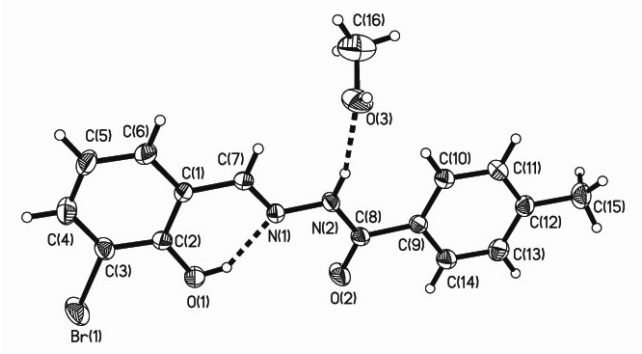
Crystal structure of *N*'-(3-bromo-2-hydroxybenzylidene)-4-methylbenzohydrazide methanol solvate, $C_{16}H_{17}BrN_2O_3$

Zeng-Xin Liu*

Experimental Center, Linyi University, Linyi 276005, Shandong Province, P. R. China

Received March 07, 2012, accepted June 12, 2012, available online August 09, 2012, CCDC no. 1267/3808



Abstract

$C_{16}H_{17}BrN_2O_3$, triclinic, $P\bar{1}$ (no. 2), a = 7.338(2) Å, b = 9.210(2) Å, c = 12.718(2) Å, α = 97.981(2)°, β = 99.889(2)°, γ = 100.486(2)°, V = 819.7 Å^3, Z = 2, $R_{gt}(F)$ = 0.0427, $wR_{ref}(F^2)$ = 0.1084, T = 298 K.

Table 1. Data collection and handling.

Crystal:	colourless needles, size 0.17×0.20×0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	25.21 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1K CCD, ω
$2\theta_{max}$:	51.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6340, 3167
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2058
$N(param)_{refined}$:	205
Programs:	SHELX [10]

Source of material

The title compound was synthesized by the reaction of 3-bromo-2-hydroxybenzaldehyde (1 mmol, 0.20 g) with 4-methylbenzohydrazide (1 mmol, 0.15 g) in absolute methanol (30 ml) at ambient condition. Colourless needle-shaped single crystals were obtained by slow evaporation of the solution at room temperature after several days.

Experimental details

The amide H atom was located in a difference map and was refined isotropically, with N–H = 0.90(1) Å. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms, with C–H = 0.93 Å for aromatic and CH and 0.96 Å for CH_3 atoms, and with O–H = 0.82 Å. The U_{iso} values were constrained to be 1.5U_{eq} of the carrier atom for methyl and hydroxyl H atoms and 1.2U_{eq} for the remaining H atoms. A rotating group model was used for the methyl groups.

Discussion

In the last few years, the compounds derived from the condensation reactions of carbonyl-containing compounds with substituted benzohydrazides have received considerable attention [1-4]. In this paper the title compound (Fig.), derived from the reaction of 3-bromo-2-hydroxybenzaldehyde with 4-methylbenzohydrazide, is reported.

The asymmetric unit of the compound contains one hydrazone molecule and a methanol molecule of crystallization. The hydrazone molecule displays a *trans*-configuration about the C(7)=N(1) bond. The torsion angle of C(7)–N(1)–N(2)–C(8) is 0.1(2)°. The dihedral angle between the C(1)–C(6) and C(9)–C(14) benzene rings is 2.5(2)°. This planarity is assisted by the formation of an intramolecular O(1)–H(1)···N(1) hydrogen bond. The bond distances are within normal values [5], and are comparable with those reported in similar compounds [6-9]. In the crystal, the methanol molecules are linked to the hydrazone molecules through intermolecular O–H···O and N–H···O hydrogen bonds.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.2772	0.3981	0.7648	0.079
H(3)	2*i*	0.9615	0.6401	0.6123	0.110
H(4)	2*i*	0.5426	0.1873	1.0854	0.069
H(5)	2*i*	0.8380	0.3012	1.0666	0.071
H(6)	2*i*	0.8679	0.4341	0.9285	0.061
H(7)	2*i*	0.7375	0.5357	0.7728	0.048
H(10)	2*i*	0.6540	0.7742	0.5194	0.055
H(11)	2*i*	0.6747	0.9185	0.3866	0.061
H(13)	2*i*	0.1144	0.8207	0.2929	0.064
H(14)	2*i*	0.0913	0.6768	0.4267	0.058
H(15A)	2*i*	0.2988	0.9563	0.1855	0.096
H(15B)	2*i*	0.5184	0.9687	0.2079	0.096
H(15C)	2*i*	0.4316	1.0872	0.2725	0.096
H(16A)	2*i*	0.9507	0.8659	0.7547	0.165
H(16B)	2*i*	1.0428	0.8891	0.6541	0.165
H(16C)	2*i*	1.1297	0.8009	0.7400	0.165
H(2)	2*i*	0.619(2)	0.630(4)	0.628(3)	0.080

* Correspondence author (e-mail: zengxin_liu@163.com)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	2*i*	0.15517(5)	0.15776(5)	0.97389(3)	0.0576(3)	0.0961(3)	0.0966(4)	0.0036(2)	0.0329(2)	0.0497(3)
N(1)	2*i*	0.4730(3)	0.5158(2)	0.7181(2)	0.036(1)	0.042(1)	0.038(1)	0.006(1)	0.006(1)	0.013(1)
N(2)	2*i*	0.5002(3)	0.5983(3)	0.6379(2)	0.032(1)	0.046(1)	0.042(1)	0.006(1)	0.010(1)	0.018(1)
O(1)	2*i*	0.2502(3)	0.3465(3)	0.8098(2)	0.030(1)	0.074(2)	0.056(1)	0.004(1)	0.005(1)	0.028(1)
O(2)	2*i*	0.1869(3)	0.5616(3)	0.5859(2)	0.031(1)	0.084(2)	0.080(2)	0.005(1)	0.010(1)	0.045(1)
O(3)	2*i*	0.8988(3)	0.6889(3)	0.6447(2)	0.037(1)	0.083(2)	0.100(2)	0.008(1)	0.021(1)	0.020(2)
C(1)	2*i*	0.5905(4)	0.4071(3)	0.8656(2)	0.035(2)	0.045(2)	0.035(2)	0.006(1)	0.004(1)	0.011(1)
C(2)	2*i*	0.4095(4)	0.3372(3)	0.8763(2)	0.033(2)	0.044(2)	0.041(2)	0.004(1)	0.007(1)	0.008(1)
C(3)	2*i*	0.3970(4)	0.2548(3)	0.9592(2)	0.043(2)	0.052(2)	0.048(2)	0.008(2)	0.016(2)	0.017(2)
C(4)	2*i*	0.5553(5)	0.2421(4)	1.0299(3)	0.063(2)	0.071(2)	0.048(2)	0.020(2)	0.016(2)	0.026(2)
C(5)	2*i*	0.7308(5)	0.3096(4)	1.0189(2)	0.047(2)	0.085(3)	0.048(2)	0.016(2)	–0.002(2)	0.028(2)
C(6)	2*i*	0.7478(4)	0.3900(4)	0.9367(2)	0.035(2)	0.068(2)	0.048(2)	0.005(2)	0.003(1)	0.016(2)
C(7)	2*i*	0.6162(4)	0.4949(3)	0.7810(2)	0.029(2)	0.049(2)	0.041(2)	0.002(1)	0.006(1)	0.012(1)
C(8)	2*i*	0.3429(4)	0.6173(3)	0.5735(2)	0.033(2)	0.041(2)	0.043(2)	0.006(1)	0.006(1)	0.012(1)
C(9)	2*i*	0.3702(4)	0.7110(3)	0.4880(2)	0.035(2)	0.037(2)	0.040(2)	0.006(1)	0.006(1)	0.010(1)
C(10)	2*i*	0.5442(4)	0.7837(3)	0.4744(2)	0.039(2)	0.051(2)	0.046(2)	0.005(1)	0.006(1)	0.015(2)
C(11)	2*i*	0.5563(5)	0.8703(3)	0.3946(2)	0.050(2)	0.049(2)	0.051(2)	–0.002(2)	0.013(2)	0.013(2)
C(12)	2*i*	0.3964(5)	0.8871(3)	0.3264(2)	0.061(2)	0.040(2)	0.040(2)	0.007(2)	0.007(2)	0.009(1)
C(13)	2*i*	0.2238(5)	0.8127(4)	0.3389(3)	0.052(2)	0.056(2)	0.052(2)	0.015(2)	0.001(2)	0.017(2)
C(14)	2*i*	0.2097(4)	0.7256(3)	0.4192(2)	0.041(2)	0.051(2)	0.051(2)	0.005(1)	0.005(2)	0.015(2)
C(15)	2*i*	0.4128(5)	0.9836(4)	0.2402(3)	0.087(3)	0.057(2)	0.053(2)	0.015(2)	0.015(2)	0.022(2)
C(16)	2*i*	1.0143(6)	0.8213(5)	0.7028(4)	0.071(3)	0.113(4)	0.130(4)	0.001(3)	0.013(3)	0.001(3)

Acknowledgments. The author thanks the Experimental Center of Linyi University for suports.

References

1. Dinda, R.; Sengupta, P.; Ghosh, S.; Mayer-Figge, H.; Sheldrick, W. S.: A family of mononuclear molybdenum-(VI), and -(IV) oxo complexes with a tridentate (ONO) ligand. J. Chem. Soc., Dalton Trans. (2002) 4434-4439.
2. Pouralimardan, O.; Chamayou, A.-C.; Janiak, C.; Hosseini-Monfared, H.: Hydrazone Schiff base-manganese(II) complexes: Synthesis, crystal structure and catalytic reactivity. Inorg. Chim. Acta **360** (2007) 1599-1608.
3. Bessy, R. B.; Prathapachandra, K. M. R.; Suresh, E.: Synthesis, spectroscopic characterization and crystal structure of mixed ligand Ni(II) complex of *N*-4-diethylaminosalicylidine-N'-4-nitrobenzoyl hydrazone and 4-picoline. Struct. Chem. **17** (2006) 201-208.
4. Rauf, A.; BAnday, M. R.; Mattoo, R. H.: Synthesis, characterization and antimicrobial activity of long-chain hydrazones. Acta Chim. Slov. **55** (2008) 448-452.
5. Allen, F. H.; Kennard, O.; Watson, D. G.; Brammer, L.; Orphen, A. G.; Taylor, R. Tables of bond lengths determined by *X*-ray and neutron-diffraction. 1. Bond lengths in organic-compounds. J. Chem. Soc. Perkin Trans. **2** (1987) S1-S19.
6. Peng, S.-J.: Synthesis, Crystal structures, and antibacterial activity of two hydrazone derivatives 3-methoxy-*N*'-(3,5-dibromo-2-hydroxy benzylidene)benzohydrazide methanol solvate and 3-methoxy-*N*'-(2,4-diclorobenzylidene)benzohydrazide. J. Chem. Crystallogr. **41** (2011) 280-285.
7. 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.
8. 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, $C_{14}H_{10}Cl_2N_2O_4$. Z. Kristallogr. NCS **223** (2008) 165-166.
9. Zareef, M.; Iqbal, R.; Qadeer, G.; Wong, W.-Y.; Akhtar, H.; Arfan, M.: Crystal structure of 2-bromobenzoic acid hydrazide, $C_7H_7BrN_2O$. Z. Kristallogr. NCS **221** (2006) 305-306.
10. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.