Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,3-Bis(ethylsulfanyl)-1,4,5,8-tetrathiafulvalene-6,7-dicarbonitrile

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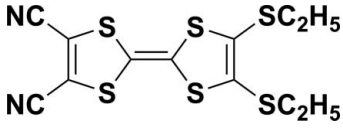
Received 1 July 2011; accepted 16 July 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.109; data-to-parameter ratio = 20.2.

In the title compound, $C_{12}H_{10}N_2S_6$, all non-H atoms, except for those in the ethyl groups, lie in the same non-crystallographic plane, with a r.m.s. deviation of 0.0366 (5) Å. In the crystal structure, molecules are linked through weak C—H···N hydrogen bonds between methyl and cyano groups, forming centrosymmetric dimers. The dimers are arranged along the a axis, due to intermolecular N···S [3.337 (4) Å] interactions.

Related literature

For synthetic uses of dicyano-substituted tetrathiafulvalene derivatives, see: Chen *et al.* (2007); Leng *et al.* (2010). For a related structure, see: Jiang *et al.* (2010). For the synthesis of the title compound, see: Chen *et al.* (2005).



Experimental

Crystal data

$C_{12}H_{10}N_2S_6$
M_r = 374.58
Triclinic, $P\overline{1}$
a = 7.8357 (16) Å
b = 8.9777 (18) Å
c = 12.618 (3) Å
α = 76.48 (3)°
β = 77.59 (3)°
γ = 73.20 (3)°
V = 815.8 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.83 mm^{-1}
T = 293 K
0.15 × 0.13 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.886, T_{max} = 0.907
8038 measured reflections
3689 independent reflections
3079 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.109
S = 1.15
3689 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10C···N2^{i}	0.96	2.73	3.659 (4)	164

Symmetry code: (i) $-x+2, -y+1, -z+2$.

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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

The authors acknowledge financial support from the National Natural Science Foundation of China (grant No. 21062022).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2367).

References

Chen, T., Wang, C. L., Cong, Z. Q., Yin, B. Z. & Imafuku, K. (2005). *Heterocycles*, **65**, 187–193.
Chen, T., Wang, C. L., Qiu, H., Jin, L. Y., Yin, B. Z. & Imafuku, K. (2007). *Heterocycles*, **71**, 549–555.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Jiang, C.-P., Li, B., Yin, B.-Z. & Wu, L.-X. (2010). *Acta Cryst.* E**66**, o2079.
Leng, F. S., Wang, X. S., Jin, L. Y. & Yin, B. Z. (2010). *Dyes Pigm.* **87**, 89–94.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2011). E**67**, o2096 [**doi:10.1107/S1600536811028601**]

2,3-Bis(ethylsulfanyl)-1,4,5,8-tetrathiafulvalene-6,7-dicarbonitrile

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Comment

Dicyano-substituted tetrathiafulvalene derivatives (TTFs) are key precursors for the preparation of the TTF-annulated prophyrazines. We have recently synthesized the symmetrical (Chen *et al.*, 2007) and the unsymmetrical TTF-annulated porphyrazines (Leng *et al.*, 2010) using such precursors. In this paper, we report the crystal structure of the title compound.

In the title compound (Fig. 1), all bond lengths and angles are in the normal ranges and comparable with those observed in a closely related compound (Jiang *et al.*, 2010). In the title compound, except for two ethyl groups, all atoms lie on the same plane. In the crystal, the molecules form dimers through weak intermolecular C—H···N hydrogen bonds (Table 1), and dimers are arranged along the *a* axis, due to N···S interactions.

Experimental

The title compound was prepared according to the literature (Chen *et al.*, 2005). Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a solution in a mixture of dichloromethane and petroleum ether, at room temperature.

Refinement

C-bound H-atoms were placed in calculated positions (C—H 0.96 or 0.97 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) = 1.5 U_{eq}(C) for methyl groups and U_{iso}(H) = 1.2 U_{eq}(C) for methylene groups.

Figures

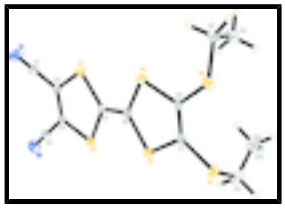

Fig. 1. The crystal structure of the title compound, with displacement ellipsoids for non-H atoms drawn at the 20% probability level.

2,3-Bis(ethylsulfanyl)-1,4,5,8-tetrathiafulvalene-6,7-dicarbonitrile

Crystal data

$C_{12}H_{10}N_2S_6$	$Z = 2$
$M_r = 374.58$	$F(000) = 384$
Triclinic, $P\overline{1}$	$D_x = 1.525$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.8357$ (16) Å	Cell parameters from 3994 reflections

b = 8.9777 (18) Å	θ = 3.2–27.5°
c = 12.618 (3) Å	μ = 0.83 mm^{-1}
α = 76.48 (3)°	T = 293 K
β = 77.59 (3)°	Block, black
γ = 73.20 (3)°	0.15 × 0.13 × 0.12 mm
V = 815.8 (3) Å^3	

Data collection

Rigaku R-AXIS RAPID diffractometer	3689 independent reflections
Radiation source: fine-focus sealed tube	3079 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.022
ω scans	θ_{max} = 27.5°, θ_{min} = 3.2°
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	h = −10→10
T_{min} = 0.886, T_{max} = 0.907	k = −11→10
8038 measured reflections	l = −16→16

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.031	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.109	H-atom parameters constrained
S = 1.15	$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2 + 0.1175P]$ where $P = (F_o^2 + 2F_c^2)/3$
3689 reflections	$(\Delta/\sigma)_{max}$ = 0.001
183 parameters	$\Delta\rho_{max}$ = 0.37 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.42 e Å^{-3}
0 constraints	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	1.3781 (3)	0.3004 (3)	1.17179 (18)	0.0471 (5)
C2	1.2353 (2)	0.2956 (2)	1.11964 (14)	0.0346 (4)
C3	1.0593 (2)	0.3581 (2)	1.15573 (14)	0.0325 (4)
C4	0.9973 (3)	0.4385 (2)	1.24706 (16)	0.0404 (4)
C5	1.0603 (2)	0.2358 (2)	0.99101 (14)	0.0312 (4)
C6	1.0078 (2)	0.1834 (2)	0.91471 (14)	0.0314 (4)
C7	0.8174 (3)	0.1324 (2)	0.78825 (14)	0.0362 (4)
C8	0.9902 (3)	0.0668 (2)	0.74847 (14)	0.0348 (4)
C9	1.2580 (3)	0.0150 (3)	0.56711 (17)	0.0514 (5)
H9A	1.3429	−0.0101	0.6183	0.062*
H9B	1.3115	−0.0489	0.5104	0.062*

C10	1.2308 (4)	0.1859 (3)	0.5143 (2)	0.0673 (7)
H10A	1.1425	0.2134	0.4662	0.101*
H10B	1.3430	0.2042	0.4725	0.101*
H10C	1.1895	0.2499	0.5705	0.101*
C11	0.5366 (3)	0.3331 (3)	0.6882 (2)	0.0658 (7)
H11A	0.4252	0.3457	0.6615	0.079*
H11B	0.5078	0.3900	0.7489	0.079*
C12	0.6628 (5)	0.4051 (4)	0.5971 (3)	0.1003 (13)
H12A	0.7769	0.3859	0.6210	0.150*
H12B	0.6122	0.5171	0.5789	0.150*
H12C	0.6801	0.3584	0.5333	0.150*
N1	1.4901 (3)	0.3032 (3)	1.21488 (19)	0.0733 (6)
N2	0.9451 (3)	0.5027 (2)	1.31940 (17)	0.0632 (5)
S1	1.28831 (6)	0.20402 (6)	1.00515 (4)	0.03914 (14)
S2	0.89965 (6)	0.33925 (6)	1.08715 (4)	0.03802 (14)
S3	0.77877 (6)	0.21620 (6)	0.90659 (4)	0.04141 (14)
S4	1.15711 (6)	0.07338 (6)	0.81983 (4)	0.03804 (14)
S5	0.62815 (7)	0.12625 (7)	0.73859 (4)	0.04650 (16)
S6	1.05259 (8)	−0.03845 (7)	0.64039 (4)	0.04750 (16)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0377 (10)	0.0631 (13)	0.0461 (11)	−0.0110 (10)	−0.0063 (9)	−0.0232 (10)
C2	0.0360 (9)	0.0404 (9)	0.0320 (9)	−0.0122 (8)	−0.0083 (7)	−0.0094 (7)
C3	0.0369 (9)	0.0348 (9)	0.0290 (8)	−0.0107 (7)	−0.0058 (7)	−0.0090 (7)
C4	0.0420 (10)	0.0436 (10)	0.0369 (10)	−0.0073 (9)	−0.0071 (8)	−0.0136 (8)
C5	0.0324 (8)	0.0345 (9)	0.0288 (8)	−0.0090 (7)	−0.0058 (7)	−0.0081 (7)
C6	0.0341 (8)	0.0355 (9)	0.0274 (8)	−0.0107 (7)	−0.0068 (7)	−0.0070 (7)
C7	0.0404 (9)	0.0443 (10)	0.0301 (9)	−0.0177 (8)	−0.0111 (7)	−0.0050 (7)
C8	0.0434 (9)	0.0416 (9)	0.0255 (8)	−0.0183 (8)	−0.0084 (7)	−0.0060 (7)
C9	0.0471 (11)	0.0647 (14)	0.0430 (11)	−0.0095 (11)	0.0012 (9)	−0.0245 (10)
C10	0.0797 (18)	0.0730 (16)	0.0516 (14)	−0.0346 (14)	0.0086 (12)	−0.0144 (12)
C11	0.0585 (14)	0.0618 (15)	0.0846 (18)	−0.0060 (12)	−0.0410 (14)	−0.0121 (13)
C12	0.115 (3)	0.085 (2)	0.110 (3)	−0.050 (2)	−0.065 (2)	0.0395 (19)
N1	0.0473 (11)	0.1120 (18)	0.0767 (15)	−0.0167 (12)	−0.0174 (11)	−0.0453 (14)
N2	0.0704 (13)	0.0684 (13)	0.0527 (12)	−0.0068 (11)	−0.0075 (10)	−0.0303 (10)
S1	0.0314 (2)	0.0513 (3)	0.0399 (3)	−0.0088 (2)	−0.00407 (19)	−0.0216 (2)
S2	0.0303 (2)	0.0491 (3)	0.0386 (3)	−0.0089 (2)	−0.00466 (18)	−0.0176 (2)
S3	0.0336 (2)	0.0591 (3)	0.0365 (3)	−0.0109 (2)	−0.00710 (19)	−0.0180 (2)
S4	0.0348 (2)	0.0504 (3)	0.0336 (3)	−0.0103 (2)	−0.00648 (18)	−0.0162 (2)
S5	0.0454 (3)	0.0585 (3)	0.0465 (3)	−0.0236 (3)	−0.0173 (2)	−0.0084 (2)
S6	0.0633 (3)	0.0552 (3)	0.0346 (3)	−0.0267 (3)	−0.0028 (2)	−0.0185 (2)

Geometric parameters (Å, °)

C1—N1	1.136 (3)	C8—S4	1.7606 (18)
C1—C2	1.430 (3)	C9—C10	1.496 (3)
C2—C3	1.352 (3)	C9—S6	1.810 (2)

C2—S1	1.7423 (19)	C9—H9A	0.9700
C3—C4	1.425 (2)	C9—H9B	0.9700
C3—S2	1.7314 (18)	C10—H10A	0.9600
C4—N2	1.132 (3)	C10—H10B	0.9600
C5—C6	1.346 (2)	C10—H10C	0.9600
C5—S2	1.7646 (19)	C11—C12	1.500 (4)
C5—S1	1.7673 (18)	C11—S5	1.800 (3)
C6—S4	1.7495 (19)	C11—H11A	0.9700
C6—S3	1.7543 (18)	C11—H11B	0.9700
C7—C8	1.348 (3)	C12—H12A	0.9600
C7—S5	1.7483 (18)	C12—H12B	0.9600
C7—S3	1.7569 (19)	C12—H12C	0.9600
C8—S6	1.7439 (19)		
N1—C1—C2	178.9 (3)	H9A—C9—H9B	107.7
C3—C2—C1	122.92 (17)	C9—C10—H10A	109.5
C3—C2—S1	117.99 (14)	C9—C10—H10B	109.5
C1—C2—S1	119.09 (15)	H10A—C10—H10B	109.5
C2—C3—C4	123.81 (17)	C9—C10—H10C	109.5
C2—C3—S2	118.14 (13)	H10A—C10—H10C	109.5
C4—C3—S2	118.05 (14)	H10B—C10—H10C	109.5
N2—C4—C3	178.8 (2)	C12—C11—S5	113.3 (2)
C6—C5—S2	120.78 (14)	C12—C11—H11A	108.9
C6—C5—S1	123.79 (15)	S5—C11—H11A	108.9
S2—C5—S1	115.42 (10)	C12—C11—H11B	108.9
C5—C6—S4	123.86 (14)	S5—C11—H11B	108.9
C5—C6—S3	121.49 (15)	H11A—C11—H11B	107.7
S4—C6—S3	114.62 (10)	C11—C12—H12A	109.5
C8—C7—S5	125.25 (15)	C11—C12—H12B	109.5
C8—C7—S3	117.19 (14)	H12A—C12—H12B	109.5
S5—C7—S3	117.34 (11)	C11—C12—H12C	109.5
C7—C8—S6	123.52 (14)	H12A—C12—H12C	109.5
C7—C8—S4	116.94 (14)	H12B—C12—H12C	109.5
S6—C8—S4	119.23 (11)	C2—S1—C5	94.04 (9)
C10—C9—S6	113.94 (17)	C3—S2—C5	94.40 (8)
C10—C9—H9A	108.8	C6—S3—C7	95.39 (9)
S6—C9—H9A	108.8	C6—S4—C8	95.48 (9)
C10—C9—H9B	108.8	C7—S5—C11	101.18 (10)
S6—C9—H9B	108.8	C8—S6—C9	102.91 (10)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10C···N2^{i}	0.96	2.73	3.659 (4)	164.

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+2.

Fig. 1