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2-(Acetamido)-4,5-dimethyl-*N*-(2-methylphenyl)thiophene-3-carboxamide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.123
Data-to-parameter ratio = 15.4

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

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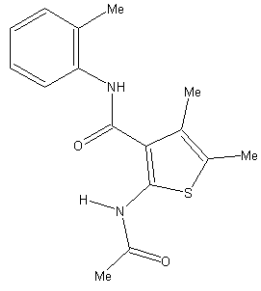
The title compound, $C_{16}H_{18}N_2O_2S$, shows antibacterial and antifungal activities. The dihedral angle between the thiophene and 2-methylphenyl groups is 83.3 (1)°. There are intra- and intermolecular N—H···O hydrogen bonds, and C—H···O intermolecular interactions.

Comment

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) possess antibacterial, antitubercular and antifungal properties. Sulfur-containing Schiff bases are the most effective. The title compound, (I), shows the above-mentioned biological properties (Mohan & Saravanan, 2002, 2003).



(I)

The molecular structure of (I) is shown in Fig. 1, and a packing diagram is shown in Fig. 2. The C2—C1—N2—C15 torsion angle is −170.81 (16)°, indicating that the acetamide group and the thiophene ring are essentialy planar (Table 1). The dihedral angle between the least-squares plane passing through the amide group (O1/C5/N1) and the 2-methylphenyl group is 60.9 (1)°, to avoid steric interaction between the methyl and carbonyl groups.

An intramolecular N—H···O hydrogen bond (Table 2) forms a pseudo-six-membered ring, which locks the molecular conformation and eliminates conformational flexibility. The crystal structure is further stabilized by N—H···O dimers and C—H···O chains running parallel to the *b* axis, which hold the dimers together to form 'chains of dimers'.

Experimental

The title compound was synthesized using the Gewald reaction (Gewald *et al.*, 1966). *o*-Cyanotoluidine (0.04 mol) was refluxed with ethyl methyl ketone (0.04 mol) in the presence of sulfur (0.04 mol), dimethylamine (4.0 ml) and ethanol (40 ml) at 323 K for 1 h. The product was mixed with acetic anhydride in the molar ratio 1:3 and heated in a beaker in a water bath for 1 h. The mixture was then

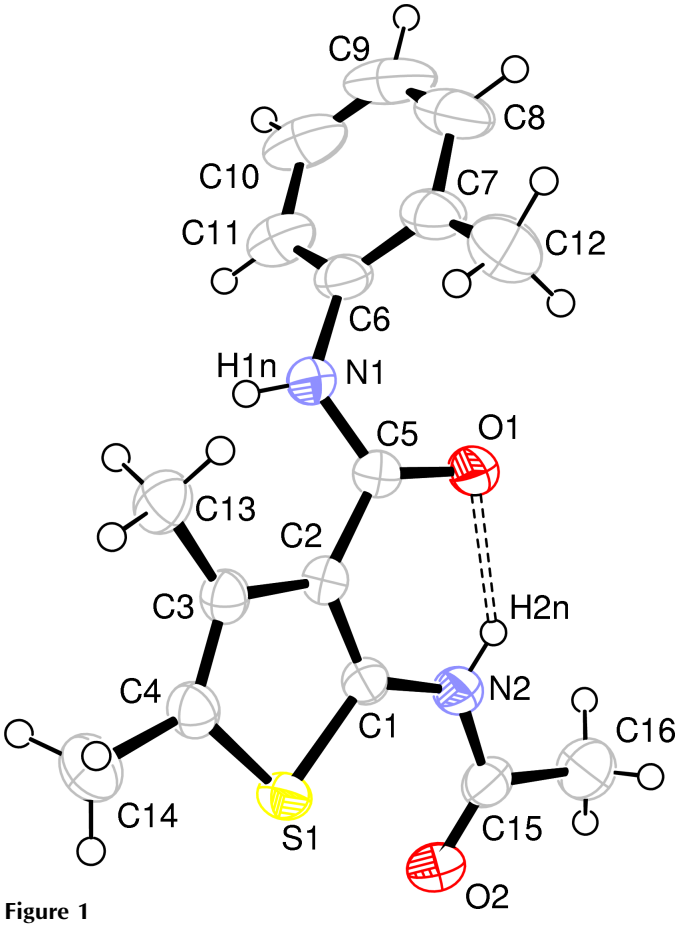


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate the intramolecular N—H···O hydrogen bond.

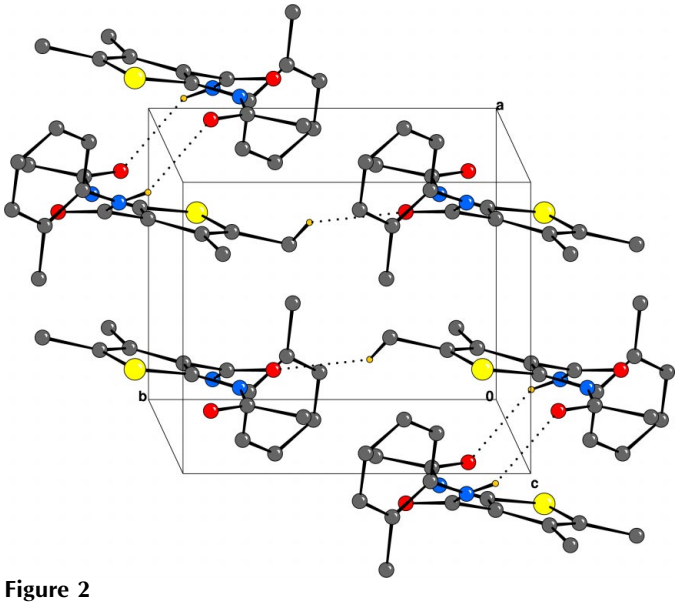


Figure 2
A packing diagram for (I). Dotted lines indicate N—H···O and C—H···O interactions. H atoms have been omitted for clarity.

cooled to room temperature and the solid which separated was filtered off. Crystals of (I) were obtained after recrystallization from ethanol (yield 72%).

Crystal data

$C_{16}H_{18}N_2O_2S$	D_x = 1.293 Mg m^{-3}
M_r = 302.39	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 650 reflections
a = 7.416 (2) Å	θ = 2.5–24.5°
b = 8.858 (3) Å	μ = 0.21 mm^{-1}
c = 23.718 (8) Å	T = 293 (2) K
β = 94.566 (6)°	Block, yellow
V = 1553.2 (9) Å^3	0.50 × 0.20 × 0.20 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3118 independent reflections
φ and ω scans	2715 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	R_{int} = 0.018
T_{min} = 0.910, T_{max} = 0.958	θ_{max} = 26.4°
11 711 measured reflections	$h = -8 \rightarrow 9$
	$k = -11 \rightarrow 11$
	$l = -29 \rightarrow 28$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1357P)^2 + 0.8P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.042	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.123	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
S = 1.06	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
3118 reflections	
202 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected torsion angles (°).

C15—N2—C1—C2	−170.81 (16)	C1—C2—C5—N1	154.78 (15)
C6—N1—C5—C2	−174.04 (16)	C5—N1—C6—C11	118.8 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2^{i}	0.786 (19)	2.46 (2)	3.155 (2)	148 (2)
N2—H2N···O1	0.829 (18)	2.029 (19)	2.692 (2)	137 (2)
C14—H14C···O1^{ii}	0.96	2.575	3.355 (3)	138

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

Amine H atoms were located in difference Fourier maps and refined isotropically. Methyl H atoms were constrained to an ideal geometry [C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C)], but were allowed to rotate freely about the C—C bond. All benzene H atoms were placed in idealized positions (C—H = 0.93 Å) and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C).

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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