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Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.041
wR factor = 0.098
Data-to-parameter ratio = 16.9

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

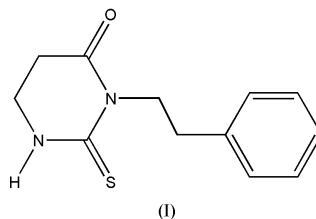
3-Phenethyl-2-thioxo-2,3,5,6-tetrahydro- pyrimidin-4(1H)-one

The tetrahydropyrimidine ring of the title molecule, $\text{C}_{12}\text{H}_{14}\text{N}_2\text{OS}$, adopts a half-chair conformation. In the crystal structure, the molecules are linked to form centrosymmetrically related hydrogen-bonded dimers.

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Comment

The 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). 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. We report here the crystal structure of 3-phenethyl-2-thioxo-2,3,5,6-tetrahydropyrimidin-4(1H)-one, (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 & Aurrecochea, 1994; Rohrer & Sundaralingam, 1968; Furberg & Jensen, 1968; Yao *et al.*, 2004). The conformation of the attachment of the phenyl ring to the tetrahydropyrimidine ring is described by the torsion angle $\text{N1}-\text{C5}-\text{C6}-\text{C7}$ of $-173.97 (14)^\circ$. In the crystal structure, centrosymmetrically related molecules form dimeric pairs through intermolecular $\text{N}-\text{H} \cdots \text{S}$ hydrogen bonds (Fig. 2 and Table 2).

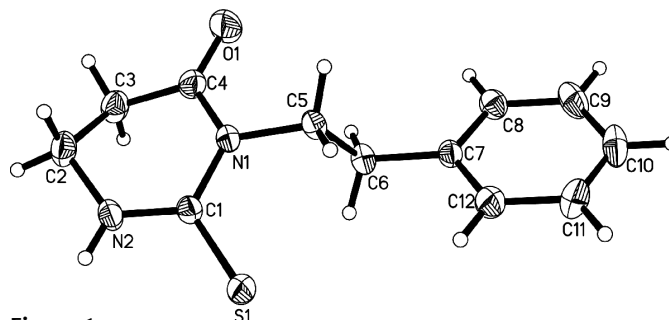


Figure 1
The structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

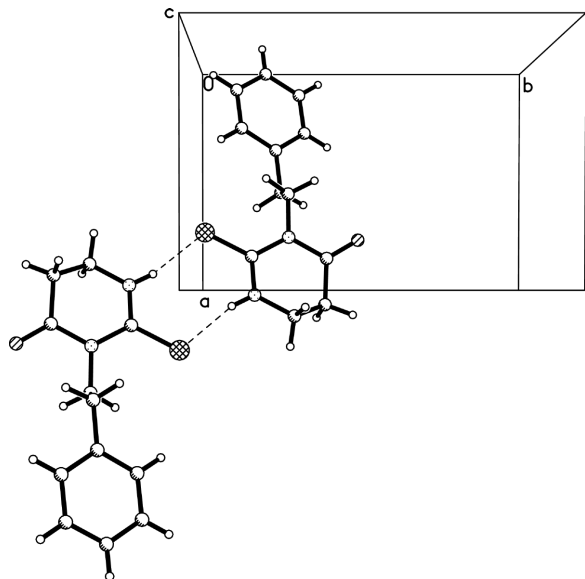


Figure 2
One of the N—H...S hydrogen-bonded dimers in (I), viewed down the *c* axis. Intermolecular hydrogen bonds are shown as dashed lines.

Experimental

According to the reported procedure of Hatam *et al.* (1996), the title compound was synthesized by refluxing methyl 3-((2-phenylethyl)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, giving single crystals suitable for X-ray diffraction. $C_{12}H_{14}N_2OS$ requires: C 61.51, H 6.02, N 11.96%; found: C 61.50, H 5.96, N 12.04%. 1H NMR (300 MHz, $CDCl_3$, p.p.m.): 2.77 (*t*, 2H, $J = 7.01$ Hz, CH_2CO); 2.97–3.02 (*m*, 2H, CH_2Ph); 3.42–3.48 (*m*, 2H, CH_2NH), 4.40–4.46 (*m*, 2H, CH_2N), 7.20–7.33 (*m*, 5H, Ph), 8.02 (*s*, 1H, MH).

Crystal data

$C_{12}H_{14}N_2OS$	$D_x = 1.311$ Mg m $^{-3}$
$M_r = 234.31$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 720 reflections
$a = 7.574$ (3) Å	$\theta = 3.0$ – 25.7°
$b = 11.104$ (4) Å	$\mu = 0.25$ mm $^{-1}$
$c = 14.122$ (5) Å	$T = 293$ (2) K
$\beta = 91.719$ (8) $^\circ$	Prism, colorless
$V = 1187.2$ (7) Å 3	0.20 × 0.16 × 0.10 mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	2530 independent reflections
φ and ω scans	1805 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{int} = 0.031$
$T_{min} = 0.934$, $T_{max} = 0.975$	$\theta_{max} = 26.8^\circ$
6813 measured reflections	$h = -8 \rightarrow 9$
	$k = -8 \rightarrow 14$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2 + 0.3285P]$
$R[F^2 > 2\sigma(F^2)] = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.099$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.20$ e Å $^{-3}$
2530 reflections	$\Delta\rho_{min} = -0.16$ e Å $^{-3}$
150 parameters	Extinction correction: SHELXL97
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.023 (2)

Table 1

Selected geometric parameters (Å, $^\circ$).

S1—C1	1.6801 (19)	C5—C6	1.518 (3)
O1—C4	1.211 (2)	C6—C7	1.511 (2)
N1—C1	1.390 (2)	C7—C8	1.383 (3)
N1—C4	1.404 (2)	C7—C12	1.387 (3)
N1—C5	1.474 (2)	C8—C9	1.392 (3)
N2—C1	1.324 (2)	C9—C10	1.369 (3)
N2—C2	1.451 (2)	C10—C11	1.367 (3)
C2—C3	1.494 (3)	C11—C12	1.382 (3)
C3—C4	1.498 (3)		
C1—N1—C4	123.06 (15)	O1—C4—C3	123.51 (18)
C1—N1—C5	119.93 (15)	N1—C4—C3	115.83 (16)
C4—N1—C5	116.70 (14)	N1—C5—C6	111.68 (14)
C1—N2—C2	124.27 (17)	C7—C6—C5	112.44 (15)
C1—N2—H2C	116.1 (14)	C8—C7—C12	118.19 (17)
C2—N2—H2C	118.6 (14)	C8—C7—C6	121.42 (17)
N2—C1—N1	116.72 (16)	C12—C7—C6	120.38 (16)
N2—C1—S1	121.38 (14)	C7—C8—C9	120.39 (19)
N1—C1—S1	121.90 (13)	C10—C9—C8	120.25 (19)
N2—C2—C3	108.75 (16)	C11—C10—C9	120.10 (19)
C2—C3—C4	110.77 (16)	C10—C11—C12	119.9 (2)
O1—C4—N1	120.62 (18)	C11—C12—C7	121.16 (19)
C2—N2—C1—N1	−7.4 (3)	C1—N1—C5—C6	89.0 (2)
C2—N2—C1—S1	172.33 (15)	C4—N1—C5—C6	−84.8 (2)
C4—N1—C1—N2	−11.8 (3)	N1—C5—C6—C7	−173.97 (14)
C5—N1—C1—N2	174.85 (16)	C5—C6—C7—C8	−110.2 (2)
C4—N1—C1—S1	168.48 (14)	C5—C6—C7—C12	70.0 (2)
C5—N1—C1—S1	−4.9 (2)	C12—C7—C8—C9	0.0 (3)
C1—N2—C2—C3	39.3 (3)	C6—C7—C8—C9	−179.76 (17)
N2—C2—C3—C4	−50.9 (2)	C7—C8—C9—C10	0.1 (3)
C1—N1—C4—O1	177.80 (18)	C8—C9—C10—C11	−0.3 (3)
C5—N1—C4—O1	−8.6 (3)	C9—C10—C11—C12	0.2 (3)
C1—N1—C4—C3	−4.4 (3)	C10—C11—C12—C7	0.0 (3)
C5—N1—C4—C3	169.16 (16)	C8—C7—C12—C11	−0.1 (3)
C2—C3—C4—O1	−145.8 (2)	C6—C7—C12—C11	179.70 (19)
C2—C3—C4—N1	36.5 (2)		

Table 2

Hydrogen-bonding geometry (Å, $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2C \cdots S1 i	0.84 (2)	2.53 (2)	3.3332 (19)	159.1 (18)

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

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 $U_{iso}(H) = 1.2U_{eq}(\text{parent atom})$. The position of the amine H atom was refined freely along with an isotropic displacement parameter.

Data collection: SMART (Bruker, 1998); cell refinement: SMART; data reduction: SAINT (Bruker, 1999); program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); 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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References

- Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (1999). *SAINTE* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Furberg, S. & Jensen, L. H. (1968). *J. Am. Chem. Soc.* **90**, 470–474.
- Gupta, S., Pulman, D. A. & Roh, T. (2004). US Patent No. 20 040 018 941.
- Hatam, M., Kopper, S. & Martens, J. (1996). *Heterocycles*, **43**, 1653–1663.
- Lorente, A. & Aurrecoechea, L. M. (1994). *Heterocycles*, **38**, 1077–1087.
- Rohrer, D. & Sundaralingam, M. (1968). *Chem. Commun.* pp. 746–747.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Soliman, R. (1979). *J. Med. Chem.* **22**, 351–353.
- South, M. S., Jones, D. E. & Rueppel, M. L. (2003). US Patent No. 20 030 023 086.
- Yao, C.-S., Song, H.-B., Zhu, Y.-Q., Gao, Y., Hu, F.-Z., Zou, X.-M. & Yang, H.-Z. (2004). *Acta Cryst.* **E60**, o943–o945.