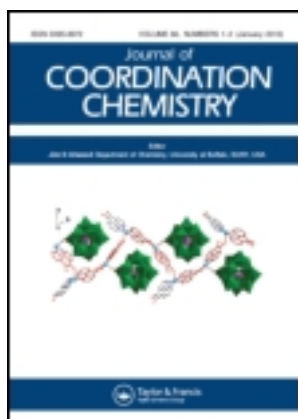


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1D, nano-dimensional pore and square grid metal-organic polymers based on 4,4'-oxybisbenzoate; synthesis, characterization, and thermal properties

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1D, nano-dimensional pore and square grid metal-organic polymers based on 4,4'-oxybisbenzoate; synthesis, characterization, and thermal properties

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Three new zinc(II) coordination polymers, $[\text{Zn}(\text{imi})_2(\mu_2\text{-oba})]_n \cdot n\text{H}_2\text{O}$ (**1**), $[\text{Zn}(2,2'\text{-bipy})(\mu_2\text{-oba})(\text{H}_2\text{O})]_n \cdot n\text{H}_2\text{O}$ (**2**), and $[\text{Zn}_2(4,4'\text{-bipy})(\mu_2\text{-oba})_2(\text{H}_2\text{O})_2(\text{DMF})_2]_n \cdot n\text{DMF}$ (**3**) (oba = 4,4'-oxybisbenzoate, imi = imidazole), have been synthesized and structurally characterized by single-crystal X-ray crystallography and FT-IR spectroscopy. The coordination polymers **1–3** have 1D, nano-dimensional pore and square grid structures. The thermal stabilities of **1–3** were studied by thermal gravimetric and differential thermal analysis.

Keywords: Nano-dimensional square grid; Zinc(II); Coordination polymer; 4,4'-Oxybis(benzoic acid)

1. Introduction

Metal-organic coordination polymers contain metal ions linked by ligands into an infinite array [1–6], incorporating architectures ranging from simple 1D chains with small ligands to large micro and mesoporous frameworks, metal-organic frameworks (MOFs) [7, 8]. In the design and construction of these frameworks ligands play crucial roles as even small changes in flexibility, length, or symmetry of the ligands result in diverse architectures and functions [9–27]. Thus, the prospect of tuning properties of MOFs through systematic change of ligands provides impetus for further research on metal-organic supramolecular architectures. However, obtaining desirable architectures of coordination polymers constructed by ligands and metal ions is still a long-term challenge due to the difficult prediction of either the compositions or the structures of the reaction products, especially those from nonrigid ligands because of their flexibility and conformational freedom in the assembly process.

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4,4'-Oxybisbenzoate, a highly flexible ligand, is widely used owing to free rotation of the two benzoate planes, which can meet the requirement of coordination geometries of metal ions in assembly [28–31]. In this article, we have synthesized and characterized three zinc(II) coordination polymers with 4,4'-oxybisbenzoate and imidazole/2,2'-bipyridine/4,4'-bipyridine, $[\text{Zn}(\text{imi})_2(\mu_2\text{-oba})]_n \cdot n\text{H}_2\text{O}$ (**1**), $[\text{Zn}(2,2'\text{-bipy})(\mu_2\text{-oba})(\text{H}_2\text{O})]_n \cdot n\text{H}_2\text{O}$ (**2**) and $[\text{Zn}_2(4,4'\text{-bipy})(\mu_2\text{-oba})_2(\text{H}_2\text{O})_2(\text{DMF})_2]_n \cdot n\text{DMF}$ (**3**). Thermal properties of **1–3** were investigated.

2. Experimental

2.1. Materials and methods

All commercially available chemicals are reagent grade quality and used without purification. IR data were recorded on a Thermo Scientific Nicoletti. Thermogravimetric analyses (TGA) were collected on a Perkin-Elmer Pyris1. TGA analyses were performed from room temperature to 800 °C with a heating rate of 20 °C min⁻¹ under nitrogen. Single-crystal analyses were performed with the SMART APEXII CCD diffractometer for **1–3** using graphite monochromated Mo- K_α radiation ($\lambda = 0.71073 \text{ \AA}$) at 298(2) K. Accurate unit cell parameters and orientation matrices for data collection were obtained from least-squares refinement. The structures were solved by direct methods and refined by full-matrix least-squares on F^2 . Crystal data and refinement parameters are given in table 1. Selected bond lengths and angles are given in tables 2–4.

Table 1. Crystallographic data for **1–3**.

Identification code	1	2	3
Empirical formula	C ₂₀ H ₁₈ N ₄ O ₆ Zn	C ₂₄ H ₂₀ N ₂ O ₇ Zn	C ₂₅ H ₂₈ N ₃ O ₈ Zn
Formula weight	475.75	513.79	563.87
<i>T</i> /K	298(2)	298(2)	298(2)
$\lambda/\text{\AA}$	0.71073	0.71073	0.71073
Crystal system	Orthorhombic	Triclinic	Triclinic
Space group	Pnna	P $\bar{1}$	P $\bar{1}$
<i>a</i> [Å]	15.302(2)	9.7165(5)	8.5866(6)
<i>b</i> [Å]	13.4686(17)	11.0980(6)	11.9990(8)
<i>c</i> [Å]	9.8714(12)	14.6325(12)	14.0365(9)
α [°]	90	104.177(2)	109.0130(10)
β [°]	90	95.665(2)	103.5130(10)
γ [°]	90	112.6330(10)	97.318(2)
<i>V</i> [Å ³]	2034.5(4)	1378.79(15)	1296.46(15)
<i>Z</i>	4	2	2
<i>D</i> _{calcd} [g cm ⁻³]	1.553	1.238	1.444
<i>F</i> (000)	976	528	586
<i>R</i> (int)	0.0203	0.0916	0.0191
Data/restraints/parameters	1895/0/142	4767/30/334	4589/24/334
<i>GOF</i> on F^2	1.102	1.065	1.075
<i>R</i> ^a [$I > 2\sigma(I)$]	0.0293	0.0771	0.0396
<i>wR</i> ₂ ^b [$I > 2\sigma(I)$]	0.0970	0.2054	0.1232
<i>R</i> ^a (all data)	0.0334	0.0886	0.0419
<i>wR</i> ₂ ^b (all data)	0.0996	0.2171	0.1255
Largest diff. peak/hole, e.Å ⁻³	0.33/−0.386	1.284/−0.600	0.848/−0.640

$$R_1^a = \Sigma |F_o| - |F_c| / \Sigma F_o, \quad wR_2^b = [\Sigma w(F_o^2 - F_c^2)^2 / \Sigma w(F_o^2)]^{1/2}.$$

Table 2. Selected bond lengths (Å) and angles (°) for 1.

Zn(1)–O(2)	1.9501(16)	O(2)–Zn(1)–O(2)#1	129.93(9)
Zn(1)–O(2)#1	1.9501(16)	O(2)–Zn(1)–N(1)#1	115.66(7)
Zn(1)–N(1)#1	2.0204(18)	O(2)#1–Zn(1)–N(1)#1	95.08(7)
Zn(1)–N(1)	2.0204(18)	O(2)–Zn(1)–N(1)	95.08(7)
		O(2)#1–Zn(1)–N(1)	115.66(7)
		N(1)#1–Zn(1)–N(1)	103.92(11)
		C(1)–O(2)–Zn(1)	116.41(14)
		C(5)–O(3)–C(5)#2	118.0(3)

Symmetry transformations used to generate equivalent atoms: #1 $x, -y + 3/2, -z + 1/2$; #2 $x, -y + 1/2, -z + 1/2$.

Table 3. Selected bond lengths (Å) and angles (°) for 2.

Zn(1)–O(2)	2.039(3)
Zn(1)–N(1)	2.108(4)
Zn(1)–O(6)	2.129(3)
Zn(1)–N(2)	2.150(3)
Zn(1)–O(1)	2.437(3)
Zn(1)–C(11)	2.567(4)
O(4)#1–Zn(1)–O(2)	101.13(13)
O(4)#1–Zn(1)–N(1)	108.54(15)
O(2)–Zn(1)–N(1)	148.96(15)
O(4)#1–Zn(1)–O(6)	92.33(13)
O(2)–Zn(1)–O(6)	92.25(15)
N(1)–Zn(1)–O(6)	95.40(14)
O(4)#1–Zn(1)–N(2)	92.20(13)
O(2)–Zn(1)–N(2)	93.52(14)
N(1)–Zn(1)–N(2)	76.72(14)
O(6)–Zn(1)–N(2)	171.85(13)
O(4)#1–Zn(1)–O(1)	158.18(13)

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

Table 4. Selected bond lengths (Å) and angles (°) for 3.

Zn(1)–O(1)	1.9982(17)	O(1)–Zn(1)–O(5)#1	109.03(8)
Zn(1)–O(5)#1	2.066(2)	O(1)–Zn(1)–O(6)	105.81(8)
Zn(1)–O(6)	2.071(2)	O(5)#1–Zn(1)–O(6)	145.07(8)
Zn(1)–O(8)	2.130(2)	O(1)–Zn(1)–O(8)	92.03(8)
Zn(1)–N(1)	2.159(3)	O(5)#1–Zn(1)–O(8)	89.97(9)
Zn(1)–O(4)#1	2.480(2)	O(6)–Zn(1)–O(8)	91.28(9)
		O(1)–Zn(1)–N(1)	91.52(8)
		O(5)#1–Zn(1)–N(1)	90.16(10)
		O(6)–Zn(1)–N(1)	86.43(10)
		O(8)–Zn(1)–N(1)	176.19(8)
		O(1)–Zn(1)–O(4)#1	165.37(8)
		O(5)#1–Zn(1)–O(4)#1	56.92(7)
		O(6)–Zn(1)–O(4)#1	88.48(7)
		O(8)–Zn(1)–O(4)#1	84.37(8)
		N(1)–Zn(1)–O(4)#1	92.52(8)

Symmetry transformations used to generate equivalent atoms: #1 $x + 1, y + 1, z + 1$; #2 $x - 1, y - 1, z - 1$; #3 $-x + 3, -y + 2, -z + 1$.

2.2. Preparation of $[Zn(imi)_2(\mu_2-oba)]_n \cdot nH_2O$ (1)

A DMF solution (10 mL) containing imidazole (0.41 mM, 0.028 g) was added slowly to a solution (10 mL) of DMF containing $Zn(NO_3)_2 \cdot 6H_2O$ (0.15 mM, 0.043 g) and 4,4'-oxybis

(benzoic acid) (0.10 mM, 0.024 g). The mixture solution was left to stand at room temperature and in two months colorless crystals were obtained (yield: 44%). (Found: C, 50.41; H, 3.75; N, 11.68%. calculated for $C_{20}H_{18}N_4O_6Zn$: C, 50.49; H, 3.81; N, 11.78%). IR (cm^{-1}) selected bonds: $\nu=1670m, 1594s, 1544m, 1543m, 1383s, 1224s, 1156m, 1089w, 867m, 846m, 781m, 763s, 735m, 659m$.

2.3. Preparation of $[Zn(2,2'-bipy)(\mu_2-oba)(H_2O)]_n \cdot nH_2O$ (2)

A DMF solution (10 mL) containing 2,2'-bipyridine (0.15 mM, 0.023 g) was added slowly to a DMF solution (10 mL) containing $Zn(NO_3)_2 \cdot 6H_2O$ (0.18 mM, 0.054 g) and 4,4'-oxybis(benzoic acid) (0.15 mM, 0.036 g). The mixture solution was left to stand at room temperature and two months later colorless crystals were obtained (yield: 42%). (Found: C, 55.97; H, 3.85; N, 5.32%. calculated for $C_{24}H_{20}N_2O_7Zn$: C, 56.10; H, 3.92; N, 5.45%). IR (cm^{-1}) selected bonds: $\nu=1671w, 1592s, 1533s, 1543m, 1388s, 1238s, 1156s, 1027w, 873m, 789m, 763s, 734m, 661m$.

2.4. Preparation of $[Zn_2(4,4'-bipy)(\mu_2-oba)_2(H_2O)_2(DMF)_2]_n \cdot nDMF$ (3)

A DMF solution (5 mL) containing 4,4'-bipyridine (0.50 mM, 0.051 g) was added slowly to a mixed solution (10 mL, V/V=1) of DMF and water containing $Zn(NO_3)_2 \cdot 6H_2O$ (0.18 mM, 0.054 g) and 4,4'-oxybis(benzoic acid) (0.15 mM, 0.036 g). The mixture solution stood at room temperature and three months later giving red crystals (yield: 40%). (Found: C, 53.29; H, 4.78; N, 7.34%. calculated for $C_{25}H_{28}N_3O_8Zn$: C, 53.25; H, 5.00; N, 7.45%). IR (KBr, cm^{-1}) selected bonds: $\nu=1675w, 1651m, 1592s, 1537m, 1405s, 1380s, 1237s, 1154m, 1089w, 1011w, 873m, 783s, 765m, 685m, 659s$.

3. Results and discussion

Reaction between zinc(II) nitrate with 4,4'-oxybis(benzoic acid)/imidazole, 4,4'-oxybis(benzoic acid)/2,2'-bipyridine, or 4,4'-oxybis(benzoic acid)/4,4'-bipyridine yielded crystalline materials formulated as $[Zn(imi)_2(\mu_2-oba)]_n \cdot nH_2O$ (1), $[Zn(2,2'-bipy)(\mu_2-oba)(H_2O)]_n \cdot nH_2O$ (2), and $[Zn_2(4,4'-bipy)(\mu_2-oba)_2(H_2O)_2(DMF)_2]_n \cdot nDMF$ (3), respectively.

An IR spectrum displays absorptions for oba^{2-} , imi, 2,2'-bipy, 4,4'-bipy, DMF, and H_2O in 1–3. The relatively weak absorptions at 3055 and 3045 cm^{-1} are assigned to C–H modes involving aromatic ring hydrogens. Absorptions with variable intensity at 1400–1580 cm^{-1} correspond to C=C and C=N ring vibrations of oba^{2-} , imi, 2,2'-bipy and 4,4'-bipy ligands. The broad absorption at 3488 cm^{-1} is assigned to the $\nu(H_2O)$. The broadness is attributed to $\nu(O-H \cdots X)$, indicating the presence of hydrogen bonds, confirmed by crystal structure determination of 1–3.

Determination of the structure of 1 by X-ray crystallography (tables 1 and 2) showed this structure, crystallizes in the orthorhombic noncentrosymmetric space group $Pca2_1$, which originates from the lack of a center of inversion in oba. The solid is a 1D coordination polymer with zinc(II) four coordinate with tetrahedral ZnN_2O_2 (figure 1). Two imi molecules coordinate to zinc(II) by nitrogen and two oba connects to zinc(II) by one oxygen (figure 1), resulting in a 1D coordination polymer (figure 2a). Waters are not

coordinated and fill space between 1D chains. Hydrogen bonds and π - π stacking grow this polymer into a 3-D supramolecule (figure 2b). Every water connects to two coordinated oxygens from oba and also every N-H of imi connects to one uncoordinated oxygen from oba²⁻ via hydrogen bonding.

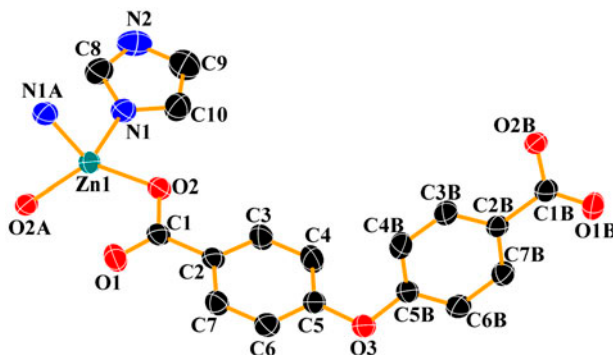


Figure 1. View of the coordination of Zn(II) in 1 with thermal ellipsoids at 50% probability. All hydrogens and waters have been omitted for clarity.

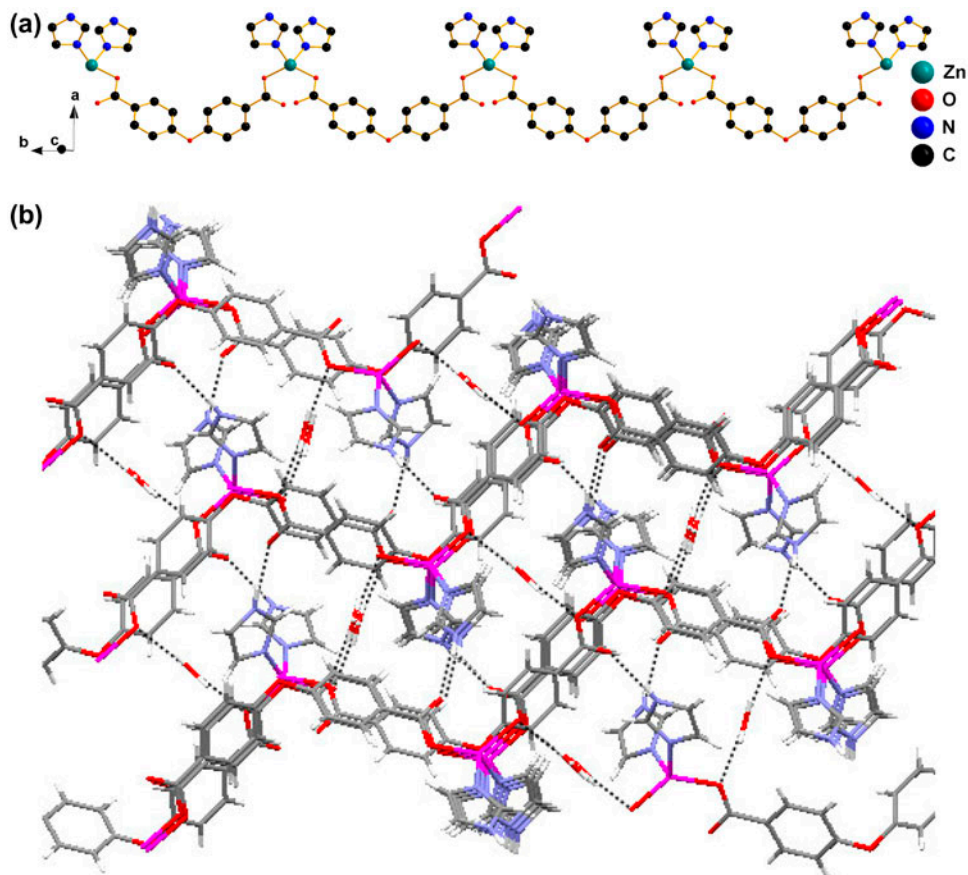


Figure 2. (a) 1-D metal-organic polymer and (b) hydrogen bonding in 1.

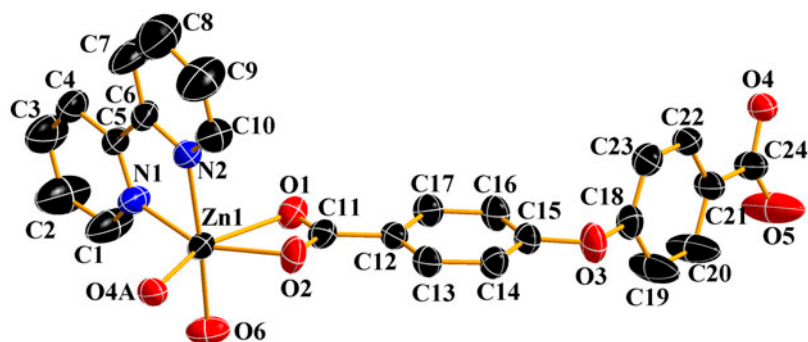


Figure 3. View of the coordination of Zn(II) in **2** with thermal ellipsoids at 50% probability. All hydrogens and waters have been omitted for clarity.

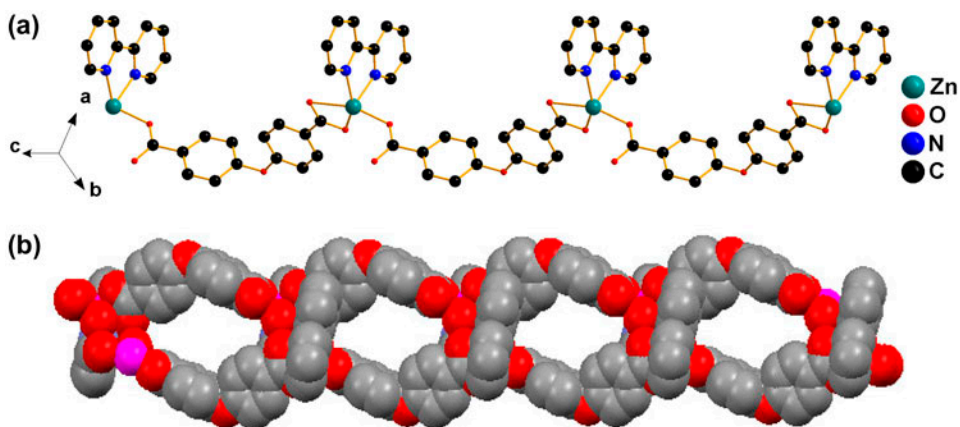


Figure 4. (a) 1-D metal-organic polymer and (b) nano-pores formed by hydrogen bonding and π - π stacking interactions in **2**.

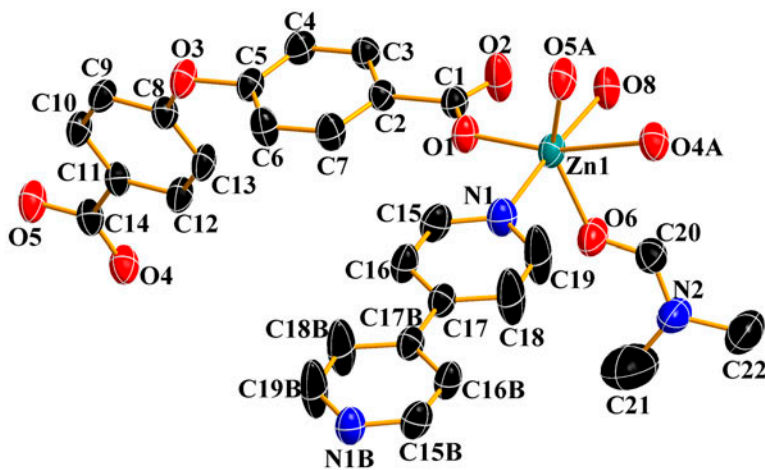


Figure 5. View of the coordination of Zn(II) in **3** with thermal ellipsoids at 50% probability. All hydrogens and waters have been omitted for clarity.

Complex **2** crystallizes in the triclinic space group $P\bar{1}$. The asymmetric unit consists of one Zn(II), one oba^{2-} , and one 2,2'-bipy. As shown in figure 3, Zn(II) is distorted octahedral, coordinated by three oxygens from oba , two nitrogens from 2,2'-bipy, and one water. In **2**, Zn(II) are connected by oba^{2-} generating two reverse parallel 1D wave-like single chains. Hydrogen bonds and π - π stacking grow this polymer into a supramolecular structure. These link two reverse parallel 1D wave-like single chains into an interesting 1D cycle (figure 4).

Compound **3** crystallizes in triclinic space group $P\bar{1}$. The asymmetric unit of **3** contains one Zn(II), one oba^{2-} , half 4,4'-bipy, one DMF, and one H_2O . As illustrated in figure 5, Zn(II) are coordinated by five oxygens from four oba^{2-} , DMF, and H_2O and one nitrogen from 4,4'-bipy, showing a ZnO_5 octahedron. Each oba and 4,4'-bipy connects two Zn(II)

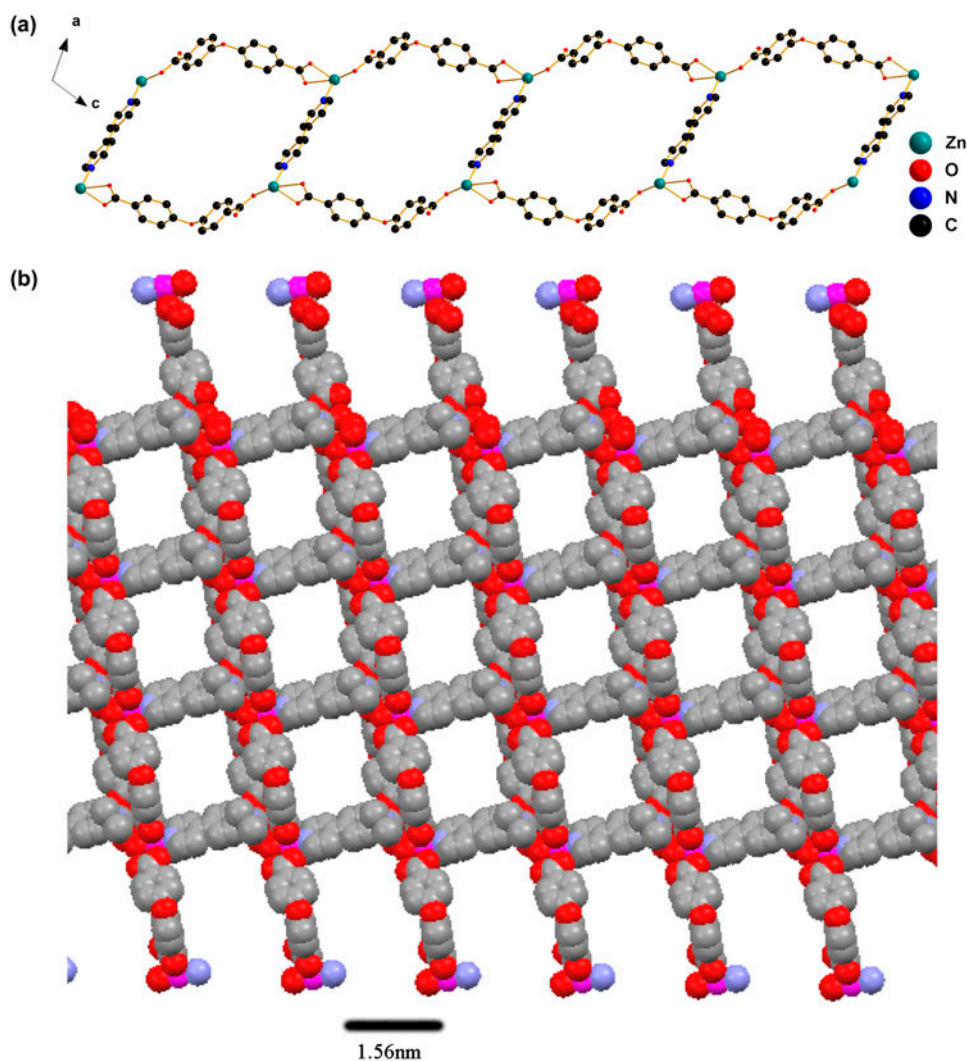


Figure 6. (a) The grid-like $\text{Zn}_4(\text{oba})_2(\text{oba})_2$ units and (b) 2D layer structure consisting of nano-dimensional 2D (4,4) square grid units in **3**.

ions to generate a 2D layer structure consisting of nano-dimensional 2D(4,4) square grid units in **3** (figure 6). The grid-like $\text{Zn}_4(\text{oba})_2(\text{oba})_2$ units have dimensions of 1.56×1.56 nm. However, the grid layers are tightly stacked parallel.

The thermal decomposition behaviors of $[\text{Zn}(\text{imi})_2(\mu_2\text{-oba})]_n \cdot n\text{H}_2\text{O}$ (**1**), $[\text{Zn}(2,2'\text{-bipy})(\mu_2\text{-oba})(\text{H}_2\text{O})]_n \cdot n\text{H}_2\text{O}$ (**2**), and $[\text{Zn}_2(4,4'\text{-bipy})(\mu_2\text{-oba})_2(\text{H}_2\text{O})_2(\text{DMF})_2]_n \cdot n\text{DMF}$ (**3**) were investigated in static nitrogen from ambient to 800 °C (Supplementary material.doc). The lattice waters in **1** are removed at 100 °C with a mass loss $\sim 4.20\%$, consistent with the calculated value 3.78% for the elimination of one H_2O and with an endothermic effect. The compound is then stable to 300 °C at which temperature it begins to decompose. Decomposition of two imi and one oba^{2-} takes place at 300–570 °C with four endothermic effects at 320, 340, 385, and 485 °C as well as an exothermic effect at 580 °C. The solid residue formed at 600 °C is assigned as ZnO. Compound **2** at 50 °C begins to decompose and removal of two waters takes place at 110 °C with experimental mass loss of 7.01%, consistent with calculated 6.60% for elimination of two H_2O with an endothermic effect at 105 °C. The weight loss from 310 to 570 °C is equivalent to the loss of one coordinated 2,2'-bipy and oba^{2-} with four exothermic effects. The solid residue formed at 600 °C is assigned as ZnO. Compound **3** is stable to 120 °C. Decomposition of **3** occurs between 120 and 660 °C with a mass loss of 85.2%. The solid residue formed at 660 °C is suggested to be ZnO (Calcd 14.38%, observed 15.38%).

4. Conclusions

Three new coordination polymers with diverse architectures were designed via self-assembly of imi, 2,2'-bipy or 4,4'-bipy with Zn(II) and H_2oba , $[\text{Zn}(\text{imi})_2(\mu_2\text{-oba})]_n \cdot n\text{H}_2\text{O}$ (**1**), $[\text{Zn}(2,2'\text{-bipy})(\mu_2\text{-oba})(\text{H}_2\text{O})]_n \cdot n\text{H}_2\text{O}$ (**2**) and $[\text{Zn}_2(4,4'\text{-bipy})(\mu_2\text{-oba})_2(\text{H}_2\text{O})_2(\text{DMF})_2]_n \cdot n\text{DMF}$ (**3**). The V-shaped oba displays distinct coordination modes and N-donors with different positions and conformations also play an important role, enriching the diversity of structures. All solids are highly thermally stable.

Supplementary material

Crystallographic data for the structure reported in the article have been deposited with the Cambridge Crystallographic Data Center as supplementary publication no, CCDC 883,780–883,782 for **1–3**, respectively. Copies of the data can be obtained on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [Fax: +44–1223/150 336,033; E-mail: deposit@ccdc.cam.ac.uk].

Acknowledgments

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