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Crystal structure of *catena*-aquatris(1*H*-imidazole-*N*)(μ-sulfato)copper(II), $Cu(H_2O)(C_3H_4N_2)_3(SO_4)$

F. Fu, J.-X. Guo, D.-S. Li*, L. Tang, M.-L. Zhang and J.-W. Wang

Yan'an University, Department of Chemistry and Chemical Engineering, Shaanxi Key Laboratory of Chemical Reaction Engineering, Shaanxi, Yan'an 716000, P. R. China

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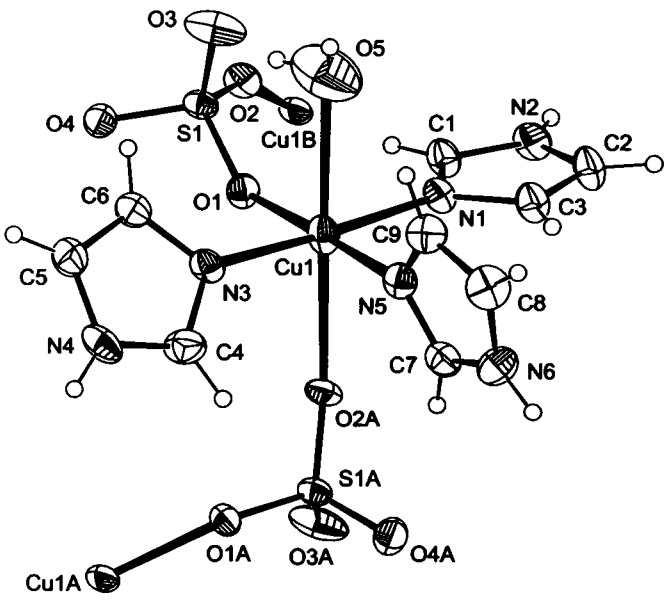

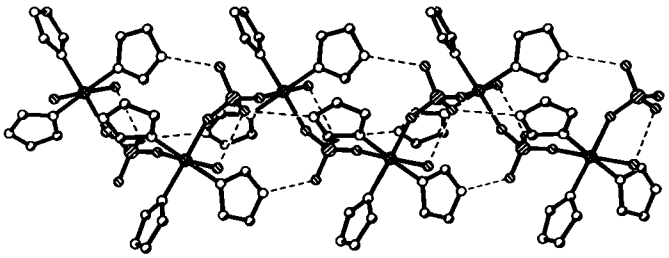

Abstract

$C_9H_{14}CuN_6O_5S$, monoclinic, $P12_1/n1$ (no. 14), a = 11.723(2) Å, b = 8.651(2) Å, c = 13.839(3) Å, β = 91.703(4)°, V = 1402.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.046, $wR_{ref}(F^2)$ = 0.133, T = 296 K.

Source of material

The title complex was synthesized by mixed solution method. A solution of $CuSO_4 \cdot 5H_2O$ (0.125 g, 0.5 mmol) in 10 ml methanol was added to a solution of 1,1′-carbonyl-diimidazole (0.081 g, 0.5 mmol) in 10 ml methanol. The reaction mixture was stirred for 2 h at room temperature and then filtered. The filtrate was diffused in ether. After two weeks, blue block-shaped crystals were obtained.

* Correspondence author (e-mail: llln1832@sina.com)

Discussion

Helical structures have received much attention in coordination chemistry and materials chemistry because helicity is an essential feature of life and also important in advanced materials such as optical devices and asymmetric catalysis [1,2]. Consequently, many single-, double- and higher-order stranded helical complexes have been generated by self-assembly processes [3,4]. Herein, we report a novel 1D helical chain coordination polymer $[Cu(C_3H_4N_2)_3(H_2O)(SO_4)]_n$.

In the crystal structure of title compound (figure, top), the Cu(II) atoms are coordinated by three N atoms from three different imidazole ligands (d(Cu—N1) = 1.998(3) Å, d(Cu—N3) = 1.984(3) Å, d(Cu—N5) = 1.997(4) Å. Imidazole molecules resulted from 1,1′-carbonyl-diimidazole which was easily hydrolyzed in methanol solution. Furthermore, three O atoms from two sulfato groups (d(Cu—O1) = 2.003(3) Å, d(Cu—O2A) = 2.534(4) Å) and one water molecule (d(Cu—O1) = 2.551(5) Å) furnish a distorted octahedral environment. The *cisoid* and *transoid* bond angles around the Cu atoms fall in the regions of 85.6(2)° – 92.1(2)° and 176.6(2)° – 178.1(2)°, respectively. Each pair of adjacent Cu (II) atoms are bridged by a bidentate sulfato group to form a 1D single helical chain running along a crystallographic 2_1 screw axis in b direction with a pitch of the lattice parameter (figure, bottom). Interestingly, the polymeric helical chains possess two types of intrachain hydrogen bonds. One type occurs between coordinating water and uncoordinated sulfato O atoms with d(O···O) = 2.714 Å and ∠O–H···O = 144.8°. The other type is between the uncoordinated –NH groups of the imidazole ligands and uncoordinated sulfato O atoms with d(N···O) = 2.850 Å – 2.873 Å and ∠N–H···O = 173° – 175°. Therefore, the polymeric helical chains are further stabilized by these relatively strong intrachain hydrogen bonds. Through the interchain N–H···O and O–H···O hydrogen bonds, neighboring helical chains are interlinked to form the 3D framework. On the other hand, sulfato groups exhibit slight deviation from T_d symmetry (d(S—O) = 1.450(3) Å – 1.479(3) Å, ∠O–S–O = 108.6(2)° – 110.9(2)°), due to the influence of sulfato groups joining in coordination.

Table 1. Data collection and handling.

Crystal:	blue rod, size 0.06 × 0.08 × 0.22 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	17.41 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.18°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6897, 2495
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1631
$N(param)_{refined}$:	199
Programs:	SHELXS-97 [5], SHELXL-97 [6]

Table 2. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{iso}
H(2A)	4e	0.5787	0.1816	0.5876	0.057
H(4A)	4e	0.6556	1.1432	0.9487	0.066
H(6A)	4e	0.2988	0.9545	0.5952	0.064
H(5A)	4e	0.515(5)	0.493(2)	0.936(4)	0.209
H(5B)	4e	0.401(4)	0.518(6)	0.934(4)	0.209
H(1)	4e	0.6714	0.3345	0.7072	0.052
H(2)	4e	0.4019	0.2995	0.5309	0.063
H(3)	4e	0.3881	0.5427	0.6191	0.056
H(4)	4e	0.6105	1.0158	0.8024	0.066
H(5)	4e	0.6656	0.9609	1.0800	0.066
H(6)	4e	0.6316	0.7097	1.0087	0.059
H(7)	4e	0.4904	0.8747	0.6195	0.054
H(8)	4e	0.1827	0.8613	0.7236	0.070
H(9)	4e	0.3129	0.7167	0.8321	0.058

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cu(1)	4e	0.57914(5)	0.65540(7)	0.78070(5)	0.0468(4)	0.0273(4)	0.0538(4)	0.0093(3)	−0.0122(3)	−0.0129(3)
S(1)	4e	0.7583(1)	0.4237(1)	0.89478(9)	0.0414(7)	0.0216(6)	0.0415(7)	0.0012(5)	0.0045(6)	−0.0007(5)
N(1)	4e	0.5413(3)	0.4833(5)	0.6890(3)	0.036(2)	0.035(2)	0.047(2)	−0.002(2)	0.000(2)	−0.007(2)
N(2)	4e	0.5529(4)	0.2684(5)	0.6080(3)	0.065(3)	0.027(2)	0.051(3)	0.004(2)	0.004(2)	−0.014(2)
N(3)	4e	0.6126(3)	0.8189(4)	0.8780(3)	0.044(2)	0.024(2)	0.051(3)	0.003(2)	−0.001(2)	−0.005(2)
N(4)	4e	0.6452(4)	1.0452(5)	0.9431(3)	0.063(3)	0.032(3)	0.069(3)	−0.012(2)	0.009(2)	−0.017(3)
N(5)	4e	0.4393(3)	0.7708(5)	0.7372(3)	0.046(3)	0.029(2)	0.044(3)	0.007(2)	−0.002(2)	−0.008(2)
N(6)	4e	0.3232(4)	0.8996(6)	0.6433(3)	0.053(3)	0.053(3)	0.053(3)	0.013(2)	−0.003(2)	0.007(2)
O(1)	4e	0.7216(3)	0.5462(4)	0.8261(2)	0.041(2)	0.033(2)	0.045(2)	0.006(2)	−0.007(2)	−0.001(2)
O(2)	4e	0.8001(3)	0.2913(4)	0.8422(2)	0.048(2)	0.026(2)	0.050(2)	0.008(2)	0.004(2)	−0.007(2)
O(3)	4e	0.6625(3)	0.3760(4)	0.9543(3)	0.074(3)	0.037(2)	0.099(3)	0.007(2)	0.054(2)	0.004(2)
O(4)	4e	0.8479(3)	0.4885(4)	0.9583(2)	0.076(2)	0.031(2)	0.047(2)	−0.007(2)	−0.018(2)	0.003(2)
O(5)	4e	0.4598(6)	0.5072(9)	0.9009(5)	0.134(6)	0.117(6)	0.169(6)	−0.029(5)	0.053(5)	−0.010(5)
C(1)	4e	0.6028(4)	0.3574(6)	0.6748(4)	0.043(3)	0.031(3)	0.056(3)	0.004(2)	−0.005(3)	−0.011(3)
C(2)	4e	0.4540(5)	0.3373(6)	0.5770(4)	0.052(3)	0.046(4)	0.059(4)	−0.004(3)	−0.010(3)	−0.019(3)
C(3)	4e	0.4466(4)	0.4707(6)	0.6260(4)	0.042(3)	0.042(3)	0.056(3)	0.002(3)	−0.006(3)	−0.010(3)
C(4)	4e	0.6208(5)	0.9693(7)	0.8626(4)	0.076(4)	0.041(4)	0.049(3)	−0.002(3)	0.009(3)	0.001(3)
C(5)	4e	0.6510(5)	0.9406(7)	1.0148(4)	0.068(4)	0.042(4)	0.055(3)	−0.005(3)	−0.008(3)	−0.006(3)
C(6)	4e	0.6319(4)	0.8029(6)	0.9752(4)	0.061(4)	0.034(3)	0.051(3)	−0.004(3)	−0.005(3)	−0.001(3)
C(7)	4e	0.4301(4)	0.8521(6)	0.6593(4)	0.047(3)	0.040(3)	0.048(3)	−0.007(3)	0.005(2)	−0.005(3)
C(8)	4e	0.2604(5)	0.8462(7)	0.7158(5)	0.045(3)	0.061(4)	0.069(4)	0.014(3)	0.008(3)	−0.005(3)
C(9)	4e	0.3323(4)	0.7662(7)	0.7753(4)	0.053(3)	0.047(4)	0.046(3)	0.007(3)	0.013(3)	−0.008(3)

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