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2-Amino-*N*-(2-chlorophenyl)-5,6-dihydro-4*H*-cyclopenta[*b*]thiophene-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 = 0.057
wR factor = 0.130
Data-to-parameter ratio = 11.7

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

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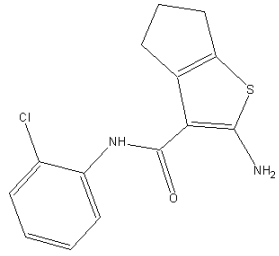
The title compound, $C_{14}H_{13}ClN_2OS$, shows antibacterial and antifungal activities. In the asymmetric unit there are two independent molecules, the dihedral angles between the thiophene moiety and the 2-chlorophenyl ring being 1.1 (3)° and 6.7 (3)°. There are intra- and intermolecular N—H···O hydrogen bonds.

Comment

Schiff bases (Csaszar & Morvay, 1983; Laksmi *et al.*, 1985; Cohen *et al.*, 1977) and their derivatives of thiophene (El-Meghraby *et al.*, 1982;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), exhibits the above-mentioned biological properties (Mohan & Saravanan, 2002, 2003).



(I)

The molecular structure and the packing diagram are shown in Figs. 1 and 2, respectively. The thiophene ring is essentially planar, with atoms C1 and C15 deviating by 0.089 (6) Å and

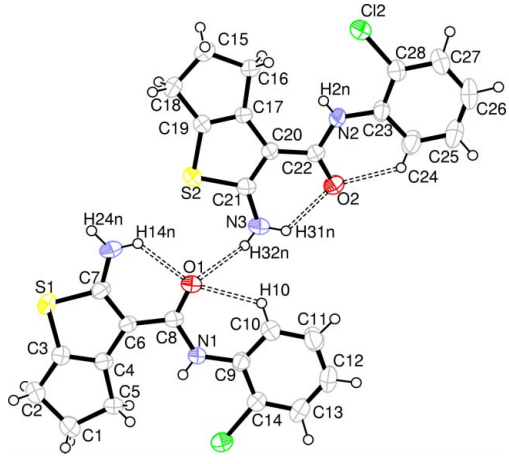


Figure 1
The structure of the asymmetric unit of (I), showing 50% probability ellipsoids. Open dashed bonds indicate N—H···O hydrogen bonds and C—H···O close contacts.

0.175 (4) Å from the plane, indicating that the cyclopentane ring has an envelope conformation. The thiophene rings exhibit normal geometry. The 2-chlorophenyl group is tilted from the thiophene ring in molecule *A* [C8—N1—C9—C10 = 9.22 (5)°], while it is almost coplanar with the thiophene ring in molecule *B* [C22—N2—C23—C24 = −1.26 (5)°]. Molecules *A* and *B* are linked *via* N3—H32N···O1 hydrogen bonds (Table 1). There is also an intramolecular N—H···O hydrogen bond, which locks each molecule into a rigid pseudo-six-membered-ring conformation and hence removes the conformational flexibility.

Experimental

The title compound, (I), was synthesized by mixing cyclopentanone (0.84 g, 0.01 mol) and *N*-(2-chlorophenyl)-2-cyanoacetamide (1.94 g, 0.01 mol) and refluxing for 1 h. To the resulting solution, 4.0 ml of diethylamine, sulfur powder (1.28 g, 0.04 mol) and 40 ml of ethanol were added, stirred and heated for 1 h at 323 K. Crystals of (I) were grown by slow evaporation using *N*,*N*-dimethylformamide and ethanol (1:1) as the solvents.

Crystal data

$C_{14}H_{13}ClN_2OS$
$M_r = 292.78$
Monoclinic, $P2_1/c$
$a = 9.939$ (3) Å
$b = 29.449$ (8) Å
$c = 9.747$ (3) Å
$\beta = 111.912$ (4)°
$V = 2646.8$ (14) Å^3
$Z = 8$
$D_x = 1.469$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 600 reflections
$\theta = 2.0$–24.5°
$\mu = 0.44$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.60 × 0.40 × 0.35 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{min} = 0.779$, $T_{max} = 0.862$
19431 measured reflections
5215 independent reflections
4005 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 26.4°$
$h = -11 \rightarrow 12$
$k = -36 \rightarrow 36$
$l = -12 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.130$
$S = 1.15$
5215 reflections
447 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 1.7113P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H24N···O2^{i}	0.86 (4)	2.27 (4)	3.094 (5)	162 (4)
N3—H32N···O1	0.83 (4)	2.39 (4)	3.186 (5)	162 (3)
N4—H14N···O1	0.86 (5)	2.13 (5)	2.755 (5)	129 (4)
N3—H31N···O2	0.88 (4)	2.14 (4)	2.752 (5)	126 (3)
C10—H10···O1	0.94 (3)	2.29 (4)	2.883 (5)	120 (2)
C24—H24···O2	0.90 (4)	2.24 (4)	2.875 (5)	127 (3)

Symmetry code: (i) $x-1, y, z$.

All the H atoms were located and refined isotropically. The C—H and N—H bond lengths are 0.89 (4)–1.01 (4) Å and 0.89 (4)–1.01 (4) Å, respectively.

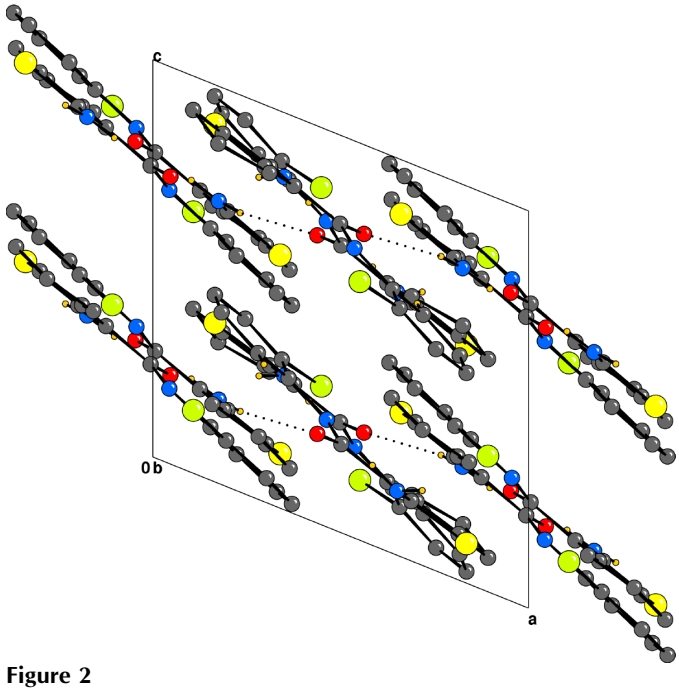


Figure 2
Packing diagram of (I), viewed down the *b* axis. The dotted lines indicate intermolecular N4—H24N···O2 hydrogen bonds between the molecules.

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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