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Benzyl *N*′-(1-methyl-1*H*-indol-3-ylmethylidene)hydrazinecarbodithioate

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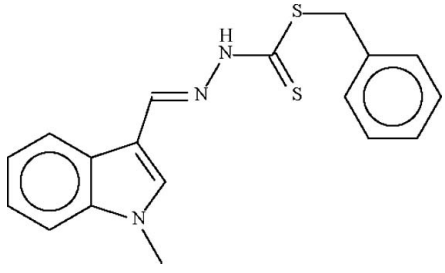
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.037; wR factor = 0.087; data-to-parameter ratio = 15.4.

The *N*′-(1-methyl-1*H*-indol-3-ylmethylidene)hydrazinecarbodithioate portion of the title molecule, $C_{18}H_{17}N_3S_2$, is nearly planar; this unit and the phenyl ring subtend an angle of 112.9 (2)° at the methylene C atom.

Related literature

For the structure of *S*-benzyl *N*-1-(1*H*-indol-3-ylmethylidenehydrazinecarbodithioate, see: Khaledi *et al.* (2008).



Experimental

Crystal data

$C_{18}H_{17}N_3S_2$ M_r = 339.47
Monoclinic, $P2_1$
a = 10.6111 (4) Å
b = 6.1134 (2) Å
c = 13.4961 (4) Å
β = 111.934 (2)°
V = 812.12 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 100 (2) K
0.30 × 0.10 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.908, T_{max} = 0.993
5513 measured reflections
3277 independent reflections
2802 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.087
S = 1.03
3277 reflections
213 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack (1983), 1261 Friedel pairs
Flack parameter: 0.01 (8)

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2334).

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supporting information

Acta Cryst. (2008). E**64**, o2482 [doi:10.1107/S1600536808039330]

Benzyl *N*′-(1-methyl-1*H*-indol-3-ylmethylidene)hydrazinecarbodithioate

Hamid Khaledi, Hapipah Mohd Ali and Seik Weng Ng

S1. Experimental

N-Methylindole-3-carbaldehyde (1.59 g, 10 mmol) and *S*-benzyl dithiocarbazate (1.98 g, 10 mmol) were heated in ethanol (60 ml) for 1 h. Several drops of acetic acid were added. The solution yielded a solid on cooling. This was recrystallized from DMSO.

S2. Refinement

Hydrogen atoms were placed at calculated positions (C–H 0.95–0.99 Å) and were treated as riding on their parent carbon atoms, with *U*(H) set to 1.2 times U_{eq}(C), 1.5 for methyl-C. The amino H-atom was located in a difference Fourier map, and was refined with a distance restraint of N–H 0.88±0.01 Å; it does not form a hydrogen bond.

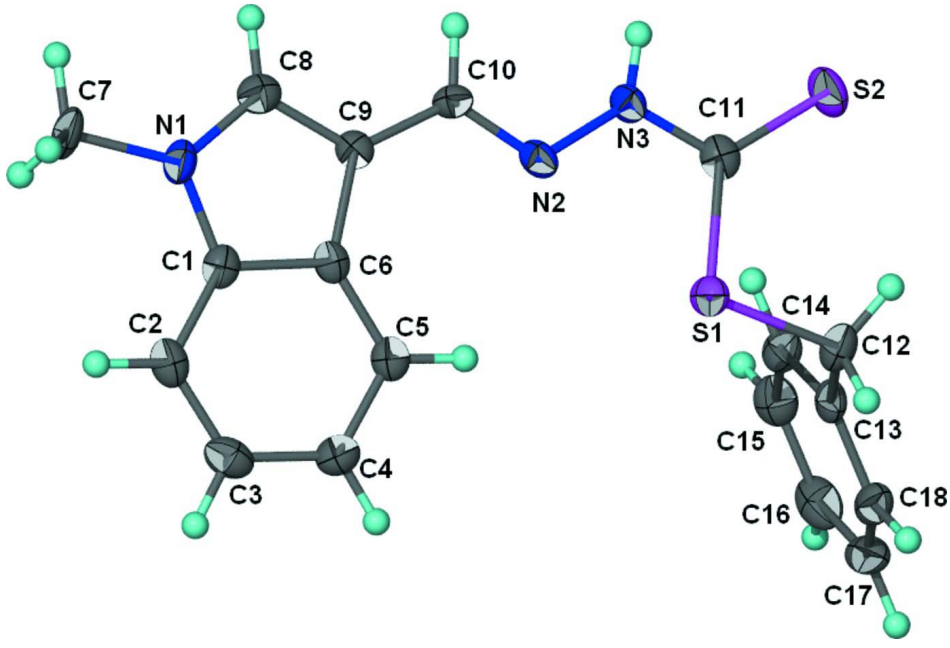

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{18}H_{17}N_2S_3$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

Benzyl *N*′-(1-methyl-1*H*-indol-3-ylmethylidene)hydrazinecarbodithioate

Crystal data

$C_{18}H_{17}N_3S_2$
$M_r = 339.47$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 10.6111\ (4)$ Å
$b = 6.1134\ (2)$ Å
$c = 13.4961\ (4)$ Å
$\beta = 111.934\ (2)°$
$V = 812.12\ (5)$ Å^3
$Z = 2$
$F(000) = 356$
$D_x = 1.388$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1721 reflections
$\theta = 3.1–27.1°$
$\mu = 0.33$ mm^{-1}
$T = 100$ K
Plate, yellow
0.30 × 0.10 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.908$, $T_{max} = 0.993$
5513 measured reflections
3277 independent reflections
2802 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.6°$
$h = -13\rightarrow13$
$k = -6\rightarrow7$
$l = -17\rightarrow17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.087$
$S = 1.03$
3277 reflections
213 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: constr
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 0.212P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Absolute structure: Flack (1983), 1261 Friedel pairs
Absolute structure parameter: 0.01 (8)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.33191 (7)	0.50000 (12)	0.67816 (5)	0.01960 (16)
S2	0.48123 (7)	0.86601 (13)	0.61270 (5)	0.02642 (18)
N1	0.2902 (2)	0.7662 (4)	1.16590 (16)	0.0202 (5)
N2	0.3791 (2)	0.7502 (4)	0.85695 (16)	0.0181 (5)
N3	0.4323 (2)	0.8477 (4)	0.78860 (16)	0.0183 (5)
H3	0.480 (3)	0.967 (3)	0.796 (2)	0.035 (9)*
C1	0.2337 (3)	0.5789 (5)	1.1079 (2)	0.0178 (6)
C2	0.1584 (3)	0.4147 (5)	1.1303 (2)	0.0215 (6)
H2	0.1422	0.4143	1.1950	0.026*
C3	0.1075 (3)	0.2515 (5)	1.0548 (2)	0.0236 (6)
H3A	0.0531	0.1389	1.0668	0.028*
C4	0.1347 (3)	0.2496 (5)	0.9610 (2)	0.0215 (6)

H4	0.0983	0.1354	0.9106	0.026*
C5	0.2133 (3)	0.4101 (4)	0.9399 (2)	0.0193 (6)
H5	0.2326	0.4051	0.8767	0.023*
C6	0.2637 (3)	0.5795 (4)	1.01374 (19)	0.0166 (6)
C7	0.2743 (3)	0.8324 (5)	1.2645 (2)	0.0261 (7)
H7A	0.3162	0.9761	1.2867	0.039*
H7B	0.3186	0.7249	1.3206	0.039*
H7C	0.1774	0.8405	1.2524	0.039*
C8	0.3524 (3)	0.8815 (5)	1.11224 (19)	0.0201 (6)
H8	0.3981	1.0166	1.1357	0.024*
C9	0.3409 (3)	0.7767 (4)	1.01840 (19)	0.0168 (6)
C10	0.3932 (2)	0.8586 (5)	0.94249 (18)	0.0166 (5)
H10	0.4392	0.9952	0.9551	0.020*
C11	0.4177 (3)	0.7519 (5)	0.6953 (2)	0.0197 (6)
C12	0.3161 (3)	0.4207 (5)	0.54473 (19)	0.0229 (6)
H12A	0.3840	0.5019	0.5251	0.028*
H12B	0.3366	0.2627	0.5445	0.028*
C13	0.1765 (3)	0.4642 (5)	0.46213 (19)	0.0192 (6)
C14	0.1145 (3)	0.6657 (5)	0.4536 (2)	0.0220 (6)
H14	0.1606	0.7800	0.5010	0.026*
C15	−0.0137 (3)	0.7043 (5)	0.3773 (2)	0.0258 (7)
H15	−0.0550	0.8438	0.3726	0.031*
C16	−0.0816 (3)	0.5379 (6)	0.3078 (2)	0.0295 (8)
H16	−0.1693	0.5638	0.2552	0.035*
C17	−0.0220 (3)	0.3353 (5)	0.3150 (2)	0.0280 (7)
H17	−0.0686	0.2212	0.2677	0.034*
C18	0.1073 (3)	0.2984 (5)	0.3919 (2)	0.0240 (7)
H18	0.1486	0.1589	0.3966	0.029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0229 (3)	0.0209 (4)	0.0149 (3)	−0.0023 (3)	0.0070 (2)	0.0006 (3)
S2	0.0317 (4)	0.0286 (4)	0.0252 (3)	−0.0013 (4)	0.0179 (3)	0.0058 (3)
N1	0.0225 (12)	0.0243 (13)	0.0158 (10)	0.0007 (10)	0.0094 (9)	−0.0026 (9)
N2	0.0191 (11)	0.0187 (13)	0.0176 (10)	−0.0022 (10)	0.0082 (9)	0.0036 (9)
N3	0.0232 (12)	0.0166 (13)	0.0178 (10)	−0.0018 (10)	0.0109 (9)	0.0023 (10)
C1	0.0179 (14)	0.0190 (15)	0.0159 (12)	0.0050 (12)	0.0056 (11)	0.0017 (11)
C2	0.0210 (14)	0.0248 (17)	0.0208 (13)	0.0049 (12)	0.0103 (11)	0.0051 (11)
C3	0.0217 (14)	0.0195 (16)	0.0298 (14)	−0.0007 (13)	0.0097 (12)	0.0052 (12)
C4	0.0225 (14)	0.0195 (15)	0.0208 (13)	−0.0007 (12)	0.0062 (11)	−0.0012 (12)
C5	0.0192 (13)	0.0213 (16)	0.0178 (12)	0.0031 (12)	0.0072 (11)	0.0025 (11)
C6	0.0145 (13)	0.0187 (15)	0.0169 (12)	0.0041 (11)	0.0063 (10)	0.0031 (11)
C7	0.0369 (17)	0.0297 (18)	0.0160 (12)	0.0016 (14)	0.0148 (12)	−0.0056 (12)
C8	0.0173 (13)	0.0194 (15)	0.0216 (12)	0.0007 (13)	0.0049 (10)	−0.0003 (12)
C9	0.0156 (13)	0.0182 (15)	0.0157 (12)	0.0003 (11)	0.0048 (10)	0.0016 (11)
C10	0.0180 (13)	0.0136 (13)	0.0168 (11)	−0.0014 (12)	0.0049 (10)	−0.0002 (11)
C11	0.0176 (13)	0.0225 (16)	0.0185 (12)	0.0038 (12)	0.0061 (11)	0.0040 (11)

C12	0.0298 (15)	0.0225 (16)	0.0182 (12)	0.0035 (12)	0.0109 (12)	−0.0016 (11)
C13	0.0230 (13)	0.0242 (17)	0.0130 (11)	−0.0008 (12)	0.0099 (10)	0.0005 (11)
C14	0.0230 (15)	0.0266 (17)	0.0157 (12)	−0.0016 (12)	0.0064 (11)	−0.0021 (11)
C15	0.0281 (16)	0.0256 (17)	0.0254 (14)	0.0045 (13)	0.0122 (12)	0.0060 (13)
C16	0.0244 (15)	0.044 (2)	0.0176 (12)	−0.0029 (14)	0.0055 (11)	0.0064 (13)
C17	0.0353 (17)	0.0324 (19)	0.0169 (12)	−0.0157 (15)	0.0105 (12)	−0.0049 (13)
C18	0.0366 (17)	0.0196 (16)	0.0202 (13)	−0.0043 (13)	0.0159 (13)	−0.0011 (11)

Geometric parameters (Å, º)

S1—C11	1.759 (3)	C7—H7A	0.9800
S1—C12	1.811 (3)	C7—H7B	0.9800
S2—C11	1.657 (3)	C7—H7C	0.9800
N1—C8	1.347 (3)	C8—C9	1.383 (4)
N1—C1	1.390 (4)	C8—H8	0.9500
N1—C7	1.460 (3)	C9—C10	1.426 (4)
N2—C10	1.290 (3)	C10—H10	0.9500
N2—N3	1.384 (3)	C12—C13	1.508 (4)
N3—C11	1.345 (3)	C12—H12A	0.9900
N3—H3	0.875 (10)	C12—H12B	0.9900
C1—C2	1.386 (4)	C13—C14	1.381 (4)
C1—C6	1.420 (4)	C13—C18	1.394 (4)
C2—C3	1.384 (4)	C14—C15	1.386 (4)
C2—H2	0.9500	C14—H14	0.9500
C3—C4	1.400 (4)	C15—C16	1.389 (4)
C3—H3A	0.9500	C15—H15	0.9500
C4—C5	1.385 (4)	C16—C17	1.378 (5)
C4—H4	0.9500	C16—H16	0.9500
C5—C6	1.398 (4)	C17—C18	1.395 (4)
C5—H5	0.9500	C17—H17	0.9500
C6—C9	1.445 (4)	C18—H18	0.9500
C11—S1—C12	102.48 (13)	C9—C8—H8	124.6
C8—N1—C1	108.8 (2)	C8—C9—C10	124.8 (3)
C8—N1—C7	126.3 (3)	C8—C9—C6	106.3 (2)
C1—N1—C7	124.7 (2)	C10—C9—C6	128.9 (2)
C10—N2—N3	115.8 (2)	N2—C10—C9	121.1 (3)
C11—N3—N2	120.3 (2)	N2—C10—H10	119.5
C11—N3—H3	109 (2)	C9—C10—H10	119.5
N2—N3—H3	131 (2)	N3—C11—S2	120.9 (2)
C2—C1—N1	129.5 (2)	N3—C11—S1	112.2 (2)
C2—C1—C6	122.6 (3)	S2—C11—S1	126.94 (17)
N1—C1—C6	108.0 (2)	C13—C12—S1	112.87 (19)
C3—C2—C1	117.4 (2)	C13—C12—H12A	109.0
C3—C2—H2	121.3	S1—C12—H12A	109.0
C1—C2—H2	121.3	C13—C12—H12B	109.0
C2—C3—C4	121.1 (3)	S1—C12—H12B	109.0
C2—C3—H3A	119.4	H12A—C12—H12B	107.8

C4—C3—H3A	119.4	C14—C13—C18	118.6 (2)
C5—C4—C3	121.6 (3)	C14—C13—C12	121.6 (2)
C5—C4—H4	119.2	C18—C13—C12	119.8 (3)
C3—C4—H4	119.2	C13—C14—C15	121.2 (3)
C4—C5—C6	118.6 (2)	C13—C14—H14	119.4
C4—C5—H5	120.7	C15—C14—H14	119.4
C6—C5—H5	120.7	C14—C15—C16	119.7 (3)
C5—C6—C1	118.8 (2)	C14—C15—H15	120.2
C5—C6—C9	135.2 (2)	C16—C15—H15	120.2
C1—C6—C9	106.0 (2)	C17—C16—C15	120.2 (3)
N1—C7—H7A	109.5	C17—C16—H16	119.9
N1—C7—H7B	109.5	C15—C16—H16	119.9
H7A—C7—H7B	109.5	C16—C17—C18	119.7 (3)
N1—C7—H7C	109.5	C16—C17—H17	120.2
H7A—C7—H7C	109.5	C18—C17—H17	120.2
H7B—C7—H7C	109.5	C13—C18—C17	120.7 (3)
N1—C8—C9	110.9 (3)	C13—C18—H18	119.7
N1—C8—H8	124.6	C17—C18—H18	119.7

C10—N2—N3—C11	−177.2 (2)	C1—C6—C9—C8	−0.2 (3)
C8—N1—C1—C2	−178.6 (3)	C5—C6—C9—C10	−1.0 (5)
C7—N1—C1—C2	−3.0 (4)	C1—C6—C9—C10	−178.3 (3)
C8—N1—C1—C6	0.3 (3)	N3—N2—C10—C9	179.1 (2)
C7—N1—C1—C6	175.9 (2)	C8—C9—C10—N2	179.5 (3)
N1—C1—C2—C3	176.3 (3)	C6—C9—C10—N2	−2.7 (4)
C6—C1—C2—C3	−2.4 (4)	N2—N3—C11—S2	−179.82 (19)
C1—C2—C3—C4	1.8 (4)	N2—N3—C11—S1	−1.8 (3)
C2—C3—C4—C5	0.0 (4)	C12—S1—C11—N3	175.97 (19)
C3—C4—C5—C6	−1.3 (4)	C12—S1—C11—S2	−6.1 (2)
C4—C5—C6—C1	0.8 (4)	C11—S1—C12—C13	−101.3 (2)
C4—C5—C6—C9	−176.2 (3)	S1—C12—C13—C14	50.8 (3)
C2—C1—C6—C5	1.1 (4)	S1—C12—C13—C18	−129.3 (2)
N1—C1—C6—C5	−177.9 (2)	C18—C13—C14—C15	0.0 (4)
C2—C1—C6—C9	178.9 (2)	C12—C13—C14—C15	179.9 (2)
N1—C1—C6—C9	−0.1 (3)	C13—C14—C15—C16	−0.1 (4)
C1—N1—C8—C9	−0.4 (3)	C14—C15—C16—C17	0.3 (4)
C7—N1—C8—C9	−175.9 (3)	C15—C16—C17—C18	−0.4 (4)
N1—C8—C9—C10	178.6 (2)	C14—C13—C18—C17	−0.2 (4)
N1—C8—C9—C6	0.4 (3)	C12—C13—C18—C17	180.0 (2)
C5—C6—C9—C8	177.1 (3)	C16—C17—C18—C13	0.4 (4)