

Liang-Zhong Xu,<sup>a\*</sup> Guan-Ping  
Yu,<sup>a</sup> Feng-Ling Xu<sup>b</sup> and  
Wei-Hua Li<sup>c</sup><sup>a</sup>College of Chemistry and Molecular  
Engineering, Qingdao University of Science and  
Technology, Qingdao 266042, People's  
Republic of China, <sup>b</sup>Science and Engineering  
College of Hainan University, Hainan  
University, Haikou 570228, People's Republic  
of China, and <sup>c</sup>College of Chemistry and  
Chemical Engineering, Ocean University of  
China, Qingdao 266033, People's Republic of  
China

Correspondence e-mail: qknhs@163169.net

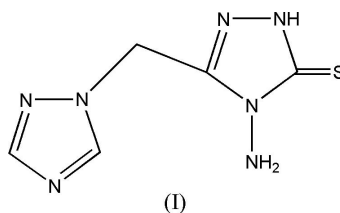
## Key indicators

Single-crystal X-ray study  
 $T = 294\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$   
 $R$  factor = 0.031  
 $wR$  factor = 0.083  
Data-to-parameter ratio = 13.2For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.4-Amino-3-(1,2,4-triazol-1-yl)-1*H*-1,2,4-triazole-  
5(4*H*)-thione

In the molecule of the title compound,  $\text{C}_5\text{H}_7\text{N}_7\text{S}$ , the essentially planar triazole ring and the 4-amino-5-mercapto-1,2,4-triazole moiety make a dihedral angle of  $70.97(5)^\circ$ . In the crystal structure, weak intermolecular  $\text{N}-\text{H}\cdots\text{N}$  and  $\text{N}-\text{H}\cdots\text{S}$  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.



In the title compound (Fig. 1), the  $\text{C}-\text{S}$  bond length (Table 1) is in good agreement with the mean value of  $1.660\text{ \AA}$  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  $0.038(3)\text{ \AA}$  for atoms  $\text{N}5$  and  $\text{S}1$ , respectively] make a dihedral angle of  $70.97(5)^\circ$ .

The crystal packing of (I) (Fig. 2) is stabilized by weak intermolecular  $\text{N}-\text{H}\cdots\text{N}$  and  $\text{N}-\text{H}\cdots\text{S}$  hydrogen bonds (Table 2) and by  $\pi-\pi$  stacking interactions between the triazole rings TR ( $\text{N}5/\text{N}6/\text{C}4/\text{N}7/\text{C}5$ ) of neighbouring molecules. The distance between the centroids of rings TR and  $\text{TR}^i$  is  $3.669(6)\text{ \AA}$  [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  $283\text{ K}$  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  $278\text{ K}$  and acidified with concentrated  $\text{HCl}$  to pH 4–5. The mixture was filtered and crystallized from ethanol to afford the title compound (yield 40%, m.p.  $483\text{--}484\text{ K}$ ). Spectroscopic analysis:  $^1\text{H}$  NMR ( $\text{DMSO}-d_6$ ,  $\delta$ , p.p.m.): 5.23 (s, 2H,  $\text{CH}_2$ ), 5.60 (s, 2H,  $\text{NH}_2$ ), 8.00 (s, 1H,  $\text{Tr}-\text{H}$ ), 8.64 (s, 1H,  $\text{Tr}-\text{H}$ ), 13.79 (s, 1H,  $\text{SH}$ ).

Received 23 May 2005

Accepted 3 June 2005

Online 10 June 2005

## Crystal data

$C_5H_7N_7S$   
 $M_r = 197.24$   
 Orthorhombic,  $Pbca$   
 $a = 11.5192(18) \text{ \AA}$   
 $b = 7.3222(11) \text{ \AA}$   
 $c = 20.048(3) \text{ \AA}$   
 $V = 1690.9(4) \text{ \AA}^3$   
 $Z = 8$   
 $D_x = 1.550 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation  
 Cell parameters from 3020 reflections  
 $\theta = 3.5\text{--}26.2^\circ$   
 $\mu = 0.35 \text{ mm}^{-1}$   
 $T = 294(2) \text{ K}$   
 Block, colourless  
 $0.32 \times 0.24 \times 0.20 \text{ mm}$

## Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.034$   
 $\theta_{\max} = 26.4^\circ$   
 $h = -7 \rightarrow 14$   
 $k = -9 \rightarrow 8$   
 $l = -25 \rightarrow 24$

## 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  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.24 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.25 \text{ e \AA}^{-3}$   
 Extinction correction: SHELXL97 (Sheldrick, 1997)  
 Extinction coefficient: 0.0063 (8)

Table 1

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

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 ( $\text{\AA}$ ,  $^\circ$ ).

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
N3—H3 $\cdots$ N6 <sup>ii</sup>	0.87 (2)	2.10 (2)	2.933 (2)	160 (2)
N1—H1B $\cdots$ N7 <sup>iii</sup>	0.85 (2)	2.43 (2)	3.226 (2)	158 (2)
N1—H1A $\cdots$ S1 <sup>iv</sup>	0.94 (2)	2.76 (2)	3.4410 (17)	130 (2)
N1—H1A $\cdots$ N7 <sup>v</sup>	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  $C\text{—}H = 0.93\text{--}0.97 \text{ \AA}$  and  $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$ .

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.

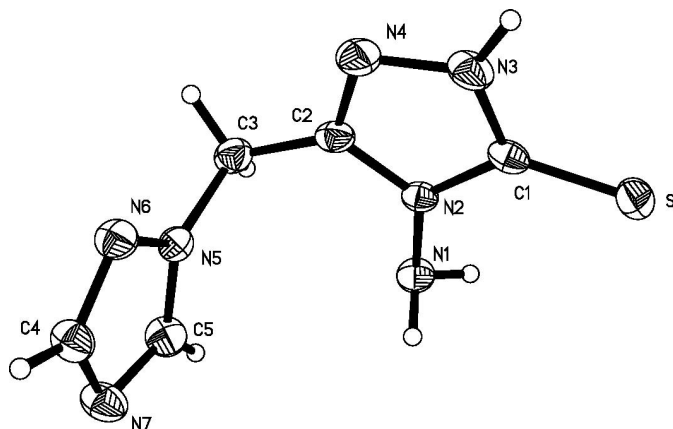


Figure 1

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.

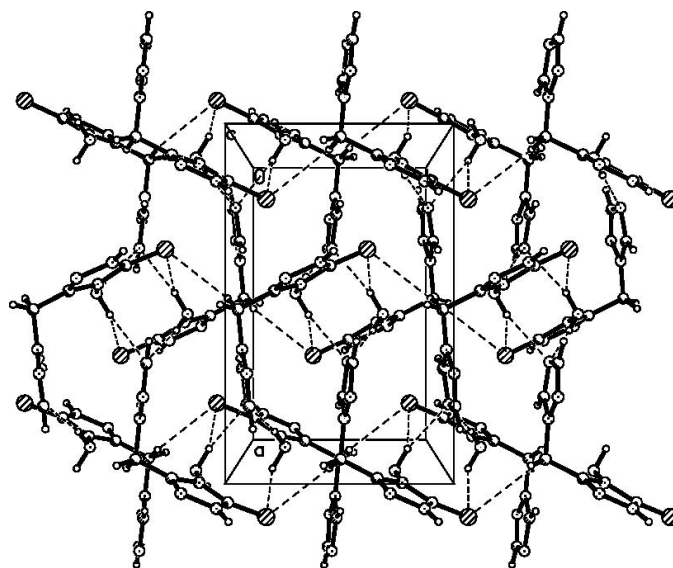


Figure 2

A packing diagram for (I), viewed along the  $c$  axis of the cell. Intermolecular  $N\text{—}H\cdots N$  and  $N\text{—}H\cdots S$  hydrogen bonds are indicated by dashed lines.

## 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.  
 Bruker (1998). SMART. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Bruker (1999). SAINT and SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Holla, B., Poojary, K., Kalluraya, B. & Gowda, P. (1996). *Farmaco*, **51**, 793–799.  
 Jantova, S., Greif, G., Paviovicova, R. & Cipak, L. (1998). *Folia Microbiol. (Prague)*, **43**, 75–80.  
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.  
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.  
 Xu, L. Z., Jiao, K., Zhang, S. S. & Kuang, S. P. (2002). *Bull. Korean Chem. Soc.* **23**, 1699–1701.