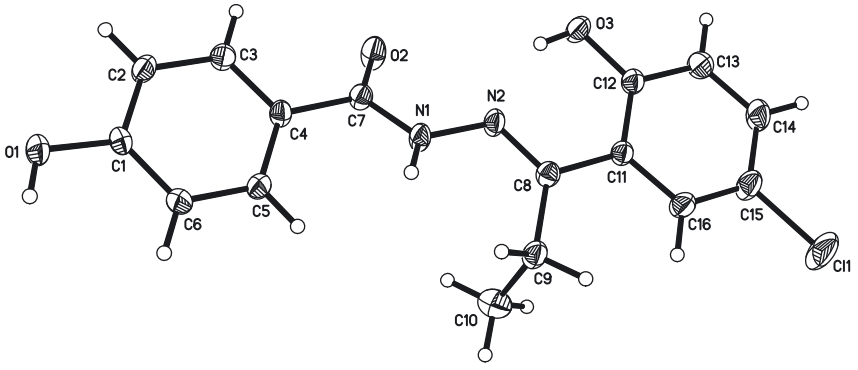
Zhaoyan Li*

Crystal structure of (*E*)-*N*′-(1-(5-chloro-2-hydroxyphenyl)propylidene)-4-hydroxybenzohydrazide, $C_{16}H_{15}ClN_2O_3$



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Abstract

$C_{16}H_{15}ClN_2O_3$, monoclinic, $P2_1/c$ (no. 14), a = 4.9561(17) Å, b = 31.458(11) Å, c = 9.764(3) Å, β = 93.581(6)°, V = 1519.3(9) Å^3, Z = 4, $R_{gt}(F)$ = 0.0497, $wR_{ref}(F^2)$ = 0.1359, T = 296(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.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.23 × 0.20 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.27 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7703, 2668, 0.078
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 1755
$N(param)_{refined}$:	202
Programs:	Bruker [1], SHELX [2]

Source of material

A mixture of 4-hydroxybenzohydrazide (1 mmol, 152.2 mg), 1-(5-chloro-2-hydroxyphenyl)propan-1-one (184.6 mg, 1 mmol), a few drops of glacial acetic acid, and 20 mL anhydrous ethanol was refluxed. The resulting solution was cooled to room temperature, and then left undisturbed for about several days to form yellow block crystals.

Experimental details

All H atoms were placed in idealized calculated positions and refined as riding on their parent atoms.

Comment

The 4-hydroxybenzoyl hydrazones as an important class of schiff-bases are widely investigated in coordination chemistry because of their excellent coordination properties [3–9].

***Corresponding author: Zhaoyan Li**, School of Biological and Chemical Engineering, Nanyang Institute of Technology, 473004, Nanyang, Henan, P. R. China, E-mail: abcdefnyist123123@163.com.
https://orcid.org/0000-0002-7034-1607

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8395 (5)	0.34834 (7)	0.5772 (3)	0.0334 (6)
C2	1.0662 (5)	0.34204 (8)	0.6659 (3)	0.0420 (7)
H2	1.161883	0.365298	0.702201	0.050*
C3	1.1499 (5)	0.30127 (8)	0.7003 (3)	0.0390 (7)
H3	1.302646	0.297311	0.759166	0.047*
C4	1.0088 (5)	0.26629 (7)	0.6480 (3)	0.0308 (6)
C5	0.7878 (5)	0.27303 (8)	0.5552 (3)	0.0374 (7)
H5	0.695093	0.249792	0.516676	0.045*
C6	0.7042 (5)	0.31355 (8)	0.5195 (3)	0.0379 (6)
H6	0.557314	0.317516	0.456822	0.046*
C7	1.1040 (5)	0.22312 (7)	0.6899 (3)	0.0318 (6)
C8	0.8394 (5)	0.11986 (7)	0.6782 (2)	0.0313 (6)
C9	0.5988 (5)	0.12448 (8)	0.5771 (3)	0.0394 (7)
H9A	0.493241	0.149044	0.601123	0.047*
H9B	0.484729	0.099510	0.581022	0.047*
C10	0.6906 (7)	0.12974 (11)	0.4337 (3)	0.0696 (10)
H10A	0.804720	0.154318	0.430281	0.104*
H10B	0.535757	0.133272	0.370620	0.104*
H10C	0.789629	0.104962	0.408978	0.104*
C11	0.9374 (5)	0.07756 (7)	0.7264 (2)	0.0306 (6)
C12	1.1625 (5)	0.07328 (7)	0.8222 (3)	0.0347 (6)
C13	1.2427 (6)	0.03315 (8)	0.8680 (3)	0.0487 (8)
H13	1.387931	0.030803	0.932617	0.058*
C14	1.1134 (6)	−0.00333 (8)	0.8207 (3)	0.0491 (8)
H14	1.169391	−0.029981	0.852448	0.059*
C15	0.8978 (6)	0.00097 (8)	0.7246 (3)	0.0459 (7)
C16	0.8107 (5)	0.04029 (8)	0.6784 (3)	0.0405 (7)
H16	0.664774	0.042065	0.614012	0.049*
Cl1	0.73066 (19)	−0.04462 (2)	0.66223 (11)	0.0828 (4)
N1	0.9102 (4)	0.19243 (6)	0.6815 (2)	0.0356 (5)
H1A	0.747153	0.198099	0.651866	0.043*
N2	0.9851 (4)	0.15165 (6)	0.7225 (2)	0.0352 (5)
O1	0.7606 (4)	0.38922 (5)	0.5508 (2)	0.0500 (5)
H1	0.622706	0.389236	0.499860	0.075*
O2	1.3387 (3)	0.21498 (5)	0.7277 (2)	0.0474 (5)
O3	1.3061 (4)	0.10718 (5)	0.87114 (19)	0.0470 (5)
H3A	1.235335	0.129114	0.841096	0.070*

The asymmetric unit of the title compound consists of one formula unit (*cf*. the figure). In the title crystal structure, the acylhydrazone molecule is in a ketone form and adopts an *E* configuration at the C=N double bond. The bond length between C8 and N2 is 1.292(3) Å, which shows a typical C=N double bond [10]. The angle of C8=N2–N1 is 119.0(2)°. The dihedral angle between the two aromatic rings of 3.5° indicates that they are all almost parallel to each other. In the solid state, the molecules are linked into two-dimensional networks along the *ac*-plane by O–H···O and N–H···O hydrogen bonds. In addition, intramolecular O–H···N hydrogen bonds further contribute to the stability of the structure.

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References

1. Bruker. APEX2, SAINT and SADABS; Bruker AXS Inc.: Madison, WI, USA, 2008.
2. Sheldrick G. M. A short history of *Shelx*. *Acta Crystallogr.* 2008, *A64*, 112–122.
3. Bao X., Tian C. Crystal structure of (*E*)-*N*′-(1-(3-chloro-4-fluorophenyl) ethylidene)-4-hydroxybenzohydrazide – tetrahydrofuran (2/1), $C_{17}H_{16}ClFN_2O_{2.5}$. *Z. Kristallogr. N. Cryst. Struct.* 2021, *236*, 1383–1384.
4. Chen J., Liu Z., Yan H. Crystal structure of (*E*)-*N*′-(2-chloro-6-hydroxybenzylidene)-4-hydroxybenzohydrazide—dihydrofuran-2(3*H*)-one (1/1), $C_{18}H_{17}ClN_2O_5$. *Z. Kristallogr. N. Cryst. Struct.* 2020, *235*, 997–998.
5. Yan X., Zhong S., Zhang H. The crystal structure of (*E*)-*N*′-(1-(3-chloro-4-fluorophenyl) ethylidene-2-hydroxybenzohydrazine, $C_{15}H_{12}ClFN_2O_2$. *Z. Kristallogr. N. Cryst. Struct.* 2020, *235*, 263–265.
6. Chantrapromma S., Kwong H. C., Prachumrat P., Kobkeatthawin T., Chia T. S., Quah C. K. Crystal structure of (*E*)-*N*′-(3,4-dihydroxybenzyl idene)-4-hydroxybenzohydrazide. *Acta Crystallogr.* 2019, *E75*, 1280–1283.
7. Chantrapromma S., Prachumrat P., Ruanwas P., Boonnak N., Kassim M. B. Crystal structure of (*E*)-4-hydroxy-*N*′-(3-methoxybenzy lidene) benzohydrazide. *Acta Crystallogr.* 2016, *E72*, 1339–1342.
8. Rassem H. H., Salhin A., Bin Salleh B., Rosli M. M., Fun H.-K. (*E*)-4-hydroxy-*N*′-(2-hydroxy-4-methoxybenzylidene) benzohydrazide. *Acta Crystallogr.* 2012, *E68*, o2279.
9. Hou J.-L. 4-Hydroxy-*N*′-(3-nitrobenzylidene)benzohydrazide. *Acta Crystallogr.* 2012, *E68*, o1352.
10. Zhao R.-G., Lu J., Chang J.-G. *N*′-[(1*E*)-1-(5-Chloro-2-hydroxyphenyl)propylidene]-4-methoxybenzohydrazide. *Acta Crystallogr.* 2010, *E66*, o140.