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4,5-Dimethyl-*N*-(2-methylphenyl)-2-{[(1*E*)-(3,4,5-trimethoxyphenyl)methylene]amino}-thiophene-3-carboxamide

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Key indicators

Single-crystal X-ray study
T = 290 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.123
Data-to-parameter ratio = 16.1

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

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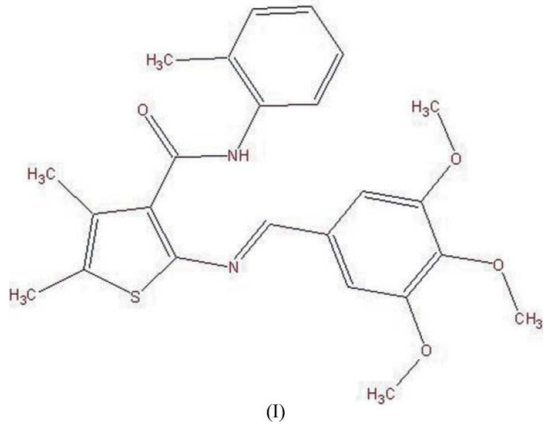
The conformation of the title molecule, $C_{24}H_{26}N_2O_4S$, is influenced by intramolecular N—H···N hydrogen bonding, and π–π interactions link the molecules into centrosymmetric dimers. The crystal packing is further stabilized by weak intermolecular C—H···O hydrogen bonds.

Comment

The title compound, (I), shows promising antibacterial activity against *subtilis aureus* and *Escherichia coli*, comparable with the activity of ampicillin (Mohan & Saravanan *et al.*, 2002, 2003). We present here the crystal structure of the title compound, (I).



(I)

In (I) (Fig. 1), all bond lengths and angles show normal values (Allen *et al.*, 1987). An intramolecular N—H···N hydrogen bond (Table 1) forms a pseudo-six-membered ring with graph-set notation *S*(6) (Bernstein *et al.*, 1995) and influences the molecular conformation. The *o*-toluidine is *gauche* to the thiophene ring, the dihedral angle being 59.24 (2)°. The geometric parameters of the methoxy groups correspond to those observed in methoxybenzene derivatives (Fun *et al.*, 1997).

The relatively short *Cg*1···*Cg*2^{iii} distance of 3.683 (3) Å (*Cg*1 and *Cg*2 are centroids of the S1/C1–C4 and C16–C21 rings, respectively) shows the presence of π–π interactions which link the molecules into centrosymmetric dimers [symmetry code: (iii) $-x+2, -y+1, -z$]. The crystal packing (Fig. 2) is further stabilized by weak intermolecular C—H···O hydrogen bonds (Table 1).

Experimental

The title compound was synthesized using the Gewald reaction (Gewald *et al.*, 1966). *o*-Cyanotoluidine (0.04 mol) was refluxed with

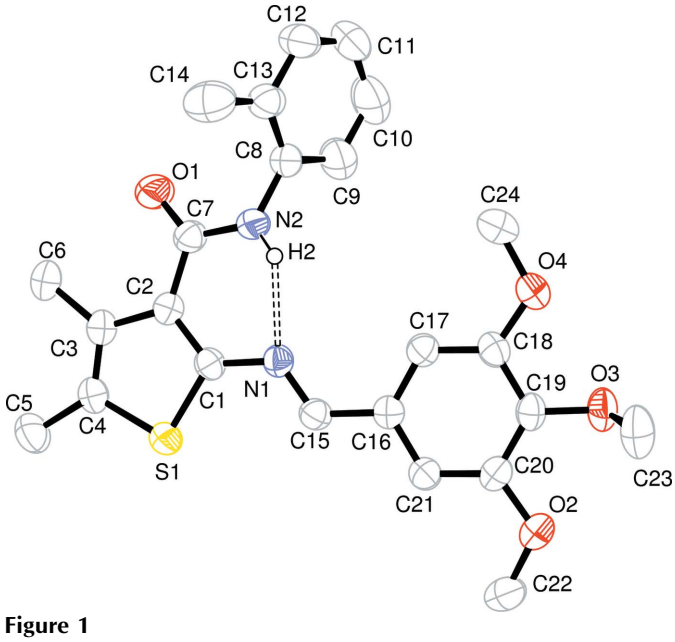


Figure 1
The molecular structure of (I), showing the atomic labeling and 50% probability displacement ellipsoids. The double dashed line denotes the N—H···N hydrogen bond. All H atoms, except H2, have been omitted for clarity.

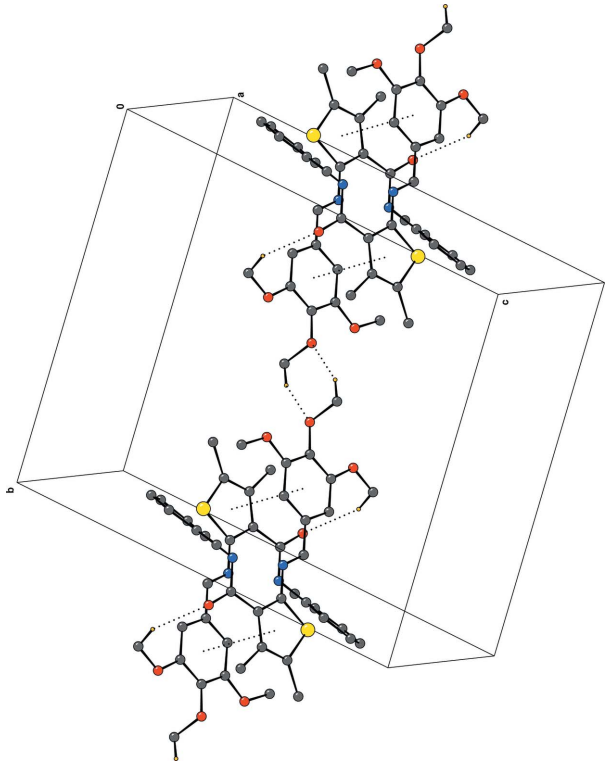


Figure 2
A portion of the crystal packing of (I). The dotted lines denote π–π interactions and C—H···O hydrogen bonds. H atoms not involved in hydrogen bonds have been omitted for clarity.

ethyl methyl ketone in the presence of sulfur (0.04 mol), dimethylamine (4 ml) and ethanol (40 ml) at 313–323 K for 1 h. The product was then reacted with 3,4,5-trimethoxybenzaldehyde in an equimolar ratio in the presence of ethanol, which yielded the title compound. This was then purified and crystallized from dimethylformamide and ethanol (1:2) by slow evaporation, yielding orange block-shaped crystals.

Crystal data

$C_{24}H_{26}N_2O_4S$
M_r = 438.54
Monoclinic, $P2_1/n$
a = 7.1802 (15) Å
b = 16.064 (3) Å
c = 19.354 (4) Å
β = 90.123 (3)°
V = 2232.3 (8) Å^3
Z = 4
D_x = 1.305 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 290 (2) K
Block, orange
0.30 × 0.25 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.909, T_{max} = 0.957
17378 measured reflections
4661 independent reflections
3427 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 27.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.123
S = 1.00
4661 reflections
290 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0751P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···N1	0.86	2.10	2.737 (2)	130
C22—H22C···O1^{i}	0.96	2.57	3.459 (3)	155
C23—H23B···O3^{ii}	0.96	2.56	3.424 (3)	150

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

All H atoms were located in a difference Fourier map, placed in idealized positions (C—H = 0.93–0.96 and N—H = 0.86 Å) and refined as riding, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; 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: *PLATON* (Spek, 2003).

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