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1D zigzag chain and 0D monomer Cd(II)/Zn(II) compounds based on flexible phenylenediacetic ligand: Synthesis, crystal structures and fluorescent properties

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A B S T R A C T

Three novel Cd(II)/Zn(II) compounds, $[Cd_2(poda)_2(phen)_3(H_2O)]_n \cdot nEtOH \cdot 3nH_2O$**(1)**, $[Zn(poda)_2(bpy)(H_2O)]_n$**(2)** and $[Zn(Hpoda)_2(bpy)]$**(3)** (H_2poda = 1,2-phenylenediacetic acid, phen = 1,10-phenanthroline, bpy = 2,2′-bipyridyl), have been synthesized and characterized by IR, TG, fluorescent spectrum and single-crystal X-ray diffraction techniques. In 1, $poda^{2-}$ anions link the adjacent Cd(II) centers to generate a 1D zigzag chain. Furthermore, an unprecedented four-footed “8-shaped” mixed water–ethanol $(H_2O)_6(C_2H_5OH)_2$ cluster connects four double chains based on 1D zigzag chain into 3D supramolecular architecture. By bis(chelate–monodentate) fashion of $poda^{2-}$ ligand, compound **2** exhibits 1D zigzag chains, which forming a dense zipper-like 2D structure *via* strong π–π stacking interactions. Differed from **1** and **2**, compound **3** has a mononuclear motif, and displays a 3D 6-connected α-Po net hydrogen-bonded topology. The structure-related solid-state fluorescence spectra of compounds **1** and **2** have been determined.

1. Introduction

Construction of metal-involved supramolecular architectures based on crystal engineering are currently of interest in the field of supramolecular chemistry and material science [1,2]. The increasing interest in this field is justified not only by their potential application in microelectronics [3], non-linear optics [4], porous materials [5], catalysis, and luminescence [6], but also by their intriguing structural diversities of the architectures [7]. A successful approach to build these supramolecular frameworks is to select suitable multi-dentate ligands as building blocks. Among them, multi-carboxylate ligands have been demonstrated as a top-rank candidates, owing to their versatile coordination modes [8], for the formation of 1D, 2D or 3D frameworks and, construction of unique interwoven extended structural motifs, such as polycatenanes, polyrotaxanes, double helices, triple helices, borromean rings, molecular braids, and other more unusual species [9]. Phenylenediacetic acid is a multifunctional ligand with two carboxyl groups, which possesses three isomers of 1,2-, 1,3-, and 1,4-phenylenediacetitic acids. As symmetrical and flexible dicarboxylate ligand, they could display versatile coordinated modes, and provide potential identified sites of hydrogen bonds and π–π stacking interactions to expand in high dimension frameworks. To our knowledge, two of them, 1,3- and 1,4-phenylenediacetitic acids, have been widely investigated for the larger distance of their two carboxylate groups, and are apt to porous metal compounds [10]. However, 1,2-phenylenediacetitic acid is rarely reported [11], which inspirits our interest in it. Recently, we take interest in building supramolecular structures using *N*-heterocyclic or aromatic carboxyl ligands, and obtain the expected results.

In view of above points, we tried to prepare Cd(II)/Zn(II) compounds with 1,2-phenylenediacetitic acid for series researches. Fortunately, we obtained two 1D compounds (**1** and **2)** and 0D mononuclear compound **3** by using 1,2-phenylenediacetitic acid as bridged ligand and NN chelated ligands as terminal ligands. In these compounds, 1,2-phenylenediacetitic acid exhibits three kinds of coordination fashions, and constructs 1D zigzag chain or 0D monomer structures, respectively. Interestingly, compound **1** shows a 3D supramolecular architecture with the inclusion of an unusual four-footed “8-shaped” mixed water–ethanol $(H_2O)_6(C_2H_5OH)_2$ clusters unreported in Ref. [12].

2. Experimental

2.1. Materials and measurements

All manipulations were carried out in air. All reagents were commercially available and were used without further purification. Elemental analyses (C, H, N) were performed on a Perkin-Elmer 2400 CHN elemental analyzer. TG analyses were recorded with a NETZSCH STA 449C microanalyzer under nitrogen atmosphere at

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a heating rate of 10 °C min^{-1}. Fluorescence spectra were performed on a Hitachi F-4500 fluorescence spectrophotometer at room temperature.

2.2. Synthesis of $[Cd_2(poda)_2(phen)_3(H_2O)]_n \cdot nEtOH \cdot 3nH_2O$ (**1**)

A solution of H_2poda (0.0194 g, 0.1 mmol) and phen (0.0198 g, 0.2 mmol) in EtOH/H_2O (40 mL, 1:1) adjusted to pH 7 by addition of aqueous NaOH solution (1.0 mmol L^{-1}), was added to the solution of $Cd(NO_3)_2 \cdot 4H_2O$ (0.0308 g, 0.1 mmol) in H_2O (20 mL). The reaction mixture was stirred for ca. 10 min to give a colorless solution and filtered. After the mixture was slowly cooled to room temperature, colorless block crystals of **1** were yielded. Elemental analysis calcd. for $C_{58}H_{54}Cd_2N_6O_{13}$ (%): C, 54.94; H, 4.29; N, 6.63. Found (%): C, 54.02; H, 4.12; N, 6.05.

2.3. Synthesis of $[Zn(poda)_2(bpy)(H_2O)]_n$ (**2**)

Compound **2** was synthesized from the reaction mixture of H_2poda (0.0194 g, 0.1 mmol) were dissolved in 2 ml of MeOH, then the solution was added dropwise with 6 ml H_2O of $Zn(NO_3)_2$ (0.0308 g, 0.1 mmol) and 2 ml MeOH of bpy (0.0156 g,0.1 mmol). The solution was adjusted to pH 8–9 by addition of 1.0 mmol L^{-1} NaOH solution, then stirred, filtered, and standing in air at room temperature for 20 days, some colorless block crystals of compound **2** were obtained. Elemental analysis calcd. for $C_{20}H_{18}N_2O_5Zn$ (%): C, 55.64; H, 4.20; N, 6.49. Found (%): C, 54.93; H, 4.01; N, 5.69.

2.4. Synthesis of $[Zn(Hpoda)_2(bpy)]$ (**3**)

The synthesis procedure was similar to that described above compound **2** except using pH 7 instead of pH 8–9, and colorless block crystals of **3** were yielded. Elemental analysis calcd. for $C_{30}H_{26}N_2O_8Zn$ (%): C, 59.27; H, 4.31; N, 4.61. Found (%): C, 58.76; H, 4.12; N, 3.95.

2.5. Determination of crystal structure

Single-crystal X-ray data were collected on a Bruker SMART APEX II CCD diffractometer equipped with graphite monochromatized Mo Kα ($\lambda = 0.071073$ nm) by Crystal clear software. Empirical absorption corrections were applied in the case. All calculations were conducted using SHELXS-97 and SHELXL-97 programs [13,14], while the structures were solved by the direct methods. All the non-hydrogen atoms were located from the trial structure and then refined anisotropically with SHELXTL via full-matrix least-square procedure [15]. Hydrogen atoms were located from difference Fourier maps. The crystallographic data of the compounds are summarized in Table 1.

3. Results and discussion

3.1. Crystal structures of compounds **1–3**

X-ray single-crystal diffraction analysis reveals compound **1** has 1D zigzag structure based on dinuclear metallic units containing solvent clusters. In the asymmetric unit of **1**, there are two Cd^{II} ions, two $poda^{2-}$ ligand, three phen groups, one water molecule, as well as three lattice water molecules, and one ethanol molecule (Fig. 1). Cd1 ion is located in a six-coordinated anti-triangular-prism geometry with four nitrogen atoms from two chelated phen ligands, two oxygen atoms from two $poda^{2-}$ groups (plane 1: N4, N2, O5 and plane 2: O1, N1, N3). Different from Cd1, Cd2 ion is seven-coordinated to four oxygen atoms from two $poda^{2-}$ groups, two nitrogen atoms from one phen, and one water molecule into a distorted one-cap triangular-prism geometry. The Cd–O bonds are in the range of 2.208–2.585 Å, Cd–N bonds range from 2.374(4) to 2.468(4) Å, which drop into the normal scope of Cd–N and Cd–O bond lengths [16]. The $poda^{2-}$ ligand adopts chelate–bidentate coordination fashion to connect the adjacent Cd^{II} atoms into 1D zigzag chain. Interestingly, the 1D chains are interlinked to form a 1D double chain via strong intermolecular hydrogen bonds between the coordinated water molecules on one chain and the $poda^{2-}$ acceptors on the other chain ($O(9)–H \cdots O(3) = 2.730$ Å, 178.49°).

The most remarkable feature of **1** is the existence of unreported mixed solvent clusters (Fig. 2a). The solvent cluster, as named four-footed "8-shaped" mixed water–ethanol $(H_2O)_6(C_2H_5OH)_2$ cluster, consists of four water (O11A, O13A, O11B, and O13B) and two oxygen atoms (O12A and O12B) of two ethanol molecules as the "8-shaped" main body, as well as two water molecules (O10A and O10B) and $-CH_2-CH_3$ groups of ethanol molecules as its four feet (Fig. 2b). All data of these hydrogen bonds are listed in Table 2. The distances of hydrogen bonds range from 2.651 to 2.875 Å, with the mean value of 2.786 Å, which is slightly longer than that observed in ice-like cyclic $(H_2O)_8$ clusters [17]. Although discrete water clusters have been the subject of theoretical calculations, and have been described in literatures [18,19], mixed solvent clusters have been rarely reported before [20]. As far as we know, such a four-footed "8-shaped" mixed water–ethanol $(H_2O)_6(C_2H_5OH)_2$ cluster is rare example and worth to investigating further. In compound **1**, each mixed solvent cluster links four double chains by its four feet to extend into 3D supramolecular framework.

In compound **2**, the Zn(II) ion lies in a distorted octahedron coordination environment. The coordinated six atoms are three carboxylic O atoms from two $poda^{2-}$ ligands, one O atom from one water molecule and two N atoms of a bpy ligand, respectively (Fig. 3). The bond lengths of Zn–O are in the range of 2.2086–2.3591 Å, and those of Zn–N within 2.3129–2.3212 Å, which accord with the Zn–O and Zn–N bond lengths reported [21]. Just for the flexible structure of the $poda^{2-}$ ligand, the adjacent Zn(II) ions are linked into an infinite 1D zigzag chain by the $poda^{2-}$ anions in a bis(chelate–monodentate) mode (Fig. 4). The chelating bpy ligands coordinated to Zn(II) ions at the opposite side to the phenyl of the $poda^{2-}$ ligand of 1D zigzag chain alternately. Strong π–π stacking interactions (the face to face distance is 3.493 Å) existing between the immediate bipy ligands and phenyl rings of $poda^{2-}$ ligands from adjacent chains, result in a dense zipper-like 2D layer in *ac* plane (Fig. 5). Then, these 2D layers stack along *b* axis according to ABAB manner.

The coordination modes of an organic ligand containing carboxyl group are affected by pH value of a reaction system. In order to verify it, we altered pH value of system of compound **2** from 8–9 to 7 and obtained compound **3**. For **3**, the Zn(II) ion is in a six-coordinated slight distorted triangular-prism environment, in which four oxygen atoms O1, O2, O1A, and O2A from two $Hpoda^{-}$ ligands, and two nitrogen atoms N1 and N1A from a bpy ligand (Fig. 6). And O1, O2A, N1A, and O1A, O2, N1 form two planes of triangular-prism. The Zn–O bond lengths are in the range of 2.2568–2.3515 Å, which are similar to those in compound **2**. The Zn–N distances (2.2530 Å) are slightly shorter than that in compound **2**. The $Hpoda^{-}$ ligand adopts the chelate coordinated mode and holds back the formation of 1D chain.

A notable feature of compound **3** is four neighboring Zn(II) ions via four $C_{12}–H \cdots O_2$ hydrogen bonds (3.453 Å, 115.42°) to form 2D [4,4] supramolecular network parallel to the *ab* plane (Fig. 7). And strong hydrogen bonds between the protonized carbonyl oxygen atoms from two different dangling ligands ($O_4–H \cdots O_1$ 2.667 Å) make 2D networks into 3D 6-connected supramolecular edifice [22]. From the point of view of topology, 3D framework of **3** is a

Table 1
Crystallographic data for compounds **1**, **2**, and **3**

	1	2	3
Empirical formula	$C_{58}H_{54}N_6O_{13}Cd_2$	$C_{20}H_{18}N_2O_5Zn$	$C_{30}H_{26}N_2O_8Zn$
Formula weight	1267.87	431.73	607.9
Crystal system	Triclinic	Monoclinic	Monoclinic
Space group	*P*-1	*P*2(1)/*n*	*C*2/*c*
a (Å)	12.7635(17)	10.2100(17)	16.398(4)
b (Å)	14.9031(19)	17.054(3)	8.6224(18)
c (Å)	15.198(2)	11.1836(18)	20.785
α (deg)	68.796(2)	90	90
β (deg)	84.210(3)	95.296(2)	111.975(2)
γ (deg)	78.093(2)	90	90
V (Å^3), Z	2636.2(6), 2	1938.99(67), 4	2725.28(242), 2
D_{cacl} (g cm^{-3})	2.023	1.47883	1.48151
μ (mm^{-1})	1.725	1.30	0.96
$F(000)$	1288	888.0	1256.0
Reflections collected/unique	13806/9133	16329/3529	12711/2536
R_{int}	0.0258	0.0344	0.0242
Goodness-of-fit on F^2	1.039	1.088	1.135
$R_1{}^a/wR_2{}^b$ [$I > 2\sigma(I)$]	0.0405/0.0747	0.0642/0.1544	0.0649/0.2163
R_1/wR_2 [all data]	0.0751/0.0910	0.0513/0.1413	0.0590/0.2072
Largest differ. peak and hole (e Å^{-3})	0.746/−0.619	0.943/−0.426	1.102/−0.512

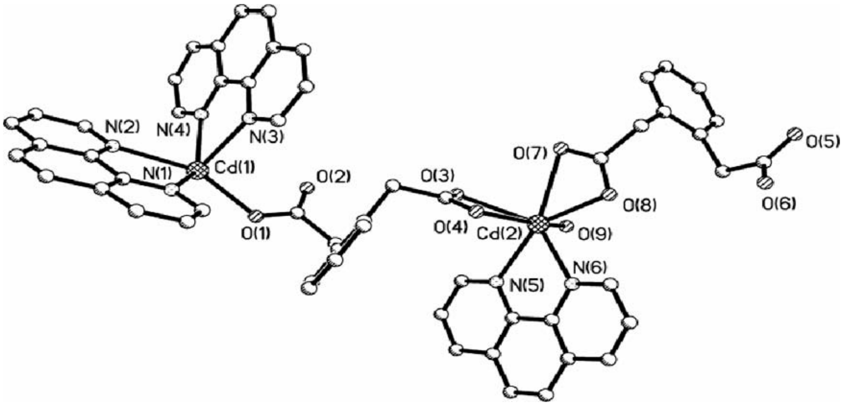

Fig. 1. ORTEP plot showing the asymmetric unit of compound **1**.

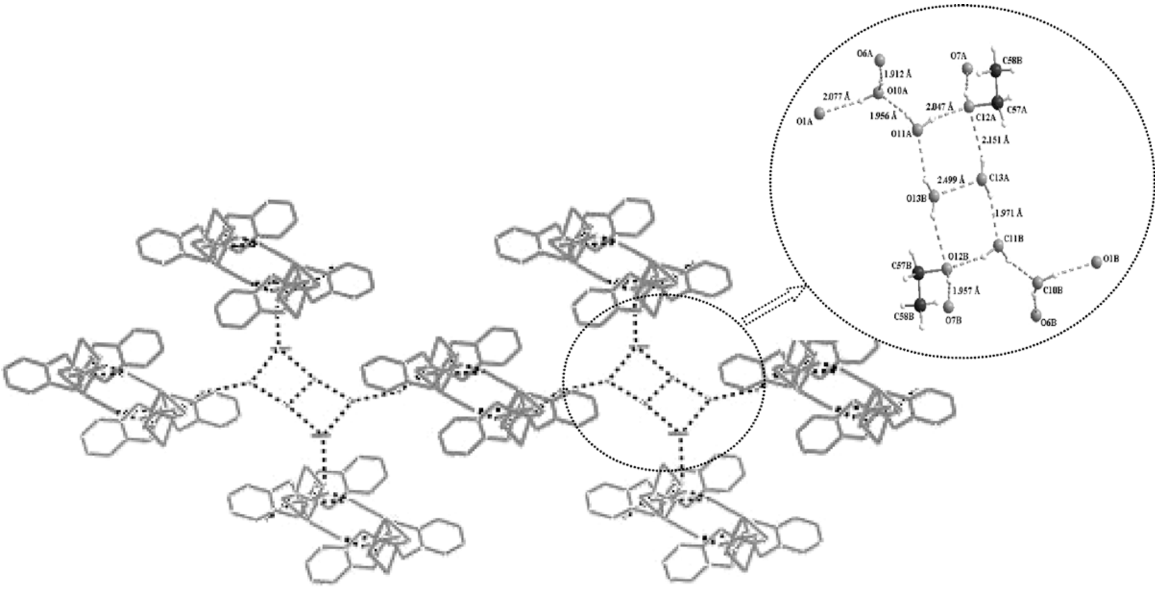

Fig. 2. 3D framework via "8-shaped" four-footed mixed solvent clusters along *a* axis in **1** (carbon atoms of 1,10-phen and hydrogen atoms are omitted for clarity).

distorted α-Po net with the Zn(II) ions as nodes and hydrogen bonds as spacers (9.263 × 9.263 × 11.052 Å) (Fig. 8).

In compounds **1**–**3**, H_2poda ligand adopts three different coordination modes: (1) bis(chelate–bidentate) mode; (2)

Table 2
Hydrogen-bond distances and angles of the mixed solvent cluster in **1**

D–H	d(H···A)/nm	∠DHA/deg	d(D···A)/nm	A
O(9)–H(9C)	0.1870	178.49	0.2719	O(2)
O(9)–H(9C)	0.1880	178.42	0.2730	O(3)
O(10)–H(10A)	0.2078	165.14	0.2908	O(1)
O(10)–H(10B)	0.1913	164.79	0.2742	O(6)
C(11)–H(11A)	0.2045	164.78	0.2874	O(12)
O(11)–H(11B)	0.1957	164.63	0.2786	O(10)
O(12)–H(12)	0.1957	169.28	0.2767	O(7)
O(13)–H(13A)	0.1971	136.38	0.2652	O(11)
O(13)–H(13B)	0.2151	137.31	0.2835	O(12)

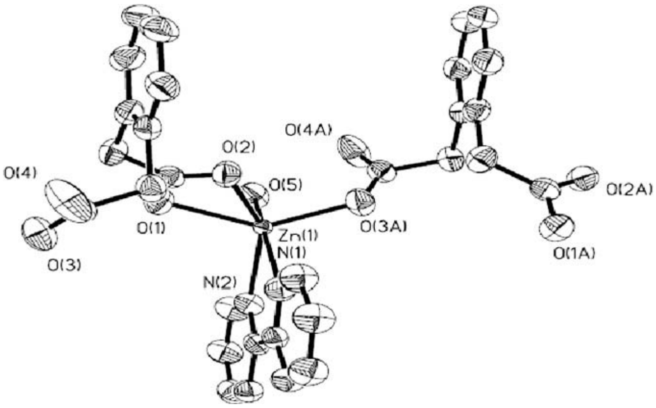

Fig. 3. The coordination environment of Zn in compound **2**.

bis(chelate–monodentate) mode and (3) mono(chelate–deprotonized) mode. These different modes make their different structures. From compounds **2** and **3**, we could discover that pH value of the reaction system plays a key role in constructing the structures of the compounds. Clearly, the pH value influence in the compounds is attributed to its effect on deprotonation of H_2poda ligand.

3.2. Thermal stabilities of compounds *1–3*

Thermogravimetric analyses (TG) have been performed in air for compounds **1–3** from 20 to 700 °C (Fig. 9). For compound **1**, its TG curve shows two main steps of weight losses. The first step started at 40 and completed at 80 °C, which corresponds to the release of one free C_2H_5OH and four H_2O. The observed weight loss of 9.77% is close to the calculated value (9.32%). The final step (310–476 °C) corresponds to the release of bpy and H_2poda ligands, giving cadmium oxides as the final decomposition product which constitute 20.47% (calcd. 20.25%). TG curve of **2** also exhibit two main steps of weight losses. The first step started at 31 °C and completed at 87 °C, which corresponds to the release of one H_2O. The observed weight loss of 5.11% is somehow larger than the calculated value; however, we deem that this is within the experimental error because the results of TG may have larger errors. The secondary step (90–430 °C) corresponds to the release of bpy ligands, giving zinc oxides as the final decomposition product which constitute 18.32% (calcd. 18.85%). For **3**, TG curve shows merely one step of weight loss from 20 to 489 °C, and zinc oxides is the final decomposition product with residue percent of 13.26% (calcd. 13.39%).

3.3. Fluorescent properties of *1* and *2*

As shown in Fig. 10, solid-state fluorescence spectra of **L** and **1**, **2** at room temperature have been determined. The free ligand **L** has fluorescence emission bands centered at ca. 391.8 nm. In comparison with that of the free ligand, compound **1** shows weak red-shifted emission centered at 566 nm ($\lambda_{ex} = 350$ nm), which could be ascribed to the ligand-to-metal-charge-transfer (LMCT), that is, probably due to σ-donations from the cooperation of H_2poda and phen ligands to the CdO core [23]. The similar emission at 375 nm was observed for **2** upon excitation at 220 nm. Compared to that of **L**, the maximum emission band of **2** is blue-shift shifted. It is electronic that the emission of **2** originated from the $\pi \rightarrow \pi^*$ electronic transition of **L** ligand [24]. By comparing the emission spectra of **2** and ligand, we could conclude that the enhancement of luminescence in **2** may be attributed to the rigidity of **2**. This rigidity is favor of energy transfer and reduces the loss of energy through a radiationless pathway. The possible explanation for the different emission properties of **1** and **2** is the different structure in **1** and **2**. Because compound **2** are usually stable and insoluble in common solvents arising from their polymeric structures, it is possible to develop high stable luminescent materials based on them.

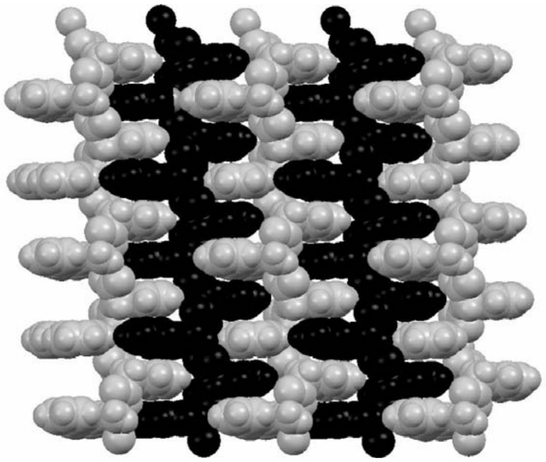

Fig. 5. 2D zipper-like network *via* π–π stacking interactions of **2**.

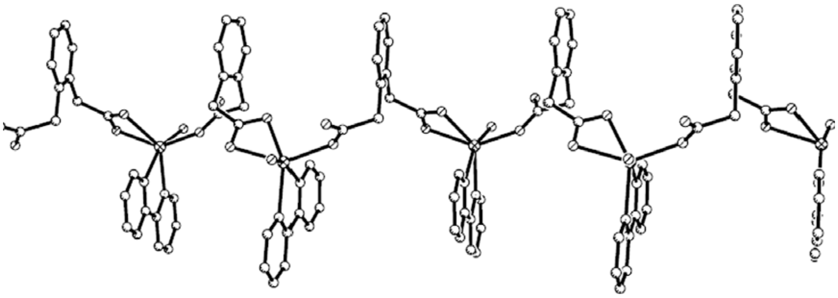

Fig. 4. 1D chain along b axis in compound **2**.

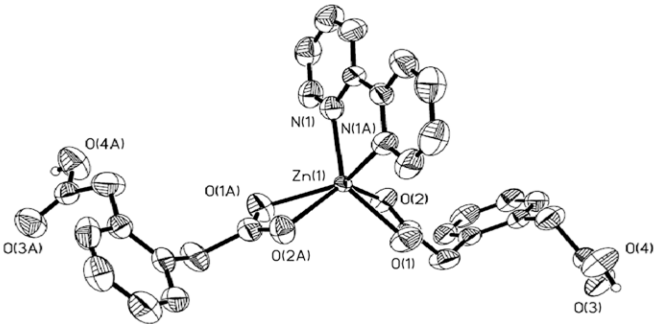

Fig. 6. The coordination environment of Zn(II) in **3**.

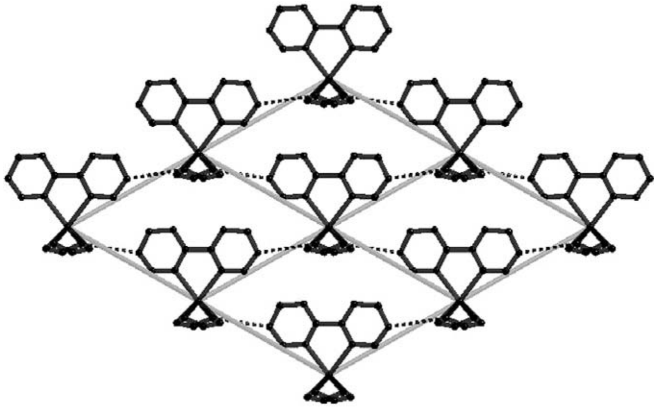

Fig. 7. The view of altemation of **2** along the *ab* plane (carbon atoms and hydrogen atoms of *O*-phenylenediacetic acid and hydrogen atoms are omitted for clarity).

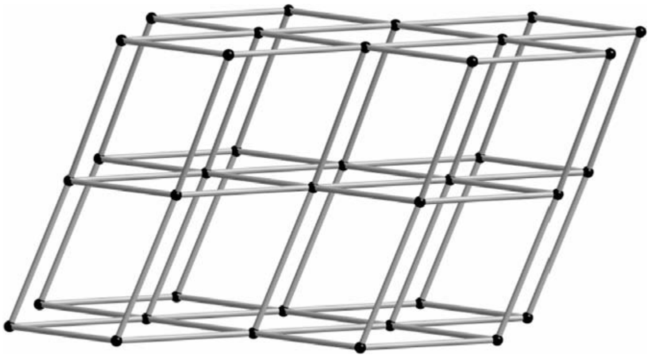

Fig. 8. The schematic topology of the compressed α-Po net in **3**.

4. Conclusions

In summary, using H_2poda ligand and NN chelated ligands, we isolated three metal (Cd/Zn)–organic compounds with 1D chains (**1** and **2**) and 0D monometer (**3**) structures. Noteworthy, in **1** an unprecedented four-footed “8-shaped” mixed water–ethanol $(H_2O)_6(C_2H_5OH)_2$ cluster links adjacent 1D double chains and extends **1** into 3D structures, and is one of rare mixed solvent cluster examples in metal–organic coordination polymer research.

5. Supplementary material

Supplementary data are available from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 1233 336033; e-mail: deposit@ccdc.cam.ac.uk or http:www.ccdc.cam.ac.uk) and can be obtained on request, quoting the deposition Nos. 683323, 683324, and 683325.

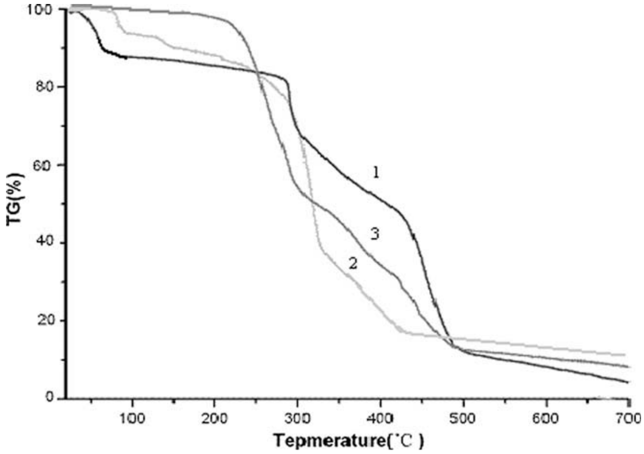

Fig. 9. TG curves of compounds **1–3**.

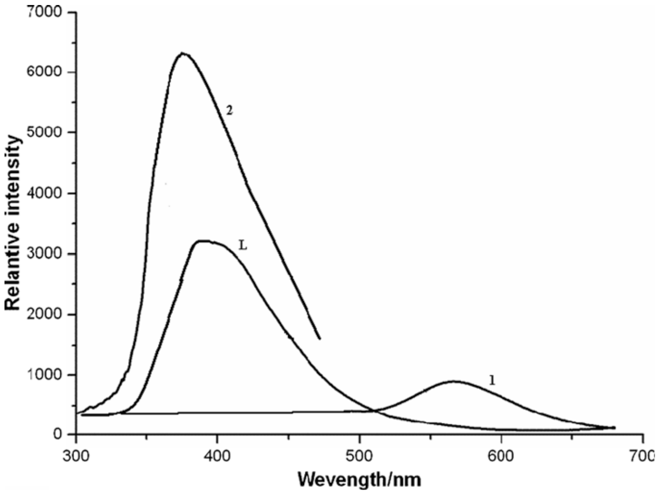

Fig. 10. Emission spectra of **1**, **2**, and free ligand H_2poda (L) in solid-state at room temperature.

Acknowledgments

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