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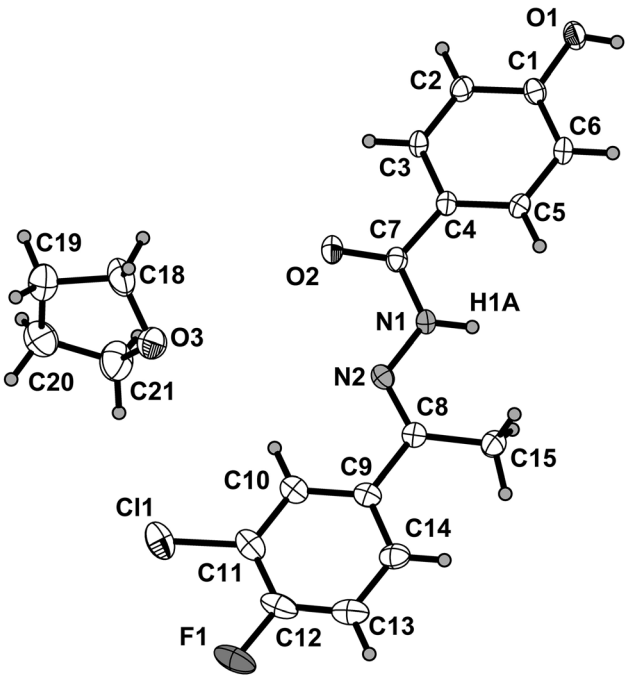
Crystal structure of (*E*)-*N*′-(1-(3-chloro-4-fluorophenyl)ethylidene)-4-hydroxy – tetrahydrofuran (2/1), $C_{17}H_{16}ClFN_2O_{2.5}$



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Abstract

$C_{17}H_{16}ClFN_2O_{2.5}$, monoclinic, $P2_1/n$ (no. 14), $a = 7.4495(6)$ Å, $b = 16.5328(13)$ Å, $c = 12.8519(10)$ Å, $\beta = 91.329(1)°$, $V = 1582.4(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0508$, $wR_{ref}(F^2) = 0.1461$, T = 100(2) K.

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The asymmetric unit of 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.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.20 × 0.18 × 0.16 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.27 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8782, 3237, 0.022
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2624
$N(param)_{refined}$:	237
Programs:	Bruker [1], Shelx [2]

Source of material

A mixture of 2-hydroxy-benzoic acid hydrazide (152.2 mg, 1 mmol) and 1-(3-chloro-4-fluoro-phenyl)-ethanone (1 mmol, 172.6 mg) in 50 mL anhydrous ethanol with a few drops of glacial acetic acid was stirred under reflux conditions for 3 h. The solvent was removed under reduced pressure and the solid product was recrystallized from 15 mL tetrahydrofuran. Crystals were obtained after three days.

Experimental details

Hydrogen atoms were placed in idealized positions and constrained to ride on their parent atoms. The U_{iso} values were set to be $1.5U_{eq}$ of the carrier atom for methyl and oxygen H atoms and $1.2U_{eq}$ for the remaining H atoms. The highest difference electron density peak is is almost 5–10% of a chlorine atom, representing a minor disorder at the Õ-position of the chloro-fluoro-phenyl moiety.

Comment

Schiff bases containing halogen atoms are attractive due to their higher biological activity [3–9].

The asymmetric unit of the title compound consists of one molecule of the organic target compound and half a molecule of tetrahydrofuran solvent located on a two-fold rotation axis (see the figure). Geometric parameters are all in the expected ranges [10]. The title compound shows an *E* configuration around the C8=N2 bond. In the crystal structure, the short distance d(N2–C8) = 1.288(3) Å has a

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.55344 (10)	0.63151 (5)	0.78700 (5)	0.0423 (2)
F1	0.5455 (2)	0.78998 (10)	0.69029 (13)	0.0438 (4)
O1	−0.1736 (2)	0.09435 (10)	0.20902 (12)	0.0279 (4)
H1	−0.219102	0.112331	0.153256	0.042*
O2	0.1487 (2)	0.32791 (10)	0.55528 (12)	0.0293 (4)
N1	0.1437 (2)	0.41662 (11)	0.42123 (13)	0.0191 (4)
H1A	0.116154	0.425879	0.355346	0.023*
N2	0.2148 (2)	0.47762 (11)	0.48274 (14)	0.0197 (4)
C1	−0.1042 (3)	0.15635 (13)	0.26577 (17)	0.0207 (5)
C2	−0.0270 (3)	0.13840 (13)	0.36309 (17)	0.0224 (5)
H2	−0.023764	0.084036	0.386987	0.027*
C3	0.0447 (3)	0.19925 (14)	0.42473 (16)	0.0205 (5)
H3	0.095975	0.186292	0.491016	0.025*
C4	0.0429 (3)	0.27967 (13)	0.39108 (16)	0.0176 (4)
C5	−0.0332 (3)	0.29686 (13)	0.29291 (16)	0.0193 (4)
H5	−0.034472	0.351088	0.268440	0.023*
C6	−0.1064 (3)	0.23651 (14)	0.23100 (16)	0.0209 (5)
H6	−0.158302	0.249423	0.164845	0.025*
C7	0.1163 (3)	0.34238 (14)	0.46269 (16)	0.0190 (4)
C8	0.2414 (3)	0.54582 (13)	0.43718 (17)	0.0197 (4)
C9	0.3228 (3)	0.61075 (14)	0.50305 (18)	0.0216 (5)
C10	0.3919 (3)	0.59196 (15)	0.60253 (18)	0.0243 (5)
H10	0.387253	0.537916	0.627448	0.029*
C11	0.4667 (3)	0.65217 (16)	0.66434 (19)	0.0290 (5)
C12	0.4732 (3)	0.73120 (16)	0.6274 (2)	0.0324 (6)
C13	0.4071 (3)	0.75104 (16)	0.5310 (2)	0.0339 (6)
H13	0.413018	0.805227	0.506723	0.041*
C14	0.3307 (3)	0.69060 (15)	0.46843 (19)	0.0279 (5)
H14	0.283403	0.704025	0.401358	0.034*
C15	0.1943 (4)	0.56159 (15)	0.32441 (18)	0.0298 (5)
H15A	0.248853	0.519845	0.281186	0.045*
H15B	0.239913	0.614838	0.304429	0.045*
H15C	0.063570	0.560342	0.314241	0.045*
O3[a]	−0.0523 (5)	0.5568 (2)	0.8940 (3)	0.0370 (9)
C18[a]	−0.1628 (17)	0.5056 (13)	0.9545 (10)	0.047 (4)
H18A[a]	−0.183287	0.453327	0.918458	0.056*
H18B[a]	−0.280567	0.531462	0.965875	0.056*
C19[a]	−0.0643 (11)	0.4924 (6)	1.0569 (7)	0.050 (2)
H19A[a]	−0.081681	0.436546	1.082702	0.060*
H19B[a]	−0.104242	0.531180	1.110242	0.060*
C20[a]	0.1302 (17)	0.5072 (15)	1.0287 (10)	0.053 (4)
H20A[a]	0.182435	0.551886	1.070863	0.064*
H20B[a]	0.203346	0.457936	1.040349	0.064*
C21[a]	0.1228 (10)	0.5292 (6)	0.9161 (7)	0.049 (2)
H21A[a]	0.211363	0.572158	0.901545	0.059*
H21B[a]	0.150242	0.481446	0.872774	0.059*

[a]Occupancy: 0.5.

value of a typical C=N double bond. The C8=N2–N1 angle of 116.15(18)°. The two aromatic rings are almost parallel to each other with the dihedral angle of 3.2°. In the crystal structure, the intermolecular O–H···O hydrogen bonds link the molecules into chains along the *c*-axis .

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