Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,3-(3,6,9-Trioxaundecane-1,11-diyldisulfanyl)-1,4,5,8-tetrathiafulvalene-6,7-dicarbonitrile

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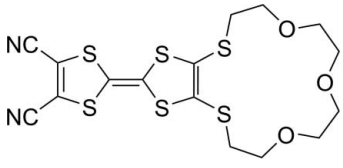
Received 12 May 2010; accepted 13 May 2010

Key indicators: single-crystal X-ray study; T = 290 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.088; data-to-parameter ratio = 19.2.

In the title compound, $C_{16}H_{16}N_2O_3S_6$, the two five-membered rings form a dihedral angle of 7.86 (9)°. Weak C—H···N hydrogen bonds link the molecules to form a chain along c; the chains are further connected by weak C—H···O hydrogen bonds to form a three-dimensional supramolecular network.

Related literature

For background to the use of dithiacrown ether annulated tetrathiafulvalenes as sensor molecules for various metal cations, see Moore *et al.* (2000); Otsubo & Ogura (1985). For the synthesis, see Yin *et al.* (2006). For a related structure, see Hou *et al.* (2009).



Experimental

Crystal data

$C_{16}H_{16}N_2O_3S_6$
M_r = 476.67
Triclinic, $P\overline{1}$
a = 8.300 (5) Å
b = 9.186 (5) Å
c = 13.892 (10) Å
α = 100.42 (3)°
β = 92.31 (3)°
γ = 95.60 (2)°
V = 1035.0 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.68 mm^{-1}
T = 290 K
0.13 × 0.12 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.917, T_{max} = 0.935
10214 measured reflections
4702 independent reflections
3936 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.088
S = 1.07
4702 reflections
245 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13A···N1^{i}	0.97	2.65	3.534 (3)	152
C14—H14A···N1^{ii}	0.97	2.74	3.691 (3)	168
C14—H14B···O1^{iii}	0.97	2.64	3.401 (3)	136
C9—H9A···O2^{iv}	0.97	2.57	3.406 (3)	144
C15—H15B···O3^{v}	0.97	2.47	3.317 (2)	146

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

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*.

The authors acknowledge financial support from the National Natural Science Foundation of China (grant No. 20662010), the Specialized Research Fund for the Doctoral Program of Higher Education (grant No. 2006184001) and the Open Project of the State Key Laboratory of Supramolecular Structure and Materials, Jilin University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2773).

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supplementary materials

Acta Cryst. (2010). E**66**, o1379 [**doi:10.1107/S1600536810017587**]

2,3-(3,6,9-Trioxaundecane-1,11-diyldisulfanyl)-1,4,5,8-tetrathiafulvalene-6,7-dicarbonitrile

R.-B. Hou, B. Li, T. Chen, B.-Z. Yin and L.-X. Wu

Comment

Dithiacrown ether annulated tetrathiafulvalenes have received great attentions as sensors molecule for various metal cations (Otsubo *et al.*, 1985; Moore *et al.*, 2000). These sensors can recognize selectively the virous metal cations to singel electrochemical information. We incorpolated TTF with a 15-membered O, S hybrid crown ether to synthesize the title compound because it should be able to bind sodium ion (Yin *et al.*, 2006). We report herein the synthesis and structure of the title compound.

The title compound, (I), as shown in Fig. 1, all bond lengths and angles are normal and comparable with those reported for the related structure (Hou *et al.*, 2009). In the crystal, weak C—H···O hydrogen bonds (table 1) link the molecules into two-dimensional network in ac plane. The crystal structure is further stablized by weak C—H···N hydrogen bonds along c dirction.

Experimental

The title compound, (I), was prepared according to literature (Yin *et al.*, 2006) 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.

Refinement

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

Figures

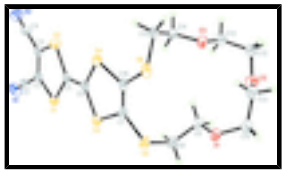


Fig. 1. The asymmetric unit of the title compound, with the atom numbering. Displacement ellipsoids of non-H atoms are drawn at the 30% probalility level.

2,3-(3,6,9-Trioxaundecane-1,11-diyldisulfanyl)-1,4,5,8-tetrathiafulvalene- 6,7-dicarbonitrile

Crystal data

$C_{16}H_{16}N_2O_3S_6$	$Z = 2$
$M_r = 476.67$	$F(000) = 492$
Triclinic, $P\overline{1}$	$D_x = 1.530$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

a = 8.300 (5) Å	Cell parameters from 8487 reflections
b = 9.186 (5) Å	θ = 3.2–27.5°
c = 13.892 (10) Å	μ = 0.68 mm^{-1}
α = 100.42 (3)°	T = 290 K
β = 92.31 (3)°	Block, black
γ = 95.60 (2)°	0.13 × 0.12 × 0.10 mm
V = 1035.0 (11) $Å^3$	

Data collection

Rigaku R-AXIS RAPID diffractometer	4702 independent reflections
Radiation source: fine-focus sealed tube	3936 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.026
ω scans	θ_{max} = 27.5°, θ_{min} = 3.2°
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$h = -10 \rightarrow 10$
T_{min} = 0.917, T_{max} = 0.935	$k = -10 \rightarrow 11$
10214 measured reflections	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.032	H-atom parameters constrained
$wR(F^2)$ = 0.088	$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 0.1948P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max}$ = 0.001
4702 reflections	$\Delta\rho_{max}$ = 0.52 e $Å^{-3}$
245 parameters	$\Delta\rho_{min}$ = −0.31 e $Å^{-3}$
0 restraints	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.027 (2)

Special details

Experimental. (See detailed section in the paper)

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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	−0.1516 (2)	0.9624 (2)	−0.20634 (12)	0.0455 (4)
C2	−0.1520 (2)	0.88171 (17)	−0.12738 (11)	0.0364 (3)
C3	−0.2869 (2)	0.84467 (18)	−0.08319 (11)	0.0377 (3)
C4	−0.4436 (2)	0.8829 (2)	−0.10925 (13)	0.0493 (4)
C5	−0.05873 (19)	0.74212 (17)	0.00468 (11)	0.0353 (3)
C6	0.02801 (19)	0.68050 (18)	0.06787 (11)	0.0374 (3)
C7	0.2533 (2)	0.60022 (18)	0.17572 (11)	0.0406 (4)
C8	0.5249 (2)	0.7500 (2)	0.28490 (13)	0.0470 (4)
H8A	0.6417	0.7629	0.2811	0.056*
H8B	0.4803	0.8258	0.2548	0.056*
C9	0.4861 (2)	0.77382 (19)	0.39115 (12)	0.0428 (4)
H9A	0.5244	0.8748	0.4233	0.051*
H9B	0.3699	0.7581	0.3967	0.051*
C10	0.5910 (3)	0.7045 (3)	0.53927 (15)	0.0621 (5)
H10A	0.6581	0.7990	0.5566	0.075*
H10B	0.6520	0.6293	0.5596	0.075*
C11	0.4441 (3)	0.7141 (2)	0.59613 (16)	0.0625 (5)
H11A	0.3571	0.6423	0.5626	0.075*
H11B	0.4667	0.6906	0.6603	0.075*
C12	0.2389 (2)	0.8716 (2)	0.64099 (13)	0.0506 (4)
H12A	0.2396	0.9632	0.6884	0.061*
H12B	0.2088	0.7892	0.6738	0.061*
C13	0.1159 (2)	0.87039 (19)	0.55895 (14)	0.0499 (4)
H13A	0.0166	0.9038	0.5853	0.060*
H13B	0.1566	0.9378	0.5171	0.060*
C14	−0.0256 (2)	0.71693 (19)	0.42162 (12)	0.0401 (4)
H14A	0.0240	0.7710	0.3747	0.048*
H14B	−0.1227	0.7616	0.4424	0.048*
C15	−0.0681 (2)	0.55669 (19)	0.37564 (12)	0.0434 (4)
H15A	−0.1587	0.5490	0.3280	0.052*
H15B	−0.1029	0.5019	0.4260	0.052*
C16	0.1146 (2)	0.56287 (17)	0.21572 (11)	0.0385 (4)
N1	−0.1521 (3)	1.0257 (2)	−0.26936 (13)	0.0688 (5)
N2	−0.5676 (2)	0.9136 (3)	−0.12990 (15)	0.0767 (6)
O1	0.56498 (15)	0.67023 (14)	0.43571 (9)	0.0494 (3)
O2	0.39558 (16)	0.85988 (14)	0.60659 (10)	0.0540 (3)
O3	0.08385 (15)	0.72386 (12)	0.50341 (8)	0.0419 (3)
S1	0.02900 (5)	0.82515 (5)	−0.08826 (3)	0.04072 (12)
S2	−0.27073 (5)	0.74152 (6)	0.00882 (3)	0.04764 (13)
S3	0.23772 (5)	0.67410 (5)	0.06743 (3)	0.04530 (12)
S4	−0.06611 (5)	0.59369 (5)	0.15614 (3)	0.04415 (12)
S5	0.44704 (6)	0.56746 (5)	0.21477 (4)	0.05140 (14)
S6	0.09787 (6)	0.47074 (5)	0.31522 (3)	0.04736 (13)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0563 (10)	0.0477 (9)	0.0365 (8)	0.0147 (8)	0.0069 (7)	0.0124 (7)
C2	0.0451 (8)	0.0380 (8)	0.0278 (7)	0.0106 (7)	0.0008 (6)	0.0076 (6)
C3	0.0410 (8)	0.0444 (8)	0.0301 (7)	0.0123 (7)	−0.0020 (6)	0.0100 (6)
C4	0.0463 (10)	0.0674 (11)	0.0386 (9)	0.0175 (9)	0.0003 (7)	0.0160 (8)
C5	0.0369 (8)	0.0431 (8)	0.0279 (7)	0.0110 (7)	0.0006 (6)	0.0083 (6)
C6	0.0407 (8)	0.0448 (8)	0.0274 (7)	0.0113 (7)	−0.0024 (6)	0.0064 (6)
C7	0.0447 (9)	0.0432 (8)	0.0333 (8)	0.0142 (7)	−0.0099 (7)	0.0031 (6)
C8	0.0429 (9)	0.0542 (10)	0.0452 (9)	0.0016 (8)	−0.0058 (7)	0.0167 (8)
C9	0.0398 (8)	0.0426 (9)	0.0456 (9)	0.0028 (7)	−0.0038 (7)	0.0097 (7)
C10	0.0551 (11)	0.0863 (15)	0.0479 (11)	0.0191 (11)	−0.0054 (9)	0.0160 (10)
C11	0.0723 (14)	0.0645 (12)	0.0598 (12)	0.0214 (11)	0.0145 (10)	0.0259 (10)
C12	0.0559 (11)	0.0452 (9)	0.0458 (10)	0.0001 (8)	0.0052 (8)	−0.0024 (8)
C13	0.0561 (11)	0.0388 (9)	0.0544 (10)	0.0115 (8)	0.0050 (8)	0.0037 (8)
C14	0.0381 (8)	0.0480 (9)	0.0399 (8)	0.0100 (7)	0.0039 (6)	0.0202 (7)
C15	0.0413 (8)	0.0516 (9)	0.0391 (8)	−0.0083 (7)	−0.0085 (7)	0.0230 (7)
C16	0.0477 (9)	0.0382 (8)	0.0299 (7)	0.0131 (7)	−0.0096 (7)	0.0048 (6)
N1	0.0948 (14)	0.0705 (11)	0.0523 (10)	0.0233 (10)	0.0155 (9)	0.0307 (9)
N2	0.0525 (10)	0.1179 (17)	0.0673 (12)	0.0318 (11)	−0.0040 (9)	0.0268 (11)
O1	0.0477 (7)	0.0621 (8)	0.0408 (6)	0.0152 (6)	−0.0050 (5)	0.0127 (6)
O2	0.0509 (7)	0.0463 (7)	0.0644 (8)	0.0010 (6)	0.0077 (6)	0.0103 (6)
O3	0.0508 (7)	0.0355 (6)	0.0399 (6)	0.0046 (5)	−0.0055 (5)	0.0104 (5)
S1	0.0380 (2)	0.0521 (2)	0.0354 (2)	0.01113 (18)	0.00434 (16)	0.01327 (17)
S2	0.0403 (2)	0.0657 (3)	0.0470 (2)	0.0160 (2)	0.00757 (18)	0.0305 (2)
S3	0.0407 (2)	0.0629 (3)	0.0344 (2)	0.0145 (2)	−0.00208 (16)	0.01077 (19)
S4	0.0418 (2)	0.0618 (3)	0.0331 (2)	0.01220 (19)	−0.00430 (16)	0.01804 (18)
S5	0.0472 (3)	0.0551 (3)	0.0507 (3)	0.0212 (2)	−0.0144 (2)	0.0019 (2)
S6	0.0675 (3)	0.0392 (2)	0.0371 (2)	0.0133 (2)	−0.0134 (2)	0.01131 (17)

Geometric parameters (Å, °)

C1—N1	1.135 (2)	C10—C11	1.480 (3)
C1—C2	1.430 (2)	C10—H10A	0.9700
C2—C3	1.343 (2)	C10—H10B	0.9700
C2—S1	1.7352 (18)	C11—O2	1.420 (3)
C3—C4	1.430 (2)	C11—H11A	0.9700
C3—S2	1.7318 (18)	C11—H11B	0.9700
C4—N2	1.133 (3)	C12—O2	1.411 (2)
C5—C6	1.348 (2)	C12—C13	1.497 (3)
C5—S1	1.7615 (18)	C12—H12A	0.9700
C5—S2	1.7625 (19)	C12—H12B	0.9700
C6—S3	1.7476 (19)	C13—O3	1.420 (2)
C6—S4	1.7490 (19)	C13—H13A	0.9700
C7—C16	1.340 (3)	C13—H13B	0.9700
C7—S5	1.7479 (19)	C14—O3	1.414 (2)
C7—S3	1.764 (2)	C14—C15	1.496 (2)

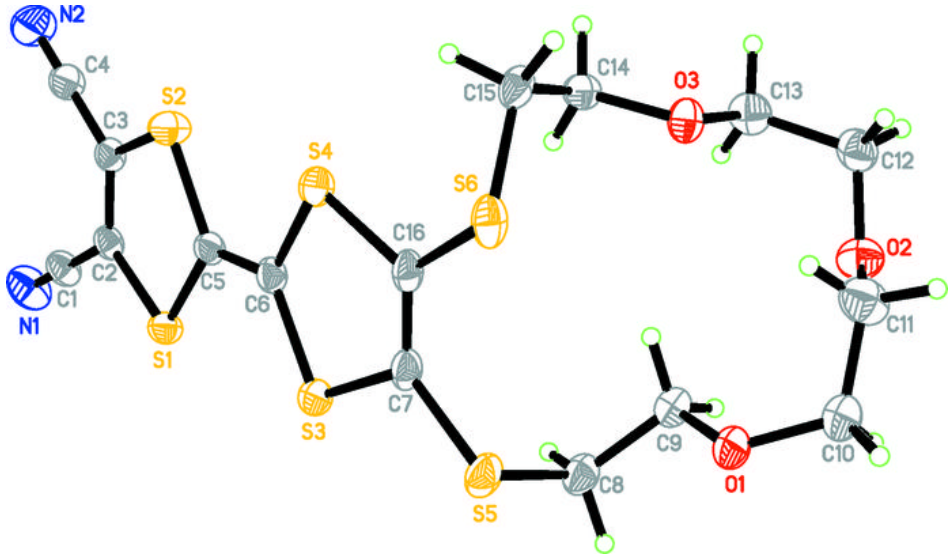
C8—C9	1.505 (3)	C14—H14A	0.9700
C8—S5	1.823 (2)	C14—H14B	0.9700
C8—H8A	0.9700	C15—S6	1.814 (2)
C8—H8B	0.9700	C15—H15A	0.9700
C9—O1	1.421 (2)	C15—H15B	0.9700
C9—H9A	0.9700	C16—S6	1.7499 (19)
C9—H9B	0.9700	C16—S4	1.7555 (18)
C10—O1	1.419 (2)		
N1—C1—C2	179.5 (2)	C10—C11—H11B	109.7
C3—C2—C1	123.03 (15)	H11A—C11—H11B	108.2
C3—C2—S1	118.17 (13)	O2—C12—C13	111.50 (16)
C1—C2—S1	118.78 (13)	O2—C12—H12A	109.3
C2—C3—C4	123.67 (16)	C13—C12—H12A	109.3
C2—C3—S2	118.12 (12)	O2—C12—H12B	109.3
C4—C3—S2	118.19 (14)	C13—C12—H12B	109.3
N2—C4—C3	179.9 (3)	H12A—C12—H12B	108.0
C6—C5—S1	123.07 (13)	O3—C13—C12	109.47 (15)
C6—C5—S2	121.53 (13)	O3—C13—H13A	109.8
S1—C5—S2	115.39 (9)	C12—C13—H13A	109.8
C5—C6—S3	124.32 (14)	O3—C13—H13B	109.8
C5—C6—S4	121.23 (14)	C12—C13—H13B	109.8
S3—C6—S4	114.42 (9)	H13A—C13—H13B	108.2
C16—C7—S5	125.91 (13)	O3—C14—C15	108.09 (13)
C16—C7—S3	117.11 (12)	O3—C14—H14A	110.1
S5—C7—S3	116.72 (11)	C15—C14—H14A	110.1
C9—C8—S5	114.18 (13)	O3—C14—H14B	110.1
C9—C8—H8A	108.7	C15—C14—H14B	110.1
S5—C8—H8A	108.7	H14A—C14—H14B	108.4
C9—C8—H8B	108.7	C14—C15—S6	113.74 (12)
S5—C8—H8B	108.7	C14—C15—H15A	108.8
H8A—C8—H8B	107.6	S6—C15—H15A	108.8
O1—C9—C8	107.67 (14)	C14—C15—H15B	108.8
O1—C9—H9A	110.2	S6—C15—H15B	108.8
C8—C9—H9A	110.2	H15A—C15—H15B	107.7
O1—C9—H9B	110.2	C7—C16—S6	125.35 (13)
C8—C9—H9B	110.2	C7—C16—S4	116.95 (13)
H9A—C9—H9B	108.5	S6—C16—S4	117.42 (11)
O1—C10—C11	116.36 (18)	C10—O1—C9	116.55 (15)
O1—C10—H10A	108.2	C12—O2—C11	113.79 (15)
C11—C10—H10A	108.2	C14—O3—C13	112.15 (13)
O1—C10—H10B	108.2	C2—S1—C5	94.00 (8)
C11—C10—H10B	108.2	C3—S2—C5	94.10 (8)
H10A—C10—H10B	107.4	C6—S3—C7	95.23 (8)
O2—C11—C10	109.80 (18)	C6—S4—C16	95.58 (9)
O2—C11—H11A	109.7	C7—S5—C8	101.42 (9)
C10—C11—H11A	109.7	C16—S6—C15	100.76 (8)
O2—C11—H11B	109.7		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13A···N1^{i}	0.97	2.65	3.534 (3)	152.
C14—H14A···N1^{ii}	0.97	2.74	3.691 (3)	168.
C14—H14B···O1^{iii}	0.97	2.64	3.401 (3)	136.
C15—H15A···O1^{iii}	0.97	2.99	3.402 (3)	107.
C9—H9A···O2^{iv}	0.97	2.57	3.406 (3)	144.
C15—H15B···O3^{v}	0.97	2.47	3.317 (2)	146.

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) −*x*, −*y*+2, −*z*; (iii) *x*−1, *y*, *z*; (iv) −*x*+1, −*y*+2, −*z*+1; (v) −*x*, −*y*+1, −*z*+1.

Fig. 1


N2
C4
S2
C3
S4
C15
C14
O3
C13
C12
S6
C16
O2
C2
C5
C6
C1
C11
N1
S1
S3
C7
C9
C10
O1
C8
S5