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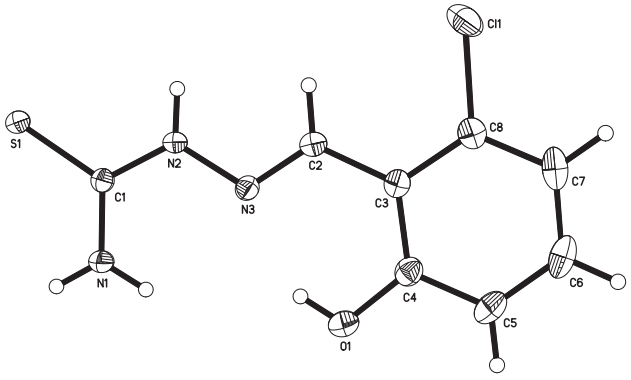
# Crystal structure of (*E*)-2-(2-chloro-6-hydroxybenzylidene)hydrazine-1-carbothioamide, $C_8H_8ClN_3O_4S$

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## Abstract

$C_8H_8ClN_3O_4S$, monoclinic, $P2_1/c$ (no. 14), $a = 12.662(2)$ Å, $b = 5.2782(8)$ Å, $c = 14.841(2)$ Å, $\beta = 94.103(3)°$, $V = 989.3(3)$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.0317$, $wR_{ref}(F^2) = 0.1232$, $T = 293(2)$ 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

The title compound was synthesized *via* the reaction of hydrazinecarbothioamide (1 mmol, 91.1 mg) with 2-chloro-6-hydroxybenzaldehyde (1 mmol, 156.6 mg) in ethanol solution containing a few drops glacial acetic acid under refluxing. When cooled to room temperature, the solution was filtered and left at room temperature. After six days, light yellow crystals were obtained.

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**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Yellow block |
| Size: | 0.26 × 0.23 × 0.20 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 0.57 mm$^{-1}$ |
| Diffractometer, scan mode: | Bruker APEX-II, $\varphi$ and $\omega$ |
| $\theta_{max}$, completeness: | 25.0°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 4752, 1745, 0.021 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 1494 |
| $N(param)_{refined}$: | 128 |
| Programs: | Bruker [1], SHELX [2] |

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

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.43980(15) | −0.6564(4) | 0.90319(13) | 0.0247(5) |
| C2 | 0.29145(16) | −0.1416(4) | 0.81694(14) | 0.0287(5) |
| H2 | 0.324296 | −0.131957 | 0.762968 | 0.034* |
| C3 | 0.20683(15) | 0.0370(4) | 0.83323(14) | 0.0289(5) |
| C4 | 0.15535(17) | 0.0383(4) | 0.91431(15) | 0.0347(5) |
| C5 | 0.07628(19) | 0.2148(5) | 0.92830(19) | 0.0452(6) |
| H5 | 0.043123 | 0.214548 | 0.982236 | 0.054* |
| C6 | 0.0474(2) | 0.3888(5) | 0.8624(2) | 0.0502(7) |
| H6 | −0.005719 | 0.505110 | 0.872205 | 0.060* |
| C7 | 0.0955(2) | 0.3949(5) | 0.7818(2) | 0.0466(7) |
| H7 | 0.075263 | 0.513492 | 0.737565 | 0.056* |
| C8 | 0.17444(17) | 0.2209(4) | 0.76843(16) | 0.0341(5) |
| Cl1 | 0.23532(6) | 0.24002(15) | 0.66736(5) | 0.0590(3) |
| N1 | 0.39513(14) | −0.6967(3) | 0.97940(12) | 0.0326(4) |
| H1A | 0.344512 | −0.600273 | 0.994368 | 0.039* |
| H1B | 0.416592 | −0.819409 | 1.014172 | 0.039* |
| N2 | 0.40402(13) | −0.4624(3) | 0.85054(11) | 0.0294(4) |
| H2A | 0.432918 | −0.432309 | 0.800937 | 0.035* |
| N3 | 0.32168(13) | −0.3110(3) | 0.87481(12) | 0.0280(4) |
| O1 | 0.18019(14) | −0.1282(3) | 0.98182(12) | 0.0487(5) |
| H1 | 0.230677 | −0.214944 | 0.968897 | 0.073* |
| S1 | 0.54036(4) | −0.83831(11) | 0.86945(4) | 0.0349(2) |

## Experimental details

All hydrogen atoms were placed in calculated positions (C—H = 0.93 Å, N—H = 0.86 Å, O—H = 0.82 Å) and refined as riding atoms. The $U_{iso}$ values were constrained to be $1.5U_{eq}$

of the carrier atom for oxygen H atoms and $1.2U_{eq}$ for the remaining H atoms.

## Comment

Thiosemicarbazones are a class of important Schiff-bases and have received considerable attention for many years because of their biological activities and coordination chemistry properties [3–14]. In order to search for new thiosemicarbazones, the title compound was synthesized and its crystal structure was determined.

The asymmetric unit of the title compound consists of one formula unit (*cf*. the figure). The title compound has an *E* configuration with the sulfur atom *trans* to the iminic nitrogen. In the crystal structure of title compound, the short distance *d*(N3—C2) = 1.279(3) Å has a value of a typical C=N double bond. Because of the consequence of repulsion between the nitrogen lone pairs and the adjacent N bonds, the C=N—N angle of 115.11(17)° (C2 = N3—N2) is much smaller than the ideal value of 120° expected for $sp^2$ N atoms. The C1 = S1 bond distance in the molecule is 1.698(2) Å, which is between the typical C=S double-bond (1.56 Å) and the typical C—S single-bond (1.82 Å), thus showing a double bond character. In the crystal structure, a hydrogen bonded dimeric structure is formed by two classical N—H···S hydrogen bonds. The adjacent dimers are further linked into three-dimensional frameworks by weak C—H···S hydrogen bonds. In addition, intramolecular O—H···N hydrogen bonds further consolidate the crystal packing. A database search gave the result that the structure of the parent compound (without the chloro substituent) [3] and another very closely related structure is known [4].

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