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Aqua(2,2′-bipyridyl-κ^2N,N'){2-[(3,5-dichloro-2-oxidobenzylidene)amino]-3-methylpentanoato-κ^3N,O,O'}copper(II) 2.5-hydrate

Xiao-Zhen Feng, Shu-Hua Zhang,* Zheng Liu,* Guang-Zhao Li and Li-Xia Jin

Key Laboratory of Non-Ferrous Metal Materials and Processing Technology, Department of Material and Chemical Engineering, Guilin University of Technology, Ministry of Education, Guilin 541004, People's Republic of China

Correspondence e-mail: zsh720108@21cn.com, lisa4.6@163.com

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In the title compound, $[Cu(C_{13}H_{13}Cl_2NO_3)(C_{10}H_8N_2)]\cdot 2.5H_2O$, the Cu^{II} atom is coordinated in a slightly distorted tetragonal–pyramidal geometry by two O atoms and one N atom from the chiral ligand 2-[(3,5-dichloro-2-oxidobenzylidene)amino]-3-methylpentanoate and two N atoms from 2,2′-bipyridine. The asymmetric unit consists of two Cu^{II} complexes and five water molecules. In the crystal structure, the water molecules are linked by O—H···O hydrogen bonds into five-membered rings that are further linked into chains.

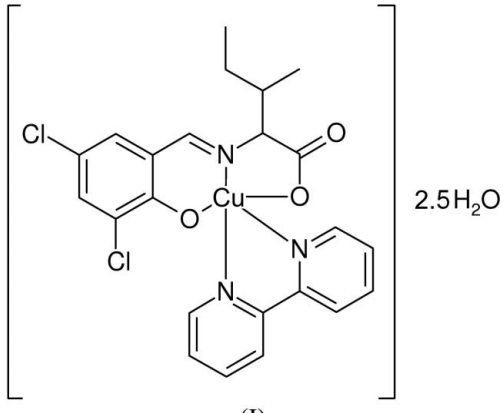
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.013 Å
R factor = 0.066
wR factor = 0.147
Data-to-parameter ratio = 11.8

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

Comment

The controlled construction of complexes with stereogenic metal centres is an important task because of its potential impact on various areas of chemical research, such as asymmetric catalysis, supramolecular chemistry, or biological recognition. Transfer of chirality from chiral, non-racemic organic ligands to metal centres with a variety of coordination geometries has attracted great interest (Knof & Von Zelewsky, 1999; Brunner, 1999; Hamann *et al.*, 2004).



(I)

Recently, compounds containing water dimers (Ghosh & Bharadwaj, 2003), water rings (Moorthy *et al.*, 2002; Ugalde *et al.*, 2000; Ghosh & Bharadwaj, 2004), water chains (Neogi & Bharadwaj, 2005; Cheruzel *et al.*, 2003; Ghosh *et al.*, 2005), metal–water chains (Ye *et al.*, 2004), water networks (Zhang *et al.*, 2005) and mixed water/methanol clusters (Raghuraman *et al.*, 2003) have also attracted interest. Water chains appear to facilitate selective permeation (Tajkhorshid *et al.*, 2002) of water across membranes and also to be important (Zaslavsky & Gennis, 2000) in the control of proton fluxes in a variety of biomolecules.

The title compound, (I), is a chiral Cu^{II} complex containing a ligand constructed from 2-amino-3-methylpentanoic acid

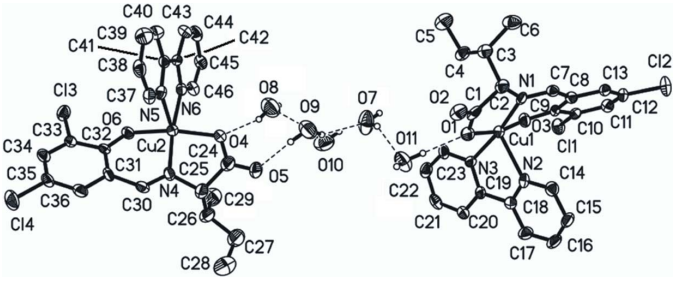


Figure 1
The asymmetric unit of (I), showing displacement ellipsoids drawn at the 30% probability level for non-H atoms. The dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted.

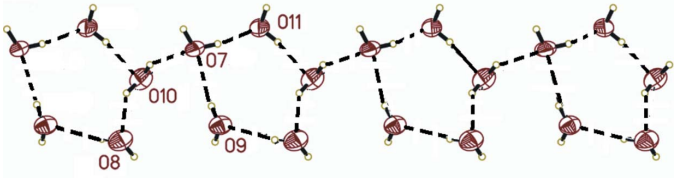


Figure 2
Water molecules linked by O—H···O hydrogen bonds (dashed lines) into five-membered rings that form chains. Displacement ellipsoids are shown at the 30% probability level for O atoms.

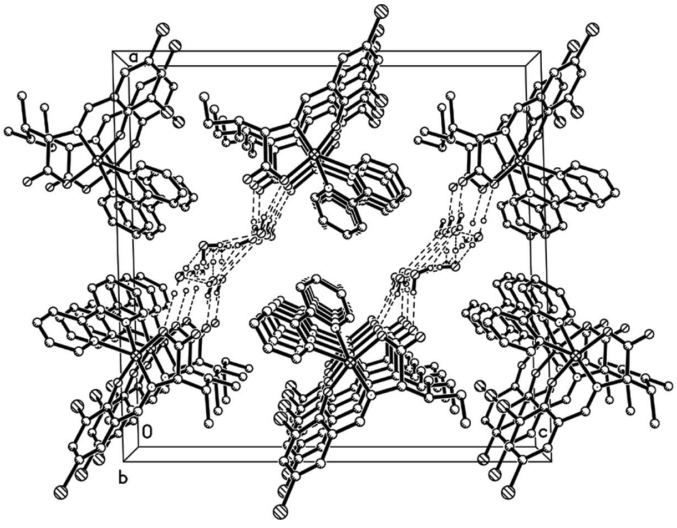


Figure 3
Projection of (I) along *b*, showing layers of Cu^{II} complexes with the water molecules lying between them. Dashed lines denote hydrogen bonds and H atoms not involved in hydrogen bonding have been omitted.

and 3,5-dichloro-2-hydroxybenzaldehyde. Each Cu^{II} atom is coordinated by two O atoms and one N atom from the chiral ligand and two N atoms from 2,2′-bipyridine, forming a slightly distorted tetragonal–pyramidal geometry (Table 1). The asymmetric unit (Fig. 1) comprises two Cu^{II} complexes and five water molecules. The water molecules are linked by O—H···O hydrogen bonds (Table 2), forming five-membered rings, which are linked further into chains along *b* (Fig. 2). The water chains lie between layers of Cu^{II} complexes (Fig. 3), forming O—H···O hydrogen bonds to the O atoms of the carboxylate groups.

Experimental

A solution of 2-amino-3-methylpentanoic acid (2 mmol, 0.262 g) and caustic potash (2 mmol, 0.112 g) in distilled water (15 ml) was added slowly to a solution of 3,5-dichloro-2-hydroxybenzaldehyde (2 mmol, 0.382 g) in ethanol (20 ml). The mixture was stirred for 30 min at 333 K then added slowly to a solution of copper(II) nitrate (1 mmol, 0.291 g) in distilled water (10 ml). This mixture was stirred and refluxed for 4 h at 333 K; 2,2′-bipyridyl (2 mmol, 0.312 g) was then added and the reaction continued for a further 2 h. The solution was filtered and the filtrate was left to stand at room temperature. Blue prisms suitable for X-ray diffraction were obtained in a yield of 46% (based on copper nitrate). Elemental analysis found: C 48.65, H 4.85, N 7.44%; calculated: C 48.73, H 4.62, N 7.41%.

Crystal data

$[Cu(C_{13}H_{13}Cl_2NO_3)(C_{10}H_8N_2)]\cdot 2.5H_2O$
$M_r = 1133.82$
Monoclinic, $P2_1$
$a = 19.463$ (3) Å
$b = 6.391$ (2) Å
$c = 20.302$ (2) Å
$\beta = 91.522$ (3)°
$V = 2524.6$ (9) Å^3
$Z = 2$
$D_x = 1.492$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.12$ mm^{-1}
$T = 298$ (2) K
Prism, blue
0.46 × 0.15 × 0.07 mm

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.627$, $T_{max} = 0.926$
13053 measured reflections
7364 independent reflections
4605 reflections with $I > 2\sigma(I)$
$R_{int} = 0.070$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.147$
$S = 0.99$
7364 reflections
622 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0458P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.89$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}
Absolute structure: Flack (1983), 2500 Friedel pairs
Flack parameter: 0.03 (2)

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.972 (6)	Cu2—O4	1.968 (6)
Cu1—O3	1.935 (6)	Cu2—O6	1.936 (5)
Cu1—N1	1.916 (7)	Cu2—N4	1.908 (7)
Cu1—N2	2.217 (7)	Cu2—N5	2.220 (7)
Cu1—N3	1.990 (7)	Cu2—N6	1.970 (7)
N1—Cu1—O3	91.5 (3)	N4—Cu2—O6	91.7 (3)
N1—Cu1—O1	82.8 (3)	N4—Cu2—O4	83.1 (3)
O3—Cu1—O1	167.6 (2)	O6—Cu2—O4	166.6 (2)
N1—Cu1—N3	172.2 (3)	N4—Cu2—N6	171.4 (3)
O3—Cu1—N3	92.5 (3)	O6—Cu2—N6	93.3 (2)
O1—Cu1—N3	91.9 (3)	O4—Cu2—N6	90.4 (3)
N1—Cu1—N2	108.6 (3)	N4—Cu2—N5	108.3 (3)
O3—Cu1—N2	94.3 (2)	O6—Cu2—N5	95.2 (2)
O1—Cu1—N2	98.0 (2)	O4—Cu2—N5	98.1 (2)
N3—Cu1—N2	77.8 (3)	N6—Cu2—N5	78.2 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O7—H47···O11	0.85	1.97	2.693 (11)	142
O7—H48···O2^{i}	0.85	1.96	2.790 (10)	164
O8—H49···O9^{i}	0.85	2.09	2.825 (14)	144
O8—H50···O4	0.85	2.06	2.880 (10)	162
O9—H51···O7	0.85	2.15	2.907 (9)	148
O9—H52···O5	0.85	1.85	2.699 (9)	175
O10—H53···O8	0.85	1.96	2.705 (11)	146
O10—H54···O7	0.85	1.95	2.695 (13)	146
O11—H55···O1	0.85	1.95	2.796 (9)	174
O11—H56···O10^{ii}	0.85	1.88	2.724 (11)	171

Symmetry codes: (i) $x, y-1, z$; (ii) $x, y+1, z$.

H atoms bound to C atoms were positioned geometrically and refined as riding atoms, with C—H distances of 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). H atoms of the water molecules were located in difference Fourier maps. The O—H distances were normalized to 0.85 Å and the H atoms were then allowed to ride on their parent O atoms, with U_{iso}(H) = 1.5U_{eq}(O). The anisotropic displacement parameters of atoms C2–C6 and C24–C29 were restrained to be identical with a standard uncertainty of 0.01 Å^2, or 0.02 Å^2 for terminal atoms.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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