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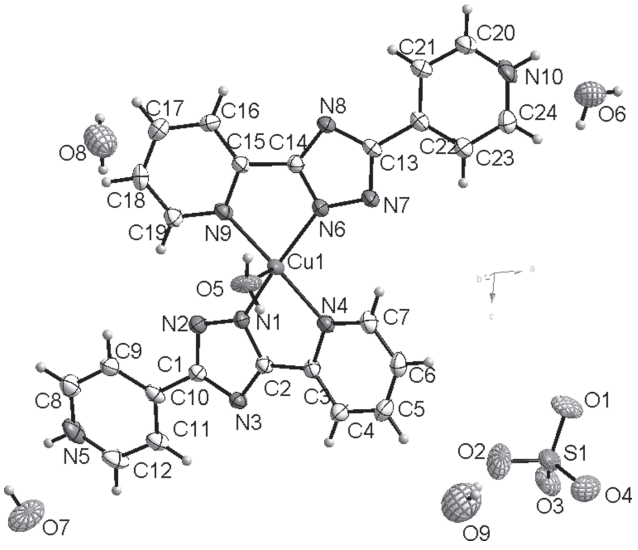
Crystal structure of aqua-bis(3-(2-pyridyl)-5-(4-pyridyl)-1*H*-1,2,4-triazole-κ²*N*,*N*′)copper(II) sulfate tetrahydrate, $C_{24}H_{28}CuN_{10}O_9S$



https://doi.org/10.1515/ncrs-2017-0224
Received October 29, 2017; accepted January 17, 2018; available online February 9, 2018

Abstract

$C_{24}H_{28}CuN_{10}O_9S$, monoclinic, $P2_1/c$ (no. 14), $a = 16.3499(16)$ Å, $b = 14.0411(13)$ Å, $c = 12.7069(13)$ Å, $\beta = 104.019(2)°$, $V = 2830.2(5)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0472$, $wR_{ref}(F^2) = 0.1165$, $T = 293(2)$ K.

CCDC no.: 694447

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The ligand, 3-(2-pyridyl)-5-(4-pyridyl)-1*H*-1,2,4-triazole (Hbpt24), was purchased from Alfa Aesar company. Copper sulfate pentahydrate (0.0125 g, 0.05 mmol), Hbpt24 (0.0223 g, 0.1 mmol) and water (20 mL) were placed in

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Table 1: Data collection and handling.

Crystal:	Needle, blue
Size:	0.38 × 0.31 × 0.24 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.92 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ_{max}, completeness:	25.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14622, 5044, 0.060
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3341
$N(param)_{refined}$:	406
Programs:	Bruker programs [1], SHELX [2, 3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.51097(3)	0.65030(3)	0.24832(4)	0.02603(16)
N1	0.45121(19)	0.6222(2)	0.3611(2)	0.0250(8)
N2	0.37061(19)	0.6041(2)	0.3667(3)	0.0290(8)
N3	0.45482(19)	0.6115(2)	0.5376(2)	0.0233(7)
N4	0.60923(19)	0.6566(2)	0.3798(2)	0.0251(7)
N5	0.1567(2)	0.5546(2)	0.5836(3)	0.0403(10)
H5	0.111509	0.546036	0.605777	0.048*
N6	0.57098(19)	0.6208(2)	0.1347(3)	0.0267(8)
N7	0.65134(19)	0.6017(2)	0.1273(2)	0.0272(8)
N8	0.56440(19)	0.6067(2)	−0.0419(2)	0.0244(8)
N9	0.41200(19)	0.6536(2)	0.1176(2)	0.0254(7)
N10	0.8518(2)	0.5342(2)	−0.1085(3)	0.0370(9)
H10	0.894351	0.522268	−0.135037	0.044*
O1	1.00940(19)	0.5410(2)	0.6277(2)	0.0579(10)
O2	0.9294(2)	0.6454(2)	0.7131(3)	0.0633(10)
O3	0.97443(18)	0.4952(2)	0.7937(2)	0.0450(8)
O4	1.07674(19)	0.6182(2)	0.7929(2)	0.0522(9)
O5	0.50831(18)	0.81163(19)	0.2479(2)	0.0417(8)
H5C	0.513537	0.835498	0.188363	0.050*
H5D	0.543107	0.836688	0.301383	0.050*
O6	0.9443(2)	0.3424(3)	−0.0062(3)	0.0713(11)
H6C	0.936345	0.356596	0.055561	0.086*
H6D	0.978305	0.295716	0.001651	0.086*
O7	0.1520(2)	0.7826(3)	0.7137(3)	0.0732(11)
H7C	0.128798	0.734764	0.735493	0.088*
H7D	0.122268	0.799054	0.651803	0.088*
O8	0.0536(2)	0.1938(3)	0.0295(3)	0.0845(13)
H8C	0.035078	0.146258	−0.010667	0.101*
H8D	0.057908	0.178038	0.095223	0.101*
O9	0.9256(3)	0.8391(3)	0.7572(3)	0.0921(13)
H9C	0.934332	0.782739	0.738040	0.111*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H9D	0.959562	0.876579	0.736740	0.111*
S1	0.99755(7)	0.57652(8)	0.73250(9)	0.0376(3)
C1	0.3755(2)	0.5987(3)	0.4728(3)	0.0229(9)
C2	0.4990(2)	0.6261(3)	0.4628(3)	0.0232(9)
C3	0.5888(2)	0.6462(3)	0.4767(3)	0.0227(8)
C4	0.6470(2)	0.6564(3)	0.5735(3)	0.0309(10)
H4	0.631076	0.649918	0.638569	0.037*
C5	0.7299(3)	0.6765(3)	0.5736(4)	0.0362(11)
H5A	0.770358	0.682925	0.638642	0.043*
C6	0.7517(2)	0.6869(3)	0.4761(4)	0.0344(10)
H6	0.806975	0.700471	0.474293	0.041*
C7	0.6900(2)	0.6767(3)	0.3810(3)	0.0315(10)
H7	0.704785	0.684085	0.315217	0.038*
C8	0.1499(3)	0.5778(3)	0.4802(4)	0.0371(11)
H8	0.096842	0.584058	0.433226	0.045*
C9	0.2206(2)	0.5924(3)	0.4429(3)	0.0321(10)
H9	0.215693	0.608972	0.370781	0.038*
C10	0.2998(2)	0.5823(3)	0.5131(3)	0.0252(9)
C11	0.3043(3)	0.5578(3)	0.6203(3)	0.0323(10)
H11	0.356257	0.550535	0.669400	0.039*
C12	0.2303(3)	0.5444(3)	0.6531(4)	0.0409(12)
H12	0.232588	0.528012	0.724656	0.049*
C13	0.6441(2)	0.5942(3)	0.0207(3)	0.0241(9)
C14	0.5222(2)	0.6228(3)	0.0343(3)	0.0226(9)
C15	0.4319(2)	0.6429(3)	0.0211(3)	0.0233(9)
C16	0.3738(2)	0.6514(3)	−0.0760(3)	0.0319(10)
H16	0.389587	0.643967	−0.141054	0.038*
C17	0.2909(3)	0.6713(3)	−0.0754(3)	0.0369(11)
H17	0.250075	0.677604	−0.140248	0.044*
C18	0.2697(2)	0.6816(3)	0.0224(3)	0.0338(11)
H18	0.214256	0.693922	0.024521	0.041*
C19	0.3312(2)	0.6736(3)	0.1164(3)	0.0292(10)
H19	0.316627	0.682193	0.182098	0.035*
C20	0.7746(3)	0.5336(3)	−0.1741(3)	0.0372(11)
H20	0.767615	0.520243	−0.247376	0.045*
C21	0.7059(3)	0.5525(3)	−0.1345(3)	0.0322(10)
H21	0.652171	0.551795	−0.180494	0.039*
C22	0.7170(2)	0.5730(3)	−0.0243(3)	0.0241(9)
C23	0.7977(2)	0.5729(3)	0.0414(3)	0.0337(10)
H23	0.806835	0.586280	0.114987	0.040*
C24	0.8647(3)	0.5529(3)	−0.0030(4)	0.0385(11)
H24	0.919203	0.552409	0.040883	0.046*

20.0 mL Teflon-lined stainless steel autoclave. It was heated at 383 K for 2 days. After cooling to room temperature, large blue, rod crystals of the title complex were obtained.

Experimental details

The nitrogen and oxygen bound hydrogen atoms were located from the difference Fourier syntheses and refined with restrained distances usind the DFIX comand (O—H: 0.85 Å; N—H: 0.86 Å), and were included with 1.2 U_{iso}(C, N). The carbon bonded hydrogen atoms were placed on calculated positions using a riding model (AFIX 43 or AFIX 137 option of the SHELX program [2, 3]. A translational pseudosymmetry was detected ($b' = b/2$) with a fit of 92% [4].

Discussion

Metal organic complexes are known for their wealth of intriguing architectures, topologies and their potential applications in gas storage, molecular recognition, magnetism, electric conductivity, heterogeneous catalysis, fluorescence probe and much more. My group has contributed to this large field of research some findings in the last years [5–8]. During our previous work, a number of coordination compounds of 3-(2-pyridyl)-5-(4-pyridyl)-1*H*-1,2,4-triazole (bpt24) ligand with cobalt and nickel were prepared and structurally characterized [9, 10]. The Ni/bpt24 complexes showed polytropic structures, and a series of Co/bpt24 complexes present crystal transformations from each other.

The asymmetric structure unit of the title compound contains one Cu(II) ion, two bptH ligands, one coordinated water molecule and a free sulfate radical as well as four free water molecules. Of particular note is that the hydrogen atom had shifted from triazolyl nitrogen to pyridyl nitrogen within the bptH ligand. The central atom Cu(II) is chelated by four N atoms from two bptH ligands and is coordinated by one water O atom (Cu—O5, 2.264(3) Å). The Cu—N bond lengths (1.960(3)−2.264(3) Å) are within the normal range of those in complexes of Cu/N-heterocyclic ligands [11, 12]. The geometry around the Cu(II) atom is best described as a seriously distorted tetragonal pyramid. In addition, the hydrogen bonds formed between the coordinated water molecule and the triazole N atom (O5—H5C···N3, 2.820 Å; O5—H5D···N8, 2.849 Å), link the title complexes into a one dimensional step-like chain. The sulfate ions and water molecules form the following hydrogen bonds: O6—H6C···O4 (2.868 Å), O6—H6D—O8 (2.714 Å), O7—H7C—O4 (2.910 Å), O7—H7D—O6 (2.848 Å), O8—H8C—O1 (2.940 Å), O8—H8D—O9 (2.690 Å) and O9—H9C—O2 (2.781 Å), O9—H9D—O3 (2.898 Å). The intermolecular hydrogen bonds from pridyl N—H N5—H5···O1 (2.608 Å) and N10—H10···O3 (2.663 Å), together with the aforementioned ones link all moieties to a supramolecular three-dimensional framework.

Acknowledgements: We are grateful for financial support from the NSF of China (Grants 21671031, 21171031).

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