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catena-Poly[[[aqua(pyrazino[2,3-*f*][1,10]phenanthroline-κ^2N,N')copper(II)]-μ-isophthalato-$\kappa^2O:O'$] *N*,*N*-dimethylformamide solvate monohydrate]

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Key indicators

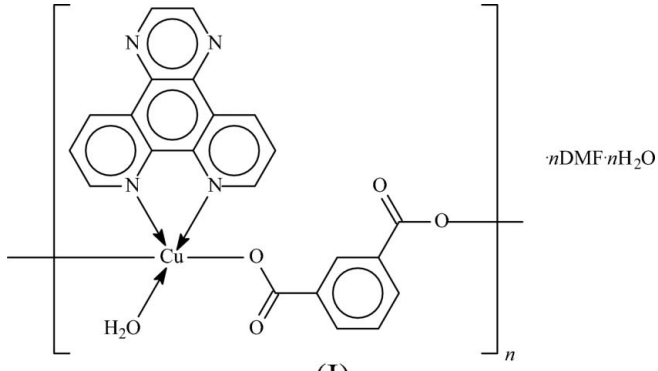
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.038
wR factor = 0.107
Data-to-parameter ratio = 13.1

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

In the title compound, $\{[Cu(C_8H_4O_4)(C_{14}H_8N_4)(H_2O)]\cdot C_3H_7NO\cdot H_2O\}_n$, the Cu^{II} atom binds a water molecule and is chelated by the pyrazinophenanthroline *N*-heterocycle. It is also linked covalently to an isophthalate dianion that lies on a special position of site symmetry 2. The dianion serves as a spacer that connects adjacent square-pyramidal metal atoms into a zigzag chain propagating along the *c* axis of the orthorhombic unit cell. The chain motif is consolidated into a layer structure by hydrogen bonds involving both the coordinated and uncoordinated water molecules. The copper atom and the coordinated water molecule lie on a mirror plane, while the *N*-heterocycle, the disordered uncoordinated water molecule and the disordered dimethylformamide solvent molecule lie across a mirror plane.

Comment

Che *et al.* (2006) describe the chain structure of aqua(pyrazino[2,3-*f*][1,10]phenanthroline)(terephthalato)copper dimethylformamide (DMF) hydrate. The principal feature is the helical chain motif that results from μ_2-bridging by the terephthalate unit. The use of isophthalic acid in place of terephthalic acid in a similar synthesis affords an analogous chain compound, which also crystallizes with dimethylformamide and water as solvent molecules, (I) (Fig. 1). The metal atom shows a square-pyramidal coordination. The copper atom and the coordinated water molecule lie on a mirror plane, while the *N*-heterocycle, the disordered uncoordinated water molecule and the disordered dimethylformamide solvent molecule lie across a mirror plane.



(I)

The isophthalate dianion connects adjacent monomeric units into a zigzag chain that propagates along the *c* axis of the unit cell (Fig. 2). The structure is consolidated by hydrogen bonds (Table 2) into layers.

Experimental

A methanol solution (4 ml) of pyrazino[2,3-*f*][1,10]phenanthroline (Che *et al.*, 2006) (59 mg, 0.25 mmol) was mixed with an aqueous (4 ml) solution of copper chloride dihydrate (43 mg, 0.25 mmol). A DMF solution (18 ml) of isophthalic acid (42 mg, 0.25 mmol) was added and the mixture heated at 348 K for 5 h. The solution was filtered; blue crystals separated from the solution after several days in 60% yield.

Crystal data

$[Cu(C_8H_4O_4)(C_{14}H_8N_4)(H_2O)]\cdot C_3H_7NO\cdot H_2O$
$M_r = 569.02$
Orthorhombic, *Pbcm*
$a = 7.237$ (2) Å
$b = 23.877$ (6) Å
$c = 13.848$ (8) Å
$V = 2393$ (2) Å^3
$Z = 4$
$D_x = 1.579$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.97$ mm^{-1}
$T = 293$ (2) K
Block, blue
0.20 × 0.19 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.552$, $T_{max} = 0.845$
20300 measured reflections
2810 independent reflections
2138 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.107$
$S = 1.05$
2810 reflections
215 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.5178P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.931 (2)	Cu1—O1*w*	2.337 (2)
Cu1—N1	2.022 (2)		
O1—Cu1—O1^{i}	91.4 (1)	O1—Cu1—O1*w*	91.0 (1)
O1—Cu1—N1	174.8 (1)	O1*w*—Cu1—N1	89.8 (1)
O1—Cu1—N1^{i}	93.7 (1)	N1—Cu1—N1^{i}	81.1 (1)

Symmetry code: (i) $x, y, -z+\frac{3}{2}$.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*w*—H1*w*1···O3	0.85 (1)	2.00 (1)	2.725 (4)	142 (1)
O1*w*—H1*w*2···O2*w*^{ii}	0.85 (1)	1.93 (1)	2.773 (5)	177 (2)
O2*w*—H2*w*1···O2	0.85 (1)	1.82 (4)	2.67 (3)	175 (6)

Symmetry code: (ii) $x-1, y, z$.

The pyrazino-phenanthroline, DMF and uncoordinated water are disordered over a mirror plane. For the $C_{14}H_8N_4$ ligand, the N—C distances were restrained to 1.35 (1) Å and the C—C distances to 1.39 (1) Å; the ligand was restrained to near-planarity. For the DMF molecule, the following distance restraints were imposed: C13—O3 = 1.25 (1) Å, C13—N3 = 1.35 (1) Å, C14—N3 = C15—N3 = 1.45 (1) Å, C13···C14 = C13···C15 = 2.43 (2) Å, C14···C15 = 2.51 (2) Å and O3···N3 = 2.25 (2) Å. The molecule was also restrained to be approximately planar, and the atoms were restrained to approximate isotropic behaviour. The uncoordinated water molecule was allowed to refine off the mirror plane. The carbon-bound H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and were included in the refinement in the riding-model approximation, with U_{iso}(H) values set at 1.2 or 1.5 times U_{eq}(C). The methyl groups were rotated to fit the electron density. The water H atoms were located in a difference Fourier map, and were refined with distance restraints of O—H = 0.85 (1) Å and H···H = 1.39 (1) Å, with U_{iso}(H) = 1.2U_{eq}(O).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: publCIF (Westrip, 2006).

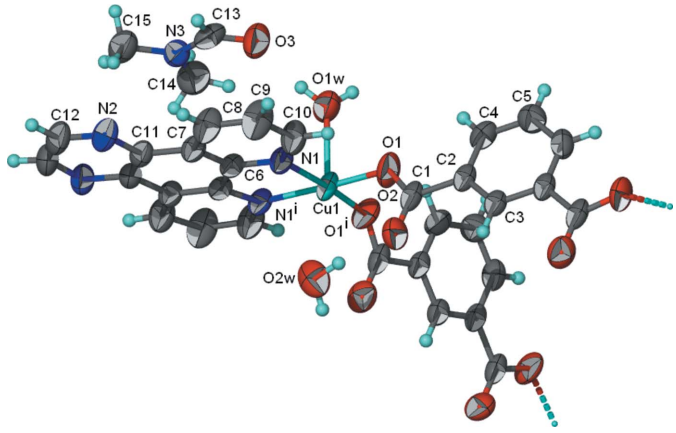


Figure 1
A portion of the chain structure of (I). Displacement ellipsoids are drawn at the 70% probability level and H atoms are drawn as spheres of arbitrary radii. [Symmetry code (i): $x, y, \frac{3}{2}-z$.]

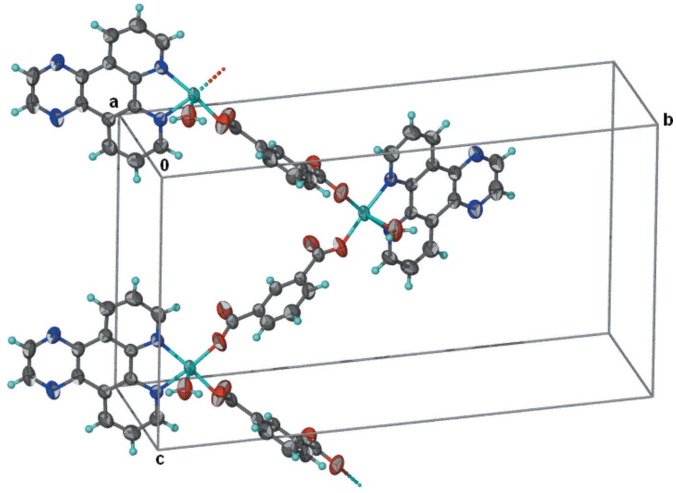


Figure 2
The chain structure; the DMF and uncoordinated water molecules are not shown. Displacement ellipsoids are drawn at the 50% probability level.

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