Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-(5-Fluoro-2-oxo-2,3-dihydro-1*H*-indol-3-ylidene)benzenesulfonohydrazide

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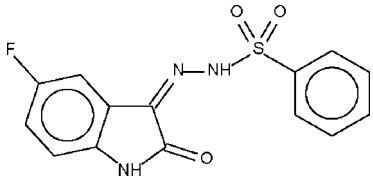
Received 29 January 2008; accepted 20 April 2008

Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.145; data-to-parameter ratio = 15.3.

The molecule of the title compound, $C_{14}H_{10}FN_3O_3S$, consists of an indole unit and a phenylsulfonyl unit that are disposed in an approximately *trans* orientation relative to the N—N single bond. Two molecules are arranged about a center of inversion, forming a hydrazide–carbonyl N—H···O hydrogen-bonded dimer; the dimers are linked by an indole–sulfonyl N—H···O hydrogen bond into a ribbon.

Related literature

For the crystal structures of related 3-indole benzenesulfonylhydrazones, see: Ali *et al.* (2007*a*,*b*,*c*). For the crystal structure of 5-fluoro-1*H*-indole-2,3-dione, see: Naumov *et al.* (2000).



Experimental

Crystal data

$C_{14}H_{10}FN_3O_3S$
M_r = 319.31
Monoclinic, $P2_1/c$
a = 8.2218 (2) Å
b = 16.4933 (3) Å
c = 10.8585 (2) Å
β = 110.249 (1)°
V = 1381.46 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 123 (2) K
0.50 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.816, T_{max} = 0.962
10513 measured reflections
3166 independent reflections
2741 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.145
S = 1.20
3166 reflections
207 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.59 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1n···O3^{i}	0.88 (1)	2.10 (2)	2.896 (2)	151 (2)
N3—H3n···O1^{ii}	0.88 (1)	2.22 (2)	2.986 (2)	145 (2)

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

The authors thank the University of Canterbury, New Zealand, for the diffraction measurements, and the Science Fund (12–02-03–2031) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2224).

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supporting information

Acta Cryst. (2008). E**64**, o921 [doi:10.1107/S1600536808011136]

N′-(5-Fluoro-2-oxo-2,3-dihydro-1*H*-indol-3-ylidene)benzenesulfonohydrazide

Hapipah M. Ali, Musalem Laila, Mohd. Razali Rizal and Seik Weng Ng

S1. Comment

We have reported the crystal structures of 3-indole benzenesulfonohydrazides (Ali *et al.*, 2007*a*, 2007*b*, 2007*c*). The studies continue with the benzenesulfonohydrazide that is obtained by condensing benzenesulfonohydrazine with a substituted 1*H*-indol-2,3-dione, 5-fluroisatin. This compound exists as a hydrogen-bonded dimer (Naumov *et al.*, 2000). The title compound (Scheme I) has the indolyl fused-ring portion and the phenylsulfonyl portion disposed in an approximately *trans*-alignment relative to the N–N single-bond (Fig. 1). Two molecules are arranged about a center-of-inversion to form an N–$H_{hydrazide}\cdots O_{carbonyl}$ hydrogen-bonded dimer; the dimers are linked by another N–$H_{indole}\cdots O_{sulfonyl}$ hydrogen bond into a ribbon structure (Fig. 2).

S2. Experimental

Benzenesulfonyl hydrazide (0. 69 g, 4 mmol) and 5-fluoroisatin (0.66 g, 4 mmol) were heated in ethanol (50 ml) for an hour. The solution when cooled afforded yellow crystals.

S3. Refinement

The carbon-bound H atoms were placed at calculated positions (C–H 0.95 Å), and were included in the refinement in the riding model approximation with U(H) set to 1.2U_{eq}(C). The amino H atoms were located in a difference Fouier map, and were refined with a distance restraint of N–H 0.88±0.01 Å.

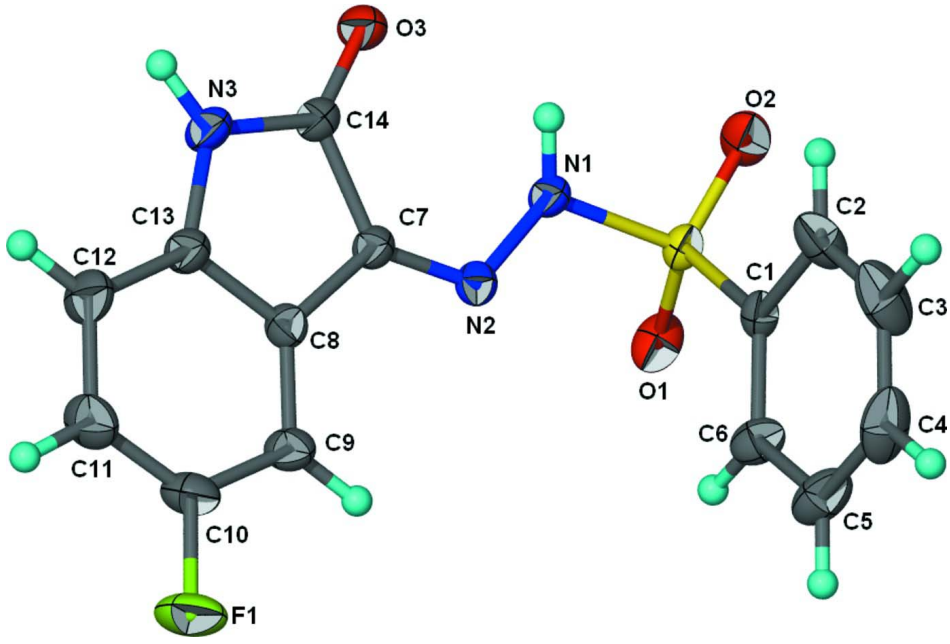

Figure 1

Thermal ellipsoid plot of $C_{14}H_{10}FN_3O_3S$. Displacement ellipsoids are drawn at the 70% probability level, and H atoms are shown as spheres of arbitrary radii.

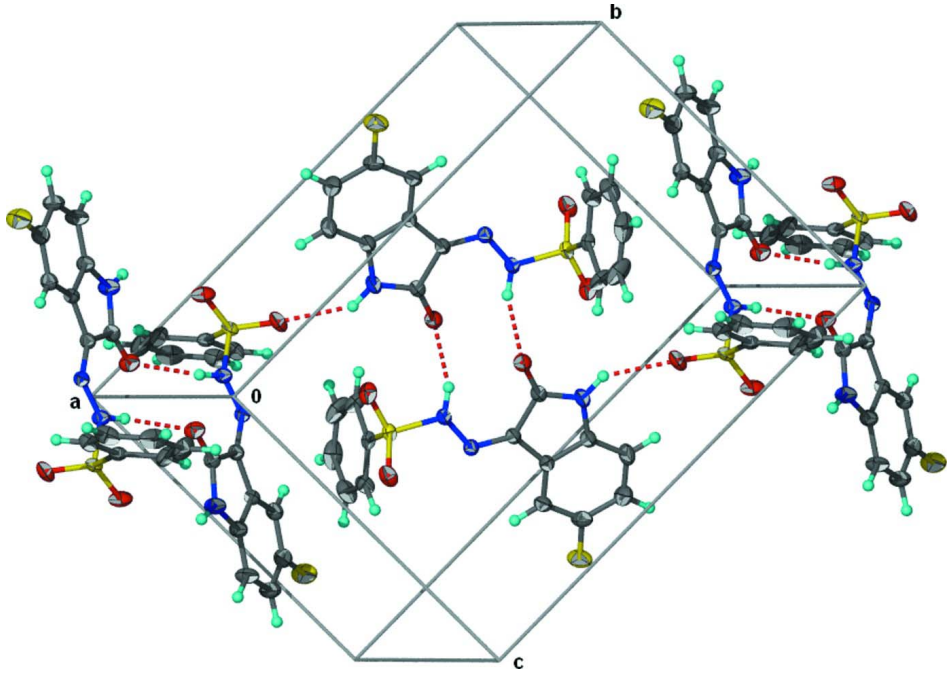

Figure 2

Ribbon structure of $C_{10}H_{10}FN_3O_3S$.

N′-(5-Fluoro-2-oxo-2,3-dihydro-1*H*-indol-3-ylidene)benzenesulfonohydrazide

Crystal data

$C_{14}H_{10}FN_3O_3S$
$M_r = 319.31$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.2218$ (2) Å
$b = 16.4933$ (3) Å
$c = 10.8585$ (2) Å
$\beta = 110.249$ (1)°
$V = 1381.46$ (5) Å^3
$Z = 4$
$F(000) = 656$
$D_x = 1.535$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6063 reflections
$\theta = 3.0$–$31.3°$
$\mu = 0.26$ mm^{-1}
$T = 123$ K
Irregular block, yellow
0.50 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: medium-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.816$, $T_{max} = 0.962$
10513 measured reflections
3166 independent reflections
2741 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.4°$
$h = -10 \rightarrow 10$
$k = -21 \rightarrow 21$
$l = -13 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.145$
$S = 1.20$
3166 reflections
207 parameters
2 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0922P)^2 + 0.2432P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.60$ e Å^{-3}

Special details

Experimental. A medium-focus collimator of 0.8 mm diameter was used on the diffractometer to measure the somewhat large crystal.

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.65386 (5)	0.28692 (2)	0.54366 (4)	0.01609 (16)
O1	0.61921 (18)	0.21966 (7)	0.61409 (14)	0.0248 (3)
O2	0.56524 (17)	0.29375 (8)	0.40497 (13)	0.0255 (3)
O3	0.42474 (17)	0.51614 (8)	0.60498 (13)	0.0240 (3)
N1	0.59808 (19)	0.37034 (9)	0.60384 (14)	0.0183 (3)

H1N	0.557 (3)	0.4089 (11)	0.546 (2)	0.036 (7)*
N2	0.67998 (18)	0.38149 (8)	0.73560 (14)	0.0172 (3)
N3	0.5337 (2)	0.57008 (9)	0.81558 (16)	0.0232 (3)
H3N	0.478 (3)	0.6168 (9)	0.799 (2)	0.038 (7)*
C1	0.8794 (2)	0.29132 (9)	0.58025 (17)	0.0165 (3)
C2	0.9467 (3)	0.33843 (13)	0.50285 (19)	0.0277 (4)
H2	0.8730	0.3696	0.4318	0.033*
C3	1.1256 (3)	0.33841 (16)	0.5329 (2)	0.0368 (5)
H3	1.1754	0.3703	0.4823	0.044*
C4	1.2315 (3)	0.29211 (13)	0.6361 (2)	0.0349 (5)
H4	1.3530	0.2914	0.6541	0.042*
C5	1.1624 (2)	0.24721 (12)	0.7127 (2)	0.0300 (4)
H5	1.2364	0.2164	0.7841	0.036*
C6	0.9846 (2)	0.24692 (10)	0.68578 (19)	0.0227 (4)
H6	0.9360	0.2167	0.7390	0.027*
C7	0.6472 (2)	0.44753 (10)	0.78623 (17)	0.0174 (3)
C8	0.7275 (2)	0.47125 (10)	0.92317 (17)	0.0182 (4)
C9	0.8561 (2)	0.43576 (10)	1.02774 (17)	0.0219 (4)
H9	0.9069	0.3853	1.0192	0.026*
C10	0.9065 (3)	0.47769 (11)	1.14518 (18)	0.0252 (4)
C11	0.8372 (3)	0.55171 (11)	1.16168 (19)	0.0285 (4)
H11	0.8765	0.5779	1.2447	0.034*
C12	0.7090 (3)	0.58763 (11)	1.05538 (19)	0.0276 (4)
H12	0.6597	0.6385	1.0641	0.033*
C13	0.6564 (2)	0.54660 (10)	0.93715 (18)	0.0204 (4)
C14	0.5206 (2)	0.51454 (10)	0.72023 (17)	0.0187 (4)
F1	1.03509 (17)	0.44573 (7)	1.25055 (11)	0.0374 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0133 (2)	0.0151 (2)	0.0192 (3)	−0.00149 (13)	0.00483 (17)	−0.00402 (14)
O1	0.0232 (7)	0.0164 (6)	0.0388 (8)	−0.0042 (5)	0.0157 (6)	−0.0018 (5)
O2	0.0199 (7)	0.0309 (7)	0.0207 (7)	−0.0001 (5)	0.0006 (5)	−0.0086 (5)
O3	0.0257 (7)	0.0238 (6)	0.0195 (6)	0.0050 (5)	0.0039 (5)	−0.0003 (5)
N1	0.0202 (7)	0.0158 (7)	0.0180 (7)	0.0032 (5)	0.0053 (6)	−0.0009 (5)
N2	0.0179 (7)	0.0163 (7)	0.0179 (7)	−0.0007 (5)	0.0069 (6)	−0.0010 (5)
N3	0.0250 (8)	0.0194 (7)	0.0219 (8)	0.0074 (6)	0.0039 (6)	−0.0024 (6)
C1	0.0132 (8)	0.0191 (8)	0.0172 (8)	−0.0017 (6)	0.0053 (6)	−0.0053 (6)
C2	0.0251 (9)	0.0409 (11)	0.0185 (9)	−0.0057 (8)	0.0093 (7)	0.0000 (8)
C3	0.0284 (10)	0.0596 (14)	0.0287 (11)	−0.0140 (10)	0.0178 (9)	−0.0058 (10)
C4	0.0153 (9)	0.0477 (13)	0.0424 (12)	−0.0030 (8)	0.0108 (9)	−0.0180 (10)
C5	0.0196 (9)	0.0262 (10)	0.0370 (11)	0.0035 (7)	0.0005 (8)	−0.0062 (8)
C6	0.0202 (8)	0.0180 (8)	0.0269 (9)	0.0000 (6)	0.0042 (7)	0.0001 (7)
C7	0.0182 (8)	0.0154 (7)	0.0191 (8)	0.0007 (6)	0.0072 (7)	0.0000 (6)
C8	0.0223 (8)	0.0144 (7)	0.0183 (8)	−0.0007 (6)	0.0076 (7)	−0.0013 (6)
C9	0.0266 (9)	0.0166 (8)	0.0207 (9)	0.0012 (6)	0.0059 (7)	0.0019 (6)
C10	0.0287 (9)	0.0225 (9)	0.0197 (9)	−0.0010 (7)	0.0023 (7)	0.0037 (7)

C11	0.0364 (11)	0.0251 (9)	0.0202 (9)	−0.0022 (8)	0.0052 (8)	−0.0058 (7)
C12	0.0327 (10)	0.0217 (9)	0.0251 (10)	0.0039 (8)	0.0059 (8)	−0.0073 (7)
C13	0.0216 (8)	0.0180 (8)	0.0206 (9)	0.0019 (6)	0.0060 (7)	−0.0008 (6)
C14	0.0184 (8)	0.0172 (8)	0.0206 (9)	0.0016 (6)	0.0069 (7)	0.0005 (6)
F1	0.0471 (8)	0.0298 (6)	0.0211 (6)	0.0055 (5)	−0.0061 (5)	0.0026 (5)

Geometric parameters (Å, º)

S1—O1	1.4309 (13)	C4—C5	1.375 (3)
S1—O2	1.4325 (14)	C4—H4	0.9500
S1—N1	1.6545 (14)	C5—C6	1.388 (3)
S1—C1	1.7582 (17)	C5—H5	0.9500
O3—C14	1.227 (2)	C6—H6	0.9500
N1—N2	1.367 (2)	C7—C8	1.456 (2)
N1—H1N	0.876 (10)	C7—C14	1.518 (2)
N2—C7	1.290 (2)	C8—C9	1.385 (2)
N3—C14	1.358 (2)	C8—C13	1.404 (2)
N3—C13	1.410 (2)	C9—C10	1.382 (3)
N3—H3N	0.884 (10)	C9—H9	0.9500
C1—C6	1.383 (2)	C10—F1	1.366 (2)
C1—C2	1.392 (3)	C10—C11	1.385 (3)
C2—C3	1.392 (3)	C11—C12	1.397 (3)
C2—H2	0.9500	C11—H11	0.9500
C3—C4	1.386 (3)	C12—C13	1.382 (3)
C3—H3	0.9500	C12—H12	0.9500
O1—S1—O2	119.99 (8)	C1—C6—C5	118.97 (18)
O1—S1—N1	107.46 (8)	C1—C6—H6	120.5
O2—S1—N1	103.90 (8)	C5—C6—H6	120.5
O1—S1—C1	107.50 (8)	N2—C7—C8	125.06 (15)
O2—S1—C1	110.32 (8)	N2—C7—C14	128.61 (16)
N1—S1—C1	106.93 (7)	C8—C7—C14	106.32 (14)
N2—N1—S1	114.96 (11)	C9—C8—C13	120.91 (16)
N2—N1—H1N	125.5 (17)	C9—C8—C7	132.16 (16)
S1—N1—H1N	114.2 (17)	C13—C8—C7	106.84 (15)
C7—N2—N1	117.39 (14)	C10—C9—C8	116.43 (16)
C14—N3—C13	111.76 (15)	C10—C9—H9	121.8
C14—N3—H3N	122.4 (17)	C8—C9—H9	121.8
C13—N3—H3N	125.6 (17)	F1—C10—C9	118.54 (17)
C6—C1—C2	122.02 (17)	F1—C10—C11	117.74 (17)
C6—C1—S1	118.29 (14)	C9—C10—C11	123.70 (17)
C2—C1—S1	119.69 (14)	C10—C11—C12	119.57 (17)
C1—C2—C3	117.85 (19)	C10—C11—H11	120.2
C1—C2—H2	121.1	C12—C11—H11	120.2
C3—C2—H2	121.1	C13—C12—C11	117.64 (17)
C4—C3—C2	120.4 (2)	C13—C12—H12	121.2
C4—C3—H3	119.8	C11—C12—H12	121.2
C2—C3—H3	119.8	C12—C13—C8	121.74 (17)

C5—C4—C3	120.72 (19)	C12—C13—N3	128.89 (16)
C5—C4—H4	119.6	C8—C13—N3	109.37 (15)
C3—C4—H4	119.6	O3—C14—N3	128.06 (16)
C4—C5—C6	119.97 (19)	O3—C14—C7	126.22 (15)
C4—C5—H5	120.0	N3—C14—C7	105.70 (15)
C6—C5—H5	120.0		

O1—S1—N1—N2	57.51 (14)	C14—C7—C8—C13	−0.74 (19)
O2—S1—N1—N2	−174.35 (12)	C13—C8—C9—C10	1.2 (3)
C1—S1—N1—N2	−57.66 (14)	C7—C8—C9—C10	177.29 (18)
S1—N1—N2—C7	176.78 (12)	C8—C9—C10—F1	−178.89 (16)
O1—S1—C1—C6	−14.56 (16)	C8—C9—C10—C11	−0.6 (3)
O2—S1—C1—C6	−147.07 (13)	F1—C10—C11—C12	178.16 (18)
N1—S1—C1—C6	100.57 (14)	C9—C10—C11—C12	−0.1 (3)
O1—S1—C1—C2	165.08 (14)	C10—C11—C12—C13	0.3 (3)
O2—S1—C1—C2	32.58 (16)	C11—C12—C13—C8	0.3 (3)
N1—S1—C1—C2	−79.78 (16)	C11—C12—C13—N3	−178.45 (19)
C6—C1—C2—C3	1.4 (3)	C9—C8—C13—C12	−1.1 (3)
S1—C1—C2—C3	−178.20 (16)	C7—C8—C13—C12	−178.09 (17)
C1—C2—C3—C4	0.5 (3)	C9—C8—C13—N3	177.87 (16)
C2—C3—C4—C5	−1.8 (3)	C7—C8—C13—N3	0.9 (2)
C3—C4—C5—C6	1.0 (3)	C14—N3—C13—C12	178.14 (19)
C2—C1—C6—C5	−2.2 (3)	C14—N3—C13—C8	−0.8 (2)
S1—C1—C6—C5	177.47 (14)	C13—N3—C14—O3	178.88 (18)
C4—C5—C6—C1	0.9 (3)	C13—N3—C14—C7	0.3 (2)
N1—N2—C7—C8	−177.31 (15)	N2—C7—C14—O3	0.9 (3)
N1—N2—C7—C14	3.5 (3)	C8—C7—C14—O3	−178.35 (17)
N2—C7—C8—C9	3.5 (3)	N2—C7—C14—N3	179.59 (17)
C14—C7—C8—C9	−177.21 (18)	C8—C7—C14—N3	0.29 (19)
N2—C7—C8—C13	179.93 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1n···O3^{i}	0.88 (1)	2.10 (2)	2.896 (2)	151 (2)
N3—H3n···O1^{ii}	0.88 (1)	2.22 (2)	2.986 (2)	145 (2)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1, *y*+1/2, −*z*+3/2.