metal-organic papers

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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.005 Å
R factor = 0.046
wR factor = 0.125
Data-to-parameter ratio = 13.7

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

Bis(μ₂-N-5-formysalicylideneglycinato-κ⁴O,N,O′:O′)bis[aquacopper(II)]

In the title controsymmetric dimeric copper(II) compound, $[Cu_2(C_{10}H_7NO_4)_2(H_2O)_2]$, the Cu^{II} ion is five-coordinated in a square-pyramidal configuration, with one imine N atom, one phenolate O atom, one carboxylate O atom of the Schiff base and one O atom of a coordinated water molecule defining the basal plane, and the phenolate O atom of another Schiff base occupying the apical position. The Cu^{II} atoms are bridged by two phenolic O atoms, with a Cu···Cu distance of 3.250 (4) Å. In the crystal structure, the molecules are linked through intermolecular O—H···O hydrogen bonds, forming chains running along the b axis.

Comment

Metal complexes with Schiff bases as ligands have played an important part in the development of inorganic chemistry as a whole. The research field dealing with Schiff base metal complexes has expanded enormously, and embraces wide and diverse subjects comprising vast numbers of organometallic compounds and various aspects of biocoordination chemistry (Kono & Fridovich, 1983; Wu *et al.*, 2001; May *et al.*, 2004). In addition, transition metal complexes of salicylaldehyde–amino acid Schiff bases are found to behave analogously to those of pyridoxal–amino acid Schiff bases (Snell *et al.*, 1963). However, complexes related to the 5-formylsalicylaldehyde derivative have seldom been reported. We therefore focused our attention on the assembly of transition metal ions with flexible ligands. We report here the structure of the new title Cu^{II} compound, (I).
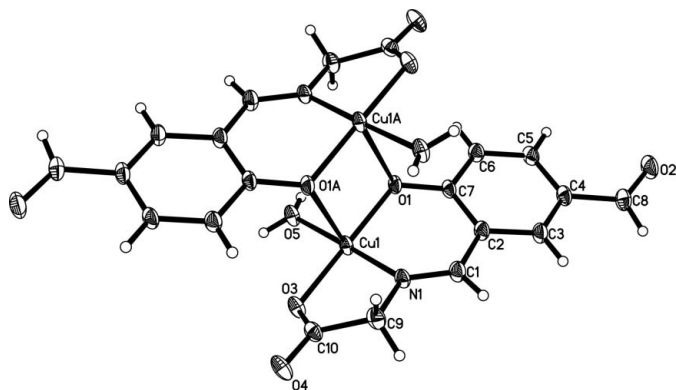

(I)
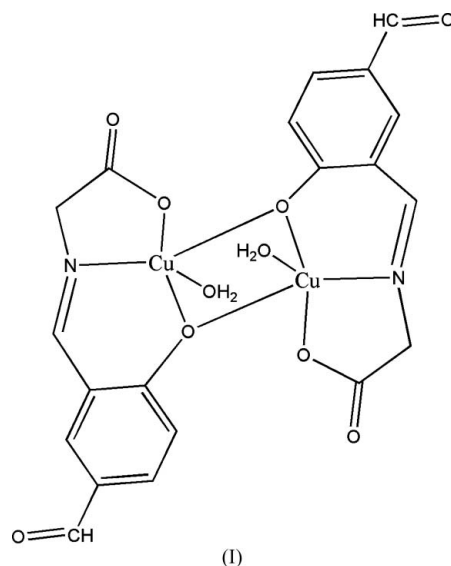
Figure 1
The dimeric structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. [Symmetry code: (A) $-x, y + \frac{1}{2}, -z + \frac{1}{2}$.]
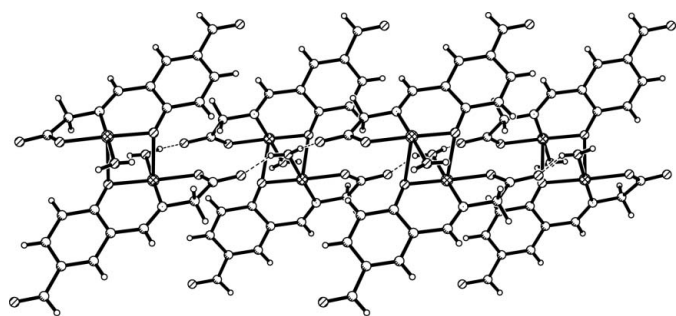

Figure 2
A fragment of the crystal structure of (I), showing the polymeric hydrogen-bonded chain (dashed lines) of Cu complexes.

The crystal structure of (I) consists of dimers, with the Cu^{II} atoms having square-pyramidal coordination (Fig. 1). The Cu^{II} atoms are bridged by two phenolic O atoms, with a Cu···Cu distance of 3.250 (4) Å. In a distorted square-pyramidal geometry around each Cu^{II} atom, the basal plane is formed by two O atoms and one N atom of the 5-formylsalicylidene-glycinate anion and one water O atom. The apical site is occupied by a bridging phenolic O atom from a neighbouring monomer unit, with a Cu—O(apical) bond distance of 2.589 (4) Å. The Cu—O and Cu—N bond lengths (Table 1) are comparable with the corresponding values observed in other Schiff base Cu^{II} complexes (Davies, 1984; Warda, 1998; Marsh & Spek, 2001; Valent *et al.*, 2002).

In the crystal structure, adjacent dimers are linked through intermolecular O—H···O hydrogen bonds (Table 2), forming chains running along the *b* axis (Fig. 2).

Experimental

All reagents were of commercially available grade and were used without further purification. 5-Formylsalicylaldehyde (0.1 mmol, 0.134 g), glycine (0.2 mmol, 0.15 g) and KOH (0.2 mmol, 0.112 g) were dissolved in aqueous methanol (80%; 20 ml). The mixture was stirred at room temperature for 10 min to give a clear yellow solution. To this solution was added an aqueous solution (10 ml) of CuSO$_4$·5H$_2$O (0.2 mmol, 0.500 g), with stirring. The mixture was stirred and refluxed at 323 K for 6 h, then cooled to room temperature. After filtration, the filtrate was left to stand at room temperature. Green crystals of (I) suitable for X-ray diffraction were obtained in a yield of 48%. Analysis, found: C 41.92, H 3.18, N 4.92%; C$_{20}$H$_{18}$N$_2$O$_{10}$Cu$_2$ requires: C 41.96, H 3.17, N 4.90%.

Crystal data

[Cu$_2$(C$_{10}$H$_7$NO$_4$)$_2$(H$_2$O)$_2$]	$Z = 2$
$M_r = 573.44$	$D_x = 1.853$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 9.281$ (16) Å	$\mu = 2.13$ mm^{-1}
$b = 9.060$ (15) Å	$T = 293$ (2) K
$c = 12.62$ (2) Å	Block, green
$\beta = 104.495$ (19)°	$0.30 \times 0.20 \times 0.20$ mm
$V = 1028$ (3) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	4900 measured reflections
	2221 independent reflections
φ and ω scans	1877 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.058$
	$\theta_{max} = 27.0°$
$T_{min} = 0.567$, $T_{max} = 0.675$	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.046$	
$wR(F^2) = 0.125$	$w = 1/[\sigma^2(F_o^2) + (0.0852P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	
2221 reflections	$(\Delta/\sigma)_{max} = 0.002$
162 parameters	$\Delta\rho_{max} = 1.02$ e Å^{-3}
	$\Delta\rho_{min} = -0.84$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Cu1—N1	1.920 (3)	Cu1—O3	1.961 (4)
Cu1—O1	1.922 (4)	Cu1—O5	1.946 (3)
Cu1—O1A	2.589 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5A···O4^i	0.808 (18)	1.99 (3)	2.705 (4)	148 (4)
O5—H5B···O4^{ii}	0.842 (18)	1.89 (2)	2.708 (4)	163 (3)

Symmetry codes: (i) $x, -y + \frac{5}{2}, z + \frac{1}{2}$; (ii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$.

H atoms bonded to C atoms were positioned geometrically, with C—H = 0.93–0.97 Å, and treated as riding atoms, with U_{iso}(H) = $1.2U_{eq}$(C). H atoms bonded to O atoms were located in difference maps and refined isotropically. The maximum electron-density peak is located 0.60 Å from atom C6.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

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