

OE22—P2—OL23	107.75 (8)	P2—OL12—P1	124.97 (8)
OE21—P2—OL23	109.85 (8)	P3—OL13—P1	126.01 (8)
OL12—P2—OL23	100.46 (7)	P3—OL23—P2	125.91 (8)
OE31—P3—OE32	118.65 (8)	P1—P2—P3	60.12 (2)
OE31—P3—OL13	111.59 (7)	P1—P3—P2	59.88 (2)
OE32—P3—OL13	107.25 (7)	P2—P1—P3	60.00 (2)
OE31—P3—OL23	110.23 (8)	H1W1—OW1—H2W1	108 (3)
OE32—P3—OL23	106.23 (8)	H1W2—OW2—H2W2	102 (4)
OL13—P3—OL23	101.38 (7)	N1—C1—C2 ⁱⁱⁱ	112.7 (2)
OE31—Na—OE12 ⁱⁱ	115.11 (6)	C1 ^{vi} —C2—C3 ^{iv}	111.9 (2)
OE31—Na—OE31 ⁱ	81.04 (5)	C4—C3—C2 ⁱⁱ	112.9 (2)
OE12 ⁱⁱ —Na—OE31 ⁱ	92.49 (6)	C3—C4—C5	112.7 (2)
OE31—Na—OE21	86.33 (5)	C6 ^v —C5—C4	111.2 (2)
OE12 ⁱⁱ —Na—OE21	96.42 (6)	N2—C6—C5 ^v	112.9 (2)
OE31 ⁱ —Na—OE21	166.78 (6)		

Symmetry codes: (i) 2 - x, 1 - y, -1 - z; (ii) x, y, z - 1; (iii) x - 1, y, z; (iv) x, y, 1 + z; (v) 1 - x, -y, -1 - z; (vi) 1 + x, y, z.

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

D—H...A	D—H	H...A	D...A	D—H...A
OW1—H1W1...OE22 ⁱ	0.74 (3)	1.99 (3)	2.729 (2)	176 (3)
OW1—H2W1...OE22 ⁱⁱ	0.74 (3)	2.08 (3)	2.816 (2)	170 (3)
OW2—H1W2...OE12 ⁱⁱⁱ	0.80 (3)	2.26 (3)	3.010 (2)	156 (3)
OW2—H2W2...OE32 ^{iv}	0.78 (5)	2.22 (5)	2.988 (2)	172 (4)
N1—H1N1...OE11 ⁱⁱⁱ	0.83 (3)	2.16 (3)	2.825 (2)	138 (3)
N1—H2N1...OE32 ^v	0.89 (3)	1.98 (3)	2.861 (2)	172 (2)
N1—H3N1...OE11	0.96 (3)	2.05 (3)	3.006 (2)	177 (3)
N2—H1N2...OE21	0.87 (3)	1.91 (3)	2.775 (2)	169 (2)
N2—H2N2...OE32 ^v	0.89 (3)	1.99 (3)	2.852 (2)	164 (2)
N2—H3N2...OW1	0.88 (3)	1.89 (3)	2.740 (3)	163 (2)

Symmetry codes: (i) 1 - x, -y, -1 - z; (ii) x, y, z - 1; (iii) 1 - x, 1 - y, -z; (iv) 2 - x, 1 - y, -1 - z; (v) x - 1, y, z.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DU1172). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). **C53**, 1184–1186

catena-Poly[[5-ethyl-2-methylpyridine-N)copper(II)]-μ-(N-salicylidene-glycinato-O,N,O':O'')]

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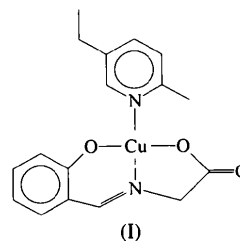
(Received 27 February 1997; accepted 8 April 1997)

Abstract

The title complex, [Cu(C₉H₇NO₃)(C₈H₁₁N)]_n, adopts a square-pyramidal Cu^{II} coordination with the tridentate N-salicylidene-glycinato Schiff base dianion (TSB²⁻) and the 5-ethyl-2-methylpyridine ligand bound in the basal plane. The apex of the pyramid is occupied by the carboxylic O atom from the neighbouring chelate at an apical distance of 2.268 (2) Å, which is the shortest apical distance in this class of substances.

Comment

Copper(II) complexes with tridentate Schiff base dianions of the N-salicylideneaminoalkanoato type (TSB²⁻) represent a relatively simple model for studies of cooperative bonding effects, which can be investigated by electron paramagnetic resonance (EPR) spectroscopy. In these complexes, which are of the general type [Cu(TSB)(L)]_n (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 Cu 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, Sivy, Plesch & Švajlenová, 1996). If a phenolic O atom from a neighbouring molecule is apically coordinated, dimeric structures are formed (n = 2; Warda, 1994; Warda, Dahlke, Wocadlo, Massa & Friebel, 1997). n = 4 is found in the case of (4-ethylpyridine)(N-salicylidene-glycinato)copper(II) (Warda, 1997). Polymeric struc-



tures ($n = \infty$) are achieved when the apical position is occupied by a carboxylic O atom from an adjacent molecule to make infinite zigzag chains (Warda, Friebel, Sivý, Plesch & Bláhová, 1997).

The molecule of the title complex, (I), is characterized by a square-pyramidal Cu^{II} coordination with the tridentate Schiff base *N*-salicylidene-glycinato dianion and a monodentate ligand (5-ethyl-2-methylpyridine) in the basal plane. The apical $\text{Cu}-\text{O}3(\frac{3}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z)$ bond length, 2.268 (2) Å, originating from

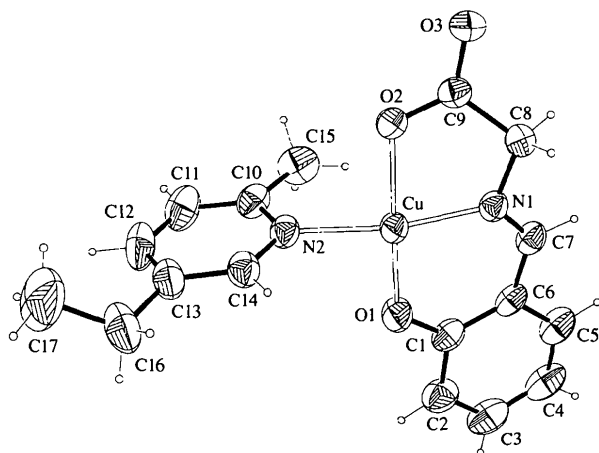


Fig. 1. A view of the asymmetric unit of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

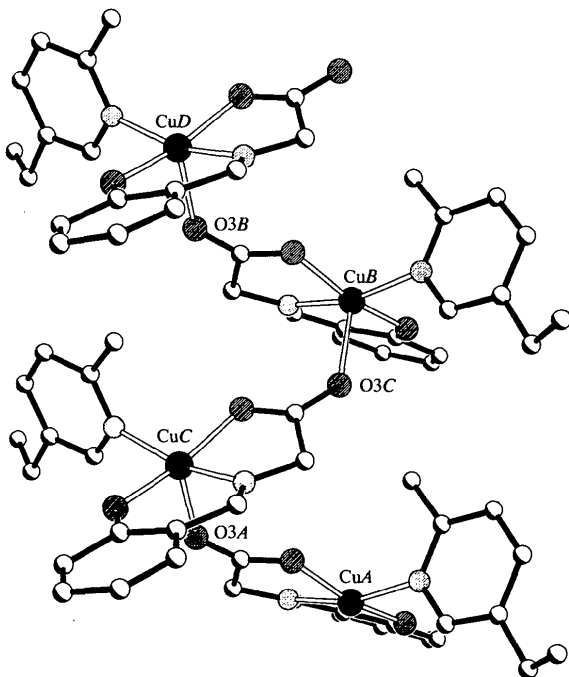


Fig. 2. Polymeric association of the title compound.

a carboxylic O atom of a neighbouring molecule, is the shortest so far for all products of *N*-salicylidene-aminoalkanoatocopper(II) with neutral donor ligands. An infinite one-dimensional chain is formed by the $\text{Cu}\cdots\text{O}3\cdots\text{Cu}$ bonds along the crystallographic *b* axis. EPR patterns display a coupled *g* tensor, indicative of distorted ferrodistoritive ordering, where the distance of the differently oriented paramagnetic centres is shorter than the critical distance of 8.3 Å (Warda, 1994); in (I), the $\text{Cu}\cdots\text{Cu}(\frac{3}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z)$ distance is 5.6557 (6) Å (PLATON; Spek, 1990). It was not possible to explain the coupling of the *g* parameters from the EPR data alone; thus, the structure determination was very important for the solution of this problem. The Cu polyhedra are also ordered in a quasi-ferrodistoritive orientation, because the molecular axes ($\text{O}-\text{Cu}-\text{O}$ *x* axis and $\text{N}-\text{Cu}-\text{N}$ *y* axis) are not exactly parallel to each other (Fig. 2). The short apical $\text{Cu}-\text{O}3(\frac{3}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z)$ distance explains the high value of the *g* tensor.

Experimental

The title compound was synthesized from aqua-*N*-salicylidene-glycinatocopper(II) hemihydrate (Ueki, Ashida, Sasada & Kakudo, 1967) and 5-ethyl-2-methylpyridine.

Crystal data

$[\text{Cu}(\text{C}_9\text{H}_7\text{NO}_3)(\text{C}_8\text{H}_{11}\text{N})]$

$M_r = 361.87$

Monoclinic

$P2_1/n$

$a = 13.5225(8)$ Å

$b = 6.9078(3)$ Å

$c = 17.6194(10)$ Å

$\beta = 102.327(5)^\circ$

$V = 1607.90(15)$ Å³

$Z = 4$

$D_x = 1.495$ Mg m⁻³

D_m not measured

Cu $K\alpha$ radiation

$\lambda = 1.54178$ Å

Cell parameters from 25

reflections

$\theta = 40.3-46.7^\circ$

$\mu = 2.057$ mm⁻¹

$T = 293(2)$ K

Prism

$0.20 \times 0.10 \times 0.10$ mm

Dark green

Data collection

Enraf-Nonius CAD-4
diffractometer

$\omega-2\theta$ scans

Absorption correction:
empirical with ψ scans
(Siemens, 1996a)

$T_{\min} = 0.668$, $T_{\max} = 0.830$

2509 measured reflections

2347 independent reflections

2233 reflections with

$I > 2\sigma(I)$

$R_{\text{int}} = 0.089$

$\theta_{\max} = 59.98^\circ$

$h = -15 \rightarrow 5$

$k = 0 \rightarrow 7$

$l = -19 \rightarrow 19$

3 standard reflections

frequency: 120 min

intensity decay: 3%

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.105$

$S = 1.070$

2347 reflections

209 parameters

H atoms riding

$\Delta\rho_{\max} = 0.379$ e Å⁻³

$\Delta\rho_{\min} = -0.499$ e Å⁻³

Extinction correction:

SHELXL96 (Sheldrick,
1996)

Extinction coefficient:

0.0053 (6)

$$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 1.8469P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$

Scattering factors from
*International Tables for
 Crystallography* (Vol. C)

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Monomeric (Pyrazine-*N*)(*N*-salicylidene- α -amino-2-methylpropanoate-*O,N,O'*)copper(II)

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Table 1. Selected geometric parameters (Å, °)

Cu—O1	1.9328 (19)	Cu—N2	2.031 (2)
Cu—N1	1.936 (2)	Cu—O3 ⁱ	2.268 (2)
Cu—O2	2.0021 (19)		
O1—Cu—N1	93.48 (9)	O2—Cu—N2	91.56 (8)
O1—Cu—O2	166.68 (9)	O1—Cu—O3 ⁱ	99.70 (9)
N1—Cu—O2	82.48 (9)	N1—Cu—O3 ⁱ	102.63 (9)
O1—Cu—N2	89.05 (9)	O2—Cu—O3 ⁱ	93.58 (8)
N1—Cu—N2	164.38 (10)	N2—Cu—O3 ⁱ	92.11 (9)

Symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$.

Data collection: CAD-4 EXPRESS (Enraf–Nonius, 1994). Cell refinement: CAD-4 EXPRESS. Data reduction: XCAD-4 (Harms, 1996). Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL96 (beta test version 03) (Sheldrick, 1996). Molecular graphics: XP in SHELXTL (Siemens, 1996b). Software used to prepare material for publication: SHELXL96.

We are grateful to the Institute of Inorganic Chemistry of the University of Marburg.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1101). Services for accessing these data are described at the back of the journal.

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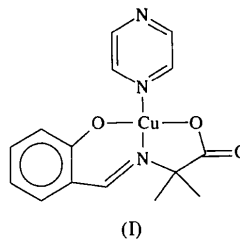
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Abstract

The title compound, [Cu(C₁₁H₁₁NO₃)(C₄H₄N₂)], adopts a square-planar Cu^{II} coordination with a tridentate *N*-salicylidene- α -amino-2-methylpropanoate [2-methyl-2-(salicylideneamino)propanoate] Schiff base dianion and a pyrazine ligand. The molecules are arranged in two magnetically inequivalent orientations.

Comment

Copper(II) complexes with tridentate Schiff base dianions of the *N*-salicylideneaminoalkanoate type (TSB²⁻) represent a relatively simple model for studies of cooperative bonding effects, which can be investigated by electron paramagnetic resonance (EPR) spectroscopy. In these complexes, which are of the general type [Cu(TSB)(L)]_n (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). Polymeric structures ($n = \infty$) are achieved when the apical position is occupied by a carboxylic oxygen from an adjacent molecule to form infinite zigzag chains (Ueki, Ashida, Sasada & Kakudo, 1967; Warda, Friebel, Sívý, Plesch & Bláhová, 1997). In this communication, we report a case with $n = 0$, (I).



In the title compound, (I), the environment of the copper(II) ion is square planar (Fig. 1), with O,N,O-donor atoms of the *N*-salicylidene- α -aminoisobutyrate