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#### Key indicators

Single-crystal X-ray study  
T = 293 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.008 \text{ \AA}$   
R factor = 0.052  
wR factor = 0.123  
Data-to-parameter ratio = 16.7

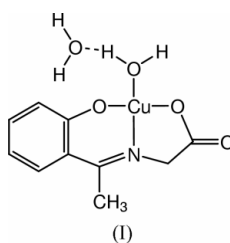
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

## Aqua[[[2-(2-hydroxyphenyl)ethylidene]amino]-acetato]copper(II) monohydrate

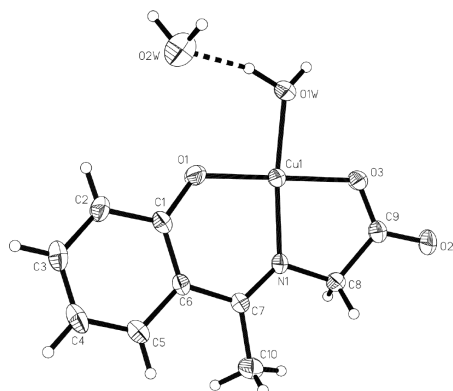
In the crystal structure of the title complex,  $[\text{Cu}(\text{C}_{10}\text{H}_9\text{NO}_3)(\text{H}_2\text{O})]\cdot\text{H}_2\text{O}$ , the Cu atom is tetracoordinated by O and N atoms, and facilitates an intermolecular  $\text{Cu}\cdots\text{O}$  short contact of  $2.809(3) \text{ \AA}$ . The two symmetrically-related coordinated Cu ions form distorted square pyramids of coordination  $\text{CuO}_4\text{N}$ . In the asymmetric unit, a free water molecule is hydrogen bonded to the coordinated water molecule. In the packing, the molecules are interconnected by two types of  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds.

#### Comment

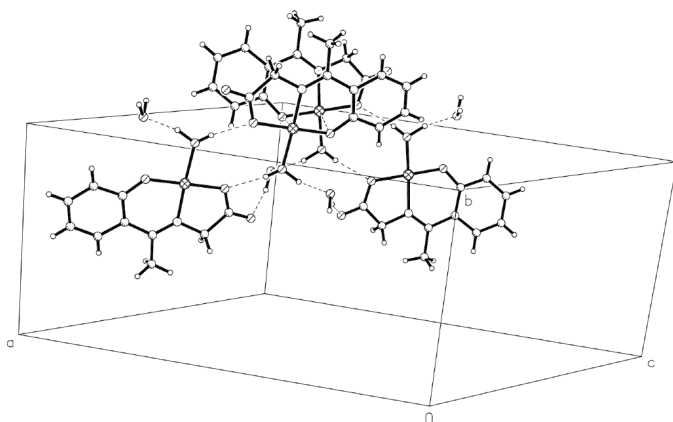
Considerable attention has been paid to the structures of Cu complexes with Schiff bases formed from salicylaldehyde-amino acid (Ueki *et al.*, 1967, 1969; Bkouche-Waksman *et al.*, 1988). These complexes are expected to be catalytic intermediates in non-enzymatic transamination reactions (Eichhorn & Marchand, 1956; Longenecker & Snell, 1957). Recently, X-ray structural studies on tin complexes with such ligands exhibited a variety of coordination and bonding for the tin<sup>IV</sup> ion (Dakternieks *et al.*, 1998; Basu Baul *et al.*, 1999, 2001, 2002). The usefulness of such systems has, in turn, prompted research into the chemistry of Cu, since the bond distances involving the N atoms are of interest. In this study, we report the X-ray structural analysis of the title complex, (I).



The asymmetric unit comprises a ligand-coordinated  $\text{Cu}^{\text{II}}$  cation, a coordinated water molecule, and a free water molecule. The Cu atom is tetracoordinated by two O atoms and one N atom from the *O,N,O*-tridentate ligand and by one O atom of a water molecule. The Cu—O and Cu—N bond distances are in the range  $1.881(3)$ – $1.959(3) \text{ \AA}$ , in agreement with reported values [ $1.892(2)$ – $1.993(2) \text{ \AA}$ ; Warda, 1997*a,b*; Warda *et al.*, 1997; Warda, 1998*a,b*]. Atom Cu1 also facilitates an intermolecular  $\text{Cu}\cdots\text{O}1^i$  short contact (Table 1), giving a distorted square pyramidal  $\text{CuO}_4\text{N}$  coordination with  $\text{O}1^i$  occupying the apical position. The basal plane is formed by  $\text{Cu}1/\text{O}1/\text{N}1/\text{O}3/\text{O}1\text{W}$ . The bond angles around Cu1 are listed in Table 1.



**Figure 1**  
The asymmetric unit of the title compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.



**Figure 2**  
Packing diagram of (I), showing the links via O—H...O hydrogen bonds.

The ligand and the atom Cu1 form two nearly planar rings, one five-membered [ring A: Cu1—O3—C9—C8—N1;  $Q_2$  puckering amplitude (Cremer & Pople, 1975) 0.090 (5) Å] and the other six-membered [ring B: Cu1—O1—C1—C6—C7—N1;  $Q_7$  puckering amplitude 0.184 (4) Å]. These two rings adopt envelope conformations, with atoms C8 and Cu1 deviating by 0.134 (1) Å and 0.222 (1) Å from the N1/Cu1/O3/C9 and O1/C1/C6/C7/N1 planes, respectively. The six-membered, five-membered and aromatic (C) rings are nearly coplanar, with ring B making dihedral angles of 7.1 (2)° and 4.8 (2)° with A and C, respectively.

In the asymmetric unit, the Cu<sup>II</sup> complex cation and the free water molecule are interconnected by O1W—H1W1...O2W hydrogen bonds. In the crystal packing, the two water molecules also play an important role in the intermolecular hydrogen-bonding scheme, in which there are two types of intermolecular O—H...O bonds, viz. O1W—H2W1...O3<sup>ii</sup> and O2W—H2W2...O2<sup>iii</sup> (Table 2 and Fig. 2). Two molecules are interconnected by the two intermolecular hydrogen bonds into molecular chains.

## Experimental

A methanolic solution of potassium ((2-(2-hydroxyphenyl)ethylidene)amino)acetate (Dakternieks *et al.*, 1998) was added dropwise to a stirred aqueous solution of Cu(OAc)<sub>2</sub>·H<sub>2</sub>O containing a few

drops of acetic acid (1:1). The filtered solution, upon slow evaporation, afforded green crystals of the title compound (I). M.p. 520 (1) K, analysis found: C 41.27, H. 4.47, N 4.80%; calc. for C<sub>10</sub>H<sub>13</sub>NO<sub>5</sub>Cu: C 41.31, H. 4.51, N 4.82%; IR (KBr): 1630 ν(OCO), 1599 ν(C=N), 1237 ν[Ph(C=O)].

## Crystal data

[Cu(C<sub>10</sub>H<sub>11</sub>NO<sub>4</sub>)]H<sub>2</sub>O  
 $M_r = 290.75$   
 Monoclinic, C2/c  
 $a = 20.2250$  (3) Å  
 $b = 10.6741$  (3) Å  
 $c = 14.2089$  (3) Å  
 $\beta = 134.294$  (1)°  
 $V = 2195.59$  (9) Å<sup>3</sup>  
 $Z = 8$

$D_x = 1.759$  Mg m<sup>-3</sup>  
 Mo K $\alpha$  radiation  
 Cell parameters from 3297 reflections  
 $\theta = 2.8$ –28.3°  
 $\mu = 2.00$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Block, green  
 0.20 × 0.12 × 0.10 mm

## Data collection

Siemens SMART CCD area-detector diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.691$ ,  $T_{\max} = 0.825$   
 7657 measured reflections

2718 independent reflections  
 1739 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.080$   
 $\theta_{\text{max}} = 28.3^\circ$   
 $h = -26 \rightarrow 20$   
 $k = -12 \rightarrow 14$   
 $l = -14 \rightarrow 18$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.052$   
 $wR(F^2) = 0.123$   
 $S = 0.95$   
 2718 reflections  
 163 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0475P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.97$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.73$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Cu1—O1	1.881 (3)	Cu1—O1W	1.959 (3)
Cu1—N1	1.936 (3)	Cu1—O1 <sup>i</sup>	2.809 (3)
Cu1—O3	1.948 (3)		
O1—Cu1—N1	93.9 (1)	O3—Cu1—O1W	88.6 (1)
O1—Cu1—O3	179.0 (1)	O1—Cu1—O1 <sup>i</sup>	78.3 (1)
N1—Cu1—O3	86.0 (1)	N1—Cu1—O1 <sup>i</sup>	101.4 (2)
O1—Cu1—O1W	91.7 (1)	O3—Cu1—O1 <sup>i</sup>	100.8 (2)
N1—Cu1—O1W	170.9 (1)	O1W—Cu1—O1 <sup>i</sup>	86.7 (1)

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
O1W—H1W1...O2W	1.02 (9)	1.65 (9)	2.649 (9)	164 (7)
O1W—H2W1...O3 <sup>ii</sup>	0.83 (7)	2.02 (7)	2.850 (8)	172 (6)
O2W—H2W2...O2 <sup>iii</sup>	1.04	1.82	2.838 (5)	166

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

All H atoms attached to C were positioned geometrically, with C—H 0.93–0.97 Å. Though the H atoms attached to O1W were calculated and refined isotropically, with OW—H 0.84 (6)–1.02 (7) Å, those attached to O2W were located in a difference map, with OW—H 1.04 Å and treated as riding atoms.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 1997); program(s) used to refine

structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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## References

- Basu Baul, T. S., Dutta, S., Rivarola, E., Butcher, R. & Smith, F. E. (2002). *J. Organomet. Chem.* **654**, 100–108.
- Basu Baul, T. S., Dutta, S., Rivarola, E., Scopelliti, M. & Chaudhuri, S. (2001). *Appl. Organomet. Chem.* **15**, 947–953.
- Basu Baul, T. S., Dutta, S. & Tiekink, E. R. T. (1999). *Z. Kristallogr.* **214**, 361–362.
- Bkouche-Waksman, L., Barbe, J. M. & Kvic, A. (1988). *Acta Cryst.* **B44**, 595–601.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Dakternieks, D., Basu Baul, T. S., Dutta, S. & Tiekink, E. R. T. (1998). *Organometallics*, **17**, 3058–3062.
- Eichhorn, G. L. & Marchand, N. D. (1956). *J. Am. Chem. Soc.* **78**, 2688–2691.
- Longenecker, J. B. & Snell, E. E. (1957). *J. Am. Chem. Soc.* **79**, 142–145.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS, Inc., Madison, Wisconsin, USA.
- Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Systems, Inc., Madison, Wisconsin, USA.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Ueki, T., Ashida, T., Sasada, Y. & Kakudo, M. (1967). *Acta Cryst.* **22**, 870–878.
- Ueki, T., Ashida, T., Sasada, Y. & Kakudo, M. (1969). *Acta Cryst.* **B25**, 328–335.
- Warda, S. A. (1997a). *Acta Cryst.* **C53**, 1588–1590.
- Warda, S. A. (1997b). *Acta Cryst.* **C53**, 1590–1593.
- Warda, S. A. (1998a). *Acta Cryst.* **C54**, 189–191.
- Warda, S. A. (1998b). *Acta Cryst.* **C54**, 302–304.
- Warda, S. A., Friebe, C., Sívý, J., Plesch, G. & Báhová, M. (1997). *Acta Cryst.* **C53**, 50–54.