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4,6,7,9,10,12-Hexahydro-1,3-dithiolo-[4,5-*f*][1,4,9]oxadithiacycloundecine-2-thione

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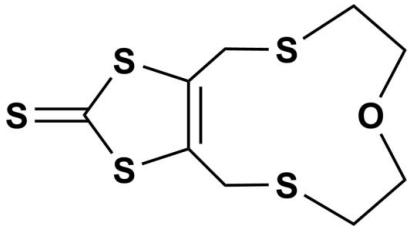
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.106; data-to-parameter ratio = 20.7.

In the title molecule, $C_9H_{12}S_5O$, the five-membered ring and attached S atom are essentially coplanar [mean deviation from the mean plane = 0.020 (1) Å]. The two S atoms belonging to the macrocycle deviate from this plane by 1.005 (1) and 1.337 (2) Å. In the crystal, π–π interactions link the molecules into centrosymmetric dimers with a short distance of 3.753 (5) Å between the centroids of the five-membered rings.

Related literature

The title compound was prepared according to Chen *et al.* (2005). For background literature concerning crown-ether-annulated 1,3-dithiol-2-thione derivatives, see: Hansen *et al.* (1992); Trippé *et al.* (2002).



Experimental

Crystal data

$C_9H_{12}OS_5$
M_r = 296.49
Triclinic, $P\overline{1}$
a = 8.3425 (17) Å
b = 8.9611 (18) Å
c = 9.820 (2) Å
α = 98.10 (3)°
β = 106.58 (3)°
γ = 112.74 (3)°
V = 622.2 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.90 mm^{-1}
T = 291 K
0.15 × 0.12 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.877, T_{max} = 0.900
6141 measured reflections
2813 independent reflections
2560 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.106
S = 1.06
2813 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 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: CV2573).

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supplementary materials

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4,6,7,9,10,12-Hexahydro-1,3-dithiolo[4,5-*f*][1,4,9]oxadithiacycloundecine-2-thione

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Comment

Crown ether annulated 1,3-dithiol-2-thione derivatives have been intensively investigated as key intermediate of the crowned tetrathiafulvalenes because the latter molecules show electrochemical signaling for various metal cations (Hansen *et al.*,1992; Trippé *et al.*, 2002). We report hererin the crystal structure of the title compound, (I).

In (I) (Fig. 1), five-membered ring and attached S2 atom are essentially coplanar with the mean deviation of 0.020 (1) Å from the mean plane *P*. The plane defined by the rest non-hydrogen atoms forms an angle of 70.25 (4) ° with *P*. The π-π interactions with the short distance of 3.753 (5) Å between the centroids of five-membered rings link the molecules into centrosymmetric dimers.

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 a mixture of dichloromethane and petroleum 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).

Figures

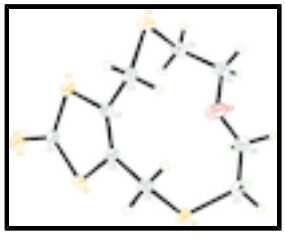

Fig. 1. The molecular structure of (I) showing the atom numbering. Displacement ellipsoids of non-H atoms are drawn at the 30% probalility level.

4,6,7,9,10,12-Hexahydro-1,3- dithiolo[4,5-*f*][1,4,9]oxadithiacycloundecine-2-thione

Crystal data

$C_9H_{12}OS_5$	$Z = 2$
$M_r = 296.49$	$F_{000} = 308$
Triclinic, $P\overline{1}$	$D_x = 1.583$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

a = 8.3425 (17) Å	Cell parameters from 5678 reflections
b = 8.9611 (18) Å	θ = 3.4–27.0º
c = 9.820 (2) Å	μ = 0.90 mm^{-1}
α = 98.10 (3)º	T = 291 K
β = 106.58 (3)º	Block, yellow
γ = 112.74 (3)º	0.15 × 0.12 × 0.12 mm
V = 622.2 (2) Å^3	

Data collection

Rigaku R-AXIS RAPID diffractometer	2813 independent reflections
Radiation source: fine-focus sealed tube	2560 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.017
T = 291 K	θ_{max} = 27.5º
ω scans	θ_{min} = 3.4º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	h = −10→10
T_{min} = 0.877, T_{max} = 0.900	k = −11→11
6141 measured reflections	l = −12→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.031	H-atom parameters constrained
$wR(F^2)$ = 0.106	$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2 + 0.2783P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max}$ = 0.003
2813 reflections	$\Delta\rho_{max}$ = 0.41 e Å^{-3}
136 parameters	$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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	0.3295 (3)	0.6916 (3)	0.4927 (2)	0.0376 (4)
C2	0.2130 (3)	0.8764 (2)	0.3463 (2)	0.0300 (4)
C3	0.1113 (3)	0.9792 (2)	0.3073 (2)	0.0354 (4)
H3A	0.1194	1.0051	0.2161	0.042*
H3B	0.1724	1.0851	0.3848	0.042*
C4	−0.2203 (3)	0.6799 (3)	0.1413 (3)	0.0408 (5)
H4A	−0.1445	0.6233	0.1746	0.049*
H4B	−0.3469	0.6075	0.1315	0.049*
C5	−0.2231 (3)	0.6956 (3)	−0.0092 (3)	0.0486 (5)
H5A	−0.2975	0.5852	−0.0811	0.058*
H5B	−0.2805	0.7677	−0.0378	0.058*
C6	−0.0193 (3)	0.6766 (3)	−0.1285 (3)	0.0466 (5)
H6A	−0.1108	0.6664	−0.2208	0.056*
H6B	−0.0423	0.5639	−0.1211	0.056*
C7	0.1742 (3)	0.7686 (3)	−0.1276 (2)	0.0439 (5)
H7A	0.1991	0.8846	−0.1229	0.053*
H7B	0.1754	0.7190	−0.2215	0.053*
C8	0.4004 (3)	0.9279 (2)	0.1782 (2)	0.0367 (4)
H8A	0.5328	1.0056	0.2243	0.044*
H8B	0.3342	0.9920	0.1443	0.044*
C9	0.3363 (2)	0.8552 (2)	0.2922 (2)	0.0294 (4)
O1	−0.0377 (2)	0.7650 (2)	−0.0088 (2)	0.0600 (5)
S1	0.43959 (7)	0.73453 (6)	0.36855 (5)	0.03533 (15)
S2	0.36763 (14)	0.58331 (12)	0.60929 (8)	0.0708 (3)
S3	0.17163 (8)	0.77394 (7)	0.48048 (6)	0.03867 (15)
S4	−0.13328 (7)	0.87314 (7)	0.28369 (6)	0.04162 (16)
S5	0.36387 (8)	0.77082 (7)	0.01819 (6)	0.04019 (15)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0448 (11)	0.0454 (11)	0.0277 (9)	0.0247 (9)	0.0138 (8)	0.0110 (8)
C2	0.0293 (8)	0.0333 (9)	0.0239 (8)	0.0121 (7)	0.0089 (7)	0.0061 (7)
C3	0.0375 (10)	0.0361 (9)	0.0347 (10)	0.0190 (8)	0.0136 (8)	0.0087 (8)
C4	0.0313 (9)	0.0420 (10)	0.0486 (12)	0.0163 (8)	0.0140 (9)	0.0140 (9)
C5	0.0318 (10)	0.0615 (14)	0.0431 (12)	0.0164 (10)	0.0121 (9)	0.0057 (10)
C6	0.0439 (11)	0.0526 (12)	0.0361 (11)	0.0199 (10)	0.0127 (9)	0.0023 (9)
C7	0.0506 (12)	0.0565 (13)	0.0329 (10)	0.0266 (10)	0.0214 (9)	0.0172 (9)
C8	0.0388 (10)	0.0339 (9)	0.0361 (10)	0.0107 (8)	0.0200 (8)	0.0097 (8)
C9	0.0283 (8)	0.0289 (8)	0.0276 (9)	0.0101 (7)	0.0105 (7)	0.0059 (7)
O1	0.0356 (8)	0.0692 (11)	0.0481 (10)	0.0048 (8)	0.0198 (7)	−0.0146 (8)
S1	0.0340 (3)	0.0422 (3)	0.0348 (3)	0.0204 (2)	0.0152 (2)	0.0112 (2)
S2	0.1147 (7)	0.0990 (6)	0.0578 (4)	0.0820 (6)	0.0517 (4)	0.0521 (4)
S3	0.0443 (3)	0.0543 (3)	0.0322 (3)	0.0285 (2)	0.0224 (2)	0.0188 (2)

S4	0.0399 (3)	0.0544 (3)	0.0438 (3)	0.0303 (2)	0.0214 (2)	0.0135 (2)
S5	0.0468 (3)	0.0511 (3)	0.0357 (3)	0.0300 (2)	0.0217 (2)	0.0135 (2)

Geometric parameters (Å, °)

C1—S2	1.642 (2)	C5—H5A	0.9700
C1—S1	1.726 (2)	C5—H5B	0.9700
C1—S3	1.726 (2)	C6—O1	1.402 (3)
C2—C9	1.346 (3)	C6—C7	1.499 (3)
C2—C3	1.495 (3)	C6—H6A	0.9700
C2—S3	1.7471 (19)	C6—H6B	0.9700
C3—S4	1.814 (2)	C7—S5	1.796 (2)
C3—H3A	0.9700	C7—H7A	0.9700
C3—H3B	0.9700	C7—H7B	0.9700
C4—C5	1.498 (3)	C8—C9	1.497 (3)
C4—S4	1.802 (2)	C8—S5	1.820 (2)
C4—H4A	0.9700	C8—H8A	0.9700
C4—H4B	0.9700	C8—H8B	0.9700
C5—O1	1.426 (3)	C9—S1	1.747 (2)
S2—C1—S1	124.30 (13)	O1—C6—H6A	109.7
S2—C1—S3	123.21 (13)	C7—C6—H6A	109.7
S1—C1—S3	112.49 (12)	O1—C6—H6B	109.7
C9—C2—C3	127.67 (18)	C7—C6—H6B	109.7
C9—C2—S3	115.53 (15)	H6A—C6—H6B	108.2
C3—C2—S3	116.79 (14)	C6—C7—S5	117.34 (17)
C2—C3—S4	112.96 (14)	C6—C7—H7A	108.0
C2—C3—H3A	109.0	S5—C7—H7A	108.0
S4—C3—H3A	109.0	C6—C7—H7B	108.0
C2—C3—H3B	109.0	S5—C7—H7B	108.0
S4—C3—H3B	109.0	H7A—C7—H7B	107.2
H3A—C3—H3B	107.8	C9—C8—S5	114.07 (14)
C5—C4—S4	116.74 (17)	C9—C8—H8A	108.7
C5—C4—H4A	108.1	S5—C8—H8A	108.7
S4—C4—H4A	108.1	C9—C8—H8B	108.7
C5—C4—H4B	108.1	S5—C8—H8B	108.7
S4—C4—H4B	108.1	H8A—C8—H8B	107.6
H4A—C4—H4B	107.3	C2—C9—C8	127.63 (18)
O1—C5—C4	110.55 (19)	C2—C9—S1	116.14 (15)
O1—C5—H5A	109.5	C8—C9—S1	116.20 (14)
C4—C5—H5A	109.5	C6—O1—C5	113.57 (18)
O1—C5—H5B	109.5	C1—S1—C9	97.73 (10)
C4—C5—H5B	109.5	C1—S3—C2	98.01 (10)
H5A—C5—H5B	108.1	C4—S4—C3	102.59 (10)
O1—C6—C7	109.68 (19)	C7—S5—C8	103.70 (11)

Fig. 1