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4-[(5-Nitrothiophen-2-ylmethylene)amino]-5-(thiophen-2-ylmethyl)-1*H*-1,2,4-triazol-5(4*H*)-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.057
wR factor = 0.127
Data-to-parameter ratio = 12.9

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

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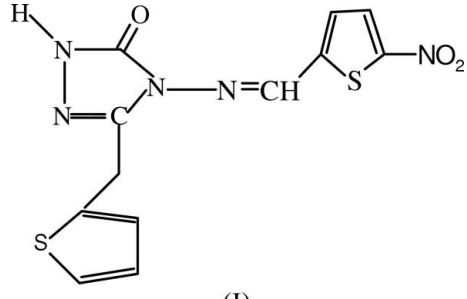
The title compound, $C_{12}H_9N_5O_3S_2$, contains three planar rings. The triazole ring makes a dihedral angle of 3.88 (15)° with the nitrothiophene ring. Intra- and intermolecular hydrogen bonding is effective in stabilizing the crystal structure.

Comment

1,2,4-Triazole compounds possess important pharmacological activities, such as antifungal and antiviral properties. Examples of such compounds having 1,2,4-triazole residues are fluconazole (Tsukuda *et al.*, 1998), the powerful azole antifungal agent, and the potent antiviral N-nucleoside ribavirin (Witkoaski *et al.*, 1972). Furthermore, various 1,2,4-triazole derivatives have been reported as having fungicidal (Heubach *et al.*, 1979), insecticidal (Tanaka, 1975) and antimicrobial activities (Griffin & Mannion, 1986), and some have shown antitumour activity (Hanna *et al.*, 1988). Other compounds in this class act as anticonvulsants (Husain & Amir, 1986), antidepressants (Chiu & Huskey, 1998), plant-growth regulators and anticoagulants (Eliott *et al.*, 1986). Other laboratories have reported the biological activity of the triazole family (Chaaban & Oji, 1984; Omar & AboulWafa, 1984). Other compounds incorporating a 1,2,4-triazole ring, with diverse pharmacological effects, have been reported as therapeutic agents in medicinal chemistry (Demirayak *et al.*, 2000), and several of these compounds have been shown to be antitumour agents. Some of them also incorporate a Schiff base structure (İkizler *et al.*, 2000).



(I)

The title compound, (I), contains three planar rings (Fig. 1), namely a triazole ring (*A*) and two thiophene rings, C1–C4/S1 (ring *B*) and C9–C12/S2 (ring *C*). The maximum atomic deviations for rings *A*, *B* and *C* from their individual planes are 0.0032 (5) Å for atom C6, 0.0136 (4) Å for atom C3 and 0.0071 (5) Å for atom C12, respectively. The dihedral angles between rings *A*/*B*, *A*/*C* and *B*/*C* are 70.98 (17), 3.88 (15) and 67.23 (16)°, respectively.

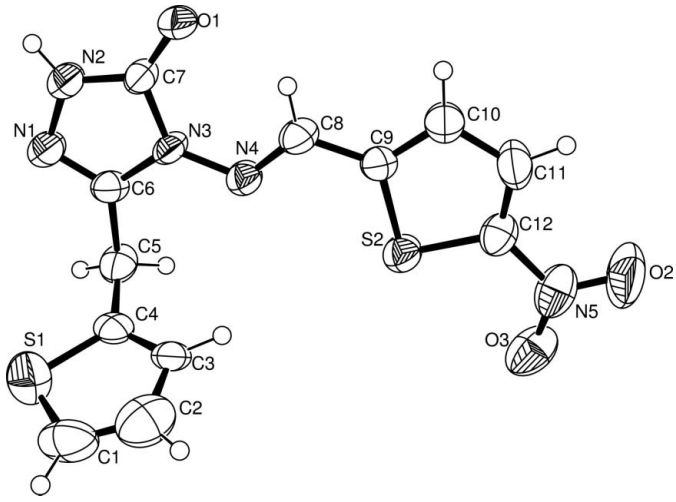


Figure 1
The molecular structure of (I), with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

In the thiophene ring, the S2—C9 bond is longer than the S2—C12 bond (Table 1). These S—C distances are in agreement with those found for other structures containing thiophene, such as 1,5-bis(3-thienyloxy)-3-oxapentane [1.701 (2) and 1.716 (3) Å; Labat & Halfpenny, 2005*a*] and 1,2-bis(3-thienyloxy)ethane [1.7129 (14) and 1.7178 (13) Å; Labat & Halfpenny, 2005*b*]. The C6—N3 and C7—N3 bond distances are longer than C7—N2 (Table 1), because of the substitution at atom N3. The N1—N2 bond length is close to that reported for similar compounds [1.403 (8) Å (Sancak *et al.*, 2005) and 1.408 (2) Å (Ciunik *et al.*, 2002)].

The crystal structure of (I) is stabilized by two intermolecular hydrogen bonds and one intramolecular hydrogen bond (Table 2).

Experimental

4-Amino-3-thiophen-2-yl-methyl-4,5-dihydro-1*H*-[1,2,4]triazole-5-one (0.01 mol) and 5-nitrothiophene-2-carboxyaldehyde (0.01 mol) were heated at 433–438 K in an oil bath for 2 h. After cooling to room temperature, a solid appeared and this was recrystallized from a solution in dimethyl sulfoxide–water (1:2) to give compound (I) (yield 82.39%).

Crystal data

$C_{12}H_9N_5O_3S_2$
M_r = 335.36
Monoclinic, $P2_1/c$
a = 8.2416 (9) Å
b = 16.9464 (19) Å
c = 10.9065 (12) Å
β = 106.663 (3)°
V = 1459.3 (3) Å^3
Z = 4
D_x = 1.526 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.39 mm^{-1}
T = 293 (2) K
Plate, yellow
0.23 × 0.20 × 0.09 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
6681 measured reflections
2564 independent reflections
1113 reflections with $I > 2\sigma(I)$
R_{int} = 0.109
θ_{max} = 25.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.127
S = 0.83
2564 reflections
199 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0436P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Selected bond lengths (Å).

S2—C12	1.710 (5)	N3—C7	1.401 (5)
S2—C9	1.724 (4)	C7—N2	1.328 (5)
N3—C6	1.385 (5)	N1—N2	1.398 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H3···O1^{i}	0.86	1.97	2.793 (5)	161
C8—H8···O1	0.93	2.13	2.847 (6)	132
C11—H11···O3^{ii}	0.93	2.26	3.184 (6)	170

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

The crystal was poorly diffracting, which explains the low ratio of observed to unique reflections of 0.43. All H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H = 0.93 (aromatic) or 0.97 Å (methylene) and with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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References

Bruker (1997). *SADABS* (Version 2.10), *SMART* (Version 5.044) and *SAINT* (Version 5.01). Bruker AXS Inc., Madison, Wisconsin, USA.
Chaaban, I. & Oji, O. O. J. (1984). *Indian Chem. Soc.* **61**, 523–525.
Chiu, S.-H. L. & Huskey, S.-E. W. (1998). *Drug. Metab. Dispos.* **26**, 838–847.
Ciunik, Z., Drabent, K. & Szterenberg, L. (2002). *J. Mol. Struct.* **641**, 175–182.
Demirayak, Ş., Benkli, K. & Güven, K. (2000). *Eur. J. Chem.* **35**, 1037–1040.
Eliott, R., Sunley, R. L. & Griffin, D. A. (1986). UK Patent Appl. GB 2 175 301.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Griffin, D. A. & Mannion, S. K. (1986). European Patent Appl. EP 199 474.
Hanna, N. B., Dimitrijevich, S. D., Larson, S. B., Robsin, R. K. & Revankar, G. R. (1988). *J. Heterocycl. Chem.* **25**, 1857–1868.
Heubach, G., Sachse, B. & Buerstell, H. (1979). *Ger. Off.* **2**, 826–876.
Husain, M. I. & Amir, M. (1986). *J. Indian Chem. Soc.* **63**, 317–319.
İkizler, A. A., Uzunali, E. & Demirbaş, A. (2000). *Indian J. Pharm.* **5**, 289–292.
Labat, G. & Halfpenny, J. (2005*a*). *Acta Cryst.* E**61**, o2813–o2814.
Labat, G. & Halfpenny, J. (2005*b*). *Acta Cryst.* E**61**, o2815–o2816.
Omar, A. M. E. & AboulWafa, O. M. (1984). *J. Heterocycl. Chem.* **21**, 1415–1417.
Sancak, K., Çoruh, U., Ünver, Y. & Vázquez-López, E. M. (2005). *Acta Cryst.* E**61**, o1785–o1787.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Tanaka, G. (1975). Jpn Kokai 973 7495.
Tsukuda, T., Shiratori, Y., Watanabe, M., Ontsuka, H., Hattori, K., Shirai, M. & Shimma, N. (1998). *Bioorg. Med. Chem. Lett.* **8**, 1819–1824.
Witkoaski, J. T., Robins, R. K., Sidwell, R. W. & Simon, L. N. (1972). *J. Med. Chem.* **15**, 1150–1154.