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

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.084
Data-to-parameter ratio = 12.8

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

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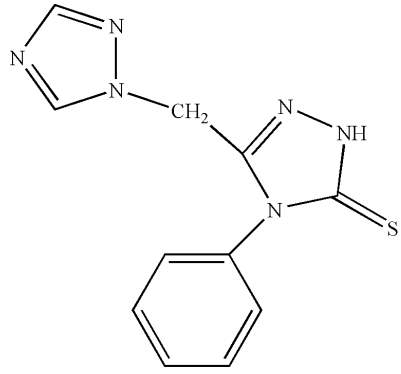
The title compound, $C_{11}H_{10}N_6S$, was prepared by the reaction of ethyl 2-(1*H*-1,2,4-triazol-1-yl)acetate with hydrazine and phenyl isothiocyanate. The molecular structure and packing are stabilized by an N—H···N intermolecular hydrogen-bond and C—H···π interactions.

Comment

Recently, compounds containing the 1*H*-1,2,4-triazole group have attracted much interest because they exhibit some fungicidal activity and plant-growth regulating activity (Xu *et al.*, 2002), and show antibacterial activity against *Puccinia recondite* and root-growth regulation for cucumber (Zhao *et al.*, 1998). As part of our search for new triazole compounds with higher bioactivity, we synthesized the title compound, (I), and describe its structure here.



(I)

In the title compound (Fig. 1), the C—S bond length is within the values observed for a C=S double bond. Atom C9 lies in the plane of the triazole ring (N4/N5/N6/C10/C11) (plane *p*1). The dihedral angles formed by the 2*H*-1,2,4-triazole-3-thione and phenyl rings with *p*1 are 83.4 (1) and 17.8 (1)°, respectively. The dihedral angle between the 2*H*-1,2,4-triazole-3-thione and phenyl rings is 84.7 (3)°. The conformation of the molecule, with nearly parallel triazole and phenyl rings, strongly suggests the presence of intramolecular π–π interactions between these planes (Glusker *et al.*, 1994; Xu, Jian, Cao *et al.*, 2004; Xu, Jian, Qin *et al.*, 2004). The centroid–centroid distance, 3.662 (3) Å, and angle between the ring normal to the phenyl plane and the above centroid vector, 22.4 (2)°, are consistent with π–π interactions (Janiak, 2000).

There is an N—H···N intermolecular interaction (see Table 2), resulting in a two-dimensional network which develops parallel to the *a* direction (Fig. 2). The molecular

structure is also stabilized by intermolecular C—H···π interactions (Jeffrey *et al.*, 1985; Xu, Jian, Cao *et al.*, 2004; Xu, Jian, Qin *et al.*, 2004) (Table 2).

Experimental

The title compound was prepared by the reaction of ethyl 2-(1*H*-1,2,4-triazol-1-yl)acetate (5.50 g, 0.02 mol) with hydrazine (0.6 g, 0.02 mol) and phenyl isothiocyanate (2.24 g, 0.02 mol) in NaOH solution (30 ml). Single crystals of (I) suitable for X-ray measurements were obtained by recrystallization from dimethylformamide solution at room temperature.

Crystal data

$C_{11}H_{10}N_6S$
M_r = 258.31
Monoclinic, $P2_1/c$
a = 9.0010 (18) Å
b = 9.5510 (19) Å
c = 14.251 (3) Å
β = 102.94 (3)°
V = 1194.0 (4) Å^3
Z = 4
D_x = 1.437 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 4–14°
μ = 0.26 mm^{-1}
T = 295 (2) K
Block, yellow
0.35 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
4343 measured reflections
2104 independent reflections
1449 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 25.0°
$h = 0 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -16 \rightarrow 16$
3 standard reflections
every 100 reflections
intensity decay: 0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.084
S = 1.02
2104 reflections
164 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0347P)^2 + 0.2841P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0035 (7)

Table 1
Selected bond lengths (Å).

S1—C7	1.669 (2)	N4—C11	1.328 (2)
N1—C8	1.381 (3)	N4—N5	1.364 (2)
N1—C7	1.382 (2)	N4—C9	1.451 (2)
N1—C6	1.447 (2)	N5—C10	1.314 (3)
N2—C7	1.335 (3)	N6—C11	1.321 (3)
N2—N3	1.376 (2)	N6—C10	1.350 (3)
N3—C8	1.295 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N6^{i}	0.86	1.96	2.794 (2)	164
C1—H1A···$Cg1^{ii}$	0.93	2.99	3.843 (2)	153
C4—H4A···$Cg2^{iii}$	0.93	2.98	3.395 (2)	108
C5—H5B···$Cg1^{iv}$	0.93	2.83	3.490 (2)	129

Symmetry codes: (i) $x-1, y, z$; (ii) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iii) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (iv) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$. Notes: $Cg1$ and $Cg2$ are the centroids of the N1/C7/N2/N3/C8 and N4/N5/C10/N6/C11 rings, respectively.

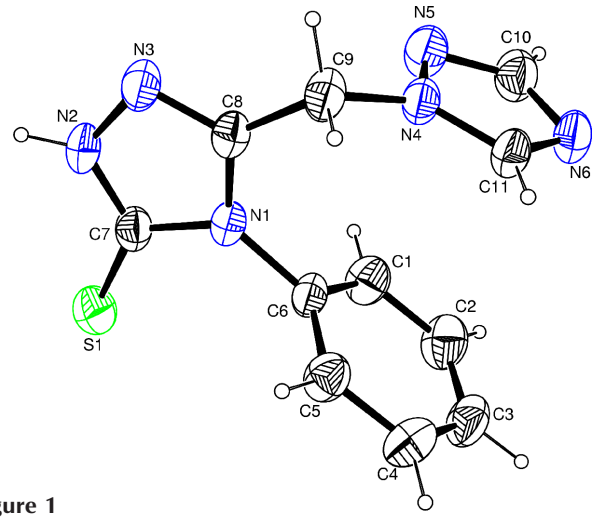


Figure 1
The structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

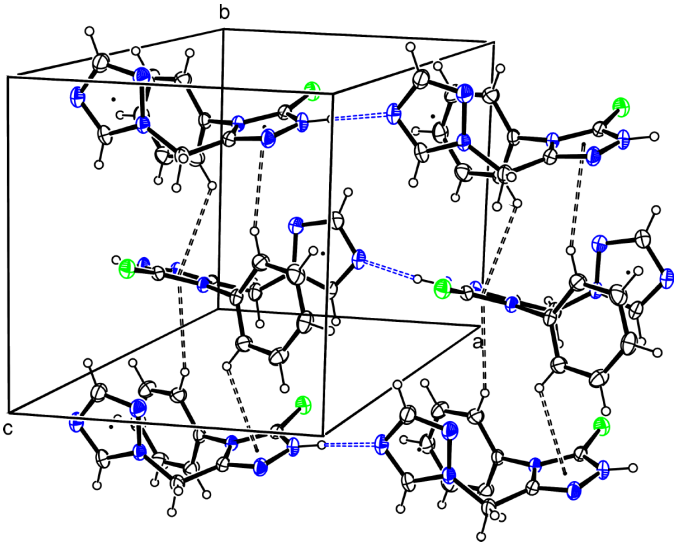


Figure 2
View of the packing in the structure of (I), showing the N—H···N and C—H···π interactions (dashed lines). Displacement ellipsoids are drawn at the 30% probability level.

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å and with U_{iso}(H) = 1.2U_{eq}(carrier atom).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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