

Aqua(pyridine- κN)(*N*-salicylidene-glycinato- $\kappa^3 N, O, O'$)copper(II)Yu-Guang Li,^{a,b} Hai-Liang Zhu,^{a*}
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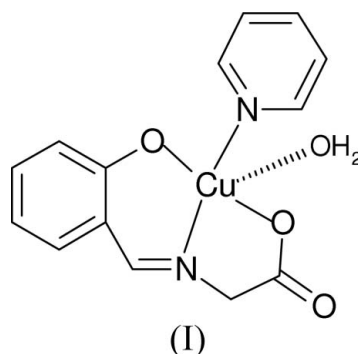
Key indicators

Single-crystal X-ray study
 $T = 295$ K
Mean $\sigma(\text{C}-\text{C}) = 0.011$ Å
 R factor = 0.041
 wR factor = 0.137
Data-to-parameter ratio = 15.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In both of the two independent molecules of the title compound, $[\text{Cu}(\text{C}_9\text{H}_7\text{NO}_3)(\text{C}_5\text{H}_5\text{N})(\text{H}_2\text{O})]$, the copper atom is in a square-pyramidal coordination with the apical site occupied by water. The two molecules are related by a non-crystallographic center of inversion. Hydrogen bonds link symmetry-equivalent molecules into zigzag chains that run along the a axis of the orthorhombic unit cell.

Comment

Schiff bases that are derived from the condensation of salicylaldehyde and amino-acids behave as N, O, O' -tridentate dianions in a large number of transition metal complexes. For copper(II), in particular, its *N*-salicylidene-glycinates are Lewis bases that accept ligands such as pyridines into their coordination sphere, as exemplified by (*N*-salicylidene-glycinato)pyridinecopper(II) (Butcher *et al.*, 2003). Pyridine adducts that also contain coordinated water are also known; crystallographically authenticated examples include the 2-methylpyridine (Warda, 1998*d*), 2-ethylpyridine (Warda, 1998*c*), 2,3-dimethylpyridine (Warda, 1998*a*) and 2,4-dimethylpyridine (Warda, 1998*b*) adducts. The anhydrous parent compound itself exists as a five-coordinate, square-pyramidal complex with a polymeric chain structure in which the double-bonded O atom of the dianion engages in binding to an adjacent metal center (Butcher *et al.*, 2003). The aquapyridine adducts also have the metal center in such an environment; additionally, hydrogen bonds are present in their crystal structures. The title aquapyridine homolog (Scheme I) displays chelation similar to that noted in these adducts.



The two independent molecules are related by a local, non-crystallographic center of inversion with $x \sim \frac{1}{8}$ and $y \sim \frac{1}{4}$; such local centers are often found in space groups $Pca2_1$ and $Pna2_1$ (Marsh *et al.*, 1998). As such, the structural differences between the two molecules in this compound are only marginal.

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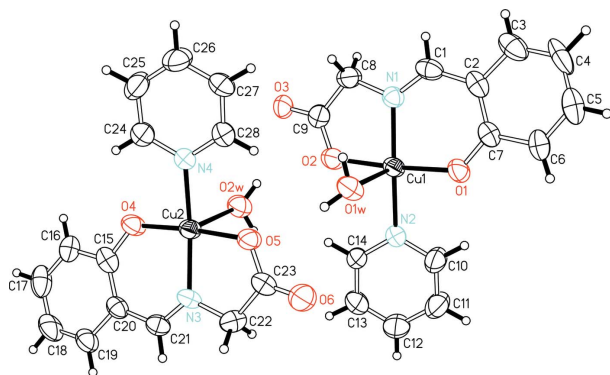


Figure 1
ORTEP (Johnson, 1976) plot of the two independent molecules of $[\text{Cu}(\text{C}_9\text{H}_7\text{NO}_3)(\text{C}_5\text{H}_5\text{N})(\text{H}_2\text{O})]$. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

A strong hydrogen bond between between the water molecule and the carbonyl oxygen atom of an adjacent, symmetry-related molecule leads to the formation of zigzag chains; all chains in the unit cell run in the same direction, along the a axis. Weaker hydrogen bonds (Table 2) link the chains into layers.

Experimental

Glycine (0.15 g, 2 mmol) and salicylaldehyde (0.24 g, 2 mmol) were dissolved in water (10 ml); the mixture turned yellow after an hour of stirring to yield *N*-salicylideneglycine. Copper(II) acetate hydrate (0.20 g, 1 mmol) dissolved in water (10 ml) was added, followed by an excess of pyridine (1 ml). Deep-blue prisms separated from solution after two weeks in about 85% yield. CH&N analysis. Found: C 61.51, H 5.2, N 10.15%; calc. for $\text{C}_{14}\text{H}_{14}\text{CuN}_2\text{O}_4$: C 61.30, H 5.15, N 10.21%.

Crystal data

$[\text{Cu}(\text{C}_9\text{H}_7\text{NO}_3)(\text{C}_5\text{H}_5\text{N})(\text{H}_2\text{O})]$	Mo $K\alpha$ radiation
$M_r = 337.81$	Cell parameters from 4366 reflections
Orthorhombic, $Pca2_1$	$\theta = 2.8\text{--}24.9^\circ$
$a = 9.6232(5) \text{ \AA}$	$\mu = 1.56 \text{ mm}^{-1}$
$b = 10.8138(6) \text{ \AA}$	$T = 295(2) \text{ K}$
$c = 27.219(2) \text{ \AA}$	Prism, deep blue
$V = 2832.4(3) \text{ \AA}^3$	$0.30 \times 0.20 \times 0.08 \text{ mm}$
$Z = 8$	
$D_x = 1.584 \text{ Mg m}^{-3}$	

Data collection

Bruker APEX area-detector diffractometer	6066 independent reflections
φ and ω scans	4788 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.032$
$T_{\text{min}} = 0.660$, $T_{\text{max}} = 0.885$	$\theta_{\text{max}} = 27.5^\circ$
16774 measured reflections	$h = -12 \rightarrow 11$
	$k = -13 \rightarrow 12$
	$l = -30 \rightarrow 35$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0839P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.137$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.48 \text{ e \AA}^{-3}$
6066 reflections	$\Delta\rho_{\text{min}} = -0.40 \text{ e \AA}^{-3}$
396 parameters	Absolute structure: Flack (1983),
H atoms treated by a mixture of independent and constrained refinement	3294 Friedel pairs
	Flack parameter: 0.48 (2)

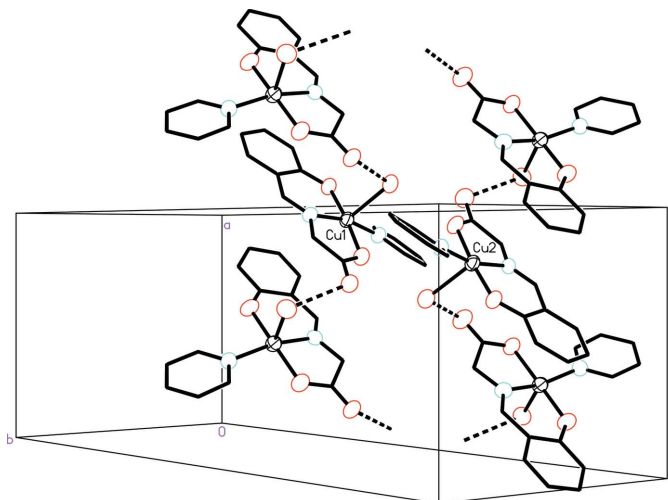


Figure 2
Schematic representation of the hydrogen-bonded (dashed lines) chain motif. H atoms have been omitted.

Table 1

Selected geometric parameters (\AA , $^\circ$).

Cu1—O1	1.924 (4)	Cu2—O4	1.908 (4)
Cu1—O2	1.977 (4)	Cu2—O5	1.987 (4)
Cu1—O1 _w	2.396 (4)	Cu2—O2 _w	2.393 (4)
Cu1—N1	1.948 (4)	Cu2—N3	1.934 (4)
Cu1—N2	2.017 (4)	Cu2—N4	2.015 (4)
O1—Cu1—O1 _w	95.3 (2)	O4—Cu2—O5	174.6 (2)
O1—Cu1—O2	174.3 (2)	O4—Cu2—N3	92.6 (3)
O1—Cu1—N2	91.6 (2)	O4—Cu2—N4	92.2 (2)
O1—Cu1—N1	92.3 (3)	O4—Cu2—O2 _w	94.9 (2)
O2—Cu1—O1 _w	89.4 (2)	O5—Cu2—O2 _w	89.5 (2)
O2—Cu1—N2	91.6 (2)	O5—Cu2—N3	83.1 (3)
O2—Cu1—N1	83.5 (3)	O5—Cu2—N4	91.1 (2)
O1 _w —Cu1—N1	102.5 (3)	O2 _w —Cu2—N3	104.7 (3)
O1 _w —Cu1—N2	90.2 (2)	O2 _w —Cu2—N4	89.7 (2)
N1—Cu1—N2	166.3 (3)	N3—Cu2—N4	164.3 (2)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
O1 _w —H1 _w 1...O3 ⁱ	0.85 (1)	1.95 (3)	2.749 (6)	155 (5)
O1 _w —H1 _w 2...O5	0.85 (1)	2.46 (5)	3.114 (6)	134 (6)
O1 _w —H1 _w 2...O6	0.85 (1)	2.41 (3)	3.169 (6)	149 (5)
O2 _w —H2 _w 2...O2	0.85 (1)	2.39 (3)	3.121 (6)	145 (5)
O2 _w —H2 _w 2...O3	0.85 (1)	2.39 (2)	3.169 (7)	153 (4)
O2 _w —H2 _w 1...O6 ⁱⁱ	0.85 (1)	1.97 (3)	2.744 (6)	151 (5)

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

The structure is an inversion twin with essentially equal components. The carbon-bound H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93 and 0.97 \AA , and with $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The water H atoms were located and refined with distance restraints of O—H = 0.85 (1) and H...H = 1.39 (1) \AA .

Data collection: SMART (Bruker, 2003); cell refinement: SAINT (Bruker, 2003); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP (Johnson, 1976); software used to prepare material for publication: SHELXL97.

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