Crystal structure of diaqua-di- μ -hydroxo-bis-(1,10-phenanthroline-*N,N'*)-dicopper(II) succinate octahydrate, $[\text{Cu}_2(\text{C}_{12}\text{H}_8\text{N}_2)_2(\text{H}_2\text{O})_2(\text{OH})_2](\text{C}_4\text{H}_4\text{O}_4) \cdot 8\text{H}_2\text{O}$

Y.-Q. Zheng*, J. Sun and J.-L. Lin

Ningbo University, Chemistry Department, Institute for Solid State Chemistry, Ningbo, Zhejiang, 315211 P. R. China

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ered from both sides by phen ligands of neighboring complexes, leading to 1D intermolecular π - π stacking interactions along [100] (spacings of about 3.94 Å and 4.21 Å). Each complex cation interacts with four neighbors to form 2D layers parallel (001). The hydrogen bonded water molecules and succinate dianions are sandwiched between the 2D layers. Bonds in the succinate anion: $d(\text{C}-\text{O}) = 1.244$ Å, 1.264 Å; $d(\text{C}-\text{C}) = 1.519$ Å; angle $\text{O}-\text{C}-\text{O}$ is 123.0°. One of the five H_2O molecules is coordinated to Cu. Hydrogen bonds between O atoms of H_2O , OH^- and $(\text{C}_4\text{H}_4\text{O}_4)^{2-}$ are responsible for the formation of the 3D network ($d(\text{O}\cdots\text{O}) = 2.719$ Å – 2.914 Å; $\angle(\text{O}-\text{H}\cdots\text{O}) = 141^\circ - 175^\circ$).

Abstract

$\text{C}_{28}\text{H}_{42}\text{Cu}_2\text{N}_4\text{O}_{16}$, triclinic, $P\bar{1}$ (No. 2), $a = 8.151(1)$ Å, $b = 9.520(1)$ Å, $c = 11.845(1)$ Å, $\alpha = 96.70(1)^\circ$, $\beta = 104.45(1)^\circ$, $\gamma = 102.38(1)^\circ$, $V = 855.2$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.031$, $wR_{\text{ref}}(F^2) = 0.091$, $T = 293$ K.

Source of material

Under stirring, 0.43 g (2.52 mmol) copper dichloride dihydrate ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$), 0.50 g (2.52 mmol) 1,10-phenanthroline monohydrate ($\text{C}_{12}\text{H}_8\text{N}_2 \cdot \text{H}_2\text{O}$) and 0.30 g (2.52 mmol) succinic acid were added in 50 ml $\text{CH}_3\text{OH}/\text{H}_2\text{O}$ (1:1 v/v) and insoluble substances were then filtered. To the filtrate Na_2CO_3 (1M) was dropwise added up to pH = 10.8. By slow evaporation, blue crystals were grown from the blue solution (yield: 75% based on the initially-used $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$).

Discussion

Hydrogen bonded H_2O molecules, succinate anions and divalent centrosymmetric $[\text{Cu}_2(\text{phen})_2(\text{H}_2\text{O})_2(\text{OH})_2]^{2+}$ complex cations form the structure. The Cu atoms are fivefold coordinated [1] by one bidentate chelating phenanthroline ligand, two bridging hydroxide groups and one water molecule to form a distorted square pyramid with two phenanthroline N and two hydroxide O atoms equatorial and the water O atom at the apical position ($d(\text{Cu}-\text{N}) = 2.018(2)$ Å, $2.020(2)$ Å; $d(\text{Cu}-\text{O})_{\text{OH}} = 1.933(1)$ Å, $1.946(1)$ Å; $d(\text{Cu}-\text{O})_{\text{H}_2\text{O}} = 2.257(2)$ Å). The Cu atom is shifted by 0.170(1) Å from the basal plane toward the apical water molecule. The $[\text{Cu}_2(\text{phen})_2(\text{H}_2\text{O})_2(\text{OH})_2]^{2+}$ complex cation results from the condensation of two square pyramids via the two OH^- ligands. The $\text{Cu}\cdots\text{Cu}$ distance of 2.889 Å is slightly shorter than that in the hydrogen carbonate [1]. The dinuclear complex cations are orientated in that way that the planar phenanthroline ligands are cov-

Table 1. Data collection and handling.

Crystal:	blue, block, size 0.28 × 0.34 × 0.36 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	13.22 cm ⁻¹
Diffractionmeter, scan mode:	Bruker P4, $\theta/2\theta$
$2\theta_{\text{max}}$:	55°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	4752, 3927
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 3355
$N(\text{param})_{\text{refined}}$:	231
Programs:	SHELXS-97 [2], SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(1)	2i	0.5033	0.7450	0.7683	0.049(3)
H(2)	2i	0.5329	0.9648	0.8889	0.049
H(3)	2i	0.6537	1.1821	0.8396	0.049
H(5)	2i	0.7846	1.3134	0.6905	0.049
H(6)	2i	0.8650	1.2977	0.5214	0.049
H(8)	2i	0.8907	1.1366	0.3390	0.049
H(9)	2i	0.8493	0.9040	0.2366	0.049
H(10)	2i	0.7251	0.7033	0.3084	0.049
H(14A)	2i	0.9453	0.9532	0.8762	0.047(5)
H(14B)	2i	1.1276	0.9500	0.9573	0.047
HO(1)	2i	0.5096	0.5006	0.6616	0.053(8)
HA(O2)	2i	0.8761	0.6592	0.7200	0.070(3)
HB(O2)	2i	0.9267	0.5976	0.6248	0.070
HA(O5)	2i	1.2380	0.5359	0.6262	0.070
HB(O5)	2i	1.1562	0.4968	0.7066	0.070
HA(O6)	2i	0.8579	0.5850	1.0990	0.070
HB(O6)	2i	0.9169	0.4549	1.1038	0.070
HA(O7)	2i	0.7105	0.5423	0.8599	0.070
HB(O7)	2i	0.5800	0.4369	0.8749	0.070
HA(O8)	2i	0.6085	0.7013	1.0169	0.070
HB(O8)	2i	0.4376	0.6472	0.9662	0.070

* Correspondence author (e-mail: zhengcm@nbu.edu.cn)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Cu	2i	0.59174(3)	0.65296(2)	0.53150(2)	0.0375(2)	0.0257(1)	0.0334(2)	0.0054(1)	0.0149(1)	0.00494(9)
N(1)	2i	0.6012(2)	0.8283(2)	0.6488(2)	0.0369(9)	0.0311(8)	0.0364(9)	0.0085(7)	0.0122(7)	0.0047(7)
N(2)	2i	0.6952(2)	0.8097(2)	0.4498(2)	0.0372(9)	0.0286(8)	0.0347(9)	0.0057(7)	0.0115(7)	0.0067(7)
C(1)	2i	0.5514(3)	0.8324(3)	0.7473(2)	0.047(1)	0.041(1)	0.041(1)	0.012(1)	0.017(1)	0.0045(9)
C(2)	2i	0.5694(4)	0.9648(3)	0.8206(2)	0.056(2)	0.055(1)	0.039(1)	0.019(1)	0.018(1)	−0.001(1)
C(3)	2i	0.6406(4)	1.0939(3)	0.7911(2)	0.055(2)	0.042(1)	0.046(1)	0.019(1)	0.009(1)	−0.005(1)
C(4)	2i	0.6940(3)	1.0930(2)	0.6871(2)	0.044(1)	0.033(1)	0.044(1)	0.0133(9)	0.006(1)	0.0005(9)
C(5)	2i	0.7689(4)	1.2218(3)	0.6462(2)	0.060(2)	0.027(1)	0.057(2)	0.009(1)	0.007(1)	0.001(1)
C(6)	2i	0.8167(4)	1.2125(3)	0.5452(2)	0.058(2)	0.028(1)	0.056(2)	0.004(1)	0.008(1)	0.010(1)
C(7)	2i	0.7944(3)	1.0733(2)	0.4735(2)	0.038(1)	0.031(1)	0.044(1)	0.0048(9)	0.0065(9)	0.0113(9)
C(8)	2i	0.8423(3)	1.0553(3)	0.3674(2)	0.046(1)	0.040(1)	0.049(1)	0.002(1)	0.012(1)	0.018(1)
C(9)	2i	0.8171(3)	0.9173(3)	0.3063(2)	0.047(1)	0.047(1)	0.041(1)	0.006(1)	0.017(1)	0.014(1)
C(10)	2i	0.7424(3)	0.7966(2)	0.3505(2)	0.042(1)	0.037(1)	0.040(1)	0.0083(9)	0.014(1)	0.0081(9)
C(11)	2i	0.7214(3)	0.9465(2)	0.5109(2)	0.031(1)	0.031(1)	0.036(1)	0.0083(8)	0.0050(8)	0.0082(8)
C(12)	2i	0.6714(3)	0.9562(2)	0.6187(2)	0.034(1)	0.031(1)	0.037(1)	0.0099(8)	0.0073(8)	0.0045(8)
C(13)	2i	0.9289(3)	0.7851(3)	0.9689(2)	0.044(1)	0.042(1)	0.037(1)	0.008(1)	0.011(1)	0.0054(9)
C(14)	2i	1.0055(3)	0.9396(2)	0.9542(2)	0.042(1)	0.041(1)	0.040(1)	0.008(1)	0.016(1)	0.0065(9)
O(1)	2i	0.4458(2)	0.5100(2)	0.5938(1)	0.0400(8)	0.0293(7)	0.0339(7)	0.0046(6)	0.0156(6)	0.0044(6)
O(2)	2i	0.8514(2)	0.6375(2)	0.6463(2)	0.044(1)	0.077(1)	0.0432(9)	0.0275(9)	0.0140(8)	0.0125(9)
O(3)	2i	0.8692(3)	0.7655(2)	1.0544(2)	0.100(2)	0.044(1)	0.060(1)	0.006(1)	0.045(1)	0.0084(9)
O(4)	2i	0.9278(3)	0.6813(2)	0.8914(2)	0.066(1)	0.0430(9)	0.049(1)	0.0113(8)	0.0206(9)	0.0004(8)
O(5)	2i	1.1441(3)	0.5437(3)	0.6470(2)	0.062(1)	0.100(2)	0.066(1)	0.047(1)	0.034(1)	0.040(1)
O(6)	2i	0.8289(3)	0.4905(2)	1.1187(2)	0.075(1)	0.064(1)	0.064(1)	0.028(1)	0.029(1)	0.028(1)
O(7)	2i	0.6045(3)	0.4807(3)	0.8211(2)	0.079(2)	0.075(1)	0.047(1)	−0.013(1)	0.003(1)	0.020(1)
O(8)	2i	0.4935(3)	0.7085(3)	1.0238(2)	0.081(2)	0.079(2)	0.068(1)	0.016(1)	0.036(1)	0.002(1)

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