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4-Amino-3-(1,2,4-triazol-1-yl)-1*H*-1,2,4-triazole-5(4*H*)-thione

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Key indicators

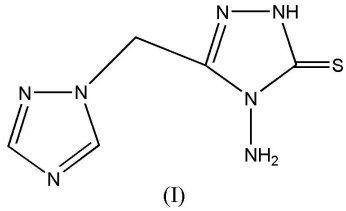
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.031
wR factor = 0.083
Data-to-parameter ratio = 13.2

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

In the molecule of the title compound, $C_5H_7N_7S$, the essentially planar triazole ring and the 4-amino-5-mercapto-1,2,4-triazole moiety make a dihedral angle of 70.97 (5)°. In the crystal structure, weak intermolecular N—H···N and N—H···S hydrogen bonds stabilize the packing.

Comment

1,2,4-Triazole and its derivatives display a broad range of biological activity, finding application as antitumour, antibacterial, antifungal and antiviral agents (Xu *et al.*, 2002; Jantova *et al.*, 1998; Holla *et al.*, 1996). In the search for compounds with better biological activity, the title compound, (I), was synthesized and we report its crystal structure here.



(I)

In the title compound (Fig. 1), the C—S bond length (Table 1) is in good agreement with the mean value of 1.660 Å reported by Allen *et al.* (1987). The essentially planar triazole ring and 4-amino-5-mercapto-1,2,4-triazole moiety [maximum deviations 0.002 (3) and 0.038 (3) Å for atoms N5 and S1, respectively] make a dihedral angle of 70.97 (5)°.

The crystal packing of (I) (Fig. 2) is stabilized by weak intermolecular N—H···N and N—H···S hydrogen bonds (Table 2) and by π–π stacking interactions between the triazole rings TR (N5/N6/C4/N7/C5) of neighbouring molecules. The distance between the centroids of rings TR and TR^{i} is 3.669 (6) Å [symmetry code: (i) $\frac{3}{2} - x$, y $- \frac{1}{2}$, z.

Experimental

2-(1*H*-1,2,4-Triazol-1-yl)acetohydrazide (0.02 mol) and potassium hydroxide (0.02 mol) were dissolved in anhydrous ethanol (80 ml). Carbon disulfide (0.02 mol) was added dropwise to the solution. The resulting mixture was stirred at 283 K for 12 h. The precipitate was then filtered to obtain the intermediate potassium salt, which was used for the next stage without further purification. Hydrazine hydrate (80%; 0.03 mol) was added to the potassium salt dissolved in water (50 ml) with stirring and the mixture was refluxed for 16 h, cooled to 278 K and acidified with concentrated HCl to pH 4–5. The mixture was filtered and crystallized from ethanol to afford the title compound (yield 40%, m.p. 483–484 K). Spectroscopic analysis: ^{1}H NMR (DMSO-d_6, δ, p.p.m.): 5.23 (*s*, 2H, CH_2), 5.60 (*s*, 2H, NH_2), 8.00 (*s*, 1H, Tr—H), 8.64 (*s*, 1H, Tr—H), 13.79 (*s*, 1H, SH).

Crystal data

$C_5H_7N_7S$
M_r = 197.24
Orthorhombic, *Pbca*
a = 11.5192 (18) Å
b = 7.3222 (11) Å
c = 20.048 (3) Å
V = 1690.9 (4) Å^3
Z = 8
D_x = 1.550 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3020 reflections
θ = 3.5–26.2°
μ = 0.35 mm^{-1}
T = 294 (2) K
Block, colourless
0.32 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.882, T_{max} = 0.933
8701 measured reflections
1727 independent reflections
1353 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 26.4°
$h = -7 \rightarrow 14$
$k = -9 \rightarrow 8$
$l = -25 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.083
S = 1.04
1727 reflections
131 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2 + 0.8731P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0063 (8)

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.6726 (19)	N3—C1	1.339 (2)
N1—N2	1.4063 (19)	N3—N4	1.376 (2)
N2—C2	1.363 (2)	N4—C2	1.299 (2)
N2—C1	1.365 (2)		
N5—C3—C2	112.34 (15)		
N1—N2—C1—S1	−1.3 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···N6^{ii}	0.87 (2)	2.10 (2)	2.933 (2)	160 (2)
N1—H1B···N7^{iii}	0.85 (2)	2.43 (2)	3.226 (2)	158 (2)
N1—H1A···S1^{iv}	0.94 (2)	2.76 (2)	3.4410 (17)	130 (2)
N1—H1A···N7^{v}	0.94 (2)	2.54 (2)	3.321 (2)	141 (2)

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

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

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*.

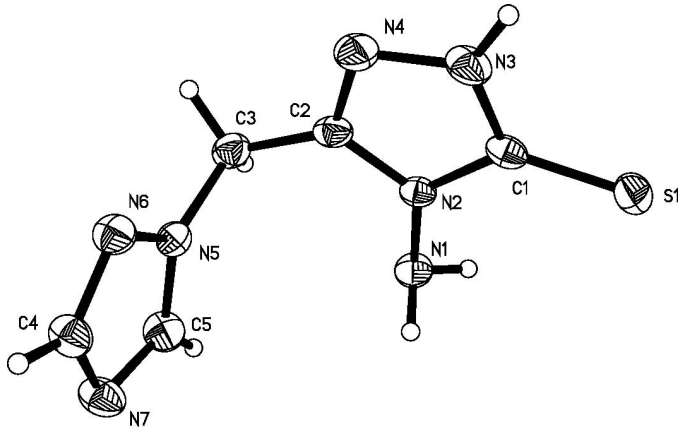


Figure 1
A view of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as small spheres of arbitrary radii.

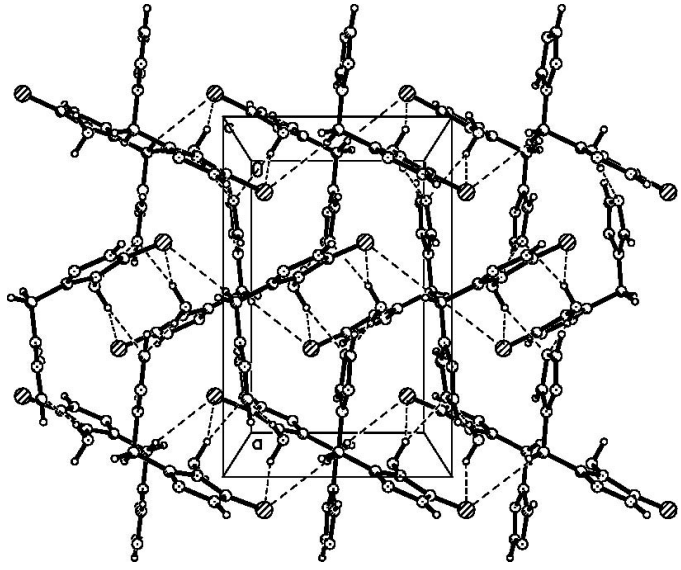


Figure 2
A packing diagram for (I), viewed along the c axis of the cell. Intermolecular N—H···N and N—H···S hydrogen bonds are indicated by dashed lines.

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