Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(4-Phenyl-3-thioxo-1,2,4-triazolidin-1-yl)-2-(1*H*-1,2,4-triazol-1-yl)ethanone

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.116
Data-to-parameter ratio = 14.9

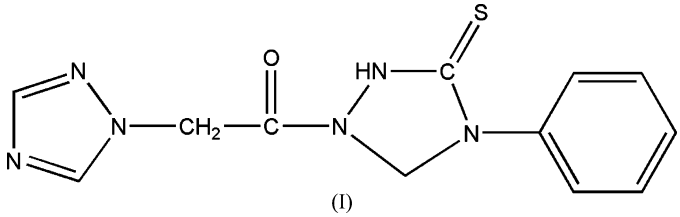
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 12 September 2005
Accepted 20 September 2005
Online 24 September 2005

In the title compound, $C_{12}H_{12}N_6OS$, the dihedral angles made by the plane of the thione-substituted triazolidine ring with the planes of the triazole ring and the benzene ring are 87.77 (2) and 52.07 (3)°, respectively. In the crystal structure, weak intermolecular N—H···N, C—H···N and C—H···O hydrogen bonds stabilize the packing.

Comment

Research findings indicate that the 1,2,4-triazole ring is associated with diverse pharmacological activities, such as analgesic, anti-asthmatic, diuretic, antifungal, antibacterial, pesticidal and anti-inflammatory activities (Bennur *et al.*, 1976; Heubach *et al.*, 1980; Sharma & Babel, 1982; Mohamed *et al.*, 1993). In view of this, the crystal structure determination of the title compound, (I), has been carried out in order to elucidate the stereochemistry and the molecular conformation.



(I)

Bond lengths and angles of the triazole ring (Table 1) are in agreement with the values in our previous report of a similar structure (Xu *et al.*, 2005). The C=S distance is essentially the same as the mean value of 1.660 Å reported by Allen *et al.* (1987). The dihedral angles made by the plane of the thione-substituted triazolidine ring (C1/C2/N1–N3/S1) with the planes of the triazole ring (C11/C12/N4–N6) and the benzene ring (C3–C8) are 87.77 (2) and 52.07 (3)°, respectively. In the crystal structure weak C—H···O, C—H···N and N—H···N intermolecular hydrogen-bond interactions stabilize the packing (Table 2).

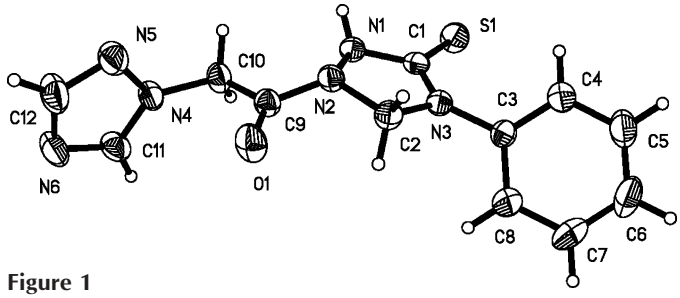


Figure 1
View of the title compound, (I), with displacement ellipsoids drawn at the 40% probability level.

Experimental

A mixture of 5-[(1*H*-1,2,4-triazol-1-yl)methyl]-1,3,4-oxadiazole-2-thiol (0.02 mol), aniline (0.02 mol) and formaldehyde (0.02 mol) was stirred in ethanol (30 ml) for 15 h at 278 K to afford the title compound (2.62 g, yield 91%). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

Crystal data

$C_{12}H_{12}N_6OS$
M_r = 288.34
Monoclinic, $P2_1/c$
a = 9.7112 (19) Å
b = 13.589 (3) Å
c = 10.711 (2) Å
β = 107.362 (4)°
V = 1349.0 (5) Å^3
Z = 4
D_x = 1.420 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1493 reflections
θ = 2.5–22.4°
μ = 0.25 mm^{-1}
T = 294 (2) K
Block, yellow
0.22 × 0.18 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.942, T_{max} = 0.966
7473 measured reflections
2748 independent reflections
1557 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 26.3°
$h = -12 \rightarrow 10$
$k = -16 \rightarrow 16$
$l = -6 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.116
S = 1.00
2748 reflections
185 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Selected interatomic distances (Å).

S1—C1	1.661 (2)	N4—N5	1.355 (3)
N1—C1	1.369 (3)	N5—C12	1.312 (3)
N1—N2	1.425 (3)	N6—C11	1.313 (3)
N4—C11	1.318 (3)	N6—C12	1.349 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N6^{i}	0.83 (3)	2.16 (3)	2.962 (3)	160 (3)
C2—H2*B*···N5^{ii}	0.97	2.61	3.138 (3)	115
C5—H5···O1^{iii}	0.93	2.54	3.288 (4)	138
C11—H11···O1^{iv}	0.93	2.28	2.978 (3)	132

Symmetry codes: (i) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+2, -y+1, -z+1$; (iii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (iv) $x, -y+\frac{1}{2}, z+\frac{1}{2}$.

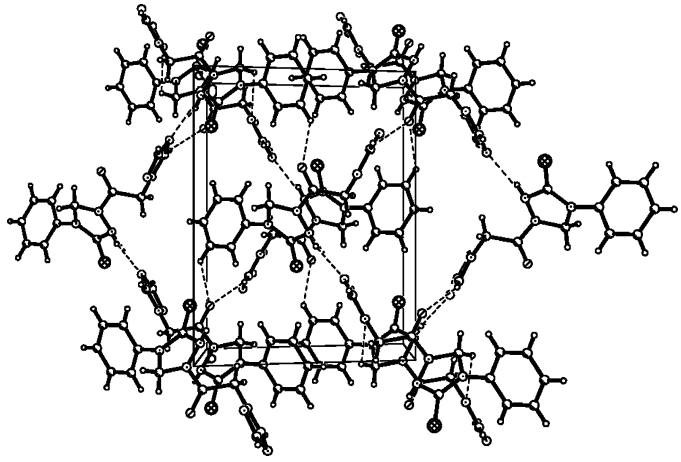


Figure 2
A packing diagram of the title compound, viewed along the *a* axis. Weak hydrogen bonds are shown as dashed lines.

All H atoms were placed in calculated positions. H atoms bonded to C atoms were constrained to ride on their parent atom (C—H = 0.93–0.97 Å), with U_{iso} values of $1.2U_{eq}$(C). The position and isotropic displacement parameter of the N-bound H atom were refined freely.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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