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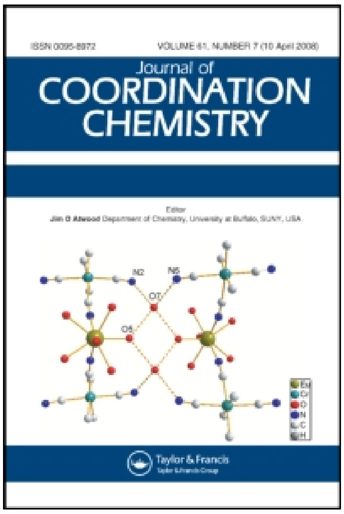

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Two diamond-type octacyanometallate(IV)-nickel(II) assemblies chelated by macrocyclic ligands

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Two diamond-type octacyanometallate(IV)–nickel(II) assemblies chelated by macrocyclic ligands

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Two octacyanometallate-based Ni^{II}–M^{IV} [M = Mo(**1**), W(**2**)] bimetallic assemblies chelated with tetradentate macrocyclic ligands have been synthesized by slow diffusion and characterized structurally. In both complexes, M and Ni centers acting as linker and connector, respectively, are connected by M–CN–Ni–NC–M linkages to form a 3-D diamond-type topological network. Magnetic behaviors of both complexes show a very weak antiferromagnetic interaction between Ni^{II} ions mediated by the diamagnetic $[M(CN)_8]^{4-}$ bridges.

Keywords: Octacyanometallates; Nickel complex; Macrocyclic ligands; Crystal structure

1. Introduction

Considerable research has been made on the construction of $[M(CN)_8]^{3-/4-}$ (M = Mo, W)-based magnets owing to their interesting functionalities [1]. The combination of $[M(CN)_8]^{3-}$ as a carrier of unpaired spin (d^1) with paramagnetic metal centers M′ (M′ = Cu^{II}, Mn^{II}, Fe^{II}, Co^{II}, Ni^{II}) has produced various dimensional structures with multifarious magnetic properties, such as photo-induced magnetism [2], reasonably high Curie temperatures [3], and single-molecule magnetism [4]. In particular, chemical modification of the Ni_9W_6 cluster by replacing the alcoholic solvent molecules with 2,2′-bipyridine yielded air-stable, bipyridine-capped Ni_9W_6 cluster ($S = 12$) exhibiting SMM behavior [4a].

As a part of our research toward molecular chemistry for magnetic materials, a rational strategy is to employ octahedral M′(II) (M′ = Ni, Cu) precursors blocked by tetradentate macrocyclic ligands in which four equatorial positions are occupied by the macrocycles and two axial sites are reserved for the incoming bridging ligands [5]. We have attempted to seek the correlation between adjusted structural parameters and resulting magnetic character by varying pendant groups on macrocyclic ligands. We have extended our work to paramagnetic $[M(CN)_8]^{3-}$ (M = Mo, W) and $[Ni(L)]^{2+}$ (L = tetradentate macrocyclic ligands having different pendant groups) (scheme 1) as building blocks to construct heterometallic assemblies with interesting magnetic properties.

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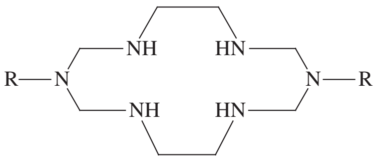

$R = CH_2CH_2OH$ (**L1**)

CH_2CH_3 (**L2**)

Scheme 1. Structure of ligands L1 and L2.

Unfortunately, the M^V ions have been reduced during the reaction process and two new 3-D diamond-type organic–inorganic hybrid materials were obtained: $[Ni^{II}(L1)]_2[Mo^{IV}(CN)_8] \cdot 3DMF \cdot 2H_2O$ (**1**) (L1 = 3,10-diethanol-1,3,5,8,10,12-hexaazacyclotetradecane) and $[Ni^{II}(L2)]_2[W^{IV}(CN)_8] \cdot 4DMF \cdot 4H_2O$ (**2**) (L2 = 3,10-diethyl-1,3,5,8,10,12-hexaazacyclotetradecane). In this article, we report the syntheses, crystal structures, and magnetic properties of both complexes.

2. Experimental

2.1. *Preparations and methods*

All chemicals purchased were of reagent grade or better and were used without purification. Elemental analyses for C, H, and N were performed with a Perkin-Elmer 240C elemental analyzer. All the magnetization data were recorded on a Quantum Design MPMS-XL7 SQUID magnetometer. Variable-temperature magnetic susceptibility measurements for **1** and **2** were performed in an applied field of 2 kOe in the temperature range 300–1.8 K. The molar magnetic susceptibilities were corrected for diamagnetism estimated from Pascal's tables [6] and for the sample holder by a previous calibration.

2.1.1. $[Ni^{II}(L1)]_2[Mo^{IV}(CN)_8] \cdot 3DMF \cdot 2H_2O$ (1). Single crystals of **1** were prepared at room temperature by slow diffusion of a dimethylformamide (DMF) solution (2 mL) containing $[Ni(L1)](ClO_4)_2$ [7] (0.075 mmol) into a DMF solution (20 mL) of $[HN(n\text{-}C_4H_9)_3]_3[Mo(CN)_8] \cdot 4H_2O$ [8] (0.05 mmol). After about 2 weeks, block-shaped yellow crystals were obtained. Yield: 26%. Elemental analysis Calcd (%) for $C_{41}H_{84}MoN_{23}Ni_2O_9$: C, 39.19; H, 6.74; N, 25.64; found: C, 39.33; H, 6.61; N, 25.71.

2.1.2. $[Ni^{II}(L2)]_2[W^{IV}(CN)_8] \cdot 4DMF \cdot 4H_2O$ (2). Single crystals of **2** were prepared at room temperature by slow diffusion of a DMF solution (2 mL) containing $[Ni(L2)](ClO_4)_2$ [7] (0.075 mmol) into a DMF solution (20 mL) of $[HN(n\text{-}C_4H_9)_3]_3[W(CN)_8] \cdot 4H_2O$ [8] (0.05 mmol). After about 2 weeks, block-shaped yellow crystals were obtained. Yield: 33%. Elemental analysis Calcd (%) for $C_{44}H_{96}N_{24}Ni_2O_8W$: C, 38.00; H, 6.96; N, 24.17; found: C, 38.16; H, 6.75; N, 24.06.

Table 1. Crystal data and structure refinement of X-ray data collection for **1** and **2**.

Complex	**1**	**2**
Empirical formula	$C_{41}H_{84}MoN_{23}Ni_2O_9$	$C_{44}H_{96}N_{24}Ni_2O_8W$
Formula weight	1256.67	1390.73
Crystal system	Monoclinic	Tetragonal
Space group	$C2/c$	$I4_1/a$
Unit cell dimensions (Å, °)		
a	17.267(2)	19.1573(9)
b	25.080(3)	19.1573(9)
c	16.686(5)	16.6532(15)
β	119.747(3)	90.00
Volume (Å^3), Z	6274(2), 4	6111.8(7), 4
Calculated density (g cm^{-3})	1.330	1.511
Absorption coefficient (Mo-Kα) (mm^{-1})	0.855	2.555
Total/unique	19347/6073	23579/3011
R_{int}	0.045	0.081
Goodness-of-fit on F^2	1.038	1.049
R_1, ωR_2 [$I > 2\sigma(I)$]	0.0499/0.0716	0.0389/0.0429
R_1, ωR_2 (all data)	0.1172/0.1231	0.1082/0.1144
Largest difference peak/hole (e Å^{-3})	0.594/−0.761	0.481/−0.966

Note: The oxidation state of molybdenum and tungsten decreasing from V to IV has also been observed in other octacyanometallate-based complexes [9], and the reason is under further investigation.

2.2. *X-ray crystallography*

Single-crystal X-ray diffraction measurements for **1** and **2** were carried out on a Bruker APEX II diffractometer equipped with Mo-Kα ($\lambda = 0.71073$ Å) radiation. Diffraction data analysis and reduction were performed within SMART and SAINT+ [10]. Correction for Lorentz, polarization, and absorption effects were performed within SADABS [11]. Structures were solved using the Patterson method within SHELXL-97 and refined using SHELXL-97 [12]. All non-hydrogen atoms were refined with anisotropic thermal parameters. All hydrogens except those bound to water were calculated at idealized positions with C–H = 0.97, or 0.96, N–H = 0.91 and O–H = 0.96 Å and included in the refinement in a riding mode with U_{iso} for H assigned as 1.2 or 1.5 times U_{eq} of the attached atoms. Hydrogens bound to water were located from difference maps and refined as riding (O–H = 0.85 Å), with $U_{iso}(H) = 1.2U_{eq}(O)$. The crystallographic data and experimental details for structural analyses of **1** and **2** are summarized in table 1. Selected bond lengths and angles are listed in tables 2 and 3.

3. Results and discussion

Single-crystal X-ray analysis reveals that **1** and **2** crystallize in the monoclinic ($C2/c$) and tetragonal ($I4_1/a$) space groups, respectively (table 1). ORTEP diagrams of **1** and **2** are shown in figure 1. In both complexes, $[M(CN)_8]^{4-}$ displays a slightly distorted square antiprismatic geometry with four bridging and four terminal cyano groups,

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

Mo1–C1	2.114(4)	Ni2–N3	2.119(3)
Mo1–C2	2.129(4)	Ni2–N8	2.037(3)
Mo1–C3	2.139(4)	Ni2–N10	2.078(3)
Mo1–C4	2.148(4)	C1–N1	1.172(5)
Ni1–N1	2.073(3)	C2–N2	1.111(5)
Ni1–N5	2.077(3)	C3–N3	1.157(5)
Ni1–N7	2.072(3)	C4–N4	1.116(5)
Mo1–C1–N1	178.0(3)	C1–Mo1–C1^{iv}	101.7(2)
Mo1–C2–N2	176.0(3)	C1–Mo1–C3^{iv}	78.33(15)
Mo1–C3–N3	176.6(3)	C1^{iv}–Mo1–C3^{iv}	143.74(15)
Mo1–C4–N4	176.6(4)	C1–Mo1–C3	143.74(15)
Ni1–N1–C1	154.6(3)	C1^{iv}–Mo1–C3	78.33(15)
Ni2–N3–C3	155.2(3)	C3^{iv}–Mo1–3	122.9(2)

Symmetry code: $^{iv}-x+2, y, -z+1/2$.

Table 3. Selected bond distances (Å) and angles (°) for **2**.

W1–C1	2.149(4)	Ni1–N5	2.059(3)
W1–C2	2.174(4)	C1–N1	1.154(5)
Ni1–N1	2.106(3)	C2–N2	1.169(5)
Ni1–N3	2.083(3)		
W1–C1–N1	175.9(3)	C1^{iii}–W1–C1	143.4(2)
W1–C2–N2	178.0(3)	C1^{ii}–W1–C1^{iv}	143.4(2)
Ni1–N1–C1	151.7(3)	C1^{iii}–W1–C1^{iv}	95.66(6)
C1^{ii}–W1–C1^{iii}	95.66(6)	C1–W1–C1^{iv}	95.66(6)
C1^{ii}–W1–C1	95.66(6)		

Symmetry codes: $^{ii}-y+5/4, x+1/4, -z+1/4$; $^{iii}-x+1, -y+3/2, z$; $^{iv}y-1/4, -x+5/4, -z+1/4$.

typical for $[M(CN)_8]^{4-}$-based complexes [2b, 13]. The average values of edge lengths of the basal plane for Mo and W are 1.1877 and 1.1924 times that of M–C bond lengths, respectively, comparable to those of the most favorable square antiprism (1.2156 times) [14]. For **1**, the mean values of M–C and C–N bond lengths are 2.133 and 1.139 Å, respectively, while 2.162 and 1.162 Å for **2**. The M–C–N bond angles are nearly linear with a maximum deviation from linearity of 4.1°.

The Ni centers in **1** and **2** adopt an axially *trans*-elongated octahedral geometry, constituted by four equatorial N atoms of the macrocyclic ligand and two axial nitrogens of bridging CN. The mean equatorial and apical Ni–N distances are 2.066 Å and 2.096 Å, respectively, for **1** and 2.071 Å and 2.106 Å for **2**. The Ni–N–C bridging units are strongly bent (for **1**, 154.6(3)° and 155.2(2)° for Ni1–N1–C1 and Ni2–N3–C3, respectively; for **2**, 151.7(3)° for Ni1–N1–C1) in opposition to almost linear M–C–N units. These geometric parameters are reminiscent of those in related $[Ni(L)]^{2+}$ (L = tetradentate macrocyclic ligands) complexes [15].

From a topological viewpoint, each $[Ni(L)]^{2+}$ in **1** and **2** is a linear two-connecting node, while each $[M(CN)_8]$ unit is a tetrahedral four-connecting one. The values of C–Mo(W)–C angles participating in propagation of the networks for **1** and **2** range from 78.33(15)° to 143.74(15)° and from 95.66(6)° to 143.4(2)°, respectively (tables 2 and 3). These nodes of $[M(CN)_8]$ units are linked to four adjacent $[M(CN)_8]$ units in a *trans* arrangement by the $[Ni(L)]^{2+}$ units. As a result, **1** and **2** both display a

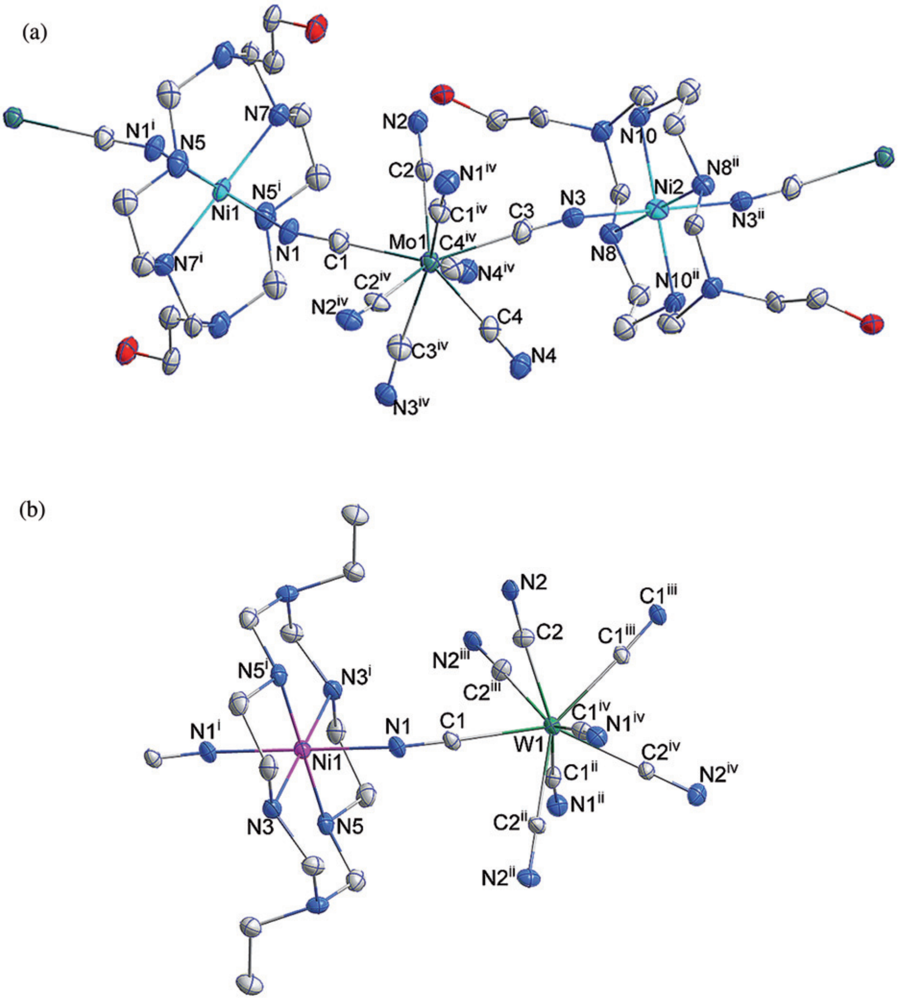

Figure 1. ORTEP diagrams of (a) **1** and (b) **2** showing the 30% probability thermal motion ellipsoid. Non-coordinated DMF and water have been omitted for clarity. Symmetry codes for **1**: $^{\mathrm{i}}-x+3/2, -y+3/2, -z$; $^{\mathrm{ii}}-x+2, -y+2, -z+1$; $^{\mathrm{iv}}-x+2, y, -z+1/2$. Symmetry codes for **2**: $^{\mathrm{i}}-x+1, -y+1, -z$; $^{\mathrm{ii}}x+1/4, -y+5/4, -z+1/4$; $^{\mathrm{iii}}-x+1, -y+3/2$, z; $^{\mathrm{iv}}-x+5/4, y-1/4, -z+1/4$.

diamond-type topological network with various degrees of distortion (figure 2), observed in other octacyanometallate-based bimetallic assemblies [9a]. The 3-D structural features for **1** and **2** are significantly different from those of other nickel- or cyanide-containing complexes with lower dimensions [16]. Moreover, to the best of our knowledge, there are limited examples of 3-D octacyano- and nickel-based heterobimetallic systems [5b, 5c, 5e, 9a, 17]. For **1**, the pendant hydroxyl groups on L1 undergo hydrogen bonds with DMF, lattice water molecules, and the secondary amines of L1. In addition, the amines on L1 and lattice water molecules, both participate in hydrogen bonding to terminal CN groups of $[\mathrm{Mo(CN)_8}]^{4-}$ (table 4). For **2**, DMF, water, and L2 are also involved in hydrogen-bonding interactions (table 5).

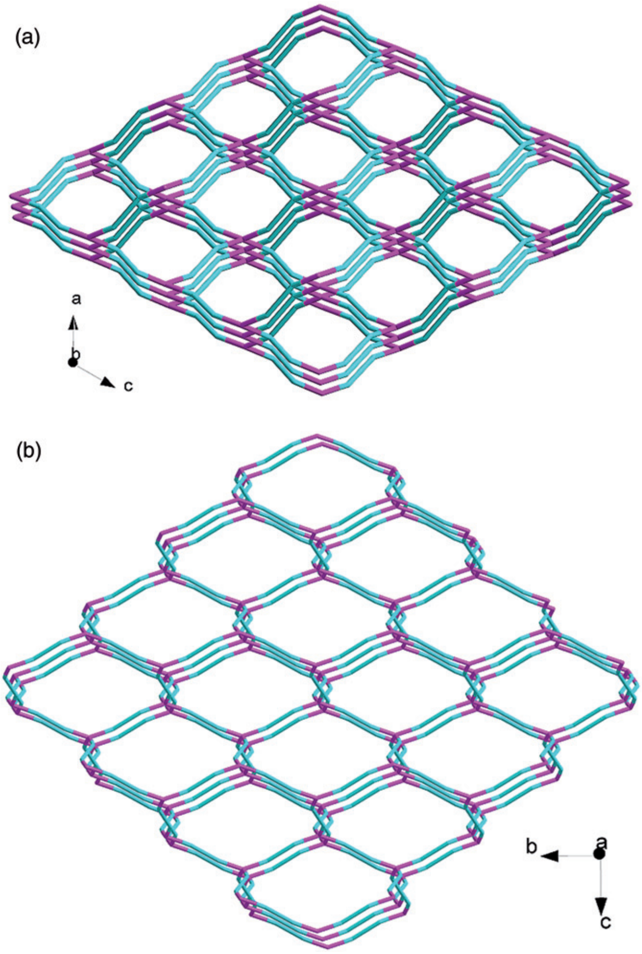

Figure 2. Topological depictions of (a) **1** and (b) **2**, where the M nodes and the Ni connectors are shown in pink and turquoise, respectively.

Table 4. Hydrogen-bond geometry (Å, °) for **1**.

D–H···A	d(D–H)	d(H···A)	d(D···A)	∠(D–H···A)
N5–H5C···O1^{iv}	0.91	2.42	3.095(4)	131
N7–H7C···N2	0.91	2.40	3.254(4)	156
N8–H8C···N4^{iv}	0.91	2.42	3.277(5)	157
O1–H1A···O5^{v}	0.96	2.57	3.047(4)	111
O2–H2A···N2	0.96	2.60	3.448(4)	147
O3–H3A···N2	0.85	2.05	2.902(7)	179
O3–H3B···O1	0.85	1.83	2.628(6)	156

Symmetry codes: $^{iv}-x+2$, y, $-z+1/2$; $^{v}x+1/2$, $y-1/2$, z.

Table 5. Hydrogen-bond geometry (Å, °) for **2**.

D–H···A	d(D–H)	d(H···A)	d(D···A)	∠(D–H···A)
N3–H3···N2^{v}	0.91	2.32	3.132(5)	149
N5–H5···O1^{vi}	0.91	2.38	3.033(5)	128
O2–H2D···O1	0.85	2.30	3.100(5)	157
O2–H2B···O1^{vii}	0.85	2.23	2.931(5)	140

Symmetry codes: $^{v}x, y-1/2, -z$; $^{vi}-y+5/4, x-1/4, z-1/4$; $^{vii}y+1/4, -x+5/4, z+1/4$.

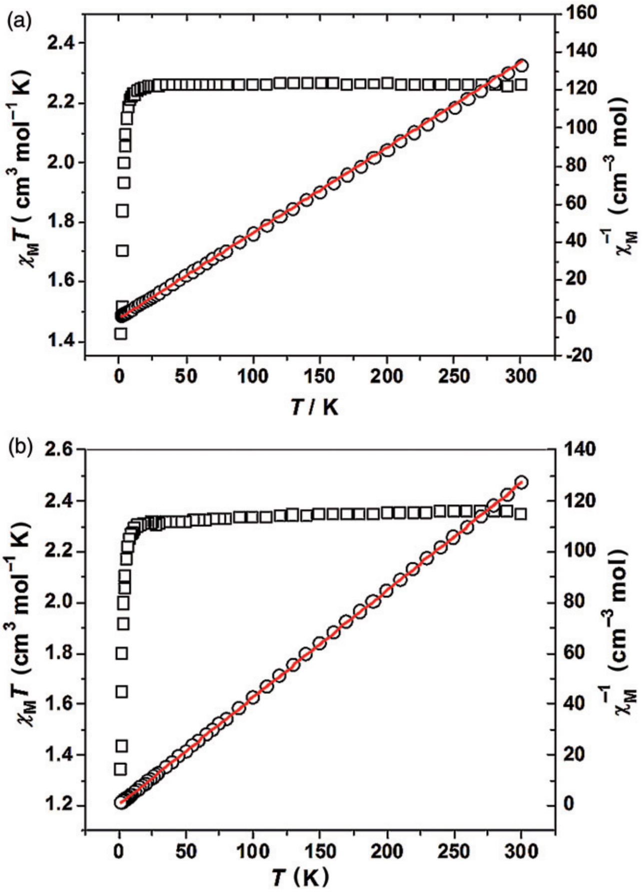

Figure 3. Temperature dependence of $\chi_M T$ (□) and $1/\chi_M$ (○) for **1** (a) and **2** (b) measured at 2 kOe. The solid line represents the fit obtained by the Curie–Weiss law.

The plots of $\chi_M T$ *versus* T and $1/\chi_M$ *versus* T are given in figure 3. At room temperature, the $\chi_M T$ values for each Ni_2M unit (M = Mo, W) are 2.26 and 2.35 cm^3 K mol^{-1} for **1** and **2**, respectively, slightly higher than the $\chi_M T$ value of 2.0 cm^3 K mol^{-1} calculated for two uncoupled Ni^{II} ions ($S = 1$, $g = 2$). On lowering the temperature, the $\chi_M T$ values remain almost constant until 8.0 K (for **1**) and 14 K (for **2**). After that, the $\chi_M T$ values decrease with further decrease in temperature to

$1.40\,cm^3\,K\,mol^{-1}$ (for **1**) and $1.34\,cm^3\,K\,mol^{-1}$ (for **2**) at 1.8 K. Plots of $1/\chi_M$ *versus* T obey the Curie–Weiss law with a negative Weiss constant $\theta = -0.2\,K$ (for **1**) and $-0.7\,K$ (for **2**). Such magnetic behavior indicates that the diamagnetic $[M(CN)_8]^{4-}$ (M = Mo, W) bridges mediate a very weak antiferromagnetic interaction between Ni^{II} ions. The more negative Weiss constant of **2** can be explained by the fact that 5d orbitals with the large diffusion are advantageous to enhance coupling between metal ions [9a, 13a].

4. Conclusion

We prepared two cyano-bridged bimetallic Ni–M (M = Mo(**1**), W(**2**)) complexes chelated with tetradentate macrocyclic ligands having different pendant groups. Although the pendant groups noticeably affect the space groups of **1** and **2**, both complexes display a similar 3-D diamond-like network. Magnetic behaviors of both complexes show a very weak antiferromagnetic interaction between Ni^{II} ions mediated by the diamagnetic $[M(CN)_8]^{4-}$ (M = Mo, W) bridges.

Supplementary material

Crystallographic data for the structure reported in this article has been deposited with the Cambridge Crystallographic Data Center, CCDC nos 754006 and 754007 for **1** and **2**, respectively. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB21EZ, UK; Fax: +44-1223-336033; Email: deposit@ccdc.cam.ac.uk.

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