Supplementary Material (ESI) for Chemical Communications

Supporting Information

A Soluble Pentacene: Synthesis, EPR and Electrochemical Studies of 2,3,9,10-Tetrakis(trimethylsilyl)pentacene

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The preparation of compounds **1** was carried out either in a nitrogen-filled glovebox or under nitrogen using standard Schlenk-line technique. All reagents and solvents were reagent grade. Further purification and drying by standard methods were employed when necessary. All organic solvents were evaporated under reduced pressure with a rotary evaporator. The plates used for thin-layer chromatography (TLC) were E. Merck silica gel $60F_{254}$ (0.25 mm thickness) precoated on aluminum plates, and they were visualized under both long (365 nm) and short (254 nm) UV light. Compounds on TLC plates were visualized with a spry of 5% dodecamolybdophosphoric acid in ethanol and with subsequent heating. Column

chromatography was performed using E. Merck silica gel (230-400 mesh).

Melting points were measured on a Reichert Microscope apparatus and were uncorrected. NMR spectra were recorded on a Bruker DPX-300 spectrometer (300.13 MHz for ^{1}H and 75.47 MHz for ^{13}C). All NMR measurements were carried out at 300K in deuterated chloroform solution unless otherwise stated. Chemical shifts are reported as parts per million (ppm) in δ unit in the scale relative to the resonance of $CDCl_3$ (7.26 ppm in the ^{1}H, 77.00 ppm for the central line of the triplet in the ^{13}C modes, respectively). Coupling constants (*J*) are reported in Hz. Splitting patterns are described by using the following abbreviations: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. ^{1}HNMR data is reported in this order: chemical shift; multiplicity; coupling constant(s), number of proton. Mass spectra (ERMS and HRMS) were obtained with a Thermofinnigan MAT 95 XL spectrometer and determined at an ionized voltage of 70 eV unless otherwise stated. Relevant data were tabulated as *m/z*. Elemental analyses were performed at Shanghai Institute of Organic Chemistry, the Chinese Academy of Sciences, China. EPR spectrum was recorded with a Bruker EMX EPR spectrometer with stated experimental condition. Cyclic voltammetric measurement was carried out at 25^{o}C under a nitrogen atmosphere using a BAS CV-50W voltammetric analyzer. A three-electrode system which consisted of a platinum ball working electrode, a silver reference electrode and a platinum foil auxiliary electrode was used with $[Bu_4N]^{+}[BF4]^{-}$ as the supporting electrolyte. All redox potentials were referenced to the ferrocenium / ferrocene redox couple as recommended by the IUPAC. The UV measurement was performed with Varian Cary 1E UV spectrophotometer

2,3,9,10-tetrakis(trimethylsilyl)pentacene (1): A mixture of aluminum (100 g), carbon tetrabromide (10 mg) and mercuric chloride (2 mg) in cyclohexanol (3 ml) was heated to reflux for 1.5 hr under N_2. After the formation of dark grey mixture, the reaction flask was

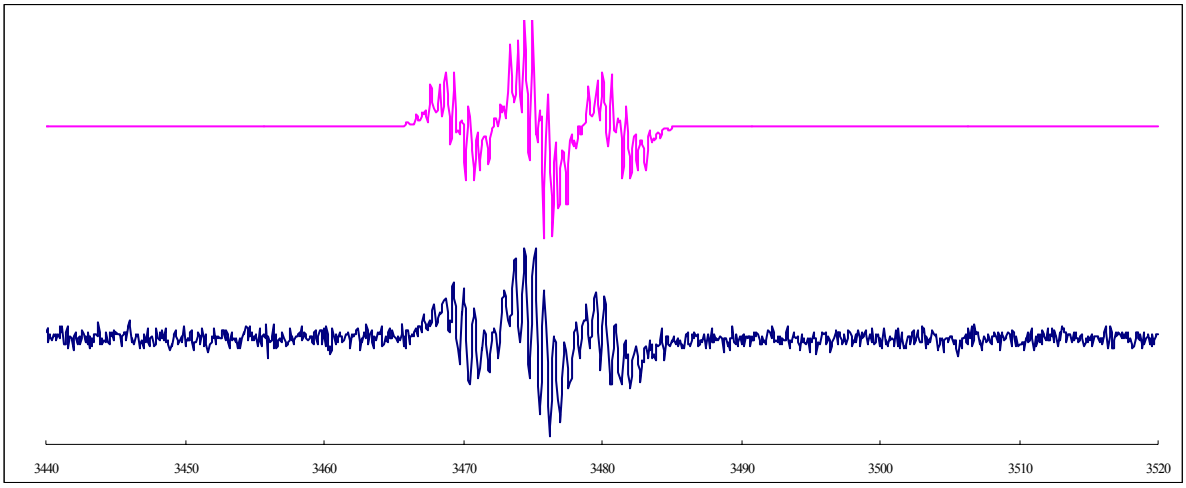
cooled to r.t.. Then solid **2** (100 mg, 0.17 mmol) was added and the mixture was heated again to reflux for 2 hr. The reaction flask was then cooled to r.t. Cyclohexanol was removed under vacuum. Degassed hexanes (5 mL) was then added to the reaction mixture via a syringe and the mixture was transferred to the silica gel column with rigorous exclusion of air (10 g, degassed hexanes). A purple band of eluent was collected under N_2 to afford **1** (76 mg, 80%) as a purple solid: m.p. 320^{o}C (decomp.); ^{1}H NMR ($CDCl_3$) δ 0.48 (s, 36H), 8.25 (s, 4H), 8.62 (s, 4H), 8.96 (s, 2H); ^{13}C NMR ($CDCl_3$) δ 1.9, 126.2, 126.8, 130.5, 130.7, 136.6, 140.6; MS *m/z* 566 (M^+). HRMS (FAB) Calcd for $C_{34}H_{46}Si_4$ (MH^+): 566.2671. Found: 566.2665. UV-Vis (CH_2Cl_2): 318 nm.

3,4,11,12,19,20,25,26-Octakis(trimethylsilyl)-7,16[2',3']:8,15[2",3"]-dinaphthalenodinaphtho[*a*,*e*]cyclooctene (**3**)**.** A solution of **1** (50 mg, 0.09 mmol) in hexane was concentrated and a colorless crystal was obtained after 2 days under N_2 and sunlight. The crystal was then filtered and washed with a minimum amount of cold hexane to afford **3** (5 mg, 5%) as a white solid: m.p. 300^{o}C (decomp); ^{1}H NMR ($CDCl_3$) δ 0.32 (s, 72H), 5.15 (s, 4H), 7.44 (s, 8H), 7.83 (s, 8H); ^{13}C NMR ($CDCl_3$) δ 1.9, 54.2, 125.4, 131.3, 135.2, 141.0, 141.5; HRMS (APCI) Calcd for $C_{68}H_{93}Si_8$ (MH^+): 1133.5353. Found: 1133.5084.

2,3,9,10-tetrakis(trimethylsilyl)-6-13-dihydro-pentacene-6,13-peroxide (**4**): Compound **1** (76 mg, 0.13 mmol) was dissolved in hexane (5 mL) in the presence of sunlight and atmospheric air. After stirring for 10 min., the solvent was then removed under reduced pressure. The resulting crude product was purified by column chromatography on silica gel (10 g, hexanes) to afford **4** (16 mg, 20%) as a light-yellow oil: ^{1}H NMR ($CDCl_3$) δ 0.42 (s, 36H), 6.25 (s, 2H), 7.83 (s, 4H), 8.12 (s, 4H); ^{13}C NMR ($CDCl_3$) δ 1.7, 79.5, 121.3, 122.2, 131.6, 135.1, 135.4; MS *m/z* 598 (M^+). HRMS (EI) Calcd for $C_{34}H_{46}Si_4O_2$ (M^+) 598.2569.

Found: 598.2560.

EPR measurement of 1. Samples **1** were prepared *in situ* before each measurement. Sample tubes and solvents were degassed before use. Samples in the inert environment should give no EPR signal. The sample tubes were then allowed to contact with atmospheric air by open the cap of the sample tube throughout the measurement. The EPR spectrum was recorded with a Bruker EMX EPR spectrometer: microwave frequency = 9.722 GHz; modulation amplitude = 0.8 G, time constant = 10.24 msec; T = 298K. The spectrum elicits an isotropic signal centered at g = 2.00.



The EPR spectrum of $\mathbf{1}^{+\cdot}$**(**bottom) and its computer simulation (top). The spectrum elicits an isotropic signal centered at g = 2.00. ^{1}H hyperfine splittings as obtained by computer simulation: 5.65 G for the two hydrogens in the 6,13-positions, 1.05 G for four hydrogens in the 5,7,12,14-positions and 0.65 G for four hydrogens in the 1,4,8,11-positions (line-width = 0.15 G). The EPR spectrum was recorded with a Bruker EMX EPR spectrometer: microwave frequency = 9.722 GHz; modulation amplitude = 0.8G, time constant = 10.24 msec; T = 298K.

Electrochemical measurement of 1. Sample was immediately recorded after preparation. The cyclic voltammetric measurements were carried out in a mixed hexane:CH_2Cl_2 (1:4)

solvent system at 25°C under a nitrogen atmosphere using a BAS CV-50W voltammetric analyzer. A three-electrode system which consisted of a platinum ball working electrode, a silver reference electrode and a platinum foil auxiliary electrode was used with $[Bu_4N]^+[BF_4]^-$ as the supporting electrolyte. All redox potentials were referenced to the ferrocenium / ferrocene redox couple as recommended by the IUPAC.

2,3,8,9-Tetrakis(trimethylsilyl)-13,14-dihydro-13,14-ethanopentacenedicarboxylic Anhydride (**5a**): A solution of **1** (10 mg, 0.018 mmol) in CH_2Cl_2 (6 mL) was added to a solution of maleic anhydride (1.5 mg, 0.015 mmol) dropwise at r.t. under N_2. After stirring overnight, the color of the reaction mixture changed from purple-red to colorless, and the solvent was removed under reduced pressure. The solid residue was washed with hexanes (0.5 mL) and **5a** (5 mg, 40%) was obtained as a white solid: m.p. 250°C (decomp.); ^{1}H NMR ($CDCl_3$) δ 0.41 (s, 18H), 0.41 (s, 18H), 3.64 (s, 2H), 5.04 (s, 2H), 7.78 (s, 2H), 7.81 (s, 2H), 8.08 (s, 2H), 8.09 (s, 2H); ^{13}C NMR ($CDCl_3$) δ 1.9, 45.3, 47.9, 122.9, 124.0, 131.5, 131.7, 135.3, 135.4, 137.9, 143.1, 170.5; MS *m/z* 664 (M^+). HRMS (EI) Calcd for $C_{38}H_{48}Si_4O_3$ (M^+) 664.2675. Found: 664.2673.

2,3,8,9-Tetrakis(trimethylsilyl)-13,14-dihydro-13,14-ethanopentacene-17-phenyl-17*H*-pyrrole-16,18-dione (**5b**): A solution of **1** (50 mg, 0.088 mmol) in CH_2Cl_2 (8 mL) was added to a solution of *N*-phenyl maleimide (15 mg, 0.088 mmol) dropwise at r.t. under N_2. After stirring overnight, the color of the reaction mixture changed from purple-red to colorless, and the solvent was removed under reduced pressure. The resulting crude product was purified by column chromatography on silica gel (10 g, hexanes: ethyl acetate / 10:1) to afford **5b** (52 mg, 80%) as a white solid: m.p. 250°C (decomp.); ^{1}H NMR ($CDCl_3$) δ 0.38 (s, 18H), 0.40 (s, 18H),

3.49 (s, 2H), 5.11 (s, 2H), 6.27 (d, *J* = 7.2 Hz, 2H), 7.06-7.18 (m, 3H), 7.77 (s, 2H), 7.84 (s, 2H), 8.06 (s, 2H), 8.12 (s, 2H); ^{13}C NMR ($CDCl_3$) δ 1.9, 2.0, 45.8, 47.0, 122.7, 123.7, 126.3, 128.7, 129.0, 131.5, 135.3, 136.1, 138.8, 142.5, 142.7, 176.2; MS *m/z* 739 (M^+). HRMS (EI) Calcd for $C_{44}H_{53}N_1Si_4O_2$ (M^+) 739.3148. Found: 739.3144.

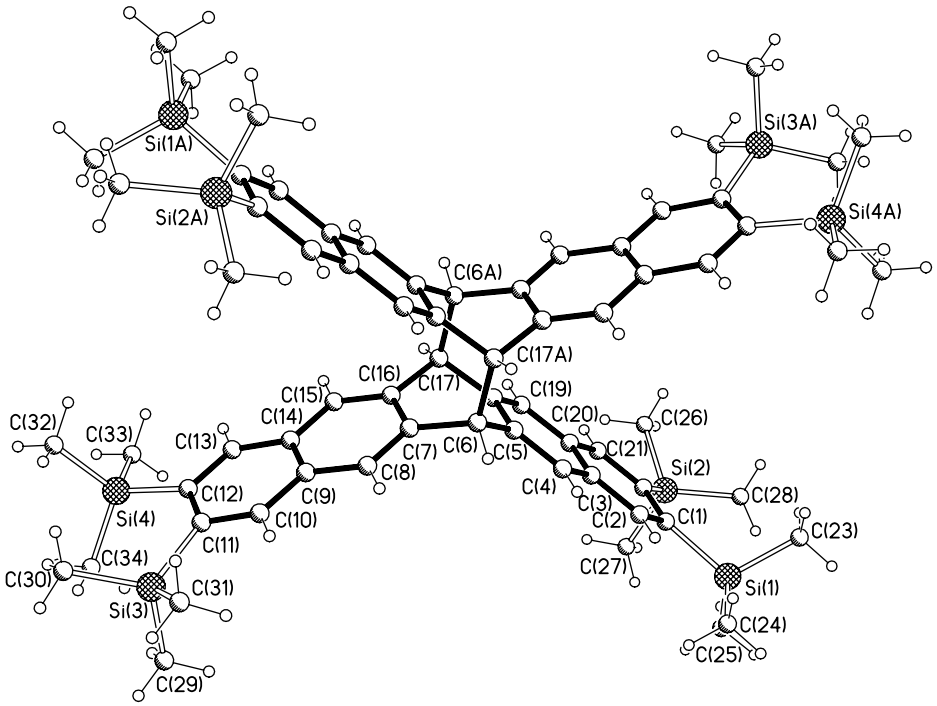
2,3,8,9-Tetrakis(trimethylsilyl)-13,14-dihydro-13,14-ethanopentacene-17-ethyl-17*H*-pyrrole-16,18-dione (**5c**). A solution of **1** (50 mg, 0.088 mmol) in CH_2Cl_2 (8 mL) was added to a solution of *N*-ethyl maleimide (11 mg, 0.088 mmol) dropwise at r.t. under N_2. After stirring overnight, the color of the reaction mixture changed from purple-red to colorless, and the solvent was removed under reduced pressure. The resulting crude product was purified by column chromatography on silica gel (10 g, hexanes: ethyl acetate / 10:1) to afford **5c** (21 mg, 35%) as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 0.22 (t, *J* = 6 Hz, 3H), 0.40 (s, 18H), 0.42 (s, 18H), 3.07 (q, *J* = 7.2 Hz, 2H), 3.29 (s, 2H), 5.01 (s, 2H), 7.70 (s, 2H), 7.79 (s, 2H), 8.02 (s, 2H), 8.09 (s, 2H); ^{13}C NMR ($CDCl_3$) δ 1.9, 11.9, 33.3, 45.5, 46.7, 122.6, 123.5, 131.5, 134.1, 135.3, 136.1, 139.1, 142.3, 142.6, 176.8; MS *m/z* 691 (M^+). HRMS (EI) Calcd for $C_{40}H_{53}N_1Si_4O_2$ (M^+) 691.3148. Found: 691.3138.

Dimethyl 2,3,8,9-tetrakis(trimethylsilyl)-13,14-dihydro-13,14-ethenopentacene-15,16-dicarboxylate (**6**). A solution of **1** (50 mg, 0.088 mmol) in CH_2Cl_2 (8 mL) was added to a solution of DMAD (0.01 mL, 0.09 mmol) dropwise at r.t. under N_2. After stirring for 7d, the color of the reaction mixture changed from purple-red to colorless, and the solvent was removed under reduced pressure. The resulting crude product was purified by column chromatography on silica gel (10 g, hexanes: ethyl acetate / 10:1) to afford **6** (6 mg, 10%) as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 0.38 (s, 36H), 3.79 (s, 6H), 5.66 (s, 2H), 7.79 (s, 4H), 8.01 (s, 4H); ^{13}C NMR ($CDCl_3$) δ 1.7, 52.2, 79.5, 120.1, 121.3, 122.2, 131.6, 135.1, 135.4, 162.4; MS

m/z 708 (M^+). HRMS (EI) Calcd for $C_{40}H_{52}Si_4O_4$ (M^+) 708.2937. Found: 708.2933.

2,3,8,9-Tetrakis(trimethylsilyl)-13,14-dihydro-13,14[1',2']benzenopentacene (**7**). To a solution of **1** (50 mg, 0.088 mmol) and phenyl [2-(trimethylsilyl)-1-phenyl]iodonium triflate (44 mg, 0.088 mmol) in CH_2Cl_2 (8 mL), TBAF (1 M in THF, 0.09 mL) was added dropwise at 0^oC. After stirring for 15 min., the mixture was poured into water (3 mL). The organic layer was collected and the aqueous residue was extracted with CH_2Cl_2 (3 x 3 mL). The combined organic extract was dried over $MgSO_4$ and concentrated under reduced pressure. Chromatography on silica gel (10 g, hexanes) afforded **7** (21 mg, 37%) as a colorless oil: ^1H NMR ($CDCl_3$) δ 0.36 (s, 18H), 5.63 (s, 2H), 7.01 (dd, *J* = 5.4, 3.3 Hz, 2H), 7.41 (dd, *J* = 5.4, 3.3 Hz, 2H), 7.81 (s, 4H), 8.00 (s, 4H); MS *m/z* 642 (M^+). HRMS (FAB) Calcd for $C_{40}H_{50}Si_4$: 642.2984. Found: 642.2976.

2,3,8,9-Tetrakis(trimethylsilyl)-15,16-dicyano-13,14-dihydro-13,14-ethanopentacene (**8**). A solution of **1** (50 mg, 0.088 mmol) in CH_2Cl_2 (8 mL) was added to a solution of fumaronitrile (7 mg, 0.088 mmol) dropwise at r.t. under N_2. After stirring for 5d, the color of the reaction mixture changed from purple-red to colorless, and the solvent was removed under reduced pressure. The resulting crude product was purified by column chromatography on silica gel (10 g, hexanes: ethyl acetate / 10:1) to afford **8** (6 mg, 10%) as a colorless oil: ^1H NMR ($CDCl_3$) δ 0.41 (s, 36H), 3.29 (s, 2H), 4.83 (s, 2H), 7.79 (s, 2H), 7.90 (s, 2H), 8.09 (s, 2H), 8.12 (s, 2H); MS *m/z* 644 (M^+). HRMS (EI) Calcd for $C_{38}H_{48}N_2Si_4$ (M^+) 644.2889. Found: 644.2879.



Molecular structure and atom labeling of **3**. Symmetry transformation used to generate equivalent atoms: #1 –x+1, -y+2, -z. Selected bond distances and angles: C6-C17#1 = 1.604 Å, C5-C6-C11#1 = 112.96^{o}

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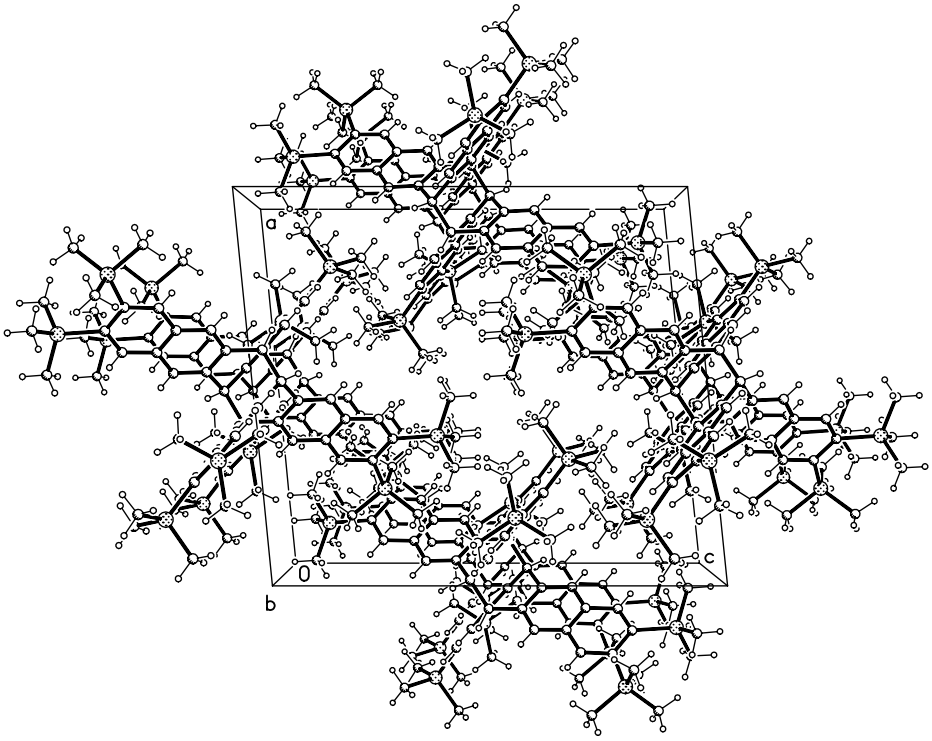

Stereoview of molecular packing of **3**. H atoms have been omitted for clarity.

Structure Determination Summary for **3**

Crystal Data

Empirical formula	C40 H60 Si4
Color and Habit	colorless block
Crystal Size (mm)	0.40 x 0.30 x 0.30
Crystal system	Monoclinic
Space group	P2(1)/n
Unit cell dimensions	
a (A)	16.574(4)
b (A)	13.868(3)
c (A)	18.762(4)
alpha (deg.)	90
beta (deg.)	95.71(2)
gamma (deg.)	90
Volume(A^3)	4291.2(16)
Z	4
Formula weight	653.24
Density(cal.)(Mg/m^3)	1.011
Absorption coefficient(mm^-1)	0.162
F(000)	1424
Data Collection	
Diffractometer Used	Bruker APEX CCD CCD
Radiation	MoK\a 0.71073A
Temperature(K)	123(2)

Monochromator	Highly oriented graphite crystal
Theta range (deg.)	2.81 to 28.00
Scan Type and Rate	Omega scan
Crystal to Detector Distance (mm)	?
Background Level	?
Reflections measured	46139
Index ranges of measured data	-21<=h<=21, -18<=k<=18, -24<=l<=24
Independent reflections	10359 (Rint = 0.0326)
Observed Reflection	8207 (>2sigma(I))
Absorption Correction	SADABS
Relative Transmission Factor	0.9380 - 0.9530
Solution and Refinement	
ystem Used	SMART for Windows NT, Bruker AXS Inc., Madison
Structure Solution	SHELXS-97 (Sheldrick, 1990)
Structure Refinement	SHELXL-97 (Sheldrick, 1997)
Refinement Method	Full-matrix least-squares on F^2
	Flack H D(1983), Acta Cryst. A39, 876-881
Weighting Scheme	calc
Parameter/Restraints/Data(obs.)	415 / 88 / 8207
Final R indices (obs.)	R1 = 0.0670, wR2 = 0.1915
R indices (all)	R1 = 0.0818, wR2 = 0.2088
Goodness-of-fit	1.069
Largest and Mean Delta/Sigma	0.001, 0.000
Largest difference peak(e.A^-3)	0.779, -0.551

Table 1. Atomic coordinates and equivalent isotropic displacement parameters (A^2).

atom	x	y	z	U(eq)	SOF	
Si(1)	0.67335(4)	0.43630(4)	0.04831(3)	0.03027(16)	1	Uani
Si(2)	0.81587(4)	0.57635(4)	0.18091(3)	0.03184(17)	1	Uani
Si(3)	0.62692(5)	1.20637(6)	-0.39284(4)	0.0445(2)	1	Uani
Si(4)	0.76719(6)	1.34636(7)	-0.25848(4)	0.0615(3)	1	Uani
C(1)	0.67596(12)	0.57044(13)	0.06553(10)	0.0239(4)	1	Uani
C(2)	0.61915(12)	0.62206(14)	0.02206(10)	0.0227(4)	1	Uani
C(3)	0.61032(11)	0.72355(13)	0.02522(10)	0.0203(4)	1	Uani
C(4)	0.55218(11)	0.77437(13)	-0.02126(9)	0.0192(3)	1	Uani
C(5)	0.54629(10)	0.87282(13)	-0.01761(9)	0.0173(3)	1	Uani
C(6)	0.48797(10)	0.93073(12)	-0.06802(9)	0.0170(3)	1	Uani
C(7)	0.53559(10)	1.01296(12)	-0.09665(9)	0.0176(3)	1	Uani
C(8)	0.53257(11)	1.03542(13)	-0.16779(10)	0.0198(4)	1	Uani
C(9)	0.58294(11)	1.10866(13)	-0.19244(10)	0.0219(4)	1	Uani
C(10)	0.58330(13)	1.13227(15)	-0.26588(11)	0.0269(4)	1	Uani
C(11)	0.63478(15)	1.19978(16)	-0.29137(12)	0.0326(5)	1	Uani
C(12)	0.69117(15)	1.24969(16)	-0.23998(12)	0.0348(5)	1	Uani
C(13)	0.68958(13)	1.22790(15)	-0.16841(12)	0.0295(4)	1	Uani
C(14)	0.63697(12)	1.15822(13)	-0.14232(10)	0.0223(4)	1	Uani
C(15)	0.63826(11)	1.13538(13)	-0.06861(10)	0.0212(4)	1	Uani
C(16)	0.58881(11)	1.06494(12)	-0.04593(10)	0.0182(3)	1	Uani
C(17)	0.59046(10)	1.03395(12)	0.03163(9)	0.0176(3)	1	Uani
C(18)	0.59919(11)	0.92518(12)	0.03301(9)	0.0182(3)	1	Uani
C(19)	0.65596(11)	0.87752(14)	0.07778(10)	0.0213(4)	1	Uani

C(20)	0.66262(11)	0.77555(13)	0.07510(10)	0.0219(4)	1	Uani
C(21)	0.72059(12)	0.72288(14)	0.12044(10)	0.0258(4)	1	Uani
C(22)	0.72888(13)	0.62357(15)	0.11762(10)	0.0261(4)	1	Uani
C(23)	0.62216(19)	0.3740(2)	0.11973(16)	0.0544(8)	1	Uani
C(24)	0.77549(17)	0.38254(19)	0.03894(17)	0.0488(7)	1	Uani
C(25)	0.61161(16)	0.41239(16)	-0.03902(13)	0.0371(5)	1	Uani
C(26)	0.8355(2)	0.6631(2)	0.25697(17)	0.0732(12)	1	Uani
C(27)	0.90846(16)	0.5712(2)	0.13204(16)	0.0478(6)	1	Uani
C(28)	0.79891(18)	0.4588(2)	0.22531(15)	0.0507(7)	1	Uani
C(29)	0.7075(3)	1.1291(4)	-0.4242(2)	0.0841(13)	1	Uani
C(30)	0.6326(4)	1.3290(3)	-0.4316(2)	0.1053(19)	1	Uani
C(31)	0.5276(2)	1.1551(3)	-0.43044(15)	0.0696(10)	1	Uani
C(32)	0.7141(4)	1.4635(3)	-0.2770(3)	0.1072(18)	1	Uani
C(33)	0.8381(3)	1.3667(3)	-0.1762(2)	0.0848(14)	1	Uani
C(34)	0.8324(3)	1.3132(4)	-0.3286(3)	0.128(3)	1	Uani
C(35)	0.9745(19)	1.130(2)	0.2852(9)	0.099(5)	0.50	Uani
C(36)	0.9147(9)	1.1668(11)	0.2265(5)	0.176(6)	0.50	Uani
C(37)	0.9165(10)	1.0997(9)	0.1634(6)	0.156(4)	0.50	Uani
C(38)	0.8430(9)	1.1182(13)	0.1129(7)	0.209(7)	0.50	Uani
C(39)	0.8430(8)	1.0501(13)	0.0509(5)	0.177(5)	0.50	Uani
C(40)	0.8963(13)	1.092(2)	-0.0011(11)	0.159(8)	0.50	Uani
C(35')	0.968(2)	1.159(2)	0.2952(10)	0.099(5)	0.50	Uani
C(36')	0.9453(10)	1.0535(15)	0.2332(5)	0.176(6)	0.50	Uani
C(37')	0.9010(15)	1.0920(16)	0.1744(9)	0.156(4)	0.50	Uani
C(38')	0.8777(12)	1.0235(9)	0.1145(6)	0.209(7)	0.50	Uani
C(39')	0.8346(6)	1.0732(14)	0.0505(5)	0.177(5)	0.50	Uani
C(40')	0.8678(14)	1.109(2)	-0.0196(15)	0.159(8)	0.50	Uani

Table 2. Bond lengths (A) and angles (deg.).

Si(1)-C(23)	1.868(3)	C(7)-C(16)	1.427(2)
Si(1)-C(25)	1.873(2)	C(8)-C(9)	1.421(3)
Si(1)-C(24)	1.874(3)	C(9)-C(14)	1.411(3)
Si(1)-C(1)	1.888(2)	C(9)-C(10)	1.417(3)
Si(2)-C(28)	1.865(3)	C(10)-C(11)	1.384(3)
Si(2)-C(27)	1.866(3)	C(11)-C(12)	1.450(3)
Si(2)-C(26)	1.870(3)	C(12)-C(13)	1.379(3)
Si(2)-C(22)	1.891(2)	C(13)-C(14)	1.421(3)
Si(3)-C(29)	1.853(4)	C(14)-C(15)	1.417(3)
Si(3)-C(30)	1.855(4)	C(15)-C(16)	1.370(3)
Si(3)-C(31)	1.865(3)	C(16)-C(17)	1.515(2)
Si(3)-C(11)	1.898(2)	C(17)-C(18)	1.515(2)
Si(4)-C(34)	1.843(4)	C(17)-C(6)#1	1.604(2)
Si(4)-C(32)	1.864(5)	C(18)-C(19)	1.368(3)
Si(4)-C(33)	1.867(4)	C(19)-C(20)	1.420(3)
Si(4)-C(12)	1.895(2)	C(20)-C(21)	1.420(3)
C(1)-C(2)	1.382(3)	C(21)-C(22)	1.386(3)
C(1)-C(22)	1.448(3)	C(35)-C(36)	1.496(5)
C(2)-C(3)	1.417(3)	C(36)-C(37)	1.509(5)
C(3)-C(20)	1.409(3)	C(37)-C(38)	1.489(5)
C(3)-C(4)	1.421(2)	C(38)-C(39)	1.497(5)
C(4)-C(5)	1.371(2)	C(39)-C(40)	1.500(5)
C(5)-C(18)	1.426(2)	C(35')-C(36')	1.88(3)
C(5)-C(6)	1.514(2)	C(36')-C(37')	1.37(2)
C(6)-C(7)	1.515(2)	C(37')-C(38')	1.49(2)
C(6)-C(17)#1	1.604(2)	C(38')-C(39')	1.501(5)
C(7)-C(8)	1.367(2)	C(39')-C(40')	1.56(2)

C(23)-Si(1)-C(25)	107.34(13)
C(23)-Si(1)-C(24)	111.24(15)
C(25)-Si(1)-C(24)	105.52(12)
C(23)-Si(1)-C(1)	109.72(13)
C(25)-Si(1)-C(1)	108.98(9)
C(24)-Si(1)-C(1)	113.74(11)
C(28)-Si(2)-C(27)	110.73(14)
C(28)-Si(2)-C(26)	104.13(17)
C(27)-Si(2)-C(26)	108.08(18)
C(28)-Si(2)-C(22)	116.35(11)
C(27)-Si(2)-C(22)	108.52(11)
C(26)-Si(2)-C(22)	108.69(11)
C(29)-Si(3)-C(30)	109.8(3)
C(29)-Si(3)-C(31)	107.23(19)
C(30)-Si(3)-C(31)	106.2(2)
C(29)-Si(3)-C(11)	108.10(16)
C(30)-Si(3)-C(11)	115.89(15)
C(31)-Si(3)-C(11)	109.32(12)
C(34)-Si(4)-C(32)	112.6(3)
C(34)-Si(4)-C(33)	105.2(2)
C(32)-Si(4)-C(33)	105.6(2)
C(34)-Si(4)-C(12)	113.71(16)
C(32)-Si(4)-C(12)	109.89(19)
C(33)-Si(4)-C(12)	109.32(13)
C(2)-C(1)-C(22)	117.77(17)
C(2)-C(1)-Si(1)	113.99(15)
C(22)-C(1)-Si(1)	128.23(14)
C(1)-C(2)-C(3)	123.83(18)
C(20)-C(3)-C(2)	118.48(17)
C(20)-C(3)-C(4)	119.24(16)
C(2)-C(3)-C(4)	122.25(17)
C(5)-C(4)-C(3)	120.71(16)
C(4)-C(5)-C(18)	119.89(16)
C(4)-C(5)-C(6)	122.77(16)
C(18)-C(5)-C(6)	117.27(15)
C(5)-C(6)-C(7)	107.34(14)
C(5)-C(6)-C(17)#1	112.96(14)
C(7)-C(6)-C(17)#1	113.19(14)
C(8)-C(7)-C(16)	119.75(16)
C(8)-C(7)-C(6)	123.23(16)
C(16)-C(7)-C(6)	116.93(15)
C(7)-C(8)-C(9)	121.01(17)
C(14)-C(9)-C(10)	118.14(17)
C(14)-C(9)-C(8)	119.07(17)
C(10)-C(9)-C(8)	122.76(18)
C(11)-C(10)-C(9)	123.7(2)
C(10)-C(11)-C(12)	118.17(19)
C(10)-C(11)-Si(3)	113.34(17)
C(12)-C(11)-Si(3)	128.27(16)
C(13)-C(12)-C(11)	117.98(19)
C(13)-C(12)-Si(4)	114.26(17)
C(11)-C(12)-Si(4)	127.74(16)
C(12)-C(13)-C(14)	123.8(2)
C(9)-C(14)-C(15)	119.23(17)
C(9)-C(14)-C(13)	118.19(18)
C(15)-C(14)-C(13)	122.56(18)
C(16)-C(15)-C(14)	120.75(17)

C(15)-C(16)-C(7)	120.16(17)
C(15)-C(16)-C(17)	123.13(16)
C(7)-C(16)-C(17)	116.62(15)
C(16)-C(17)-C(18)	106.94(14)
C(16)-C(17)-C(6)#1	112.61(14)
C(18)-C(17)-C(6)#1	112.14(14)
C(19)-C(18)-C(5)	120.20(16)
C(19)-C(18)-C(17)	123.39(16)
C(5)-C(18)-C(17)	116.37(15)
C(18)-C(19)-C(20)	120.71(17)
C(3)-C(20)-C(19)	119.24(16)
C(3)-C(20)-C(21)	117.99(17)
C(19)-C(20)-C(21)	122.77(17)
C(22)-C(21)-C(20)	123.50(18)
C(21)-C(22)-C(1)	118.41(17)
C(21)-C(22)-Si(2)	113.11(15)
C(1)-C(22)-Si(2)	128.34(15)
C(35)-C(36)-C(37)	107.6(5)
C(38)-C(37)-C(36)	108.4(5)
C(37)-C(38)-C(39)	108.8(5)
C(38)-C(39)-C(40)	107.7(5)
C(37')-C(36')-C(35')	104.5(19)
C(36')-C(37')-C(38')	116(2)
C(37')-C(38')-C(39')	112.1(12)
C(38')-C(39')-C(40')	129.8(18)

Symmetry transformations used to generate equivalent atoms: #1 -x+1,-y+2,-z

Table 3. Anisotropic displacement parameters (A^2).

atom	U11	U22	U33	U23	U13	U12
Si(1)	0.0371(3)	0.0191(3)	0.0333(3)	0.0035(2)	-0.0028(2)	0.0045(2)
Si(2)	0.0384(3)	0.0302(3)	0.0247(3)	0.0003(2)	-0.0081(2)	0.0118(2)
Si(3)	0.0660(5)	0.0465(4)	0.0240(3)	0.0019(3)	0.0183(3)	-0.0183(3)
Si(4)	0.0853(6)	0.0638(5)	0.0389(4)	-0.0023(4)	0.0246(4)	-0.0480(5)
C(1)	0.0304(10)	0.0208(9)	0.0210(9)	0.0033(7)	0.0053(7)	0.0046(7)
C(2)	0.0255(9)	0.0212(9)	0.0213(9)	-0.0007(7)	0.0021(7)	0.0018(7)
C(3)	0.0223(8)	0.0201(8)	0.0187(8)	0.0006(6)	0.0034(7)	0.0026(7)
C(4)	0.0210(8)	0.0187(8)	0.0179(8)	-0.0009(6)	0.0021(7)	-0.0002(6)
C(5)	0.0177(8)	0.0191(8)	0.0153(8)	0.0006(6)	0.0030(6)	0.0012(6)
C(6)	0.0197(8)	0.0161(8)	0.0151(8)	-0.0002(6)	0.0010(6)	-0.0002(6)
C(7)	0.0171(8)	0.0172(8)	0.0188(8)	0.0000(6)	0.0031(6)	0.0016(6)
C(8)	0.0221(8)	0.0189(8)	0.0184(8)	-0.0005(6)	0.0029(7)	0.0004(7)
C(9)	0.0247(9)	0.0209(8)	0.0212(9)	0.0008(7)	0.0082(7)	0.0010(7)
C(10)	0.0345(11)	0.0265(9)	0.0209(9)	0.0002(7)	0.0081(8)	-0.0013(8)
C(11)	0.0437(12)	0.0310(11)	0.0254(10)	0.0012(8)	0.0146(9)	-0.0043(9)
C(12)	0.0446(12)	0.0290(10)	0.0336(11)	0.0009(9)	0.0181(10)	-0.0105(9)
C(13)	0.0334(11)	0.0271(10)	0.0299(10)	-0.0030(8)	0.0121(8)	-0.0075(8)
C(14)	0.0248(9)	0.0198(8)	0.0236(9)	-0.0006(7)	0.0093(7)	-0.0007(7)
C(15)	0.0218(8)	0.0191(8)	0.0231(9)	-0.0030(7)	0.0044(7)	-0.0016(7)
C(16)	0.0186(8)	0.0175(8)	0.0190(8)	-0.0012(6)	0.0037(6)	0.0011(6)
C(17)	0.0175(8)	0.0183(8)	0.0167(8)	0.0002(6)	0.0009(6)	0.0000(6)
C(18)	0.0192(8)	0.0188(8)	0.0171(8)	-0.0012(6)	0.0036(6)	0.0003(6)
C(19)	0.0223(8)	0.0229(9)	0.0181(8)	-0.0014(7)	0.0000(7)	0.0014(7)
C(20)	0.0242(9)	0.0224(9)	0.0189(8)	0.0004(7)	0.0012(7)	0.0040(7)
C(21)	0.0290(10)	0.0271(10)	0.0201(9)	-0.0018(7)	-0.0030(7)	0.0059(8)
C(22)	0.0311(10)	0.0276(10)	0.0193(9)	0.0020(7)	0.0014(7)	0.0094(8)
C(23)	0.0591(17)	0.0516(16)	0.0490(16)	0.0234(13)	-0.0123(13)	-0.0224(13)
C(24)	0.0474(14)	0.0339(12)	0.0621(17)	-0.0136(12)	-0.0092(12)	0.0180(11)

C(25)	0.0454(13)	0.0250(10)	0.0392(13)	-0.0061(9)	-0.0044(10)	0.0067(9)
C(26)	0.097(3)	0.066(2)	0.0472(17)	-0.0242(15)	-0.0433(18)	0.0424(19)
C(27)	0.0354(13)	0.0575(17)	0.0490(15)	0.0117(12)	-0.0028(11)	0.0058(11)
C(28)	0.0531(16)	0.0568(16)	0.0392(14)	0.0226(12)	-0.0104(12)	0.0042(13)
C(29)	0.083(3)	0.120(3)	0.056(2)	-0.036(2)	0.0372(19)	-0.013(2)
C(30)	0.193(6)	0.075(3)	0.0455(19)	0.0261(18)	-0.001(3)	-0.051(3)
C(31)	0.076(2)	0.104(3)	0.0279(13)	0.0116(15)	0.0046(14)	-0.030(2)
C(32)	0.171(5)	0.053(2)	0.095(3)	0.020(2)	-0.002(3)	-0.047(3)
C(33)	0.090(3)	0.102(3)	0.066(2)	-0.005(2)	0.023(2)	-0.068(2)
C(34)	0.131(4)	0.175(5)	0.091(3)	-0.057(3)	0.081(3)	-0.114(4)
C(35)	0.087(5)	0.147(15)	0.066(5)	-0.014(6)	0.021(5)	-0.048(7)
C(36)	0.148(9)	0.293(16)	0.081(5)	0.054(9)	-0.014(6)	-0.074(11)
C(37)	0.167(9)	0.189(8)	0.115(7)	-0.027(6)	0.023(6)	0.005(7)
C(38)	0.276(16)	0.190(13)	0.136(9)	-0.038(9)	-0.098(10)	0.046(12)
C(39)	0.150(8)	0.150(10)	0.235(11)	0.043(7)	0.036(8)	0.015(7)
C(40)	0.106(14)	0.126(11)	0.256(15)	-0.074(10)	0.072(12)	-0.017(11)
C(35')	0.087(5)	0.147(15)	0.066(5)	-0.014(6)	0.021(5)	-0.048(7)
C(36')	0.148(9)	0.293(16)	0.081(5)	0.054(9)	-0.014(6)	-0.074(11)
C(37')	0.167(9)	0.189(8)	0.115(7)	-0.027(6)	0.023(6)	0.005(7)
C(38')	0.276(16)	0.190(13)	0.136(9)	-0.038(9)	-0.098(10)	0.046(12)
C(39')	0.150(8)	0.150(10)	0.235(11)	0.043(7)	0.036(8)	0.015(7)
C(40')	0.106(14)	0.126(11)	0.256(15)	-0.074(10)	0.072(12)	-0.017(11)

Table 4. Hydrogen coordinates and isotropic displacement parameters (A^2).

	x	y	z	U(eq)
H(2A)	0.5840	0.5875	-0.0119	0.027
H(4A)	0.5170	0.7398	-0.0552	0.023
H(6A)	0.4689	0.8882	-0.1094	0.020
H(8A)	0.4962	1.0016	-0.2013	0.024
H(10A)	0.5461	1.0999	-0.2995	0.032
H(13A)	0.7258	1.2614	-0.1346	0.035
H(15A)	0.6738	1.1693	-0.0345	0.025
H(17A)	0.6396	1.0626	0.0589	0.021
H(19A)	0.6913	0.9130	0.1110	0.026
H(21A)	0.7555	0.7576	0.1545	0.031
H(23A)	0.6210	0.3044	0.1106	0.082
H(23B)	0.5666	0.3981	0.1195	0.082
H(23C)	0.6520	0.3868	0.1665	0.082
H(24A)	0.7698	0.3131	0.0303	0.073
H(24B)	0.8111	0.3935	0.0831	0.073
H(24C)	0.7990	0.4129	-0.0014	0.073
H(25A)	0.6099	0.3429	-0.0483	0.056
H(25B)	0.6362	0.4453	-0.0777	0.056
H(25C)	0.5563	0.4365	-0.0367	0.056
H(26A)	0.8803	0.6392	0.2903	0.110
H(26B)	0.7867	0.6693	0.2820	0.110
H(26C)	0.8499	0.7262	0.2384	0.110
H(27A)	0.9541	0.5465	0.1641	0.072
H(27B)	0.9213	0.6360	0.1157	0.072
H(27C)	0.8986	0.5283	0.0906	0.072
H(28A)	0.8475	0.4413	0.2568	0.076
H(28B)	0.7877	0.4087	0.1888	0.076
H(28C)	0.7526	0.4645	0.2537	0.076
H(29A)	0.7046	1.1315	-0.4766	0.126
H(29B)	0.6999	1.0625	-0.4087	0.126
H(29C)	0.7607	1.1525	-0.4038	0.126
H(30A)	0.6279	1.3245	-0.4840	0.158

H(30B)	0.6847	1.3585	-0.4146	0.158
H(30C)	0.5883	1.3686	-0.4168	0.158
H(31A)	0.5234	1.1585	-0.4828	0.104
H(31B)	0.4834	1.1921	-0.4127	0.104
H(31C)	0.5240	1.0877	-0.4155	0.104
H(32A)	0.7537	1.5132	-0.2863	0.161
H(32B)	0.6862	1.4822	-0.2354	0.161
H(32C)	0.6743	1.4567	-0.3190	0.161
H(33A)	0.8685	1.3073	-0.1640	0.127
H(33B)	0.8071	1.3848	-0.1364	0.127
H(33C)	0.8760	1.4186	-0.1850	0.127
H(34A)	0.8594	1.2517	-0.3162	0.192
H(34B)	0.8734	1.3634	-0.3324	0.192
H(34C)	0.7992	1.3067	-0.3746	0.192
H(35A)	0.9755	1.1727	0.3267	0.149
H(35B)	1.0285	1.1273	0.2683	0.149
H(35C)	0.9586	1.0648	0.2989	0.149
H(36A)	0.9295	1.2330	0.2130	0.211
H(36B)	0.8597	1.1684	0.2427	0.211
H(37A)	0.9660	1.1114	0.1391	0.188
H(37B)	0.9170	1.0319	0.1797	0.188
H(38B)	0.8433	1.1856	0.0956	0.250
H(4AB)	0.7936	1.1084	0.1376	0.250
H(39A)	0.7871	1.0417	0.0276	0.213
H(39B)	0.8639	0.9863	0.0676	0.213
H(40A)	0.8972	1.0493	-0.0425	0.239
H(40B)	0.9515	1.0999	0.0223	0.239
H(40C)	0.8752	1.1555	-0.0172	0.239
H(35D)	0.9978	1.1367	0.3397	0.149
H(35E)	0.9164	1.1885	0.3061	0.149
H(35F)	0.9999	1.2067	0.2721	0.149
H(36C)	0.9962	1.0243	0.2198	0.211
H(36D)	0.9137	1.0036	0.2560	0.211
H(37C)	0.9326	1.1452	0.1557	0.188
H(37D)	0.8508	1.1203	0.1900	0.188
H(38C)	0.8420	0.9729	0.1313	0.250
H(38D)	0.9271	0.9918	0.1002	0.250
H(39C)	0.8096	1.1309	0.0703	0.213
H(39D)	0.7890	1.0298	0.0343	0.213
H(40D)	0.8238	1.1387	-0.0507	0.239
H(40E)	0.8897	1.0539	-0.0444	0.239
H(40F)	0.9109	1.1562	-0.0078	0.239