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[*N*,*N*-Bis(3,5-dimethylpyrazol-1-ylmethyl)-1-hydroxy-2-aminoethane](3,5-dimethylpyrazole)copper(II) Diperchlorate†

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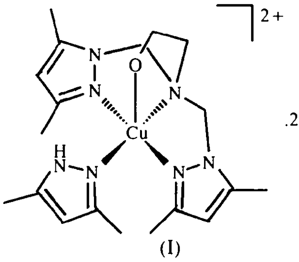
Abstract

The title compound, $[Cu(C_5H_8N_2)(C_{14}H_{23}N_5O)](ClO_4)_2$, consists of a five-coordinate square-pyramidal copper(II) complex as its perchlorate salt. The copper(II) ion is coordinated in the basal plane by azole and amine N atoms of the *N*,*N*-bis[(3,5-dimethyl-1-pyrazolyl)methyl]-1-hydroxy-2-aminoethane (bpmhe) ligand and 3,5-dimethylpyrazole, and in the apical position by an alcohol O atom.

Comment

Copper(II) complexes of the tripodal ligand bpmhe (Driessen, 1982) of the type [Cu(bpmhe)*L*]*X* (*L* = Br, Cl, NO_3, N_3; *X* = Cl, Br, BF_4) have been reported as mononuclear five-coordinate complexes (Malachowski, Davidson & Hoffman, 1989). The ligand bpmhe may act as a tetradentate ligand with two pyrazole nitrogen, one tertiary amine and one alcohol oxygen donor. We have been interested in the formation of such complexes with *L* as a neutral donor ligand and have isolated the title compound [Cu(bpmhe)*L*]$(ClO_4)_2$, where *L* is 3,5-dimethylpyrazole.



(I)

The coordination geometry around the copper(II) ion consists of two pyrazole N atoms, an alcohol O atom and an amine N atom of the bpmhe ligand and one N atom of the 3,5-dimethylpyrazole molecule in a square-pyramidal environment. The four N atoms form the base of the pyramid with the alcohol O atom at the apex. The Cu—N bond distances and angles are similar to those found in other *N*,*N*-bis(pyrazol-1-ylmethyl)amine complexes of copper(II) (Blonk, Driessen & Reedijk, 1985; Driessen, De Graaff & Wiesmeijer, 1987). The

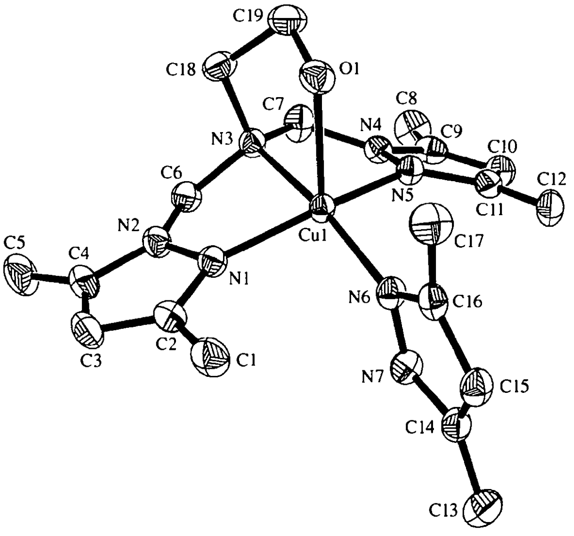


Fig. 1. The structure of the cation showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

† Alternative name: {2-{*N*,*N*-bis[(3,5-dimethylpyrazol-1-yl-*N*^{2})methyl]amino-*N*}ethanol-*O*}(3,5-dimethylpyrazole-*N*^{2})copper(II) diperchlorate.

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copper(II) ion lies 0.354 (1) Å out of the plane defined by N5, N3, N2 and N6 toward O1, with a Cu—O distance of 2.273 (2) Å. This distance is shorter than observed in other pyramidal complexes with coordinated alcohols (2.33–2.382 Å; Luneau, Oshio, Okawa, Koikawa & Kida, 1990; Antolini & Menabue, 1984; Antolini, Menabue, Sola, Battaglia & Corradi, 1986) where the corresponding out-of-plane displacement of the copper(II) ion is smaller. In a related structure, the [*N*,*N*-bis(pyrazol-1-ylmethyl)amine](pyrazole) bis(tetrafluoroborate) complex (Driessen, De Graaff & Wiesmeijer, 1987), with only a weakly coordinated F atom of the tetrafluroborate ion (Cu—F 2.517 Å), the copper(II) ion is coplanar with the base of the pyramid.

Experimental

The title compound was prepared by reaction of stoichiometric amounts of *N*,*N*-bis(3,5-dimethylpyrazol-1-ylmethyl)-1-hydroxy-2-aminoethane (bpmhe) (Driessen, 1982), copper(II) perchlorate and 3,5-dimethylpyrazole in methanol using a similar procedure to that reported by Malachowski *et al.* (1989). Blue crystals suitable for X-ray measurements were grown by slow evaporation of methanol solutions of the complex.

Crystal data

$[Cu(C_5H_8N_2)(C_{14}H_{23}N_5O)]$-$(ClO_4)_2$
$M_r = 635.95$
Triclinic
$P\overline{1}$
$a = 11.4923 (5)$ Å
$b = 14.3227 (7)$ Å
$c = 8.1715 (4)$ Å
$\alpha = 95.655 (1)°$
$\beta = 94.838 (1)°$
$\gamma = 89.567 (1)°$
$V = 1333.7 (1)$ Å^3
$Z = 2$
$D_x = 1.584$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 5784 reflections
$\theta = 1.4$–$26.0°$
$\mu = 1.080$ mm^{-1}
$T = 203 (2)$ K
Prism
0.27 × 0.27 × 0.20 mm
Blue

Data collection

Siemens SMART diffractometer
Area-detector scans
Absorption correction: empirical (Blessing, 1995)
$T_{min} = 0.677$, $T_{max} = 0.806$
13 652 measured reflections
5426 independent reflections

4921 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 26.37°$
$h = -14 \rightarrow 14$
$k = -17 \rightarrow 17$
$l = -10 \rightarrow 10$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.116$
$S = 1.042$
5426 reflections
346 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0625P)^2 + 2.0217P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.013$
$\Delta\rho_{max} = 0.940$ e Å^{-3}
$\Delta\rho_{min} = -0.550$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Cu1—N6	1.968 (2)	Cu1—N3	2.073 (2)
Cu1—N5	1.977 (2)	Cu1—O1	2.273 (2)
Cu1—N1	1.980 (2)		
N6—Cu1—N5	97.10 (9)	N1—Cu1—N3	81.97 (9)
N6—Cu1—N1	94.62 (9)	N6—Cu1—O1	109.22 (9)
N5—Cu1—N1	155.85 (9)	N5—Cu1—O1	92.55 (9)
N6—Cu1—N3	169.23 (9)	N1—Cu1—O1	103.40 (9)
N5—Cu1—N3	82.62 (9)	N3—Cu1—O1	81.53 (8)

The data collection nominally covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle for the crystal and each exposure covered 0.3° in ω. The crystal-to-detector distance was 5.02 cm. Coverage of the unique set is over 97% complete to at least 26° in θ. Crystal decay was monitored by repeating the initial frames at the end of data collection and analyzing the duplicate reflections. The H atom on the alcohol O atom, O1, was located from a difference map and allowed to refine with a displacement parameter 20% greater than O1. The remaining H atoms were placed geometrically and refined with a riding model (including free rotation about C—C bonds for methyl groups) and with U_{iso} constrained to be 1.2 (1.5 for methyl groups) times U_{eq} of the carrier atom.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SMART*. Data reduction: *SAINT* (Siemens, 1995). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL* (Sheldrick, 1994). Software used to prepare material for publication: *SHELXL*93.

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Chlorobis(*N*,*N*′-di-2-pyridyl ketone oximato)gold(III) Hydrate

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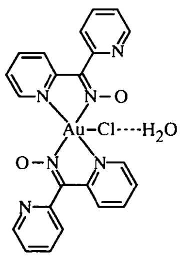
Abstract

Structural analysis of $[AuCl(C_{11}H_8N_3O)_2].H_2O$ revealed the Au atom to be in a square-pyramidal coordination environment with the $Au(C_{11}H_8N_3O)_2Cl$ moieties linked by hydrogen bonds to water molecules to form centrosymmetric dimers. The Au atom achieves a coordination number of five with four N atoms nearly coplanar and the fifth position occupied by a Cl atom.

Comment

The study of di-2-pyridyl ketone oxime (dpk-o) stems from its structural similarities to the di-2-pyridyl ketone (dpk) ligand (Sommerer, Westcott & Abboud, 1994) and its potential to bridge metals to form dimers and infinite chains (Sommerer, Westcott, Jircitano & Abboud, 1995).



(I)

The crystals of the title compound, (I), were found to consist of centrosymmetrically related $Au(dpk\text{-}o)_2Cl$ (Fig. 1) moieties linked by hydrogen bonds to water molecules to form dimers (Fig. 2). The Au atom achieves a coordination number of five with four N atoms nearly coplanar and the fifth position occupied by a Cl atom. The maximum deviation from the mean plane of the N atoms is 0.031 (2) Å and the Au atom is located 0.043 (2) Å below the plane. The geometry is clearly square pyramidal since there is no atom capable of coordination in a sixth position closer than 4.0 Å. The dimer is formed through hydrogen bonds between the chlorine and two separate water molecules, with Cl···O distances of 3.274 (6) and 3.461 (7) Å, respectively, for *OW*···Cl and *OW*···Cl^i [symmetry code: (i) $-x$, $1-y$, $-z$]. Each dpk-o ligand is deprotonated at the oxime function which together with the single chloride results in a neutral complex. Two observations provide evidence for the deprotonation of the dpk-o ligand: first, no electron-density peak

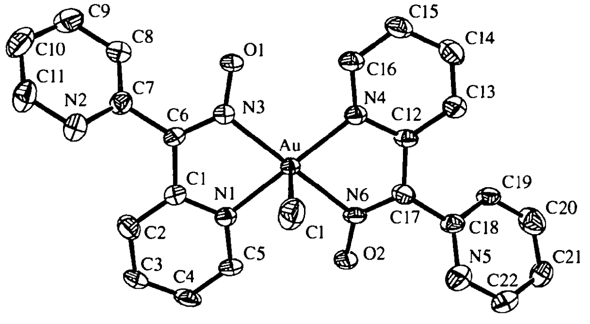


Fig. 1. Displacement ellipsoid drawing of the title compound with ellipsoids drawn at the 50% probability level.

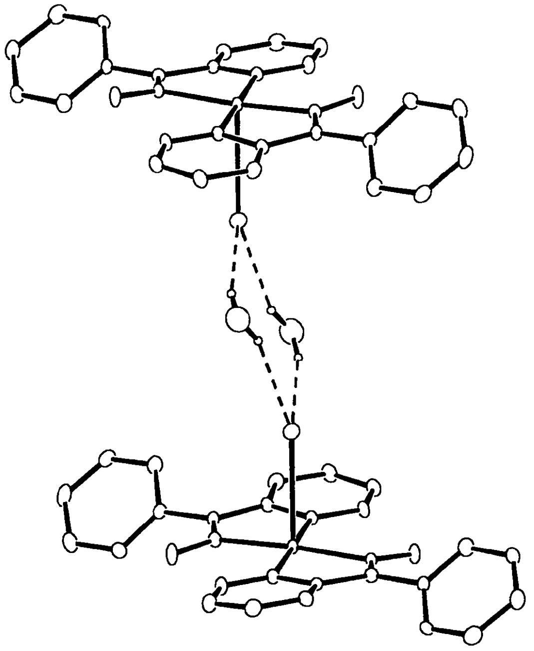


Fig. 2. Displacement ellipsoid drawing of the hydrogen-bonded dimer with the ellipsoids drawn at the 20% probability level.