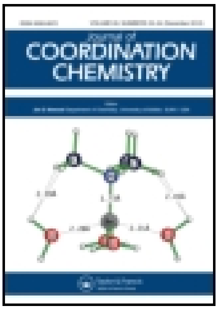

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A cubane-like [Ni_4O_4] cluster and a chloro-bridged dinuclear copper complex incorporating a hydroxyl-rich ligand: syntheses and crystal structures

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A cubane-like [Ni_4O_4] cluster and a chloro-bridged dinuclear copper complex incorporating a hydroxyl-rich ligand: syntheses and crystal structures

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A tetranuclear cubane-like cluster $[Ni_4(HL)_4(DMSO)_2]\cdot 2DMSO\cdot H_2O$ (**1**) and a dinuclear compound $[Cu_2Cl_2(H_2L)_2]\cdot 1.5MeOH$ (**2**) ($H_3L = $ 3-(2-hydroxy-benzylideneamino)propane-1,2-diol) have been prepared and characterized by X-ray single crystal diffraction, IR, and elemental analysis. The X-ray diffraction analysis indicated that the four metals of **1** showed a Ni_4O_4 cubane arrangement, and two of the six faces of the Ni_4O_4 cubane were capped by DMSO. Complex **1** is the first reported DMSO capped Ni_4O_4 cubane cluster. Complex **2** contains a u_2-Cl bridged dinuclear core with each Cu^{II} ion exhibiting pseudo square pyramidal geometry.

Keywords: Tetranuclear; Cubane-like; Nickel; Copper

1. Introduction

Molecular 3d-metal clusters continue to attract attention [1] due to potential applications as catalysts for organic synthesis [2–5], models of metal-containing sites in biology [6–8], and single molecular magnets [9–12] or single chain magnets [13, 14]. A synthetic challenge involves discovering simple and suitable ligands, which possess arrays of coordination pockets organized to create high-nuclearity molecules.

One route for preparation of paramagnetic metal clusters is using hydroxyl-rich Schiff-base ligands. These ligands possess nitrogen donors that can bind to a metal and alkoxides that participate in cluster formation by bridging two or three metals.

Schiff bases derived from condensation between salicylaldehyde and hydroxyl-rich amino alcohols have been reported for construction of high-nuclearity molecules [15–18]. Surprisingly, coordination potential of H_3L ($H_3L = $ 3-(2-hydroxybenzylideneamino)-propane1,2-diol; scheme 1a), which was prepared by condensation between

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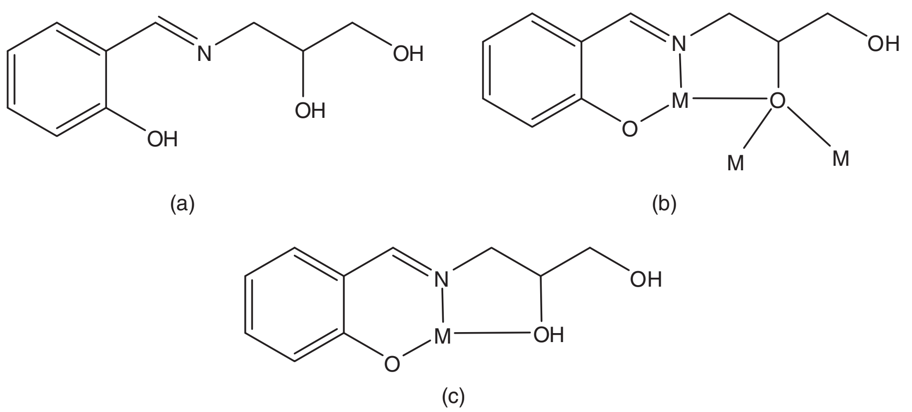


Scheme 1. (a) The structure of H_3L; (b) coordination mode of $[HL]^{2-}$ in **1**; and (c) coordination of $[H_2L]^{-}$ in **2**.

salicylaldehyde and 3-aminopropane-1,2-diol, has been little explored [19, 20]. Ni^{II}/H_3L or Cu^{II}/H_3L high-nuclearity clusters might be possible and thus we have investigated the reaction of H_3L with $NiCl_2 \cdot 6H_2O$ and $CuCl_2 \cdot 2H_2O$. A Ni_4O_4 cubane-like cluster $[Ni_4(HL)_4(DMSO)_2] \cdot 2DMSO \cdot H_2O$ (**1**) and a dinuclear complex $[Cu_2Cl_2(H_2L)_2] \cdot 1.5MeOH$ (**2**) were prepared. Herein, we report the syntheses and characterization of these complexes.

2. Experimental

2.1. *General considerations*

All reagents were of analytical grade. Elemental analyses for carbon, hydrogen, and nitrogen were performed with a Carlo-Erba EA1110 CHNO-S microanalyzer. Infrared spectra (4000–400 cm^{-1}) were recorded by using KBr pellets with a Nicolet MagNa-IR500 FT-IR spectrometer. Crystal determination was performed with a Bruker SMART APEX II CCD diffractometer equipped with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å).

2.2. *Syntheses*

2.2.1. Synthesis of $[Ni_4(HL)_4(DMSO)_2] \cdot 2DMSO \cdot H_2O$ (1). To a solution of H_3L (0.5836 g, 3 mmol) in CH_3OH (20 mL) was added LiOH (0.2534 g, 6 mmol) and $NiCl_2 \cdot 6H_2O$ (0.6969 g, 3 mmol) in CH_3OH (10 mL). Blue precipitates formed immediately. The blue mixture was stirred for 20 h, mixed with DMSO and left to slowly evaporate at room temperature. Blue prismatic crystals were generated after being evaporated for 40 days. Elemental analysis: Calcd for $C_{48}H_{70}Ni_4N_4O_{17}S_4$ (%): C, 43.92; H, 5.19; N, 4.41; S, 8.20. Found (%): C, 44.32; H, 5.28; N, 4.17; S, 8.64. Selected IR data (KBr, cm^{-1}): 3423 (m), 2913 (m), 2829 (m), 1640 (s), 1598 (m), 1540 (s), 1447 (m), 1303 (m), 1005 (s), 872 (m), 755 (m).

2.2.2. Synthesis of [$Cu_2Cl_2(H_2L)_2$] • 1.5MeOH (2). To a solution of H_3L (0.7870 g, 4 mmol) in CH_3OH (30 mL) was added a solution of $CuCl_2 \cdot 2H_2O$ (0.6869 g, 4 mmol) in CH_3OH (10 mL). The resulting dark-green solution was stirred at room temperature for 36 h and left undisturbed at room temperature. Blue crystals were obtained after 20 days. Elemental analysis: Calcd for $C_{43}H_{56}Cl_4Cu_4N_4O_{15}$ (%): C, 40.83; H, 4.46; N, 4.43. Found (%): C, 41.24; H, 4.68; N, 4.72. Selected IR data (KBr, cm^{-1}): 3422 (m), 2918 (s), 2850 (m), 1638 (s), 1600 (m), 1544 (s), 1448 (s), 1289 (m), 1198 (s), 1031 (m), 758 (m).

2.3. *X-ray crystallography*

Single crystals were placed in a Bruker SMART APEX II CCD. The diffraction data were obtained by using graphite-monochromated Mo-Kα radiation with an ω–2θ scan technique at room temperature. The structure was solved by direct methods with SHELX-97 [21]. A full-matrix least-squares refinement on F^2 was carried out using SHELXL-97 [21].

3. Results and discussion

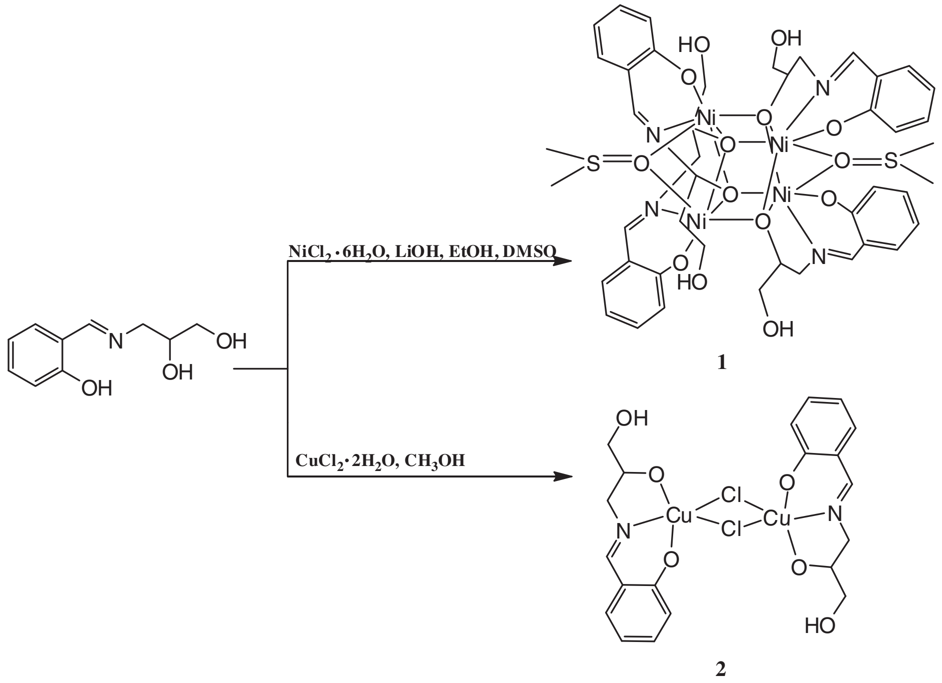
3.1. *Syntheses and IR spectra*

Complex **1** was prepared by mixing H_3L, $NiCl_2 \cdot 6H_2O$ and LiOH in EtOH, and then dissolving the resulting slurry in DMSO to generate a homogenous solution. [$Ni_4(HL)_4(DMSO)_2$] · 2DMSO · H_2O (**1**) was obtained as blue prismatic crystals after evaporating the homogeneous solution at room temperature for 40 days (scheme 2). DMSO played a dual role in the synthesis of **1**, serving as an ancillary ligand and as solvent for H_3L, $NiCl_2 \cdot 6H_2O$, and LiOH.

The presence of the solvate water and the uncoordinated –OH in $[HL]^{2-}$ in **1** was manifested by a broad IR band of medium intensity at 3423 cm^{-1}. Bands at 2918 and 2850 cm^{-1} were assigned to ν(C–C) of $[HL]^{2-}$. The bands between 1640 and 1447 cm^{-1} were ascribed to ν(C=C) of phenyl. The ν(C=N) was observed at 1598 cm^{-1}. The strong intensity band at 1005 cm^{-1} has been assigned to ν(S=O) of DMSO.

Complex **2** was synthesized by mixing H_3L and $CuCl_2 \cdot 2H_2O$ in CH_3OH, then evaporating the resulting blue solution. [$Cu_2Cl_2(H_2L)_2$] · 1.5MeOH (**2**) was obtained as blue crystals after evaporating the blue solution at room temperature for 20 days (scheme 2).

MeOH and uncoordinated –OH in $[H_2L]^-$ in **2** were confirmed by an IR band of medium intensity at 3422 cm^{-1}. Bands at 2918 and 2850 cm^{-1} were assigned to ν(C–C) of $[H_2L]^-$. Bands between 1600 and 1448 cm^{-1} were ascribed to ν(C=C) of phenyl. The ν(C=N) vibration was observed at 1638 cm^{-1}.



Scheme 2. Syntheses of **1** and **2**.

3.2. *Structural descriptions of 1 and 2*

3.2.1. Structural description of 1. Single crystal diffraction analysis reveals that **1** crystallizes in the triclinic space group $P\bar{1}$. Crystallographic data of **1** are listed in table 1; selected bond distances and angles are summarized in table 2. The structure of **1** consists of one [Ni$_4$(HL)$_4$(DMSO)$_2$] (figure 1), one solvate H$_2$O and two solvate DMSO molecules. [Ni$_4$(HL)$_4$(DMSO)$_2$] contains a cubane-like Ni$_4$O$_4$ cluster constructed from four nickels and four oxygen atoms on alternating corners. The coordination environment of each nickel is identical, six-coordinate by one deprotonated phenoxide oxygen, one imine nitrogen, and one deprotonated alkoxide (μ_3-O) from one [HL]$^{2-}$, two deprotonated alkoxide oxygens (μ_3-O) from another two [HL]$^{2-}$ ligands, and one oxygen (μ_2-O) from one DMSO. Therefore, the geometry of each nickel can be described as a distorted octahedron. Each [HL]$^{2-}$ functions as a 3.3110 ligand (Harrison notation [22]), forming a six-membered NiOCCCN chelating ring and a five-membered NiNCCO chelating ring (scheme 1b). Two DMSO molecules serve as bridges, capping two opposite faces of the cubane.

The six faces of the cubane can be classified into two groups according to the bond distances and angles: two opposite faces (OF) capped by DMSO molecules exhibit short Ni–Ni distances and smaller Ni–O–Ni angles than the remaining faces (RF) which have different Ni–O–Ni angles. The structural parameters for the OF are Ni1–Ni2 = 2.8053(15) Å, Ni3–Ni4 = 2.8093(14) Å, Ni1–O2–Ni2 = 85.09(18)°, Ni1–O5–Ni2 = 85.14(17)°, Ni3–O8–Ni4 = 84.79(18)°, and Ni3–O11–Ni4 = 85.52(19)°. The corresponding parameters for the RF are larger than those in OF.

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

	1	**2**
Empirical formula	$C_{46.50}H_{65.50}N_4Ni_4O_{15.75}S_{3.25}$	$C_{43}H_{56}Cl_4Cu_4N_4O_{15}$
Formula weight ($g\,mol^{-1}$)	1271.56	1264.88
Temperature (K)	298 (2)	298(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P2(1)/c$
Unit cell dimensions (Å, °)		
a	13.746(2)	15.459(7)
b	14.725(2)	14.030(7)
c	16.639(3)	24.185(12)
α	100.982(2)	90
β	94.864(2)	95.746(7)
γ	115.519(3)	90
Volume ($Å^3$)	2930.1(8)	5219(4)
Z	2	4
Calculated density ($g\,cm^{-3}$)	1.441	1.610
$F(000)$	1325	2584
Crystal size (mm^3)	0.56 × 0.45 × 0.43	0.56 × 0.43 × 0.29
θ range for data collection (°)	1.67–25.01	1.68–25.01
Limiting indices	$-14 \le h \le 16$; $-17 \le k \le 16$; $-17 \le l \le 19$	$-18 \le h \le 18$; $-16 \le k \le 16$; $-19 \le l \le 28$
Reflections collected/unique	15,063/10,052	25,729/9046
Data/restraints/parameters	10,052/317/710	9046/12/631
Goodness-of-fit	1.053	1.006
R_1, wR_2 [$I > 2\sigma(I)$]	$R_1 = 0.0748$, $wR_2 = 0.1885$	$R_1 = 0.0956$, $wR_2 = 0.2215$
R indices (all data)	$R_1 = 0.1357$, $wR_2 = 0.2567$	$R_1 = 0.2558$, $wR_2 = 0.3223$
Largest difference peak and hole ($e\,Å^{-3}$)	1.209 and −1.202	2.181 and −1.037

Complex **1** is a tetranuclear Ni^{II} cluster with Ni_4O_4 cubane core [15–18, 23–28]. This complex is also the first compound in which the two faces of the Ni_4O_4 cubane were capped by DSMO molecules.

3.2.2. Structural description of 2.

The asymmetric unit of **2** contains two independent [$Cu_2Cl_2(H_2L)_2$] molecules with the same basic structure but different bond parameters (molecules **2a** and **2b**, see "Supplementary material"). The crystal structure of **2a** is shown in figure 2. Crystallographic data of **2** are listed in table 1, and selected bond lengths and angles of **2** are summarized in table 2. Complex **2a** is dinuclear consisting of two Cu^{II} ions, two $[H_2L]^-$, and two chlorides (μ_2-Cl). Each Cu^{II} is five-coordinate by one deprotonated phenoxide, one imine nitrogen, and one protonated alkoxide from one $[H_2L]^-$. The remaining two sites of Cu^{II} are occupied by two μ_2-Cl^-. The geometry around each Cu^{II} may be described as a distorted square pyramid with Cl1–Cu1–Cl2, Cl1–Cu1–O2, Cl1–Cu1–N1, and Cl1–Cu1–O1 angles of 94.38(14)°, 82.70(3)°, 105.4(4)°, and 96.10(4)°, respectively.

The {Cu1(μ-Cl1)(μ-Cl2)Cu2} motif is asymmetric with Cu1–Cl1, Cu1–Cl2, Cu2–Cl1, and Cu2–Cl2 bond lengths being 2.726(4), 2.261(4), 2.265(4), and 2.709(4) Å, respectively.

Each $[H_2L]^-$ functions as a 1.1110 ligand (Harrison notation [22]), forming a six-membered CuOCCCN chelating ring and a five-membered CuNCCO chelating ring (scheme 1c).

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

1			
Ni1–O1	1.967(6)	Ni1–N1	1.968(6)
Ni1–O5	2.045(5)	Ni1–O11	2.053(5)
Ni1–O2	2.111(5)	Ni1–O13	2.243(7)
Ni1–Ni2	2.8053(15)	Ni2–O4	1.954(6)
Ni2–N2	1.986(7)	Ni2–O2	2.037(5)
Ni2–O8	2.043(5)	Ni2–O5	2.101(5)
Ni2–O13	2.573(8)	Ni3–O7	1.962(6)
Ni3–N3	1.986(7)	Ni3–O11	2.041(5)
Ni3–O2	2.054(5)	Ni3–O8	2.108(5)
Ni3–O14	2.384(7)	Ni3–Ni4	2.8093(14)
Ni4–O10	1.951(6)	Ni4–N4	1.977(6)
Ni4–O8	2.058(5)	Ni4–O5	2.066(5)
Ni4–O11	2.096(5)	Ni4–O14	2.389(7)
Ni2–O2–Ni3	101.1(2)	Ni2–O2–Ni1	85.09(18)
Ni3–O2–Ni1	99.8(2)	Ni1–O5–Ni4	100.5(2)
Ni1–O5–Ni2	85.14(17)	Ni4–O5–Ni2	99.4(2)
Ni2–O8–Ni4	101.6(2)	Ni2–O8–Ni3	99.1(2)
Ni4–O8–Ni3	84.79(18)	Ni3–O11–Ni1	102.2(2)
Ni3–O11–Ni4	85.52(19)	Ni1–O13–Ni2	70.86(18)
2			
Cu1–O1	1.883(10)	Cu1–Cl2	2.261(4)
Cu1–N1	1.916(12)	Cu1–Cl1	2.726(4)
Cu1–O2	2.024(10)	Cu2–O4	1.871(11)
Cu2–N2	1.930(10)	Cu2–Cl2	2.709(4)
Cu2–O5	1.965(10)	Cu2–Cl1	2.265(4)
Cl2–Cu1–Cl1	94.38(14)	N1–Cu1–Cl2	158.6(4)
N1–Cu1–Cl1	105.4(4)	O1–Cu1–Cl2	92.4(3)
O1–Cu1–O2	175.1(4)	O2–Cu1–Cl1	82.7(3)
O2–Cu1–Cl2	92.4(3)	Cu2–Cl1–Cu1	83.88(13)
O1–Cu1–N1	93.6(6)	O1–Cu1–Cl1	96.1(4)
N1–Cu1–O2	82.3(5)	O5–Cu2–Cl1	89.5(3)
O4–Cu2–Cl1	93.5(3)	O4–Cu2–Cl2	91.2(3)
O4–Cu2–O5	175.7(4)	N2–Cu2–Cl2	98.2(3)
Cl1–Cu2–Cl2	94.76(14)	O5–Cu2–Cl2	91.7(3)
N2–Cu2–Cl1	165.0(4)	N2–Cu2–O5	82.5(5)
Cu1–Cl2–Cu2	84.34(14)		

Complex **2** joins a large family of dinuclear copper complexes with μ-Cl bridges [29–32].

3.3. *TG-DTG analyses*

TG-DTG analyses were carried out on **1** and **2**. The TG curve of **1** displays two weight losses. The first is 18.55% from 114.9°C to 269.0°C, corresponding to removal of one water and three DMSO molecules (theoretical value is 18.86%). The second loss occurs from 269.0°C to 999.7°C, with weight loss of 71.06%, assigned to loss of one DMSO and four $[HL]^{2-}$.

Two steps of weight loss were observed for **2** of 5.57% and 45.75% from 20.0°C to 155.9°C and 155.9°C to 915.0°C, respectively. The former may result from removal of two methanols (calculated, 5.85%), while the latter is consistent with the removal of three $[H_2L]^-$ ligands (calculated, 45.77%).

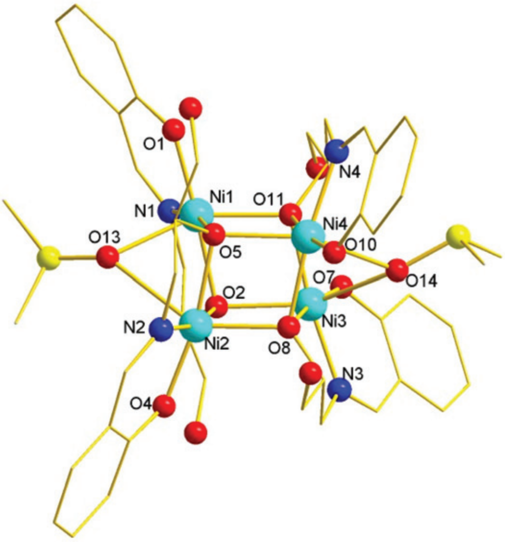


Figure 1. Molecule structure of **1**; hydrogens and solvated molecules have been omitted for clarity.

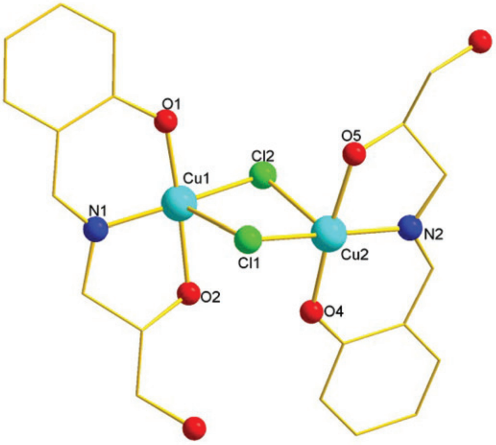


Figure 2. Molecule structure of **2a**; hydrogens and solvated molecules have been omitted for clarity.

4. Conclusion

A tetranuclear cubane-like Ni_4O_4 complex and a dinuclear μ_2-Cl bridged Cu^{II} complex have been prepared and structurally characterized. Two of the six faces of Ni_4O_4 of **1** were capped by DMSO. Complex **1** is the first compound in which two faces of Ni_4O_4 cubane were capped by DMSO. Complex **2** contains μ_2-Cl bridged dinuclear core with each Cu^{II} ion exhibiting pseudo square pyramidal geometry. Application of DMSO to other metal cubane clusters is underway and will be reported in due course.

Supplementary material

Figures S1 and S2 display the crystal structures for **1** and **2**. Tables S1–S4 give the crystallographic data and bond lengths and angles for **1** and **2**. Figures S3 and S4 give the IR spectra for **1** and **2**. Figures S5 and S6 give the TG-DTG analyses for **1** and **2**. Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 657372 for **1** and 628114 for **2**. These data can be obtained free of charge from CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44 1223336033; E-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

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