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Ethyl 2-amino-5,6,7,8-tetrahydro-4*H*-cyclohepta[*b*]thiophene-3-carboxylate

K. A. Nirmala,[a]* Vasu,[b] Deepak Chopra,[c] S. Mohan[d] and M. Raghu Prasad[d]

[a]Department of Physics, Bangalore University, Bangalore 560 056, Karnataka, India, [b]Vivekananda Degree College, Bangalore 560 055, Karnataka, India, [c]Solid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560 012, Karnataka, India, and [d]PES College of Pharmacy, Hanumanthanagar, Bangalore 560 050, Karnataka, India

Correspondence e-mail: nirmalaka47@yahoo.com

Key indicators

Single-crystal X-ray study
T = 290 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.102
Data-to-parameter ratio = 11.7

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

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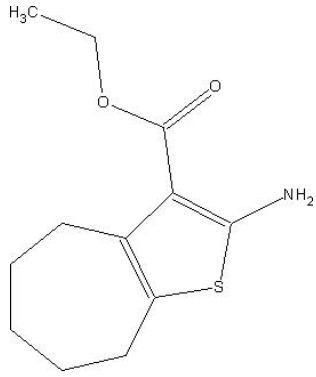
In the structure of the title compound, $C_{12}H_{17}NO_2S$, the terminal ester group lies in the plane of the thiophene ring system. The cycloheptene ring adopts a half-chair conformation. There are intramolecular N—H···O and C—H···O interactions, and intermolecular N—H···O and C—H···π interactions.

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, φ_2 = 156.8 (3)° and τ = 31.8 (2)°, also indicating a half-chair conformation.



(I)

There is an intramolecular N1—H1···O1 hydrogen bond (Table 2), which locks the molecular conformation and eliminates conformational flexibility. This conformation is further stabilized by an intramolecular C5—H4···O2 interaction. Molecules are connected by N1—H2···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 C—H···π 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

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

$C_{12}H_{17}NO_2S$	D_x = 1.288 Mg m^{-3}
M_r = 239.34	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 635 reflections
a = 9.580 (3) Å	θ = 1.4–25.4°
b = 9.552 (2) Å	μ = 0.25 mm^{-1}
c = 13.677 (4) Å	T = 290 (2) K
β = 99.489 (4)°	Block, yellow
V = 1234.3 (6) Å^3	0.39 × 0.35 × 0.14 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2487 independent reflections
φ and ω scans	2221 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	R_{int} = 0.021
T_{min} = 0.918, T_{max} = 0.966	θ_{max} = 26.4°
9377 measured reflections	$h = -11 \rightarrow 11$
	$k = -11 \rightarrow 11$
	$l = -17 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 0.2147P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.102	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
S = 1.10	$\Delta\rho_{min}$ = −0.32 e Å^{-3}
2487 reflections	
213 parameters	
All H-atom parameters refined	

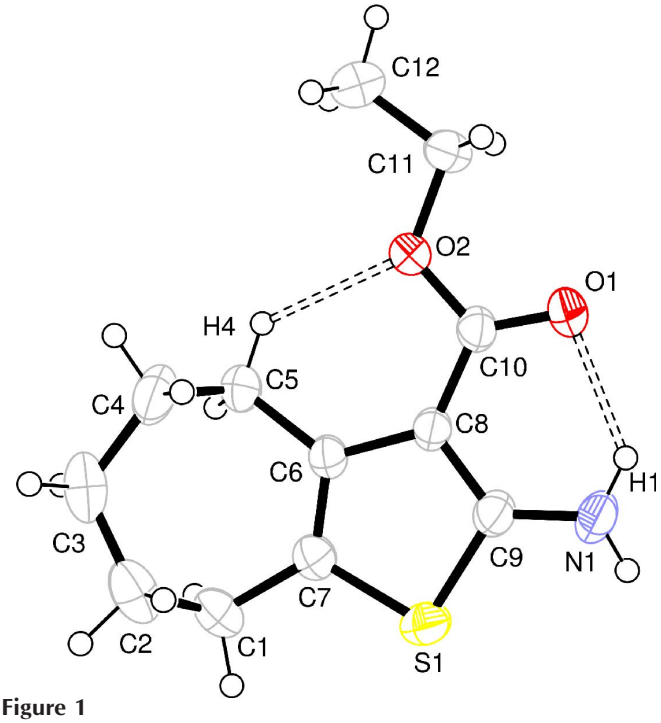


Figure 1
The molecular structure of (I), showing 50% probability ellipsoids. The broken lines indicate hydrogen bonds.

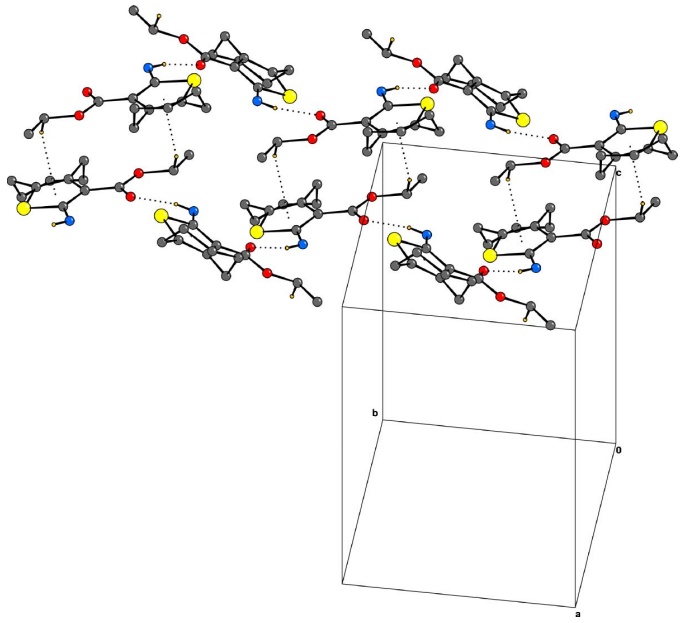


Figure 2
Packing diagram of (I). Dotted lines indicate N—H···O hydrogen bonds and C—H···π interactions. H atoms have been omitted unless they are involved in hydrogen bonding.

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, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.87 (2)	2.06 (2)	2.717 (2)	132 (2)
N1—H2···O1^{i}	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···$Cg1^{ii}$	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: *SIR*92 (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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