

# ChemistryEurope

Supporting Information

## **Molecular Actuation of a Bistable Liquid-Crystalline [c2]Daisy Chain Rotaxane**

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## 1. General methods

**Solvents and chemical reagents:** All solvents and all commercially available reagents were purchased at the highest quality and used as received unless otherwise noted. Dry solvents were obtained by passing them through a double column SolvTech purification system. Water was deionized by using a milli-gradient system (Millipore, Molsheim, France). Microwave reactions were carried out with a single mode cavity Discover SP Microwave (CEM Corporation, NC, USA), producing continuous irradiation at 2455 MHz and equipped with simultaneous external air-cooling system. Yields refer to spectroscopically purified ( $^1\text{H}$  Nuclear Magnetic Resonance) homogeneous materials.

**Chromatographic methods:** Thin Layer Chromatographies (TLC) were performed using silica on TLC Al foils (silica gel matrix with fluorescent indicator 254 nm, thickness: 500  $\mu\text{m}$ , Sigma-Aldrich). In most cases, irradiation using a *Bioblock VL-4C* UV-Lamp (6 W, 254 nm and/or 365 nm) as well as potassium permanganate stain were used for visualization. Flash Column Chromatographies were performed using silica gel (60  $\text{\AA}$ , 230 - 400 mesh, 40 - 63  $\mu\text{m}$ , Sigma-Aldrich). Ultra-performance liquid chromatography with mass spectrometry (UPLC-MS) analyses were conducted on a Waters Acquity UPLC-SQD apparatus equipped with a SQD mass spectrometer, a PDA detector (190 - 500 nm, 80 Hz), using a reverse phase column (Waters, BEH C18 1.7  $\mu\text{m}$ , 2.1  $\times$  50 mm), the MassLynx 4.1 - XP software and a gradient of water/acetonitrile + 0.1 % TFA as eluent.

**Nuclear magnetic resonance (NMR):** All the spectra were recorded on a Bruker Avance 400 at 25 $^\circ\text{C}$  unless otherwise noted.  $^1\text{H}$  NMR spectra were recorded at 400 MHz and  $^{13}\text{C}$  NMR spectra were recorded at 100 MHz. The observed signals are reported as follows: chemical shift in ppm from the signal of TMS, with the residual solvent signal as internal standard ( $^1\text{H}$  NMR:  $\text{CDCl}_3$   $\delta$  7.26 ppm;  $^{13}\text{C}$  NMR:  $\text{CDCl}_3$   $\delta$  77.16 ppm); multiplicity (singlet (s), doublet (d), triplet (t), quartet (q), quintuplet (qt), septuplet (sept), multiplet (m), broad (br)); integration. Coupling constants  $J$  are given in Hz.

**Mass spectrometry.** Electron Spray Ionization (ESI) was recorded on a SQD mass spectrometer from Waters either by direct injection or after chromatography. High Resolution Mass Spectrometry (HR-MS) was performed on a micrOTOF-Q instrument from Bruker.

**Differential Scanning Calorimetry (DSC).** DSC experiments were performed on a DSC 8000 from Perkin-Elmer at the Institut Charles Sadron (ICS). The samples were prepared using hermetic aluminium pans and the experiments conducted under a flow of nitrogen (20 mL/min). The samples were heated from 25 °C to 240 °C at 20 °C/min and cooled to -10 °C at the same rate during the first cycle. Second and third cycles were performed from -10 °C to 240 °C at 10 and 5 °C/min, respectively.

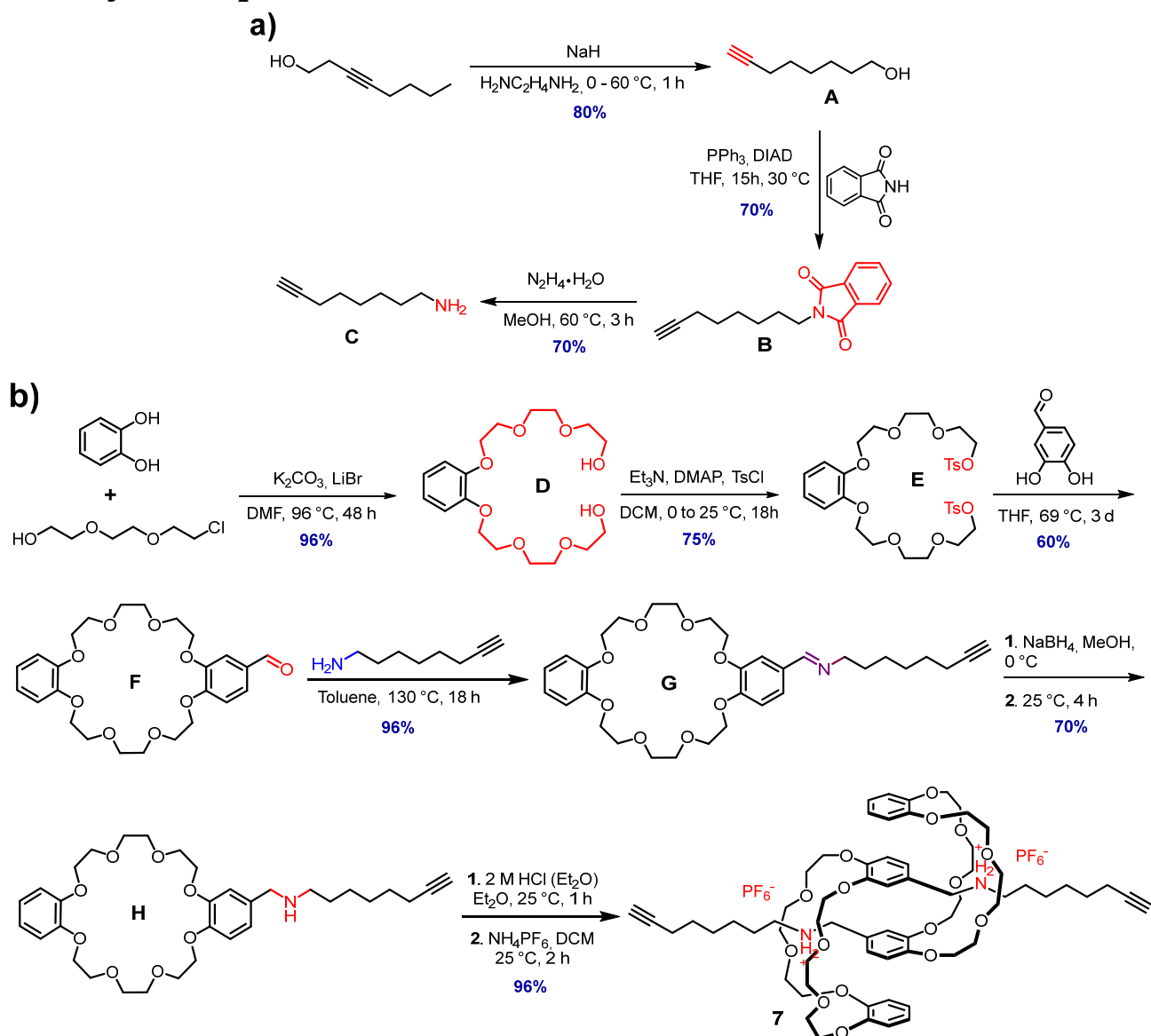
**Thermogravimetric Analysis (TGA).** TGA experiments were performed on a TGA2 Star System instrument from Mettler Toledo. A heating rap from 25 °C to 400 °C at a rate of 10 °C/min under an argon flow of 10 mL/min was subjected to each material (2 to 5 mg).

**Polarising optical microscopy (POM).** POM experiments were performed with an OLYMPUS model BX-51 polarizing optical microscope equipped with a Mettler FP-82HT hot stage.

**Molecular mechanics calculations.** Theoretical MM2 calculations of [c2]daisy chain rotaxanes **9<sub>Ext</sub>** and **9<sub>Cont</sub>** were performed using the Chem3D<sup>®</sup> Pro 22.2.0 software (CambridgeSoft). The minimization of the energy was performed by using a minimum RMS gradient of 0.100 after 10000 iterations.

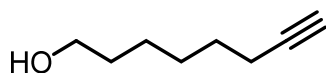
**Small-Angle X-ray Scattering experiments (SAXS)** were performed on the X-ray scattering platform *Différix* of the Institut Charles Sadron (Strasbourg) using a Rigaku rotating anode generator (Micromax<sup>TM</sup>-007 HF, operating power 40 kV at 30 mA, wavelength  $\lambda = 1.54 \text{ \AA}$ ). The beam was monochromated and collimated with a confocal Max-Flux Optics<sup>TM</sup> mirror (Osmics, Inc.) combined to a system of three pinholes. The scattered intensity was collected on a 2D multiwires detector (Rigaku) located at 81 cm from the sample position. This setup has a scattering vector range from  $q = 0.01$  to  $0.3 \text{ \AA}^{-1}$  with  $q = (4\pi/\lambda) \sin \theta$ , where  $2\theta$  is the scattering angle. The samples were filled into circular brass cells with 18  $\mu\text{m}$  thick mica windows and 1 mm beam path. Data reduction was done in Igor Pro 7, according to usual procedures for isotropic scattering (angular averaging, electronic background, sample transmission and thickness corrections).  $q$  vectors were calibrated using a silver behenate powder standard. Intensities were converted into absolute scale using a calibrated Lupolen standard. An empty cell was used as reference.

## 2. Synthetic procedures



**Scheme S1.** Synthetic route for compound 7.

### Oct-7-yn-1-ol (A)

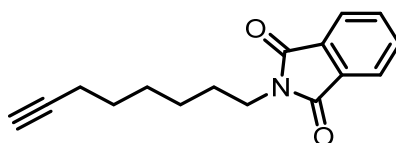


In a 500 mL two-neck round-bottom flask equipped with a stirring bar and a findenser containing freshly distilled and dried ethylene-1,2-diamine (150 mL) at 4 °C was added sodium hydride (60% suspension in oil, 7.0 g, 175 mmol). The grey cloudy mixture was allowed to warm to 65 °C under vigorous stirring for

one hour to give a deep blue mixture. After cooling down the mixture to 45 °C, 3-octyn-1-ol (6.5 mL, 45 mmol) was added dropwise over fifteen minutes. The solution was stirred at 65 °C for one hour and then cooled to 4 °C using a cold bath containing a water: ice mixture. Water (100 mL) and diethyl ether (200 mL) were then added slowly to the reaction mixture, which was acidified using 12 M hydrochloric acid carefully until reaching pH 1. The aqueous layer was extracted with diethyl ether (3 x 200 mL) and the organic layers were combined, dried over sodium sulfate, and concentrated. The crude residue was purified by column chromatography (SiO<sub>2</sub> (200 g), *n*-pentane | EtOAc 10% → *n*-pentane | EtOAc 40%) to provide compound **A** (4.6 g, 80%) as a pale yellow liquid. *Analyses were in accordance with the literature.*<sup>1</sup>

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C): δ = 3.63 (t, *J* = 6.7 Hz, 2H), 2.19 (td, *J* = 7.0, 2.7 Hz, 2H), 1.91 (t, *J* = 2.6 Hz, 1H), 1.61 – 1.51 (m, 4H), 1.49 – 1.33 (m, 4H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz, 25 °C): δ = 84.7, 68.3, 63.0, 32.7, 28.6, 28.5, 25.4, 18.5.

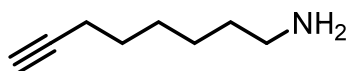
#### 2-(oct-7-yn-1-yl)isoindoline-1,3-dione (**B**)



In a 100 mL one-neck round-bottom flask equipped with a stirring bar was prepared a mixture of alcohol **A** (0.50 g, 3.96 mmol), PPh<sub>3</sub> (1.1 g, 4.2 mmol), and phthalimide (0.61 g, 4.2 mmol) in anhydrous tetrahydrofuran (15 mL). Diisopropyl azodicarboxylate (DIAD; 0.8 mL, 4.2 mmol) was slowly added and the reaction mixture was stirred for 3 hours at 0 °C, and then during two days at 25 °C. Water (30 mL) was added and the aqueous phase was extracted with dichloromethane (2 x 30 mL). The organic layers were combined, dried over sodium sulfate, evaporated, and the crude residue was purified by column chromatography (SiO<sub>2</sub> (100 g), *n*-hexane | EtOAc 10%) to yield compound **B** (0.7 g, 70%) as a white solid. *Analyses were in accordance with the literature.*<sup>2</sup>

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C): δ = 7.90 – 7.80 (m, 2H), 7.76 – 7.67 (m, 2H), 3.68 (td, *J* = 7.5, 1.3 Hz, 2H), 2.18 (td, *J* = 7.0, 2.7 Hz, 2H), 1.91 (t, *J* = 2.6 Hz, 1H), 1.68 (tt, *J* = 7.4, 6.8 Hz, 2H), 1.52 – 1.25 (m, 6H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz, 25 °C): δ = 169.1, 132.4, 132.0, 122.1, 84.5, 68.1, 41.9, 33.7, 28.6, 27.5, 27.4, 18.5. **ESI-MS**: *m/z* calcd. For C<sub>16</sub>H<sub>18</sub>NO<sub>2</sub>: 256.13 [M+H]<sup>+</sup>, found: 256.26.

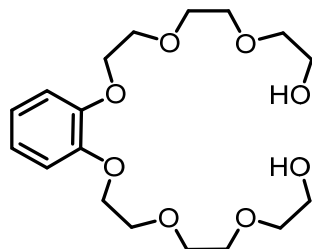
### Oct-7-yn-1-amine (C)



In a 250 mL one-neck round-bottom flask equipped with a stirring bar and a condenser was prepared a solution of compound **B** (3.0 g, 11.8 mmol) in methanol (100 mL). Hydrazine hydrate 25% (3 mL, 60 mmol) was added to the mixture, which was stirred at reflux for four hours and then cooled down to room temperature. After removing the solvent, the residue was taken back in a 2 M potassium hydroxide solution (50 mL). The solution was extracted three times with dichloromethane (3 x 50 mL) and then the organic layers were dried over sodium sulfate and concentrated to yield compound **C** (1.0 g, 69%) as a pale yellow liquid. *Analyses were in accordance with the literature.*<sup>2</sup>

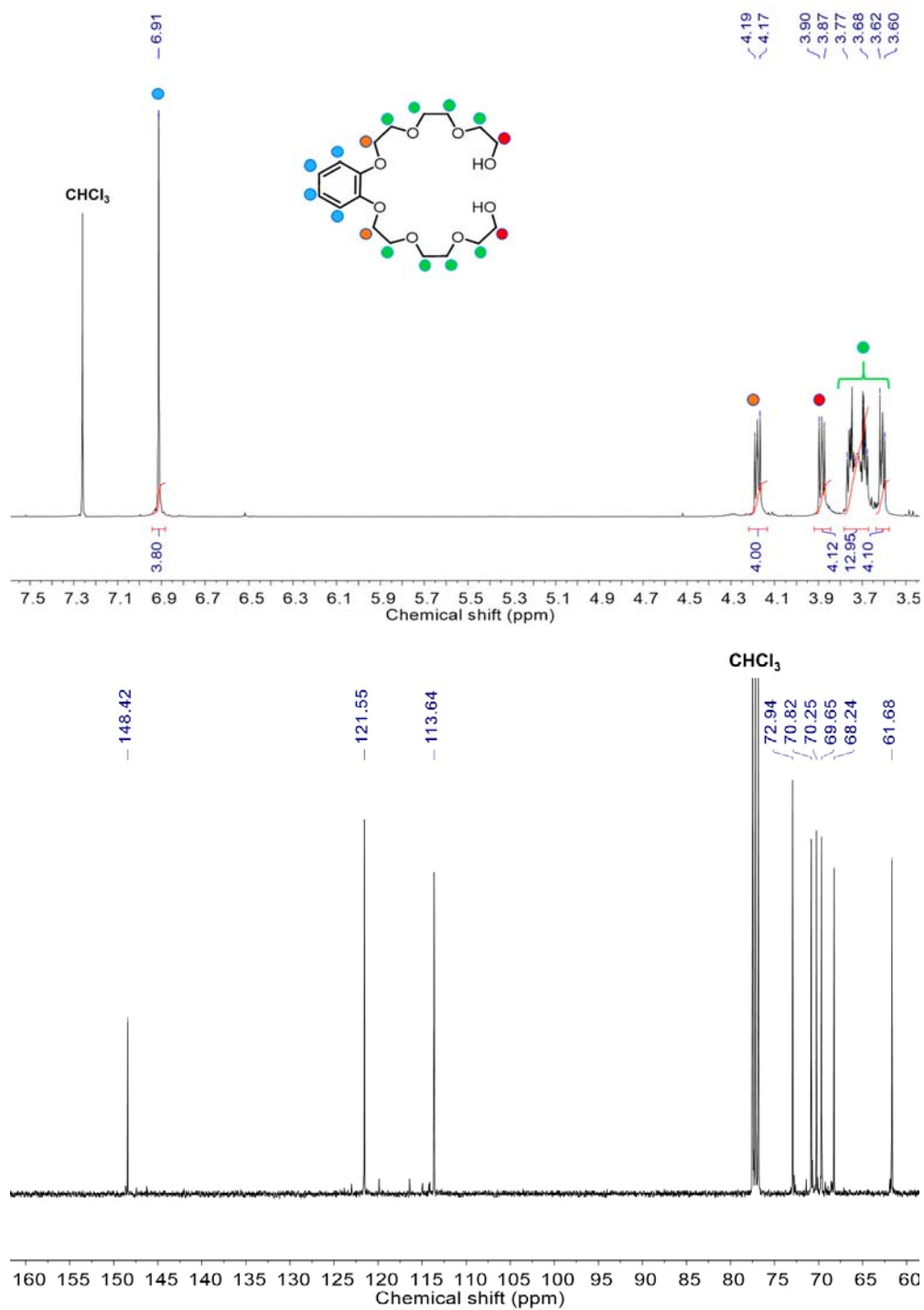
<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C): δ = 2.69 (t, *J* = 6.7 Hz, 2H), 2.18 (td, *J* = 7.0, 2.7 Hz, 2H), 1.91 (t, *J* = 2.6 Hz, 1H), 1.59 – 1.42 (m, 2H), 1.40 – 1.23 (m, 6H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz, 25 °C): δ = 84.5, 68.1, 41.9, 33.7, 28.6, 27.5, 27.4, 18.5.

### 1,2-bis(2-(2-(2-hydroxyethoxy)ethoxy)ethoxy)benzene (D)



A 250 mL two-neck round-bottom flask equipped with a stirring bar and a condenser was charged with catechol (2.7 g, 24.5 mmol), potassium carbonate (8.4 g, 61.5 mmol), and lithium bromide (2.2 g, 25 mmol) suspended in dry *N,N*-dimethylformamide (120 mL). 2-[2-(2-chloroethoxy)ethanol (7.5 mL, 52 mmol) was added dropwise under argon and the reaction mixture was stirred at 96 °C during three days. Then, the solvent was evaporated, the residue dissolved in CH<sub>2</sub>Cl<sub>2</sub> (120 mL) and washed with 10% K<sub>2</sub>CO<sub>3</sub> solution (3 x 100 mL). The organic layers were combined, dried over sodium sulfate, and concentrated under reduced pressure to yield compound **D** (8.80 g, 96%) as a brown oil, which was pure enough to be used as such in the next step. *Analyses were in accordance with the literature.*<sup>3</sup>

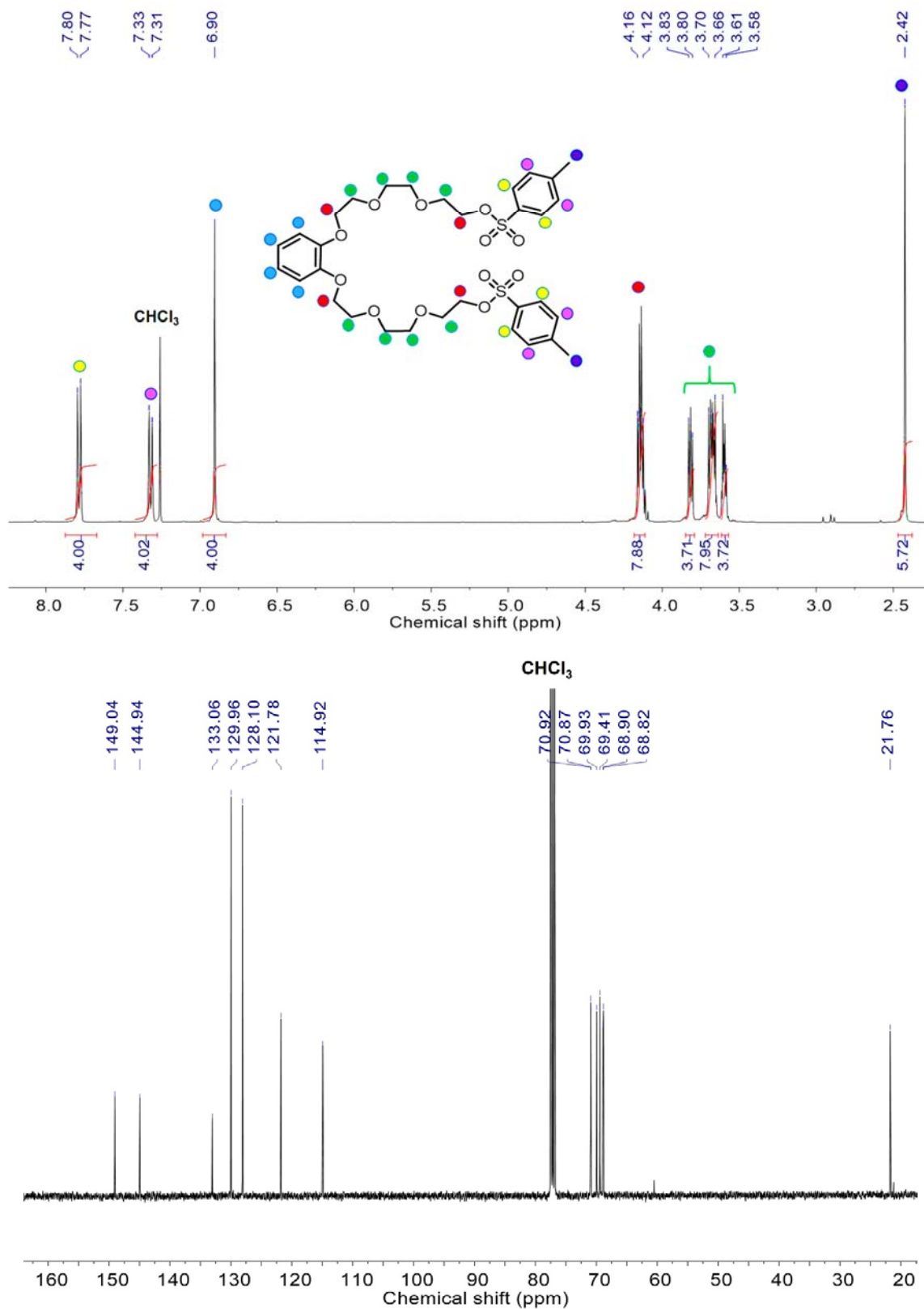
$^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz, 25 °C):  $\delta$  = 6.91 (s, 4H), 4.19 – 4.17 (m, 4H), 3.90 – 3.87 (m, 4H), 3.77 – 3.68 (m, 12H), 3.62 – 3.60 (m, 4H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz, 25 °C):  $\delta$  = 148.4, 121.6, 113.6, 72.9, 70.8, 70.2, 69.6, 68.2, 61.7. ESI-MS:  $m/z$  calcd. For  $\text{C}_{18}\text{H}_{31}\text{O}_8$ : 375.20  $[\text{M}+\text{H}]^+$ , found: 375.69



**Figure S1.**  $^1\text{H}$  (top) and  $^{13}\text{C}$  (bottom) NMR spectra of compound D in  $\text{CDCl}_3$  at 25 °C.

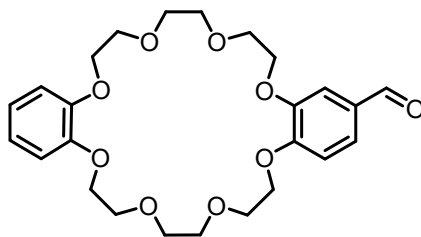






**Figure S2.** <sup>1</sup>H (top) and <sup>13</sup>C (bottom) NMR spectra of compound E in CDCl<sub>3</sub> at 25 °C.

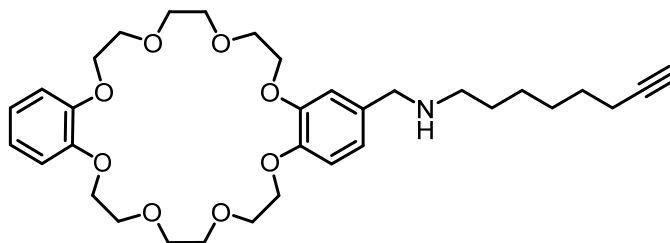
### (2-formyl)dibenzo[24]crown-8 (F)



A 1 L two-neck round-bottom flask equipped with a stirring bar, a condenser and a dropping funnel was charged with cesium carbonate (22.0 g, 67.5 mmol) and 4-formylcatechol (2.2 g, 16 mmol) dissolved in dry tetrahydrofuran (200 mL). Bistosylate **E** (12.0 g, 17.5 mmol) was dissolved in dry tetrahydrofuran (100 mL) and charged in a dropping funnel, while the suspension was maintained under stirring at 65 °C for 30 minutes. The bistosylate solution was added dropwise over 90 minutes and the reaction mixture was stirred at 65 °C for 3 days. Then, the solvent was removed, and the residue was taken back in chloroform (200 mL) and washed with 10% potassium carbonate (200 mL). The organic phase was dried over sodium sulfate, the solvent evaporated, and the crude residue was purified by column chromatography (SiO<sub>2</sub> (400 g), EtOAc → EtOAc: MeOH 2%) to yield compound **F** (4.5 g, 59%) as a white solid powder. *Analyses were in accordance with the literature.*<sup>4</sup>

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C): δ = 9.81 (s, 1H), 7.41 (dd, *J* = 8.1, 1.9 Hz, 1H), 7.37 (d, *J* = 8.2 Hz, 1H), 6.94 (d, *J* = 8.2 Hz, 1H), 6.89-6.86 (m, 4H), 4.22-4.18 (m, 8H), 3.96 - 3.92 (m, 8H), 3.84 - 3.82 (m, 8H).  
<sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz, 25 °C): δ = 190.9, 154.4, 149.3, 149.0, 130.3, 126.9, 121.5, 114.2, 112.0, 111.2, 71.7, 71.5, 71.4, 70.1, 69.7, 69.8. **ESI-MS**: *m/z* calcd. for C<sub>25</sub>H<sub>34</sub>O<sub>10</sub>: 494.20 [M+H<sub>2</sub>O]<sup>+</sup>, found: 494.16.

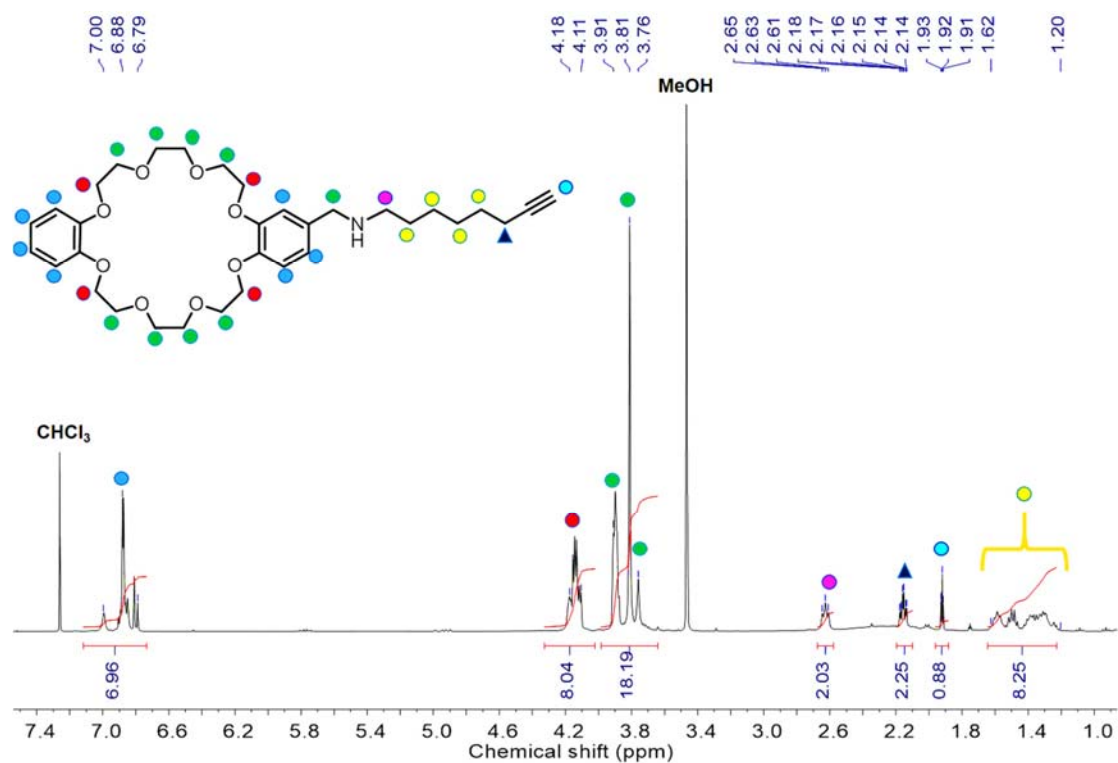
### Compound G



A 500 mL two-neck round-bottom flask equipped with a stirring bar and a condenser was charged with compound **F** (2.0 g, 4.2 mmol) and oct-7-yn-1-amine (0.53 g, 4.3 mmol) in dry toluene (100 mL). The

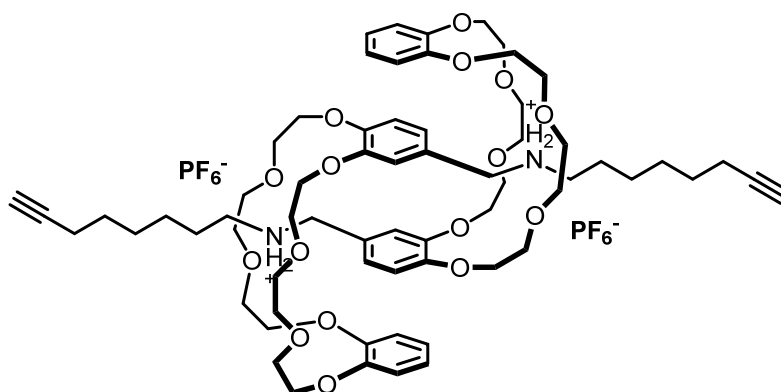
solution was heated during 15h at reflux using a Dean-Stark apparatus. The solvent was then evaporated under reduced pressure providing the imine derivative as a yellow oil (compound F<sup>9</sup>). This oil was dissolved in anhydrous methanol (100 mL), and then, sodium borohydride (0.8 g, 21 mmol) was added portion wise at 0 °C. The solution was then stirred at room temperature during four hours, then a 1 M aqueous solution of HCl (100 mL) was added to the mixture. The solvent was evaporated, and the residue was taken back in dichloromethane (150 mL) and the organic phase was washed with a 2 M aqueous solution of NaOH (100 mL), dried over sodium sulfate and concentrated under reduced pressure. The crude was purified by column chromatography (SiO<sub>2</sub> (100 g), CH<sub>2</sub>Cl<sub>2</sub>|MeOH|NH<sub>4</sub>OH 85|10|5) to yield compound G (1.8 g, 69%) as a pale yellow oil. *Analyses were in accordance with the literature.*<sup>2</sup>

**<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C):** δ = 6.96 – 6.76 (m, 7H), 4.18 – 4.11 (m, 8H), 3.92 – 3.82 (m, 16H), 3.69 (s, 2H), 2.59 (t, *J* = 7.3 Hz, 2H), 2.17 (td, *J* = 7.0, 2.6 Hz, 2H), 1.93 (t, *J* = 2.6 Hz, 1H), 1.55 – 1.25 (m, 8H). **<sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz, 25 °C):** δ = 149.0, 148.9, 147.9, 133.9, 121.5, 120.9, 114.2, 114.0, 84.7, 71.3, 70.7, 69.6, 69.5, 68.3, 53.8, 49.4, 30.0, 28.7, 28.4, 26.9, 18.4. **ESI-MS:** *m/z* calcd. for C<sub>33</sub>H<sub>48</sub>NO<sub>8</sub>: 586.34 [M+H]<sup>+</sup>, found: 586.43.



**Figure S3.** <sup>1</sup>H NMR spectra of compound G in CDCl<sub>3</sub> and CD<sub>3</sub>OD at 25 °C.

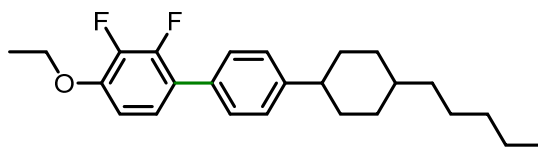
### Pseudo[c2]daisy chain rotaxane (7)



In a 100 mL one-neck round-bottom flask equipped with a stirring bar was prepared a solution of amine G (1.8g, 5.7 mmol) in diethyl ether (30 mL). A 2 M HCl solution in diethyl ether (10 mL) was slowly added to the suspension, which was stirred for one hour at room temperature. The solvent was evaporated and the solid taken back in a biphasic mixture of an aqueous solution of ammonium hexafluorophosphate (2.0 g in 20 mL of water) and dichloromethane (20 mL). The mixture was vigorously stirred for two hours, then the organic phase was extracted with dichloromethane (2 x 10 mL), dried over sodium sulfate, and concentrated under reduced pressure to yield compound 7 (2.0 g, 92%) as a beige solid powder. *Analyses were in accordance with the literature.*<sup>2,5</sup>

**<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C):**  $\delta$  = 6.91 (d,  $J$  = 8.3 Hz, 2H), 6.85-6.70 (m, 10H), 6.57 (s, 2H), 4.44-4.38 (m, 2H), 4.35 - 4.29 (m, 2H), 4.23 - 3.63 (m, 48H), 3.56 - 3.36 (m, 4H), 2.09 (td,  $J$  = 6.9, 1.8 Hz, 4H), 1.92 (br, 2H), 1.48 - 1.14 (m, 16H). **<sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz, 25 °C):**  $\delta$  = 147.9, 147.8, 147.0, 146.5, 146.3, 124.8, 123.2, 121.4, 121.2, 113.4, 112.7, 112.0, 84.4, 72.6, 72.0, 71.3, 71.0, 70.6, 69.6, 68.8, 67.8, 67.2, 67.0, 66.9, 52.4, 49.0, 28.2, 26.8, 26.4, 18.0. **ESI-MS:**  $m/z$  calcd. for C<sub>66</sub>H<sub>96</sub>N<sub>2</sub>O<sub>16</sub>P<sub>2</sub>F<sub>12</sub>: 586.84 [M-2PF<sub>6</sub>]<sup>2+</sup>, found: 586.45.

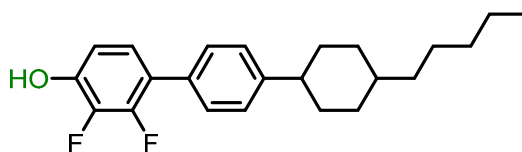
#### 4-ethoxy-2,3-difluoro-4'-(4-pentylcyclohexyl)-1,1'-biphenyl (H)



A 50 mL two-neck round-bottom flask equipped with a stirring bar and a condenser was charged with (4-ethoxy-2,3-difluorophenyl)boronic acid (2.0 g, 9.90 mmol), potassium carbonate (1.65 g, 11.94 mmol) and 1-iodo-4-(4-pentylcyclohexyl)benzene (3.6 g, 10.10 mmol) dissolved in a mixture of water (6 mL), toluene (13 mL) and ethanol (6 mL). The resulting mixture was degassed with argon at room temperature during five minutes under vigorously stirring. Then, tetrakis(triphenylphosphine)palladium (0.12 g, 0.104 mmol) was added leading to a colour change from white to dark brown. The reaction mixture was heated up to 80 °C under continuous stirring for twenty hours and then, cooled at room temperature. The mixture was poured in water (10 mL) and the aqueous phase was extracted with dichloromethane (3 x 10 mL). The organic phases were washed with water (10 mL), dried over sodium sulfate, evaporated under reduced pressure and the crude residue was purified by silica column chromatography using cyclohexane to yield compound **H** as a white solid (3.46 g, 90%). *Analyses were in accordance with the literature.*<sup>6</sup>

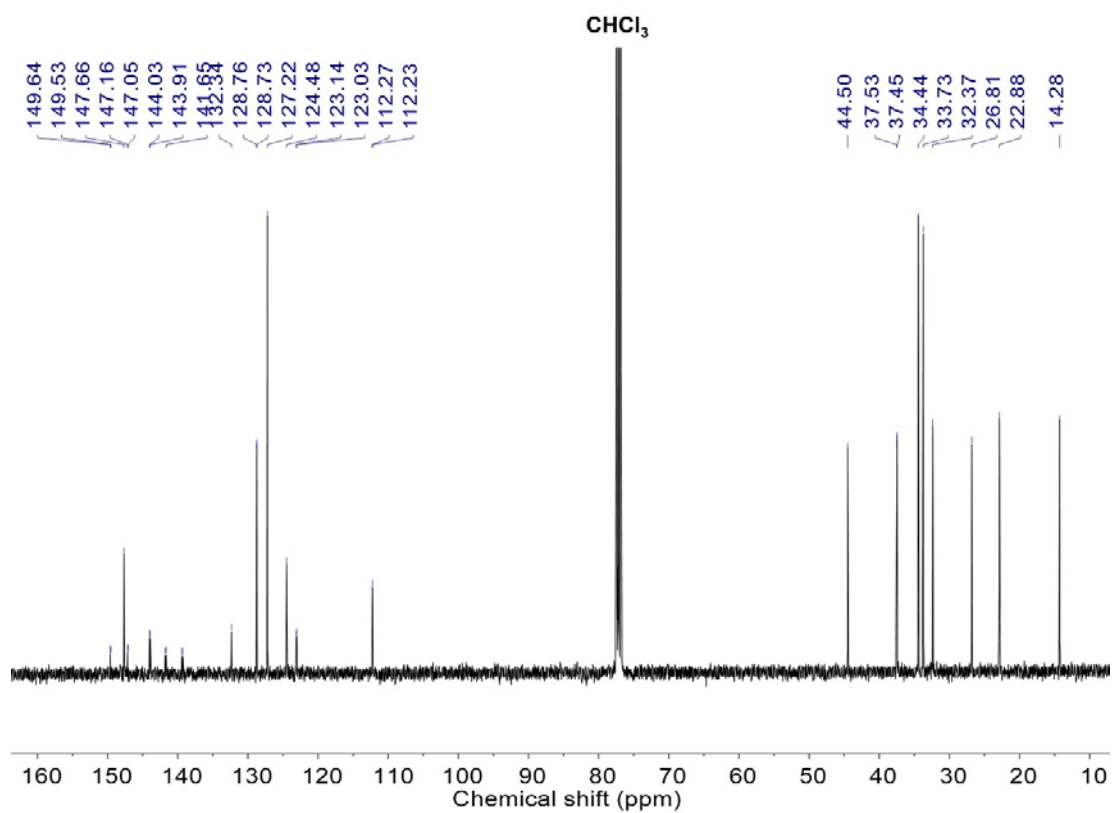
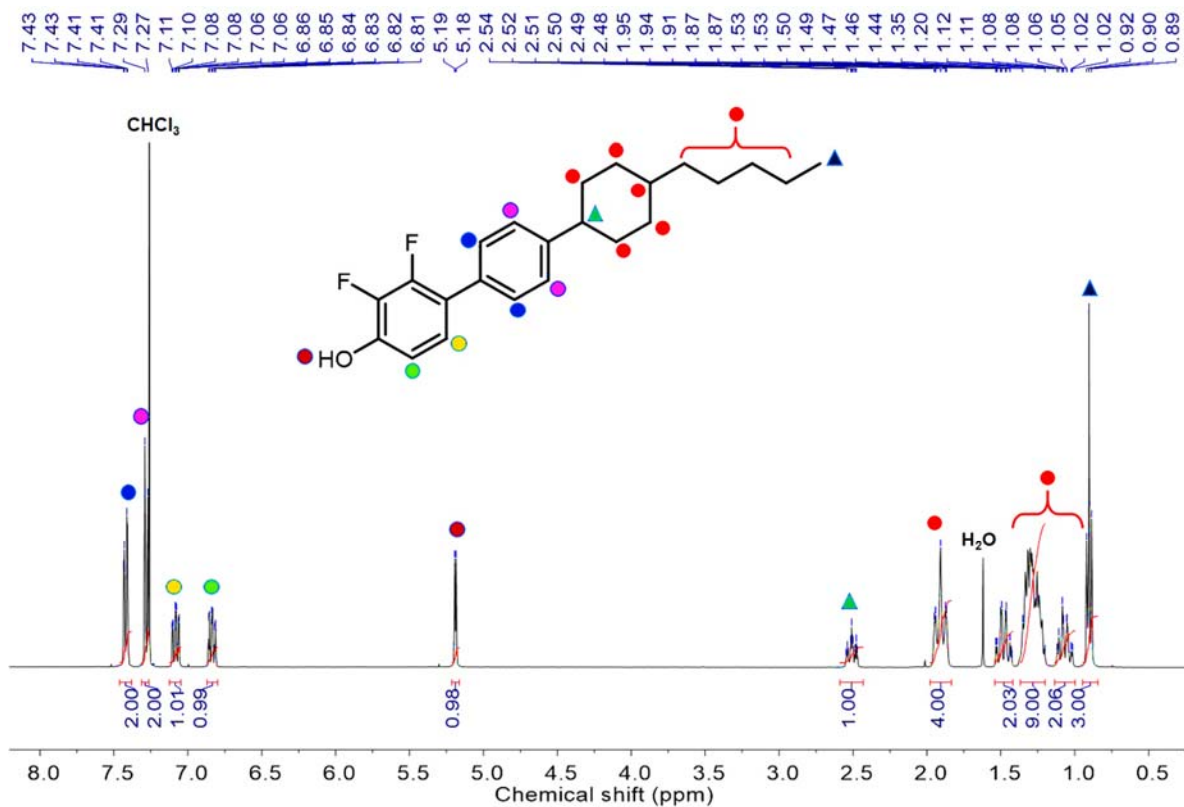
**<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C):**  $\delta$  = 7.43 (d,  $J$  = 8.1 Hz, 2H), 7.28 (d,  $J$  = 8.1 Hz, 2H), 7.09 (td,  $J$  = 8.4, 2.1 Hz, 1H), 6.99–6.94 (m, 1H), 4.13 (s, 2H), 2.51 (t,  $J$  = 12.1 Hz, 1H), 1.91 (t,  $J$  = 14.1 Hz, 2H), 1.49–1.44 (m, 8H), 1.36–1.21 (m, 8H), 1.11 – 1.02 (m, 2H), 0.91 (t,  $J$  = 6.5 Hz, 3H). **<sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz, 25 °C):**  $\delta$  = 149.6, 147.7, 147.1, 144.0, 141.7, 139.3, 132.3, 128.7, 127.2, 124.5, 123.1, 112.3, 64.6, 44.5, 37.5, 37.4, 34.4, 33.7, 32.4, 31.0, 29.6, 26.9, 22.2, 14.8, 14.3.

**2,3-difluoro-4'-(4-pentylcyclohexyl)-[1,1'-biphenyl]-4-ol (1)**



In a 100 mL one-neck round-bottom flask equipped with a stirring bar was dissolved compound **H** (3.46 g, 8.96 mmol) in dried dichloromethane (20 mL). The solution was cooled down at -70 °C using a dry ice: acetone cool bath and boron tribromide (1.15 mL, 12.2 mmol) was slowly added under argon atmosphere. The mixture was stirred during one hour at -70 °C and then, the mixture was allowed to reach room temperature under continuous stirring for 20 hours. Water (10 mL) was added dropwise to avoid exothermic reactions and the aqueous phase was extracted with dichloromethane (2 x 20 mL). The organic phases were dried over sodium sulfate and evaporated under reduced to yield compound **1** (3.13 g, 96%) as a white solid. The product was used as such for the next step without any further purification process. *Analyses were in accordance with the literature.*<sup>6</sup>

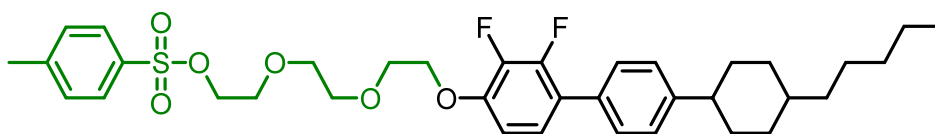
**<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C):**  $\delta$  = 7.42 (dd,  $J$  = 8.3, 1.6 Hz, 2H), 7.28 (d,  $J$  = 8.1 Hz, 2H), 7.08 (td,  $J$  = 8.3, 2.3 Hz, 1H), 6.84 (ddd,  $J$  = 8.7, 8.0, 2.0 Hz, 1H), 5.19 (d,  $J$  = 3.7 Hz, 1H), 2.50 (tt,  $J$  = 12.3, 3.1 Hz, 1H), 1.95–1.86 (m, 4H), 1.48 (qd,  $J$  = 13.9, 3.6 Hz, 2H), 1.35–1.20 (m, 9H), 1.12–1.02 (m, 2H), 0.90 (t,  $J$  = 7.0 Hz, 3H). **<sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz, 25 °C):**  $\delta$  = 149.6, 147.7, 147.1, 144.0, 141.7, 139.3, 132.3, 128.7, 127.2, 124.5, 123.1, 112.3, 44.5, 37.5, 37.4, 34.4, 33.7, 32.4, 26.8, 22.9, 14.3. **ESI-MS:**  $m/z$  calcd. for C<sub>23</sub>H<sub>28</sub>F<sub>2</sub>O: 380.19 [M-H + Na]<sup>+</sup>, found: 380.33.



**Figure S4.** <sup>1</sup>H (top) and <sup>13</sup>C (bottom) NMR spectra of compound 1 in CDCl<sub>3</sub> at 25 °C.

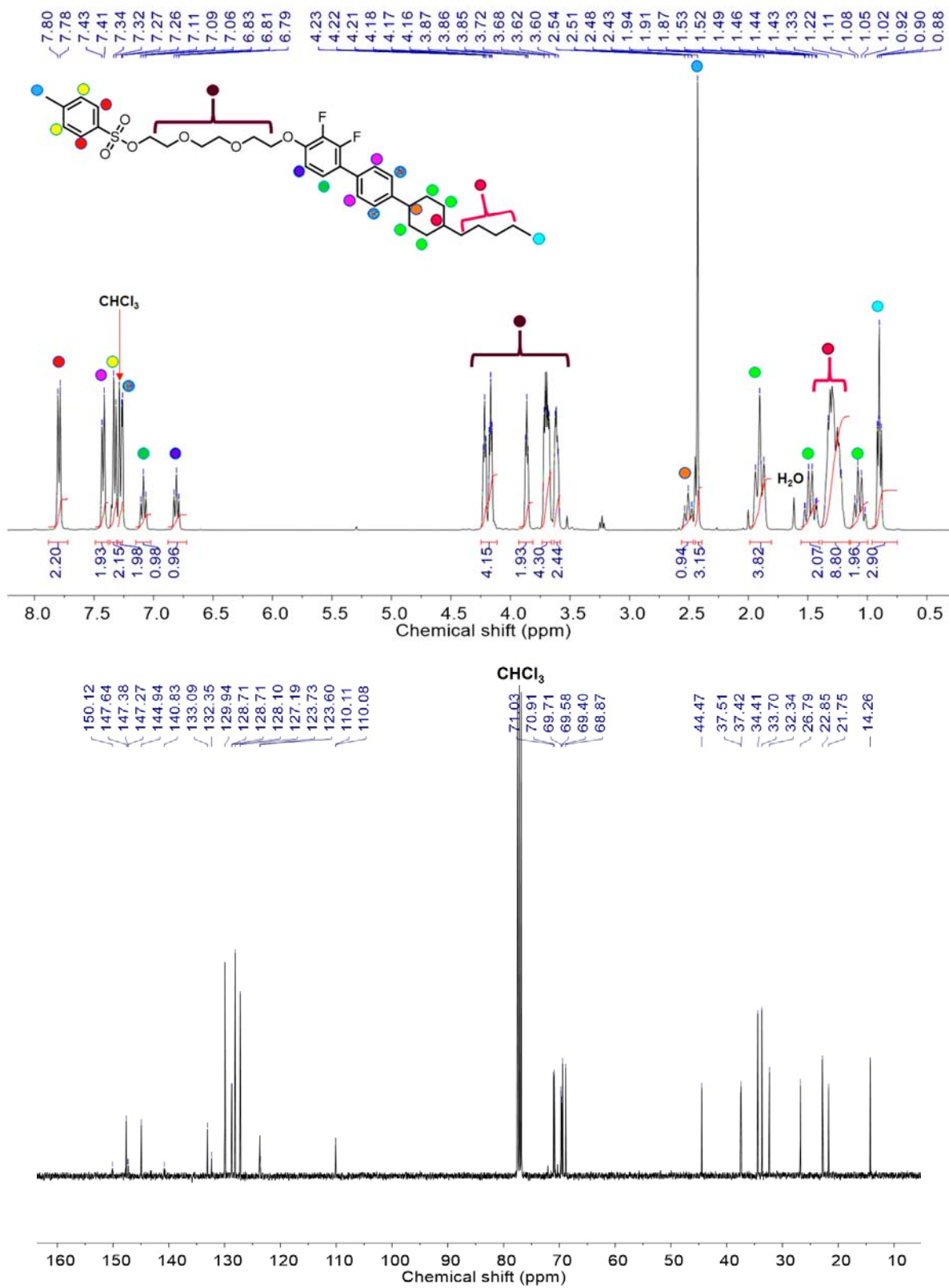


## Compound 2



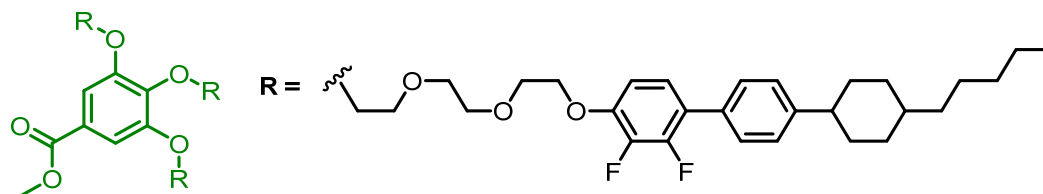
In a 100 mL one-neck round-bottom flask equipped with a stirring bar and a condenser was charged the phenolic derivative **1** (2.75 g, 7.7 mmol), triethylene glycol di(*p*-toluenesulfonate) (10.5 g, 23 mmol), anhydrous potassium carbonate (1.59 g, 11.5 mmol) and potassium iodide (0.13 g, 0.77 mmol). Dry acetone (50 mL) was added and the solution was refluxed under an argon atmosphere during 24 hours. The reaction was cooled down to room temperature, the solvent was removed under reduced pressure and the crude residue was purified by column chromatography (SiO<sub>2</sub>, Cyclohexane/Ethyl Acetate 20%) yielding compound **2** as a white solid (3.0 g, 60%). *Analyses were in accordance with the literature.*<sup>7</sup>

**<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C):** δ = 7.79 (d, *J* = 8.3 Hz, 2H), 7.42 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 8.6 Hz, 2H), 7.28 (d, *J* = 8.5 Hz, 2H), 7.09 (td, *J* = 8.4, 2.3 Hz, 1H), 6.81 (ddd, *J* = 8.9, 7.7, 1.9 Hz, 1H), 4.24 – 4.19 (m, 2H), 4.18 – 4.15 (m, 2H), 3.89 – 3.85 (m, 2H), 3.73 – 3.66 (m, 4H), 3.64 – 3.60 (m, 2H), 2.51 (tt, *J* = 12.3, 3.4 Hz, 1H), 2.43 (s, 3H), 1.90 (td, *J* = 16.2, 2.1 Hz, 4H), 1.48 (dd, *J* = 12.7, 2.8 Hz, 2H), 1.33–1.23 (m, 9H), 1.06 (qd, *J* = 12.3, 11.6, 3.1 Hz, 2H), 0.91 (t, *J* = 6.5 Hz, 3H). **<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz, 25 °C):** δ = 150.1, 147.6, 147.4, 144.9, 140.9, 133.1, 132.3, 129.9, 128.7, 128.1, 127.2, 123.7, 123.6, 110.1, 71.0, 70.9, 69.7, 69.6, 69.4, 68.9, 44.5, 37.5, 37.4, 34.4, 33.7, 32.3, 26.8, 22.9, 21.7, 14.2. **ESI-MS:** *m/z* calcd. for C<sub>36</sub>H<sub>48</sub>F<sub>2</sub>O<sub>7</sub>S: 662.31 [M+H<sub>2</sub>O]<sup>+</sup>, found: 662.54.



**Figure S5.** <sup>1</sup>H (top) and <sup>13</sup>C (bottom) NMR spectra of compound **2** in CDCl<sub>3</sub> at 25 °C.

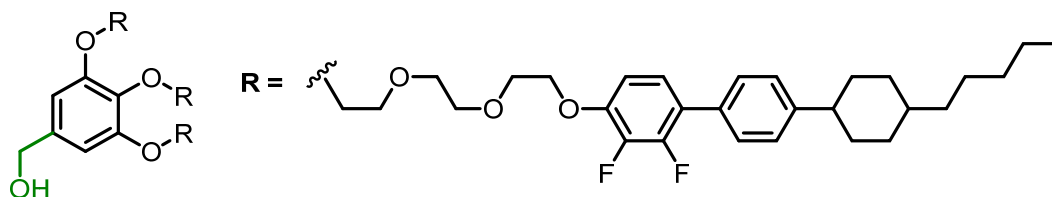
### Compound 3



In a 100 mL one-neck round-bottom flask equipped with a stirring bar and a condenser was dissolved compound **2** (1.4 g, 2.2 mmol), methyl gallate (0.10 g, 0.55 mmol), and potassium carbonate anhydrous (0.45 g, 3.2 mmol) in dried methyl ethyl ketone (5 mL). The reaction mixture was stirred for 24 h at 70 °C under an argon atmosphere. After cooling to room temperature, water (10 mL) was added and the product was extracted with ethyl acetate (3 x 10 mL). The organic layers were washed with brine (2 x 5 mL), dried over sodium sulfate and the solvent was evaporated under reduced pressure. The crude residue was purified by column chromatography (SiO<sub>2</sub>, Cyclohexane/Ethyl acetate 20%→50%→70%) yielding compound **3** (0.4 g, 50%) as a white waxy solid. *Analyses were in accordance with the literature.*<sup>7</sup>

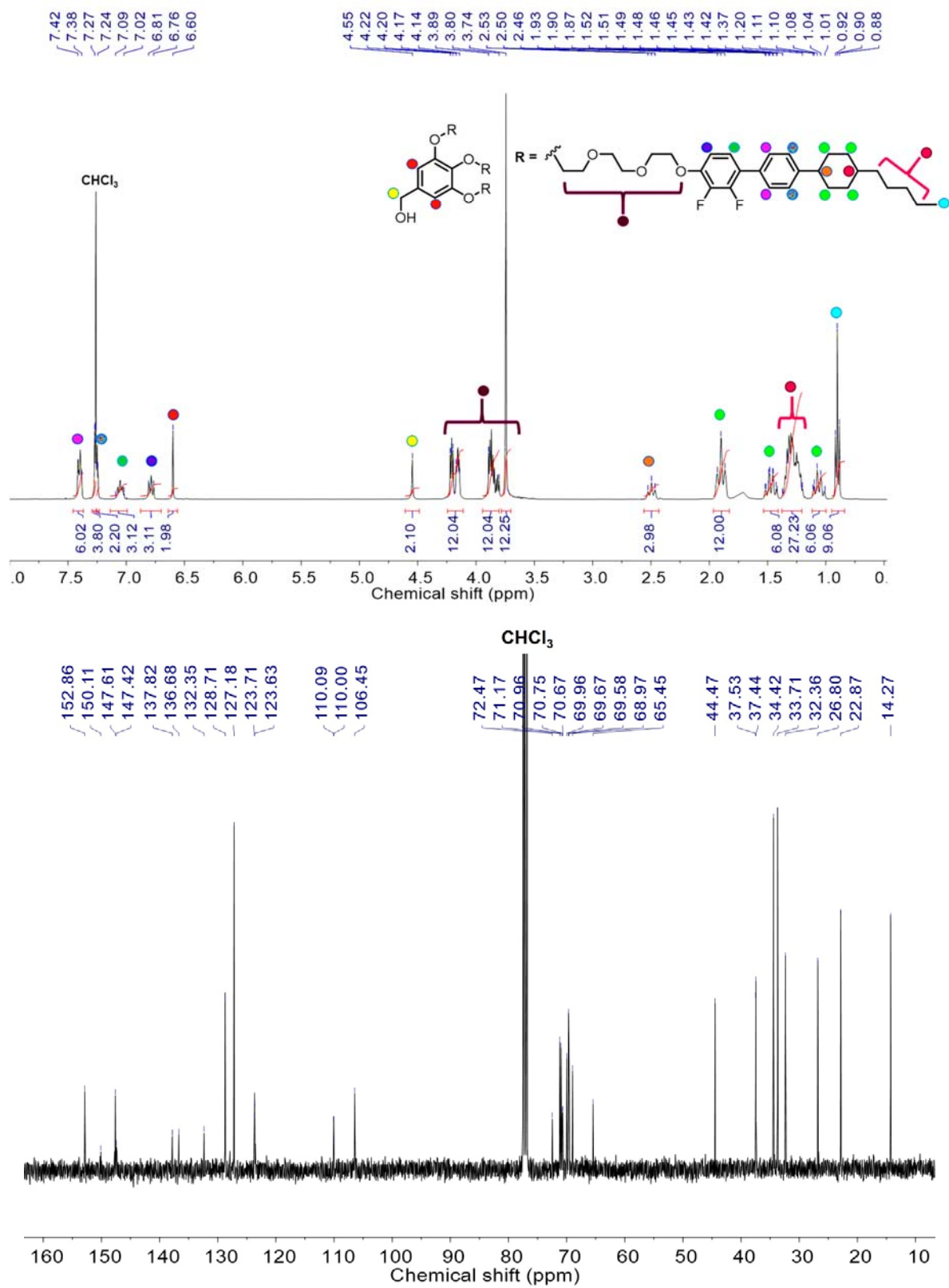
<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C): δ = 7.44-7.38 (m, 6H), 7.31 (s, 2H), 7.29-7.24 (m, 6H), 7.10-7.02 (m, 3H), 6.84-6.75 (m, 3H), 4.26-4.18 (m, 12H), 3.89-3.81 (m, 15H), 3.75-3.73 (m, 12H), 2.49 (t, *J* = 12.1 Hz, 3H), 1.90 (t, *J* = 12.8 Hz, 9H), 1.47 (qd, *J* = 14.2, 3.5 Hz, 6H), 1.36-1.20 (m, 30H), 1.12 - 1.00 (m, 6H), 0.91 (t, *J* = 6.5 Hz, 9H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz, 25 °C): δ = 166.1, 152.9, 150.1, 147.6, 147.4, 137.8, 136.7, 132.4, 128.7, 127.2, 123.7, 123.6, 110.1, 110.0, 106.4, 72.5, 71.2, 70.7, 69.9, 69.6, 68.9, 65.4, 44.5, 37.5, 37.4, 34.4, 33.7, 32.4, 26.8, 22.9, 14.3.

## Compound 4



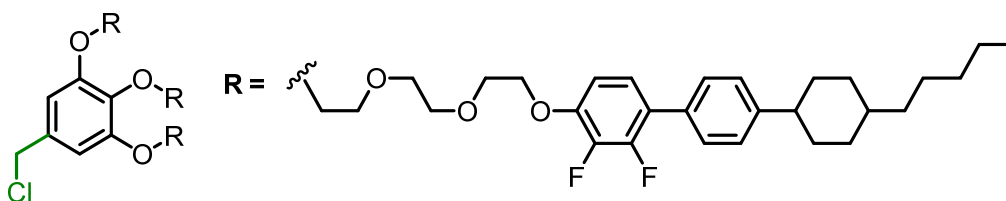
In a 25 mL one-neck round bottom flask equipped with a stirring bar was prepared a solution of ester **3** (0.20 g, 0.12 mmol) in dry tetrahydrofuran (10 mL). The solution was cooled down to 4 °C and lithium aluminium hydride (0.175 g, 0.44 mmol) was added under argon atmosphere. The reaction mixture was stirred at 25 °C for six hours and monitored by thin layer chromatography. When no remaining starting material was observed, a 1 M HCl solution (10 mL) was added at 4 °C and the aqueous phase was extracted with dichloromethane (2 x 20 mL). The combined organic layers were dried over sodium sulfate and evaporated under reduced pressure to yield alcohol **4** (0.18 g, 92%) as a waxy white solid.

**<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C):** δ = 7.42-7.38 (m, 6H), 7.27-7.24 (m, 6H), 7.09-7.02 (m, 3H), 6.81-6.76 (m, 3H), 6.60 (s, 2H), 4.55 (s, 2H) 4.22-4.14 (m, 12H), 3.90-3.80 (m, 12H), 3.74 (s, 12H), 2.50 (t, *J* = 12.0 Hz, 3H), 1.90 (t, *J* = 12.8 Hz, 12H), 1.47 (qd, *J* = 13.9, 3.2 Hz, 6H), 1.36-1.16 (m, 27H), 1.11-1.01 (m, 6H), 0.90 (t, *J* = 7.0 Hz, 9H). **<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz, 25 °C):** δ = 152.9, 150.1, 147.6, 147.4, 137.8, 136.7, 132.4, 128.7, 127.2, 123.7, 123.6, 110.1, 110.0, 106.4, 72.5, 71.2, 70.8, 70.7, 69.9, 69.6, 68.9, 65.4, 44.5, 37.5, 37.4, 34.4, 33.7, 32.4, 26.8, 22.9, 14.3. **HR-MS:** *m/z* calcd. for C<sub>94</sub>H<sub>122</sub>F<sub>6</sub>O<sub>13</sub>: 1596.8716 [M + Na]<sup>+</sup>, found: 1596.8723.



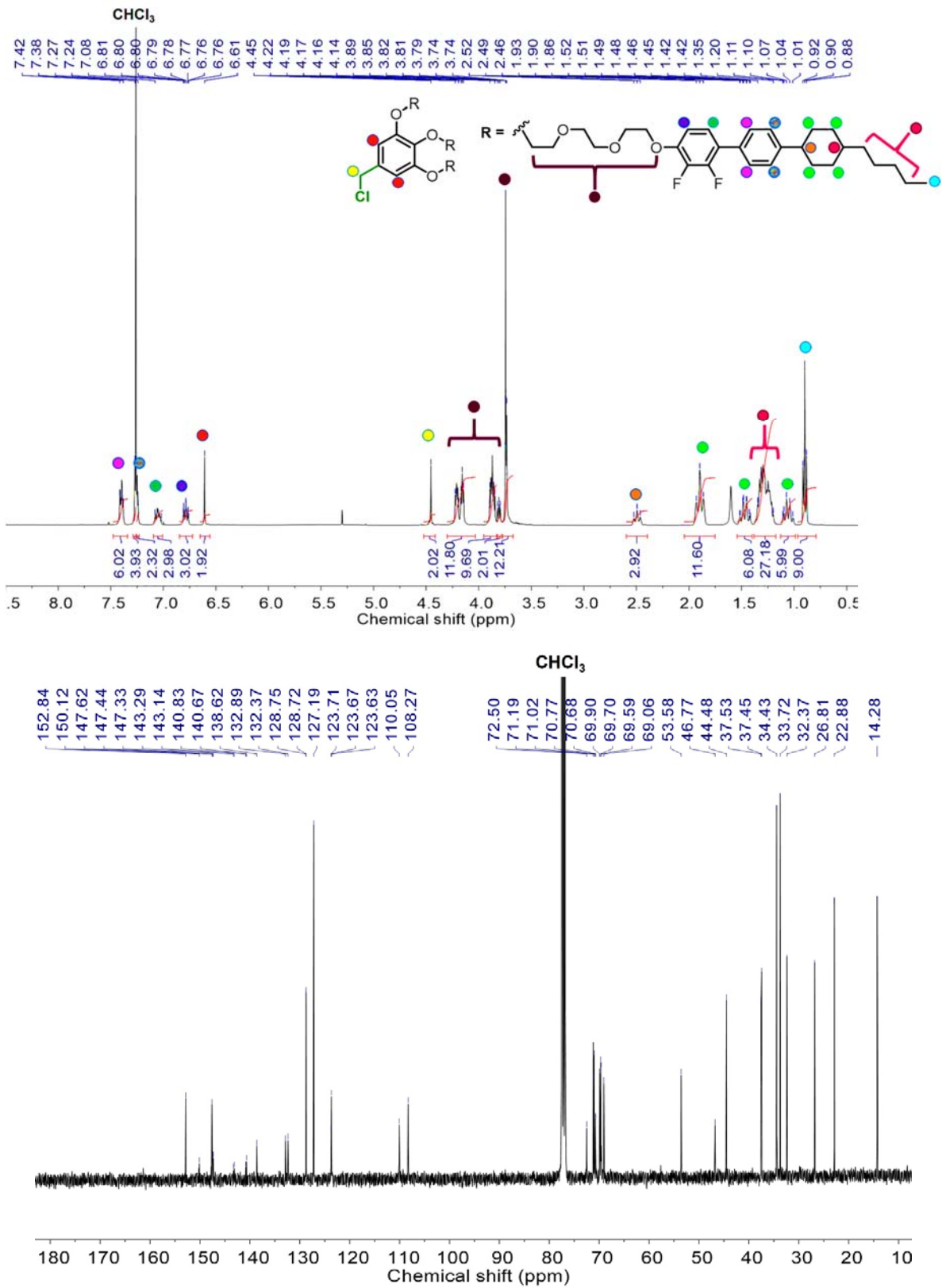
**Figure S6.** <sup>1</sup>H (top) and <sup>13</sup>C (bottom) NMR spectra of compound **4** in CDCl<sub>3</sub> at 25 °C.

## Compound 5



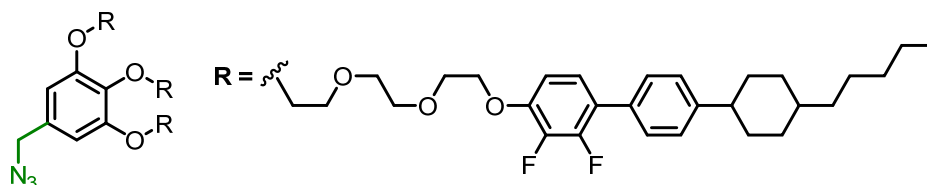
A 25 mL one-neck round bottom flask equipped with a stirring bar was charged with aryl alcohol **4** (0.18 g, 0.11 mmol) and a catalytic amount of dry *N,N*-dimethylformamide (one drop). Dry dichloromethane (2 mL) was added and thionyl chloride (0.019 g, 12  $\mu$ L, 0.16 mmol) was added carefully to the reaction mixture. After continuous stirring for two hours, the volatile compounds were removed, and the residual mixture was dissolved in dichloromethane (10 mL) and washed with water (2 x 5 mL). The solvent was evaporated under reduced pressure yielding compound **5** (0.17 g, 93%) which was pure enough to be used in the next step without further purification.

**<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C):**  $\delta$  = 7.42-7.38 (m, 6H), 7.27-7.24 (m, 6H), 7.08-7.02 (m, 3H), 6.79 (ddd,  $J$  = 9.0, 5.7, 1.9 Hz, 3H), 6.61 (s, 2H), 4.45 (s, 2H) 4.22-4.14 (m, 12H), 3.90-3.85 (m, 10H), 3.82 - 3.79 (m, 2H), 3.75-3.70 (m, 12H), 2.49 (t,  $J$  = 12.0 Hz, 3H), 1.90 (t,  $J$  = 12.9 Hz, 12H), 1.47 (qd,  $J$  = 13.9, 3.4 Hz, 6H), 1.40-1.20 (m, 27H), 1.11-1.01 (m, 6H), 0.90 (t,  $J$  = 7.0 Hz, 9H). **<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz, 25 °C):**  $\delta$  = 152.9, 150.1, 147.6, 147.4, 143.3, 140.8, 138.6, 132.9, 132.4, 128.8, 128.7, 127.2, 123.7, 123.6, 110.1, 108.3, 72.5, 71.2, 70.7, 69.9, 69.6, 69.1, 53.6, 46.8, 44.5, 37.5, 37.4, 34.4, 33.7, 32.4, 26.8, 22.9, 14.3. **HR-MS:**  $m/z$  calcd. for C<sub>94</sub>H<sub>121</sub>ClF<sub>6</sub>O<sub>12</sub>: 1614.8377 [M+Na]<sup>+</sup>, found: 1614.8375.



**Figure S7.** <sup>1</sup>H (top) and <sup>13</sup>C (bottom) NMR spectra of compound **5** in CDCl<sub>3</sub> at 25 °C.

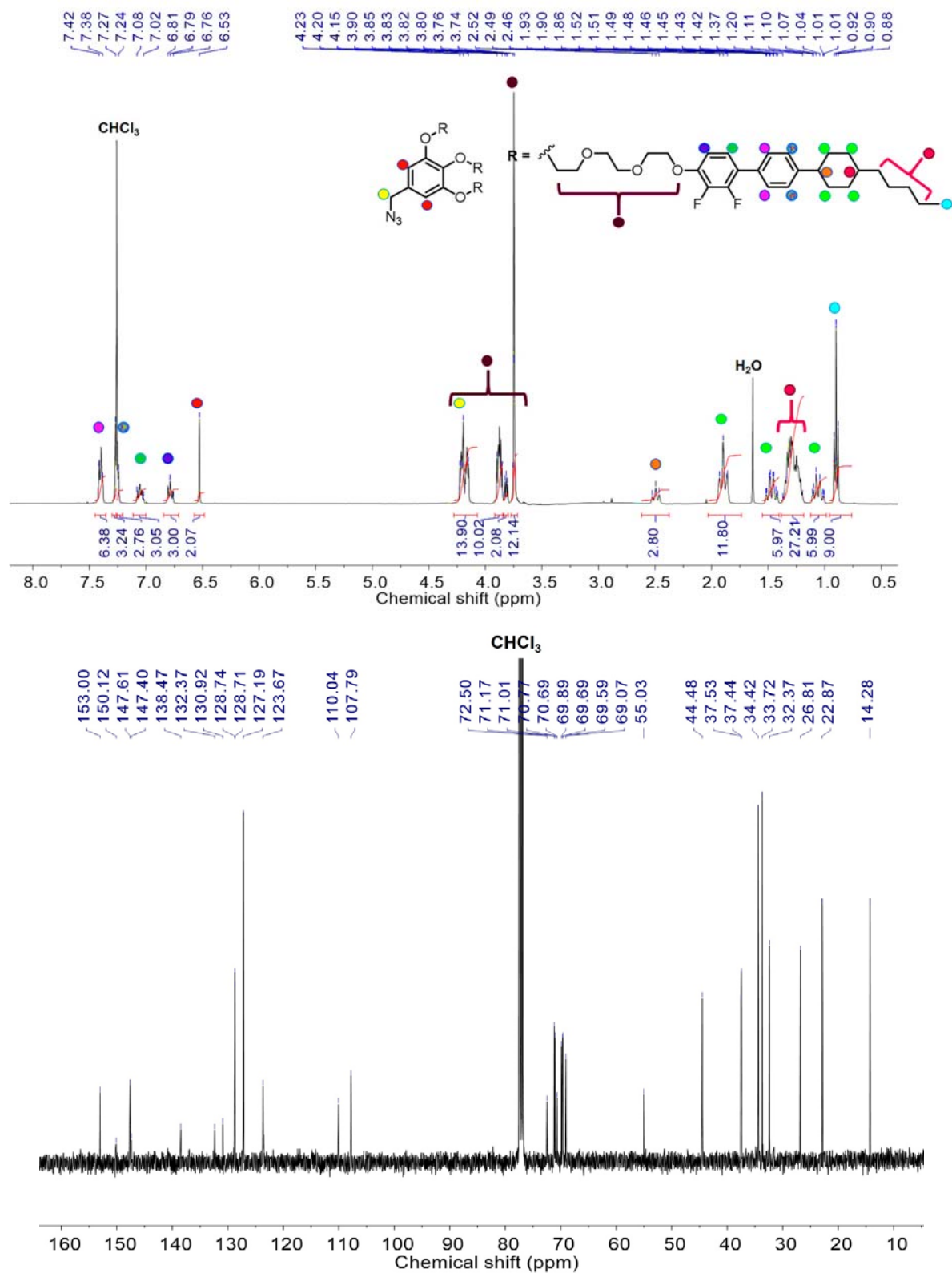
## Compound 6



In a 25 mL one-neck round bottom flask equipped with a stirring bar was dissolved the aryl chloride **5** (0.69 g, 0.43 mmol) in dry *N,N*-dimethylformamide (5 mL). Sodium azide (0.11 g, 1.73 mmol) was added and the solution was stirred for twenty hours at 80 °C. Water (15 mL) was added and the aqueous phase was extracted with dichloromethane (2 x 20 mL). The organic phase was dried over sodium sulfate, the solvent evaporated under reduced pressure and the crude residue purified by column chromatography (SiO<sub>2</sub>, Cyclohexane: Ethyl acetate 30% → 50%) to yield compound **6** (0.55 g, 80%) as a white waxy solid.

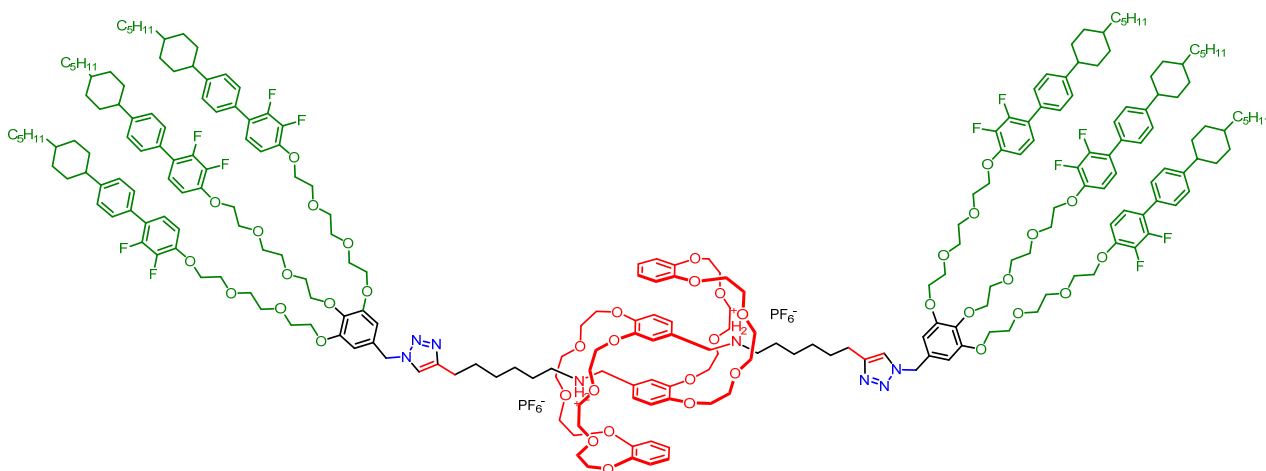
**<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C):** δ = 7.42-7.38 (m, 6H), 7.27-7.24 (m, 6H), 7.08-7.02 (m, 3H), 6.81-6.76 (m, 3H), 6.53 (s, 2H), 4.23-4.15 (m, 14H), 3.90-3.85 (m, 10H), 3.83 - 3.80 (m, 2H), 3.76-3.74 (m, 12H), 2.52 - 2.46 (m, 3H), 1.93-1.86 (m, 12H), 1.52-1.42 (m, 6H), 1.37-1.20 (m, 27H), 1.11-1.01 (m, 6H), 0.90 (t, *J* = 7.0 Hz, 9H). **<sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz, 25 °C):** δ = 153.0, 150.1, 147.6, 147.4, 138.5, 132.4, 130.9, 128.7, 128.7, 127.2, 123.7, 110.0, 107.8, 72.5, 71.2, 71.0, 70.8, 70.7, 69.9, 69.6, 69.1, 55.0, 44.5, 37.5, 37.4, 34.4, 33.7, 32.7, 26.8, 22.9, 14.3. **HR-MS:** *m/z* calcd. for C<sub>94</sub>H<sub>121</sub>F<sub>6</sub>N<sub>3</sub>O<sub>12</sub>: 1621.8781 [M + Na]<sup>+</sup>, found: 1621.8779.





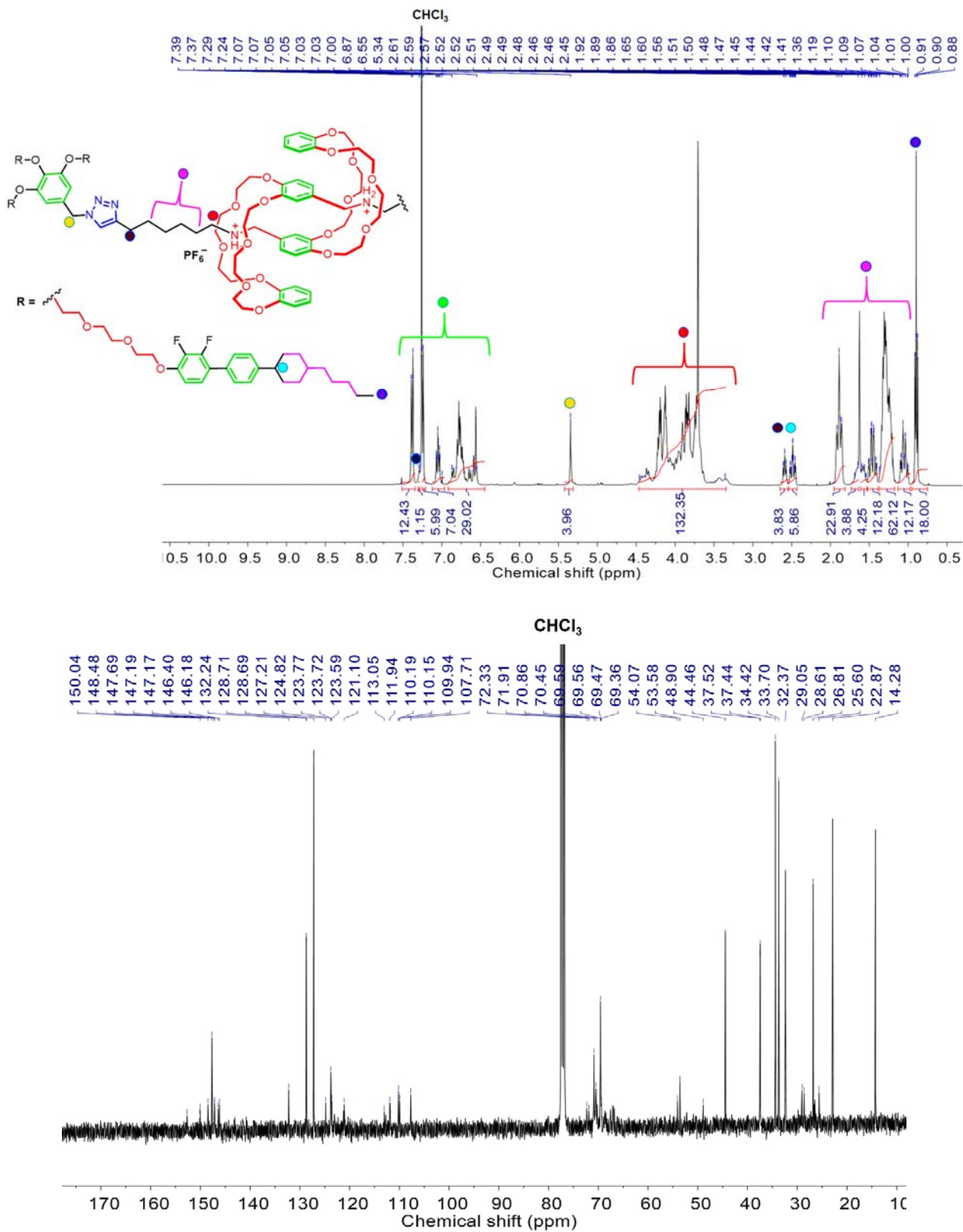
**Figure S8.** <sup>1</sup>H (top) and <sup>13</sup>C (bottom) NMR spectra of compound **6** in CDCl<sub>3</sub> at 25 °C.

## Compound 8



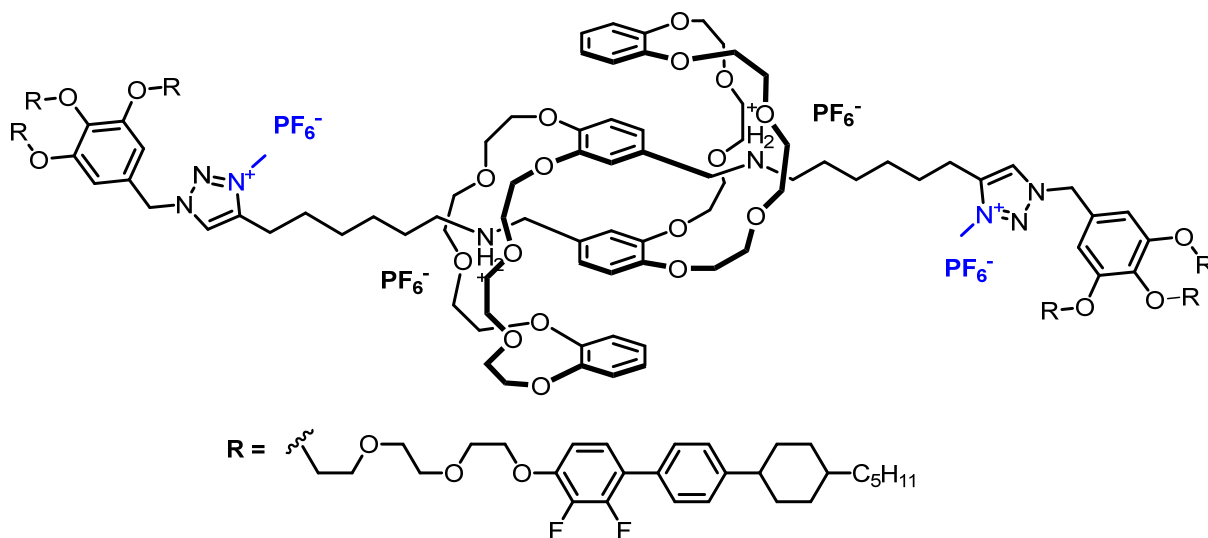
In a microwave tube equipped with a stirring bar were dissolved pseudo[*c*2]daisy chain rotaxane **7** (0.15 g, 0.10 mmol) and benzyl azide **6** (0.35 g, 0.22 mmol) in dry dichloromethane (3 mL).  $\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$  (0.076 g, 0.20 mmol) and 2,6-lutidine (0.016 g, 18  $\mu\text{L}$ , 0.15 mmol) were added and the solution was purged with argon. The reaction was stirred for three hours at 35°C under microwave (MW) irradiation (dynamic mode, 40W), and then it was stirred overnight at 25 °C without MW irradiation. Ethylenediaminetetraacetic acid (2 mL) was added to the reaction mixture which was stirred for 30 minutes. The aqueous phase was extracted with dichloromethane (2 x 10 mL), the solvent was evaporated under reduced pressure and the product was purified by column chromatography ( $\text{SiO}_2$ , dichloromethane:methanol 1%  $\rightarrow$  2%) to yield obtain [*c*2]daisy chain rotaxane **8** as a waxy grey solid (0.135 g, 60%).

$^1\text{H NMR}$  ( $\text{CDCl}_3$ , 400 MHz, 25 °C):  $\delta$  = 7.38 (d,  $J$  = 8.1 Hz, 12H), 7.29 (bs, 2H), 7.24 (s, 6H), 7.05 (td,  $J$  = 8.6, 2.1 Hz, 7H), 6.90 – 6.50 (m, 29H), 5.34 (s, 4H), 4.50 – 3.50 (m, 128H), 3.48 – 3.28 (m, 4H), 2.59 (t,  $J$  = 7.6 Hz, 4H), 2.49 (tt,  $J$  = 12.3, 3.0 Hz, 6H), 1.89 (t,  $J$  = 12.0 Hz, 24H), 1.72 – 1.65 (m, 4H), 1.60 – 1.54 (m, 4H), 1.46 (qd,  $J$  = 13.7, 13.2, 3.4 Hz, 12H), 1.36 – 1.16 (m, 62H), 1.10 – 1.00 (m, 12H), 0.90 (t,  $J$  = 7.0 Hz, 18H).  $^{13}\text{C NMR}$  ( $\text{CDCl}_3$ , 125 MHz, 25 °C):  $\delta$  = 152.7, 150.2, 150.0, 148.5, 147.7, 147.2, 147.1, 146.4, 146.2, 132.2, 128.7, 128.6, 127.2, 124.8, 123.8, 123.7, 123.6, 121.1, 113.0, 111.9, 110.2, 110.1, 109.9, 107.7, 72.3, 71.9, 70.9, 70.4, 70.3, 69.6, 69.5, 69.4, 69.3, 54.1, 53.6, 48.9, 44.5, 37.5, 37.4, 34.4, 33.7, 32.4, 29.0, 26.8, 25.6, 22.9, 14.3. **HR-MS**:  $m/z$  calcd. for  $\text{C}_{254}\text{H}_{338}\text{F}_{12}\text{N}_8\text{O}_{40}$ : 2185.2263 [ $\text{M}-2\text{PF}_6$ ] $^{2+}$ , found: 2185.2241.



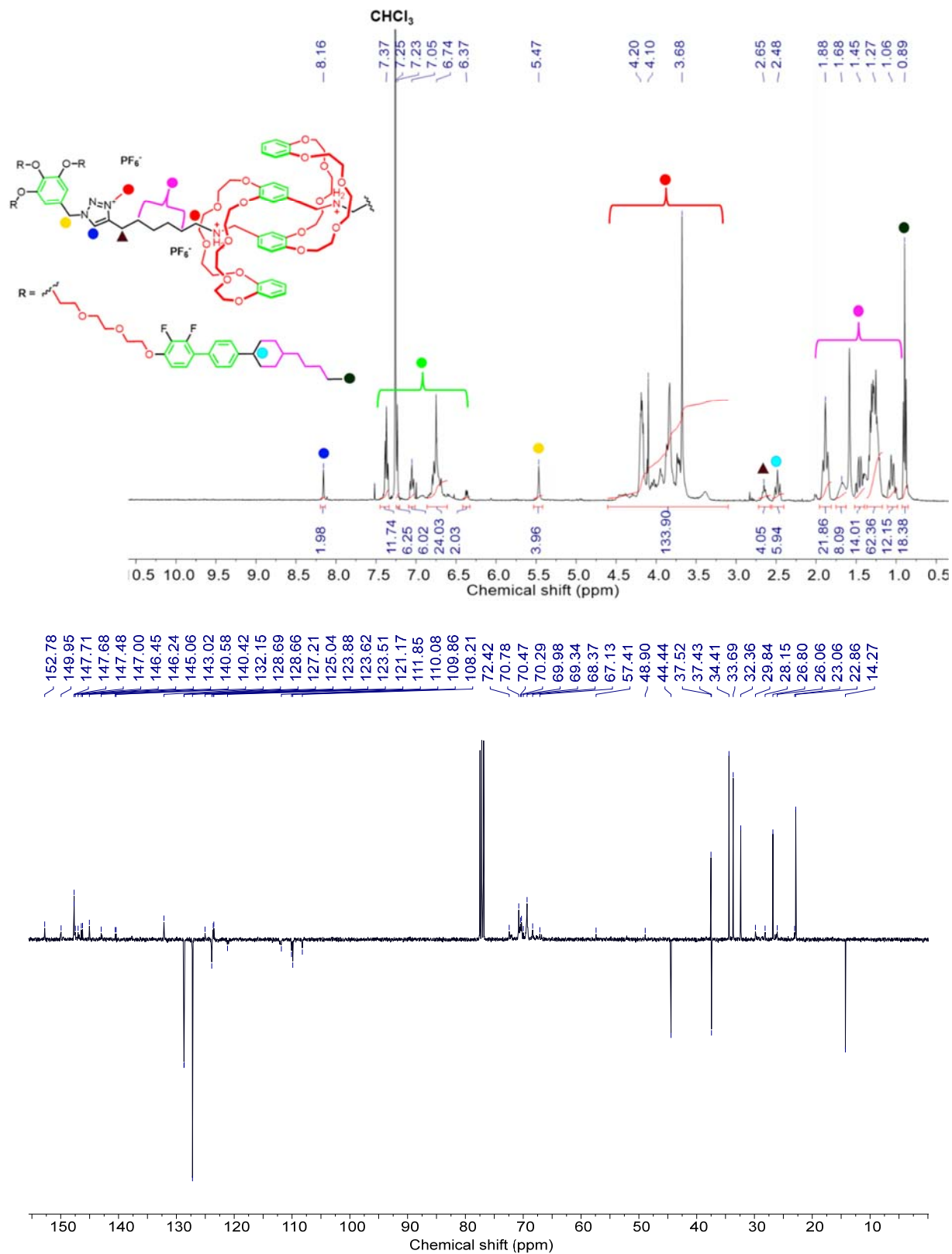
**Figure S9.** <sup>1</sup>H (top) and <sup>13</sup>C (bottom) NMR spectra of compound **8** in CDCl<sub>3</sub> at 25 °C.

## Compound 9<sub>Ext</sub>



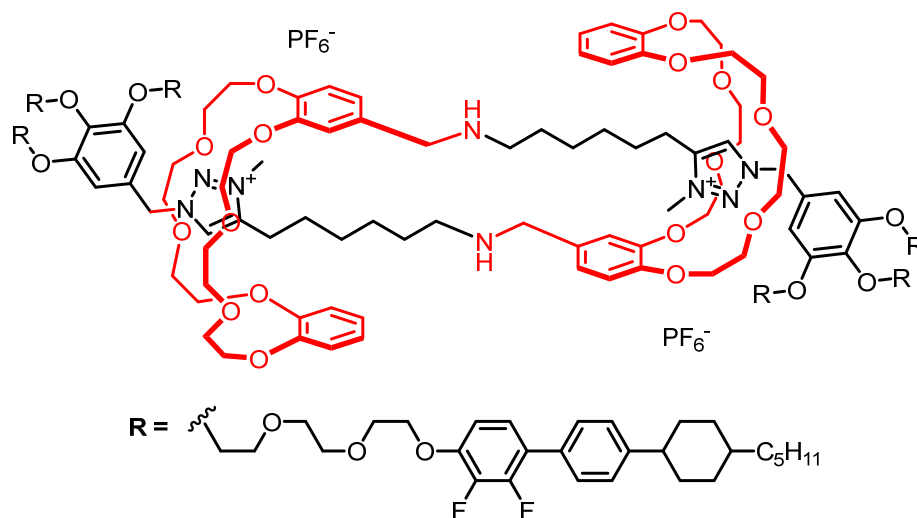
In a 10 mL one-neck round-bottom flask equipped with a stirring bar was dissolved [c2]daisy chain rotaxane **8** (0.10 g, 0.022 mmol) in methyl iodide (2 mL) and dry chloroform (1 mL). The mixture was stirred during four days under inert atmosphere at 30 °C. The solvent was evaporated to remove methyl iodide, then a 1:1 solution of CH<sub>2</sub>Cl<sub>2</sub>: NH<sub>4</sub>PF<sub>6</sub> (aq) (6 mL) was added and the solution was stirred for two hours. The aqueous phase was extracted with dichloromethane (2 x 10 mL), the organic layer was dried over sodium sulfate and the solvent was evaporated under reduced pressure affording compound **9<sub>Ext</sub>** (0.11 g, 96%) as a pale yellow solid.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C): δ = 8.16 (s, 2H), 7.45 – 7.35 (m, 12H), 7.25 – 7.23 (m, 12H), 7.05 (ddt, J = 8.3, 5.9, 2.3 Hz, 6H), 6.85 – 6.75 (m, 24H), 6.37 (d, J = 8.5 Hz, 2H), 5.47 (s, 4H), 4.50 – 3.20 (m, 134H), 2.70 – 2.60 (m, 4H), 2.48 (tt, J = 12.0, 2.8 Hz, 6H), 1.89 (t, J = 11.7 Hz, 22H), 1.75 – 1.65 (m, 8H), 1.46 (qd, J = 14.4, 3.8 Hz, 14H), 1.36 – 1.16 (m, 62H), 1.10 – 1.01 (m, 12H), 0.89 (t, J = 7.0 Hz, 18H).  
<sup>13</sup>C NMR (CDCl<sub>3</sub>, 101 MHz, 25 °C): δ = 152.8, 150.1, 150.0, 147.7, 147.7, 147.6, 147.5, 147.0, 147.0, 146.9, 146.8, 146.5, 146.2, 145.1, 143.0, 142.9, 140.6, 140.4, 132.2, 128.7, 128.7, 127.2, 125.0, 123.9, 123.6, 123.5, 121.2, 111.9, 110.1, 109.9, 108.2, 72.4, 70.8, 70.6, 70.5, 70.3, 70.0, 69.5, 69.4, 69.3, 69.2, 68.4, 67.1, 57.4, 48.9, 44.4, 37.5, 37.4, 34.4, 33.7, 32.4, 29.8, 28.2, 26.8, 26.4, 26.2, 26.1, 23.1, 22.9, 14.3. **HR-MS**: m/z calcd. for C<sub>256</sub>H<sub>342</sub>F<sub>18</sub>N<sub>8</sub>O<sub>40</sub>P: 4543.4486 [M-3PF<sub>6</sub>-2H]<sup>+</sup>, found: 4543.4433.



**Figure S10.** <sup>1</sup>H (top) and <sup>13</sup>C (bottom) NMR spectra of compound **9<sub>Ext</sub>** in CDCl<sub>3</sub> at 25 °C.

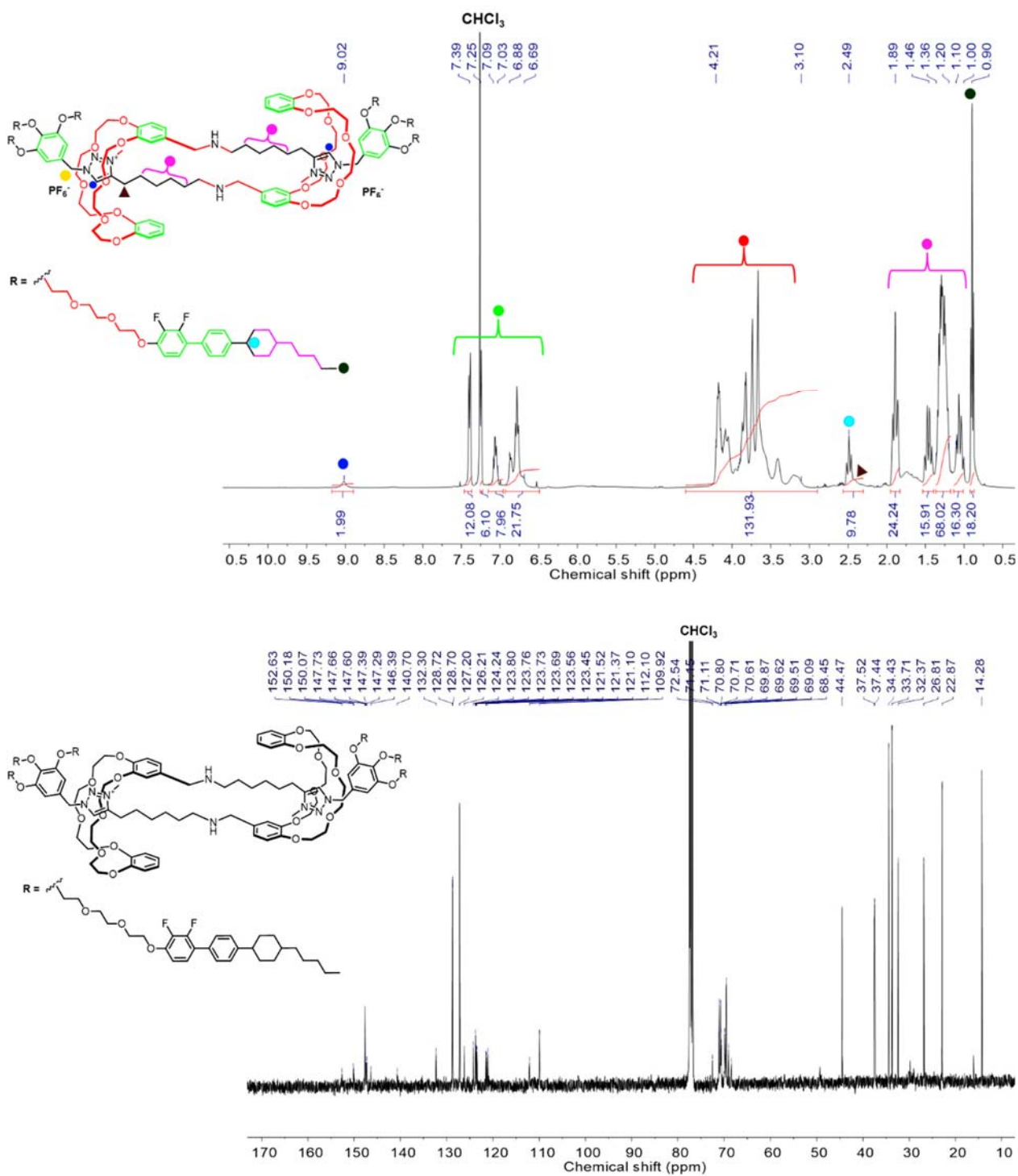
## Compound **9**<sub>Cont</sub>



In a 10 mL one-neck round-bottom flask equipped with a stirring bar was dissolved compound **9**<sub>Ext</sub> (0.10 g, 0.020 mmol) in chloroform (5 mL). Then, a 1 M NaOH solution (1 mL) was added, and the solution was stirred vigorously for two minutes. The aqueous phase was extracted with chloroform (2 x 5 mL) and washed with water (5 mL). The combined organic layers were dried over sodium sulfate and the solvent was evaporated under reduced pressure to yield contracted [c2]daisy chain **9**<sub>Cont</sub> (0.090 g) quantitatively as a beige solid.

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C):  $\delta$  = 9.02 (s, 2H), 7.40 – 7.35 (m, 12H), 7.27 – 7.24 (m, 6H), 7.10 – 7.00 (m, 8H), 6.95 – 6.56 (m, 22H), 4.60 – 2.90 (m, 132H), 2.53 – 2.35 (m, 10H), 1.89 (t,  $J$  = 12.4 Hz, 24H), 1.46 (qd,  $J$  = 13.7, 13.3, 3.2 Hz, 16H), 1.40 – 1.20 (m, 68H), 1.10 – 1.00 (m, 16H), 0.90 (t,  $J$  = 7.0 Hz, 18H).

<sup>13</sup>C NMR (CDCl<sub>3</sub>, 101 MHz, 25 °C):  $\delta$  = 152.6, 150.2, 150.1, 147.7, 147.6, 147.4, 147.3, 146.4, 140.7, 132.3, 128.7, 127.2, 126.2, 124.2, 123.8, 123.7, 123.6, 123.5, 121.5, 121.4, 121.1, 112.1, 109.9, 72.5, 71.2, 71.1, 70.8, 70.7, 70.6, 69.9, 69.6, 69.5, 69.1, 68.4, 44.5, 37.5, 37.4, 34.4, 33.7, 32.4, 26.8, 22.9, 14.3. **HR-MS**:  $m/z$  calcd. for C<sub>256</sub>H<sub>342</sub>F<sub>12</sub>N<sub>8</sub>O<sub>40</sub>: 2199.2419 [M-2PF<sub>6</sub>]<sup>2+</sup>, found: 2199.2453.

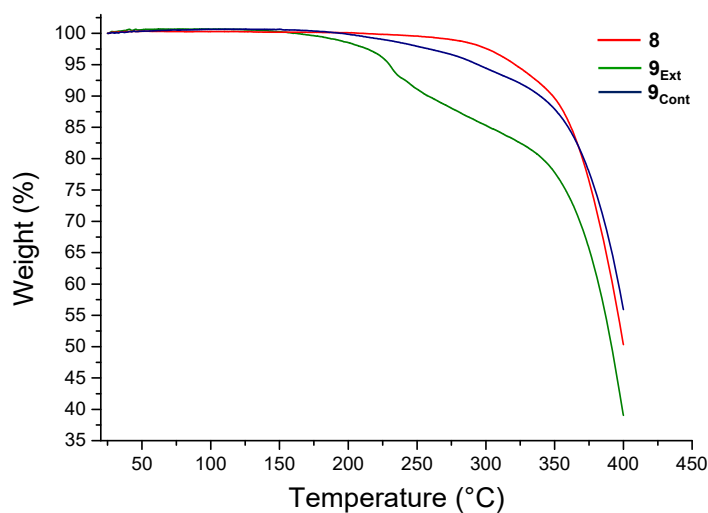


**Figure S11.** <sup>1</sup>H (top) and <sup>13</sup>C (bottom) NMR spectra of compound **9<sub>cont</sub>** in CDCl<sub>3</sub> at 25 °C.

### Re-extension from **9<sub>cont</sub>** to **9<sub>ext</sub>**:

The reversibility of the motion was obtained by dissolving compound **9<sub>cont</sub>** (0.006 g) in deuterated chloroform (0.6 mL). Then, deuterated trifluoroacetic acid (10  $\mu$ L, 10 equiv) was added and the tube was shaken manually for a few seconds. The  $^1\text{H}$  NMR experiment was recorded displaying a spectrum similar to the one obtained for the extended molecule **9<sub>ext</sub>**. The extension / contraction procedures can be repeated several times without degradation of the product.

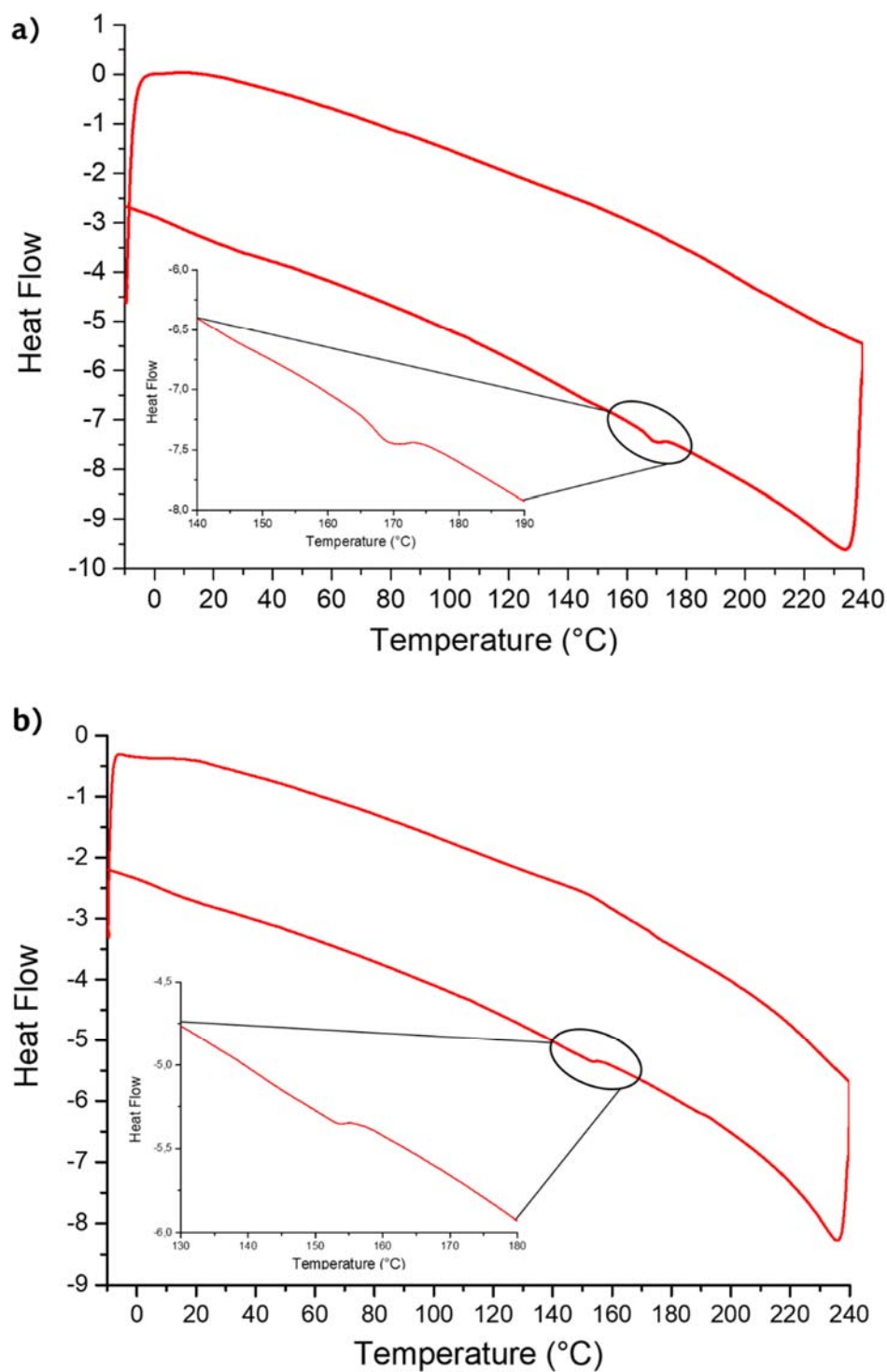
### 3. TGA experiments



**Figure S12.** Thermogravimetric analysis (TGA) thermograms of **8** (red), **9<sub>Ext</sub>** (green) and **9<sub>Cont</sub>** (blue).

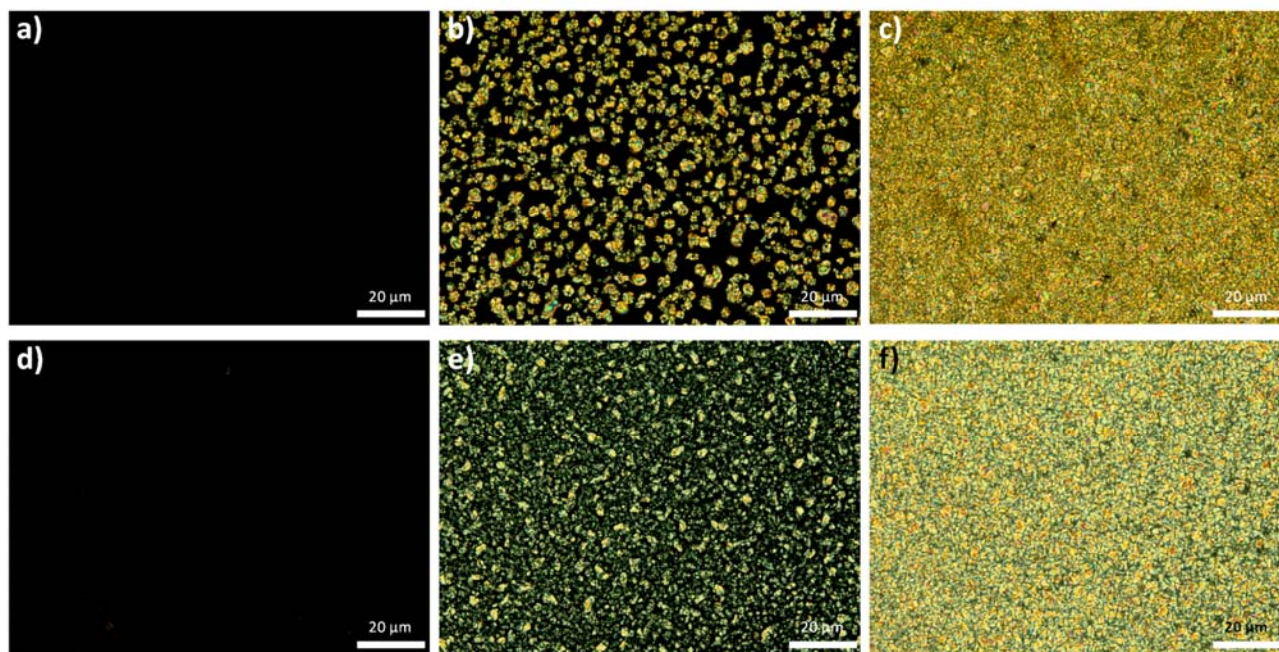


#### 4. DSC experiments



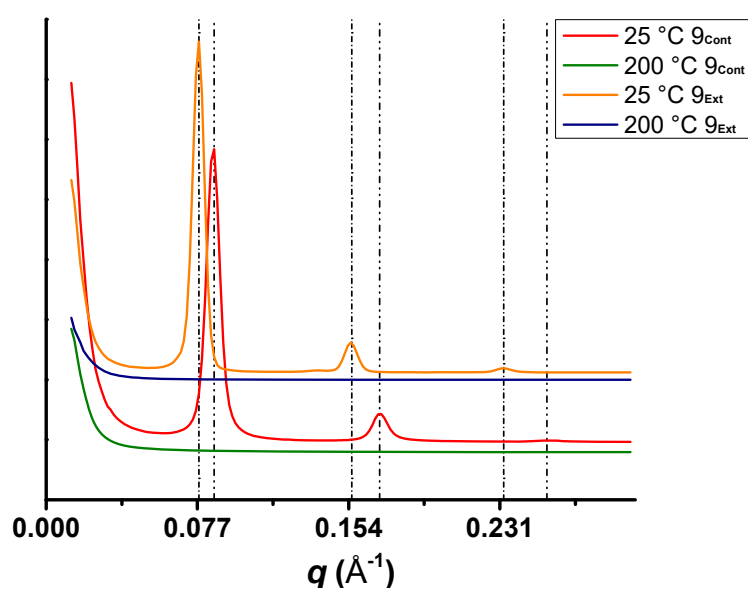
**Figure S13.** Differential scanning calorimetry (DSC) thermograms of  $9_{\text{Ext}}$  (top) and  $9_{\text{Cont}}$  (bottom) in the temperature range from -10 °C to 240 °C for heating and cooling with a rate of 5 °C/min under nitrogen.

## 5. POM experiments



**Figure S14.** Typical POM images of (a-c)  $9_{\text{Ext}}$  and (d-f)  $9_{\text{Cont}}$  under crossed polarizer in the isotropic phase (a,d), at the isotropic-LC phase transition (b,e), and at room temperature (c,f).

## 6. X-ray scattering experiments



**Figure S15.** Scattered intensity  $I(q)$  for compounds  $9_{\text{Cont}}$  and  $9_{\text{Ext}}$  at room temperature and at 200°C.

## 7. References

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