

一维镉(II)和镍(II)配位聚合物的合成、晶体结构、荧光及磁性质

黎 彧 陈泳璇 赵 娜* 冯安生 邹训重*

(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)

摘要: 在水热条件下, 用联苯类四羧酸配体(H_4L)和菲咯啉(phen)分别与 $CdCl_2 \cdot H_2O$ 和 $NiCl_2 \cdot 6H_2O$ 反应, 合成了 2 个具有一维链结构的配合物 $\{[M_2(\mu_3-L)(phen)_3] \cdot 5H_2O\}_n$ ($M=Cd$ (**1**), Ni (**2**)), 并对其结构、荧光和磁性质进行了研究。结构分析结果表明 2 个配合物是异质同心的, 属于正交晶系, $Pbca$ 空间群。2 个配合物具有一维链结构, 而且这些链通过 $O-H \cdots O$ 氢键作用进一步形成了二维超分子网络。研究表明, 配合物 **1** 在室温下能发出蓝色荧光, 配合物 **2** 中相邻 $Ni(II)$ 离子间存在铁磁相互作用。

关键词: 配位聚合物; 四羧酸配体; 荧光; 磁性

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Syntheses, Crystal Structures, Luminescent and Magnetic Properties of Two 1D Cadmium(II) and Nickel(II) Coordination Polymers

LI Yu CHEN Yong-Xuan ZHAO Na* FENG An-Sheng ZOU Xun-Zhong*

(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)

Abstract: Two 1D cadmium(II) and nickel(II) coordination polymers, namely $\{[M_2(\mu_3-L)(phen)_3] \cdot 5H_2O\}_n$ ($M=Cd$ (**1**), Ni (**2**)), were constructed hydrothermally using H_4L ($H_4L=3$ -(2,4-dicarboxyphenyl)-2,6-pyridinedicarboxylic acid), phen (phen=1,10-phenanthroline), and cadmium or nickel chlorides. Single-crystal X-ray diffraction analyses reveal that both compounds are isostructural and crystallize in the orthorhombic system, space group $Pbca$. Compound **1** or **2** feature a 1D metal-organic chain, which is assembled to a 2D supramolecular network through $O-H \cdots O$ hydrogen bond. The luminescent and magnetic properties for two compounds were also investigated. CCDC: 1915948, **1**; 1915949, **2**.

Keywords: coordination polymer; tetracarboxylic acid; luminescent properties; magnetic properties

0 Introduction

In the past few decades, the design and hydrothermal syntheses of functional coordination polymers

have attracted tremendous attention owing to their fascinating architectures and topologies, as well as potential applications in catalysis, magnetism, luminescence, and gas absorption^[1-8]. However, it is difficult

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*通信联系人。E-mail: 2017018009@gdip.edu.cn, 2012009035@gdip.edu.cn

to predict the structures of coordination polymers, 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^[9-15].

In this regard, various types of aromatic polycarboxylic acids have been proved to be versatile and efficient candidates for constructing diverse coordination polymers due to their rich coordination chemistry, tunable degree of deprotonation, and ability to act as H-bond acceptors and donors^[11,13,15-19].

As a combination of the aforementioned aspects and our previous research work, we have selected a novel biphenyl tetracarboxylate ligand, 3-(2,4-dicarboxyphenyl)-2,6-pyridinedicarboxylic acid (H_4L) and explored it for the construction of novel coordination polymers. The H_4L block possesses the following features: (1) it can twist and rotate freely to generate different angles between the two aromatic planes via the C-C bond to furnish a subtle conformational adaptation; (2) it has nine potential coordination sites (eight carboxylate O donors and one N donor), which can lead to diverse coordination patterns and high dimensionalities, especially when acting as a multiply bridging spacer; (3) apart from a limited number of coordination compounds derived from H_4L , this acid block remains poorly used for the generation of coordination polymers^[20]. Given these features, the main objective of the present study consisted in the exploration of H_4L as a novel biphenyl tetracarboxylate block for the assembly of diverse metal-organic networks.

In this work, we report the syntheses, crystal structures, luminescent and magnetic properties of two Cd(II) and Ni(II) coordination polymers constructed from biphenyl-type tetracarboxylate ligands.

1 Experimental

1.1 Reagents and physical measurements

All chemicals and solvents were of AR grade and used without further purification. Carbon, hydrogen and nitrogen were determined using an Elementar Vario EL elemental analyzer. IR spectra were recorded by a Bruker EQUINOX 55 spectrometer using KBr

pellets. Thermogravimetric analysis (TGA) data were collected on a LINSEIS STA PT1600 thermal analyzer with a heating rate of $10\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$. Excitation and emission spectra were recorded on an Edinburgh FLS920 fluorescence spectrometer using the solid samples at room temperature. Magnetic susceptibility data were collected in the 2~300 K temperature range on a Quantum Design SQUID Magnetometer MPMS XL-7 with a field of 0.1 T. A correction was made for the diamagnetic contribution prior to data analysis.

1.2 Synthesis of $\{[Cd_2(\mu_3-L)(phen)_3]\cdot 5H_2O\}_n$ (**1**)

A mixture of $CdCl_2\cdot H_2O$ (0.040 g, 0.20 mmol), H_4L (0.033 g, 0.10 mmol), phen (0.060 g, 0.3 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 $160\text{ }^{\circ}\text{C}$ for 3 days, followed by cooling to room temperature at a rate of $10\text{ }^{\circ}\text{C}\cdot\text{h}^{-1}$. Colourless block-shaped crystals of **1** were isolated manually, and washed with distilled water. Yield: 52% (based on H_4L). Anal. Calcd. for $C_{51}H_{39}Cd_2N_7O_{13}(\%)$: C 51.79, H 3.32, N 8.29; Found(%): C 51.64, H 3.31, N 8.32. IR (KBr, cm^{-1}): 3 540w, 3 055w, 1 607s, 1 556s, 1 515m, 1 454w, 1 424m, 1 357s, 1 275w, 1 250w, 1 220w, 1 184w, 1 138w, 1 102w, 1 016w, 908w, 853m, 817w, 781w, 730w, 684w, 638w.

1.3 Synthesis of $\{[Ni_2(\mu_3-L)(phen)_3]\cdot 5H_2O\}_n$ (**2**)

The preparation of **2** was similar to that of **1** except $NiCl_2\cdot 6H_2O$ was used instead of $CdCl_2\cdot H_2O$. After cooling the reaction mixture to room temperature, green block-shaped crystals of **2** were isolated manually, washed with distilled water, and dried. Yield: 61% (based on H_4L). Anal. Calcd. for $C_{51}H_{39}Ni_2N_7O_{13}(\%)$: C, 56.97; H, 3.66; N, 9.12. Found(%): C, 57.13; H, 3.65; N, 9.07%. IR (KBr, cm^{-1}): 3 572m, 3 045w, 1 618s, 1 541s, 1 510m, 1 464w, 1 408m, 1 367s, 1 275w, 1 209w, 1 184w, 1 148w, 1 097w, 1 010w, 954w, 913w, 857m, 817w, 786w, 765w, 724m, 678w, 664w.

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 $0.26\text{ mm}\times$

0.24 mm×0.23 mm (**1**) and 0.26 mm×0.22 mm×0.21 mm (**2**) were collected at 293(2) K on a Bruker SMART APEX II CCD diffractometer with Mo $K\alpha$ radiation ($\lambda=0.071\ 073$ nm) for **1** and Cu $K\alpha$ radiation ($\lambda=0.154\ 184$ nm) for **2**. The structures were solved by direct methods and refined by full matrix least-square on F^2 using the SHELXTL-2014 program^[21]. All non-hydrogen atoms were refined anisotropically. All the hydrogen atoms were positioned geometrically and refined using a riding model. Disordered solvent

molecules in **1** and **2** were removed using the SQUEEZE routine in PLATON^[22]. The number of solvent water molecules was obtained on the basis of elemental and thermogravimetric analyses. 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 compounds **1** and **2** are given in Table 3 and 4.

CCDC: 1915948, **1**; 1915949, **2**.

Table 1 Crystal data for compounds **1** and **2**

Compound	1	2
Chemical formula	C ₅₁ H ₃₉ Cd ₂ N ₇ O ₁₃	C ₅₁ H ₃₉ Ni ₂ N ₇ O ₁₃
Molecular weight	1 182.72	1 075.31
Crystal system	Orthorhombic	Orthorhombic
Space group	<i>Pbca</i>	<i>Pbca</i>
<i>a</i> / nm	1.845 88(7)	1.809 05(5)
<i>b</i> / nm	1.691 95(6)	1.676 82(4)
<i>c</i> / nm	3.012 97(11)	3.016 46(8)
<i>V</i> / nm ³	9.409 9(6)	9.150 3(4)
<i>Z</i>	8	8
<i>F</i> (000)	4 512	4 272
θ range for data collection / (°)	3.336~25.049	3.816~ 69.987
Limiting indices	$-12 \leq h \leq 21, -19 \leq k \leq 20, -35 \leq l \leq 35$	$-20 \leq h \leq 21, -20 \leq k \leq 13, -36 \leq l \leq 36$
Reflection collected, unique (R_{int})	35 201, 8 304 (0.087 9)	36 483, 8 579 (0.099 0)
D_c / (g·cm ⁻³)	1.593	1.509
μ / mm ⁻¹	0.972	1.633
Data, restraint, parameter	8 304, 0, 631	8 579, 0, 640
Goodness-of-fit on F^2	1.142	1.054
Final <i>R</i> indices [$I \geq 2\sigma(I)$] R_1, wR_2	0.05 63, 0.097 6	0.075 3, 0.157 5
<i>R</i> indices (all data) R_1, wR_2	0.078 1, 0.092 4	0.125 2, 0.183 5
Largest diff. peak and hole / (e·nm ⁻³)	1 079 and -464	359 and -666

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

1					
Cd(1)-O(1)	0.243 6(4)	Cd(1)-O(3)	0.232 7(4)	Cd(1)-O(8)A	0.220 6(4)
Cd(1)-N(1)	0.228 7(4)	Cd(1)-N(2)	0.232 7(5)	Cd(1)-N(3)	0.238 6(5)
Cd(2)-O(2)	0.226 4(4)	Cd(2)-O(5)	0.227 4(4)	Cd(2)-N(4)	0.234 3(5)
Cd(2)-N(5)	0.235 8(5)	Cd(2)-N(6)	0.229 4(5)	Cd(2)-N(7)	0.239 4(5)
O(8)A-Cd(1)-N(1)	104.30(15)	O(8)A-Cd(1)-N(2)	148.44(18)	N(1)-Cd(1)-N(2)	103.70(18)
O(8)A-Cd(1)-O(3)	105.04(15)	N(1)-Cd(1)-O(3)	71.99(15)	N(2)-Cd(1)-O(3)	97.21(17)
O(8)A-Cd(1)-N(3)	81.51(16)	N(1)-Cd(1)-N(3)	173.90(17)	N(2)-Cd(1)-N(3)	70.21(19)
O(3)-Cd(1)-N(3)	108.59(16)	O(8)A-Cd(1)-O(1)	92.88(15)	N(1)-Cd(1)-O(1)	67.87(15)
N(2)-Cd(1)-O(1)	84.41(16)	O(3)-Cd(1)-O(1)	138.96(14)	N(3)-Cd(1)-O(1)	110.42(15)

Continued Table 2

O(2)-Cd(2)-O(5)	86.00(15)	O(2)-Cd(2)-N(6)	102.27(16)	O(5)-Cd(2)-N(6)	90.42(18)
O(2)-Cd(2)-N(4)	92.71(16)	O(5)-Cd(2)-N(4)	105.03(16)	N(6)-Cd(2)-N(4)	159.25(18)
O(2)-Cd(2)-N(5)	161.74(15)	O(5)-Cd(2)-N(5)	89.59(17)	N(6)-Cd(2)-N(5)	95.46(18)
N(4)-Cd(2)-N(5)	71.34(18)	O(2)-Cd(2)-N(7)	98.13(15)	O(5)-Cd(2)-N(7)	161.38(17)
N(6)-Cd(2)-N(7)	71.0(2)	N(4)-Cd(2)-N(7)	92.95(18)	N(5)-Cd(2)-N(7)	91.68(16)
2					
Ni(1)-O(1)	0.210 0(4)	Ni(1)-O(5)	0.204 0(3)	Ni(1)-N(2)	0.212 4(4)
Ni(1)-N(3)	0.213 3(4)	Ni(1)-N(4)	0.217 0(4)	Ni(1)-N(5)	0.213 1(4)
Ni(2)-O(4)A	0.203 2(4)	Ni(2)-O(6)	0.215 4(3)	Ni(2)-O(7)	0.214 7(4)
Ni(2)-N(1)	0.205 8(4)	Ni(2)-N(6)	0.213 6(5)	Ni(2)-N(7)	0.214 8(5)
O(5)-Ni(1)-O(1)	86.47(15)	O(5)-Ni(1)-N(2)	88.73(15)	O(1)-Ni(1)-N(2)	102.10(16)
O(5)-Ni(1)-N(5)	98.26(16)	O(1)-Ni(1)-N(5)	88.49(16)	N(2)-Ni(1)-N(5)	167.69(17)
N(3)-Ni(1)-O(5)	163.93(15)	O(1)-Ni(1)-N(3)	88.01(16)	N(3)-Ni(1)-N(2)	77.69(17)
N(5)-Ni(1)-N(3)	96.67(17)	N(4)-Ni(1)-O(5)	96.27(15)	O(1)-Ni(1)-N(4)	165.73(16)
N(2)-Ni(1)-N(4)	91.98(17)	N(5)-Ni(1)-N(4)	77.25(17)	N(4)-Ni(1)-N(3)	92.78(16)
O(4)A-Ni(2)-N(1)	97.75(15)	O(4)A-Ni(2)-N(6)	160.40(17)	N(1)-Ni(2)-N(6)	99.69(17)
O(4)A-Ni(2)-O(7)	100.94(15)	N(1)-Ni(2)-O(7)	76.07(15)	N(6)-Ni(2)-O(7)	91.90(16)
O(4)A-Ni(2)-N(7)	86.65(16)	N(1)-Ni(2)-N(7)	175.53(17)	N(6)-Ni(2)-N(7)	76.09(18)
N(7)-Ni(2)-O(7)	102.40(15)	O(6)-Ni(2)-O(4)A	92.34(14)	N(1)-Ni(2)-O(6)	74.99(14)
N(6)-Ni(2)-O(6)	83.65(16)	O(7)-Ni(2)-O(6)	149.48(14)	N(7)-Ni(2)-O(6)	105.72(15)

Symmetry codes: A: $-x+1, y-1/2, -z+3/2$ for **1**; A: $-x+1, y+1/2, -z+1/2$ for **2**.Table 3 Hydrogen bond parameters of compound **1**

D-H...A	$d(\text{D-H}) / \text{nm}$	$d(\text{H}\cdots\text{A}) / \text{nm}$	$d(\text{D}\cdots\text{A}) / \text{nm}$	$\angle \text{DHA} / (^\circ)$
O(9)-H(1W)···O(7)	0.085	0.188 2	0.273 1	178.6
O(9)-H(2W)···O(2)A	0.085	0.212 7	0.297 7	179.4
O(10)-H(3W)···O(6)	0.085	0.203 8	0.288 8	179.7
O(10)-H(4W)···O(4)B	0.085	0.200 4	0.285 4	179.3

Symmetry codes: A: $-x+1, y+1/2, -z+3/2$; B: $-x+1/2, y+1/2, z$.Table 4 Hydrogen bond parameters of compound **2**

D-H...A	$d(\text{D-H}) / \text{nm}$	$d(\text{H}\cdots\text{A}) / \text{nm}$	$d(\text{D}\cdots\text{A}) / \text{nm}$	$\angle \text{DHA} / (^\circ)$
O(9)-H(1W)···O(3)A	0.085	0.19 54	0.280 4	178.8
O(9)-H(2W)···O(11)B	0.085	0.203 5	0.288 5	179.5
O(10)-H(3W)···O(8)	0.085	0.200 1	0.285 1	178.9
O(10)-H(4W)···O(2)B	0.085	0.220 8	0.305 8	179.0
O(11)-H(5W)···O(8)	0.085	0.201 9	0.286 9	179.0
O(11)-H(6W)···O(2)B	0.085	0.211 5	0.296 5	179.3

Symmetry codes: A: $x, y+1, z$; B: $-x+1/2, y+1/2, z$.

2 Results and discussion

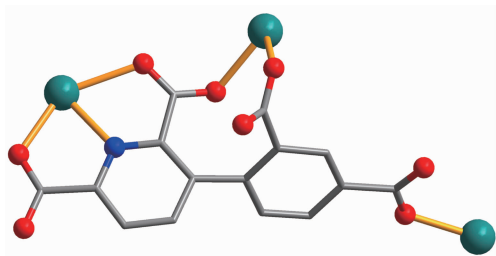
2.1 Description of the structure

2.1.1 Structure of $[\text{Cd}_2(\mu_3\text{-L})(\text{phen})_3] \cdot 5\text{H}_2\text{O}$ (**1**)

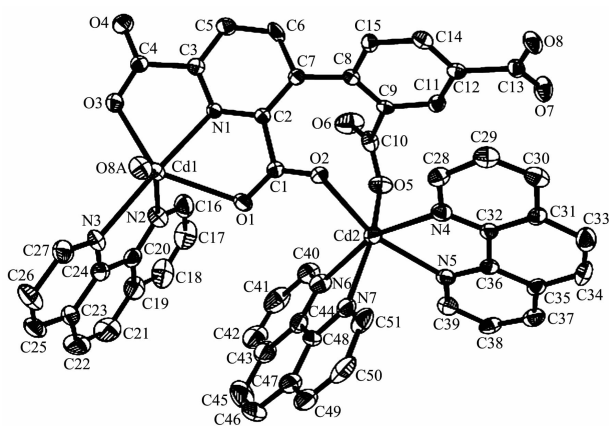
Compounds **1** and **2** are isostructural (Table 1)

and the structure of **1** is discussed in detail as an example. Asymmetric unit of **1** comprises two Cd(II) centers (Cd1 and Cd2), one $\mu_3\text{-L}^{4-}$ spacer, three phen moieties, and five lattice water molecules (Fig.1). The six-coordinated Cd1 atom adopts a distorted

octahedral $\{CdN_3O_3\}$ geometry, which is populated by one N and two O atoms from one μ_3 - L^{4-} spacer and a carboxylate O donor from another μ_3 - L^{4-} ligand, in addition to two phen N donors. The Cd2 center is also six-coordinated with a distorted octahedral $\{CdN_4O_2\}$ environment, which is filled by two O atoms of μ_3 - L^{4-} block and two pairs of phen N donors. The lengths of the Cd-O and Cd-N bonds are 0.220 6(4)~0.243 6(4) and 0.228 7(4)~0.239 4(5) nm, respectively, which are within the normal values for related Cd(II) derivatives^[11,15,23]. The L^{4-} block acts as a hexadentate μ_3 -bridging ligand (Scheme 1), in which the four carboxylate groups adopt different monodentate or bidentate bridging modes. The dihedral angle of two aromatic rings in the L^{4-} ligand is 62.53°. The carboxylate groups of L^{4-} blocks bridge alternately adjacent Cd(II) atoms to form a 1D chain (Fig.2). These chains are further extended into a 2D supramolecular network via the O-H...O hydrogen-bonding interactions (Fig.3 and Table 3).

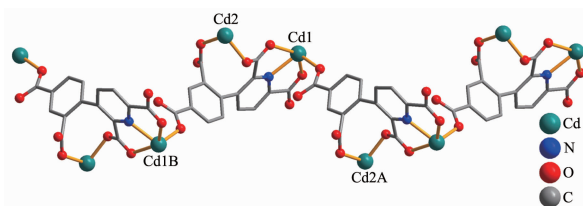


Scheme 1 Coordination mode of L^{4-} ligand in compound **1**



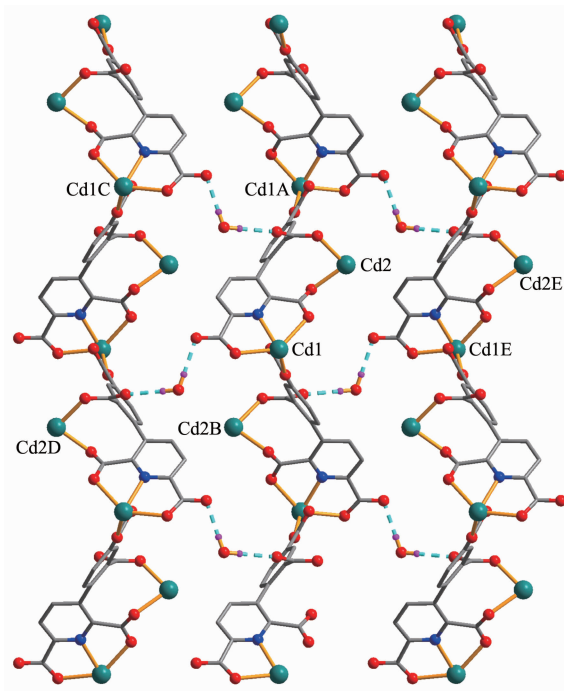
H atoms and lattice water molecules are omitted for clarity; Symmetry code: A: $-x+1, y-1/2, -z+3/2$

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



Phen ligands are omitted for clarity; Symmetry codes: A: $-x+1, y-1/2, -z+3/2$; B: $-x+1, y+1/2, -z+3/2$

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

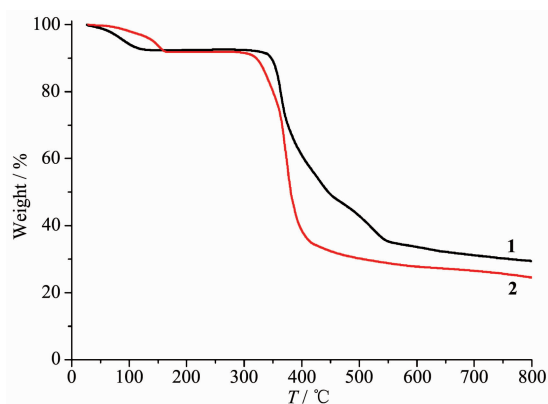


Phen ligands are omitted for clarity; Dotted lines present the H-bonds; symmetry codes: A: $-x+1, y+1/2, -z+3/2$; B: $-x+1, y-1/2, -z+3/2$; C: $-x+1/2, y+1/2, z$; D: $-x+1/2, y-1/2, z$; E: $x+1/2, y, -z+3/2$

Fig.3 Perspective of 2D supramolecular network parallel to ab plane in **1**

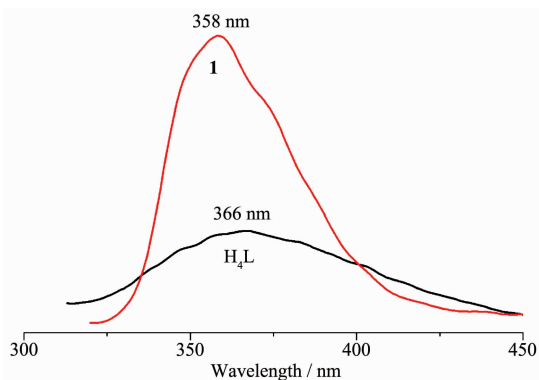
2.2 TGA analysis

To determine the thermal stability of polymers **1** and **2**, their thermal behaviors were investigated under nitrogen atmosphere by thermogravimetric analysis (TGA). As shown in Fig.4, TGA curve of compound **1** showed that there was a loss of five lattice water molecules between 30 and 130 °C (Obsd. 7.4%; Calcd. 7.6%); further heating above 312 °C led to a decomposition of the dehydrated sample. Compound **2** lost its five lattice water molecules in a range of 30~160 °C (Obsd. 8.2%; Calcd. 8.4%), followed by the decomposition at 294 °C.

Fig.4 TGA curves of compounds **1** and **2**

2.3 Luminescent properties

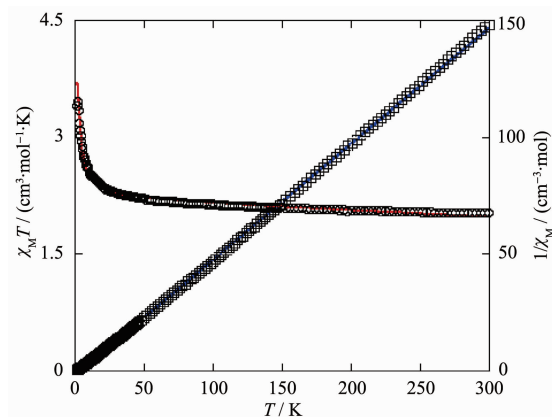
Solid-state emission spectra of H₄L and cadmium (II) compound **1** were measured at room temperature (Fig.5). The spectrum of H₄L revealed a weak emission with a maximum at 366 nm ($\lambda_{\text{ex}}=325$ nm). In comparison with H₄L, the coordination compound **1** exhibited more extensive emission with a maximum at 358 nm ($\lambda_{\text{ex}}=315$ nm). These emissions correspond to intraligand $\pi-\pi^*$ or $n-\pi^*$ transition of H₄L^[11,13,15]. Enhancement of the luminescence in **1** compared to H₄L 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^[13,15,23].

Fig.5 Solid-state emission spectra of H₄L and compound **1** at room temperature

2.4 Magnetic properties

Variable-temperature magnetic susceptibility measurements were performed on powder samples of **2** in the 2~300 K temperature range (Fig.6). For **2**, as shown in Fig.6, the $\chi_{\text{M}}T$ value at room temperature was $2.03 \text{ cm}^3 \cdot \text{mol}^{-1} \cdot \text{K}$, which is close to the value ($2.00 \text{ cm}^3 \cdot \text{mol}^{-1} \cdot \text{K}$) for two magnetically isolated high-spin

Ni(II) ion ($S=1$, $g=2.0$). The $\chi_{\text{M}}T$ values increased slowly on lowering the temperature until about 48 K, and then increased quickly to $3.42 \text{ cm}^3 \cdot \text{mol}^{-1} \cdot \text{K}$ at 2.0 K. Between 2 and 300 K, the magnetic susceptibilities can be fitted to the Curie-Weiss law with $C=1.99 \text{ cm}^3 \cdot \text{mol}^{-1} \cdot \text{K}$ and $\theta=5.76$ K. These results indicate a ferromagnetic interaction between the adjacent Ni(II) centers in compound **2**.



Red line represents the best fit to the equation in the text, and the blue line shows the Curie-Weiss fitting

Fig.6 Temperature dependence of $\chi_{\text{M}}T$ (○) and $1/\chi_{\text{M}}$ (□) for compound **2**

The magnetic data for **2** can be fitted with the expression for a dinuclear Ni(II) unit^[24]:

$$\chi_{\text{M, dimer}} = \frac{N_{\text{A}} g^2 \mu_{\text{B}}^2}{9kT} \frac{5 + \exp(-5.75x)}{5 + 3\exp(-5.75x) + \exp(-8.63x)}$$

where $x=J/(kT)$. The best fit for the experimentally observed data was obtained with a J value of 2.48 cm^{-1} , which indicates reasonable ferromagnetic interactions.

According to the chain structure of **2** (Fig.2), adjacent Ni(II) centers within the Ni₂ unit possess a single type of the magnetic exchange path, namely through one *syn-anti* carboxylate bridge, which explains a ferromagnetic exchange observed in this compound. The *syn-anti* carboxylate bridging moiety has been observed in some Ni(II) complexes with ferromagnetic interactions reported elsewhere^[25-28].

3 Conclusions

In summary, we have successfully synthesized and characterized two new cadmium/nickel coordination polymers by using one biphenyl-type tetracarboxylic

acid as bridging ligand under hydrothermal condition. Two polymers all feature 1D chain. Besides, the magnetic (for **2**) and luminescent (for **1**) 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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