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Key indicators

Single-crystal X-ray study

$T = 120$ K

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

R factor = 0.028

wR factor = 0.078

Data-to-parameter ratio = 18.6

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

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

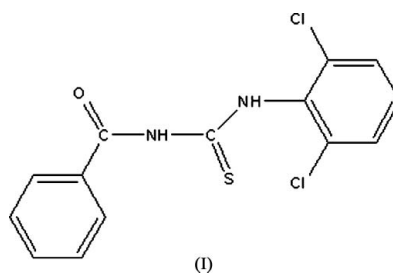
The asymmetric unit of the title compound, $\text{C}_{14}\text{H}_{10}\text{Cl}_2\text{N}_2\text{OS}$, contains two independent molecules with slightly different conformations with respect to the aromatic ring planes. The two independent molecules form dimers *via* intermolecular $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds; the dimers are stacked along [100].

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Comment

N,N' -Disubstituted thiourea derivatives have attracted attention due to their coordination behavior with transition metals. The complexation capacity of thiourea derivatives has been reported in several studies (Schuster *et al.*, 1990). In addition, thioureas have been shown to possess antibacterial, antifungal, antitubercular, antithyroid and insecticidal properties (Madan *et al.*, 1991). The biological activities of complexes with thiourea derivatives have been successfully screened for various biological activities (Frech *et al.*, 1970).



The molecular structure of (I) is shown in Fig. 1. There are two independent molecules (*A* and *B*) in the asymmetric unit having slightly different geometries. Within each molecule, the dihedral angles formed between the phenyl and benzene rings are $72.43(4)$ and $75.11(4)^\circ$ for molecules *A* and *B*, respectively. Intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2) appear to control the essentially planar conformation of the central thiourea units 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 dimers which are stacked along [100] (Fig. 2). The shortest $\text{C}-\text{H}\cdots\text{Cl}$ distance is 2.89 Å and there are no significant $\pi-\pi$ stacking interactions. The 2,6-dichloro substitution in (I) does not lead to any significant changes in bond lengths compared to the molecular structure of the related 2-chlorophenyl compound (Yamin & Yusof, 2003).

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. Afterwards, a solution

of 2,6-dichloroaniline (1.63 g, 10 mmol) in acetone (15 ml) was 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 give crystals of the title compound, in an overall yield of 82%. Full spectroscopic and physical characterization will be reported elsewhere.

Crystal data

$C_{14}H_{10}Cl_2N_2OS$
 $M_r = 325.20$
 Triclinic, $P\bar{1}$
 $a = 8.2731$ (4) Å
 $b = 12.9178$ (7) Å
 $c = 14.1746$ (7) Å
 $\alpha = 70.500$ (1)°
 $\beta = 85.118$ (1)°
 $\gamma = 77.804$ (1)°
 $V = 1395.55$ (12) Å³

$Z = 4$
 $D_x = 1.548$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 9938 reflections
 $\theta = 2.5$ – 28.3 °
 $\mu = 0.61$ mm⁻¹
 $T = 120$ (2) K
 Prism, colorless
 $0.48 \times 0.45 \times 0.38$ mm

Data collection

Bruker SMART-CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.759$, $T_{\max} = 0.801$
 13966 measured reflections

6714 independent reflections
 6195 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.014$
 $\theta_{\text{max}} = 28.1$ °
 $h = -10 \rightarrow 10$
 $k = -17 \rightarrow 17$
 $l = -18 \rightarrow 18$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.078$
 $S = 1.05$
 6714 reflections
 361 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 0.4524P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.44$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

| | | | |
|-------------|-------------|-------------|-------------|
| C11A—C10A | 1.7337 (13) | C11B—C14B | 1.7343 (14) |
| C12A—C14A | 1.7286 (13) | C12B—C10B | 1.7321 (13) |
| S1A—C8A | 1.6611 (12) | S1B—C8B | 1.6634 (12) |
| O1A—C1A | 1.2196 (15) | O1B—C1B | 1.2193 (15) |
| N1A—C1A | 1.3816 (15) | N1B—C1B | 1.3801 (15) |
| N1A—C8A | 1.3874 (15) | N1B—C8B | 1.3839 (15) |
| N2A—C8A | 1.3409 (15) | N2B—C8B | 1.3399 (15) |
| N2A—C9A | 1.4184 (15) | N2B—C9B | 1.4181 (15) |
| C1A—N1A—C8A | 127.52 (10) | C1B—N1B—C8B | 127.64 (10) |
| C8A—N2A—C9A | 124.10 (10) | C8B—N2B—C9B | 122.80 (10) |
| N1A—C1A—C2A | 115.67 (10) | N1B—C1B—C2B | 116.12 (10) |
| N2A—C8A—N1A | 115.49 (10) | N2B—C8B—N1B | 116.03 (10) |

Table 2

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|----------------------|-------|-------------|-------------|---------------|
| N1A—H1A \cdots S1B | 0.88 | 2.62 | 3.4414 (11) | 155 |
| N2A—H2A \cdots O1A | 0.88 | 1.97 | 2.6269 (14) | 131 |
| N1B—H1B \cdots S1A | 0.88 | 2.71 | 3.4772 (11) | 146 |
| N2B—H2B \cdots O1B | 0.88 | 1.95 | 2.6240 (14) | 132 |

H atoms were included in calculated positions ($C-H = 0.95$, $N-H = 0.88$ Å) and refined as riding, with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C, N)$.

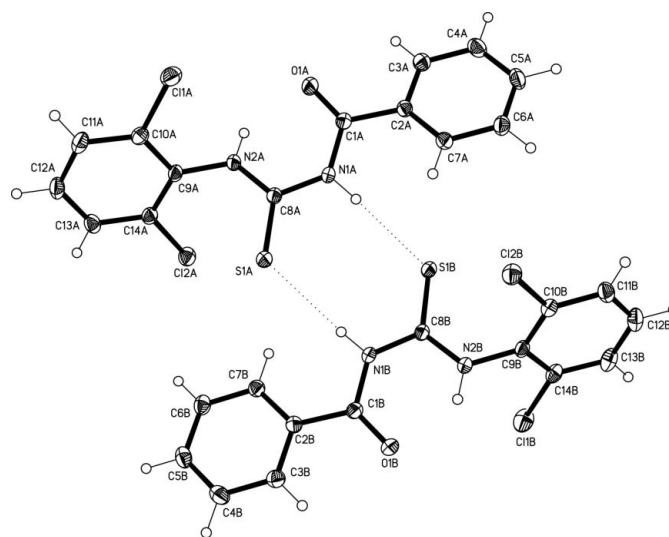


Figure 1

The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level. Dotted lines indicate hydrogen bonds.

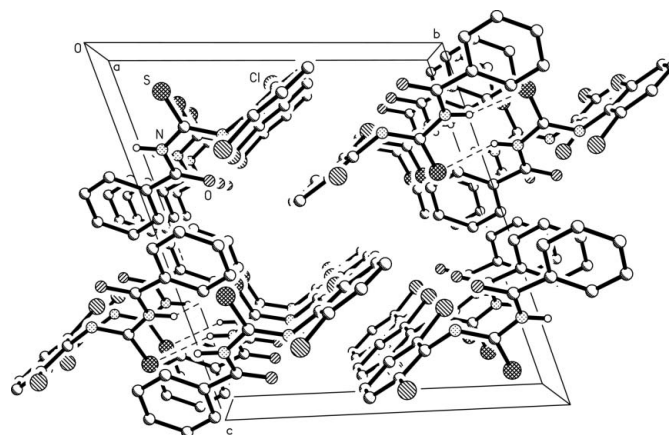


Figure 2

The packing, viewed along [100], with hydrogen bonds indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted.

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.

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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.
- Frech, F. A., Blanz, E. J., Amaral, J. R. D. & French, D. A. (1970). *J. Med. Chem.* **13**, 1117–1124.
- Madan, V. K. & Taneja, A. D. (1991). *J. Indian Chem. Soc.* **68**, 471–472.
- Schuster, M., Kugler, B. & Konig, K. H. (1990). *J. Anal. Chem.* **338**, 717–720.
- Yamin, B. M. & Yusof, M. S. M. (2003). *Acta Cryst.* **E59**, o151–o152.