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Key indicators

Single-crystal X-ray study

$T = 173$ K

Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å

R factor = 0.031

wR factor = 0.083

Data-to-parameter ratio = 17.4

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

1-(3-Chlorobenzoyl)-3-(2,6-dichlorophenyl)-thiourea

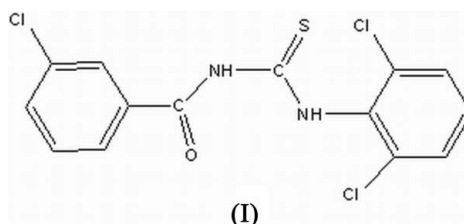
The asymmetric unit of the title compound, $\text{C}_{14}\text{H}_9\text{Cl}_3\text{N}_2\text{OS}$, contains two independent molecules with slightly different conformations with respect to the aromatic ring planes. These two independent molecules form dimers *via* intermolecular $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds and which are stacked along [100].

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Comment

N,N' -Disubstituted thiourea derivatives have attracted attention due to their coordination behaviour with transition metals and their biological activities (Schuster *et al.*, 1990; Madan *et al.*, 1991). The biological activities of complexes with thiourea derivatives have also been successfully screened (Frech *et al.*, 1970). Against this background, we present here the crystal structure of the title compound, (I).



There are two independent molecules in the asymmetric unit (Fig. 1), each having slightly different geometries. Within these molecules, the dihedral angles formed between the benzene rings are $68.69(5)$ and $63.30(5)^\circ$, respectively, for the molecule labelled by numerals only and that labelled by numerals plus the suffix A.

Intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 1) appear to control the essentially planar conformation of the central thiourea groups of each molecule. In the crystal structure, independent molecules are linked by two intermolecular $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds to form centrosymmetric dimers [$R_2^2(8)$ in the notation of Etter *et al.* (1990)], which are stacked along [100] (Fig. 2). The 2,6-dichloro substituents and the 3-chloro substituent do not lead to any significant changes in bond lengths compared with the molecular structures of the related 3,4-dichlorophenyl (Khawar Rauf, Badshah, Flörke & Saeed, 2006) and 2-chlorobenzoyl compounds (Khawar Rauf, Badshah, Saeed & Bolte, 2006).

Experimental

A solution of 3-chlorobenzoyl chloride (1.75 g, 10 mmol) in acetone (30 ml) was added to a suspension of KSCN ((1.00 g, 10 mmol) in acetone (30 ml). The reaction mixture was heated under reflux for a few minutes, and neat 2,6-dichloroaniline (1.63 g, 10 mmol) was

added and the resulting mixture was stirred for 1 h. The reaction mixture was then poured into water and stirred well. The solid product was separated, washed with water and purified by recrystallization from toluene to give crystals of the title compound, with an overall yield of 82%. Full spectroscopic and physical characterization will be reported elsewhere.

Crystal data

$C_{14}H_9Cl_3N_2OS$ $V = 1507.91 (18) \text{ \AA}^3$
 $M_r = 359.64$ $Z = 4$
 Triclinic, $P\bar{1}$ $D_x = 1.584 \text{ Mg m}^{-3}$
 $a = 7.8338 (6) \text{ \AA}$ $\text{Mo K}\alpha$ radiation
 $b = 14.4275 (9) \text{ \AA}$ $\mu = 0.74 \text{ mm}^{-1}$
 $c = 14.5450 (10) \text{ \AA}$ $T = 173 (2) \text{ K}$
 $\alpha = 100.858 (5)^\circ$ Block, colourless
 $\beta = 104.460 (6)^\circ$ $0.34 \times 0.32 \times 0.27 \text{ mm}$
 $\gamma = 101.767 (5)^\circ$

Data collection

Stoe IPDS-II two-circle diffractometer 21597 measured reflections
 6903 independent reflections
 6338 reflections with $I > 2\sigma(I)$
 Absorption correction: multi-scan $R_{int} = 0.048$
 (MULABS; Spek, 2003; Blessing, 1995) $\theta_{max} = 27.5^\circ$
 $T_{min} = 0.786, T_{max} = 0.824$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0438P)^2 + 0.6473P]$
 $R[F^2 > 2\sigma(F^2)] = 0.031$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.083$ $(\Delta/\sigma)_{max} = 0.001$
 $S = 1.03$ $\Delta\rho_{max} = 0.33 \text{ e \AA}^{-3}$
 6903 reflections $\Delta\rho_{min} = -0.31 \text{ e \AA}^{-3}$
 396 parameters Extinction correction: SHELXL97 (Sheldrick, 1997)
 H atoms treated by a mixture of independent and constrained refinement Extinction coefficient: 0.0162 (10)

Table 1 Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots S1^i$	0.81 (2)	2.85 (2)	3.6135 (13)	158.0 (18)
$N2-H2\cdots O1$	0.87 (2)	1.92 (2)	2.6437 (16)	139.0 (19)
$N1A-H1A\cdots S1A^{ii}$	0.89 (2)	2.54 (2)	3.4261 (13)	171.6 (17)
$N2A-H2A\cdots O1A$	0.89 (2)	1.92 (2)	2.6440 (16)	136.8 (19)

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

N-bound H atoms were located in a difference map and refined freely, with refined N–H distances in the range 0.81 (2)–0.89 (2) \AA . C-bound H atoms were included in calculated positions, with C–H = 0.95 \AA , and refined as riding on their parent C atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

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

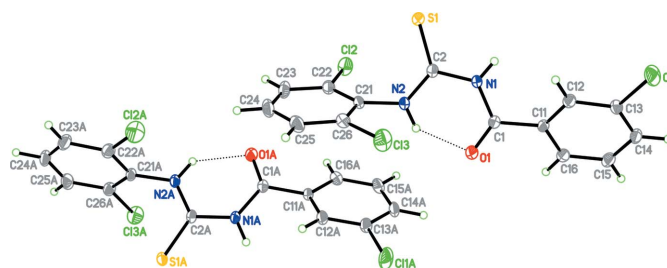


Figure 1 The molecular structures of the two independent molecules of (I). Displacement ellipsoids are drawn at the 50% probability level. Dotted lines indicate hydrogen bonds.

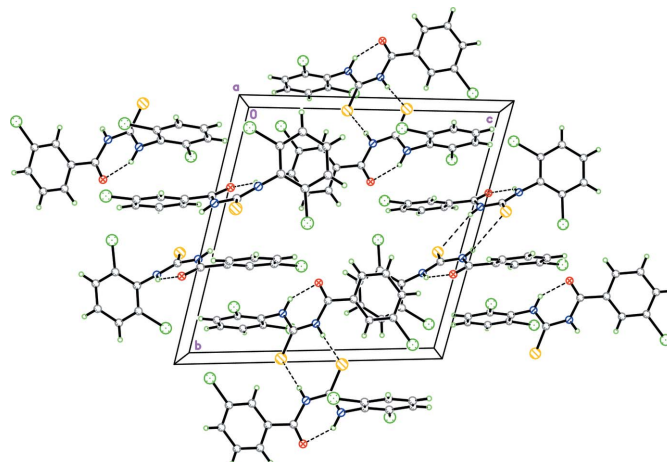


Figure 2 The crystal packing of (I), viewed along [100]. Hydrogen bonds are indicated by dashed lines.

SHELXTL-Plus (Sheldrick, 1991); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2003).

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