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# Hydrogen bonded supra-molecular framework in inorganic–organic hybrid compounds of Mn(II) and Zn(II): Syntheses, structures, and photoluminescent studies

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### HIGHLIGHTS

• Two compounds have been synthesized and characterized.

• Weak interactions provide additional assembly forces. The fluorescence spectrum of the complexes are investigated.

### G R A P H I C A L A B S T R A C T

The reaction of  $Mn(SO_4)_2 \cdot H_2O$ ,  $ZnCl_2$  with *N*-heterocyclic ligands and poly-carboxylate ligands yielded two novel coordination polymers. All compounds were characterized by elemental analysis, FT-IR, thermogravimetric analysis and X-ray crystallography. Furthermore, we studied the solid-state and solvent fluorescence spectrum, as well as the ligands.

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### ABSTRACT

In our efforts to tune the structures of compounds by selection of polycarboxylic acid ligands and *N*-heterocyclic ligands, two novel unreported compounds  $[Mn(cipt)(m-BDC)\cdotH_2O]_n(1)$  and  $[Zn(mip)(NDC)]_n(2)$ were obtained by hydrothermal reaction, where cipt = 2-(3-chlorophenyl)-1H-imidazo[4,5-f][1,10]phenanthroline, m-BDC = isophthalic acid, mip = 2-(3-methoxyphenyl)-1H-imidazo[4,5-f][1,10]phenanthroline, and NDC = naphthalene-1,4-dicarboxylic acid. All compounds have been characterized by IR, elemental analyses, single crystal X-ray diffraction, and thermogravimetric analysis (TGA). Structural analyses show that compounds 1 and 2 possess mononuclear structures and exhibit 1D zigzag chain structure. The intermolecular O-H···O and N-H···O interactions extend the compounds into 2D sheet networks. There are H-bonds and  $\pi$ - $\pi$  interactions in the title compounds. Furthermore, the solid-state fluorescence spectrum of compounds 1 and 2 were also investigated.

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### 1. Introduction

Recently, the rational design and synthesis of new polymeric transition metal–organic frameworks (MOFs) have received intense interest and attentions for their potential applications as







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function materials as well as their structural diversity and intriguing variety of topologies [1-6]. These MOFs can be specially designed by the careful selection of metal centers with preferred coordination geometries. Selection of appropriate multidentate ligands to coordinate metal ions is a key strategy for construction. The selection of polycarboxylate anions is extremely important because changing the structures of the anions can control and adjust the structures of coordination polymers. Among poly-carboxylates, the best studied are dicarboxylates, tricarboxylates, and biphenyldicarboxylates. 2-(Pyridin-2-yl)pyridine, 4-(pyridin-4yl)pyridine, and 1,10-phenanthroline can act as terminal ligands and provide supramolecular interaction sites for molecular recognition [7–10]. So far, 1,10-phenanthroline (phen) has been widely used to build supra-molecular architectures for its excellent coordinating ability and large-conjugated system. However, far less attention has been given to their derivatives. In this paper, two important phen derivative possesses fruitful aromatic systems 2-(3-chlorophenyl)-1H-imidazo[4,5have been prepared: f][1,10]phenanthroline (cipt) and 2-(3-methoxyphenyl)-1H-imidazo[4,5-f][1,10]phenanthroline (mip) in view of their following characteristics: (1) they possess extended aromatic system, which potentially provide supramolecular interactions such as  $\pi - \pi$  interactions between the aryl rings to construct intriguing structures; (2) they have strong coordination with two nitrogen atoms; (3) they possess rigidity of coordination to metal atoms. Nowadays the interest in Zn(II) compounds comes from their diverse applications especially in photochemistry due to their luminescent properties [11,12]. In this paper, we synthesized two polymers containing Mn<sup>2+</sup> and Zn<sup>2+</sup> ions and reported the syntheses, crystal structures, thermogravimetric analysis (TGA), as well as photoluminescence properties of coordination polymers 1-2.

### 2. Experimental section

### 2.1. Materials and physical measurements

The ligands cipt and mip were prepared according to the description in the literature procedures [13]. All the other chemicals from commercial sources were commercially available, and used without further purification. The FT-IR spectrum was measured with KBr pellets in the range of 4000–400 cm<sup>-1</sup> on a Perkin–Elmer 240C spectrometer. TGA was performed using a Perkin–Elmer TG-7 analyzer at the rate of 10 °C/min rise of temperature in nitrogen atmosphere. Crystal structures were determined on a Bruker SMART APEX II CCD X-ray diffractometer. Carbon, hydrogen and nitrogen elemental analyses were performed with a PE-2400 elemental analyzer. Fluorescence spectra were recorded on a FLSP 920 Edinburgh fluorescence spectrometer.

### 2.2. Syntheses

[Mn(cipt)(m-BDC)·H<sub>2</sub>O]<sub>n</sub> (**1**): A mixture of cipt (0.100 g, 0.3 mmol), Mn(SO<sub>4</sub>)<sub>2</sub>·H<sub>2</sub>O (0.051 g, 0.3 mmol), isophthalic acid (m-BDC) (0.100 g, 0.6 mmol) in distilled H<sub>2</sub>O (18 mL) was stirred at room temperature and adjusted the pH value to 6.0 with NaOH. The cloudy solution were put into a 30-mL Teflon-lined stainless vessel at 170 °C for 3 days and afterwards cooled to room temperature at a rate of 5 °C/h. The yellow crystals of compound **1** were obtained in 81% yield based on  $Mn.C_{54}H_{31}Cl_2Mn_2N_8O_{10}$ : calcd. C 57.26%, H 2.75%, N 9.89%; found: C 57.35%, H 2.89%, N 9.81%.

 $[Zn(mip)(NDC)]_n$  (2): Compound 2 was synthesized by a procedure similar to that used for 1 except for some syntheses condition. The mixture of mip,  $ZnCl_2$ , and NDC was adjusted the pH value to 8.5 with NaOH, and the cloudy solution were put into a 30-mL Teflon-lined stainless vessel at 180 °C for 3 days and afterwards cooled to room temperature at a rate of 5 °C/h. Compound **2** were collected in 76% yield based on Zn.  $C_{32}H_{20}N_4O_5Zn$ : calcd. C 63.43% H 3.33%, N 9.25%; found: C 63.52%, H 3.36%, N 9.41%.

The synthesis parameters play an important role in the formation of fascinating new compounds. However, a detailed understanding of the role that these variables play in the synthesis of hybrid inorganic–organic systems is, to date, a challenging work in this field. The differences in the synthetic condition in **1** and **2** are the pH value and temperature of the reactions. We found that higher pH value and temperature will favor the coordination polymer **2** produced, and it is clear that the pH value and temperature of the reaction play an important role in controlling the structures of MOFs.

### 2.3. X-ray crystallography

Single-crystal X-ray diffraction data for compounds **1** and **2** were collected at 293(2) K with a Bruker SMART APEX II CCD diffractometer equipped with a graphite-monochromatized Mo K $\alpha$  radiation ( $\lambda = 0.71073$  A) in the range of  $1.72 \le \theta \le 26.06^{\circ}$  for **1** and  $2.10 \le \theta \le 26.09^{\circ}$  for **2**. Absorption corrections were applied using multi-scan technique and all the structures were solved by direct methods and refined by full-matrix least-squares based on  $F^2$  using the programs SHELXS-97 [14] and SHELXL-97 [15]. Non-hydrogen atoms were refined with anisotropic temperature parameters and all hydrogen atoms were refined isotropically. Experimental details for crystallographic data and structure refinement parameters for compounds **1** and **2** are listed in Table 1.

### 3. Results and discussion

### 3.1. Structural analysis of compound 1

The molecular structure is shown in Fig. 1. The 1D zigzag chain structure is suggested in Fig. 2, and 2D sheet structure is shown in Fig. 3. Selected bond lengths and bond angles are given in Table 2. Single-crystal X-ray structural analysis reveals that the asymmetric

Crystal data and details of structure refinement parameters for 1 and 2.

Compound	1	2
Empirical formula	C <sub>54</sub> H <sub>32</sub> Cl <sub>2</sub> Mn <sub>2</sub> N <sub>8</sub> O <sub>10</sub>	$C_{32}H_{20}N_4O_5Zn$
Formula weight	1133.66	605.89
Crystal system	monoclinic	monoclinic
space group	C2/c	p2(1)/n
a (nm)	1.4124(6)	1.27411(8)
b (nm)	2.3311(1)	1.49255(9)
c (nm)	1.5703(1)	1.35881(8)
$\beta$ (°)	102.655(6)	90.989(1)
Volume (nm <sup>3</sup> )	5.045(5)	2.5836(3)
Z Density (Mg/m <sup>3</sup> ) (calculated) Absorption coefficient (mm <sup>-1</sup> )	4 1.493 0.675	4 1.558 1.003
(init) 7	2304	1240
F(000)	0.318 × 0.115 × 0.091	0.272 × 0.177 × 0.160
Crystal size (mm3)	1.7226.06	2.1026.09
Theta range (°)	13808	14000
Reflections collected	4976 [0.0608]	5107 [0.0502]
Unique reflections [ $R_{int}$ ]	0.983	1.002
Goodness-of-fit on $F^2$	R1 = 0.0707	<i>R</i> 1 = 0.0456
Final $R$ indices [ $I > 2\sigma(I)$ ]	wR2 = 0.1827	<i>wR</i> 2 = 0.1001
R indices (all data)	R1 = 0.1191	<i>R</i> 1 = 0.0855
Largest difference peak and hole (e. Å <sup>-3</sup> )	wR2 = 0.2134 2.202, -0.545	wR2 = 0.814 3.487, -0.291



Fig. 1. The molecular structure of compound 1 (hydrogen atoms were omitted).

unit of compound **1** crystallizes in monoclinic, space group *C*2/*c*. Each Mn atom is coordinated by one m-BDC acid, one cipt molecule, and one water molecule (Fig. 1). Mn(II) metal exhibits penta-, six-, or seven-coordinated mode [16–19]. The Mn(II) atom in compound **1** shows a hexa-coordinated arrangement, which arises from three oxygen atoms [Mn–Ocarboxylic = 2.082(4)–2.348(4)Å, Mn–Owater = 2.135(4)Å] from two distinct m-BDC ligands, one

coordinated water molecule, and two nitrogen atoms donors [Mn-N = 2.265(4)-2.272(4) Å] from one chelating cipt ligand, forming a distorted octahedral geometry. The angle of O(2)-Mn(1)-O(1), O(1)-Mn(1)-N(1), N(1)-Mn(1)-O(1W) and O(2)-Mn(1)-O(1W) is 56.84°, 100.34°, 113.34° and 90.95°, and the sum is 361.47°. For the coordination environment of Mn(1), the Mn1, O(1W), O(1), N(1), O(2) atoms define the basal plane, and the N(2) and O(3) atoms occupy the apical axial positions. The equation of a plane is 10.1820(0.0141)x + 2.9168(0.0293)y + 7.9639(0.0175)z = 4.0545(0.0068). The deviation of atoms Mn1, O(1W), O(1), N(1), O(2), N(2) and O(3) to the plane is -0.1226, -0.1282, -0.2429, 0.1913, 0.3024, 2.1083 and -2.1488 Å, respectively. The distances of Mn-O/N bond lengths are all consistent with corresponding bond lengths found in the literature [20-25]. The m-BDC acid bridges two adjacent Mn centers to form a 1D zigzag chain structure in two different coordination modes: one m-BDC acid bridges two adjacent Mn centers in mono-bridging coordination mode with distance of Mn...Mn is 11.0335 Å, and one m-BDC acid bridges two adjacent Mn centers in bidentate chelating coordination mode with distance of Mn ... Mn is 9.4487 Å (Fig. 2). Two different m-BDC ligands arranged almost perpendicular to each other while coordinating to metal center. Hydrogen bonding interactions are usually important in the synthesis of supramolecular architectures. There are three types of H-bonds interactions in compound 1: O-H···O, N-H···O and C-H···O interactions. The most interesting aspect of the structure in 1 concerns 0-H· · · 0 the intermolecular  $[H(1 WA) \cdots O(4) = 1.800 Å,$ 



Fig. 2. 1D zigzag chain structure of compound 1 (hydrogen atoms were omitted).



Fig. 3. 2D layer structure of compound 1 linked by O-H...O hydrogen bonds (dotted lines represent hydrogen bonds).

### Table 2

Selected bond lengths	[Å]	and bo	ond angles	[°	for	compound	1
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Bond	Dist.	Bond	Dist.
Mn(1)-O(1)	2.263(4)	Mn(1)-O(2)	2.348(4)
Mn(1)-O(3)	2.082(4)	Mn(1)-O(1W)	2.135(4)
Mn(1)-N(1)	2.272(4)	Mn(1)-N(2)	2.265(4)
Angle	(°)	Angle	(°)
O(3)-Mn(1)-O(1)	91.38(17)	O(3)-Mn(1)-O(1W)	93.14(16)
O(1W) - Mn(1) - O(1)	146.27(13)	O(1)-Mn(1)-O(2)	56.84(12)
O(1W)-Mn(1)-O(2)	90.95(13)	O(3)-Mn(1)-O(2)	113.27(15)
O(3)-Mn(1)-N(2)	156.73(16)	O(1W) - Mn(1) - N(2)	91.10(15)
O(1)-Mn(1)-N(2)	97.70(15)	O(3)-Mn(1)-N(1)	84.76(15)
O(1W) - Mn(1) - N(1)	113.34(14)	O(1)-Mn(1)-N(1)	100.34(14)
N(2)-Mn(1)-O(2)	89.51(14)	N(1)-Mn(1)-O(2)	149.47(14)
N(2)-Mn(1)-N(1)	72.57(14)		



Fig. 4. The molecular structure of compound 2 (hydrogen atoms were omitted).

O(1W)···O(4) = 2.615 Å and O(1W)–H(1WA)···O(4) = 140°] interactions, which help in the construction of the 2D layer structure (Fig. 3). Moreover, there are  $\pi$ - $\pi$  interactions between the aryl ring of cipt ligands in coordination polymer **1** with distance of cg(1) cg(2) ring centroid is 3.614 Å. Cg(1): C1–C2–C3–C4–C11–N1, Cg(2): C4–C5–C6–C7–C11–C12.

### 3.2. Structural analysis of compound 2

The molecular structure is shown in Fig. 4, and the 1D zigzag chain structure is shown in Fig. 5. The 2D layer structure is shown in Fig. 6. Selected bond lengths and bond angles are listed in Table 3. As shown in Fig. 4, the asymmetric unit of compound **2** 

consists of one Zn(II) atom, one mip ligand, and one NDC ligand. Zn(II) metal exhibits four-, penta-, six- mode [26–29]. The Zn(II) atom is four-coordinated with two nitrogen atoms (N(1), N(2))from one chelating mip ligand and two oxygen atoms (O(2)), O(4)) from two different monodentate bridging NDC ligands, forming a distorted trigonal pyramidal geometry. The N(O)–Zn–O(N) angles range are from 113.12(10)° to 123.08(11)°. The bond distances of Zn–O in compound **2** are from 1.958(3) to 1.987(2) Å, and those of Zn-N bond distances fall in the 2.070(3) to 2.071(3) Å range, which are similar with the values reported [30– 32,11,33]. The average bond of Zn–O is 1.973 Å, smaller than the average bonds of Zn-N (2.071 Å). Each Zn(II) atom is linked to another two Zn(II) atoms by bis-bridging NDC ligands, and this lead to the formation of a 1D zigzag chain network with the distances of Zn...Zn are 10.6168 Å (Fig. 5). The *N*-heterocyclic ligands mip are attached to both sides of this zigzag chain regularly, and mip ligands on the same sides are parallel with the angular separation of the zigzag chain are 89.323°. The most interesting aspect of the structure in **2** concerns the intermolecular N-H···O  $[H(3A) \cdots O(2) = 2.090 \text{ Å}, N(3) \cdots O(2) = 2.934 \text{ Å}$ and N(3) - $H(3A) \cdots O(2) = 167^{\circ}$  interactions, which help in the construction of the 2D layer structure (Fig. 6). Moreover, there are  $\pi$ - $\pi$  interactions between the aryl ring of mip ligands in coordination polymer 2 with distance of cg(1) cg(2) ring centroid is 3.619 Å. Cg(1): C14– C15-C16-C17-C18-C19, Cg(2): C4-C5-C6-C7-C11-C12.

Dicarboxylates are widely used in the assembly of supra-molecular architectures because of their diverse coordination modes and bridging ability. For example, the BDC ligand can function in chelating bis-bidentate, bis-monodentate, mono-bidentate, bridging bis-bidentate, monodentate-bidentate or chelating/bridging bisbidentate modes [26]. In the title compounds, we can see the final structures are always decided by the diversity of coordination modes of dicarboxylates ligands, and coordination modes (chelating bis-bidentate or bis-monodentate) usually raise the 1D chain or 1D double-chain structure.

### 3.3. IR spectra

The FT-IR spectra absorption peaks at  $3214 \text{ cm}^{-1}$  for **1**, and 3132,  $3206 \text{ cm}^{-1}$  for **2** correspond to the C–H stretching mode for the aromatic rings. The infrared spectrum of the compound **1** exhibited absorption at  $3466 \text{ cm}^{-1}$ , which is assigned to O–H stretching vibration of the water molecules. It is a clear indication of the presence of water molecules. There are two peaks at 1625 and 1554 cm<sup>-1</sup>, which correspond to the antisymmetric stretching of carboxyl, and the absorption of 1398 cm<sup>-1</sup> corresponds to the



Fig. 5. The 1D zigzag chain of compound 2 (hydrogen atoms were omitted).



Fig. 6. The 2D layer structure of compound 2 by N-H···O hydrogen bonds (dotted lines represent hydrogen bonds).

 Table 3
 Selected bond lengths [Å] and bond angles [°] for compound 2.

Bond	Dist.	Bond	Dist.
Zn(1)-O(2)	1.987(2)	Zn(1)-O(4)	1.958(3)
Zn(1)-N(1)	2.070(3)	Zn(1)-N(2)	2.071(3)
Angle	(°)	Angle	(°)
O(4)-Zn(1)-O(2)	109.18(11)	O(4)-Zn(1)-N(1)	119.81(12)
O(2)-Zn(1)-N(1)	113.12(10)	O(4)-Zn(1)-N(2)	109.41(11)
O(2)-Zn(1)-N(2)	123.08(11)	N(1)-Zn(1)-N(2)	80.45(10)

symmetric stretching of carboxyl. The  $\Delta v (v_{as}(COO^{-})-v_{s}(COO^{-}))$  are 227 and 156 cm<sup>-1</sup>, indicates that the carboxyls are monodentately and bidentately coordinated with Mn(II) atoms. In **2**, the strong characteristic absorption peak at 1662 cm<sup>-1</sup> is attributed to the antisymmetric stretching vibration of the coordinated carboxyl groups, and the peak at 1397 cm<sup>-1</sup> corresponds to the symmetric stretching of carboxyl. The separation is 265 cm<sup>-1</sup>, which indicates that the carboxyls groups adopt monodentate coordination modes. The IR results are good agreement with their solid structural features from the results of their crystal structures.

### 3.4. Thermal properties

To examine the thermal stability of compounds 1 and 2, TG curves have been obtained from crystalline samples in the flowing nitrogen atmosphere at a heating rate of 10 °C/min (Fig. 7). In compound 1, First weight loss of 3.11% from 262 to 267 °C reveals the loss of the water molecule (calcd. 3.17%). The second weight loss of 27.50% from 267 to 501 °C corresponds to the loss of m-BDC ligands (calcd. 29.29%), and the last weight loss of 59.10% from 501 to 670 °C reveals the loss of the cipt ligands (calcd. 58.35%). Compound **2** exhibits good thermal stability and can stable before the temperature of 361 °C, and the first weight loss of 32.13% from 361 to 397 °C corresponds to the loss of NDC ligands (calcd. 35.68%). The last weight loss of 52.87% in the temperature range of 397-598 °C can be ascribed to the release of mip ligands (calcd. 53.86%). Compared with the similar compounds without H-bonds or  $\pi - \pi$  interactions [34–36,18], our compounds shows good thermal stability, and it is noteworthy that the existence of hydrogen



Fig. 7. TGA curves of compounds 1 and 2.

bonds and  $\pi - \pi$  interactions reinforce the structural thermal stability for MOFs.

### 3.5. Photoluminescent properties

Luminescence property is very important in photochemistry and photophysics. So in this study, we research the luminescence of compounds **1** and **2**, as well as the free ligands cipt and mip (Figs. 8 and 9). The free ligands exhibit emissions at 539 nm (excitation at 347 nm) for cipt, and at 541 nm for mip (excitation at 320 nm). Compound **1** shows one broad emission band with the maximum intensity at 612 nm upon excitation at 330 nm, which is red-shifted by 73 nm relative to the emission wavelength of free ligand cipt. Compound **2** shows one broad strong emission band with the maximum intensity at 619 nm upon excitation at 340 nm, which is red-shifted by 78 nm relative to the emission wavelength of free ligand mip. The energy of the luminescence suggests that the most possible assignment for the emissions of **1** originates from ligand-to-metal charge transfer (LMCT), and the emissions of **2** mainly originate from the intra-ligand fluorescent



Fig. 8. Luminescent spectrum of ligand cipt and compound 1 in solid state at room temperature.



Fig. 9. Luminescent spectrum of ligand mip and compound 2 in solid state at room temperature.



Fig. 10. Luminescent spectrum of compound 2 in different solvents.

emissions of ligands [37]. In order to explore the potential of compound **2** in terms of sensing small molecules, its luminescent properties in different solvent emulsions were investigated. The **2**solvent emulsions were prepared by introducing powder of **2** into 4 mL methanol, ethanol, and N, N-dimethylformamide (DMF), respectively. As shown in Fig. 10, the luminescent intensity of **2** is largely dependent on the solvent molecules, which exhibits different degrees of quenching effects in the solvents. Compounds **1** and **2** exhibit strong emissions, the reason may be attributed to the rigidity of *N*-heterocyclic ligands. The rigidity is favor of energy transfer and reduces the loss of energy through a radiationless pathway. The coordination polymers **1** and **2** may be good candidate for potential photoluminescence material because it is highly thermally stable and insoluble in water and common organic solvents.

### 4. Conclusions

In summary, we have reported two compounds formed by polycarboxylate and *N*-heterocyclic ligands. The polycarboxylates ligands function in mono-bridging and bidentate-chelating coordination modes to form 1D zigzag chain structure in compound **1**, and function in bis-bridging coordination modes to form a 1D zigzag chain network in compound **2**. It is noteworthy that non-covalent interactions ( $\pi \cdots \pi$  interactions, H-bond and coordination bonds) can be one of the most powerful force for instructing and directing the supra-molecular architectures. The results also present a feasible strategy for the construction of framework architectures by synthesis condition design, such as pH value, temperature, and so on. This material will give new impetus to the construction of novel functional material with potentially useful physical properties. Further studies are now under way in our laboratory.

### Supplementary material

CCDC 912838 and 910139 contain the supplementary crystallographic data for **1** and **2**, respectively. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data request.cif.

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