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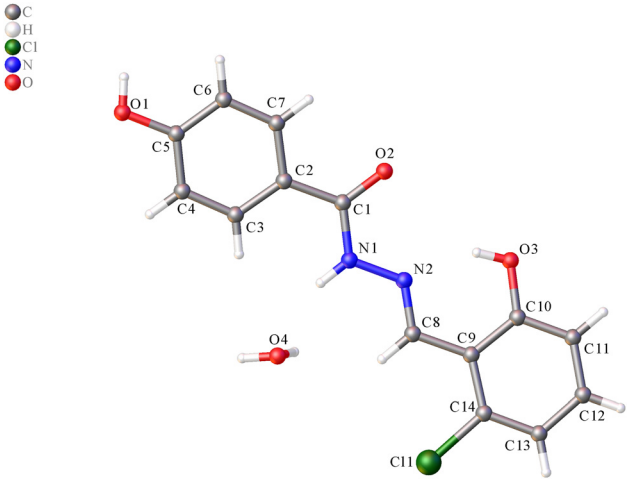
Crystal structure of (*E*)-*N*′-(2-chloro-6-hydroxybenzylidene)-4-hydroxybenzohydrazide-water (1/1), $C_{14}H_{13}Cl_1N_2O_4$



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Abstract

$C_{14}H_{13}Cl_1N_2O_4$, monoclinic, *I*2/*a* (no. 15), a = 13.257(5) Å, b = 16.161(4) Å, c = 13.262(3) Å, β = 97.47(3)°, V = 2817.2(14) Å^3, Z = 8, $R_{gt}(F)$ = 0.0578, $wR_{ref}(F^2)$ = 0.1714, T = 293 K.

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The asymmetric unit of the title crystal 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:	0.15 × 0.12 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.29 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7970, 2878, 0.059
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1556
$N(param)_{refined}$:	192
Programs:	Bruker [1], Olex2 [2], SHELX [3]

Source of material

A mixture of 4-hydroxybenzohydrazide (1 mmol, 152.2 mg) and 2-chloro-6-hydroxybenzaldehyde (1 mmol, 156.6 mg) in anhydrous ethanol (15 ml) with a few drops of glacial acetic acid was stirred under reflux conditions (353 K) for 3 h. The solvent was removed under reduced pressure and the solid product was recrystallized from 10 mL of anhydrous tetrahydrofuran. Colourless block crystals were obtained after five days.

Experimental details

All hydrogen atoms were placed in the calculated positions (C—H = 0.93 Å, O—H = 0.82–0.85 Å, N—H = 0.86 Å) and refined using a riding model approximation. The U_{iso} values were constrained to be 1.5U_{eq} of the carrier atom for oxygen H atoms and 1.2U_{eq} for the remaining H atoms.

Comment

In general, as a kind of well known Schiff-bases, hydrazones are widely studied in coordination chemistry for many years. And many hydrazones structures have been reported [4–7]. In order to search for new hydrazones, the title compound was synthesized and its crystal

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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	0.0735 (2)	0.35609 (18)	0.8730 (2)	0.0442 (7)
C2	−0.03652 (19)	0.37122 (17)	0.8694 (2)	0.0407 (7)
C3	−0.1108 (2)	0.31042 (18)	0.8521 (2)	0.0537 (8)
H3	−0.091723	0.255873	0.842667	0.064*
C4	−0.2121 (2)	0.32979 (19)	0.8486 (3)	0.0575 (9)
H4	−0.260960	0.288477	0.836163	0.069*
C5	−0.2414 (2)	0.40943 (18)	0.8635 (2)	0.0470 (7)
C6	−0.1695 (2)	0.47048 (18)	0.8828 (2)	0.0552 (9)
H6	−0.189080	0.524572	0.894287	0.066*
C7	−0.0686 (2)	0.45094 (18)	0.8852 (2)	0.0509 (8)
H7	−0.020218	0.492648	0.897713	0.061*
C8	0.2390 (2)	0.18814 (18)	0.8735 (2)	0.0457 (7)
H8	0.193614	0.143976	0.866715	0.055*
C9	0.3484 (2)	0.17369 (18)	0.8846 (2)	0.0445 (7)
C10	0.4179 (2)	0.2395 (2)	0.8921 (2)	0.0493 (7)
C11	0.5213 (2)	0.2252 (2)	0.9048 (2)	0.0607 (9)
H11	0.566435	0.269490	0.910864	0.073*
C12	0.5576 (3)	0.1460 (3)	0.9085 (3)	0.0716 (10)
H12	0.627445	0.137072	0.917060	0.086*
C13	0.4926 (3)	0.0791 (2)	0.8998 (3)	0.0677 (10)
H13	0.517799	0.025378	0.901489	0.081*
C14	0.3896 (2)	0.0937 (2)	0.8885 (2)	0.0547 (8)
Cl1	0.30838 (7)	0.00874 (5)	0.87773 (8)	0.0805 (4)
N1	0.10424 (17)	0.27646 (14)	0.86669 (17)	0.0461 (6)
H1	0.061068	0.236513	0.858806	0.055*
N2	0.20666 (16)	0.26212 (15)	0.87335 (17)	0.0434 (6)
O1	−0.34286 (13)	0.42525 (13)	0.85927 (19)	0.0621 (7)
H1A	−0.351628	0.475006	0.866598	0.093*
O2	0.13688 (14)	0.41191 (12)	0.88265 (17)	0.0591 (6)
O3	0.38714 (15)	0.31897 (13)	0.88702 (17)	0.0599 (6)
H3A	0.325734	0.321064	0.889667	0.090*
O4	0.01026 (17)	0.12368 (14)	0.7914 (2)	0.0852 (8)
H4A	−0.050427	0.112224	0.766364	0.128*
H4B	0.039603	0.136584	0.740334	0.128*

structure is reported here. Unlike Chen's team's findings, the asymmetric unit of the title compound consists of a molecular formula unit of the target hydrazone plus a water molecule [8].

The asymmetric unit of the title compound consists of one molecule of the organic target compound and a water molecule (see the figure). The molecule displays an *E* configuration around the C8=N2 bond. In the crystal structure, the short distance *d*(N2—C8) = 1.270(3) Å exhibits a typical C=N double bond. The C8=N2–N1 angle of 119.2(2)° is smaller than the ideal value of 120° expected for sp^2-hybridized N atoms. The dihedral angle formed by the two aromatic rings is 10.147(1)°.

In the crystal structure, the molecules are linked into two-dimensional networks by O—H···O and N—H···O hydrogen bonds. In addition, the intramolecular O—H···N hydrogen bond further consolidates the crystal structure. The bond lengths and angles are in the expected ranges.

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

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