Jin-Xia Ma, Ping Wei, Qing-Lin Li, Tao Fu, Ji-Xing Zhao and Li Zhao*

Crystal structure of (*E*)-1-(4-(((*E*)-2-bromo-6-hydroxybenzylidene)amino)phenyl)ethan-1-one *O*-methyl oxime, C₁₆H₁₅BrN₂O₂

<https://doi.org/10.1515/ncrs-2019-0435>

Received June 25, 2019; accepted August 18, 2019; available online August 23, 2019

Abstract

C₁₆H₁₅BrN₂O₂, monoclinic, *P*2₁ (no. 4), *a* = 10.9311(8) Å, *b* = 3.9554(3) Å, *c* = 16.5215(12) Å, β = 92.743(2)°, *V* = 713.52(9) Å³, *Z* = 2, *R*_{gt}(*F*) = 0.0335, *wR*_{ref}(*F*²) = 0.0655, *T* = 173(2) K.

CCDC no.: 1940969

The crystal 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.

Source of material

2-Bromo-6-hydroxy-benzaldehyde (200.3 mg, 1 mmol) dissolved in ethanol solution (3 mL) was added to 1-(4-aminophenyl)ethanone *O*-methyl oxime (164.5 mg, 1 mmol) dissolved in ethanol solution (3 mL). The solution had been stirred at 328 K. Five hours later, the mixture was filtered at room temperature. The filtrate was allowed to stand for 9 days at room temperature by slow evaporation. Several block clear light yellow crystals were obtained (yield 62.2%, m.p. 372–379 K). Anal. calcd. for C₁₆H₁₅BrN₂O₂: C, 55.35%; H, 4.35%; N, 8.07%; Found: C, 55.89%; H, 4.93%; N, 7.34%.

*Corresponding author: Li Zhao, School of Chemical and Biological Engineering, Lanzhou Jiaotong University, Lanzhou 730070, P.R. China, e-mail: zhaoli_72@163.com

Jin-Xia Ma, Ping Wei, Qing-Lin Li and Tao Fu: School of Chemical and Biological Engineering, Lanzhou Jiaotong University, Lanzhou 730070, P.R. China

Ji-Xing Zhao: Shihezi University, Analysis and Testing Center, Xinjiang 832003, P.R. China

Table 1: Data collection and handling.

Crystal:	Block
Size:	0.17 × 0.14 × 0.13 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	2.89 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ _{max} , completeness:	25.5°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	7892, 2622, 0.076
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2263
<i>N</i> (<i>param</i>) _{refined} :	194
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3]

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	−1.51230(4)	−1.15197(17)	−1.16170(3)	0.03161(16)
N2	−1.1824(4)	−1.7579(12)	−1.6462(3)	0.0336(13)
O1	−1.0669(3)	−1.6696(19)	−1.12664(18)	0.0345(8)
H1	−1.0855	−1.7202	−1.1750	0.052*
N1	−1.1922(3)	−1.664(2)	−1.2640(2)	0.0265(9)
C4	−1.3537(4)	−1.175(3)	−1.0228(3)	0.0302(12)
H4	−1.4188	−1.0609	−0.9981	0.036*
C12	−1.1362(4)	−1.7190(17)	−1.4818(3)	0.0284(17)
H12	−1.0730	−1.6595	−1.5167	0.034*
C8	−1.2116(4)	−1.7395(12)	−1.3479(3)	0.0238(14)
C5	−1.2458(5)	−1.2521(14)	−0.9784(3)	0.0348(17)
H5	−1.2372	−1.1873	−0.9231	0.042*
C13	−1.1188(4)	−1.669(3)	−1.3996(2)	0.0280(11)
H13	−1.0425	−1.5845	−1.3781	0.034*
C15	−1.3762(4)	−2.0781(16)	−1.6366(3)	0.0333(17)
H15A	−1.3635	−2.1507	−1.6923	0.050*
H15B	−1.4466	−1.9247	−1.6363	0.050*
H15C	−1.3919	−2.2763	−1.6031	0.050*
C11	−1.2453(5)	−1.8563(14)	−1.5146(3)	0.0241(12)
C7	−1.2805(4)	−1.5265(12)	−1.2269(3)	0.0228(12)
H7	−1.3556	−1.4828	−1.2564	0.027*
C9	−1.3190(5)	−1.8856(13)	−1.3787(3)	0.0254(12)
H9	−1.3814	−1.9464	−1.3433	0.030*
O2	−1.2111(4)	−1.7967(10)	−1.7298(2)	0.0446(12)
C16	−1.1181(5)	−1.618(3)	−1.7723(3)	0.0479(17)
H16A	−1.0371	−1.7065	−1.7554	0.072*
H16B	−1.1219	−1.3765	−1.7594	0.072*

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H16C	−1.1324	−1.6499	−1.8308	0.072*
C3	−1.3638(4)	−1.2658(13)	−1.1027(3)	0.0266(13)
C10	−1.3355(5)	−1.9431(14)	−1.4615(3)	0.0262(13)
H10	−1.4095	−2.0433	−1.4822	0.031*
C1	−1.1624(5)	−1.5079(14)	−1.0941(3)	0.0247(12)
C14	−1.2650(5)	−1.9002(14)	−1.6036(3)	0.0268(12)
C6	−1.1516(5)	−1.4201(15)	−1.0130(3)	0.0306(14)
H6	−1.0794	−1.4757	−0.9814	0.037*
C2	−1.2696(4)	−1.4360(14)	−1.1420(3)	0.0239(12)

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Schiff base compounds and their derivatives have been playing an important role in the development of coordination chemistry [4, 5]. So far, we have synthesized a variety of Schiff base type compounds and their derivatives [6–8]. The Schiff base ligands form a stable N₂O₂ type complexes with transition metals through the nitrogen and oxygen atoms [9–11]. Particular attention has been paid to the synthesis and the application of Schiff base type metal complexes.

The current work reports the synthesis of title compound. The single crystal structure of the title compound was determined by X-ray crystallography. All bond lengths are in normal ranges [6–8]. There is a strong intramolecular O1–H1···N1 hydrogen bond interaction ($d(N1 \cdots H1) = 1.846$, $d(O1-H1) = 0.84$ and $d(O1 \cdots N1) = 2.594$) in the title structure (*cf.* the figure).

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