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Key indicators

Single-crystal X-ray study
T = 296 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.043
wR factor = 0.127
Data-to-parameter ratio = 14.0

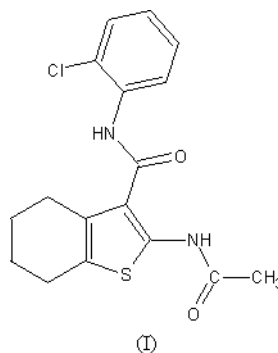
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

2-Acetamido-N-(2-chlorophenyl)-4,5,6,7-tetrahydrobenzothiophene-3-carboxamide

In the title compound, $\text{C}_{17}\text{H}_{17}\text{ClN}_2\text{O}_2\text{S}$, the *o*-chlorophenyl group is almost coplanar with the thiophene ring and the C—N bond distance indicates delocalization of π bonding across the ring systems. There are intramolecular N—H \cdots O, N—H \cdots Cl and C—H \cdots O hydrogen bonds, and intermolecular C—H \cdots O interactions.

Comment

The title compound, (I), is one of a series of 3-aryl-carboxamides. These compounds were screened against Gram-positive and Gram-negative bacteria, and 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 derivatives of thiophene (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) possess antibacterial, antitubercular and antifungal activity. Also, Schiff bases containing sulfur are the most effective. The structure of (I) has been determined in order to study the geometry of amide linkage and the hydrogen bonds in the molecule.



The molecular structure of (I) is shown in Fig. 1. In the thiophene ring, the C8—C9 bond distance [1.354 (3) Å] is the shortest (Table 1), indicating the absence of delocalization of the double bonds. This is also reflected in the S1—C2 and S1—C8 bond lengths, which are greater than the normal C—S distance. The tetrahydrobenzothiophene moiety is not planar, with atoms C5 and C6 above and below the mean plane of the thiophene ring. The *o*-chlorophenyl group is nearly coplanar with the thiophene moiety, making a dihedral angle of 1.5 (4)°. The C2—N1 bond [1.385 (3) Å] is shorter than C11—N2 [1.405 (3) Å], indicating greater delocalization of π bonding across the ring systems. Maximum deviations in the bond angles from the normal values are observed at atoms N1 and

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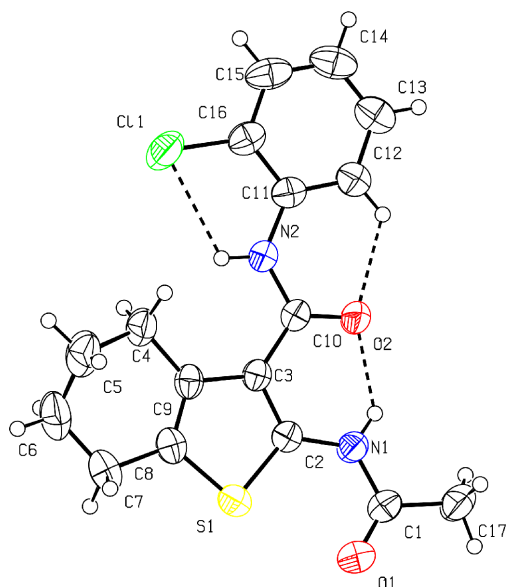


Figure 1

The structure of (I), showing displacement ellipsoids at the 50% probability level. Hydrogen bonds are shown as dashed lines.

N2, with $C10-N2-C11 = 128.86 (17)^\circ$ and $C2-N1-C1 = 125.57 (18)^\circ$. The $C1=O1$ carboxyl group is in the plane of the thiophene ring, and O1 is eclipsed with the ring S atom. The other carboxyl group, $C10=O2$, is antiperiplanar with the benzothiofene ring. Intramolecular $N1-H1 \cdots O2$ and $C-H \cdots O$ hydrogen bonds (Table 2) form pseudo-six-membered rings locking the molecular conformation and eliminating conformational flexibility. Molecules are linked via $C-H \cdots O$ interactions, forming zigzag chains along the c axis (Fig. 2).

Experimental

Compound (I) was synthesized by refluxing a mixture of cyclohexanone (0.98 g, 0.01 mol) and *o*-chlorocycanoacetanilide (1.94 g, 0.01 mol) in the presence of 4 ml of diethylamine. The mixture was heated at 325 K using microwaves, 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 (70%). This was purified and crystallized from ethanol by slow evaporation.

Crystal data

$C_{17}H_{17}ClN_2O_2S$	$D_x = 1.439 \text{ Mg m}^{-3}$
$M_r = 348.84$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 450 reflections
$a = 8.2523 (7) \text{ \AA}$	$\theta = 2.1-25.4^\circ$
$b = 19.6807 (16) \text{ \AA}$	$\mu = 0.38 \text{ mm}^{-1}$
$c = 10.2857 (8) \text{ \AA}$	$T = 296 (2) \text{ K}$
$\beta = 105.388 (1)^\circ$	Prism, colourless
$V = 1610.6 (2) \text{ \AA}^3$	$0.43 \times 0.19 \times 0.12 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	2948 independent reflections
φ and ω scans	2597 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.021$
$T_{\text{min}} = 0.920$, $T_{\text{max}} = 0.956$	$\theta_{\text{max}} = 25.4^\circ$
11 817 measured reflections	$h = -9 \rightarrow 9$
	$k = -23 \rightarrow 23$
	$l = -12 \rightarrow 12$

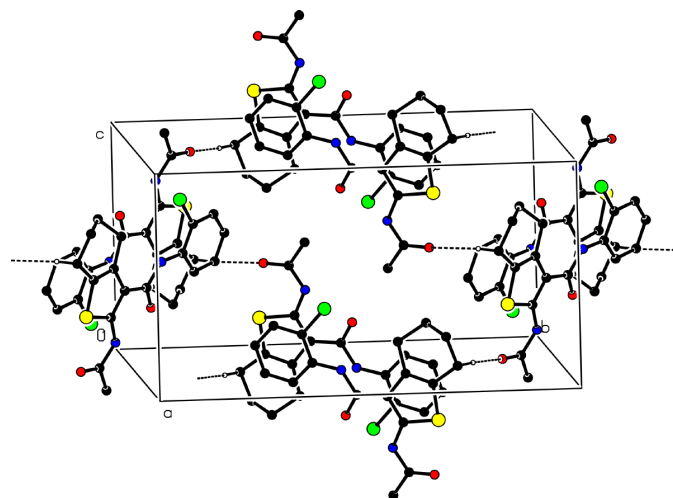


Figure 2

The crystal structure of (I). H atoms have been omitted for clarity. Dashed lines indicate $C-H \cdots O$ interactions.

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.127$
 $S = 1.05$
 2948 reflections
 210 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0719P)^2 + 0.7245P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.007$
 $\Delta\rho_{\text{max}} = 0.60 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0157 (19)

Table 1

Selected geometric parameters (\AA , $^\circ$).

S1—C2	1.717 (2)	N2—C11	1.405 (3)
S1—C8	1.726 (2)	C2—C3	1.384 (3)
O1—C1	1.209 (3)	C3—C9	1.452 (3)
O2—C10	1.233 (2)	C8—C9	1.354 (3)
N1—C2	1.385 (3)		
C1—N1—C2	125.57 (18)	C10—N2—C11	128.86 (17)
C1—N1—C2—S1	−6.7 (3)	C9—C3—C10—O2	171.7 (2)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1 \cdots O2	0.86	1.98	2.616 (2)	130
N2—H2 \cdots C11	0.86	2.47	2.948 (2)	116
C7—H7B \cdots O1 ⁱ	0.97	2.59	3.535 (3)	163
C12—H12 \cdots O2	0.93	2.22	2.820 (3)	121

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

H atoms were placed in idealized positions [$C-H = 0.93-0.97 \text{ \AA}$ and $N-H = 0.86 \text{ \AA}$] and constrained to ride on their parent atoms [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$, or $1.5U_{\text{eq}}(\text{parent atom})$ for methyl groups]. A rotating group model was used for the methyl groups.

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: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *PARST* (Nardelli, 1995), *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

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