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Crystal structure of (*E*)-4-((2-fluoro-3-(trifluoromethyl)benzylidene)amino)-3-methyl-1*H*-1,2,4-triazole-5(4*H*)-thione, C₁₁H₈F₄N₄S

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.27 × 0.25 × 0.22 mm
Wavelength:	Cu K α radiation (1.54178 Å)
μ :	2.73 mm ⁻¹
Diffractometer, scan mode:	Bruker X8 Prospector APEX-II,
$\theta_{\max}$, completeness:	67.2°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	19933, 2239, 0.041
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1920
$N(\text{param})_{\text{refined}}$:	182
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

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Abstract

C₁₁H₈F₄N₄S, monoclinic, $P2_1/c$ (no. 14), $a = 4.4773(4)$ Å, $b = 4.1089(11)$ Å, $c = 20.1449(16)$ Å, $\beta = 95.425(4)^\circ$, $V = 1266.85(18)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0383$, $wR_{\text{ref}}(F^2) = 0.1176$, $T = 273(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
F0	0.4372(3)	0.44835(8)	0.25276(6)	0.0739(4)
S1	1.12989(14)	0.49484(4)	0.40955(3)	0.0806(3)
F1	−0.0909(3)	0.46474(11)	0.17394(8)	0.0922(5)
F2	0.2600(3)	0.46174(11)	0.10948(7)	0.0910(5)
F3	−0.1212(3)	0.37171(13)	0.09074(8)	0.1029(5)
N1	1.4424(4)	0.36895(13)	0.48894(9)	0.0706(5)
H1	1.5369	0.4126	0.5122	0.085*
N2	1.4923(4)	0.27423(14)	0.50090(9)	0.0705(5)
N3	1.1452(3)	0.29822(11)	0.41752(7)	0.0550(4)
N4	0.9438(4)	0.26523(12)	0.36596(8)	0.0589(4)
C1	1.2355(4)	0.38815(14)	0.43840(10)	0.0598(5)
C2	1.3105(5)	0.23221(15)	0.45667(10)	0.0619(5)
C3	0.7799(4)	0.32394(15)	0.33195(10)	0.0602(5)
H3	0.7888	0.3881	0.3426	0.072*
C4	0.5765(4)	0.29044(14)	0.27588(9)	0.0548(4)
C5	0.4121(4)	0.35589(14)	0.23676(9)	0.0548(4)
C6	0.2257(4)	0.33152(14)	0.18070(9)	0.0573(5)
C7	0.1981(5)	0.23676(16)	0.16425(11)	0.0719(6)
H7	0.0724	0.2183	0.1271	0.086*
C8	0.3564(6)	0.16922(17)	0.20271(12)	0.0811(7)
H8	0.3363	0.1055	0.1914	0.097*
C9	0.5441(5)	0.19564(15)	0.25783(11)	0.0684(6)
H9	0.6502	0.1495	0.2832	0.082*
C10	1.2794(6)	0.12916(16)	0.44686(13)	0.0832(7)
H10A	1.0763	0.1106	0.4518	0.125*
H10B	1.4126	0.0968	0.4795	0.125*
H10C	1.3290	0.1128	0.4030	0.125*
C11	0.0687(5)	0.40661(17)	0.13883(11)	0.0680(6)

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and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

Preparation: 1 mmol of 2-fluoro-3-(trifluoromethyl) benzaldehyde and 1 mmol 4-amino-3-methyl-1*H*-1,2,4-triazole-5(4*H*)-thione was mixed in 100 mL ethanol; then refluxed for 6 h. The reaction mixture was left to stand for 2 days in an open beaker. When the solvent was almost evaporated a crystalline product appeared, which was filtrated and washed with 100 mL of diethyl ether.

Experimental details

Hydrogen atoms were placed at the calculated positions and refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ or $1.5U_{\text{eq}}$ of the adjacent non-hydrogen atom. H3 Atom position was localized from the difference map and refined with DFIX 0.87 and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O3})$.

Comment

Schiff bases are N-ligands owing a ($>\text{C}=\text{N}-$) azomethine functional group which were reported by Hugo Schiff in 1864 [5]. They can be prepared via condensation of a primary amine with ketone or aldehyde compounds, and generally take place under acid catalysis or without catalysts [6]. Schiff bases are useful chelators because of their structural varieties, ease of preparation, and electronic controlling [7]. These ligands coordinate ions via the *N*-imine and some cases the O-carbonyl to form very stable complexes [5–7]. The azomethine moiety is involved in essential biological activity and interferes in cell processes. Therefore Schiff-base complexes could be promising medicinal materials due to their diverse pharmacological applications [6–9].

The molecule crystallizes monoclinically, space group $P2_1/c$. According to the results of the single crystal diffraction

the thione tautomeric structure was found to be favored over the thiol tautomeric one as seen in the figure. The plane of the aryl ring makes a dihedral angle of 0.6° with the plane of the 1,2,4-triazole ring indicating the *exo*-isomer favoured over the *endo*-one. The molecule exhibits one $\text{N}-\text{H}\cdots\text{S}$ classical hydrogen with 2.444 Å. No $\pi-\pi$ stacking interactions were detected in the crystal.

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