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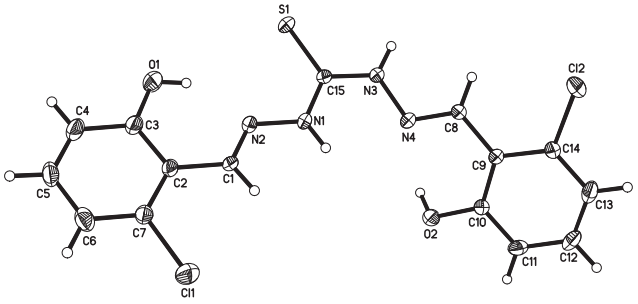
Crystal structure of *N′*,2-bis((*E*)-2-chloro-6-hydroxybenzylidene)hydrazine-1-carbothiohydrazide, $C_{15}H_{12}Cl_2N_4O_2S$



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Abstract

$C_{15}H_{12}Cl_2N_4O_2S$, monoclinic, $P2_1/c$ (no. 14), $a = 12.236(7)$ Å, $b = 20.199(10)$ Å, $c = 6.992(4)$ Å, $\beta = 105.987(9)°$, $V = 1661.2(16)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0707$, $wR_{ref}(F^2) = 0.1917$, $T = 293(2)$ 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.

Source of material

The title compound was prepared by the Schiff-base condensation of thiocarbohydrazide (1 mmol, 106.2 mg) with 2-chloro-6-hydroxybenzaldehyde (2 mmol, 313.1 mg) in ethanol (25 mL) containing a few drops glacial acetic acid under refluxing. The resulting solution was cooled to room temperature, and then left in air for a few days, yielding yellow block crystals suitable for X-ray diffraction determination.

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.25 × 0.23 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.53 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}:	9168, 3378, 0.069
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2520
$N(param)_{refined}$:	220
Programs:	Bruker [1], SHELX [2], PLATON [3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.31512(18)	0.21522(9)	−0.1292(3)	0.0594(6)
Cl2	0.94512(15)	0.15008(9)	1.1821(3)	0.0539(5)
S1	0.38836(14)	−0.00951(7)	0.7008(3)	0.0413(4)
O1	0.1927(5)	0.0069(3)	0.1848(9)	0.0676(18)
H1B	0.242408	0.020337	0.281267	0.101*
O2	0.5682(4)	0.2751(2)	0.8937(9)	0.0478(13)
H2B	0.536599	0.246184	0.941218	0.072*
N1	0.4327(4)	0.1050(2)	0.5477(8)	0.0352(13)
H1	0.469142	0.141719	0.554194	0.042*
N2	0.3486(4)	0.0885(2)	0.3793(8)	0.0339(12)
N3	0.5419(4)	0.0806(2)	0.8591(9)	0.0398(14)
H3	0.567780	0.052397	0.952908	0.048*
N4	0.5887(4)	0.1441(2)	0.8734(8)	0.0354(12)
C1	0.3400(5)	0.1216(3)	0.2229(9)	0.0318(15)
H1A	0.388574	0.157097	0.224392	0.038*
C2	0.2545(5)	0.1043(3)	0.0404(10)	0.0346(15)
C3	0.1859(5)	0.0466(3)	0.0305(11)	0.0460(19)
C4	0.1094(6)	0.0304(4)	−0.1542(13)	0.055(2)
H4A	0.064647	−0.007335	−0.163118	0.067*
C5	0.0988(6)	0.0675(4)	−0.3182(11)	0.050(2)
H5A	0.047441	0.054759	−0.437151	0.061*
C6	0.1630(6)	0.1245(4)	−0.3129(11)	0.0493(19)
H6A	0.155354	0.150320	−0.426102	0.059*
C7	0.2384(5)	0.1415(3)	−0.1343(10)	0.0364(16)
C8	0.6894(5)	0.1497(3)	0.9934(9)	0.0327(14)
H8A	0.728025	0.112274	1.053934	0.039*
C9	0.7427(5)	0.2146(3)	1.0342(9)	0.0299(13)
C10	0.6825(5)	0.2744(3)	0.9868(9)	0.0341(14)

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C11	0.7335(6)	0.3355(3)	1.0375(10)	0.0408(16)
H11A	0.690843	0.374159	1.007099	0.049*
C12	0.8483(6)	0.3386(3)	1.1336(11)	0.0439(17)
H12A	0.882653	0.379574	1.169175	0.053*
C13	0.9127(5)	0.2817(3)	1.1776(11)	0.0436(16)
H13A	0.990466	0.284324	1.237444	0.052*
C14	0.8608(5)	0.2209(3)	1.1320(10)	0.0346(14)
C15	0.4569(5)	0.0632(3)	0.7003(9)	0.0304(14)

Experimental details

Hydrogen atoms attached were positioned geometrically and refined using a riding model approximation, with C—H = 0.93 Å, N—H = 0.86 Å and O—H = 0.82 Å. The U_{iso} values were refined with U_{iso}(H) = 1.2 U_{eq}(C, N) and 1.5 U_{eq}(O).

Comment

Thiocarbohydrazones are a class of important Schiff bases derived from the condensation of thiocarbohydrazide and aldehyde or ketone. Because of the existence of thione (>C=S) or thiol (>C—SH) groups in thiocarbohydrazones or their metal complexes, thiocarbohydrazones and their derivatives exhibit interesting biological properties and thus, have gained increasingly more and more attention [4–10]. In order to search for new thiocarbohydrazones, the title compound was synthesized and its crystal structure reported here.

The asymmetric unit of the title compound consists of one formula unit (*cf*. the figure) with bonding parameters in the normal ranges [11]. In the crystal structure of the title compound, the partial double bond between C15 and S1 has a length of 1.691(6) Å, which is between the typical C—S single-bond (1.82 Å) and the typical C=S double-bond (1.56 Å). The C=N double bond lengths are 1.260(8) Å (C1 = N2) and 1.291(7) Å (C8 = N4), respectively, exhibiting typical C=N double-bond characters. The title compound is a non-planar molecule. The dihedral angle between the two aromatic rings is 55.5°. In the crystal structure, a dimer structure is formed by weak N—H···S hydrogen bonds. The O—H···N intramolecular hydrogen bonds further stabilize the crystal structure.

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