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Crystal structure of (*E*)-*N'*-(1-(5-chloro-2-hydroxyphenyl) ethylidene)thiophene-2-carbohydrazide, C₁₃H₁₁ClN₂O₂S

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Abstract

C₁₃H₁₁ClN₂O₂S, orthorhombic, *Pna*2₁/*n* (no. 33), *a* = 33.781(4) Å, *b* = 5.2087(6) Å, *c* = 7.2979(8) Å, *β* = 90°, *V* = 1284.1(3) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0429, *wR*_{ref}(*F*²) = 0.1267, *T* = 296 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.

Source of material

A mixture of thiophene-2-carbohydrazide (142.2 mg, 1 mmol) and 1-(5-chloro-2-hydroxyphenyl)ethanone (170.6 mg, 1 mmol) was dissolved in 25 mL anhydrous ethanol containing a catalytic amount of glacial acetic acid. The reaction mixture was stirred under reflux for 3 h, then cooled to room temperature and filtered, and then

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.23 × 0.20 × 0.18 mm
Wavelength:	MoKα radiation (0.71073 Å)
<i>μ</i> :	0.46 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, <i>φ</i> and <i>ω</i>
<i>θ</i> _{max} , completeness:	25.0°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	6160, 2235, 0.036
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 <i>σ</i> (<i>I</i> _{obs}), 1794
<i>N</i> (<i>param</i>) _{refined} :	175
Programs:	Bruker [1], SHELX [2]

left at room temperature. After six days, yellow block crystals were obtained.

Experimental details

All hydrogen atoms were positioned geometrically (C–H = 0.93–0.96 Å, O–H = 0.82 Å, N–H = 0.86 Å) and refined using a riding model approximation. The *U*_{iso} values were constrained to be 1.5 *U*_{eq} of the carrier atom for oxygen atom and methyl groups and 1.2 *U*_{eq} for the remaining H atoms.

Comment

Thiophene hydrazone schiff bases are attracting more and more attention due to their biological activities and wide applications in coordination chemistry [3–10].

The asymmetric unit of the title compound consists of one molecule of the organic target compound (see the figure). The title compound shows an *E* configuration around the C6=N2 bond. In the crystal structure, the short distance *d*(C6–N2) = 1.296(7) Å shows a typical C=N double bond. The angle of C6=N2–N1 is 118.4(5)°. In general the bond lengths and angles are as expected [11, 12]. The thiophene and the 5-chloro-2-hydroxyphenyl ring are almost parallel, with the dihedral angle of 1.6°. In the crystal structure, the molecules are linked into chains along the *b*-axis by O–H⋯N and C–H⋯O hydrogen bonds.

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

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.9810 (2)	0.8480 (12)	−0.2536 (10)	0.0548 (19)
H1	0.999426	0.835520	−0.347966	0.066*
C2	0.95310 (19)	1.0332 (12)	−0.2452 (9)	0.0493 (17)
H2	0.950049	1.158192	−0.335087	0.059*
C3	0.92900 (15)	1.0200 (10)	−0.0878 (8)	0.0317 (12)
H3	0.908686	1.133749	−0.059482	0.038*
C4	0.94041 (15)	0.8075 (10)	0.0204 (8)	0.0352 (13)
C5	0.92251 (16)	0.7127 (11)	0.1901 (8)	0.0363 (13)
C6	0.85330 (16)	0.9566 (10)	0.5007 (8)	0.0329 (12)
C7	0.83529 (17)	1.1750 (12)	0.3945 (9)	0.0453 (16)
H7A	0.851286	1.325874	0.409198	0.068*
H7B	0.809073	1.208206	0.439383	0.068*
H7C	0.834009	1.130062	0.267014	0.068*
C8	0.83714 (16)	0.8753 (11)	0.6793 (8)	0.0313 (13)
C9	0.85195 (17)	0.6599 (11)	0.7735 (8)	0.0363 (13)
C10	0.83517 (18)	0.5908 (11)	0.9432 (9)	0.0436 (15)
H10	0.845315	0.450029	1.005952	0.052*
C11	0.80461 (18)	0.7239 (12)	1.0179 (9)	0.0440 (14)
H11	0.793521	0.673025	1.128706	0.053*
C12	0.79030 (15)	0.9362 (10)	0.9260 (9)	0.0368 (13)
C13	0.80551 (16)	1.0129 (11)	0.7604 (8)	0.0340 (13)
H13	0.795003	1.155584	0.701210	0.041*
Cl1	0.75141 (5)	1.1113 (3)	1.0210 (3)	0.0548 (5)
N1	0.90097 (14)	0.8956 (9)	0.2811 (7)	0.0406 (12)
H1A	0.898602	1.048873	0.238615	0.049*
N2	0.88332 (13)	0.8246 (8)	0.4430 (7)	0.0369 (11)
O1	0.92519 (13)	0.4914 (9)	0.2437 (6)	0.0507 (12)
O2	0.88196 (13)	0.5141 (8)	0.7116 (6)	0.0451 (11)
H2A	0.888517	0.563296	0.609249	0.068*
S1	0.97918 (5)	0.6426 (3)	−0.0765 (3)	0.0503 (5)

One intramolecular N–H⋯O hydrogen bond exists, which stabilizes the conformation of the title molecule (see the figure).

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