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2-(Acetylamido)-*N*-(4-chlorophenyl)-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxamide

K. Chandrakumar,[a] M. K. Kokila,[b]* Puttaraja,[b] S. Mohan[c] and K. S. Manjunath Shetty[d]

[a]Department of Engineering Physics, HKBK College of Engineering, Nagawara, Bangalore 560 045, Karnataka, India, [b]Department of Physics, Bangalore University, Bangalore 560 056, Karnataka, India, [c]PES College of Pharmacy, Hanumanthanagar, Bangalore 560 050, Karnataka, India, and [d]M. S. Ramaiah Clinical and Bioavailability/Bioequivalence Centre, MSRIT Post, Bangalore 560 054, Karnataka, India

Correspondence e-mail:
prmkk_group@rediffmail.com

The cyclohexene ring of the title compound, $C_{17}H_{17}ClN_2O_2S$, shows disorder, with a 60% major conformer and a 40% minor conformer. Both adopt half-chair conformations. In addition to intramolecular N—H···N and C—H···O hydrogen bonds, there are intermolecular N—H···O hydrogen bonds and very weak C—H···N and C—H···O interactions.

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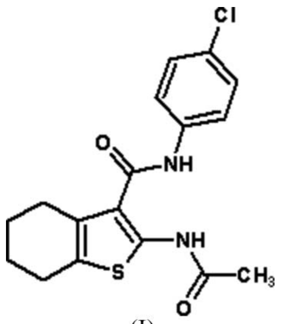
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.055
wR factor = 0.138
Data-to-parameter ratio = 13.2

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

Comment

The title compound, (I), is one of a series of 3-arylcarboxamides screened against Gram-positive and -negative bacteria, and which have shown promising antibacterial activity when compared with ampicillin (Mohan & Saravanan, 2003). Schiff bases (Csaszar & Morvay, 1983; Lakshmi *et al.*, 1985; Cohen *et al.*, 1977) and their thiophene derivatives (El-Maghraby *et al.*, 1984; Dzhurayev *et al.*, 1992; Gewald *et al.*, 1966), and a few tetrahydrobenzothiophenes with various groups at C2 and C3 (Vasu *et al.*, 2004; Chandra Kumar *et al.*, 2005), possess antibacterial, antitubercular and antifungal activity.



(I)

The molecular structure of (I) is shown in Fig. 1, and selected bond lengths and angles of the molecule are given in Table 1. In the thiophene ring, the shortest bond distance, C8—C9 [1.356 (4) Å], indicates the absence of delocalization of the double bonds, and this is reflected in the S1—C2 and S1—C8 bond distances, which are longer than normal C—S bonds.

The tetrahydrobenzothiophene ring system (S1/C2/C3/C9/C4–C7), including the thiophene ring, makes dihedral angles of 43.9 (1) and 21.2 (2)° with the planar carboxamide (O2/C10/N2) and acetamide (N1/C1/O1/C17) groups, respectively. The thiophene ring makes dihedral angles of 19.3 (2) and 5.5 (4)° with, respectively, the chlorophenyl and cyclohexene (C4–C9) rings.

The C1═O1 carbonyl group, where atoms C1 and O1 deviate by 0.129 and 0.637 Å, respectively, from the thiophene

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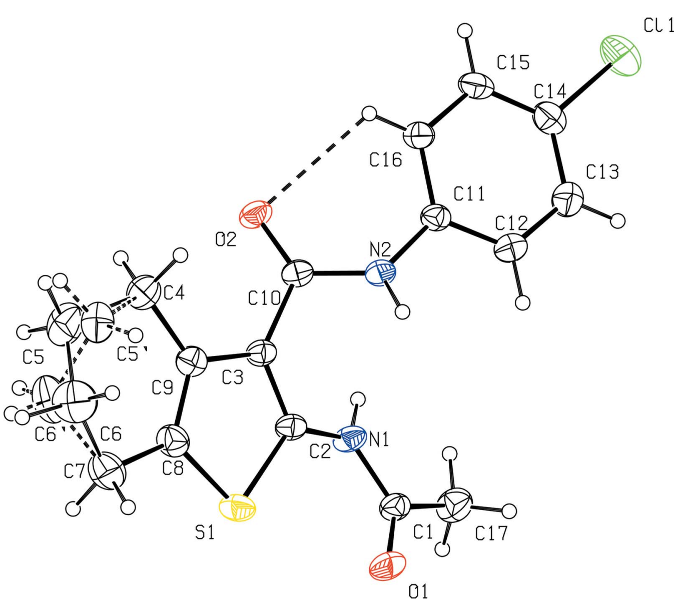

Figure 1
The structure of (I), showing the atom-numbering scheme and with displacement ellipsoids at the 30% probability level. Primes indicate the minor-site atoms and the dashed line indicates the intramolecular hydrogen bond.

ring, and the C1—N1—C2—S1 torsion angle of −27.8 (4)°, show a *cis* conformation for this part of the molecule. The C10═O2 bond and the C9—C3—C10—O2 torsion angle of −43.8 (4)° also define a *cis* conformation.

Intramolecular N—H⋯N and C—H⋯O hydrogen bonds (Table 2) form pseudo-six-membered rings, locking the molecular conformation and eliminating conformational flexibility. Molecules linked by N—H⋯O hydrogen bonds (Table 2) and very weak C—H⋯O and C—H⋯N interactions form zigzag chains along the [100] axis (Fig. 2).

Experimental

Compound (I) was synthesized by refluxing a mixture of cyclohexanone (0.98 g, 0.01 mol) and *p*-chloro-2-cyanoacetanilide (1.94 g, 0.01 mol) in the presence of diethylamine (4 ml). The mixture was heated at 325 K using a microwave oven, with the addition of sulfur powder (1.28 g, 0.04 mol) and ethanol (40 ml). The product was then mixed with acetic anhydride in the molar ratio 1:3 and heated using microwave irradiation, to yield a white solid, (I) (70%). This was then purified and crystallized from ethanol by slow evaporation.

Crystal data

$C_{17}H_{17}ClN_2O_2S$
$M_r = 348.84$
Monoclinic, $P2_1/n$
$a = 9.3999$ (18) Å
$b = 20.274$ (4) Å
$c = 9.7668$ (19) Å
$\beta = 111.764$ (3)°
$V = 1728.6$ (6) Å^3
$Z = 4$

$D_x = 1.340$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 540 reflections
$\theta = 2.0$–26.4°
$\mu = 0.35$ mm^{-1}
$T = 294$ (2) K
Prism, colourless
0.38 × 0.19 × 0.16 mm

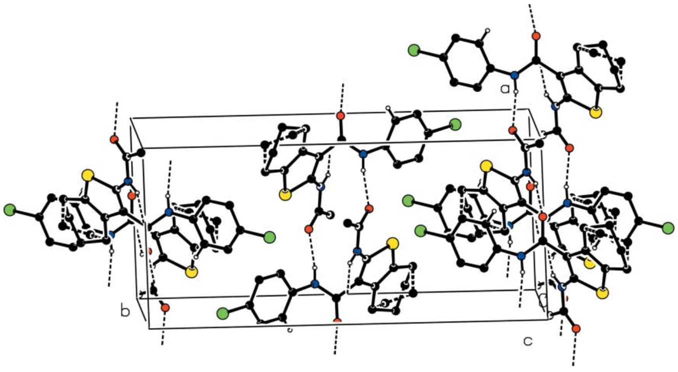

Figure 2
The crystal structure of (I). H atoms bonded to C atoms have been omitted for clarity. Dotted lines indicate N—H⋯O interactions.

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.925$, $T_{\max} = 0.946$
12791 measured reflections

3206 independent reflections
2553 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.034$
$\theta_{\max} = 25.5°$
$h = -11 \rightarrow 11$
$k = -24 \rightarrow 23$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.138$
$S = 1.09$
3206 reflections
243 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0675P)^2 + 0.6634P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.32$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C14	1.748 (3)	S1—C2	1.730 (3)
N1—C1	1.345 (3)	S1—C8	1.730 (3)
N2—C10	1.355 (3)	C8—C9	1.356 (4)
C1—N1—C2	125.5 (2)	C10—N2—C11	129.1 (2)
C1—N1—C2—S1	−27.8 (4)	C9—C3—C10—O2	−43.8 (4)

Table 2
Hydrogen-bond geometry (Å, °).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1⋯N2$^{\rm i}$	0.86	2.64	2.950 (2)	103
C16—H16⋯O2$^{\rm i}$	0.93	2.49	2.990 (3)	114
N1—H1⋯O2$^{\rm ii}$	0.86	2.05	2.863 (3)	158
N2—H2⋯O1$^{\rm iii}$	0.86	2.02	2.869 (3)	170

Symmetry codes: (i) x, y, z; (ii) $-x+1, -y, -z+2$; (iii) $-x+2, -y, -z+2$.

Disorder for atoms C5 and C6 was observed during anisotropic refinement of non-H atoms; atoms C5 and C6 were assigned occupancies of 0.6, and atoms C5′ and C6′ were assigned occupancies of 0.4. The H atoms attached to atoms C4 and C7 were found in difference Fourier maps and refined isotropically. All other H atoms of the molecule, including those belonging to the major and minor conformers of the cyclohexene ring, were placed in idealized positions [C—H = 0.93 for aromatic CH, 0.96 for methyl CH_3 and 0.97 Å for methylene CH_2; N—H = 0.86 Å] and constrained to ride on their parent atoms [U_{iso}(H) = 1.2U_{eq}(parent atom) for NH, aromatic CH and methylene CH_2; U_{iso}(H) = 1.5U_{eq}(parent atom) for methyl CH_3]. A rotating-group model was used for the methyl group.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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