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由醚氧桥联四羧酸配体构筑的两个一维镉(Ⅱ)配位聚合物的合成、晶体结构及荧光性质

黎 彧[1] 赵振宇*,[2] 邹训重[1] 冯安生[1] 邱文达*,[1]
([1]广东轻工职业技术学院,广东省特种建筑材料及其绿色制备工程技术研究中心/佛山市特种功能性建筑材料及其绿色制备技术工程中心,广州 510300)
([2]深圳信息职业技术学院智能制造与装备学院,深圳 518172)

摘要: 采用水热方法,用醚氧桥联四羧酸配体(H_4L)和菲咯啉(phen)、2,2′-联吡啶(2,2′-bipy)与$CdCl_2·H_2O$反应,合成了2个一维配位聚合物$[Cd_2(\mu_5\text{-}L)(phen)_2]_n$ (**1**)和$\{[Cd_2(\mu_4\text{-}L)(2,2'\text{-}bipy)_2(H_2O)_2]·2H_2O\}_n$ (**2**),并对其结构和荧光性质进行了研究。结构分析结果表明2个配合物分别属于单斜和三斜晶系,$P2_1/c$和$P\bar{1}$空间群。聚合物**1**和**2**分别具有基于四核Cd_4和Cd_2单元的一维链结构。研究表明,配合物**1**和**2**在室温下能发出蓝色荧光。

关键词: 配位聚合物; 四羧酸配体; 荧光
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Syntheses, Crystal Structures, and Luminescent Property of Two 1D Cadmium(Ⅱ) Coordination Polymers Assembled from an Ether-Bridged Tetracarboxylic Acid

LI Yu[1] ZHAO Zhen-Yu*,[2] ZOU Xun-Zhong[1] FENG An-Sheng[1] QIU Wen-Da*,[1]
([1]*Guangdong Research Center for Special Building Materials and Its Green Preparation Technology/Foshan Research Center for Special Functional Building Materials and Its Green Preparation Technology, Guangdong Industry Polytechnic, Guangzhou* 510300, *China*)
([2]*School of Intelligent Manufacturing and Equipment, Shenzhen Institute of Information Technology, Shenzhen, Guangdong* 518172, *China*)

Abstract: Two 1D cadmium(Ⅱ) coordination polymers, namely $[Cd_2(\mu_5\text{-}L)(phen)_2]_n$ (**1**) and $\{[Cd_2(\mu_4\text{-}L)(2,2'\text{-}bipy)_2(H_2O)_2]·2H_2O\}_n$ (**2**), have been constructed hydrothermally using H_4L (H_4L=2,3,3′,4′-diphenyl ether tetracarboxylic acid), phen (phen=1,10-phenanthroline), 2,2′-bipy (2,2′-bipy=2,2′-bipyridine), and cadmium chloride. Single-crystal X-ray diffraction analyses reveal that two polymers crystallize in the monoclinic or triclinic systems, space groups $P2_1/c$ or $P\bar{1}$. Polymers **1** and **2** show 1D chains composed of Cd_4 or Cd_2 units. The luminescent properties for two compounds were also investigated. CCDC: 1971327, **1**; 1971328, **2**.

Keywords: coordination polymer; tetracarboxylic acid; luminescent properties

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*通信联系人。E-mail:qiuwd123@163.com,shzhzhao123@163.com

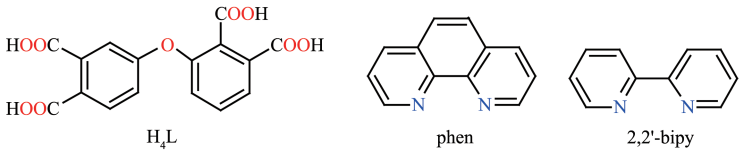
0 Introduction

The design and synthesis of functional coordination polymers have become the subjects of extensive investigation owing to their intriguing architectures and functional properties, such as magnetism, catalysis, luminescence, and gas storage[1-5]. However, there still remains a great challenge for directional construction of functional coordination polymers with predictable molecular structures and expected properties, because a lot of factors influence the construction of complexes, such as the structural features of organic ligands, the coordination requirements of metal ions, solvent systems, temperatures, and pH values[6-10].

In this context, various types of aromatic polycarboxylic acids have been extensively utilized to synthesize various coordination polymers owing to their strong coordination ability in diverse modes and the fact that they are able to satisfy the geometric requirement of the metal centers[2-3,6,10-14].

As a combination of the aforementioned aspects and our previous research work, we have selected a new asymmetric tetracarboxylate ligand, 2,3,3′,4′-diphenyl ether tetracarboxylic acid (H_4L, Scheme 1) and explored it for the construction of novel coordination polymers. The selection of H_4L has been governed by the following reasons. (1) This ligand contains two phenyl rings that are interconnected by a rotatable *O*-ether group providing a subtle conformational adaptation. (2) H_4L contains two different types of functionalities (*i.e.*, -COOH and *O*-ether) and has nine potential coordination sites, which can result in diverse coordination patterns and high dimensionalities, especially when acting as a multiply bridging spacer. (3) This tetracarboxylic acid block remains poorly used for the generation of coordination polymers.

In this work, we report the syntheses, crystal structures, and luminescent properties of two Cd(Ⅱ) coordination polymers constructed from the tetracarboxylate ligand.



H_4L　　phen　　2,2'-bipy

Scheme 1　Structures of H_4L and the auxiliary ligands

1 Experimental

1.1 Reagents and physical measurements

All chemicals and solvents were of AR grade and used without further purification. H_4L was acquired from Jinan Henghua Sci. & Technol. Co., Ltd. Carbon, hydrogen and nitrogen were determined using an Elementar Vario EL elemental analyzer. IR spectra were recorded using KBr pellets and a Bruker EQUINOX 55 spectrometer. Thermogravimetric analysis (TGA) data were collected on a LINSEIS STA PT1600 thermal analyzer with a heating rate of 10 ℃·min^{-1}. Excitation and emission spectra were recorded on an Edinburgh FLS920 fluorescence spectrometer using the solid samples at room temperature.

1.2 Synthesis of $[Cd_2(\mu_5\text{-}L)(phen)_2]_n$ (1)

A mixture of $CdCl_2\cdot H_2O$ (0.040 g, 0.20 mmol), H_4L (0.035 g, 0.10 mmol), phen (0.040 g, 0.20 mmol), NaOH (0.016 g, 0.40 mmol), and H_2O (10 mL) was stirred at room temperature for 15 min, and then sealed in a 25 mL Teflon-lined stainless steel vessel, and heated at 160 ℃ for 3 days, followed by cooling to room temperature at a rate of 10 ℃·h^{-1}. Colourless block-shaped crystals of **1** were isolated manually, and washed with distilled water. Yield: 43% (based on H_4L). Anal. Calcd. for $C_{40}H_{22}Cd_2N_4O_9$(%): C 51.80, H 2.39, N 6.04; Found(%): C 51.93, H 2.38, N 6.01. IR (KBr, cm^{-1}): 1 588s, 1 515w, 1 431w, 1 397s, 1 363m, 1 256w, 1 240w, 1 144w, 1 099w, 1 060w, 980w, 924w, 851m, 783w, 727m, 699w, 637w.

1.3 Synthesis of $\{[Cd_2(\mu_4\text{-}L)(2,2'\text{-bipy})_2(H_2O)_2]\cdot 2H_2O\}_n$ (2)

The preparation of **2** was similar to that of **1** except 2,2′-bipy was used instead of phen. After cool-

ing the reaction mixture to room temperature, colourless block-shaped crystals of **2** were isolated manually, washed with distilled water, and dried. Yield: 48% (based on H_4L). Anal. Calcd. for $C_{36}H_{30}Cd_2N_4O_{13}$(%): C 45.44, H 3.18, N 5.89; Found(%): C 45.21, H 3.16, N 5.93. IR (KBr, cm^{-1}): 3 390w, 3 074w, 1 567s, 1 470w, 1 436m, 1 391s, 1 313w, 1 256w, 1 234m, 1 154w, 1 060w, 1 015w, 980w, 856w, 823w, 806w, 766m, 733w, 699w, 654w.

The compounds are insoluble in water and common organic solvents, such as methanol, ethanol, acetone, and DMF.

1.4 Structure determinations

Two single crystals with dimensions of 0.22 mm×0.21 mm×0.19 mm (**1**) and 0.21 mm×0.20 mm×0.18 mm (**2**) were collected at 293(2) K on a Bruker SMART APEX Ⅱ CCD diffractometer with Mo $K\alpha$ radiation (λ= 0.071 073 nm). The structures were solved by direct methods and refined by full matrix least-square on F^2 using the SHELXTL-2014 program[15]. All non-hydrogen atoms were refined anisotropically. All the hydrogen atoms were positioned geometrically and refined using a riding model. A summary of the crystallography data and structure refinements for **1** and **2** is given in Table 1. The selected bond lengths and angles for compounds **1** and **2** are listed in Table 2. Hydrogen bond parameters of compound **2** are given in Table 3.

CCDC: 1971327, **1**; 1971328, **2**.

Table 1 Crystal data for compounds 1 and 2

Compound	1	2
Chemical formula	$C_{40}H_{22}Cd_2N_4O_9$	$C_{36}H_{30}Cd_2N_4O_{13}$
Formula weight	927.41	951.44
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/c$	$P\bar{1}$
a / nm	1.184 09(8)	0.930 77(8)
b / nm	1.178 61(11)	1.191 97(11)
c / nm	2.389 62(18)	1.713 64(11)
α / (°)		97.116(6)
β / (°)	91.964(6)	92.986(6)
γ / (°)		109.187(8)
V / nm^3	3.332 9(5)	1.773 0(3)
Z	4	2
F(000)	1 832	948
θ range for data collection / (°)	3.412~25.042	3.389~25.050
Limiting indices	$-14 \leqslant h \leqslant 14$, $-14 \leqslant k \leqslant 11$, $-28 \leqslant l \leqslant 23$	$-11 \leqslant h \leqslant 11$, $-14 \leqslant k \leqslant 13$, $-20 \leqslant l \leqslant 17$
Reflection collected, unique (R_{int})	20 338, 5 885 (0.107 9)	11 174, 6 248 (0.068 6)
D_c / ($Mg \cdot m^{-3}$)	1.848	1.782
μ / mm^{-1}	1.345	1.274
Data, restraint, parameter	5 885, 0, 496	6 248, 0, 496
Goodness-of-fit on F^2	0.848	1.056
Final R indices [$I \geqslant 2\sigma(I)$] R_1, wR_2	0.056 7, 0.080 6	0.063 9, 0.080 4
R indices (all data) R_1, wR_2	0.101 2, 0.110 8	0.103 8, 0.131 1
Largest diff. peak and hole / ($e \cdot nm^{-3}$)	916 and −707	785 and −928

Table 2 Selected bond distances (nm) and bond angles (°) for compounds 1 and 2

1					
Cd(1)-O(1)	0.253 3(5)	Cd(1)-O(3)	0.232 3(5)	Cd(1)-O(4)A	0.224 7(5)
Cd(1)-O(8)B	0.218 9(6)	Cd(1)-N(1)	0.237 0(6)	Cd(1)-N(2)	0.238 1(6)
Cd(2)-O(1)	0.233 1(5)	Cd(2)-O(2)	0.236 8(5)	Cd(2)-O(6)B	0.240 1(5)

Continued Table 1

Cd(2)-O(7)B	0.231 8(5)	Cd(2)-N(3)	0.232 2(7)	Cd(2)-N(4)	0.228 6(6)
O(8)B-Cd(1)-O(4)A	97.8(2)	O(8)B-Cd(1)-O(3)	151.7(2)	O(4)A-Cd(1)-O(3)	96.82(18)
N(1)-Cd(1)-O(8)B	91.3(2)	O(4)A-Cd(1)-N(1)	151.6(2)	O(3)-Cd(1)-N(1)	87.15(19)
O(8)B-Cd(1)-N(2)	123.8(2)	O(4)A-Cd(1)-N(2)	82.67(19)	O(3)-Cd(1)-N(2)	82.0(2)
N(1)-Cd(1)-N(2)	70.0(2)	O(8)B-Cd(1)-O(1)	75.1(2)	O(1)-Cdo(1)-O(4)A	106.77(18)
O(3)-Cd(1)-O(1)	77.60(18)	O(1)-Cd(1)-N(1)	101.6(2)	O(1)-Cd(1)-N(2)	158.38(17)
O(7)B-Cd(2)-N(4)	132.1(2)	N(3)-Cd(2)-N(4)	72.2(2)	O(7)B-Cd(2)-N(3)	103.3(2)
N(4)-Cd(2)-O(1)	117.2(2)	O(1)-Cd(2)-O(7)B	99.83(19)	O(1)-Cd(2)-N(3)	134.6(2)
O(2)-Cd(2)-N(4)	134.2(2)	O(7)B-Cd(2)-O(2)	91.06(19)	O(2)-Cd(2)-N(3)	85.06(19)
O(1)-Cd(2)-O(2)	55.76(17)	O(6)B-Cd(2)-N(4)	84.4(2)	O(7)B-Cd(2)-O(6)B	55.24(17)
N(3)-Cd(2)-O(6)B	118.7(2)	O(1)-Cd(2)-O(6)B	106.64(19)	O(2)-Cd(2)-O(6)B	140.98(18)
		2			
Cd(1)-O(1)	0.256 2(6)	Cd(1)-O(2)	0.228 4(5)	Cd(1)-O(4)A	0.227 9(6)
Cd(1)-O(10)	0.235 5(6)	Cd(1)-N(1)	0.233 3(7)	Cd(1)-N(2)	0.232 3(6)
Cd(2)-O(6)B	0.250 0(6)	Cd(2)-O(7)B	0.236 2(7)	Cd(2)-O(8)	0.236 6(5)
Cd(2)-O(9)	0.242 7(6)	Cd(2)-O(11)	0.238 4(6)	Cd(2)-N(3)	0.229 7(8)
Cd(2)-N(4)	0.230 2(7)				
O(2)-Cd(1)-O(4)A	108.2(2)	O(4)A-Cd(1)-N(2)	97.8(2)	O(2)-Cd(1)-N(2)	153.9(2)
N(1)-Cd(1)-O(4)A	117.7(2)	O(2)-Cd(1)-N(1)	97.2(2)	N(2)-Cd(1)-N(1)	69.8(2)
O(4)A-Cd(1)-O(10)	79.0(2)	O(10)-Cd(1)-O(2)	95.0(2)	O(10)-Cd(1)-N(2)	89.9(2)
N(1)-Cd(1)-O(10)	154.6(2)	O(4)A-Cd(1)-O(1)	149.9(2)	O(1)-Cdo(1)-O(2)	53.56(18)
N(2)-Cd(1)-O(1)	102.6(2)	O(1)-Cd(1)-N(1)	90.2(2)	O(1)-Cd(1)-O(10)	79.3(2)
N(3)-Cd(2)-N(4)	70.9(3)	N(3)-Cd(2)-O(7)B	96.5(3)	O(7)B-Cd(2)-N(4)	130.1(2)
N(3)-Cd(2)-O(8)	99.3(2)	N(4)-Cd(2)-O(8)	139.7(2)	O(7)B-Cd(2)-O(8)	89.1(2)
O(11)-Cd(2)-N(3)	165.4(3)	O(11)-Cd(2)-N(4)	95.8(3)	O(7)B-Cd(2)-O(11)	96.8(2)
O(8)-Cd(2)-O(11)	86.9(2)	O(9)-Cd(2)-N(3)	88.5(2)	O(9)-Cd(2)-N(4)	86.4(2)
O(7)B-Cd(2)-O(9)	142.8(2)	O(8)-Cd(2)-O(9)	53.73(19)	O(11)-Cd(2)-O(9)	84.5(2)
N(3)-Cd(2)-O(6)B	109.5(2)	N(4)-Cd(2)-O(6)B	85.9(2)	O(7)B-Cd(2)-O(6)B	52.1(2)
O(8)-Cd(2)-O(6)B	132.9(2)	O(11)-Cd(2)-O(6)B	74.5(2)	O(9)-Cd(2)-O(6)B	156.8(3)

Symmetry transformations used to generate equivalent atoms: A: $-x$, $-y$+2, $-z$+1; B: x−1, y, z for **1**; A: $-x$+1, $-y$+1, $-z$+1; B: $-x$, $-y$, $-z$+2 for **2**.

Table 3　Hydrogen bond parameters of compound 2

D−H···A	d(D-H) / nm	d(H···A) / nm	d(D···A) / nm	∠DHA / (°)
O(10)−H(1W)···O(1)A	0.085 0	0.196 5	0.281 5	179.74
O(10)−H(2W)···O(12)	0.089 0	0.177 9	0.264 7	164.37
O(11)−H(3W)···O(8)B	0.081 3	0.194 6	0.275 3	172.06
O(11)−H(4W)···O(13)C	0.072 2	0.208 2	0.278 5	164.98
O(12)−H(5W)···O(2)D	0.085 0	0.202 0	0.287 0	179.20
O(12)−H(6W)···O(3)	0.085 0	0.187 3	0.272 3	178.95
O(13)−H(7W)···O(7)E	0.079 9	0.158 0	0.237 2	170.57

Symmetry codes: A: $-x$+2, $-y$+1, $-z$+1; B: $-x$, $-y$, $-z$+2; C: x, y−1, z+1; D: $-x$+1, $-y$+1, $-z$+1; E: x, y+1, z−1.

2　Results and discussion

2.1　Description of the structures

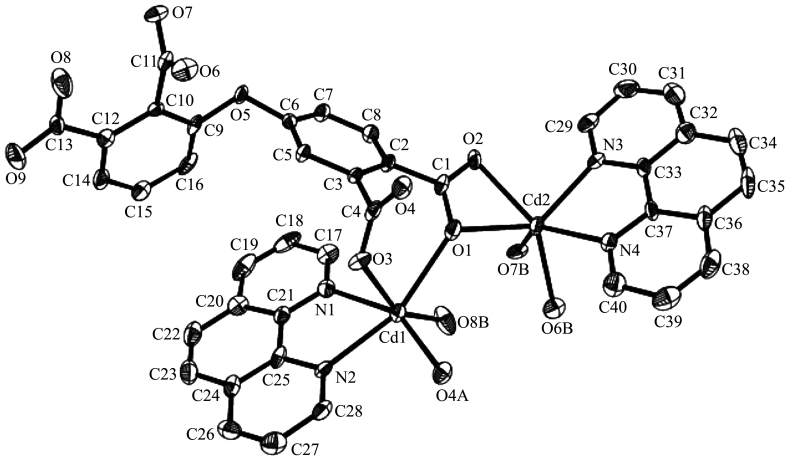
2.1.1　$[Cd_2(\mu_5\text{-L})(phen)_2]_n$ (**1**)

The molecular structure of compound **1** is shown in Fig. 1. The asymmetric unit contains two Cd atoms (Cd1 and Cd2), a μ_5-L^{4-} ligand, and two phen moieties.

The six-coordinated Cd1 center displays a distorted octahedral $\{CdN_2O_4\}$ environment filled by four carboxylate O atoms from three individual μ_5-L^{4-} blocks and two N atoms from the phen moiety. The Cd2 center is also six-coordinated and features a distorted octahedral $\{CdN_2O_4\}$ geometry that is taken by four carboxylate O atoms from two different μ_5-L^{4-} blocks and two N atoms from the phen moiety. The lengths of the Cd-O and Cd-N bonds are 0.218 9(6)~0.253 3(5) and 0.228 6(6)~0.238 1(6) nm, respectively, which are within the normal values for related Cd(Ⅱ) derivatives[6,10,16]. In **1**, the L^{4-} block acts as a μ_5-ligand, wherein the COO^- groups are monodentate, bidentate or tridentate (mode Ⅰ, Scheme 2). The carboxylate groups of four μ_5-L^{4-} blocks link the adjacent Cd(Ⅱ) centers into a Cd_4 subunit (Fig. 2). Such subunits are further assembled via the remaining COO^- groups of μ_5-L^{4-} blocks to a 1D chain (Fig.2).

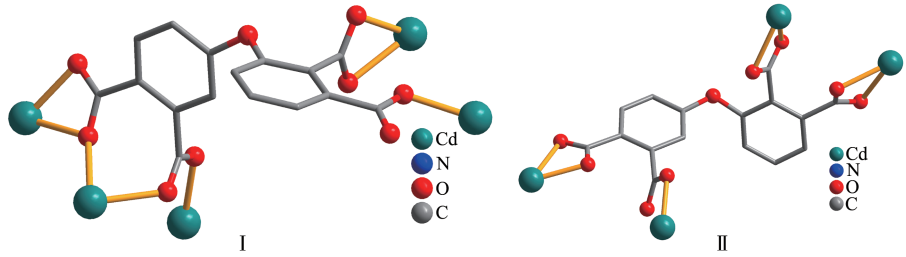
2.1.2 $\{[Cd_2(\mu_4\text{-L})(2,2'\text{-bipy})_2(H_2O)_2]\cdot 2H_2O\}_n$ (**2**)

The asymmetric unit of **2** bears two Cd(Ⅱ) centers (Cd1 and Cd2), one μ_4-L^{4-} linker, two 2,2′-bipy moieties, two terminal water ligands and two lattice water molecules (Fig. 3). The seven-coordinated Cd1 center adopts a distorted pentagonal bipyramid $\{CdN_2O_5\}$ environment taken by four carboxylate O atoms from two independent μ_4-L^{4-} blocks, one O atom from the H_2O li-

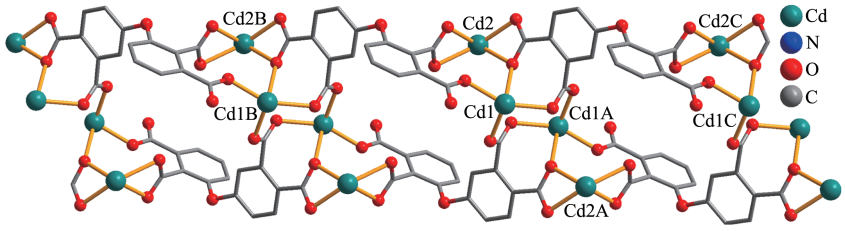


H atoms were omitted for clarity; Symmetry codes: A: $-x$, $-y+2$, $-z+1$; B: $x-1$, y, z

Fig.1 Drawing of asymmetric unit of compound **1** with 30% probability thermal ellipsoids



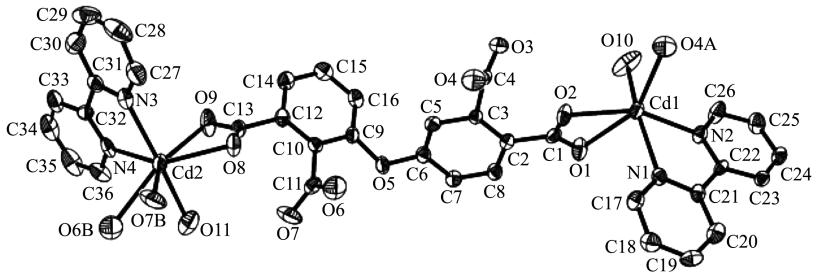
Scheme 2 Coordination modes of L^{4-} ligands in compounds **1** and **2**



Phen ligands are omitted for clarity; Symmetry codes: A: $-x$, $-y+2$, $-z+1$; B: $x-1$, y, z; C: $x+1$, y, z

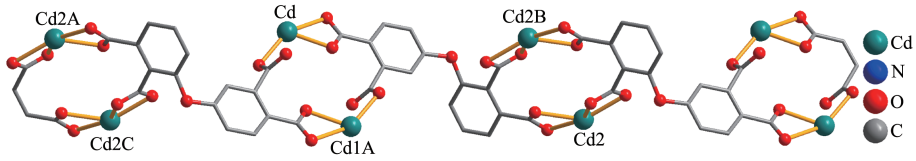
Fig.2 View of 1D chain in compound **1** along *b* axis

gand, and a pair of N atoms from the 2,2′-bipy moiety. The Cd2 center is six-coordinated and reveals a distorted octahedral $\{CdN_2O_4\}$ geometry. It is completed by three carboxylate O donors from two μ_4-L^{4-} blocks, one O atom from the H_2O ligand, and two N donors from the 2,2′-bipy moiety. The Cd-O (0.227 9(6)~0.256 2(6) nm) and Cd-N (0.229 7(8)~0.233 3(7) nm) bonds are within standard values[14,16-17]. The L^{4-} block behaves as a heptadentate μ_4-linker (Scheme 2, mode Ⅱ). Two μ_4-L^{4-} blocks link adjacent Cd(Ⅱ) centers to form a Cd_2 unit (Fig. 4). Furthermore, these Cd_2 units are further extended by μ_4-L^{4-} blocks into a 1D chain (Fig.4). Compounds **1** and **2** were obtained under similar reaction conditions except using different auxiliary ligands (phen for **1** and 2,2′-bipy for **2**), which result in different structures.



Lattice water molecule and H atoms were omitted for clarity; Symmetry codes: A: −*x*+1, −*y*+1, −*z*+1; B: −*x*, −*y*, −*z*+2

Fig.3　Drawing of asymmetric unit of compound **2** with 30% probability thermal ellipsoids



2,2′−Bipy ligands are omitted for clarity; Symmetry codes: A: −*x*+1, −*y*+1, −*z*+1; B: −*x*, −*y*, −*z*+2; C: *x*+1, *y*+1, *z*−1

Fig.4　View of 1D chain in compound **2** along *a* axis

2.2　TGA analysis

To determine the thermal stability of compounds **1** and **2**, their thermal behaviors were investigated under nitrogen atmosphere by thermogravimetric analysis (TGA). As shown in Fig.5, TGA curve of compound **1** shows that the sample remained stable until 327 ℃. Compound **2** lost its two lattice water molecules and two H_2O ligands in a range of 45~157 ℃ (Obsd. 7.2%; Calcd. 7.6%), followed by the decomposition at 254 ℃.

2.3　Luminescent properties

Solid-state emission spectra of H_4L and cadmium(Ⅱ) compounds **1** and **2** were measured at room temperature (Fig.6). The spectrum of H_4L revealed a weak emis-

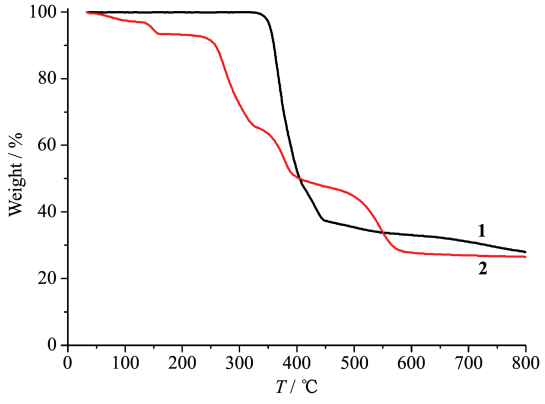


Fig.5　TGA curves of compounds **1** and **2**

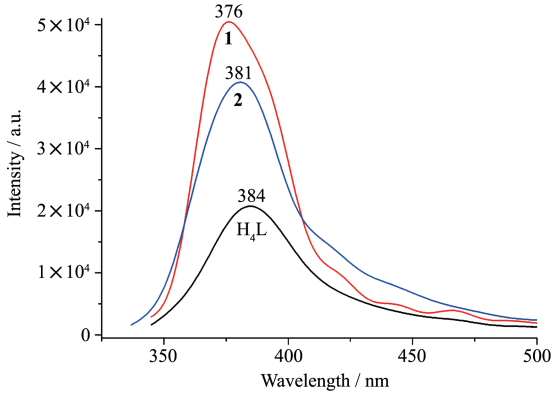


Fig.6　Solid-state emission spectra of H_4L and compounds **1** and **2** at room temperature

sion with a maximum at 384 nm (λ_{ex}=320 nm). In comparison with H_4L, compounds **1** and **2** exhibited more extensive emission with maximum at 376 and 381 nm (λ_{ex}=320 nm). These emissions correspond to intraligand π-π* or n-π* transition of H_2L[6,10,17]. Enhancement of the luminescence in **1** and **2** compared to H_4L can be explained by the coordination of ligands to Cd(Ⅱ); the coordination can augment a rigidity of ligands and reduce an energy loss due to radiationless decay[10,16-17].

3 Conclusions

In summary, we have successfully synthesized and characterized two new cadmium coordination polymers by using a tetracarboxylate acid as a ligand under hydrothermal condition. Compounds **1** and **2** show 1D chains composed of Cd_4 and Cd_2 units, respectively. Besides, the luminescent properties were also investigated and discussed. The results show that such tetracarboxylic acid can be used as a versatile multifunctional building block toward the generation of new coordination polymers.

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