RSC Advances

Rational Synthesis of Bis(hexyloxy)-Tetra(hydroxy)-Triphenylenes and their Derivatives.

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SUPPORTING INFORMATION
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I. General Synthetic Procedures

*General Methods:* Chemicals were purchased from Aldrich, Strem, Acros, TCI America, or Cambridge Isotope Labs and used as received. Solvents were dried using an Innovative Technologies SPS-400-5 solvent purification system. Thin layer chromatography (TLC) was performed on alumina-backed sheets coated with silica gel 60 F254. TLC plates were visualized using a UV/Vis lamp and/or by staining with iodine or p-anisaldehyde solution. Column chromatography was performed using glass columns over Dynamic Absorbents 60 Å, 32-63 μm silica gel. Melting points were determined on a Mettler Toledo Mel-Temp II melting point apparatus and are uncorrected. UV/Vis spectroscopy was recorded on a Varian Cary 100 Bio UV-Visible spectrophotometer. All $^1$H and $^{13}$C NMR spectra were recorded with a Varian Mercury (300 MHz and 75 MHz, respectively) or Varian Unity Plus (400 MHz and 100 MHz, respectively) spectrometer using residual solvent as the internal standard. All chemical shifts are quoted using the $\delta$ scale and all coupling constants are expressed in Hertz (Hz). Accurate mass EI/GCMS and ESI/APCI mass spectrometric analysis of compounds 1-6, 9-10, 13-14, 16-18, 20-23, and 26 were performed at the UC Riverside Mass Spec Facility. Differential scanning calorimetry was recorded with a TA DSC Q20 equipped with a TA refrigerated cooling system 90. Compounds 7, 8, 11, 12, 15, 19, and 24 were prepared according to literature procedures 1-7.
II. Synthesis of Compounds

**Compound 13:** Under an inert atmosphere, Compound 12 (1.5g, 5.49 mmol) was dissolved in dimethylformamide (5.5 mL) and diisopropylethylamine (1.5 mL, 8.79 mmol) was added dropwise. The solution was stirred for 15 minutes, tertbutyldimethylsilylchloride (1.2 g, 8.24 mmol) was added, and the solution stirred overnight. Water was added (20 mL) and the crude product extracted with hexanes (3x15mL). The combined organic extracts were washed with brine (30mL), dried over MgSO4, and concentrated under reduced pressure. The crude material was purified by column chromatography, eluting with 10% dichloromethane in hexanes, to afford the pure product (1.5 g, 70%) as a colorless oil. El/GCMS (m/z) [M]+ calculated for C18H31O2SiBr, 386.1271; found 386.1282. 

\[ \begin{array}{c}
\text{Br} \\
\text{OC}_{6}H_{13} \\
\text{OTBDMS}
\end{array} \]

\[ \delta 7.03-6.92 \text{ (m, 2H), 6.73 (d, } J = 8.8 \text{ Hz, 1H), 3.92 (t, } J = 6.45 \text{ ppm, 2H), 1.87-1.78 \text{ (m, 2H), 1.58-1.43 \amp; 1.43-1.30 \text{ (m, 4H), 1.03 (s, 9H), 0.94 (t, } J = 5.7 \text{, 3H), 0.18 (s, 6H) ppm.} \]

\[ \begin{array}{c}
\text{Br} \\
\text{OH} \\
\text{OC}_{6}H_{13}
\end{array} \]

\[ \delta 7.08 \text{ (d, } J = 2.4 \text{ Hz, 1H), 6.95 (dd, } J = 8.7, 2.5 \text{ Hz, 1H), 6.71 (d, } J = 8.8 \text{ Hz, 1H), 5.67 (s, 1H), 4.02 (t, } J = 6.7 \text{, 2H), 1.86-1.77 \text{ (m, 2H), 1.49-1.40 \amp; 1.38-1.30 \text{ (m, 4H), 0.92 (t, } J = 7.3 \text{ Hz, 3H) ppm.} \]

**Compound 16:** To a solution of 15 (2.3 g, 8.01 mmol) in dichloromethane (52mL) was added meta-chloroperoxybenzoic acid (2.6 g, 14.9 mmol) in small portions, and the solution stirred at 40°C overnight. A 2M solution of ammonia in methanol (12.1 mL) was added, and the mixture was stirred for 2 hours. Saturated sodium bicarbonate was added (30 mL), and the product was extracted with diethyl ether (3 x 30mL). The combined organic layers were washed with saturated sodium bicarbonate (100mL) and brine (100 mL), dried over MgSO4, and concentrated in vacuo to afford the pure product, isolated as an off-white solid (2.2 g, 99%). Mp = 41.8-42.6°C. El/GCMS (m/z) [M]+ calculated for C12H17O2Br, 272.0406; found 272.0339. 

\[ \delta 7.08 \text{ (d, } J = 2.4 \text{ Hz, 1H), 6.95 (dd, } J = 8.7, 2.5 \text{ Hz, 1H), 6.71 (d, } J = 8.8 \text{ Hz, 1H), 5.67 (s, 1H), 4.02 (t, } J = 6.7 \text{, 2H), 1.86-1.77 \text{ (m, 2H), 1.49-1.40 \amp; 1.38-1.30 \text{ (m, 4H), 0.92 (t, } J = 7.3 \text{ Hz, 3H) ppm.} \]

\[ \begin{array}{c}
\text{Br} \\
\text{OH} \\
\text{OC}_{6}H_{13}
\end{array} \]

\[ \delta 7.08 \text{ (d, } J = 2.4 \text{ Hz, 1H), 6.95 (dd, } J = 8.7, 2.5 \text{ Hz, 1H), 6.71 (d, } J = 8.8 \text{ Hz, 1H), 5.67 (s, 1H), 4.02 (t, } J = 6.7 \text{, 2H), 1.86-1.77 \text{ (m, 2H), 1.49-1.40 \amp; 1.38-1.30 \text{ (m, 4H), 0.92 (t, } J = 7.3 \text{ Hz, 3H) ppm.} \]

\[ \begin{array}{c}
\text{Br} \\
\text{OH} \\
\text{OC}_{6}H_{13}
\end{array} \]
Compound 17: Under an inert atmosphere, Compound 16 (541 mg, 1.98 mmol) was dissolved in dimethylformamide (2 mL) and diisopropylethylamine (0.54 mL, 3.17 mmol) was added dropwise. The solution was stirred for 15 minutes, tertbutlydimethylsilylchloride (448 mg, 2.97 mmol) was added, and the solution stirred overnight. Water was added (10 mL) and the crude product extracted with hexanes (3x10mL). The combined organic extracts were washed with brine (20mL), dried over MgSO₄, and concentrated under reduced pressure. The crude material was subjected to high vacuum to remove volatiles and afford the pure product (725 mg, 95%) as a yellow oil. El/GCMS (m/z) [M]+ calculated for C₁₈H₃₁O₂SiBr, 386.1271; found 386.1258. ¹H NMR (CDCl₃, 300 MHz): δ 6.96 (d, J = 2.5 Hz, 1H), 6.91 (dd, J = 8.2, 2.4 Hz, 1H), 6.70 (d, J = 8.5, 1H), 3.90 (t, J = 7.0 Hz, 2H), 1.85-1.76 (m, 2H), 1.52-1.42 (m, 2H), 1.36-1.28 (m, 4H), 1.00 (s, 9H), 0.89 (t, J = 7.6 Hz, 3H), 0.15 (s, 6H) ppm. ¹³C NMR (CDCl₃, 75 MHz): 150.03, 145.83, 124.37, 124.01, 113.93, 111.94, 68.61, 31.61, 29.28, 25.78, 25.62, 22.61, 18.33, 14.04, -4.70 ppm.

General procedure to prepare aryl pinacolboranes from aryl halides.

A mixture of aryl halide, bis(acetonitrile)dichloropalladium (II) (1 mol%), and Sphos Buchwald ligand (4 mol %) was prepared in a pressure flask, and immediately subjected to a vacuum/N₂ cycle (3x). To the solids was added dry 1,4-dioxane (1.7M with respect to aryl halide), and dry triethylamine (1.5 equivalents) under N₂. Last, pinacolborane (1.5 equivalents) was added quickly and the flask capped tightly. The mixture was stirred at 100°C until the reaction mixture darkened and thickened (around 3 hours). The mixture was allowed to cool, diluted with diethyl ether, and filtered over a pad of Celite. The filtrate was concentrated under reduced pressure, and the crude material purified by column chromatography.

Compound 14: Reaction scale: Compound 13 (1.5 g, 3.87 mmol). The product eluted from the column with 20% dichloromethane in hexanes, and was isolated as a yellow oil (1.28 g, 76%). ESI/APCI (m/z) [MH]^+ calculated for C₂₄H₄₄¹¹BO₃Si, 435.3096; found 435.3110. ¹H NMR (CDCl₃, 300 MHz): δ 7.32-7.27 (m, 1H), 6.35 (d, J = 7.9 Hz, 1H), 3.99 (t, J = 6.9, 1H), 1.90-1.76 (m, 2H), 1.51-1.45 (m, 2H), 1.34 (s, 12H), 1.31-1.18 (m, 4H), 1.01 (s, 9H), 0.91 (t, J = 6.9 Hz, 3H), 0.17 (s, 6H) ppm. ¹³C NMR (CDCl₃, 75 MHz): 150.20, 147.94, 128.00, 120.63, 118.48, 83.53, 68.33, 31.65, 29.48, 25.84, 25.70, 25.67, 24.86, 22.62, 18.42, 14.07, -4.61 ppm.
**Compound 18**: Reaction Scale: Compound 17 (355 mg, 0.916 mmol). The product eluted from the column with 30% dichloromethane in hexanes, and was isolated as a yellow solid (312 mg, 78%). Mp = 69.2 - 71.1°C. ESI/APCI (m/z) [MH]+ calculated for C_{24}H_{44}^{13}BO_4Si, 435.3096; found 435.3104. ^1H NMR (CDCl₃, 300 MHz): δ 7.38 (dd, J = 8.1, 1.6 Hz, 1H), 7.27-7.24 (m, 1H), 6.84 (d, J = 8.2 Hz, 1H), 3.96 (t, J = 6.7 Hz, 2H), 1.87-1.76 (m, 2H), 1.52-1.42 (m, 2H), 1.33 (s, 12H), 1.30-1.23 (m, 4H), 1.01 (s, 9H), 0.91 (t, J = 6.6 Hz, 3H), 0.17 (s, 6H) ppm. ^13C NMR (CDCl₃, 75 MHz): 159.29, 144.16, 129.20, 126.90, 111.85, 83.38, 68.15, 31.62, 29.28, 25.78, 25.73, 25.71, 24.81, 22.59, 18.36, 14.03, -4.59 ppm.

**Compound 26**: Hexakis(monomethyl di(ethylene glycol)) triphenylene: To a mixture of 2,3,6,7,1011-hexahydroxytriphenylene (105 mg, 0.324 mmol), potassium carbonate (447 mg, 3.24 mg), and catalytic 18-C-6 was added dimethylformamide (3.2 mL) and di(ethylene glycol) monomethyl ether tosylate (710 mg, 2.59 mmol) under nitrogen. The reaction solution was stirred at 80°C overnight. The solution was allowed to cool, and water was added. Excess di(ethylene glycol) monomethyl ether tosylate was extracted from the aqueous layer with diethyl ether (2x). The combined ethereal extracts were washed with aqueous hydrochloric acid (1M), and the aqueous layers combined. The product was extracted from the aqueous phase with ethyl acetate (3x), and the ethyl acetate phase washed with brine, dried over MgSO₄, and concentrated under reduced pressure to afford analytically pure product (183 mg, 30%). The produt was isolated as a dark oil which gradually solidified. ESI/APCI (m/z) [MNa]+ calculated for C_{48}H_{72}O_{18}Na, 959.4611; found 959.4634. ^1H NMR (300 MHz, CDCl₃): δ 7.88 (s, 6H), 4.42 (t, J = 4.6 Hz, 12H), 4.01 (t, J = 4.4 Hz, 12H), 3.80-3.83 (m, 12H), 3.61-3.63 (m, 12H), 3.41 (s, 18H) ppm. ^13C NMR (CDCl₃, 75 MHz): 148.41, 123.62, 107.61, 71.76, 70.54, 69.64, 68.89, 58.81 ppm.
III. $^1$H and $^{13}$C NMR Spectra

Compound 1.
Compound 2

\[ \text{C}_6\text{H}_{13}\text{O}-\text{H}_2\text{O}-\text{OC}_6\text{H}_{13} \]

**CD}_3\text{COCD}_3**

298 K

300 MHz (\text{^1H})

75 MHz (\text{^13C})
Compound 3

CD$_3$COCD$_3$
298 K
300 MHz (H)
75 MHz (C)

Chemical Shift (ppm)

Normalized Intensity

Chemical Shift (ppm)
Compound 4

CDCl₃
298 K
300 MHz (¹H)
75 MHz (¹³C)

Chemical Shift (ppm)

Normalized Intensity

Chemical Shift (ppm)
Compound 5

CDCl₃
298 K
300 MHz (¹H)
75 MHz (¹³C)
Compound 6

Chemical Shift (ppm)

Normalized Intensity

CDCl₃
298 K
300 MHz (¹H)
75 MHz (¹³C)
Compound 9

CDCl₃
298 K
300 MHz (¹H)
75 MHz (¹³C)
Compound 10

[Chemical structure image]

CDCl₃
298 K
300 MHz (^1H)
75 MHz (^13C)
Compound 13

Chemical Shift (ppm)

Normalized Intensity

Chemical Shift (ppm)

Normalized Intensity

CDCl₃
298 K
300 MHz (¹H)
75 MHz (¹³C)
Compound 14

[Chemical structure image]

CDCl$_3$
298 K
300 MHz (H)
75 MHz (C)
Compound 16

CDCl₃
298 K
300 MHz (¹H)
75 MHz (¹³C)
Compound 17

\[
\begin{align*}
\text{CDCl}_3 & \quad 298 \text{ K} \\
300 \text{ MHz } (^1\text{H}) & \quad 75 \text{ MHz } (^{13}\text{C})
\end{align*}
\]
Compound 18

Chemical Shift (ppm)

Normalized Intensity

CDCl₃
298 K
300 MHz (¹H)
75 MHz (¹³C)

Chemical Structure

OTBDMS
OC₆H₁₃
Compound 20

CDCl₃
298 K
300 MHz (¹H)
75 MHz (¹³C)
Compound 21

![NMR spectra of Compound 21](image)

- Chemical Shift (ppm)
  - Normalized Intensity

---

**CDCl₃**

- 298 K
- 300 MHz (¹H)
- 75 MHz (¹³C)
Compound 22

\begin{align*}
\text{Chemical Shift (ppm)} & \quad \text{Normalized Intensity} \\
& \quad 0.1 \quad 0.2 \quad 0.3 \quad 0.4 \quad 0.5 \quad 0.6 \quad 0.7 \quad 0.8 \quad 0.9 \quad 1.0 \\
& \quad 0.1 \quad 0.2 \quad 0.3 \quad 0.4 \quad 0.5 \quad 0.6 \quad 0.7 \quad 0.8 \quad 0.9 \quad 1.0
\end{align*}

\begin{align*}
\text{Chemical Shift (ppm)} & \quad \text{Normalized Intensity} \\
& \quad 200 \quad 180 \quad 160 \quad 140 \quad 120 \quad 100 \quad 80 \quad 60 \quad 40 \quad 20 \quad 0 \\
& \quad 200 \quad 180 \quad 160 \quad 140 \quad 120 \quad 100 \quad 80 \quad 60 \quad 40 \quad 20 \quad 0
\end{align*}
Compound 23

Chemical Shift (ppm)

Normalized Intensity

MKS-10-6

180 160 140 120 100 80 60 40 20 0

Chemical Shift (ppm)

Normalized Intensity

MKS-10-6

10 8 6 4 2 0

Normalized Intensity

Chemical Shift (ppm)

Normalized Intensity

180 160 140 120 100 80 60 40 20 0

Chemical Shift (ppm)

Normalized Intensity

10 8 6 4 2 0
Compound 26

CDCl₃
298 K
300 MHz (¹H)
75 MHz (¹³C)
DEPTs

**Compound 1 (processed CH up)**

[Graph showing the DEPT spectrum for Compound 1 with normalized intensity on the y-axis and chemical shift (ppm) on the x-axis.]

**Compound 2 (processed CH down)**

[Graph showing the DEPT spectrum for Compound 2 with normalized intensity on the y-axis and chemical shift (ppm) on the x-axis.]

**Compound 3 (processed CH down)**

[Graph showing the DEPT spectrum for Compound 3 with normalized intensity on the y-axis and chemical shift (ppm) on the x-axis.]
IV. Mass Spectrometric Data

Compound 1.
Compound 2

Measured Mass: 493.2583

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![Chemical Structure of Compound 2](image)
Compound 3

Measured Mass 493.2554

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Formula C30 H37 O6  Calculated Mass 493.2555  mDaError -0.1  ppmError -0.1  PDB 12.5

\[
\text{HO}\text{-}\begin{array}{c}
\text{HO}
\end{array}\begin{array}{c}
\text{HO}
\end{array}\text{HO}
\]

\[
\text{C}_8\text{H}_{13}\text{O} \quad \text{OC}_8\text{H}_{13}
\]
Compound 4

Produced a measured mass of 923.5148.

Element | Low Limit | High Limit
--- | --- | ---
C | 46 | 56
H | 70 | 95
O | 5 | 15
Na | 0 | 1

Formula | Calculated Mass | mDaError | ppmError | RDB
--- | --- | --- | --- | ---
C52 H75 O14 | 923.5181 | -0.3 | -0.4 | 15.5
C50 H76 O14 Na | 923.5127 | 2.1 | 2.2 | 12.5
Compound 5

Measured Mass: 323.5137

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\[
\text{H}_3\text{C(OC}_2\text{H}_4\text{)}_2\text{O} \quad \text{O(C}_2\text{H}_4\text{O)}_2\text{CH}_3 \\
\text{O}_6\text{H}_{13}\text{O} \quad \text{O}_6\text{H}_{13} \\
\text{H}_3\text{C(OC}_2\text{H}_4\text{)}_2\text{O} \quad \text{O(C}_2\text{H}_4\text{O)}_2\text{CH}_3
\]
Compound 6

![Graph with peaks labeled MN2+ and MNH4+]

**Measured Mass:** 923.5153

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![Chemical structure diagram]

H₃C(OC₂H₄)₂O       O(C₂H₄O)₂CH₃

H₃C(OC₂H₄)₂O       O(C₂H₄O)₂CH₃

C₆H₁₃O            OC₆H₁₃
Compound 9

![Mass Spectrogram](image)

**Measured Mass**: 951.613

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![Chemical Structure](image)
Compound 10

**Measured Mass**

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```
C6H13O
O\_C6H13

TBDMSO

OTBDMS

TBDMOS

OTBDMS
```
Compound 13

Measured Mass

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<td>0</td>
<td>90</td>
</tr>
<tr>
<td>N</td>
<td>0</td>
<td>2</td>
</tr>
<tr>
<td>O</td>
<td>0</td>
<td>4</td>
</tr>
<tr>
<td>Br</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>Si</td>
<td>0</td>
<td>1</td>
</tr>
</tbody>
</table>

Formula | Calculated Mass | mDa Error | ppm Error | RDB |
--------|-----------------|-----------|-----------|-----|
C16 H13 O2 Si Br | 366.1271 | 1.1 | 2.9 | 4 |
C22 H18 N2 O4 | 366.1261 | 2.1 | 5.4 | 16 |
C22 H17 O Br | 366.1240 | 4.2 | 10.9 | 9 |
C16 H19 N2 O2 Br | 366.1235 | -4.3 | -11.2 | 4.5 |
C25 H18 N2 Si | 366.1234 | 4.9 | 12.5 | 20 |
C20 H22 O3 Si | 366.1333 | -5.1 | -13.1 | 15 |
C13 H31 N2 O4 Si Br | 366.1231 | 5.1 | 13.2 | 0 |

Accurate mass done by ESI/GCMS for same reasons as sample FR3
again see expected M+
Compound 14

![Mass Spectroscopy Graph]

**Measured Mass**

<table>
<thead>
<tr>
<th>Element</th>
<th>Low Limit</th>
<th>High Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>19</td>
<td>29</td>
</tr>
<tr>
<td>H</td>
<td>35</td>
<td>55</td>
</tr>
<tr>
<td>O</td>
<td>2</td>
<td>6</td>
</tr>
<tr>
<td>N</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>SI</td>
<td>0</td>
<td>1</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Formula</th>
<th>Calculated Mass</th>
<th>mCalError</th>
<th>ppmError</th>
<th>RDB</th>
</tr>
</thead>
<tbody>
<tr>
<td>C\textsubscript{2}6\textsubscript{H}4\textsubscript{3}O\textsubscript{5}</td>
<td>435.3105</td>
<td>0.5</td>
<td>1.1</td>
<td>5.5</td>
</tr>
<tr>
<td>C\textsubscript{2}4\textsubscript{H}4\textsubscript{4}N\textsubscript{1}B\textsubscript{4}O\textsubscript{4}SI</td>
<td>435.3096</td>
<td>1.4</td>
<td>3.1</td>
<td>4.6</td>
</tr>
</tbody>
</table>

\[
\begin{align*}
\text{OTBDMS} & \\
\text{OC}_6\text{H}_{13} & \\
\text{B} & \\
\end{align*}
\]
Compound 16

Accurate mass done via EI/GCMS and shows expected M+

\[
\begin{align*}
\text{Br} & \quad \text{OH} \\
& \quad \text{O}_6\text{H}_{13}
\end{align*}
\]
Compound 17

![Graph with molecular structure]

Table of measured mass and elemental analysis:

<table>
<thead>
<tr>
<th>Element</th>
<th>Low Limit</th>
<th>High Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>2</td>
<td>60</td>
</tr>
<tr>
<td>H</td>
<td>0</td>
<td>90</td>
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<tr>
<td>N</td>
<td>0</td>
<td>2</td>
</tr>
<tr>
<td>O</td>
<td>0</td>
<td>4</td>
</tr>
<tr>
<td>Br</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>Si</td>
<td>0</td>
<td>1</td>
</tr>
</tbody>
</table>

Table of calculated mass, mDaPrmX, ppmEpmX, and RDB:

<table>
<thead>
<tr>
<th>Formula</th>
<th>Calculated Mass</th>
<th>mDaPrmX</th>
<th>ppmEpmX</th>
<th>RDB</th>
</tr>
</thead>
<tbody>
<tr>
<td>C23 H18 N2 O4</td>
<td>386.1261</td>
<td>-0.3</td>
<td>-0.8</td>
<td>16</td>
</tr>
<tr>
<td>C19 H31 O2 Si Br</td>
<td>386.1271</td>
<td>-1.3</td>
<td>5.4</td>
<td>6</td>
</tr>
<tr>
<td>C22 H27 O Br</td>
<td>386.1240</td>
<td>1.8</td>
<td>4.7</td>
<td>9</td>
</tr>
<tr>
<td>C26 H18 N2 Si</td>
<td>386.1234</td>
<td>2.4</td>
<td>6.3</td>
<td>20</td>
</tr>
<tr>
<td>C13 H31 N2 O4 Si Br</td>
<td>386.1231</td>
<td>2.7</td>
<td>7.0</td>
<td>0</td>
</tr>
<tr>
<td>C26 H18 O2</td>
<td>386.1301</td>
<td>-4.3</td>
<td>-11.2</td>
<td>20</td>
</tr>
<tr>
<td>C23 H20 N O3 Si</td>
<td>386.1207</td>
<td>5.1</td>
<td>13.2</td>
<td>15.5</td>
</tr>
</tbody>
</table>
Compound 18

\[
\begin{align*}
\text{Measured Mass} & \quad 435.3104 \\
\text{Element} & \quad \text{Low Limit} & \quad \text{High Limit} \\
C & \quad 19 & \quad 29 \\
H & \quad 35 & \quad 37 \\
O & \quad 2 & \quad 6 \\
11B & \quad 0 & \quad 1 \\
\text{SI} & \quad 0 & \quad 1 \\
\text{Formula} & \quad \text{Calculated Mass} & \quad \text{mDaError} & \quad \text{ppmError} & \quad \text{RDB} \\
C_{26}H_{43}O_{5} & \quad 435.3105 & \quad -0.1 & \quad -0.2 & \quad 5.5 \\
C_{24}H_{44}11B\text{O}_{4}\text{SI} & \quad 435.3096 & \quad 0.8 & \quad 1.7 & \quad 4.8 \\
\end{align*}
\]

\[
\begin{align*}
\text{Structure:}
\end{align*}
\]
Compound 20

Measured Mass: 951.62

<table>
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<tr>
<th>Element</th>
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<th>High Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>49</td>
<td>59</td>
</tr>
<tr>
<td>H</td>
<td>85</td>
<td>105</td>
</tr>
<tr>
<td>O</td>
<td>4</td>
<td>8</td>
</tr>
<tr>
<td>S</td>
<td>3</td>
<td>5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Formula</th>
<th>Calculated Mass</th>
<th>mDaError</th>
<th>ppmError</th>
<th>RDB</th>
</tr>
</thead>
<tbody>
<tr>
<td>C84 H80 O6 Si4</td>
<td>951.6200</td>
<td>0.0</td>
<td>0.0</td>
<td>11.5</td>
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<tr>
<td>C85 H81 O6 Si4</td>
<td>951.6169</td>
<td>3.1</td>
<td>3.3</td>
<td>16.5</td>
</tr>
<tr>
<td>C50 H99 O7 Si5</td>
<td>951.6232</td>
<td>-3.2</td>
<td>-3.3</td>
<td>6.5</td>
</tr>
</tbody>
</table>

```
  TBDMOSO  OTBDMS
     |       |
     |       |
     |       |
  C6H13O  TBDMOSO  OTBDMS
  C6H13O  C6H13
```
Compound 21

**Measured Mass**: 949.6031

<table>
<thead>
<tr>
<th>Element</th>
<th>Low Limit</th>
<th>High Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>46</td>
<td>56</td>
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<tr>
<td>H</td>
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<tr>
<td>O</td>
<td>5</td>
<td>15</td>
</tr>
<tr>
<td>Si</td>
<td>2</td>
<td>5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Formula</th>
<th>Calculated Mass</th>
<th>mDaError</th>
<th>ppmError</th>
<th>RDB</th>
</tr>
</thead>
<tbody>
<tr>
<td>C₅S H₆S O S₂</td>
<td>949.6040</td>
<td>-0.9</td>
<td>-0.9</td>
<td>13.5</td>
</tr>
<tr>
<td>C₅S H₆S O S₄</td>
<td>949.6044</td>
<td>-1.3</td>
<td>-1.3</td>
<td>12.5</td>
</tr>
<tr>
<td>C₅₁ H₆₅ O₁₀ S₃</td>
<td>949.6071</td>
<td>-4.0</td>
<td>-4.2</td>
<td>8.5</td>
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</table>

Diagram of Compound 21:
Compound 22

Measured Mass: 951.6196

<table>
<thead>
<tr>
<th>Element</th>
<th>Low Limit</th>
<th>High Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>49</td>
<td>59</td>
</tr>
<tr>
<td>H</td>
<td>85</td>
<td>105</td>
</tr>
<tr>
<td>O</td>
<td>4</td>
<td>8</td>
</tr>
<tr>
<td>Si</td>
<td>3</td>
<td>5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Formula</th>
<th>Calculated Mass</th>
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<th>ppmError</th>
<th>RD</th>
</tr>
</thead>
<tbody>
<tr>
<td>C54 H1506 Si4</td>
<td>951.5200</td>
<td>-0.4</td>
<td>-0.5</td>
<td>11.5</td>
</tr>
<tr>
<td>C58 H6105 Si3</td>
<td>951.6169</td>
<td>2.7</td>
<td>2.8</td>
<td>16.5</td>
</tr>
<tr>
<td>C50 H9907 Si5</td>
<td>951.6232</td>
<td>-3.6</td>
<td>-3.8</td>
<td>6.5</td>
</tr>
</tbody>
</table>

TBDMSO

OTBDM50

C6H13O

O2C6H13
Compound 23

Measured Mass: 949.5034

Element          | Low Limit | High Limit |
------------------|-----------|------------|
C                 | 49        | 59         |
H                 | 55        | 105        |
O                 | 4         | 5          |
S                 | 3         | 5          |

Formula | Calculated Mass | mDaError | ppmError | PDB
-------|-----------------|----------|----------|-----
C54 H36 O6 S4   | 949.5044      | -1.0     | -1.0     | 12.5
C58 H49 O6 S3   | 949.5012      | 2.2      | 2.3      | 17.5
C60 H57 O7 S5   | 949.4976      | -4.1     | -4.3     | 7.6
Compound 26

![Mass Spectrogram](image)

**Measured Mass**: 959.4634

<table>
<thead>
<tr>
<th>Element</th>
<th>Low Limit</th>
<th>High Limit</th>
</tr>
</thead>
<tbody>
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<td>55</td>
</tr>
<tr>
<td>H</td>
<td>70</td>
<td>95</td>
</tr>
<tr>
<td>O</td>
<td>16</td>
<td>20</td>
</tr>
<tr>
<td>Na</td>
<td>0</td>
<td>1</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
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<th>ppmError</th>
<th>RSD</th>
</tr>
</thead>
<tbody>
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<td>C50 H1 O18</td>
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<td>-0.1</td>
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<tr>
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<td>959.4611</td>
<td>2.3</td>
<td>2.4</td>
<td>12.5</td>
</tr>
</tbody>
</table>

![Chemical Structure](image)
V. UV/Vis & Fluorescence Spectroscopy

**Figure S1**: Overlaid UV/Vis spectra of triphenylene tetra-ols 1-3. All spectra were collected in THF (1.0x10⁻⁵ M) at 298 K.

**Figure S2**: Overlaid UV/Vis spectra of amphiphilic triphenylenes 4-6. All spectra were collected in THF (1.0x10⁻⁵ M) at 298 K.
VI. Differential Scanning Calorimetry

**Figure S3.** DSC cooling thermal diagram traces for **Compounds 4-6, 25,** and **26** at 5°C/min.

**Figure S4.** DSC second heating thermal diagram traces for **Compounds 4-6, 25,** and **26** at 10°C/min.
VII. Supporting References