

3-Amino-2-methyl-5,6,7,8-tetrahydro-1-benzothieno[2,3-*d*]pyrimidin-4(3*H*)-oneVasu,^a K. A. Nirmala,^b
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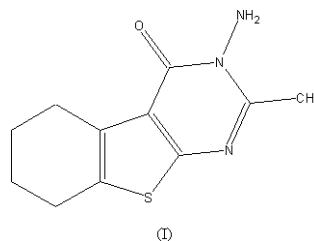
Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$
R factor = 0.090
wR factor = 0.234
Data-to-parameter ratio = 15.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_{11}\text{H}_{13}\text{N}_3\text{OS}$, the dihedral angle between the fused thiophene and pyrimidinone moieties is $3.3(1)^\circ$. The crystal structure is stabilized by an intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond and weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions.

Comment

Pyrimidinones are an important class of compound and found to exhibit different biological activities (Narr & Woitun, 1973). Thieno[2,3-*d*]pyrimidine and 2-aminothiophene derivatives possess platelet aggregation inhibition (Kikugawa & Ichino, 1973) and antitumor activities (Patil *et al.*, 1974). In view of the diverse applications of these compounds in different biological activities, a structure analysis has been carried out for the title compound, (I).



The torsion angle $\text{C}11-\text{C}10-\text{N}1-\text{N}2$ is $-2.8(6)^\circ$, indicating that the methyl and amino groups are in a *cis* orientation (Fig. 1). The cyclohexene ring adopts a half-chair conformation, with C2 and C3 out of the mean plane of the other atoms, on opposite sides. There is an intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond (Table 1) which locks the molecule in a pseudo-five-membered ring. There are also intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions, forming molecular chains parallel to the *b* axis (Fig. 2).

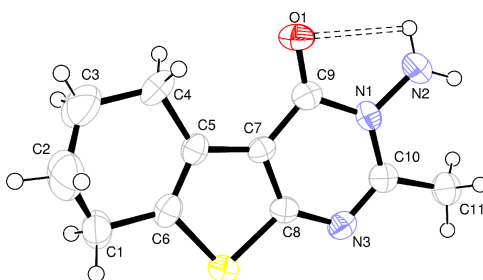


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

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Experimental

The title compound, (I), was synthesized using the Gewald reaction (Gewald *et al.*, 1966). A mixture of cyclohexanone (3.92 g, 0.04 mol), ethyl cyanoacetate (4.52 g, 0.04 mol) and sulfur (1.2 g, 0.04 mol) was dissolved in ethanol (40 ml) and to the resulting solution diethylamine (4.0 ml) was added with constant stirring. This was continued for 1 h at 325 K and then the solution was allowed to cool overnight. The resulting solid was washed and dried with ethanol and finally refluxed with hydrazine for 4 h in methanol to obtain the title compound (yield 65%). Crystals of (I) of suitable size and quality were grown from an acetone solution by slow evaporation.

Crystal data

C₁₁H₁₃N₃O₅ $D_x = 1.441 \text{ Mg m}^{-3}$
 $M_r = 235.31$ Mo $K\alpha$ radiation
 Monoclinic, $P2_1/c$ Cell parameters from 660 reflections
 $a = 10.001(3) \text{ \AA}$ $\theta = 1.4\text{--}25.8^\circ$
 $b = 11.929(3) \text{ \AA}$ $\mu = 0.28 \text{ mm}^{-1}$
 $c = 9.544(3) \text{ \AA}$ $T = 293(2) \text{ K}$
 $\beta = 107.656(4)^\circ$ Block, yellow
 $V = 1085.0(5) \text{ \AA}^3$ $0.35 \times 0.25 \times 0.20 \text{ mm}$
 $Z = 4$

Data collection

Bruker SMART CCD area-detector 2252 independent reflections
 diffractometer 1972 reflections with $I > 2\sigma(I)$
 φ and ω scans $R_{int} = 0.028$
 Absorption correction: multi-scan $\theta_{max} = 27.2^\circ$
 (SADABS; Sheldrick, 1997) $h = -12 \rightarrow 12$
 $T_{min} = 0.899, T_{max} = 0.946$ $k = -15 \rightarrow 15$
 8148 measured reflections $l = -11 \rightarrow 12$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.121P)^2 + 2.7775P]$
 $R[F^2 > 2\sigma(F^2)] = 0.090$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.234$ $(\Delta/\sigma)_{max} < 0.001$
 $S = 1.01$ $\Delta\rho_{max} = 0.87 \text{ e \AA}^{-3}$
 2252 reflections $\Delta\rho_{min} = -0.46 \text{ e \AA}^{-3}$
 148 parameters
 H-atom parameters constrained

Table 1

Hydrogen-bonding geometry ($\text{\AA}, ^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—HN2B \cdots O1	0.86	2.37	2.657 (5)	100
C11—H11A \cdots O1 ¹	0.96	2.57	3.500 (6)	165

Symmetry code: (i) $1 - x, y - \frac{1}{2}, -\frac{1}{2} - z$.

The position of the H atoms on the amino group were constrained to an ideal geometry, with $N-H = 0.86 \text{ \AA}$ and $U_{iso}(H) = 1.2U_{eq}(N)$. The methyl H atoms were constrained to an ideal geometry, with $C-H = 0.96 \text{ \AA}$ and $U_{iso}(H) = 1.5U_{eq}(C)$, but were allowed to rotate freely about the $C-C$ bond. All the remaining H atoms were placed in idealized positions and constrained to ride on their parent atoms with $C-H = 0.97 \text{ \AA}$ and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: SMART (Bruker, 1998); cell refinement: SMART; data reduction: SAINT (Bruker, 1998); program(s) used to solve

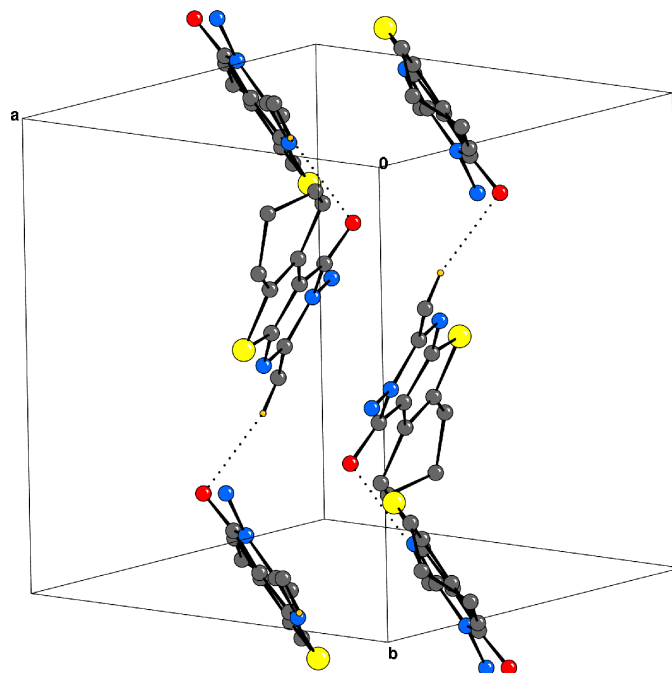


Figure 2 Packing diagram of (I), showing intermolecular $C-H\cdots O$ chains along the b axis.

structure: SIR92 (Altomare *et al.*, 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997) and CAMERON (Watkin *et al.*, 1993); software used to prepare material for publication: PLATON (Spek, 2003).

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