Syntheses, Structures, and Properties of Two-Dimensional Honeycomb Networks from the Assembly of the Tripodal Ligand 2,4,6-Tris[4-(imidazol-1-ylmethyl)phenyl]-1,3,5-triazine with Metal Salts

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Two novel coordination networks, $[Zn_2(timpt)_2(\mu-OH)](NO_3)_3$ (**1**) and $[Ni_2(timpt)_2(H_2O)_2(SO_4)_2]\cdot0.5H_2O$ (**2**) were obtained by assembly of 2,4,6-tris[4-(imidazol-1-ylmethyl)phenyl]-1,3,5-triazine (timpt) with the corresponding metal salts. The X-ray single-crystal diffraction analysis reveals that complex **1** possesses μ-OH-bridged dinuclear zinc(II) subunits (Zn_2OH) and each timpt ligand connects three metal atoms to form a two-dimensional (2D) honeycomb network structure, in which each independent sheet contains two sublayers. While in the case of complex **2**, the nickel(II) atom is coordinated by N atoms of timpt, O atoms of sulfate anions and

water molecules to give a 2D honeycomb network. The luminescence properties of timpt, **1** and **2** in the solid state were investigated at room temperature. The maximum emission wavelength of **1** is red-shifted by 17 nm compared with that of timpt. While complex **2** showed an emission with remarkably reduced intensity under the same conditions. The results imply that the metal ions have great influence on the structure and properties of the complexes.

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Introduction

Employing multidentate organic ligands and suitable metal salts to construct supramolecular architectures has been a major strategy in recent years.[1] Metal-organic frameworks (MOFs) with specific topologies such as cage-like structures, honeycomb and interpenetrating networks have been obtained by self-assembly of suitable metal salts with rigid or flexible tripodal ligands, for example 1,3,5-tricyanobenzene,[2] 1,3,5-tris(4-ethynylbenzonitrile)benzene,[3] 2,4,6-tris(4-pyridyl)-1,3,5-triazine,[4] 1,3,5-benzenetricarboxylate,[5] 1,3,5-benzenetribenzoate,[6] 1,3,5-tris(pyrazol-1-ylmethyl)-2,4,6-triethylbenzene,[7] 1,3,5-tris(4-pyridylmethyl)benzene,[8] 1,3,5-tris(benzimidazol-1-ylmethyl)-2,4,6-trimethylbenzene.[9] In contrast to the rigid tripodal ligands with little or no conformational changes when they interact with metal salts, the flexible tripodal ligands have much more possible coordination modes than the rigid ones due to their flexibility and low symmetry, since the flexible ligands can adopt different conformations according to the geo-

metric needs of different metal ions. Accordingly, we designed and synthesized a series of flexible tripodal ligands with aromatic core rings as spacers and systematically investigated their assembly reactions with metal salts.[10] Most recently, we prepared a novel nanometer-sized tripodal ligand 2,4,6-tris[4-(imidazol-1-ylmethyl)phenyl]-1,3,5-triazine (timpt) which reacted with $Pb(NO_3)_2$ to give a two-dimensional (2D) 4.8^2 network with threefold interpenetrations.[11] We report herein two novel 2D honeycomb coordination polymers $[Zn_2(timpt)_2(\mu-OH)](NO_3)_3$ (**1**) and $[Ni(timpt)_2(H_2O)_2(SO_4)_2]\cdot0.5H_2O$ (**2**) obtained by solvothermal reactions of timpt with $Zn(NO_3)_2\cdot6H_2O$ and $Ni(SO_4)_2\cdot7H_2O$, respectively.

Results and Discussion

Crystal Structure Description

The structures of complexes **1** and **2** were determined by X-ray crystallography. The crystal and data collection parameters are summarized in Table 3 and selected bond lengths and angles are listed in Table 1.

Crystal Structure of 1

The complex crystallizes in rhombohedral space group of $R\bar{3}c$ and all the zinc(II) atoms have a tetrahedral coordination geometry with an N_3O donor set as illustrated in Figure 1. The three N atoms are from the imidazole groups

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Table 1. Selected bond lengths [Å] and angles [°] for **1** and **2**

$[Zn_2(timpt)_2(\mu\text{-}OH)](NO_3)_3$ (**1**)			
Zn(1)−O(1)	1.874(2)	Zn(1)−N(1)	2.001(3)
O(1)−Zn(1)−N(1)[a]	104.3(2)	N(1)−Zn(1)−N(1)[a]	106.95(11)

$[Ni(timpt)_2(H_2O)_2(SO_4)_2]\cdot0.5H_2O$ (**2**)			
Ni(1)−N(12)	2.033(5)	Ni(1)−N(112)	2.038(4)
Ni(1)−N(22)	2.078(4)	Ni(1)−O(11)	2.049(4)
Ni(1)−O(1)	2.225(4)	Ni(1)−O(2)	2.152(4)
Ni(2)−N(32A)[b]	2.018(4)	Ni(2)−N(122)	1.978(5)
Ni(2)−N(132)	2.024(4)	Ni(2)−O(21)	2.058(4)
Ni(2)−O(22)	2.067(3)		
N(12)−Ni(1)−N(112)	97.50(18)	N(112)−Ni(1)−O(11)	91.50(16)
N(12)−Ni(1)−O(11)	170.49(17)	N(112)−Ni(1)−N(22)	97.10(17)
N(12)−Ni(1)−N(22)	96.88(18)	N(12)−Ni(1)−O(2)	81.18(19)
O(11)−Ni(1)−N(22)	85.01(17)	O(11)−Ni(1)−O(2)	89.42(18)
N(112)−Ni(1)−O(2)	169.87(17)	N(12)−Ni(1)−O(1)	91.46(17)
N(22)−Ni(1)−O(2)	93.03(18)	O(11)−Ni(1)−O(1)	86.06(16)
N(112)−Ni(1)−O(1)	86.05(16)	O(2)−Ni(1)−O(1)	83.95(16)
N(22)−Ni(1)−O(1)	170.60(18)	N(122)−Ni(2)−N(132)	99.09(16)
N(122)−Ni(2)−N(32A)[b]	97.30(16)	N(122)−Ni(2)−O(21)	107.82(17)
N(32A)−Ni(2)−N(132)[b]	100.70(16)	N(132)−Ni(2)−O(21)	148.54(16)
N(32A)−Ni(2)−O(21)[b]	91.90(15)	N(32A)−Ni(2)−O(22)[b]	157.23(16)
N(122)−Ni(2)−O(22)	99.97(15)	N(132)−Ni(2)−O(22)	91.13(14)
		O(21)−Ni(2)−O(22)	68.74(14)

[a] Equivalent atoms generated by $-x + y, -x + 1, z$. [b] Equivalent atoms generated by $x, y - 1, z - 1$.
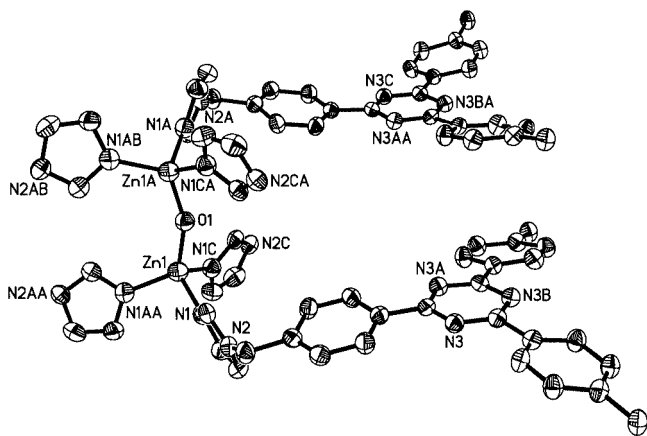

Figure 1. The coordination environment of Zn^{II} in **1**; thermal ellipsoids are drawn at 30% probability; the carbon atoms are unlabelled and hydrogen atoms are omitted for clarity

of three different timpt ligands with the Zn−N bond lengths of 2.001(3) Å. The fourth site was completed by an O atom from a hydroxy group which connects another zinc(II) atom to form a μ-hydroxy bridge. The Zn−O bond length is 1.874(2) Å which is slightly shorter than the Zn−O distances in the reported hydroxy-bridged zinc(II) complexes.[12] The Zn−O−Zn bond angle is 156.7° and the Zn⋯Zn distance is 3.67 Å, which are within the ranges of the reported Zn−O−Zn angles and Zn⋯Zn separations in the hydroxy-bridged zinc(II) complexes.[12] The N−Zn−N and N−Zn−O bond angles are ranging from 106.95(11)

to 104.3(2)°, which are close to the ideal tetrahedral angle of 109.5°.

Each timpt ligand coordinated to three different zinc(II) atoms to form an equilateral triangle with an edge length (Zn⋯Zn separation) of 17.57 Å. Such units repeated along the *ab* plane to generate an infinite 2D honeycomb network as shown in Figure 2 (part a). It is noteworthy that two such networks are bridged by hydroxy groups through the Zn−O coordination bonds to form an independent layer (Figure 2, b). Namely, every independent 2D layer in complex **1** contains two sublayers which are connected by the hydroxy bridges. This is, to the best of our knowledge, the first example of 2D honeycomb networks in which each independent layer contains two sublayers linked by μ-hydroxy bridges through the M−O(H)−M (M represents the metal ion) bonds. Disordered nitrate anions are located between the two sublayers of an independent layer and held there by O(1B)−H(1AB)⋯O(3) (NO_3^-) with an O(1B)⋯O(3) distance of 3.269(12) Å and C−H⋯O (NO_3^-) hydrogen bonds with C⋯O distances of 3.381(15) and 3.398(11) Å (Figure 3, a). The 2D independent layers are closely stacked with each other and connected by the NO_3^- anions through the C−H⋯O hydrogen bond with C⋯O distance of 3.212(8) Å to produce a 3D framework as exhibited in Figure 3 (part b). The hydrogen-bonding data are summarized in Table 2.

In addition to the hydrogen bonds as mentioned above, there are weak π−π interactions between the benzene rings of the timpt ligands. The two benzene rings from two different ligands are nearly parallel with a dihedral angle of 2.2° and a centroid-to-centroid distance of 4.28 Å. The two tri-
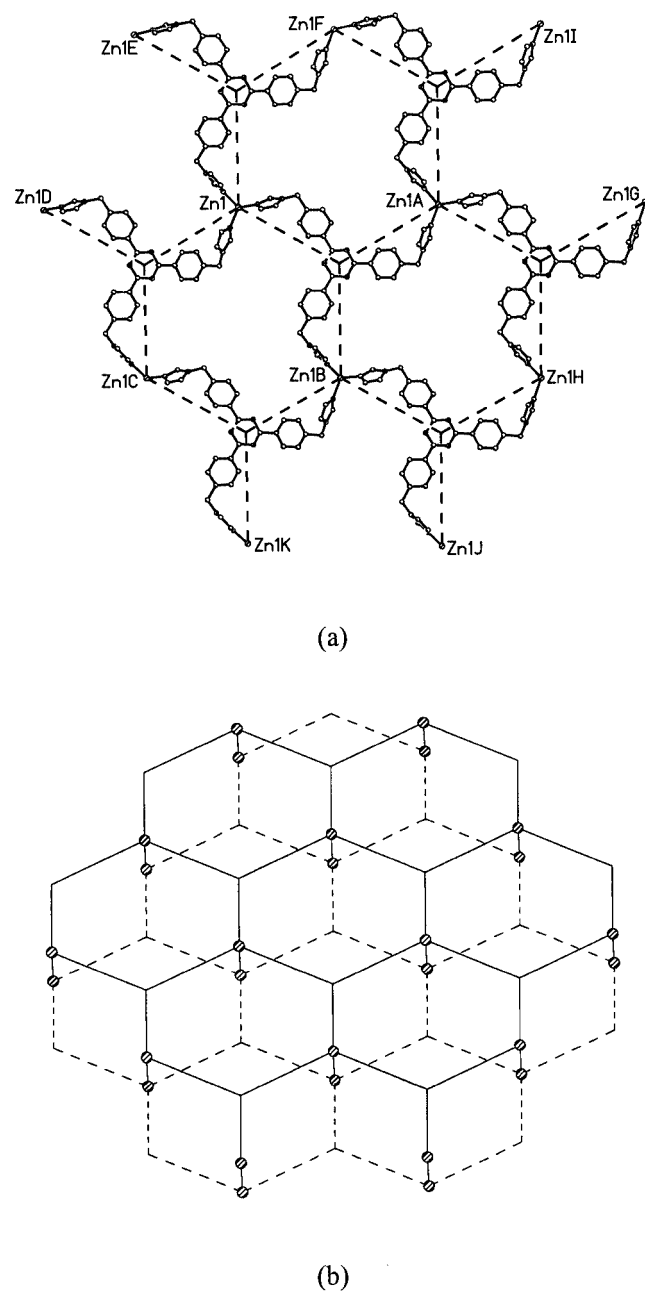
(a)

(b)

Figure 2. (a) The perspective view of the 2D cationic sublayer with the honeycomb structure (dashed lines) in the *ab* plane; (b) schematic drawing of 2D honeycomb network in which the timpt ligands are represented by three spokes radiating from a point and Zn centers by circles; the hydroxy bridges (Zn−O−Zn) are represented by solid lines; the solid and dashed lines refer to timpt ligands of each sublayer

azine rings of the two ligands of the neighboring sublayers of the two different layers adopt a face-to-face conformation and are strictly parallel to each other with a centroid-to-centroid distance of 3.44 Å, indicating the presence of π−π stacking interactions (Figure 4). The hydrogen bonding and π−π interactions as well as Zn−O−Zn bridges may play an important role in stabilizing the layered structures in the titled complex.

Crystal Structure of 2

In contrast to the tetrahedral coordination geometry of the zinc(II) atom in complex **1**, nickel(II) atoms usually adopt octahedral geometry when they interact with N-donor ligands. Thus, the reaction of timpt with nickel(II) sulfate was carried out and complex **2** was obtained. From the X-ray analysis, it is interesting that there are two kinds of Ni^{II} atoms with different coordination environments: the Ni(1) atom is six-coordinate with a distorted octahedral geometry while the Ni(2) atom is five-coordinate with a distorted trigonal-bipyramidal geometry as shown in Figure 5. The Ni1 atom lies in the plane defined by the O(1), O(2), N(22), and N(112) atoms since the deviation of Ni(1) from this plane is 0.08 Å. Three of the six sites of Ni(1) are coordinated by three N atoms of imidazole groups from three different timpt ligands with Ni−N bond lengths varying from 2.033(5) to 2.078(4) Å (Table 1), and the remaining three sites are occupied by O atoms from a monodentate sulfate anion and two water molecules. The Ni(2) atom lies above the plane of the O(21), N(122), and N(132) atoms by 0.23 Å. The three sites of Ni(2) are coordinated by three imidazole-group N atoms with Ni−N bond lengths ranging from 1.978(5) to 2.024(4) Å, while the additional two sites are completed by two O atoms of a sulfate anion in a bidentate mode.

An infinite 2D honeycomb network sheet of **2** is achieved by the coordination of the timpt ligands to Ni^{II} atoms as shown in Figure 6. The triangles formed by three Ni^{II} atoms connected by one timpt ligand are non-equilateral which are different from those observed in complex **1**. Furthermore, there are two kinds of triangles in **2**: one is formed by two five-coordinate Ni atoms and one six-coordinate Ni^{II} atom with the edge lengths of 19.85, 19.52, and 16.47 Å, respectively, e.g. Ni(2), Ni(2B), and Ni(1B) in Figure 6; another one is formed by one five-coordinate Ni^{II} atom and two six-coordinate Ni^{II} atoms [e.g. Ni(2B), Ni(1A), and Ni(1C) in Figure 6] with the edge lengths of 19.19, 19.11, and 16.47 Å, respectively. Disordered water molecules are located in the voids between the adjacent sheets through C−H···O hydrogen bonds (Table 2). The 2D honeycomb sheets are linked through the C−H···O (coordinated water molecules and sulfate anions) hydrogen-bonding interactions to form a 3D structure (Figure 7).

Face-to-face π−π interactions were found between the triazine rings of the two layers since the two triazine ring planes are strictly parallel to each other with a centroid-to-centroid distance of 3.86 Å. In addition, π−π interactions are also found between the triazine rings and the benzene rings of two adjacent layers. The dihedral angle between them is 11.8° and the centroid-to-centroid separation is 3.88 Å (Figure S1 in Supporting Information, see footnote on page 1 of this article).

Although both complexes **1** and **2** have the honeycomb network structures, there are distinct differences between these two complexes. First, they crystallize in different crystal systems with **1** in rhombohedral $R\bar{3}c$ and **2** in triclinic $P\bar{1}$. Another important difference is that each independent layer in **1** contains two sublayers which feature the honey-
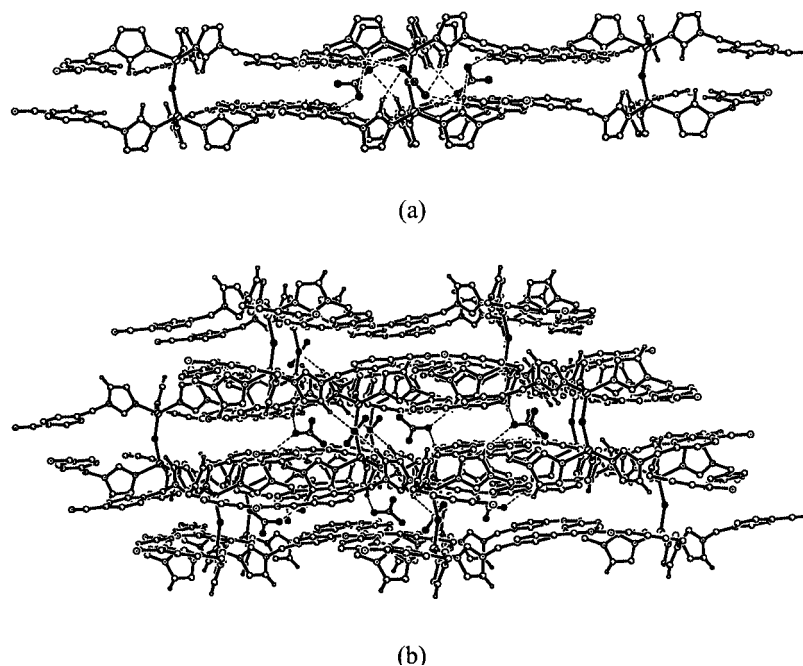
(a)

(b)

Figure 3. (a) View of an independent layer with nitrate anions located within the layer through hydrogen bonds (dashed lines); (b) crystal packing diagram of **1**; the two adjacent independent layers are connected by nitrate anions through hydrogen-bonding interactions

comb structures and are connected by hydroxy bridges, while **2** contains no sublayers. Furthermore, the conformations of the timpt ligand in **1** and **2** are obviously different. In complex **1**, the dihedral angle between the central triazine ring plane and the benzene ring plane is 12.6°. While the one between the terminal imidazole ring plane and the benzene ring plane is 109.4°, indicating a near perpendicular orientation of the imidazole ring plane to the benzene ring plane (Figure 2, a). The plane defined by three coordinated imidazolyl N atoms of one timpt ligand is parallel to the triazine ring plane with a separation of 1.35 Å. In the previously reported metal complexes of 1,3,5-tris(imidazol-1-ylmethyl)-2,4,6-trimethylbenzene (timtb) with *cis,-cis,cis* conformation, the distance between the plane defined by three coordinated imidazolyl N atoms and the central benzene ring plane is ca. 3.1 Å.[10f,10g] Therefore, the conformation of timpt in **1** is a propeller-type rather than *cis,cis,cis* or *cis,trans,trans* as observed in complexes of timtb (Scheme 1).[10] In complex **2**, the three coordinated imidazolyl N atoms of the timpt ligand are at both sides of the central triazine ring plane and the dihedral angles between the plane defined by the three N atoms and the triazine ring plane are 16.9 and 19.8°, respectively, since there are two kinds of timpt ligand in **2** as mentioned above. This causes the backbone of complex **2** to be different from that of **1**. In addition, the vacancies between the two sublayers in **1** are occupied by nitrate anions, while in the case of **2**, the sulfate anions are coordinated with the Ni[II] atoms and the voids between the 2D layers are filled with disordered lattice water molecules.

In the reported 2D coordination polymers with honeycomb structures, rigid tripodal ligands, e.g., 1,3,5-tricyanobenzene or 1,3,5-benzenetricarboxylate, enclosing an angle of 120° were used to link the linear metal centers.[2,5] In our previously reported honeycomb complexes, the flexible tripodal ligand timtb with the *cis,cis,cis* conformation, connected three six-coordinate metal centers to direct the formation of the 2D honeycomb network.[10f] However, in the present case, the metal atom in **1** is four-coordinate with three sites occupied by three N atoms from three ligands and the fourth site finished by a hydroxy O atom. If the Zn−O−Zn units are considered as one metal center M, then the metal center M is six-coordinate and the backbone of **1** is identical with those of the complexes with honeycomb structures reported previously.[10f] However, the backbone of **2** is different from **1** and other reported complexes with timtb ligand.[10f]
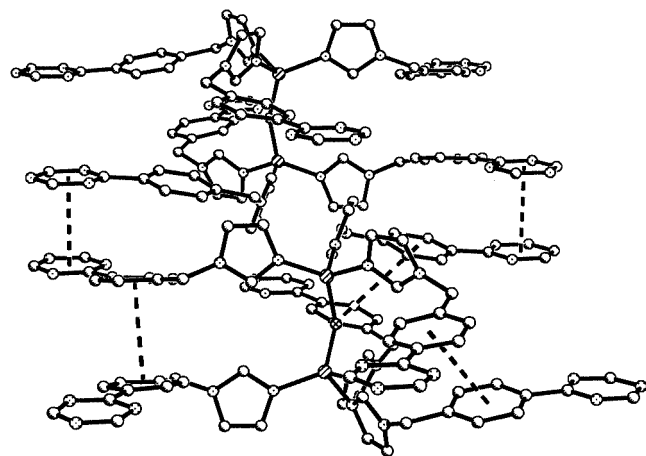
Luminescent Properties

The photoluminescence studies of timpt, **1** and **2** in the solid state under the same conditions showed that, although the maximum-excitation wavelengths of the complexes **1** and **2** are the same as that of the timpt ligand at 396 nm, the maximum emission wavelength of **1** is red-shifted by 17 nm from 443 to 460 nm compared with that of the ligand, accompanied by an increase of luminescent intensity (Figure 8). However, the maximum-emission wavelength of **2** is red-shifted by 25 nm from 443 to 468 nm relative to that of the ligand, accompanied by a greatly decreased luminescent intensity. The red shift of emission maxima
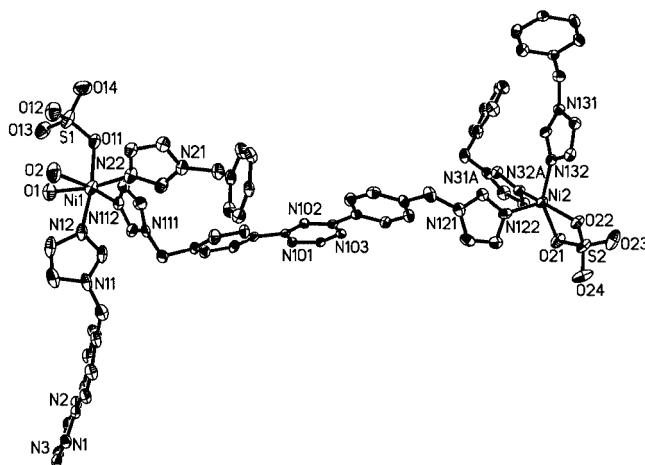
Table 2. Hydrogen-bond data for complexes **1** and **2**

D−H···A	Distance of D···A [Å]	Angles of D−H−A [°]
$[Zn_2(timpt)_2(\mu\text{-}OH)](NO_3)_3$ (**1**)		
O(1B)−H(1AB)···O(3)[a]	3.269(12)	154
C(2)−H(2)···O(2)[b]	3.212(8)	155
C(3)−H(3)···O(3)	3.398(11)	156
C(6)−H(6)···O(3)[c]	3.381(15)	147
$[Ni_2(timpt)_2(H_2O)_2(SO_4)_2]\cdot0.5H_2O$ (**2**)		
C(17)−H(5)···O(12)[d]	3.387(7)	158
C(17)−H(6)···O(2)[e]	3.373(7)	150
C(18)−H(7)···O(12)[e]	3.329(7)	150
C(27)−H(15)···O(14)[e]	3.307(7)	146
C(28)−H(16)···O(14)[e]	3.043(7)	141
C(33)−H(20)···O(23)[f]	3.339(8)	162
C(38)−H(25)···O(24)[f]	3.491(7)	172
C(115)−H(30)···O(14)[e]	3.343(7)	154
C(118)−H(34)···O(13)[e]	3.101(6)	142
C(127)−H(42)···O(22)[g]	3.463(8)	162
C(128)−H(43)···O(24)[h]	3.123(7)	154
C(133)−H(47)···O(22)[h]	3.412(6)	166
C(137)−H(50)···O(23)[h]	3.285(7)	139
C(138)−H(52)···O(23)[h]	3.027(6)	126
C(11D)−H(29A)···O(3)[i]	3.304(6)	130
C(11H)−H(32A)···O(3)[i]	3.106(7)	139

[a] Equivalent atoms generated by $x + 1/3$, $y + 5/3$, $z + 2/3$. [b] Equivalent atoms generated by $1/3 - x$, $5/3 - y$, $5/3 - z$. [c] Equivalent atoms generated by $2/3 + x - y$, $4/3 - y$, $1/6 - z$. [d] Equivalent atoms generated by $2 - x$, $2 - y$, $1 - z$. [e] Equivalent atoms generated by $-1 + x$, y, z. [f] Equivalent atoms generated by $1 + x$, $1 + y$, $1 + z$. [g] Equivalent atoms generated by $-1 - x$, $1 - y$, $-z$. [h] Equivalent atoms generated by $1 + x$, y, z. [i] Equivalent atoms generated by x, $y - 1$, z.

Figure 5. The coordination environment of Ni[II] in **2**; thermal ellipsoids are drawn at 30% probability; the carbon atoms are unlabelled and hydrogen atoms are omitted for clarity
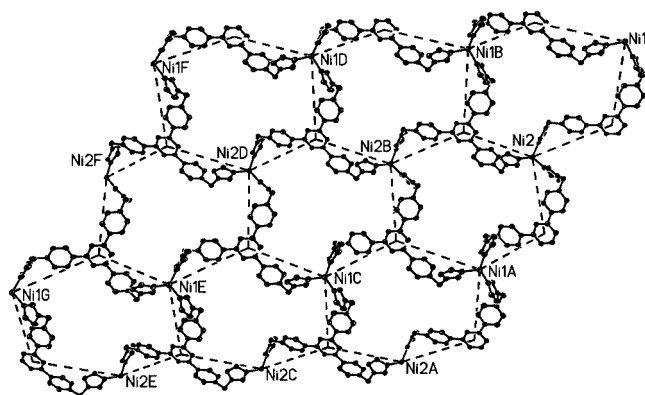
Figure 6. Perspective view of the 2D cationic sheet with the honeycomb structure (dashed lines) in the *bc* plane

Conclusion

The present study evidences that flexible tripodal ligands are efficient in constructing novel coordination networks with metal salts. The nanometer-sized flexible tripodal ligand timpt, using its imidazolyl N atoms, coordinates to metal atoms to form 2D coordination polymers with honeycomb network structures. In the zinc(II) complex **1**, each independent layer has two sublayers which are linked by hydroxy bridges through Zn−O coordination bonds. The emission maximum of timpt is red-shifted and the intensity is enhanced by coordination of timpt with zinc(II) nitrate. While in the case of the Ni[II] complex of timpt (**2**), the luminescent intensity was remarkably reduced compared with that of timpt. The results show that the metal ions have great influence on the structure and properties of the timpt complexes.

Figure 4. The $\pi-\pi$ interactions in **1** indicated by dashed lines

may be a result of the above-mentioned multiple $\pi-\pi$ interactions in **1** and **2**.[13] The results indicate that photoluminescent properties of timpt can be controlled by coordination of different metal salts.
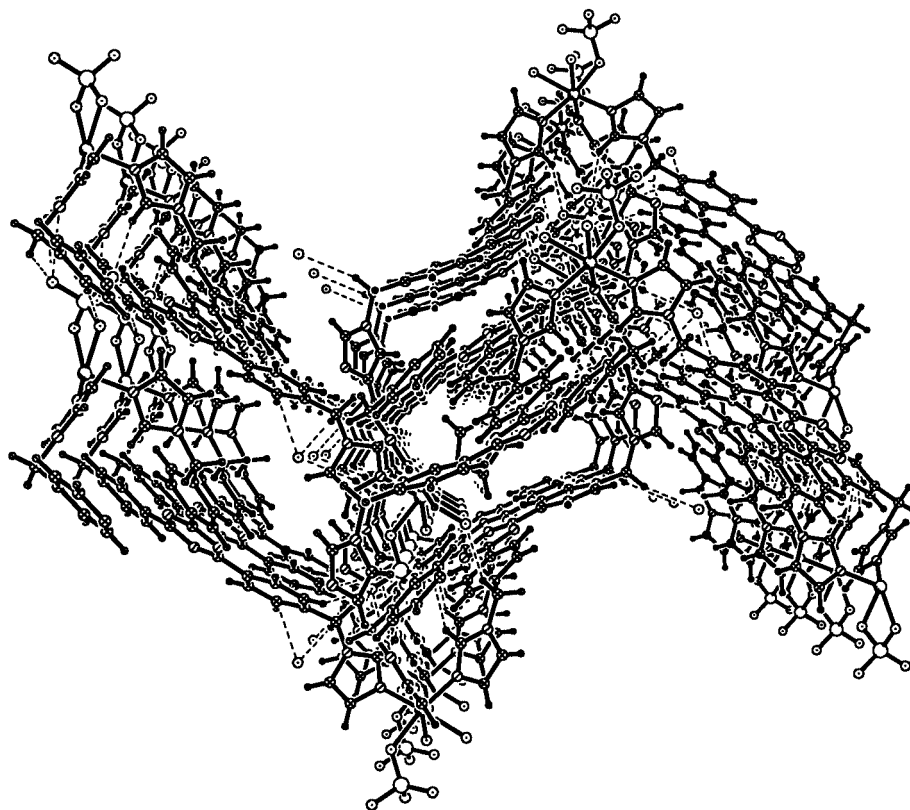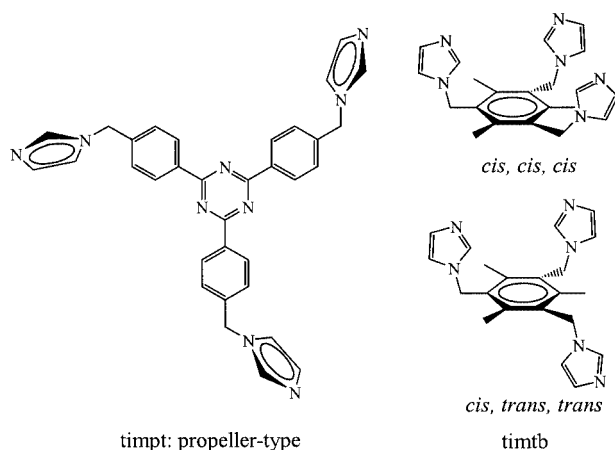
Figure 7. Crystal packing diagram of **2** with voids formed between two adjacent sheets and occupied by disordered water molecules; the hydrogen bonds are shown with dashed lines
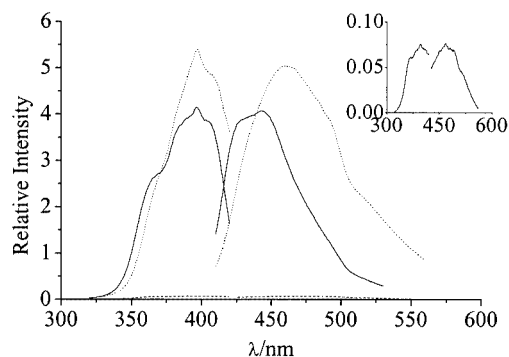

timpt: propeller-type

timtb

Scheme 1

Figure 8. Excited (left) and emission (right) spectra of timpt (solid lines), **1** (dotted lines), and **2** (dashed lines) in the solid state at room temperature; the enlarged spectral picture of **2** is at top right corner

Experimental Section

General: The timpt ligand was synthesized as described previously.[11] All the chemicals were of reagent grade and used as received without further purification. C, H, and N analyses were carried out with a Perkin−Elmer 240C elemental analyzer at the analysis center of Nanjing University. Luminescence spectra for the solid samples were recorded with a Hitachi 850 fluorescence spectrophotometer at room temperature (25 °C). TGA measurement was performed with a simultaneous SDT 2960 thermal analyzer under nitrogen with a heating rate of 10 °C·min^{-1}.

Synthesis of [Zn$_2$(timpt)$_2$(μ-OH)](NO$_3$)$_3$ (1): The complex was prepared by solvothermal reaction. The timpt ligand (61.9 mg, 0.11 mmol) was added to a water/ethanol (6:1, v/v) solution (14 mL) of Zn(NO$_3$)$_2$·6H$_2$O (46.0 mg, 0.15 mmol). After stirring at room temperature for ca. 15 min, the reaction mixture was transferred to an autoclave and kept at 150 °C for 3 d. Light yellow cubic crystals (50.2 mg) were obtained in 62% yield (based on ligand) after cooling the autoclave. C$_{66}$H$_{55}$N$_{21}$O$_{10}$Zn$_2$ (1433.05): calcd. C 55.32, H 3.87, N 20.52; found C 55.49, H 3.82, N 20.55.

Synthesis of [Ni$_2$(timpt)$_2$(H$_2$O)$_2$(SO$_4$)$_2$]·0.5H$_2$O (2): Complex **2** was synthesized in a similar manner to that of **1**. The timpt ligand (28.3 mg, 0.052 mmol) was added to a water/ethanol (6:1, v/v) solu-

tion (14 mL) of $NiSO_4 \cdot 7H_2O$ (15.7 mg, 0.056 mmol). After stirring at room temperature for ca. 15 min, the reaction mixture was placed in an autoclave and kept at 150 °C for 1 d. Green plate crystals (23.0 mg) were obtained in 61% yield (based on ligand) after cooling the autoclave. $C_{66}H_{59}N_{18}Ni_2O_{10.5}S_2$ (1453.81): calcd. C 54.53, H 4.09 N 17.34; found C 54.57, H 4.17, N 17.31. TGA data of **2** show that the weight loss of 3.06% between 10 and 90 °C corresponds to the loss of 2.5 water molecules per molecule of **2** (calcd. 3.10%). No further weight loss was observed from 90 to 310 °C.

X-ray Crystal Structure Analysis for Complexes 1 and 2: Data collection for **1** (Table 3) was performed with a Smart Apex CCD diffractometer at 293 K, using graphite-monochromated Mo-K_α radiation ($\lambda = 0.71073$ Å). The structure was solved by direct methods with SHELXTL and refined by full-matrix least-squares calculations. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were generated geometrically. The O1 atom of the hydroxy ion is disordered in three positions [O(1), O(1A), and O(1B)], each assigned 1/3 site occupancy. The O(2) and O(3) atoms of the nitrate anion are disordered in two positions, each with 1/2 site occupancy. The intensity data for complex **2** (Table 3) were collected with a Rigaku RAXIS-RAPID Imaging Plate diffractometer at 200 K, using graphite-monochromated Mo-K_α radiation ($\lambda = 0.71069$ Å). The structure was solved by direct method

with SIR92, and expanded with Fourier techniques. All data were refined anisotropically by full-matrix least-squares calculations for non-hydrogen atoms. The hydrogen atoms were generated geometrically except those of water molecules. CCDC-201837 (**1**) and -201838 (**2**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (internat.) + 44-1223/336-033; E-mail: deposit@ccdc.cam.ac.uk].

Table 3. Crystallographic data for complexes **1** and **2**

Compound	**1**	**2**
Empirical formula	$C_{66}H_{55}N_{21}O_{10}Zn_2$	$C_{66}H_{59}N_{18}Ni_2O_{10.5}S_2$
Formula mass	1433.05	1453.81
Crystal system	rhombohedral	triclinic
Space group	$R\bar{3}c$	$P\bar{1}$
a [Å]	17.573(1)	8.3122(2)
b [Å]	17.573(1)	16.4661(5)
c [Å]	35.410(3)	23.2800(7)
α [°]	90	90.7639(4)
β [°]	90	90.7241(6)
γ [°]	90	94.6208(5)
V [Å^3]	9470.0(11)	3175.38(16)
Z	6	2
D_c [g·cm^{-3}]	1.508	1.530
μ [cm^{-1}]	0.839	0.738
Crystal habit, color	colorless block	green platelet
Crystal size [mm]	0.40×0.25×0.12	0.30×0.15×0.10
$2\theta_{max}$ [°]	50	55
Index ranges	$-17 \leq h \leq 0$	$-10 \leq h \leq 10$
	$0 \leq k \leq 20$	$-21 \leq k \leq 21$
	$0 \leq l \leq 40$	$-30 \leq l \leq 30$
Reflns. collected	14831	28186
Independent reflns.	1848	13964
R_{int}	0.0209	0.0608
Obsd. reflns.[$I > 2\sigma(I)$]	1089	6616
Parameters refined	149	893
R (obsd. data)	0.0484	0.0706
wR (obsd. data)	0.1397[a]	0.1639[b]
Goodness of fit	0.972	0.930
Residuals [e·Å^{-3}]	0.861; −0.529	1.045; −0.593

[a] $\omega = 1/[\sigma^2(F_o^2) + (0.0800P)^2 + 1.9900P]$, where $P = (F_o^2 + 2F_c^2)/3$. [b] $\omega = 1/[\sigma^2(F_o^2) + (0.1000P)^2]$, where $P = (F_o^2 + 2F_c^2)/3$.

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