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

Hamid Khaledi, Hapipah Mohd Ali and Seik Weng Ng*

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

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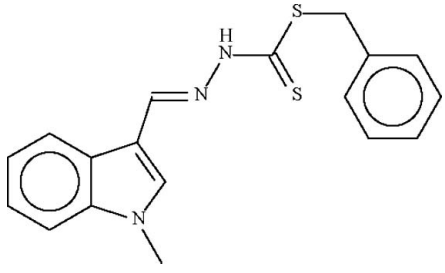
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.037; wR factor = 0.087; data-to-parameter ratio = 15.4.

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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supplementary materials

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

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

H. Khaledi, H. Mohd Ali and S. W. Ng

Comment

(type here to add)

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.

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.

Figures

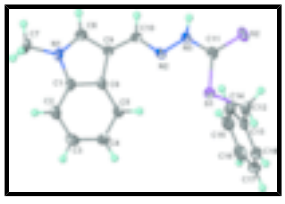


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{18}H_{17}N_2S_3$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

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

Crystal data

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

$Z = 2$

Data collection

Bruker SMART APEX diffractometer	3277 independent reflections
Radiation source: fine-focus sealed tube	2802 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.027$
$T = 100(2)$ K	$\theta_{max} = 27.5°$
ω scans	$\theta_{min} = 1.6°$
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	$h = -13\rightarrow13$
$T_{min} = 0.908$, $T_{max} = 0.993$	$k = -6\rightarrow7$
5513 measured reflections	$l = -17\rightarrow17$

Refinement

Refinement on F^2	Hydrogen site location: constr
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.037$	$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 0.212P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.087$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.03$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
3277 reflections	$\Delta\rho_{min} = -0.28$ e Å^{-3}
213 parameters	Extinction correction: none
2 restraints	Absolute structure: Flack (1983), 1261 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.01 (8)
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	0.33191 (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 (Å^2)

	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)

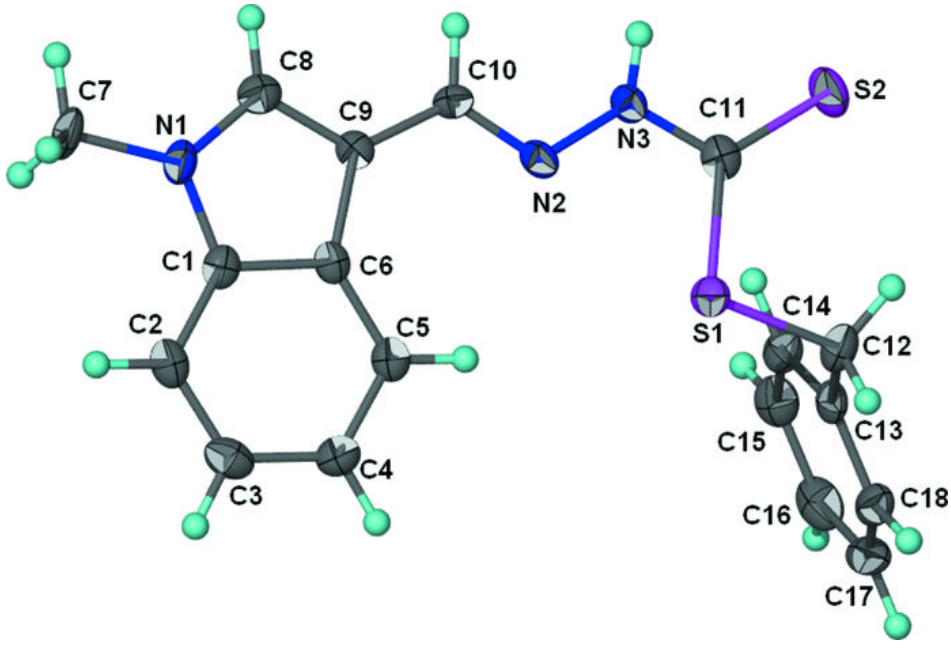
C14	0.0230 (15)	0.0266 (17)	0.0157 (12)	−0.0016 (12)	0.0064 (11)	−0.0021 (11)
C15	0.0281 (16)	0.0256 (17)	0.0254 (14)	0.0045 (13)	0.0122 (12)	0.0060 (13)
C16	0.0244 (15)	0.044 (2)	0.0176 (12)	−0.0029 (14)	0.0055 (11)	0.0064 (13)
C17	0.0353 (17)	0.0324 (19)	0.0169 (12)	−0.0157 (15)	0.0105 (12)	−0.0049 (13)
C18	0.0366 (17)	0.0196 (16)	0.0202 (13)	−0.0043 (13)	0.0159 (13)	−0.0011 (11)

Geometric parameters (Å, °)

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)

Fig. 1


C7
N1
C8
C9
C10
N2
N3
C11
S2
C1
C6
C2
C5
S1
C14
C12
C15
C13
C4
C3
C16
C18
C17