Tai Xi-Shi*, Zhou Xiao-Jing, Liu Li-Li, Cao Shu-Hua and Wang Li-Hua

The crystal structure of (*E*)-2-(((2-(1*H*-indol-3-yl)ethyl)iminio)methyl)-6-bromophenolate, $C_{17}H_{15}N_2BrO$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.12 × 0.1 × 0.08 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	2.76 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω -scans
$\theta_{\max}$, completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6287, 2621, 0.036
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2215
$N(\text{param})_{\text{refined}}$:	190
Programs:	Bruker programs [1], OLEX2 [2], SHELX [3], DIAMOND [4]

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Abstract

$C_{17}H_{15}N_2BrO$, monoclinic, $P2_1/n$ (no. 14), $a = 8.9886(8)$ Å, $b = 8.2159(7)$ Å, $c = 20.164(2)$ Å, $\beta = 92.580(9)^\circ$, $V = 1487.6(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0370$, $wR_{\text{ref}}(F^2) = 0.0716$, $T = 293(2)$ K.

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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 materials

0.201 g 3-Bromo-2-hydroxybenzaldehyde (1.0 mmol) and 0.160 g tryptamin (1.0 mmol) were dissolved into water-ethanol (v:v = 1:3) solution at room temperature. After the mixture was stirred for 0.5 h, 0.102 g magnesium chloride

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.01093(3)	0.71888(4)	0.43099(2)	0.02124(12)
O1	0.1602(2)	0.7267(2)	0.29932(10)	0.0162(5)
N1	0.8847(3)	0.6616(3)	0.23417(12)	0.0157(6)
H1	0.947906	0.658987	0.267466	0.019*
N2	0.3544(3)	0.6513(3)	0.21366(12)	0.0141(6)
H2	0.276789	0.705076	0.224102	0.017*
C1	0.6801(3)	0.7261(4)	0.17092(15)	0.0139(7)
C2	0.7550(3)	0.7515(4)	0.23043(16)	0.0160(7)
H2A	0.723078	0.819791	0.263725	0.019*
C3	0.8961(3)	0.5768(4)	0.17582(15)	0.0134(7)
C4	0.7677(3)	0.6123(4)	0.13503(15)	0.0136(7)
C5	0.7512(3)	0.5417(4)	0.07184(15)	0.0166(7)
H5	0.668476	0.564892	0.044124	0.020*
C6	0.8598(4)	0.4375(4)	0.05173(16)	0.0224(8)
H6	0.849814	0.389411	0.010017	0.027*
C7	0.9858(3)	0.4023(4)	0.09310(16)	0.0215(8)
H7	1.057218	0.330888	0.078108	0.026*
C8	1.0064(3)	0.4704(4)	0.15503(16)	0.0174(7)
H8	1.090189	0.446843	0.182052	0.021*
C9	0.5341(3)	0.7949(4)	0.14547(15)	0.0168(7)
H9A	0.544085	0.832008	0.100221	0.020*
H9B	0.509991	0.888733	0.172146	0.020*
C10	0.4053(3)	0.6728(4)	0.14673(15)	0.0172(7)
H10A	0.323313	0.711241	0.117981	0.021*
H10B	0.437816	0.568835	0.129798	0.021*
C11	0.4157(3)	0.5583(4)	0.25873(15)	0.0150(7)
H11	0.497701	0.496423	0.247834	0.018*

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

Zhou Xiao-Jing, Liu Li-Li and Cao Shu-Hua: College of Chemistry and Chemical Engineering, Weifang University, Weifang, Shandong 261061, P.R. China

Wang Li-Hua: College of Information and Control Engineering, Weifang University, Weifang, Shandong 261061, P.R. China

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C12	0.3639(3)	0.5457(3)	0.32437(15)	0.0129(7)
C13	0.2326(3)	0.6338(4)	0.34117(15)	0.0135(7)
C14	0.1885(3)	0.6102(4)	0.40787(15)	0.0164(7)
C15	0.2665(3)	0.5151(4)	0.45276(16)	0.0195(7)
H15	0.233917	0.505002	0.495698	0.023*
C16	0.3954(3)	0.4322(4)	0.43483(16)	0.0195(8)
H16	0.448111	0.367470	0.465543	0.023*
C17	0.4424(3)	0.4483(4)	0.37139(16)	0.0172(7)
H17	0.527679	0.393752	0.359253	0.021*

hexahydrate (0.5 mmol) was added to the above solution. The mixture was stirred for 5 h at 75 °C. Then the reactant was filtered. The colorless crystals of (*E*)-2-(((2-(1*H*-indol-3-yl)ethyl)iminio)methyl)-6-bromophenolate were obtained by slow evaporation in 120 days.

Experimental details

The hydrogen atoms were positioned geometrically (C—H = 0.93–0.97 Å and N—H = 0.86 Å). Their *U*_{iso} values were set to 1.2*U*_{eq} of the parent atoms.

Comment

Over the past decades, many ylide compounds have been received considerable attention, because they display widespread applications in organic chemistry [5–8]. Our goal was to synthesize a new Schiff base ligand and its manganese(II) complex. However, the Mg(II) ion does not coordinate with the title compound. Only a new ylide compound, (*E*)-2-(((2-(1*H*-indol-3-yl)ethyl)iminio)methyl)-6-bromophenolate has been obtained.

In the title compound, the C11=N2 bond distance is 1.291(4) Å, which is shorter than those of C10–N2 (1.455(4) Å), C3–N1 (1.375(4) Å), C2–N1 (1.380(4) Å), indicating the C11=N2 bond is double bond which was expected. The dihedral angles of plane 1 (C1–C2–N1–C3–C4) and plane 2 (C3–C4–C5–C6–C7–C8), plane 1 (C1–C2–N1–C3–C4) and plane 3 (C12–C13–C14–C15–C16–C17), plane 2 (C3–C4–

C5–C6–C7–C8) and plane 3 (C12–C13–C14–C15–C16–C17) are 0.5°, 40.2° and 40.0°, respectively, indicating that the whole (*E*)-2-(((2-(1*H*-indol-3-yl)ethyl)iminio)methyl)-6-bromophenolate molecule is not planar (see the figure). Geometric parameters are all in the expected ranges [9].

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