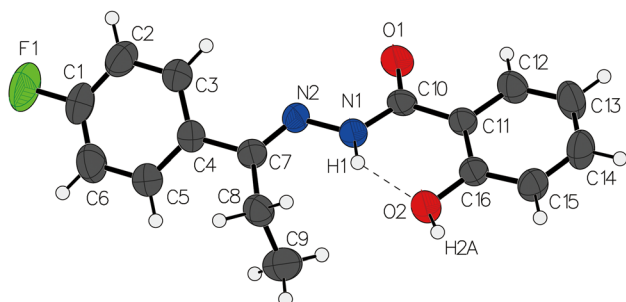


Sha Yang, Jing Men and Wenqiang Tang*

The crystal structure of (*E*)-*N'*-(1-(4-fluorophenyl) propylidene)-2-hydroxybenzohydrazide, $C_{16}H_{15}FN_2O_2$

**Table 1:** Data collection and handling.

Crystal:	Yellow block
Size:	0.46 × 0.43 × 0.37 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffractometer, scan mode:	Bruker D8 Venture, φ and ω
$\theta_{\max}$, completeness:	29.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11,714, 3392, 0.025
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2448
$N(\text{param})_{\text{refined}}$:	193
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

<https://doi.org/10.1515/ncrs-2023-0113>

Received March 12, 2023; accepted March 24, 2023;

published online April 6, 2023

Abstract

$C_{16}H_{15}FN_2O_2$, monoclinic, $P2_1/c$ (no. 14), $a = 9.2751(7)$ Å, $b = 13.1232(7)$ Å, $c = 11.7155(8)$ Å, $\beta = 90.011(7)^\circ$, $V = 1426.00(16)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0570$, $wR_{\text{ref}}(F^2) = 0.1462$, $T = 293$ K.

CCDC no.: 2251418

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.

1 Source of materials

A single crystal of (*E*)-*N'*-(1-(4-fluorophenyl) propylidene)-2-hydroxybenzohydrazide was synthesized through a facile

method. Initially, salicyl hydrazide (0.15 g, 1 mmol) and 4'-fluoropropiophenone (0.15 g, 1 mmol) were dissolved in 25 mL of absolute alcohol in a round-bottom flask equipped with a stir bar and rubber septum. The mixture was further treated with five drops of glacial acetic acid and stirred for 10 min at 60 °C. Following the addition of acetic acid, the reaction mixture turned yellowish, and stirring was continued for 6 h at the same temperature. The solvent was then evaporated to dryness using a rotary evaporator, yielding the crude product that was then purified to obtain a single crystal of high quality. For crystal growth, the crude product was dissolved in a minimal amount of hot ethanol and slowly cooled to room temperature.

2 Experimental details

The Direct Methods implemented in SHELXT [2] were utilized in order to solve the crystal structure, and it was further refined using the SHELXL program [3]. All hydrogen atoms were placed at calculated positions and refined isotropically. Finally, the OLEX2 software package [4] was employed for generating the graphical representation of the crystal structure.

3 Comment

The *N*-acylhydrazone moiety has gained considerable attention in the development of biologically active compounds,

*Corresponding author: Wenqiang Tang, Collaborative Innovation Center of Green Manufacturing Technology for Traditional Chinese Medicine in Shaanxi Province, School of Pharmacy, Shaanxi Institute of International Trade & Commerce, Xi'an, China, E-mail: twqscxq@163.com. <https://orcid.org/0000-0001-5150-4549>

Sha Yang, Collaborative Innovation Center of Green Manufacturing Technology for Traditional Chinese Medicine in Shaanxi Province, School of Pharmacy, Shaanxi Institute of International Trade & Commerce, Xi'an, China

Jing Men, Xi'an Wanlong Pharmaceutical Co. Ltd., Xi'an, China

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

Atom	x	y	z	U _{iso} [*] /U _{eq}
C1	1.0138 (3)	0.47461 (19)	0.74180 (18)	0.0621 (6)
C2	0.8893 (3)	0.43123 (16)	0.70312 (18)	0.0641 (6)
H2	0.860663	0.367323	0.728896	0.077*
C3	0.8067 (2)	0.48401 (15)	0.62506 (17)	0.0547 (5)
H3	0.722544	0.454800	0.596918	0.066*
C4	0.84766 (19)	0.58012 (13)	0.58804 (15)	0.0447 (4)
C5	0.9741 (2)	0.62134 (16)	0.63075 (17)	0.0580 (5)
H5	1.002600	0.686048	0.607547	0.070*
C6	1.0590 (2)	0.56774 (18)	0.70754 (19)	0.0660 (6)
H6	1.144805	0.595135	0.734801	0.079*
C7	0.7588 (2)	0.63509 (13)	0.50206 (15)	0.0441 (4)
C8	0.7567 (2)	0.75048 (14)	0.50179 (16)	0.0526 (5)
H8A	0.658952	0.773642	0.488052	0.063*
H8B	0.785808	0.774961	0.576452	0.063*
C9	0.8548 (3)	0.79586 (18)	0.4128 (2)	0.0767 (7)
H9A	0.825323	0.773114	0.338454	0.115*
H9B	0.952155	0.774540	0.426907	0.115*
H9C	0.849287	0.868846	0.416217	0.115*
C10	0.54761 (19)	0.57292 (12)	0.26588 (15)	0.0427 (4)
C11	0.46200 (18)	0.62964 (12)	0.17896 (14)	0.0398(4)
C12	0.3931 (2)	0.57202 (14)	0.09568 (17)	0.0538(5)
H12	0.402777	0.501499	0.097119	0.065*
C13	0.3110 (2)	0.61596 (17)	0.01124(18)	0.0648(6)
H13	0.265243	0.575751	−0.043187	0.078*
C14	0.2975 (2)	0.72066 (16)	0.00852 (17)	0.0608 (6)
H14	0.242352	0.751136	−0.048322	0.073*
C15	0.3643 (2)	0.78005 (14)	0.08844 (16)	0.0521 (5)
H15	0.354330	0.850520	0.085530	0.063*
C16	0.44699 (18)	0.73583 (12)	0.17398 (15)	0.0422 (4)
F1	1.09534 (17)	0.42254 (12)	0.81874 (13)	0.0963 (5)
N1	0.60765 (16)	0.62681 (10)	0.35036 (12)	0.0454 (4)
H1	0.595414	0.691702	0.353578	0.055*
N2	0.68935 (16)	0.57799 (10)	0.43243 (12)	0.0444 (4)
O1	0.56368 (17)	0.48054 (9)	0.25904 (13)	0.0685 (5)
O2	0.51725 (17)	0.79355 (9)	0.25231 (12)	0.0629 (4)
H2A	0.493628	0.853386	0.244749	0.094*

making it a promising structural motif for drug discovery [5]. An essential step in understanding the properties and behavior of organic compounds is the preparation and study of single crystal structures [6]. Herein, we report the single crystal structure of (*E*)-*N'*-(1-(4-fluorophenyl)propylidene)-2-hydroxybenzohydrazide, which provides significant insights into its molecular structure and packing arrangement.

There is one molecule in the asymmetric unit (see the figure). In terms of molecular geometry, the bond lengths and angles were found to be consistent with those that are typically observed for similar compounds. Notably, the C–N bond length was determined to be 1.338(3) Å, while the C=N bond length was observed to be 1.281(3) Å, indicating the presence of partial single and double bond character [7–10]. The angles between the C7–N2–N1 and C10–N1–N2 atoms

were measured to be 116.58(14)° and 119.86(14)°, respectively, and these values fell within the ranges of what is typically observed in similar chemical systems [11, 12].

The crystal structure of (*E*)-*N'*-(1-(4-fluorophenyl)propylidene)-2-hydroxybenzohydrazide displays a complex three-dimensional network of hydrogen bonds, which involve the carbonyl oxygen and amide nitrogen atoms. The crystal structure is composed of layers of molecules, and the hydrogen bonds (N1–H1...O2 and O2–H2A...O1) facilitate the cohesion of these layers.

The investigation of the crystal structure of 1-(2-chlorophenyl)-3-cycloheptylurea can contribute to a better understanding of the structure-property relationships in similar derivatives [13, 14].

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: Natural Science Foundation of Shaanxi Province (2021JQ-883), The Key Breeding Program by the Collaborative Innovation Center of Green Manufacturing Technology for Traditional Chinese Medicine in Shaanxi Province (2019XT-1-02), Effective Substances of Traditional Chinese Medicine Innovative Team in Shaanxi Institute of International Trade & Commerce (SSY18TD01), and Key Laboratory of Molecular Imaging and Drug Synthesis of Xianyang City (2021QXNL-PT-0008).

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

References

1. Bruker. *SAINT, APEX2 and SADABS*; Bruker AXS Inc.: Madison, WI, USA, 2012.
2. Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. 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.* 2009, *42*, 339–341.
5. Zhang B., Zhao Y., Zhai X., Wang L., Yang J., Tan Z., Gong P. Design, synthesis and anticancer activities of diaryl urea derivatives bearing *N*-acylhydrazone moiety. *Chem. Pharm. Bull.* 2013, *60*, 1046–1054.
6. Ajayeoba T. A., Woods J. O., Ayeni A. O., Ajayi T. J., Akeem R. A., Hosten E. C., Akinyele O. F. Synthesis, crystallographic, computational and molecular docking studies of new acetophenone-benzoylhydrazones. *J. Mol. Struct.* 2021, *1237*, 130275.
7. Wang X.-S., Sheng J., Lu L., Yang K., Li Y.-L. Combinatorial synthesis of 3-arylideneaminoquinazolin-4(1*H*)-one derivatives catalyzed by iodine in ionic liquids. *ACS Comb. Sci.* 2011, *13*, 196–199.
8. Sreeja P. B., Sithambaresan M., Aiswarya N., Kurup M. R. P. *N'*-[(*E*)-2-Fluorobenzylidene]benzohydrazide. *Acta Crystallogr.* 2013, *E69*, o1828.

9. Sivajeyanthi P., Jeevaraj M., Edison B., Balasubramani K. Crystal structure and Hirshfeld surface analysis of (*E*)-4-amino-*N'*-[1-(4-methylphenyl)ethylidene]benzohydrazide. *Acta Crystallogr.* 2017, *E73*, 1029–1032.
10. Ding Y.-W., Ni L.-L. 4-Dimethylamino-*N'*-(3-pyridylmethylidene) benzohydrazide. *Acta Crystallogr.* 2010, *E66*, o2636.
11. Mague J. T., Mohamed S. K., Akkurt M., Potgieter H., Albayati M. R. 4-Chloro-*N'*-[(*E*)-2-chlorobenzylidene]benzohydrazide monohydrate. *Acta Crystallogr.* 2014, *E70*, o612.
12. Xi-Shi T., Yi-Min F. 2-Hydroxy-*N'*-[1-(3-methylpyrazin-2-yl) ethylidene] benzohydrazide. *Acta Crystallogr.* 2008, *E64*, o707.
13. Biswas N., Bera S., Sepay N., Pal A., Halder T., Ray S., Acharyya S., Biswas A. K., Drew M. G. B., Ghosh T. Simultaneous formation of non-oxidovanadium(iv) and oxidovanadium(v) complexes incorporating phenol-based hydrazone ligands in aerobic conditions. *New J. Chem.* 2020, *44*, 3700–3716.
14. Tang C.-B. 2-Methyl-*N'*-[1-(2-pyridyl)ethylidene]benzohydrazide. *Acta Crystallogr.* 2011, *E67*, o271.