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# Four metal–organic networks based on benzene-1,4-dioxyacetic acid and dipyrido[3,2-a:2',3'-c]phenazine ligand

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#### 1. Introduction

In the area of crystal engineering, the deliberate control of structure, property and function, is a long-term objective being vigorously pursued by chemists [1-4]. In this regard, much progress has been made on the design and synthesis of novel coordination frameworks and the relationships between their structures and properties [5–10]. The  $\pi$ - $\pi$  interaction is one of the most powerful non-covalent intermolecular interactions for directing the supramolecular architectures or influencing the physical properties of final products [11-14]. Therefore, the design of versatile functional ligands which are capable of providing the  $\pi$ -conjugated system for organizing their complexes into extended networks is quite desirable. 1,10-Phenanthroline derivatives such as pyrazino[2,3-f][1,10]phenanthroline and 2-phenyl-1H-1,3,7,8,tetraaza-cyclopenta[*l*]- phenanthrene are good candidates for construction of metal-organic supramolecular architectures through  $\pi$ - $\pi$  interactions. Up to now, the reports of this field are scarce [15-18]. In this contribution, we selected dipyrido[3,2-a:2',3'c]phenazine (dppz) as the neutral chelating ligands (Scheme 1). Benzene-1,4-dioxyacetic acid (H<sub>2</sub>bdoa) was selected as organic acid (Scheme 2). This ligand has rarely been used in the field of crystal engineering and supramolecular chemistry to construct coordination polymers. A potentially valuable feature of the ligand

# ABSTRACT

Four structurally diverse complexes,  $[Cd(dppz)(bdoa)]_n$  (1),  $[Zn(dppz)(bdoa)(H_2O)]_n$  (2),  $[Fe(dppz)_2(bdoa)]_n \cdot 2nH_2O$  (3), and  $[Co_2(dppz)_2(bdoa)_2(H_2O)]_n \cdot 3nH_2O$  (4), where  $H_2bdoa = benzene - 1, 4$ -dioxyacetic acid and dppz = dipyrido[3,2-a:2',3'-c]phenazine, have been hydrothermally synthesized. Compounds 1–4 feature chain structures. There exist  $\pi$ – $\pi$  interactions in the structures of 1, 2 and 4. Two neighboring chains of 1 are linked through the  $\pi$ – $\pi$  interactions into a double chain supramolecular structure. The chains of 2 and 4 are further extended by the  $\pi$ – $\pi$  interactions to form 3D and 2D supramolecular structures, respectively. The structural differences among such complexes show that the transition metals have important influences on their structures. The photoluminescent property of complex 2 and the magnetic property of complex 4 have also been investigated.

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is that the  $-OCH_2$ - group makes this ligand more flexible in comparison with the corresponding benzenedicarboxylate [3]. Herein, we report four novel complexes with different structures by varying the transition metals. The photoluminescent property of complex **2** and the magnetic property of complex **4** have also been studied.

# 2. Experimental

# 2.1. General

All reagents of analytical grade were purchased and used without further purification. The neutral chelating ligand dppz was synthesized according to the literature [19,20]. A Perkin–Elmer 240 elemental analyzer was used to collect microanalytical data. The photoluminescent properties of the ligands and compounds were measured on a Perkin–Elmer LS55 spectrometer.

# 2.2. Syntheses of the complexes

## 2.2.1. Synthesis of $[Cd(dppz)(bdoa)]_n$ (1)

A mixture of  $Cd(OH)_2$  (0.059 g, 0.40 mmol),  $H_2bdoa$  (0.090 g, 0.40 mmol), dppz (0.113 g, 0.40 mmol), and water (8 mL) was stirred for 10 min in air. The mixture was transferred and sealed in a Teflon reactor (15 mL) and heated at 160 °C for 3 days. After the mixture had been cooled to room temperature at 10 °C/h, colorless block crystals were obtained. Yield: 45% based on Cd(II). IR (KBr,



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#### Scheme 1. Structure of the dppz ligand.



Scheme 2. Structure of the H2bdoa acid.

cm<sup>-1</sup>): 3604 s, 3421 s, 3125 m, 2942 m, 2364 w, 1616 s, 1546 s, 1461 s, 1390 s, 1137 m, 1081 m, 939 w, 855 w, 742 s. Anal. Calc. for  $C_{28}H_{18}CdN_4O_6$ : C, 54.34; H, 2.93; N, 9.05. Found: C, 54.44; H, 2.85; N, 9.12%.

## 2.2.2. Synthesis of $[Zn(dppz)(bdoa)(H_2O)]_n$ (2)

The preparation of **2** was similar to that of **1** except that  $ZnCO_3$  was used instead of  $Cd(OH)_2$ . Colorless block crystals were obtained in a 41% yield based on Zn(II). IR (KBr, cm<sup>-1</sup>): 3357 s, 3104 s, 2357 w, 1626 s, 1476 s, 1358 s, 1148 m, 1073 m, 954 m, 834 m, 790 s, 700 s. *Anal.* Calc. for  $C_{28}H_{20}ZnN_4O_7$ : C, 57.01; H, 3.42; N, 9.50. Found: C, 57.15; H, 3.47; N, 9.43%.

## 2.2.3. Synthesis of $[Fe(dppz)_2(bdoa)]_n \cdot 2nH_2O(3)$

The preparation of **3** was similar to that of **1** except that FeCl<sub>2</sub> was used instead of Cd(OH)<sub>2</sub>. Red block crystals were obtained in a 27% yield based on Fe(II). IR (KBr, cm<sup>-1</sup>): 3479 s, 3119 s, 1626 s, 1574 s, 1526 m, 1504 m, 1439 m, 1342 s, 1248 m, 1111 m, 1095 m, 1032 w, 953 m, 768 s, 733 s, 656 m. *Anal.* Calc. for  $C_{23}H_{16}Fe_{0.50}N_4O_4$ : C, 62.74; H, 3.66; N, 12.72. Found: C, 62.61; H, 3.64; N, 12.81%.

## Table 1

Crystallographic data for compounds 1-4.

Compound	1	2	3	4
Formula	$C_{28}H_{18}CdN_4O_6$	$C_{28}H_{20}ZnN_4O_7$	$C_{23}H_{16}Fe_{0.50}N_4O_4$	C <sub>56</sub> H <sub>40</sub> Co <sub>2</sub> N <sub>8</sub> O <sub>16</sub>
Formula mass	618.86	589.85	440.32	1198.82
Space group	ΡĪ	ΡĪ	$P\bar{1}$	$P2_1/c$
Crystal size (mm)	$0.15 \times 0.10 \times 0.09$	$0.18 \times 0.15 \times 0.10$	$0.21 \times 0.17 \times 0.11$	$0.26 \times 0.16 \times 0.15$
a (Å)	8.5900(17)	9.5276(14)	8.1485(16)	20.0618(14)
b (Å)	12.220(2)	11.3497(17)	9.9529(19)	13.1722(9)
<i>c</i> (Å)	12.960(3)	11.9147(18)	12.467(2)	21.1529(15)
α (°)	109.23(5)	101.551(2)	110.590(2)	90
β(°)	100.10(4)	92.166(2)	96.225(4)	111.5340(10)
γ (°)	96.42(3)	93.645(2)	95.175(2)	90
V (Å <sup>3</sup> )	1243.4(4)	1258.1(3)	931.9(3)	5199.7(6)
Ζ	2	2	2	4
$M (mm^{-1})$	0.931	1.033	0.478	0.720
Goodness-of-fit (GOF) on F <sup>2</sup>	1.074	0.848	1.021	1.033
Reflections collected/unique	10 787/4883	10 906/4938	8092/3667	44 016/10 331
$D_{\text{calc}}$ (Mg m <sup>-3</sup> )	1.653	1.557	1.569	1.531
$\theta$ Range (°)	1.99-26.09	2.14-26.09	2.21-26.07	2.18-26.14
$R\left(I > 2\sigma(I)\right)$	0.0705, 0.2007	0.0437, 0.0572	0.0524, 0.1155	0.0402, 0.1030
R (all data)	0.0885. 0.2214	0.0942, 0.0681	0.0899. 0.1351	0.0611. 0.1144

Table 2
Selected bond distances (Å) and angles (°) for 1-4.

Complex <b>1</b>			
Cd-O(6)	2.369(11)	O(6)-Cd-O(2)	81.0(4)
Cd-O(2)	2.455(11)	O(2)-Cd-O(1)	50.8(4)
Cd-O(1)	2.616(10)	O(1)-Cd-N(1)	126.7(4)
Cd-N(1)	2.670(12)	O(6)-Cd-N(1)	85.1(4)
Cd-N(2)	2.717(10)	O(2)-Cd-N(1)	77.2(4)
Complex 2			
Zn-O(1W)	1.984(3)	O(1W) - Zn - O(4)	91.61(13)
Zn-O(4)	2.020(2)	O(4) - Zn - N(2)	168.35(10)
Zn-O(1)	2.074(2)	O(4) - Zn - O(1)	93.04(10)
Zn-N(1)	2.110(3)	O(1W)-Zn-N(1)	105.26(11)
Zn-N(2)	2.196(3)	O(4) - Zn - N(1)	91.70(10)
Complex 3			
$Fe_{-}O(1)$	2.054(2)	N(2)A - Fe - N(1)	106 78(9)
Fe-N(2)	2,051(2)	O(1)A-Fe-N(1)	86 44(9)
Fe-N(1)	2.255(3)	O(1)-Fe- $O(1)$ A	180.0
10 11(1)	2.202(0)	O(1)A-Fe-N(2)A	92,45(9)
		N(2) - Fe - N(1)	73.22(9)
Commissi A			
Complex 4	2.0401(10)	$O(11)$ $C_{2}(1)$ $O(1)$	170.00(0)
$C_0(1) = O(11)$	2.0481(18)	O(11) - O(1) - O(1)	178.96(8)
$C_0(1) = O(7)$	2.1138(18)	O(7) - CO(1) - O(4vv)	94.73(7)
$C_0(1) = O(1)$	2.1149(19) 2.124(2)	O(7) - O(1) - N(2)	91.54(7)
CO(1) - IN(2)	2.124(2) 2.1492(10)	N(2) = CO(1) = N(1)	77.10(8)
Co(1) = O(4vv)	2.1483(19)	O(4vv) = CO(1) = N(1)	97.67(8)
$C_0(1) = IN(1)$	2.133(2) 2.0478(10)	O(1) - O(1) - N(1) O(8) Co(2) O(5)	93.00(8)
$C_0(2) = O(12)$	2.0478(19)	O(5) - CO(2) - O(5)	02.06(0)
$C_0(2) = O(8)$	2.0048(19)	O(12) = CO(2) = N(0)	92.00(9)
$C_0(2) = O(3)$	2.103(2) 2.125(2)	O(12) - O(2) - O(400) O(400) - O(2) - N(6)	93.29(7)
$C_0(2) = I_1(3)$	2.12J(2) 2.12/6(18)	O(400) = CO(2) = N(0) O(12) = Co(2) = N(5)	80 37(8)
$C_0(2) = O(4VV)$	2.1240(10) 2.150(2)	O(12) - O(2) - N(5)	80.08(8)
CO(2) - IN(0)	2.130(2)	O(3) = O(2) = N(3)	09.90(0)

Symmetry code for A: -x + 2, -y + 2, -z.



**Fig. 1.** (a) The coordination environment of the Cd(II) center in compound **1**. (b) The 1D chain structure. (c) The supramolecular chain formed through  $\pi$ - $\pi$  stacking interactions. Symmetry codes: (A) x + 1, -1 + y, z; (B) x, y + 1, z.

# 2.2.4. Synthesis of $[Co_2(dppz)_2(bdoa)_2(H_2O)]_n \cdot 3nH_2O(4)$

The preparation of **4** was similar to that of **1** except that CoCO<sub>3</sub> was used instead of Cd(OH)<sub>2</sub>. Purple block crystals were obtained in a 33% yield based on Co(II). IR (KBr, cm<sup>-1</sup>): 3566 s, 3447 s, 3268 s, 2939 m, 2327 w, 1805 w, 1551 s, 1416 s, 1267 s, 1193 m, 1133 m, 999 m, 864 m, 789 s, 730 s, 625 w. *Anal.* Calc. for C<sub>56</sub>H<sub>38</sub>Co<sub>2</sub>-N<sub>8</sub>O<sub>16</sub>: C, 56.20; H, 3.20; N, 9.36. Found: C, 56.26; H, 3.15; N, 9.51%.

# 2.3. X-ray crystallography

Diffraction data for complexes **1–4** were recorded on a Bruker-AXS Smart CCD diffractometer, using a  $\omega$  scan technique with Mo K $\alpha$  radiation ( $\lambda$  = 0.71073 Å) at 293(2) K. All the structures were solved by direct method of SHELXS-97 [21] and refined by full-matrix least-squares techniques using the SHELXL-97 [22] program.



**Fig. 2.** (a) The coordination environment of the Zn(II) center in compound **2**. (b) The 1D chain structure. (c) The 3D supramolecular structure formed through  $\pi$ - $\pi$  stacking interactions. Symmetry codes: (A) –*x*, –*y* – 1, –*z*; (B) –*x*–1, –*y*, 1 – *z*.



Fig. 3. (a) The coordination environment of the Fe(II) center in compound 3. (b) The 1D chain structure. Symmetry codes: (A) 2 - x, -y + 2, -z; (B) 3 - x, 1 - y, -z; (c) -1 + x, 1 + y, z.

Non-hydrogen atoms were refined with anisotropic temperature parameters, and hydrogen atoms of the ligands were refined as rigid groups. The H atoms of two lattice water molecules in compound 4 could not be positioned reliably. Other H atoms of water molecules were located from difference Fourier maps. Further details for structural analysis are summarized in Table 1. Selected bond distances and angles for compounds 1–4 are shown in Table 2.

#### 3. Results and discussion

## 3.1. Description of crystal structures

#### 3.1.1. Structure description of $[Cd(dppz)(bdoa)]_n$ (1)

Single-crystal X-ray diffraction reveals that the structure of compound 1 is a 1D chain. As shown in Fig. 1a, the cadmium ion is five-coordinated in a distorted square-pyramidal environment by two nitrogen atoms from one dppz ligand and three oxygen atoms from two bdoa anions. The bond lengths of Cd-O (2.369(11)–2.616(10) Å) and Cd–N1 (2.670(12) Å) are all within the normal ranges; the bond length of Cd-N2 (2.717(10) Å) is a little bit longer, but shorter than the van der Waals contact distance for Cd–N of 3.17 Å [23]. Cd ions are bridged by bdoa anions to form a 1D chain structure (Fig. 1b). As shown in Fig. 1c, there exist faceto-face  $\pi - \pi$  interactions between the dppz ring and phenyl ring of bdoa anion of adjacent chain (with centroid-to-centroid distance of 3.56 Å, face-to-face distance of 3.38 Å, and dihedral of 4.09°), resulting in a double chain structure of **1**.

# 3.1.2. Structure description of $[Zn(dppz)(bdoa)(H_2O)]_n$ (2)

As shown in Fig. 2a, the Zn atom of **2** is five-coordinated by two nitrogen atoms from one dppz ligand, two carboxylate oxygen atoms from two different bdoa anions, and one water molecule. The Zn–O (1.984(3)–2.074(2)Å) and Zn–N (2.110(3)–2.196(3)Å) bond lengths are all within the normal ranges [24,25]. Each bdoa anion coordinates to two Zn atoms with its bis-monodentate carboxylate oxygen atoms, and the Zn atoms are bridged by bdoa anions to form a chain (Fig. 2b). The dppz ligands are attached to the Zn atoms, acting as a bidentate ligand. Unlike the structure of 1, the dppz ligands in 2 are extended on two sides of the chain. As shown in Fig. 2c, the dppz ligands from neighboring chains are well matched with each other, and adjacent chains are linked through  $\pi$ - $\pi$  stacking interactions between two dppz rings (with the centroid-centroid distance of 3.53 Å, face-to-face distance of 3.45 Å, and dihedral of 4.04°), which extend the chains into a 3D supramolecular structure. It is believed that the very strong  $\pi$ - $\pi$  stacking interactions play an important role in stabilizing the 1D chain and the 3D supramolecular structure [26-28].

#### 3.1.3. Structure description of $[Fe(dppz)_2(bdoa)]_n \cdot n2H_2O(\mathbf{3})$

As shown in Fig. 3a. Fe atom lies at an inversion center and is six-coordinated by four nitrogen atoms from two dppz ligands, and two carboxylate oxygen atoms from two bdoa anions. The distances of the Fe-O bond (2.054(2) Å) and Fe-N bonds (2.253(3)-2.262(3) Å) are near to those of reported [29,30]. The Fe atoms are bridged by bdoa molecules forming a 1D chain. The dppz ligands are attached on both sides of the chains. Unlike the structures of **1** and **2**, there exists no  $\pi - \pi$  interaction in the structure.



**Fig. 4.** (a) The coordination environment of the Co(II) center in compound **4.** (b) Polyhedral representation of the 1D chain structure of **4.** (c) The  $\pi$ - $\pi$  interactions. (d) The 2D supramolecular layer formed through  $\pi$ - $\pi$  stacking interactions. Symmetry codes: (A) -x, -y + 2, -z; (B) x, -y + 1.5, 0.5 + z.



Fig. 5. Solid-state photoluminescent spectrum of 2 at room temperature.

The lattice water molecule is hydrogen-bonded to carboxylate oxygen atom, stabilizing the chain structure of **3**.

## 3.1.4. Structure description of $[Co_2(dppz)_2(bdoa)_2(H_2O)]_n \cdot 3nH_2O$ (4)

As shown in Fig. 4a, the structure of 4 contains two kinds of unique Co atoms. Both Co1 and Co2 are six-coordinated by three oxygen atoms from three bdoa anions (Co-O 2.0478(19)-2.1149(19) Å), one µ2-H2O (Co-O4W 2.1246(18)-2.1483(19) Å) and two N atom from dppz ligand (Co-N 2.124(2)-2.153(2) Å). Two carboxylate groups of bdoa anion adopt bis-monodentate mode, and coordinate with Co1 and Co2, respectively. The rest of bdoa anion displays a  $\mu$ -4 bis-bidentate coordination mode. The Co centers are connected by  $\mu_2$ -H<sub>2</sub>O and bdoa anions to form a dinuclear cobalt-oxygen cluster. The cobalt-oxygen clusters are connected through bdoa anions to form a 1D chain (Fig. 4b). There exist two types of  $\pi$ - $\pi$  interactions among adjacent layers: one is between dppz ligands (with the centroid-centroid distance of 3.91 Å, face-to-face distance of 3.35 Å, and dihedral of 3.39°), and another is between dppz ligand and bdoa anion (with the centroid-centroid distance of 3.57 Å, face-to-face distance of 3.40 Å, and dihedral of 7.47°, Fig. 4c). The chains of 4 are connected by the two kinds of  $\pi$ - $\pi$  interactions to form a 2D supramolecular layer (Fig. 4d). Obviously, the  $\pi$ - $\pi$  stacking interactions play an important role in the formation and stabilization of the 2D supramolecular structure of 4 [26-28]. In addition, the intramolecular and intermolecular O-H-O hydrogen bonds involving the water molecules and the carboxylic groups of bdoa anions further stabilize the 2D framework (O4W-H4WB-01: 0.868(18), 2.263(18), and 2.898(3) Å, 130(2)°; O4W-H4WB-O2: 0.868(18), 1.73(2), and 2.578(3) Å, 164(3)°; 04W-H4WA...06: 0.838(17), 1.738(18), and 2.554(3) Å, 164(3)°; O3W-H3WB-05: 0.922(19), 2.29(2), and 3.214(5) Å, 177(6)°, symmetry code: x, y – 1, z + 1).

#### 3.2. Photoluminescent property

The solid-state photoluminescent spectrum of compound **2** is depicted in Fig. 5. Compound **2** exhibit emissions at about 539 nm ( $\lambda_{ex}$  = 360 nm). In order to understand the nature of this emission band, the photoluminescence properties of free ligands dppz and H<sub>2</sub>dboa were studied. The main emission peak of dppz is at 444 nm, which may be attributed to the  $\pi^* \rightarrow \pi$  transition [16]. The dicarboxylate ligands H<sub>2</sub>dboa ( $\lambda_{em}$  = 380 nm) can also exhibit fluorescence at room temperature. The emission bands can be assigned to the  $\pi^* \rightarrow \pi$  transition as previously reported [31–33]. Compared with the photoluminescent spectra, the emission spectra of **2** are obviously similar to that of dppz. Thus, the green pho-



**Fig. 6.** Plots of the temperature dependence of  $\chi_m T$  (open squares) and  $\chi_m^{-1}$  (open triangles) for compound **4**.

toluminescent emission of **2** may be attributed to the intraligand fluorescent emissions of dppz [8,34].

# 3.3. Magnetic property

The temperature-dependent magnetic susceptibility data of compound **4** has been measured for polycrystalline samples at an applied magnetic field of 1000 Oe in the temperature range of 2–300 K (Fig. 6). For **4**, the  $\chi_m T$  value at 300 K is 3.85 cm<sup>3</sup> mol<sup>-1</sup> K (5.55  $\mu_B$ ), which is consistent with the expected value (3.75 cm<sup>3</sup> mol<sup>-1</sup> K, 5.48  $\mu_B$ ) of two isolated S = 3/2 spin-only Co<sup>II</sup> ions (g = 2.0). Upon cooling, the values of  $\chi_m T$  keep smoothly decreasing in 300–2.0 K range, indicating an overall antiferromagnetic coupling in compound **4** [35,36]. The magnetic can be well fit to Curie–Weiss law with C = 4.03 cm<sup>3</sup> mol<sup>-1</sup> K,  $\theta = -16.88$  K. The negative  $\theta$  value further confirms the presence of antiferromagnetic interaction in **4**.

#### 4. Conclusions

Four complexes with dppz and H<sub>2</sub>bdoa ligands have been hydrothermally synthesized under similar synthetic conditions. The complexes form 1D polymeric structure, and three of which connected by  $\pi$ - $\pi$  interactions to form supramolecular structures. The luminescent property of compound **2** demonstrates that it may be good candidates for luminescent materials. The magnetic study shows the presence of antiferromagnetic interaction in **4**. Further research on rational design and construction of related compounds with interesting supramolecular structures as well as physical properties is currently underway in our laboratory.

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#### **Appendix A. Supplementary material**

CCDC 751617, 751618, 751619 and 751620 contain the supplementary crystallographic data for  $[Cd(dppz)(bdoa)]_n$ ,  $[Zn(dppz)(bdoa)(H_2O)]_n$ ,  $[Fe(dppz)_2(bdoa)]_n \cdot 2nH_2O$ , and  $[Co_2 - (dppz)_2(bdoa)_2(H_2O)]_n \cdot 3nH_2O$ . These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data\_request/cif. Supplementary data

associated with this article can be found, in the online version, at doi:10.1016/j.ica.2009.12.056.

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