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4,4′-[(1,3,4-Thiadiazole-2,5-diyl)bis(thiomethylene)]dibenzonitrile

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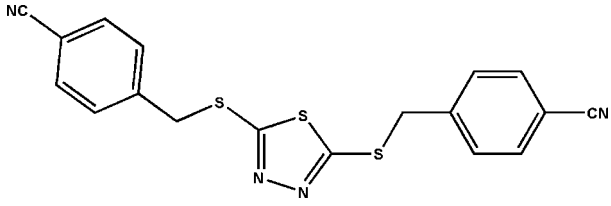
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.053; wR factor = 0.116; data-to-parameter ratio = 17.8.

The title molecule, $C_{18}H_{12}N_4S_3$, consists of three essentially planar fragments, *viz*. two methyl-substituted benzonitrile rings and a substituted thiadiazole ring. The dihedral angles between the substituted benzonitrile rings and the central thiadiazole ring are 28.29 (10) and 78.83 (6)°, and the dihedral angle between the two benzonitrile rings is 72.89 (7)°.

Related literature

For related literature, see: Tarafder *et al.*, (2000); El-Shekeil *et al.* (1988); Jinxia *et al.* (2003).



Experimental

Crystal data

$C_{18}H_{12}N_4S_3$
M_r = 380.50
Monoclinic, $P2_1/c$
a = 7.6974 (15) Å
b = 8.4375 (17) Å
c = 27.272 (6) Å
β = 92.53 (3)°
V = 1769.5 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.43 mm^{-1}
T = 293 (2) K
0.45 × 0.40 × 0.30 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.814, T_{max} = 0.903
15327 measured reflections
4028 independent reflections
2754 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.115
S = 1.05
4028 reflections
226 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: FL2207).

References

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supporting information

Acta Cryst. (2008). E**64**, o1599 [doi:10.1107/S1600536808022848]

4,4′-[(1,3,4-Thiadiazole-2,5-diyl)bis(thiomethylene)]dibenzonitrile

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S1. Comment

1,3,4-thiadiazole-2,5-dithiol and its derivatives are interesting compounds that have attracted the attention of researchers because of their wide range of applications in many fields, such as the determination of trace elements, the synthesis of novel heterocyclic compounds with antimicrobial activity(El-Shekeil *et al.* 1988), advanced materials, and battery cathodes (Jinxia *et al.*2003, Tarafder *et al.*, (2000)). In this paper, we report the structure of one such derviative, the title compound (I).

In (I, Fig. 1) all bond lengths and angles are normal. There are three planar fragments in the molecule, *viz*. the central S1—S2—S3—C1—C2—N1—N2 plane with C1 farthest out at (0.0077 (19) Å) and the two benzonitrile systems C11—C18 and N4 with C11 farthest out at 0.0280 (19) Å) and C3—C19 and N3 with N3 farthest out at 0.0738 (21) Å. The dihedral angles between the central substituted ring and benzonitrile systems are 28.29 (10)° and 78.83 (6)° and 72.89 (7)° between the two benzonitrile moieties. There is no evidence of typical hydrogenn bonding or intermolecular π–π interactions. Only van-der-waals interactions are observed in the crystal is packing (Fig.2).

S2. Experimental

A dry 50 ml flask was charged with 1,3,4-thiadiazole-2,5-dithiol (10 mmol), 4-(bromomethyl)benzonitrile (20 mmol), K_2CO_3(10 mmol), and methanol (30 mL). The mixture was stirred with refluxing for 4 h and then was poured into water (40 ml), the precipitate was washed with water for 2–3 times and purified by recrystallization from methanol to give the crystals of the target material.

S3. Refinement

All the C—H hydrogen atoms were calculated geometrically and were allowed to ride on the C atoms to which they are bonded, with acyclic C—H distances ranging as 0.97 Å, phenyl C—H distances ranging as 0.93 Å, and with U_{iso}(H) = 1.2Ueq(C).

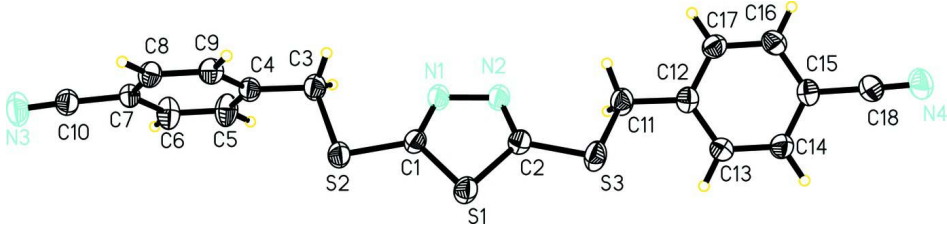

Figure 1

A view of the compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 30% probability level.

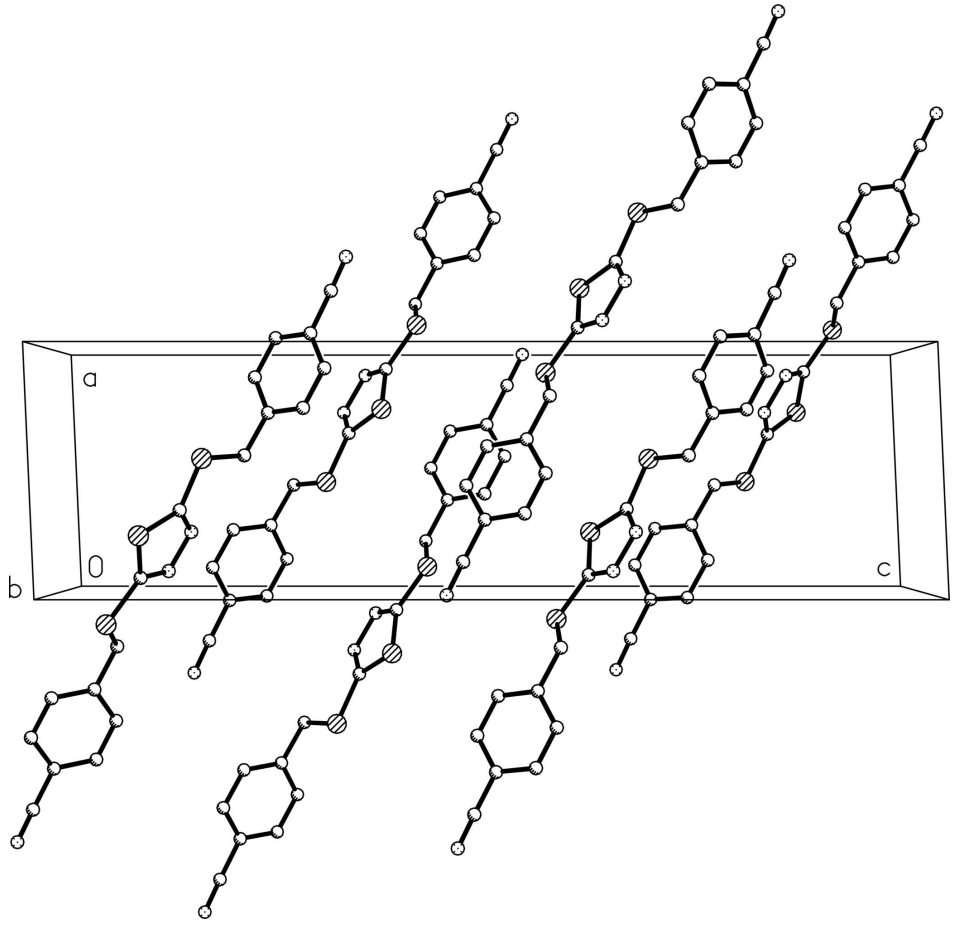

Figure 2

Packing diagram of the title compound, showing the structure along the *b* axis.

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Crystal data

$C_{18}H_{12}N_4S_3$
$M_r = 380.50$
Monoclinic, $P2_1/c$
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$a = 7.6974\ (15)$ Å
$b = 8.4375\ (17)$ Å
$c = 27.272\ (6)$ Å
$\beta = 92.53\ (3)°$
$V = 1769.5\ (6)$ Å^3
$Z = 4$
$F(000) = 784$
$D_x = 1.428$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 12716 reflections
$\theta = 3.3–27.3°$
$\mu = 0.43$ mm^{-1}
$T = 293$ K
Prism, colorless
0.45 × 0.40 × 0.30 mm

Data collection

Rigaku Mercury2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.814$, $T_{max} = 0.903$
15327 measured reflections
4028 independent reflections
2754 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$

θ_{max} = 27.5°, θ_{min} = 3.0°
h = −9→9
k = −10→10
l = −35→35

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.115
S = 1.05
4028 reflections
226 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
$w = 1/[\sigma^2(F_o^2) + (0.0435P)^2 + 0.4638P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 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}
C1	0.6555 (3)	0.3789 (3)	0.34124 (8)	0.0469 (5)
C2	0.9214 (3)	0.3764 (3)	0.38800 (8)	0.0476 (6)
C3	0.4395 (3)	0.2503 (3)	0.27094 (10)	0.0619 (7)
H3A	0.5357	0.2535	0.2491	0.074*
H3B	0.4455	0.1514	0.2891	0.074*
C4	0.2700 (3)	0.2614 (3)	0.24188 (9)	0.0494 (6)
C5	0.1254 (3)	0.1815 (3)	0.25715 (10)	0.0681 (8)
H5	0.1344	0.1193	0.2853	0.082*
C6	−0.0313 (3)	0.1926 (3)	0.23136 (10)	0.0675 (8)
H6	−0.1277	0.1379	0.2419	0.081*
C7	−0.0448 (3)	0.2853 (3)	0.18983 (8)	0.0463 (5)
C8	0.0971 (3)	0.3662 (3)	0.17434 (8)	0.0504 (6)
H8	0.0873	0.4293	0.1464	0.060*
C9	0.2533 (3)	0.3540 (3)	0.20014 (9)	0.0521 (6)
H9	0.3494	0.4087	0.1894	0.062*
C10	−0.2123 (3)	0.3038 (3)	0.16434 (9)	0.0576 (7)
C11	1.2030 (3)	0.2210 (3)	0.42467 (10)	0.0602 (7)
H11A	1.1265	0.1432	0.4386	0.072*
H11B	1.2195	0.1920	0.3908	0.072*
C12	1.3756 (3)	0.2188 (3)	0.45268 (8)	0.0489 (6)
C13	1.4027 (3)	0.2929 (3)	0.49739 (9)	0.0571 (6)
H13	1.3126	0.3500	0.5106	0.068*
C14	1.5604 (3)	0.2838 (3)	0.52286 (9)	0.0568 (6)

H14	1.5767	0.3346	0.5530	0.068*
C15	1.6945 (3)	0.1987 (3)	0.50351 (9)	0.0486 (6)
C16	1.6682 (3)	0.1221 (3)	0.45909 (9)	0.0563 (6)
H16	1.7575	0.0631	0.4462	0.068*
C17	1.5102 (3)	0.1328 (3)	0.43390 (9)	0.0553 (6)
H17	1.4936	0.0815	0.4039	0.066*
C18	1.8610 (3)	0.1876 (3)	0.52986 (9)	0.0558 (6)
N1	0.7442 (3)	0.2519 (3)	0.33425 (8)	0.0576 (5)
N2	0.9002 (3)	0.2507 (2)	0.36166 (8)	0.0573 (5)
N3	−0.3451 (3)	0.3216 (4)	0.14549 (9)	0.0848 (8)
N4	1.9930 (3)	0.1766 (3)	0.54989 (9)	0.0740 (7)
S1	0.75230 (9)	0.51067 (8)	0.38233 (2)	0.0594 (2)
S2	0.45247 (8)	0.41683 (8)	0.31298 (2)	0.0595 (2)
S3	1.10283 (9)	0.41341 (8)	0.42665 (3)	0.0642 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0514 (13)	0.0435 (13)	0.0451 (13)	−0.0003 (11)	−0.0051 (10)	−0.0021 (10)
C2	0.0521 (14)	0.0403 (13)	0.0499 (13)	−0.0007 (11)	−0.0046 (10)	0.0007 (10)
C3	0.0557 (15)	0.0595 (16)	0.0689 (17)	0.0079 (13)	−0.0167 (12)	−0.0152 (13)
C4	0.0475 (13)	0.0459 (14)	0.0540 (14)	0.0037 (11)	−0.0076 (11)	−0.0056 (11)
C5	0.0654 (17)	0.0724 (19)	0.0653 (17)	−0.0093 (15)	−0.0122 (14)	0.0269 (14)
C6	0.0509 (15)	0.081 (2)	0.0694 (18)	−0.0132 (14)	−0.0042 (13)	0.0272 (15)
C7	0.0423 (13)	0.0497 (14)	0.0463 (13)	0.0028 (11)	−0.0039 (10)	−0.0007 (10)
C8	0.0535 (14)	0.0511 (14)	0.0464 (13)	−0.0022 (12)	−0.0002 (11)	0.0072 (11)
C9	0.0470 (14)	0.0515 (15)	0.0577 (15)	−0.0082 (12)	0.0031 (11)	−0.0010 (12)
C10	0.0511 (16)	0.0691 (18)	0.0523 (15)	0.0021 (14)	0.0005 (12)	0.0068 (13)
C11	0.0546 (15)	0.0506 (15)	0.0741 (17)	−0.0006 (12)	−0.0122 (13)	−0.0093 (13)
C12	0.0502 (14)	0.0396 (13)	0.0561 (15)	−0.0038 (11)	−0.0052 (11)	0.0033 (11)
C13	0.0517 (14)	0.0607 (16)	0.0584 (15)	0.0103 (13)	−0.0014 (12)	−0.0096 (13)
C14	0.0565 (15)	0.0585 (16)	0.0547 (15)	0.0084 (13)	−0.0059 (12)	−0.0061 (12)
C15	0.0464 (13)	0.0427 (13)	0.0564 (15)	−0.0002 (11)	−0.0037 (11)	0.0103 (11)
C16	0.0526 (15)	0.0525 (15)	0.0640 (16)	0.0085 (12)	0.0038 (12)	0.0001 (13)
C17	0.0603 (16)	0.0527 (15)	0.0526 (15)	0.0027 (13)	−0.0019 (12)	−0.0080 (12)
C18	0.0553 (16)	0.0512 (15)	0.0607 (16)	0.0061 (13)	0.0001 (12)	0.0081 (12)
N1	0.0526 (12)	0.0538 (13)	0.0648 (13)	0.0076 (10)	−0.0160 (10)	−0.0139 (10)
N2	0.0525 (12)	0.0532 (13)	0.0647 (13)	0.0061 (10)	−0.0147 (10)	−0.0131 (10)
N3	0.0503 (14)	0.126 (2)	0.0771 (17)	0.0071 (15)	−0.0094 (12)	0.0204 (16)
N4	0.0572 (14)	0.0870 (18)	0.0765 (16)	0.0146 (13)	−0.0119 (12)	0.0068 (13)
S1	0.0657 (4)	0.0441 (4)	0.0664 (4)	0.0074 (3)	−0.0187 (3)	−0.0101 (3)
S2	0.0572 (4)	0.0556 (4)	0.0640 (4)	0.0118 (3)	−0.0159 (3)	−0.0095 (3)
S3	0.0646 (4)	0.0468 (4)	0.0786 (5)	0.0012 (3)	−0.0278 (3)	−0.0094 (3)

Geometric parameters (Å, º)

C1—N1	1.289 (3)	C9—H9	0.9300
C1—S1	1.725 (2)	C10—N3	1.134 (3)

C1—S2	1.742 (2)	C11—C12	1.503 (3)
C2—N2	1.287 (3)	C11—S3	1.799 (3)
C2—S1	1.727 (2)	C11—H11A	0.9700
C2—S3	1.740 (2)	C11—H11B	0.9700
C3—C4	1.499 (3)	C12—C13	1.378 (3)
C3—S2	1.814 (2)	C12—C17	1.382 (3)
C3—H3A	0.9700	C13—C14	1.374 (3)
C3—H3B	0.9700	C13—H13	0.9300
C4—C5	1.381 (3)	C14—C15	1.381 (3)
C4—C9	1.382 (3)	C14—H14	0.9300
C5—C6	1.372 (3)	C15—C16	1.380 (3)
C5—H5	0.9300	C15—C18	1.445 (3)
C6—C7	1.376 (3)	C16—C17	1.373 (3)
C6—H6	0.9300	C16—H16	0.9300
C7—C8	1.371 (3)	C17—H17	0.9300
C7—C10	1.446 (3)	C18—N4	1.136 (3)
C8—C9	1.370 (3)	N1—N2	1.386 (3)
C8—H8	0.9300		

N1—C1—S1	114.56 (17)	C12—C11—S3	111.49 (17)
N1—C1—S2	123.94 (18)	C12—C11—H11A	109.3
S1—C1—S2	121.49 (14)	S3—C11—H11A	109.3
N2—C2—S1	114.47 (17)	C12—C11—H11B	109.3
N2—C2—S3	124.36 (18)	S3—C11—H11B	109.3
S1—C2—S3	121.18 (14)	H11A—C11—H11B	108.0
C4—C3—S2	107.93 (16)	C13—C12—C17	118.7 (2)
C4—C3—H3A	110.1	C13—C12—C11	122.7 (2)
S2—C3—H3A	110.1	C17—C12—C11	118.6 (2)
C4—C3—H3B	110.1	C14—C13—C12	121.2 (2)
S2—C3—H3B	110.1	C14—C13—H13	119.4
H3A—C3—H3B	108.4	C12—C13—H13	119.4
C5—C4—C9	118.5 (2)	C13—C14—C15	119.6 (2)
C5—C4—C3	120.3 (2)	C13—C14—H14	120.2
C9—C4—C3	121.2 (2)	C15—C14—H14	120.2
C6—C5—C4	120.9 (2)	C16—C15—C14	119.7 (2)
C6—C5—H5	119.6	C16—C15—C18	119.9 (2)
C4—C5—H5	119.6	C14—C15—C18	120.4 (2)
C5—C6—C7	119.6 (2)	C17—C16—C15	120.0 (2)
C5—C6—H6	120.2	C17—C16—H16	120.0
C7—C6—H6	120.2	C15—C16—H16	120.0
C8—C7—C6	120.3 (2)	C16—C17—C12	120.7 (2)
C8—C7—C10	120.2 (2)	C16—C17—H17	119.6
C6—C7—C10	119.4 (2)	C12—C17—H17	119.6
C9—C8—C7	119.8 (2)	N4—C18—C15	178.6 (3)
C9—C8—H8	120.1	C1—N1—N2	112.22 (19)
C7—C8—H8	120.1	C2—N2—N1	112.35 (19)
C8—C9—C4	120.9 (2)	C1—S1—C2	86.40 (12)
C8—C9—H9	119.5	C1—S2—C3	99.23 (11)

C4—C9—H9	119.5	C2—S3—C11	98.82 (11)
N3—C10—C7	177.8 (3)		