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

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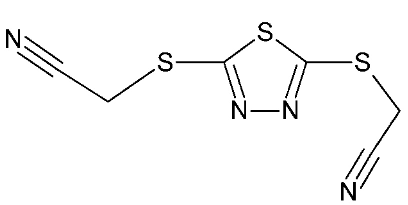
Key indicators: single-crystal X-ray study; $T$ = 150 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.029; $wR$ factor = 0.074; data-to-parameter ratio = 20.8.

In the title compound, $C_6H_4N_4S_3$, the 1,3,4-thiadiazole ring is essentially planar, with an r.m.s. deviation of 0.001 Å. The two N—C—S—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, C—H⋯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*

$C_6H_4N_4S_3$
$M_r$ = 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) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.76 mm$^{-1}$
$T$ = 150 K
0.24 × 0.08 × 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 $I > 2\sigma(I)$
$R_{int}$ = 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⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| C3—H3$A$⋯N1$^{i}$ | 0.99 | 2.60 | 3.407 (2) | 139 |
| C5—H5$B$⋯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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supporting information

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

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

## S1. Comment

Thiadiazole acts as a constrained pharmacophore. It is the core structure of several medicinal drugs such as acetazolamide, atibeprone, 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 precurser 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($H_2$)–C4≡N3 and –C5($H_2$)–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).

## S2. 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 e thanol 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.

## S3. Refinement

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

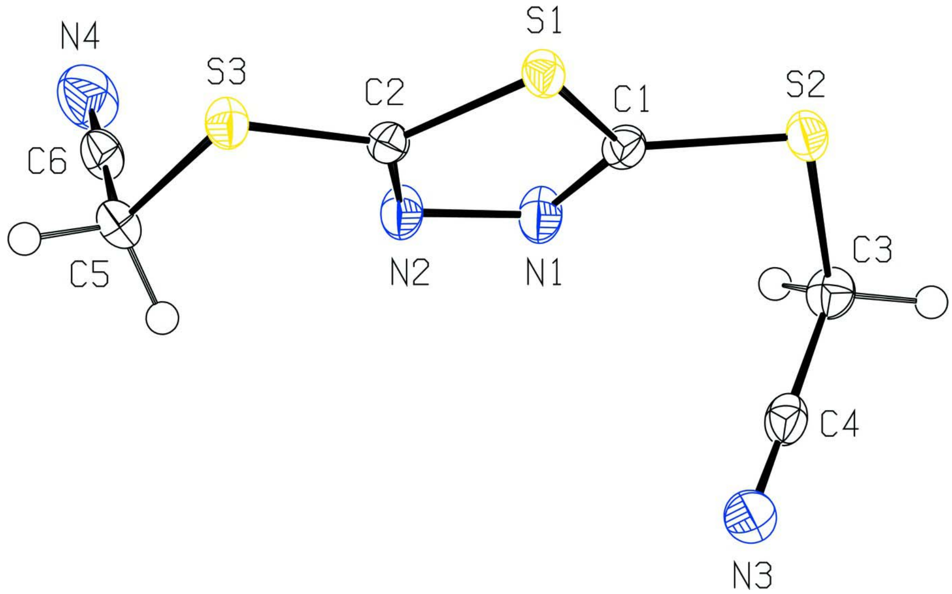


**Figure 1**

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

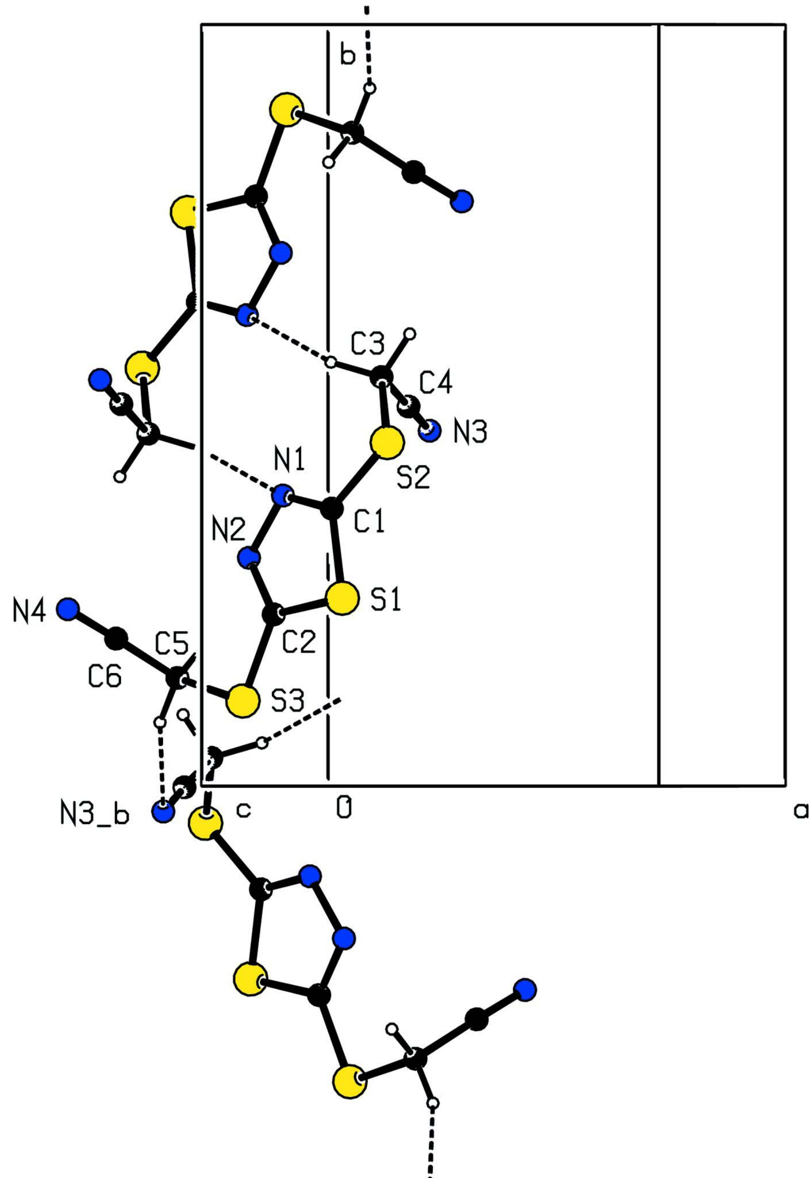


**Figure 2**

The hydrogen bonding (dashed lines) viewed along the *b*-axis of the title compound. [Symmetry code: (*b*) 1 + *x*, ½ - *y*, ½ + *z*].

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

*Crystal data*

$C_6H_4N_4S_3$
$M_r$ = 228.34
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 8.5305 (7) Å
$b$ = 14.2102 (11) Å
$c$ = 7.8803 (6) Å
$\beta$ = 104.3810 (11)°
$V$ = 925.32 (13) Å$^3$
$Z$ = 4
$F$(000) = 464
$D_x$ = 1.639 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 9961 reflections
$\theta$ = 2.9–29.1°
$\mu$ = 0.76 mm$^{-1}$

$T = 150$ K
Column, pale gold

0.24 × 0.08 × 0.06 mm

*Data collection*

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3660 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
$T_{min} = 0.82$, $T_{max} = 0.96$

16625 measured reflections
2450 independent reflections
2155 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 29.1°$, $\theta_{min} = 2.5°$
$h = -11 \rightarrow 11$
$k = -19 \rightarrow 19$
$l = -10 \rightarrow 10$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.074$
$S = 1.05$
2450 reflections
118 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$W = 1/[\Sigma^2(FO^2) + (0.0348P)^2 + 0.4414P]$ Where $P = (FO^2 + 2FC^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.56$ e Å$^{-3}$
$\Delta\rho_{min} = -0.24$ e Å$^{-3}$

*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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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, º)*

| | | | |
|---|---|---|---|
| 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, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C3—H3*A*···N1$^{i}$ | 0.99 | 2.60 | 3.407 (2) | 139 |
| C5—H5*B*···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.