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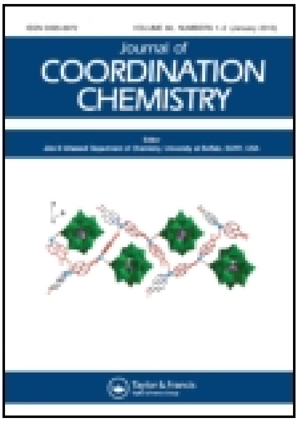

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Syntheses, crystal structures, and photophysical properties of two 2-D coordination polymers with different geometries of lead(II)

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Syntheses, crystal structures, and photophysical properties of two 2-D coordination polymers with different geometries of lead(II)

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Two complexes constructed from aromatic acid and *N*-heterocyclic ligands have been synthesized by hydrothermal reaction: $[Pb(cipt)(NDC)]_n$ (**1**) [cipt = 2-(3-chlorophenyl)-1H-imidazo [4,5-f][1,10]phenanthroline, NDC = naphthalene-1,4-dicarboxylic acid] and $[Pb(ipm)(BDC)_2]_n$ (**2**) [BDC = terephthalic acid, ipm = 5-(1H-imidazo[4,5-f][1,10]phenanthrolin-2-yl)-2-methoxyphenol]. Single-crystal X-ray analysis shows that **1** exhibits an interesting arm-shaped chain structure. 1-D ladder chain structure is formed by N–H···O bonding interactions and further into a 2-D network by N–H···O hydrogen bonds and interchain π–π stacking interactions. Complex **2** shows a 2-D butterfly wings structure, which has been rarely reported. The structure in **2** has intermolecular N–H···O interactions, which help in construction of the 3-D framework. In **1**, the coordination sphere of Pb(II) is hemi-directed, whereas the Pb(II) geometry in **2** is holo-directed. The solid-state fluorescence spectra of **1** and **2** are also investigated, as well as the ligands cipt and ipm.

Keywords: Coordination complexes; Crystal structures; Hydrothermal synthesis; Fluorescence

1. Introduction

Design and syntheses of metal–organic coordination polymers attracts attention for topologies, fascinating structures, and potential applications in optical, electrical, magnetic, catalytic, and microporous materials [1–7]. However, design and controllable preparation of metal–organic coordination polymers still remains a long-term challenge. Structures of coordination polymers are determined by coordination nature of metal ions, ligand structures, pH, counter anions, temperature, etc. [8]. Therefore, systematic research is necessary. Generally, there are two different types of interactions, covalent bonds and non-covalent interactions, which can be used to construct varied supramolecular architectures. Much research has focused on controling motifs of metal–organic complexes through coordination bonds and relatively less attention has been given to non-covalent ($\pi\cdots\pi$ and H-bond) interactions, although non-covalent interactions can be a powerful force for directing supramolecular architectures. To obtain complexes and investigate their structure–function

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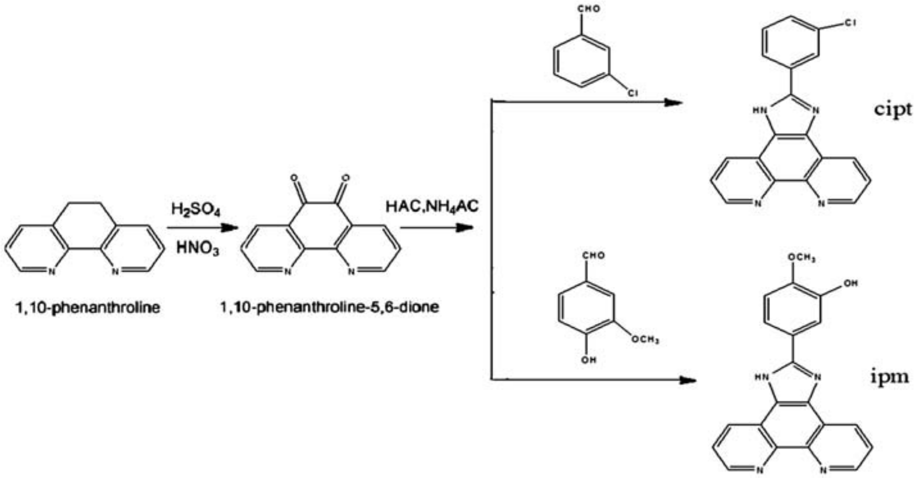


Scheme 1. The synthesis of cipt and ipm.

relationships, we have synthesized 2-(3-chlorophenyl)-1H-imidazo[4,5-f][1,10]phenanthroline (cipt) and 5-(1H-imidazo[4,5-f][1,10]phenanthrolin-2-yl)-2-methoxyphenol (ipm). The syntheses of cipt and ipm are shown in scheme 1.

These *N*-heterocyclic ligands possess an extended aromatic system, which can provide supramolecular interactions such as aromatic stacking to construct structural topologies. Our group has worked on synthesis of ligands similar to cipt and ipm [8–10]. However, investigation for this type of *N*-heterocyclic ligand is not enough, especially cipt and naphthalene-1,4-dicarboxylic acid (NDC) polymers have not been reported. Lead(II), as a heavy *p*-block metal, has a large radius and flexible coordination. Moreover, the stereochemical activity of the lone pair of electrons is an interesting issue.

We have designed and synthesized two Pb(II) coordination polymers with different geometries, $[Pb(cipt)(NDC)]_n$ (**1**) and $[Pb(ipm)(BDC)_2]_n$ (**2**). Complex **2** shows binuclear butterfly wings structure, which has been rarely reported. The existence of hydrogen bond and π–π stacking interactions reinforce the structural stability and favor construction of higher dimensional supermolecular frameworks. **1** and **2** are good luminescent materials.

2. Experimental

2.1. *Materials*

The ligands cipt and ipm were prepared according to the literature procedures [11, 12]. The metal salt, BDC, NDC, and NaOH were purchased commercially and used without purification.

2.2. *Physical measurements*

FT-IR spectra were measured with KBr pellets from 4000–400 cm^{-1} on a Perkin-Elmer 240C spectrometer. Thermogravimetric analysis was performed using a Perkin-Elmer TG-7 analyzer at 10 °C min^{-1} in nitrogen. Crystal structures were determined on a Bruker SMART

APEX II CCD X-ray diffractometer. Carbon, hydrogen, and nitrogen elemental analyzes were performed with a PE-2400 elemental analyzer.

2.3. *Syntheses*

$[Pb(cipt)(NDC)]_n$ (**1**): A mixture of cipt (0.100 g, 0.3 mmol), $Pb(Ac)_2·3H_2O$ (0.114 g, 0.3 mmol) and NDC (0.130 g, 0.6 mmol) in distilled H_2O (18 mL) was stirred at room temperature and the pH adjusted to about 7.0 with NaOH. We put the cloudy solution into a 30 mL Teflon-lined stainless vessel at 170 °C for 3 days and afterward cooled to room temperature at a rate of 5 °C h^{-1}. Yellow crystals of **1** were obtained in 80% yield based on Pb. $C_{31}H_{17}Cl_{0.99}N_4O_{4.50}Pb$: Calcd C, 49.00; H, 2.25; N, 7.37%. Found: C, 49.15; H, 2.16; N, 7.46%. IR (KBr, cm^{-1}): 2355(s), 2330(s), 1576(vs), 1410(s), 1380(vs), 830(m), 772(m), 555(m), 438(m).

$[Pb(ipm)(BDC)_2]_n$ (**2**): A mixture of ipm (0.103 g, 0.3 mmol), $Pb(Ac)_2·3H_2O$ (0.114 g, 0.3 mmol), and BDC (0.100 g, 0.6 mmol) in distilled H_2O (18 mL) was stirred at room temperature and the pH adjusted to about 7.0 with NaOH. We put the cloudy solution into a 30 mL Teflon-lined stainless vessel at 180 °C for 3 days. The reaction was cooled to room temperature at a rate of 5 °C h^{-1}, and then small yellow crystals of **2** were collected in 70% yield based on Pb. $C_{28}H_{16}N_4O_6Pb$: Calcd C, 47.25; H, 2.26; N, 7.87%. Found: C, 47.18; H, 2.32; N, 7.81%. IR (KBr, cm^{-1}): 3400(s), 2369(s), 2324(s), 1548(vs), 1509(vs), 1338(vs), 1286(s), 817(m), 510(m).

2.4. *Crystallographic data collection and structure determination*

Single-crystal X-ray diffraction data for **1** and **2** were collected at 293(2) K with a Bruker SMART APEX II CCD diffractometer equipped with graphite-monochromated MoKα

Table 1. Crystal data and details of structure refinement parameters for **1** and **2**.

Complex	**1**	**2**
Empirical formula	$C_{31}H_{17}Cl_{0.99}N_4O_{4.50}Pb$	$C_{28}H_{16}N_4O_6Pb$
Formula weight	759.90	711.64
Crystal system	Monoclinic	Triclinic
Space group	*C*2/*c*	*P*−1
a (Å)	16.160(3)	9.895(2)
b (Å)	20.138(3)	11.414(3)
c (Å)	15.898(3)	12.873(3)
β (°)	94.664(3)	91.031(4)
Volume (Å^3)	5156.4(15)	1268.9(5)
Z	8	2
Density (Mg m^{-3}) (calculated)	1.958	1.863
Absorption coefficient (mm^{-1})	6.697	6.700
F(0 0 0)	2927	684
Crystal size (mm^3)	0.310 × 0.255 × 0.210	0.547 × 0.420 × 0.338
Theta range (°)	1.62–25.02	2.04–26.14
Reflections collected	15,190	6861
Unique reflections [R_{int}]	4554 [0.0876]	4858 [0.0175]
Goodness-of-fit on F^2	0.961	1.037
Final *R* indices [$I > 2\sigma(I)$]	$R1 = 0.0480$ $wR2 = 0.0758$	$R1 = 0.0329$ $wR2 = 0.0798$
R indices (all data)	$R1 = 0.0967$ $wR2 = 0.0884$	$R1 = 0.0387$ $wR2 = 0.0825$
Largest diff. peak and hole (e Å^{-3})	1026, −597	1780, −891

radiation ($\lambda = 0.71073$ Å) from $1.62 \leqslant \theta \leqslant 25.02°$ for **1** and $2.04 \leqslant \theta \leqslant 26.14°$ for **2**. Absorption corrections were applied using multiscan technique and all structures were solved by direct methods and refined by full-matrix least-squares based on F^2 using SHELXS-97 [13] and SHELXTL-97 [14]. Non-hydrogen atoms were refined with anisotropic temperature parameters and all hydrogens were refined isotropically. Experimental details for crystallographic data and structure refinement parameters for **1** and **2** are listed in table 1.

3. Results and discussion

3.1. *Description of the crystal structures*

3.1.1. [Pb(cipt)(NDC)]$_n$ (1). The molecular structure is shown in figure 1 and the 1-D arm-shaped chain structure is shown in figure 2. The 1-D ladder chain structure is suggested in figure 3 and the 2-D layer structure is shown in figure 4. Selected bond lengths and angles are listed in table 2.

Single-crystal X-ray diffraction analysis reveals that [Pb(cipt)(NDC)]$_n$ crystallizes in the *C*2/*c* space group. As shown in figure 1, the asymmetric unit of **1** consists of one Pb(II), one cipt, and one NDC. Pb(II) is six-coordinate with N(1) and N(2) from one chelating cipt and four oxygens (O(1), O(2), O(3A), O(4A) from two chelating bidentate NDC, forming a distorted [:PbN2O4] pentagonal bipyramidal geometry. For the coordination environment of Pb(1), Pb(1), O2, O3, O4, N1 and N2 define the basal plane, and O1 and the lone pair of electrons occupy apical positions. The N(O)–Pb–O(N) angles range from 69.2(2) to 124.0(2)°. Pb–O bond distances in **1** are 2.507(6) to 2.708(6) Å and Pb–N bond distances are 2.453(7) to 2.548(7) Å, similar with values reported [15–18]. The average bond length of Pb–O is 2.576 Å, larger than the average bonds of Pb–N (2.500 Å), suggesting that the coordination ability of cipt is a little stronger than carboxylate. Each

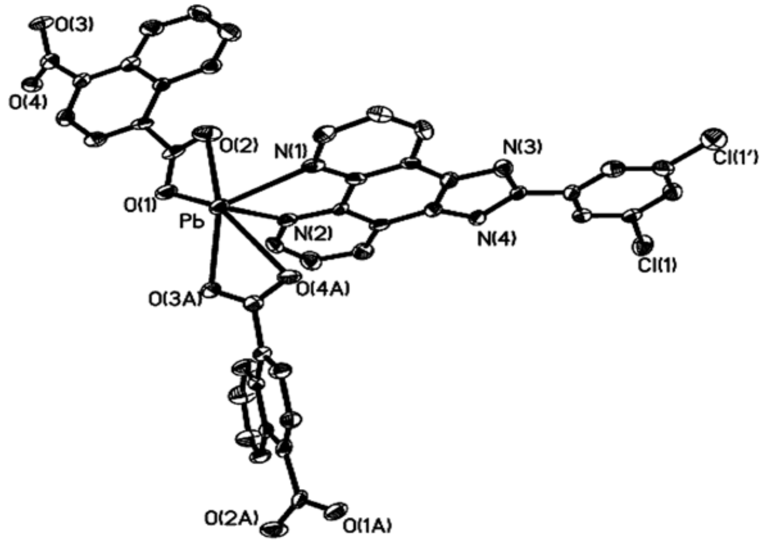


Figure 1. The molecular structure of **1** (hydrogens were omitted).

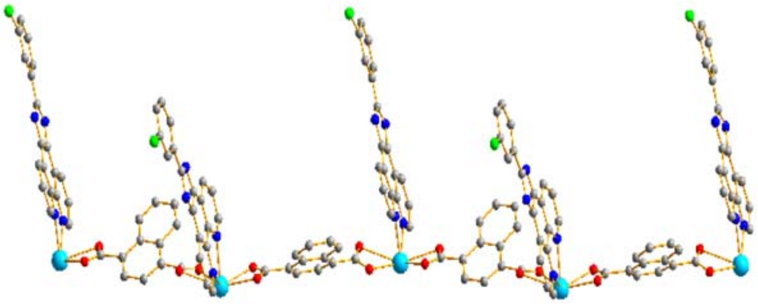


Figure 2. 1-D arm-shaped chain structure of **1** (hydrogens were omitted).

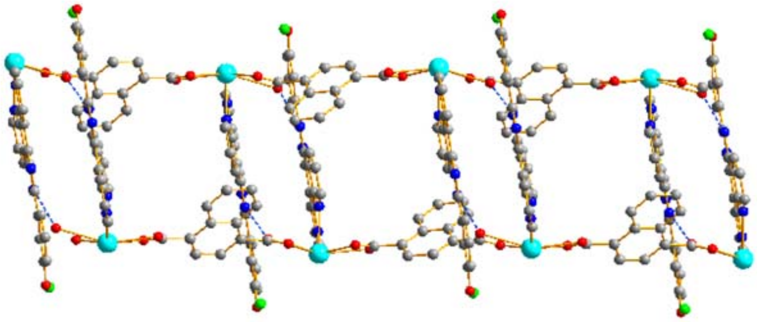


Figure 3. 1-D ladder chain structure by N–H···O bonding interactions of **1** (dotted lines represent hydrogen bonds).

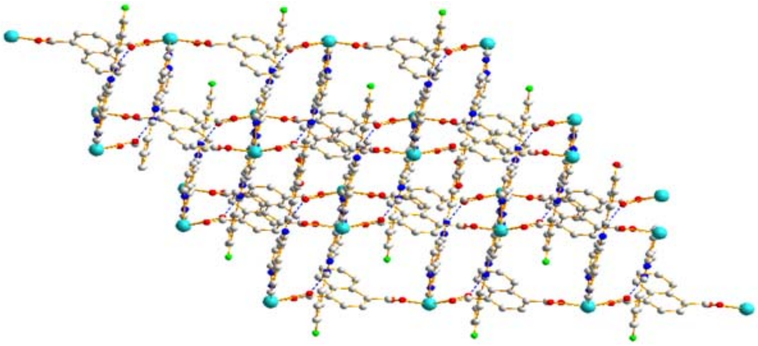


Figure 4. 2-D layer structure of **1** linked by N–H···O hydrogen bonds and inter-chain π–π stacking interactions (dotted lines represent hydrogen bonds).

NDC links two symmetry-related leads into an infinite 1-D arm-shaped chain framework with Pb–Pb distance of 11.5744 Å (figure 2). The cipt in **1** are attached on one side of the chain. There are strong N–H···O hydrogen bonds, which play an important role in stabilizing the network structure and form the 1-D ladder chain structure (figure 3). 2-D layer frameworks are formed through π–π stacking between two cipt (centroid-to-centroid distance of 3.72 Å and face-to-face distance of 3.63 Å), figure 4. Hydrogen bonds and π–π stacking interactions reinforce the structural stability of **1**, as shown by TG analysis.

Table 2. Selected bond lengths [Å] and angles [°] for **1**.

Bond	Distance	Bond	Distance
Pb–O(1)	2.507(6)	Pb–O(3)$^{\#1}$	2.542(6)
Pb–O(2)	2.550(6)	Pb–O(4)$^{\#1}$	2.708(6)
Pb–N(1)	2.548(7)	Pb–N(2)	2.453(7)
Angle	(°)	Angle	(°)
O(1)–Pb–O(3)$^{\#1}$	88.9(2)	O(1)–Pb–O(2)	51.6(2)
O(1)–Pb–O(4)$^{\#1}$	131.5(2)	O(3)$^{\#1}$–Pb–O(4)$^{\#1}$	49.79(19)
O(2)–Pb–O(4)$^{\#1}$	148.0(2)	O(3)$^{\#1}$–Pb–O(2)	138.5(2)
N(2)–Pb–O(1)	78.0(2)	N(2)–Pb–O(3)$^{\#1}$	76.3(2)
O(1)–Pb–N(1)	118.9(2)	N(2)–Pb–O(2)	82.8(2)
O(3)$^{\#1}$–Pb–N(1)	124.0(2)	N(2)–Pb–O(4)$^{\#1}$	69.2(2)
N(1)–Pb–O(2)	76.0(2)	N(1)–Pb–O(4)$^{\#1}$	78.4(2)
N(2)–Pb–N(1)	65.3(2)		

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

3.1.2. [Pb(ipm)(BDC)$_2$]$_n$ (2).

The molecular structure is shown in figure 5 and the 2-D layer structure is shown in figure 6. The 3-D structure of **2** is suggested in figure 7. Selected bond lengths and angles are listed in table 3.

Complex **2** crystallizes in triclinic space group *P*−1 and the asymmetric unit consists of one Pb(II), one ipm, and two BDC, as illustrated in figure 5. Pb(II) is five-coordinate with N(1) and N(2) from one chelating ipm and three oxygens (O(1), O(2) from one chelating bidentate NDC and O(4) from a different bridging NDC). The secondary bond of Pb(II) with carboxylic oxygen of an adjacent molecule Pb(1)···O(3) with a distance of 2.830(4) Å (dashed lines in figure 5) is longer than the sum of the ionic radii but significantly shorter

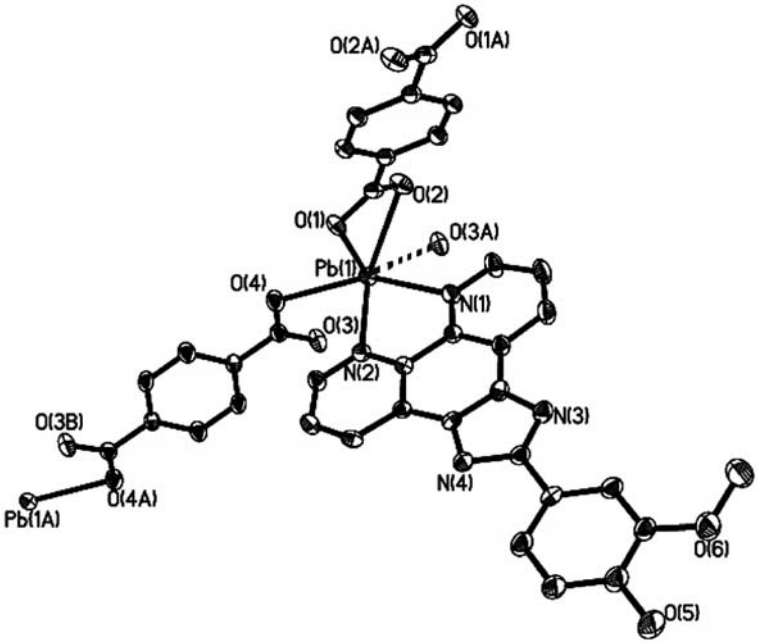


Figure 5. The molecular structure of **2** (hydrogens were omitted).

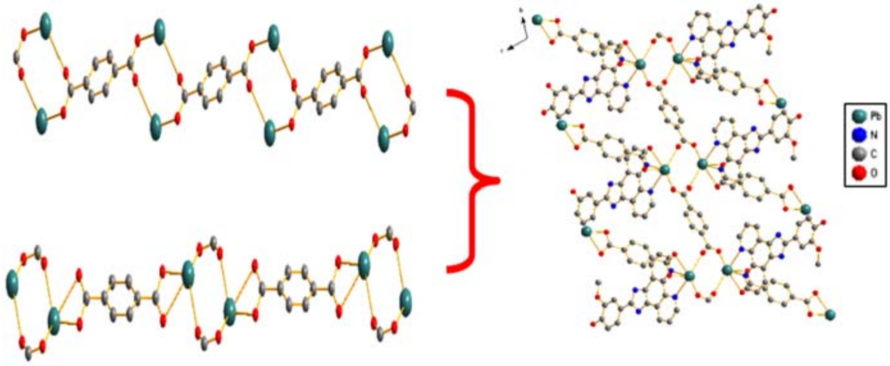


Figure 6. The 2-D butterfly wings structure of **2** (hydrogens were omitted).

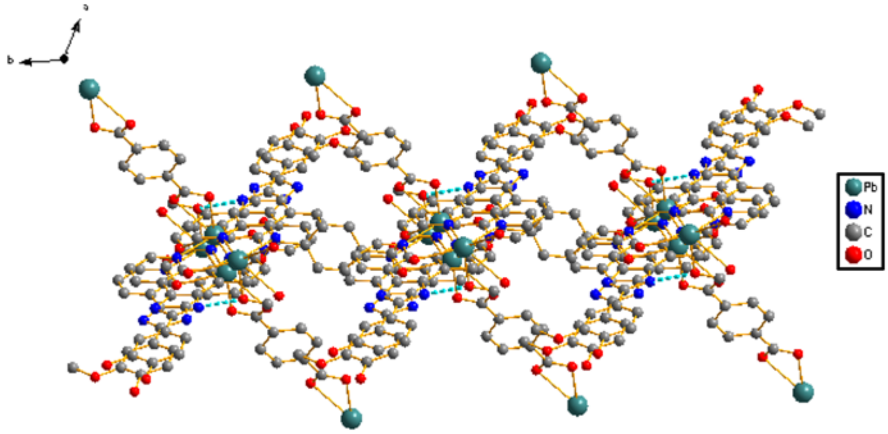


Figure 7. The 3D structure of **2** (dotted lines represent hydrogen bonds).

Table 3. Selected bond lengths [Å] and angles [°] for **2**.

Bond	Distance	Bond	Distance
Pb(1)–O(1)	2.466(4)	Pb(1)–O(4)	2.530(4)
Pb(1)–O(2)	2.681(4)	Pb(1)–O(3)	2.830(4)
Pb(1)–N(2)	2.617(5)	Pb(1)–N(1)	2.617(4)
Angle	(°)	Angle	(°)
O(1)–Pb(1)–O(4)	84.32(13)	O(4)–Pb(1)–O(2)	124.52(14)
O(3)–Pb(1)–O(4)	132.06(8)	O(2)–Pb(1)–O(3)	74.62(7)
O(1)–Pb(1)–O(2)	51.06(12)	O(1)–Pb(1)–N(1)	84.40(14)
O(4)–Pb(1)–N(1)	141.79(15)	O(1)–Pb(1)–N(2)	80.12(13)
O(4)–Pb(1)–N(2)	79.49(15)	N(1)–Pb(1)–O(2)	72.69(14)
N(2)–Pb(1)–O(2)	115.95(13)	N(1)–Pb(1)–N(2)	62.63(14)

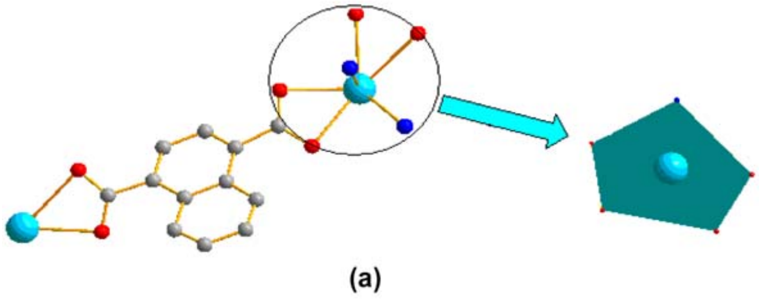
than the sum of the van der Waals radii (3.54 Å), which can be explained by the presence of an active lone electron pair in proximity of the oxygen. If the Pb(1)···O(3) bond is taken into account, then the geometry around lead(II) can be described as six-coordinate [:PbN2O4] pentagonal bipyramidal geometry [19]. For the coordination environment of Pb

(1), Pb(1), O2, N1, N2, O4, and the lone pair of electrons define the basal plane, and O1 and O(3A) occupy the apical positions. Pb–O and Pb–N distances are 2.466(4)–2.830(4) and 2.617(4)–2.617(5) Å, respectively, similar to reported Pb–O and Pb–N distances [16, 20–23]. The N(O)–Pb–O(N) angles are from 72.69° to 141.79(15)°.

Dicarboxylates are widely used in assembly of supramolecular architectures for their diverse coordination modes and bridging ability. In **2**, BDC has two different coordination modes, bridging bis-bidentate and chelating bis-bidentate [24]. BDC links four symmetry-related leads in bridging bis-bidentate coordination into an infinite 1-D chain. At the same time, BDC links two symmetry-related leads in chelating bis-bidentate coordination into another infinite 1-D chain framework with shorter Pb–Pb separation (3.8684 Å). The existence of these two 1-D chains gives a 2-D binuclear butterfly wings structure, as shown in figure 6. Two BDC and ipm ligands are attached to both sides of this chain regularly, just like the wings of butterflies. In the unit of **2**, an eight-membered ring forms, which contains Pb(1)–O(4)–C(21)–O(3)–Pb(1A)–O(4A)–C(21A)–O(3A)–Pb(1) (A: $1-x$, $2-y$, $1-z$), and the angle of O(3)–C21–O(4) is 123.809°.

The structure in **2** has intermolecular N–H···O [H(4A)···O(1) = 2.020 Å, N(4)···O(1) = 2.828 Å, and N(4)–H(4A)···O(1) = 156°] interactions, which help in construction of the 3-D framework (figure 7). Existence of hydrogen bond interactions reinforces the structural stability of **2**, as proved by TG analysis.

Geometries of Pb(II) complexes can be classified as (1) holo-directed, in which the bonds to ligands are distributed throughout the surface of an encompassing globe and (2) hemi-directed, in which the bonds to ligands are directed throughout only part of the globe [25]. The coordination environment of Pb(II) in **1** is hemi-directed coordination with a void in the distribution of bonds to the ligands. Complex **2** is holo-directed coordination with bonds to the ligands distributed throughout the surface of the encompassing Pb(II) sphere (figure 8). The diversity of coordination modes of dicarboxylate ligand result in



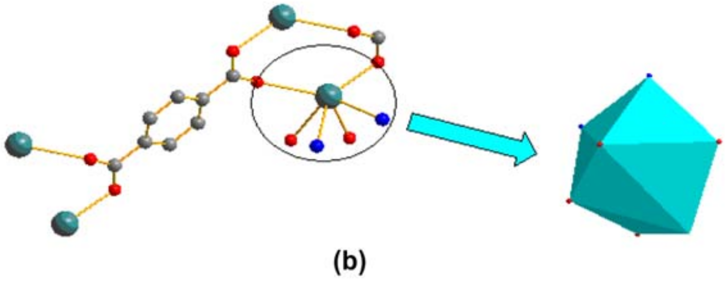


Figure 8. Coordination modes and sphere of the central metal ions in **1** (a) and **2** (b).

difference of structures in **1** and **2**. Coordination modes (chelating bis-bidentate or bis-monodentate) usually have 1-D chain or 1-D double-chain structures and diverse coordination (monodentate-bidentate or chelating/bridging bis-bidentate) usually have higher dimensional crystal structures. Multidentate ligands coordinate to metal by the N donors on heterocyclic rings and O donors of carboxyl groups. As proton donors and acceptors, the carboxylic oxygens and nitrogens in heterocyclic carboxylic acids not only coordinate as monodentate or multidentate ligands, but also provide intermolecular hydrogen bonds for assembling the complex into high-dimensional supramolecular networks [26, 27]. Existence of hydrogen bonds reinforces the structural stability of complexes.

3.2. *IR spectra*

In **1**, peaks at 1576 and 1380 cm^{-1} correspond to the antisymmetric and symmetric stretching of carboxyl. The $\Delta\nu$ ($\nu_{as}(\text{COO}^-)$–$\nu_s(\text{COO}^-)$) is 196 cm^{-1} (smaller than 200 cm^{-1}), indicating bidentate carboxyls. In **2**, the strong absorptions at 1548 and 1509 cm^{-1} are the antisymmetric stretching of carboxyl and the absorption at 1338 cm^{-1} is the symmetric stretch of carboxyl. The separation is 210 and 171 cm^{-1}, which indicates the carboxyls adopt monodentate and bidentate coordination. The IR results agree with their solid structural features from crystal structures.

3.3. *Thermal properties*

TG analysis was used to investigate the stability of **1** and **2** (Supplementary material). Complex **1** exhibits good stability, stable below 333 °C. Weight loss of 29.00% from 333 to 389 °C reveals loss of NDC (Calcd 28.45%). The second weight loss of 45.00% from 389 to 642 °C corresponds to loss of cipt (Calcd 43.53%). In **2**, the first weight loss of 24.00% from 402 to 442 °C corresponds to loss of BDC (Calcd 23.33%). The second weight loss from 442–605 °C can be ascribed to the release of ipm (obsd. 49.20%, Calcd 48.11%). The final formation may be PbO. The analysis results indicate that the frameworks of **1** and **2** are very stable.

3.4. *Photoluminescent properties*

Luminescent complexes are of interest because of applications in photochemistry and photophysics [28, 29]. Therefore, we report the luminescence of the free ligands, **1** and **2** (see figures 9 and 10). The free cipt exhibits one emission band at 533 nm upon excitation at 325 nm and the ipm exhibits one emission band at 497 nm upon excitation at 320 nm. Complex **1** shows two emission bands: the emission peak at 420 nm (excitation at 340 nm) probably originating from the π–π^* electronic transition of NDC because NDC exhibits emission at 472 nm upon excitation at 392 nm; the second emission peak at 619 nm is red-shifted by 86 nm relative to that of free cipt. Complex **2** shows one strong emission at 532 nm (excitation at 307 nm), which is red-shifted by 35 nm relative to that of free ipm. The luminescence suggests emission bands of the complexes are the result of poly-carboxylate and *N*-heterocyclic ligands interacting on each other. Our complexes exhibit strong emissions, attributed to the rigidity of complexes. The rigidity favors energy transfer and reduces the loss of energy through a radiationless pathway [30]. However, the effect of the microenvironment between ligands and complexes on luminescence still needs further investigations. Complexes **1** and **2** may be candidates as photoluminescence materials

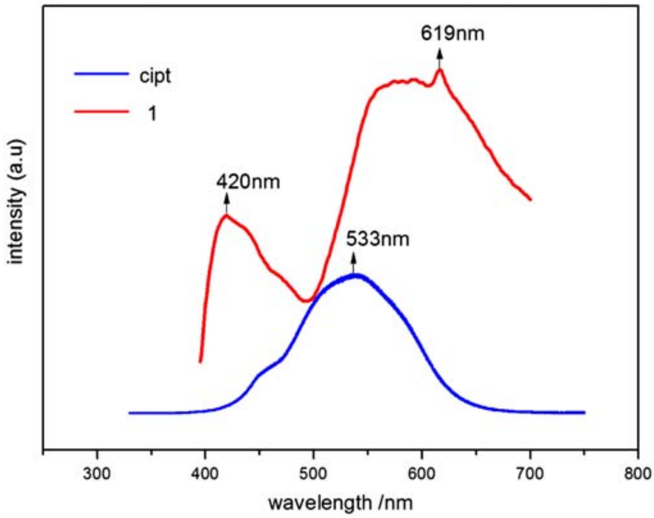


Figure 9. Luminescence spectrum of cipt and **1** in solid state at room temperature.

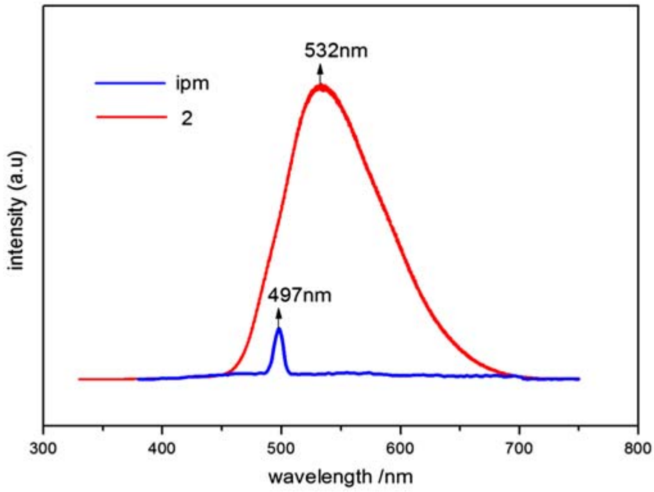


Figure 10. Luminescence spectrum of ipm and **2** in solid state at room temperature.

because they are highly thermally stable and insoluble in water and common organic solvents.

4. Conclusions

Two complexes have been synthesized using planar multifunctional ligands cipt, ipm, NDC, and BDC. The coordination environment of Pb(II) in **1** is a good example of hemi-directed coordination, whereas **2** is a holo-directed coordination, with bonds to the ligand distributed throughout the surface of the encompassing Pb sphere. Our complexes exhibit strong emissions attributed to the rigidity of complexes. Hydrogen bonds and π–π interactions reinforce the structural stability of **1** and **2**. TG analysis reveals that they are very stable. Complexes **1** and **2**, highly thermally stable and insoluble in water and common organic solvents, are worthy of further study as potential photoluminescence materials.

Supplementary material

CCDC 832020 and 858668 contain the supplementary crystallographic data for **1** and **2**, respectively. These data can be obtained free of charge from the Cambridge Crystallographic Data Center via http://www.ccdc.cam.ac.uk/data_request/cif.

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