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

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.044
wR factor = 0.111
Data-to-parameter ratio = 14.7

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

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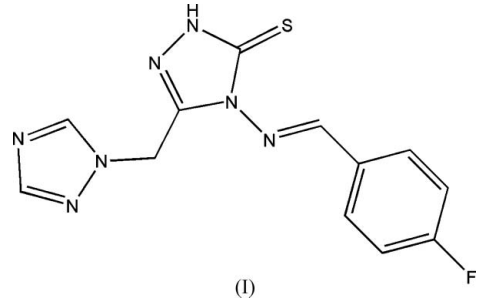
In the title compound, $C_{12}H_{10}FN_7S$, the dihedral angles made by the plane of the thione-substituted triazole ring with the planes of the other triazole ring and the benzene ring are 71.94 (3) and 40.10 (2)°, respectively. Inter- and intramolecular hydrogen-bond and π–π stacking interactions stabilize the structure.

Comment

Compounds containing the 1,2,4-triazole ring possess a broad pharmacological activity spectrum encompassing anti-inflammatory (Prasad *et al.*, 1989), sedative, smooth-muscle relaxation (Gall *et al.*, 1976), anticonvulsant (Kane *et al.*, 1990), antituberculosis (Mir *et al.*, 1970) and platelet-aggregation inhibitory activities (Lagorce *et al.*, 1992). In search of better biological activity, the title compound, (I), was synthesized. We report here the crystal structure of (I).



(I)

The bond lengths and angles of the thione-substituted triazole ring and the other triazole ring (Table 1) are in agreement with the values quotes in previous reports (Li *et al.*, 2005; Xu *et al.*, 2005). The molecule exists in the thione tautomeric form, with an S═C distance of 1.668 (2) Å, which indicates substantial double-bond character (Allen *et al.*, 1987). The planes C1–C3/N1–N3 and C6–C12/N7/F1 make angles of 71.94 (3) and 40.10 (2)°, respectively, with the thione-substituted triazole plane C4/C5/N4–N6/S1. The crystal structure of (I) is stabilized by weak intra- and intermolecular hydrogen bonds and double π–π stacking interactions. The π–π stacking interactions involve the thione-substituted triazole ring (C4/C5/N4–N6/S1) *R*1 and the benzene ring (C7–C12) *R*2. The distance between the centroids of rings *R*1 and *R*2 are 3.589 (8) Å at $(-\frac{1}{2}+x, \frac{1}{2}-y, -z)$ and 3.857 (4) Å at $(\frac{1}{2}+x, \frac{1}{2}-y, -z)$.

Experimental

A mixture of 4-amino-3-(1,2,4-triazol-1-yl)-1*H*-1,2,4-triazole-5(4*H*)-thione (0.02 mol) and 4-fluorobenzaldehyde (0.02 mol) was refluxed

at 391 K for 15–20 min in glacial acetic acid to yield a yellow crystalline precipitate which was recrystallized from ethanol to afford yellow crystals of the title compound (5.45 g, yield 90%; m.p. 497–498 K). ^{1}H NMR (DMSO, 600 MHz): δ 14.16 (*s*, 1H), 10.00 (*s*, 1H), 8.70 (*s*, 1H), 7.99 (*s*, 1H),7.41–7.99 (*m*, 4H), 5.70 (*s*, 2H). IR (KBr, cm^{-1}): 3437, 3109, 2889, 1600, 1509, 1273. Analysis calculated for $C_{12}H_{10}FN_7S$ (Mr = 303.3): C 47.52, H 3.32, N 32.32%; found C 47.53, H 3.30, N 32.33%. Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

Crystal data

$C_{12}H_{10}FN_7S$
M_r = 303.33
Orthorhombic, *Pbca*
a = 7.146 (4) Å
b = 17.906 (9) Å
c = 21.697 (11) Å
V = 2776 (3) Å^3
Z = 8
D_x = 1.452 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2822 reflections
θ = 2.3–25.1°
μ = 0.25 mm^{-1}
T = 294 (2) K
Block, yellow
0.32 × 0.22 × 0.10 mm

Data collection

Bruker SMART CCD area detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.910, T_{max} = 0.971
14610 measured reflections
2855 independent reflections
1673 reflections with $I > 2\sigma(I)$
R_{int} = 0.064
θ_{max} = 26.5°
$h = -8 \rightarrow 6$
$k = -17 \rightarrow 22$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.111
S = 1.01
2855 reflections
194 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 0.939P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C5	1.668 (2)	N4—N5	1.375 (3)
N2—N3	1.360 (3)	N6—N7	1.409 (3)
C6—N7—N6	115.6 (2)	N3—C3—C4	113.6 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5···N1^{i}	0.94 (3)	1.94 (3)	2.867 (3)	174 (3)
C6—H6···S1	0.93	2.70	3.244 (3)	118
C9—H9···N4^{ii}	0.93	2.58	3.451 (4)	157
C11—H11···S1^{iii}	0.93	2.76	3.519 (3)	139

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

All C-bound H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C). The position and isotropic displacement parameter of the N-bound H atoms 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*.

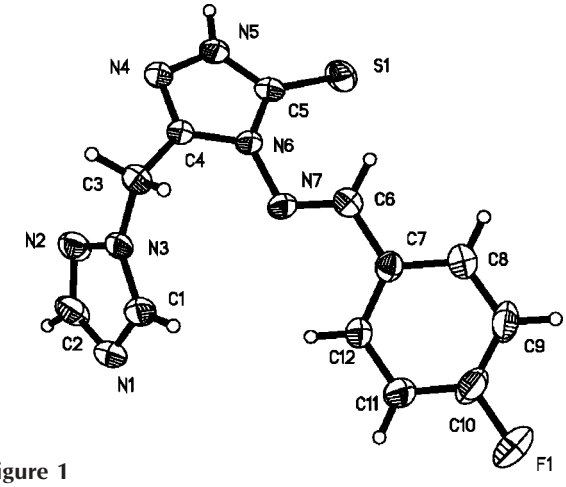


Figure 1
View of the title compound, (I), with displacement ellipsoids for non-H atoms drawn at the 40% probability level. H atoms are represented as spheres of arbitrary radii.

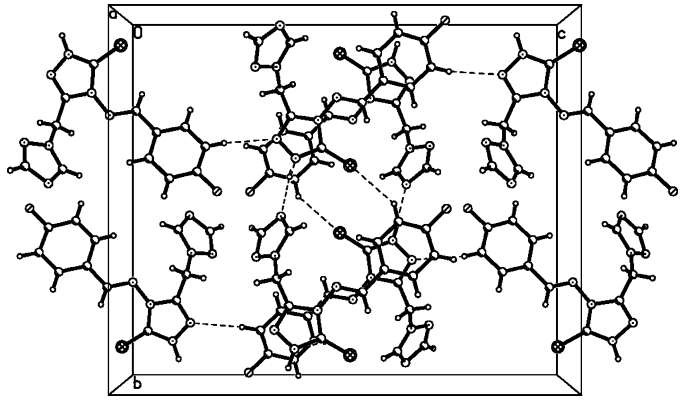


Figure 2
A packing diagram of the title compound, viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

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