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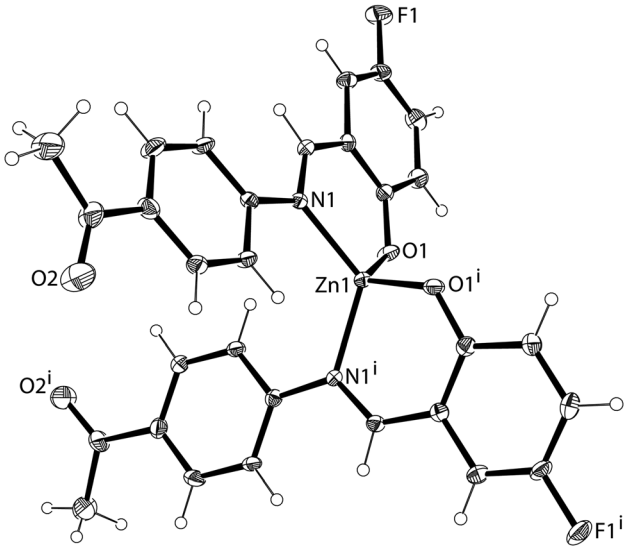
Synthesis and crystal structure of bis{2-(((4-acetophenone)imino)methyl)-4-fluorophenolato-κ^2N,O}zinc(II), $C_{30}H_{22}F_2N_2O_4Zn$



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Abstract

$C_{30}H_{22}F_2N_2O_4Zn$, monoclinic, $P2/c$ (no. 13), a = 9.5936(8) Å, b = 11.2561(10) Å, c = 11.9682(10) Å, β = 104.598(3)°, V = 1250.68(19) Å^3, Z = 2, $R_{gt}(F)$ = 0.0661, $wR_{ref}(F^2)$ = 0.1259, T = 173(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.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.12 × 0.10 × 0.09 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.04 mm^{-1}
Diffractometer, scan mode:	Bruker D8 Venture, φ and ω
θ_{max}, completeness:	26.8°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11,343, 2650, 0.089
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2039
$N(param)_{refined}$:	178
Programs:	Bruker [1], SHELX [2]

1 Source of material

Synthesis of the ligand: Synthesis of the title complex was prepared by a similar method reported earlier [3–5]. The batch size was: An ethanol solution (3 mL) of 5-fluoro-2-hydroxybenzaldehyde (420 mg, 3 mmol) was added dropwise to an ethanol solution (3 mL) of 1-(4-amino-phenyl)ethanone (405 mg, 3 mmol). The mixture was stirred at 238 K for 6 h. We filtered and purified the mixture to obtain the title ligand.

Synthesis of the zinc(II) complex: A methanol solution (2 mL) of the zinc acetate dihydrate (43.9 mg, 0.2 mmol) was added dropwise to a dichloromethane solution (2 mL) of the aforementioned ligand (102.9 mg, 0.4 mmol). The mixture was filtered after being stirred for 1 h at room temperature, and the filtrate was allowed to stand for three weeks at a quiet environment. After partial evaporation of the solvent, several clear light yellow block crystals were obtained.

2 Experimental details

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

3 Comment

Schiff bases have become very popular ligands due to their versatility and ease of preparation [6]. The fluoroaromatic Schiff base ligands and their metal complexes have demonstrated to be important in several applications [7]. They can form stable complexes with alkali metals [8] and transition metals [9, 10] and so on. So far, we have designed and synthesized a variety of bis–Schiff base N_2O_2 type ligands

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.500000	0.50312 (6)	0.750000	0.02094 (19)
F1	−0.0886 (3)	0.3000 (3)	0.3550 (2)	0.0434 (7)
O1	0.3271 (3)	0.4151 (3)	0.7440 (2)	0.0285 (7)
N1	0.4258 (3)	0.5871 (3)	0.5981 (3)	0.0189 (7)
C1	0.2304 (4)	0.3901 (4)	0.6473 (4)	0.0232 (9)
O2	0.8369 (4)	1.0277 (4)	0.5818 (3)	0.0671 (13)
C2	0.2228 (4)	0.4488 (4)	0.5415 (3)	0.0209 (8)
C3	0.1132 (4)	0.4163 (4)	0.4424 (4)	0.0261 (9)
H3	0.107054	0.454527	0.370547	0.031*
C4	0.0172 (4)	0.3301 (4)	0.4510 (4)	0.0297 (10)
C5	0.0218 (5)	0.2712 (4)	0.5522 (4)	0.0339 (11)
H5	−0.046314	0.210773	0.555341	0.041*
C6	0.1270 (5)	0.3015 (4)	0.6491 (4)	0.0313 (10)
H6	0.130238	0.261505	0.719569	0.038*
C7	0.3143 (4)	0.5436 (4)	0.5238 (3)	0.0221 (9)
H7	0.290980	0.578927	0.449336	0.027*
C9	0.4573 (5)	0.7476 (4)	0.4643 (4)	0.0327 (11)
H9	0.372022	0.724272	0.409095	0.039*
C8	0.5011 (4)	0.6866 (3)	0.5678 (3)	0.0204 (8)
C10	0.5377 (5)	0.8426 (4)	0.4411 (4)	0.0389 (12)
H10	0.508269	0.882757	0.369127	0.047*
C11	0.6610 (5)	0.8798 (4)	0.5220 (4)	0.0310 (10)
C12	0.7035 (4)	0.8192 (4)	0.6255 (4)	0.0260 (9)
H12	0.787594	0.843274	0.681641	0.031*
C13	0.6242 (4)	0.7242 (4)	0.6475 (3)	0.0245 (9)
H13	0.654574	0.683478	0.719078	0.029*
C14	0.7488 (5)	0.9832 (4)	0.5013 (4)	0.0420 (12)
C15	0.7259 (7)	1.0315 (5)	0.3818 (5)	0.0618 (18)
H15A	0.739698	0.967965	0.329595	0.093*
H15B	0.795229	1.095389	0.381584	0.093*
H15C	0.627748	1.062833	0.355738	0.093*

and complexes [11, 12]. More attention has been paid to exploring their mechanisms and applications.

In this article, we designed and synthesized a new zinc(II) complex. The single crystal structure of the title complex has been characterized by X-ray crystallography. Zn1 is tetracoordinated and forms a slightly distorted tetrahedral geometry configuration by two O atoms and two N atoms (cf. the Figure). The Zn1–N1 bond lengths are 2.013(3) and 2.014(3) Å respectively, while the Zn1–O1 bond lengths are both 1.918(3) Å. The angles of N1–Zn1–O1 and N1–Zn1–O1^i are 95.93(12)° and 112.45(12)°, respectively. All geometric parameters are similar to those reported in the previous literature [13, 14].

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

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