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Methyl 2-amino-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxylate

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The molecule of the title compound, $C_{10}H_{13}NO_2S$, is planar. The crystal structure is stabilized by intra- and intermolecular N—H···O hydrogen bonds.

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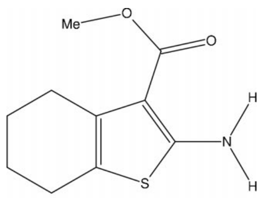
Comment

2-Aminothiophenes are a useful class of compounds because they display different biological properties, such as anti-tubercular, bacteriostatic and antifungal activities (Nakanishi *et al.*, 1970). The structure of the title compound, (I), is composed of a thiophene ring substituted by a 2-amino group and a 3-methyl ester group. The molecule is planar (Fig. 1). The terminal ester group lies in the plane of the thiophene ring system, the torsion angle C10—O2—C9—C2 being 178.0 (4)°.



(I)

The carbonyl group has a *cis* orientation with respect to the C2═C3 double bond. This leads to a strong intramolecular hydrogen bond (Table 1) between one of the H atoms, *viz.* H2N of the amino group, and the O atom of the carbonyl group. The other H atom, H1N, is involved in molecular chains along the *c* axis (Fig. 2).

Experimental

The title compound, (I), was synthesized by the Gewald reaction (Gewald *et al.*, 1966) by mixing cyclohexanone (0.04 mol), ethyl cyanoacetate (0.04 mol), sulfur (0.04 mol) and 40 ml of ethanol and stirring the mixture at 325 K for 1 h, with dropwise addition of 4 ml of dimethylamine to yield the ester, followed by alkaline hydrolysis

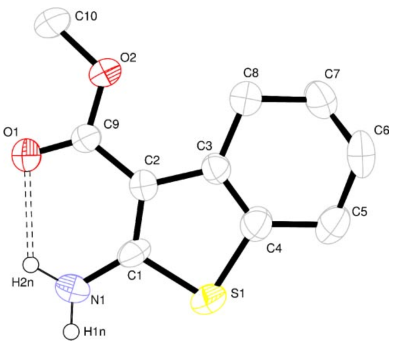


Figure 1
View of the molecule of (I), drawn with 50% probability displacement ellipsoids. The hydrogen bond is shown as dashed lines.

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.078
wR factor = 0.190
Data-to-parameter ratio = 13.3

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

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using sodium hydroxide solution. It was recrystallized from methanol and crystals of suitable size were removed for single-crystal studies.

Crystal data

$C_{10}H_{13}NO_2S$
$M_r = 211.28$
Monoclinic, $C2/c$
$a = 16.915$ (3) Å
$b = 14.003$ (3) Å
$c = 10.142$ (2) Å
$\beta = 121.018$ (3)°
$V = 2058.9$ (7) Å^3
$Z = 8$
$D_x = 1.363$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 450 reflections
$\theta = 1.6$–25.6°
$\mu = 0.29$ mm^{-1}
$T = 293$ (2) K
Plate, yellow
0.17 × 0.16 × 0.07 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{\min} = 0.918$, $T_{\max} = 0.980$
8343 measured reflections
1808 independent reflections
1337 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.054$
$\theta_{\max} = 27.8°$
$h = -21 \rightarrow 21$
$k = -18 \rightarrow 17$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.078$
$wR(F^2) = 0.190$
$S = 1.18$
1808 reflections
136 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0929P)^2 + 1.4124P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.54$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1$^{\rm i}$	0.84 (5)	2.17 (5)	2.972 (5)	161 (4)
N1—H2N···O1	0.94 (6)	2.14 (6)	2.766 (6)	123 (4)

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

The H atoms of the amino group were located in difference Fourier maps and refined freely. The N—H bond lengths are 0.84 (5) and 0.94 (6) Å. The methyl H atoms were constrained to an ideal geometry [C—H = 0.96 Å and $U_{\rm iso} = 1.5U_{\rm eq}$(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 [C—H = 0.97 Å and $U_{\rm iso} = 1.2U_{\rm eq}$(C)].

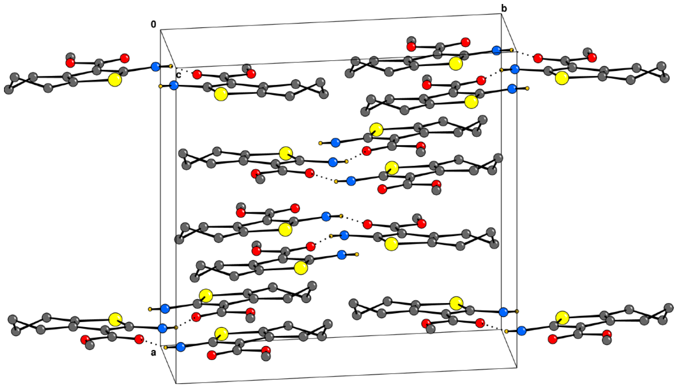

Figure 2
Packing diagram of (I), showing N—H···O hydrogen-bond chains as dashed lines.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (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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