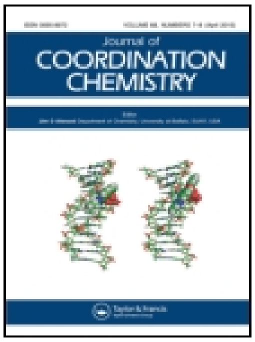

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Self-assembly of 1-D and 3-D transition-metal coordination polymers based on 4-(1H-l,3-benzimidazol-2-yl)pyridine 1-oxide

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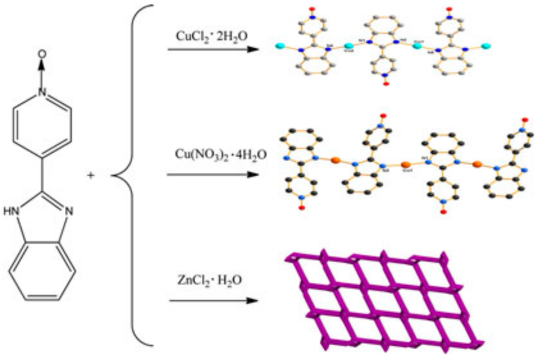

Three metal-organic coordination polymers based on 4-(1H-l,3-benzimidazol-2-yl)pyridine 1-oxide (BImPyO) with the molecular structures $[Cu_2(C_{12}H_8N_3O)_2]_n$ (**1**), $[Cu(C_{12}H_8N_3O)]_n$ (**2**), and $[Zn(C_{12}H_8N_3O)Cl]_n$ (**3**) were synthesized under hydrothermal conditions. They showed diverse coordination modes and were further characterized by elemental analysis, infrared spectroscopy, and single crystal X-ray structure analysis, respectively. In **1** and **2**, BImPyO generated a 1-D chain by adopting μ_2-kN : kN′ coordination to bridge two Cu(II) ions with bis-N-chelation. In **3**, by adopting μ_3-kN : kN′:kO coordination, BImPyO bridged three crystallographically independent Zn(II) ions to form a 3-D framework; **3** exhibits strong fluorescent emission in the solid state at room temperature.

Keywords: Coordination polymers; Crystal structure; 4-(1H-l,3-benzimidazol-2- yl)pyridine 1-oxide; Hydrothermal condition; Luminescent property

1. Introduction

During the past several decades, design and synthesis of metal-organic coordination polymers have attracted interest not only because of their intriguing structural motifs but also the potential application in catalysis, conductivity, luminescence, molecular magnetism, gas

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*Corresponding author. Email: jkdzgy@just.edu.cn storage, etc. [1–8]. Most of the reported materials consist of metal ions as connecting centers and multidentate polyfunctional-bridging ligands as linkers [9, 10]. Recent efforts have been devoted to the design of suitable ligands to construct new coordination polymers. Heterocyclic organic ligands such as benzotriazoly-, tetrazoly-, pyridine-, and pyrazole- as building blocks have attracted much attention [11–18]. Among these heterocyclic ligands, 4-(1H-l,3-benzimidazol-2 -yl)pyridine 1-oxide(BImPyO) has been attractive since the conjugate aromatic nucleus will contribute to interesting structures through aromatic π–π stacking interactions [19–22]. Furthermore, as a bridging ligand, BImPyO has three donors allowing formation of high dimensional structures, from which the dihedral angle between the benzimidazole ring and the pyridine ring can be formed in various angles. Frameworks based on BImPyO have rarely been studied. Therefore, we chose BImPyO as building block to construct metal-organic coordination polymers with new structural features. Herein, we report the syntheses and crystal structures of three metal-organic polymers based on BImPyO with the molecular structures $[Cu_2(C_{12}H_8N_3O)_2]_n$ (**1**), $[Cu(C_{12}H_8N_3O)]_n$ (**2**), and $[Zn(C_{12}H_8N_3O)Cl]_n$ (**3**).

2. Experimental section

2.1. *Materials and instruments*

All chemicals except BImPyO were obtained from Shanghai Jingchun Scientifical Company of reagent grade and used without purification. BImPy was prepared by the method reported previously [23]; BImPyO ligand was prepared as given below. Infrared spectra were recorded on a SHIMADZU IRprestige-21 FTIR-8400S spectrometer from 4000 to 400 cm^{-1}. Elemental analyses were taken on a Perkin–Elmer 240C elemental analyzer. The solid-state fluorescent spectra were recorded on a SHIMADZURF-5301PC.

2.2. *Preparation of BImPyO*

In a 100 mL flask, isonicotic acid (12.3 g, 0.10 mol) was dissolved in acetic acid (30 mL) with a magnetic stirrer at room temperature. Then hydrogen peroxide (30 mL, 30%) was added, and the mixture was heated to reflux for 20 h with appearance of a white solid. After cooling, the precipitate was collected with suction filtration, washed with water, and dried at 60 °C affording 10.5 g N-oxide isonicotic acid with 75.5% yield.

1, 2-Diaminobenzene (5.4 g, 0.05 mol) and N-oxide isonicotic acid (6.97 g, 0.05 mol) were suspended in polyphosphoric acid (30 mL) with a mechanic stirrer, and this suspension was heated in a oil bath at 220 °C for 8 h. The cooled mixture was poured into ice water (50 mL) and the resulting solution was then neutralized to pH 7 with 10% NaOH aqueous solution. The precipitated product was filtered, washed with water, and dried. The final BImPyO was recrystalized from ethanol giving 6.2 g *p*-BIPO as pale yellow powder in 56.0% yield. IR spectra of BImPyO: 3650(br), 1676(m), 1591(w), 1541(w), 1494(m), 1478(s), 1462(s), 1438(s), 1380(w), 1318(m), 1285(m), 1234(s), 1182(s), 1153(w), 1121(m), 1041(w), 966(m), 846(s), 809(s), 749(s), 678(w), 648(w), 543(m), 521(m), 479(w), 439(w).

2.3. *Preparation of 1*

BImPyO (0.1 mmol, 0.021 g), $CuCl_2 \cdot 2H_2O$ (0.1 mmol, 0.017 g), 12 mL of H_2O, and 3 mL of DMF were placed in a 25 mL Teflon-lined stainless-steel vessel, and then the mixture was heated to 125 °C for 24 h. After that, the reaction system was slowly cooled down (a descent rate of 5 °C h^{-1}) to room temperature. Light yellow bulk crystals were obtained, washed with distilled water, and dried in air. Yield of 49% (0.013 g based on BImPyO). Anal. Calcd (%) for $C_{24}H_{16}Cu_2N_6O_2$ for: C, 52.65; H, 2.95; N, 15.35; Found: C, 52.61; H, 2.90; N, 15.38. IR spectra of **1**: 3447(s), 2922(w), 2846(w), 2026(m), 1630(m), 1544(m), 1494(w), 1385(w), 1257(w), 1121(m), 1172(m), 1104(w), 1020(w), 935(w), 859(s), 674(m), 572(w), 523(m).

2.4. *Preparation of 2*

A mixture of $Cu(NO_3)_2{\cdot}3H_2O$ (0.1 mmol, 0.024 g), BImPyO (0.1 mmol, 0.021 g), 4.5 mL of H_2O, and 9 mL of ethanol were placed in a 25 mL Teflon-lined stainless-steel vessel, and then the mixture was heated to 125 °C for 24 h. After that, the reaction system was slowly cooled (a descent rate of 5 °C h^{-1}) to room temperature. Light green bulk crystals were obtained, washed with distilled water, and dried in air. Yield 51% (0.014 g based on BImPyO). Anal. Calcd (%) for $C_{12}H_8CuN_3O$ for: C, 52.65; H, 2.95; N, 15.35; Found: C, 52.69; H, 2.91; N, 15.29. IR spectra of complex **2**: 3413(s), 3108(w), 3032(s), 2948(w), 1603(w), 1510(m), 1460(s), 1426(s), 1384(s), 1265(s), 1172(m), 1104(w), 1020(w), 1112 (w) 918(w), 858(m), 809(m), 750(s), 648(m), 580(w), 525(m).

2.5. *Preparation of 3*

A mixture of $ZnCl_2$ (0.1 mmol, 0.014 g) and BImPyO (0.1 mmol, 0.021 g) was placed in a thick Pyrex tube (ca. 20 cm in length). After ethanol (0.5 mL) and water (1.7 mL) were added, the tube was frozen with liquid N_2, evacuated under vacuum, and sealed with a torch. The tube was heated at 150 °C for three days. After being cooled slowly to room temperature, colorless crystals of **3** were isolated manually, washed with distilled water, and dried in air. Yield 51% (0.016 g based on BImPyO). Anal. Calcd (%) $C_{12}H_8ZnN_3OCl$ for: C, 46.34; H, 2.59; N, 13.51; Found: C, 46.31; H, 2.62; N, 13.48. IR spectra of complex **3**: 3501(s), 3225(s), 3103(m), 3072(m), 1621(m), 1597(m), 1535(m), 1469(s), 1437(s), 1376 (w), 1325(m), 1285(w), 1264(m), 1232(s), 1195(s), 1153(m), 1125(m), 1042(w), 1005(w), 985(m), 945(w), 858(s), 808(s), 752(s), 654(s), 613(s), 547(s).

2.6. *Single-crystal X-ray diffraction measurements*

Single crystals **1**–**3** were selected respectively for data collection on a Bruker SMART APEX-II CCD diffractometer equipped with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) using an ω scan mode at room temperature. The absorption correction was carried out by the multiscan method. The structures were solved by direct method with SHELXS-97 and refined by full-matrix least-squares on F^2 with SHELXL-97 [24]. Topology information for **3** was obtained using TOPOS 4.0 [25]. All non-H atoms were refined anisotropically. All hydrogens attached to C and N were added theoretically and refined with a riding model and fixed isotropic thermal parameters. Detailed data collection and refinement of **1**−**3** are summarized in table 1. The hydrogen bond distances of BImPyO are listed in table 2. The selected bond distances and angles of **1**−**3** are listed in table 3.

Table 1. Data collection and processing parameters for **1−3** and BImPyO ligand.

	Complex **1**	Complex **2**	Complex **3**	Ligand
Formula	$C_{24}H_{16}Cu_2N_6O_2$	$C_{12}H_8CuN_3O$	$C_{12}H_8ClN_3ZnO$	$C_{12}H_{13}N_3O_3$
M_r	547.51	273.76	311.05	247.25
Temperature (K)	296(2)	293(2)	293(2)	293(2)
Crystal system	Monoclinic	Monoclinic	Orthorhombic	Monoclinic
Space group	$P2_1/n$	$P2_1/n$	$Fdd2$	$P2_1/n$
a (Å)	13.868(9)	5.768(3)	20.735(7)	6.818(2)
b (Å)	7.895(15)	11.330(60)	30.647(10)	9.312(3)
c (Å)	18.968(2)	15.505(9)	7.379(2)	18.510(6)
α (°)	90	90	90	90.000
β (°)	92.393(9)	90.397(7)	90.000(0)	96.340(5)
γ (°)	90	90	90	90.000
V (Å^3)	2075.0(2)	1013.3(10)	4689.0(30)	1168.0(7)
Z	4	4	16	4
ρ_{calcd} (g cm^{-3})	1.753	1.794	1.763	1.407
μ (mm^{-1})	2.088	2.137	2.311	0.104
Total, unique	14,306, 3649	1783, 1598	2057, 1117	9721, 2635
GOF on F^2	1.019	1.172	0.996	0.959
R_1, wR_2 [$I > 2\sigma(I)$]	0.0773, 0.1991	0.0774, 0.1932	0.0388, 0.0852	0.0461, 0.1146
R_1, wR_2 (all data)	0.1063, 0.2356	0.0832, 0.1997	0.0440, 0.0884	0.1022, 0.1563

Table 2. Hydrogen bond lengths (Å) and bond angles (°) of ligand.

D−H···A	d(D−H)	d(H···A)	d(D···A)	(DHA)
N1−H1···O1 W	0.95(3)	1.88(3)	2.805(3)	166(3)
O1 W−H1A···O2 W$^{\#1}$	0.91(4)	1.89(4)	2.791(4)	171(3)
O1 W−H1B···O1$^{\#2}$	0.91(4)	1.95(5)	2.847(3)	168(3)
O2 W−H2A···O1$^{\#3}$	0.93(4)	1.90(4)	2.834(3)	174(3)
O2 W−H2B···N2$^{\#4}$	0.89(4)	1.98(4)	2.863(3)	179(5)
C5−H5···O2 W$^{\#5}$	0.93(0)	2.55(0)	3.451(4)	162(0)
C12−H12···N2	0.93(0)	2.60(0)	2.911(3)	100(0)

Symmetry transformation: $^{\#1}1-x,\ 1-y,\ 1-z$; $^{\#2}x,\ 1+y,\ z$; $^{\#3}1/2+x,\ 1/2-y,\ -1/2+z$; $^{\#4}x,\ 1+y,\ -1+z$; $^{\#5}x,\ y,\ 1+z$.

Table 3. Selected bond lengths (Å) and bond angles (°) of **1−3**.

Cu(1)–N(5)$^{\#1}$	1.871(5)	Cu(2)–N(4)	1.889(5)
Cu(1)–N(2)	1.877(5)	Cu(2)–N(1)	1.892(5)
Cu(1)–N(1)	1.876(7)	Cu(1)–N(2)$^{\#2}$	1.877(7)
Zn(1)−O(1)	1.990(4)	Zn(1)−Cl(1)	2.221 (1)
Zn(1)−N(2)	2.035(4)	Zn(1)−N(3)	2.038(4)
N(5)$^{\#1}$–Cu(1)–N(2)	177.0(3)	N(4)–Cu(2)–N(1)	173.7(2)
N(1)–Cu(1)–N(2)$^{\#2}$	174.3(3)		
O(1)−Zn (1)−N(2)	106.8(2)	N(2)−Zn(1)−N(3)	98.1(2)
O(1)−Zn (1)−N(3)	111.9(2)	O(1)−Zn (1)−Cl(1)	106.0(1)
N(2)−Zn(1)−Cl(1)	118.4(2)	N(3)−Zn (1)−Cl(1)	115.5(1)

Symmetry transformation: $^{\#1}x + 1/2,\ -y + 1/2$; $z + 1/2$. ; $^{\#2}-x + 1/2,\ y - 1/2,\ -z + 3/2$.

3. Result and discussion

3.1. *Description of the crystal structure*

Single crystal X-ray diffraction reveals that BImPyO crystallizes in the monoclinic system, space group $P2_1/n$, which is shown in figure 1(a). Furthermore, the dihedral angle between benzimidazole ring plane and pyridine 1-oxide ring plane is 5.261°. The ligand structure is stabilized by intermolecular N–H···O, O–H···O and C–H···O and intramolecular C–H···N hydrogen bonds. The H-bonds forming the 2-D are shown in figure 1(b).

Complex **1** crystallizes in the monoclinic system, space group $P2_1/n$. The asymmetric unit of **1**, $[Cu_2(C_{12}H_8N_3O)_2]$, which is shown in figure 2(a), contains two Cu(II) ions and two BImPyO ligands. The structure comprises endless chains along the angled direction of about 30-degree with the *c* axis, in which Cu ions and BImPyO ligands alternate. The position of the Cu ions correspond with a pseudo-inversion center in the structure, among which, Cu1 has an angle of 177.0(3)° coordinated by N2 of one ligand and N5 of another; Cu2 is 173.7(2)° coordinated by N1 of one ligand and N4 of another. The benzimidazole ring and pyridine 1-oxide ring of BImPyO are bisected by the pseudo-mirror plane [figure 2(b)] at $z \approx 30°$, being attached to two copper ions by its two nitrogens. The C−C single bond which is connected to the benzimidazole ring and pyridine 1-oxide ring of BImPyO can rotate freely given appropriate activation energy. The two planes only rotate to a larger angle and finally form a more stable complex when Cu(II) is coordinated with BImPyO. Thus, the dihedral angles between imidazole ring plane and pyridine 1-oxide ring plane are 35.899° and 38.070°, respectively, which are comparable to that in BImPyO (5.261°). The coordination geometry around Cu is close to linear, with the average bond length of Cu−N [1.882(5) Å] comparable to those in $[Cu_2(SCN)_2(4\text{-PyHBIm})]_n$ [4-PyHBIm=2-(4-pyridyl) benzimidazole], in which the bond length of Cu1−N1A is 2.051(3) Å [26]. In addition, the distances of Cu2 ions and oxygens which link adjacent layer pyridine 1-oxide rings are 3.632(8) Å and 5.807(9) Å, respectively. There are weak interactions between Cu2 and O. The overall structure of **1** is a 1-D chain. The accumulation of 1-D chains shows a wavy shape along the *c* axis as illustrated in figure 2(c).

Complex **2** also crystallizes in the monoclinic space group $P2_1/n$. Complexes **2** and **1** have similar basic structures, but the asymmetric unit of **2** is different from **1**. As shown in figure 3(a), the asymmetric unit of **2** consists of one Cu ion and one BImPyO ligand. Complex **2** also displays a 1-D chain [figure 3(b)], with two coordinated Cu from two nitrogens and the angle of N1−Cu1−N2 is 174.3(3)°. The benzimidazole and pyridine 1-oxide rings of BImPyO are separated by the pseudo-mirror plane at $b = 0$, being attached to two copper ions via two nitrogens. In addition, the dihedral angle between imidazole ring plane and pyridine 1-oxide ring plane is 35.146°, which has spun 29.885° comparable to BImPyO. The dihedral angles are 0.753° and 2.924° shorter than that in **1**. The bond lengths of Cu1−N1 and $\text{Cu1−N2}^{\#1}$ in **2** are 1.876(7) Å and 1.877(7) Å, respectively, 0.006(7) Å shorter than the average bond lengths of Cu−N in **1**. Also the bond lengths of Cu−N in **2** are slight shorter than those found in $[Cu_2(l\text{-}SCN)_2(SCN)_2(pybzim)_2]$[pybzim=2-(2-pyridyl) benzimidazole] [1.933(3) Å−2.045(2) Å] [27] and $[Cu(Hbtc)(PyBIm)_2]_n$ [H_3btc=1,3,5-benzen etri-carboxylic acid, PyBIm=2-(4-pyridyl)benzimidazole] [2.020(1) Å−2.034(1) Å] [28].

Single crystal analysis reveals that **3** crystallizes in the monoclinic system, space group *Fdd*2 and displays a 3-D coordination framework. In addition, the asymmetric unit of **3** contains one Zn(II), one Cl^- atom, and one BImPyO. As shown in figure 4(a), each Zn is

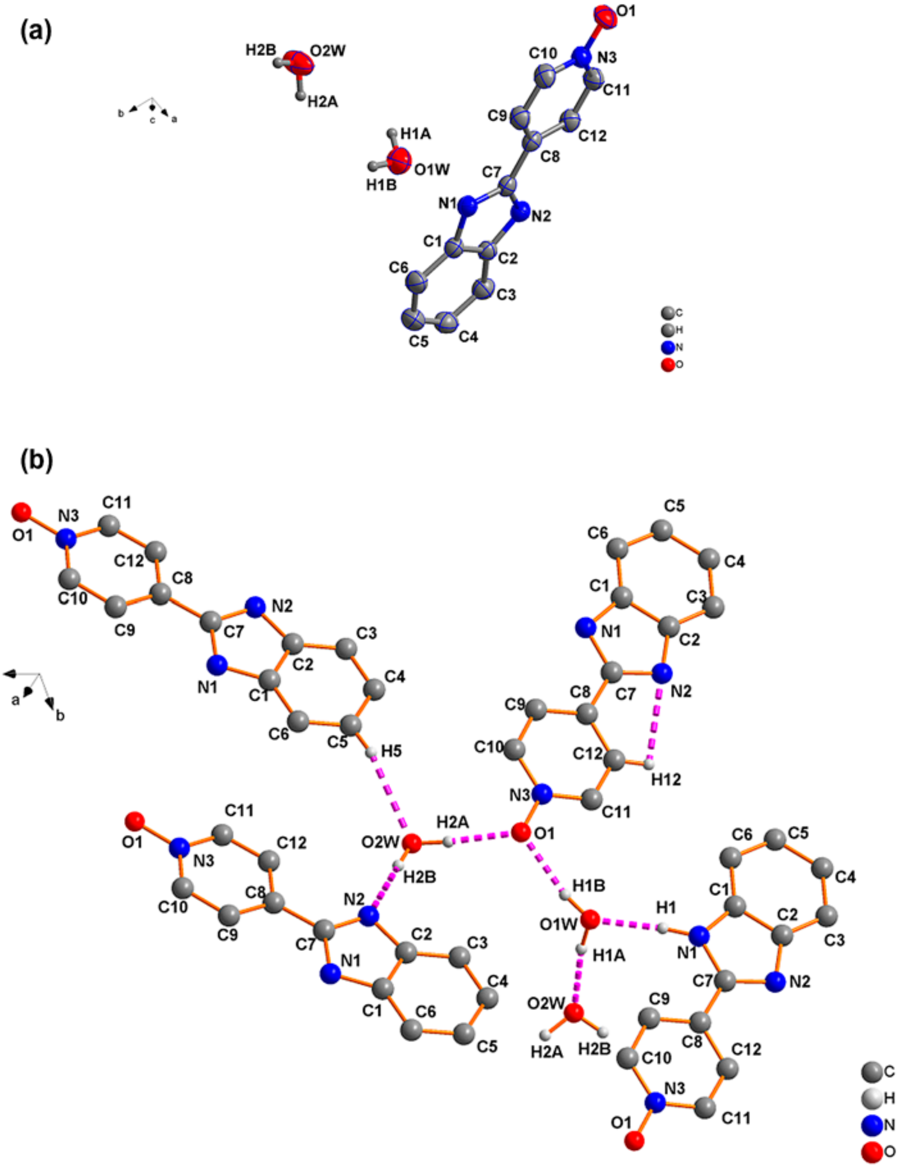


Figure 1. (a) The asymmetric unit of BImPyO dihydrate with the ellipsoids drawn at the 25% probability level. Hydrogens are omitted for clarity. (b) The intramolecular and intermolecular hydrogen bonds (dashed lines) in BImPyO forming a 2-D network.

located in a distorted tetrahedral coordination environment. Zn1 is coordinated by one chloride, one O from the monodentate pyridine N-oxide of BImPyO and two nitrogens from two different BImPyO ligands. Such coordination of Zn1 leads to a large dihedral angle between benzimidazole ring plane and pyridine 1-oxide ring plane, up to 51.591°. The bond lengths of Zn1−N2 and Zn1−N3 are 2.035(4) Å and 2.038(4) Å, respectively, comparable with those in [Zn(4-PyHBIm)(oba)] [4-PyHBIm=2-(4-pyridyl)benzimidazole, oba=4,

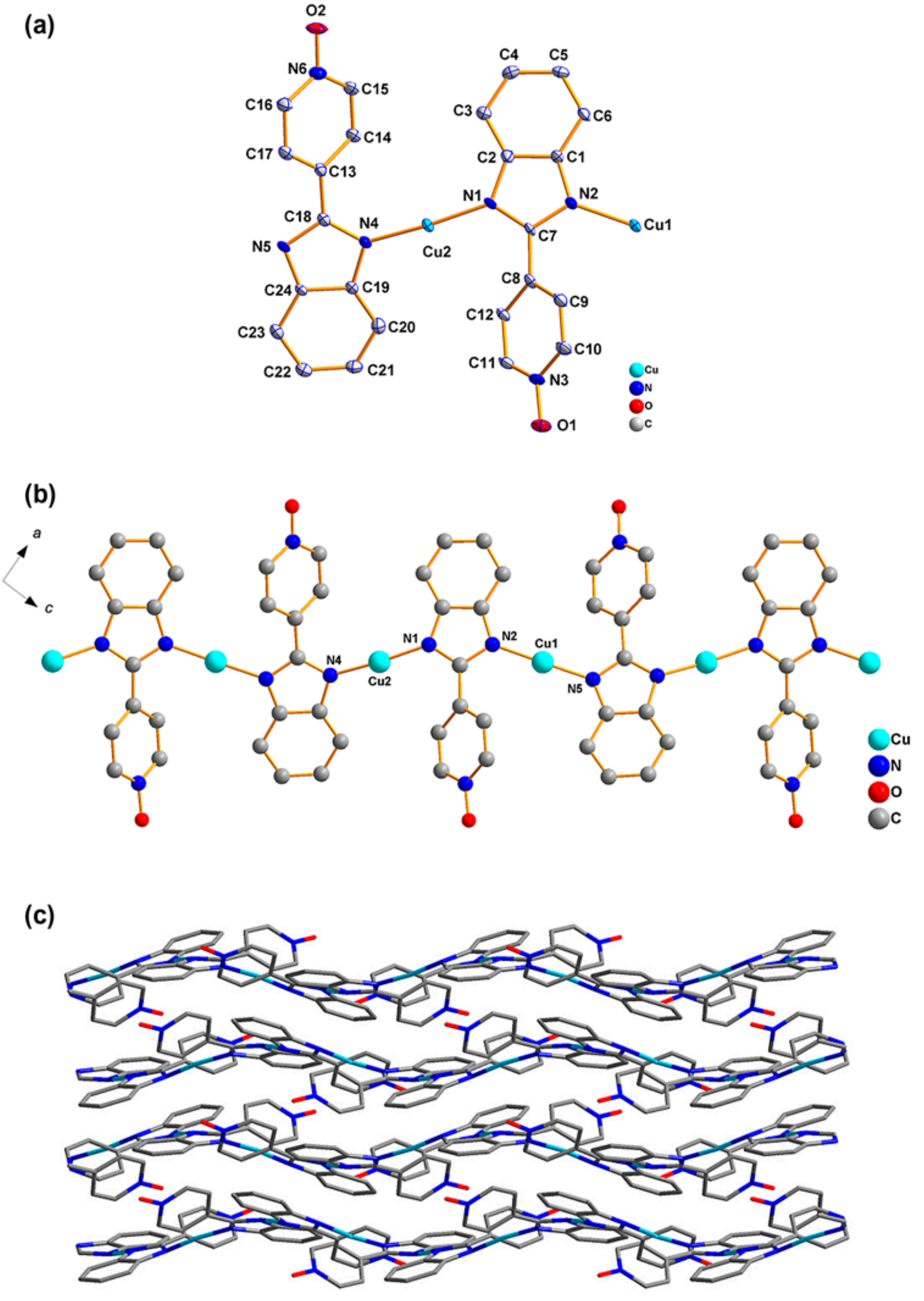


Figure 2. (a) The asymmetric unit of **1** with the ellipsoids drawn at 25% probability. Hydrogens are omitted for clarity. (b) 1-D chain of **1** viewed parallel to the *b*-axis. (c) The chain stacking along the *c*-axis.

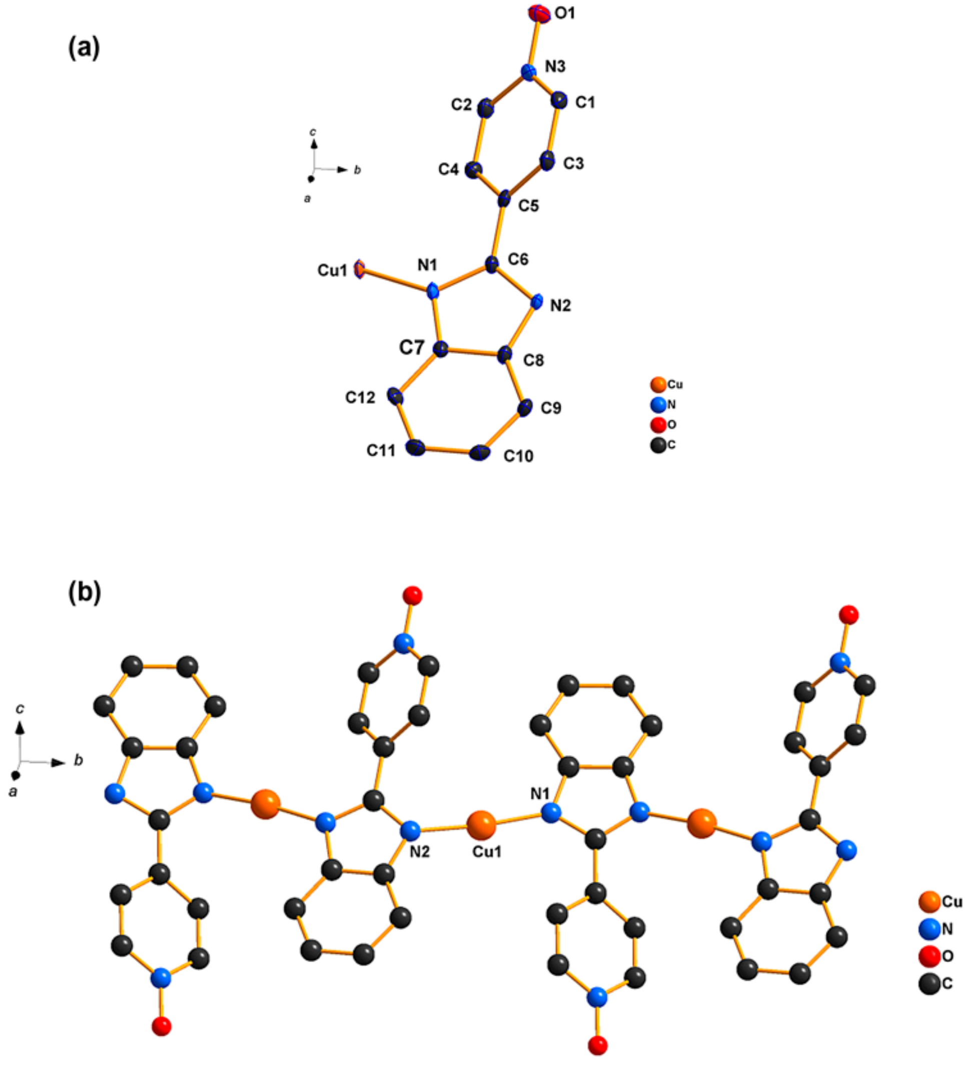


Figure 3. (a) The asymmetric unit of **2** with the ellipsoids drawn at the 25% probability level. Hydrogens are omitted for clarity. (b) 1-D chain of **2**.

40-oxybis-(benzoate)] [2.076(4) Å−2.103(4) Å] [29]. Furthermore, the bond lengths of Zn−N in **3** are similar to those found in coordination polymers based on 2-(4-pyridyl)benzimidazole ligands [30]. Moreover, the bond lengths of Zn1−O1 and Zn1−Cl1 in **3** are 1.990(4) Å and 2.221(1) Å, respectively. With Cl^- as the link center, BImPyO is tridentate, binding three Zn ions to result in the formation of a 3-D structure as shown in figure 4(b). Furthermore, **3** can be simplified as 4-connected parallelogram planar nodes (organic linker), and the assembly of these nodes generates a 3-D framework with the $8^2{\cdot}10$ topology [figure 4(c)].

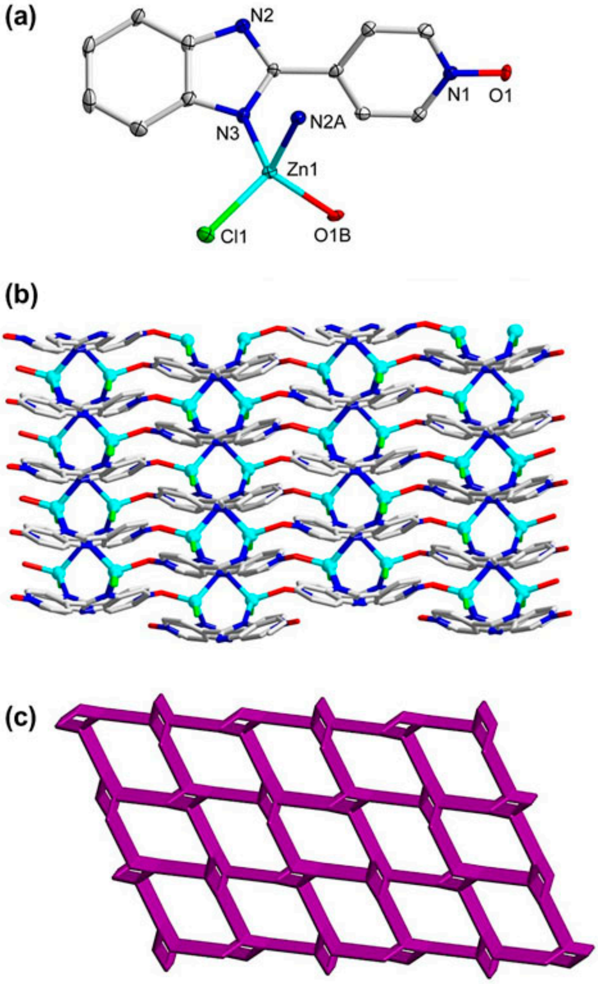


Figure 4. (a) The coordination environment of Zn(II) in **3** with the ellipsoids drawn at the 30% probability level. Hydrogens are omitted for clarity. Symmetry Code: (i) $(0.5-x, -y, -0.5+z)$, (ii) $(-0.25+x, 0.25-y, -0.25+z)$. (b) The 3-D structure of **3**. (c) The total void value of the channel is estimated (calculated by PLATON) to be 412.1 Å^3, approximately 8.8% of the total crystal volume of 4689.0 Å^3.

3.2. *Fluorescence of 3*

Aromatic organic molecules, organic polymers, and mixed inorganic–organic hybrid coordination polymers have been applied as fluorescence-emitting materials [31]. Organic materials affect emission wavelengths, so it is of significance to select proper organic spacers and metal centers (Zn, Cd, Pb, Ca, B, etc.) for the syntheses of inorganic–organic coordination polymers [32–34]. The solid-state fluorescence spectra of powdered **3** at room temperature (figure 5) show maximal emission peaks at 465 nm (with $\lambda_{ex} = 420$ nm), suggesting that **3** may be a good, visible, light-emitting material. The photoluminescent mechanism is tentatively attributed to ligand-to-ligand transitions, being in reasonable agreement with this class of metal complexes [35, 36].

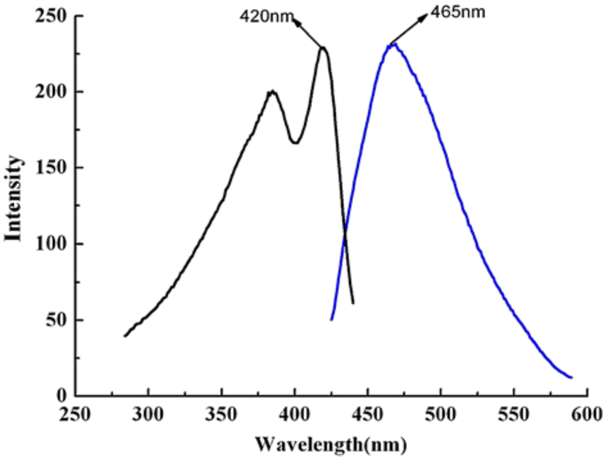
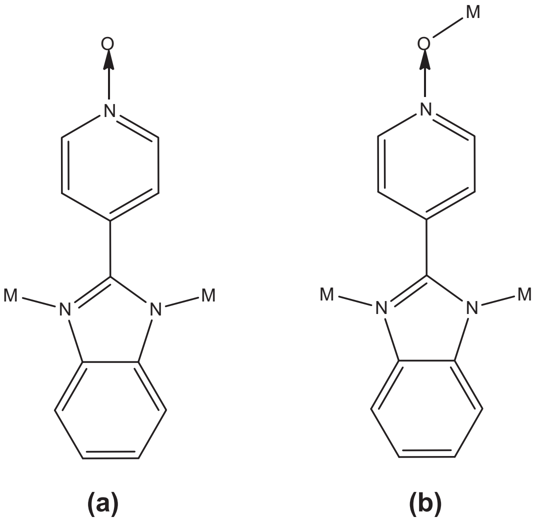


Figure 5. Solid state photoluminescence spectra of **3** at room temperature.

In conclusion, three metal-organic coordination polymers based on 4-(1H-l,3-benzimidazol-2-yl)pyridine N-oxide were successfully synthesized under hydrothermal conditions and structurally characterized. Complexes **1** and **2** were 1-D chains adopting a μ_2-kN : kN′ coordination mode. Complex **3** generated a 3-D framework having the $8^2{\cdot}10$ topology and exhibits strong fluorescent emission in the solid state at room temperature. Flexible dihedral angles between benzimidazole ring plane and pyridine N-oxide plane lead to more coordination of BImPyO, comparable to rigid ligands such as 2,6-dimethylbenzo[1,2-d : 4,5-d′]diimidazole [37]. In summary, BImPyO exhibits diverse coordination and is thus a good candidate for constructing frameworks (scheme 1).



Scheme 1. (a) Coordination modes of BImPyO in **1** and **2**. (b) Coordination modes of BImPyO ligand in **3**.

Supplementary materials

CCDC Nos. 1044330, 1044334, 1044338, and 1044457 contains the supplementary crystallographic data of ligand, **1**, **2,** and **3** for this article. These data can be obtained free of charge from the Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

Disclosure statement

No potential conflict of interest was reported by the authors.

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