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# Methyl 2-(acetylamino)-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxylate

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**Key indicators**

Single-crystal X-ray study
$T$ = 293 K
Mean $\sigma$(C–C) = 0.005 Å
Disorder in main residue
$R$ factor = 0.061
$wR$ factor = 0.208
Data-to-parameter ratio = 14.1

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



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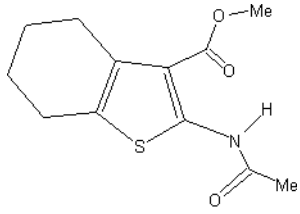
The title compound, $C_{12}H_{15}NO_3S$, exhibits antibacterial and antifungal activities. The cyclohexene ring exhibits disorder, indicating two possible conformations of the half-chair form. The molecular structure is approximately planar, supported by an intramolecular N—H···O hydrogen bond.

## Comment

Schiff bases (Csaszar & Morvay, 1983; Lakshmi *et al.*, 1985; Cohen *et al.*, 1977) and their derivatives of thiophene (El-Maghraby *et al.*, 1984; Dzhurayev *et al.*, 1992; Gewald *et al.*, 1966) possess antibacterial, antitubercular and antifungal properties. Sulfur-containing Schiff bases are most effective. The title compound, (I), shows the above-mentioned biological properties (Mohan & Saravanan, 2002, 2003).




(I)

The molecular structure of (I) is shown in Fig. 1. The deviations of atoms C5*a* and C6*a*, which constitute the portion of the cyclohexene ring of the major conformer, from the

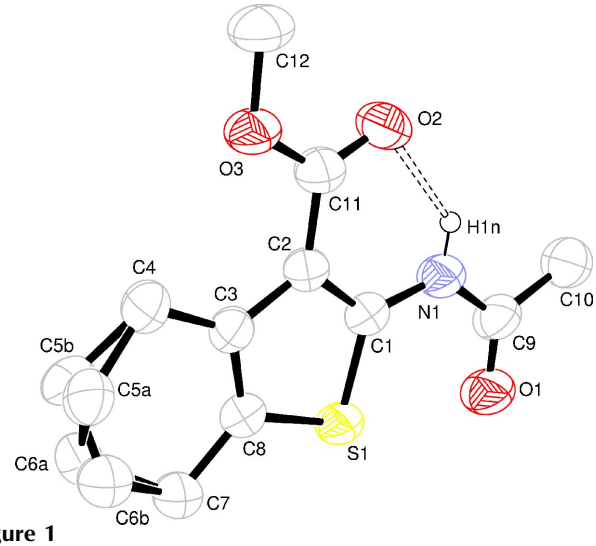



**Figure 1**
The molecular structure of (I), showing 50% probability ellipsoids. Both disorder components are shown. H atoms other than H1*n* have been omitted. Dashed lines indicate N—H···O hydrogen bonds.

C4/C3/C8/C7 plane are 0.412 (15) and −0.415 (15) Å, respectively. The torsion angles (Table 1) indicate that the molecule is approximately planar. An intramolecular N—H···O hydrogen bond (Table 2) forms a pseudo-six-membered ring, thus locking the molecular conformation and eliminating conformational flexibility.

## Experimental

Cyclohexanone (3.92 g, 0.04 mol), methyl cyanoacetate (4.52 g, 0.04 mol) and sulfur (1.2 g, 0.04 mol) were mixed with ethanol (40 ml) and stirred at 315 K for 1 h with dropwise addition of diethylamine (4 ml). The product obtained was then reacted with acetic anhydride (10 ml) and heated on a water bath until the solid dissolved. The mixture was allowed to cool and the resulting solid (yield 64%) was recrystallized from ethanol.

*Crystal data*

$C_{12}H_{15}NO_3S$
$M_r$ = 253.32
Monoclinic, $C2/c$
$a$ = 15.289 (14) Å
$b$ = 10.644 (10) Å
$c$ = 15.790 (15) Å
$\beta$ = 105.212 (13)°
$V$ = 2480 (4) Å$^3$
$Z$ = 8
$D_x$ = 1.357 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 710 reflections
$\theta$ = 1.6–26.4°
$\mu$ = 0.26 mm$^{-1}$
$T$ = 293 (2) K
Block, yellow
0.39 × 0.30 × 0.20 mm

*Data collection*

Bruker SMART APEX CCD area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.852, $T_{max}$ = 0.953
6489 measured reflections
2519 independent reflections
1881 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.039
$\theta_{max}$ = 26.4°
$h = -17 \rightarrow 18$
$k = -13 \rightarrow 7$
$l = -17 \rightarrow 19$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.208
$S$ = 0.91
2519 reflections
179 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1396P)^2 + 2.1364P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$

**Table 1**
Selected torsion angles (°).

| | | | |
|---|---|---|---|
| C7—C8—C3—C2 | −178.7 (3) | O3—C11—C2—C1 | −179.8 (3) |
| C9—N1—C1—C2 | 178.7 (3) | C1—N1—C9—C10 | −179.9 (3) |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$n$···O2 | 0.83 (4) | 2.01 (4) | 2.683 (5) | 138 (4) |

Atoms C5 and C6 show positional disorder and the occupancy factors of two possible sites, C5*a*/C6*a* and C5*b*/C6*b*, are 0.58 (2) and 0.42 (2), respectively. The amine H atom was located in a difference Fourier map and refined isotropically. H atoms bonded to the C atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.96–0.97 Å and $U_{iso}$ = 1.2$U_{eq}$(C) or 1.5$U_{eq}$($C_{methyl}$).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; 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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