
Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1218). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Hydrogen Bis[(8-quinolinol-*N*)(8-quinolinolato-*N*,*O*)(triphenylphosphine-*P*)silver(I)] Acetate Monohydrate

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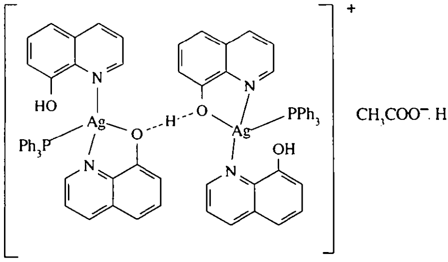
(*Received 11 December 1995; accepted 26 June 1996*)

Abstract

The asymmetric unit of the title compound, [$Ag_2H(C_9H_6NO)_2(C_9H_7NO)_2(C_{18}H_{15}P)_2](C_2H_3O_2).H_2O$, contains two exactly centrosymmetric [QHQ]$^+$ complex cations, Q = [$Ag(PPh_3)(C_9H_6NOH)(C_9H_6NO)$]. The two electroneutral silver complex units, Q, in each cation are connected by a short hydrogen bond [O···O 2.452 (5) and 2.463 (6) Å], with the proton lying at an inversion center. Each Ag atom has a distorted tetrahedral environment, bonding to two quinoline ligands and a PPh_3 ligand; both O and N atoms of the anionic quinolinol ligand are attached to the Ag atom, while in the neutral quinolinol ligand, the bonding is only through the N atom. The fourth coordination site is occupied by the P atom of a PPh_3 ligand. Some differences in the coordination of the two independent Ag atoms are observed. The [QHQ]$^+$ complex cations are linked by the trapped acetate ions through hydrogen bonding which also connects the water molecule to one of the acetate O atoms. In each [QHQ]$^+$ cation, there are intramolecular contacts between the Ag and the phenolic O atom of the neutral quinolinol moieties [Ag···O 2.827 (4) and 2.901 (4) Å].

Comment

Crystallographic studies on AgL(LH), L = 8-hydroxyquinolinate, show the existence of short O···H···O′ hydrogen bonds [O···O 2.457 Å] between adjacent molecules (Fleming & Lynton, 1968). In $Cu^{II}L_2$, a dimeric structure is found with the Cu atom forming a fifth bond to the O atom of a centrosymmetrically related molecule (Cu—O 2.830 Å; Palenik, 1964). The structural study of the title complex, (1), continues our exploration of the reactions of the dimeric binuclear complex, bis[acetato(triphenylphosphine)silver(I)] (Ng & Othman, 1995; Othman, Fun & Sivakumar, 1996).



(1)

An interesting feature of compound (1) is the structure of the [QHQ]$^+$ cation. A displacement ellipsoid plot of one of the two independent Q residues, which have a common numbering scheme, is shown in Fig. 1. In each cation, equivalent Q residues are connected by a short hydrogen bond [O1A···O1A^{i} 2.452 (5) and O1B···O1B^{ii} 2.463 (6) Å; symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x, -y+2, -z+1$]. In each case, the proton lies on a center of inversion. Inspection of the atomic coordinates of the two independent Q residues, A and B, revealed the existence of the pseudo-symmetry relationships $x_B \simeq \frac{1}{2} - x_A$ and $y_B \simeq y_A$, the z coordinates being independent. The structure of compound (1) bears significant resemblence to that of hydrogen bis(1-methyl-2-quinolone) hexafluoroarsenate(V), where there is a similar short contact involving a hydrogen bond between the two quinolone moieties [O···O 2.439 (12) Å;

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Calleri & Speakman, 1969], although the hydrogen bond does not lie across a symmetry element.

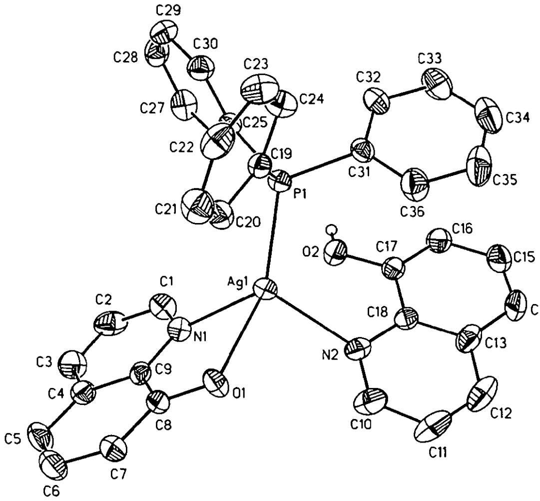

Fig. 1. A 30% displacement ellipsoid plot of an $[Ag(PPh_3)(C_9H_7NO)(C_9H_6NO)]$ unit in (1). A common atomic numbering scheme has been used for the two crystallographically independent units of this type.

In compound (1), the quinoline ligands are planar, the dihedral angles between the two quinoline ligands attached to the same Ag atom being similar [34.10 (8) in *A* and 33.97 (9)° in *B*]. The Ag—N bond distances [average 2.333 (4) Å] are longer than those in Ag*L*(*L*H) [average 2.150 (4) Å; Fleming & Lynton, 1968]. The relatively short Ag···O(phenolic) contacts [2.827 (4) in *A* and 2.901 (4) Å in *B*] involving the monodentate ligand suggest some interaction between the two atoms (Procter, Britton & Dunitz, 1981). In Ag*L*(*L*H), however, the difference in the Ag—O bond lengths is small compared with that in compound (1). The angles about the Ag atom in (1) deviate significantly from the ideal tetrahedral value, the smallest being N(1)—Ag—O(1) [68.9 (1) in *B* and 69.4 (1)° in *A*] and the largest N(1)—Ag—P [127.7 (1) in *A* and 135.4 (1)° in *B*]; the small N—Ag—O angle is a consequence of the constrained geometry of the quinolinate group.

The acetate ion bridges the cations through O—H···O hydrogen bonds; O2*A*···O4 2.565 (6) Å and O2*A*—H2*A*···O4 166 (7)°, and O2*B*···O3 2.568 (7) Å and O2*B*—H2*B*···O3 174 (7)°. There is a short intermolecular contact between the solvent water molecule and one of the acetate O atoms [O1···O4 2.727 (7) Å]. Hydrogen bonding involving the acetate ion and a water molcule has been observed previously (Perello, Verdaguer, Aymamie & Fita, 1994). The cations are thus connected into a chain parallel to the long *a* axis (see Fig. 2). In addition, the following possible C—H···O hydrogen bonds (< 3.5 Å) are observed: C10*A*···O1*A* 3.400 (7) Å, C10*A*—H10*A*···O1*A* 131.3 (6)°; C16*A*···O3 3.430 (8) Å, C16*A*—H16*A*···O3 152.6 (6)°; C26*A*···O4 3.456 (7) Å, C26*A*—H26*A*···O4 127.4 (5)°; C16*B*···O3 3.201 (8) Å, C16*B*—H16*B*···O3 124.3 (6)°; C7*A*···O1*A*^{i} 3.312 (7) Å, C7*A*—H7*A*···O1*A*^{i} 121.6 (6)°; C7*B*···O1*B*^{ii} 3.325 (7), C7*B*—H7*B*··· O1*B*^{ii} 121.4 (5)° (symmetry codes are as above).

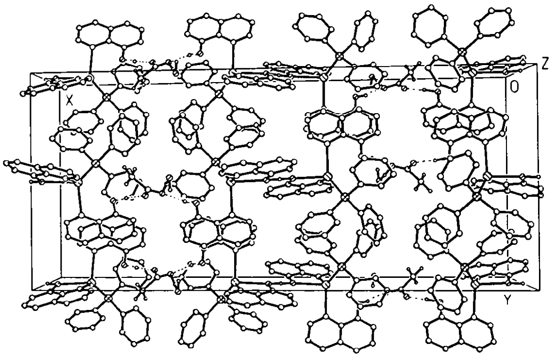

Fig. 2. Contents of the unit cell in (1) viewed down the *c* axis, showing the O—H···O hydrogen bonds. Symmetrical hydrogen bonds are shown as full lines, while other hydrogen bonds are shown as broken lines.

Experimental

The title compound was prepared by the reaction of excess 8-quinolinol with bis[acetato(triphenylphosphine)silver(I)] (molar ratio 4:1) in methanol. The mixture was heated with constant stirring. The hot solution was filtered and evaporated to give a yellow crystalline product. Single crystals were obtained by recrystallization from ethanol.

Crystal data

$[Ag_2H(C_9H_6NO)_2(C_9H_7NO)_2(C_{18}H_{15}P)_2](C_2H_3O_2).H_2O$
$M_r = 1396.96$
Monoclinic
$P2_1/c$
$a = 35.800\,(4)$ Å
$b = 15.564\,(1)$ Å
$c = 11.387\,(1)$ Å
$\beta = 96.21\,(1)°$
$V = 6307.5\,(10)$ Å^3
$Z = 4$
$D_x = 1.471$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 35 reflections
$\theta = 8$–$25°$
$\mu = 0.732$ mm^{-1}
$T = 293\,(2)$ K
Thick plate
0.54 × 0.48 × 0.32 mm
Yellow

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical *via* ψ scans (*XSCANS*; Siemens, 1994)
$T_{min} = 0.71$, $T_{max} = 0.79$
13 737 measured reflections
10 997 independent reflections
7783 observed reflections [$I > 2\sigma(I)$]

$R_{int} = 0.0549$
$\theta_{max} = 25°$
$h = -42 \rightarrow 42$
$k = -18 \rightarrow 1$
$l = -1 \rightarrow 13$
3 standard reflections monitored every 97 reflections
intensity decay: <4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0530$
$wR(F^2) = 0.2075$
$S = 1.056$
10 956 reflections
832 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0937P)^2 + 3.7086P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 1.62$ e Å^{-3}
$\Delta\rho_{\min} = -0.81$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Ag1*A*	0.40398 (1)	1.00990 (2)	0.42728 (3)	0.04396 (14)
P1*A*	0.37253 (4)	0.91853 (8)	0.55236 (11)	0.0387 (3)
O1*A*	0.47426 (11)	1.0032 (2)	0.4198 (3)	0.0505 (9)
O2*A*	0.33588 (12)	1.1025 (2)	0.3863 (4)	0.0518 (9)
N1*A*	0.41744 (12)	0.9828 (3)	0.2403 (4)	0.0424 (10)
N2*A*	0.40511 (13)	1.1602 (3)	0.4684 (4)	0.0505 (11)
C1*A*	0.3906 (2)	0.9722 (4)	0.1534 (5)	0.0527 (13)
C2*A*	0.3969 (2)	0.9462 (5)	0.0387 (6)	0.069 (2)
C3*A*	0.4324 (2)	0.9298 (4)	0.0173 (5)	0.067 (2)
C4*A*	0.4627 (2)	0.9378 (4)	0.1074 (5)	0.0516 (13)
C5*A*	0.4999 (2)	0.9167 (4)	0.0921 (6)	0.067 (2)
C6*A*	0.5274 (2)	0.9255 (4)	0.1829 (6)	0.068 (2)
C7*A*	0.5191 (2)	0.9555 (4)	0.2934 (5)	0.0518 (13)
C8*A*	0.48312 (15)	0.9762 (3)	0.3154 (4)	0.0402 (11)
C9*A*	0.45367 (15)	0.9660 (3)	0.2197 (4)	0.0404 (11)
C10*A*	0.4383 (2)	1.1926 (4)	0.5055 (7)	0.069 (2)
C11*A*	0.4438 (2)	1.2617 (4)	0.5817 (7)	0.083 (2)
C12*A*	0.4142 (2)	1.2983 (4)	0.6246 (7)	0.079 (2)
C13*A*	0.3775 (2)	1.2688 (3)	0.5851 (5)	0.060 (2)
C14*A*	0.3448 (2)	1.3056 (4)	0.6239 (6)	0.078 (2)
C15*A*	0.3106 (2)	1.2747 (4)	0.5809 (7)	0.078 (2)
C16*A*	0.3068 (2)	1.2062 (4)	0.4999 (6)	0.062 (2)
C17*A*	0.33778 (15)	1.1683 (3)	0.4628 (5)	0.0456 (12)
C18*A*	0.37417 (15)	1.1987 (3)	0.5057 (5)	0.0438 (12)
C19*A*	0.40369 (14)	0.8510 (3)	0.6512 (4)	0.0388 (11)
C20*A*	0.43979 (15)	0.8400 (3)	0.6259 (5)	0.0496 (13)
C21*A*	0.4647 (2)	0.7878 (4)	0.6958 (6)	0.064 (2)
C22*A*	0.4522 (2)	0.7457 (4)	0.7910 (6)	0.061 (2)
C23*A*	0.4162 (2)	0.7557 (4)	0.8168 (5)	0.0590 (15)
C24*A*	0.3917 (2)	0.8094 (4)	0.7489 (5)	0.0537 (14)
C25*A*	0.33976 (14)	0.8439 (3)	0.4693 (5)	0.0418 (11)
C26*A*	0.3222 (2)	0.8725 (4)	0.3622 (5)	0.0511 (13)
C27*A*	0.2987 (2)	0.8189 (4)	0.2929 (6)	0.063 (2)
C28*A*	0.2920 (2)	0.7370 (4)	0.3290 (6)	0.061 (2)
C29*A*	0.3095 (2)	0.7073 (4)	0.4344 (6)	0.061 (2)
C30*A*	0.3335 (2)	0.7601 (3)	0.5041 (5)	0.0498 (13)
C31*A*	0.3435 (2)	0.9756 (3)	0.6490 (5)	0.0432 (12)
C32*A*	0.3047 (2)	0.9676 (4)	0.6423 (6)	0.065 (2)
C33*A*	0.2838 (2)	1.0172 (5)	0.7107 (8)	0.080 (2)
C34*A*	0.3016 (2)	1.0766 (5)	0.7876 (6)	0.078 (2)
C35*A*	0.3396 (3)	1.0878 (5)	0.7936 (6)	0.085 (2)
C36*A*	0.3606 (2)	1.0378 (5)	0.7237 (6)	0.068 (2)
Ag1*B*	0.09663 (1)	1.00445 (2)	0.49008 (3)	0.04521 (14)
P1*B*	0.12475 (4)	0.91765 (8)	0.64714 (11)	0.0366 (3)
O1*B*	0.02572 (11)	1.0012 (2)	0.4364 (3)	0.0509 (10)
O2*B*	0.16779 (12)	1.0898 (2)	0.4716 (3)	0.0533 (9)
N1*B*	0.08179 (12)	0.9871 (2)	0.2928 (4)	0.0397 (9)
N2*B*	0.10135 (14)	1.1574 (3)	0.5199 (4)	0.0535 (12)
C1*B*	0.1085 (2)	0.9843 (4)	0.2208 (5)	0.0554 (15)
C2*B*	0.1018 (2)	0.9602 (4)	0.1017 (5)	0.060 (2)
C3*B*	0.0667 (2)	0.9360 (4)	0.0590 (5)	0.062 (2)
C4*B*	0.0370 (2)	0.9379 (3)	0.1318 (5)	0.0485 (13)
C5*B*	0.0001 (2)	0.9126 (4)	0.0924 (5)	0.060 (2)
C6*B*	−0.0269 (2)	0.9168 (4)	0.1675 (5)	0.064 (2)
C7*B*	−0.0192 (2)	0.9472 (4)	0.2832 (5)	0.0522 (13)
C8*B*	0.01687 (15)	0.9725 (3)	0.3272 (5)	0.0425 (12)
C9*B*	0.04594 (14)	0.9660 (3)	0.2497 (4)	0.0374 (11)
C10*B*	0.0696 (2)	1.1930 (4)	0.5403 (6)	0.072 (2)
C11*B*	0.0674 (3)	1.2600 (5)	0.6211 (8)	0.093 (3)
C12*B*	0.0994 (3)	1.2908 (4)	0.6828 (8)	0.088 (3)
C13*B*	0.1342 (2)	1.2563 (3)	0.6616 (6)	0.065 (2)
C14*B*	0.1689 (3)	1.2850 (4)	0.7210 (6)	0.083 (2)
C15*B*	0.2012 (3)	1.2502 (5)	0.6963 (6)	0.084 (2)
C16*B*	0.2022 (2)	1.1840 (4)	0.6117 (5)	0.065 (2)
C17*B*	0.1689 (2)	1.1536 (3)	0.5528 (5)	0.0474 (13)
C18*B*	0.1338 (2)	1.1895 (3)	0.5761 (5)	0.0483 (13)
C19*B*	0.09255 (14)	0.8514 (3)	0.7203 (4)	0.0387 (11)
C20*B*	0.05519 (15)	0.8463 (3)	0.6724 (5)	0.0496 (13)
C21*B*	0.0300 (2)	0.7956 (4)	0.7245 (6)	0.065 (2)
C22*B*	0.0418 (2)	0.7489 (4)	0.8244 (6)	0.064 (2)
C23*B*	0.0784 (2)	0.7531 (4)	0.8729 (5)	0.061 (2)
C24*B*	0.1037 (2)	0.8045 (3)	0.8218 (5)	0.0509 (13)
C25*B*	0.15960 (14)	0.8424 (3)	0.6002 (4)	0.0410 (11)
C26*B*	0.1896 (2)	0.8746 (4)	0.5487 (6)	0.064 (2)
C27*B*	0.2164 (2)	0.8215 (4)	0.5101 (6)	0.072 (2)
C28*B*	0.2134 (2)	0.7338 (4)	0.5222 (6)	0.064 (2)
C29*B*	0.1835 (2)	0.7011 (4)	0.5722 (6)	0.064 (2)
C30*B*	0.1565 (2)	0.7550 (3)	0.6120 (5)	0.0523 (13)
C31*B*	0.15072 (15)	0.9781 (3)	0.7658 (4)	0.0392 (11)
C32*B*	0.1857 (2)	0.9549 (4)	0.8209 (5)	0.0563 (14)
C33*B*	0.2042 (2)	1.0056 (5)	0.9080 (6)	0.077 (2)
C34*B*	0.1871 (3)	1.0797 (5)	0.9430 (6)	0.081 (2)
C35*B*	0.1520 (3)	1.1027 (4)	0.8916 (6)	0.075 (2)
C36*B*	0.1339 (2)	1.0532 (4)	0.8039 (5)	0.0533 (14)
O1*W*	0.2933 (2)	0.9893 (4)	0.0710 (5)	0.0783 (14)
O3	0.23457 (14)	1.0674 (3)	0.4144 (4)	0.0834 (14)
O4	0.27280 (12)	1.0567 (3)	0.2756 (4)	0.0677 (12)
C37	0.2146 (2)	0.9827 (6)	0.2499 (9)	0.072 (2)
C38	0.2430 (2)	1.0403 (4)	0.3182 (6)	0.0571 (15)

Table 2. *Selected geometric parameters (Å, °)*

Ag1*A*—N1*A*	2.272 (4)	Ag1*B*—N1*B*	2.268 (4)
Ag1*A*—P1*A*	2.3802 (13)	Ag1*B*—P1*B*	2.3765 (13)
Ag1*A*—N2*A*	2.386 (4)	Ag1*B*—N2*B*	2.407 (4)
Ag1*A*—O1*A*	2.529 (4)	Ag1*B*—O1*B*	2.546 (4)
O1*A*—C8*A*	1.332 (6)	O1*B*—C8*B*	1.327 (6)
O2*A*—C17*A*	1.343 (6)	O2*B*—C17*B*	1.356 (6)
N1*A*—C1*A*	1.313 (7)	N1*B*—C1*B*	1.325 (7)
N1*A*—C9*A*	1.368 (7)	N1*B*—C9*B*	1.363 (6)
N2*A*—C10*A*	1.318 (7)	N2*B*—C10*B*	1.309 (8)
N2*A*—C18*A*	1.367 (7)	N2*B*—C18*B*	1.360 (7)
N1*A*—Ag1*A*—P1*A*	127.71 (11)	N1*B*—Ag1*B*—P1*B*	135.43 (10)
N1*A*—Ag1*A*—N2*A*	111.4 (2)	N1*B*—Ag1*B*—N2*B*	105.23 (15)
P1*A*—Ag1*A*—N2*A*	117.87 (11)	P1*B*—Ag1*B*—N2*B*	116.00 (12)
N1*A*—Ag1*A*—O1*A*	69.41 (14)	N1*B*—Ag1*B*—O1*B*	68.86 (13)
P1*A*—Ag1*A*—O1*A*	122.17 (9)	P1*B*—Ag1*B*—O1*B*	119.96 (9)
N2*A*—Ag1*A*—O1*A*	92.92 (14)	N2*B*—Ag1*B*—O1*B*	96.12 (15)

The title structure was solved by direct methods and refined by full-matrix least-squares method on F^2. The H atoms attached to the phenyl rings and quinoline moieties were fixed geometrically and allowed to ride on their respective parent atoms; the H atoms attached to the OH groups (O2*A* and O2*B*) and those of the acetate and water molecules were located from the difference Fourier maps and refined isotropically.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93. Geometrical calculations: *PARST* (Nardelli, 1983).

The authors thank the Universiti Kebangsaan Malaysia for grant S/2/96, Professor Ibrahim Abdullah for his interest and the Malaysian Government and

Universiti Sains Malaysia for research grant R&D No. 123-3417-2201. KS thanks Universiti Sains Malaysia for a Visiting Post Doctoral Research Fellowship.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1240). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 2763–2766

Aqua(3,5-dimethylpyrazole)(*N*-salicylideneglycinato)copper(II)

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(Received 29 November 1995; accepted 26 June 1996)

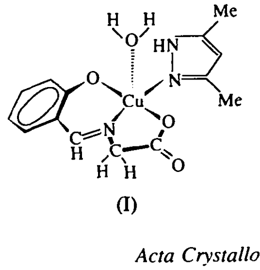
Abstract

Aqua(3,5-dimethylpyrazole-N^1)(N-salicylideneglycinato-O,N,O')copper(II), $[Cu(C_9H_7NO_3)(C_5H_8N_2)(H_2O)]$, adopts a square-pyramidal Cu^{II} coordination with the tridentate *N*-salicylideneglycinato Schiff base dianion and the 3,5-dimethylpyrazole ligand bound in the basal plane. The water molecule occupies the apical site. The complex molecules are arranged in four magnetically non-equivalent orientations, two at a time forming zigzag chains. Both the interchain Cu···Cu spacing of 7.396 Å and the intrachain Cu···Cu distance of 6.880 Å are too large for effective dipolar coupling between the paramagnetic Cu^{II} centres, in agreement with the results of electron paramagnetic resonance (EPR) spectroscopy.

Comment

Copper(II) complexes with a tridentate Schiff base dianion of the *N*-salicylideneamino alkanoate type (TSB^{2-}) represent a relatively simple model for intermediates in enzymatic reactions of amino acids (Krätsmár-Šmogrovič, Bergendi, Duncková, Švajlenová & Seressová, 1988). In recent years, however, much attention has been devoted to their structural and spectroscopic properties in the solid state, since they show an extraordinary richness of cooperative orderings. In these complexes, which are of the general type $[Cu(TSB)(L)(H_2O)_x]$, three donor atoms (O, N and 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. The apical site can be occupied by different types of donor atoms, giving rise to the observed variety of structural orderings; when a carboxylic O atom from an adjacent molecule takes the apex of the pyramid, infinite chains may be formed (Sivý, Kettmann, Krätsmár-Šmogrovič, Švajlenová, Friebel & Plesch, 1990). The apical bonding of phenolic oxygen from an adjacent molecule results in the formation of dimers (Pavelčík, Krätsmár-Šmogrovič, Švajlenová & Majer, 1991). Finally, the apical position can be occupied by a water molecule, giving rise to an isolated square-pyramidal complex (Ueki, Ashida, Sasada & Kakudo, 1969). If the coordination polyhedra are sufficiently close to one another, magnetic coupling between the Cu^{2+} ions, ranging from ferrodistortive to antiferrodistortive, distinctly influences the EPR spectra (Warda, 1994; Plesch, Friebel, Švajlenová & Krätsmár-Šmogrovič, 1995).

Since the EPR spectrum of the title compound, (I), gave no evidence for a coupled g tensor (Plesch, Friebel, Švajlenová & Krätsmár-Šmogrovič, 1987), it was of interest to analyze its crystal structure by X-ray diffraction.



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Acta Crystallographica Section C
ISSN 0108-2701 © 1996