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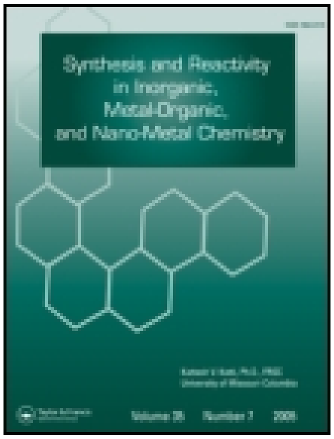

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Hydrogen Bonded Supramolecular Framework in Inorganic-Organic Hybrid Compounds: Syntheses, Crystal Structures, and Photoluminescent Properties

Li Yan [a], Yufei Xiao [a], Chuanbi Li [a], Meiling Liu [a], Wenyu Li [a] & Nan Yang [a]
[a] College of Chemistry, Jilin Normal University, Siping, P. R. China

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Hydrogen Bonded Supramolecular Framework in Inorganic-Organic Hybrid Compounds: Syntheses, Crystal Structures, and Photoluminescent Properties

Li Yan, Yufei Xiao, Chuanbi Li, Meiling Liu, Wenyu Li, and Nan Yang
College of Chemistry, Jilin Normal University, Siping, P. R. China

Two novel compounds [Zn(bip)(BDC)] (1) and [Co(bip)$_2$Cl$_2$·4H$_2$O] (2) [bip = 2-(4-bromophenyl)-1H-imidazo[4,5-f][1,10]phenanthroline, BDC = phthalate acid] have been synthesized by hydrothermal reactions and characterized by elemental analysis, thermogravimetric (TG) analysis, infrared spectrum (IR), and single-crystal X-ray diffraction. Single-crystal X-ray diffraction reveals that compounds 1 and 2 are zero-dimensional (0D) architecture, and the existence of hydrogen bonds and π-π interactions lead the 0D to 2D novel frameworks. Hydrogen bonds and π-π interactions are powerful interactions for directing supramolecular architectures. TG analysis shows clear courses of weight loss, which corresponds to the decomposition of different ligands. The luminescent properties for the ligand bip and compounds 1–2 are also discussed.

Keywords crystal structures, fluorescence, thermal stability analysis

INTRODUCTION

During the past decades, metal–organic frameworks (MOFs) are of enormous interest because of their potential applications in gas storage, separation, catalysis, nonlinear optics and so on.[1–6] Nowadays, the method of hydrothermal synthesis is widely used to synthesis novel crystal structures, because it can solve the problems of ligands solubility and enhance the reactivity of reactants in the crystallization process to obtain perfect crystal. However, it is still a great challenge to rationally prepare and predict the exact structures and composition of target products in crystal engineering, because the supramolecular structures are dependent on a number of parameters such as the nature of the metal-ligand bonds, coordination geometries of the metal centers, the ligating topologies of the ligands used, the metal-ligand ratio, the counterions and the experimental conditions such as solvent, temperature, pH value and crystallization process.[7,8] In reported MOFs, *N*-heterocyclic ligands, such as 2,2′-bipyridine, 4,4′-bipyridine, 1,10-phenanthroline, and their substituted derivatives played an important role in the rational design to obtain anticipant structures and desired properties. Our strategy of rational design and synthesis of coordination polymers is to employ a new designed long-conjugated unsymmetrical ligand bip (2-(4-bromophenyl)-1H-imidazo[4,5-f][1,10]phenanthroline) (as shown in Scheme 1): in view of the following characteristics, (a) it possesses extended long-conjugated unsymmetrical aromatic system to provide supramolecular interactions; (b) it has two chelating nitrogen atoms, which is similar with 2,2’-bipyridyl-like bidentate chelating ligands; and (c) it possesses strong and rigidity of coordination ability to metal atoms to construct intriguing structures.[9] For the past several years, we have worked on the synthesis of *N*-heterocyclic compounds.[10–12] However, the investigation for this type of ligands is not enough. Based on the previous reasons, we design and synthesize of the polymers, namely: [Zn(bip)(BDC)] **1** and [Co(bip)$_2$Cl$_2$·4H$_2$O] **2**. Compounds **1** and **2** exhibit zero-dimensional (0D) structures, and display 2D layer structures via hydrogen bonds and interchain π-π stacking interactions. The research shows that **1** and **2** are luminescent material.

EXPERIMENTAL

Materials

The ligand bip was prepared according to the description in the literature procedures.[13] The metallic salt, BDC, and NaOH were purchased commercially and used without further purification.

Physical Measurements

The FT-IR spectrum was measured with KBr pellets in the range of 4000–400 cm^{-1} on a Perkin-Elmer 240C spectrometer. TGA (thermogravimetric analysis) was performed using a Perkin-Elmer TG-7 analyzer at the rate of 10°C/min rise of temperature in nitrogen atmosphere. Crystal structures were determined on a Bruker SMART APEX II CCD X-ray diffractometer. Carbon, hydrogen and nitrogen elemental analyses

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Address correspondence to Li Yan, College of Chemistry, Jilin Normal University, Siping 136000, P. R. China. E-mail: yanli820618@163.com

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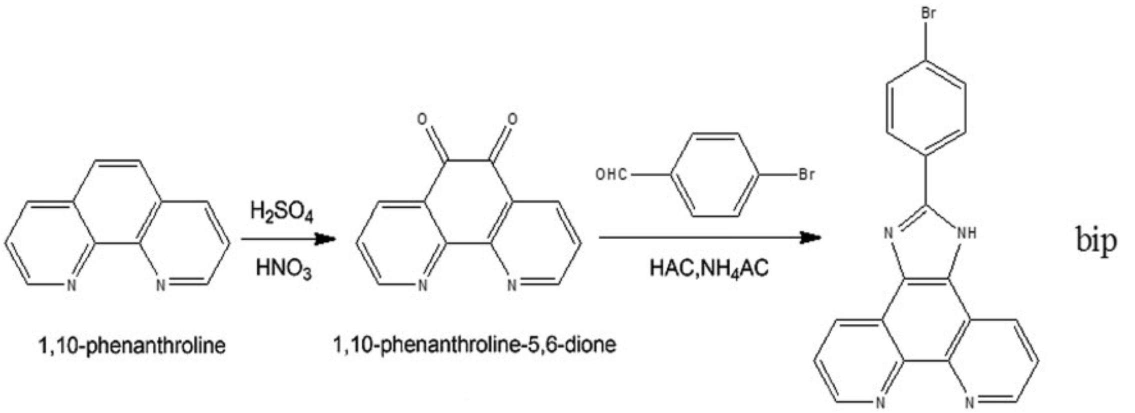


SCH. 1. The synthesis of bip.

were performed with a PE-2400 elemental analyzer. X-ray powder diffraction (XRD) measurements were performed using an X'pert diffractometer of Philips with monochromated Co-Kα radiation.

Syntheses of 1–2

*Synthesis of [Zn(bip)(BDC)] (**1**)*

A mixture of $ZnCl_2$ (0.082 g, 0.3 mmol), bip (0.112 g, 0.3 mmol), BDC (0.100 g, 0.6 mmol) and H_2O (18 mL) was stirred at room temperature and adjusted the pH value to about 7.0 with NaOH. We put the cloudy solution into a 30-mL Teflon-lined stainless vessel under autogenous pressure at 443 K for 72 h, and afterwards cooled to room temperature at a rate of 5°C/ h. The yellow crystals of compound **1** were collected in 80% yield based on Zn. $C_{54}H_{30}Br_2N_8O_8Zn_2$: Anal. Calcd. (%): C 53.62, H 2.50, N 9.26; Found: C 53.55, H 2.39, N 9.41%. IR (KBr, cm^{-1}): 1605(vs), 1566(vs), 1453(m), 1389(vs), 1061(s), 991(s), 837(m), 732(m), 538(m), 439(m).

*Synthesis of [Co(bip)$_2$Cl$_2$·4H$_2$O] (**2**)*

A mixture of $CoCl_2$ (0.114 g, 0.3 mmol), bip (0.112 g, 0.3 mmol), and H_2O (18 mL) was stirred at room temperature and adjusted the pH value to about 7.0 with NaOH. We put the cloudy solution into a 30-mL Teflon-lined stainless vessel under autogenous pressure at 443 K for 72 h, and afterwards cooled to room temperature at a rate of 5°C/ h. Small red crystals of compound **2** were collected in 85% yield based on Co. $C_{19}H_{15}BrClCo_{0.50}N_4O_2$: Anal. Calcd. (%) C 47.92, H 3.17, N 11.76; Found: C 48.05, H 3.36, N 11.52%. IR (KBr, cm^{-1}): 3372(s), 1701(s), 1605(s), 1459(s), 1277(s), 837(s), 725(s), 656(m), 580(m), 439(m).

X-Ray Crystallography

Single-crystal X-ray diffraction data were collected at room temperature with a Bruker SMART APEX II CCD diffractometer equipped with a graphite-monochromatized MoKα radiation ($\lambda = 0.71073$ Å) at 293(2) K in the range of $1.67 \leq \theta \leq 25.67°$ for **1** and $1.46 \leq \theta \leq 25.05°$ for **2**. Absorption corrections were applied using multi-scan technique and all the structures were solved by direct methods with SHELXS-97[14] and refined with SHELXL-97[15] by full-matrix least-squares techniques on F^2. Non-hydrogen atoms were refined with anisotropic temperature parameters. Experimental details for crystallographic data and structure refinement parameters for **1** and **2** are listed in Table 1.

RESULTS AND DISCUSSION

Description of Crystal Structures

*[Zn(bip)(BDC)$_2$] (**1**)*

The molecular structure of compound **1** is shown in Figure 1. The 1D chain structure linked hydrogen bonds is suggested in Figure 2, and the 2D layer structure linked hydrogen bonds is suggested in Figure 3. Selected important bond lengths and bond angles are given in Table 2.

As shown in Figure 1, the asymmetric unit contains two Zn (II) atoms, two bip ligands, and two BDC ligands. The Zn (II) atom is penta-coordinated with three carboxylate oxygen atoms (O(3), O(4) from one chelating BDC ligand, O(1) from another bridging BDC ligand) and two nitrogen atoms donors (N(1), N(2) from one chelating bip ligand).

The bond distances of Zn–O in compound **1** are from 1.958(5) to 2.373(6) Å, and those of Zn–N bond distances fall in the 2.069(5) to 2.084(6) Å range, which are similar with the values reported.[16–23] The N(O)–Zn–O(N) angles range are from 87.2(2) to 149.1(2)°.

As showed in the Figure 1, the BDC ligand coordinates to two Zn (II) ions through three carboxylic oxygen atoms in mono-bridging and bidentate chelating fashion, which give rise to a 0D structure. It is interesting that in the unit of compound **1**, 14-membered ring is formed, in which the angle of O(1)–Zn(1)–O(4) is 104.392°; *N*-heterocyclic ligands bip are attached to both sides of this chain regularly, and the ligands on the same side are parallel nearly.

Hydrogen bonding interactions are usually important in the synthesis of supramolecular architectures, and the most

TABLE 1
Crystal data and details of structure refinement parameters for **1** and **2**

	1	2
Empirical formula	$C_{54}H_{30}Br_2N_8O_8Zn_2$	$C_{19}H_{15}BrClCo_{t0.50}N_4O_2$
Formula weight	1209.42	476.18
Crystal system	monoclinic	orthorhombic
Space group	*C*2/*c*	*Pbcn*
a (nm)	1.57481(1)	1.47427(11)
b (nm)	2.4457(3)	0.89536(7)
c (nm)	1.39872(1)	2.7832(2)
β (°)	119.783(2)	90
Volume (nm^3)	4.6756(8)	3.6738(5)
Z	4	8
Density (Mg/m^3) (calcd.)	1.718	1.722
Absorption coefficient (mm^{-1})	2.804	2.843
F(000)	2416	1908
Crystal size (mm^3)	0.43 × 0.35 × 0.30	0.54 × 0.42 × 0.32
Theta range (°)	1.67–25.07	1.46–25.05
Reflections collected	12506	20427
Unique reflections [R_{int}]	4136 [0.0758]	3263 [0.0624]
Goodness-of-fit on F^2	0.987	1.002
Final *R* indices [$I2\sigma(I)$]	$R1 = 0.0618$	$R1 = 0.0371$
	$wR2 = 0.1293$	$wR2 = 0.0880$
R indices (all data)	$R1 = 0.1414$	$R1 = 0.0566$
	$wR2 = 0.1604$	$wR2 = 0.0964$
Largest difference peak and hole (e. Å^{-3})	1.089, –0.746	0.472, –0.446

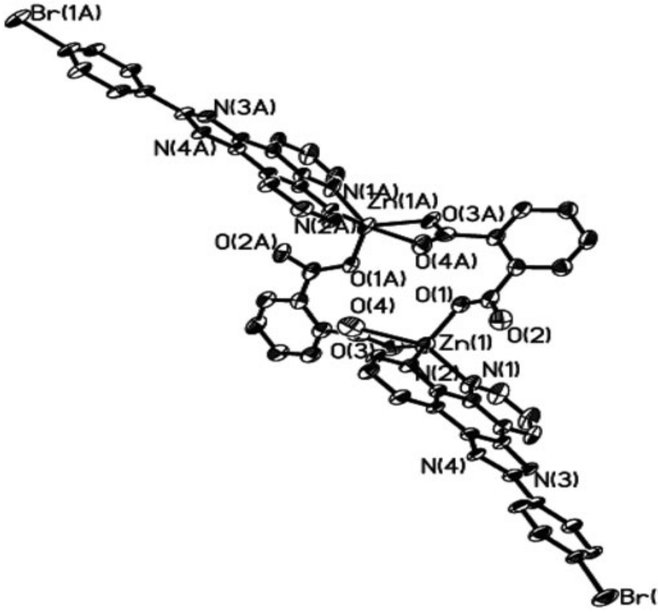


FIG. 1. The molecular structure of compound **1** (hydrogen atoms were omitted).

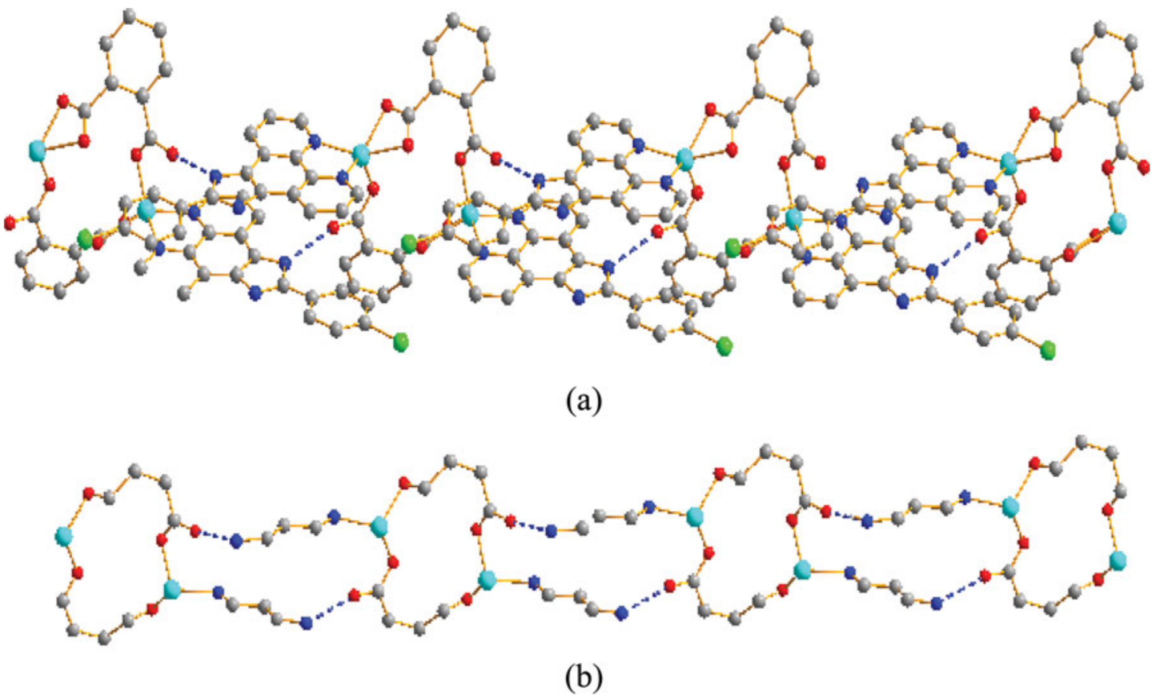
interesting aspect of the structure in **1** concerns the intermolecular N–H···O interactions. Every pair of molecules linked by intermolecular N3–H3A···O2 hydrogen bonding. The existence of N3–H3A···O2 hydrogen bonds interactions [H3A···O2 = 1.90 Å, N3···O2 = 2.743(8) Å and N3–H3A···O2 = 168°] lead the 0D zigzag chain to 1D chain infinite structure. It is interesting that in the unit of compound **1**, 18-membered rings are formed using N3–H3A···O2 hydrogen bonds interactions, and these two types of rings (14-membered ring and 18-membered ring) arranged alternately in the chain structure (see Figure 2). There is another type of Hydrogen bonding interaction C–H···O interaction, which expand 1D chain structure to 2D framework, as illustrated in Figure 3. The two parallel bip ligands in the same side recognize each other through aromatic *π*-*π* stacking interaction with the face-to-face distance being 3.499 Å.[9]

*[Co(bip)$_2$Cl$_2$·4H$_2$O] (**2**)*

The molecular structure is shown in Figure 4, and the 2D layer structure linked hydrogen bonds is suggested in Figure 5. Selected important bond lengths and bond angles are also listed in Table 2.

As shown in Figure 4, the asymmetric unit of compound **2** consists of half Co (II) atom, one bip ligand, one chloride ion, and one lattice water molecule. The coordinated chloride ions occupy two coordination points of Co (II) atom, and the Co atom is on a special position. The Co (II) atom is hexa-coordinated with four nitrogen atoms from two chelating bip ligands and two chloride ions from metal salts, furnishing a slightly distorted octahedral geometry. The angle of N(1)–Co(1)–N(2), N(2)–Co(1)–Cl(1A), Cl(1A)–Co(1)–N(2A) and N(2A)–Co(1)–N(1) is 76.98°, 92.34°, 99.41°, and 91.31°, and the sum is 360.04°(A: –x – 1, y, –z + 3/2). For the coordination environment of Co(1), the Co(1), N1, N2, N2A, Cl1A atoms define the basal plane, and the N1A, Cl1A atoms occupy the apical axial positions. The bond distance of Co–Cl in compound **2** is 2.4715 Å, and those of Co–N bond distance fall in the 2.150 to 2.165Å range, which are similar with the values reported.[24–27]

Hydrogen bonding interactions are usually important in the synthesis of supramolecular architectures, there are six type of hydrogen bonding interactions in compound **2**: N–H···Cl, C–H···Br, C–H···Cl, C–H···O, and the most interesting aspect of the structure in **2** concerns the intermolecular C–H···Cl



(a)

(b)

FIG. 2. 1D chain structure linked by N–H···O hydrogen bonds of compound **1** (hydrogen atoms were omitted) (a), two types of rings in the chain structure(b).

[H(8A)···Cl(1) = 2.70 Å, C(8)···Cl(1) = 3.587 Å and C(8)–H(8A)···Cl(1) = 159°] interactions, which help in the construction of the 2D layer structure (Figure 5). Moreover, there are π-π interactions between the aryl ring of bip ligands and symmetry of the two adjacent equivalent of aromatic ring (symmetry codes: –x, –1 + y, 1.5 – z, 0.5+x, –1.5 + y, 1.5 – z). The distance between cg(1)→cg(2) ring centroid is 3.628 Å and cg(3)→cg(4) is 3.669 Å.[9] Cg(1):C14→C15→C16→C17→C18→C19, Cg(2): C1→C2→C3→C4→C11→N1(),Cg(3): C4→C5→C6→C7→C11→C12,y Cg(4): C5→C6→C13→N3→N4.

The Zn (II) atom is penta-coordinated with three carboxylate oxygen atoms and two nitrogen atoms donors, forming distorted tetrahedron in the compound **1**. Zn (II) metal exhibits four-, penta-, or six-coordinated mode, especially for four- and six-coordinated modes are common.[28,29] The most interesting aspect of the structure in **1** concerns the intermolecular of H-bond interactions, which formed two types of rings (14-membered ring and 18-membered ring) alternately in the chain structure. The coordination environment around Co(II) center in **2** is in a distorted octahedral geometry, surrounded by four N atoms

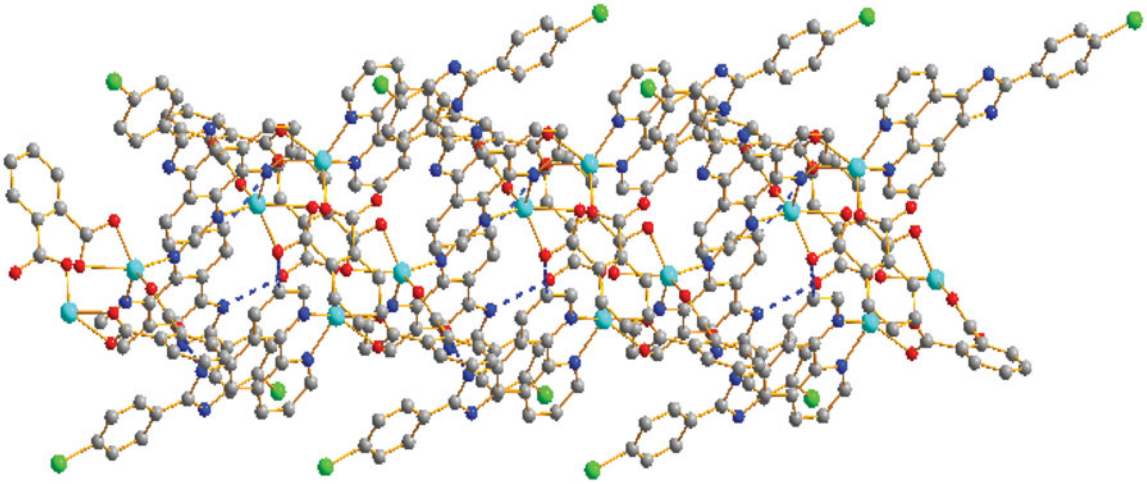


FIG. 3. 2D layer structure of compound **1** linked by N–H···O and C–H···O hydrogen bonds (dotted lines represent hydrogen bonds).

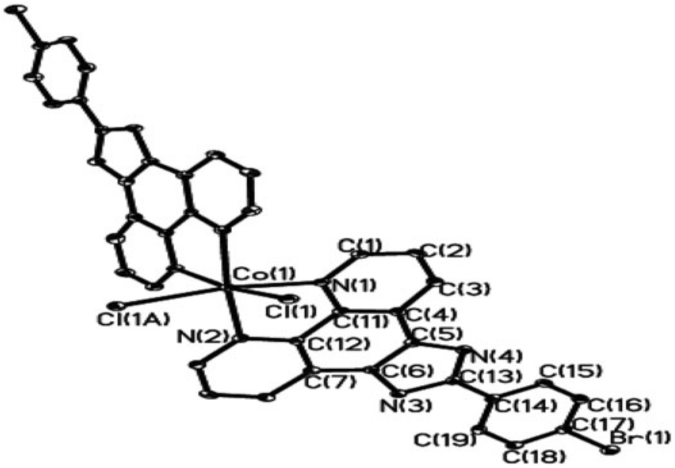


FIG. 4. The molecular structure of compound **2** (hydrogen atoms were omitted).

from two bip ligands and two terminal Cl atoms. The structures of coordinated chloride ions in **2** are symmetric.

IR Spectra

In compound **1**, the two peaks at 1605 and 1566 cm^{-1} correspond to the antisymmetric stretching vibrations of carboxyl, and 1389 cm^{-1} correspond to the symmetric stretching vibration of carboxyl. The Δv ($v_{as}(COO^-)$-$v_s(COO^-)$) are 216 and 177 cm^{-1}, indicating that the carboxyls are bidentately and monodentately coordinated with Zn (II) atoms.[30] In compound **2**, the infrared spectrum exhibited absorption at 3372 cm^{-1}, which is assigned to O–H stretching vibration.[31] The spectrum of compound **2** shows the strong bands at 1459 and 837 cm^{-1} are due to C=C and C=N stretching vibrations of the coordinated phenanthroline molecules. Below 700 cm^{-1} it seems to be a lot of bands, which correspond to the Co–N stretching mode. The IR results are good agreement with their solid structural features from the results of their crystal structures.

XRD Spectra

The simulated and experimental power XRD patterns of **1** and **2** are shown in Figure 6. The patterns calculated from the single-crystal X-ray data of **1** and **2** are in good agreement with the observed ones in almost identical peak position but with different peak intensities, which indicates the phase purity of the products. The differences in intensity may be due to the preferred orientation of the powder samples.[32,33]

Thermal Analysis

To examine the stability of compounds **1** and **2**, we examined the TG curves of crystalline samples in the flowing nitrogen atmosphere at a heating rate of 10°C/min (Figure 7). As expected, in compound **1**, the first weight loss corresponding to the removal of lattice BDC is 24.50% (calcd. 27.48%) from 137 to 451°C. The second weight loss of 65.10% from 451 to 651°C corresponds to the loss of bip ligands (calcd. 62.03%). In

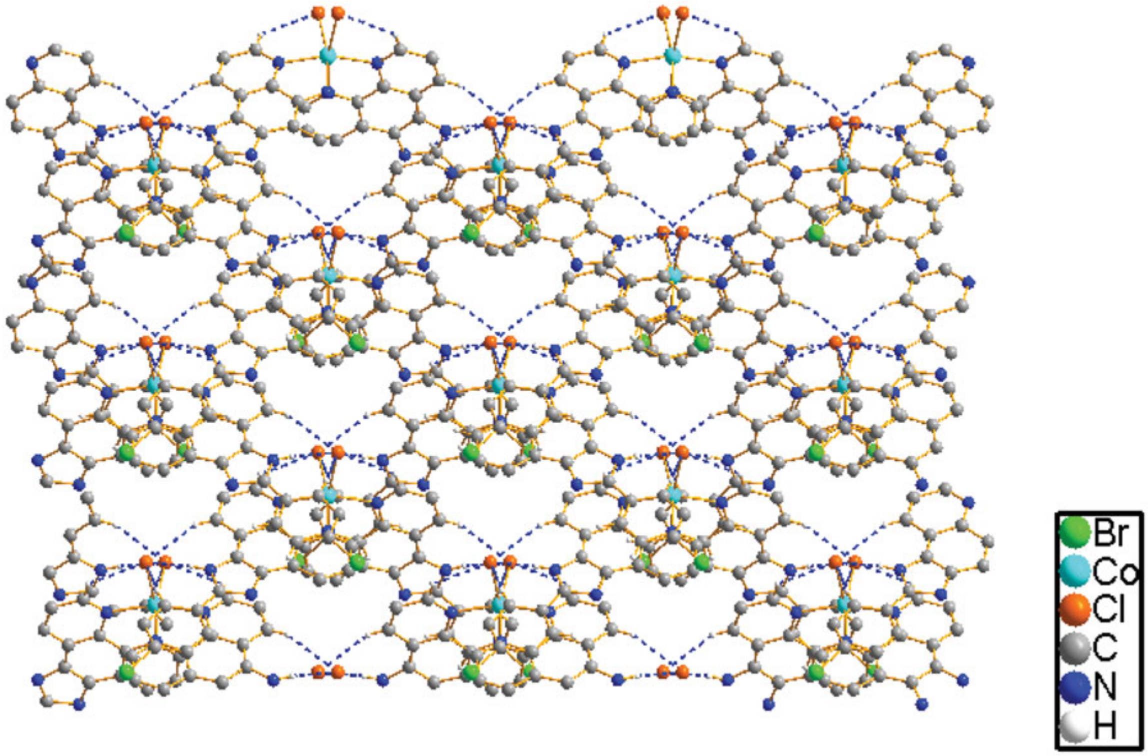


FIG. 5. 2D layer structure of compound **2** linked by C–H···Cl hydrogen bonds (dotted lines represent hydrogen bonds).

TABLE 2
Selected bond lengths (Å) and bond angles (°) for compounds **1** and **2**

1/			
Bond	Dist.	Bond	Dist.
Zn(1)–O(1)	1.958(5)	Zn(1)–O(3)	2.031(5)
Zn(1)–O(4)	2.373(6)	Zn(1)–N(1)	2.084(6)
Zn(1)–N(2)	2.069(5)		
Angle	(°)	Angle	(°)
O(1)–Zn(1)–O(3)	103.1(2)	O(3)–Zn(1)–O(4)	59.3(2)
O(1)–Zn(1)–O(4)	104.42(1)	O(1)–Zn(1)–N(1)	106.1(2)
O(3)–Zn(1)–N(1)	108.4(2)	N(2)–Zn(1)–O(4)	87.2(2)
N(1)–Zn(1)–O(4)	149.1(2)	O(1)–Zn(1)–N(2)	122.7(2)
O(3)–Zn(1)–N(2)	129.3(2)	N(2)–Zn(1)–N(1)	80.4(2)
		2	
Co(1)–N(1)	2.165(3)	Co(1)–N(2)	2.150(3)
Co(1)–Cl(1)	2.4715(1)		
Angle	(°)	Angle	(°)
N(2)$^{\#1}$–Co(1)–N(2)	163.30(17)	N(2)–Co(1)–N(1)	76.98(12)
N(2)$^{\#1}$–Co(1)–N(1)	91.31(12)	N(1)$^{\#1}$–Co(1)–N(1)	91.64(17)
N(1)–Co(1)–Cl(1)	89.91(9)	N(2)–Co(1)–Cl(1)	99.41(9)
N(1)$^{\#1}$–Co(1)–Cl(1)	169.24(8)	N(2)–Co(1)–Cl(1)$^{\#1}$	92.34(9)
Cl(1)$^{\#1}$–Co(1)–Cl(1)	90.55(5)		

Symmetry transformations used to generate equivalent atoms: (2) $^{\#1}$ –x – 1, y, –z + 3/2.

compound **2**, the first weight loss of 16.50% corresponds to the loss of chloride ions and two water molecules (calcd. 15.01%). The second weight loss in the temperature range of 245–595°C can be ascribed to the release of bip ligands (obsvd. 76.50%, calcd. 78.77%).

Photoluminescent Properties

In this study, we research the luminescence of compounds **1** and **2**, as well as the free ligand bip (Figure 8). The free ligand exhibits emission at 579 nm (excitation at 325 nm) for bip. Compound **1** shows one broad emission band with the maximum intensity at 495 nm upon excitation at 280 nm, which is blue-shifted by 84 nm relative to the emission wavelength of free ligand bip. Compound **2** exhibits one broad strong emission band with the maximum intensity at 395 nm upon excitation at 247 nm, which is blue-shifted by 184 nm relative to the emission wavelength of free ligand bip. It is electronic that the blue-shifted emission of **1** and **2** originated from the π-π^* electronic transition of bip ligand.[34] It is worth to mention that luminescent intensity of compounds **1** and **2** is weaker than the ligand bip. The reason for that is probably attributed to the quenching effect.[35] The coordination polymers **1** and **2** may be good candidate for potential photoluminescence material.

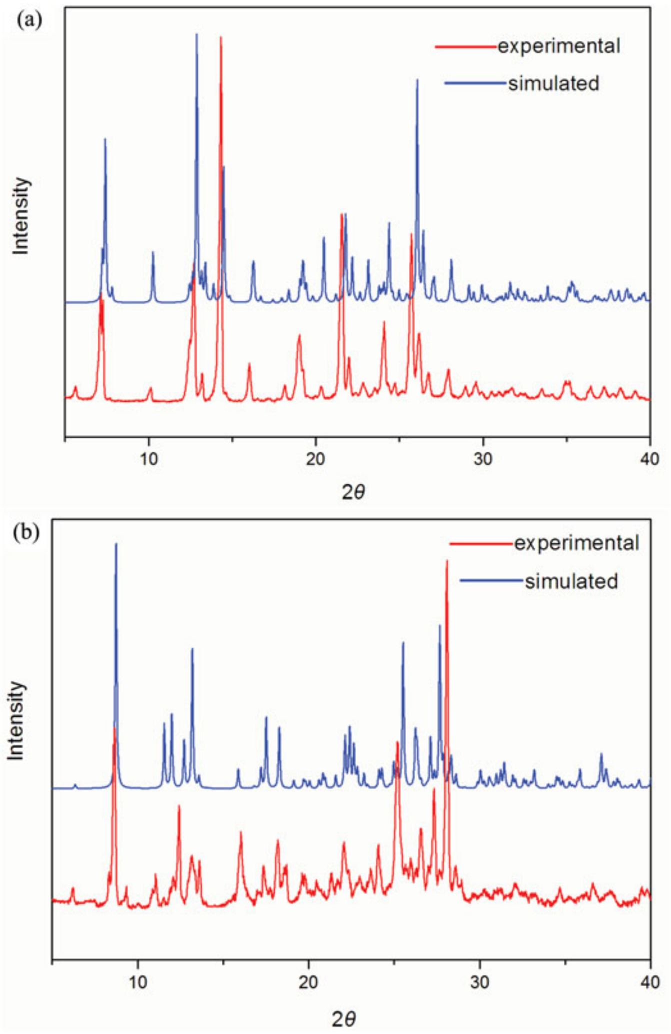


FIG. 6. Experimental and simulated X-ray powder diffraction patterns of **1** (a) and **2** (b).

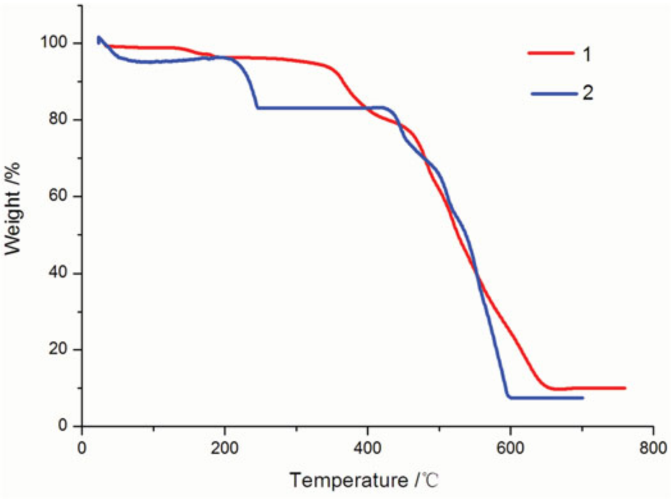


FIG. 7. TG curves of compounds **1** and **2**.

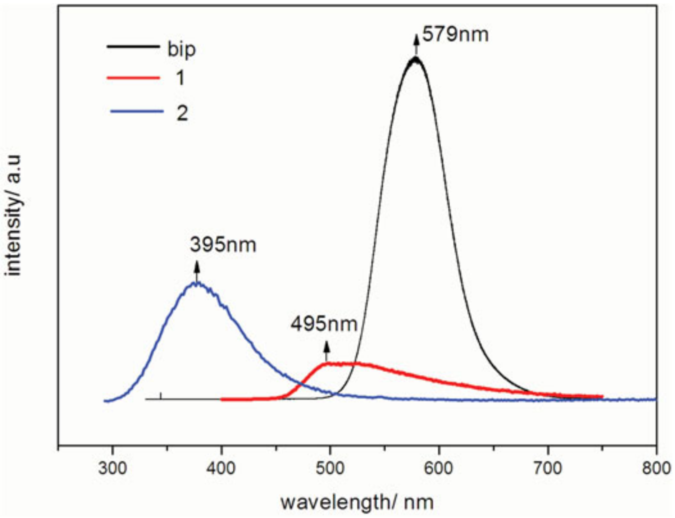


FIG. 8. Luminescent spectrum of ligand bip and compounds **1** and **2** in solid state at room temperature.

CONCLUSIONS

In conclusion, two compounds have been synthesized by using plane multifunctional ligands bip, BDC, and metallic salt. The results reveal that the title compounds are worthy of further study as candidate of potential photoluminescent material.

FUNDING

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SUPPLEMENTAL MATERIALS

CCDC 894755 and 894754 contain the supplementary crystallographic data for **1** and **2**, respectively. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/datarequest.cif.

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