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N-(3-Flurobenzoyl)-*N*′-phenylthiourea

You-Ming Zhang, Jing-Dong Qin, Qi Lin and Tai-Bao Wei*

College of Chemistry and Chemical Engineering, Gansu Key Laboratory of Polymer Materials, Northwest Normal University, Lanzhou, Gansu 730070, People's Republic of China

Correspondence e-mail: weitaibao@126.com

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.002 Å
R factor = 0.033
wR factor = 0.095
Data-to-parameter ratio = 14.2

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

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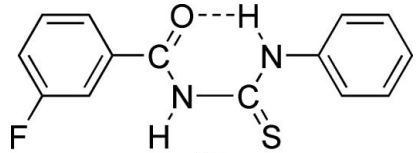
In the crystal structure of the title compound, $C_{14}H_{11}FN_2OS$, the molecules form centrosymmetric hydrogen-bonded dimers, with the S atom forming an intermolecular hydrogen bond with the H atom of an NH group.

Comment

Acylthiourea derivatives have been patented as antidiabetic (Sohda *et al.*, 1990), anti-arthritic (Missbach, 1995), anti-neoplastic (Hwang *et al.*, 1995) and anticoagulant (Bisacchi *et al.*, 2000) agents, and for the treatment of cognitive problems (Wu *et al.*, 2002) and prostate disorders (Holt, 1995). Herbicidal (Hackmann, 1960), fungicidal (Hackmann, 1960), bactericidal (Huang *et al.*, 1995), insecticidal (Joos & Wirtz, 1972) and plant growth regulator activities (Wei *et al.*, 1992) have also been reported. In addition, N-substituted *N*′-carbonylthiourea compounds have attracted considerable attention in recent years, due to their coordination ability with transition metal ions (Shen *et al.*, 1999). Owing to their strong coordination ability, many acylthiourea derivatives are extensively utilized as sequestering agents for copper sulfides and precious metals (Fairthorne *et al.*, 1997). Biological activity is closely related to structure, and in continuation of our previous work on the coordination behaviour, synthesis and biological activities of acylthiourea derivatives (Zhang *et al.*, 2000, 2001, 2003, 2006), we have determined the structure of the title thiourea derivative, (I).



(I)

The structure of compound (I), with the atomic numbering scheme and the intra- and intermolecular hydrogen bonding, is shown in Fig. 1. Between the H atom attached to N2 and atom O1 an intramolecular hydrogen bond is found (Table 1). From this arrangement an almost planar six-membered ring is formed, in which the mean deviation of the non-H atoms is 0.0111 (2) Å.

The 3-flurobenzoyl ring and the benzene ring have different orientations with respect to the thiourea group, with torsion angles C8—N1—C7—C4 = 176.4 (1)° and N1—C8—N2—C9 = −176.9 (1)°, respectively. The torsion angle C7—N1—C8—S1 is −176.7 (1)°.

In the crystal structure, each pair of molecules is connected into a dimer by two intermolecular N—H···S hydrogen bonds.

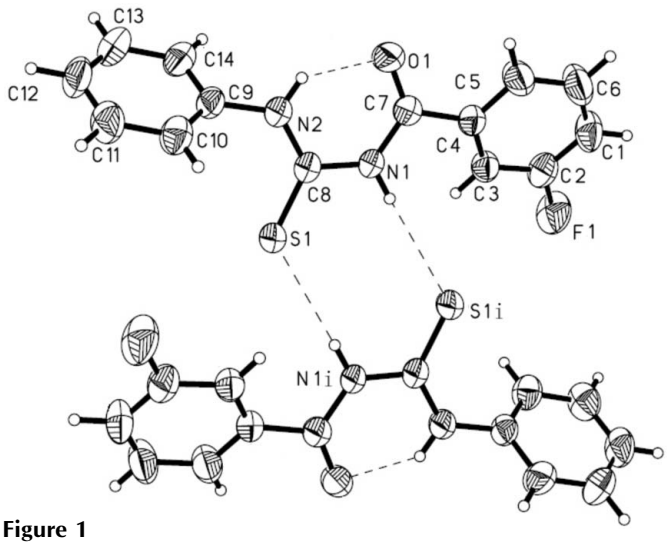


Figure 1
The crystal structure of the title compound, with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonding is shown as dashed lines. [Symmetry code: (i) $1-x$, $-y$, $-z$.]

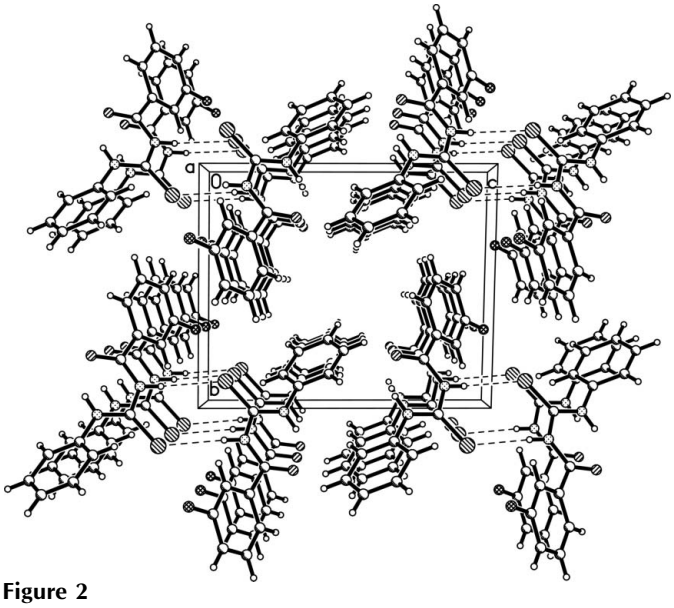


Figure 2
A packing diagram for (I), projected in the direction of the *a* axis. Dashed lines indicate hydrogen bonds

The N1—H···S1^{i} separation is 2.64 Å and the N1—H···S1 angle is 173° [symmetry code: (i) $1-x$, $-y$, $-z$] (Table 1 and Fig. 1).

Experimental

The synthesis of (I) was carried out by adding NH_4SCN (13 mmol) and polyethyleneglycol-400 (0.1 ml) as phase-transfer catalyst to a solution of 3-fluorobenzoyl chloride (10 mmol) in dry ethyl acetate (10 ml), followed by stirring at room temperature for 1 h. After filtration to remove the inorganic solid, aniline (10 mmol) was slowly added to the reaction mixture with constant stirring. The reaction mixture was then again stirred at room temperature for 30 min. After evaporating the ethyl acetate, a crude product was obtained, which was recrystallized from ethanol. Colourless single crystals of (I) were obtained by slow evaporation of an EtOAc solution of compound (I) for about one week.

Crystal data

$C_{14}H_{11}FN_2OS$
M_r = 274.31
Triclinic, $P\overline{1}$
a = 5.6511 (4) Å
b = 10.0628 (7) Å
c = 12.0169 (9) Å
α = 90.769 (3)°
β = 91.033 (3)°
γ = 98.626 (3)°
V = 675.42 (8) Å^3
Z = 2
D_x = 1.349 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 294 (2) K
Block, colourless
0.59 × 0.51 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
3655 measured reflections
2464 independent reflections
2213 reflections with $I > 2\sigma(I)$
R_{int} = 0.011
θ_{max} = 25.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.095
S = 0.99
2464 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.1964P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.034 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.86	1.93	2.6239 (17)	137
N1—H1···S1^{i}	0.86	2.64	3.4989 (12)	173

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

All H atoms were positioned with idealized geometry, with C—H distances of 0.93 Å and N—H distances of 0.86 Å, and were refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(N) or 1.5U_{eq}(C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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