

Redetermination of (2,2'-bipyridyl- κ^2N,N')chloro-(glycinato- $\kappa^2N:O$)copper(II) dihydrate

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Key indicators

Single-crystal X-ray study
 $T = 123\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.030
 wR factor = 0.086
Data-to-parameter ratio = 16.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The redetermined structure of the title compound, $[\text{Cu}(\text{C}_2\text{H}_4\text{NO}_2)\text{Cl}(\text{C}_{10}\text{H}_8\text{N}_2)] \cdot 2\text{H}_2\text{O}$, confirms that reported earlier [Neitzel & Desiderato (1975). *Cryst. Struct. Commun.* **4**, 333–336] with respect to the non-H atoms. All the H atoms have been located in the present study and the network of $\text{N}-\text{H} \cdots \text{O}$, $\text{N}-\text{H} \cdots \text{Cl}$, $\text{O}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{Cl}$ hydrogen bonds has been elucidated.

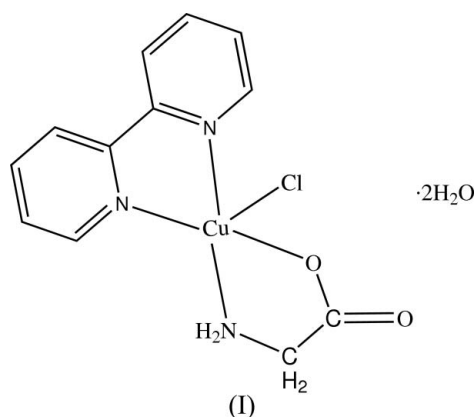
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Comment

Certain transition metal complexes have remarkable antimicrobial or fungicidal activity (Okide *et al.*, 2000; Patel *et al.*, 1999), or have redox activity and act as catalysts for metal-induced toxicity or carcinogenesis through processes which are interpreted as Fenton-type reactions (Kasprzak, 2002). Furthermore, aromatic heterocycles can stack with nucleobases and enhance complex formation with DNA, which is the principal target in the chemotherapy of tumours (Shehata, 2001).



For these reasons, various ternary copper complexes with N,N' -bidentate heterocyclic ligands, such as bipyridine (bpy), 1,10-phenanthroline (phen) or bipyridyl amine (bpa), and O,O' -bidentate ligands, such as phthalic acid or *p*-hydroxybenzenecarboxylic acid, have been synthesized and their structures determined (Wang & Okabe, 2004; Wang & Okabe, 2005*a,b*).

As part of these studies, we have redetermined the structure of the title complex, (I), which was previously studied by Neitzel & Desiderato (1975). In this study, all the H atoms have been located and the hydrogen-bonding scheme has been elucidated.

The structure of (I) is shown in Fig. 1 and compares well with the previous study (Neitzel & Desiderato, 1975). The Cu atom has distorted square-pyramidal geometry, being bonded

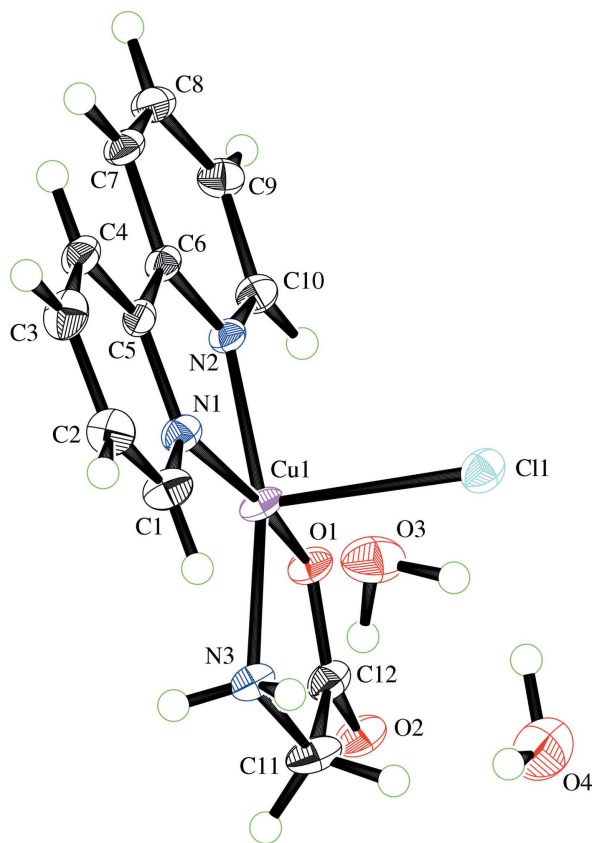


Figure 1
A view of (I), showing 50% probability displacement ellipsoids.

to two N atoms of a bidentate bpy ligand, the N and O atoms of a glycinate ligand in a distorted square plane, and a Cl⁻ anion at the apical site of the pyramid (Table 1). The deviation of atom Cu1 from the mean plane formed by the atoms N1, N2, N3 and O1 is 0.2141 (11) Å. The long Cu–Cl bond length is explained by the well known Jahn–Teller effect.

Complex molecules and non-coordinated water molecules are connected to each other by way of a network of hydrogen bonds (Fig. 2, Table 2). In graph theory notation (Bernstein *et al.*, 1995), these linkages result in an $R_2^2(8)$ ring formed through atoms O4, Cl1, Cu1, N3 and Cl1ⁱ (see Fig. 2 for symmetry codes) and two short $C_2^2(5)$ chains, through atoms N3, O3 and Cl1, as well as through atoms N3, O3 and O2.

Experimental

Firstly, bipyridine [120 mg dissolved in 4 ml of an 80% (v/v) methanol–water mixture] was reacted with CuCl₂·2H₂O [130 mg in 1 ml of an 80% (v/v) methanol–water mixture] (molar ratio 1:1) at room temperature, and 150 mg of the resulting light-blue precipitate was collected and dried. This was then dissolved [5.0 mg in 3 ml of a 70% (v/v) methanol–water mixture] and reacted with glycine [1.14 mg in 1 ml of a 70% (v/v) methanol–water mixture] (molar ratio 1:1) at 343 K until the volume of the reaction mixture was concentrated to ca 1 ml. The concentrated mixture was left to stand at room temperature, and yielded blue prismatic crystals of (I) over a period of days.

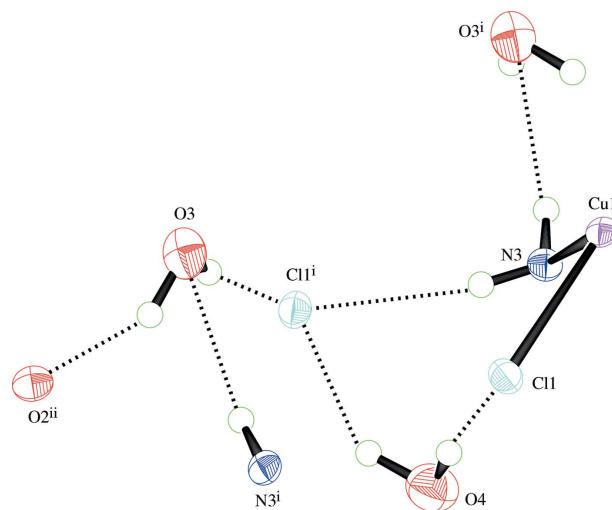


Figure 2
A fragment of (I), showing the different kinds of hydrogen bonds (dashed lines). [Symmetry codes: (i) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (ii) $-x, \frac{1}{2} + y, \frac{1}{2} - z$.]

Crystal data

[Cu(C₂H₄NO₂)Cl(C₁₀H₈N₂)]·2H₂O
M_r = 365.28
 Monoclinic, $P2_1/c$
a = 10.47 (2) Å
b = 18.32 (2) Å
c = 7.646 (9) Å
 β = 103.92 (5)°
V = 1424 (4) Å³
Z = 4

D_x = 1.704 Mg m⁻³
 Mo K α radiation
 Cell parameters from 10955 reflections
 θ = 3.0–27.5°
 μ = 1.74 mm⁻¹
T = 123.1 K
 Prism, blue
 0.30 × 0.10 × 0.05 mm

Data collection

Rigaku R-Axis RAPID diffractometer
 ω scans
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
T_{min} = 0.812, *T_{max}* = 0.917
 13847 measured reflections

3249 independent reflections
 2104 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.032
 θ_{max} = 27.5°
h = -13 → 13
k = -23 → 23
l = -9 → 9

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.086$
S = 0.94
 3249 reflections
 203 parameters

All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0561P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.40 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{min} = -0.63 \text{ e } \text{Å}^{-3}$

Table 1
Selected bond lengths (Å).

Cu1–O1	1.957 (2)	Cu1–N3	2.006 (2)
Cu1–N1	2.002 (2)	Cu1–Cl1	2.637 (5)
Cu1–N2	1.999 (2)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N3-H9\cdots Cl1^i$	0.90	2.60	3.453 (7)	157
$N3-H10\cdots O3^i$	0.90	2.40	3.227 (7)	153
$O3-H13\cdots O2^{ii}$	0.95	1.94	2.799 (3)	148
$O3-H14\cdots Cl1^i$	0.95	2.26	3.199 (7)	168
$O4-H15\cdots Cl1$	0.99	2.15	3.137 (7)	172
$O4-H16\cdots Cl1^i$	0.99	2.33	3.194 (7)	145

Symmetry codes: (i) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$.

The water H atoms were located in difference maps and refined as riding in their as-found relative positions, with $U_{iso}(H) = 1.2U_{eq}(O)$. The other H atoms were positioned geometrically (N–H = 0.90 and C–H = 0.93–0.97 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(\text{carrier})$.

Data collection: *RAPID AUTO* (Rigaku, 2003); cell refinement: *RAPID AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2004) and *CRYSTALS* (Betteridge *et al.*, 2003); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999) and *DIRDIF99* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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