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

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.093
Data-to-parameter ratio = 13.6

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

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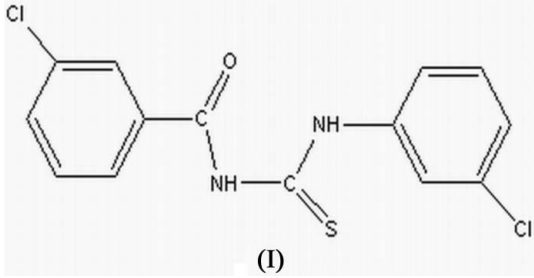
The chlorophenyl and benzoyl groups in the title compound, $C_{14}H_{10}Cl_2N_2OS$, are *cis* and *trans*, respectively, with respect to the C=S bond. The crystal packing is characterized by N—H···O, N—H···S and N—H···Cl hydrogen bonds.

Comment

N-Substituted and *N*,*N*′-disubstituted thiourea derivatives are of practical interest, due to their coordination behaviour towards transition metals (Schuster *et al.*, 1990) and their biological activity (Frech *et al.*,1970; Madan & Taneja, 1991).



(I)

The title compound, (I) (Fig. 1), is a typical *N*,*N*′-disubstituted thiourea derivative with normal geometric parameters (Cambridge Structural Database, Version 5.27 plus one update; *MOGUL* Version 1.1; Allen, 2002). The C2=S1 and C1=O1 bonds (Table 1) both show the expected full double-bond character, while the short values for the C1—N1, C2—N1, C2—N2, and C21—N2 bond lengths indicate partial double-bond character.

The dihedral angle between the aromatic rings is 8.06 (5)°, and the corresponding angles to the thiourea plane are 43.56 (5)° for the C11–C16 ring and 48.93 (5)° for the C21–C26 ring.

An intramolecular N—H···O hydrogen bond is present (Table 2), forming a six-membered ring commonly observed in this class of compounds (Arslan *et al.*, 2004; Khawar Rauf *et al.*, 2006). In the crystal packing, intermolecular N—H···S and C—H···Cl hydrogen bonds link the molecules into centrosymmetric dimers stacked along the direction of the *a* axis (Fig. 2).

Experimental

Freshly prepared 3-chlorobenzoyl chloride (1.75 g, 10 mmol) was added to a suspension of KSCN (1.00 g, 10 mmol) in acetone (20 ml). The reaction mixture was stirred for 15 min, and then neat 3-chloroaniline (1.27 g, 10 mmol) was added and the resulting mixture stirred for 1 h. The reaction mixture was then poured into acidified

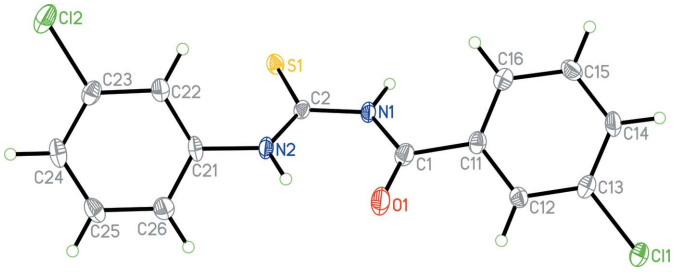


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

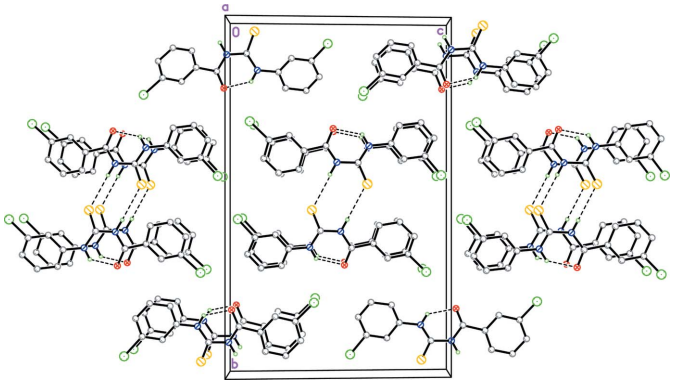


Figure 2
The crystal packing of (I), viewed along [100], with hydrogen bonds indicated as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

water and stirred well. The solid product which formed was separated, washed with deionized water and purified by recrystallization from methanol–dichloromethane (1:1) to give fine crystals of the title compound, (I), in an overall yield of 85%. Full spectroscopic and physical characterization will be reported elsewhere.

Crystal data

$C_{14}H_{10}Cl_2N_2OS$
M_r = 325.20
Monoclinic, $P2_1/c$
a = 3.9523 (4) Å
b = 23.762 (3) Å
c = 14.8970 (14) Å
β = 93.887 (8)°
V = 1395.8 (3) Å^3
Z = 4
D_x = 1.547 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.61 mm^{-1}
T = 173 (2) K
Needle, colourless
0.34 × 0.14 × 0.13 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.820, T_{max} = 0.925
8762 measured reflections
2591 independent reflections
2024 reflections with $I > 2\sigma(I)$
R_{int} = 0.080
θ_{max} = 25.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.093
S = 0.99
2591 reflections
190 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0073 (14)

Table 1
Selected bond lengths (Å).

S1—C2	1.679 (3)	N1—C2	1.409 (3)
C1—O1	1.224 (3)	C2—N2	1.335 (3)
C1—N1	1.387 (3)	N2—C21	1.442 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.90 (4)	1.97 (3)	2.674 (3)	134 (3)
N1—H1···S1^{i}	0.92 (4)	2.66 (4)	3.571 (2)	175 (3)
N2—H2···Cl1^{ii}	0.90 (4)	2.98 (4)	3.773 (3)	149 (3)

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

H atoms bonded to C were included in calculated positions, with C—H = 0.95 Å, and refined as riding on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C). 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, 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*.

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References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Arslan, H., Flörke, U. & Külcü, N. (2004). *Turk. J. Chem.* **28**, 673–678.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Frech, F. A., Blanz, E. J., Amaral, J. R. D. & French, D. A. (1970). *J. Med. Chem.* **13**, 1117–1124.
Khawar Rauf, M., Badshah, A., Saeed, A. & Bolte, M. (2006). *Acta Cryst.* E**62**, o1262–o1263.
Madan, V. K. & Taneja, A. D. (1991). *Indian Chem. Soc.* **68**, 471–472.
Schuster, M., Kugler, B. & Konig, K. H. (1990). *J. Anal. Chem.* **338**, 717–720.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 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*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.