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2,2'-[(1,3,4-Thiadiazole-2,5-diyl)bis-(sulfanediyl)]diacetonitrile

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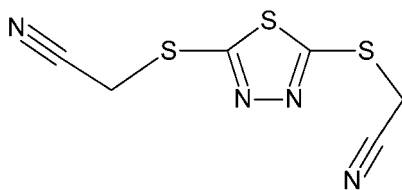
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 Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.029; wR factor = 0.074; data-to-parameter ratio = 20.8.

In the title compound, $\text{C}_6\text{H}_4\text{N}_4\text{S}_3$, the 1,3,4-thiadiazole ring is essentially planar, with an r.m.s. deviation of 0.001 Å. The two $\text{N}-\text{C}-\text{S}-\text{C}$ torsion angles in the molecule are -23.41 (15) and 0.62 (14)°. One acetonitrile group is above the plane of the 1,3,4-thiadiazole ring and the other is below it, indicating *syn* and *anti* orientations. In the crystal, $\text{C}-\text{H}\cdots\text{N}$ hydrogen bonds link the molecules into ribbons along [010].

Related literature

For the broad spectrum of biological activities of thiadiazole-containing compounds, see: Padmavathi *et al.* (2009); Karegoudar *et al.* (2008); Wei *et al.* (2009); Gupta *et al.* (2009); Pattanayak *et al.* (2009); Cressier *et al.* (2009).



Experimental

Crystal data

 $\text{C}_6\text{H}_4\text{N}_4\text{S}_3$
 $M_r = 228.34$

 Monoclinic, $P2_1/c$
 $a = 8.5305$ (7) Å

 $b = 14.2102$ (11) Å

 $c = 7.8803$ (6) Å

 $\beta = 104.3810$ (11)°

 $V = 925.32$ (13) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.76$ mm⁻¹
 $T = 150$ K

 $0.24 \times 0.08 \times 0.06$ mm

Data collection

Bruker SMART APEX CCD diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2013)

 $T_{\min} = 0.82$, $T_{\max} = 0.96$

16625 measured reflections

2450 independent reflections

 2155 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.074$
 $S = 1.05$

2450 reflections

118 parameters

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.56$ e Å⁻³
 $\Delta\rho_{\min} = -0.24$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C3}-\text{H3A}\cdots\text{N1}^i$	0.99	2.60	3.407 (2)	139
$\text{C5}-\text{H5B}\cdots\text{N3}^{ii}$	0.99	2.35	3.267 (2)	153

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

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXT* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5363).

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supplementary materials

Acta Cryst. (2013). E69, o1855 [doi:10.1107/S1600536813032194]

2,2'-[(1,3,4-Thiadiazole-2,5-diyl)bis(sulfaneyl)]diacetonitrile

Joel T. Mague, Mehmet Akkurt, Shaaban K. Mohamed, Ahmed M. M. El-Saghier and Mustafa R. Albayati

1. Comment

Thiadiazole acts as a constrained pharmacophore. It is the core structure of several medicinal drugs such as acetazolamide, atibepnone, tebuthiuron and methazolamide. In addition, thiadiazole containing compounds have a wide spectrum of biological activities such as antimicrobial (Padmavathi *et al.*, 2009), antiinflammatory (Karegoudar *et al.*, 2008), anticancer (Wei *et al.*, 2009), anticonvulsant (Gupta *et al.*, 2009), antidepressant (Pattanayak *et al.*, 2009), and antioxidant (Cressier *et al.*, 2009). Based on such facts, the title compound has been synthesized in our lab as a precursor for further study.

The 1,3,4-thiadiazole ring (S1/N1/N2/C1/C2) of the title compound, (I, Fig. 1), is essentially planar [r.m.s deviation = 0.001 Å]. The N1–C1–S2–C3 and N2–C2–S3–C5 torsion angles in (I) are 23.41 (15) and -0.62 (14)°, respectively. The two acetonitrile groups [–C3(H₂)–C4≡N3 and –C5(H₂)–C6≡N4] of (I) are above and below the plane of the 1,3,4-thiadiazole ring, indicating *syn*- and *anti*- orientations, respectively.

In the crystal, molecules are linked by intermolecular C—H···N hydrogen bonds to form ribbons along the *b*-axis (Table 1, Fig. 2).

2. Experimental

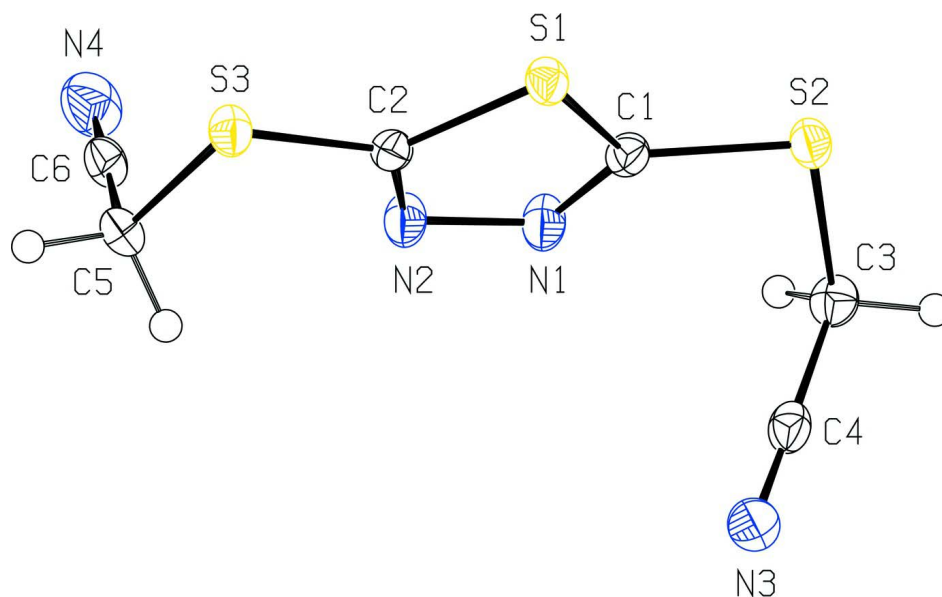
A mixture of 1,3,4-thiadiazolidine-2,5-dithione (150 mg, 1 mmol), chloroacetonitrile (149 mg, 2 mmol), sodium acetate (36 mg, 0.5 mmol) in 30 ml ethanol was refluxed for 4 h. The reaction mixture was allowed to cool to room temperature to afford the solid product which was filtered off under vacuum, dried and recrystallized from ethanol. Pure single crystals were prepared by slow evaporation of an ethanolic solution of the title compound in air over 24 h. M.P. 396 K.

3. Refinement

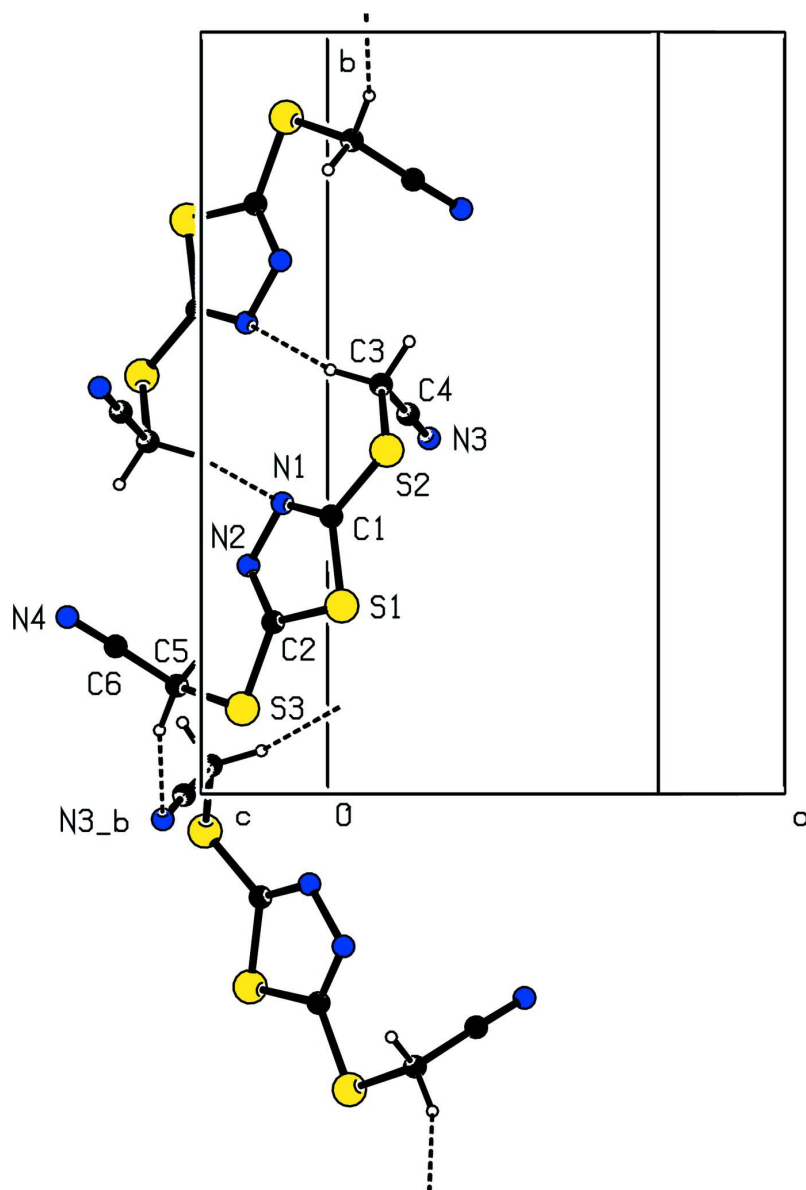
The methylene H atoms were positioned geometrically and refined by using a riding model with C—H = 0.99 Å and, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{iso}}(\text{C})$.

Computing details

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: *SHELXT* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

**Figure 1**

Perspective view of the title molecule with 50% probability displacement ellipsoids.


Figure 2

The hydrogen bonding (dashed lines) viewed along the *b*-axis of the title compound. [Symmetry code: (*b*) $1 + x, \frac{1}{2} - y, \frac{1}{2} + z$].

2,2'-[(1,3,4-Thiadiazole-2,5-diyl)bis(sulfaneyl)]diacetonitrile

Crystal data

$C_6H_4N_4S_3$

$M_r = 228.34$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 8.5305\ (7)\ \text{\AA}$

$b = 14.2102\ (11)\ \text{\AA}$

$c = 7.8803\ (6)\ \text{\AA}$

$\beta = 104.3810\ (11)^\circ$

$V = 925.32\ (13)\ \text{\AA}^3$

$Z = 4$

$F(000) = 464$

$D_x = 1.639\ \text{Mg m}^{-3}$

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

Cell parameters from 9961 reflections

$\theta = 2.9\text{--}29.1^\circ$

$\mu = 0.76\ \text{mm}^{-1}$

$T = 150$ K $0.24 \times 0.08 \times 0.06$ mm
 Column, pale gold

Data collection

Bruker SMART APEX CCD diffractometer	16625 measured reflections
Radiation source: fine-focus sealed tube	2450 independent reflections
Graphite monochromator	2155 reflections with $I > 2\sigma(I)$
Detector resolution: 8.3660 pixels mm^{-1}	$R_{\text{int}} = 0.040$
φ and ω scans	$\theta_{\text{max}} = 29.1^\circ$, $\theta_{\text{min}} = 2.5^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2013)	$h = -11 \rightarrow 11$
$T_{\text{min}} = 0.82$, $T_{\text{max}} = 0.96$	$k = -19 \rightarrow 19$
	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.029$	$W = 1/[\Sigma^2(FO^2) + (0.0348P)^2 + 0.4414P]$ Where
$wR(F^2) = 0.074$	$P = (FO^2 + 2FC^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\text{max}} < 0.001$
2450 reflections	$\Delta\rho_{\text{max}} = 0.56 \text{ e } \text{\AA}^{-3}$
118 parameters	$\Delta\rho_{\text{min}} = -0.24 \text{ e } \text{\AA}^{-3}$
0 restraints	

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating $-R$ -factor-obs *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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.24557 (4)	0.24679 (2)	0.77612 (5)	0.0199 (1)
S2	0.35261 (4)	0.45127 (3)	0.80614 (5)	0.0225 (1)
S3	-0.02136 (4)	0.11179 (3)	0.59651 (5)	0.0217 (1)
N1	0.07063 (16)	0.38153 (9)	0.61222 (18)	0.0246 (4)
N2	-0.01784 (15)	0.29982 (9)	0.56140 (18)	0.0232 (4)
N3	0.31454 (18)	0.46664 (10)	0.33409 (18)	0.0286 (4)
N4	-0.40861 (18)	0.23221 (12)	0.5806 (2)	0.0370 (5)
C1	0.20800 (17)	0.36440 (10)	0.72254 (19)	0.0188 (4)
C2	0.05731 (17)	0.22555 (10)	0.63639 (18)	0.0178 (4)
C3	0.29143 (18)	0.53720 (10)	0.6312 (2)	0.0223 (4)
C4	0.30417 (18)	0.49844 (10)	0.4636 (2)	0.0222 (4)
C5	-0.20837 (18)	0.14204 (11)	0.4383 (2)	0.0240 (4)
C6	-0.32061 (18)	0.19370 (11)	0.5168 (2)	0.0247 (4)
H3A	0.17830	0.55670	0.62240	0.0270*
H3B	0.36100	0.59370	0.65920	0.0270*
H5A	-0.18280	0.18080	0.34420	0.0290*

H5B -0.26140 0.08360 0.38420 0.0290*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0170 (2)	0.0195 (2)	0.0210 (2)	-0.0004 (1)	0.0001 (1)	0.0025 (1)
S2	0.0207 (2)	0.0212 (2)	0.0224 (2)	-0.0041 (1)	-0.0007 (1)	0.0015 (1)
S3	0.0205 (2)	0.0189 (2)	0.0243 (2)	-0.0020 (1)	0.0029 (1)	-0.0006 (1)
N1	0.0195 (6)	0.0210 (6)	0.0302 (7)	-0.0028 (5)	0.0006 (5)	0.0037 (5)
N2	0.0185 (6)	0.0212 (6)	0.0274 (7)	-0.0031 (5)	0.0012 (5)	0.0025 (5)
N3	0.0310 (7)	0.0273 (7)	0.0261 (7)	-0.0034 (6)	0.0042 (6)	0.0016 (6)
N4	0.0244 (7)	0.0490 (9)	0.0352 (8)	0.0019 (7)	0.0031 (6)	-0.0032 (7)
C1	0.0195 (7)	0.0175 (6)	0.0194 (7)	-0.0010 (5)	0.0050 (5)	0.0017 (5)
C2	0.0156 (6)	0.0217 (7)	0.0164 (6)	-0.0014 (5)	0.0043 (5)	0.0004 (5)
C3	0.0232 (7)	0.0173 (7)	0.0260 (8)	0.0014 (5)	0.0051 (6)	0.0023 (6)
C4	0.0202 (7)	0.0180 (7)	0.0265 (8)	-0.0026 (5)	0.0025 (6)	0.0041 (6)
C5	0.0233 (7)	0.0272 (8)	0.0187 (7)	-0.0049 (6)	0.0000 (5)	-0.0028 (6)
C6	0.0187 (7)	0.0299 (8)	0.0215 (7)	-0.0047 (6)	-0.0026 (6)	0.0009 (6)

Geometric parameters (Å, °)

S1—C1	1.7340 (15)	N3—C4	1.140 (2)
S1—C2	1.7333 (15)	N4—C6	1.142 (2)
S2—C1	1.7538 (15)	C3—C4	1.460 (2)
S2—C3	1.8184 (15)	C5—C6	1.460 (2)
S3—C2	1.7486 (15)	C3—H3A	0.9900
S3—C5	1.8155 (16)	C3—H3B	0.9900
N1—N2	1.3885 (19)	C5—H5A	0.9900
N1—C1	1.297 (2)	C5—H5B	0.9900
N2—C2	1.2984 (19)		
C1—S1—C2	85.82 (7)	S3—C5—C6	112.64 (11)
C1—S2—C3	98.35 (7)	N4—C6—C5	178.33 (17)
C2—S3—C5	97.88 (7)	S2—C3—H3A	109.00
N2—N1—C1	111.85 (12)	S2—C3—H3B	109.00
N1—N2—C2	112.14 (13)	C4—C3—H3A	109.00
S1—C1—S2	121.11 (9)	C4—C3—H3B	109.00
S1—C1—N1	115.19 (11)	H3A—C3—H3B	108.00
S2—C1—N1	123.63 (11)	S3—C5—H5A	109.00
S1—C2—S3	121.93 (8)	S3—C5—H5B	109.00
S1—C2—N2	114.99 (11)	C6—C5—H5A	109.00
S3—C2—N2	123.07 (11)	C6—C5—H5B	109.00
S2—C3—C4	111.14 (10)	H5A—C5—H5B	108.00
N3—C4—C3	178.79 (16)		
C2—S1—C1—S2	-177.61 (10)	C5—S3—C2—N2	0.62 (14)
C2—S1—C1—N1	-0.51 (12)	C2—S3—C5—C6	-68.78 (12)
C1—S1—C2—S3	179.01 (10)	C1—N1—N2—C2	0.16 (19)
C1—S1—C2—N2	0.61 (12)	N2—N1—C1—S1	0.32 (17)
C3—S2—C1—S1	153.44 (9)	N2—N1—C1—S2	177.33 (11)

C3—S2—C1—N1	-23.41 (15)	N1—N2—C2—S1	-0.57 (17)
C1—S2—C3—C4	-60.50 (12)	N1—N2—C2—S3	-178.96 (11)
C5—S3—C2—S1	-177.66 (9)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C3—H3 <i>A</i> ...N1 ⁱ	0.99	2.60	3.407 (2)	139
C5—H5 <i>B</i> ...N3 ⁱⁱ	0.99	2.35	3.267 (2)	153

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