Supporting Information for

The unusual cis- and trans- architecture of dihydrofunctional double-decker shaped silsesquioxane and synthesis of its ethyl bridged π-conjugated arene derivatives

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1. General Considerations ••• S -2-
2. Measurements ••• S -2-
3. Synthetic procedures ••• S -3-
3.1 Synthesis and analysis (also XRD) of 9,19-di(hydro)octaphenyl double-decker silsesquioxane (DDSQ-2SiH) (1) ••• S -3-
3.2 Synthesis of styryl- and bis(styryl)arenes ••• S -6-
3.3 General Procedure for FT-IR in situ monitored hydrosilylation reaction ••• S -6-
3.4 General procedure for the synthesis of 9,19-bis(ethyl-aryl)-octaphenyl double-decker shaped silsesquioxanes - molecular systems (3a-d) ••• S -6-
3.5 General procedure for the synthesis of co-oligomeric aryl-ethyl- double-decker shaped silsesquioxanes - macromolecular systems (5a-d) ••• S -7-
4. Table of isolated compounds ••• S -8-
5. Spectroscopic analysis of obtained molecular (3a-d) products along with the copies of 1H, 13C, 29Si NMR, UV-VIS spectra ••• S -9-
6. Spectroscopic analysis of obtained macromolecular (5a-d) products along with the copies of 1H, 13C, 29Si NMR, UV-VIS spectra ••• S -21-
7. GPC Calibration Curve ••• S -33-
8. References ••• S -34-
1. General Considerations

The chemicals were obtained from the following sources: toluene, dichloromethane, tetrahydrofuran, potassium carbonate and trimethylamine were purchased from Fluka. Methanol and ethanol were purchased from POCH. DDSQ-4OH (TetraSilanolPhenyl POSS) was purchased from Hybrid Plastics. Methyldichlorosilane, 4-vinylplenyboronic acid, 1-bromo-naphtalene, bromopentafluorobenzene, bromobenzene, 9-bromoanthracene, 1,4-dibromobenzene, 1,4-dibromotetrafluorobenzene, 9,10-dibromoanthracene, 1,4-dibromo-naphtalene and Celite®, Karstedt’s catalyst (platinum(0)-1,3-divinyl-1,1,3,3-tetramethyldisiloxane complex [Pt(dvs)], solution in xylene with 2% of Pt), tetrais(triphenylphosphine) palladium(0) ([Pd(PPh)₃]₄) were purchased from Sigma-Aldrich Chemical Company (A Part of a Merck Group). The above mentioned solvents were stored over molecular sieves type 4Å. All liquid substrates were also dried and degassed by bulb-to-bulb distillation.

2. Measurements

**Nuclear magnetic resonance spectroscopy (NMR)**

1H and 13C NMR measurements were conducted using a spectrometer (Bruker Ultrashield 300 MHz), 29Si NMR spectra were recorded using spectrometer (BruckerAscend™ 400 MHz Nanobay) in CDCl₃.

**FT-IR spectroscopy**

Fourier Transform-Infrared (FT-IR) spectra were recorded on a Bruker Tensor 27 Fourier transform spectrophotometer that was equipped with a SPECAC Golden Gate, diamond ATR unit with a resolution of 2 cm⁻¹.

**Real-time FT-IR spectroscopy**

Real-time FT-IR measurements were performed on a Mettler Toledo ReactIR 15 equipped with a DS 6.3mm AgXDiComp Fiber Probe with a diamond sensor, and a Mercury Cadmium Telluride detector. For all the spectra 256 scans were recorded with the resolution of 1 cm⁻¹ in 1,2,5 and 10 min intervals.

**UV-vis spectroscopy**

The UV-visible spectra were recorded on NICOLET EVOLUTION 220 PC spectrophotometer, and baseline corrections and normalizations were carried out using INSIGHT software. HPLC pure grade CHCl₃ was used as a solvent.

**Gel Permeation Chromatography (GPC)**

GPC analyses were performed using a Waters Alliance 2695 system equipped with Waters 2414 RI detector and a set of three serially connected Waters HR columns (Waters Styragel HR1, HR2 and HR4). The measurements were carried out with THF as a mobile phase in a flow rate of 0.60 mL/min; column oven temperature was 35°C and detector temperature 40°C. All molecular weight (Mₙ, Mₓ) and polydispersity index (PDI) values were calculated based on calibration curve using polystyrene standards (Shodex) in a range from 1.31×10³ to 3.64×10⁶ Da.

**Thermogravimetric Analysis (TGA)**

TGA analyses were performed using a TA Instruments TGA Q50 thermal gravimetric analyzer. The measurements were conducted in nitrogen atmosphere (flow of 60 mL/min), from ambient temperature to 800°C at the heating rate of 10°C/min. The temperature of initial degradation (T_d) was taken as the onset temperature at which 5 wt% of mass loss occurs.

**Matrix-assisted ultraviolet laser desorption/ionization time-of-flight mass spectroscopy (MALDI-TOF-MS)**

MALDI-TOF mass spectra were recorded on a UltrafleXtreme mass spectrometer (Bruker Daltonics), equipped with a SmartBeam II laser (355 nm) in 500-4000 m/z range. 2,5-Dihydroxybenzoic acid (DHB, Bruker Daltonics, Bremen, Germany) served as matrix and was prepared in TA30 solvent (30:70 v/v acetonitrile: 0.1% TFA in water) at a concentration of 20 mg/mL. Studied samples were dissolved in dichloromethane (2 mg/mL) and then mixed in a ratio 1:1 v/v with matrix solution.
Matrix/sample mixtures (1 µL) were spotted onto the MALDI target and dried in air. Mass spectra were measured in reflection mode. The data were analyzed using the software provided with the Ultraflex instrument - FlexAnalysis (version 3.4). Mass calibration (cubic calibration based on five to seven points) was performed using external standards (Peptide Calibration Standard).

X-ray crystallography

Diffraction data were collected at 130(1) K by the ω-scan technique on Agilent Technologies four-circle Xcalibur diffractometer with Eos CCD detector and graphite-monochromated Mo Kα radiation (λ=0.71069 Å). The data were corrected for Lorentz-polarization as well as for absorption effects. Precise unit-cell parameters were determined by a least-squares fit of 6786 reflections of the highest intensity, chosen from the whole experiment. The structure was solved with SIR92 and refined with the full-matrix least-squares procedure on F² by SHELXL-2013. The scattering factors incorporated in SHELXL97 were used. All non-hydrogen atoms were refined anisotropically, hydrogen atoms were placed in idealized positions and refined as ‘riding model’ with isotropic displacement parameters set at 1.2 (1.5 for methyl groups) times Ueq of appropriate carrier atoms. There are two symmetry-independent halves of C8-symmetrical molecules of 1 in the asymmetric part of the unit cell. Interestingly, in both these molecules the Si-CH3 fragments are disordered. In molecule A the Si atom has been found in two alternative positions (s.o.f.’s refined at 69.7(5)% and 30.3(5)%), while in the molecule B the methyl groups are disordered over two positions with s.o.f.’s of 58.1% and 41.9%.

Crystallographic data (excluding structure factors) for the molecule B the methyl groups are disordered over two positions with s.o.f.’s of 58.1% and 41.9%. Crystallographic data (excluding structure factors) for the structural analysis has been deposited with the Cambridge Crystallographic Data Centre, Nos. CCDC-1481315. Copies of this information may be obtained free of charge from: The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK. Fax: +44(1223)336-033, e-mail:deposit@ccdc.cam.ac.uk, or www: www.ccdc.cam.ac.uk.

3. Synthetic Procedures

3.1.1 Synthesis of 9,19-di(hydro)octaphenyl double-decker silsesquioxane (DDSQ-2SiH) (1)

DDSQ-2SiH(1) was synthesized via well-known hydrolytic condensation of tetrasilanol form of silsesquioxane with methylidichlorosilane. Typically, DDSQ-4OH (10 g, 9.35 mmol) and Et3N (5.52 mL, 39.60 mmol) were added into two-neck round-bottom flask equipped with a magnetic stirrer, anhydrous THF (450 mL) was added with vigorous stirring. The reaction mixture was placed in ice-water bath and purged with highly pure argon. Then methylidichlorosilane (1.96 mL, 18.87 mmol) was added. Reaction was carried out for 24 hours at room temperature. Then insoluble solid of triethylammonium chloride was removed by filtration on a glass frit, volatiles and THF were eliminated via rotary evaporation. Crude product was dissolved in DCM and extracted three times with water. After solvent removal, the solid was dried in vacuo for 4 hours the product (8.2 g) was obtained with yield of 76%.

M.p. = 273°C. FT-IR (cm⁻¹) 3073, 3051 (C-Hphenyl); 2960 (Si-CH3), 2190 (Si-H), 1594 (C=CHphenyl), 1258 (Si-CH3), 1050, 1030 (Si-O), 997 (C-Hphenyl), 900, (Si-H). 1H NMR (ppm, CDCl3) 0.39 (d, 6H, JHH = 1.5 Hz, Si-CH3), 5.01(qu, 2H, JHH = 1.5 Hz, Si-H), 7.08-7.78 (m, 40H, C̃-H). 13C NMR (ppm, CDCl3) 0.63 (-CH3), 127.60; 130.40; 130.48; 131.57; 133.90 (C̃-CH3). 29Si NMR (ppm, CDCl3) -32.77, -77.80, -79.09, -79.28, -79.49

Crystal data:
1: C50H30O4Si10, Mr = 1151.77, triclinic, P-1, a = 10.5118(5) Å, b = 13.8528(8) Å, c = 19.5175(6) Å, α = 98.402(4)°, β = 90.407(3)°, γ = 90.950(4)°, V = 2811.1(2) Å³, Z=2, d=1.36 g.cm⁻³, F(000)=1196, μ=0.296 mm⁻¹, 19356 reflections collected, of which 9894 unique (Rint=0.032), 7282 with I>2σ(I), R(F) seals 0.047, wR(F²)(I>2σ(I))=0.125, R(F)[all data]=0.073, wR(F²)[all data]=0.136, S=1.06, max/min Δρ in the final ΔF map: 0.90/-0.53 e·Å⁻³.

$^1H$ NMR (CDCl₃, 400MHz):

$^{13}$C NMR (CDCl₃, 125 MHz):
$^{29}\text{Si NMR (CDCl}_3, 99 \text{ MHz):}$

Fragment of $^{29}\text{Si NMR spectrum of trans- di}[9,19- \text{hydro-methyl}]$-$1,3,5,7,11,13,15,17$-$\text{octa(phenyl)pentacyclo}[11.7.1.1^{5,11}.1^{5,17}.1^{7,15}]$-$\text{decasiloxane (1)}$
3.2 Synthesis of styryl- and bis(styryl)arenes
Styryl- and bis(styryl)arenes (2a-d and 4a-d) were obtained according to the literature. Typically, a mixture of aryl bromine and 4-vinylphenylboronic acid were placed in a two-necked round-bottom flask equipped with magnetic stirrer and condenser. Then, anhydrous toluene and ethanol (3:1) were added. The suspension was heated in oil bath at 60-90°C and catalyst ([Pd(PPh_3)_4], 1 mol%) and the solution of K_2CO_3 (2M) was added. After the reaction was completed (monitored by GCMS analysis) obtained mixture was filtered off by flash chromatography (silica gel 40, Celite®, DCM) to remove impurities from catalyst. Next DCM and other solvents were eliminated via rotary evaporation. Crude product was dissolved in DCM and extracted with water three times. After solvent removal, the solid was dried in vacuo for 4 hours, products (2a-d and 4a-d) were obtained with yield of 73-95%.

3.3 General Procedure for FT-IR in situ monitored hydrosilylation reaction
A mixture containing 0.2g (0.173mmol) of DDSQ and equimolar amount of bis(styryl)arene or 0.346 mmol of styrylarene and 2mL of toluene was placed in a glass reactor set (10mL) (described below) and heated at 90°C, then, the catalyst was added. Reaction mixture was stirred at 90°C and monitored by in-situ FT-IR spectroscopy to designate the endpoint of process. For quantitative description of DDSQ-2SiH conversion band at 907cm\(^{-1}\) derived from Si-H bond was analyzed.

3.4 General procedure for the synthesis of 9, 19-bis(ethyl-aryl)-octaphenyl double-decker shaped silsesquioxanes - molecular systems (3a-d)
To a two-neck round-bottom flask equipped with a magnetic stirrer, condenser and IR in-situ probe, DDSQ-2SiH (0.2 g, 0.173 mmol), an anhydrous toluene (2 mL) and stoichiometric amount of olefin (2 a-d) were added. The reaction mixture was heated up to 90°C and 2% solution of Karstedt’s catalyst in xylene (Pt(dvs) 5×10^{-4} – 10^{-3} mol per 1 mol of I was added. Reactions were carried out until the conversion of 1 was near to 100%, that was precisely specified due to IR in-situ spectroscopy. After that reaction mixture was filtered off by flash chromatography (silica gel 40, Celite®, DCM) to remove impurities from catalyst. If necessary (present of the unreacted olefin), product was purified by precipitation in methanol/DCM to afford 3(a-d) (76-82% yield).
3.5 General procedure for the synthesis of co-oligomeric aryl-ethyl- double-decker shaped silsesquioxanes - macromolecular systems (5a-d)

A two-neck round-bottom flask equipped with a magnetic stirrer and IR *in-situ* probe was charged with \( \text{1} \) (0.2 g, 0.173 mmol), anhydrous toluene (2 mL) and stoichiometric amount of bis(styryl)arene (4 a-d). After heated up to 90°C, 2% solution of Karstedt catalyst was added. Reactions were controlled by IR *in-situ* spectroscopy, that provided define substrate conversion and its real reaction time consequently. Polymeric products were obtained by precipitation in methanol/DCM. Obtained solids (5a-d) were isolated by decantation and dried under reduced pressure. The prepared polymers were characterized by \(^1\text{H}, ^{13}\text{C} \) and \(^{29}\text{Si} \) NMR spectroscopy as well as GPC and TGA analyses were performed.
4. Table of isolated compounds:

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<th>Structure</th>
<th>Compound #</th>
<th>NMR spectra page:</th>
<th>UV-Vis spectrum page:</th>
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<tr>
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</tr>
<tr>
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<td>5d</td>
<td>S -30-</td>
<td>S -32-</td>
</tr>
</tbody>
</table>
5. Spectroscopic analysis of obtained molecular (3a-d) products:

*trans*-di[9,19-(4-phenylethylbenzene)]-1,3,5,7,11,13,15,17-octaphenylpentacyclo[11.7.1.1^{3,11}.1^{5,17}.1^{7,15}]-decasiloxane (3a).

M.p. = 203°C. FT-IR (cm⁻¹): 3073, 3052, 3027 (C=Hphenyl), 1594, 1430 (C=Hphenyl), 1261 (Si-CH₃), 1050, 1027 (Si-O), 997 (C-Hphenyl), 804, 773 (Si-CH₂). ²H NMR (CDCl₃, 400 MHz): δ 0.30 (s, 6H, Si-CH₃), 1.13 (t, 2H, J_HH = 6 Hz, Si-CH₂), 2.73 (t, 2H, J_HH = 6 Hz, Ar-CH₂), 6.99-7.60 (m, 58 H, Ar-H). ¹³C NMR (CDCl₃, 125 MHz): δ 0.77 (-CH₃), 18.88 (-CH₂), 28.58 (-CH₂), 127.11; 127.99; 128.80; 131.06; 132.10; 133.98; 138.55; 141.34; 143.68 (C_Ar). ²⁹Si NMR (CDCl₃, 99 MHz): δ -18.20, -78.51, -79.49. MALDI-ToF MS (m/z): 1536.2 ([M + Na + H]+, calcd. for C₇₈H₇₃NaO₁₄Si₁₀: 1536.2)

¹H NMR (CDCl₃, 400MHz):

![NMR谱图](image-url)
$^{13}$C NMR (CDCl$_3$, 125 MHz):

$^{29}$Si NMR (CDCl$_3$, 99 MHz):
UV-visible absorption spectra of 1, 2a and 3a product in CHCl₃ solution:
trans- di[9,19-(4-(1-naphthyl)ethylbenzene)]-1,3,5,7,11,13,15,17-octaphenylpentacyclo-
M.p. = 137°C. FT-IR (cm\(^{-1}\)): 3072, 3048 (Si-P); 2961, 2924 (Si-CH\(_3\)), 1593, 1430 (C=C\(\text{phenyl}\)), 1260 (Si-CH\(_3\)), 1078, 1027 (Si-O), 997 (C-H\(\text{phenyl}\)), 799, 777 (Si-CH\(_2\)). \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta\) 0.42 (s, 6H, Si-CH\(_3\)), 1.19 (t, 2H, \(J_{HH} = 15\) Hz, Si-CH\(_2\)).2.78(t, 2H, \(J_{HH} = 48\) Hz, C\(\text{Ar}\)-CH\(_2\)).7.07-7.70 (m, 62 H, C\(\text{Ar}\)-H). \(^{13}\)C NMR (CDCl\(_3\), 125 MHz): \(\delta\) 1.07 (-CH\(_3\)), 15.60 (-CH\(_2\)), 28.77 (-CH\(_2\)), 125.19; 126.36; 127.00; 127.67; 127.73; 128.31; 130.32; 131.40; 133.97; 134.20 (C\(\text{Ar}\)). \(^{29}\)Si NMR (CDCl\(_3\), 99 MHz): \(\delta\) -18.38, -78.50, -79.51. MALDI-ToF MS (m/z): 1652.2 [M + K + H]\(^+\), calcd. for C\(_{86}\)H\(_{77}\)KO\(_{14}\)Si\(_{10}\): 1652.2

\(^1\)H NMR (CDCl\(_3\), 400MHz):

![NMR spectrum](image)
$^{13}$C NMR (CDCl$_3$, 125 MHz):

$^{29}$Si NMR (CDCl$_3$, 99 MHz):
UV-visible absorption spectra of 1, 2b and 3b product in CHCl₃ solution:
cis- and trans- mixture of di[9,19-(4-(9-anthracenyl)ethylbenzene]-1,3,5,7,11,13,15,17-octaphenylpentacyclo[11.7.1.1<sup>3,11</sup>.1<sup>5,17</sup>.1<sup>7,15</sup>]decasiloxane (3c).

M.p. = 211°C. FT-IR (cm<sup>-1</sup>): 3074 (C-Hphenyl), 2962, 2927 (Si-CH3), 2851; 1595, 1430 (C=C phenyl), 1080, 1027 (Si-O), (Si-CH3), 998 (C-H phenyl), 798, 738 (Si-CH2). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz): δ 0.49 (s, 6H, Si-CH<sub>3</sub>), 1.32 (t, 2H, <i>J</i> <sub>HH</sub> = 21 Hz, Si-CH<sub>2</sub>), 2.95 (t, 2H, <i>J</i> <sub>HH</sub> = 15 Hz, C<sub>Ar</sub>-CH<sub>2</sub>-), 6.99-8.00 (m, 66H, C<sub>Ar</sub>-H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ 1.07 (-CH<sub>3</sub>), 15.34 (-CH<sub>2</sub>), 28.71 (-CH<sub>2</sub>-), 127.71; 127.86; 128.18; 130.06; 130.43; 134.01; 145.61 (C<sub>Ar</sub>). <sup>29</sup>Si NMR (CDCl<sub>3</sub>, 99 MHz): δ -18.03, -78.52, -79.39, -79.48, -79.58. MALDI-ToF MS (m/z): 1751.2 [M + K]<sup>+</sup>, calcd. for C<sub>94</sub>H<sub>80</sub>KO<sub>14</sub>Si<sub>10</sub>: 1751.2. 

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400MHz):

![NMR spectrum image]
$^{13}$C NMR (CDCl$_3$, 125 MHz):

$^{29}$Si NMR (CDCl$_3$, 99 MHz):

UV-visible absorption spectra of 1, 2c and 3c product in CHCl$_3$ solution:
cis- and trans- mixture of di[9,19-(4-(2,3,4,5,6-penta-fluorophenyl)ethylbenzene)]-1,3,5,7,11,13,15,17-octaphenyl-penta-cyclo-[11.7.1.1^{3,11}.1^{5,17}.1^{7,15}]decasiloxane (3d).

M.p. = 203°C. FT-IR (cm\(^{-1}\)): 3073, 3051 (C-H\(_{\text{phenyl}}\)), 2960 (Si-CH\(_3\)), 1594, 1430 (C=C\(_{\text{phenyl}}\)), 1258 (Si-CH\(_3\)), 1050, 1030 (Si-O), 997 (C-H\(_{\text{phenyl}}\)) 795, (Si-CH\(_3\)). \(^1\)H NMR (CDCl\(_3\), 400 MHz): δ0.32, 0.41 (s, 6H, Si-C\(_{\text{H}}\)\(_3\)), 1.25 (t, 2H, J\(_{HH}\) = 15 Hz, Si-CH\(_2\)), 2.86 (t, 2H, J\(_{HH}\) = 18 Hz, C\(_A\)-CH\(_2\)), 7.13-7.68 (m, 48H, C\(_A\)-H).

\(^{13}\)C NMR (CDCl\(_3\), 125 MHz): δ 1.05 (-CH\(_3\)), 18.47 (-CH\(_2\)), 28.69 (-CH\(_2\)), 127.58; 128.19; 129.93; 130.43; 133.91; 134.06; 145.34 (C\(_A\)). \(^{29}\)Si NMR (CDCl\(_3\), 99 MHz): δ -18.03, -78.52, -79.36, -79.44, -79.63. MALDI-ToF MS (m/z): 1716.1 [M + Na]\(^+\), calcd. for C\(_{78}\)H\(_{63}\)F\(_{10}\)NaO\(_{14}\)Si\(_{10}\): 1716.1

\(^1\)H NMR (CDCl\(_3\), 400MHz):

![1H NMR spectrum](image)
$^{13}$C NMR (CDCl$_3$, 125 MHz):

$^{29}$Si NMR (CDCl$_3$, 99 MHz):

silicone grease
UV-visible absorption spectra of 1, 2d and 3d product in CHCl₃ solution:
6. Spectroscopic analysis of obtained macromolecular (5a-d) products

oligo[[9,19-(4-phenylethylbenzene)-1,3,5,7,11,13,15,17-octaphenylpentacyclo-
[11.7.1.1\(^{3,11}\).1\(^{5,17}\).1\(^{7,15}\)]decasiloxane]s (5a).

M.p. = 247 °C. \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta\) 0.27, 0.32, 0.37, 0.40 (s, Si-CH\(_3\)), 1.19 (m, Si-CH\(_2\)), 2.82 (m, C\(_{Ar}\)-CH\(_2\)), 7.11-7.61 (m, C\(_{Ar}\)-H). \(^{13}\)C NMR (CDCl\(_3\), 125 MHz): \(\delta\) 1.07 (-CH\(_3\)), 15.34 (-CH\(_2\)), 28.71 (-CH\(_2\)), 127.67, 127.86, 128.27, 130.07, 130.47, 133.90, 133.97, 145.61, (C\(_{Ar}\)). \(^{29}\)Si NMR (CDCl\(_3\), 99 MHz): \(\delta\) -18.39, -78.50, -79.54

\(^1\)H NMR (CDCl\(_3\), 400MHz):

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{spectrum}
\caption{NMR spectrum of the compound.}
\end{figure}
$^{13}C$ NMR (CDCl$_3$, 125 MHz):

$^{29}$Si NMR (CDCl$_3$, 99 MHz):
UV-visible absorption spectra of 1, 4a and 5a product in CHCl₃ solution:
oligo[{9,19-(4-(1-naphthyl)ethylbenzene)]-1,3,5,7,11,13,15,17-
octaphenylpentacyclo[11.7.1.1^{3,11}.1^{5,17}.1^{7,15}]decasiloxanes (5b).
M.p. = 254 °C. $^1$H NMR (CDCl$_3$, 400 MHz): δ 0.23, 0.28 (s, Si-CH$_3$), 1.15 (m, Si-CH$_2$), 2.76 (m, C$_{Ar}$-CH$_2$), 7.08-7.60 (m, C$_{Ar}$-H). $^{13}$C NMR (CDCl$_3$, 125 MHz): δ 1.06 (-CH$_3$), 14.59, 15.44 (-CH$_2$), 28.69, 29.73 (-CH$_2$), 123.50, 127.54, 127.93, 130.56, 133.87, 134.23, 140.38, (C$_{Ar}$). $^{29}$Si NMR (CDCl$_3$, 99 MHz): δ -18.12, -78.47, -79.46

$^1$H NMR (CDCl$_3$, 400MHz):
$^{13}$C NMR (CDCl₃, 125 MHz):

$^{29}$Si NMR (CDCl₃, 99 MHz):
UV-visible absorption spectra of 1, 4b and 5b product in CHCl$_3$ solution:
oligo[[9,19-(4-(9-anthracenyl)ethylbenzene)]-1,3,5,7,11,13,-15,17-octaphenylpentacyclo-[11.7.1.1^{3,11}.1^{5,17}.1^{7,15}]decasiloxane]s (5c).

M.p. = 340 °C. $^1$H NMR (CDCl$_3$, 400 MHz): $\delta$ 0.07, 0.32, 0.37, 0.40 (s, Si-CH$_3$), 1.19 (m, Si-CH$_2$-), 2.82 (m, C$_{Ar}$-CH$_2$-), 7.11-7.61 (m, C$_{Ar}$-H). $^{13}$C NMR (CDCl$_3$, 125 MHz): $\delta$ 0.79 (-CH$_3$), 18.78 (-CH$_2$), 29.85 (-CH$_2$-), 123.49, 127.73, 127.87, 130.19, 130.50, 130.81, 133.98, 140.39 (C$_{Ar}$). $^{29}$Si NMR (CDCl$_3$, 99 MHz): $\delta$ -18.32, -78.47, -79.39.

$^1$H NMR (CDCl$_3$, 400MHz):
$^{13}$C NMR (CDCl$_3$, 125 MHz):

$^{29}$Si NMR (CDCl$_3$, 99 MHz):
UV-visible absorption spectra of 1, 4c and 5c product in CHCl₃ solution:
poly[[9,19-(4-(2,3,4,5,6-penta-fluorophenyl)ethylbenzene)]-1,3,5,7,11,13,15,17-octaphenylpentacyclo-[11.7.1.1³⁷.1⁷.1⁵.1⁷]decasiloxanes (5d).

M.p. = 254 °C. $^1$H NMR (CDCl$_3$, 400 MHz): δ 0.27, 0.31, 0.37, 0.40 (s, Si-CH$_3$), 1.19 (m, Si-CH$_2$), 2.82 (m, CAr-CH$_2$), 6.90-7.79 (m, CAr-H). $^{13}$C NMR (CDCl$_3$, 125 MHz): δ 0.92 (-CH$_3$), 14.58 (-CH$_2$), 28.69 (-CH$_2$), 123.49, 127.73, 127.87, 130.19, 130.50, 130.81, 133.98, 140.39, (CAr). $^{29}$Si NMR (CDCl$_3$, 99 MHz): δ -18.32, -78.45, -79.46

$^1$H NMR (CDCl$_3$, 400 MHz):
$^{13}$C NMR (CDCl$_3$, 125 MHz):

$^{29}$Si NMR (CDCl$_3$, 99 MHz):
UV-visible absorption spectra of 1, 4d and 5d product in CHCl₃ solution:
7. GPC Calibration Curve
8. References


