Supplementary Information

Synthesis, Functionalization, and Isolation of Planar-Chiral Pillar[5]arenes with Bulky Substituents Using a Chiral Derivatization Agent

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**Single crystal X-ray diffraction data:**

**Table 1S.** Summary on the nature of the crystals and various crystallographic parameters of Pillar-1.

<table>
<thead>
<tr>
<th>Crystal Name</th>
<th>Pillar-1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crystal Dimension/mm</td>
<td>0.20 X 0.16 X 0.11</td>
</tr>
<tr>
<td>Crystal Color, Habit</td>
<td>Colorless, Block</td>
</tr>
<tr>
<td>Formula</td>
<td>C_{93}H_{144}Cl_{8}O_{10}</td>
</tr>
<tr>
<td>Crystal system</td>
<td>Orthorhombic</td>
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<tr>
<td>Space group(no.)</td>
<td>P b c n (60)</td>
</tr>
<tr>
<td>T/K</td>
<td>150</td>
</tr>
<tr>
<td>a/Å</td>
<td>21.8547(8)</td>
</tr>
<tr>
<td>b/Å</td>
<td>22.2502(9)</td>
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<tr>
<td>c/Å</td>
<td>20.4982(14)</td>
</tr>
<tr>
<td>α</td>
<td>90</td>
</tr>
<tr>
<td>β</td>
<td>90</td>
</tr>
<tr>
<td>γ</td>
<td>90</td>
</tr>
<tr>
<td>V/ Å³</td>
<td>9967.7(9)</td>
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<tr>
<td>Z</td>
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<tr>
<td>μ(CuKα) / mm⁻¹</td>
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<tr>
<td>ρ&lt;sub&gt;calc&lt;/sub&gt;/g cm⁻³</td>
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<tr>
<td>Unique reflections</td>
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</tr>
<tr>
<td>R&lt;sub&gt;int&lt;/sub&gt;</td>
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<tr>
<td>R (I &gt; 2σ)</td>
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<td>R (all data)</td>
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<tr>
<td>R&lt;sub&gt;ω&lt;/sub&gt;(all data)</td>
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<tr>
<td>Δρ&lt;sub&gt;max&lt;/sub&gt;e Å⁻³</td>
<td>0.627</td>
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</table>
Figure S1. Thermal Ellipsoid representation of the crystal structure Pilar-1. Color code Gray-carbon; red-oxygen; green –chlorine and black-hydrogen.

Figure S2. Presentation of the stacking patterns of Pillar-1 (a) side view and (b) top view (only the rigid equilateral pillar parts are shown for clarity).
Figure S3. $^1$H NMR spectra (600 MHz, chloroform-$d$, 25 °C) of Pilar-1 and OMA (a); 6.3 mM OMA (b); 1.25 mM Pilar-1 and 6.3 mM OMA (c); 2.52 mM Pilar-1 and 6.3 mM OMA (d); 3.78 mM Pilar-1 and 6.3 mM OMA (e); 5.04 mM Pilar-1 and 6.3 mM OMA.
Figure S4. Partial $^1$H-$^1$H COSY spectrum (600 MHz, chloroform-$d$, 25 °C) of Pilar-1 (6.3 mM) and OMA (6.3 mM).

Figure S5. Partial ROSEY spectrum (600 MHz, chloroform-$d$, 25 °C) of Pilar-1 (6.3 mM) and OMA (6.3 mM).
Figure S6. $^{1}$HNMR (600 MHz, CDCl$_3$) spectrum of 1,4-bis(neopentyloxy)benzene.

Figure S7. $^{13}$CNMR (150 MHz, CDCl$_3$) spectrum of 1,4-bis(neopentyloxy)benzene.
Figure S8. $^1$HNMR (600 MHz, CDCl$_3$) spectrum of 1-(benzyloxy)-4-(neopentyloxy)benzene.

Figure S9. $^{13}$CNMR (100 MHz, CDCl$_3$) spectrum of 1-(benzyloxy)-4-(neopentyloxy)benzene.
Figure S10. $^1$HNMR (600 MHz, CDCl$_3$) spectrum of Pillar-1.

Figure S11. $^{13}$CNMR (150 MHz, CDCl$_3$) spectrum of Pillar-1.
Figure S12. $^1$HNMR (600 MHz, CDCl$_3$) spectrum of Pillar-2.

Figure S13. $^{13}$CNMR (150 MHz, CDCl$_3$) spectrum of Pillar-2.
Figure S14. $^1$HNMR (600 MHz, CDCl$_3$) spectrum of Pillar-3.

Figure S15. $^{13}$CNMR (150 MHz, CDCl$_3$) spectrum of Pillar-3.
Figure S16. $^1$HNMR (600 MHz, CDCl$_3$) spectrum of Pillar-4a.

Figure S17. $^{13}$CNMR (150 MHz, CDCl$_3$) spectrum of Pillar-4a.
Figure S18. $^1$HNMR (600 MHz, CDCl$_3$) spectrum of Pillar-4b.

Figure S19. $^{13}$CNMR (150 MHz, CDCl$_3$) spectrum of Pillar-4b.
Figure S20. $^1$H-$^1$H COSY NMR (600 MHz, CDCl$_3$) spectrum of first fraction (Pillar-4a).

Figure S21. $^1$H-$^1$H COSY NMR (600 MHz, CDCl$_3$) spectrum of second fraction (Pillar-4b).
Figure S22. CD spectra of the first and second fractions (12 μLmol⁻¹cm⁻¹) in hexane at 25 °C.