metal-organic papers

Di-μ-chloro-bis({3-[(2-hydroxy-1-methyl-2-phenyl-ethyl)iminomethyl]-2-naphtholato-κ^3N,O,O'}-copper(II)) methanol solvate

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In the title Schiff base complex, $[Cu_2(C_{20}H_{18}NO_2)_2Cl_2]\cdot CH_3OH$, two Cu^{II} ions are bridged by two Cl^- anions. Each Cu^{II} ion has an NO_2Cl_2 coordination environment with a distorted square-pyramidal geometry.

Comment

Phenylpropanolamine (PPA) has been used as an ingredient in many cold and cough remedies for many years. Recently, there have been many reports on PPA because it has been associated with an increased risk of hemorrhagic stroke (Kerman et al., 2001). Modification of PPA has been an active area, but studies on the corresponding derivatives of phenylpropanolamine Schiff base are still very scanty. We report here the preparation and crystal structure of the title compound, (I).
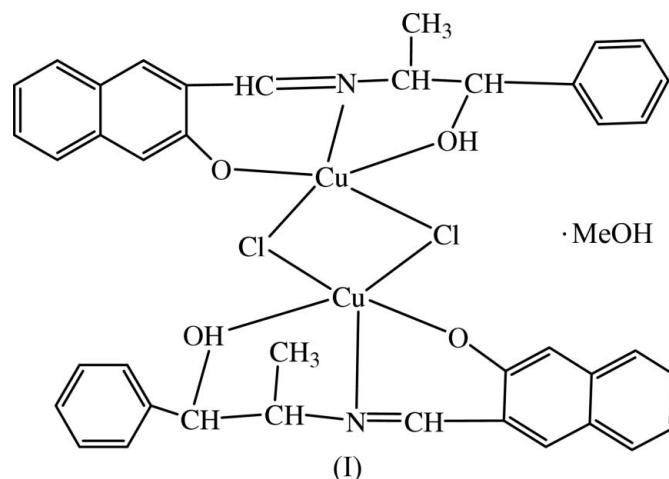
The molecular structure of (I) is shown in Fig. 1. Two Cu^{II} ions are bridged by two Cl^- anions, forming a dimer. Each Cu^{II} ion has a distorted square-pyramidal coordination geometry, with a longer apical Cu—Cl bond (Table 1). The PPA chelates to the Cu^{II} ion, forming a planar six-membered chelate ring and an envelope five-membered chelate ring.

Both intra- and inter-molecular hydrogen bonding occur in the crystal structure of (I) (Table 2).

Experimental

To a stirred solution of 2-hydroxy-1-naphthaldehyde (0.172 g, 1.0 mmol) and $Cu(ClO_4)_2\cdot6H_2O$ (0.372 g, 1.0 mmol) in absolute methanol (25 ml), a solution of phenylpropanolamine hydrochloride (0.187 g, 1.0 mmol) in absolute methanol (10 ml) was added dropwise. Sodium hydroxide (0.040 g, 1.0 mmol) was then added to the mixture. After stirring for 3 h at 320 K, the precipitate was filtered
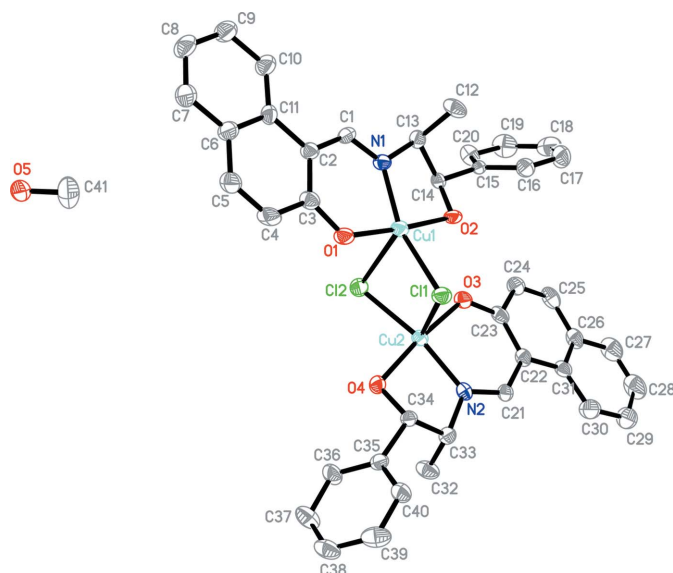
Figure 1
The molecular structure of (I), shown with 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

off. Single crystals of (I) were obtained by slow evaporation of the filtrate after 10 d.

Crystal data

$[Cu_2(C_{20}H_{18}NO_2)_2Cl_2]\cdot CH_4O$	$Z = 4$
$M_r = 838.73$	$D_x = 1.464$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 15.252\ (5)$ Å	$\mu = 1.31$ mm^{-1}
$b = 8.884\ (3)$ Å	$T = 298\ (2)$ K
$c = 28.866\ (10)$ Å	Block, blue
$\beta = 103.410\ (6)°$	$0.42 \times 0.16 \times 0.08$ mm
$V = 3805\ (2)$ Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	19209 measured reflections
φ and ω scans	6695 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)	3217 reflections with $I > 2\sigma(I)$
$T_{min} = 0.610$, $T_{max} = 0.904$	$R_{int} = 0.118$
	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0716P)^2 + 4.9577P]$
$R[F^2 > 2\sigma(F^2)] = 0.066$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.211$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.01$	$\Delta\rho_{max} = 0.79$ e Å^{-3}
6695 reflections	$\Delta\rho_{min} = -0.90$ e Å^{-3}
473 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Cu1—Cl1	2.245 (2)	Cu2—Cl1	2.689 (2)
Cu1—Cl2	2.827 (3)	Cu2—Cl2	2.255 (2)
Cu1—N1	1.912 (6)	Cu2—N2	1.920 (6)
Cu1—O1	1.871 (5)	Cu2—O3	1.903 (5)
Cu1—O2	1.981 (5)	Cu2—O4	1.992 (5)
O1—Cu1—N1	92.9 (2)	O3—Cu2—N2	92.5 (2)
O1—Cu1—O2	175.4 (2)	O3—Cu2—O4	174.5 (2)
N1—Cu1—O2	82.7 (2)	N2—Cu2—O4	81.9 (2)
O1—Cu1—Cl1	93.61 (17)	O3—Cu2—Cl1	91.82 (17)
N1—Cu1—Cl1	161.6 (2)	N2—Cu2—Cl2	166.5 (2)
O2—Cu1—Cl1	90.97 (15)	O4—Cu2—Cl2	93.58 (16)
O1—Cu1—Cl2	93.82 (19)	O3—Cu2—Cl1	94.77 (18)
N1—Cu1—Cl2	106.1 (2)	N2—Cu2—Cl1	98.34 (19)
O2—Cu1—Cl2	86.04 (17)	O4—Cu2—Cl1	85.98 (17)
Cl1—Cu1—Cl2	90.57 (8)	Cl2—Cu2—Cl1	93.98 (8)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2···O3	0.85	1.80	2.647 (8)	177
O4—H4···O5[i]	0.85	1.74	2.594 (8)	178
O5—H5···O1[i]	0.82	2.09	2.764 (8)	140

Symmetry code: (i) $-x + 1, -y + 1, -z + 1$.

Hydroxy atoms H2 and H4 were located in a difference Fourier map and refined with a distance constraint of O—H = 0.85 Å. Other H atoms were placed in calculated positions, with C—H = 0.93 (aromatic), 0.96 (methyl) and 0.98 Å (methylene) and O—H = 0.82 Å (methanol), and refined in riding mode, with $U_{iso}(H) = 1.5U_{eq}(C,O)$ for methyl and O-bound H atoms, and $U_{iso}(H) = 1.2U_{eq}(C)$ for the remaining H atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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