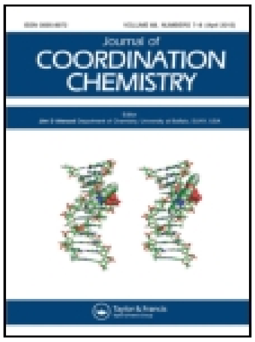

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Synthesis, crystal structure, and characterization of a cadmium(II) complex containing an octacarboxylate ligand

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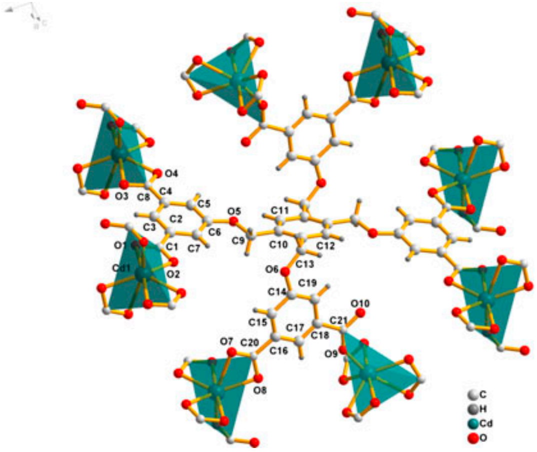

We present a MOF obtained by solvothermal synthesis from assembly of an octacarboxylate ligand and Cd(II) salt in DMF. The title metal–organic framework possesses a porous 3-D framework with the total solvent-accessible volume about 1800.7 A^3 per unit cell, comprising 52.7% of the total crystal volume. We studied the photoluminescent properties of L and **1** in the solid state. The solid-state diffuse reflectance UV–vis spectra of **1** show possible semiconductor property.

A cadmium(II) complex containing an octacarboxylate ligand (**L**), $Cd_2(C_{42}H_{22}O_{20})\cdot 5(H_2O)\cdot 4(CH_3NH_2CH_3)$, has been solvothermally synthesized and characterized by elemental analysis, spectroscopic studies (IR spectrum, diffuse reflectance spectrum, and solid-state emission spectrum), powder X-ray diffraction, and single-crystal X-ray diffraction. In **1**, Cd1 is coordinated by four carboxylates from four different **L** and **L** is coordinated to eight Cd ions with its eight carboxylates. Compound **1** possesses a porous 3-D framework with total solvent-accessible volume of 1800.7 Å^3 per unit cell, comprising 52.7% of the total crystal volume.

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[1] J.S. and L.Y. contributed equally.

Keywords: Cd(II) complex; Octacarboxylate ligand; Diffuse reflectance spectrum

1. Introduction

With knowledge of metal–organic framework (MOF) properties, gas storage [1] and separation [2], catalysis [3], and sensing [4] have made some significant breakthroughs. Properties such as water absorption [5], temperature-dependent luminescence [6], proton conduction [7], and explosive detectors [8] have been observed, too. Generally, the properties of MOFs are based on the structural diversity. During the design of MOFs, carboxylic ligands which have transformable coordination modes are always good choices [9].

Among the carboxylic ligands, ligands derived from 3,5-substituted dicarboxylates (isophthalates) that serve as versatile molecular building blocks for construction of a range of interesting coordination polymers with Cu(II) ions have been widely reported [10]. However, ligands based on tetra-isophthalates assembled with Cd(II) salts are rarely reported [11]. As Ma *et al.* reported, augmented pcu 3D networks containing two distinct polyhedral cages were assembled with a porphyrin-based tetra-isophthalate and Cd(II) salts [11(a)].

In a previous study, octacarboxylate 5,5′,5″,5‴-[1,2,4,5phenyltetramethoxy]tetraisophthalate [12] (**L**) (scheme 1) assembled with Cu(II) in square paddlewheel dimers generated three polyhedral cages: truncated tetrahedron, truncated cube, and truncated cuboctahedron. To study the coordination modes of **L** and the assembled structures with other metal ions, herein, we present a MOF obtained by solvothermal synthesis from the assembly of octacarboxylate **L** and $Cd(NO_3)_2 \cdot 6H_2O$ in DMF.

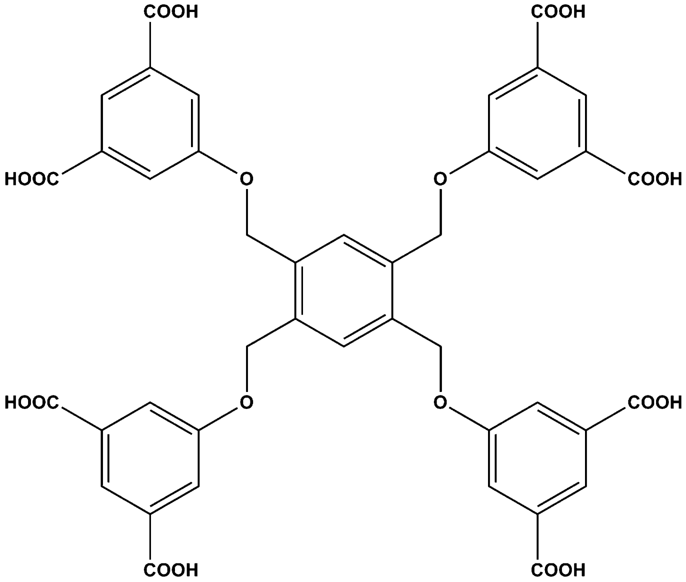
2. Experimental

2.1. *Materials and physical measurements*

L was prepared according to the literature method [12]. Other reagents and solvents were commercially available and used as received. C, H, and N elemental analyses were performed on a Vario ELIII. FT-IR data were recorded on a VERTEX 80 with KBr pellets from 4000 to 400 cm^{-1}. TGA data were obtained on a TGA-50 (SHIMADZU) thermogravimetric analyzer with a heating rate of 10 °C min^{-1} under N_2. The powder X-ray diffraction patterns were collected with a scan speed of 0.5 s deg^{-1} on a Bruker Avance D8 (40 kV, 40 mA) diffractometer with Cu radiation ($\lambda = 1.54056$ Å) at room temperature. Diffuse reflectance spectra were recorded on a U-41000 Spectrophotometer (Liquid) using $BaSO_4$ powder as a 100% reflectance reference. The solid-state luminescence spectra were measured on an F-4500 FL spectrophotometer with the EX Slit: 20.0 nm, EM Slit: 20.0 nm, and PMT Voltage: 500 V for **L** and **1**.

2.2. *Synthesis of $Cd_2L \cdot 5(H_2O) \cdot 4(CH_3NH_2CH_3)$ (1)*

To the solution of **L** (0.070 g, 0.082 mmol) in 7 mL DMF was added a solution of $Cd(NO_3)_2 \cdot 4H_2O$ (0.025 g, 0.081 mmol) in 3 mL DMF, then a drop of 12 mol L^{-1} HCl was added. The mixture was heated to 150 °C in a sealed vial for 72 h, then allowed to cool to room temperature. Colorless block crystals (0.0064 g) of **1** were obtained by filtration and washed with ethyl acetate three times. Yield 11.74% (based on Cd). Calcd for



Scheme 1. The structure of L.

$C_{50}H_{64}O_{25}Cd_2N_4$ (Fw = 1345.85): C, 43.50; H, 4.95; N, 4.18%. Found: C, 43.41; H, 5.08; N, 4.06%. FT-IR (KBr pellet, cm^{-1}): 3386 m, b, 3069 m, b, 2793 m, 2476 w, 1663 w, 1613 w, 1564 vs 1454 m, 1371 vs, 1259 m, 1123 w, 1042 m, 891 w, 779 m, 729 m, 662 w.

2.3. *X-ray diffraction crystallography*

Single-crystal X-ray crystallographic data were collected on a Bruker Smart APEX II diffractometer with a CCD area detector. Raw data collection and reduction were done using APEX2 software [13]. Adsorption corrections were applied using the SADABS routine. The structures were solved by direct methods and refined by full-matrix least-squares on F^2 using the SHELXTL software package [14]. Nonhydrogen atoms were refined with anisotropic displacement parameters during the final cycles. All hydrogens were calculated in ideal positions with isotropic displacement parameters and refined with a riding model. Many guest water molecules and dimethylammonium cations existed in the channels; they were all located in the difference Fourier map and their atomic parameters were refined subsequently. The phase purity of bulk crystal samples was confirmed by X-ray powder diffraction studies (figure 1). Details of the crystal parameters, data collection, and refinement results are summarized in table 1. Selected bond lengths and angles of **1** are given in table 2. The hydrogen-bond geometry is shown in table 3.

3. Results and discussion

3.1. *IR spectra*

Infrared spectrum of **1** shows strong peaks at 1371 and 1564 cm^{-1} attributed to asymmetric and symmetric stretches of carboxylates [15], respectively. IR peaks attributable to the

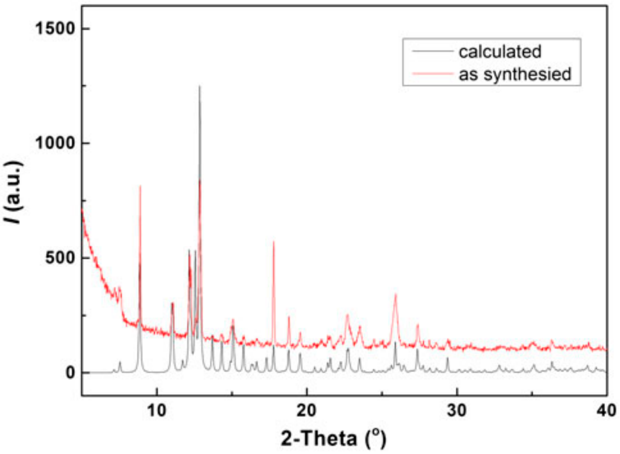


Figure 1. X-ray powder diffraction patterns of **1**.

Table 1. Crystal data and structure refinement parameters for **1**.

Empirical formula	$C_{50}H_{64}Cd_2N_4O_{25}$
Formula weight	1345.85
Temperature (K)	291(2)
Crystal system	Monoclinic
Wavelength (Å)	0.71073
Space group	*P2(1)/c*
Unit cell dimensions (Å, °)	
a	10.031(2)
b	14.541(3)
c	23.562(5)
β	96.381(3)
Volume (Å^3, *Z*)	3415.3(1)
Calculated density (g cm^{-3})	1.309
F(0 0 0)	1376
Absorption coefficient, μ (mm^{-1})	0.695
No. of reflections measured	16,552
No. of independent reflections	5968
R_{int}	0.0671
No. of formula units per unit cell, *Z*	4
R_1, wR_2 [$I \geq 2\sigma(I)$]	0.0825/0.2122
R_1, wR_2 [all data]	0.1382/0.2293
GOF	1.069
Largest diff. peak and hole (e Å^{-3})	1.491/−0.646

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

aromatic ring in **1** (3069, 1613, 1454, and 1123 cm^{-1}) are observed. The bands corresponding to asymmetric and symmetric stretches $\nu_{c\text{–}o\text{–}c}$ are at 1042 and 1259 cm^{-1} in spectra of **1**.

3.2. *Thermal analysis*

The weight loss of 6.6% (Calcd 6.7%) from room temperature to 126 °C (figure 2) is assigned to release of guest water molecules. The loss of uncoordinated water molecules

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

Cd(1)–O(9)#4	2.148(1)	Cd(1)–O(1)	2.486(7)
Cd(1)–O(8)#3	2.216(7)	Cd(1)–O(7)#3	2.453(7)
Cd(1)–O(2)	2.315(6)	Cd(1)–O(3)#5	2.546(7)
Cd(1)–O(4)#5	2.295(7)		
O(9)#4–Cd(1)–O(8)#3	103.3(4)	O(8)#3–Cd(1)–O(7)#3	53.6(3)
O(9)#4–Cd(1)–O(2)	106.3(4)	O(2)–Cd(1)–O(7)#3	95.7(3)
O(8)#3–Cd(1)–O(2)	134.2(3)	O(4)#5–Cd(1)–O(7)#3	96.3(3)
O(9)#4–Cd(1)–O(4)#5	96.5(4)	O(1)–Cd(1)–O(7)#3	93.6(3)
O(8)#3–Cd(1)–O(4)#5	130.6(3)	O(9)#4–Cd(1)–O(3)#5	86.1(4)
O(2)–Cd(1)–O(4)#5	79.5(2)	O(8)#3–Cd(1)–O(3)#5	84.7(2)
O(9)#4–Cd(1)–O(1)	92.3(4)	O(2)–Cd(1)–O(3)#5	131.1(2)
O(8)#3–Cd(1)–O(1)	91.6(2)	O(4)#5–Cd(1)–O(3)#5	51.9(2)
O(2)–Cd(1)–O(1)	53.4(2)	O(1)–Cd(1)–O(3)#5	175.5(2)
O(4)#5–Cd(1)–O(1)	132.6(2)	O(7)#3–Cd(1)–O(3)#5	86.2(3)
O(9)#4–Cd(1)–O(7)#3	156.2(4)		

Notes: Symmetry transformations used to generate equivalent atoms: #3 $-x+1$, $-y+1$, $-z+1$; #4 x, $-y+1/2$, $z-1/2$; #5 $x+1$, y, z.

Table 3. Hydrogen-bond geometry (Å, °) of **1**.

D-H···A	D-H	H···A	D···A	D-H···A
C(13)–H(13B)···O(13)	0.97	2.57	3.54(5)	172.6
C(22)–H(22B)···O(8)#7	0.96	2.36	3.24(2)	152.9
C(22)–H(22B)···O(3)#8	0.96	2.94	3.46(2)	115.5
C(22)–H(22C)···O(11)#3	0.96	2.19	3.14(3)	169.3
C(23)–H(23C)···O(2)#3	0.96	2.69	3.405(1)	132.2
N(1)–H(1A)···O(4)#9	0.90	2.18	2.98(2)	147.4
N(1)–H(1B)···O(10)#10	0.90	2.13	2.875(1)	140.0
O(11)–H(11A)···O(3)#11	0.85	2.52	3.29(4)	151.0
O(14)–H(14A)···O(11)#9	0.85	2.36	3.16(5)	156.7
O(14)–H(14A)···O(13)#9	0.85	2.79	3.63(6)	168.4
O(15)–H(15A)···O(8)#7	0.85	2.64	3.06(5)	112.5
O(15)–H(15B)···O(1)#12	0.85	2.89	3.63(5)	146.4

Notes: Symmetry transformations used to generate equivalent atoms: #3 $-x+1$, $-y+1$, $-z+1$; #7 $-x+1$, $y+1/2$, $-z+3/2$; #8 $x+1$, $-y+3/2$, $z+1/2$; #9 $-x$, $-y+1$, $-z+1$; #10 x, $y+1$, z; #11 $-x$, $y-1/2$, $-z+1/2$; #12 x, $-y+3/2$, $z+1/2$.

does not have obvious limits because they are kept in the channels by H-bonds with the framework [16]. Upon heating, the release of organic amines is incomplete and is followed immediately by framework decomposition. At 500 °C, the residue (39.9%) was probably the mixture of CdO (Calcd 19.1%) and carbon, then the carbon was lost and the line falls very slowly [17].

3.3. *Diffuse reflectance spectrum*

The solid-state UV–vis spectra of 5,5′,5″,5‴-[1,2,4,5-phenyltetramethoxy]tetraisophthalate (**L**) and **1** are shown in figure 3(a), which show that these compounds have absorption onset at 367 and 410 nm for **L** and **1** [18], respectively. The band is slightly red-shifted upon coordination. Considering that semiconductive MOF-5 with a band gap of about 3.4 eV has been proven by density functional theory calculations and experimental measurements using photoluminescence spectroscopy and UV–vis spectroscopy [19], we calculated the band gap of **1** through Kubelka–Munck function at 3.64 eV [see figure 3(b)].

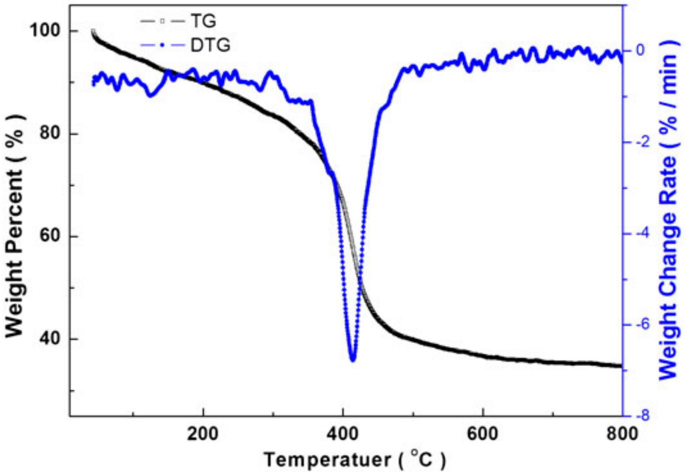


Figure 2. The TGA (black line) and DTG (blue line) plots of **1** (see http://dx.doi.org/10.1080/00958972.2015.1031655 for color version).

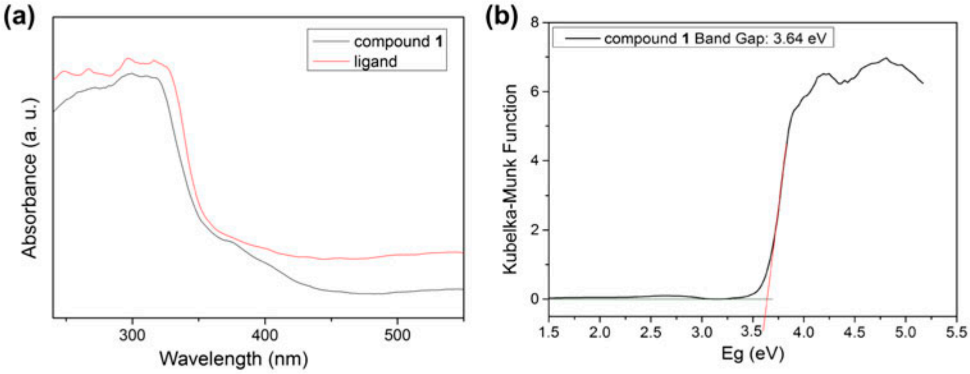


Figure 3. (a) The solid-state UV–vis absorption spectra of **L** and **1** at ambient temperature. (b) The solid-state diffuse reflectance of **1**.

3.4. *Luminescent properties*

The photoluminescent properties of **L** and **1** in the solid state were investigated at ambient temperature (see figure 4). Upon excitation at 296 nm, **L** and **1** exhibited photoluminescence with main emission maxima at 399, 465 and 395, 465 nm, respectively. The emission peaks of **1** are almost the same as that of free **L**, which may be attributed to the coordination of **L** [20]. Increase in the peak height may be attributed to the coordination effect of the ligands to Cd ions, which increases the ligand conformational rigidity and reduces the nonradiative decay [21].

3.5. *Description of the structure of 1*

Single-crystal X-ray diffraction analyses reveal that **1** crystallizes in the monoclinic space group $P2_1/c$. The asymmetric unit [see figure 5(a)] contains one Cd ion, a half of **L**, five

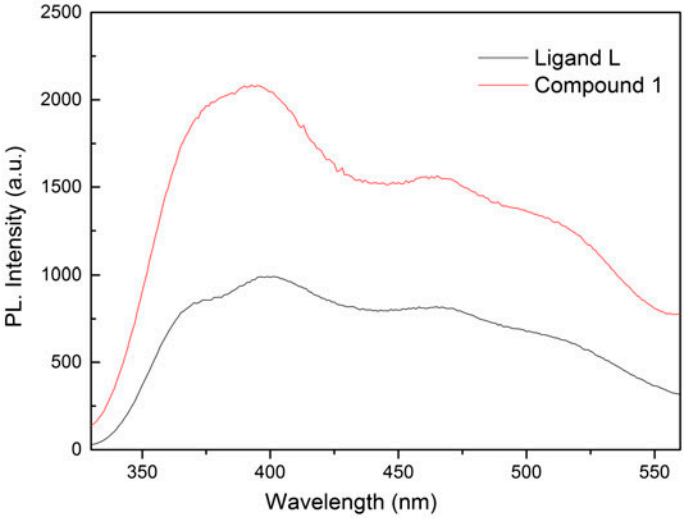


Figure 4. The solid-state emission spectra of **L** and **1** at ambient temperature.

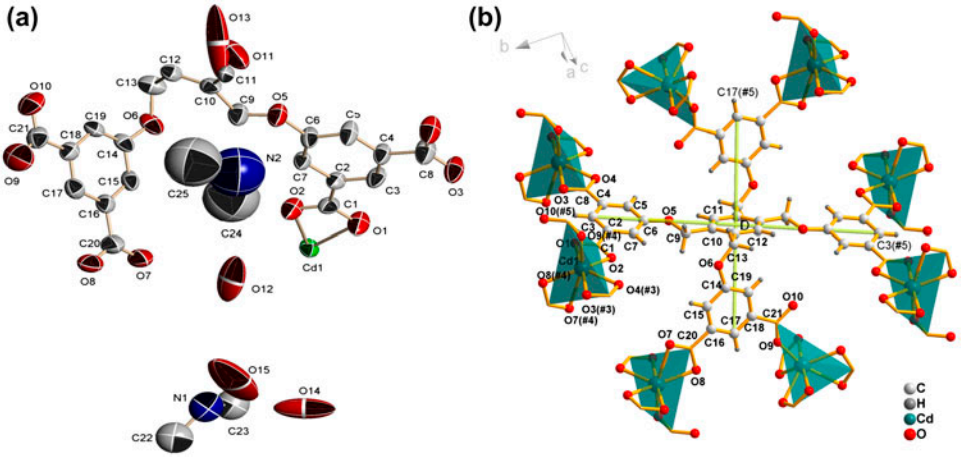


Figure 5. (a) The asymmetric unit of **1**. Displacement ellipsoids are drawn at the 50% probability level. Hydrogens are omitted for clarity. (b) The coordination environment of **L** and Cd1 in **1** [#3 $-x + 1, -y + 1, -z + 1$; #4 $x, -y + 1/2, z - 1/2$; #5 $x + 1, y, z$].

waters, and two dimethylammonium cations as a consequence of DMF hydrolysis [22]. As shown in figure 5(b), Cd1 adopts a distorted tetrahedron Cd[$(COO)_3O$] coordination geometry (the center is Cd1 and the four vertices are occupied by carbons from the four coordinated carboxylate groups) with O–Cd–O angles ranging from 51.9(2) to 175.5(2)°. The edge lengths of Cd1 centered distorted tetrahedra range from 4.1347(1) to 5.0905(1) Å. Cd1 is coordinated by four carboxylates from four different **L** with three in chelating bidentate coordination and one monodentate. The Cd1–O9(#4) distance in the monodentate coordination is 2.148(1) Å, shorter than those in chelating bidentate coordination with distance ranging from 2.216(7) to 2.546(7) Å (see table 2). These Cd–O distances are comparable with those in reported Cd(II)-carboxylic complexes [16, 10(b), 10(c)]. The **L** is coordinated to eight Cd ions with its eight carboxylates, six in chelating bidentate modes, and two monodentate. The center (marked as D) of the central benzene ring is a symmetric center of **L**. **L** can be considered as a rhomboid in which the angle C3–D–C17 is 89.7(8)°, the side lengths are 9.65(9) and 9.69(8) Å, the diagonals (C3–D–C3(#4) and C17–D–C17 (#4)) are 15.38(8) and 11.72(8) Å, respectively. This geometry of **L** is different than the reported work [12].

The 3D framework of **1** [see figure 6(a)] is constructed from the four-connected Cd ions and the eight-connected **L**. Within the 3D structure, channels running along the crystallographic [2 0 1] vertical direction have maximum diameter of the opening sizes of ca. 3.22 Å. The rhomboid channels run along the crystallographic *a*, *b* direction with maximum diagonal distance of the opening sizes of ca. 6.32(1) × 5.78(1) Å^2 and ca. 5.18(1) × 2.46(1) Å^2, respectively. In these channels, there are guest water molecules and dimethylammonium cations held with H-bonds (see figure 7; the H-bond geometry is shown in table 3). The stability of the framework has been benefited from those H-bonds. PLATON [23] calculations show that the total solvent-accessible volume is 1800.7 Å^3 per unit cell, comprising 52.7% of the total crystal volume. The solvent-accessible surface area estimated on the basis of the crystal structure is 2058 m^2 g^{-1} (using a spherical probe of 2.8 Å in diameter). These calculations show the porous nature of **1**.

From a topological point of view, **1** can be constructed from 4-connected Cd1 ions and 8-connected **L**. Each Cd1 is linked by four **L** and each **L** is linked by eight Cd ions. This gives a binodal (4,8)-connected topology [figure 6(b)]. The 4-connected Cd1 nodes have the point symbol of $\{4^5{\cdot}6\}$. The **L** node has the point symbol of $\{4^{10}{\cdot}6^{14}{\cdot}8^4\}$. The network of **1** can thus be topologically represented as a binodal (4,8) net with stoichiometry of $\{4^{10}{\cdot}6^{14}{\cdot}8^4\}\{4^5{\cdot}6\}_2$ [24].

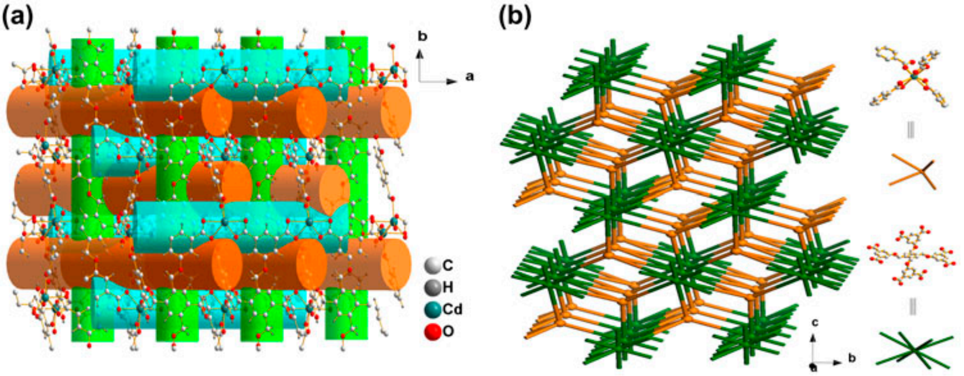


Figure 6. (a) The ball-and-stick views and channels in direction *a* (in brown color), *b* (in green color), and [2 0 1] (in light blue color) of the microporous framework of **1**. (b) Topological representations of **1** with stoichiometry of $\{4^{10}{\cdot}6^{14}{\cdot}8^4\}\{4^5{\cdot}6\}_2$. (see http://dx.doi.org/10.1080/00958972.2015.1031655 for color version).

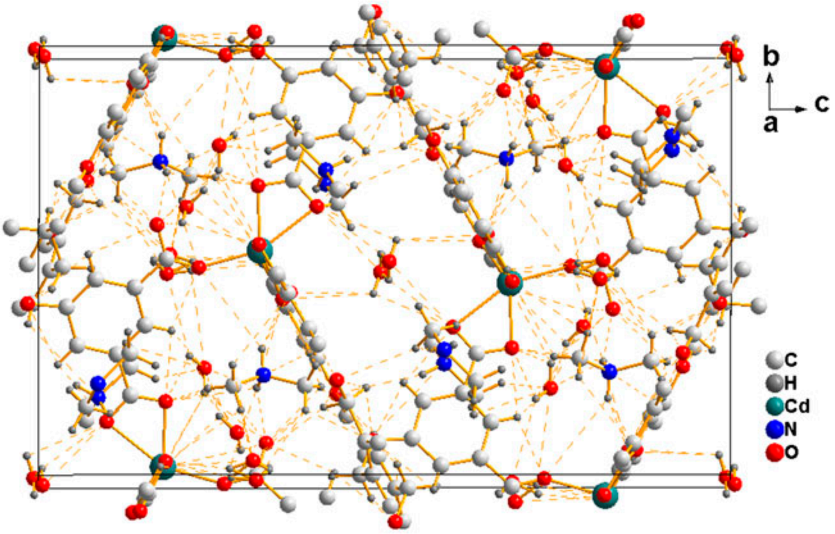


Figure 7. The H-bond (dotted line) interaction between the framework, water molecules, and dimethylammonium cations in the unit cell.

4. Conclusion

A MOF assembled by octacarboxylate **L** and Cd(II) has been synthesized and characterized. It provides an excellent MOF structure to evaluate the impact of different metal ions based on **L**. The solid diffuse reflectance result shows the presence of an optical gap of 3.64 eV, which suggests that **1** may have semiconductor property. Further work for preparing new MOFs based on **L** and other metal ions is in progress.

Supplementary material

CCDC number reference number: 1026521. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: + 44-1223-336-033; Email: deposit@ccdc.cam.ac.uk).

Disclosure statement

No potential conflict of interest was reported by the authors.

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