Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

13-[4,5-Bis(methylsulfanyl)-1,3-dithiol-2-ylidene]-6-oxa-3,9,12,14-tetrathiabicyclo[9.3.0]tetradec-1(11)-ene

Rui-Bin Hou,[a] Bao Li,[b] Tie Che,[a] Bing-Zhu Yin[a]* and Li-Xin Wu[b]

[a]Key Laboratory of Organism Functional Factors of Changbai Mountain, Yanbian University, Ministry of Education, Yanji 133002, People's Republic of China, and [b]State Key Laboratory of Supramolecular Structure and Materials, College of Chemistry, Jilin University, Changchun 130012, People's Republic of China
Correspondence e-mail: zqcong@ybu.edu.cn

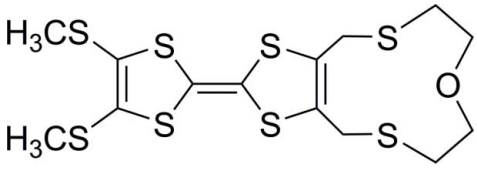
Received 14 September 2009; accepted 15 September 2009

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.006 Å; R factor = 0.056; wR factor = 0.170; data-to-parameter ratio = 21.8.

In the title molecule, $C_{14}H_{18}OS_8$, one O atom, two S atoms and six C atoms form an 11-membered ring with a chair-like conformation; the planes of the two five-membered rings connected by a carbon–carbon double bond form a dihedral angle of 29.97 (11)°. In the crystal, pairs of weak intermolecular C—H···S hydrogen bonds link two molecules into inversion dimers.

Related literature

For background to crown ether-annulated 1,3-dithiol-2-thiones, see: Hansen *et al.* (1993). For the synthesis, see: Chen *et al.* (2005). For a related structure, see: Hou *et al.* (2009)



Experimental

Crystal data

$C_{14}H_{18}OS_8$
M_r = 458.76
Triclinic, $P\overline{1}$
a = 8.4542 (17) Å
b = 10.158 (2) Å
c = 13.612 (3) Å
α = 105.00 (3)°
β = 97.83 (3)°
γ = 112.22 (3)°
V = 1008.8 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.88 mm^{-1}
T = 291 K
0.14 × 0.12 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.886, T_{max} = 0.901
9961 measured reflections
4572 independent reflections
3655 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.170
S = 1.10
4572 reflections
210 parameters
18 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.09 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7B···S2^{i}	0.97	3.00	3.793 (6)	140

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

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 Programm 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: NG2642).

References

Chen, T., Liu, W. J., Cong, Z. Q. & Yin, B. Z. (2005). *Chin. J. Org. Chem.* **25**, 570–575.
Hansen, T. K., Jorgensen, T., Jensen, F., Thygesen, P. H., Christiansen, K., Hursthouse, M. B., Harman, M. E., Malik, M. A. & Girmay, B. (1993). *J. Org. Chem.* **58**, 1359–1366.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Hou, R.-B., Li, B., Yin, B.-Z. & Wu, L.-X. (2009). *Acta Cryst.* E**65**, o1710.
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.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2009). E**65**, o2538 [doi:10.1107/S1600536809037301]

13-[4,5-Bis(methylsulfanyl)-1,3-dithiol-2-ylidene]-6-oxa-3,9,12,14-tetrathiabicyclo[9.3.0]tetradec-1(11)-ene

Rui-Bin Hou, Bao Li, Tie Che, Bing-Zhu Yin and Li-Xin Wu

S1. Comment

Tetrathiafulvalene (TTF) derivatives with a fused crown ether ring have received much attention as component molecules for cation sensors (Hansen *et al.*, 1993). We are incorporated TTF with a sulfur hybrid crown ether to synthesize the title compound

The molecule structure of tiltle compound, (I), $C_{14}H_{18}S_8O$, as shown in Fig. 1, all bond lengths and angles are normal and comparable with the related structure (Hou *et al.*, 2009). In the crystal, weak intermolecular C—H···S hydrogen bonds (Table 1) link the molecules into dimer.

S2. Experimental

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

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 to 0.97 Å) and were included in the refinement in the riding model with U_{iso}(H) = 1.2 or 1.5 U_{eq}(C).

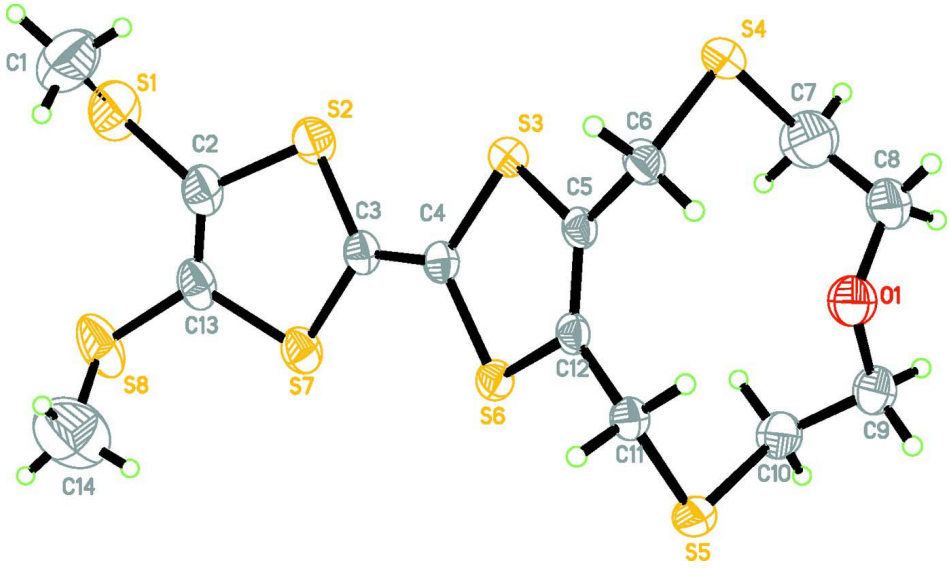


Figure 1

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

13-[4,5-bis(methylsulfanyl)-1,3-dithiol-2-ylidene]-6-oxa-3,9,12,14- tetrathiabicyclo[9.3.0]tetradec-1(11)-ene

Crystal data

$C_{14}H_{18}OS_8$
$M_r = 458.76$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.4542\ (17)$ Å
$b = 10.158\ (2)$ Å
$c = 13.612\ (3)$ Å
$\alpha = 105.00\ (3)°$
$\beta = 97.83\ (3)°$
$\gamma = 112.22\ (3)°$
$V = 1008.8\ (3)$ Å^3
$Z = 2$
$F(000) = 476$
$D_x = 1.510$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8201 reflections
$\theta = 3.1–27.5°$
$\mu = 0.88$ mm^{-1}
$T = 291$ K
Block, yellow
0.14 × 0.12 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.886$, $T_{max} = 0.901$
9961 measured reflections
4572 independent reflections
3655 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -10\rightarrow10$
$k = -11\rightarrow13$
$l = -17\rightarrow17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.170$
$S = 1.10$
4572 reflections
210 parameters
18 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0884P)^2 + 0.6622P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.016$
$\Delta\rho_{max} = 1.09$ e Å^{-3}
$\Delta\rho_{min} = -0.64$ e Å^{-3}

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	0.0932 (11)	0.6629 (10)	0.3460 (6)	0.147 (3)
H1A	0.1923	0.7484	0.3443	0.220*
H1B	0.0094	0.6954	0.3720	0.220*
H1C	0.0378	0.5904	0.2761	0.220*
C2	0.3190 (6)	0.5333 (5)	0.3702 (3)	0.0698 (11)
C3	0.4341 (5)	0.4102 (4)	0.2243 (3)	0.0507 (7)
C4	0.4512 (4)	0.3545 (3)	0.1275 (2)	0.0461 (7)
C5	0.3940 (4)	0.2608 (3)	−0.0741 (2)	0.0413 (6)
C6	0.2894 (5)	0.2050 (4)	−0.1861 (3)	0.0525 (8)
H6A	0.3691	0.2087	−0.2314	0.063*
H6B	0.2366	0.2724	−0.1949	0.063*
C7	0.2424 (8)	−0.0950 (6)	−0.2145 (6)	0.1025 (17)
H7A	0.3145	−0.0519	−0.1423	0.123*
H7B	0.1583	−0.1969	−0.2240	0.123*
C8	0.3534 (6)	−0.1067 (5)	−0.2800 (5)	0.0943 (17)
H8A	0.2905	−0.1288	−0.3517	0.113*
H8B	0.3794	−0.1915	−0.2786	0.113*
C9	0.6658 (6)	0.0075 (5)	−0.2091 (4)	0.0740 (12)
H9A	0.7526	0.0358	−0.2487	0.089*
H9B	0.6321	−0.0978	−0.2166	0.089*
C10	0.7491 (5)	0.1017 (4)	−0.0953 (3)	0.0649 (10)
H10A	0.8265	0.0635	−0.0660	0.078*
H10B	0.6563	0.0882	−0.0589	0.078*
C11	0.7040 (4)	0.3605 (4)	−0.0996 (3)	0.0469 (7)
H11A	0.7589	0.4682	−0.0874	0.056*
H11B	0.6438	0.3094	−0.1740	0.056*
C12	0.5700 (4)	0.3284 (3)	−0.0371 (2)	0.0407 (6)
C13	0.4928 (6)	0.6013 (4)	0.4070 (3)	0.0661 (10)
C14	0.7619 (13)	0.8815 (12)	0.4955 (7)	0.176 (4)

H14A	0.8239	0.8368	0.4545	0.264*
H14B	0.8442	0.9598	0.5587	0.264*
H14C	0.7053	0.9239	0.4552	0.264*
O1	0.5140 (4)	0.0229 (3)	−0.2531 (2)	0.0707 (7)
S1	0.1683 (2)	0.5780 (2)	0.43099 (13)	0.1162 (6)
S2	0.22953 (15)	0.37585 (11)	0.25246 (8)	0.0674 (3)
S3	0.26931 (11)	0.23691 (10)	0.01898 (7)	0.0513 (2)
S4	0.11564 (12)	0.01405 (12)	−0.22817 (9)	0.0704 (3)
S5	0.87487 (12)	0.30094 (10)	−0.06741 (8)	0.0602 (3)
S6	0.65644 (11)	0.38520 (10)	0.10015 (6)	0.0504 (2)
S7	0.61409 (14)	0.52278 (11)	0.33593 (7)	0.0619 (3)
S8	0.6096 (2)	0.74971 (15)	0.52682 (9)	0.1030 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.123 (4)	0.173 (5)	0.156 (5)	0.087 (4)	0.053 (4)	0.031 (4)
C2	0.087 (3)	0.059 (2)	0.060 (2)	0.022 (2)	0.044 (2)	0.0171 (18)
C3	0.062 (2)	0.0419 (16)	0.0491 (17)	0.0220 (15)	0.0185 (14)	0.0151 (13)
C4	0.0529 (18)	0.0395 (15)	0.0474 (16)	0.0223 (13)	0.0130 (13)	0.0134 (13)
C5	0.0474 (16)	0.0328 (13)	0.0428 (15)	0.0202 (12)	0.0075 (12)	0.0091 (11)
C6	0.0540 (19)	0.0458 (17)	0.0492 (17)	0.0240 (15)	−0.0016 (14)	0.0069 (14)
C7	0.090 (3)	0.068 (3)	0.143 (4)	0.032 (2)	0.033 (3)	0.029 (3)
C8	0.068 (3)	0.057 (2)	0.122 (4)	0.027 (2)	0.009 (3)	−0.017 (3)
C9	0.065 (2)	0.053 (2)	0.098 (3)	0.0339 (19)	0.016 (2)	0.005 (2)
C10	0.063 (2)	0.0484 (19)	0.089 (3)	0.0315 (18)	0.0179 (19)	0.0217 (19)
C11	0.0480 (17)	0.0389 (15)	0.0520 (17)	0.0195 (13)	0.0122 (13)	0.0119 (13)
C12	0.0467 (16)	0.0331 (13)	0.0423 (15)	0.0210 (12)	0.0087 (12)	0.0083 (11)
C13	0.089 (3)	0.0504 (19)	0.0500 (19)	0.019 (2)	0.0335 (19)	0.0126 (16)
C14	0.172 (5)	0.168 (5)	0.142 (5)	0.025 (4)	0.065 (4)	0.040 (4)
O1	0.0666 (17)	0.0519 (15)	0.090 (2)	0.0287 (13)	0.0162 (14)	0.0151 (14)
S1	0.1153 (12)	0.1136 (11)	0.1043 (11)	0.0336 (9)	0.0798 (10)	0.0109 (9)
S2	0.0694 (6)	0.0561 (5)	0.0642 (6)	0.0132 (5)	0.0322 (5)	0.0144 (4)
S3	0.0450 (4)	0.0472 (4)	0.0537 (5)	0.0173 (3)	0.0116 (3)	0.0093 (3)
S4	0.0446 (5)	0.0586 (6)	0.0766 (7)	0.0161 (4)	0.0007 (4)	−0.0092 (5)
S5	0.0433 (5)	0.0514 (5)	0.0791 (6)	0.0218 (4)	0.0133 (4)	0.0108 (4)
S6	0.0480 (4)	0.0542 (5)	0.0435 (4)	0.0262 (4)	0.0036 (3)	0.0059 (3)
S7	0.0723 (6)	0.0586 (5)	0.0457 (5)	0.0226 (5)	0.0151 (4)	0.0123 (4)
S8	0.1440 (13)	0.0685 (7)	0.0543 (6)	0.0108 (8)	0.0411 (7)	0.0005 (5)

Geometric parameters (Å, º)

C1—S1	1.794 (9)	C8—O1	1.405 (6)
C1—H1A	0.9600	C8—H8A	0.9700
C1—H1B	0.9600	C8—H8B	0.9700
C1—H1C	0.9600	C9—O1	1.421 (5)
C2—C13	1.319 (7)	C9—C10	1.495 (6)
C2—S1	1.744 (4)	C9—H9A	0.9700

C2—S2	1.768 (4)	C9—H9B	0.9700
C3—C4	1.344 (5)	C10—S5	1.798 (4)
C3—S2	1.747 (4)	C10—H10A	0.9700
C3—S7	1.756 (4)	C10—H10B	0.9700
C4—S3	1.747 (3)	C11—C12	1.498 (4)
C4—S6	1.753 (3)	C11—S5	1.809 (3)
C5—C12	1.335 (4)	C11—H11A	0.9700
C5—C6	1.496 (4)	C11—H11B	0.9700
C5—S3	1.762 (3)	C12—S6	1.763 (3)
C6—S4	1.814 (4)	C13—S8	1.752 (4)
C6—H6A	0.9700	C13—S7	1.765 (4)
C6—H6B	0.9700	C14—S8	1.665 (9)
C7—C8	1.397 (8)	C14—H14A	0.9600
C7—S4	1.833 (6)	C14—H14B	0.9600
C7—H7A	0.9700	C14—H14C	0.9600
C7—H7B	0.9700		

S1—C1—H1A	109.5	C10—C9—H9A	108.9
S1—C1—H1B	109.5	O1—C9—H9B	108.9
H1A—C1—H1B	109.5	C10—C9—H9B	108.9
S1—C1—H1C	109.5	H9A—C9—H9B	107.7
H1A—C1—H1C	109.5	C9—C10—S5	116.1 (3)
H1B—C1—H1C	109.5	C9—C10—H10A	108.3
C13—C2—S1	125.9 (3)	S5—C10—H10A	108.3
C13—C2—S2	117.2 (3)	C9—C10—H10B	108.3
S1—C2—S2	116.8 (3)	S5—C10—H10B	108.3
C4—C3—S2	123.3 (3)	H10A—C10—H10B	107.4
C4—C3—S7	123.7 (3)	C12—C11—S5	113.4 (2)
S2—C3—S7	112.98 (18)	C12—C11—H11A	108.9
C3—C4—S3	122.6 (3)	S5—C11—H11A	108.9
C3—C4—S6	123.2 (3)	C12—C11—H11B	108.9
S3—C4—S6	114.08 (18)	S5—C11—H11B	108.9
C12—C5—C6	127.3 (3)	H11A—C11—H11B	107.7
C12—C5—S3	116.9 (2)	C5—C12—C11	127.1 (3)
C6—C5—S3	115.8 (2)	C5—C12—S6	117.2 (2)
C5—C6—S4	113.5 (3)	C11—C12—S6	115.7 (2)
C5—C6—H6A	108.9	C2—C13—S8	125.2 (3)
S4—C6—H6A	108.9	C2—C13—S7	116.7 (3)
C5—C6—H6B	108.9	S8—C13—S7	117.7 (3)
S4—C6—H6B	108.9	S8—C14—H14A	109.5
H6A—C6—H6B	107.7	S8—C14—H14B	109.5
C8—C7—S4	119.7 (5)	H14A—C14—H14B	109.5
C8—C7—H7A	107.4	S8—C14—H14C	109.5
S4—C7—H7A	107.4	H14A—C14—H14C	109.5
C8—C7—H7B	107.4	H14B—C14—H14C	109.5
S4—C7—H7B	107.4	C8—O1—C9	114.7 (4)
H7A—C7—H7B	106.9	C2—S1—C1	100.8 (3)
C7—C8—O1	114.6 (4)	C3—S2—C2	94.04 (19)

C7—C8—H8A	108.6	C4—S3—C5	94.20 (15)
O1—C8—H8A	108.6	C6—S4—C7	102.0 (2)
C7—C8—H8B	108.6	C10—S5—C11	102.29 (17)
O1—C8—H8B	108.6	C4—S6—C12	93.92 (15)
H8A—C8—H8B	107.6	C3—S7—C13	94.02 (19)
O1—C9—C10	113.4 (3)	C14—S8—C13	104.6 (3)
O1—C9—H9A	108.9		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*B*···S2^{i}	0.97	3.00	3.793 (6)	140

Symmetry code: (i) −*x*, −*y*, −*z*.