

4-(2-Pyridylmethyleneamino)-3-(2-thienylmethyl)-1*H*-1,2,4-triazol-5(4*H*)-oneIsil Yılmaz,^{a*} N. Burcu Arslan,^a
Canan Kazak,^a Kemal Sancak^b
and Yasemin Unver^b^aDepartment of Physics, Faculty of Art and Science, Ondokuz Mayıs University, 55139 Samsun, Turkey, and ^bDepartment of Chemistry, Faculty of Art and Sciences, Karadeniz Technical University, Trabzon, Turkey

Correspondence e-mail: isil_ylmaz@yahoo.com

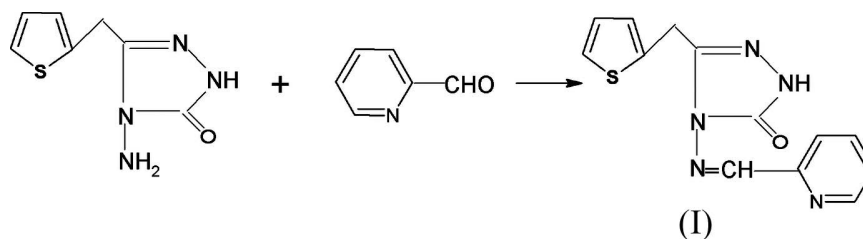
Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.043
wR factor = 0.107
Data-to-parameter ratio = 14.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.In the title compound, $\text{C}_{13}\text{H}_{11}\text{N}_5\text{OS}$, molecules are linked into centrosymmetric dimers through intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

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Comment

Azole derivatives, such as derivatives of pyrazole, imidazole, triazole (including benzotriazole), tetrazole, indole *etc.*, exhibit extensive biological activities. They have become the central focus in the study of agricultural chemicals, adjustment reagents for plant growth and so on (Haddock & Hopwood, 1982). Triazole ring systems are typical planar 6π -electron partially aromatic systems, and 1,2,4-triazole and its derivatives are used as starting materials for the synthesis of many heterocycles (Desenko, 1995). Recently, much attention has been focused on 1,2,4-triazole derivatives for their broad-spectrum activities, such as fungicidal, insecticidal, herbicidal, anticonvulsant, antitumour and plant growth regulatory activities (Jenkins *et al.*, 1989; Er-Rahimini & Mornet, 1992; Nakib *et al.*, 1994; Chai *et al.*, 2003; Tsuda *et al.*, 2004).In the title compound, (I), the thiophene ring presents C—S bond lengths similar to each other (Table 1) and also to other values reported in the literature (Vrábel *et al.*, 2005). The C6=N1 bond is clearly a double bond, being much shorter than the other C—N bonds in the triazole ring. This distance is also comparable to literature data (Çoruh *et al.*, 2003; Yılmaz *et al.*, 2005). The crystal structure features centrosymmetric dimers, formed by classical intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, involving the amine functionality and the carbonyl of the triazole ring (Table 2).

Experimental

4-Amino-5-thiophen-2-ylmethyl-2,4-dihydro-1,2,4-triazol-3-one (0.196 g, 1 mmol) was mixed with pyridine-2-carbaldehyde (0.09 ml, 0.107 g, 1 mmol) at 373–383 K for 1 h. The resulting solid crude product was crystallized from alcohol–water. The crystals (0.22 g, yield 81.4%) were improved by crystallizing several times from the same solvent mixture and were dried *in vacuo*.

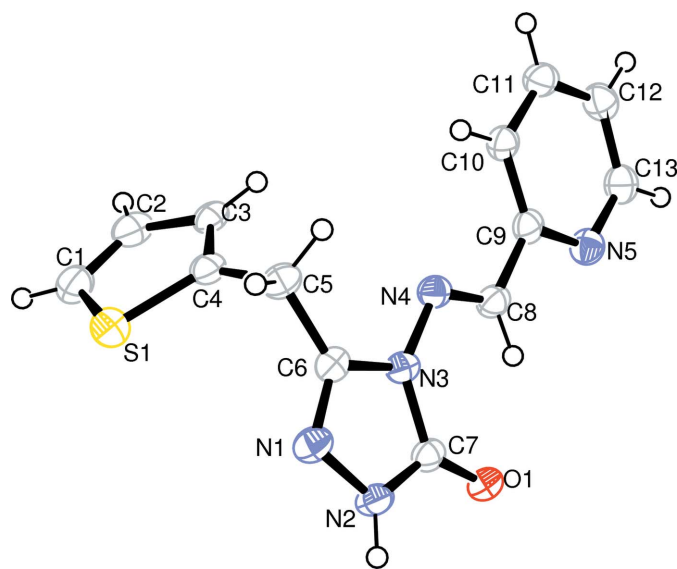


Figure 1
A view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids.

Crystal data

$C_{13}H_{11}N_5OS$
 $M_r = 285.33$
 Monoclinic, $P2_1/n$
 $a = 5.6726$ (4) Å
 $b = 18.3669$ (11) Å
 $c = 12.5690$ (9) Å
 $\beta = 92.651$ (6)°
 $V = 1308.14$ (15) Å³

$Z = 4$
 $D_x = 1.449$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.25$ mm⁻¹
 $T = 293$ (2) K
 Prism, colourless
 $0.48 \times 0.20 \times 0.02$ mm

Data collection

Stoe IPDS-II diffractometer
 ω scans
 Absorption correction: integration
 (*X-RED32*; Stoe & Cie, 2002)
 $T_{min} = 0.910$, $T_{max} = 0.988$

13756 measured reflections
 2564 independent reflections
 1785 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.061$
 $\theta_{max} = 26.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.107$
 $S = 0.96$
 2564 reflections
 182 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0622P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.28$ e Å⁻³
 $\Delta\rho_{min} = -0.38$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0091 (19)

Table 1
Selected bond lengths (Å).

C1—S1	1.701 (3)	C7—O1	1.239 (3)
C4—S1	1.712 (2)	C7—N2	1.341 (3)
C6—N1	1.290 (3)	C7—N3	1.393 (3)
C6—N3	1.384 (3)	N1—N2	1.389 (3)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N2-H6\cdots O1^i$	0.86	1.95	2.777 (2)	161

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

All H atoms were positioned geometrically and treated using a riding model, constraining the aromatic C—H distances at 0.93 Å, methylene C—H distances at 0.97 Å and the N—H distance at 0.86 Å. Isotropic displacement parameters for H atoms were fixed at $U_{iso}(H) = 1.2U_{eq}(\text{carrier atom})$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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