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Synthesis and crystal structure of bis{5-fluorine-2-(((4-(1-(methoxy-imino)ethyl)phenyl) imino)methyl)phenolato- $\kappa^2 N,O$ }copper(II), $C_{32}H_{28}CuF_2N_4O_4$

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Abstract

$C_{32}H_{28}CuF_2N_4O_4$, monoclinic, $P2_1/n$ (no. 14), $a = 10.5489(3)$ Å, $b = 6.2601(1)$ Å, $c = 21.3299(7)$ Å, $\beta = 99.401(1)^\circ$, $V = 1389.65(6)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0312$, $wR_{ref}(F^2) = 0.0849$, $T = 173(2)$ K.

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The crystal 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 Schiff base ligand was prepared by a similar method reported earlier [5]. The copper(II) acetate tetrahydrate (2.5 mg, 10 mmol) dissolved in ethanol solution (4 mL) was added dropwise to the ligand (8.3 mg, 20 mmol) dissolved in chloroform solution (4 mL) at room temperature. The ligand is named 1-[4-[(4-fluoro-2-hydroxy-benzylidene) amino] phenyl]ethanone *O*-methyl oxime. The mixture solution turns to dark brown, immediately. The mixture was kept being stirred for 1 h. The filtrate was allowed to stand for 23 days at quiet environment. Several clear dark brown crystals

Table 1: Data collection and handling.

Crystal:	Clear dark brown, block
Size:	$0.19 \times 0.17 \times 0.14$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.85 mm^{-1}
Diffractometer, scan mode:	Bruker D8 Venture, φ and ω -scans
$\theta_{\max}$, completeness:	26.8° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	23571, 2942, 0.080
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2406
$N(\text{param})_{\text{refined}}$:	198
Programs:	Bruker programs [1], SHELX [2, 3], OLEX2 [4]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	−0.04431(19)	0.8840(3)	0.62336(9)	0.0241(4)
C2	−0.1184(2)	0.9158(3)	0.67199(10)	0.0286(4)
H2	−0.1628	1.0465	0.6750	0.034*
C3	−0.1251(2)	0.7557(4)	0.71458(10)	0.0305(5)
C4	−0.0646(2)	0.5598(4)	0.71372(11)	0.0365(5)
H4	−0.0745	0.4513	0.7437	0.044*
C5	0.0103(2)	0.5297(3)	0.66774(11)	0.0334(5)
H5	0.0545	0.3980	0.6663	0.040*
C6	0.02374(19)	0.6879(3)	0.62253(10)	0.0251(4)
C7	0.11116(19)	0.6489(3)	0.57909(10)	0.0254(4)
H7	0.1631	0.5244	0.5858	0.030*
C8	0.22548(18)	0.7159(3)	0.49583(9)	0.0231(4)
C9	0.24052(19)	0.5119(3)	0.47333(10)	0.0265(4)
H9	0.1866	0.3995	0.4833	0.032*
C10	0.3343(2)	0.4709(3)	0.43618(10)	0.0277(4)
H10	0.3434	0.3303	0.4208	0.033*
C11	0.41483(18)	0.6314(3)	0.42102(9)	0.0243(4)
C12	0.4006(2)	0.8351(3)	0.44580(10)	0.0295(5)
H12	0.4563	0.9468	0.4372	0.035*
C13	0.3076(2)	0.8770(3)	0.48239(10)	0.0292(5)
H13	0.2994	1.0168	0.4986	0.035*
C14	0.51170(19)	0.5911(3)	0.37899(10)	0.0268(4)
C15	0.5164(2)	0.3788(4)	0.34776(12)	0.0416(6)
H15A	0.5693	0.3889	0.3141	0.062*
H15B	0.4292	0.3346	0.3293	0.062*
H15C	0.5539	0.2731	0.3793	0.062*
C16	0.7361(2)	0.8956(4)	0.31841(13)	0.0423(6)
H16A	0.7946	0.8684	0.2880	0.063*
H16B	0.7853	0.9470	0.3585	0.063*

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H16C	0.6730	1.0040	0.3010	0.063*
Cu1	0.0000	1.0000	0.5000	0.02223(11)
F1	−0.19833(13)	0.7879(2)	0.76061(6)	0.0427(3)
N1	0.12534(15)	0.7694(2)	0.53126(8)	0.0233(3)
N2	0.58482(17)	0.7520(3)	0.37133(9)	0.0333(4)
O1	−0.03973(15)	1.0363(2)	0.58198(7)	0.0304(3)
O2	0.67108(15)	0.7026(2)	0.32947(8)	0.0392(4)

were obtained. **Anal. Calcd.** for C₃₂H₂₈CuF₂N₄O₄: C, 60.61%; H, 4.45%; N, 8.84%. Found: C, 61.43%; H, 4.79%; N, 8.24%.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Schiff base containing fluorine substituents is a new class of ligands in metal organic chemistry [6]. Schiff base ligands have excellent coordination ability and can form complexes with various transition metals [7, 8]. So far, we have designed and synthesized a series of bidentate Schiff base ligands [9–11]. Particular attention has been paid to the structural and spectroscopic properties of Schiff base type metal complexes [12–14].

In this paper, we describe a new copper(II) complex. The single crystal structure of the title complex was determined by X-ray crystallography. The complex is located at an inversion center. In the title complex, Cu1 is four-coordinated. The Cu1–N1 bond lengths are both 1.9973(16) and the Cu1–O1 bond lengths are both 1.8774(14). The angles of N1–Cu1–O1 and N1–Cu1–O1ⁱ are 90.74(6) and 89.26(6), respectively. All geometric parameters are in the typical ranges [8–10, 12].

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