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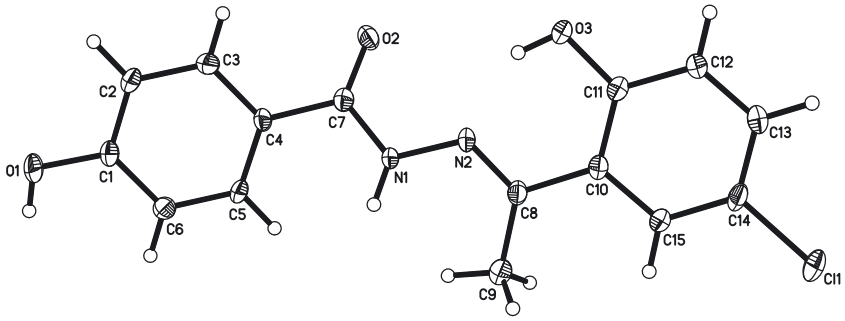
Crystal structure of (*E*)-*N*′-(1-(5-chloro-2-hydroxyphenyl) ethylidene)-4-hydroxybenzohydrazide, $C_{15}H_{13}ClN_2O_3$



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Abstract

$C_{15}H_{13}ClN_2O_3$, monoclinic, $P2_1/c$ (no. 14), a = 4.9709(11) Å, b = 31.691(7) Å, c = 8.512(2) Å, β = 92.376(4)°, V = 1339.8(5) Å^3, Z = 4, $R_{gt}(F)$ = 0.0559, $wR_{ref}(F^2)$ = 0.1676, T = 296(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

To a solution of 4-hydroxybenzohydrazide (1 mmol, 152.2 mg) in 10 mL anhydrous ethanol were added 1-(5-chloro-2-hydroxyphenyl)ethanone (1 mmol, 170.6 mg) and a catalytic amount of glacial acetic acid. The reaction mixture was heated under reflux conditions for 3 h. The resulting solution left quietly at room temperature, and evaporated naturally. After four days, light yellow block crystals were obtained.

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.22 × 0.20 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.30 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}:	6634, 2343, 0.047
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1555
$N(param)_{refined}$:	193
Programs:	Bruker [1], SHELX [2]

Experimental details

Coordinates of hydrogen atoms were placed in idealized positions (C–H = 0.93–0.96 Å, O–H = 0.82 Å, N–H = 0.86 Å) and constrained to ride on their parent atoms. The U_{iso} values were constrained to be 1.5U_{eq} of the carrier atom for methyl H atoms and oxygen H atoms and 1.2U_{eq} for the remaining H atoms.

Comment

Aroylhydrazones ligands and their metal complexes are an important class of compounds and widely investigated due to their various biological activities, such as antiinflammatory, anticonvulsing, antimicrobial, anticancer, and antitubercular activities [3–6].

The asymmetric unit of the title structure contains one formula unit (*cf*. the figure). The bond lengths and angles are in the expected ranges [7–10]. The C8=N2 double bond shows the *E* configuration of the substituents in the title compound.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1513 (7)	0.34764 (9)	0.3898 (4)	0.0327 (8)
C2	−0.0678 (7)	0.33926 (10)	0.2904 (5)	0.0406 (10)
H2	−0.153880	0.361110	0.235425	0.049*
C3	−0.1591 (7)	0.29846 (10)	0.2726 (4)	0.0368 (9)
H3	−0.308301	0.293055	0.206074	0.044*
C4	−0.0331 (6)	0.26539 (9)	0.3518 (4)	0.0271 (8)
C5	0.1837 (6)	0.27453 (9)	0.4534 (4)	0.0317 (9)
H5	0.269107	0.252776	0.509284	0.038*
C6	0.2748 (7)	0.31507 (10)	0.4730 (4)	0.0344 (9)
H6	0.419780	0.320661	0.542427	0.041*
C7	−0.1322 (6)	0.22190 (9)	0.3242 (4)	0.0294 (8)
C8	0.1208 (6)	0.11936 (9)	0.3546 (4)	0.0280 (8)
C9	0.3465 (7)	0.12262 (10)	0.4734 (5)	0.0430 (10)
H9A	0.363939	0.151334	0.508360	0.064*
H9B	0.510523	0.113851	0.427475	0.064*
H9C	0.311189	0.104797	0.561412	0.064*
C10	0.0317 (6)	0.07786 (9)	0.2918 (4)	0.0285 (8)
C11	−0.1886 (6)	0.07373 (9)	0.1837 (4)	0.0311 (8)
C12	−0.2654 (7)	0.03443 (10)	0.1295 (4)	0.0390 (10)
H12	−0.411975	0.032208	0.058544	0.047*
C13	−0.1328 (7)	−0.00134 (10)	0.1769 (4)	0.0384 (9)
H13	−0.189375	−0.027677	0.140432	0.046*
C14	0.0860 (7)	0.00240 (9)	0.2796 (4)	0.0332 (9)
C15	0.1674 (7)	0.04088 (9)	0.3382 (4)	0.0325 (9)
H15	0.314068	0.042429	0.409448	0.039*
Cl1	0.2629 (2)	−0.04288 (3)	0.33606 (13)	0.0542 (4)
N1	0.0576 (5)	0.19130 (7)	0.3467 (3)	0.0313 (7)
H1	0.215020	0.196838	0.387156	0.038*
N2	−0.0157 (5)	0.15076 (7)	0.3011 (3)	0.0309 (7)
O1	0.2390 (5)	0.38819 (6)	0.4026 (4)	0.0516 (8)
H1A	0.357028	0.389769	0.472633	0.077*
O2	−0.3664 (4)	0.21341 (7)	0.2869 (3)	0.0437 (7)
O3	−0.3338 (5)	0.10751 (6)	0.1313 (3)	0.0408 (7)
H3A	−0.266281	0.129195	0.167757	0.061*

The bond length of this double bond is with the distance *d*(C8–N2) of 1.278(4) Å in the normal range. The C8=N2–N1 angle of 119.5(3)° is very close to the ideal value. The two aromatic rings form a dihedral angle of 13.29°. In the crystal structure, O–H···O and N–H···O hydrogen bonds link the molecules into two-dimensional networks in the *ac* plane. In addition, intramolecular O–H···N hydrogen bonds further consolidate the structure.

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

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