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# Supramolecular coordination networks constructed from infinite one-dimensional chains with 5-nitroisophthalate as bridge

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#### Abstract

Four chain-like coordination polymers  $[Zn(NIPH)(py)_3]_n$  (1),  $[Mn(NIPH)(py)_3]_n$  (2),  $\{[Co(NIPH)(py)_3](H_2O)\}_n$  (3), and  $\{[Cd(NI-PH)(im)_2(H_2O)\}_n$  (4), (where NIPH = 5-nitroisophthalate, py = pyridine, and im = imidazole) were synthesized and characterized by elemental analysis, IR, single-crystal X-ray diffraction, and TGA analysis. Crystal data for 1: monoclinic, C2/c, a = 20.044(4) Å, b = 16.182(3) Å, c = 14.539(3) Å,  $\beta = 111.96(3)^\circ$ , Z = 8. Crystal data for 2: monoclinic, C2/c, a = 19.3590(13) Å, b = 16.5100(9) Å, c = 14.6392(7) Å,  $\beta = 111.844(3)^\circ$ , Z = 8. Crystal data for 3: monoclinic, C2/c, a = 19.797(4) Å, b = 16.256(3) Å, c = 14.619(3) Å,  $\beta = 111.82(3)^\circ$ , Z = 8. Crystal data for 4: monoclinic, P2(1)/c, a = 7.6882(15) Å, b = 17.861(4) Å, c = 13.219(3) Å,  $\beta = 92.53(3)^\circ$ , Z = 4. Single-crystal X-ray diffraction analysis reveals that 1, 2, 3, and 4 have 1D zig-zag chain structures constructed from  $\mu_2$ -bridge NIPH ligands and metal atoms. All of these four chain-like structures are finally packed into three-dimensional supramolecular networks through hydrogen bonds and  $\pi \cdots \pi$  stacking interactions. The thermal properties of these complexes have been measured.

Keywords: Coordination polymer; Crystal structure; Chain; Hydrogen bonds

## 1. Introduction

The design and construction of functional supramolecular coordination polymers have attracted great attention for their intriguing network topologies and their potential applications as absorption, molecular separation, selective catalysis, optoelectronic and magnetic materials [1–9]. Most coordination polymers reported so far involve the using of polyfunctional organic ligands to bridge metal ions by metal coordination covalent bonding, with the aid of hydrogen bonding,  $\pi \cdots \pi$  stacking interaction, and other weak intermolecular interactions [10–13]. Polycarboxylic acids and their derivatives are very important ligands for desired extended networks [14,15]. Recent studies have demonstrated that 5-nitroisophthalate (NIPH) is a versatile ligand in the synthesis of multidimensional supra-

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molecular coordination polymers because its two carboxylic groups can bond with metal centers and one nitro group can act as a hydrogen-bond acceptor [16–20].

On the other hand, 1D chain is the basic building block in coordination polymers, as well as several infinite 1D coordination polymers with zig-zag, linear, ladder, double and helix chain motifs have been described recently [21-24]. In our previous work, we have performed some 1D chain-like coordination polymers with luminescence and magnetism [25]. In this contribution, four coordination polymers,  $[Zn(NIPH)(py)_3]_n$  (1),  $[Mn(NIPH)(py)_3]_n$  (2),  $\{[Co(NIPH)(py)_3](H_2O)\}_n$  (3) and  $\{[Cd(NIPH)(im)_2]$  $(H_2O)$ ] $(H_2O)$ }<sub>n</sub> (4), were synthesized by the use of 5-nitroisophthalate (NIPH) as a bridge ligand and pyridine (py) and imidazole (im) as terminal ligands. The detailed characterizations of elemental analysis, IR, single-crystal Xray diffraction, and TGA analysis are reported for the coordination polymers 1, 2, 3, and 4. All of these coordination polymers display 1D chain structure feature and the 1D chains pack into different three-dimensional supramo-

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Table 1	
Crystal data and	structure refinement for 1–4

	1	2	3	4
Empirical formula	C <sub>23</sub> H <sub>18</sub> N <sub>4</sub> O <sub>6</sub> Zn	C23H18N4O6Mn	C <sub>23</sub> H <sub>19</sub> N <sub>4</sub> O <sub>7</sub> Co	C14H15N5O8Cd
Formula weight	511.78	501.35	523.36	493.71
Crystal size	0.30*0.19*0.16	0.37*0.21*0.16	0.35*0.14*0.11	0.44*0.15*0.14
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	C2/c	C2/c	C2/c	P2(1)/c
a (Å)	20.044(4)	19.3590(13)	19.797(4)	7.6882(15)
b (Å)	16.182(3)	16.5100(9)	16.256(3)	17.861(4)
c (Å)	14.539(3)	14.6392(7)	14.619(3)	13.219(3)
χ (°)	90	90	90	90
β (°)	111.96(3)	111.844(3)	111.82(3)	92.53(3)
v (°)	90	90	90	90
Volume (Å <sup>3</sup> )	4373.6(15)	4343.0(4)	4367.3(15)	1813.5(6)
Z	8	8	8	4
$D_{\rm C}({\rm mg/m^3})$	1.554	1.534	1.592	1.808
Limiting indices	$0 \leq h \leq 25, 0 \leq k \leq 20,$	$0 \leq h \leq 25, 0 \leq k \leq 21,$	$0 \leq h \leq 25, -21 \leq k \leq 21,$	$0 \leq h \leq 9, 0 \leq k \leq 23,$
	$-18 \leq l \leq 17$	$-19 \leqslant l \leqslant 17$	$-18 \leqslant l \leqslant 17$	$-17 \leqslant l \leqslant 17$
$\mu ({\rm mm}^{-1})$	1.172	0.657	0.841	1.259
F(000)	2096	2056	2152	984
Data/restraints/ parameters	5003/0/379	5138/0/307	4881/0/316	4142/0/269
GooF	0.998	1.007	0.871	1.004
$R_1 \left[I > 2\sigma(I)\right]^a$	0.0309	0.0318	0.0642	0.0209
$vR_2 [I \ge 2\sigma(I)]^a$	0.0588	0.0685	0.1485	0.0414
$R_1$ (all data) <sup>a</sup>	0.0616	0.0574	0.1245	0.0318
$wR_2$ (all data) <sup>a</sup>	0.0741	0.0778	0.1737	0.0440

<sup>a</sup>  $R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|; wR_2 = \{\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)]^2 \}^{1/2}.$ 

lecular networks based on hydrogen bonding and  $\pi \cdots \pi$  stacking interactions.

#### 2. Experimental

#### 2.1. Materials and methods

5-Nitroisophthalic acid (H<sub>2</sub>NIPH), pyridine (py), and imidazole (im) were purchased from Fluka and used without further purification. All of other reagents were procured from commercial sources and used as received. Elemental analysis was performed on a Perkin–Elmer 2400 CHN Elemental Analyzer. Thermogravimetric analysis (TGA) was conducted on a Perkin–Elmer TGA 7 unit with a heating rate of 10 °C/min in air. IR spectra were measured on a Nicolet 5PC FT-IR spectrophotometer in the range 4000–400 cm<sup>-1</sup>.

# 2.2. Preparation of $[Zn(NIPH)(py)_3]_n$ (1)

A mixture of  $Zn(NO_3)_2 \cdot 6H_2O$  (0.149 g, 0.5 mmol), H<sub>2</sub>NIPH (0.106 g, 0.5 mmol), py (1 ml), DMF (5 ml), and water (5 ml) was sealed in a 25 ml Teflon-lined stainless steel autoclave and was heated at 120 °C for 72 h. After cooling to room temperature, colorless block-shaped crystals were obtained with yield of 65% based on Zn. Anal. calcd. for C<sub>23</sub>H<sub>18</sub>N<sub>4</sub>O<sub>6</sub>Zn: C, 53.98%; H, 3.54%; N, 10.95%. Found: C, 53.57%; H, 3.48%; N, 11.12%. IR (KBr, cm<sup>-1</sup>): 3379(m), 3100(w), 1636(s), 1571(s), 1533(s), 1448(s), 1401(m), 1348(s), 1221(w), 1070(m), 1037(m), 927(w), 787(w), 731(m).

## 2.3. Preparation of $[Mn(NIPH)(py)_3]_n$ (2)

The synthesis procedure of **2** was similar to the above description for **1** except that  $Zn(NO_3)_2 \cdot 6H_2O$  was replaced by  $MnCl_2 \cdot 4H_2O$  (0.099 g, 0.5 mmol). The yield was 55% based on Mn. Anal. calcd. for  $C_{23}H_{18}N_4O_6Mn$ : C, 55.10%; H, 3.62%; N 11.18%. Found: C, 55.04%; H, 3.53%; N, 11.35%. IR (KBr, cm<sup>-1</sup>): 3404(m), 3092(w), 1641(vs), 1598(m), 1534(s), 1441(m), 1407(m), 1346(s), 1220(w), 1070(w), 1037(w), 931(w), 790(w), 761(w), 733(m).

## 2.4. Preparation of $[Co(NIPH)(py)_3(H_2O)]_n(3)$

The synthesis procedure of **3** was similar to the above description for **1** except that  $Zn(NO_3)_2 \cdot 6H_2O$  was replaced by  $Co(NO_3)_2 \cdot 6H_2O(0.145 \text{ g}, 0.5 \text{ mmol})$ . The yield was 55% based on Co. Anal. calcd. for  $C_{23}H_{19}N_4O_7Co$ : C, 52.78%; H, 3.85%; N, 10.70%. Found: C, 52.64%; H, 3.78%; N, 10.82%. IR (KBr, cm<sup>-1</sup>): 3442(m), 3059(w), 2787(w), 1719(m), 1586(vs), 1541(m), 1468(s), 1389(s), 1348(s), 1218(w), 1079(m), 1022(w), 920(w), 804(s), 732(w).

#### 2.5. Preparation of $[Cd(NIPH)(im)_2(H_2O)] \cdot H_2O$ (4)

A mixture of CdCl<sub>2</sub>·2.5H<sub>2</sub>O (0.114 g, 0.5 mmol), H<sub>2</sub>NIPH (0.106 g, 0.5 mmol), im (0.136 g, 1 mmol), and H<sub>2</sub>O (10 ml) was stirred for 30 min and heated at 140 °C

Table 2 Selected bond lengths [Å] and angles [°] for 1–4

$\begin{array}{l} 1 \\ Zn-O(3)^i \\ Zn-N(3) \end{array}$	2.003(2) 2.129(2)	Zn-O(2) Zn-N(2)	2.109(2) 2.227(2)	Zn-O(1) Zn-N(4)	2.475(2) 2.218(2)
$\begin{array}{l} O(3)^{i} - Zn - O(2) \\ O(2) - Zn - N(4) \\ O(3A) - Zn - N(2) \\ O(3)^{i} - Zn - O(1) \\ N(3) - Zn - O(1) \end{array}$	121.96(7) 89.48(7) 90.17(8) 178.44(7) 86.67(7)	$\begin{array}{l} O(3)^{i} - Zn - N(3) \\ N(3) - Zn - N(4) \\ O(2) - Zn - N(2) \\ O(2) - Zn - O(1) \\ N(4) - Zn - O(1) \end{array}$	94.86(8) 92.14(8) 86.77(7) 56.50(6) 90.16(7)	$\begin{array}{c} O(2)-Zn-N(3)\\ O(3)^i-Zn-N(4)\\ N(4)-Zn-N(2)\\ N(3)-Zn-N(2)\\ N(2)-Zn-O(1) \end{array}$	143.15(7) 90.05(8) 175.72(7) 92.10(8) 89.50(7)
2 Mn–O(1) Mn–N(2)	2.049(2) 2.226(2)	$Mn-O(3)^i$ Mn-N(3)	2.207(2) 2.298(2)	$\begin{array}{c} Mn\text{-}O(4)^{i} \\ Mn\text{-}N(4) \end{array}$	2.362(2) 2.303(2)
$\begin{array}{l} O(1)-Mn-O(3)^{i} \\ O(3)^{i}-Mn-N(2) \\ O(1)-Mn-N(3) \\ N(2)-Mn-N(3) \\ O(1)-Mn-N(4) \end{array}$	123.67(5) 145.65(6) 90.48(6) 90.36(6) 89.73(6)	$\begin{array}{l} N(3)-Mn-N(4)\\ O(3)^{i}-Mn-O(4)^{i}\\ N(2)-Mn-O(4)^{i}\\ N(4)-Mn-O(4)^{i}\\ N(2)-Mn-N(4) \end{array}$	177.53(6) 55.68(5) 89.98(6) 89.80(5) 92.10(6)	$\begin{array}{c} O(1)-Mn-O(4)^{i}\\ O(1)-Mn-N(2)\\ N(3)-Mn-O(4)^{i}\\ O(3)^{i}-Mn-N(3)\\ O(3)^{i}-Mn-N(4) \end{array}$	179.22(6) 90.67(6) 89.95(5) 90.30(5) 87.53(5)
<b>3</b> Co–O(3) <sup>i</sup> Co–N(4)	2.012(3) 2.206(4)	Co–N(3) Co–O(1)	2.136(4) 2.206(3)	Co-O(2) Co-N(2)	2.192(4) 2.195(4)
$\begin{array}{l} O(3)^{i}-Co-N(3) \\ N(3)-Co-O(2) \\ O(3)^{i}-Co-N(2) \\ O(2)-Co-N(2) \\ O(3)^{i}-Co-N(4) \end{array}$	93.71(15) 151.74(14) 89.15(14) 89.83(13) 89.83(13)	O(2)-Co-N(4) O(3A)-Co-O(1) N(3)-Co-O(1) N(2)-Co-O(1) N(4)-Co-O(1)	87.04(13) 173.84(13) 92.45(14) 90.61(12) 89.80(13)	O(3A)-Co-O(2) N(2)-Co-N(4) O(2)-Co-O(1) N(3)-Co-N(2) N(3)-Co-N(4)	114.23(13) 176.09(15) 59.32(12) 91.77(14) 92.10(14)
4 Cd–O(1) Cd–N(2)	2.221(2) 2.293(2)	Cd-N(4) $Cd-O(3)^{i}$	2.256(2) 2.535(2)	$\begin{array}{c} Cd{-}O(7)\\ Cd{-}O(4)^i\end{array}$	2.360(2) 2.420(2)
$\begin{array}{l} O(1)-Cd-N(4) \\ N(4)-Cd-N(2) \\ O(1)-Cd-O(7) \\ N(2)-Cd-O(7) \\ O(1)-Cd-O(4)^{i} \end{array}$	137.75(6) 91.69(6) 88.77(6) 177.45(6) 129.70(5)	$\begin{array}{c} N(2)-Cd-O(4)^{i}\\ O(1)-Cd-O(3)^{i}\\ N(4)-Cd-O(3)^{i}\\ O(7)-Cd-O(3)^{i}\\ O(4)^{i}-Cd-O(3)^{i} \end{array}$	88.87(6) 77.28(5) 144.97(5) 91.02(6) 52.51(5)	$O(7)-Cd-O(4)^{i}$ O(1)-Cd-N(2) $N(2)-Cd-O(3)^{i}$ N(4)-Cd-O(7) $N(4)-Cd-O(4)^{i}$	89.26(6) 91.10(6) 86.47(6) 90.12(6) 92.50(6)

Symmetry transformations used to generate equivalent atoms: for 1: i. -x + 3/2, y-1/2, -z + 1/2. For 2: i. -x + 1/2, y + 1/2, -z + 3/2. For 3: i. -x + 1/2, y-1/2, -z + 3/2. For 4: i. -x, y + 1/2, -z + 1/2.

for 120 h in a Teflon-lined stainless steel autoclave (25 ml) under autogenous pressure. After cooling to room temperature, the block-shaped crystals were obtained and washed with water and dried in air. The yield was 60% based on Cd. Element analysis calcd (%) for  $C_{14}H_{15}N_5O_8Cd$ : C 34.06%; H 3.06%; N 14.19%; found: C 33.95%; H 2.98%; N 14.28%. IR (KBr, cm<sup>-1</sup>) data; 3384(m), 1610(vs), 1558(s), 1528(w), 1449(m), 1370(s), 1340(s), 1071(m), 926(w), 732(s), 654(w), 610(w).

#### 2.6. X-ray data collection and structure determination

The crystal structures of 1–4 were determined by singlecrystal X-ray diffraction experiment. The reflection data were collected on a Rigaku R-AXIS RAPID diffractometer (MoK $\alpha$  radiation, graphite monochromator) at room temperature with  $\omega$ -scan mode. Empirical absorption correction was applied for all data. The structure was solved by direct methods and refined by full-matrix least squares on  $F^2$  using SHELXTL97 software [26]. The heaviest atoms were firstly located. O and C atoms were subsequently located in difference Fourier maps. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms of 1–4 were located based on theoretical riding models except those in water molecules of 4 which were deduced from difference Fourier maps and refined isotropically.



Fig. 1. Perspective view of coordination environment of metal atom for 1-3 [M = Zn (1), Mn (2), Co (3). Hydrogen atoms are omitted for clarity.



Fig. 2. Perspective view of coordination environment of Cd(II) in 4.



Fig. 3. View of the one-dimensional zig-zag chains in (a) 1-3 and (b) 4.

Experimental details for the structure determination are given in Table 1. The selected bond distance and angles are tabulated in Table 2. The hydrogen bonds are listed in Table S1.

Crystallographic data for 1–4 have been deposited at the Cambridge Crystallographic Data Center with the deposition number of CCDC 284161–284163 and 607755. These data can be obtained free of charge from the Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/ conts/retrieving.html [or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; e-mail:deposit@ccdc.cam.ac.uk].

# 3. Result and discussion

## 3.1. Crystal structures of complexes 1, 2, and 3

Single-crystal structure analysis reveals that 1, 2, and 3 are composed of 1D zig-zag chains. The molecular structures of 1, 2, and 3 are shown in Figs. 1, S1 and S2, respectively. In the asymmetric units of 1, 2, and 3, there is one central metal atom, one NIPH and three pyridine molecules, except that asymmetric unit of 3 contains additional uncoordinated water molecule. The coordination environ-



Fig. 4. Packing structures for 1 (a) and 4 (b).



Fig. 5. Perspective view of the hydrogen-bonds between chains in 1, (a) along the a axis and (b) along b axis, and in 4, (c) along the a axis and (d) along c axis.

ments of central metal atoms of 1, 2, and 3 can be described as a distorted octahedron formed by three carboxylic-oxygen atoms from two different NIPH ligands and three nitrogen atoms from three different pyridine molecules. The average bond distances of Zn-O, Mn-O, Co-O, Zn-N, Mn-N, and Co-N are 2.196, 2.206, 2.137, 2.191, 2.276, and 2.179 Å, respectively, which are in good agreement with those found in other extended structures constructed from NIPH ligands [16–20]. Each  $\mu_2$ -bridge NIPH ligand coordinates with two metal atoms in bidentate chelating/monodentate modes, resulting in a zig-zag chain (Fig. 3). The three-dimensional networks of 1, 2, and 3 are formed from one-dimensional chains via C-H···O hydrogen bonds and  $\pi \cdot \cdot \pi$  stacking interactions (Fig. 4). It is found that there are  $C-H \cdots O$  hydrogen bonding patterns between py ligand C-H groups and nitro-oxygen atoms of NIPH, py ligand C-H groups and carboxylate oxygen atoms of NIPH (Figs. 5 and S3). Moreover, there are  $\pi \cdots \pi$  stacking interactions with contact distance of 3.393 Å between the aromatic rings of the py molecules of adjacent chains.

# 3.2. Crystal structures of complex 4

In order to investigate the influence of the terminal ligand on structures, im was employed to replace py ligand, the  $\{[Cd(NIPH)(im)_2(H_2O)](H_2O)\}_n$  (4) and  $[M(NIPH)(im)_2]$  (M = Zn, Co) were isolated respectively.  $[M(NIPH)(im)_2]$  (M = Zn, Co) have been reported previously [22,27], therefore we only describe the structure of 4. In the asymmetric unit of 4, there is one Cd ion, one NIPH ligand dianion, two im ligands, one coordination water molecule and one uncoordination water molecule. The environment of Cd atom can be described as a distorted octahedral with three oxygen atoms (O1, O3A, O4A) from two bridging NIPH ligands and one nitrogen

atom (N4) from one im ligand in the equatorial plane, and one oxygen atom (O7) of the water molecules and one nitrogen atom (N2) from one im ligand at the axial position (Fig. 2). The average bond Cd-O and Cd-N distances are 2.384 and 2.275 Å, respectively. Similar to 1, 2, and 3, each NIPH ligand links two metal centers and each metal center connects two NIPH ligands to form the zigzag chain structure (Fig. 3). The supramolecular structure of 4 is the three-dimensional network constructed from chains via hydrogen bonds (Figs. 4 and 5). There are three types of hydrogen bonds involving the O-H···O hydrogen bond between the coordinated water molecules and carboxylic-oxygen of NIPH ligands, C-H···O hydrogen bond between the C-H groups of im ligands and carboxylic-oxygen of NIPH ligands, N-H···O hvdrogen bond between the N-H groups of im ligands and nitro group of NIPH ligands. The uncoordinated water molecules are located between the chains with strong O-H···O hydrogen bonding interactions (Figs. 5 and S4).

## 3.3. Thermography analyses of complexes 1-4

The thermal stabilities of these complexes were studied by thermogravimetric analyses (TGA) under atmosphere (Fig. S5). The TGA results of the 1 and 2 indicate that the thermal decomposition of 1 and 2 is initiated at 107 and 112 °C, respectively. The two stages of weight loss of 1 and 2 correspond to that of py and NIPH ligands. The residue products weighted 15.54% and 17.19% are metal oxides (ZnO and MnO<sub>2</sub>, respectively), which is in agreement with the value calculated (15.90% and 17.34%) for 1 and 2, respectively. The TGA of the 3 shows three stages of weight loss, the first 3.78% up to 72 °C, which is ascribed to the loss of water molecules (calculated value 3.44%), and the other two steps are equal to the loss of py and NIPH ligands. The residue product weighted of 14.83% is metal oxides (Co<sub>2</sub>O<sub>3</sub>, calculated value of 15.85%). For **4**, TGA curve shows three significant weight loses, first weight loss of 7.22% from 86 to 252 °C is in accordance with the loss of free and coordinated water molecule (calcd 7.30%). The the other two are equal to the loss of im and NIPH ligands. The residue product weighted of 27.86% is metal oxides (CdO, calculated value of 26.01%).

### 4. Conclusions

In summary, we have successfully synthesized four coordination polymers based on infinite one-dimensional chains, formulated as  $[Zn(NIPH)(py)_3]_n$  (1),  $[Mn(NIPH)(-py)_3]_n$  (2),  $\{[Co(NIPH)(py)_3] \cdot (H_2O)\}_n$  (3), and  $\{[Cd(NI-PH)(im)_2(H_2O)] \cdot (H_2O)\}_n$  (4). Each NIPH ligand links two metal centers and each metal center connects two NIPH ligands to form the one-dimensional zig-zag chain structure. All of these chain-like structures are finally packed into three-dimensional supramolecular networks through hydrogen bonds. It is indicative that the hydrogen bonds can enhance the thermal stability of the coordination polymers.

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#### Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molstruc. 2007.03.005.

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