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Diaqua-κ^2O-tris(1,10-phenanthroline)-$1\kappa^2N,N';2\kappa^4N,N'$-$\mu$-5-sulfonatoisophthalato-$1:2\kappa^4O^1,O^{1'}:O^3,O^{3'}$-dicopper(II) perchlorate trihydrate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.009 Å
R factor = 0.065
wR factor = 0.158
Data-to-parameter ratio = 12.1

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

In the title compound, $[Cu_2(C_8H_3O_7S)(C_{12}H_8N_2)_3(H_2O)_2]$-$ClO_4\cdot3H_2O$, the 5-sulfonatoisophthalate trianion functions as a bridge between two Cu atoms to form an asymmetric dinuclear cation. One Cu atom is surrounded by four O atoms, two from a 5-sulfonatoisophthalate trianion and two from water ligands, and by two N atoms from a 1,10-phenanthroline ligand. The second Cu atom is bound to two O atoms from a 5-sulfonatoisophthalate trianion and four N atoms from two 1,10-phenanthroline ligands. Both copper centers have a distorted octahedral geometry.

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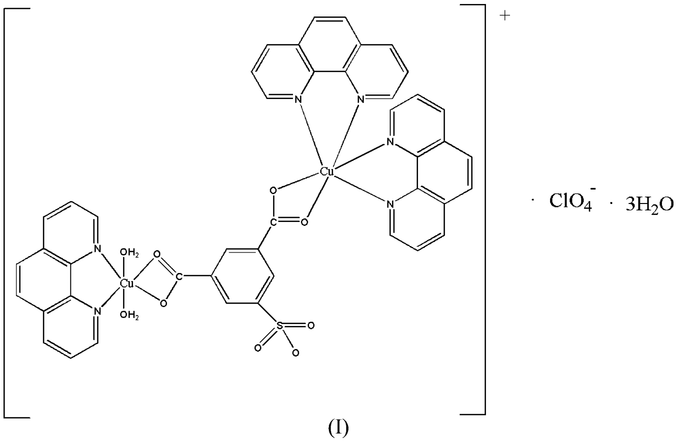
Comment

We obtained a series of dinuclear complexes and one-dimensional zigzag chain coordination polymers whilst studying the structures of Cu^{2+}/phen/H_2tp (phen is 1,10-phenanthroline and H_2tp is terephthalic acid) complexes (Cano *et al.*, 1997; Chen *et al.*, 2004; Sun *et al.*, 2000, 2001; Xiao *et al.*, 2004; Zhu *et al.*, 2004) and Cu^{2+}/2,2′-bipy/H_2tp (2,2′-bipy is 2,2′-bipyridine) (Xiao & Zhu, 2003; Li & Xiao, 2004). These complexes display a diversity of structures. Even small changes, such as the use of different solvents, temperature, synthesis conditions or H-atom acceptors, have produced different products. All of these factors encouraged us to research these systems in more detail. Here, 5-sulfoisophthalatic acid was substituted for H_2tp and the title compound $[Cu_2(C_8H_3O_7S)(phen)_3(H_2O)_2]ClO_4\cdot3H_2O$, (I), was obtained.



(I)

Compound (I) consists of $[Cu_2(C_8H_3O_7S)(C_{12}H_8N_2)_3$-$(H_2O)_2]^+$ cations, perchlorate anions, and solvent water molecules. Each Cu atom is six-coordinate and the geometry is distorted octahedral. Atom Cu1 is surrounded by a carboxylate group from a 5-sulfonatoisophthalate trianion, two water ligands and a 1,10-phenanthroline ligand. Atom

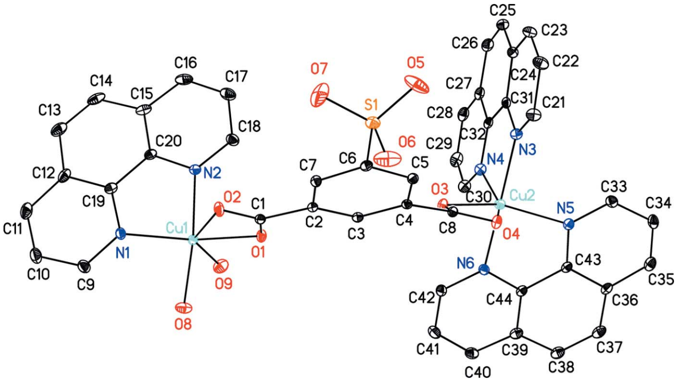


Figure 1
The structure of (I) with the atom numbering, showing displacement ellipsoids at the 50% probability level. H atoms, water molecules and the perchlorate anion have been omitted for clarity.

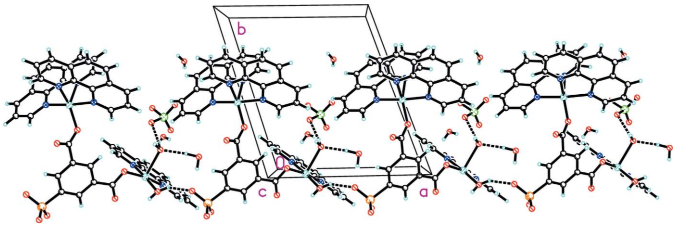


Figure 2
Perspective view of the packing of (I), with hydrogen bonds shown as dotted lines.

Cu2 is surrounded by a carboxylate from a 5-sulfonatoisophthalate trianion and two 1,10-phenanthroline ligands (Fig. 1 and Table 1). The 5-sulfonatoisophthalate trianion functions as a bridge between the two Cu atoms in a tetradentate coordination mode, and the 1,10-phenanthroline group acts as a chelating ligand. The dinuclear cations, perchlorate anions and solvent water molecules interact through O—H$\cdots$O interactions (Table 2), generating columnar stacks parallel to the *a* axis (Fig. 2).

Experimental

An aqueous solution (10 ml) of copper chloride dihydrate (0.3 mmol) was added dropwise to a solution (10 ml) of dimethylformamide containing 1,10-phenanthroline (0.3 mmol), 5-sulfoisophthalic acid (0.3 mmol) and 2,2′-dithiosalicylic acid (0.3 mmol) at room temperature. The reaction mixture was filtered and the filtrate was left to stand for about four weeks until blue single crystals were obtained.

Crystal data

$[Cu_2(C_8H_3O_7S)(C_{12}H_8N_2)_3(H_2O)_2]$-$ClO_4 \cdot 3H_2O$
$M_r = 1100.39$
Triclinic, $P\overline{1}$
$a = 11.7224$ (9) Å
$b = 15.1064$ (12) Å
$c = 15.2183$ (11) Å
$\alpha = 117.583$ (10)°
$\beta = 95.397$ (2)°
$\gamma = 103.732$ (10)°
$V = 2254.3$ (3) Å^3
$Z = 2$
$D_x = 1.621$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 7887 reflections
$\theta = 1.6$–25.1°
$\mu = 1.13$ mm^{-1}
$T = 298$ (2) K
Block, blue
0.22 × 0.21 × 0.14 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.78$, $T_{max} = 0.85$
11966 measured reflections
7887 independent reflections
6173 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 25.1°$
$h = -13 \rightarrow 12$
$k = -17 \rightarrow 18$
$l = -18 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.158$
$S = 1.06$
7887 reflections
651 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0735P)^2 + 2.2465P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.021$
$\Delta\rho_{max} = 0.67$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.950 (3)	Cu1—O9	2.309 (4)
Cu1—O8	1.958 (3)	Cu2—O3	1.966 (3)
O1—Cu1—N1	168.72 (16)	N6—Cu2—N5	82.01 (17)
N1—Cu1—N2	82.21 (17)	O3—Cu2—N4	110.68 (14)
O1—Cu1—O9	92.68 (14)	N3—Cu2—N4	79.73 (16)
O3—Cu2—N5	153.08 (15)		

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

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
O16—H16*B*$\cdots$O9	0.95 (4)	1.90 (4)	2.843 (8)	168 (5)
O15—H15*B*$\cdots$O13^{i}	0.76 (4)	2.37 (5)	3.101 (10)	162 (7)
O15—H15*A*$\cdots$O7^{ii}	0.88 (4)	1.99 (4)	2.858 (8)	169 (6)
O14—H14*B*$\cdots$O1^{iii}	0.79 (4)	2.11 (5)	2.831 (5)	151 (6)
O14—H14*A*$\cdots$O6^{iv}	0.84 (4)	1.95 (5)	2.765 (6)	164 (6)
O9—H9*A*$\cdots$O11	0.82	2.04	2.851 (8)	171
O9—H9*B*$\cdots$O14^{v}	0.82	2.01	2.793 (7)	159
O8—H8*A*$\cdots$O4^{vi}	0.82	1.89	2.643 (5)	152
O8—H8*B*$\cdots$O5^{vii}	0.82	1.87	2.603 (5)	148

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

Uncoordinated water H atoms were refined subject to the restraint O—H = 0.82 (6) Å. Other H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of 0.82 (O—H) and 0.93 Å (C—H), with $U_{iso} = 1.2U_{eq}$(parent atom).

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* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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References

Bruker (2002). *SMART* (Version 5.618), *SAINT* (Version 6.02a), *SADABS* (Version 2.03), *SHELXTL* (Version 5.03, including *XP*). Bruker AXS Inc., Madison, Wisconsin, USA.

Cano, J., De Munno, G., Sanz, J. L., Ruiz, R., Faus, J., Lloret, F., Julve, M. & Caneschi, A. (1997). *J. Chem. Soc. Dalton Trans.* pp. 1915–1923.

Chen, F., Li, X.-H., Xiao, H.-P. & Hu, M.-L. (2004). *Acta Cryst.* E**60**, m708–m710.

Li, X.-H. & Xiao, H.-P. (2004). *Acta Cryst.* E**60**, m898–m900.

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

Sun, D.-F., Cao, R., Liang, Y.-C., Hong, M.-C., Su, W.-P. & Weng, J.-B. (2000). *Acta Cryst.* C**56**, e240–e241.

Sun, D.-F., Cao, R., Liang, Y.-C., Shi, Q., Su, W.-P. & Hong, M.-C. (2001). *J. Chem. Soc. Dalton Trans.* pp. 2335–2340.

Xiao, H.-P., Li, X.-H., Ye, M.-D. & Hu, M.-L. (2004). *Acta Cryst.* E**60**, m253–m254.

Xiao, H.-P. & Zhu, L.-G. (2003). *Chin. J. Inorg. Chem.* **19**, 1179–1184.

Zhu, L.-G., Xiao, H.-P. & Lu, J.-Y. (2004). *Inorg. Chem. Commun.* **7**, 94–96.