

## 新型 N,O-双官能团席夫碱配体及其镉配合物的合成、晶体结构及荧光性质

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**摘要:** 合成了一个新颖的席夫碱 N,O-双官能团有机配体(4-(吡啶基-2-亚甲基氨基)苯甲酸, 4,2-pmabaH) (**1**), 并在水和乙醇体系中合成了它的镉配合物 $[\text{Cd}(4,2\text{-pmaba})(\text{H}_2\text{O})_3](4,2\text{-pmaba})\cdot\text{H}_2\text{O}$  (**2**)。利用元素分析及 X-射线单晶结构测定对其进行了表征。标题化合物 **1** 属于正交晶系,  $Pna2_1$  空间群。化合物 **2** 属于正交晶系,  $Pbcn$  空间群。在化合物 **2** 中, Cd(II) 的配位环境为七配位。其中有 2 种不同配位模式的配体, 其  $\text{M}_2\text{L}_2$  环状结构单元通过氢键作用连接成一个具有三维结构的超分子化合物。对化合物 **1, 2** 的荧光性质也进行了测定。

**关键词:** 晶体结构; 镉配合物; C-H $\cdots\pi$  相互作用; 氢键; 席夫碱

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## Synthesis, Crystal Structure and Photoluminescent Properties of A Novel N,O-bifunctional Schiff-Base Ligand and Its Cadmium(II) Complex

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**Abstract:** A novel schiff-base ligand based on N,O-bifunctional coordination group and its cadmium(II) complex, namely 4-(pyridin-2-ylmethylamino)benzoic acid (4,2-pmabaH) (**1**) and  $[\text{Cd}_2(4,2\text{-pmaba})_2(\text{H}_2\text{O})_6](4,2\text{-pmaba})_2\cdot 2\text{H}_2\text{O}$  (**2**), respectively, were obtained in ethanol/water system. Both the ligand and complex were characterized by elemental analysis and X-ray crystal structure analysis. The ligand **1** crystallizes in orthorhombic, space group  $Pna2_1$  with  $a=2.913\ 1(6)$  nm,  $b=1.054\ 8(2)$  nm,  $c=0.722\ 08(16)$  nm,  $V=2.218\ 7(9)$  nm<sup>3</sup>,  $Z=8$ ,  $D_c=1.367\ \text{g}\cdot\text{cm}^{-3}$ ,  $F(000)=960$ ,  $\mu=0.094\ \text{cm}^{-1}$ , the final  $R=0.077\ 6$ ,  $wR=0.185\ 8$ ; in which C-H $\cdots\pi$  interactions play important roles in structural stabilization. While the complex **2** crystallizes in orthorhombic, space group  $Pbcn$  with  $a=1.335\ 39(12)$  nm,  $b=0.944\ 24(8)$  nm,  $c=4.094\ 5(4)$  nm,  $V=5.162\ 9(8)$  nm<sup>3</sup>,  $Z=8$ ,  $D_c=1.644\ \text{g}\cdot\text{cm}^{-3}$ ,  $F(000)=2\ 608$ ,  $\mu=0.904\ \text{cm}^{-1}$ , the final  $R=0.064\ 9$ ,  $wR=0.132\ 8$ . In complex **2**, the coordination environment of Cd(II) is a distorted pentagonal-bipyramid with  $\text{N}_2\text{O}_5$  set. Two types of ligand with different conformations and coordination modes are found in complex **2**. The  $\text{M}_2\text{L}_2$  cyclic structural units in **2** are further connected by hydrogen bonds to give a three-dimensional structure. Their photoluminescent properties were also investigated. CCDC: 690113, **1**; 690114, **2**.

**Key words:** crystal structure; Cd(II) complex; C-H $\cdots\pi$  interaction; hydrogen bond; Schiff-base

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Coordination polymers consisting of inorganic and organic components have drawn great interests in recent years, mainly because of their structural diversity<sup>[1,2]</sup> as well as potential applications as functional materials<sup>[3-5]</sup>. Up to now, to the best of our knowledge, multidentate N- or O-donor ligands have been widely employed and many metal-organic frameworks (MOFs) have been constructed by the reactions of N-containing or O-containing ligands with corresponding metal salts, such as *N,N',N''*-tris (pyrid-3-ylmethyl-1,3,5-benzenetricarboxamide)<sup>[6]</sup>, 1,3,5-benzenetricarboxylic acid<sup>[7,8]</sup>.

Due to the different coordination abilities of N, O atoms to transition or rare-earth metals, the ligands containing both N and O as coordination atoms are now often used to synthesize hetero-metallic lanthanide-transition metal complexes<sup>[8]</sup>, which have attracted many attentions due to their special value in investigation on the nature of the magnetic exchange interactions between 3*d* and 4*f* metal ions in magnetic materials that contain rare earth metals<sup>[9]</sup>. On the other hand, schiff-base ligands have also attracted many attentions of chemists during the past decades because of the convenience in synthesis, good coordination ability and wide applications of Schiff-base complexes including catalysis and biomimetic chemistry<sup>[10]</sup>. Furthermore, cadmium(II) ions are extensively studied for its variable coordination symmetry and applications including photoluminescence and adsorption etc., and further studies are needed<sup>[11]</sup>. In this paper, we report a novel schiff-base ligand based on N,O-bifunctional coordination group and its Cd(II) complex, namely 4-(pyridin-2-ylmethylamino)benzoic acid (4,2-pmabaH) (**1**) and [Cd(4,2-pmaba)(H<sub>2</sub>O)<sub>3</sub>](4,2-pmaba)·H<sub>2</sub>O (**2**), respectively, as well as their photoluminescent properties.

## 1 Experimental

### 1.1 General

All commercially available chemicals are of reagent grade and used as received without further purification. C, H and N analyses were made on Elementar Vario EL-III elemental analyzer. Infrared (IR) spectra were recorded on Nicolet AVATAR 360 FTIR spectrophotometer by using KBr discs. <sup>1</sup>H NMR

spectra were recorded on the Bruker DRX-500 spectrometer. Thermogravimetric and differential thermal analyses were performed on a simultaneous NETZSCH STA 409 PC LXXX thermal analyzer. Powder samples were loaded into alumina pans and heated under N<sub>2</sub> at a heating rate of 20 °C·min<sup>-1</sup>. Luminescent spectra were recorded on HITACHI F4500 fluorescence spectrophotometer.

### 1.2 Synthesis of 4-(pyridin-2-ylmethylamino)benzoic acid (4,2-pmabaH) (**1**)

The ligand was synthesized according to the previously reported literatures<sup>[12]</sup>. A mixture of NaOH (50 mmol, 2.00 g) and *p*-aminobenzoic acid (50 mmol, 6.85 g) in 20 mL CH<sub>3</sub>OH/H<sub>2</sub>O (*V:V*=4:1) was stirred for 30 min. Then a solution of α-piccolinaldehyde (50 mmol, 5.36 g) in 15 mL CH<sub>3</sub>OH was added. The resulting yellow solution was stirred over night and then refluxed for 8 h. After been cooled to room temperature, excess NaBH<sub>4</sub> was added. The pH value of the solution was adjusted to 5.5 with HCl and the pale yellow precipitate was collected, washed with water and recrystallized in ethanol/H<sub>2</sub>O solution (Yield 80%). FTIR (KBr pellet, cm<sup>-1</sup>): 1 667s, 1 607s, 1 569m, 1 530s, 1 475s, 1 436m, 1 417m, 1 317s, 1 296s, 1 176s, 841m, 776m, 765m. <sup>1</sup>H NMR (500 MHz, sodium salt in D<sub>2</sub>O): δ 8.32(d, 1H); δ 7.65(t, 1H); δ 7.55(d, 2H); δ 7.31(t, 1H); δ 7.18(t, 1H); δ 6.58(d, 2H); δ 4.39(s, 2H).

### 1.3 Synthesis of [Cd(4,2-pmaba)(H<sub>2</sub>O)<sub>3</sub>](4,2-pmaba)·H<sub>2</sub>O (**2**)

Complex **2** was synthesized by layering method. The aqueous solution of the tetrabutylammonium salt of 4-(pyridin-2-ylmethylamino)benzoic acid was readily prepared by the reaction of tetrabutylammonium hydroxide with 4,2-pmabaH in water until pH=6.5. The title compound **2** was obtained by slow diffusion between two layers of an aqueous solution (5 mL) of the tetrabutylammonium salt of 4,2-pmabaH (11.4 mg, 0.05 mmol) and Cd(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O (15.4 mg, 0.05 mmol) in ethanol (5 mL) at room temperature. About 2 week later, colorless flake-like crystals suitable for X-ray analysis were obtained in 40% yield. Anal. Calcd. for compound **2**, C<sub>26</sub>H<sub>30</sub>CdN<sub>4</sub>O<sub>8</sub> (%): C, 48.87; H, 4.74; N, 8.77. Found(%): C, 49.03; H, 4.65; N, 8.84.

### 1.4 Structure determination

Suitable crystals of **1** and **2** with dimensions of 0.40 mm×0.20 mm×0.10 mm and 0.42 mm×0.20 mm×0.02 mm, respectively, were selected for data collection at 293 K, using a Bruker Smart Apex II CCD diffractometer equipped with a Mo  $K\alpha$  radiation ( $\lambda=0.071\ 073$  nm). The structures were solved by direct methods using SHELX-97 and refined by full-matrix least-squares methods anisotropically for non-hydrogen

atoms<sup>[13]</sup>. The hydrogen atoms except for those of water molecules were generated geometrically. Calculations were performed on a personal computer with the SHELXTL program package<sup>[14]</sup>. The details of the crystal parameters, data collection and refinement for the compounds are summarized in Table 1, and selected bond lengths and angles with their estimated standard deviations of **2** are listed in Table 2.

CCDC: 690113, **1**; 690114, **2**.

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

Compound	<b>1</b>	<b>2</b>
Empirical formula	C <sub>13</sub> H <sub>12</sub> N <sub>2</sub> O <sub>2</sub>	C <sub>26</sub> H <sub>30</sub> CdN <sub>4</sub> O <sub>8</sub>
Formula weight	228.24	638.94
Temperature / K	293	293
Crystal system	Orthorhombic	Orthorhombic
Space group	<i>Pna</i> 2 <sub>1</sub>	<i>Pbcn</i>
<i>a</i> / nm	2.913 1(6)	1.335 39(12)
<i>b</i> / nm	1.054 8(2)	0.944 24(8)
<i>c</i> / nm	0.722 08(16)	4.094 5(4)
<i>V</i> / nm <sup>3</sup>	2.218 7(9)	5.162 9(8)
<i>Z</i>	8	8
<i>D<sub>c</sub></i> / (g·cm <sup>-3</sup> )	1.367	1.644
Absorption coefficient / cm <sup>-1</sup>	0.094	0.904
<i>F</i> (000)	960	2 608
$\theta$ range / (°)	2.05~24.99	1.82~27.50
Reflections collected	10 751	28 780
Independent reflections ( <i>R<sub>int</sub></i> )	2 130 (0.086 4)	5 916 (0.168 3)
Observed reflections	2 010	3 928
Goodness of-fit on <i>F</i> <sup>2</sup>	1.210	1.010
<i>R</i> [ <i>I</i> >2 $\sigma$ ( <i>I</i> )]	0.077 6	0.064 9
<i>wR</i> [ <i>I</i> >2 $\sigma$ ( <i>I</i> )]	0.185 8 <sup>a</sup>	0.132 8 <sup>b</sup>

<sup>a</sup>  $w=1/[\sigma^2(F_o^2)+(0.085\ 1P)^2+1.066\ 3P]$  where  $P=(F_o^2+2F_c^2)/3$ ;

<sup>b</sup>  $w=1/[\sigma^2(F_o^2)+(0.054\ 6P)^2]$  where  $P=(F_o^2+2F_c^2)/3$ .

Table 2 Selected bond lengths (nm) and bond angles (°) for complex **2**

Cd1-N1	0.275 5(4)	Cd1-N2	0.228 9(4)	Cd1-O1	0.229 7(3)
Cd1-O2	0.244 2(4)	Cd1-O3	0.232 8(4)	Cd1-O4	0.230 3(5)
Cd1-O5	0.234 7(4)				
N1-Cd1-N2	67.19(13)	N1-Cd1-O1	95.69(12)	N1-Cd1-O2	75.64(12)
N1-Cd1-O3	153.35(14)	N1-Cd1-O4	79.09(15)	N1-Cd1-O5	125.53(14)
N2-Cd1-O1	162.79(13)	N2-Cd1-O2	115.93(13)	N2-Cd1-O3	104.14(17)
N2-Cd1-O4	90.58(18)	N2-Cd1-O5	81.96(15)	O1-Cd1-O2	54.95(12)
O1-Cd1-O3	92.00(16)	O1-Cd1-O4	87.59(17)	O1-Cd1-O5	108.37(15)
O2-Cd1-O3	128.57(15)	O2-Cd1-O4	131.32(16)	O2-Cd1-O5	79.70(15)
O3-Cd1-O4	75.77(16)	O3-Cd1-O5	75.26(16)	O4-Cd1-O5	147.24(17)

## 2 Results and discussion

### 2.1 Structure description

The X-ray crystallographic analysis reveals that the **1** crystallizes in orthorhombic system, space group  $Pna2_1$ . The two ligands in the asymmetric unit of **1** (**A** and **B**), which both adopt “linear” shape, occurs different torsions (Fig.1). The dihedral angle between two aromatic planes of ligand **A** (e.g. planes containing C1, C2, C3, C4 C5, C6 and C9, C10, C11, C12, C13, N2, respectively) is  $4.3^\circ$ , while the dihedral angle between two aromatic planes of ligand **B** (e.g. planes containing C14#1, C15#1, C16#1, C17#1, C18#1, C19#1 and C22#1, C23#1, C24#1, C25#1, C26#1,

N4#1, respectively) is  $10.1^\circ$ . In **1**, ligands of different types are linked by  $O-H\cdots O$  hydrogen bond to give a ligand pair with  $O\cdots O$  distances and  $O-H\cdots O$  angles of 0.260 6(5), 0.260 7(5) nm, and  $171(12)^\circ$ ,  $163(13)^\circ$ , respectively (Fig.1, Table 3). It is noteworthy that in **1**, the distances between H3, H6, H10, H13, H16, H18, H26 and the corresponding centroids of adjacent aromatic rings vary from 0.277 83(5) to 0.311 40(6) nm, with the corresponding angles of  $C-H\cdots$ Centroid in the range of  $127.3(4)^\circ$  to  $138.3(3)^\circ$  (Table 4), which indicate the  $C-H\cdots\pi$  interactions between each two adjacent distinct ligands<sup>[15]</sup>. Thus, the ligand pairs are further stacked to give a three-dimensional structure and stabled (Fig.2).

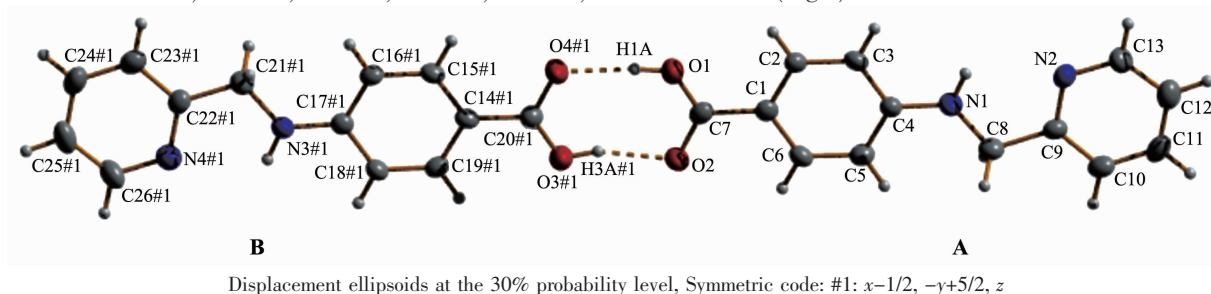


Fig.1 Hydrogen bonded ligand pair in **1**

Table 3 Hydrogen bonding data for **1** and **2**

D-H $\cdots$ A	Distance of D $\cdots$ A / nm	Angle of D-H-A / ( $^\circ$ )
<b>1</b>		
O1-H1A $\cdots$ O4#1	0.260 6(5)	171(12)
O3-H3A $\cdots$ O2#2	0.260 7(5)	163(13)
<b>2</b>		
O1W-H1WB $\cdots$ N4#1	0.283 2(7)	164(7)
O1W-H1WA $\cdots$ O7	0.273 0(7)	164(5)
O5-H5B $\cdots$ O1#2	0.298 0(5)	151(7)
O4-H4B $\cdots$ O6#1	0.268 8(6)	169(6)
O3-H3B $\cdots$ O1#2	0.291 1(6)	158(6)
O5-H5A $\cdots$ O2#3	0.276 8(6)	161(4)
O4-H4A $\cdots$ O7#4	0.265 7(6)	169(7)
O3-H3A $\cdots$ O6#1	0.272 8(6)	171(6)
N3-H3N $\cdots$ O1W#4	0.315 7(7)	142.0
N1-H1N $\cdots$ O4	0.323 9(6)	157.5

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

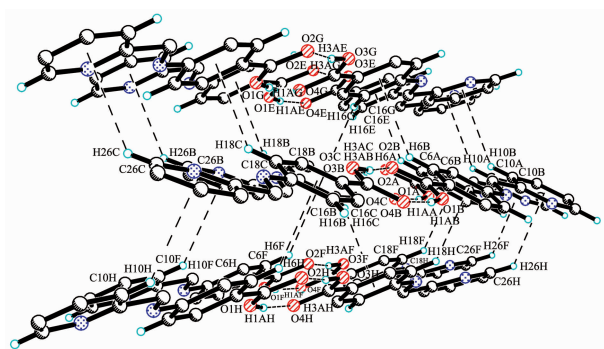
Table 4 Bond lengths (nm) and bond angles ( $^\circ$ ) of  $C-H\cdots\pi$  interaction in **1**

	$d(C-H)$ / nm	$d(H\cdots\text{centroid})$ / nm	$\angle C-H\cdots\text{centroid}$ / ( $^\circ$ )
C3#1-H3#1 $\cdots$ centroid	0.093	0.291 73(6)	136.0(4)
C6#2-H6#2 $\cdots$ centroid	0.093	0.291 74(6)	138.3(3)

Continued Table 4

C10#2-H10#2...centroid	0.093	0.311 40(6)	131.3(4)
C13#3-H13#3...centroid	0.093	0.284 52(5)	133.9(4)
C16#4-H16#4...centroid	0.093	0.292 61(6)	127.3(4)
C18#5-H18#5...centroid	0.093	0.277 83(5)	136.0(3)
C26#5-H26#5...centroid	0.093	0.301 12(6)	123.9(4)

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

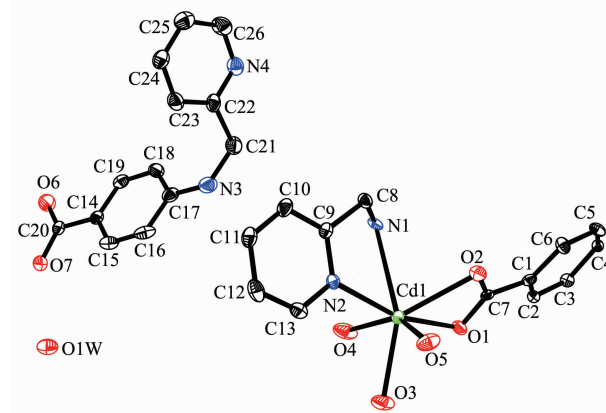


Hydrogen bonds and C-H... $\pi$  interactions were indicated by dashed lines, where the other H atoms were omitted for clarity

Fig.2 Three-dimensional packing diagram of ligand **1** viewing along the *b* axis with the corresponding atoms marked

Complex **2** was offered by treatment of  $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  with the tetrabutylammonium salt of 4,2-pmabaH ligand in ethanol/water system and crystallized in orthorhombic system, space group *Pbcn*. As shown in Fig.3, there are one Cd(II) ion, two ligands of different types, three coordination water molecules and one free water molecule in the asymmetric unit of **2**. Five of the seven sites of Cd(II) ion are coordinated by two carboxylic O atoms from same ligand and three water molecules with Cd-O bond lengths varying from 0.229 7(3) to 0.244 2(4) nm, and the remaining two sites are occupied by two N atoms of another ligand with Cd-N distances of 0.228 9(4) and 0.275 5(4) nm, respectively (Table 2). The N-Cd-N, N-Cd-O and O-Cd-O bond angles around Cd(II) ions are in the range of 54.95(12) $^\circ$  to 162.79(13) $^\circ$ . Thus, the coordination geometry of the Cd(II) center can be regarded as a distorted pentagonal-bipyramid with  $\text{N}_2\text{O}_3$  donor set. On the other hand, each 4,2-pmaba ligand coordinates to two Cd(II) ions and each Cd(II) ion links two ligands to give a 22-membered  $\text{M}_2\text{L}_2$  cycle with Cd...Cd distance of 0.945 15(8) nm (Fig.4a). The two benzene rings in the same cycle are

offset parallel stacked with centroid...centroid distance of 0.397 49(3) nm indicating  $\pi \cdots \pi$  interactions between the two aromatic rings<sup>[15]</sup>, which stabilizes the  $\text{M}_2\text{L}_2$  cycle unit. It is noteworthy that there are two kinds of 4,2-pmaba $^-$  ligands in **2** (Fig.3, Fig.4a). The first one is in a “linear” shape and coordinates to two Cd(II) ions with their carboxylate groups in  $\mu_1-\eta^1:\eta^1$  mode and two N atoms in chelating mode, where the dihedral angle of the two aromatic rings is 54.2 $^\circ$ . The second type of ligand does not participate in the coordination and serves as counter anion, where the two aromatic rings are nearly perpendicular to each other with the dihedral angle of 93.6 $^\circ$ . On the other hand, the second type of ligand adopts different conformation with “L” shape rather than the “linear” one, indicating that the flexible ligand like 4,2-pmaba can have varied conformations.



Thermal ellipsoids were drawn at 30% probability

Fig.3 Structure of asymmetric unit of complex **2** with atom numbering scheme where the hydrogen atoms were omitted for clarity

The  $\text{M}_2\text{L}_2$  cycles are further connected by O-H...O and N-H...O hydrogen bonds to give a three-dimensional (3D) structure (Table 3)<sup>[16-18]</sup>. First, the O5-O2 distance of 0.276 8(6) nm and O5-H5A-O2 angle of 161 (4) $^\circ$  indicate the formation of O5-H5A...O2 hydrogen



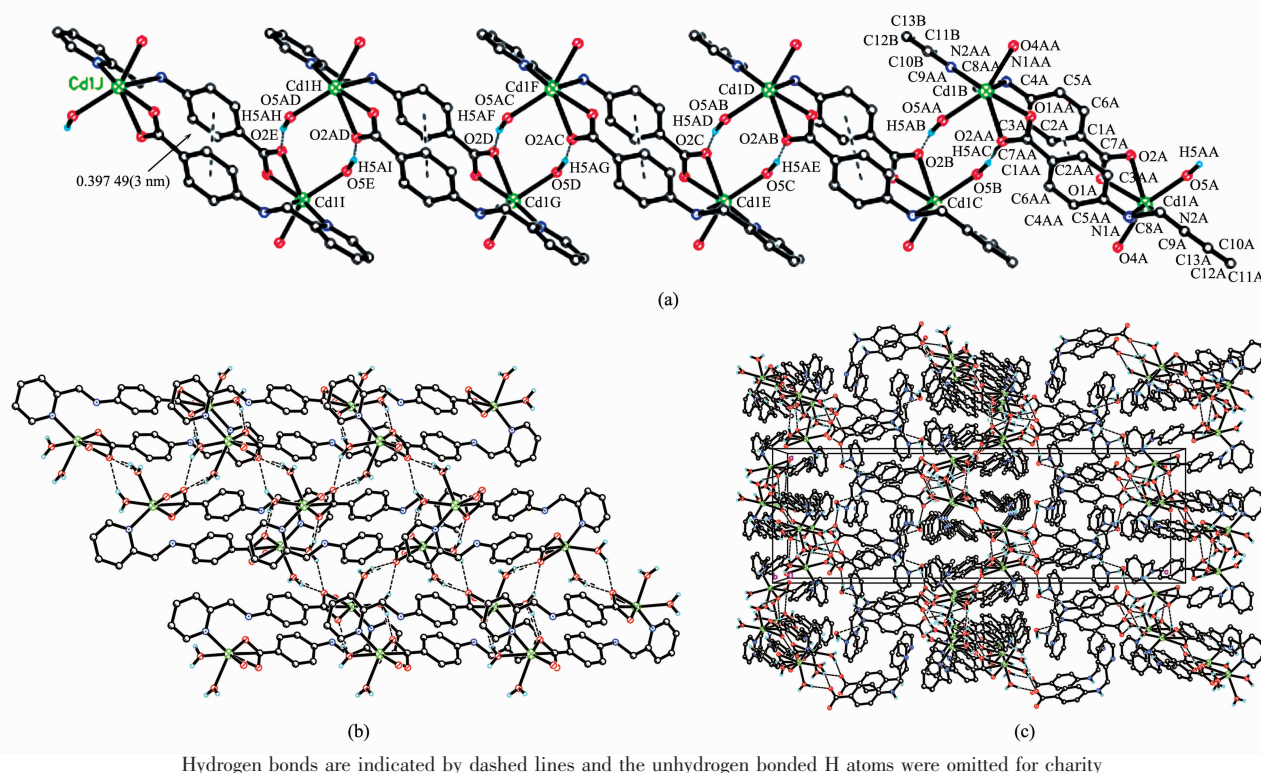


Fig.4 (a) 1D infinite chain structure linked by O5–H5A...O2 hydrogen bonds viewing along the *a* axis; (b) 2D network in **2** linked by O3–H3B...O1 and O5–H5B...O1 hydrogen bonds viewing along the *c* axis; (c) 3D diagram of **2** linked by O–H...O and N–H...O hydrogen bonds viewing along the *b* axis

bond between the neighboring cycles, which link the cyclic units to produce a one-dimensional (1D) infinite chain (Fig.4a). Second, the O3–O1, O5–O1 distances of 0.291 1(6) nm, 0.298 0(5) nm and O3–H3B–O1, O5–H5B–O1 angles of 158 (6)°, 151 (7)° indicate the formation of O3–H3B...O1 and O5–H5B...O1 hydrogen bonds between the adjacent 1D chains, which joint the 1D chains to produce a two-dimensional (2D) network in ABAB mode (Fig.4b). Furthermore, although some ligands and water molecules do not coordinate to Cd (II) ions, they play important roles in structure stabilization. They occupy the vacuum between each two 2D layers and form O–H...O and N–H...O hydrogen bonds with skeletons of **2** (Table 3), which links the 2D networks to generate 3D framework of **2** (Fig.4c). The results reveal that the hydrogen bonds play distinguished role in stabilizing the whole structure of complex **2**.

The Thermogravimetric analysis of complex **2** was carried out in the range 20~800 °C under nitrogen atmosphere. The TGA data show that the initial weight

loss of 11.7% (calcd. 11.3%) occurred from 40 to 180 °C corresponding to the loss of the water molecules; the second weight loss begins at 320 °C where the decomposition of the residue starts. A distinction between the loss of the coordinated and uncoordinated water molecules was not clearly observed from the TGA data. This nondistinctive behavior is also reported in the

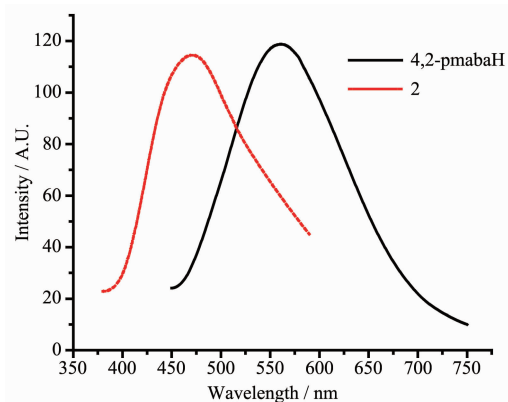


Fig.5 Photoluminescent emission spectra of the free 4,2-pmabaH ( $\lambda_{\text{em, max}}$  at 561 nm,  $\lambda_{\text{ex, max}}$  at 409 nm) and complex **2** ( $\lambda_{\text{em, max}}$  at 470 nm,  $\lambda_{\text{ex, max}}$  at 365 nm) in the solid state at room temperature (em=emission, ex=excitation, and max=maximum)

literature<sup>[19]</sup>. The solid state emission spectra of title complexes at room temperature are depicted in Fig.5. Complex **2** exhibits intense blue emissions maximum at 470 nm, when excited at 365 nm. Comparably, the free 4,2-pmabaH ligand shows emission at 561 nm with excitation at 409 nm. The luminescence of **2** is tentatively assigned to the intraligand fluorescent emission and the blue-shift in **2** is probably due to the coordination of 4,2-pmaba<sup>-</sup> to the Cd(II) centers<sup>[20]</sup>. The thermal stability as well as the photoluminescent emission makes **2** a potentially useful photoactive solid-state material. The investigations on the reactions of 4,2-pmabaH with other metal salts are still in progress.

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