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4-Bromo-2-[(phenylimino)methyl]phenol

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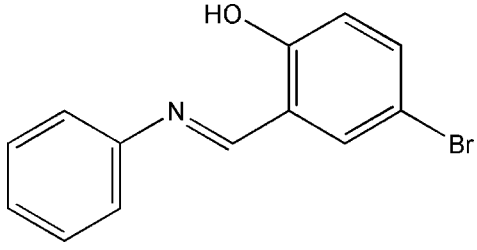
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.039; wR factor = 0.078; data-to-parameter ratio = 11.2.

The title compound, $C_{13}H_{10}BrNO$, is essentially planar (r.m.s. deviation = 0.026 Å) and the dihedral angle between the planes of the two aryl rings is 1.5 (3)°. An intramolecular O—H···N hydrogen bond generates an S(6) ring.

Related literature

For background to the biological activity of Schiff bases, see: Han *et al.* (2012); Rehman *et al.* (2008); Ritter *et al.* (2009); Vanco *et al.* (2008). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{13}H_{10}BrNO$
M_r = 276.13
Orthorhombic, $Pca2_1$
a = 12.353 (3) Å
b = 4.5092 (9) Å
c = 19.778 (4) Å
V = 1101.7 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.71 mm^{-1}
T = 298 K
0.20 × 0.15 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
T_{min} = 0.524, T_{max} = 0.836
4926 measured reflections
1674 independent reflections
1444 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.078
S = 1.04
1674 reflections
149 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Absolute structure: Flack (1983), 924 Friedel pairs
Absolute structure parameter: 0.039 (18)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.89 (6)	1.81 (5)	2.583 (6)	144 (5)

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL/PC* (Sheldrick, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: NK2224).

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supporting information

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4-Bromo-2-[(phenylimino)methyl]phenol

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S1. Experimental

S1.1. Synthesis and crystallization

4.0204g (20.0 mmol) 5-bromo-salicylaldehyde was dissolved in 30 mL of absolute ethanol. To it 1.822 mL (20.0 mmol) of aniline was added dropwise with a constant stirring. The reaction mixture was heated under refluxing for 3h. After cooling slowly, the light orange powder was separated out. The separated compound, (I), was filtered, washed thoroughly with absolute ethanol and dried in a vacuum desiccator with P_2O_5. Yield 91%. 0.2761g of (I) (1.0mmol) dissolved in 15 mL of absolute ethanol was heated under refluxing until thoroughly dissolved and 0.163 g (1.0 mmol) of $VOSO_4$ in 5 mL of water was added dropwise with a constant stirring. The reaction mixture was adjusted to pH = 7 with NaOH solution, and then it was heated under refluxing for 3h. After cooling slowly, the yellow-green precipitates were separated out. Orange-red crystal (I) was obtained from the filtrate after two weeks. Selected IR(KBr, cm^{-1}): 1614s.

S1.2. Refinement

H atoms attached to C of (I) were placed in geometrically idealized positions with Csp^2—H = 0.93Å. H atom attached to O of (I) was refined freely with the distance of O—H = 0.89 (6) Å.

S2. Results and discussion

We report here the synthesis and characterization a potentially bidentate Schiff base derivative, (I), and prepared from the condensation reaction of an equimolar proportion of 5-bromo-salicylaldehyde and aniline in absolute ethanol. A Schiff base is condensed by primary amines and carbonyl compounds, containing strong electronegative with atoms O and N, so it is easily coordinated with metal ions to form stable complexes (Rehman *et al.*, 2008). It is reported that metal complexes of schiff base derivatives have a variety of important biological activity,such as anti-bacterial, anti-cancer, anti-tumor, hypoglycemic and so on.(Vanco *et al.*, 2008; Ritter *et al.*, 2009).Our reports indicated that copper and vanadium complexes of Schiff bases are potential inhitors over protein tyrosine phosphatases. As part of the ongoing study of vanadium complexes inhibiting protein tyrosine phosphatases (Han *et al.*, 2012), the aim of us is to synthesize new vanadium complex. Unfortunately, only the crystal structure of the title compound (I) was obtained.

The molecular structure and the crystal packing are depicted in Figure 1. X-ray structural analysis confirmed the title compound,(I), the dihedral angle between the two benzene rings is nearly 180° and and all non-H atoms are roughly coplanar with an r.m.s. deviation of 0.0255 Å for a mean plane fitted atomsin the model. There is a strong intramolecular O—H···N hydrogen bonds with a distance of 2.583 (6) Å between donor and acceptor, which generate S(6) ring.

The strong band in IR at 1614 cm^{-1} corresponds to the C7===N1, with a bond length of 1.283 (7) Å, stretching frequency of the imine group of Schiff base.

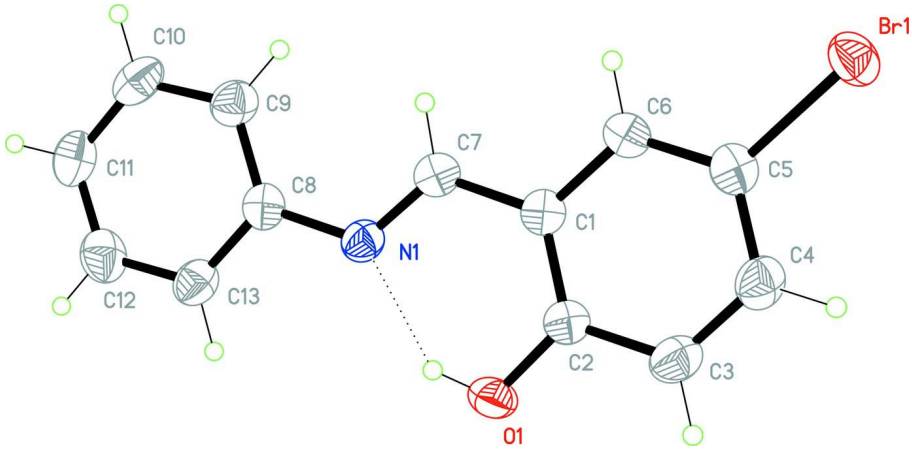

Figure 1

A view of the structure of (I) with displacement ellipsoids drawn at the 50% probability level. Dot line indicates hydrogen bonding interaction.

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Crystal data

$C_{13}H_{10}BrNO$
$M_r = 276.13$
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
$a = 12.353$ (3) Å
$b = 4.5092$ (9) Å
$c = 19.778$ (4) Å
$V = 1101.7$ (4) Å^3
$Z = 4$
$F(000) = 552$
$D_x = 1.665$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2025 reflections
$\theta = 2.1$–$26.0°$
$\mu = 3.71$ mm^{-1}
$T = 298$ K
Block, orange-red
0.20 × 0.15 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
$T_{min} = 0.524$, $T_{max} = 0.836$
4926 measured reflections
1674 independent reflections
1444 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 25.1°$, $\theta_{min} = 3.3°$
$h = -13 \rightarrow 14$
$k = -5 \rightarrow 5$
$l = -23 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.078$
$S = 1.04$
1674 reflections
149 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0329P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}
Absolute structure: Flack (1983), 924 Friedel pairs
Absolute structure parameter: 0.039 (18)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.57515 (4)	1.03271 (10)	0.49809 (4)	0.04069 (18)
C1	0.6938 (4)	0.5393 (12)	0.3358 (3)	0.0283 (13)
C2	0.8024 (4)	0.6143 (12)	0.3347 (3)	0.0310 (14)
C3	0.8434 (5)	0.8206 (15)	0.3824 (3)	0.0401 (16)
H3	0.9163	0.8720	0.3817	0.048*
C4	0.7755 (5)	0.9449 (13)	0.4299 (3)	0.0394 (14)
H4	0.8022	1.0819	0.4608	0.047*
C5	0.6671 (5)	0.8650 (13)	0.4313 (3)	0.0340 (14)
C6	0.6257 (5)	0.6708 (12)	0.3845 (3)	0.0315 (13)
H6	0.5523	0.6255	0.3850	0.038*
C7	0.6489 (4)	0.3280 (12)	0.2878 (3)	0.0305 (13)
H7	0.5750	0.2881	0.2882	0.037*
C8	0.6684 (4)	−0.0149 (12)	0.1984 (3)	0.0314 (13)
C9	0.5609 (5)	−0.1095 (13)	0.1966 (3)	0.0379 (15)
H9	0.5108	−0.0327	0.2271	0.045*
C10	0.5286 (5)	−0.3203 (13)	0.1487 (3)	0.0428 (17)
H10	0.4573	−0.3866	0.1481	0.051*
C11	0.6026 (5)	−0.4316 (12)	0.1020 (3)	0.0401 (15)
H11	0.5810	−0.5689	0.0695	0.048*
C12	0.7067 (6)	−0.3361 (15)	0.1046 (4)	0.0445 (16)
H12	0.7565	−0.4105	0.0736	0.053*
C13	0.7409 (5)	−0.1310 (12)	0.1521 (3)	0.0373 (14)
H13	0.8130	−0.0710	0.1528	0.045*
N1	0.7101 (4)	0.1963 (10)	0.2449 (2)	0.0304 (12)
O1	0.8726 (3)	0.4998 (10)	0.2894 (2)	0.0398 (10)
H1	0.840 (4)	0.348 (13)	0.269 (3)	0.029 (17)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0408 (3)	0.0520 (3)	0.0293 (3)	0.0062 (3)	0.0020 (4)	−0.0049 (6)
C1	0.033 (3)	0.028 (3)	0.023 (3)	0.000 (3)	−0.002 (2)	0.006 (3)
C2	0.032 (3)	0.033 (3)	0.029 (4)	−0.001 (3)	0.000 (3)	0.003 (3)
C3	0.034 (4)	0.047 (4)	0.039 (4)	−0.006 (3)	−0.005 (3)	0.000 (3)
C4	0.042 (3)	0.046 (3)	0.030 (4)	−0.002 (3)	−0.007 (3)	−0.002 (3)
C5	0.040 (3)	0.034 (3)	0.028 (4)	0.002 (3)	0.000 (3)	0.004 (3)

C6	0.031 (3)	0.033 (3)	0.031 (4)	0.003 (3)	0.001 (3)	0.003 (3)
C7	0.031 (3)	0.032 (3)	0.029 (4)	−0.001 (3)	−0.002 (3)	0.005 (3)
C8	0.039 (3)	0.029 (3)	0.026 (3)	0.000 (3)	0.000 (2)	0.003 (3)
C9	0.035 (3)	0.044 (3)	0.034 (4)	−0.003 (3)	−0.005 (3)	−0.002 (3)
C10	0.039 (4)	0.038 (4)	0.051 (5)	−0.010 (3)	−0.014 (3)	0.008 (3)
C11	0.056 (4)	0.034 (3)	0.029 (4)	−0.004 (3)	−0.010 (3)	0.001 (3)
C12	0.057 (4)	0.044 (4)	0.032 (4)	0.001 (4)	0.007 (3)	−0.004 (3)
C13	0.039 (4)	0.036 (3)	0.037 (4)	−0.007 (3)	0.001 (3)	0.001 (3)
N1	0.030 (3)	0.032 (2)	0.029 (3)	−0.002 (2)	−0.002 (3)	0.000 (2)
O1	0.027 (2)	0.054 (3)	0.038 (3)	0.000 (2)	0.0047 (19)	−0.012 (2)

Geometric parameters (Å, º)

Br1—C5	1.899 (6)	C8—C13	1.385 (8)
C1—C2	1.383 (7)	C8—C9	1.396 (7)
C1—C6	1.411 (8)	C8—N1	1.421 (7)
C1—C7	1.454 (8)	C9—C10	1.400 (8)
C2—O1	1.350 (7)	C9—H9	0.9300
C2—C3	1.419 (9)	C10—C11	1.394 (9)
C3—C4	1.378 (9)	C10—H10	0.9300
C3—H3	0.9300	C11—C12	1.358 (9)
C4—C5	1.387 (8)	C11—H11	0.9300
C4—H4	0.9300	C12—C13	1.384 (8)
C5—C6	1.372 (8)	C12—H12	0.9300
C6—H6	0.9300	C13—H13	0.9300
C7—N1	1.283 (7)	O1—H1	0.89 (6)
C7—H7	0.9300		
C2—C1—C6	119.1 (5)	C13—C8—C9	118.9 (5)
C2—C1—C7	121.4 (5)	C13—C8—N1	116.6 (5)
C6—C1—C7	119.6 (5)	C9—C8—N1	124.5 (5)
O1—C2—C1	122.6 (5)	C8—C9—C10	119.7 (6)
O1—C2—C3	117.6 (5)	C8—C9—H9	120.1
C1—C2—C3	119.8 (6)	C10—C9—H9	120.1
C4—C3—C2	120.1 (6)	C11—C10—C9	120.4 (6)
C4—C3—H3	119.9	C11—C10—H10	119.8
C2—C3—H3	119.9	C9—C10—H10	119.8
C3—C4—C5	119.8 (6)	C12—C11—C10	118.8 (6)
C3—C4—H4	120.1	C12—C11—H11	120.6
C5—C4—H4	120.1	C10—C11—H11	120.6
C6—C5—C4	120.7 (6)	C11—C12—C13	121.8 (7)
C6—C5—Br1	120.0 (4)	C11—C12—H12	119.1
C4—C5—Br1	119.2 (5)	C13—C12—H12	119.1
C5—C6—C1	120.5 (5)	C12—C13—C8	120.3 (6)
C5—C6—H6	119.8	C12—C13—H13	119.8
C1—C6—H6	119.8	C8—C13—H13	119.8
N1—C7—C1	120.6 (5)	C7—N1—C8	121.6 (5)
N1—C7—H7	119.7	C2—O1—H1	108 (4)

C1—C7—H7	119.7		
C6—C1—C2—O1	−179.6 (5)	C2—C1—C7—N1	2.9 (8)
C7—C1—C2—O1	0.5 (9)	C6—C1—C7—N1	−177.0 (5)
C6—C1—C2—C3	0.1 (8)	C13—C8—C9—C10	0.6 (9)
C7—C1—C2—C3	−179.8 (5)	N1—C8—C9—C10	−179.7 (5)
O1—C2—C3—C4	180.0 (5)	C8—C9—C10—C11	−1.4 (9)
C1—C2—C3—C4	0.3 (9)	C9—C10—C11—C12	1.2 (9)
C2—C3—C4—C5	0.8 (9)	C10—C11—C12—C13	−0.3 (10)
C3—C4—C5—C6	−2.2 (9)	C11—C12—C13—C8	−0.5 (10)
C3—C4—C5—Br1	178.8 (5)	C9—C8—C13—C12	0.4 (9)
C4—C5—C6—C1	2.6 (9)	N1—C8—C13—C12	−179.4 (5)
Br1—C5—C6—C1	−178.4 (4)	C1—C7—N1—C8	178.8 (5)
C2—C1—C6—C5	−1.6 (8)	C13—C8—N1—C7	176.6 (5)
C7—C1—C6—C5	178.4 (5)	C9—C8—N1—C7	−3.1 (9)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.89 (6)	1.81 (5)	2.583 (6)	144 (5)