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# Hydrothermal synthesis, crystal structures and TGA of two metal–organic frameworks from 2'-(1H-tetrazol-5-yl)-[1,1'-biphenyl]-4-carboxylic acid

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# 1. Introduction

Self-assembled metal-organic frameworks (MOFs) containing transition metal ions and organic building blocks have been rapidly expanding in recent years owing to their novel and diverse topologies [1-6] and potential applications in the areas of nonlinear optics, gas storage, catalysis, electrical conductivity, and magnetism [7-11]. To get designed and predictable frameworks and properties, an enormous amount of research is being focused on constructing novel MOFs by choosing versatile organic ligands and functional metal ions. Heterocyclic carboxylic acids such as tetrazoly-, pyridine-, pyrazole-, and imidazole-carboxylic acids as building blocks have attracted much attention [12-19]. Especially, the tetrazolyl group can either coordinate in two monodentate modes, three  $\mu_2$ -modes, two  $\mu_3$ -modes or  $\mu_4$ -modes while carboxylate groups can coordinate in monodentate, chelate or bridged mode resulting in the formations of various MOFs with specific topologies and useful properties [20-26]. Our group has also been dedicated to the design of new tetrazolyl-carboxylate contained ligand including their derivatives and the study on their coordination chemistry [27]. As a continuation of our work, a new fivemembered nitrogen heterocyclic carboxylic derivative 2'-(1H-tetrazol-5-yl)-[1,1'-biphenyl]-4-carboxylic acid (H2tbpc) attracts our attention. The four tetrazole nitrogen atoms of this ligand and carboxylic acid functional group may work as the useful coordination sites. Novel structures might be produced by employing this ligand

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#### ABSTRACT

Two novel coordination compounds  $[Zn(tbpc)]_n$  (**1**) and  $[Cd(tpbc)(H_2O)]_n$  (**2**), where  $H_2tbpc = 2'-(1H-tet-razol-5-yl)-[1,1'-biphenyl]-4-carboxylic acid, have been synthesized through hydrothermal method. In$ **1**and**2**, the tbpc adopts different quadridentate bridging modes to connect metal (<math>Zn/Cd) centers to form distinct two-dimensional layers, which are stacked in different sequence. The  $C_{phenyl}$ - $H\cdots\pi$  interactions play a central role in packing 2D layers to form 3D structure. The preliminary investigation on the thermal stability of the complexes is presented.

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to react with various metals. In this work,  $H_2$ tbpc was synthesized, and two novel MOFs {[Zn(tbpc)]<sub>n</sub> (1) and [Cd(tbpc)( $H_2O$ )]<sub>n</sub> (2)} have been isolated under hydrothermal conditions. Then, their crystal structure and thermogravimetric analysis is reported.

#### 2. Experimental

# 2.1. Materials and measurements

All commercially available chemicals were of reagent grade and were used as received without further purification. Elemental analysis of C, H and N were taken on a Perkin-Elmer 240 element-alanalyzer. Infrared (IR) spectra were recorded on a Bruker Vector22 FT-IR spectro-photometer by using KBr plates. <sup>1</sup>H NMR and <sup>13</sup>CNMR spectra were obtained in a Bruker 300 MHz NMR spectrometer. Electrospray ionization mass (ESI-MS) spectra were recorded on a Finnigan Ion LCQ Advantage Max mass spectrometer in a scan range 50–2000 amu. The crystal structures were determined by Rigaku SCX mini diffractometer. The X-ray powder diffraction data was obtained on a Rigaku X-ray powder diffractometer D/MAX 2000/PC. Thermogravimetric analyses were performed on a Diamond TG/DTA (PE) simultaneous thermal analyzer under flowing N<sub>2</sub> (100 mL/min) with a heating rate of 10 °C/min from ambient temperature to 810 °C.

#### 2.2. Preparation of the ligand (H<sub>2</sub>tbpc)

The ligand, H<sub>2</sub>tbpc, was prepared according to the literature method [27] from 4-methyl biphenyl-2-carbonitrile through two

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Scheme 1. Synthesis of H<sub>2</sub>tbpc.

steps (Scheme 1). Anal. Calcd. for  $C_{14}H_{10}N_4O_2$ : C, 63.15; H, 3.79; N, 21.04. Found: C, 63.09; H, 3.88; N, 20.92. <sup>1</sup>H NMR (300 MHz, CD<sub>3</sub>OD):  $\delta$  7.98 (*d*, *J* = 7.98 Hz, 2H), 7.72 (*t*, 2H), 7.63 (*t*, 2H), 7.24 (*d*, *J* = 7.98 Hz 2H). <sup>13</sup>CNMR (300 MHz, CD<sub>3</sub>OD):  $\delta$  167.90, 155.30, 143.92, 141.10, 131.26, 130.48, 130.34, 129.79, 128.87, 128.22, 122.92. ESI-MS *m/z* (%): [M–H]<sup>-</sup> 265.32; [M+H]<sup>+</sup> 267.33. Main IR (cm<sup>-1</sup>, KBr): 3446 (b, w), 3130 (b, s), 2999 (w), 1680 (s), 1608 (m), 1560 (w), 1481 (w), 1402 (s), 1323 (w), 1290 (m), 1251 (w), 1186 (w), 1167 (w), 1125 (w), 1049 (w), 1011 (w), 935 (w), 860

#### Table 1

Crystal data and structure refinement parameters for 1-2.

Compound	1	2
Empirical formula	C <sub>14</sub> H <sub>8</sub> N <sub>4</sub> O <sub>2</sub> Zn	C <sub>14</sub> H <sub>10</sub> N <sub>4</sub> O <sub>3</sub> Cd
Formula weight	329.61	394.67
Crystal system	Monoclinic	Orthorhombic
Space group	P2 <sub>1</sub> /c	Pbca
a (Å)	11.036(2)	7.5811(15)
b (Å)	7.6673(15)	15.449(3)
c (Å)	15.828(3)	25.163(5)
α (°)	90	90
β(°)	98.02(3)	90
γ (°)	90	90
V (Å <sup>3</sup> )	1326.3(5)	2947.1(10)
Ζ	4	1
T (K)	293	293(2)
$D_{\text{calc}}$ (g cm <sup>-3</sup> )	1.651	1.770
$\mu$ (mm <sup>-1</sup> )	1.861	1.499
F (000)	664	1536
$\theta$ range (°)	3.25-27.48	3.09-27.48
Index ranges	$-14\leqslant h\leqslant 14$ ,	$-9\leqslant h\leqslant 9$ ,
	$-9\leqslant k\leqslant 9$ ,	$-20\leqslant k\leqslant 20$ ,
	$-20 \leqslant l \leqslant 20$	$-32\leqslant l\leqslant 32$
Collected reflections	13,348	28,226
Independent reflections	3039 [R(int) = 0.0454]	3369 [R(int) = 0.0854]
Data/restraints/	3039/0/190	3369/0/207
parameters		
$R_1^{a}, wR_2^{b} [I > 2\sigma(I)]$	0.0401, 0.0851	0.0529, 0.0724
R <sub>1</sub> <sup>a</sup> ,wR <sub>2</sub> <sup>b</sup> [all data]	0.0551, 0.0906	0.0810, 0.0792
Minimum, maximum	0.326, -0.358	0.824, -2.224
$ ho/$ e Å $^{-3}$		
GOF	1 097	1 105

<sup>a</sup>  $R_1 = \Sigma ||F_0| - |F_c| / \Sigma |F_0|$ .

<sup>b</sup>  $wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma w[(F_o^2)^2]\}^{1/2}.$ 

#### Table 2

Selected bond lengths (Å) and angles (°) for 1.

Zn—O2A	1.931(2)	Zn—O1B	1.937(2)
Zn—N3C	2.001(3)	Zn—N1	2.004(2)
02A—Zn—O1B	111.44(9)	O2A—Zn—N3C	115.66(10)
01B—Zn—N3C	108.35(10)	O2A—Zn—N1	101.71(10)
01B—Zn—N1	116.55(10)	N(3)C—Zn—N1	103.02(10)

Symmetry codes: (A) -x + 2, -y + 1, -z, (B) x, y - 1, z, (C) -x + 2, y - 1/2, -z + 1/2.

(m), 779 (w), 754 (s), 705 (w), 675 (w), 555 (w), 511 (w), 472 (w), 422 (w).

# 2.3. Synthesis of the complexes 1 and 2

#### 2.3.1. $[Zn (tbpc)]_n$ (1)

 $H_2tbpc\ (1\ mmol,\ 0.0265\ g)\ and\ ZnNO_3\cdot 6H_2O\ (1\ mmol,\ 0.0297\ g)$  were placed in a thick Pyrex tube (ca. 20 cm in length). After addi-

# Table 3

Selected bond lengths (Å) and angles (°) for  ${\bf 2}.$ 

Cd—N2 Cd—O1B Cd—O1w	2.268(4) 2.284(3) 2.315(4)	Cd—N3A Cd—O2C Cd—O1C	2.280(4) 2.305(3) 2.446(3)
N2-Cd-N3A N3A-Cd-O1B N3A-Cd-O2C N2-Cd-O1w O1B-Cd-O1w N2-Cd-O1C O1B-Cd-O1C O1w-Cd-O1C	117.82(14) 88.03(12) 138.39(12) 80.64(14) 159.75(14) 154.30(12) 113.68(11) 85.95(13)	N2-Cd-O1B N2-Cd-O2C O1B-Cd-O2C N3A-Cd-O1w O2C-Cd-O1w N3A-Cd-O1C O2C-Cd-O1C	82.84(12) 103.39(13) 103.28(12) 89.39(17) 91.91(16) 83.63(12) 55.03(10)

Symmetry codes: (A) x + 1/2, y, -z + 1/2, (B) -x - 2, y + 1/2, -z + 1/2. (C) -x - 3/2, y + 1/2, z.



**Fig. 1.** ORTEP view of **1** with thermal ellipsoids drawn at the 50% probability level. Hydrogen atoms have been omitted for clarity.



Scheme 2. Coordination modes of H<sub>2</sub>tbpc in complexes 1–2.

tion of ethanol (0.6 mL) and water (0.3 mL), the tube was frozen with liquid N<sub>2</sub>, evacuated and sealed with a torch. The tube was heated at 110 °C for 4 days, and after slowly cooling to room temperature, pale-yellow rhombic crystals were obtained in 62% yield (based on Zn<sup>2+</sup>). Anal. (%) Calc. for **1**,  $C_{14}H_8N_4O_2Zn$ : C, 51.01; H, 2.45; N, 17.00. Found: C, 50.89; H, 253; N, 17.12. IR (cm<sup>-1</sup>, KBr): 3466 (s), 3130 (b, s), 1597 (s), 1545 (s), 1512 (w), 1456 (m), 1404 (s), 1367 (m), 1286 (w), 1244 (w), 1176 (w), 1138 (w), 1105 (w), 1009 (w), 852 (w), 791 (w), 768 (m), 716 (w), 692 (w), 648 (w), 588 (w), 490 (w).

# 2.3.2. [Cd(tbpc)(H<sub>2</sub>O)] (2)

H<sub>2</sub>tbpc (1 mmol, 0.0265 g) and CdCl<sub>2</sub> (1 mmol, 0.0228 g) were placed in a thick Pyrex tube (ca. 20 cm in length). After addition of 2-butanol (1.5 mL), triethylamino (0.1 mL) and water (0.5 mL), the tube was frozen with liquid N<sub>2</sub>, evacuated and sealed with a torch. The tube was heated at 165 °C for 5 days, and after slow cooling to room temperature, colorless blocked crystals were obtained in 45% yield (based on Cd<sup>2+</sup>). Anal. (%) Calc. for **2**, C<sub>14</sub>H<sub>10</sub>N<sub>4</sub>O<sub>3</sub>Cd: C, 42.61; H, 2.55; N, 14.20. Found: C, 42.49; H, 2.68; N, 14.37. IR (cm<sup>-1</sup>, KBr): 3543 (m), 3128 (b, s), 1607 (m), 1580 (m), 1518 (s), 1458 (m), 1402 (s), 1240 (w), 1182 (w), 1161



Fig. 3. The (3,6)-connected topologic structure of 1.

(w), 1103 (w), 1011 (w), 866 (w), 789 (w), 762 (m), 711 (w), 690 (w), 596 (w), 488 (w), 449 (w).

#### 2.4. Single-crystal X-ray diffraction measurements

Single-crystal data were collected at 293(2) K on a Rigaku SCXmini CCD diffractometer equipped with graphite-monochromated Mo Ka radiation. The structure was solved using direct methods and successive Fourier difference synthesis (SHELXS-97) [28], and refined using the full-matrix least-squares method on  $F_2$  with anisotropic thermal parameters for all nonhydrogen atoms (SHEL-XL-97) [29]. Hydrogen atoms bonded to the carbon atoms were



Fig. 2. A two-dimensional network structure for complex 1 in bc plane. Biphenyl groups have been omitted for clarity.

placed in calculated positions and refined as riding mode, with C—H = 0.93 Å and  $U_{iso}(H) = 1.2 U_{eq}(C)$ . The water hydrogen atoms were located in the difference Fourier maps and refined with an O—H distance restraint [0.90 Å] and  $U_{iso}(H) = 1.5U_{eq}(O)$ . The crystal data, details on the data collection and refinement of two complexes **1** and **2** are summarized in Table 1. Selected bond distances and angles are given in Tables 2 and 3.

# 3. Results and discussion

#### 3.1. IR analysis

In contrast to H<sub>2</sub>tbpc, the absence of any strong bands around 1680  $\text{cm}^{-1}$  in the IR spectra of **1** and **2** indicates that the –COOH has been completely deprotonated to generate COO<sup>-</sup> anion, which are agreement with their X-ray single structures.  $v_{as}(COO^{-})$  and  $v_{\rm s}({\rm COO^{-}})$  were observed at 1597 cm<sup>-1</sup> and 1404 cm<sup>-1</sup> for **1**, 1607 (s)  $cm^{-1}$  and 1404 (s) for **2**. The difference between asymmetric stretching and symmetric stretching bands of the carboxyl groups  $(\Delta v = v_{as}(C=0) - v_s(C=0))$  are 193 cm<sup>-1</sup> and 205 cm<sup>-1</sup>, respectively, which indicates strong coordination of the carboxylate oxygen to the transition metal center in a 1,3-bidentate bridging fashion. In compound **2**,  $v_{as}(COO^{-})$  and  $v_{s}(COO^{-})$  were observed at 1518 cm<sup>-1</sup> and 1404 cm<sup>-1</sup>, the difference between asymmetric stretching and symmetric stretching bands of the carboxyl group  $(\Delta v = v_{as}(C=0) - v_{s}(C=0))$  is 104 cm<sup>-1</sup>. The  $\Delta v$  value is in agreement with chelate coordination modes of carboxylates [30,31]. The presence of new bands at 1456, 1458 cm<sup>-1</sup> assigned to the stretching of the ring of tetrazole, and the broad bands at about 3130 cm<sup>-1</sup> indicate the presence of water molecules.

#### 3.2. Crystal structure of [Zn (tbpc)] (1)

The single crystal X-ray diffraction analysis reveals that compound **1** crystallizes in the centric space group  $P2_1/c$ . The asymmetric unit of compound **1** contains one Zn atom, one tbpc ligand (Fig. 1). The coordination geometry of each Zn(II) ion is a slightly distorted tetrahedron, in which the Zn(II) ion is fourcoordinated by two carboxylate oxygen atoms (O1B and O2A) and two tetrazole nitrogen atoms (N1 and N3C) from four symmetrically related tbpc ligands, with the angles N/O–Zn–N/O varying from 101.71(10) to 116.55(10)°. The two Zn–O bond lengths are 1.931(2) and 1.937(2) Å, and the Zn–N bond lengths are 2.001(3) and 2.004(2) Å, respectively. In tbpc, the dihedral angle between tetrazole ring and each phenyl ring is 76.43° or 50.82°, and that between phenyl rings is 76.72°.

In complex **1**, each tbpc ligand adopts a  $\mu^4$ -kN, kN', kO, kO' quadridentate bridging mode (Scheme 2a). Two oxygen atoms of the carboxylate group bridge two metal centers while the tetrazolate group bridges two metal centers through its 1- and 3-nitrogen atoms. Through the 1- and 2-oxygen atoms of carboxylate groups, two ligands bridge adjacent two Zn atoms to form a dimer (Zn<sub>2</sub>C<sub>2</sub>O<sub>4</sub>), which is a 8-membered metallocycle. The distance of Zn...Zn is 3.8969(9) Å in a dimer. In bc plane, each dimer is connected with four dimers through nitrogen atoms (N1, N3) of tetrazole rings to form a 2D layer (Fig. 2). Biphenyl groups connected tetrazole rings and carboxylate groups in a layer (Fig. S1, Fig. S2).



**Fig. 5.** ORTEP view of **2** with thermal ellipsoids drawn at the 50% probability level. Hydrogen atoms have been omitted for clarity.



**Fig. 4.** The C–H··· $\pi$  interaction between adjacent layers in **1**.



Fig. 6. A two-dimensional network structure for complex 2 in ab plane. Water molecules have been omitted for clarity.



Fig. 7. The (4,4)-connected topologic structure of 2.

To get better insight into the framework structure of **1**, topological analysis was carried out [32–34]. As discussed above, each dimer may be considered as a six-connecting node, and each residue of tbpc can be considered as three-connecting spacer. According to the simplification principle, the resulting structure of **1** is "kgd" and has a topology with Schläfli symbol of {4<sup>3</sup>}2{4<sup>6</sup>.6<sup>6</sup>.8<sup>3</sup>} (Fig. 3). As shown in Fig. S3, the layers are parallel to the *bc* plane and stack in an AAAA sequence to form a 3D supramolecular architecture. With a closer inspection of the structure,  $C_{phenyl}$ —H… $\pi$  [2.881 Å, 161.162(243)°] interactions are observed (Fig. 4), which is responsible for strengthening of the 3D assembly along *a* axis [35,36].

# 3.3. Crystal structure of $[Cd(tbpc)(H_2O)]$ (2)

Compound 2 crystallizes in the centric space group Pbca. The asymmetric unit of 2 contains one Cd atom, one tbpc ligand and one coordinated water molecule. The coordination geometry of each Cd(II) ion is a severely distorted octahedron, in which the Cd(II) ion is coordinated by one water oxygen atom (O1w), three carboxylate oxygen atoms (O1B, O1C and O2C), and two nitrogen atoms (N2 and N3A) from four tbpc ligands (Fig. 5). The four Cd-O bond lengths range from 2.284(3) to 2.446(3) Å, and the Cd-N bond lengths are 2.268(4) and 2.280(4) Å, respectively, which are close to those reported in Cd complexes. The bond angles around Cd vary from  $55.03(10)^{\circ}$  to  $159.75(14)^{\circ}$ . In tbpc, the dihedral angle between tetrazole ring and each phenyl ring is 42.94° or 57.17°, respectively, and that between phenyl rings is 54.07°. Obviously, the dihedral angles in 2 are very different from corresponding ones in **1**. In complex **2**, each tbpc ligand, exhibiting a quadridentate  $\mu^4$ kN, kN', kO, OkO' coordination mode [Scheme 2b], is bound to four Cd(II) atoms while each Cd(II) atom is linked to four tbpc groups, thus resulting in a 2D layered  $\{Cd(tbpc)(H_2O)\}_n$  (Fig. S4, Fig. 6). The overall structure of 2 is a (4,4)-connected binodal layer when Cd and tbpc are considered as nodes. An analysis by Topos reveals that Schläfli symbol is  $\{4^4.6^2\}\{4^4.6^6.8^4.10\}\{6\}$  (in Fig. 7). As shown in Fig. 8, the linking order between tbpc and Cd ions is reversed along *b* axis in adjacent layers, which are parallel to the *ab* plane and are stacked in an *ABAB* sequence. The  $C_{phenyl}$ — $H \cdots \pi$  [3.094 Å, 134.637(343)°] interaction is also observed between adjacent  $\{Cd(tbpc)(H_2O)\}_n$  layers. The 3D supramolecular architecture is formed through  $C_{phenyl}$ — $H \cdots \pi$  interactions.

#### 3.4. XRPD patterns of compounds 1 and 2

Compounds **1** and **2** were characterized by X-ray powder diffraction (XRPD) at room temperature. As shown in Figs. 9 and 10,



**Fig. 8.** The layers are stacked in an *ABAB* sequence with the C–H··· $\pi$  interactions.



Fig. 9. Experimental and simulated X-ray powder diffraction patterns of 1.



Fig. 11. TG-DTA curve of 1.





Fig. 10. Experimental and simulated X-ray powder diffraction patterns of 2.

Fig. 12. TG-DTA curve of 2.

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.

#### 3.5. Thermogravimetric analysis

Thermal stability of **1** and **2** was studied in this context. The thermogravimetric analysis (TGA) on the crystalline sample (**1**, 3.187 mg and **2**, 4.763 mg) was performed under a nitrogen atmosphere in flowing N<sub>2</sub> with a heating rate of 10 °C min<sup>-1</sup>. As depicted in Figs. 11 and 12, one can see that complexes are stable enough at room temperature. There is no noticeable weight loss for **1** until a temperature above 315 °C is reached, presumably due to decomposition of the network. One consecutive weight loss 4.86% occurs from 110 °C to 268 °C (calc. 4.56%) for **2**, which is corresponded to the loss of one coordinating water molecule per formula. There is no weight loss for it until a temperature above 309 °C is reached, presumably attribute to decomposition of the network. It is easily concluded that the  $[Zn(tbpc)]_n$  and  $[Cd(tbpc)]_n$  framework are very stable and worthy of further investigation as candidates of thermally stable materials.

#### 4. Conclusions

In this work, 2'-(1H-tetrazol-5-yl)-[1,1'-biphenyl]-4-carboxylic acid has been synthesized and characterized. Two 2D metal–organic frameworks: **1** and **2** have been successfully isolated by the reaction of Zn(II)/Cd(II) salts with H<sub>2</sub>tbpc under hydrothermal conditions. Since the different reaction temperature, assistant solvent and coordination numbers of center metal atom, the connection modes of tbpc adopt  $\mu^4$ -kN, kN', kO, kO' and  $\mu^4$ -kN, kN', kO, OkO' in **1** and **2**, respectively. C<sub>phenyl</sub>—H··· $\pi$  stacking interactions contribute to the stabilization of the crystal packing of **1** and **2** into 3D supramolecular structure. The thermogravimetric analysis reveals that the two complexes are potential thermally stable materials.

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#### Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molstruc.2010.03.074.

CCDC 763392 and 7763393 contain the supplementary crystallographic data for **1** and **2**. These data can be obtained free of charge from the Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data\_request/cif.

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