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Synthesis, crystal structures, and properties of three helical coordination polymers with quinolinecarboxylate ligand

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Three mixed-ligand complexes $[M(L)(Phen)(H_2O)]_n \cdot nH_2O$ [M = Ni (1), Co (2), Zn (3)], (L=2-methylquinoline-3,4-dicarboxylate dianion; phen = 1,10-phenanthroline) have been synthesized by the reaction of MCl₂ with 2-methylquinoline-3,4-dicarboxylate acid, sodium hydroxide, and 1,10-phenanthroline under hydrothermal conditions and were characterized by single-crystal X-ray diffraction. Complexes 1–3 crystallize isomorphously in the orthorhombic space group *P*bca and feature 1-D helical chains. These helical chains are linked to form a 2-D layer through π - π stacking interactions, which are further linked by hydrogen bonds to form a 3-D network. Solid-state properties such as thermal stability and fluorescence emission of the polymeric Zn complex 3 have also been investigated.

Keywords: Crystal structure; Coordination polymers; Helical chains; Fluorescent properties; Quinolinecarboxylate acid

1. Introduction

There has been intense research on metal–organic framework structures (MOFs) [1, 2]. The rapid development of MOFs is due to their intriguing structural topologies and properties, such as adsorption, ion-exchange, non-linear optical (NLO) materials, and heterogeneous catalysis [3–7]. A particularly interesting and challenging area is the synthesis of helical coordination polymers and exploration of their potential utility in asymmetric catalysis and non-linear optics [8, 9]. The synthesis of such species is often based on self-assembly of suitable building blocks to give supramolecular networks constructed by coordination and/or hydrogen bonds or other weak supramolecular interactions, such as π – π stacking interactions. Metal–organic helical structures containing single-, double-, and multi-stranded helices [10–12] have been constructed; the chemistry of coordination polymers with helical structures has been reviewed [13].

The pyridinecarboxylic acid unit is widely used in the synthesis of coordination polymers [14, 15]. In contrast, research on metal quinolinecarboxylate systems is

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limited [16]. 2-Methylquinoline-3,4-dicarboxylate, which is the close analog of pyridinecarboxylic acid, presents different functional groups and a large conjugated π -system, allowing for diversity in the coordination mode. Chelating bipyridine-like ligands such as 1,10-phenanthroline and 2,2'-bipyridine are important in maintaining the one dimensionality of the coordination polymers and may provide supramolecular recognition sites for π - π aromatic stacking interactions to form interesting supramolecular structures, such as double-stranded helices and molecular zippers [17, 18]. Many helical architectures based on bipy-like and carboxylate ligands have been reported [19].

As a continuation of our research, we report herein three, 1-D coordination polymers with 2-methylquinoline-3,4-dicarboxylate (L), including $[Ni(L)(Phen)(H_2O)]_n \cdot nH_2O$ (1), $[Co(L)(Phen)(H_2O)]_n \cdot nH_2O$ (2), and $[Zn(L)(Phen)(H_2O)]_n \cdot nH_2O$ (3). Thermal stability and fluorescence emission of 3 has also been explored.

2. Experimental

2.1. Materials and analyses

All reagents and solvents were commercial materials of analytical grade and used without purification, except that 2-methylquinoline-3,4-dicarboxylate was synthesized according to the reported procedure [20]. Elemental analyses for C, H, and N were carried out on a Perkin-Elmer 2400 II elemental analyzer. Infrared spectra on KBr pellets were performed on a BRUKER EQUINOX-55 spectrometer from 4000 to 400 cm⁻¹. Diamond TG/DTA thermal analyzer was used to record simultaneous TG and DTG curves in static air at a heating rate of 10 K min⁻¹ from 25°C to 1000°C using platinum crucibles. Fluorescence spectra were recorded on Perkin-Elmer instruments LS55 Luminescence Spectrometer.

2.2. Syntheses of the complexes

2.2.1. Synthesis of $[Ni(L)(Phen)(H_2O)]_n \cdot nH_2O$ (1). A solution of phen (0.1982 g, 1 mmol) in 10 mL methanol was added dropwise with stirring at room temperature to a solution of NiCl₂ · 6H₂O (0.2377 g, 1 mmol), NaOH (0.0800 g, 2 mmol), and 2-methylquinoline-3,4-dicarboxylate (0.2312 g, 1 mmol) in 5 mL water. The resulting mixture was sealed in a 23 mL Teflon-lined stainless reactor, kept under autogenous pressure at 120°C for 5 days, and then slowly cooled to room temperature at a rate of 5°Ch⁻¹. Green block crystals suitable for X-ray diffraction were isolated directly, washed with ethanol, and dried in air (Yield: 68%, based on Ni). Anal. Calcd for C₂₄H₁₉N₃O₆Ni (%): C, 57.18; H, 3.80; N, 8.34. Found: C, 57.32; H, 3.91; N, 8.32. IR data (KBr pellets, cm⁻¹): 3510–3100w ν (O–H), 1589s ν (C=O).

2.2.2. Synthesis of $[Co(L)(Phen)(H_2O)]_n \cdot nH_2O$ (2). The same synthetic procedure as for 1 was used except that NiCl₂ · 6H₂O was replaced by CoCl₂ · 6H₂O (0.2379 g, 1 mmol). Red block crystals of 2 were obtained in 54% yield based on Co. Anal. Calcd for C₂₄H₁₉N₃O₆Co (%): C, 57.15; H, 3.80; N, 8.33. Found: C, 57.20; H, 3.89; N, 8.30. IR data (KBr pellets, cm⁻¹): 3510–3100w ν (O–H), 1585s ν (C=O).

Complex	1	2	3
Empirical formula	C24H19N3O6Ni	C ₂₄ H ₁₉ N ₃ O ₆ Co	C24H19N3O6Zn
Formula weight	504.11	504.35	510.81
Crystal system	Orthorhombic	Orthorhombic	Orthorhombic
Space group	Pbca	Pbca	Pbca
Unit cell dimensions (Å)			
a	19.64(2)	19.882(2)	19.949(6)
b	9.115(9)	9.133(1)	9.190(3)
С	23.50(2)	23.539(3)	23.493(7)
V	4207(7)	4274.3(9)	4307(2)
Volume (Z)	8	8	8
Calculated density $(g cm^{-3})$	1.592	1.568	1.576
Absorption coefficient (mm^{-1})	0.972	0.852	1.189
F(000)	2080	2072	2096
Reflections collected, Independent	22960/4133	24328/4199	24145/4229
Max. and min. transmission	0.8219/0.7725	0.8549/0.8347	0.7883/0.7474
Data/restraints/parameters	4133/0/308	4199/0/308	4229/0/308
Goodness-of-fit on F^2	0.962	0.955	1.035
Final R indices $[I > 2\sigma(I)]$	$R_1 = 0.0429,$	$R_1 = 0.0446,$	$R_1 = 0.0418$,
	$wR_2 = 0.1228$	$wR_2 = 0.1230$	$wR_2 = 0.1142$
R indices (all data)	$R_1 = 0.0673,$	$R_1 = 0.0862,$	$R_1 = 0.0645$,
	$wR_1 = 0.1318$	$wR_2 = 0.1378$	$wR_2 = 0.1260$

Table 1. Crystal data and structure refinement of 1-3.

2.2.3. Synthesis of $[Zn(L)(Phen)(H_2O)]_n \cdot nH_2O$ (3). The same synthetic procedure as for 1 was used except that NiCl₂ · 6H₂O was replaced by ZnCl₂ (0.1363 g, 1 mmol). Light yellow block crystals of 3 were obtained in 72% yield based on Zn. Anal. Calcd for C₂₄H₁₉N₃O₆Zn (%): C, 56.43; H, 3.75; N, 8.23. Found: C, 56.40; H, 3.80; N, 8.29. IR data (KBr pellets, cm⁻¹): 3510–3100w ν (O–H), 1572 ν (C=O).

2.3. X-ray diffraction data collection and refinement

Suitable single crystals of 1–3 were mounted on a Bruker SMART CCD diffractometer equipped with graphite-monochromated Mo-K α radiation ($\lambda = 0.71073$ Å) at 298 K. All absorption corrections were performed with SADABS [21]. The structures were solved by direct methods and refined with full-matrix least-squares and expanded using Fourier techniques. Anisotropic thermal parameters were assigned to all non-hydrogen atoms. Hydrogens were fixed at calculated positions and refined by using a riding mode. All calculations were performed using SHELXTL-97 [22]. The crystallographic data are summarized in table 1 and selected bond distances and angles for 1–3 are listed in table 2. Crystal drawings were produced by Diamond soft.

3. Result and discussion

3.1. Crystal structures of 1–3

Single-crystal X-ray diffraction on 1–3 reveal that they are extremely similar in structure with the formula $[M(L)(Phen)(H_2O)]_n \cdot nH_2O$ [M = Ni(1), Co(2), Zn(3)]. All the

1			
Ni1–O3	2.113(3)	Nil-O4	2.135(3)
Ni1–O5	2.039(3)	Nil-Nl	2.054(3)
Ni1–N2	2.066(3)	Nil-O1A	2.051(3)
O3-Ni1-O4	62.08(10)	N1-Ni1-O1A	93.40(11)
O3-Ni1-O5	89.45(11)	N2-Ni1-O1A	93.89(11)
O4-Ni1-O5	86.09(10)	O5–Ni1–O1A	90.80(11)
N1-Ni1-N2	80.45(13)	O3–Ni1–O1A	100.42(10)
N2-Ni1-O3	93.09(12)	N1-Ni1-O4	104.34(10)
N2–Ni1–O4	90.47(11)	N1–Ni1–O5	95.84(12)
2			
Co1–O3	2.145(2)	Col-O4	2.193(2)
Co1–O5	2.075(2)	Co1-N1	2.115(3)
Co1-N2	2.129(3)	Col-OlA	2.064(2)
O3-Co1-O4	60.64(9)	N1-Co1-O1A	94.65(10)
O3–Co1–O5	91.55(10)	N2-Co1-O1A	95.90(10)
O4–Co1–O5	86.31(10)	O5–Co1–O1A	89.93(10)
N1-Co1-N2	78.21(11)	O3–Co1–O1A	100.29(9)
N2-Co1-O3	93.43(10)	N1-Co1-O4	104.86(10)
N2-Co1-O4	90.17(10)	N1-Co1-O5	95.25(11)
3			
Zn1–O3	2.157(2)	Zn1–O4	2.260(3)
Zn1–O5	2.085(2)	Zn1–N1	2.123(3)
Zn1-N2	2.154(3)	Zn1–O1A	2.041(2)
O3-Zn1-O4	59.66(8)	N1–Zn1–O1A	98.05(10)
O3–Zn1–O5	92.82(10)	N2–Zn1–O1A	97.18(10)
O4–Zn1–O5	85.31(9)	O5–Zn1–O1A	90.75(9)
N1-Zn1-N2	78.23(11)	O3–Zn1–O1A	99.40(9)
N2-Zn1-O3	92.23(10)	N1–Zn1–O4	103.47(9)
N2-Zn1-O4	89.58(10)	N1–Zn1–O5	94.34(11)

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

Symmetry code: A = 2 - x, 0.5 + y, 0.5 - z.

complexes consist of 1-D helical chains formed by distorted MN_2O_4 octahedra linked by the dicarboxylic acid. Therefore, the structure of **3** is described in detail to represent their frameworks.

A single crystal X-ray diffraction study shows **3** crystallizes in the orthorhombic *Pbca* space group. As shown in figure 1, each six-coordinate Zn(II) is in a slightly distorted octahedral geometry, defined by two nitrogens (N1, N2) from one chelating phen, three oxygens (O1, O3, and O4) from carboxylate of two different L, and O5 from H₂O. The Zn–O(N) bond distances are in the range 2.041–2.260 Å, with an average value of 2.136 Å. Each quinolinecarboxylate links two zincs, giving a half turn unit of the helical 1-D chain (figure 2). The neighboring Zn–Zn distances are 5.619 Å and the pitches of the helical chains are 9.190 Å. Face-to-face $\pi \cdots \pi$ stacking between two well-overlapping phen molecules from two adjacent chains cause the 2-D supramolecular network [23]. The two adjacent phen molecules from different helical chains are almost parallel (dihedral angle 0.428°). The interchain π - π interactions between the two adjacent phen molecules of two adjacent helical chains are at very short distances (3.530 and 3.531 Å) (figure 3). Coordination water molecules (O5) are situated within a tube-like helix through O5-H5C···O2 and O5-H5D···O3 hydrogen bonding. Lattice water molecule (O6) is stabilized by forming hydrogen bonds (table 3) between



Figure 1. Molecular structure of **3** with 30% thermal ellipsoids. All hydrogens are omitted for clarity. Symmetry codes: A = 2 - x, 0.5 + y, 0.5 - z.

different helical chains. These weak interactions $(O6-H6C\cdots O2 = 2.900(4) \text{ Å}, O6-H6D\cdots N3 = 3.002(5) \text{ Å})$ play a vital role in determining the crystal packing and construction of the extended 3-D supramolecular network.

3.2. Vibrational spectroscopy

IR spectra of all three complexes are similar, indicating similar structures. The strong and broad absorptions at $3510-3100 \text{ cm}^{-1}$ are attributed to symmetric O–H stretch of lattice and/or coordinated water in the crystal. The IR spectrum of free ligand shows strong bands at 1712 cm^{-1} , which can be assigned as ν (C=O). In the complexes, the band shifted by $123-140 \text{ cm}^{-1}$ for ν (C=O). The shift suggests that the relevant oxygen of the ligand coordinates to metal.

3.3. Photoluminescence properties

Coordination polymers of d^{10} metal ions and conjugated ligands are potential hybrid photoactive materials due to their thermal stability and photoluminescence properties.



Figure 2. View of the helices in 3: one is left-handed (left) and the other right-handed (right). Unnecessary atoms are omitted for clarity.

Solid-state emission spectrum of **3** at room temperature is shown in figure 4. The fluorescence spectrum shows that **3** exhibits a broad and strong emission band with a maximum wavelength of 414 nm upon photoexcitation at 347 nm. In addition, free L exhibits a broad and strong emission band with a maximum wavelength of 390 nm when excited with light of 348 nm, assigned to intraligand π - π * charge transfer. Further, the free phen generally displays a weak luminescence at *ca* 530 and 365 nm [24] in the solid state at room temperature. Hence, the emission of **3** can be tentatively attributed to coordination of **L**. The red-shift of the emission energy from the free ligand to the complex may be related to the inter-planar π - π stacking interactions, which result in decrease in HOMO-LUMO energy gap of the complex [25].

3.4. Thermal analysis

To investigate thermal stabilities, TG and DTG of **3** were examined at a heating rate of 10° C min⁻¹ from 25°C to 1000°C under N₂. As shown in figure 5, the TGA indicates that **3** loses 7.7% of total weight from 25°C to 216°C (DTG_{max} = 209°C), corresponding to the removal of the lattice and coordinated water molecules. Good agreement between experimental and calculated values was observed for the weight loss (Calcd 7.05%). The anhydrous complex is stable to 240°C. The products lose 36.2% of the total weight



Figure 3. View of the π - π intramolecular stacking interaction in 3. All hydrogens are omitted for clarity.

Donor– $H \cdots$ Acceptor	D–H	$H \cdots A$	$D \cdots A$	$D\!\!-\!\!H\cdots A$
O5–H5C · · · O2a	0.85	1.77	2.614(4)	174.1
O5–H5D···O3b	0.85	2.01	2.853(3)	174.5
O6–H6C···O2c	0.85	2.06	2.900(4)	170.8
O6–H6D····N3d	0.85	2.16	3.002(5)	171.0

Table 3. Distances of the hydrogen bond interactions (Å) for 3.

Symmetry code: a = 2 - x, 0.5 + y, 0.5 - z; b = 2 - x, -0.5 + y, 0.5 - z; c = x, 1 + y, z; d = 2 - x, 1 - y, -z.

from 240°C to 470°C, which is related to the endothermic decomposition of phen (Calcd 35.3%; $DTG_{max} = 265^{\circ}C$, 286°C, 314°C). Upon temperature increase, the quinolinecarboxylate decomposes gradually, a series of complicated and consecutive weight losses occur.



Figure 4. Room-temperature solid-state emission spectrum of L and 3.



Figure 5. The TG-DTG curves of 3.

4. Conclusion

Three helical coordination polymers $[\mathbf{M}(\mathbf{L})(\text{Phen})(\text{H}_2\text{O})]_n \cdot n\text{H}_2\text{O}$ $[\mathbf{M} = \text{Ni}$ (1), Co (2), Zn (3)] were synthesized under hydrothermal conditions. Theoretically, the rigid 2methylquinoline-3,4-dicarboxylate building blocks can be considered as T-shaped connectors to construct high-dimensional coordination polymers. Nevertheless, it seems not the case here. In fact, the larger conjugated π -systems as well as the steric hindrance of benzene rings and the methyl group weaken the coordination abilities of the quinoline N donor, leading to 1-D helical architectures.

The preparation of 1–3 shows that π – π stacking interactions and hydrogen bonds play important roles in affecting the final structure of the coordination polymers and provides a valuable approach for the construction of other helical complexes containing 2,2'-bipyridyl-like and carboxylate ligands.

Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre as Supplementary Publication Nos CCDC 773537–773539. Copies of the data can be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB21EZ, UK (Fax: +44 1223 336 033; E-mail: deposit@ccdc.cam.ac.uk).

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