Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-[(4-Phenyl-1*H*-1,2,3-triazol-1-yl)methyl]-1,3,4-oxadiazole-2-thione

Xiao-Ru Zhang, Min-Hua Xu, Sai Bi and Shu-Sheng Zhang*

College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, 266042 Qingdao, Shandong, People's Republic of China

Correspondence e-mail: shushzhang@126.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.103
Data-to-parameter ratio = 13.4

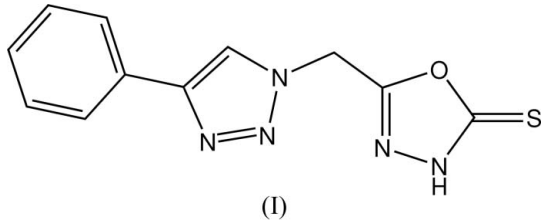
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 29 August 2006
Accepted 4 September 2006

In the title compound, $C_{11}H_9N_5OS$, the molecules are linked into chains along the *c* axis *via* N—H···N hydrogen bonds. The packing is further stabilized by C—H···π and π–π interactions.

Comment

A large number of oxadiazoles (Hwang *et al.*, 2005), triazoles (Lebouvier *et al.*, 2006) and triazolothiadiazines have been shown to exhibit significant antimicrobial activity against *S. aureus*, *C. albicans*, *C. krusei*, *C. parapsilosis*, *T. paradoxa*, *E. coli*, *B. subtilis* and *P. aeruginosa*. These initial reports prompted us to combine 1,3,4-oxadiazole and 1,2,3-triazole units, since these systems possess well documented antimicrobial activity. The title 1,3,4-oxadiazole, (I), was synthesized in high yield.



(I)

In the molecule of (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The dihedral angle between the triazole ring and the benzene ring is 18.13 (1)°, while the oxadiazole ring makes dihedral angles of 62.93 (1) and 63.31 (1)° with the triazole and benzene rings, respectively. The N4—C10 [1.276 (2) Å] bond shows double-bond character, while the N5—C11 bond [1.324 (2) Å] has a character intermediate between single and double (Table 1).

In the crystal structure of (I), molecules are linked into chains along the *c* axis (Fig. 2) *via* N5—H5*A*···N1 hydrogen bonds (Table 2). The packing is further stabilized by C—H···π (Table 2) and π—π interactions involving the triazole rings: *Cg*2···*Cg*2($-x$, $-y$, $1-z$) = 3.797 Å, where *Cg*2 is the centroid of the triazole (N1–N3/C7/C8) ring.

Experimental

To a 273 K solution of ethyl 2-(4-phenyl-1*H*-1,2,3-triazole-1-yl) acetate (1 g, 4.3 mmol) in methanol (50 ml), 80% hydrazine hydrate (0.5 ml, 8.6 mmol) in methanol (10 ml) was added slowly and the mixture was stirred for 1 h at room temperature. A white solid, 2-(4-phenyl-1H-1,2,3-triazol-1-yl)acetohydrazide, was obtained after filtration. To a 273 K solution of this acetohydrazide (0.93 g, 4.3 mmol) and potassium hydroxide (0.48 g, 8.6 mmol) in absolute ethanol (50 ml), carbon disulfide (0.52 ml, 8.6 mmol) in absolute

ethanol (10 ml) was added slowly. The resulting mixture was then stirred and refluxed for 8 h. The solvent was removed *in vacuo*, and the residue was acidified with 2 *M* hydrochloric acid and then extracted with ethyl acetate (2 × 20 ml). The organic portions were washed with water and dried with anhydrous sodium sulfate. After filtration and concentration *in vacuo*, the residue was recrystallized from ethanol to give the title compound, (I) (1 g, 91%). A solution of (I) in ethanol was allowed to stand at room temperature for 2 d and yellow crystals suitable for X-ray crystallographic analysis were grown by slow evaporation.

Crystal data

$C_{11}H_9N_5OS$
M_r = 259.29
Triclinic, $P\overline{1}$
a = 8.3037 (15) Å
b = 8.5820 (15) Å
c = 8.7191 (15) Å
α = 105.911 (2)°
β = 97.441 (2)°
γ = 93.643 (2)°
V = 589.30 (18) Å^3
Z = 2
D_x = 1.461 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 293 (2) K
Plate, yellow
0.38 × 0.29 × 0.11 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.905, T_{max} = 0.971
3267 measured reflections
2245 independent reflections
2003 reflections with $I > 2\sigma(I)$
R_{int} = 0.009
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.103
S = 1.06
2245 reflections
167 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 0.1861P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected bond lengths (Å).

S1—C11	1.6364 (18)	N4—C10	1.276 (2)
O1—C10	1.364 (2)	N5—C11	1.324 (2)
O1—C11	1.384 (2)		

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

*Cg*1 denotes the centroid of the N4/N5/C10/C11/O1 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3*A*···*Cg*1^{i}	0.93	2.98	3.682	133
N5—H5*A*···N1^{ii}	0.87 (2)	1.97 (2)	2.822 (2)	168 (2)

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $x, y, z-1$.

N-bound atom H5*A* was located in a difference map and refined isotropically [N5—H5*A* = 0.87 (2) Å]. All other H atoms were located in difference Fourier maps and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å, and with U_{iso}(H) = 1.2 U_{eq}(C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

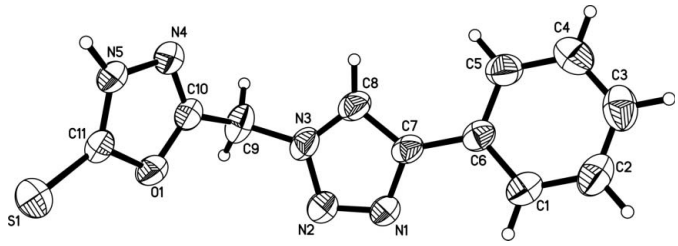


Figure 1
The molecular structure of compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

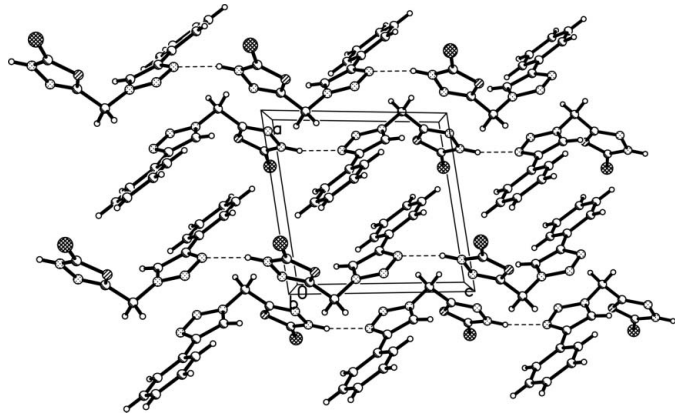


Figure 2
A packing diagram of (I), viewed down the *b* axis. Hydrogen bonds are indicated by dashed lines.

This project was supported by the Special Project of Qingdao for Leadership of Science and Technology (grant No. 05-2-JC-80) and the Outstanding Young Adult Scientific Research Encouraging Foundation of Shandong Province (grant No. 2005BS04007).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Hwang, J. Y., Choi, H. S., Lee, D. H. & Gong, Y. D. (2005). *J. Comb. Chem.* **7**, 816–819.
Lebouvier, N., Giraud, F., Corbin, T., Young, M. N., Baut, G. L., Marchand, P. & Borgne, M. L. (2006). *Tetrahedron Lett.* **47**, 6479–6483.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.