

M. Khawar Rauf,<sup>a</sup> Amin  
Badshah,<sup>a</sup> Aamer Saeed<sup>a</sup> and  
Michael Bolte<sup>b\*</sup><sup>a</sup>Department of Chemistry, Quaid-i-Azam  
University, Islamabad 45320, Pakistan, and<sup>b</sup>Institut für Anorganische Chemie, J. W.  
Goethe-Universität Frankfurt, Marie-Curie-  
Strasse 11, 60439 Frankfurt/Main, GermanyCorrespondence e-mail:  
bolte@chemie.uni-frankfurt.de

## Key indicators

Single-crystal X-ray study  
 $T = 173\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$   
 $R$  factor = 0.037  
 $wR$  factor = 0.106  
Data-to-parameter ratio = 22.4For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

## 1-(2-Chlorobenzoyl)-3-phenylthiourea

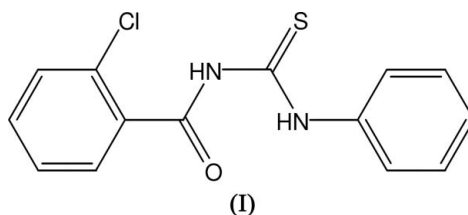
The phenyl and benzoyl groups in the title molecule,  $\text{C}_{14}\text{H}_{11}\text{ClN}_2\text{OS}$ , are *cis* and *trans*, respectively, with respect to the  $\text{C}=\text{S}$  bond. The molecular conformation is stabilized by an  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond and the crystal packing is characterized by  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{N}-\text{H}\cdots\text{S}$  hydrogen bonds.

Received 22 February 2006

Accepted 28 February 2006

## Comment

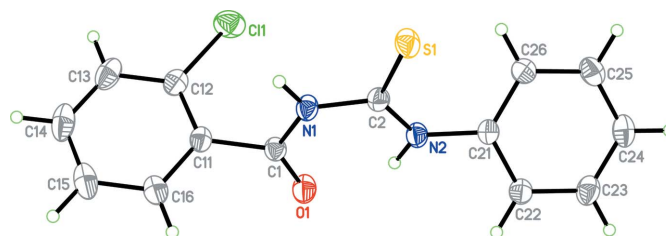
The background to this study has been set out in the preceding paper (Rauf *et al.*, 2006).



The molecular structure of (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 5.27 plus one update; *MOGUL* Version 1.1; Allen, 2002). The central carbonylthiourea group is almost planar (r.m.s. deviation for  $\text{O1}/\text{C1}/\text{N1}/\text{C2}/\text{S1}/\text{N2}$  is  $0.054\text{ \AA}$ ). The chlorophenyl and phenyl rings form dihedral angles of  $74.31(3)^\circ$  and  $40.22(4)^\circ$ , respectively, with the central carbonylthiourea plane. The dihedral angle between the two aromatic rings is  $34.92(5)^\circ$ . An intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond gives rise to a six-membered ring. The crystal packing is stabilized by  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{N}-\text{H}\cdots\text{S}$  hydrogen bonds (Table 2).

## Experimental

A solution of 2-chlorobenzoyl chloride (1.750 g, 10 mmol) in acetone (50 ml) was added dropwise to a suspension of  $\text{KSCN}$  (1.00 g, 10 mmol) in acetone (30 ml). The reaction mixture was heated under reflux for 45 min, and then cooled to room temperature. Afterwards a solution of aniline (0.93 g, 10 mmol) in acetone (15 ml) was added



**Figure 1**  
Perspective view of the title compound, with displacement ellipsoids drawn at the 50% probability level.

and the resulting mixture was stirred for 3 h. The reaction mixture was then poured into crushed ice and stirred well. The solid product was separated and washed with deionized water and purified by recrystallization from toluene to give crystals of the title compound, in an overall yield of 85%.

#### Crystal data

$C_{14}H_{11}ClN_2OS$	$D_x = 1.410 \text{ Mg m}^{-3}$
$M_r = 290.76$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 24244 reflections
$a = 20.5099 (13) \text{ \AA}$	$\theta = 2.5\text{--}30.4^\circ$
$b = 7.3281 (3) \text{ \AA}$	$\mu = 0.42 \text{ mm}^{-1}$
$c = 18.5344 (12) \text{ \AA}$	$T = 173 (2) \text{ K}$
$\beta = 100.437 (5)^\circ$	Plate, colourless
$V = 2739.6 (3) \text{ \AA}^3$	$0.37 \times 0.35 \times 0.17 \text{ mm}$
$Z = 8$	

#### Data collection

Stoe IPDS-II two-circle diffractometer	4055 independent reflections
$\omega$ scans	3793 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	$R_{\text{int}} = 0.059$
$T_{\text{min}} = 0.859$ , $T_{\text{max}} = 0.932$	$\theta_{\text{max}} = 30.3^\circ$
24244 measured reflections	$h = -28 \rightarrow 28$
	$k = -10 \rightarrow 10$
	$l = -22 \rightarrow 26$

#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 1.3534P]$
$R[F^2 > 2\sigma(F^2)] = 0.037$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.106$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.40 \text{ e \AA}^{-3}$
4055 reflections	$\Delta\rho_{\text{min}} = -0.50 \text{ e \AA}^{-3}$
181 parameters	Extinction correction: <i>SHELXL97</i>
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.0112 (10)

**Table 1**

Selected bond lengths ( $\text{\AA}$ ).

Cl1—C12	1.7419 (13)	C1—N1	1.3725 (13)
S1—C2	1.6758 (11)	C2—N2	1.3391 (13)
C1—O1	1.2269 (13)		

**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—H1 $\cdots$ S1 <sup>i</sup>	0.903 (18)	2.492 (18)	3.3743 (9)	165.6 (16)
N2—H2 $\cdots$ O1	0.89 (2)	1.95 (2)	2.6888 (12)	138.6 (17)
N2—H2 $\cdots$ O1 <sup>ii</sup>	0.89 (2)	2.57 (2)	3.2917 (13)	138.0 (15)

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

H atoms were located in a difference electron-density map, but those bonded to C atoms were refined with fixed individual displacement parameters [ $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ ] using a riding model, with  $C-H = 0.95 \text{ \AA}$ . The H atoms bonded to nitrogen were refined freely.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

MKR is grateful to The Higher Education Commission of Pakistan for financial support for PhD programme vide Scholarship No. (PIN) ILC(0363104).

#### References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.  
 Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.  
 Rauf, M. K., Badshah, A., Flörke, U., Saeed, A. & Saeed, M. A. (2006). *Acta Cryst.* **E62**, o1060–o1061.  
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.  
 Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Sheldrick, G. M. (1997). *SHELXL97*. Univ. of Göttingen, Germany.  
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.  
 Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.