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4-Methyl-1-(3-pyridylmethylidene)thiosemicarbazide

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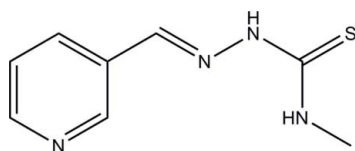
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.043; wR factor = 0.118; data-to-parameter ratio = 13.2.

All the non-H atoms of the title compound, $\text{C}_8\text{H}_{10}\text{N}_4\text{S}$, lie on a crystallographic mirror plane and an intramolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bond helps to stabilize the molecular conformation. In the crystal, molecules are linked through intermolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds, forming zigzag $C(7)$ chains along the a axis.

Related literature

For background to Schiff bases derived from thiosemicarbazone and its derivatives, see: Casas *et al.* (2001); Beraldo *et al.* (2001); Jouad *et al.* (2002); Swearingen *et al.* (2002). For bond-length data, see: Allen *et al.* (1987). For similar structures, see: Selvanayagam *et al.* (2002); Karakurt *et al.* (2003); Bernhardt *et al.* (2003); Sampath *et al.* (2003).



Experimental

Crystal data

$\text{C}_8\text{H}_{10}\text{N}_4\text{S}$
 $M_r = 194.26$
 Monoclinic, $P2_1/m$
 $a = 7.276$ (3) Å
 $b = 6.581$ (2) Å
 $c = 10.297$ (3) Å
 $\beta = 92.997$ (2)°

$V = 492.4$ (3) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.29$ mm⁻¹
 $T = 298$ K
 $0.17 \times 0.15 \times 0.15$ mm

Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 2004)
 $T_{\min} = 0.953$, $T_{\max} = 0.958$

3208 measured reflections
 1106 independent reflections
 640 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.041$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.118$
 $S = 1.02$
 1106 reflections
 84 parameters
 2 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.14$ e Å⁻³
 $\Delta\rho_{\min} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N4}-\text{H4}\cdots\text{N2}$ | 0.90 (2) | 2.14 (3) | 2.585 (4) | 109 (2) |
| $\text{N3}-\text{H3}\cdots\text{N1}^1$ | 0.90 (1) | 2.09 (1) | 2.989 (3) | 176 (3) |

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

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5755).

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supporting information

Acta Cryst. (2010). E66, o3324 [https://doi.org/10.1107/S1600536810048853]

4-Methyl-1-(3-pyridylmethylidene)thiosemicarbazide

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S1. Comment

Thiosemicarbazone and its derivatives are important materials for the preparation of Schiff bases (Casas *et al.*, 2001; Beraldo *et al.*, 2001; Jouad *et al.*, 2002; Swearingen *et al.*, 2002). In this paper, the title new Schiff base compound derived from the condensation of 3-formylpyridine with 4-methylthiosemicarbazone is reported.

The molecule of the title compound, Fig. 1, possess a crystallographic mirror plane symmetry. The bond lengths have normal values (Allen *et al.*, 1987), and are comparable to those observed in similar compounds (Selvanayagam *et al.*, 2002; Karakurt *et al.*, 2003; Bernhardt *et al.*, 2003; Sampath *et al.*, 2003).

In the crystal, molecules are linked through intermolecular N—H···N hydrogen bonds (Table 1), to form zigzag chains along the *a* axis (Fig. 2).

S2. Experimental

The title compound was prepared by the Schiff base condensation of equimolar quantities of 3-formylpyridine (0.107 g, 1 mmol) with 4-methylthiosemicarbazone (0.105 g, 1 mmol) in methanol. The excess methanol was removed by distillation. Colourless blocks were obtained by slow evaporation of an ethanol solution of the product in air.

S3. Refinement

The amino H atoms were located in a difference map and refined with N—H distance restrained to 0.90 (1) Å. The remaining H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{C8})$.

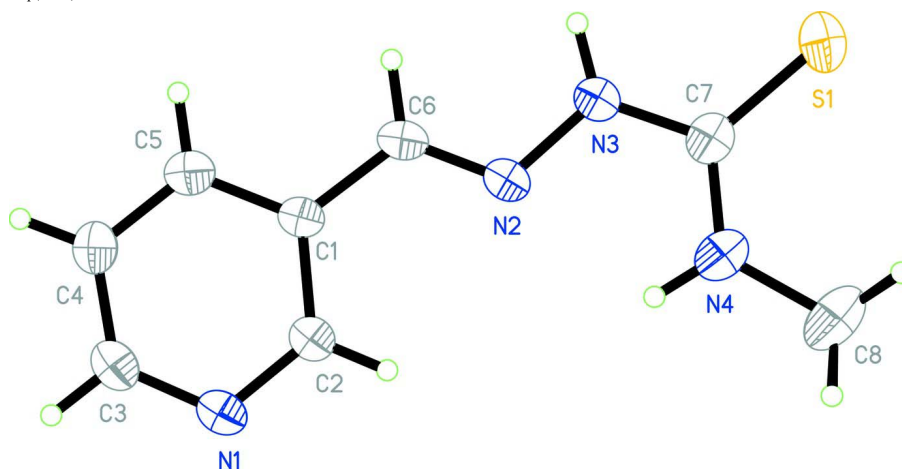


Figure 1

The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

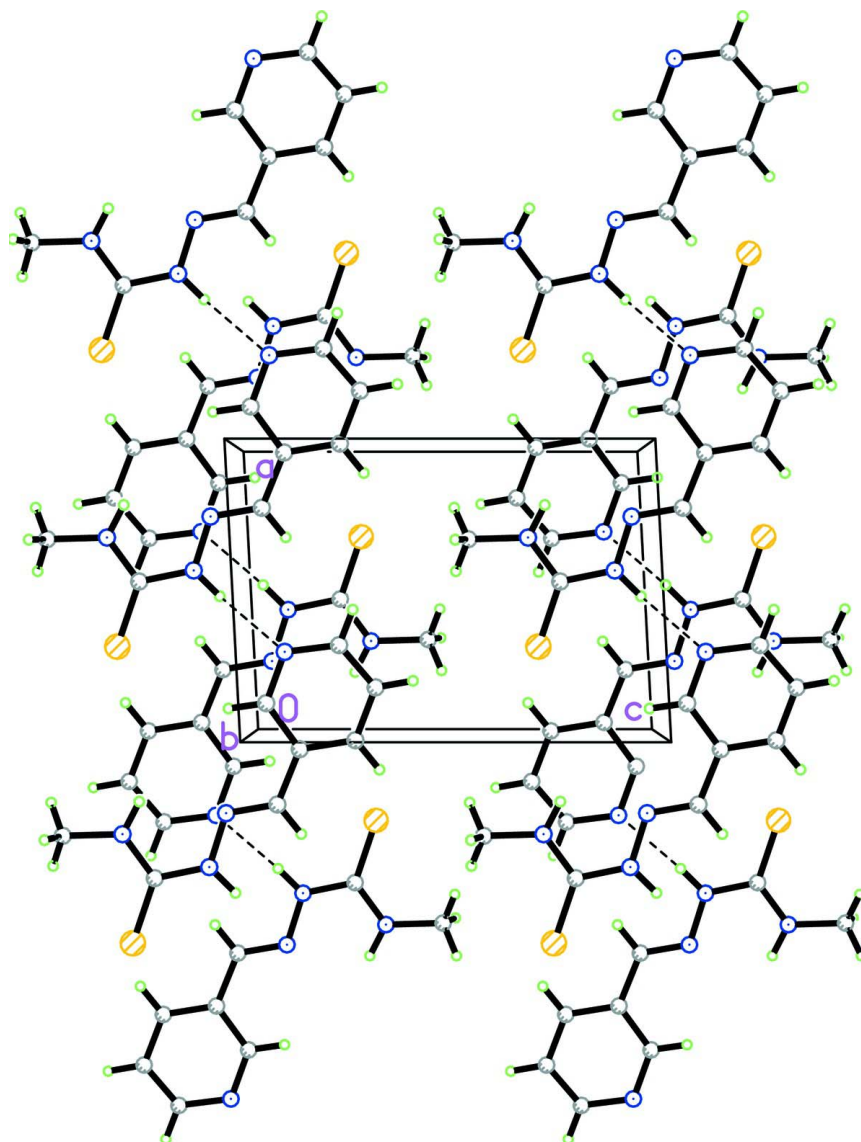


Figure 2

The crystal packing of the title compound, viewed along the *b* axis.

4-Methyl-1-(3-pyridylmethylidene)thiosemicarbazide

Crystal data

$C_8H_{10}N_4S$

$M_r = 194.26$

Monoclinic, $P2_1/m$

Hall symbol: $-P\ 2_1y$

$a = 7.276\ (3)\ \text{\AA}$

$b = 6.581\ (2)\ \text{\AA}$

$c = 10.297\ (3)\ \text{\AA}$

$\beta = 92.997\ (2)^\circ$

$V = 492.4\ (3)\ \text{\AA}^3$

$Z = 2$

$F(000) = 204$

$D_x = 1.310\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 669 reflections

$\theta = 2.7\text{--}24.5^\circ$

$\mu = 0.29\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Block, colourless

$0.17 \times 0.15 \times 0.15\ \text{mm}$

Data collection

| | |
|---|--|
| Bruker APEXII CCD diffractometer | 3208 measured reflections |
| Radiation source: fine-focus sealed tube | 1106 independent reflections |
| Graphite monochromator | 640 reflections with $I > 2\sigma(I)$ |
| ω scans | $R_{\text{int}} = 0.041$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 2004) | $\theta_{\text{max}} = 26.5^\circ$, $\theta_{\text{min}} = 2.8^\circ$ |
| $T_{\text{min}} = 0.953$, $T_{\text{max}} = 0.958$ | $h = -9 \rightarrow 9$ |
| | $k = -8 \rightarrow 8$ |
| | $l = -12 \rightarrow 10$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.043$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.118$ | $w = 1/[\sigma^2(F_o^2) + (0.0547P)^2]$ |
| $S = 1.02$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 1106 reflections | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 84 parameters | $\Delta\rho_{\text{max}} = 0.14 \text{ e } \text{\AA}^{-3}$ |
| 2 restraints | $\Delta\rho_{\text{min}} = -0.20 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | |

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|--------------|--------|-------------|----------------------------------|-----------|
| S1 | 0.68926 (12) | 0.2500 | 0.29184 (9) | 0.0835 (4) | |
| N1 | -0.2929 (3) | 0.2500 | -0.1051 (2) | 0.0558 (7) | |
| N2 | 0.2508 (3) | 0.2500 | 0.0542 (2) | 0.0507 (6) | |
| N3 | 0.4324 (3) | 0.2500 | 0.1009 (2) | 0.0566 (7) | |
| N4 | 0.3217 (4) | 0.2500 | 0.3028 (3) | 0.0699 (8) | |
| C1 | 0.0350 (3) | 0.2500 | -0.1287 (3) | 0.0496 (7) | |
| C2 | -0.1201 (3) | 0.2500 | -0.0547 (3) | 0.0510 (8) | |
| H2 | -0.1020 | 0.2500 | 0.0354 | 0.061* | |
| C3 | -0.3159 (4) | 0.2500 | -0.2351 (3) | 0.0616 (9) | |
| H3A | -0.4353 | 0.2500 | -0.2720 | 0.074* | |
| C4 | -0.1733 (4) | 0.2500 | -0.3164 (3) | 0.0697 (10) | |
| H4A | -0.1954 | 0.2500 | -0.4061 | 0.084* | |
| C5 | 0.0049 (4) | 0.2500 | -0.2620 (3) | 0.0673 (10) | |
| H5 | 0.1041 | 0.2500 | -0.3154 | 0.081* | |
| C6 | 0.2213 (4) | 0.2500 | -0.0687 (3) | 0.0581 (8) | |

| | | | | | |
|-----|------------|--------|------------|-------------|------|
| H6 | 0.3209 | 0.2500 | -0.1217 | 0.070* | |
| C7 | 0.4698 (4) | 0.2500 | 0.2318 (3) | 0.0554 (8) | |
| C8 | 0.3272 (5) | 0.2500 | 0.4451 (3) | 0.0994 (13) | |
| H8A | 0.3310 | 0.1125 | 0.4762 | 0.149* | 0.50 |
| H8B | 0.2193 | 0.3162 | 0.4743 | 0.149* | 0.50 |
| H8C | 0.4349 | 0.3213 | 0.4781 | 0.149* | 0.50 |
| H3 | 0.519 (3) | 0.2500 | 0.042 (2) | 0.080* | |
| H4 | 0.212 (2) | 0.2500 | 0.259 (3) | 0.080* | |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-------------|-------------|-------------|----------|--------------|----------|
| S1 | 0.0681 (6) | 0.1189 (9) | 0.0617 (6) | 0.000 | -0.0150 (4) | 0.000 |
| N1 | 0.0392 (13) | 0.0640 (17) | 0.0642 (17) | 0.000 | 0.0028 (12) | 0.000 |
| N2 | 0.0365 (12) | 0.0615 (16) | 0.0542 (15) | 0.000 | 0.0019 (11) | 0.000 |
| N3 | 0.0416 (13) | 0.0775 (18) | 0.0508 (16) | 0.000 | 0.0024 (11) | 0.000 |
| N4 | 0.0738 (18) | 0.085 (2) | 0.0518 (16) | 0.000 | 0.0136 (14) | 0.000 |
| C1 | 0.0369 (14) | 0.0618 (19) | 0.0503 (17) | 0.000 | 0.0058 (12) | 0.000 |
| C2 | 0.0425 (15) | 0.0593 (19) | 0.0512 (18) | 0.000 | 0.0011 (13) | 0.000 |
| C3 | 0.0450 (16) | 0.074 (2) | 0.064 (2) | 0.000 | -0.0062 (15) | 0.000 |
| C4 | 0.0587 (19) | 0.102 (3) | 0.0478 (19) | 0.000 | -0.0054 (16) | 0.000 |
| C5 | 0.0505 (18) | 0.097 (3) | 0.055 (2) | 0.000 | 0.0091 (15) | 0.000 |
| C6 | 0.0389 (15) | 0.078 (2) | 0.0579 (19) | 0.000 | 0.0108 (13) | 0.000 |
| C7 | 0.0624 (19) | 0.0541 (19) | 0.0499 (18) | 0.000 | 0.0033 (15) | 0.000 |
| C8 | 0.126 (3) | 0.121 (4) | 0.053 (2) | 0.000 | 0.023 (2) | 0.000 |

Geometric parameters (Å, °)

| | | | |
|----------|------------|-----------|-----------|
| S1—C7 | 1.682 (3) | C1—C6 | 1.460 (4) |
| N1—C2 | 1.335 (3) | C2—H2 | 0.9300 |
| N1—C3 | 1.340 (3) | C3—C4 | 1.367 (4) |
| N2—C6 | 1.273 (4) | C3—H3A | 0.9300 |
| N2—N3 | 1.382 (3) | C4—C5 | 1.385 (4) |
| N3—C7 | 1.361 (4) | C4—H4A | 0.9300 |
| N3—H3 | 0.899 (10) | C5—H5 | 0.9300 |
| N4—C7 | 1.334 (4) | C6—H6 | 0.9300 |
| N4—C8 | 1.463 (4) | C8—H8A | 0.9600 |
| N4—H4 | 0.898 (10) | C8—H8B | 0.9600 |
| C1—C5 | 1.379 (4) | C8—H8C | 0.9600 |
| C1—C2 | 1.394 (4) | | |
| C2—N1—C3 | 117.0 (2) | C3—C4—H4A | 120.8 |
| C6—N2—N3 | 117.1 (2) | C5—C4—H4A | 120.8 |
| C7—N3—N2 | 118.9 (2) | C1—C5—C4 | 119.9 (3) |
| C7—N3—H3 | 124 (2) | C1—C5—H5 | 120.0 |
| N2—N3—H3 | 117 (2) | C4—C5—H5 | 120.0 |
| C7—N4—C8 | 124.7 (3) | N2—C6—C1 | 121.7 (3) |
| C7—N4—H4 | 117 (2) | N2—C6—H6 | 119.2 |

| | | | |
|-----------|-----------|------------|-----------|
| C8—N4—H4 | 119 (2) | C1—C6—H6 | 119.2 |
| C5—C1—C2 | 117.0 (2) | N4—C7—N3 | 114.7 (3) |
| C5—C1—C6 | 121.1 (3) | N4—C7—S1 | 125.2 (2) |
| C2—C1—C6 | 121.9 (3) | N3—C7—S1 | 120.1 (2) |
| N1—C2—C1 | 124.1 (2) | N4—C8—H8A | 109.5 |
| N1—C2—H2 | 118.0 | N4—C8—H8B | 109.5 |
| C1—C2—H2 | 118.0 | H8A—C8—H8B | 109.5 |
| N1—C3—C4 | 123.6 (3) | N4—C8—H8C | 109.5 |
| N1—C3—H3A | 118.2 | H8A—C8—H8C | 109.5 |
| C4—C3—H3A | 118.2 | H8B—C8—H8C | 109.5 |
| C3—C4—C5 | 118.5 (3) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|--------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N4—H4 \cdots N2 | 0.90 (2) | 2.14 (3) | 2.585 (4) | 109 (2) |
| N3—H3 \cdots N1 ⁱ | 0.90 (1) | 2.09 (1) | 2.989 (3) | 176 (3) |

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