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3-[(6-Chloropyridin-3-yl)methyl]-2-thioxo-2,3,5,6-tetrahydropyrimidin-4(1*H*)-one

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Key indicators

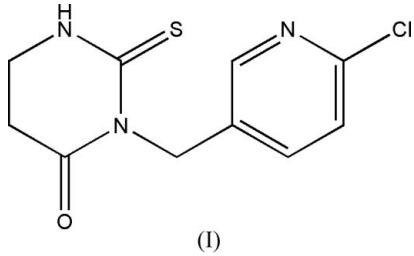
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.049
wR factor = 0.144
Data-to-parameter ratio = 14.8

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

The tetrahydropyrimidine ring of the title molecule, $C_{10}H_{10}ClN_3OS$, adopts a half-chair conformation. In the crystal structure, molecules are linked through N—H···S hydrogen bonding, forming a one-dimensional structure.

Comment

Derivatives of uracil and thiouracil are generally bioactive (Gupta *et al.*, 2004; South *et al.*, 2003). For example, lenacil, bromacil, butafenacil, flupropacil, isocil and terbacil are widely used herbicides, some also having antidiabetic activity (Soliman, 1979). In addition, many compounds incorporating the pyridine ring also show significant bioactivity (Kreutter *et al.*, 2006). This has led us to focus our attention on the synthesis and structure of uracil and thiouracil derivatives that contain pyridine rings. We report here the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The tetrahydropyrimidine ring adopts 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 pyridine ring to the tetrahydropyrimidine ring is described by the C1—N2—C5—C6 torsion angle of 99.9 (3)°. In the crystal structure, molecules are linked through intermolecular N—H···S hydrogen bonds (Fig. 2 and Table 1), forming a one-dimensional structure.

Experimental

The title compound was synthesized according to Hatam *et al.* (1996) by refluxing methyl 3-({[(6-chloropyridin-3-yl)methanamino]-carbonothioyl}amino)propanoate (4.14 mmol) in triethylamine (10 ml) for about 2 h. After cooling, the precipitate was filtered off and recrystallized from a mixture of acetone and ethanol (1:1), which gave single crystals suitable for X-ray diffraction (yield 88%; m.p. 441–443 K). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 2.84 (*t*, *J* = 6.8 Hz, 2H, CH_2CO), 3.51–3.65 (*m*, 2H, CH_2py), 7.25–8.54 (*m*, 3H, py—H), 8.17 (*s*, 1H, NH).

Crystal data

$C_{10}H_{10}ClN_3OS$
$M_r = 255.72$
Monoclinic, $P2_1/n$
$a = 10.131\ (3)$ Å
$b = 5.7977\ (14)$ Å
$c = 20.957\ (5)$ Å
$\beta = 101.562\ (4)^\circ$
$V = 1205.9\ (5)$ Å^3
$Z = 4$
$D_x = 1.408$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.47$ mm^{-1}
$T = 294\ (2)$ K
Block, colorless
$0.24 \times 0.20 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (SABABS; Sheldrick, 1996)
$T_{min} = 0.895$, $T_{max} = 0.954$
6496 measured reflections
2470 independent reflections
1277 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 26.4^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.146$
$S = 1.01$
2470 reflections
167 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2 + 0.3547P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ 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}	0.83 (3)	2.49 (3)	3.315 (3)	169 (3)

Symmetry code: (i) $-x+2, -y+1, -z$.

The N-bound H atom was located in a difference Fourier map and was refined freely. Other H atoms were placed in calculated positions, with C—H = 0.93 or 0.97 Å, and included in the refinement using a riding model, with U_{iso}(H) = $1.2U_{eq}$(parent atom). In the crystal structure, atoms C2 and C3 and their associated H atoms are disordered over two sites, with refined site-occupancy factors of 0.453 (14) and 0.547 (14), respectively. Atoms N3 and C9 and their associated H atoms are also disordered over two sites, with refined site-occupancy factors of 0.47 (3) and 0.53 (3), respectively. Atoms N3 and C9′, and N3′ and C9 were set to have the same positional parameters and anisotropic displacement parameters. The C2—C3 and C2′—C3′ bond lengths were restrained to 1.52 (1) Å. Atom C3 was restrained with an effective standard deviation of 0.01 so that its U^{ij} components approximate to isotropic behavior.

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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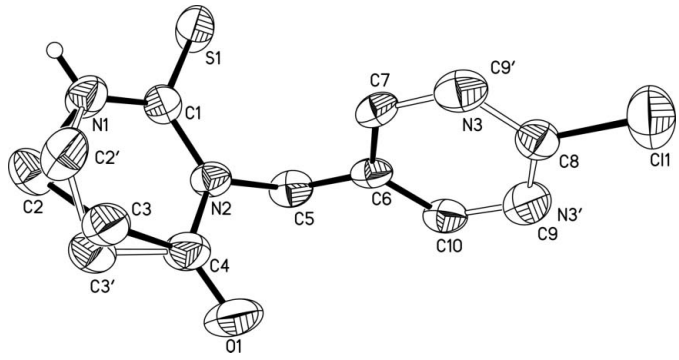

Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

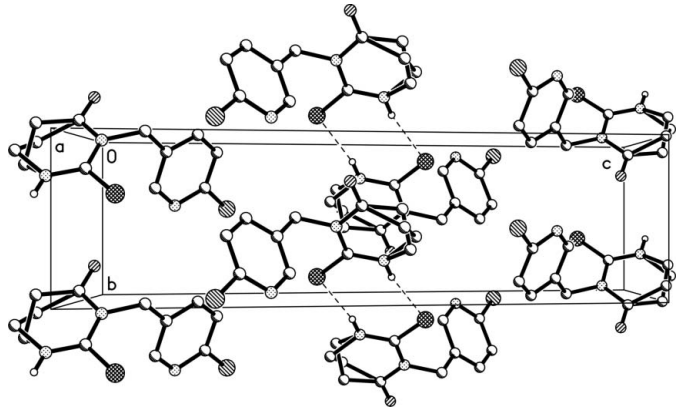

Figure 2
A packing diagram of (I). Intermolecular hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted. Both disorder components are shown. H atoms have been omitted.

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