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Benzyl *N'*-(1*H*-indol-2-ylmethylene)-hydrazinecarbodithioate ethanol hemisolvate

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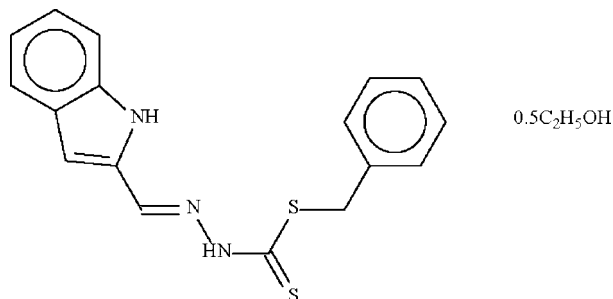
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 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; disorder in solvent or counterion; R factor = 0.033; wR factor = 0.088; data-to-parameter ratio = 16.8.

In the crystal of the title compound, $\text{C}_{17}\text{H}_{15}\text{N}_3\text{S}_2 \cdot 0.5\text{C}_2\text{H}_6\text{O}$, the molecules are linked by a pair of $\text{N}-\text{H}_{\text{aliphatic}} \cdots \text{S}$ hydrogen bonds across a center of inversion, forming a dimer. The ethanol solvent molecule, which is statistically disordered about a crystallographic twofold rotation axis, accepts an $\text{N}-\text{H}_{\text{aromatic}} \cdots \text{O}$ hydrogen bond; the hydroxy group of the solvent molecule is not engaged in hydrogen bonding.

Related literature

 For references to benzyl esters of hydrazinecarbodithioic acids, see: Khaledi *et al.* (2008).


Experimental

Crystal data

 $\text{C}_{17}\text{H}_{15}\text{N}_3\text{S}_2 \cdot 0.5\text{C}_2\text{H}_6\text{O}$
 $M_r = 348.47$

 Monoclinic, $C2/c$
 $a = 13.4225$ (2) Å
 $b = 15.4088$ (2) Å
 $c = 16.8120$ (3) Å
 $\beta = 102.637$ (1)°
 $V = 3392.90$ (9) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.32$ mm⁻¹
 $T = 100$ (2) K
 $0.25 \times 0.20 \times 0.15$ mm

Data collection

 Bruker SMART APEX CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.924$, $T_{\text{max}} = 0.954$

 14302 measured reflections
 3897 independent reflections
 3332 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.088$
 $S = 1.03$
 3897 reflections
 232 parameters
 5 restraints

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.35$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.40$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N1}-\text{H1}n \cdots \text{S2}^i$	0.88 (1)	2.52 (1)	3.350 (1)	159 (2)
$\text{N3}-\text{H3}n \cdots \text{O1}$	0.87 (1)	2.25 (1)	3.047 (3)	153 (2)

 Symmetry code: (i) $-x + 1, -y + 1, -z + 1$.

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: pubCIF (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2851).

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supplementary materials

Acta Cryst. (2008). E64, o2445 [doi:10.1107/S1600536808038609]

Benzyl *N'*-(1*H*-indol-2-ylmethylene)hydrazinecarbodithioate ethanol hemisolvate

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

Comment

For background references, see: Khaledi *et al.* (2008).

Experimental

Indole-2-carbaldehyde (0.36 g, 2.5 mmol) and *S*-benzylidithiocarbazate (0.495 g, 2.5 mmol) were heated in ethanol (40 ml) for 3 h. The solution was set aside for the formation of yellow blocks of (I).

Refinement

C-bound hydrogen atoms were placed at calculated positions (C–H = 0.95–0.99 Å) and refined as riding with $U(H) = 1.2$ – 1.5 times $U_{eq}(C)$. The amino and hydroxy H atoms were located in a difference map, and were refined with distance restraints of N–H = 0.88 ± 0.01 and O–H = $0.84 +_{-0.01}$ Å; their U_{iso} values were freely refined.

The ethanol molecule is statistically disordered about a two-fold axis: the O–C distance was restrained to 1.45 ± 0.01 Å and the C–C distance to $1.50 +_{-0.01}$ Å; the displacement factors of the two C atoms were restrained to be equal.

Figures

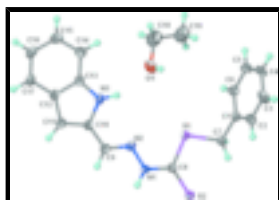


Fig. 1. View of (I) at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius. Only one orientation of the ethanol molecule is shown.

Benzyl *N'*-(1*H*-indol-2-ylmethylene)hydrazinecarbodithioate ethanol hemisolvate

Crystal data

$C_{17}H_{15}N_3S_2 \cdot 0.5C_2H_6O$

$M_r = 348.47$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 13.4225$ (2) Å

$b = 15.4088$ (2) Å

$c = 16.8120$ (3) Å

$F_{000} = 1464$

$D_x = 1.364$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 5259 reflections

$\theta = 2.5$ – 28.3°

$\mu = 0.32$ mm⁻¹

$T = 100$ (2) K

supplementary materials

$\beta = 102.637 (1)^\circ$ Block, yellow
 $V = 3392.90 (9) \text{ \AA}^3$ $0.25 \times 0.20 \times 0.15 \text{ mm}$
 $Z = 8$

Data collection

Bruker SMART APEX CCD diffractometer 3897 independent reflections
Radiation source: fine-focus sealed tube 3332 reflections with $I > 2\sigma(I)$
Monochromator: graphite $R_{\text{int}} = 0.028$
 $T = 100(2) \text{ K}$ $\theta_{\text{max}} = 27.5^\circ$
 ω scans $\theta_{\text{min}} = 2.0^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996) $h = -16 \rightarrow 17$
 $T_{\text{min}} = 0.924$, $T_{\text{max}} = 0.954$ $k = -19 \rightarrow 20$
14302 measured reflections $l = -21 \rightarrow 17$

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
 $R[F^2 > 2\sigma(F^2)] = 0.033$ H atoms treated by a mixture of independent and constrained refinement
 $wR(F^2) = 0.088$ $w = 1/[\sigma^2(F_o^2) + (0.0411P)^2 + 3.3308P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.03$ $(\Delta/\sigma)_{\text{max}} = 0.001$
3897 reflections $\Delta\rho_{\text{max}} = 0.35 \text{ e \AA}^{-3}$
232 parameters $\Delta\rho_{\text{min}} = -0.40 \text{ e \AA}^{-3}$
5 restraints Extinction correction: none
Primary atom site location: structure-invariant direct methods

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
S1	0.58648 (3)	0.69507 (2)	0.36508 (2)	0.01972 (10)	
S2	0.56141 (3)	0.50160 (2)	0.38844 (2)	0.02263 (11)	
N1	0.48149 (10)	0.62574 (8)	0.46106 (8)	0.0201 (3)	
H1N	0.4612 (15)	0.5843 (10)	0.4893 (11)	0.036 (5)*	
N2	0.45977 (10)	0.71147 (8)	0.47237 (8)	0.0200 (3)	
N3	0.39716 (10)	0.88130 (8)	0.48940 (8)	0.0205 (3)	
H3N	0.4341 (14)	0.8771 (13)	0.4528 (10)	0.039 (6)*	
C1	0.72885 (12)	0.71544 (9)	0.27832 (9)	0.0199 (3)	
C2	0.83286 (12)	0.71386 (11)	0.31290 (10)	0.0254 (3)	
H2	0.8608	0.6671	0.3476	0.031*	
C3	0.89612 (13)	0.77961 (12)	0.29733 (10)	0.0297 (4)	
H3	0.9671	0.7776	0.3212	0.036*	

C4	0.85687 (13)	0.84829 (11)	0.24721 (10)	0.0260 (3)	
H4	0.9004	0.8937	0.2372	0.031*	
C5	0.75366 (13)	0.85010 (10)	0.21189 (10)	0.0242 (3)	
H5	0.7263	0.8968	0.1770	0.029*	
C6	0.68972 (12)	0.78405 (9)	0.22712 (10)	0.0222 (3)	
H6	0.6190	0.7858	0.2024	0.027*	
C7	0.66018 (12)	0.64525 (9)	0.29845 (10)	0.0211 (3)	
H7A	0.7011	0.5962	0.3264	0.025*	
H7B	0.6144	0.6232	0.2482	0.025*	
C8	0.53949 (11)	0.60480 (9)	0.40842 (9)	0.0180 (3)	
C9	0.40256 (11)	0.72590 (9)	0.52304 (9)	0.0203 (3)	
H9	0.3811	0.6794	0.5524	0.024*	
C10	0.37149 (11)	0.81355 (9)	0.53466 (9)	0.0197 (3)	
C11	0.31225 (12)	0.84426 (9)	0.58549 (9)	0.0208 (3)	
H11	0.2848	0.8111	0.6233	0.025*	
C12	0.29954 (11)	0.93534 (9)	0.57094 (9)	0.0192 (3)	
C13	0.35401 (11)	0.95615 (9)	0.51045 (9)	0.0197 (3)	
C14	0.35929 (12)	1.04048 (10)	0.48156 (10)	0.0246 (3)	
H14	0.3960	1.0533	0.4407	0.030*	
C15	0.30886 (13)	1.10461 (10)	0.51477 (10)	0.0269 (4)	
H15	0.3116	1.1628	0.4967	0.032*	
C16	0.25378 (13)	1.08549 (10)	0.57456 (10)	0.0259 (3)	
H16	0.2196	1.1309	0.5960	0.031*	
C17	0.24827 (12)	1.00197 (10)	0.60284 (10)	0.0229 (3)	
H17	0.2105	0.9897	0.6431	0.027*	
O1	0.48913 (18)	0.92618 (16)	0.34417 (14)	0.0289 (5)	0.50
H1O	0.5523 (10)	0.922 (3)	0.363 (3)	0.045 (13)*	0.50
C18	0.4709 (7)	1.0002 (6)	0.2875 (4)	0.0472 (9)	0.50
H18A	0.3966	1.0099	0.2687	0.057*	0.50
H18B	0.5016	1.0533	0.3157	0.057*	0.50
C19	0.5162 (7)	0.9824 (6)	0.2172 (4)	0.0472 (9)	0.50
H19A	0.5044	1.0320	0.1798	0.071*	0.50
H19B	0.4847	0.9304	0.1888	0.071*	0.50
H19C	0.5898	0.9729	0.2360	0.071*	0.50

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0245 (2)	0.01412 (17)	0.0230 (2)	0.00081 (13)	0.01047 (15)	0.00013 (13)
S2	0.0342 (2)	0.01388 (17)	0.0233 (2)	-0.00028 (14)	0.01385 (17)	-0.00209 (14)
N1	0.0271 (7)	0.0148 (6)	0.0210 (7)	0.0021 (5)	0.0108 (5)	0.0004 (5)
N2	0.0225 (6)	0.0171 (6)	0.0203 (7)	0.0026 (5)	0.0042 (5)	-0.0024 (5)
N3	0.0223 (6)	0.0210 (6)	0.0204 (7)	0.0009 (5)	0.0092 (5)	-0.0024 (5)
C1	0.0255 (8)	0.0185 (7)	0.0183 (7)	-0.0008 (6)	0.0104 (6)	-0.0024 (5)
C2	0.0261 (8)	0.0299 (8)	0.0217 (8)	0.0038 (6)	0.0082 (7)	0.0082 (6)
C3	0.0207 (8)	0.0439 (10)	0.0241 (9)	-0.0035 (7)	0.0045 (7)	0.0074 (7)
C4	0.0293 (9)	0.0299 (8)	0.0202 (8)	-0.0086 (7)	0.0087 (7)	0.0026 (6)
C5	0.0316 (9)	0.0207 (7)	0.0204 (8)	-0.0001 (6)	0.0062 (7)	0.0028 (6)

supplementary materials

C6	0.0229 (8)	0.0213 (7)	0.0222 (8)	-0.0002 (6)	0.0044 (6)	-0.0009 (6)
C7	0.0258 (8)	0.0179 (7)	0.0221 (8)	0.0001 (6)	0.0110 (6)	-0.0025 (6)
C8	0.0199 (7)	0.0170 (6)	0.0169 (7)	-0.0003 (5)	0.0031 (6)	0.0001 (5)
C9	0.0224 (7)	0.0199 (7)	0.0182 (7)	0.0022 (6)	0.0038 (6)	-0.0003 (6)
C10	0.0199 (7)	0.0209 (7)	0.0178 (7)	0.0013 (6)	0.0028 (6)	-0.0012 (6)
C11	0.0242 (8)	0.0206 (7)	0.0181 (8)	0.0021 (6)	0.0057 (6)	-0.0003 (6)
C12	0.0184 (7)	0.0217 (7)	0.0164 (7)	0.0012 (6)	0.0014 (6)	-0.0025 (6)
C13	0.0187 (7)	0.0209 (7)	0.0190 (8)	0.0007 (6)	0.0032 (6)	-0.0043 (6)
C14	0.0281 (8)	0.0227 (7)	0.0235 (8)	-0.0033 (6)	0.0066 (7)	-0.0018 (6)
C15	0.0333 (9)	0.0183 (7)	0.0270 (9)	0.0001 (6)	0.0017 (7)	-0.0020 (6)
C16	0.0265 (8)	0.0234 (7)	0.0262 (9)	0.0053 (6)	0.0020 (7)	-0.0082 (6)
C17	0.0224 (8)	0.0263 (8)	0.0203 (8)	0.0030 (6)	0.0057 (6)	-0.0046 (6)
O1	0.0238 (12)	0.0401 (13)	0.0217 (12)	-0.0033 (10)	0.0026 (10)	0.0035 (10)
C18	0.041 (2)	0.050 (3)	0.0478 (15)	0.0096 (19)	0.0033 (12)	-0.001 (2)
C19	0.041 (2)	0.050 (3)	0.0478 (15)	0.0096 (19)	0.0033 (12)	-0.001 (2)

Geometric parameters (Å, °)

S1—C8	1.7506 (15)	C9—C10	1.439 (2)
S1—C7	1.8187 (15)	C9—H9	0.9500
S2—C8	1.6647 (14)	C10—C11	1.372 (2)
N1—C8	1.3397 (19)	C11—C12	1.428 (2)
N1—N2	1.3748 (16)	C11—H11	0.9500
N1—H1N	0.875 (9)	C12—C17	1.406 (2)
N2—C9	1.285 (2)	C12—C13	1.413 (2)
N3—C13	1.3713 (18)	C13—C14	1.394 (2)
N3—C10	1.3792 (19)	C14—C15	1.383 (2)
N3—H3N	0.873 (9)	C14—H14	0.9500
C1—C2	1.390 (2)	C15—C16	1.403 (2)
C1—C6	1.392 (2)	C15—H15	0.9500
C1—C7	1.507 (2)	C16—C17	1.379 (2)
C2—C3	1.383 (2)	C16—H16	0.9500
C2—H2	0.9500	C17—H17	0.9500
C3—C4	1.383 (2)	O1—C18	1.472 (8)
C3—H3	0.9500	O1—H1O	0.839 (10)
C4—C5	1.383 (2)	C18—C19	1.469 (5)
C4—H4	0.9500	C18—H18A	0.9900
C5—C6	1.391 (2)	C18—H18B	0.9900
C5—H5	0.9500	C19—H19A	0.9800
C6—H6	0.9500	C19—H19B	0.9800
C7—H7A	0.9900	C19—H19C	0.9800
C7—H7B	0.9900		
C8—S1—C7	102.42 (7)	C11—C10—N3	109.34 (13)
C8—N1—N2	119.62 (12)	C11—C10—C9	129.24 (14)
C8—N1—H1N	118.6 (13)	N3—C10—C9	121.37 (14)
N2—N1—H1N	121.7 (13)	C10—C11—C12	107.22 (13)
C9—N2—N1	115.70 (13)	C10—C11—H11	126.4
C13—N3—C10	108.88 (12)	C12—C11—H11	126.4
C13—N3—H3N	125.6 (14)	C17—C12—C13	118.83 (14)

C10—N3—H3N	125.5 (14)	C17—C12—C11	134.62 (15)
C2—C1—C6	118.72 (14)	C13—C12—C11	106.55 (13)
C2—C1—C7	120.08 (14)	N3—C13—C14	129.59 (14)
C6—C1—C7	121.17 (14)	N3—C13—C12	108.01 (13)
C3—C2—C1	120.66 (15)	C14—C13—C12	122.40 (14)
C3—C2—H2	119.7	C15—C14—C13	117.29 (15)
C1—C2—H2	119.7	C15—C14—H14	121.4
C4—C3—C2	120.53 (15)	C13—C14—H14	121.4
C4—C3—H3	119.7	C14—C15—C16	121.36 (15)
C2—C3—H3	119.7	C14—C15—H15	119.3
C3—C4—C5	119.29 (15)	C16—C15—H15	119.3
C3—C4—H4	120.4	C17—C16—C15	121.27 (14)
C5—C4—H4	120.4	C17—C16—H16	119.4
C4—C5—C6	120.45 (15)	C15—C16—H16	119.4
C4—C5—H5	119.8	C16—C17—C12	118.83 (15)
C6—C5—H5	119.8	C16—C17—H17	120.6
C5—C6—C1	120.35 (15)	C12—C17—H17	120.6
C5—C6—H6	119.8	C18—O1—H10	108 (3)
C1—C6—H6	119.8	C19—C18—O1	109.7 (7)
C1—C7—S1	106.10 (10)	C19—C18—H18A	109.7
C1—C7—H7A	110.5	O1—C18—H18A	109.7
S1—C7—H7A	110.5	C19—C18—H18B	109.7
C1—C7—H7B	110.5	O1—C18—H18B	109.7
S1—C7—H7B	110.5	H18A—C18—H18B	108.2
H7A—C7—H7B	108.7	C18—C19—H19A	109.5
N1—C8—S2	121.15 (11)	C18—C19—H19B	109.5
N1—C8—S1	113.45 (10)	H19A—C19—H19B	109.5
S2—C8—S1	125.40 (9)	C18—C19—H19C	109.5
N2—C9—C10	119.03 (14)	H19A—C19—H19C	109.5
N2—C9—H9	120.5	H19B—C19—H19C	109.5
C10—C9—H9	120.5		
C8—N1—N2—C9	179.09 (14)	N2—C9—C10—C11	-179.23 (16)
C6—C1—C2—C3	0.6 (2)	N2—C9—C10—N3	3.6 (2)
C7—C1—C2—C3	-177.57 (15)	N3—C10—C11—C12	0.47 (17)
C1—C2—C3—C4	0.2 (3)	C9—C10—C11—C12	-176.97 (15)
C2—C3—C4—C5	-0.8 (3)	C10—C11—C12—C17	179.98 (17)
C3—C4—C5—C6	0.6 (2)	C10—C11—C12—C13	-0.39 (17)
C4—C5—C6—C1	0.2 (2)	C10—N3—C13—C14	179.53 (16)
C2—C1—C6—C5	-0.8 (2)	C10—N3—C13—C12	0.12 (17)
C7—C1—C6—C5	177.36 (14)	C17—C12—C13—N3	179.86 (13)
C2—C1—C7—S1	106.98 (14)	C11—C12—C13—N3	0.16 (16)
C6—C1—C7—S1	-71.14 (16)	C17—C12—C13—C14	0.4 (2)
C8—S1—C7—C1	-163.14 (11)	C11—C12—C13—C14	-179.30 (14)
N2—N1—C8—S2	-176.00 (11)	N3—C13—C14—C15	-179.11 (15)
N2—N1—C8—S1	3.72 (18)	C12—C13—C14—C15	0.2 (2)
C7—S1—C8—N1	179.96 (11)	C13—C14—C15—C16	-0.6 (2)
C7—S1—C8—S2	-0.33 (12)	C14—C15—C16—C17	0.3 (3)
N1—N2—C9—C10	-176.43 (13)	C15—C16—C17—C12	0.3 (2)
C13—N3—C10—C11	-0.38 (17)	C13—C12—C17—C16	-0.7 (2)

supplementary materials

C13—N3—C10—C9

177.30 (13)

C11—C12—C17—C16

178.94 (16)

Hydrogen-bond geometry (\AA , $^\circ$)

D—H \cdots *A*

D—H

H \cdots *A*

D \cdots *A*

D—H \cdots *A*

N1—H1n \cdots S2ⁱ

0.88 (1)

2.52 (1)

3.350 (1)

159 (2)

N3—H3n \cdots O1

0.87 (1)

2.25 (1)

3.047 (3)

153 (2)

Symmetry codes: (i) $-x+1, -y+1, -z+1$.

Fig. 1

