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4,6,7,9,10,12,13,15-Octahydro-2*H*-1,3-dithiolo[4,5-*i*][1,4,7,12]dioxadithiacyclotetradecine-2-thione

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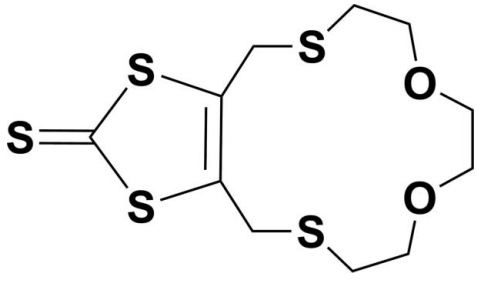
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.004 Å; R factor = 0.034; wR factor = 0.088; data-to-parameter ratio = 19.6.

In the title molecule, $C_{11}H_{16}O_2S_5$, the two S atoms from the macrocycle are situated on opposite sides of the mean plane of the five-membered ring, deviating from it by 1.288 (3) and 1.728 (3) Å. In the crystal, weak intermolecular C—H···S and C—H···O hydrogen bonds link the molecules into layers parallel to (100). The crystal studied was a racemic twin.

Related literature

For crown ether annulated 1,3-dithiol-2-thiones, see: Hansen *et al.* (1992); Trippé *et al.* (2002). For details of the synthesis, see: Chen *et al.* (2005). For a related structure, see: Hou *et al.* (2009)



Experimental

Crystal data

$C_{11}H_{16}O_2S_5$
M_r = 340.54
Monoclinic, $P2_1$
a = 8.9201 (18) Å
b = 8.5317 (17) Å
c = 10.128 (2) Å
β = 97.00 (3)°
V = 765.0 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.75 mm^{-1}
T = 291 K
0.13 × 0.12 × 0.11 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.909, T_{max} = 0.922
7527 measured reflections
3221 independent reflections
3100 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.088
S = 1.06
3221 reflections
164 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Absolute structure: Flack (1983); 1359 Friedel pairs
Flack parameter: 0.42 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7A···S1^{i}	0.97	2.86	3.695 (3)	145
C10—H10A···O2^{ii}	0.97	2.51	3.317 (3)	140

Symmetry codes: (i) $x-1, y, z-1$; (ii) $-x+1, y+\frac{1}{2}, -z+1$.

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2592).

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supplementary materials

Acta Cryst. (2009). E**65**, o2042 [**doi:10.1107/S1600536809029468**]

4,6,7,9,10,12,13,15-Octahydro-2*H*-1,3-dithiolo[4,5-*i*][1,4,7,12]dioxadithiacyclotetradecine-2-thione

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Comment

In the context of redox-responsive ligands, TTF is an ideal redox-active unit in view of its unique π-electron donating properties. Attachment of ligands such as crown ethers to TTF in many cases results in the electrochemical tunable ligands (Trippé *et al.*, 2002). Crowned 1,3-dithiole-2-thiones, important precursors to TTF derivatives, have also attracted attention (Hansen *et al.*, 1992). In this paper, we report the crystal structure of the title compound.

In the title compound (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 intermolecular C—H···S and C—H···O hydrogen bonds (Table 1) link the molecules into layers parallel to (*a*+*c*)*b* plane.

Experimental

The title compound was prepared according to the literature (Chen *et al.*, 2005) and single crystals suitable for X-ray diffraction were prepared by slow evaporation a mixture of dichloromthane and petroleum (60–90 °C) at room temperatue.

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). The refined value of Flack parameter of 0.42 (9) suggests that the crystal studied was a racemic twin.

Figures

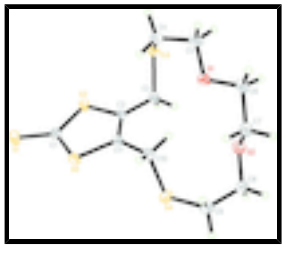

Fig. 1. The molecular structure of the title compound showing the atomic numbering. Displacement ellipsoids of non-H atoms are drawn at the 30% probalility level.

4,6,7,9,10,12,13,15-Octahydro-2*H*-1,3- dithiolo[4,5-*i*][1,4,7,12]dioxadithiacyclotetradecine-2-thione

Crystal data

$C_{11}H_{16}O_2S_5$	F_{000} = 356
M_r = 340.54	D_x = 1.478 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, λ = 0.71073 Å

Hall symbol: P 2yb	Cell parameters from 7211 reflections
a = 8.9201 (18) Å	θ = 3.1–27.5º
b = 8.5317 (17) Å	μ = 0.75 mm^{-1}
c = 10.128 (2) Å	T = 291 K
β = 97.00 (3)º	Block, yellow
V = 765.0 (3) Å^3	0.13 × 0.12 × 0.11 mm
Z = 2	

Data collection

Rigaku R-AXIS RAPID diffractometer	3221 independent reflections
Radiation source: fine-focus sealed tube	3100 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.028
T = 291 K	θ_{max} = 27.5º
ω scans	θ_{min} = 3.1º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	h = −11→11
T_{min} = 0.909, T_{max} = 0.922	k = −11→10
7527 measured reflections	l = −13→11

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.034	$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.1939P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.088	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.06	$\Delta\rho_{max}$ = 0.58 e Å^{-3}
3221 reflections	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
164 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983); 1359 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.42 (9)
Secondary atom site location: difference Fourier map	

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	0.9104 (3)	0.5403 (4)	1.0514 (2)	0.0344 (5)
C2	0.6930 (2)	0.4126 (3)	0.8914 (2)	0.0264 (5)
C3	0.5748 (3)	0.2952 (3)	0.8447 (2)	0.0342 (5)
H3A	0.6206	0.1920	0.8492	0.041*
H3B	0.5391	0.3164	0.7521	0.041*
C4	0.3178 (3)	0.4733 (4)	0.8901 (3)	0.0406 (6)
H4A	0.2524	0.4990	0.9568	0.049*
H4B	0.3929	0.5556	0.8915	0.049*
C5	0.2242 (3)	0.4741 (4)	0.7555 (3)	0.0445 (7)
H5A	0.1638	0.3795	0.7439	0.053*
H5B	0.1566	0.5637	0.7482	0.053*
C6	0.2480 (3)	0.4848 (4)	0.5262 (3)	0.0426 (6)
H6A	0.1704	0.5650	0.5179	0.051*
H6B	0.2005	0.3842	0.5047	0.051*
C7	0.3637 (3)	0.5190 (4)	0.4329 (3)	0.0418 (6)
H7A	0.3150	0.5253	0.3421	0.050*
H7B	0.4128	0.6185	0.4558	0.050*
C8	0.5855 (3)	0.4162 (4)	0.3590 (3)	0.0427 (7)
H8A	0.5382	0.4495	0.2722	0.051*
H8B	0.6331	0.3156	0.3480	0.051*
C9	0.7060 (3)	0.5337 (4)	0.4090 (3)	0.0470 (7)
H9A	0.6589	0.6351	0.4175	0.056*
H9B	0.7765	0.5438	0.3438	0.056*
C10	0.6889 (3)	0.5696 (4)	0.6801 (2)	0.0381 (6)
H10A	0.6885	0.6826	0.6690	0.046*
H10B	0.5861	0.5322	0.6586	0.046*
C11	0.7448 (2)	0.5293 (3)	0.8207 (2)	0.0290 (5)
O1	0.3233 (2)	0.4825 (3)	0.65686 (18)	0.0432 (5)
O2	0.4715 (2)	0.3969 (2)	0.44464 (17)	0.0385 (4)
S1	1.03598 (9)	0.58767 (12)	1.17933 (8)	0.0558 (2)
S2	0.78149 (7)	0.39272 (8)	1.05411 (6)	0.03282 (15)
S3	0.41352 (8)	0.29178 (9)	0.93755 (8)	0.04456 (18)
S4	0.80970 (8)	0.48036 (12)	0.56811 (7)	0.0539 (2)
S5	0.89230 (7)	0.64009 (8)	0.90156 (7)	0.03733 (16)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0240 (11)	0.0456 (15)	0.0325 (11)	0.0034 (10)	−0.0003 (9)	−0.0064 (11)
C2	0.0195 (10)	0.0336 (13)	0.0252 (10)	0.0033 (8)	−0.0005 (8)	−0.0022 (9)
C3	0.0310 (12)	0.0346 (13)	0.0356 (12)	0.0012 (10)	−0.0020 (9)	−0.0022 (11)
C4	0.0330 (13)	0.0503 (17)	0.0400 (13)	0.0006 (12)	0.0105 (10)	−0.0063 (12)
C5	0.0259 (12)	0.0616 (19)	0.0473 (15)	0.0072 (12)	0.0094 (11)	0.0041 (14)
C6	0.0275 (13)	0.0597 (19)	0.0388 (13)	0.0049 (12)	−0.0030 (10)	0.0054 (13)

C7	0.0369 (14)	0.0520 (17)	0.0346 (12)	0.0011 (12)	−0.0028 (11)	0.0071 (12)
C8	0.0417 (15)	0.0582 (19)	0.0283 (11)	−0.0032 (13)	0.0048 (10)	0.0000 (12)
C9	0.0413 (15)	0.069 (2)	0.0313 (12)	−0.0104 (14)	0.0065 (11)	0.0044 (13)
C10	0.0318 (13)	0.0507 (16)	0.0315 (12)	0.0060 (11)	0.0028 (10)	0.0054 (11)
C11	0.0184 (10)	0.0390 (13)	0.0292 (11)	0.0012 (9)	0.0020 (8)	−0.0002 (10)
O1	0.0233 (8)	0.0716 (14)	0.0349 (9)	0.0071 (9)	0.0045 (7)	0.0047 (9)
O2	0.0346 (10)	0.0433 (11)	0.0377 (9)	−0.0016 (8)	0.0049 (7)	0.0018 (9)
S1	0.0336 (4)	0.0887 (7)	0.0416 (4)	−0.0110 (4)	−0.0092 (3)	−0.0101 (4)
S2	0.0298 (3)	0.0407 (3)	0.0267 (3)	−0.0002 (2)	−0.0015 (2)	0.0031 (2)
S3	0.0319 (3)	0.0508 (4)	0.0507 (4)	−0.0110 (3)	0.0041 (3)	0.0134 (3)
S4	0.0314 (3)	0.0975 (7)	0.0325 (3)	0.0086 (4)	0.0027 (3)	−0.0032 (4)
S5	0.0254 (3)	0.0466 (4)	0.0399 (3)	−0.0089 (3)	0.0031 (2)	0.0025 (3)

Geometric parameters (Å, °)

C1—S1	1.656 (3)	C6—H6A	0.9700
C1—S2	1.708 (3)	C6—H6B	0.9700
C1—S5	1.730 (3)	C7—O2	1.413 (4)
C2—C11	1.341 (4)	C7—H7A	0.9700
C2—C3	1.489 (3)	C7—H7B	0.9700
C2—S2	1.747 (2)	C8—O2	1.424 (3)
C3—S3	1.812 (3)	C8—C9	1.511 (4)
C3—H3A	0.9700	C8—H8A	0.9700
C3—H3B	0.9700	C8—H8B	0.9700
C4—C5	1.510 (4)	C9—S4	1.815 (3)
C4—S3	1.805 (3)	C9—H9A	0.9700
C4—H4A	0.9700	C9—H9B	0.9700
C4—H4B	0.9700	C10—C11	1.490 (3)
C5—O1	1.414 (3)	C10—S4	1.824 (3)
C5—H5A	0.9700	C10—H10A	0.9700
C5—H5B	0.9700	C10—H10B	0.9700
C6—O1	1.409 (3)	C11—S5	1.741 (2)
C6—C7	1.510 (4)		
S1—C1—S2	124.03 (17)	C6—C7—H7A	110.0
S1—C1—S5	123.24 (18)	O2—C7—H7B	110.0
S2—C1—S5	112.70 (14)	C6—C7—H7B	110.0
C11—C2—C3	127.4 (2)	H7A—C7—H7B	108.4
C11—C2—S2	115.48 (18)	O2—C8—C9	113.8 (2)
C3—C2—S2	117.08 (18)	O2—C8—H8A	108.8
C2—C3—S3	114.98 (18)	C9—C8—H8A	108.8
C2—C3—H3A	108.5	O2—C8—H8B	108.8
S3—C3—H3A	108.5	C9—C8—H8B	108.8
C2—C3—H3B	108.5	H8A—C8—H8B	107.7
S3—C3—H3B	108.5	C8—C9—S4	113.3 (2)
H3A—C3—H3B	107.5	C8—C9—H9A	108.9
C5—C4—S3	115.9 (2)	S4—C9—H9A	108.9
C5—C4—H4A	108.3	C8—C9—H9B	108.9
S3—C4—H4A	108.3	S4—C9—H9B	108.9
C5—C4—H4B	108.3	H9A—C9—H9B	107.7

S3—C4—H4B	108.3	C11—C10—S4	110.08 (18)
H4A—C4—H4B	107.4	C11—C10—H10A	109.6
O1—C5—C4	108.3 (2)	S4—C10—H10A	109.6
O1—C5—H5A	110.0	C11—C10—H10B	109.6
C4—C5—H5A	110.0	S4—C10—H10B	109.6
O1—C5—H5B	110.0	H10A—C10—H10B	108.2
C4—C5—H5B	110.0	C2—C11—C10	125.7 (2)
H5A—C5—H5B	108.4	C2—C11—S5	116.22 (18)
O1—C6—C7	107.8 (2)	C10—C11—S5	118.0 (2)
O1—C6—H6A	110.1	C6—O1—C5	113.3 (2)
C7—C6—H6A	110.1	C7—O2—C8	113.1 (2)
O1—C6—H6B	110.1	C1—S2—C2	98.09 (12)
C7—C6—H6B	110.1	C4—S3—C3	103.16 (13)
H6A—C6—H6B	108.5	C9—S4—C10	99.88 (14)
O2—C7—C6	108.3 (2)	C1—S5—C11	97.47 (13)
O2—C7—H7A	110.0		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7A···S1^{i}	0.97	2.86	3.695 (3)	145
C10—H10A···O2^{ii}	0.97	2.51	3.317 (3)	140

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

Fig. 1