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Crystal structure of (*E*)-*N'*-(2-chloro-6-hydroxybenzylidene)-3-hydroxybenzo-hydrazide monohydrate, C₁₄H₁₃ClN₂O₄

<https://doi.org/10.1515/ncrs-2021-0465>

Received December 6, 2021; accepted January 13, 2022;

published online January 28, 2022

Abstract

C₁₄H₁₃ClN₂O₄, monoclinic, *P*₂₁/*n* (no. 14), *a* = 9.7353(13) Å, *b* = 13.7790(18) Å, *c* = 10.5227(13) Å, β = 97.961(2)°, *V* = 1397.9(3) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0367, *wR*_{ref}(*F*²) = 0.1181, *T* = 296(2) K.

CCDC no.: 2141831

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 3-hydroxy-benzoic acid hydrazide (0.76 g, 5 mmol), 2-chloro-6-hydroxybenzaldehyde (0.78 g, 5 mmol) and

Table 1: Data collection and handling.

Crystal:	Yellow block
Size	0.25 × 0.23 × 0.20 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.29 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} :	6983, 2466, 0.020
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2029
<i>N</i> (<i>param</i>) _{refined} :	200
Programs:	Bruker [1], SHELX [2]

ethanol (20 mL) were stirred and heated at 351 K for 3 h, then cooled to room temperature. The resulting mixture was filtered, and then allowed to stand at room temperature. After a few days, yellow block crystals were obtained.

Experimental details

Hydrogen atoms were placed in idealized positions and refined as riding atoms. The *U*_{iso} values were set to be 1.2*U*_{eq} of the carrier atom for carbon H atoms and 1.5*U*_{eq} for the remaining H atoms.

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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	0.45501 (15)	0.30698 (11)	0.49765 (15)	0.0280 (4)
C2	0.57322 (15)	0.32606 (11)	0.58459 (15)	0.0297 (4)
H2	0.650819	0.286353	0.587292	0.036*
C3	0.57452 (16)	0.40433 (11)	0.66683 (15)	0.0324 (4)
C4	0.45907 (18)	0.46345 (13)	0.66320 (17)	0.0394 (4)
H4	0.459656	0.515381	0.719725	0.047*
C5	0.34346 (18)	0.44508 (13)	0.57564 (17)	0.0409 (4)
H5	0.266548	0.485511	0.572262	0.049*
C6	0.34051 (16)	0.36731 (13)	0.49281 (16)	0.0349 (4)
H6	0.261935	0.355388	0.433914	0.042*
C7	0.44727 (15)	0.22489 (11)	0.40575 (15)	0.0289 (4)
C8	0.62926 (16)	0.00972 (11)	0.37641 (15)	0.0316 (4)
H8	0.691870	0.014761	0.451346	0.038*
C9	0.63853 (16)	−0.07203 (11)	0.29098 (15)	0.0314 (4)
C10	0.74147 (18)	−0.14351 (12)	0.31941 (17)	0.0392 (4)
C11	0.7506 (2)	−0.22351 (14)	0.2418 (2)	0.0505 (5)
H11	0.818605	−0.270349	0.263467	0.061*
C12	0.6571 (2)	−0.23241 (15)	0.1318 (2)	0.0544 (5)
H12	0.661946	−0.286193	0.079184	0.065*
C13	0.55722 (19)	−0.16373 (14)	0.0982 (2)	0.0502 (5)
H13	0.496027	−0.170647	0.022624	0.060*
C14	0.54684 (17)	−0.08356 (12)	0.17673 (17)	0.0373 (4)
Cl1	0.86153 (6)	−0.13198 (4)	0.45584 (5)	0.0654 (2)
N1	0.53422 (13)	0.14968 (9)	0.43621 (13)	0.0295 (3)
H1	0.587676	0.148490	0.508467	0.035*
N2	0.53606 (14)	0.07495 (9)	0.34998 (12)	0.0317 (3)
O1	0.68933 (13)	0.42826 (9)	0.75131 (12)	0.0462 (4)
H1A	0.742272	0.381724	0.760737	0.069*
O2	0.36418 (12)	0.22458 (8)	0.30501 (11)	0.0416 (3)
O3	0.44661 (13)	−0.01869 (10)	0.13772 (12)	0.0501 (4)
H3	0.444725	0.022850	0.193253	0.075*
O4	0.25017 (16)	0.38138 (10)	0.14667 (13)	0.0536 (4)
H4A	0.263 (2)	0.4353 (5)	0.1858 (14)	0.069 (7)*
H4B	0.2904 (19)	0.3394 (8)	0.1980 (13)	0.070 (7)*

Comment

The 3-hydroxybenzoyl hydrazones are an important class of schiff bases and play an important role in coordination chemistry due to their extensive application in assembling metal complexes and the formation of interesting hydrogen bonding in the crystal structures [3–9].

The asymmetric unit of the title compound consists of one molecule of the organic compound and one water molecule (see the Figure). The molecule exhibits an *E* configuration around the C8=N2 bond. In the crystal structure, the short distance $d(\text{C8}–\text{N2}) = 1.280(2)$ Å exhibits a

typical C=N double bond. The C8=N2–N1 angle is 117.15(13)°. The two aromatic rings form the dihedral angle of 17.42(1)°.

In the crystal structure, the water molecules are linked to the schiff base molecules through intermolecular O–H...O and N–H...O hydrogen bonds, forming two-dimensional network. In addition, an intramolecular O–H...N hydrogen bond exists and further consolidate the crystal packing.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was financial supported by Nanyang Institute of Technology.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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