Crystal structure of diaqua-(2-carboxyethylene-8-hydroxyquinolinato)-copper(II) monohydrate, $Cu(C_{12}H_9O_3N)(H_2O)_2 \cdot H_2O$

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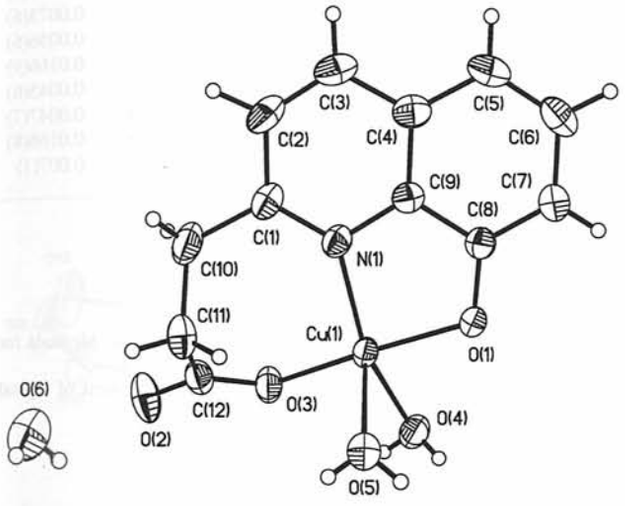

Abstract

$C_{12}H_{15}CuNO_6$, triclinic, $P\bar{1}$ (No. 2), $a = 7.363(2)$ Å, $b = 8.985(2)$ Å, $c = 10.349(2)$ Å, $\alpha = 97.438(4)°$, $\beta = 100.846(4)°$, $\gamma = 106.334(4)°$, $V = 633.1$ Å^3, $Z = 2$, $R_{gt}(F) = 0.029$, $wR_{obs}(F^2) = 0.073$, $T = 298$ K

Source of material

To an aqueous dioxane solution of 2-bis(ethoxycarbonyl)ethylene-8-hydroxyquinoline, 3 eq. copper(II) perchlorate aqueous solution was added. The green crystals were recrystallized from hot water.

Discussion

8-Hydroxyquinoline and its substituted analogues are widely known as extracting agents for transition metals. In the case of the copper-complexes, the two 8-hydroxyquinoline ligands are coordinated to the central Cu atom through their O and N atoms, forming a four-membered chelating ring [1–3]. We synthesized a 8-hydroxyquinoline having a carboxyethanyl group at the 2-position to act as an additional group with the ability to form a coordinate bond to the metal, and reacted it with aqueous Cu (II) perchlorate.

In the title complex, the coordination of Cu (II) atom is close to square-pyramidal with three O atoms (one from a water molecule) and one N atom in the basal plane and the other H_2O molecule in the apical position. The Cu—N bond length is 2.039(2) Å. The Cu—O bond lengths in the basal positions are in the range from 1.919(2) Å to 2.028(2) Å. The axial Cu—O(H_2O) bond length (2.329(2) Å), however, is significantly longer than the basal Cu—O bonds. These Cu—O and Cu—N bond distances are in agreement with those in other bis-(8-hydroxyquinoline)Cu(II) complexes. The five-membered chelate ring and the 8-hydroxyquinoline ligands are coplanar.

Table 1. Data collection and handling.

Crystal:	green block, size 0.2 × 0.4 × 0.5 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	17.51 cm^{-1}
Diffractometer, scan mode:	Bruker Smart 1000, $\theta/2\theta$
$2\theta_{max}$:	53.5°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3367, 2330
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2187
$N(param)_{refined}$:	241
Programs:	SHELXS-97 [4], SHELXL-97 [5]

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

Atom	Site	x	y	z	U_{iso}
H(2)	2*i*	0.617(4)	–0.210(3)	0.227(3)	0.050(7)
H(3)	2*i*	0.551(4)	–0.317(3)	0.003(3)	0.042(7)
H(5)	2*i*	0.572(4)	–0.269(3)	–0.233(3)	0.038(6)
H(6)	2*i*	0.671(4)	–0.101(4)	–0.371(3)	0.058(8)
H(7)	2*i*	0.850(4)	0.156(3)	–0.278(3)	0.041(7)
H(4A)	2*i*	1.204(4)	0.567(4)	0.114(3)	0.042(8)
H(4B)	2*i*	1.296(5)	0.549(4)	0.214(3)	0.05(1)
H(5A)	2*i*	0.847(5)	0.534(4)	0.243(4)	0.06(1)
H(5B)	2*i*	0.864(4)	0.563(4)	0.140(3)	0.045(8)
H(6A)	2*i*	0.436(5)	0.384(4)	0.598(3)	0.06(1)
H(6B)	2*i*	0.587(5)	0.414(4)	0.578(3)	0.05(1)
H(10A)	2*i*	0.701(5)	0.012(4)	0.384(3)	0.067(9)
H(10B)	2*i*	0.918(5)	0.034(4)	0.411(3)	0.062(8)
H(11A)	2*i*	0.767(3)	0.286(3)	0.347(2)	0.035(6)
H(11B)	2*i*	0.794(4)	0.253(3)	0.484(3)	0.043(7)

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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}
C(1)	2*i*	0.7664(3)	0.0089(2)	0.2123(2)	0.029(1)	0.034(1)	0.033(1)	0.0155(8)	0.0071(8)	0.0086(8)
C(2)	2*i*	0.6587(3)	–0.1528(3)	0.1622(3)	0.039(1)	0.034(1)	0.050(1)	0.020(1)	0.013(1)	0.0035(9)
C(3)	2*i*	0.6146(3)	–0.2155(3)	0.0304(3)	0.036(1)	0.026(1)	0.053(1)	0.008(1)	0.008(1)	0.0018(9)
C(4)	2*i*	0.6759(3)	–0.1184(2)	–0.0606(2)	0.027(1)	0.029(1)	0.041(1)	0.0050(8)	0.0053(8)	0.0081(8)
C(5)	2*i*	0.6354(3)	–0.1715(3)	–0.2000(2)	0.035(1)	0.030(1)	0.042(1)	–0.0062(9)	0.0007(9)	0.0060(9)
C(6)	2*i*	0.6976(4)	–0.0681(3)	–0.2803(2)	0.046(1)	0.046(1)	0.028(1)	–0.0055(9)	0.0011(9)	0.016(1)
C(7)	2*i*	0.8035(3)	0.0911(3)	–0.2260(2)	0.047(1)	0.038(1)	0.027(1)	0.0071(9)	0.0074(9)	0.012(1)
C(8)	2*i*	0.8477(3)	0.1473(2)	–0.0897(2)	0.031(1)	0.028(1)	0.027(1)	0.0062(8)	0.0041(8)	0.0096(8)
C(9)	2*i*	0.7831(3)	0.0407(2)	–0.0049(2)	0.0241(9)	0.0254(9)	0.029(1)	0.0056(7)	0.0039(7)	0.0081(7)
C(10)	2*i*	0.8043(4)	0.0665(3)	0.3605(2)	0.049(1)	0.047(1)	0.033(1)	0.020(1)	0.009(1)	0.004(1)
C(11)	2*i*	0.8409(3)	0.2417(3)	0.4123(2)	0.037(1)	0.053(1)	0.029(1)	0.012(1)	0.0145(9)	0.015(1)
C(12)	2*i*	1.0544(3)	0.3324(2)	0.4448(2)	0.038(1)	0.037(1)	0.023(1)	0.0056(8)	0.0071(8)	0.0158(9)
Cu(1)	2*i*	1.01344(3)	0.32868(2)	0.15998(2)	0.0351(2)	0.0252(2)	0.0202(2)	0.0069(1)	0.0050(1)	0.0041(1)
N(1)	2*i*	0.8298(2)	0.1037(2)	0.1303(2)	0.0266(8)	0.0280(8)	0.0274(8)	0.0098(6)	0.0041(6)	0.0073(6)
O(1)	2*i*	0.9459(2)	0.2971(2)	–0.0322(1)	0.0487(9)	0.0244(7)	0.0228(7)	0.0075(5)	0.0065(6)	0.0056(6)
O(2)	2*i*	1.1522(2)	0.3626(2)	0.5620(2)	0.047(1)	0.080(1)	0.0222(8)	0.0065(8)	0.0051(7)	0.0166(9)
O(3)	2*i*	1.1324(2)	0.3709(2)	0.3492(1)	0.0358(8)	0.0391(8)	0.0233(7)	0.0081(6)	0.0070(6)	0.0045(6)
O(4)	2*i*	1.2397(3)	0.5121(2)	0.1466(2)	0.0408(9)	0.0325(8)	0.0316(9)	0.0115(7)	0.0042(7)	0.0047(7)
O(5)	2*i*	0.8249(3)	0.4963(2)	0.1765(2)	0.056(1)	0.0368(9)	0.0316(9)	0.0094(8)	0.0145(8)	0.0166(8)
O(6)	2*i*	0.5318(3)	0.3591(3)	0.6004(2)	0.041(1)	0.057(1)	0.050(1)	0.0178(9)	0.0048(9)	0.007(1)

References

1. Okabe, N.; Saishu, H.: Bisaquabis(8-hydroxyquinolinato-*N,O*)copper(II). Acta Crystallogr. E**57** (2001) 251-252.
2. Shoja, M.; Gershon, H.; Clarke, D. D.: Crystal structure of bis(7-nitro-8-quinolinolato)copper(II). $CuC_{18}H_{10}O_6N_4$. Z. Kristallogr. NCS **215** (2000) 273-274.
3. Jian, F.; Wang, Y.; Lu, L.; Yang, X.; Wang, X.; Chantrapromma, S.; Fun, H.-K.; Ibrahim, A.: (2-Methylquinoline-8-olato)iron(III) and -copper(II) complex. Acta Crystallogr. C**57** (2001) 714-716.
4. Sheldrick, G. M.: Phase Annealing in SHELX-90: Direct Methods for Larger Structures. Acta Crystallogr. A**46** (1990) 467-473.
5. Sheldrick, G. M.: SHELXL-97. Program for the refinement of crystal structures. University of Göttingen, Germany 1997.