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Crystal structure of [6,6'-(((2,2-dimethylpropane-1,3-diyl)bis(azanylylidene))bis(methanylylidene))bis(2-chlorophenolato)- κ^4N,N',O,O']copper(II)

Table 1: Data collection and handling.

Crystal:	Block, clear greenish black
Size	0.20 × 0.14 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.47 mm ⁻¹
Diffractometer, scan mode:	Xcalibur, ϕ and ω -scans
$\theta_{\max}$, completeness:	26.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, Rint:	R _{int} : 8248, 1902, 0.100
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1526
$N(\text{param})_{\text{refined}}$:	120
Programs:	CrysAlis ^{PRO} [1], OLEX2 [2], SHELX [3,4]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cu1	0.000000	0.01438 (6)	0.250000	0.0261 (3)
Cl1	0.13532 (5)	-0.32244 (10)	0.27252 (16)	0.0388 (3)
O1	0.06290 (13)	-0.1035 (3)	0.2386 (4)	0.0297 (7)
N1	0.04804 (16)	0.1346 (3)	0.1521 (4)	0.0293 (8)
C1	0.1442 (2)	0.0173 (4)	0.1451 (5)	0.0275 (9)
C2	0.11978 (19)	-0.0901 (4)	0.1992 (5)	0.0274 (9)
C3	0.1624 (2)	-0.1881 (4)	0.2091 (5)	0.0305 (10)
C4	0.2242 (2)	-0.1796 (4)	0.1709 (6)	0.0347 (11)
H4	0.251430	-0.246814	0.181136	0.042*
C5	0.2461 (2)	-0.0744 (5)	0.1184 (6)	0.0394 (12)
H5	0.288190	-0.069007	0.091237	0.047*
C6	0.2068 (2)	0.0227 (5)	0.1055 (6)	0.0368 (11)
H6	0.222149	0.095143	0.069167	0.044*
C7	0.1052 (2)	0.1211 (4)	0.1174 (5)	0.0293 (9)
H7	0.123246	0.186522	0.068779	0.035*
C8	0.0122 (2)	0.2433 (4)	0.0982 (5)	0.0280 (9)
H8A	-0.030023	0.222788	0.026076	0.034*
H8B	0.036864	0.290208	0.027509	0.034*
C9	0.000000	0.3188 (6)	0.250000	0.0314 (14)
C10	-0.0596 (3)	0.3944 (5)	0.1857 (7)	0.0518 (14)
H10A	-0.052208	0.441473	0.088005	0.078*
H10B	-0.067401	0.446689	0.277602	0.078*
H10C	-0.097360	0.343683	0.150312	0.078*

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Abstract

C₁₉H₁₈Cl₂CuN₂O₂, monoclinic, space group *C2/c* (no. 15), $a = 21.010(3)$ Å, $b = 11.3886(15)$ Å, $c = 7.9578(10)$ Å, $\beta = 100.725(4)^\circ$, $V = 1870.8(4)$ Å³, $Z = 4$, $wR_2 = 0.1620$, $T = 170.0$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The structure was solved with the OLEX2 program [2] as an interface together with the SHELXT and SHELXL

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programs [3, 4]. All H atoms were placed in geometrically idealized positions and refined using a riding model, with C–H = 0.98 (methylene), 0.95 Å (benzene), and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ for H atoms.

Experimental details

3,5-Dibromosalicylaldehyde (0.0279 g, 0.10 mmol) was dissolved in methanol (30 mL) with stirring and 1,3-diaminopropane (0.05 mmol) was added. The mixture was stirred and refluxed at 80 °C for 3 h. The solvent was removed by a rotary evaporator to give a yellow powder which was dissolved in a mixture containing 20 mL methanol and 10 mL ethanol. Then, 0.1 g copper nitrate was added to the above solution and the resulting dark green mixture was further stirred at room temperature overnight. Dark greenish crystals of the title compound were isolated after two weeks.

Comment

Schiff base is an excellent ligand for 3d and 4f metal ion owing to its characteristic $\text{C}=\text{N}-$ group, in which the N atom has a lone pair of electrons, creating an electron-rich chemical environment [5, 6]. Meanwhile, the introduction of electron-rich atoms such as O, N and S into the Schiff base can create more favorable electron-rich chemical environment for coordination. As a part of our current research interest on the exploration of the regulating effect of Schiff base complexes [7, 8], we report herein a new complex based.

The asymmetric unit of the title structure contains one half of Cu(II) complex. As is shown in Figure, the central Cu(II) exhibits a tetra-coordinated environment which is defined by N_2O_2 . The bond lengths of Cu–O(N) are in the range of 1.898(3)–1.948(4) Å which is compared favorably with the corresponding values observed in salen-type Cu(II) analogues [9, 10].

The dihedral angle calculated between the planes of two benzene rings is 38.135(16)°. Owing to the presence of $\pi-\pi$ interactions (the distance between $\text{Cg1}\cdots\text{Cg1}^i$ are 3.603(2)), where Cg1 is the centroid of the C1–C6 ring). Two neighboring complexes are further linked to each other to form a self-assembled supramolecular dimer.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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