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Key indicators

Single-crystal X-ray study
T = 120 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.036
wR factor = 0.091
Data-to-parameter ratio = 17.4

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

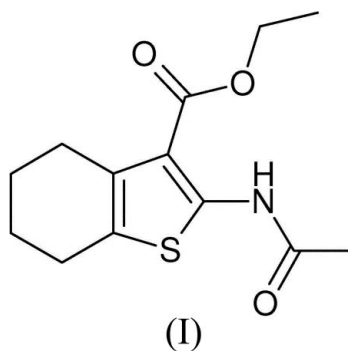
Ethyl 2-acetylamino-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxylate

The geometrical parameters for the title compound, $\text{C}_{13}\text{H}_{17}\text{NO}_3\text{S}$, are normal. The planar molecular conformation is reinforced by an intramolecular $\text{N}-\text{H}\cdots\text{O}$ interaction.

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Comment

Thiophene derivatives are known to exhibit an array of biological effects, including analgesic and anti-inflammatory activities (Ramanathan & Namboothiri, 1978; Cannito *et al.*, 1990). As part of our own research in this area, the structure of the title compound, (I) (Fig. 1), is presented.



The geometric parameters for (I) are normal. The five-membered C1/C2/C6/C7/S1 ring is almost planar (r.m.s. deviation from the mean plane = 0.013 Å). The C1–C6 ring is in a half-chair conformation (Table 1), with atoms C1, C2, C5 and C6 almost co-planar (r.m.s. deviation = 0.003 Å) and atoms C3 and C4 displaced from this plane by −0.480 (3) and 0.277 (3) Å, respectively. An intramolecular $\text{N}-\text{H}\cdots\text{O}$ bond (Table 2) helps to establish the molecular conformation. Overall, the molecule of (I) is approximately planar.

The molecular packing for (I) comprises undulating sheets lying parallel to the (10 $\bar{1}$) plane (Fig. 2). Within these sheets, the shortest intermolecular contacts are $\text{C}-\text{H}\cdots\text{O}$ interactions (Table 2).

Experimental

Ethyl-2-amino-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxylate, (II), was prepared from cyclohexanone, sulfur and ethyl cyanoacetate by a one-pot thiolation-heterocyclization reaction (Gewald *et al.*, 1966). A mixture of (II) (3.5 g, 0.015 mol), acetic anhydride (14 ml) and zinc dust (0.883 g, 0.015 mol) was refluxed for 2 h. The reaction mixture was cooled to room temperature and the solid product was recovered. The crude product was dissolved in warm (318 K) methanol (35 ml) and filtered. The product was recrystallized from acetone to yield colourless crystals of (I) (yield 84.3%; m.p. 388 K). IR (KBr, cm^{-1}): 3436 and 3244 (–NH–), 2931 and 2873 (–CH–),

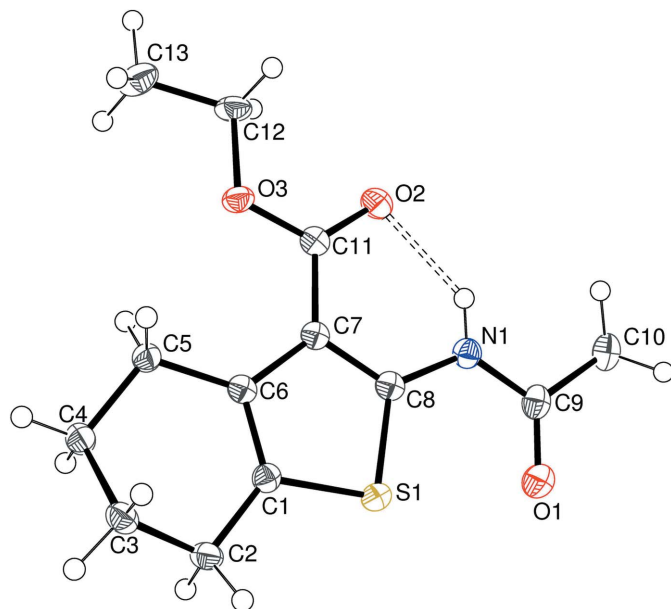


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and arbitrary spheres for the H atoms. The intramolecular hydrogen bond is indicated by a dashed line.

1666 and 1546 (C=O) and 1250 (C–O). Elemental analysis, found: C 58.18, H 6.32, N 5.16%; calculated: C58.40, H 6.41, N 5.24%.

Crystal data

$C_{13}H_{17}NO_3S$
 $M_r = 267.34$
 Monoclinic, $P2_1/c$
 $a = 10.2987$ (4) Å
 $b = 16.6174$ (5) Å
 $c = 7.8510$ (3) Å
 $\beta = 108.4381$ (18)°
 $V = 1274.63$ (8) Å³

$Z = 4$
 $D_x = 1.393$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.25$ mm⁻¹
 $T = 120$ (2) K
 Cut block, colourless
 $0.44 \times 0.30 \times 0.18$ mm

Data collection

Nonius KappaCCD diffractometer
 ω and φ scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2003)
 $T_{\min} = 0.897$, $T_{\max} = 0.958$

16538 measured reflections
 2917 independent reflections
 2338 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$
 $\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.091$
 $S = 1.05$
 2917 reflections
 168 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 + 0.574P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.28$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³

Table 1

Selected torsion angles (°).

C1–C2–C3–C4	48.83 (16)	C4–C5–C6–C1	–10.54 (19)
C2–C3–C4–C5	–63.48 (17)	C5–C6–C1–C2	–0.9 (2)
C3–C4–C5–C6	42.28 (18)	C6–C1–C2–C3	–18.7 (2)

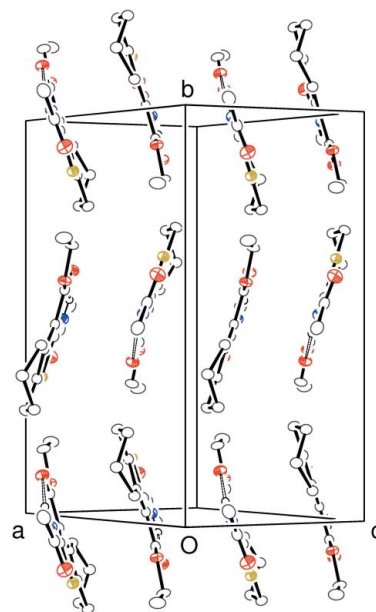


Figure 2

A view of the unit-cell contents of (I), with H atoms (except H1) omitted for clarity.

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N1-H1 \cdots O2$	0.858 (18)	1.979 (18)	2.6798 (17)	138.1 (16)
$C2-H2A \cdots O2^i$	0.99	2.44	3.370 (2)	157

Symmetry code: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$.

The N-bound H atom was located in a difference map and its position was refined freely, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. The C-bound H atoms were placed in idealised locations (C–H = 0.98–0.99 Å) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$. The methyl groups were allowed to rotate about their local threefold axes to fit the electron density.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997) and *SORTAV* (Blessing, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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References

- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
 Bruker (2003). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Cannito, A., Perrisin, M., LuuDuc, C., Huguer, F., Gaultier, C. & Narcisse, G. (1990). *Eur. J. Med. Chem.* **25**, 635–639.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Gewald, K., Schinke, E. & Bottcher, H. (1966). *Chem. Ber.* **99**, 94–100.

Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Ramanathan, J. C. D. G. & Namboothiri, D. G. (1978). *J. Indian Chem. Soc.* **55**, 822–823.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.