Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Non-merohedral twin crystal of 2,3,9,10-tetrakis(triisopropylsilylethynyl)-6,13-bis(trimethylsilylethynyl)pentacene[1]

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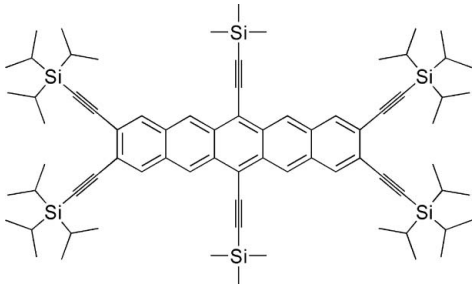
Received 5 July 2007; accepted 6 August 2007

Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.010 Å; disorder in main residue; R factor = 0.053; wR factor = 0.154; data-to-parameter ratio = 20.2.

All investigated crystals of the title pentacene derivative, $C_{76}H_{110}Si_6$, obtained from benzene solution and examined at 120 K, were identified as non-merohedral twins. The crystal examined in this study had an approximate 2:1 domain ratio. The molecules have crystallographic inversion symmetry and approximate C_{2h} symmetry, with a planar conjugated central core, including the peripheral C≡C triple bonds. The molecules pack in layers perpendicular to the a axis, with a dihedral angle between individual molecules in adjacent layers of 5.25 (2)°; π–π stacking is prevented by the bulky substituents. One methyl group is disordered over two positions with occupancies of 0.6 and 0.4.

Related literature

For related literature, see: Dimitrakopoulos *et al.* (1998); Klauk *et al.* (2000); Nelson *et al.* (1998); Wurthner (2001); Holmes *et al.* (1999); Jiang *et al.* (2006); Desiraju & Gavezzotti (1989). For related structures, see: Campbell *et al.* (1962); Mattheus *et al.* (2001); Houk *et al.* (2001); Fokin *et al.* (1998). For twinning analyis tools, see: Bruker (1998, 2000).



Experimental

Crystal data

$C_{76}H_{110}Si_6$
M_r = 1192.18
Monoclinic, $P2_1/n$
a = 10.6492 (12) Å
b = 19.1865 (18) Å
c = 18.6632 (14) Å
β = 91.236 (4)°
V = 3812.4 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 120 (2) K
0.26 × 0.25 × 0.25 mm

Data collection

Bruker SMART 6000 diffractometer
Absorption correction: multi-scan (*TWINABS*; Sheldrick, 1999)
T_{min} = 0.718, T_{max} = 1.000
(expected range = 0.692–0.964)
26858 measured reflections
7479 independent reflections
6159 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.154
S = 1.32
7479 reflections
370 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.64 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2005); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXTL*.

This work was supported by the Office of Naval Research (grant No. N00014-05-1-0372). The crystallographic facility of the Ohio Crystallography Consortium located at the University of Toledo was established with grants from the Ohio Board of Regents and a Naval Research grant given to Professor A. Alan Pinkerton. BRK thanks the University Research Board of the American University of Beirut for a Faculty Development Fund. The authors gratefully acknowledge Professor A. Alan Pinkerton for many fruitful crystallographic discussions and thank Professor Brigitte Wex for her assistance in preparing the manuscript.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2122).

References

Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Version 3.1. Crystal Impact GbR, Bonn, Germany.
Bruker (1998). *RLATT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2000). *GEMINI*. Version 1.02. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2003). *SMART* (Version 5.630) and *SAINT* (Version 6.45). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *SHELXTL*. Version 6.14. Bruker AXS Inc., Madison, Wisconsin, USA.
Campbell, R. B., Robertson, J. M. & Trotter, J. (1962). *Acta Cryst.* **15**, 289–290.
Desiraju, G. R. & Gavezzotti, A. (1989). *Acta Cryst.* B**45**, 473–482.
Dimitrakopoulos, C. D., Furman, B. K., Graham, T., Hedge, S. & Purushothaman, S. (1998). *Synth. Met.* **92**, 47–52.
Fokin, A. A., Jiao, H. & von Schleyer, P. R. (1998). *J. Am. Chem. Soc.* **120**, 9364–9365.

[1] Contribution No. 615 from the Center for Photochemical Sciences.

  doi:10.1107/S1600536807038664

Holmes, D., Kumaraswamy, S., Matzger, A. J. & Vollhardt, K. P. C. (1999). *Chem. Eur. J.* **5**, 3399–3412.
Houk, K. N., Lee, P. S. & Nendel, M. (2001). *J. Org. Chem.* **66**, 5517–5521.
Jiang, J., Kaafarani, B. R. & Neckers, D. C. (2006). *J. Org. Chem.* **71**, 2155–2158.
Klauk, H., Gundlach, D. J., Nichols, J. A., Sheraw, C. D., Bonse, M. & Jackson, T. N. (2000). *Solid State Technol.* **43**, 63–64, 66–67, 72, 75–76.
Mattheus, C. C., Dros, A. B., Baas, J., Meetsma, A., Boer, J. L. de & Palstra, T. T. M. (2001). *Acta Cryst.* C**57**, 939–941.
Nelson, S. F., Lin, Y. Y., Gundlach, D. J. & Jackson, T. N. (1998). *Appl. Phys. Lett.* **72**, 1854–1856.
Sheldrick, G. M. (1999). *TWINABS*. University of Göttingen, Germany.
Wurthner, F. (2001). *Angew. Chem. Int. Ed.* **40**, 1037–1039.

supplementary materials

Acta Cryst. (2007). E**63**, o3760-o3761 [**doi:10.1107/S1600536807038664**]

Non-merohedral twin crystal of 2,3,9,10-tetrakis(triisopropylsilylethynyl)-6,13-bis(trimethylsilylethynyl)pentacene

J. Jiang, B. R. Kaafarani, K. Kirschbaum, Y. Hu and D. C. Neckers

Comment

The practical uses of pentacene, the most promising polyacene in the area of organic field-effect transistors (Dimitrakopoulos *et al.*, 1998; Klauk *et al.*, 2000; Nelson *et al.*, 1998; Wurthner, 2001), are limited by its sensitivity to oxygen, poor solubility in organic solvents and herringbone packing in the solid state (Holmes *et al.*, 1999). To overcome these disadvantages, we reported new ethynylated pentacenes (Jiang *et al.*, 2006). In this paper, we present the crystal structure of the title compound (I)·The molecular structure of (I) is shown in Figure 1. The X-ray crystal structure of (I) confirms its chemical structure ($C_{76}H_{110}Si_6$). The five fused benzene rings and the six carbon-carbon triple bonds are coplanar within ±0.14 (1) Å. In the pentacene core of (I), the bond lengths and their alternation are very similar to those in pentacene itself (Campbell *et al.*, 1962; Mattheus *et al.*, 2001). The cross-ring aromatic bonds are consistently longer than the peripheral aromatic bonds. The former average 1.445 (8) Å in length, and the latter 1.401 (8) Å, suggesting that the peripheral bonds form two parallel, delocalized polyacetylenic ribbons as previously reported (Houk *et al.*, 2001; Fokin *et al.*, 1998). The 'single' bonds between 'triple' bonds and the pentacene ring, for example, C1—C12 (1.425 (8) Å), C6—C17 (1.433 (8) Å) and C7—C28 (1.433 (8) Å), are significantly shorter than typical carbon-carbon single bonds, indicating the extended conjugation. The triple bond lengths average 1.20 Å.

The bond angles C17—C18—Si2 and C28—C29—Si3 are 173.4 (6)° and 176.7 (7)°, respectively, indicating that the steric crowding of terminal triisopropylsilyl (TIPS) moieties is released by bending. The two TIPS at the same end also contribute to this release by assuming different conformations. As required by the crystallographic inversion center in the middle of the molecule, the two trimethylsilyl groups at C13 and C13' (= 2 − *x*,-*y*,2 − *z*) assume a staggered conformation.

The molecules of (I) pack in layers perpendicular to the *a* axis (Figure 2). The dihedral angle between adjacent layers is 5.25 (2)° and alternate layers are parallel to each other as required by the translational symmetry. In contrast to the packing of pentacene, no herringbone packing exists (Holmes *et al.*, 1999; Desiraju *et al.*, 1989). The bulky substituents prevent π–π stacking.

Experimental

The synthesis is described by Jiang *et al.* (2006).

Refinement

As for several other crystals, the one used for data collection was identified as a non-merohedral twin using RLATT (Bruker, 1998). Two orientation matrices were assigned to the two different twin components (GEMINI; Bruker, 2000). Integration of the data using both orientation matrices deconvoluted the data set into overlapped reflections and reflections belonging to only one of the twin components. Corrections for absorption, decay and inhomogeneity of the X-ray beam were applied using TWINABS (Sheldrick, 1999). The twin law is a 180° rotation about the c* reciprocal axis; the ratio of the two twin

components was refined to 0.363:0.637 (1). The disordered carbon atom C15 was refined isotropically; the occupancy factors were initially refined with a common displacement parameter, and then fixed at 0.6:0.4 for the final refinement. The hydrogen atoms were positioned geometrically and included in the refinement as riding atoms, with C—H = 0.95–1.00 Å and U_{iso}(H) = U_{eq}(C). The non-merohedral twinning prevents the complete merging of equivalent reflections before refinement. A table of structure factors including the calculated contributions from the two twin components, obtained by the undocumented LIST 7 instruction of *SHELXTL*, is provided in the Supplementary Material.

Figures

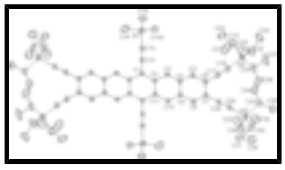


Fig. 1. The molecular structure of (I) at 120 K, showing the atomic labeling and 50% probability ellipsoids.

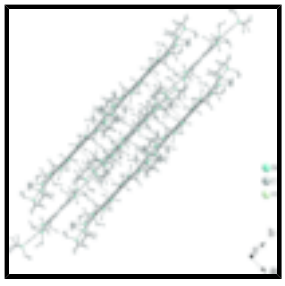


Fig. 2. A packing diagram of (I) viewed down the *c* axis. Hydrogen atoms are omitted for clarity.

2,3,9,10-Tetrakis(triisopropylsilylethynyl)- 6,13-bis(trimethylsilylethynyl)pentacene

Crystal data

$C_{76}H_{110}Si_6$	F_{000} = 1300
M_r = 1192.18	D_x = 1.039 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 10.6492 (12) Å	Cell parameters from 1328 reflections
b = 19.1865 (18) Å	θ = 2.2–30.1º
c = 18.6632 (14) Å	μ = 0.15 mm^{-1}
β = 91.236 (4)º	T = 120 (2) K
V = 3812.4 (6) Å^3	Cuboid, very dark turquoise
Z = 2	0.26 × 0.25 × 0.25 mm

Data collection

Bruker SMART 6000 diffractometer	7479 independent reflections
Radiation source: fine-focus sealed tube	6159 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.044
T = 120(2) K	θ_{max} = 26.0º
ω scans	θ_{min} = 1.5º
Absorption correction: multi-scan	h = −13→13

(TWINABS; Sheldrick, 1999)

T_{min} = 0.718, T_{max} = 1.000

26858 measured reflections

k = 0→23

l = 0→23

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.053

$wR(F^2)$ = 0.154

S = 1.32

7479 reflections

370 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

$w = 1/[\sigma^2(F_o^2) + (0.16P)^2 + 15.6P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max}$ = 0.64 e Å^{-3}

$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Extinction correction: none

Special details

Experimental. As several crystals before, this crystal was identified as a non-merohedral twin using RLATT. Two orientation matrices were assigned to the two different twin components (GEMINI 1.02). Integration of the data with *SAINT* 6.45 A using both orientation matrices deconvoluted the data set into overlapped reflections and reflections originated by only one of the twin components. Correction for absorption, decay and inhomogeneity of the X-ray beam were applied using TWINABS. The twinning law is a 180 degree rotation around c*, the ratio of the two twin components was refined to 0.363 (1):0.637 (1).

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>2sigma(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}	Occ. (<1)
Si1	0.9658 (2)	0.17271 (10)	0.73900 (10)	0.0343 (5)	
Si2	0.96038 (17)	−0.31575 (9)	0.55599 (9)	0.0268 (4)	
Si3	1.0477 (2)	−0.51484 (9)	0.82961 (11)	0.0353 (5)	
C1	0.9887 (5)	0.0421 (3)	0.9372 (3)	0.0187 (11)	
C2	0.9908 (5)	−0.0317 (3)	0.9308 (3)	0.0176 (11)	
C3	0.9825 (6)	−0.0645 (3)	0.8638 (3)	0.0201 (12)	
H3A	0.9735	−0.0367	0.8218	0.024*	
C4	0.9872 (5)	−0.1363 (3)	0.8570 (3)	0.0203 (12)	
C5	0.9799 (6)	−0.1697 (3)	0.7887 (3)	0.0229 (12)	
H5A	0.9706	−0.1420	0.7468	0.027*	

C6	0.9859 (6)	−0.2407 (3)	0.7819 (3)	0.0232 (12)	
C7	0.9996 (6)	−0.2838 (3)	0.8454 (3)	0.0230 (12)	
C8	1.0047 (6)	−0.2527 (3)	0.9114 (3)	0.0222 (12)	
H8A	1.0119	−0.2813	0.9528	0.027*	
C9	0.9996 (5)	−0.1789 (3)	0.9201 (3)	0.0197 (11)	
C10	1.0071 (5)	−0.1474 (3)	0.9866 (3)	0.0189 (12)	
H10A	1.0150	−0.1758	1.0282	0.023*	
C11	1.0033 (5)	−0.0743 (3)	0.9945 (3)	0.0180 (11)	
C12	0.9787 (6)	0.0835 (3)	0.8740 (3)	0.0215 (12)	
C13	0.9723 (6)	0.1179 (3)	0.8200 (3)	0.0269 (13)	
C14	1.1282 (8)	0.1937 (5)	0.7130 (5)	0.050 (2)	
H14A	1.1667	0.2249	0.7487	0.076*	
H14B	1.1266	0.2166	0.6660	0.076*	
H14C	1.1774	0.1506	0.7104	0.076*	
C15A	0.8610 (14)	0.1311 (8)	0.6700 (8)	0.042 (3)*	0.60
H15A	0.7767	0.1513	0.6725	0.063*	0.60
H15B	0.8565	0.0809	0.6790	0.063*	0.60
H15C	0.8947	0.1393	0.6223	0.063*	0.60
C15B	0.913 (2)	0.1141 (11)	0.6637 (11)	0.043 (5)*	0.40
H15D	0.9838	0.0852	0.6488	0.064*	0.40
H15E	0.8832	0.1424	0.6231	0.064*	0.40
H15F	0.8450	0.0840	0.6799	0.064*	0.40
C16	0.8795 (9)	0.2531 (5)	0.7634 (6)	0.064 (3)	
H16A	0.9225	0.2758	0.8040	0.096*	
H16B	0.7937	0.2409	0.7766	0.096*	
H16C	0.8767	0.2849	0.7223	0.096*	
C17	0.9778 (6)	−0.2711 (3)	0.7118 (3)	0.0264 (13)	
C18	0.9685 (6)	−0.2927 (3)	0.6515 (3)	0.0282 (14)	
C19	0.9126 (9)	−0.2335 (5)	0.5079 (4)	0.051 (2)	
H19A	0.9156	−0.2428	0.4552	0.062*	
C20	1.0012 (11)	−0.1723 (5)	0.5249 (6)	0.070 (3)	
H20A	0.9721	−0.1307	0.4989	0.105*	
H20B	1.0864	−0.1841	0.5102	0.105*	
H20C	1.0016	−0.1629	0.5765	0.105*	
C21	0.7793 (10)	−0.2129 (5)	0.5252 (8)	0.085 (4)	
H21A	0.7571	−0.1699	0.4996	0.128*	
H21B	0.7731	−0.2053	0.5769	0.128*	
H21C	0.7216	−0.2502	0.5102	0.128*	
C22	1.1217 (7)	−0.3480 (5)	0.5310 (5)	0.050 (2)	
H22A	1.1207	−0.3992	0.5410	0.060*	
C23	1.2276 (9)	−0.3191 (7)	0.5779 (6)	0.075 (3)	
H23A	1.3079	−0.3379	0.5620	0.113*	
H23B	1.2146	−0.3325	0.6278	0.113*	
H23C	1.2287	−0.2681	0.5741	0.113*	
C24	1.1454 (10)	−0.3417 (6)	0.4502 (5)	0.067 (3)	
H24A	1.2297	−0.3590	0.4400	0.101*	
H24B	1.1388	−0.2927	0.4358	0.101*	
H24C	1.0828	−0.3692	0.4234	0.101*	
C25	0.8394 (7)	−0.3864 (4)	0.5443 (4)	0.0378 (17)	

H25A	0.7568	−0.3660	0.5578	0.045*
C26	0.8649 (9)	−0.4476 (5)	0.5946 (7)	0.064 (3)
H26A	0.8002	−0.4833	0.5869	0.096*
H26B	0.8633	−0.4314	0.6443	0.096*
H26C	0.9476	−0.4675	0.5848	0.096*
C27	0.8254 (8)	−0.4107 (6)	0.4669 (6)	0.070 (3)
H27A	0.7617	−0.4475	0.4635	0.105*
H27B	0.9060	−0.4289	0.4507	0.105*
H27C	0.7996	−0.3713	0.4365	0.105*
C28	1.0113 (6)	−0.3579 (3)	0.8384 (3)	0.0269 (14)
C29	1.0234 (7)	−0.4199 (3)	0.8331 (4)	0.0328 (15)
C30	0.8883 (8)	−0.5580 (4)	0.8309 (5)	0.046 (2)
H30A	0.9024	−0.6095	0.8338	0.055*
C31	0.8160 (9)	−0.5367 (5)	0.8975 (5)	0.053 (2)
H31A	0.7337	−0.5596	0.8967	0.080*
H31B	0.8045	−0.4860	0.8977	0.080*
H31C	0.8634	−0.5508	0.9406	0.080*
C32	0.8074 (10)	−0.5441 (6)	0.7641 (6)	0.066 (3)
H32A	0.7264	−0.5677	0.7685	0.099*
H32B	0.8502	−0.5617	0.7218	0.099*
H32C	0.7935	−0.4938	0.7590	0.099*
C33	1.1273 (10)	−0.5363 (4)	0.7429 (5)	0.054 (2)
H33A	1.0588	−0.5356	0.7054	0.065*
C34	1.2203 (11)	−0.4849 (5)	0.7188 (6)	0.076 (4)
H34A	1.2562	−0.5006	0.6737	0.113*
H34B	1.2874	−0.4802	0.7553	0.113*
H34C	1.1792	−0.4397	0.7115	0.113*
C35	1.1790 (10)	−0.6106 (4)	0.7422 (6)	0.061 (3)
H35A	1.2196	−0.6194	0.6964	0.092*
H35B	1.1099	−0.6437	0.7481	0.092*
H35C	1.2405	−0.6164	0.7815	0.092*
C36	1.1365 (8)	−0.5365 (4)	0.9154 (5)	0.0458 (19)
H36A	1.0913	−0.5118	0.9543	0.055*
C37	1.2701 (9)	−0.5080 (6)	0.9180 (6)	0.065 (3)
H37A	1.3108	−0.5209	0.9637	0.098*
H37B	1.2678	−0.4572	0.9136	0.098*
H37C	1.3175	−0.5278	0.8784	0.098*
C38	1.1356 (11)	−0.6140 (5)	0.9360 (6)	0.070 (3)
H38A	1.1835	−0.6206	0.9809	0.106*
H38B	1.1740	−0.6414	0.8979	0.106*
H38C	1.0488	−0.6294	0.9422	0.106*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0557 (13)	0.0245 (9)	0.0226 (9)	−0.0043 (8)	−0.0026 (8)	0.0091 (7)
Si2	0.0293 (9)	0.0326 (9)	0.0183 (8)	0.0021 (7)	−0.0003 (7)	−0.0083 (7)
Si3	0.0578 (13)	0.0169 (8)	0.0316 (10)	0.0026 (8)	0.0103 (9)	−0.0032 (7)

C1	0.022 (3)	0.016 (3)	0.018 (3)	0.001 (2)	0.002 (2)	0.000 (2)
C2	0.021 (3)	0.016 (3)	0.017 (3)	0.001 (2)	0.001 (2)	−0.002 (2)
C3	0.027 (3)	0.018 (3)	0.015 (3)	0.000 (2)	0.002 (2)	0.000 (2)
C4	0.024 (3)	0.019 (3)	0.018 (3)	0.000 (2)	−0.001 (2)	−0.003 (2)
C5	0.029 (3)	0.022 (3)	0.017 (3)	0.002 (2)	−0.001 (2)	−0.003 (2)
C6	0.027 (3)	0.023 (3)	0.020 (3)	0.000 (2)	−0.002 (2)	−0.005 (2)
C7	0.028 (3)	0.017 (3)	0.024 (3)	−0.002 (2)	0.000 (2)	−0.004 (2)
C8	0.030 (3)	0.018 (3)	0.019 (3)	0.000 (2)	0.001 (2)	−0.001 (2)
C9	0.023 (3)	0.017 (3)	0.019 (3)	0.001 (2)	0.002 (2)	−0.002 (2)
C10	0.023 (3)	0.017 (3)	0.017 (3)	0.001 (2)	0.000 (2)	0.001 (2)
C11	0.020 (3)	0.017 (3)	0.016 (3)	0.000 (2)	0.001 (2)	−0.001 (2)
C12	0.028 (3)	0.017 (3)	0.020 (3)	0.001 (2)	0.001 (2)	−0.005 (2)
C13	0.042 (4)	0.019 (3)	0.019 (3)	0.001 (3)	0.002 (3)	0.000 (2)
C14	0.066 (6)	0.045 (5)	0.041 (5)	0.009 (4)	0.014 (4)	0.018 (4)
C16	0.061 (6)	0.040 (5)	0.091 (8)	0.014 (4)	0.018 (5)	0.023 (5)
C17	0.033 (4)	0.021 (3)	0.025 (3)	0.000 (3)	0.000 (3)	−0.005 (2)
C18	0.035 (4)	0.025 (3)	0.024 (3)	0.002 (3)	−0.004 (3)	−0.006 (2)
C19	0.076 (6)	0.048 (5)	0.030 (4)	0.008 (4)	−0.013 (4)	0.002 (4)
C20	0.085 (8)	0.048 (5)	0.076 (7)	−0.004 (5)	0.014 (6)	0.020 (5)
C21	0.063 (7)	0.042 (6)	0.149 (12)	0.007 (5)	−0.042 (7)	0.007 (7)
C22	0.036 (4)	0.070 (6)	0.045 (5)	−0.001 (4)	0.010 (3)	−0.021 (4)
C23	0.042 (5)	0.117 (10)	0.067 (7)	0.005 (6)	0.004 (4)	−0.008 (7)
C24	0.063 (6)	0.091 (8)	0.049 (6)	−0.008 (5)	0.027 (5)	−0.025 (5)
C25	0.027 (4)	0.043 (4)	0.044 (4)	−0.001 (3)	0.001 (3)	−0.022 (3)
C26	0.050 (6)	0.037 (5)	0.105 (9)	−0.010 (4)	0.006 (5)	−0.007 (5)
C27	0.035 (5)	0.106 (8)	0.069 (7)	−0.006 (5)	0.002 (4)	−0.066 (6)
C28	0.036 (4)	0.023 (3)	0.022 (3)	−0.001 (3)	0.002 (3)	−0.006 (2)
C29	0.047 (4)	0.023 (3)	0.029 (3)	0.000 (3)	0.007 (3)	−0.006 (3)
C30	0.066 (6)	0.025 (4)	0.047 (5)	−0.006 (3)	−0.003 (4)	−0.002 (3)
C31	0.057 (6)	0.048 (5)	0.056 (6)	−0.011 (4)	0.005 (4)	0.007 (4)
C32	0.075 (7)	0.069 (7)	0.053 (6)	−0.013 (5)	−0.012 (5)	−0.005 (5)
C33	0.086 (7)	0.030 (4)	0.046 (5)	0.003 (4)	0.023 (4)	−0.008 (4)
C34	0.103 (9)	0.048 (6)	0.079 (8)	−0.018 (5)	0.058 (6)	−0.018 (5)
C35	0.087 (7)	0.034 (5)	0.063 (6)	0.013 (4)	0.029 (5)	−0.013 (4)
C36	0.054 (5)	0.039 (5)	0.044 (5)	0.007 (4)	0.005 (4)	0.002 (4)
C37	0.050 (6)	0.076 (7)	0.069 (7)	0.008 (5)	0.002 (4)	−0.003 (6)
C38	0.093 (8)	0.051 (6)	0.067 (7)	0.014 (5)	−0.007 (5)	0.024 (5)

Geometric parameters (Å, °)

Si1—C13	1.841 (6)	C20—H20B	0.980
Si1—C14	1.851 (9)	C20—H20C	0.980
Si1—C16	1.856 (9)	C21—H21A	0.980
Si1—C15A	1.865 (14)	C21—H21B	0.980
Si1—C15B	1.88 (2)	C21—H21C	0.980
Si2—C18	1.837 (6)	C22—C23	1.518 (13)
Si2—C25	1.880 (7)	C22—C24	1.540 (12)
Si2—C19	1.880 (9)	C22—H22A	1.00
Si2—C22	1.893 (8)	C23—H23A	0.980

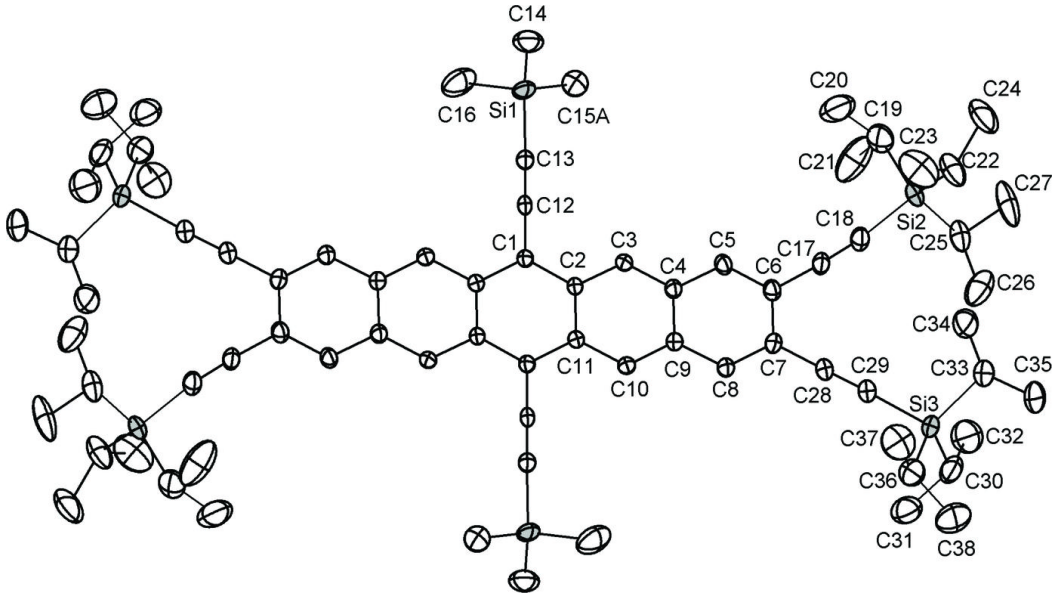
Si3—C29	1.841 (7)	C23—H23B	0.980
Si3—C33	1.888 (8)	C23—H23C	0.980
Si3—C36	1.887 (9)	C24—H24A	0.980
Si3—C30	1.890 (9)	C24—H24B	0.980
C1—C11^{i}	1.417 (8)	C24—H24C	0.980
C1—C2	1.420 (8)	C25—C27	1.522 (11)
C1—C12	1.425 (8)	C25—C26	1.524 (13)
C2—C3	1.402 (7)	C25—H25A	1.00
C2—C11	1.447 (8)	C26—H26A	0.980
C3—C4	1.384 (8)	C26—H26B	0.980
C3—H3A	0.950	C26—H26C	0.980
C4—C5	1.426 (8)	C27—H27A	0.980
C4—C9	1.438 (8)	C27—H27B	0.980
C5—C6	1.369 (8)	C27—H27C	0.980
C5—H5A	0.950	C28—C29	1.202 (9)
C6—C17	1.433 (8)	C30—C32	1.524 (13)
C6—C7	1.450 (8)	C30—C31	1.531 (13)
C7—C8	1.368 (8)	C30—H30A	1.00
C7—C28	1.433 (8)	C31—H31A	0.980
C8—C9	1.426 (8)	C31—H31B	0.980
C8—H8A	0.950	C31—H31C	0.980
C9—C10	1.381 (8)	C32—H32A	0.980
C10—C11	1.412 (8)	C32—H32B	0.980
C10—H10A	0.950	C32—H32C	0.980
C11—C1^{i}	1.417 (8)	C33—C34	1.474 (13)
C12—C13	1.206 (8)	C33—C35	1.529 (12)
C14—H14A	0.980	C33—H33A	1.00
C14—H14B	0.980	C34—H34A	0.980
C14—H14C	0.980	C34—H34B	0.980
C15A—H15A	0.980	C34—H34C	0.980
C15A—H15B	0.980	C35—H35A	0.980
C15A—H15C	0.980	C35—H35B	0.980
C15B—H15D	0.980	C35—H35C	0.980
C15B—H15E	0.980	C36—C37	1.523 (13)
C15B—H15F	0.980	C36—C38	1.537 (12)
C16—H16A	0.980	C36—H36A	1.00
C16—H16B	0.980	C37—H37A	0.980
C16—H16C	0.980	C37—H37B	0.980
C17—C18	1.202 (9)	C37—H37C	0.980
C19—C21	1.514 (15)	C38—H38A	0.980
C19—C20	1.537 (14)	C38—H38B	0.980
C19—H19A	1.00	C38—H38C	0.980
C20—H20A	0.980		
C13—Si1—C14	108.7 (3)	H21A—C21—H21B	109.5
C13—Si1—C16	106.5 (4)	C19—C21—H21C	109.5
C14—Si1—C16	110.7 (4)	H21A—C21—H21C	109.5
C13—Si1—C15A	109.5 (5)	H21B—C21—H21C	109.5
C14—Si1—C15A	117.4 (6)	C23—C22—C24	113.6 (8)

C16—Si1—C15A	103.4 (6)	C23—C22—Si2	113.9 (6)
C13—Si1—C15B	106.1 (7)	C24—C22—Si2	112.6 (7)
C14—Si1—C15B	101.5 (8)	C23—C22—H22A	105.2
C16—Si1—C15B	122.7 (8)	C24—C22—H22A	105.2
C15A—Si1—C15B	20.2 (7)	Si2—C22—H22A	105.2
C18—Si2—C25	107.7 (3)	C22—C23—H23A	109.5
C18—Si2—C19	105.6 (3)	C22—C23—H23B	109.5
C25—Si2—C19	111.8 (4)	H23A—C23—H23B	109.5
C18—Si2—C22	107.0 (3)	C22—C23—H23C	109.5
C25—Si2—C22	111.0 (4)	H23A—C23—H23C	109.5
C19—Si2—C22	113.2 (5)	H23B—C23—H23C	109.5
C29—Si3—C33	108.2 (3)	C22—C24—H24A	109.5
C29—Si3—C36	104.8 (4)	C22—C24—H24B	109.5
C33—Si3—C36	116.9 (4)	H24A—C24—H24B	109.5
C29—Si3—C30	107.8 (4)	C22—C24—H24C	109.5
C33—Si3—C30	109.6 (4)	H24A—C24—H24C	109.5
C36—Si3—C30	109.1 (4)	H24B—C24—H24C	109.5
$C11^{i}$—C1—C2	120.6 (5)	C27—C25—C26	111.2 (8)
$C11^{i}$—C1—C12	120.2 (5)	C27—C25—Si2	112.6 (6)
C2—C1—C12	119.2 (5)	C26—C25—Si2	111.8 (5)
C3—C2—C1	121.4 (5)	C27—C25—H25A	107.0
C3—C2—C11	118.9 (5)	C26—C25—H25A	107.0
C1—C2—C11	119.7 (5)	Si2—C25—H25A	107.0
C4—C3—C2	121.8 (5)	C25—C26—H26A	109.5
C4—C3—H3A	119.1	C25—C26—H26B	109.5
C2—C3—H3A	119.1	H26A—C26—H26B	109.5
C3—C4—C5	121.9 (5)	C25—C26—H26C	109.5
C3—C4—C9	119.5 (5)	H26A—C26—H26C	109.5
C5—C4—C9	118.6 (5)	H26B—C26—H26C	109.5
C6—C5—C4	121.9 (5)	C25—C27—H27A	109.5
C6—C5—H5A	119.0	C25—C27—H27B	109.5
C4—C5—H5A	119.0	H27A—C27—H27B	109.5
C5—C6—C17	119.2 (6)	C25—C27—H27C	109.5
C5—C6—C7	119.6 (5)	H27A—C27—H27C	109.5
C17—C6—C7	121.1 (5)	H27B—C27—H27C	109.5
C8—C7—C28	120.8 (6)	C29—C28—C7	178.8 (8)
C8—C7—C6	119.2 (5)	C28—C29—Si3	176.7 (7)
C28—C7—C6	119.9 (5)	C32—C30—C31	109.4 (8)
C7—C8—C9	122.3 (5)	C32—C30—Si3	113.9 (7)
C7—C8—H8A	118.8	C31—C30—Si3	111.2 (6)
C9—C8—H8A	118.8	C32—C30—H30A	107.4
C10—C9—C8	122.4 (5)	C31—C30—H30A	107.4
C10—C9—C4	119.4 (5)	Si3—C30—H30A	107.4
C8—C9—C4	118.2 (5)	C30—C31—H31A	109.5
C9—C10—C11	121.9 (5)	C30—C31—H31B	109.5
C9—C10—H10A	119.1	H31A—C31—H31B	109.5
C11—C10—H10A	119.1	C30—C31—H31C	109.5
C10—C11—$C1^{i}$	121.8 (5)	H31A—C31—H31C	109.5

C10—C11—C2	118.5 (5)	H31B—C31—H31C	109.5
C1^{i}—C11—C2	119.7 (5)	C30—C32—H32A	109.5
C13—C12—C1	178.7 (7)	C30—C32—H32B	109.5
C12—C13—Si1	178.0 (6)	H32A—C32—H32B	109.5
Si1—C14—H14A	109.5	C30—C32—H32C	109.5
Si1—C14—H14B	109.5	H32A—C32—H32C	109.5
H14A—C14—H14B	109.5	H32B—C32—H32C	109.5
Si1—C14—H14C	109.5	C34—C33—C35	112.2 (9)
H14A—C14—H14C	109.5	C34—C33—Si3	115.6 (6)
H14B—C14—H14C	109.5	C35—C33—Si3	112.3 (6)
Si1—C15A—H15A	109.5	C34—C33—H33A	105.2
Si1—C15A—H15B	109.5	C35—C33—H33A	105.2
H15A—C15A—H15B	109.5	Si3—C33—H33A	105.2
Si1—C15A—H15C	109.5	C33—C34—H34A	109.5
H15A—C15A—H15C	109.5	C33—C34—H34B	109.5
H15B—C15A—H15C	109.5	H34A—C34—H34B	109.5
Si1—C15B—H15D	109.5	C33—C34—H34C	109.5
Si1—C15B—H15E	109.5	H34A—C34—H34C	109.5
H15D—C15B—H15E	109.5	H34B—C34—H34C	109.5
Si1—C15B—H15F	109.5	C33—C35—H35A	109.5
H15D—C15B—H15F	109.5	C33—C35—H35B	109.5
H15E—C15B—H15F	109.5	H35A—C35—H35B	109.5
Si1—C16—H16A	109.5	C33—C35—H35C	109.5
Si1—C16—H16B	109.5	H35A—C35—H35C	109.5
H16A—C16—H16B	109.5	H35B—C35—H35C	109.5
Si1—C16—H16C	109.5	C37—C36—C38	110.5 (8)
H16A—C16—H16C	109.5	C37—C36—Si3	113.5 (6)
H16B—C16—H16C	109.5	C38—C36—Si3	114.8 (7)
C18—C17—C6	176.0 (7)	C37—C36—H36A	105.7
C17—C18—Si2	173.4 (6)	C38—C36—H36A	105.7
C21—C19—C20	109.3 (8)	Si3—C36—H36A	105.7
C21—C19—Si2	111.2 (7)	C36—C37—H37A	109.5
C20—C19—Si2	112.6 (6)	C36—C37—H37B	109.5
C21—C19—H19A	107.8	H37A—C37—H37B	109.5
C20—C19—H19A	107.8	C36—C37—H37C	109.5
Si2—C19—H19A	107.8	H37A—C37—H37C	109.5
C19—C20—H20A	109.5	H37B—C37—H37C	109.5
C19—C20—H20B	109.5	C36—C38—H38A	109.5
H20A—C20—H20B	109.5	C36—C38—H38B	109.5
C19—C20—H20C	109.5	H38A—C38—H38B	109.5
H20A—C20—H20C	109.5	C36—C38—H38C	109.5
H20B—C20—H20C	109.5	H38A—C38—H38C	109.5
C19—C21—H21A	109.5	H38B—C38—H38C	109.5
C19—C21—H21B	109.5		

Symmetry codes: (i) −*x*+2, −*y*, −*z*+2.

Fig. 1


C14
C16
Si1
C15A
C13
C12
C1
C2
C3
C4
C5
C6
C17
C18
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C19
C24
C23
C21
C22
Si2
C27
C25
C26
C34
C11
C10
C9
C8
C7
C29
C28
C33
C35
Si3
C37
C32
C36
C30
C31
C38

Fig. 2