organic papers

1-Benzoyl-3-(3,4-dichlorophenyl)thiourea

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.097
Data-to-parameter ratio = 18.3

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

In the molecule of the title compound, $C_{14}H_{10}Cl_2N_2OS$, the dihedral angle between the two aromatic rings is 38.58 (6)°. In the crystal structure, centrosymmetric dimers are formed *via* intermolecular N—H···S hydrogen bonds [N···S = 3.4798 (16) Å]. These dimeric units are, in turn, connected by weak intermolecular C—H···O hydrogen bonds, forming one-dimensional chains along [100].

Comment

The background to this study has been set out in a previous paper (Rauf *et al.*, 2006a). The geometric parameters in (I) (Fig. 1) are typical for *N,N′*-disubstituted thiourea derivatives. The C7—S1 and C8—O1 bonds (Table 1) both show the expected double-bond character. The C1—N1, C7—N1, C7—N2 and C8—N2 bond lengths indicate partial double-bond character. Compared to the 3,4-chloro compound, the C—Cl and C1—N1 bonds are somewhat shorter than the corresponding bonds in the related 3-chloro- and 4-chloro-substituted compounds (Khawar Rauf *et al.*, 2006a,b). The dihedral angle between the benzene and phenyl rings is 38.58 (6)°.

(I)
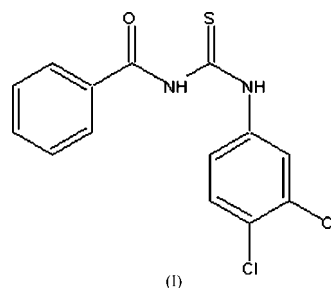
In the crystal structure, intermolecular N—H···S hydrogen bonds (Table 2) link molecules, forming centrosymmetric dimers which are, in turn, connected *via* weak C—H···O interactions to form one-dimensional chains along [100] and which are stacked along [010] (Fig. 2).

Experimental

A solution of benzoyl chloride (1.50 g, 10 mmol) in acetone (50 ml) was added dropwise to a suspension of KSCN (1.00 g, 10 mmol) in acetone (30 ml). The reaction mixture was heated under reflux for 45 min and then cooled to room temperature. A solution of 3,4-dichloroaniline (1.62 g, 10 mmol) in acetone (15 ml) was then added and the resulting mixture was stirred for 3 h. The reaction mixture was then poured into crushed ice and stirred well. The solid product was separated and washed with deionized water and purified by recrystallization from toluene to obtain crystals of the title compound, with an overall yield of 85%. Full spectroscopic and physical characterization are reported elsewhere.
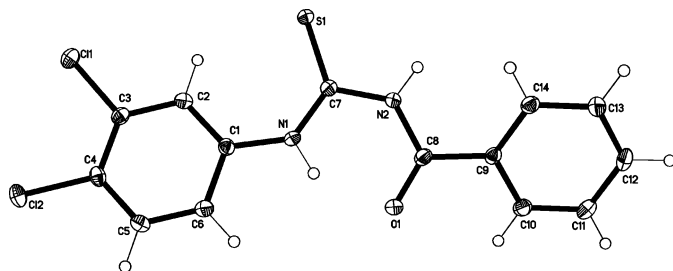
Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

Crystal data

$C_{14}H_{10}Cl_2N_2OS$	$D_x = 1.576$ Mg m^{-3}
$M_r = 325.20$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 3197
$a = 25.070$ (2) Å	reflections
$b = 3.8928$ (4) Å	$\theta = 2.9$–$28.0°$
$c = 28.137$ (3) Å	$\mu = 0.62$ mm^{-1}
$\beta = 93.326$ (2)°	$T = 120$ (1) K
$V = 2741.3$ (5) Å^3	Needle, colorless
$Z = 8$	$0.46 \times 0.04 \times 0.03$ mm

Data collection

Bruker SMART CCD diffractometer	3320 independent reflections
	2845 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.033$
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)	$\theta_{max} = 28.1°$
	$h = -32 \rightarrow 32$
$T_{min} = 0.763$, $T_{max} = 0.982$	$k = -5 \rightarrow 5$
11673 measured reflections	$l = -37 \rightarrow 32$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.040$	$+ 2.0546P]$
$wR(F^2) = 0.097$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} < 0.001$
3320 reflections	$\Delta\rho_{max} = 0.45$ e Å^{-3}
181 parameters	$\Delta\rho_{min} = -0.24$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

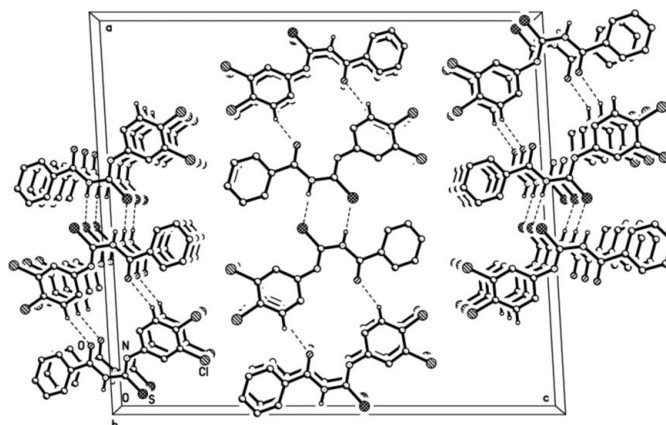|			
Cl1—C3	1.7304 (19)	N1—C7	1.343 (2)
Cl2—C4	1.7277 (18)	N1—C1	1.406 (2)
S1—C7	1.6579 (17)	N2—C8	1.375 (2)
O1—C8	1.233 (2)	N2—C7	1.398 (2)
C7—N1—C1	132.23 (15)	N1—C7—N2	114.26 (15)
C8—N2—C7	128.49 (15)	N2—C8—C9	116.79 (16)
C7—N1—C1—C6	−178.47 (19)	N2—C8—C9—C10	−153.84 (18)

Figure 2
The crystal packing, viewed along [010], with hydrogen bonds indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted.

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$A\cdots$O1	0.88	1.87	2.610 (2)	140
N2—H2$B\cdots$S1^i	0.88	2.63	3.4798 (16)	163
C5—H5$A\cdots$O1^{ii}	0.95	2.40	3.265 (2)	152

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

H atoms were included in idealized positions (C—H = 0.95 Å and N—H = 0.88 Å) in a riding-model approximation, with U_{iso}(H) = $1.2U_{eq}$(C,N).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

Bruker (2002). *SMART* (Version 5.62), *SAINT* (Version 6.02), *SHELXTL* (Version 6.10) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.

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Khawar Rauf, M., Badshah, A., Flörke, U. & Saeed, A. (2006b). *Acta Cryst.* E**62**., o1419–o1420.