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2,3-Bis(methylsulfanyl)-1,4,5,8-tetrathiafulvalene

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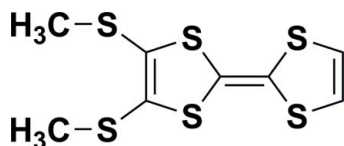
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å;
 R factor = 0.031; wR factor = 0.083; data-to-parameter ratio = 21.5.

In the title compound, $\text{C}_8\text{H}_8\text{S}_6$, the five-membered rings form a dihedral angle of $25.06(9)^\circ$. In the absence of short intermolecular contacts, the molecules are packed by van der Waals forces in the crystal.

Related literature

For applications of tetrathiafulvalenes, see: Wudl *et al.* (1972); Jørgensen *et al.* (1994). For details of the synthesis, see: Fourmigué *et al.* (1993). For a related structure, see: Hou *et al.* (2010).



Experimental

Crystal data

$\text{C}_8\text{H}_8\text{S}_6$
 $M_r = 296.50$
Monoclinic, $C2/c$
 $a = 19.368(11)$ Å

$b = 7.703(4)$ Å
 $c = 17.150(8)$ Å
 $\beta = 108.59(2)^\circ$
 $V = 2425(2)$ Å³

$Z = 8$
Mo $K\alpha$ radiation
 $\mu = 1.09$ mm⁻¹

$T = 291$ K
 $0.12 \times 0.10 \times 0.09$ mm

Data collection

Rigaku R-Axis RAPID
diffractometer
Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)
 $T_{\min} = 0.881$, $T_{\max} = 0.909$

11301 measured reflections
2773 independent reflections
2480 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.083$
 $S = 1.03$
2773 reflections

129 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.58$ e Å⁻³
 $\Delta\rho_{\min} = -0.66$ e Å⁻³

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 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: NG5069).

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supporting information

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2,3-Bis(methylsulfonyl)-1,4,5,8-tetrathiafulvalene

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S1. Comment

Tetrathiafulvalenes (TTFs) have attracted much interest due to their electron-donating ability, which have been used for the synthesis of new organic metals and superconductors (Wudl *et al.* 1972) and recently for supramolecular architectures (Jørgensen *et al.* 1994.). In this paper, we report the crystal structure of the title compound.

The title compound, as shown in Fig. 1, crystallizes in monoclinic system with the space group $C2/c$. All bond lengths and angles of the title compound are normal and comparable with those reported for the related structure (Hou *et al.*, 2010). In the crystal, the molecules are packed by van der Waal's forces.

S2. Experimental

The title compound was prepared according to literature (Fourmigué *et al.*, 1993) 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.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions with C—H = 0.93 or 0.96 Å and were included in the refinement in the riding model with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5 U_{\text{eq}}(\text{C})$.

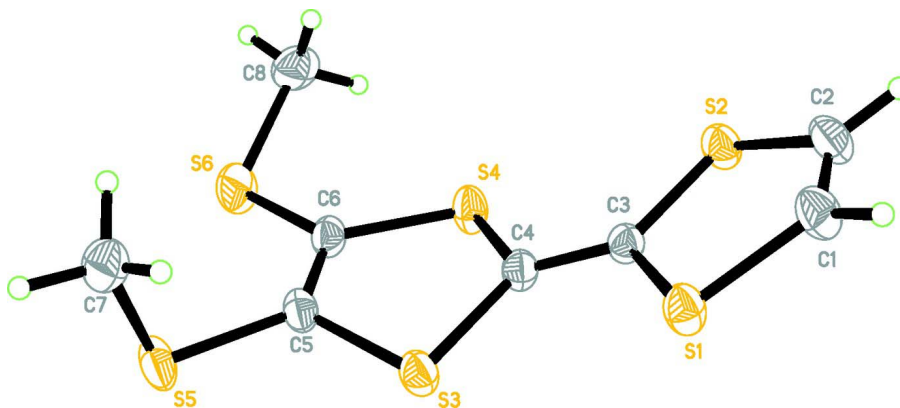


Figure 1

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

2-(2H-1,3-dithiol-2-ylidene)-4,5-bis(methylsulfanyl)-2H-1,3-dithiole

Crystal data

C₈H₈S₆ $M_r = 296.50$ Monoclinic, *C2/c*

Hall symbol: -C 2yc

 $a = 19.368 (11) \text{ \AA}$ $b = 7.703 (4) \text{ \AA}$ $c = 17.150 (8) \text{ \AA}$ $\beta = 108.59 (2)^\circ$ $V = 2425 (2) \text{ \AA}^3$ $Z = 8$ $F(000) = 1216$ $D_x = 1.624 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 9674 reflections

 $\theta = 3.3\text{--}27.6^\circ$ $\mu = 1.09 \text{ mm}^{-1}$ $T = 291 \text{ K}$

Block, yellow

 $0.12 \times 0.10 \times 0.09 \text{ mm}$

Data collection

Rigaku R-AXIS RAPID

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 ω scans

Absorption correction: multi-scan

(ABSCOR; Higashi, 1995)

 $T_{\min} = 0.881$, $T_{\max} = 0.909$

11301 measured reflections

2773 independent reflections

2480 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.025$ $\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.3^\circ$ $h = -25 \rightarrow 25$ $k = -10 \rightarrow 9$ $l = -21 \rightarrow 22$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.031$ $wR(F^2) = 0.083$ $S = 1.03$

2773 reflections

129 parameters

0 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

 $w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 2.0438P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.006$ $\Delta\rho_{\max} = 0.58 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.66 \text{ e \AA}^{-3}$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.02208 (11)	0.7309 (3)	1.15260 (11)	0.0490 (5)
H1	-0.0004	0.7911	1.1849	0.059*
C2	0.06543 (12)	0.5980 (3)	1.18273 (11)	0.0494 (5)

H2	0.0744	0.5615	1.2368	0.059*
C3	0.07086 (9)	0.6402 (2)	1.03633 (10)	0.0331 (3)
C4	0.09038 (9)	0.6398 (2)	0.96816 (10)	0.0348 (3)
C5	0.12943 (10)	0.7609 (2)	0.84888 (10)	0.0375 (4)
C6	0.17548 (9)	0.6286 (2)	0.87949 (10)	0.0381 (4)
C7	0.18062 (13)	1.0834 (3)	0.83038 (14)	0.0552 (5)
H7A	0.1538	1.1248	0.8651	0.083*
H7B	0.1850	1.1747	0.7941	0.083*
H7C	0.2283	1.0472	0.8638	0.083*
C8	0.30945 (12)	0.4634 (3)	0.92717 (15)	0.0608 (6)
H8A	0.3209	0.5384	0.9743	0.091*
H8B	0.3531	0.4385	0.9141	0.091*
H8C	0.2890	0.3571	0.9391	0.091*
S1	0.00813 (3)	0.79005 (6)	1.05124 (3)	0.04253 (13)
S2	0.10518 (3)	0.49359 (6)	1.11799 (3)	0.04402 (13)
S3	0.05498 (2)	0.78415 (6)	0.88578 (3)	0.04151 (13)
S4	0.15503 (3)	0.49566 (6)	0.95261 (3)	0.04458 (14)
S5	0.13348 (3)	0.90281 (7)	0.77079 (3)	0.05321 (15)
S6	0.24524 (3)	0.56800 (10)	0.84168 (4)	0.06234 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0601 (12)	0.0557 (12)	0.0353 (9)	0.0035 (9)	0.0211 (8)	-0.0054 (8)
C2	0.0591 (11)	0.0599 (13)	0.0293 (9)	0.0019 (10)	0.0142 (8)	0.0037 (8)
C3	0.0323 (7)	0.0330 (8)	0.0354 (8)	0.0023 (6)	0.0127 (6)	0.0048 (6)
C4	0.0353 (8)	0.0347 (9)	0.0377 (8)	0.0030 (6)	0.0160 (7)	0.0063 (6)
C5	0.0468 (9)	0.0376 (9)	0.0311 (8)	-0.0072 (7)	0.0166 (7)	0.0002 (6)
C6	0.0405 (8)	0.0442 (10)	0.0343 (8)	-0.0055 (7)	0.0185 (7)	-0.0019 (7)
C7	0.0602 (12)	0.0458 (12)	0.0593 (13)	-0.0100 (10)	0.0187 (10)	0.0034 (9)
C8	0.0425 (11)	0.0773 (16)	0.0651 (14)	0.0057 (10)	0.0206 (10)	-0.0037 (12)
S1	0.0492 (3)	0.0424 (3)	0.0420 (2)	0.01172 (19)	0.0229 (2)	0.00695 (18)
S2	0.0462 (3)	0.0476 (3)	0.0400 (2)	0.01051 (19)	0.0161 (2)	0.01361 (18)
S3	0.0447 (2)	0.0423 (3)	0.0404 (2)	0.00839 (19)	0.01765 (19)	0.01208 (18)
S4	0.0482 (3)	0.0449 (3)	0.0505 (3)	0.0142 (2)	0.0295 (2)	0.01503 (19)
S5	0.0789 (4)	0.0510 (3)	0.0320 (2)	-0.0161 (3)	0.0210 (2)	0.00457 (19)
S6	0.0543 (3)	0.0919 (5)	0.0539 (3)	0.0106 (3)	0.0356 (3)	0.0085 (3)

Geometric parameters (Å, °)

C1—C2	1.321 (3)	C5—S3	1.759 (2)
C1—S1	1.733 (2)	C6—S6	1.7386 (19)
C1—H1	0.9300	C6—S4	1.7591 (18)
C2—S2	1.737 (2)	C7—S5	1.794 (2)
C2—H2	0.9300	C7—H7A	0.9600
C3—C4	1.339 (2)	C7—H7B	0.9600
C3—S1	1.7525 (18)	C7—H7C	0.9600
C3—S2	1.7565 (17)	C8—S6	1.784 (2)

C4—S3	1.7565 (18)	C8—H8A	0.9600
C4—S4	1.7562 (18)	C8—H8B	0.9600
C5—C6	1.346 (3)	C8—H8C	0.9600
C5—S5	1.7498 (18)		
C2—C1—S1	118.21 (15)	S5—C7—H7A	109.5
C2—C1—H1	120.9	S5—C7—H7B	109.5
S1—C1—H1	120.9	H7A—C7—H7B	109.5
C1—C2—S2	117.70 (15)	S5—C7—H7C	109.5
C1—C2—H2	121.1	H7A—C7—H7C	109.5
S2—C2—H2	121.1	H7B—C7—H7C	109.5
C4—C3—S1	122.09 (13)	S6—C8—H8A	109.5
C4—C3—S2	123.66 (14)	S6—C8—H8B	109.5
S1—C3—S2	114.26 (9)	H8A—C8—H8B	109.5
C3—C4—S3	123.67 (14)	S6—C8—H8C	109.5
C3—C4—S4	123.23 (13)	H8A—C8—H8C	109.5
S3—C4—S4	113.10 (10)	H8B—C8—H8C	109.5
C6—C5—S5	125.86 (14)	C1—S1—C3	94.57 (9)
C6—C5—S3	117.01 (13)	C2—S2—C3	94.58 (10)
S5—C5—S3	116.99 (11)	C4—S3—C5	94.04 (8)
C5—C6—S6	123.71 (14)	C4—S4—C6	94.24 (9)
C5—C6—S4	116.55 (14)	C5—S5—C7	100.79 (10)
S6—C6—S4	119.21 (11)	C6—S6—C8	103.63 (10)
