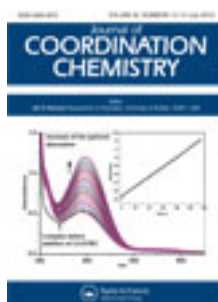


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Zn^{II}/Ni^{II} coordination polymers with 2-phenylsuccinate and 1,3-bis(4-pyridyl)propane

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Zn^{II}/Ni^{II} coordination polymers with 2-phenylsuccinate and 1,3-bis(4-pyridyl)propane

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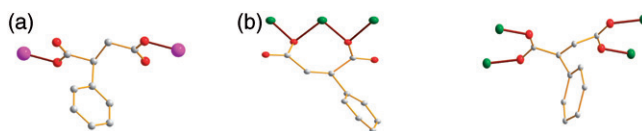
Two coordination polymers, [ZnCl(PHSU)_{0.5}(bpp)]_n (**1**) and [Ni₃(PHSU)₃(bpp)₂(H₂O)₂]_n (**2**) (PHSU = 2-phenylsuccinate, bpp = 1,3-bis(4-pyridyl)propane), have been synthesized by hydrothermal reactions of ZnCl₂ or NiCl₂·6H₂O with PHSU²⁻ and bpp. Single-crystal X-ray diffractions reveal that **1** is a 1-D coordination ladder with tetrahedral Zn^{II}, which is further linked into a 2-D network by hydrogen bonds. Complex **2** displays a 2-D coordination network with (4, 4) topology containing octahedral Ni^{II} with linear trinuclear Ni₃^{II} clusters. Flexible PHSU²⁻ in **1** and **2** adopts different coordination modes. Thermogravimetric analysis, powder X-ray diffraction, EA, and IR of **1** and **2** were also investigated.

Keywords: Coordination polymer; Ladder; Linear trinuclear cluster

1. Introduction

Coordination polymers have intriguing structural topologies and properties [1–4]. Aromatic polycarboxylates are usually employed to construct coordination polymers due to their versatile coordination and high structural stability [5–7]. Flexible dipyrindine ligands are good linkers because of their strong coordination, suitable length and flexibility. A series of interesting structural topologies, helical, interwoven, and other species, with flexible dipyrindine ligands have been reported [8–10]. We recently investigated a series of coordination polymers with metal clusters extended by 1,3-bis(4-pyridyl) propane (bpp) [11,12]. To study the influence of different metal nodes on construction of coordination polymers, we report herein two coordination polymers based on Zn^{II}/Ni^{II}-2-phenylsuccinate subunit extended by bpp, including a 1-D coordination ladder with tetrahedral Zn^{II} [ZnCl(PHSU)_{0.5}(bpp)]_n (**1**) and a 2-D coordination network with octahedral Ni^{II} in linear trinuclear clusters [Ni₃(PHSU)₃(bpp)₂(H₂O)₂]_n (**2**). PHSU²⁻ in **1** and **2** displays different coordination and conformations (scheme 1).

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Scheme 1. Coordination modes of PHSU^{2-} in (a) **1** and (b) **2**.

2. Experimental

2.1. Materials and methods

Reagents and solvents employed were commercially available and used as received. FT-IR spectra were recorded from KBr pellets, $4000\text{--}400\text{ cm}^{-1}$, on a Bio-Rad FTS-7 spectrometer. C, H, and N elemental analyses were carried out with an Elementar Vario-EL CHNS elemental analyzer. Thermogravimetric analysis (TGA) was performed at a rate of $10^\circ\text{C min}^{-1}$ using a NETZSCH system. Powder X-ray diffraction intensities were measured on a Bruker ADVANCE X-ray diffractometer (Cu-K α , $\lambda = 1.54056\text{ \AA}$).

2.2. Synthesis

2.2.1. Synthesis of $[\text{ZnCl}(\text{PHSU})_{0.5}(\text{bpp})]_n$ (1**).** A mixture of ZnCl_2 (0.068 g, 0.5 mmol), H_2PHSU (48.5 mg, 0.25 mmol), NaOH (0.02 g, 0.5 mmol), bpp (0.099 g, 0.5 mmol) and water (15 mL) was heated at 140°C for 2 days. After cooling to room temperature at a rate of 5°C h^{-1} , block colorless crystals were obtained in 40% yield (based on ZnCl_2). Anal. Calcd for $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_2\text{ClZn}$ (%): C, 54.71; H, 4.59; N, 7.09. Found (%): C, 54.78; H, 4.68; N, 7.02. IR (KBr, ν (cm^{-1})): 3036(w), 3038(w), 2944(w), 1634(m), 1604(s), 1420(m), 1362(s), 1072(s), 820(s), 739(m), 697(s), 642(m), 547(m), 478(m).

2.2.2. Synthesis of $[\text{Ni}_3(\text{PHSU})_3(\text{bpp})_2(\text{H}_2\text{O})_2]_n$ (2**).** A mixture of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (118.9 mg, 0.5 mmol), H_2PHSU (97.1 mg, 0.5 mmol), NaOH (0.02 g, 0.5 mmol), bpp (99.0 mg, 0.5 mmol), and water (15 mL) in a 23 mL Teflon reactor was heated at 160°C for 72 h and then cooled to room temperature at a rate of 5°C h^{-1} . Blue block crystals were obtained in 60% yield (based on $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$). Anal. Calcd for $\text{C}_{56}\text{H}_{52}\text{N}_4\text{O}_{14}\text{Ni}_3$ (%): C, 56.95; H, 4.44; N, 4.74. Found (%): C, 57.09; H, 4.60; N, 4.61. IR (KBr, cm^{-1}): 3368(s), 3036(w), 1634(m), 1604(s), 1420(m), 1362(s), 1072(s), 820(s), 739(m), 697(s), 642(m).

2.3. Crystal structure determination

Diffraction intensities of **1** and **2** were collected on a Bruker Apex CCD area-detector diffractometer (Mo-K α , $\lambda = 0.71073\text{ \AA}$). Absorption corrections were applied by using the multiscan program SADABS [13]. The structures were solved with direct methods and refined with full-matrix least-squares with SHELXTL [14]. Anisotropic thermal

parameters were applied to all nonhydrogen atoms. Hydrogen atoms attached to carbon and oxygen were inserted at calculated positions and allowed to ride on their respective parents. Crystal data as well as details of data collection and refinements for **1** and **2** are summarized in table 1. Selected bond distances and angles are listed in table 2.

3. Results and discussion

3.1. Synthesis

Recently, we found an *in situ* reaction of bpp under hydrothermal conditions, which provides a pathway for generating functional building blocks and coordination polymers [11]. In this work, the hydrothermal reactions of Zn^{II}/Ni^{II} chloride, bpp and 2-phenylsuccinate resulted in a 1-D coordination ladder with tetrahedral Zn^{II} [ZnCl(PHSU)_{0.5}(bpp)]_n (**1**) and a 2-D coordination network with linear trinuclear Ni^{II} clusters [Ni₃(PHSU)₃(bpp)₂(H₂O)₂]_n (**2**). TGA experiments were conducted to determine the thermal stability of **1** and **2** (figure S1). The TG curve of **1** demonstrates that it is thermally stable below 260°C and then begins to decompose. The major weight loss occurred at 260–460°C, corresponding to release of PHSU²⁻ and bpp. For **2**, the first weight loss of 3.3% at 100–185°C corresponds to removal of two coordinated water molecules (Calcd 3.1%). A major weight loss occurred at 185–450°C. The phase purities of bulk **1** and **2** were confirmed by comparison of their XRPD with those calculated from single crystal X-ray diffraction (figures S2 and S3).

3.2. Crystal structure of [ZnCl(PHSU)_{0.5}(bpp)]_n (**1**)

Single-crystal X-ray diffraction reveals that **1** crystallized in the triclinic space group of *P* $\bar{1}$. The asymmetrical unit contains one Zn^{II}, one coordinated Cl⁻, half PHSU²⁻ and one bpp (figure 1a). Tetrahedral Zn1 coordinates to two nitrogen atoms from two different bpp ligands, one chloride and one oxygen from PHSU²⁻ (the phenyl groups of PHSU²⁻ are disordered in a 1:1 ratio in two positions). Zn1–O, Zn1–N, and Zn1–Cl distances are 1.936(4)–2.252(2) Å (table 2). The bpp possess the TT conformation (*T* = *trans*) with N···N distance of 9.625 Å [10]. PHSU²⁻ exhibits *trans*-monodentate bridging (scheme 1a). Four tetrahedral Zn^{II} centers are linked by two bpp with TT conformation and two *trans*-monodentate PHSU²⁻ to form a basic grid unit of the 1-D ladder. Adjacent parallel 1-D ladders are linked *via* hydrogen bonds between coordinated Cl⁻ and H–C (the D···A distances are 3.476–3.775 Å), resulting in a 2-D coordination network along the *bc* plane (figure 1b). As shown in figure 1(c), adjacent 2-D coordination networks are stacked along the *a*-axis. Although PHSU²⁻ in **1** and terephthalate in [Co(terephthalate)(bpp)(H₂O)]_n have similar bridging roles, the latter is a 1-D to 3-D polycatenation, indicating influence of axial Cl⁻ on Zn^{II} and the whole coordination network [15].

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

Compound	1	2
Empirical formula	C ₁₈ H ₁₈ N ₂ O ₂ ClZn	C ₃₆ H ₅₂ N ₄ O ₁₄ Ni ₃
Formula weight	395.19	1181.11
Temperature (K)	298	298
Wavelength (Å)	0.71073	0.71073
Crystal size (mm ³)	0.21 × 0.15 × 0.12	0.31 × 0.23 × 0.17
Crystal system	Triclinic	Monoclinic
Space group	<i>P</i> $\bar{1}$	<i>C</i> 2/ <i>m</i>
Unit cell dimensions (Å, °)		
<i>a</i>	5.4781(7)	22.719(2)
<i>b</i>	13.018(2)	12.701(1)
<i>c</i>	15.287(2)	9.6532(8)
α	109.181(2)	90
β	93.861(2)	92.356(8)
γ	92.658(2)	90
Volume (Å ³), <i>Z</i>	1024.6(2), 2	2783.0(4), 2
Calculated density (Mg m ⁻³)	1.279	1.414
Absorption coefficient (mm ⁻¹)	1.339	1.072
<i>F</i> (000)	405	1232
Reflections collected	6730	7839
Independent reflections	3755 [<i>R</i> _{int} = 0.0258]	2762 [<i>R</i> _{int} = 0.0153]
Completeness (%)	98.5	96.4
Goodness-of-fit on <i>F</i> ²	1.125	1.018
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> ₁ ^a = 0.0715, <i>wR</i> ₂ ^b = 0.1912	<i>R</i> ₁ ^a = 0.0566, <i>wR</i> ₁ ^b = 0.1493

$$^a R_1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}$$

$$^b wR_2 = \left[\frac{\sum w(F_o^2 - F_c^2)^2}{\sum w(F_o^2)^2} \right]^{1/2}$$

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

1			
Zn1–O1	1.936(4)	Zn1–N2#1	2.038(4)
Zn1–N1	2.045(4)	Zn1–Cl1	2.252(2)
O1–Zn1–N2#1	110.44(18)	O1–Zn1–N1	112.50(1)
N1–Zn1–Cl1	106.19(4)	N2#1–Zn1–Cl1	105.27(3)
N2#1–Zn1–N1	112.75(7)	O1–Zn1–Cl1	109.29(3)
2			
Ni1–O1	2.039(3)	Ni1–O3	2.092(2)
Ni1–O1W	2.070(3)	Ni1–N1	2.072(3)
Ni2–O2	2.024(3)	O1–Ni1–N1	92.12(1)
Ni2–O3#2	2.036(2)	O1W–Ni1–N1	92.12(1)
Ni1–O3–Ni2	94.04(9)	N1–Ni1–O3	89.24(1)
O1–Ni1–O1W	173.57(2)	Ni2–O2#1	2.024(3)
O1–Ni1–O3	84.94(9)	Ni2–O3#1	2.036(2)
O3–Ni2–O3#3	86.78(2)	O1–Ni1–N1#3	92.11(1)
O1W–Ni1–N1#3	92.13(1)	N1–Ni1–N1#3	97.46(5)
N1–Ni1–O3#3	172.78(1)	N1–Ni1–O3#3	172.79(1)
O1W–Ni1–O3#3	90.29(9)	O2–Ni2–O3#3	87.50(9)
O2–Ni2–O2#1	180.0	O2–Ni2–O3#1	92.50(9)
O2–Ni2–O3	87.50(9)	O2–Ni2–O3#2	92.50(9)
O2–Ni2–O3#1	92.50(9)	O3–Ni2–O3#1	180.0
O1–Ni1–O3	84.94(9)	O1W–Ni1–O3	90.29(9)

Symmetry codes for **1**, #1: *x*, *y* – 1, *z*; #2: –*x* + 1, 1 – *y*, –*z*.

Symmetry codes for **2**, #1: 1 – *x*, 1 + *y*, 1 – *z*; #2: *x* + 3/2, 1/2 – *y*, 1 – *z*; #3: 1/2 – *x*, 3/2 + *y*, –*z*.

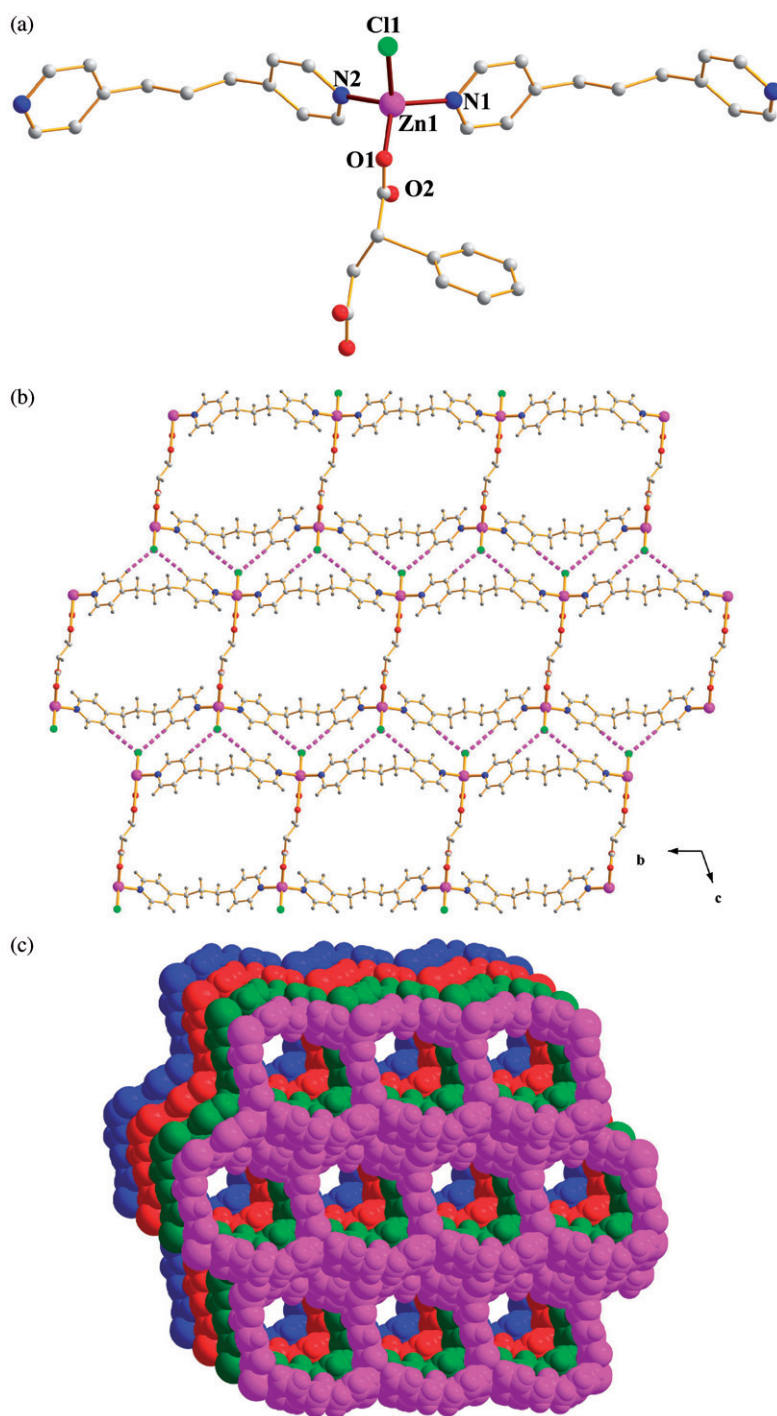


Figure 1. (a) The coordination environment of Zn^{II}, (b) the 2-D coordination network along the *bc* plane linked by hydrogen bonds, and (c) the space-filling model of the stacked coordination networks of **1** (disordered phenyls of PHSU²⁻ are omitted for clarity).

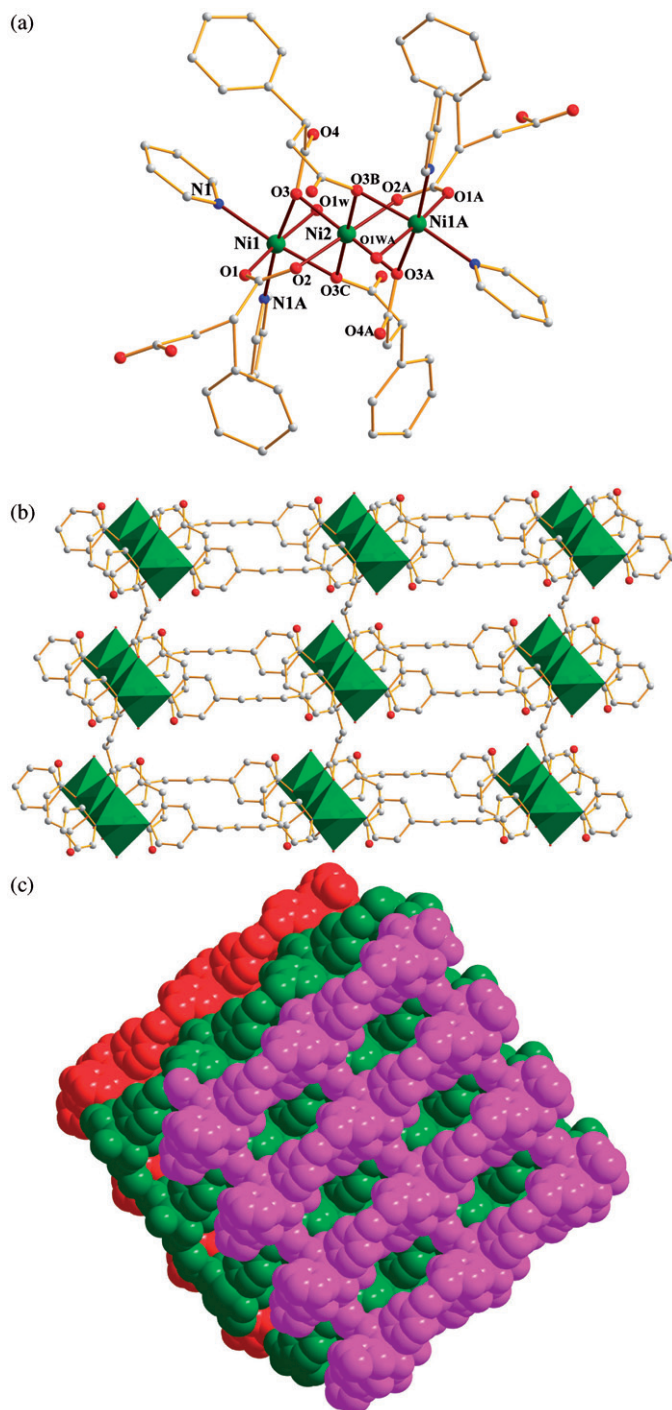


Figure 2. (a) Coordination environment of linear trinuclear Ni_3^{II} , (b) the 2-D coordination network with (4, 4) topology, and (c) the space-filling model of the stacked coordination networks of **2** (disordered phenyls of PHSU^{2-} are omitted for clarity).

3.3. Crystal structure of [Ni₃(PHSU)₃(bpp)₂(H₂O)₂]_n (**2**)

Complex **2** in the monoclinic space group *C2/m* consists of a linear trinuclear Ni₃^{II} cluster coordinated by four PHSU²⁻ in *cis*- and *trans*-conformations, two water molecules and four bpp (figure 2a). PHSU²⁻ with *cis*-conformation is μ_3 -bridging monodentate or μ_4 -bridging bidentate (scheme 1b; the phenyl groups of PHSU²⁻ are disordered in a 1:1 ratio over two positions). All three nickels are octahedral. The central Ni2 sits on a crystallographic inversion center, bonded to four oxygen atoms from two PHSU²⁻ with *cis*-conformations, two oxygen atoms from two PHSU²⁻ with *trans*-conformations. Ni1 and Ni1A are coordinated with two oxygen atoms from two *cis*-PHSU²⁻, one oxygen from *trans*-PHSU²⁻, one coordinated water molecule, Ni1–O1W 2.070(3) Å, and two nitrogen atoms of two bpp with Ni1–N1 distance of 2.072(3) Å. Intramolecular separations of Ni1···Ni2 are 3.020 Å. The Ni1–O3–Ni2 bridging angle is 94.04(9)°. Linear trinuclear clusters are linked into a 1-D chain *via trans*-PHSU²⁻ ligands. The bpp link adjacent 1-D chains into 2-D layers (figure 2b). Adjacent 2-D layers are stacked offset with each other along the *a*-axis (figure 2c). In **2**, the intramolecular Ni···Ni separations are 3.020 Å, longer than the corresponding distances in some other linear trinuclear Ni₃^{II} clusters [16], indicating steric hindrance of PHSU²⁻ and geometric dimensions of the coordination network on structures and properties of these trinickel string complexes.

4. Conclusions

Two coordination polymers have been synthesized by hydrothermal reactions of Zn^{II}/Ni^{II} chloride with flexible PHSU²⁻ and bpp. Compound **1** is a 1-D coordination ladder with tetrahedral Zn^{II}, which is further linked into a 2-D network by hydrogen bonds. Compound **2** displays a 2-D coordination network with octahedral Ni^{II} in linear trinuclear Ni₃^{II} clusters. PHSU²⁻ in **1** and **2** adopt different coordination modes. Compared with other coordination complexes with PHSU²⁻ in *cis*- and *trans*-conformations [16], the flexible linker of bpp extending the coordination unit proves to be an important factor because of its strong coordination ability, suitable length and flexible conformation. Furthermore, the coordination sphere of the metal nodes influences the construction of coordination polymers, and the appropriate match of metal nodes with mixed ligands of flexible aromatic polycarboxylate and dipyridine represents a rational route to coordination polymers.

Supplementary material

Crystallographic information of **1** and **2** has been deposited with the Cambridge Crystallographic Data Centre, CCDC numbers 870064 and 870065. 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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