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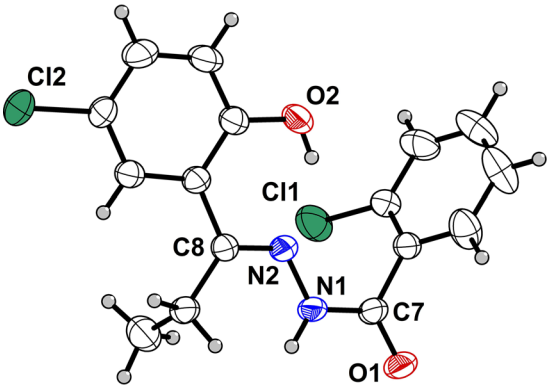
Crystal structure of *E*-2-chloro-*N'*-(1-(5-chloro-2-hydroxyphenyl)propylidene)benzohydrazide, $C_{16}H_{14}Cl_2N_2O_2$



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Abstract

$C_{16}H_{14}Cl_2N_2O_2$, triclinic, $P\bar{1}$ (no. 2), a = 8.9215(15) Å, b = 10.0013(15) Å, c = 10.5713(15) Å, α = 62.517(2)°, β = 75.325(2)°, γ = 68.9930(10)°, V = 776.8(2) Å^3, Z = 2, $R_{gt}(F)$ = 0.0417, $wR_{ref}(F^2)$ = 0.1414, 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:	0.20 × 0.18 × 0.16 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.43 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}:	3964, 2724, 0.014
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2304
$N(param)_{refined}$:	201
Programs:	Bruker [1], SHELX [2]

Source of material

A mixture of 2-chlorobenzohydrazide (170.6 mg, 1 mmol), 1-(5-chloro-2-hydroxyphenyl)propan-1-one (184.6 mg, 1 mmol), a few drops of glacial acetic acid, and anhydrous ethanol (25 mL) was stirred under reflux conditions for 2 h. The solvent was removed under reduced pressure and the solid product was recrystallized from 10 mL of anhydrous ethanol. After five days, yellow block crystals were obtained.

Experimental details

All hydrogen atoms were placed in idealized positions (C—H = 0.93–0.97 Å, O—H = 0.82 Å, N—H = 0.86 Å) and refined using a riding model approximation. The U_{iso} values were constrained to be 1.5 U_{eq} of the carrier atom for methyl groups and oxygen atom and 1.2 U_{eq} for the remaining H atoms.

Comment

Acylhydrazones are well known ligands in coordination chemistry and have been widely used [3–8]. In order to search for new acylhydrazones containing halogen substituents with higher biological activities, the title compound was synthesized.

The asymmetric unit of the title compound consists of one molecule of the organic compound (see the figure). The molecule is in the *E* configuration at the C8—N2 double bond with a short distance 1.290(2) Å. The C8=N2–N1 angle is 119.30(15)°. The dihedral angle formed by the two

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6504 (2)	0.8061 (2)	0.20593 (19)	0.0356 (4)
C2	0.6094 (2)	0.9561 (2)	0.1979 (2)	0.0390 (5)
C3	0.6574 (3)	1.0751 (3)	0.0782 (3)	0.0564 (6)
H3	0.628186	1.175707	0.074740	0.068*
C4	0.7484 (3)	1.0435 (3)	−0.0354 (3)	0.0668 (7)
H4	0.782915	1.122617	−0.115836	0.080*
C5	0.7890 (4)	0.8955 (3)	−0.0309 (3)	0.0688 (8)
H5	0.850173	0.874529	−0.108518	0.083*
C6	0.7388 (3)	0.7779 (3)	0.0891 (2)	0.0541 (6)
H6	0.764915	0.678240	0.090957	0.065*
C7	0.5934 (3)	0.6766 (2)	0.3308 (2)	0.0376 (4)
C8	0.8306 (2)	0.6103 (2)	0.58589 (19)	0.0324 (4)
C9	0.7765 (2)	0.4849 (2)	0.7200 (2)	0.0368 (4)
H9A	0.863154	0.425813	0.781461	0.044*
H9B	0.753932	0.412906	0.694680	0.044*
C10	0.6272 (3)	0.5531 (3)	0.8015 (2)	0.0541 (6)
H10A	0.653758	0.610934	0.840077	0.081*
H10B	0.587838	0.469501	0.878406	0.081*
H10C	0.545426	0.622005	0.737800	0.081*
C11	0.9520 (2)	0.6779 (2)	0.5887 (2)	0.0328 (4)
C12	1.0171 (2)	0.7846 (2)	0.4641 (2)	0.0385 (5)
C13	1.1365 (3)	0.8380 (3)	0.4729 (2)	0.0496 (6)
H13	1.182196	0.905280	0.389331	0.060*
C14	1.1887 (3)	0.7944 (3)	0.6010 (2)	0.0495 (5)
H14	1.268316	0.831527	0.605244	0.059*
C15	1.1203 (2)	0.6943 (2)	0.7234 (2)	0.0381 (5)
C16	1.0054 (2)	0.6358 (2)	0.7187 (2)	0.0368 (4)
H16	0.962468	0.567180	0.803238	0.044*
Cl1	0.49233 (9)	1.00034 (8)	0.33955 (7)	0.0674 (3)
Cl2	1.18209 (7)	0.63822 (8)	0.88898 (6)	0.0558 (2)
N1	0.6569 (2)	0.61445 (19)	0.45538 (17)	0.0410 (4)
H1	0.624703	0.540805	0.528952	0.049*
N2	0.7733 (2)	0.66874 (19)	0.46502 (17)	0.0369 (4)
O1	0.4947 (2)	0.62461 (19)	0.31963 (16)	0.0550 (5)
O2	0.9717 (2)	0.8395 (2)	0.33297 (15)	0.0531 (4)
H2	0.909603	0.793559	0.336855	0.080*

aromatic rings is 66.556(1)°. All parameters are in the expected ranges [9–11]. In the crystal structure, a dimer structure is formed through intermolecular N—H···O hydrogen bonds. In addtion, the intramolecular O—H···N hydrogen bonds are also presented in the crystal.

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

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