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Di-μ-hydroxo-bis[aqua(1,10-phenanthroline-κ^2N,N')-copper(II)] terephthalate octahydrate

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The title compound, $[Cu_2(OH)_2(C_{12}H_8N_2)_2(H_2O)_2](C_8H_4O_4)\cdot 8H_2O$, was prepared by the hydrothermal reaction of $CuCl_2$, 1,10-phenanthroline, terephthalic acid and water at 443 K. It consists of a double-hydroxo-bridged dinuclear complex cation [Cu···Cu = 2.911 (3) Å], a terephthalate anion and eight water molecules. Both cation and anion possess inversion symmetry. A network of O—H···O hydrogen bonds stabilizes the structure.

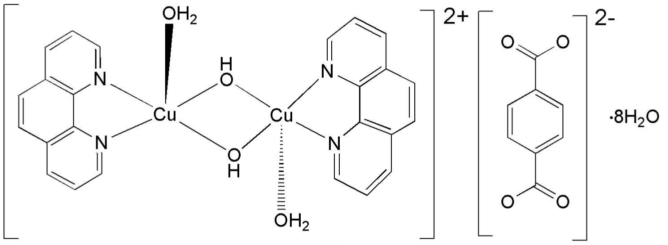
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.019 Å
R factor = 0.098
wR factor = 0.253
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.

Comment

In recent years, the design and synthesis of coordination polymers has attracted much attention, due to their potential application in catalysis, ion exchange and gas adsorption (Eddaoudi *et al.*,2002; Moulton & Zaworotko, 2001). As a multidentate bridging ligand, terephthalic acid (ta^{2-}) has been much used in this field because of its ability to form short metal–metal bridges *via* one carboxylate end group or long bridges *via* the intervening benzene ring (Ma *et al.*, 2003; Zhang *et al.*, 2003; Yuan *et al.*, 2003). Numerous complexes with the ta^{2-} ligand have been reported (Hagrman *et al.*, 1999; Li *et al.*, 1999; Groenman *et al.*, 1999), while new complexes are constantly being synthesized. We report here another such structure, the title compound, (I) (Fig. 1), consisting of an uncoordinated ta^{2-} anion, a $[Cu_2(C_{12}H_8N_2)(OH)_2(H_2O)_2]^{2+}$ dinuclear cation and water molecules of crystallization. Both cation and anion are generated by inversion symmetry from the atoms of the asymmetric unit.



(I)

In the $[Cu_2(C_{12}H_8N_2)(OH)_2(H_2O)_2]^{2+}$ cation of (I), the Cu^{2+} environment is that of a distorted square pyramid, composed of two phen N atoms and two O atoms of bridging hydroxide groups at the corners of the basal square and a fifth weakly coordinated water molecule at the apical position. Because of inversion symmetry, the two apical water molecules are in a trans conformation. This dinuclear cation is similar to others reported previously (Zheng *et al.*, 2000; Lu *et al.*, 2003, 2004). The Cu···Cu distance of 2.911 (3) Å in (I) is longer than that found in $[Cu_2(C_{12}H_8N_2)(OH)_2(H_2O)_2]$

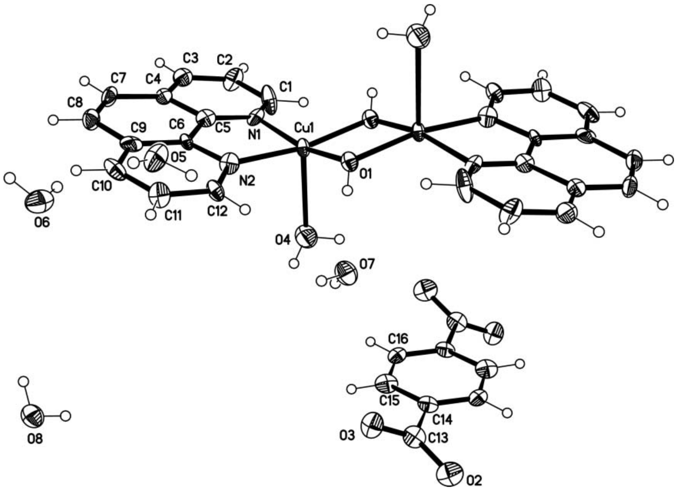


Figure 1
The structure of (I), with displacement ellipsoids drawn at the 30% probability level and H atoms shown as small spheres of arbitrary radii. Unlabelled atoms in the cation and anion are related to labelled atoms by the symmetry operators $(1 - x, 2 - y, 2 - z)$ and $(-x, 1 - y, 2 - z)$, respectively.

[2.902 (1) Å; Zheng *et al.*, 2000; Lu *et al.*, 2004] and shorter than that found [2.933 (1) Å] by Lu *et al.* (2003).

The hydrogen-bonding geometry in (I) is listed in Table 2 and illustrated in Fig. 2. These O—H···O hydrogen bonds link $[Cu_2(C_{12}H_8N_2)(OH)_2(H_2O)_2]^{2+}$ cations and ta^{2-} anions, as well as uncoordinated water molecules, resulting in a three-dimensional network.

Experimental

All chemicals were of reagent grade and commercially available from the Beijing Chemical Reagents Company of China, and were used without further purification. A mixture of $CuCl_2 \cdot 2H_2O$ (0.0851 g, 0.5 mmol), 1,10-phenanthroline (0.0992 g, 0.5 mmol) and H_2O (15 ml) was stirred for 35 min at 323 K. The mixture and terephthalic acid (0.0801 g, 0.5 mmol) were then sealed in a 20 ml Teflon-lined autoclave and heated at 443 K for 86 h. After cooling, blue crystals of (I) were recovered.

Crystal data

$[Cu_2(OH)_2(C_{12}H_8N_2)_2(H_2O)_2]$-$(C_8H_4O_4) \cdot 8H_2O$
$M_r = 865.78$
Triclinic, $P\overline{1}$
$a = 9.295$ (4) Å
$b = 10.639$ (5) Å
$c = 11.258$ (5) Å
$\alpha = 114.603$ (8)°
$\beta = 112.540$ (8)°
$\gamma = 95.065$ (8)°
$V = 893.2$ (7) Å^3
$Z = 1$
$D_x = 1.609$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 279 reflections
$\theta = 2.2$–17.9°
$\mu = 1.27$ mm^{-1}
$T = 298$ (2) K
Plate, blue
0.20 × 0.20 × 0.01 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
$T_{min} = 0.785$, $T_{max} = 0.987$
4159 measured reflections
2960 independent reflections
1399 reflections with $I > 2\sigma(I)$
$R_{int} = 0.107$
$\theta_{max} = 25.0°$
$h = -11 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -8 \rightarrow 13$

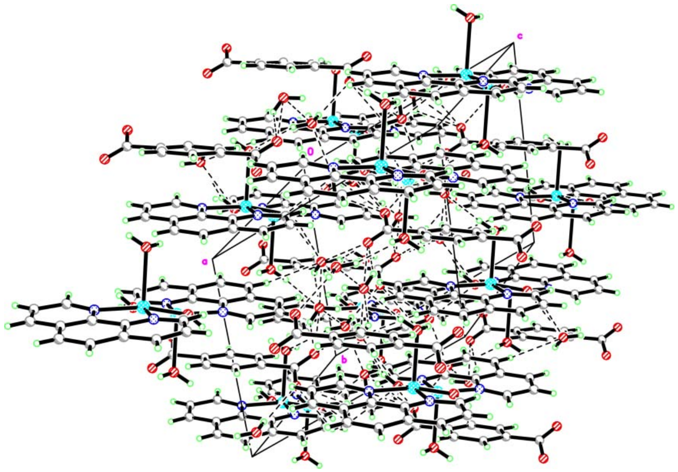


Figure 2
The packing of (I), with dashed lines indicating hydrogen bonds.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.098$
$wR(F^2) = 0.253$
$S = 0.84$
2960 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1026P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.59$ e Å^{-3}
$\Delta\rho_{min} = -0.83$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.925 (7)	Cu1—N1	2.024 (9)
Cu1—O1^i	1.930 (7)	Cu1—O4	2.427 (9)
Cu1—N2	2.023 (10)		
O1—Cu1—O1^i	81.9 (3)	N2—Cu1—N1	84.0 (4)
O1—Cu1—N2	94.7 (4)	O1—Cu1—O4	99.4 (3)
O1^i—Cu1—N2	167.6 (3)	O1^i—Cu1—O4	99.3 (3)
O1—Cu1—N1	166.7 (3)	N2—Cu1—O4	93.0 (3)
O1^i—Cu1—N1	96.5 (4)	N1—Cu1—O4	93.9 (3)

Symmetry code: (i) $-x + 1, -y + 2, -z + 2$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O8—H82···O2^{ii}	0.84	2.34	2.783 (13)	113
O7—H72···O6^{iii}	0.82	2.08	2.822 (12)	149
O7—H71···O3^{iv}	0.81	2.24	2.713 (12)	118
O6—H61···O3^{iii}	0.82	2.17	2.828 (12)	138
O5—H52···O2^{iv}	0.82	2.10	2.802 (12)	144
O5—H51···O4^{v}	0.82	2.30	3.088 (13)	161
O4—H41···O6^{iii}	0.83	2.19	2.983 (13)	160
O1—H13···O7	0.83	1.96	2.756 (11)	159

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

H atoms attached to C atoms were placed in geometrically idealized positions (C—H = 0.93 Å) and refined as riding [$U_{iso}(H) = 1.2U_{eq}(C)$]. H atoms attached to O were located in a difference Fourier map, relocated in idealized positions (O—H = 0.80–0.84 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(O)$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve

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structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL/PC*.

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