

Plumbum(II) and Zinc(II) Complexes with 5-Chlorosalicylic Acid: Structure and Property

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Two new transition metal complexes, containing the acidic ligand 5-chlorosalicylic acid (HL5), and 1,10-phenanthroline (phen) and 1,3-di(4-pyridyl)propane (bpp) as secondary ligands are reported, these two are formulated as $\{[\text{Pb}(\text{HL5})_2(\text{phen})(\text{H}_2\text{O})]\cdot\text{H}_2\text{O}\}_n$ (1), and $[\text{Zn}_2(\text{HL5})_4(\text{bpp})_2]_n$ (2), and are characterized by single-crystal X-ray diffraction. Compound 1 crystallizes in the triclinic *P-1* space group while compound 2 in the monoclinic system with space group *C2/c*. In compound 1, the carboxylate group of the coordinated HL5⁻ adopt κ^2O,O' coordination fashion to connect one Pb(II) ion but all the carboxylate groups adopt the different $\mu_1-\eta^1: \eta^1$ mode in the crystal of compound 2 compared to 1. The solid-state photoluminescent properties at room temperature and thermal decomposition process of complexes were investigated.

Keywords: Transition metal complexes, Single-crystal X-ray diffractions, Solid-state photoluminescent properties, Thermal decomposition process

INTRODUCTION

In recent years, the rational design and synthesis of metal-carboxylate complexes are one of the most attraction areas of materials research. The intense interests in this area are driven by not only their aesthetically beautiful architectures and topologies but also their potential applications in magnetism, luminescence, absorption, and chemical sensing [1-5]. Despite some recent advancement, the ability to predict and control the supramolecular assembly remains a long-standing challenge, and much more elaborate and systematic work is required to comprehend the inter- and intramolecular forces that determine the fashions of molecular structure and crystal packing in the solid state [6-14]. Recently, salicylic derivatives have attracted considerable attention not only for their structural varieties but also for their biological applications and spectroscopic and electrochemical behavior [15]. In

addition, the carboxylate group and hydroxyl can partly or entirely be deprotonated, which makes it possible to coordinate to the metal ion or participate in hydrogen bonding interactions [16]. The carboxyl group has various coordination motifs dependent on the reaction conditions, such as temperature, solvent, and auxiliary ligand. In the previous studies, we found that the pH value would play an important influence on the structure of the solid product [17]. Also, *N*-heterocyclic ligand are usually used as ancillary ligands, chelating bipyridine-like ligands such as 1,10-phenanthroline and 2,2'-bipyridine may provide supramolecular recognition sites for $\pi-\pi$ stacking interactions to form interesting supramolecular structures, and bridging bipyridine-like ligands such as 4,4'-pyridine and 1,3-di(4-pyridyl)propane can generate a 1D infinite zigzag chain through M-N coordination bonds [18,19]. The chemistry of metal-organic molecular architectures of bpy-like and carboxylate ligands has been reviewed [20].

Herein, we report two new coordination polymers, $\{[\text{Pb}(\text{HL5})_2(\text{phen})(\text{H}_2\text{O})]\cdot\text{H}_2\text{O}\}_n$ (1) and $[\text{Zn}_2(\text{HL5})_4(\text{bpp})_2]_n$

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(2), prepared by the reaction of plumbum(II) and zinc(II) salts with 5-chlorosalicylic acid and N-donor ligands. Furthermore, the fluorescence property in the solid state and thermal decomposition process were investigated in order to understand the relationships between the structures and the properties.

EXPERIMENTAL

All chemicals were commercial materials of analytical grade and used without purification. Elemental analysis for C, H and N was carried out on a Perkin-Elmer 2400 II elemental analyzer. The FT-IR spectrum was obtained on a PE Spectrum One FT-IR spectrophotometer in the 4000-400 cm^{-1} regions, using KBr pellets. Fluorescence spectra were recorded with F-2500 FL spectrophotometer analyzer.

Preparation of $\{[\text{Pb}(\text{HL5})_2(\text{phen})(\text{H}_2\text{O})]\cdot\text{H}_2\text{O}\}_n$ (1). The reagents of $\text{Pb}(\text{NO}_3)_2$ (0.1656 g, 0.5 mmol), $\text{H}_2\text{L5}$ (0.0863 g, 0.5 mmol), phen (0.0991 g, 0.5 mmol) were dissolved in 18 ml mixed solvent of $\text{H}_2\text{O}/\text{MeOH}/\text{DMF}$ (volume ratio 4:3:2) followed by three drops of KOH aqueous solution (1 M) and then stirred for 0.5 h. The resulting mixture was sealed in a 25 ml Teflon-lined stainless container and heated at 145 °C for 72 h, and then slowly cooled to room temperature at a rate of 20 °C per hour. The yellow block crystals suitable for X-ray diffraction were isolated directly, Yield: 70 %, based on Pb. Anal. Calcd. for $\text{C}_{26}\text{H}_{20}\text{N}_2\text{Cl}_2\text{O}_8\text{Pb}$ (%): C, 40.74; H, 2.63; N, 3.65; Cl, 9.25. Found: C, 40.71; H, 2.67; N, 3.60; Cl, 9.31; IR data (KBr pellets, cm^{-1}): 3438(w), 1618(m), 1579(m), 1547(m), 1512(m), 1469(s), 1421(s), 1362(s), 1283(m), 1241(s), 1111(vs), 893(w), 860(w), 837(w), 821(m), 717(m), 616(vs), 535(w), 417(w).

Preparation of $[\text{Zn}_2(\text{HL5})_4(\text{bpp})_2]_n$ (2). A mixture of $\text{Zn}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$ (0.1487 g, 0.5 mmol), $\text{H}_2\text{L5}$ (0.0863 g, 0.5 mmol), and bpp (0.0991 g, 0.5 mmol) was dissolved in 9 ml $\text{DMF}/\text{H}_2\text{O}$ (1/2, v/v). The final mixture was placed in a Parr Teflon-lined stainless vessel and heated at 135 °C for 3 days, and then slowly cooled to room temperature at a rate of 20 °C per hour. Yellow block crystals were obtained. Yield of the reaction was *ca.* 64% based on Zn. Anal. Calcd. for $\text{C}_{54}\text{H}_{44}\text{N}_4\text{Cl}_4\text{O}_{12}\text{Zn}_2$ (%): C, 53.45; H, 3.65; N, 4.62; Cl, 11.69. Found: C, 53.39; H, 3.61; N, 4.57; Cl, 11.58. IR (KBr pellets, cm^{-1}): 3431(w), 3063(w), 2957(w), 2923(w),

1617(m), 1585(m), 1566(m), 1505(w), 1474(m), 1420(s), 1386(m), 1359(m), 1286(m), 1247(m), 1215(m), 1109(vs), 1028(m), 904(w), 893(w), 826(m), 717(m), 616(s), 540(w), 523(w), 506(w), 456(w), 420(w).

X-Ray Data Collection and Structure Refinement

A high quality crystal of each complex was selected and mounted on the top of a glass fiber. The data were collected on a Bruker SMART CCD diffractometer at 293(2) K. Using graphite-monochromatized Mo $\text{K}\alpha$ radiation ($\lambda = 0.71069$ Å). Absorption corrections were applied using SADABS [21]. All structures were solved by direct methods and refined by full-matrix least squares on F^2 using the SHELXTL-97 program package [22]. All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms attached to carbon were placed in geometrically idealized position and refined using a riding model. The crystallographic data as well as details of the data collection and refinement for the complex are listed in Table 1. Selected bond lengths and angles are given in Table 2, and possible hydrogen bond geometries are given in Table 3.

RESULTS AND DISCUSSION

Crystal Structure Descriptions

The single-crystal X-ray diffraction analysis reveals that compound 1 crystallizes in the triclinic system, space group of $P\bar{1}$. The asymmetric unit of 1 consists of one Pb(II) ion in the center of the structure, two HL5^- , one phen ligand, one coordinated water molecule and a free water molecule. As depicted in Fig. 1, the Pb1 ion possesses a seven-coordinated sphere via coordinating with four oxygen atoms (O3, O4, O6, O7) from two $\text{H}_2\text{L5}$ and one oxygen atom (O2) from one water molecule as well as two nitrogen atoms (N1, N2) from one phen composing the equatorial plane, in a slightly distorted decahedral geometry (Fig. 2). The Pb-O bond distances vary from 2.468(4) to 2.807(5) Å, while the Pb-N bond lengths are 2.574(5) and 2.626(5) Å. In 1, two carboxylate groups of HL5^- ligand adopts $\kappa^2\text{O}, \text{O}'$ coordination mode and the dihedral angle between two benzene rings is 93.66°. The structure is stabilized by intermolecular O-H...O hydrogen bond (Table 3, Fig. 3). The most fascinating feature of crystalline 1 is the presence

Table 1. Crystallographic Data for Polymers 1 and 2

Polymer	1	2
Formula	C ₂₆ H ₂₀ Cl ₂ N ₂ O ₈ Pb	C ₅₄ H ₄₄ Cl ₄ N ₄ O ₁₂ Zn ₂
Formula weight	766.54	1213.47
Crystal system	Triclinic	Monoclinic
Space group	<i>P</i> -1	<i>C</i> 2/ <i>c</i>
<i>a</i> (Å)	9.141(2)	21.727
<i>b</i> (Å)	12.810(3)	14.185
<i>c</i> (Å)	13.023(3)	18.321
α (°)	63.427(3)	90
β (°)	79.428(3)	108.10
γ (°)	77.298(3)	90
Volume (Å ³)	1324.1(5)	5367.2
D _{calc.} (mg m ⁻³)	1.923	1.502
<i>Z</i>	2	4
Absorption coefficient (mm ⁻¹)	6.626	1.159
<i>F</i> (000)	740	2480
Crystal size (mm ³)	0.33 × 0.25 × 0.18	0.24 × 0.23 × 0.22
θ range (°)	1.76-25.00	1.74-25.00
Reflections collected	7219	13804
Unique reflections	4611 [R(int.) = 0.0222]	4727 [R(int.) = 0.0503]
Completeness to $\theta = 25.00$	98.6%	99.9 %
Max. and min. transmission	0.8214 and 0.6054	0.8013 and 0.5950
Data/restraints/parameters	4611/0/3530	4727/0/345
GOF on <i>F</i> ²	1.068	1.045
<i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	R1 = 0.0285, wR2 = 0.0766	R1 = 0.0473, wR2 = 0.1203
<i>R</i> indices (all data)	R1 = 0.0335, wR2 = 0.0791	R1 = 0.0617, wR2 = 0.1289
Largest diff. peak and hole (e Å ⁻³)	2.082 and -0.754	0.788 and -0.594

Table 2. Selected Bond Lengths (Å) and Angles (°) for 1 and 2

1			
Pb(1)-O(4)	2.468(4)	Pb(1)-N(2)	2.573(5)
Pb(1)-N(1)	2.626(4)	Pb(1)-O(3)	2.633(4)
Pb(1)-O(7)	2.679(4)	Pb(1)-O(2)	2.781(4)
Pb(1)-O(6)	2.807(4)	Pb(1)-O(7)#1	2.978(5)
O(4)-Pb(1)-N(2)	83.27(15)	O(4)-Pb(1)-N(1)	77.65(13)
N(2)-Pb(1)-N(1)	63.65(15)	O(4)-Pb(1)-O(3)	51.12(12)
N(2)-Pb(1)-O(3)	79.06(15)	N(1)-Pb(1)-O(3)	119.63(13)
O(4)-Pb(1)-O(7)	83.43(14)	N(2)-Pb(1)-O(7)	140.65(14)
N(1)-Pb(1)-O(7)	77.37(13)	O(3)-Pb(1)-O(7)	118.83(13)
O(4)-Pb(1)-O(2)	123.36(13)	N(2)-Pb(1)-O(2)	83.78(16)
N(1)-Pb(1)-O(2)	139.82(15)	O(3)-Pb(1)-O(2)	72.32(13)
O(7)-Pb(1)-O(2)	133.67(15)	O(4)-Pb(1)-O(6)	84.76(14)
N(2)-Pb(1)-O(6)	163.90(14)	N(1)-Pb(1)-O(6)	123.88(13)
O(3)-Pb(1)-O(6)	85.05(13)	O(7)-Pb(1)-O(6)	47.66(12)
O(2)-Pb(1)-O(6)	93.90(15)	O(4)-Pb(1)-O(7)#1	153.10(12)
N(2)-Pb(1)-O(7)#1	93.50(13)	N(1)-Pb(1)-O(7)#1	77.03(12)
O(3)-Pb(1)-O(7)#1	154.32(12)	O(7)-Pb(1)-O(7)#1	82.35(13)
O(2)-Pb(1)-O(7)#1	82.51(13)	O(6)-Pb(1)-O(7)#1	102.02(11)
2			
Zn(1)-O(3)	1.957(2)	Zn(1)-O(4)	1.986(2)
Zn(1)-N(2)#1	2.066(3)	Zn(1)-N(1)	2.072(3)
O(3)-Zn(1)-O(4)	137.48(11)	O(3)-Zn(1)-N(2)#1	106.54(11)
O(4)-Zn(1)-N(2)#1	99.76(10)	O(3)-Zn(1)-N(1)	96.56(10)
O(4)-Zn(1)-N(1)	107.03(10)	N(2)#1-Zn(1)-N(1)	107.05(10)

Symmetry transformations used to generate equivalent atoms: for polymer 1: #1 - x, -y + 1, -z + 1; for polymer 2: #1x + 1/2, y + 1/2, z.

Table 3. Hydrogen-bond Geometry (Å) for Polymers 1 and 2

D-H...A	D-H	H...A	D...A	D-H...A
Polymer 1				
O1-H1C...O4	0.85	2.12	2.909(7)	154
O1-H1D...O7	0.85	2.27	3.054(7)	154
O2-H2C...O1	0.85	1.96	2.806(7)	178
O2-H2D...O5	0.85	2.13	2.980(8)	178
O5-H5...O3	0.82	1.81	2.535(7)	147
O8-H8...O6	0.82	1.83	2.541(7)	144
C10-H10...O8	0.93	2.45	3.357(11)	164
C12-H12...O7	0.93	2.42	3.153(10)	135
C23-H23...O6	0.93	2.53	3.416(8)	160
Polymer 2				
O1-H1...O2	0.82	1.85	2.574(4)	147
O6-H6...O5	0.82	1.83	2.552(4)	146
C2-H2...O2	0.93	2.57	3.371(5)	145
C3-H3...O1	0.93	2.53	3.449(6)	168

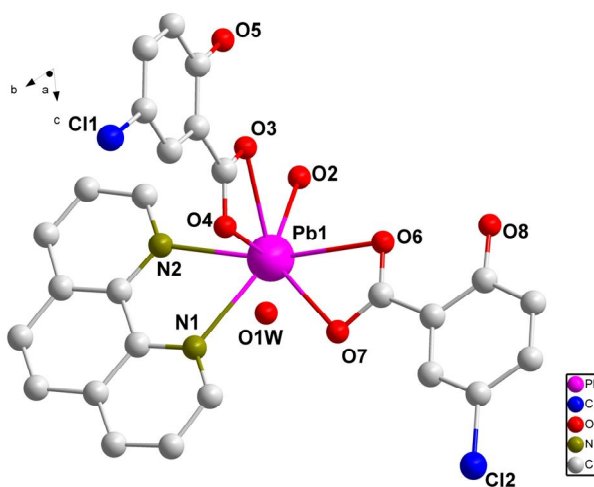


Fig. 1. The coordination environment of Pb(II) ion of the complex 1. All the hydrogen atoms are omitted for clarity

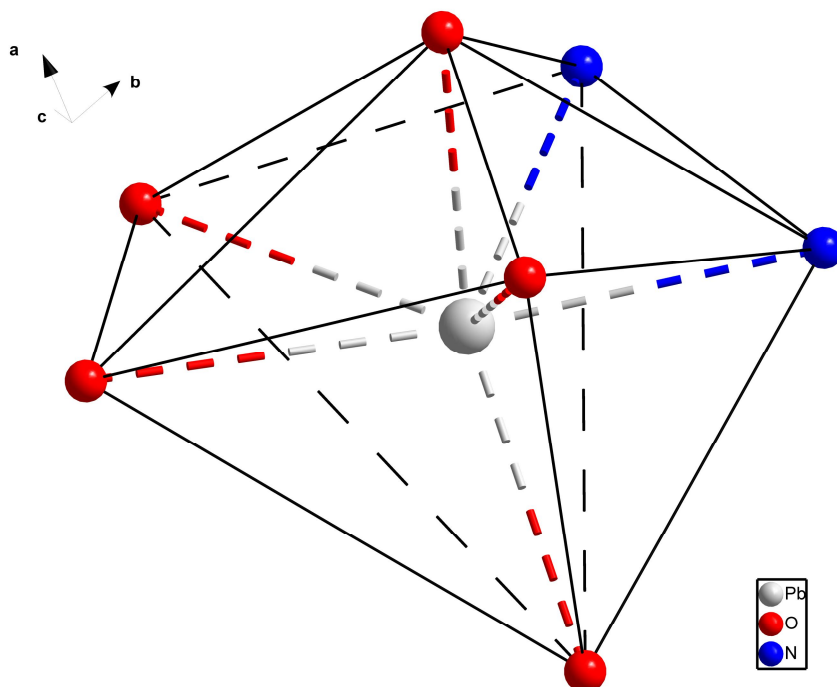


Fig. 2. The coordination polyhedron of the Pb(II) ion of complex 1. Unnecessary atoms are omitted for clarity.

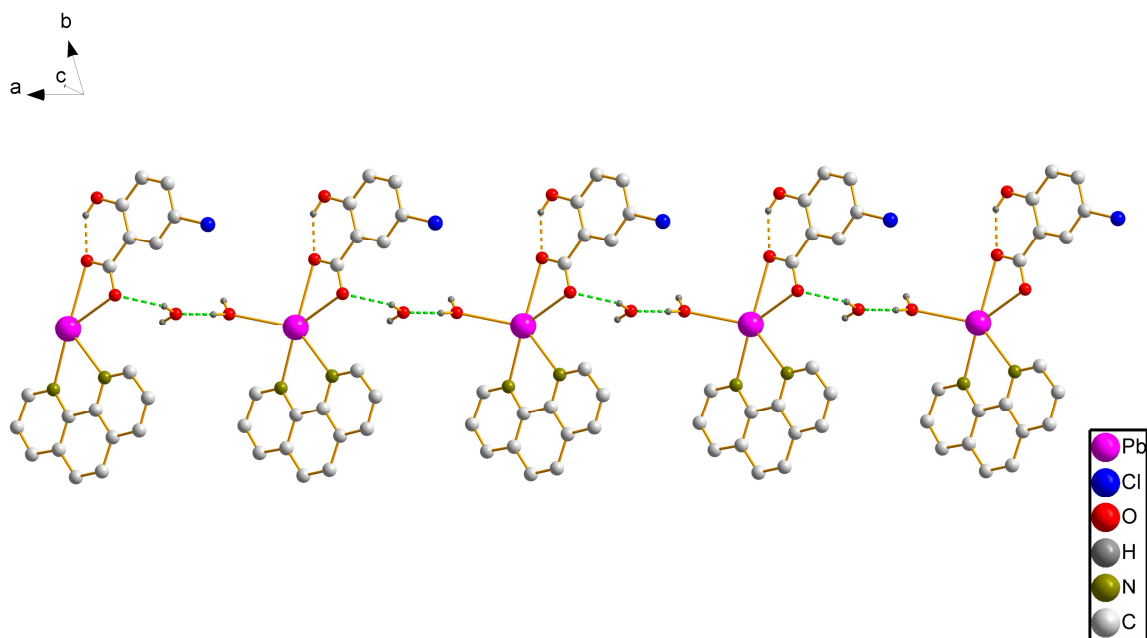


Fig. 3. Infinite 1D chain of 1 connected by O-H...O hydrogen bonds. Unnecessary atoms are omitted for clarity.

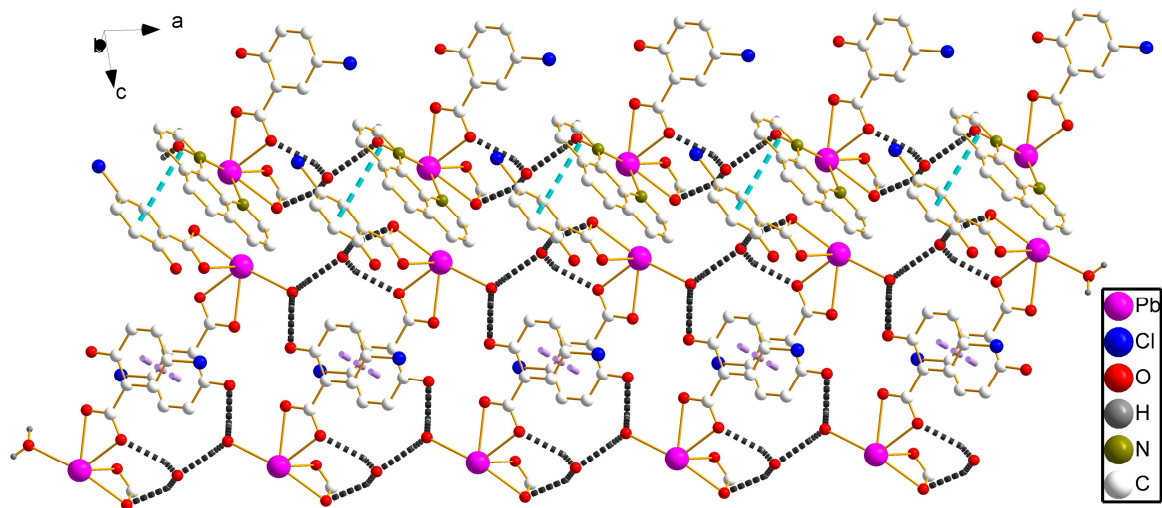


Fig. 4. View of π - π stacking interactions and H-bonding of complex 1. Unnecessary atoms are omitted for clarity.

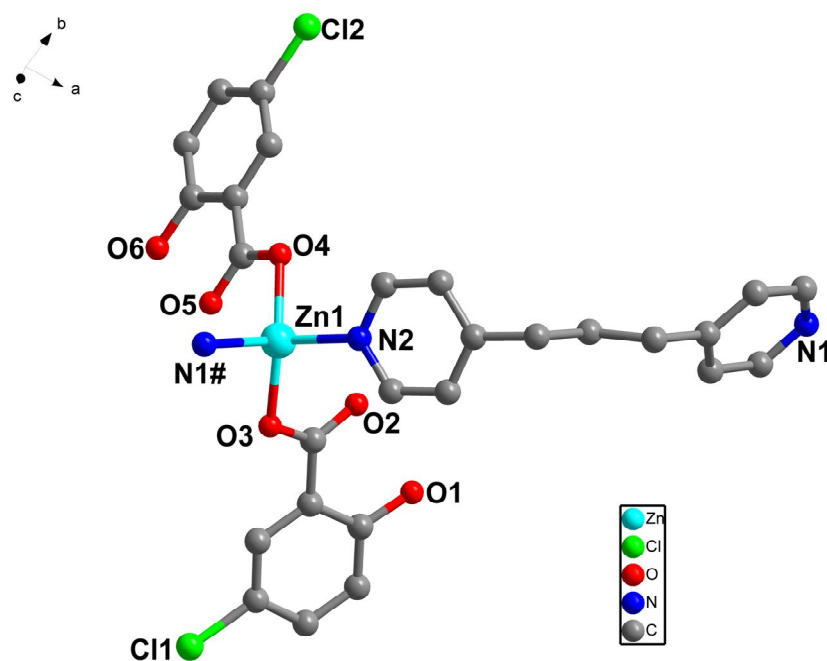


Fig. 5. The coordination environment of Zn(II) ion of the complex 2. All the hydrogen atoms are omitted for clarity.

of two kinds of alternant face-to-face π - π stacking between the adjacent 1,10-phenanthroline and between the adjacent HL5, which π - π stacking play a important part in the structure construction. The complex packed into a 2D layered structure *via* perfect face-to-face π - π stacking interactions and O-H \cdots O hydrogen bonds (Fig. 4).

A single-crystal X-ray diffraction study reveals that 2 crystallizes in the monoclinic system with space group $C2/c$. As shown in Fig. 5, asymmetric unit of 2 is composed of one Zn center, two HL5⁻ anions, and two bpp ligands. The Zn center is four-coordinated by two single dentate bridging carboxylate oxygen atoms from two HL5⁻ ligands, two pyridyl nitrogen atoms from the bpp ligands. The geometry around this Zn(II) can be described as a distorted tetrahedron (Fig. 6). As for the deprotonated HL5⁻ ligands, all the carboxylate groups adopt the different μ_1 - η^1 : η^1 mode in the crystal of compound 2 compared to 1. As shown in Fig. 7, Zn centers are linked together by bpp, through Zn-N coordination bonds to generate a 1D infinite zigzag chain [Zn(bpp)]_n with Zn \cdots Zn distance of 12.974(2) Å. Moreover, the π - π stacking of HL5 and bpp or HL5 with the centroid-centroid distances of 3.65 and 3.88 Å seem to be effective in the stabilization of the structure, resulting in the formation of a 2D supramolecular network (Fig. 8).

IR Spectra

In the IR spectra of two complexes, the broad bands at 3438 cm⁻¹ which can be attribute to the coordination water molecules' O-H stretching vibration for 1. The COO⁻ is coordinated with its asymmetric and symmetric stretching appearing at 1618 and 1362 cm⁻¹ in 1, 1617 and 1386 cm⁻¹ in 2, For polymer 1, the medium intensity peak at 1512 cm⁻¹ is attributed for C=N, additional peaks at 1241 and 616 cm⁻¹ are attributed for C-OH and C-Cl. For polymer 2, the strong peak at 1420 is attributed for C=N, additional peaks at 1247 and 616 cm⁻¹ is consistent with C-OH and C-Cl.

Thermogravimetric (TG) Analyses

Polymers 1 and 2 are air stable and can retain their structural integrity at room temperature for a considerable length of time; thus, TGA was conducted to determine the thermal stability of these crystalline materials, and the TG curves are shown in Fig. 9. The TG curve of 1 shows the first loss of 4.61% in the temperature range of 94-150 °C,

which indicates the exclusion of free water and coordinated water molecules (Calcd., 4.71%); The second stage occurs between 250 and 380 °C, the anhydrous polymer loses 21.66% of total weight, which is due to the decomposition of phen (Calcd., 23.51%). When the temperature holds on rising, the product lost 42.87% of the total weight in the temperature range of 380-750 °C, which is related to the loss of HL5⁻ (Calcd., 44.76%). Then the remaining weight is assigned to PbO (Obsd., 27.55%; Calcd., 29.12%).

As there is no water molecules in the crystal of polymer 2 compared to 1, it shows higher thermal stability owing to the facile release of water molecules from the framework. The TGA curve of 2 shows two main weight losses. The first one occurs in the temperature range of 150-355 °C corresponding to the release of two HL5⁻ per formula unit. The observed weight loss (55.03%) is in agreement with the calculated one (56.56%). Upon temperature increase, the remaining sample suffers incessant weight loss at 350-850 cm⁻¹, with a total of 30.89% which corresponds to the removal of bpp (Calcd., 32.68%). The residual weight percentage (Obsd., 15.22%) at the end of the decomposition of the complex is consistent with the formation of ZnO (Calcd., 13.41%).

CONCLUSIONS

This paper reports the synthesis and characterization of complexes {[Pb(HL5)₂(phen)(H₂O)]·H₂O}_n and [Zn₂(HL5)₄(bpp)₂]_n by IR method, elemental analysis, fluorescence spectroscopy method, and single-crystal X-ray diffraction techniques. The above result shows that the configuration of ligand plays an important role in affecting the final structure of the coordination polymers. The successful preparation of the complexes manifest that the conformations and functions of π - π stacking interactions, hydrogen bonds and van der Waals' forces are important factors in influencing the architecture of metal-ligand complex. In addition, compound 1 and 2 exhibit good fluorescence in the solution of methanol at room temperature.

SUPPLEMENTARY MATERIAL

Crystallographic data for the structures reported here have been deposited with CCDC (Deposition No. CCDC-

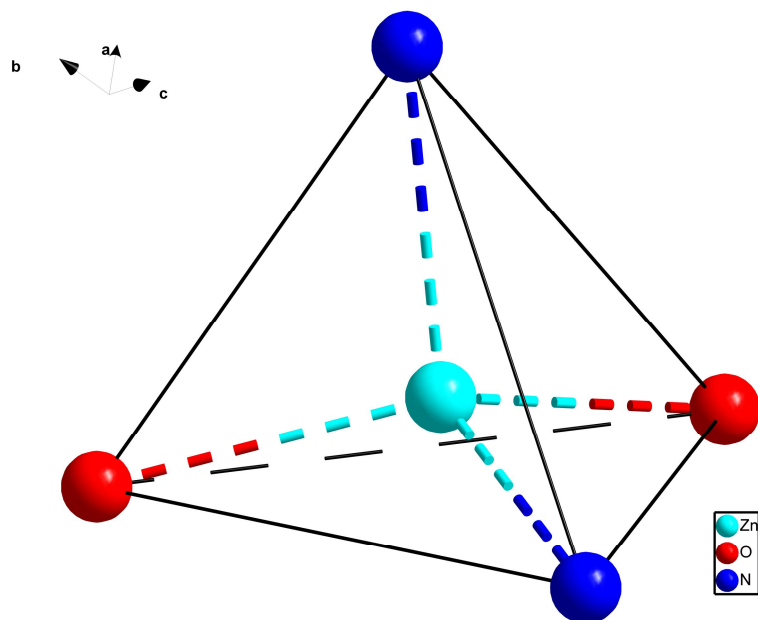


Fig. 6. The coordination polyhedron of the Zn(II) ion of complex 2. Unnecessary atoms are omitted for clarity.

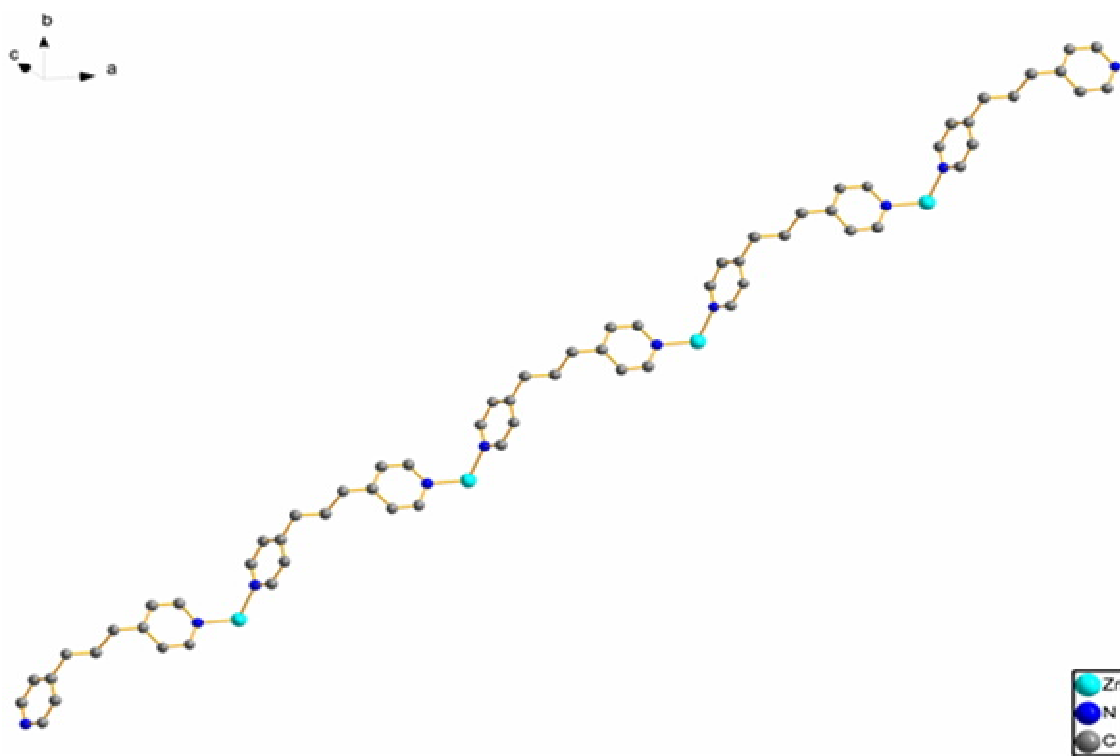


Fig. 7. One-dimensional zigzag chain $[Zn(bpp)]_n$ of polymer 2. Unnecessary atoms are omitted for clarity.

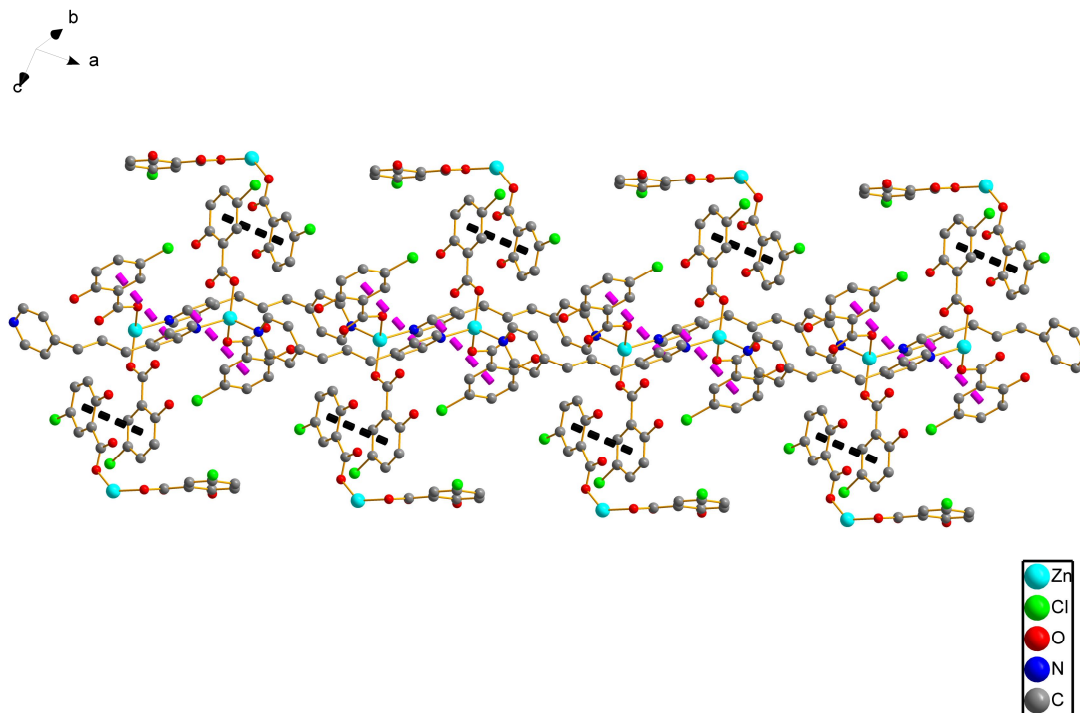


Fig. 8. View of the π - π stacking interactions of 2. Unnecessary atoms are omitted for clarity.

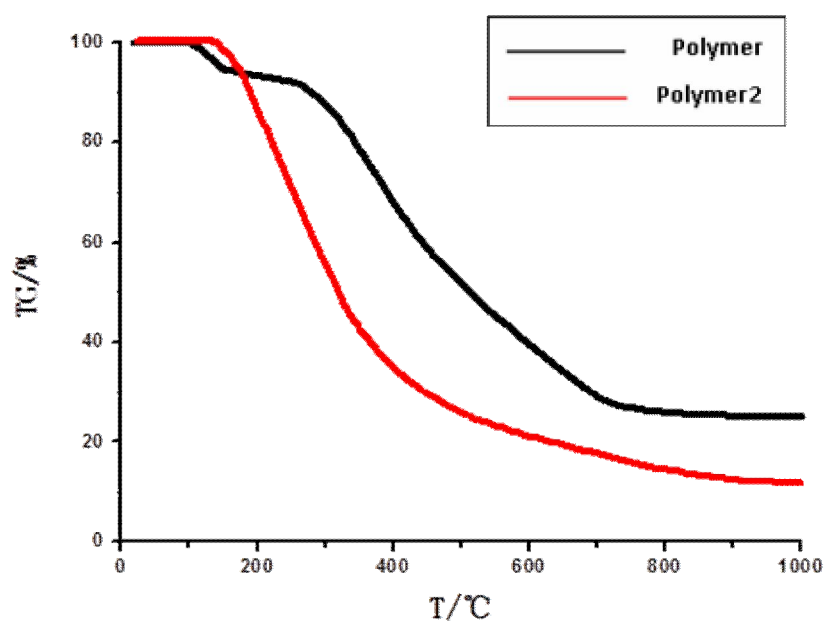


Fig. 9. The TG curves for complexes 1-2.

997743 (1), No. CCDC-1036509 (2)). These data can be obtained free of charge *via* <http://www.ccdc.cam.ac.uk/conts/retrieving.html> or from CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, E-mail: deposit@ccdc.cam.ac.uk.

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