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Syntheses, crystal structures, and properties of four new coordination compounds of transition metals and imidazoledicarboxylic acid derivatives

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Syntheses, crystal structures, and properties of four new coordination compounds of transition metals and imidazoledicarboxylic acid derivatives

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Four new transition metal coordination complexes, $[\text{Cd}(\text{H}_2\text{pimdc})_2(\text{H}_2\text{O})_2] \cdot 4\text{H}_2\text{O}$ (**1**), $[\text{Zn}(\text{H}_2\text{pimdc})_2(\text{H}_2\text{O})_2] \cdot 7\text{H}_2\text{O}$ (**2**), and $[\text{M}(\text{H}_2\text{pimdc})_2]$ ($\text{M} = \text{Cu}$ (**3**) or Ni (**4**), $\text{H}_2\text{pimdc}^- = 2\text{-propyl-4,5-imidazoledicarboxylate}$), have been prepared by conventional synthesis and characterized by elemental analyses, IR, TG, and single-crystal X-ray diffraction. H_2pimdc^- is a bidentate chelating ligand in **1** and **2**, leading to 3-D supramolecular structures through hydrogen bonds. However, H_2pimdc^- is a tridentate chelating-bridge ligand in **3** and **4**, which exhibit 2-D layer structures. Thermal properties and photoluminescence spectra of **1–4** were measured.

Keywords: Coordination complex; Imidazoledicarboxylic acid; Transition metal; Photoluminescence

1. Introduction

Research in metal–organic coordination polymers continues to be interesting for their intriguing architectures and topologies and potential applications in gas adsorption [1], catalysis [2], luminescence probes [3], and magnetic materials [4]. Many ligands with both N- and O-donors, such as 4,5-imidazoledicarboxylic acid (H_3imdc), have been used to construct metal–organic coordination polymers with different topologies [5–14], because it can provide different coordination modes with their six-donor sites, forming interesting structures, and can be partially or fully deprotonated to generate anions with one, two, or three charges at different pH values giving acidity-dependent coordination modes. A number of fascinating metal–organic coordination polymers have been prepared from H_3imdc . However, H_3imdc derivatives are still rarely used, with only a few examples reported [15–17]. H_3imdc derivatives with small substituents, such as a methyl or ethyl or other groups at the 2-position, may lead to the formation

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of new structures. Herein, we report the synthesis, structure, and properties of four new metal–organic complexes of 2-propyl-4,5-imidazoledicarboxylic acid (H_3pimdc): $[Cd(H_2pimdc)_2(H_2O)_2] \cdot 4H_2O$ (**1**), $[Zn(H_2pimdc)_2(H_2O)_2]_2 \cdot 7H_2O$ (**2**), and $[M(H_2pimdc)_2]$ ($M = Cu$ (**3**) or Ni (**4**)).

2. Experimental

2.1. General remarks

All chemicals were commercially purchased, of analytical reagent grade, and used without purification. Elemental analyses were performed on a PLASMA-SPEC(I) ICP atomic emission spectrometer. IR spectra were recorded from 400 to 4000 cm^{-1} on a Magna 560 FT/IR spectrophotometer using KBr pellets. Photoluminescence spectra were measured with pure solid samples at room temperature using a FL-2T2 instrument (SPEX, USA) with 450 W Xenon lamp monochromated by double grating (1200). TG analyses were performed with a Perkin Elmer TGA7 instrument in N_2 at a heating rate of $10^\circ\text{C min}^{-1}$.

2.2. Synthesis of 1–4

2.2.1. Synthesis of $[Cd(H_2pimdc)_2(H_2O)_2] \cdot 4H_2O$ (1**).** In a typical synthesis procedure for **1**, a mixture of 0.4 mmol (0.12 g) of $Cd(NO_3)_2 \cdot 4H_2O$, 0.1 mmol (0.02 g) of H_3pimdc , and 20 mL of H_2O was stirred in air at 80°C for 4 h. After 1 week, colorless block crystals of **1** were obtained in 87% yield (based upon Cd). Anal. Calcd for $C_{16}H_{28}CdN_4O_{14}$: H, 4.6; N, 9.1; C, 31.4; and Cd, 18.4. Found: H, 4.7; N, 9.0; C, 31.3; and Cd, 18.4. FT/IR (cm^{-1}): 1529vs, 1576vs, 1399s, 1272s, 1127s, 972s, 2969s, 3175m, 515m, 1058m, 3440m, 2875m, 650w, 445w, and 777w.

2.2.2. Synthesis of $[Zn(H_2pimdc)_2(H_2O)_2]_2 \cdot 7H_2O$ (2**).** The preparation of **2** was similar to **1** except that $Zn(NO_3)_2 \cdot 6H_2O$ (0.12 g) was used in place of $Cd(NO_3)_2 \cdot 4H_2O$ (yield: 81% based upon Zn). Anal. Calcd for $C_{16}H_{28}ZnN_4O_{14}$: H, 4.6; N, 9.2; C, 31.7; and Zn, 10.8. Found: H, 4.5; N, 9.3; C, 31.6; and Zn, 10.8. FT/IR (cm^{-1}): 1537vs, 1463s, 1716s, 1386s, 1284m, 2967m, 1340m, 988m, 1228m, 769m, 1107m, 527w, 893w, 3428w, 656w, 477w, 1067w, 730w, 413w, and 687w.

2.2.3. Synthesis of $M(H_2pimdc)_2$, $M = Cu$ (3**) or Ni (**4**).** The preparation of **3** was similar to **1** except that $CuSO_4 \cdot 5H_2O$ (0.10 g) was used in place of $Cd(NO_3)_2 \cdot 4H_2O$ (yield: 65% based upon Cu). Anal. Calcd for $C_{16}H_{14}CuN_4O_8$: H, 3.1; N, 12.3; C, 42.4; and Cu, 14.0. Found: H, 3.1; N, 12.4; C, 42.3; and Cu, 14.1. FT/IR (cm^{-1}) for **3**: 1540vs, 3448s, 1712s, 1399s, 2970s, 1280m, 512m, 659m, 775m, 1055w, 1124w, 1025w, 951w, 1089w, and 887w.

The preparation of **4** was similar to **1** except that $NiSO_4 \cdot 7H_2O$ (0.09 g) was used in place of $Cd(NO_3)_2 \cdot 4H_2O$ (yield: 68% based upon Ni). Anal. Calcd for $C_{16}H_{14}NiN_4O_8$: H, 3.1; N, 12.5; C, 42.8; and Ni, 13.1. Found: H, 3.2; N, 12.6; C, 42.8; and Ni, 13.2.

FT/IR (cm^{-1}) for **4**: 1539vs, 3424s, 1715s, 1393m, 2971m, 1277m, 512w, 661w, 773w, 1055w, 864w, and 1119w.

2.3. X-ray crystallography

Crystals of **1–4** suitable for single-crystal X-ray diffraction were placed into four thin glass tubes, respectively, and mounted to a Rigaku R-AXIS RAPID IP diffractometer with graphite monochromated Mo-K α ($\lambda = 0.71073 \text{ \AA}$) radiation. Diffraction data of **1–4** were collected in the ψ scanning mode at 293 K. Multi-scan absorption corrections was applied. The structures were solved by direct methods and refined by full-matrix-least squares on F^2 using the SHELXTL-97 software [18]. All non-hydrogen atoms were refined anisotropically. Hydrogens were located from Fourier difference maps. Crystal parameters and details of the data collection and structure refinement for **1–4** are listed in table 1.

3. Results and discussion

3.1. Crystal structure of 1–4

Compounds **1–4** were prepared with traditional aqueous reactions, and the crystal structures of these compounds were determined by single-crystal X-ray diffraction. In **1**, there is one crystallographically independent Cd^{2+} , two H_2pimdc^- and six waters. The cadmium ion is in the center of a distorted octahedron CdN_2O_4 (figure 1) and

Table 1. Crystal data and structure refinement for **1–4**.

	1	2	3	4
Empirical formula	$\text{C}_{16}\text{H}_{28}\text{CdN}_4\text{O}_{14}$	$\text{C}_{32}\text{H}_{54}\text{Zn}_2\text{N}_8\text{O}_{27}$	$\text{C}_{16}\text{H}_{14}\text{CuN}_4\text{O}_8$	$\text{C}_{16}\text{H}_{14}\text{NiN}_4\text{O}_8$
Formula weight	612.83	1113.61	453.86	449.00
T (K)	293(2)	293(2)	293(2)	293(2)
Wavelength (\AA)	0.71069	0.71069	0.71069	0.71069
Space group	$P\bar{1}$	$P\bar{1}$	$P2(1)/n$	$P2(1)/n$
Unit cell dimensions (\AA , $^\circ$)				
a	10.638	10.638	8.321	8.303
b	10.708	14.321	10.152	10.150
c	11.375	16.051	11.217	11.203
α	83.653	85.887	90	90
β	81.706	83.585	91.122	91.075
γ	87.419	85.727	90	90
V (\AA^3)	1273.78	2418.32	947.37	944.07
Z	2	2	2	2
Calculated density (g cm^{-3})	1.598	1.529	1.591	1.580
Absorption coefficient (mm^{-1})	0.929	1.008	1.206	1.081
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0543$, $wR_2 = 0.1454$	$R_1 = 0.0475$, $wR_2 = 0.1333$	$R_1 = 0.0405$, $wR_2 = 0.1202$	$R_1 = 0.0488$, $wR_2 = 0.1344$
R indices (all data)	$R_1 = 0.0720$, $wR_2 = 0.1579$	$R_1 = 0.0564$, $wR_2 = 0.1406$	$R_1 = 0.0444$, $wR_2 = 0.1231$	$R_1 = 0.0597$, $wR_2 = 0.1383$
CCDC reference No.	793999	422166	794000	794001

$$R_1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}, \quad \omega R_2 = \frac{\sum [\omega(F_o^2) - F_c^2]}{\sum [\omega(F_o^2)]^{1/2}}$$

coordinated by two H_2pimdc^- ligands in single-chelating mode (mode a, scheme 1) and two waters (table 2).

There exist hydrogen bonds among the building fragments in **1**. The H-bonds between Ow5 and Ow6 of the coordinated waters and the non-coordinated oxygens of the carboxyl in another building fragment lead to the formation of 1-D chains (figure 2). Between these chains, there are plenty of hydrogen bonds among the lattice waters and the oxygens in H_2pimdc^- coordinated to Cd^{2+} . Therefore, a 3-D supramolecular structure forms through hydrogen bond interactions (figure 3). Data of hydrogen bonds are listed in table 3.

In **2**, there are two crystallographically independent Zn^{2+} ions (figure 4), and the coordination arrangements are the same as in **1**, only with differences in bond length and angles (table 2). There are seven lattice waters in the asymmetrical unit of **2** that make **1** and **2** with different asymmetrical units. However, the hydrogen bond packing in **2** is similar to that in **1**. For **1** and **2**, the propyl of H_2pimdc^- has an obvious influence on the crystal packing.

Compounds **3** and **4** crystallize in the same space group $P2(1)/n$ with $Z=2$. Since **3** and **4** are isomorphous, the structure of **3** is described as the representative. In the asymmetrical unit of **3**, there is only one crystallographically independent Cu^{2+} and two H_2pimdc^- . Each Cu^{2+} exhibits octahedral coordination of $\{\text{CuN}_2\text{O}_4\}$ elongated in the apical direction; the equatorial plane comprises two oxygens and two nitrogens from two distinct H_2pimdc^- ligands with Cu–O bond length 2.003(3) Å and Cu–N bond length 1.971(3) Å; the other two oxygens from two different H_2pimdc^- ligands occupy the remaining apical coordination sites with Cu–O bond lengths 2.509(4) Å, as depicted in figure 5. H_2pimdc^- takes a mono-chelating-mono-bridge coordination

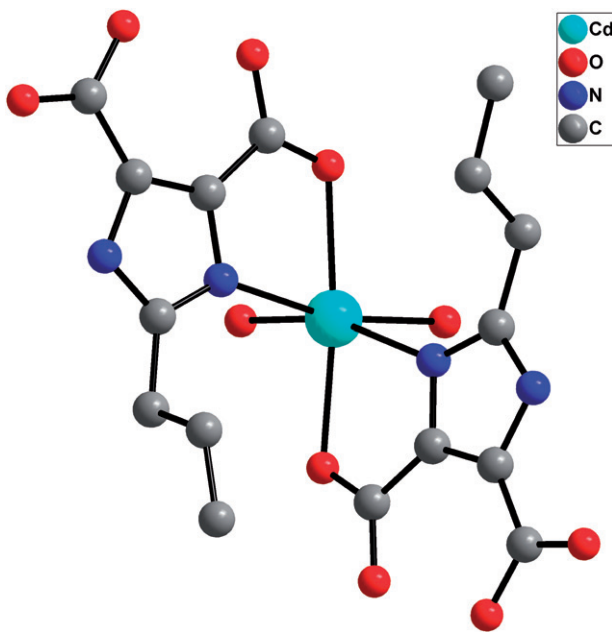
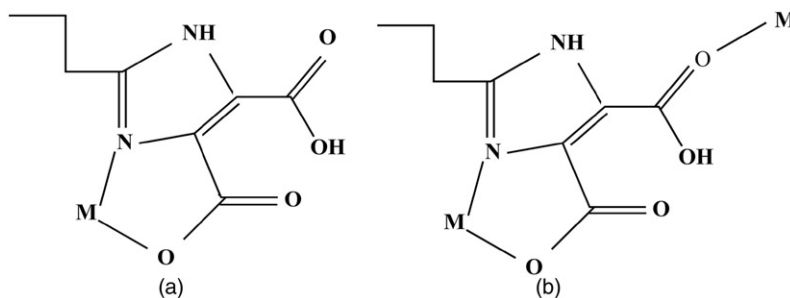
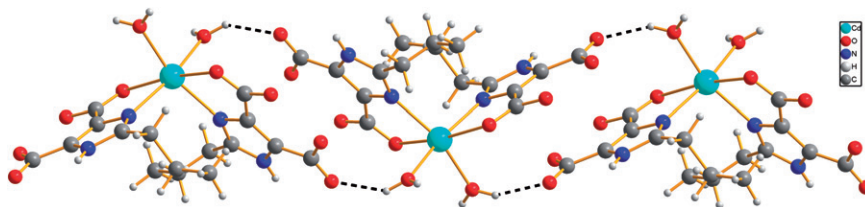


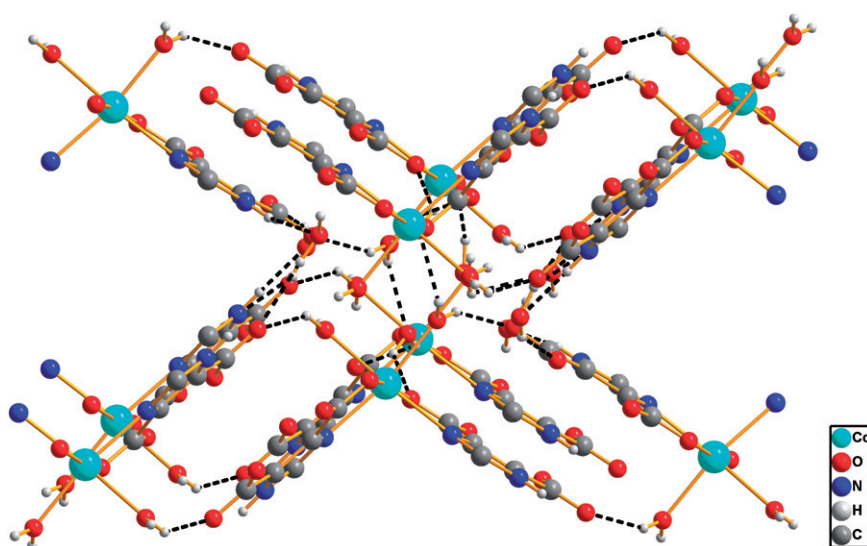
Figure 1. Ball-stick representation of Cd^{2+} coordination (hydrogens are omitted for clarity).

Scheme 1. Two coordination modes of H_2pimdc in **1**–**4**.Table 2. Selected bond lengths (Å) and angles (°) of **1** and **2**.

1		2	
Cd1–O5W	2.246(5)	Zn3–N40	2.117(18)
Cd1–O6W	2.299(4)	Zn3–O37	2.120(14)
Cd1–N3	2.294(5)	Zn3–O42	2.089(16)
Cd1–N1	2.309(4)	Zn3–O71	2.129(8)
Cd1–O2	2.345(4)	Zn3–N15	2.109(18)
Cd1–O3	2.368(4)	Zn3–O22	2.25(2)
O5W–Cd1–N3	162.52(17)	N40–Zn3–O71	76.8(5)
N3–Cd1–O2	72.46(14)	O37–Zn3–O42	88.9(6)
O5W–Cd1–O2	91.64(18)	N40–Zn3–O42	163.7(5)
O6W–Cd1–N1	165.83(17)	N40–Zn3–O37	90.1(7)
N3–Cd1–N1	100.03(16)	O42–Zn3–N15	169.0(7)
O5W–Cd1–N1	89.12(18)	O37–Zn3–N15	86.6(6)
N3–Cd1–O6W	85.63(17)	N40–Zn3–N15	97.0(7)
O5W–Cd1–O6W	89.0(2)	O42–Zn3–O71	92.6(5)
O2–Cd1–O3	169.32(14)	O37–Zn3–O71	87.0(4)
N1–Cd1–O3	71.72(14)	O71–Zn3–N15	97.2(5)
O6W–Cd1–O3	94.28(17)	N40–Zn3–O22	101.3(6)
N3–Cd1–O3	105.76(15)	O37–Zn3–O22	95.0(6)
O5W–Cd1–O3	91.20(17)	O42–Zn3–O22	96.9(7)
N1–Cd1–O2	98.04(15)	O71–Zn3–O22	170.3(6)
O6W–Cd1–O2	96.06(18)	N15–Zn3–O22	73.5(7)

Figure 2. 1-D chains in **1** formed by hydrogen bonds.

mode (mode b in scheme 1). The connection of alternating H_2pimdc^- and coppers result in a 2-D layer with parallelogram-like rings in the [101] plane, as shown in figure 6. This 2-D layer can be described as a (4,4) network with the topology symbol 4^4 with Cu^{2+} as four connection points and H_2pimdc^- as connection rods.

Figure 3. The net of hydrogen bonds in **1** (propyls are omitted for clarity).Table 3. Hydrogen bonds in **1**.

D–H...A	H...A (Å)	D...A (Å)	D–H...A (°)
O14–H6D...OW6	2.0504	2.8248	143.508
O19–H7A...OW5	1.9559	2.7546	147.044
OW3–H9B...O14	2.1824	3.0793	173.599
N4–H4...OW3	1.9155	2.7465	161.918
OW4–H7C...O3	2.0790	2.8333	140.707
OW4–H7B...OW5	2.0517	2.6510	122.875
OW2–H8A...O9	2.1064	2.9238	150.552
OW4–H7D...O8	2.2305	2.7949	120.294
N2–H2C...OW2	1.9064	2.7618	170.591

The propyl groups extend toward two sides of the layer, resulting in a hydrophobic core around the layer. Between two adjacent 2-D layers, there exist only intermolecular forces, as shown in figure 7. These weak interaction forces between adjacent 2-D layers result in their low thermal stability. Additionally, the propyl of H_2pimdc^- brings large steric constraints, which terminate the extension of **3** and **4**.

H_3pimdc with propyl group at the 2-position should have higher electron delocalization ability, but at the same time larger steric constraints. So H_3pimdc displays different coordination abilities and geometries in contrast to related structures in the literature [5–17].

3.2. Photoluminescent spectra

Photoluminescence properties of **1–4** were measured in the solid state at room temperature. Compound **1** exhibits a broad fluorescence band centered at

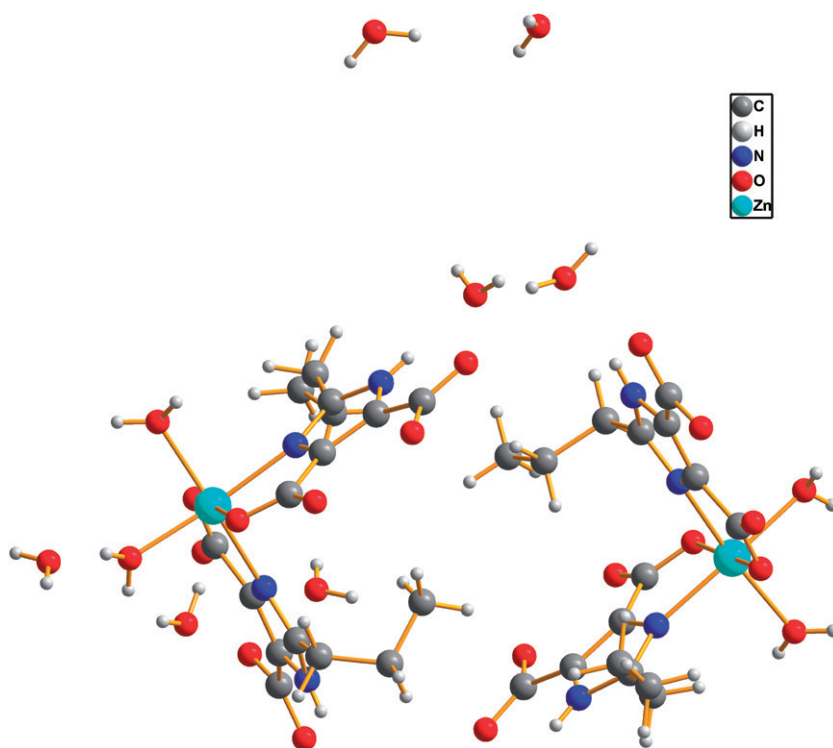


Figure 4. Ball-stick representation of Zn^{2+} coordination in **2**.

$\lambda_{\text{max}} = 453$ nm upon excitation at 363 nm (figure 8). Free H_3PIMDC was also measured under identical experimental conditions, showing a broad emission band at $\lambda_{\text{max}} = 464$ nm when excited at 363 nm (figure 8). Obviously, the origin of the emissions of **1** can be tentatively attributed to intramolecular transitions of the ligand. The blue-shifted emission of **1** compared to the pure H_3PIMDC is assigned to coordination effect. Compounds **2**, **3**, and **4** have no obvious emission, showing that coordination of Zn^{2+} , Cu^{2+} , and Ni^{2+} with H_3PIMDC quenches the luminescence of H_3PIMDC .

3.3. IR spectra

In infrared spectra of **1–4** (figure S1), strong bands between 1716 and 1386 cm^{-1} are assigned to carboxylates. Peaks in the high-frequency region are due to symmetrical stretching vibrations of carboxylate, and bands in the low-frequency region are attributed to asymmetrical stretching vibrations of carboxylate. Bands between 1386 and 1110 cm^{-1} are assigned to imidazole; peaks around 2970 cm^{-1} are due to stretching of C–H bonds in propyl groups.

3.4. Thermal analysis

The TG curves of **1** and **2** show weight losses of 17.96% in the region 40 – 150°C of **1** and 14.77% in the region 50 – 110°C of **2**, corresponding to the release of lattice waters (calcd

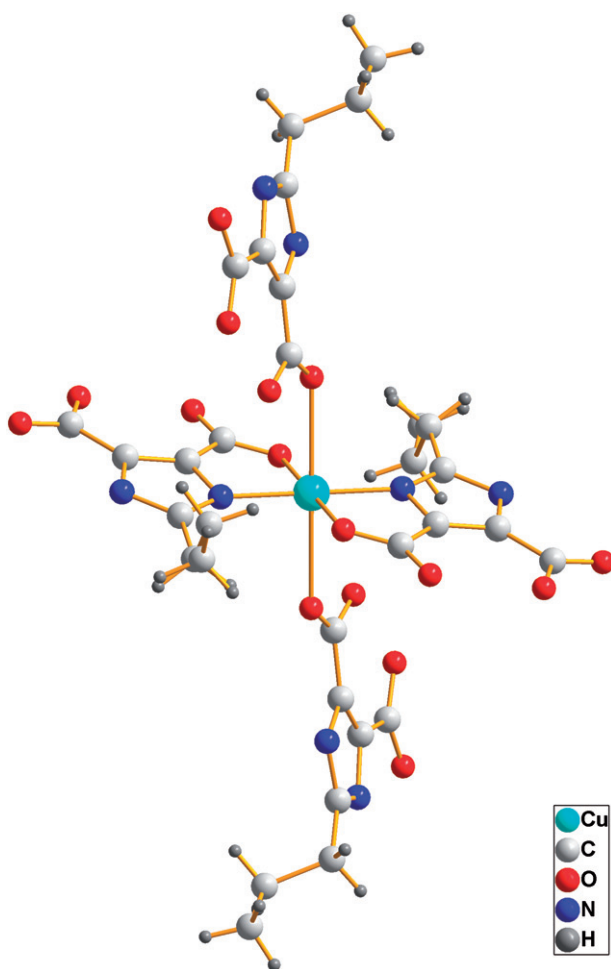
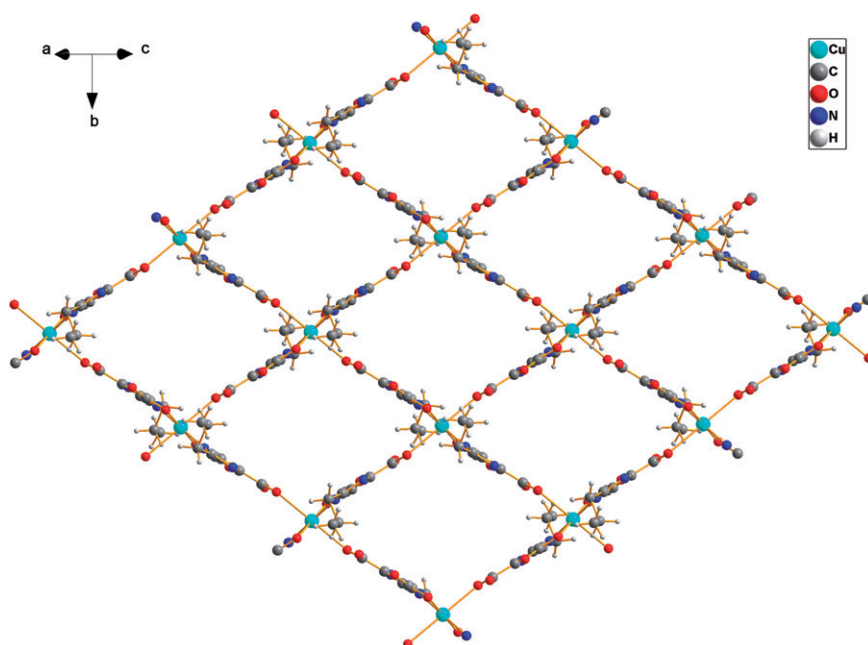
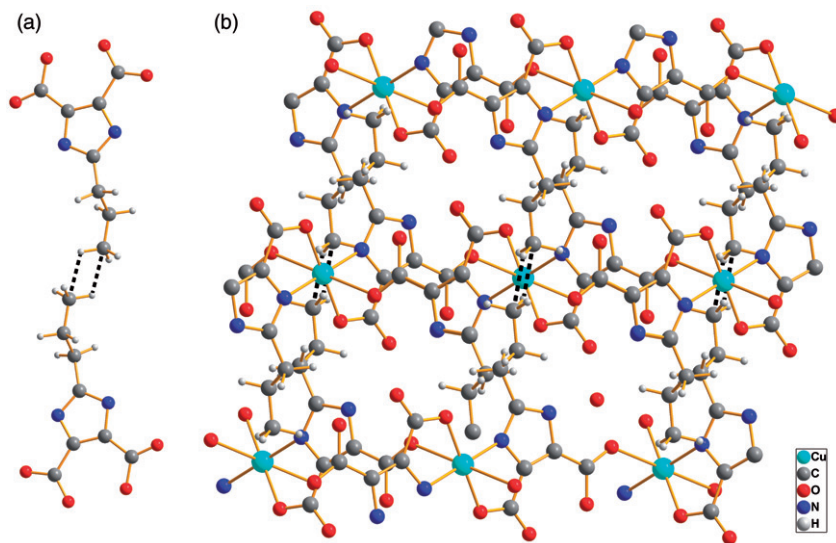


Figure 5. Perspective views of the coordination environments of Cu^{2+} in **3**.

11.75% in **1**; 11.31% in **2**) and coordinated waters (calcd 5.78% in **1**; 3.23% in **2**). The weight of two compounds remain until 290°C of **1** and 250°C of **2**, and then go through another weight loss, assigned to decomposition of the frameworks. In **3** and **4**, there are no waters, so that the two compounds do not lose weight from room temperature to 290°C of **3** and 240°C of **4**. After that, the weight losses can be attributed to collapse of the frameworks. Finally, the residue of the four compounds may be metal oxide.

4. Conclusions

We have obtained four new transition metal coordination complexes based on H_3imdc derivatives in conventional method in aqueous solution.

Figure 6. 2-D layer structure of **3**.Figure 7. Interactions between 2-D layers in **3**.

These supramolecular compounds have low thermal stability and only **1** exhibits obvious emission similar to the ligand; the combination of Zn^{2+} , Cu^{2+} , and Ni^{2+} with H_3pimdc quenches the luminescence of H_3pimdc . Compared with other organic ligands in metal–organic coordination polymers [19–21], H_3imdc derivatives with an electron

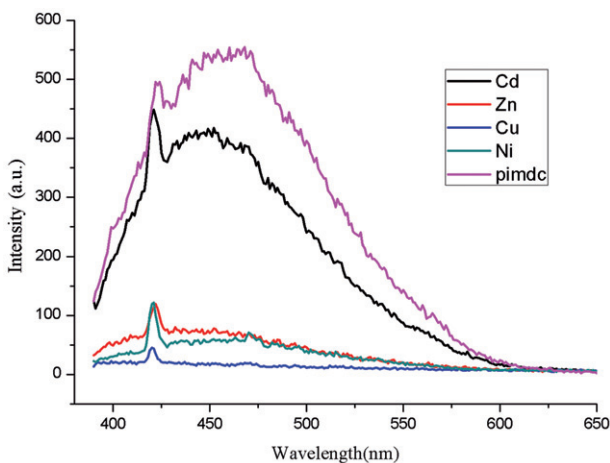


Figure 8. Photoluminescent spectra of **1–4** and H_3pimdc .

donor substituent may lead to the formation of a series of new and interesting structures.

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