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Two-dimensional and three-dimensional nickel(II) supramolecular complexes based on the new chelating ligand *N*-(4-carboxyphenyl)iminodiacetic acid: hydrothermal synthesis and crystal structures

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Abstract

Two new nickel (II) supramolecular complexes, [Ni(HCPIDA)(H_2O)$_3$] (**1**) and [Ni_2(HCPIDA)$_2$(bpy)(H_2O)$_4$]·(H_2O)$_2$ (**2**) (H_3CPIDA = *N*-(4-carboxyphenyl) iminodiacetic acid, bpy = 4,4′-bipyridine) were synthesized through hydrothermal method. These supramolecular complexes have been structurally characterized by X-ray single crystal diffraction. The structure of **1** exhibits a new three-dimensional supramolecular framework with one-dimensional channels, formed by hydrogen bonding interactions. The structure of **2** shows a new two-dimensional joint-ladder like architecture constructed by hydrogen bonding and π–π staking interactions. The compounds **1** and **2** contain the zigzag-shape and ladder-shape supramolecular chains, respectively, which further extend into the three-dimensional and two-dimensional supramolecular network, respectively.

Keywords: Hydrothermal synthesis; Crystal structure; Supramolecular network; Nickel(II) complex; N-(4-carboxyphenyl)iminodiacetic acid

1. Introduction

The rational design and synthesis of metal-directed supramolecular framework have received much attention in coordination chemistry because of their interesting molecular topologies and potential applications in catalysis, non-linear optics, sensors, magnetism and molecular recognition [1–5]. During the last decade, many high-dimensional coordination complexes have been designed and prepared through molecular self-assembly process [6–11]. The construction of metal-organic supramolecular framework, so far, can be achieved via three kinds of interactions, i.e. coordinate covalent bonds, hydrogen bonds and π–π staking interactions. It should be noted that weaker intermolecular forces (hydrogen bond and π–π stacking) play an important role in the formation of these high dimensional frameworks [12–20]. Doubtless, hydrogen bond provides an ideal organizing force for supramolecular networks, because its moderately directional intermolecular interaction can effectively control short-range packing. In the construction of new supramolecular networks based on metal-containing complex, the polycarboxylate ligands are of special interest [21–24], because they can be regarded not only as hydrogen-bonding accepters but also as hydrogen-bonding donors, depending upon the number of deprotonated carboxylate groups.

In addition, recent practice has proved that hydrothermal synthesis is an effective method for the construction of new supramolecular complexes [18]. Although most of these insoluble solids have been synthesized by controlled mixing of suitable soluble molecular components, hydrothermal conditions have demonstrated increasing success in providing alternative pathways to the preparation of crystalline

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supramolecular solids. Hydrothermal synthesis carried out in superheated solvent systems provides ideal conditions for the crystal growth owing to the enhanced transport ability of the solvents [25].

We focused on the synthesis of the polycarboxylate ligands and prepared a new chelating ligand *N*-(4-carboxyphenyl)iminodiacetic acid (H_3CPIDA). In this paper, we report the two new supramolecular complexes, $[Ni(HCPIDA)(H_2O)_3]$ (**1**) and $[Ni_2(HCPIDA)_2(bpy)(H_2O)_4]\cdot(H_2O)_2$ (**2**). The compound **1** is a new three-dimensional supramolecular network with one-dimensional channels. The compound **2** exhibits a new two-dimensional joint-ladder like supramolecular network.

2. Experimental section

2.1. Materials and methods

All chemicals purchased were of reagent grade and used without further purification. FTIR spectra were recorded in the range 400–4000 cm^{-1} on a Bruker VECTOR-22 FT-IR spectrophotometer using KBr pellets. ^1H NMR spectrum was measured on a Avance AV400 NMR spectrometer at room temperature. TG analysis were performed under air with a heating rate of 10 °C min^{-1} using a Shimadzu TGA-50H TG analyzer.

2.2. Synthesis

2.2.1. Preparation of N-(4-carboxyphenyl)iminodiacetic acid (H_3CPIDA)

A solution of KOH (33.6 g, 0.6 mol) in water (100 ml) was added drop-wise to a solution of monochloroacetic acid (28.4 g, 0.3 mol) in water (100 ml). To the resulting alkaline solution, *p*-aminobenzoic acid (13.7 g, 0.1 mol) was slowly added and the mixture was refluxed 30 h at 86 °C. Then the reaction mixture was cooled to room temperature and acidified with HCl (6 mol/l) until the desired white acid precipitated (pH ∼2.5), which were collected by filtration, washed with water and recrystallized in water (Scheme S1). Yield: 32% based on *p*-aminobenzoic acid. FT-IR (KBr pellet, cm^{-1}): 3458b, 2919m, 1714m, 1612s, 1530m, 1462m, 1388s, 1278m, 1239m, 1192s, 975w, 772s, 693m, 549w. ^1H NMR (400 MHz, D_2O, 25 °C): 7.84 (d, 2 H, *m*), 6.58 (d, 2 H, *o*), 4.23 (s, 4 H, CH_2–COO).

2.2.2. Hydrothermal synthesis of $[Ni(HCPIDA)(H_2O)_3]$ (1) and $[Ni_2(HCPIDA)_2(bpy)(H_2O)_4]\cdot(H_2O)_2$ (2)

Complexes **1** and **2** were synthesized hydrothermally in the heavy-walled Pyrex tubes under autogenous pressure, which contain $Ni(NO_3)_2\cdot 6H_2O$, H_3CPIDA, KOH and H_2O in a molar ratio of 2:2:1:556 for $[Ni(HCPIDA)(H_2O)_3]$ **1**; $Ni(NO_3)_2\cdot 6H_2O$, H_3CPIDA, 4,4′-bpy·$2H_2O$, KOH, ethanol and H_2O in a molar ratio of 2:2:2:1:78:222 for $[Ni_2(HCPIDA)_2(bpy)(H_2O)_4]\cdot(H_2O)_2$ **2**. The tubes were frozen in liquid N_2, sealed under vacuum, and placed inside an oven at 120 °C for 2 days (Scheme S1). *Caution: the sealed tube is potentially explosive and the volume of solvents should be controlled*. The green crystals of **1** and **2** were collected and washed with ethanol. Yield: 65% for **1** and 21% for **2** based on Ni. FT-IR (KBr pellets, cm^{-1}) for **1**: 3444b, 3003w, 2923m, 1694s, 1580vs, 1451m, 1411vs, 1355w, 1325m, 1251s, 1196s, 1128m, 991w, 920w, 853m, 771w, 700m, 638w, 544w; for **2**: 3392b, 3238b, 2934w, 1707s, 1591vs, 1515w, 1488w, 1405s, 1323m, 1289s, 1219m, 1197s, 1146m, 1122m, 1046w, 974w, 914w, 806m, 772m, 731w, 696w, 641m.

2.3. X-ray crystallographic studies of 1 and 2

Single-crystal X-ray diffraction measurements of complexes **1** and **2** were carried out with a Bruker Smart CCD diffractometer at 293(2) K. The determination of unit cell parameters and the data collections were performed with Mo Kα radiation ($\lambda = 0.71073$ Å). The structures were solved by direct methods using SHELXS-97 [26] and refined by full-matrix least-squares methods against F^2 (SHELXL-97) [27]. All non-hydrogen atoms were refined anisotropically. The C–H hydrogen atoms were added theoretically and riding on the concerned atoms. The O–H hydrogen atoms were located in successive difference Fourier syntheses. Crystallographic data and experimental details for structural analysis are summarized in Table 1. Selected bond lengths and angles for **1** and **2** are listed in Table 2.

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

Compound	1	2
Formula	$C_{11}H_{15}NiNO_9$	$C_{32}H_{30}Ni_2N_4O_{18}$
M_r	363.95	876.02
Crystal system	Orthorhombic	Monoclinic
Space group	*Aba2*	$P2_1/n$
Unit cell dimensions		
a (Å)	7.0064(18)	7.4820(15)
b (Å)	30.208(8)	8.7950(18)
c (Å)	12.604(3)	26.382(5)
β (°)	90.00	96.11(3)
V (Å^3)	2667.8(12)	1726.2(6)
Z	8	2
T (K)	293(2)	293(2)
D_{calc} (g cm^{-3})	1.812	1.685
μ (mm^{-1})	1.505	1.181
F(000)	1504	900
Reflections collected	3041	6626
Independent reflections	1972	2275
R_{int}	0.0614	0.0563
Observed reflections	1414	2046
Final R_1, wR_2 [$I > 2\sigma(I)$]	0.0523, 0.1194	0.0911, 0.1729
Flack parameter	−0.03 (4)	

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

1		2	
Ni–O(1)	2.019 (7)	Ni(1)–O(1)	2.030 (6)
Ni–O(3)	2.043 (6)	Ni(1)–O(3)	2.030 (6)
Ni–O(7)	2.054 (8)	Ni(1)–O(7)	2.080 (8)
Ni–O(8)	2.087 (8)	Ni(1)–O(8)	2.037 (7)
Ni–O(9)	2.049 (7)	Ni(1)–N(1)	2.092 (8)
Ni–N	2.197 (8)	Ni(1)–N(2)	2.196 (7)
O(1)–Ni–O(3)	92.2 (3)	O(1)–Ni(1)–O(3)	161.6 (2)
O(1)–Ni–O(7)	175.5 (3)	O(1)–Ni(1)–O(7)	86.9 (3)
O(1)–Ni–O(8)	87.8 (3)	O(1)–Ni(1)–O(8)	97.3 (3)
O(1)–Ni–O(9)	94.8 (3)	O(3)–Ni(1)–O(7)	87.4 (3)
O(3)–Ni–O(7)	91.5 (3)	O(3)–Ni(1)–O(8)	99.7 (3)
O(3)–Ni–O(8)	179.1 (3)	O(7)–Ni(1)–O(8)	86.0 (3)
O(3)–Ni–O(9)	95.1 (3)	O(1)–Ni(1)–N(1)	92.3 (3)
O(7)–Ni–O(8)	88.5 (3)	O(1)–Ni(1)–N(2)	81.2 (2)
O(7)–Ni–O(9)	87.6 (3)	O(3)–Ni(1)–N(1)	95.2 (3)
O(8)–Ni–O(9)	85.8 (3)	O(3)–Ni(1)–N(2)	81.2 (2)
O(1)–Ni–N	82.0 (3)	O(7)–Ni(1)–N(1)	173.6 (3)
O(3)–Ni–N	78.1 (3)	O(7)–Ni(1)–N(2)	88.6 (3)
O(7)–Ni–N	96.1 (3)	O(8)–Ni(1)–N(1)	87.8 (3)
O(8)–Ni–N	101.0 (3)	O(8)–Ni(1)–N(2)	174.5 (3)
O(9)–Ni–N	172.3 (3)	N(1)–Ni(1)–N(2)	97.5 (3)

3. Results and discussion

3.1. Crystal structure of [Ni(HCPIDA)(H_2O)$_3$] (*1*)

The single-crystal X-ray structural analysis shows that the structure of compound **1** is a new three-dimensional supramolecular framework in which the asymmetric unit contains one Ni atom, one HCPIDA^{2-} ligand and three coordinated water molecules. The nickel (II) atom is coordinated by two oxygen atoms (Ni–O(1) = 2.019(7) Å, Ni–O(3) = 2.043(6) Å) and one nitrogen atom (Ni–N = 2.197(8) Å) of one HCPIDA^{2-} ligand, and three coordinated water molecules (Ni–O(7) = 2.054(8) Å, Ni–O(8) = 2.087(8) Å, Ni–O(9) = 2.049(7) Å), showing a lightly distorted octahedral geometry (Fig. 1). As can be seen from the above description, a HCPIDA^{2-} ligand acts as a tridentate ligand chelating a nickel atom. The positions of oxygen atoms (O(1), O(3)) define the *cis* conformation of the HCPIDA^{2-} ligand. The bond angles around the Ni atom at the equatorial plane defined by O(1), O(3), O(7) and O(8) sum to 360° within experimental error, showing that they are coplanar. The bond lengths of Ni–N and Ni–O are comparable with those of other nickel(II) complexes [18,28].

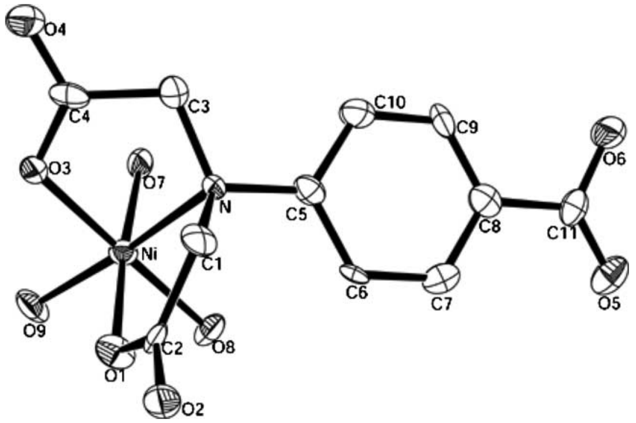

Fig. 1. ORTEP diagram of the coordination environment of the nickel atom in **1**; thermal ellipsoids are drawn at the 50% probability level; all hydrogen atoms are omitted for clarity.

It is interesting that the basic building units ([Ni(HCPIDA)(H_2O)$_3$]) are connected into a one-dimensional zigzag chain running parallel to the *a* direction via extensive hydrogen bonds formed between the coordinated water molecules and carboxylate oxygen atoms (See Fig. 2 and Fig. S1). The typical hydrogen bonds are O(7)···O(4) of 2.673 Å and O(9)···O(3) of 2.784 Å. It is notable that the phenylcarboxylic groups of HCPIDA^{2-} ligands are alternatively extended outwards at both sides of the chain (Fig. S1). Each pair of adjacent phenylcarboxylic groups at the same side forms one pitch of the chain, which is virtually oriented in a parallel fashion with a separation of 7.01 Å (equal to the length of *a* axis). The zigzag-shape chains are further linked into a two-dimensional supramolecular network in the *ab* plane through hydrogen bonding interactions as shown in Fig. 2. The representative hydrogen bonds are O(8)···O(5) (both 2.846 Å). It is interesting that all lateral phenylcarboxylic groups from neighboring chains are intercalated in a zipper-like fashion, as shown in Fig. 2. There are offset π–π staking interactions between the intercalated phenylcarboxylic groups with the centroid separation of 4.06 Å. In addition, the two-dimensional layers further assemble into three-dimensional framework through hydrogen bonding interactions with O(2)···O(6) of 2.653 Å, O(2)···O(7) of 2.879 Å and O(4)···O(9) of 2.783 Å (Fig. S2). The most interesting feature of compound **1** is that its three-dimensional supramolecular framework consists of one-dimensional channels along [100] direction (Fig. 3).

3.2. Crystal structure of [Ni_2(HCPIDA)$_2$(bpy)(H_2O)$_4$]·(H_2O)$_2$ (*2*)

The single-crystal X-ray structural analysis exhibits that the structure of compound **2** is a unique two-dimensional supramolecular network in which the asymmetric unit contains 1 nickel atom, 1 HCPIDA^{2-} ligand, 0.5 4,4′-bpy ligand, 2 coordinated water molecules and 1 uncoordinated water molecule. The nickel (II) atom is coordinated by two oxygen atoms (Ni(1)–O(1) = 2.030(6) Å, Ni(1)–O(3) = 2.030(6) Å) of HCPIDA^{2-} ligand, two nitrogen atoms (Ni(1)–N(1) = 2.092(8) Å, Ni(1)–N(2) = 2.196(7) Å) of one 4,4′-bipy ligand and one HCPIDA^{2-} ligand, respectively, and two coordinated water molecules (Ni(1)–O(7) = 2.080(8) Å, Ni(1)–O(8) = 2.037(7) Å), showing a lightly distorted octahedral geometry in which two *cis* conformational oxygen atoms (O(1),O(3)) of HCPIDA^{2-} ligand act

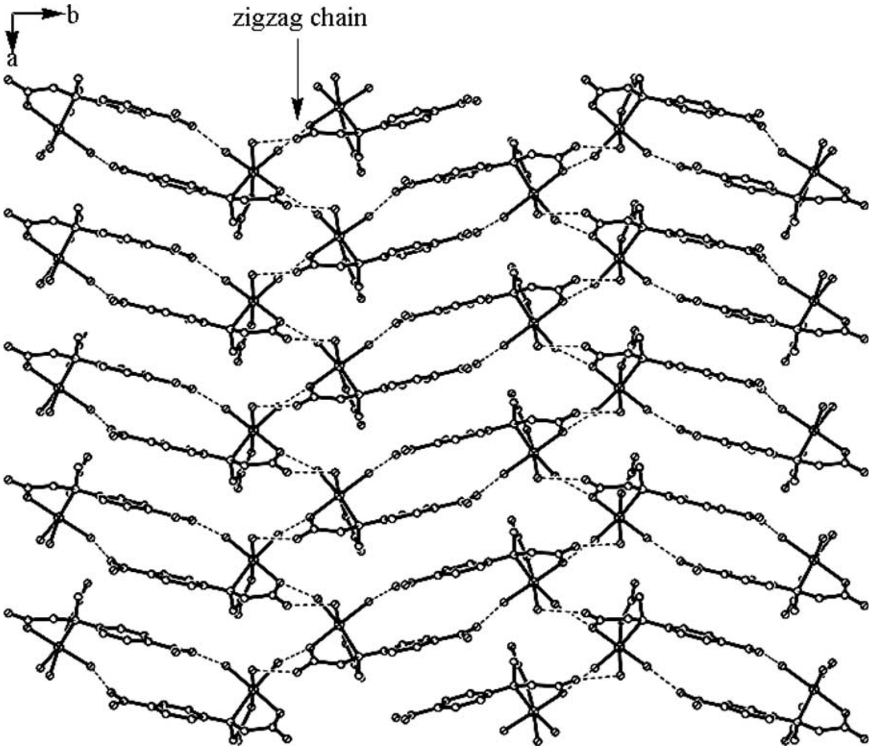

Fig. 2. Two-dimensional supramolecular network in **1** as viewed down the *c* axis, showing the double hydrogen-bond-linked zigzag-shape chains, the dotted lines represent hydrogen bonds.

as the apices (Fig. 4). In the asymmetric unit, the $HCPIDA^{2-}$ ligand also acts as a tridentate ligand chelating a nickel atom. Two asymmetric units then form a Z-shape dimmer which contains two Ni atoms, two $HCPIDA^{2-}$ ligands, one 4,4′-bpy ligand and four coordinated water molecules. In the Z-shape dimmer unit, two phenylcarboxylic groups are located above and below the 4,4′-bpy ring, respectively, which exhibit strong intramolecular π–π staking interactions between 4,4′-bpy ring and $HCPIDA^{2-}$ aromatic rings with centroid··· centroid of 3.41 Å. In addition, these aromatic π–π stakes show offset or slipped packing rather than perfect face-to-face array of atoms [29].

A notable feature of the title compound **2** resides in the formation of a new two-dimensional joint-ladder like supramolecular network. As shown in Fig. 5, the coordinated water molecules (O(8)) link the oxygen atoms (O(2)) of the carboxylate groups through double intermolecular hydrogen bonds (O(8)···O(2) = 2.676 Å), giving a one-dimensional ladder-shape structure along *a* direction (the neighboring Ni···Ni separation of 7.482 Å), as shown in Fig. S3. On the other hand, in the ladder-shape chain, there are intermolecular aromatic π–π staking interactions between slipped $HCPIDA^{2-}$ aromatic rings with centroid···centroid of 3.96 Å. Furthermore, the coordinated water molecules (O(7)) in one ladder link the oxygen atoms of the carboxylate groups in the neighboring ladder through hydrogen bonds (O(7)···O(1) = 2.691 Å, O(7)···O(4) = 2.712 Å) to form the unique two-dimensional joint-ladder like architecture (Fig. 5) [19], in which the Ni···Ni separation of two adjacent ladders is 5.785 Å. It should be noted that the uncoordinated water molecule (O(9)) links the Z-shape $[Ni_2(HCPIDA)_2(bpy)(H_2O)_4]$ units through multiple hydrogen bonds (O(9)···O(4) = 2.695 Å, O(9)···O(6) = 2.734 Å, O(9)···O(8) = 2.790 Å), which further stabilize this two-dimensional supramolecular network.

3.3. IR spectroscopy

The IR spectrum of compound **1** shows characteristic bands of carboxylate group at 1580 cm^{-1} for the antisymmetric stretching and at 1411 cm^{-1} for symmetric stretching. The separation (Δ) between ν_{asym} (CO_2) and ν_{sym} (CO_2) indicates the presence of monodentate (169 cm^{-1}) coordination mode in compound **1** [30], which is consistent with the crystal structure of **1**. The presence of the characteristic band at 1694 cm^{-1} in compound **1** indicates that the deprotonation of H_3CPIDA ligand is incomplete. The absorption peak at 3444 cm^{-1} is assigned to ν_{O-H} of the coordinated water molecules. The IR spectrum of compound **2** exhibits that characteristic bands of carboxylate group at 1591 cm^{-1} for the antisymmetric stretching and at 1405 cm^{-1} for symmetric stretching. The separation (Δ) between ν_{asym} (CO_2) and ν_{sym} (CO_2) indicates the presence of monodentate (186 cm^{-1}) coordination mode in compound **2**. The deprotonation of H_3CPIDA ligand in compound **2** is also incomplete, because there exists

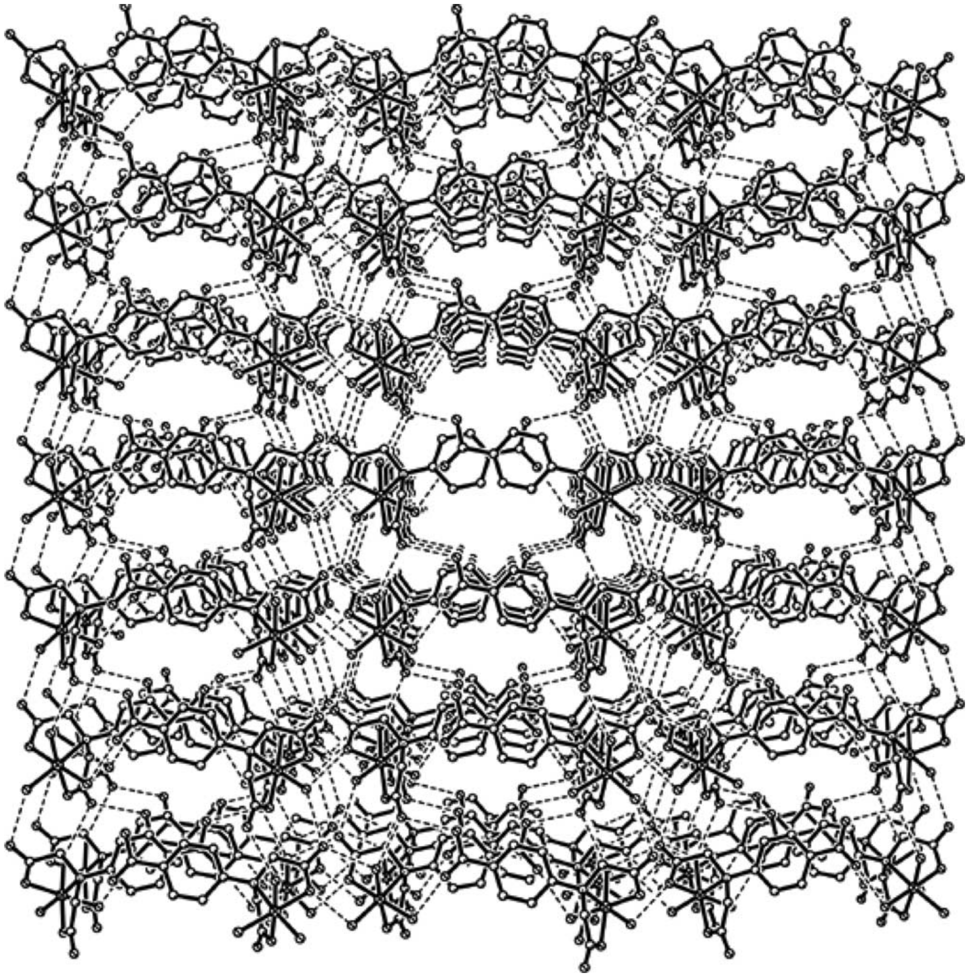

Fig. 3. View of the three-dimensional supramolecular network of **1** along *a* axis, showing one-dimensional channels.

the characteristic band at 1707 cm^{-1}. The two absorption peaks at 3392 and 3238 cm^{-1} are assigned to $\nu_{\mathrm{O-H}}$ of the coordinated water molecules and uncoordinated water molecules.

3.4. Thermal analysis

The TG curve of compound **1** exhibits the continuous weight loss stages in the temperature range 185–450 °C (Fig. S4a), corresponding to the release of the coordinated water molecules and HCPIDA^{2-} ligand. The remaining weight of 19.75% corresponds to the percentage (20.52%) of Ni and O components, indicating that the final product is NiO. The whole weight loss (80.25%) is in agreement with the calculated value (79.48%). Compound **2** also shows the continuous weight loss stages in the temperature range 142–505 °C (Fig. S4b), corresponding to the release of the uncoordinated water molecules, coordinated water molecules, and 4,4′-bpy and HCPIDA^{2-} ligands. The remaining weight of 8.77% corresponds to the percentage (8.53%) of Ni and O components, indicating that the final product is NiO.

4. Conclusions

In this paper, two new two-dimensional and three-dimensional supramolecular networks, constructed through

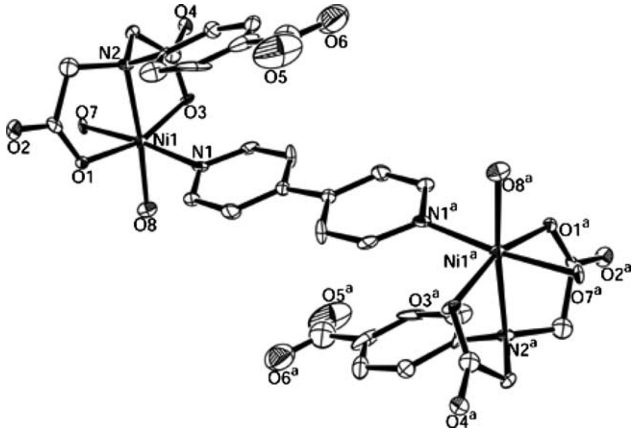

Fig. 4. Perspective view of the coordination environment of the nickel atoms in compound **2**, showing Z-shape dimmer; thermal ellipsoids are drawn at the 50% probability level; all hydrogen atoms and uncoordinated water molecules are omitted for clarity; Symmetry code: $a = -x, -y, -z$.

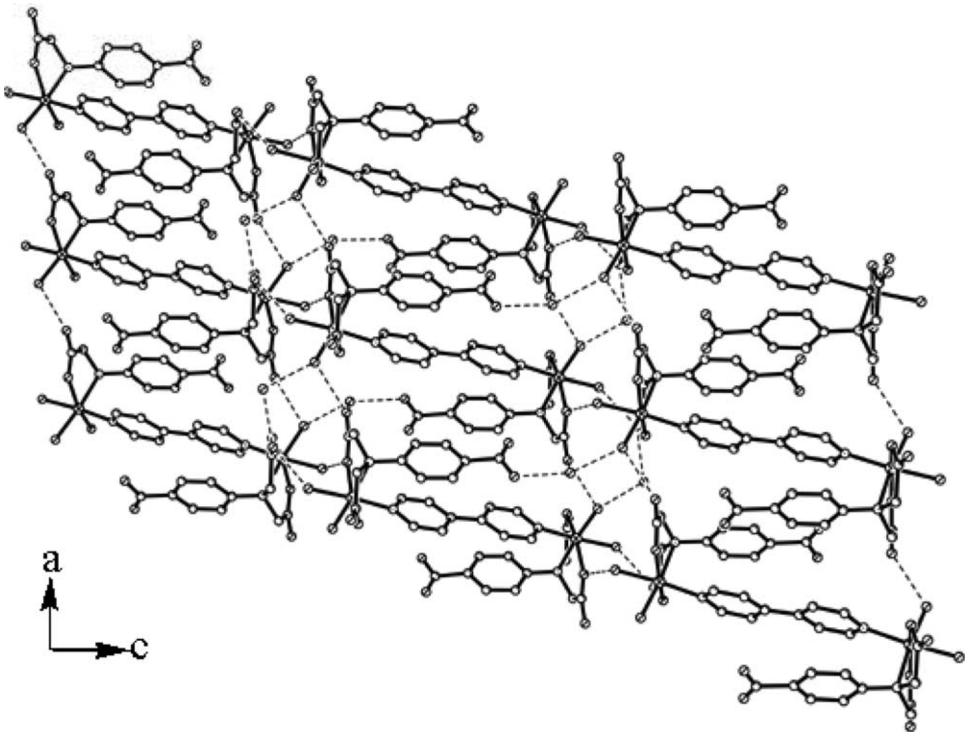

Fig. 5. View of two-dimensional joint-ladder like supramolecular network of **2** down the *b* axis, showing the double hydrogen-bond-linked ladder-shape chains, the dotted lines represent hydrogen bonds.

hydrogen bonding and π–π stacking interactions, have been synthesized and structurally characterized. The successful synthesis of the new compounds **1** and **2** not only proves that the strong capability of hydrothermal reaction in preparing new supramolecular complexes but also shows that weaker intermolecular interactions (hydrogen bonding and π–π stacking interactions) contribute to the formation of higher-dimensional supramolecular network.

5. Supplementary materials

Crystallographic data for the crystal structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre (CCDC nos 223189 for *1* and 227501 for **2**). These materials can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk).

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Appendix. Supplementary material

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molstruc.2004.07.024

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