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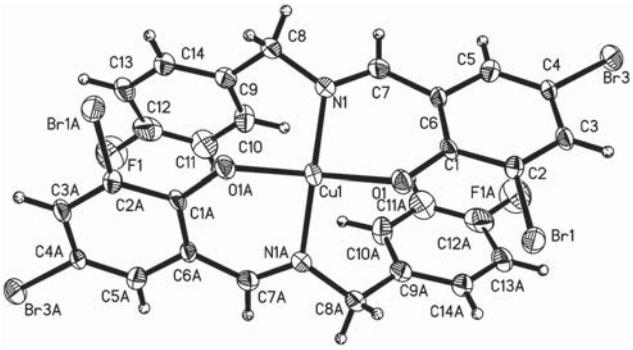
Crystal structure of bis(2,4-dibromo-6-{(*E*)[(4-fluorobenzyl)imino]methyl}phenolato-κ^2*N*,*O*) copper(II), $C_{28}H_{18}Br_4F_2N_2O_2Cu$



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Abstract

$C_{28}H_{18}Br_4CuF_2N_2O_2$, monoclinic, *C*2 (no. 5), $a = 14.6518(8)$ Å, $b = 9.6738(5)$ Å, $c = 11.1907(6)$ Å, $\beta = 121.182(8)°$, $V = 1357.00(17)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0632$, $wR_{ref}(F^2) = 0.0762$, $T = 293$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

3,5-dibromo-2-hydroxybenzaldehyde 2.80 g (10 mmol) and (4-fluorobenzyl)methanamine 1.25 g (10 mmol) were dissolved in ethanol and stirred for 2 h at a temperature of 65 °C. After evaporation, a crude product was recrystallized twice from ethanol solution to give a pure yellow product. [2-((E)-(4-fluorobenzylimino)methyl)-4,6-dibromophenol 3.157 g (8.2 mmol)]. 1 mmol (0.3851 g) of this product and 0.5 mmol (0.1221 g) $Cu_2NO_3 \cdot 3H_2O$ were dissolved in ethanol and stirred for 10 minutes. Evaporation at room temperature yielded blue crystals of $C_{28}H_{18}Br_4CuF_2N_2O_2$.

Table 1: Data collection and handling.

Crystal:	Blue, block, size 0.12×0.21×0.33 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	67.38 cm^{-1}
Diffractometer, scan mode:	SuperNova, Eos, ω scans
$2\theta_{max}$:	50.48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6394, 2469
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1994
$N(param)_{refined}$:	177
Programs:	CrysAlis^{PRO} [6], SHELX [7], DIAMOND [8]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(3)	4*c*	0.4884	0.3411	0.8425	0.043
H(5)	4*c*	0.3194	0.1301	0.4876	0.048
H(7)	4*c*	0.1369	0.1726	0.3340	0.051
H(8A)	4*c*	−0.0198	0.1986	0.1670	0.061
H(8B)	4*c*	−0.0836	0.3245	0.1745	0.061
H(10)	4*c*	−0.0445	0.0222	0.3700	0.065
H(11)	4*c*	−0.1824	−0.1238	0.3462	0.078
H(13)	4*c*	−0.3917	0.0809	0.0006	0.065
H(14)	4*c*	−0.2562	0.2298	0.0315	0.051

Experimental details

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms (C—H = 0.93 Å, 0.97 Å). The U_{iso} values of the hydrogen atoms were set to $1.2U_{eq}$(C).

Discussion

Schiff base complexes, due to the novel structure and many potential applications, have been widely studied for many

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Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cu(1)	2*b*	0	0.3384(2)	0.5	0.023(1)	0.056(1)	0.036(1)	0.0	0.0149(8)	0.0
Br(1)	4*c*	0.3350(1)	0.4994(1)	0.8793(1)	0.0361(7)	0.0625(9)	0.0434(7)	0.0032(7)	0.0175(5)	−0.0149(6)
Br(3)	4*c*	0.5416(1)	0.1366(2)	0.6959(1)	0.0339(7)	0.070(1)	0.0579(8)	0.0161(7)	0.0255(6)	−0.0012(7)
F(1)	4*c*	−0.375(1)	−0.114(2)	0.160(1)	0.083(8)	0.12(1)	0.113(9)	−0.053(8)	0.049(7)	0.001(8)
O(1)	4*c*	0.1422(6)	0.3894(9)	0.6250(8)	0.029(4)	0.042(5)	0.043(4)	0.004(4)	0.020(4)	−0.003(4)
N(1)	4*c*	0.0345(8)	0.263(1)	0.362(1)	0.032(5)	0.055(7)	0.035(5)	−0.002(5)	0.019(4)	0.002(5)
C(1)	4*c*	0.2278(9)	0.341(1)	0.632(1)	0.020(5)	0.044(7)	0.036(6)	0.010(5)	0.017(5)	0.006(5)
C(2)	4*c*	0.3286(9)	0.372(1)	0.746(1)	0.026(6)	0.045(8)	0.032(6)	0.005(5)	0.011(5)	0.004(5)
C(3)	4*c*	0.4227(9)	0.317(2)	0.765(1)	0.022(5)	0.044(7)	0.043(6)	0.000(5)	0.018(5)	−0.005(6)
C(4)	4*c*	0.4151(9)	0.226(2)	0.665(1)	0.019(5)	0.057(8)	0.033(6)	0.003(5)	0.014(5)	−0.003(5)
C(5)	4*c*	0.3216(9)	0.193(1)	0.552(1)	0.033(7)	0.047(8)	0.035(6)	0.003(6)	0.014(5)	0.001(5)
C(6)	4*c*	0.2240(9)	0.254(1)	0.529(1)	0.021(6)	0.045(7)	0.032(6)	0.000(5)	0.013(5)	−0.009(5)
C(7)	4*c*	0.129(1)	0.224(2)	0.398(1)	0.038(7)	0.057(9)	0.037(6)	−0.005(6)	0.023(6)	−0.011(6)
C(8)	4*c*	−0.051(1)	0.237(2)	0.218(1)	0.032(7)	0.09(1)	0.038(7)	−0.013(8)	0.021(6)	−0.005(7)
C(9)	4*c*	−0.137(1)	0.141(2)	0.202(1)	0.039(7)	0.053(8)	0.042(6)	0.003(7)	0.025(5)	−0.005(6)
C(10)	4*c*	−0.114(1)	0.036(2)	0.297(2)	0.037(7)	0.06(1)	0.059(8)	0.003(7)	0.018(6)	0.006(7)
C(11)	4*c*	−0.196(1)	−0.053(2)	0.283(2)	0.07(1)	0.049(9)	0.08(1)	0.008(8)	0.043(9)	0.018(8)
C(12)	4*c*	−0.296(1)	−0.028(2)	0.173(2)	0.07(1)	0.08(1)	0.07(1)	−0.04(1)	0.047(9)	−0.02(1)
C(13)	4*c*	−0.322(1)	0.071(2)	0.076(2)	0.034(7)	0.08(1)	0.054(8)	−0.015(8)	0.024(6)	−0.009(8)
C(14)	4*c*	−0.240(1)	0.158(2)	0.094(1)	0.036(7)	0.052(9)	0.039(6)	−0.003(6)	0.018(5)	−0.004(6)

years [1–4]. Schiff base and its transition metal complexes have become the hot topic of research [5]. Schiff base ligands are formed by amine and carbonyl compounds via condensation and coordinate by a single tooth to multiple teeth, from symmetrical to asymmetrical, from chain to ring.

Schiff base ligands synthesized from 2-hydroxy-3,5-dibromo benzaldehyde and fluorine benzylamine may form Cu complexes to study their inhibitory effect on jack bean urease.

The title Schiff base complex, $[Cu(C_{14}H_9Br_2F_NO)_2]$, possesses a crystallographic two fold symmetry, with the Cu^{II} atom lying on the rotation axis. The coordination of the metal atom by the two N,O-bidentate ligands results in a flattened CuN_2O_2 tetrahedron. The six-membered chelate ring is in an envelope conformation, with the metal atom as the flap. The dihedral angle between the planes of the aromatic rings within each ligand is 61.4(2)°. The Cu1—O1 distance of 1.877(8) Å is shorter than the distance of Cu1—N1 distance [1.997(10) Å]. The bond lengths and bond angles in title complex are within normal ranges.

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