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Crystal structure of bis(μ_2 -azido- k^2N,N)-bis(2-amino-1-(N -(3-bromosalicylaldiminato))ethane)-dicopper(II), $C_{20}H_{18}Br_4N_2O_2$

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.12 × 0.11 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	5.21 mm ⁻¹
Diffractometer, scan mode:	CCD, φ and ω
$\theta_{\max}$, completeness:	25.0°, 98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4023, 2046, 0.035
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1436
$N(\text{param})_{\text{refined}}$:	154
Programs:	CrysAlis ^{PRO} [1], SHELX [2], Olex2 [3], Diamond [4]

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Abstract

$C_{20}H_{18}Br_4N_2O_2$, monoclinic, $P2_1/c$ (no. 14), $a = 9.5994(7)$ Å, $b = 11.9156(10)$ Å, $c = 10.3508(7)$ Å, $\beta = 91.025(6)^\circ$, $V = 1183.76(15)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0472$, $wR_{\text{ref}}(F^2) = 0.1008$, $T = 293(2)$ 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

3-Bromo-5-chlorosalicylaldehyde 0.471 g (2.0 mmol) and 0.23 mL ethylenediamine (2.0 mmol) were stirred in a mixture of 30 mL ethanol and 10 mL methanol at room temperature for 20 min. Then, 0.241 g $Cu(NO_3)_2 \cdot H_2O$ (1.0 mmol) and 0.065 g NaN_3 (1.0 mmol) was added to the above solution and the resulting yellow mixture was further stirred for another

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.29015(7)	0.96575(6)	0.12269(5)	0.0590(3)
Cu1	0.14869(7)	0.99948(6)	0.56851(6)	0.0370(2)
O1	0.2340(4)	1.0027(3)	0.4032(3)	0.0384(9)
N1	0.2772(4)	1.1066(4)	0.6440(4)	0.0404(12)
N2	0.0558(5)	1.0074(4)	0.7396(4)	0.0408(12)
H2A	−0.0374	1.0069	0.7283	0.049*
H2B	0.0802	0.9478	0.7883	0.049*
N3	0.0353(4)	0.8639(4)	0.5195(4)	0.0410(12)
N4	0.0826(5)	0.7854(5)	0.4682(4)	0.0422(12)
N5	0.1217(6)	0.7046(5)	0.4170(5)	0.0664(16)
C1	0.3478(5)	1.0562(5)	0.3727(5)	0.0357(13)
C2	0.3974(6)	1.0497(5)	0.2458(5)	0.0450(15)
C3	0.5193(6)	1.1000(5)	0.2062(6)	0.0501(16)
H3	0.5479	1.0925	0.1213	0.060*
C4	0.5988(6)	1.1618(5)	0.2944(6)	0.0572(18)
H4	0.6816	1.1955	0.2698	0.069*
C5	0.5521(6)	1.1722(5)	0.4193(6)	0.0509(16)
H5	0.6049	1.2136	0.4785	0.061*
C6	0.4293(5)	1.1230(5)	0.4597(5)	0.0385(13)
C7	0.3875(6)	1.1414(5)	0.5914(5)	0.0466(15)
H7	0.4481	1.1832	0.6434	0.056*
C8	0.2507(6)	1.1310(6)	0.7814(5)	0.0549(17)
H8A	0.2749	1.2082	0.8012	0.066*
H8B	0.3067	1.0822	0.8365	0.066*
C9	0.0998(7)	1.1116(6)	0.8040(5)	0.0574(18)
H9A	0.0836	1.1060	0.8960	0.069*
H9B	0.0458	1.1742	0.7702	0.069*

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30 min and filtered. The black-blue crystals of the title compound were obtained after 5 days by slow evaporation.

Experimental details

All hydrogen atoms were placed in calculated positions using a riding model. Aromatic C—H distances were set to 0.93 Å and their U_{iso} set to 1.2 times the U_{eq} of the parent atom.

Comment

Coordination chemistry is a multidisciplinary research field that ranges from crystal engineering to materials science. As a part of current research interest on the exploration of the regulating effect of Schiff base ligands on transition metal complexes [5–7], we report herein a new copper dinuclear complex based on a halogenated Schiff base ligand.

The title compound crystallizes in monoclinic space group $P2_1/c$. The asymmetric of the title structure contains a mononuclear copper(II) center coordinated to four donor atoms in the equatorial plane, three of which from the ‘half-salen’ tridentate Schiff base ligand and one from azide anion. The coordination is completed by another N atom from the other azide anion at axial position, and resulting in an overall centrosymmetry out-of-plane dimer structure (see the figure). The bond length of bridging Cu—N³ⁱ is 2.556(2) Å, and the bond angle of N(3)—Cu(1)—N(3)ⁱ is 93.036(3) (symmetry code: (i): $x, 2 - y, 1 - z$). All other Cu—O/N bond lengths are in the expected ranges (Cu1—O1 = 1.910(3) Å; Cu1—N1 = 1.931(4) Å; Cu1—N2 = 1.999(4) Å; Cu1—N3 = 2.008(5) Å) [8, 9].

It should be mention that bromine atoms form N⋯Br halogen bonding lay a key role in stabilizing the stacking arrangement. (Interactions: (N(5)⋯Br(1)ⁱⁱ = 3.337(2) Å, symmetry code (ii): $x, 1.5 - y, 0.5 + z$).

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