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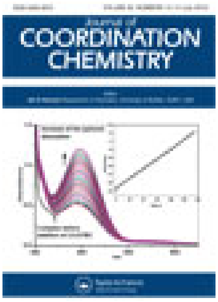

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A binuclear Cu(II) complex of a Schiff-base type polyhydroxyl ligand with asymmetrical phenoxo and methoxo bridges: crystal structure and magnetic properties

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A binuclear Cu(II) complex of a Schiff-base type polyhydroxyl ligand with asymmetrical phenoxo and methoxo bridges: crystal structure and magnetic properties

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$[Cu_2(H_4L)(OCH_3)(H_2O)]\cdot 2MeOH$ (**1**) has been synthesized from the reaction of 2-hydroxy-5-methylisophthalaldehyde, tris(hydroxymethyl)aminomethane and copper(II) perchlorate in methanol with the Schiff-base polyhydroxyl ligand $(H_4L)^{3-}$ formed *in situ*. Single-crystal X-ray structure analysis of **1** reveals a binuclear structure with Cu(II) asymmetrically bridged by a methoxo and a phenoxo group with Cu–O–Cu bridging angles of 102.93(7)° and 100.17(7)°, respectively. The binuclear Cu(II) moieties utilize the coordinated and noncoordinated alkoxyl groups for formation of intermolecular hydrogen bonds, thus affording a 3-D supramolecular structure. Variable temperature magnetic measurements reveal strong antiferromagnetic coupling in **1** with the $2J$-value of $-729\,\mathrm{cm}^{-1}$, which was mediated cooperatively by the methoxo and phenoxo bridges. Magneto-structural correlations are discussed based on the crystal structure.

Keywords: Crystal structure; Cu(II) complex; O-Bridge; Magnetic properties

1. Introduction

Attention has been focused on polynuclear Cu(II) complexes and supramolecular Cu(II) coordination assemblies in view of their importance in metalloproteins, as metalloenzyme mimics [1–3], and for applications in catalysis [4–6], chemical sensors [7, 8], magnetochemistry [9–15], and pharmaceutical materials [16–19].

Design and synthesis of ligands is essential for developing Cu(II) complexes. Hydroxyl-rich polydentate ligands have been demonstrated to form Cu(II) complexes with structural diversity. Hydroxyl groups may coordinate in the neutral or deprotonated form. For the latter, it may coordinate terminal, or bridge two or three metal centers to form polynuclear structures. Thus, a variety of binuclear and polynuclear Cu(II) complexes with hydroxo [20–23], alkoxo [24–26], and phenoxo [27–29] bridges have been synthesized and magneto-structural correlations have

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been proposed. In addition to bridging abilities, hydroxyl groups may also be involved in forming intermolecular hydrogen bonds to afford supramolecular assemblies. We have reported supramolecular coordination assemblies and magnetic properties of a variety of binuclear, tetranuclear, and hexanuclear metal complexes synthesized from bidentate, tridentate, and multidentate hydroxyl-rich ligands [30–34].

Based on this background, in this work we synthesize a binuclear Cu(II) complex, $[Cu_2(H_4L)(OCH_3)(H_2O)]\cdot 2MeOH$ (**1**), wherein the polydentate hydroxyl-rich ligand $(H_4L)^{3-}$ was formed *in situ* by reaction of a dialdehyde and an amino compound tris(hydroxymethyl)aminomethane (Tris). Complex **1** has a binuclear structure with the Cu(II) centers asymmetrically bridged by phenoxo and methoxo groups. The binuclear moieties are linked by hydrogen bonds to form a 3-D supramolecular assembly. Antiferromagnetic interactions between Cu(II) centers are observed with 2J of $-729\,cm^{-1}$.

2. Experimental

2.1. *Safety notes and materials*

Perchlorate salts of metal complexes with organic ligands are potentially explosive. Only a small amount should be used and should be handled with extreme care. All chemicals were of reagent grade quality and used as received from commercial sources. 2-Hydroxy-5-methylisophthalaldehyde was synthesized by a procedure modified from that reported previously [35].

2.2. *Preparation of $[Cu_2(H_4L)(OCH_3)(H_2O)]\cdot 2MeOH$ (1)*

To a methanol (100 mL) solution of 2-hydroxy-5-methylisophthalaldehyde (66 mg, 0.4 mmol) was added tris(hydroxymethyl)aminomethane (97 mg, 0.8 mmol). After stirring for 2 h, $Cu(ClO_4)_2\cdot 6H_2O$ (296.4 mg, 0.8 mmol) was added. Then NaOH (0.4 mL, 4 mol L^{-1}, 1.6 mmol) was added dropwise with stirring. After refluxing for 4 h, the resulting solution was concentrated *in vacuo* to 10 mL, filtered, and left undisturbed at room temperature for several days, yielding plate-like dark green single crystals of **1** suitable for X-ray diffraction analysis. Yield: 0.093 g, 39%. Anal. Calcd for $(C_{20}H_{36}Cu_2N_2O_{11})$ (%): C, 39.53; H, 5.97; N, 4.61. Found: C, 39.32; H, 6.24; N, 4.91. IR (KBr, cm^{-1}): 3376(vs), 2920(s), 2850(s), 1638(vs), 1556(vs), 1451(m), 1395(w), 1326(m), 1050(vs), 649(m).

2.3. *Crystallographic data collection and refinement of the structure*

Crystals suitable for X-ray analyses were grown by slow evaporation of a methanol solution of the complex. X-ray diffraction data were collected on a Bruker-AXS APEX utilizing Mo-Kα radiation ($\lambda = 0.71073$ Å). The structure was solved by direct methods and refined with full-matrix least-squares technique. Anisotropic thermal parameters were applied to all nonhydrogen atoms. All of the hydrogen atoms in the structure are located from the difference electron density map and constrained to ideal positions in

Table 1. Crystallographic data and structure refinement summary for **1**.

Empirical formula	$C_{20}H_{36}Cu_2N_2O_{11}$
Formula weight	607.59
Space group	$P2_1/n$
Temperature (K)	293(2)
Crystal system	Monoclinic
Unit cell dimensions (Å, °)	
a	12.0287(9)
b	12.6483(10)
c	16.8129(13)
α	90
β	91.702(1)
γ	90
Volume (Å^3), Z	2556.8(3), 4
Calculated density (Mg m^{-3})	1.578
Absorption coefficient	1.722
Crystal size (mm^3)	0.32 × 0.30 × 0.30
No. of reflections [$I > 2\sigma(I)$]	4474
Parameters	298
Goodness-of-fit on F^2	1.054
Final R_1[a] [$I > 2\sigma(I)$]	0.0505
wR_2[b] (all data)	0.1599

[a]$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; [b]$wR_2 = [\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]]^{1/2}$.

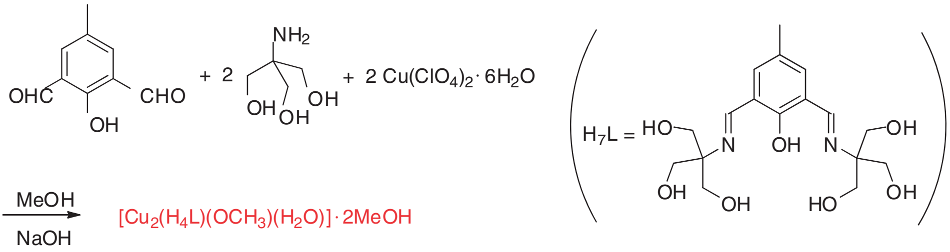
Table 2. Selected bond lengths (Å) and angles (°) for **1**.

Cu(1)–O(4)	1.901(3)	Cu(2)–O(8)–Cu(1)	102.93(7)
Cu(1)–O(8)	1.903(3)	O(5)–Cu(2)–O(8)	102.05(11)
Cu(1)–N(1)	1.928(2)	O(5)–Cu(2)–N(2)	87.81(9)
Cu(1)–O(1)	1.946(2)	O(8)–Cu(2)–N(2)	167.47(10)
Cu(2)–O(5)	1.891(3)	O(5)–Cu(2)–O(1)	179.61(10)
Cu(2)–O(8)	1.911(3)	O(8)–Cu(2)–O(1)	78.29(9)
Cu(2)–N(2)	1.931(2)	N(2)–Cu(2)–O(1)	91.83(7)
Cu(2)–O(1)	1.944(2)	O(8)–Cu(1)–O(1)	78.43(9)
Cu(2)–O(10)	2.612(1)	N(1)–Cu(1)–O(1)	91.84(7)
Cu(1)···Cu(2)	2.984(1)	Cu(2)–O(1)–Cu(1)	100.17(7)
O(4)–Cu(1)–O(8)	102.23(11)	O(5)–Cu(2)–O(10)	93.43(2)
O(4)–Cu(1)–N(1)	87.50(9)	N(2)–Cu(2)–O(10)	92.64(2)
O(8)–Cu(1)–N(1)	170.27(9)	O(1)–Cu(2)–O(10)	86.73(1)
O(4)–Cu(1)–O(1)	179.34(9)	O(8)–Cu(2)–O(10)	94.44(2)

the refinement procedure. Absorption correction was performed using SADABS [36]. All calculations were performed using SHELX-97 software package [37]. Crystal data and experimental details for the crystal are summarized in table 1 and selected bond lengths and angles are given in table 2.

2.4. *Other physical measurements*

FT-IR spectra were recorded from 400 cm^{-1} to 4000 cm^{-1} on a Thermo Electron Avatar 380 FT-IR instrument (KBr discs). Elemental analyses were carried out with an Elementar Vario EL-III analyzer. Fluorescence measurements were made on a Varian Cary Eclipse fluorescence spectrophotometer. Variable temperature magnetic susceptibility data were collected with a Quantum Design MPMS7 SQUID magnetometer



Scheme 1. Synthesis of **1** and the molecular structure of H_7L.

between 2 and 300 K. The data were corrected for diamagnetism of the sample holder and for diamagnetic contributions with Pascal's constants; a value of $60 \times 10^{-6}\,cm^3\,mol^{-1}$ was used for the temperature-independent paramagnetism of Cu(II).

3. Results and discussion

3.1. *Synthesis and spectral characterization*

In the preparation of **1**, the Schiff-base type ligand was formed *in situ* by condensation of the dialdehyde and the amino compound in MeOH (scheme 1). Similar reactions have been reported previously [38, 39]. The molecule contains phenol and alkoxyl groups. To promote the deprotonation and subsequent coordination of these groups, aqueous NaOH solution was added. Upon addition of NaOH, the reaction solution turned from light green to dark green immediately, indicating deprotonation, and coordination of the phenol. The MeOH solution was concentrated and left for slow evaporation to obtain single crystals. The presence of the C=N bonds in the final complex can be clearly observed by the strong absorption at $1638\,cm^{-1}$ in the infrared (IR) spectrum.

3.2. *Description of the structure*

To understand the molecular structure of **1**, a single crystal was analyzed by X-ray diffraction measurements. Selected bond lengths and angles for **1** are listed in table 2. In general, the crystal structure of **1** consists of a binuclear Cu(II) structure with the Cu(II) centers asymmetrically bridged by phenoxo and methoxo groups.

H_7L contains six alkoxyl groups, two imino nitrogen atoms, and one phenol. Thus, it is potentially a nonadentate ligand. In **1**, the ligand coordinates pentadentate (figure 1), utilizing the deprotonated phenoxo, the two imino nitrogen atoms, and two deprotonated alkoxo, with four other alkoxyl not coordinated. Thus, H_7L coordinates in a trianionic form of $(H_4L)^{3-}$. The coordination geometry of Cu(1) can be described as square planar, with one phenoxo oxygen, one imino nitrogen, and one alkoxo oxygen from $(H_4L)^{3-}$ in addition to one methoxide. Cu(2) is equatorially coordinated to O(1), N(2), and O(5) of $(H_4L)^{3-}$, in addition to one methoxo, and axially coordinated to one water molecule. The final coordination sphere of Cu(2) can be described as square pyramidal. Cu–N and Cu–O distances are similar to those observed in a similar

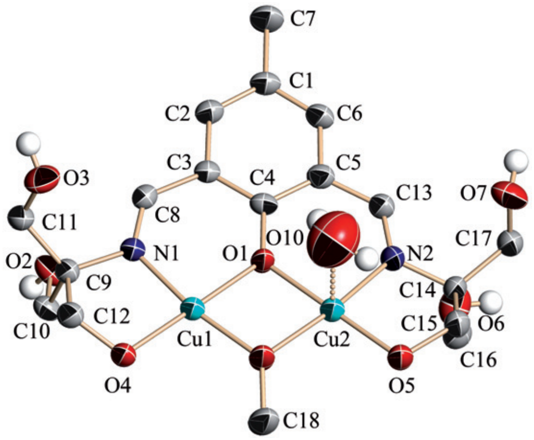


Figure 1. The molecular structure of **1** showing 50% probability displacement ellipsoids and the atom labels. All hydrogen atoms attached to carbon and solvent molecules are omitted for clarity.

Table 3. Hydrogen bond lengths (Å) and angles (°) for **1**.

D–H···A	*d*(H···A)	∠DHA	*d*(D···A)
C13–H13···O2^{i}	2.35	156	3.220(5)
C2–H2···O6^{i}	2.49	149	3.317(5)
O2–H2A···O4^{ii}	1.87	168	2.673(5)
O3^{iii} –H3^{iii}···O5	1.86	173	2.678(5)

Symmetry operations: i: $-x$, $-y+1$, $-z$; ii: $-x-1/2$, $y+1/2$, $-z+1/2$; iii: $x+1/2$, $-y-1/2$, $z+1/2$.

binuclear Cu(II) complex of H_7L, where two Cu(II) atoms are bridged by one phenoxo and one azide, with the Cu–O–Cu bridging angle of 101.61(8)°, larger than the corresponding value observed in **1** [38]. The Cu(1)···O(11) and Cu(2)···O(11) distances are 3.3 Å and 4.6 Å, respectively, significantly longer than normal Cu–O bond lengths [40, 41], indicating that they are nonbonded. The Cu(1)···Cu(2) distance of 2.984 Å lies in the normal range for this type of binuclear copper(II) complex [42, 43]. Two Cu(II) centers are bridged by the phenoxo of the ligand, and the positive charge of Cu(II) is further balanced by a bridging methoxide, which originates from deprotonation of MeOH under the alkaline synthetic condition. Finally, a binuclear Cu(II) structure was formed with Cu(II) centers asymmetrically bridged by phenoxo and methoxo. The Cu–O–Cu bridging angles are 100.17(7)° and 102.93(7)°, respectively.

Deprotonated and neutral alkoxy groups coexist in **1**, involved in intermolecular hydrogen bonds to afford a supramolecule. A hydrogen-bonded dimer (table 3) was formed by quadruple C–H···O hydrogen bonds between noncoordinated alkoxyl and the phenyl carbon hydrogen atoms (figure 2a), with H···O lengths of 2.35 Å and 2.49 Å, and C–H···O angles of 156° and 149°, respectively. In addition, binuclear moieties are also linked by multiple intermolecular hydrogen bonds between coordinated alkoxo groups and the noncoordinated alkoxy groups with O···H lengths of 1.86 Å and 1.87 Å, and O–H···O angles of 173° and 168°, respectively (figure 2b). Finally, the binuclear moieties are linked by these multiple H-bonds to afford a 3-D supramolecular structure (figure 3).

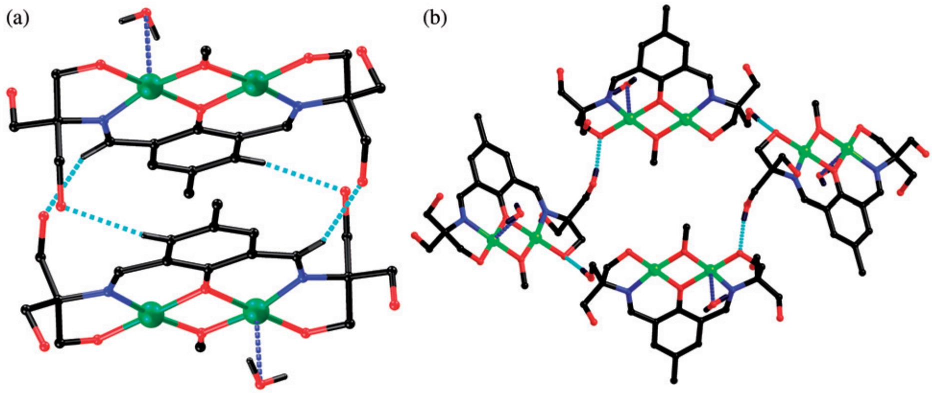


Figure 2. (a) The dimeric structure linked through hydrogen bonds between the alkoxyl groups and the C–H hydrogen atoms; (b) a tetramer approximately along the *ab* plane linked by intermolecular hydrogen bonds between coordinated and noncoordinated alkoxyl groups.

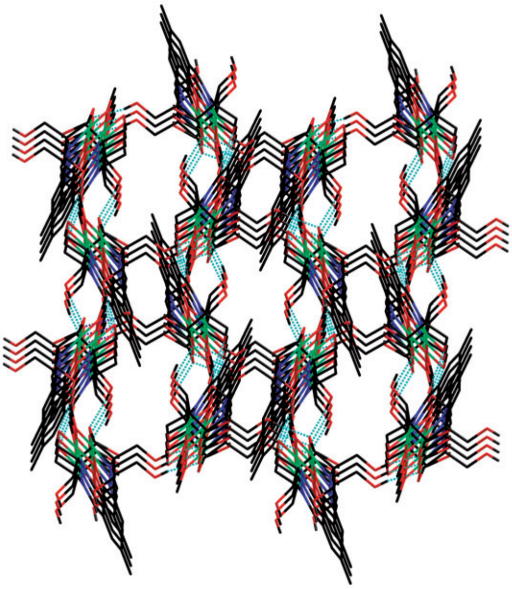


Figure 3. View of the 3-D network of **1** approximately along the *c*-axis.

3.3. *Magnetic properties*

In the crystal structure of **1**, the Cu(II) centers are asymmetrically bridged by a methoxo and a phenoxo group, which may mediate magnetic interactions between Cu(II) centers. To understand the magnetic interactions, variable temperature magnetic susceptibilities for **1** were measured, and the plot of $\chi_m T$ *versus* T is illustrated in figure 4.

The $\chi_m T$ product at room temperature is 0.22 $cm^3 mol^{-1}$ K. Gradual decrease of this product is observed as the temperature decreases (figure 4), reaching a value of

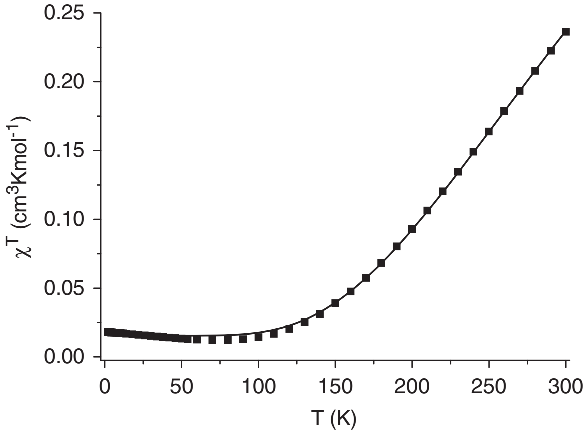


Figure 4. Plot of $\chi_m T$ products *vs*. temperature for **1**; the solid line represents the best calculated fit (see text for the fitted parameters).

0.018 $cm^3\,mol^{-1}$ K at 2 K, indicative of a strong antiferromagnetic interaction. The data were fitted to the Bleaney–Bowers equation (equation (1)) [44]:

$$\chi_m T = (1-\rho)\cdot\frac{2Ng^2\mu_B^2}{k}\cdot\frac{1}{3+e^{-2J/kT}}+\rho\cdot\frac{N\beta^2 g^2}{2k}, \tag{1}$$

where all the symbols have their usual meanings. The best fit gave $\rho = 0.019(1)$, $g = 2.080(10)$, $2J = -729(6)\,cm^{-1}$, and $R = 3.6\times10^{-6}$. Linear correlations have been found between Cu–O–Cu bridging angle (ϕ) and spin coupling (J) between the metal centers of binuclear copper(II) complexes equatorially bridged by pairs of hydroxide [20, 21] or alkoxide [24] groups. A similar linear correlation has also been proposed for binuclear copper(II) complexes bridged by a pair of phenoxides [27]. In these cases, ferromagnetism is associated with smaller bridging angles, and antiferromagnetism with larger ones. In **1**, the Cu(II) centers are asymmetrically bridged by phenoxo and methoxo O atom groups with large Cu–O–Cu bridging angles of 100.17(7)° and 102.93(7)°, and the average bridging angle ϕ of 101.55°. The strong antiferromagnetic interaction with $2J$-value of $-729(6)\,cm^{-1}$ associated with these large bridging angles for **1** is consistent with reported results [20–27]. We utilize the average bridging angle of 101.55° for further magneto-structural correlation analyses. Thus, the expected $2J$-value calculated from the equation ($2J = -82.1\phi + 7857\,cm^{-1}$) [24] proposed for copper(II) complexes bridged by a pair of alkoxides is $-480\,cm^{-1}$, and the corresponding value obtained from the equation ($2J = -31.95\phi + 2462\,cm^{-1}$) [27] proposed for the copper(II) complexes bridged by a pair of phenoxides is $-783\,cm^{-1}$. It is obvious that the $2J$-value of $-729\,cm^{-1}$ obtained for **1** lies in between these two values, indicating that the strong antiferromagnetic interactions between the Cu(II) centers in **1** are cooperatively propagated by the phenoxo and the alkoxo bridges.

4. Conclusion

We report the synthesis of a binuclear Cu(II) complex, $[Cu_2(H_4L)(OCH_3)(H_2O)]\cdot 2MeOH$ (**1**), of a potentially nonadentate hydroxyl-rich ligand H_7L, which was generated *in situ* from the reaction of 2-hydroxy-5-methylisophthalaldehyde, and tris(hydroxymethyl)aminomethane. Complex **1** has a binuclear structure with $(H_4L)^{3-}$ coordinated pentadentate and the Cu(II) ions asymmetrically bridged by methoxo and phenoxo groups with Cu–O–Cu bridging angles of 102.93(7)° and 100.17(7)°, respectively. The binuclear Cu(II) moieties utilize coordinated and noncoordinated alkoxyl groups for formation of intermolecular hydrogen bonds, affording a 3-D supramolecular structure. Variable temperature magnetic measurements reveal strong antiferromagnetic coupling, which was cooperatively mediated by methoxo and phenoxo bridges with large bridging angles. This work provides further insight into the design and synthesis of relevant binuclear and polynuclear Cu(II) complexes with supramolecular structures and interesting magnetic properties.

Supplementary material

Crystallographic data (excluding structure factors) for the structure reported in this article have been deposited with the Cambridge Crystallographic Center, CCDC No. 856715. Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336 033, E-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk

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