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Synthesis, crystal structure and properties of cobalt(II) and nickel(II) coordination polymers supported by functionalized dicarboxylate ligands

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ABSTRACT

Four new coordination polymers of cobalt(II) and nickel(II) with functionalized dicarboxylate ligands, namely, $[Co^{II}L^1(2,2'-bpy)(H_2O)]$ (1), $[Ni^{II}L^1(2,2'-bpy)(H_2O)] \cdot H_2O$ (2), $[Co^{II}_2(L^2)_2(2,2'-bpy)_2(H_2O)]$ (3) and $[Ni^{II}_2(L^2)_2(2,2'-bpy)_2(H_2O)]$ (4), where $H_2L^1 = 2,5$ -dibenzoylterephthalic acid, $H_2L^2 = 4,6$ -bis(4-methylbenzoyl) isophthalic acid and 2,2'-bpy = 2,2'-bipyridine, were synthesized and characterized by elemental analysis, IR spectra and thermogravimetric analysis. Complex 1 exhibits a zigzag chain with a C-H··· π interaction between the phenyl ring proton and the phenyl ring of an adjacent chains to form a 2D supramolecular sheet. Complex 2 contains two helical chains which extend into 2D via a C-H··· π interaction between the pyridine ring. Complexes 3 and 4 are isomorphous with helical chains that extend in the same direction and further link to one another by supramolecular forces into a 2D structure. Moreover, magnetic and luminescence properties have been investigated for 1 and 2, respectively.

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1. Introduction

The design and synthesis of metal–organic frameworks (MOFs) are continuously attractive among coordination and material chemistry, not only due to the frequently novel motifs of their architectures but also their potential applications in gas storage, chemical separations, ion exchange, non-linear optics, magnetism and heterogeneous catalysis [1–17]. A great variety of interesting architectures have been reported including helix, brick wall, honeycomb, layer, ladder, grids, boxes and herringbone [18–26], of which helical coordination polymers are particularly attractive because of their fascinating structures and applications in many fields [27–34]. Although discrete helices and low-dimensional helical coordination polymers are now abundant, the assembly of such helical units into higher order networks is still a challenge.

In an attempt to construct helical frameworks, we choose functionalized dicarboxylate ligands to give birth to new complexes. So four new coordination polymers of Co(II) and Ni(II) were obtained by using benzoyl-substituted terephthalic acid (H_2L^1) and methylbenzoyl-substituted isophthalic acid (H_2L^2) (Chart 1) as ligands. The choice of these ligands was based on the following considerations: (1) Each ligand has six oxygen atoms potentially available for metal coordination; (2) the aromatic substituent may rotate around the central phenyl ring. This flexibility may be significant in realizing interesting structures; (3) the extended π -system of the substituted ligands, which are derived from terephthalic and isophthalic acid, may be useful for luminescent and/or magnetic properties of coordination polymers. In addition, in this work we also introduce 2,2'-bipyridine as an auxiliary ligand to control the structure and dimensionality of the resulting coordination polymers [35–37]. The synthesis and structural characterization of four new coordination polymers $[Co^{II}L^1(2,2'-bpy)(H_2O)]$ (1), $[Ni^{II}L^1(2,2'-bpy)(H_2O)] \cdot H_2O$ (2), $[Co^{II}_2(L^2)_2(2,2'-bpy)_2(H_2O)]$ (3) and $[Ni^{II}_2(L^2)_2(2,2'-bpy)_2(H_2O)]$ (4) are reported here. Furthermore, interesting luminescence and magnetic properties have been studied for complexes 1 and 2, respectively.

2. Experimental

2.1. Materials and physical measurements

Reagents were purchased commercially and were used without further purification. The ligands H_2L^1 and H_2L^2 were synthesized according to the literature method [38]. The FI-IR spectra were recorded using KBr pellets in the range 4000–400 cm⁻¹ on a Mattson Alpha-Centauri spectrometer. Elemental analyses for C, H and N were performed on a Perkin–Elmer 2400 Elemental Analyzer. The thermal behaviors were studied by TGA on a Perkin–Elmer Thermal Analyzer under N₂ with a heating rate of 5 °C/min. The fluorescence spectrum for **2** was obtained using a Cary Eclipse luminescence spectrophotometer (VARIAN. USA). The temperature-dependent magnetic susceptibility of **1** was measured with crystalline samples on a Quantum Design MPMS XL-5 Squid magnetometer in a magnetic field of 1000 Oe under the temperature range 2–300 K.





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2.2. Synthesis

2.2.1. Synthesis of $[Co^{II}L^{1}(2,2'-bpy)(H_{2}O)]$ (1)

To a mixture containing H_2L^1 (18.7 mg, 0.05 mmol), $Co(NO_3)_2 \cdot 6H_2O$ (0.73 mg, 0.025 mmol) and 20 mL H_2O , was added aqueous NaOH (0.05 M) to adjust the pH of the mixture to be 5.0, and then 2,2'-bpy (0.40 mg, 0.025 mmol) was added. The resulting mixture was stirred for 30 min and then filtered. Upon standing the filtrate for about a month, the purple-colored cube-shaped crystals were obtained, washed by H_2O and air-dried (yield: 54% based on $Co(NO_3)_2 \cdot 6H_2O$). *Anal.* Calc. for $C_{32}H_{21}CoN_2O_7$: C, 63.53; H, 3.47; N, 4.63. Found: C, 63.43; H, 3.6; N, 4.42%. IR (KBr, cm⁻¹): 1678s, 1594m, 1560m, 1395s, 1326m, 1246m, 1020w, 951w, 929w, 770s, 532s.

2.2.2. Synthesis of $[Ni^{II}L^{1}(2,2'-bpy)(H_{2}O)] \cdot H_{2}O(2)$

To a mixture containing H_2L^1 (18.7 mg, 0.05 mmol), Ni(NO₃)₂·6H₂O (0.72 mg, 0.025 mmol) and 20 mL H₂O, was added aqueous NaOH (0.05 M) to adjust the pH of the mixture to be 8.0, and then 2,2'-bpy (0.40 mg, 0.025 mmol) was added. The resulting mixture was stirred for 30 min and then transferred to a Teflon-lined stainless steel vessel (25 mL), sealed, heated to 160 °C and maintained at that temperature for 3 days, then finally cooled at a rate of 5 °C/h to room temperature. Blue-colored cubeshaped crystals were obtained, washed by H₂O and air-dried

Table	1
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Crystal	data	and	structure	refinement	for	complexes	1-4	١.

(Yield: 72% based on Ni(NO₃)₂·6H₂O). *Anal.* Calc. for $C_{32}H_{23}NiN_2O_8$: C, 61.71; H, 3.7; N, 4.5. Found: C, 61.62; H, 3.85; N, 4.42%. IR (KBr, cm⁻¹): 1673s, 1660m, 1576m, 1552s, 1490w, 1353s, 1249s, 1026w, 772s, 634w, 521m.

2.2.3. Synthesis of $[Co_2^{II}(L^2)_2(2,2'-bpy)_2(H_2O)]$ (3)

The pH of an aqueous solution containing H_2L^2 (20.2 mg, 0.05 mmol), $Co(NO_3)_2$ · $6H_2O$ (0.73 mg, 0.025 mmol) and 18 mL H_2O was adjusted with K_2CO_3 to 6.0, and then 2,2'-bpy (0.40 mg, 0.025 mmol) was added. The resulting mixture was stirred for 30 min, filtered, and then transferred to a Teflon-lined stainless steel vessel (25 mL), sealed, heated to 150 °C and maintained at that temperature for 4 days, then finally cooled at a rate of 5 °C/h to room temperature. This afforded purple-colored cube-shaped crystals of **3**, which were washed by H_2O and air-dried (Yield: 66% based on $Co(NO_3)_2$ · $6H_2O$). Anal. Calc. for $C_{68}H_{49}Co_2N_4O_{13}$: C, 65.39; H, 3.93; N, 4.49. Found: C, 65.28; H, 4.14; N, 4.30%. IR (KBr, cm⁻¹): 1657s, 1603s, 1582m, 1384s, 1260m, 920w, 896m, 768s, 738m, 655w, 506w.

2.2.4. Synthesis of $[Ni_2^{II}(L^2)_2(2,2'-bpy)_2(H_2O)]$ (4)

Complex **4** was synthesized by starting with $Ni(NO_3)_2 \cdot 6H_2O$ (0.72 mg, 0.025 mmol) under similar conditions to **3**, except that the pH of the reaction mixture was 8.5. Blue-colored cube-shaped crystals of **4** were obtained (Yield 58% based on $Ni(NO_3)_2 \cdot 6H_2O$).

Complex	1	2	3	4
Formula	C ₃₂ H ₂₁ N ₂ O ₇ Co	C ₃₂ H ₂₃ N ₂ O ₈ Ni	C ₆₈ H ₄₉ N ₄ O ₁₃ Co ₂	C68H49N4O13Ni2
fw	604.44	622.22	1247.97	1247.53
T (K)	293(2)	296(2)	296(2)	293(2)
λ (Å)	0.71069	0.71073	0.71073	0.71069
Crystal system	triclinic	monoclinic	monoclinic	monoclinic
Space group	$P\overline{1}$	$P2_1/c$	$P2_1/c$	$P2_1/c$
a (Å)	9.373(12)	13.9956(8)	16.0141(9)	15.852(12)
b (Å)	11.673(15)	20.7637(12)	17.5279(10)	17.539(13)
c (Å)	13.808(18)	10.2864(6)	22.2579(13)	22.255(17)
α (°)	75.243(2)	90.00	90.00	90.00
β (°)	75.941(2)	110.7600(10)	102.5850(10)	102.687(10)
γ (°)	71.844(2)	90.00	90.00	90.00
V (Å ³)	1366.1(3)	2795.2(3)	6097.5(6)	6036.4(8)
Ζ	2	4	4	4
Dc (g/cm ³)	1.469	1.474	1.359	1.373
$\mu ({\rm mm^{-1}})$	0.682	0.751	0.612	0.693
$R_1^{a} \left[I > 2\sigma(I) \right]$	0.0356	0.0337	0.0389	0.0369
$wR_2^{b} [I > 2\sigma(I)]$	0.0920	0.0941	0.0839	0.0831
R1 (all data)	0.0428	0.0379	0.0805	0.0652
wR ₂ (all data)	0.0953	0.0966	0.0965	0.0914
Goodness-of-fit	1.103	1.066	0.992	1.008
Transmission (max/min)	0.903/0.822	0.874/0.835	0.907/0.863	0.901/0.861
Δ/σ (max/min)	0.460/-0.269	0.746/-0.718	0.248/-0.262	0.331/-0.332
F(0 0 0)	620	1276	2572	2580

^a $R_1 = \sum ||F_0| - |Fc|| / \sum |F_0|.$

^b $wR_2 = \sum [w(F_0^2 - Fc^2)2] / \sum [w(F_0^2)^2]^{1/2}$.



Fig. 1a. Coordination environment of Co atom in **1** with the ellipsoids draw at the 30% probability level, all hydrogen atoms are omitted for clarity.

Anal. Calc. for $C_{68}H_{49}Ni_2N_4O_{13}$: C, 65.41; H, 3.93; N, 4.49. Found: C, 65.32; H, 4.14; N, 4.30%. IR (KBr, cm⁻¹): 1658s, 1604s, 1579s, 1552s, 1490m, 1388s, 1042w, 896m, 769s, 739m, 571w, 413w.

2.3. X-ray crystallography

Single crystals of **1–4** were glued to a fiberglass for data collection on a SMART (Bruker, 2002) diffractometer equipped with Mo K α radiation. Empirical absorption corrections were applied to the data using ABSCOR [39]. The structure was solved by the direct method followed by the difference Fourier method and refined by full-matrix least-squares on F^2 using the SHELXTL-97 package [40]. All non-hydrogen atoms were refined anisotropically, the hydrogen atoms of organic ligands and coordinated water were located geometrically. Due to the small crystallographic disorder for **2**, hydrogen atoms of the lattice water molecule were not located. The crystal data and structure refinements of **1–4** are summarized in Table 1 and selected bond lengths and angles are listed in the supplementaryinformation (S1 Table S1).

3. Results and discussion

3.1. Structure description

3.1.1. Structure description of 1

Crystallographic analysis shows that complex **1** is an infinite zigzag chain coordination polymer. As shown in Fig. 1a, the Co atom is five-coordinate with two carboxylate O atoms (O1 and O3) of two different L^1 ligands, two N atoms (N1 and N2) of the chelating 2,2'-bpy and one O atom (O1 W) of the aqua ligand. The coordination geometry can be described as distorted trigonal



Fig. 1b. A view of a 2D diagram in **1** via C–H··· π interactions.



Fig. 2a. Coordination environment of the Ni atom in **2** with the ellipsoids draw at the 30% probability level, all hydrogen atoms are omitted for clarity.

bipyramidal with its equatorial plane defined by the O(1), N(2) and O(1 W) atoms; the axial positions are occupied by O(3) and N(1). The Co–O bond lengths, from 2.012(2) to 2.107(2) Å, and the Co–N bond lengths, from 2.086(2) to 2.131(2) Å, are within the normal range [41–43]. The two carboxylate groups show monodentate coordination (Scheme 1a) and the dihedral angles between the 1-, 4-carboxylate groups and their corresponding phenyl rings are both 173.26°. In the overall structure of **1**, units of $[Co(bpy)]^{2+}$ are connected by the bridging L¹ ligands to generate a zigzag chain with a pitch of 15.65 Å. Adjacent chains extend into



Scheme 1. The L¹ ligand adopts two distinct coordination modes, namely bridging bis(mono-dentate) and chelating bisdentate coordination modes.



Fig. 2b. A perspective views of two infinite helical chains in 2.

a 2D supramolecular sheet via a C-H $\cdots \pi$ interaction between the C-H moiety of the benzene rings in one chain and the benzene rings of adjacent chains (H $\cdots \pi$ 3.60 Å, C-H $\cdots \pi$ 146°, Fig. 1b).

3.1.2. Structure description of 2

Crystallographic determination shows that there are two helical chains with opposite directions. In complex **2** the Ni atom is six-



Fig. 3a. Coordination environment of the Co(1) and Co(2) atoms in **3** with the ellipsoids drawn at the 30% probability level, all hydrogen atoms are omitted for clarity.

coordinate with a distorted octahedral geometry. The six-coordinate atoms are two N atoms of the 2,2'-bpy ligand (N1 and N2), two O atoms (O1 and O2) of a chelating carboxylate group, an O atom (O4) from the other monodentate carboxylate group of the L¹ ligand and an O atom (O1 W) of an aqua ligand (Fig. 2a). The average Ni–O and Ni–N bond lengths are 2.093(2) and 2.043(2) Å, respectively. The dihedral angles between the 1-, 4-carboxylate groups and their corresponding phenyl rings are both 145.89 (or 148.1)°. Doubly chelate (both carboxylate groups chelating) and doubly monodentate L¹ ligands alternate with the Ni atoms in complex **2** (Scheme 1a and b).

It is clear from the above description that subtle differences exist in the coordination of the same ligand with Ni and Co ions. The combination of two twisted carboxylate groups and the rotated phenyl ring result in two strand chains in **2**, the two distinct chains coexist and are oppositely arranged via a C–H··· π interaction (Fig. 2b). The [Ni(bpy)]²⁺ units are joined by independent L¹ spacers to generate an infinite helical chain with a period of 20.09 Å. Those chains further extend into 2D via a C–H··· π interaction between the pyridine ring proton and the pyridine ring of adjacent chains (H··· π 3.18 Å, C–H··· π 96°, Fig. 2c).



Fig. 2c. The perspective view of a 2D helical diagram in **2** via C–H··· π interactions.



Scheme 2. The coordination modes of the deprotonated H_2L^2 ligand.



Fig. 3b. The 2D diagram of helical chain via $C-H\cdots\pi$ interactions in **3**.

3.1.3. Structure description of **3** and **4**

Complex **3** is also a helical chain. As shown in Fig. 3a, there are two kinds of Co atoms (Co(1) and Co(2)) in **3**. The Co(1) is six-coordinate with a distorted octahedral geometry. The equatorial plane is defined by N(1), N(2), O(2) and O(1 W) atoms; the axial positions are occupied by O(3) and O(6) atoms (O(3)–Co(1)–O(6) 160.84(8)°). The Co(1)–O and Co(1)–N bond lengths are 2.024(2)–2.246(2) Å and 2.090(2)–2.100(2) Å, respectively. Co(2) is also six-coordinate, but the coordination polyhedron is more like a trigonal antiprism. In this complex Co(1) adopts the chelate-monodentate carboxylate groups coordination (Scheme 2a and b) to afford an infinite helical chain with a period of 25.60 Å. These chains progress in one direction and further extend into a 2D supramolecular sheet via C–H··· π interactions between the pyridine ring proton and a phenyl ring (H··· π 3.50, 3.46 Å and C–H··· π 117, 101°, Fig. 3b).

Complex **4** is isostructural with **3** but Ni atoms instead of Co atoms.

The different coordination polymers formed under slightly different reaction conditions, such as the pH is lower for **1** and **3** than for **2** and **4**, otherwise we can only obtain a precipitate not a crystalline product, which suggests that the assembly is sensitive to the synthetic conditions, including temperature (ambient or under hydrothermal conditions), pH and the nature of ligand and metal ion. At the same time, supramolecular forces also play an important role in constructing the MOFs.

3.2. Thermal analysis

The thermal stability of **1**–**4** were studied by TGA and are recorded in Fig. S1a–d (in the Supplementary data). For **1**, the weight loss incurred between 118 and 256 °C corresponds to the loss of the coordinated water molecule (Obsd. 3.01%, Calc. 2.98%). Subse-



Fig. 4. Solid-state photoluminescent spectra of 2.



Fig. 5. Plots of the temperature dependence of $\chi_M T$ and χ_M^{-1} at 2–300 K.

quent weight loss from 300 to 402 °C is probably due to organic ligand decomposition (Obsd. 64.7%, Calc. 63.5%). The residue is Co_2O_3 (Obsd. 27.57%, Calc. 27.46%). For **2**, the TG curve exhibits three steps of weight loss. The first step is 2.84% from 100 to 140 °C and is possibly due to the loss of the water molecule of crystallization (Calc. 2.89%). The second weight loss is 2.99% in the temperature range 140–180 °C, ascribable to the loss of the aqua ligand (Calc. 2.89%). The third step from 200 to 400 °C is due to the decomposition of the organic ligand (Obsd. 65.71%, Calc. 61.71%). The remaining residue is probable NiO (Obsd. 13.62%, Calc. 12.05%). Complexes **3** and **4** are isomorphic with three steps of weight loss. For **3**, the first step is 1.40% in the temperature range 100–200 °C, corresponding to the loss of the coordinated water molecule (Calc. 1.44%). The second step is due to the release of the organic ligand in the temperature range 200–550 °C (Obsd. 64.17%, Calc. 65.38%), and the remaining weight (Obsd. 12.46%, Cacl. 13.30%) is assigned to Co_2O_3 .

3.3. Luminescence properties

The solid-state luminescence of **2** was investigated. An intense emission at $\lambda em = 435$ nm upon excitation at $\lambda ex = 360$ nm (Fig. 4) was observed. The solid-state photoluminescence property of the free H₂L¹ ligand was analyzed (Fig. S2) in comparison. It is clear that the emission of **2** is blue shifted compared with the free H₂L¹ ligand, and this is assigned to an intraligand $\pi \rightarrow \pi^*$ transition and charge-transfer transition between the coordinated ligand and the metal center [44–47].

3.4. Magnetic properties

The magnetic properties of **1** were studied in the temperature range 2–300 K (Fig. 5). The $\chi_M T$ value at 300 K is 3.528 cm³ K mol⁻¹, larger than the value for a spin-only S = 3/2 system (1.875 emu K mol⁻¹). This is expected as there is a significant first-order orbital contribution to the magnetic moment for a five-coordinate high spin cobalt(II) ion [48]. Upon lowering the temperature $\chi_M T$ decreases to 1.191 cm³ K mol⁻¹ at 2 K. The fitting of the curve for the χ_M^{-1} versus *T* plot to the Curie–Weiss law gives a good result in the temperature range of 50–300 K with C = 3.931 cm³ K mol⁻¹ and $\theta = -41.191$ K, consistent with the presence of an antiferromagnetic interaction in **1**.

4. Conclusions

In summary, $[Co^{II}L^1(2,2'-bpy)(H_2O)]$ (1), $[Ni^{II}L^1(2,2'-bpy)(H_2O)]$. H₂O(2), $[Co^{II}_2(L^2)_2(2,2'-bpy)_2(H_2O)]$ (3) and $[Ni^{II}_2(L^2)_2(2,2'-bpy)_2(H_2O)]$ (4), four new coordination polymers, were synthesized and characterized. In addition, magnetic and luminescence properties have been studied for 1 and 2, respectively. It is anticipated that more metal–organic frameworks (MOFs) employing L¹, L² or other analogous ligands will be produced and possibly realized in functioned materials.

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Appendix A. Supplementary data

CCDC 691998, 703442, 701829 and 702993 contain the supplementary crystallographic data for compounds **1**, **2**, **3** and **4**. These data can be obtained free of charge via http://www.ccdc.cam.ac. uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.poly.2009.06.022.

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