

1-(2-Chlorobenzoyl)-3-(3-chlorophenyl)thiourea

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Key indicators

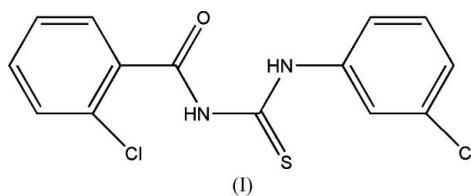
Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.032
 wR factor = 0.085
Data-to-parameter ratio = 14.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title compound, $\text{C}_{14}\text{H}_{10}\text{Cl}_2\text{N}_2\text{OS}$, the geometric parameters do not show unusual features. The thiocarbonyl and carbonyl groups are almost coplanar with the chlorophenyl ring, as reflected by the torsion angles of 0.7 (3) and -5.8 (3)°, respectively. The structure exhibits inter- and intramolecular hydrogen-bonding interactions.

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Comment

Thiourea derivatives are very useful building blocks for the synthesis of a wide range of aliphatic macromolecular and heterocyclic compounds. Benzothiazoles have been prepared from arylthioureas in the presence of bromine (Patil & Chedekel, 1984), while condensation of thiourea with halocarbonyl compounds forms 2-aminothiazoles (Baily *et al.*, 1996). 2-Methyl-aminothiazolines have been synthesized by cyclization of *N*-(2-hydroxyethyl)-*N'*-methylthioureas (Namgun *et al.*, 2001). Thioureas are efficient guanylating agents (Maryanoff *et al.*, 1986). *N,N*-Dialkyl-*N*-aroylthioureas have been used efficiently for the extraction of nickel, palladium and platinum metals (Koch, 2001). Aliphatic and acylthioureas are well known for their fungicidal, antiviral, pesticidal and plant-growth regulating activities (Upadlgaya & Srivastava, 1982; Wegner *et al.*, 1986). Symmetrical and unsymmetrical thioureas have shown antifungal activity against the plant pathogens *Pyricularia oryzae* and *Drechslera oryzae* (Krishnamurthy *et al.*, 1999). We became interested in the synthesis of these thioureas as intermediates in the synthesis of novel guanidines and heterocyclic compounds for the systematic study of bioactivity and complexation behaviour and we present here the crystal structure of the title compound (I).



Compound (I) (Fig. 1) shows the typical thiourea $\text{C}=\text{S}$ and $\text{C}=\text{S}$ double bonds, as well as shortened $\text{C}-\text{N}$ bond lengths (Table 1). The thiocarbonyl and carbonyl groups are almost coplanar with the chlorophenyl ring, as shown by the $\text{C}2-\text{N}1-\text{C}1-\text{O}1$ [0.7 (3)°] and $\text{N}2-\text{C}2-\text{N}1-\text{C}1$ [-5.8 (3)°] torsion angles. This is associated with the expected typical thiourea intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond (Table 2). The dihedral angle formed by the two benzene ring planes is 46.89 (7)°. Other geometric parameters present no unusual

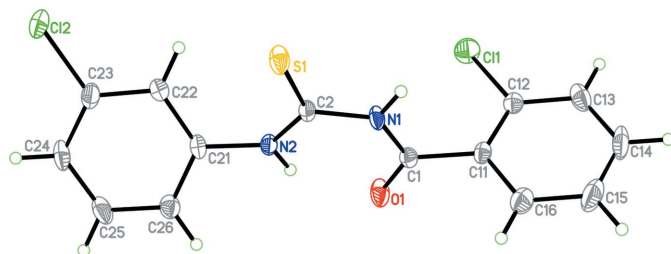


Figure 1
Molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

features (Khawar Rauf, Badshah, Bolte & Zaeem Akhtar, 2006, Khawar Rauf, Badshah & Bolte, 2006). The crystal packing shows intermolecular N—H···S hydrogen bonds (Table 2, Fig. 2), forming dimers. The Cl atoms are not involved in any type of hydrogen bonding.

Experimental

Freshly prepared 2-chlorobenzoyl chloride (1.75 g, 10 mmol) was added to a suspension of KSCN ((1.00 g, 10 mmol) in acetone (30 ml). The reaction mixture was stirred for 15 minutes. Afterwards neat 3-chloroaniline (1.27 g, 10 mmol) was added and the resulting mixture was stirred for 1 h. The reaction mixture was then poured into acidified water and stirred well. The solid product was separated, washed with deionized water and purified by recrystallization from methanol–dichloromethane (1:1, v/v) to give fine crystals of the title compound in an overall yield of 85%. The full spectroscopic and physical characterization will be reported elsewhere.

Crystal data

$C_{14}H_{10}Cl_2N_2OS$	$Z = 4$
$M_r = 325.20$	$D_x = 1.493 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 10.7919 (10) \text{ \AA}$	$\mu = 0.59 \text{ mm}^{-1}$
$b = 9.9331 (6) \text{ \AA}$	$T = 173 (2) \text{ K}$
$c = 13.7534 (12) \text{ \AA}$	Block, colourless
$\beta = 101.002 (7)^\circ$	$0.39 \times 0.35 \times 0.34 \text{ mm}$
$V = 1447.2 (2) \text{ \AA}^3$	

Data collection

Stoe IPDS II two-circle diffractometer	11996 measured reflections
ω scans	2704 independent reflections
Absorption correction: multi-scan <i>MULABS</i> (Spek, 2003; Blessing, 1995)	2283 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.803$, $T_{\max} = 0.825$	$R_{\text{int}} = 0.055$
	$\theta_{\text{max}} = 25.6^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2 + 0.3673P]$
$R[F^2 > 2\sigma(F^2)] = 0.032$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.085$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$
2704 reflections	$\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$
190 parameters	Extinction correction: <i>SHELXL97</i>
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.0134 (14)

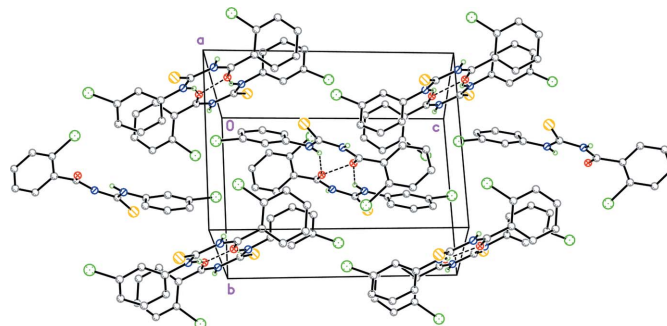


Figure 2
Packing diagram of (I), viewed along the a axis. Dashed lines indicate hydrogen bonds.

Table 1

Selected bond lengths (\AA).

S1—C2	1.6730 (18)	N1—H1	0.82 (2)
C1—C12	1.7405 (18)	C2—N2	1.343 (2)
C1—O1	1.222 (2)	N2—C21	1.432 (2)
C1—N1	1.371 (2)	N2—H2	0.87 (2)
N1—C2	1.403 (2)		

Table 2

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2···O1	0.87 (2)	1.98 (2)	2.701 (2)	138.7 (18)
N1—H1···S1 ⁱ	0.82 (2)	2.61 (2)	3.4009 (16)	162.9 (19)
N2—H2···O1 ⁱⁱ	0.87 (2)	2.48 (2)	3.2127 (19)	142.6 (18)

Symmetry codes: (i) $-x, -y + 1, -z + 1$; (ii) $-x + 1, -y + 1, -z + 1$.

All H atoms were found in a difference map, but those bonded to C were positioned geometrically and allowed to ride on their parent atoms at a distance of 0.95 \AA and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The H atoms bonded to N 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, 1997); 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*.

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