Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN*. Program(s) used to refine structure: *MolEN*. Molecular graphics: *ORTEP*-3 (Farrugia, 1997). Software used to prepare material for publication: *MolEN*.

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Aqua[N,N'-bis(salicylidene)-1,3-propane-diaminato]copper(II)†

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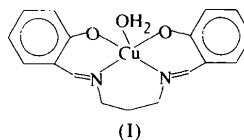
Abstract

The title compound, $[Cu(C_{17}H_{16}N_2O_2)(H_2O)]$, has *m* symmetry. The Cu atom has an approximately square-pyramidal environment involving the two O atoms and two N atoms of the N,N'-bis(salicylidene)-1,3-propane-

† Systematic name: aqua-μ-{2,2'-[1,3-propanediylbis(nitrilomethylidyne)]diphenolato}-1κ^4O,N,N',O'-copper(II).

diaminate ligand and one O atom of the water molecule in the apical position. In the coordination sphere, the O—Cu—N and N—Cu—O(water) angles are 89.6 (1) and 96.7 (1)°, respectively.

Comment

The Schiff base reactions of aldehydes with a symmetrical amino group such as 1,3-propanediamine or ethylenediamine are of interest because of their metal-complexing behaviour. The structure of the Schiff base complex aqua[N,N'-bis(salicylidene)-1,3-propanediamine]nickel(II) has been determined previously (Elerman *et al.*, 1993). Nickel complexes of the diamine Schiff bases generally have square-planar coordination (Akhtar & Drew, 1982; Manfredotti & Guastini, 1983; Radha *et al.*, 1985; Drew *et al.*, 1985). The title copper complex, aqua[N,N'-bis(salicylidene)-1,3-propanediaminato]copper(II), (I), has the same environment.

(I)

The coordination sphere around the Cu atom in (I) is a square pyramid (Fig. 1). The copper ion is coordinated with the four equatorial positions occupied by the two N and two O atoms of the N,N'-bis(salicylidene)-1,3-propanediaminate ligand $(SALPD^{2-})$. The Cu atom is located 0.157 (1) Å from the coordination best plane (N1, N1', O1 and O1'). The axial position of the square-pyramidal polyhedron is occupied by the O atom of the water molecule. The displacement of the O2 atom from the basal coordination plane is 2.472 (1) Å.

The metal complex has *m* symmetry. The $SALPD^{2-}$ ligand is not planar. The chelate ring composed of atoms Cu1, N1, C8, C9, C8' and N1' has a chair conformation. The distances of the two *para*-positioned
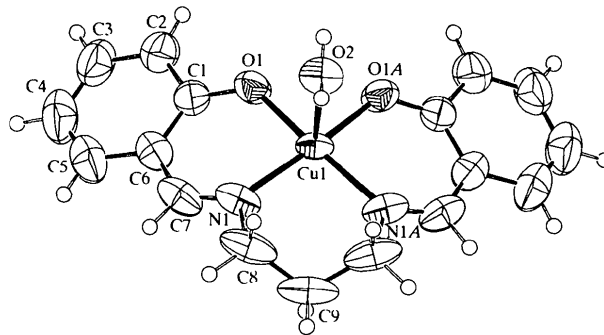

Fig. 1. *PLATON*99 (Spek, 1999) drawing of (I) with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small circles of arbitrary radii.

chair atoms, Cu1 and C9, from the best plane of the six atoms are 0.004 (1) and 0.358 (4) Å, respectively. The value of the dihedral angle formed by the coordination basal plane and the propane bridge is 46.0 (3)°. In the Cu^{2+} coordination sphere, the Cu1—O1(SALPD^{2-}) bond length [1.939 (2) Å] is shorter than the Cu1—O2(aqua) distance [2.309 (3) Å]. Within the coordination square-pyramidal polyhedron, the maximum deviation of the bond angles from 90° is 6.73 (2)°. The hydrogen bonds of the water molecule and the propane bridge contribute to the molecular packing. Details of the hydrogen-bonding geometry are given in Table 2.

Experimental

To a solution of N,N'-bis(salicylidene)-1,3-propanediamine (2.82 g, 10 mmol) in hot ethanol (50 ml), ammonia solution (10 ml, 20%) was added. A solution of $CuCl_2 \cdot 2H_2O$ (1.71 g, 10 mmol) in hot water (20 ml) was added and the resulting mixture was set aside for 1–2 h. The green copper complex was filtered off and the green crystals obtained dissolved in a mixture of hot EtOH (100 ml) and hot MeCN (20 ml). Hot water (30 ml) was added to the mixture. The final mixture was set aside for 2 d, after which time part of the complex had recrystallized as needle-shaped [Cu(C$_{17}$H$_{16}$N$_2$O$_2$)]. The rest of the complex crystallized as light-green [Cu(C$_{17}$H$_{16}$N$_2$O$_2$)-(H$_2$O)]. These crystals were filtered off and dried in air.

Crystal data

[Cu(C$_{17}$H$_{16}$N$_2$O$_2$)(H$_2$O)]	Mo $K\alpha$ radiation
$M_r = 361.89$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 15
Pbnm	reflections
$a = 7.5352 (11)$ Å	$\theta = 9.90–11.13°$
$b = 9.2242 (12)$ Å	$\mu = 1.375$ mm^{-1}
$c = 23.1197 (13)$ Å	$T = 293 (2)$ K
$V = 1607.0 (3)$ Å^3	Prismatic
$Z = 4$	$0.35 \times 0.20 \times 0.15$ mm
$D_x = 1.496$ Mg m^{-3}	Light green
D_m not measured	

Data collection

Nonius CAD-4 diffractom-	1296 reflections with
eter	$I > 2\sigma(I)$
$\omega/2\theta$ scans	$\theta_{max} = 26.28°$
Absorption correction:	$h = -9 \rightarrow 0$
ψ scan (Fair, 1990)	$k = -11 \rightarrow 0$
$T_{min} = 0.645$, $T_{max} = 0.820$	$l = 0 \rightarrow 28$
1671 measured reflections	3 standard reflections
1671 independent reflections	frequency: 120 min
	intensity decay: 0.002%

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.381$ e Å^{-3}
$R(F) = 0.046$	$\Delta\rho_{min} = -0.630$ e Å^{-3}
$wR(F^2) = 0.063$	Extinction correction:
$S = 1.07$	SHELXL93 (Sheldrick,
1296 reflections	1993)
114 parameters	Extinction coefficient:
H atoms constrained	0.0047 (13)

$w = 1/[\sigma^2(F_o^2) + (0.0897P)^2 + 0.2277P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.0006$

Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Cu1—O1	1.939 (2)	N1—C8	1.483 (4)
Cu1—N1	2.005 (3)	O1—C1	1.293 (3)
Cu1—O2	2.309 (3)	C8—C9	1.502 (5)
N1—C7	1.261 (5)		
O1—Cu1—O1^i	83.3 (1)	C7—N1—Cu1	124.5 (2)
O1—Cu1—N1^i	168.9 (1)	C8—N1—Cu1	119.6 (3)
O1—Cu1—N1	89.6 (1)	C1—O1—Cu1	129.7 (2)
N1^i—Cu1—N1	96.0 (2)	O1—C1—C6	123.7 (3)
O1—Cu1—O2	92.2 (1)	N1—C7—C6	128.0 (3)
N1—Cu1—O2	96.7 (1)	N1—C8—C9	112.7 (3)
C7—N1—C8	115.9 (3)		

Symmetry code: (i) $x, y, \frac{1}{2} - z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H22···O1^i	0.72	2.16	2.773 (3)	142
C8—H82···O1^i	0.97	2.52	3.438 (4)	157

Symmetry code: (i) $\frac{1}{2} - x, y - \frac{1}{2}, z$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993). Data reduction: *X-RED* (Stoe & Cie, 1996). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Software used to prepare material for publication: *SHELXL93*.

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