

# 1*H*-Indole-3-carbaldehyde thiosemi-carbazone

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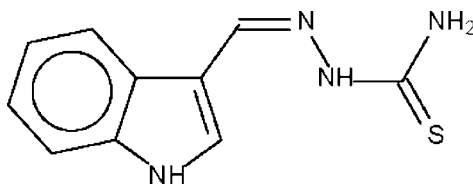
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 Key indicators: single-crystal X-ray study;  $T = 123$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  
 $R$  factor = 0.045;  $wR$  factor = 0.161; data-to-parameter ratio = 14.9.

The molecules of the title compound,  $\text{C}_{10}\text{H}_{10}\text{N}_4\text{S}$ , are linked by  $\text{N}-\text{H}_{\text{indole}} \cdots \text{S}$  hydrogen bonds to form a linear hydrogen-bonded chain. There are two independent molecules in the asymmetric unit.

## Related literature

For the synthesis and bacteriostatic activity of indole-3-carbaldehyde semithiocarbazone, see: Doyle *et al.* (1956); Fujikawa *et al.* (1966); Libermann *et al.* (1953); Weller *et al.* (1954). For metal complexes of the compound, see: Bhardwaj & Singh (1994); Dalvi *et al.* (2004); Garg & Tandon (1988); Kanoongo *et al.* (1988, 1990); Kiran *et al.* (1986); Kumari *et al.* (1992*a,b*; 1993*a,b*); Rodriguez-Argueelles *et al.* (2005); Saxena & Singh (1994); Saxena *et al.* (1993, 1994); Singh & Singh (1990); Singh *et al.* (1987, 1988); Varshney & Tandon (1989); Varshney *et al.* (1989, 1996).



## Experimental

### Crystal data

$\text{C}_{10}\text{H}_{10}\text{N}_4\text{S}$   
 $M_r = 218.28$   
 Triclinic,  $P\bar{1}$   
 $a = 7.1893$  (1) Å  
 $b = 11.1654$  (2) Å  
 $c = 13.5373$  (3) Å  
 $\alpha = 68.887$  (1)°  
 $\beta = 85.048$  (1)°

$\gamma = 82.467$  (1)°  
 $V = 1004.07$  (3) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.29$  mm<sup>-1</sup>  
 $T = 123$  (2) K  
 $0.44 \times 0.24 \times 0.04$  mm

### Data collection

Bruker SMART APEX  
 diffractometer

Absorption correction: multi-scan  
 (SADABS; Sheldrick, 1996)  
 $T_{\text{min}} = 0.857$ ,  $T_{\text{max}} = 0.988$

9295 measured reflections  
 4527 independent reflections

3142 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.036$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$   
 $wR(F^2) = 0.161$   
 $S = 1.10$   
 4527 reflections  
 303 parameters  
 8 restraints

H atoms treated by a mixture of  
 independent and constrained  
 refinement  
 $\Delta\rho_{\text{max}} = 0.45$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.45$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{N4}-\text{H4n} \cdots \text{S1}^i$	0.89 (1)	2.56 (2)	3.383 (3)	156 (3)
$\text{N8}-\text{H8n} \cdots \text{S2}^i$	0.88 (3)	2.49 (2)	3.325 (2)	157 (3)

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

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); 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).

The authors thank the University of Canterbury, New Zealand, for the diffraction measurements, and the Science Fund (12-02-03-2031) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2196).

## References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.  
 Bhardwaj, N. C. & Singh, R. V. (1994). *Proc. Ind. Acad. Sci. Chem. Sci.* **106**, 15–22.  
 Bruker (2005). APEX2 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Dalvi, K., Pal, M., Garje, S. & Shivram, S. (2004). *Indian J. Chem.* **43A**, 1667–1671.  
 Doyle, F. P., Ferrier, W., Holland, D. O., Mehta, M. D. & Nayler, J. H. C. (1956). *J. Chem. Soc.* pp. 2853–2857.  
 Fujikawa, F., Yamashita, I., Seno, T., Sasaki, M., Naito, M. & Tsukuma, S. (1966). *Yakugaku Zasshi*, **86**, 801–814.  
 Garg, A. & Tandon, J. P. (1988). *Syn. React. Inorg. Met.-Org. Chem.* **18**, 705–715.  
 Kanoongo, N., Singh, R. V. & Tandon, J. P. (1988). *J. Prakt. Chem. (Leipzig)*, **330**, 479–483.  
 Kanoongo, N., Singh, R. V. & Tandon, J. P. (1990). *J. Prakt. Chem. (Leipzig)*, **332**, 815–819.  
 Kiran, R. V., Singh, J. P. & Tandon, J. P. (1986). *Syn. React. Inorg. Met.-Org. Chem.* **16**, 1341–1350.  
 Kumari, A., Sharma, N., Singh, R. V. & Tandon, J. P. (1992*a*). *Phosphorus, Sulfur Silicon Rel. Elem.* **71**, 225–229.  
 Kumari, A., Singh, D., Singh, R. V. & Tandon, J. P. (1993*a*). *Syn. React. Inorg. Met.-Org. Chem.* **23**, 575–587.  
 Kumari, A., Singh, D., Singh, R. V. & Tandon, J. P. (1993*b*). *Phosphorus, Sulfur Silicon Rel. Elem.* **80**, 117–125.  
 Kumari, A., Singh, R. V. & Tandon, J. P. (1992*b*). *Phosphorus, Sulfur Silicon Rel. Elem.* **66**, 195–200.  
 Libermann, D., Moyeux, M., Rouaix, A., Maillard, J., Hengl, L. & Himbert, J. (1953). *Bull. Soc. Chim. Fr.* pp. 957–962.  
 Rodriguez-Argueelles, M. C., Lopez-Silva, E., Sanmartin, J., Pelagatti, P. & Zani, F. (2005). *J. Inorg. Biochem.* **99**, 2231–2239.

- Saxena, C., Bhardwaj, N. C., Singh, D. & Singh, R. (1993). *Syn. React. Inorg. Met.-Org. Chem.* **23**, 1391–1405.
- Saxena, C. & Singh, R. V. (1994). *Appl. Organomet. Chem.* **9**, 267–276.
- Saxena, C., Singh, R. V. & Singh, S. (1994). *Syn. React. Inorg. Met.-Org. Chem.* **24**, 1311–1124.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Singh, D. & Singh, R. V. (1990). *Main Group Met. Chem.* **13**, 19–27.
- Singh, K., Singh, R. V. & Tandon, J. P. (1987). *Syn. React. Inorg. Met.-Org. Chem.* **17**, 385–398.
- Singh, K., Singh, R. V. & Tandon, J. P. (1988). *Polyhedron*, **7**, 151–154.
- Varshney, S., Singh, H. & Varshney, A. K. (1996). *ACGC Chem. Res. Commun.* **4**, 26–29.
- Varshney, A. K. & Tandon, J. P. (1989). *Egypt. J. Chem.* **32**, 109–114.
- Varshney, A. K., Verma, P. S. & Varshney, S. (1989). *Main Group Met. Chem.* **12**, 249–257.
- Weller, L. E., Sell, H. M. & Gottshall, R. Y. (1954). *J. Am. Chem. Soc.* **76**, 1959–???
- Westrip, S. P. (2008). *publCIF*. In preparation.

**supplementary materials**

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## 1*H*-Indole-3-carbaldehyde thiosemicarbazone

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### Comment

The bacteriostatic activity of indole-3-carboxaldehyde thiosemicarbazone is known for a long time (Doyle *et al.*, 1956; Fujikawa *et al.*, 1966; Libermann *et al.*, 1953; Weller *et al.*, 1954). The compound yields complexes with main group as well as transition metal ions.

### Experimental

Thiosemicarbazide (0.3 g, 3.3 mmol) and indole-3-carboxaldehyde (0.5 g, 3.3 mmol) were refluxed in ethanol (50 ml) for 2 h. The solvent was removed to give the product Schiff base, and crystals were obtained upon recrystallization from ethanol.

### Refinement

The carbon-bound H atoms were placed at calculated positions (C–H 0.95 Å), and were included in the refinement in the riding model approximation with  $U(\text{H})$  set to  $1.2U_{\text{eq}}(\text{C})$ . The amino H atoms were located in a difference Fourier map, and were refined with a distance restraint of N–H  $0.88 \pm 0.01$  Å.

### Figures

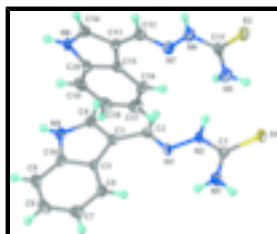


Fig. 1. Thermal ellipsoid plot of the two independent molecules of the title compound. Displacement ellipsoids are drawn at the 70% probability level, and H atoms are shown as spheres of arbitrary radii.

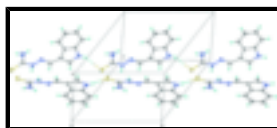


Fig. 2. Chain structure of the title compound. Intermolecular H bonds are shown as dashed lines.

## 1*H*-Indole-3-carbaldehyde thiosemicarbazone

### Crystal data

$\text{C}_{10}\text{H}_{10}\text{N}_4\text{S}$

$M_r = 218.28$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$Z = 4$

$F_{000} = 456$

$D_x = 1.444 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ \AA}$

# supplementary materials

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$a = 7.1893$  (1) Å  
 $b = 11.1654$  (2) Å  
 $c = 13.5373$  (3) Å  
 $\alpha = 68.887$  (1)°  
 $\beta = 85.048$  (1)°  
 $\gamma = 82.467$  (1)°  
 $V = 1004.07$  (3) Å<sup>3</sup>

Cell parameters from 4065 reflections

$\theta = 3.6$ – $30.8$ °  
 $\mu = 0.29$  mm<sup>-1</sup>  
 $T = 123$  (2) K  
Wedge, colorless  
 $0.44 \times 0.24 \times 0.04$  mm

## Data collection

Bruker APEXII diffractometer	4527 independent reflections
Radiation source: medium-focus sealed tube	3142 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.036$
$T = 123$ (2) K	$\theta_{\text{max}} = 27.5$ °
$\varphi$ and $\omega$ scans	$\theta_{\text{min}} = 1.6$ °
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -8 \rightarrow 9$
$T_{\text{min}} = 0.857$ , $T_{\text{max}} = 0.988$	$k = -14 \rightarrow 14$
9295 measured reflections	$l = -17 \rightarrow 17$

## Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.161$	$w = 1/[\sigma^2(F_o^2) + (0.0864P)^2 + 0.0582P]$
$S = 1.10$	where $P = (F_o^2 + 2F_c^2)/3$
4527 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
303 parameters	$\Delta\rho_{\text{max}} = 0.45$ e Å <sup>-3</sup>
8 restraints	$\Delta\rho_{\text{min}} = -0.45$ e Å <sup>-3</sup>
Primary atom site location: structure-invariant direct methods	Extinction correction: none

## Special details

**Experimental.** A medium-focus collimator of 0.8 mm diameter was used on the diffractometer to measure the somewhat large crystal.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.61344 (11)	0.11515 (7)	0.35357 (6)	0.0218 (2)
S2	0.81997 (11)	0.34262 (7)	0.04922 (6)	0.0250 (2)
N1	0.7013 (4)	0.1337 (3)	0.5349 (2)	0.0272 (6)
H1N1	0.721 (5)	0.173 (3)	0.577 (2)	0.044 (11)*

H1N2	0.680 (6)	0.0521 (16)	0.568 (3)	0.053 (13)*
N2	0.6604 (3)	0.3220 (2)	0.39282 (18)	0.0179 (5)
H2N	0.642 (5)	0.354 (3)	0.3237 (10)	0.042 (11)*
N3	0.7031 (3)	0.3921 (2)	0.45314 (18)	0.0171 (5)
N4	0.7758 (4)	0.7985 (2)	0.45494 (19)	0.0208 (5)
H4N	0.762 (5)	0.8832 (11)	0.440 (3)	0.040 (11)*
N5	0.5325 (4)	0.4371 (3)	0.1469 (2)	0.0239 (6)
H5N1	0.450 (4)	0.502 (2)	0.147 (3)	0.037 (10)*
H5N2	0.487 (5)	0.365 (2)	0.154 (3)	0.037 (10)*
N6	0.7531 (4)	0.5739 (2)	0.0624 (2)	0.0223 (5)
H6N	0.855 (3)	0.590 (4)	0.021 (3)	0.047 (11)*
N7	0.6520 (3)	0.6664 (2)	0.09750 (19)	0.0199 (5)
N8	0.6103 (3)	1.0744 (2)	0.11636 (19)	0.0210 (5)
H8N	0.640 (5)	1.1508 (18)	0.111 (3)	0.037 (10)*
C1	0.6607 (4)	0.1932 (3)	0.4337 (2)	0.0173 (6)
C2	0.6936 (4)	0.5146 (3)	0.4011 (2)	0.0175 (6)
H2	0.6579	0.5459	0.3296	0.021*
C3	0.7347 (4)	0.6064 (3)	0.4466 (2)	0.0161 (6)
C4	0.7238 (4)	0.7375 (3)	0.3922 (2)	0.0196 (6)
H4	0.6857	0.7794	0.3213	0.024*
C5	0.7953 (4)	0.5851 (3)	0.5511 (2)	0.0148 (5)
C6	0.8287 (4)	0.4758 (3)	0.6422 (2)	0.0188 (6)
H6	0.8136	0.3924	0.6426	0.023*
C7	0.8843 (4)	0.4918 (3)	0.7319 (2)	0.0230 (6)
H7	0.9067	0.4184	0.7944	0.028*
C8	0.9078 (4)	0.6146 (3)	0.7319 (2)	0.0237 (7)
H8	0.9464	0.6227	0.7943	0.028*
C9	0.8759 (4)	0.7244 (3)	0.6426 (2)	0.0211 (6)
H9	0.8925	0.8074	0.6426	0.025*
C10	0.8183 (4)	0.7082 (3)	0.5529 (2)	0.0174 (6)
C11	0.6925 (4)	0.4572 (3)	0.0883 (2)	0.0192 (6)
C12	0.7333 (4)	0.7692 (3)	0.0773 (2)	0.0189 (6)
H12	0.8548	0.7738	0.0434	0.023*
C13	0.6459 (4)	0.8771 (3)	0.1045 (2)	0.0164 (6)
C14	0.7289 (4)	0.9880 (3)	0.0867 (2)	0.0191 (6)
H14	0.8527	1.0017	0.0576	0.023*
C15	0.4594 (4)	0.8983 (3)	0.1488 (2)	0.0156 (6)
C16	0.3047 (4)	0.8256 (3)	0.1817 (2)	0.0210 (6)
H16	0.3122	0.7412	0.1790	0.025*
C17	0.1416 (4)	0.8801 (3)	0.2180 (2)	0.0247 (7)
H17	0.0372	0.8312	0.2414	0.030*
C18	0.1256 (4)	1.0053 (3)	0.2214 (2)	0.0245 (7)
H18	0.0106	1.0403	0.2453	0.029*
C19	0.2771 (4)	1.0783 (3)	0.1898 (2)	0.0231 (6)
H19	0.2682	1.1629	0.1922	0.028*
C20	0.4422 (4)	1.0232 (3)	0.1547 (2)	0.0185 (6)

## supplementary materials

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### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0312 (4)	0.0162 (4)	0.0216 (4)	-0.0052 (3)	-0.0014 (3)	-0.0101 (3)
S2	0.0274 (4)	0.0203 (4)	0.0321 (4)	-0.0073 (3)	0.0061 (3)	-0.0150 (3)
N1	0.0433 (17)	0.0196 (14)	0.0201 (13)	-0.0059 (13)	-0.0052 (12)	-0.0070 (11)
N2	0.0242 (13)	0.0160 (12)	0.0171 (12)	-0.0055 (10)	-0.0017 (10)	-0.0089 (10)
N3	0.0168 (12)	0.0196 (13)	0.0199 (12)	-0.0058 (10)	0.0008 (9)	-0.0120 (10)
N4	0.0269 (13)	0.0135 (13)	0.0233 (13)	-0.0045 (11)	-0.0012 (10)	-0.0072 (10)
N5	0.0218 (13)	0.0207 (14)	0.0303 (14)	-0.0038 (11)	0.0041 (11)	-0.0108 (12)
N6	0.0231 (13)	0.0180 (13)	0.0280 (14)	-0.0031 (11)	0.0032 (11)	-0.0114 (11)
N7	0.0221 (12)	0.0150 (12)	0.0232 (12)	0.0008 (10)	-0.0018 (10)	-0.0081 (10)
N8	0.0269 (14)	0.0162 (13)	0.0244 (13)	-0.0094 (11)	0.0015 (10)	-0.0105 (10)
C1	0.0165 (13)	0.0175 (14)	0.0188 (14)	-0.0034 (11)	0.0013 (11)	-0.0074 (11)
C2	0.0157 (13)	0.0213 (15)	0.0173 (13)	-0.0058 (12)	0.0007 (10)	-0.0079 (11)
C3	0.0144 (13)	0.0178 (14)	0.0177 (13)	-0.0029 (11)	0.0015 (10)	-0.0081 (11)
C4	0.0223 (15)	0.0182 (15)	0.0188 (14)	-0.0035 (12)	-0.0005 (11)	-0.0069 (12)
C5	0.0110 (12)	0.0163 (14)	0.0194 (13)	-0.0042 (11)	0.0029 (10)	-0.0089 (11)
C6	0.0176 (14)	0.0181 (15)	0.0220 (14)	-0.0036 (12)	-0.0004 (11)	-0.0080 (12)
C7	0.0224 (15)	0.0254 (16)	0.0189 (14)	-0.0020 (13)	-0.0019 (12)	-0.0051 (12)
C8	0.0193 (14)	0.0349 (18)	0.0221 (15)	-0.0027 (13)	-0.0025 (12)	-0.0159 (13)
C9	0.0171 (14)	0.0239 (16)	0.0279 (15)	-0.0020 (12)	-0.0003 (12)	-0.0163 (13)
C10	0.0138 (13)	0.0175 (15)	0.0225 (14)	-0.0024 (11)	0.0023 (11)	-0.0096 (12)
C11	0.0210 (15)	0.0170 (15)	0.0215 (14)	-0.0031 (12)	-0.0030 (11)	-0.0083 (12)
C12	0.0204 (14)	0.0175 (15)	0.0176 (13)	-0.0009 (12)	-0.0011 (11)	-0.0053 (11)
C13	0.0206 (14)	0.0125 (14)	0.0152 (13)	-0.0041 (11)	-0.0022 (11)	-0.0025 (11)
C14	0.0199 (14)	0.0208 (15)	0.0177 (13)	-0.0047 (12)	-0.0003 (11)	-0.0072 (12)
C15	0.0175 (13)	0.0149 (14)	0.0133 (13)	-0.0013 (11)	-0.0039 (10)	-0.0030 (11)
C16	0.0237 (15)	0.0165 (15)	0.0216 (14)	-0.0060 (12)	-0.0035 (12)	-0.0032 (12)
C17	0.0172 (14)	0.0294 (17)	0.0258 (15)	-0.0066 (13)	-0.0009 (12)	-0.0065 (13)
C18	0.0201 (15)	0.0312 (18)	0.0223 (15)	0.0027 (13)	-0.0005 (12)	-0.0114 (13)
C19	0.0299 (16)	0.0206 (16)	0.0207 (14)	0.0018 (13)	-0.0057 (12)	-0.0101 (12)
C20	0.0237 (15)	0.0180 (14)	0.0161 (13)	-0.0040 (12)	-0.0050 (11)	-0.0072 (11)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

S1—C1	1.696 (3)	C4—H4	0.9500
S2—C11	1.689 (3)	C5—C6	1.399 (4)
N1—C1	1.329 (4)	C5—C10	1.415 (4)
N1—H1N1	0.87 (3)	C6—C7	1.388 (4)
N1—H1N2	0.89 (3)	C6—H6	0.9500
N2—C1	1.341 (4)	C7—C8	1.403 (4)
N2—N3	1.393 (3)	C7—H7	0.9500
N2—H2N	0.887 (10)	C8—C9	1.386 (4)
N3—C2	1.289 (4)	C8—H8	0.9500
N4—C4	1.365 (3)	C9—C10	1.395 (4)
N4—C10	1.376 (4)	C9—H9	0.9500
N4—H4N	0.886 (10)	C12—C13	1.436 (4)

N5—C11	1.335 (4)	C12—H12	0.9500
N5—H5N1	0.88 (3)	C13—C14	1.379 (4)
N5—H5N2	0.88 (3)	C13—C15	1.446 (4)
N6—C11	1.344 (4)	C14—H14	0.9500
N6—N7	1.385 (3)	C15—C16	1.409 (4)
N6—H6N	0.88 (3)	C15—C20	1.414 (4)
N7—C12	1.287 (4)	C16—C17	1.384 (4)
N8—C14	1.350 (4)	C16—H16	0.9500
N8—C20	1.384 (4)	C17—C18	1.405 (4)
N8—H8N	0.88 (3)	C17—H17	0.9500
C2—C3	1.443 (4)	C18—C19	1.391 (4)
C2—H2	0.9500	C18—H18	0.9500
C3—C4	1.376 (4)	C19—C20	1.391 (4)
C3—C5	1.443 (4)	C19—H19	0.9500
C1—N1—H1N1	124 (3)	C8—C7—H7	119.5
C1—N1—H1N2	120 (3)	C9—C8—C7	121.4 (3)
H1N1—N1—H1N2	114 (4)	C9—C8—H8	119.3
C1—N2—N3	121.7 (2)	C7—C8—H8	119.3
C1—N2—H2N	113 (2)	C8—C9—C10	117.4 (3)
N3—N2—H2N	125 (2)	C8—C9—H9	121.3
C2—N3—N2	113.1 (2)	C10—C9—H9	121.3
C4—N4—C10	109.1 (2)	N4—C10—C9	129.9 (3)
C4—N4—H4N	125 (2)	N4—C10—C5	108.0 (2)
C10—N4—H4N	125 (2)	C9—C10—C5	122.1 (3)
C11—N5—H5N1	121 (2)	N5—C11—N6	117.1 (3)
C11—N5—H5N2	115 (2)	N5—C11—S2	123.0 (2)
H5N1—N5—H5N2	116 (3)	N6—C11—S2	119.9 (2)
C11—N6—N7	120.0 (2)	N7—C12—C13	122.0 (3)
C11—N6—H6N	118 (3)	N7—C12—H12	119.0
N7—N6—H6N	122 (3)	C13—C12—H12	119.0
C12—N7—N6	114.4 (2)	C14—C13—C12	124.2 (3)
C14—N8—C20	109.3 (2)	C14—C13—C15	105.9 (2)
C14—N8—H8N	123 (2)	C12—C13—C15	129.9 (2)
C20—N8—H8N	127 (2)	N8—C14—C13	110.7 (3)
N1—C1—N2	117.8 (2)	N8—C14—H14	124.6
N1—C1—S1	123.6 (2)	C13—C14—H14	124.6
N2—C1—S1	118.6 (2)	C16—C15—C20	118.8 (3)
N3—C2—C3	123.1 (3)	C16—C15—C13	134.5 (3)
N3—C2—H2	118.5	C20—C15—C13	106.7 (2)
C3—C2—H2	118.5	C17—C16—C15	118.4 (3)
C4—C3—C2	123.6 (3)	C17—C16—H16	120.8
C4—C3—C5	106.6 (2)	C15—C16—H16	120.8
C2—C3—C5	129.8 (3)	C16—C17—C18	122.0 (3)
N4—C4—C3	110.1 (3)	C16—C17—H17	119.0
N4—C4—H4	125.0	C18—C17—H17	119.0
C3—C4—H4	125.0	C19—C18—C17	120.4 (3)
C6—C5—C10	119.4 (2)	C19—C18—H18	119.8
C6—C5—C3	134.3 (2)	C17—C18—H18	119.8
C10—C5—C3	106.3 (2)	C20—C19—C18	117.7 (3)



## supplementary materials

C7—C6—C5	118.6 (3)	C20—C19—H19	121.2
C7—C6—H6	120.7	C18—C19—H19	121.2
C5—C6—H6	120.7	N8—C20—C19	129.9 (3)
C6—C7—C8	121.1 (3)	N8—C20—C15	107.4 (2)
C6—C7—H7	119.5	C19—C20—C15	122.6 (3)
C1—N2—N3—C2	-179.2 (2)	C3—C5—C10—C9	179.8 (2)
C11—N6—N7—C12	173.1 (3)	N7—N6—C11—N5	0.9 (4)
N3—N2—C1—N1	0.7 (4)	N7—N6—C11—S2	-177.8 (2)
N3—N2—C1—S1	-178.20 (19)	N6—N7—C12—C13	177.7 (2)
N2—N3—C2—C3	-179.0 (2)	N7—C12—C13—C14	178.5 (3)
N3—C2—C3—C4	-179.5 (3)	N7—C12—C13—C15	-4.9 (5)
N3—C2—C3—C5	1.5 (5)	C20—N8—C14—C13	-0.3 (3)
C10—N4—C4—C3	-1.2 (3)	C12—C13—C14—N8	177.4 (2)
C2—C3—C4—N4	-178.3 (3)	C15—C13—C14—N8	0.1 (3)
C5—C3—C4—N4	0.9 (3)	C14—C13—C15—C16	178.4 (3)
C4—C3—C5—C6	178.6 (3)	C12—C13—C15—C16	1.3 (5)
C2—C3—C5—C6	-2.3 (5)	C14—C13—C15—C20	0.1 (3)
C4—C3—C5—C10	-0.2 (3)	C12—C13—C15—C20	-177.0 (3)
C2—C3—C5—C10	178.9 (3)	C20—C15—C16—C17	0.3 (4)
C10—C5—C6—C7	-0.2 (4)	C13—C15—C16—C17	-177.8 (3)
C3—C5—C6—C7	-178.8 (3)	C15—C16—C17—C18	1.0 (4)
C5—C6—C7—C8	-0.3 (4)	C16—C17—C18—C19	-1.4 (4)
C6—C7—C8—C9	0.3 (4)	C17—C18—C19—C20	0.4 (4)
C7—C8—C9—C10	0.4 (4)	C14—N8—C20—C19	-177.0 (3)
C4—N4—C10—C9	-179.3 (3)	C14—N8—C20—C15	0.4 (3)
C4—N4—C10—C5	1.0 (3)	C18—C19—C20—N8	178.0 (3)
C8—C9—C10—N4	179.5 (3)	C18—C19—C20—C15	0.9 (4)
C8—C9—C10—C5	-0.9 (4)	C16—C15—C20—N8	-178.9 (2)
C6—C5—C10—N4	-179.5 (2)	C13—C15—C20—N8	-0.3 (3)
C3—C5—C10—N4	-0.5 (3)	C16—C15—C20—C19	-1.3 (4)
C6—C5—C10—C9	0.8 (4)	C13—C15—C20—C19	177.4 (3)

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N4—H4n $\cdots$ S1 <sup>i</sup>	0.89 (1)	2.56 (2)	3.383 (3)	156 (3)
N8—H8n $\cdots$ S2 <sup>i</sup>	0.88 (3)	2.49 (2)	3.325 (2)	157 (3)

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

Fig. 1

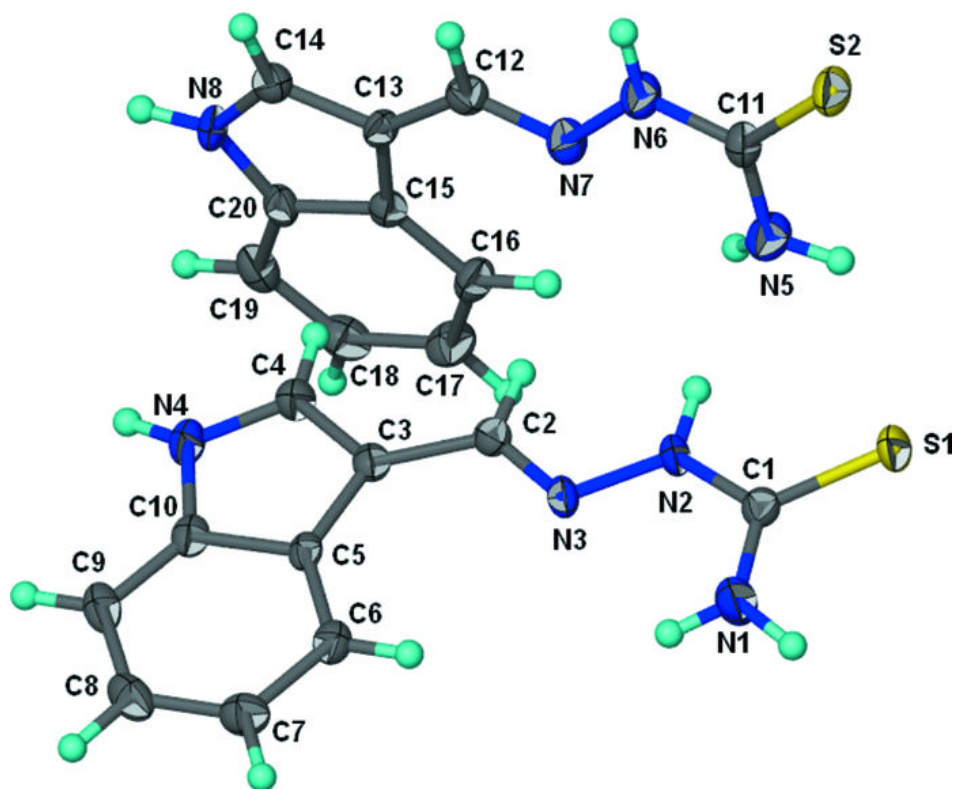


Fig. 2

