

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

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, 1996*b*). Software
 used to prepare material for publication: *SHELXL96*.

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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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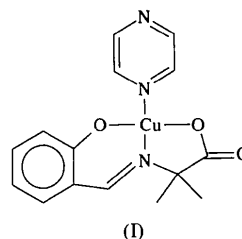
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Acta Cryst. C52, 2763–2766.

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 coop-
 erative 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 api-
 cal 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 re-
 port 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

dianion and an N atom of the pyrazine ligand. All non-H atoms except C10 lie in the mirror plane at $y = 0.25$. In the present case, the pyrazine acts as a monofunctional ligand only; it can act as a bifunctional ligand with other copper(II) tridentate systems, *e.g.* in the polymeric structure of (pyrazine)bis[*N*-salicylidene-(*R,S*)-alaninatocopper(II)] (Warda, 1994). The copper(II) layers are stacked parallel to [010] (Fig. 2), leading to a ferrodistorptive ordering, which in turn gives rise to a molecular (not coupled) *g* tensor. The EPR-powder and single-crystal patterns (Warda, 1994) display a weak coupling. We define the molecular axes O1—Cu—O2 as

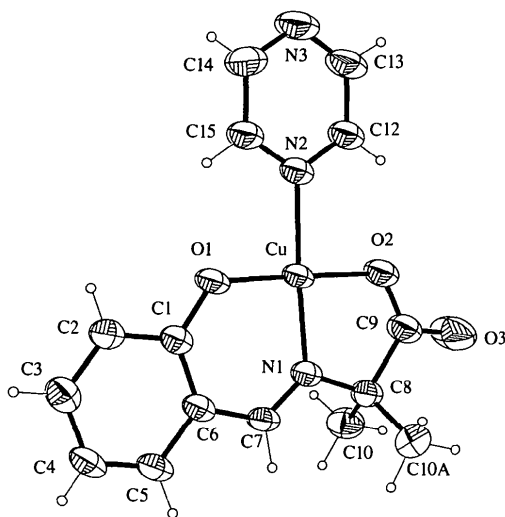


Fig. 1. A view of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

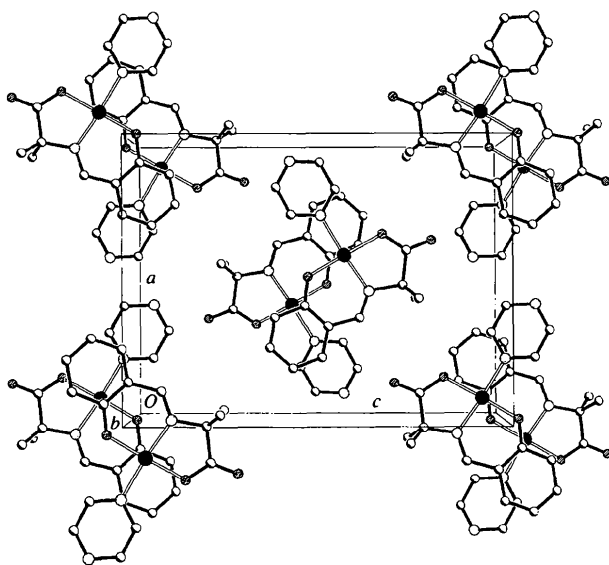


Fig. 2. Packing projected along the *b* axis.

x and N1—Cu—N2 as y , which are related to g as g_x and g_y , respectively, and should of course be distinguished clearly from the crystallographic axes with the same labels. The small electronic difference between these two axes results in a significant orthorhombic splitting of the *g* tensor; in the crystallographic *ac* plane there are two pairs of distinctly oriented x and y magnetic axes. The x axes define a type of tilting angle (2ξ) amounting to $60.4(7)^\circ$. If $2\xi = 90^\circ$, the orthorhombic splitting of the *g* tensor cannot be observed, and an axially symmetrical *g* tensor will be detected. We denote such a system with two non-collinear magnetic axes as 'quasi-ferrodistorptive'.

Experimental

The title compound was synthesized from aqua(*N*-salicylidene- α -aminoisobutyrate)copper(II) (Fujimaki, Oonishi, Muto, Nakahara & Komiyama, 1971) and pyrazine in dioxane–water (1:2).

Crystal data

[Cu(C₁₁H₁₁NO₃)(C₄H₄N₂)]
 $M_r = 348.84$
 Orthorhombic
Pnma
 $a = 12.7240(4) \text{ \AA}$
 $b = 6.7030(5) \text{ \AA}$
 $c = 16.8484(13) \text{ \AA}$
 $V = 1437.00(16) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.612 \text{ Mg m}^{-3}$
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 18.51\text{--}23.19^\circ$
 $\mu = 1.536 \text{ mm}^{-1}$
 $T = 293(2) \text{ K}$
 Prism
 $0.275 \times 0.250 \times 0.225 \text{ mm}$
 Orange

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω scans
 Absorption correction: empirical with ψ scans, (XPREP; Siemens, 1996a)
 $T_{\min} = 0.574$, $T_{\max} = 0.708$
 3160 measured reflections
 1592 independent reflections

1395 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$
 $\theta_{\max} = 26.33^\circ$
 $h = 0 \rightarrow 15$
 $k = -7 \rightarrow 8$
 $l = -21 \rightarrow 0$
 3 standard reflections
 frequency: 120 min
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.081$
 $S = 1.087$
 1592 reflections
 131 parameters
 H atoms riding
 $w = 1/[\sigma^2(F_o^2) + (0.0495P)^2 + 0.3196P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.272 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.328 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL96
 Extinction coefficient: 0.0016(5)
 Scattering factors from International Tables for Crystallography (Vol. C)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)
$$U_{eq} = (1/3) \sum_i \sum_j U^{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U _{eq}
Cu	0.41220 (2)	1/4	0.429551 (17)	0.03767 (14)
O1	0.48653 (13)	1/4	0.52604 (11)	0.0467 (5)
N1	0.53630 (15)	1/4	0.36423 (12)	0.0378 (5)
C1	0.58869 (17)	1/4	0.53476 (17)	0.0386 (6)
N2	0.27490 (16)	1/4	0.49067 (13)	0.0409 (5)
O2	0.33900 (14)	1/4	0.32988 (11)	0.0502 (5)
C2	0.6307 (2)	1/4	0.61258 (16)	0.0467 (6)
N3	0.08450 (19)	1/4	0.57191 (17)	0.0727 (11)
C3	0.7363 (2)	1/4	0.62682 (18)	0.0518 (7)
O3	0.35974 (18)	1/4	0.20051 (14)	0.1018 (12)
C4	0.8075 (2)	1/4	0.56476 (17)	0.0491 (7)
C5	0.77047 (19)	1/4	0.48833 (18)	0.0455 (6)
C6	0.66164 (18)	1/4	0.47180 (15)	0.0387 (5)
C7	0.63115 (19)	1/4	0.38929 (15)	0.0403 (6)
C8	0.5157 (2)	1/4	0.27742 (15)	0.0432 (6)
C9	0.3954 (2)	1/4	0.26703 (18)	0.0547 (8)
C10	0.56147 (17)	0.0633 (3)	0.23918 (12)	0.0556 (5)
C12	0.1833 (2)	1/4	0.45291 (18)	0.0515 (7)
C13	0.0894 (2)	1/4	0.4933 (2)	0.0665 (10)
C14	0.1756 (2)	1/4	0.60890 (19)	0.0687 (10)
C15	0.2706 (2)	1/4	0.56971 (16)	0.0524 (7)

Table 2. Selected geometric parameters (Å, °)

Cu—O1	1.8808 (18)	Cu—N1	1.925 (2)
Cu—O2	1.9204 (18)	Cu—N2	2.028 (2)
O1—Cu—O2	178.83 (7)	O1—Cu—N2	89.67 (8)
O1—Cu—N1	94.69 (8)	O2—Cu—N2	91.50 (8)
O2—Cu—N1	84.14 (8)	N1—Cu—N2	175.64 (9)

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* (Sheldrick, 1996). Molecular graphics: *XP* (Siemens, 1996b). Software used to prepare material for publication: *SHELXL96* (Sheldrick, 1996).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1198). Services for accessing these data are described at the back of the journal.

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Tetracarbonylbis[(2-methoxyphenyl)diphenylphosphine-P]tungsten Dichloromethane 0.25-Solvate

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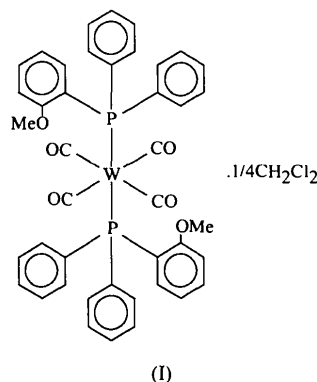
(Received 26 November 1996; accepted 22 April 1997)

Abstract

In the title complex, [W{P(C₆H₅)₂C₆H₄OCH₃-2}₂(CO)₄].1/4CH₂Cl₂, the W atom exhibits a slightly distorted octahedral coordination with the phosphine ligands in *trans* positions.

Comment

The title compound, (I), was prepared and its crystal structure examined as part of our continuing investigations on the coordination chemistry of functional phosphine ligands (Pietsch *et al.*, 1994; Pietsch, Dahlenburg, Wolski, Berke & Eremenko, 1995; Dahlenburg, Herbst & Kühnlein, 1997).



The structure consists of discrete molecules of the tungsten complex and dichloromethane solvate molecules lying on 4e positions with site symmetry