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3,5-Bis(trimethylsilyl)triphenyleno[1,12-*bcd*]thiophene

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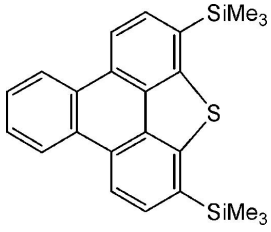
Key indicators: single-crystal X-ray study; T = 103 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.089; data-to-parameter ratio = 14.9.

In the title compound, $C_{24}H_{26}SSi_2$, the fused ring system plus the two Si atoms are coplanar, consistent with planar triphenylene but contrary to bowl-shaped triphenyleno[1,12-*bcd*:4,5-*b*′*c*′*d*′:8,9-*b*″*c*″*d*″]trithiophene. The bond alternation pattern of the C—C bonds in the title compound is similar to that found in triphenyleno[1,12-*bcd*:4,5-*b*′*c*′*d*′:8,9-*b*″*c*″*d*″]-trithiophene, although the lengths of the C—C bonds around the central and thiophene rings of the title compound are slightly different from the corresponding bonds of triphenyleno[1,12-*bcd*:4,5-*b*′*c*′*d*′:8,9-*b*″*c*″*d*″]trithiophene.

Related literature

Synthesis of triphenyleno[1,12-*bcd*]thiophene and its derivatives: Klemm & Lawrence (1979); Klemm *et al.* (1987); Ashe *et al.* (1990). X-ray crystallographic study of triphenylene: Ahmed & Trotter (1963). Related compounds: Chantson *et al.* (2003); Imamura *et al.* (1999).



Experimental

Crystal data

$C_{24}H_{26}SSi_2$
M_r = 402.69
Monoclinic, $P2_1/n$
a = 7.0230 (3) Å
b = 14.4933 (7) Å
c = 20.8358 (10) Å
β = 92.707 (1)°
V = 2118.44 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 103 K
0.45 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.937, T_{max} = 0.947
12122 measured reflections
3730 independent reflections
3375 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.089
S = 1.05
3730 reflections
250 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2001); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2130).

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supplementary materials

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3,5-Bis(trimethylsilyl)triphenyleno[1,12-*bcd*]thiophene

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Comment

The structure of triphenyleno[1,12-bcd]thiophene (2) where one of the three bay regions of triphenylene (4) is connected by a sulfur bridge is of interest in terms of comparison to the bowl shaped triphenyleno[1,12-bcd:4,5 − b'c'd':8,9 − b"c"d"]trithiophene (3) (Imamura *et al.*, 1999) (Fig. 3). Although the synthesis of triphenyleno[1,12-bcd]thiophene (2) and its derivatives has been already reported (Klemm and Lawrence, 1979; Klemm *et al.*, 1987; Ashe, *et al.*, 1990), no reports on the X-ray crystallographic analysis of (2) have appeared. This paper presents the synthesis and first X-ray crystallographic analysis of 3,5-bis(trimethyl- silyl)-triphenyleno[1,12-bcd]thiophene (1) having a triphenyleno[1,12-bcd]- thiophene (2) skeleton.

The X-ray structural analysis reveals that the fused ring system including the two silicon atoms of (1) is planar, consistent with the planar triphenylene (4) (Ahmed & Trotter, 1963) but contrary to the bowl shaped trithiophene derivative (3) (Imamura *et al.*, 1999). A pattern of bond alternation of C—C bonds in (1) is similar to that found in (4), although the lengths of the C—C bonds around the central and thiophene rings of (1) are slightly different from those of the corresponding bonds of (4). The C5—C6 and C11—C12 distances (1.467 (2) and 1.465 (2) Å) are slightly longer than those of (4) (*ca* 1.44 Å), while the C17—C18 distance (1.412 (2) Å) is slightly shorter than the corresponding bonds of (4) (*ca* 1.44 Å) and 4,6-bis(trimethyl- silyl)dibenzothiophene (1.448 (2) Å) (Chantson *et al.*, 2003). The angles of C4—C5—C6 and C11—C12—C13 are 127.45 (15) and 127.38 (15) °, respectively, larger than the corresponding angles of triphenylene (4) (Ahmed & Trotter, 1963) (*ca* 120 °), reflecting a formation of the thiophene ring in (1).

Experimental

To a hexane (4 ml) solution of triphenyleno[1,12-bcd]thiophene (2) was added TMEDA (0.12 ml, 0.8 mmol) and butyllithium (1.58 *M* in hexane; 0.51 ml, 0.80 mmol) at room temperature. The resulting mixture was heated at 60 °C for 3 h. After being cooled to room temperature, the mixture was treated with chlorotrimethylsilane (0.10 ml, 0.80 mmol). After the mixture was poured into water (50 ml), the organic layer was extracted with chloroform and dried over anhydrous magnesium sulfate. After removal of volatile substances, the residue was subjected to gel permeation chromatography to afford the title compound, 3,5-bis(trimethylsilyl)- triphenyleno[1,12-bcd]thiophene (1) (57 mg, 71%). Suitable crystals for X-ray crystallographic analysis were obtained by slow evaporation of a chloroform/ethanol solution of (1).

Refinement

H atoms attached to Csp^3 and Csp^2 carbon atoms were treated as riding with C—H distances of 0.96 and 0.93 Å, while all the other atoms were refined anisotropically.

Figures

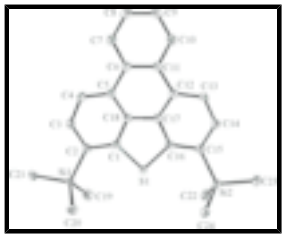


Fig. 1. Top view of the molecule of (1) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level. All H atoms are omitted for clarity.

Fig. 2. Side view of the molecule of (1). Displacement ellipsoids are drawn at the 40% probability level. All H atoms are omitted for clarity.

Fig. 3. Triphenylene derivatives with sulfur bridges, (1), (2) and (3), and Triphenylene (4).

3,5-Bis(trimethylsilyl)triphenyleno[1,12-bcd]thiophene

Crystal data

$C_{24}H_{26}SSi_2$	F_{000} = 856
M_r = 402.69	D_x = 1.263 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P2yn	Cell parameters from 7365 reflections
a = 7.0230 (3) Å	θ = 2.4–27.9º
b = 14.4933 (7) Å	μ = 0.27 mm^{-1}
c = 20.8358 (10) Å	T = 103 K
β = 92.707 (1)º	Cube, colourless
V = 2118.44 (17) Å^3	0.45 × 0.20 × 0.20 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3730 independent reflections
Radiation source: fine-focus sealed tube	3375 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.044
T = 103 K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −8→5
T_{min} = 0.937, T_{max} = 0.947	k = −17→16
12122 measured reflections	l = −24→24

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	H-atom parameters constrained
$wR(F^2) = 0.089$	$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.6989P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.001$
3730 reflections	$\Delta\rho_{max} = 0.41$ e Å^{-3}
250 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 " $F^2 > 2$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}
S1	0.78242 (6)	0.79366 (3)	0.043181 (19)	0.01977 (12)
Si1	0.78958 (7)	0.77637 (3)	0.21285 (2)	0.02029 (13)
Si2	0.81669 (7)	0.89550 (3)	−0.11203 (2)	0.02055 (13)
C18	0.7605 (2)	0.61485 (11)	0.04568 (7)	0.0181 (3)
C10	0.7419 (2)	0.40497 (12)	−0.09549 (8)	0.0236 (4)
H10	0.7478	0.4198	−0.1388	0.028*
C17	0.7684 (2)	0.63865 (11)	−0.01986 (8)	0.0178 (3)
C11	0.7501 (2)	0.47600 (11)	−0.04956 (8)	0.0196 (4)
C2	0.7662 (2)	0.67842 (12)	0.15410 (8)	0.0196 (3)
C16	0.7815 (2)	0.73368 (11)	−0.03077 (8)	0.0184 (3)
C5	0.7483 (2)	0.52327 (11)	0.06642 (8)	0.0191 (3)
C6	0.7412 (2)	0.45186 (11)	0.01646 (8)	0.0197 (4)
C12	0.7656 (2)	0.57279 (11)	−0.06884 (8)	0.0188 (3)
C8	0.7170 (3)	0.29028 (12)	−0.01351 (9)	0.0263 (4)
H8	0.7059	0.2287	−0.0017	0.032*
C13	0.7782 (2)	0.60757 (12)	−0.13114 (8)	0.0229 (4)
H13	0.7778	0.5671	−0.1658	0.028*
C1	0.7684 (2)	0.69009 (11)	0.08752 (8)	0.0178 (3)
C3	0.7531 (2)	0.58564 (12)	0.17410 (8)	0.0228 (4)
H3	0.7504	0.5741	0.2180	0.027*
C15	0.7926 (2)	0.76950 (11)	−0.09278 (8)	0.0196 (4)
C14	0.7914 (2)	0.70169 (12)	−0.14167 (8)	0.0228 (4)

H14	0.8000	0.7217	−0.1838	0.027*
C7	0.7253 (2)	0.35799 (12)	0.03256 (8)	0.0241 (4)
H7	0.7202	0.3414	0.0756	0.029*
C4	0.7440 (2)	0.51047 (12)	0.13279 (8)	0.0231 (4)
H4	0.7350	0.4512	0.1494	0.028*
C21	0.7877 (3)	0.73179 (13)	0.29661 (8)	0.0274 (4)
H21A	0.8391	0.7777	0.3258	0.041*
H21B	0.8638	0.6769	0.3003	0.041*
H21C	0.6591	0.7178	0.3070	0.041*
C22	1.0565 (3)	0.93544 (13)	−0.08124 (9)	0.0279 (4)
H22A	1.0699	1.0001	−0.0899	0.042*
H22B	1.1534	0.9017	−0.1022	0.042*
H22C	1.0695	0.9251	−0.0357	0.042*
C19	1.0212 (3)	0.83549 (14)	0.20191 (9)	0.0319 (4)
H19A	1.0347	0.8861	0.2315	0.048*
H19B	1.0247	0.8582	0.1587	0.048*
H19C	1.1238	0.7926	0.2099	0.048*
C9	0.7252 (2)	0.31397 (12)	−0.07794 (9)	0.0263 (4)
H9	0.7194	0.2682	−0.1093	0.032*
C23	0.7884 (3)	0.91238 (13)	−0.20085 (8)	0.0295 (4)
H23A	0.7840	0.9772	−0.2104	0.044*
H23B	0.6723	0.8837	−0.2167	0.044*
H23C	0.8945	0.8849	−0.2211	0.044*
C20	0.5925 (3)	0.86107 (14)	0.19876 (9)	0.0346 (5)
H20A	0.4723	0.8306	0.2027	0.052*
H20B	0.5986	0.8865	0.1564	0.052*
H20C	0.6051	0.9097	0.2299	0.052*
C24	0.6303 (3)	0.96322 (13)	−0.07238 (9)	0.0318 (4)
H24A	0.6375	0.9509	−0.0271	0.048*
H24B	0.5067	0.9458	−0.0900	0.048*
H24C	0.6504	1.0278	−0.0796	0.048*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0259 (2)	0.0170 (2)	0.0164 (2)	−0.00113 (16)	0.00125 (17)	−0.00041 (15)
Si1	0.0239 (3)	0.0207 (3)	0.0163 (2)	−0.00040 (19)	0.00071 (19)	−0.00153 (17)
Si2	0.0216 (3)	0.0208 (3)	0.0192 (2)	−0.00127 (19)	0.00036 (18)	0.00232 (18)
C18	0.0153 (8)	0.0209 (9)	0.0182 (8)	0.0008 (7)	0.0005 (6)	0.0001 (6)
C10	0.0214 (9)	0.0246 (9)	0.0250 (9)	−0.0003 (7)	0.0019 (7)	−0.0038 (7)
C17	0.0136 (8)	0.0208 (8)	0.0188 (8)	0.0005 (6)	0.0003 (6)	−0.0003 (7)
C11	0.0143 (8)	0.0218 (9)	0.0227 (9)	0.0008 (7)	0.0004 (6)	−0.0021 (7)
C2	0.0181 (8)	0.0226 (8)	0.0182 (8)	0.0001 (7)	0.0008 (6)	−0.0010 (7)
C16	0.0153 (8)	0.0200 (8)	0.0198 (8)	−0.0008 (7)	0.0004 (6)	−0.0019 (7)
C5	0.0158 (8)	0.0199 (8)	0.0216 (8)	0.0009 (7)	−0.0002 (6)	0.0005 (6)
C6	0.0154 (8)	0.0205 (8)	0.0232 (9)	0.0007 (7)	−0.0005 (6)	−0.0018 (7)
C12	0.0147 (8)	0.0214 (8)	0.0203 (8)	−0.0008 (7)	0.0003 (6)	−0.0021 (7)
C8	0.0238 (9)	0.0172 (9)	0.0377 (11)	0.0005 (7)	−0.0009 (8)	−0.0011 (7)

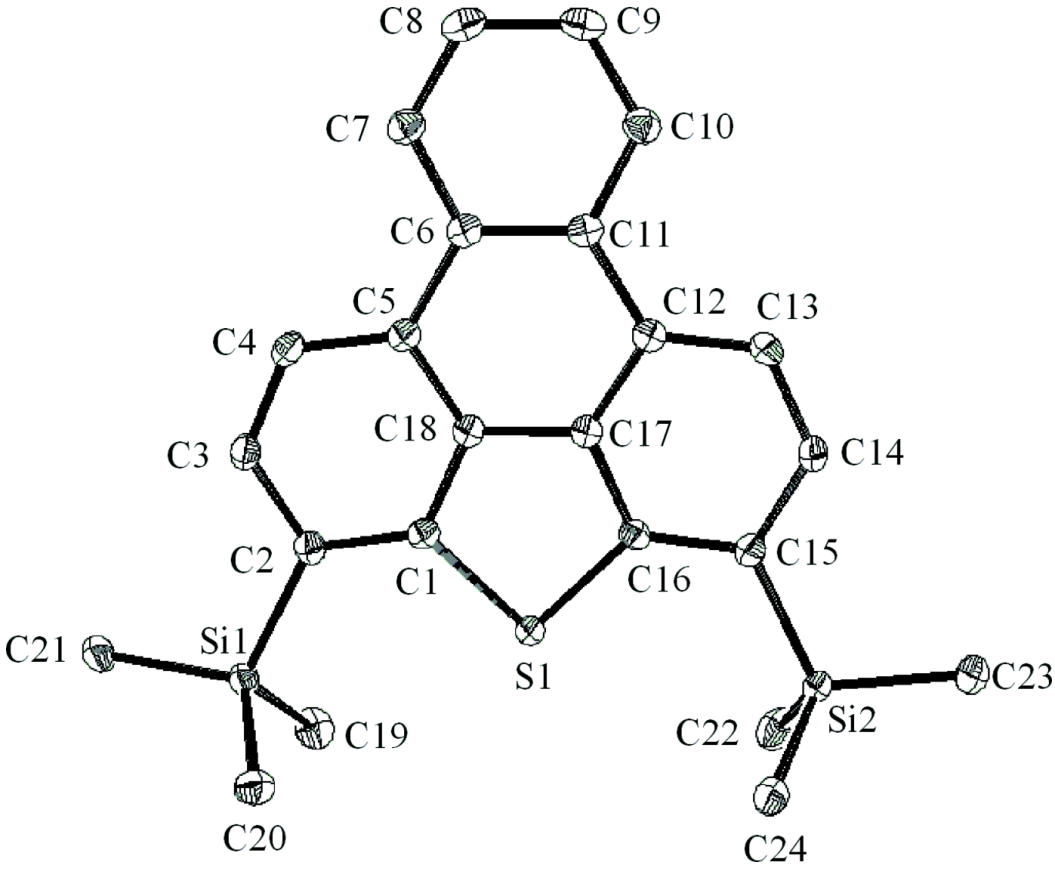
C13	0.0239 (9)	0.0264 (9)	0.0186 (8)	−0.0013 (7)	0.0016 (7)	−0.0054 (7)
C1	0.0161 (8)	0.0170 (8)	0.0203 (8)	−0.0005 (6)	0.0004 (6)	0.0016 (6)
C3	0.0268 (9)	0.0244 (9)	0.0171 (8)	0.0003 (7)	0.0004 (7)	0.0027 (7)
C15	0.0159 (8)	0.0233 (9)	0.0195 (8)	−0.0010 (7)	0.0008 (6)	0.0020 (7)
C14	0.0239 (9)	0.0277 (9)	0.0170 (8)	−0.0014 (7)	0.0015 (7)	0.0010 (7)
C7	0.0241 (9)	0.0220 (9)	0.0259 (9)	0.0015 (7)	−0.0012 (7)	0.0019 (7)
C4	0.0280 (9)	0.0176 (8)	0.0235 (9)	−0.0003 (7)	0.0003 (7)	0.0039 (7)
C21	0.0328 (10)	0.0300 (10)	0.0198 (9)	−0.0001 (8)	0.0044 (7)	−0.0019 (7)
C22	0.0274 (9)	0.0263 (9)	0.0298 (10)	−0.0037 (8)	−0.0007 (8)	−0.0024 (7)
C19	0.0382 (11)	0.0361 (11)	0.0213 (9)	−0.0127 (9)	−0.0004 (8)	−0.0002 (8)
C9	0.0234 (9)	0.0233 (9)	0.0321 (10)	0.0012 (7)	0.0004 (7)	−0.0095 (8)
C23	0.0370 (10)	0.0286 (10)	0.0226 (9)	−0.0074 (8)	−0.0014 (8)	0.0061 (7)
C20	0.0404 (11)	0.0328 (10)	0.0302 (10)	0.0107 (9)	−0.0020 (9)	−0.0056 (8)
C24	0.0314 (10)	0.0301 (10)	0.0342 (10)	0.0076 (8)	0.0035 (8)	0.0040 (8)

Geometric parameters (Å, °)

S1—C1	1.7678 (16)	C8—H8	0.9300
S1—C16	1.7688 (16)	C13—C14	1.385 (2)
Si1—C21	1.8617 (18)	C13—H13	0.9300
Si1—C19	1.8622 (19)	C3—C4	1.388 (2)
Si1—C20	1.8629 (19)	C3—H3	0.9300
Si1—C2	1.8766 (17)	C15—C14	1.415 (2)
Si2—C24	1.8607 (19)	C14—H14	0.9300
Si2—C22	1.8654 (18)	C7—H7	0.9300
Si2—C23	1.8680 (18)	C4—H4	0.9300
Si2—C15	1.8790 (17)	C21—H21A	0.9600
C18—C1	1.395 (2)	C21—H21B	0.9600
C18—C5	1.400 (2)	C21—H21C	0.9600
C18—C17	1.412 (2)	C22—H22A	0.9600
C10—C9	1.375 (2)	C22—H22B	0.9600
C10—C11	1.405 (2)	C22—H22C	0.9600
C10—H10	0.9300	C19—H19A	0.9600
C17—C12	1.397 (2)	C19—H19B	0.9600
C17—C16	1.400 (2)	C19—H19C	0.9600
C11—C6	1.424 (2)	C9—H9	0.9300
C11—C12	1.465 (2)	C23—H23A	0.9600
C2—C1	1.398 (2)	C23—H23B	0.9600
C2—C3	1.412 (2)	C23—H23C	0.9600
C16—C15	1.398 (2)	C20—H20A	0.9600
C5—C4	1.397 (2)	C20—H20B	0.9600
C5—C6	1.467 (2)	C20—H20C	0.9600
C6—C7	1.407 (2)	C24—H24A	0.9600
C12—C13	1.399 (2)	C24—H24B	0.9600
C8—C7	1.372 (2)	C24—H24C	0.9600
C8—C9	1.389 (3)		
C1—S1—C16	92.28 (8)	C16—C15—C14	114.12 (15)
C21—Si1—C19	108.62 (9)	C16—C15—Si2	124.59 (13)
C21—Si1—C20	109.77 (9)	C14—C15—Si2	121.25 (12)

C19—Si1—C20	108.93 (10)	C13—C14—C15	124.61 (16)
C21—Si1—C2	110.16 (8)	C13—C14—H14	117.7
C19—Si1—C2	108.56 (8)	C15—C14—H14	117.7
C20—Si1—C2	110.75 (8)	C8—C7—C6	121.73 (16)
C24—Si2—C22	109.05 (9)	C8—C7—H7	119.1
C24—Si2—C23	109.07 (9)	C6—C7—H7	119.1
C22—Si2—C23	110.61 (9)	C3—C4—C5	120.46 (15)
C24—Si2—C15	110.21 (8)	C3—C4—H4	119.8
C22—Si2—C15	108.49 (8)	C5—C4—H4	119.8
C23—Si2—C15	109.41 (8)	Si1—C21—H21A	109.5
C1—C18—C5	123.31 (15)	Si1—C21—H21B	109.5
C1—C18—C17	114.27 (15)	H21A—C21—H21B	109.5
C5—C18—C17	122.41 (15)	Si1—C21—H21C	109.5
C9—C10—C11	121.54 (16)	H21A—C21—H21C	109.5
C9—C10—H10	119.2	H21B—C21—H21C	109.5
C11—C10—H10	119.2	Si2—C22—H22A	109.5
C12—C17—C16	123.55 (15)	Si2—C22—H22B	109.5
C12—C17—C18	122.67 (15)	H22A—C22—H22B	109.5
C16—C17—C18	113.78 (14)	Si2—C22—H22C	109.5
C10—C11—C6	118.41 (15)	H22A—C22—H22C	109.5
C10—C11—C12	121.09 (15)	H22B—C22—H22C	109.5
C6—C11—C12	120.50 (15)	Si1—C19—H19A	109.5
C1—C2—C3	114.32 (15)	Si1—C19—H19B	109.5
C1—C2—Si1	123.43 (12)	H19A—C19—H19B	109.5
C3—C2—Si1	122.20 (12)	Si1—C19—H19C	109.5
C15—C16—C17	121.49 (15)	H19A—C19—H19C	109.5
C15—C16—S1	128.64 (13)	H19B—C19—H19C	109.5
C17—C16—S1	109.86 (12)	C10—C9—C8	120.12 (16)
C4—C5—C18	115.85 (15)	C10—C9—H9	119.9
C4—C5—C6	127.38 (15)	C8—C9—H9	119.9
C18—C5—C6	116.76 (14)	Si2—C23—H23A	109.5
C7—C6—C11	118.40 (15)	Si2—C23—H23B	109.5
C7—C6—C5	120.88 (15)	H23A—C23—H23B	109.5
C11—C6—C5	120.71 (15)	Si2—C23—H23C	109.5
C17—C12—C13	115.61 (15)	H23A—C23—H23C	109.5
C17—C12—C11	116.93 (15)	H23B—C23—H23C	109.5
C13—C12—C11	127.45 (15)	Si1—C20—H20A	109.5
C7—C8—C9	119.80 (16)	Si1—C20—H20B	109.5
C7—C8—H8	120.1	H20A—C20—H20B	109.5
C9—C8—H8	120.1	Si1—C20—H20C	109.5
C14—C13—C12	120.61 (16)	H20A—C20—H20C	109.5
C14—C13—H13	119.7	H20B—C20—H20C	109.5
C12—C13—H13	119.7	Si2—C24—H24A	109.5
C18—C1—C2	121.56 (15)	Si2—C24—H24B	109.5
C18—C1—S1	109.79 (12)	H24A—C24—H24B	109.5
C2—C1—S1	128.65 (13)	Si2—C24—H24C	109.5
C4—C3—C2	124.49 (15)	H24A—C24—H24C	109.5
C4—C3—H3	117.8	H24B—C24—H24C	109.5
C2—C3—H3	117.8		

Fig. 1


C8
C9
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Si1
S1
C23
C19
C22
Si2
C20
C24

Fig. 2

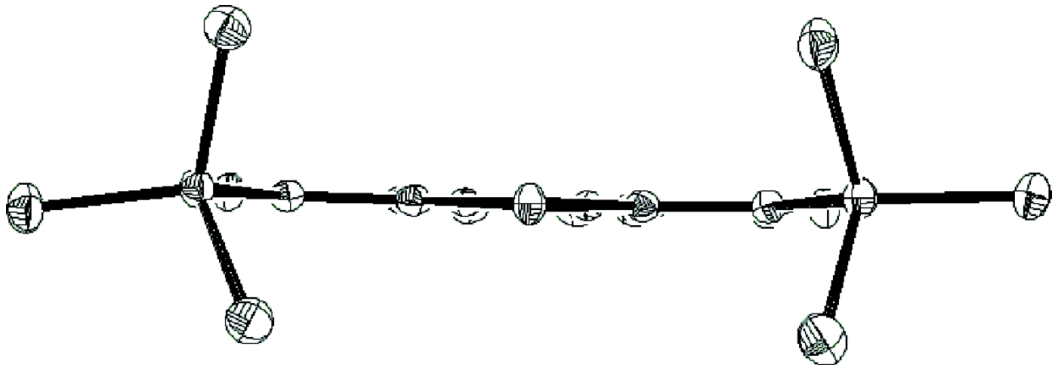
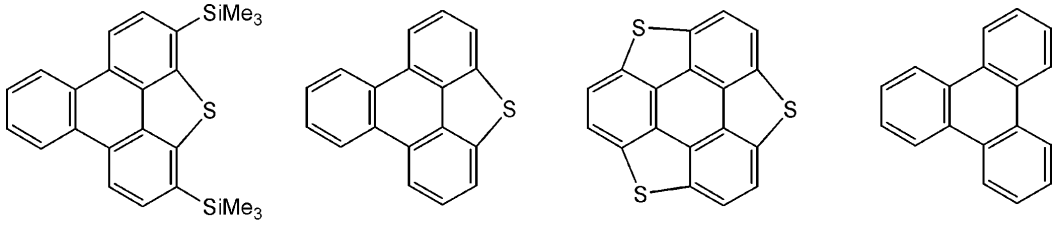


Fig. 3



1 **2** **3** **4**