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2′-(5-Bromo-1*H*-indol-3-ylmethylene)-benzenesulfonohydrazine

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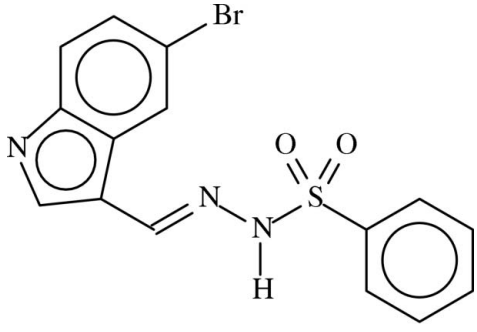
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.027; wR factor = 0.082; data-to-parameter ratio = 16.3.

The molecules of the title compound, $C_{15}H_{12}BrN_3O_2S$, are linked by N_{amino}–H···$O_{sulfonyl}$ and $N_{indolyl}$–H···N_{imino} hydrogen bonds into a layer motif. The dihedral angle between the aromatic ring mean planes in the molecule is 76.44 (8)°.

Related literature

For related structures, see: Ali, Yusnita *et al.* (2007); Ali, Yusnita & Ng (2007).



Experimental

Crystal data

$C_{15}H_{12}BrN_3O_2S$
M_r = 378.25
Orthorhombic, *Pbcn*
a = 33.1196 (4) Å
b = 8.9260 (1) Å
c = 9.9158 (1) Å
V = 2931.36 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 2.96 mm^{-1}
T = 173 (2) K
0.50 × 0.40 × 0.40 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.330, T_{max} = 0.384
(expected range = 0.263–0.306)
59138 measured reflections
3375 independent reflections
3270 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.082
S = 1.07
3375 reflections
207 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1n···O1^{i}	0.87 (1)	2.17 (2)	2.986 (2)	155 (3)
N3—H3n···N2^{ii}	0.88 (1)	2.24 (2)	3.028 (2)	150 (3)

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

Data collection: *APEXII* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2389).

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supplementary materials

Acta Cryst. (2007). E**63**, o2733 [**doi:10.1107/S1600536807019927**]

2'-(5-Bromo-1*H*-indol-3-ylmethylene)benzenesulfonohydrazine

H. M. Ali, J. Yusnita and S. W. Ng

Comment

The crystal structure of 2'-(1*H*-indol-3-ylmethylene)benzenesulfonohydrazine consists of molecules that are linked by N_{amino}–*H*···$O_{sulfonoy}$ and $N_{indolyl}$–*H*···N_{imino} hydrogen bonds into layers (Ali, Yusnita, Wan Jefrey & Ng, 2007; Ali, Yusnita & Ng, 2007. The presence of the bromine substituent in the 5-position of the indolyl portion leads to a similar layer structure for the title compound (I).

Experimental

Benzenesulfohydrazine (0.3 g, 2 mmol) and 5-bromoindole-3-carbaldehyde (0.3 g, 2 mmol) were dissolved in ethanol (50 ml). The reactants were heated under reflux for 1 h. The solvent was removed to give the Schiff base, which was purified by recrystallzation from ethanol to yield faint yellow blocks of (I).

Refinement

The carbon-bound H atoms were placed at calculated positions (C–H = 0.95-0.98 Å), and they were included in the refinement in the riding model approximation with U(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The N-bound H atoms were located in a difference Fourier map, and were refined with a distance restraint [N–H = 0.88 (1) Å]; their U_{iso} values were freely refined.

Figures

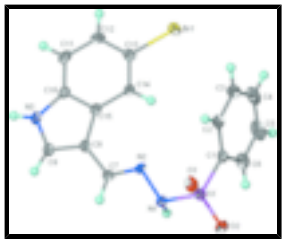


Fig. 1. View of the molecular structure of (I). Displacement ellipsoids are drawn at the 70% probability level and H atoms are shown as spheres of arbitrary radius.

2'-(5-Bromo-1*H*-indol-3-ylmethylene)benzenesulfonohydrazine

Crystal data

$C_{15}H_{12}BrN_3O_2S$	F_{000} = 1520
M_r = 378.25	D_x = 1.714 Mg m^{-3}
Orthorhombic, *Pbcn*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2n 2ab	Cell parameters from 9228 reflections
a = 33.1196 (4) Å	θ = 2.4–33.5º

b = 8.9260 (1) Å	μ = 2.96 mm^{-1}
c = 9.9158 (1) Å	T = 173 (2) K
V = 2931.36 (6) Å^3	Block, faint yellow
Z = 8	0.50 × 0.40 × 0.40 mm

Data collection

Bruker APEXII CCD diffractometer	3375 independent reflections
Radiation source: medium-focus sealed tube	3270 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.030
T = 173(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 1.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −43→43
T_{min} = 0.330, T_{max} = 0.384	k = −11→11
59138 measured reflections	l = −12→12

Refinement

Refinement on F^2	Hydrogen site location: difmap and geom
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.027	$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 4.5513P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.082	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.07	$\Delta\rho_{max}$ = 0.41 e Å^{-3}
3375 reflections	$\Delta\rho_{min}$ = −0.39 e Å^{-3}
207 parameters	Extinction correction: none
2 restraints	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.517836 (6)	0.70758 (2)	0.45443 (2)	0.02107 (9)
S1	0.702481 (14)	0.49278 (5)	0.24367 (5)	0.01426 (11)
O1	0.70538 (5)	0.58662 (18)	0.12640 (15)	0.0204 (3)
O2	0.72824 (5)	0.36427 (18)	0.25464 (16)	0.0226 (3)
N1	0.71431 (5)	0.59551 (19)	0.37560 (17)	0.0140 (3)
H1N	0.7194 (9)	0.536 (3)	0.443 (2)	0.031 (8)*
N2	0.68493 (5)	0.70889 (18)	0.40448 (18)	0.0132 (3)
N3	0.64834 (5)	1.0919 (2)	0.68965 (18)	0.0180 (4)
H3N	0.6499 (9)	1.164 (2)	0.749 (2)	0.026 (7)*
C1	0.65175 (6)	0.4387 (2)	0.2608 (2)	0.0151 (4)
C2	0.62208 (7)	0.5210 (2)	0.1940 (2)	0.0189 (4)

H2	0.6293	0.6011	0.1357	0.023*
C3	0.58184 (7)	0.4838 (3)	0.2140 (2)	0.0252 (5)
H3	0.5612	0.5398	0.1705	0.030*
C4	0.57173 (7)	0.3650 (3)	0.2974 (2)	0.0283 (5)
H4	0.5441	0.3404	0.3113	0.034*
C5	0.60142 (8)	0.2821 (3)	0.3604 (2)	0.0276 (5)
H5	0.5941	0.1996	0.4157	0.033*
C6	0.64192 (7)	0.3184 (3)	0.3435 (2)	0.0217 (4)
H6	0.6624	0.2622	0.3875	0.026*
C7	0.69757 (6)	0.8008 (2)	0.4945 (2)	0.0136 (4)
H7	0.7251	0.7987	0.5215	0.016*
C8	0.67028 (6)	0.9073 (2)	0.55495 (19)	0.0144 (4)
C9	0.68164 (6)	1.0224 (2)	0.6400 (2)	0.0168 (4)
H9A	0.7087	1.0489	0.6606	0.020*
C10	0.61404 (6)	1.0226 (2)	0.6410 (2)	0.0149 (4)
C11	0.57348 (6)	1.0512 (2)	0.6664 (2)	0.0179 (4)
H11	0.5655	1.1323	0.7223	0.021*
C12	0.54506 (6)	0.9581 (2)	0.6078 (2)	0.0173 (4)
H12	0.5171	0.9745	0.6233	0.021*
C13	0.55780 (6)	0.8393 (2)	0.5253 (2)	0.0152 (4)
C14	0.59775 (6)	0.8105 (2)	0.4965 (2)	0.0135 (4)
H14	0.6054	0.7299	0.4394	0.016*
C15	0.62673 (6)	0.9053 (2)	0.55482 (19)	0.0132 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.01373 (12)	0.02226 (13)	0.02720 (14)	−0.00269 (7)	−0.00048 (8)	−0.00370 (8)
S1	0.0148 (2)	0.0144 (2)	0.0136 (2)	−0.00029 (17)	0.00231 (17)	−0.00240 (17)
O1	0.0248 (8)	0.0238 (8)	0.0126 (7)	−0.0057 (6)	0.0030 (6)	−0.0004 (6)
O2	0.0194 (8)	0.0200 (8)	0.0284 (8)	0.0047 (6)	0.0012 (6)	−0.0081 (6)
N1	0.0145 (8)	0.0136 (8)	0.0137 (8)	0.0023 (6)	−0.0012 (6)	−0.0020 (6)
N2	0.0128 (8)	0.0124 (8)	0.0143 (8)	0.0025 (6)	0.0011 (6)	0.0003 (6)
N3	0.0209 (9)	0.0140 (8)	0.0191 (8)	0.0015 (7)	−0.0034 (7)	−0.0061 (7)
C1	0.0155 (9)	0.0150 (9)	0.0147 (9)	−0.0024 (7)	0.0023 (7)	−0.0037 (7)
C2	0.0200 (10)	0.0159 (10)	0.0207 (10)	−0.0011 (8)	−0.0017 (8)	−0.0029 (8)
C3	0.0174 (10)	0.0268 (12)	0.0315 (12)	0.0020 (9)	−0.0022 (9)	−0.0099 (9)
C4	0.0224 (11)	0.0362 (13)	0.0264 (11)	−0.0104 (10)	0.0065 (9)	−0.0130 (10)
C5	0.0340 (13)	0.0326 (13)	0.0163 (10)	−0.0158 (10)	0.0048 (9)	0.0002 (9)
C6	0.0264 (11)	0.0214 (10)	0.0171 (10)	−0.0039 (9)	−0.0013 (8)	0.0009 (8)
C7	0.0129 (9)	0.0135 (9)	0.0145 (9)	−0.0003 (7)	−0.0004 (7)	0.0016 (7)
C8	0.0166 (9)	0.0125 (9)	0.0141 (9)	−0.0005 (7)	−0.0023 (7)	0.0000 (7)
C9	0.0186 (9)	0.0143 (9)	0.0175 (9)	0.0001 (7)	−0.0026 (8)	−0.0018 (7)
C10	0.0184 (9)	0.0126 (9)	0.0137 (9)	0.0029 (7)	−0.0028 (7)	−0.0006 (7)
C11	0.0226 (10)	0.0172 (9)	0.0138 (9)	0.0067 (8)	−0.0003 (8)	−0.0024 (8)
C12	0.0144 (9)	0.0207 (10)	0.0169 (9)	0.0043 (8)	0.0005 (8)	0.0007 (8)
C13	0.0156 (9)	0.0169 (9)	0.0132 (9)	−0.0008 (8)	−0.0020 (7)	0.0010 (7)
C14	0.0148 (9)	0.0124 (8)	0.0132 (9)	0.0009 (7)	0.0005 (7)	−0.0006 (7)

C15	0.0161 (9)	0.0118 (8)	0.0118 (8)	0.0015 (7)	0.0000 (7)	0.0016 (7)

Geometric parameters (Å, °)

Br1—C13	1.905 (2)	C4—H4	0.9500
S1—O2	1.4337 (16)	C5—C6	1.390 (3)
S1—O1	1.4363 (15)	C5—H5	0.9500
S1—N1	1.6448 (17)	C6—H6	0.9500
S1—C1	1.756 (2)	C7—C8	1.442 (3)
N1—N2	1.433 (2)	C7—H7	0.9500
N1—H1N	0.872 (10)	C8—C9	1.381 (3)
N2—C7	1.282 (3)	C8—C15	1.442 (3)
N3—C9	1.358 (3)	C9—H9A	0.9500
N3—C10	1.381 (3)	C10—C11	1.390 (3)
N3—H3N	0.876 (10)	C10—C15	1.416 (3)
C1—C6	1.390 (3)	C11—C12	1.383 (3)
C1—C2	1.394 (3)	C11—H11	0.9500
C2—C3	1.388 (3)	C12—C13	1.405 (3)
C2—H2	0.9500	C12—H12	0.9500
C3—C4	1.386 (4)	C13—C14	1.378 (3)
C3—H3	0.9500	C14—C15	1.404 (3)
C4—C5	1.380 (4)	C14—H14	0.9500
O2—S1—O1	119.23 (10)	C5—C6—H6	120.8
O2—S1—N1	104.12 (9)	C1—C6—H6	120.8
O1—S1—N1	107.63 (9)	N2—C7—C8	120.41 (19)
O2—S1—C1	110.00 (10)	N2—C7—H7	119.8
O1—S1—C1	107.60 (10)	C8—C7—H7	119.8
N1—S1—C1	107.71 (9)	C9—C8—C15	106.38 (18)
N2—N1—S1	113.01 (13)	C9—C8—C7	124.99 (19)
N2—N1—H1N	114 (2)	C15—C8—C7	128.18 (18)
S1—N1—H1N	108 (2)	N3—C9—C8	109.91 (18)
C7—N2—N1	111.66 (16)	N3—C9—H9A	125.0
C9—N3—C10	109.67 (17)	C8—C9—H9A	125.0
C9—N3—H3N	122.2 (19)	N3—C10—C11	130.53 (19)
C10—N3—H3N	127.9 (19)	N3—C10—C15	107.34 (17)
C6—C1—C2	121.5 (2)	C11—C10—C15	122.12 (19)
C6—C1—S1	119.57 (17)	C12—C11—C10	118.13 (19)
C2—C1—S1	118.92 (16)	C12—C11—H11	120.9
C3—C2—C1	118.9 (2)	C10—C11—H11	120.9
C3—C2—H2	120.6	C11—C12—C13	119.59 (18)
C1—C2—H2	120.6	C11—C12—H12	120.2
C2—C3—C4	120.0 (2)	C13—C12—H12	120.2
C2—C3—H3	120.0	C14—C13—C12	123.36 (19)
C4—C3—H3	120.0	C14—C13—Br1	118.43 (15)
C5—C4—C3	120.5 (2)	C12—C13—Br1	118.18 (15)
C5—C4—H4	119.7	C13—C14—C15	117.34 (18)
C3—C4—H4	119.7	C13—C14—H14	121.3
C4—C5—C6	120.5 (2)	C15—C14—H14	121.3
C4—C5—H5	119.7	C14—C15—C10	119.42 (18)

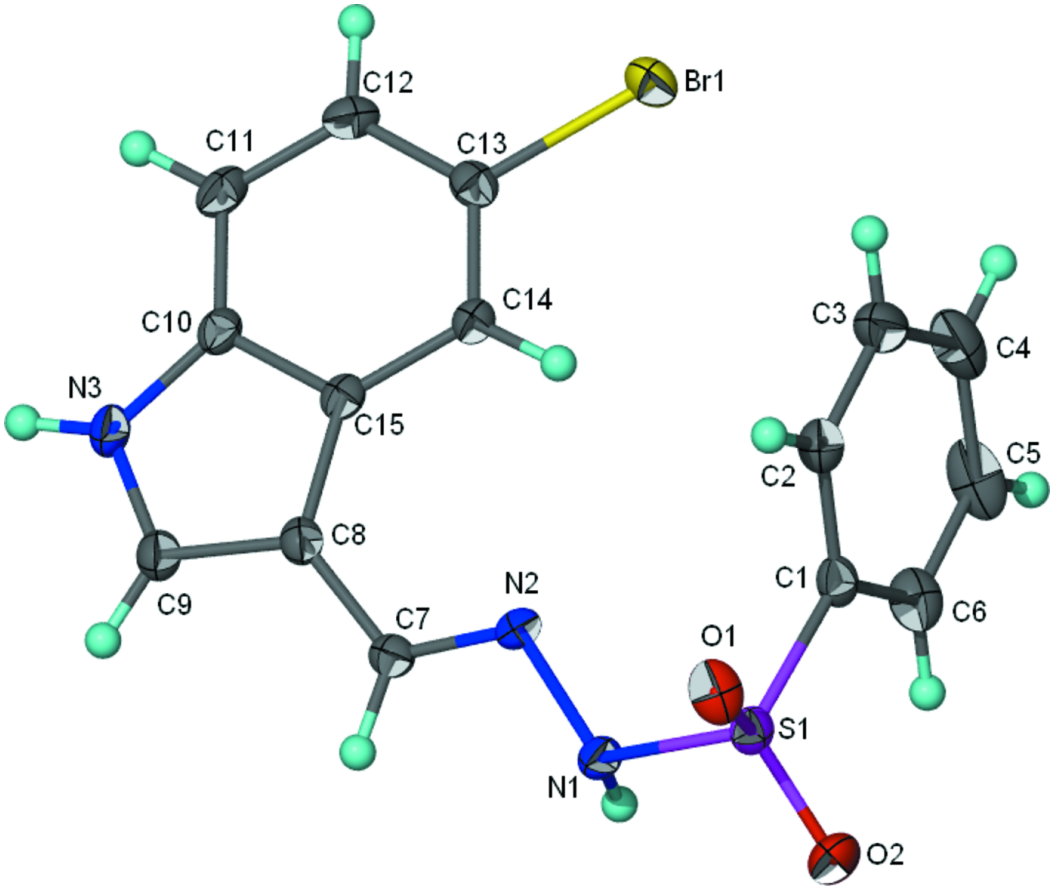
C6—C5—H5	119.7	C14—C15—C8	133.76 (18)
C5—C6—C1	118.5 (2)	C10—C15—C8	106.69 (17)
O2—S1—N1—N2	−164.15 (13)	C15—C8—C9—N3	−1.0 (2)
O1—S1—N1—N2	68.38 (15)	C7—C8—C9—N3	−173.81 (19)
C1—S1—N1—N2	−47.37 (16)	C9—N3—C10—C11	178.1 (2)
S1—N1—N2—C7	−170.42 (14)	C9—N3—C10—C15	−1.0 (2)
O2—S1—C1—C6	32.1 (2)	N3—C10—C11—C12	−177.0 (2)
O1—S1—C1—C6	163.45 (17)	C15—C10—C11—C12	1.9 (3)
N1—S1—C1—C6	−80.77 (18)	C10—C11—C12—C13	−0.1 (3)
O2—S1—C1—C2	−149.89 (16)	C11—C12—C13—C14	−1.2 (3)
O1—S1—C1—C2	−18.54 (19)	C11—C12—C13—Br1	176.74 (16)
N1—S1—C1—C2	97.24 (17)	C12—C13—C14—C15	0.7 (3)
C6—C1—C2—C3	1.8 (3)	Br1—C13—C14—C15	−177.22 (14)
S1—C1—C2—C3	−176.16 (16)	C13—C14—C15—C10	1.1 (3)
C1—C2—C3—C4	−1.1 (3)	C13—C14—C15—C8	176.2 (2)
C2—C3—C4—C5	−0.5 (3)	N3—C10—C15—C14	176.72 (17)
C3—C4—C5—C6	1.4 (4)	C11—C10—C15—C14	−2.4 (3)
C4—C5—C6—C1	−0.7 (3)	N3—C10—C15—C8	0.3 (2)
C2—C1—C6—C5	−0.9 (3)	C11—C10—C15—C8	−178.80 (19)
S1—C1—C6—C5	177.05 (17)	C9—C8—C15—C14	−175.2 (2)
N1—N2—C7—C8	−169.22 (17)	C7—C8—C15—C14	−2.7 (4)
N2—C7—C8—C9	−171.7 (2)	C9—C8—C15—C10	0.4 (2)
N2—C7—C8—C15	17.1 (3)	C7—C8—C15—C10	172.88 (19)
C10—N3—C9—C8	1.3 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1n···O1^{i}	0.87 (1)	2.17 (2)	2.986 (2)	155 (3)
N3—H3n···N2^{ii}	0.88 (1)	2.24 (2)	3.028 (2)	150 (3)

Symmetry codes: (i) *x*, −*y*+1, *z*+1/2; (ii) *x*, −*y*+2, *z*+1/2.

Fig. 1


C12
Br1
C11
C13
C14
C10
N3
C15
C8
C9
C7
N2
N1
O1
S1
O2
C1
C2
C3
C4
C5
C6