

M. Sukeri M. Yusof,^a
S. Kamilah C. Soh,^a Nurziana
Ngah^b and Bohari M. Yamin^{b*}^aDepartment of Chemical Sciences, Faculty of
Science and Technology, Kolej Universiti Sains
dan Teknologi Malaysia, Mengabang Telipot,
21030 Kuala Terengganu, Malaysia, and^bSchool of Chemical Sciences and Food
Technology, Universiti Kebangsaan Malaysia,
43600 Bangi, Selangor, MalaysiaCorrespondence e-mail:
bohari@pkrisc.cc.ukm.my

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ (C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.123
Data-to-parameter ratio = 14.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

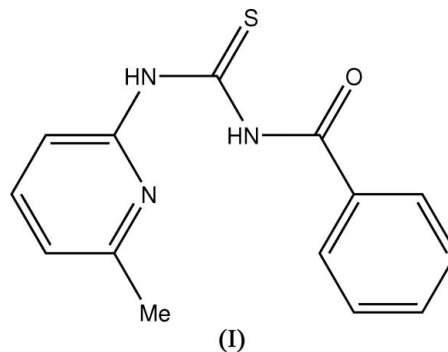
1-Benzoyl-3-(6-methylpyridin-2-yl)thiourea

The molecular structure of the title compound, C₁₄H₁₃N₃OS, adopts a *cis-trans* configuration with respect to the positions of the benzoyl and 6-methylpyridin-2-yl groups relative to the S atom across the thiourea C–N bonds. In the crystal structure, the molecules are linked by N–H···S, C–H···S and C–H···O interactions, forming a one-dimensional chain parallel to the *b* axis.

Received 3 March 2006
Accepted 13 March 2006

Comment

In most benzoylthiourea derivatives, such as *N*-benzoyl-*N'*-phenylthiourea, (II) (Yamin & Yusof, 2003*a*), and *N*-benzoyl-*N'*-4-bromophenylthiourea, (III) (Yamin & Yusof, 2003*b*), the benzoyl group prefers a *trans* configuration with respect to the S atom. The title compound, (I), is an example of a *cis* configuration being displayed by the benzoyl group.



The molecular structure of (I) adopts a *cis-trans* configuration with respect to the positions of the benzoyl and 6-methylpyridin-2-yl groups relative to the S atom across the C8–N1 and C8–N2 bonds, respectively (Fig. 1). The bond lengths and angles (Table 1) are in normal ranges (Allen *et al.*, 1987) and in agreement with the corresponding ones in (II) and (III). The central carbonylthiourea group (S1/C8/N1/N2/C7), phenyl (C1–C6) and 6-methylpyridin-2-yl (N3/C9–C14) fragments are each planar, with a maximum deviation of 0.024 (2) Å for atom N1.

The central carbonylthiourea group makes dihedral angles with the phenyl and 6-methylpyridin-2-yl fragments of 12.92 (12) and 3.53 (10)°, respectively. The phenyl ring is inclined to the 6-methylpyridin-2-yl fragment by 11.77 (12)°; this is smaller than the corresponding value [33.3 (1)°] in (II), indicating a more nearly planar behaviour adopted by the molecule.

There is an intramolecular hydrogen bond, N1–H1···N3 (Table 2) and, as a result, a pseudo-six-membered ring (N1–H1···N3–C9–N2–C8) is formed (Fig. 1). In the crystal

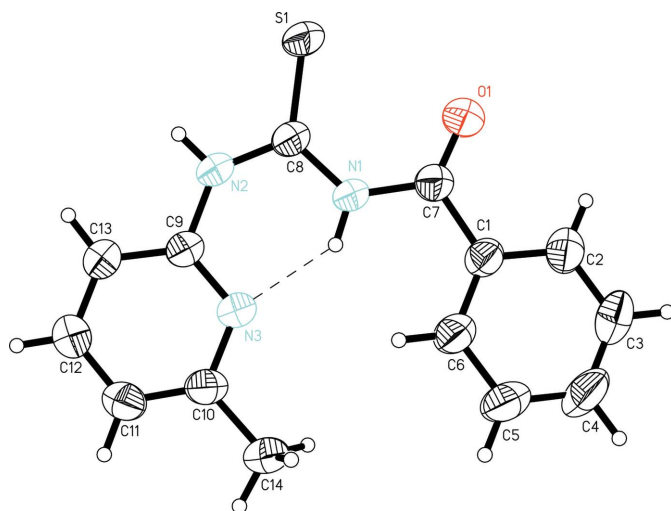


Figure 1

The molecular structure with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular hydrogen bond is shown as a dashed line.

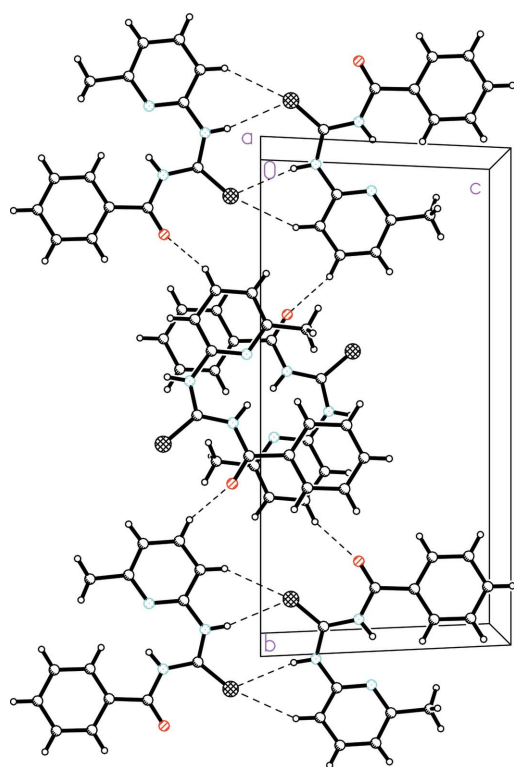


Figure 2

A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

structure, the molecules are linked by hydrogen bonds (Fig.2), forming a one-dimensional chain parallel to the *b* axis.

Experimental

A solution of benzoylchloride (2.0 g, 14 mmol) and ammonium thiocyanate (1.06 g, 14 mmol) in acetone (15 ml) was added dropwise to an acetone solution (15 ml) containing an equimolar amount of 2-

amino-6-methylpyridine (1.51 g, 14 mmol). The solution was refluxed for 1 h and then poured into a beaker containing ice. The resulting white precipitate was filtered and washed with cold ethanol–distilled water (2:1 v/v) before being dried under vacuum (yield 2.85 g, 75%, m.p. 404.85–406.1 K). Recrystallization from chloroform yielded single crystals suitable for X-ray analysis.

Crystal data

$C_{14}H_{13}N_3OS$
 $M_r = 271.33$
 Monoclinic, $P2_1/c$
 $a = 7.2687$ (14) Å
 $b = 18.938$ (4) Å
 $c = 10.1216$ (19) Å
 $\beta = 109.756$ (4)°
 $V = 1311.3$ (4) Å³
 $Z = 4$

$D_x = 1.374$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2710 reflections
 $\theta = 2.1$ – 26.0°
 $\mu = 0.24$ mm⁻¹
 $T = 298$ (2) K
 Block, colorless
 $0.40 \times 0.30 \times 0.23$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2000)
 $T_{\min} = 0.910$, $T_{\max} = 0.947$
 7196 measured reflections

2560 independent reflections
 2000 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = -8 \rightarrow 8$
 $k = -17 \rightarrow 23$
 $l = -12 \rightarrow 11$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.123$
 $S = 1.03$
 2560 reflections
 173 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0591P)^2 + 0.4366P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.31$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.26$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

S1–C8	1.665 (2)	N1–C7	1.393 (2)
N1–C8	1.366 (2)	N2–C8	1.355 (3)
O1–C7–N1	122.83 (18)	N2–C8–S1	119.12 (14)
N2–C8–N1	114.92 (16)	N1–C8–S1	125.95 (15)

Table 2

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>
N1–H1...N3	0.86	1.95	2.676 (2)	141
N2–H2...S1 ⁱ	0.86	2.64	3.4831 (18)	167
C12–H12...O1 ⁱⁱ	0.93	2.51	3.364 (3)	153
C13–H13...S1 ⁱ	0.93	2.79	3.627 (2)	150

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$.

H atoms were positioned geometrically, with N–H = 0.86 Å, and C–H = 0.93 and 0.96 Å for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C}, \text{N})$, where $x = 1.5$ for methyl and $x = 1.2$ for all other H atoms.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; 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).

The authors thank the Malaysian Government, Universiti Kebangsaan Malaysia and Kolej Universiti Sains dan Teknologi Malaysia for research grants IRPA No 09-02-02-993 and KUSTEM fundamental research grant No 54221.

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). *SADABS* (Version 2.01), *SMART* (Version 5.630) and *SAINTE* (Version 6.36a). Bruker AXS Inc., Madison, Wisconsin, USA.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Sheldrick, G. M. (1997a). *SHELXS97* and *SHELXL97*, University of Göttingen, Germany.
- Sheldrick, G. M. (1997b). *SHELXTL*. Version 5.1. Bruker AXS, Inc., Madison, Wisconsin, USA.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Yamin, B. M. & Yusof, M. S. M (2003a). *Acta Cryst.* **E59**, o151–o152.
- Yamin, B. M. & Yusof, M. S. M (2003b). *Acta Cryst.* **E59**, o340–o341.