

# 四核铜化合物 $(\text{Cu}_4\text{L}_4)(\text{CH}_3\text{CN})(\text{H}_2\text{L}=(R)\text{-1-(1-羟基正丁烷-2-氨基)萘-2-醇})$ 的合成、晶体结构及磁性质

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**摘要:** 采用具有手性的席夫碱配体,  $(R)\text{-1-(1-羟基正丁烷-2-氨基)萘-2-醇}$  ( $\text{H}_2\text{L}$ ), 和醋酸铜反应得到四核铜的化合物 $(\text{Cu}_4\text{L}_4)(\text{CH}_3\text{CN})$ 。用 X-射线衍射对化合物的晶体结构进行了测定, 结果表明此化合物含有 4 个铜和 4 个配体, 铜通过配位作用和 4 个氧原子形成类立方烷中心。磁性测定表明此化合物中金属离子之间有弱的反铁磁相互耦合作用。

**关键词:** 四核铜; 配合物; 磁性; 手性席夫碱配体

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## Synthesis, Crystal Structure and Magnetic Properties of a Tetranuclear Copper(II) Complex

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**Abstract:** An antiferromagnetic complex based on copper(II) and chiral Schiff-base ligand  $(R)\text{-1-}((1\text{-hydroxybutan-2-ylimino)methyl)naphthalen-2-ol}$  ( $\text{H}_2\text{L}$ ) with formula of  $(\text{Cu}_4\text{L}_4)(\text{CH}_3\text{CN})$  was synthesized and structurally characterized. Single-crystal structure shows that the complex consists of an inorganic core formed by four Cu(II) ions and four oxygen atoms from four ligands respectively. Magnetic measurements show that a weak antiferromagnetic coupling occurs in the complex. CCDC: 620500.

**Key words:** tetranuclear copper(II); coordination complex; magnetic behavior; chiral schiff-base ligand

## 0 Introduction

There has been continuous interest in high-nuclearity transition-metal complexes with aims at the elucidation of the fundamental science of magnetic interactions and magneto-structural correlations in molecular systems<sup>[1-5]</sup>. In particular, much attention has been given to copper because of the central role it plays in biology<sup>[6]</sup>. On the other hand, the flexibility of the coordination sphere around Cu(II), with varied

distortions due to a pseudo-Jahn-Teller effect, in combination with steric and crystal packing forces, leads to its tremendous structural diversity. Small changes in structure can have far reaching effects on the magnetic properties of these systems<sup>[7,8]</sup>. In addition, since Rikken and Raupach observed the weak magneto-chiral dichroism (MChD) effects in a chiral paramagnetic material<sup>[9]</sup>, the investigation of chiral magnets combining magnetism and optical activity has become an active research topic for their important potential application as multif-

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unctional materials<sup>[10-13]</sup>. The present work stems from our interest in exploring this chemistry by using the multidentate chiral Schiff-base ligand ( $R$ )-1-((1-hydroxybutan-2-ylimino) methyl)naphthalen-2-ol ( $\text{H}_2\text{L}$ ).

## 1 Experimental

### 1.1 Materials and physical measurement

All chemicals were of reagent grade quality obtained from commercial sources and used without further purification. Elemental analyses (C, H and N) were carried out on a Perkin-Elmer 240C analytical instrument. IR spectra were recorded on a VECTOR 22 Bruker spectrophotometer with KBr pellets in the  $4000\sim 400\text{ cm}^{-1}$  regions,  $^1\text{H}$  NMR spectra on DRX500 Bruker spectrometer at 298K with TMS as an internal reference. Electrospray mass spectrum was carried out on a LCQ system (Finnigan MAT, USA) with methanol as mobile phase. Magnetic susceptibility data on crushed single crystals were collected over the temperature range 20~300 K using a Quantum Design MPMS-XL super-conducting quantum interference device (SQUID) magnetometer, and the experimental data were corrected for diamagnetism of the constituent atoms estimated from Pascal's constants.

### 1.2 Synthesis of the ligand $\text{H}_2\text{L}$

$R$ -(-)-2-Amino-1-butanol (2.8 g, 31 mmol) was slowly added to a solution of 2-Hydroxy-1-naphthaldehyde (5.445 g, 26 mmol) in THF (30 mL). The reaction mixture was refluxed for about 1h, and the solvent was subsequently removed under reduced pressure, and the dark-brown result was obtained. Yield: 6.0g, 93%. Anal. Calc. (%) for  $\text{H}_2\text{L}$ ,  $\text{C}_{15}\text{H}_{17}\text{NO}_2$  (243.30): C, 74.05; H, 7.04; N, 5.76. Found (%): C, 74.21; H, 6.78; N, 5.83. IR (solid KBr pellet): 3 166 (m), 2 964 (m), 2 928 (m), 2 856 (m), 1 633 (s), 1 545 (m), 1 518 (m), 1 493 (m), 1 461 (m), 1 404 (m), 1 348 (s), 1 271 (m), 1 213 (m), 1 188 (m), 1 142 (w), 1 086 (m), 1 063 (m),

999 (w), 868 (w), 839 (m), 754 (s), 496 (w).

### 1.3 Synthesis of the title complex

A methanolic solution (10 mL) of  $\text{Et}_3\text{N}$  (0.6 g, 5.8 mmol) was added to a methanolic solution (30 mL) containing  $\text{H}_2\text{L}$  (0.7 g, 2.9 mmol) and  $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$  (0.58 g, 2.9 mmol). A green precipitate immediately formed. The result mixture was stirred overnight at room temperature and was filtered off. The green block-shaped crystals were obtained recrystallization of the above filtrate from  $\text{CH}_3\text{CN}$  in *ca.* 80% yield after one day. Anal. calc. (%) for  $\text{C}_{62}\text{H}_{63}\text{Cu}_4\text{N}_5\text{O}_8$  (1260.33): C, 59.08; H, 5.04; N, 5.56. Found (%): C, 59.19; H, 5.25; N, 5.40. IR (solid KBr pellet): 3 425 (w), 2 960 (w), 2 926 (w), 2 867 (w), 1 620 (s), 1 539 (s), 1 507 (m), 1 455 (m), 1 434 (m), 1 394 (m), 1 356 (m), 1 184 (m), 1 040 (w), 987 (w), 934 (w), 828 (m), 746 (m), 588 (w), 555 (w), 493 (w).

### 1.4 Crystal structure determination

Suitable crystal was selected for single-crystal X-ray diffraction structural analysis and the data were collected on a Siemens SMART-CCD diffractometer with graphite-monochromatized Mo  $K\alpha$  radiation  $\lambda = 0.071\,073\text{ nm}$ , using the SMART and SAINT programs<sup>[14]</sup>. 45 frames of data were collected at 298K with an oscillation range of  $1^\circ/\text{frame}$  and an exposure time of 10 s/frame. Indexing and unit cell refinement were based on all observed reflections from those 45 frames. The structures were solved by direct method and refined on  $F^2$  by full-matrix least-squares methods with SHELXTL version 5.1<sup>[15]</sup>. Anisotropic thermal parameters were refined for non-hydrogen atoms. Hydrogen atoms were localized in their calculation positions and refined by using the riding model. Parameters for data collection and refinement of the compound and the crystallographic data are summarized in Table 1.

CCDC: 620500.

Table 1 Crystal data and refinement parameters of complex

Formula	$\text{C}_{62}\text{H}_{63}\text{N}_5\text{O}_8\text{Cu}_4$	$Z$	2
Formula weight	1 260.33	$F(000)$	1300
Crystal system	Monoclinic	$\mu / \text{mm}^{-1}$	1.385
Space group	$P2_1$	No. reflection measured	15 926
$a / \text{nm}$	1.509 6(3)	No. unique reflections	9 054

Continued Table 1

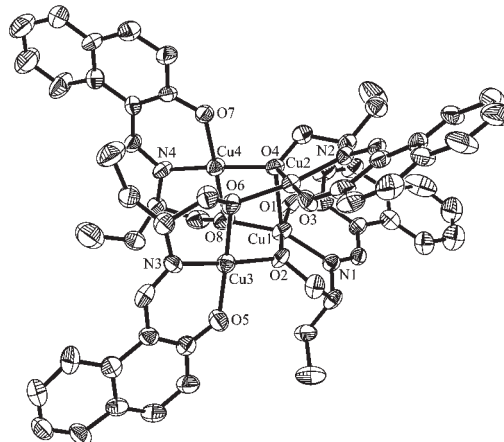
$b / \text{nm}$	1.443 2(3)	$R_{\text{int}}$	0.087 6
$c / \text{nm}$	1.598 6(4)	$R_1$	0.062 3
$\beta / (^\circ)$	115.155(4)	$wR_2$ (all data)	0.151
$V / \text{nm}^3$	3.1526(1)	Goodness-of-fit	0.989

## 2 Results and discussion

### 2.1 Crystal structure

Ligand  $\text{H}_2\text{L}$  was synthesized in 93% yield by the condensation of 2-Hydroxy-1-naphthaldehyde and  $R(-)-2$ -Amino-1-butanol. The reaction of  $\text{H}_2\text{L}$  and  $\text{Cu}(\text{OAc})_2$  in methanol afforded a green complex of with formula  $(\text{Cu}_4\text{L}_4)(\text{CH}_3\text{CN})$ . The crystal structure revealed a molecule consisting of an inorganic core formed by four  $\text{Cu}(\text{II})$  ions and four oxygen atoms form four ligands respectively (Fig.1). A disordered  $\text{CH}_3\text{CN}$  molecule is also found to be associated to the molecular unit. The naphthalene rings point outward from the core in four directions. The copper center  $\text{Cu}(1)$  is 4 + 1 coordinated with two alkoxo oxygen atoms  $\text{O}(1)$ ,  $\text{O}(2)$ , one amine nitrogen atom  $\text{N}(1)$  from a same  $\text{H}_2\text{L}$  ligand and one alkoxo oxygen atom  $\text{O}(8)$  from another ligand forming the basal plane and the weak coordinated alkoxo oxygen atom  $\text{O}(4)$  from the other organic ligand occupies the axial position [ $\text{Cu}(1)-\text{O}(4)=0.242\ 3(7)\ \text{nm}$ ]. The pentacoordination geometry might be described as a distorted square pyramidal with  $\tau=0.24$  [16]. Other copper centers ( $\text{Cu}(2)$ ,  $\text{Cu}(3)$ ,  $\text{Cu}(4)$ ) are weakly bonded to an alkoxo oxygen atom from a neighboring  $\text{H}_2\text{L}$  ligand [ $\text{Cu}-\text{O}=0.256\ 1\sim 0.284\ 1\ \text{nm}$ ], which might imply that the metal-coordination sphere should be regarded as predominantly square planar. However, in the structure under discussion, the alkoxo oxygen atoms bridge three copper atoms rather than two: each of the oxygen atoms  $\text{O}(2)$ ,  $\text{O}(4)$ ,  $\text{O}(6)$ , and  $\text{O}(8)$ , besides being bound to two copper atoms, are also weakly coordinated to a third copper atom ( $\text{Cu}(2)$ ,  $\text{Cu}(1)$ ,  $\text{Cu}(4)$ , and  $\text{Cu}(3)$ , respectively). This bridge mode results in the formation of a  $\text{Cu}_4\text{O}_4$  cluster that is made up of a copper tetrahedron interlocked with an oxygen tetrahedron and that is closely related to the cubanetype structures. Meanwhile, because of the above weak coordination interaction, the  $\text{Cu}_4\text{O}_4$  unit should be also

considered as an open cubane-like core or a boat-like conformation. The  $\text{Cu}\cdots\text{Cu}$  separations are range from  $0.314\ 7(2)\ \text{nm}$  [ $\text{Cu}(1)\cdots\text{Cu}(4)$ ] to  $0.346\ 0(2)\ \text{nm}$  [ $\text{Cu}(3)\cdots\text{Cu}(4)$ ].



Hydrogen atoms and lattice solvent molecule  $\text{CH}_3\text{CN}$  are omitted for clarity. Selected bond lengths (nm) and angles ( $^\circ$ ):

$\text{Cu}(1)-\text{O}(1)$ 0.189 1(7),	$\text{Cu}(1)-\text{O}(8)$ 0.193 1(8),
$\text{Cu}(1)-\text{O}(2)$ 0.193 9(7),	$\text{Cu}(1)-\text{N}(1)$ 0.194 6(8),
$\text{Cu}(1)-\text{O}(4)$ 0.242 3(7),	$\text{Cu}(2)-\text{O}(3)$ 0.185 6(7),
$\text{Cu}(2)-\text{N}(2)$ 0.191 9(8),	$\text{Cu}(2)-\text{O}(4)$ 0.193 4(7),
$\text{Cu}(2)-\text{O}(6)$ 0.195 6(6),	$\text{Cu}(3)-\text{O}(5)$ 0.188 4(8),
$\text{Cu}(3)-\text{N}(3)$ 0.191 6(8),	$\text{Cu}(3)-\text{O}(2)$ 0.192 5(6),
$\text{Cu}(3)-\text{O}(6)$ 0.192 9(7),	$\text{Cu}(4)-\text{O}(7)$ 0.189 8(7),
$\text{Cu}(4)-\text{N}(4)$ 0.191 2(9),	$\text{Cu}(4)-\text{O}(4)$ 0.192 5(7),
$\text{Cu}(4)-\text{O}(8)$ 0.194 8(7),	$\text{Cu}(1)\cdots\text{Cu}(2)$ 0.339 3(2),
$\text{Cu}(1)\cdots\text{Cu}(3)$ 0.330 9(2),	$\text{Cu}(1)\cdots\text{Cu}(4)$ 0.314 7(2),
$\text{Cu}(2)\cdots\text{Cu}(3)$ 0.321 5(2),	$\text{Cu}(2)\cdots\text{Cu}(4)$ 0.318 8(2),
$\text{Cu}(3)\cdots\text{Cu}(4)$ 0.346 0(2)	
$\text{Cu}(1)-\text{O}(2)-\text{Cu}(3)$ 117.8(3),	$\text{Cu}(2)-\text{O}(4)-\text{Cu}(4)$ 111.4(3),
$\text{Cu}(2)-\text{O}(6)-\text{Cu}(3)$ 111.6(3),	$\text{Cu}(1)-\text{O}(8)-\text{Cu}(4)$ 108.4(4)

Fig.1 ORTEP drawing of molecular structure of  $(\text{Cu}_4\text{L}_4)(\text{CH}_3\text{CN})$  with the atomic partially numbering scheme (30% displacement ellipsoids)

### 2.2 CD spectra characterization of the title complex

CD spectra of the title complex, as shown in Fig. 2, exhibits a positive Cotton effects at  $\lambda_{\text{max}}=675\ \text{nm}$  in the visible region, which can easily be assigned to the  $d-d$  transition, indicating that the chirality of ligand

leading to the formation of a single enantiomer of the polynuclear complex.

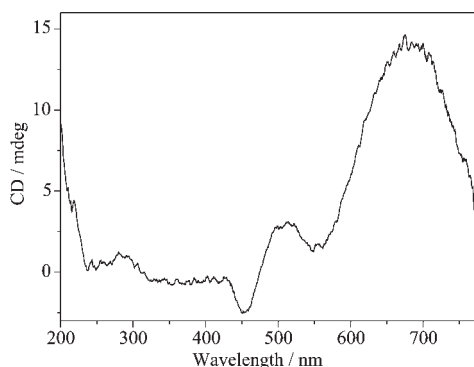


Fig.2 Circular dichroism spectra of the complex in KBr pellets

### 2.3 Magnetic susceptibility

Magnetic susceptibility measurement for the complex was obtained on the Quantum Design MPMS-XL7 SQUID magnetometer at temperatures range from 20 to 300 K. As shown in Fig.3, the  $\chi_M T$  value gradually decreases from 1.53 emu · K · mol<sup>-1</sup> at room temperature to 1.49 emu · K · mol<sup>-1</sup> at 120 K; then, it rapidly decreases to 1.14 emu · K · mol<sup>-1</sup> at 20 K. The  $\mu_{\text{eff}}$  value is 3.50  $\mu_B$  at 300 K and 3.03  $\mu_B$  at 20 K, which is marginally smaller than the magnetic moment of 4.90  $\mu_B$  for a molecule in the ground state with a total spin of 2 (spin-only value, assuming that the *g* factor is 2). All the magnetic data indicate that substantial antiferromagnetic coupling interactions among the Cu(II) spins in cubane rather than the magnetic moment of the Cu(II) cluster. Interestingly, some clos-

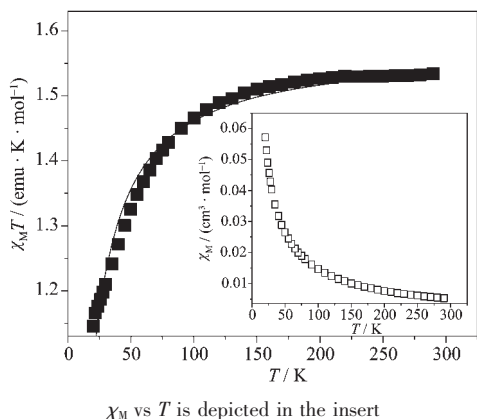


Fig.3 Temperature dependence of the magnetic susceptibility in the form of  $\chi_M T$  vs *T* at 2 kOe; the solid line represents the best-fit curve from 300 to 20 K

ely related compounds, [Cu<sub>4</sub>(NH<sub>3</sub>)<sub>4</sub>(HL)<sub>4</sub>][CdBr<sub>4</sub>]Br<sub>2</sub> · 3dmf · H<sub>2</sub>O (H<sub>2</sub>L=diethanolamine), [Cu<sub>4</sub>(hpda)<sub>4</sub>][ClO<sub>4</sub>]<sub>4</sub> · H<sub>2</sub>O (Hhpda=N-(2-hydroxyethyl)-1,3-propane-diamine), [Cu<sub>4</sub>(HL')<sub>4</sub>], [Cu<sub>4</sub>L<sub>2</sub>(OH)<sub>2</sub>], and [Cu<sub>4</sub>L<sub>2</sub>(OMe)<sub>2</sub>] (H<sub>3</sub>L'=N, N'-(2-hydroxypropane-1,3-diyl)bis(acetylacetoneimine) and H<sub>3</sub>L=N'-(2-hydroxypropane-1,3-diyl)-bis(salicylaldehydeimine)), exhibited very different magnetic behavior<sup>[6,8,17]</sup>, with the  $\mu_{\text{eff}}$  value increasing as the temperature decreased.

Because the inorganic cores are well shielded by the organic ligands, it is very impossible that they include significant intermolecular interactions. The structure of the tetranuclear Cu(II) core shows that the copper atoms are weakly bonded to an alkoxo oxygen atom [Cu-O=0.242 4~0.284 1 nm], hence there is exchange pathway between each pair of copper atoms. Besides, the long Cu···Cu separation falls in the range 0.315~0.346 nm. Based on these structural and magnetic data, we usually assume some degree of symmetry so that certain exchange integrals are equal and can be described as in Fig.4 with *J* (*J*=*J*<sub>13</sub>=*J*<sub>14</sub>=*J*<sub>23</sub>=*J*<sub>24</sub>) and *J'* (*J'*=*J*<sub>12</sub>=*J*<sub>34</sub>). The magnetic data were analyzed using the Heisenberg Hamiltonian<sup>[18]</sup> and the magnetic susceptibility can then be expressed as<sup>[19]</sup>:

$$H = -2[J(S_1S_3 + S_1S_4 + S_2S_3 + S_2S_4) + J'(S_1S_2 + S_3S_4)]$$

$$\chi_M = \frac{2N\beta^2 g^2}{kT} \times \frac{A}{B} (1 - \rho) + \frac{N\beta^2 g^2}{kT} \times \rho + N_\alpha$$

$$A = 2\exp(2J'/kT) + \exp[(4J' - 2J)/kT] + 5\exp[(4J' + 2J)/kT]$$

$$B = 1 + 6\exp(2J'/kT) + \exp[(4J' - 4J)/kT] + 3\exp[(4J' - 2J)/kT] + 5\exp[(4J' + 2J)/kT]$$

Where  $\rho$  is the (molar) fraction of mononuclear Cu(II) ions and the other symbols have their usual meanings. The magnetic susceptibility data was least-

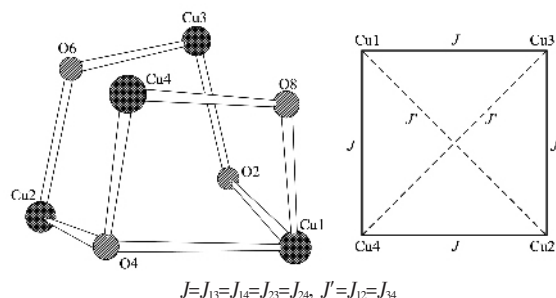


Fig.4 Schematic representation of the magnetic coupling model in (Cu<sub>4</sub>L<sub>4</sub>)(CH<sub>3</sub>CN)

squares fitted to the equation and the best fitting parameters (corresponding to the full line in Fig.3) were found to be  $J=J'=-3.3(8) \text{ cm}^{-1}$ ,  $g=2.05$ ,  $\rho=5 \times 10^{-4}$ , and  $N_{\alpha}$  (temperature independent magnetic susceptibility) taken as  $240 \times 10^{-6} \text{ emu} \cdot \text{mol}^{-1}$ , and agreement factor  $R=(\chi_{\text{m}}^{\text{obs}}-\chi_{\text{m}}^{\text{calc}})^2/(\chi_{\text{m}}^{\text{obs}})^2=3.9 \times 10^{-4}$ . The results show the presence of weakly antiferromagnetic interactions as well as most of the reported tetranuclear copper(II) complexes<sup>[19,20]</sup>.

In summary, this paper describes a complex based on copper(II) and chiral Schiff-base ligand. Preliminary investigation of the magnetic properties reveals that it shows weak antiferromagnetic coupling with  $J=J'=-3.3(8) \text{ cm}^{-1}$ . The synthesis of species using chiral ligand that contain chiral coordination metal core is currently being pursued, as are further investigations into the weak MChD effects in a chiral paramagnetic material.

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