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5-Bromo-1*H*-indole-3-carbaldehyde thiosemicarbazone

Mohd. Razali Rizal, 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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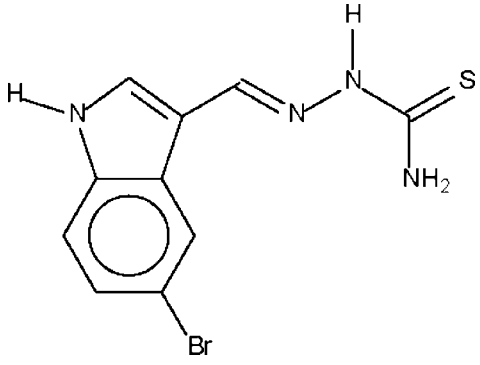
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.025; wR factor = 0.066; data-to-parameter ratio = 15.9.

In the essentially planar title molecule, $C_{10}H_9BrN_4S$, the C=N double bond is in a *trans* configuration. In the crystal structure, the S atom acts as a hydrogen-bond acceptor for the aromatic NH, aliphatic NH and terminal NH_2 groups of three symmetry-related molecules, forming a weak hydrogen-bonded layer structure.

Related literature

For a previous synthesis of the title compound, see: Dubey & Babu (2006). For related literature, see: Doyle *et al.* (1956); French & Blanz (1966); Fukukawa *et al.* (1966); Libermann *et al.* (1953); Usi (1968); Weller *et al.* (1954).



Experimental

Crystal data

$C_{10}H_9BrN_4S$
M_r = 297.18
Triclinic, $P\overline{1}$
a = 6.7731 (2) Å
b = 8.7551 (2) Å
c = 10.6539 (2) Å
α = 69.280 (1)°
β = 79.969 (1)°
γ = 72.886 (1)°
V = 563.00 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.81 mm^{-1}
T = 100 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.381, T_{max} = 0.516
(expected range = 0.344–0.467)
6176 measured reflections
2563 independent reflections
2281 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.066
S = 1.06
2563 reflections
161 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*n*···S1^{i}	0.88 (1)	2.60 (2)	3.390 (2)	150 (3)
N3—H3*n*···S1^{ii}	0.88 (1)	2.65 (1)	3.508 (2)	167 (2)
N4—H4*n*1···S1^{iii}	0.88 (1)	2.74 (1)	3.569 (2)	158 (2)

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

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: LH2609).

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supplementary materials

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5-Bromo-1*H*-indole-3-carbaldehyde thiosemicarbazone

M. R. Rizal, H. M. Ali and S. W. Ng

Comment

Indole-3-carboxaldehyde thiosemicarbazone and its substituted analogs possess useful medicinal properties; such activity has been known for a long time (Doyle *et al.*, 1956; French & Blanz, 1966; Fukukawa *et al.*, 1966; Libermann *et al.*, 1953; Usi, 1968; Weller *et al.*, 1954). The compounds, in the form of their metal derivatives, have been assesses for similar activity.

In the title compound (I) (Fig. 1), the double-bonded sulfur atom is a hydrogen-bond acceptor for the aromatic -N-H, aliphatic -N-H and terminal -NH_2 groups of three adjacent molecules, forming a weak hydrogen-bonded layer structure.

Experimental

5-Bromoindole-3-carboxaldehyde (0.3 g, 1.3 mmol) and thiosemicarbazide (0.12 g, 1.3 mmol) were heated in ethanol (50 ml) for an hour. The solvent was removed and the product and recrystallized from ethanol.

Refinement

Carbon-bound H atoms were placed in calculated positions, and were included in the refinement in the riding model approximation. The nitrogen-bound H atoms were located in a difference Fourier map, and were refined with a distance restraint of N–H 0.88±0.01 Å; their temperature factors were freely refined.

Figures

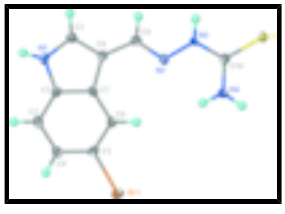

Fig. 1. The title molecule drawn using 70% probabilty ellipsoids. Hydrogen atoms are drawn as spheres of arbitrary radius.

5-Bromo-1H-indole-3-carboxaldehyde thiosemicarbazone

Crystal data

$C_{10}H_9BrN_4S$	$Z = 2$
$M_r = 297.18$	$F_{000} = 296$
Triclinic, $P\overline{1}$	$D_x = 1.753$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.7731$ (2) Å	Cell parameters from 6604 reflections

b = 8.7551 (2) Å	θ = 4.0–28.3º
c = 10.6539 (2) Å	μ = 3.81 mm^{-1}
α = 69.280 (1)º	T = 100 (2) K
β = 79.969 (1)º	Block, yellow
γ = 72.886 (1)º	0.30 × 0.20 × 0.20 mm
V = 563.00 (2) Å^3	

Data collection

Bruker SMART APEX diffractometer	2563 independent reflections
Radiation source: fine-focus sealed tube	2281 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.025
T = 100(2) K	θ_{max} = 27.5º
ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −6→8
T_{min} = 0.381, T_{max} = 0.516	k = −11→11
6176 measured reflections	l = −13→13

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.024	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.066	$w = 1/[\sigma^2(F_o^2) + (0.0383P)^2 + 0.1P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max}$ = 0.001
2563 reflections	$\Delta\rho_{max}$ = 0.36 e Å^{-3}
161 parameters	$\Delta\rho_{min}$ = −0.40 e Å^{-3}
4 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_{iso}*/U_{eq}
Br1	0.51693 (4)	0.13022 (2)	0.31237 (2)	0.02102 (9)
S1	0.01187 (9)	0.22591 (6)	1.06142 (5)	0.01534 (12)
N1	0.2717 (3)	0.8577 (2)	0.27383 (17)	0.0153 (4)
N2	0.1383 (3)	0.4698 (2)	0.68281 (16)	0.0116 (3)
N3	0.0718 (3)	0.4342 (2)	0.81750 (16)	0.0118 (3)
N4	0.1492 (3)	0.1567 (2)	0.83268 (18)	0.0169 (4)
C1	0.1846 (3)	0.8475 (3)	0.4006 (2)	0.0151 (4)
H1	0.1266	0.9415	0.4320	0.018*
C2	0.3383 (3)	0.6981 (2)	0.2626 (2)	0.0122 (4)

C3	0.4355 (3)	0.6471 (3)	0.1529 (2)	0.0142 (4)
H3	0.4641	0.7265	0.0687	0.017*
C4	0.4889 (3)	0.4765 (3)	0.1711 (2)	0.0131 (4)
H4	0.5573	0.4359	0.0990	0.016*
C5	0.4422 (3)	0.3633 (2)	0.2960 (2)	0.0127 (4)
C6	0.3464 (3)	0.4108 (2)	0.40581 (19)	0.0118 (4)
H6	0.3184	0.3300	0.4893	0.014*
C7	0.2917 (3)	0.5832 (2)	0.38933 (19)	0.0109 (4)
C8	0.1921 (3)	0.6825 (2)	0.4770 (2)	0.0120 (4)
C9	0.1249 (3)	0.6266 (2)	0.6176 (2)	0.0127 (4)
H9	0.0693	0.7074	0.6633	0.015*
C10	0.0822 (3)	0.2737 (2)	0.8937 (2)	0.0123 (4)
H1N	0.254 (5)	0.953 (2)	0.207 (2)	0.028 (7)*
H3N	0.037 (4)	0.510 (2)	0.858 (2)	0.013 (6)*
H4N1	0.137 (4)	0.0533 (17)	0.876 (2)	0.023 (7)*
H4N2	0.173 (5)	0.190 (4)	0.7452 (11)	0.035 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02971 (15)	0.01243 (12)	0.02039 (12)	−0.00512 (9)	0.00410 (9)	−0.00765 (8)
S1	0.0217 (3)	0.0111 (2)	0.0101 (2)	−0.0029 (2)	0.00152 (19)	−0.00200 (18)
N1	0.0190 (10)	0.0104 (8)	0.0119 (8)	−0.0022 (7)	0.0015 (7)	−0.0005 (6)
N2	0.0105 (8)	0.0144 (8)	0.0100 (8)	−0.0056 (6)	0.0021 (6)	−0.0034 (6)
N3	0.0154 (9)	0.0112 (8)	0.0091 (7)	−0.0032 (7)	0.0020 (6)	−0.0051 (6)
N4	0.0239 (10)	0.0117 (8)	0.0144 (8)	−0.0050 (7)	0.0044 (7)	−0.0056 (7)
C1	0.0176 (11)	0.0133 (9)	0.0127 (9)	−0.0021 (8)	0.0005 (8)	−0.0045 (8)
C2	0.0120 (10)	0.0108 (9)	0.0134 (9)	−0.0032 (7)	−0.0023 (8)	−0.0025 (7)
C3	0.0121 (10)	0.0167 (10)	0.0119 (9)	−0.0036 (8)	−0.0015 (8)	−0.0022 (7)
C4	0.0109 (10)	0.0174 (10)	0.0111 (9)	−0.0028 (8)	−0.0020 (7)	−0.0049 (7)
C5	0.0113 (10)	0.0106 (9)	0.0165 (9)	−0.0031 (7)	−0.0014 (8)	−0.0042 (7)
C6	0.0102 (10)	0.0124 (9)	0.0117 (9)	−0.0030 (7)	−0.0017 (7)	−0.0021 (7)
C7	0.0090 (10)	0.0132 (9)	0.0103 (9)	−0.0028 (7)	−0.0012 (7)	−0.0032 (7)
C8	0.0114 (10)	0.0116 (9)	0.0131 (9)	−0.0035 (8)	−0.0011 (7)	−0.0037 (7)
C9	0.0107 (10)	0.0137 (9)	0.0133 (9)	−0.0024 (8)	−0.0003 (8)	−0.0049 (7)
C10	0.0103 (10)	0.0129 (9)	0.0130 (9)	−0.0029 (7)	0.0001 (7)	−0.0038 (7)

Geometric parameters (Å, °)

Br1—C5	1.9032 (19)	C1—H1	0.9500
S1—C10	1.699 (2)	C2—C3	1.388 (3)
N1—C1	1.359 (3)	C2—C7	1.418 (3)
N1—C2	1.378 (3)	C3—C4	1.379 (3)
N1—H1N	0.878 (10)	C3—H3	0.9500
N2—C9	1.284 (3)	C4—C5	1.399 (3)
N2—N3	1.378 (2)	C4—H4	0.9500
N3—C10	1.339 (3)	C5—C6	1.372 (3)
N3—H3N	0.876 (10)	C6—C7	1.398 (3)
N4—C10	1.331 (3)	C6—H6	0.9500

N4—H4N1	0.880 (10)	C7—C8	1.447 (3)
N4—H4N2	0.873 (10)	C8—C9	1.437 (3)
C1—C8	1.376 (3)	C9—H9	0.9500
C1—N1—C2	109.25 (17)	C3—C4—H4	120.1
C1—N1—H1N	122.5 (19)	C5—C4—H4	120.1
C2—N1—H1N	126.2 (18)	C6—C5—C4	123.94 (18)
C9—N2—N3	115.12 (17)	C6—C5—Br1	118.76 (15)
C10—N3—N2	119.25 (16)	C4—C5—Br1	117.30 (15)
C10—N3—H3N	117.5 (16)	C5—C6—C7	117.04 (18)
N2—N3—H3N	122.8 (16)	C5—C6—H6	121.5
C10—N4—H4N1	119.9 (17)	C7—C6—H6	121.5
C10—N4—H4N2	118.1 (19)	C6—C7—C2	119.09 (17)
H4N1—N4—H4N2	120 (3)	C6—C7—C8	134.17 (18)
N1—C1—C8	110.69 (18)	C2—C7—C8	106.75 (17)
N1—C1—H1	124.7	C1—C8—C9	124.92 (18)
C8—C1—H1	124.7	C1—C8—C7	105.86 (17)
N1—C2—C3	129.70 (18)	C9—C8—C7	129.10 (18)
N1—C2—C7	107.45 (17)	N2—C9—C8	121.41 (18)
C3—C2—C7	122.85 (18)	N2—C9—H9	119.3
C4—C3—C2	117.27 (18)	C8—C9—H9	119.3
C4—C3—H3	121.4	N4—C10—N3	117.36 (18)
C2—C3—H3	121.4	N4—C10—S1	122.58 (16)
C3—C4—C5	119.81 (18)	N3—C10—S1	120.06 (15)
C9—N2—N3—C10	−179.30 (19)	C3—C2—C7—C6	0.3 (3)
C2—N1—C1—C8	0.5 (3)	N1—C2—C7—C8	0.4 (2)
C1—N1—C2—C3	180.0 (2)	C3—C2—C7—C8	179.9 (2)
C1—N1—C2—C7	−0.6 (2)	N1—C1—C8—C9	176.0 (2)
N1—C2—C3—C4	178.9 (2)	N1—C1—C8—C7	−0.2 (3)
C7—C2—C3—C4	−0.6 (3)	C6—C7—C8—C1	179.4 (2)
C2—C3—C4—C5	0.8 (3)	C2—C7—C8—C1	−0.2 (2)
C3—C4—C5—C6	−0.8 (3)	C6—C7—C8—C9	3.4 (4)
C3—C4—C5—Br1	179.01 (16)	C2—C7—C8—C9	−176.2 (2)
C4—C5—C6—C7	0.6 (3)	N3—N2—C9—C8	178.80 (19)
Br1—C5—C6—C7	−179.25 (15)	C1—C8—C9—N2	−179.4 (2)
C5—C6—C7—C2	−0.3 (3)	C7—C8—C9—N2	−4.0 (4)
C5—C6—C7—C8	−179.8 (2)	N2—N3—C10—N4	−2.8 (3)
N1—C2—C7—C6	−179.18 (18)	N2—N3—C10—S1	177.10 (15)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*n*···S1^{i}	0.88 (1)	2.60 (2)	3.390 (2)	150 (3)
N3—H3*n*···S1^{ii}	0.88 (1)	2.65 (1)	3.508 (2)	167 (2)
N4—H4*n*1···S1^{iii}	0.88 (1)	2.74 (1)	3.569 (2)	158 (2)

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

Fig. 1