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4,6,7,9,10,12,13,15,16,18-Decahydro-1,3-dithiolo[4,5-*l*][1,4,7,10,15]-trioxadithiacycloheptadecine-2-thione

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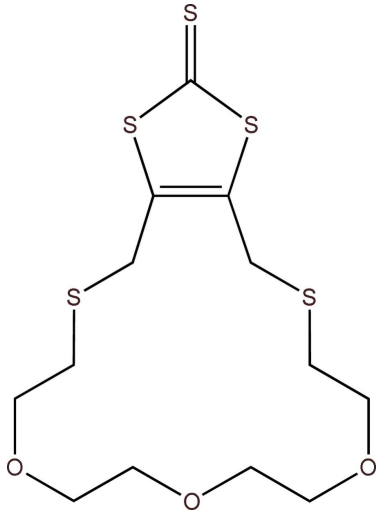
Key indicators: single-crystal X-ray study; T = 290 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.039; wR factor = 0.103; data-to-parameter ratio = 17.7.

The title compound, $C_{13}H_{20}O_3S_5$, is bisected by a crystallographic twofold rotation axis, which relates the two halves of the molecule to one another: one S, one C and one O atom lie on the axis. The thione S atom lies in the plane of the five-membered rings with an r.m.s. deviation of 0.0042 (5) Å. Parts of the 17-membered macrocycle were refined using a two-part disorder model [occupancies of 0.553 (14) and 0.447 (14)]. There are no noteworthy intermolecular interactions.

Related literature

Thiacrown ether annulated 1,3-dithiol-2-thione is a key intermediate of the crown ether-bearing redox-active tetrathiafulvalene moiety, see: Moore *et al.* (2000). For details of the synthesis, see: Chen *et al.* (2005). For a related structure, see: Hou *et al.* (2009)



Experimental

Crystal data

$C_{13}H_{20}O_3S_5$
M_r = 384.59
Monoclinic, $C2/c$
a = 14.040 (3) Å
b = 13.616 (3) Å
c = 10.004 (2) Å
β = 110.89 (3)°
V = 1786.6 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.65 mm^{-1}
T = 290 K
0.13 × 0.13 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.920, T_{max} = 0.926
8683 measured reflections
2054 independent reflections
1795 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.103
S = 1.05
2054 reflections
116 parameters
31 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 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: NK2030).

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supplementary materials

Acta Cryst. (2010). E**66**, o1335 [**doi:10.1107/S1600536810016946**]

4,6,7,9,10,12,13,15,16,18-Decahydro-1,3-dithiolo[4,5-*l*][1,4,7,10,15]trioxadithiacycloheptadecine-2-thione

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

Comment

Thiacrown ether annulated 1,3-dithiol-2-thione has been intensively investigated because it is a key intermediate of the crown ether bearing redox-active tetrathiafulvalene moiety (Moore *et al.*, 2000). Herein, we report the crystal structure of the title compound, (I).

The molecule structure of title compound, $C_{13}H_{20}O_3S_5$, is shown in Fig. 1. All bond lengths and angles are unexceptional and comparable with the related structure (Hou *et al.*, 2009). The C6, C7 and C6', C7' atoms were refined using a two-part disorder model with a major:minor occupancy ratio of 55:45.

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 evaperation a mixture of dichloromethane and petroleum (60-90 °C) at room temperature.

Refinement

C-bound H-atoms were placed in calculated positions (C—H 0.97 Å) and were included in the refinement with U_{iso}(H) = 1.2 U_{eq}(C). Atoms C6, C7 and C6', C7' were refined using a two-part disorder model with a major:minor occupancy ratio of 55:45. Mild rigid bond restraints were used on the disordered components.

Figures

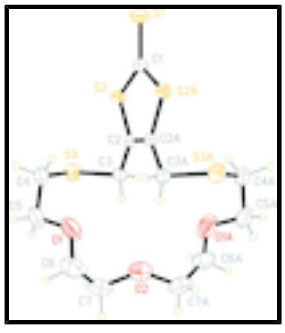


Fig. 1. The asymmetric of title compound, with the atom numbering. The disordered C6', C7' and their attached H atoms are omitted for clarity. Displacement ellipsoids of non-H atoms are drawn at the 30% probalility level.

4,6,7,9,10,12,13,15,16,18-Decahydro-1,3-dithiolo[4,5-*l*][1,4,7,10,15]trioxadithiacycloheptadecine-2-thione

Crystal data

$C_{13}H_{20}O_3S_5$ $F(000) = 808$

M_r = 384.59	D_x = 1.430 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 7120 reflections
a = 14.040 (3) Å	θ = 3.0–27.5°
b = 13.616 (3) Å	μ = 0.65 mm^{-1}
c = 10.004 (2) Å	*T* = 290 K
β = 110.89 (3)°	Block, yellow
V = 1786.6 (6) Å^3	0.13 × 0.13 × 0.12 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID diffractometer	2054 independent reflections
Radiation source: fine-focus sealed tube	1795 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.022
ω scans	θ_{max} = 27.5°, θ_{min} = 3.0°
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	*h* = −18→17
T_{min} = 0.920, T_{max} = 0.926	*k* = −15→17
8683 measured reflections	*l* = −12→12

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.039	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.103	H-atom parameters constrained
S = 1.05	$w = 1/[\sigma^2(F_o^2) + (0.0458P)^2 + 1.730P]$ where $P = (F_o^2 + 2F_c^2)/3$
2054 reflections	$(\Delta/\sigma)_{max} < 0.001$
116 parameters	$\Delta\rho_{max}$ = 0.38 e Å^{-3}
31 restraints	$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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}	Occ. (<1)
S1	0.0000	−0.12281 (6)	0.2500	0.0692 (3)	
S2	0.05129 (4)	0.06857 (4)	0.14909 (6)	0.04829 (17)	
S3	0.17462 (5)	0.26384 (5)	0.11969 (8)	0.0693 (2)	
O1	0.17749 (17)	0.40044 (19)	0.3861 (3)	0.1067 (8)	
O2	0.0000	0.5091 (3)	0.2500	0.1381 (16)	
C1	0.0000	−0.0016 (2)	0.2500	0.0456 (6)	
C2	0.02260 (13)	0.18371 (13)	0.2012 (2)	0.0390 (4)	
C3	0.04873 (15)	0.27043 (15)	0.1292 (2)	0.0493 (5)	
H3A	0.0440	0.3295	0.1807	0.059*	
H3B	−0.0010	0.2757	0.0330	0.059*	
C4	0.25511 (19)	0.2730 (2)	0.3037 (4)	0.0816 (9)	
H4A	0.2278	0.2308	0.3594	0.098*	
H4B	0.3221	0.2481	0.3138	0.098*	
C5	0.2672 (2)	0.3743 (2)	0.3658 (4)	0.0896 (10)	
H5A	0.3238	0.3758	0.4564	0.108*	
H5B	0.2813	0.4205	0.3013	0.108*	
C6	0.1737 (6)	0.4901 (6)	0.4396 (11)	0.092 (2)	0.553 (14)
H6A	0.2409	0.5199	0.4753	0.111*	0.553 (14)
H6B	0.1473	0.4868	0.5169	0.111*	0.553 (14)
C7	0.1044 (10)	0.5461 (6)	0.3178 (12)	0.099 (3)	0.553 (14)
H7A	0.1004	0.6126	0.3501	0.118*	0.553 (14)
H7B	0.1351	0.5497	0.2451	0.118*	0.553 (14)
C6'	0.1649 (8)	0.5194 (7)	0.3562 (16)	0.083 (3)	0.447 (14)
H6'1	0.2130	0.5549	0.4360	0.100*	0.447 (14)
H6'2	0.1785	0.5356	0.2703	0.100*	0.447 (14)
C7'	0.0574 (11)	0.5476 (8)	0.3385 (11)	0.092 (3)	0.447 (14)
H7'1	0.0459	0.5333	0.4266	0.110*	0.447 (14)
H7'2	0.0491	0.6178	0.3219	0.110*	0.447 (14)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0894 (6)	0.0347 (4)	0.0982 (7)	0.000	0.0513 (5)	0.000
S2	0.0524 (3)	0.0401 (3)	0.0612 (3)	0.00140 (19)	0.0310 (2)	−0.0020 (2)
S3	0.0700 (4)	0.0671 (4)	0.0918 (5)	−0.0035 (3)	0.0543 (4)	0.0127 (3)
O1	0.0793 (14)	0.1025 (17)	0.142 (2)	−0.0370 (13)	0.0438 (14)	−0.0511 (16)
O2	0.110 (3)	0.067 (2)	0.225 (5)	0.000	0.044 (3)	0.000
C1	0.0432 (13)	0.0374 (13)	0.0576 (15)	0.000	0.0199 (12)	0.000
C2	0.0321 (8)	0.0354 (9)	0.0478 (9)	−0.0004 (6)	0.0123 (7)	0.0006 (7)
C3	0.0459 (10)	0.0444 (10)	0.0561 (11)	−0.0011 (8)	0.0165 (9)	0.0090 (9)
C4	0.0423 (12)	0.0699 (17)	0.122 (2)	−0.0026 (11)	0.0168 (14)	0.0213 (16)
C5	0.0518 (15)	0.086 (2)	0.115 (2)	−0.0222 (13)	0.0109 (15)	0.0036 (18)
C6	0.109 (5)	0.077 (4)	0.080 (5)	−0.023 (3)	0.021 (3)	−0.014 (3)
C7	0.112 (5)	0.054 (3)	0.126 (5)	−0.018 (4)	0.038 (5)	−0.010 (3)

C6'	0.092 (5)	0.063 (5)	0.100 (7)	−0.029 (4)	0.043 (5)	−0.029 (5)
C7'	0.108 (7)	0.081 (5)	0.094 (5)	−0.003 (5)	0.045 (5)	−0.037 (4)

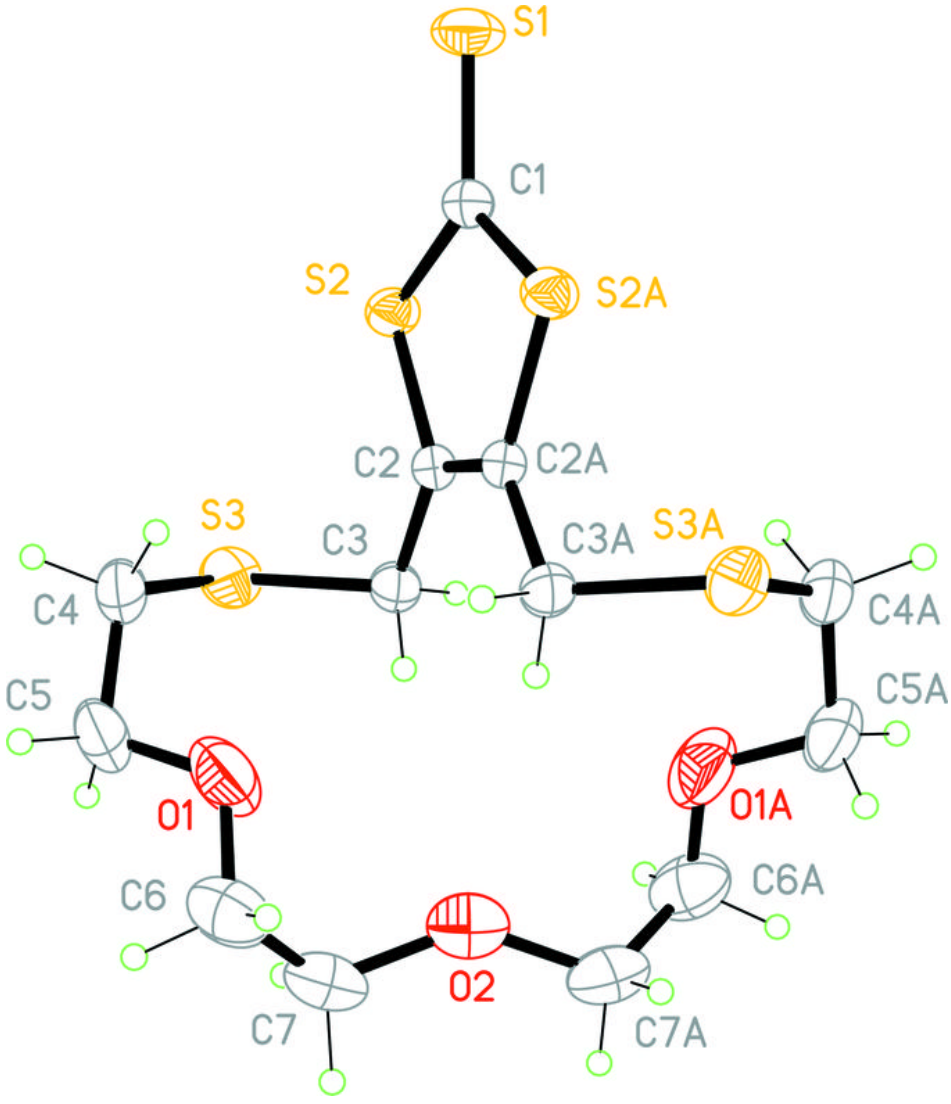
Geometric parameters (Å, °)

S1—C1	1.650 (3)	C3—H3B	0.9700
S2—C1	1.7231 (16)	C4—C5	1.497 (4)
S2—C2	1.7440 (18)	C4—H4A	0.9700
S3—C4	1.788 (3)	C4—H4B	0.9700
S3—C3	1.806 (2)	C5—H5A	0.9700
O1—C6	1.342 (7)	C5—H5B	0.9700
O1—C5	1.391 (4)	C6—C7	1.472 (14)
O1—C6'	1.645 (12)	C6—H6A	0.9700
O2—C7'	1.095 (11)	C6—H6B	0.9700
O2—C7'^{i}	1.095 (11)	C7—H7A	0.9700
O2—C7	1.467 (11)	C7—H7B	0.9700
O2—C7^{i}	1.467 (11)	C6'—C7'	1.506 (15)
C1—S2^{i}	1.7231 (16)	C6'—H6'1	0.9700
C2—C2^{i}	1.340 (4)	C6'—H6'2	0.9700
C2—C3	1.495 (3)	C7'—H7'1	0.9700
C3—H3A	0.9700	C7'—H7'2	0.9700
C1—S2—C2	97.69 (10)	O1—C5—H5B	109.9
C4—S3—C3	102.32 (13)	C4—C5—H5B	109.9
C6—O1—C5	117.2 (4)	H5A—C5—H5B	108.3
C6—O1—C6'	32.8 (4)	O1—C6—C7	104.4 (8)
C5—O1—C6'	105.6 (4)	O1—C6—H6A	110.9
C7'—O2—C7'^{i}	122.7 (13)	C7—C6—H6A	110.9
C7'—O2—C7	30.3 (5)	O1—C6—H6B	110.9
C7'^{i}—O2—C7	122.3 (7)	C7—C6—H6B	110.9
C7'—O2—C7^{i}	122.3 (7)	H6A—C6—H6B	108.9
C7'^{i}—O2—C7^{i}	30.3 (5)	O2—C7—C6	117.5 (8)
C7—O2—C7^{i}	139.8 (8)	O2—C7—H7A	107.9
S1—C1—S2^{i}	123.68 (8)	C6—C7—H7A	107.9
S1—C1—S2	123.68 (8)	O2—C7—H7B	107.9
S2^{i}—C1—S2	112.65 (15)	C6—C7—H7B	107.9
C2^{i}—C2—C3	127.72 (11)	H7A—C7—H7B	107.2
C2^{i}—C2—S2	115.95 (6)	C7'—C6'—O1	108.1 (8)
C3—C2—S2	116.30 (14)	C7'—C6'—H6'1	110.1
C2—C3—S3	113.52 (14)	O1—C6'—H6'1	110.1
C2—C3—H3A	108.9	C7'—C6'—H6'2	110.1
S3—C3—H3A	108.9	O1—C6'—H6'2	110.1
C2—C3—H3B	108.9	H6'1—C6'—H6'2	108.4
S3—C3—H3B	108.9	O2—C7'—C6'	113.0 (9)
H3A—C3—H3B	107.7	O2—C7'—C7'^{i}	28.7 (6)
C5—C4—S3	115.3 (2)	C6'—C7'—C7'^{i}	125.7 (13)
C5—C4—H4A	108.4	O2—C7'—H7'1	109.0

S3—C4—H4A	108.4	C6'—C7'—H7'1	109.0
C5—C4—H4B	108.4	C7^{i}—C7'—H7'1	118.7
S3—C4—H4B	108.4	O2—C7'—H7'2	109.0
H4A—C4—H4B	107.5	C6'—C7'—H7'2	109.0
O1—C5—C4	108.8 (2)	C7^{i}—C7'—H7'2	80.4
O1—C5—H5A	109.9	H7'1—C7'—H7'2	107.8
C4—C5—H5A	109.9		

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

Fig. 1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