Li-Lang Li, Chang-Jin Yu, Qing Shi, Qi-Long Zhang* and Xian-Jiong Yang

Crystal structure of (*E*)-2-amino-*N'*-(2-hydroxy-4-(2-(piperidin-1-yl)ethoxy)benzylidene)benzohydrazide monohydrate, $C_{21}H_{26}N_4O_3 \cdot H_2O$

<https://doi.org/10.1515/ncrs-2020-0283>

Received June 11, 2020; accepted June 24, 2020; available online July 2, 2020

Abstract

$C_{21}H_{26}N_4O_3$, triclinic, $P\bar{1}$ (no. 2), $a = 6.5543(12)$ Å, $b = 11.875(2)$ Å, $c = 14.386(3)$ Å, $\alpha = 71.803(3)^\circ$, $\beta = 81.663(3)^\circ$, $\gamma = 89.928(3)^\circ$, $V = 1051.3(3)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0548$, $wR_{\text{ref}}(F^2) = 0.1762$, $T = 273(2)$ K.

CCDC no.: 2011918

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

2-Aminobenzohydrazide (151 mg, 1 mmol) in ethanol (25 mL) was added to a stirred ethanol solution (25 mL) of 2-hydroxy-4-(2-(piperidin-1-yl)ethoxy)benzaldehyde (249 mg, 1 mmol) in a three-necked flask. The reaction mixture was refluxed for

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.22 \times 0.20 \times 0.18$ 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:	27.6°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	13938, 4859, 0.038
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3281
$N(\text{param})_{\text{refined}}$:	277
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.7279(3)	0.31552(14)	−0.06447(13)	0.0469(4)
C2	0.6844(3)	0.24614(17)	−0.12331(15)	0.0589(5)
H2	0.7851	0.2411	−0.1743	0.071*
C3	0.4998(3)	0.18637(18)	−0.10782(16)	0.0658(6)
H3	0.4769	0.1405	−0.1475	0.079*
C4	0.3450(3)	0.1933(2)	−0.03324(16)	0.0689(6)
H4	0.2182	0.1528	−0.0230	0.083*
C5	0.3818(3)	0.26044(17)	0.02510(14)	0.0548(5)
H5	0.2774	0.2653	0.0747	0.066*
C6	0.5716(2)	0.32220(14)	0.01255(12)	0.0420(4)
C7	0.6142(2)	0.38981(14)	0.07819(12)	0.0422(4)
C8	0.3514(3)	0.47669(16)	0.27211(13)	0.0483(4)
H8	0.2192	0.4469	0.2739	0.058*
C9	0.3854(3)	0.54607(15)	0.33540(12)	0.0457(4)
C10	0.2186(3)	0.57527(18)	0.39392(14)	0.0563(5)
H10	0.0873	0.5460	0.3935	0.068*
C11	0.2395(3)	0.64558(18)	0.45237(14)	0.0591(5)
H11	0.1249	0.6640	0.4902	0.071*
C12	0.4370(3)	0.68848(17)	0.45342(13)	0.0534(5)
C13	0.6062(3)	0.66126(17)	0.39658(13)	0.0560(5)
H13	0.7372	0.6900	0.3981	0.067*
C14	0.5826(3)	0.59190(17)	0.33760(13)	0.0490(4)
C15	0.3103(3)	0.7870(2)	0.57027(15)	0.0662(6)
H15A	0.2383	0.7151	0.6157	0.079*
H15B	0.2135	0.8332	0.5304	0.079*
C16	0.3992(3)	0.85776(17)	0.62658(14)	0.0569(5)
H16A	0.5004	0.8118	0.6636	0.068*
H16B	0.4690	0.9297	0.5802	0.068*
C17	0.1140(4)	0.9858(2)	0.64551(17)	0.0743(6)
H17A	0.0535	0.9648	0.5951	0.089*
H17B	0.2018	1.0571	0.6132	0.089*

*Corresponding author: Qi-Long Zhang, State Key

Laboratory of Functions and Applications of Medicinal Plants, Guizhou Medical University, Guiyang 550025, P.R. China, e-mail: gzuqlzhang@126.com. <https://orcid.org/0000-0002-6715-9548>

Li-Lang Li: State Key Laboratory of Functions and Applications of Medicinal Plants, the key laboratory of Chemistry for Natural Products of Guizhou Province and Chinese Academy of Sciences, Guizhou Medical University, Guiyang 550025, P.R. China

Chang-Jin Yu, Qing Shi and Xian-Jiong Yang: School of Basic Medical Science, Guizhou Medical University, Guiyang 550025, P.R. China

Table 2 (continued)

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C18	−0.0566(4)	1.0097(2)	0.7201(2)	0.0935(7)
H18A	−0.1320	1.0769	0.6867	0.112*
H18B	−0.1526	0.9411	0.7469	0.112*
C19	0.0301(4)	1.0353(2)	0.8041(2)	0.0897(7)
H19A	0.1061	1.1114	0.7793	0.108*
H19B	−0.0824	1.0395	0.8546	0.108*
C20	0.1691(4)	0.94094(19)	0.84825(16)	0.0695(6)
H20A	0.0887	0.8672	0.8819	0.083*
H20B	0.2344	0.9629	0.8969	0.083*
C21	0.3323(3)	0.92280(17)	0.77007(14)	0.0544(5)
H21A	0.4167	0.9955	0.7384	0.065*
H21B	0.4209	0.8610	0.8006	0.065*
N1	0.9144(3)	0.37452(18)	−0.08607(15)	0.0705(5)
H1A	0.999(4)	0.359(2)	−0.1266(18)	0.085*
H1B	0.951(4)	0.411(2)	−0.0442(18)	0.085*
N2	0.4603(2)	0.39449(12)	0.15142(10)	0.0454(3)
H2A	0.3411	0.3601	0.1584	0.054*
N3	0.5022(2)	0.45605(12)	0.21375(10)	0.0466(3)
N4	0.2379(2)	0.88913(12)	0.69518(10)	0.0492(4)
O1	0.78382(18)	0.44023(12)	0.07083(10)	0.0575(4)
O2	0.75415(19)	0.56876(14)	0.28213(11)	0.0683(4)
H2B	0.713(4)	0.530(2)	0.2490(16)	0.082*
O3	0.4792(2)	0.75835(13)	0.50855(10)	0.0688(4)
O4	1.0320(2)	0.31756(13)	0.20996(11)	0.0600(4)
H4A	0.958(3)	0.2536(17)	0.2395(17)	0.090*
H4B	0.966(4)	0.363(2)	0.1719(17)	0.090*

8 h, then the solution was cooled to room temperature. The white precipitated residue was removed by filtration and then washed with ethanol and ether 2 times. The white solids (0.1 mmol) were dissolved in 20 mL ethanol. Single crystals were obtained from 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 $d(\text{C}—\text{H}) = 0.97–0.99 \text{ \AA}$, $U_{\text{iso}}(\text{H}) = 1.2 \text{ times } U_{\text{eq}}(\text{C})$ and $U_{\text{iso}}(\text{H}) = 1.5 \text{ times } U_{\text{eq}}(\text{O})$.

Comment

Hydrazones are a class of organic compounds with common structure $\text{R}_1\text{R}_2\text{C}=\text{NNR}_3\text{R}_4$, 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 [5].

In the crystal structure of the title compound, the asymmetric unit of the title compound consists of one formula unit and one water molecule. The bond angles and bond lengths are in the normal ranges [6–8]. There are intramolecular hydrogen bonds ($\text{O2}—\text{H2B} \cdots \text{N3}$ and $\text{N1}—\text{H1B} \cdots \text{O1}$) in the molecule. Through further $\text{N}—\text{H} \cdots \text{O}$ and $\text{O}—\text{H} \cdots \text{N}$ hydrogen bonds between the target compound and water molecule the connection extends into a 3D structure.

Acknowledgements: This work was supported by the Science and Technology Foundation of Guizhou Province (grant number [2019]2792, grant number [2018]5779–14, grant number [2016]7362), the Provincial General Project of University Student Innovation and Entrepreneurship Fund, China (no. 20195200127).

References

- Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: Olex2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **42** (2009) 339–341.
- Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2012).
- Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
- Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Ver. 4.0. Crystal Impact, Bonn, Germany (2015).
- Tian, F. F.; Jiang, F. L.; Han, X. L.; Xiang, C.; Ge, Y. S.; Li, J. H.; Zhang, Y.; Li, R.; Ding, X. L.; Liu, Y.: Synthesis of a novel hydrazone derivative and biophysical studies of its interactions with bovine serum albumin by spectroscopic, electrochemical, and molecular docking methods. *J. Phys. Chem. B* **114** (2010) 14842–14853.
- Park, H.; Kim, J.; Kwon, E.; Kim, T. H.: Crystal structure of flucetosulfuron. *Acta Crystallogr.* **E73** (2017) 1439–1442.
- Weng, Q.; Zhao, L.: 2-Chloro-*N'*-(2-hydroxy-4-methoxybenzylidene) benzohydrazine. *Acta Crystallogr.* **E65** (2009) o808.
- Lee, S. M.; Lo, K. M.; Tiekink, E. R. T.: Crystal structure of 4-chloro-*N'*-[(1*E*)-(3-ethoxy-2-hydroxyphenyl)methylidene] benzohydrazide – a $Z' = 3$ structure, $\text{C}_{16}\text{H}_{15}\text{ClN}_2\text{O}_3$. *Z. Kristallogr. NCS* **234** (2019) 1341–1344.