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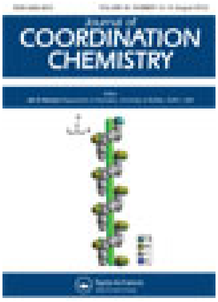

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Two new Pb(II) coordination polymers derived from fatty dicarboxylates and phenanthroline derivatives: syntheses, structures, and properties

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Two new Pb(II) coordination polymers derived from fatty dicarboxylates and phenanthroline derivatives: syntheses, structures, and properties

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Two new Pb(II) coordination polymers, $[Pb(3\text{-pdip})(L_1)]\cdot 0.5H_2O$ (**1**) and $[Pb(4\text{-pdip})(L_2)]$ (**2**) [L_1 = heptane diacid, L_2 = hexane diacid, 3-pdip = 2-(3-pyridyl)imidazo[4,5-f]1,10-phenanthroline, 4-pdip = 2-(4-pyridyl)imidazo[4,5-f]1,10-phenanthroline], have been obtained from hydrothermal reactions of Pb(II) nitrate with two flexible fatty carboxylic acids and two phenanthroline derivatives. Single-crystal X-ray diffraction analysis reveals that **1** is a 1-D staircase-like double chain coordination polymer extended by binuclear $[Pb_2N_4O_8]$ subunits and pairs of L_1 ligands. Compound **2** exhibits a 2-D (4,4) network based on bi-metallic $[Pb_2N_6O_8]$ units and $(L_2)_2/(4\text{-pdip})_2$ double linkers. Furthermore, adjacent chains of **1** and layers of **2** are extended into 3-D supramolecular networks by π–π stacking interactions between aromatic rings of phenanthroline groups. The organic carboxylic acids with different lengths show great influence on the shapes of Pb(II)-carboxylate double chains. Phenanthroline derivatives possessing different sites of N-donors (3-pyridyl and 4-pyridyl) determine the final coordination frameworks of **1** and **2**. Photoluminescence and thermal stabilities of **1** and **2** were also investigated.

Keywords: Hydrothermal syntheses; Crystal structures; Lead coordination polymers; Photoluminescent property

1. Introduction

Design and synthesis of metal–organic coordination polymers have developed because of structural diversities and potential applications in gas storage, luminescence, and magnetic materials [1–4]. However, rational design of structures with specific properties is still a long-term challenge [5]. Careful selection of the ligands, such as length, substituent, and site of donor, has been an effective way to obtain target compounds [6–8]. Two types of N-donor ligands, including chelating ligands (2,2′-bipyridine and 1,10-phenanthroline) and bridging ligands (multi-pyridine and multi-imidazole), have attracted great interest. A number of coordination polymers with various structures

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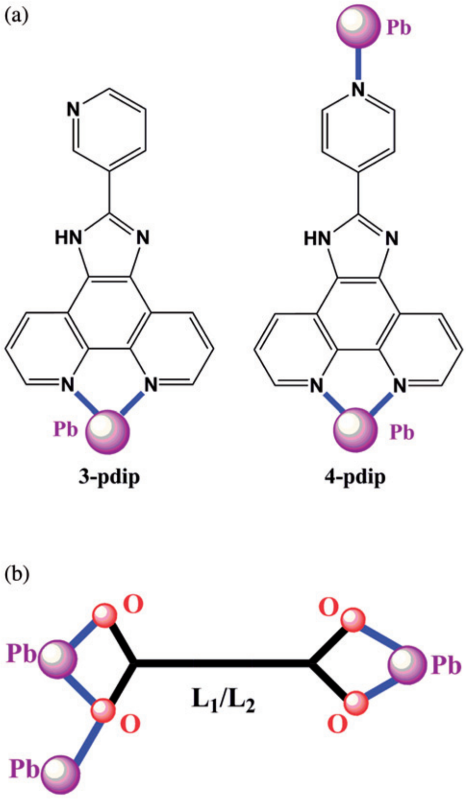
have been prepared from N-donor ligands [9–13]. Some 1,10-phenanthroline derivatives, such as 2-(4-hydroxylbenzene)imidazo[4,5-f]1,10-phenanthroline (HOIP), 2-(3-pyridyl)imidazo[4,5-f]1,10-phenanthroline (3-pdip), and 2-(4-pyridyl)imidazo[4,5-f]1,10-phenanthroline (4-pdip), possess advantages of the N-donors mentioned above, and some metal–organic coordination polymers have been prepared by Yang, Wang, and our group [14–19]. However, investigation of the influence of 1,10-phenanthroline derivatives on the formation of coordination polymers remains largely unexplored [14, 19]. Fatty carboxylic acids showing remarkable coordination ability and versatile coordination modes have been widely used in constructing functional coordination polymers. Several reports demonstrate that the spacer length of this kind of anionic ligand impacts topological structures of the final products [15].

Here, we chose heptane diacid (L_1) and hexane diacid (L_2) as anionic ligands, 3-pdip and 4-pdip as the N-donor ligands based on the following considerations (scheme 1): (i) as multidentate O-donor ligands, L_1 and L_2 with different spacer length possess multi $–CH_2–$ groups for free twist, which may induce different metal–carboxylate coordination frameworks; (ii) 3-pdip and 4-pdip, as bifunctional N-donor ligands with different sites of donor, possess the advantage of chelating and bridging ligands, but also have large aromatic-ring system and may provide potential supramolecular



Scheme 1. (a) The coordination modes of 3-pdip and 4-pdip and (b) the schematic view of the coordination mode of L_1 = heptane dicarboxylate and L_2 = hexane dicarboxylate in **1** and **2**.

recognition sites for π–π stacking interactions, influencing formation of the final supramolecular networks.

In this article, we report a 1-D double-chain coordination polymer [Pb(3-pdip)(L_1)] · 0.5H_2O (**1**) and a 2-D (4,4) network [Pb(4-pdip)(L_2)] (**2**) with two kinds of double linkers. The differences of coordination modes of metal ions and N-donor ligands, conformations of anionic ligands, and the final structures of **1** and **2** reveal that the length of organic carboxylic acids and the site of donors play important roles on synthesis of metal–organic coordination polymers.

2. Experimental

2.1. *Materials and methods*

All reagents employed were commercially available and used as received. 3-pdip and 4-pdip were synthesized by the literature method [20]. FT-IR spectra (KBr pellets) were taken on a Magna FT-IR 560 Spectrometer and elemental analyses (C, H, and N) were carried out on a Perkin-Elmer 240 C elemental analyzer. Thermogravimetric analysis (TGA) was carried out with a Pyris Diamond TG-DTA instrument and luminescence spectra for the samples were measured on a HITACHI F–4500 Fluorescence Spectrophotometer.

2.2. *Syntheses*

2.2.1. Synthesis of [Pb(3-pdip)(L_1)] · 0.5H_2O (1). A mixture of $Pb(NO_3)_2$ (0.1 mmol), L_1 (0.1 mmol), 3-pdip (0.1 mmol), H_2O (12 mL), and NaOH (0.2 mmol) was stirred for 20 min in air, then transferred and sealed in a 25 mL Teflon reactor, which was heated at 180°C for 3 days leading to formation of yellow block crystals of **1** (~18% yield based on Pb). Anal. Calcd for $C_{50}H_{44}Pb_2N_{10}O_9$ (%): C, 44.70; H, 3.30; N, 10.43. Found (%): C, 44.69; H, 3.32; N, 10.46. IR (KBr, cm^{-1}): 3391 w, 2359 w, 1614 s, 1551 s, 1387 s, 801 s, 736 s.

2.2.2. Synthesis of [Pb(4-pdip)(L_2)] (2). The synthetic procedure for **2** is the same as that for **1** except that L_2 (0.1 mmol) and 4-pdip (0.1 mmol) were used instead of L_1 and 3-pdip. Yellow block crystals of **2** were obtained (~24% yield based on Pb). Anal. Calcd for $C_{24}H_{19}PbN_5O_4$ (%): C, 44.44; H, 2.95; N, 10.80. Found (%): C, 44.42; H, 2.92; N, 10.82. IR (KBr, cm^{-1}): 2360 w, 1611 s, 1549 s, 1385 s, 1181 w, 1064 m, 797 s, 738 s.

2.3. *X-ray crystallographic study*

Crystallographic data for **1** and **2** were collected on a Bruker Smart 1000 CCD diffractometer with Mo-Kα radiation ($\lambda = 0.71073$ Å for **1** and 0.71069 Å for **2**) by ω scan mode from $1.94° \leq \theta \leq 25.0°$ for **1** and $1.67° \leq \theta \leq 25.0°$ for **2**. Structural solution and refinement based on observed reflections gave the final $R_1(wR_2) = 0.0357$ (0.0745) for **1** and $R_1(wR_2) = 0.0322$ (0.1524) for **2**. The structures were solved by direct methods using SHELXS of the SHELXTL package and refined by full-matrix least-squares

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

Empirical formula	$C_{50}H_{44}Pb_2N_{10}O_9$	$C_{24}H_{19}PbN_5O_4$
Formula weight	1343.34	648.64
Temperature (K)	293(2)	296(2)
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$C2/c$
Unit cell dimensions (Å, °)		
a	8.6331(12)	23.2903(16)
b	10.8843(16)	15.0873(16)
c	13.0022(19)	14.0655(12)
α	80.341(2)	90
β	87.758(2)	117.894(2)
γ	77.471(2)	90
Volume ($Å^3$), Z	1175.7(3), 2	4368(2), 8
Calculated density ($g\ cm^{-3}$)	1.894	1.973
Absorption coefficient (mm^{-1})	7.220	7.768
$F(000)$	648	2496
θ maximum for data collection (°)	25	25
$R(int)$	0.0261	0.0249
Goodness-of-fit on F^2	0.994	1.036
R_1^a $[I > 2\sigma(I)]$	0.0357	0.0322
$wR_2{}^b$ (all data)	0.0745	0.154
Largest difference peak and hole ($e\ Å^{-3}$)	0.736 and −0.862	0.925 and −1.326

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

methods with SHELXL [21, 22]. All non-hydrogen atoms were refined anisotropically and all hydrogen atoms were placed in geometrically idealized positions and refined isotropically. The details of crystallographic information for **1** and **2** are summarized in table 1. Selected bond lengths and angles are listed in table S1.

3. Results and discussion

3.1. *Structure descriptions*

3.1.1. [Pb(3-pdip)(L_1)]·0.5H_2O (1). Single-crystal X-ray analysis shows that **1** is a 3-D supramolecular network derived from 1-D double chains extended by π–π stacking interactions. The crystal structure analysis indicates that Pb is seven-coordinate by five oxygen atoms from three different L_1 and two nitrogen atoms from one 3-pdip with a chelating coordination mode showing a hemidirected coordination geometry (figure 1). The Pb–O distances range from 2.467(4) to 2.936(5) Å and Pb–N distances vary from 2.645(5) to 2.652(5) Å, which are near to the reported $[Pb_2(L)_2(1,4\text{-bdc})_2]_n$ (1,4-bdc = 1,4-benzenedicarboxylate and L = dipyrido[7,6-*a*:6′,7′-*c*]-3-chloropyrido[2,3-*b*]quinoxaline) [23]. The N–Pb–N angle is 61.79(16)°. The O–Pb–O and N–Pb–O angles are 48.1(2)–139.8(6)° and 76.88(17)–165.5(4)°, respectively. The Pb coordination polyhedron is slightly distorted, which may be caused by the steric effect of the ligands.

In **1**, the two carboxylic groups of L_1 exhibit asymmetric chelating–bridging coordination. The μ_2 O3 from carboxylates with chelating–bridging coordination (μ_2–$\eta^2:\eta^1$) link adjacent Pb(II)'s to form a bi-metallic $[Pb_2N_4O_8]$ subunit with Pb(II)···Pb(II) distance of 4.7268(6) Å. The subunits are extended into a staircase-like double chain coordination polymer through the pairs of L_1 (figure 2a) and the distance between the

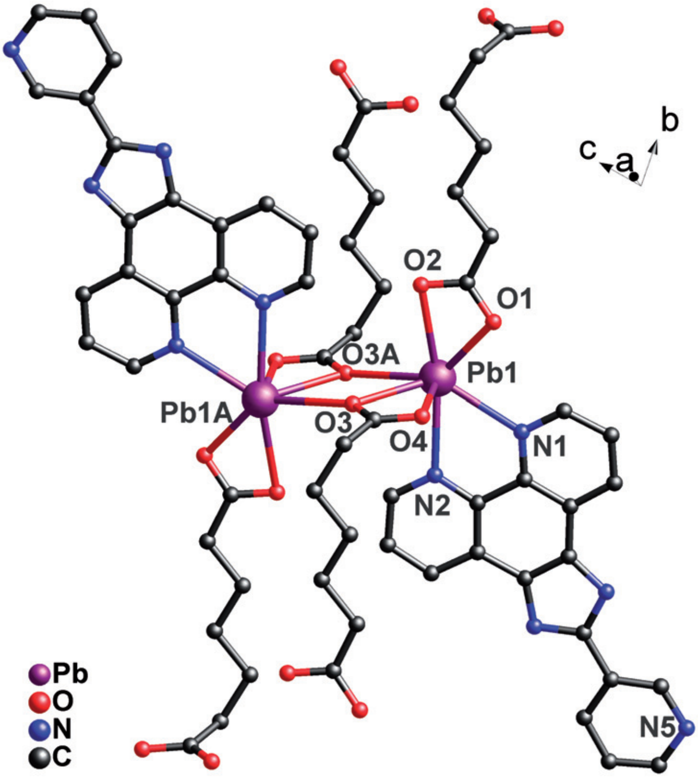


Figure 1. The coordination environment of Pb(II) in **1** (A: $2-x$, $1-y$, $2-z$).

cores of $[Pb_2N_4O_8]$ subunits is 10.884(6) Å. Intermolecular π–π stacking interactions exist between aromatic rings of 3-pdip. Adjacent chains are interlinked through π–π stacking interactions between pyridine rings (containing N1 and N5) of 3-pdip to form a 2-D supramolecular layer [Cg–Cg (distance between ring centroids) is 3.854(4) Å and the dihedral angle is 2.53°] (figure 2a). The 2-D layers are extended into 3-D supramolecular architecture by π–π stacking interactions between pyridine rings (containing N1) and the center phenyl ring of 3-pdip [Cg–Cg is 3.635(2) Å and the dihedral angle is 0°] (figure 2b and c).

To better understand the structure of **1**, it is necessary to simplify the building blocks from the 3-D supramolecular network. Considering the binuclear Pb cluster linked by O3 as a six-connected node, pairs of L_1 as the double linkers, and the two kinds of supramolecular π–π stacking interactions as the non-covalent spacers, the whole structure of **1** can be considered to be a distorted *a*-Po topology framework (figure S1).

3.1.2. [Pb(4-pdip)(L_2)] (2).

X-ray study reveals that **2** is a 2-D bi-layer coordination polymer exhibiting (4,4) network based on Pb(II) binuclear units and two kinds of double linkers $(4\text{-pdip})_2/(L_2)_2$. As shown in figure 3, **2** consists of one Pb(II), one 4-pdip, and one L_2 anion. Each Pb(II) is holodirected and eight-coordinate by two nitrogen atoms (N1 and N2) of phenanthroline and one N5 of pyridine from two different 4-pdip ligands, two chelating carboxylate oxygen atoms of one L_2 anion and three oxygen

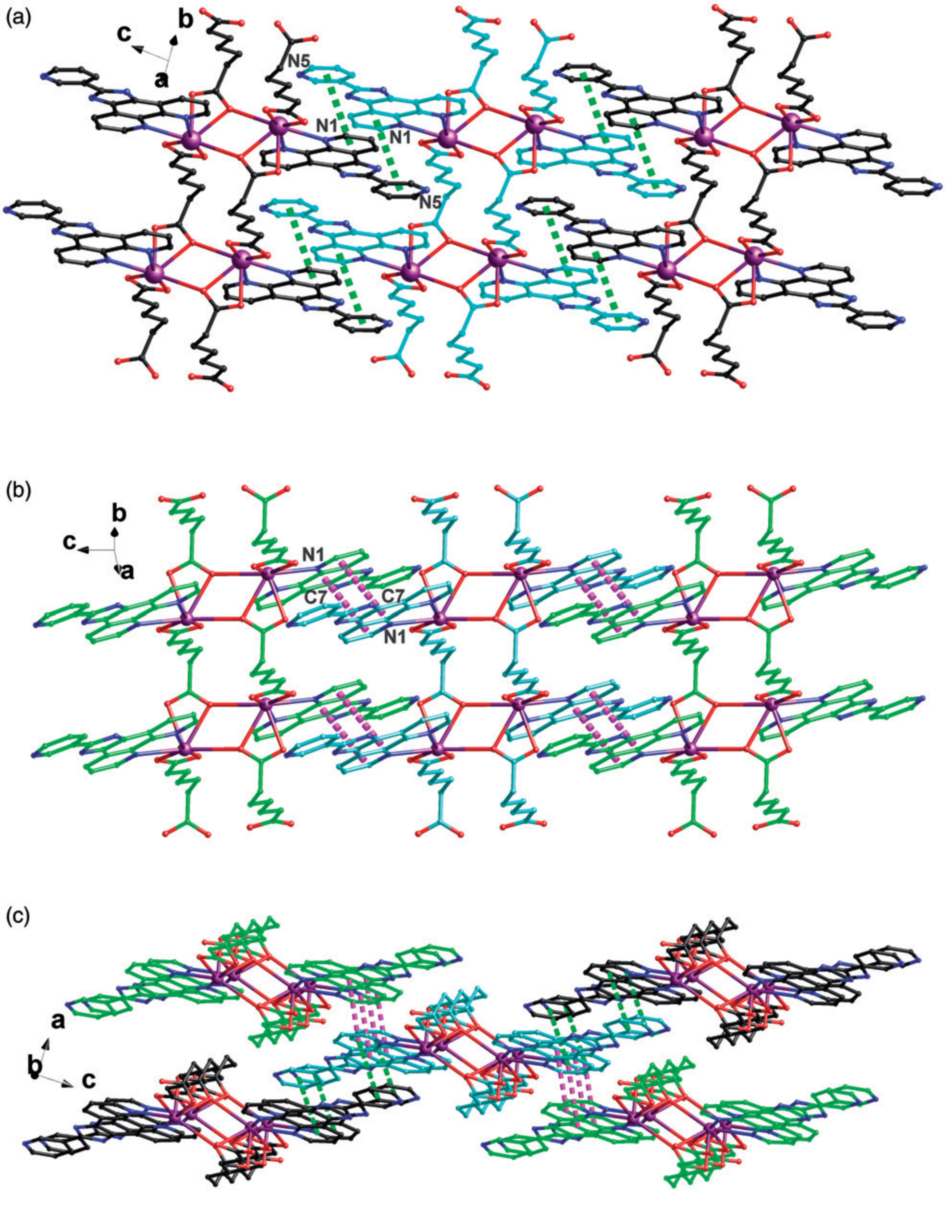


Figure 2. (a) The 2-D supramolecular layer extended through π–π stacking interactions between pyridine rings (containing N1 and N5), (b) the 2-D supramolecular layer extended *via* π–π stacking interactions from the pyridine rings (containing N1) and center phenyl of 3-pdip (containing C7), and (c) view of the 3-D supramolecular framework along the *b*-axis.

atoms of chelating–bridging carboxylates from two different L_2 anions. The distances of Pb–N are 2.619(7), 2.852(7), and 2.858(5) Å. The distances of Pb–O are 2.429(5)–2.941(7) Å, which are also near to the reported ones [24]. The O–Pb–O and N–Pb–O angles are in the range of 49.0(2)–167.2(2)° and 75.235(2)–132.965(2)°, respectively. The N–Pb–N angles vary from 59.52° to 154.714(2)°.

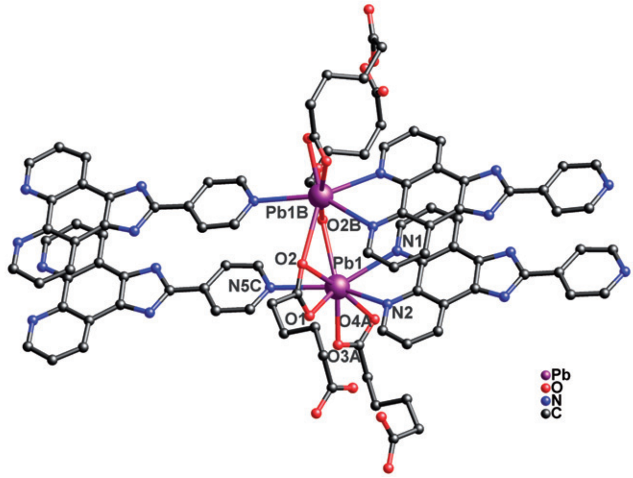


Figure 3. The coordination environment of Pb(II) in **2** (A: $-x+1/2$, $-y+1/2$, $-z+1$, B: $-x, y, -z+1/2$, C: $x, y+1, z$).

In **2**, the two μ_2 O2 of chelating–bridging carboxylates (μ_2–$\eta^2:\eta^1$ mode) from L_2 connect two adjacent Pb(II) centers, forming a binuclear [$Pb_2N_6O_8$] secondary building unit (SBU) with Pb$\cdots$Pb distance of 4.1973 Å (figure 4a). Two adjacent SBUs are connected by two L_2 anions to generate 1-D –[$Pb_2(L_2)_2$]$_n$– double chains extended along the *a* direction. The distance between cores of SBUs is 10.509 Å. The 1-D double chains are linked by pairs of 4-pdip ligands with parallel array mode to form a 2-D bi-layer coordination polymer and the distance between the cores of SBUs linked by 4-pdip ligands is 15.087 Å (figure 4b). Considering the binuclear SBU as a four-connected node, keeping $(L_2)_2$ and (4-pdip)$_2$ as the double spacers, **2** exhibits a (4,4) network (figure 4c). The 2-D bi-layer is stabilized by intramolecular π–π stacking interactions between aromatic rings of phenanthrolines from 4-pdip [Cg–Cg are 3.621(2) – 3.694(2) Å and the dihedral angles vary from 0.67° to 2.23°] (figure S2a). Furthermore, the 2-D networks are linked by intermolecular π–π stacking interactions between pyridine rings (containing N2) and the center phenyl ring of phenanthroline from 4-pdip to generate a 3-D supramolecular framework [Cg–Cg is 3.74(2) and the dihedral angle is 1.07°] (figure S2).

From the structure description, the effects of the organic-acid anions with different lengths and the N-containing ligands possessing different sites of donors on the structures of **1** and **2** have been clearly demonstrated. In **1** and **2**, Pb is hemidirected in **1** and holodirected in **2**, due to the different organic ligands used in these two reaction systems (figure 5a). L_1 anions link Pb's to form a rectangular 20-membered ring (9.8 × 10.9 Å) and a staircase-like metal–organic double chain in **1** (figure 5b top). While a shorter carboxylate (L_2) is used in **2**, a circular 18-membered ring (7.6 × 8.5 Å) is formed and a carriage-like metal–organic double chain is obtained (figure 5b bottom), which may be due to the different lengths and configurations of carboxylates. In **1**, 3-pdip shows chelating coordination as a terminal group and N5 does not coordinate to extend the 1-D double chain, perhaps due to the steric effect of 3-pyridine. When the site of pyridine was changed and 4-pdip with 4-pyridine group was used in **2**, it adopts a chelating–bridging coordination mode (μ_2–$\kappa N:\kappa N, N'$) and N5 of the 4-pyridine shows excellent ability in

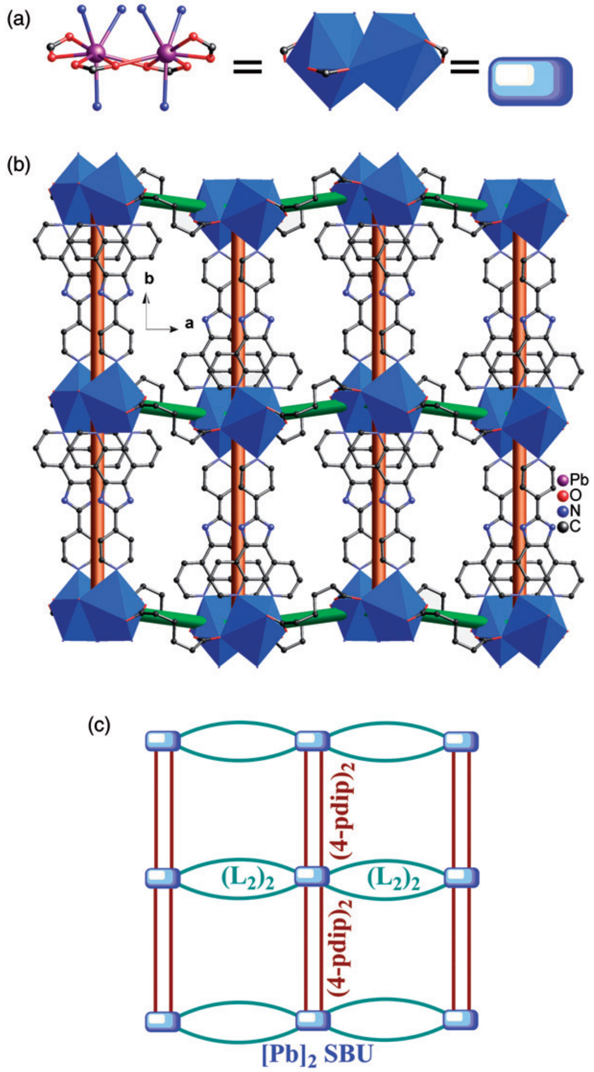


Figure 4. (a) View of the binuclear Pb(II) SBU in **2**, (b) 2-D (4,4) network along the *ab* plane, and (c) the schematic view of the 2-D network.

linking the Pb-carboxylate chain into a 2-D network. The results demonstrate that rational selection of ligands with different lengths and the site of N-donors is a good way to tune the structure of coordination polymers. Some Pb(II) complexes with 0-D, 1-D, 2-D, and 3-D architectures have been constructed from carboxylates by Xu, Morsali, and Che [25–27]. However, the examples derived from 1,10-phenanthroline derivatives with different pyridine groups are limited [24].

3.2. *IR spectra*

The main features in IR spectra of **1** and **2** concern the N-containing ligands and carboxylates. Bands at 800 and 737 cm^{-1} may be attributed to $\nu_{C\text{–}N}$ of the

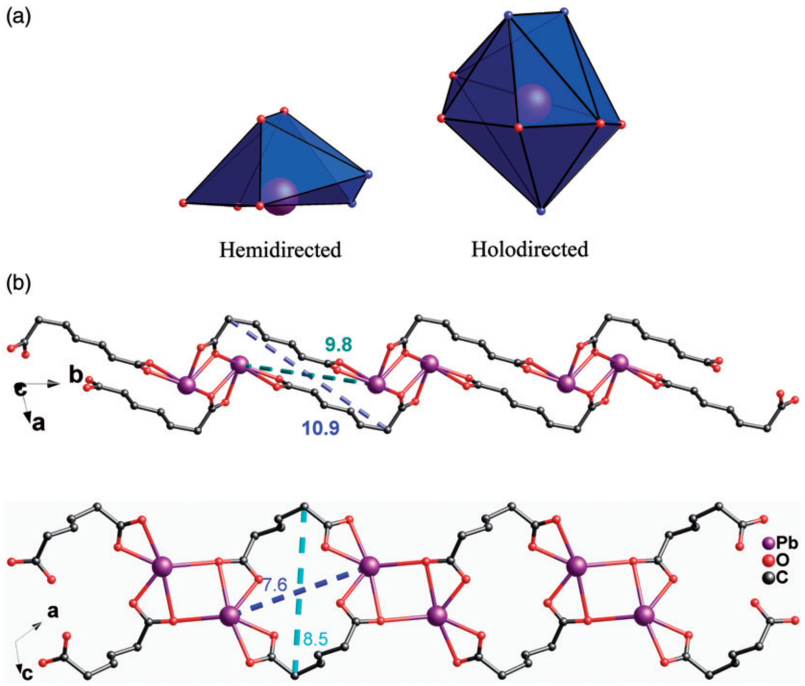


Figure 5. (a) Lone-pair stereochemical properties and (b) the 1-D staircase-like (top) and carriage-like (bottom) metal-carboxylate double chains in **1** and **2**.

N–heterocyclic rings from 3-pdip and 4-pdip [14–19]. No strong absorptions around 1700 cm^{-1} for –COOH are observed, indicating that all carboxyl groups in **1** and **2** are deprotonated [28]. For **1**, the asymmetric and symmetric vibrations appear at 1614, 1551, and 1387 cm^{-1}, respectively. The Δ (227 and 164 cm^{-1}) reflects the coordination modes of carboxylate in **1**. For **2**, carboxylate bands appear at 1611, 1549, and 1385 cm^{-1} for the asymmetric and symmetric vibrations, respectively. The Δ is 226 and 164 cm^{-1}, in agreement with the results of the crystal structure [29]. Similar IR spectra of **1** and **2** may be attributed to the same coordination modes of carboxylates and similar N-containing ligands in **1** and **2** [30]. The band at 3391 cm^{-1} in **1** indicates lattice water [31].

3.3. *Thermal properties*

To examine the thermal stability of **1** and **2**, decomposition behaviors were examined by TGA with a heating rate of 10°C min^{-1} from 30°C to 600°C, as shown in figure S3. The TG curve of **1** exhibits three weight loss stages. The first of 1.35% at 80–115°C corresponds to release of lattice water (Calcd 1.34%). The second and third weight losses at 250–435°C can be attributed to the decomposition of the framework, forming PbO as a final product (Observed 33.20%, Calcd 33.23%). In comparison with **1**, **2** is slightly more stable (to 340°C), where decomposition of the framework starts a rapid and significant weight loss of 65.57% from 340°C to 520°C, and the resulting residue is PbO (Observed 34.43%, Calcd 34.41%). The different decomposition temperature for the frameworks may be due to the difference of their structural dimensionality [32].

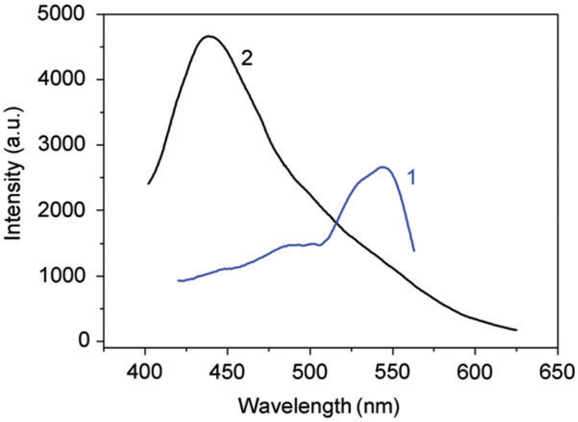


Figure 6. Fluorescence spectra for **1** and **2** in the solid state at room temperature.

3.4. *Photoluminescence properties*

We have examined the photoluminescent properties of **1** and **2** in the solid state at room temperature due to excellent luminescent properties of phenanthroline derivatives, as shown in figure 6. Complex **1** exhibits green photoluminescence with an emission maximum at *ca* 543 nm upon excitation at 341 nm. Free 3-pdip displays a photoluminescent emission at 422 nm upon excitation at 260 nm in the solid state [18]. Comparing with the emission of 3-pdip, a red shift of *ca* 120 nm for **1** has been observed. Therefore, the emission band for **1** can be mainly assigned to a $\pi^* \rightarrow \pi$ transition of coordinated 3-pdip [19]. The emission maximum of **2** was observed at 438 nm upon excitation at 335 nm. Comparing with the emission of 4-pdip ($\lambda_{ex} = 360$ nm, $\lambda_{em} = 535$ nm), a blue shift of *ca* 100 nm for **2** is observed. Therefore, the emission band for **2** may be ascribed to ligand-to-metal charge transfer [19]. The different emission bands of **1** and **2** may be due to the different phenanthroline derivatives [32].

4. Conclusion

We have synthesized two new coordination polymers constructed from Pb(II), flexible fatty carboxylic acids and phenanthroline derivatives. Complex **1** is a 1-D double chain coordination polymer, whereas **2** features a 2-D (4,4) network. Structural analyses indicate that the organic carboxylic anions with different lengths and the N-containing ligands with different N-donor sites have significant effects on the formation and dimensionality of the resulting structures. The blue/green fluorescent properties and excellent thermal stability suggest that **1** and **2** may be used as luminescent materials.

Supplementary material

CCDC 848470 for **1** and 848471 for **2** contain the supplementary crystallographic data for this article. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif

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