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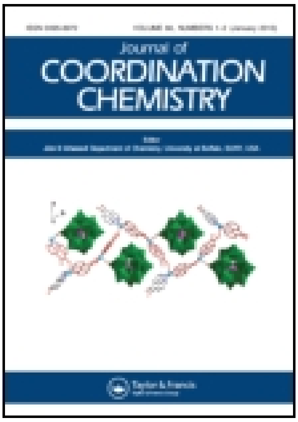

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Dan Tian [a] , Li Guan [a] , Yanhong Zhou [a] & Hong Zhang [a]
[a] Key Laboratory of Polyoxometalate Science of Ministry of Education, Department of Chemistry , Northeast Normal University , Changchun , Jilin 130024 , China

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# Syntheses and crystal structures of two Co(II) coordination polymers based on 4,6-bis(4-methylbenzoyl)isophthalic acid and 4,4′-bipyridine

DAN TIAN, LI GUAN, YANHONG ZHOU and HONG ZHANG*

Key Laboratory of Polyoxometalate Science of Ministry of Education, Department of Chemistry, Northeast Normal University, Changchun, Jilin 130024, China

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Two coordination complexes, $[Co_2L_2(4,4'\text{-bpy})_2(H_2O)_4]\cdot 6H_2O$ (**1**) and $[CoL(4,4'\text{-bpy})]$ (**2**) ($H_2L$ = 4,6-bis(4-methylbenzoyl)isophthalic acid and 4,4′-bpy = 4,4′-bipyridine), have been synthesized with the same starting materials under conventional and hydrothermal condition, respectively. Their structures have been characterized by X-ray diffraction, elemental analysis, IR spectra, and thermogravimetric analysis. Complex **1** features a 2-D sheet structure (space group *C2/c*) with (4,4) grid units. The non-covalent interactions (O–H $\cdots$ O, C–H $\cdots$ $\pi$, and weak $\pi \cdots \pi$ interactions) extend **1** into a 3-D supramolecular network. Complex **2** displays a (3,5)-connected network (space group $P\bar{1}$) with a $(4^2\cdot6)(4^2\cdot6^8)$ topology.

*Keywords*: Coordination polymers; Cobalt(II); 4,6-Bis(4-methylbenzoyl)isophthalic acid; Crystal structure

## 1. Introduction

Metal-organic coordination polymers are of interest because of their variety of structures and tremendous potential applications [1–9]. Functionalization and modification of ligands to construct new complexes are significant work in crystal engineering [10–13]; a simple change in substitutions on the organic linker dramatically changes the structures. For instance, introduction of small functional groups on polycarboxylic acids, such as $-NH_2$, $-OH$, and $-NO_2$ [14], may change the electron density, solubility, and coordination modes, generating products different from those of common ligand as long as steric hindrance of the substituent does not have a significant effect [15–18].

To consider the effect of big substituent, we have synthesized a series of coordination polymers with modified aromatic carboxylic acids [19–21]. We choose 4,6-bis(4-methylbenzoyl)isophthalic acid ($H_2L$) and 4,4′-bipyridine as auxiliary ligand to give new complexes. In contrast to basic $H_2BDC$ (benzenedicarboxylic acid) [22–26], $H_2L$ not only contains two bridging carboxylates, but also possesses a large substituent

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*Corresponding author. Email: zhangh@nenu.edu.cn

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which may rotate around the central phenyl ring to generate distinct structures for steric hindrance. In addition, the extended $\pi$-system derived from 1,3-BDC may be useful for the stability of the coordination polymer.

Herein, we synthesize two Co(II) coordination complexes, $[Co_2L_2(4,4'\text{-bpy})_2(H_2O)_4]\cdot 6H_2O$ (**1**) and $[CoL(4,4'\text{-bpy})]$ (**2**), by conventional and hydrothermal synthetic methods, respectively. Complex **1** shows a 2-D layer, the neighboring 2-D sheets stack together in an ABAB fashion through weak interactions to form a 3-D structure. The structure of **2** is a 2-D (3,5)-connected $(4^2\cdot 6)(4^2\cdot 6^8)$ net.

## 2. Experimental

### 2.1. *Materials and instrumentation*

All chemicals were purchased as reagent grade and used without purification. $H_2L$ is synthesized according to the literature [27]. C, H, and N elemental analyses were performed on a Perkin-Elmer 240C elemental analyzer. FT-IR spectra were recorded from KBr pellets (4000–400 cm$^{-1}$) on a Mattson Alpha-Centauri spectrometer. Thermogravimetric analysis (TG) was performed on a Perkin-Elmer TG-7 analyzer heated from 40°C to 900°C under $N_2$ with a heating rate of 10°C min$^{-1}$.

### 2.2. *Syntheses*

**2.2.1. Synthesis of $[Co_2L_2(4,4'\text{-bpy})_2(H_2O)_4]\cdot 6H_2O$ (1).** A mixture of $H_2L$ (40.2 mg, 0.1 mmol), $Co(NO_3)_2\cdot 6H_2O$ (29.1 mg, 0.1 mmol), 4,4′-bpy (19.2 mg, 0.1 mmol), NaOH (8.0 mg, 0.2 mmol), and $H_2O$ (20 mL) was stirred for 30 min, the pH of the mixture was adjusted to 7.0, the resulting solution was filtered and the filtrate was placed at room temperature without disturbance. Pink single crystals of **1** were obtained after 2 weeks. Yield: 52% based on $Co(NO_3)_2\cdot 6H_2O$. Elemental anal. Calcd for $C_{68}H_{68}Co_2N_4O_{22}$ (%): C, 57.88; H, 4.86; N, 3.97. Found: C, 57.84; H, 4.88; N, 3.95. IR data (KBr, cm$^{-1}$): 3628 (s), 3589 (m), 3406 (s), 1677 (s), 1645 (m), 1606 (s), 1539 (m), 1516 (w), 1484 (m), 1414 (m), 1368 (s), 809 (s).

**2.2.2. Synthesis of $[CoL(4,4'\text{-bpy})]$ (2).** A mixture of $H_2L$ (40.2 mg, 0.1 mmol), $Co(NO_3)_2\cdot 6H_2O$ (29.1 mg, 0.1 mmol), 4,4′-bpy (19.2 mg, 0.1 mmol), NaOH (8.0 mg, 0.2 mmol), and $H_2O$ (10 mL) was stirred for 30 min, the pH of the mixture was adjusted to 7.0, then transferred and sealed in a 25 mL Teflon-lined stainless-steel container which was heated at 160 °C for 72 h, then slowly cooled to room temperature at 3.25 °C h$^{-1}$. Purple single crystals of **2** were collected, yield: 60% based on $Co(NO_3)_2\cdot 6H_2O$. Elemental anal. Calcd for $C_{34}H_{24}CoN_2O_6$ (%): C, 66.34; H, 3.94; N, 4.55. Found: C, 66.37; H, 3.91; N, 4.60. IR data (KBr, cm$^{-1}$): 1675 (s), 1605 (s), 1564 (m), 1540 (w), 1412 (s), 1178 (m), 927 (w), 808 (s), 769 (m), 741 (m).

Table 1. Crystal data and structure refinement for **1** and **2**.

| | **1** | **2** |
|---|---|---|
| Empirical formula | $C_{68}H_{68}Co_2N_4O_{22}$ | $C_{34}H_{24}CoN_2O_6$ |
| Formula weight | 1411.09 | 615.48 |
| Crystal system | Monoclinic | Triclinic |
| Space group | *C2/c* | $P\bar{1}$ |
| Unit cell dimensions (Å, °) | | |
| $a$ | 15.662(3) | 9.942(3) |
| $b$ | 22.902(5) | 11.405(4) |
| $c$ | 19.948(4) | 14.653(5) |
| $\alpha$ | 90 | 106.754(3) |
| $\beta$ | 112.31(3) | 95.531(2) |
| $\gamma$ | 90 | 106.056(3) |
| Volume (Å$^3$), $Z$ | 6620(2), 4 | 1500.6(9), 2 |
| Absorption coefficient (mm$^{-1}$) | 0.582 | 0.620 |
| Calculated density (g cm$^{-3}$) | 1.412 | 1.362 |
| $R$(int) | 0.0742 | 0.0304 |
| Goodness-of-fit on $F^2$ | 1.054 | 1.092 |
| Final $R$ indices $[I > 2\sigma(I)]^{a,b}$ $wR_2{}^{b}$ $[I > 2\sigma(I)]$ | $R_1 = 0.0586$, $wR_2 = 0.1642$ | $R_1 = 0.0487$, $wR_2 = 0.1145$ |

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

### 2.3. *X-ray crystallography*

Single-crystal X-ray diffraction data for **1** and **2** were recorded on a Bruker Apex CCD diffractometer with graphite-monochromated Mo-K$\alpha$ radiation ($\lambda = 0.71073$ Å) at 293 K. Empirical absorption corrections were applied using the multiscan technique. The structures were solved by Direct Methods of SHELXS-97 [28] and refined by full-matrix least-squares on $F^2$ using the SHELXL-97 program [29] within WINGX [30]. All non-hydrogen atoms were refined anisotropically, hydrogens of organic ligand are located geometrically, and hydrogens of water were located in difference Fourier maps. Due to crystallographic disorder for **1**, hydrogens of free water (O3W) were not located. Crystal data and structure refinement parameters for **1** and **2** are summarized in table 1. Selected bond distances and angles are listed in table 2.

## 3. Results and discussion

### 3.1. *Description of the structures*

**3.1.1. $[Co_2L_2(4,4'\text{-bpy})_2(H_2O)_4]\cdot 6H_2O$ (1).** Single-crystal X-ray structural analysis indicates that **1** crystallizes in the monoclinic space group *C2/c* and shows a 2-D (4,4) network. The asymmetric unit of **1** contains two different Co(II) cations, two $L^{2-}$ and two 4,4′-bpy ligands. There are two six-coordinate Co(II) cations located on the C2 symmetry axis. As shown in figure 1(a), Co1 is coordinated by N1 and N3 from two 4,4′-bpy in axial positions, two carboxylate oxygens (O1, O1A) from two $L^{2-}$ and two coordination waters (O1W, O1WA) in the equatorial plane, while Co2 is coordinated by two carboxylate oxygens (O3B, O3C) from two $L^{2-}$ and two waters (O2W, O2WA)

Table 2. Bond lengths [Å] and angles [°] for **1** and **2**.[a]

| | | | |
|---|---|---|---|
| Complex **1** | | | |
| Co1–O1 | 2.078(3) | Co1–O1W | 2.087(3) |
| Co1–N1 | 2.182(5) | Co1–N3 | 2.203(5) |
| Co2–O3B | 2.052(3) | Co2–O2W | 2.108(3) |
| Co2–N4 | 2.162(5) | Co2–N2D | 2.164(5) |
| Co1–O1A | 2.078(3) | Co1–O1WA | 2.087(3) |
| Co2–O3C | 2.052(3) | Co2–O2WA | 2.108(3) |
| O1–Co1–O1A | 172.73(18) | O1–Co1–O1W | 90.55(13) |
| O1A–Co1–O1W | 89.74(13) | O1WA–Co1–O1W | 175.47(2) |
| O1A–Co1–O1WA | 90.55(13) | O1–Co1–O1WA | 89.74(13) |
| O1–Co1–N1 | 86.37(9) | O1W–Co1–N1 | 92.26(10) |
| O1–Co1–N3 | 93.63(9) | O1W–Co1–N3 | 87.74(10) |
| N1–Co1–N3 | 180.000(1) | O2W–Co2–N2D | 87.43(10) |
| O3B–Co2–O2W | 92.13(13) | O3B–Co2–N2D | 90.03(9) |
| O3C–Co2–O2W | 87.88(13) | O1A–Co1–N1 | 86.37(9) |
| O1WA–Co1–N1 | 92.26(10) | O3B–Co2–N4 | 89.97(9) |
| O2W–Co2–N4 | 92.57(10) | O3B–Co2–O3C | 179.93(18) |
| O2W–Co2–O2WA | 174.9(2) | N4–Co2–N2D | 180.000(1) |
| O1A–Co1–N3 | 93.63(9) | O1WA–Co1–N3 | 87.74(10) |
| O3B–Co2–O2WA | 87.88(13) | O3C–Co2–O2WA | 92.13(13) |
| O3C–Co2–N4 | 89.97(9) | O2WA–Co2–N4 | 92.57(10) |
| O3C–Co2–N2D | 90.03(9) | O2WA–Co2–N2D | 87.43(10) |
| Complex **2** | | | |
| Co1–O5 | 2.0219(17) | Co1–O6 | 2.0267(16) |
| Co1–N1 | 2.1498(19) | Co1–O2 | 2.1632(16) |
| Co1–N2 | 2.1784(19) | Co1–O1 | 2.1843(17) |
| O5–Co1–O6 | 108.61(6) | O5–Co1–N1 | 86.73(7) |
| O6–Co1–N1 | 88.12(7) | O5–Co1–O2 | 95.55(6) |
| O6–Co1–O2 | 155.57(6) | N1–Co1–O2 | 89.73(7) |
| O5–Co1–N2 | 92.73(7) | O6–Co1–N2 | 93.44(7) |
| N1–Co1–N2 | 178.44(7) | O2–Co1–N2 | 88.87(7) |
| O5–Co1–O1 | 156.20(7) | O6–Co1–O1 | 95.02(6) |
| N1–Co1–O1 | 91.34(7) | O2–Co1–O1 | 60.70(6) |
| N2–Co1–O1 | 88.57(7) | | |

[a] Symmetry transformations used to generate equivalent atoms for **1**: A: $1-x$, $y$, $1/2-z$; B: $1/2+x$, $1/2+y$, $z$; C: $1/2-x$, $1/2+y$, $1/2-z$; D: $x$, $1+y$, $z$.

in equatorial positions and N2D and N4 from two 4,4′-bpy in axial positions, showing a slightly distorted octahedral geometry. Two 4-methylbenzoyl groups of $L^{2-}$ are located in the ipsilateral positions of phenyl ring with dihedral angles of 84.29° and 75.29° corresponding to the central benzene.

$L^{2-}$ in **1** are bis(monodentate) linking two Co(II) ions (chart S1a), and forming an infinite Co-L 1-D covalent chain (figure 1b). In addition, 4,4′-bpy ligands bridge Co(II) ions from neighboring chains to form a 2-D network with (4,4) grid units (figure 1c), the two pyridines of 4,4′-bpy are not coplanar with dihedral angles of 10° and 37°, respectively. Finally, neighboring 2-D sheets stack together in an ABAB fashion to generate a 3-D structure, stabilized by O–H $\cdots$ O interactions (O2W $\cdots$ O5W, 2.843 Å; O4W $\cdots$ O5, 2.832 Å; O5W $\cdots$ O6, 2.746 Å; O1W $\cdots$ O5W, 2.728 Å; O5W $\cdots$ O4W, 2.710 Å; O4W $\cdots$ O1, 3.109 Å), C–H $\cdots\pi$ interactions (C15–H15 $\cdots\pi$ and C32–H32 $\cdots\pi$, the edge-to-face separations of 2.84 Å and 3.00 Å), and weak $\pi\cdots\pi$ interactions between pyridyl rings and benzene rings with centroid-to-centroid distances of 4.242 and 4.836 Å (table S1). This further confirms that non-covalent interactions play a crucial role in formation of the crystal structure.

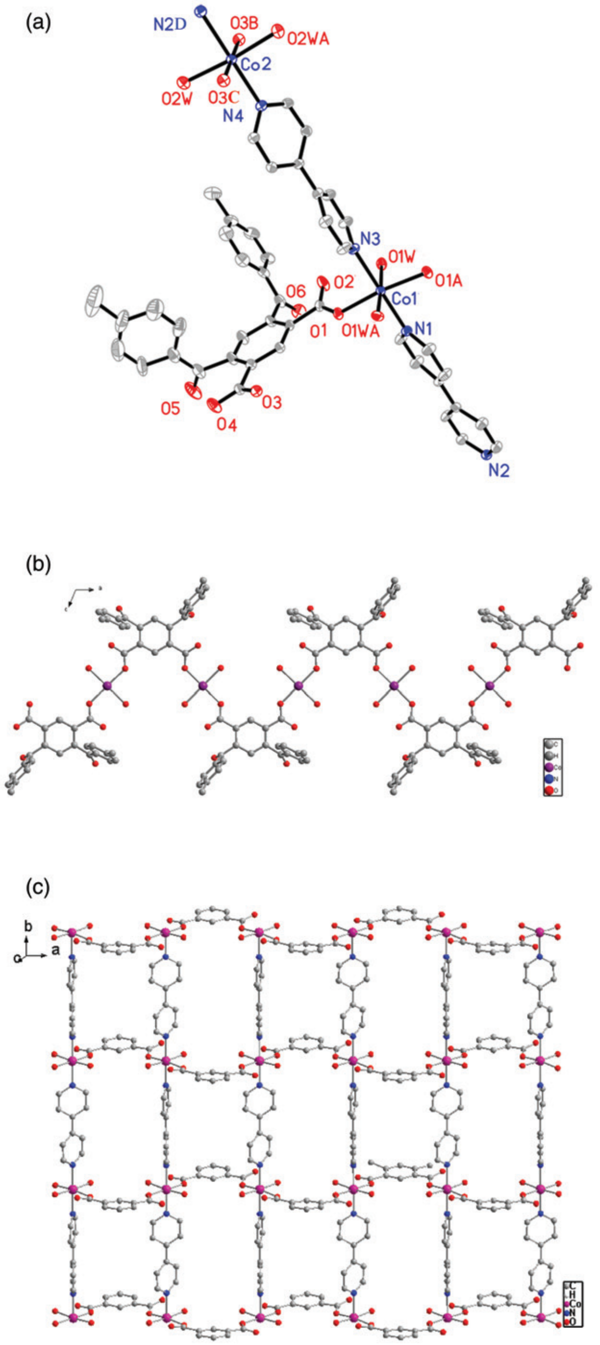


Figure 1. (a) ORTEP diagram showing the coordination environments for cobalt in **1** at 30% probability. Symmetry codes: A: $1-x$, $y$, $1/2-z$; B: $1/2+x$, $1/2+y$, $z$; C: $1/2-x$, $1/2+y$, $1/2-z$; D: $x$, $1+y$, $z$. All hydrogens and water molecules are omitted for clarity. (b) Infinite Co–L 1-D covalently bond chain. (c) The 2-D (4,4) network in **1**. The 4-methylbenzoyl groups of dicarboxylic acid have been omitted for clarity.

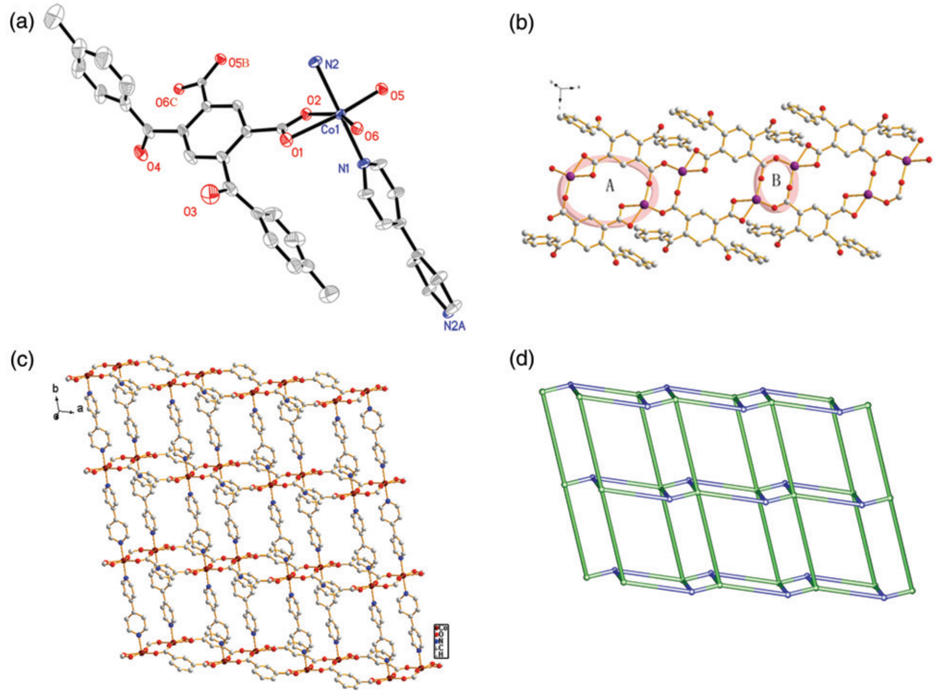
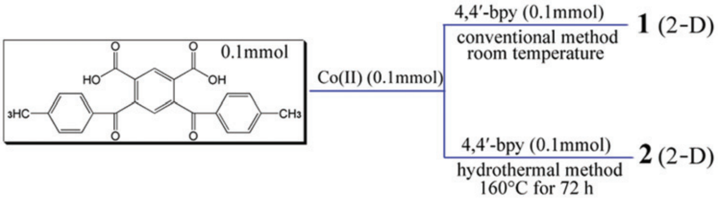


Figure 2. (a) ORTEP diagram showing the coordination environments for cobalt in **2** at 30% probability. All hydrogens are omitted for clarity. Symmetry codes: A: $x$, $-1+y$, $z$, B: $-x$, $1-y$, $-z$, C: $-1+x$, $y$, $z$. (b) Infinite Co–L 1-D double chain structure with rings A and B. (c) The 2-D network of **2**; 4-methylbenzoyl groups of dicarboxylic acid have been omitted for clarity. (d) Schematic diagram (OLEX) of the (3,5)-connected 2-D network with $(4^2\cdot6)(4^2\cdot6^8)$ topological notation.

**3.1.2. [CoL(4,4′-bpy)] (2).** Single-crystal X-ray analysis shows that **2** crystallizes in the triclinic space group $P\bar{1}$ with the asymmetric unit of **2** containing one Co(II), one $L^{2-}$ and one 4,4′-bpy. As depicted in figure 2(a), the Co(II) is six-coordinate with four carboxylate oxygens (O1, O2, O5, and O6) from three different $L^{2-}$ in the equatorial plane and N1 and N2 from two 4,4′-bpy ligands in the apical positions. The coordination geometry around Co(II) can be described as slightly distorted octahedral. The Co–O/N bond distances in **1** and **2** are 2.0219(17)–2.203(5) Å, while O/N–Co–O/N bond angles are 60.70(6)–180.000°, all in the regular range [31, 32]. The two 4-methylbenzoyl groups of $L^{2-}$ are located above and below the basal phenyl ring with dihedral angles of 78.6° and 75.8° corresponding to the central benzene, different from that in **1**.

$L^{2-}$ in **2** is bidentate chelating and bidentate bridging (chart S1b). Each $L^{2-}$ binds three Co(II) ions to form 1-D double chains with 16-membered rings (A) and 8-membered rings (B) [33–35], which are arranged alternately (figure 2b). Adjacent 1-D chains are connected by 4,4′-bpy ligands to form a 2-D network (figure 2c) with dihedral angle between a pair of pyridine rings of 37°. From the topological view, $L^{2-}$ and Co(II) can be viewed as 3- and5-connected nodes, respectively, and the 2-D network can be classified as a (3,5)-connected structure with a $(4^2\cdot6)(4^2\cdot6^8)$ topology (figure 2d) [36].



Scheme 1. The two different syntheses of **1** and **2**.

From the above structural descriptions, we find that the structures of **1** and **2** are very different for conventional and hydrothermal methods with the same starting materials (scheme 1). In complex **1**, $L^{2-}$ is bis(monodentate) but for **2**, $L^{2-}$ is tetradentate, bidentate chelating, and bidentate bridging leading to a 2-D network. Carboxylic acid may tend to monodentate at lower temperature but bidentate or mixed at higher temperature with reaction temperature playing a crucial role in formation of structures.

### 3.2. *Thermal analyses*

To examine thermal stability of the two complexes, thermal gravimetric (TG) analyses were performed under $N_2$ using single crystals of **1** and **2** (figures S1 and S2). Complex **1** shows a weight loss of 11.45% from 40°C to 110°C corresponding to lattice and coordinated water molecule (Calcd 12.76%). Organic components are removed from 280°C to 400°C (Obsd. 76.62%; Calcd 78.88%); the remaining product is $Co_2O_3$ (Obsd. 11.93%, Calcd 11.75%). The TGA curve of **2** is similar to that of **1**, but does not have water molecule. Complex **2** begins to collapse at 280°C, attributed to the loss of organic ligands (Obsd. 88.95%, Calcd 90.43%). The remaining weight (11.05%) indicated $Co_2O_3$ as final product (Calcd 13.47%).

## 4. Conclusion

We synthesized two new Co(II) coordination polymers, $[Co_2L_2(4,4'\text{-bpy})_2(H_2O)_4]\cdot 6H_2O$ (**1**) and $[CoL(4,4'\text{-bpy})]$ (**2**), with the same starting materials under diverse synthetic approaches (conventional and hydrothermal condition); reaction temperature plays a very important role in constructing MOFs. Subsequent work will focus on the construction of coordination polymers by reacting isomorphous ligand, and flexible ligands with more metal ions.

## Supplementary material

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Center, CCDC Nos. 702867 and 712986 for **1** and **2**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB21EZ, UK (Fax: +44-1223-336033; E-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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