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Synthesis, X-ray structure and magnetic properties of trinuclear copper(II) tridentate Schiff base complexes containing a partial cubane Cu_3O_4 core

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Abstract

Two copper(II) complexes with tridentate Schiff bases AE and SE, which are condensed from *N*,*N*-dimethylethylenediamine and acetylacetone or salicylaldehyde, respectively, have been synthesized, characterized by X-ray structural analysis, magnetic measurement, IR and UV spectra. Both of the complexes contain a partial cubane Cu_3O_4 core consisting of [Cu(AE)] or [Cu(SE)], nonbonded ClO_4^- anions and water molecules. The two complexes comprise three [Cu(AE)] or [Cu(SE)] subunits, respectively, which are interconnected through two types of oxygen bridges afforded by the oxygen atoms of the ligands and the central OH^- group. The average Cu–O distances involving hydroxy OH^- are 2.053 Å in complex **1** and 2.078 Å in complex **2**. Complex **1** exhibits an antiferromagnetic interactions between the copper ions with $J = -2.40$ cm^{-1} and $g = 2.038$. Complex **2** exhibits a ferromagnetic interaction between the copper ions with $J = 7.83$ cm^{-1} and $g = 2.02$.

Keywords: Trinuclear copper complexes; Crystal structures; Magnetic properties

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1. Introduction

In the past decades many efforts have been made to synthesize and investigate polynuclear Cu(II) complexes with various bridges between the metal centers [1–4], particularly in connection with their magnetic behaviour [5]. Exchange-coupled polynuclear copper(II) complexes have aroused extensive interest due to their importance in biological processes and in inorganic materials science [6,7]. Magneto-structural correlations in binuclear copper(II) complexes bridged by pairs of alkoxide or phenoxide groups [8,9] show that the major factor controlling spin coupling (J) between the metal centers is the Cu–O–Cu angle (Φ).

The ligands condensation from salicylaldehyde or acetylacetone with diamine forms the basis of an extensive class of chelating ligands that has enjoyed popular use in the coordination chemistry of transition metal elements [10–14]. The magnetic properties of the Schiff base metal complexes with partial and complete cube structure have been studied extensively [15–17]. In this paper we present the synthesis, magnetic properties and X-ray crystal structure of two trinuclear hydroxo-bridged, Cu(II) tridentate Schiff base complexes. Although both the complexes have partial cubane Cu_3O_4 cores, they are different in magnetic properties. Complex **1** exhibits an antiferromagnetic interactions, while complex **2** exhibits rare ferromagnetic interactions.

2. Experimental

2.1. Materials and synthesis

All starting materials were of analytical grade.

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2.1.1. *($[Cu_3(\mu_3\text{-}OH)(AE)_3](ClO_4)_2\cdot 1.5H_2O$ (1))*

To *N*,*N*-dimethylethylenediamine (0.5 mmol) in methanol (10 ml) was added acetylacetone (0.5 mmol). The mixture was refluxed for 1 h to get a bright yellow solution and then was evaporated to yield a deep red oil. The oil was dissolved in 5 ml methanol and $Cu(ClO_4)_2\cdot 6H_2O$ (0.5 mmol) was added slowly. The solution was stirred for 10 min and then Et_3N (0.14 ml) was added. The resulting mixture was stirred for 2 h. Deep green single crystals suitable for X-ray analysis were separated after several weeks. Yield: 106.68 mg (68%). *Anal*. Calc. for $C_9H_{18.33}Cl_{0.67}CuN_2O_{4.50}$: C, 34.5; H, 5.9; N, 8.9%. Found: C, 34.6; H, 6.0; N, 8.6%.

2.1.2. *($[Cu_3(\mu_3\text{-}OH)(SE)_3](ClO_4)_2\cdot 0.5H_2O$ (2))*

To *N*,*N*-dimethylethylenediamine (0.5 mmol) in methanol (10 ml) was added salicylaldehyde (0.5 mmol). The mixture was refluxed for 1 h and then $Cu(ClO_4)_2\cdot 6H_2O$ (0.5 mmol) in 3 ml water was added slowly. The resulting mixture was stirred for 2 h. Deep green single crystals suitable for X-ray analysis were separated after several weeks. Yield: 117.06 mg (71%). *Anal*. Calc. for $C_{33}H_{47}Cl_2Cu_3N_6O_{12.50}$: C, 40.1, H, 4.8, N, 8.5%. Found: C, 40.1; H, 4.6; N, 8.7%.

2.2. *Physical measurements*

IR spectra were recorded as KBr discs on a Shimadzu IR-408 infrared spectrophotometer in the 4000–600 cm^{-1} region. The ultraviolet and visible spectra were measured on a Shimadzu UV-2101 PC spectrophotometer, methanol was the solvent in this study. Elemental analyses (C, H, N) were performed on a Perkin–Elmer 240C analyser. Magnetic susceptibility measurement of crystalline samples were carried out in the temperature range 4–300 K on a Maglab system 2000 magnetometer at a field strength of 10 000 G.

2.3. *X-ray crystallography*

Diffraction intensity data for single crystals of **1** and **2** were collected at room temperature on a Bruker Smart 1000 CCD area detector equipped with graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å). Empirical absorption corrections were applied using the SADABS program. The structure was solved by the direct method and refined by the full-matrix least-squares method on F^2 with anisotropic thermal parameters for all non-hydrogen atoms [18]. The hydrogen atoms of solvent molecules were not added, and the other hydrogen atoms were located geometrically and refined isotropically. Further details of the structure analysis are given in Table 1.

Table 1
Crystallographic data for complex **1** and **2**

Complexes	1	2
Formula	$C_9H_{18.33}Cl_{0.67}CuN_2O_{4.50}$	$C_{33}H_{47}Cl_2Cu_3N_6O_{12.50}$
Formula weight	313.77	989.29
T (K)	293(2)	293(2)
Crystal system	trigonal	monoclinic
Space group	$R\bar{3}c$	$P2_1/c$
Unit cell dimensions		
a (Å)	11.959(6)	11.167(5)
b (Å)	11.959(6)	30.019(13)
c (Å)	110.32(3)	13.362(6)
β (°)	106.366(7)	
γ (°)	120	
Z	36	4
μ (mm^{-1})	1.563	1.659
F(0 0 0)	5855	2032
Reflections collected/unique	16 638/2609 [$R_{int} = 0.0797$]	174 42/7536 [$R_{int} = 0.0597$]
Data/restraints/parameters	2609/0/177	7536/20/509
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0523$, $wR_2 = 0.1343$	$R_1 = 0.0508$, $wR_2 = 0.1044$
R indices (all data)	$R_1 = 0.0771$, $wR_2 = 0.1477$	$R_1 = 0.1084$, $wR_2 = 0.1200$

3. Results and discussion

3.1. *Spectroscopic characterization*

In complex **1** there is an absorption band at 3200 cm^{-1} which is assigned to ν(OH) of the triply-bridging hydroxy group. The absorption of ν(OH) of water appears at about 3400 cm^{-1}. The absorption bands of C=N appear at about 1600 and 1520 cm^{-1}. There is a broad band at 1105 cm^{-1}, which is due to the perchlorate anions v_3 mode in Td symmetry.

In complex **2**, the broad absorption band at 3200 and 3450 cm^{-1} are assigned to ν(OH) of the triply-bridging hydroxy group and water, respectively. The absorption band at 1615 and 1540 cm^{-1} is attributed to the ν(C=N). The absorption band of perchlorate appears at 1095 cm^{-1}.

Complexes **1** and **2** show a broad band centered at about 605 nm ($\varepsilon = 2.15 \times 10^2$ $dm^3\cdot mol^{-1}\cdot cm^{-1}$) and 638 nm ($\varepsilon = 3.58 \times 10^2$ $dm^3\cdot mol^{-1}\cdot cm^{-1}$) in methanol at 298 K, respectively, which is tentatively attributed to the electronic transitions $^2B_1 \rightarrow {}^2E$ and $^2B_1 \rightarrow {}^2B_2$. This spectrum is typical d–d charge-transfer bands in the square-pyramidal Cu(II) surrounding [19,20].

3.2. *Description of the structure*

Complex **1** and **2** are isostructural complexes. A view of the trinuclear cations $[Cu_3(AE)_3(OH)]^{2+}$ and $[Cu_3(SE)_3(OH)]^{2+}$ is given in Fig. 1. Details of data

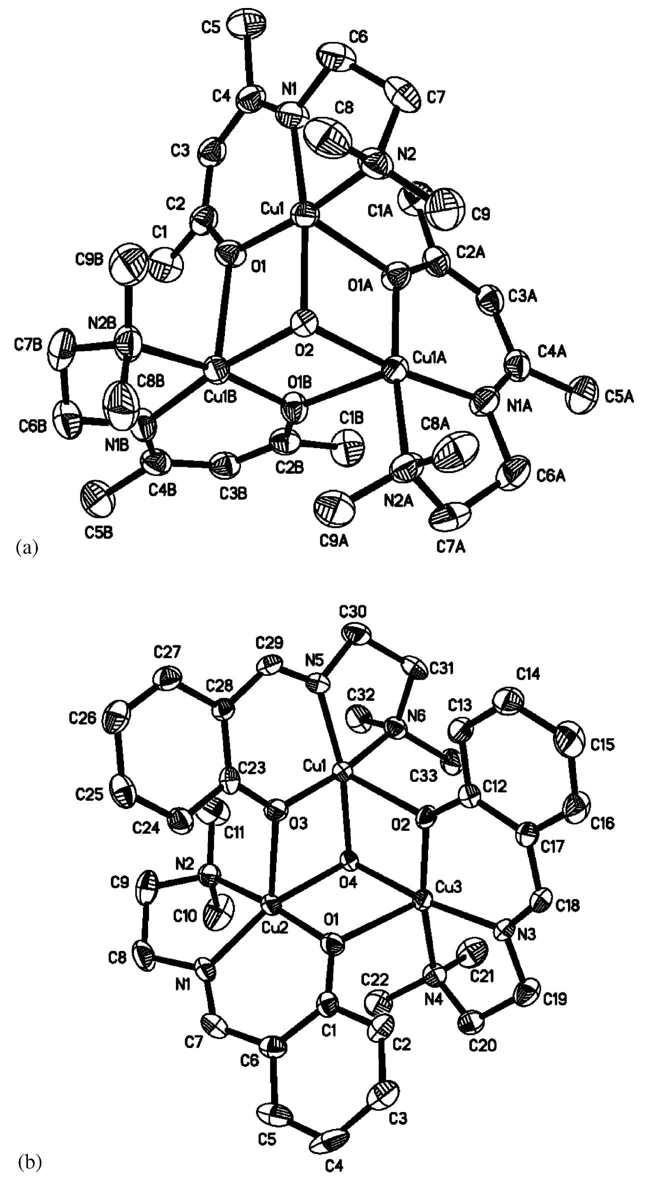

Fig. 1. Perspective view of trinuclear unit of complex **1** (a) and complex **2** (b) with the atom numbering scheme.

collection procedures and structures are given in Table 1 and the selected bond distances and angles are listed in Table 2.

Each complex contains a partial cubane Cu_3O_4 core, nonbonded ClO_4^- anions and water molecules. In contrast with the trinuclear complexes derived from the half-unit ligand [21], the crystal structures of complex **1** and **2** include solvent water molecules.

It is an approximate description that the trinuclear cations of the two complexes comprise three [Cu(AE)] and [Cu(SE)] subunits, respectively, which are interconnected through two types of oxygen bridges afforded by the oxygen atoms of the ligands and the central OH^- group, respectively. The copper center has square pyramidal geometry with the basal plane comprising two nitrogen atoms and an oxygen atom from the tridentate ligand [AE] or [SE] and the hydroxy OH^-, while oxygen atom of the ligand completes the coordination sphere in the apical site. In each subunit, the axial Cu–O bonds (2.294 Å for complex **1** and 2.293 Å average for complex **2**) are longer than those of equatorial oxygen atoms of the ligands (1.922 Å for complex **1** and 1.953 Å average for complex **2**). The Cu–N bonds of tertiary nitrogen atoms (2.060 Å in complex **1** and 2.085 Å average in complex **2**) are longer than those of the *sec*-nitrogen atoms (1.943 Å in complex **1** and 1.971 Å average in complex **2**).

The presence of Cu–OH is confirmed by the trigonal pyramid formed by the Cu_3O fragment; here the oxygen occupies the apex of the pyramid, and the three copper atoms are located at the corners. The distance of the copper atoms and the central oxygen atoms in Cu_3OH subunits are 2.052 Å in complex **1** and 2.078 Å average in complex **2**, respectively. The distance of Cu···Cu in complex **2** (3.210 Å average) is the same as that in complex **1** (3.208 Å). The apical hydroxy OH^- is located 0.8806 Å above the Cu_3 plane in complex **1** and 0.932(3) Å in complex **2**.

3.3. Magnetic properties

The magnetic susceptibilities (and effective magnetic moment μ_{eff}) of the two complexes over 4–300 K are illustrated in Figs. 2 and 3. If the trimeric unit has equivalent metal atoms that form an equilateral triangle, the spin Hamiltonial will describe the interactions of the spins. The isotropic exchange interaction between three ions of $S = 1/2$ located at the apices of an equilateral triangle results in a splitting of the degenerate energy levels into one quartet state with a total spin $S' = 3/2$ and two degenerate doublet states with $S' = 1/2$. A closed form of the magnetic susceptibility has been derived [22].

$$\chi_{\text{M}} = \frac{Ng^2(\mu_{\text{B}})^2}{4\kappa(T-\theta)}\,\frac{1+5\mathrm{e}^{3J/\kappa T}}{1+\mathrm{e}^{3J/\kappa T}} \tag{1}$$

(complex **1**: $\theta = 0$, complex **2**: $\theta = 0.2$)

The magnetic behaviour of complex **1** is shown in Fig. 2 by plotting μ_{eff} and χ_{M} vs. T. At 4 K the moment per Cu_3 unit in complex **1** is practically equal to 1.79 μ_{B}, so that the ground state has one unpaired electron per unit. As the temperature rises, μ_{eff} increases, and at room temperature it reaches 3.04 μ_{B}, which compares well to the expected value for three uncoupled $S = 1/2$ centres (theoretical value with $g = 2$, $\mu_{\text{eff}} = 3.0\ \mu_{\text{B}}$). A least-squares fit of the data to the equation yields the values of $J = -2.40\ \text{cm}^{-1}$, $g = 2.038$ and $R = 3.68 \times 10^{-3}$. This is indicative of the presence of antiferromagnetic interactions in complex **1**. The value of $J = -2.40\ \text{cm}^{-1}$ found for complex **1** is considerably lower than the values reported for trinuclear hydroxo-bridged com-

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

Complex **1**					
Bond distances					
Cu(1)–O(1)	1.922(3)	Cu(1)–N(1)	1.943(4)	Cu(1)–O(2)	2.052(2)
O(2)–Cu(1B)	2.052(2)	Cu(1)–O(1A)	2.294(3)	O(2)–Cu(1A)	2.052(2)
Bond angles					
O(1)–Cu(1)–N(1)	93.48(16)	O(1)–Cu(1)–O(1A)	94.38(19)	O(1)–Cu(1)-O(2)	83.23(13)
O(2)–Cu(1)–O(1A)	74.62(12)	N(1)–Cu(1)–O(2)	172.17(14)	Cu(1)–O(1)–Cu(1B)	98.77(13)
O(1)–Cu(1)–N(2)	170.03(18)	N(2)–Cu(1)–O(1A)	95.27(16)	N(1)–Cu(1)–N(2)	85.08(17)
N(1)–Cu(1)–O(1A)	112.82(15)	O(2)–Cu(1)–N(2)	96.92(15)	Cu(1A)–O(2)–Cu(1B)	102.94(14)
Cu(1)–O(2)–Cu(1A)	102.94(14)	Cu(1)–O(2)–Cu(1B)	102.94(14)		
Complex **2**					
Bond distances					
Cu(1)–O(3)	1.951(4)	Cu(1)–N(5)	1.982(5)	Cu(1)–N(6)	2.070(5)
Cu(1)–O(4)	2.080(4)	Cu(1)–O(2)	2.292(3)	Cu(2)–O(1)	1.947(4)
Cu(2)–N(1)	1.970(5)	Cu(2)–N(2)	2.085(5)	Cu(2)–O(4)	2.111(3)
Cu(2)–O(3)	2.238(4)	Cu(3)–O(2)	1.961(4)	Cu(3)–N(3)	1.960(4)
Cu(3)–O(4)	2.043(3)	Cu(3)–N(4)	2.091(5)	Cu(3)–O(1)	2.350(4)
Bond angles					
O(3)–Cu(1)–N(5)	92.24(18)	O(3)–Cu(1)–N(6)	169.70(17)	N(5)–Cu(1)–N(6)	84.2(2)
O(3)–Cu(1)–O(4)	82.91(14)	N(5)–Cu(1)–O(4)	168.10(16)	N(6)–Cu(1)–O(4)	98.71(17)
O(3)–Cu(1)–O(2)	93.27(15)	N(5)–Cu(1)–O(2)	115.81(17)	N(6)–Cu(1)–O(2)	96.98(16)
O(4)–Cu(1)–O(2)	75.44(13)	O(1)–Cu(2)–N(1)	92.65(18)	O(1)–Cu(2)–N(2)	173.37(18)
N(1)–Cu(2)–N(2)	83.7(2)	O(1)–Cu(2)–O(4)	84.91(15)	N(1)–Cu(2)–O(4)	161.37(18)
N(2)–Cu(2)–O(4)	96.78(17)	O(1)–Cu(2)–O(3)	91.29(15)	N(1)–Cu(2)–O(3)	122.87(18)
N(2)–Cu(2)–O(3)	95.34(18)	O(4)–Cu(2)–O(3)	75.70(13)	O(2)–Cu(3)–N(3)	92.31(17)
O(2)–Cu(3)–O(4)	84.01(14)	N(3)–Cu(3)–O(4)	172.16(17)	O(2)–Cu(3)–N(4)	167.60(17)
N(3)–Cu(3)–N(4)	84.16(19)	O(4)–Cu(3)–N(4)	97.98(16)	O(2)–Cu(3)–O(1)	97.55(15)
N(3)–Cu(3)–O(1)	110.55(17)	O(4)–Cu(3)–O(1)	76.88(13)	N(4)–Cu(3)–O(1)	94.82(16)
Cu(2)–O(1)–Cu(3)	96.46(15)	Cu(3)–O(2)–Cu(1)	97.99(14)	Cu(1)–O(3)–Cu(2)	99.99(16)
Cu(3)–O(4)–Cu(1)	102.56(14)	Cu(3)–O(4)–Cu(2)	101.46(15)	Cu(1)–O(4)–Cu(2)	100.18(15)

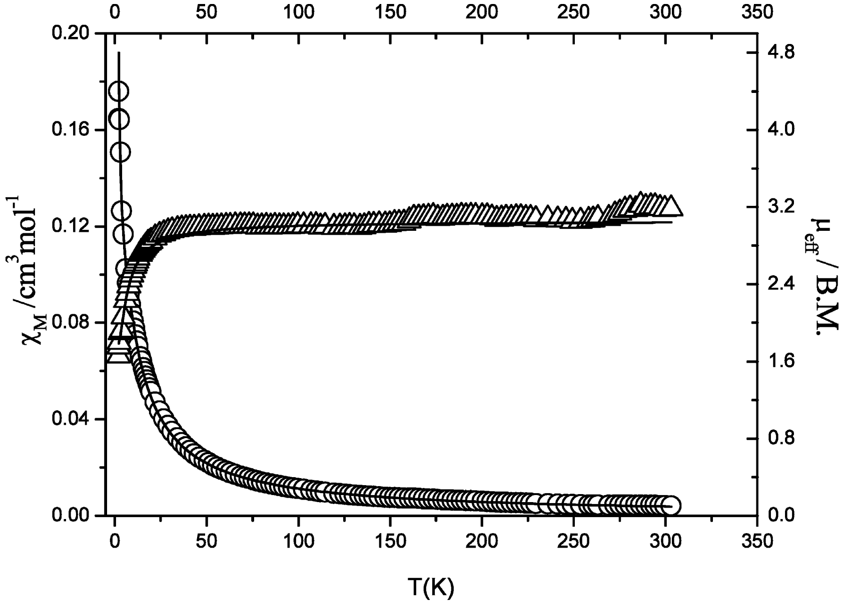

Fig. 2. A plot for complex **1** of temperature dependence of χ_M (○) vs. T and μ_{eff} (△) vs. T. The solid lines are theoretical fits based on Eq. (1).

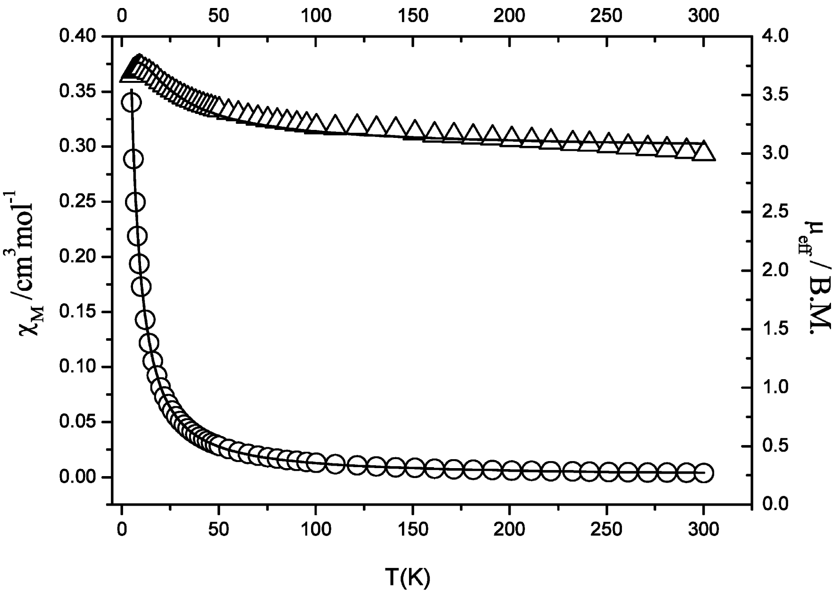

Fig. 3. A plot for complex **2** of temperature dependence of χ_M (○) vs. T and μ_{eff} (△) vs. T. The solid lines are theoretical fits based on Eq. (1).

plexes with imine-oximato (-122 to -1000 cm^{-1}) and pyrrazolato ligands (-200 cm^{-1}) [22,23]. On the other hand, the observed value corresponds well with the value of -12 cm^{-1} found for $[(CuL)_3(OH)](ClO_4)_2$ (LH = 7-amino-4-methyl-5-aza-3-hepten-2-one) [21].

For complex **2**, the same equation is applicable. The μ_{eff} at room temperature compares well to the theoretical value. As the temperature is lowed, μ_{eff} increases from 3.09 at 300 K to 3.77 μ_B at 9 K and then decreases to 3.73 μ_B at 4 K. A least-squares fit of the data to the equation yields the values of $J = 7.83$ cm^{-1}, $g = 2.02$ and $R = 6.89 \times 10^{-4}$. This behaviour is characteristic of ferromagnetic interaction between the copper ions, and the decrease of μ_{eff} from 3.77 μ_B at 9 K to 3.73 μ_B at 4 K maybe due to intermolecular interactions. Most of the tricopper complexes previously reported exhibit antiferromagnetic interactions [21–26], while ferromagnetic interactions in μ_3-oxo and μ_3-hydroxo tricopper complexes are rare [27].

It is of interest to compare the structural and magnetic properties of trinuclear complexes involving hydroxy OH$^-$ bridges. In $[L_3Cu_3OH(ClO_4)]^+$ and $[L'{}^3Cu_3O(ClO_4)]_2$ (LH = 3-(phenylimino)butanone 2-oxime, L′H = 1,2-diphenyl-2-(methylimino)ethanone 1-oxime), the oxygen is located 0.695 and 0.352 Å above the Cu_3 plane, respectively [21], while in complex **1** and **2** the oxygen atoms are farther from the Cu_3 plane: 0.8806 Å in complex **1** and 0.9323 Å in complex **2**. The coordination planes (O_1, O_2, N_1, N_2; O_{1A}, O_2, N_{1A}, N_{2A}; O_{1B}, O_2, N_{1B}, N_{2B}) in complex **1** make angles 96.8°, 96.8°, 83.2° with each other, and in complex **2** the angles of the planes (O_3, O_4, N_5, N_6; O_1, O_4, N_1, N_2; O_2, O_4, N_3, N_4) are increased to 98.8°, 103.7°, 87.5°. In the oximato complexes the angles are 40.3, 28.5, 31.1° in $[L_3Cu_3OH(ClO_4)]^+$ showing $J = -120$ cm^{-1} and 14.6°, 20.4° and 26.6° in $[L'{}^3Cu_3O(ClO_4)]_2$ showing $J = -1000$ cm^{-1} [21]. These observations imply that the degree of coplanarity of the three CuN_2O_2 coordination planes influences the magnetic coupling. According to the Kahn's model [5], the exchange integral (J) can be decomposed in two terms, one ferromagnetic (J_F) and the other antiferromagnetic contributions (J_{AF}). In this model, the value of J_{AF} is proportional to the square of the integral overlap (S^2). In our case the large distance between the O atom and Cu_3 plane causes large dihedral angle of the base planes. The low degree of coplanarity hinders $d_{x^2+y^2}$ overlap, which results in antiferromagnetic interactions in complex **1** and ferromagnetic interactions in complex **2**.

4. Supplementary data

Supplementary data are available from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk) on request, quoting the deposition number CCDC 189196 for complex **1** ad 189197 for complex **2**.

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