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Crystal structure of {2,2'-{cyclohexane-1,2-diylbis[(azanylylidene)methylylidene]}bis(2,4-dibromophenolato)- $\kappa^4 N, N', O, O'$ }copper(II) – diethylformamide (1/1), $C_{23}H_{23}Br_4CuN_3O_3$

Table 1: Data collection and handling.

Crystal:	Block, clear blackish black
Size:	0.16 × 0.14 × 0.12 mm
Wavelength:	MoK α radiation (0.71073 Å)
μ :	7.18 mm ⁻¹
Diffractometer, scan mode:	Agilent CCD detector, φ and ω -scans
$\theta_{\max}$, completeness:	26.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8469, 5230, 0.022
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4520
$N(\text{param})_{\text{refined}}$:	309
Programs:	CrysAlis ^{PRO} [1], OLEX2 [2], SHELX [3, 4]

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Abstract

$C_{23}H_{23}Br_4CuN_3O_3$, $P\bar{1}$ (no. 2), $a = 10.4769(4)$ Å, $b = 10.7829(5)$ Å, $c = 12.9824(5)$ Å, $\alpha = 98.153(2)^\circ$, $\beta = 111.484(2)^\circ$, $\gamma = 105.035(2)^\circ$, $V = 1271.43(9)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0338$, $wR_{\text{ref}}(F^2) = 0.1158$, $T = 273.15$ 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 and refined with the OLEX2 program [2] as an interface together with the SHELXT and SHELXL

programs [3, 4]. All H atoms were placed in geometrically idealized positions and refined using a riding model.

Experimental details

3,5-Dibromosalicylaldehyde (0.0263 g, 0.094 mmol) was dissolved in methanol (30 mL) with stirring, adding 1,2-cyclohexanediamine (0.047 mmol). The mixture was stirred and refluxed at 80 °C for 3 h. The solvent was removed using a rotary evaporator to give the Schiff-base ligand (+-)-3,5-BrsalcyH₂. After this (+-)-3,5-BrsalcyH₂ (0.1 mmol) was dissolved in a mixture containing 20 mL *N,N*-dimethylformamide (DMF) 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 brown crystals of the title compound were isolated after two weeks.

Comment

Schiff bases have a variety of chemical and biological activities [5, 6]. The research concerning halogenated salen-type Cu-complexes is still at an early stage [7–10]. As a part of our current research interest on the regulating effect of Schiff base ligands on transition metal complexes, we report herein a new Cu(II) complex.

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Br2	0.63313 (5)	0.23839 (5)	0.73782 (4)	0.01910 (13)
Br1	0.96491 (6)	0.70727 (5)	1.09894 (4)	0.02541 (14)
Br4	0.18198 (6)	0.01430 (5)	0.34252 (5)	0.02474 (14)
Cu1	0.34896 (6)	0.48150 (6)	0.52712 (5)	0.01562 (15)
Br3	−0.28775 (7)	0.07518 (6)	−0.01305 (5)	0.04215 (19)
O2	0.2553 (4)	0.3058 (3)	0.4296 (3)	0.0197 (7)
O1	0.4954 (4)	0.4229 (3)	0.6236 (3)	0.0187 (7)
N1	0.4484 (4)	0.6665 (4)	0.6160 (3)	0.0163 (8)
N2	0.2062 (5)	0.5473 (4)	0.4296 (4)	0.0178 (9)
C3	0.7808 (6)	0.4841 (5)	0.9077 (4)	0.0189 (10)
H3	0.8319	0.4369	0.9510	0.023*
C13	0.2303 (5)	0.6883 (4)	0.4780 (4)	0.0143 (9)
H13	0.1973	0.7297	0.4152	0.017*
C9	0.4276 (6)	0.8976 (5)	0.6178 (5)	0.0212 (11)
H9A	0.4057	0.9526	0.5652	0.025*
H9B	0.5307	0.9354	0.6685	0.025*
C4	0.8110 (5)	0.6180 (5)	0.9512 (4)	0.0180 (10)
C1	0.5909 (6)	0.4879 (5)	0.7264 (4)	0.0175 (10)
N3	0.2973 (6)	0.3072 (5)	0.7716 (5)	0.0318 (11)
C20	0.1406 (5)	0.2638 (5)	0.3338 (4)	0.0161 (9)
C7	0.5559 (6)	0.7073 (5)	0.7140 (4)	0.0185 (10)
H7	0.5935	0.7983	0.7484	0.022*
C8	0.3952 (5)	0.7578 (5)	0.5499 (4)	0.0153 (9)
H8	0.4406	0.7662	0.4961	0.018*
C19	0.0851 (5)	0.1270 (5)	0.2726 (4)	0.0154 (9)
C2	0.6729 (5)	0.4218 (5)	0.7982 (4)	0.0165 (10)
C15	0.0592 (5)	0.3421 (5)	0.2807 (4)	0.0155 (9)
C6	0.6252 (5)	0.6240 (4)	0.7768 (4)	0.0139 (9)
C14	0.0969 (5)	0.4817 (5)	0.3332 (4)	0.0167 (10)
H14	0.0384	0.5271	0.2949	0.020*
C18	−0.0358 (6)	0.0709 (5)	0.1701 (4)	0.0196 (10)
H18	−0.0662	−0.0188	0.1330	0.023*
O3	0.2322 (6)	0.4707 (5)	0.6960 (6)	0.0577 (15)
C10	0.3399 (6)	0.9002 (5)	0.6886 (5)	0.0223 (11)
H10A	0.3660	0.8503	0.7449	0.027*
H10B	0.3625	0.9914	0.7293	0.027*
C5	0.7374 (5)	0.6894 (5)	0.8889 (4)	0.0178 (10)
H5	0.7606	0.7799	0.9194	0.021*
C17	−0.1118 (6)	0.1533 (5)	0.1234 (4)	0.0244 (11)
C12	0.1428 (6)	0.6958 (5)	0.5493 (5)	0.0233 (11)
H12A	0.0394	0.6575	0.4994	0.028*
H12B	0.1655	0.6445	0.6050	0.028*
C11	0.1789 (6)	0.8403 (5)	0.6115 (5)	0.0221 (11)
H11A	0.1524	0.8910	0.5558	0.026*
H11B	0.1235	0.8438	0.6568	0.026*
C16	−0.0666 (6)	0.2866 (6)	0.1770 (4)	0.0226 (11)
H16	−0.1192	0.3394	0.1447	0.027*
C24	0.3728 (8)	0.2668 (7)	0.8723 (6)	0.0367 (15)
H24A	0.3045	0.1961	0.8821	0.055*
H24B	0.4166	0.3412	0.9388	0.055*
H24C	0.4471	0.2365	0.8626	0.055*
C23	0.2168 (7)	0.2056 (7)	0.6616 (6)	0.0412 (16)
H23A	0.2843	0.1784	0.6379	0.062*
H23B	0.1608	0.2417	0.6049	0.062*
H23C	0.1526	0.1301	0.6701	0.062*

Table 2: (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C21	0.2945 (8)	0.4308 (7)	0.7783 (7)	0.0433 (17)
H21	0.3429	0.4913	0.8503	0.052*

The basic molecular structure of the title compound shows a classic Cu-salen complex configuration, in which the copper center possesses tetra-coordinated environment. Neighbouring monomers are further linked by weak inter-molecular halogen-bonding interactions: Br1⋯Br4ⁱ, D⋯A: 3.740(2) Å; Br2⋯Br3ⁱⁱ, D⋯A: 3.740(2) Å, symmetry code: (i): symmetry code: (i): (1 + x, 1 + y, 1 + z). Another interesting feature of the title compound is the DMF solvent molecules located between Cu-salen molecules contributing the stability of the supramolecular networks.

According to the result of PLATON software [19], the classic π - π stacking interactions is another important driving force in linking the adjacency molecules in to supermolecular networks.

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

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