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Aquachloro[5-hydroxyisophthalato(1−)](1,10-phenanthroline)copper(II) 2.5-hydrate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
H-atom completeness 96%
Disorder in solvent or counterion
R factor = 0.050
wR factor = 0.156
Data-to-parameter ratio = 12.5

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

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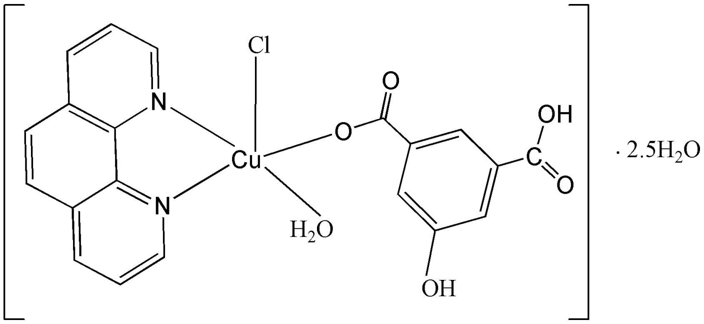
In the title compound, $[Cu(C_8H_5O_5)Cl(C_{12}H_8N_2)(H_2O)]\cdot 2.5H_2O$, the Cu^{II} atom is in a square-pyramidal geometry defined by one Cl$^-$ anion, one aqua O atom, one carboxyl O atom belonging to one 5-hydroxyisophthalate anion, and two N atoms from a 1,10-phenanthroline molecule. O—H···O hydrogen bonds involving 5-hydroxyisophthalate anions, the aqua ligands and the uncoordinated water molecules, along with weak O—H···Cl hydrogen bonds, link the mononuclear units into a three-dimensional network structure.

Comment

Numerous isophthalate and 5-hydroxyisophthalate complexes have been extensively studied. They show a diversity of structures and a variety of framework topologies (Chen & Liu, 2002; Hou *et al.*, 2003; Plater *et al.*, 2001; Xu & Li, 2004; Wen *et al.*, 2004). It should be pointed out that the synthetic conditions, such as H-atom receptors, temperature, solvents *etc.*, play an important role in determining the compositions of these complexes. For example, the previously reported complex $[Cu_2(C_{12}H_8N_2)_2(C_8H_4O_5)_2]\cdot 3H_2O$ (Xiao *et al.*, 2004) and the title complex, $[CuCl(C_{12}H_8N_2)(C_8H_5O_5)(H_2O)]\cdot 2.5H_2O$, (I), were synthesized by the reaction of $CuCl_2\cdot 2H_2O$, 5-hydroxyisophthalic acid and 1,10-phenanthroline. The former was obtained in a 20 ml mixture of *N*,*N*-dimethylformamide, water and methane (2:1:1 *v*/*v*/*v*), while (I) was obtained in a 20 ml solution in *N*,*N*-dimethylformamide.



(I)

In (I), the Cu^{II} atom has a five-coordinate environment defined by one Cl$^-$ anion, one aqua O atom, one carboxyl O atom belonging to one 5-hydroxyisophthalate anion, and two N atoms from a 1,10-phenanthroline molecule (Fig. 1). The geometry around the Cu^{II} atom is square pyramidal. The basal plane (atoms O1, O6, N1 and N2) consists of two N atoms from a 1,10-phenanthroline molecule, one carboxyl O atom from a 5-hydroxyisophthalate anion, and one aqua O atom.

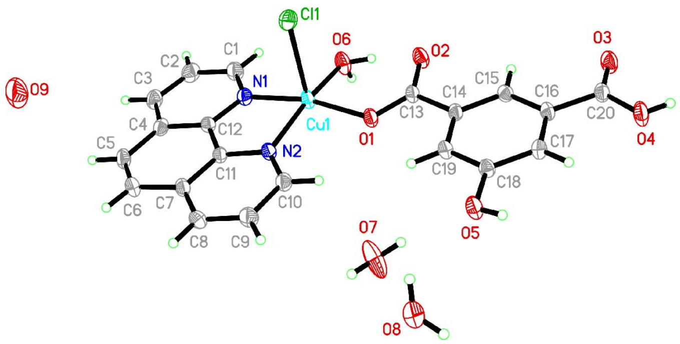


Figure 1
The molecular structure of the asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

The O1—Cu1—N1 and O6—Cu1—N2 bond angles are 167.95 (13) and 165.74 (15)°, respectively. The other angles around the Cu^{II} centre are in the range 81.89 (14)–98.51 (11) Å (Table 1). The apical position is occupied by a Cl^- anion, the Cu1—Cl1 bond distance of 2.5463 (12) Å being longer than the apical Cu—Cl bond distance found in the previously reported compound $[CuCl(C_{12}H_8N_2)_2]\cdot(C_8H_4NO_6)\cdot 2H_2O$ [2.287 (2) Å; Ye *et al.*, 2004]. The 1,10-phenanthroline system is almost coplanar with the phenyl ring of the 5-hydroxyisophthalate anion, the dihedral angle between the planes being 1.9 (2)°.

In the crystal structure of (I), there is an intramolecular hydrogen bond between the coordinated aqua O6 atom and the uncoordinated carboxylate O2 atom. The hydroxy group of the 5-hydroxyisophthalate anion is involved in a weak intermolecular O5—H5···Cl1($1 - x, 2 - y, 1 - z$) hydrogen bond and an intermolecular O7—H7*B*···O5 hydrogen bond. Moreover, O—H···O intermolecular hydrogen bonds are formed between the water molecules and the carboxylate O atoms (Table 2). These interactions link the mononuclear units to form a three-dimensional network structure (Fig. 2).

Experimental

A solution of $CuCl_2\cdot 2H_2O$ (0.5 mmol, 0.852 g) and 5-hydrogenisophthalic acid (0.5 mmol, 0.910 g) in 15 ml *N*,*N*-dimethylformamide was added slowly to a solution of 1,10-phenanthroline (0.5 mmol, 0.991 g) in 10 ml of *N*,*N*-dimethylformamide . The mixture was left to stand at room temperature for about a month to afford blue crystals of (I).

Crystal data

$[Cu(C_8H_5O_5)Cl(C_{12}H_8N_2)(H_2O)]\cdot 2.5H_2O$
$M_r = 523.37$
Triclinic, $P\overline{1}$
$a = 8.8508\ (10)$ Å
$b = 10.7516\ (12)$ Å
$c = 12.7592\ (15)$ Å
$\alpha = 114.218\ (2)°$
$\beta = 95.770\ (2)°$
$\gamma = 94.022\ (2)°$
$V = 1093.4\ (2)$ Å^3
$Z = 2$
$D_x = 1.590$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2931 reflections
$\theta = 2.3$–$24.4°$
$\mu = 1.17$ mm^{-1}
$T = 298\ (2)$ K
Prism, blue
0.33 × 0.15 × 0.13 mm

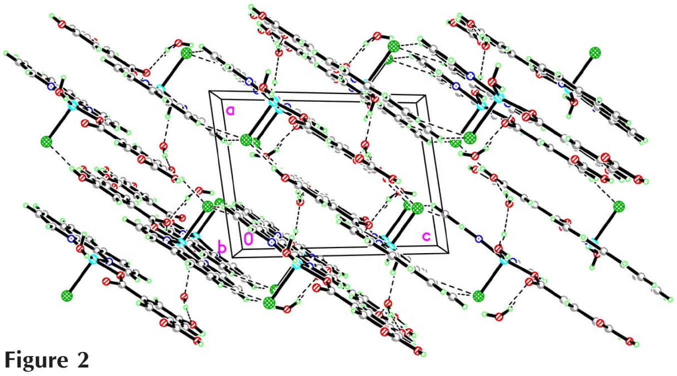


Figure 2
The crystal packing of (I), viewed along the *b* axis, showing the three-dimensional network structure formed by the hydrogen-bonding interactions (dashed lines).

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.810$, $T_{max} = 0.862$
8038 measured reflections
3911 independent reflections
3445 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 25.2°$
$h = -10 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.156$
$S = 1.13$
3911 reflections
313 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0785P)^2 + 1.351P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.28$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—Cl1	2.5463 (12)	Cu1—N1	2.009 (4)
Cu1—O1	1.957 (3)	Cu1—N2	2.010 (4)
Cu1—O6	1.980 (4)		
Cl1—Cu1—O1	95.62 (8)	O1—Cu1—N1	167.95 (13)
Cl1—Cu1—O6	94.93 (11)	O1—Cu1—N2	90.42 (14)
Cl1—Cu1—N1	94.68 (10)	O6—Cu1—N1	92.33 (15)
Cl1—Cu1—N2	98.51 (11)	O6—Cu1—N2	165.74 (15)
O1—Cu1—O6	92.98 (14)	N1—Cu1—N2	81.89 (14)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O8^{i}	0.82	1.82	2.623 (6)	167
O5—H5···Cl1^{ii}	0.82	2.18	2.998 (4)	172
O6—H6*A*···O2	0.82	1.86	2.601 (5)	149
O6—H6*B*···O7^{iii}	0.89	1.77	2.642 (5)	167
O7—H7*A*···O3^{iv}	0.84 (4)	1.93 (4)	2.754 (6)	167 (6)
O7—H7*B*···O5	0.83 (4)	1.97 (5)	2.771 (6)	162 (4)
O8—H8*A*···O2^{ii}	0.82 (3)	2.05 (4)	2.844 (6)	163 (4)
O8—H8*B*···O9^{v}	0.81 (3)	2.05 (4)	2.831 (10)	162 (5)

Symmetry codes: (i) $x, y + 1, z$; (ii) $-x + 1, -y + 2, -z + 1$; (iii) $-x + 2, -y + 2, -z + 1$; (iv) $x, y - 1, z$; (v) $x, y + 1, z + 1$.

The H atoms of the water molecules were located in a Fourier difference map and refined isotropically, with O—H distances restrained to 0.82 (2) Å and the H7*A*···H7*B* and H8*A*···H8*B*

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distances restrained to 1.45 (2) Å. The remaining H atoms were positioned geometrically (C—H = 0.93 Å and O—H = 0.82 Å) and allowed to ride on their parent atoms, with $U_{\rm iso}$(H) values equal to $1.2U_{\rm eq}$(C) or $1.5U_{\rm eq}$(O). At this stage, the maximum difference density of 3.59 Å^{-3} indicated the presence of a possible atom site. This peak was found near atom H8*B*, at a hydrogen-bonding distance of 2.27 Å. Attempts to refine this peak as a water O atom (O9) with full occupancy resulted in a high $U_{\rm iso}$ value, and hence it was refined with occupancy 0.5. The occupancy factor of atom O9 was initially found to be 0.51 (1) and later fixed at 0.50. The H atoms of this water molecule could not be located in the difference map.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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References

Bruker (2002). *SADABS* (Version 2.03), *SAINT* (Version 6.02), *SMART* (Version 5.62) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Winsonsin, USA.

Chen, X. M. & Liu, G. F. (2002). *Chem. Eur. J.* **8**, 4811–4817.

Hou, Y., Shen, E., Wang, S., Wang, E., Xiao, D., Li, Y., Xu, L. & Hu, C. (2003). *Inorg. Chem. Commun.* **6**, 1347–1349.

Plater, J. M., Foreman, M. R. St. J., Howie, R. A., Skakle, J. M. S., McWilliam, S. A., Coronado, E. & Gomez-Garcia, C. J. (2001). *Polyhedron*, **20**, 2293–2303.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Wen, Y. H., Cheng, J. K., Zhang, J., Li, Z. J., Kao, Y. & Yao, Y. G. (2004). *Inorg. Chem. Commun.* **7**, 1120–1123.

Xiao, H. P., Shi, Q. & Ye, M. D. (2004). *Acta Cryst.* E**60**, m1498–m1500.

Xu, H. & Li, Y. (2004). *(2004). J. Mol. Struct.* **690**, 137–143.

Ye, M. D., Xiao, H. P. & Hu, M. L. (2004). *Acta Cryst.* E**60**, m1516–m1518.

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