

Syntheses, Structures and Luminescent Properties of Two Novel M(II)-Phen-SIP Supramolecular Compounds (M = Co, Ni)

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Two metal compounds, [Co(phen)₂(H₂O)₂] \cdot 2H₂SIP \cdot 2H₂O **1** and [Ni(phen)₃] \cdot 2H₂SIP \cdot 3H₂O **2**, have been obtained by incorporating 1,10-phenanthroline (phen) and 5-sulfoisophthalic acid monosodium salt (NaH₂SIP) ligands under hydrothermal conditions. Meanwhile, the two compounds were characterized by element analysis, IR, XRD, TG-DTA and single-crystal X-ray diffraction. Both **1** and **2** present 3D supramolecular structures *via* O–H \cdots O hydrogen bond interactions. Luminescent properties for **1** and **2** were also studied. The compound **1** has two fluorescence emission peaks centered at 398 nm attributed to the intraligand emission from the SIP ligand and at 438 nm assigned to the combined interaction of intraligand π^* - π transitions of the phen ligand and ligand-to-metal-charge-transfer (LMCT) transitions ($\lambda_{\text{ex}} = 233$ nm). The compound **2** shows one emission band centered at 423 nm with a shoulder peak at 434 nm which may be originated from the intraligand π^* - π transitions of the phen ligand ($\lambda_{\text{ex}} = 266$ nm).

Key Words : Hydrothermal synthesis, Crystal structure, Supramolecule, Luminescent properties

Introduction

The design and synthesis of supramolecular architectures have attracted considerable attention from scientific communities owing to their interesting framework topologies as well as potential applications of supramolecular architectures in various fields such as catalysis, luminescence, and gas storage.¹⁻⁶ Many supramolecular architectures have been prepared based on metal ions which can yield various coordination geometries as connected centers and poly-functional organic ligands such as pyridine-type *N*-donors and carboxylate-type *O*-donors as linkers usually.⁷⁻¹⁰ It is well-known that 2,2'-bipyridyl-like ligands, for example 1,10-phenanthroline (phen), are bidentate chelating ligands, which may inhibit the expansion of the polymeric framework to give coordination polymers of low-dimensionality or zero-dimensional molecules.^{11,12} Among carboxylate-type *O*-donors, aromatic multicarboxylate ligands containing sulfonate groups, for example 5-sulfoisophthalic acid (H₃SIP), play an important role in construction of new coordination polymers as a linker.¹³⁻¹⁵ The H₃SIP ligand can manifest various coordination modes with metal ions through two carboxylate and one sulfonate groups, resulting in diverse topologies and multidimensional frameworks.¹⁰ The reactions of Zn(II),¹⁶ Cu(II),^{17,18} Pb(II),¹⁹ Tb(III),²⁰ La(III)²¹ and Eu(III)²² metal ions in combination with NaH₂SIP and phen ligands have been studied. [Co(HSIP)(phen)(H₂O)₃] \cdot H₂O has been reported by B.-Y. Zhang and co-workers in 2008.²³ In 2010, [Co(H₂SIP)(phen)(H₂O)₃] \cdot H₂O has been synthesized by T.-T. Cao and co-workers.²⁴ Our group has also synthesized the similar compound in our previous work. However, luminescent properties of these Co(II) compounds

are not studied. Moreover, to the best of our knowledge, the Ni(II)-phen-SIP system has never been reported in the previous literature. Herein, we chose Co(II)/Ni(II), phen and NaH₂SIP to construct new supramolecular architectures and studied their luminescent properties, respectively. In this context, the new 3D supramolecular compounds [Co(phen)₂(H₂O)₂] \cdot 2H₂SIP \cdot 2H₂O **1** and the first example of [Ni(phen)₃] \cdot 2H₂SIP \cdot 3H₂O **2** were introduced. In addition, luminescent properties for **1** and **2** were also investigated.

Experimental Section

All reagents were purchased from commercial sources and used without further purification. Element analyses were performed on a Perkin-Elmer 2400LS elemental analyzer. IR spectra were recorded from 4000 to 400 cm⁻¹ with a Nicolet AVATAR360 instrument. The thermal gravimetric analyses (TGA) were carried out with a TGA/SDTA851e differential thermal analyzer with a rate of 10 °C/min. Power X-ray diffraction patterns were performed on an ARLX'TRA diffractometer using Cu K α radiation. Emission spectra in the solid state at room temperature were taken on a Perkin-Elmer LS-55 fluorescence spectrophotometer.

Preparation of [Co(phen)₂(H₂O)₂] \cdot 2H₂SIP \cdot 2H₂O **1.** An aqueous water solution (10 mL) of CoCl₂ \cdot 6H₂O (0.095 g, 0.4 mmol), NaH₂SIP (0.053 g, 0.2 mmol) and 1,10-phenanthroline (0.040 g, 0.2 mmol) was dissolved in a 23 mL Teflon-lined stainless steel vessel and heated at 120 °C for 3 days, and then cooled to room temperature. The orange prism-shaped crystals of **1** were obtained. Yield: 31%. Anal. Calcd (%) for **1**: C, 51.34; H, 3.46; N, 5.70; found (%): C, 51.65; H, 3.68; N, 5.90. IR (KBr, cm⁻¹): 3244 w, 1689 m, 1608 w,

1426 w, 1383 w, 1193 s, 1111 s, 1047 s, 1000 w, 851 w, 726 w, 620 w, 458 w.

Preparation of [Ni(phen)₃]₂·2H₂SIP·3H₂O **2.** An aqueous water solution (10 mL) of NiSO₄·6H₂O (0.053 g, 0.2 mmol), NaH₂SIP (0.053 g, 0.2 mmol) and 1,10-phenanthroline (0.079 g, 0.4 mmol) was dissolved in a 23 mL Teflon-lined stainless steel vessel and heated at 120 °C for 5 days, and then cooled to room temperature. The dark red block-shaped crystals of **2** were obtained. Yield: 36%. Anal. Calcd (%) for **2**: C, 54.56; H, 3.50; N, 7.34; found (%): C, 54.78; H, 3.65; N, 7.55. IR (KBr, cm⁻¹): 3412 w, 1711 s, 1624 w, 1606 w, 1518 w, 1426 m, 1384 w, 1236 s, 1204 s, 1184 s, 1141 m, 1105 m, 1042 s, 998 w, 869 w, 848 m, 726 m, 674 w, 624 m, 489 w.

X-ray Crystallography. Single crystal of compounds **1-2** were mounted on a Bruker Smart CCD using Mo K α radiation ($\lambda = 0.71073$ Å) and a graphite monochromator at room temperature. All structures were solved by the direct method and successive Fourier difference syntheses, and refined by the full-matrix least-squares method on F^2 with anisotropic thermal parameters for all non-hydrogen atoms using *SHELXS-97*²⁵ and *SHELXL-97*,²⁶ respectively. The positions of hydrogen atoms were either located by difference Fourier maps or calculated geometrically and their contributions in structural factor calculations were included. The details of the crystal structures and refinements are summarized in Table 1. CCDC Nos. 767946 **1** and 770108 **2** contain the supplementary crystallographic data for compounds **1-2**, respectively. These data can be obtained free of charge from The Cambridge Crystallographic Data

Table 1. Crystallographic data and structure refinement parameters for **1-2**

	1	2
Empirical formal	C ₄₀ H ₃₄ N ₄ O ₁₈ S ₂ Co	C ₅₂ H ₄₀ N ₆ O ₁₇ S ₂ Ni
Mr	981.78	1143.73
Crystal system	Monoclinic	Triclinic
Space group	<i>P</i> 2 ₁ / <i>n</i>	<i>P</i> $\bar{1}$
<i>a</i> /Å	11.4083(2)	14.071(2)
<i>b</i> /Å	29.525(4)	14.309(2)
<i>c</i> /Å	13.1924(2)	15.058(4)
α /°	90.00	100.761(3)
β /°	113.026(2)	98.000(3)
γ /°	90.00	118.659(2)
<i>V</i> /Å ³	4089.5(10)	2519.9(8)
<i>Z</i>	4	2
<i>D</i> _c /Mg·m ⁻³	1.595	1.507
μ /mm ⁻¹	0.608	0.550
θ range	1.38–25.00	1.43–25.00
Unique reflections	25832	17945
Observed reflections	4122	5696
Parameters	592	703
<i>F</i> (000)	2020	1180
<i>R</i> ₁ , <i>wR</i> ₂ [<i>I</i> > 2 σ (<i>I</i>)]	0.0564, 0.1268	0.0569, 0.1545
<i>R</i> ₁ , <i>wR</i> ₂ [all data]	0.1158, 0.1591	0.0944, 0.1853
GOF	1.025	1.023
Largest peak and hole (e Å ⁻³)	0.668, -0.574	0.921, -0.605

Centre via www.ccdc.cam.ac.uk/data_request/cif.

Results and Discussion

Descriptions of Crystal Structures.

[Co(phen)₂(H₂O)₂]₂·2H₂SIP·2H₂O **1:** The crystal structure of **1** is depicted in Figure 1, and the asymmetric unit consists of one Co(II) ion, two phen molecules, two coordinated water molecules, two H₂SIP⁻ ligands and two lattice water molecules. Co(1) ion, which is bonded to four N atoms (N(1), N(2), N(3), N(4)) from two phen molecules and two O atoms (O(1), O(2)) from two coordinated water molecules, displays a distorted [CoN₄O₂] octahedral coordination geometry. In the octahedral coordination geometry, N(1), N(2), N(4) and O(2) atoms lie in the equatorial position, while O(1) and N(3) atoms occupy the axial direction. The Co-N bond lengths are in the range of 2.122(4)–2.156(4) Å, and the Co-O bond distances are 2.090(4) Å for

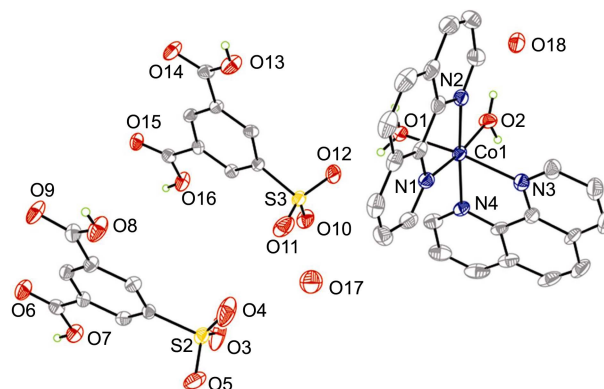


Figure 1. View of **1** with 30% probability displacement ellipsoids (H atoms are omitted for clarity).

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

1			
Co1–O1	2.090(4)	Co1–N2	2.145(4)
Co1–O2	2.083(4)	Co1–N3	2.156(4)
Co1–N1	2.122(4)	Co1–N4	2.135(4)
O1–Co1–O2	88.79(1)	N1–Co1–N2	78.56(2)
O1–Co1–N1	88.61(1)	N1–Co1–N3	92.48(2)
O1–Co1–N2	89.99(1)	N1–Co1–N4	99.10(2)
O1–Co1–N3	171.92(2)	N2–Co1–N3	98.08(1)
O1–Co1–N4	94.48(1)	N2–Co1–N4	174.92(1)
O2–Co1–N2	94.30(2)	O2–Co1–N4	88.23(2)
2			
Ni1–N1	2.106(4)	Ni1–N4	2.089(4)
Ni1–N2	2.106(5)	Ni1–N5	2.071(4)
Ni1–N3	2.096(4)	Ni1–N6	2.086(3)
N1–Ni1–N2	79.27(2)	N2–Ni1–N6	91.84(2)
N1–Ni1–N3	97.50(1)	N2–Ni1–N3	172.33(2)
N1–Ni1–N4	92.44(1)	N2–Ni1–N4	93.85(2)
N1–Ni1–N5	94.42(1)	N2–Ni1–N5	93.84(2)
N1–Ni1–N6	168.89(1)		

Table 3. Hydrogen bond lengths (Å) and angles (°) for **1-2**

	D–H	H···A	D···A	D–H···A
1				
O1–H1A···O12	0.85	1.88	2.666(6)	153
O1–H1B···O5 ^{#1}	0.86	1.84	2.670(6)	162
O2–H2C···O18	0.85	1.87	2.691(6)	162
O2–H2D···O15 ^{#2}	0.85	1.92	2.726(5)	159
O7–H7O···O10 ^{#3}	0.85	1.77	2.597(5)	163
O8–H8O···O14 ^{#4}	0.79(6)	1.81(6)	2.595(5)	170(7)
O13–H13O···O9 ^{#4}	0.86	1.76	2.612(5)	173
O16–H16O···O3 ^{#5}	0.84	1.77	2.612(6)	177
O17–H17B···O4	0.85	2.01	2.852(7)	173
O17–H17A···O11	0.87(6)	1.99(7)	2.783(6)	151(7)
O18–H18A···O17 ^{#1}	0.86	1.98	2.767(6)	151
O18–H18B···O6 ^{#6}	0.86	2.09	2.903(6)	159
C6–H6···O5 ^{#7}	0.93	2.52	3.379(9)	153
C12–H12···O18	0.93	2.49	3.410(7)	172
2				
O4–H4A···O17 ^{#1}	0.85	1.97	2.813(1)	173
O6–H6···O9 ^{#2}	0.82	1.77	2.570(6)	165
O11–H11A···O12 ^{#3}	0.85	1.80	2.639(6)	169
O14–H14A···O15 ^{#4}	0.76	1.91	2.547(7)	142
O15–H15A···O2	0.79	1.95	2.704(7)	159
O15–H15B···O1 ^{#5}	0.85	1.94	2.757(6)	161
O16–H16A···O17	0.92	2.30	2.808(1)	114
O17–H17A···O7 ^{#5}	0.85	2.17	2.984(9)	160
O17–H17B···O8 ^{#3}	0.85	1.97	2.783(8)	160
C11–H11···O10 ^{#6}	0.93	2.56	3.278(9)	134
C23–H23···O1 ^{#7}	0.93	2.58	3.264(8)	131
C24–H24···O5 ^{#8}	0.93	2.57	3.280(7)	134
C25–H25···O10 ^{#2}	0.93	2.42	3.207(5)	142
C26–H26···O8 ^{#9}	0.93	2.58	3.079(8)	114
C34–H34···O3 ^{#10}	0.93	2.59	3.287(8)	132
C36–H36···O13 ^{#5}	0.93	2.40	3.200(7)	144

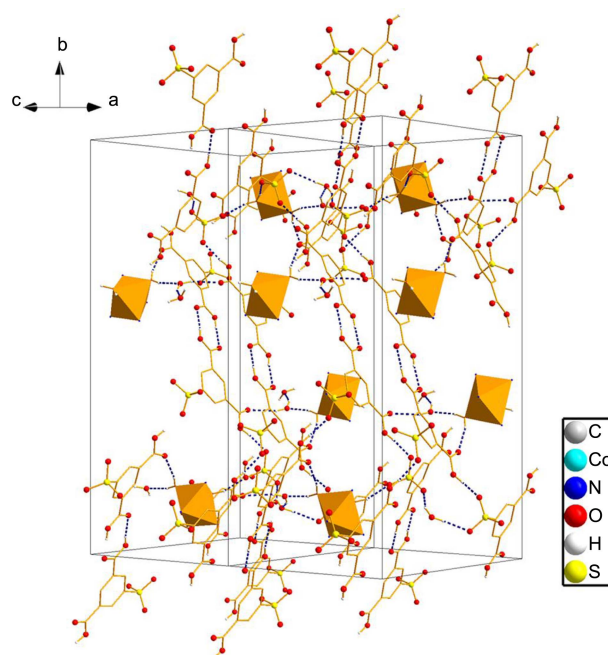
Symmetry code for **1**: #1 $x-1, y, z$; #2 $x, y, z+1$; #3 $x+1/2, -y+1/2, z-1/2$; #4 $-x+1, -y, -z$; #5 $x-1/2, -y+1/2, z-1/2$; #6 $x-1, y, z+1$; #7 $2-x, -y, 1-z$. **2**: #1 $x, y, z-1$; #2 $-x+1, -y+1, -z$; #3 $-x+1, -y, -z+1$; #4 $x+1, y, z$; #5 $-x+1, -y+1, -z+1$; #6 $-1+x, 1+y, z$; #7 $-1+x, y, z$; #8 $-x, 1-y, -z$; #9 $x, 1+y, z$; #10 $1-x, 2-y, 1-z$.

O(1), and 2.083(4) Å for O(2), respectively. These bond lengths are similar to those reported previously.^{23,24} The selected bond lengths and angles for **1** are listed in Table 2.

Table 4. Aromatic interactions in **1-2**

Ring atoms	Dihedral angle (°)	Distance (Å)
1		
[N(3), C(13), C(14), C(15), C(16), C(17)]...[C(25), C(26), C(27), C(28), C(29), C(30)] ^{#1}	8.1(3)	3.799(3)
[N(3), C(13), C(14), C(15), C(16), C(17)]...[C(33), C(34), C(35), C(36), C(37), C(38)] ^{#1}	3.5(3)	3.699(3)
[C(4), C(5), C(9), C(8), C(7), C(6)]...[C(4), C(5), C(9), C(8), C(7), C(6)] ^{#2}	0.0	3.843(3)
[C(16), C(17), C(24), C(20), C(19), C(18)]...[C(25), C(26), C(27), C(28), C(29), C(30)] ^{#1}	7.0(3)	3.839(3)
2		
[N(2), C(6), C(7), C(10), C(11), C(12)]...[N(2), C(6), C(7), C(10), C(11), C(12)] ^{#1}	0.0	3.952(4)
[N(2), C(6), C(7), C(10), C(11), C(12)]...[C(4), C(5), C(6), C(7), C(8), C(9)] ^{#1}	0.8(3)	3.937(4)
[C(37), C(38), C(39), C(40), C(41), C(42)]...[C(4), C(5), C(6), C(7), C(8), C(9)] ^{#2}	12.3(3)	3.783(4)

Symmetry code for **1**: #1 $x, y, 1+z$; #2 $1-x, -y, 1-z$. **2**: #1 $-x, 2-y, -z$; #2 $-x, 1-y, -z$.

**Figure 2.** 3D hydrogen-bonded framework (C atoms of phen ligands are omitted for clarity).

In the compound **1**, the SIP units are not coordinated with metal ion. For the sake of charge balance, the two carboxylate groups of the uncoordinated SIP ligands should be protonated H_2SIP^- .

In the structure, there exist hydrogen bonds, involving O(17) and O(18) atoms of the lattice water molecules, O(1) and O(2) atoms of the coordinated water molecules and oxygen atoms from the uncoordinated H_2SIP^- (Table 3). Each $[\text{CoN}_4\text{O}_2]$ unit is linked to the H_2SIP^- ligands by the O(1)–H(1A)···O(12) (2.665(6) Å, 153°), O(1)–H(1B)···O(5)^{#1} (2.670(6) Å, 162°), O(2)–H(2D)···O(15)^{#2} (2.726(5) Å, 159°) hydrogen bonds between the coordinated water molecules and oxygen atoms of H_2SIP^- . The H_2SIP^- ligands connect with each other through the hydrogen bonds (2.595(5)–2.612(6) Å, 163–177°) between H_2SIP^- ligands. There are also hydrogen bonds (2.691(6)–2.903(6) Å, 151–173°) between the lattice water molecules and the coordinated water molecules and between the lattice water molecules and oxygen atoms of H_2SIP^- . The $[\text{CoN}_4\text{O}_2]$ units connect with

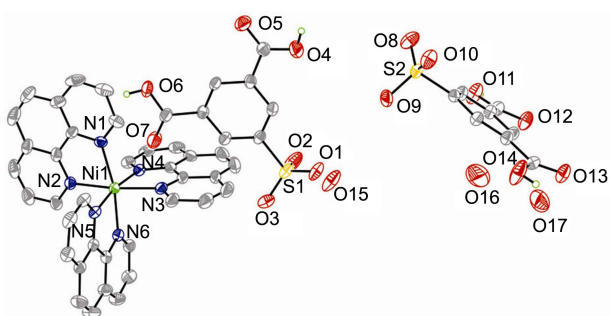
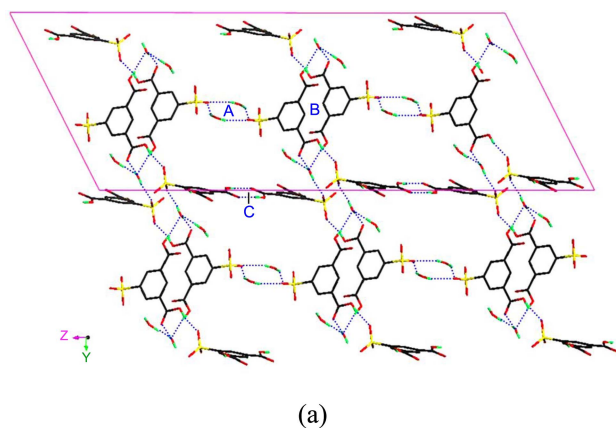


Figure 3. View of **2** with 30% probability displacement ellipsoids (H atoms are omitted for clarity).

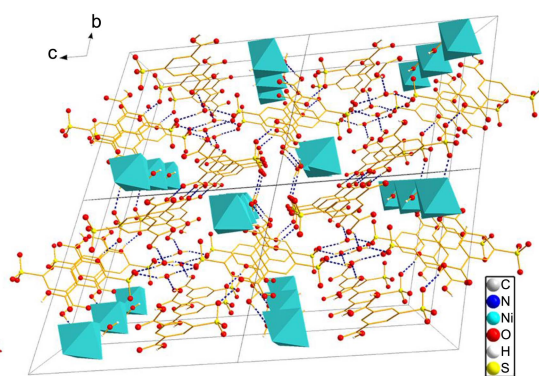
each other through these extensive hydrogen bond interactions to construct a 3D supramolecular framework (Figure 2). In **1**, there are also π - π interactions between the phen ligands and between the aromatic rings of the SIP and the phen. And the distances and dihedral angles are in the range of 3.699(3)-3.843(3) Å and 0.0-8.1(3) $^\circ$, respectively (Table 4), which further stabilize the 3D framework of **1**.

[Ni(phen)₃]·2H₂SIP·3H₂O **2:** The asymmetric unit of **2** comprises one Ni(II) ion, three phen molecules, two H₂SIP⁻ ligands and three lattice water molecules (Figure 3). The Ni(II) ion shows a distorted octahedral geometry coordination environment containing six N atoms (N(1), N(2), N(3), N(4), N(5), N(6)) from three phen molecules. The Ni-N (2.071(4)-2.106(5) Å) bond lengths are comparable to those reported previously.²⁷ The Ni-N bond lengths and the N-Ni-N angles are summarized in Table 2.

Both two H₂SIP⁻ ligands which take part in the formation of hydrogen bonds are also uncoordinated, similar to that of **1**. In the structure of **2**, 1D anionic chain is formed *via* hydrogen bonds (2.704(7)-2.984(9) Å, 159-173 $^\circ$) between oxygen atoms of H₂SIP⁻ and the lattice water molecules along the *c* direction. Within the 1D chain, there are the 12- and 20-membered rings (marked A and B, respectively). The 1D chain through the O(11)-H(11A)⋯O(12)^{#3} (2.639(6) Å, 169 $^\circ$), O(6)-H(6)⋯O(9)^{#2} (2.570(6) Å, 165 $^\circ$) and O(17)-H(17B)⋯O(8)^{#3} (2.783(8) Å, 160 $^\circ$) hydrogen bonds generate 2D supramolecular anionic layer with 8-membered rings marked C in the *bc* plane (Figure 4(a)). The O(14)-H(14A)



(a)



(b)

Figure 4. View of compound **2**: (a) 1D chain along the *c* direction and 2D layer in the *bc* plane ([Ni(phen)₃]²⁺ are omitted) (b) 3D framework (C atoms of phen ligands are omitted for clarity).

⋯O(15)^{#4} (2.547(7) Å, 142 $^\circ$) hydrogen bonds link 2D layers into 3D supramolecular framework, as illustrated in Figure 4(b). In addition, [Ni(phen)₃]²⁺ are placed in voids of the framework *via* the C-H⋯O (3.079(8)-3.287(8) Å, 114-144 $^\circ$) hydrogen bonds. The hydrogen bonds for **2** are listed in Table 3. The 3D supramolecular framework is further stabilized by π - π interactions. The π - π interactions are observed between the rings of the phen ligands and the aromatic rings of the SIP ligands and between the rings of the phen ligands. And the corresponding distances and dihedral angles are in the range of 3.783(4)-3.952(4) Å and 0.0-12.3(3) $^\circ$, respectively (Table 4).

Powder XRD Analysis. In order to check the phase purity, the PXRD patterns of title compounds **1-2** were checked at room temperature, in Figure S1 (Supporting Information). The experimental patterns correspond well with the simulated results, indicating the good phase purity of the compounds. The difference in reflection intensities between the simulated and experimental patterns was due to the variation in preferred orientation of the powder samples during the collection of the experimental data.

Thermal Analysis. To estimate the stability of the coordination architectures, TGA experiments were carried out (Figure 5). The TGA curves of **1** and **2** show that **1** released

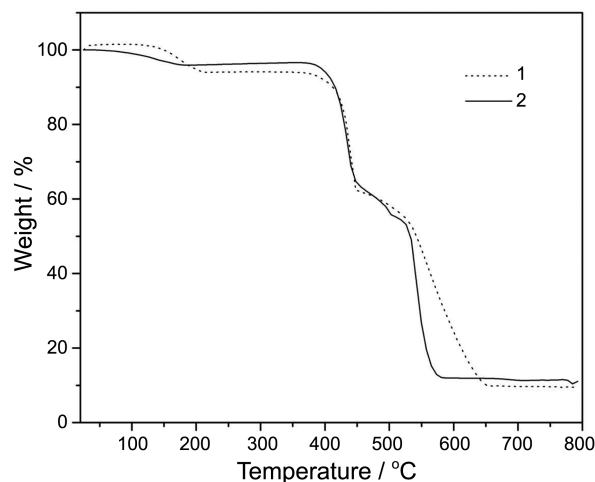


Figure 5. TGA curves of **1** and **2**.

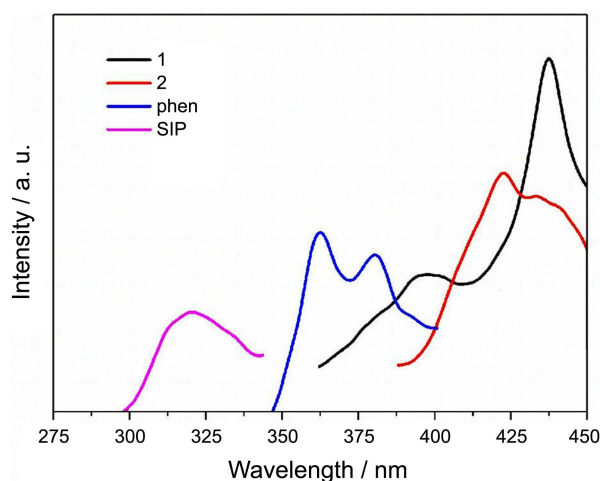


Figure 6. Solid-state emission spectra of 1-2.

two lattice water molecules and two coordinated water molecules at 25-210 °C (exp. 6.62%, calcd. 7.33%), while **2** lost three free water molecules from 25-200 °C (exp. 4.04%, calcd. 4.72%). Then there is a platform with no weight loss for **1** and **2**. For **1**, the framework began to collapse with the decomposition of the phen molecules and the SIP ligand after 340 °C (exp. 33.6%, calcd. 36.67%). After 470 °C, SIP organic groups were continuously decomposed. The total weight loss for **1** is 90.09% (calcd. 93.90%). The final residue of **1** corresponds to CoO. For **2**, the framework started to collapse with the decomposition of the phen molecules and the SIP ligand after 350 °C. The total weight loss in the range of 350 °C to 600 °C is 84.64% (calcd. 76.06%). The final residue of **2** corresponds to NiO, and the decomposing process is not completed.

Luminescent Properties. The luminescent properties of **1** and **2** have been measured in the solid state at room temperature. As shown in Figure 6, the free NaH₂SIP exhibits an emission peak at 321 nm ($\lambda_{\text{ex}} = 238$ nm), whereas the free phen molecule displays two intense emission bands at 363 and 381 nm ($\lambda_{\text{ex}} = 234$ nm), which may be attributed to the $\pi^* \rightarrow \pi$ transition. In Figure 6, both **1** and **2** have fluorescence emission peaks at 398 nm and 438 nm ($\lambda_{\text{ex}} = 233$ nm) for **1**, 423 nm with a shoulder peak (434 nm) ($\lambda_{\text{ex}} = 266$ nm) for **2**, respectively. In comparison with those of the free ligands and based on the shape of emission peaks, the emission band at 398 nm of **1** may be attributed to the intraligand emission from the SIP ligand, while the emission peak at 438 nm may originate from the combined interaction of intraligand $\pi^* \rightarrow \pi$ transitions of the phen ligand and ligand-to-metal-charge-transfer (LMCT) transitions. For **2**, the emission band at 423 nm with a shoulder at 434 nm may be ascribed to the intraligand $\pi^* \rightarrow \pi$ transitions of the phen ligand. In comparison with the free ligands, the red-shift of the emission bands in **1** and **2** are mainly due to intermolecular interactions.¹⁶

Conclusion

Two new supramolecular architectures with phen and

NaH₂SIP have been successfully synthesized under hydrothermal conditions. The results show that not only the H₂SIP units play a significant role in the formation of hydrogen bonds, but also the final extended structures are influenced by the noncovalent interactions. Furthermore, both the compounds show good fluorescence properties at room temperature. It is anticipated that more metal compounds containing the *N*-heterocycle and SIP ligands will be synthesized.

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