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2-[(1,3-Benzothiazol-2-yl)iminomethyl]-4-bromophenol

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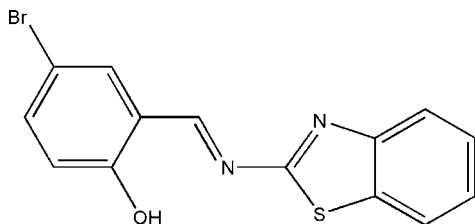
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.045; wR factor = 0.118; data-to-parameter ratio = 12.9.

In the title compound, $\text{C}_{14}\text{H}_9\text{BrN}_2\text{OS}$, the dihedral angle between the benzene rings is 3.1 (3)°. An intramolecular $\text{O}-\text{H}\cdots\text{N}$ (imine) hydrogen bond occurs. The crystal structure is stabilized by weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions.

Related literature

For the uses of Schiff bases, see: Da Silva *et al.* (2011); Dhar & Taploo (1982); Przybylski *et al.* (2009); Guo *et al.* (2007); Bringmann *et al.* (2004). For the structures of closely related imines, see: Liu *et al.* (2009); Asiri *et al.* (2010).



Experimental

Crystal data

$\text{C}_{14}\text{H}_9\text{BrN}_2\text{OS}$
 $M_r = 333.20$
 Monoclinic, $P2_1/c$
 $a = 26.1607$ (2) Å
 $b = 4.0565$ (2) Å
 $c = 12.1435$ (3) Å
 $\beta = 91.5720$ (1)°

$V = 1288.19$ (7) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 3.34$ mm⁻¹
 $T = 296$ K
 $0.45 \times 0.40 \times 0.38$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2001)
 $T_{\min} = 0.242$, $T_{\max} = 0.281$

11848 measured reflections
 2232 independent reflections
 1466 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.138$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.118$
 $S = 0.93$
 2232 reflections

173 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.36$ e Å⁻³
 $\Delta\rho_{\min} = -0.54$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.82	1.87	2.600 (6)	148
$\text{C7}-\text{H7}\cdots\text{O1}^{\text{i}}$	0.93	2.47	3.345 (6)	157

Symmetry code: (i) $x, -y + \frac{1}{2}, z + \frac{1}{2}$.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2345).

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2-[(1,3-Benzothiazol-2-yl)iminomethyl]-4-bromophenol

Hai-Peng Diao, Ti-Jian Sun and Wen Liu

S1. Comment

Schiff bases are some of the most widely used organic compounds. They are used as pigments and dyes, catalysts, intermediates in organic synthesis, and as polymer stabilisers (Da Silva *et al.*, 2011). They have also been shown to exhibit a broad range of biological activities, including antifungal, antibacterial, antimalarial, antiproliferative, anti-inflammatory, antiviral, and antipyretic properties (Dhar & Taploo, 1982; Przybylski *et al.*, 2009). The imine group present in such compounds has been shown to be critical to their biological activities (Guo *et al.*, 2007; Bringmann *et al.*, 2004). It was thus of interest to synthesize the title compound.

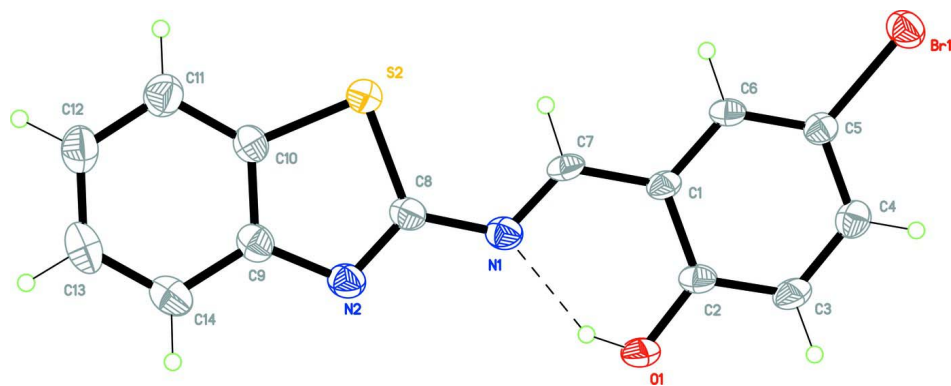
The X-ray structural analysis confirmed the assignment of the structure of the title compound (Fig. 1). The bond length of C8—N1 is 1.396 (6) Å, which is shorter than normal C—N [1.47 Å]. The dihedral angle between the two benzene rings (C1···C6 and C9···C14) is 3.1 (3)°, it is a little larger than 2.81 (9)° or 2.6 (1)° found in a related structure (Liu *et al.*, 2009; Asiri *et al.*, 2010). In the crystal structure (Fig. 2), the compound is further stabilized by intramolecular O—H···N and weak intermolecular C—H···O hydrogen-bond interactions.

S2. Experimental

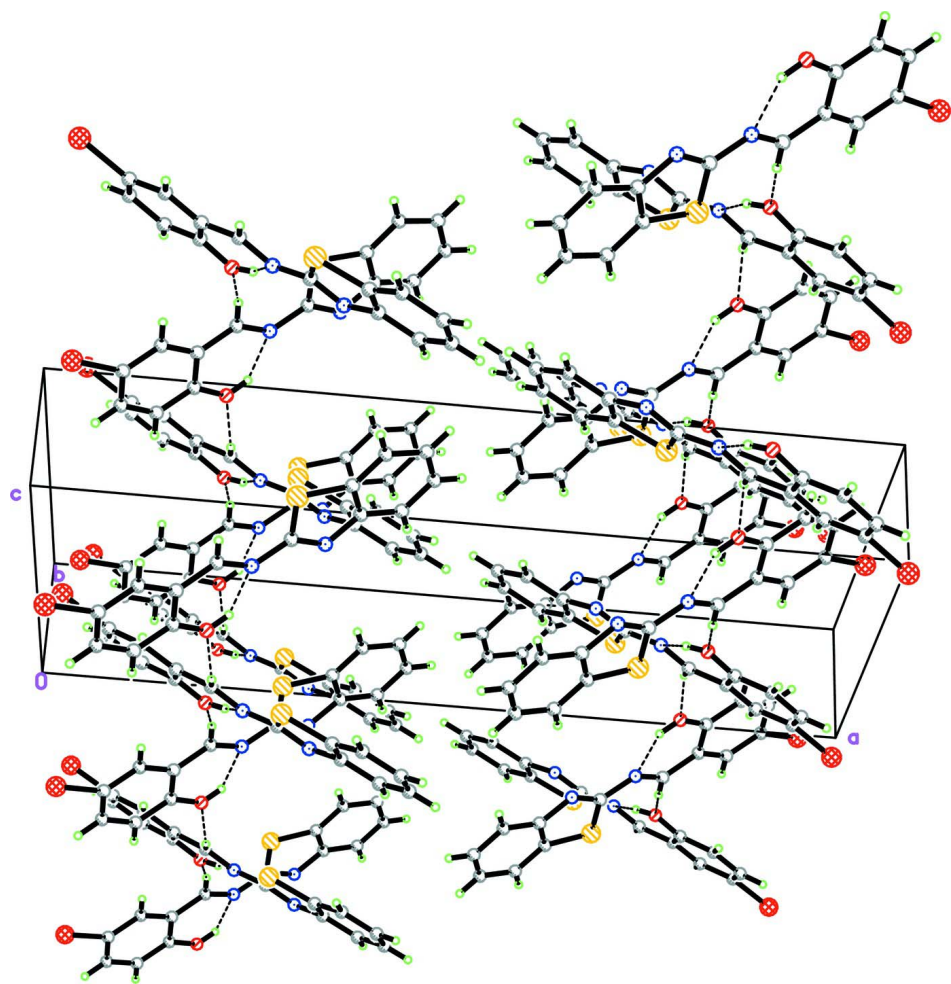
10 mL of 5 mmol 5-bromo-2-hydroxybenzaldehyde ethanol solution was added to 10 mL of the 5 mmol (0.7515 g) of 2-aminobenzothiazole ethanol solution. The resulting solution was refluxed for about 3 h, and then cooled to room temperature. Yellow crystals of title compound were obtained after 2 weeks of slow evaporation of the filtrate at room temperature.

S3. Refinement

H atoms attached to C and O atoms were placed in geometrically idealized positions with $C_{sp^2}-H = 0.93$ Å and $O-H = 0.82$ Å. The isotropic displacement parameters for H atoms were fixed as $U_{iso}(H) = 1.2U_{eq}(\text{carrier C atom})$ and $U_{iso}(H1) = 1.5U_{eq}(O1)$.

**Figure 1**

A view of the structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

**Figure 2**

Crystal packing of the title compound. Hydrogen bonds are shown as dashed lines.

2-[(1,3-Benzothiazol-2-yl)iminomethyl]-4-bromophenol

Crystal data

C₁₄H₉BrN₂OS $M_r = 333.20$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 26.1607 (2) \text{ \AA}$ $b = 4.0565 (2) \text{ \AA}$ $c = 12.1435 (3) \text{ \AA}$ $\beta = 91.5720 (1)^\circ$ $V = 1288.19 (7) \text{ \AA}^3$ $Z = 4$ $F(000) = 664$ $D_x = 1.718 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ $\theta = 3.4\text{--}21.4^\circ$ $\mu = 3.34 \text{ mm}^{-1}$ $T = 296 \text{ K}$

Block, yellow

 $0.45 \times 0.40 \times 0.38 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scansAbsorption correction: multi-scan
(SADABS; Bruker, 2001) $T_{\min} = 0.242$, $T_{\max} = 0.281$

11848 measured reflections

2232 independent reflections

1466 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.138$ $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 1.6^\circ$ $h = -30 \rightarrow 30$ $k = -4 \rightarrow 4$ $l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.045$ $wR(F^2) = 0.118$ $S = 0.93$

2232 reflections

173 parameters

0 restraints

0 constraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2)]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.36 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.54 \text{ e \AA}^{-3}$ Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.03658 (2)	1.69312 (15)	0.65038 (5)	0.0536 (2)
S2	0.29866 (5)	0.7609 (3)	0.70906 (12)	0.0488 (4)
C1	0.17288 (19)	1.3148 (12)	0.5361 (4)	0.0370 (11)
O1	0.20795 (15)	1.3280 (10)	0.3543 (3)	0.0582 (10)
H1	0.2300	1.2208	0.3877	0.087*
C12	0.4377 (2)	0.3239 (15)	0.7216 (6)	0.0633 (17)
H12	0.4606	0.2193	0.7700	0.076*
C4	0.0898 (2)	1.6691 (13)	0.4506 (4)	0.0458 (13)
H4	0.0620	1.7888	0.4225	0.055*
C6	0.13249 (18)	1.4049 (11)	0.6017 (4)	0.0372 (12)
H6	0.1335	1.3473	0.6758	0.045*
N2	0.33356 (16)	0.7896 (11)	0.5088 (4)	0.0471 (11)
N1	0.25338 (15)	1.0410 (11)	0.5215 (3)	0.0432 (10)

C2	0.17096 (19)	1.4091 (12)	0.4229 (4)	0.0399 (12)
C8	0.29517 (19)	0.8711 (13)	0.5676 (4)	0.0437 (13)
C5	0.09127 (19)	1.5761 (12)	0.5601 (4)	0.0381 (12)
C7	0.21554 (18)	1.1329 (12)	0.5801 (4)	0.0386 (12)
H7	0.2160	1.0782	0.6545	0.046*
C9	0.3704 (2)	0.6300 (13)	0.5745 (5)	0.0469 (14)
C3	0.1294 (2)	1.5847 (13)	0.3824 (4)	0.0501 (14)
H3	0.1279	1.6472	0.3087	0.060*
C11	0.3918 (2)	0.4411 (14)	0.7602 (5)	0.0596 (16)
H11	0.3837	0.4206	0.8340	0.071*
C10	0.35821 (19)	0.5906 (12)	0.6847 (4)	0.0432 (13)
C13	0.4499 (2)	0.3597 (14)	0.6131 (6)	0.0653 (18)
H13	0.4812	0.2816	0.5897	0.078*
C14	0.4168 (2)	0.5088 (15)	0.5376 (5)	0.0581 (16)
H14	0.4253	0.5279	0.4640	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0431 (3)	0.0658 (4)	0.0521 (4)	0.0039 (3)	0.0053 (2)	-0.0054 (3)
S2	0.0435 (8)	0.0614 (9)	0.0415 (8)	0.0046 (6)	0.0040 (6)	0.0028 (7)
C1	0.046 (3)	0.041 (3)	0.023 (3)	-0.001 (2)	-0.005 (2)	-0.003 (2)
O1	0.058 (2)	0.089 (3)	0.0284 (19)	0.013 (2)	0.0052 (18)	0.008 (2)
C12	0.046 (4)	0.064 (4)	0.080 (5)	0.001 (3)	-0.003 (3)	0.010 (4)
C4	0.043 (3)	0.050 (3)	0.044 (3)	-0.001 (3)	-0.010 (3)	-0.001 (3)
C6	0.042 (3)	0.045 (3)	0.025 (3)	-0.006 (2)	0.001 (2)	-0.004 (2)
N2	0.043 (3)	0.060 (3)	0.039 (3)	-0.004 (2)	0.002 (2)	-0.005 (2)
N1	0.040 (2)	0.054 (3)	0.036 (2)	-0.006 (2)	0.001 (2)	-0.004 (2)
C2	0.043 (3)	0.050 (3)	0.027 (3)	-0.007 (2)	0.001 (2)	-0.003 (2)
C8	0.039 (3)	0.054 (3)	0.038 (3)	-0.005 (3)	0.005 (2)	-0.004 (3)
C5	0.042 (3)	0.038 (3)	0.034 (3)	-0.003 (2)	-0.003 (2)	-0.002 (2)
C7	0.044 (3)	0.050 (3)	0.021 (3)	-0.008 (2)	-0.003 (2)	0.002 (2)
C9	0.039 (3)	0.050 (3)	0.052 (3)	-0.008 (3)	0.003 (3)	-0.004 (3)
C3	0.056 (4)	0.067 (4)	0.027 (3)	0.003 (3)	-0.007 (3)	0.005 (3)
C11	0.054 (4)	0.065 (4)	0.060 (4)	-0.001 (3)	-0.003 (3)	0.004 (3)
C10	0.036 (3)	0.043 (3)	0.051 (3)	-0.006 (2)	0.002 (3)	-0.002 (3)
C13	0.042 (3)	0.060 (4)	0.094 (5)	0.002 (3)	0.006 (4)	-0.012 (4)
C14	0.051 (4)	0.065 (4)	0.059 (4)	-0.008 (3)	0.012 (3)	-0.011 (3)

Geometric parameters (Å, °)

Br1—C5	1.887 (5)	C6—H6	0.9300
S2—C10	1.737 (5)	N2—C8	1.291 (6)
S2—C8	1.775 (5)	N2—C9	1.393 (7)
C1—C6	1.390 (6)	N1—C7	1.290 (6)
C1—C2	1.426 (6)	N1—C8	1.396 (6)
C1—C7	1.430 (7)	C2—C3	1.380 (7)
O1—C2	1.335 (5)	C7—H7	0.9300

O1—H1	0.8200	C9—C10	1.393 (7)
C12—C13	1.372 (9)	C9—C14	1.395 (7)
C12—C11	1.384 (8)	C3—H3	0.9300
C12—H12	0.9300	C11—C10	1.392 (7)
C4—C5	1.382 (7)	C11—H11	0.9300
C4—C3	1.387 (7)	C13—C14	1.382 (8)
C4—H4	0.9300	C13—H13	0.9300
C6—C5	1.367 (6)	C14—H14	0.9300
C10—S2—C8	87.6 (2)	C6—C5—Br1	121.1 (4)
C6—C1—C2	118.3 (5)	C4—C5—Br1	119.2 (4)
C6—C1—C7	121.3 (4)	N1—C7—C1	123.1 (4)
C2—C1—C7	120.4 (4)	N1—C7—H7	118.5
C2—O1—H1	109.5	C1—C7—H7	118.5
C13—C12—C11	121.0 (6)	C10—C9—N2	115.5 (5)
C13—C12—H12	119.5	C10—C9—C14	119.5 (6)
C11—C12—H12	119.5	N2—C9—C14	125.0 (5)
C5—C4—C3	120.3 (5)	C2—C3—C4	120.6 (5)
C5—C4—H4	119.8	C2—C3—H3	119.7
C3—C4—H4	119.8	C4—C3—H3	119.7
C5—C6—C1	121.8 (5)	C12—C11—C10	117.7 (6)
C5—C6—H6	119.1	C12—C11—H11	121.2
C1—C6—H6	119.1	C10—C11—H11	121.2
C8—N2—C9	109.8 (4)	C11—C10—C9	121.7 (5)
C7—N1—C8	121.7 (4)	C11—C10—S2	127.9 (4)
O1—C2—C3	118.9 (4)	C9—C10—S2	110.4 (4)
O1—C2—C1	121.9 (5)	C12—C13—C14	121.7 (6)
C3—C2—C1	119.2 (5)	C12—C13—H13	119.1
N2—C8—N1	121.2 (5)	C14—C13—H13	119.1
N2—C8—S2	116.7 (4)	C13—C14—C9	118.4 (6)
N1—C8—S2	122.1 (4)	C13—C14—H14	120.8
C6—C5—C4	119.7 (5)	C9—C14—H14	120.8
C2—C1—C6—C5	0.8 (7)	C8—N2—C9—C10	0.7 (6)
C7—C1—C6—C5	-179.3 (4)	C8—N2—C9—C14	-180.0 (5)
C6—C1—C2—O1	-179.1 (5)	O1—C2—C3—C4	178.9 (5)
C7—C1—C2—O1	1.1 (7)	C1—C2—C3—C4	0.0 (7)
C6—C1—C2—C3	-0.1 (7)	C5—C4—C3—C2	-0.4 (8)
C7—C1—C2—C3	-180.0 (5)	C13—C12—C11—C10	1.2 (9)
C9—N2—C8—N1	178.7 (4)	C12—C11—C10—C9	-1.6 (8)
C9—N2—C8—S2	-0.6 (6)	C12—C11—C10—S2	-179.6 (4)
C7—N1—C8—N2	-177.8 (5)	N2—C9—C10—C11	-178.8 (5)
C7—N1—C8—S2	1.5 (7)	C14—C9—C10—C11	1.8 (8)
C10—S2—C8—N2	0.3 (4)	N2—C9—C10—S2	-0.4 (6)
C10—S2—C8—N1	-179.0 (4)	C14—C9—C10—S2	-179.8 (4)
C1—C6—C5—C4	-1.3 (7)	C8—S2—C10—C11	178.3 (5)
C1—C6—C5—Br1	179.5 (4)	C8—S2—C10—C9	0.1 (4)
C3—C4—C5—C6	1.1 (7)	C11—C12—C13—C14	-1.0 (9)

C3—C4—C5—Br1	-179.7 (4)	C12—C13—C14—C9	1.2 (9)
C8—N1—C7—C1	178.3 (4)	C10—C9—C14—C13	-1.5 (8)
C6—C1—C7—N1	179.0 (4)	N2—C9—C14—C13	179.1 (5)
C2—C1—C7—N1	-1.1 (7)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O1—H1...N1	0.82	1.87	2.600 (6)	148
C7—H7...O1 ⁱ	0.93	2.47	3.345 (6)	157

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