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2,3-Bis[(2-cyanoethyl)sulfanyl]-1,4,5,8-tetrathiafulvalene-6,7-dicarbonitrile

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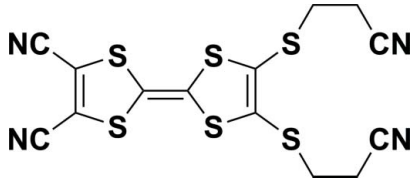
Key indicators: single-crystal X-ray study; T = 290 K; mean σ(C–C) = 0.002 Å; R factor = 0.026; wR factor = 0.072; data-to-parameter ratio = 18.5.

In the title compound, $C_{14}H_8N_4S_6$, the two five-membered rings lie in the same plane with an r.m.s. deviation of 0.0334 (5) Å. The crystal structure features intermolecular S···N interactions of 3.295 (4) Å.

Related literature

For background to the electrical properties of tetrathiafulvalene derivatives, see: Fabre (2000); Batail (2004). For the synthesis, see Chen *et al.* (2005). For a related structure, see: Hou *et al.* (2010).



Experimental

Crystal data

$C_{14}H_8N_4S_6$
M_r = 424.66
Triclinic, $P\overline{1}$
a = 7.3055 (15) Å
b = 8.5193 (17) Å
c = 15.386 (3) Å
α = 82.52 (3)°
β = 76.97 (3)°
γ = 72.43 (3)°
V = 887.4 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.77 mm^{-1}
T = 290 K
0.13 × 0.12 × 0.11 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.906, T_{max} = 0.920
8751 measured reflections
4020 independent reflections
3415 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.072
S = 1.09
4020 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2799).

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supporting information

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2,3-Bis[(2-cyanoethyl)sulfanyl]-1,4,5,8-tetrathiafulvalene-6,7-dicarbonitrile

Cui-Ping Jiang, Bao Li, Bing-Zhu Yin and Li-Xin Wu

S1. Comment

The derivants of tetrathiafulvalene (TTF), especially functionalized with hydroxyl or amine groups, have been studies extensively for their interesting electrical properties (Fabre, 2000; Batail, 2004). Herein, we report the crystal structure of the title compound.

The title compound, as shown in Fig. 1, all bond lengths and angles are normal and comparable with those reported for the related structure (Hou *et al.*, 2010). In the crystal, the two five-membered rings lie in the same plane with an r.m.s. deviation of 0.0334 (5) Å. The intermolecular S···N interactions with the distance of 3.295 (4) Å link the molecules into one-dimensional chain along a+c dirction.

S2. Experimental

The title compound was synthesized as described in the literature for the analogous compound 2,3-Bis(butylthio)-6,7-dicarbonitrile-1,4,5,8- tetrathiafulvalene (Chen *et al.*, 2005). Single crystals and single crystals suitable for X-ray diffraction were prepared by slow evaporation a mixture of dichloromethane and petroleum (60–90 °C) at room temperature.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions with C—H = 0.97 Å and were included in the refinement in the riding model with U_{iso}(H) = 1.2 U_{eq}(C).

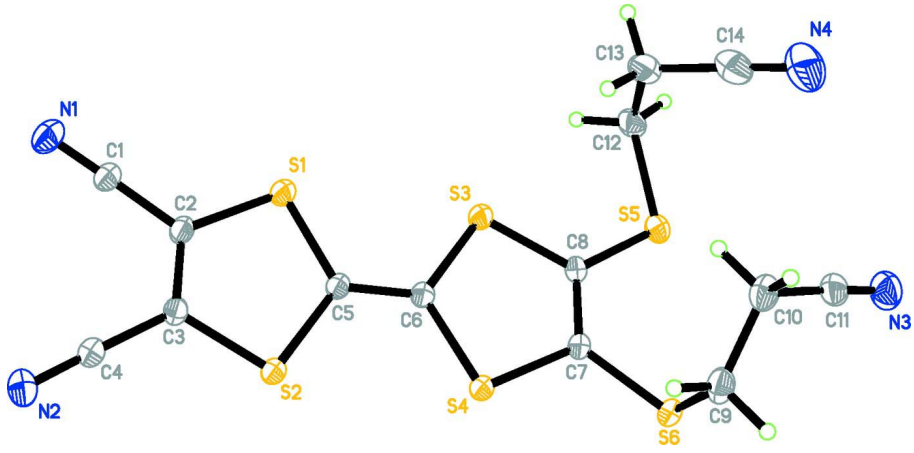

Figure 1

The asymmetric of title compound, with the atom numbering. Displacement ellipsoids of non-H atoms are drawn at the 30% probalility level.

2-{4,5-bis[(cyanomethyl)sulfanyl]-1,3-dithiolan-2-ylidene}-1,3-dithiolane- 4,5-dicarbonitrile

Crystal data

$C_{14}H_8N_4S_6$
$M_r = 424.66$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.3055$ (15) Å
$b = 8.5193$ (17) Å
$c = 15.386$ (3) Å
$\alpha = 82.52$ (3)°
$\beta = 76.97$ (3)°
$\gamma = 72.43$ (3)°
$V = 887.4$ (3) Å^3
$Z = 2$
$F(000) = 432$
$D_x = 1.589$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7465 reflections
$\theta = 3.0$–$27.5°$
$\mu = 0.77$ mm^{-1}
$T = 290$ K
Block, black
0.13 × 0.12 × 0.11 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.906$, $T_{max} = 0.920$
8751 measured reflections
4020 independent reflections
3415 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 9$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.072$
$S = 1.09$
4020 reflections
217 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
$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 0.0566P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Special details

Experimental. (See detailed section in the paper)

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	−0.3844 (2)	0.29787 (18)	−0.14033 (10)	0.0352 (3)
C2	−0.2172 (2)	0.25475 (16)	−0.09903 (9)	0.0298 (3)

C3	−0.0448 (2)	0.14665 (16)	−0.13307 (9)	0.0292 (3)
C4	−0.0139 (2)	0.06331 (18)	−0.21181 (10)	0.0343 (3)
C5	0.01227 (19)	0.24707 (15)	0.00609 (9)	0.0275 (3)
C6	0.09080 (19)	0.27024 (15)	0.07290 (9)	0.0276 (3)
C7	0.3151 (2)	0.25405 (16)	0.18511 (9)	0.0292 (3)
C8	0.14603 (19)	0.36850 (16)	0.21550 (8)	0.0277 (3)
C9	0.4763 (2)	0.01342 (18)	0.30608 (10)	0.0420 (4)
H9A	0.4424	−0.0567	0.2711	0.050*
H9B	0.5960	−0.0498	0.3258	0.050*
C10	0.3135 (2)	0.05787 (19)	0.38801 (10)	0.0428 (4)
H10A	0.1927	0.1205	0.3690	0.051*
H10B	0.2928	−0.0424	0.4210	0.051*
C11	0.3623 (2)	0.1552 (2)	0.44647 (10)	0.0433 (4)
C12	−0.1478 (2)	0.56270 (19)	0.33796 (10)	0.0423 (4)
H12A	−0.1782	0.6517	0.3770	0.051*
H12B	−0.2004	0.6090	0.2847	0.051*
C13	−0.2516 (3)	0.4360 (2)	0.38520 (11)	0.0539 (4)
H13A	−0.3915	0.4887	0.3985	0.065*
H13B	−0.2281	0.3495	0.3454	0.065*
C14	−0.1865 (3)	0.3618 (2)	0.46835 (13)	0.0552 (4)
N1	−0.5150 (2)	0.33249 (19)	−0.17432 (10)	0.0528 (4)
N2	0.0141 (2)	−0.00538 (19)	−0.27451 (10)	0.0527 (4)
N3	0.4075 (2)	0.22886 (19)	0.49019 (10)	0.0591 (4)
N4	−0.1328 (3)	0.3044 (2)	0.53217 (12)	0.0805 (6)
S1	−0.23311 (5)	0.34738 (4)	−0.00256 (2)	0.03481 (10)
S2	0.14881 (5)	0.11197 (4)	−0.07788 (2)	0.03252 (10)
S3	−0.04110 (5)	0.40991 (4)	0.15451 (2)	0.03209 (10)
S4	0.33230 (5)	0.16723 (4)	0.08462 (2)	0.03296 (10)
S5	0.11523 (6)	0.48537 (4)	0.30581 (2)	0.03523 (10)
S6	0.52255 (5)	0.19092 (5)	0.23438 (2)	0.03667 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0287 (7)	0.0404 (7)	0.0378 (8)	−0.0074 (6)	−0.0108 (6)	−0.0051 (6)
C2	0.0279 (7)	0.0340 (7)	0.0304 (7)	−0.0099 (5)	−0.0106 (5)	−0.0011 (5)
C3	0.0290 (7)	0.0339 (7)	0.0276 (7)	−0.0101 (5)	−0.0094 (5)	−0.0024 (5)
C4	0.0298 (7)	0.0405 (7)	0.0339 (8)	−0.0080 (6)	−0.0107 (6)	−0.0040 (6)
C5	0.0256 (6)	0.0296 (6)	0.0279 (6)	−0.0058 (5)	−0.0082 (5)	−0.0033 (5)
C6	0.0266 (7)	0.0298 (6)	0.0265 (6)	−0.0061 (5)	−0.0071 (5)	−0.0036 (5)
C7	0.0284 (7)	0.0358 (7)	0.0247 (6)	−0.0073 (5)	−0.0090 (5)	−0.0039 (5)
C8	0.0299 (7)	0.0316 (7)	0.0232 (6)	−0.0087 (5)	−0.0079 (5)	−0.0026 (5)
C9	0.0485 (9)	0.0365 (8)	0.0406 (8)	−0.0009 (6)	−0.0209 (7)	−0.0058 (6)
C10	0.0468 (9)	0.0429 (8)	0.0410 (8)	−0.0129 (7)	−0.0177 (7)	0.0061 (7)
C11	0.0442 (9)	0.0469 (9)	0.0318 (8)	−0.0025 (7)	−0.0095 (7)	0.0002 (7)
C12	0.0417 (9)	0.0431 (8)	0.0338 (8)	0.0021 (6)	−0.0044 (6)	−0.0126 (6)
C13	0.0451 (10)	0.0769 (12)	0.0416 (9)	−0.0216 (9)	0.0003 (8)	−0.0158 (8)
C14	0.0595 (12)	0.0595 (11)	0.0473 (10)	−0.0259 (9)	0.0031 (9)	−0.0079 (9)

N1	0.0366 (8)	0.0634 (9)	0.0596 (9)	−0.0057 (6)	−0.0221 (7)	−0.0064 (7)
N2	0.0516 (9)	0.0652 (9)	0.0444 (8)	−0.0106 (7)	−0.0157 (7)	−0.0181 (7)
N3	0.0653 (11)	0.0632 (9)	0.0453 (8)	−0.0030 (8)	−0.0172 (8)	−0.0165 (7)
N4	0.1064 (17)	0.0812 (13)	0.0587 (11)	−0.0397 (12)	−0.0161 (11)	0.0099 (10)
S1	0.02631 (18)	0.0407 (2)	0.0367 (2)	−0.00195 (14)	−0.01004 (15)	−0.01111 (15)
S2	0.02509 (18)	0.03996 (19)	0.03258 (19)	−0.00337 (14)	−0.00934 (14)	−0.01026 (15)
S3	0.02685 (18)	0.03621 (19)	0.03086 (18)	−0.00055 (13)	−0.00891 (14)	−0.00827 (14)
S4	0.02534 (18)	0.0421 (2)	0.02945 (18)	−0.00093 (14)	−0.00827 (14)	−0.01089 (14)
S5	0.0400 (2)	0.03828 (19)	0.02918 (18)	−0.01030 (15)	−0.00711 (15)	−0.00961 (14)
S6	0.02798 (19)	0.0510 (2)	0.03197 (19)	−0.00714 (15)	−0.01181 (15)	−0.00518 (16)

Geometric parameters (Å, º)

C1—N1	1.1341 (19)	C9—C10	1.524 (2)
C1—C2	1.4316 (19)	C9—S6	1.8232 (17)
C2—C3	1.355 (2)	C9—H9A	0.9700
C2—S1	1.7352 (15)	C9—H9B	0.9700
C3—C4	1.424 (2)	C10—C11	1.464 (2)
C3—S2	1.7417 (14)	C10—H10A	0.9700
C4—N2	1.1404 (19)	C10—H10B	0.9700
C5—C6	1.3462 (19)	C11—N3	1.136 (2)
C5—S2	1.7611 (15)	C12—C13	1.520 (2)
C5—S1	1.7648 (14)	C12—S5	1.8066 (17)
C6—S3	1.7527 (15)	C12—H12A	0.9700
C6—S4	1.7558 (14)	C12—H12B	0.9700
C7—C8	1.351 (2)	C13—C14	1.465 (3)
C7—S6	1.7553 (14)	C13—H13A	0.9700
C7—S4	1.7628 (14)	C13—H13B	0.9700
C8—S5	1.7483 (14)	C14—N4	1.136 (2)
C8—S3	1.7536 (14)		
N1—C1—C2	178.90 (18)	C11—C10—C9	111.21 (14)
C3—C2—C1	122.75 (13)	C11—C10—H10A	109.4
C3—C2—S1	118.32 (11)	C9—C10—H10A	109.4
C1—C2—S1	118.92 (11)	C11—C10—H10B	109.4
C2—C3—C4	123.46 (13)	C9—C10—H10B	109.4
C2—C3—S2	117.65 (11)	H10A—C10—H10B	108.0
C4—C3—S2	118.88 (10)	N3—C11—C10	177.38 (19)
N2—C4—C3	178.71 (16)	C13—C12—S5	115.32 (11)
C6—C5—S2	121.71 (11)	C13—C12—H12A	108.4
C6—C5—S1	122.52 (11)	S5—C12—H12A	108.4
S2—C5—S1	115.77 (8)	C13—C12—H12B	108.4
C5—C6—S3	122.05 (11)	S5—C12—H12B	108.4
C5—C6—S4	123.27 (11)	H12A—C12—H12B	107.5
S3—C6—S4	114.67 (8)	C14—C13—C12	112.75 (15)
C8—C7—S6	125.66 (11)	C14—C13—H13A	109.0
C8—C7—S4	117.08 (10)	C12—C13—H13A	109.0
S6—C7—S4	117.20 (8)	C14—C13—H13B	109.0

C7—C8—S5	123.02 (11)	C12—C13—H13B	109.0
C7—C8—S3	117.09 (10)	H13A—C13—H13B	107.8
S5—C8—S3	119.69 (8)	N4—C14—C13	178.8 (2)
C10—C9—S6	114.19 (10)	C2—S1—C5	94.05 (7)
C10—C9—H9A	108.7	C3—S2—C5	94.21 (7)
S6—C9—H9A	108.7	C6—S3—C8	95.71 (7)
C10—C9—H9B	108.7	C6—S4—C7	95.29 (7)
S6—C9—H9B	108.7	C8—S5—C12	102.98 (7)
H9A—C9—H9B	107.6	C7—S6—C9	100.68 (7)

N1—C1—C2—C3	−50 (9)	C1—C2—S1—C5	−178.05 (12)
N1—C1—C2—S1	129 (9)	C6—C5—S1—C2	−179.46 (12)
C1—C2—C3—C4	−1.2 (2)	S2—C5—S1—C2	−0.12 (8)
S1—C2—C3—C4	−179.88 (11)	C2—C3—S2—C5	0.78 (12)
C1—C2—C3—S2	177.64 (11)	C4—C3—S2—C5	179.67 (11)
S1—C2—C3—S2	−1.04 (16)	C6—C5—S2—C3	179.05 (12)
C2—C3—C4—N2	−168 (8)	S1—C5—S2—C3	−0.29 (9)
S2—C3—C4—N2	14 (8)	C5—C6—S3—C8	178.64 (12)
S2—C5—C6—S3	177.67 (7)	S4—C6—S3—C8	−2.39 (9)
S1—C5—C6—S3	−3.04 (18)	C7—C8—S3—C6	−0.40 (12)
S2—C5—C6—S4	−1.21 (18)	S5—C8—S3—C6	174.61 (8)
S1—C5—C6—S4	178.08 (7)	C5—C6—S4—C7	−177.40 (12)
S6—C7—C8—S5	5.36 (19)	S3—C6—S4—C7	3.65 (9)
S4—C7—C8—S5	−171.73 (7)	C8—C7—S4—C6	−4.05 (12)
S6—C7—C8—S3	−179.81 (8)	S6—C7—S4—C6	178.61 (8)
S4—C7—C8—S3	3.10 (16)	C7—C8—S5—C12	−162.25 (12)
S6—C9—C10—C11	−62.05 (14)	S3—C8—S5—C12	23.05 (10)
C9—C10—C11—N3	−17 (4)	C13—C12—S5—C8	73.13 (13)
S5—C12—C13—C14	59.88 (18)	C8—C7—S6—C9	93.08 (14)
C12—C13—C14—N4	−35 (11)	S4—C7—S6—C9	−89.84 (9)
C3—C2—S1—C5	0.68 (12)	C10—C9—S6—C7	−72.76 (12)