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N-(2-Chlorobenzoyl)-N′-(4-nitrophenyl)thiourea

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.100
Data-to-parameter ratio = 15.4

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

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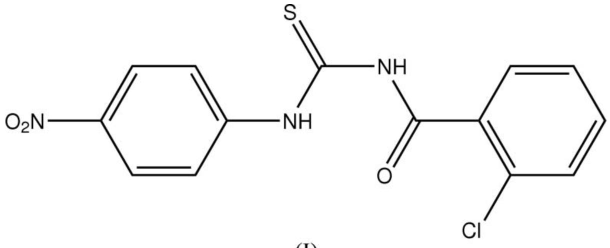
The title compound, $C_{14}H_{10}ClN_3O_3S$, adopts a *trans–cis* configuration for the positions of the 2-chlorobenzoyl and 4-nitrophenyl groups with respect to the thione S atom, across the thiourea C—N bonds. The two aromatic rings are nearly coplanar. The molecule is stabilized by intermolecular hydrogen bonds, forming a three-dimensional network.

Comment

Thiourea derivatives receive considerable attention because of their potential applications in materials science (Wei *et al.*, 2004) and their biological activities (Baruah *et al.*, 2002).



(I)

The title compound, (I), is analogous with *N*-(4-methylbenzoyl)-*N*′-(4-nitrophenyl)thiourea, (II) (Yusof *et al.*, 2006), with the methyl group replaced by a Cl atom in the 2-position of the benzene ring (Fig. 1). The bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and comparable with those in (II). The molecule adopts a *trans–cis* configuration for the positions of the 2-chlorobenzoyl and 4-nitrophenyl groups, respectively, with respect to the thiono S atom, across the thiourea C—N bonds. The central carbonylthiourea (S1/N1/N2/C7/C8) fragment is planar with a maximum deviation of 0.021 (2) Å for atom N1 from the least-squares plane. The central carbonylthiourea fragment makes dihedral angles of 66.95 (10) and 64.38 (9)° with the benzene rings C1–C6 and C9–C14, respectively. The two benzene rings are nearly coplanar, the dihedral angle between them being 3.89 (11)°.

There is one intramolecular hydrogen bond, N2—H2···O1 (Table 1), forming a pseudo-six-membered ring (O1/H2/N2/C8/N1/C7). In the crystal structure, the molecules are linked by intermolecular N—H···S, N—H···O and C—H···O interactions, forming a three-dimensional network (Fig.2).

Experimental

To a stirred acetone solution (75 ml) of 2-chlorobenzoyl chloride (2.0 g, 11 mmol) and ammonium thiocyanate (0.87 g, 11 mmol), 4-nitrophenylamine (1.58 g, 11 mmol) in 30 ml of acetone was added dropwise. The solution 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 78% (2.87 g); m.p. 478.57–480.74 K.

Crystal data

$C_{14}H_{10}ClN_3O_3S$
$M_r = 335.76$
Triclinic, $P\bar{1}$
$a = 5.9832\ (13)$ Å
$b = 9.783\ (2)$ Å
$c = 13.274\ (3)$ Å
$\alpha = 90.045\ (4)^\circ$
$\beta = 100.432\ (4)^\circ$
$\gamma = 102.271\ (4)^\circ$
$V = 746.0\ (3)$ Å^3
$Z = 2$
$D_x = 1.495$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.41$ mm^{-1}
$T = 298\ (2)$ K
Slab, light yellow
0.40 × 0.38 × 0.06 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.853$, $T_{\max} = 0.976$
7979 measured reflections
3071 independent reflections
2370 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.034$
$\theta_{\max} = 26.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.100$
$S = 1.03$
3071 reflections
199 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2 + 0.1293P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.20$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.86	2.03	2.704 (2)	134
N1—H1···S1$^{\rm i}$	0.86	2.67	3.4014 (19)	143
N2—H2···O1$^{\rm ii}$	0.86	2.27	3.000 (2)	143
C3—H3···O3$^{\rm iii}$	0.93	2.60	3.298 (4)	132
C11—H11···O2$^{\rm iv}$	0.93	2.43	3.274 (3)	151

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $-x, -y, -z+2$; (iii) $x+2, y, z+1$; (iv) $-x-1, -y, -z+1$.

After their location in a difference Fourier map, all H atoms were positioned geometrically and allowed to ride on the parent C or N atoms with C—H = 0.93 Å and N—H = 0.86 Å and with $U_{\rm iso}$(H)= $1.2U_{\rm eq}$(C,N).

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).

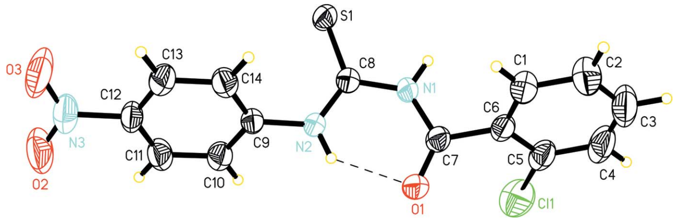


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. The dashed line indicates a hydrogen bond.

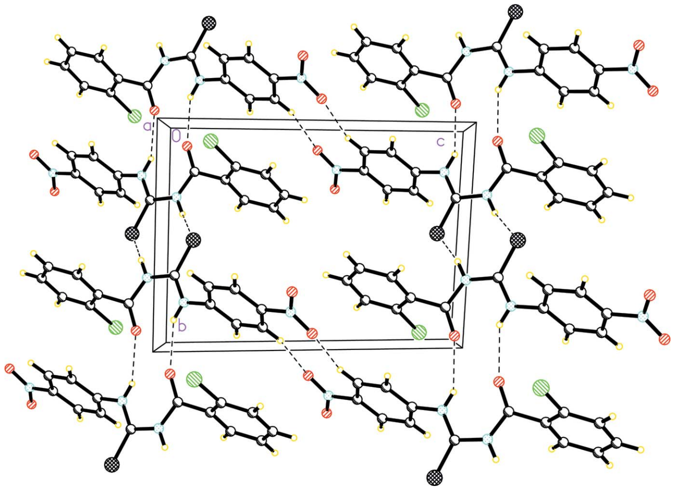


Figure 2
Packing diagram of (I), viewed down the *a* axis. The dashed lines denote the N—H···S, N—H···O and C—H···O hydrogen bonds.

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