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

Vasu,^a K. A. Nirmala,^b Deepak Chopra,^{c*} S. Mohan^d and J. Saravanan^e

^aVivekananda Degree College, Bangalore 560 055, Karnataka, India, ^bDepartment of Physics, Bangalore University, Bangalore 560 056, Karnataka, India, ^cSolid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560 012, Karnataka, India, ^dPES College of Pharmacy, Hanumanthanagar, Bangalore 560 050, Karnataka, India, and ^eMS Ramaiah College of Pharmacy, Bangalore 560 054, Karnataka, India

Correspondence e-mail: deepak@sscu.iisc.ernet.in

The title compound, C₁₂H₁₅NO₃S, 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).

Key indicators

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

The molecular structure of (I) is shown in Fig. 1. The deviations of atoms C5a and C6a, 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 H1n 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

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

Data collection

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

Refinement

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

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, °).

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
N1—H1 μ ···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, C5a/C6a and C5b/C6b, 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 $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$.

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