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The crystal structure of (*E*)-*N'*-(1-(3-chloro-4-fluorophenyl) ethylidene)-2-hydroxy-benzohydrazide, C₁₅H₁₂ClFN₂O₂

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.20 × 0.15 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.29 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	19612, 6361, 0.084
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4081
$N(\text{param})_{\text{refined}}$:	381
Programs:	Bruker [1], SHELX [2]

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	1.0604(2)	0.1301(2)	0.87188(19)	0.0507(7)
H1	1.0593	0.1998	0.8651	0.061*
C2	1.0925(2)	0.0916(2)	0.95159(19)	0.0513(7)
H2	1.1138	0.1346	0.9982	0.062*
C3	1.0924(2)	−0.0121(2)	0.96110(19)	0.0514(7)
H3	1.1128	−0.0391	1.0143	0.062*
C4	1.0626(2)	−0.0756(2)	0.89280(18)	0.0513(7)
H4	1.0640	−0.1451	0.9004	0.062*
C5	1.0304(2)	−0.0370(2)	0.81273(18)	0.0497(7)
C6	1.0294(2)	0.06784(19)	0.80076(18)	0.0504(7)
C7	0.9952(2)	0.1184(2)	0.71837(18)	0.0502(7)
C8	0.8638(2)	0.0364(2)	0.49458(19)	0.0525(8)
C9	0.8362(2)	0.0955(2)	0.41578(18)	0.0505(7)
C10	0.8011(2)	0.0419(2)	0.33914(18)	0.0510(7)
H10	0.8037	−0.0283	0.3409	0.061*
C11	0.7634(2)	0.08997(19)	0.26193(18)	0.0494(7)
C12	0.7603(2)	0.1938(2)	0.25817(19)	0.0514(7)
C13	0.7950(2)	0.2521(2)	0.33139(18)	0.0533(7)
H13	0.7933	0.3223	0.3287	0.064*
C14	0.8322(2)	0.20163(19)	0.40882(19)	0.0538(8)
H14	0.8557	0.2397	0.4585	0.065*
C15	0.8372(2)	−0.07834(19)	0.49145(18)	0.0527(7)
H15A	0.8685	−0.1016	0.5500	0.079*
H15B	0.8741	−0.1150	0.4693	0.079*
H15C	0.7527	−0.0892	0.4531	0.079*
C16	0.5796(2)	0.14467(19)	0.87763(18)	0.0494(7)
H16	0.5765	0.0748	0.8714	0.059*
C17	0.6282(2)	0.18537(19)	0.96035(19)	0.0512(7)

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Abstract

C₁₅H₁₂ClFN₂O₂, monoclinic, $P2_1/c$ (no. 14), $a = 13.9210(13)$ Å, $b = 13.2329(15)$ Å, $c = 18.7191(19)$ Å, $\beta = 126.250(2)^\circ$, $V = 2780.9(5)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0499$, $wR_{\text{ref}}(F^2) = 0.1420$, $T = 293(2)$ 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 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H17	0.6581	0.1437	1.0093	0.061*
C18	0.6317(2)	0.28978(19)	0.96899(18)	0.0487(7)
H18	0.6653	0.3184	1.0244	0.058*
C19	0.5861(2)	0.3513(2)	0.89660(18)	0.0511(7)
H19	0.5871	0.4210	0.9032	0.061*
C20	0.5385(2)	0.31049(19)	0.81365(18)	0.0483(7)
C21	0.5347(2)	0.20525(19)	0.80263(18)	0.0489(7)
C22	0.4876(2)	0.1529(2)	0.71805(18)	0.0496(7)
C23	0.3919(2)	0.2306(2)	0.50090(19)	0.0532(8)
C24	0.3444(2)	0.16985(19)	0.41862(18)	0.0500(7)
C25	0.2960(2)	0.2210(2)	0.33834(17)	0.0510(7)
H25	0.2888	0.2909	0.3370	0.061*
C26	0.2592(2)	0.17142(19)	0.26227(18)	0.0504(7)
C27	0.2680(2)	0.06791(19)	0.26216(18)	0.0497(7)
C28	0.3124(2)	0.0105(2)	0.33861(18)	0.0509(7)
H28	0.3163	−0.0597	0.3382	0.061*
C29	0.3502(2)	0.06377(19)	0.41518(18)	0.0510(7)
H29	0.3810	0.0274	0.4670	0.061*
C30	0.4129(2)	0.34686(19)	0.50572(18)	0.0559(8)
H30A	0.3633	0.3804	0.5183	0.084*
H30B	0.3932	0.3703	0.4500	0.084*
H30C	0.4950	0.3615	0.5518	0.084*
Cl1	0.71888(6)	0.02090(5)	0.16886(5)	0.0551(2)
Cl2	0.20314(5)	0.23827(5)	0.16556(4)	0.0528(2)
F1	0.72159(12)	0.24023(11)	0.18107(10)	0.0539(4)
F2	0.23185(12)	0.01976(11)	0.18642(10)	0.0520(4)
N1	0.45966(17)	0.21313(16)	0.64992(15)	0.0488(6)
H1A	0.4694	0.2772	0.6590	0.059*
N2	0.41626(17)	0.17763(16)	0.56630(15)	0.0497(6)
N3	0.95095(17)	0.05715(16)	0.64765(15)	0.0493(6)
H3A	0.9464	−0.0064	0.6547	0.059*
N4	0.91240(18)	0.09048(16)	0.56430(15)	0.0519(6)
O1	1.00053(14)	−0.09903(13)	0.74459(12)	0.0511(5)
H1E	1.0059	−0.1680	0.7625	0.077*
O2	1.00823(14)	0.21007(13)	0.71545(12)	0.0520(5)
O3	0.49501(14)	0.37106(12)	0.74203(12)	0.0500(5)
H3E	0.5034	0.4405	0.7596	0.075*
O4	0.47437(15)	0.06042(13)	0.71121(12)	0.0533(5)

Source of material

A mixture of 2-hydroxy-benzoic acid hydrazide (1 mmol, 152.2 mg) and 1-(3-chloro-4-fluoro-phenyl)-ethanone (1 mmol, 172.6 mg) in anhydrous ethanol (25 mL) was kept under reflux conditions (351 K) for 3 h. The solvent was removed under reduced pressure and the solid product was recrystallized from 15 mL of anhydrous ethanol. After 4 days, colourless block-shaped crystals were obtained.

Experimental details

Coordinates of hydrogen atoms were refined without any constraints or restraints. The U_{iso} values were set to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms.

Comment

In recent years, Schiff bases as one of the important synthetic intermediates have attracted more and more attention due to the wide applications in the fields of coordination chemistry, biochemistry, pharmacy, nanotechnology, and photochemistry [3–10]. From the structural point of view, *o*-hydroxy Schiff bases are attractive because of their intramolecular H-bond, and thus have been extensively investigated and reported [11–17]. In addition, the heteroatoms (such as N, F, Cl, Br, S) can act as hydrogen bonding acceptors for bioactive molecules in order to exhibit higher biological activity [18, 19]. But, the unsymmetrical *o*-hydroxy Schiff bases containing heteroatoms especial of F and Cl in one molecule are less investigated. Herein, in order to search for new *o*-hydroxy Schiff bases containing F and Cl heteroatoms, we have undertaken the synthesis and single crystal structure determination of the title compound (*cf.* the figure).

In the title compound, there are two crystallographically independent molecules in the asymmetric unit, which have very similar conformations. Compared with those of closely related structure, such a structural feature is uncommon [15]. The bond lengths and angles are in the expected ranges which fit with those of related compounds in literature [11–17]. The C=N double bond lengths in the independent molecule are 1.277(3) Å (C8=N4) and 1.271(4) Å (C23=N2), respectively, exhibiting the double bond character. The aromatic rings form dihedral angles of 3.9° and 4.2° in the two independent molecules. In the crystal structure, the molecules are linked into double infinite chains by O–H···O hydrogen bonds. There also exist intramolecular N–H···O hydrogen bonds, which further consolidate the crystal packing.

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