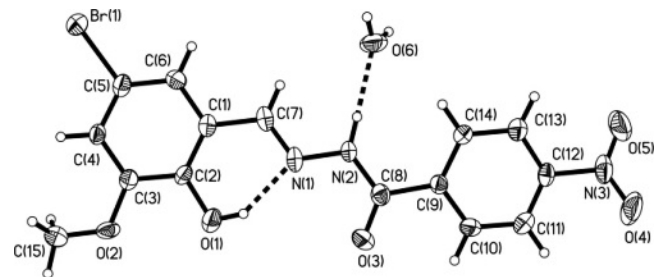


Crystal structure of *N'*-(5-bromo-2-hydroxy-3-methoxybenzylidene)-4-nitrobenzohydrazide monohydrate, $C_{15}H_{12}BrN_3O_5 \cdot H_2O$, $C_{15}H_{14}BrN_3O_6$

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Received February 21, 2012, accepted July 19, 2012, available online September 28, 2012, CCDC no. 1267/3833



Abstract

$C_{15}H_{14}BrN_3O_6$, monoclinic, $P2_1/n$ (no. 14), $a = 4.546(2)$ Å, $b = 13.831(3)$ Å, $c = 26.124(2)$ Å, $\beta = 91.146(2)^\circ$, $V = 1642.1$ Å³, $Z = 4$, $R_{gt}(F) = 0.0704$, $wR_{ref}(F^2) = 0.1827$, $T = 298$ K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.17×0.17×0.18 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	25.42 cm ⁻¹
Diffractometer, scan mode:	Bruker SMART 1000 CCD, ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11647, 3066
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1729
$N(param)_{refined}$:	238
Programs:	SHELX [12]

Source of material

5-Bromo-3-methoxysalicylaldehyde (0.23 g, 1 mmol) and 4-nitrobenzohydrazide (0.18 g, 1 mmol) were stirred at room temperature in MeOH (25 mL) for 30 min. Colourless block crystals were formed by slow evaporation of the solution in air for a week.

Experimental details

The amino and water H atoms were located from a difference Fourier map and refined isotropically, with the N–H, O–H, and H···H distances restrained to 0.90(1), 0.85(1), and 1.37(2) Å, respectively. The remaining hydrogen atoms were constrained to ideal geometries, with C–H distances of 0.93–0.96 Å, O–H distance of 0.82 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$ and $1.5U_{eq}(O1 \text{ and } C15)$.

Discussion

Hydrazone derivatives have been widely investigated for their biological properties [1–4]. In recent years, the crystal structures of a number of hydrazone derivatives have been reported [5–8]. In the present work, a new hydrazone derivative, *N'*-(5-bromo-2-hydroxy-3-methoxybenzylidene)-4-nitrobenzohydrazide

monohydrate (see the Figure), was prepared and structurally characterized. The compound contains a hydrazone molecule and a water molecule of crystallization. The hydrazone molecule is approximately coplanar with mean deviation from the plane defined by the non-hydrogen atoms of 0.070(2) Å. The dihedral angle between the two substituted benzene rings is 6.9(3)°. The intramolecular O(1)–H(1)···N(1) hydrogen bond may contribute to the planarity of the molecule. All the bond values in the compound are similar to those reported in the hydrazone derivatives [9–11]. The crystal structure is stabilized by intermolecular N–H···O and O–H···O hydrogen bonds.

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	4e	0.2987	0.8583	0.2033	0.095
H(4)	4e	−0.3761	0.7529	0.0599	0.050
H(6)	4e	0.1110	0.5652	0.1431	0.062
H(7)	4e	0.4360	0.6190	0.2128	0.057
H(10)	4e	1.0446	0.9034	0.3691	0.067
H(11)	4e	1.3639	0.8727	0.4366	0.073
H(13)	4e	1.4031	0.5956	0.3939	0.067
H(14)	4e	1.0785	0.6275	0.3262	0.061
H(15A)	4e	−0.3475	0.9122	0.0389	0.097
H(15B)	4e	−0.4433	1.0026	0.0707	0.097
H(15C)	4e	−0.5931	0.9022	0.0799	0.097
H(2)	4e	0.74(1)	0.664(1)	0.270(2)	0.05(2)
H(6B)	4e	0.81(2)	0.485(4)	0.259(2)	0.08
H(6A)	4e	0.70(2)	0.489(4)	0.307(2)	0.08

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Br(1)	4e	−0.2933(2)	0.54771(6)	0.05474(3)	0.0945(7)	0.0501(5)	0.0692(6)	−0.0056(5)	−0.0310(4)	−0.0058(4)
N(1)	4e	0.498(1)	0.7513(4)	0.2324(2)	0.040(3)	0.068(4)	0.036(3)	0.005(3)	−0.009(3)	0.006(3)
N(2)	4e	0.700(1)	0.7272(4)	0.2701(2)	0.045(3)	0.046(4)	0.039(3)	0.002(3)	−0.014(3)	0.003(3)
N(3)	4e	1.607(1)	0.7108(6)	0.4648(2)	0.056(4)	0.099(6)	0.047(4)	0.017(4)	−0.018(3)	−0.010(4)
O(1)	4e	0.174(1)	0.8761(4)	0.1822(2)	0.059(3)	0.068(3)	0.062(3)	0.001(3)	−0.021(3)	−0.010(3)
O(2)	4e	−0.207(1)	0.9131(3)	0.1075(2)	0.044(3)	0.033(3)	0.103(4)	0.001(2)	−0.029(3)	−0.012(3)
O(3)	4e	0.740(1)	0.8830(4)	0.2940(2)	0.104(4)	0.045(3)	0.065(4)	0.011(3)	−0.023(3)	0.007(3)
O(4)	4e	1.722(1)	0.7776(5)	0.4868(2)	0.099(5)	0.125(6)	0.070(4)	−0.016(4)	−0.042(4)	−0.011(4)
O(5)	4e	1.644(2)	0.6269(6)	0.4767(3)	0.148(7)	0.110(6)	0.092(5)	0.050(5)	−0.067(5)	−0.010(4)
C(1)	4e	0.161(1)	0.7055(5)	0.1661(2)	0.038(4)	0.057(4)	0.035(4)	0.001(3)	−0.002(3)	−0.001(3)
C(2)	4e	0.074(1)	0.7989(5)	0.1551(2)	0.042(4)	0.045(4)	0.037(4)	−0.007(3)	−0.007(3)	−0.007(3)
C(3)	4e	−0.134(1)	0.8175(5)	0.1161(2)	0.044(4)	0.047(4)	0.039(4)	−0.003(3)	−0.002(3)	0.000(3)
C(4)	4e	−0.242(1)	0.7415(5)	0.0866(2)	0.040(4)	0.046(4)	0.038(4)	−0.007(3)	−0.012(3)	0.002(3)
C(5)	4e	−0.148(1)	0.6486(5)	0.0976(3)	0.048(4)	0.057(5)	0.043(4)	−0.004(3)	−0.009(3)	−0.002(3)
C(6)	4e	0.051(1)	0.6284(5)	0.1365(3)	0.054(4)	0.039(4)	0.061(5)	0.002(3)	−0.011(4)	0.003(3)
C(7)	4e	0.382(1)	0.6828(5)	0.2064(2)	0.038(4)	0.066(5)	0.038(4)	0.006(4)	−0.004(3)	0.009(3)
C(8)	4e	0.811(2)	0.7992(5)	0.2997(2)	0.052(4)	0.052(5)	0.035(4)	0.001(4)	−0.001(3)	0.004(3)
C(9)	4e	1.025(1)	0.7694(4)	0.3412(2)	0.039(4)	0.039(4)	0.038(4)	−0.005(3)	−0.003(3)	0.007(3)
C(10)	4e	1.115(2)	0.8409(5)	0.3742(3)	0.078(5)	0.035(4)	0.054(4)	0.000(4)	−0.013(4)	0.002(3)
C(11)	4e	1.305(2)	0.8233(6)	0.4146(3)	0.065(5)	0.055(5)	0.062(5)	−0.009(4)	−0.017(4)	−0.009(4)
C(12)	4e	1.404(1)	0.7318(5)	0.4213(2)	0.043(4)	0.060(5)	0.031(4)	−0.003(3)	−0.007(3)	0.003(3)
C(13)	4e	1.327(2)	0.6574(5)	0.3889(3)	0.061(5)	0.055(5)	0.050(4)	−0.001(4)	−0.015(4)	0.001(4)
C(14)	4e	1.135(2)	0.6768(5)	0.3485(3)	0.058(4)	0.043(4)	0.050(4)	0.004(3)	−0.015(3)	−0.009(3)
C(15)	4e	−0.412(2)	0.9340(5)	0.0717(3)	0.082(6)	0.047(5)	0.064(5)	−0.009(4)	−0.005(4)	0.009(4)
O(6)	4e	0.746(2)	0.5228(3)	0.2814(2)	0.129(6)	0.037(3)	0.072(4)	−0.009(3)	−0.012(4)	−0.002(3)

Acknowledgments. The author acknowledges the Eastern Liaoning University for funding this study.

References

- El-Dissouky, A.; Al-Fulaij, O.; Awad, M. K.; Rizk, S.: Synthesis, characterization, and biological activity studies of copper(II)-metal(II) binuclear complexes of dipyritylglyoxal *bis*(2-hydroxybenzoyl hydrazone). *J. Coord. Chem.* **63** (2010) 330-345.
- El-Sherif, A. A.: Synthesis, spectroscopic characterization and biological activity on newly synthesized copper(II) and nickel(II) complexes incorporating bidentate oxygen-nitrogen hydrazone ligands. *Inorg. Chim. Acta* **362** (2009) 4991-5000.
- Abu Affan, M.; Foo, S. W.; Jusoh, I.; Hanapi, S.; Tiekink, E. R. T.: Synthesis, characterization and biological studies of organotin(IV) complexes with hydrazone ligand. *Inorg. Chim. Acta* **362** (2009) 5031-5037.
- Horiuchi, T.; Nagata, M.; Kitagawab, M.; Akahane, K.; Uoto, K.: Discovery of novel thieno[2,3-d]pyrimidin-4-yl hydrazone-based inhibitors of Cyclin D1-CDK4: Synthesis, biological evaluation and structure-activity relationships. *Bioorg. Med. Chem.* **17** (2009) 7850-7860.
- Wang, X.-L.; You, Z.-L.; Wang, C.: Syntheses, characterization and crystal structures of two isostructural hydrazone compounds *N'*-(3-bromo-5-chloro-2-hydroxybenzylidene)-4-chlorobenzohydrazide methanol solvate and *N'*-(3,5-dichloro-2-hydroxybenzylidene)-4-chlorobenzohydrazide methanol solvate. *J. Chem. Crystallogr.* **41** (2011) 621-624.
- Peng, S.-J.: Synthesis, Crystal structures, and antibacterial activity of two hydrazone derivatives 3-methoxy-*N'*-(3,5-dibromo-2-hydroxybenzylidene)benzohydrazide methanol solvate and 3-methoxy-*N'*-(2,4-dichlorobenzylidene)benzohydrazide. *J. Chem. Crystallogr.* **41** (2011) 280-285.
- Loh, W.-S.; Fun, H.-K.; Kayarmar, R.; Viveka, S.; Nagaraja, G. K.: 4-Chlorobenzaldehyde (1-isobutyl-1H-imidazo[4,5-c]quinolin-4-yl)hydrazone monohydrate. *Acta Crystallogr. E* **67** (2011) o407.
- Diao, Y.-P.; Huang, S.-S.; Zhang, H.-L.; Kang, T.-G.: Crystal structure of 2,4-dihydroxybenzoic acid [1-(3,5-dichloro-2-hydroxyphenyl)-methylidene]hydrazide, C₁₄H₁₀Cl₂N₂O₄. *Z. Kristallogr. NCS* **223** (2008) 165-166.
- Zareef, M.; Iqbal, R.; Qadeer, G.; Wong, W.-Y.; Akhtar, H.; Arfan, M.: Crystal structure of 2-bromobenzoic acid hydrazide, C₇H₇BrN₂O. *Z. Kristallogr. NCS* **221** (2006) 305-306.
- Fun, H.-K.; Loh, W. S.; Nayak, S. P.: 4-Methoxybenzaldehyde (5-bromopyrimidin-2-yl)hydrazone monohydrate. *Acta Crystallogr. E* **66** (2010) o2467.
- Pyta, K.; Przybylski, P.; Huczynski, A.; Hoser, A.; Wozniak, K.; Schilf, W.; Kamiński, B.; Grech, E.; Brzezinski, B.: X-ray, spectroscopic and computational studies of the tautomeric structure of a new hydrazone of 5-nitrosalicylaldehyde with indole-3-acetic hydrazide. *J. Mol. Struct.* **970** (2010) 147-154.
- Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr. A* **64** (2008) 112-122.