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1-(3-Chlorobenzoyl)-3-(2,4,6-trichlorophenyl)thiourea

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.087
Data-to-parameter ratio = 14.3

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

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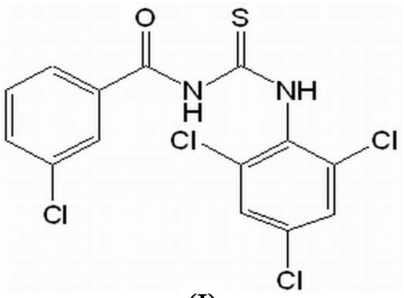
The title compound, $C_{14}H_8Cl_4N_2OS$, shows the typical geometric parameters of substituted thiourea derivatives. Whereas one NH group is involved in an intramolecular hydrogen bond, the other is shielded, so that no hydrogen bond is observed.

Comment

N-substituted and *N*,*N*′-disubstituted thiourea derivatives have potential applications due to their coordination behaviour towards transition metals (Schuster *et al.*, 1990) and their biological activity (Frech *et al.*, 1970; Madan *et al.*, 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 1.7; 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 84.17 (6)°. The dihedral angle between the thiourea plane (O1/C1/N1/C2/S1/N2) and the ring formed by atoms C11–C16 is 3.86 (13)°, and that between the thiourea plane and the ring formed by atoms C21–C26 is 86.58 (5)°.

An intramolecular N—H···O hydrogen bond is present (Table 2), which results in the formation of a six-membered ring, as is commonly observed in most benzoylthiourea derivatives (Arslan *et al.*, 2004; Khawar Rauf *et al.*, 2006). The other NH group is shielded, so that no hydrogen-bond acceptor can approach it to form an intermolecular hydrogen bond. A weak hydrogen bond between C23—H of an aromatic ring and S is noted (Table 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

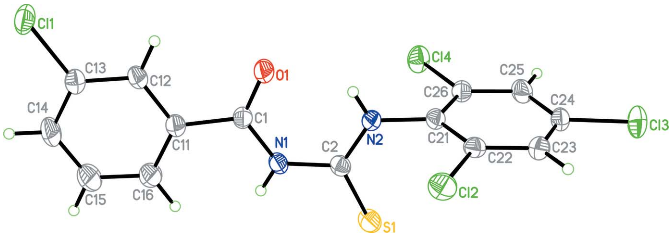


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

(30 ml). The reaction mixture was stirred for 15 min. Neat 2,4,6-trichloroaniline (1.97 g, 10 mmol) was then 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), to give fine crystals of (I) (overall yield 85%; m.p. 453 K).

Crystal data

$C_{14}H_8Cl_4N_2OS$
$M_r = 394.08$
Monoclinic, $P2_1/n$
$a = 12.4021$ (12) Å
$b = 9.2723$ (9) Å
$c = 14.5212$ (15) Å
$\beta = 107.379$ (8)°
$V = 1593.6$ (3) Å^3
$Z = 4$
$D_x = 1.643$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.87$ mm^{-1}
$T = 173$ (2) K
Block, colourless
0.48 × 0.46 × 0.43 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
$T_{\min} = 0.679$, $T_{\max} = 0.705$
8381 measured reflections
2968 independent reflections
2559 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.046$
$\theta_{\max} = 25.6°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.087$
$S = 1.03$
2968 reflections
208 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.8237P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.28$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0115 (11)

Table 1
Selected bond lengths (Å).

S1—C2	1.662 (2)	C1—O1	1.226 (3)
Cl1—C13	1.750 (2)	C1—N1	1.372 (3)
Cl2—C22	1.737 (2)	C2—N2	1.340 (3)
Cl3—C24	1.743 (2)	N2—C21	1.426 (3)
Cl4—C26	1.738 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.82 (3)	2.01 (3)	2.650 (2)	135 (3)
C23—H23···S1$^{\rm i}$	0.95	2.74	3.638 (2)	158

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

C-bound H atoms were included in the riding-model approximation, with C—H = 0.95 Å and with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C). N-bound H atoms were refined freely; N—H = 0.82 (3) Å.

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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