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Structure of Bis[2-(salicylideneamino)ethanolato]copper(II), [Cu(C₉H₁₀NO₂)₂]

BY FRANÇOISE NEPVEU AND HELMUT PAULUS

Institut für Physikalische Chemie, Technische Hochschule Darmstadt, Petersenstrasse 20, D-6100 Darmstadt, Federal Republic of Germany

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Abstract. $M_r = 391.9$, monoclinic, $P2_1/c$, $a = 18.323$ (4), $b = 4.813$ (1), $c = 19.770$ (4) Å, $\beta = 98.84$ (1)°, $V = 1722.8$ Å³, $Z = 4$, $D_m = 1.49$ (2), $D_x = 1.511$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 1.293$ mm⁻¹, $F(000) = 812$, $T = 293$ K, final $R = 0.029$ for 2072 observed reflections. The asymmetric unit consists of two independent half molecules (*A* and *B*) which are similar. Each of the Cu atoms has a square-planar environment with Cu bonded to two phenolic O atoms and two imino N atoms with distances Cu–O 1.877 (2), 1.882 (2) Å in molecules *A* and *B*, respectively, and Cu–N 2.010 (2) and 1.998 (2) Å. The ethanolic O atoms are not coordinated to Cu.

Introduction. The crystal structure of the title compound has been determined as a part of an investigation of the coordination properties of some Cu complexes in an attempt to correlate molecular structure and magnetic properties (Mergehenn & Haase, 1977; Walz, Paulus, Haase, Langhof & Nepveu, 1983). Schiff-base complexes prepared from salicylaldehyde and amino-alcohols have been characterized and different oligomeric species have been obtained (Brown &

Wardeska, 1982, and references therein; Muhonen, 1982; Galešić, Trojko, Cimerman & Štefanac, 1984).

Experimental. Slow evaporation of a methanolic solution containing copper(II) acetate, salicylaldehyde and 2-aminoethanol (molar ratio 1:2:2) in air gave dark-green parallelepipeds. Crystal $0.16 \times 0.27 \times 1.2$ mm. D_m by flotation in CH₂Cl₂/CCl₄. Stoe-Siemens AED2 diffractometer, Mo $K\alpha$ radiation. Cell constants from the 2θ values of 62 reflections. 4671 intensities scanned in range $6.6 \leq 2\theta \leq 45^\circ$, 2250 unique, $R_{\text{int}} = 0.022$, 178 unobserved [$F_o < 2\sigma(F_o)$]. Index range $h \pm 19$, $k 0/5$, $l 0/20$. Three standard reflections measured every hour, intensity variations < 2%. Numerical absorption correction, transmission factors 0.72–0.84. Structure determined by direct methods (MULTAN80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Anisotropic refinement on *F*. H atoms from difference Fourier synthesis, placed at calculated positions (C–H = 0.96 Å); U_H fixed at $1.1 \times U_{\text{eq}}$ of the carrying atom. $(\Delta/\sigma)_{\text{max}} = 0.09$. Final difference Fourier map with peaks within ± 0.25 e Å⁻³. Final refinement, 230 variables, gave $R = 0.029$, $wR = 0.033$ [$w = 1/\sigma^2(F)$]

Table 1. Atomic parameters

$$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}(\text{\AA}^2)$
Molecule A				
Cu(1)	0.0000	0.5000	0.5000	0.040 (2)
O(1)	-0.0728 (1)	0.5571 (3)	0.4235 (1)	0.059 (1)
O(2)	0.2023 (1)	0.2798 (3)	0.4204 (1)	0.061 (1)
N(1)	0.0486 (1)	0.2010 (3)	0.4516 (1)	0.044 (1)
C(1)	-0.0872 (1)	0.4189 (5)	0.3660 (1)	0.049 (1)
C(2)	-0.1509 (2)	0.4880 (5)	0.3199 (1)	0.064 (2)
C(3)	-0.1682 (2)	0.3537 (6)	0.2586 (1)	0.069 (2)
C(4)	-0.1237 (2)	0.1457 (6)	0.2401 (1)	0.071 (2)
C(5)	-0.0620 (2)	0.0724 (5)	0.2837 (1)	0.065 (2)
C(6)	-0.0421 (1)	0.2056 (4)	0.3472 (1)	0.049 (1)
C(7)	0.0229 (1)	0.1119 (5)	0.3912 (1)	0.050 (1)
C(8)	0.1158 (1)	0.0623 (4)	0.4860 (1)	0.048 (1)
C(9)	0.1837 (1)	0.2396 (4)	0.4868 (1)	0.052 (1)
Molecule B				
Cu(2)	0.5000	0.5000	0.0000	0.048 (1)
O(3)	0.4960 (1)	0.3472 (4)	0.0870 (1)	0.074 (1)
O(4)	0.2691 (1)	0.7221 (3)	-0.0938 (1)	0.055 (1)
N(2)	0.4112 (1)	0.7346 (4)	0.0059 (1)	0.050 (1)
C(10)	0.4439 (1)	0.3549 (5)	0.1250 (1)	0.059 (2)
C(11)	0.4498 (2)	0.1856 (6)	0.1833 (1)	0.073 (2)
C(12)	0.3967 (2)	0.1838 (7)	0.2245 (1)	0.082 (2)
C(13)	0.3351 (2)	0.3513 (7)	0.2105 (2)	0.084 (2)
C(14)	0.3279 (2)	0.5230 (6)	0.1548 (1)	0.073 (2)
C(15)	0.3817 (1)	0.5272 (5)	0.1108 (1)	0.055 (2)
C(16)	0.3717 (1)	0.7156 (5)	0.0541 (1)	0.056 (2)
C(17)	0.3866 (1)	0.9389 (5)	-0.0483 (1)	0.056 (2)
C(18)	0.3395 (1)	0.8036 (5)	-0.1084 (1)	0.057 (2)

for 2072 observed reflections used in the analysis. Secondary-extinction factor 1.29×10^{-4} . All calculations performed on a Data General Eclipse S 140 computer using a program package developed in the application laboratory of Stoe & Cie (Darmstadt). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. The $\text{Cu}(\text{C}_9\text{H}_{10}\text{NO}_2)_2$ complex crystallizes with two independent molecules (*A* and *B*) in the asymmetric unit. The Cu atoms are located on special positions [Cu(1): 0.0,0.5,0.5; Cu(2): 0.5,0.0,0.5]. Final positional parameters are given in Table 1.* Molecules *A* and *B* have very similar geometries. Selected bond lengths and angles are reported in Fig. 1. A projection of the unit cell is shown in Fig. 2.

The ethanolic O atoms [O(2) in *A*, O(4) in *B*] are not involved in the Cu coordination. The resulting bidentate ligand is coordinated to Cu *via* its phenolic O atom and its imino N atom. The molecules are centrosymmetric with the Cu atom at the centre. The coordination of Cu is therefore square planar consisting of two N and two O atoms from two salicyaldimine moieties.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a complete set of bond lengths and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42054 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Calculation of the least-squares plane through all atoms except C(9) and O(2) in molecule *A* shows that the largest atomic deviation is for O(1), 0.062 (1) Å; C(9) and O(2) lie 1.306 (6) and 2.305 (7) Å, respectively, above the plane. Molecule *B* is less planar. The dihedral angle between the least-squares plane through all atoms except Cu(2), O(3), O(4), N(2) and C(18), and the Cu(2),O(3),N(2) plane is 10.2 (1)°. C(18) and O(4) lie -1.430 (6) and -2.104 (7) Å from the former plane. Hydrogen bonding occurs between molecules *A* and *B* through the ethanolic O atoms with distances O(2)···O(4)($x, \frac{1}{2}-y, \frac{1}{2}+z$) = 2.726 (2) and O(2)···O(4)($x, \frac{1}{2}-y, \frac{1}{2}+z$) = 2.742 (2) Å, linking the molecules into sheets in the *ab* plane.

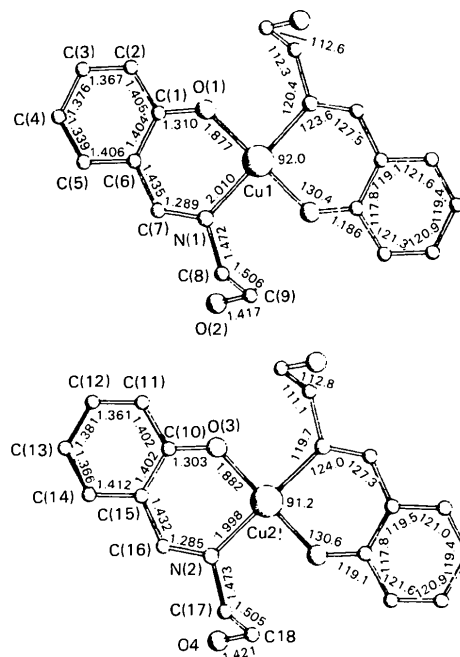


Fig. 1. Perspective view of the $\text{Cu}(\text{C}_9\text{H}_{10}\text{NO}_2)_2$ complex showing bond distances (Å) and angles (°) in molecules *A* (top) and *B* (bottom). The Cu atoms lie on inversion centres. Bond distances and angles involving Cu have e.s.d.'s of 0.002 Å and 0.10°; other distances and angles have e.s.d.'s of 0.003–0.005 Å and 0.2–0.4°.

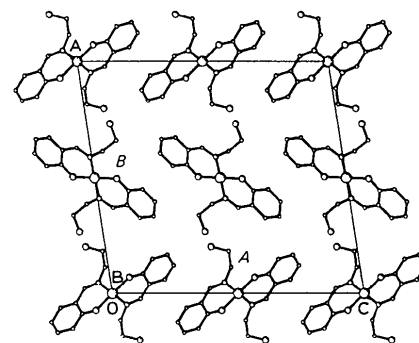


Fig. 2. View of the unit cell along *b*.

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Structure of Trichloro(7-8- η -morphinium)platinum(II) Hemihydrate Acetone Solvate, $[\text{PtCl}_3(\text{C}_{17}\text{H}_{20}\text{NO}_3)] \cdot \text{C}_3\text{H}_6\text{O} \cdot 0.5\text{H}_2\text{O}$

BY JEAN-PIERRE MACQUET

Laboratoire de Pharmacologie et de Toxicologie Fondamentales du CNRS, 205 route de Narbonne, 31400-Toulouse, France

AND ANDRÉ L. BEAUCHAMP

Département de Chimie, Université de Montréal, CP 6210, Succ. A, Montréal, Québec, Canada H3C 3V1

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Abstract. $M_r = 654.9$, tetragonal, $P4_2,2,2$, $a = 9.468$ (4), $c = 52.30$ (1) Å, $V = 4688.3$ Å³, $Z = 8$, $D_m = 1.85$ (1), $D_x = 1.855$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å (graphite monochromator), $\mu(\text{Mo } K\alpha) = 6.42$ mm⁻¹, $F(000) = 2552$, $T = 293$ K, $R = 0.039$, 1537 nonzero reflections [$I > 3\sigma(I)$]. Pt forms a square-planar complex with three Cl atoms [Pt–Cl 2.320 (4), 2.291 (5), 2.313 (5) Å] and the middle of the C(7)–C(8) double bond [Pt–C(7) 2.168 (13), Pt–C(8) 2.132 (14) Å]. The PtCl₃ units stand on the top of the horizontal arms of the T-shaped morphine moiety.

Introduction. In a previous paper (Macquet & Beauchamp, 1984), we reported the preparation of the title compound and briefly commented on its crystal structure. A detailed account of the crystallographic work is given in the present paper.

Experimental. Crystals obtained as described earlier (Macquet & Beauchamp, 1984). Density measured by flotation in CCl₄–CHBr₃. Yellow prism, 0.15 mm (102–102) × 0.18 mm (001–001) × 0.42 mm (012–012). Cell parameters derived from least-squares fit on angles for 24 reflections ($12 < \theta < 16^\circ$) centered on Enraf–Nonius CAD-4 diffractometer. Laue symmetry (4/*mmm*) and systematic absences (00*l*, $l \neq 4n$; *h*00, $h \neq 2n$; 0*k*0, $k \neq 2n$) derived from precession and

cone-axis photographs were consistent with space groups $P4_2,2,2$ and $P4_3,2,2$.

Data collected as described elsewhere (Bélanger-Gariépy & Beauchamp, 1980). Reflection sphere: $2\theta \leq 50^\circ$, $0 \leq h \leq 11$, $k \leq h$, $0 \leq l \leq 61$. Fluctuations of 3 standards $< \pm 2\%$. 2531 unique reflections measured, 994 unobserved [$I < 3\sigma(I)$], 1537 observed. Data corrected for the Lorentz effect, polarization and absorption (Gaussian integration, grid $10 \times 10 \times 10$, transmission range 0.34–0.44).

Structure solved by the heavy-atom method and refined on $|F|$ by full-matrix least squares. H atoms whose positions were predictable from C skeleton fixed at calculated positions ($B = 5.0$ Å²). Remaining H atoms neglected. Refinement of scale factor, coordinates and anisotropic temperature factors of non-hydrogen atoms. H atoms not refined, but repositioned after each cycle. $R = 0.039$, $wR = 0.044$, $S = 1.46$ for $m = 1537$ and $n = 271$. w based on counting statistics (Authier-Martin & Beauchamp, 1977). Δ/σ in last cycle, max. 0.12, av. 0.04. Background in final difference Fourier map 0.55 e Å⁻³, 6 peaks within ± 10.6 – 1.1 e Å⁻³ near Pt or Cl.

No attempt was made to refine the enantiomorphic structure (in $P4_2,2,2$), since torsion angles corresponded to the known absolute configuration of morphine (Bye, 1976; Gylbert, 1973).