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

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Key indicators

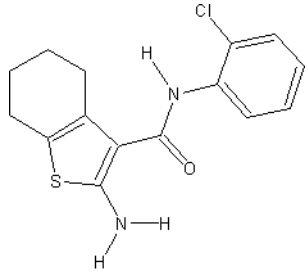
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.052
wR factor = 0.143
Data-to-parameter ratio = 10.2

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

The title compound, $C_{15}H_{15}ClN_2OS$, shows antibacterial and antifungal activities. The dihedral angle between the thiophene moiety and the 2-chlorophenyl ring is 22.3 (1)°. There are intramolecular N—H···O and N—H···Cl hydrogen bonds and an intramolecular C—H···O interaction, which remove the conformational flexibility. Also intermolecular N—H···O interactions form chains of molecules in the crystal structure.

Comment

Schiff bases (Csaszar & Morvay, 1983; Laksmi *et al.*, 1985; Cohen *et al.*, 1977) of thiophene compounds (El-Maghraby *et al.*, 1982; Dzhurayev *et al.*, 1992; Gewald *et al.*, 1966) contain structural motifs which find application in many pharmacologically active antibacterial, antitubercular and antifungal compounds. 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 and the packing diagram of (I) are shown in Figs. 1 and 2, respectively. The thiophene ring is essentially planar with atoms C6 and C7 deviating by 0.289 (4) and −0.359 (4) Å, respectively, from the plane. The C5—C6—C7—C8 torsion angle of 62.4 (4)° indicates that the cyclohexene ring has a half-chair conformation. The thiophene rings exhibit normal geometry. The dihedral angle between the thiophene moiety and the 2-chlorophenyl ring is 22.3 (1)°. The molecule is conformationally locked by intramolecular hydrogen bonds of the types N—H···O and C—H···O, forming a six-membered ring; there is also an intramolecular N—H···Cl interaction forming a five-membered ring. The crystal structure is stabilized by intermolecular N—H···O interactions, which link the molecules into chains running parallel to the *c* axis (Table 1 and Fig. 2).

Experimental

The title compound, (I), was synthesized by mixing cyclohexanone (0.98 g, 0.01 mol) and *o*-chlorocyanoacetanilide (1.94 g, 0.01 mol) and

refluxing the mixture for 1 h (Gewald *et al.*, 1966) in the presence of 4.0 ml of diethylamine. Sulfur powder (1.28 g, 0.04 mol) and 40 ml of ethanol were then added, and the resulting solution was stirred and heated for 1 h at 323 K. Crystals of (I) were grown by slow evaporation of a solution in *N*,*N*-dimethylformamide and ethanol (1:1) (yield 68%).

Crystal data

$C_{15}H_{15}ClN_2OS$
M_r = 306.81
Monoclinic, $P2_1/c$
a = 11.432 (3) Å
b = 14.722 (3) Å
c = 9.321 (2) Å
β = 113.320 (3)°
V = 1440.5 (6) Å^3
Z = 4
D_x = 1.415 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 750 reflections
θ = 1.8–25.4°
μ = 0.41 mm^{-1}
T = 293 (2) K
Block, yellow
0.50 × 0.30 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.823, T_{max} = 0.923
9632 measured reflections
2450 independent reflections
2074 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°
$h = -13 \rightarrow 13$
$k = -17 \rightarrow 17$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.143
S = 1.06
2450 reflections
241 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0706P)^2 + 1.144P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.67 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1n···O1	0.85 (3)	2.09 (3)	2.722 (4)	131 (3)
N2—H3n···Cl1	0.77 (4)	2.50 (4)	2.955 (3)	120 (3)
C15—H15···O1	0.91 (4)	2.22 (5)	2.854 (4)	126 (4)
N1—H2n···O1^{i}	0.84 (4)	2.22 (5)	3.062 (4)	173 (4)

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

All the H atoms were located and refined isotropically. The C—H and N—H bond lengths are 0.87 (5)–1.00 (4) and 0.76 (4)–0.85 (4) Å, respectively.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); 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).

We thank Professor T. N. Guru Row, Indian Institute of Science, Bangalore, for data collection on the CCD facility set up under the IRHPA–DST program and Bangalore University. Vasu thanks Vivekananda Degree College for support.

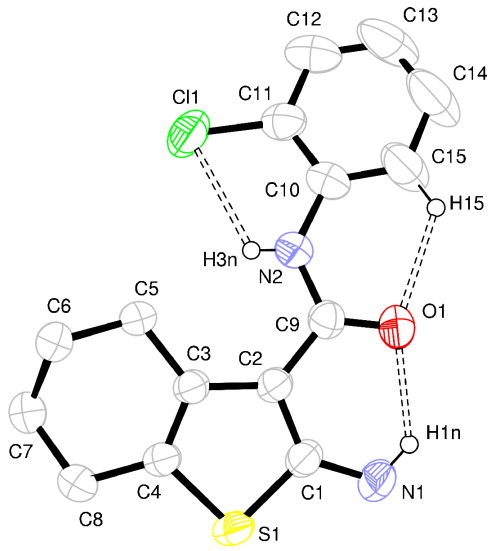


Figure 1
View of the molecule of (I), with displacement ellipsoids drawn at the 50% probability level. Only H atoms involved in intramolecular hydrogen bonds (dashed lines) are shown.

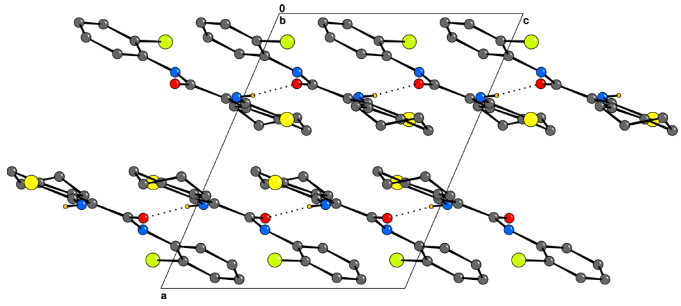


Figure 2
Packing diagram of (I), viewed along the *b* axis. Hydrogen bonds are shown as dashed lines. Only H atoms involved in intermolecular hydrogen bonds (dashed lines) are shown.

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