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A monoclinic polymorph of *catena*-poly[[[diaqua(pyridine-2-carboxamide-κ^2N^1,O)copper(II)]-μ-sulfato-$\kappa^2O:O'$] monohydrate]

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Key indicators

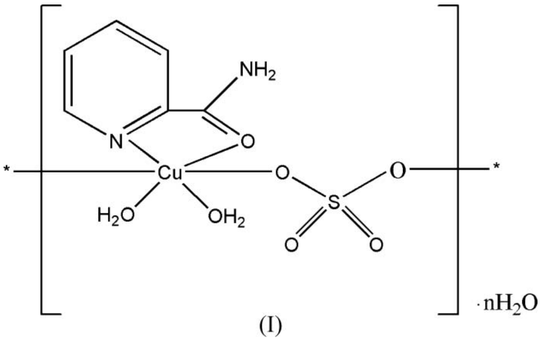
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.022
wR factor = 0.055
Data-to-parameter ratio = 13.1

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

The title crystal structure, $\{[Cu(SO_4)(C_6H_6N_2O)(H_2O)_2]\cdot H_2O\}_6$, is a monoclinic polymorph of the previously reported triclinic structure [Sieroń & Bukowska-Strzyżewska (1999). *Acta Cryst.* C**55**, 491–494] in which sulfate ligands bridge six-coordinate Cu atoms, generating an extended one-dimensional chain structure along [010]. Neighboring chains are connected by N—H···O(sulfate) hydrogen bonds into ladders, which are further connected into two-dimensional layers perpendicular to the [100] direction by N—H···O(water) and O(water)—H···O hydrogen bonds. These layers are connected by O—H···O hydrogen bonds into a three-dimensional network.

Comment

Recently, the rational design and synthesis of polymeric metal-organic coordination networks has been an active research area because of its application in many fields (Moulton & Zaworotko, 2001; MacGillivray *et al.*, 1998). It is well known that the utilization of multidentate *O*- or *N*-donor ligands is an effective strategy in the construction of these types of complex networks (Zheng *et al.*, 2004; Kamiyama *et al.*, 2000). As part of our recent research, we selected 2,4,6-tri-2-pyridyl-1,3,5-triazine (tptz) in the hope of constructing a coordination polymer with potential applications, but instead we obtained the title compound, (I), from the reaction of tptz and $CuSO_4\cdot 5H_2O$. This was most likely the result of tptz undergoing hydrolysis, yielding pyridine-2-carboxamide (Cantarero *et al.*, 1988; Lerner & Lippard, 1977).



(I)

Compound (I) is a monoclinic polymorph of the previously reported triclinic structure (Sieroń & Bukowska-Strzyżewska, 1999). Part of the one-dimensional chain structure of (I) is shown in Fig.1. The unique Cu atom is coordinated in a slightly distorted octahedral environment by N and O atoms from a chelating pyridine-2-carboxamide ligand, two O atoms from two bridging sulfate ligands and two O atoms from two water

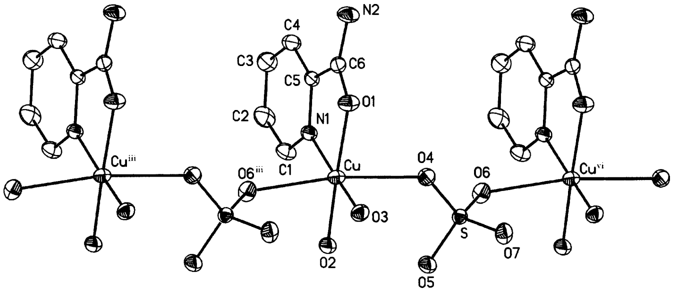


Figure 1

Part of the one-dimensional chain structure of (I). Displacement ellipsoids are drawn at the 45% probability level. H atoms have been omitted. [Symmetry codes: (iii) $x, y + 1, z$; (vi) $x, y - 1, z$.]

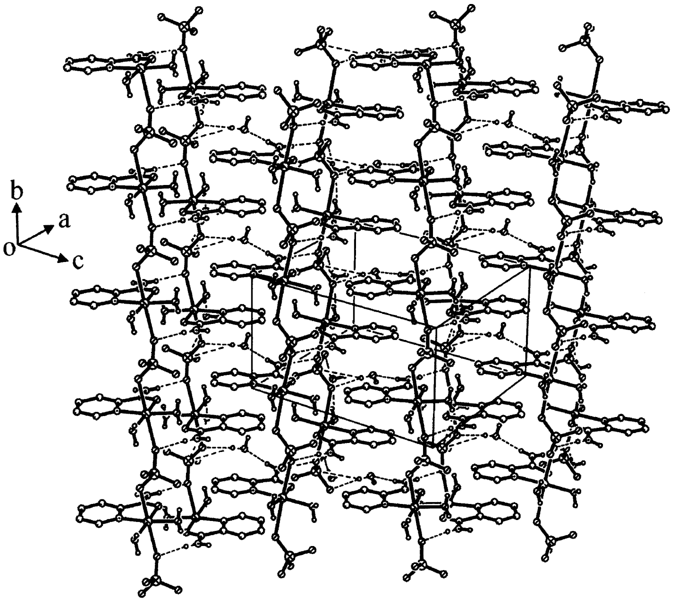


Figure 2

A two-dimensional supramolecular layer of (I). Hydrogen bonds are indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted.

ligands. The resulting one-dimensional chain structure propagates along [010]. Selected bond distances and bond angles are listed in Table 1.

There are strong intrachain hydrogen bonds between atom O2 of the coordinated water molecule and atom O5 of a sulfate ligand (see Table 2 for hydrogen-bonding geometry). Neighboring chains are connected by interchain N2—H$\cdots$O4^{i} (symmetry codes as in Table 2) hydrogen bonds into ladders, which are further assembled into two-dimensional layers perpendicular to the [100] direction by N2—H$\cdots$O8^{ii}and O8—H$\cdots$O6^{v} hydrogen bonds (Fig. 2). The two-dimensional layers are stabilized by the remaining hydrogen bonds between O atoms of the sulfate ligand and the uncoordinated water molecules, forming a three-dimensional network (Fig. 3).

Experimental

Addition of 2,4,6-tri-2-pyridyl-1,3,5-triazine (tptz) (1.25 g, 4.0 mmol) to a stirred aqueous solution (30 ml) of $CuSO_4{\cdot}5H_2O$ (2.0 g, 8.0 mmol) yielded a turbid blue solution. This was refluxed for 30 min at 363 K followed by filtration after cooling. The resulting blue filtrate was maintained at room temperature and slow evaporation afforded blue crystals one month later (yield: 40% based on the initial $CuSO_4{\cdot}5H_2O$ input).

Crystal data

$[Cu(SO_4)(C_6H_6N_2O)(H_2O)_2]{\cdot}H_2O$
$M_r = 335.78$
Monoclinic, $P2_1/n$
$a = 12.635$ (3) Å
$b = 6.8276$ (14) Å
$c = 13.414$ (3) Å
$\beta = 101.62$ (3)°
$V = 1133.5$ (5) Å^3
$Z = 4$
$D_x = 1.968$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 2.15$ mm^{-1}
$T = 298$ (2) K
Block, blue
0.45 × 0.27 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.504$, $T_{max} = 0.773$
10304 measured reflections
2559 independent reflections
2284 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.055$
$S = 1.14$
2559 reflections
195 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0211P)^2 + 0.8198P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

Cu—O3	1.9492 (15)	Cu—O1	2.0045 (13)
Cu—O2	1.9787 (14)	Cu—O4	2.3788 (14)
Cu—N1	1.9806 (16)	Cu—O6^{i}	2.4173 (14)
O3—Cu—O2	93.96 (7)	N1—Cu—O4	96.20 (6)
O3—Cu—N1	172.45 (6)	O1—Cu—O4	88.12 (5)
O2—Cu—N1	92.68 (7)	O3—Cu—O6^{i}	87.81 (6)
O3—Cu—O1	92.33 (6)	O2—Cu—O6^{i}	85.57 (6)
O2—Cu—O1	172.17 (6)	N1—Cu—O6^{i}	89.13 (5)
N1—Cu—O1	81.36 (6)	O1—Cu—O6^{i}	99.34 (5)
O3—Cu—O4	87.68 (6)	O4—Cu—O6^{i}	171.43 (5)
O2—Cu—O4	87.48 (6)		

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

Table 2

Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H4$B\cdots$O4^{ii}	0.80 (3)	2.11 (3)	2.900 (3)	169 (3)
N2—H4$A\cdots$O8^{iii}	0.86 (3)	2.20 (3)	3.056 (3)	170 (3)
O2—H2$B\cdots$O8^{i}	0.78 (3)	2.03 (3)	2.792 (3)	167 (3)
O2—H2$A\cdots$O5	0.79 (3)	1.90 (3)	2.668 (3)	162 (3)
O3—H3$B\cdots$O7^{iv}	0.74 (3)	2.07 (3)	2.796 (3)	166 (3)
O3—H3$A\cdots$O7^{i}	0.82 (3)	1.84 (3)	2.649 (3)	169 (3)
O8—H8$A\cdots$O5	0.74 (3)	2.10 (3)	2.828 (3)	170 (3)
O8—H8$B\cdots$O6^{v}	0.78 (3)	2.13 (3)	2.897 (3)	169 (3)

Symmetry codes: (i) $x, y + 1, z$; (ii) $-x + 1, -y, -z + 2$; (iii) $x - \frac{1}{2}, -y - \frac{1}{2}, z + \frac{1}{2}$; (iv) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$; (v) $-x + 2, -y - 1, -z + 2$.

H atoms bonded to C atoms were placed in geometrically calulated positons (C—H = 0.93 Å) and refined using a riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$. H atoms bonded to O and N atoms were found in difference Fourier maps and refined freely.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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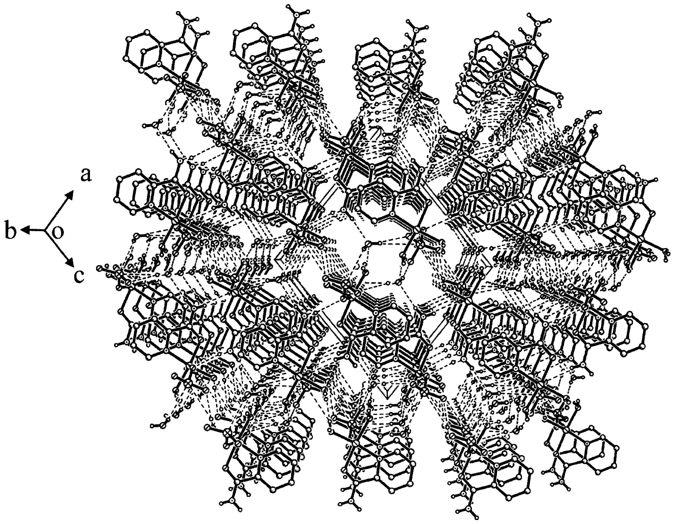

Figure 3
The packing of (I). Hydrogen bonds are indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted.

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