Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(4-Chlorophenyl)-3-(2,6-dichlorobenzoyl)-thiourea

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.032
wR factor = 0.084
Data-to-parameter ratio = 14.4

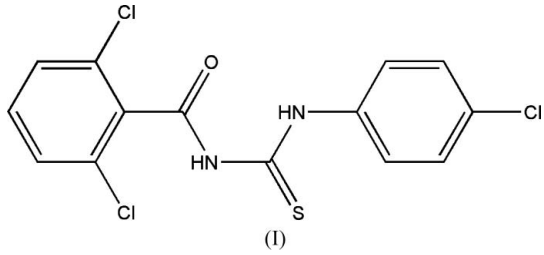
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 4 August 2006
Accepted 6 August 2006

The title compound, $C_{14}H_9Cl_3N_2OS$, shows the typical geometric parameters of substituted thiourea derivatives. The crystal packing is characterized by N—H···O and N—H···S hydrogen bonds.

Comment

N,*N*′-Disubstituted thiourea derivatives are very useful building blocks for the synthesis of a wide range of aliphatic macromolecular and heterocyclic compounds. Thus, benzothiazoles have been prepared from arylthioureas in the presence of bromine (Patil & Chedekel, 1984), and condensation of thiourea with halocarbonyl compounds form 2-aminothiazoles (Baily *et al.*, 1996). 2-Methylaminothiazolines 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). 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 metal complexation behaviour and we present here the crystal structure of the title compound, (I).



(I)

Compound (I) (Fig. 1) shows the typical thiourea C═S and C═S double bonds, as well as shortened C—N bond lengths (Table 1). The thiocarbonyl and carbonyl groups are almost coplanar with the dichlorophenyl ring, as reflected by the torsion angles C2—N1—C1—O1 [8.3 (3)°] and N2—C2—N1—C1 [−2.6 (3)°]. This is associated with the expected typical thiourea intramolecular N—H···O hydrogen bond (Table 2). The dihedral angle formed by the two benzene ring planes is 18.30 (7)°. Other geometric parameters present no unusual features (Khawar Rauf *et al.*, 2006*a*,*b*). Intermolecular

N—H···S hydrogen bonds (Table 2 and Fig. 2) link the molecules into chains.

Experimental

Freshly prepared 2,6-dichlorobenzoyl chloride (2.1 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 min. Afterwards, neat 4-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 resulting solid product was separated and washed with deionized water and purified by recrystallization from methanol–dichloromethane (1:1 *v*/*v*) to give fine crystals of the title compound, (I), with an overall yield of 80%.

Crystal data

$C_{14}H_9Cl_3N_2OS$
M_r = 359.64
Triclinic, $P\overline{1}$
a = 7.1485 (9) Å
b = 8.8020 (10) Å
c = 12.8530 (16) Å
α = 79.102 (9)°
β = 76.787 (10)°
γ = 81.645 (10)°
V = 768.70 (16) Å^3
Z = 2
D_x = 1.554 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.73 mm^{-1}
T = 173 (2) K
Block, colourless
0.48 × 0.47 × 0.42 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.721, T_{max} = 0.749
6863 measured reflections
2860 independent reflections
2688 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 25.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.085
S = 1.07
2860 reflections
199 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2 + 0.4277P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.026 (3)

Table 1
Selected bond lengths (Å).

S1—C2	1.6748 (16)	O1—C1	1.223 (2)
Cl1—C12	1.7452 (18)	N1—C1	1.371 (2)
Cl2—C16	1.7389 (18)	N1—C2	1.405 (2)
Cl3—C24	1.7562 (18)	N2—C2	1.334 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S1^{i}	0.84 (2)	2.54 (2)	3.3548 (15)	161 (2)
N2—H2···O1	0.82 (3)	2.01 (3)	2.6743 (19)	137 (2)
N2—H2···O1^{ii}	0.82 (3)	2.33 (3)	2.9250 (19)	130 (2)

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

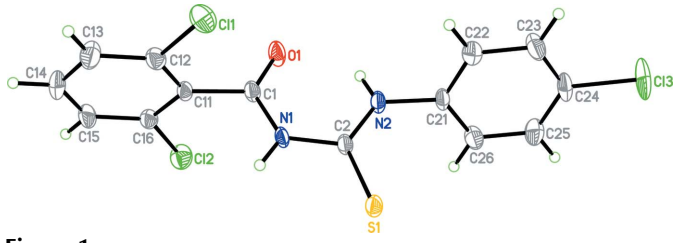


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

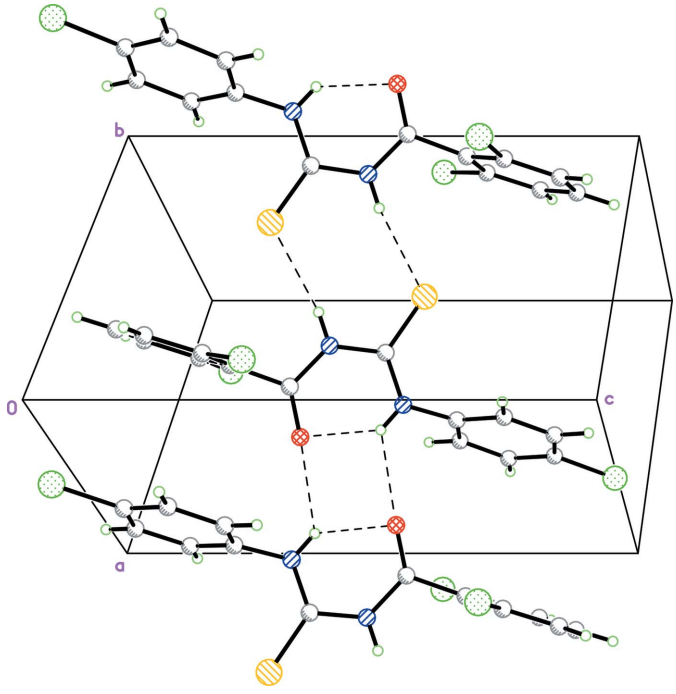


Figure 2
Packing diagram of (I) viewed along the the face diagonal of the *ab* plane. Hydrogen bonds shown as dashed lines.

H atoms were located in a difference map, but those bonded to C were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C)] using a riding model, with C—H = 0.95 Å. 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*.

MKR is grateful to The Higher Education Commission of Pakistan for financial support for the PhD program – scholarship No. (PIN) ILC (0363104).

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