Crystal structure of chloro(glycinato-*N*,*O*)(1,10-phenanthroline-*N*,*N*′)-copper(II) trihydrate, $[CuCl(C_2H_4NO_2)(C_{12}H_8N_2)] \cdot 3H_2O$

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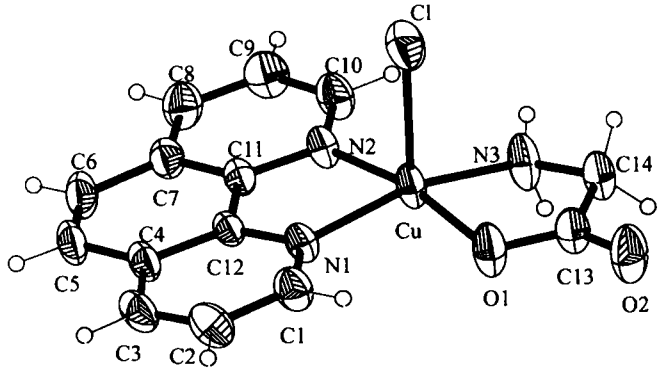

Abstract

$C_{14}H_{18}ClCuN_3O_5$, monoclinic, $P12_1/n1$ (No. 14), $a = 7.065(2)$ Å, $b = 12.272(3)$ Å, $c = 20.242(5)$ Å, $\beta = 94.88(1)°$, $V = 1748.7$ Å^3, $Z = 4$, $R_{gt}(F) = 0.064$, $wR_{ref}(F^2) = 0.166$, $T = 293$ K.

Source of material

Under continuous stirring, 0.264 g (1.33 mmol) phenanthroline monohydrate, 0.100 g (1.33 mmol) glycine, and 0.227 g (1.33 mmol) $CuCl_2 \cdot 2H_2O$ were successively dissolved in 20 ml of the CH_3OH/H_2O mixture (1:1 v/v). Then, 1.0 ml of the 1M Na_2CO_3 solution was dropwise added and pale blue precipitate was formed. After the deposit was removed, the filtrate was allowed to stand at room temperature. Blue crystals grew by slow evaporation for several weeks.

Discussion

The crystal structure consists of $[CuCl(C_2H_4NO_2)(C_{12}H_8N_2)]$ complex molecules and hydrogen bonded H_2O molecules. Within each complex molecules, the central Cu atom is square pyramidally coordinated by one Cl atom, one O atom and three N atoms from one bidentate chelating phenanthroline ligand and one bidentate chelating glycinato ligand, respectively, with the Cl atom at the apical position. The coordination environment around the central Cu atom is significantly distorted with the Cu atom shifted by 0.244(2) Å away from the basal plane towards the apical Cl atom. The Cu—N bond lengths vary from 2.013 Å to 2.022 Å while the Cu—O and Cu—Cl bond lengths are 1.941 Å and 2.576 Å, respectively. The bidentate phen ligands exhibit nearly perfect coplanarity. Along the [100], the complex molecules, *via* intermolecular π–π stacking interactions between the chelating phenanthroline rings with the mean spacings of 3.38 Å, are stacked with the Cu—Cl bonds alternatively upwards and downwards into 1D columns. The formed columns are arranged to meet the requirements of pseudo 1D close-packing. For the glycinato ligands, the C—O bond for the coordinating carboxyl O atom is 1.288 Å substantially longer than 1.231 Å for the non-coordinating one. In the crystal structure exist three crystallographically independent H_2O molecules, of which two crystallographic positions for water O5 and O6 atoms are half occupied. As hydrogen bonding donors, the H_2O mole-cules are located in the cavities to connect the 1D columns.

Table 1. Data collection and handling.

Crystal:	blue, block, size 0.156 × 0.267 × 0.422 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	14.30 cm^{-1}
Diffractometer, scan mode:	Bruker P4, θ/2θ
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5412, 4001
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3174
$N(param)_{refined}$:	223
Programs:	SHELXS-97 [1], SHELXL-97 [2]

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(1)	4*e*		0.3822	0.7134	0.0743	0.061(7)
H(2)	4*e*		0.2772	0.6805	–0.0361	0.061
H(3)	4*e*		0.1805	0.8218	–0.1039	0.061
H(5)	4*e*		0.1233	1.0279	–0.1165	0.061
H(6)	4*e*		0.1415	1.1961	–0.0723	0.061
H(8)	4*e*		0.2123	1.3172	0.0319	0.061
H(9)	4*e*		0.3169	1.3259	0.1428	0.061
H(10)	4*e*		0.4102	1.1694	0.1999	0.061
O(6)	4*e*	0.50	0.427(2)	0.486(1)	–0.0561(8)	0.122(5)
H(14A)	4*e*		0.5546	0.8708	0.3192	0.050
H(14B)	4*e*		0.7801	0.879	0.3069	0.050
HA(N3)	4*e*		0.7241	0.9801	0.2416	0.050
HB(N3)	4*e*		0.5615	1.0323	0.2679	0.050
HA(O3)	4*e*		0.4285	0.4860	0.2074	0.050
HB(O3)	4*e*		0.4873	0.6046	0.2047	0.050
HA(O4)	4*e*		0.3090	1.1072	0.3322	0.050
HB(O4)	4*e*		0.4011	1.2083	0.3307	0.050
HA(O5)	4*e*	0.50	0.2146	1.4734	–0.0587	0.050
HB(O5)	4*e*	0.50	0.0331	1.4449	–0.0885	0.050
HA(O6)	4*e*	0.50	0.4889	0.4907	–0.0372	0.050
HB(O6)	4*e*	0.50	0.4745	0.4723	–0.0932	0.050

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cu	4*e*		0.4391(1)	0.91951(5)	0.16464(3)	0.0571(4)	0.0350(3)	0.0243(3)	–0.0001(3)	–0.0094(2)	–0.0002(2)
Cl	4*e*		0.1244(2)	0.8818(1)	0.21608(7)	0.067(1)	0.0645(9)	0.0402(7)	–0.0034(8)	0.0011(6)	0.0123(7)
N(1)	4*e*		0.3523(6)	0.8712(4)	0.0718(2)	0.046(2)	0.039(2)	0.025(2)	–0.003(2)	–0.001(2)	–0.004(2)
N(2)	4*e*		0.3668(6)	1.0672(3)	0.1259(2)	0.048(2)	0.038(2)	0.025(2)	–0.005(2)	–0.003(2)	0.001(2)
N(3)	4*e*		0.5961(8)	0.9702(4)	0.2466(2)	0.077(3)	0.039(2)	0.036(2)	–0.011(2)	–0.020(2)	0.003(2)
C(1)	4*e*		0.3443(8)	0.7717(5)	0.0470(3)	0.060(4)	0.042(3)	0.036(3)	–0.001(3)	–0.001(2)	–0.005(2)
C(2)	4*e*		0.2801(9)	0.7514(5)	–0.0197(3)	0.066(4)	0.049(3)	0.041(3)	–0.008(3)	0.002(3)	–0.017(3)
C(3)	4*e*		0.2227(8)	0.8351(5)	–0.0598(3)	0.047(3)	0.069(4)	0.029(2)	–0.012(3)	–0.001(2)	–0.014(3)
C(4)	4*e*		0.2267(7)	0.9422(5)	–0.0351(2)	0.037(3)	0.058(3)	0.023(2)	–0.008(2)	–0.002(2)	–0.002(2)
C(5)	4*e*		0.1689(8)	1.0361(5)	–0.0723(2)	0.043(3)	0.073(4)	0.024(2)	–0.005(3)	–0.007(2)	0.005(3)
C(6)	4*e*		0.1775(8)	1.1364(5)	–0.0459(3)	0.048(3)	0.065(4)	0.033(3)	0.000(3)	–0.005(2)	0.017(3)
C(7)	4*e*		0.2422(7)	1.1529(5)	0.0231(2)	0.040(3)	0.048(3)	0.033(2)	–0.002(2)	0.001(2)	0.006(2)
C(8)	4*e*		0.2495(9)	1.2542(5)	0.0550(3)	0.060(4)	0.044(3)	0.048(3)	0.003(3)	–0.002(3)	0.010(3)
C(9)	4*e*		0.3122(9)	1.2592(5)	0.1209(3)	0.068(4)	0.037(3)	0.052(3)	0.000(3)	0.001(3)	–0.005(3)
C(10)	4*e*		0.3692(9)	1.1644(5)	0.1551(3)	0.065(4)	0.039(3)	0.035(3)	–0.004(3)	–0.005(2)	–0.007(2)
C(11)	4*e*		0.3009(7)	1.0612(4)	0.0608(2)	0.034(2)	0.039(3)	0.025(2)	–0.004(2)	0.000(2)	0.002(2)
C(12)	4*e*		0.2943(7)	0.9558(4)	0.0319(2)	0.032(2)	0.041(3)	0.023(2)	–0.004(2)	0.001(2)	–0.001(2)
C(13)	4*e*		0.6235(8)	0.7689(5)	0.2476(3)	0.056(3)	0.047(3)	0.035(3)	0.002(3)	–0.007(2)	0.003(2)
C(14)	4*e*		0.6443(9)	0.8719(5)	0.2876(3)	0.061(4)	0.047(3)	0.032(2)	0.000(3)	–0.010(2)	0.004(2)
O(1)	4*e*		0.5509(7)	0.7785(3)	0.1873(2)	0.087(3)	0.041(2)	0.037(2)	0.009(2)	–0.020(2)	–0.003(2)
O(2)	4*e*		0.6735(7)	0.6816(4)	0.2735(2)	0.098(4)	0.044(2)	0.051(2)	0.012(2)	–0.019(2)	0.008(2)
O(3)	4*e*		0.429(1)	0.5512(5)	0.1739(3)	0.191(8)	0.057(3)	0.096(5)	–0.030(4)	–0.015(5)	–0.008(3)
O(4)	4*e*		0.434(1)	1.1346(5)	0.3358(3)	0.206(8)	0.072(4)	0.085(4)	0.008(5)	0.023(5)	–0.004(4)
O(5)	4*e*	0.50	0.077(2)	1.480(1)	–0.0537(5)	0.23(2)	0.070(8)	0.053(6)	–0.038(9)	0.028(8)	–0.021(6)

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