

N-Hexanoyl-*N'*-(6-methyl-2-pyridyl)thioureaM. Sukeri M. Yusof,^{a*}
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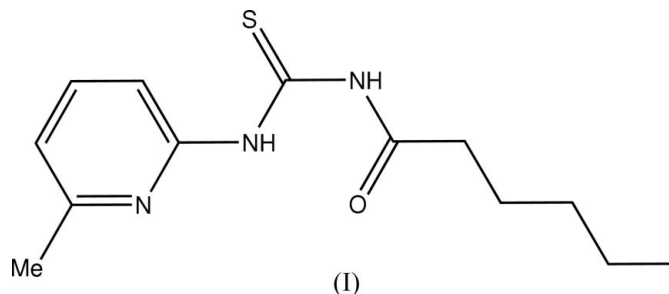
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ (C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.136
Data-to-parameter ratio = 17.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The asymmetric unit of the title compound, C₁₃H₁₉N₃OS, consists of two independent molecules. Both molecules adopt a *trans*–*cis* configuration with respect to the positions of the hexanoyl and 6-methyl-2-pyridyl groups relative to the S atom across their C–N bonds. In the crystal structure, the molecules are linked by intermolecular N–H···S and C–H···O hydrogen bonds to form a molecular tape running along the [310] direction.

Comment

Most benzoylthiourea derivatives have *trans*–*cis* configurations with respect to the position of the benzoyl and the other *N*-terminal substituent group relative to the thiono S atom across their C–N bonds (Yamin & Yusof, 2003). Only a few derivatives, such as 1-benzoyl-3-(6-methyl-2-pyridyl)thiourea, (II) (Yusof *et al.*, 2006), prefer *cis*–*trans* configurations. The title compound, (I), is similar to (II) except that the benzoyl group is replaced by hexanoyl (Fig. 1).



The asymmetric unit consists of two independent molecules. In contrast to (II), the molecules adopt the common *trans*–*cis* configuration. The bond lengths and angles in both molecules are comparable to those in (II) and in normal ranges (Allen *et al.*, 1987). The two independent molecules are essentially planar with maximum deviations of 0.171 (1) Å for atom S1 and 0.173 (2) Å for atom C23 from their least-squares planes. There are two intramolecular hydrogen interactions in each molecule (Table 1). In the crystal structure, the molecules are linked by N–H···S and C–H···O intermolecular hydrogen bonds (Table 1) into a molecular tape running along the [310] direction (Fig. 2).

Experimental

An equimolar amount of 2-amino-6-methylpyridine (1.61 g, 14 mmol) in acetone 20 ml was added dropwise to a stirred acetone solution (75 ml) containing hexanoyl chloride (2.0 g, 14 mmol) and ammonium thiocyanate (1.06 g, 14 mmol). The mixture was refluxed

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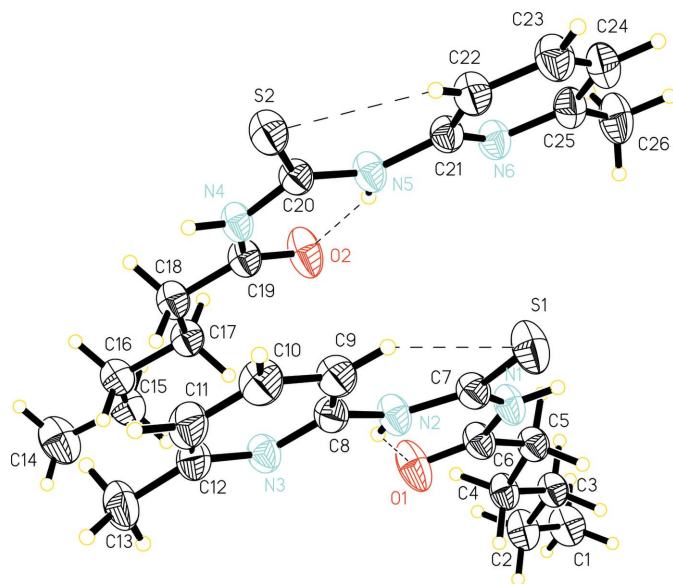


Figure 1
The asymmetric unit of (I), with 50% probability displacement ellipsoids. The dashed lines indicate N—H...O and C—H...S hydrogen bonds.

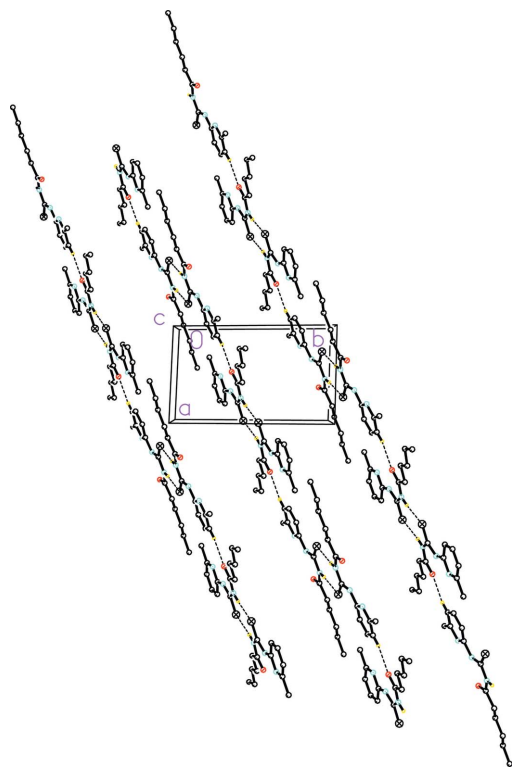


Figure 2
A packing diagram of (I), viewed down the *c* axis. The dashed lines denote N—H...S and C—H...O hydrogen bonds. H atoms not involved in these interactions have been omitted.

for 1 h. The resulting solution was poured into a beaker containing some ice blocks. The white precipitate was filtered off, washed with distilled water and cold ethanol, and dried under vacuum (yield 78%, 2.72 g). Single crystals were obtained by recrystallization from methanol.

Crystal data

$C_{13}H_{19}N_3OS$
 $M_r = 265.37$
Triclinic, $P\bar{1}$
 $a = 8.112$ (4) Å
 $b = 13.631$ (7) Å
 $c = 14.267$ (7) Å
 $\alpha = 72.840$ (8)°
 $\beta = 73.600$ (8)°
 $\gamma = 87.145$ (9)°

$V = 1445.0$ (13) Å³
 $Z = 4$
 $D_x = 1.220$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.22$ mm⁻¹
 $T = 298$ (2) K
Block, colourless
0.43 × 0.37 × 0.36 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.912$, $T_{max} = 0.926$

14881 measured reflections
5632 independent reflections
4013 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.025$
 $\theta_{max} = 26.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.136$
 $S = 1.02$
5632 reflections
329 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0781P)^2 + 0.0881P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.28$ e Å⁻³
 $\Delta\rho_{min} = -0.15$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N2—H2...O1	0.86	1.88	2.616 (3)	143
N5—H5...O2	0.86	1.85	2.596 (2)	144
C9—H9...S1	0.93	2.56	3.200 (3)	126
C22—H22...S2	0.93	2.60	3.229 (3)	126
N1—H1...S1 ⁱ	0.86	2.55	3.402 (2)	169
N4—H4...S2 ⁱⁱ	0.86	2.68	3.518 (2)	166
C11—H11...O2 ⁱⁱⁱ	0.93	2.41	3.230 (3)	147

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

All H atoms were located in a difference map, and then placed in geometrically idealized positions and allowed to ride on the parent C or N atoms with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$ (for CH, CH₂ and NH) or $1.5U_{eq}(C)$ (for CH₃).

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON (Spek, 2003).

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References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2000). SMART (Version 5.630) and SAINT (Version 6.36a). Bruker AXS Inc., Madison, Wisconsin, USA.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.

Sheldrick, G. M. (1997a). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Sheldrick, G. M. (1997b). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Yamin, B. M. & Yusof, M. S. M. (2003). *Acta Cryst.* **E59**, o151–o152.

Yusof, M. S. M., Soh, S. K. C., Ngah, N. & Yamin, B. M. (2006). *Acta Cryst.* **E62**, o1446–o1448.