

1-Acetyl-4-(3,5-diethyl-4*H*-1,2,4-triazol-4-yl)-  
3-(2-thienylmethyl)-1*H*-1,2,4-triazol-5(4*H*)-oneReşat Ustaşa,<sup>a</sup> Ufuk Çoruh,<sup>b</sup>  
Kemal Sancak,<sup>c</sup> Esra Düğdü<sup>c</sup> and  
Ezequiel M. Vázquez-López<sup>d\*</sup><sup>a</sup>Department of Physics, Graduate School of  
Natural and Applied Sciences, Ondokuz Mayıs  
University, Kurupelit 55139, Samsun, Turkey,<sup>b</sup>Department of Computer Education and  
Instructional Technology, Educational Faculty,  
Ondokuz Mayıs University, Atakum 55200,  
Samsun, Turkey, <sup>c</sup>Department of Chemistry,  
Faculty of Arts and Sciences, Karadeniz Teknik  
University, 61080 Trabzon, Turkey, and<sup>d</sup>Departamento de Química Inorgánica,  
Facultade de Ciencias-Química, Universidade  
de Vigo, 36200 Vigo, Galicia, Spain

Correspondence e-mail: rustabas@omu.edu.tr

## Key indicators

Single-crystal X-ray study

 $T = 293\text{ K}$ Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$  $R$  factor = 0.052 $wR$  factor = 0.092

Data-to-parameter ratio = 14.1

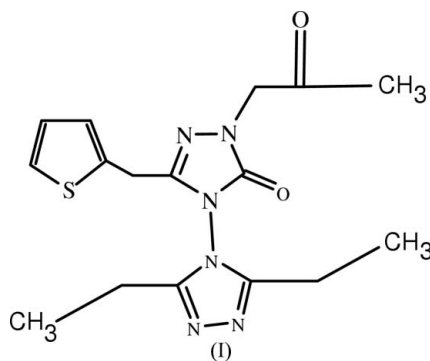
For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.The title compound,  $\text{C}_{16}\text{H}_{20}\text{N}_6\text{O}_2\text{S}$ , has two similar independent molecules in the asymmetric unit, with planar triazole and thiophene rings. Intermolecular  $\text{C}-\text{H}\cdots\text{N}$  and  $\text{C}-\text{H}\cdots\text{O}$  interactions stabilize the crystal structure.

Received 24 August 2006

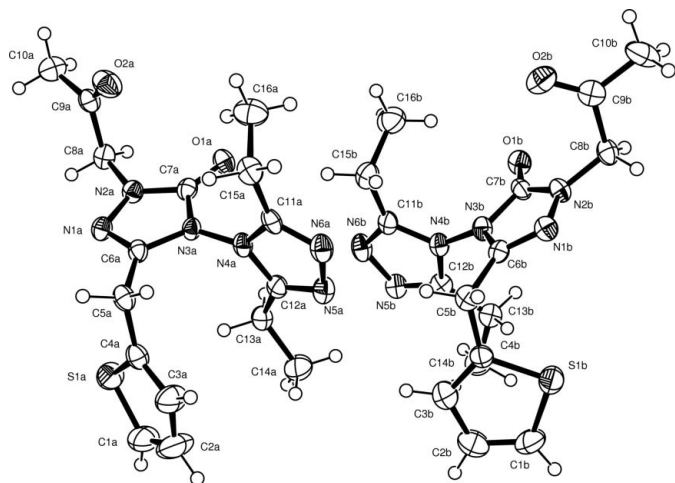
Accepted 29 August 2006

## Comment

Triazole compounds and their derivatives have many applications in industry and medicine. In recent years, ionic liquids consisting of imidazolium and triazolium salts have attracted increasing interest as an alternative to classical organic solvents for a wide range of chemical syntheses (Wasserscheid & Keim, 2000), biocatalysis (Cull *et al.*, 2000), electrochemical applications (Dupont *et al.*, 2002), energetic materials (Gelesky *et al.*, 2005), nano-rods (Wang & Yang, 2005; Swatloski, Spear *et al.*, 2002), liquid-liquid separation (Swatloski, Visser *et al.*, 2002) and polymerization (Hardacre *et al.*, 2002). These interesting liquids containing imidazole and triazole ring systems have unique physical and chemical properties: low melting point, very low vapour pressure, a large liquid phase range, tunable miscibility, and good hydrolytic and thermal stability (Huddleston *et al.*, 2001). Recently, attention has been focused on 1,2,4-triazole derivatives for their broad spectrum of activities. In the continuing search for pharmacologically active compounds, azole fungicides including 1,2,4-triazole and 1,2,4-triazol-5-one compounds have been developed for diseases of cereal crops; examples include fluconazole (Ichikawa *et al.*, 2001), ravuconazole (Ueda, 2003) and posaconazole (Kim *et al.*, 2003). In this paper, we present the synthesis and crystal structure of the title compound, (I).



In compound (I), two independent molecules with similar conformations, labelled *A* and *B*, are present in the asymmetric unit (Fig. 1). There are no significant differences between their interplanar and dihedral angles, *e.g.* the angles



**Figure 1**  
The asymmetric unit of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

between the planar thiophene ring and the two triazole rings are 78.77 (16) and 25.79 (16)° in molecule *A* and 77.93 (17) and 26.19 (16)° in molecule *B*. Atoms N2*A* and N2*B* have a trigonal configuration, the sums of the three bond angles around them being 359.4 and 359.3°, respectively.

In the thiophene ring, the S1—C4 bond is longer than the S1—C1 bond in both molecules (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 N5—N6 bond lengths (Table 1) are similar to some reported values for 1-(benzoylmethyl)-4-(3,5-dimethyl-4*H*-1,2,4-triazol-4-yl)-3-(2-thienylmethyl)-1*H*-1,2,4-triazol-5(4*H*)-one [1.403 (8) Å; Sancak *et al.*, 2005] and *N*-(4-*H*-1,2,4-triazole-4-yl)-*N*-(2-methylbenzylidene)amine [1.408 (2) Å; Ciunik *et al.*, 2002], but longer than that in 4-[(4-hydroxybenzylidene)amino]-4*H*-1,2,4-triazole hemihydrate [1.383 (2) Å; Zhu *et al.*, 2000].

The crystal structure of (I) is stabilized by two intermolecular C(methylene)—H···N and three intermolecular C(methylene)—H···O hydrogen bonds (Table 2).

## Experimental

4-(3,5-Diethyl-4*H*-1,2,4-triazol-4-yl)-3-(2-thienylmethyl)-1*H*-1,2,4-triazol-5(4*H*)-one (0.001 mol) was refluxed with sodium metal (0.001 mol) in absolute ethanol (100 ml) for 1 h. Chloroacetone (0.001 mol) was added and the solution refluxed for 6 h. The solvent was evaporated and the resultant material, (I), was recrystallized from ethanol–diethyl ether (1:8) (yield 87.2%; m.p. 435–436 K). IR and NMR data are available in the archived CIF.

## Crystal data

C<sub>16</sub>H<sub>20</sub>N<sub>6</sub>O<sub>2</sub>S  
*M<sub>r</sub>* = 360.44  
 Triclinic, *P* $\bar{1}$   
*a* = 8.4340 (9) Å  
*b* = 9.8963 (11) Å  
*c* = 21.124 (2) Å  
 $\alpha$  = 88.989 (2)°  
 $\beta$  = 86.672 (2)°  
 $\gamma$  = 89.909 (2)°

*V* = 1759.9 (3) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.360 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 $\mu$  = 0.21 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Prism, colourless  
 0.28 × 0.23 × 0.21 mm

## Data collection

Bruker SMART CCD area-detector  
 diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: none  
 12473 measured reflections

6451 independent reflections  
 3260 reflections with *I* > 2σ(*I*)  
*R*<sub>int</sub> = 0.068  
 $\theta$ <sub>max</sub> = 26.0°

## Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.052  
*wR*(*F*<sup>2</sup>) = 0.092  
*S* = 0.81  
 6451 reflections  
 457 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0244P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.31 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.24 \text{ e \AA}^{-3}$

**Table 1**

Selected bond lengths (Å).

S1 <i>A</i> —C1 <i>A</i>	1.704 (3)	O1 <i>A</i> —C7 <i>A</i>	1.218 (3)
S1 <i>A</i> —C4 <i>A</i>	1.724 (3)	O1 <i>B</i> —C7 <i>B</i>	1.219 (3)
S1 <i>B</i> —C1 <i>B</i>	1.701 (3)	N5 <i>B</i> —N6 <i>B</i>	1.418 (3)
S1 <i>B</i> —C4 <i>B</i>	1.717 (3)	N5 <i>A</i> —N6 <i>A</i>	1.414 (3)

**Table 2**

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C5 <i>B</i> —H5 <i>B</i> 1···N5 <i>A</i>	0.97	2.42	3.382 (4)	171
C15 <i>B</i> —H15 <i>D</i> ···O1 <i>A</i>	0.97	2.60	3.561 (3)	174
C5 <i>A</i> —H5 <i>A</i> 2···N5 <i>B</i> <sup>i</sup>	0.97	2.43	3.386 (4)	170
C8 <i>B</i> —H8 <i>B</i> 1···O2 <i>A</i> <sup>ii</sup>	0.97	2.55	3.397 (4)	147
C8 <i>A</i> —H8 <i>A</i> 2···O2 <i>B</i> <sup>iii</sup>	0.97	2.55	3.413 (4)	148

Symmetry codes: (i) *x* − 1, *y*, *z*; (ii) *x* + 1, *y* − 1, *z*; (iii) *x*, *y* + 1, *z*.

The crystal data were of poor quality, reflected in several low-angle ‘outlier’ reflections. All H atoms were positioned geometrically and refined using a riding model, with aromatic C—H = 0.93 Å, methylene C—H = 0.97 Å and methyl C—H = 0.97 Å, and with *U*<sub>iso</sub>(H) = 1.5*U*<sub>eq</sub>(C) for non-aromatic H atoms and 1.2*U*<sub>eq</sub>(C) for aromatic H atoms.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SHELXTL* (Bruker, 1997); 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).

The authors thank the Faculty of Education, Ondokuz Mayıs University, Turkey, for supporting this work under grant No. EF.107 of the University Research Fund.

## References

- Bruker (1997). *SHELXTL*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Ciunik, Z., Drabent, K. & Szterenber, L. (2002). *J. Mol. Struct.* **641**, 175–182.
- Cull, S. G., Holbrey, J. D., Vargas-Mora, V., Seddon, K. R. & Lye, G. J. (2000). *Biotechnol. Bioeng.* **69**, 227–233.
- Dupont, J., de Souza, R. F. & Suarez, P. A. Z. (2002). *Chem. Rev.* **102**, 3667–3691.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Gelesky, M. A., Umpierre, A. P., Machado, G., Correia, R. R. B., Mango, W. C., Morais, J., Ebeling, G. & Dupont, J. (2005). *J. Am. Chem. Soc.* **127**, 4588–4589.
- Hardacre, C., Holbrey, J. D., Katdare, S. P. & Seddon, K. R. (2002). *Green Chem.* **4**, 143–147.
- Huddleston, J. G., Visser, A. E., Reichert, W. M., Willauer, H. D., Broker, G. A. & Rogers, R. D. (2001). *Green Chem.* **3**, 156–164.
- Ichikawa, T., Kitazaki, T., Matsushita, Y., Yamada, M., Hayashi, R., Yamaguchi, M., Kiyota, Y., Okonogi, K. & Itoh, K. (2001). *Chem. Pharm. Bull.* **49**, 1102–1108.
- Kim, H., Kumar, P., Laughlin, M., Hilbert, M. J., Indelicato, S. R. & Lim, J. (2003). *J. Chromatogr. A*, **987**, 243–248.
- Labat, G. & Halfpenny, J. (2005a). *Acta Cryst.* **E61**, o2813–o2814.
- Labat, G. & Halfpenny, J. (2005b). *Acta Cryst.* **E61**, o2815–o2816.
- Sancak, K., Çoruh, U., Ünver, Y. & Vázquez-López, E. M. (2005). *Acta Cryst.* **E61**, o1785–o1787.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Swatloski, R. P., Spear, S. K., Holbrey, J. D. & Rogers, R. D. (2002). *J. Am. Chem. Soc.* **124**, 4974–4975.
- Swatloski, R. P., Visser, A. E., Reichert, W. M., Broker, G. A., Farina, L. M., Holbrey, J. D. & Rogers, R. D. (2002). *Green Chem.* **4**, 81–87.
- Ueda, Y. (2003). *Bioorg. Med. Chem. Lett.* **13**, 669–3672.
- Wang, Y. & Yang, H. (2005). *J. Am. Chem. Soc.* **127**, 5316–5317.
- Wasserscheid, P. & Keim, W. (2000). *Angew. Chem. Int. Ed.* **39**, 3772–3789.
- Zhu, D.-R., Xu, Y., Liu, Y.-J., Song, Y., Zhang, Y. & You, X.-Z. (2000). *Acta Cryst.* **C56**, 242–243.