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N-(3-Chlorophenyl)-2-{[(1*E*)-(4-methylphenyl)-methylene]amino}-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxamide

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The crystal structure of the title compound, $C_{23}H_{21}ClN_2OS$, is stabilized by intramolecular N—H···N, C—H···S and C—H···O hydrogen bonds and a weak intermolecular C—H···O interaction.

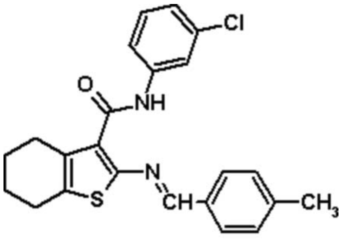
Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.124
Data-to-parameter ratio = 14.6

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

Comment

The title compound, $C_{23}H_{21}ClN_2OS$, (I), belongs to a series of 3-arylcarboxamides. The majority of 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) possess antibacterial, antitubercular and antifungal activities. Sulfur-containing Schiff bases are particularly effective (Mohan & Saravanan, 2003). In view of the pharmacological and biological activities of this class of compounds, the structure analysis of (I) has been undertaken.



(I)

The molecular structure of (I) is shown in Fig. 1, and selected geometrical parameters are given in Table 1. The

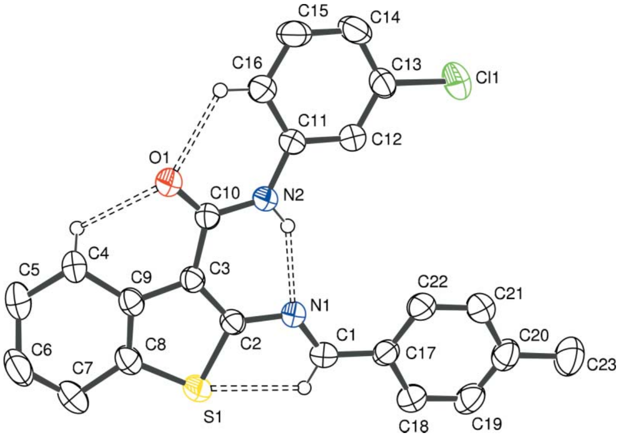


Figure 1
A drawing of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate intramolecular hydrogen bonds; H atoms not involved in hydrogen bonding have been omitted.

thiophene ring is planar. The six-membered cyclohexane ring adopts a half-chair conformation, with the puckering parameters q_2 = 0.383 (3) Å, φ_2 = −88.4 (4)° and θ_2 = 128.1 (3)° (Cremer & Pople, 1975; Spek, 2003).

The dihedral angle between the *p*-toluidine group (atoms C17–C23/C21/C22) and the thiophene ring is 10.2 (1)°. The *o*-chlorophenyl ring (C11–C16/Cl1) makes a dihedral angle of 4.8 (3)° with the thiophene ring. The C3—C10—N2—C11 [178.53 (17)°] and C2—N1—C1—C17 torsion angles [−176.78 (18)°] show the *anti* conformation of the two groups about the C10—N2 and N1—C1 bonds.

The intramolecular N—H···N and C—H···O hydrogen bonds form pseudo-six-membered rings, and the C—H···S hydrogen bond forms a pseudo-five-membered ring, thus locking the molecular conformation and eliminating conformational flexibility. In the crystal structure, molecules are linked by weak C—H···O interactions, forming along the *c* axis.

Experimental

The title compound, (I), was synthesized using the Gewald reaction (Gewald *et al.*, 1966). *m*-Chlorophenyl 2-cyanoacetamide (0.04 mol) was refluxed with cyclohexanone (0.98 g, 0.01 mol) in the presence of ammonium acetate (1.00 g) and glacial acetic acid (2 ml) in benzene. This mixture was treated with sulfur (1.28 g, 0.04 mol), dimethylamine (4 ml) and ethanol at 323 K. The product was treated with 4-methyl benzaldehyde in an equimolar ratio in the presence of 2-propanol and a catalytic amount of glacial acetic acid under microwave irradiation, which yielded (I). This was purified and crystallized from *N*,*N*-dimethylformamide and ethanol (1:2) by slow evaporation.

Crystal data

$C_{23}H_{21}ClN_2OS$
M_r = 408.93
Monoclinic, $P2_1/n$
a = 7.828 (3) Å
b = 19.652 (6) Å
c = 13.086 (4) Å
β = 98.854 (5)°
V = 1989.2 (11) Å^3
Z = 4

D_x = 1.365 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 470 reflections
θ = 1.2–25.5°
μ = 0.31 mm^{-1}
T = 291 (2) K
Block, yellow
0.34 × 0.24 × 0.21 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick 1996)
$T_{\min}$ = 0.915, $T_{\max}$ = 0.945
19713 measured reflections

3711 independent reflections
3209 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.023
$\theta_{\max}$ = 25.5°
$h = -9 \rightarrow 9$
$k = -23 \rightarrow 23$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.124
S = 1.09
3711 reflections
254 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0676P)^2 + 0.4901P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.26 e Å^{-3}
$\Delta\rho_{\min}$ = −0.16 e Å^{-3}

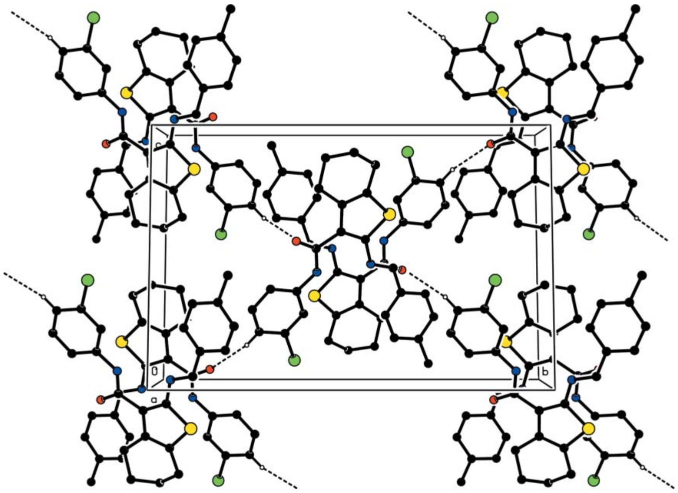


Figure 2
Packing diagram of (I); C—H···O hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

Table 1
Selected geometric parameters (Å, °).

S1—C8	1.719 (2)	N2—C10	1.359 (2)
S1—C2	1.7365 (19)	N2—C11	1.402 (2)
N1—C1	1.278 (2)	C8—C9	1.358 (3)
N1—C2	1.392 (2)		
C10—N2—C11	128.59 (16)	C22—C17—C18	118.04 (19)
C3—C2—N1	126.20 (16)	C19—C20—C21	117.8 (2)
C2—C3—C10	126.21 (16)		
C2—N1—C1—C17	−176.78 (18)	C11—N2—C10—C3	178.53 (17)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···N1	0.86	2.04	2.772 (3)	143
C1—H1···S1	0.93	2.53	2.997 (2)	112
C4—H4*A*···O1	0.97	2.49	2.830 (3)	100
C16—H16···O1	0.93	2.28	2.868 (3)	121
C14—H14···O1$^{\rm i}$	0.93	2.46	3.363 (3)	163

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

H atoms were positioned geometrically [N—H = 0.86 Å, and C—H = 0.93 (CH), 0.97 (CH_2) and 0.96 Å (CH_3)] and constrained to ride on their parent atoms with $U_{\rm iso}$(H) values of 1.2 (1.5 for methyl) times $U_{\rm eq}$(C,N). 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: *ORTEP-3 for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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o2842 Anilkumar *et al.* • $C_{23}H_{21}ClN_2OS$ *Acta Cryst.* (2005). E**61**, o2841–o2843

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Acta Cryst. (2005). E**61**, o2841–o2843 Anilkumar *et al.* • $C_{23}H_{21}ClN_2OS$ **o2843**