

Supporting Information

Disclosing chirality in consecutive supramolecular polymerizations. Chiral induction by light in *N*-annulated perylenetetracarboxamides

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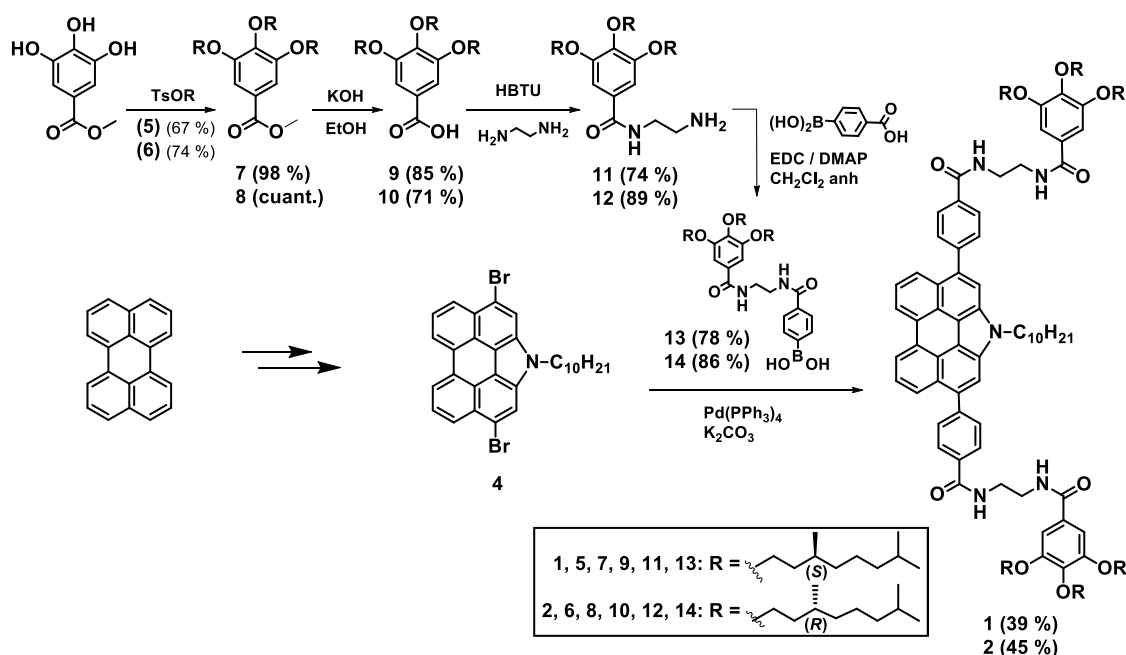
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1. Experimental section

General. All solvents were dried according to standard procedures. Reagents were used as purchased. All air-sensitive reactions were carried out under argon atmosphere. Flash chromatography was performed using silica gel (Merck, Kieselgel 60, 230-240 mesh or Scharlau 60, 230-240 mesh). Analytical thin layer chromatography (TLC) was performed using aluminium-coated Merck Kieselgel 60 F254 plates. NMR spectra were recorded on a Bruker Avance 300 (^1H : 300 MHz; ^{13}C : 75 MHz) spectrometer at 298 K using partially deuterated solvents as internal standards. Coupling constants (J) are denoted in Hz and chemical shifts (δ) in ppm. Multiplicities are denoted as follows: s = singlet, d = doublet, t = triplet, m = multiplet, br = broad. FT-IR spectra were recorded on a Bruker Tensor 27 (ATR device) spectrometer. UV-Vis spectra were recorded on a Varian Cary 50 spectrophotometer. High resolution mass spectra (HRMS) were recorded on a FTMS Bruker APEX Q IV spectrometer. Circular dichroism (CD) measurements were performed on a JASCO-1500 dichrograph equipped with a Peltier thermoelectric temperature controller. The spectra were recorded in the continuous mode between 700 and 200 nm, with a wavelength increment of 1 nm, a response time of 4 s, and a bandwidth of 1 nm. A 1 cm path-length quartz cuvette (Hellma) was used. VCD spectra were recorded in a JASCO FVS-6000 over an average of 6000 scans using a cell equipped with BaF_2 lens and a Teflon spacer generating a 150 μm path length. CPL spectra were recorded on a JASCO CPL-300 at a scanning rate of 100 nm/min over an average of 5 scans, using a 10 mm quartz cell. CPL irradiation was performed with a JASCO CPL irradiation instrument equipped with a 500 W Deep UV lamp and a Babinet-Soleil Compensator using a 10 mm quartz cell.

Preparation of AggI and AggII species. In good agreement with our previous results on achiral compound **3**^{S1} a stock solution of chiral **1** and **2** in MCH as solvent and at $c_T = 500 \mu\text{M}$ is initially prepared. A dilution of this stock solution is carried out to attain the desired final concentration (10 μM). Registering the UV-Vis spectra of this diluted solution results in the broad absorption spectrum shown in Figure 2a that coincides with that reported for AggII species of achiral **3** (reference 10 of the manuscript). Heating this 10 μM solutions of **1** or **2** to 90 $^\circ\text{C}$, yields a heating curve with two clear transitions ascribable to AggI and the monomeric species. Once the initial solution is completely disassembled to the monomeric species, a further cooling of this solution to 20 $^\circ\text{C}$ allows the reassembly of this monomeric species to form AggI species, as demonstrate the UV-Vis spectra shown in Figure 2b. A similar strategy is followed for the solutions in which Tol is utilized as solvent. However, in this case, only more concentrated solutions of **1** and **2** ($c_T = 100 \mu\text{M}$ or higher) allows detecting the formation of AggII. In good correlation with that reported for achiral **3**, the UV-Vis spectra of diluted solutions of **1** and **2** ($c_T = 20 \mu\text{M}$) yields AggI species as the only aggregated state, as demonstrates the identical absorption pattern to that registered for the MCH solution upon applying the heating/cooling cycle mentioned above.

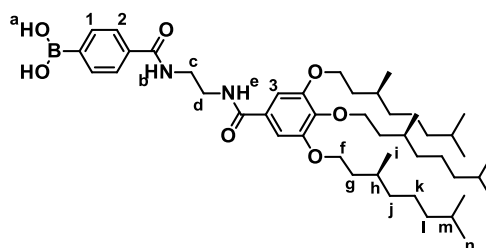
2. Synthetic details and characterization



Scheme S1. Synthesis of the reported *N*-annulated tricarboxamides **1-3**.

Compounds **4-12** were prepared according to previously reported synthetic procedures and showed identical spectroscopic properties to those reported therein.^{S2,S3}

4-(2-(3,4,5-tris((*S*)-3-methyloctyloxy)benzamido)ethylcarbamoyl)phenylboronic acid (**13**)

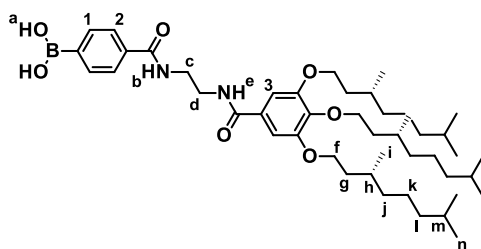


$C_{46}H_{77}BN_2O_7$
 Exact Mass: 780.5824
 Mol. Wt.: 780.9238

4-(Dihydroxyboryl)benzoic acid (0.18 g, 1.06 mmol) was dissolved in dry DMSO (2 mL) and in dry methylene chloride (17 mL) under Argon atmosphere. The solution was cooled to 0 °C and 4-dimethylaminopyridine (0.14 g, 1.17 mmol) and 1-ethyl-3-(3-dimethylaminopropyl)-carbodiimide hydrochloride (0.22 g, 1.17 mmol) were slowly added. The mixture was stirred for 30 minutes. After that, compound **11** (0.74 g, 1.17 mmol) was added portionwise. The reaction mixture was stirred at room temperature for 66 hours. The organic layer was washed with water, HCl 1 M, dried over $MgSO_4$ and evaporation of the solvent under reduced pressure. The residue was purified by column chromatography (silica gel, chloroform/methanol 10/1) affording compound **13** as a white solid (0.65 g, 78 %). 1H NMR (DMSO- d_6 , 500 MHz, 328 K) δ 8.61 (1H, H_e , br), 8.51 (1H, H_b , br), 8.18 (2H, H_a , s), 7.85 (2H, H_2 , d, $J=8.1$ Hz), 7.80 (2H, H_1 , d, $J=8.1$

Hz), 7.13 (2H, H₃, s), 4.03 (4H, H_f, m), 3.93 (2H, H_f, m), 3.46 (4H, H_{c+d}, br), 1.77 (3H, H_m, m), 1.70 (3H, H_h, br), 1.51 (6H, H_g, m), 1.33–1.14 (18H, H_{j+k+l}, br), 0.92 (6H, H_i, d, *J*=6.6 Hz), 0.89 (3H, H_i, d, *J*=6.6 Hz), 0.85 (18H, H_n, d, *J*=6.6 Hz); ¹³C NMR (DMSO-*d*₆, 125 MHz, 328 K) δ 166.6, 165.9, 152.0, 139.8, 135.6, 133.6, 129.3, 125.7, 105.9, 70.5, 66.7, 38.5, 38.4, 36.6, 36.6, 36.5, 35.7, 29.0, 28.9, 27.1, 23.9, 23.8, 22.2, 22.1, 19.2, 19.1; FTIR (neat) 715, 761, 858, 1016, 1045, 1115, 1231, 1337, 1365, 1380, 1427, 1465, 1497, 1542, 1580, 1636, 2870, 2926, 2954, 3317 cm⁻¹. HRMS (MALDI-TOF) calcd. for C₄₆H₇₈BN₂O₇ [M+H]⁺, 781.590; found, 781.358.

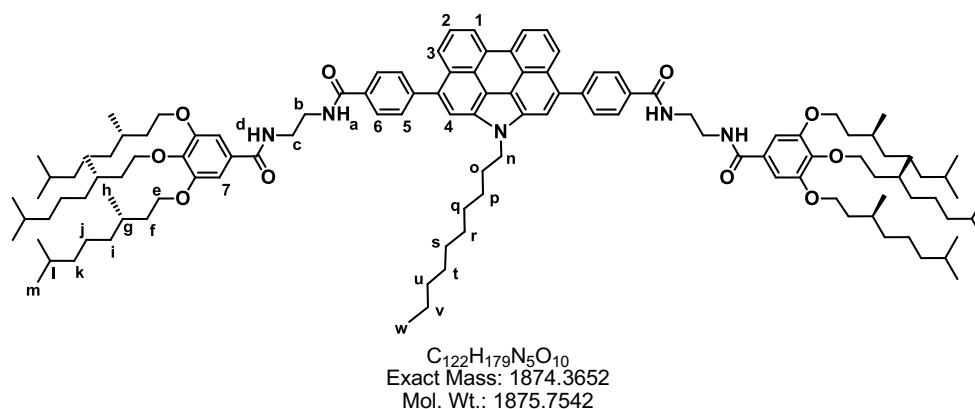
4-(2-(3,4,5-tris((*R*)-3-methyloctyloxy)benzamido)ethylcarbamoyl)phenylboronic acid (**14**)



C₄₆H₇₇BN₂O₇
 Exact Mass: 780.5824
 Mol. Wt.: 780.9238

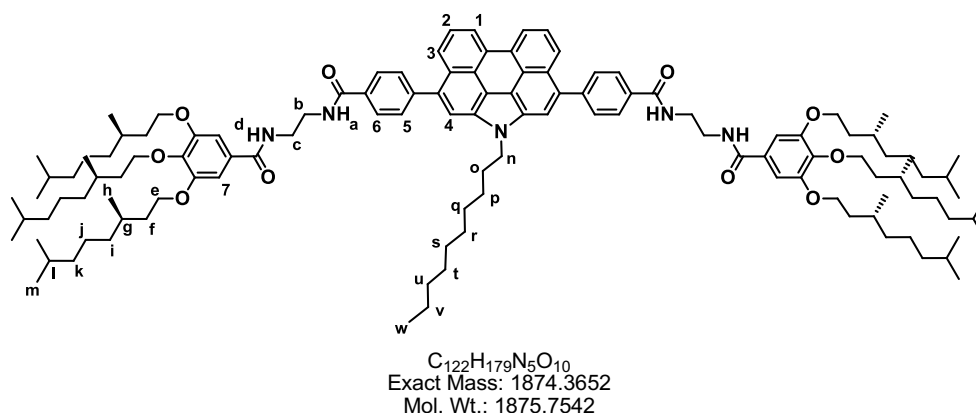
4-(Dihydroxyboryl)benzoic acid (0.19 g, 1.12 mmol) was dissolved in dry DMSO (1 mL) and in dry methylene chloride (14 mL) under Argon atmosphere. The solution was cooled to 0 °C and 4-dimethylaminopyridine (0.15 g, 1.23 mmol) and 1-ethyl-3-(3-dimethylaminopropyl)-carbodiimide hydrochloride (0.24 g, 1.23 mmol) were slowly added. The mixture was stirred for 30 minutes. After that, compound **12** (0.78 g, 1.23 mmol) was dissolved in dry methylene chloride (4mL) and added portionwise. The reaction mixture was stirred at room temperature for 44 hours. The organic layer was washed with water, HCl 1 M, dried over MgSO₄ and evaporation of the solvent under reduced pressure. The residue was purified by column chromatography (silica gel, chloroform/methanol 10/1) affording compound **14** as a white solid (0.76 g, 86 %). ¹H NMR (DMSO-*d*₆, 300 MHz, 328 K) δ 8.44 (1H, H_e, br), 8.34 (1H, H_b, br), 7.94 (2H, H_a, s), 7.85 (2H, H₂, d, *J*=8.1 Hz), 7.80 (2H, H₁, d, *J*=8.1 Hz), 7.13 (2H, H₃, s), 4.03 (4H, H_f, m), 3.94 (2H, H_f, m), 3.46 (4H, H_{c+d}, br), 1.72 (6H, H_{m+h}, br), 1.51 (6H, H_g, m), 1.32–1.14 (18H, H_{j+k+l}, br), 0.92 (9H, H_i, d, *J*=6.6 Hz), 0.85 (18H, H_n, d, *J*=6.6 Hz); ¹³C NMR (DMSO-*d*₆, 75 MHz, 328 K) δ 166.7, 166.0, 152.2, 139.5, 135.7, 133.9, 129.5, 126.1, 105.6, 70.6, 66.5, 36.8, 36.8, 36.7, 35.9, 29.1, 29.0, 27.4, 24.2, 24.1, 22.5, 22.5, 22.4, 19.4, 19.3; FTIR (neat) 668, 718, 762, 858, 997, 1017, 1047, 1115, 1230, 1337, 1365, 1402, 1427, 1465, 1497, 1540, 1580, 1635, 2870, 2926, 2954, 3320 cm⁻¹. HRMS (MALDI-TOF) calcd. for C₄₆H₇₈BN₂O₇ [M+H]⁺, 781.590; found, 781.530.

(S,S,S)-N,N'-(((4,4'-(1-decyl-1H-phenanthro[1,10,9,8-cdefg]carbazole-3,10-diyl)bis(benzoyl))bis(azanediyl))bis(ethane-2,1-diyl))bis(3,4,5-tris(((S)-3,7-dimethyloctyl)-oxy)benzamide) (1)



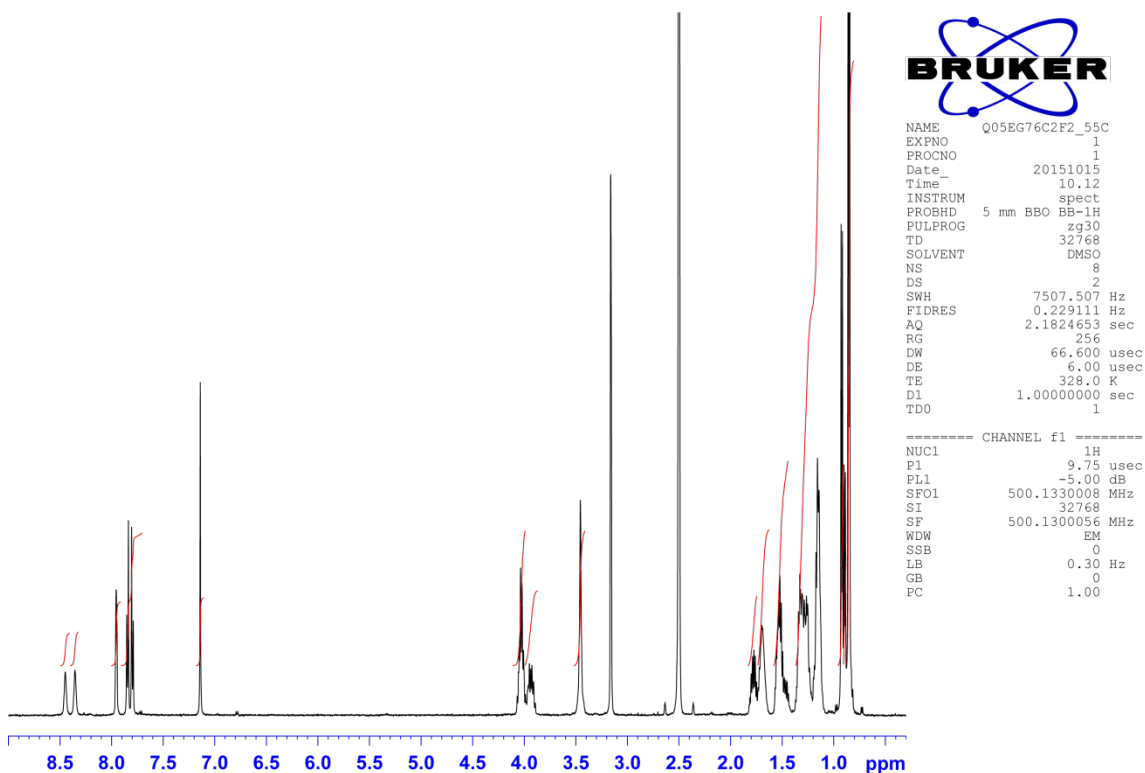
Compound **4** (111 mg, 0.20 mmol), compound **13** (340 mg, 0.44 mmol), tetrakis-(triphenylphosphine) palladium(0) (23 mg, 0.02 mmol) were dissolved in dry THF (42 mL). K_2CO_3 (137 mg, 0.99 mmol) was dissolved in water (2.3 mL) and added to the solution under Argon atmosphere. The reaction mixture was heated at reflux for 26 hours. After evaporation of the solvent under reduced pressure, the residue was washed with water, extracted with chloroform, dried over $MgSO_4$ and evaporation of the solvent under reduced pressure. After that, the residue was purified by column chromatography (silica gel, chloroform/methanol 10/0.05) affording compound **1** as a yellow solid (145 mg, 39 %). 1H NMR ($CDCl_3$, 300 MHz, 298 K) δ 8.62 (2H, H_1 , d, $J=7.7$ Hz), 8.03 (4H, H_{3+6} , m), 7.72 (6H, H_{2+5} , m), 7.62 (2H, H_4 , s), 7.52 (2H, H_a , br), 7.42 (2H, H_d , br), 7.12 (4H, H_7 , s), 4.43 (2H, H_n , t, $J=7.2$ Hz), 4.06 (12H, H_e , m), 3.81 (8H, H_{b+c} , br), 1.96 (2H, H_o , quin, $J=7.2$ Hz), 1.85 (6H, H_i , m), 1.63 (6H, H_g , br), 1.49 (14H, H_{f+p} , m), 1.30–1.11 (48H, $H_{i+j+k+q+r+s+t+u+v}$, br), 0.91 (18H, H_n , d, $J=6.6$ Hz), 0.85 (12H, H_m , d, $J=6.6$ Hz), 0.81 (27H, H_{m+w} , m); ^{13}C NMR ($CDCl_3$, 175 MHz, 313 K) δ 168.7, 168.7, 153.2, 145.6, 141.3, 136.4, 132.5, 131.9, 130.6, 130.5, 128.8, 127.3, 124.8, 123.9, 121.1, 117.1, 114.1, 105.8, 71.7, 67.7, 45.8, 41.5, 41.1, 39.4, 39.3, 37.5, 37.4, 37.4, 36.4, 35.4, 32.0, 31.8, 31.2, 29.9, 29.7, 29.7, 29.4, 29.3, 29.2, 29.2, 28.0, 27.9, 27.1, 24.7, 24.7, 22.7, 22.6, 22.6, 22.5, 19.6, 14.1, 14.0; FTIR (neat) 668, 718, 760, 802, 851, 1116, 1232, 1303, 1342, 1380, 1430, 1466, 1500, 1543, 1581, 1634, 2860, 2925, 2953, 3289 cm^{-1} . HRMS (MALDI-TOF, exact mass) calcd. for $C_{122}H_{179}N_5O_{10}$ [M], 1874.3652; found, 1874.3656.

(*R,R,R*)-N,N'-(((4,4'-(1-decyl-1H-phenanthro[1,10,9,8-cdefg]carbazole-3,10-diyl)bis(benzoyl))bis-(azanediyl))bis(ethane-2,1-diyl))bis(3,4,5-tris(((*R*)-3,7-dimethyloctyl)-oxy)benzamide) (2)

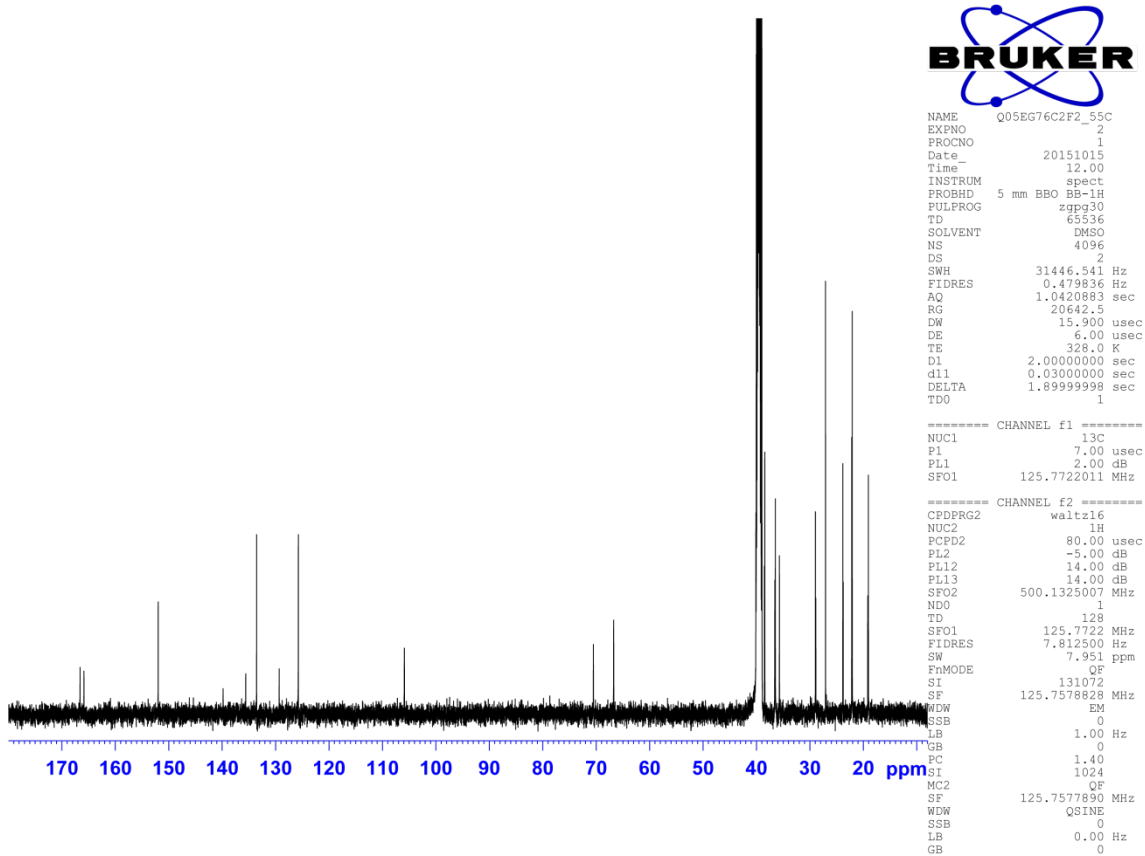


Compound **4** (115 mg, 0.20 mmol), compound **14** (350 mg, 0.45 mmol), tetrakis-(triphenylphosphine) palladium(0) (24 mg, 0.02 mmol) were dissolved in dry THF (43 mL). K_2CO_3 (141 mg, 1.02 mmol) was dissolved in water (2.4 mL) and added to the solution under Argon atmosphere. The reaction mixture was heated at reflux for 72 hours. After evaporation of the solvent under reduced pressure, the residue was washed with water, extracted with chloroform, dried over $MgSO_4$ and evaporation of the solvent under reduced pressure. After that, the residue was purified by column chromatography (silica gel, chloroform/methanol 10/0.05) affording compound **2** as a yellow solid (170 mg, 45 %). 1H NMR ($CDCl_3$, 700 MHz, 298 K) δ 8.63 (2H, H_1 , d, $J=7.7$ Hz), 8.05 (2H, H_3 , d, $J=8.2$ Hz), 8.02 (4H, H_6 , d, $J=7.7$ Hz), 7.74 (6H, H_{2+5} , m), 7.64 (2H, H_4 , s), 7.47 (2H, H_a , br), 7.37 (2H, H_d , br), 7.12 (4H, H_7 , s), 4.48 (2H, H_n , t, $J=7.2$ Hz), 4.04 (12H, H_e , m), 3.83 (4H, $H_{b\text{ or }c}$, br), 3.78 (4H, $H_{b\text{ or }c}$, br), 1.97 (2H, H_o , quin, $J=7.2$ Hz), 1.87 (4H, H_l , m), 1.82 (2H, H_r , m), 1.69 (6H, H_g , br), 1.59 (6H, H_{r+p} , br), 1.49 (8H, H_f , m), 1.31–1.10 (48H, $H_{i+j+k+q+r+s+t+u+v}$, br), 0.91 (18H, H_h , d, $J=6.5$ Hz), 0.85 (12H, H_m , d, $J=6.5$ Hz), 0.82 (24H, H_m , d, $J=6.5$ Hz), 0.80 (3H, H_w , t, $J=7.2$ Hz); ^{13}C NMR ($CDCl_3$, 175 MHz, 298 K) δ 168.9, 168.8, 153.3, 145.7, 141.2, 136.5, 132.6, 131.9, 130.7, 128.9, 127.4, 125.0, 124.9, 124.0, 121.3, 117.2, 114.2, 105.6, 71.8, 67.6, 45.8, 41.5, 41.1, 39.5, 39.4, 37.7, 37.5, 37.5, 36.5, 31.9, 31.3, 30.0, 29.8, 29.6, 29.4, 28.1, 28.1, 27.3, 24.9, 22.9, 22.8, 22.8, 22.7, 19.7, 14.2; FTIR (neat) 736, 802, 851, 1060, 1117, 1232, 1303, 1343, 1382, 1429, 1465, 1501, 1543, 1581, 1634, 2854, 2925, 2955, 3291 cm^{-1} . HRMS (MALDI-TOF, exact mass) calcd. for $C_{122}H_{179}N_5O_{10}$ [M], 1874.3652; found, 1874.3680.

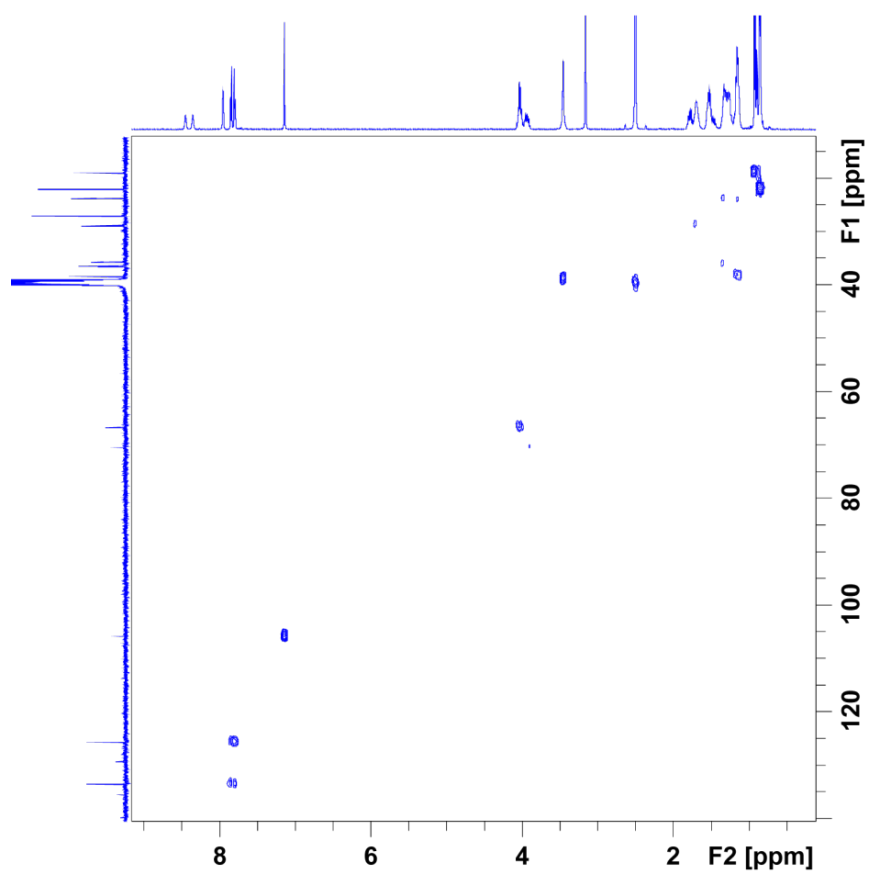
3. Collection of spectra



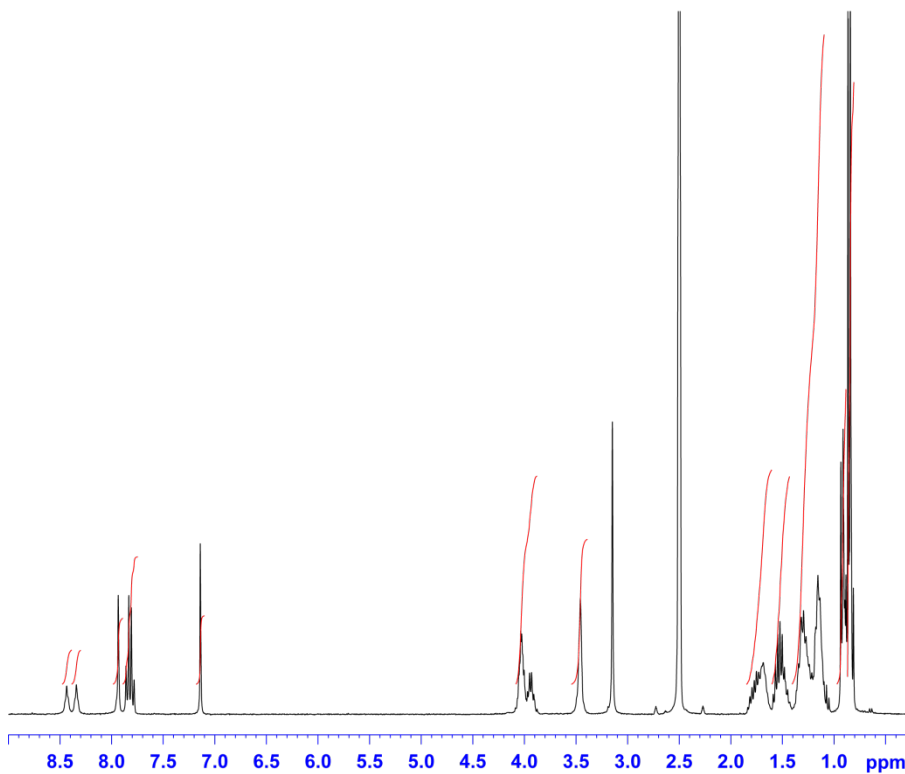
¹H NMR (DMSO-*d*₆, 500 MHz, 328 K) of compound **13**.



¹³C NMR (DMSO-*d*₆, 125 MHz, 328 K) of compound **13**.



$^1\text{H}, ^{13}\text{C}$ -HMQC spectrum (DMSO- d_6 , 328 K) of compound 13.



```

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PROCNO    1
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Time_     14.47
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PULPROG   zg30
TD         32768
SOLVENT   DMSO
NS         16
DS         2
SWH        4496.403 Hz
FIDRES     0.137219 Hz
AQ         3.6438515 sec
RG         322.5
DW         111.200 usec
DE         6.50 usec
TE         328.2 K
D1         1.0000000 sec
D11        1
TDO        1

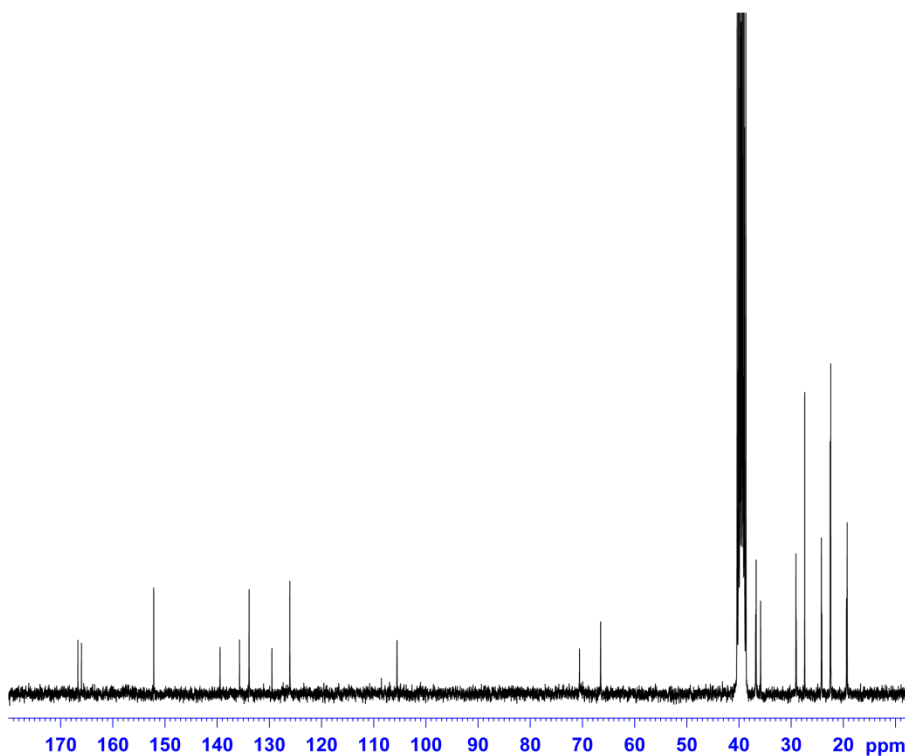
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PL1        -6.00 dB
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SI         32768
SF         300.1300012 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

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¹H NMR (DMSO-*d*₆, 300 MHz, 328 K) of compound 14.

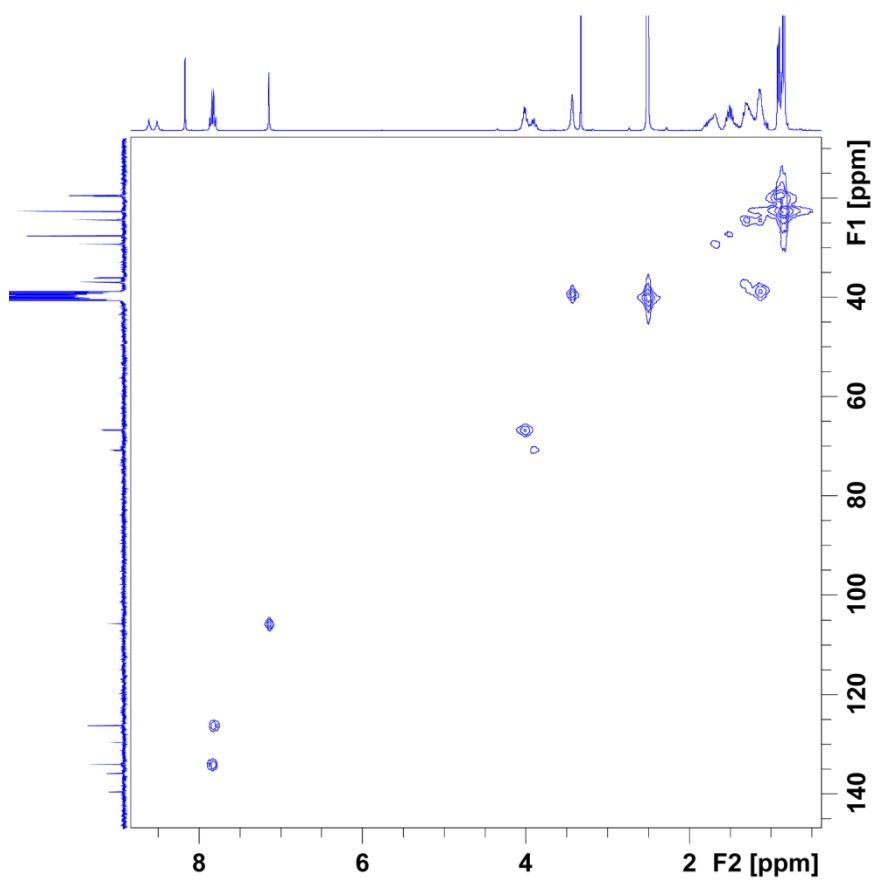


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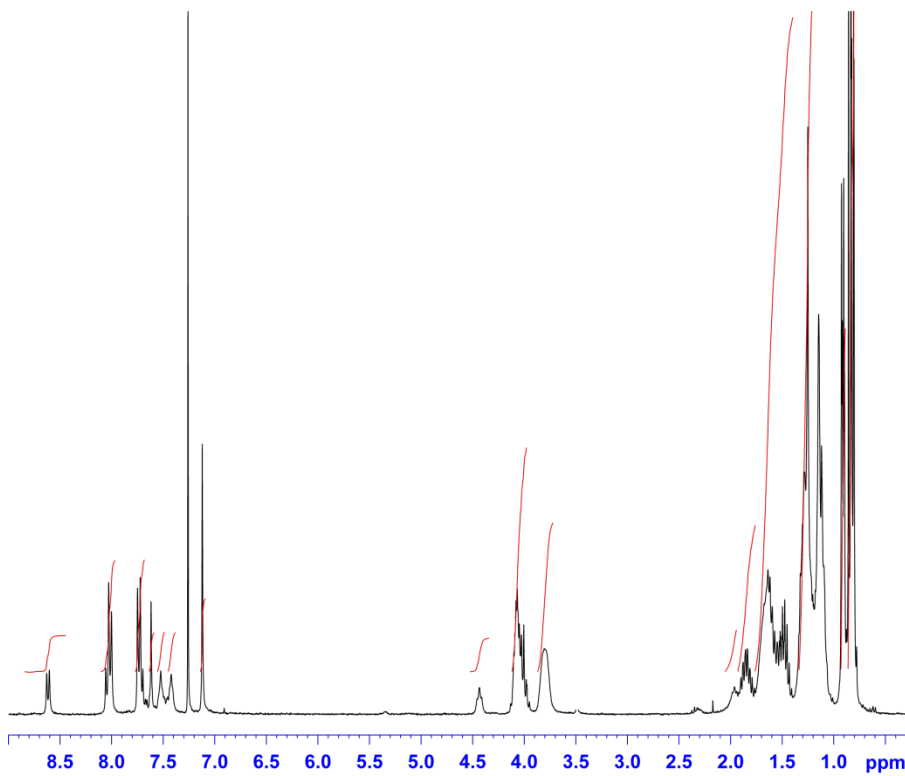
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TD         65536
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NS         10000
DS         4
SWH        18115.941 Hz
FIDRES     0.552855 Hz
AQ         1.8088436 sec
RG         13.95
DW         27.600 usec
DE         6.50 usec
TE         298.0 K
D1         2.0000000 sec
D11        0.0300000 sec
D10        1
SFO1      75.4835188 MHz
NUC1      13C
P1         10.00 usec
SI         32768
SF         75.4753279 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

```

¹³C NMR (DMSO-*d*₆, 75 MHz, 328 K) of compound 14.



$^1\text{H},^{13}\text{C}$ -HMQC spectrum (DMSO- d_6 , 328 K) of compound 14.



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NAME      Q05EG79F2CN
EXPNO     160
PROCNO    1
Date_     20151125
Time_     14.51
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
FULPROG   zg30
TD        16384
SOLVENT   CDCl3
NS        16
DS        2
SWH       4789.272 Hz
FIDRES    0.292314 Hz
AQ        1.7105396 sec
RG        456.1
DW        104.400 usec
DE        6.00 usec
TE        300.0 K
D1        1.0000000 sec
TDO       1

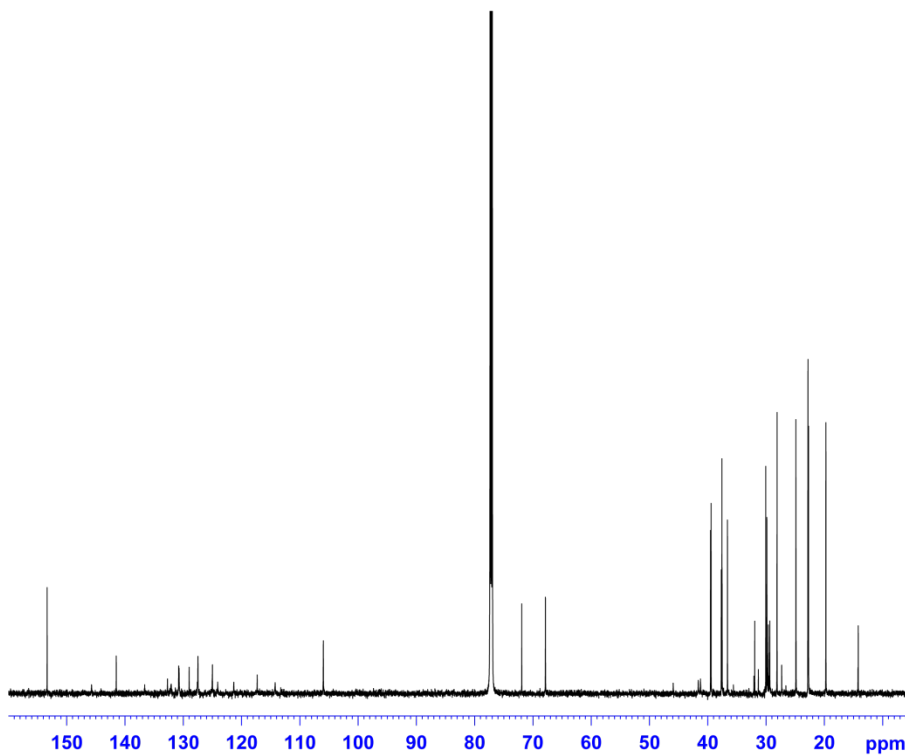
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===== CHANNEL f1 =====
NUC1      1H
P1        10.50 usec
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SI        32768
SF        300.1600114 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

```

¹H NMR (CDCl₃, 300 MHz, 298 K) of compound 1.



```

NAME      Q05EG78CARBONO_40C
EXPNO     2
PROCNO    1
Date_     20160112
Time_     13.35
INSTRUM   spect
PROBHD    5 mm CPTCI 1H-
FULPROG   zgpg30
TD        65356
SOLVENT   CDCl3
NS        800
DS        4
SWH       48076.922 Hz
FIDRES    0.735616 Hz
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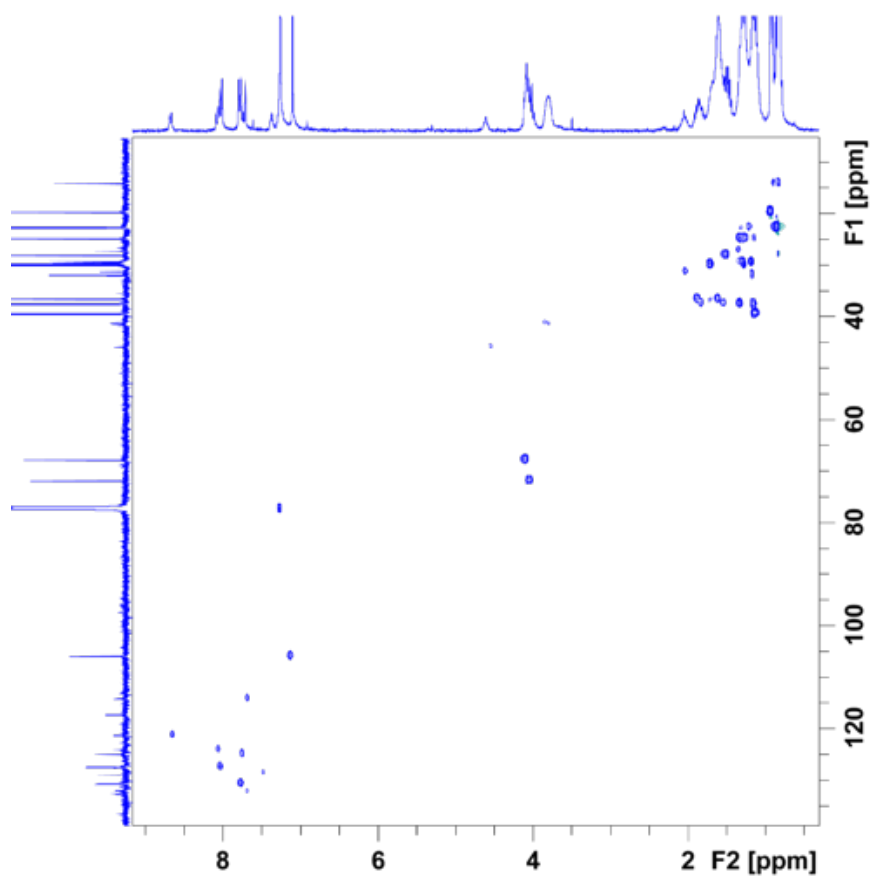
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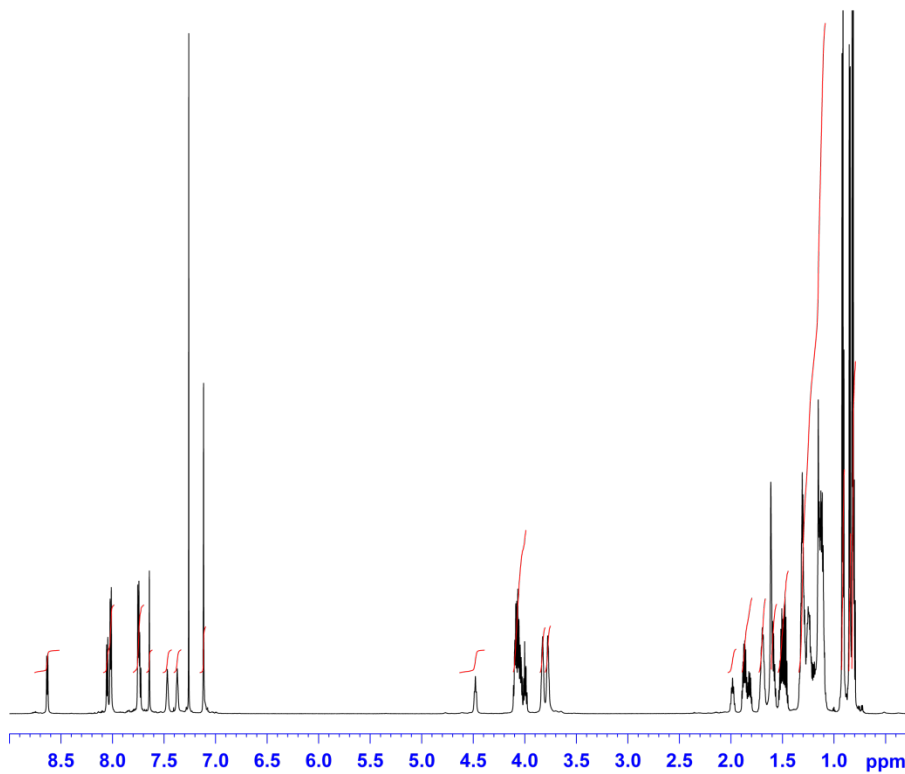
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LB        1.00 Hz
GB        0
PC        1.40

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¹³C NMR (CDCl₃, 175 MHz, 313 K) of compound 1.



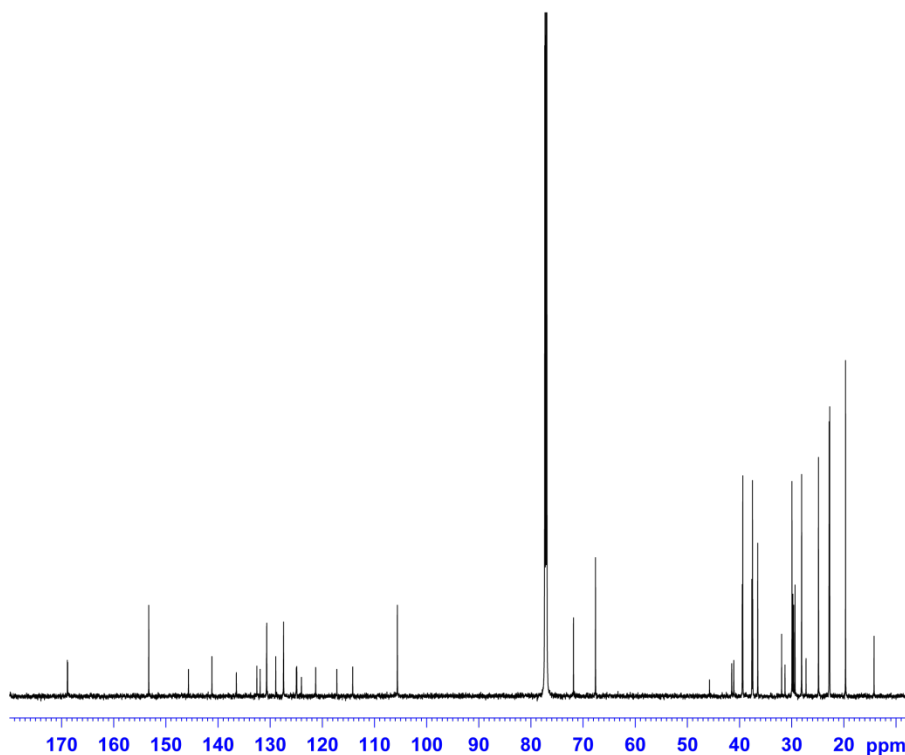
$^1\text{H}, ^{13}\text{C}$ -HMQC spectrum (CDCl_3 , 313 K) of compound **1**.



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PULPROG       zg30
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DS            2
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FIDRES        0.641126 Hz
AQ            1.5598068 sec
RG            5.6
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DE            6.50 usec
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TD0           1
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SSB           0
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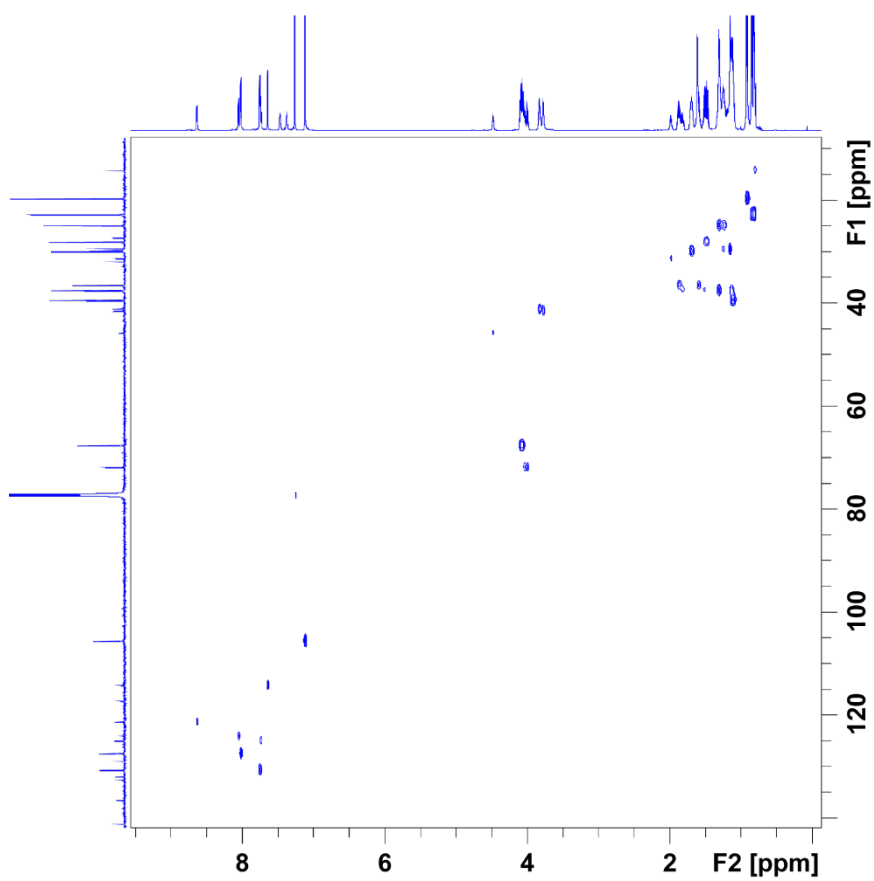
¹H NMR (CDCl₃, 700 MHz, 298 K) of compound 2.



```

NAME          Q05EG124
EXPNO         2
PROCNO        1
Date_         20180215
Time_         10.59 h
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PROBHD        Z75813_0001 (C
PULPROG       zgpg30
TD            65356
SOLVENT       CDCl3
NS            1024
DS            4
SWH           48076.922 Hz
FIDRES        1.471232 Hz
AQ            0.6797524 sec
RG            1030
DW            10.400 usec
DE            18.00 usec
TE            298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
SF01          176.0772534 MHz
NUC1          13C
P1            13.75 usec
SI            32768
SF            176.0578634 MHz
WDW           EM
SSB           0
LB            2.00 Hz
GB            0
PC            1.40
  
```

¹³C NMR (CDCl₃, 175 MHz, 298 K) of compound 2.



$^1\text{H},^{13}\text{C}$ -HMOC spectrum (CDCl_3 , 298 K) of compound 2.

4. Supplementary Figures and Tables

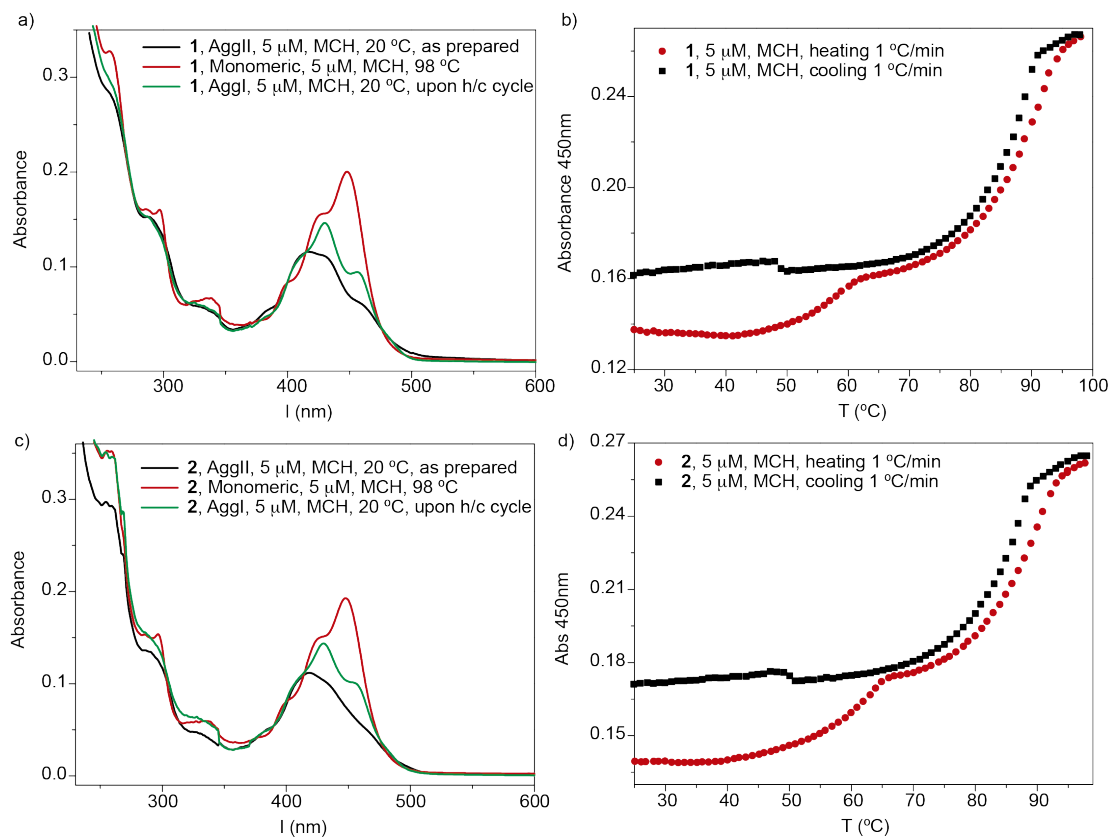


Figure S1. UV-Vis spectra of **1** (a) and **2** (c) in MCH, at $c_T = 5 \mu\text{M}$, at different temperatures and as-prepared or upon applying a heating/cooling (h/c) cycle. Heating and cooling curves of **1** (b) and **2** (d) in MCH, at $c_T = 10 \mu\text{M}$ utilizing a heating and a cooling rate of 1 $^\circ\text{C}/\text{min}$.

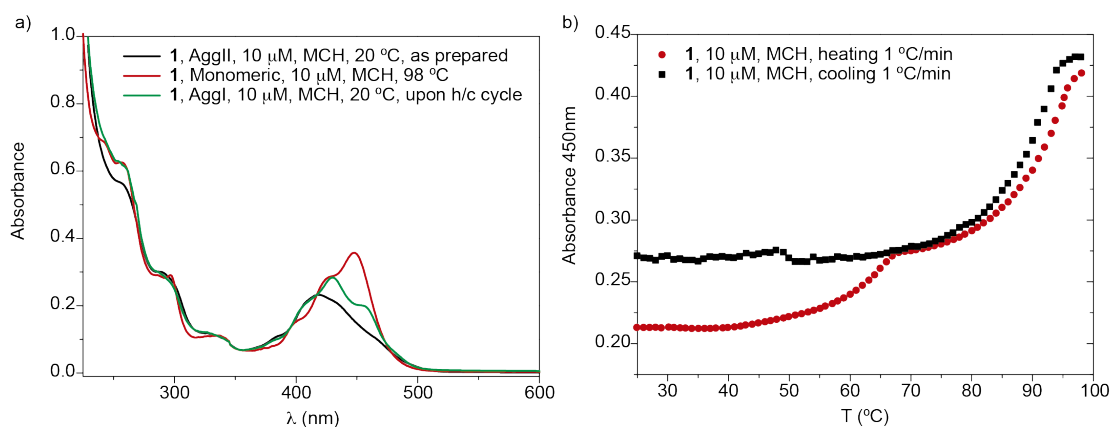


Figure S2. (a) UV-Vis spectra of **1** in MCH, at $c_T = 10 \mu\text{M}$, at different temperatures and as-prepared or upon applying a heating/cooling (h/c) cycle. (b) Heating and cooling curves of **1** in MCH at $c_T = 10 \mu\text{M}$ utilizing a heating and a cooling rate of 1 $^\circ\text{C}/\text{min}$.

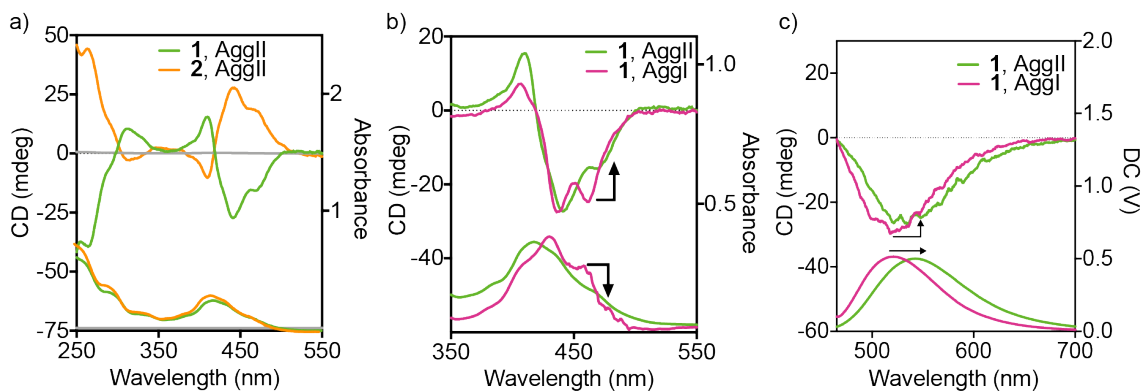


Figure S3. (a) UV-Vis (bottom) and CD (top) spectra of the AggII species formed from **1** and **2**. Comparison of the UV-Vis (b, bottom), CD (b, top) fluorescence (c, bottom) and CPL (c, top) spectra of Aggl and AggII formed from the self-assembly of **1**. Experimental conditions: MCH, $c_T = 20 \mu\text{M}$, $\lambda_{\text{exc}} = 380 \text{ nm}$, $20 \text{ }^\circ\text{C}$.

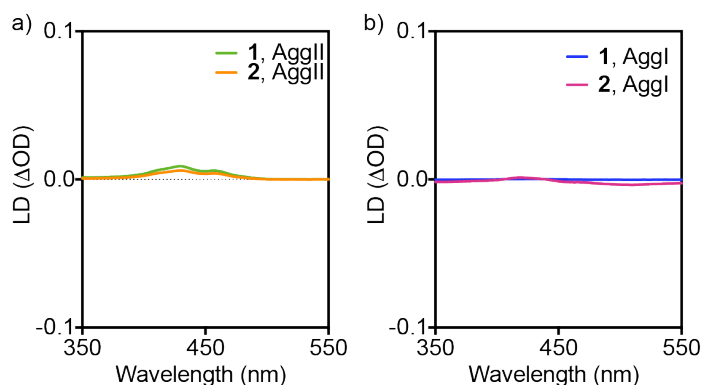


Figure S4. LD spectra of chiral **1** and **2** for both the AggII (a) and Aggl (b) species obtained in MCH and Tol, respectively. Experimental conditions: $c_T = 20 \mu\text{M}$, $20 \text{ }^\circ\text{C}$.

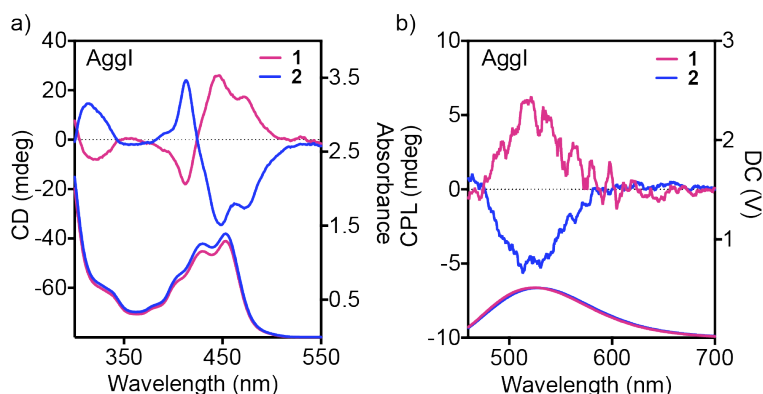


Figure S5. (a) UV-Vis (a,bottom), CD (b, top), fluorescence (b, bottom) and CPL (b, top) spectra of the Aggl species formed from **1** and **2** in Tol. Experimental conditions: $c_T = 20 \mu\text{M}$, $\lambda_{\text{exc}} = 380 \text{ nm}$, $20 \text{ }^\circ\text{C}$.

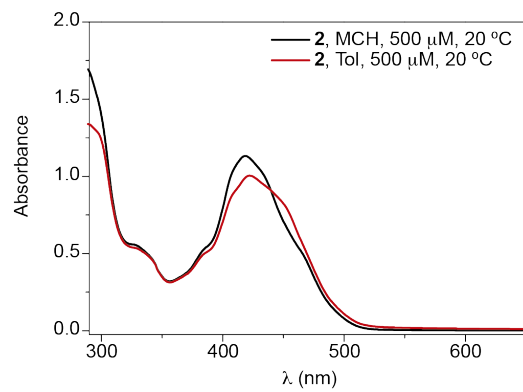


Figure S6. UV-Vis spectra of **2** at $c_T = 500 \mu\text{M}$ in MCH and Tol, 20 °C.

References

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- (S2) S. Ghosh, X-Q. Li, V. Stepanenko and F. Würthner, *Chem. Eur. J.* 2008, **14**, 11343.
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