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N-(3,4-Dichlorophenyl)-*N*′-(3-nitrobenzoyl)thiourea

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Key indicators

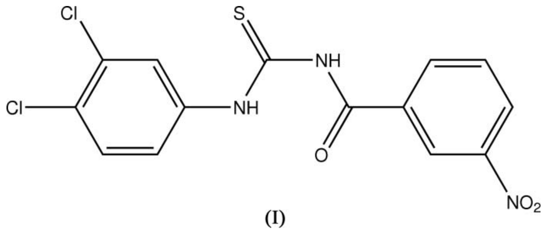
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.105
Data-to-parameter ratio = 14.3

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

In the title molecule, $C_{14}H_9N_3O_3Cl_2S$, the dihedral angles between the mean plane of the central carbonylthiourea group, N_2CSCO, and the mean planes of the 3-nitrobenzoyl and 3,4-dichlorophenyl groups are 22.82 (9) and 69.75 (11)°, respectively. The crystal packing is stabilized by weak intermolecular C—H···O hydrogen bonds, forming a one-dimensional chain along the *b* axis.

Comment

The title compound, (I), is analogous to *N*-(4-chloro-3-nitrophenyl)-*N*′-(3-nitrobenzoyl)thiourea, (II) (Yusof *et al.*, 2006), except that the bulky nitro group in the disubstituted benzene ring is replaced by a Cl atom (Fig.1). The molecule maintains its *trans–cis* configuration with respect to the position of the 3-nitrobenzoyl and 3,4-dichlorophenyl groups relative to the thiono S1 atom across the C8—N2 and C8—N3 bonds, respectively. The bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and are comparable to those in (II). The central carbonylthiourea (S1/C8/N2/N3/C7/O3), 3-nitrophenyl (C1–C6/N1/O1/O2) and 3,4-dichlorophenyl (C9–C14/Cl1/Cl2) fragments are all planar, with a maximum deviation of 0.107 (3)Å for atom O2. The mean plane of the central carbonylthiourea group, N_2CSCO, makes dihedral angles of 22.82 (9) and 69.75 (11)°, respectively, with the mean planes of the 3-nitrobenzoyl and 3,4-dichlorophenyl groups. The dihedral angle between the two aromatic rings is 48.25 (10)°.



(I)

There are two intramolecular hydrogen bonds, N3—H3···O3 and C14—H14···S1 (Table 2), forming two pseudo-six-membered rings, *viz.* N3—H3···O3—C7—N2—C8—N3 and C14—H14···S1—C8—N3—C9—C14. In the crystal structure, the molecules are linked by intermolecular interactions, C1—H1*A*···O2^{i} (symmetry code as in Table 2), forming a one-dimensional chain along the *b* axis (Fig. 2).

Experimental

An equimolar amount of 3,4-dichloroaniline (1.78 g, 11 mmol) in 20 ml acetone was added dropwise to a stirred acetone solution (75 ml) containing 3-nitrobenzoyl chloride (2.0 g, 11 mmol) and

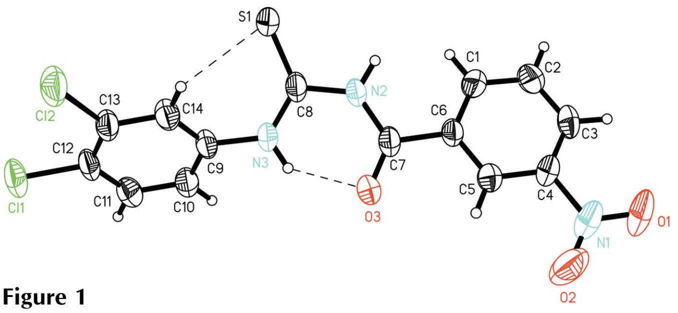


Figure 1
The molecular structure of the title compound, (I), shown with 50% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines.

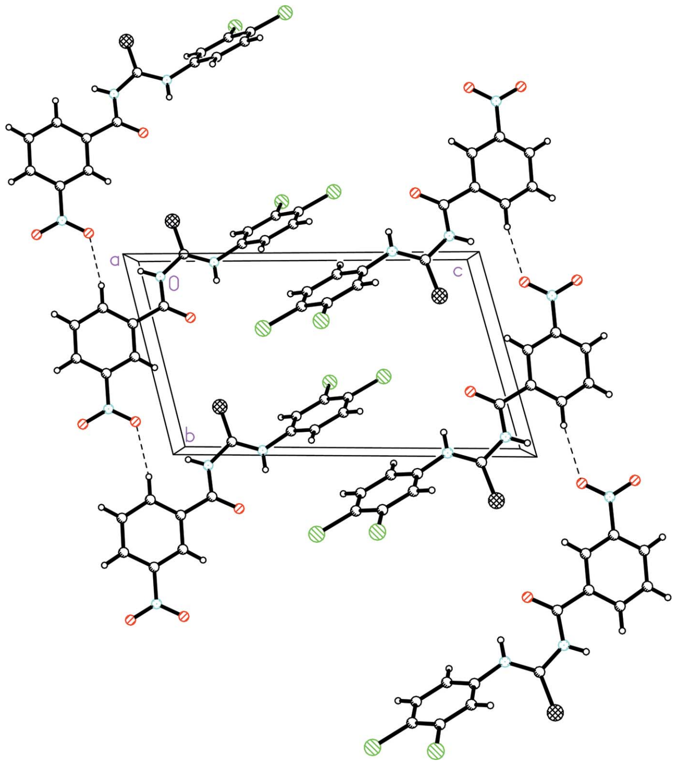


Figure 2
Packing diagram for compound (I), viewed down the *a* axis. Dashed lines denote C—H···O hydrogen bonds.

ammonium thiocyanate (0.82 g, 11 mmol). The solution mixture was refluxed for 1 h. The resulting solution was poured into a beaker containing some ice blocks. The light-yellow precipitate was filtered off and washed with distilled water and cold ethanol before being dried under vacuum. Good quality crystals were obtained by recrystallization from methanol (yield 67%, 2.73 g; m.p. 489.3–491.7 K).

Crystal data

$C_{14}H_9Cl_2N_3O_3S$
$M_r = 370.20$
Triclinic, $P\overline{1}$
$a = 7.1648$ (15) Å
$b = 8.1522$ (17) Å
$c = 13.866$ (3) Å
$\alpha = 73.990$ (3)°
$\beta = 81.912$ (3)°
$\gamma = 79.552$ (4)°
$V = 762.0$ (3) Å^3
$Z = 2$
$D_x = 1.613$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.58$ mm^{-1}
$T = 293$ (2) K
Slab, light yellow
0.50 × 0.22 × 0.08 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.760$, $T_{max} = 0.955$
7867 measured reflections
2964 independent reflections
2400 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 26.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.105$
$S = 1.04$
2964 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.2184P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C12	1.730 (2)	S1—C8	1.648 (2)
Cl2—C13	1.735 (2)	N2—C8	1.397 (3)
N2—C7—C6	116.1 (2)	N3—C8—S1	126.61 (16)
N3—C8—N2	114.6 (2)	N2—C8—S1	118.80 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O3	0.86	1.94	2.648 (3)	138
C14—H14···S1	0.93	2.83	3.225 (2)	107
C1—H1A···O2^i	0.93	2.42	3.322 (4)	165

Symmetry code: (i) $x, y - 1, z$.

After their location in a difference map, all H atoms were positioned geometrically, with N—H = 0.86 Å and C—H = 0.93–0.96 Å, and constrained to ride on their parent atoms, with U_{iso}(H)= xU_{eq}(C,N), where $x = 1.5$ for methyl H and 1.2 for all other H atoms.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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