Jianlian Liu*, Hong Hao and Chaojun Du

Crystal structure of (*E*)-*N'*-(2-chloro-6-hydroxybenzylidene)-2-hydroxybenzohydrazide, C₁₄H₁₁ClN₂O₃

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Abstract

C₁₄H₁₁ClN₂O₃, monoclinic, *P*₂₁/*n* (no. 14), *a* = 7.0242(11) Å, *b* = 25.375(4) Å, *c* = 7.6789(13) Å, β = 111.2752°, *V* = 1275.4(4) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0349, *wR*_{ref}(*F*²) = 0.1123, *T* = 296(2) K.

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The asymmetric unit of the title 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.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.25 × 0.23 × 0.20 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.31 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ _{max} , completeness:	25.0°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	6399, 2245, 0.023
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 1909
<i>N</i> (<i>param</i>) _{refined} :	183
Programs:	Bruker [1], SHELX [2]

Source of material

A mixture of 2-hydroxy-benzoic acid hydrazide (152.2 mg, 1 mmol) and 2-chloro-6-hydroxybenzaldehyde (156.6 mg, 1 mmol) in 50 mL anhydrous ethanol containing a few drops of glacial acetic acid was stirred under reflux conditions for 2 h. The mixture was cooled to room temperature and filtered, and then left at room temperature. After a few days, yellow block-shaped crystals suitable were obtained.

Experimental details

The hydrogen atoms were positioned geometrically (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 1.2*U*_{eq} of the carrier atom for carbon bound H atoms and 1.5*U*_{eq} for the remaining H atoms.

*Corresponding author: Jianlian Liu, School of Chemical Engineering, Northwest University, 710069, Xi'an, Shaanxi, People's Republic of China; and School of Biological and Chemical Engineering, Nanyang Institute of Technology, 473004, Nanyang, Henan, People's Republic of China, E-mail: zgqjd2005@sohu.com. <https://orcid.org/0000-0001-9270-2293>

Hong Hao, School of Chemical Engineering, Northwest University, 710069, Xi'an, Shaanxi, People's Republic of China, E-mail: tianxtpljh2002@163.com

Chaojun Du, School of Biological and Chemical Engineering, Nanyang Institute of Technology, 473004, Nanyang, Henan, People's Republic of China

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

Atom	x	y	z	U _{iso} [*] /U _{eq}
C1	0.6707 (2)	0.32492 (6)	0.6496 (2)	0.0264 (4)
C2	0.6835 (2)	0.37973 (6)	0.6306 (2)	0.0311 (4)
C3	0.7307 (3)	0.41154 (7)	0.7874 (3)	0.0418 (4)
H3	0.743319	0.447752	0.776416	0.050*
C4	0.7590 (3)	0.38989 (8)	0.9589 (3)	0.0453 (5)
H4	0.790634	0.411732	1.062837	0.054*
C5	0.7414 (3)	0.33620 (8)	0.9798 (2)	0.0423 (4)
H5	0.758484	0.321968	1.096153	0.051*
C6	0.6979 (2)	0.30405 (6)	0.8250 (2)	0.0328 (4)
H6	0.686594	0.267871	0.837962	0.039*
C7	0.6280 (2)	0.29130 (6)	0.4830 (2)	0.0272 (3)
C8	0.7219 (2)	0.16077 (6)	0.4004 (2)	0.0290 (4)
H8	0.767364	0.149250	0.523688	0.035*
C9	0.7070 (2)	0.12389 (6)	0.2529 (2)	0.0272 (3)
C10	0.6617 (2)	0.14042 (6)	0.0664 (2)	0.0328 (4)
C11	0.6581 (3)	0.10471 (8)	−0.0710 (3)	0.0442 (5)
H11	0.631030	0.116142	−0.192701	0.053*
C12	0.6948 (3)	0.05238 (8)	−0.0267 (3)	0.0513 (5)
H12	0.691889	0.028650	−0.119913	0.062*
C13	0.7357 (3)	0.03403 (7)	0.1519 (3)	0.0462 (5)
H13	0.757917	−0.001654	0.179249	0.055*
C14	0.7429 (2)	0.06981 (6)	0.2892 (2)	0.0351 (4)
Cl1	0.79733 (9)	0.04664 (2)	0.51507 (7)	0.0589 (2)
N1	0.69976 (19)	0.24167 (5)	0.51329 (18)	0.0310 (3)
H1A	0.761624	0.230390	0.625330	0.037*
N2	0.6730 (2)	0.20921 (5)	0.36294 (18)	0.0300 (3)
O1	0.6557 (2)	0.40315 (5)	0.46567 (18)	0.0452 (3)
H1	0.618765	0.381112	0.381920	0.068*
O2	0.53252 (19)	0.30788 (4)	0.32239 (16)	0.0380 (3)
O3	0.6208 (2)	0.19117 (5)	0.01546 (18)	0.0471 (4)
H3A	0.631904	0.209004	0.107700	0.071*

Comment

Halogenated Schiff bases are attracting more and more attention in several fields such as biochemistry, pharmacy, and coordination chemistry [3]. In addition, halogenated compounds containing the hydrazide moiety form intramolecular H-bonds [4–11]. In order to search for new Schiff bases containing halogen atoms, the title compound was synthesized and its crystal structure is reported here.

The asymmetric unit of the title compound consists of one formula unit (*cf.* the figure). The molecule exhibits an *E* configuration. The bond length of C8=N2 is 1.2804(19) Å exhibiting that the C8=N2 bond is a double bond. The two aromatic rings form the dihedral angle of 14.3°. The angle of C8=N2–N1 is 116.29(13)°, smaller than the ideal value of 120°.

In the crystal packing, the chain structure was formed by intermolecular N–H···O hydrogen bonds. The intramolecular O–H···O and N–H···O hydrogen bonds also exist, which further consolidate the crystal.

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