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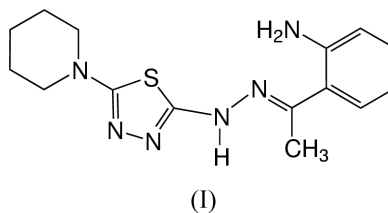
Key indicators

Single-crystal X-ray study
T = 291 K
Mean $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$
R factor = 0.047
wR factor = 0.090
Data-to-parameter ratio = 13.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

2-[[1-(2-Aminophenyl)ethylidene]hydrazino]-5-piperidyl-1,3,4-thiadiazole

The molecules of the title compound, $\text{C}_{15}\text{H}_{20}\text{N}_6\text{S}$, are linked into centrosymmetric dimers by $\text{N}-\text{H}\cdots\text{N}$ intermolecular hydrogen bonds, forming a monoclinic crystal structure.Received 10 May 2002
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Comment

The expected product of condensation between 2-aminoacetophenone and 3-piperidylthiosemicarbazide is the 2-aminoacetophenone *N*(4)-piperidylthiosemicarbazone (which was not obtained despite repeated attempts), but because a second molecule of 3-piperidylthiosemicarbazide reacted with the newly formed thiosemicarbazone, the title compound, (I), was obtained. The reaction takes place at the thiosemicarbazone thione C atom by the nucleophilic attack of the hydrazine N atom of 3-piperidylthiosemicarbazide, resulting in the formation of (I), as well as hydrogen sulfide and piperidine.Atoms S1 and N1 are in a *syn* conformation with respect to the C8—N2 bond. The two C—S bonds are single bonds, while the C8—N3 and N4—C9 bonds are essentially equal in length and can be considered double bonds.One of the amino group H atoms forms an intramolecular $\text{N6}-\text{H6A}\cdots\text{N2}$ hydrogen bond, but the expected hydrogen bond involving H6B is not observed. The molecules form dimers through self-complementary $\text{N2}-\text{H2A}\cdots\text{N3}$ intermolecular hydrogen bonds.

Experimental

3-Piperidylthiosemicarbazide was prepared according to the method of Scovill (1991). The thiosemicarbazide was condensed with 2-aminoacetophenone (Aldrich) as reported previously (West *et al.*, 1998*a,b*) to produce (I). Crystals were obtained by slow evaporation of a dilute ethanol solution.

Crystal data

 $\text{C}_{15}\text{H}_{20}\text{N}_6\text{S}$
 $M_r = 316.43$
Monoclinic, $P2_1/c$
 $a = 11.291 (1) \text{ \AA}$
 $b = 13.644 (2) \text{ \AA}$
 $c = 11.384 (2) \text{ \AA}$
 $\beta = 115.94 (1)^\circ$
 $V = 1577.1 (4) \text{ \AA}^3$
 $Z = 4$ $D_x = 1.333 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 37 reflections
 $\theta = 4.7-9.4^\circ$
 $\mu = 0.21 \text{ mm}^{-1}$
 $T = 291 (2) \text{ K}$
Prism, dark red
 $0.40 \times 0.12 \times 0.12 \text{ mm}$

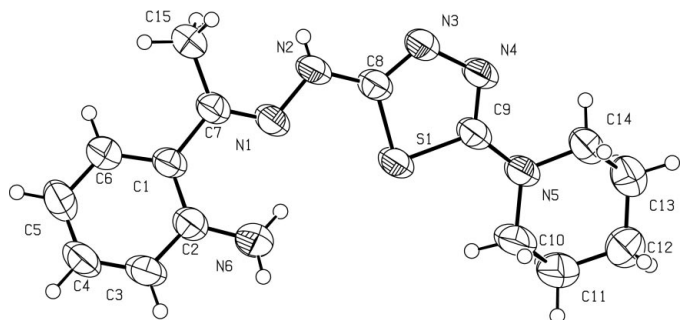


Figure 1
A view of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

Data collection

Siemens P4/PC diffractometer
 ω scans
Absorption correction: none
2925 measured reflections
2777 independent reflections
1302 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.044$

$\theta_{\text{max}} = 25.0^\circ$
 $h = 0 \rightarrow 13$
 $k = 0 \rightarrow 16$
 $l = -13 \rightarrow 12$
3 standard reflections
every 97 reflections
intensity decay: <2%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.090$
 $S = 0.88$
2777 reflections
208 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0186P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.012$
 $\Delta\rho_{\text{max}} = 0.16 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.19 \text{ e } \text{\AA}^{-3}$

Table 1
Selected geometric parameters (\AA , $^\circ$).

S1—C8	1.736 (3)	N3—N4	1.398 (4)
S1—C9	1.757 (3)	N4—C9	1.292 (4)
N1—C7	1.292 (4)	N5—C9	1.350 (4)
N1—N2	1.387 (3)	N5—C14	1.460 (4)
N2—C8	1.347 (4)	N5—C10	1.460 (4)
N3—C8	1.297 (4)	N6—C2	1.388 (5)
C8—S1—C9	86.22 (15)	N6—C2—C1	122.6 (3)
C7—N1—N2	118.4 (3)	N1—C7—C1	117.9 (3)
C8—N2—N1	116.3 (3)	N3—C8—N2	125.1 (3)
C8—N3—N4	112.8 (3)	N3—C8—S1	114.5 (2)
C9—N4—N3	112.1 (3)	N2—C8—S1	120.5 (2)
C9—N5—C14	117.9 (3)	N4—C9—N5	123.8 (3)
C9—N5—C10	120.4 (3)	N4—C9—S1	114.4 (3)
C14—N5—C10	117.0 (3)	N5—C9—S1	121.7 (2)
C2—C1—C7	124.1 (3)		

Table 2
Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N2—H2A \cdots N3 ⁱ	0.76 (3)	2.28 (3)	2.995 (3)	156 (4)
N6—H6A \cdots N1	0.95 (4)	1.98 (4)	2.698 (4)	131 (3)
N6—H6A \cdots S1	0.95 (4)	2.91 (4)	3.806 (3)	159 (3)

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

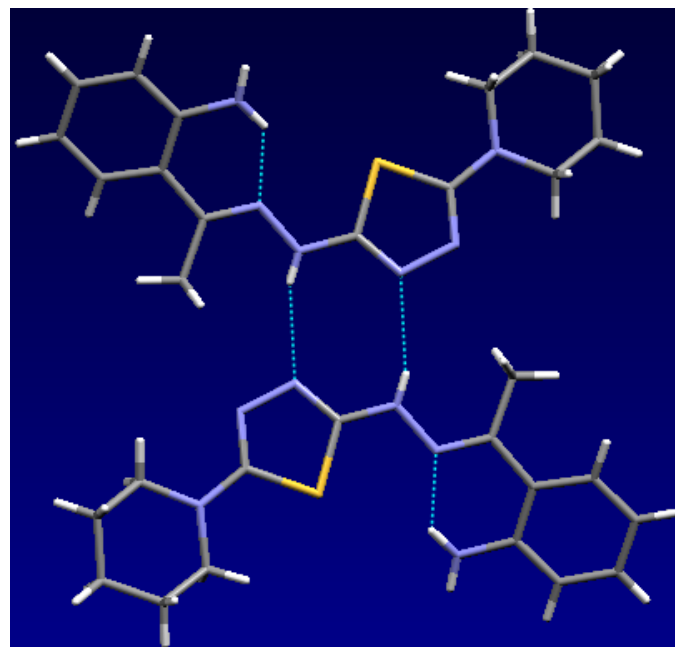


Figure 2
The hydrogen-bonded molecular dimers in (I).

H atoms attached to N atoms were located in a difference Fourier map and refined isotropically. H atoms attached to C atoms were positioned geometrically ($C-H = 0.93-0.97 \text{ \AA}$) and refined using a riding model.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1999) and *Mercury Visualizer Program* (Cambridge Crystallographic Data Centre, 2002); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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