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

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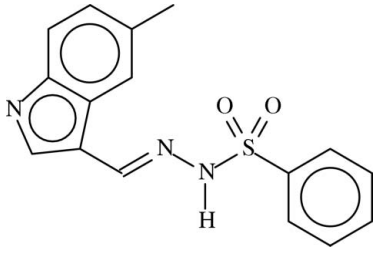
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.099; data-to-parameter ratio = 17.5.

The molecules of the title compound, $C_{16}H_{15}N_3O_2S$, 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 78.85 (5)°.

Related literature

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



Experimental

Crystal data

$C_{16}H_{15}N_3O_2S$
M_r = 313.37
Orthorhombic, *Pbca*
a = 13.3080 (2) Å
b = 10.0043 (1) Å
c = 23.7968 (3) Å
V = 3168.24 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 173 (2) K
0.54 × 0.53 × 0.50 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: none
41089 measured reflections
3637 independent reflections
3334 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.099
S = 1.01
3637 reflections
208 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 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)	2.05 (1)	2.924 (1)	173 (2)
N3—H3n···N1^{ii}	0.87 (1)	2.16 (1)	3.032 (1)	177 (2)

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

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: HB2390).

References

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  doi:10.1107/S1600536807020065

supplementary materials

Acta Cryst. (2007). E**63**, o2734 [**doi:10.1107/S1600536807020065**]

2'-(5-Methyl-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 methyl 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-methylindole-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

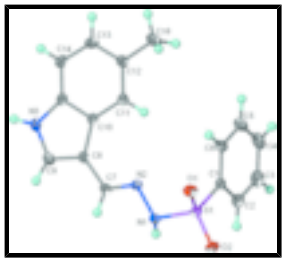

Fig. 1. View of the molecular structure of (I). Displacement ellipsoids are drawn at the 70% probability level and H atoms are shown as spheres of arbitrary radius.

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

Crystal data

$C_{16}H_{15}N_3O_2S$	F_{000} = 1312
M_r = 313.37	D_x = 1.314 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 9953 reflections

a = 13.3080 (2) Å	θ = 2.3–33.9º
b = 10.0043 (1) Å	μ = 0.21 mm^{-1}
c = 23.7968 (3) Å	T = 173 (2) K
V = 3168.24 (7) Å^3	Irregular block, faint yellow
Z = 8	0.54 × 0.53 × 0.50 mm

Data collection

Bruker APEXII CCD diffractometer	3334 reflections with $I > 2\sigma(I)$
Radiation source: medium-focus sealed tube	R_{int} = 0.025
Monochromator: graphite	θ_{max} = 27.5º
T = 173(2) K	θ_{min} = 1.7º
φ and ω scans	h = −17→17
Absorption correction: none	k = −12→12
41089 measured reflections	l = −30→30
3637 independent reflections	

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
$R[F^2 > 2\sigma(F^2)]$ = 0.031	$w = 1/[\sigma^2(F_o^2) + (0.0598P)^2 + 1.5134P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.099	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.01	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
3637 reflections	$\Delta\rho_{min}$ = −0.39 e Å^{-3}
208 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}
S1	0.23027 (2)	0.11903 (3)	0.421914 (12)	0.01747 (10)
O1	0.21446 (7)	0.25306 (8)	0.40210 (4)	0.0244 (2)
O2	0.14557 (6)	0.04044 (9)	0.43913 (4)	0.0248 (2)
N1	0.27929 (7)	0.03416 (10)	0.36867 (4)	0.0181 (2)
H1N	0.2819 (13)	−0.0515 (10)	0.3758 (7)	0.035 (5)*
N2	0.37106 (7)	0.09299 (10)	0.35018 (4)	0.0191 (2)
N3	0.64312 (8)	0.08303 (12)	0.23137 (4)	0.0247 (2)
H3N	0.6840 (12)	0.0675 (19)	0.2036 (6)	0.046 (5)*
C1	0.32075 (9)	0.12280 (11)	0.47563 (5)	0.0190 (2)
C2	0.32850 (10)	0.01370 (13)	0.51174 (5)	0.0265 (3)
H2	0.2858	−0.0617	0.5074	0.032*

C3	0.40021 (12)	0.01753 (15)	0.55427 (6)	0.0342 (3)
H3	0.4063	−0.0554	0.5796	0.041*
C4	0.46263 (12)	0.12737 (15)	0.55972 (6)	0.0346 (3)
H4	0.5111	0.1294	0.5890	0.041*
C5	0.45525 (10)	0.23442 (14)	0.52298 (6)	0.0299 (3)
H5	0.4990	0.3088	0.5269	0.036*
C6	0.38383 (9)	0.23295 (12)	0.48040 (5)	0.0230 (2)
H6	0.3782	0.3059	0.4550	0.028*
C7	0.41556 (9)	0.02655 (12)	0.31144 (5)	0.0211 (2)
H7	0.3866	−0.0540	0.2979	0.025*
C8	0.50919 (9)	0.07311 (12)	0.28826 (5)	0.0207 (2)
C9	0.55733 (10)	0.01550 (13)	0.24299 (5)	0.0251 (3)
H9	0.5338	−0.0604	0.2229	0.030*
C10	0.57019 (9)	0.18475 (11)	0.30554 (5)	0.0190 (2)
C11	0.56175 (9)	0.28304 (12)	0.34724 (5)	0.0210 (2)
H11	0.5058	0.2831	0.3720	0.025*
C12	0.63569 (10)	0.38036 (13)	0.35217 (5)	0.0251 (3)
C13	0.72032 (10)	0.37621 (13)	0.31648 (6)	0.0277 (3)
H13	0.7718	0.4411	0.3212	0.033*
C14	0.73080 (9)	0.28053 (13)	0.27478 (6)	0.0258 (3)
H14	0.7880	0.2789	0.2509	0.031*
C15	0.65415 (9)	0.18704 (12)	0.26929 (5)	0.0211 (2)
C16	0.62308 (12)	0.49234 (16)	0.39414 (7)	0.0379 (3)
H16A	0.5575	0.5349	0.3886	0.057*
H16B	0.6764	0.5586	0.3887	0.057*
H16C	0.6271	0.4562	0.4323	0.057*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01618 (16)	0.01306 (16)	0.02316 (17)	0.00111 (9)	−0.00103 (10)	0.00258 (10)
O1	0.0252 (4)	0.0141 (4)	0.0339 (5)	0.0034 (3)	−0.0041 (4)	0.0049 (3)
O2	0.0187 (4)	0.0206 (4)	0.0350 (5)	−0.0012 (3)	0.0028 (3)	0.0033 (4)
N1	0.0188 (5)	0.0137 (4)	0.0218 (5)	−0.0015 (3)	−0.0007 (4)	0.0017 (4)
N2	0.0189 (5)	0.0178 (5)	0.0207 (5)	−0.0026 (4)	−0.0012 (4)	0.0031 (4)
N3	0.0255 (5)	0.0275 (5)	0.0213 (5)	0.0030 (4)	0.0038 (4)	−0.0028 (4)
C1	0.0199 (5)	0.0182 (5)	0.0189 (5)	0.0033 (4)	0.0002 (4)	−0.0006 (4)
C2	0.0303 (7)	0.0240 (6)	0.0253 (6)	0.0022 (5)	0.0004 (5)	0.0045 (5)
C3	0.0405 (8)	0.0373 (8)	0.0247 (6)	0.0098 (6)	−0.0038 (6)	0.0072 (5)
C4	0.0333 (7)	0.0449 (9)	0.0254 (6)	0.0097 (6)	−0.0091 (6)	−0.0064 (6)
C5	0.0256 (6)	0.0302 (7)	0.0340 (7)	0.0026 (5)	−0.0043 (5)	−0.0106 (5)
C6	0.0230 (6)	0.0188 (5)	0.0271 (6)	0.0027 (4)	−0.0007 (5)	−0.0031 (4)
C7	0.0216 (5)	0.0191 (5)	0.0226 (5)	−0.0009 (4)	−0.0035 (4)	−0.0008 (4)
C8	0.0219 (6)	0.0202 (6)	0.0200 (5)	0.0014 (5)	−0.0014 (4)	−0.0010 (4)
C9	0.0262 (6)	0.0241 (6)	0.0251 (6)	0.0012 (5)	−0.0009 (5)	−0.0046 (5)
C10	0.0204 (5)	0.0182 (5)	0.0183 (5)	0.0024 (4)	−0.0007 (4)	0.0030 (4)
C11	0.0229 (6)	0.0215 (6)	0.0187 (5)	0.0021 (5)	−0.0002 (4)	0.0004 (4)
C12	0.0273 (6)	0.0227 (6)	0.0252 (6)	0.0001 (5)	−0.0028 (5)	−0.0021 (5)

C13	0.0263 (6)	0.0245 (6)	0.0325 (7)	−0.0053 (5)	−0.0011 (5)	0.0018 (5)
C14	0.0234 (6)	0.0271 (6)	0.0269 (6)	−0.0002 (5)	0.0035 (5)	0.0041 (5)
C15	0.0230 (6)	0.0210 (6)	0.0193 (5)	0.0038 (5)	0.0005 (4)	0.0024 (4)
C16	0.0369 (8)	0.0349 (8)	0.0418 (8)	−0.0054 (6)	−0.0011 (6)	−0.0160 (6)

Geometric parameters (Å, °)

S1—O2	1.4340 (9)	C6—H6	0.9500
S1—O1	1.4369 (9)	C7—C8	1.4401 (17)
S1—N1	1.6589 (10)	C7—H7	0.9500
S1—C1	1.7566 (12)	C8—C9	1.3794 (17)
N1—N2	1.4253 (13)	C8—C10	1.4406 (17)
N1—H1N	0.874 (9)	C9—H9	0.9500
N2—C7	1.2816 (16)	C10—C11	1.4015 (16)
N3—C9	1.3551 (17)	C10—C15	1.4119 (16)
N3—C15	1.3851 (16)	C11—C12	1.3892 (18)
N3—H3N	0.870 (9)	C11—H11	0.9500
C1—C6	1.3899 (17)	C12—C13	1.4112 (19)
C1—C2	1.3929 (17)	C12—C16	1.5101 (18)
C2—C3	1.3914 (19)	C13—C14	1.3858 (19)
C2—H2	0.9500	C13—H13	0.9500
C3—C4	1.384 (2)	C14—C15	1.3901 (18)
C3—H3	0.9500	C14—H14	0.9500
C4—C5	1.386 (2)	C16—H16A	0.9800
C4—H4	0.9500	C16—H16B	0.9800
C5—C6	1.3894 (18)	C16—H16C	0.9800
C5—H5	0.9500		
O2—S1—O1	119.35 (5)	N2—C7—H7	119.7
O2—S1—N1	104.28 (5)	C8—C7—H7	119.7
O1—S1—N1	106.54 (5)	C9—C8—C7	124.46 (11)
O2—S1—C1	110.04 (6)	C9—C8—C10	106.57 (11)
O1—S1—C1	108.61 (5)	C7—C8—C10	128.97 (11)
N1—S1—C1	107.30 (5)	N3—C9—C8	110.02 (11)
N2—N1—S1	111.18 (7)	N3—C9—H9	125.0
N2—N1—H1N	115.5 (12)	C8—C9—H9	125.0
S1—N1—H1N	111.7 (12)	C11—C10—C15	118.99 (11)
C7—N2—N1	113.81 (10)	C11—C10—C8	134.50 (11)
C9—N3—C15	109.32 (10)	C15—C10—C8	106.50 (10)
C9—N3—H3N	126.3 (13)	C12—C11—C10	119.66 (11)
C15—N3—H3N	124.3 (13)	C12—C11—H11	120.2
C6—C1—C2	121.75 (12)	C10—C11—H11	120.2
C6—C1—S1	119.36 (9)	C11—C12—C13	119.59 (12)
C2—C1—S1	118.87 (10)	C11—C12—C16	119.81 (12)
C3—C2—C1	118.55 (13)	C13—C12—C16	120.56 (12)
C3—C2—H2	120.7	C14—C13—C12	122.11 (12)
C1—C2—H2	120.7	C14—C13—H13	118.9
C4—C3—C2	120.12 (13)	C12—C13—H13	118.9
C4—C3—H3	119.9	C13—C14—C15	117.27 (12)
C2—C3—H3	119.9	C13—C14—H14	121.4

C3—C4—C5	120.80 (13)	C15—C14—H14	121.4
C3—C4—H4	119.6	N3—C15—C14	130.13 (11)
C5—C4—H4	119.6	N3—C15—C10	107.58 (11)
C4—C5—C6	120.02 (13)	C14—C15—C10	122.29 (11)
C4—C5—H5	120.0	C12—C16—H16A	109.5
C6—C5—H5	120.0	C12—C16—H16B	109.5
C5—C6—C1	118.75 (12)	H16A—C16—H16B	109.5
C5—C6—H6	120.6	C12—C16—H16C	109.5
C1—C6—H6	120.6	H16A—C16—H16C	109.5
N2—C7—C8	120.50 (11)	H16B—C16—H16C	109.5
O2—S1—N1—N2	−174.17 (7)	C7—C8—C9—N3	179.62 (11)
O1—S1—N1—N2	58.73 (8)	C10—C8—C9—N3	0.16 (14)
C1—S1—N1—N2	−57.45 (8)	C9—C8—C10—C11	178.40 (13)
S1—N1—N2—C7	175.77 (8)	C7—C8—C10—C11	−1.0 (2)
O2—S1—C1—C6	−151.28 (9)	C9—C8—C10—C15	−0.72 (13)
O1—S1—C1—C6	−18.95 (11)	C7—C8—C10—C15	179.85 (12)
N1—S1—C1—C6	95.85 (10)	C15—C10—C11—C12	−0.04 (17)
O2—S1—C1—C2	29.91 (11)	C8—C10—C11—C12	−179.07 (13)
O1—S1—C1—C2	162.25 (10)	C10—C11—C12—C13	−2.34 (18)
N1—S1—C1—C2	−82.95 (11)	C10—C11—C12—C16	175.45 (12)
C6—C1—C2—C3	1.35 (19)	C11—C12—C13—C14	2.5 (2)
S1—C1—C2—C3	−179.87 (10)	C16—C12—C13—C14	−175.25 (13)
C1—C2—C3—C4	−0.6 (2)	C12—C13—C14—C15	−0.2 (2)
C2—C3—C4—C5	−0.4 (2)	C9—N3—C15—C14	178.29 (13)
C3—C4—C5—C6	0.7 (2)	C9—N3—C15—C10	−0.94 (14)
C4—C5—C6—C1	0.01 (19)	C13—C14—C15—N3	178.60 (13)
C2—C1—C6—C5	−1.04 (18)	C13—C14—C15—C10	−2.28 (18)
S1—C1—C6—C5	−179.81 (9)	C11—C10—C15—N3	−178.27 (10)
N1—N2—C7—C8	179.93 (10)	C8—C10—C15—N3	1.01 (13)
N2—C7—C8—C9	−173.47 (12)	C11—C10—C15—C14	2.43 (17)
N2—C7—C8—C10	5.86 (19)	C8—C10—C15—C14	−178.29 (11)
C15—N3—C9—C8	0.48 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1n···O1^{i}	0.87 (1)	2.054 (9)	2.924 (1)	173.3 (16)
N3—H3n···N1^{ii}	0.87 (1)	2.163 (9)	3.032 (1)	176.7 (18)

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

Fig. 1