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3-(2-Chlorobenzyl)-2-thioxoperhydropyrimidin-4-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.103
Data-to-parameter ratio = 16.6

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

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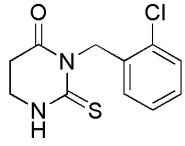
The tetrahydropyrimidine ring of the title molecule, $C_{11}H_{11}ClN_2OS$, adopts a half-chair conformation. In the crystal structure, the molecules are linked to form centrosymmetric hydrogen-bonded dimers.

Comment

The derivatives of uracil and thiouracil are very attractive because of their varied bioactivity (Gupta *et al.*, 2004; South *et al.*, 2003). For example, lenacil, bromacil, butafenacil, flupropacil, isocil and terbacil are widely used as herbicides. Besides, some of them possess antidiabetic activity (Soliman, 1979). This led us to pay more attention to the synthesis and structure determination of these compounds. Recently, we have synthesized a series of derivatives of uracil and thiouracil to study the relationship between the structure and herbicidal activity. 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). The attachment of the chlorobenzyl ring to the tetrahydropyrimidine ring is described by the torsion angle C1—N1—C5—C6 of 89.1 (2)°. In the crystal structure, centrosymmetrically related molecules form dimeric pairs through intermolecular N—H···S hydrogen bonds (Fig. 2 and Table 2).

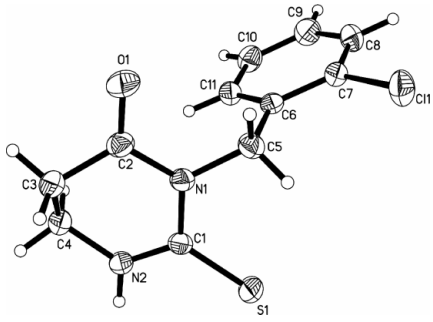


Figure 1
The structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

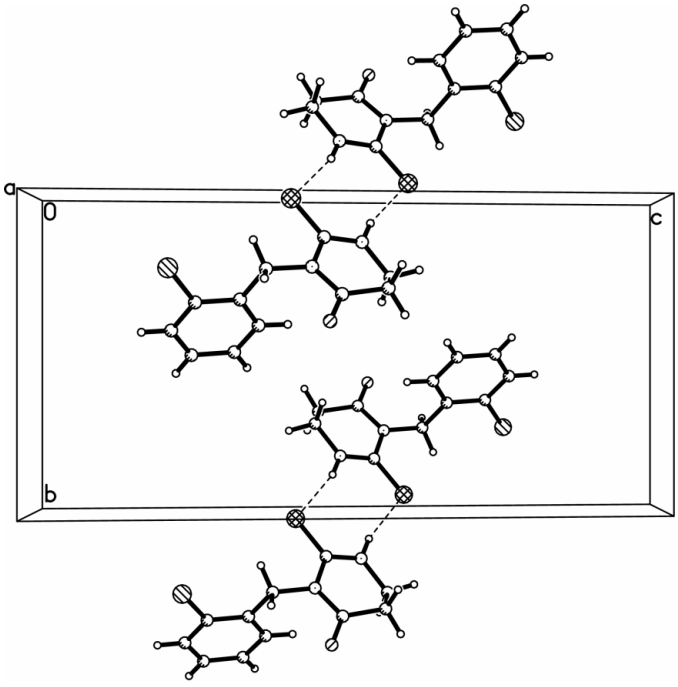


Figure 2
The N—H···S hydrogen-bonded dimers in (I), viewed down the *a* axis. Intermolecular hydrogen bonds are shown as dashed lines.

Experimental

According to the reported procedure of Hatam *et al.* (1996), the title compound was synthesized by refluxing 3-({[(2-chlorobenzyl)-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, which gave single crystals suitable for X-ray diffraction.

Crystal data

$C_{11}H_{11}ClN_2OS$
M_r = 254.73
Monoclinic, $P2_1/c$
a = 5.3014 (19) Å
b = 10.502 (4) Å
c = 21.102 (7) Å
β = 95.591 (5)°
V = 1169.3 (7) Å^3
Z = 4
D_x = 1.447 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 781 reflections
θ = 2.7–26.3°
μ = 0.48 mm^{-1}
T = 293 (2) K
Prism, colorless
0.26 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.807, T_{max} = 0.908
6631 measured reflections
2410 independent reflections
1925 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 26.4°
$h = -6 \rightarrow 5$
$k = -13 \rightarrow 11$
$l = -26 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.103
S = 1.07
2410 reflections
145 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.4892P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.676 (2)	C3—C4	1.491 (3)
Cl1—C7	1.739 (2)	C5—C6	1.511 (3)
N1—C1	1.392 (2)	C6—C11	1.388 (3)
N1—C2	1.398 (3)	C6—C7	1.395 (3)
N1—C5	1.471 (3)	C7—C8	1.381 (3)
N2—C1	1.315 (3)	C8—C9	1.374 (3)
N2—C4	1.455 (3)	C9—C10	1.371 (3)
O1—C2	1.210 (2)	C10—C11	1.386 (3)
C2—C3	1.495 (3)		
C1—N1—C2	122.90 (17)	N1—C5—C6	112.91 (17)
C1—N1—C5	119.62 (16)	C11—C6—C7	116.86 (19)
C2—N1—C5	117.41 (16)	C11—C6—C5	123.08 (18)
C1—N2—C4	123.95 (17)	C7—C6—C5	120.06 (18)
N2—C1—N1	116.71 (17)	C8—C7—C6	122.1 (2)
N2—C1—S1	121.72 (15)	C8—C7—Cl1	118.82 (16)
N1—C1—S1	121.56 (15)	C6—C7—Cl1	119.11 (17)
O1—C2—N1	120.7 (2)	C9—C8—C7	119.6 (2)
O1—C2—C3	123.57 (19)	C10—C9—C8	119.7 (2)
N1—C2—C3	115.68 (17)	C9—C10—C11	120.5 (2)
C4—C3—C2	110.65 (18)	C10—C11—C6	121.23 (19)
N2—C4—C3	108.42 (19)		
C4—N2—C1—N1	5.4 (3)	C5—N1—C2—C3	−175.25 (19)
C4—N2—C1—S1	−173.80 (18)	O1—C2—C3—C4	144.7 (2)
C2—N1—C1—N2	15.3 (3)	N1—C2—C3—C4	−36.2 (3)
C5—N1—C1—N2	−167.77 (18)	C1—N2—C4—C3	−39.4 (3)
C2—N1—C1—S1	−165.51 (16)	C2—C3—C4—N2	51.9 (3)
C5—N1—C1—S1	11.4 (3)	C1—N1—C5—C6	89.1 (2)
C1—N1—C2—O1	−179.2 (2)	C2—N1—C5—C6	−93.8 (2)
C5—N1—C2—O1	3.8 (3)	N1—C5—C6—C11	6.9 (3)
C1—N1—C2—C3	1.7 (3)	N1—C5—C6—C7	−173.86 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···S1^{i}	0.86	2.48	3.321 (2)	166

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

H atoms were placed in calculated positions, with C—H = 0.93 or 0.97 Å and N—H = 0.86 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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