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Journal of Coordination Chemistry

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713455674>

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Online publication date: 21 July 2010

To cite this Article Han, Sol , Lough, Alan J. and Chang Kim, Ju(2010) 'Synthesis and crystal structures of nickel(II) supramolecules containing hexaazamacrocycles and aromatic carboxylate ligands', *Journal of Coordination Chemistry*, 63: 12, 2069 – 2078

To link to this Article: DOI: 10.1080/00958972.2010.499456

URL: <http://dx.doi.org/10.1080/00958972.2010.499456>

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Synthesis and crystal structures of nickel(II) supramolecules containing hexaazamacrocycles and aromatic carboxylate ligands

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(Received 1 October 2009; in final form 22 April 2010)

Two new nickel(II) complexes, $\{[\text{Ni}(\text{L})(4,4'\text{-bpdc})] \cdot 3\text{H}_2\text{O}\}_n$ (**1**) and $\{[\text{Ni}(\text{L})(2,6\text{-ndc})] \cdot 2\text{CH}_3\text{CN}\}_n$ (**2**) (**L** = 1,8-dihydroxyethyl-1,3,6,8,10,13-hexaazacyclotetradecane, 4,4'-bpdc = 4,4'-biphenyldicarboxylate, 2,6-ndc = 2,6-naphthalenedicarboxylate), have been synthesized and structurally characterized by spectroscopic and X-ray diffraction methods. Compound **1** shows a 3-D supramolecule which is composed of two different series of 1-D coordination polymers, where each 1-D chain runs in different directions and interacts by π - π stacking at the intersection. Compound **2** contains 1-D coordination polymers in which 1-D chains run in the same direction. The 1-D chains are interconnected by hydrogen bonds in an undulated fashion to form a 3-D supramolecule.

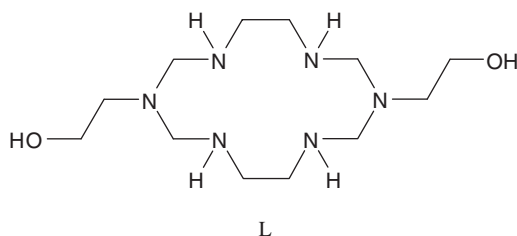
Keywords: Nickel(II) complex; Hexaazamacrocycle; Crystal structure; Carboxylate ligands

1. Introduction

Supramolecules constructed from the self-assembly of coordination polymers composed of metal ion building blocks and organic molecules are of great importance due to the potential applications in molecular devices, catalysis, molecular recognition, protein structure stabilization, etc. [1–6]. In the assembly of supramolecules, noncovalent interactions, such as hydrogen bonding, π - π stacking, and C–H \cdots π interaction in addition to metal–ligand coordination are usually involved [7–10]. Coordination polymers built from metallomacrocycles and bridging aromatic carboxylates can be utilized to induce metallosupramolecules by self-assembly processes [11–13]. In particular, the coordination polymers composed of the present metallohexaazamacrocycle and aromatic carboxylate bridging ligands are advantageous in constructing metallosupramolecules because of the presence of hydroxyl groups and aromatic carboxylate ligands, which can act as hydrogen bond donors and/or acceptors or π - π interaction for the formation of metallosupramolecules during self-assembly [10–12].

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By using hexaazamacrocyclic and aromatic carboxylates, we have synthesized and structurally characterized two 3-D supramolecules **1** and **2**. In both complexes, various types of hydrogen bonding as well as π - π (in **1**) interactions play an important role in building 3-D supramolecules. The details of structures and properties of **1** and **2** are discussed in this report.



2. Experimental

2.1. Physical measurements

All chemicals utilized in this investigation were obtained from commercial sources of reagent grade and were used without purification. Distilled water was used for all procedures. Infrared (IR) spectra of solid samples were recorded on a Perkin-Elmer Paragon 1000 FT-IR spectrophotometer between 4000 and 400 cm^{-1} as Nujol mulls on KBr discs. Solid-state electronic spectra were measured by the diffuse reflectance method using **1** and **2**, diluted with BaSO_4 on a Shimadzu 2401 PC UV-Vis recording spectrophotometer. Elemental analyses were performed at the analytical laboratory of Korea Research Institute of Chemical Technology, Daejeon, Korea.

Caution! Perchlorate salts are potentially explosive and should be handled in small quantities.

2.2. Synthesis of $[\text{Ni}(\text{L})](\text{ClO}_4)_2$

The precursor complex $[\text{Ni}(\text{L})](\text{ClO}_4)_2$ was prepared by a template method according to the literature procedures [14].

2.3. Syntheses of $\{[\text{Ni}(\text{L})(4,4'\text{-bpd})] \cdot 3\text{H}_2\text{O}\}_n$ (**1**)

To a DMF (5 mL) solution of $[\text{Ni}(\text{L})](\text{ClO}_4)_2$ (137 mg, 0.25 mmol) was added a stoichiometric amount of disodium 4,4'-bpd (70 mg, 0.25 mmol) dissolved in water (5 mL), which was allowed to stand in an open beaker at ambient temperature. After 1 week, pale pink blocks of **1** were obtained. Suitable crystals of **1** were picked under a microscope, while they were wet for X-ray diffraction studies and subsequent spectroscopic measurements. Yield: 90%. Anal. Calcd (%) for $\text{C}_{26}\text{H}_{44}\text{N}_6\text{O}_9\text{Ni}$ (**1**) $\cdot\text{H}_2\text{O}$: C, 47.1; H, 7.0; N, 12.7. Found (%): C, 46.9; H, 6.9; N, 12.7. IR (Nujol, cm^{-1}): 3380 (νOH), 3195 (νNH), 1583 ($\nu_{\text{as}}\text{COO}$), 1273 ($\nu_{\text{s}}\text{COO}$).

2.4. Syntheses of $\{[Ni(L)(2,6-ndc)] \cdot 2MeCN\}_n$ (**2**)

Complex **2** was prepared by adding an aqueous solution of dipotassium 2,6-ndc (73 mg, 0.25 mmol) to a CH₃CN/H₂O (1:1, 10 mL) solution of [Ni(L)](ClO₄)₂ (137 mg, 0.25 mmol). Slow evaporation of the solution provided pale pink needles of **2**. Suitable crystals for X-ray diffraction studies and other measurements were manually collected under a microscope. Yield: 90%. Microanalytical measurement for **2'** was determined by using thoroughly dried **2**. Anal. Calcd (%) for C₂₄H₃₆N₆O₆Ni (**2'**): C, 51.1; H, 6.4; N, 14.9. Found (%): C, 51.0; H, 6.4; N, 14.9. IR (Nujol, cm⁻¹): 3435 (νOH), 3197 (νNH), 1579 (ν_{as}COO), 1292 (ν_sCOO), 2258 (νC≡N).

2.5. X-ray crystallography

A summary of selected crystallographic data for **1** and **2** is given in table 1. X-ray data were collected on a Nonius Kappa CCD diffractometer using graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å). A combination of 1° φ and ω (with κ offsets) scans were used to collect sufficient data. The data frames were integrated and scaled using

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

	1	2
Empirical formula	C ₂₆ H ₄₄ NiN ₆ O ₉	C ₂₈ H ₄₂ NiN ₈ O ₆
Formula weight	643.38	645.41
Temperature (K)	150(1)	150(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic
Space group	C2/c	P2 ₁ /c
Unit cell dimensions (Å, °)		
<i>a</i>	19.8112(12)	11.5124(5)
<i>b</i>	17.5106(9)	15.6636(10)
<i>c</i>	9.3803(6)	8.5530(7)
β	96.663(3)	93.006(3)
<i>V</i> (Å ³)	3232.1(3)	1540.20(17)
<i>Z</i>	4	2
<i>D</i> _{calcd} (mg m ⁻³)	1.322	1.392
Absorption coefficient (mm ⁻¹)	0.657	0.684
<i>F</i> (000)	1368	684
Crystal size (mm ³)	0.34 × 0.21 × 0.20	0.16 × 0.08 × 0.06
θ range for data collection	2.58–27.47°	2.60–27.44°
Index ranges	–25 ≤ <i>h</i> ≤ 25 0 ≤ <i>k</i> ≤ 22 0 ≤ <i>l</i> ≤ 12	–10 ≤ <i>h</i> ≤ 14 –20 ≤ <i>k</i> ≤ 18 –11 ≤ <i>l</i> ≤ 11
Reflections collected	3657	8380
Independent reflections	3657 [<i>R</i> (int) = 0.0000]	3470 [<i>R</i> (int) = 0.0751]
Completeness to θ	98.7% (θ = 27.47°)	99.0% (θ = 27.44°)
Absorption correction	Semi-empirical from equiv	Semi-empirical from equiv
Max. and min. transmission	0.881 and 0.823	0.964 and 0.863
Refinement method	Full-matrix least-squares on <i>F</i> ²	Full-matrix least-squares on <i>F</i> ²
Data/restraints/parameters	3657/0/196	3470/0/201
Goodness-of-fit on <i>F</i> ²	1.007	0.979
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> ₁ = 0.0535, <i>wR</i> ₂ = 0.1326	<i>R</i> ₁ = 0.0587, <i>wR</i> ₂ = 0.1237
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0931, <i>wR</i> ₂ = 0.1481	<i>R</i> ₁ = 0.1435, <i>wR</i> ₂ = 0.1583
Largest difference peak and hole (e Å ⁻³)	0.591 and –0.344	0.522 and –0.569

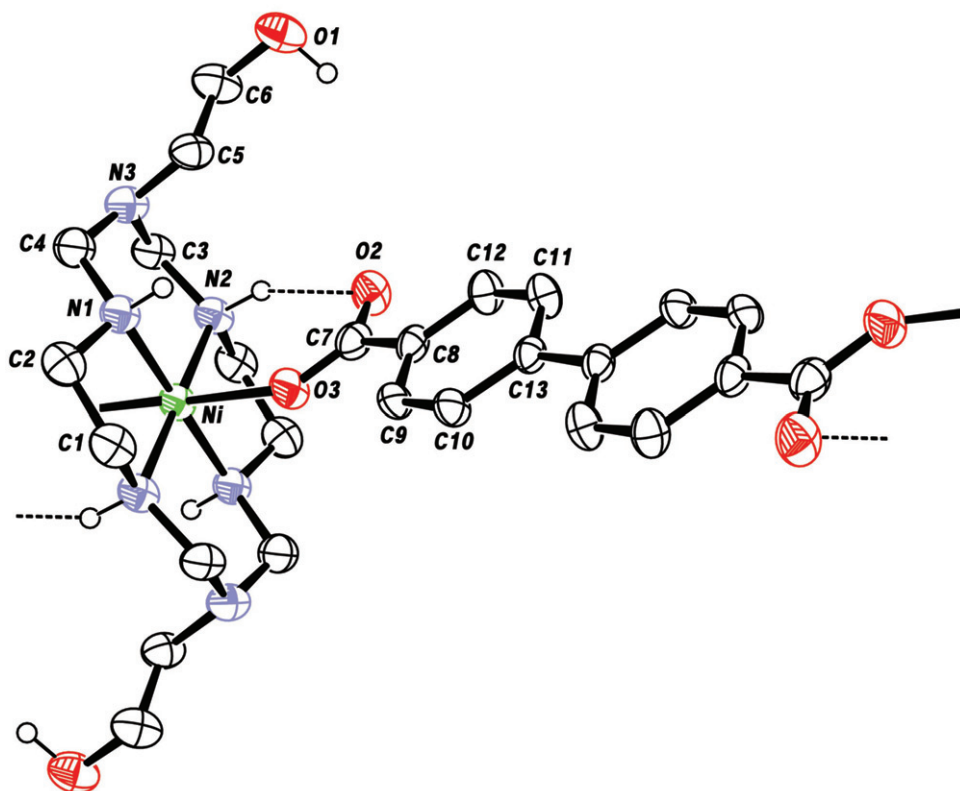


Figure 1. Molecular structure of **1** with atom-labeling scheme. Hydrogens other than those participating in hydrogen bonding are omitted for clarity.

the Denzo-SMN package [15]. Structures were solved and refined using the SHELXTL\PC V6.1 package [16]. Refinement was performed by full-matrix least squares on F^2 using all data (negative intensities included). Hydrogens were included in calculated positions. During the refinement of the structure for **1**, electron density peaks were located that were believed to be highly disordered solvent molecules (possibly H_2O and/or DMF). Attempts to model the solvent molecule were not successful. The SQUEEZE option in PLATON [17] indicated that there was a solvent cavity of volume $155.0 \text{ e} \text{ \AA}^{-3}$ containing approximately 27 electrons. In the final cycles of refinement, this contribution to the electron density was removed from the observed data. The density, the $F(000)$ value, the molecular weight, and the formula are given without taking into account the results obtained with the SQUEEZE option PLATON [17]. Similar treatments of disordered solvent molecules were reported in other systems [18–21].

3. Results and discussion

3.1. Structure of **1**

The molecular structure of **1** with atom labeling is shown in figure 1; selected bond distances and angles are given in table 2. The nickel(II) sits on an inversion center and

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

Ni1–N1	2.066(2)	Ni1–N2	2.056(2)
Ni1–O3	2.0976(16)		
N2–Ni–N1	86.00(9)	N2–Ni1–N1 ^{#1}	94.00(9)
N2–Ni1–O3	92.26(8)	N2 ^{#1} –Ni1–O3	87.74(8)
N1–Ni1–O3	91.62(8)	N1 ^{#1} –Ni1–O3	88.38(8)

Symmetry transformations used to generate equivalent atoms: ^{#1} $-x+1/2, -y+1/2, -z+1$.

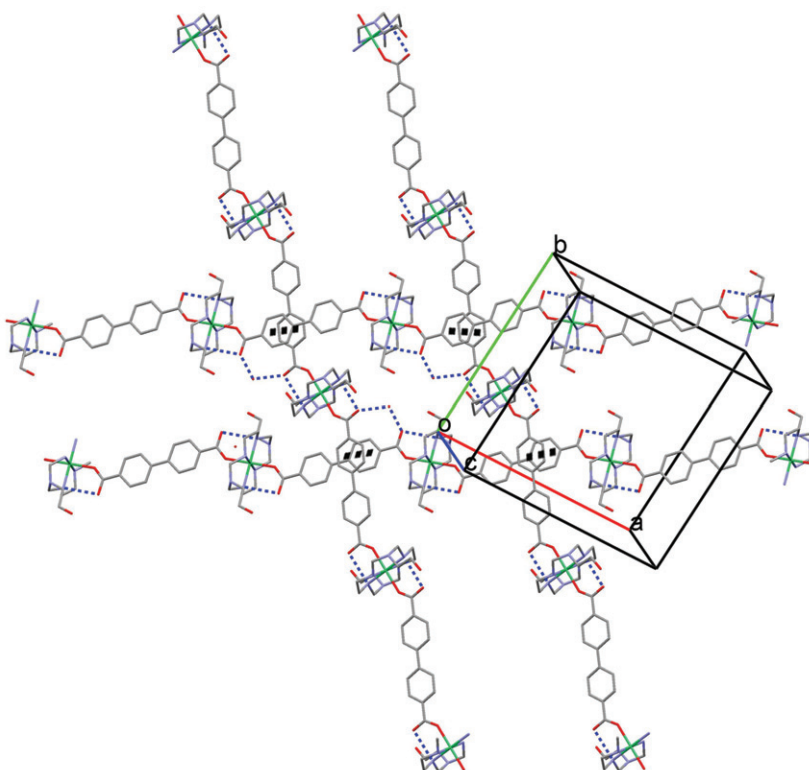


Figure 2. Diagram showing how a 3-D network is constructed from two different series of 1-D coordination polymers. Dashed lines (· · ·) indicate hydrogen bonds. Square dotted (■ ■ ■) lines indicate π – π interactions between the phenyl rings.

the coordination geometry around nickel(II) displays a tetragonally distorted octahedron, with four secondary nitrogens from the macrocycle and two oxygens from carboxylates. The basic skeleton of nickel(II) macrocycle in **1** is close to those reported for $[\text{Ni}(\text{L})]^{2+}$ compounds, although the central nickel(II) has different axial ligands. The Ni–N distances of 2.066(2) and 2.056(2) Å are comparable to those of related examples ($[\text{Ni}(\text{L})(\text{TCNQ})_2]$: Ni–N = 2.053(4), 2.056(5) Å; $[\text{Ni}(\text{L})_3[\text{Cr}(\text{CN})_5(\text{NO})]_2 \cdot 8\text{H}_2\text{O}$: Ni–N = 2.044(8), 2.072(7), 2.059(8), 2.086(8) Å; *catena*-(μ - CrO_4 -*O,O'*)[Ni(L)] $\cdot 5\text{H}_2\text{O}$: Ni–N = 2.051(2), 2.052(2), 2.070(3) Å; $[\text{Ni}(\text{L})_2[\text{Mo}(\text{CN})_8] \cdot 8\text{H}_2\text{O}$: Ni–N = 2.014(3),

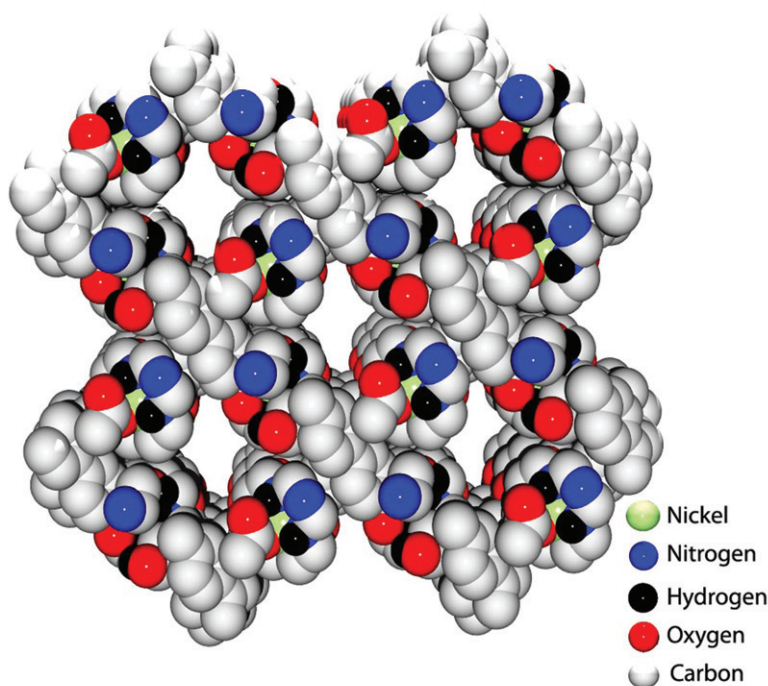


Figure 3. Extended space-filling view of **1** showing 1-D channels. Water molecules inside the channels are omitted for clarity.

2.072(2) Å; TCNQ = 7,7,8,8-tetracyano-*p*-quinodimethane) [14, 22–24]. The axial Ni–O bond distance of 2.0976(16) Å is slightly shorter than previously reported values in $\{[\text{Ni}(\text{L})(\text{tp})]\cdot 4\text{H}_2\text{O}\}_n$: Ni–O = 2.129(5) Å; $[\text{Ni}(\text{cyclam})(\text{Hdipic})_2]\cdot 2\text{H}_2\text{O}$: Ni–O = 2.1242(19) Å; $\{[\text{Ni}(\text{L1})(\text{dipic})]\cdot (\text{H}_2\text{O})\}_n$: Ni–O = 2.115(4) Å; tp = terephthalate, cyclam = 1,4,8,11-tetraazacyclotetradecane, dipic = dipicolinate, L1 = 2,5,9,12-tetramethyl cyclam) [11, 25, 26]. The structure of **1** has two different series of 1-D coordination polymers which run in different directions and cross at one of the aromatic rings of the bridging 4,4'-bpdc ligands. Two phenyl rings of 4,4'-bpdc ($\angle \text{C11}-\text{C13}-\text{C13}\#2-\text{C11}\#2 = 180^\circ$, symmetry code: #2 - x , - $y + 1$, - z) have π - π stacking interactions at the phenyl ring intersection $\{d(\text{centroid} \cdots \text{centroid}) = 3.673 \text{ \AA}\}$ (figure 2). Recent report by Liu *et al.* [9] shows the importance of π - π and C–H $\cdots\pi$ interactions in the formation of 1-D assembly of metal complexes. The 1-D chains in compound **1** cross in a way of crosswise stacked logs to generate a 3-D supramolecule having 1-D channels. Lattice waters are contained in the channels (figure 3), where water interacts with uncoordinated oxygen of 4,4'-bpdc ligands to form hydrogen bonds between the 1-D polymeric chains (table 3).

Suh *et al.* [11] created a similar self-assembled polymer $\{[\text{Ni}(\text{L})(\text{tp})]\cdot 4\text{H}_2\text{O}\}_n$ from the nickel(II) tetraazamacrocyclic and tp ligands, in which two different series of 1-D polymeric chains are crosswise stacked logs, and each 1-D chain is connected by hydrogen-bonding interactions yielding 1-D channels.

Table 3. Hydrogen bonds for **1** (Å and °).

D–H...A	d(D–H)	d(H...A)	d(D...A)	∠(DHA)
O1–H1O...O2W	0.98(4)	1.75(4)	2.722(4)	170(3)
N1–H1...O1 ^{#3}	0.93	2.28	3.114(3)	149.4
N2–H2...O2	0.93	2.13	2.967(3)	150.0
O1W–H1WA...O2	0.85	1.94	2.789(3)	178.9
O2W–H2WB...O2	0.84	1.97	2.806(3)	180

Symmetry transformations used to generate equivalent atoms: ^{#3}*x*, *y*, *z* + 1.

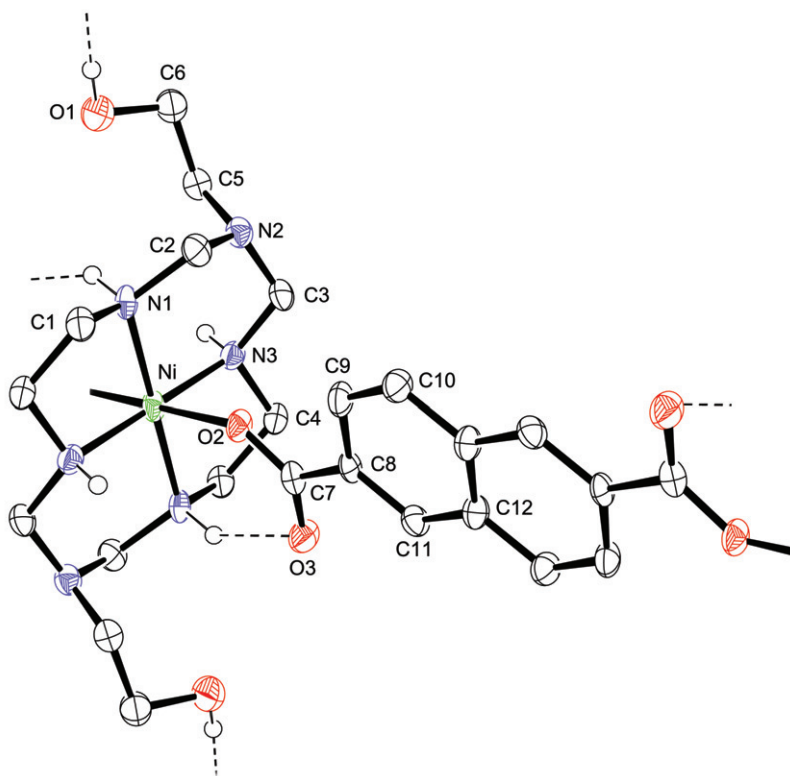


Figure 4. Molecular structure of **2** with atom-labeling scheme. Hydrogens are omitted for clarity.

3.2. Structure of **2**

The molecular structure of **2** with atom-labeling scheme is shown in figure 4. Important bond distances and angles are given in table 4. Nickel(II) sits on an inversion center. The structure of **2** consists of a 1-D coordination polymer with a basic $\{[\text{Ni}(\text{L})(2,6\text{-ndc})] \cdot 2\text{CH}_3\text{CN}\}$ unit. The coordination environment around nickel(II) is described as a distorted octahedron with four Ni–N bonds from the macrocycle and two Ni–O bonds from 2,6-ndc ligands. The Ni–N bond distances are from 2.049(3) to 2.057(3) Å and similar to those of **1**. Weaker Ni–N bonds are involved in the octahedral species

Table 4. Selected interatomic distances (Å) and angles (°) for **2**.

Ni1–N1	2.049(3)	Ni1–N3	2.057(3)
Ni1–O2	2.142(2)		
N1–Ni1–N3	93.87(11)	N1–Ni1–N3 ^{#1}	86.13(11)
N1–Ni1–O2	85.90(10)	N3–Ni1–O2	93.21(10)
N1 ^{#1} –Ni1–O2	94.10(10)	N3 ^{#1} –Ni1–O2	86.79(10)

Symmetry transformations used to generate equivalent atoms: ^{#1} $-x+1, -y+1, -z+1$.

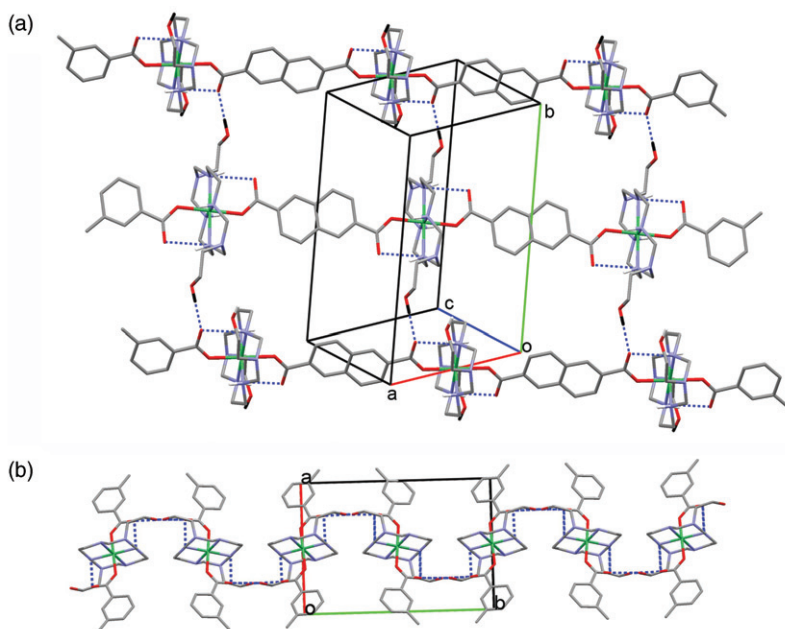


Figure 5. (a) View of 1-D coordination polymers and hydrogen-bonding interactions between 1-D chains in **2**; (b) undulating hydrogen bonds between 1-D chains. Dashed lines indicate hydrogen bonds.

(Ni–N = 2.07 ~ 2.10 Å) than in the square-planar species (Ni–N = 1.88 ~ 1.91 Å) [27, 28]. The Ni–O bond distance of 2.142(2) Å is comparable to the previously reported value in the related system ([Ni(L2)(isonicotinate)₂].H₂O: Ni–O = 2.135(4) Å; L2 = 3,10-di(4-methyl-pyridine)-1,3,5,8,10,12-hexaazacyclotetradecane) [8]. The carboxylates of the bridging 2,6-ndc are nearly in the naphthalene ring plane (\angle C11–C8–C7–O2 = 177°). As illustrated in figure 5, two different series of 1-D coordination polymers with tilted macrocycles are observed in **2**, running in the same direction. The 1-D polymers are linked *via* hydrogen bonds between the hydroxyethyl pendant groups of the hexaazamacrocycles and uncoordinated oxygens of 2,6-ndc in an undulated fashion, giving a 3-D supramolecule (table 5). The lattice acetonitriles in **2** are interconnected by hydrogen bonds between the secondary nitrogens of the hexaazamacrocycles and the nitrogen of acetonitrile.

Table 5. Hydrogen bonds for **2** (Å and °).

D–H...A	d(D–H)	d(H...A)	d(D...A)	∠(DHA)
O1–H1O...O3 ^{#3}	0.94(5)	1.78(5)	2.719(4)	175(5)
N1–H1...O3 ^{#1}	0.93	2.09	2.921(4)	147.7
N3–H3...N4 ^{#4}	0.93	2.44	3.194(6)	138.0

Symmetry transformations used to generate equivalent atoms: ^{#1} $-x+1, -y+1, -z+1$; ^{#3} $-x+1, y+1/2, -z+3/2$; ^{#4} $-x+1, -y+1, -z+2$.

3.3. Analytical, physical, and spectroscopic properties for **1** and **2**

Microanalysis for **1** was consistent with a molecule of water in addition to the formula **1**. Due to the rapid desolvation of acetonitrile of **2** even at room temperature in air, the microanalytical analysis for the desolvated compound **2'** was obtained and gave a satisfactory result.

The presence of carboxylate in **1** was suggested by the strong absorptions at 1583 ($\nu_{\text{as}}\text{COO}$) and 1273 ($\nu_{\text{s}}\text{COO}$) cm^{-1} in the IR spectrum (Nujol mull). In addition, a broad band at 3380 (νOH) cm^{-1} and a weak band at 3195 (νNH) cm^{-1} indicate that the macrocyclic ligand is present in **1**. In the IR spectrum of **2**, strong bands were observable at 1597 ($\nu_{\text{as}}\text{COO}$) and 1292 ($\nu_{\text{s}}\text{COO}$) cm^{-1} , with a weak band at 3197 (νNH) cm^{-1} and a broad at 3425 (νOH) cm^{-1} .

The solid-state electronic spectra for **1** and **2** exhibit d–d transitions at 320, 503, and 665 nm (for **1**) and 328, 506, and 649 nm (for **2**). The bands are expected from a high-spin d^8 nickel(II) ion in a distorted octahedral environment (${}^3\text{B}_{1\text{g}} \rightarrow {}^3\text{E}_{\text{g}}$, ${}^3\text{B}_{1\text{g}} \rightarrow {}^3\text{E}_{\text{g}}$, ${}^3\text{B}_{1\text{g}} \rightarrow {}^3\text{B}_{2\text{g}} + {}^3\text{B}_{1\text{g}} \rightarrow {}^3\text{A}_{2\text{g}}$) [29, 30].

4. Conclusion

We prepared and characterized two macrocyclic nickel(II) supramolecules by self-assembly of macrocyclic nickel(II) complexes containing hydroxyethyl pendant groups with aromatic carboxylates. In **1**, two different series of 1-D coordination polymers which run in different directions are interconnected by π – π stacking and hydrogen-bonding interactions, resulting in the formation of a 3-D supramolecule. In **2**, 1-D chains running in the same direction are interconnected by hydrogen bonds between pendant hydroxyethyl groups of the macrocycle and bridging aromatic carboxylates to yield a 3-D metallosupramolecule.

Supplementary material

CCDC 749268 and 749269 contain the supplementary crystallographic data for **1** and **2**, respectively. These data can be obtained free of charge *via* <http://www.ccdc.cam.ac.uk/conts/retrieving.html>, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax (+44) 1223-336-033; or E-mail: deposit@ccdc.cam.ac.uk.

Acknowledgments

This work was supported by the Korea Research Foundation Grant funded by the Korean Government (MOEHRD), Basic Research Promotion Fund (KRF-2008-521-C00137).

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