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{3-[(*E*)-(5-Bromo-2-oxidophenyl)methylamino]propanoato-κ^3*O*,*N*,*O*′}(1,10-phenanthroline-κ^2*N*,*N*′)copper(II) dihydrate

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Key indicators

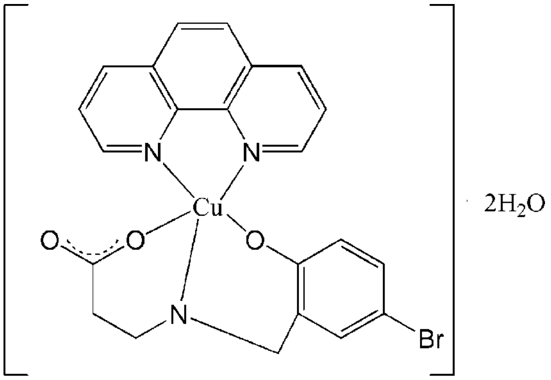
Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.014 Å
R factor = 0.057
wR factor = 0.122
Data-to-parameter ratio = 11.7

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

In the title complex, [Cu(L)(phen)]·2H_2O, {LH_2 = 3-[(*E*)-(5-bromo-2-oxidophenyl)methyleneamino]propanoic acid ($C_{10}H_{10}BrNO_3$) and phen = 1,10-phenanthroline ($C_{12}H_8N_2$)}, the Cu^{II} atom is coordinated in a slightly distorted square-pyramidal geometry by two O atoms and one N atom from an L^{2-} ligand, and two N atoms from a phen ligand. One of the carboxylate O atoms is in the apical position. In the crystal structure, a two-dimensional supramolecular network is formed through intermolecular N—H···O and O—H···O hydrogen bonds.

Comment

Over the past few decades, significant progress has been made in understanding the coordination chemistry of the copper(II) complexes of various Schiff base ligands (Casella & Gullotti, 1981; Wagner & Walker, 1983; Xiao *et al.*, 2006). Most model studies of metal complexes of Schiff base ligands containing salicylaldehyde and amino acids have focused on the binding mode of these ligands (Nakagima *et al.* 1989, Kettmann *et al.*, 1993). Crystal structures of the complexes obtained demonstrate that the Schiff base ligands act in a tridentate mode, coordinating through the phenolate O, imine N and carboxylate O atoms. Our research group is interested in tridentate reduced Schiff bases, as they are more flexible because the C═N bond of the Schiff base has been reduced, and this helps to overcome the ligand instability.



(I)

The molecular structure of (I) is shown in Fig. 1 and selected bond lengths and angles are given in Table 1. The Cu^{II} atom is coordinated by two O atoms and one N atom from an L^{2-} ligand and two N atoms from a phen ligand with atom O2 in the apical position, giving a slightly distorted square-pyramidal geometry [LH_2 = 3-(5-bromo-2-hydroxybenzylamino)propionic acid and phen = 1,10-phenanthroline]. In the

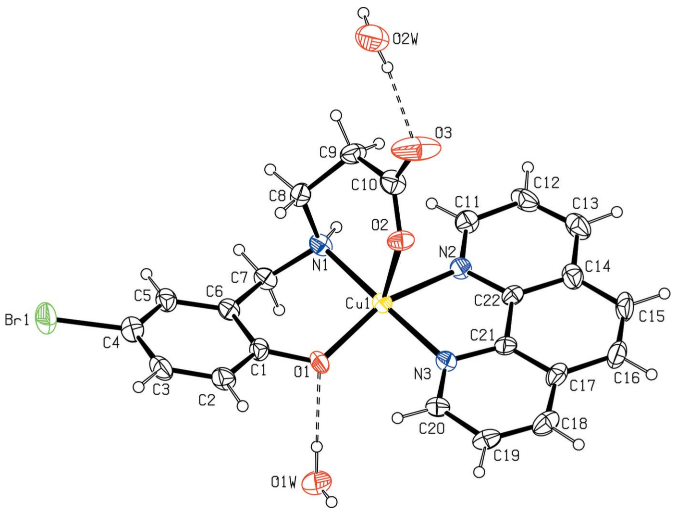


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids. The dashed lines indicate hydrogen bonds.

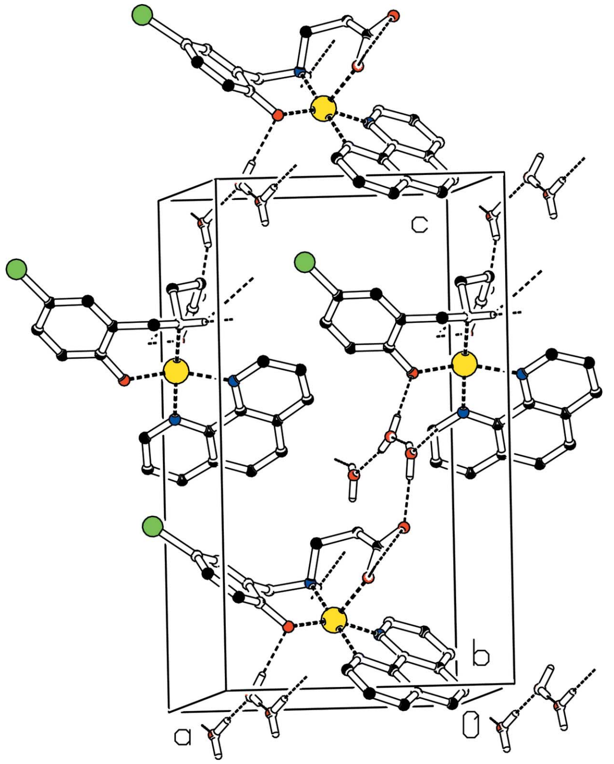


Figure 2
Part of the crystal structure of (I), showing hydrogen bonds as dashed lines. Only H atoms included in the hydrogen bonds are shown.

crystal structure, a two-dimensional network is formed *via* intermolecular N—H···O and O—H···O hydrogen bonds (Table 2 and Fig. 2).

Experimental

A solution of 3-aminopropionic acid (2 mmol, 0.178 g) and potassium hydroxide (2 mmol, 0.112 g) in distilled water (20 ml) was slowly added to a solution of 5-bromo-2-hydroxybenzaldehyde (2 mmol, 0.404 g) in methanol (10 ml). The mixture was stirred for 30 min at room temperature, then the solution was added to solid sodium borohydride (2 mmol, 0.076 g) and stirred for 2 h until the yellow solution became colourless. This solution was slowly added to a solution of copper(II) nitrate (1 mmol, 0.291 g) in distilled water (10 ml). The mixture was stirred and refluxed for 4 h at room temperature. Phen (2 mmol, 0.36 g) was added and the reaction continued for a further 2 h. The solution was filtered and the filtrate was left to stand at room temperature. Blue prisms suitable for X-ray diffraction were obtained in a yield of 66% (based on copper nitrate). Analysis found: C 47.69, H 4.23, N 7.57, Cu 11.49%; $C_{22}H_{22}BrCuN_3O_5$ requires: C 47.88, H 4.02, N 7.61, Cu 11.52%.

Crystal data

$[Cu(C_{10}H_{10}BrNO_3)(C_{12}H_8N_2)]\cdot 2H_2O$
$M_r = 551.88$
Monoclinic, Pn
$a = 11.120$ (2) Å
$b = 5.3808$ (11) Å
$c = 18.393$ (4) Å
$\beta = 93.47$ (3)°
$V = 1098.5$ (4) Å^3
$Z = 2$
$D_x = 1.668$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 2.85$ mm^{-1}
$T = 293$ (2) K
Prism, blue
0.18 × 0.08 × 0.06 mm

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.628$, $T_{max} = 0.848$
5630 measured reflections
3605 independent reflections
2695 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 26.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.122$
$S = 0.97$
3605 reflections
309 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0239P)^2 + 4.2874P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.007$
$\Delta\rho_{max} = 0.77$ e Å^{-3}
$\Delta\rho_{min} = -0.62$ e Å^{-3}
Absolute structure: Flack (1983), 1462 Friedel pairs
Flack parameter: 0.00 (1)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	1.01 (9)	1.88 (9)	2.841 (9)	157 (7)
N1—H1A···O3^{i}	1.01 (9)	2.54 (9)	3.349 (12)	136 (6)
O1W—H1WB···O1	0.84 (6)	1.96 (7)	2.800 (9)	178 (9)
O1W—H1WA···O2W^{ii}	0.84 (6)	2.03 (5)	2.865 (13)	172 (18)
O2W—H2WA···O1W^{iii}	0.84 (6)	2.12 (5)	2.926 (13)	162 (6)
O2W—H2WB···O3	0.84 (7)	1.88 (3)	2.667 (13)	157 (8)

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

Water H atoms and the amido H atom were located in difference Fourier maps. They were refined isotropically but a distance restraint [0.84 (1) Å] was applied to the O—H distances. All other H atoms were positioned geometrically and were treated as riding atoms, with C—H = 0.93–0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Bruker, 2001).

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