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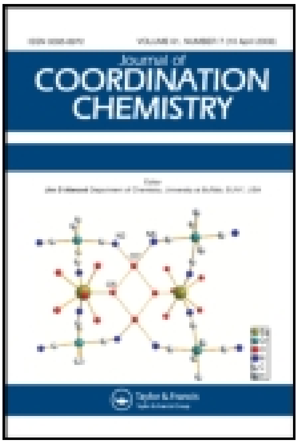

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1-D zigzag double-chain coordination polymers of transition metals derived from pyridine-2,3,5,6-tetracarboxylic acid

Ai-Hong Yang [a] , Hong-Ling Gao [a] , Su-Rong Fang [a] & Jian-Zhong Cui [a]

[a] Department of Chemistry , Tianjin University , Tianjin 300072, P.R. China

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1-D zigzag double-chain coordination polymers of transition metals derived from pyridine-2,3,5,6-tetracarboxylic acid

AI-HONG YANG, HONG-LING GAO, SU-RONG FANG and JIAN-ZHONG CUI*

Department of Chemistry, Tianjin University, Tianjin 300072, P.R. China

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Three new 1-D zigzag coordination polymers $\{[M_2(pdtc)(bpy)_2(H_2O)_3]\cdot 4H_2O\}_n$ [M = Zn (**1**), Ni (**2**), Co (**3**)] (H_4pdtc = pyridine-2,3,5,6-tetracarboxylic acid, bpy = 2,2′-bipyridine) have been synthesized by the reactions of H_4pdtc and corresponding metal salts in the presence of bpy at room temperature and characterized by elemental analyses, IR, TGA, fluorescence, and X-ray diffraction. The main structural feature of **1**–**3** is the presence of octahedrally coordinated M1(II) and M2(II) centers sequentially bridged by pdtc ligands; bpy acts as a blocking ligand to avoid further polymerization. These coordination modes lead to 1-D double-chain structures. The 2-D or 3-D supramolecular structures of **1**–**3** are built up by $\pi\cdots\pi$ and hydrogen-bond interactions. Two $(H_2O)_3$ clusters are observed in **1**–**3**. The zinc complex shows strong fluorescent emission at 325 nm.

Keywords: Pyridine-2,3,5,6-tetracarboxylic acid; 1-D double-chain; Fluorescence measurement; Water cluster

1. Introduction

Supramolecular networks based on bridging ligands containing N- or/and O-donors have intriguing network architectures [1]. Pyridine polycarboxylic acids are versatile ligands due to the fact that they have many coordination sites to construct complexes with various coordination modes and have potential applications as functional materials. Numerous coordination polymers containing pyridine di- [2–9] or tri- [10–13] carboxylates have been reported. Compared with di- or tri-pyridinecarboxylic acids, pyridine-2,3,5,6-tetracarboxylic acid (H_4pdtc) has more coordination groups, making it possible to construct high-dimensional complexes. H_4pdtc exhibits low symmetry, has diverse function groups, and can give various possibilities to form complexes with novel structures and coordination modes. Since H_4pdtc complexes might exhibit interesting structures, H_4pdtc becomes a good candidate for the construction of metal–organic frameworks.

Hydrogen-bonded water clusters and the morphology of water clusters are also fascinating. Large water clusters, novel water chains, and water layers were observed

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*Corresponding author. Email: cuijianzhong@tju.edu.cn

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and reported [14]. In this article, two $(H_2O)_3$ clusters are observed in three 1-D zigzag coordination polymers $\{[M_2(pdtc)(bpy)_2(H_2O)_3]\cdot 4H_2O\}_n$ [M = Zn (**1**), Ni (**2**), Co (**3**)], which are synthesized with H_4pdtc in the presence of 2,2′-bipyridine (bpy) as the second ligand. They are characterized by X-ray single-crystal diffraction, element analysis, IR, fluorescence, and thermal analysis. The zinc complex shows the strong fluorescent emission at 325 nm with excitation at 294 nm.

2. Experimental

2.1. *General*

Pyridine-2,3,5,6-tetracarboxylic acid was synthesized by the literature method [14(b)]. Other chemicals purchased were of reagent grade and used without purification. Elemental analyses for C, H, and N were performed on a Perkin-Elmer 240 CHN elemental analyzer. IR spectra were recorded from 400 to 4000 cm^{-1} with a Bruker TENSOR 27 spectrophotometer using a KBr pellet. TGA experiments were performed on a NETZSCH TG 209 instrument with a heating rate of 10°C min^{-1}. Fluorescence spectra were measured on a Cary Eclipse EL06063917 fluorescence spectrophotometer with a xenon arc lamp as the light source.

2.2. *Syntheses of the complexes*

A mixture of H_4pdtc (0.1 mmol, 0.0255 g), $Zn(NO_3)_2\cdot 6H_2O$ (0.1 mmol, 0.0297 g), 2,2-bipyridine (0.1 mmol, 0.0156 g), and H_2O (10 mL) was refluxed for 3 h. Colorless crystals of **1** were obtained after the filtrate was allowed to stand at room temperature for 2 weeks. Yield 63% based on Zn. Elemental analysis, found (%): C, 42.89; H, 3.85; N, 8.65; Calcd for $C_{29}H_{31}N_5O_{15}Zn_2$ (fw = 820.33) (%): C, 42.46; H, 3.78; N, 8.54. IR (KBr, cm^{-1}): ν = 3435 s, 1636 s, 1576 s, 1478 m, 1366 s, 1328 m, 1159 m, 767 m.

Complex **2** was prepared by similar method to **1** only using the $Ni(NO_3)_2\cdot 6H_2O$ (0.1 mmol, 0.0291 g) instead of $Zn(NO_3)_2\cdot 6H_2O$. Green crystals of **2** were obtained after the filtrate was allowed to stand at room temperature for 10 days. Yield 61% based on Ni. Elemental analysis, found (%): C, 43.25; H, 3.95; N, 8.55; Calcd for $C_{14.5}H_{15.5}N_{2.5}O_{7.5}Ni_2$ (fw = 403.50) (%): C, 43.16; H, 3.84; N, 8.67. IR (KBr, cm^{-1}): ν = 3348 s, 1612 s, 1575 s, 1431 m, 1356 s, 1325 m, 1156 m, 770 m.

Complex **3** was prepared with the same method as **1** using $Co(NO_3)_2\cdot 6H_2O$ (0.1 mmol, 0.0291 g) instead of $Zn(NO_3)_2\cdot 6H_2O$. Crystals of **3** were obtained after the filtrate was allowed to stand at room temperature for 2 weeks. Yield 64% based on Co. Elemental analysis, found (%): C, 43.36; H, 3.97; N, 8.76; Calcd for $C_{29}H_{31}N_5O_{15}Co_2$ (fw = 807.45) (%): C, 43.14; H, 3.84; N, 8.67. IR (KBr, cm^{-1}): ν = 3423 s, 1627 s, 1567 s, 1423 m, 1348 s, 1324 m, 1159 m, 771 m.

2.3. *X-ray crystallography*

Crystal diffraction data were collected on a computer-controlled Rigaku Saturn detector for **1** and **2**, and SCX min for **3**. All diffractometers were equipped with

Table 1. Crystal data and structure refinement information for **1**–**3**.

Complex	**1**	**2**	**3**
Empirical formula	$C_{29}H_{31}N_5O_{15}Zn_2$	$C_{29}H_{31}N_5O_{15}Ni_2$	$C_{29}H_{31}N_5O_{15}Co_2$
Formula weight	820.33	807.01	807.45
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1$	$P2_1$	$P2_1$
Unit cell dimensions (Å, °)			
a	10.938(2)	10.823(2)	11.009(2)
b	10.727(2)	10.750(2)	10.857(2)
c	13.658(3)	13.582(3)	13.715(3)
β	94.44(3)	94.64(3)	94.61(3)
Volume (Å^3), Z	1597.7(5), 2	1575.0(5), 2	1634.0(6), 2
Calculated density, D_c (Mg m^{-3})	1.705	1.702	1.641
Absorption coefficient (mm^{-1})	1.585	1.280	1.097
F (000)	840	832	828
Goodness-of-fit on F^2	1.038	1.034	0.969
Parameters	461	461	460
θ range (°)	1.50–27.87	1.50–25.02	3.38–25.00
Flack parameters	0.00(2)	0.00(8)	0.00(3)
Reflections collected/unique	12,348/6741 [R(int) = 0.0246]	9717/5205 [R(int) = 0.0504]	13,803/5683 [R(int) = 0.1834]
Reflections [$I > 2\sigma(I)$]	6281	4615	3240
Final R [$I > 2\sigma(I)$]	$R_1 = 0.0318$, $wR_2 = 0.0685$	$R_1 = 0.0518$, $wR_2 = 0.0952$	$R_1 = 0.0992$, $wR_2 = 0.1207$
R indices (all data)	$R_1 = 0.0347$, $wR_2 = 0.0706$	$R_1 = 0.0599$, $wR_2 = 0.1003$	$R_1 = 0.1782$, $wR_2 = 0.1389$
Largest difference peak and hole (e Å^{-3})	0.427 and −0.520	0.423 and −0.519	0.542 and −0.488

graphite-monochromated Mo-Kα radiation with a radiation wavelength of 0.71073 Å. For **1**–**3**, the ω-scan technique was used. Lorentz polarization and absorption corrections were applied. The structures were solved by direct methods and refined with a full-matrix least-squares technique based on F^2 using SHELXS-97 and SHELXL-97 programs [15, 16]. Anisotropic thermal parameters were assigned to all non-hydrogen atoms. The crystallographic data are listed in table 1.

3. Results and discussion

3.1. *Structure description*

The asymmetric unit of **1** comprises two crystallographically independent Zn's, one pdtc, two bpy, three coordinated water molecules, and four uncoordinated water molecules. Both Zn1 and Zn2 are six-coordinate with distorted octahedra (figure 1a). Equatorial coordination to Zn1 is provided by tridentate chelate ONO and one N3 of bpy. The two axial sites are occupied by N2 of bpy and a carboxylate oxygen from a neighboring pdtc. For Zn2, equatorial positions are coordinated by a bpy (N4, N5) and two coordinated water molecules (O9, O10). The axial sites are occupied by one carboxylate (O4) and one coordinated water molecule (O11). The average bond lengths of Zn1–O and Zn1–N are 2.162 and 2.103 Å; the average bond lengths of Zn2–O and

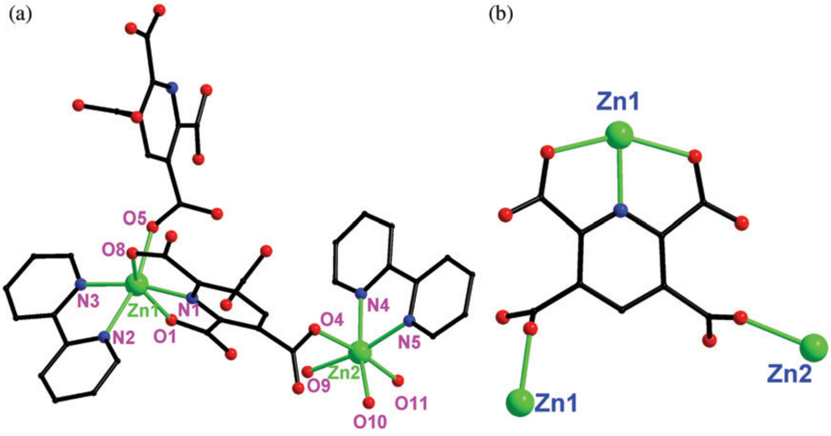

Figure 1. The coordination environments of (a) Zn and (b) pdtc. Hydrogens and uncoordinated water molecules are omitted.

Table 2. Selected bond lengths (Å) and angles (°) for **1**–**3**.

	1 (Zn)	**2** (Ni)	**3** (Co)		**1** (Zn)	**2** (Ni)	**3** (Co)
M(1)–N(1)	2.056(2)	1.980(5)	2.060(8)	M(2)–O(10)	2.061(2)	2.012(5)	2.047(7)
M(1)–N(3)	2.061(2)	2.028(5)	2.091(8)	M(2)–O(9)	2.072(2)	2.043(4)	2.074(7)
M(1)–O(5)^{i}	2.073(2)	2.068(4)	2.079(7)	M(2)–N(4)	2.126(3)	2.048(5)	2.134(9)
M(1)–O(8)	2.177(2)	2.133(4)	2.167(7)	M(2)–N(5)	2.144(2)	2.069(5)	2.141(9)
M(1)–N(2)	2.193(2)	2.101(5)	2.164(9)	M(2)–O(11)	2.1485(19)	2.112(4)	2.191(6)
M(1)–O(1)	2.2352(19)	2.142(4)	2.196(7)	M(2)–O(4)	2.1662(19)	2.111(4)	2.148(7)
N(1)–M(1)–N(3)	167.21(10)	172.3(2)	169.0(3)	O(10)–M(2)–O(9)	99.41(9)	93.0(2)	97.8(3)
N(1)–M(1)–O(5)^{i}	98.40(9)	96.35(16)	97.3(3)	O(10)–M(2)–N(4)	166.78(10)	173.3(2)	168.7(4)
N(3)–M(1)–O(5)^{i}	91.89(10)	90.00(19)	90.3(3)	O(9)–M(2)–N(4)	93.45(9)	93.75(18)	93.4(4)
N(1)–M(1)–O(8)	76.68(9)	77.81(17)	77.2(3)	O(10)–M(2)–N(5)	89.71(10)	93.4(2)	91.6(4)
N(3)–M(1)–O(8)	111.54(9)	106.96(17)	111.1(3)	O(9)–M(2)–N(5)	169.15(9)	172.3(2)	169.1(3)
O(5)^{i}–M(1)–O(8)	87.41(9)	87.27(16)	88.6(3)	N(4)–M(2)–N(5)	77.19(10)	79.9(2)	77.2(4)
N(1)–M(1)–N(2)	94.34(9)	95.32(19)	96.8(3)	O(10)–M(2)–O(11)	90.23(9)	88.74(18)	89.9(3)
N(3)–M(1)–N(2)	76.60(9)	78.94(19)	76.9(3)	O(9)–M(2)–O(11)	90.13(8)	91.20(15)	91.5(3)
O(5)^{i}–M(1)–N(2)	165.16(9)	166.44(18)	164.1(3)	N(4)–M(2)–O(11)	92.83(9)	91.36(17)	91.6(3)
O(8)–M(1)–N(2)	88.16(9)	88.46(17)	87.3(3)	N(5)–M(2)–O(11)	95.74(8)	93.22(16)	94.2(3)
N(1)–M(1)–O(1)	75.36(8)	78.30(17)	75.2(3)	O(10)–M(2)–O(4)	88.77(8)	90.91(18)	89.8(3)
N(3)–M(1)–O(1)	96.54(9)	97.11(17)	96.3(3)	O(9)–M(2)–O(4)	81.77(8)	83.16(15)	82.0(3)
O(5)^{i}–M(1)–O(1)	93.58(8)	92.46(16)	95.1(3)	N(4)–M(2)–O(4)	90.03(8)	89.65(17)	90.0(3)
O(8)–M(1)–O(1)	151.86(7)	155.93(15)	152.4(3)	N(5)–M(2)–O(4)	92.64(8)	92.45(17)	92.4(3)
N(2)–M(1)–O(1)	96.98(8)	96.65(16)	95.6(3)	O(11)–M(2)–O(4)	171.56(7)	174.33(15)	173.4(3)

Symmetry code: $^{i}-x+1, y+1/2, -z$.

Zn2–N are 2.112 and 2.135 Å. Selected bond lengths and angles of **1**–**3** are shown in table 2.

For pdtc, the carboxylates are completely deprotonated and coordinate with three metals (figure 1b). The head side of pdtc connects one Zn1 with tridentate chelate mode and the tail side bridges Zn1 and Zn2 monodentate. These coordination modes lead to the formation of infinite 1-D zigzag chains running along the *b*-axis (figure 2). In the zigzag chain, Zn1 is almost coplanar with the pyridine ring with a deviation of 0.0231 Å,

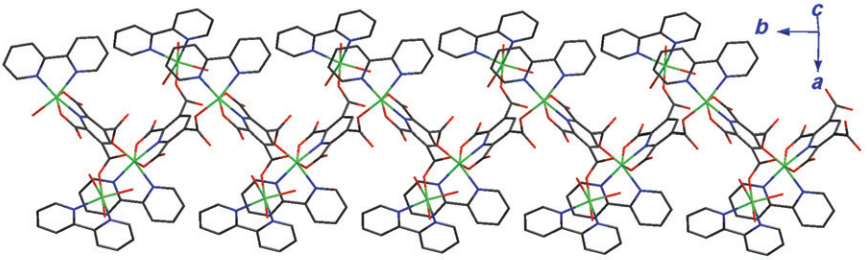

Figure 2. The 1-D zigzag chain of **1**.

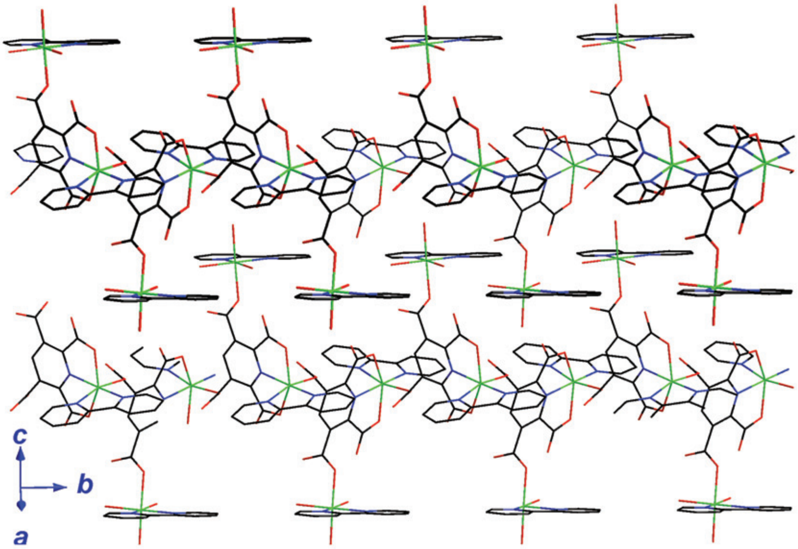

Figure 3. The chains occlude each other on the positions of bpy rings to form 2-D layer structure.

and two neighboring pyridine rings are approximately perpendicular with dihedral angle of 80.9°. The 1-D chain forms because bpy prevents continued growth, just like in two Bi^{3+} complexes, they formed mono or dinuclear complexes because bpy/phen acted as blocking ligand to avoid further polymerization [17].

For **1**–**3**, the planes of bpy of adjacent chains are almost parallel. Then the chains occlude each other on the positions of bpy rings to form 2-D layer structure (figure 3). The 2-D structure also depends on an extensive hydrogen-bonding network involving coordinated and uncoordinated water molecules. As connecters of the zigzag chains, two V-shaped $(H_2O)_3$ clusters form in the space between adjacent chains (figure 4). The coordinated water molecule O9 with two uncoordinated water molecules (O14 and O12) forms an $(H_2O)_3$ cluster with an average O ··· O distance of 2.692 Å, and the O9 ··· O14 ··· O12 angle of 117.31°. The other three water molecules (O11, O13, and O15) link to form the other $(H_2O)_3$ cluster with an average O ··· O distance of 2.887 Å, and the O11 ··· O13 ··· O15 angle of 125.90°. The average O ··· O distances of 2.692 and 2.887 Å are close to the distances in regular ice (2.74 Å) and liquid water

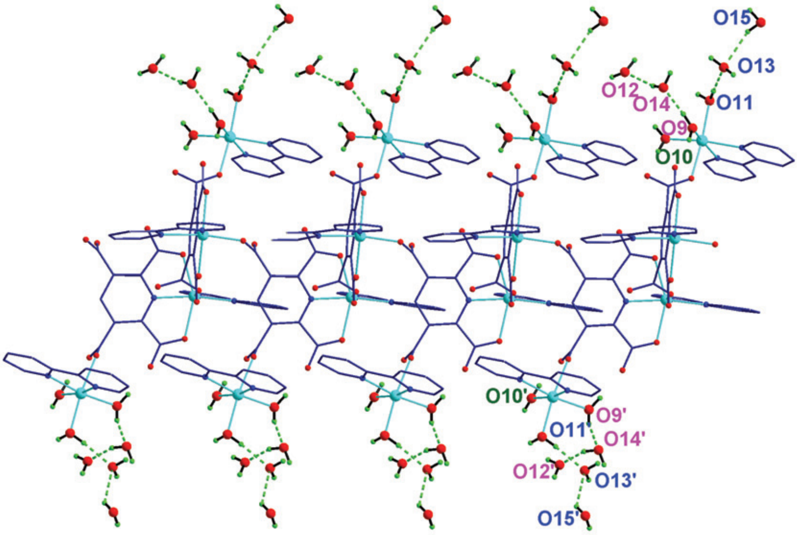

Figure 4. View showing the interactions between the two $(H_2O)_3$ clusters and the 1-D chain.

Table 3. Hydrogen-bond data (Å, °) in **1**; the hydrogen-bond data in water trimers are in bold.

D–H···A^{a}	*d*(D–H)	*d*(H···A)	*d*(D···A)	∠(DHA)
O9–H9B···O14	**0.856**	**1.798**	**2.629**	**163.06**
O11–H11B···O13^{i}	**0.858**	**1.951**	**2.807**	**175.18**
O14–H14A···O12^{ii}	**0.865**	**1.931**	**2.754**	**158.35**
O15–H15B···O13	**0.894**	**2.260**	**2.967**	**135.74**
O9–H9A···O2	0.849	2.006	2.846	169.44
O10–H10A···O2^{ii}	0.848	1.851	2.686	167.63
O10–H10B···O3	0.856	1.811	2.654	167.75
O10–H10B···O4	0.856	2.532	2.958	111.71
O11–H11A···O1^{ii}	0.846	2.017	2.855	170.66
O12–H12A···O4	0.859	2.103	2.960	176.24
O12–H12B···O6^{iii}	0.852	1.987	2.746	147.79
O13–H13A···O3	0.860	2.060	2.810	145.36
O13–H13B···O7^{iv}	0.853	2.046	2.890	169.92
O14–H14B···O7^{v}	0.859	1.928	2.758	161.90
O15–H15A···O6	0.881	2.126	2.916	148.74

Symmetry code: $^{i}-x+1,\ y+1/2,\ -z+1$; $^{ii}-x+1,\ y-1/2,\ -z+1$; $^{iii}-x+1,\ y+1/2,\ -z$; $^{iv}-x+1,\ y-1/2,\ -z$; $^{v}x,\ y,\ z+1$.
^{a}D = donor; A = acceptor.

(2.85 Å), respectively. The O···O···O angles (117.31, 125.90°) are close to the theoretical value of 120° for the acyclic trimer in the gas phase [18, 19].

The $(H_2O)_3$ clusters are hydrogen bonded to carboxylate of pdtc to form a rich hydrogen-bond network, resulting in a 2-D layer arrangement. Hydrogen-bond data are shown in table 3. The 2-D structures further assemble into a 3-D supramolecular framework by hydrogen bonds and $\pi\cdots\pi$ interactions between adjacent bpy ligands with interplanar distance of 3.771 Å (figure 5).

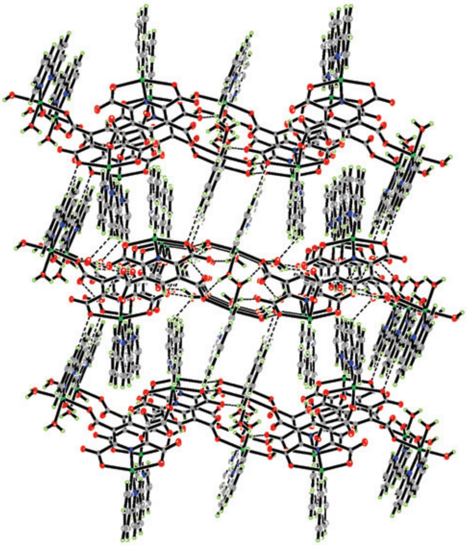

Figure 5. 3-D supramolecular structure of **1** built up by $\pi\cdots\pi$ and hydrogen-bond interactions.

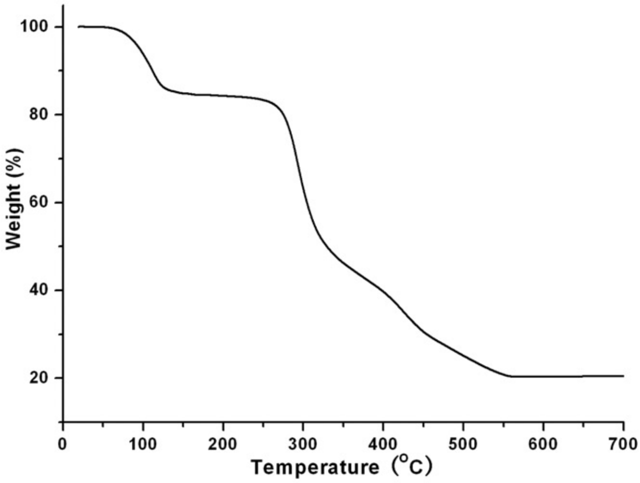

Figure 6. TG curve of **1**.

3.2. *Thermal analysis*

The structures of **1**–**3** are very similar, therefore only the TGA of **1** is described. The thermal behavior of **1** was examined in air (figure 6). The first mass loss of 15.01% from 50°C to 145°C is due to the loss of three coordinated and four lattice water molecules (calculated value 15.36%). From 145°C to 250°C, there is no weight change in the TG curve. The second weight loss from 250°C to 550°C corresponds to degradation of bpy and pdtc. The remaining mass of 20.45% is presumably ZnO, in agreement with the calculated value of 19.85%.

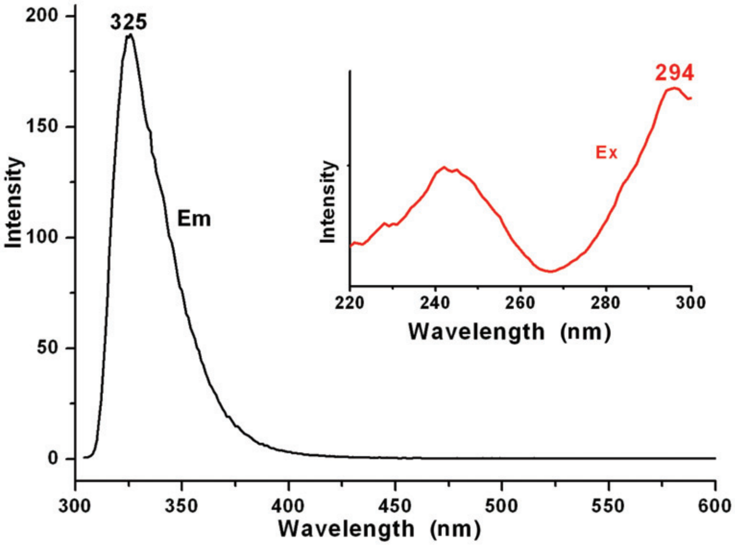

Figure 7. Emission and excitation spectra of **1**.

3.3. *Fluorescence*

The emission and excitation spectra of **1** in aqueous solution at room temperature are depicted in figure 7. There is a strong emission at $\lambda_{max} = 325$ nm with an excitation at 294 nm. The emission peak of 325 nm is due to $\pi^* \rightarrow \pi$ or $\pi^* \rightarrow n$ transitions of the ligands. The fluorescent properties of pdtc and bpy ligands were also measured, with only very weak emission peaks at 381 and 374 nm, respectively. The high fluorescence efficiency of **1** may be attributed to coordination of pdtc and bpy to zinc(II) that increases the rigidity of the ligand and reduces the loss of energy *via* radiationless thermal vibrations [20, 21]. The emission spectrum is similar with another Zn complex from pyrazine-2,3,5,6-tetracarboxylate and bpy [22], in which the double-chain structure also avoided further growth into 2-D because bpy acted as a blocking ligand. The similar structures may result in similar fluorescence.

4. Conclusion

Three 1-D complexes have been synthesized by the reaction of pyridine-2,3,5,6-tetracarboxylic acid and 2,2′-bipyridine (bpy) with corresponding salts. Two water trimers are observed in **1**–**3**, acting as connecters in the space between adjacent chains and making a 2-D layer. The 3-D supramolecular frameworks are built up by an intricate network of non-covalent interactions including hydrogen bonds and $\pi \cdots \pi$ interactions. The luminescence of **1** shows strong photoluminescence. Efforts to further investigate other luminescent chelating complexes, especially rare earth metal coordination polymers, are underway in our laboratory.

Supplementary material

CCDC-702809 **1**, CCDC-778746 **2**, and CCDC-778745 **3** contain the supplementary crystallographic data for this article. These data can be obtained free of charge *via* www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Centre, 12 Union Road, Cambridge CB21EZ, UK; Fax: (+44) 1223-336033; or E-mail: deposit@ccdc.cam.ac.uk).

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