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## Structure Reports

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

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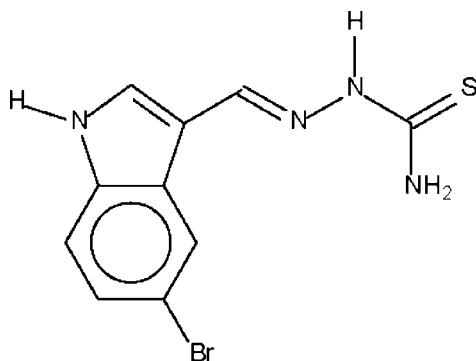
Received 27 March 2008; accepted 20 April 2008

Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  
 $R$  factor = 0.025;  $wR$  factor = 0.066; data-to-parameter ratio = 15.9.

In the essentially planar title molecule,  $\text{C}_{10}\text{H}_9\text{BrN}_4\text{S}$ , the  $\text{C}=\text{N}$  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  $\text{NH}_2$  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

$\text{C}_{10}\text{H}_9\text{BrN}_4\text{S}$   
 $M_r = 297.18$   
Triclinic,  $P\bar{1}$   
 $a = 6.7731$  (2) Å  
 $b = 8.7551$  (2) Å

$c = 10.6539$  (2) Å  
 $\alpha = 69.280$  (1)°  
 $\beta = 79.969$  (1)°  
 $\gamma = 72.886$  (1)°  
 $V = 563.00$  (2) Å<sup>3</sup>

$Z = 2$   
Mo  $K\alpha$  radiation  
 $\mu = 3.81$  mm<sup>-1</sup>

$T = 100$  (2) K  
 $0.30 \times 0.20 \times 0.20$  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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.025$

## 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<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.40$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}n\cdots\text{S1}^i$	0.88 (1)	2.60 (2)	3.390 (2)	150 (3)
$\text{N3}-\text{H3}n\cdots\text{S1}^{ii}$	0.88 (1)	2.65 (1)	3.508 (2)	167 (2)
$\text{N4}-\text{H4}n1\cdots\text{S1}^{iii}$	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 assessed 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<sub>2</sub> 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 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

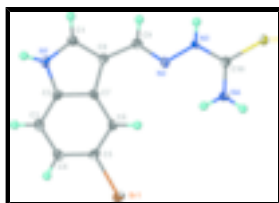


Fig. 1. The title molecule drawn using 70% probability ellipsoids. Hydrogen atoms are drawn as spheres of arbitrary radius.

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

### Crystal data

C<sub>10</sub>H<sub>9</sub>BrN<sub>4</sub>S

*M<sub>r</sub>* = 297.18

Triclinic, *P* $\bar{1}$

Hall symbol: -P 1

*a* = 6.7731 (2) Å

*Z* = 2

*F*<sub>000</sub> = 296

*D<sub>x</sub>* = 1.753 Mg m<sup>-3</sup>

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 6604 reflections

# supplementary materials

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$b = 8.7551$  (2) Å  
 $c = 10.6539$  (2) Å  
 $\alpha = 69.280$  (1)°  
 $\beta = 79.969$  (1)°  
 $\gamma = 72.886$  (1)°  
 $V = 563.00$  (2) Å<sup>3</sup>

$\theta = 4.0$ – $28.3$ °  
 $\mu = 3.81$  mm<sup>-1</sup>  
 $T = 100$  (2) K  
Block, yellow  
 $0.30 \times 0.20 \times 0.20$  mm

## Data collection

Bruker SMART APEX  
diffractometer  
Radiation source: fine-focus sealed tube  
Monochromator: graphite  
 $T = 100$ (2) K  
 $\omega$  scans  
Absorption correction: multi-scan  
(SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.381$ ,  $T_{\max} = 0.516$   
6176 measured reflections

2563 independent reflections  
2281 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.025$   
 $\theta_{\text{max}} = 27.5$ °  
 $\theta_{\text{min}} = 2.1$ °  
 $h = -6 \rightarrow 8$   
 $k = -11 \rightarrow 11$   
 $l = -13 \rightarrow 13$

## Refinement

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.024$   
 $wR(F^2) = 0.066$   
 $S = 1.06$   
2563 reflections  
161 parameters  
4 restraints  
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map  
Hydrogen site location: inferred from neighbouring sites  
H atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0383P)^2 + 0.1P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.36$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.40$  e Å<sup>-3</sup>  
Extinction correction: none

## Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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 ( $\text{\AA}$ ,  $^\circ$ )

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

## supplementary materials

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 ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 <i>n</i> ⋯S1 <sup>i</sup>	0.88 (1)	2.60 (2)	3.390 (2)	150 (3)
N3—H3 <i>n</i> ⋯S1 <sup>ii</sup>	0.88 (1)	2.65 (1)	3.508 (2)	167 (2)
N4—H4 <i>n</i> 1⋯S1 <sup>iii</sup>	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

