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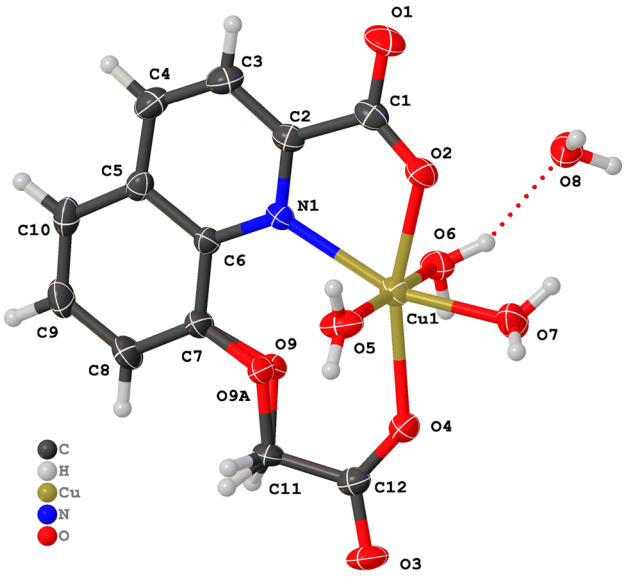
The crystal structure of [triaqua-(8-carboxymethoxy-quinoline-2-carboxylato-κ^3*N*,*O*,*O*)copper(II)]monohydrate, $C_{12}H_{15}NO_9Cu$



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Abstract

$C_{12}H_{15}NO_9Cu$, triclinic, $P\bar{1}$ (no. 2), a = 7.3596(3) Å, b = 8.1735(2) Å, c = 12.0982(4) Å, α = 85.754(3)°, β = 80.318(3)°, γ = 84.852(2)°, V = 702.61(5) Å^3, Z = 2, $R_{gt}(F)$ = 0.0494, $wR_{ref}(F^2)$ = 0.1463, T = 298 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Pink block
Size:	0.28 × 0.26 × 0.22 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.61 mm^{-1}
Diffractometer, scan mode:	XtaLAB AFC12 (RINC),
θ_{max}, completeness:	25.3°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	17674, 2564, 0.036
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2489
$N(param)_{refined}$:	228
Programs:	Bruker,[1] Olex2,[2] SHELX,[3] Diamond[4]

1 Source of materials

The title Cu(II) complex has been synthesized by the following method: 0.1336 g 8-carboxymethoxy-quinoline-2-carboxylic acid (0.5 mmol), 0.1011 g 2-(2-methyl-imidazol-1-yl)benzoic acid (0.5 mmol), 0.040 g NaOH (1.0 mmol) were added to the solution of 15 ml water-ethanol (v:v = 2:1) with stirring. After dissolution, 0.0998 g cupric acetate monohydrate (0.5 mmol) solid was added to the above solution with stirring. Then the mixture solution was stirred for 4 h at 75 °C and for another 3 h at room temperature. Blue block crystals were obtained after 10 days.

2 Experimental details

The hydrogen atoms were positioned geometrically (C–H = 0.93–0.97 Å, O–H = 0.85–0.86 Å). Their U_{iso} values were set to 1.2 U_{eq} or 1.5 U_{eq} of the parent atoms.

3 Comment

The Cu(II), Ni(II), Co(II), and Mn(II) complexes constructed by 8-carboxymethoxyquinoline-2-carboxylate ligand have showed multiple coordination modes and potential applications such as magnetic property, DNA/BSA binding and DNA cleavage ability.[5,6] Our group has also synthesized and structurally characterized Cu(II), Ni(II), Cd(II), and Zn(II) complexes using 8-carboxymethoxy-quinoline-2-carboxylic

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.69719 (8)	0.72438 (7)	0.14864 (5)	0.0285 (2)
O1	0.2107 (5)	0.4914 (5)	0.1937 (3)	0.0426 (9)
O2	0.4692 (5)	0.6120 (4)	0.1209 (3)	0.0322 (8)
O3	1.1907 (5)	0.9957 (5)	0.1490 (3)	0.0419 (9)
O4	0.9665 (5)	0.8446 (4)	0.1205 (3)	0.0331 (8)
O5	0.5716 (5)	0.9547 (5)	0.1303 (4)	0.0393 (9)
H5A	0.456769	0.968792	0.123955	0.059*
H5B	0.610948	1.049945	0.116167	0.059*
O6	0.8352 (5)	0.5007 (4)	0.1823 (3)	0.0315 (8)
H6A	0.952960	0.511117	0.179111	0.047*
H6B	0.833077	0.435526	0.129494	0.047*
O7	0.7923 (7)	0.7118 (5)	−0.0229 (3)	0.0445 (10)
H7A	0.793041	0.802046	−0.063067	0.067*
H7B	0.756373	0.644107	−0.062950	0.067*
N1	0.5163 (5)	0.6921 (4)	0.3211 (3)	0.0224 (7)
C1	0.3464 (6)	0.5689 (6)	0.2017 (4)	0.0275 (9)
C2	0.3652 (6)	0.6186 (5)	0.3171 (4)	0.0246 (9)
C3	0.2297 (7)	0.5877 (6)	0.4139 (4)	0.0329 (10)
H3	0.123806	0.536308	0.407750	0.040*
C4	0.2542 (7)	0.6332 (6)	0.5160 (4)	0.0342 (11)
H4	0.164941	0.612958	0.579844	0.041*
C5	0.4134 (6)	0.7105 (5)	0.5252 (4)	0.0250 (9)
C6	0.5430 (6)	0.7371 (5)	0.4233 (4)	0.0234 (9)
C7	0.7072 (7)	0.8122 (7)	0.4275 (4)	0.0335 (11)
C8	0.7421 (8)	0.8612 (7)	0.5275 (4)	0.0393 (12)
H8	0.850036	0.912678	0.529019	0.047*
C9	0.6149 (8)	0.8336 (7)	0.6272 (4)	0.0358 (11)
H9	0.640291	0.865659	0.694945	0.043*
C10	0.4546 (7)	0.7608 (6)	0.6271 (4)	0.0321 (10)
H10	0.371740	0.743983	0.694448	0.039*
C11	0.9639 (7)	0.9289 (6)	0.3043 (4)	0.0316 (11)
H11A	1.061518	0.907845	0.350063	0.038*
H11B	0.900246	1.036242	0.319517	0.038*
H11C	1.044331	0.863874	0.350113	0.038*
H11D	0.950139	1.041444	0.326268	0.038*
C12	1.0454 (6)	0.9222 (6)	0.1808 (4)	0.0268 (9)
O9	0.835 (2)	0.805 (2)	0.3290 (9)	0.034 (3)
O9A	0.788 (3)	0.864 (5)	0.3189 (10)	0.028 (4)
O8	0.7405 (5)	0.2571 (5)	0.0574 (3)	0.0337 (8)
H8A	0.829357	0.210581	0.012400	0.051*
H8B	0.666147	0.302194	0.015025	0.051*

acid ligand.[7–10] According to the above studies, it was found that the synthesis conditions and pH could affect the structure of the complex, for example, under the same hydrothermal conditions, nickel(II) acetate tetrahydrate and nickel (II) nitrate hexahydrate respectively form six-coordinated complexes with different structures with 8-carboxymethoxyquinoline-2-carboxylic acid ligand.[6] However, under ordinary pressure conditions, nickel(II) acetate tetrahydrate forms seven-coordinated complex with 8-carboxymethoxyquinoline-2-carboxylic acid ligand.[10] To further explore the effect of the synthesis conditions on the structure of metal complexes with the 8-carboxymethoxyquinoline-2-carboxylic acid, in this paper, we synthesized and structurally characterized a new Cu(II) complex using nickel(II) acetate tetrahydrate, 8-carboxymethoxy-quinoline-2-carboxylic acid, 2-(2-methyl-imidazol-1-yl)benzoic acid, and NaOH. The molecular structure of Cu(II) complex is shown in Figure. The Cu(II) complex contains one central Cu(II) ion, one completely deprotonated 8-carboxymethoxy-quinoline-2-carboxylate ligand, three coordinated water molecules, and one uncoordinated water molecule. The Cu(II) ion is six-coordinated by two carboxylic O atoms (O2 and O4) and one N atom (N1) from 8-carboxymethoxy-quinoline-2-carboxylate ligand, three O atoms from three coordinated water molecules, adopting a distorted octahedral coordination geometry. Unfortunately, the 2-(2-methyl-imidazol-1-yl)benzoic acid ligand does not take part in coordination with Cu(II) ion. The bond angle of O5–Cu1–O6 is 173.52(15)°, indicating that the O5 and O6 of coordinated water molecules occupy the axial positions. And the N1, O2, O4 and O7 atoms from the basal plane. The angles around the Cu(II) ion within the basal plane vary from 74.88(13) to 122.87(13)°, and the sum of bond angles is 360.02°. The bond lengths of Cu–O and Cu–N are 2.031(4)–2.239(3) Å and 2.264(4) Å, respectively, which is consistent with other Cu(II) complexes reported in the literature.[11–14] The Cu(II) complex molecules form 1D chained structure by intermolecular O–H·O hydrogen bonds.

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