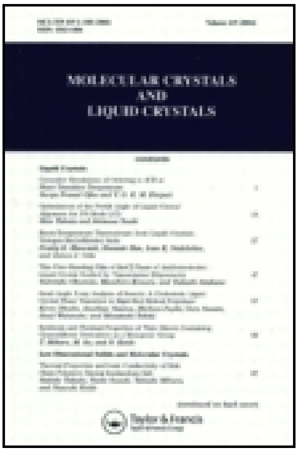

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Synthesis and Characterization of a New Coordination Polymer Constructed from 2,2′-Dibenzyl-1,1′-Binaphthyl-3,3′-dicArboxylic Acid and Cobalt(II)

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Synthesis and Characterization of a New Coordination Polymer Constructed from 2,2′-Dibenzyl-1,1′-Binaphthyl-3,3′-dicArboxylic Acid and Cobalt(II)

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*A novel metal-organic framework Co(bnbc)(C_5H_5N)$_3$ (complex **1**) (H_2bnbc = 2,2′-dibenzyl-1,1′-binaphthyl- 3,3′-dicarboxylic acid) has been synthesized under mild conditions and structurally characterized. IR spectra indicates that the coordination mode of carboxyl group of complex **1** is chelating bidentate with the Co^{2+} ion. Crystal structural analysis reveals that complex **1** adopts a one-dimensional (1D) infinite line structure, and the distance between two carbon atoms of the pair carboxyl groups is 8.226 Å which is longer than other analogs. The complex **1** is further assembled into a 2D supramolecular network through the hydrogen bonds. Thermogravimetric analysis (TGA) shows that complex **1** is highly thermally stable up to 150°C. X-ray powder diffraction (PXRD) of complex **1** shows that single crystalline structure is as the same as the huge mass small crystalline structure confirming its purity of phase. Meanwhile, the photoluminescent property of the complex **1** is investigated.*

Keywords 2,2′-Dibenzyl-1,1′-binaphthyl-3,3′-dicarboxylic acid; Cobalt (II); crystal structure; metal organic frameworks; photoluminescent; thermogravimetric analysis

Metal organic frameworks (MOFs), also called coordination polymer or coordination networks, are a class of hybrid materials formed by the self-assembly of metal ions or clusters and polydentate bridging ligands topically under mild conditions [1]. MOFs are unprecedentedly highly porosity, tunable and can be built from a wide variety of inorganic connecting points and an infinite selection of organic bridging ligands. As a result, numerous MOFs have been engineered for different potential applications, including gas storage [2–4], chemical sensing [5–7], catalysis [8–10], and drug delivery [11,12].

The functionalization of binol has been investigated for several years. Such an interest is due to the great versatility of its derivatives, which has been found a wide array of applications in many different areas of chemistry [13]. Due to their axial chirality with C_2 symmetry and exhibiting a stable configuration in a broad range of conditions, binol

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derivatives have become important molecules in several fields [13]. The binol core has been conveniently functionalized at both the 3,3′ 4,4′, 5,5′ and 6,6′ positions [14–18]. We are particularly interested in 3,3′- bicarboxyl binol since it provides an ideal platform for design catalysts because of the identical secondary environments, and bicarboxyl groups suited to bind metal ions to construct coordination polymer [19,20]. Herein, we have synthesized a ligand of 3,3′-disubstituted binol derivative and constructed a novel MOFs complex **1**. Single crystal X-ray diffraction analysis reveals that complex **1** adopts a one-dimensional (1D) infinite line structure, and the distance between two carbon atoms of the pair carboxyl groups is 8.226 Å which is longer than other analogs [20–22].

Experimental

Materials and Methods

All chemicals were purchased commercially and used without further purification. Infrared spectra were obtained with a Nicolet Impact 410 FTIR spectrometer in the range 400–4000 cm^{-1} using the KBr pellets. A Perkin–Elmer thermogravimetric analys (TGA) thermogravimetric analyzer was used to obtain TGA curve in air with a heating rate of 20°C min^{-1}. ^{1}H NMR spectra were run at 25°C using a Bruker 400 (400 MHz) spectrometer. PXRD spectra were obtained with a Bruker D8 ADVANCE. Photoluminescence spectra was tested by Steady/transient fluorescence spectrometer (Edinburgh, FLS920). Its stimulate light source adopts 450 W of xenon lamp.

Synthesis of Compounds

*Synthesis of 2,2′-Dihydroxy-l,l′-Dinaphthyl-3,3′-Dicarboxy Acid (**2**).* 2,2′-dihydroxy-l,l′-dinaphthyl-3,3′-dicarboxy acid was synthesized according to a published procedure [23]. A mixture of compound **1** 3-hydroxy-2-naphthoic acid (9.4 g, 50 mmol) and $FeCl_3{\cdot}6H_2O$ (20.3 g, 56 mmol) was grinded, the mixture was conducted in microwave tube at 70°C and 500 W for 35 min. The mixture was kept at room temperature with occasional grinding for a certain period of the reaction time until the reaction was completed. The residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (5:1) to afford the product compound **2** (7.0 g, 18.7 mmol, 74%, mp $>$ 290°C). IR (KBr, cm^{-1}): 3059, 1661, 1499, 1455, 1272, 1228, 1150, 1072, 886, 796, 736 cm^{-1}. ^{1}H NMR ($(CD_3)_2CO$, 400 MHz): δ 7.14$\sim$7.17 (m, 2H), 7.39$\sim$7.42 (m, 4H), 8.09$\sim$8.11 (m, 2H), 8.84 (s, 2H).

*Synthesis of 2,2′-Dibenzyloxy-1,1′- Binaphthyl-3,3′-Dicarboxylic Acid Dibenzyl ester (**3**).* 2,2′-dibenzyloxy-1,1′-binaphthyl-3,3′-dicarboxylic acid dibenzyl ester was synthesized according to a published procedure [23]. Compound **2** (0.37 g, 1 mmol) and NaH (0.12 g, 2.62 mmol) was added in DMF (5 mL), then the mixture was stirring at 50°C for 1.5 h. Benzyl chloride (1.5 mL) was slowly added. NaH (0.08 g, 1.78 mmol) was added into the mixture per hour. Tracking the reaction by TLC, until compound **2** reacted completely. Then, H_2O (30 mL) and ethyl acetate (3 $\times$ 50 mL) were added to the mixture. The organic layers were dried over $MgSO_4$ and the solvent was removed under reduced pressure. The solid residue purified by column chromatography to afford the compound **3** (0.46 g, 0.632 mmol, 63.2%, mp: 138°C). IR (KBr, cm^{-1}): 3062, 3029, 1720, 1622, 1588, 1497, 1454, 1373, 1351, 1298, 1273, 1232, 1202, 1162, 1070, 1003, 911, 793, 753, 695 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.51 (d, $J = 8$ Hz, 2H), 4.90 (d, $J = 4$ Hz, 2H), 5.42 (s, 4H), 6.61 (d, $J = 4$ Hz, 4H), 6.98 (t, $J = 4$ Hz, 4H), 7.06 (d, $J = 8$ Hz, 2H), 7.22 (d, $J = 8$ Hz 2H), 7.33∼7.39 (m, 8H), 7.44∼7.49 (m, 6H), 7.97 (d, $J = 8$ Hz, 2H), 8.56 (s, 2H).

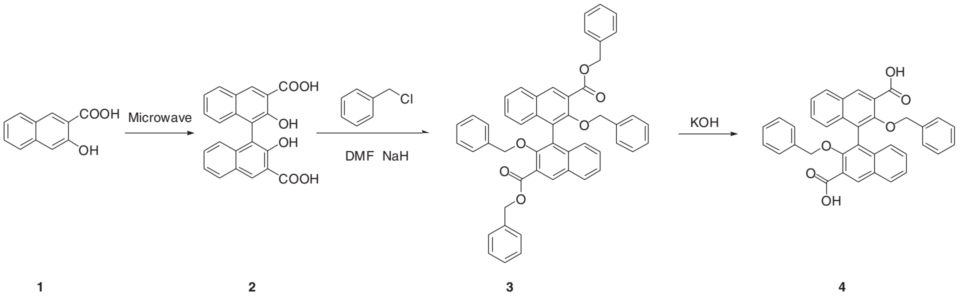
*Synthesis of 2,2′-Dibenzyl-1,1′-Binaphthyl-3,3′-Dicarboxylic Acid (**4**).* 2,2′-dihydroxy-1,1′-binaphthyl-3,3′-dicarboxylic acid was synthesized according to a published procedure [24]. A solution of compound **3** (0.73 g, 1 mmol) in alcohol (2 mL) was added NaOH (5 mL, 10%) and then stirred at 60°C for 10 h, then remove the solvent under reduced pressure and then acidification with hydrochloric acid to afford the compound **4** (0.51 g, 91.6%, mp:188∼190°C), IR (KBr, cm^{-1}): 3062, 2942, 1691, 1620, 1591, 1497, 1452, 1384, 1347, 1291, 1266, 1226, 1204, 1071, 988, 910, 796, 749, 696 cm^{-1}. ^1H NMR (DMSO-d_6, 400 MHz): δ 4.44 (d, $J = 8$ Hz, 2H), 4.88 (d, $J = 8$ Hz, 2H), 6.66 (d, $J = 8$ Hz, 4 H), 7.05∼7.14 (m, 8H,), 7.43 (t, $J = 8$ Hz, 3H), 7.52 (t, $J = 8$ Hz, 2H), 8.17 (d, $J = 8$ Hz, 2H), 8.62 (s, 2H), 13.23 (s, 2H).

*Synthesis of Co(bnbc)(C_5H_5N)$_3$ (Complex **1**).* $Co(NO_3)_2{\cdot}6H_2O$ (16 mg, 0.05 mmol) was dissolved in a mixture of 2 mL DMF and H_2O (V/V = 1:1) as the under layer in a tube. A mixture of DMSO (1 mL) and pyridine (V/V = 1:1) was carefully layered as the middle layer in the tube. The mixture of DMSO (1 mL) and pyridine (1 mL) was put into the tube as the intermediate layer. Compound **4** (0.0075 g, 0.02 mmol) was dissolved in a mixture of DMSO and pyridine (V/V = 1:3, 2 mL) as the upper layer. The tube was then sealed. Diffusion between the three phases over a period of 40 days produced transparent rhombic block crystals of complex **1**. IR (KBr, cm^{-1}): 3056, 1593, 1490, 1445, 1390, 1231, 1073, 752. Anal. Calcd for $C_{51}H_{39}CoN_3O_6$: C, 72.17; H, 4.63; N, 4.95. Found: C, 72.15; H, 4.66; N, 4.92%.

Result and Discussion

Synthesis and Characterization

The ligand of 2,2′-dibenzyl-1,1′-binaphthyl-3,3′-dicarboxylic acid (**4**) is synthesized from 3-hydroxy-2-naphthoic acid through microwave coupling, esterification and hydrolysis of the substrate (Scheme 1).Then, the complex **1** is obtained through the method of solvent diffusion.



Scheme 1. Synthesis of 2,2′-dibenzyl-1,1′-binaphthyl-3,3′-dicarboxylic acid.

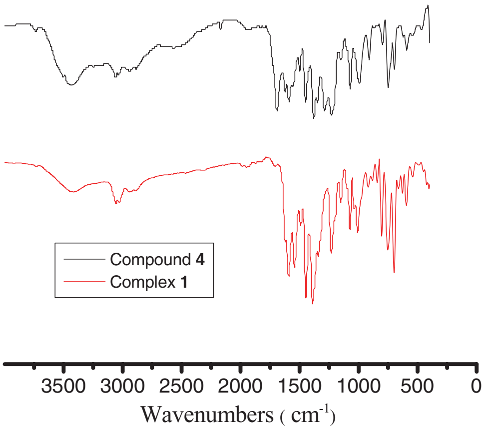


Figure 1. IR spectra of compound **4** and complex **1** (Color figure available online).

IR Study

The infrared spectra were recorded in the region from 4000 cm^{-1} to 400 cm^{-1}. The spectrum of compound **4** exhibits a very strong absorption at 1691 cm^{-1} (Fig. 1), due to the asymmetrical vibration of the C=O of carboxyl group, but the absorption has disappeared when the compound **4** coordinated Co^{2+} ion. The typical bands of the asymmetrical and symmetrical vibrations of C–H of CH_2 are at 2942 cm^{-1} and 2885 cm^{-1}. (Fig. 1) The spectrum of complex **1** exhibits absorption at 1594 cm^{-1} and 1444 cm^{-1} corresponding with the asymmetrical and symmetrical vibrations of the carbonyl groups respectively. The value of Δ [νas-νs] is 140 cm^{-1}, which is smaller than 200 cm^{-1}, indicates that the coordination mode of carboxyl group is chelating bidentate [25–27].

Determination of Crystal Structure

Single crystal X-ray diffraction shows that complex **1** crystallizes in the orthorhombic space group *P2(1)/c*. As showed in Fig. 2(a), the asymmetric unit of the coordination polymer contains one Co(II) ion, one (bnbc) molecule, and three pyridine molecules. The Co(II) ion adopts distorted tetrahedron bipyramidal coordination geometries by coordinated to four carboxyl oxygen atoms from two different ligands and two nitrogen atoms from two pyridine molecules (Fig. 2(b)). The last pyridine molecule is freely filled in the space of the unit. The dihedral angle between the pair of naphthyl rings is 91.6°. The angles of the pair carboxylic acid are difference: one is 125.4°, the other is 118.1°. The Co1-N1 (pyridine) bond length (2.092 Å) is similar to the Co1-N2 (pyridine) bond length (2.102 Å), but the Co1-O2 (carboxylic oxygen) bond length (2.207 Å) is longer than Co1-O1 (carboxylic oxygen) bond length (2.093 Å), which is listed in Table 2. Comparing the distance between two carboxyl groups of the pair naphthyl rings of complex 1 with other reported, there is a longer distance than benzyl has been employed. The H_2(bnbc) ligands act as bridging molecules, which link the Co^{2+} centers into an infinite chain running along the c-axis (Fig. 2(c)). The oxygen atoms of hydroxyl of ligand form donor hydrogen bonds with the Hydrogen atom of naphthalene rings or benzene ring from adjacent molecules. Through interchain hydrogen bonds these 1D chains are further assembled into a 2D supramolecular layer (Fig. 3). On these basis of analysis, the result is in good agreement with our design.

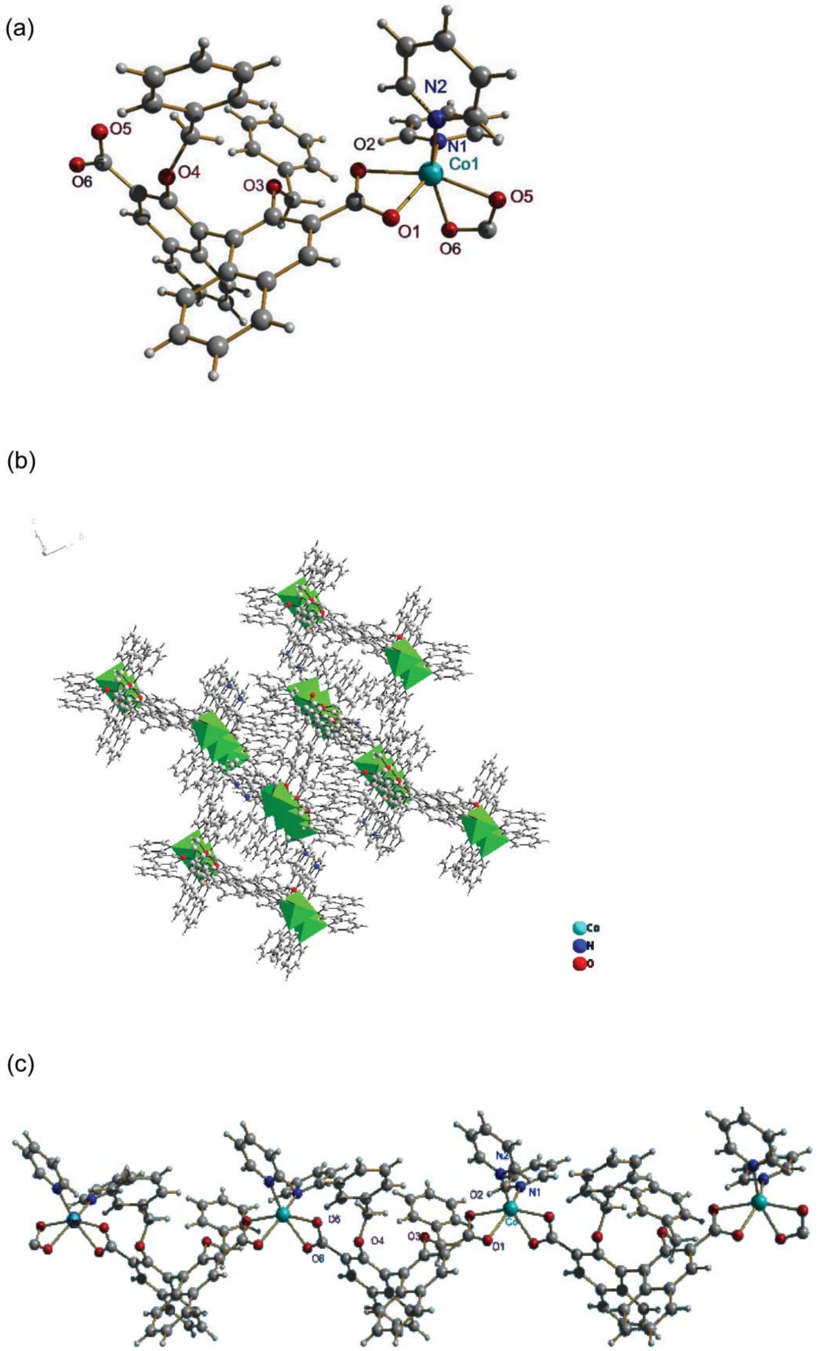


Figure 2. (a) The coordination environments of Co atoms with 30% thermal ellipsoids of complex **1**; (b)distorted tetrahedron bipyramidal coordination geometryis of Co; (c) the 1D chain of complex **1**. All free pyridine molecules are omitted for clarity. (Color figure available online).

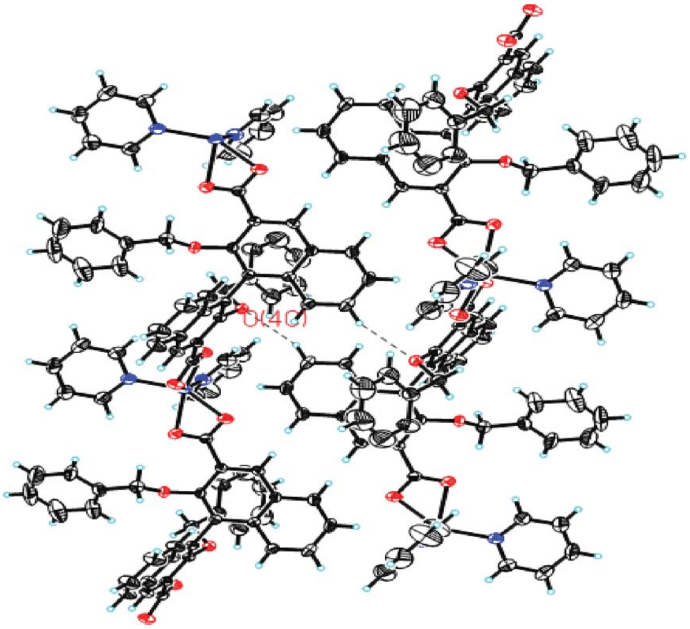


Figure 3. The chainlink structure of Co(bnbc)(C_5H_5N)$_3$ formed by hydrogen bond (Color figure available online).

X-ray Powder Diffraction

In order to confirm the phase purity of the bulk materials, X-ray powder diffraction (PXRD) experiment of complex **1** was carried out. The simulation and actual measurement PXRD pattern of the complex **1** is showed in Fig. 4. Although the experimental patterns have

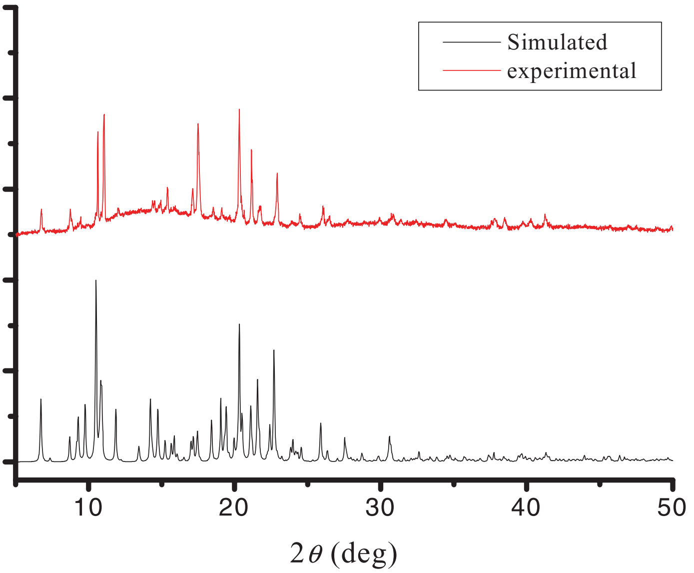


Figure 4. Experimental and simulated PXRD patterns for complex **1** (Color figure available online).

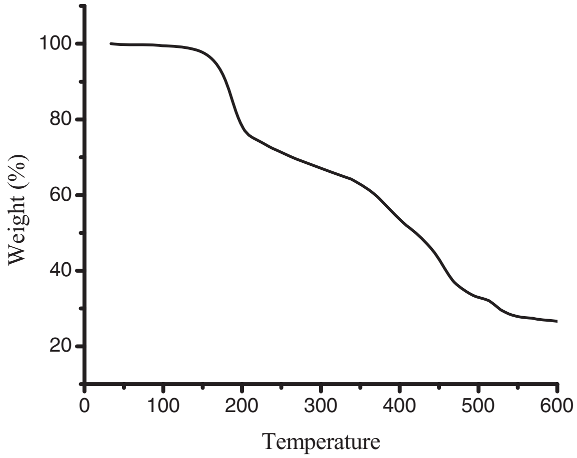


Figure 5. TGA curve of Co(bnbc)(C_5H_5N)$_2$.

a few un-indexed diffraction lines and some are slightly broadened in comparison with those simulated from the single-crystal model, it can still be well considered that the bulk synthesized material and the as-grown crystals are homogeneous for complex **1**, confirming its purity of phase.

Thermogravimetric Analysis

High thermal stability is an important precondition in the conversion of porous coordination frameworks from laboratory curiosities to practical materials. Thus, TGA of complex **1** has been performed in the temperature range 20°C–800°C at a rate of 10°C min^{-1} under N_2 atmosphere. (Fig. 5) The TGA curve shows that complex **1** exhibits a remarkable thermal stability at about 150°C. The TGA curve of complex **1** shows two weight loss steps. The first weight loss is 25.6% in the range of 120°C–220°C, which ascribe to the loss of three pyridine molecules per formula unit (calculated 27.9%). These results show that metal-pyridine bonds are weaker than metal-carboxyl bonds. With a further heating the organic ligand become decomposed between 220°C–350°C, which ascribe to the loss of two benzyl groups. At last, the framework in two naphthyl rings decomposition between 350°C and 520°C, remain finally constant fade away.

Lumminescent Properties

The solid-state emission spectra of ligand H_2bnbc and complex **1** were measured at room temperature (Fig. 6). In the solid state, ligand and complex **1** exhibit green fluorescent emission bands at 560 nm and 561 nm upon excitation at 265 nm and 280 nm, respectively. The intensity increase of the luminescence for the complex **1** may be attributed to the chelation of the ligand to the metal center, which increases the rigidity of bnbc^{2-} and reduces the nonradiative relaxation process. Consequently, the luminescence nature of the complex **1** is metal-perturbed intraligand transition.

Table 1. Crystal data and structure refinement of complex **1**

Identification code	Complex **1**
Empirical formula	$C_{51}H_{39}CoN_3O_6$
Formula weight	848.78
Temperature (K)	296(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	P2(1)/c
a (Å)	$a = 12.2902$ (5)
b (Å)	$b = 19.0083$ (7)
c (Å)	$c = 18.5218$ (8)
α /deg	90
β /deg	102.388 (2)
γ /deg	90
Volume (Å^3)	4226.2(3)
Z	4
Calculated density (Mg m^{-3})	1.334
Absorption coefficient (mm^{-1})	0.461
F(000)	1764
Crystal size (mm^3)	0.1 × 0.2 × 1.0
range for data collection	1.70° to 22.02°
Reflections collected	16711
Independent reflections	5175 ($R_{\mathrm{int}} = 0.0532$)
Completeness to theta = 22.02	99.9%
Refinement method	Full-matrix least-squares on F2
Data/restraints/parameters	5175/0/545
Limiting indices	$-12 \leq h \leq 12, -20 \leq k \leq 20, -18 \leq l \leq 19$
Goodness-of-fit on F2	1.023
Final R indices [$I > 2$ sigma(I)]	$R^1 = 0.0509$, $wR^2 = 0.1224$
R indices (all data)	$R^1 = 0.0828$, $wR^2 = 0.1397$
Extinction coefficient	0.0000 (2)
Largest diff. peak and hole (eA^{-3})	0.744 and −0.346

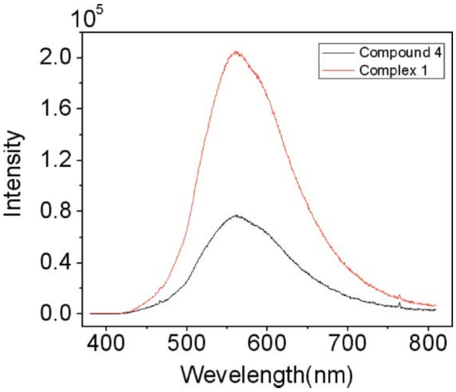


Figure 6. Solid-state emission spectra for compound **4** and complex **1** at rome temperature (Color figure available online).

Table 2. Selected bond lengths [Å] and angles [deg (°).] for complex **1.** Symmetry code for complex 1: #1 $x - 1, y, z$, #2 $x + 1, y, z$

Distances			
Co(1)–N(1)	2.092(4)	$Co(1)–O(6)^{\#1}$	2.207(3)
Co(1)–O(1)	2.093(3)	$Co(1)–O(2)$	2.214(3)
Co(1)–N(2)	2.102(4)	$Co(1)–C(22)^{\#1}$	2.500(5)
Co(1)–O(5)$^{\#1}$	2.107(3)	$O(6)–Co(1)^{\#2}$	2.207(3)
C(22)–Co(1)$^{\#2}$	2.500(5)	$O(5)–Co(1)^{\#2}$	2.107(3)
Angles			
N(1)–Co(1)–O(1)	149.02(14)	$O(6)^{\#1}–Co(1)–O(2)$	106.47(13)
N(1)–Co(1)–N(2)	100.96(16)	$C(37)–N(1)–Co(1)$	122.4(4)
O(1)–Co(1)–N(2)	96.79(14)	$C(41)–N(1)–Co(1)$	121.1(4)
N(1)–Co(1)–O(2)	93.64(14)	$C(46)–N(2)–Co(1)$	122.7(4)
O(1)–Co(1)–O(2)	59.85(12)	$C(42)–N(2)–Co(1)$	120.4(4)
N(2)–Co(1)–O(2)	93.69(15)	$C(21)–O(1)–Co(1)$	92.9(3)
O(5)$^{\#1}$–Co(1)–O(2)	160.42(13)	$C(21)–O(2)–Co(1)$	87.6(3)

X-ray Crystal-structure Analysis of Complex 1

The single crystal data of the Co(bnbc)(C_5H_5N)$_3$ (complex **1**) was collected on a Bruker Smart Apex II CCD diffractometer using the graphite monochromated Mo K radiation ($\lambda = 0.71073$ Å). The data of complex **1** were collected at 296 K. A total of 16711 reflections, including 5175 unique reflections ($R_{\text{int}} = 0.0532$) were measured in the 1.70° $< \theta <$ 22.02°. The structure was solved with a direct method using SHELXS-97 and was refined by full-matrix least-square methods using SHELXTL-97. All H atoms were placed geometrically. The crystallographic data and structure experimental details of the complex **1** were given in Table 1, and selected bond lengths and bond angles were presented in Table 2.

Conclusions

A novel metal-organic framework has been synthesized and characterized. IR spectra indicates the coordination mode of carboxyl group is chelating bidentate with the Co^{2+}ion. Crystal structural analysis reveals that complex **1** adopts a 1D infinite line structure, the complex **1** is further assembled into a 2D supramolecular network through the hydrogen bonds. TGA shows that complex **1** is highly thermally stable up to 150°C. PXRD of complex **1** shows its purity of phase. Meanwhile, the photoluminescent property of this coordination polymer has better luminescent activity.

Acknowledgments

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Cambridge, CB21EZ, UK (fax: + 44 1223 336033; email: deposit@ccdc.cam.ac.ukor www:http://www.ccdc.cam.ac.uk).

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