

A Series of Coordination Polymers Constructed by 2-Phenylsuccinic Acid and Flexible Bis(imidazole) Ligands: Syntheses, Structures, and Photoluminescent Properties

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Abstract

Eight coordination polymers (CPs), namely, $[\text{Co}(\text{m-phsuc})(\text{m-obix})]_n$ (**1**), $\{[\text{Co}_2(\text{m-phsuc})_2(\text{m-pbix})_{1.5}] \times 2\text{H}_2\text{O}\}_n$ (**2**), $[\text{Zn}(\text{m-phsuc})(\text{m-obix})]_n$ (**3**), $\{[\text{Zn}(\text{m-phsuc})(\text{m-mbix})] \times \text{H}_2\text{O}\}_n$ (**4**), $\{[\text{Zn}(\text{m-phsuc})(\text{m-pbix})] \times \text{H}_2\text{O}\}_n$ (**5**), $\{[\text{Zn}(\text{m-phsuc})(\text{m-pbix})] \times 1.5\text{H}_2\text{O} \times \text{CH}_3\text{OH}\}_n$ (**6**), $[\text{Cd}(\text{m-phsuc})(\text{m-obix})]_n$ (**7**) and $[\text{Cd}(\text{m}_3\text{-phsuc})(\text{m-pbix})_{0.5}]_n$ (**8**) (phsucH₂ = 2-phenylsuccinic acid, obix = 1,2-bis((imidazol-1-yl)methyl)benzene, mbix = 1,3-bis((imidazol-1-yl)methyl)benzene, pbix = 1,4-bis((imidazol-1-yl)methyl)benzene), were solvothermally synthesized and characterized by IR spectroscopy, elemental analysis, X-ray single-crystal diffraction, X-ray powder diffraction (PXRD), and thermal analyses. The structural analyses revealed that the compounds were 2D coordination polymers, in which metal ions were in tetrahedral coordination geometries in **1-6** and in octahedral coordination geometries in **2, 7** and **8**. Compounds **1, 3, 4, 5, 7** and **8** displayed a 4-connected 2D wave sheets with the point symbol of $\{4^4.6^2\}$. Compound **2** was rare example exhibiting a 2D polyrotaxane structure. Compound **6** showed a 2D two-fold interpenetrating 3-connected hcb net with the point symbol of $\{6^3\}$. In the compounds, the neighboring 2D layers were further assembled by p××p stacking and weak C–H××p interactions into the 3D supramolecular framework. The effects of the bis(imidazole) linkers and metals ions on the framework assemblies have been discussed. In addition, thermal and photoluminescent properties of the compounds were investigated.

1. Introduction

The design and construction of coordination polymers (CPs) have been drawn attention by researchers in the field of crystal engineering owing to their intriguing architectures and application areas such as gas adsorption, sensor, magnetism, luminescence, dye removal, etc [1–7]. In the construction of CPs with well-defined architecture, the selection of appropriate ligands is a useful strategy. However, in addition to metal ions and ligands, reaction conditions, the solvent can also affect the final structure of CPs [8, 9].

As known, rigid or flexible carboxylic acids have been widely utilized to provide charge balance of metal ions in the construction of CPs [10]. Especially; flexible ligands have been preferred to obtain an unpredicted structure with structural diversity [11, 12]. Recently, the combination of flexible N-donor ligands and flexible dicarboxylic acids (the mixed ligand strategy) has been widely employed to build unexpected CPs with intriguing architectures [13, 14]. In the literature, the coordination polymers synthesized with flexible dicarboxylic acids such as succinic acid, glutaric acid, adipic acid, etc, have been well documented and the ligands displayed several conformations to connect to metal ions resulting in versalite structures [15–19]. In our previous studies, we used glutaric acid and substitute glutaric acid with semi-flexible bis(imidazole) linkers in the synthesis of coordination polymers. They showed diverse coordination modes and conformations in the CPs with various dimensions [20–22]. In this study, 2-phenylsuccinic acid (phsucH₂) containing phenyl side group, which was an asymmetric ligand, was used as an anionic linker. The phenyl side group on the succinate backbone can affect the coordination ability of the carboxylate group, orientation of the aliphatic skeleton, induce conformation ability, and reduce dimensionality in its compounds [23–25]. In the literature, some coordination polymers

with phsucH₂ were synthesized and characterized [25–28]. However, there has been still need to investigate the effect of phenyl side group on succinate backbone on its compounds. Moreover, as an auxiliary ligand, we used semi-isomeric bis(imidazole) linker in the mixed-ligand assembly to investigate the effect of mixed ligand strategy on the dimensionality of phsuc²⁻ coordination polymers and also isomeric neutral ligands on the final structure of CPs. The imidazole rings of bis(imidazole) ligand around –CH₂– groups freely rotate to show various conformations, leading to interesting intriguing architecture [29].

In this context, eight coordination polymers, [Co(μ-phsuc)(μ-obix)]_n (**1**), {[Co₂(μ-phsuc)₂(μ-pbix)_{1.5}]·2H₂O}_n (**2**), [Zn(μ-phsuc)(μ-obix)]_n (**3**), {[Zn(μ-phsuc)(μ-mbix)]·H₂O}_n (**4**), {[Zn(μ-phsuc)(μ-pbix)]·H₂O}_n (**5**), {[Zn(μ-phsuc)(μ-pbix)]·1.5H₂O·CH₃OH}_n (**6**), [Cd(μ-phsuc)(μ-obix)]_n (**7**) and [Cd(μ₃-phsuc)(μ-pbix)_{0.5}]_n (**8**), with phsucH₂ and isomeric bis(imidazole) linkers in the presence of Co(II), Zn(II) and Cd(II) ions were hydrothermally synthesized and characterized by elemental analysis, IR spectroscopy, single crystal, and powder X-ray diffraction and thermal analysis.

2. Materials And Methods

2.1. Materials and measurements

All chemicals were received from commercial sources except that bis(imidazole) ligands were synthesized according to literature [29]. IR spectra in the range 4000-400 cm⁻¹ were taken with a Bruker Tensor FT-IR spectrometer with the use of KBr pellet. Elemental analyses of the compounds for C, H, N were performed with a Perkin Elmer 2400 Series II device. A Perkin Elmer Diamond TG/DTA Thermal Analyzer were utilized to thermal analysis curves under a dry air atmosphere in the range 30-1000 °C. Photoluminescence spectra of the compounds were gained with a Perkin-Elmer LS-55 spectrophotometer. A Panalytical Emperian X-ray diffractometer with Cu-Kα radiation was used to record powder X-ray diffraction patterns. A Bruker Smart Apex II CCD diffractometer equipped with Mo-Kα radiation (λ = 0.71073 Å) was used for single crystal X-ray diffraction data collection. SHELXT-2015 program in conjunction with the OLEX2 was used to solve the structures with direct methods and the structures were refined by full-matrix least-squares on all F² data using SHELXL [30–32]. In the structures, all hydrogen atoms were calculated and refined using riding and free modes. The crystal structures were drawn using Mercury program [33]. ToposPro software was used for topological analyses of the compounds [34]. Table 1 shows the crystal data parameters and Tables S1-S4 indicate the selected bond distances, angles, and hydrogen bond geometries.

Table 1
Crystal data and structure refinement parameters for compounds **1-4**.

	1	2	3	4
Empirical formula	C ₂₄ H ₂₂ CoN ₄ O ₄	C ₈₂ H ₈₂ Co ₃ N ₁₂ O ₂₀	C ₂₄ H ₂₂ N ₄ O ₄ Zn	C ₂₄ H ₂₄ N ₄ O ₅ Zn
Formula weight (gmol⁻¹)	489.38	1732.38	495.82	513.84
Diffractometer	Bruker APEX II CCD	Bruker APEX II CCD	Bruker APEX II CCD	Bruker APEX II CCD
Rad. /λ (Å)	Mo-K _α /0.71073	Mo-K _α /0.71073	Mo-K _α /0.71073	Mo-K _α /0.71073
Temperature (K)	293	293	293	293
Color	Violet	Violet	Colorless	Colorless
Crystal system	Orthorhombic	Triclinic	Monoclinic	Monoclinic
Space group	<i>Pbca</i>	<i>P</i> -1	<i>P</i> 2 ₁ / <i>n</i>	<i>P</i> 2 ₁ / <i>c</i>
a (Å)	14.5561 (17)	12.748 (2)	7.7463 (10)	11.297 (2)
b (Å)	16.342 (2)	12.962 (3)	17.386 (2)	16.368 (4)
c (Å)	18.610 (2)	13.500 (3)	16.051 (2)	12.430 (3)
α (°)	90	80.592 (10)	90	90
β (°)	90	72.718 (9)	91.171 (5)	99.248 (7)
γ (°)	90	72.326 (9)	90	90
V (Å³)	4427.0 (9)	2022.9 (7)	2161.3 (5)	2268.4 (9)
Z	8	1	4	4
D_c (g cm⁻³)	1.469	1.422	1.524	1.505
μ (mm⁻¹)	0.82	0.69	1.18	1.13
θ range (°)	2.9–28.4	3.2–26.8	2.9–28.4	3.0–28.3
Measured refls.	106193	53440	65779	71545
Independent refls.	5547	8139	5352	5659
R_{int}	0.098	0.072	0.079	0.035
R1/wR2	0.124/0.228	0.106/0.230	0.130/0.271	0.039/0.086
Δρ_{max}/Δρ_{min} (eÅ⁻³)	0.75/−0.68	0.98/−0.86	1.99/−1.19	0.60/−0.38

	1	2	3	4
S	1.43	1.23	1.14	1.13

Table 2
Crystal data and structure refinement parameters for complexes **5-8**.

	5	6	7	8
Empirical formula	C ₂₄ H ₂₃ N ₄ O ₅ Zn	C ₅₀ H ₆₂ N ₈ O ₁₅ Zn ₂	C ₂₄ H ₂₂ CdN ₄ O ₄	C ₁₇ H ₁₅ CdN ₂ O ₄
Formula weight (gmol⁻¹)	510.82	1135.73	542.82	423.71
Diffractometer	Bruker APEX II CCD	Bruker APEX II CCD	Bruker APEX II CCD	Bruker APEX II CCD
Rad. /λ (Å)	Mo-K _α /0.71073	Mo-K _α /0.71073	Mo-K _α /0.71073	Mo-K _α /0.71073
Temperature (K)	293	293	293	293
Color	Colorless	Colorless	Colorless	Colorless
Crystal system	Monoclinic	Monoclinic	Monoclinic	Triclinic
Space group	<i>P</i> 2 ₁ / <i>n</i>	<i>C</i> 2/ <i>c</i>	<i>P</i> 2 ₁ / <i>n</i>	<i>P</i> 2 ₁ / <i>n</i>
a (Å)	12.0883 (12)	23.450(3)	7.5704 (6)	6.8249 (8)
b (Å)	16.1314 (17)	13.7109(15)	17.6491 (15)	10.6963 (12)
c (Å)	13.1076 (13)	17.524(2)	16.6555 (15)	11.0880 (13)
α (°)	90	90	90	94.735 (4)
β (°)	114.208 (4)	91.838(5)	93.127 (3)	92.701 (4)
γ (°)	90	90	90	93.412 (4)
V (Å³)	2331.2 (4)	5631.4(12)	2222.0 (3)	804.14 (16)
Z	4	4	4	2
D_c (g cm⁻³)	1.455	1.340	1.623	1.750
μ (mm⁻¹)	1.10	0.92	1.02	1.38
θ range (°)	3.0–28.3	2.9–26.5	3.1–26.4	3.0–28.4
Measured refls.	75188	61332	58628	33460
Independent refls.	5752	5726	4533	3959
R_{int}	0.052	0.069	0.047	0.030
R1/wR2	0.094/0.222	0.150/0.121	0.052/0.107	0.043/0.113
Δρ_{max}/Δρ_{min} (eÅ⁻³)	1.78/-1.23	1.76/-1.53	1.14/-0.91	1.62/-0.89

	5	6	7	8
S	1.07	1.22	1.26	1.26

2.2. Syntheses of the compounds

[Co(μ -phsuc)(μ -obix)]_n(1)

A mixture of Co(NO₃)₂·6H₂O (0.29 g, 1 mmol), phsucH₂ (0.194 g, 1 mmol) and obix (0.240 g, 1 mmol) was stirred at room temperature for 30 min in 30 mL distilled water. 6.0 M HNO₃ (3 drops) was added dropwise into the mixture to obtain a clear solution. Then the clear solution was placed in a 45 mL Teflon-lined stainless steel vessel and heated at 120 °C for 3 days. After cooling to room temperature, the clear solution was waited about 20 days to obtain violet crystals were obtained. FT-IR (KBr, cm⁻¹): 3433(w), 3116(m), 2962(w), 2893(w), 1612(vs), 1521(m), 1373(s), 1298(m), 1244(m), 1109(m), 966(m), 843(m), 742(m), 655(m). Anal. calcd (%) for C₂₄H₂₂N₄O₄Co (489.40 g/mol): C, 58.90; H, 4.53; N, 11.45. Found: C, 58.92; H, 4.33; N, 11.59.

{[Co₂(μ -phsuc)₂(μ -pbix)_{1.5}·2H₂O]}_n(2)

A mixture of Co(CH₃COO)₂·4H₂O (0.25 g, 1 mmol), phsucH₂ (0.194 g, 1 mmol) and pbix (0.240 g, 1 mmol) was stirred at room temperature for 30 min in 15 mL distilled water. The solution was placed in a 25 mL closed capped bottle and heated at 120 °C for 3 days. Upon cooling to room temperature, violet crystals were obtained. FT-IR (KBr, cm⁻¹): 3429(s), 3134(s), 3028(w), 1616(vs), 1585(s), 1520(s), 1381(s), 1232(m), 1087(m), 992(m), 903(m), 842(m). Anal. calcd (%) for C₄₁H₄₁N₆O₁₀Co₂ (895.70 g/mol): C, 54.98; H, 4.61; N, 9.38. Found: C, 54.88; H, 4.63; N, 9.44.

[Zn(μ -phsuc)(μ -obix)]_n(3)

The complex was prepared in a similar manner as **1** except for the use of Zn(NO₃)₂·6H₂O (0.297 g, 1 mmol) instead of Co(NO₃)₂·6H₂O. Colorless crystals of **3** were obtained. FT-IR (KBr, cm⁻¹): 3433(s), 3116(s), 2963(w), 2893(w), 1612(vs), 1522(s), 1373(s), 1109 (s), 953(m), 891(m), 844(m), 742(m), 656(m). Anal. calcd (%) for C₂₄H₂₂N₄O₄Zn (495.80 g/mol): C, 58.14; H, 4.47; N, 11.30. Found: C, 58.23; H, 4.45; N, 11.33.

{[Zn(μ -phsuc)(μ -mbix)]·H₂O}_n(4)

A mixture of Zn(NO₃)₂·6H₂O (0.297g, 1 mmol), phsucH₂ (0.194 g, 1 mmol), mbix (0.240 g, 1 mmol) and NaOH (0.080 g, 2 mmol) were dissolved in solution of H₂O (15 mL), DMF (1 mL) and CH₃OH (1 mL). Precipitation formed after stirring the mixture for 30 min and then the mixture was placed in a 25 mL closed capped bottle and heated at 120 °C for 3 days. Upon cooling to room temperature, colorless crystals were obtained. FT-IR (KBr, cm⁻¹): 3522(s), 3408(s), 3126(s), 3087(w), 3051(w), 2922(w), 1606(vs), 1598(vs), 1511(m), 1402(vs), 1306(w), 1221(m), 1090(m), 1022(m), 952(m), 873(m), 743(m), 657(m).

Anal. calcd (%) for $C_{24}H_{22}N_4O_4Zn$ (513.90 g/mol): C, 56.10; H, 4.71; N, 10.90. Found: C, 55.98; H, 4.93; N, 11.03.

$\{[Zn(\mu\text{-phsuc})(\mu\text{-pbix})]\cdot H_2O\}_n$ (5)

A mixture of $Zn(CH_3COO)_2\cdot 2H_2O$ (0.249 g, 1 mmol), phsucH₂ (0.194 g, 1 mmol), pbix (0.240 g, 1 mmol) and NaOH (0.35 mL, 0.65 M) were dissolved in solution of H₂O (30 mL) and C₂H₅OH (3.5 mL).

Precipitation formed after stirring the mixture for 30 min and then the mixture was placed in a 45 mL Teflon-lined stainless steel vessel and heated at 140 °C for 3 days. Upon cooling to room temperature, colorless crystals were obtained. FT-IR (KBr, cm⁻¹): 3508(s), 3500(s), 3124(s), 2984(w), 1604(vs), 1598(vs), 1522(m), 1389(s), 1359(s), 1223(m), 1097(s), 1022(w), 955(m), 894(w), 788(m), 657(m). Anal. calcd (%) for $C_{24}H_{24}N_4O_5Zn$ (513.90 g/mol): C, 56.10; H, 4.71; N, 10.90. Found: C, 56.18; H, 4.54; N, 10.92.

$\{[Zn(\mu\text{-phsuc})(\mu\text{-pbix})]\cdot 1.5H_2O\cdot CH_3OH\}_n$ (6)

A mixture of $Zn(CH_3COO)_2\cdot 2H_2O$ (0.249 g, 1 mmol), phsucH₂ (0.194 g, 1 mmol), pbix (0.240 g, 1 mmol) and NaOH (0.080 g, 2 mmol) were dissolved in solution of H₂O (5 mL), DMF (5 mL) and CH₃OH (5 mL). This mixture was stirred for 30 min and then transferred to a 25 mL Teflon-lined stainless steel vessel, heated to 140°C for 3 days, After slow cooling to room temperature, colorless crystals of **6** were obtained. FT-IR (KBr, cm⁻¹): 3441(s), 3134(w), 2968(w), 1610(vs), 1525(m), 1373(s), 1251(m), 1090(m), 963(m), 857(w), 754(m), 656(m). Anal. calcd (%) for $C_{25}H_{32}N_4O_8Zn$ (581.90 g/mol): C, 51.60; H, 5.54; N, 9.63. Found: C, 51.18; H, 5.34; N, 9.43.

$[Cd(\mu\text{-phsuc})(\mu\text{-obix})]_n$ (7)

The compound was obtained in a similar method to that of **1** except for the use of 3CdSO₄·8H₂O (0.256 g, 1 mmol) instead of Co(NO₃)₂·6H₂O. Colorless crystals of **7** were obtained. FT-IR (KBr, cm⁻¹): 3444(w), 3122(m), 3020(w), 2974(w), 1572(vs), 1519(s), 1410(vs), 1291(m), 1242(m), 1085(s), 927(m), 873(m), 831(m), 742(s), 653(m). Anal. calcd (%) for $C_{24}H_{22}N_4O_4Cd$ (542.90 g/mol): C, 53.10; H, 4.08; N, 10.32. Found: C, 53.03; H, 4.14; N, 10.09.

$[Cd(\mu_3\text{-phsuc})(\mu\text{-pbix})_{0.5}]_n$ (8)

A mixture of 3CdSO₄·8H₂O (0.256g, 1 mmol), phsucH₂ (0.194 g, 1 mmol) and pbix (0.240 g, 1 mmol) was stirred at room temperature for 30 min in 30 mL distilled water. The solution was transferred in a 45 mL Teflon-lined stainless steel vessel and heated at 120 °C for 3 days. Upon cooling to room temperature, violet crystals were obtained. FT-IR (KBr, cm⁻¹): 3502(m), 3420(m), 3109(m), 3030(w), 2988(w), 1560(vs), 1419(s), 1298(w), 1095(m), 998(m), 942(m), 897(w), 735(m), 658(m). Anal. calcd (%) for $C_{17}H_{15}N_2O_4Cd$ (423.75 g/mol): C, 48.19; H, 3.57; N, 6.61. Found: C, 48.22; H, 3.71; N, 6.49.

3. Results And Discussion

3.1. Syntheses and Characterizations

The eight coordination polymers were hydrothermally synthesized with phsucH₂ and isomeric bis(imidazole) linkers in the presence of diverse metal ions and characterized with several techniques. Compound **1** was not reproduced repeatedly. Hence, only single crystal analysis result was given to determine the effect of isomeric bis(imidazole) linkers on the structure. Elemental analysis results are agreed with the given formulations. IR spectra of compounds **2, 4-6** indicate that the broad bands observed at about 3429 cm⁻¹ are attributed to the ν(OH) stretchings of crystal water molecules. The aromatic and aliphatic ν(CH) stretchings appeared at between 3134 and 2922 cm⁻¹ in all compounds. In the compounds, asymmetric (ν_{as}COO⁻) and symmetric (ν_sCOO⁻) stretching vibrations of carboxylate groups of phsuc²⁻ are observed in the range of 1616-1560 cm⁻¹ and 1419-1359 cm⁻¹, respectively. The asymmetric stretching vibration of phsucH₂ shifts to a lower frequency after connecting to metal ions (Figs. S1-S8). Moreover, ν(C=N) stretching vibrations of imidazole rings are observed at 1520 cm⁻¹ in the compounds.

Descriptions of crystal structures

[Co(μ-phsuc)(μ-obix)]_n (1). X-ray crystallographic analysis indicates that compound **1** crystallizes in the orthorhombic *Pbca* space group, and the asymmetric unit of **1** contains one Co(II) ion, one phsuc and one obix ligand (Fig. 1a). Co1 ion is in a slightly distorted tetrahedral coordination geometry with two nitrogen atoms from two different obix ligands and two oxygen atoms from two different phsuc ligands. Each phsuc ligand adopts bis(monodentate) coordination mode linking Co(II) ions leading to a 1D linear chain with the intrachain Co...Co distance 7.546 Å (Fig. 1b). Furthermore, the adjacent 1D chains are connected by obix ligands into a 2D wave network (Fig. 1c.). This 2D network can be clarified as a 4-connected sql/Shubnikov tetragonal topology with the point symbol of {4⁴.6²} (Fig. 1d).

{[Co₂(μ-phsuc)₂(μ-pbix)_{1.5}]·2H₂O}_n (2). Compound **2** crystallizes in the triclinic system, *P*-1 space group. The asymmetric unit of **2** contains two independent Co(II) ions, two phsuc, one and a half pbix ligands and two crystal water molecules. As shown in Fig. 2a, there are two cobalt centres (Co1 and Co2) in the compound with different coordination environments. Co1 is coordinated to two pbix ligands via two nitrogen atoms of the imidazole groups and two oxygen atoms of two carboxylate groups from two different phsuc ligands, showing a distorted tetrahedral geometry. Whereas, Co2 is six-coordinated with four carboxylate oxygen atoms from four different phsuc ligands to form a distorted octahedral geometry with N₂O₄ donor set. The basal plane is constructed by the coordination of four carboxylate oxygen atoms, whereas, the axial site of Co2 ion is occupied by two nitrogen atoms from two symmetry-related pbix ligands. The carboxylate groups of phsuc ligands are completely deprotonated and adopt the bis(monodentate) coordination modes. Co1 and Co2 are bridged by phsuc anions into a 1D double chain with four metallic 28-member rings (Fig. 2b). Then, the adjacent two 1D double chains are further connected by two nitrogen atoms from pbix ligands with bimetallic 26-member rings, thus generating a 2D tubular structure along the *b*-axis (Fig. 2c). As shown in Figs. 2d and 2e, pbix ligand adopts two

different conformations, namely *gauche* and *trans*, to generate a 2D polyrotaxane sheet. The values of the Co...Co separation through these bridges are 7.221, 7.943 and 11.001 Å. The 2D framework can be clarified as a 6-nodal net and the point symbol for net is $\{4.8^3.12^2\}_2\{4\}_2\{8^4.12^9.16^2\}\{8\}_5$ (Fig. 2f).

[Zn(μ -phsuc)(μ -obix)]_n(3). X-ray diffraction result indicates that compound **3** crystallizes in the monoclinic space group $P2_1/n$ and features a two-dimensional (2D) network. The asymmetric unit consists of one Zn(II) ion, one phsuc and one obix ligand. As shown in Fig. 3a, the Zn1 ion is four-coordinated by two carboxylate oxygen atoms from two phsuc ligands and two nitrogen atoms from obix ligands, showing a distorted tetrahedral structure. The Zn–O and Zn–N bond lengths of previously reported Zn(II) complexes based on succinate ligands are similar to those of compound **3** [35]. The phsuc ligand is fully deprotonated and acts as bridging ligands with bis(monodentate) coordination modes to connect the Zn(II) ions to form a 1D linear structure (Fig. 3b.). Two adjacent 1D units are connected by obix ligands to give a 2D wave network (Fig. 3c). The distances between the closest Zn(II) ions are 7.746 and 13.563 Å. The compound shows a uninodal 4-connected sql topology with the point symbol of $\{4^4.6^2\}$ (Fig. 3d).

{[Zn(μ -phsuc)(μ -mbix)]·H₂O}_n(4). Single crystal X-ray structural analysis reveals that **4** crystallizes in the monoclinic system, $P2_1/c$ space group. The asymmetric unit of compound **4** contains one Zn(II) ion, one phsuc anion, one mbix ligand, and one lattice water molecule. As shown in Fig. 4a, Zn1 is four coordinated in a tetrahedral coordination geometry, coordinated by two oxygen atoms from two different phsuc anions and two nitrogen atoms from two different mbix ligands. The Zn–O/N bond lengths are in the normal range of 1.910(17)–2.0276(17) Å as other Zn(II)-containing coordination polymers. Two symmetry-related Zn1 ions are connected by two carboxylate groups from phsuc ligand to generate a 1D linear chain with the Zn...Zn distance of 6.358 Å. Each phsuc ligand acts as a bridging ligand through the two carboxylate groups as a bis(monodentate) mode (Fig. 4b). The adjacent 1D chains are further bridged by the mbix ligands to generate a 2D network (Figs. 4c and 4d). In addition, there are intrachain O–H...O hydrogen-bonding interactions (Fig. 4e), which further stabilize the 1D chains of **4**. Topologically, compound **4** can be simplified as a sql/Shubnikov tetragonal plane net with a point symbol of $\{4^4.6^2\}$ (Fig. 4f).

{[Zn(μ -phsuc)(μ -pbix)]·H₂O}_n(5). Single-crystal X-ray diffraction shows that **5** crystallizes in monoclinic system with $P2_1/n$ space group. The asymmetric unit of **5** contains one Zn(II) ion, one phsuc, and one pbix ligand and one crystal water molecule. The Zn1 is four-coordinated in a tetrahedral coordination environment, coordinated by two oxygen atoms from two different phsuc ligands and two nitrogen atoms from two different pbix ligands (Fig. 5a). The phsuc ligand acts as a bis(monodentate) ligand to connect with two Zn(II) ions to generate a 1D chain structure with the Zn...Zn distance of 6.948 Å. The Zn1–O distances are 1.939(4) and 1.941(4) Å, and Zn1–N distances are 1.997(4) and 2.038(5) Å, respectively. Adjacent the 1D chains are further linked by nitrogen atoms from pbix ligands generating a 2D wave structure (Figs. 5b and 5c). Topologically, the 2D layer can be simplified as a 4-connected sql/Shubnikov tetragonal plane net with a point symbol of $\{4^4.6^2\}$ (Fig. 5d). The 2D layers interact with each other through C–H... π interactions between C7–H7, C8–H8 and phenyl (Cg1) and imidazole rings of pbix

ligand, respectively to form a 3D supramolecular frameworks [Cg1 = C15–C16–C17b–C15b–C16b–C17, Cg2 = N3–C18–C19–N4–C20, C7...Cg1 = 3.645(13) and C8...Cg2 = 3.644(10) Å].

{[Zn(μ -phsuc)(μ -pbix)]·1.5H₂O·CH₃OH}_n (6). It is very surprising that when solvent mixture (H₂O (30 mL) and C₂H₅OH (3.5 mL)) for the synthesis of **5** is replaced with solvent mixture (H₂O (5 mL), DMF (5 mL) and CH₃OH (5 mL)), two-fold interpenetrated network of **6** is observed. Compound **6** shows a 2D network and it crystallizes in monoclinic system *C2/c*. The asymmetric unit consists of one Zn(II) ion, one phsuc, one pbix, one methanol and one and a half water molecules (Fig. 6a). The Zn1 atom is four-coordinated and forms a disordered tetrahedral geometry, which is completed by two carboxylate oxygen atoms from two distinct phsuc moieties and two nitrogen donors from two pbix ligands. The lengths of the Zn–O (1.951(6)–1.959(6) Å) and Zn–N (2.002(7) and 2.000(7) Å) bonds are comparable to those observed in the related literature [35]. The adjacent Zn(II) ions are connected by bridging carboxylate groups of phsuc ligands into a 1D double chain with the Zn...Zn distance of 9.291 Å. Further, every Zn(II) ion are coordinated with two pbix ligands showing gauche form, and a two-fold interpenetrating 2D structure with bimetallic 26-member rings is observed (Fig. 6b.) Two adjacent 2D networks interpenetrate each other to form a 2D + 2D → 2D interpenetration network (Fig. 6c). The final framework of **6** can be considered as a uninodal 3-connected hcb Shubnikov hexagonal plane net with the point symbol of {6³} (Fig. 6d).

[Cd(μ -phsuc)(μ -obix)]_n (7). X-ray diffraction shows that **7** crystallizes in monoclinic system with *P2₁/n* space group. The asymmetric unit contains one Cd(II) ion, one phsuc and obix ligands. As shown in Fig. 7a, the Cd1 ion is coordinated by four oxygen atoms from two different phsuc ligands and two nitrogen atoms from two different obix ligands. The Cd–O/N bond lengths are in the range of 2.245(4) to 2.470(4) Å. The phsuc ligands in **7** adopt a bis(bidentate) bridging coordination mode linking the neighbouring Cd(II) ions to generate 1D linear chain along the *a*-axis with Cd(II)...Cd(II) distances of 7.570 Å (Fig. 7b). The adjacent 1D chains are linked to form a 2D wave-like structure along the *c*-axis by obix ligands (Fig. 7c). There are π ... π interactions between the imidazole rings (Cg1 = N1–C11–C12–N2–C13, Cg1...Cg1ⁱ = 3.544(3) Å, (i) 1-*x*, 1-*y*, 1-*z*). Furthermore, there are also weak C–H... π interactions between the C13–H13 and phenyl ring (Cg2) of phsuc ligand (Cg2 = C5–C6–C7–C8–C9–C10, C13...Cg2 = 3.516(5) Å). All of these intermolecular interactions give a three-dimensional supramolecular network (Fig. 7d). Topologically, compound **7** can be simplified as a 4-connected sql/Shubnikov tetragonal plane net with a point symbol of {4⁴·6²} (Fig. 7e).

[Cd(μ_3 -phsuc)(μ -pbix)_{0.5}]_n (8). Compound **8** crystallizes in a triclinic with space group *P2₁/n*. There is one Cd(II) ion, one phsuc ligand, a half pbix ligands in an asymmetric unit of **8**. As shown in Fig. 8a, the Cd1 is six-coordinated by five oxygen atoms from two different phsuc ligands, and one nitrogen atom from pbix ligand. The lengths of the Cd–O (2.278 (4)–2.385 (4) Å) and Cd–N (2.209 (3) Å) bonds are comparable to those observed in the related literature [36–39]. There are two coordination modes for the carboxylate groups of the phsuc ligand, with one in a μ - η^1 : η^1 chelating mode and the other in a μ_3 - η^1 : η^2 tridentate bridging mode (Fig. 8b). The closest three Cd(II) ions are bridged together through the carboxylate groups

of the phsuc ligands to generate a 14-membered rings based on the $[\text{Cd}_2(\text{CO}_2)_4]$ unit with a Cd1...Cd1 distance of 6.139 Å. The $[\text{Cd}_2(\text{CO}_2)_4]$ clusters are further connected by pbix ligands to form a 2D network (Fig. 8c). The neighboring 2D layers are further assembled by weak $\pi\cdots\pi$ stacking interactions, with centroid-centroid (Cg1= C5-C6-C7-C8-C9-C10) distance of 4.095(3) Å, and C-H... π (C16...Cg1 = 3.598(6) Å and C16-H16...Cg1 = 141 °) interactions into the 3D supramolecular framework (Fig. 8d). Topologically, if the Cd(II) center is regarded as a four-connected node and the phsuc ligand is considered as linker, the structure of **8** can be described as a 4-connected layer with sql/Shubnikov tetragonal plane topology with the point symbol of $\{4^4\cdot 6^2\}$ (Fig. 8e).

3.3. Powder X-ray diffraction and thermal properties

The phase purities of the compounds were determined with powder X-ray diffraction (PXRD) measurements (Figs. S9-S15.). All the major peaks of the recorded PXRD patterns are agreed with those of the simulated patterns, demonstrating the phase purities of the compounds.

Thermal analysis curves of the compounds were recorded in the temperature range 30-1000 °C under a dry air atmosphere to determine the thermal behaviors of the compounds (Fig. S16-S23). For compounds **2** and **4-6**, the first weight losses are related to eliminations of crystal water molecules (Exp.: 3.8% calc.: 4.1 % for **2**; exp.: 4.1 % calc.: 3.5% for **4**; exp.: 3.0% calc.: 3.5% for **5**; exp.: 10.54% calc.:10.63% for **6**). After the steps, compounds **2** and **4-6** displayed stability up to 200-280°C and anhydrous compounds **1, 3, 7** and **8** showed stability up to 300 °C. When further heating, all compounds are exothermally decomposed and the final decomposition products can be Co_3O_4 which turns to CoO over 905 °C for **1** and **2**, and ZnO for **3-6** and CdO for **7** and **8**. (CoO, exp.:17.8%, calc.:15.31% for **1**; exp.: 9.27%, calc.: 8.36% for **2**; ZnO, exp.: 16.11%, calc.: 16.42% for **3**; exp.: 13.07%, calc.: 15.76% for **4**; exp.: 17.22%, calc.: 15.77% for **5**; exp.:8.4%, calc.: 14.6% for **6**; CdO, exp.: 21.83%, calc.: 23.65% for **7**; exp.: 27.8%, calc.: 30.3% for **8**)

3.4. Photoluminescence properties

Solid-state photoluminescence spectra of compounds **3-8** with the d^{10} metal center were recorded under the same conditions at room temperature. As seen in Fig. 9, phsucH₂ displayed very weak emission at 360 and 425 nm upon excitation at 300 nm. In our previous study, isomeric bis(imidazole) linkers showed emissions in the range of 409-428 nm with the excitation at 300 nm. These emissions were assigned to $\pi^*\rightarrow\pi$ or $\pi^*\rightarrow n$ transitions of the ligands [40]. Compounds **3-8** showed emission maxima at 440, 384, 368, 422, 440 and 382 nm, respectively upon excitation at 300 nm. The shifted emissions of the compounds, when compared to free ligands, could be assigned to formation of coordination bonds.

4. Conclusion

Eight coordination polymers were prepared with phsucH₂ and isomeric semi-flexible bis(imidazole) linkers in the presence of diverse M(II) ions and characterized by several techniques. X-ray results showed that the phsuc²⁻ connected the metal ions to form one dimensional chains which were extended to 2D structures by coordination of bis(imidazole) linkers. Moreover, all compounds were extended to 3D

supramolecular structure via $\pi\cdots\pi$ stacking and weak C–H $\cdots\pi$ interactions. The phsuc²⁻ ligand displayed diverse coordination modes in the compounds and pbix ligand showed *trans*- and *gauche*- conformations due to its flexible nature. The emissions of the compounds were assigned to the intra-ligand transition of bis(imidazole) linkers because it was difficult to oxidize or reduce the d¹⁰ metal ions

Declarations

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Supporting Information

The Cambridge Crystallographic Data Centre with CCDC No. 2142948-2142955 for **1-8** have deposited the crystallographic data for the structural analysis. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: <http://www.ccdc.cam.ac.uk>).

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Scheme

Schemes 1 is available in the Supplementary Files section.

Figures

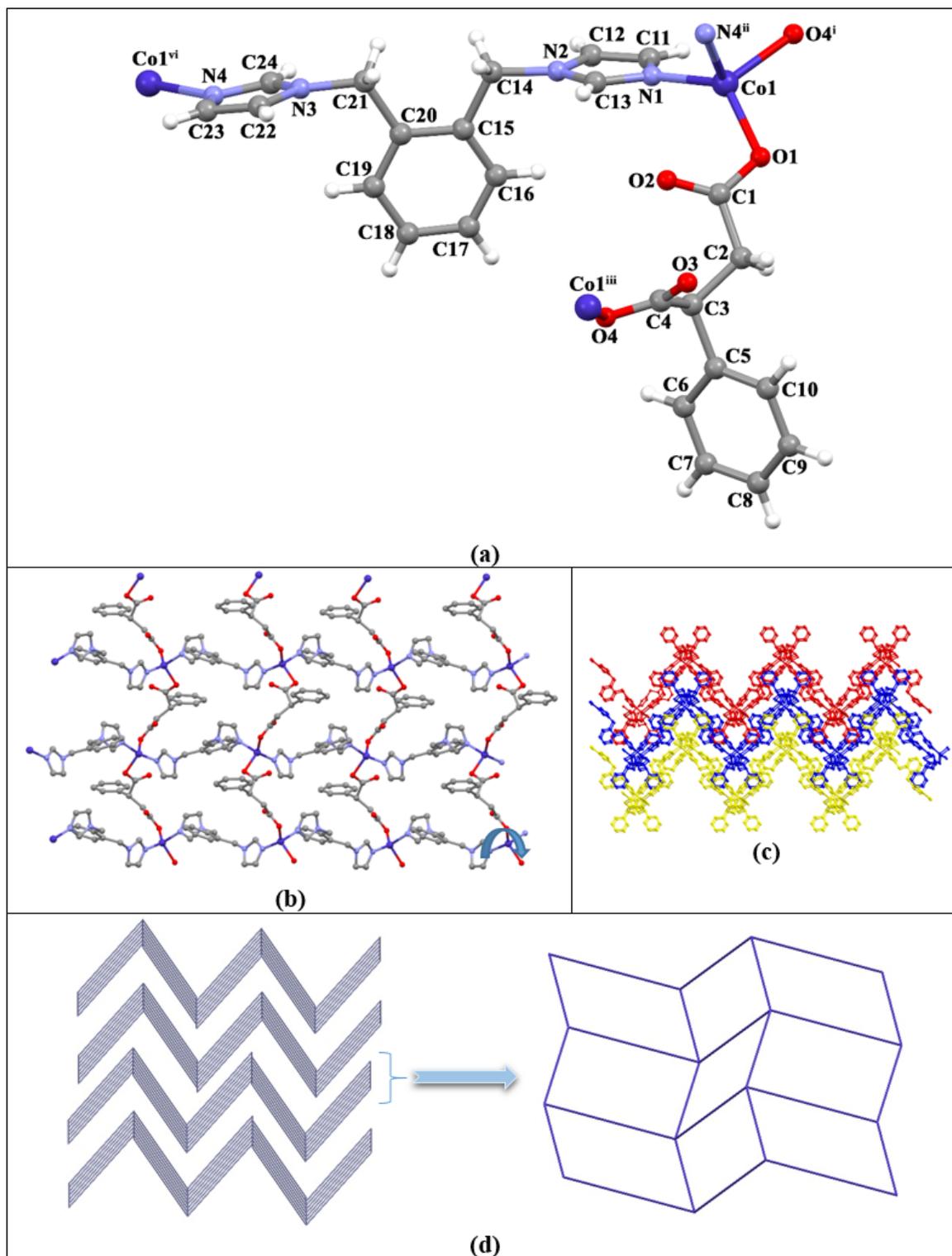


Figure 1

(a) View of the molecular structure of **1** with the atom numbering scheme **(b)** View of the two-dimensional network of **1**. (Hydrogen atoms have been omitted for clarity), **(c)** View of the 3D supramolecular structure of **1** **(d)** The schematic view of sql topology of **1** ((i) $x+\frac{1}{2}, y, -z+\frac{1}{2}$; (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x-\frac{1}{2}, y, -z+\frac{1}{2}$; (iv) $x, -y+\frac{1}{2}, z+\frac{1}{2}$).

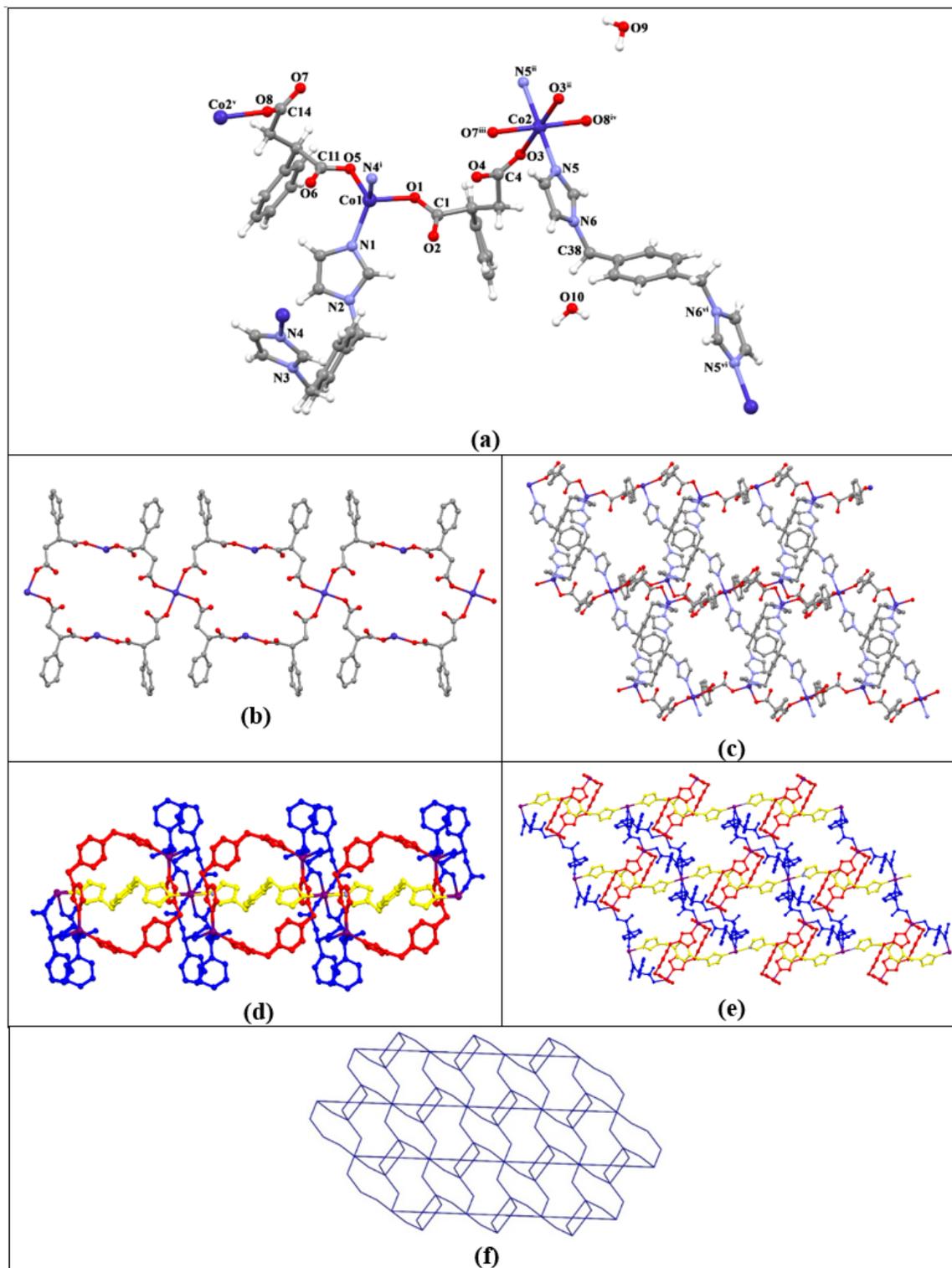


Figure 2

(a) View of the molecular structure of **2** with the atom numbering scheme **(b)** View of the 1D structure of **2** (Hydrogen atoms have been omitted for clarity), **(c-e)** View of the 2D supramolecular structure of **2** **(f)** The schematic view of topology of **2** ((i) $-x, -y, -z$; (ii) $-x+1, -y+1, -z$; (iii) $-x+1, -y, -z$; (iv) $x, y+1, z$; (v) $x, y-1, z$; (vi) $-x, -y+2, -z$)

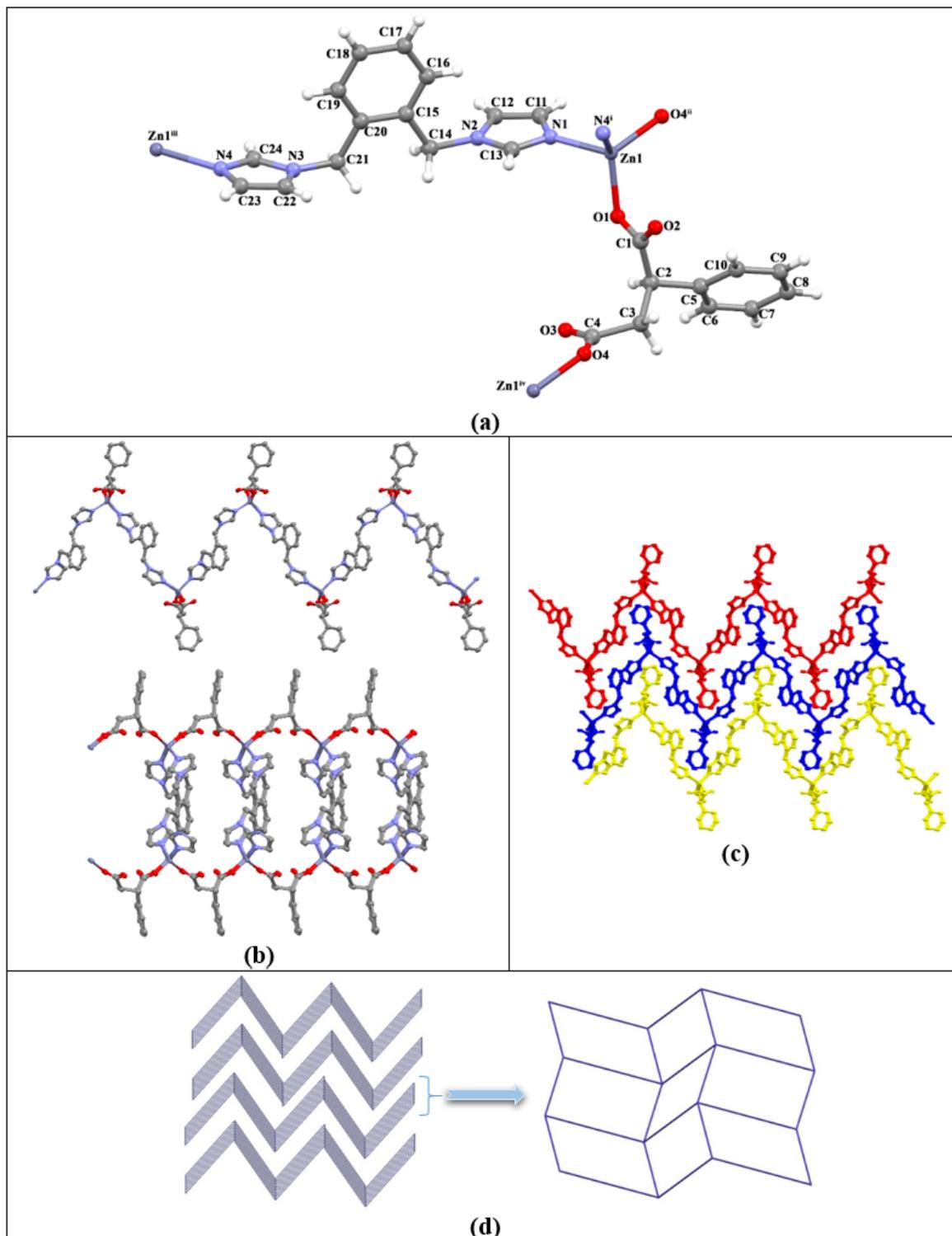


Figure 3

(a) View of the molecular structure of **3** with the atom numbering scheme **(b)** View of the two-dimensional network of **3** (Hydrogen atoms have been omitted for clarity), **(c)** View of the 3D supramolecular structure of **3** **(d)** The schematic view of sql topology of **3** ((i) $x-\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x-1, y, z$, (iii) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (iv) $x+1, y, z$.)

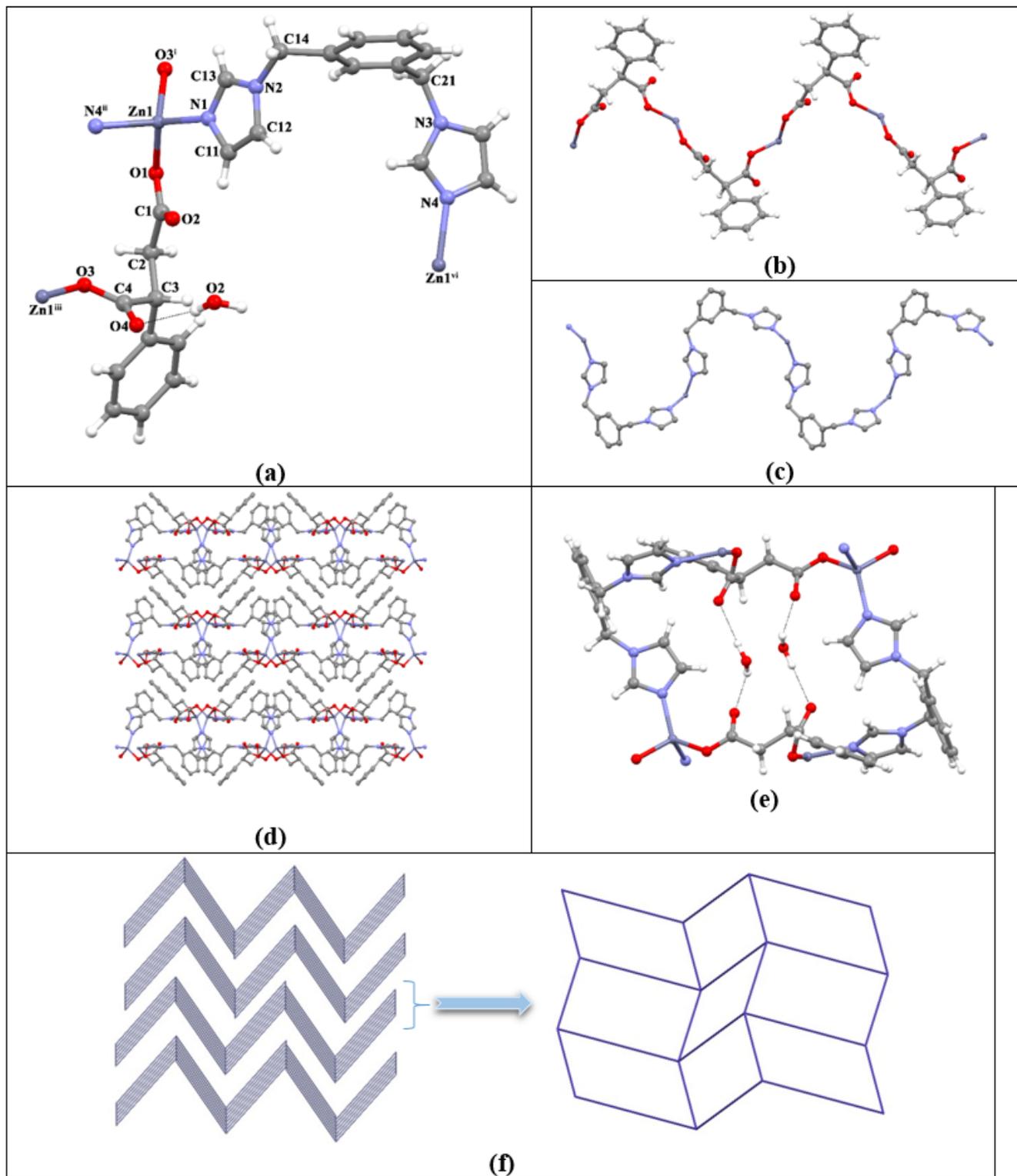


Figure 4

(a) View of the molecular structure of **4** with the atom numbering scheme **(b)** View of the one-dimensional chain constructed by phsuc ligands (Hydrogen atoms have been omitted for clarity) **(c)** View of the one-dimensional chain constructed by pbix ligands **(d)** View of the 3D supramolecular structure of **4** **(e)** A view of the hydrogen bonding interaction **(f)** The schematic view of sql topology of **4** ((i) $x, -y+1/2, z-1/2$; (ii) $-x+1, y-1/2, -z+1/2$; (iii) $x, -y+1/2, z+1/2$; (iv) $-x+1, y+1/2, -z+1/2$.)

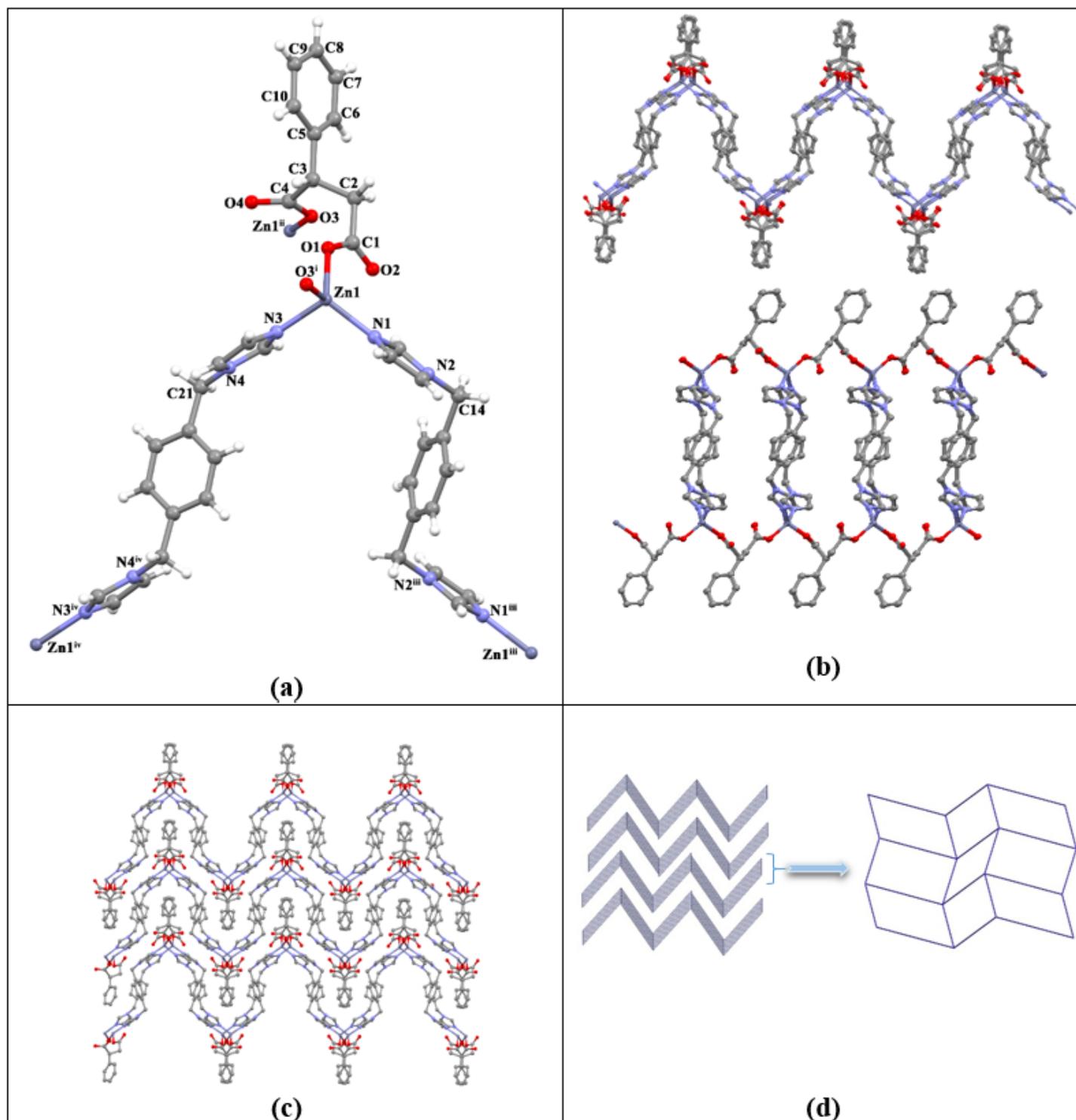


Figure 5

(a) View of the molecular structure of **5** with the atom numbering scheme **(b)** View of the two-dimensional network of **5** (Hydrogen atoms have been omitted for clarity) **(c)** View of the 3D supramolecular structure of **5** **(d)** The schematic view of sql topology of **5** ((i) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (iii) $-x+1, -y, -z$; (iv) $-x+1, -y+1, -z$)

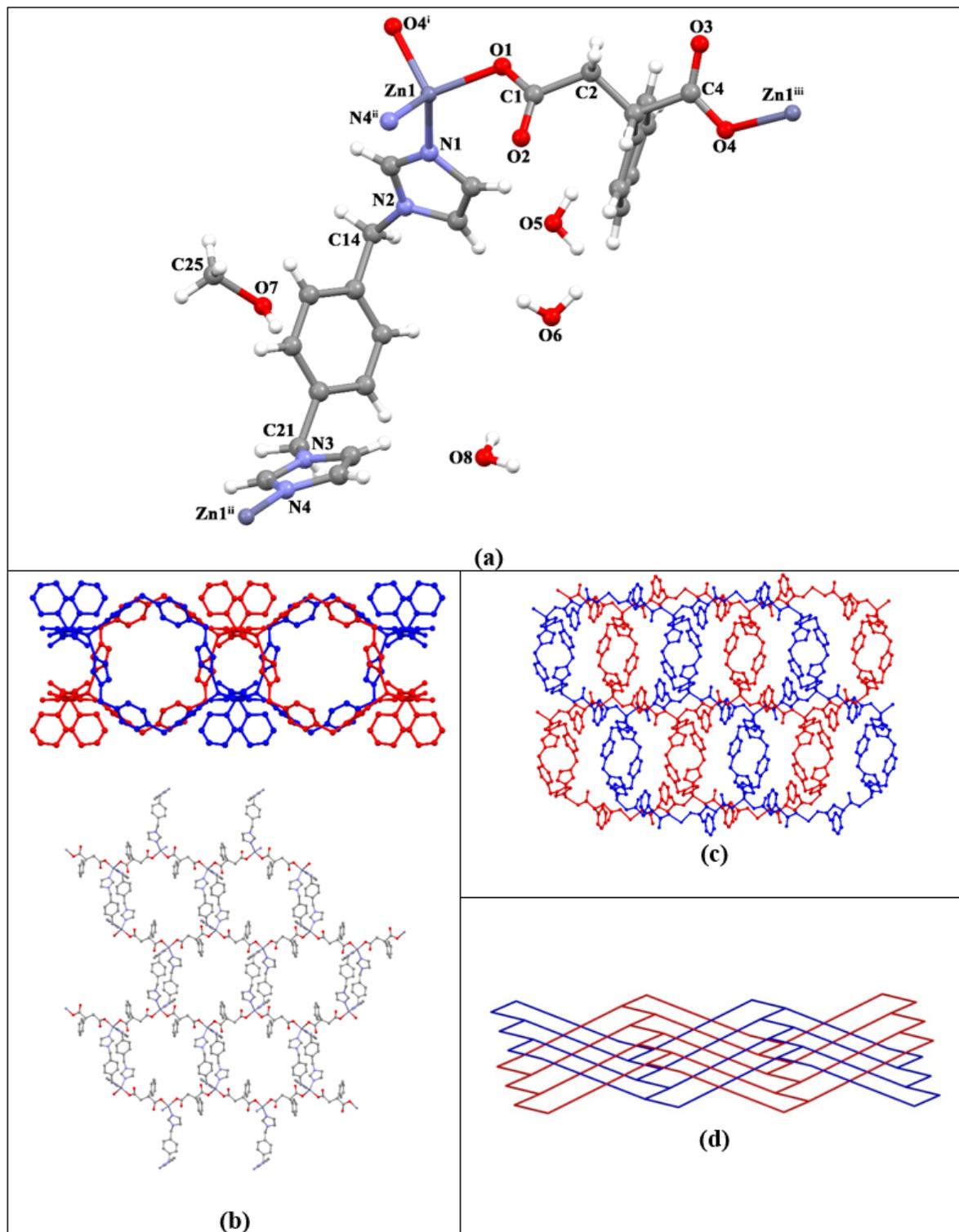


Figure 6

(a) View of the molecular structure of **6** with the atom numbering scheme **(b)** View of the two-dimensional network of **6** (Hydrogen atoms have been omitted for clarity), **(c)** View of the 2D+2D@2D interpenetration structure of **6** **(d)** The schematic view of hcb topology of **6** ((i) $x, -y+2, z+\frac{1}{2}$; (ii) $-x+1, -y+1, -z+1$; (iii) $x, -y+2, z-\frac{1}{2}$)

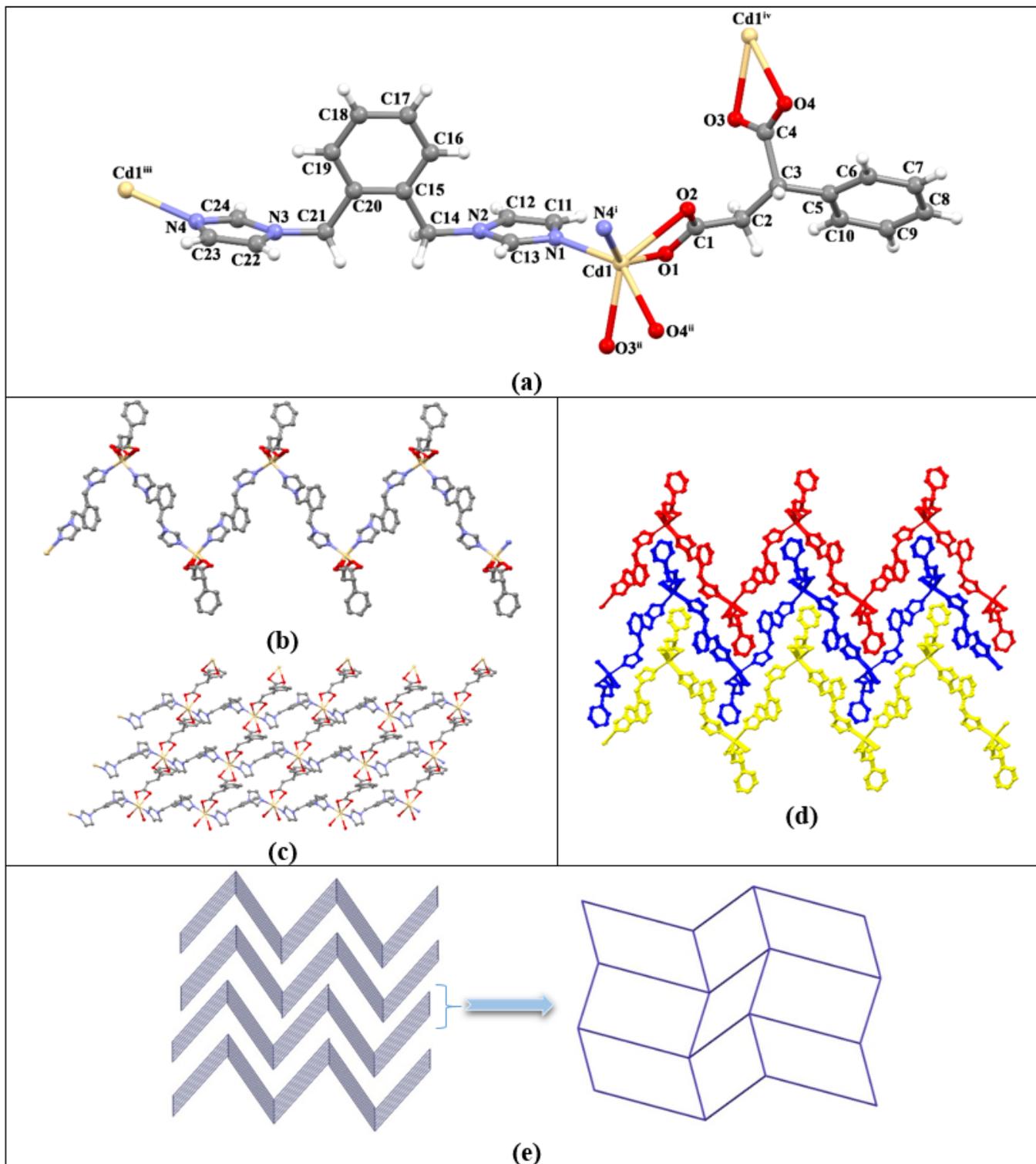


Figure 7

(a) View of the molecular structure of **7** with the atom numbering scheme **(b)** View of the one-dimensional structure of **7** **(c)** View of the two-dimensional structure of **7** (Hydrogen atoms have been omitted for clarity) **(d)** The schematic view of sql topology of **7** ((i) $x+1/2, -y+1/2, z-1/2$; (ii) $x-1, y, z$; (iii) $x-1/2, -y+1/2, z+1/2$; (iv) $x+1, y, z$).

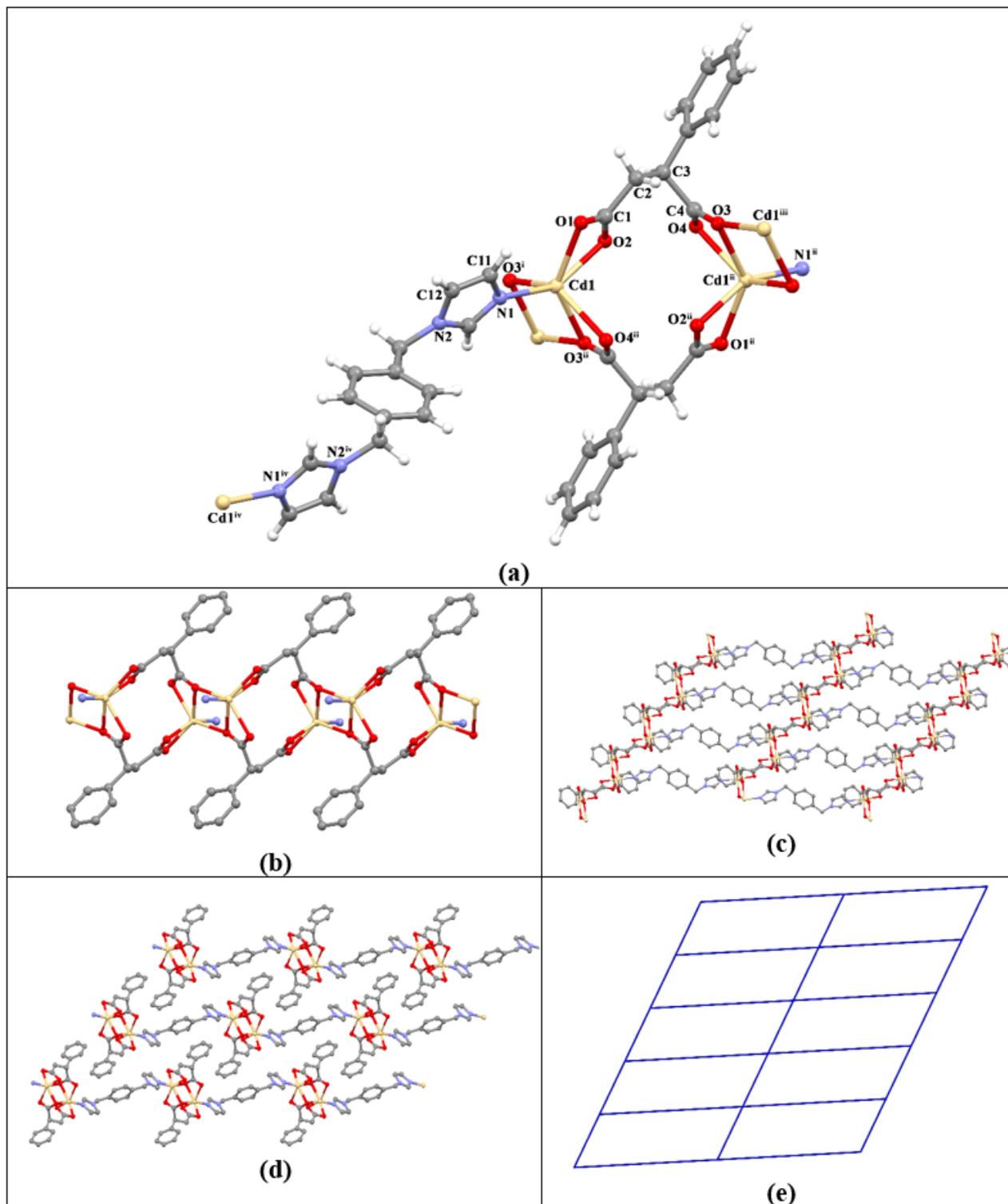


Figure 8

(a) View of the molecular structure of **8** with the atom numbering scheme (b) View of the one-dimensional double chain of **8** (Hydrogen atoms have been omitted for clarity) (c) View of the two-dimensional network of **8** (d) View of the 3D supramolecular structure of **8** (e) The schematic view of sql topology of **8** ((i) $x+1, y, z$; (ii) $-x, -y+1, -z+1$; (iii) $x-1, y, z$; (iv) $-x+2, -y, -z$)

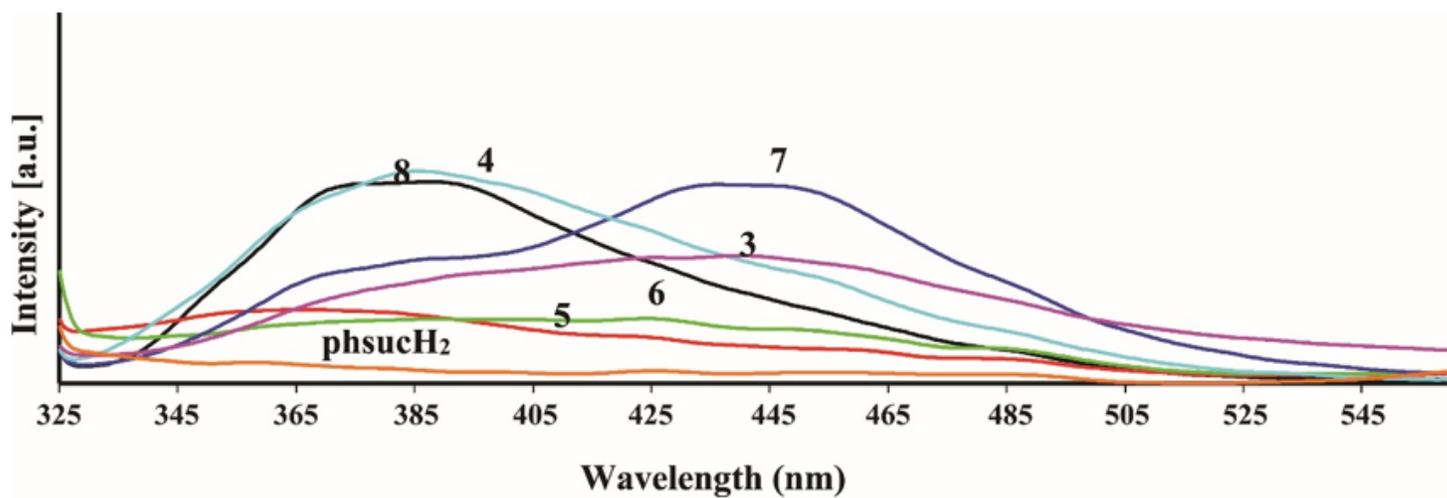


Figure 9

Solid state photoluminescence spectra of phsucH₂ and compounds **3-8**

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- [Sl.docx](#)
- [Scheme1.png](#)