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## Journal of Coordination Chemistry

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/gcoo20>

### Two transition metal coordination polymers constructed from 3-aminobenzoate and 4,4'-bipyridine

Zhili Fang<sup>a</sup> & Qixiang Nie<sup>b</sup>

<sup>a</sup> School of Basic Science, East China Jiaotong University, Nanchang 330013, P.R. China

<sup>b</sup> School of Civil Engineering and Architecture, East China Jiaotong University, Nanchang 330013, P.R. China

Published online: 26 Jul 2011.

To cite this article: Zhili Fang & Qixiang Nie (2011) Two transition metal coordination polymers constructed from 3-aminobenzoate and 4,4'-bipyridine, Journal of Coordination Chemistry, 64:14, 2573-2582, DOI: [10.1080/00958972.2011.602072](https://doi.org/10.1080/00958972.2011.602072)

To link to this article: <http://dx.doi.org/10.1080/00958972.2011.602072>

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## Two transition metal coordination polymers constructed from 3-aminobenzoate and 4,4'-bipyridine

ZHILI FANG<sup>†</sup> and QIXIANG NIE<sup>\*‡</sup>

<sup>†</sup>School of Basic Science, East China Jiaotong University,  
Nanchang 330013, P.R. China

<sup>‡</sup>School of Civil Engineering and Architecture,  
East China Jiaotong University, Nanchang 330013, P.R. China

(Received 19 April 2011; in final form 16 June 2011)

Two new transition metal coordination polymers,  $[\text{Zn}(\text{abz})(\text{bipy})](\text{ClO}_4)$  (**1**) and  $[\text{Mn}(\text{bipy})_5(\text{H}_2\text{O})_6](\text{ClO}_4)_2 \cdot 2(\text{abz}) \cdot 2(\text{H}_2\text{O})$  (**2**) (bipy = 4,4'-bipyridine, abz = 3-aminobenzoate), were prepared and characterized by elemental analysis, FTIR spectroscopy, and single-crystal X-ray diffraction. Complex **1** is a 2-D-layered network with a (4,4) net built up by bipy and 3-aminobenzoate ligands connecting two zinc nodes. Complex **2** is a 3-D supramolecular grid network constructed from close stacking bipy ligands of H-type cation units with large cavities hosting 3-aminobenzoate, perchlorate ions, and water. Solid state photoluminescence for **1** was also investigated.

*Keywords:* Crystal structure; 3-Aminobenzoate; 4,4'-Bipyridine; Photoluminescence

### 1. Introduction

Interest in polymeric coordination networks is rapidly expanding for their intriguing architectures [1–6], potential applications in catalysis, gas storage, and molecular recognition, and their magnetic, electrical, and nonlinear optical properties [7–20]. A number of such materials with predictable structures have been prepared through self-assembly of organic ligand spacer and metal node. Noncovalent intermolecular interactions, i.e., hydrogen-bonding and  $\pi$ - $\pi$  stacking, are an efficient approach to construct molecular assemblies with well-defined structures in the micro- and nanoscopic regimes.

Ligands containing carboxylate or bipyridine functionalities are useful in self-assembly because they give predictable patterns of hydrogen bonding that add extra dimensionality to supramolecular structures [21–23]. Self assembly of these ligands with various transition metal salts (such as those of Zn, Cu, Cd, and Mn) results in 1-D chain, zigzag, helical, ladder, 2-D square grid, rectangular grid, herringbone, brick wall, bilayer, 3-D cubical, diamondoid, etc. [24, 25]. Recently, aminobenzoate and 4,4'-bipyridine have been chosen to construct transition metal coordination compounds

\*Corresponding author. Email: zhilifang@ecjtu.jx

[7, 26, 27]; however, no examples of metal coordination polymers enclathrating mixtures of 3-aminobenzoate, and 4,4'-bipy ligands have been reported.

In our continuing efforts for the preparation of new coordination frameworks [28], herein we prepare two new polymeric frameworks,  $[\text{Zn}(\text{abz})(\text{bipy})](\text{ClO}_4)$  (**1**) and  $[\text{Mn}(\text{bipy})_5(\text{H}_2\text{O})_6](\text{ClO}_4)_2 \cdot 2(\text{abz}) \cdot 2(\text{H}_2\text{O})$  (**2**) (bipy = 4,4'-bipyridine, abz = 3-aminobenzoate), and characterize them by elemental analysis, IR spectroscopy, and single-crystal X-ray diffraction.

## 2. Experimental

### 2.1. Materials and instruments

All materials and reagents were obtained commercially and used without purification. Elemental (C, H, and N) analyses were performed on a Perkin–Elmer 2400 element analyzer. Infrared (IR) samples were prepared as KBr pellets and spectra were obtained from 4000 to  $400\text{ cm}^{-1}$  using a Nicolet Avatar 360 FT-IR spectrophotometer. Thermogravimetric analyses (TGA) were carried out on a Perkin–Elmer TGA 7 thermogravimetric analyzer at  $10^\circ\text{C min}^{-1}$  from  $35^\circ\text{C}$  to  $750^\circ\text{C}$  under dry air. Fluorescence spectra were recorded with an Edinburgh FLS920 Spectrophotometer analyzer.

### 2.2. Synthesis of **1**

A mixture of  $\text{Zn}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$  (0.372 g, 1 mmol), 4,4'-bipy (0.156 g, 1 mmol), 3-aminobenzoic acid (0.137 g, 1 mmol),  $\text{H}_2\text{O}$  (10 mL), and ethanol (5 mL) was sealed in a 20 mL Teflon-lined reactor, which was heated in an oven to  $150^\circ\text{C}$  for 48 h then cooled to room temperature at  $5^\circ\text{C h}^{-1}$ . Colorless block single crystals of **1** were obtained (yield: 82% based on Zn). Elemental Anal Calcd (%) for **1**,  $\text{C}_{17}\text{H}_{14}\text{ZnClN}_3\text{O}_6$ : C, 44.66; H, 3.09; N, 9.19. Found (%): C, 44.50; H, 2.98; N, 9.25. IR data (KBr,  $\text{cm}^{-1}$ ): 3454, 3374, 1632, 1429, 1367, 1200, 1165, 1095, 1004, 812, 701.

### 2.3. Synthesis of **2**

A mixture of  $\text{Mn}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$  (0.331 g, 1 mmol), 4,4'-bipy (0.390 g, 2.5 mmol), and 75% ethanol (20 mL) was stirred for 30 min at  $80^\circ\text{C}$ , then added to 3-aminobenzoic acid (0.137 g, 1 mmol), and adjusted to  $\text{pH}=7.0$  with an aqueous solution of sodium hydroxide ( $0.1\text{ mol L}^{-1}$ ). The mixture was continuously stirred for another 30 min and filtered. Colorless single crystals were obtained at room temperature by slow evaporation of the filtrate after several days (yield: 69% based on Mn). Elemental Anal Calcd (%) for **2**,  $\text{C}_{64}\text{H}_{68}\text{Cl}_2\text{Mn}_2\text{N}_{12}\text{O}_{20}$ : C, 51.04; H, 4.55; N, 11.16. Found (%): C, 51.10; H, 4.48; N, 11.02. IR data (KBr,  $\text{cm}^{-1}$ ): 3443, 3364, 1625, 1530, 1485, 1410, 1376, 1215, 1165, 1089, 1045, 1006, 801, 705.

Table 1. Crystal data details for **1** and **2**.

Complexes	<b>1</b>	<b>2</b>
Empirical formula	C <sub>17</sub> H <sub>14</sub> ClZnN <sub>3</sub> O <sub>6</sub>	C <sub>64</sub> H <sub>68</sub> Cl <sub>2</sub> Mn <sub>2</sub> N <sub>12</sub> O <sub>20</sub>
Formula weight	457.13	1506.08
Temperature (K)	296(2)	296(2)
Crystal system	Triclinic	Monoclinic
Space group	<i>P</i> $\bar{1}$	<i>P</i> 2 <sub>1</sub> / <i>n</i>
Unit cell dimensions (Å, °)		
<i>a</i>	9.0060(2)	7.4842(2)
<i>b</i>	10.5937(3)	19.0831(5)
<i>c</i>	11.4443(3)	23.5635(6)
$\alpha$	99.182(2)	90
$\beta$	109.182(1)	91.341(2)
$\gamma$	112.350(1)	90
Volume (Å <sup>3</sup> ), <i>Z</i>	901.77(4), 2	3364.46(15), 2
Calculated density (g cm <sup>-3</sup> )	1.684	1.487
Absorption coefficient (mm <sup>-1</sup> )	1.552	0.539
<i>F</i> (000)	464	1560
Goodness-of-fit on <i>F</i> <sup>2</sup>	1.010	1.019
<i>R</i> <sub>1</sub> [ <i>I</i> > 2σ( <i>I</i> )] <sup>a</sup>	0.0378	0.0517
<i>wR</i> <sub>2</sub> (all data) <sup>b</sup>	0.0963	0.1482

$$^a R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|.$$

$$^b wR_2 = [\sum_w (F_o^2 - F_c^2)^2 / \sum_w (F_o^2)^2]^{1/2}.$$

## 2.4. X-ray crystallography

Single crystal X-ray diffraction data collections of **1** and **2** were performed on a Bruker Apex II CCD diffractometer operating at 50 kV and 30 mA using Mo-K $\alpha$  radiation ( $\lambda = 0.71073$  Å). Data collection and reduction were performed using APEX II software. Multi-scan absorption corrections were applied for all the data sets using APEX II [29]. The structures were solved by direct methods and refined by full-matrix least squares on *F*<sup>2</sup> using the SHELXTL program package [29]. All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogens attached to carbon were placed in geometrically idealized positions and refined using a riding model. Hydrogens on water were located from difference Fourier maps and refined using a riding model. The 3-aminobenzoate in **2** showed significantly elongated thermal ellipsoids, indicating disorder over two positions. It was refined as being disordered over two sites in an occupancy ratio of 0.660(6):0.340(6). Crystal data and refinement parameters are presented in table 1.

## 3. Results and discussion

### 3.1. Synthesis and characterization

The synthesis of **1** and **2** is described in section 2. In FT-IR spectra, absorptions at  $\sim 1632$ – $1367$  cm<sup>-1</sup> for **1** and **2** are associated with asymmetric (COO) and symmetric (COO) stretching. Broad bands at  $3454$  and  $3443$  cm<sup>-1</sup> for **1** and **2**, respectively, may be assigned to  $\nu$ (O–H) of water. Features at  $1165$ – $1095$  cm<sup>-1</sup> and  $1165$ – $1089$  cm<sup>-1</sup> for **1** and **2**, respectively, are consistent with free ClO<sub>4</sub><sup>-</sup>.

### 3.2. Crystal structures

**3.2.1. Synthesis.** The hydrothermal method is a powerful technique in preparing metal-organic framework materials. Complex **1** could only be isolated under hydrothermal conditions, where 3-aminobenzoate is a bridging ligand; under conventional solution reactions, 3-aminobenzoate is a counteranion (see the structure of **2** below).

**3.2.2. The structure of 1.** X-ray structure analysis reveals that **1** is a 2-D layered network constructed from bipy and 3-aminobenzoate connecting two zinc centers, crystallizing in the triclinic space group  $P\bar{1}$ . An ORTEP view of the asymmetric unit of **1** is shown in figure 1(a). **1** contains one zinc, one bipy, one abz, and one lattice perchlorate. Each Zn(II) is in a distorted square-pyramidal coordination geometry formed by two oxygens from two abz ligands, one nitrogen from abz, and two nitrogens from two different bipy. The Zn–O and Zn–N bond distances and their bond angles (table 2), all within the range observed for other Zn(II) complexes with oxygen or nitrogen donors [7], range from 1.989(2) to 2.202(2) Å and 87.00(9) to 176.55(10)°, respectively. In **1**, 4,4'-bipy and abz both coordinate to zinc in a bridging coordination mode. The carboxylate and amino groups of abz connect to two zincs, resulting in an infinite Zn(abz) chain along the *a*-axis; 4,4'-bipy's bridge neighboring Zn(abz) chains in the *c*-axis direction, finally giving a layered network in the *ac*-plane of the unit cell (figure 1b). The network is a (4,4) net if 4,4'-bipy and abz are regarded as links and the two zinc ions as nodes (figure 1c). These layers connect through N–H...O (N...O = 2.945(2)) hydrogen bonding interactions (table 3) between amino groups of abz and perchlorate.

**3.2.3. The structure of 2.** X-ray structure analysis reveals that **2** is a 3D supramolecular grid network constructed from close stacking of bipy ligands of H-type cations with large cavities hosting 3-aminobenzoate, perchlorate, and water in monoclinic space group  $P2_1/n$ . The asymmetric unit of **2** consists of  $[\text{Mn}(\text{bipy})_{2.5}(\text{H}_2\text{O})_3]^{2+}$ , one abz ion, one  $\text{ClO}_4^-$ , and one water molecule (figure 2a). The free abz anion is disordered over two positions in an occupancy ratio of 0.660(6):0.340(6). Each Mn(II) is six-coordinate by three nitrogens from three different bipy ligands and three water molecules in a distorted octahedral coordination environment. The Mn–O and Mn–N bond distances and their bond angles (table 2) fall in the normal range [30]. Only one bipy bridges between Mn ions while the other two function only as monodentate terminal ligands. As a result, an H-type host unit is constructed (figure 2b). The H-type host units are infinitely extended *via* face to face  $\pi \cdots \pi$  interactions among pyridine rings of bipy to form a grid network, in which four H-type units form a rectangular grid with a large cavity of *ca*  $9.5 \times 23.6 \text{ \AA}^2$  (figure 2b). The interplanar distances between bipy ligands range from 3.55 to 3.74 Å, indicating normal  $\pi \cdots \pi$  stacking interactions. These cavities encapsulate abz, perchlorate, and water *via* strong hydrogen bonding interactions (table 3). Aside from the  $\pi \cdots \pi$  stacking interactions, the terminal bipy ligands are H-bonded to the  $\text{Mn}(\text{H}_2\text{O})_3$  units (O...N = 2.751(3)–3.088(4) Å), further supporting the grid network. The free water molecules and carboxylate oxygens of abz anions surround  $\text{Mn}(\text{H}_2\text{O})_3$  units through strong O–H...O (O...O = 2.650(4)–2.878(7) Å) hydrogen bonds. The free perchlorates are stabilized by N–H...O (N...O = 3.062(7)–3.231(6) Å) hydrogen bonds.

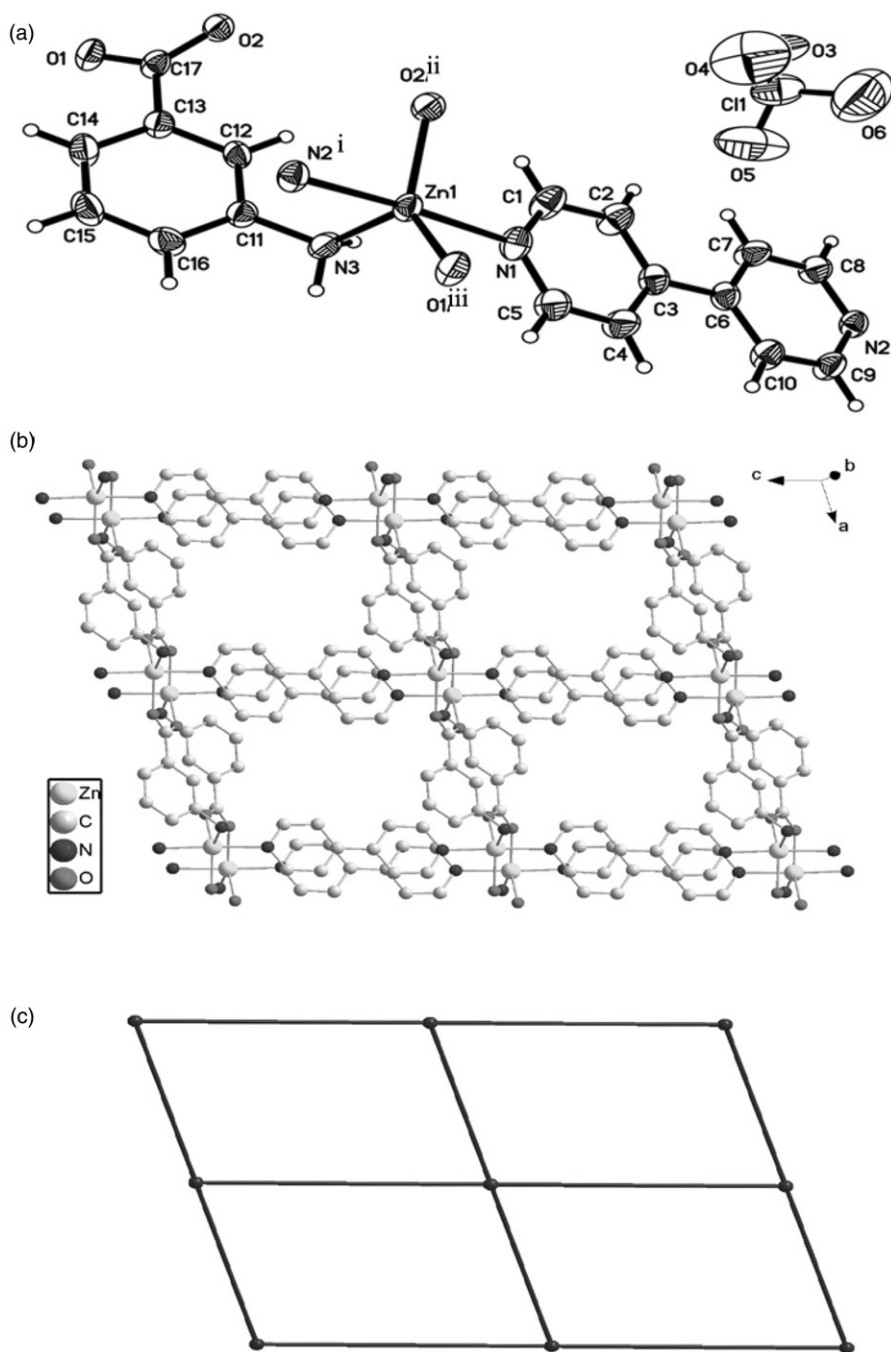


Figure 1. (a) ORTEP view of atom numbering diagram for **1** showing 50% probability displacement ellipsoids. (b) View of a 2-D-layered network of **1**. (c) A (4,4) net. Symmetry codes: (i)  $x, y, -1+z$ ; (ii)  $2-x, 2-y, 2-z$ ; (iii)  $-1+x, y, z$ .

Table 2. Selected bond lengths (Å) and angles (°) for **1** and **2**.

<b>Compound 1</b>			
Zn1–O2#1	1.989(2)	Zn1–O1#2	1.9919(19)
Zn1–N3	2.122(2)	Zn1–N2#3	2.148(2)
Zn1–N1	2.202(2)	O2#1–Zn1–O1#2	130.91(9)
O2#1–Zn1–N3	106.42(9)	O1#2–Zn1–N3	122.65(9)
O2#1–Zn1–N2#3	89.56(9)	O1#2–Zn1–N2#3	87.77(9)
N3–Zn1–N2#3	91.99(9)	O2#1–Zn1–N1	93.89(9)
O1#2–Zn1–N1	90.00(9)	N3–Zn1–N1	87.00(9)
N2#3–Zn1–N1	176.55(10)		
<b>Compound 2</b>			
Mn1–O2W	2.128(2)	Mn1–O1W	2.209(2)
Mn1–O3W	2.215(2)	Mn1–N4	2.265(2)
Mn1–N4	2.303(2)	Mn1–N2	2.310(2)
O2w–Mn1–O1w	87.45(10)	O2w–Mn1–O3w	90.96(10)
O1w–Mn1–O3w	174.69(8)	O2w–Mn1–N1	177.1(1)
O1w–Mn1–N1	89.79(9)	O3w–Mn1–N1	91.71(9)
O2w–Mn1–N4	94.87(10)	O1w–Mn1–N4	93.17(8)
O3w–Mn1–N4	92.02(9)		

Symmetry transformations used to generate equivalent atoms in compound **1** are #1:  $2-x, 2-y, -z$ ; #2:  $-1+x, y, z$ ; #3:  $x, y, -1+z$ .

Table 3. Hydrogen bond lengths (Å) and angles (°) for **1** and **2**. Symmetry transformations used to generate equivalent atoms are given as footnotes.

<b>Compound 1<sup>a</sup></b>				
Donor–H...Acceptor	D–H	H...A	D...A	D–H...A
N3–H2A...O3#1	0.90	2.06	2.945(2)	169(1)
<b>Compound 2<sup>b</sup></b>				
O1w–H1w...N3#1	0.83	1.94	2.751(3)	167(3)
O1w–H2w...N5#2	0.82	2.27	3.088(4)	174(3)
O2w–H3w...O6#3	0.83	2.06	2.878(7)	171(4)
O2w–H4w...O4w	0.83	1.82	2.650(4)	172(4)
O3w–H5w...O6	0.83	2.08	2.839(7)	152(3)
N6–H6A...O5#4	0.86	2.29	3.062(7)	149(2)
N6–H6B...O1#5	0.86	2.53	3.231(6)	140(2)
O3w–H6w...N5#6	0.83	2.12	2.957(4)	178(3)
O4w–H7w...O5	0.85	1.99	2.681(6)	138(4)
O4w–H8w...O6#7	0.85	1.97	2.814(8)	176(5)

<sup>a</sup>#1:  $x, y, -1+z$ .

<sup>b</sup>#1:  $-1/2+x, 1/2-y, 1/2+z$ ; #2:  $-1/2+x, 1/2-y, -1/2+z$ ; #3:  $-1+x, y, z$ ; #4:  $1+x, y, z$ ; #5:  $3/2-x, -1/2+y, 1/2-z$ ; #6:  $1/2+x, 1/2-y, -1/2+z$ ; #7:  $1-x, -y, -z$ .

### 3.3. Thermal behaviors, PXRD patterns, and luminescent properties

Thermogravimetric analyses of **1** and **2** were done from 35°C to 800°C at a heating rate of 5°C min<sup>-1</sup> in dry air. The TGA trace of **1** indicates that the framework is stable to 350°C, while **2** first lost weight corresponding to two lattice water molecules and six coordinated water molecules (observed 9.58%, calculated 9.56%) from 60°C to 150°C. Upon further heating, the framework collapsed until the residue was MnO (figure 3). Simulated and experimental powder X-ray diffraction (PXRD) patterns of **1** and **2** are shown in figure S1. They are in fairly good agreement with the experimental patterns, confirming the phase purity of the products.

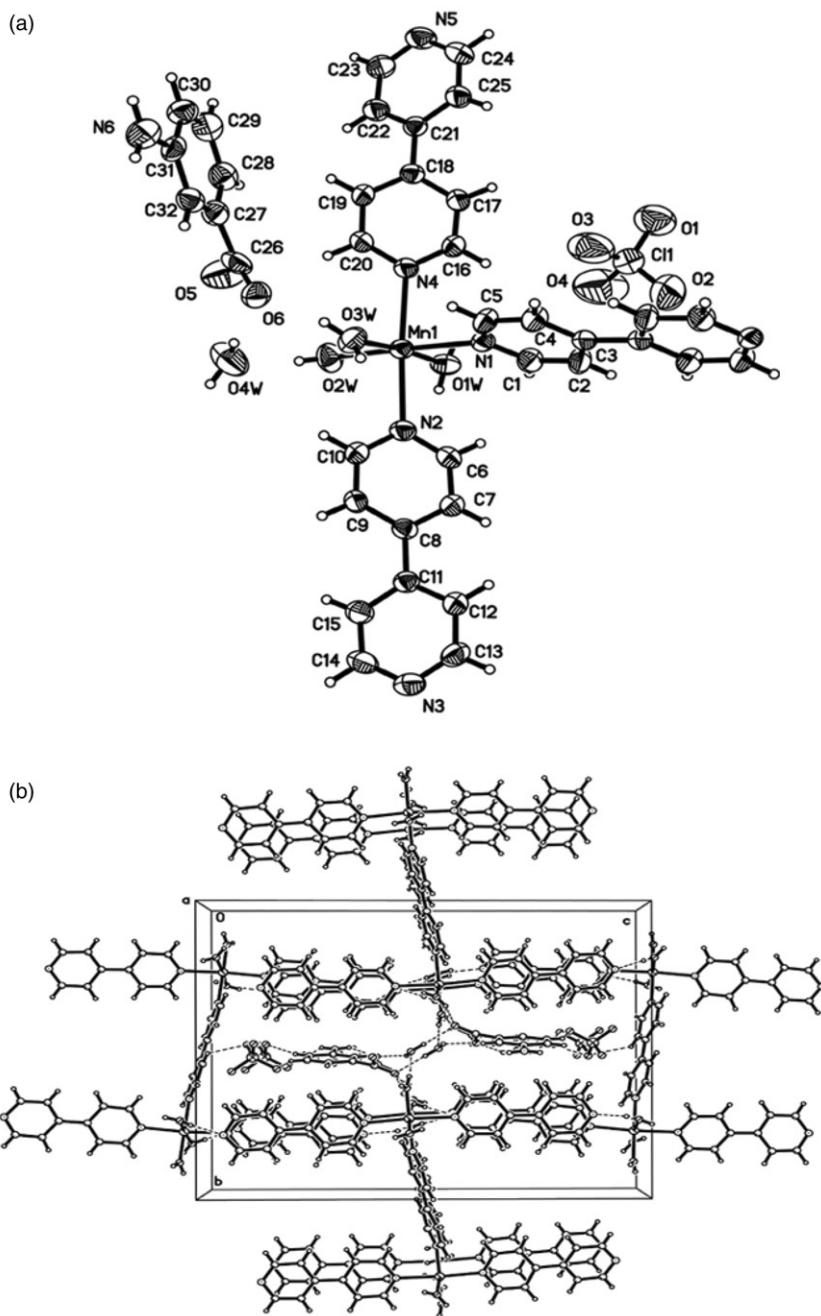


Figure 2. (a) ORTEP view of atom numbering diagram for **2** showing 50% probability displacement ellipsoids. (b) View of a 3-D supramolecular network of **2**. Hydrogen bonds are shown as dashed lines.



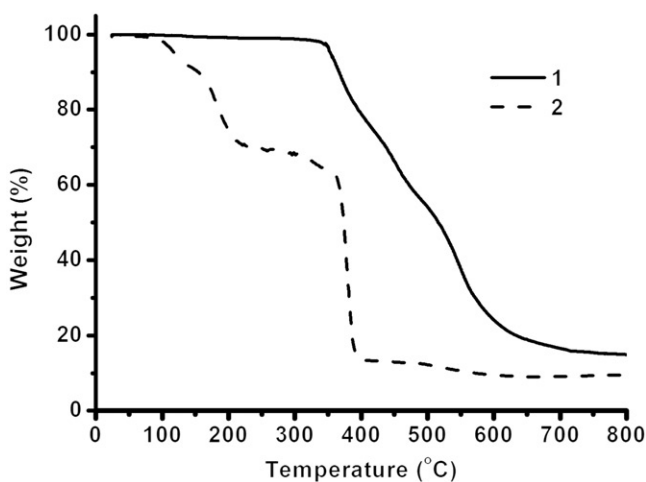


Figure 3. TGA traces of 1 and 2.

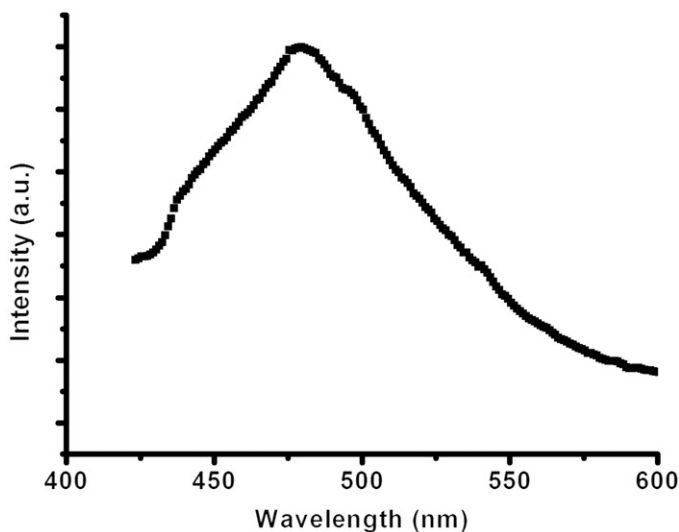


Figure 4. Solid-state emission spectrum of 1.

Owing to the excellent luminescence properties of Zn complexes, the luminescence spectrum of **1** was investigated in the solid state when excited by 320 nm at room temperature (figure 4). The 4,4'-bipy alone exhibits no photoluminescence, and the abz ligand shows a 350 nm emission band. Therefore, the emission at *ca* 478 nm may be tentatively ascribed to ligand-to-metal charge transfer (LMCT) [31] compared to the emission for free ligands. The red-shift emission and much more enhanced fluorescence intensity than  $[\text{Zn}_2(\text{bipy})_3(\text{H}_2\text{O})_8(\text{ClO}_4)_2(\text{paba})_2] \cdot 2(\text{bipy}) \cdot 4\text{H}_2\text{O}$  (bipy = 4,4'-bipyridine, paba = p-aminobenzoate) [7] is attributed to more rigidity of the 2-D-layered network (abz ligand as a bridging ligand), which effectively reduces the loss of energy.

#### 4. Conclusion

We have prepared two new transition metal coordination polymers derived from 3-aminobenzoate and 4,4'-bipyridine. Both compounds were characterized by elemental analysis, FTIR spectroscopy, and single-crystal X-ray diffraction. Complex **1** is a 2-D-layered network while **2** is a 3-D supramolecular grid network. Complex **1** exhibits strong luminescence and is a potential candidate as a photoactive material.

#### Supplementary material

Crystallographic data for the structure reported in this article have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 822215 & 822216. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44 1223 336 033, Email: deposit@ccdc.cam.ac.uk or www: <http://www.ccdc.cam.ac.uk>), or is also available from the author on request.

#### Acknowledgments

This work was supported by East China Jiaotong University.

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