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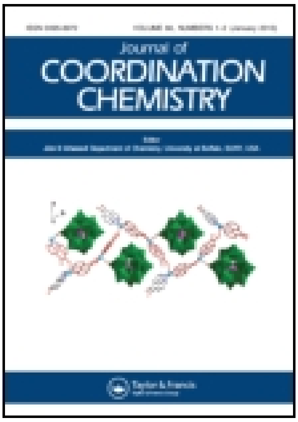

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Synthesis and crystal structures of three new complexes derived from 3-(pyridin-3′-yl)-5-(pyridin-2′′-yl)-1,2,4-triazole

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Synthesis and crystal structures of three new complexes derived from 3-(pyridin-3′-yl)-5-(pyridin-2″-yl)-1,2,4-triazole

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Three new coordination complexes, $[Cd_2(ppt)_2(cpba)]\cdot(H_2O)_2$ (**1**), $[Zn(ppt)_2]\cdot(H_2O)$ (**2**), and $[Zn(MoO_4)(Hppt)]$ (**3**) (H_2cpba: 3-(2′-carboxy-phenoxy)-benzoic acid; Hppt: 3-(pyridin-3′-yl)-5-(pyridin-2″-yl)-1,2,4-triazole), were synthesized under hydrothermal conditions. Their structures have been determined by single crystal X-ray diffraction analyses and further characterized by elemental analyses, IR spectra, and thermogravimetric analyses. X-ray diffraction analyses revealed that Cd(II) ions are linked by ppt^- to form a ladder-shaped structure along the *a*-axis and further displays a 2-D supramolecular architecture with $cpba^{2-}$ along the *c*-axis. In **2**, each $Zn(ppt)_2$ fragment is linked by the nitrogen of pyridin-3′-yl from the neighboring $Zn(ppt)_2$ forming a V-shaped chain. Compound **3** consists of a ladder structure, in which each $\{MoO_4\}$ unit is a bridge linking three $Zn(Hppt)^{2+}$ fragments.

Keywords: 3-(2′-Carboxy-phenoxy)-benzoic acid; 3-(Pyridin-3′-yl)-5-(pyridin-2″-yl)1,2,4-triazole; Cadmium; Zinc; X-ray diffraction analyses

1. Introduction

The design and synthesis of porous metal–organic frameworks (MOFs), which provide pores of well-defined sizes, shapes, and chemical environments, has attracted research interest for topological novelty and potential applications in gas storage, chemical separations, ion exchange, microelectronics, nonlinear optics, and heterogeneous catalysis [1, 2].

Although some target networks can be constructed by combined use of metal ions and suitable ligands, true crystal engineering of coordination polymers with desired topologies and specific properties remains a challenge. A major problem is the complexity of one-pot assembly in a multicomponent system, such as flexible coordination modes of commonly used ligands. Polypyridines afford more predictable

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coordination modes and have been used to design and assemble finite architectures with coordination strict metal cations [3–8].

As an extension of our previous work [9], we herein report syntheses and crystal structures of three new coordination complexes constructed from 3-(pyridin-3′-yl)-5-(pyridin-2′′-yl)-1,2,4-triazole and transition metal cations, $[Cd_2(ppt)_2(cpba)]\cdot(H_2O)_2$ (**1**), $[Zn(ppt)_2]\cdot(H_2O)$ (**2**), and $[Zn(MoO_4)(Hppt)]$ (**3**) (H_2cpba: 3-(2′-carboxy-phenoxy)-benzoic acid; Hppt: 3-(pyridin-3′-yl)-5-(pyridin-2′′-yl)-1,2,4-triazole.

2. Experimental

2.1. *General*

H_2cpba and Hppt were purchased from Jinan Henghua Sci. & Tec. Co. Ltd and used without purification. Elemental analysis was conducted on a Perkin-Elmer 240C elemental analyzer. Thermogravimetric analysis (TGA) measurements were carried out on a Perkin-Elmer Diamond TG/DTA instrument.

2.2. *Synthesis*

2.2.1. Synthesis of 1. The synthesis is performed in a 25 mL Teflon-lined stainless steel vessel. Cadmium sulfate octahydrate (0.20 mmol, 0.051 g), H_2cpba (0.20 mmol, 0.052 g), sodium hydroxide (0.40 mmol, 0.016 g), Hppt (0.20 mmol, 0.045 g), and water (14 mL) were mixed and heated to 170°C for 3 days. Colorless crystals were obtained after cooling to room temperature in 42% yield. Anal. Calcd for $C_{76}H_{56}Cd_4N_{20}O_{14}$ (%): C, 47.42; H, 2.91; N, 14.56. Found (%): C, 47.32; H, 2.58; N, 14.32. IR (KBr pellet, cm^{-1}): 3545, 3394, 3063, 2927, 1942, 1707, 1596, 1539, 1400, 1313, 1274, 1233, 1175, 1157, 1105, 1035, 942, 875, 807, 750, 709, 640, 527, 478.

2.2.2. Synthesis of 2. The synthesis is performed in a 25 mL Teflon-lined stainless steel vessel. Ammonium molybdate tetrahydrate (0.08 mmol, 0.10 g), zinc acetate dihydrate (0.23 mmol, 0.05 g), Hppt (0.34 mmol, 0.05 g), and water (14 mL) were mixed and heated to 170°C for 3 days. Colorless crystals were obtained after cooling to room temperature in 32% yield. Anal. Calcd for $C_{24}H_{18}N_{10}OZn$ (%): C, 54.56; H, 3.41; N, 25.56. Found (%): C, 54.36; H, 3.28; N, 25.39. IR (KBr pellet, cm^{-1}): 3381, 3055, 2445, 1960, 1868, 1646, 1607, 1574, 1526, 1458, 1418, 1290, 1270, 1158, 1105, 1022, 995, 940, 880, 825, 798, 746, 706, 638, 536, 452.

2.2.3. Synthesis of 3. The synthesis is performed in a 25 mL Teflon-lined stainless steel vessel. Sodium molybdate dihydrate (0.08 mmol, 0.10 g), zinc acetate dihydrate (0.23 mmol, 0.05 g), Hppt (0.34 mmol, 0.05 g), and water (14 mL) were mixed and heated to 170°C for 3 days. Colorless crystals were obtained after cooling to room temperature with 27% yield. Anal. Calcd for $C_{12}H_9MoN_5O_4Zn$ (%): C, 32.10; H, 2.01; N, 15.60. Found (%): C, 32.01; H, 1.83; N, 15.32. IR (KBr pellet, cm^{-1}): 3440, 3126, 3039, 2911, 2652, 2540, 1739, 1605, 1561, 1454, 1414, 1384, 1279, 1249, 1189, 1151, 1040, 987, 909, 855, 732, 695, 446.

2.3. *Structure determination*

Intensity data collections were 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. The structures were solved by direct methods and refined by full-matrix least-squares using SHELX-TL [10]. Crystallographic data for **1–3** are given in table 1. Selected bond lengths and angles are listed in tables 2–4.

3. Results and discussion

3.1. *Structure of 1*

The molecular structure of **1** is shown in figure 1. Selected bond lengths and angles are given in table 2. The Cd–N and Cd–O bond distances are in normal ranges,

Table 1. Crystallographic data and details of diffraction experiments for **1–3**.

	1	**2**	**3**
Formula	$C_{76}H_{56}Cd_4N_{20}O_{14}$	$C_{24}H_{18}N_{10}OZn$	$C_{12}H_9MoN_5O_4Zn$
Molecular mass (g mol^{-1})	1923.01	527.85	448.55
Crystal system	Monoclinic	Monoclinic	Orthorhombic
Space group	*P*-1	*P*2(1)/*c*	*Pna*2(1)
Unit cell dimensions (Å, °)			
a	9.4494(17)	16.8858(14)	22.883(3)
b	9.6079(18)	8.9194(8)	9.4049(11)
c	21.179(4)	14.9508(13)	6.3918(8)
α	77.339(6	90	90
β	84.574(8)	102.0260(10)	90
γ	86.536(6)	90	90
Volume (Å^3), *Z*	2871.6(7), 1	2202.3(3), 4	484.4(4)
Calculated density (g cm^{-3})	1.711	1.592	2.166
Absorption coefficient (mm^{-1})	1.204	1.158	2.684
Temperature (K)	298(2)	298(2)	298(2)
Reflections collected	9845	4899	9222
Independent reflection	6372	3879	2413
Refined parameters	526	333	209
R_{int}	0.0281	0.0331	0.0485
θ range for data collection (°)	$1.98 < \theta < 25.00$	$2.47 < \theta < 25.00$	$2.34 < \theta < 24.99$
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0494^a$ $wR_2 = 0.1238^b$	$R_1 = 0.0300^a$ $wR_2 = 0.0887^b$	$R_1 = 0.0241^a$ $wR_2 = 0.0642^b$
R indices (all data)	$R_1 = 0.0727^a$ $wR_2 = 0.1346^b$	$R_1 = 0.0485^a$ $wR_2 = 0.1016^b$	$R_1 = 0.0249^a$ $wR_2 = 0.0651^b$
Goodness-of-fit on $F^{2\ c}$	1.003	1.000	1.001
Largest difference peak and hole (e Å^{-3})	1.238 and −0.720	0.303 and −0.482	0.441 and −0.547

[a] $R_1 = \Sigma||F_{obsd.}| - |F_{calcd}||/\Sigma|F_{obsd.}|$. [b] $wR_2 = \{\Sigma[w(F^2_{obsd.} - F^2_{calcd})^2]/\Sigma[w(F^2_{obsd.})^2]\}^{1/2}$. $w = 1/[\sigma^2(F^2_o) + xP + (yP)^2]$; with $P = (F^2_o + 2F^2_c)/3$ and $x = 0.0810$, $y = 0.2734$ (**1**), $x = 0.0700$, $y = 0.2734$ (**2**); $x = 0.0430$, $y = 1.1742$ (**3**). [c] Goodness-of-fit on $F^2 = [\Sigma w(F^2_{obsd.} - F^2_{calcd})^2/(n - p)]^{1/2}$, where n = number of reflections, p = parameter used.

Table 2. Selected bond lengths and angles for **1**.

Cd(1)–O(2)	2.285(4)	Cd(2)–O(3)	2.367(5)
Cd(1)–N(6)	2.319(5)	Cd(2)–O(4)	2.367(5)
Cd(1)–O(1)	2.383(4)	Cd(2)–N(5)	2.470(5)
Cd(2)–N(3)	2.265(5)		
O(2)–Cd(1)–N(6)	96.06(16)	O(3)–Cd(2)–O(4)	55.26(16)
O(2)–Cd(1)–O(1)	56.48(15)	N(3)–Cd(2)–N(5)	70.44(17)
N(6)–Cd(1)–O(1)	148.45(16)	O(3)–Cd(2)–N(5)	78.72(16)
O(2)–Cd(1)–C(1)	28.19(17)	O(4)–Cd(2)–N(5)	88.71(16)
N(6)–Cd(1)–C(1)	122.22(19)	N(3)–Cd(2)–C(14)	124.29(19)
O(1)–Cd(1)–C(1)	28.38(17)	O(3)–Cd(2)–C(14)	27.20(18)
N(3)–Cd(2)–O(3)	143.04(16)	O(4)–Cd(2)–C(14)	28.25(18)
N(3)–Cd(2)–O(4)	103.24(17)	N(5)–Cd(2)–C(14)	80.50(17)

Table 3. Selected bond lengths and angles for **2**.

N(2)–N(1)	1.366(7)	Zn(1)–N(9)	2.166(5)
Zn(1)–N(1)	2.061(5)	Zn(1)$^{\#1}$–N(10)	2.069(5)
Zn(1)–N(4)	2.161(5)	Zn(1)–N(10)$^{\#2}$	2.069(5)
Zn(1)–N(6)	2.064(5)		
N(1)–Zn(1)–N(6)	158.7(2)	N(10)$^{\#2}$–Zn(1)–N(4)	115.9(2)
N(1)–Zn(1)–N(10)$^{\#2}$	100.1(2)	N(1)–Zn(1)–N(9)	94.0(2)
N(6)–Zn(1)–N(10)$^{\#2}$	101.1(2)	N(6)–Zn(1)–N(9)	77.6(2)
N(1)–Zn(1)–N(4)	77.63(19)	N(10)$^{\#2}$–Zn(1)–N(9)	107.0(2)
N(6)–Zn(1)–N(4)	95.0(2)	N(4)–Zn(1)–N(9)	137.1(2)

Symmetry transformations used to generate equivalent atoms: $^{\#1}-x+1,\ y-1/2,\ -z+1/2$; $^{\#2}-x+1,\ \underline{y}+1/2,\ -z+1/2$.

Table 4. Selected bond lengths and angles for **3**.

Mo(1)–O(4)	1.725(2)	Zn(1)–O(1)	1.988(7)
Mo(1)–O(2)	1.743(2)	Zn(1)–O(2)$^{\#2}$	2.044(2)
Mo(1)–O(3)	1.762(9)	Zn(1)–N(1)	2.087(2)
Mo(1)–O(1)	1.750(7)	Zn(1)–N(2)	2.181(3)
Zn(1)–O(3)$^{\#1}$	1.957(9)		
O(4)–Mo(1)–O(2)	108.58(14)	O(1)–Zn(1)–O(2)$^{\#2}$	91.5(4)
O(4)–Mo(1)–O(3)	106.7(4)	O(3)$^{\#1}$–Zn(1)–N(1)	118.2(4)
O(2)–Mo(1)–O(3)	112.9(5)	O(1)–Zn(1)–N(1)	115.6(4)
O(4)–Mo(1)–O(1)	111.2(4)	O(2)$^{\#2}$–Zn(1)–N(1)	91.26(11)
O(2)–Mo(1)–O(1)	107.0(5)	O(3)$^{\#1}$–Zn(1)–N(2)	88.9(3)
O(3)–Mo(1)–O(1)	110.53(14)	O(1)–Zn(1)–N(2)	94.6(3)
O(3)$^{\#1}$–Zn(1)–O(1)	125.71(13)	O(2)$^{\#2}$–Zn(1)–N(2)	170.13(14)
O(3)$^{\#1}$–Zn(1)–O(2)$^{\#2}$	93.7(4)	N(1)–Zn(1)–N(2)	79.13(10)

Symmetry transformations used to generate equivalent atoms: $^{\#1}\ x,\ y,\ z+1$; $^{\#2}\ -x,\ -y+1,\ z+1/2$.

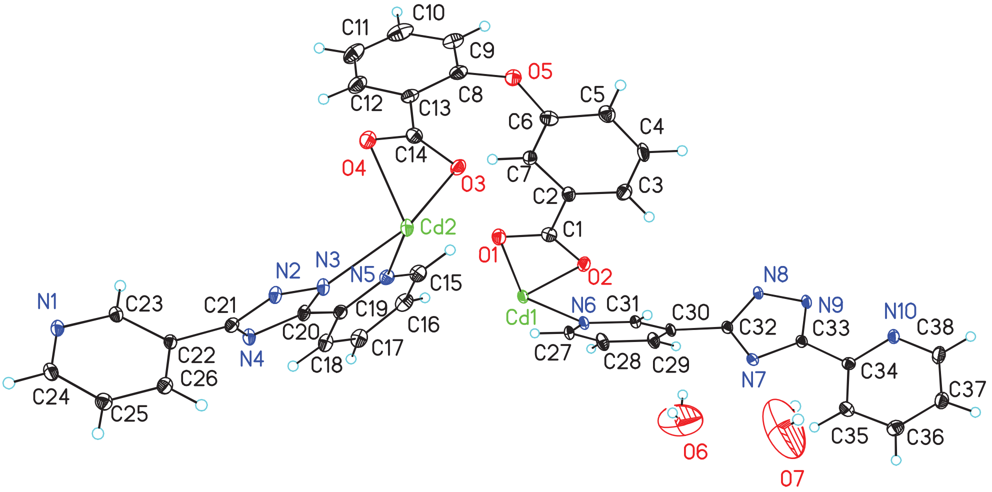

Figure 1. ORTEP view of **1** (30% thermal ellipsoids) showing the atom-labeling scheme.

2.258(4)–2.470(5) and 2.285(4)–2.367(5) Å, respectively. Single crystal X-ray diffraction analyses revealed that **1** consists of a dinuclear Cd(II) plane linked by two ppt$^-$ ligands forming one solid six-membered ring, in which the cadiums are linked by two triazole nuclei *via* N–N bridging. Deviations from the least-squares plane defined by two Cd's and four nitrogens are less than 0.07(1) Å. The neighboring dihedral angle between the two planes defined around the Cd's is 66.9(4) Å. Along the *a*-axis, each cadium is linked by nitrogen of pyridin-3′-yl forming a ladder as shown in figure 2. Along the *c*-axis, the ladder is further linked by cpba^{2-} *via* two carboxylates forming a 2-D supramolecular architecture, shown in figure 3.

3.2. *Structure of 2*

The molecular structure of **2** is shown in figure 4. Selected bond lengths and angles are given in table 3. The Zn–N bond distances are in the normal range of 2.061(5)–2.166(5) Å. Zn is in a N_5 coordination environment, slightly above the least-squares planes through nitrogens N(1), N(4), N(6), and N(9) at the same side of the molecule. Each Zn(ppt)$_2$ is linked by nitrogen of pyridin-3′-yl from the neighboring Zn(ppt)$_2$ fragments, forming a V-shaped chain (Zn…Zn: 8.55 Å; figure 5).

3.3. *Structure of 3*

The molecular structure of **3** is shown in figure 6. Selected bond lengths and angles are given in table 4. The Zn–N, Zn–O, and Mo–O bond distances are in the normal ranges, 2.087(2)–2.181(3), 1.957(9)–2.044(2), and 1.725(2)-1.762(9) Å, respectively. As shown in figure 7, the structure of [Zn(MoO$_4$)(Hppt)] (**3**) consists of 1-D chains comprising

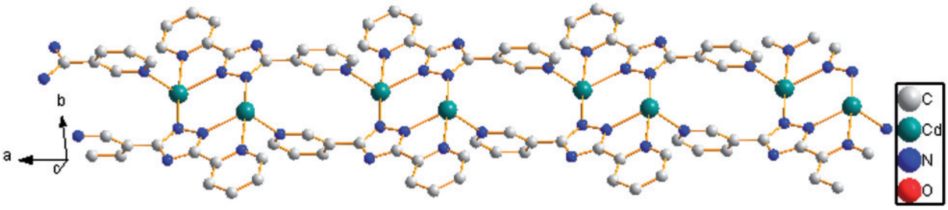

Figure 2. The ladder-shaped architecture linked by ppt$^-$ ligands, consisting of dinuclear Cd(II) planes; hydrogens are omitted for clarity.

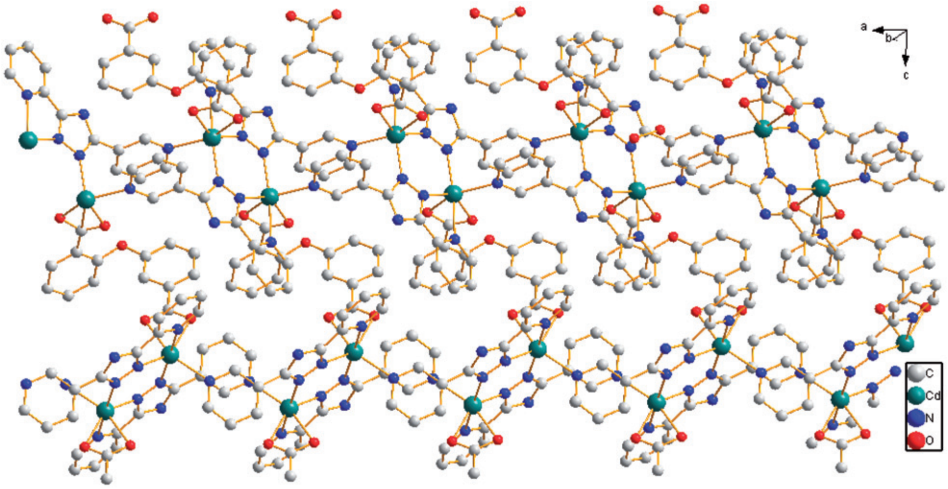

Figure 3. The 2-D structure assembled from ppt$^-$ and cpba^{2-}; hydrogens are omitted for clarity.

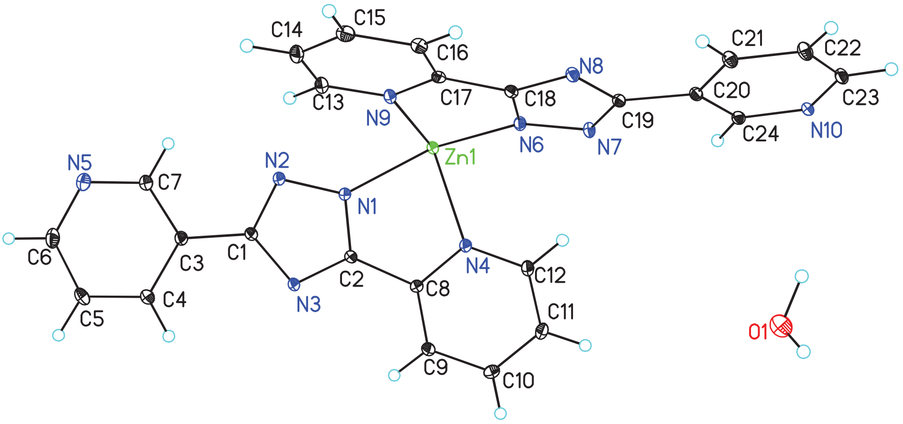

Figure 4. ORTEP view of **2** (30% thermal ellipsoids) showing the atom-labeling scheme.

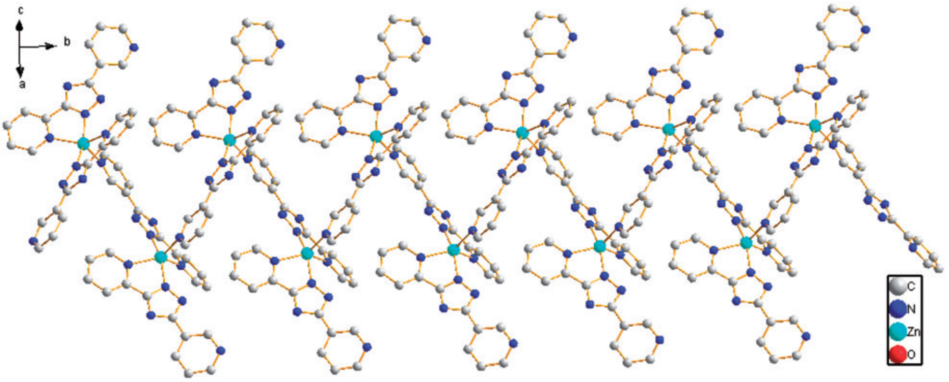

Figure 5. The V-shaped architecture of **2** linked by ppt$^-$ ligands; hydrogens are omitted for clarity.

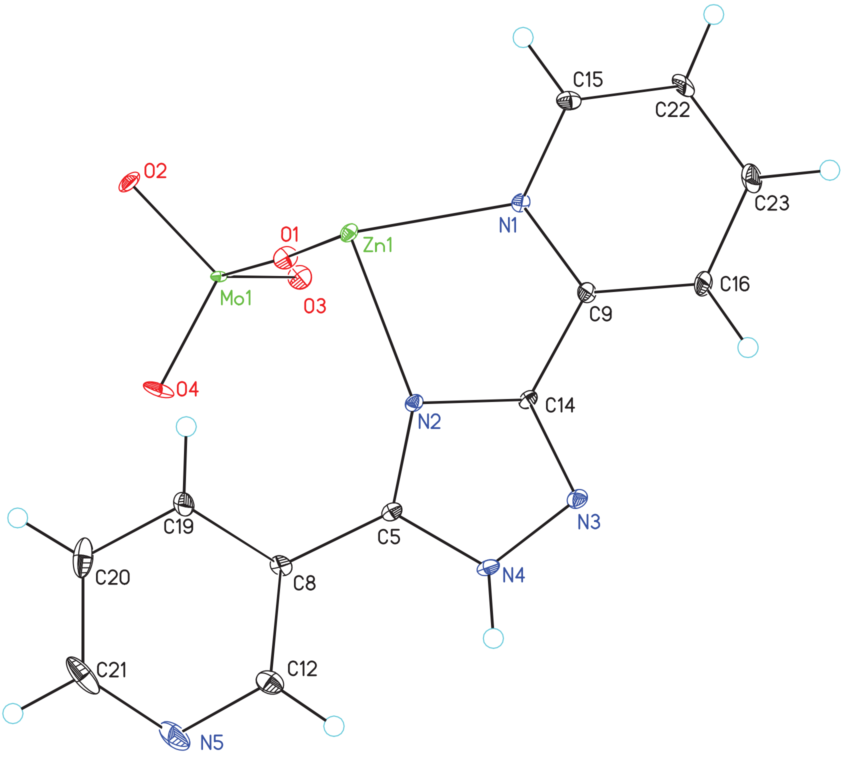

Figure 6. ORTEP view of **3** (30% thermal ellipsoids) showing the atom-labeling scheme.

{ZnN_2O_3} trigonal biyramids and {MoO_4} tetrahedra. Each {MoO_4} tetrahedral corner shares three terminal oxygens with three Zn(Hppt) fragments.

Multipoint hydrogen-bonding also exists between hydrogens from organic amines and crystallization water molecules (or terminal oxygens for **3**), stabilizing the packing diagrams of **1–3**, as shown in figures 8–10.

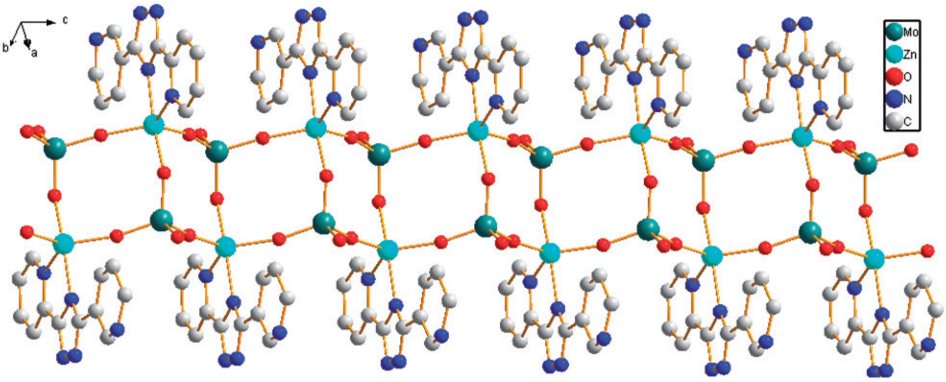

Figure 7. The ladder-shaped architecture of **3** constructed from $MoO4^{2-}$ and Zn^{2+}; hydrogens are omitted for clarity.

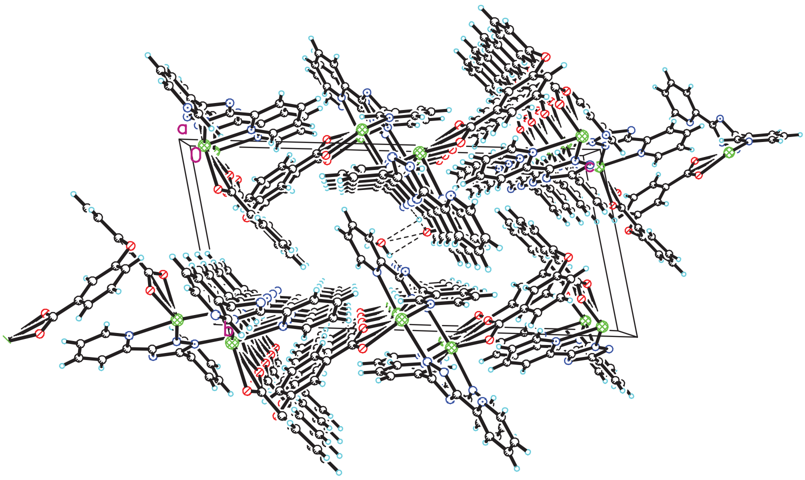

Figure 8. The crystal packing of **1** viewed along the *a*-axis, interwoven by hydrogen contacts.

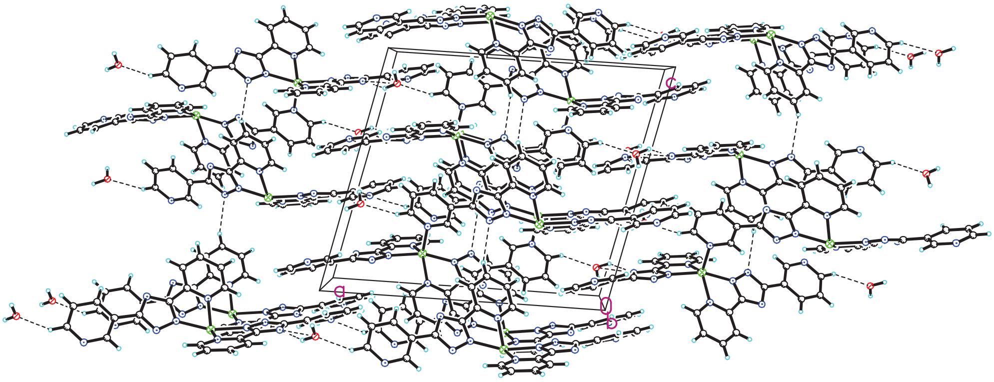

Figure 9. The crystal packing of **2** viewed along the *b*-axis, interwoven by hydrogen contacts.

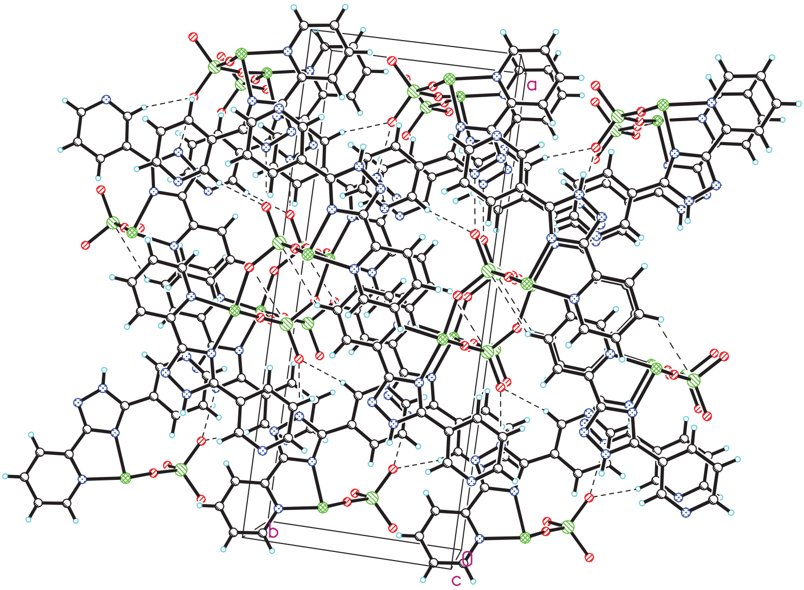

Figure 10. The crystal packing of **3** viewed along the *a*-axis, interwoven by hydrogen contacts.

3.4. *TG analysis*

To reveal the thermal stabilities of **1–3**, TGA has been carried out on a Perkin-Elmer Diamond TG/DTA instrument with a flow of dry air and a heating rate of 5°C min^{-1} from room temperature to 1000°C. TGA curve (Supplementary material) indicates that **1** is stable to 227°C. Weight loss from 227 to 393°C is 30.9%, attributed to elimination of cpba^{2-} and crystallization water molecules (Calcd 30.6%). From 393 to 763°C, ppt^{-} was lost and Cd(II) was oxidized to CdO. The residual weight of the sample is *ca.* 25.1% (Calcd for CdO: 25.8%). TGA curve (Supplementary material) indicates that **2** is stable to 162°C. Weight loss from 162 to 436°C is 3.0%, attributed to elimination of crystallization water molecules (Calcd 3.4%). From 436 to 736°C, ppt^{-} was lost and Zn(II) formed ZnO. The residual weight of the sample is *ca.* 21.0% (Calcd for ZnO: 15.1%). The TG experiment of **3** is difficult to separate into steps. Weight loss from 244 to 610°C is attributed to elimination of ppt^{-} (Supplementary material).

4. Conclusion

MOF topology depends on acidity, stoichiometry, *etc*., and also on the transition metal salt. This proved meaningful during the synthesis of **1–3** with the tetradentate 3-(pyridin-3-yl)-5-(pyridin-2-yl)-1,2,4-triazole. Further investigations of the optical, electronic, magnetic, and catalytic properties of these newly synthesized complexes continue.

Supplementary material

Further details on the crystal structure investigations of **1–3** 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; http://www.ccdc.cam.ac.uk/deposit], on quoting the depository number CCDC-819953 for **1**, 819951 for **2** and 819952 for **3**.

Acknowledgments

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