

1-(2,2-Dimethylpropionyl)-3-phenylthiourea

M. Sukeri M. Yusof,^{a*}
Nurul 'Izzah A. Ramadan^a 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:
mohdsukeri@kustem.edu.my

Key indicators

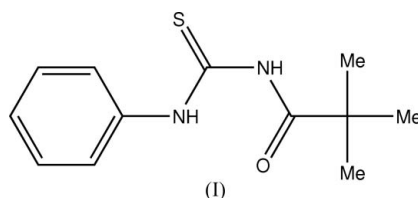
Single-crystal X-ray study
 $T = 273\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.047
 wR factor = 0.118
Data-to-parameter ratio = 18.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_{12}\text{H}_{16}\text{N}_2\text{OS}$, the molecules adopt a *trans-cis* configuration of the 2,2-dimethylpropionyl and phenyl groups relative to the S atom across the C—N bonds. The molecules are linked by $\text{N}-\text{H}\cdots\text{S}$ interactions, forming one-dimensional zigzag chains along the b axis.

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Comment

Most benzoylthiourea derivatives, such as *N*-benzoyl-*N'*-phenylthiourea (Yamin & Yusof, 2003), adopt a *trans-cis* configuration with respect to the position of the benzoyl and phenyl groups relative to the thiono S atom across the C—N bonds. The title compound, (I), is analogous to *N*-benzoyl-*N'*-phenylthiourea, with the benzoyl group replaced by a non-aromatic butyl group, and maintains the same *trans-cis* configuration (Fig. 1). The central fragment, (C1/C5—C7/S1/N1/N2/O1), is essentially planar with a maximum deviation from the least-squares plane of 0.042 (2) Å for atom N1. This plane makes an angle of 81.41 (9)° with the phenyl ring. There is an intramolecular hydrogen bond, N2—H2···O1 (Table 1), and as a result, a pseudo-six-membered ring (O1/H2/N2/C6/N1/C5) is formed. The molecules are linked by an intermolecular $\text{N}-\text{H}\cdots\text{S}$ hydrogen bond (Table 1) forming one-dimensional zigzag chains along the b axis (Fig. 2).



Experimental

An equimolar amount of benzamide (2.06 g, 17 mmol) in acetone (20 ml) was added dropwise to a stirred acetone solution (75 ml)

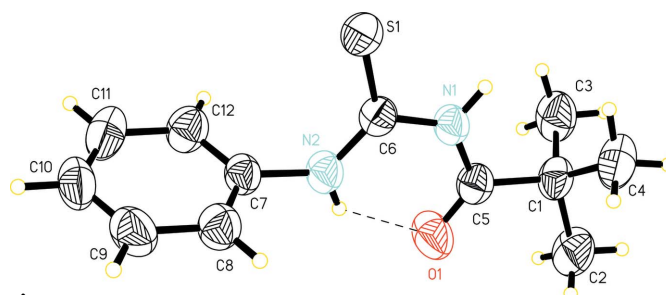


Figure 1
Molecular structure of (I), with 50% probability displacement ellipsoids. The dashed line indicates the $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond.

containing pivaloyl chloride (2.0 g, 17 mmol) and ammonium thiocyanate (1.16 g, 17 mmol). The mixture was refluxed for 1 h. The resulting solution was poured into a beaker containing some ice blocks. The white precipitate was filtered off and washed with distilled water and cold ethanol before being dried under vacuum. Good quality crystals were obtained by recrystallization from ethanol. Yield 72% (2.63 g).

Crystal data

$C_{12}H_{16}N_2OS$ $Z = 4$
 $M_r = 236.33$ $D_x = 1.232 \text{ Mg m}^{-3}$
 Monoclinic, $P2_1/c$ Mo $K\alpha$ radiation
 $a = 10.8783 (18) \text{ \AA}$ $\mu = 0.24 \text{ mm}^{-1}$
 $b = 6.3632 (11) \text{ \AA}$ $T = 273 (2) \text{ K}$
 $c = 18.963 (3) \text{ \AA}$ Block, colourless
 $\beta = 103.839 (4)^\circ$ $0.49 \times 0.31 \times 0.29 \text{ mm}$
 $V = 1274.5 (4) \text{ \AA}^3$

Data collection

Bruker SMART APEX CCD area-detector diffractometer 7283 measured reflections
 ω scans 2776 independent reflections
 Absorption correction: multi-scan 2016 reflections with $I > 2\sigma(I)$
 (SADABS; Sheldrick, 1996) $R_{int} = 0.025$
 $T_{min} = 0.893, T_{max} = 0.935$ $\theta_{max} = 27.0^\circ$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 0.3088P]$
 $R[F^2 > 2\sigma(F^2)] = 0.047$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.118$ $(\Delta/\sigma)_{max} < 0.001$
 $S = 1.08$ $\Delta\rho_{max} = 0.18 \text{ e \AA}^{-3}$
 2776 reflections $\Delta\rho_{min} = -0.13 \text{ e \AA}^{-3}$
 148 parameters
 H-atom parameters constrained

Table 1
 Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N2-H2 \cdots O1$	0.86	1.94	2.630 (2)	136
$N1-H1 \cdots S1^i$	0.86	2.82	3.638 (2)	159

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

After their location in a difference Fourier map, all H atoms were positioned geometrically and allowed to ride on the parent C or N atoms, with $C-H = 0.93-0.96 \text{ \AA}$ and $N-H = 0.86 \text{ \AA}$, with $U_{iso}(H) = 1.2(C,N) (CH_2 \text{ and } NH) \text{ or } 1.5U_{eq}(C) (CH_3)$.

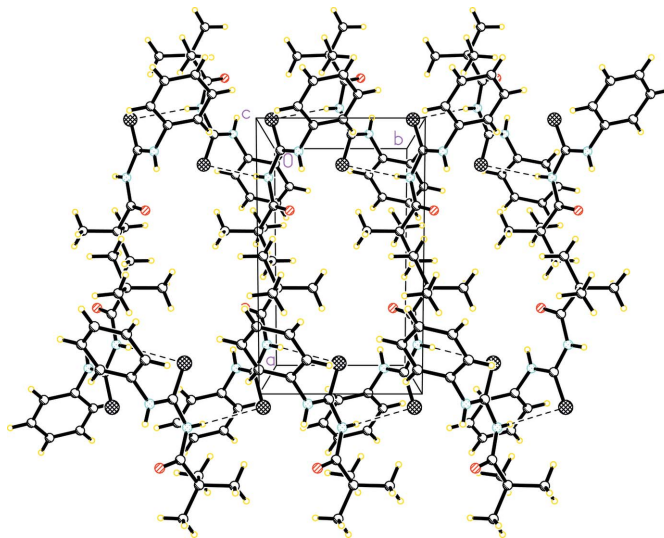


Figure 2
 Packing diagram of (I), viewed down the c axis. The dashed lines denote the $N-H \cdots S$ hydrogen bonds.

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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