

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

5-Methylphenanthro[2,3-*b*]thiopheneS. Ranjith,^a A. SubbiahPandi,^{a*} V. Dhayalan^b and A. K. MohanaKrishnan^b^aDepartment of Physics, Presidency College (Autonomous), Chennai 600 005, India, and ^bDepartment of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India

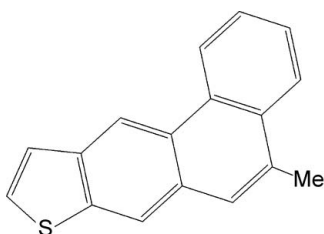
Correspondence e-mail: a_sp59@yahoo.in

Received 30 March 2011; accepted 20 April 2011

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.055; wR factor = 0.195; data-to-parameter ratio = 21.4.

The title compound, $\text{C}_{17}\text{H}_{12}\text{S}$, which crystallises with two molecules in the asymmetric unit, features four fused rings forming an essentially planar molecule, with maximum deviations from the mean plane of 0.078 (2) and 0.080 (2) Å for C atoms of the thiophene and phenanthrene groups in both the molecules. The crystal packing features weak $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For a related structure, see: Gunasekaran *et al.* (2010).

Experimental

Crystal data

$\text{C}_{17}\text{H}_{12}\text{S}$
 $M_r = 248.33$
 Monoclinic, $P2_1/n$
 $a = 18.7011$ (10) Å

$b = 5.8199$ (3) Å
 $c = 23.4546$ (14) Å
 $\beta = 105.158$ (2)°
 $V = 2463.9$ (2) Å³

$Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.24$ mm⁻¹

$T = 293$ K
 $0.25 \times 0.22 \times 0.19$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.981$, $T_{\max} = 0.985$

31917 measured reflections
 6983 independent reflections
 4446 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.195$
 $S = 1.06$
 6983 reflections
 327 parameters

2 restraints
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.47$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.30$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C2}-\text{H2}\cdots\text{Cg4}^i$	0.93	2.84	3.612 (3)	141
$\text{C2}'-\text{H2}'\cdots\text{Cg14}^{ii}$	0.93	2.85	3.619 (3)	141
$\text{C11}-\text{H11}\cdots\text{Cg1}^i$	0.93	2.72	3.515 (3)	144
$\text{C11}'-\text{H11}'\cdots\text{Cg11}^{ii}$	0.93	2.82	3.610 (3)	143
$\text{C17}'-\text{H17B}'\cdots\text{Cg14}^{iii}$	0.96	2.98	3.581 (3)	121
$\text{C17}-\text{H17F}\cdots\text{Cg4}^{iii}$	0.96	2.92	3.565 (3)	125

Symmetry codes: (i) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (iii) $x, y - 1, z$. Cg1, Cg4, Cg11 and Cg14 are the centroids of the S1/C1-C4, C9-C14, S1'/C1'-C4' and C9'-C14' rings, respectively.

Data collection: APEX2 (Bruker, 2004); cell refinement: APEX2; data reduction: SAINT (Bruker, 2004); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

SR and ASP thank Dr Babu Varghese, SAIF, IIT, Chennai, India, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5506).

References

- Bruker (2004). APEX2 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Gunasekaran, B., Dhayalan, V., Mohanakrishnan, A. K., Chakkaravarthi, G. & Manivannan, V. (2010). *Acta Cryst.* **E66**, o1449.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supplementary materials

Acta Cryst. (2011). E67, o1243 [doi:10.1107/S1600536811014838]

5-Methylphenanthro[2,3-*b*]thiophene

S. Ranjith, A. SubbiahPandi, V. Dhayalan and A. K. MohanaKrishnan

Comment

X-Ray analysis confirms the molecular structure and atom connectivity as illustrated in Fig. 1. The bond lengths and angles in (Fig. 1) agree with those observed in other benzothiophene derivative (Gunasekaran *et al.*, 2010). Both molecules are essentially planar, with maximum deviation of 0.078 (2) and 0.080 (2) Å for atoms C2 and C10'. The molecules lack hydrogen bonding functionality and pack in layers parallel to the (010) planes. In addition to van der Waals interaction, the crystal packing is stabilized by C–H \cdots π hydrogen bonds (Table. 1).

Experimental

To a solution of diethyl-2-((3-(bromomethyl)thiophen-2-yl)methylene) malonate (1 g, 2.88 mmol) in dry 1,2-dichloroethane (10ml), 1-methylnaphthalene (0.49 g, 3.44 mmol) and anhydrous ZnBr₂ (0.64 g, 2.84 mmol) were added. It was then stirred at room temperature for 8 h and then refluxed for 1 h under N₂ atmosphere. The solvent was removed and the residue was quenched with ice-water (50 ml) containing 1 ml of conc. HCl, extracted with chloroform (3 x 10 ml) and dried (Na₂SO₄). Removal of the solvent followed by flash column chromatography purification (n-hexane/ethyl acetate 99:1) led to the isolation of 5-methyl phenanthro[2,3-*b*]thiophene as a colorless crystals. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution of the title compound in methanol at room temperature.

Refinement

All H atoms were fixed geometrically and allowed to ride on their parent C atoms, with C–H distances fixed in the range 0.93–0.97 Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H 1.2 $U_{\text{eq}}(\text{C})$ for other H atoms. The displacement ellipsoids of C1' and C2' were restrained to be equal within an effective e.s.d. of 0.005.

Figures

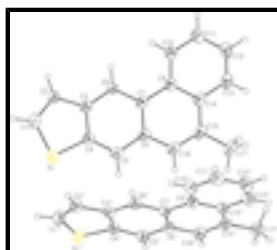


Fig. 1. The structure of showing the atom-numbering scheme and intramolecular hydrogen bond. Displacement ellipsoids are drawn at the 30% probability level.

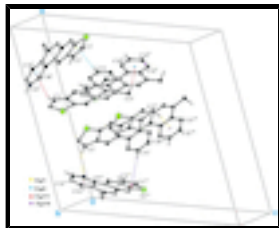


Fig. 2. C—H... π interactions (dotted lines) in the title compound. Cg denotes ring centroid. [Symmetry code: (i) $3/2-X, 1/2+Y, 1/2-Z$; (ii) $X, -1+Y, Z$; (iii) $X, -1+Y, Z$; (iv) $1/2-X, 1/2+Y, 1/2-Z$]

5-Methylphenanthro[2,3-*b*]thiophene

Crystal data

$C_{17}H_{12}S$

$M_r = 248.33$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 18.7011(10)\ \text{\AA}$

$b = 5.8199(3)\ \text{\AA}$

$c = 23.4546(14)\ \text{\AA}$

$\beta = 105.158(2)^\circ$

$V = 2463.9(2)\ \text{\AA}^3$

$Z = 8$

$F(000) = 1040$

$D_x = 1.339\ \text{Mg m}^{-3}$

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

Cell parameters from 6983 reflections

$\theta = 1.3\text{--}29.7^\circ$

$\mu = 0.24\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, colourless

$0.25 \times 0.22 \times 0.19\ \text{mm}$

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

ω and ϕ scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.981$, $T_{\max} = 0.985$

31917 measured reflections

6983 independent reflections

4446 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$

$\theta_{\max} = 29.7^\circ$, $\theta_{\min} = 1.3^\circ$

$h = -25 \rightarrow 26$

$k = -8 \rightarrow 8$

$l = -32 \rightarrow 32$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.055$

$wR(F^2) = 0.195$

$S = 1.06$

6983 reflections

327 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.1022P)^2 + 0.5162P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.47\ \text{e \AA}^{-3}$

2 restraints

$$\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$$

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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1'	0.33522 (15)	0.2874 (5)	0.07093 (11)	0.0702 (7)
H1'	0.3227	0.3551	0.0337	0.084*
C1	0.55737 (13)	0.7887 (4)	0.06335 (10)	0.0601 (6)
H1	0.5490	0.8546	0.0261	0.072*
C2	0.60214 (13)	0.8818 (4)	0.11144 (10)	0.0516 (5)
H2	0.6274	1.0193	0.1114	0.062*
C2'	0.31625 (12)	0.3776 (4)	0.11600 (10)	0.0538 (5)
H2'	0.2897	0.5136	0.1144	0.065*
C3	0.60700 (11)	0.7451 (3)	0.16387 (9)	0.0431 (4)
C3'	0.34245 (11)	0.2359 (3)	0.16957 (9)	0.0449 (4)
C4	0.56138 (11)	0.5470 (3)	0.15084 (9)	0.0457 (4)
C4'	0.38181 (11)	0.0412 (3)	0.15862 (9)	0.0451 (4)
C5	0.55612 (11)	0.3950 (3)	0.19459 (10)	0.0487 (5)
H5	0.5255	0.2669	0.1856	0.058*
C5'	0.41350 (11)	-0.1131 (4)	0.20223 (10)	0.0496 (5)
H5'	0.4394	-0.2397	0.1939	0.059*
C6'	0.40615 (10)	-0.0763 (3)	0.25924 (9)	0.0438 (4)
C6	0.59727 (11)	0.4349 (3)	0.25262 (9)	0.0446 (4)
C7	0.64532 (10)	0.6288 (3)	0.26671 (9)	0.0411 (4)
C7'	0.36605 (10)	0.1153 (3)	0.27204 (8)	0.0406 (4)
C8'	0.33449 (11)	0.2692 (3)	0.22664 (9)	0.0461 (4)
H8'	0.3079	0.3951	0.2345	0.055*
C8	0.64871 (11)	0.7820 (3)	0.22128 (9)	0.0437 (4)
H8	0.6794	0.9101	0.2298	0.052*
C9'	0.35973 (10)	0.1459 (3)	0.33238 (9)	0.0431 (4)
C9	0.68848 (11)	0.6597 (3)	0.32765 (8)	0.0416 (4)
C10'	0.31838 (11)	0.3236 (4)	0.34787 (10)	0.0517 (5)
H10'	0.2927	0.4236	0.3187	0.062*
C10	0.74001 (12)	0.8395 (4)	0.34412 (9)	0.0489 (5)
H10	0.7475	0.9395	0.3153	0.059*
C11'	0.31458 (12)	0.3548 (5)	0.40558 (11)	0.0601 (6)

supplementary materials

H11'	0.2870	0.4754	0.4149	0.072*
C11	0.77956 (13)	0.8707 (4)	0.40190 (10)	0.0567 (5)
H11	0.8128	0.9921	0.4119	0.068*
C12'	0.35180 (13)	0.2064 (5)	0.44909 (10)	0.0620 (6)
H12'	0.3492	0.2269	0.4878	0.074*
C12	0.76975 (13)	0.7205 (4)	0.44519 (10)	0.0597 (6)
H12	0.7965	0.7414	0.4843	0.072*
C13'	0.39265 (13)	0.0289 (4)	0.43557 (10)	0.0573 (6)
H13'	0.4176	-0.0697	0.4653	0.069*
C13	0.72101 (13)	0.5424 (4)	0.43057 (10)	0.0561 (6)
H13	0.7153	0.4424	0.4600	0.067*
C14'	0.39736 (10)	-0.0063 (3)	0.37744 (9)	0.0444 (4)
C14	0.67910 (12)	0.5061 (3)	0.37200 (9)	0.0459 (5)
C15'	0.44026 (11)	-0.1964 (4)	0.36283 (10)	0.0502 (5)
C15	0.62704 (12)	0.3163 (4)	0.35639 (10)	0.0503 (5)
C16	0.59013 (12)	0.2845 (4)	0.29960 (10)	0.0516 (5)
H16	0.5584	0.1591	0.2901	0.062*
C16'	0.44238 (12)	-0.2279 (4)	0.30675 (10)	0.0517 (5)
H16'	0.4684	-0.3534	0.2980	0.062*
C17'	0.48297 (13)	-0.3532 (4)	0.41088 (11)	0.0644 (6)
H17A	0.5035	-0.4779	0.3935	0.097*
H17B	0.4503	-0.4131	0.4328	0.097*
H17C	0.5223	-0.2686	0.4369	0.097*
C17	0.61402 (14)	0.1585 (4)	0.40359 (12)	0.0658 (7)
H17D	0.5801	0.0392	0.3856	0.099*
H17E	0.5935	0.2445	0.4304	0.099*
H17F	0.6602	0.0911	0.4248	0.099*
S1	0.51607 (4)	0.53496 (12)	0.07611 (3)	0.0620 (2)
S1'	0.38490 (4)	0.03471 (13)	0.08505 (3)	0.0655 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1'	0.0742 (16)	0.0787 (17)	0.0525 (13)	-0.0132 (14)	0.0075 (12)	0.0174 (12)
C1	0.0656 (14)	0.0640 (14)	0.0512 (12)	0.0067 (11)	0.0162 (10)	0.0135 (11)
C2	0.0656 (13)	0.0447 (11)	0.0494 (12)	-0.0016 (10)	0.0235 (10)	0.0059 (9)
C2'	0.0551 (12)	0.0461 (11)	0.0569 (13)	0.0003 (9)	0.0087 (10)	0.0039 (10)
C3	0.0492 (10)	0.0382 (9)	0.0471 (10)	0.0007 (8)	0.0218 (8)	0.0017 (8)
C3'	0.0431 (10)	0.0442 (10)	0.0451 (10)	-0.0050 (8)	0.0073 (8)	-0.0005 (8)
C4	0.0462 (10)	0.0438 (10)	0.0495 (11)	-0.0004 (8)	0.0171 (9)	-0.0004 (9)
C4'	0.0434 (10)	0.0489 (11)	0.0426 (10)	-0.0073 (8)	0.0103 (8)	-0.0040 (9)
C5	0.0506 (11)	0.0390 (10)	0.0594 (13)	-0.0073 (8)	0.0193 (10)	-0.0007 (9)
C5'	0.0480 (11)	0.0456 (11)	0.0548 (12)	0.0030 (9)	0.0130 (9)	-0.0045 (9)
C6'	0.0407 (10)	0.0410 (10)	0.0475 (11)	0.0008 (8)	0.0077 (8)	-0.0011 (8)
C6	0.0484 (10)	0.0378 (10)	0.0541 (12)	0.0000 (8)	0.0248 (9)	0.0038 (8)
C7	0.0473 (10)	0.0356 (9)	0.0466 (10)	0.0028 (8)	0.0232 (8)	0.0019 (8)
C7'	0.0368 (9)	0.0419 (10)	0.0416 (10)	-0.0029 (7)	0.0075 (7)	-0.0029 (8)
C8'	0.0467 (10)	0.0422 (10)	0.0483 (11)	0.0028 (8)	0.0105 (8)	-0.0032 (9)

C8	0.0555 (11)	0.0358 (9)	0.0447 (10)	-0.0065 (8)	0.0219 (9)	-0.0012 (8)
C9'	0.0379 (9)	0.0459 (10)	0.0449 (10)	-0.0017 (8)	0.0098 (8)	-0.0009 (8)
C9	0.0482 (10)	0.0391 (10)	0.0434 (10)	0.0057 (8)	0.0225 (8)	0.0021 (8)
C10'	0.0453 (10)	0.0554 (12)	0.0540 (12)	0.0048 (9)	0.0124 (9)	-0.0041 (10)
C10	0.0553 (11)	0.0482 (11)	0.0473 (11)	-0.0019 (9)	0.0208 (9)	0.0000 (9)
C11'	0.0498 (12)	0.0714 (15)	0.0624 (14)	0.0064 (11)	0.0205 (10)	-0.0104 (12)
C11	0.0562 (12)	0.0620 (13)	0.0537 (13)	0.0018 (11)	0.0178 (10)	-0.0061 (11)
C12'	0.0543 (12)	0.0867 (18)	0.0479 (12)	-0.0021 (12)	0.0187 (10)	-0.0068 (12)
C12	0.0649 (14)	0.0696 (15)	0.0446 (12)	0.0140 (12)	0.0145 (10)	-0.0021 (11)
C13'	0.0513 (12)	0.0740 (15)	0.0457 (12)	-0.0044 (11)	0.0113 (9)	0.0064 (11)
C13	0.0670 (14)	0.0600 (13)	0.0466 (12)	0.0194 (11)	0.0244 (10)	0.0099 (10)
C14'	0.0374 (9)	0.0502 (11)	0.0436 (10)	-0.0045 (8)	0.0072 (8)	0.0029 (9)
C14	0.0542 (11)	0.0428 (10)	0.0483 (11)	0.0135 (8)	0.0272 (9)	0.0082 (8)
C15'	0.0454 (10)	0.0468 (11)	0.0546 (12)	-0.0005 (9)	0.0060 (9)	0.0073 (9)
C15	0.0589 (12)	0.0428 (11)	0.0599 (13)	0.0107 (9)	0.0346 (10)	0.0149 (9)
C16	0.0565 (12)	0.0402 (10)	0.0648 (14)	-0.0026 (9)	0.0280 (10)	0.0101 (9)
C16'	0.0501 (11)	0.0443 (11)	0.0591 (13)	0.0091 (9)	0.0113 (9)	0.0001 (10)
C17'	0.0607 (14)	0.0607 (14)	0.0667 (15)	0.0089 (11)	0.0077 (11)	0.0168 (12)
C17	0.0743 (15)	0.0596 (14)	0.0740 (16)	0.0110 (12)	0.0380 (13)	0.0299 (12)
S1	0.0603 (4)	0.0672 (4)	0.0536 (4)	-0.0073 (3)	0.0061 (3)	0.0019 (3)
S1'	0.0684 (4)	0.0788 (4)	0.0530 (4)	-0.0042 (3)	0.0223 (3)	-0.0040 (3)

Geometric parameters (Å, °)

C1'—C2'	1.310 (3)	C9'—C14'	1.417 (3)
C1'—S1'	1.725 (3)	C9—C10	1.407 (3)
C1'—H1'	0.9300	C9—C14	1.417 (3)
C1—C2	1.331 (3)	C10'—C11'	1.386 (3)
C1—S1	1.729 (3)	C10'—H10'	0.9300
C1—H1	0.9300	C10—C11	1.376 (3)
C2—C3	1.447 (3)	C10—H10	0.9300
C2—H2	0.9300	C11'—C12'	1.379 (3)
C2'—C3'	1.476 (3)	C11'—H11'	0.9300
C2'—H2'	0.9300	C11—C12	1.387 (3)
C3—C8	1.385 (3)	C11—H11	0.9300
C3—C4	1.419 (3)	C12'—C13'	1.371 (3)
C3'—C8'	1.398 (3)	C12'—H12'	0.9300
C3'—C4'	1.411 (3)	C12—C13	1.364 (4)
C4—C5	1.378 (3)	C12—H12	0.9300
C4—S1	1.737 (2)	C13'—C14'	1.404 (3)
C4'—C5'	1.373 (3)	C13'—H13'	0.9300
C4'—S1'	1.742 (2)	C13—C14	1.408 (3)
C5—C6	1.397 (3)	C13—H13	0.9300
C5—H5	0.9300	C14'—C15'	1.459 (3)
C5'—C6'	1.396 (3)	C14—C15	1.455 (3)
C5'—H5'	0.9300	C15'—C16'	1.339 (3)
C6'—C7'	1.419 (3)	C15'—C17'	1.507 (3)
C6'—C16'	1.444 (3)	C15—C16	1.343 (3)
C6—C7	1.426 (3)	C15—C17	1.507 (3)

supplementary materials

C6—C16	1.441 (3)	C16—H16	0.9300
C7—C8	1.404 (3)	C16'—H16'	0.9300
C7—C9	1.457 (3)	C17'—H17A	0.9600
C7'—C8'	1.399 (3)	C17'—H17B	0.9600
C7'—C9'	1.461 (3)	C17'—H17C	0.9600
C8'—H8'	0.9300	C17—H17D	0.9600
C8—H8	0.9300	C17—H17E	0.9600
C9'—C10'	1.395 (3)	C17—H17F	0.9600
C2'—C1'—S1'	115.65 (19)	C11'—C10'—H10'	119.2
C2'—C1'—H1'	122.2	C9'—C10'—H10'	119.2
S1'—C1'—H1'	122.2	C11—C10—C9	121.5 (2)
C2—C1—S1	114.30 (17)	C11—C10—H10	119.3
C2—C1—H1	122.9	C9—C10—H10	119.3
S1—C1—H1	122.9	C12'—C11'—C10'	119.7 (2)
C1—C2—C3	112.4 (2)	C12'—C11'—H11'	120.1
C1—C2—H2	123.8	C10'—C11'—H11'	120.1
C3—C2—H2	123.8	C10—C11—C12	119.9 (2)
C1'—C2'—C3'	111.3 (2)	C10—C11—H11	120.1
C1'—C2'—H2'	124.3	C12—C11—H11	120.1
C3'—C2'—H2'	124.3	C13'—C12'—C11'	120.3 (2)
C8—C3—C4	119.30 (18)	C13'—C12'—H12'	119.8
C8—C3—C2	129.41 (19)	C11'—C12'—H12'	119.8
C4—C3—C2	111.29 (19)	C13—C12—C11	120.2 (2)
C8'—C3'—C4'	118.48 (18)	C13—C12—H12	119.9
C8'—C3'—C2'	130.0 (2)	C11—C12—H12	119.9
C4'—C3'—C2'	111.52 (19)	C12'—C13'—C14'	121.0 (2)
C5—C4—C3	121.2 (2)	C12'—C13'—H13'	119.5
C5—C4—S1	127.90 (17)	C14'—C13'—H13'	119.5
C3—C4—S1	110.93 (15)	C12—C13—C14	121.6 (2)
C5'—C4'—C3'	122.2 (2)	C12—C13—H13	119.2
C5'—C4'—S1'	127.17 (17)	C14—C13—H13	119.2
C3'—C4'—S1'	110.59 (15)	C13'—C14'—C9'	119.2 (2)
C4—C5—C6	119.36 (19)	C13'—C14'—C15'	121.09 (19)
C4—C5—H5	120.3	C9'—C14'—C15'	119.68 (19)
C6—C5—H5	120.3	C13—C14—C9	118.5 (2)
C4'—C5'—C6'	118.82 (19)	C13—C14—C15	121.65 (19)
C4'—C5'—H5'	120.6	C9—C14—C15	119.8 (2)
C6'—C5'—H5'	120.6	C16'—C15'—C14'	119.61 (19)
C5'—C6'—C7'	120.86 (18)	C16'—C15'—C17'	120.4 (2)
C5'—C6'—C16'	120.13 (18)	C14'—C15'—C17'	120.0 (2)
C7'—C6'—C16'	118.95 (18)	C16—C15—C14	119.40 (18)
C5—C6—C7	120.71 (18)	C16—C15—C17	120.3 (2)
C5—C6—C16	120.42 (19)	C14—C15—C17	120.3 (2)
C7—C6—C16	118.84 (19)	C15—C16—C6	123.3 (2)
C8—C7—C6	118.51 (19)	C15—C16—H16	118.4
C8—C7—C9	122.88 (18)	C6—C16—H16	118.4
C6—C7—C9	118.61 (17)	C15'—C16'—C6'	123.1 (2)
C8'—C7'—C6'	118.90 (18)	C15'—C16'—H16'	118.5
C8'—C7'—C9'	122.30 (18)	C6'—C16'—H16'	118.5

C6'—C7'—C9'	118.80 (17)	C15'—C17'—H17A	109.5
C3'—C8'—C7'	120.70 (19)	C15'—C17'—H17B	109.5
C3'—C8'—H8'	119.7	H17A—C17'—H17B	109.5
C7'—C8'—H8'	119.7	C15'—C17'—H17C	109.5
C3—C8—C7	120.92 (18)	H17A—C17'—H17C	109.5
C3—C8—H8	119.5	H17B—C17'—H17C	109.5
C7—C8—H8	119.5	C15—C17—H17D	109.5
C10'—C9'—C14'	118.04 (19)	C15—C17—H17E	109.5
C10'—C9'—C7'	122.22 (18)	H17D—C17—H17E	109.5
C14'—C9'—C7'	119.73 (18)	C15—C17—H17F	109.5
C10—C9—C14	118.38 (19)	H17D—C17—H17F	109.5
C10—C9—C7	121.69 (17)	H17E—C17—H17F	109.5
C14—C9—C7	119.93 (18)	C1—S1—C4	91.03 (11)
C11'—C10'—C9'	121.7 (2)	C1'—S1'—C4'	90.93 (12)
S1—C1—C2—C3	1.1 (3)	C6—C7—C9—C14	-3.5 (3)
S1'—C1'—C2'—C3'	-0.4 (3)	C14'—C9'—C10'—C11'	1.0 (3)
C1—C2—C3—C8	178.3 (2)	C7'—C9'—C10'—C11'	-178.1 (2)
C1—C2—C3—C4	-1.1 (3)	C14—C9—C10—C11	-1.4 (3)
C1'—C2'—C3'—C8'	179.8 (2)	C7—C9—C10—C11	178.70 (19)
C1'—C2'—C3'—C4'	0.6 (3)	C9'—C10'—C11'—C12'	-0.6 (4)
C8—C3—C4—C5	2.0 (3)	C9—C10—C11—C12	1.0 (3)
C2—C3—C4—C5	-178.43 (19)	C10'—C11'—C12'—C13'	0.2 (4)
C8—C3—C4—S1	-178.83 (15)	C10—C11—C12—C13	0.0 (3)
C2—C3—C4—S1	0.7 (2)	C11'—C12'—C13'—C14'	-0.2 (4)
C8'—C3'—C4'—C5'	-1.2 (3)	C11—C12—C13—C14	-0.5 (3)
C2'—C3'—C4'—C5'	178.05 (19)	C12'—C13'—C14'—C9'	0.6 (3)
C8'—C3'—C4'—S1'	-179.91 (15)	C12'—C13'—C14'—C15'	-179.4 (2)
C2'—C3'—C4'—S1'	-0.6 (2)	C10'—C9'—C14'—C13'	-0.9 (3)
C3—C4—C5—C6	-0.9 (3)	C7'—C9'—C14'—C13'	178.15 (18)
S1—C4—C5—C6	-179.89 (16)	C10'—C9'—C14'—C15'	179.06 (18)
C3'—C4'—C5'—C6'	0.3 (3)	C7'—C9'—C14'—C15'	-1.9 (3)
S1'—C4'—C5'—C6'	178.75 (16)	C12—C13—C14—C9	0.1 (3)
C4'—C5'—C6'—C7'	0.7 (3)	C12—C13—C14—C15	-179.93 (19)
C4'—C5'—C6'—C16'	-176.45 (19)	C10—C9—C14—C13	0.9 (3)
C4—C5—C6—C7	-1.0 (3)	C7—C9—C14—C13	-179.26 (17)
C4—C5—C6—C16	177.11 (19)	C10—C9—C14—C15	-179.12 (17)
C5—C6—C7—C8	1.8 (3)	C7—C9—C14—C15	0.8 (3)
C16—C6—C7—C8	-176.34 (18)	C13'—C14'—C15'—C16'	178.6 (2)
C5—C6—C7—C9	-178.46 (17)	C9'—C14'—C15'—C16'	-1.4 (3)
C16—C6—C7—C9	3.4 (3)	C13'—C14'—C15'—C17'	-2.5 (3)
C5'—C6'—C7'—C8'	-0.7 (3)	C9'—C14'—C15'—C17'	177.52 (19)
C16'—C6'—C7'—C8'	176.44 (18)	C13—C14—C15—C16	-177.8 (2)
C5'—C6'—C7'—C9'	179.81 (18)	C9—C14—C15—C16	2.2 (3)
C16'—C6'—C7'—C9'	-3.0 (3)	C13—C14—C15—C17	2.9 (3)
C4'—C3'—C8'—C7'	1.2 (3)	C9—C14—C15—C17	-177.15 (18)
C2'—C3'—C8'—C7'	-177.96 (19)	C14—C15—C16—C6	-2.3 (3)
C6'—C7'—C8'—C3'	-0.2 (3)	C17—C15—C16—C6	177.00 (19)
C9'—C7'—C8'—C3'	179.21 (17)	C5—C6—C16—C15	-178.7 (2)
C4—C3—C8—C7	-1.2 (3)	C7—C6—C16—C15	-0.5 (3)

supplementary materials

C2—C3—C8—C7	179.37 (19)	C14'—C15'—C16'—C6'	2.4 (3)
C6—C7—C8—C3	-0.7 (3)	C17'—C15'—C16'—C6'	-176.44 (19)
C9—C7—C8—C3	179.61 (17)	C5'—C6'—C16'—C15'	177.0 (2)
C8'—C7'—C9'—C10'	3.6 (3)	C7'—C6'—C16'—C15'	-0.2 (3)
C6'—C7'—C9'—C10'	-176.94 (18)	C2—C1—S1—C4	-0.56 (19)
C8'—C7'—C9'—C14'	-175.43 (18)	C5—C4—S1—C1	178.9 (2)
C6'—C7'—C9'—C14'	4.0 (3)	C3—C4—S1—C1	-0.11 (16)
C8—C7—C9—C10	-3.9 (3)	C2'—C1'—S1'—C4'	0.0 (2)
C6—C7—C9—C10	176.38 (17)	C5'—C4'—S1'—C1'	-178.2 (2)
C8—C7—C9—C14	176.23 (17)	C3'—C4'—S1'—C1'	0.36 (16)

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C2—H2 \cdots Cg4 ⁱ	0.93	2.84	3.612 (3)	141
C2'—H2' \cdots Cg14 ⁱⁱ	0.93	2.85	3.619 (3)	141
C11—H11 \cdots Cg1 ⁱ	0.93	2.72	3.515 (3)	144
C11'—H11' \cdots Cg11 ⁱⁱ	0.93	2.82	3.610 (3)	143
C17'—H17B \cdots Cg14 ⁱⁱⁱ	0.96	2.98	3.581 (3)	121
C17—H17F \cdots Cg4 ⁱⁱⁱ	0.96	2.92	3.565 (3)	125

Symmetry codes: (i) $-x+3/2, y+1/2, -z+1/2$; (ii) $-x+1/2, y+1/2, -z+1/2$; (iii) $x, y-1, z$.

Fig. 1

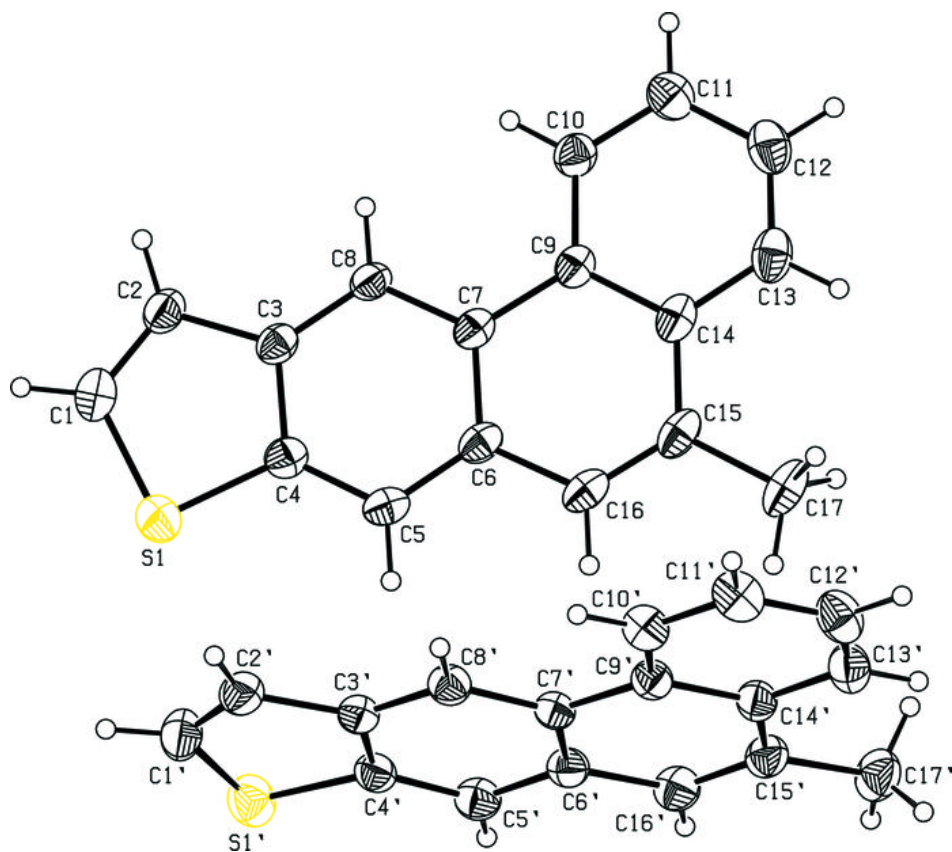


Fig. 2

