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Crystal structure of (*E*)-*N'*-(2-hydroxy-4-(2-(piperidin-1-yl)ethoxy)benzylidene)nicotinohydrazide monohydrate, $C_{20}H_{24}N_4O_3 \cdot H_2O$

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Abstract

$C_{20}H_{26}N_4O_4$, monoclinic, $P2_1/a$ (no. 14), $a = 12.107(3)$ Å, $b = 7.8431(19)$ Å, $c = 21.794(5)$ Å, $\beta = 98.233(5)^\circ$, $V = 2048.1(8)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0458$, $wR_{ref}(F^2) = 0.1208$, $T = 293(2)$ 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.

Source of material

A solution of 2-hydroxy-4-(2-(piperidin-1-yl)ethoxy)benzaldehyde (249 mg, 1 mmol) in ethanol (20 mL) was added to a stirred ethanol solution (20 mL) of nicotino-hydrazide (137 mg, 1 mmol) in a three-necked flask. The reaction mixture was refluxed for 12 h, then the mixture was cooled to room temperature. The white precipitated residue was removed from the solution by filtration and then washed with ethanol and ether 3 times. The white solids (0.1 mmol) were dissolved in 15 mL ethanol solution. Single crystals

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.25 × 0.24 × 0.23 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	D8/APEX2, φ and ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	21499, 3619, 0.072
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 2203
$N(param)_{refined}$:	258
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U_{iso}^*/U_{eq}
O1	0.70046(13)	0.1550(2)	−0.30779(7)	0.0660(5)
O1W	1.05067(12)	0.4726(2)	−0.23883(7)	0.0512(4)
H1WA	1.0601	0.5789	−0.2438	0.077*
H1WB	1.1020	0.4256	−0.2557	0.077*
O2	0.71636(15)	0.0595(3)	−0.11985(7)	0.0805(6)
H2	0.7267	0.0962	−0.1538	0.121*
O3	0.85216(13)	0.0826(2)	0.08991(6)	0.0629(5)
N1	0.85393(14)	0.2816(2)	−0.25669(7)	0.0417(4)
H1	0.9115	0.3446	−0.2586	0.050*
N2	0.83031(14)	0.2235(2)	−0.19984(8)	0.0456(5)
N3	0.91183(13)	0.1795(2)	0.25557(7)	0.0415(4)
N4	0.95211(16)	0.3935(3)	−0.43186(8)	0.0581(5)
C1	0.83202(18)	0.1703(3)	0.30013(9)	0.0526(6)
H1A	0.8102	0.0524	0.3044	0.063*
H1B	0.7656	0.2343	0.2840	0.063*
C2	0.87848(18)	0.2395(3)	0.36328(9)	0.0520(6)
H2A	0.8244	0.2225	0.3915	0.062*
H2B	0.8913	0.3611	0.3601	0.062*
C3	0.98691(19)	0.1524(3)	0.38905(10)	0.0586(7)
H3A	1.0197	0.2086	0.4270	0.070*
H3B	0.9725	0.0344	0.3987	0.070*
C4	1.06696(18)	0.1599(3)	0.34181(10)	0.0583(7)
H4A	1.0885	0.2774	0.3363	0.070*
H4B	1.1338	0.0957	0.3570	0.070*
C5	1.01452(18)	0.0878(3)	0.28036(10)	0.0538(6)
H5A	1.0674	0.0953	0.2510	0.065*
H5B	0.9973	−0.0317	0.2855	0.065*

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C6	0.86058(19)	0.1067(3)	0.19627(9)	0.0541(6)
H6A	0.7836	0.1445	0.1878	0.065*
H6B	0.8602	−0.0166	0.1999	0.065*
C7	0.91929(19)	0.1551(3)	0.14256(9)	0.0520(6)
H7A	0.9235	0.2780	0.1386	0.062*
H7B	0.9942	0.1084	0.1476	0.062*
C8	0.86955(19)	0.1308(3)	0.03203(9)	0.0475(6)
C9	0.79060(19)	0.0748(3)	−0.01527(10)	0.0535(6)
H9	0.7314	0.0081	−0.0064	0.064*
C10	0.79861(19)	0.1169(3)	−0.07553(10)	0.0504(6)
C11	0.88776(17)	0.2165(3)	−0.09008(9)	0.0444(5)
C12	0.96699(19)	0.2678(3)	−0.04115(10)	0.0524(6)
H12	1.0275	0.3319	−0.0498	0.063*
C13	0.95960(19)	0.2278(3)	0.01951(10)	0.0548(6)
H13	1.0136	0.2648	0.0514	0.066*
C14	0.90051(19)	0.2662(3)	−0.15251(10)	0.0476(6)
H14	0.9620	0.3317	−0.1587	0.057*
C15	0.78645(18)	0.2387(3)	−0.30831(10)	0.0422(5)
C16	0.81753(16)	0.2976(2)	−0.36844(9)	0.0381(5)
C17	0.73562(18)	0.3033(3)	−0.41924(10)	0.0586(7)
H17	0.6630	0.2703	−0.4157	0.070*
C18	0.7623(2)	0.3580(4)	−0.47511(10)	0.0707(8)
H18	0.7078	0.3642	−0.5097	0.085*
C19	0.8703(2)	0.4033(3)	−0.47911(10)	0.0589(7)
H19	0.8871	0.4428	−0.5169	0.071*
C20	0.92423(18)	0.3422(3)	−0.37777(9)	0.0502(6)
H20	0.9805	0.3359	−0.3440	0.060*

suitable for X-ray diffraction were obtained from an ethanol by slow evaporation at room temperature.

Experimental details

The structure was solved using Charge Flipping and refined with the ShelXL [3] refinement. All hydrogen atoms were positioned geometrically, with the $d(C-H) = 0.97-0.99$ Å, $U_{iso}(H) = 1.2$ times $U_{eq}(C)$ and $U_{iso}(H) = 1.5$ times $U_{eq}(O)$.

Comment

Hydrazones are a class of organic compounds with common structure $R_1R_2C=NNR_3R_4$, which can be converted into ketones and aldehydes *via* the oxidation of the NNR_3R_4 functional group. They are usually prepared *via* the condensation of aldehydes or ketones with hydrazines in the presence of an acid as catalyst. Hydrazones have received much attention because of their particular physical,

chemical, and biological activities based on their unique structure [5].

In the crystal structure of the title compound, the asymmetric unit of the title compound consists of one target molecule and one water molecule. The dihedral angle of the pyridyl ring and benzylidene moiety is 158.73°. The bond lengths and bond angles are in the normal ranges [6, 7]. There is intramolecular hydrogen bonds (for example: $O2-H2 \cdots N2$) in the molecules, which is typical for such compounds [8]. The hydrogen bonds $O1W-H1WA \cdots N3$, $O1W-H1WB \cdots O1$, $N1-H1 \cdots O1W$ between the target compound and water molecule extends the framework into a 2D structure.

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