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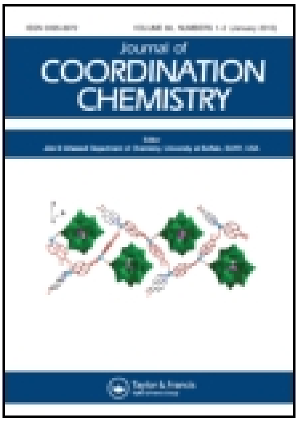

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Two threefold interpenetrated metal-organic frameworks (Co^{II}, Ni^{II}) with (6^5.8) topology constructed from biphenyl-3,4′-dicarboxylic acid

Zhong Sun [a] , Yuanshuai Ding [a] , Laijin Tian [a] & Xiutang Zhang [a] [b]
[a] College of Chemistry and Chemical Engineering, Qufu Normal University , Qufu , China
[b] Department of Chemistry and Chemical Engineering , Qilu Normal University , Jinan , China

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Two threefold interpenetrated metal-organic frameworks (Co^{II}, Ni^{II}) with (6^5.8) topology constructed from biphenyl-3,4′-dicarboxylic acid

ZHONG SUN†, YUANSHUAI DING†, LAIJIN TIAN*† and XIUTANG ZHANG*†‡

†College of Chemistry and Chemical Engineering, Qufu Normal University, Qufu, China;
‡Department of Chemistry and Chemical Engineering, Qilu Normal University, Jinan, China

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[Co(bmib)(bpda)]·H_2O (**1**) and [Ni(bmib)(bpda)] (**2**) (H_2bpda = biphenyl-3,4′-dicarboxylic acid; bmib = 4,4′-bis(2-methylimidazol-1-ylmethyl)benzene) were synthesized under hydrothermal conditions. The structures have been determined by single-crystal X-ray diffraction analyzes and further characterized by elemental analyzes, IR, PXRD, and TGA. Compounds **1** and **2** possess a threefold interpenetrated (6^5.8) architecture that incorporates left- and right-handed helical chains. The difference of angles between the coordination spacers in **1** and **2** causes a large difference along the *c*-axis.

Keywords: Biphenyl-3, 4′-dicarboxylic acid, 4,4′-bis(2-methylimidazol-1-ylmethyl)benzene; Nickel(II); Cobalt(II); Helical

1. Introduction

Crystal engineering based on metal-organic frameworks (MOFs) attracts interest for appealing structures and potential applications in gas storage, microelectronics, ion exchange, chemical separations, nonlinear optics, and heterogeneous catalysis [1–8]. Generally, structures of such materials are dependent on many factors, such as metal ion, template, metal–ligand ratio, pH, counteranion, and number of coordination sites provided by organic ligands [9–16]. In assembly of MOFs, the use of ligands is very important in obtaining the desired MOFs [17–20].

Compared to other biphenyl carboxylic acids, such as 4,4′-biphenyl-dicarboxylic acid [21, 22], 2,4′-biphenyl-tetracarboxylic acid [23], 3,3′,5,5′-biphenyl-dicarboxylic acid [24–26], and 3,3′,4,4′-biphenyl-tetracarboxylic acid [27], biphenyl-3,4′-dicarboxylic acid (H_2bpda) is less common, and the angle between carboxyl groups makes large porous coordination polymers possible. Coordination assemblies using polycarboxylate and N-donor ligands are a reliable strategy to obtain new topological prototypes of coordination networks [28–34]. MOFs containing flexible imidazoline derivatives of $(N\text{–im})_2(CH_2)_n$ ($n = 1$–4) are much reported [35]. The N-donor of 4,4′-bis(2-methylimidazol-1-ylmethyl)

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*Corresponding authors. Email: laijintian@yahoo.com.cn (L. Tian); xiutangzhang@yahoo.com.cn (X. Zhang)

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benzene is seldom used. These considerations inspired us to explore new coordination frameworks with biphenyl-3,4′-dicarboxylic acid (H_2bpda) and 4,4′-bis(2-methylimidazol-1-ylmethyl)benzene. From inspection of reported cases, we found that such flexible N-donor ligands are good because the flexibility allows it to bend and rotate when it coordinates to metal centers, allowing structural diversity. On the other hand, structures and properties can be optimized by changing the length of the spacer.

Herein, we report the syntheses and characterizations of {[Co(bmib)(bpda)]·H_2O (**1**) and [Ni(bmib)(bpda)] (**2**). Compounds **1** and **2** have a 3D structure with threefold interpenetrated (6^5.8) topology; the difference of angles between coordination spacers in one and two causes a large difference along the *c*-axis. Crystal structures, elemental analyzes, infrared spectra, topological analyzes, TGA, and PXRD properties are studied in detail.

2. Experimental

2.1. *General material and methods*

Biphenyl-3,4′-dicarboxylic acid and 4,4′-bis(2-methylimidazol-1-ylmethyl)benzene were purchased from Jinan Henghua Sci. & Tec. Co. Ltd. and used without purification. Other chemicals purchased were of reagent grade and used without purification. IR spectra were recorded from KBr pellets (4000–400 cm^{-1}) on a Bruker EQUINOX-55 spectrometer. Elemental analyzes were determined with an Elementar Vario EL III elemental analyzer. Thermogravimetric measurements were carried out in a nitrogen stream using a Netzsch STA449C apparatus with a heating rate of 10 °C min^{-1}. Powder X-ray diffraction (PXRD) measurements were carried out on a RIGAKU DMAX2500 apparatus.

2.2. *Preparation of coordination polymers*

2.2.1. Synthesis of [Co(bmib)(bpda)]·H_2O (1). The synthesis is performed in a 25-mL Teflon-lined stainless steel vessel. A mixture of H_2bpda (0.20 mmol, 0.052 g), bmib (0.22 mmol, 0.060 g), cobalt chloride hexahydrate (0.20 mmol, 0.048 g), NaOH (0.30 mmol, 0.012 g), and 10 mL H_2O was heated to 170 °C for 3 days, followed by slow cooling (a descent rate of 10 °C/h) to room temperature, giving purple block crystals. Yield of 76% (based on Co). Anal. (%) calcd for $C_{30}H_{28}N_4CoO_5$ (583.49): C, 61.70; H, 4.80; N, 9.60. Found: C, 61.18; H, 4.69; N, 9.21. IR (KBr pellet, cm^{-1}): 3938(vs), 2985(v), 2847(vs), 1603(vs), 1505(vs), 1307(vs), 1128(m), 1012(m), 938(m), 746(v), 555(m).

2.2.2. Synthesis of [Ni(bmib)(bpda)] (2). The synthesis is also performed in a 25-mL Teflon-lined stainless steel vessel. A mixture of H_2bpda (0.20 mmol, 0.052 g), bmib (0.22 mmol, 0.060 g), nickel chloride hexahydrate (0.20 mmol, 0.048 g), NaOH (0.40 mmol, 0.016 g), and 10 mL H_2O was heated to 170 °C for 3 days, followed by slow cooling (a descent rate of 10 °C/h) to room temperature, giving green block crystals. Yield of 81% (based on Ni). Anal. (%) calcd for $C_{30}H_{26}N_4NiO_4$ (565.26): C, 63.69; H, 4.60; N, 9.90. Found: C, 62.98; H, 4.59; N, 9.37. IR (KBr pellet, cm^{-1}): 3918(vs), 2984(v), 2829(vs), 1599(v), 1521(vs), 1315(vs), 1120(m), 1024(v), 935(m), 746(v), 554(m).

2.3. *X-ray crystallography*

Intensity data collection was carried out on a Siemens SMART diffractometer equipped with a CCD detector using Mo-Kα monochromated radiation ($\lambda = 0.71073$ Å) at 293(2) K. The absorption correction was based on multiple and symmetry-equivalent reflections in the data set using SADABS based on the method of Blessing. The structures were solved

Table 1. Crystallographic data and details of diffraction experiments for **1** and **2**.

Formula	**1** $C_{30}H_{28}N_4CoO_5$	**2** $C_{30}H_{26}N_4NiO_4$
M_r	583.49	565.26
Crystal system	Monoclinic	Monoclinic
Space group	*P2(1)/n*	*P2(1)/n*
a (Å)	9.940(3)	10.3556(11)
b (Å)	21.520(6)	16.8325(19)
c (Å)	13.463(4)	16.4542(18)
α (°)	90.00	90.00
β (°)	92.517(6)	107.475(2)
γ (°)	90.00	90.00
V (Å^3)	2877.0(14)	2735.8(5)
Z	4	4
ρ (gcm^{-3})	1.347	1.372
μ (mm^{-1})	0.641	0.751
T (K)	293(2)	293(2)
R_{int}	0.0319	0.0372
R [$I > 2\sigma(I)$]	$R_1 = 0.0483$ $wR_2 = 0.1376$	$R_1 = 0.0436$ $wR_2 = 0.1619$
R (all data)	$R_1 = 0.0926$ $wR_2 = 0.1577$	$R_1 = 0.0858$ $wR_2 = 0.1830$
Goof	1.004	1.005
$(\Delta\rho)_{max}$ (e-/Å^3)	0.798	0.357
$(\Delta\rho)_{min}$ (eÅ^{-3})	−0.351	−0.406

[a] $R1 = \Sigma||F_{obsd.}| - |F_{calcd}||/\Sigma|F_{obsd.}|$.
[b] $wR_2 = \{\Sigma[w(F_{obsd.}^2 - F_{calcd}^2)^2]/\Sigma[w(F_{obsd.}^2)^2]\}^{1/2}$. $w = 1/[\sigma^2(F_o^2) + xP + (yP)^2]$; with $P = (F_o^2 + 2F_c^2)/3$,
[c] $goof = [\Sigma w(F_{obsd.}^2 - F_{calcd}^2)^2/(n-p)]^{1/2}$, where n = number of reflections, p = parameter used.

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

Complex 1							
Co1-N1	2.041(2)	Co1-N3	2.023(2)	Co1-O1	1.969(2)	Co1-O2	2.704(22)
Co1-O3	2. 647(93)	Co1-O4	1.987(2)				
N3-Co1-N1	105.88(10)	N3-Co1-O4	107.54(89)	N1-Co1-O4	94.05(10)	N3-Co1-O1	95.45(68)
N1-Co1-O1	118.03(87)	O4-Co1- O1	133.56(10)	N3-Co1-O2	148.19(18)	N1-Co1-O2	90.00(38)
O4-Co1-O2	98.41(58)	O1-Co1-O2	52.81 (98)	N3-Co1-O3	98.93(18)	N1-Co1-O3	144.12(88)
O4-Co1-O3	54.38(79)	O1-Co1-O3	95.53(89)	O2-Co1-O3	81.75(69)		
Symmetry code: (i) $-x+1, -y, -z$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x-1/2$, $y+1/2, -z+1/2$; (iv) $-x-1/2$, $y-1/2, -z+1/2$							
Complex 2							
Ni1-N1	2.0595(19)	Ni1-N3	2.040(2)	Ni1-O1	2.0957(16)	Ni1-O2	2.1228(16)
Ni1-O3	2.1643(16)	Ni1-O4	2.0733(16)				
N3-Ni1-N1	90.51(8)	N3-Ni1-O4	94.76(8)	N1-Ni1-O4	99.35(7)	N3-Ni1-O1	103.85(8)
N1-Ni1-O1	102.13(7)	O4-Ni1- O1	151.27(6)	N3-Ni1-O2	165.98(8)	N1-Ni1-O2	95.54(7)
O4-Ni1-O2	96.71(7)	O1-Ni1-O2	62.53(6)	N3-Ni1-O3	92.02(7)	N1-Ni1-O3	161.68(8)
O4-Ni1-O3	62.36(6)	O1-Ni1-O3	94.87(6)	O2-Ni1-O3	86.20(7)		
Symmetry code: (i) $-x+2, -y+1, -z$; (ii) $-x+3, -y+2, -z+1$; (iii) $-x+1, y+1/2, -z+1/2$; (iv) $-x+1, y-1/2, -z+1/2$							

by direct methods and refined by full-matrix least-squares using the SHELXTL package [36, 37]. Crystallographic data for **1** and **2** are given in table 1; selected bond lengths and angles are listed in table 2.

3. Results and discussion

3.1. *Structure descriptions*

3.1.1. [Co(bmib)(bpda)]·H_2O (1). The single-crystal X-ray diffraction analysis reveals that 1 crystallizes in the monoclinic system with space group *P2₁/n,* and its structure shows a 3D polymeric framework. The asymmetric unit of 1 consists of one Co^{II}, one bmib, one $bpda^{2-}$, and one associated water. Each Co^{II} locates in a distorted octahedral geometry, coordinated by four oxygens (O1, O2, O3 and O4) from two $bpda^{2-}$ and two nitrogens (N1 and N3) from two different bmib ligands (figure 1). The bond angles around Co^{II} are 94.46(13)-126.73(12)°. The Co–N bond distances are 2.041(2) (Co–N1) and 2.023(2) (Co–N3) Å, and Co–O bond distances are 1.969(2)–2.704(2) Å, respectively.

The two deprotonated carboxylates of $bpda^{2-}$ show μ_1-η^1:η^1 coordination, and $bpda^{2-}$ is an L-shaped ligand linking Co^{II} centers to a 1D zigzag polymeric $[Co(bpda)]_n$ chain containing right-handed helices along the crystallographic [101] direction. Adjacent Co···Co separation is 10.76 Å [figure 2(a)]. Co^{II} ions are coordinated by bmib forming left-handed helices $[Co(bmib)]_n$ with Co–Co distance of 12.69 Å [figure 2(c)] and resulting 2D layer along the *bc* plane [figure 2(b)]. The layers are further bridged via bmib ligands along the *a* direction giving a triply interpenetrated structure with (6^5.8) topology (figure 3). There is intramolecular hydrogen-bond contact (O5–H2 W···O3, 2.854(9) Å) in the interlamellar regions of **1**, playing an important role in stabilization of the packing structure.

Angles between adjacent ligands (bmib to bmib, bmib to $bpda^{2-}$, $bpda^{2-}$ to $bpda^{2-}$) in **1** are 98.681 to 106.935° as listed in table 3, but smaller than 109°28′.

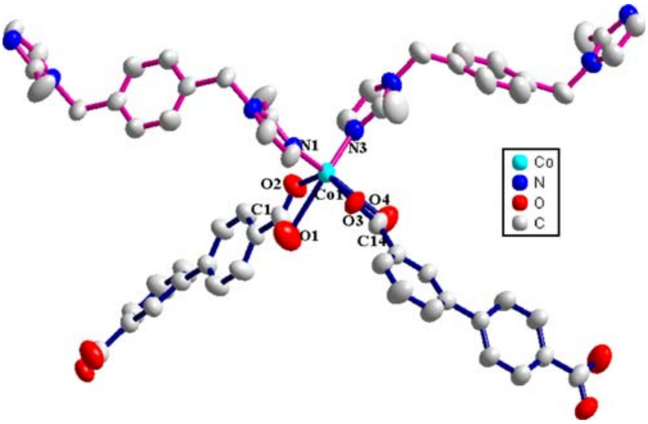

Figure 1. Coordination environment of Co^{II} in **1** with thermal ellipsoids at 30% probability level.

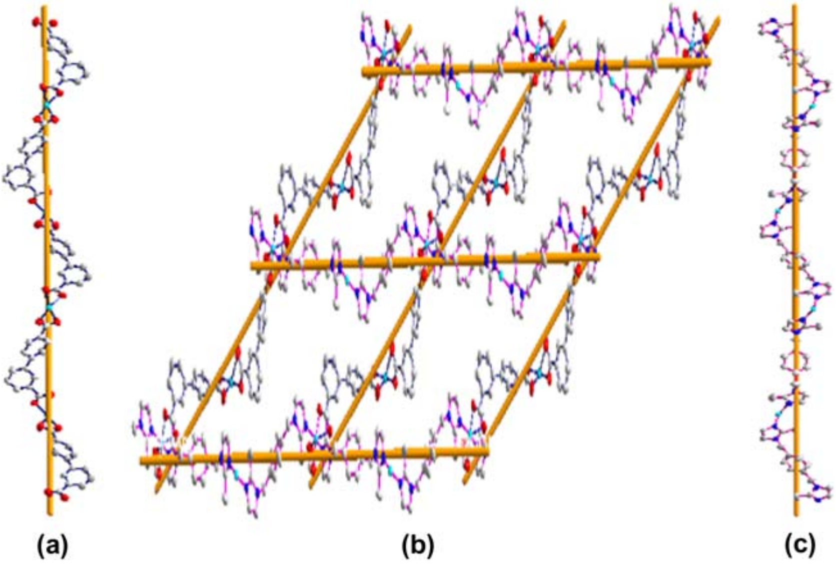

Figure 2. (a) The right-handed helices composed by $bpda^{2-}$ in **1**. (b) The 2D structure of 1 along the *bc* plane. (c) The left-handed helices composed by bmib in **1**.

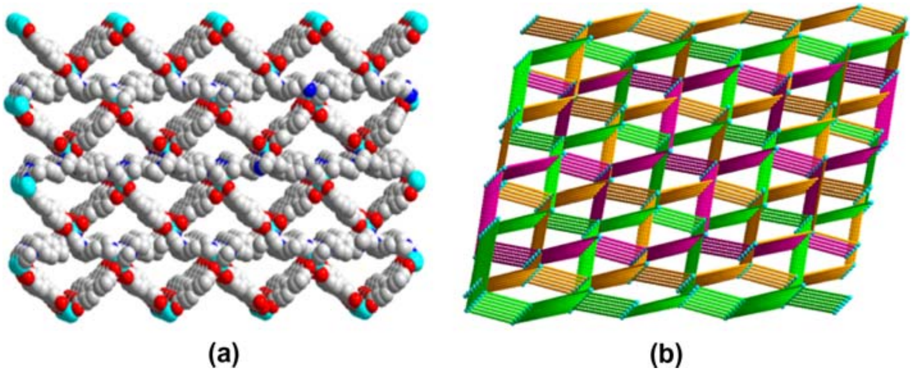

Figure 3. (a) The 3D structure of **1** along the *ab* plane. (b) The 3D threefold interpenetrated ($6^5.8$) topology of **1**.

Table 3. Angles between adjacent ligands of **1** and **2**.

Angle Complex 1	(°)	Angle Complex 2	(°)
C(1)-Co(1)-N(1)	104.20(9)	C(1)-Ni(1)-N(1)	102.82(9)
N(3)-Co(1)-N(1)	105.95(3)	N(3)-Ni(1)-N(1)	90.66(8)
C(1)-Co(1)-C(14)	98.68(1)	C(1)-Ni(1)-C(14)	106.53(4)
N(3)-Co(1)-C(14)	106.93(5)	N(1)-Ni(1)-C(14)	95.72(6)

3.1.2. [Ni(bmib)(bpda)] (2).

Single-crystal X-ray diffraction analysis reveals that two crystallizes in the monoclinic system with space group $P2_1/n$, and its structure shows a 3D polymeric framework. Each Ni^{II} is in a distorted octahedral geometry coordinated by four

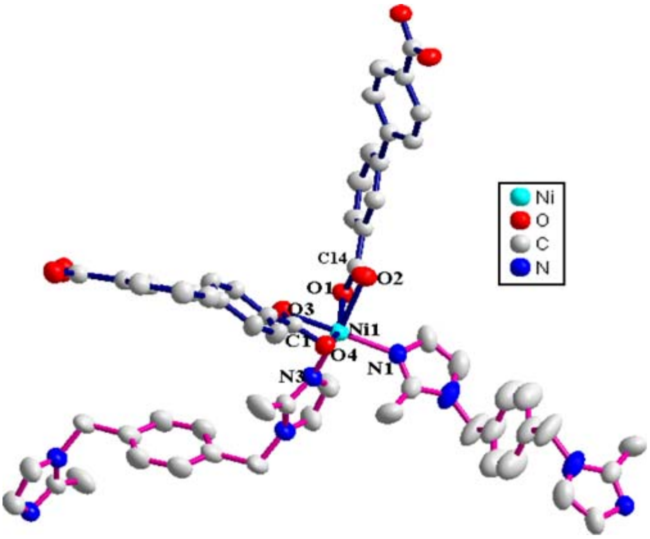

Figure 4. Coordination environment of Ni^{II} in **2** with thermal ellipsoids at 30% probability level.

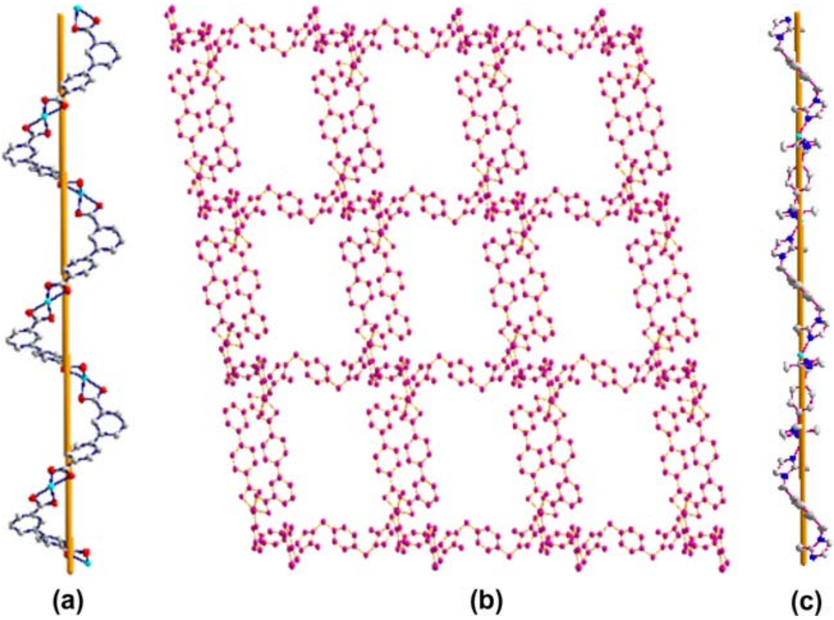

Figure 5. (a) The right-handed helices composed by bpda^{2-} in **2**. (b) The 3D structure of 2 along the *ac* plane. (c) The left-handed helices composed by bmib in **2**.

oxygens (O1, O2, O3, and O4) from two bpda^{2-} and two nitrogens (N1 and N3) from two different bmib ligands (figure 4). The Ni–O and Ni–N distances are 2.073(3)–2.164(3) and 2.040(2)-2.059(5) Å, respectively.

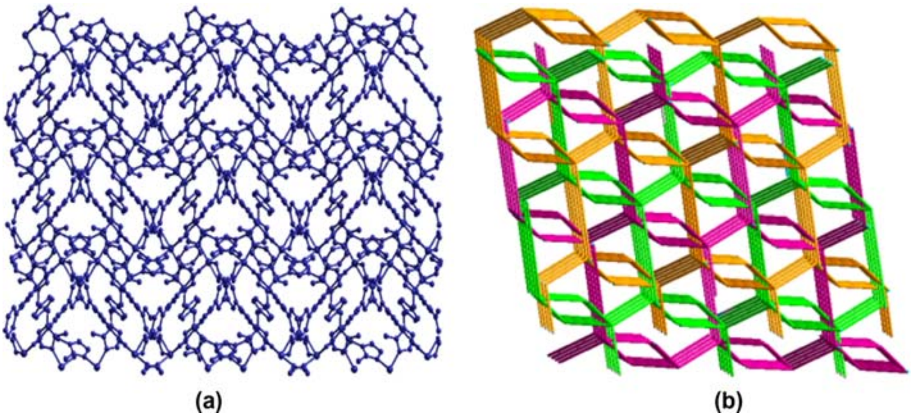

Figure 6. (a) The 3D structure of **2** along the *ab* plane. (b) The 3D threefold interpenetrated (6^5.8) topology of **2**.

In two, the angles between adjacent ligands (bmib to bmib, bmib to $bpda^{2-}$, $bpda^{2-}$ to $bpda^{2-}$) (90.67–106.53°, listed in table 3) are smaller than the ones in 1, leading to a shorter Ni···Ni distance in the right-handed helices of $[Ni(bpda)]_n$ (8.416 Å) [figure 5(a)] and the left-handed helices of $[Ni(bmib)]_n$ (Ni···Ni 12.692 Å) [figure 5(c)], futher connecting to a 3D framework [figure 5(b)]. The Jahn-Teller effect also exists in the 3D triply interpenetrated structure with the (6^5.8) topology (figure 6).

3.2. *The powder X-ray diffraction (PXRD) and thermal analysis*

To determine phase purity of **1** and **2**, PXRD patterns were obtained at room temperature. As shown in Supplementary material, peak positions of the simulated and experimental PXRD patterns are in agreement, demonstrating phase purity of the complexes. The dissimilarities in intensity may be due to the preferred orientation of the crystalline powder samples.

Thermal behaviors of **1** and **2** were studied by thermogravimetric analysis (TGA). The experiments were performed on samples consisting of numerous single crystals under N_2 with a heating rate of 10 °C min^{-1}, as shown in Supplementary Material. The TGA curves for **1** and **2** suggests that the host frameworks are stable to ~300 °C.

4. Conclusion

We have developed a synthetic strategy for coordination polymers using biphenyl-3,4′-dicarboxylic acid and 4,4′-bis(2-methylimidazol-1-ylmethyl)benzene. [Co(bmib)(bpda)]·H_2O (**1**) and [Ni(bmib)(bpda)] (**2**) have been synthesized under solvothermal reaction. Structural comparisons to the reported ones based on biphenyl-3,4′-dicarboxylic acid [38, 39] show H_2bpda plays an important role in governing the structures of these metal-organic coordination architectures. Further systematic studies for the design and synthesis of such crystalline materials with H_2bpda and other N-donor building blocks are underway.

Supplementary material

For **1** and **2**, further details on the crystal structure investigations may be obtained from the Cambridge Crystallographic Data Centre, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [Telephone: +44-(0)1223-762-910; Fax: +44-(0)1223-336-033; E-mail: deposit@ccdc.cam.ac.uk; www: http://www.ccdc.cam.ac.uk/deposit], on quoting the depository number CCDC-886755 for **1** and 886756 for **2**.

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