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3-(3-Fluorobenzyl)-2-thioxo-2,3,5,6-tetrahydropyrimidin-4(1*H*)-one

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.038
wR factor = 0.103
Data-to-parameter ratio = 12.4

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

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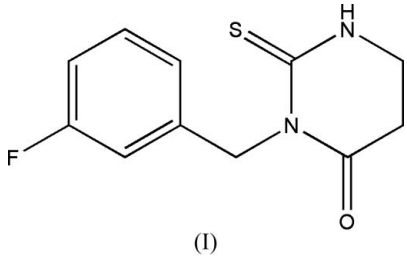
In the title molecule, $C_{11}H_{11}FN_2OS$, conformational disorder is observed in the tetrahydropyrimidine ring and both components adopt half-chair conformations. In the crystal structure, the molecules are linked by C—H···O and N—H···S hydrogen bonds, forming a sheet-like structure parallel to the *ab* plane.

Comment

Derivatives of uracil and thiouracil are very attractive for their varied bioactivity (Gupta *et al.*, 2004; South *et al.*, 2003). For example, lenacil, bromacil, butafenacil, flupropacil, isocil and terbacil are widely used herbicides, while some have been shown to possess antidiabetic activity (Soliman, 1979). Compounds that contain fluorine have special bioactivity; for example, flumioxazin is a widely used herbicide (Hermann *et al.*, 2003; Ulrich, 2004). This led us to study the synthesis and structure of these compounds. To further investigate the relationship between the structure and herbicidal activity, we have synthesized a series of derivatives of uracil and thiouracil containing fluorine. We report here the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. Both the major and minor conformers of the tetrahydropyrimidine ring adopt a half-chair conformation, similar to that observed in related structures (Lorente & Aurrecoechea, 1994; Rohrer & Sundaralingam, 1968; Furberg & Jensen, 1968; Yao *et al.*, 2004*a*,*b*). The conformation of the attachment of the benzene ring to the tetrahydropyrimidine ring is described by the torsion angle C1—N2—C5—C6 of 107.7 (2)°.

In the crystal structure, centrosymmetrically related molecules form dimeric pairs through intermolecular C—H···O hydrogen bonds. Intermolecular N—H···S hydrogen bonds between adjacent dimers generate a sheet-like structure running parallel to the *ab* plane (Fig. 2 and Table 2).

Experimental

According to the reported procedure of Hatam *et al.* (1996), the title compound was synthesized by refluxing methyl 3-({[(3-fluorobenz-

yl)amino]carbonothioyl}amino)propanoate in triethylamine for about 2 h. After cooling, the precipitate was filtered off and recrystallized from a mixture of acetone and ethanol (1:1 *v*/*v*), giving single crystals suitable for X-ray diffraction.

Crystal data

$C_{11}H_{11}FN_2OS$
$M_r = 238.28$
Orthorhombic, *Pbca*
$a = 9.4664\ (18)$ Å
$b = 9.5316\ (17)$ Å
$c = 23.823\ (5)$ Å
$V = 2149.6\ (7)$ Å^3
$Z = 8$
$D_x = 1.473$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.29$ mm^{-1}
$T = 294\ (2)$ K
Block, colourless
0.20 × 0.16 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.944$, $T_{max} = 0.971$
11291 measured reflections
2209 independent reflections
1434 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 26.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.103$
$S = 1.00$
2209 reflections
178 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0417P)^2 + 1.099P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.679 (2)	N2—C4	1.401 (3)
O1—C4	1.205 (3)	N2—C5	1.475 (3)
N2—C1	1.391 (3)	C5—C6	1.505 (3)
C1—N2—C4	123.30 (18)	N2—C1—S1	123.57 (16)
C4—N2—C5	114.88 (17)	N2—C5—C6	112.03 (17)
N1—C1—S1	120.04 (17)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···S1^{i}	0.84 (2)	2.65 (2)	3.428 (2)	155 (2)
C9—H9···O1^{ii}	0.93	2.49	3.369 (3)	158

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

The amine H atom was located in a difference map and refined freely. The remaining H atoms were placed in calculated positions, with C—H = 0.93 or 0.97 Å, and included in the final cycles of refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}$(parent atom). Atoms C2 and C3 are disordered over two sites, with occupation factors of 0.888 (8) and 0.112 (8), respectively. The C2—C3/C2′—C3′, C3—C4/C3′—C4 and C2—N1/C2′—N1 bond lengths were restrained to 1.54 (1), 1.50 (1) and 1.48 (1) Å, respectively. The F atom is also disordered over two positions with site-occupancy factors of 0.947 (4) and 0.053 (4), respectively. The C—F bond lengths were restrained to 1.35 (1) Å. The U^{ij} components of the disordered atoms were restrained to approximate isotropic behaviour.

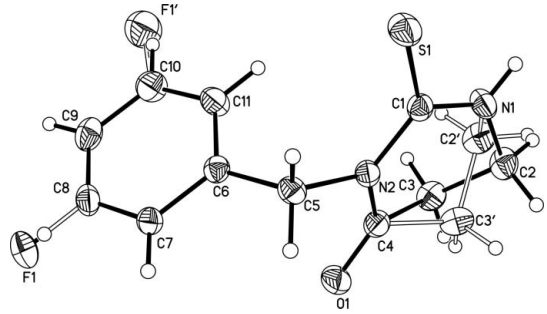


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. All disorder components are shown.

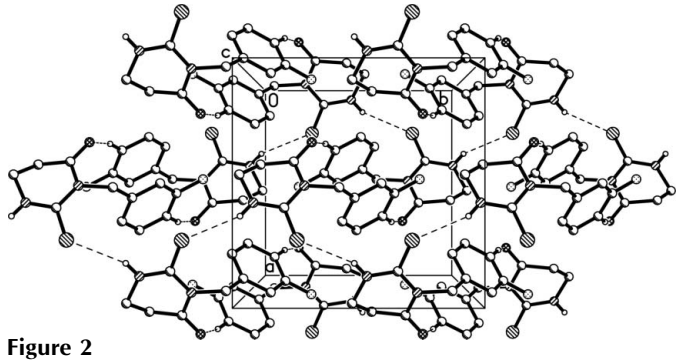


Figure 2
A packing diagram of (I). Intermolecular hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); 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, 1999); software used to prepare material for publication: *SHELXTL*.

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