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## Key indicators

Single-crystal X-ray study

T = 120 K

Mean  $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$ 

R factor = 0.043

wR factor = 0.109

Data-to-parameter ratio = 18.8

For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

## 1-(4-Chlorophenyl)-3-(4-methylbenzoyl)thiourea

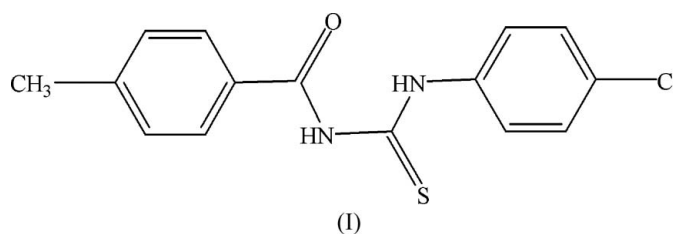
Received 30 May 2006

Accepted 12 June 2006

In the structure of the title compound,  $\text{C}_{15}\text{H}_{13}\text{ClN}_2\text{OS}$ , the dihedral angle between the two aromatic ring planes is  $30.46(8)^\circ$ . Intermolecular  $\text{N}-\text{H}\cdots\text{S}$  hydrogen bonds link the molecules into dimeric units which are stacked along  $[010]$ .

## Comment

Thiourea derivatives are extremely versatile building blocks for the manufacture of a wide variety of heterocyclic compounds. This, and our resulting interest in the synthesis of these thioureas as intermediates for the synthesis of novel heterocycles and for the systematic study of their bioactivity and complexation behavior, have been discussed previously (Saeed & Flörke, 2006). In the title compound, (I), the observed  $\text{C}=\text{S}$  and  $\text{C}=\text{O}$  double bonds, as well as the shortened  $\text{C}-\text{N}$  bond lengths, are typical of thiourea compounds (Table 1 and Fig. 1). The conformation of the molecule with respect to the thiocarbonyl and carbonyl units is nearly planar, as reflected by the torsion angles  $\text{C}7-\text{N}2-\text{C}8-\text{O}$  and  $\text{C}8-\text{N}2-\text{C}7-\text{N}1$  of  $2.5(3)$  and  $-3.8(3)^\circ$ , respectively. In the related 2-chlorophenyl compound (Saeed & Flörke, 2006), this conformational twist is more pronounced, with average torsion angles of  $-5.8$  and  $16.2^\circ$ , respectively. The dihedral angle formed by the chlorophenyl and tolyl rings of (I) is  $30.46(8)^\circ$  and the relevant torsion angle  $\text{N}2-\text{C}8-\text{C}9-\text{C}10$  is  $24.6(2)^\circ$ .



In (I), there is an intramolecular  $\text{N}1-\text{H}\cdots\text{O}$  hydrogen bond (Table 2), which is typical for thiourea. The crystal packing shows intermolecular  $\text{N}-\text{H}\cdots\text{S}$  hydrogen bonds which link molecules into dimers which are stacked along  $[010]$  (Fig. 2). This dimer pattern is common for these thiourea compounds (Arslan *et al.*, 2003; Rauf *et al.*, 2006).

## Experimental

A solution of 4-methylbenzoyl chloride (1.75 g, 10 mmol) in acetone (50 ml) was added dropwise to a suspension of potassium thiocyanate (0.97 g, 10 mmol) in acetone (30 ml) and the reaction mixture was refluxed for 30 min. After cooling to room temperature, a solution of

2-chloroaniline (1.28 g, 10 mmol) in acetone (10 ml) was added and the resulting mixture refluxed for 2.0 h. The reaction mixture was poured into cold water at which point the thiourea was precipitated as a solid. Recrystallization from ethanol gave colorless crystals (2.58 g, 8.5 mmol, m.p. 425 K). IR (KBr,  $\text{cm}^{-1}$ ): 3351 (free NH), 3200 (assoc. NH), 1667 (CO), 1610 (aromatic), 1529 (thioureido I) 1325 II, 1160 III, 744, 762. Analysis calculated for  $\text{C}_{15}\text{H}_{13}\text{ClN}_2\text{OS}$ : C 59.11, H 4.30, N 9.19, S 10.52%; found: C 59.32, H 4.29, N 9.15, S 10.56%.

#### Crystal data

$\text{C}_{15}\text{H}_{13}\text{ClN}_2\text{OS}$	$Z = 4$
$M_r = 304.78$	$D_x = 1.443 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 12.0097 (8) \text{ \AA}$	$\mu = 0.42 \text{ mm}^{-1}$
$b = 6.3118 (4) \text{ \AA}$	$T = 120 (2) \text{ K}$
$c = 18.8101 (13) \text{ \AA}$	Plate, colorless
$\beta = 100.337 (1)^\circ$	$0.48 \times 0.24 \times 0.09 \text{ mm}$
$V = 1402.72 (16) \text{ \AA}^3$	

#### Data collection

Bruker SMART APEX-I diffractometer	13563 measured reflections
$\varphi$ and $\omega$ scans	3401 independent reflections
Absorption correction: multi-scan (SADABS; Bruker, 2002)	2905 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.825$ , $T_{\max} = 0.963$	$R_{\text{int}} = 0.031$
	$\theta_{\text{max}} = 28.1^\circ$

#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.5904P]$
$R[F^2 > 2\sigma(F^2)] = 0.043$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.109$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.49 \text{ e \AA}^{-3}$
3401 reflections	$\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$
181 parameters	
H-atom parameters constrained	

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

C11–C4	1.7393 (17)	N1–C1	1.422 (2)
S1–C7	1.6696 (18)	N2–C8	1.379 (2)
O1–C8	1.223 (2)	N2–C7	1.388 (2)
N1–C7	1.333 (2)		
C7–N1–C1	130.71 (15)	C8–N2–C7	129.34 (15)
C8–N2–C7–N1	−3.8 (3)	N2–C8–C9–C10	24.6 (2)
C7–N2–C8–O1	2.5 (3)		

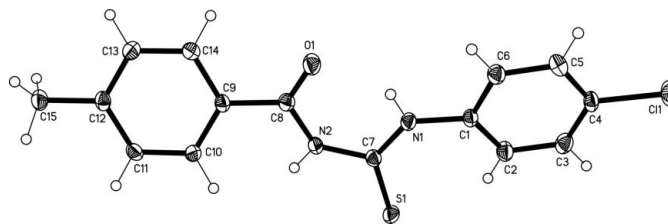
**Table 2**

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1–H1A $\cdots$ O1	0.88	1.92	2.6567 (19)	140
N2–H2B $\cdots$ S1 <sup>i</sup>	0.88	2.68	3.4250 (15)	144

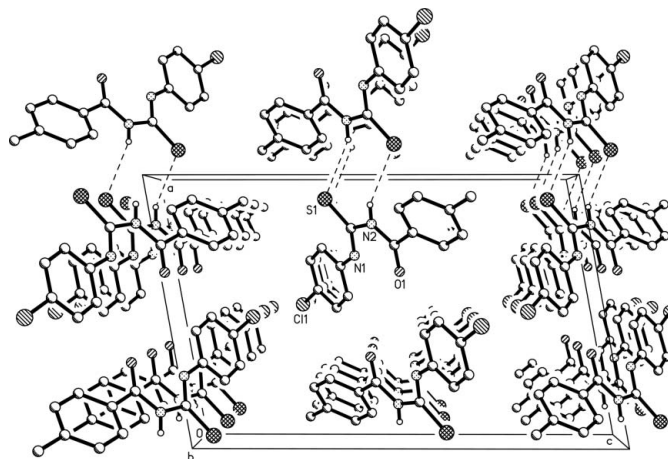
Symmetry code: (i)  $-x, -y + 2, -z$ .

H atoms were located in difference syntheses and refined at idealized positions ( $N-H = 0.88$ ,  $C-H = 0.95-0.98 \text{ \AA}$ ) riding on the



**Figure 1**

The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.



**Figure 2**

The crystal packing viewed along  $[010]$ , with intermolecular hydrogen bonds shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

C or N atoms, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$  or  $1.5U_{\text{eq}}(\text{methyl C})$ . Methyl H atoms were refined on the basis of rigid groups allowed to rotate but not tip.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 2002); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

AS gratefully acknowledges the Higher Education Commission of Pakistan for financial assistance.

#### References

- Arslan, H., Flörke, U. & Külcü, N. (2003). *J. Chem. Crystallogr.* **33**, 919–924.  
 Bruker (2002). SMART (Version 5.62), SAINT (Version 6.02), SHELXTL (Version 6.10) and SADABS (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.  
 Rauf, K., Badshah, A., Flörke, U. & Saeed, A. (2006). *Acta Cryst.* **E62**, o1419–o1420.  
 Saeed, A. & Flörke, U. (2006). *Acta Cryst.* **E62**, o2403–o2405.