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# Journal of Molecular Structure

journal homepage: www.elsevier.com/locate/molstruc



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#### ARTICLE INFO

Article history: Received 6 July 2011 Received in revised form 4 August 2011 Accepted 4 August 2011 Available online 17 August 2011

Keywords: Metal-organic framework 1,3,5-Benzenetricarboxylic acid d<sup>10</sup> Transition metal Hydrothermal synthesis Structure Thermal property

#### ABSTRACT

Two novel transition metal-organic frameworks,  $[Zn_6(OH)_3(BTC)_3(H_2O)_3]$ · $(H_2O)_3$ ]· $(H_2O)_3$ · $(H_2O)_3$ 

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# 1. Introduction

Metal-organic frameworks (MOFs) on the basis of self-assembly of metal ions and multifunctional ligands have attracted much attention for their enormous variety of interesting architectures [1-5] and beneficial properties such as porosity, catalysis, magnetism, luminescence and nonlinear optics [6-14]. In the selfassembly process, the construction of molecular architecture depends on the judicious choice of the organic ligands and metal centers [15-18]. Multicarboxylic ligands with suitable spacers, such as 1,4-benzenedicarboxylic acid (H<sub>2</sub>BDC) [19-26], 1,3,5benzenetricarboxylic acid (H<sub>3</sub>BTC) [27-37] and 1,2,4,5-benzenetetracarboxylic acid (H<sub>4</sub>BTEC) [38–41], have been widely researched, owing to the various coordination modes and structural features. H<sub>3</sub>BTC has been extensively chosen to construct novel metal-organic frameworks in view of its following characteristics: (1) it has three carboxylic ions for coordination, which exhibit different coordination fashions to be favored to construct different frameworks; (2) these carboxylate donor groups may be completely or partially deprotonated to compensate for the charge and (3) the carboxyl oxygen atoms are facilitating to form hydrogen bonds, and  $\pi$ - $\pi$  stacking interactions are easy to form between aromatic rings, which are good for stabilizing the whole supramolecular structure. Following our recent interest in the systematic studies on 1,3,5-benzenetricarboxylate coordinated with transition metals, we have synthesized nine new structures,  $[Co(H_2BTC)_2(H_2O)_4]$ ,  $[M_3(BTC)(HCOO)_4(H_2O)]$ ·  $[(CH_3)_2NH_2]$ ·H<sub>2</sub>O (M = Mn, Co, Ni),  $[Zn_3Na_2$  (BTC)\_2(HCOO)\_2(H\_2O)\_8], [ZnNa(BTC)(H\_2O)\_2]·H<sub>2</sub>O and  $[Zn_3Na(BTC)_2(HCOO)(H_2O)_3]$ ·2H<sub>2</sub>O,  $[Cd_3Na_6(BTC)_4(H_2O)_{12}]$ ·H<sub>2</sub>O and  $[Cd_3Na_2(BTC)_3(H_2O)_3]$ ·[H<sub>2</sub>N(CH<sub>3</sub>)\_2 ]·H<sub>2</sub>O [42–45]. As a sequel, we report here two novel 3D and 2D metal–organic frameworks based on d<sup>10</sup> transition metal (Zn(II) or Cd(II)) with 1,3,5-benzenetricarboxylate ligands,  $[Zn_6(OH)_3$  (BTC)<sub>3</sub>(H<sub>2</sub>O)<sub>3</sub>]·7H<sub>2</sub>O (**1**) and  $[Cd(BTC)(H_2O)]$ ·[HIM] (**2**). The syntheses, crystal structures and thermal properties of them have been discussed in detail.

# 2. Experimental

# 2.1. Materials and characterizations

All solvents and reagents for the syntheses were of analytical grade and used as received from commercial sources without further purification. Variable temperature powder X-ray diffraction (VTPXRD) data of the studied samples were collected on a Bruker D8 Discover diffractometer with Cu K $\alpha$  radiation ( $\lambda$  = 1.5418 Å) at 40 kV, 40 mA and a graphite monochromator at the secondary beam. The heating rate between two temperatures was 10 °C min<sup>-1</sup>. Each pattern was recorded after the corresponding temperature retained 600 s, within the 5–40° range (2 $\theta$ ) with a step size of 0.02° and a





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scanning rate of 10 s step<sup>-1</sup>. IR spectra were recorded on a Magna-IR 750 FTIR spectrophotometer in the region 4000–650 cm<sup>-1</sup>. Elemental analyses for C, H and N were carried out on an Elementar VarioEL III microanalyzer. Thermo-gravimetric-mass spectrometric (TG– MS) analyses were performed in air at a heating rate of 10 °C min<sup>-1</sup> from 50 to 800 °C, using a NETZSCH STA449C instrument.

# 2.2. Syntheses

# 2.2.1. [Zn<sub>6</sub>(OH)<sub>3</sub>(BTC)<sub>3</sub>(H<sub>2</sub>O)<sub>3</sub>]·7H<sub>2</sub>O (**1**)

A mixture of ZnCl<sub>2</sub> (2.0 mmol, 0.2725 g), H<sub>3</sub>BTC (1.0 mmol, 0.2101 g), NaOH (4 mmol, 0.1600 g) and distilled water (10.0 mL) was placed in a 23 mL Teflon-lined stainless autoclave. The autoclave was sealed, heated at 170 °C for 5 days. After cooling to room temperature, colorless crystals were filtered, washed with distilled water and left to air-dry to give about 0.27 g of **1** (yield about 65% based on H<sub>3</sub>BTC). Anal. Calcd for Zn<sub>6</sub>C<sub>27</sub>H<sub>32</sub>O<sub>31</sub> (fw 1244.75): C 26.05, H 2.59. Found: C 26.36, H 2.21.

#### 2.2.2. [Cd(BTC)(H<sub>2</sub>O)]·[HIM] (**2**)

**2** was hydrothermally prepared from Cd(OAc)<sub>2</sub>·2H<sub>2</sub>O (1.0 mmol, 0.2665 g), H<sub>3</sub>BTC (1.0 mmol, 0.2101 g), IM (IM = imidazole) (1 mmol, 0.0681 g) and distilled water (10.0 mL). The mixture was sealed in the 23 mL Teflon-lined stainless autoclave and heated at 170 °C for 5 days, and then cooled to room temperature. The colorless crystal-line products were filtered, washed with deionized water, and then dried in air to give about 0.36 g of **2** (yield 88% based on H<sub>3</sub>BTC). Anal. Calcd for CdC<sub>12</sub>H<sub>10</sub>O<sub>7</sub>N<sub>2</sub> (fw 406.62): C 35.44, H 2.47, N 6.89. Found: C 35.43, H 2.48, N 6.90.

#### 2.3. Crystallographic studies

Suitable single crystals of 1 and 2 were carefully selected under an optical microscope and glued to thin glass fibers with epoxy resin. The intensity data were collected on a Bruker SMART X-ray diffractometer, equipped with an APEX-CCD area detector and using graphite-monochromatic Mo K $\alpha$  ( $\lambda$  = 0.71073 Å) radiation at room temperature. The data absorption corrections were applied on the basis of symmetry-equivalent reflections using the ABSCOR program [46]. The structures were solved by the direct method and refined on  $F^2$  with full-matrix least-squares methods using the SHELXS-97 and SHELXL-97 programs, respectively [47,48]. All of the non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added in the riding model and refined isotropically with O-H = 0.82 Å (H<sub>2</sub>O) or O-H = 0.98 Å (OH), C-H = 0.93 Å and N-H = 0.86 Å. The crystallographic data and structural refinement parameters are presented in Table 1, and the selected bond lengths and angles are listed in Table S1 of the Supporting Information (SI).

# 3. Results and discussion

#### 3.1. Infrared (IR) spectra

IR spectra of **1** and **2** (Fig. S1 in SI) confirm the presence of the organic ligands used in the syntheses (through the characteristic bands of aromatic and carboxylate groups). The broad absorption bands of the asymmetric and symmetric stretching vibrations of water (compounds **1** and **2**) and imidazole (compound **2**) appear at 3700–2600 cm<sup>-1</sup> [49–51]. The bands at 1629–1544 cm<sup>-1</sup> and 1444–1357 cm<sup>-1</sup> correspond to the asymmetric and symmetric stretching vibrations of the bound carboxylate groups (CO<sub>2</sub>M), respectively [49–53]. The out-of-plane deformation vibrations of the C–H groups in the benzene ring give absorption bands from 773 to 713 cm<sup>-1</sup> [50,51]. The absence of the absorption bands from

#### Table 1

Crystallographic and structural refinement parameters of 1 and 2.

	1	2
Formula	Zn <sub>6</sub> C <sub>27</sub> H <sub>32</sub> O <sub>31</sub>	CdC <sub>12</sub> H <sub>10</sub> O <sub>7</sub> N <sub>2</sub>
Fw	1244.75	406.62
Color, habit	Colorless, needle	Colorless, block
Crystal dimensions (mm)	$0.15 \times 0.15 \times 0.30$	$0.10 \times 0.15 \times 0.30$
Temperature (K)	298	298
Crystal system	Trigonal	Monoclinic
Space group	R3c	$P2_1/c$
a (Å)	30.159(4)	6.5160(13)
b (Å)	30.159(4)	9.6687(19)
c (Å)	6.9280(14)	19.524(4)
$\beta$ (deg)	90.00	96.23(3)
$V(Å^3)$	5457.2(16)	1222.8(4)
Ζ	6	4
$D_{\text{calcd}}$ (g cm <sup>-3</sup> )	2.273	2.209
λ (Mo Kα) (Å)	0.71073	0.71073
$\mu (\mathrm{mm}^{-1})$	4.013	1.829
Absorption correction	Multi-scan	Multi-scan
Theta range for data collection	2.34, 33.49	2.00, 32.50
(deg)		
F(000)	3732	800
GOF on F <sup>2</sup>	1.004	1.008
R <sub>int</sub>	0.0437	0.0219
R1, wR2 $[I > 2(I)]$	0.0519, 0.1281	0.0340, 0.0989
R1, wR2 (all data)	0.0619, 0.1348	0.0382, 0.1019
Largest diff. peak and hole $(Å^{-3})$	2.42, -0.79	1.48, -1.12
CCDC number	823406	829422

1680 to 1800 cm<sup>-1</sup> indicates the complete deprotonation of BTC ligands [52].

# 3.2. Crystal structure of [Zn<sub>6</sub>(OH)<sub>3</sub>(BTC)<sub>3</sub>(H<sub>2</sub>O)<sub>3</sub>]·7H<sub>2</sub>O (**1**)

The single crystal X-ray diffraction analysis suggests compound **1** is in the trigonal space group *R*3*c* with the Flack parameter about 0.029(18), indicating that the absolute structure given by the structure refinement is likely correct. In the asymmetric unit of 1, there are two crystallographic independent Zn(II) cations, one hydroxyl group, one BTC ligand, one coordinated H<sub>2</sub>O and three solvated water molecules (Fig. 1a). Each Zn(1) center exhibits a four coordinated tetrahedron configuration composed by two carboxyl oxygen atoms (O(2), O(4)) from two BTC ligands, and two oxygen atoms  $(O(1) \text{ and } O(1)^i)$  from two hydroxyl groups. While, each Zn(2) atom is six-coordinated with a distorted octahedron by four BTC ligands (O(3), O(5), O(7) and O(8)), one hydroxyl group (O(1))and a water molecule (O(6)). The lengths of Zn–O bonds are in the range 1.959–2.280 Å (Table S1 in SI). Each BTC in 1 is completely deprotonated, bridging two separate Zn(1) and four Zn(2) atoms (Fig. S2a in SI). All of the three carboxyl groups adopt the bidentate fashion: both the first and the second one connect one Zn(1) and one Zn(2), and the third one links two Zn(2).

As shown in Fig. 1b,  $Zn(1)O_4$  tetrahedra and  $Zn(2)O_6$  octahedra link to each other through  $\mu_3$ -O atoms of hydroxyl groups to form a zigzag chain of  $(Zn_2O_8)_n$  along *c* axis. Such zigzag chains are connected by the bridging BTC ligands parallel the *ab* plane to form a 3D network with 1D channels of *ca*. 5.6 × 5.6 Å along *c* direction (Fig. 1c). The guest water molecules occupy the void of the structure and interact with the framework through hydrogen bonds (Table S2 in SI). The effective free volume of the dehydrated framework was estimated by PLATON/SOLV to be 23.2% of the total volume (1265.0 Å<sup>3</sup> out of the 5457.2 Å<sup>3</sup> unit cell volume). However, N<sub>2</sub> and H<sub>2</sub> adsorption appeared to fail for dehydrated form of **1**.

# 3.3. Crystal structure of [Cd(BTC)(H<sub>2</sub>O)]·[HIM] (2)

Crystallographic analysis reveals that compound **2** crystallizes in the monoclinic space group  $P2_1/c$ . As illustrated in Fig. 2a, the



**Fig. 1.** Structural details of **1** drawn by Software Atoms 6.0 [54]. (a) Thermal ellipsoid plot (50%) drawing of the coordination environment of Zn(II) centers. Symmetry codes: (i) -y, -x, z + 1/2; (ii) -y, x - y, z; (iii) x, x - y, z + 1/2; (iv) -x + y + 1/3, y + 2/3, z + 7/6; (v) -x + y + 1/3, -x + 2/3, z + 2/3. (b) Zigzag chain of (Zn<sub>2</sub>O<sub>8</sub>)<sub>n</sub> along the *c* axis. (c) 3D framework viewed along the *c* axis. (Color codes: Zn(1), ciel; Zn(2), green; C, gray; O, red and yellow; Zn(1)O<sub>4</sub> tetrahedra, ciel; Zn(2)O<sub>6</sub> octahedra, green. The free water molecules and hydrogen atoms are omitted for clarity.) (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)



**Fig. 2.** Structural details of **2** drawn by Software Atoms 6.0 [54]. (a) Thermal ellipsoid plot (50%) drawing of the coordination environment of Cd(II) ions. Symmetry codes: (i) -x, y + 1/2, -z + 1/2; (ii) -x, -y, -z. (b) The 2D layer parallel the *bc* plane. (c) 3D framework viewed along the *b* axis. (Color codes: Cd, ciel; C, gray; O, red; N, purple; Cd(1)O<sub>6</sub> octahedra, ciel. The hydrogen atoms are omitted for clarity.) (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

asymmetric unit of **2** contains one crystallographic unique Cd(II) cation, one BTC ligand, one terminal water molecule and one imidazole cation. Each Cd(1) center shows six-coordinated fashions to form distorted octahedra bound with five carboxyl oxygen atoms (O(1), O(2), O(3), O(5) and O(6)) from four BTC ligands and one oxygen atom (O(4)) from one terminal water. The distances of the Cd–O bonds range from 2.217 to 2.558 Å (Table S1 in SI). Compound **2** presents one type of completely deprotonated BTC connecting four Cd(1) atoms (see Fig. S2b in SI): the first carboxyl group is of the chelating bidentate mode linking one Cd(1) atom, the second one uses the bidentate fashion to connect with two Cd(1) atoms, while the third one adopts the unidentate way to bind with one Cd(1) atom.

Such BTC ligands link  $Cd(1)O_6$  octahedra to build up a 2D network placed in the *bc* plane of the unit cell (Fig. 2b). The imidazole cations reside in the void of the structure. The cooperative N–H···O and O–H···O interactions connect with imidazole cations, coordinated water molecules and carboxylate groups to form hydrogen-bonded network (Table S2 in SI), which establishes physical connections between neighboring layers (Fig. 2c).

#### 3.4. Thermal properties

The thermal stabilities of compound 1 and 2 were measured by TG-MS and VTPXRD (shown in Fig. 3) in air, and related data indicated that 1 and 2 were stable up to  $320 \,^{\circ}$ C and  $220 \,^{\circ}$ C, respectively.

Thermal decomposition of compound **1** proceeded with two distinct weight losses between 50 and 800 °C. The first weight loss was attributed to the departure of all the water molecules in the temperature range of 80-200 °C (Found 14.01 wt.%, Calc.

14.47 wt.%), without further weight loss up to 320 °C (Fig. 3a). The second loss corresponded to the release of the organic part occurred from 320 to 500 °C (Found 46.11 wt.%, Calc. 46.30 wt.%), leading to ZnO as the residue (Found 39.88 wt.%, Calc. 39.22 wt.%). The TG analysis was confirmed by VTPXRD patterns (Fig. 3b). The ion currents of m/z = 17 (HO<sup>+</sup>) and 18 (H<sub>2</sub>O<sup>+</sup>) appeared in above two steps, whereas the MS peaks of ions with m/z = 28 (CO<sup>+</sup>) and 44 (CO<sup>+</sup><sub>2</sub>) were observed in the second process (Fig. 3a).

The TG-MS analysis of compound 2 was demonstrated in Fig. 3c. Compound 2 lost the coordinated water molecules in the range of 100-220 °C (Found 4.79 wt.%, Calc. 4.43 wt.%). The ion currents showed peaks at these temperature only for ions with m/z = 17 and 18, which may be assigned to HO<sup>+</sup> and H<sub>2</sub>O<sup>+</sup>, respectively. And the destruction of the material occurred from 220 to 470 °C (Found 63.75 wt.%, Calc. 63.99 wt.%), founding HO<sup>+</sup>, H<sub>2</sub>O<sup>+</sup>,  $CO^+$  and  $CO^{2+}$  (*m*/*z* = 17, 18, 28 and 44, respectively) by MS. The VTPXRD studies (Fig. 3d) revealed the structure change of compound 2 in the weight loss process. The diffraction pattern measured at 220 °C showed an apparent shift towards the lower angle  $2\theta$  of the peaks due to expansion of the framework with the removal of coordinated water molecules. Interestingly, the VTPXRD analysis exhibited that **2** undergone a distinct structural transformation to form a new compound at 300 °C. And the resulting phase at the higher temperatures (above 500 °C) was CdO corresponding to the TG residue (Found 31.46 wt.%, Calc. 31.58 wt.%).

# 4. Conclusions

We have successfully combined d<sup>10</sup> transition metal (Zn(II) or Cd(II)) and the aromatic BTC ligands to afforded two novel metal–organic frameworks through hydrothermal syntheses.



Fig. 3. (a) TG-MS curves for 1; (b) VTPXRD patterns for 1; (c) TG-MS curves for 2; (d) VTPXRD patterns for 2.

Compound **1** presents a 3D framework formed by  $(Zn_2O_8)_n$  zigzag chains of  $(Zn_2O_8)_n$  and bridging BTC anions with 1D channels of ca. 5.6  $\times$  5.6 Å along [0 0 1] direction, while **2** constructs a 2D network placed in the *bc* plane, consisting of  $Cd(1)O_6$  octahedra and BTC ligands. The two illustrations demonstrate that cooperating d<sup>10</sup> transition metals with bridging BTC ligands may provide a useful platform for novel metal-organic frameworks with priming constructions.

#### Acknowledgment

This work is supported the National Key Basic Research Project of China (Grant 2010CB833103).

#### Appendix A. Supplementary data

CCDC 823406 and 829422 contain the supplementary crystallographic data for 1 and 2. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via http:// www.ccdc.cam.ac.uk/data\_request/cif. Supplementary data associated with this article can be found, in the online version, at doi: XXX. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molstruc.2011.08.013.

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