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The crystal structure of *N'*-(2-chloro-6-hydroxybenzylidene)thiophene-2-carbohydrazide monohydrate, C₁₂H₁₁ClN₂O₃S

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Abstract

C₁₂H₁₁ClN₂O₃S, monoclinic, *P*₂₁/*n* (no. 14), *a* = 4.7305(8) Å, *b* = 21.252(4) Å, *c* = 13.134(2) Å, β = 96.286(2)°, *Z* = 4, *V* = 1312.5(4) Å³, *R*_{gt}(*F*) = 0.1002, *wR*_{ref}(*F*²) = 0.1118, *T* = 296 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of materials

A mixture of thiophene-2-carbohydrazide (142.2 mg, 1 mmol) and 2-chloro-6-hydroxybenzaldehyde (156.6 mg, 1 mmol) was dissolved in 20 mL ethanol. A catalytic amount of glacial acetic acid was added and used as

Table 1: Data collection and handling.

Crystal:	Block, yellow
Size:	0.23 × 0.18 × 0.16 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.46 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ _{max} , completeness:	26.4°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	7324, 2659, 0.041
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 1745
<i>N</i> (<i>param</i>) _{refined} :	222
Programs:	Bruker programs [1], OLEX2 [2], SHELX [3]

catalyst. The reaction mixture was refluxed for 5 h, then cooled to room temperature and filtered, and then left at room temperature quietly. After a few days, light yellow block crystals were obtained.

Experimental details

All hydrogen atoms were positioned geometrically, with C–H = 0.93 Å, N–H = 0.86 Å and O–H = 0.82–0.85 Å. The thiophene ring is disordered over two positions. The two parts were disordered over opposite direction and overlapped each other with site occupation factors of 0.642 and 0.358. The disorder is omitted in the figure for clarity.

Comment

Some thiophene-based Schiff bases have attracted attention due to their biological activities such as antioxidant, antitumor, anti-inflammatory, antimicrobial activities and structural chemistry [4–12]. In order to synthesize new thiophene-based Schiff bases, the title compound was synthesized and its crystal structure determined and reported here.

The asymmetric unit of the title compound consists of one formula unit (*cf.* the figure). The bond length between C6 and N2 is 1.277(3) Å, indicating that the reaction of Schiff base has taken place, and the C=N bond was also formed. The dihedral angle between the thiophene and benzene

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Cl1	0.4677 (2)	0.41023 (5)	0.07264 (7)	0.0794 (3)
S1 ^a	−0.5774 (5)	0.18023 (13)	0.17528 (12)	0.0359 (5)
S1A ^b	−0.5741 (13)	0.1350 (2)	0.3732 (5)	0.0447 (10)
O1	−0.1764 (4)	0.23982 (9)	0.43652 (13)	0.0423 (5)
O2	0.3735 (5)	0.38140 (11)	0.45479 (15)	0.0622 (6)
H2	0.257828	0.354991	0.431318	0.093*
N1	−0.1034 (4)	0.26832 (9)	0.27581 (16)	0.0313 (5)
H1	−0.143715	0.262513	0.211040	0.038*
N2	0.0920 (4)	0.31340 (9)	0.31192 (16)	0.0326 (5)
C1 ^a	−0.8024 (13)	0.1201 (3)	0.2042 (5)	0.0337 (13)
H1A ^a	−0.930103	0.100100	0.155902	0.040*
C2 ^a	−0.7712 (15)	0.1056 (4)	0.3049 (4)	0.0366 (13)
H2A ^a	−0.872661	0.074147	0.333992	0.044*
C3 ^a	−0.560 (3)	0.1452 (5)	0.3620 (10)	0.0389 (16)
H3 ^a	−0.511765	0.142273	0.432393	0.047*
C4 ^a	−0.442 (3)	0.1868 (7)	0.3015 (8)	0.0268 (11)
C5	−0.2301 (5)	0.23355 (11)	0.3434 (2)	0.0284 (6)
C6	0.2079 (5)	0.34486 (12)	0.2449 (2)	0.0336 (6)
H6	0.157538	0.336658	0.175699	0.040*
C7	0.4170 (5)	0.39317 (11)	0.2748 (2)	0.0330 (6)
C8	0.5518 (6)	0.42630 (12)	0.2020 (2)	0.0397 (7)
C9	0.7537 (6)	0.47199 (13)	0.2278 (3)	0.0498 (8)
H9	0.842199	0.492646	0.177544	0.060*
C10	0.8213 (6)	0.48633 (14)	0.3294 (3)	0.0602 (10)
H10	0.956530	0.517172	0.348116	0.072*
C11	0.6925 (7)	0.45590 (15)	0.4034 (3)	0.0585 (9)
H11	0.739815	0.466481	0.471841	0.070*
C12	0.4919 (6)	0.40935 (13)	0.3775 (2)	0.0425 (7)
O3	−0.1429 (4)	0.26026 (11)	0.05977 (13)	0.0520 (6)
H3B	−0.012476	0.256708	0.020446	0.078*
H3C	−0.296377	0.260587	0.019542	0.078*
C4A ^b	−0.438 (6)	0.1887 (12)	0.2979 (14)	0.031 (2)
C1A ^b	−0.790 (3)	0.1057 (6)	0.2723 (8)	0.037 (2)
H1AA ^b	−0.914296	0.072253	0.277537	0.044*
C2A ^b	−0.761 (2)	0.1356 (5)	0.1846 (9)	0.0316 (18)
H2AA ^b	−0.862352	0.126485	0.121661	0.038*
C3A ^b	−0.559 (4)	0.1821 (9)	0.2017 (10)	0.033 (2)
H3A ^b	−0.508760	0.207596	0.149013	0.039*

^aOccupancy: 0.642 (3), ^bOccupancy: 0.358 (3).

rings is 12.154(1)° in the major component. Due to the consequence of repulsion between the nitrogen lone pairs and the adjacent N bonds, the angle of C6=N2–N1 is 116.7(2)°. Geometric parameters are in the expected ranges [6–10, 13]. In the crystal packing, the molecules are linked into one-dimensional chains along the *b*-axis by O–H···O hydrogen bonds. In addition, intramolecular O–H···N and N–H···O hydrogen bonds further consolidate the crystal structure.

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