Refinement of the crystal structure of diaqua-(3,10-*C-meso*-3,5,7,7,10,12,14,14-octamethyl-1,4,8,11-tetraazacyclotetradecane)copper(II) diperchlorate, $[\text{Cu}(\text{C}_{18}\text{H}_{40}\text{N}_4)(\text{H}_2\text{O})_2](\text{ClO}_4)_2$, at 173 K

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Abstract

$\text{C}_{18}\text{H}_{44}\text{Cl}_2\text{CuN}_4\text{O}_{10}$, monoclinic, $P12_1/c1$ (No. 14), $a = 8.111(2) \text{ \AA}$, $b = 15.993(4) \text{ \AA}$, $c = 10.344(4) \text{ \AA}$, $\beta = 102.56(2)^\circ$, $V = 1309.7 \text{ \AA}^3$, $Z = 2$, $R_{\text{int}}(F) = 0.045$, $wR_{\text{ref}}(F) = 0.042$, $T = 173 \text{ K}$.

Source of material

The title compound was prepared as per the literature method [1].

Discussion

New data is presented for the title compound [1]. The copper(II) cation is located on a crystallographic centre of inversion and exists in a tetragonally distorted octahedral geometry defined by a N_4O_2 donor set. The Cu—N1, N4 distances of 2.037(2) Å and 2.028(3) Å, respectively, are experimentally equivalent, mirror those found in the room temperature structure [1] and match those found in an isomeric complex [2]. The Cu—O1 distance is 2.763(3) Å and is noteworthy that this separation is significantly less than that found in the room temperature structure of 2.815(3) Å [1]. The four N-chiral centres of the 14-membered ring are in the 1*RS*, 4*RS*, 8*SR* and 11*SR* configuration with two NH groups above the CuN_4 equatorial plane and two below. The methyl groups bound to the chiral C3 atoms occupy axial positions and those bound to chiral-C5 occupy equatorial positions.

There are several significant H-bonding interactions in the structure. Each of the water-bound H atoms forms a donor interaction to a perchlorate-oxygen atom: $d(\text{O1}—\text{H1o})$ 0.91 Å, $d(\text{H1o} \cdots \text{O5})$ 2.05 Å, $d(\text{O1} \cdots \text{O5})$ 2.798(3) Å and the angle at H1o 138°; $d(\text{O1}—\text{H2o})$ is 0.94 Å, $d(\text{H2o} \cdots \text{O3}^i)$ is 1.98 Å, $d(\text{O1} \cdots \text{O3}^i)$ 2.895(2) Å and the angle at H2o is 167°; symmetry operation $i: x, 1/2-y, 1/2+z$. Similarly, the amine H atoms form donor interactions to perchlorate-oxygen: $d(\text{H1n} \cdots \text{O5}^{ii})$ 2.52 Å, $d(\text{N1} \cdots \text{O5}^{ii})$ 3.462(2) Å and the angle at H1n is 170° with symmetry operation $ii: -x, -y, -z$; $d(\text{H2n} \cdots \text{O4}^i)$ 2.34 Å, $d(\text{N4} \cdots \text{O4}^i)$ 3.209(2) Å and the angle at H2n is 152°. This mode of association results in the formation of a two-dimensional structure.

Table 1. Data collection and handling.

Crystal:	red block, size 0.26 × 0.57 × 0.57 mm
Wavelength:	Mo K_{α} radiation (0.7107 Å)
μ :	10.95 cm ⁻¹
Diffraction, scan mode:	Rigaku AFC7R, $\omega/2\theta$
$2\theta_{\text{max}}$:	55°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	3338, 3129
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 3 \sigma(I_{\text{obs}})$, 2462
$N(\text{param})_{\text{refined}}$:	160
Programs:	teXsan [3], DIRDIF [4], SHELXL-97 [5], ORTEPII [6]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4e	0.0240	0.0895	0.3321	0.026
H(2)	4e	0.1287	0.1202	0.2325	0.026
H(3)	4e	-0.2833	0.0858	0.2715	0.034
H(4)	4e	-0.3802	0.1143	0.1323	0.034
H(5)	4e	-0.2912	0.0279	0.1500	0.034
H(6)	4e	-0.1287	0.1822	0.1725	0.024
H(7)	4e	-0.3496	0.1060	-0.0820	0.024
H(8)	4e	-0.3076	0.2400	0.0228	0.037
H(9)	4e	-0.2243	0.2678	-0.0917	0.037
H(10)	4e	-0.4135	0.2426	-0.1214	0.037
H(11)	4e	-0.3036	0.1598	-0.2844	0.025
H(12)	4e	-0.1120	0.1549	-0.2213	0.025
H(13)	4e	-0.1362	0.0960	-0.4372	0.034
H(14)	4e	-0.1551	-0.0003	-0.4523	0.034
H(15)	4e	-0.3144	0.0562	-0.4785	0.034
H(16)	4e	-0.3777	-0.0103	-0.1783	0.031
H(17)	4e	-0.4664	0.0172	-0.3202	0.031
H(18)	4e	-0.3584	-0.0637	-0.2992	0.031
H(1o)	4e	0.2483	0.1404	-0.1006	0.014
H(2o)	4e	0.3026	0.1476	0.0332	0.014
H(1n)	4e	-0.0356	-0.0297	0.2255	0.021
H(2n)	4e	-0.0207	0.1519	0.0075	0.021

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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	2a	0	0	0	0.0223(1)	0.0187(1)	0.0147(1)	0.0069(1)	0.0010(1)	-0.0021(1)
Cl	4e	0.25492(6)	0.19170(3)	-0.38781(5)	0.0305(2)	0.0246(2)	0.0403(3)	0.0032(2)	-0.0047(2)	-0.0025(2)
O(1)	4e	0.2584(2)	0.10625(9)	-0.0285(1)	0.0451(8)	0.0467(9)	0.0421(8)	-0.0067(7)	0.0064(7)	-0.0062(7)
O(2)	4e	0.3550(2)	0.1572(1)	-0.4725(2)	0.083(1)	0.090(2)	0.104(2)	0.001(1)	0.039(1)	-0.027(1)
O(3)	4e	0.3444(2)	0.2639(1)	-0.3303(2)	0.078(1)	0.043(1)	0.072(1)	-0.0002(8)	-0.0304(9)	-0.0106(9)
O(4)	4e	0.0990(2)	0.2143(1)	-0.4674(2)	0.0417(9)	0.049(1)	0.131(2)	0.0042(8)	-0.029(1)	-0.000(1)
O(5)	4e	0.2393(2)	0.1284(1)	-0.2997(2)	0.117(2)	0.106(2)	0.075(1)	-0.025(1)	0.022(1)	0.032(1)
N(1)	4e	0.0550(1)	-0.00008(8)	0.2018(1)	0.0174(6)	0.0172(6)	0.0169(6)	0.0006(6)	0.0028(5)	-0.0005(6)
N(4)	4e	-0.1066(1)	0.11321(8)	0.0157(1)	0.0174(6)	0.0166(7)	0.0177(6)	0.0002(5)	0.0036(5)	-0.0013(5)
C(2)	4e	0.0337(2)	0.0881(1)	0.2422(2)	0.0260(9)	0.0196(8)	0.0192(8)	0.0008(7)	0.0023(7)	-0.0046(7)
C(3')	4e	-0.2848(2)	0.0843(1)	0.1795(2)	0.0277(9)	0.030(1)	0.0293(9)	0.0045(8)	0.0130(7)	0.0005(8)
C(3)	4e	-0.1250(2)	0.1240(1)	0.1548(2)	0.0231(8)	0.0178(8)	0.0203(8)	0.0026(7)	0.0049(6)	-0.0040(7)
C(5)	4e	-0.2565(2)	0.1408(1)	-0.0878(2)	0.0185(8)	0.0197(8)	0.0221(8)	0.0033(6)	0.0023(7)	0.0004(7)
C(5')	4e	-0.3051(2)	0.2314(1)	-0.0676(2)	0.034(1)	0.0235(9)	0.030(1)	0.0102(8)	-0.0034(8)	-0.0040(8)
C(6)	4e	-0.2190(2)	0.1300(1)	-0.2239(2)	0.0200(8)	0.0179(8)	0.0230(8)	0.0007(6)	0.0006(7)	0.0041(7)
C(7')	4e	-0.2038(2)	0.0492(1)	-0.4258(2)	0.037(1)	0.028(1)	0.0190(8)	0.0009(8)	0.0023(7)	0.0038(7)
C(7)	4e	-0.2127(2)	0.0420(1)	-0.2793(1)	0.0193(8)	0.0182(8)	0.0166(8)	0.0004(6)	0.0015(6)	0.0032(6)
C(7'')	4e	-0.3684(2)	-0.0085(1)	-0.2682(2)	0.0209(8)	0.0241(9)	0.0305(9)	-0.0027(7)	0.0020(7)	0.0006(8)

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