achieve a total of eighteen, and from that for the complex [Fe(CO)$_3$(C$_5$H$_4$CPh$_2$)], where the metal has the same configuration (d^8) as Rh$^+$ but requires a smaller number of electrons (four) to reach eighteen. In terms of the number of fulvene C atoms involved in the bonding to the metal, the complexes (I) and (II) are clearly intermediate between these two cases.

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Structure of (Di-2-pyridylamine)salicylaldehydatocopper(II) Perchlorate, [Cu(C$_7$H$_5$O$_2$)(C$_{10}$H$_9$N$_3$)]ClO$_4$

BY M. T. GARLAND AND J. Y. LE MAROUILLE

Laboratoire de Chimie du Solide et Inorganique Moléculaire, LA au CNRS n° 254, Campus de Beaulieu, 35042 Rennes CEDEX, France

AND E. SPODINE

Facultad de Ciencias Básicas y Farmacéuticas, Universidad de Chile, Casilla 233, Santiago, Chile

Abstract. $M_r = 455.31$, triclinic, $P\bar{1}$, $a = 9.363$ (2), $b = 9.964$ (3), $c = 10.093$ (2) Å, $\alpha = 75.39$ (2), $\beta = 73.49$ (4), $\gamma = 83.34$ (3)°, $V = 872.6$ (4) Å^3, $Z = 2$, $D_m = 1.70$ (2), $D_x = 1.733$ Mg m^{-3}, λ(Mo $K\bar{\alpha}$) = 0.71073 Å, $\mu = 1.448$ mm^{-1}, $F(000) = 462$, $T = 291$ K. Final $R = 0.045$ for 2434 unique observed reflections. The structure consists of a dimeric unit involving two perchlorate anions with a positional disorder. The coordination sphere of copper can be described as an elongated octahedron due to the Jahn–Teller effect. The basal plane is formed by two nitrogen atoms of the two heterocycles of the dipyridylamine and two oxygen atoms of the salicylaldehyde group. Two large apical copper–oxygen distances are found: one toward the oxygen atom of the perchlorate anion and the other one, linking two monomeric units, toward the oxygen atom of the nearest salicylaldehyde molecule.

Introduction. The limited information on metal complexes of heterocyclic Schiff bases prompted us to undertake a systematic research in this field (Osipov, Minkin, Verkhovodova & Knyazhanskii, 1967; Mahmoud & El-Haty, 1980; Hatfield & Bunger, 1969; Atria, Blasquez & Spodine, 1982; Spodine & Atria, 1984; Galešić, Trojko, Cimerman & Štefanac, 1984). However, X-ray determinations of the crystal structures of several heterocyclic Schiff bases appear in the literature (Moustakali-Mavridis & Hadjoudis, 1978; Moustakali-Mavridis, Hadjoudis & Mavridis, 1980; Escobar & Garland, 1983, 1984).

Attempts to obtain single crystals for the mixed ligand (di-2-pyridylamine)(sb)Cu^{II} complexes, where sb stands for the deprotonated form of the ligand 2-amino-N-salicylidenepyridine or its methyl derivatives, yielded [Cu(dpa)(sal)]ClO$_4$. The salicylaldehyde molecule (sal) was formed from the original Schiff base by hydrolysis. These complexes are very unstable towards traces of water in the solvents used for the synthesis and recrystallizations (Atria *et al.*, 1982; Spodine & Atria, 1984).

Experimental. Cu(ClO$_4$)$_2$.6H$_2$O was first refluxed in acetonitrile (AN) in order to eliminate some of the molecules of hydration [Cu(ClO$_4$)$_2$.4AN.2H$_2$O was obtained]. A 1 mmol sample of cupric perchlorate was combined with 1 mmol of the Schiff base in freshly distilled ethanol and slightly refluxed. After this, 1 mmol of di-2-pyridylamine dissolved in the same solvent was added to the warm reaction mixture and heated during 1 h. When cooled, a brown microcrystalline solid appeared. After a few days dark-green crystals formed, which were purified by repeated crystallizations from methanol. A direct synthesis using salicylaldehyde as reagent is possible.

Crystal dimensions 0·12 × 0·14 × 0·06 mm, D_m measured in a mixture of thallium formate and malonate; Nonius CAD-4 four-circle diffractometer, cell dimensions from high 2θ angles of 25 reflections; 3244 integrated reflections collected up to sin θ/λ ≤ 0·639 Å^{-1}; ω–2θ scan technique, scan width (1·0 + 0·35 tanθ)°; −11 ≤ h ≤ 11, −12 ≤ k ≤ 12, 0 ≤ l ≤ 12; no significant decline in intensities of three standard reflections; decay 1·1% during 60 h of irradiation; no absorption correction and no time-decay corrections applied; 2800 unique reflections after averaging (R_{int} = 0·011); 2434 with $F^2 > 3\sigma(F^2)$; structure solution by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); refinement by full-matrix least squares based on F_o, weights based on counting statistics: $1/w = \sigma^2(F)$ = $\frac{1}{4}\{[\sigma^2(I) + (0.06I)^2]/I\}$ (Stout & Jensen, 1968); atomic scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974); hydrogen atoms placed at idealized positions with fixed isotropic thermal parameters and not refined (5 Å^2);

Table 1. *Fractional atomic coordinates and equivalent isotropic B (Å^2) with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\Sigma_i \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	B_{eq}
Cu	0·48458 (4)	0·07865 (4)	0·63177 (4)	2·630 (9)
Cl	0·2414 (1)	0·34912 (9)	0·8024 (1)	3·55 (2)
O(1)	0·2447 (5)	0·4655 (4)	0·6875 (5)	7·4 (1)
O(2)	0·2529 (5)	0·2173 (6)	0·7637 (5)	4·81 (9)*
O(2B)	0·186 (2)	0·241 (1)	0·774 (1)	7·1 (3)*
O(3)	0·1056 (7)	0·3661 (6)	0·9000 (7)	7·7 (2)*
O(3B)	0·199 (1)	0·339 (1)	0·962 (1)	5·4 (2)*
O(4)	0·3575 (7)	0·3539 (6)	0·8580 (6)	7·3 (1)*
O(4B)	0·405 (1)	0·339 (1)	0·796 (1)	5·4 (2)*
O(5)	0·3530 (2)	0·0635 (2)	0·5200 (3)	2·78 (5)
O(6)	0·5453 (3)	0·2568 (3)	0·4974 (3)	3·98 (6)
N(1)	0·6416 (3)	0·0864 (3)	0·7291 (3)	2·97 (7)
N(2)	0·5946 (4)	−0·1349 (3)	0·8882 (4)	3·67 (8)
N(3)	0·3928 (3)	−0·0836 (3)	0·7838 (3)	2·82 (7)
C(1)	0·3339 (4)	0·3042 (4)	0·4046 (4)	3·12 (8)
C(2)	0·2798 (4)	0·1698 (4)	0·4571 (4)	2·68 (8)
C(3)	0·1438 (4)	0·1468 (4)	0·4370 (4)	3·53 (9)
C(4)	0·0652 (4)	0·2545 (5)	0·3672 (5)	4·6 (1)
C(5)	0·1200 (5)	0·3873 (5)	0·3125 (5)	5·2 (1)
C(6)	0·2532 (5)	0·4113 (4)	0·3312 (5)	4·6 (1)
C(7)	0·7316 (4)	0·1974 (4)	0·6791 (5)	3·71 (9)
C(8)	0·8536 (4)	0·2047 (4)	0·7254 (5)	4·2 (1)
C(9)	0·8875 (4)	0·0967 (5)	0·8297 (5)	4·3 (1)
C(10)	0·7992 (4)	−0·0138 (5)	0·8814 (5)	4·3 (1)
C(11)	0·6770 (4)	−0·0178 (4)	0·8314 (4)	3·25 (9)
C(12)	0·4631 (4)	−0·1687 (4)	0·8748 (4)	2·93 (8)
C(13)	0·4057 (5)	−0·2964 (4)	0·9582 (5)	3·8 (1)
C(14)	0·2720 (5)	−0·3324 (4)	0·9537 (5)	4·2 (1)
C(15)	0·1933 (5)	−0·2410 (5)	0·8675 (5)	4·2 (1)
C(16)	0·2565 (4)	−0·1219 (4)	0·7859 (4)	3·60 (9)
C(17)	0·4691 (5)	0·3357 (4)	0·4229 (5)	3·73 (9)

* Population parameters of O(2), O(3), O(4), O(2B), O(3B) and O(4B) are 0·3, 0·3, 0·3, 0·7, 0·7 and 0·7, respectively, and their temperature factors were refined isotropically.

anisotropic thermal parameters for all other atoms except for the oxygen of the disordered anion; refinement converged to R = 0·045, wR = 0·064, goodness-of-fit S = 1·855 for 251 refined parameters; largest shift-over-error in last cycle 0·39; largest residual peak in final difference Fourier map 0·71 e Å^{-3}. All computer programs from Enraf–Nonius *SDP* described by Frenz (1978).

Discussion. Final atomic parameters are in Table 1,* bond distances and angles in Table 2. The atomic numbering is shown in Fig. 1.

The crystal can be described as consisting of dimeric units involving two perchlorate anions. Cu—O(5') bonds of 2·436 (2) Å link two molecules in order to form the dimeric unit.

The coordination sphere of copper can be described as an elongated octahedron, the basal plane being formed by two nitrogen and two oxygen atoms from the

Table 2. *Intramolecular bond lengths* (Å) *and bond angles* (°) *and selected intermolecular bond lengths* (Å) *with e.s.d.'s in parentheses*

Cu—O(2)	2·642 (3)		
Cu—O(5)	1·932 (2)	N(3)—C(12)	1·348 (4)
Cu—O(6)	1·974 (2)	N(3)—C(16)	1·367 (4)
Cu—N(1)	2·005 (3)	C(1)—C(2)	1·407 (4)
Cu—N(3)	2·007 (2)	C(1)—C(6)	1·411 (4)
Cl—O(1)	1·414 (3)	C(1)—C(17)	1·410 (4)
Cl—O(2)	1·446 (4)	C(2)—C(3)	1·397 (4)
Cl—O(2B)	1·376 (12)	C(3)—C(4)	1·385 (5)
Cl—O(3)	1·392 (5)	C(4)—C(5)	1·395 (6)
Cl—O(3B)	1·522 (10)	C(5)—C(6)	1·369 (5)
Cl—O(4)	1·368 (6)	C(7)—C(8)	1·367 (4)
Cl—O(4B)	1·505 (10)	C(8)—C(9)	1·381 (5)
O(5)—C(2)	1·317 (3)	C(9)—C(10)	1·358 (5)
O(6)—C(17)	1·253 (4)	C(10)—C(11)	1·383 (4)
N(1)—C(7)	1·372 (4)	C(12)—C(13)	1·406 (4)
N(1)—C(11)	1·352 (4)	C(13)—C(14)	1·358 (5)
N(2)—C(11)	1·380 (4)	C(14)—C(15)	1·392 (5)
N(2)—C(12)	1·365 (4)	C(15)—C(16)	1·353 (4)
O(5)—Cu—O(6)	87·97 (8)	C(6)—C(1)—C(17)	118·3 (3)
O(5)—Cu—N(1)	172·86 (9)	O(5)—C(2)—C(1)	123·4 (2)
O(5)—Cu—N(3)	90·63 (9)	O(5)—C(2)—C(3)	118·3 (3)
O(6)—Cu—N(1)	90·65 (9)	C(1)—C(2)—C(3)	118·3 (3)
O(6)—Cu—N(3)	170·24 (9)	C(2)—C(3)—C(4)	120·4 (3)
N(1)—Cu—N(3)	91·9 (1)	C(3)—C(4)—C(5)	121·5 (3)
O(1)—Cl—O(2)	113·8 (2)	C(4)—C(5)—C(6)	118·8 (3)
O(1)—Cl—O(2B)	108·2 (6)	C(1)—C(6)—C(5)	120·9 (3)
O(1)—Cl—O(3)	104·4 (3)	N(1)—C(7)—C(8)	123·3 (3)
O(1)—Cl—O(3B)	130·7 (4)	C(7)—C(8)—C(9)	118·9 (3)
O(1)—Cl—O(4)	108·6 (3)	C(8)—C(9)—C(10)	118·7 (3)
O(1)—Cl—O(4B)	97·6 (4)	C(9)—C(10)—C(11)	120·7 (3)
O(2)—Cl—O(3)	110·9 (3)	N(1)—C(11)—N(2)	121·3 (3)
O(2)—Cl—O(4)	108·4 (3)	N(1)—C(11)—C(10)	121·7 (3)
O(2B)—Cl—O(3B)	110·3 (7)	N(2)—C(11)—C(10)	117·0 (3)
O(2B)—Cl—O(4B)	117·1 (7)	N(2)—C(12)—N(3)	121·0 (3)
O(3)—Cl—O(4)	110·6 (3)	N(2)—C(12)—C(13)	117·2 (3)
O(3B)—Cl—O(4B)	91·2 (6)	N(3)—C(12)—C(13)	121·8 (3)
C(7)—N(1)—C(11)	116·6 (3)	C(12)—C(13)—C(14)	119·6 (3)
C(11)—N(2)—C(12)	132·9 (3)	C(13)—C(14)—C(15)	119·1 (3)
C(12)—N(3)—C(16)	116·5 (2)	C(14)—C(15)—C(16)	118·5 (3)
C(2)—C(1)—C(6)	120·2 (3)	N(3)—C(16)—C(15)	124·2 (3)
C(2)—C(1)—C(17)	121·5 (3)	O(6)—C(17)—C(1)	125·9 (3)
Cu—O(5ⁱ)	2·436 (2)	O(2B)—C(9ⁱⁱⁱ)	3·131 (13)
O(1)—C(17ⁱⁱ)	3·281 (4)	O(5)—O(5ⁱ)	2·856 (3)

Symmetry operations: (i) 1−x, −y, 1−z; (ii) 1−x, 1−y, 1−z; (iii) x−1, y, z.

A comparison of the structural features of the title compound containing the CuO_4N_2 moiety with those of [Cu(saldien)(H_2O)]PF_6 (Cusmano Priolo, Rotondo, Rizzardi, Bruno & Bombieri, 1983) and [Cu(ClO_4)-(saltrien)]ClO_4 (Rotondo, Cusmano Priolo, Bombieri & Bruno, 1984) shows a good similarity in bond distances. The Cu—O(perchlorate) distance [2·642 (3) Å] is in the range of the distances 2·575 (6)–2·676 (10) Å found in complexes containing the CuN_4O_2 coordination group.

The copper distance to the mean plane of the benzene ring is −0·700 Å which is not comparable with the previous values found by Galešić *et al.* (1984) in the [4-methoxymethyl-6-methyl-2-(salicylideneamino)-3-(salicylideneaminomethyl)pyridinato(2—)-*N,N',O,O'*]-copper 1·5 hydrate. The out-of-plane displacements of copper from the two pyridine rings of the dipyridyl-amine are 0·250 and 0·379 Å. The dipyridylamine chelate rings moderately deviate from planarity by 0·035 and 0·059 Å respectively.

We found positional disorder in three oxygen atoms of the perchlorate anion. This disorder was clearly detectable by difference electron density maps based on accurately refined coordinates for the rest of the molecule. It can be explained by a rotation of 33° around the Cl—O(1) bond, each site being occupied approximately ⅓ and ⅔ of the time.

The crystal packing is mainly stabilized by van der Waals forces between the oxygen atoms of the perchlorate and the carbon atoms of the aromatic rings (Table 2 and Fig. 1).

dipyridylamine moiety and from the salicylaldehyde moiety respectively. If we take into account the Jahn–Teller effect of copper, the octahedral 4+2 coordination of copper is completed by two large apical copper—oxygen distances in the octahedron [Cu—O(5') = 2·436 (2) and Cu—O(2) = 2·642 (3) Å, compared to basal copper—ligand bonds: Cu—O(6) = 1·974 (2), Cu—O(5) = 1·932 (2), Cu—N(1) = 2·005 (3) and Cu—N(3) = 2·007 (2) Å].

The unweighted mean plane through the four donor atoms N(1), N(3), O(5) and O(6) shows a lack of planarity with displacements of 0·136, −0·138, 0·147 and −0·145 Å, respectively. The copper atom is displaced from this plane by 0·024 Å in the direction of the apical O(5') atom. It should be noted that this apical atom O(5') is the most negative oxygen atom of the nearest molecule. The dihedral angle between the planes through Cu, O(5), O(6) and through Cu, N(1), N(3) is 11·8° indicating a distortion of the basal plane towards a tetrahedral geometry.
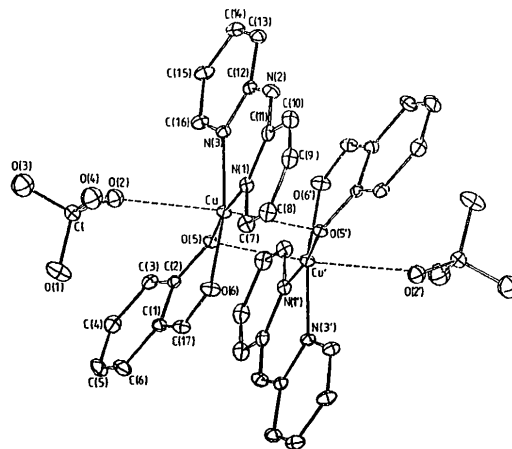

Fig. 1. A perspective view of the molecule with atom numbering and with hydrogen atoms excluded for clarity.

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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

Abstract. $M_r = 391 \cdot 9$, monoclinic, $P2_1/c$, $a = 18 \cdot 323\,(4)$, $b = 4 \cdot 813\,(1)$, $c = 19 \cdot 770\,(4)$ Å, $\beta = 98 \cdot 84\,(1)°$, $V = 1722 \cdot 8$ Å^3, $Z = 4$, $D_m = 1 \cdot 49\,(2)$, $D_x = 1 \cdot 511$ Mg m^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0 \cdot 71073$ Å, $\mu = 1 \cdot 293$ mm^{-1}, $F(000) = 812$, $T = 293$ K, final $R = 0 \cdot 029$ 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 $1 \cdot 877\,(2)$, $1 \cdot 882\,(2)$ Å in molecules A and B, respectively, and Cu–N $2 \cdot 010\,(2)$ and $1 \cdot 998\,(2)$ 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 $0 \cdot 16 \times 0 \cdot 27 \times 1 \cdot 2$ mm. D_m by flotation in CH$_2$Cl$_2$/CCl$_4$. Stoe–Siemens AED2 diffractometer, Mo $K\alpha$ radiation. Cell constants from the 2θ values of 62 reflections. 4671 intensities scanned in range $6 \cdot 6 \leq 2\theta \leq 45°$, 2250 unique, $R_{\text{int}} = 0 \cdot 022$, 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 $0 \cdot 72$–$0 \cdot 84$. 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 $= 0 \cdot 96$ Å); U_H fixed at $1 \cdot 1 \times U_{\text{eq}}$ of the carrying atom. $(\Delta/\sigma)_{\text{max}} = 0 \cdot 09$. Final difference Fourier map with peaks within $\pm 0 \cdot 25$ e Å^{-3}. Final refinement, 230 variables, gave $R = 0 \cdot 029$, $wR = 0 \cdot 033$ $[w = 1/\sigma^2(F)]$