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1-(4-Fluorophenyl)thiourea

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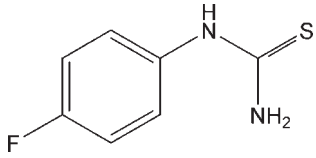
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.097; data-to-parameter ratio = 18.0.

In the title compound, $C_7H_7FN_2S$, the aromatic ring plane and the thiourea unit are twisted with a torsion angle C—C—N—C7 of 44.6 (2)°. In the crystal, N—H···S and N—H···F intermolecular hydrogen bonds link the molecules into infinite sheets that are stacked along the c axis.

Related literature

For the biological activity of fluorinated thioureas, see: Sun *et al.* (2006); Saeed *et al.* (2009); Xu *et al.* (2003). For the use of fluorinated thioureas in organic synthesis, see: Nosova *et al.* (2006, 2007); Lipunova *et al.* (2008); Berkessel *et al.* (2006). *N*′-(2-fluorobenzoyl)thiourea derivatives are suitable substrates for studying intramolecular hydrogen bonds and Fermi resonance, see: Hritzová & Koščík (2008).



Experimental

Crystal data

$C_7H_7FN_2S$
M_r = 170.21
Monoclinic, $P2_1/c$
a = 9.1384 (8) Å
b = 8.4338 (7) Å
c = 10.5334 (9) Å
β = 109.796 (2)°
V = 763.85 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 120 K
0.43 × 0.39 × 0.29 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.857, T_{max} = 0.900
6816 measured reflections
1814 independent reflections
1645 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.097
S = 1.05
1814 reflections
101 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···S1^{i}	0.88	2.43	3.2841 (12)	163
N2—H2B···F1^{ii}	0.88	2.30	3.0989 (15)	152

Symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, -y+1, -z+2$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PB2028).

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S1. Comment

Fluorinated thioureas are an imoprtant class of thioureas. Theser are versatile synthons for various fluorine-containing heterocycles: [1,3]-benzothiazin-4-ones (Nosova *et al.*,2006, 2007) 1-aryl-2-ethylthio-quinazolin-4-one, thiazolidine and 1*H*-1,2,4-triazoles (Lipunova *et al.*, 2008). Fluorinated thioureas exhibit a variety of biological activities: potent influenza virus neuraminidase inhibitors (Sun *et al.*, 2006), antimicrobial (Saeed *et al.*, 2009) and insecticidal activities (Xu *et al.*, 2003). Moreover, fluorinated bis-thiourea derivatives are also used as organocatalyst in Morita-Baylis-Hillman reaction (Berkessel *et al.*, 2006) and N-Substituted *N*′-(2-fluorobenzoyl)thiourea derivatives are suitable substrates for studying Intramolecular Hydrogen Bonds and Fermi Resonance (Hritzová & Koščík 2008). The aromatic ring plane and the thio-urea moiety are twisted with a torsion angle C2–C1–N1–C7 of 44.6 (2)°. N(1)–H···S and N(2)–H···F intermolecular hydrogen bonds link molecules to endless 2D sheets that are stacked along the c axis.

S2. Experimental

4-Fluorobenzoylisothiocyante (1 mmol) in acetone was treated with ammonia (1 mmol) under a nitrogen atmosphere at reflux for 3 h. Upon cooling, the reaction mixture was poured into aq HCl and the precipitated product was rerystallized from in methanol to afforded the title compound (78 %) as colourless crystals: Anal. calcd. for $C_7H_7N_2O_2FS$: C, 49.40; H, 4.15; N, 16.46; S, 18.84%; found: C, 49.02; H, 4.17; N, 16.41; S, 18.91%.

S3. Refinement

Hydrogen atoms were clearly identified in difference Fourier syntheses, idealized and refined at calculated positions riding on the carbon atoms with isotropic displacement parameters $U_{iso}(H) = 1.2U(C/N_{eq})$.

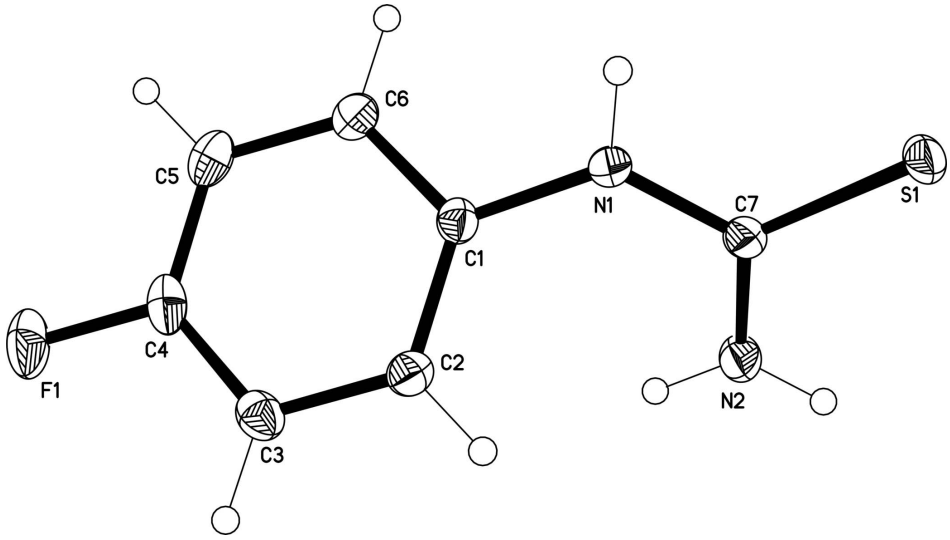

Figure 1

Molecular structure of title compound. Displacement ellipsoids are drawn at the 50% probability level.

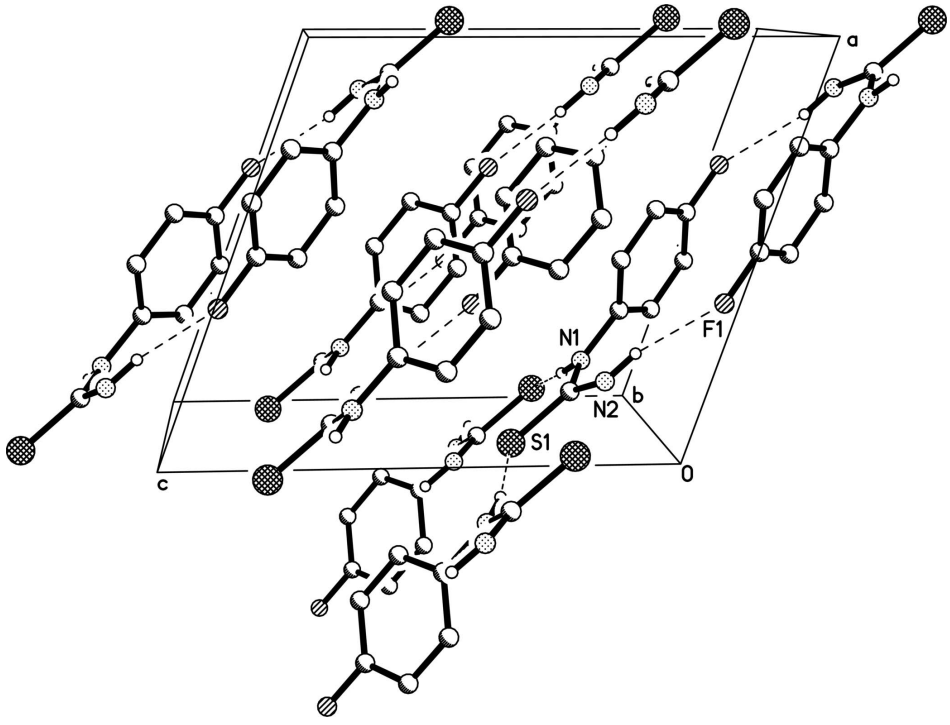

Figure 2

Crystal packing viewed along [001] with intermolecular hydrogen bonds indicated as dashed lines. H-atoms not involved in hydrogen bonding are omitted.

1-(4-Fluorophenyl)thiourea

Crystal data

$C_7H_7FN_2S$
M_r = 170.21

Monoclinic, $P2_1/c$
a = 9.1384 (8) Å

b = 8.4338 (7) Å
c = 10.5334 (9) Å
β = 109.796 (2)°
V = 763.85 (11) Å^3
Z = 4
F(000) = 352
D_x = 1.480 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3596 reflections
θ = 3.2–28.3°
μ = 0.37 mm^{-1}
T = 120 K
Block, colourless
0.43 × 0.39 × 0.29 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.857, T_{max} = 0.900

6816 measured reflections
1814 independent reflections
1645 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.9°, θ_{min} = 2.4°
h = −12→12
k = −10→11
l = −13→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.097
S = 1.05
1814 reflections
101 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 0.3192P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.009 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	1.02935 (4)	0.53586 (4)	0.71645 (4)	0.02753 (15)
F1	0.35073 (11)	0.23014 (12)	0.98827 (10)	0.0374 (3)
N1	0.84161 (13)	0.39046 (13)	0.81980 (12)	0.0224 (3)
H1A	0.8874	0.3066	0.8006	0.027*
N2	0.85423 (14)	0.66261 (14)	0.84570 (13)	0.0257 (3)
H2B	0.7864	0.6574	0.8882	0.031*
H2C	0.8920	0.7550	0.8332	0.031*
C1	0.71613 (14)	0.35888 (15)	0.86719 (13)	0.0195 (3)

C2	0.57627 (15)	0.44244 (16)	0.81997 (14)	0.0214 (3)
H2A	0.5656	0.5286	0.7594	0.026*
C3	0.45251 (16)	0.39984 (17)	0.86130 (14)	0.0247 (3)
H3A	0.3572	0.4568	0.8308	0.030*
C4	0.47151 (16)	0.27309 (18)	0.94755 (14)	0.0254 (3)
C5	0.60776 (17)	0.18705 (17)	0.99488 (14)	0.0261 (3)
H5A	0.6167	0.0995	1.0538	0.031*
C6	0.73102 (16)	0.23144 (17)	0.95434 (14)	0.0233 (3)
H6A	0.8262	0.1745	0.9863	0.028*
C7	0.89954 (15)	0.53115 (15)	0.80043 (14)	0.0206 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0258 (2)	0.0168 (2)	0.0483 (3)	−0.00188 (12)	0.02351 (17)	−0.00191 (14)
F1	0.0391 (5)	0.0376 (5)	0.0475 (6)	−0.0133 (4)	0.0304 (4)	−0.0076 (4)
N1	0.0228 (5)	0.0154 (5)	0.0330 (6)	0.0021 (4)	0.0145 (5)	0.0018 (4)
N2	0.0267 (6)	0.0174 (6)	0.0386 (7)	−0.0017 (4)	0.0184 (5)	−0.0025 (5)
C1	0.0198 (6)	0.0188 (6)	0.0207 (6)	−0.0022 (5)	0.0080 (5)	−0.0014 (5)
C2	0.0233 (6)	0.0190 (6)	0.0222 (6)	0.0008 (5)	0.0080 (5)	0.0007 (5)
C3	0.0218 (6)	0.0250 (7)	0.0284 (7)	−0.0005 (5)	0.0101 (5)	−0.0055 (5)
C4	0.0272 (7)	0.0276 (7)	0.0262 (7)	−0.0106 (5)	0.0154 (5)	−0.0091 (5)
C5	0.0353 (7)	0.0230 (7)	0.0207 (6)	−0.0069 (6)	0.0103 (6)	0.0000 (5)
C6	0.0250 (6)	0.0201 (6)	0.0235 (6)	−0.0010 (5)	0.0066 (5)	0.0013 (5)
C7	0.0164 (6)	0.0189 (6)	0.0261 (7)	0.0003 (4)	0.0066 (5)	0.0014 (5)

Geometric parameters (Å, º)

S1—C7	1.7035 (14)	C1—C2	1.3954 (18)
F1—C4	1.3616 (15)	C2—C3	1.3897 (19)
N1—C7	1.3427 (17)	C2—H2A	0.9500
N1—C1	1.4222 (15)	C3—C4	1.375 (2)
N1—H1A	0.8800	C3—H3A	0.9500
N2—C7	1.3274 (17)	C4—C5	1.380 (2)
N2—H2B	0.8800	C5—C6	1.3846 (19)
N2—H2C	0.8800	C5—H5A	0.9500
C1—C6	1.3899 (19)	C6—H6A	0.9500
C7—N1—C1	128.69 (11)	C2—C3—H3A	120.9
C7—N1—H1A	115.7	F1—C4—C3	118.71 (13)
C1—N1—H1A	115.7	F1—C4—C5	118.34 (13)
C7—N2—H2B	120.0	C3—C4—C5	122.95 (13)
C7—N2—H2C	120.0	C4—C5—C6	118.37 (13)
H2B—N2—H2C	120.0	C4—C5—H5A	120.8
C6—C1—C2	119.93 (12)	C6—C5—H5A	120.8
C6—C1—N1	117.80 (12)	C5—C6—C1	120.32 (13)
C2—C1—N1	122.03 (12)	C5—C6—H6A	119.8
C3—C2—C1	120.14 (13)	C1—C6—H6A	119.8

C3—C2—H2A	119.9	N2—C7—N1	119.76 (12)
C1—C2—H2A	119.9	N2—C7—S1	121.59 (10)
C4—C3—C2	118.28 (13)	N1—C7—S1	118.64 (10)
C4—C3—H3A	120.9		
C7—N1—C1—C6	−141.01 (15)	F1—C4—C5—C6	−179.50 (12)
C7—N1—C1—C2	44.6 (2)	C3—C4—C5—C6	0.5 (2)
C6—C1—C2—C3	0.9 (2)	C4—C5—C6—C1	−0.5 (2)
N1—C1—C2—C3	175.14 (12)	C2—C1—C6—C5	−0.1 (2)
C1—C2—C3—C4	−0.9 (2)	N1—C1—C6—C5	−174.66 (12)
C2—C3—C4—F1	−179.79 (12)	C1—N1—C7—N2	10.3 (2)
C2—C3—C4—C5	0.2 (2)	C1—N1—C7—S1	−169.25 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···S1^{i}	0.88	2.43	3.2841 (12)	163
N2—H2*B*···F1^{ii}	0.88	2.30	3.0989 (15)	152

Symmetry codes: (i) −*x*+2, *y*−1/2, −*z*+3/2; (ii) −*x*+1, −*y*+1, −*z*+2.