Wang Li-Hua, Zhou Xiao-Jing, Liu Li-Li, Zhang Ai-Ling and Tai Xi-Shi*

The crystal structure of (*E*)-2-(5-bromo-2-hydroxybenzylidene)-*N*-phenylhydrazine-1-carboxamide monohydrate, C₁₄H₁₄BrN₃O₃

<https://doi.org/10.1515/ncrs-2020-0471>

Received September 10, 2020; accepted October 13, 2020;

published online November 4, 2020

Abstract

C₁₄H₁₄BrN₃O₃, orthorhombic, *Pca*2₁ (no. 29), *a* = 32.932(3) Å, *b* = 5.8490(7) Å, *c* = 7.3606(8) Å, *V* = 1417.8(3) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0260, *wR*_{ref}(*F*²) = 0.0553, *T* = 150 K.

CCDC no.: 2036778

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:	0.13 × 0.12 × 0.10 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	2.91 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ _{max} , completeness:	25.0°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	8422, 2473, 0.032
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2348
<i>N</i> (<i>param</i>) _{refined} :	194
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

Source of material

A mixture of 0.1005 g 5-bromosalicylaldehyde (0.5 mmol) and 0.0756 g 4-phenylsemicarbazide (0.5 mmol) in ethanol

(10 mL) was stirred at room temperature. After 0.5 h, 2 mL water solution of 0.0595 g calcium perchlorate hydrate (0.25 mmol) was added. The reaction mixture was continually stirred for 3 h at 70 °C and cooled to room temperature. The reactant was filtered to a small flask. The colorless crystals of the title compound were obtained by slow evaporation in 20 days.

*Corresponding author: Tai Xi-Shi, College of Chemistry and Chemical Engineering, Weifang University, Weifang, Shandong, 261061, P. R. China, e-mail: zhangal@wfu.edu.cn. <https://orcid.org/0000-0002-0050-1900>

Wang Li-Hua, Zhou Xiao-Jing, Liu Li-Li and Zhang Ai-Ling, College of Chemistry and Chemical Engineering, Weifang University, Weifang, Shandong, 261061, P. R. China

Experimental details

The hydrogen atoms were positioned geometrically (C-H = 0.93 Å, N-H = 0.86 Å and O-H = 0.82 Å). Their *U*_{iso} values were set to 1.2*U*_{eq} or 1.5*U*_{eq} of the parent atoms.

Comment

Some hydrazone compounds and their metal complex have found considerable applications in catalysis [5], anticancer and antimicrobial activities [6, 7], as well as electrochemical sensors [8]. Our research group has been devoted to the study of hydrazone compounds [9–11]. We wanted to synthesize a Ca(II) complex with (*E*)-2-(5-bromo-2-hydroxybenzylidene)-*N*-phenylhydrazine-1-carboxamide, however, the Ca(II) does not take part in coordination. Only a new hydrazone compound has been obtained.

The molecular structure of the title compound is depicted in the Figure. The compound consists of one (*E*)-2-(5-bromo-2-hydroxybenzylidene)-*N*-phenylhydrazine-1-carboxamide molecule and one water molecule. The bond distances of

C-N are 1.284(5) Å (C7-N1), 1.374(5) Å (C8-N2) and 1.421(5) Å (C9-N3), respectively. The bond distance of C7-N1 is shorter than those of C8-N2 and C9-N3, indicating the C7-N1 bond is a double bond. The dihedral angle between plane 1 (C1-C2-C3-C4-C5-C6) and plane 2 (C9-C10-C11-C12-C13-C14) is 47.8°. In the crystal packing, the hydrazone molecules form a 3D network by intermolecular N-H...O, O-H...N and O-H...O hydrogen bonds.

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

Research funding: This project was supported by the National Natural Science Foundation of China (No. 21171132), the Natural Science Foundation of Shandong (ZR2014BL003), the project of Shandong Province Higher Educational Science and Technology Program (J14LC01) and Science Foundation of Weifang.

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

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Br1	0.51097 (2)	0.38439 (7)	0.72739 (9)	0.03716 (15)
O1	0.67461 (8)	0.7160 (4)	0.4622 (4)	0.0262 (6)
H1	0.692648	0.622504	0.481506	0.039*
O2	0.78571 (9)	0.4150 (4)	0.5381 (4)	0.0240 (6)
N1	0.70558 (10)	0.3172 (5)	0.5857 (4)	0.0202 (7)
N2	0.73580 (10)	0.1605 (5)	0.6187 (4)	0.0233 (8)
H2	0.729854	0.025668	0.656964	0.028*
N3	0.80263 (10)	0.0499 (5)	0.6232 (4)	0.0238 (7)
H3	0.792778	−0.079701	0.656494	0.029*
C1	0.63868 (13)	0.6347 (6)	0.5286 (5)	0.0193 (8)
C2	0.60427 (12)	0.7719 (6)	0.5056 (5)	0.0220 (9)
H2A	0.606793	0.913742	0.449679	0.026*
C3	0.56676 (12)	0.6998 (7)	0.5645 (5)	0.0228 (9)
H3A	0.544076	0.792307	0.548176	0.027*
C4	0.56291 (12)	0.4883 (6)	0.6485 (5)	0.0220 (9)
C5	0.59657 (12)	0.3501 (6)	0.6724 (4)	0.0197 (9)
H5	0.593559	0.208760	0.728769	0.024*
C6	0.63526 (12)	0.4193 (6)	0.6132 (5)	0.0181 (8)
C7	0.66957 (12)	0.2649 (6)	0.6383 (5)	0.0184 (8)
H7	0.665247	0.124258	0.694014	0.022*
C8	0.77559 (12)	0.2212 (6)	0.5898 (5)	0.0199 (8)
C9	0.84558 (12)	0.0651 (6)	0.6084 (5)	0.0211 (8)
C10	0.86736 (12)	0.2567 (6)	0.6646 (4)	0.0202 (9)
H10	0.853603	0.385403	0.705996	0.024*
C11	0.90932 (13)	0.2564 (7)	0.6592 (5)	0.0257 (9)
H11	0.923584	0.384325	0.698650	0.031*
C12	0.93050 (13)	0.0662 (7)	0.5954 (5)	0.0295 (10)
H12	0.958723	0.066914	0.591294	0.035*
C13	0.90894 (14)	−0.1236 (7)	0.5381 (5)	0.0276 (10)
H13	0.922824	−0.251318	0.495698	0.033*
C14	0.86663 (13)	−0.1254 (6)	0.5433 (5)	0.0230 (9)
H14	0.852420	−0.253328	0.503507	0.028*
O3	0.75611 (9)	0.7192 (4)	0.2769 (3)	0.0272 (7)
H3B	0.766837	0.640466	0.361224	0.041*
H3C	0.747314	0.620749	0.201607	0.041*

References

1. Bruker. *SAINT and SADABS*; Bruker AXS Inc.: Madison, Wisconsin, USA, 2000.
2. 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.
3. Sheldrick G. M. Crystal structure refinement with *SHELXL*. *Acta Crystallogr.* 2015, C71, 3–8.
4. Brandenburg K. *DIAMOND. Visual Crystal Structure Information System. Ver. 4.0*; Crystal Impact: Bonn, Germany, 2015.
5. Taghizadeh M.-J., Karimi H., Mirzaie A. Zeolite-encapsulated Mn(II) complex: a new nano catalyst for the synthesis of modafinil and its derivatives. *Lett. Org. Chem.* 2019, 16, 541–549.
6. Muluk M.-B., Dhupal S.-T., Rehman N.-N.-M.-A., Dixit P.-P., Kharat K.-R., Haval K.-P. Synthesis, anticancer and antimicrobial evaluation of new (*E*)-benzylidene-2-(2-ethylpyridin-4-yl)-4-methylthiazole-5-carbohydrazides. *ChemistrySelect* 2019, 4, 8993–8997.
7. Aradhyula B.-P.-R., Joshi N., Poluri K.-M., Kollipara M.-R. Synthesis and antibacterial studies of rhodium and iridium complexes comprising of dipyrindyl hydrazones. *J. Mol. Struct.* 2018, 1164, 191–199.
8. Ling L., Hu J.-F., Zhang H. Ferrocene containing *N*-tosyl hydrazones as optical and electrochemical sensors for Hg²⁺, Cu²⁺ and F[−] ions. *Tetrahedron* 2019, 75, 2472–2481.
9. Tai X.-S., Meng Q.-G., Liu L.-L. Synthesis, crystal structure, and cytotoxic activity of a novel eight-coordinated dinuclear Ca(II)-schiff base complex. *Crystals* 2016, 6, 109.
10. Tai X.-S., Wang L.-H., Liu L.-L., Zhou X.-J. The crystal structure of (*E*)-*N'*-((2-hydroxynaphthalen-1-yl)methylene)-2-phenylacetohydrazide. C₁₉H₁₆O₂N₂. *Z. Kristallogr. NCS* 2019, 234, 723–724.
11. Tai X.-S., Li P.-F. The crystal structure (*E*)-4-methyl-*N'*-(2-nitrobenzylidene)benzenesulfonylhydrazide, C₁₄H₁₃N₃O₄S. *Z. Kristallogr. NCS* 2017, 232, 1031–1032.