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Crystal structure of (6,6'-((ethane-1,2-diylbis(azanylylidene))bis(methanylylidene))bis(2-chlorophenolato- $\kappa^2 N,O$))copper(II), $C_{16}H_{12}Cl_2CuN_2O_2$

Table 1: Data collection and handling.

Crystal:	Black block
Size:	0.16 × 0.12 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.78 mm ⁻¹
Diffractometer, scan mode:	Multiwire proportional, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11780, 2695, 0.053
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2003
$N(\text{param})_{\text{refined}}$:	208
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3], Diamond [4]

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Abstract

$C_{16}H_{12}Cl_2CuN_2O_2$, orthorhombic, *Pbca* (no. 61), $a = 12.8449(5)$ Å, $b = 12.5266(6)$ Å, $c = 19.0540(11)$ Å, $V = 3065.8(3)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0423$, $wR_{\text{ref}}(F^2) = 0.1164$, $T = 173(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The halogenated tetradentate Schiff base ligand (3Cl-salen) was prepared by mixing 3-chlorosalicylaldehyd (0.147 g, 1.0 mmol) and ethylenediamine (0.03 g, 0.5 mmol) in 30 mL of methanol. Then, $Cu(NO_3)_2 \cdot 3H_2O$ (0.100 g, 0.41 mmol) was

added to above yellow solution, the resulting dark red suspension was heated for another 2 h. After cooling, the brownish red filtrate was sealed in a beaker and kept undisturbed at room temperature. The red block crystals of title compound were afforded after one week.

Experimental details

Hydrogen atoms were placed in calculated positions and included in the refinement in the riding model approximation, with $U_{\text{iso}}(H)$ set to $1.2U_{\text{eq}}(C)$. The highest peak (0.56 e Å⁻³) and the deepest hole (−0.39 e Å⁻³) in the difference Fourier map are located 1.13 Å and 0.83 Å from Cu1, respectively.

Comment

Copper(II) containing complexes have received much attention owing to their superior biological and catalytic properties. In the last several years, related research is becoming one of the most developing fields in coordination polymers [5, 6].

With the aim of exploring the relationship between the constitution of Schiff-base ligands and the complex structures, we have engaged in the research of isolating Cu complexes with different halogenated Schiff base. In this paper, we present one new copper coordination complex based on Cu(II) and 3Cl-salenH₂. The topology of this class of ligands enables a coordination to transition metals like Cu(II) [7], but also other metal ions are possible [8, 9], and are well known for decades [10].

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cu1	0.12642(3)	0.47053(3)	0.49464(3)	0.0370(2)
Cl2	0.09318(8)	0.47984(8)	0.24615(6)	0.0553(3)
Cl1	−0.06125(10)	0.16275(9)	0.41990(7)	0.0708(4)
O2	0.13738(18)	0.48691(19)	0.39622(15)	0.0402(7)
N1	0.1133(2)	0.4639(2)	0.59533(19)	0.0407(8)
O1	0.0393(2)	0.3489(2)	0.48131(15)	0.0467(7)
N2	0.2230(2)	0.5860(2)	0.51383(18)	0.0407(8)
C16	0.1850(3)	0.5625(3)	0.3621(2)	0.0390(9)
C11	0.2484(3)	0.6429(3)	0.3936(2)	0.0395(9)
C1	0.0080(3)	0.2815(3)	0.5296(2)	0.0405(10)
C6	0.0209(3)	0.2947(3)	0.6026(2)	0.0401(9)
C7	0.0701(3)	0.3888(3)	0.6310(2)	0.0453(10)
H7	0.0706	0.3960	0.6806	0.054*
C10	0.2626(3)	0.6489(3)	0.4683(2)	0.0411(10)
H10	0.3054	0.7050	0.4854	0.049*
C15	0.1747(3)	0.5702(3)	0.2883(2)	0.0414(10)
C12	0.2956(3)	0.7221(3)	0.3520(3)	0.0524(11)
H12	0.3369	0.7754	0.3740	0.063*
C13	0.2834(4)	0.7243(3)	0.2809(3)	0.0618(13)
H13	0.3171	0.7775	0.2535	0.074*
C2	−0.0438(3)	0.1866(3)	0.5083(2)	0.0470(11)
C9	0.2469(3)	0.5974(3)	0.5889(2)	0.0518(11)
H9A	0.2620	0.6730	0.6001	0.062*
H9B	0.3087	0.5540	0.6012	0.062*
C4	−0.0680(3)	0.1291(4)	0.6267(3)	0.0639(14)
H4	−0.0941	0.0781	0.6592	0.077*
C3	−0.0797(3)	0.1136(4)	0.5569(3)	0.0618(14)
H3	−0.1134	0.0507	0.5409	0.074*
C8	0.1533(3)	0.5597(3)	0.6295(2)	0.0514(11)
H8A	0.1732	0.5435	0.6785	0.062*
H8B	0.0992	0.6160	0.6301	0.062*
C14	0.2211(3)	0.6479(3)	0.2492(2)	0.0546(11)
H14	0.2106	0.6497	0.1998	0.066*
C5	−0.0181(3)	0.2195(3)	0.6504(3)	0.0532(11)
H5	−0.0100	0.2309	0.6994	0.064*

The title compound crystallizes in the orthorhombic space group *Pbca*, which is constructed by one crystallographically unique Cu(II) and one chlorinated Schiff-base ligand 3*Cl*-salen. As shown in the figure, the central Cu atom exhibit a tetra-coordinated geometry, which is defined by N₂O₂ in the equatorial plane from 3*Cl*-salen ligand. The bond lengths of Cu(1)-N(1), Cu(1)-N(2), Cu(1)-O(1) and Cu(1)-O(1) are 1.928(4), 1.940(3), 1.908(3) and 1.892(3) Å, respectively. The packing of neighbouring molecules is supported

by weak C—H···Cl hydrogen bonds and face-to-face π - π stacking interactions.

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