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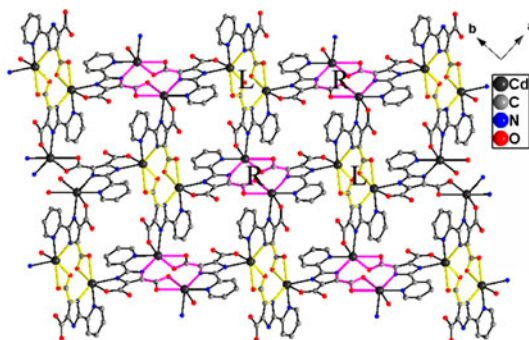
A new 3-D Cd(II) coordination polymer with metallacalix[4] arene building blocks based on 2-(pyridin-2-yl)-1H-imidazole-4,5-dicarboxylic acid

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A new metal–organic framework based on a multifunctional ligand, 2-(pyridin-2-yl)-1H-imidazole-4,5-dicarboxylic acid (H_3PIDC), $[Cd(HPIDC)]_n$ (**1**), has been obtained under hydrothermal conditions and characterized by single-crystal X-ray diffraction analysis, elemental analysis, IR spectrum, and powder X-ray diffraction. Compound **1** is constructed by metallacalix[4]arene building blocks $Cd_4(HPIDC)_4$ with single-left (L) and single-right-helical (R) chains sitting on the four corners of $Cd_4(HPIDC)_4$ units. The structure of **1** can be described as a (3,3)-connected net with the $(8^2 \times 10)$ topology. The solid-state photoluminescence has been carried out at room temperature.

Keywords: Cd(II) coordination polymer; Metallacalix[4]arene building block; Photoluminescence property

1. Introduction

The science of functional coordination polymers has become one of the most intense areas of investigation due to their fascinating topological structures and potential applications in

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gas storage, separation, catalysis, photoluminescence, magnetism, etc. [1–5]. The resultant networks are highly dependent on the geometries and coordination environments of the metal centers and ligands [6–10]. In order to build unique structures, judicious selection of multifunctional organic ligands is an effective approach, and N-heterocyclic carboxylates, such as 4,5-imidazoledicarboxylic acid [11, 12], 3,4-pyridinedicarboxylic acid [13, 14], and 2,3-dicarboxypyrazine [15, 16] have been employed. 2-(Pyridin-2-yl)-1H-imidazole-4,5-dicarboxylic acid (H₃PIDC) is an unsymmetric N-heterocyclic carboxylic acid, which can be used to construct novel structures. The ortho position of pyridine-N and the imidazole-N, carboxylic-O and imidazole-N enhances its coordination ability. Multiple coordination points endow it with multiform coordination modes. Although several coordination polymers based on H₃PIDC have been studied by our group and others [17–22], more research needs to be done to explore new compounds with H₃PIDC.

The coordination chemistry and crystal engineering of Cd(II) have attracted attention from chemists owing to the attractive structural diversities and potential applications of photoluminescent and nonlinear optical properties [23–29]. The d¹⁰ configuration makes Cd(II) compounds adopt varied coordination geometries such as tetrahedral, trigonal bipyramidal, square pyramidal, or octahedral [23–29]. Therefore, the Cd(II) salt is particularly suitable for the assembly of coordination frameworks.

Here, we report a new 3-D coordination polymer constructed by metallacalix[4]arene building blocks based on H₃PIDC, [Cd(HPIDC)]_n (**1**). Compound **1** owns a (8² × 10) 3-D network, which is constructed by metallacalix[4]arene building blocks Cd₄(HPIDC)₄, and single-left (L) and single-right-handed (R) helical chains are on the four corners of Cd₄(HPIDC)₄ units. Solid-state photoluminescence has been carried out at room temperature.

2. Experimental

2.1. Materials and physical measurements

All reagents were purchased commercially and used without further purification. Elemental analyses for C, H, and N were performed on a Perkin-Elmer 2400 Elemental Analyzer. IR (KBr pellet) spectrum was recorded from 4000 to 450 cm⁻¹ using a Perkin-Elmer Spectrum One FT-IR spectrometer. The thermal behavior was studied by thermogravimetric analyses (TGA) on a Perkin-Elmer thermal analysis system under N₂ with a heating rate of 10 °C min⁻¹. Fluorescence data were obtained using a Hitachi F7000 fluorescence spectrophotometer. Powder X-ray diffraction (PXRD) patterns were collected using a Rigaku Dmax 2000 X-ray diffractometer with graphite-monochromated Cu-Kα radiation (λ = 0.15418 nm) in the 2θ range of 5°–50° with an increment of 0.02°.

2.2. Synthesis of **1**

A mixture of H₃PIDC (0.1 mmol, 0.0233 g), CdCl₂·2.5H₂O (0.3 mmol, 0.0685 g), pyrazole (0.1 mmol, 0.0068 g), and distilled water (10 mL) was stirred at room temperature for half an hour. The pH was adjusted to 3.0 with HCl solution. The mixed solution was continued stirring for 1 h, then sealed in a 15-mL Teflon-lined stainless-steel reactor and heated at 150 °C for 82 h. The mixture was cooled to room temperature at a rate of 6 °C h⁻¹ and orange block crystals were obtained. Yield, 53% (based on H₃PIDC). Anal. Calcd for C₁₀H₅CdN₃O₄ (Mr = 343.58): C, 39.93; H, 1.46; N, 12.22%. Found: C, 39.77; H, 1.51; N,

12.38%. IR (KBr, cm^{-1}): 3438(m), 1680(s), 1606(m), 1525(s), 1486(s), 1419(m), 1383(m), 1384(m), 1330(m), 1250(m), 1157(m), 1117(w), 1050(w), 1015(w), 998(w), 863(m), 797(m), 781(w), 772(w), 750(s), 719(s), 636(m), 563(m).

2.3. X-ray crystallography

Crystallographic data for **1** were collected on an Agilent Technology SuperNova Eos Dual system with a (Cu) microfocus source ($\lambda = 1.54184 \text{ \AA}$) and focusing multilayer mirror optics. The structure was then solved by the direct method followed by the difference Fourier method and refined by full-matrix least-squares on F^2 using the SHELXS-97 package. Anisotropic thermal parameters were applied to all non-hydrogen atoms. All hydrogens were placed geometrically. Details of the final refinement of **1** are given in table 1. Selected bond lengths and angles are listed in table 2.

3. Results and discussion

3.1. Synthesis

Pyrazole was added in the reaction system in the process of preparing **1**, and pyrazole plays an important role in the synthesis of **1**. If pyrazole was removed from the system, we obtain a 3-D coordination polymer $[\text{Cd}_4(\text{HPIDC})_4(\text{H}_2\text{O})]_n$ [20], which could be also obtained when 4,4'-bipyridine and pyridine are added in the reaction system. If CdCl_2 and H_3PIDC were added at the ratio of 1 : 2 in the reaction system, we got a 0-D coordination compound $[\text{Cd}(\text{H}_2\text{PIDC})_2(\text{H}_2\text{O})] \cdot \text{H}_2\text{O}$ [17]. If we added CdCl_2 , H_3PIDC and oxalic acid at the ratio of 4 : 1 : 1 in the reaction system, a 1-D coordination compound $\{[\text{Cd}(\text{H}_2\text{PIDC})_2(\text{H}_2\text{O})] \cdot 2\text{H}_2\text{O}\}_n$ was obtained [17] (scheme 1).

Table 1. Crystal data and structure refinements for **1**.

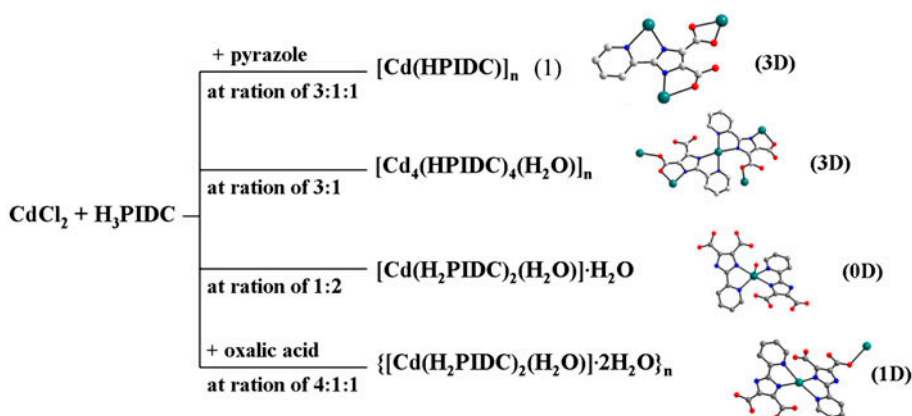
Empirical formula	$\text{C}_{10}\text{H}_5\text{CdN}_3\text{O}_4$
Formula weight	343.57
Crystal system	Orthorhombic
Space group	$Fdd2$
a (\AA)	22.7949(4)
b (\AA)	25.4892(5)
c (\AA)	7.4201(2)
α ($^\circ$)	90
β ($^\circ$)	90
γ ($^\circ$)	90
V (\AA^3)	4311.25(16)
Z	16
μ (mm^{-1})	16.380
D (g cm^{-3})	2.117
$F(0\ 0\ 0)$	2656
R (int)	0.0288
GOF on F^2	1.078
Final R^{ab} indices [$I > 2\sigma(I)$]	$R_1 = 0.0334$, $wR_2 = 0.0833$
R indices (all data)	$R_1 = 0.0335$, $wR_2 = 0.0835$

$$^{\text{a}}R_1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}$$

$$^{\text{b}}wR_2 = \frac{[\sum w(|F_o|^2 - |F_c|^2)^2 / \sum |w(F_o)|^2]^{1/2}}$$

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

Cd(1)–N(2)	2.212(5)	Cd(1)–O(1) ^{#1}	2.313(5)
Cd(1)–N(1) ^{#2}	2.340(5)	Cd(1)–O(4) ^{#2}	2.355(6)
Cd(1)–N(3)	2.401(6)	Cd(1)–O(2) ^{#1}	2.471(4)
N(2)–Cd(1)–O(1) ^{#1}	108.52(17)	N(1) ^{#2} –Cd(1)–N(3)	89.79(19)
N(2)–Cd(1)–N(1) ^{#2}	141.89(17)	O(4) ^{#2} –Cd(1)–N(3)	98.4(2)
O(1) ^{#1} –Cd(1)–N(1) ^{#2}	107.33(17)	N(2)–Cd(1)–O(2) ^{#1}	127.34(17)
N(2)–Cd(1)–O(4) ^{#2}	78.06(18)	O(1) ^{#1} –Cd(1)–O(2) ^{#1}	54.72(17)
O(1) ^{#1} –Cd(1)–O(4) ^{#2}	160.4(2)	N(1) ^{#2} –Cd(1)–O(2) ^{#1}	84.60(16)
N(1) ^{#2} –Cd(1)–O(4) ^{#2}	72.48(18)	O(4) ^{#2} –Cd(1)–O(2) ^{#1}	106.3(2)
N(2)–Cd(1)–N(3)	71.0(2)	N(3)–Cd(1)–O(2) ^{#1}	151.6(2)
O(1) ^{#1} –Cd(1)–N(3)	101.2(2)		

Symmetry codes: ^{#1}–*x* + 1/2, –*y* + 1, *z* + 1/2.^{#2}–*x* – 1/4, –*y* + 3/4, *z* + 1/4.

Scheme 1.

3.2. Crystal structure analysis

Single-crystal analysis reveals that **1** crystallizes in the orthorhombic space group *Fdd2*. The asymmetric unit of **1** comprises one crystallographically independent Cd(II) and a partially deprotonated HPIDC²⁻. As shown in figure 1, Cd1 is six-coordinate by three nitrogens (N1^{#2}, N2, and N3) and three oxygens (O1^{#1}, O2^{#1}, and O4^{#2}) from three individual HPIDC²⁻ ligands, respectively, exhibiting a distorted octahedral geometry. In the IR spectrum of **1** (figure S1, see online supplemental material at <http://dx.doi.org/10.1080/00958972.2015.1075240>), the band at 1680 cm⁻¹ indicates a protonated carboxylic acid. Each HPIDC²⁻ connects with three Cd ions, adopting μ_3 -*kN*, N': *kO*, N'': *kO'*, O'' coordination, in which N2 and N3 chelate Cd1, O1 and O2 chelate Cd1^{#3}, and O4 and N1 chelate Cd1^{#4}. As shown in scheme S1, 10 coordination modes of H₃PIDC ligand have been reported [17–21], and the coordination in **1** has not previously been reported. HPIDC²⁻ employs pyridine N3 and the *ortho* imidazole N2 (named NN) to chelate to Cd1, and the bond distances (Cd1–N2 = 2.212(5) Å and Cd1–N3 = 2.401(6) Å) compare to Cd–H₃PIDC coordination polymers [17, 20]. Another imidazole N1 and the *ortho* carboxylate O4 (named NO) chelate to the metal center, and the Cd–O distance (Cd1–O4^{#2} = 2.355(6) Å) is slightly longer than the Cd–N distance (Cd1–N1^{#2} = 2.340(5) Å), which is close to those in $[\text{Cd}_4(\text{HPIDC})_4(\text{H}_2\text{O})]_n$ (for which Cd–O(*ortho* carboxylato) distances are 2.330(2) and 2.349(2)

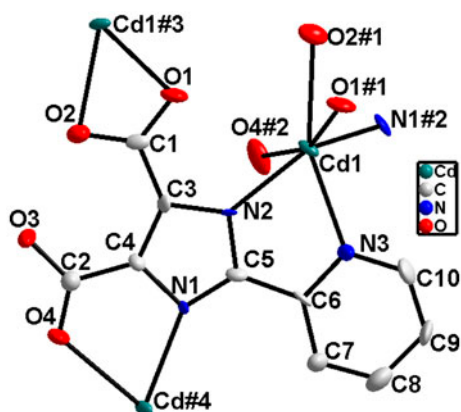


Figure 1. The coordination mode of HPIDC²⁻ and the coordination environment of Cd(II) with 50% probability thermal ellipsoids. Hydrogens are omitted for clarity. Symmetry codes: #1, $-x + 1/2, -y + 1, z + 1/2$; #2, $x - 1/4, -y + 3/4, z + 1/4$; #3, $-x + 1/2, -y + 1, z - 1/2$; #4, $x + 1/4, -y + 3/4, z - 1/4$.

Å; Cd–N_(imidazole) distances are 2.355(2) and 2.372(2) Å [20]. In the Cd–H₃BuPhIDC compounds reported by Yin *et al.* [23(b)], Cd–O_(ortho carboxylato) distances (2.456(4), 2.371(4), 2.387(2), and 2.442(2) Å) are longer than Cd–N_(imidazole) distances (2.206(4), 2.260(4), 2.271(2) and 2.310(2) Å), which is different from **1**. The carboxylic group also chelates (named OO), and the Cd–O bond distances (Cd1–O1^{#1} = 2.313(5) Å, Cd1–O2^{#1} = 2.471(4) Å) are consistent with reported Cd(II)-organic carboxylic ligand coordination polymers [30].

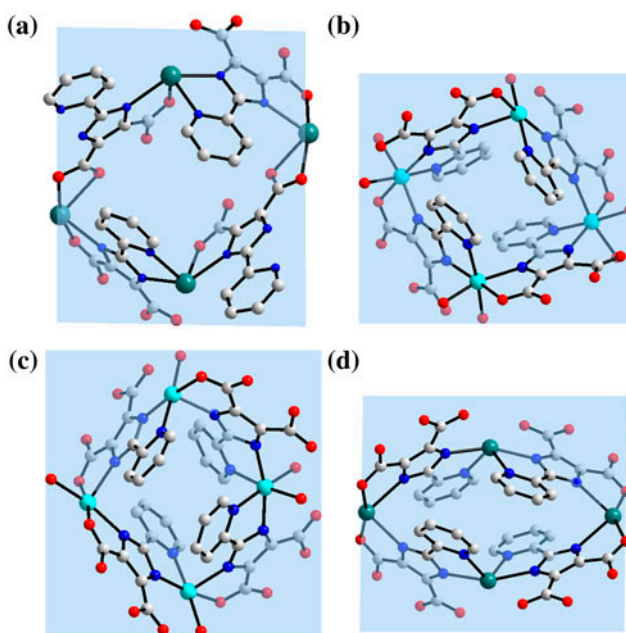


Figure 2. The metallacalix[4]arene Cd₄(HPIDC)₄ unit in **1** (a) and in the references reported by Li and Zheng (b) [20, 21], (mode (a): OO–NO–NN–NO–OO–NO–NN–NO; mode (b): NN–NO–NN–NO–NN–NO–NN–NO; mode (c): NO–NN–NO–NN–NN–NO–NN–NO; mode (d): NO–NN–NN–NO–NO–NN–NN–NO).

Four kinds of metallacalix[4]arene building blocks $M_4(\text{HPIDC})_4$ ($M=\text{Ni}$, Cd and Co) have been reported by Li, Zheng, and our group [20, 21] (figure 2). In **1**, four cadmium ions are connected by four HPIDC^{2-} ligands into a metallacalix[4]arene building block $\text{Cd}_4(\text{HPIDC})_4$ with $\text{Cd}\cdots\text{Cd}\cdots\text{Cd}$ angles of 73.43° and 99.17° and $\text{Cd}\cdots\text{Cd}$ distances along the edges being 6.54 and 8.35 Å (figures 2(a) and S2). In one $\text{Cd}_4(\text{HPIDC})_4$, two opposite HPIDC^{2-} ligands use OO and NO and the other two HPIDC^{2-} ligands use NN and NO to connect four Cd ions. Four pyridine rings of the ligands are all on the same side of the four Cd(II) ions. This structure is different from the structure of the three metallacalix[4]arenes $M_4(\text{HPIDC})_4$ reported by Li *et al.* [20] and Zheng *et al.* [21] [figure 2(b)–(d)], in which all four HPIDC^{2-} ligands use NN and NO to coordinate four metal ions into a metallacalix[4]arene $M_4(\text{HPIDC})_4$, and there are two pyridine rings on each side of the four metal ions; those metals are all coordinated by four NN and NO but in different order.

In **1**, Cd(II) ions connect neighboring $\text{Cd}_4(\text{HPIDC})_4$ units into a 3-D open framework with apertures along the c axis (figure S3). PLATON calculations show that the guest accessible volume (4311.3 \AA^3 per unit cell) comprises only 7.5% of the unit cell volume. Compound **1** contains single-left (L) and single-right-helical (R) chains with the same pitch 7.42 Å, which are on the four corners of $\text{Cd}_4(\text{HPIDC})_4$ units along the c axis [figure 3(a)]. The two helices have the same repeat unit $-\text{Cd1}-\text{N2}-\text{C3}-(\text{O1C1O2})-\text{Cd1}-$ [figure 3(b)]. From a topological point of view, if HPIDC^{2-} anions are reduced to 3-connected nodes, and Cd(II) ions are viewed as 3-connected nodes, the structure of **1** can be described as a (3,3)-connected net with the $(8^2 \times 10)$ topology (figure S4).

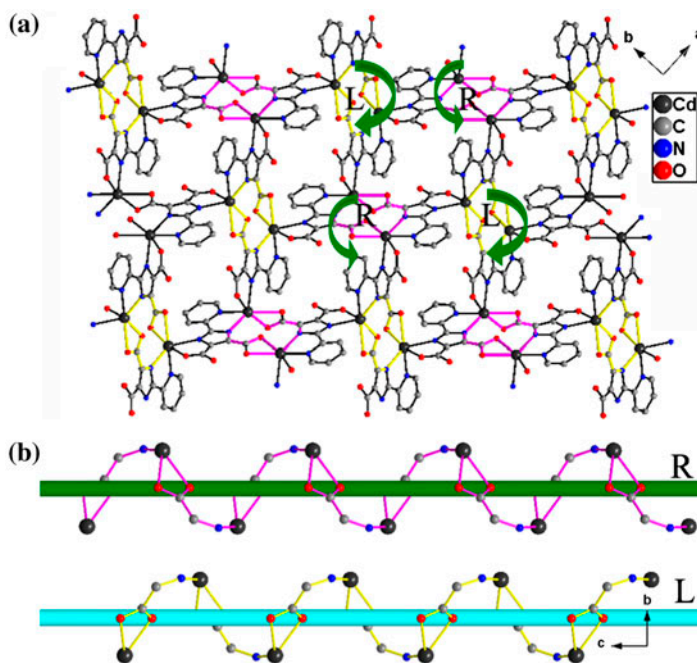


Figure 3. (a) Two kinds of helical chains on the four corners of a $\text{Cd}_4(\text{HPIDC})_4$ unit and (b) the single-left (L) and single-right-helical (R) chains have the same repeat unit.

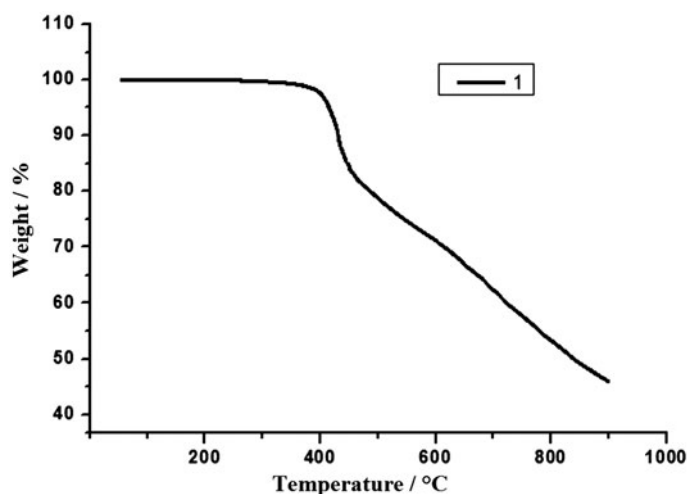


Figure 4. The TG analysis curve of **1**.

3.3. Powder X-ray diffraction and TGA

In order to check the phase purity of **1**, the original sample was measured by PXRD at room temperature. The peak positions of the measured PXRD pattern are in agreement with the simulated patterns from the respective single-crystal data for the complex (figure S5).

The thermogravimetric (TG) analysis of **1** (figure 4) was carried out on a Perkin-Elmer thermal analysis system from room temperature to 900 °C under a nitrogen atmosphere with a heating rate of 10 °C min⁻¹. The TG analysis curve of **1** shows that there is no obvious weight loss from 50 to 370 °C. The decomposition of the organic ligand began from 370 °C. However, complete decomposition was not achieved at 900 °C. The weight loss was observed to be about 56.14% from 370 to 900 °C, smaller than the theoretical value 62.63% on consideration of the organic ligand release.

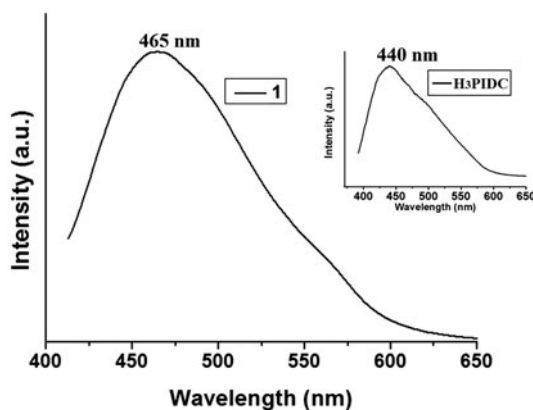


Figure 5. The solid-state fluorescence spectra of H₃PIDC ($\lambda_{\text{ex}} = 365$ nm) and **1** ($\lambda_{\text{ex}} = 391$ nm) at room temperature.

3.4. Fluorescence property

As shown in figure 5, free H₃PIDC exhibits an emission peak with maximum at 440 nm upon excitation at 365 nm, which can be attributed to the $\pi \rightarrow \pi^*$ and/or $n \rightarrow \pi^*$ transitions of the aromatic rings. The emissions are observed at 465 nm ($\lambda_{\text{ex}} = 391$ nm) for **1**. The emission of the compound is neither metal-to-ligand charge transfer nor ligand-to-metal charge transfer since Cd(II) is difficult to oxidize or to reduce due to d¹⁰ configuration [30–32]. The emissions are attributed to intra-ligand fluorescence emission. Comparing with the emission spectrum of free H₃PIDC, the emission peak of **1** is red-shifted by 25 nm, which is considered to mainly arise from the increased rigidity of the organic ligand when coordinating to Cd(II) [32], probably due to decreased energy gaps between the highest occupied molecular orbital and the lowest unoccupied molecular orbital of the ligands when coordinating to metal ions [33].

4. Conclusion

A new 3-D coordination polymer based on H₃PIDC has been hydrothermally synthesized. Compound **1** is constructed with metallacalix[4]arene building blocks Cd₄(HPIDC)₄. Single-left and single-right-helical chains can be found at the four corners of the metallacalix[4]arene building block. The structure of **1** can be described as a (3,3)-connected net with the (8² × 10) topology. H₃PIDC exhibits a new coordination mode. In addition, **1** may be a candidate as one of the advanced materials for light emitter applications owing to its strong fluorescence emission and high thermal stability.

Supplementary material

Crystallographic data reported in this article have been deposited with the Cambridge Crystallographic Data Center as supplementary publication. CCDC number is 975830. This data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html> (or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336 033; E-mail: deposit@ccdc.cam.ac.uk).

Disclosure statement

No potential conflict of interest was reported by the authors.

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