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### A (3,5-Dimethylpyridine-*N*)(*N*-salicylidene-glycinato-*O,N,O'*:*O'*)copper(II) Polymer

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#### Abstract

The title compound, [Cu(C<sub>9</sub>H<sub>7</sub>NO<sub>3</sub>)(C<sub>7</sub>H<sub>9</sub>N)], adopts a square-pyramidal Cu<sup>II</sup> coordination with the tridentate *N*-salicylidene-glycinato Schiff base dianion and the 3,5-dimethylpyridine ligand bound in the basal plane. The apex of the pyramid is occupied by a carboxylic O atom from the neighbouring chelate at an apical distance of 2.386 (2) Å leading to infinite one-dimensional chains along the crystallographic *a* axis.

#### Comment

Copper(II) complexes with tridentate Schiff base dianions of the *N*-salicylideneaminoalkanoato type (TSB<sup>2-</sup>) represent a relatively simple model for studies of cooperative bonding effects, which can be investigated by EPR (electron paramagnetic resonance) spectroscopy. In these complexes, which are of the general type [Cu(TSB)(*L*)]<sub>*n*</sub> (Warda, 1994), three donor atoms (O, N, O) of the Schiff base and a fourth donor atom from the neutral ligand *L* (N, O or S) normally define the base of a square pyramid. For isolated (monomeric) structures, the copper coordination can be square planar or square pyramidal when a neutral donor ligand is located in the apical site (*n* = 1; Ueki, Ashida, Sasada & Kakudo, 1969; Warda, Friebel, Sívý, Plesch & Švajlenová, 1996); *n* = 4 is found in the case of (4-ethylpyridine)(*N*-

salicylidene-glycinato)copper(II) (Warda, 1997). Polymeric structures (*n* = ∞) are achieved when the apical position is occupied by a carboxylic O atom from an adjacent molecule to form infinite zigzag chains (Warda, Friebel, Sívý, Plesch & Bláhová, 1997).

The title molecule, (I), is characterized by a square-pyramidal Cu<sup>II</sup> coordination with the tridentate Schiff base *N*-salicylidene-glycinato dianion and a monodentate ligand (3,5-dimethylpyridine) in the basal plane. The apical Cu—O3A bond length [2.386 (2) Å; symmetry code:  $x, \frac{1}{2} - y, \frac{1}{2} + z$ ], originating from a carboxylic O atom of a neighbouring chelate, is comparable with the parent compound of this structure type, namely aqua(*N*-salicylidene-glycinato)copper(II) hemihydrate [2.334 (6) Å]. In a new refinement of this crystal structure (Warda, 1994), the apical distance is 2.331 (2) Å. In the title compound, an infinite one-dimensional chain is formed by the Cu···O3···Cu bonds along the crystallographic *a* axis (Fig. 2). EPR patterns display a coupled *g* tensor indicating a distorted antiferrodistortive ordering with  $2\gamma = 85^\circ$  (EPR result), where the distance of the differently oriented paramagnetic centres is shorter than the critical distance of 8.3 Å (Warda, 1994). From X-ray data, the intrachain Cu···Cu spacing is 6.0742 (7) Å ( $x, \frac{1}{2} - y, \frac{1}{2} + z$ ) and the tilting angle is 84.60 (11)°, in good agreement with the results of the EPR measurement.

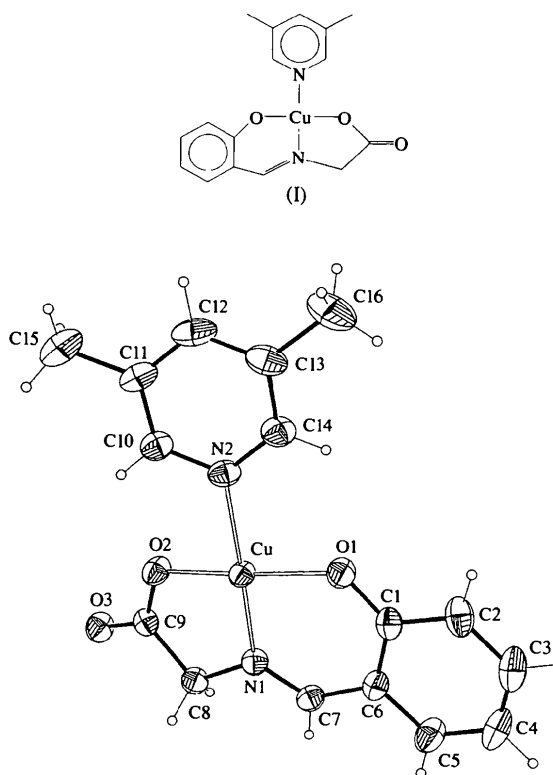


Fig. 1. The asymmetric unit of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

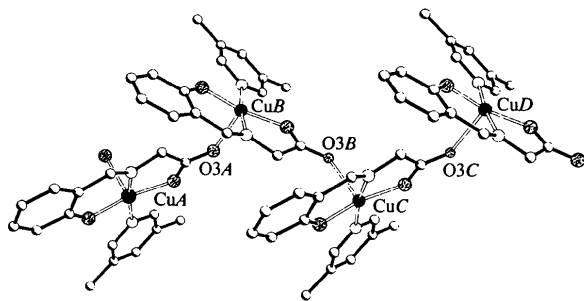


Fig. 2. The polymeric association of the title compound.

At this stage in the documentation of EPR data for a systematic study of the  $g$  tensors, the X-ray crystal structure determinations are indispensable for the verification of spectroscopic results. Furthermore, the structural results show the cooperative ordering. Monomeric and dimeric structures, for example, were found in monoclinic and different chain structures in orthorhombic crystal systems.

## Experimental

The compound was synthesized from aqua(*N*-salicylidene-glycinato)copper(II) hemihydrate (Ueki, Ashida, Sasada & Kakudo, 1967) and 3,5-dimethylpyridine.

### Crystal data

[Cu(C<sub>9</sub>H<sub>7</sub>NO<sub>3</sub>)(C<sub>7</sub>H<sub>9</sub>N)]  
 $M_r = 347.85$   
 Monoclinic  
 $P2_1/c$   
 $a = 14.1995(7) \text{ \AA}$   
 $b = 11.3316(5) \text{ \AA}$   
 $c = 9.7483(5) \text{ \AA}$   
 $\beta = 106.453(4)^\circ$   
 $V = 1504.30(13) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.536 \text{ Mg m}^{-3}$   
 $D_m$  not measured

### Data collection

Enraf–Nonius CAD-4  
 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction:  
 empirical with  $\psi$  scans  
 (Siemens, 1996a)  
 $T_{\min} = 0.405$ ,  $T_{\max} = 0.581$   
 2375 measured reflections  
 2224 independent reflections

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.055$   
 $wR(F^2) = 0.147$

Cu  $K\alpha$  radiation  
 $\lambda = 1.54178 \text{ \AA}$   
 Cell parameters from 25  
 reflections  
 $\theta = 17.29\text{--}19.66^\circ$   
 $\mu = 2.174 \text{ mm}^{-1}$   
 $T = 293(2) \text{ K}$   
 Prism  
 $0.44 \times 0.28 \times 0.25 \text{ mm}$   
 Dark green

2175 reflections with  
 $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.027$   
 $\theta_{\text{max}} = 59.93^\circ$   
 $h = -15 \rightarrow 15$   
 $k = -12 \rightarrow 0$   
 $l = 0 \rightarrow 10$   
 3 standard reflections  
 frequency: 120 min  
 intensity decay: none

$(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 1.079 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -1.273 \text{ e \AA}^{-3}$

$S = 1.143$   
 2224 reflections  
 202 parameters  
 H atoms riding, idealized  
 methyl groups  
 $w = 1/[\sigma^2(F_o^2) + (0.0964P)^2 + 1.8517P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

Extinction correction:  
*SHELXL96* (Sheldrick,  
 1996b)  
 Extinction coefficient:  
 0.0065 (8)  
 Scattering factors from  
*International Tables for  
 Crystallography* (Vol. C)

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Cu—O1	1.920 (2)	Cu—N2	2.017 (3)
Cu—N1	1.940 (3)	Cu—O3'	2.386 (2)
Cu—O2	1.977 (2)		
O1—Cu—N1	92.25 (10)	O2—Cu—N2	91.39 (11)
O1—Cu—O2	165.75 (11)	O1—Cu—O3'	98.62 (10)
N1—Cu—O2	83.09 (10)	N1—Cu—O3'	97.27 (10)
O1—Cu—N2	91.88 (11)	O2—Cu—O3'	95.33 (10)
N1—Cu—N2	172.38 (11)	N2—Cu—O3'	88.44 (10)

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1992).  
 Cell refinement: *CAD-4 EXPRESS*. Data reduction: *XCAD-4*  
 (Harms, 1997). Program(s) used to solve structure: *SHELXS86*  
 (Sheldrick, 1990). Program(s) used to refine structure:  
*SHELXL96* (Sheldrick, 1996a). Molecular graphics: *XP*  
 (Siemens, 1996b). Software used to prepare material for pub-  
 lication: *SHELXL96* (Sheldrick, 1996b).

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Supplementary data for this paper are available from the IUCr  
 electronic archives (Reference: JZ1196). Services for accessing these  
 data are described at the back of the journal.

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