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Bis(4*H*-1,2,4-triazol-3-yl)disulfane

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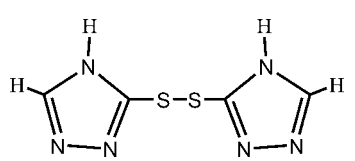
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(N–C) = 0.002 Å; R factor = 0.029; wR factor = 0.080; data-to-parameter ratio = 13.6.

The title compound, $C_4H_4N_6S_2$, was synthesized by the reaction of 3-mercapto-1*H*-1,2,4-triazole with sodium hydroxide in ethanol. The molecule possesses a crystallographically imposed twofold axis. Intermolecular N—H$\cdots$N hydrogen bonds link the molecules into chains along the *c* axis.

Related literature

For related literature, see: De Luca (2006); Di Santo, Tafi, Costi, Botta, Artico, Corelli, Forte, Caporuscio, Angiolella & Palamara (2005); Fringuelli *et al.* (2005); Menozzi *et al.* (2004).



Experimental

Crystal data

$C_4H_4N_6S_2$
M_r = 200.25
Monoclinic, $C2/c$
a = 14.052 (3) Å
b = 6.4044 (13) Å
c = 9.928 (2) Å
β = 122.18 (3)°
V = 756.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.65 mm^{-1}
T = 293 (2) K
0.12 × 0.09 × 0.06 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.932, T_{max} = 0.962
3518 measured reflections
859 independent reflections
742 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.081
S = 1.09
859 reflections
63 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$N3^{i}	0.89 (2)	1.97 (2)	2.8617 (19)	174.9 (19)

Symmetry code: (i) $x, -y+1, z+\frac{1}{2}$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO* (Rigaku, 1998); data reduction: *RAPID-AUTO* (Rigaku, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1993); software used to prepare material for publication: *SHELXL97* (Sheldrick,1997).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2177).

References

De Luca, L. (2006). *Curr. Med. Chem.* **13**, 1–23.
Di Santo, R., Tafi, A., Costi, R., Botta, M., Artico, M., Corelli, F., Forte, M., Caporuscio, F., Angiolella, L. & Palamara, A. T. (2005). *J. Med. Chem.* **48**, 5140–5153.
Fringuelli, R., Milanese, L. & Schiaffella, F. (2005). *Mini-Rev. Med. Chem.* **5**, 1061–1073.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Menozzi, G., Merello, L., Fossa, P., Schenone, S., Ranise, A., Mosti, L., Bondavalli, F., Loddo, R., Murgioni, C., Mascia, V., La Colla, P. & Tamburini, E. (2004). *Bioorg. Med. Chem.* **12**, 5465–5483.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (1993). *SHELXTL/PC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.

supporting information

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Bis(4*H*-1,2,4-triazol-3-yl)disulfane

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S1. Comment

It is well known that derivatives of pyrazole, imidazole, triazole, tetrazole and indole exhibit extensive biological activities (De Luca, 2006; Fringuelli *et al.*, 2005; Di Santo *et al.*, 2005; Menozzi *et al.*, 2004). In a search for more efficient antibacterial medicines, we have synthesized a new azole derivative and its crystal structure is reported here.

In the molecule of the title compound (Fig. 1), which possesses a crystallographically imposed twofold axis, the torsion angles C1—S1—S1^{i}—C1^{i} and S1^{i}—S1—C1—N3 are 83.69 (8) and -93.69 (13)°, respectively [symmetry code: (i) -*x*, *y*, -0.5 - *z*]. The dihedral angle formed by the triazole rings is 21.80 (7)°. In the crystal structure (Fig. 2 and 3), molecules are linked by N—H···N hydrogen bonding interactions (Table 1) to form stepped chains running parallel to the *c* axis.

S2. Experimental

3-Mercapto-1*H*-1,2,4-triazole (0.025 mol, 5.05 g) and sodium hydroxide (0.025 mol, 1.01 g) were dissolved in ethanol (15 ml). The mixture was refluxed at 353 K for five hours, cooled to room temperature, acidified with HCl (12 *M*) and filtered. Colourless crystal of the title compound were obtained on slow evaporation of the solvent after several days at room temperature.

S3. Refinement

All H atoms were located in a difference Fourier map and refined isotropically.

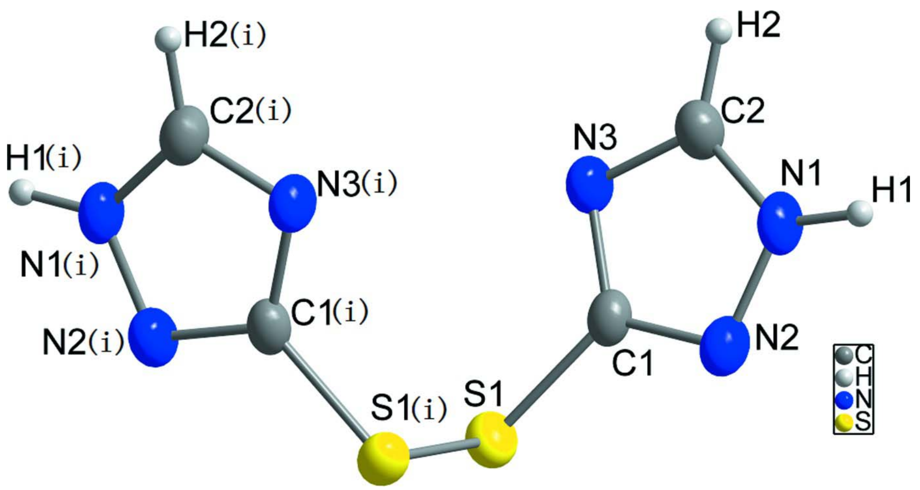

Figure 1

The molecular structure of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) -*x*, *y*, -*z* - 1/2]

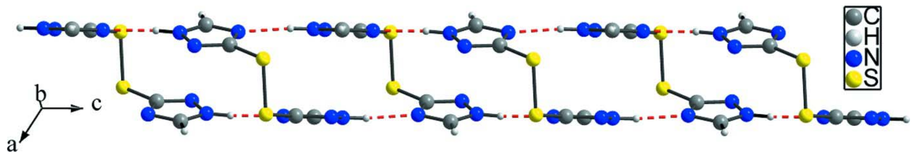

Figure 2

The chain of hydrogen-bonded molecules running along the *c* axis. Hydrogen bonding interactions are shown as red dashed lines.

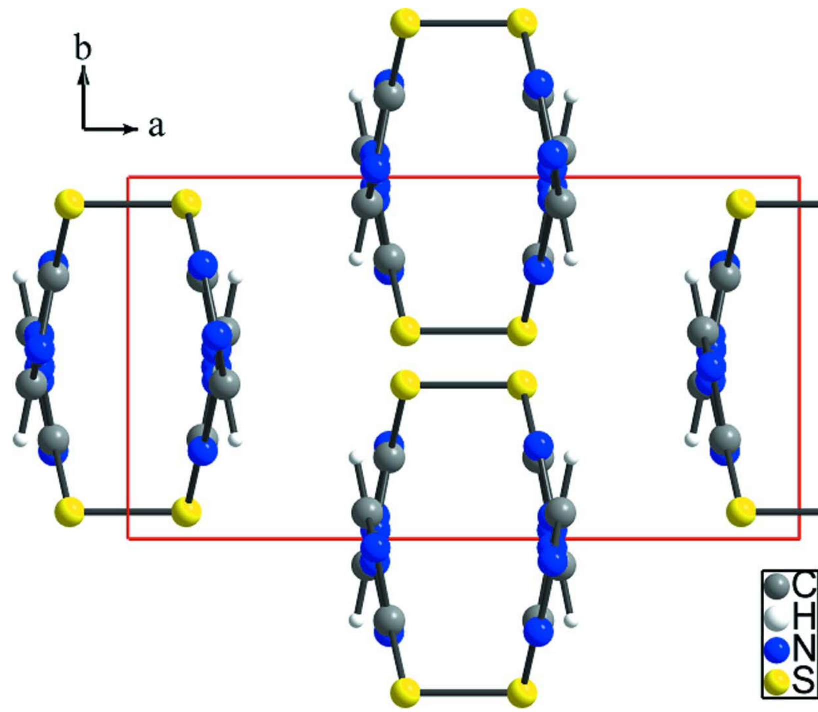

Figure 3

Packing diagram of the title compound viewed along the *c* axis.

Bis(4*H*-1,2,4-triazol-3-yl)disulfane

Crystal data

$C_4H_4N_6S_2$
M_r = 200.25
Monoclinic, $C2/c$
Hall symbol: -C 2yc
a = 14.052 (3) Å
b = 6.4044 (13) Å
c = 9.928 (2) Å
β = 122.18 (3)°
V = 756.2 (4) Å^3
Z = 4
$F(000)$ = 408
D_x = 1.759 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 12–18°
μ = 0.65 mm^{-1}
T = 293 K
Block, colourless
0.12 × 0.09 × 0.06 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Oscillation scans

Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.932, T_{max} = 0.962
3518 measured reflections
859 independent reflections
742 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.5°, θ_{min} = 3.4°
$h = -18 \rightarrow 18$
$k = -8 \rightarrow 7$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.081$
$S = 1.09$
859 reflections
63 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.08684 (3)	0.07452 (6)	−0.17828 (4)	0.03333 (18)
N1	0.13246 (12)	0.4379 (2)	0.15290 (16)	0.0330 (3)
N2	0.11117 (12)	0.2436 (2)	0.08916 (15)	0.0350 (3)
N3	0.13151 (11)	0.4757 (2)	−0.06502 (15)	0.0315 (3)
C1	0.11097 (11)	0.2743 (2)	−0.04273 (16)	0.0275 (3)
C2	0.14361 (13)	0.5723 (3)	0.06089 (18)	0.0331 (4)
H1	0.1365 (17)	0.462 (4)	0.244 (3)	0.053 (6)*
H2	0.1600 (16)	0.725 (3)	0.083 (2)	0.042 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0383 (3)	0.0327 (3)	0.0299 (3)	0.00536 (14)	0.0188 (2)	−0.00149 (13)
N1	0.0378 (7)	0.0423 (8)	0.0238 (7)	0.0035 (5)	0.0197 (6)	−0.0008 (5)
N2	0.0434 (7)	0.0387 (8)	0.0284 (7)	0.0037 (5)	0.0228 (6)	0.0035 (5)
N3	0.0383 (7)	0.0365 (7)	0.0258 (7)	−0.0025 (5)	0.0212 (6)	−0.0017 (5)
C1	0.0280 (7)	0.0346 (8)	0.0215 (7)	0.0031 (5)	0.0143 (6)	0.0022 (5)
C2	0.0357 (8)	0.0387 (9)	0.0270 (8)	−0.0029 (6)	0.0181 (7)	−0.0034 (6)

Geometric parameters (Å, º)

S1—C1	1.7541 (15)	N2—C1	1.3225 (19)
S1—S1^{i}	2.0693 (11)	N3—C2	1.322 (2)
N1—C2	1.324 (2)	N3—C1	1.3653 (19)
N1—N2	1.3549 (18)	C2—H2	1.004 (19)
N1—H1	0.89 (2)		
C1—S1—S1^{i}	101.72 (5)	N2—C1—N3	114.30 (13)
C2—N1—N2	110.63 (13)	N2—C1—S1	123.30 (12)
C2—N1—H1	128.6 (15)	N3—C1—S1	122.40 (11)
N2—N1—H1	120.8 (15)	N3—C2—N1	110.21 (15)
C1—N2—N1	102.11 (13)	N3—C2—H2	124.4 (12)
C2—N3—C1	102.74 (13)	N1—C2—H2	125.4 (12)

Symmetry code: (i) $-x$, y, $-z-1/2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N3^{ii}	0.89 (2)	1.97 (2)	2.8617 (19)	174.9 (19)

Symmetry code: (ii) x, $-y+1$, $z+1/2$.