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3,3′-Bis(2-thienylmethyl)-4,4′-(butane-1,4-diyl)-bis(4,5-dihydro-1*H*-1,2,4-triazol-5-one)

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.102
Data-to-parameter ratio = 17.1

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

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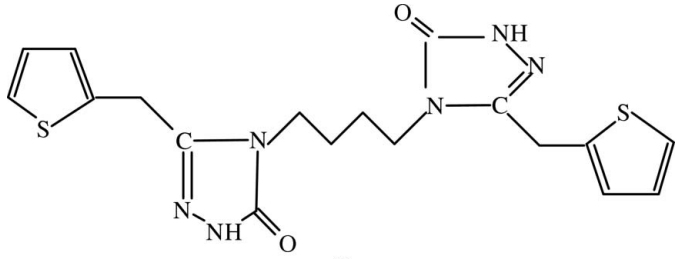
The title compound, $C_{18}H_{20}N_6O_2S_2$, has a centre of symmetry. The thiophene ring makes an angle of 70.58 (6)° with the triazole ring. Molecules are linked *via* N—H···O hydrogen bonds, forming a three-dimensional network.

Comment

1,2,4-Triazole compounds possess important pharmacological properties such as antifungal and antiviral activities. Examples of such compounds bearing the 1,2,4-triazole residue 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 showing fungicidal (Heubach *et al.*, 1979), antimicrobial (Griffin & Mannion, 1986) and antitumor activity (Hanna *et al.*, 1988), as well as having applications as anticonvulsants (Husain & Amir, 1986), antidepressants (Chiu & Huskey, 1998) and plant growth regulator anticoagulants (Eliott *et al.*, 1986). In the present paper, we report the structure of the title compound, (I).



(I)

In (I), the molecule has a centre of symmetry at the midpoint of the central C—C bond (Fig. 1). The 1,2,4-triazole ring is planar. The C6—N3 and C7—N3 bond distances are longer than C7—N2 (Table 1), because atom N3 has an alkyl substituent. The N1—N2 bond length is close to that reported for a similar compound [1.3823 (17) Å; Ocak Ískeleli *et al.*, 2005]. The dihedral angle between the thiophene (S1/C1–C4) and triazole (N1/N2/C7/N3/C6) rings is 70.58 (6)°. The molecules are linked *via* N—H···O hydrogen bonds (Table 2), forming a three-dimensional network.

Experimental

To a solution of *N*′-(1-ethoxy-2-thiophen-2-yl-ethylidene)hydrazine carboxylic acid ethyl ester (5.12 g, 0.02 mol) in water (50 ml), 1,4-diaminobutane (0.88 g, 0.01 mol) was added and refluxed for 4 h. The resulting precipitate, (I), was filtered off and washed with water. Crystals of (I) were grown from ethanol/water (1:1 *v*/*v*) solution by

slow evaporation for 7 d at room temperature (yield 69.7%, m.p. 528–529 K). IR (KBr, ν, cm^{-1}): 3188 (NH), 1701 (C═O), 1577 (C═N). ^{1}H NMR (DMSO-d_6): δ 1.21 (*bs*, 2CH$_2$), 3.40 (*bs*, 2NCH$_2$), 4.13 (4 H, 2CH$_2$, thiophene), 6.93–7.42 (*m*, 6 H, 6CH ABC system, for two thiophene ring), 11.58 (2 H, *s*, 2NH). ^{13}C NMR (DMSO-d_6): δ 25.21 (CH$_2$), 26.08 (thiophene-CH$_2$), 40.21 (–NCH$_2$), 126.92, 126.49, 126.92 (thiophene CH), 137.56 (thiophene C), 145.59 (triazole C-3), 154.87 (triazole C-5).

Crystal data

$C_{18}H_{20}N_6O_2S_2$	$Z = 2$
$M_r = 416.54$	$D_x = 1.470$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 9.6148$ (15) Å	$\mu = 0.31$ mm^{-1}
$b = 7.2699$ (11) Å	$T = 173$ (2) K
$c = 13.523$ (2) Å	Prism, colourless
$\beta = 95.289$ (3)°	0.30 × 0.22 × 0.13 mm
$V = 941.2$ (2) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2175 independent reflections
φ and ω scans	1491 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{int} = 0.061$
5511 measured reflections	$\theta_{max} = 28.0°$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.043$	$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.102$	$(\Delta/\sigma)_{max} = 0.001$
$S = 0.92$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
2175 reflections	$\Delta\rho_{min} = -0.34$ e Å^{-3}
127 parameters	

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.235 (2)	N3—C6	1.380 (2)
C8—N3	1.460 (2)	N3—C7	1.385 (2)
S1—C1	1.706 (2)	N1—N2	1.388 (2)
S1—C4	1.723 (2)	C7—N2	1.348 (3)
C7—N3—C6—N1	0.3 (2)	N3—C7—N2—N1	0.8 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2′···O1^{i}	0.86	1.91	2.737 (2)	161

Symmetry code: (i) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$.

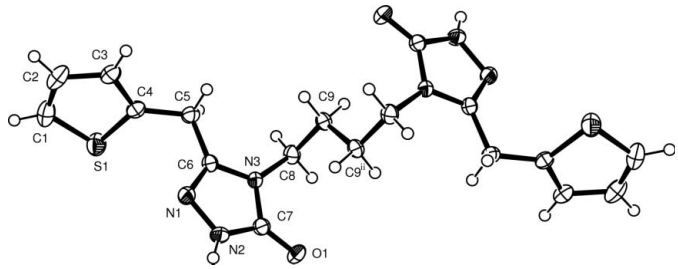


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 70% probability level. [Symmetry code: (ii) $2-x, 1-y, 1-z$.]

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N).

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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