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1-Methyl-2-methylsulfanyl-6-nitro-1*H*-benzimidazole

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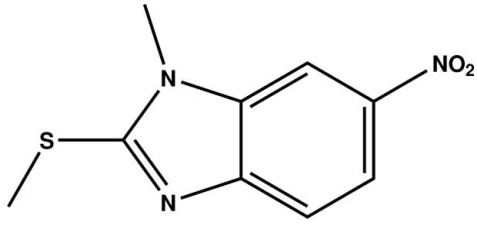
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.116; data-to-parameter ratio = 20.4.

The molecule of the title compound, $C_9H_9N_3O_2S$, is built up from fused five- and six-membered rings connected to methylsulfanyl and nitro groups, respectively. The mean plane through the fused ring system is inclined slightly relative to the plane passing through the nitro group [dihedral angle = 3.6 (2)°]. In the crystal, molecules are linked by C—H···O hydrogen bonds and π–π interactions between imidazole rings [inter-centroid distance = 3.667 (3) Å], forming a three-dimensional network.

Related literature

For the biological activity of benzimidazoles, see: Achar *et al.* (2010); Boiani & Gonzalez (2005); Ishida *et al.* (2006); Kamal *et al.* (2008); Kus *et al.* (2004); LaPlante *et al.* (2004).



Experimental

Crystal data

$C_9H_9N_3O_2S$
M_r = 223.25
Monoclinic, $P2_1/c$
a = 11.7213 (4) Å
b = 11.8991 (4) Å
c = 7.3025 (3) Å
β = 103.523 (1)°
V = 990.26 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 296 K
0.42 × 0.31 × 0.26 mm

Data collection

Bruker X8 APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.658, T_{max} = 0.746
11751 measured reflections
2772 independent reflections
2350 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.116
S = 1.07
2772 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···O1^{i}	0.96	2.53	3.393 (2)	150
C3—H3···O2^{ii}	0.93	2.65	3.3038 (19)	128
C9—H9A···O2^{iii}	0.96	2.67	3.563 (2)	155

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5298).

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1-Methyl-2-methylsulfanyl-6-nitro-1*H*-benzimidazole

Mohamed El Ghozlani, El Mostapha Rakib, Abdelouahid Medaghri-Alaoui, Mohamed Saadi and Lahcen El Ammari

S1. Structural commentary

Benzimidazole derivatives are of wide interest because of their diverse biological activities and clinical applications. This fused-ring system exhibits a broad spectrum of biological activities, that is, anti-cancer, anti-viral, anti-bacterial, anti-inflammatory, anti-oxidant and anti-leukaemic (LaPlante *et al.*, 2004; Ishida *et al.*, 2006; Boiani & Gonzalez, 2005; Achar *et al.*, 2010; Kus *et al.*, 2004; Kamal *et al.*, 2008).

The molecule of the title compound, $C_9H_9N_3O_2S$, is formed by a fused five- and six-membered rings as shown in Fig.1. The mean plane through the fused ring system (N1,N2,C1 to C7) is slightly inclined relative to the mean plane passing through the nitro group with a dihedral angle of 3.6 (2)°. In the crystal, molecules are linked by C—H···O hydrogen bonds and π–π interactions between indazole rings [inter-centroid distance = 3.667 (3) Å], forming a three-dimensional network as shown in Fig.2 and Table 1.

S2. Synthesis and crystallization

To a solution of 5-nitro-1*H*-benzimidazole-2-thiol (5.12 mmol) in DMSO (15 ml) was added potassium carbonate (5.2 mmol). After 15 min. at 298 K, methyl iodide (7.68 mmol) was added drop wise. Upon disappearance of the starting material, as indicated by TLC, the resulting mixture was evaporated. The crude material was dissolved with EtOAc (60 ml), washed with water and brine, dried over $MgSO_4$ and the solvent was evaporated *in vacuo*. The resulting residue was purified by column chromatography (EtOAc/hexane 3/7). The title compound was recrystallized from acetone at room temperature giving colourless crystals (M.pt: 475 K, yield: 47%).

S3. Refinement

H atoms were located in a difference map and treated as riding with C–H = 0.96 Å and C–H = 0.93 Å for methyl- and aromatic-H, respectively. All hydrogen with U_{iso}(H) = 1.5 U_{eq} for methyl-H and U_{iso}(H) = 1.2 U_{eq} for aromatic-H.

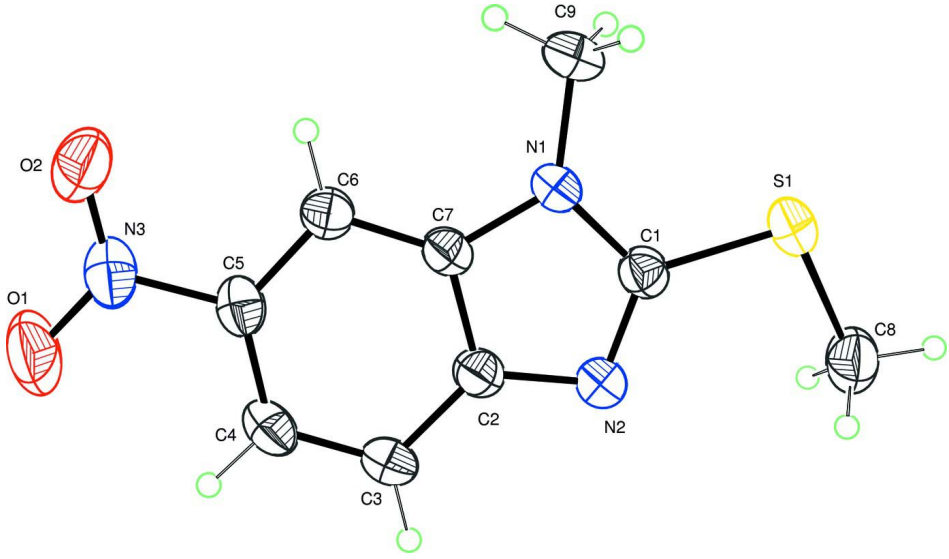

Figure 1

Plot of the molecule of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

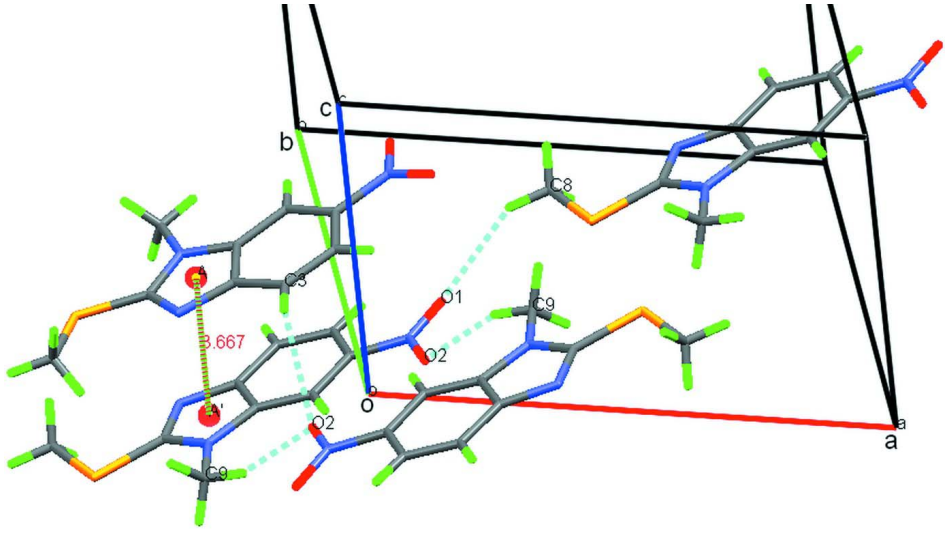

Figure 2

Partial crystal packing for the title compound showing molecules linked by π–π interactions and hydrogen bonds (dashed lines).

1-Methyl-2-methylsulfanyl-6-nitro-1*H*-benzimidazole

Crystal data

$C_9H_9N_3O_2S$
$M_r = 223.25$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.7213$ (4) Å
$b = 11.8991$ (4) Å
$c = 7.3025$ (3) Å
$\beta = 103.523$ (1)°
$V = 990.26$ (6) Å^3
$Z = 4$
$F(000) = 464$
$D_x = 1.497$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2772 reflections

θ = 2.5–29.6°
μ = 0.31 mm^{-1}
T = 296 K

Block, colourless
0.42 × 0.31 × 0.26 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.658, T_{max} = 0.746

11751 measured reflections
2772 independent reflections
2350 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 29.6°, θ_{min} = 2.5°
h = −16→16
k = −16→15
l = −10→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.116
S = 1.07
2772 reflections
136 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0644P)^2 + 0.2102P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.37904 (11)	0.74865 (11)	0.42804 (17)	0.0319 (3)
C2	0.21992 (11)	0.66649 (11)	0.29323 (17)	0.0315 (3)
C3	0.13098 (12)	0.58951 (11)	0.2205 (2)	0.0385 (3)
H3	0.1435	0.5126	0.2365	0.046*
C4	0.02393 (12)	0.63119 (12)	0.1243 (2)	0.0388 (3)
H4	−0.0373	0.5820	0.0752	0.047*
C5	0.00707 (11)	0.74680 (12)	0.10023 (18)	0.0344 (3)
C6	0.09247 (11)	0.82662 (11)	0.17081 (17)	0.0331 (3)
H6	0.0793	0.9034	0.1538	0.040*
C7	0.19868 (11)	0.78276 (10)	0.26849 (16)	0.0295 (3)
C8	0.58418 (14)	0.64247 (14)	0.5740 (2)	0.0502 (4)
H8A	0.6651	0.6472	0.6400	0.075*
H8B	0.5788	0.6101	0.4518	0.075*

H8C	0.5424	0.5961	0.6440	0.075*
C9	0.32546 (14)	0.95396 (12)	0.3712 (3)	0.0487 (4)
H9A	0.2561	0.9940	0.3088	0.073*
H9B	0.3884	0.9716	0.3123	0.073*
H9C	0.3470	0.9756	0.5013	0.073*
N1	0.30262 (9)	0.83409 (9)	0.35759 (15)	0.0326 (2)
N2	0.33342 (10)	0.64715 (10)	0.39349 (16)	0.0353 (2)
N3	−0.10606 (11)	0.78650 (12)	−0.00931 (19)	0.0451 (3)
O1	−0.17998 (12)	0.71648 (13)	−0.0807 (2)	0.0775 (5)
O2	−0.12317 (11)	0.88741 (11)	−0.02760 (19)	0.0610 (3)
S1	0.52130 (3)	0.78014 (3)	0.54830 (5)	0.04086 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0322 (6)	0.0338 (6)	0.0290 (6)	−0.0034 (5)	0.0058 (4)	−0.0008 (5)
C2	0.0328 (6)	0.0299 (6)	0.0312 (6)	−0.0036 (4)	0.0066 (4)	−0.0010 (4)
C3	0.0407 (7)	0.0282 (6)	0.0445 (7)	−0.0073 (5)	0.0062 (5)	−0.0029 (5)
C4	0.0351 (6)	0.0374 (7)	0.0426 (7)	−0.0098 (5)	0.0062 (5)	−0.0089 (6)
C5	0.0290 (6)	0.0422 (7)	0.0312 (6)	−0.0004 (5)	0.0053 (5)	−0.0059 (5)
C6	0.0342 (6)	0.0306 (6)	0.0340 (6)	0.0001 (5)	0.0068 (5)	−0.0027 (5)
C7	0.0311 (6)	0.0291 (6)	0.0281 (5)	−0.0047 (4)	0.0067 (4)	−0.0022 (4)
C8	0.0382 (7)	0.0525 (9)	0.0537 (9)	0.0044 (6)	−0.0016 (6)	−0.0025 (7)
C9	0.0460 (8)	0.0291 (7)	0.0657 (10)	−0.0091 (6)	0.0024 (7)	−0.0022 (6)
N1	0.0318 (5)	0.0282 (5)	0.0356 (5)	−0.0054 (4)	0.0032 (4)	−0.0020 (4)
N2	0.0338 (5)	0.0316 (5)	0.0379 (5)	−0.0027 (4)	0.0033 (4)	0.0007 (4)
N3	0.0337 (6)	0.0560 (8)	0.0427 (6)	0.0032 (5)	0.0032 (5)	−0.0095 (5)
O1	0.0439 (7)	0.0760 (10)	0.0950 (11)	−0.0049 (6)	−0.0193 (7)	−0.0228 (8)
O2	0.0467 (6)	0.0567 (8)	0.0720 (8)	0.0149 (5)	−0.0012 (6)	−0.0006 (6)
S1	0.0342 (2)	0.0423 (2)	0.0412 (2)	−0.00665 (13)	−0.00108 (13)	−0.00503 (13)

Geometric parameters (Å, º)

C1—N2	1.3210 (17)	C6—H6	0.9300
C1—N1	1.3730 (17)	C7—N1	1.3819 (15)
C1—S1	1.7342 (13)	C8—S1	1.7882 (17)
C2—N2	1.3796 (16)	C8—H8A	0.9600
C2—C3	1.3960 (17)	C8—H8B	0.9600
C2—C7	1.4098 (18)	C8—H8C	0.9600
C3—C4	1.379 (2)	C9—N1	1.4504 (17)
C3—H3	0.9300	C9—H9A	0.9600
C4—C5	1.395 (2)	C9—H9B	0.9600
C4—H4	0.9300	C9—H9C	0.9600
C5—C6	1.3888 (18)	N3—O2	1.2196 (18)
C5—N3	1.4578 (18)	N3—O1	1.2270 (18)
C6—C7	1.3841 (17)		
N2—C1—N1	113.95 (11)	S1—C8—H8A	109.5

N2—C1—S1	126.34 (11)	S1—C8—H8B	109.5
N1—C1—S1	119.71 (10)	H8A—C8—H8B	109.5
N2—C2—C3	129.35 (12)	S1—C8—H8C	109.5
N2—C2—C7	110.56 (11)	H8A—C8—H8C	109.5
C3—C2—C7	120.09 (12)	H8B—C8—H8C	109.5
C4—C3—C2	117.86 (12)	N1—C9—H9A	109.5
C4—C3—H3	121.1	N1—C9—H9B	109.5
C2—C3—H3	121.1	H9A—C9—H9B	109.5
C3—C4—C5	120.27 (12)	N1—C9—H9C	109.5
C3—C4—H4	119.9	H9A—C9—H9C	109.5
C5—C4—H4	119.9	H9B—C9—H9C	109.5
C6—C5—C4	123.99 (12)	C1—N1—C7	105.98 (10)
C6—C5—N3	117.79 (13)	C1—N1—C9	127.50 (11)
C4—C5—N3	118.20 (12)	C7—N1—C9	126.52 (12)
C7—C6—C5	114.64 (12)	C1—N2—C2	104.24 (11)
C7—C6—H6	122.7	O2—N3—O1	122.70 (14)
C5—C6—H6	122.7	O2—N3—C5	118.97 (13)
N1—C7—C6	131.58 (12)	O1—N3—C5	118.32 (14)
N1—C7—C2	105.28 (11)	C1—S1—C8	100.32 (7)
C6—C7—C2	123.13 (11)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O1^{i}	0.96	2.53	3.393 (2)	150
C3—H3···O2^{ii}	0.93	2.65	3.3038 (19)	128
C9—H9*A*···O2^{iii}	0.96	2.67	3.563 (2)	155

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