

Ethyl 2-amino-5,6,7,8-tetrahydro-4*H*-cyclohepta[*b*]thiophene-3-carboxylateK. A. Nirmala,<sup>a\*</sup> Vasu,<sup>b</sup> Deepak Chopra,<sup>c</sup> S. Mohan<sup>d</sup> and M. Raghu Prasad<sup>d</sup><sup>a</sup>Department of Physics, Bangalore University, Bangalore 560 056, Karnataka, India, <sup>b</sup>Vivekananda Degree College, Bangalore 560 055, Karnataka, India, <sup>c</sup>Solid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560 012, Karnataka, India, and <sup>d</sup>PES College of Pharmacy, Hanumanthanagar, Bangalore 560 050, Karnataka, IndiaCorrespondence e-mail:  
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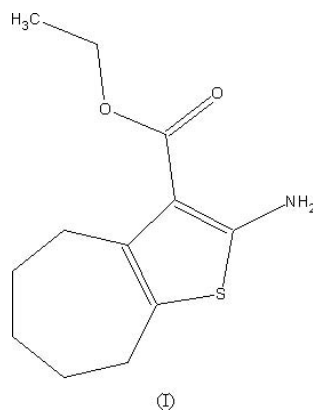
## Key indicators

Single-crystal X-ray study  
 $T = 290$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.038  
 $wR$  factor = 0.102  
Data-to-parameter ratio = 11.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the structure of the title compound,  $\text{C}_{12}\text{H}_{17}\text{NO}_2\text{S}$ , the terminal ester group lies in the plane of the thiophene ring system. The cycloheptene ring adopts a half-chair conformation. There are intramolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  interactions, and intermolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\pi$  interactions.

## Comment

2-Aminothiophenes have been a useful class of compounds because they display different biological activities, such as antitubercular, bacteriostatic and antifungal activities (Nakanishi *et al.*, 1970). The terminal ester group of the title compound, (I), lies in the plane of the thiophene ring system (Fig. 1 and Table 1). The seven-membered cycloheptene ring shows a half-chair conformation, with atoms C2, C4, C1 and C5 deviating from the mean plane by 0.280 (2), 0.288 (2),  $-0.427$  (2) and  $-0.420$  (2) Å, respectively. The puckering parameters (Cremer & Pople, 1975) calculated by *PLATON* (Spek, 2003) for this ring are  $q_2 = 0.392$  (2) Å,  $\varphi_2 = 156.8$  (3)° and  $\tau = 31.8$  (2)°, also indicating a half-chair conformation.



There is an intramolecular  $\text{N1}-\text{H1}\cdots\text{O1}$  hydrogen bond (Table 2), which locks the molecular conformation and eliminates conformational flexibility. This conformation is further stabilized by an intramolecular  $\text{C5}-\text{H4}\cdots\text{O2}$  interaction. Molecules are connected by  $\text{N1}-\text{H2}\cdots\text{O1}^i$  [symmetry code (i):  $-x, y - \frac{1}{2}, -z - \frac{1}{2}$ ] hydrogen bonds, forming chains along the *b* axis (Fig. 2). The molecules in adjacent parallel chains are held by  $\text{C}-\text{H}\cdots\pi$  intermolecular interactions (Table 2).

## Experimental

Compound (I) was synthesized *via* the Gewald reaction (Gewald *et al.*, 1966) by mixing cycloheptanone (0.04 mol), ethyl cyanoacetate (0.04 mol), sulfur (0.04 mol) and ethanol (40 ml) and the resulting

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mixture was stirred at 325 K for 1 h with dropwise addition of dimethylamine (4 ml) to yield the ester, followed by alkaline hydrolysis using sodium hydroxide solution. Crystals of (I) were grown from a methanol solution by slow evaporation (yield 75%).

Crystal data

C<sub>12</sub>H<sub>17</sub>NO<sub>2</sub>S  
*M<sub>r</sub>* = 239.34  
 Monoclinic, *P*<sub>2</sub><sub>1</sub>/*c*  
*a* = 9.580 (3) Å  
*b* = 9.552 (2) Å  
*c* = 13.677 (4) Å  
 β = 99.489 (4)°  
*V* = 1234.3 (6) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.288 Mg m<sup>-3</sup>  
 Mo Kα radiation  
 Cell parameters from 635 reflections  
 θ = 1.4–25.4°  
 μ = 0.25 mm<sup>-1</sup>  
*T* = 290 (2) K  
 Block, yellow  
 0.39 × 0.35 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer  
 φ and ω scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
*T<sub>min</sub>* = 0.918, *T<sub>max</sub>* = 0.966  
 9377 measured reflections

2487 independent reflections  
 2221 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.021  
 θ<sub>max</sub> = 26.4°  
*h* = -11 → 11  
*k* = -11 → 11  
*l* = -17 → 14

Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.038  
*wR* (*F*<sup>2</sup>) = 0.102  
*S* = 1.10  
 2487 reflections  
 213 parameters  
 All H-atom parameters refined

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0573*P*)<sup>2</sup> + 0.2147*P*]  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 (Δ/σ)<sub>max</sub> < 0.001  
 Δρ<sub>max</sub> = 0.20 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.32 e Å<sup>-3</sup>

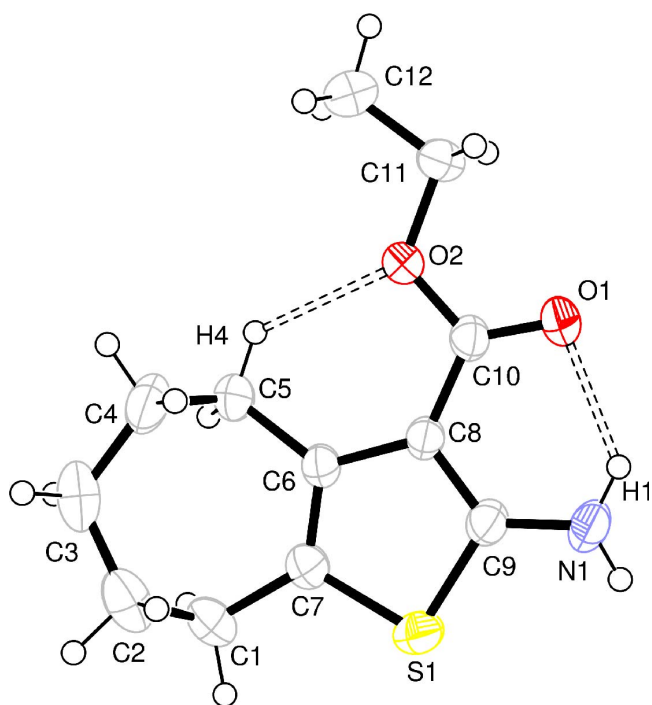


Figure 1 The molecular structure of (I), showing 50% probability ellipsoids. The broken lines indicate hydrogen bonds.

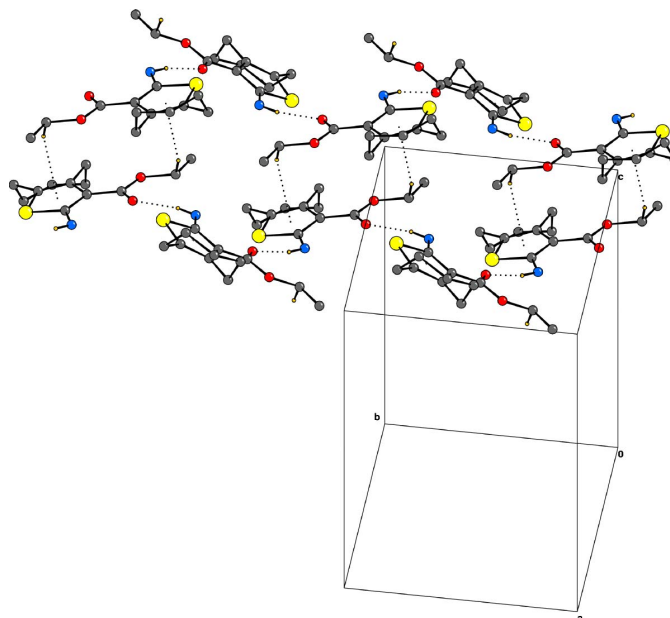


Figure 2 Packing diagram of (I). Dotted lines indicate N–H···O hydrogen bonds and C–H···π interactions. H atoms have been omitted unless they are involved in hydrogen bonding.

Table 1 Selected torsion angles (°).

C11–O2–C10–C8	177.79 (12)	C10–O2–C11–C12	-178.65 (15)
O1–C10–C8–C9	3.2 (2)	C6–C5–C4–C3	-77.66 (19)
O2–C10–C8–C6	3.2 (2)	C7–C1–C2–C3	73.9 (2)
C5–C6–C7–C1	-2.2 (2)		

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

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N1–H1···O1	0.87 (2)	2.06 (2)	2.717 (2)	132 (2)
N1–H2···O1 <sup>i</sup>	0.84 (2)	2.15 (2)	2.964 (2)	166 (2)
C5–H4···O2	0.93 (2)	2.27 (2)	2.920 (2)	127 (1)
C11–H16···Cg1 <sup>ii</sup>	0.96 (2)	2.76	3.578	143

Symmetry codes: (i)  $-x, y - \frac{1}{2}, -\frac{1}{2} - z$ ; (ii)  $-x, 1 - y, -z$ . Cg1 is the centroid of the thiophene ring.

All H atoms were located in difference Fourier maps and refined freely with isotropic displacement parameters. The N–H and C–H bond lengths are in the ranges 0.84 (2)–0.87 (2) and 0.92 (2)–1.00 (2) Å, respectively.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve 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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