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Key indicators

Single-crystal X-ray study

T = 213 K

Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$

R factor = 0.053

wR factor = 0.148

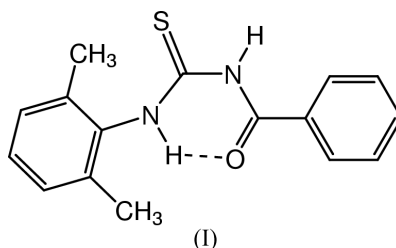
Data-to-parameter ratio = 16.3

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**N-Benzoyl-N'-(2,6-dimethylphenyl)thiourea**

In the title compound, $\text{C}_{16}\text{H}_{16}\text{N}_2\text{OS}$, the benzoyl and dimethylphenyl substituents have *trans* and *cis* configurations, respectively, with respect to the thiourea S atom. The aromatic rings of the two substituents are nearly perpendicular to each other. The molecules are interconnected into molecular chains along the *b* direction.

Comment

Although several thiourea derivatives, which are used as ligands in metal complexes (Razak *et al.*, 2001, 2002), have been synthesized, knowledge of their molecular structures is still scarce. This may be due to the difficulty in obtaining a suitable crystal for X-ray diffraction studies. As part of our ongoing studies of thiourea–metal complexes, we have prepared the title compound, (I), in order to explore new ligands. Its X-ray crystal structure determination has been undertaken to elucidate its conformation, and the result is reported here.



The bond lengths and angles in (I) (Fig. 1 and Table 1) are within normal ranges (Allen *et al.*, 1987). The dimethylphenyl and benzoyl substituents lie *cis* and *trans*, respectively, to the S atom across the thiourea C–N bonds, as was observed in the 3,4-dimethylphenyl analogue (Shanmuga Sundara Raj *et al.*, 1999). The bond lengths and angles are typical for thiourea compounds and are comparable with the corresponding values in the literature (Zhang *et al.*, 1996; Cao *et al.*, 1996). However, the bulky 2,6-dimethyl groups result in a significant reduction in the C8–N1–C9 bond angle [$123.2(2)^\circ$] compared with that in its 3,4-dimethylphenyl analogue [$127.9(2)^\circ$]. Also, the carbonyl C7–O1 bond length is shortened from $1.233(2) \text{ \AA}$ in its 3,4-dimethylphenyl analogue to $1.219(2)^\circ$ in the title compound.

The carbonyl–thiourea (S1/C8/N1/N2/C7/O1) moiety joining the two phenyl rings of the dimethylphenyl and benzoyl rings is planar, with a maximum deviation of $0.054(1) \text{ \AA}$ for atom C7. The planarity of the carbonyl–thiourea moiety is due to the sp^2 states of the N and C atoms. An intramolecular N1–H1A \cdots O1 interaction (Table 2), which forms a six-membered ring (O1–C7–N2–C8–N1–H1A)

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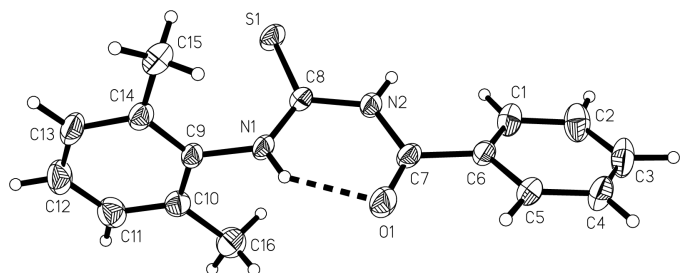


Figure 1
The molecular structure of the title compound, shown with 50% probability displacement ellipsoids

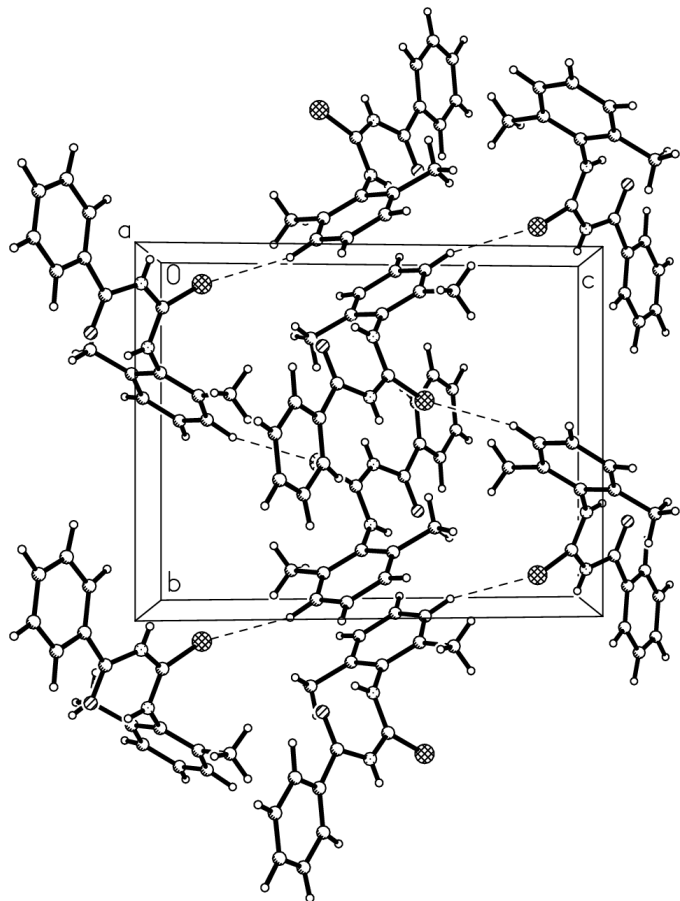


Figure 2
Packing diagram of the title complex viewed down the *a* axis. The dashed lines denote the C—H...S intermolecular contacts.

(Fig. 1) and increases the π -conjugation of the carbonyl and thiourea, may also contribute to the planarity. The carbonyl–thiourea plane makes dihedral angles of 23.1 (1) and 76.2 (1) $^\circ$ with the aromatic rings of the dimethylphenyl and benzoyl substituents, respectively.

The aromatic rings are nearly perpendicular to each other, the corresponding dihedral angle being 81.3 (1) $^\circ$. The two methyl groups are on opposite sides of the aromatic ring and deviate from the plane by 0.013 (2) Å (C15) and -0.050 (2) Å (C16).

In the title structure, the thiourea atom S1 is involved in a weak intermolecular C13—H13...S1ⁱ interaction [Table 2;

symmetry code: (i) $1 - x, y - \frac{1}{2}, \frac{3}{2} - z$], which interconnects the molecules into infinite zigzag molecular chains along the *b* direction (Fig. 2). The packing is stabilized by this interaction along with the dipole–dipole and van der Waals interactions.

Experimental

A solution of 2,6-dimethylaniline (0.33 g, 20 mmol) in acetone (40 ml) was added dropwise to 40 ml of an acetone solution containing an equimolar amount of benzoyl thiocyanate in a two-neck round-bottomed flask. The solution was refluxed for about 1 h and then cooled in ice. The white precipitate which formed was filtered off and washed with ethanol–distilled water, then dried in a vacuum (yield 75%). Recrystallization from DMF yielded single crystals suitable for X-ray analysis.

Crystal data

$C_{16}H_{16}N_2OS$	$D_x = 1.286 \text{ Mg m}^{-3}$
$M_r = 284.37$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 5482 reflections
$a = 9.5077$ (3) Å	$\theta = 2.8\text{--}28.3^\circ$
$b = 11.1226$ (4) Å	$\mu = 0.22 \text{ mm}^{-1}$
$c = 14.0147$ (5) Å	$T = 213$ (2) K
$\beta = 97.823$ (1) $^\circ$	Slab, colorless
$V = 1468.27$ (9) Å ³	$0.40 \times 0.28 \times 0.12 \text{ mm}$
$Z = 4$	

Data collection

Siemens SMART CCD area-detector diffractometer	3564 independent reflections
ω scans	2416 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.084$
$T_{\text{min}} = 0.918, T_{\text{max}} = 0.974$	$\theta_{\text{max}} = 28.3^\circ$
8741 measured reflections	$h = -11 \rightarrow 12$
	$k = -14 \rightarrow 14$
	$l = -18 \rightarrow 12$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.053$	$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2]$
$wR(F^2) = 0.148$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.94$	$(\Delta/\sigma)_{\text{max}} = 0.001$
3564 reflections	$\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
219 parameters	$\Delta\rho_{\text{min}} = -0.52 \text{ e \AA}^{-3}$

Table 1

Selected interatomic distances (Å).

S1—C8	1.6599 (19)	N1—C9	1.436 (2)
O1—C7	1.219 (3)	N2—C7	1.373 (2)
N1—C8	1.331 (3)	N2—C8	1.395 (2)

Table 2

Hydrogen-bonding geometry (Å, $^\circ$).

<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—H1A...O1	0.82 (2)	1.99 (2)	2.670 (2)	140 (2)
C13—H13...S1 ⁱ	0.98 (3)	2.94 (3)	3.862 (2)	157 (2)

Symmetry codes: (i) $1 - x, y - \frac{1}{2}, \frac{3}{2} - z$.

The H atoms were located from a difference Fourier map and were refined isotropically, except for the H atoms attached to C12, C15 and C16 which were fixed geometrically and treated as riding atoms, with C—H = 0.93–0.96 Å and $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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