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Di-μ-5-hydroxyisophthalato-bis[aqua(1,10-phenanthroline)copper(II)] trihydrate

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In the title compound, $[Cu_2(C_8H_4O_5)_2(C_{12}H_8N_2)_2(H_2O)_2]\cdot 3H_2O$, the Cu complex forms a centrosymmetric dimer with a Cu···Cu separation of 8.198 (3) Å. In the 5-hydroxyisophthalate ligands, two carboxylate groups coordinate two copper(II) cations in μ_2-bridging bidentate coordination modes.

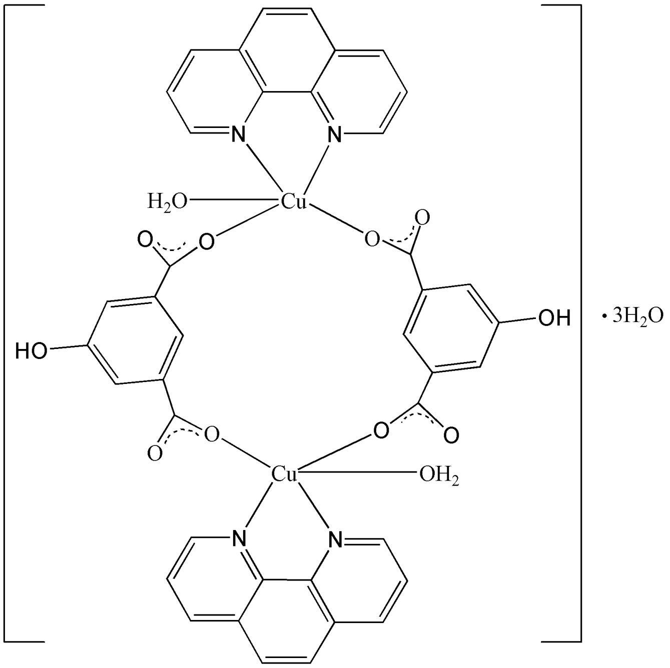
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.010 Å
Disorder in solvent or counterion
R factor = 0.084
wR factor = 0.172
Data-to-parameter ratio = 11.4

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

Comment

Encouraged by our successful synthesis of coordination polymers using transition metals, 1,4-benzenedicarboxylic acid and 1,10-phenanthroline, which showed a diversity of structures (Sun *et al.*, 2001; Xiao, Hu & Li, 2004; Xiao, Li *et al.*, 2004; Zhu *et al.*, 2004), we investigated the effect of replacing 1,4-benzenedicarboxylic acid with 5-hydroxyisophthalic acid. In a mixed solvent of dimethylformamide and methanol at room temperature, we obtained the title compound, (I).



(I)

In (I), each Cu^{II} cation has a five-coordinate environment consisting of one aqua O atom, two carboxyl O atoms belonging to two 5-hydroxyisophthalate anions and two N atoms from a 1,10-phenanthroline molecule (Fig. 1). The geometry around the Cu^{II} cation is distorted square-pyramidal. The basal plane [O3^{i}/O6/N1/N2; symmetry code: (i) $-x+2$, $y+2$, $-z+2$] is built of two 1,10-phenanthroline N

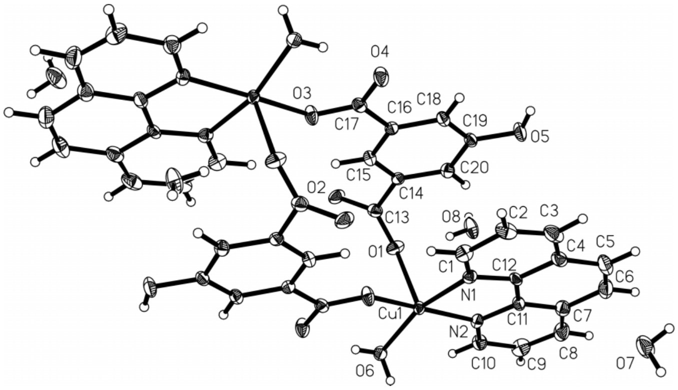


Figure 1
The coordination environment of copper in (I), showing the atom numbering and displacement ellipsoids at the 30% probability level. Unlabelled atoms are related to labelled atoms by $2 - x, 2 - y, 2 - z$.

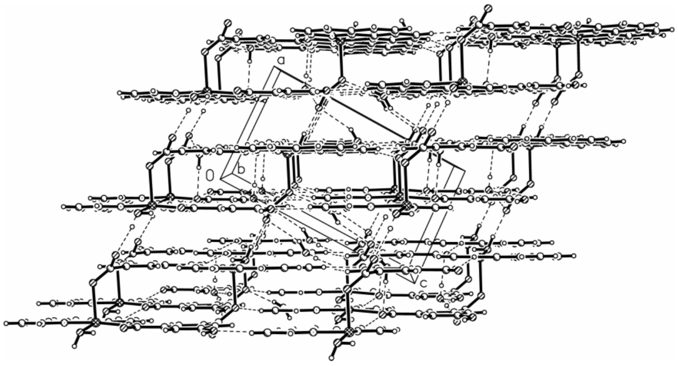


Figure 2
The crystal packing of (I), viewed along the *b* axis. Intermolecular hydrogen bonds are indicated by dashed lines.

atoms, one carboxyl O atom from each of two 5-hydroxyisophthalate anions and one aqua O atom. The apical position is occupied by one carboxyl O atom (O1), the corresponding axial bond length [2.274 (5) Å for Cu1—O1] being longer than the equatorial Cu1—O3^{i} [1.942 (4) Å] and Cu1—O6 [1.974 (4) Å] bond lengths (Table 1). The two 5-hydroxyisophthalate carboxylate groups coordinate two copper(II) cations in μ_2-bridging bidentate coordination modes. The Cu complex forms a centrosymmetric dimer with a Cu$\cdots$Cu separation of 8.198 (3) Å.

The phenol H atom of the 5-hydroxyisophthalate anion is involved in an intermolecular O—H$\cdots$O hydrogen bond (Table 2). The coordinated and free water molecules form hydrogen bonds, with the carboxylate O atoms as acceptors (Table 2). These interactions link the dimer units into a three-dimensional network structure (Fig. 2).

Experimental

A solution of $CuCl_2 \cdot 2H_2O$ (0.5 mmol) and 5-hydroxyisophthalic acid (0.5 mmol) in a mixture of dimethylformamide and water (15 ml, 2:1 *v*/*v*) was added slowly to a solution of 1,10-phenanthroline (0.5 mmol) in methanol (5 ml). The mixture was left to stand at room temperature for about a month to afford blue crystals.

Crystal data

$[Cu_2(C_8H_4O_5)_2(C_{12}H_8N_2)_2(H_2O)_2] \cdot 3H_2O$
$M_r = 937.79$
Triclinic, $P\overline{1}$
$a = 7.2751$ (10) Å
$b = 10.4797$ (14) Å
$c = 12.7575$ (17) Å
$\alpha = 97.482$ (2)°
$\beta = 92.284$ (2)°
$\gamma = 101.475$ (2)°
$V = 942.9$ (2) Å^3
$Z = 1$
$D_x = 1.652$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1209 reflections
$\theta = 2.9$–23.3°
$\mu = 1.21$ mm^{-1}
$T = 293$ (2) K
Prism, blue
0.25 × 0.16 × 0.10 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.752$, $T_{max} = 0.889$
6956 measured reflections
3383 independent reflections
3353 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 25.2°$
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.084$
$wR(F^2) = 0.172$
$S = 1.34$
3383 reflections
296 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0361P)^2 + 4.7073P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.91$ e Å^{-3}
$\Delta\rho_{min} = -1.16$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O3^{i}	1.942 (4)	Cu1—N1	2.013 (5)
Cu1—O6	1.974 (4)	Cu1—O1	2.274 (5)
Cu1—N2	1.992 (5)		
O3^{i}—Cu1—O6	94.36 (18)	N2—Cu1—N1	82.1 (2)
O3^{i}—Cu1—N2	169.7 (2)	O3^{i}—Cu1—O1	94.25 (19)
O6—Cu1—N2	92.6 (2)	O6—Cu1—O1	90.84 (18)
O3^{i}—Cu1—N1	89.20 (19)	N2—Cu1—O1	93.33 (19)
O6—Cu1—N1	164.33 (19)	N1—Cu1—O1	104.13 (18)

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

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

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
O5—H5$\cdots$O7^{ii}	0.82	1.88	2.673 (8)	164
O6—H6*B*$\cdots$O2^{iii}	0.82	1.85	2.645 (6)	164
O6—H6*C*$\cdots$O4^{i}	0.82	1.82	2.630 (6)	168
O6—H6*C*$\cdots$O3^{i}	0.82	2.46	2.872 (6)	112
O8—H8*B*$\cdots$O2^{iv}	0.80 (3)	2.35 (7)	3.084 (11)	155 (14)
O8—H8*C*$\cdots$O1	0.79 (3)	2.12 (4)	2.896 (10)	168 (15)
O7—H7*B*$\cdots$O2^{v}	0.81 (3)	2.03 (3)	2.820 (8)	167 (8)
O7—H7*C*$\cdots$O4^{vi}	0.81 (3)	2.06 (3)	2.867 (8)	171 (7)

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

H atoms attached to the C atoms were included in the refinement in calculated positions in the riding-model approximation [C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$]. The water H atoms were located in a difference Fourier map and refined with distance restraints [O—H = 0.82 (3) Å, H$\cdots$H = 1.39 (1) Å] and with $U_{iso}(H) = 1.5U_{eq}(O)$. The

Acta Cryst. (2004). E**60**, m1498–m1500 Xiao *et al.* • $[Cu_2(C_8H_4O_5)_2(C_{12}H_8N_2)_2(H_2O)_2] \cdot 3H_2O$ **m1499**

maximum positive electron-density peak is located 0.97 Å from atom O3.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL* (Bruker, 2002).

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