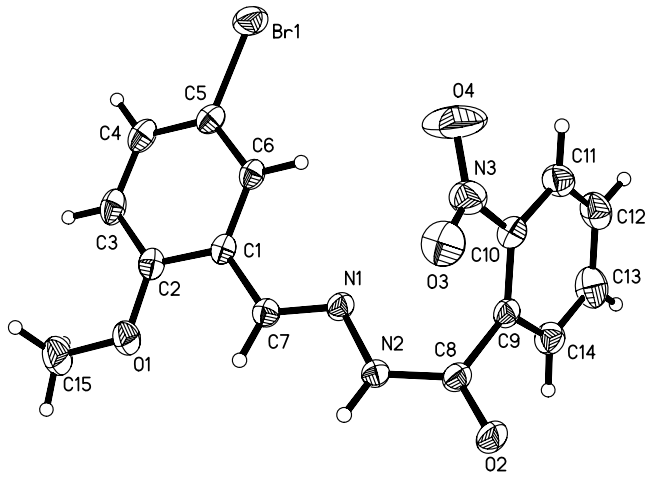
Crystal structure of 2-nitro-*N′*-(5-bromo-2-methoxybenzylidene)-benzohydrazide, $C_{15}H_{12}BrN_3O_4$

Congshan Zhou* and Tao Yang

Hunan Institute of Science and Technology, Department of Chemistry and Chemical Engineering, Yueyang 414006, P. R. .China

Received September 9, 2008, accepted and available on-line November 16, 2008; CCDC no. 1267/2453



Abstract

$C_{15}H_{12}BrN_3O_4$, monoclinic, $P12_1/c1$ (no. 14), a = 11.3077(3) Å, b = 7.4907(2) Å, c = 18.3286(5) Å, β = 90.124(1)°, V = 1552.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.037, $wR_{ref}(F^2)$ = 0.102, T = 298 K.

Source of material

Both starting materials of AR grade were purchased from Aldrich and were used as obtained. 5-Bromo-2-methoxybenzaldehyde (215.1 mg, 1.0 mmol) and 2-nitrobenzohydrazide (181.2 mg, 1.0 mmol) were dissolved in a methanol solution (50 ml). The mixture was stirred for 30 min at room temperature. The resulting solution was left in air for a few days, yielding colorless block-shaped crystals.

Experimental details

H2 atom was located in a difference map and refined with N—H distance restrained to 0.90(1) Å. The remaining H atoms were positioned geometrically [d(C—H) = 0.93 - 0.96 Å] and refined using a riding model, with U_{iso}(H) = 1.2 U_{eq}(C) and 1.5 U_{eq}(O). A rotating group model was used for the methyl group.

Discussion

Schiff bases are a kind of versatile compounds, which possess excellent biological properties [1-3]. Recently, a large number of Schiff bases derived from the reaction of aldehydes with benzohydrazides have been reported [4-6].

In the crystal structure of the title compound, all the bond lengths are comparable with those observed in other similar compounds [4-8]. The dihedral angle between two substituted benzene rings is 73.0(2)°. The dihedral angle between the O3/N3/O4 plane and the C9-C14 benzene ring is 20.2(2)°. The C15 atom lies in the least-squares plane of C1-C6, with mean deviation from the plane of 0.001(2) Å. The torsion angles of C7-N1-N2-C8 and N1-N2-C8-C9 are 1.9(2) and 11.5(2)°, respectively. The crystal structure is stabilized by intermolecular N–H···O hydrogen bonds.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.28 × 0.28 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	26.72 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, ω
$2\theta_{max}$:	54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9004, 3371
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2326
$N(param)_{refined}$:	212
Programs:	SHELXS-97 [9], SHELXL-97 [10], SHELXTL [11]

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

Atom	Site	x	y	z	U_{iso}
H(3)	4*e*	–0.1658	0.2121	–0.1173	0.075
H(4)	4*e*	–0.2507	0.3247	–0.0124	0.074
H(6)	4*e*	0.0612	0.3034	0.0893	0.056
H(7)	4*e*	0.2217	0.0929	–0.0533	0.056
H(11)	4*e*	0.2996	0.5027	0.2658	0.076
H(12)	4*e*	0.3969	0.7608	0.2381	0.087
H(13)	4*e*	0.5218	0.7750	0.1392	0.094
H(14)	4*e*	0.5526	0.5265	0.0682	0.071
H(15A)	4*e*	–0.0712	0.0072	–0.1914	0.134
H(15B)	4*e*	0.0437	0.0405	–0.2372	0.134
H(15C)	4*e*	–0.0396	0.2006	–0.2175	0.134
H(2)	4*e*	0.395(3)	0.066(3)	–0.005(1)	0.080

* Correspondence author (e-mail: zhoucongsh@gmail.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)	4*e*	–0.17214(3)	0.41529(5)	0.13373(2)	0.0509(2)	0.0949(3)	0.0716(2)	0.0093(2)	0.0081(1)	0.0009(2)
O(1)	4*e*	0.0572(2)	0.1179(3)	–0.1353(1)	0.068(1)	0.083(2)	0.051(1)	–0.008(1)	–0.013(1)	–0.008(1)
O(2)	4*e*	0.5630(2)	0.1377(3)	0.0703(1)	0.037(1)	0.077(1)	0.091(2)	0.013(1)	–0.007(1)	–0.031(1)
O(3)	4*e*	0.3406(3)	0.0567(3)	0.1784(2)	0.099(2)	0.054(1)	0.097(2)	–0.008(1)	0.003(2)	0.010(1)
O(4)	4*e*	0.2187(4)	0.2088(5)	0.2390(2)	0.161(3)	0.133(3)	0.192(4)	–0.059(3)	0.125(3)	–0.047(3)
N(1)	4*e*	0.2653(2)	0.2213(3)	0.0326(1)	0.036(1)	0.048(1)	0.054(1)	0.005(1)	–0.0030(9)	–0.004(1)
N(2)	4*e*	0.3791(2)	0.1547(3)	0.0263(1)	0.037(1)	0.057(1)	0.057(1)	0.007(1)	–0.002(1)	–0.016(1)
N(3)	4*e*	0.3055(3)	0.1969(4)	0.2006(2)	0.074(2)	0.068(2)	0.068(2)	–0.012(2)	0.014(1)	–0.004(1)
C(1)	4*e*	0.0693(2)	0.2147(3)	–0.0146(1)	0.041(1)	0.044(1)	0.051(2)	–0.005(1)	–0.007(1)	0.005(1)
C(2)	4*e*	–0.0009(2)	0.1870(4)	–0.0768(2)	0.053(2)	0.049(2)	0.051(2)	–0.008(1)	–0.008(1)	0.005(1)
C(3)	4*e*	–0.1198(3)	0.2292(4)	–0.0758(2)	0.053(2)	0.072(2)	0.062(2)	–0.010(2)	–0.024(1)	0.008(2)
C(4)	4*e*	–0.1706(3)	0.2969(4)	–0.0131(2)	0.040(2)	0.068(2)	0.077(2)	–0.003(1)	–0.012(1)	0.010(2)
C(5)	4*e*	–0.1024(2)	0.3230(4)	0.0481(2)	0.043(1)	0.053(2)	0.059(2)	–0.000(1)	–0.002(1)	0.008(1)
C(6)	4*e*	0.0160(2)	0.2837(4)	0.0476(1)	0.039(1)	0.051(2)	0.051(2)	–0.002(1)	–0.009(1)	0.006(1)
C(7)	4*e*	0.1938(2)	0.1659(4)	–0.0160(1)	0.045(1)	0.049(2)	0.047(1)	0.000(1)	–0.002(1)	–0.002(1)
C(8)	4*e*	0.4646(2)	0.2049(4)	0.0723(2)	0.037(1)	0.051(2)	0.060(2)	0.002(1)	0.000(1)	–0.008(1)
C(9)	4*e*	0.4407(2)	0.3612(4)	0.1220(1)	0.036(1)	0.051(2)	0.052(2)	0.007(1)	–0.007(1)	–0.003(1)
C(10)	4*e*	0.3664(2)	0.3598(4)	0.1808(2)	0.046(2)	0.052(2)	0.059(2)	–0.001(1)	–0.004(1)	0.002(1)
C(11)	4*e*	0.3496(3)	0.5084(5)	0.2256(2)	0.062(2)	0.064(2)	0.063(2)	0.016(2)	–0.003(2)	–0.013(2)
C(12)	4*e*	0.4077(3)	0.6602(5)	0.2092(2)	0.083(2)	0.059(2)	0.076(2)	0.013(2)	–0.014(2)	–0.020(2)
C(13)	4*e*	0.4830(4)	0.6689(5)	0.1501(2)	0.098(3)	0.052(2)	0.084(2)	–0.014(2)	–0.015(2)	–0.001(2)
C(14)	4*e*	0.5005(3)	0.5208(4)	0.1073(2)	0.057(2)	0.060(2)	0.061(2)	–0.009(2)	–0.000(1)	0.003(2)
C(15)	4*e*	–0.0077(4)	0.0892(6)	–0.2007(2)	0.094(3)	0.116(3)	0.058(2)	–0.019(2)	–0.018(2)	–0.014(2)

Acknowledgment. The author acknowledges the Project Supported by Scientific Research Fund of Hunan Provincial Education Department (grant no. 08B031), P. R. China.

References

1. Sinha, D.; Tiwari, A. K.; Singh, S.; Shukla, G.; Mishra, P.; Chandra, H.; Mishra, K.: Synthesis, characterization and biological activity of Schiff base analogues of indole-3-carboxaldehyde. Eur. J. Med. Chem. **43** (2008) 160-165.
2. Singh, B. N.; Shukla, S. K.; Singh, M.: Synthesis and biological activity of sulphadiazine Schiff's bases of isatin and their *N*-Mannich bases. Asian J. Chem. **19** (2007) 5013-5018.
3. Singh, K.; Barwa, M. S.; Tyagi, P.: Synthesis, characterization and biological studies of Co(II), Ni(II), Cu(II) and Zn(II) complexes with bidentate Schiff bases derived by heterocyclic ketone. Eur. J. Med. Chem. **41** (2006) 147-153.
4. Song, Q.-B.; Zhang, J.: Crystal structure of (1,8-diethyl-1,3,4,9-tetrahydro-pyrano[3,4-b]indol-1-yl)acetic acid (2-hydroxy-benzylidene)-hydrazide, $C_{24}H_{27}N_3O_3$. Z. Kristallogr. NCS **221** (2006) 285-286.
5. Tai, X.-S.; He, Y.; Feng, Y.-M.: Crystal structure of (*E*)-*N*′-(4- hydroxy-benzylidene)benzohydrazide monohydrate, $C_{14}H_{12}N_2O_2 \cdot H_2O$. Z. Kristallogr. NCS **222** (2007) 343-344.
6. Yang, D.-S.: Syntheses, characterization and crystal structures of two structurally similar Schiff bases isonicotinic acid [1-(3-methoxy-2-hydroxyphenyl)methylidene]hydrazide and isonicotinic acid [1-(4-dimethylaminophenyl)methylidene]hydrazide monohydrate. J. Chem. Crystallogr. **37** (2007) 343-348.
7. Yathirajan, H. S.; Sarojini, B. K.; Narayana, B.; Sunil, K.; Bolte, M.: 2-Bromo-5-methoxy-*N*′-[(*E*)-(2-nitrophenyl)methylene]benzohydrazide. Acta Crystallogr. **E63** (2007) o2719.
8. Salhin, A.; Tameem, A. A.; Saad, B.; Ng, S.-L.; Fun, H.-K.: 4-Hyroxy-*N*′-[(*E*)-(2-hydroxyphenyl)methylidene]benzohydrazide. Acta Crystallogr. **E63** (2007) o2880.
9. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
10. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
11. Sheldrick, G. M.: SHELXTL. Structure Determination Software Suite. Version 6.14. Bruker AXS, Madison, Wisconsin, USA 2000.