Instrumental Achievements

Crystal Structure of Bis[4-(5-methyl-2-oxyphenyl-κO,2κO)-4-imino-2-penten-2-olato-*O*,*O*′,*N*′]copper(II)

Orhan ATAKOL*†, Filiz ERCAN, Dincer ÜLKÜ** and Nazife YILMAZ***

**Department of Chemistry, Science Faculty, Ankara University, Tandogan 06100 Ankara, Turkey*
***Department of Engineering Physics, Haceteppe University, Beytepe 06322, Ankara, Turkey*

The title compound, $Cu_2(C_{12}H_{13}NO_2)_2$, is a doubly oxygen-bridged dimeric copper(II) complex. Doubly oxygen bridged complexes with subnormal magnetic moments are known to exhibit super exchange interaction.[1-3] The Cu atoms have a distorted square-planar environment, with bond angles in the range 76.9(1)–100.3(1)°. The average Cu–O distance is 1.916(2) Å and the average Cu–N distance is 1.941(3) Å. The Cu1 and Cu2 atoms are located 0.0339(5) and 0.0336(5) Å off the coordination best plane, respectively. The bridging angles Cu1–O1–Cu2, O1–Cu2–O3, Cu1–O3–Cu2 and O1–Cu1–O3 are 102.8(1), 76.9(1), 102.5(1) and 77.5(1)°, respectively. The dihedral angles between the Cu1–O1–Cu2–O3 bridging plane and the coordination planes (O1, N1, O2, O3 and O1, O3, N2, O4) around coppers are 2.9(9) and 8.0(3)°, respectively. The Cu···Cu distance [3.021(2) Å] in the bridging plane is rather long, to have a direct interaction. This distance is in between the two values: 2.994(2) Å[4] and 3.073(2) Å[5], reported previously.

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

4-(5-Methyl-2-hydroxyphenyl)iminomethyl-2-hydroxy-2-penten (0.205 g) was dissolved in 30 ml hot MeCN. To this mixture, a solution of $Cu(CH_3COO)_2 \cdot H_2O$ (0.199 g, 1 mmol) in 30 ml hot MeOH was added and mixed. After 2 h the dark green Cu complex was filtered and dried.

This complex (0.100 g) was crystallized from 70 ml *N*,*N*-dimethylformamide.

Table 1 Crystal and experimental data

Formula: $Cu_2C_{24}H_{26}N_2O_4$	
Formula weight=533.57	
Crystal system: monoclinic	
Space group: $P2_1/n$	Z=4
a=10.963(2) Å	
b=17.051(1) Å	
c=11.953(1) Å	
β=101.956(8)°	
V=2185.6(4) Å^3	
D_x=1.62 Mg/m^3	
μ(Mo K$_\alpha$)=1.98 mm^{-1}	
T=295 K	
Dark green	
F(000)=1096	
Crystal size: 0.35×0.35×0.18 mm	
Radiation=Mo K$_\alpha$	
R=0.043	
Rw=0.052	
No. of reflections used=3294	
No. of parameters=289	
Goodness-of-fit=1.09	
Measurement: Enraf Nonius CAD-4 diffractometer	
Program system: CAD-4 EXPRESS Software	
Structure determination: MolEN	
Refinement: full matrix least-squares (MolEN)	
Hydrogen atoms: H atoms ridging	
$w=[\sigma F^2+(0.002F)^2+1.15]^{-1}$	

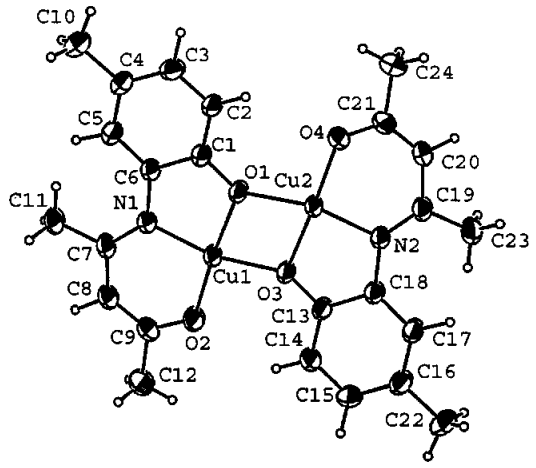

Fig. 1 The ORTEP drawing of the title compound with atom labeling.

† To whom correspondence should be addressed.

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

Atom	x	y	z	B_{eq}/Å^2
Cu1	0.65019(4)	0.02569(3)	0.58843(4)	2.791(9)
Cu2	0.84795(4)	-0.02582(3)	0.46108(4)	2.782(9)
O1	0.7706(3)	0.0685(2)	0.5126(3)	3.27(6)
O2	0.5352(3)	-0.0248(2)	0.6585(3)	4.28(7)
O3	0.7350(3)	-0.0683(2)	0.5475(3)	3.22(6)
O4	0.9538(3)	0.0210(2)	0.3744(3)	3.36(6)
N1	0.6132(3)	0.1337(2)	0.6199(3)	2.73(7)
N2	0.8599(3)	-0.1329(2)	0.4096(3)	2.70(7)
C1	0.7741(4)	0.1474(2)	0.5150(4)	2.65(8)
C2	0.8553(4)	0.1896(2)	0.4642(4)	3.03(8)
C3	0.8583(4)	0.2706(3)	0.4712(4)	3.10(9)
C4	0.7808(4)	0.3104(2)	0.5295(4)	3.26(9)
C5	0.6984(4)	0.2676(3)	0.5793(4)	3.41(9)
C6	0.6910(4)	0.1861(2)	0.5729(3)	2.60(8)
C7	0.5243(4)	0.1519(2)	0.6752(4)	2.72(8)
C8	0.4543(4))	0.0933(3)	0.7182(4)	3.01(8)
C9	0.4620(4)	0.0134(3)	0.7111(4)	3.18(9)
C10	0.7861(5)	0.3984(3)	0.5392(5)	4.9(1)
C11	0.4888(4)	0.2347(3)	0.6966(4)	3.54(9)
C12	0.3804(4)	-0.0385(3)	0.7665(4)	4.1(1)
C13	0.7339(4)	-0.1473(2)	0.5480(4)	2.75(8)
C14	0.6711(4)	-0.1901(3)	0.6158(4)	3.31(9)
C15	0.6810(4)	-0.2710(3)	0.6186(4)	3.41(9)
C16	0.7524(4)	-0.3103(2)	0.5533(4)	3.10(9)
C17	0.8147(4)	-0.2669(2)	0.4847(4)	3.02(8)
C18	0.8046(4)	-0.1853(2)	0.4770(4)	2.69(8)
C19	0.9006(4)	-0.1494(2)	0.3156(4)	2.72(8)
C20	0.9526(4)	-0.0905(3)	0.2558(4)	3.17(9)
C21	0.9767(4)	-0.0133(3)	0.2850(4)	3.14(9)
C22	0.7626(5)	-0.3984(3)	0.5571(5)	4.21(1)
C23	0.8951(4)	-0.2301(3)	0.2636(4)	3.44(9)
C24	1.0354(5)	0.0390(3)	0.2095(4)	4.4(1)

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

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

Cu1-Cu2	3.021(2)	N1 - C7	1.323(6)
Cu1 - O1	1.895(3)	N2 - C18	1.421(6)
Cu1 - O2	1.862(4)	N2 - C19	1.324(6)
Cu1 - O3	1.965(3)	C4 - C10	1.507(6)
Cu1 - N1	1.941(3)	C7 - C8	1.419(6)
Cu2 - O1	1.976(3)	C7 - C11	1.501(6)
Cu2 - O3	1.912(3)	C8 - C9	1.369(6)
Cu2 - O4	1.885(3)	C9 - C12	1.505(7)
Cu2 - N2	1.941(3)	C16 - C22	1.508(6)
O1 - C1	1.348(5)	C19 - C20	1.419(6)
O2 - C9	1.294(6)	C19 - C23	1.508(6)
O3 - C13	1.348(5)	C20 - C21	1.374(6)
O4 - C21	1.289(6)	C21 - C24	1.505(7)
N1-C6	1.428(6)		
O1 - Cu1 - O2	175.1(1)	O1 - C1 - C2	122.1(4)
O1 - Cu1 - O3	77.5(1)	O1 - C1 - C6	117.2(4)
O1 - Cu1 - N1	85.4(1)	C3 - C4 - C10	120.7(5)
O2 - Cu1 - O3	97.6(1)	C5 - C4 - C10	120.6(5)
O2 - Cu1 - N1	99.5(1)	N1 - C6 - C1	113.5(4)
O3 - Cu1 - N1	162.1(1)	N1 - C6 - C5	129.4(4)
O1 - Cu2 - O3	76.9(1)	N1 - C7 - C8	121.6(4)
O1 - Cu2 - O4	100.3(1)	N1 - C7 - C11	123.3(4)
O1 - Cu2 - N2	158.0(1)	C8 - C7 - C11	115.1(4)
O3 - Cu2 - O4	176.9(1)	O2 - C9 - C8	125.8(4)
O3 - Cu2 - N2	84.7(1)	C8 - C9 - C12	113.7(4)
O4 - Cu2 - N2	97.7(1)	O3 - C13 - C14	120.6(4)
Cu1 - O1 - Cu2	102.8(1)	O3 - C13 - C18	122.6(4)
Cu1 - O1 - C1	113.3(3)	O3 - C13 - C18	116.6(4)
Cu2 - O1 - C1	143.9(3)	C15 - C16 - C22	120.8(4)
Cu1 - O2 - C9	122.0(3)	C17 - C16 - C22	120.6(4)
Cu1 - O3 - Cu2	102.5(1)	N2 - C18 - C13	113.7(3)
Cu1 - O3 - C13	144.1(3)	N2 - C18 - C17	128.8(4)
Cu2 - O3 - C13	112.9(3)	N2 - C19 - C20	121.3(4)
Cu2 - O4 - C21	120.8(3)	N2 - C19 - C23	123.6(4)
Cu1 - N1 - C6	110.6(3)	C20 - C19 - C23	115.1(4)
Cu1 - N1 -C7	121.6(3)	C19 - C20 - C21	128.8(4)
C6 - N1 - C7	127.7(4)	O4 - C21 - C20	125.9(4)
Cu2 - N2 - C18	110.3(3)	O4 - C21 - C24	114.2(4)
Cu2 - N2 - C19	121.8(3)	C20 - C21 - C24	119.9(4)
C18 -N2 - C19	127.4(4)		

References

1. R. J. Butcher and E. Sinn, *Inorg. Chem.*, **15**, 1604 (1975).
2. M. Kato, H. B. Jonassen and J. Fanning, *Chem. Rev.*, **1964**, 99.
3. M. Kato and Y. Muto, *Coord. Chem. Rev.*, **92**, 45 (1988).
4. D. Ülkü, F. Ercan, O. Atakol, I. Ercan and A. Gencer, *Acta Crystallogr.*, **C53**, 79 (1997).
5. M. N. Tahir, D. Ülkü, O. Atakol and M. A. Akay, *Acta Crystallogr.*, **C52**, 2676 (1996).

(Received April 7, 1997)
(Accepted September 8, 1997)