

Mohamed El Kodadi,^a Malek Fouad,^a Abdelkrim Ramdani,^a Driss Eddike,^{b*} Monique Tillard^c and Claude Belin^c

^aLaboratory of Organo-Physical Chemistry, Department of Chemistry, Faculty of Sciences, University Mohammed The First, BP 524, 60 000 Oujda, Morocco, ^bLaboratory of Inorganic Solid State Chemistry, Department of Chemistry, Faculty of Sciences, University Mohammed The First, BP 524, 60 000 Oujda, Morocco, and ^cLaboratoire des Agrégats Moléculaires et Matériaux Inorganiques, UMR 5072 CC15, Université de Montpellier II, Sciences et Techniques du Languedoc, 2 Place Eugène Bataillon, 34095 Montpellier Cédex 5, France

Correspondence e-mail: eddike@sciences.univ-oujda.ac.ma

Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.038
wR factor = 0.109
Data-to-parameter ratio = 19.6

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Di- μ -chloro-bis[[N,N-bis(3,5-dimethylpyrazol-1-ylmethyl)-1-hydroxyethanamine]copper(II)] dichloride dihydrate

The title compound, $[\text{Cu}_2\text{Cl}_2(\text{C}_{14}\text{H}_{23}\text{N}_5\text{O})_2]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$, crystallizes with the dinuclear molecule located on a centre of symmetry. The coordination geometry around the Cu atom is distorted octahedral, with three N atoms and one Cl atom in the equatorial plane, whereas the axial positions are occupied by the other Cl atom and the hydroxy group. There are two additional Cl^- anions and two water molecules in the asymmetric unit. Infinite hydrogen-bonded chains involving $\text{O}-\text{H} \cdots \text{Cl}$ interactions are formed.

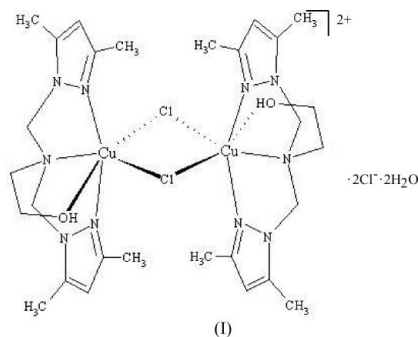
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Comment

Few copper complexes with the *N,N*-bis(3,5-dimethylpyrazol-1-ylmethyl)-1-hydroxy-2-aminoethane ligand (*L*) are known and only two papers related to these complexes have been published; they deal with the syntheses and X-ray structures of $[\text{Cu}(\text{L})(\text{Lo})(\text{ClO}_4)_2]$ (Boyd *et al.*, 1997) and $[\text{Cu}(\text{L})(\text{Lo})(\text{NO}_3)_2]$ (El Kodadi *et al.*, 2003), where *Lo* is 3,5-dimethylpyrazol. In the present work, we are concerned with the same ligand in a copper chloride complex (Fig. 1).



While the Cu atom is found in distorted square-pyramidal coordination in previous complexes (Boyd *et al.*, 1997; El Kodadi *et al.*, 2003), in the present salt two $[\text{C}_{14}\text{H}_{23}\text{ClCuN}_5\text{O}]^+$ cations are related by an inversion centre and linked to each other through two asymmetrical Cu—Cl—Cu bridges. The Cu atoms thus display a distorted octahedral coordination. The equatorial plane of the pseudo-octahedron is formed by three N atoms, *viz.* N1, N3 and N5 [Cu—N1 = 2.1060 (15) Å, Cu—N3 = 2.0045 (17) Å and Cu—N5 = 2.0233 (17) Å], and one Cl atom [Cu—Cl1 = 2.2465 (5) Å]. The axial positions are occupied by the O atom of the hydroxy group [Cu—O1 = 2.3375 (18) Å] and by a Cl atom [Cu—Cl1 = 3.0233 (7) Å]. The cationic dimer $[\text{C}_{28}\text{H}_{46}\text{Cl}_2\text{Cu}_2\text{N}_{10}\text{O}_2]^{2+}$ is linked to the chloride ions *via* $\text{O}-\text{H} \cdots \text{Cl}$ hydrogen bonds [O1 \cdots Cl2 = 3.0255 (18) Å]. Starting from this unit, infinite chains (Fig. 2) are then formed *via* $\text{O}-\text{H} \cdots \text{Cl}$ hydrogen bonds involving two water molecules [O2 \cdots Cl2 = 3.192 (2) Å and O2 \cdots Cl2ⁱⁱ = 3.194 (3) Å; symmetry code: (ii) $2 - x, -y, 2 - z$].

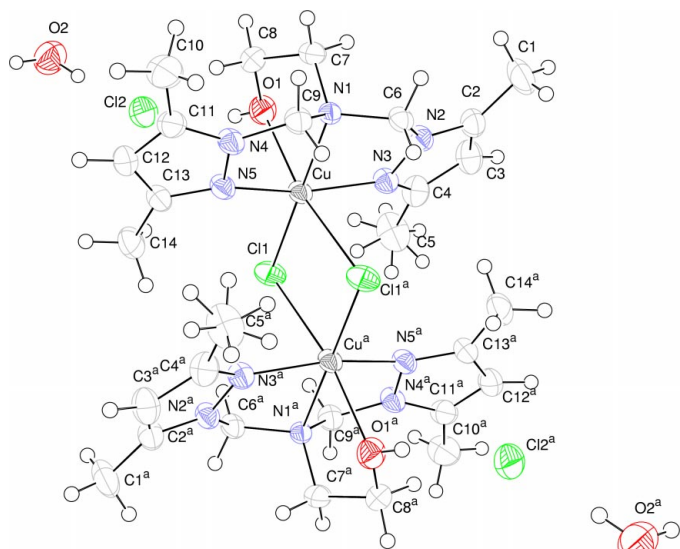


Figure 1

Twice the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level and both disorder components of the methyl groups are shown. Atoms labelled with a superscript 'a' are at the symmetry position $1 - x, 1 - y, 1 - z$.

The strain imposed on the metal coordination environment by the tripodal ligand is also reflected in the chelate angles $[81.14(6) - 78.94(6)^\circ]$, which are less than 90° and very similar to those found by Boyd *et al.* (1997) for the perchlorate $(81.97 - 82.62^\circ)$. The copper–pyrazole bond lengths are considerably shorter than the copper–amine bond distances (Boyd *et al.*, 1997; Driessen *et al.*, 1994). This difference is a result of the greater π -bonding ability of pyrazole ligands compared with alkylamines, which are exclusively σ donors. The Cu–Cl bond lengths are comparable to those reported for $[\text{Cu}(\text{ddae})\text{Cl}_2] \cdot \text{C}_2\text{H}_5\text{OH}$ {ddae is bis[2-(3,5-dimethyl-1-pyrazolyl)ethyl]-amine} (Driessen *et al.*, 1994) and the Cu–O distance is similar to that reported for the copper(II) complex with coordinated alcohols (Luneau *et al.*, 1990; Antolini & Menabue, 1984; Antolini *et al.*, 1986).

Experimental

The copper(II) complex was prepared by the addition of a solution of the tridentate ligand *N,N*-bis(3,5-dimethylpyrazol-1-ylmethyl)-1-hydroxy-2-aminoethane (0.277 g, 1 mmol) in methanol (3 ml) to a solution of copper(II) dichloride $[\text{CuCl}_2 \cdot 2\text{H}_2\text{O}]$ (0.170 g, 1 mmol) in methanol (3 ml). The resulting solution was filtered and allowed to stand at 298 K. Blue crystals formed after a few days and were filtered off and washed with small amounts of cold methanol and then dried in air.

Crystal data

$[\text{Cu}_2\text{Cl}_2(\text{C}_{14}\text{H}_{23}\text{N}_5\text{O})_2]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$
 $M_r = 859.67$
 Triclinic, $P\bar{1}$
 $a = 8.5418(6) \text{ \AA}$
 $b = 11.1878(8) \text{ \AA}$
 $c = 11.6189(7) \text{ \AA}$
 $\alpha = 66.625(6)^\circ$
 $\beta = 69.332(6)^\circ$
 $\gamma = 73.642(6)^\circ$
 $V = 940.57(11) \text{ \AA}^3$

$Z = 1$
 $D_x = 1.518 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 5565 reflections
 $\theta = 3.0 - 30.3^\circ$
 $\mu = 1.46 \text{ mm}^{-1}$
 $T = 293(2) \text{ K}$
 Parallelepiped, blue
 $0.32 \times 0.15 \times 0.13 \text{ mm}$

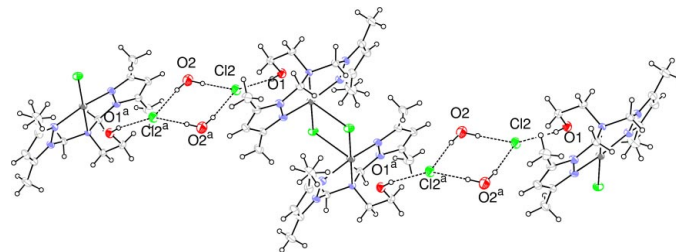


Figure 2

A view of the hydrogen-bonded chains in the crystal structure of (I). Displacement ellipsoids are drawn at the 30% probability level. Atoms labelled with a superscript 'a' are at the symmetry position $1 - x, 1 - y, 1 - z$.

Data collection

Oxford Diffraction Xcalibur CCD diffractometer
 ω scans
 15 083 measured reflections
 4526 independent reflections
 3849 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.062$
 $\theta_{\text{max}} = 28.0^\circ$
 $h = -11 \rightarrow 11$
 $k = -14 \rightarrow 14$
 $l = -15 \rightarrow 15$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.109$
 $S = 1.10$
 4526 reflections
 231 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0617P)^2 + 0.1229P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.72 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.67 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Cu–N3	2.0045 (17)	Cu–Cl1	2.2465 (5)
Cu–N5	2.0233 (17)	Cu–O1	2.3375 (18)
Cu–N1	2.1060 (15)	Cu–Cl1 ⁱ	3.0233 (7)
N3–Cu–N5	161.77 (7)	N1–Cu–O1	78.94 (6)
N3–Cu–N1	81.14 (6)	Cl1–Cu–O1	102.37 (5)
N5–Cu–N1	81.42 (6)	N3–Cu–Cl1 ⁱ	86.70 (5)
N3–Cu–Cl1	99.50 (5)	N5–Cu–Cl1 ⁱ	86.93 (5)
N5–Cu–Cl1	97.81 (5)	N1–Cu–Cl1 ⁱ	86.37 (4)
N1–Cu–Cl1	178.50 (4)	Cl1–Cu–Cl1 ⁱ	92.30 (2)
N3–Cu–O1	91.59 (7)	O1–Cu–Cl1 ⁱ	165.30 (4)
N5–Cu–O1	90.26 (7)		

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

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1–HO1 \cdots Cl2	0.74 (3)	2.30 (3)	3.0255 (18)	165 (3)
O2–HO21 \cdots Cl2	0.92 (3)	2.29 (3)	3.192 (2)	170 (3)
O2–HO22 \cdots Cl2 ⁱⁱ	0.90 (3)	2.30 (3)	3.194 (3)	172 (3)

Symmetry code: (ii) $2 - x, -y, 2 - z$.

Except for the hydroxy and water H atoms, which were located in a Fourier difference map and whose positions were refined freely

[O1–H O1 = 0.74 (3) Å, and O2–H = 0.92 (3) and 0.90 (3) Å], all others H atoms were placed in idealized positions (C–H = 0.93–0.97 Å) and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ [$1.5U_{\text{eq}}(\text{C})$ for H atoms of methyl groups]. The isotropic displacement parameter was refined for the hydroxy H atom and fixed at 0.05 \AA^2 for water H atoms.

Data collection: *CrysAlisCCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlisRED*, Oxford Diffraction, 2003); data reduction: *CrysAlisRED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

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