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Methyl 2-{[(2*E*)-3-phenylprop-2-enoyl]amino}-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.126
Data-to-parameter ratio = 12.3

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

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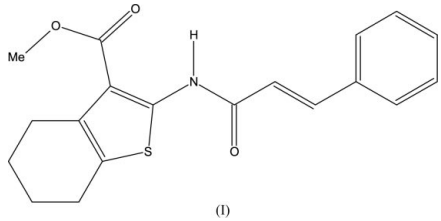
In the title compound, $C_{19}H_{19}NO_3S$, the thiophene ring is almost coplanar with the cinnamide moiety. An intramolecular N—H···O hydrogen bond forms a pseudo-six-membered ring, thus locking the molecular conformation and removing the conformational flexibility. In addition, the packing of molecules is stabilized by C—H···O interactions which form dimers.

Comment

Most Schiff bases (Pellis & West, 1968; Cohen *et al.*, 1977; Csaszar & Morvay, 1983; Lakshmi *et al.*, 1985) and their thiophene derivatives (El-Maghraby *et al.*, 1984; Dzhurayev *et al.*, 1992; Gewald *et al.*, 1966) possess pharmacological activity such as antibacterial, anticancer, anti-inflammatory and antitoxic. Sulfur-containing Schiff bases are most effective. The title compound, (I), is found to exhibit antibacterial and antifungicidal activities (Mohan & Saravanan, 2002, 2003). In view of the medicinal application of such classes of compounds, a single-crystal study has been carried out on the title compound.



(I)

The bond lengths and angles in the cinnamoyl part of the molecule are similar to the reported values [C13—C14, C12—C13 and C11—C12 = 1.467 (2), 1.315 (3) and 1.475 (2) Å, respectively, compared with 1.467 (2), 1.329 (3) and 1.485 (2) Å (Schmidt, 1964; Iwamoto *et al.*, 1989)]. The molecule is almost planar. The torsion angle C11—N1—C1—S1 is 7.4 (3)°, indicating that the plane of the thiophene ring is almost coplanar with the cinnamide moiety. The bond angle C14—C13—C12 of 126.6 (2)° is widened because of steric repulsion between the atoms H12 and H15. The angle between the mean planes of the cinnamoyl group and thiophene ring in the molecule is 9.94 (6)°. The observed dihedral angle between the vinyl moiety and the plane of the phenyl ring is 14 (2)° due to intramolecular H(*ortho*)···H(ethylenic) repulsion (Leiserowitz & Tuval, 1978). There is an intermolecular C19—H19···O3($2-x$, $1-y$, $2-z$) hydrogen bond, leading to formation of dimers. There is also an intramolecular N1—H1N···O1 hydrogen bond, leading to the formation of a conformationally locked pseudo-six-membered ring.

Experimental

The title compound was synthesized by mixing cyclohexanone (0.04 mol), ethyl cyanoacetate (0.04 mol), sulfur (0.04 mol) and 40 ml of ethanol and stirring the mixture at 325 K for 1 h with dropwise addition of 4 ml of dimethylamine to yield the ester. Alkaline hydrolysis of the ester group using sodium hydroxide solution yielded the acid which, on treatment with 3 mol of cinnamoyl chloride in presence of dioxan, yielded the final compound, (I). Single crystals of suitable quality were grown using a mixture of dimethylformamide and ethanol by slow evaporation at room temperature.

Crystal data

$C_{19}H_{19}NO_3S$
M_r = 341.42
Monoclinic, $P2_1/n$
a = 9.811 (3) Å
b = 9.590 (3) Å
c = 18.870 (5) Å
β = 102.893 (5)°
V = 1730.7 (9) Å^3
Z = 4
D_x = 1.310 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 575 reflections
θ = 1.5–21.5°
μ = 0.20 mm^{-1}
T = 293 (2) K
Prism, yellow
0.30 × 0.25 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.870, T_{max} = 0.961
13095 measured reflections
3600 independent reflections
2765 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 27.3°
$h = -12 \rightarrow 12$
$k = -11 \rightarrow 12$
$l = -23 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$) = 0.045
$wR(F^2)$ = 0.126
S = 1.04
3600 reflections
293 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0709P)^2 + 0.2357P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C19—H19···O3^{i}	0.88 (2)	2.62 (2)	3.455 (2)	159 (2)
N1—H1N···O1	0.83 (2)	2.03 (2)	2.689 (2)	136 (2)

Symmetry code: (i) $2-x, 1-y, 2-z$.

All H atoms were located and refined isotropically. The C—H and N—H bond lengths are 0.91 (4)–1.02 (3) and 0.83 (2) Å, respectively.

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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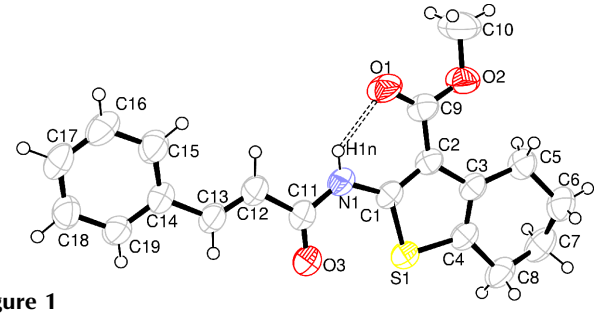

Figure 1
ORTEP diagram, with 50% probability displacement ellipsoids. Dashed lines indicate the N—H···O hydrogen bond.

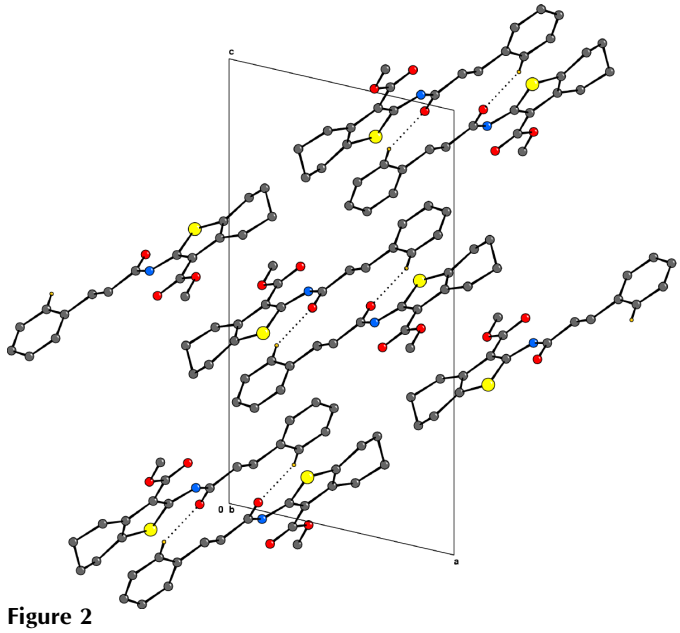

Figure 2
Packing diagram, viewed down the *b* axis, showing the C—H···O dimers. Other H atoms have been omitted.

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