

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 **Pilla-1**.

Crystal Name	Pillar-1
Crystal Dimension/mm	0.20 X 0.16 X 0.11
Crystal Color, Habit	Colorless, Block
Formula	C ₉₃ H ₁₄₄ Cl ₈ O ₁₀
Crystal system	Orthorhombic
Space group(no.)	P b c n (60)
T/K	150
a/Å	21.8547(8)
b//Å	22.2502(9)
c/Å	20.4982(14)
α	90
β	90
γ	90
V/ Å ³	9967.7(9)
Z	4
$\mu(\text{CuK}\alpha) / \text{mm}^{-1}$	0.277
$\rho_{\text{calcd}}/\text{g cm}^{-3}$	1.137
$\theta_{\text{max}}/\text{deg}$	25.030
Reflections collected	54825
Unique reflections	8766
R _{int}	0.0720
R (I > 2 σ)	0.0767
R (all data)	0.1201
R _w (all data)	0.2499
$\Delta \rho _{\text{max}} \text{e } \text{Å}^{-3}$	0.627

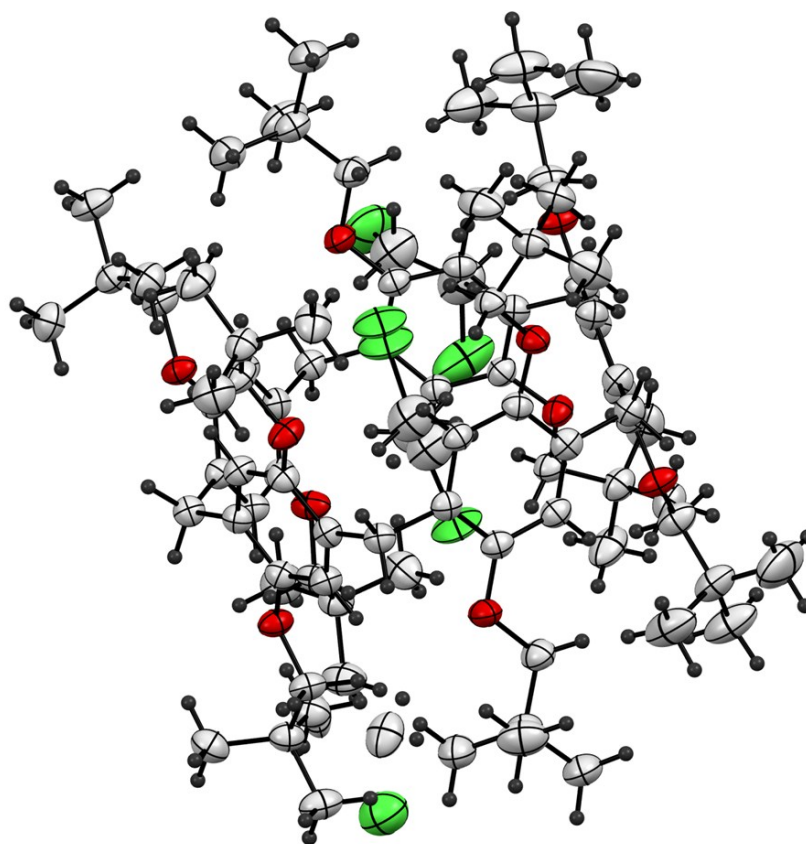


Figure S1. Thermal Ellipsoid representation of the crystal structure **Pillar-1**. Color code Gray-carbon; red-oxygen; green –chlorine and black-hydrogen.

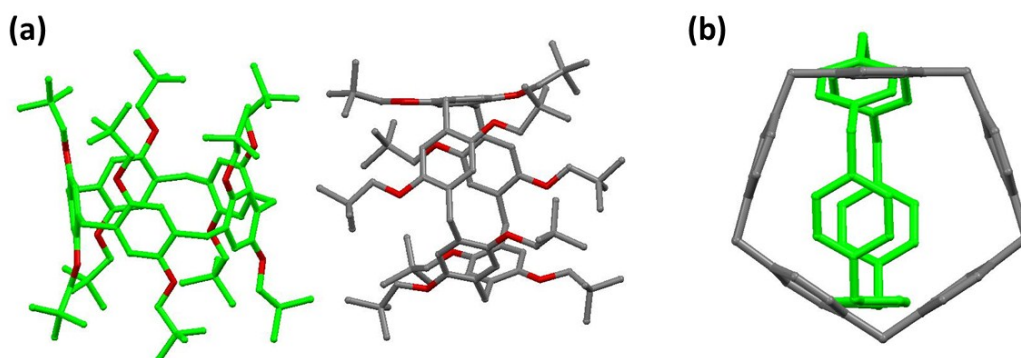


Figure S2. Presentation of the stacking patterns of **Pillar-1** (a) side view and (b) top view (only the rigid equilateral pillar parts are shown for clarity).

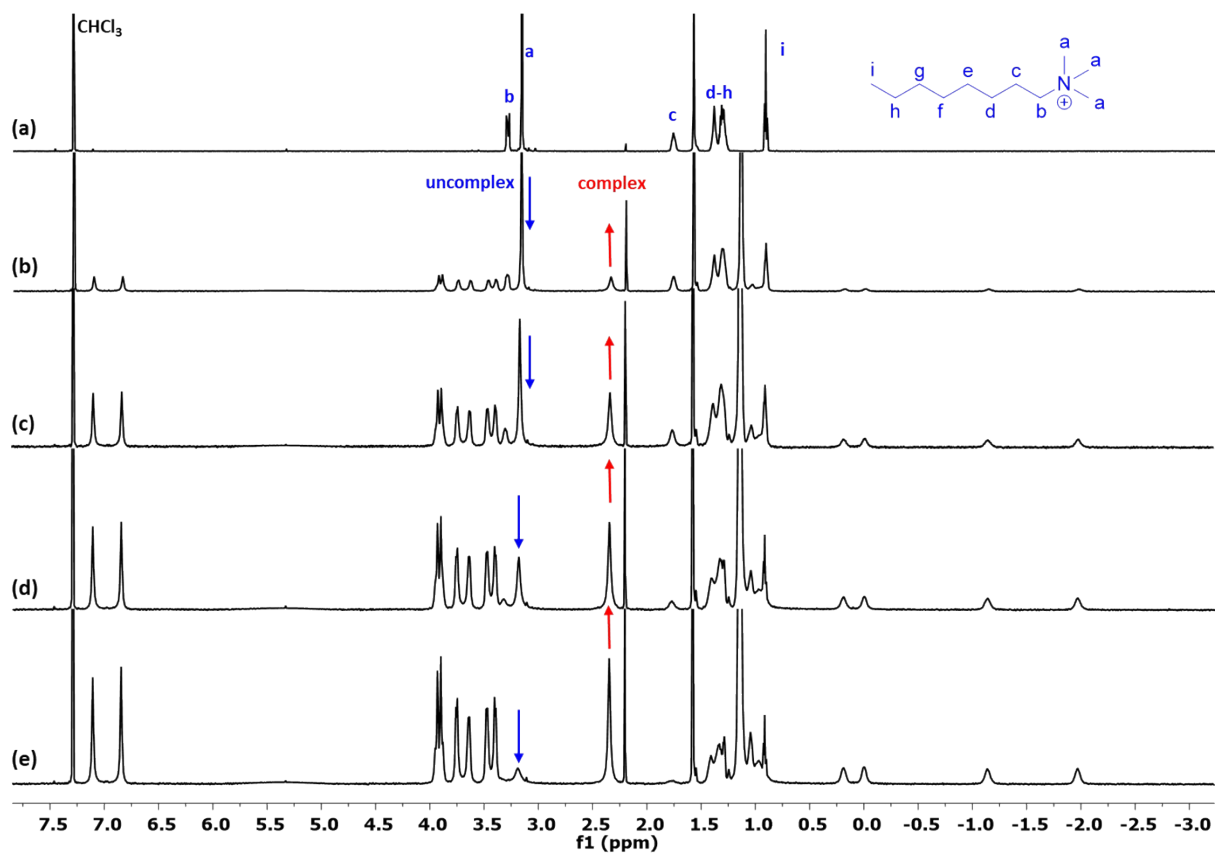


Figure S3. ^1H NMR spectra (600 MHz, chloroform-*d*, 25 $^\circ\text{C}$) of **Pilar-1** and **OMA** (a); 6.3 mM **OMA** (b); 1.25 mM **Pilar-1** and 6.3 mM **OMA** (c); 2.52 mM **Pilar-1** and 6.3 mM **OMA** (d); 3.78 mM **Pilar-1** and 6.3 mM **OMA** (e); 5.04 mM **Pilar-1** and 6.3 mM **OMA**.

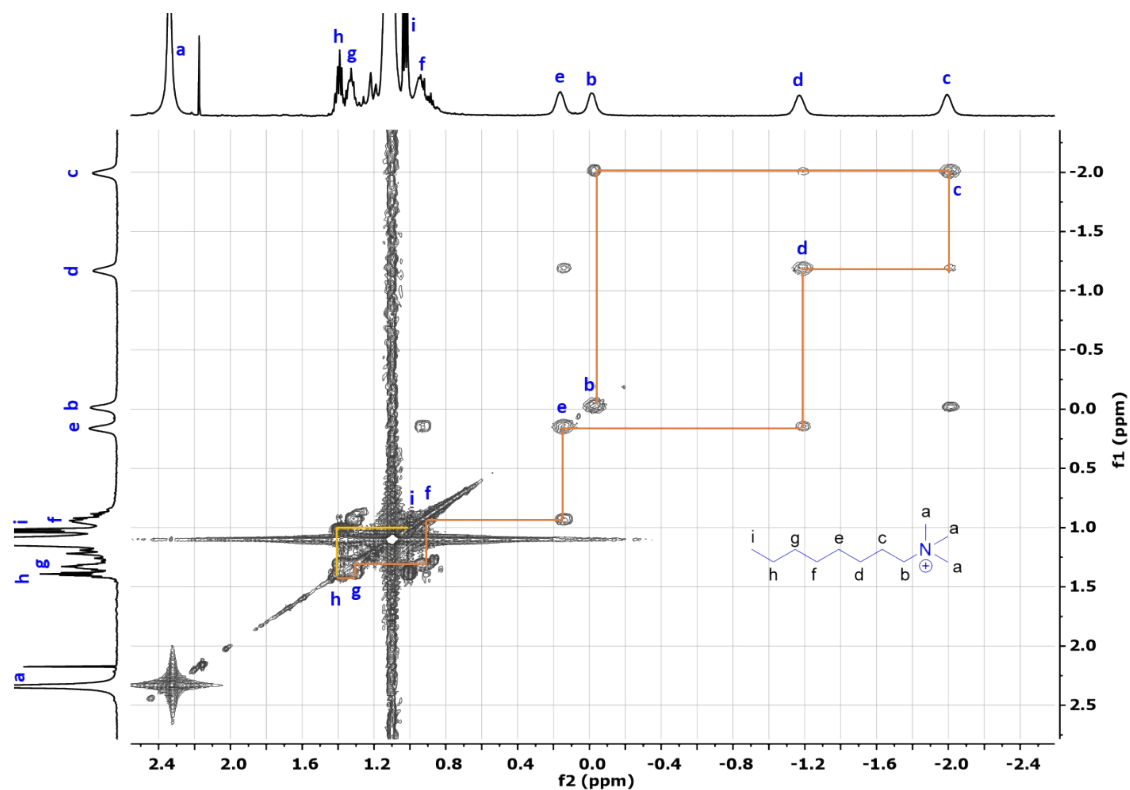


Figure S4. Partial ^1H - ^1H COSY spectrum (600 MHz, chloroform-*d*, 25 °C) of **Pilar-1** (6.3 mM) and **OMA** (6.3 mM).

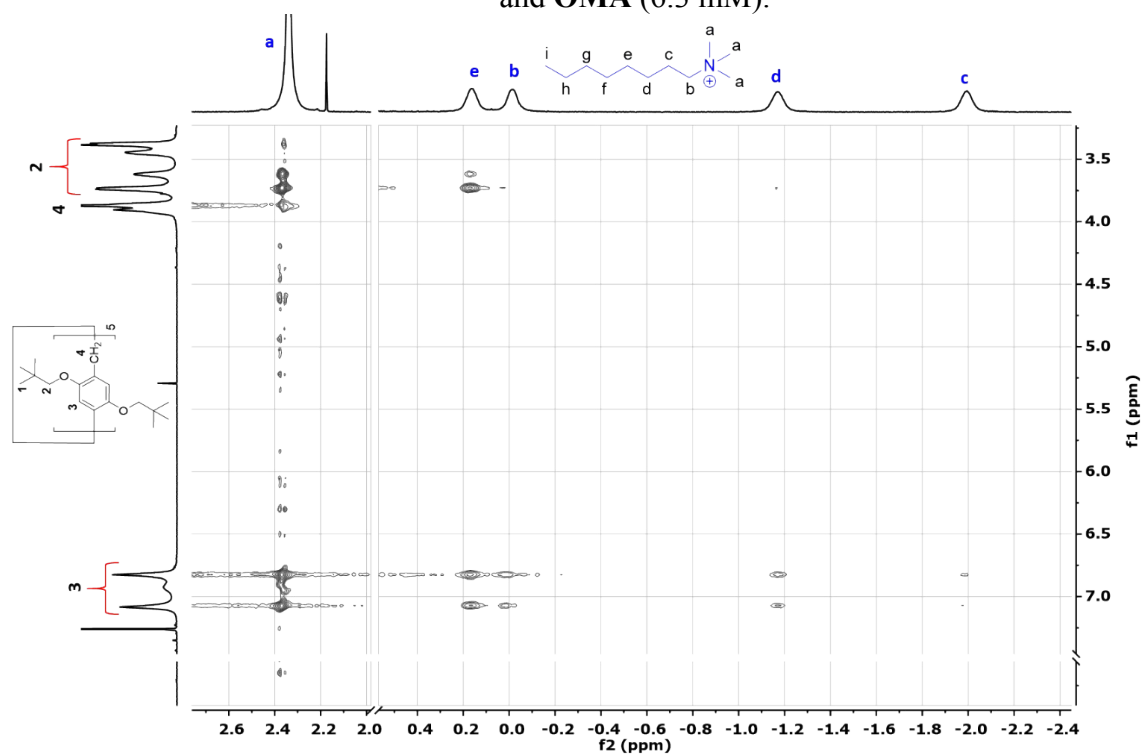


Figure S5. Partial ROSEY spectrum (600 MHz, chloroform-*d*, 25 °C) of **Pilar-1** (6.3 mM) and **OMA** (6.3 mM).

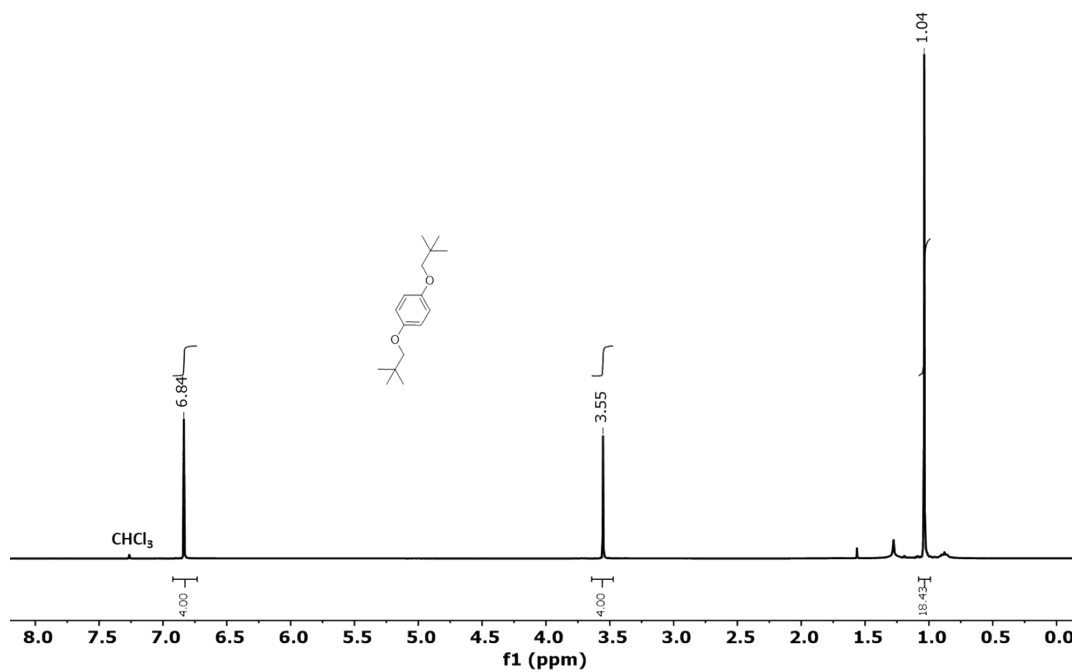


Figure S6. ¹H NMR (600 MHz, CDCl₃) spectrum of 1,4-bis(neopentyloxy)benzene.

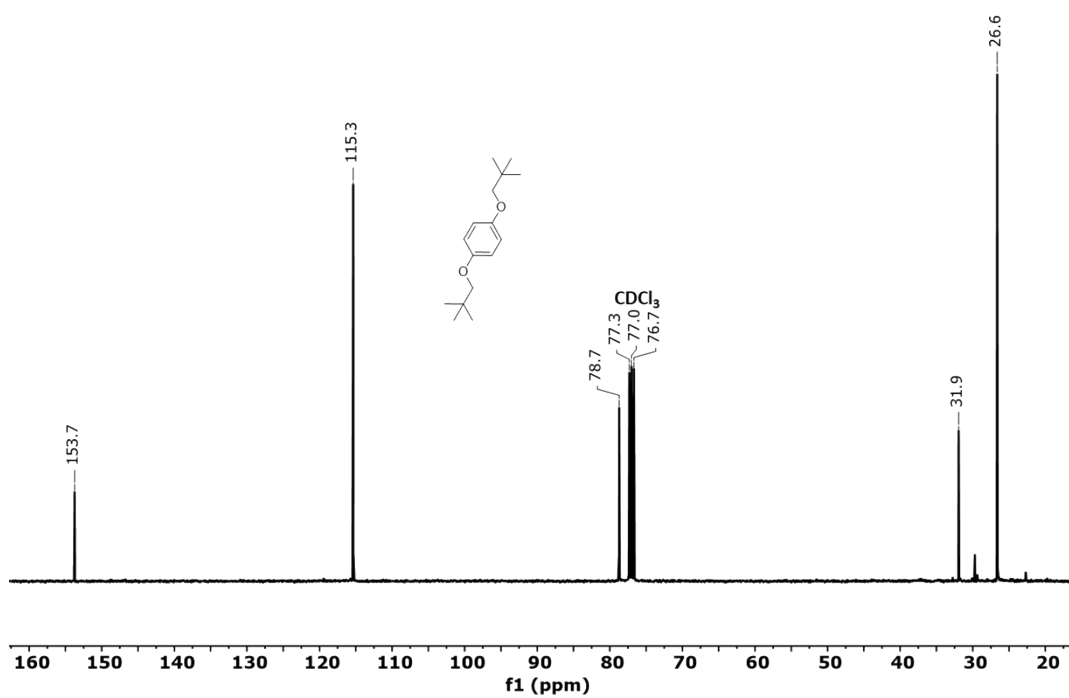


Figure S7. ¹³C NMR (150 MHz, CDCl₃) spectrum of 1,4-bis(neopentyloxy)benzene.

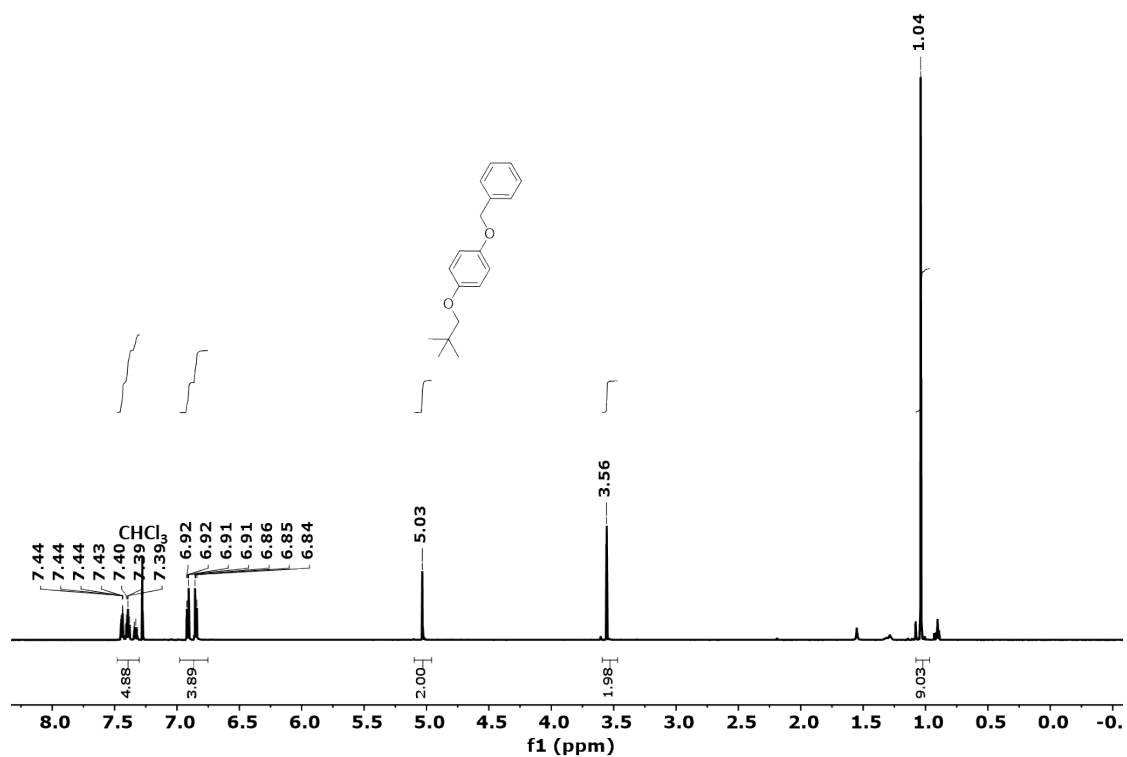


Figure S8. ^1H NMR (600 MHz, CDCl_3) spectrum of 1-(benzyloxy)-4-(neopentyloxy)benzene.

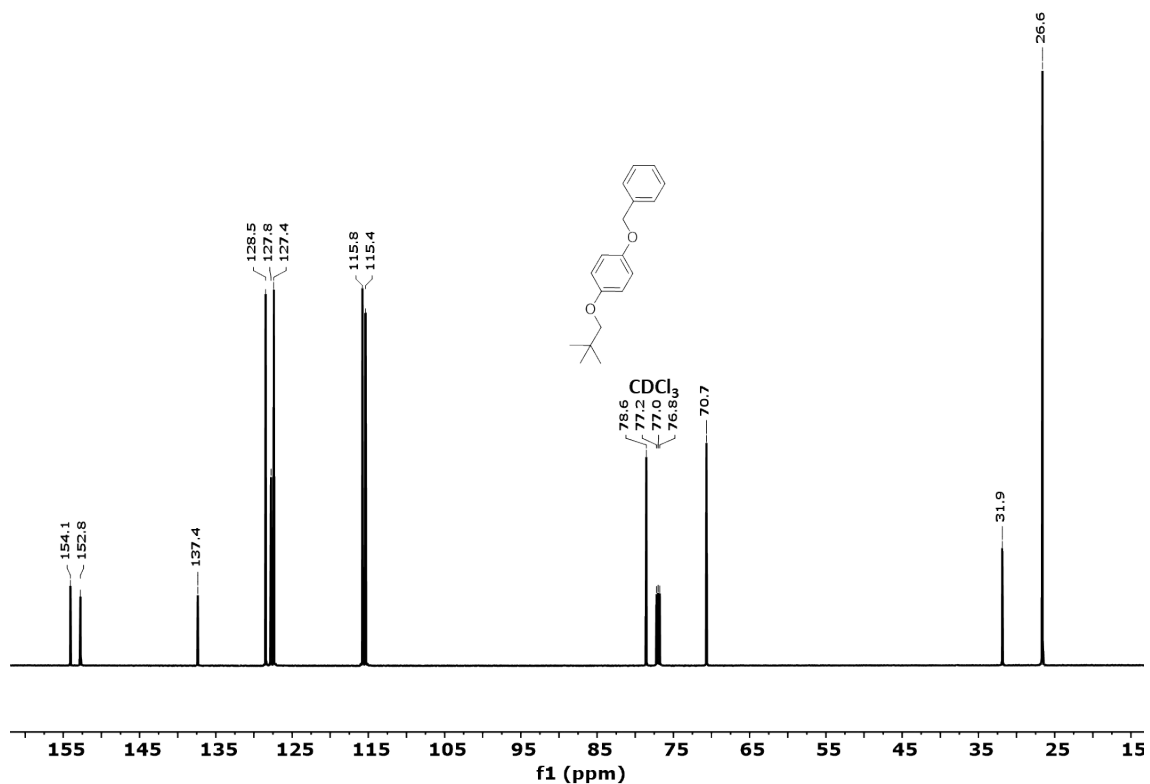


Figure S9. ^{13}C NMR (100 MHz, CDCl_3) spectrum of 1-(benzyloxy)-4-(neopentyloxy)benzene.

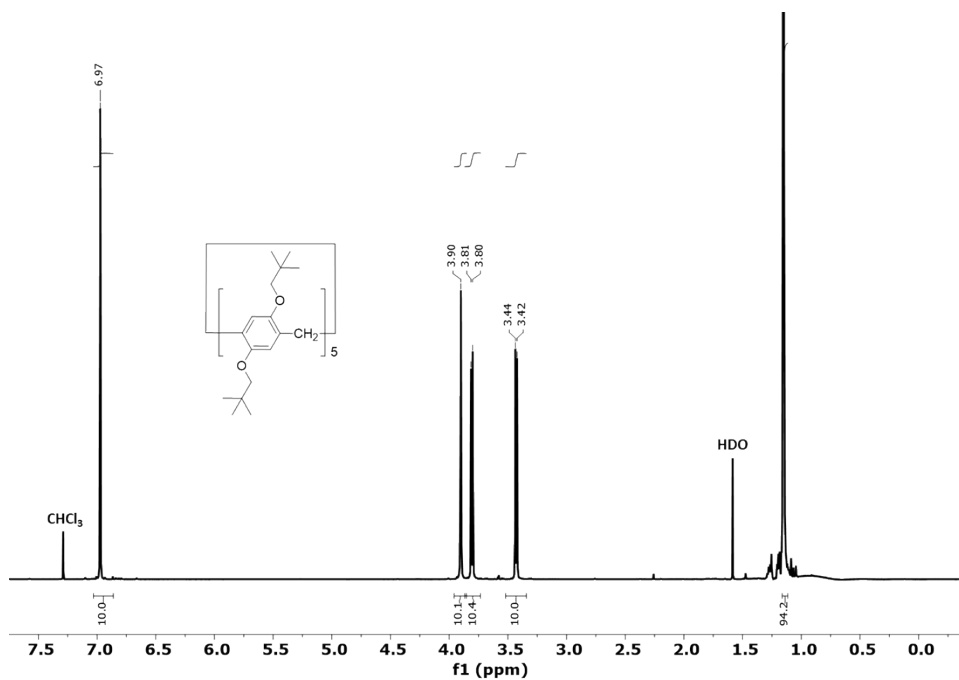


Figure S10. $^1\text{H NMR}$ (600 MHz, CDCl_3) spectrum of Pillar-1.

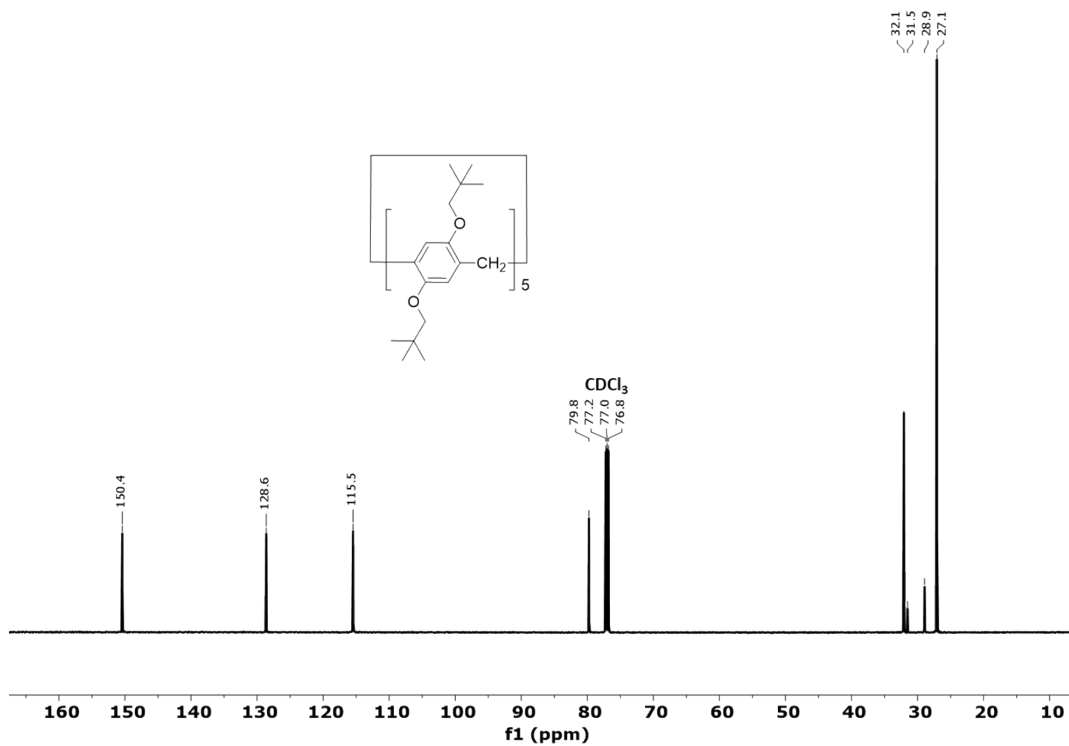


Figure S11. $^{13}\text{C NMR}$ (150 MHz, CDCl_3) spectrum of Pillar-1.

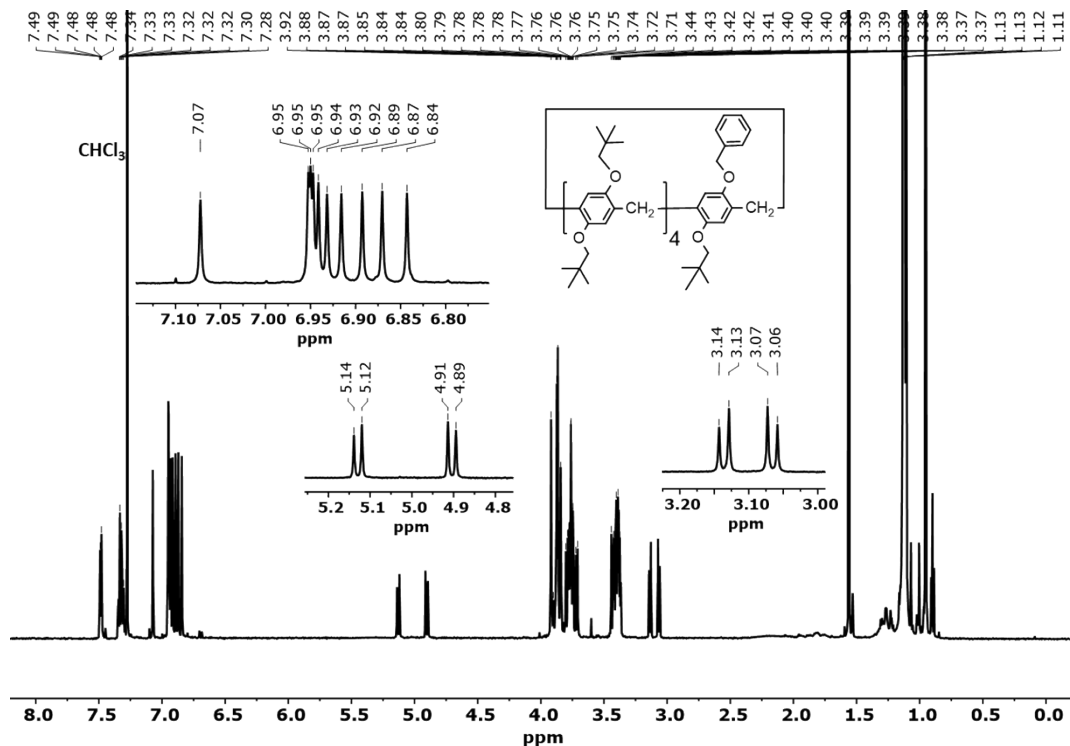


Figure S12. ^1H NMR (600 MHz, CDCl_3) spectrum of Pillar-2.

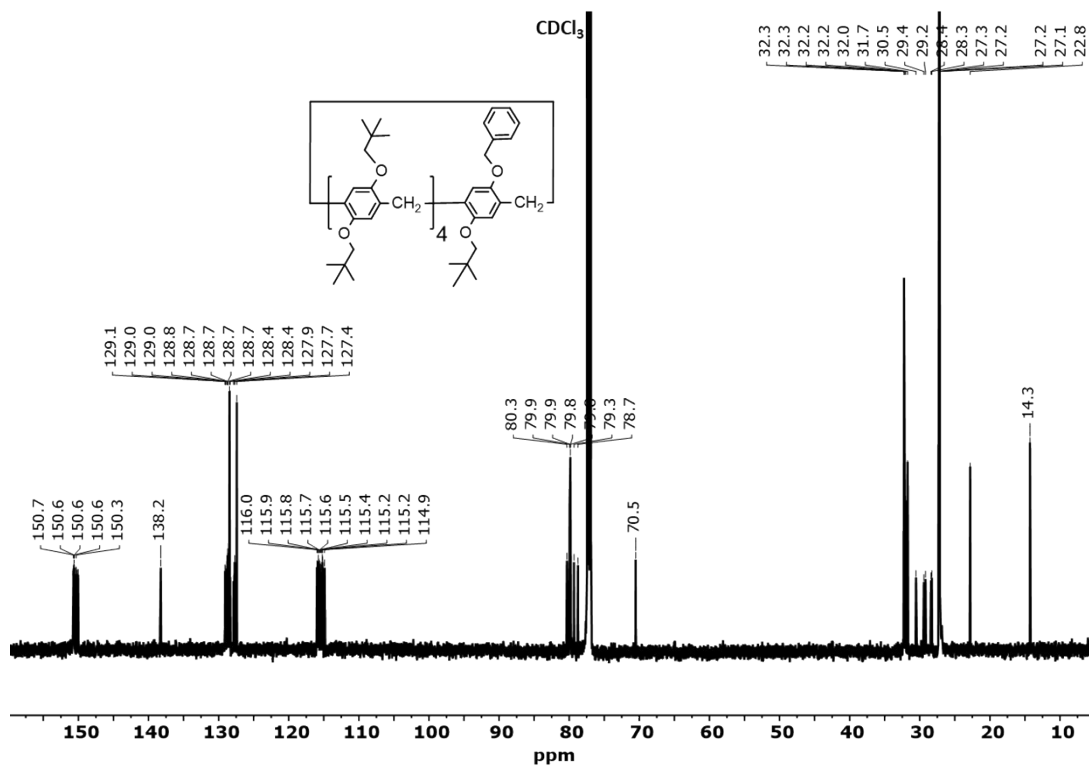


Figure S13. ^{13}C NMR (150 MHz, CDCl_3) spectrum of Pillar-2.

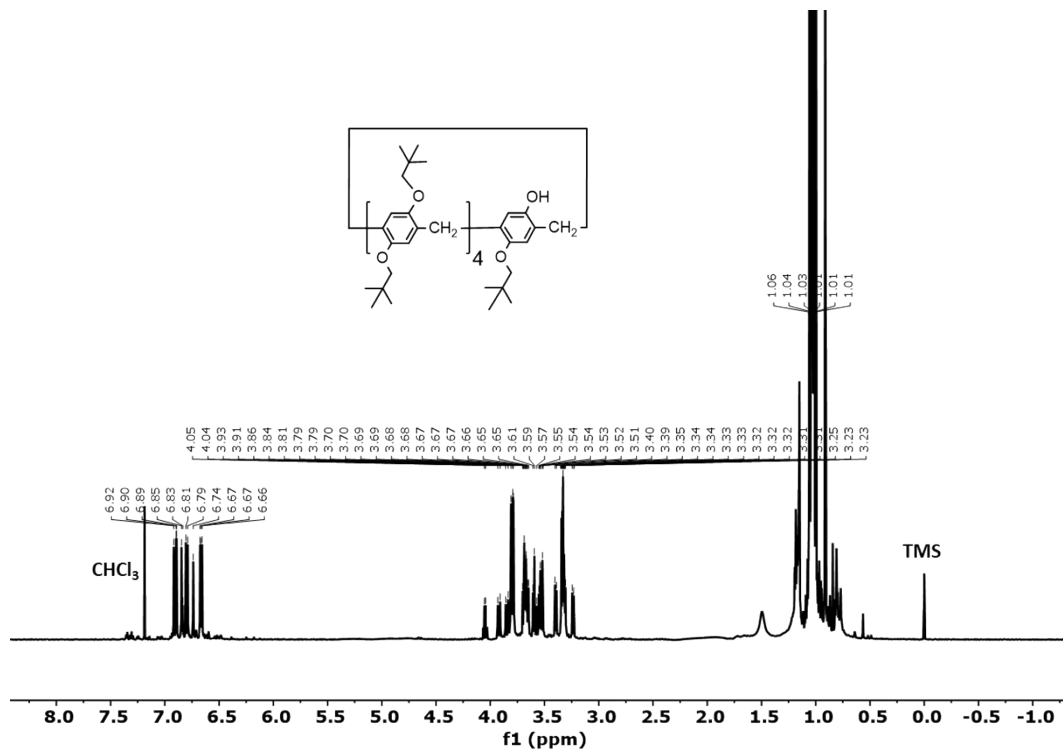


Figure S14. ¹H NMR (600 MHz, CDCl₃) spectrum of Pillar-3.

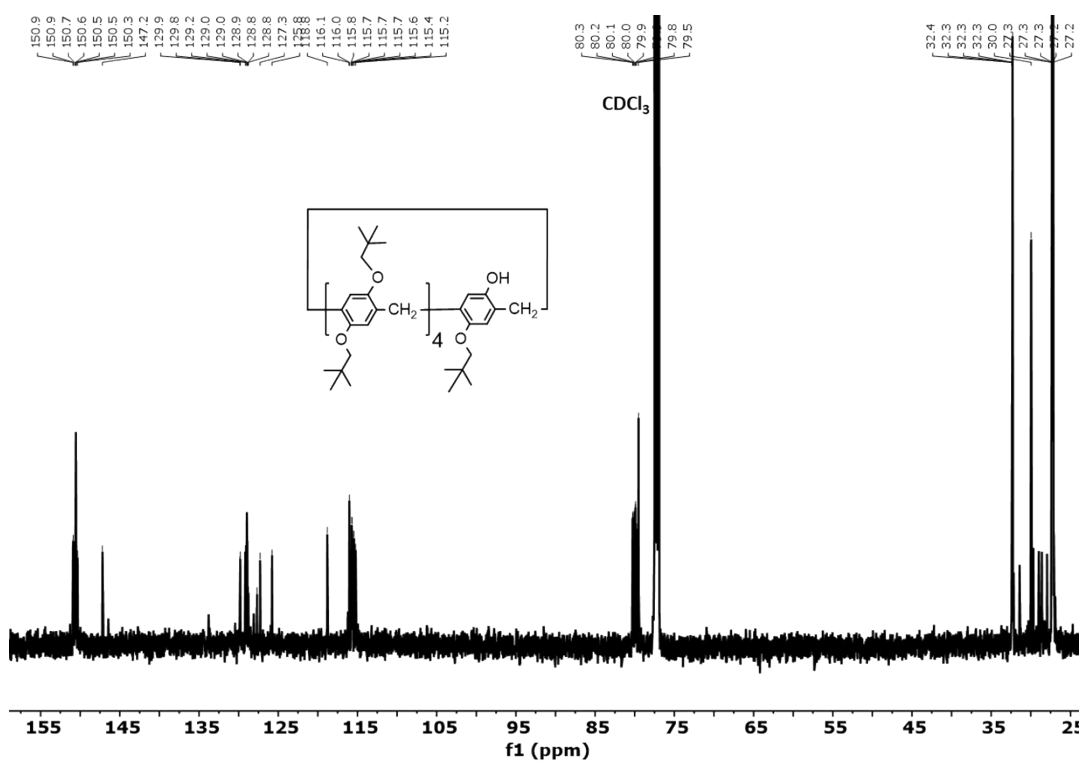
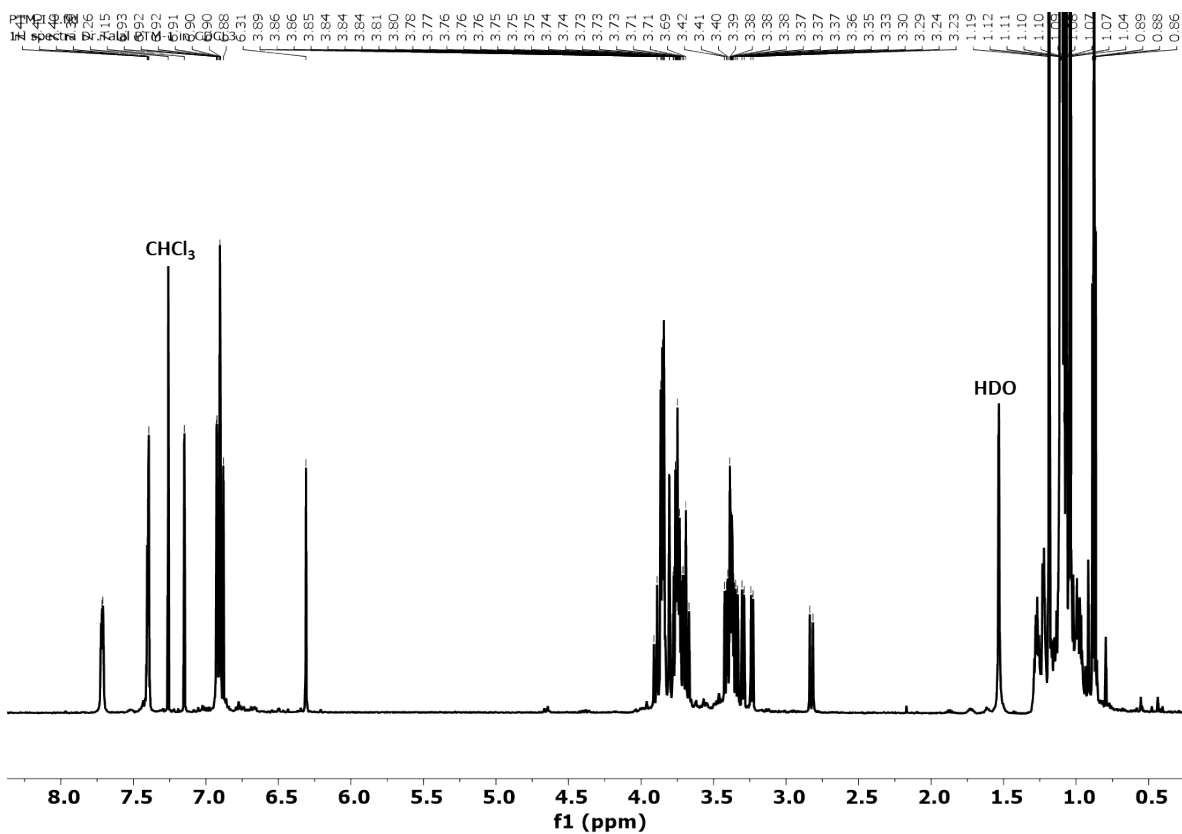
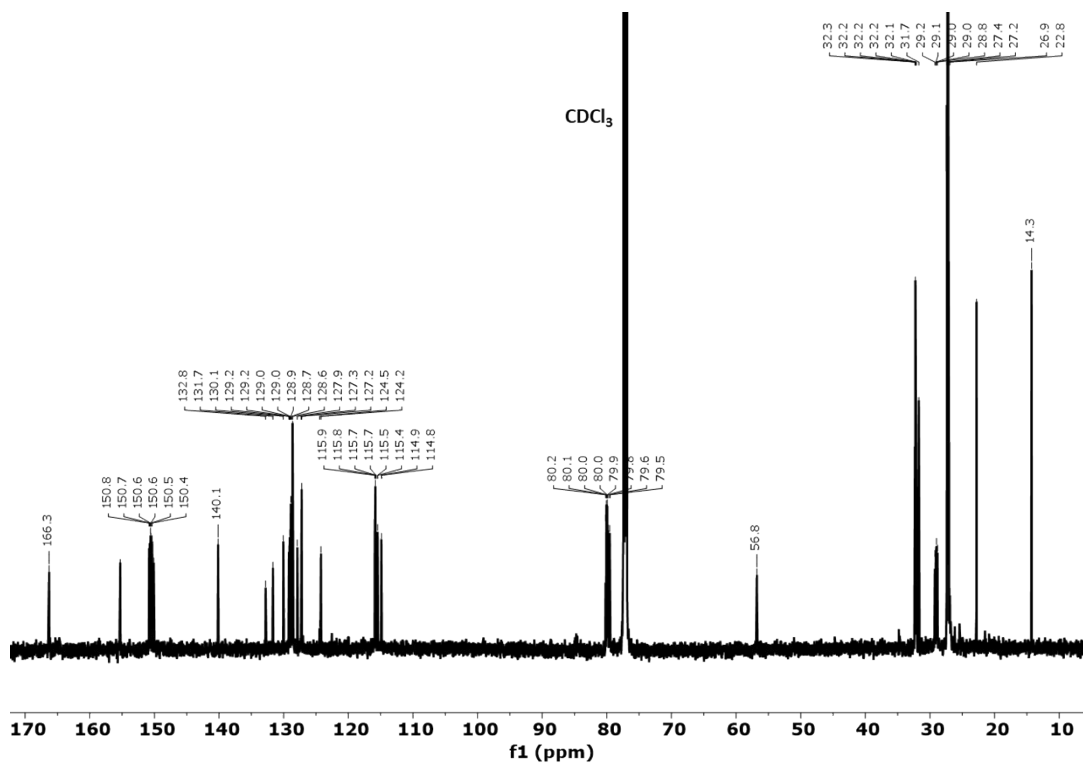


Figure S15. ¹³C NMR (150 MHz, CDCl₃) spectrum of Pillar-3.

Figure S16. ¹HNMR (600 MHz, CDCl₃) spectrum of Pillar-4a.Figure S17. ¹³CNMR (150 MHz, CDCl₃) spectrum of Pillar-4a.

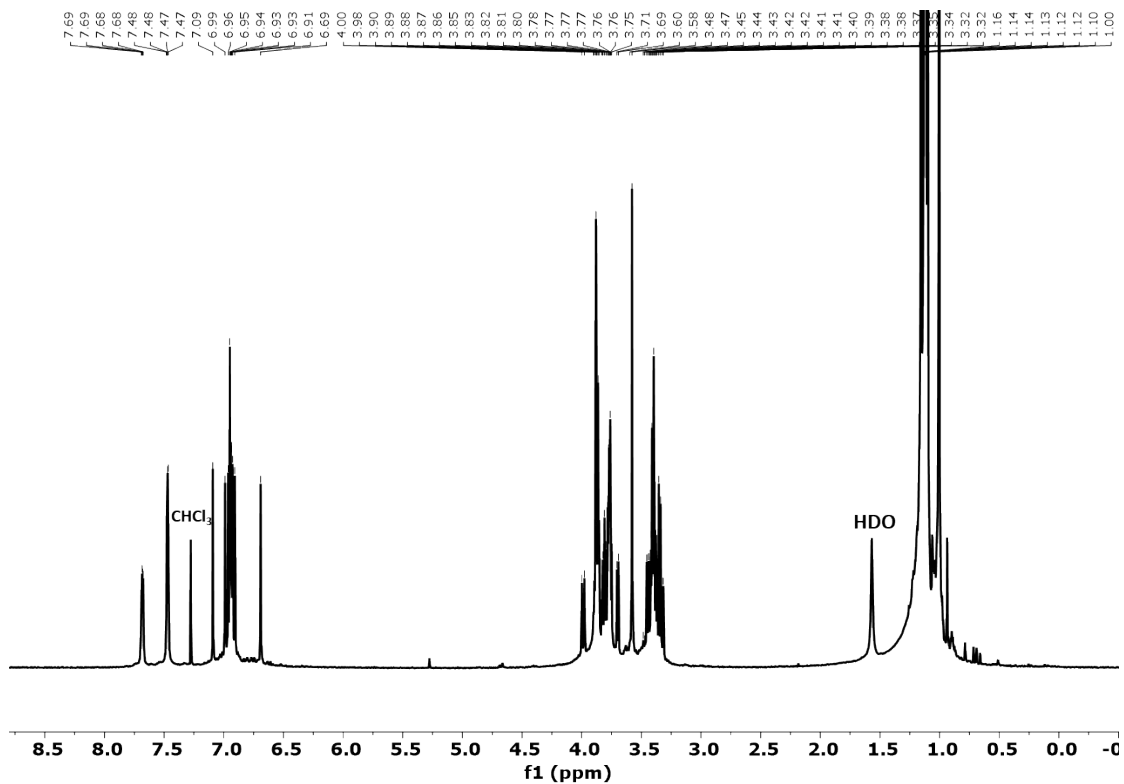


Figure S18. ¹H NMR (600 MHz, CDCl₃) spectrum of Pillar-4b.

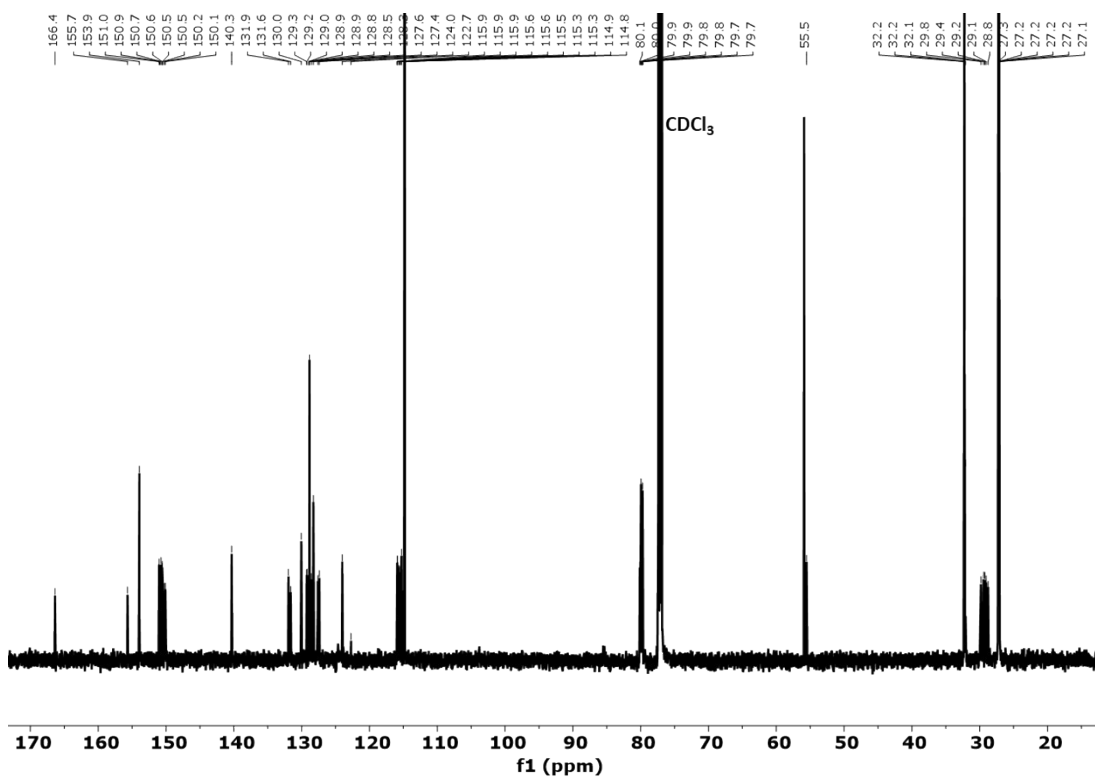


Figure S19. ¹³C NMR (150 MHz, CDCl₃) spectrum of Pillar-4b.

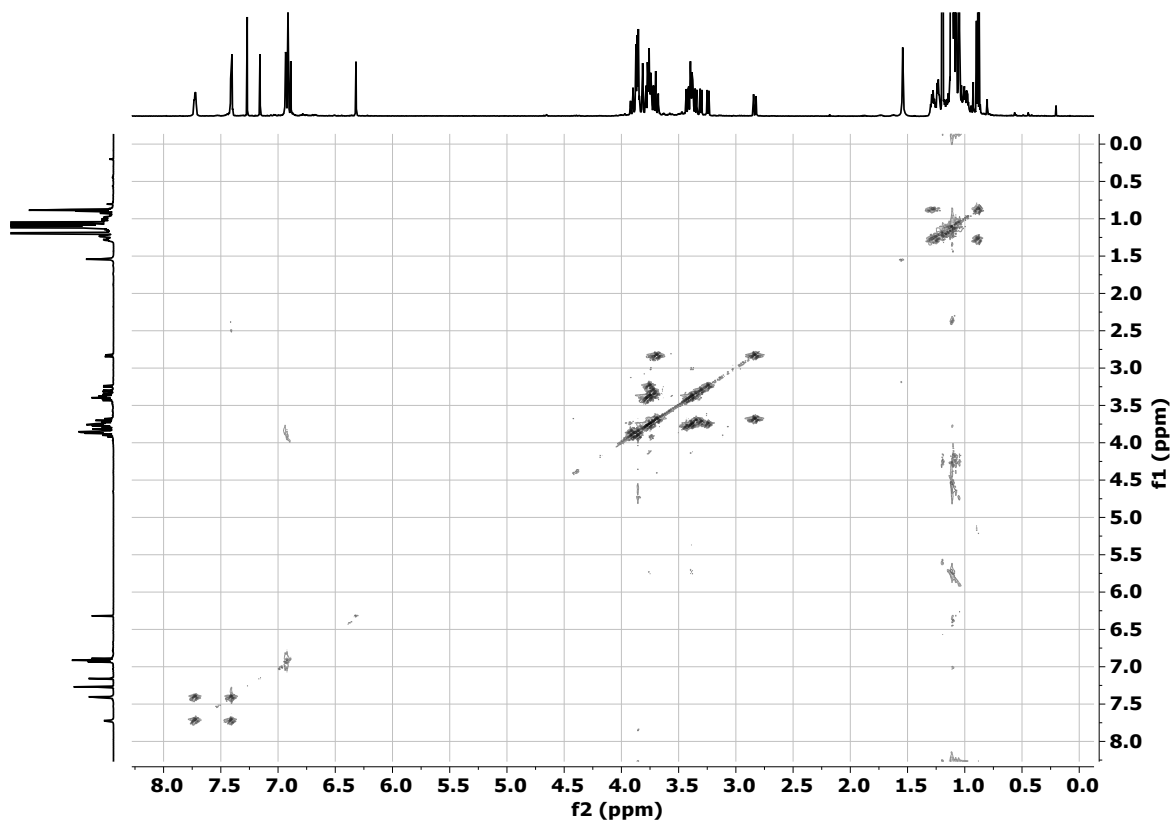


Figure S20. ^1H - ^1H COSY NMR (600 MHz, CDCl_3) spectrum of first fraction (**Pillar-4a**).

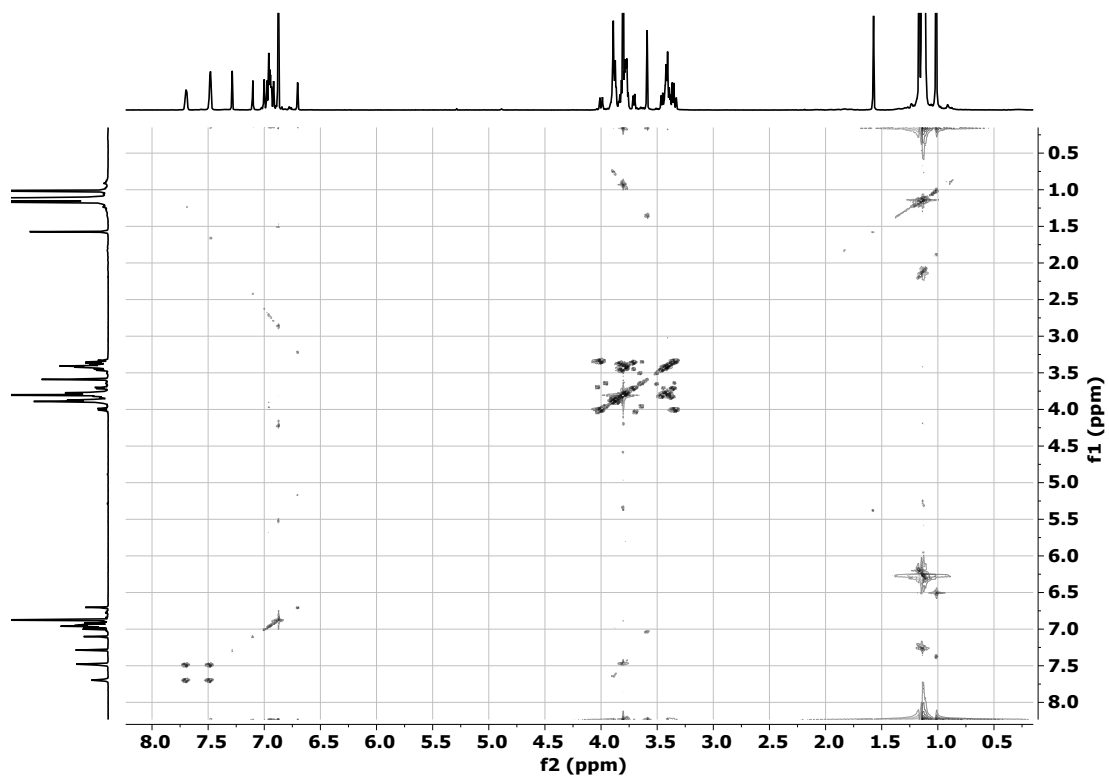


Figure S21. ^1H - ^1H COSY NMR (600 MHz, CDCl_3) spectrum of second fraction (**Pillar-4b**).

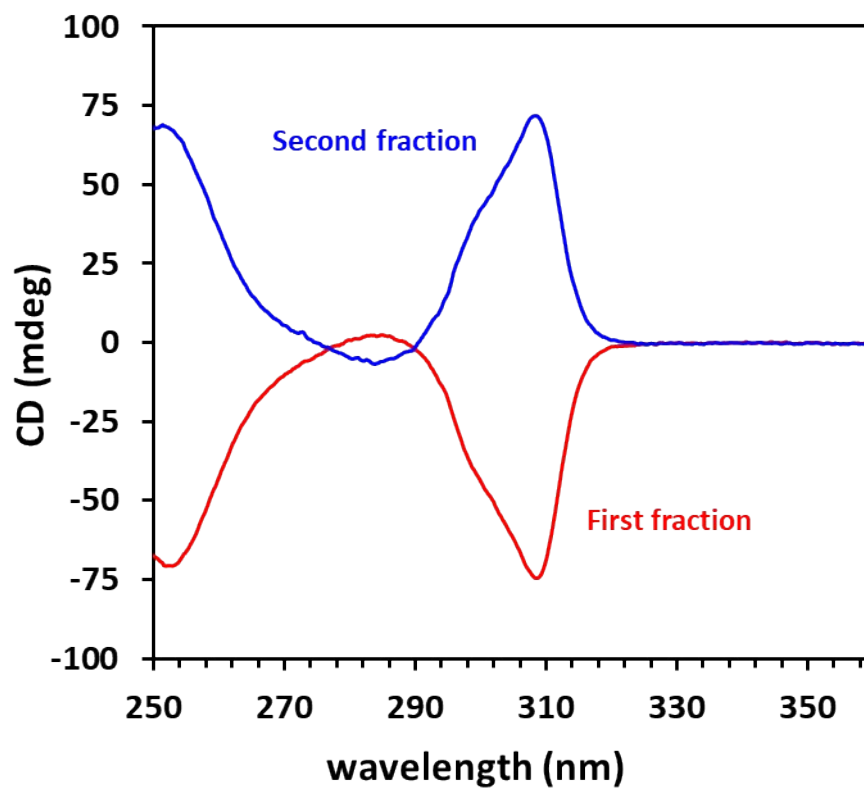


Figure S22. CD spectra of the first and second fractions ($12 \mu\text{Lmol}^{-1}\text{cm}^{-1}$) in hexane at 25 °C.