Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl 5-oxo-4-[(2-pyridylmethylene)amino]-3-(2-thienylmethyl)-4,5-dihydro-1*H*-1,2,4-triazole-1-acetate (TF–*S*-pyridine-3-ester)

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Received 4 May 2006
Accepted 23 May 2006

The title compound, $C_{17}H_{17}N_5O_3S$, displays van der Waals and C—H···O interactions, and also C—H···π stacking which is effective in the crystal packing.

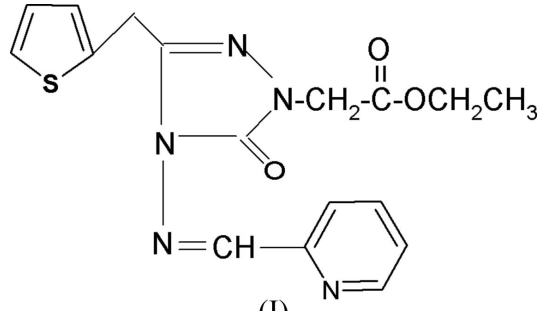
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.088
wR factor = 0.275
Data-to-parameter ratio = 15.9

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

Comment

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 (Tsuda *et al.*, 2004; Chai *et al.*, 2003; Er-Rahimini & Mornet, 1992; Nakib *et al.*, 1994; Jenkins *et al.*, 1989). Di- or trisubstituted 1,2,4-triazole derivatives have also been reported to show antitubercular activities (İkizler *et al.*, 1998). In a previous work, we reported that some 1,2,4-triazol-5-one compounds have antimicrobial effects (Demirbas *et al.*, 2004). 3-Amino-1,2,4-triazole has been recognized as an inhibitor of chloroplast development, with both carotenoid and chlorophyll pigments being effective (Wolf *et al.*, 1960). The coordination chemistry of azoles acting as ligands for the production of organometallic compounds in the context of modelling biological systems has gained much interest (İkizler & & Sancak, 1992). Previously, spectroscopic and crystal-structure data of some 1,2,4-triazoles have been reported (Çoruh *et al.*, 2004; Zhu *et al.*, 2000; Li *et al.*, 2004).



(I)

The title compound, (I), contains three rings, *viz*. 1,2,4-triazole ring *A*, thiophene ring *B* and pyridine ring *C*. The r.m.s. deviations for rings *A*, *B* and *C* are 0.0118, 0.0352 and 0.0078Å, respectively, indicating that all the rings are planar. The relative twisting of these rings can be described by the dihedral angles *A*/*B*, *A*/*C* and *B*/*C* of 86.51 (1), 8.01 (3) and 81.41 (1)°, respectively. Selected bond lengths and angles are listed in Table 1. The C7═O1 is in agreement with the values in similar 1,2,4-triazole rings (Arslan *et al.*, 2004; Ocak, Kahveci *et al.*, 2003; Ocak, Cöruh *et al.*, 2003). The C—S bond lengths are similiar to each other and also compare well with literature reports (Vrabel *et al.*, 2005).

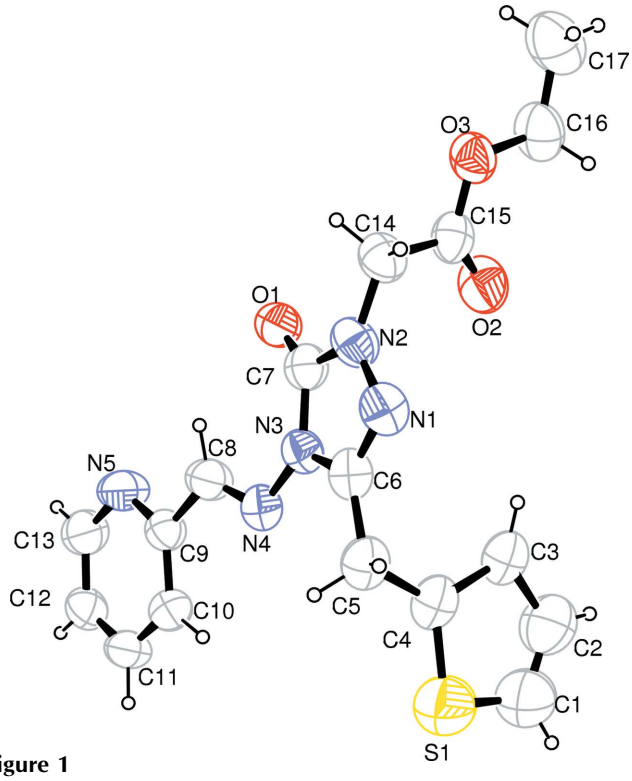


Figure 1
A view of (I), showing the atom-numbering scheme and 50% probability displacement ellipsoids.

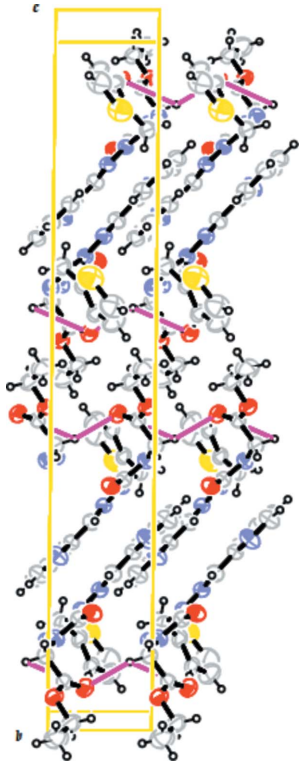


Figure 2
A packing diagram of (I), viewed along the *b*axis.

There is a strong intermolecular C—H···O hydrogen bond as well as a C—H···π interaction, involving atom C5 and the thiophene ring (Table 2).

Experimental

To 4-[(2-pyridylmethylene)amino]-5-(2-thienylmethyl)-2,4-dihydro-1,2,4-triazol-3-one (0.371 g, 0.001 mol) was added a solution of sodium (0.01 mol) in absolute alcohol (0.001 mol), and the mixture was placed in a round-bottomed flask (250 ml). After refluxing for half an hour, a solution of bromoethyl acetate in absolute alcohol was added dropwise. The mixture was refluxed for another 6 h and then cooled. The solid residue (yield 0.39 g, 81.25%) was recrystallized from alcohol–water. IR (cm-1): triazole C═O (1706), C═O (1744), C═N (1610), C═C (1578), mono subs. (700–744).

Crystal data

$C_{17}H_{17}N_5O_3S$
M_r = 371.42
Monoclinic, $P2_1/c$
a = 4.7932 (16) Å
b = 11.326 (3) Å
c = 33.233 (16) Å
β = 90.66 (3)°
V = 1804.0 (12) Å^3
Z = 4
D_x = 1.368 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 293 (2) K
Prism, colourless
0.4 × 0.2 × 0.03 mm

Data collection

Stoe IPDS-2 diffractometer
Rotation method scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.932, T_{max} = 0.993
24512 measured reflections
3548 independent reflections
1546 reflections with $I > 2\sigma(I)$
R_{int} = 0.166
θ_{max} = 26.1°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.088
$wR(F^2)$ = 0.275
S = 0.90
3548 reflections
223 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1655P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.013
$\Delta\rho_{max}$ = 0.82 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

Table 1
Selected bond lengths (Å).

C5—C6	1.490 (7)	C13—N5	1.355 (7)
C7—O1	1.213 (6)	N3—N4	1.390 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14A···O2^{i}	0.97	2.41	3.358 (7)	167
C5—H5B···$Cg1^{i}$	0.97	2.86	3.782 (7)	159

Symmetry code: (i) $x+1, y, z$. $Cg1$ is the centroid of the thiophene ring.

The high value of R_{int} indicates that the overall quality of the data may be poor due to the crystal quality. All H atoms were positioned geometrically (C—H distances at 0.93Å and methylene C—H = 0.97Å) and refined using a riding model, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (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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