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1,3-Dimethyl-5,6,7,8-tetrahydro-4H-cyclohepta[c]thiophene-4,8-dione

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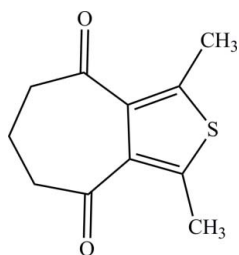
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.039; wR factor = 0.109; data-to-parameter ratio = 14.1.

In the title compound, $\text{C}_{11}\text{H}_{12}\text{O}_2\text{S}$, the C and S atoms of the central thiophene and the methyl groups, and the two carbonyl groups of the cycloheptanedione are almost coplanar [maximum deviation from the mean plane = 0.221 (2) Å]. The packing is stabilized by π - π interactions between the conjugated thiophenes, the shortest centroid-centroid distance between thiophene rings being 3.9759 (10) Å.

Related literature

The title compound was obtained as the product of our ongoing research of conjugated thiophenes for electronic devices and dye-sensitized solar cells (DSSCs). For applications of conjugated thiophenes, see: Amaresh *et al.* (2002); Nielsen & Bjonholm (2004). For related structures, see: Dufresne *et al.* (2007); Kuroda *et al.* (2005).



Experimental

Crystal data

$\text{C}_{11}\text{H}_{12}\text{O}_2\text{S}$	$V = 2074.75$ (13) Å ³
$M_r = 208.27$	$Z = 8$
Orthorhombic, $Pbca$	Mo $K\alpha$ radiation
$a = 15.9875$ (6) Å	$\mu = 0.28$ mm ⁻¹
$b = 7.6354$ (3) Å	$T = 298$ K
$c = 16.9963$ (6) Å	$0.30 \times 0.20 \times 0.18$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer	15732 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	1822 independent reflections
$T_{\min} = 0.920$, $T_{\max} = 0.951$	1430 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$	129 parameters
$wR(F^2) = 0.109$	H-atom parameters constrained
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.16$ e Å ⁻³
1822 reflections	$\Delta\rho_{\text{min}} = -0.29$ e Å ⁻³

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT-Plus (Bruker, 2001); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL-Plus (Sheldrick, 2008); 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: VM2059).

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supporting information

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1,3-Dimethyl-5,6,7,8-tetrahydro-4H-cyclohepta[c]thiophene-4,8-dione**Lijuan Yu, Yinghui Yin, Xiaole Zhou, Renjie Li and Tianyou Peng****S1. Comment**

Since the sulfur atom can contribute to the peripheral conjugation either by two *p*-electron moieties with its lone pair electrons or by *p*-sulfurane type conjugation, conjugated thiophenes have received much attention because of many new possibilities for constructing devices displaying unique optical, electrical, and mechanical properties (Nielsen *et al.*, 2004). Certain applications of conjugated thiophenes involve organic light emitting diodes and molecular wires, to be used in flexible light displays and/or low power consumption products (Amaresh *et al.*, 2002). Here, we report the structure of a novel conjugated thiophenes.

The crystal structure of the title compound is given in Fig.1. The crystallographic analysis confirms that the title compound consists of a central thiophene capped by two methyl groups. The molecular symmetry can be described by point group C₂. The cycloheptane ring shows a twisted boat conformation. The C—C bond lengths with each methyl are almost equal, with an average value of 1.506 (3) Å. Furthermore, π - π interactions stabilize the packing (Fig. 2). The closest centroid distance of approximate parallel thiophene rings is 3.9759 (10) Å.

S2. Experimental

The title compound was prepared according to the literature (Kuroda *et al.*, 2005), using diffusion of hexane into a toluene solution of the title compound at room temperature. ¹H NMR (CDCl₃, δ , p.p.m.): 2.45 (m, 4H), 2.32 (s, 6H), 1.93 (m, 2H). Analysis calculated (%): C 63.43, H 5.81; found (%): C 63.20, H 6.05.

S3. Refinement

All H-atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.96 and 0.97 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

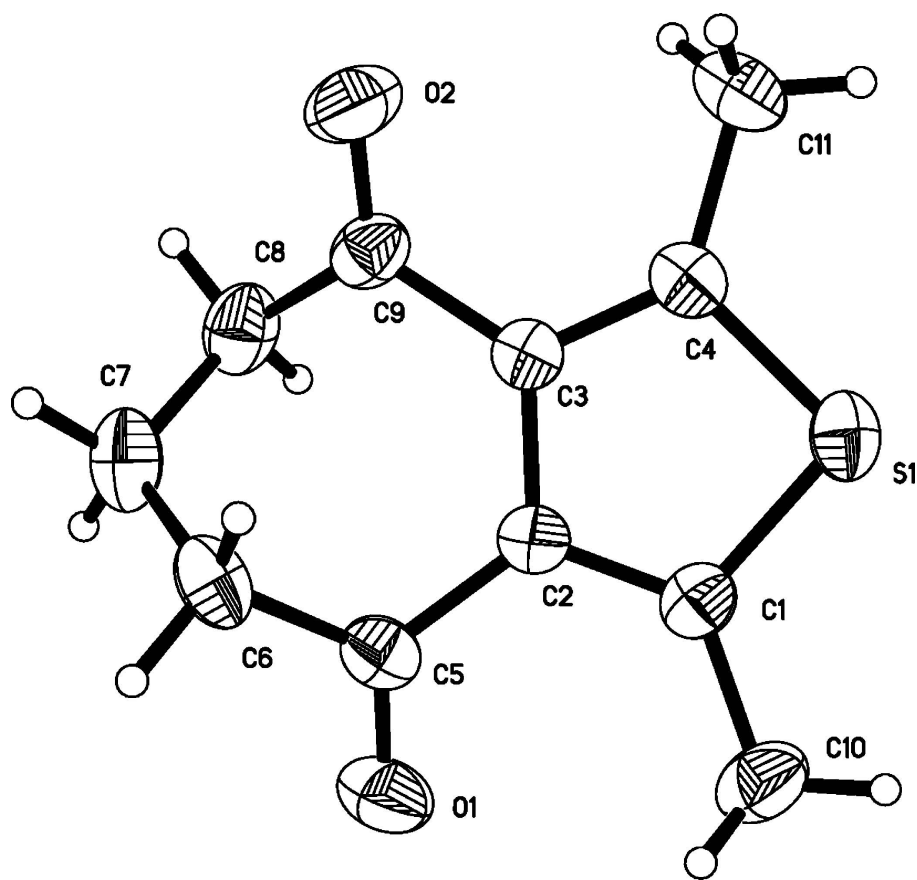


Figure 1

The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

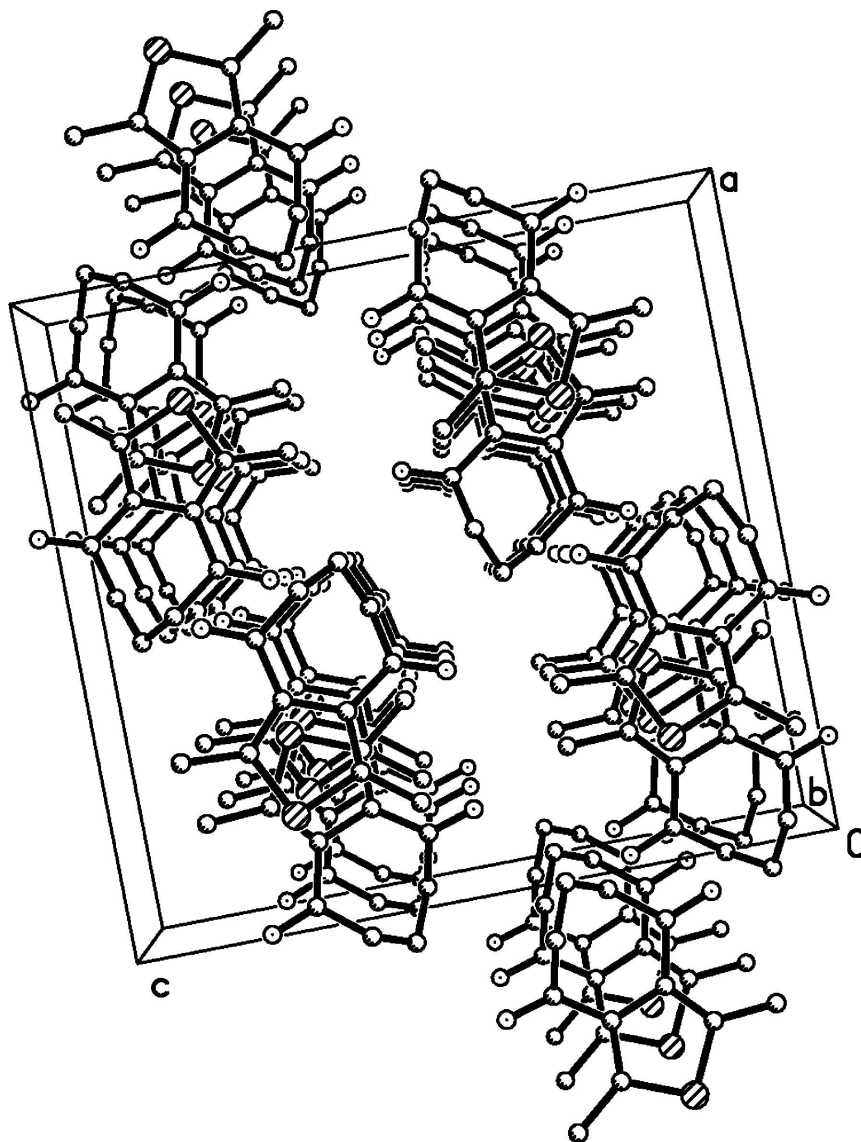


Figure 2

The packing of (I) viewed along the *b*-direction.

1,3-Dimethyl-5,6,7,8-tetrahydro-4*H*-cyclohepta[*c*]thiophene- 4,8-dione

Crystal data

$C_{11}H_{12}O_2S$

$M_r = 208.27$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 15.9875 (6) \text{ \AA}$

$b = 7.6354 (3) \text{ \AA}$

$c = 16.9963 (6) \text{ \AA}$

$V = 2074.75 (13) \text{ \AA}^3$

$Z = 8$

$F(000) = 880$

$D_x = 1.333 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4563 reflections

$\theta = 2.4\text{--}22.2^\circ$

$\mu = 0.28 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Block, yellow

$0.30 \times 0.20 \times 0.18 \text{ mm}$

Data collection

Bruker APEXII CCD area-detector
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2004)
 $T_{\min} = 0.920$, $T_{\max} = 0.951$

15732 measured reflections
1822 independent reflections
1430 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$
 $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.4^\circ$
 $h = -16 \rightarrow 19$
 $k = -9 \rightarrow 8$
 $l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.109$
 $S = 1.02$
1822 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
 $w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 0.5349P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.20027 (4)	0.11743 (8)	0.72032 (3)	0.0670 (2)
C2	0.35875 (12)	0.1124 (2)	0.70942 (10)	0.0496 (5)
C3	0.32681 (12)	0.1405 (2)	0.63092 (10)	0.0493 (5)
C1	0.29639 (14)	0.0980 (3)	0.76406 (11)	0.0575 (5)
C9	0.37901 (14)	0.1832 (3)	0.56162 (11)	0.0614 (5)
O2	0.35621 (12)	0.1474 (2)	0.49522 (8)	0.0898 (6)
C4	0.24140 (14)	0.1457 (2)	0.62814 (11)	0.0557 (5)
O1	0.47614 (12)	0.0907 (2)	0.79362 (10)	0.0882 (5)
C5	0.44794 (14)	0.0756 (3)	0.72741 (12)	0.0593 (5)
C6	0.50182 (13)	0.0112 (3)	0.66046 (14)	0.0688 (6)
H6A	0.4706	-0.0746	0.6303	0.083*
H6B	0.5508	-0.0467	0.6819	0.083*
C11	0.18386 (16)	0.1778 (3)	0.55955 (15)	0.0779 (7)
H11A	0.2030	0.2778	0.5305	0.117*
H11B	0.1282	0.1991	0.5785	0.117*
H11C	0.1836	0.0768	0.5259	0.117*

C10	0.30152 (17)	0.0610 (4)	0.85104 (12)	0.0842 (7)
H10A	0.3411	-0.0312	0.8602	0.126*
H10B	0.2475	0.0258	0.8700	0.126*
H10C	0.3192	0.1648	0.8782	0.126*
C8	0.45990 (15)	0.2776 (3)	0.57771 (14)	0.0766 (7)
H8A	0.4501	0.3667	0.6174	0.092*
H8B	0.4778	0.3364	0.5300	0.092*
C7	0.53003 (16)	0.1575 (3)	0.60577 (16)	0.0838 (7)
H7A	0.5568	0.1056	0.5601	0.101*
H7B	0.5716	0.2277	0.6328	0.101*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0548 (4)	0.0684 (4)	0.0779 (4)	0.0012 (3)	0.0116 (3)	-0.0081 (3)
C2	0.0545 (12)	0.0435 (10)	0.0509 (10)	0.0014 (8)	-0.0025 (8)	-0.0017 (8)
C3	0.0536 (12)	0.0423 (10)	0.0520 (10)	0.0016 (8)	-0.0011 (8)	-0.0048 (8)
C1	0.0666 (14)	0.0511 (12)	0.0549 (11)	0.0028 (9)	0.0053 (9)	-0.0034 (8)
C9	0.0753 (15)	0.0547 (12)	0.0542 (12)	0.0090 (11)	0.0068 (10)	0.0051 (9)
O2	0.1081 (14)	0.1103 (15)	0.0510 (9)	0.0094 (11)	0.0021 (8)	0.0020 (8)
C4	0.0569 (13)	0.0482 (11)	0.0620 (11)	0.0026 (9)	-0.0061 (9)	-0.0085 (8)
O1	0.0824 (12)	0.1029 (13)	0.0792 (11)	0.0074 (10)	-0.0269 (9)	0.0002 (9)
C5	0.0603 (13)	0.0496 (11)	0.0679 (12)	-0.0008 (10)	-0.0089 (10)	0.0025 (9)
C6	0.0524 (13)	0.0577 (13)	0.0962 (16)	0.0081 (10)	-0.0042 (11)	-0.0077 (11)
C11	0.0685 (15)	0.0810 (16)	0.0841 (16)	0.0095 (12)	-0.0247 (12)	-0.0115 (13)
C10	0.105 (2)	0.0922 (17)	0.0554 (13)	0.0103 (15)	0.0135 (12)	0.0038 (12)
C8	0.0785 (16)	0.0637 (14)	0.0875 (16)	-0.0077 (13)	0.0204 (13)	0.0109 (12)
C7	0.0628 (15)	0.0810 (17)	0.1076 (18)	-0.0070 (13)	0.0210 (14)	-0.0048 (14)

Geometric parameters (Å, °)

S1—C4	1.713 (2)	C6—H6A	0.9700
S1—C1	1.714 (2)	C6—H6B	0.9700
C2—C1	1.367 (3)	C11—H11A	0.9600
C2—C3	1.445 (2)	C11—H11B	0.9600
C2—C5	1.485 (3)	C11—H11C	0.9600
C3—C4	1.367 (3)	C10—H10A	0.9600
C3—C9	1.480 (3)	C10—H10B	0.9600
C1—C10	1.507 (3)	C10—H10C	0.9600
C9—O2	1.217 (2)	C8—C7	1.525 (4)
C9—C8	1.506 (3)	C8—H8A	0.9700
C4—C11	1.505 (3)	C8—H8B	0.9700
O1—C5	1.218 (2)	C7—H7A	0.9700
C5—C6	1.509 (3)	C7—H7B	0.9700
C6—C7	1.521 (3)		
C4—S1—C1	93.64 (10)	C4—C11—H11A	109.5
C1—C2—C3	112.43 (18)	C4—C11—H11B	109.5

C1—C2—C5	123.04 (18)	H11A—C11—H11B	109.5
C3—C2—C5	123.89 (17)	C4—C11—H11C	109.5
C4—C3—C2	112.91 (17)	H11A—C11—H11C	109.5
C4—C3—C9	121.98 (17)	H11B—C11—H11C	109.5
C2—C3—C9	124.64 (18)	C1—C10—H10A	109.5
C2—C1—C10	129.9 (2)	C1—C10—H10B	109.5
C2—C1—S1	110.63 (15)	H10A—C10—H10B	109.5
C10—C1—S1	119.38 (16)	C1—C10—H10C	109.5
O2—C9—C3	121.3 (2)	H10A—C10—H10C	109.5
O2—C9—C8	122.2 (2)	H10B—C10—H10C	109.5
C3—C9—C8	116.45 (18)	C9—C8—C7	113.58 (19)
C3—C4—C11	129.9 (2)	C9—C8—H8A	108.8
C3—C4—S1	110.39 (14)	C7—C8—H8A	108.8
C11—C4—S1	119.62 (18)	C9—C8—H8B	108.8
O1—C5—C2	121.9 (2)	C7—C8—H8B	108.8
O1—C5—C6	121.1 (2)	H8A—C8—H8B	107.7
C2—C5—C6	117.02 (17)	C6—C7—C8	114.50 (19)
C5—C6—C7	113.00 (18)	C6—C7—H7A	108.6
C5—C6—H6A	109.0	C8—C7—H7A	108.6
C7—C6—H6A	109.0	C6—C7—H7B	108.6
C5—C6—H6B	109.0	C8—C7—H7B	108.6
C7—C6—H6B	109.0	H7A—C7—H7B	107.6
H6A—C6—H6B	107.8		
C1—C2—C3—C4	-0.3 (2)	C9—C3—C4—C11	5.3 (3)
C5—C2—C3—C4	170.75 (18)	C2—C3—C4—S1	0.23 (19)
C1—C2—C3—C9	171.89 (18)	C9—C3—C4—S1	-172.24 (14)
C5—C2—C3—C9	-17.0 (3)	C1—S1—C4—C3	-0.06 (15)
C3—C2—C1—C10	177.1 (2)	C1—S1—C4—C11	-177.88 (17)
C5—C2—C1—C10	5.9 (3)	C1—C2—C5—O1	-26.3 (3)
C3—C2—C1—S1	0.3 (2)	C3—C2—C5—O1	163.48 (19)
C5—C2—C1—S1	-170.90 (15)	C1—C2—C5—C6	151.22 (19)
C4—S1—C1—C2	-0.14 (16)	C3—C2—C5—C6	-19.0 (3)
C4—S1—C1—C10	-177.35 (18)	O1—C5—C6—C7	-103.5 (2)
C4—C3—C9—O2	-34.1 (3)	C2—C5—C6—C7	78.9 (2)
C2—C3—C9—O2	154.3 (2)	O2—C9—C8—C7	-102.1 (3)
C4—C3—C9—C8	144.01 (19)	C3—C9—C8—C7	79.8 (2)
C2—C3—C9—C8	-27.6 (3)	C5—C6—C7—C8	-49.7 (3)
C2—C3—C4—C11	177.76 (19)	C9—C8—C7—C6	-37.6 (3)