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## Structure Reports

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## 5,5'-(Disulfanediy)bis(1-methyl-1H-tetrazole)

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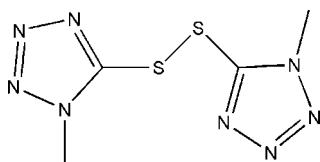
Received 7 July 2011; accepted 23 July 2011

Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{N}-\text{C}) = 0.004$  Å;  $R$  factor = 0.036;  $wR$  factor = 0.106; data-to-parameter ratio = 12.6.

In the title molecule,  $\text{C}_4\text{H}_6\text{N}_8\text{S}_2$ , two tetrazole rings linked by a disulfide bridge form a dihedral angle of  $71.32(7)^\circ$  [ $\text{C}-\text{S}-\text{S}-\text{C}$  torsion angle =  $-80.51(10)^\circ$ ]. In the crystal, strong intermolecular  $\pi-\pi$  interactions between the tetrazole rings [centroid-centroid distance =  $3.285(3)$  Å] link pairs of molecules into centrosymmetric dimers. Weak intermolecular  $\text{C}-\text{H}\cdots\text{N}$  hydrogen bonds further link these dimers, related by translation in the  $[100]$  direction, into columns.

## Related literature

For related structures, see: Kim *et al.* (2003); Brito *et al.* (2007); Tamilselvi & Mugesh (2010). For their use as ligands in transition-metal coordination chemistry, see: She *et al.* (2006); Carballo *et al.* (2009); Wang *et al.* (2010); Aromi *et al.* (2011).



## Experimental

## Crystal data

$\text{C}_4\text{H}_6\text{N}_8\text{S}_2$   
 $M_r = 230.29$   
 Monoclinic,  $P2_1/n$

$a = 6.3232(3)$  Å  
 $b = 8.1625(3)$  Å  
 $c = 18.3623(7)$  Å

$\beta = 98.906(2)^\circ$   
 $V = 936.31(7)$  Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation

$\mu = 0.54$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.40 \times 0.20 \times 0.20$  mm

## Data collection

Bruker APEXII CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2005)  
 $T_{\min} = 0.812$ ,  $T_{\max} = 0.899$

8223 measured reflections  
 1606 independent reflections  
 1527 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.019$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$   
 $wR(F^2) = 0.106$   
 $S = 1.09$   
 1606 reflections

127 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.38$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.35$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C4}-\text{H4C}\cdots\text{N6}^i$	0.96	2.61	3.518 (4)	158

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

Data collection: APEX2 (Bruker, 2005); cell refinement: APEX2; data reduction: SAINT (Bruker, 2005); program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5132).

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## supporting information

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**5,5'-(Disulfanediyl)bis(1-methyl-1*H*-tetrazole)****Ai-Yun Cui and Jing-Lin Liu****S1. Comment**

In recent years, the interesting coordination chemistry and increasingly biomedical properties of complexes derived from bis(1-methyl-1*H*-tetrazol) disulfide ligand have received much attention (Kim *et al.*, 2003; She *et al.*, 2006; Brito *et al.*, 2007; Carballo *et al.*, 2009; Tamilselvi & Mugesh, 2010; Wang *et al.*, 2010). Herein we report the synthesis and crystal structure of the title compound, (I).

In (I) (Fig. 1), the bond lengths and angles are normal and correspond to those observed in the related compounds (Kim *et al.*, 2003; Brito *et al.*, 2007; Tamilselvi & Mugesh, 2010). The dihedral angle between the two tetrazol rings is 71.32 (7)°. The S—S bond length is 2.0474 (8) Å. The C—S—S—C torsion angle of -80.51 (10)° compares well with that of -79.71 (10)° reported by Tamilselvi & Mugesh (2010). The C—S—S—C torsion angle in two bis-tetrazol disulfides reported by Kim *et al.* (2003) and Brito *et al.* (2007) are 81.54 (5)° and 80.42 (6)°, respectively.

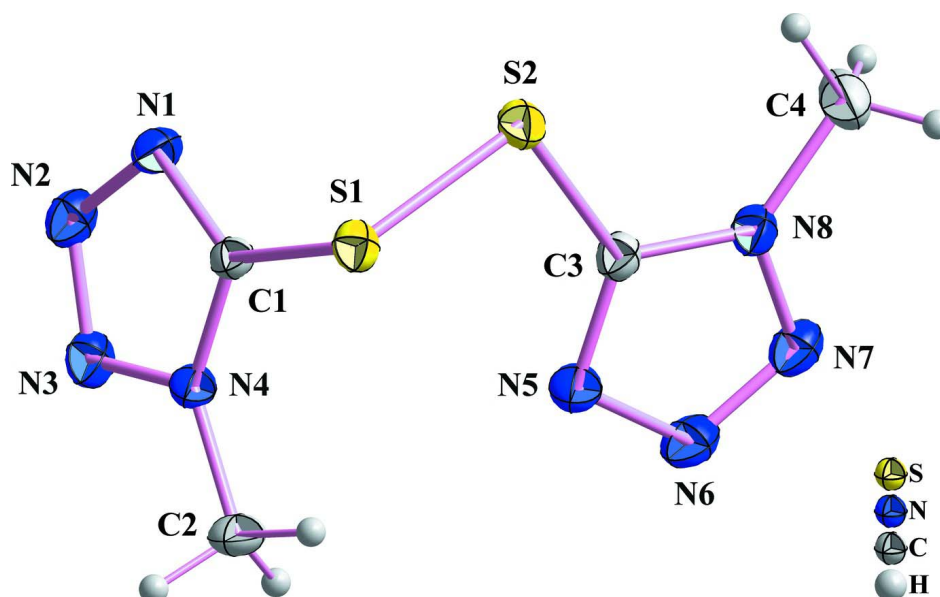
In the crystal structure, strong intermolecular  $\pi$ — $\pi$  interaction between the tetrazole rings [centroid-centroid distance of 3.285 (3) Å] link two molecules into centrosymmetric dimer. Weak intermolecular C—H $\cdots$ N hydrogen bonds (Table 1) link further these dimers related by translation in [100] into columns.

**S2. Experimental**

A water solution (5 ml) of Fe(NO<sub>3</sub>)<sub>3</sub> (0.25 mmol) was added slowly to the water solution (15 ml) of 1-methyl-5-mercaptotetrazole (0.10 mmol). The reaction mixture was stirred at room temperature for 3 h. The solvent was removed and the solid product recrystallized from ethanol. After six days, the colourless crystals suitable for X-ray diffraction were obtained.

**S3. Refinement**

All H atoms were placed in idealized positions and refined using a riding model (C—H = 0.96 Å) with  $U_{iso}(H) = 1.5 U_{eq}(C)$ .

**Figure 1**

View of (I) showing the atomic labeling and 30% probability displacement ellipsoids.

### 5,5'-(Disulfanediy)bis(1-methyl-1H-tetrazole)

#### Crystal data

$C_4H_6N_8S_2$

$M_r = 230.29$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2_1n$

$a = 6.3232\ (3)\ \text{\AA}$

$b = 8.1625\ (3)\ \text{\AA}$

$c = 18.3623\ (7)\ \text{\AA}$

$\beta = 98.906\ (2)^\circ$

$V = 936.31\ (7)\ \text{\AA}^3$

$Z = 4$

$F(000) = 472$

$D_x = 1.634\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 9988 reflections

$\theta = 2.5\text{--}40.5^\circ$

$\mu = 0.54\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Block, colourless

$0.40 \times 0.20 \times 0.20\ \text{mm}$

#### Data collection

Bruker APEXII CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2005)

$T_{\min} = 0.812$ ,  $T_{\max} = 0.899$

8223 measured reflections

1606 independent reflections

1527 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.019$

$\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 2.7^\circ$

$h = -7 \rightarrow 5$

$k = -9 \rightarrow 9$

$l = -21 \rightarrow 21$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.106$

$S = 1.09$

1606 reflections

127 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

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.6856P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.38 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.35 \text{ e } \text{\AA}^{-3}$

#### 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 ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.21952 (9)	0.05034 (7)	0.39949 (3)	0.0367 (2)
S2	0.13427 (9)	0.20613 (8)	0.47738 (3)	0.0427 (2)
N2	0.2328 (4)	0.3565 (3)	0.24114 (12)	0.0461 (5)
N1	0.1217 (3)	0.2818 (2)	0.28933 (11)	0.0393 (5)
C1	0.2630 (3)	0.1878 (2)	0.32986 (11)	0.0296 (4)
N4	0.4531 (3)	0.2035 (2)	0.30732 (10)	0.0348 (4)
N6	0.7056 (3)	0.3555 (3)	0.55018 (13)	0.0484 (5)
N5	0.5698 (3)	0.2755 (3)	0.49736 (12)	0.0448 (5)
C3	0.3824 (4)	0.2790 (3)	0.52098 (12)	0.0351 (5)
N8	0.4035 (3)	0.3574 (2)	0.58550 (10)	0.0386 (4)
N3	0.4312 (3)	0.3089 (3)	0.25086 (11)	0.0430 (5)
C2	0.6534 (4)	0.1195 (4)	0.33142 (15)	0.0503 (6)
H2B	0.6382	0.0491	0.3722	0.075*
H2C	0.7640	0.1985	0.3465	0.075*
H2A	0.6908	0.0551	0.2915	0.075*
N7	0.6092 (3)	0.4054 (3)	0.60292 (12)	0.0485 (5)
C4	0.2441 (5)	0.3978 (4)	0.63164 (16)	0.0627 (8)
H4C	0.1080	0.3535	0.6101	0.094*
H4A	0.2330	0.5146	0.6355	0.094*
H4B	0.2858	0.3518	0.6799	0.094*

#### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0401 (4)	0.0366 (3)	0.0343 (3)	-0.0056 (2)	0.0089 (2)	0.0033 (2)
S2	0.0329 (3)	0.0600 (4)	0.0367 (3)	-0.0085 (2)	0.0095 (2)	-0.0078 (2)
N2	0.0515 (12)	0.0450 (11)	0.0406 (11)	-0.0020 (9)	0.0028 (9)	0.0106 (9)
N1	0.0344 (10)	0.0417 (10)	0.0408 (11)	0.0010 (8)	0.0024 (8)	0.0035 (8)

C1	0.0268 (10)	0.0327 (10)	0.0293 (10)	-0.0033 (8)	0.0041 (8)	-0.0015 (8)
N4	0.0300 (9)	0.0411 (10)	0.0337 (10)	-0.0034 (7)	0.0065 (8)	0.0021 (7)
N6	0.0352 (11)	0.0539 (12)	0.0547 (13)	-0.0054 (9)	0.0024 (10)	0.0047 (10)
N5	0.0364 (11)	0.0549 (12)	0.0439 (11)	-0.0038 (9)	0.0088 (9)	0.0025 (9)
C3	0.0346 (12)	0.0382 (12)	0.0326 (11)	-0.0034 (9)	0.0051 (9)	0.0065 (9)
N8	0.0379 (10)	0.0422 (10)	0.0348 (10)	-0.0049 (8)	0.0028 (8)	0.0005 (8)
N3	0.0453 (12)	0.0498 (11)	0.0348 (10)	-0.0081 (9)	0.0090 (9)	0.0071 (8)
C2	0.0261 (11)	0.0749 (18)	0.0508 (14)	0.0049 (11)	0.0086 (10)	0.0060 (13)
N7	0.0397 (11)	0.0527 (12)	0.0503 (12)	-0.0071 (9)	-0.0023 (10)	0.0009 (10)
C4	0.0513 (16)	0.092 (2)	0.0472 (15)	-0.0110 (16)	0.0141 (13)	-0.0212 (15)

*Geometric parameters (Å, °)*

S1—C1	1.754 (2)	N5—C3	1.324 (3)
S1—S2	2.0474 (8)	C3—N8	1.335 (3)
S2—C3	1.752 (2)	N8—N7	1.349 (3)
N2—N3	1.299 (3)	N8—C4	1.452 (3)
N2—N1	1.357 (3)	C2—H2B	0.9600
N1—C1	1.317 (3)	C2—H2C	0.9600
C1—N4	1.337 (3)	C2—H2A	0.9600
N4—N3	1.338 (3)	C4—H4C	0.9600
N4—C2	1.448 (3)	C4—H4A	0.9600
N6—N7	1.287 (3)	C4—H4B	0.9600
N6—N5	1.359 (3)		
C1—S1—S2	101.53 (7)	C3—N8—C4	130.1 (2)
C3—S2—S1	102.49 (8)	N7—N8—C4	121.8 (2)
N3—N2—N1	111.26 (18)	N2—N3—N4	106.28 (18)
C1—N1—N2	104.84 (19)	N4—C2—H2B	109.5
N1—C1—N4	109.43 (19)	N4—C2—H2C	109.5
N1—C1—S1	127.98 (17)	H2B—C2—H2C	109.5
N4—C1—S1	122.48 (16)	N4—C2—H2A	109.5
C1—N4—N3	108.17 (18)	H2B—C2—H2A	109.5
C1—N4—C2	130.23 (19)	H2C—C2—H2A	109.5
N3—N4—C2	121.46 (19)	N6—N7—N8	106.3 (2)
N7—N6—N5	111.64 (19)	N8—C4—H4C	109.5
C3—N5—N6	104.7 (2)	N8—C4—H4A	109.5
N5—C3—N8	109.2 (2)	H4C—C4—H4A	109.5
N5—C3—S2	128.82 (19)	N8—C4—H4B	109.5
N8—C3—S2	121.87 (17)	H4C—C4—H4B	109.5
C3—N8—N7	108.06 (19)	H4A—C4—H4B	109.5
C1—S1—S2—C3	-80.51 (10)	S1—S2—C3—N5	17.9 (2)
N3—N2—N1—C1	0.9 (3)	S1—S2—C3—N8	-165.44 (17)
N2—N1—C1—N4	-0.2 (2)	N5—C3—N8—N7	0.3 (3)
N2—N1—C1—S1	-176.46 (16)	S2—C3—N8—N7	-176.89 (16)
S2—S1—C1—N1	-65.6 (2)	N5—C3—N8—C4	177.5 (3)
S2—S1—C1—N4	118.62 (17)	S2—C3—N8—C4	0.3 (4)

N1—C1—N4—N3	-0.5 (2)	N1—N2—N3—N4	-1.2 (3)
S1—C1—N4—N3	175.98 (15)	C1—N4—N3—N2	1.0 (2)
N1—C1—N4—C2	-176.1 (2)	C2—N4—N3—N2	177.1 (2)
S1—C1—N4—C2	0.4 (3)	N5—N6—N7—N8	0.3 (3)
N7—N6—N5—C3	-0.1 (3)	C3—N8—N7—N6	-0.4 (3)
N6—N5—C3—N8	-0.2 (3)	C4—N8—N7—N6	-177.8 (2)
N6—N5—C3—S2	176.80 (18)		

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C4—H4C $\cdots$ N6 <sup>i</sup>	0.96	2.61	3.518 (4)	158

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