Crystal structure of dichloro((*N*-(*N*′,*N*′-dimethylammonio)ethyl)-2,4-dichlorosalicylaldiminato-*N*,*O*)copper(II), $Cu(C_{11}H_{14}Cl_2N_2O)Cl_2$

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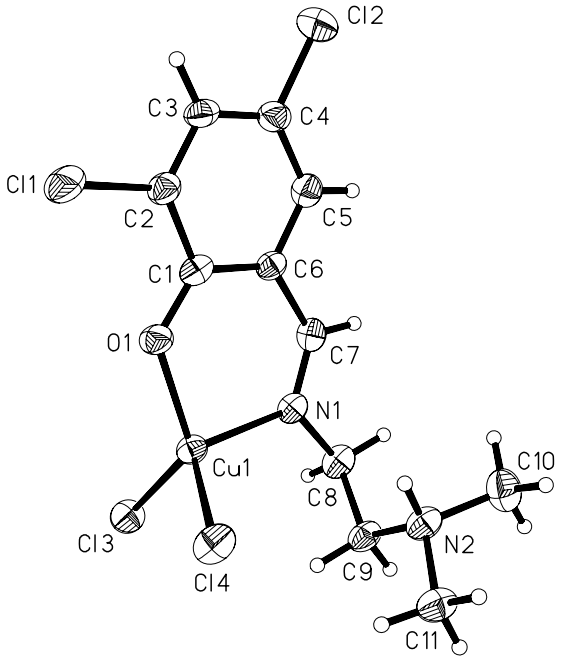

Abstract

$C_{11}H_{14}Cl_4CuN_2O$, orthorhombic, $P2_12_12_1$ (no. 19), a = 7.737(2) Å, b = 10.131(2) Å, c = 20.229(3) Å, V = 1585.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.040, $wR_{ref}(F^2)$ = 0.079, T = 298 K.

Source of material

Reagents and solvents used were of commercially available quality. *N*,*N*-Dimethylethane-1,2-diamine (44.3 mg, 0.5 mmol), 3,5-dichloro-2-hydroxybenzaldehyde (95.3 mg, 0.5 mmol), and $CuCl_2 \cdot 2H_2O$ (85.2 mg, 0.5 mmol) were stirred and refluxed in ethanol at about 351 K for 1 h. Then the mixture was cooled to room temperature and filtered. Prismatic crystals suitable for X-ray structural determination were formed by slow evaporation of the solvent from the filtrate after about a week.
Elemental analysis: found – C, 33.23 %; H, 3.45 %; N, 7.17 %; calc. for $C_{11}H_{14}Cl_4CuN_2O$ – C, 33.40 %; H, 3.57 %; N, 7.08 %.

Experimental details

Atom H2 was located in a Fourier difference map and refined isotropically, with the N—H distance restrained to 0.90(1) Å. All other H atoms were positioned geometrically and constrained as riding atoms, with C—H distances of 0.93 Å – 0.97 Å and U_{iso}(H) set to 1.2 or 1.5U_{eq}(C) of the parent atom.

Discussion

Metal complexes of Schiff bases have been extensively studied, and copper(II) complexes play a major role in both synthetic and structural research [1-3]. Schiff base metal complexes are one of the most important stereochemical models in transition metal coordination chemistry with their easy preparation and structural variation [4-6]. As an extension of our work on the structural characterization of Schiff base complexes [7], a new mononuclear copper(II) complex is reported.
The Cu^{II} ion in the title complex is four-coordinated by one O and one imine N atom of the Schiff base, and by two terminal Cl atoms, giving a tetrahedral environment. All the bond lengths related to the Cu center are comparable to the corresponding values observed in other Schiff base copper(II) complexes [8-10].

Table 1. Data collection and handling.

Crystal:	blue, prismatic, size 0.10 × 0.11 × 0.17 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	20.43 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	56.54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13798, 3759
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2771
$N(param)_{refined}$:	178
Program:	SHELXTL [11]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*a*	0.736(5)	0.607(3)	–0.046(1)	0.06(1)
H(3)	4*a*	0.6688	0.8053	0.3096	0.057
H(5)	4*a*	0.4687	0.5389	0.1872	0.061
H(7)	4*a*	0.6131	0.4494	0.1010	0.052
H(8A)	4*a*	0.6839	0.3554	0.0148	0.058
H(8B)	4*a*	0.8739	0.3049	0.0245	0.058
H(9A)	4*a*	0.9705	0.4575	–0.0539	0.058
H(9B)	4*a*	0.8381	0.3580	–0.0848	0.058
H(10A)	4*a*	0.4946	0.5799	–0.1046	0.105
H(10B)	4*a*	0.5069	0.4788	–0.0461	0.105
H(10C)	4*a*	0.5517	0.4340	–0.1183	0.105
H(11A)	4*a*	0.9318	0.6348	–0.1272	0.088
H(11B)	4*a*	0.7470	0.6789	–0.1500	0.088
H(11C)	4*a*	0.8202	0.5413	–0.1718	0.088

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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}
Cu(1)	4*a*	1.06242(6)	0.57655(4)	0.07093(2)	0.0459(3)	0.0319(2)	0.0387(2)	–0.0014(2)	0.0006(2)	–0.0048(2)
Cl(1)	4*a*	0.9920(2)	0.8593(1)	0.26030(6)	0.0772(9)	0.0766(8)	0.0673(7)	–0.0171(7)	0.0056(6)	–0.0358(6)
Cl(2)	4*a*	0.3576(2)	0.6681(2)	0.30139(7)	0.0594(8)	0.124(1)	0.0735(8)	0.0108(8)	0.0161(7)	–0.0118(8)
Cl(3)	4*a*	1.2623(1)	0.41617(9)	0.06926(5)	0.0602(6)	0.0486(5)	0.0564(6)	0.0121(5)	–0.0016(5)	–0.0021(6)
Cl(4)	4*a*	1.1218(2)	0.7142(1)	–0.01311(5)	0.0830(9)	0.0463(6)	0.0509(6)	–0.0154(6)	–0.0011(6)	0.0048(5)
O(1)	4*a*	1.0028(3)	0.6759(2)	0.1491(1)	0.050(2)	0.046(2)	0.046(2)	–0.010(1)	0.007(1)	–0.013(1)
N(1)	4*a*	0.8325(4)	0.4823(3)	0.0647(2)	0.051(2)	0.028(2)	0.040(2)	–0.002(1)	–0.003(2)	–0.003(1)
N(2)	4*a*	0.7407(5)	0.5435(3)	–0.0766(2)	0.067(2)	0.036(2)	0.043(2)	0.002(2)	–0.002(2)	–0.007(2)
C(1)	4*a*	0.8567(6)	0.6721(4)	0.1799(2)	0.058(3)	0.030(2)	0.034(2)	0.004(2)	–0.002(2)	0.002(2)
C(2)	4*a*	0.8273(5)	0.7535(4)	0.2363(2)	0.053(3)	0.042(2)	0.044(2)	0.005(2)	–0.003(2)	–0.007(2)
C(3)	4*a*	0.6793(5)	0.7519(4)	0.2724(2)	0.059(3)	0.049(2)	0.036(2)	0.017(2)	0.001(2)	–0.004(2)
C(4)	4*a*	0.5450(5)	0.6709(4)	0.2539(2)	0.051(3)	0.062(3)	0.041(2)	0.012(2)	0.002(2)	0.003(2)
C(5)	4*a*	0.5608(5)	0.5924(4)	0.1996(2)	0.052(2)	0.053(2)	0.047(2)	–0.002(2)	–0.004(2)	0.002(2)
C(6)	4*a*	0.7131(5)	0.5908(3)	0.1622(2)	0.048(2)	0.036(2)	0.034(2)	0.002(2)	–0.002(2)	0.001(2)
C(7)	4*a*	0.7123(5)	0.5000(3)	0.1066(2)	0.047(2)	0.033(2)	0.049(2)	–0.008(2)	–0.005(2)	0.002(2)
C(8)	4*a*	0.8044(6)	0.3819(3)	0.0142(2)	0.066(3)	0.028(2)	0.050(2)	–0.002(2)	–0.006(2)	–0.005(2)
C(9)	4*a*	0.8502(5)	0.4303(4)	–0.0537(2)	0.065(3)	0.041(2)	0.038(2)	0.010(2)	–0.003(2)	–0.010(2)
C(10)	4*a*	0.5572(6)	0.5057(5)	–0.0873(2)	0.059(3)	0.068(3)	0.082(3)	0.001(3)	–0.009(3)	0.012(3)
C(11)	4*a*	0.8166(6)	0.6051(4)	–0.1367(2)	0.073(3)	0.057(3)	0.046(2)	0.005(2)	0.006(2)	0.003(2)

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