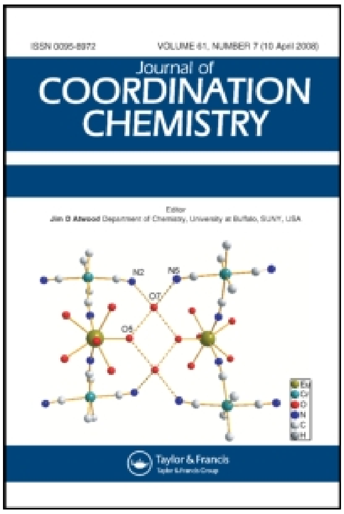

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Coordination polymers constructed from 2-(2-thienyl)imidazo[4,5-*f*]1,10-phenanthroline and isophthalate: synthesis, characterization and luminescent property

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Coordination polymers constructed from 2-(2-thienyl)imidazo[4,5-*f*]1,10-phenanthroline and isophthalate: synthesis, characterization and luminescent property

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Two new coordination polymers $[Cd(TIP)(1,3\text{-bdc})]_n$ (**1**) and $\{[La(TIP)(1,3\text{-bdc})_{1.5}] \cdot 0.2H_2O\}_n$ (**2**) (TIP = 2-(2-thienyl)imidazo[4,5-*f*]1,10-phenanthroline and 1,3-H_2bdc = isophthalic acid) were synthesized from hydrothermal reactions and characterized by elemental analyses, IR, and single-crystal X-ray diffraction. Complexes **1** and **2** are ribbon structures with different configurations bridged by 1,3-bdc ligand, further extended into 3-D supramolecular structures by π–π stacking and hydrogen bonding interactions. The most interesting feature of these two complexes is that TIP with the large π-system and additional hydrogen bonding group plays a crucial role in the formation of different weak interactions. Thermal properties and fluorescence of **1** and **2** are also investigated in the solid state.

Keywords: Coordination polymer; Chelating ligand; Isophthalate; Crystal structure; Fluorescence

1. Introduction

Design and synthesis of new coordination polymers is focused on the development of functional solid materials [1–3]. The self-assembly process of metal–organic coordination polymers greatly depends on the judicious selection of organic ligands and metal centers [4]. Rational synthesis of these complexes still faces many challenges because of the uncontrollable factors dominating the formation of the final products [5, 6]. Therefore, many new synthetic strategies have been applied in which the mixed-ligand approach has been proven to be one of the most effective and important approaches [7–9]. Organic ligands with hetero-aromatic rings can effectively enhance the fluorescence emissions of coordination polymers and are currently of interest in the development of fluorescent materials [10].

An important aspect of crystal engineering is to understand and attempt to control the manner in which molecules are arranged in crystal lattices through noncovalent

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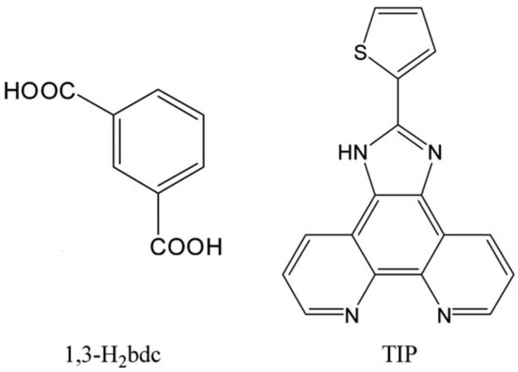
interactions [11]. One approach to forming high-dimensional networks of discrete or low-dimensional complexes is to use a chelating ligand that has additional functionality attached to its backbone, such as additional coordination sites, hydrogen bonding groups, or extended π-system [12]. As an important 1,10-phenanthroline (phen) derivative, 2-(2-thienyl)imidazo[4,5-*f*]1,10-phenanthroline (TIP) (scheme 1) contains an extended π-system and potential hydrogen bonding groups involving two hetero-aromatic rings. It is a good candidate for the construction of metal–organic supramolecular architecture [13]. Multi-carboxylates as the bridging ligands are of interest in the construction of polymeric coordination architectures with structural diversities and its potential applications as functional materials [14].

In this work, the assembly of $CdCl_2$ or $La(NO_3)_3 \cdot 6H_2O$, TIP, and isophthalic acid (1,3-H_2bdc) under hydrothermal conditions generates two new 1-D coordination polymers, $[Cd(TIP)(1,3\text{-bdc})]_n$ (**1**) and $\{[La(TIP)(1,3\text{-bdc})_{1.5}] \cdot 0.2H_2O\}_n$ (**2**). Both structures are extended into 3-D supramolecular frameworks by weak interactions. On the basis of synthesis and structural characterization, the role of weak intermolecular forces in the creation of molecular architectures is discussed. Moreover, the thermal properties and fluorescence of **1** and **2** are also investigated.

2. Experimental

2.1. *Materials and instrumentation*

All chemicals purchased were of reagent grade and used without purification. TIP [15] was synthesized by literature methods and characterized by FT-IR spectra and ^{1}H NMR. FT-IR spectra (KBr pellets) were taken on a Magna FT-IR 560 spectrometer. Elemental analyses (C, H, and N) were performed on a Perkin-Elmer 240C analyzer. Thermogravimetric data for **1** and **2** were collected on a Pyris Diamond thermal analyzer. Fluorescence spectra were performed on a Hitachi F-4500 fluorescence/phosphorescence spectrophotometer at room temperature.



Scheme 1. Structures of the ligands.

2.2. *Syntheses*

2.2.1. [Cd(TIP)(1,3-bdc)]$_n$ (1). A mixture of $CdCl_2$ (0.018 g, 0.1 mmol), TIP (0.014 g, 0.05 mmol), isophthalic acid (0.017 g, 0.1 mmol), H_2O (8 mL), 0.1 mol L^{-1} NaOH (0.007 g, 1.8 mL) was stirred for 30 min in air, then transferred and sealed in a 25-mL Teflon reactor, which was heated at 160°C for 4 days and then cooled to room temperature. Yellow block crystals of **1** were obtained in 25% yield. $C_{25}H_{14}N_4O_4SCd$ (578.88): Anal. Calcd (%) C, 51.82; H, 2.42; N, 9.67; Found (%): C, 51.97; H, 2.26; N, 9.79. IR (KBr, cm^{-1}): 2365 (w), 1613 (m), 1542 (s), 1486 (m), 1435 (m), 1355 (s), 1076 (m), 800 (m), 782 (m), 709 (s), 638 (m).

2.2.2. {[La(TIP)(1,3-bdc)$_{1.5}$] · 0.2H$_2$O}$_n$ (2). The preparation of **2** was similar to that of **1** except that $La(NO_3)_3 \cdot 6H_2O$ was used instead of $CdCl_2$. Yellow block crystals of **2** were obtained in 30% yield. $C_{29}H_{16.40}N_4O_{6.02}SLa$ (688.15): Anal. Calcd (%) C, 50.57; H, 2.38; N, 8.14; Found (%): C, 50.65; H, 2.32; N, 8.06. IR (KBr, cm^{-1}): 3435 (w), 3070 (w), 2361 (m), 1614 (s), 1569 (m), 1524 (m), 1484 (m), 1400 (s), 1080 (m), 849 (m), 810 (m), 737 (m), 720 (s), 697 (m), 652 (w).

2.3. *X-ray crystallography*

All diffraction data were collected using a Bruker Apex CCD diffractometer (Mo-Kα radiation, graphite monochromator, $\lambda = 0.71073$ Å). The structures were solved by direct methods with *SHELXS-97* and Fourier techniques and refined by full-matrix least-squares on F^2 with *SHELXL-97* [16, 17]. All nonhydrogen atoms were refined anisotropically, hydrogens from nitrogen of imidazole in TIP were located in different Fourier synthesis maps, and other hydrogens of the ligands were generated theoretically onto the specific atoms and refined isotropically with fixed thermal factors. Crystal data and structure refinement details for the two complexes are given in table 1. Relevant bond distances and angles are listed in table 2.

3. Results and discussion

3.1. *Description of crystal structures*

Structural analysis of [Cd(TIP)(1,3-bdc)]$_n$ (**1**): Complex **1** is a ribbon framework with zigzag configuration. The coordination environment of Cd^{II} is six-coordinate, bound by two nitrogens from a chelating TIP with bond distances of 2.364(2) Å [Cd1–N1] and 2.351(2) Å [Cd1–N2], and four oxygens of three 1,3-bdc ligands [the bond lengths of Cd–O range from 2.238(2) Å to 2.395(2) Å] (figure 1). The Cd–N and Cd–O bond lengths lie in normal ranges [18, 19]. Thus, the coordination sphere of Cd^{II} can be described as distorted octahedral.

In **1**, all 1,3-bdc ligands adopt the same bridging coordination mode, chelating mono-bidentate and bridging mono-bidentate (I, scheme 2). Two adjacent Cd^{II} ions are bridged by a pair of 1,3-bdc μ-carboxylates to afford a dinuclear $[Cd_2N_4O_4]$ unit, the intradimer Cd···Cd distance is 3.999 Å. The V-shaped 1,3-bdc anion with

Table 1. Crystal data and structure refinement for **1** and **2**.

Complex	**1**	**2**
Empirical formula	$C_{25}H_{14}N_4O_4SCd$	$C_{29}H_{16.40}N_4O_{6.20}SLa$
Formula weight	578.88	688.15
Crystal system	Monoclinic	Monoclinic
Space group	$C2/c$	$P2_1/m$
Unit cell dimensions (Å, °)		
a	14.9998(11)	9.8669(6)
b	15.6250(11)	18.5627(11)
c	19.0258(13)	15.17 32(9)
α	90	90
β	110.8040(10)	102.115 0(10)
γ	90	90
Volume (Å^3), Z	4168.4(5), 8	2717.2(3), 4
D_{calcd} (mg cm^{-3})	1.845	1.682
Absorption coefficient (mm^{-1})	1.193	1.700
$F(000)$	2304	1358
θ range (°)	$1.95 < \theta < 25.00$	$1.76 < \theta < 25.20$
Reflections collected/unique	10464 / 3667	14145 / 5056
Final R indices $[I > 2\sigma(I)]$	$R_1 = 0.0265$, $wR_2 = 0.0637$	$R_1 = 0.0254$, $wR_2 = 0.0592$
R indices (all data)	$R_1 = 0.0356$, $wR_2 = 0.0669$	$R_1 = 0.0332$, $wR_2 = 0.0624$
Goodness-of-fit	1.017	1.058

$R_1 = \sum \| F_o| - |F_c \| / \sum |F_o|$; $wR_2 = \sum[w(F_o^2 - F_c^2)^2] / \sum[w(F_o^2)^2]^{1/2}$.

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

1			
Cd(1)–O(1)	2.238(2)	Cd(1)–O(2)	2.252(2)
Cd(1)–O(3)	2.395(2)	Cd(1)–O(4)	2.386(2)
Cd(1)–N(1)	2.364(2)	Cd(1)–N(2)	2.351(2)
O(1)–Cd(1)–O(2)	102.60(9)	O(1)–Cd(1)–N(2)	91.72(8)
O(1)–Cd(1)–N(1)	161.18(8)	O(1)–Cd(1)–O(4)	109.93(8)
O(1)–Cd(1)–O(3)	92.86(7)	O(2)–Cd(1)–N(2)	121.98(8)
O(2)–Cd(1)–N(1)	84.28(9)	O(2)–Cd(1)–O(4)	82.95(8)
O(2)–Cd(1)–O(3)	137.33(8)	N(1)–Cd(1)–O(3)	93.67(8)
N(1)–Cd(1)–O(4)	88.15(8)	N(2)–Cd(1)–N(1)	70.00(8)
N(2)–Cd(1)–O(4)	143.21(8)	N(2)–Cd(1)–O(3)	96.60(8)
2			
La(1)–O(1)	2.750(2)	La(1)–O(2)	2.461(2)
La(1)–O(3)	2.491(2)	La(1)–O(4)#1	2.432(2)
La(1)–O(5)	2.456(2)	La(1)–O(6)#1	2.503(2)
La(1)–N(1)	2.672(2)	La(1)–N(2)	2.703(2)
O(2)–La(1)–O(1)	49.66(6)	O(2)–La(1)–O(3)	76.07(7)
O(3)–La(1)–O(1)	69.50(7)	O(5)–La(1)–O(3)	88.42(7)
O(5)–La(1)–O(2)	127.00(7)	O(5)–La(1)–O(1)	77.38(7)
O(2)–La(1)–O(6)#1	91.96(7)	O(5)–La(1)–O(6)#1	132.08(7)
O(4)#1–La(1)–O(1)	149.86(7)	O(4)#1–La(1)–O(2)	128.26(8)
O(4)#1–La(1)–O(3)	76.40(7)	O(4)#1–La(1)–O(5)	138.98(7)
O(2)–La(1)–N(2)	68.21(7)	O(4)#1–La(1)–N(1)	82.38(8)
O(5)–La(1)–N(1)	82.07(7)	O(2)–La(1)–N(1)	82.77(8)
O(6)#1–La(1)–N(1)	135.56(7)	O(3)–La(1)–N(1)	144.67(7)
O(4)#1–La(1)–N(2)	81.65(8)	O(6)#1–La(1)–N(2)	76.23(7)
O(3)–La(1)–N(2)	131.87(8)	N(1)–La(1)–N(2)	60.80(7)
N(1)–La(1)–O(1)	75.22(7)	N(2)–La(1)–O(1)	106.66(7)
O(4)#1–La(1)–O(6)#1	80.68(7)	O(6)#1–La(1)–O(1)	131.68(7)
O(3)–La(1)–O(6)#1	73.82(7)		

Symmetry transformations used to generate equivalent atoms: **2**: #1 $-x$, $-y$, $2-z$.

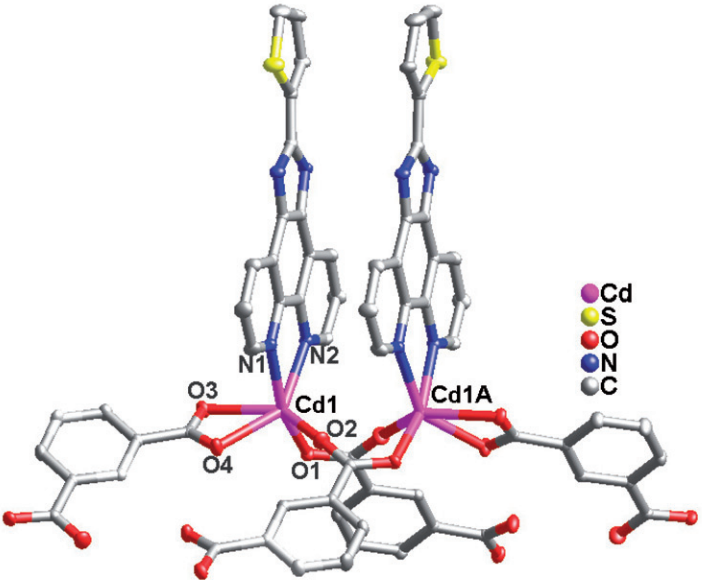
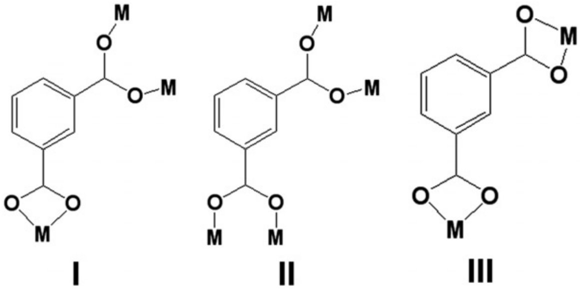


Figure 1. The coordination environment for Cd^{II} in **1** (at 30% probability level).



Scheme 2. Coordination modes of 1,3-bdc ligand.

chelate-bidentate coordination further links the adjacent Cd^{II} ions to a 1-D chain based on a double-strand helix that has a 9.866 Å pitch (figure 2). Three types of π–π interactions are observed in **1**, π–π interaction between pyridine ring and pyridine ring with face-to-face distance of 3.603 Å, π–π interaction between pyridine ring and benzene ring with face-to-face distance of 3.835 Å, and π–π interaction between pyridine ring and thiofuran ring with face-to-face distance of 3.898 Å. Moreover, TIP is a hydrogen bond donor to the coordinated oxygen, O3, of 1,3-bdc ligand as one kind of hydrogen bonding interaction. Meanwhile, a R_2^2 (28) hydrogen bonding ring involving N–H$\cdots$O (N4–H4A$\cdots$O3 $(-1/2-x,\ 1/2+y,\ -1/2-z) = 2.05$ Å, N$\cdots$O $= 2.838$ Å) is obtained. Finally, the chain structure of **1** was extended into a 3-D supramolecular network by weak interactions (figure 3). Weak noncovalent interactions play an important role in the formation of the final supramolecular structure of **1**.

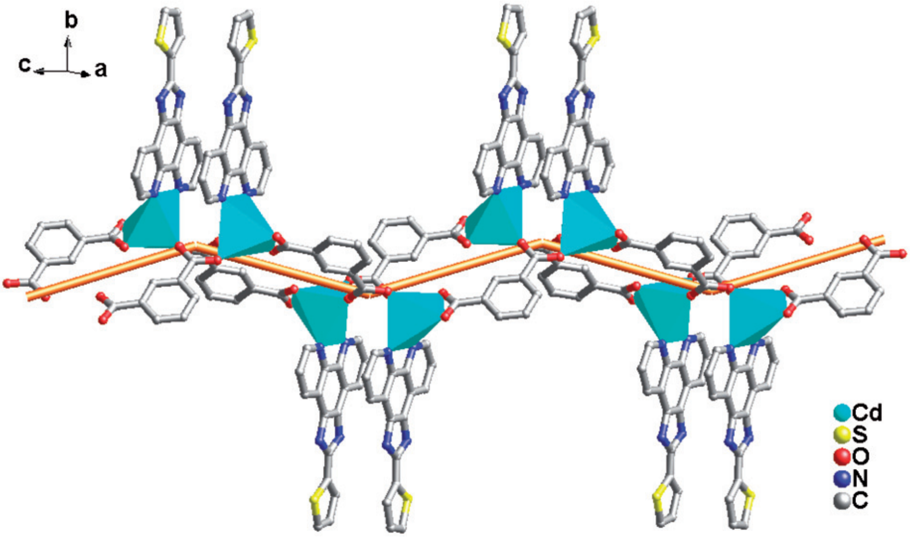


Figure 2. View of the 1-D ribbon structure with the zigzag configuration in **1**.

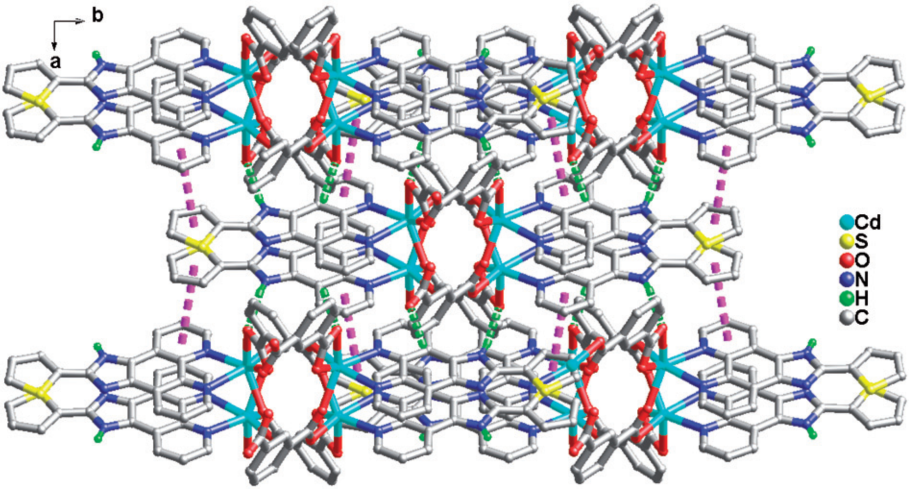


Figure 3. View of the 3-D supramolecular framework of **1** formed through weak interactions (purple broken lines represent π–π stacking interactions; green broken lines represent H-bonding interactions).

Structural analysis of $\{[La(TIP)(1,3\text{-bdc})_{1.5}] \cdot 0.2H_2O\}_n$ (**2**): Complex **2**, obtained by using $La(NO_3)_3 \cdot 6H_2O$, is also a 1-D ribbon with the line configuration based on dimetal $[La_2N_4O_{12}]$ units with separations of La···La of 4.147 Å (figure 4). Obviously, the M···M distance in **2** is longer than that in **1**. Figure 5 illustrates the coordination environments of La^{III}. Each La^{III} is coordinated by two nitrogens of TIP (La1–N1, 2.672(2) Å; La1–N2, 2.703(2) Å) and six carboxylic oxygens of five different 1,3-bdc

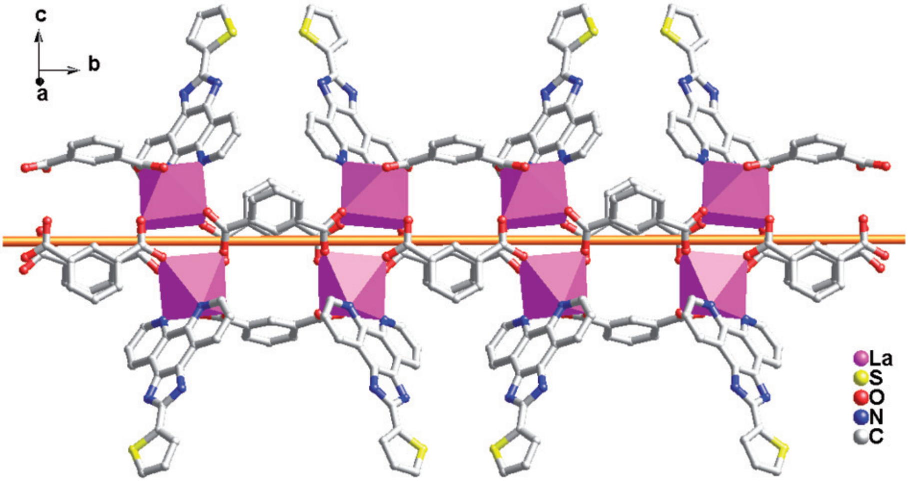


Figure 4. View of the 1-D ribbon structure with the line configuration in **2**.

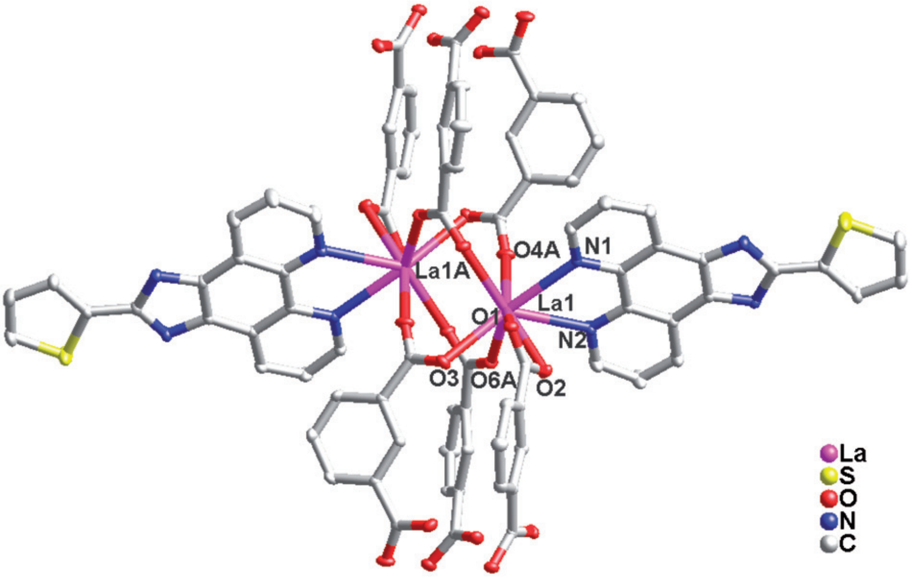


Figure 5. The coordination environment for La^{III} in **2** (at 30% probability level).

ligands with La–O bond lengths ranging from 2.432(2) to 2.750(2) Å in a distorted tetragonal prism environment. There are disordered uncoordinated water molecules in **2**.

Unlike **1**, the 1,3-bdc ligands in **2** exhibit two kinds of coordination modes, bridging bis-bidentate and chelating bis-bidentate (II and III, scheme 2), comparable to those in previously reported 1,3-bdc complexes [20, 21]. Complex **2** has only one kind of π–π stacking interaction between benzene ring and thiofuran ring with face-to-face distance

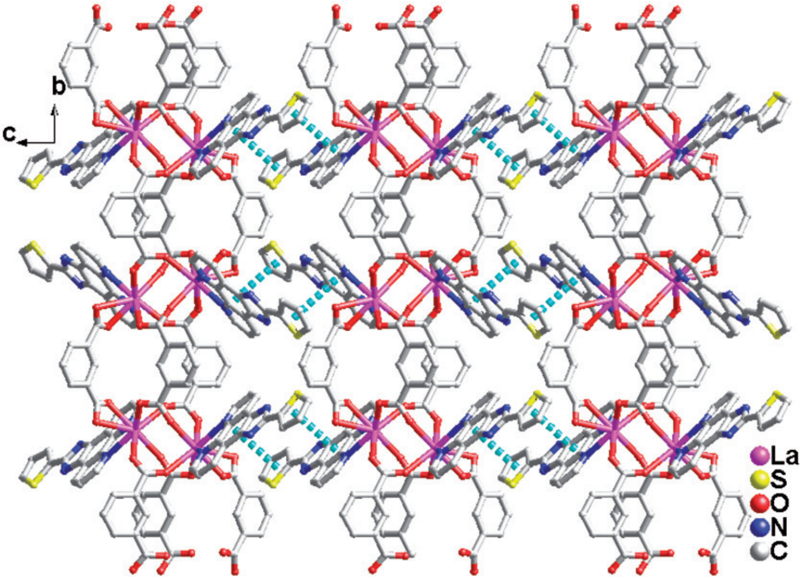


Figure 6. View of the 2-D supramolecular layer of **2** formed *via* π–π interactions (blue broken lines represent π–π stacking interactions).

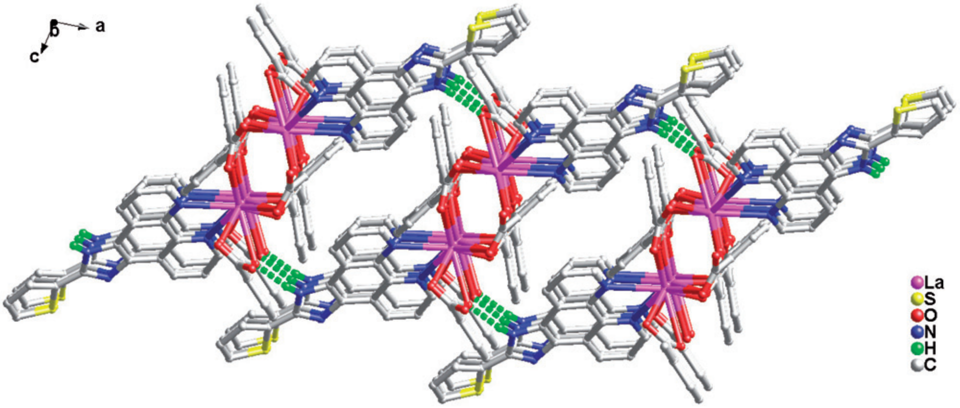


Figure 7. View of the 3-D supramolecular structure of **2** formed *via* H-bonding interactions (green broken lines represent H-bonding interactions).

of 3.590 Å (the dihedral angle is 10.20°). Therefore, the 1-D chain structure is extended into an interesting supramolecular 2-D network (figure 6). Compared with **1**, **2** shows strong hydrogen bonding interactions (N4–H4A···O1 $(1 + x, y, z) = 1.95$ Å, N···O = 2.792 Å). Thus, a R_2^2 (24) hydrogen bonding ring involving N–H···O (N4–H4A···O4, 2.896(6) Å) is formed and the 2-D chain is extended into a 3-D supramolecular framework by hydrogen bonding interactions (figure 7).

Many metal complexes based on N-donor ligands or polycarboxylate, such as 1,10-phenanthroline, isophthalate, benzene-1,4-dicarboxylate, and pyridyl polycarboxylate, have been reported [22–25], but those constructed from TIP and isophthalate mixed ligands are rare. Compared with previously reported related complex $[La(phen)(HNip)(Nip)]_n$ (phen = 1,10-phenanthroline, Nip = 5-nitroisophalate) [22], in which a 2-D network was observed, **2** is quite different. Moreover, the coordination mode of 5-nitroisophthalate in $[La(phen)(HNip)(Nip)]_n$ is also different from isophthalate in **2**. The differences indicate that the structures of complexes and the coordination modes of organic polycarboxylates can be changed by the N-donor ligand and additional groups from polycarboxylate. In addition, the carboxyl groups of isophthalate are at 1,3-positions, which are convenient bridging units for linking the adjacent metal centers to form a dinuclear building unit. Thus, similar to those of reported analogous complexes [14, 25, 26], dimetal units $[Cd_2N_4O_4]$ and $[La_2N_4O_{12}]$ were obtained in the title complexes. Notably, compared with M-phen-polycarboxylate complexes [14, 22], the most important feature of **1** and **2** is that the N-donor ligand TIP is a donor for hydrogen bonding to isophthalate resulting in the formation of the final supramolecular framework.

3.2. *Effect of TIP ligand on the supramolecular structures of the complexes*

Although both the phen and TIP molecules are planar, there are two additional hetero-aromatic rings present in TIP, which have important function in the formation of the final frameworks. The bulky hetero-aromatic ring in the backbone significantly increases the steric hindrance of TIP, leading to structural differences in their complexes compared with M-phen complexes [26]. Comparing **1** and **2**, the coordination orientation of TIP and the coordination number of metal ion play important roles in the formation of the different weak interactions. In **1**, TIP's are almost co-planar with dihedral angle of 0.345°, attached to both sides of the 1-D chain. As a result, the TIP ligands have more chance to form π–π stacking interactions in **1**. Thus, three kinds of π–π interactions are found in **1** (figure 8). For **2**, the TIP ligands are also attached to both sides of 1-D chain with the big dihedral angle of 82.079°. Complex **2** possesses only one kind of π–π stacking interaction (figure 8). However, the π–π stacking interactions of **2** are stronger than those in **1**. Similar hydrogen bonding interactions involving $N–H\cdots O$ were found in **1** and **2** (figure 8). Different hydrogen bonding ring structures indicate that the metal center may become the decisive factor.

3.3. *Thermogravimetric analysis*

Thermogravimetric analyses (TGA) were carried out for **1** and **2**, as shown in "Supplementary material". For **1**, TGA shows that decomposition starts at 332°C and ends at 456°C with 77.88% weight loss, equivalent to the loss of organic ligands (Calcd 77.62%); the remaining weight corresponds to CdO. The decomposition of **2** was similar to **1**, but showed higher thermal stability. Here mass decrease began at 439°C and was completed by 546°C, corresponding to the loss of 53.34% (Calcd 52.63%). The remaining weight corresponds to La_2O_3.

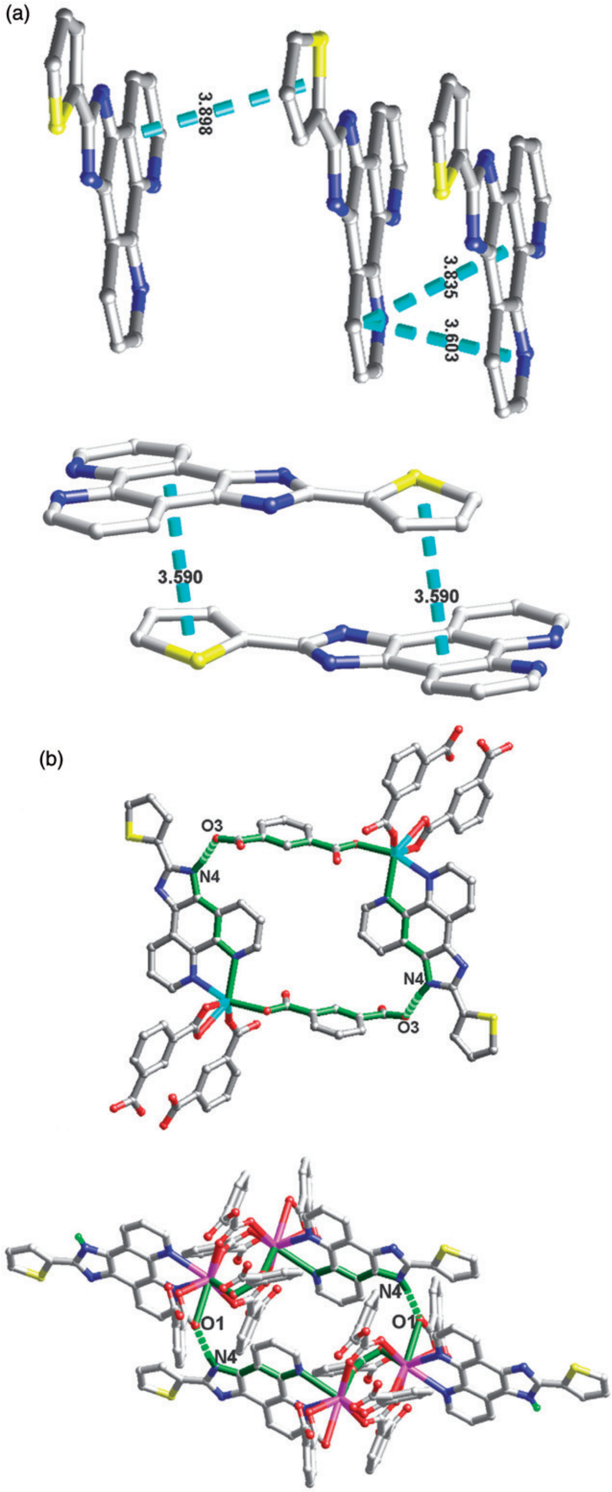


Figure 8. View of (a) the π–π stacking interactions; (b) the H-bonding interactions in **1** and **2**, respectively.

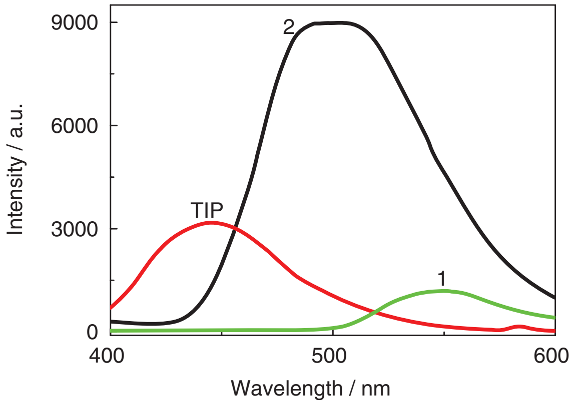


Figure 9. Fluorescence emission spectra for TIP, **1** and **2** in the solid state at room temperature.

3.4. *Fluorescence properties of 1 and 2*

Luminescence is of great importance in photochemistry and photophysics [27]. The fluorescence properties of **1** and **2** were investigated in the solid state at room temperature together with free ligands for comparison (figure 9). The free ligands display emission at 350 nm ($\lambda_{ex} = 310$ nm) for 1,3-H_2bdc [28] and 443 nm ($\lambda_{ex} = 293$ nm) for TIP. These emission bands of free ligands can be assigned to the $\pi \rightarrow \pi^*$ transitions [29]. For complexes, there are emission bands at 551 nm ($\lambda_{ex} = 310$ nm) for **1** and 502 nm ($\lambda_{ex} = 339$ nm) for **2**, respectively. On comparison with the emission of the free ligands, a red-shift is observed upon coordination of the ligands. Thus, the fluorescence behavior of **1** and **2** are best ascribed to the ligand-to-metal charge transfer (LMCT) [30, 31]. The red-shift emission peak probably is related to the intraligand fluorescent emission [32]. Differences in intensity and emission maxima between **1** and **2** are probably due to the different metal ions and supramolecular environments [33, 34].

4. Conclusions

Two new coordination polymers $[\mathrm{Cd(TIP)(1,3\text{-}bdc)}]_n$ (**1**) and $\{[\mathrm{La(TIP)(1,3\text{-}bdc)}_{1.5}] \cdot 0.2\mathrm{H_2O}\}_n$ (**2**) have been isolated under hydrothermal conditions by reactions of the TIP and metal salts together with isophthalic acid. Complexes **1** and **2** exhibit 1-D ribbon structures with different configurations, which are further extended into 3-D supramolecular frameworks by weak interactions. TIP with the large π-system and additional hydrogen bonding group plays the most crucial role in the formation of supramolecular framework by π–π stacking and hydrogen bonding interactions. The fluorescence properties of **1** and **2** have also been investigated.

Supplementary material

Crystallographic data for the structures reported in this article have been deposited in the Cambridge Crystallographic Data Center with CCDC reference numbers 753612 and

737499 for **1** and **2**, respectively. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

Acknowledgments

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