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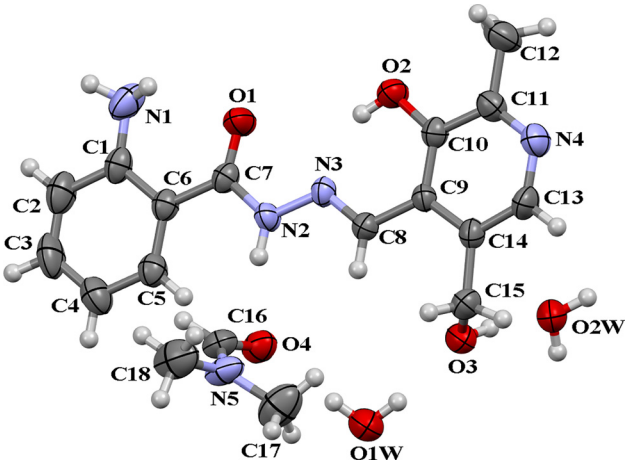
Crystal structure of (*E*)-2-amino-*N*′-((3-hydroxy-5-(hydroxymethyl)-2-methylpyridin-4-yl)methylene) benzohydrazide – dimethylformamide – water (1/1/2), $C_{15}H_{16}N_4O_3 \cdot C_3H_7NO \cdot 2H_2O$



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Abstract

$C_{18}H_{27}N_5O_6$, monoclinic, $P21/n$ (no. 14), a = 11.6588(12) Å, b = 7.0645(6) Å, c = 25.700(3) Å, β = 94.899(3)°, V = 2109.0(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0630, $wR_{ref}(F^2)$ = 0.1632, T = 273 K.

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The 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.

1 Source of materials

A solution of 2-aminobenzohydrazide (151 mg, 1 mmol) in ethanol (25 ml) was added to a stirred ethanol solution (25 ml) of 3-hydroxy-5-(hydroxymethyl)-2-methylisonicotinaldehyde (167 mg, 1 mmol) in a three-necked flask. The reaction mixture was refluxed for 12 h, then the solution was cooled to room temperature. The yellow precipitated residue was removed from the solution by filtration and then washed with ethanol 3 times. The yellow solids (0.2 mmol) were dissolved in a solution mixture (45 ml, CH_3OH/DMF, 8:1) within 7 days. Single crystals suitable for X-ray diffraction were obtained by slow evaporation at room temperature.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.2 × 0.2 × 0.15 mm
Wavelength	Mo *K*α radiation (0.71073 Å)
μ:	0.1 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ_{max}, completeness:	26°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12424, 4070, 0.102
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1844
$N(param)_{refined}$:	274
Programs:	OLEX2 [1], Bruker programs [2], SHELX [3], DIAMOND [4]

2 Experimental details

Crystal data, data collection and structure refinement details are summarized in Table 1. Using Olex2 [1], 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).

3 Comment

Acylhydrazones as a special kind of Schiff base have been widely investigated because their structures are similar to that of natural biological substances [5]. Acylhydrazones exhibit strong coordination abilities and flexible coordination

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	0.25686 (16)	0.7385 (3)	0.56082 (7)	0.0499 (6)
H2	0.317899	0.744932	0.547259	0.075*
O3	0.58899 (16)	0.7475 (3)	0.71957 (8)	0.0546 (6)
H3	0.554795	0.841077	0.729097	0.082*
O4	0.76673 (17)	0.5615 (3)	0.59111 (8)	0.0552 (6)
O1	0.44549 (18)	0.7995 (3)	0.45887 (8)	0.0591 (6)
N3	0.47432 (19)	0.7123 (3)	0.55708 (9)	0.0387 (6)
N2	0.56592 (19)	0.7175 (3)	0.52767 (9)	0.0410 (6)
H2A	0.634301	0.691214	0.540902	0.049*
N4	0.1945 (2)	0.6393 (4)	0.69189 (10)	0.0552 (7)
N5	0.8088 (2)	0.2478 (4)	0.59874 (11)	0.0569 (7)
N1	0.5183 (3)	0.8405 (4)	0.36565 (10)	0.0670 (8)
H1A	0.520579	0.812343	0.333088	0.080*
H1B	0.468888	0.767860	0.378926	0.080*
C8	0.4888 (2)	0.6711 (4)	0.60559 (11)	0.0387 (7)
H8	0.562010	0.646382	0.621366	0.046*
C9	0.3894 (2)	0.6637 (4)	0.63563 (10)	0.0350 (7)
C10	0.2779 (2)	0.6954 (4)	0.61193 (11)	0.0398 (7)
C7	0.5450 (3)	0.7663 (4)	0.47624 (11)	0.0400 (7)
C6	0.6441 (2)	0.7765 (4)	0.44447 (11)	0.0410 (7)
C14	0.3998 (2)	0.6215 (4)	0.68922 (11)	0.0402 (7)
C5	0.7569 (3)	0.7514 (4)	0.46639 (12)	0.0478 (8)
H5	0.769695	0.728869	0.502064	0.057*
C11	0.1830 (2)	0.6820 (4)	0.64169 (13)	0.0464 (8)
C1	0.6255 (3)	0.8128 (4)	0.39011 (12)	0.0471 (8)
C16	0.7837 (3)	0.4039 (5)	0.57231 (13)	0.0554 (9)
H16	0.778224	0.394951	0.536082	0.066*
C15	0.5149 (3)	0.5877 (4)	0.71894 (12)	0.0511 (8)
H15A	0.502894	0.553478	0.754617	0.061*
H15B	0.552247	0.481755	0.703321	0.061*
C13	0.3015 (3)	0.6112 (5)	0.71469 (12)	0.0545 (9)
H13	0.309135	0.582744	0.750155	0.065*
C4	0.8493 (3)	0.7590 (4)	0.43674 (15)	0.0624 (9)
H4	0.923764	0.741537	0.451989	0.075*
C2	0.7218 (3)	0.8189 (4)	0.36098 (14)	0.0642 (10)
H2B	0.711275	0.840936	0.325219	0.077*
C3	0.8294 (3)	0.7934 (5)	0.38373 (16)	0.0710(11)
H3A	0.891478	0.798947	0.363330	0.085*
C12	0.0643 (3)	0.7146 (5)	0.61596 (14)	0.0712 (10)
H12A	0.040354	0.605789	0.595432	0.107*
H12B	0.064725	0.823843	0.593771	0.107*
H12C	0.011725	0.734930	0.642211	0.107*
C17	0.8150 (4)	0.2473 (5)	0.65483 (14)	0.0864 (13)
H17A	0.859477	0.353886	0.668085	0.130*
H17B	0.850993	0.132417	0.667792	0.130*
H17C	0.738740	0.255224	0.666090	0.130*
C18	0.8172 (3)	0.0686 (5)	0.57124(15)	0.0847 (13)
H18A	0.755625	−0.013245	0.579521	0.127*
H18B	0.889698	0.009787	0.581809	0.127*
H18C	0.811873	0.091351	0.534289	0.127*
O2W	0.50095 (18)	1.0797 (3)	0.75177 (8)	0.0569 (6)
H2WA	0.558553	1.132257	0.768455	0.085*
H2WB	0.441112	1.098348	0.767793	0.085*
O1W	0.81272 (18)	0.7480 (4)	0.69001 (10)	0.0659 (6)
H1WA	0.747355	0.746871	0.702423	0.099*
H1WB	0.800454	0.698770	0.659905	0.099*

modes due to the N, O donor atoms. It has also been found that acylhydrazones containing pyridine ring are easier to coordinate because of the additional N donor atom. In the crystal structure of the title compound, the asymmetric unit of the title compound consists of one formula unit of the title molecule, one DMF and two water molecules. The dihedral angle of the amino-phenyl ring and pyridine ring is 2.83°. The bond angles and bond lengths are in the normal ranges [6, 7]. There are intramolecular hydrogen bonds (O2–H2B···N3 and N1–H1B···O1) in the molecules. The hydrogen bonds between the target compound and water molecules extend the framework into a 2*D* structure.

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

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