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1-(3-Chlorobenzoyl)-3-[3-(trifluoromethyl)-phenyl]thiourea

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.057
wR factor = 0.120
Data-to-parameter ratio = 16.7

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

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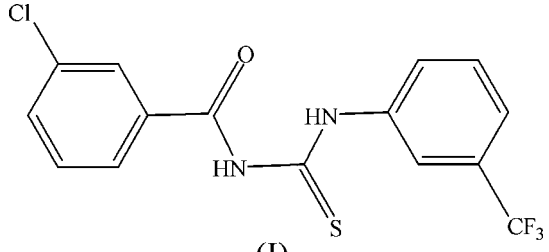
In the crystal structure of the title compound, $C_{15}H_{10}ClF_3N_2OS$, the dihedral angle between the two aromatic rings is 32.68 (8)°. The crystal packing shows intermolecular N—H···O and C—H···S hydrogen bonds which link molecules into sheets which are stacked along [001].

Comment

N-Substituted and *N*,*N*′-disubstituted thiourea derivatives are very useful building blocks for the synthesis of a wide range of aliphatic macromolecular and heterocyclic compounds. Thus, benzothiazoles have been prepared from arylthioureas in the presence of bromine (Patil & Chedekel, 1984), and the condensation of thiourea with α-halocarbonyl compounds forms 2-aminothiazoles (Baily *et al.*, 1996). 2-Methyl-aminothiazolines have been synthesized by cyclization of *N*-(2-hydroxyethyl)-*N*′-methylthioureas (Namgun *et al.*, 2001). Thioureas are efficient guanylating agents (Maryanoff *et al.*, 1986). *N*,*N*-Dialkyl-*N*-aroylthioureas have been efficiently used for the extraction of nickel, palladium and platinum metals (Koch, 2001). Aliphatic and acylthioureas are well known for their fungicidal, antiviral, pesticidal and plant-growth regulating activities (Upadlgaya & Srivastava, 1982; Wegner *et al.*, 1986). Symmetrical and unsymmetrical thioureas have shown antifungal activity against the plant pathogens *Pyricularia oryzae* and *Drechslera oryzae* (Krishnamurthy *et al.*, 1999). We became interested in the synthesis of these thioureas as intermediates in the synthesis of novel guanidines and heterocyclic compounds for the systematic study of bioactivity and complexation behaviour and we present here the crystal structure of the title compound, (I).



(I)

Compound I (Fig. 1) shows the typical thiourea C=S and C=O double bonds, as well as shortened C—N bonds (Table 1). The thiocarbonyl and carbonyl groups are almost coplanar, as reflected by the torsion angles C8—N2—C9—O1 = 10.6 (5)° and C9—N2—C8—N1 = −18.1 (5)°. This is asso-

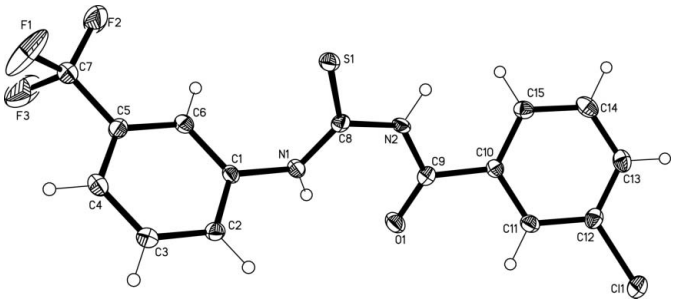


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

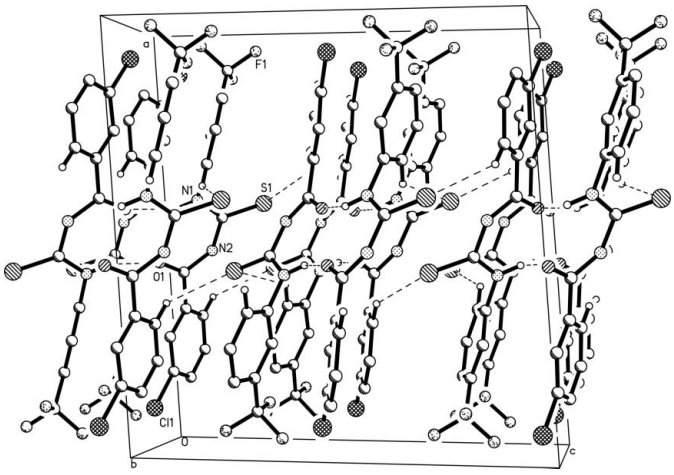


Figure 2
The crystal packing of (I), viewed along [010], with hydrogen bonds indicated as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

ciated with the expected typical thiourea intramolecular N1—H1*A*···O1 hydrogen bond (Table 2). The dihedral angle formed by the two benzene ring planes, C1–C6 and C10–C15, is 32.68 (8)°. Other geometric parameters present no unusual features (Khawar Rauf, Badshah & Bolte, 2006; Khawar Rauf, Badshah & Flörke, 2006).

The crystal packing of (I) shows intermolecular N—H···O and C—H···S hydrogen bonds (Table 2, Fig. 2) which link the molecules into sheets stacked along [001]. The Cl atoms and N2—H group are not involved in any hydrogen bonds.

Experimental

Freshly prepared 3-chlorobenzoyl chloride (1.75 g, 10 m mol) was added to a suspension of KSCN ((1.00 g, 10 mmol) in acetone (30 ml). The reaction mixture was stirred for 15 min. Neat 3-(trifluoromethyl)aniline (1.6 g, 10 mmol) was then added and the resulting mixture was stirred for 1 h. The reaction mixture was then poured into acidified water (500 ml) and stirred well. The solid product was separated, washed with deionized water and purified by recrystallization from methanol–dichloromethane (1:1) to give fine crystals of (I), with an overall yield of 85%. The full spectroscopic and physical characterization of the compound will be reported elsewhere.

Crystal data

$C_{15}H_{10}ClF_3N_2OS$
M_r = 358.76
Monoclinic, $P2_1/c$
a = 15.249 (4) Å
b = 6.5459 (16) Å
c = 14.643 (4) Å
β = 94.066 (5)°
V = 1457.9 (7) Å^3
Z = 4
D_x = 1.634 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.44 mm^{-1}
T = 120 (2) K
Plate, colourless
0.48 × 0.22 × 0.03 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.816, T_{max} = 0.987
9777 measured reflections
3468 independent reflections
1968 reflections with $I > 2\sigma(I)$
R_{int} = 0.094
θ_{max} = 27.9°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.121
S = 0.89
3468 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0464P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.94 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C12	1.736 (3)	N1—C1	1.438 (4)
S1—C8	1.667 (3)	N2—C9	1.375 (4)
O1—C9	1.217 (4)	N2—C8	1.392 (4)
N1—C8	1.326 (4)	C9—C10	1.488 (4)
C8—N1—C1	121.3 (3)	N2—C8—S1	117.5 (2)
C9—N2—C8	128.1 (3)	O1—C9—N2	122.3 (3)
N1—C8—N2	116.8 (3)	O1—C9—C10	123.1 (3)
N1—C8—S1	125.7 (2)	N2—C9—C10	114.5 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1	0.88	2.05	2.683 (3)	128
N1—H1*A*···O1^{i}	0.88	2.53	3.199 (3)	133
C2—H2*A*···S1^{ii}	0.95	2.93	3.599 (3)	128
C15—H15*A*···S1^{iii}	0.95	2.84	3.666 (3)	146

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

H atoms were located in a difference Fourier map and refined in idealized positions riding on their parent C and N atoms, with C—H = 0.95 Å and N—H = 0.88 Å, and with isotropic displacement parameters U_{iso}(H) = 1.2U_{eq}(C,N). The anisotropic displacement parameters of the trifluoromethyl group indicate some degree of disorder (rotation along the C5—C7 axis) but this could not be resolved.

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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