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[*N*,*N*′-Bis(1-{[2-oxido-4-(2-hydroxyethoxy)phenyl]-methyl}ethylidene)ethane-1,2-diamine]copper(II)

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Key indicators

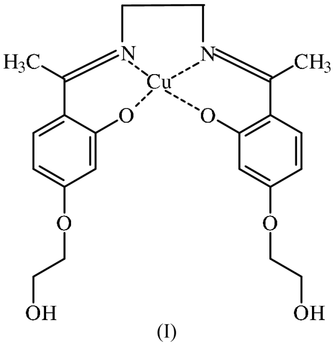
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.108
Data-to-parameter ratio = 16.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title complex, [$Cu(C_{22}H_{26}N_2O_6)$], the coordination geometry about the Cu atom is a 6–5–6 chelate ring structure with a tetrahedrally distorted square plane formed by the four-coordinate N_2O_2 donor set of the Schiff base imine–phenol ligand. The crystal packing is stabilized by intermolecular O—H···O and C—H···O hydrogen bonds.

Comment

Metal derivatives of Schiff bases have been studied extensively, and copper(II) complexes play a major role in both synthesis and structural research (Garnovskii *et al.*, 1993). Copper(II) complexes with N_2O_2 Schiff base ligands derived from salicylaldehyde have long been investigated for their colour isomerism (Llewellyn & Waters, 1960; Hall & Waters, 1960), conformational influences (Clark *et al.*, 1969; Baker *et al.*, 1970*a*,*b*) and their potential as radiopharmaceuticals (John *et al.*, 1994). A neutral and lipophilic Cu^{II} complex has been investigated as a potential Cu tracer for imaging the heart and brain (Green, 1987). Also, neutral and lipophilic complexes of ^{67}Cu with a series of tetradentate Schiff base N_2O_2 ligands have been evaluated in terms of their potential as cerebral blood-flow imaging agents (John *et al.*, 1994). As part of our studies to examine the complexes in this series, we report the crystal structure of the title complex, (I).



(I)

The X-ray analysis reveals that (I) is a monomer with four-coordinate geometry which is consistent with that of copper(II)–imine–phenols with a four-coordinate structure (Baker *et al.*, 1970*a*; Bhadbhade & Srinivas, 1993; Cheeseman *et al.*, 1966; Yao *et al.*, 1997). The coordination of the metal atom is distorted square planar, as indicated by the angles around the Cu atom, which vary from 85.5 (1) (O3—Cu—O4) to 93.6 (1)° (O3—Cu—N1). The Cu—O [1.885 (2) (Cu—O3) and 1.867 (2) Å (Cu—O4)] and Cu—N [1.927 (2) and 1.940 (2) Å] bond lengths are comparable to those in the related reported structures (Baker *et al.*, 1970*a*,*b*; Bhadbhade & Srinivas, 1993; Cheeseman *et al.*, 1966).

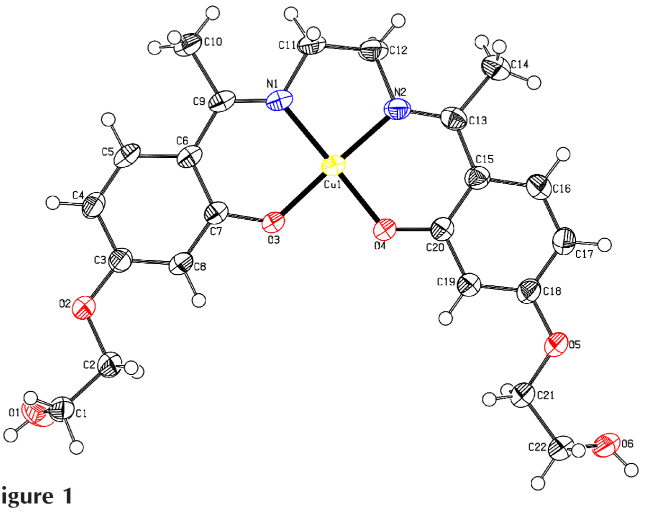


Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

The Cu atom and its equatorial donor atoms are practically coplanar and the metal atom is displaced by 0.033 (1) Å from the mean plane (O3/O4/N1/N2). The salicylideneiminate frame of nine atoms (O4—C20···C13—N2) and its equivalent (O3—C7···C9—N1) are also fairly planar, the largest deviations of the atoms from their corresponding least-squares planes being only 0.068 (3) and 0.032 (2) Å for atoms C13 and C8, respectively. The dihedral angle between these nine-atom planes is 6.1 (1)°. The ethylene bridge C atoms on either side of the CuN plane adopt a *gauche* conformation, as shown by the torsion angle N1—C11—C12—N2 of −36.9 (3)°. The conformations around C1—C2 and C21—C22 are *gauche* [O1—C1—C2—O2 = 71.4 (3)° and O5—C21—C22—O6 = −65.8 (3)°]. In addition to van der Waals interactions, the molecular packing is stabilized by O—H···O and C—H···O intermolecular hydrogen bonds.

Experimental

To ethanol (120 ml) was added *N*,*N*′-bis(1-{[2-hydroxy-4-(2-hydroxyethoxy)phenyl]methyl}ethylidene)ethane-1,2-diamine (4 g, 9.6 mmol), which was prepared by the condensation of a stirred solution of 2-hydroxy-4-(2-hydroxyethoxy)acetophenone (4.9 g, 2.5 mmol) with ethylenediamine (0.75 g, 1.25 mmol) in methanol at room temperature for 12 h. To this mixture, copper(II) acetate (1.9 g, 9.6 mmol) was added and the reaction mixture was refluxed for 8 h. The product, (I), that precipitated was filtered off, washed with ethanol and dried *in vacuo* at 383 K (m.p. 583–585 K); it was crystallized by slow evaporation of a dimethylformamide solution.

Crystal data

$[Cu(C_{22}H_{26}N_2O_6)]$
$M_r = 477.99$
Monoclinic, $P2_1/c$
$a = 11.8535$ (7) Å
$b = 8.6424$ (5) Å
$c = 21.0047$ (13) Å
$\beta = 101.451$ (1)°
$V = 2108.9$ (2) Å^3
$Z = 4$
$D_x = 1.505$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4421 reflections
$\theta = 2.4$–27.2°
$\mu = 1.08$ mm^{-1}
$T = 293$ (2) K
Block, red
0.24 × 0.20 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
12545 measured reflections
4633 independent reflections
3861 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.026$
$\theta_{\rm max} = 28.0°$
$h = -15 \rightarrow 15$
$k = -11 \rightarrow 10$
$l = -27 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.108$
$S = 1.04$
4633 reflections
284 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0651P)^2 + 0.3364P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.36$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.31$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O4	1.867 (2)	O2—C2	1.431 (3)
Cu1—O3	1.885 (2)	O6—C22	1.411 (3)
Cu1—N1	1.927 (2)	O5—C18	1.369 (3)
Cu1—N2	1.940 (2)	O5—C21	1.429 (3)
O3—C7	1.316 (2)	N1—C9	1.306 (3)
N2—C13	1.302 (3)	N1—C11	1.469 (3)
N2—C12	1.471 (3)	O4—C20	1.309 (3)
O2—C3	1.368 (3)	O1—C1	1.410 (3)
O4—Cu1—O3	85.5 (1)	O4—Cu1—N2	92.7 (1)
O4—Cu1—N1	178.8 (1)	O3—Cu1—N2	175.2 (1)
O3—Cu1—N1	93.6 (1)	N1—Cu1—N2	88.3 (1)
O1—C1—C2—O2	71.4 (3)	O5—C21—C22—O6	−65.8 (3)
N1—C11—C12—N2	−36.9 (3)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*B*···O1$^{\rm i}$	0.97	2.52	3.297 (3)	138
O1—H1···O6$^{\rm ii}$	0.82	2.00	2.775 (2)	157
O6—H6···O3$^{\rm iii}$	0.82	1.98	2.783 (2)	168
C22—H22*A*···O4$^{\rm iii}$	0.97	2.43	3.188 (3)	135

Symmetry codes: (i) $1 - x, 1 - y, -z$; (ii) $x - 1, y - 1, z$; (iii) $2 - x, \frac{1}{2} + y, \frac{1}{2} - z$.

The H atoms were positioned geometrically and were treated as riding on their parent C atoms. They were then refined isotropically with aromatic C—H distances of 0.93 Å, methyl C—H distances of 0.96 Å, methylene C—H distances of 0.97 Å and O—H distances of 0.82 Å, and with $U_{\rm iso} = 1.5U_{\rm eq}$(C or O) for methyl and hydroxy H atoms, and $1.2U_{\rm eq}$(C) for other H atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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m1774 G. Usha *et al.* • [Cu($C_{22}H_{26}N_2O_6$)] *Acta Cryst.* (2004). E**60**, m1772–m1774