Instrumental Achievements

Crystal Structure of Bis[4-(2-oxy-1-naphthylmethyliminomethyl-*N,O:O*)-2-penten-2-olato-*O*]dicopper(II) Complex

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The structures of dimeric Cu complexes of $Cu_2(C_{11}H_{10}ClNO_2)_2$,[1] $Cu_2(C_{14}H_{11}NO_2)_2$,[2] and $Cu_2(C_{12}H_{13}NO_2)_2$,[3] in the presence of different tridentate ONO type Schiff-base ligands are reported from this laboratory. The title compound is a continuation of forming dimeric Cu complexes and a determination of the crystal structure by our group. The dimeric Cu^{II} complexes bridged with an O atom possess subnormal magnetic moments due to a super-exchange interaction.[4-6] In the present work concerning $Cu_2(C_{16}H_{15}NO_2)_2$ our aim was to investigate the effect of the ligand stereochemistry around the Cu atom.

2-Hydroxy-1-naphthaldehyde (0.73 g, 0.01 mol) was dissolved in ethanol (50 ml) by heating. Into this solution acetylacetone (1.0 g, 0.01 mol) was added and mixed. The mixture was set aside for 24 h. The precipitated ligand was filtered out and dried in air; (0.255 g, 0.001 mol) of this ligand was dissolved in 50 ml of DMF:MeCN:MeOH (1:1:1) and mixed with $Cu(CH_3COO)_2 \cdot H_2O$ (0.199 g, 0.001 mol) in hot 20 ml MeOH (see also Fig. 1). The mixture was heated to nearly 80° C and crystals were obtained after allowing the solution to stand for 24 h at room temperature.

Table 1 shows the crystal and experimental data, while the final atomic parameters are given in Table 2. The bond distances and angles are given in Table 3.

The reported complex is centrosymmetric, and the asymmetric unit is bridged *via* the O atom of the 2-oxy-1-naphthylmethyliminomethyl group of the ligand (Fig. 2). In this complex the Cu–O bond distances for the non-

Table 1 Crystal and experimental data

Formula: $C_{32}H_{30}Cu_2N_2O_4$	
Formula weight=316.84	
Crystal system: monoclinic	
Space group: $P2_1/c$	Z=2
a=7.7295(12) Å	
b=19.2524(14) Å	
c=8.9716(11) Å	
β=93.108(3)°	
V=1333.1(5) Å^3	
D_x=1.579 g/cm^3	
μ=(Mo K$_\alpha$)=1.640 mm^{-1}	
T=295 K	
Dark red	
F(000)=652	
0.4×0.1×0.06 mm	
Radiation=Mo K$_\alpha$	
R=0.067	
Rw=0.068	
No. of reflections used=1264	
No. of parameters=181	
Goodness-of-fit=0.80	
Measurements: Enraf Nonius CAD-4 diffractometer	
Program system: CAD-4 EXPRESS Software	
Structure determination: MolEN	
Treatment of hydrogen atoms: all H-atoms were geometrically calculated, the coordinates of ethyl and methyl H-atoms were refined for few cycles and then a riding model was used for all H atoms	
Refinement: full matrix least-squares (MolEN)	

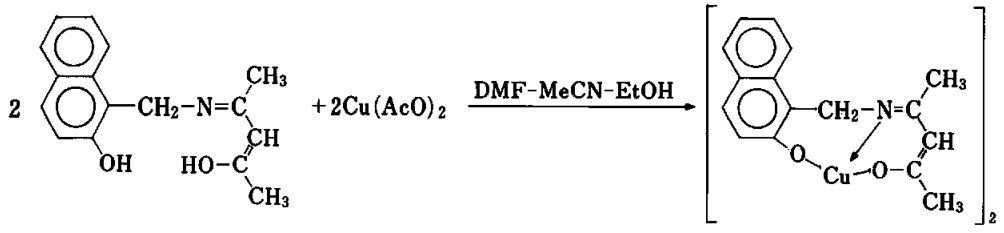

Fig. 1 Reaction scheme.

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Table 2 Final atomic coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms

Atom	x	y	z	B_{eq}/Å^2
Cu	0.0247(2)	0.92419(6)	0.9600(1)	2.85(2)
O1	0.1972(9)	0.8587(4)	1.0159(7)	3.4(2)
O2	-0.1042(8)	1.0068(3)	0.8949(7)	2.8(1)
N1	-0.106(1)	0.8647(4)	0.8239(8)	2.6(2)
C1	0.220(1)	0.8024(5)	0.942(1)	3.2(2)
C2	0.106(1)	0.7769(5)	0.834(1)	3.5(2)
C3	-0.052(1)	0.8044(5)	0.777(1)	2.6(2)
C4	0.384(1)	0.7643(6)	0.986(1)	4.8(3)
C5	-0.153(2)	0.7618(6)	0.666(1)	5.0(3)
C6	-0.270(1)	0.8969(5)	0.765(1)	2.5(2)
C7	-0.235(1)	0.9594(5)	0.675(1)	2.5(2)
C8	-0.280(1)	0.9665(5)	0.519(1)	2.7(2)
C9	-0.365(1)	0.9136(6)	0.431(1)	4.3(3)
C10	-0.404(1)	0.9228(7)	0.282(1)	4.9(3)
C11	-0.367(1)	0.9832(7)	0.213(1)	5.0(3)
C12	-0.288(1)	1.0374(6)	0.291(1)	3.8(2)
C13	-0.242(1)	1.0285(5)	0.444(1)	3.0(2)
C14	-0.156(1)	1.0830(6)	0.526(1)	3.3(2)
C15	-0.113(1)	1.0751(6)	0.674(1)	2.8(2)
C16	-0.154(1)	1.0142(5)	0.749(1)	2.3(2)

$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* (\boldsymbol{a}_i \cdot \boldsymbol{a}_j)$.

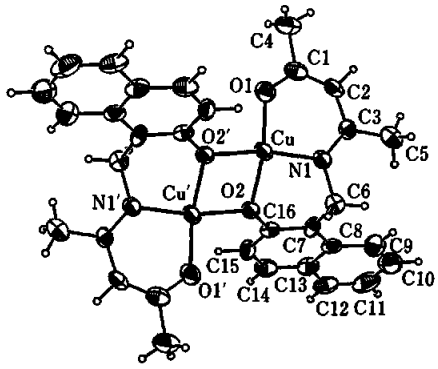

Fig. 2 ORTEP drawing of the title compound with the atomic labeling scheme. The thermal ellipsoids are drawn at the 50% probability level. Hydrogen atoms are shown as small circles with arbitratry radii. The symmetry code for Cu', O2', O1' and O2' is $-x$, $2-y$, $2-z$.

bridging O1 atom is shorter, 1.883(7) Å. The bond distances for the bridging O2 (1.949(7) Å) and O2' atoms (1.937(6) Å) are practically equal. The Cu–N bond distance is 1.922(8) Å. The coordination angle around the Cu atom has values of between 77.3(3) – 96.4(3)°.

Table 3 Bond distances (Å) and angles (°)

Cu - Cu'	3.035(2)	C6 - C7	1.48(1)
Cu - O1	1.883(7)	C7 - C8	1.43(1)
Cu - O2	1.949(7)	C7 - C16	1.38(1)
Cu - O2'	1.937(6)	C8 - C9	1.43(2)
Cu - N1	1.922(8)	C8 - C13	1.41(1)
O1 - C1	1.29(1)	C9 - C10	1.37(2)
O2 - C16	1.35(1)	C10- C11	1.35(2)
N1 - C3	1.31(1)	C11- C12	1.38(2)
N1 - C6	1.48(1)	C12- C13	1.41(1)
C1 - C2	1.37(1)	C13- C14	1.42(2)
C1 - C4	1.50(2)	C14- C15	1.36(1)
C2 - C3	1.40(1)	C15- C16	1.39(1)
C3 - C5	1.48(1)		

Cu - O2 - Cu'	102.7(3)	N1 - C6 - C7	111.0(8)
O1 - Cu - O2'	94.9(3)	C6 - C7 - C8	124.5(9)
O1 - Cu - O2	165.7(3)	C6 - C7 - C16	117.1(9)
O2 - Cu - O2'	77.3(3)	C8 - C7 - C16	118(1)
O1 - Cu - N1	96.4(3)	C7 - C8 - C9	124(1)
O2 - Cu - N1	93.0(3)	C7 - C8 - C13	120(1)
O2' - Cu - N1	166.6(3)	C9 - C8 - C13	116(1)
Cu - O1 - C1	122.8(7)	C8 - C9 - C10	121(1)
Cu - O2 - C16	119.3(6)	C9 - C10- C11	121(1)
Cu - N1 - C3	124.3(7)	C10- C11- C12	121(1)
Cu - N1 - C6	112.9(6)	C11- C12- C13	119(1)
C3 - N1 - C6	122.6(9)	C8 - C13- C12	121(1)
O1 - C1 - C2	124.(1)	C8 - C13- C14	119(1)
O1 - C1 - C4	114.(1)	C12- C13- C14	120(1)
C2 - C1 - C4	121.(1)	C13- C14- C15	120(1)
C1 - C2 - C3	130.(1)	O2 - C16- C7	118.7(9)
N1 - C3 - C2	120.5(9)	O2 - C16- C15	119.9(9)
N1 - C3 - C5	123.(1)	C7 - C16- C15	121.3(9)
C2 - C3 - C5	117(1)		

The dihedral angle between the bridging plane (Cu, O2, Cu', O2') and the coordination plane (O1, N1, O2, O2') is 8(1)°. The two moieties of the ligand (O1, C1, C2, C3, C4, C5, N1) and ((O2, C6 – C16) make a dihedral angle of 63.7(2). In this complex the Cu···Cu distance is 3.035(2) Å.

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