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Structural diversity and properties of six coordination polymers derived from 1,2/1,3-phenylenedioxydiacetic acids and varied N-donor co-ligands

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ABSTRACT

Based on two positional isomers of phenylenedioxydiacetate ligands and different N-containing co-ligands, six novel coordination polymers, formulated as $\{[Zn(o-PDOA)(bpp)]\cdot 2H_2O\}_n$ (1), $\{[Cd(o-PDOA)(bix)]\cdot 3H_2O\}_n$ (2), $[Cd(m-PDOA)(bix)]_n$ (3), $[Cd(m-PDOA)(bib)]_n$ (4), $[Zn(m-PDOA)(bix)]_n$ (5), $\{[Zn(m-PDOA)(bib)]\cdot 2H_2O\}_n$ (6), $(o/m-H_2PDOA = 1,2/1,3$ -phenylenedioxydiacetic acid; bix = 1,4-bis(imidazol-ylmethyl)benzene; bib = 1,4-bis(1H-imidazol-1-yl)-butane; bpp = 1,3-bis(4-pyridyl)propane), have been synthesized under hydrothermal conditions. Compound 1 features a three-dimensional (3D) 2-fold interpenetrating **cds** topology. In 2, the (H₂O)₆ clusters interlink the 1D double $[Cd_2(o-PDOA)_2(bix)_2]_n$ chains into a 3D supramolecular network. Compound 3 is a (3,5)-connected 2D layer with (4².6)(4².6⁷.8) topology. Compound 4 exhibits a 3-fold interpenetrating **pcu** topological network, while, 5 is a 2D puckered meso-helical 4⁴-**sql** layer built by alternately arranged right-handed and left-handed helical chains. In contrast to 1–5, compound 6 possesses a 1D loop-like chain and, interestingly, rare D4 tetrameric (H₂O)₄ cluster extends the chains into a 3D supramolecular framework. Moreover, the thermal stabilities and luminescent properties of compounds 1–6 have also been investigated.

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1. Introduction

Crystal engineering to synthesize rationally coordination polymers (CPs) or metal-organic frameworks (MOFs) has evoked an immense amount interest as a result of the fascinating architectures and topologies as well as the potential applications in the areas of magnetism, luminescence, catalysis, gas storage/separation, sensing, ion exchange and optics [1-3]. Up to now, many effective synthetic approaches have been applied to construct desired and attractive CPs through the assembly of various metal centers and elaborately designed organic spacers. In this context, some excellent organic tectons, mainly including the rigid multicarboxylate ligands, such as 1,2/1,3/1,4-benzenedicarboxylic acid, 1,3, 5-benzene tricarboxylic acid. and 1.3.5-tri(4-carboxyphenyl) benzene, have been extensively used for fabricating functional crystalline materials [4–6]. However, the combination study on positional isomeric effects of organic linkers and the diversity of structure in assembly of CPs remains less explored yet, which is of great importance in understanding the relationship between their structure and intrinsic properties [7]. For these reasons, we have focused on utilizing positional isomeric benzenedicarboxylate ligands, such

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as 1,2/1,3/1,4-benzenedicarboxylic acid and 1,2/1,3/1,4-phenylenediacetate acid, to construct coordination polymers and to promote the investigation of the structure–property relationship. Many interesting frameworks with diverse topologies and desired properties have been observed [8]. To evaluate the influence of positional isomeric effect on the formation of the coordination frameworks, here we select a couple of positional isomeric dicarboxylate ligands, 1,2-phenylenedioxydiacetic acid (o-H₂PDOA) and 1,3-phenylenedioxydiacetic acid (m-H₂PDOA) as multidentate ligands based on the following reason: The –OCH₂– group makes these ligands more flexible and more coordination modes in comparison with the corresponding benzenedicarboxylate, whilst the existence of the benzene ring provides a rigid element.

On the other hand, polymeric d^{10} metal (Cu¹, Ag¹, Au¹, Zn^{II}, or Cd^{II}) complexes have extensive interests in recent years because they not only exhibit appealing structures but also possess photoluminescent properties [9]. A search of the Cambridge Structural Database (CSD version 5.34, may 2013) reveals only 13 coordination polymers constructed by a d¹⁰ metal and *o/m*-PDOA ligand have been reported hitherto (seven for *o*-PDOA, six for *m*-PDOA). Moreover, because of the strong coordination affinity and mediating geometric need of metal centers, it is effective to introduce auxiliary connectors in preparing metal carboxylate coordination polymers with novel topology and properties [10]. In view of the







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above experience, two isomeric phenylenedioxydiacetate ligands, 1,2/1,3-phenylenedioxydiacetic acid (Scheme 1), were used to assemble with Zn(II) or Cd(II) salt in this work, incorporating different secondary *N*-donor spacers. As a result, six coordination polymers with distinct topological motifs, that is, {[Zn(*o*-PDOA)(bpp]]·2H₂O}_{*n*} (1), {[Cd(*o*-PDOA)(bix)]·3H₂O}_{*n*} (2), [Cd(*m*-PDOA)(bix)]_{*n*} (3), [Cd(*m*-PDOA)(bib)]_{*n*} (4), [Zn(*m*-PDOA)(bix)]_{*n*} (5), {[Zn (*m*-PDOA)(bib)]·2H₂O}_{*n*} (6), are reported. Their syntheses, crystal structures, thermal stabilities, and photoluminescent properties have also been investigated.

2. Experimental

2.1. Materials and methods

All the chemicals were received as reagent grade and used without any further purification. FT-IR spectra were recorded as KBr pellets on a Thermo Electron NEXUS 670 FTIR spectrometer. Elemental analyses were performed on a Perkin-Elmer 2400 Series II analyzer. Thermogravimetric (TG) curves were recorded on a NETZSCH 449C thermal analyzer with a heating rate of 10 °C min⁻¹ under an air atmosphere. Power X-ray diffraction (PXRD) analyses were recorded on a Rigaku Ultima IV diffractometer (Cu K α radiation, λ = 1.5406 Å). Solid-state fluorescence spectra were recorded on a FLS920 fluorescence spectrophotometer at room temperature. The simulated powder patterns were calculated using Mercury 2.0. The purity and homogeneity of the bulk products were determined by comparison of the simulated and experimental X-ray powder diffraction patterns.

2.2. Synthesis of $\{[Zn(o-PDOA)(bpp)] \cdot 2H_2O\}_n$ (1)

A mixture of $Zn(CH_3COO)_2 \cdot 2H_2O$ (21.9 mg, 0.1 mmol), $o-H_2PDOA$ (22.6 mg, 0.1 mmol), bpp (19.8 mg, 0.1 mmol,) and NaOH (8.0 mg, 0.2 mmol) in H₂O (8 mL) was stirred for 30 min. The reaction mixture was placed in a 23 mL Teflon-lined stainless steel autoclave and was sealed and heated at 120 °C for 120 h. The autoclave was allowed to cool to room temperature for 48 h. Pale yellow crystals of compound **1** were obtained (30.9 mg). Yield: 59.0% (based on Zn). *Anal.* Calc. for $C_{23}H_{26}ZnN_2O_8$ ($M_r = 523.83$): C, 52.71; H, 5.00; N, 5.35. Found: C, 52.12; H, 4.86; N, 5.42%. IR (KBr pellet, cm⁻¹): 3458 w, 2933 w, 1622 s, 1509 m, 1432 m, 1329 w, 1250 s, 1210 s, 1137 s, 1105 m, 1021 m, 821 m, 752 m.

2.3. Synthesis of $\{[Cd(o-PDOA)(bix)] \cdot 3H_2O\}_n$ (2)

A mixture of $Cd(CH_3COO)_2 \cdot 2H_2O$ (26.7 mg, 0.1 mmol), *o*-H₂PDOA (22.6 mg, 0.1 mmol), bix (23.8 mg, 0.1 mmol) and NaOH (8.0 mg, 0.2 mmol) in H₂O (8 mL) was stirred for 30 min. The



Scheme 1. Schematic molecular structures of the ligands.

reaction mixture was placed in a 23 mL Teflon-lined stainless steel autoclave and was sealed and heated at 140 °C for 120 h. The autoclave was allowed to cool to room temperature for 48 h. Pale yellow crystals of compound **2** were obtained (35.0 mg). Yield: 55.7% (based on Cd). *Anal*. Calc. for C₂₄H₂₈CdN₄O₉ (M_r = 628.90): C, 45.83; H, 4.49; N, 8.91. Found: C, 45.42; H, 4.63; N, 8.76%. IR (KBr pellet, cm⁻¹): 3412 m, 2937 w, 1615 s, 1502 m, 1422 s, 1329 m, 1182 s, 1250 m, 1213 m, 1133 m, 1040 m, 924 w, 825 m, 755 s.

2.4. Synthesis of $[Cd(m-PDOA)(bix)]_n$ (3)

The same synthetic method as that of **2** was used except that *o*-H₂PDOA (22.6 mg, 0.1 mmol)was replaced by *m*-H₂PDOA(22.6 mg, 0.1 mmol). Colorless crystals were obtained (32.2 mg). Yield: 56% (based on Cd). *Anal.* Calc. for $C_{24}H_{22}CdN_4O_6$ (M_r = 574.86): C, 50.14; H, 3.86; N, 9.75. Found: C, 50.36; H, 3.58; N, 9.91%. IR (KBr pellet, cm⁻¹): 3139 s, 2894 s, 2362 m, 2336 m, 1618 s, 1592 s, 1509 m, 1416 s, 1329 m, 1293 w, 1183 s, 1080 s, 1060 m, 931 m, 825 m, 732 s, 688 m.

2.5. Synthesis of $[Cd(m-PDOA)(bib) \cdot H_2O]_n$ (4)

The same synthetic method as that of **3** was used except that bix (23.8 mg, 0.1 mmol) was replaced by bib (19.0 mg, 0.1 mmol,). Colorless crystals were obtained (31.1 mg). Yield: 57% (based on Cd). *Anal.* Calc. for $C_{20}H_{24}CdN_4O_7$ (M_r = 544.83): C, 44.09; H, 4.44; N, 10.28. Found: C, 44.31; H, 4.12; N, 10.46%. IR (KBr pellet, cm⁻¹): 3516 s, 3116 s, 2900 w, 2342 w, 1625 s, 1558 m, 1482 m, 1406 s, 1313 s, 1286 s, 1183 s, 1097 s, 964 m, 841 s, 778 m, 695 m.

2.6. Synthesis of $[Zn(m-PDOA)(bix)]_n$ (5)

The same synthetic method as that of **3** was used except that $Cd(CH_3COO)_2 \cdot 2H_2O(26.7 \text{ mg}, 0.1 \text{ mmol})$ was replaced by $Zn(CH_3COO)_2 \cdot 2H_2O$ (21.9 mg, 0.1 mmol). Pale yellow crystals were obtained (26.9 mg). Yield 51% (based on Zn). *Anal.* Calc. for $C_{24}H_{22}$. ZnN_4O_6 (M_r = 527.83): C, 54.61; H, 4.20; N, 10.61. Found: C, 54.87; H, 4.03; N, 10.76%. IR (KBr pellet, cm⁻¹): 3425 w, 3242 w, 1681 m, 1502 s, 1446 m, 1349 m, 1280 s, 1226 s, 1077 s, 924 m, 818 m, 768 s, 748 s, 675 s.

2.7. Synthesis of $\{[Zn(m-PDOA)(bib)] \cdot 2H_2O\}_n$ (6)

The same synthetic method as that of **5** was used except that bix (23.8 mg, 0.1 mmol) was replaced by bib (19.0 mg, 0.1 mmol). Pale yellow crystals were obtained (21.7 mg). Yield: 42% (based on Zn). *Anal.* Calc. for $C_{20}H_{26}ZnN_4O_8$ (M_r = 515.82): C, 46.57; H, 5.08; N, 10.86. Found: C, 46.91; H, 4.83; N, 11.04%. IR (KBr pellet, cm⁻¹): 3123 m, 2362 m, 1635 s, 1602 s, 1532 s, 1495 m, 1396 s, 1273 m, 1183 s, 1107 m, 1050 m, 957 w, 838 s, 758 m.

2.8. Crystal structure determination and refinement

Single crystal X-ray diffraction analyses of compounds **1–6** were carried out on a Rigaku XtaLAB mini diffractometer with graphite monochromated Mo K α radiation (λ = 0.71073 Å). The collected data were reduced using the program CRYSTALCLEAR [11] and an empirical absorption correction was applied. The structure was solved by direct methods and refined based on F^2 by the full matrix least-squares methods using SHELXTL [12,13]. All non-H atoms were refined anisotropically. The position of hydrogen atoms attached to carbon atoms were generated geometrically. The crystallographic data and structural refinementsfor complexes **1–6** are summarized in Table 1. Selected bond lengths and angles for **1–6** are listed in Table S1.

3. Results and discussion

3.1. Description of the crystal structures

3.1.1. {[Zn(o-PDOA)(bpp)]·2H₂O}_n (**1**)

Single-crystal X-ray diffraction analysis reveals that **1** crystallizes in the orthorhombic space group of *Pbcn* and the asymmetric unit of **1** contains one Zn(II) ion, one *o*-PDOA²⁻ ligand, one bpp ligand and two lattice water molecules (Fig. 1a). The Zn(II) ion is five-coordinated by three carboxylate oxygen atoms from two *o*-PDOA²⁻ ligands (Zn1–O1 = 2.151(4), Zn1–O2 = 2.218(4), Zn1–O6^{#1} = 2.009(4) Å, #1: x + 1/2, -y + 1/2, -z + 1) and two nitrogen atoms from two bpp ligands (Zn1–N1 = 2.065(4), Zn1–N2^{#2} = 2.047(4) Å, #2: x - 1/2, y - 1/2, -z + 1/2) in a distorted square pyramid coordination geometry. Each *o*-PDOA²⁻ ligand act as μ_2 -bridging mode with one carboxylate group in a μ_1 - $\eta^1:\eta^1$ chelating mode and the other in a μ_1 - $\eta^1:\eta^0$ monodentate mode(mode **II** in Scheme 2). The Zn(II) ions are bridged by *o*-PDOA²⁻ ligands to generate zigzag neutral [Zn(*o*-PDOA)]_n chains with the Zn···Zn distance of 8.850(9) Å. The chains are linked by TT conformational bpp ligands which adopt two different orientations (N1–Zn1–N2, 109.0(1)°), thus extending the chains into a 3D 6⁵.8-**cds** framework (Fig. 1b). Furthermore, the cds-topological architecture of **1** produces large channels that have an opening of about 13.0 × 16.3 Å² along the crystallographic *a* axis (Fig. 1b). So, a pair of **cds**-nets is interlocked with each other, leading to the formation of the 2-fold interpenetrated 3D → 3D architecture (Fig. 1c, d). Although a number of **cds** frameworks have been reported, there are only a few compounds that exhibit interpenetration [14]. The 2-fold interpenetration in the crystal effectively reduces the void

Table 1

Crystallographic data and structure refinement details for 1-6.

Compound	1	2	3	4	5	6
Formula	C23H26ZnN2O8	$C_{24}H_{28}CdN_4O_9$	$C_{24}H_{22}CdN_4O_6$	C20H24CdN4O7	$C_{24}H_{22}ZnN_4O_6$	C20H26ZnN4O8
Formula weight	523.83	628.90	574.86	544.83	527.83	515.82
Crystal system	orthorhombic	triclinic	triclinic	triclinic	monoclinic	triclinic
a (Å)	14.094(2)	9.605(2)	10.034(8)	9.601(9)	20.728(4)	9.908(2)
b (Å)	17.093(8)	12.262(3)	10.942(5)	10.713(8)	13.207(2)	10.501(1)
<i>c</i> (Å)	20.192(9)	12.708(3)	12.101(7)	11.565(7)	16.843(3)	12.880(3)
α (°)	90.00	66.916(3)	67.629(1)	110.919(2)	90.00	105.483(8)
β(°)	90.00	72.173(3)	78.023(1)	97.986(2)	106.30(3)	93.881(8)
γ (°)	90.00	84.090(4)	79.469(1)	92.316(2)	90.00	113.079(1)
$V(Å^3)$	4864.9(7)	1310.5(5)	1194.14(1)	1095.37(1)	4425.5(1)	1165.4(4)
Ζ	8	2	2	2	8	2
$D_{\rm calc}~({\rm g~cm^{-3}})$	1.430	1.594	1.599	1.652	1.584	1.470
F(000)	2176	640	580	552	2176	536
Data/restraints/parameters	4270/21/319	4584/9/361	4143/144/316	5261/121/289	5401/0/317	4090/0/299
R _{int}	0.0556	0.0113	0.1142	0.0402	0.0213	0.1599
GOF	1.033	1.050	1.093	1.019	1.011	1.046
$R_1^{a} (I > 2\sigma(I))$	0.0677	0.0228	0.0682	0.0518	0.0331	0.0597
wR_2^b $(I > 2\sigma(I))$	0.1918	0.0617	0.2110	0.0783	0.0814	0.1531
R_1 (all data)	0.0910	0.0249	0.0724	0.0658	0.0547	0.0648
wR_2 (all data)	0.2073	0.0633	0.2181	0.0827	0.0902	0.1605
$\Delta ho_{ m maximum,minimum}$ (e Å ⁻³)	1.470/-0.861	0.364/-0.384	2.244/-1.786	0.903/-0.559	0.328/-0.498	1.265 / -0.641

^a $R_1 = \Sigma ||F_0|| - |F_c|/\Sigma |F_0|.$

 $wR_2 = \{\Sigma[w(F_0^2 - F_c^2)^2] / \Sigma[w F_0^2]^2\}^{1/2}.$



Fig. 1. (a) The coordination environment of Zn(II) ion in 1 (symmetry codes: A: -1/2 + x, -1/2 + y, 1/2 - z; B: 1/2 + x, 1/2 - y, 1 - z). (b) The view of 3D **cds** network. (c) The 3D packing of 2-fold **cds** network. (d) Schematic representation of 2-fold **cds** topology.



Scheme 2. Diverse coordination modes of *o*/*m*-PDOA in complexes 1–6 and previously reported polymers.

space, and it still shows a porous structure. After removal of the lattice water molecules, the effective free void space of **1** is calculated by PLATON program [15] as being 15.6% of the crystal volume (757.1 Å³ out of the 4864.9 Å³ unit cell volume). In addition, intramolecular O–H···O hydrogen-bonding interactions (Table S2) exist among the lattice water molecules and carboxylate oxygen atoms, which further stabilized the 3D structure.

3.1.2. {[Cd(o-PDOA)(bix)]·3H₂O}_n (**2**)

Single-crystal X-ray diffraction analysis reveals that **2** crystallizes in the triclinic space group of *P*1. The structure of **2** exhibits a double-stranded chain and each asymmetric unit of 2 contains one Cd(II) ion, one o-PDOA²⁻ ligand, one bix ligand and three lattice water molecules. As shown in Fig. 2a, Cd(II) ion displays distorted pentagonal bipyramidal coordination geometry, defined by three carboxyl oxygen atoms and two ether oxygen atoms from two different o-PDOA²⁻ ligands (Cd(1)-O(1) = 20291)(1), Cd(1)-O(3) = 2.637(3), Cd(1)-O(4) = 2.647(2), Cd(1)-O(5) = 2.647(2)2.367(0), Cd(1)–O(5)^{#1} = 2.331(4) Å, #1: -x + 2, -y + 1, -z), as well as two nitrogen atoms from two bix ligands (Cd(1)–N(1) = 2.221(9), Cd(1)– $N(4)^{#2} = 2.235(5)$ Å, #2: x + 1, y - 1, z). One carboxylate group of PDOA²⁻ ligand acts as bridging ligand, which links the two Cd(II) ions in μ_2 - η^2 : η^0 mode, the other carboxylate group adopts μ_1 - η^1 : η^0 mode, resulting the pentadentate coordination mode (mode **Im** in Scheme 2), with the addition of two coordinated ether oxygen atoms. In the structure, two Cd(II) ions are connected by a pair of pentadentate o-PDOA²⁻ ligands to form a binuclear unit $(Cd \cdots Cd = 3.848(9) \text{ Å})$. Then such binuclear blocks are linked through trans conformational bix ligands in two different directions giving rise to a unique 1D infinite double [Cd₂(o-PDOA)₂(bix)₂]_n chain (Fig. 2b). If viewed along the *b*-axis, these double chains have interesting $\pi \cdots \pi$ packing interactions of adjacent aromatic rings between the *o*-PDOA²⁻ molecules (centroid-to-centroid distance = 3.976(2) Å), resulting a 2D supramolecular network as shown in Fig. 2c. It is worth noting that an interesting hexameric water cluster (Fig. 2e) self-assembled by means of hydrogen bonding interactions (Table S2) between lattice water molecules is observed in the solid structure of **2**. Each hexameric water cluster consists of a cyclic water tetramer and two dangling water monomers. Within the cluster, the four water molecules are fully coplanar in the plane and each water monomer acts as both a single hydrogen bond donor and acceptor. The remaining hydrogen atom of O1WA is below/above of the ring, while the remaining hydrogen atom of O1WC is above/below the ring. In addition, the hanged water molecules also form hydrogen bonds with carboxylate group of PDOA²⁻ ligand, resulting in a 3D supramolecular network (Fig. 2d).

3.1.3. $[Cd(m-PDOA)(bix)]_n$ (3)

Compound **3** crystallized in triclinic space group of $P\overline{1}$. As shown in Fig. 3a, there are one crystallography independent Cd(II) ion, one *m*-PDOA^{2–} ligand and one bix ligand. Each Cd(II) center adopts distorted octahedral coordination geometry completed by four O atoms from two *m*-PDOA^{2–} ligands and two N atoms from two bix liangs. Each *m*-PDOA^{2–} anion acts as a μ_3 -bridge (mode **III** in Scheme 2) linking three Cd(II) ions, in which one carboxylate group adopts a $\mu_2-\eta^1:\eta^1$ bridging mode to connect two Cd(II) ions, while the other adopts a $\mu_1-\eta^1:\eta^1$ mode to chelate one Cd(II) ion. The adjacent Cd(II) ions are bridged by *m*-PDOA^{2–} ligands to form a 1D [Cd₂(*m*-PDOA)₂]_{*n*} ribbon chain. The infinite chain comprises 8and 24-membered rings built up by Cd(II) ion and *m*-PDOA^{2–} anion. The 8-membered ring is formed by two symmetry related bridging carboxylate groups with a Cd…Cd distance of 4.710(3) Å. Such 1D chains are further interlinked by *trans* confor-



Fig. 2. (a) The coordination environment of Cd(II) ion in **2** (symmetry codes: A: 2 - x, 1 - y, -z; B: 1 + x, -1 + y, z); (b) The view of 1D infinite double chain like structure. (c) The 2D supramolecular packing sheet constructed by $\pi \cdots \pi$ packing interactions. (d) The view of H-bonded 3D network. (e) The enlarged view of the hexameric (H₂O)₆ cluster formed by a planar water tetramer and two dangling water monomers.



Fig. 3. (a) The coordination environment of Cd(II) ion in 3 (symmetry codes: A: 1 - x, -y, 1 - z; B: 1 + x, y, -1 + z; C: 1 + x, -1 + y, z). (b) The view of the 2D layer. (c) Schematic representation of (3,5)-connected (4².6)(4².6⁷.8) topology. (d) The view of the 3D supramolecular network linked by hydrogen bonding and $\pi \cdots \pi$ packing interactions.

mational bix ligands with a dihedral angle between imidazole and phenyl of $87.45(7)^{\circ}$ and $79.31(1)^{\circ}$ to form a 2D layer (Fig. 3b). Analysis of the network topology of **3** reveals that each Cd(II) center acts as a 5-connected node to connect three *m*-PDOA^{2–} ligands and two bix spacers. And the *m*-PDOA^{2–} ligands serve as the 3-con-

nected nodes. Thus, a (3,5)-connected 2D network with the point symbol of $(4^2.6)(4^2.6^7.8)$ is constituted (Fig. 3c). The 2D layers are further linked by hydrogen bonding between the C atom and the carboxylate O atom (C7–H7A···O5^{#1}, #1: -x, 1 - y, 2 - z), as well as the $\pi \cdot \cdot \pi$ packing interactions of adjacent imidazole rings be-

tween the m-PDOA²⁻ molecules (centroid-to-centroid distance = 3.898 (6) Å), resulting in a 3D supramolecular network (Fig. 3d).

3.1.4. {[Cd(m-PDOA)(bib)]·H₂O}_n (**4**)

X-ray single-crystal diffraction analysis reveals that 4 is a 3-fold interpenetrated **pcu** network and crystallizes in the triclinic space group of $P\overline{1}$. The asymmetric unit contains one Cd(II) ion, one m-PDOA²⁻ ligand and a pair of half bbi ligand as well as one lattice water molecule. The structure contains a centrosymmetric binuclear Cd(II) unit (Fig. 4a) built through two bridging carboxylates (Cd1-O6 2.240(1) Å), while the other four coordination sites of each metal ion are occupied by two chelating carboxylate O atoms and two imidazole N atoms forming a distorted octahedral geometry (Cd1-O1 2.290(5), Cd1-O2 2.598(2), Cd1-N1 2.281(3), Cd1-N3 2.231(2) Å). Each *m*-PDOA²⁻ anion acts as a μ_3 -bridge linking three Cd(II) ions (mode IIm in Scheme 2), in which one carboxylate group adopts a μ_2 - η^2 : η^0 bridging mode to connect two Cd(II) ions, while the other adopts a μ_1 - η^1 : η^1 mode to chelate one Cd(II) ion. The m-PDOA²⁻ ligands link the binuclear Cd(II) units to form a 1D double chain with the $[Zn_2(m-PDOA)_2]_n$ ring arranging alternately, which is then connected by the TTT conformational bbi ligands into a loop-like ladder 2D network along the (111) plan (Fig. 4b). Further, these 2D layers are pillared by the GTG (G = gauche, T = trans) conformational bbi spacers to result in an interesting 3D pillar-layered framework with 1D rectangular channels with dimensions of $14.597(6) \times 13.098(8)$ Å². Because of the spacious nature of the single network, the potential voids are filled via mutual interpenetration of an identical 3D framework generating a 3-fold interpenetrating architecture (Fig. 4c). Topologically, if we consider the dimeric Cd(II)-unit as six-connected node, while m-PDOA²⁻ and bbi ligands serve as linkers. Thus, the whole framework is a 3-fold interpenetrating **pcu** network (Fig. 4d).

3.1.5. $[Zn(m-PDOA)(bix)]_n$ (5)

The single-crystal X-ray diffraction study performed on compound 5 reveals that it is a 2D framework, crystallizing in the monoclinic space group C2/c. As shown in Fig. 5a, the asymmetric unit consists of two crystallographically independent Zn(II) ions, one m-PDOA²⁻ ligand, a pair of half bix liand. Each Zn(II) center is tetra-coordinated by two carboxyl O atoms from two m-PDOA²⁻ ligands and two imidazolyl N atoms from two bix ligands, showing a distorted tetrahedral coordination geometry. The two carboxylate groups of *m*-PDOA²⁻ ligand all exhibit μ_1 - η^1 : η^0 monodentate mode (mode IIe in Scheme 2). The Zn(II) ions are linked by μ_1 -bridging *m*-PDOA²⁻ ligands to form the alternate left- and right-handed [Zn(m-PDOA)]_n helical chains running along the crystallographic a axis(Fig. 5b) with a pitch of 20.728(4) Å. Righthanded and left-handed helical chains are alternately arranged and interlinked by *trans*-bix ligands, resulting a 2D puckered meso-helical (4,4) layer (Fig. 5c) with grid dimensions 10.3×14.9 Å² (based on the separation of the metal ions). Furthermore, the C–H···O and C–H···Cg weak bonding interactions which is ranging from 3.176 (6) to 3.306(2) Å not only bring further stability for the structure but also link the 2D layer structure to form a 3D supramolecular architecture (Fig. 5d, S1).

3.1.6. { $[Zn(m-PDOA)(bib)] \cdot 2H_2O_n(\mathbf{6})$

Single-crystal X-ray diffraction analysis reveals that complex **6** crystallizes in the triclinic $P\overline{1}$ space group. As shown in Fig. 6a, each Zn(II) center displays a distorted tetrahedral coordination geometry, where two oxygen atoms (O1, O5A) are part of two separate m-PDOA²⁻ ligands and two nitrogen atoms (N1, N4B) belong to two different bib ligands. The two carboxylate groups of m-PDOA²⁻ ligand all exhibit μ_1 - η^1 : η^0 monodentate mode (mode **IIe** in Scheme 2), whereas the GTT conformational bib ligand with its terminal nitrogen atom coordinate two Zn(II) centers, and the dihedral angle between two imidazolyl ring is 64.62°. In this way,







Fig. 5. (a) The coordination environment of Zn(II) ion in **5** (symmetry codes: A: 1 - x, y, 1/2 - z; B: -x, y, 1/2 - z). (b) The view of 1D infinite right-handed and left-handed helical $[Zn(m-PDOA)]_n$ chains along the crystallographic *a* axis. (c) The view of 2D puckered meso-helical (4, 4) layer constructed by helical chains and interlinked bix ligands. (d) View of 3D crystal packing of **5**.



Fig. 6. (a) Coordination environment of Zn(II) ion in **6** (symmetry codes: A: -x, 1 - y, -1 - z; B: -x, -y, -1 - z). (b) The view of 1D loop-like chain formed by Zn(II) ions and organic ligands. (c), (d) The 3D supramolecular framework constructed by 1D chains and (H₂O)₄ water clusters viewed along the *b* (or *a*) axis. (e) View of the tetramer water cluster which links the four adjacent chains; (f) The enlarged view of the tetramer water cluster.

two bib ligands link two Zn(II) centers into a -Zn-bib-Zn-bib-22-member ring in head-to-tail mode with the Zn \cdots Zn distance being 9.362(9) Å. The bis-monodentate *m*-PDOA^{2–} ligands further extend the metal rings into a 1D loop-like chain (Fig. 6b).

Most strikingly, the two symmetric independent lattice water molecules (O7, and O8) are assembled into a curved tetrameric cluster (H₂O)₄ notated as D4 according to Infantes' classification [16] by hydrogen bonding interactions with the O···O distances of 2.706 (8) Å (O7···O8) and 2.756 (6) Å (O8···O8A) and an O···O··O angle of 122.94° (O7···O8···O8A). As we know, such an arrangement has scarcely observed so far [17], although cyclic (H₂O)₄ consisting of two or four independent water molecules have been reported [18]. In this structure, the guest water cluster, (H₂O)₄, just like a "glue" agglutinates and supports the host 1D loop-like chains to form a 3D supramolecular framework (Fig. 6c, d) by abundant hydrogen bonding interactions involving carboxylic oxygen atoms and crystallization water molecules (Fig. 6e, f).

3.2. The diverse coordination modes of o/m-PDOA

The coordination chemistry of H₂PDOA is interesting, since its two carboxylate groups can be partially or fully deprotonated to generate HPDOA⁻ and PDOA²⁻ anions under different synthesis conditions. Therefore, it can coordinate with metal ions in multiple ways to form a series of coordination polymers with interesting topologies and properties. Furthermore, the flexible -O-CH₂-COO group can twist to meet different coordination modes. As shown in Scheme 2, according to the search of the Cambridge Structural Database (CSD version 5.34, may 2013), so far 26 kinds of coordination mode of o/m-PDOA ligands were observed in the previously reported ones and complexes 1–6 [19,20]. In 1, the o-PDOA ligand takes the μ_2 -bridging mode with one carboxylate group in a $\mu_1 - \eta^1 : \eta^1$ chelating mode and the other in a $\mu_1 - \eta^1 : \eta^0$ monodentate mode (see mode II in Scheme 2). In 2, the *m*-PDOA ligand displays a first reported pentadentate mode (see mode Im in Scheme 2). In 3, the o-PDOA ligand acts the μ_3 -bridging mode with one carboxylate group in a μ_1 - η^1 : η^1 chelating mode and the other in a μ_2 - η^1 : η^1 bridging mode (see mode III in Scheme 2). In 4, the *m*-PDOA ligand acts as μ_3 -bridging mode with one carboxylate group in a μ_1 - η^1 : η^1 chelating mode and the other in a μ_2 - η^2 : η^0 bridging mode (see mode **IIm** in Scheme 2). In **5** and **6**, the *m*-PDOA ligands fearture μ_2 -bridging modes with two carboxylate groups in μ_1 - η^1 : η^0 monodentate mode (see mode IIe in Scheme 2). In comparison with the reported compounds, as we know, the coordination modes Im, III and **IIm** are first reported in this presentation.

3.3. Structural diversity of 1–6

It is noteworthy that a variety of framework structures can be achieved on the basis of the choice of the phenyl dicarboxylate isomers with differently oriented carboxyl groups and N-containing auxiliary bridging ligands. The phenomenon of structural diversification in 1-6, may arise from three different sources: (i) the positional isomeric effect of the PDOA ligands. The phenylenedioxydiacetate isomers with different orientations, torsions, and binding modes of the carboxylate groups can adopt the chelating and bridging modes. From PDOA-containing CPs 1-6, PDOA ligand shows five types of coordination modes: mode II for 1, mode Im for 2, mode III for 3, mode IIm for 4, mode IIe for 5 and 6, resulting in three different dimensional frameworks (two for 1D, two for 2D, and two for 3D, Table 2); (ii) the N-containing co-ligands with different flexibility were employed to investigate their influence on the structure of the polymers, which will influence the final coordination architectures. Compare 3 and 4: when a semi-rigid ligand, bix was chosen as an auxiliary ligand, and is found to act as a pillar connector to

Table 2

Summary about the coordinated	l geometry of metal	centers in 1–6 .
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	Formula	Dimension	Coordination atoms	Geometry of metal
1	$\begin{array}{c} C_{23}H_{26}ZnN_2O_8\\ C_{24}H_{28}CdN_4O_9\\ C_{24}H_{22}CdN_4O_6\\ C_{20}H_{24}CdN_4O_7\\ C_{24}H_{22}ZnN_4O_6\\ C_{20}H_{26}ZnN_4O_8 \end{array}$	3D	N2O3	square pyramid
2		1D	N2O5	pentagonal bipyramid
3		2D	N2O4	octahedron
4		3D	N2O4	octahedron
5		2D	N2O2	tetrahedron
6		1D	N2O2	tetrahedron

link 1D double $[Cd_2(m-PDOA)_2]_n$ chains into a 2D layer with $(4^2.6)(4^2.6^7.8)$ topology. In **4**, when a flexible spacer (bib) was taken into the reaction, a 3D threefold interpenetrating pcu network was obtained. In 5 and 6, semi-rigid bix and flexible bib was used in the reactions, respectively. 5 is a puckered mesohelical (4,4) layer network wihle 6 exhibits a 1D loop-like chain structure; (iii) the versatile coordinated geometries of Zn(II)/ Cd(II) centers maybe benefit the structural diversification of the resultant extended networks of coordination polymers (Table 1). For example, with regard to the *m*-PDOA-bib- bridged CPs **4** and **6** in this series of crystalline materials, along with the variation of metal centers (from Cd(II) to Zn(II) ion), their network arrays are changed from 3D 3-fold interpenetrating **pcu** topology to1D loop-like chain motif. The results mentioned above indicate that the backbone of phenylenedioxydiacetate ligands, the different flexibility of the N-containing auxiliary bridging ligands as well as the coordination geometry of the metal centers play a key role in governing the ultimate crystalline architectures.

3.4. XRPD and TG results

To check the purity and homogeneity of the bulk products of compounds **1–6**, the as-synthesized samples were measured by X-ray powder diffraction (XRPD) at room temperature. As shown in Figs. S2–S7 (ESI), the peak positions of the experimental patterns are in good agreement with the simulated patterns generated from single-crystal diffraction data, which clearly indicate the good purity and homogeneity of the compounds.

To investigate the thermal stability of these compounds, the thermogravimetric analyses were carried out on under an air atmosphere. As shown in Fig. S8 (ESI), compound 1 shows the first mass loss of 6.47% below 135 °C due to the release of the two guest water molecules (calcd 6.87%); the further weight loss of 78.41% between 230 and 450 $^\circ C$ is attributed to the loss of organic ligands(calcd 80.63%), and the residual weight at ca. 15.12% (calcd for ZnO, 15.54%). For 2, the first mass loss of 9.31% below 175 °C due to the release of the three guest water molecules (calcd 8.59%), the residual weight at ca. 21.32% above 550 °C (calcd for CdO, 20.41%). For **3**, the framework is stable up to 290 °C, and then a sharp weight loss (75.41%) from 290 to 700 °C corresponding to the decomposition of the organic ligands, and the residual weight at ca. 22.58% (calcd for CdO, 22.33%). For 4, the first mass loss of 3.51% below 110 °C due to the release of the one guest water molecule (calcd 3.30%), and the residual weight at ca. 23.71% (calcd for CdO, 23.56%). For 5, the framework is stable up to 240 °C, and the residual weight at ca. 15.15% (calcd for ZnO, 15.42%)). For 6, the first mass loss of 7.36% below 120 °C due to the release of the two guest water molecules (calcd 6.98%); the second step is from 250 to 600 °C, where the organic ligands began to decompose with the residual weight of 17.54% (calcd for ZnO, 15.72).

3.5. Photoluminescent properties

The emission spectra of **1–6** were examined in the solid state at room temperature and shown in Fig. 7. According to the previous



Fig. 7. Solid-state emission spectrum of 1–6 at room temperature.

 Table 3

 Luminescent properties of free ligands and 1–6 in the solid state.

Compound	$\lambda_{\rm ex}/\lambda_{\rm em}$ (nm)	Compound	$\lambda_{\rm ex}/\lambda_{\rm em}$ (nm)
o-H ₂ PDOA	284/568	2	375/461
$m-H_2PDOA$	350/394	3	315/429
bpp	373/508	4	315/441
bix	379/451	5	388/452
bib	278/320	6	372/431
1	440/503		

study, it is found that the emission bands for the free ligand bpp. bix, bib, o-H₂PDOA and *m*-H₂PDOA display photoluminescence with emission maxima at 508 nm [21], 451 nm [22], 320 nm [22], 568 nm [23] and 394 nm [20b] respectively. The emission bands of these free organic ligands can be assigned to the $\pi^* \rightarrow n$ or $\pi^* \rightarrow \pi$ transition. The emission spectra show maximum emission peaks at 503 nm (λ_{ex} = 440 nm) for **1**, 461 nm (λ_{ex} = 375 nm) for **2**, 429 nm (λ_{ex} = 315 nm) for **3**, 441 nm (λ_{ex} = 315 nm) for **4**, 452 nm (λ_{ex} = 388 nm) for **5**, 431 nm (λ_{ex} = 372 nm) for **6**, respectively (Table 3). The emissions are neither metal-to-ligand charge transfer (MLCT) nor ligand-tometal charge transfer (LMCT) in nature since Zn(II)/Cd(II) ion is difficult to oxidize or reduce due to its d¹⁰ configuration [24]. Thus, they may be assigned to a mixture characteristics of intraligand ($\pi^* \rightarrow n$ or $\pi^* \rightarrow \pi$ transition) and ligand-to-ligand charge transition (LLCT), as reported for other Zn(II)/Cd(II) CPs constructed from mixed N-donor and O-donor ligands [25]. The enhancement of luminescence in d¹⁰ complexes may be attributed to ligand chelation to the metal center which effectively increases the rigidity of the ligand and reduces the loss of energy by radiationless decay [26]. The difference of the emission behaviors for 1-6 probably derive from the differences in the rigidity of solid-state crystal packing.

4. Conclusions

In summary, six coordination polymers have been isolated under hydrothermal conditions by the employment of isomeric H₂PDOA and different N-containing ligands. Compounds **1–6** display appealing structural features from 1D double or loop-like chains to 2D puckered meso-helical layer to 3D **cds/pcu** topological frameworks. Three new coordination modes of PDOA ligands were first observed in coordination polymers and reported. Of particular interest, **2** and **6** feature interesting (H₂O)₆ and D4 tetrameric $(H_2O)_4$ water clusters. The effects of the positions of the carboxylate groups, the flexibility of the N-containing co-ligands as well as the coordination geometry of the metal centers are discussed in detail. Many more systematic studies for the design and construction of such crystalline materials with isomeric H₂PDOA and other N-containing building blocks are underway in our group.

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Appendix A. Supplementary material

CCDC 932838–932843 contain the supplementary crystallographic data for **1–6**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc. cam.ac.uk/data_request/cif. X-ray crystallographic files in CIF format for compounds **1–6**, selected bond lengths and angles, hydrogen bond distances and angles for **1** and **2**, PXRD, TG curves for **1–6**, respectively. Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/ j.ica.2013.12.027.

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