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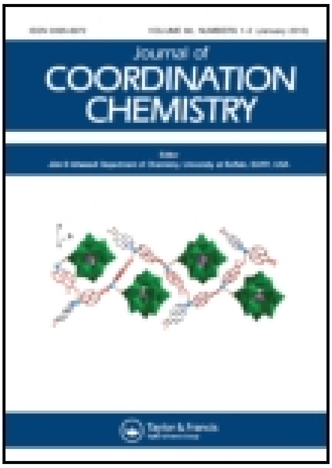

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Syntheses, crystal structures, and photoluminescence of two Cd(II) complexes with simple ligands

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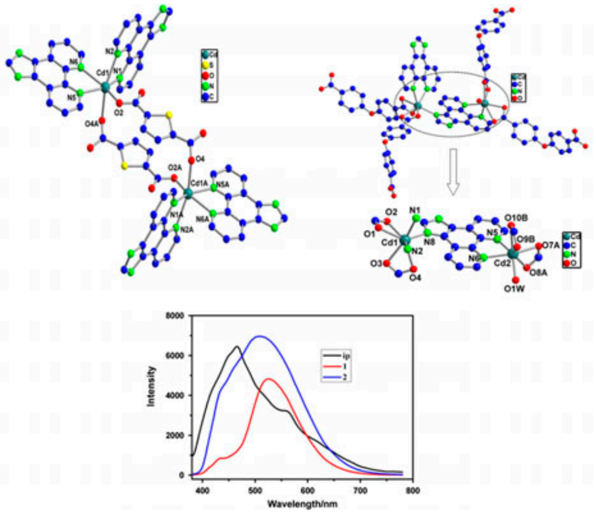

Two cadmium complexes, $\{[Cd_2(2,5\text{-tda})_2(ip)_4]\cdot 4H_2O\}_n$ (**1**) and $\{[Cd_2(4,4'\text{-obb})_2(ip)_2\cdot H_2O]\cdot H_2O\}_n$ (**2**) (2,5-tda = thiophene-2,5-dicarboxylic acid, 4,4′-obb = 4,4′-oxybisbenzoic acid, ip = 1H-imidazo [4,5-f][1, 10]-phenanthroline), were synthesized and characterized by IR, elemental analysis, powder X-ray diffraction, and single-crystal X-ray diffraction. X-ray analysis revealed that **1** is a dinuclear complex with the 2,5-tda anion connecting two Cd ions in a μ_1-η^1:η^0/μ_1-η^1:η^0 coordination mode. Each dinuclear complex is further connected with neighboring complexes via hydrogen-bonding interactions. Compound **2** displays a 2-D layer structure with opened windows occupied by crystallographic water molecules. The layers are further packed via hydrogen-bonding interactions. Luminescent properties for **1** and **2** are also investigated in the solid state at room temperature.

Keywords: Cadmium complexes; Crystal structures; Photoluminescence

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1. Introduction

Design and construction of functional metal–organic coordination polymers is of considerable interest due to diverse topologies and potential applications in gas storage, magnetism, catalysis, and fluorescence [1–5]. Metal–organic complexes with 0-D, 1-D, 2-D, or 3-D inorganic networks and interesting properties are synthesized through molecular self-assembly [6, 7]. The construction of coordination polymers is affected by the selection of appropriate multidentate ligand, metal ions, pH, temperature, solvent, etc. [8, 9]. It is a challenge to design and construct coordination polymers in crystal engineering because of the difficult prediction of either the composition or the structure of the reaction product.

Complexes constructed by ligands, especially those with d^{10} metal centers, have been investigated for fluorescent properties and potential applications as fluorescent materials, such as light-emitting diodes [10]. Ligands with heteroaromatic rings which can enhance the fluorescence of coordination polymers are of interest in the construction of complexes [11]. As a 1,10-phenanthroline derivative, 1H-imidazo[4,5-f][1, 10]-phenanthroline, which contains an extended π-system and potential hydrogen groups involving heteroaromatic rings, is a good candidate for construction of metal–organic supramolecular architecture with good luminescence. The use of both rigid and flexible multicarboxylate ligands form diverse networks [12]. As a result, our effort has been devoted to the construction of complexes using rigid and flexible multicarboxylate ligands with imidazo[4,5-f][1, 10]-phenanthroline. In this article, we use thiophene-2,5-dicarboxylic as the rigid carboxylate ligand, 4,4′-oxybisbenzoic acid as the flexible multicarboxylate ligand, and 1H-imidazo[4,5-f] [1, 10]-phenanthroline as a N-donor to construct $\{[Cd_2(2,5\text{-tda})_2(ip)_4]\cdot 4H_2O\}_n$ (**1**) and $\{[Cd_2(4,4'\text{-obb})_2(ip)_2\cdot H_2O]\cdot H_2O\}_n$ (**2**).

2. Experimental

2.1. *General material and method*

The starting materials used in this work were commercially purchased and used without purification. Elemental analyses for C, H, and N were performed on an Elementar Vario Micro Cube analyzer. The IR spectra were recorded on a Thermo Nicolet IR200 FT-IR spectrometer as KBr pellets (4000–400 cm^{-1}). Powder X-ray diffraction (PXRD) analysis was performed on a MiniFlex2 X-ray Diffractometer Cu-Kα radiation ($\lambda = 0.1542$ nm) in the 2θ range of 5°–50° with a scanning rate of 5° min^{-1}. Thermal stability studies were carried out on a NETSCHZ STA-449C thermoanalyzer with a heating rate of 10 °C min^{-1} under a N_2 gas flow. The fluorescent spectra were obtained on a Hitachi F-4600 spectrofluorometer.

2.2. *Synthesis of* $\{[Cd_2(2,5\text{-tda})_2(ip)_4]\cdot 4H_2O\}_n$ (**1**)

Hydrothermal reaction of $Cd(NO_3)_2\cdot 4H_2O$ (0.0308 g, 0.1 mM), H_2tda (0.0172 g, 0.1 mM), NaOH (0.008 g, 0.2 mM), ip (0.0220 g, 0.1 mM), and distilled water (8 mL) was heated to 160 °C for 72 h in a 25-mL stainless steel reactor with a Teflon liner, followed by slow cooling to room temperature. The resulting reaction products were orange block crystals that were washed with distilled water to give pure samples (Yield: 52% based on Cd). Elemental analyses Calcd for $C_{64}H_{44}Cd_2N_{16}O_{12}S_2$ (1518.07): C, 50.63; H, 2.92; N, 14.76%. Found: C, 50.49; H, 2.78; N, 14.70%. IR data (KBr pellet): 3432m, 2919m, 2802m, 1608m, 1544s, 1425m, 1384s, 1354s, 1245w, 1139m, 1075s, 933m, 824s, 736s, 637s, 542s.

2.3. *Synthesis of* $\{[Cd_2(4,4'\text{-}obb)_2(ip)_2\cdot H_2O]\cdot H_2O\}_n$ (**2**)

Compound **2** was obtained by hydrothermal procedure as for preparation of **1** only using 4,4′-obb (0.1 mM) instead of 2,5-tda. Orange block crystals of **2** were collected in 46% yield based on Cd after wash by distilled water several times. Elemental analyses Calcd for $C_{54}H_{36}Cd_2N_8O_{12}$ (1213.71): C, 53.44; H, 2.99; N, 9.23%. Found: C, 53.19; H, 2.87; N, 9.11%. IR data (KBr pellet): 3403m, 2974m, 1596s, 1546s, 1406s, 1301m, 1249m, 1160s, 1096m, 1076w, 952w, 880s, 809m, 733s, 639s.

2.4. *X-ray crystallography*

Diffraction intensity data of the single crystals of **1** and **2** were collected on a Bruker SMART APEXII CCD diffractometer equipped with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) using a ω-scan mode. Empirical absorption correction was applied using SADABS [13]. All the structures were solved by direct methods and refined by full-matrix least-squares on F^2 using SHEXL 97 [14]. The single suite WINGX was used as an integrated system for all the crystallographic programs [15]. All non-hydrogen atoms were refined anisotropically. Hydrogens were located by geometric calculations, and their positions and thermal parameters were fixed during the structure refinement. The crystallographic data are summarized in table 1. The data of relevant bond lengths and angles are listed in table 2.

Table 1. Summary of crystallographic data for **1** and **2**.

Complex	**1**	**2**
Formula	$C_{64}H_{44}Cd_2N_{16}O_{12}S_2$	$C_{54}H_{36}Cd_2N_8O_{12}$
Fw	1518.07	1213.71
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/c$	$P\text{-}1$
a (Å)	11.688(5)	12.445(3)
b (Å)	17.433(5)	14.957(3)
c (Å)	14.497(5)	15.857(4)
β (°)	102.998(5)	72.822(10)
Volume (Å^3)	2878.2(18)	2466.9(10)
Z	2	2
Density (calculated)	1.752	1.634
Abs. coeff. (mm^{-1})	0.896	0.936
Total reflections	24,763	21,642
Unique reflections	6577	11,160
Goodness-of-fit on F^2	1.066	0.896
Final R indices [$I > 2\sigma(I2)$]	$R1 = 0.0863$, $wR2 = 0.2330$	$R1 = 0.0660$, $wR2 = 0.1816$
R (all data)	$R1 = 0.0889$, $wR2 = 0.2410$	$R1 = 0.0865$, $wR2 = 0.2312$

$R = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$, $wR = \{\Sigma w[(F_o^2 - F_c^2)^2]/\Sigma w[(F_o^2)^2]\}^{1/2}$.

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

Complex **1**			
Cd(1)–O(2)	2.314(6)	Cd(1)–N(2)	2.369(8)
Cd(1)–O(4)A	2.330(7)	Cd(1)–N(5)	2.363(7)
Cd(1)–N(1)	2.333(7)	Cd(1)–N(6)	2.465(7)
		N(2)–Cd(1)–N(6)	83.3(2)
O(2)–Cd(1)–O(4)A	89.0(2)	O(4)A–Cd(1)–N(2)	155.7(2)
O(2)–Cd(1)–N(1)	122.4(2)	N(1)–Cd(1)–N(2)	71.2(2)
O(4)A–Cd(1)–N(1)	85.8(2)	N(5)–Cd(1)–N(2)	114.8(2)
O(2)–Cd(1)–N(5)	84.9(2)	O(2)–Cd(1)–N(6)	150.2(2)
O(4)A–Cd(1)–N(5)	89.2(2)	O(4)A–Cd(1)–N(6)	103.1(2)
N(1)–Cd(1)–N(5)	152.0(3)	N(1)–Cd(1)–N(6)	86.0(2)
O(2)–Cd(1)–N(2)	96.8(2)	N(5)–Cd(1)–N(6)	68.4(2)
Complex **2**			
Cd(1)–O(1)	2.431(7)	Cd(2)–O(1W)	2.284(7)
Cd(1)–O(2)	2.361(7)	Cd(2)–O(7)A	2.284(6)
Cd(1)–O(3)	2.381(7)	Cd(2)–O(8)A	2.466(8)
Cd(1)–O(4)	2.431(7)	Cd(2)–O(9)B	2.421(6)
Cd(1)–N(8)	2.332(6)	Cd(2)–O(10)B	2.426(7)
N(8)–Cd(1)–O(2)	88.2(2)	O(7)A–Cd(2)–O(1W)	108.8(3)
N(8)–Cd(1)–N(1)	84.9(2)	O(7)A–Cd(2)–N(6)	159.5(2)
O(2)–Cd(1)–N(1)	93.7(2)	O(1W)–Cd(2)–N(6)	86.4(3)
N(8)–Cd(1)–O(3)	119.0(3)	O(7)A–Cd(2)–O(9)B	102.4(2)
O(2)–Cd(1)–O(3)	89.7(2)	O(1W)–Cd(2)–O(9)B	77.5(2)
N(1)–Cd(1)–O(3)	156.0(3)	O(7)A–Cd(2)–O(10)B	88.5(2)
O(2)–Cd(1)–O(4)	128.3(2)	O(1W)–Cd(2)–O(10)B	131.7(2)
O(3)–Cd(1)–O(1)	82.5(3)	O(7)A–Cd(2)–N(5)	91.3(2)
N(8)–Cd(1)–N(2)	127.2(2)	O(1W)–Cd(2)–N(5)	140.2(2)

Note: Symmetry codes: (**1**): (A) $-x+1$, $-y$, $-z+1$; (**2**): (A) $x-1$, y, $z-1$; (B) $x+1$, y, $z-1$.

3. Results and discussion

3.1. *Description of crystal structures*

3.1.1. $\{[Cd_2(2,5\text{-tda})_2(ip)_4]\cdot 4H_2O\}_n$ (1). Structure determination shows that **1** crystallized in the monoclinic $P_{2}1/c$ space group. There are two Cd(II) ions, four ip ligands, two tda ligands, and four free waters in the asymmetric unit. As shown in figure 1(a), Cd(II) is six coordinate by two oxygens from two 2,5-tda ligands [Cd(1)–O(2) = 2.313(6) Å and Cd(1)–O(4A) = 2.331(7) Å] and four nitrogens from two ip ligands [Cd–N bond lengths varying from 2.332(7) to 2.465(7) Å] in a highly distorted octahedral geometry. The Cd–O and Cd–N distances are in agreement with corresponding values in [Cd(tda)(phen)] [16]. Thiophene-2,5-dicarboxylic as a rigid multidentate carboxylate is often used to build complexes because of its variety of coordination models, such as $[Cd(tda)(H_2O)]\cdot 4H_2O$ [17] and $[Zn_4O(tda)_3(MeOH)]\cdot DMF\cdot 4H_2O$ [18]. In our work, we added 1H-imidazo[4,5-f][1, 10]-phenanthroline as a N-donor to construct the complex with thiophene-2,5-dicarboxylic.

In **1**, the carboxylic groups of tda adopt monodentate bridging mode [scheme 1(a)] forming a dinuclear Cd(II) subunit, which is similar to previously reported complexes [19, 20]. N2 and O4A lie axial of the octahedron and the equatorial plane was occupied by O2, N1, N5, and N6. Strong intermolecular hydrogen bonds between the N–H from ip ligands and uncoordinated O from tda ligands are observed (table 3). As a result, the 0-D structure was extended into a 3-D supramolecular network via hydrogen bonds involving the thiophene-2,5-dicarboxylic, imidazo[4,5-f][1, 10]-phenanthroline, and free water molecules [figure 1(b), table 3].

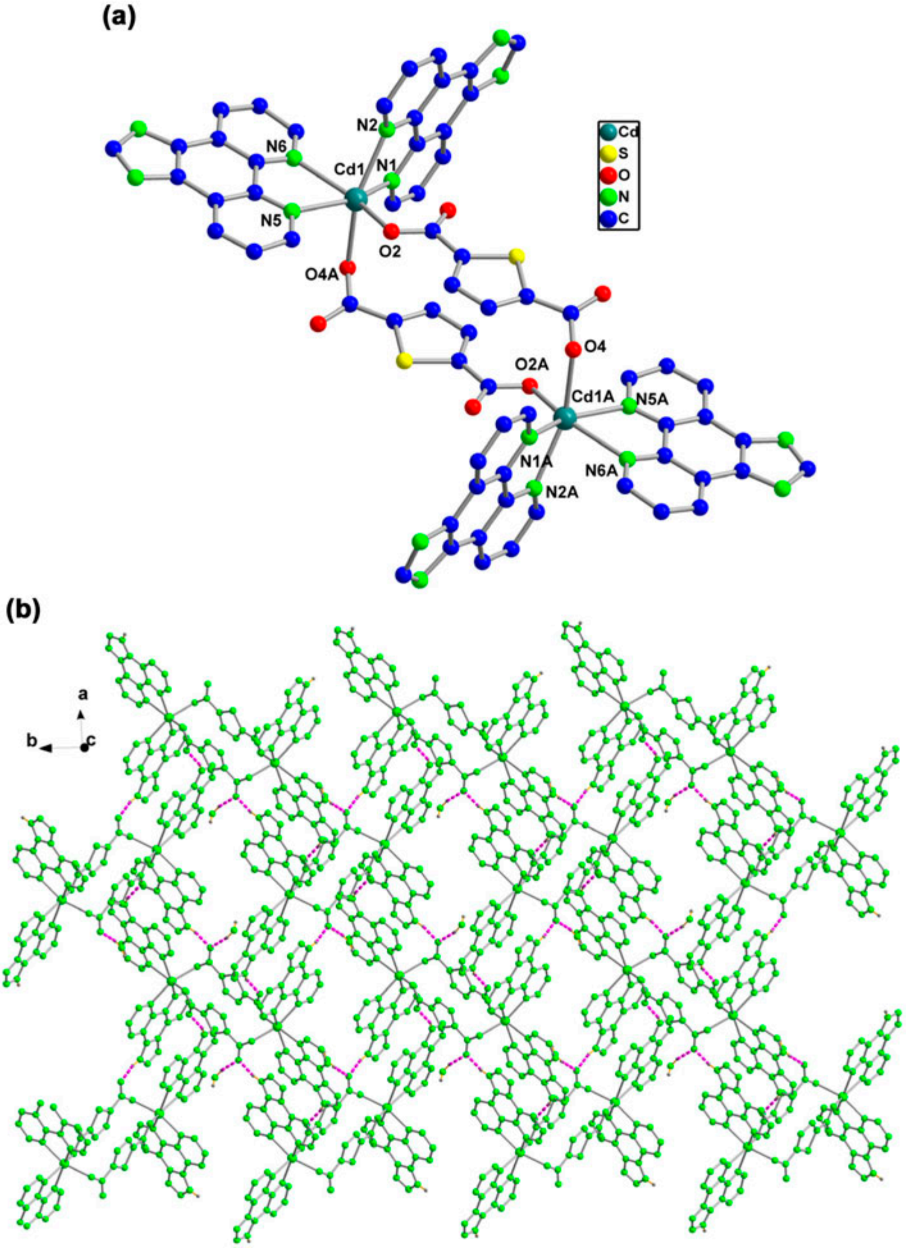


Figure 1. (a) View of the coordination environments of Cd(II) in **1**; hydrogens and guest molecules are omitted for clarity. Symmetry code: (A) −*x* + 1, −*y*, −*z* + 1. (b) The 3-D structure of **1** constructed by hydrogen bonds (the pink dashed lines involving tda, ip, and water, see the online article for the colour version of this figure: http://dx.doi.org/10.1080/00958972.2014.897336).

3.1.2. $\{[Cd_2(4,4'\text{-obb})_2(ip)_2 \cdot H_2O] \cdot H_2O\}_n$ (2). When 2,5-tda was replaced by 4,4′-obb, a totally different structure of **2** was obtained. X-ray crystallography reveals that **2** crystallizes in the triclinic *P-1* space group. As shown in figure 2(a), the asymmetric unit of **2** contains

Table 3. Hydrogen bond lengths (Å) and angles (°) for **1** and **2**.

Complex	D–H···A	d(D–H)	d(H···A)	d(D···A)	∠(DHA)
1	N(4)–H(4A)···O(3)A	0.860	1.949	2.794	167.18
	N(4)–H(4A)···O(4)B	0.860	2.638	3.148	119.16
	N(8)–H(8A)···O(1)C	0.860	1.953	2.759	155.59
	O(1W)–H(1W)···N(7)D	0.852	2.235	2.858	129.98
	O(2W)–H(4W)···S(1)E	0.852	2.034	2.851	160.25
	O(2W)–H(4W)···O(3)F	0.852	2.879	3.404	121.57
2	O(2W)–H(4W)···O(3)	0.85	1.93	2.755(11)	163.1
	O(2W)–H(3W)···O(7)A	0.85	2.07	2.865(11)	154.1
	O(1W)–H(1W)···O(9)B	0.85	1.99	2.789(9)	155.4
	O(1W)–H(2W)···N(3)A	0.85	1.94	2.732(10)	153.3
	N(4)–H(4)···O(1)C	0.86	1.86	2.677(9)	157.8
	N(7)–H(7)···O(2W)D	0.86	2.54	3.018(10)	115.7
	N(7)–H(7)···O(10)E	0.86	2.06	2.854(9)	153.3

Note: Symmetry codes: (**1**): (A) $-x-1, y-1/2, -z+1/2$; (B) $-x-1, y-1/2, -z+1/2$; (C) $x+1, -y+3/2, z+1/2$; (D) $-x+1, y+1/2, -z+1/2$; (E) $x+1, y, z$ (F) $x+1, y, z$; (**2**): (A) $x-1, y, z$; (B) $-x, -y+1, -z+1$; (C) $-x+2, -y+1, -z+1$; (D) $-x+1, -y+2, -z+1$; (E) $-x, -y+2, -z+1$.

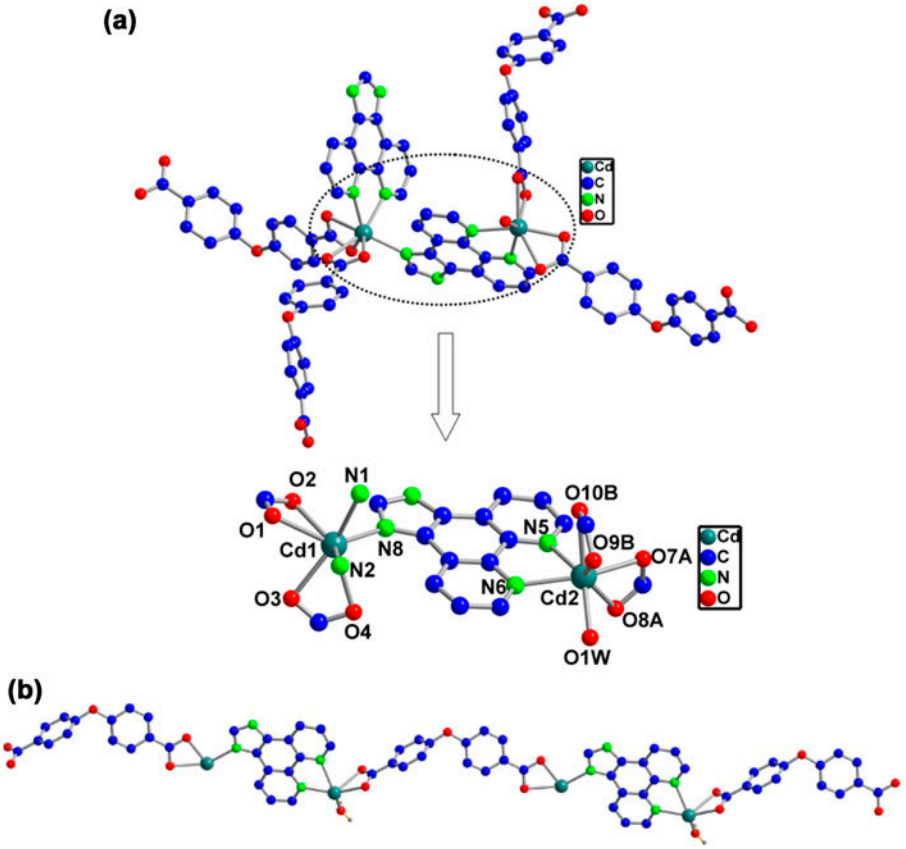


Figure 2. (a) View of the coordination environments of Cd(II) in **2**; hydrogens and guest molecules are omitted for clarity. Symmetry codes: (A) $x-1, y, z-1$; (B) $x+1, y, z-1$. (b) The 1-D chain of **2**. (c) The 2-D layer of **2**. (d) The 3-D structure of **2** constructed by hydrogen bonds (the pink dashed lines involving obb, ip, and water, see the online article for the colour version of this figure: http://dx.doi.org/10.1080/00958972.2014.897336).

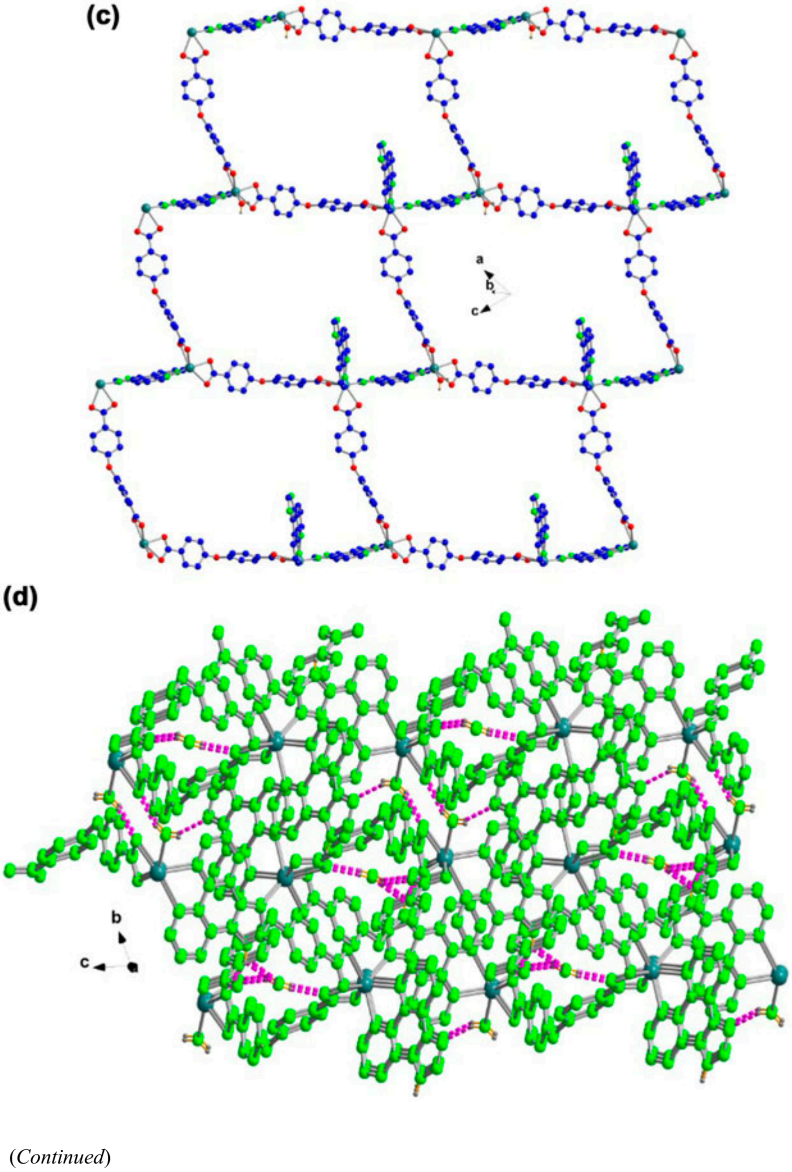
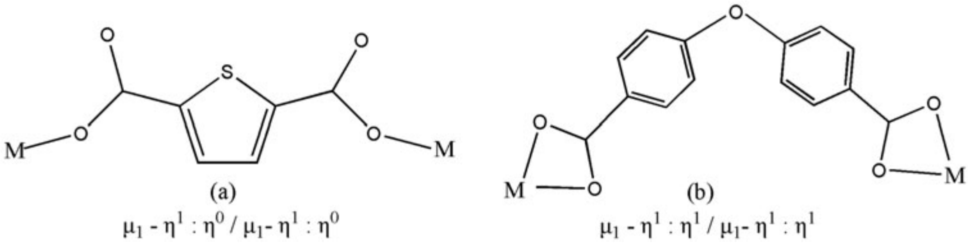


Figure 2. (*Continued*)

two crystallographically independent Cd(II) ions (Cd1 and Cd2), two 4,4′-obb, two ip, one coordinated water, and one free water. Both Cd1 and Cd2 adopt highly distorted pentagonal bipyramidal coordination geometry. Cd1(II) is coordinated by four oxygens from two 4,4′-obb ligands and three nitrogens from two ip ligands, while Cd2(II) is surrounded by four oxygens from two carboxylates of two 4,4′-obb ligands, one coordinated water, and two nitrogens from one ip. The Cd–O and Cd–N distances are 2.284(6)–2.466(8) Å, well within the normal range of Cd–O and Cd–N previously reported in similar compounds [21, 22]. The carboxyls in **2** adopt chelate [scheme 1(b)]. Different from **1**, the dinuclear Cd (II) is formed by connection of the ip. Cd(II) centers are connected by alternate ip and



(a) $\mu_1 - \eta^1 : \eta^0 / \mu_1 - \eta^1 : \eta^0$

(b) $\mu_1 - \eta^1 : \eta^1 / \mu_1 - \eta^1 : \eta^1$

Scheme 1. Coordination of 2,5-tda and 4,4′-obb anions.

4,4′-obb ligands into a 1-D chain [figure 2(b)], which is different from the reported complex [23]. The neighboring 1-D inorganic chains are extended into a 2-D layer framework by 4,4′-obb ligands with one carboxyl connecting Cd1, while the other connects Cd2 of the neighboring chain [figure 2(c)]. In addition, the free waters are connected by hydrogen bonds, so the 2-D layer was extended into a 3-D supramolecular network via O–H···O, O–H···N and N–H···O hydrogen bonds involving 4,4′-obb, ip, and water [figure 2(d), table 3].

3.2. *Thermogravimetric analysis and photoluminescence properties*

To confirm the phase of the bulk materials, PXRD experiments were carried out on **1** and **2** (figure S1, see online supplemental material at http://dx.doi.org/10.1080/00958972.2014.897336). As shown in figure S1, experimental PXRD data match well with simulated powder pattern, it can be considered that the as-synthesized materials are homogeneous.

The decomposition of **1** and **2** was examined via thermogravimetric analysis (TGA) (figure S2). For **1**, first weight loss of 4.5% at 30–270 °C corresponds to loss of water (Calcd 4.7%). For **2**, the TGA reveals weight loss (found: 2.76%) from ca. 30 to 362 °C, corresponding to loss of free and coordinated water molecules (Calcd: 2.96%).

Solid-state luminescent emission spectra of **1** and **2** were studied at room temperature. The emission spectra of **1** and **2** are depicted in figure 3. Excitation of the microcrystalline

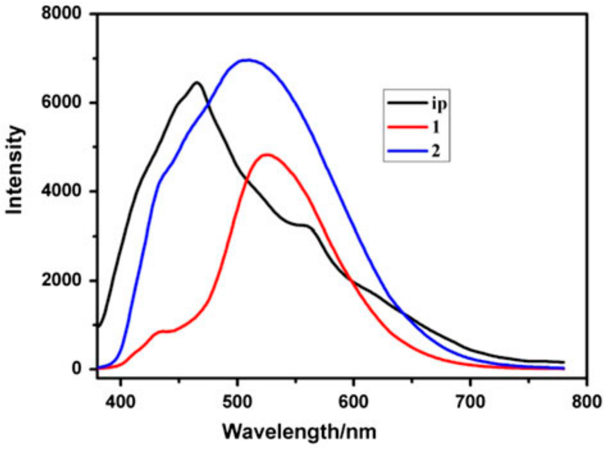


Figure 3. The photoluminescence of **1**, **2**, and free ip in the solid state.

samples at 350 nm leads to fluorescent emissions, with peak maxima occurring at 524 nm for **1** and 508 nm for **2**. To further understand the origin of these emissions, the fluorescent spectrum of ip has also been measured, showing an emission band at 465 with excitation at 315 nm which can be attributed to π^*–π transition of the π-electrons of the aromatic rings. The fluorescent emission of the carboxylate deriving from the π^*–n transition is much weaker than that of the π^*–π transition of the aromatic ligands, thus the carboxylate ligands have almost no contribution to the fluorescent emission of the complexes [24]. Compared with the luminescence of ip, the emissions of **1** ($\lambda_{em} = 524$ nm, $\lambda_{ex} = 350$ nm) and **2** ($\lambda_{em} = 508$ nm, $\lambda_{ex} = 350$ nm) are neither metal-to-ligand charge transfer nor ligand-to-metal charge transfer since the Cd^{2+} is difficult to oxidize or reduce due to its d^{10} configuration. Emission bands of **1** and **2** are located in red-shift positions compared to free ip, which can be assigned to the π^*–π transitions, whereas the significant red-shift of the band in comparison to that of ip would be attributed to metal–ligand coordination and its topological structure [25]. Besides the emission band at 465 nm (free ip), a weak blue-shift of a 34 nm shoulder at 431 nm for **1** and **2** is observed. Taking the crystal structures of **1** and **2** into consideration, the blue-shift band of 431 nm is probably ascribed to the weak edge π-interactions between neighboring ip molecules [26]. The difference of the emission peak intensity for **1** and **2** mainly derives from the differences of the coordination environments of the metal ions and the rigidity of solid-state crystal packing.

4. Conclusion

A number of Cd complexes with dicarboxylates have been reported as chelating or monodentate [27–29]. We have synthesized two new complexes with Cd^{2+} under hydrothermal conditions using thiophene-2,5-dicarboxylic, 4,4′-oxybisbenzoic acid, and 1H-imidazo[4,5-f][1, 10]-phenanthroline. Complexes **1** and **2** showed variety in coordination with dicarboxylates. Compound **1** shows 0-D structure with tda anions connecting Cd^{2+} centers forming a dinuclear structure. Compound **2** shows 2-D structure with opened windows. The Cd ions in **1** and **2** exhibited different geometries of the coordination environment. The Cd in **1** displayed a highly distorted octahedral geometry, Cd in **2** adopt highly distorted pentagonal bipyramidal coordination geometry, in agreement with reported Cd(II) complexes [30, 31]. The preparation of **1** and **2** provides a valuable approach for construction of many other complexes from dicarboxylate and N-donor ligands. Solid-state luminescent spectra demonstrate that **1** and **2** are promising candidates for fluorescent materials.

Supplementary material

CCDC Nos. 884510 and 884509 contain the supplementary crystallographic data for this article. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK. Fax: C44 1223336033.

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