

# Supporting Information

## Facile Synthesis of an Eight-armed Star-shaped Polymer via Coordination-driven Self-assembly of a Four-armed Cavitand

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## References

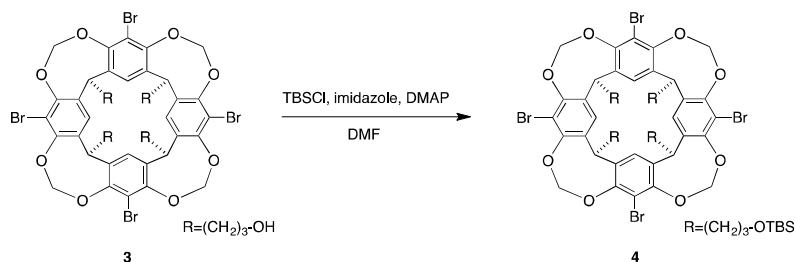
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**General:** All reagents and solvents were of the commercial reagent grade and were used without further purification except where noted. DMF was obtained by distillation over  $\text{CaH}_2$ .  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a Varian mercury-300 or JEOL ECA-500 spectrometer at 25 °C in  $\text{CDCl}_3$  and chemical shifts were reported as the delta scale in ppm relative to  $\text{CHCl}_3$  ( $\delta = 7.260$  for  $^1\text{H}$  and 77.3 for  $^{13}\text{C}$ ). ESI-Mass spectra were recorded on Thermo Scientific LTQ Orbitrap XL hybrid FTMS. Preparative separations were performed by silica gel gravity column chromatography (Silica Gel 60N (spherical, neutral)).

**DOSY.** *Poly-1* and *poly-2* were dissolved in chloroform- $d_1$ , and the sample solutions were placed in a 3 mm NMR sample tube. The pulse-field gradient diffusion NMR spectra were collected using a bipolar pulse pair stimulated echo (BPP-STE) pulse sequence on a JEOL Delta 500 spectrometer with a 3 mm inverse H3X/FG probe. The pulse-field gradient strength was arrayed from  $\sim 0.003$  to  $0.803 \text{ T m}^{-1}$  with a pulse gradient time of 1 ms and a diffusion time of 100 ms. The data were processed using the MestReNova program. The signal intensity as a function of the pulse-field gradient strength was fitted to the Stejskal-Tanner equation to determine the diffusion coefficients.

**Dynamic Light Scattering (DLS).** DLS measurements were carried out by using a Zetasizer Nano ZS (Malvern Instruments) setup equip with a He-Ne laser (633 nm, 40 mW power), and the scattering angle was  $173^\circ$  in  $10 \times 10 \text{ mm}$  quartz cuvette. *Poly-1* and *poly-2* were dissolved in chloroform, and the sample solutions were made at a concentration of  $1.0 \text{ mmol L}^{-1}$  and  $0.5 \text{ mmol L}^{-1}$  respectively. Each sample was measured at 20°C, and the particle size (hydrodynamic diameter) was volume-weighted averaged using Dispersion Technology Software (DTS) (V4.20).

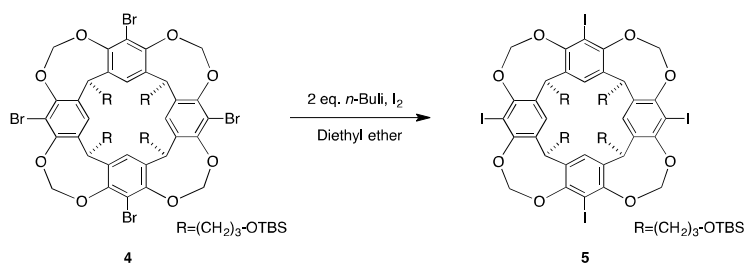
## Synthesis of 4



To a solution of **3** (4.01 g, 3.69 mmol) in dry *N,N*-dimethylformamide (35 mL) was added dimethylaminopyridine (3.60 g, 29.5 mmol) and imidazole (2.02 g, 29.5 mmol) at room temperature. The mixture was cooled to 0 °C and *tert*-butylchlorodimethylsilane (4.45 g, 29.5 mmol) was added to the mixture. The solution was allowed to room temperature and was stirred for 2 h. The solvent was removed under reduced pressure. The organic residue was dissolved in ethyl acetate and the solution was washed with 1N aqueous hydrogen chloride, aqueous sodium hydrogen carbonate, and brine. The organic layer was dried over anhydrous sodium sulfonate. The residual solid was purified by column chromatography on silica gel (ethyl acetate : hexane = 1 : 99) to afford **4**. Recrystallization of **4** from a mixed solution of toluene and hexane at room temperature afforded pure product (3.68 g, 1.17 mmol) in 65% yield.

M.p. 162–163 °C;  $^1\text{H}$  NMR (300 MHz, chloroform- $d_1$ ):  $\delta$  7.04 (s, 4H), 5.96 (d, 4H,  $J = 7.3$  Hz), 4.88 (t, 4H,  $J = 8.2$  Hz), 4.39 (d, 4H,  $J = 7.3$  Hz), 3.70 (t, 8H,  $J = 6.3$  Hz), 2.26 (m, 8H), 1.58 (m, 8H), 0.94 (s, 36H), 0.08 (s, 24H);  $^{13}\text{C}$  NMR (75 MHz, chloroform- $d_1$ ):  $\delta$  152.3, 139.2, 119.1, 113.8, 98.6, 62.5, 37.2, 30.6, 26.2, 26.1, 18.6, –5.1; HRMS (ESI-Orbitrap)  $m/z$ :  $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{68}\text{H}_{101}\text{O}_{12}\text{Br}_4\text{Si}_4$  1537.3098, found 1537.3138.

## Synthesis of 5

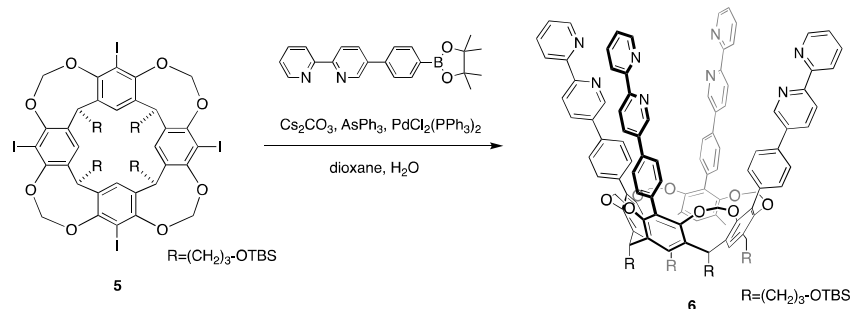


Under  $\text{N}_2$  atmosphere to a solution of **4** (2.0 g, 1.3 mmol) in dry diethyl ether (170 mL) was added *n*-butyllithium in hexane (6.4 mL, 10.4 mmol) at –80 °C. Iodine (ca. 3 g) in dry diethyl ether (30 mL) was slowly added to the solution. The reaction was quenched with aqueous sodium thiosulfate. The mixture was warmed to room temperature. The solvent was removed under reduced pressure. The organic residue was extracted with ethyl acetate. The organic layer was washed with aqueous sodium thiosulfate, aqueous sodium hydrogen carbonate, and then brine, and dried over anhydrous sodium sulfate. The solvent was removed under reduced pressure. The residual solid was purified by column chromatography on silica gel (ethyl acetate : hexane = 1 : 99) to afford **5** (1.75 g, 1.01 mmol) in 77% yield.

M.p. 172–173 °C;  $^1\text{H}$  NMR (300 MHz, chloroform- $d_1$ ):  $\delta$  7.07 (s, 4H), 5.97 (d, 4H,  $J = 7.4$  Hz), 4.87 (t, 4H,  $J = 8.2$  Hz), 4.31 (d, 4H,  $J = 7.4$  Hz), 3.69 (t, 8H,  $J = 6.3$  Hz), 2.25 (m, 8H), 1.56 (m, 8H), 0.93 (s, 36H), 0.08 (s, 24H);  $^{13}\text{C}$  NMR (75

MHz, chloroform-*d*<sub>1</sub>):  $\delta$  155.1, 138.6, 120.7, 98.8, 93.3, 62.5, 37.5, 30.7, 26.3, 26.2, 18.6, -5.1; HRMS (ESI-Orbitrap) *m/z*: [M + H]<sup>+</sup> calcd for C<sub>68</sub>H<sub>101</sub>O<sub>12</sub>I<sub>4</sub>Si<sub>4</sub> 1729.2543, found 1729.2572.

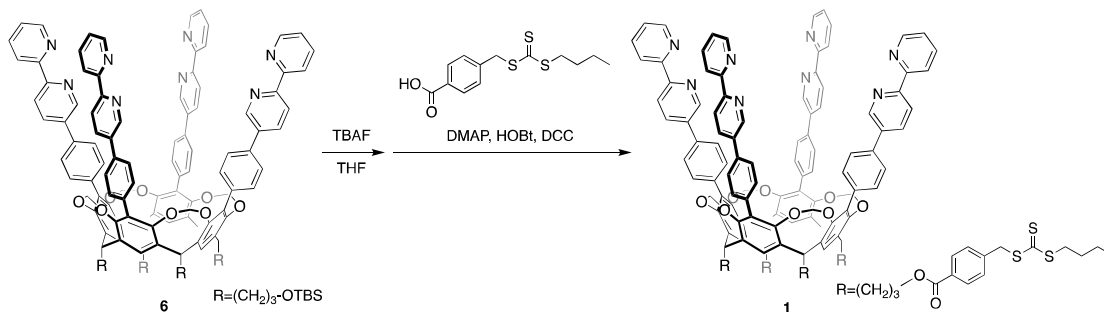
## Synthesis of 6



To a solution of **5** (450 mg, 0.260 mmol) in a degassed mixed solution of 1,4-dioxane and water (10 mL, 1,4-dioxane : water = 25 : 1), cesium carbonate (2.50 g, 7.80 mmol), triphenylarsine (320 mg, 1.04 mmol), 5-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-2,2'-bipyridine (0.76 g, 2.1 mmol), and bis(triphenylphosphine)palladium dichloride (53 mg, 0.052 mmol). The mixture was stirred at 110 °C of 15 min under Ar atmosphere. The solution was cooled to room temperature and filtered with magnesium silicate. Filtrate was concentrated under reduced pressure. The organic residue was purified by gel permeation chromatography (eluent: chloroform) to afford **6** (451 mg, 0.210 mmol) in 81% yield.

M.p. > 300 °C; <sup>1</sup>H NMR (300 MHz, chloroform-*d*<sub>1</sub>):  $\delta$  8.92 (d, 4H, *J* = 2.3 Hz), 8.66 (d, 4H, *J* = 4.5 Hz), 8.44 (d, 4H, *J* = 8.2 Hz), 8.42 (d, 4H, *J* = 7.8 Hz), 8.02 (dd, 4H, *J* = 8.2, 2.3 Hz), 7.82 (td, 4H, *J* = 7.8, 1.8 Hz), 7.64 (d, 8H, *J* = 8.0 Hz), 7.41 (s, 4H), 7.29 (m, 4H), 7.21 (d, 8H, *J* = 8.0 Hz), 5.41 (d, 4H, *J* = 7.0 Hz), 4.95 (t, 4H, *J* = 8.0 Hz), 4.39 (d, 4H, *J* = 7.0 Hz), 3.80 (t, 8H, *J* = 6.4 Hz), 2.45 (m, 8H), 1.72 (m, 8H), 0.99 (s, 36H), 0.13 (s, 24H); <sup>13</sup>C NMR (75 MHz, chloroform-*d*<sub>1</sub>):  $\delta$  155.8, 155.0, 152.7, 149.2, 147.5, 138.3, 137.0, 136.3, 135.8, 135.1, 133.8, 130.8, 126.5, 123.7, 121.1, 121.0, 100.6, 62.8, 36.6, 30.9, 26.6, 26.1, 18.5, -5.1; HRMS (ESI-Orbitrap) *m/z*: [M + H]<sup>+</sup> calcd for C<sub>132</sub>H<sub>146</sub>N<sub>8</sub>O<sub>12</sub>Si<sub>4</sub> 1073.5063 [M+2H]<sup>2+</sup>, found 1073.5088.

## Synthesis of 1

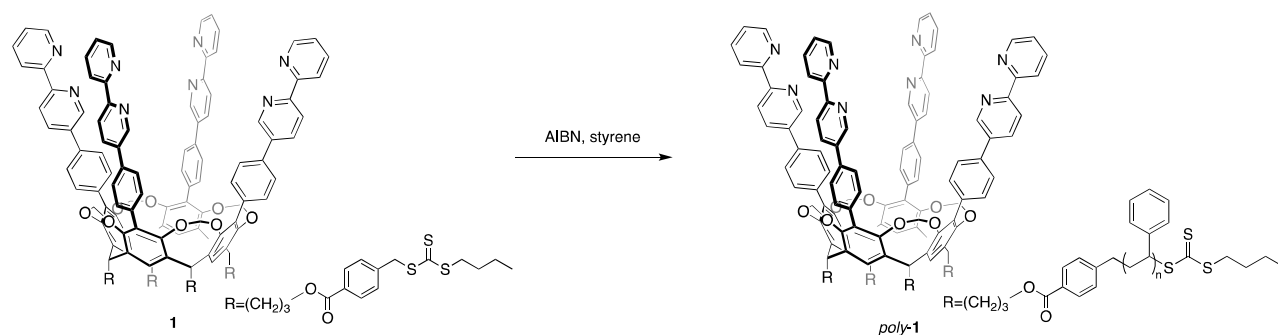


To a solution of **6** (415 mg, 193 μmol) in dry THF (4 mL) was added tetrabutylammonium fluoride (977 mg, 3.10 mmol) at room temperature. The solution was stirred for 5 h. The reaction was quenched with water. Precipitate was

filtered off and dissolved in chloroform. Addition of hexane into the solution gave precipitate of deprotected cavitand (297 mg, 176  $\mu$ mol). This precipitate was used in the next reaction without further purification. To a solution of the precipitate (172 mg, 0.101 mmol) in freshly distilled *N,N*-dimethylformamide (1.2 mL) was dimethylaminopyridine (123 mg, 1.00 mmol), 1-hydroxybenzotriazole (87 mg, 0.64 mmol), 4-((((butylthio)carbon-thioyl)thio)methyl)benzoic acid (181 mg, 0.602 mmol), and *N,N'*-dicyclohexylcarbodiimide (128 mg, 0.620 mmol) at room temperature. The mixture was stirred at 50 °C for 24 under Ar atmosphere. The reaction was quenched with ethyl acetate. The solution was filtered and the filtrate was washed with aqueous hydrogen chloride (0.1 N), aqueous sodium hydrogen carbonate, and brine. The organic layer was dried over anhydrous sodium sulfate. The solvent was removed under reduced pressure. The organic residue was purified with gel permeation chromatography (eluent: chloroform) to afford **1** (176 mg, 80  $\mu$ mol) in 80 % yield.

M.p. > 300 °C;  $^1\text{H}$  NMR (300 MHz, chloroform- $d_1$ ):  $\delta$  8.92 (d, 4H,  $J$  = 1.8 Hz), 8.64 (d, 4H,  $J$  = 4.0 Hz), 8.46 (d, 4H,  $J$  = 8.8 Hz), 8.44 (d, 4H,  $J$  = 7.8 Hz), 8.02 (m, 4H), 8.01 (d, 8H,  $J$  = 8.4 Hz), 7.83 (dd, 4H,  $J$  = 1.5, 7.8 Hz), 7.64 (d, 8H,  $J$  = 8.2 Hz), 7.45 (s, 4H), 7.39 (d, 8H,  $J$  = 8.4 Hz), 7.28 (m, 4H), 7.20 (d, 8H,  $J$  = 8.2 Hz), 5.41 (d, 4H,  $J$  = 6.8 Hz), 5.05 (t, 4H,  $J$  = 8.0 Hz), 4.62 (s, 8H), 4.51 (t, 8H,  $J$  = 6.6 Hz), 4.40 (d, 4H,  $J$  = 6.8 Hz), 3.36 (t, 8H,  $J$  = 7.4 Hz), 2.59 (m, 8H), 1.97 (m, 8H), 1.67 (quint, 8H,  $J$  = 7.4 Hz), 1.41 (sext, 8H,  $J$  = 7.4 Hz), 0.92 (t, 12H,  $J$  = 7.4 Hz);  $^{13}\text{C}$  NMR (75 MHz, chloroform- $d_1$ ):  $\delta$  232.9, 223.1, 166.3, 155.8, 155.1, 153.1, 149.2, 147.6, 140.9, 138.3, 137.1, 136.5, 135.9, 135.2, 133.6, 130.9, 130.1, 129.7, 129.4, 126.7, 123.8, 121.2, 119.8, 100.7, 77.4, 75.0, 65.0, 40.8, 37.1, 30.1, 27.5, 27.3, 22.2, 13.7; HRMS (ESI-Orbitrap)  $m/z$ :  $[\text{M} + \text{H}]^{2+}$  calcd for  $\text{C}_{160}\text{H}_{146}\text{O}_{16}\text{N}_8\text{S}_{12}$  1409.3747, found 1409.3744.

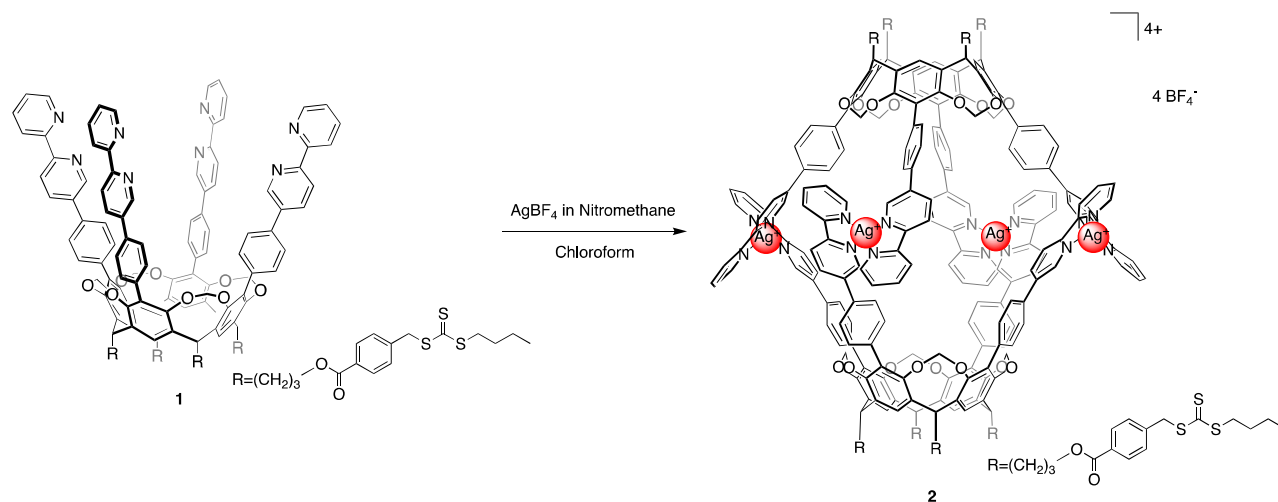
## RAFT polymerization of **1**



A mixture of **1** (100 mg, 35.4  $\mu\text{mol}$ ), azobisisobutyronitrile (2.9 mg, 17.7  $\mu\text{mol}$ ), styrene (743 mg, 7.09 mmol) was degassed by freeze drying. The mixture was stirred at 90  $^{\circ}\text{C}$  for 24 h under Ar atmosphere. Purification of the product was achieved by gel permeation chromatography (eluent: chloroform) to afford *poly-1* (666 mg) in 75 % yield.

M.p. 174–175  $^{\circ}\text{C}$ ;  $^1\text{H}$  NMR (300 MHz, chloroform- $d_1$ ):  $\delta$  8.93 (s, 4H), 8.70 (d, 4H,  $J = 4.2$  Hz), 8.47 (m, 8H), 8.02 (d, 4H,  $J = 8.5$  Hz), 7.85 (br, 12H), 7.64 (d, 8H,  $J = 7.9$  Hz), 7.42 (s, 4H), 6.33–7.25 (br, 440H), 5.42 (br, 4H), 5.03 (br, 8H), 4.47 (br, 12H), 3.26 (m, 8H), 2.55 (br, 8H), 1.20–2.35 (br, 300H), 0.92 (m, 12 H);  $M_{n,\text{SEC}} = 10,100 \text{ g mol}^{-1}$ ,  $M_{n,\text{NMR}} = 10,200 \text{ g mol}^{-1}$ .

## Synthesis of capsule-1 $\cdot(\text{BF}_4)_4$

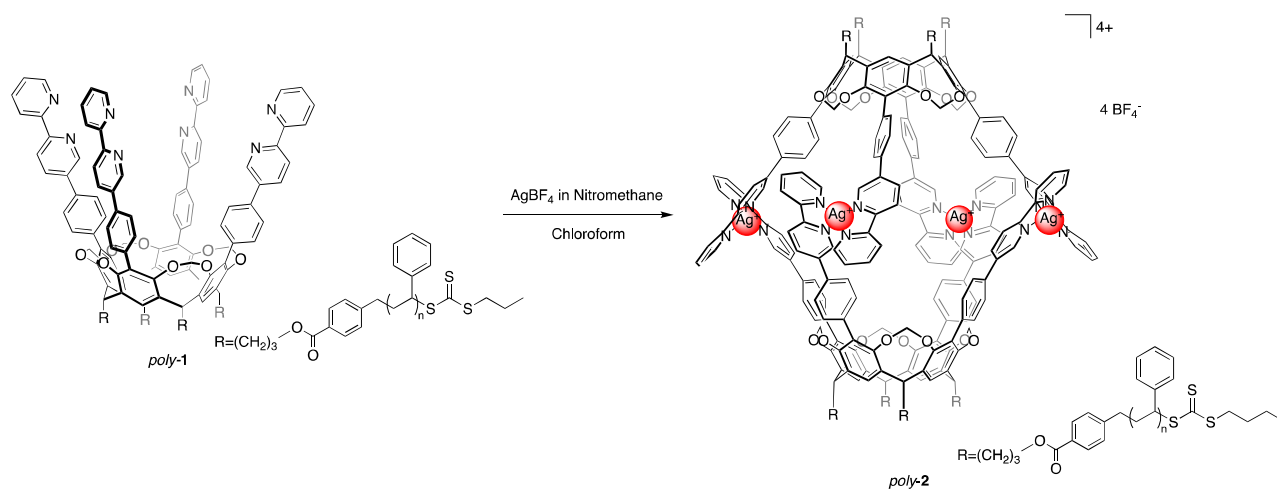


To a solution of **1** (6.4 mg, 2.3  $\mu\text{mol}$ ) in chloroform was added silver tetrafluoroborate (0.8 mg, 4.1  $\mu\text{mol}$ ) in nitromethane (113  $\mu\text{L}$ ). The mixture was stirred for 1 h at room temperature. The solvent was removed under reduced pressure to afford **2** (7.2 mg, 1.1  $\mu\text{mol}$ ) in 99% yield.

M.p. > 300  $^{\circ}\text{C}$ ;  $^1\text{H}$  NMR (300 MHz, chloroform- $d_1$ ):  $\delta$  8.85–8.90 (m, 16H), 8.49–8.59 (m, 16H), 8.25 (d, 8H,  $J = 8.2$  Hz), 8.17 (t, 8H,  $J = 7.6$  Hz), 7.96 (d, 16H,  $J = 8.1$  Hz), 7.30–7.48 (m, 48H), 7.05 (d, 16H,  $J = 7.7$  Hz), 5.30 (d, 8H,  $J = 6.3$  Hz), 4.94 (t, 8H,  $J = 7.4$  Hz), 4.58 (s, 16H), 4.46 (t, 16H,  $J = 6.0$  Hz), 4.24 (d, 8H,  $J = 6.3$  Hz), 3.34 (t, 16H,  $J = 7.4$  Hz), 2.53 (br, 16H), 1.91 (br, 16H), 1.65 (quint, 16H,  $J = 7.4$  Hz), 1.40 (sext, 16H,  $J = 7.4$  Hz), 0.91 (t, 24H,  $J = 7.4$  Hz);  $^{13}\text{C}$  NMR (75 MHz, chloroform- $d_1$ ):  $\delta$  223.2, 166.3, 153.0, 152.7, 152.1, 150.6, 150.5, 149.0, 140.9, 139.9, 138.4, 138.2,

135.7, 133.7, 131.2, 130.1, 129.7, 129.4, 128.6, 127.0, 125.5, 124.2, 120.0, 100.8, 77.4, 65.0, 40.8, 37.1, 30.1, 29.8, 27.5, 27.1, 22.2, 13.7.

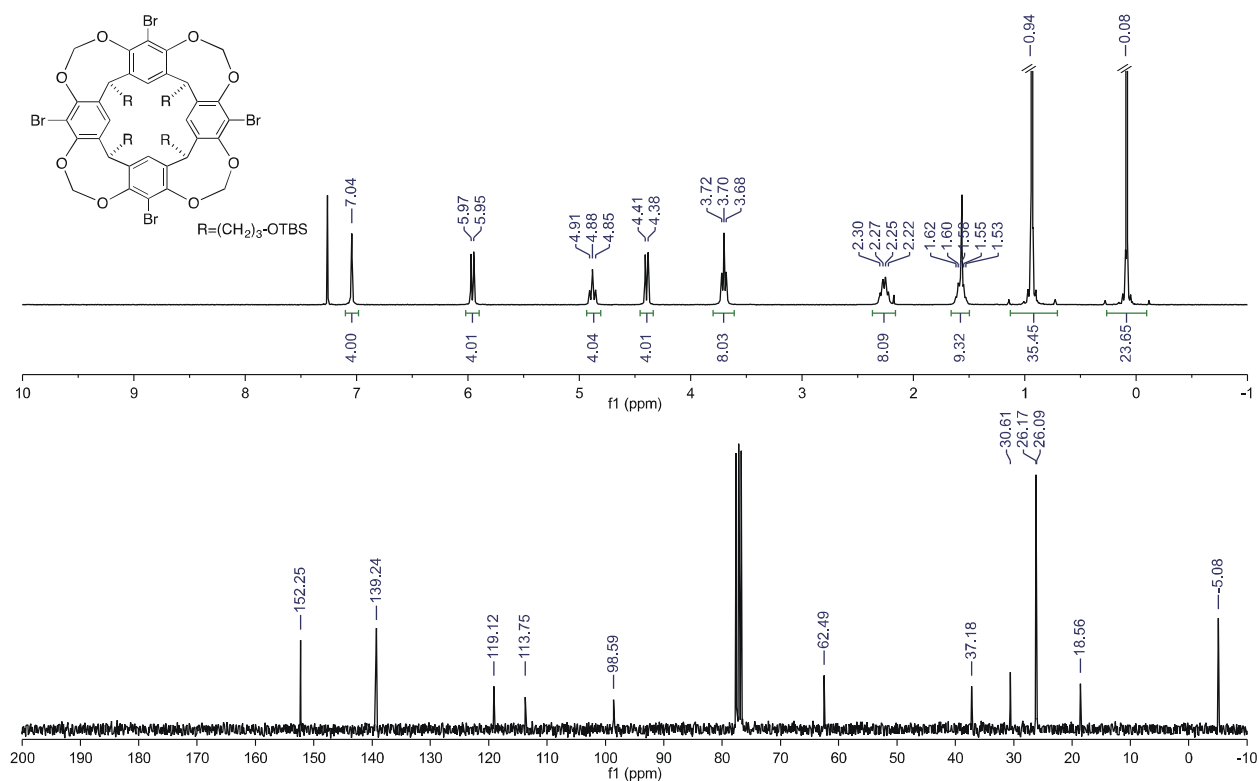
### Synthesis of *poly-capsule-1*·(BF<sub>4</sub>)<sub>4</sub>



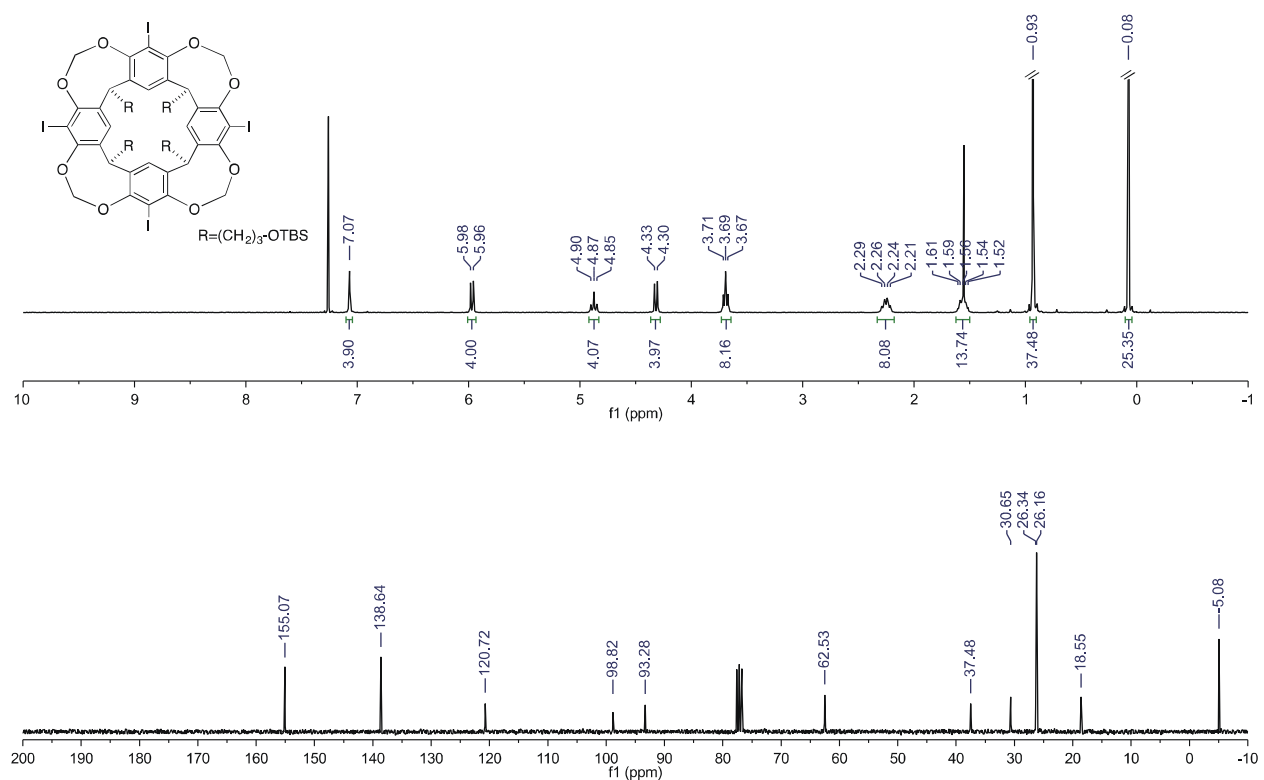
To a solution of *poly-1* (1.04, (calc. sec 0.0595) mmol) in chloroform was added to silver tetrafluoroborate (23.2 mg, 0.112 mol) in nitromethane (422  $\mu$ L). The mixture was stirred for 0.5 h at room temperature. The solvent was removed under reduced pressure to afford *poly-2* (1.06 mg, 0.0295 mmol) in 99% yield.

M.p. 260–263 °C; <sup>1</sup>H NMR (300 MHz, chloroform-*d*<sub>1</sub>):  $\delta$  8.82 (br, 16H), 8.49 (br, 16H), 8.06–8.30 (br, 16H), 7.81 (br, 16H), 6.16–7.22 (br, 2100H), 5.28 (br, 8H), 4.91 (br, 16H), 4.42 (br, 16H), 4.24 (br, 8H), 3.27 (br, 16H), 1.17–2.61 (br, 2200H), 0.90 (br, 24H).

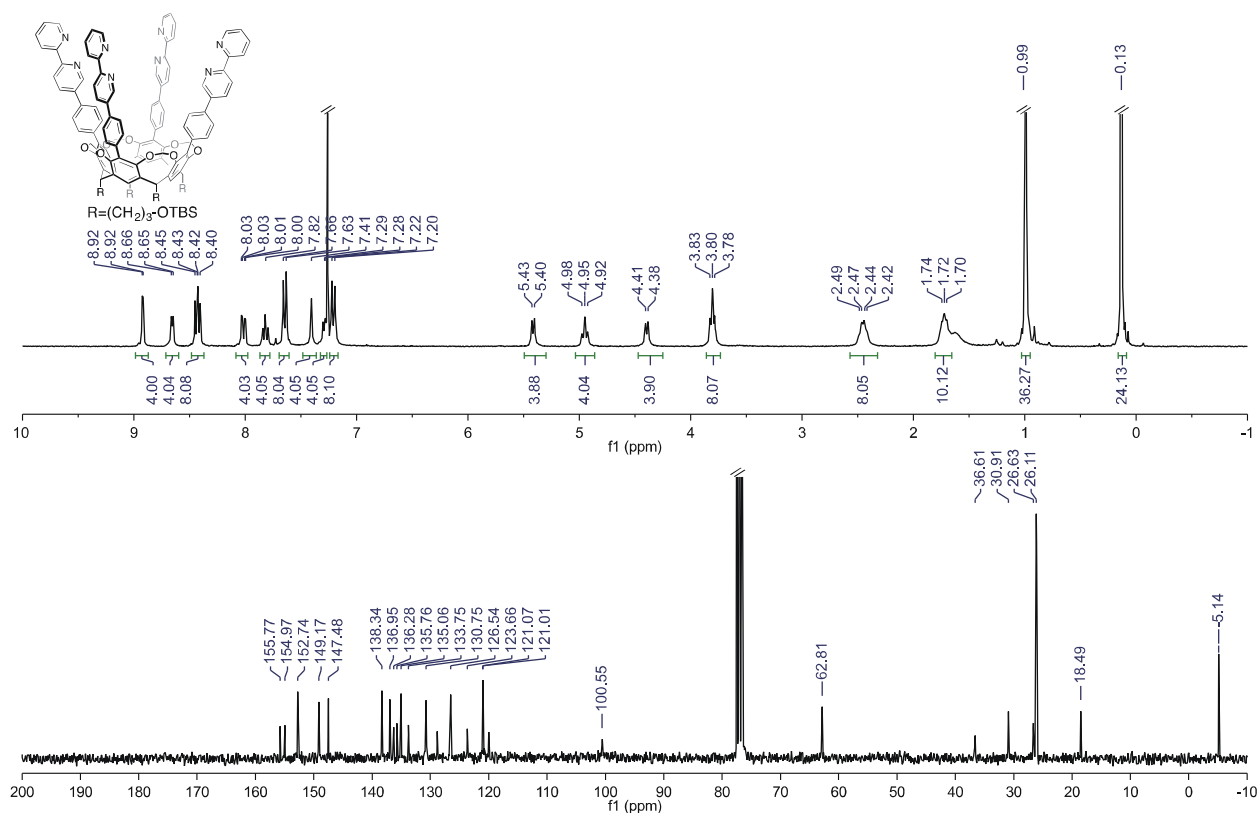




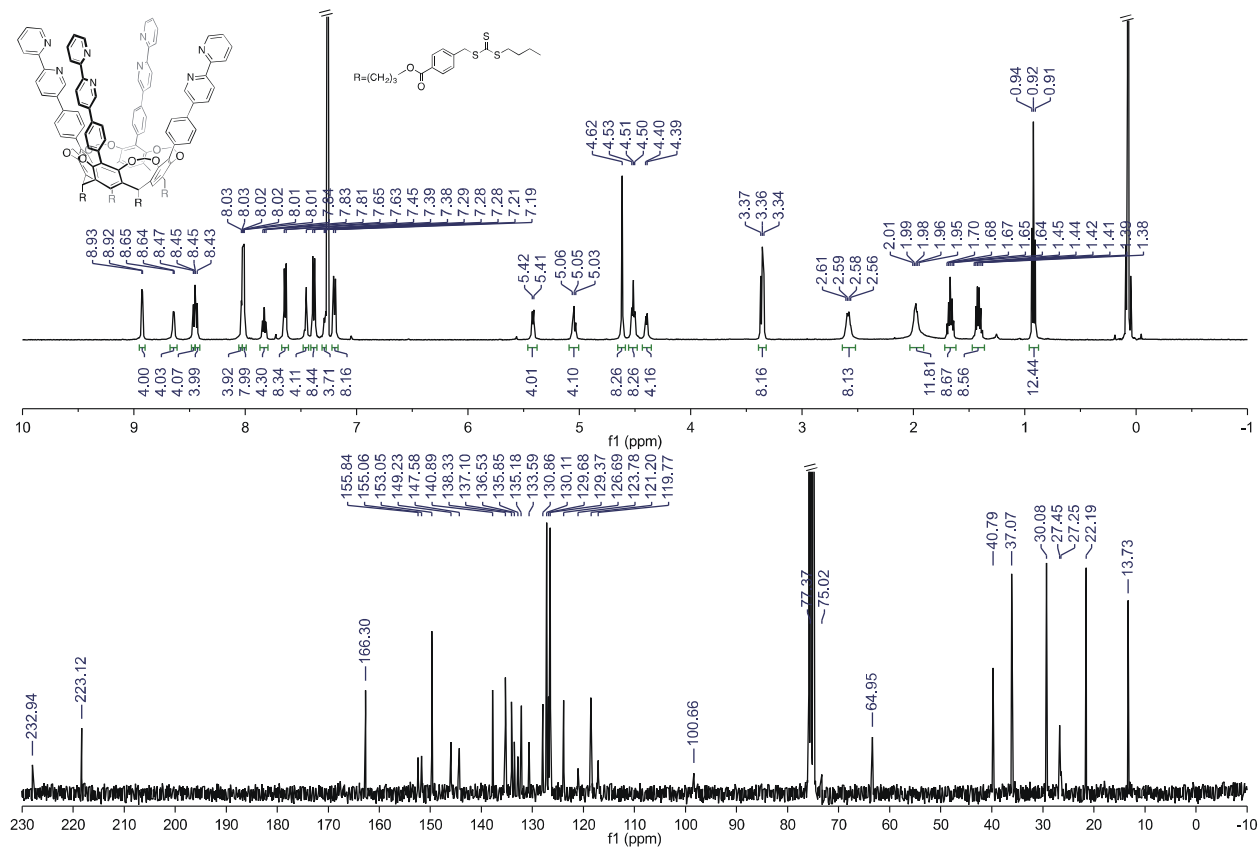
**Figure S1.**  $^1H$  (300 MHz,  $CDCl_3$ , 293 K) and  $^{13}C$  (75 MHz,  $CDCl_3$ , 293 K) NMR spectra of **4**.



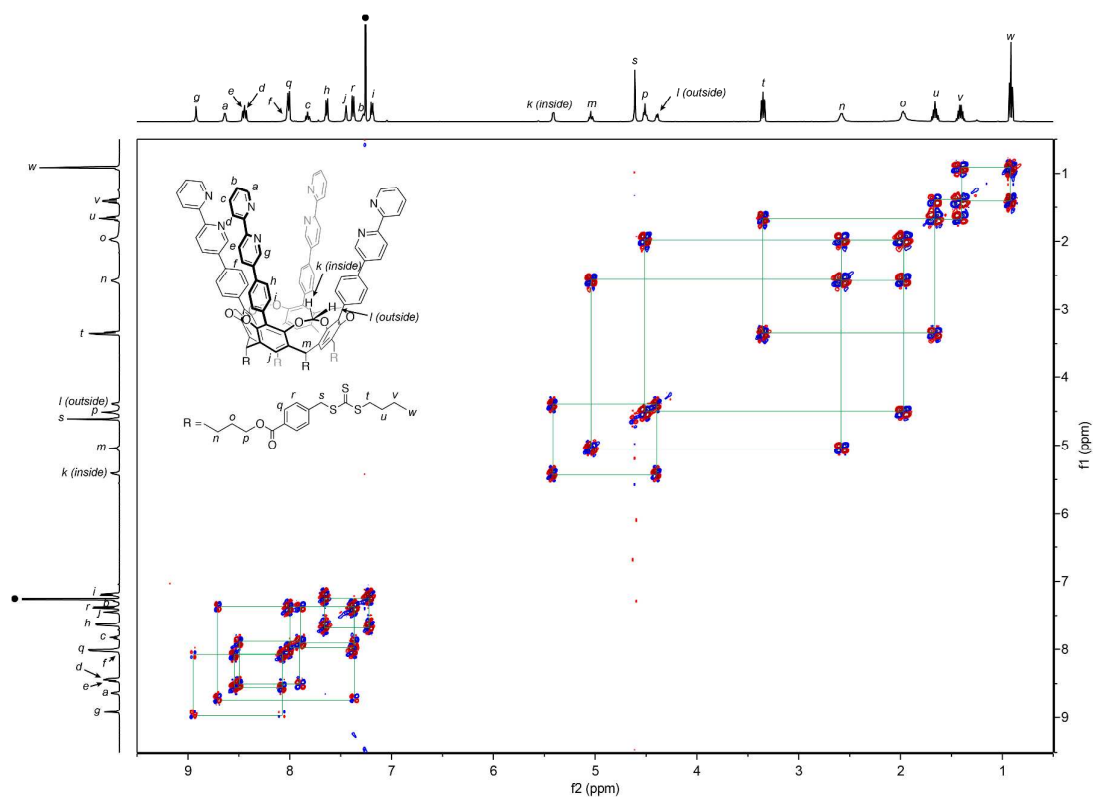
**Figure S2.**  $^1H$  (300 MHz,  $CDCl_3$ , 293 K) and  $^{13}C$  (75 MHz,  $CDCl_3$ , 293 K) NMR spectra of **5**.



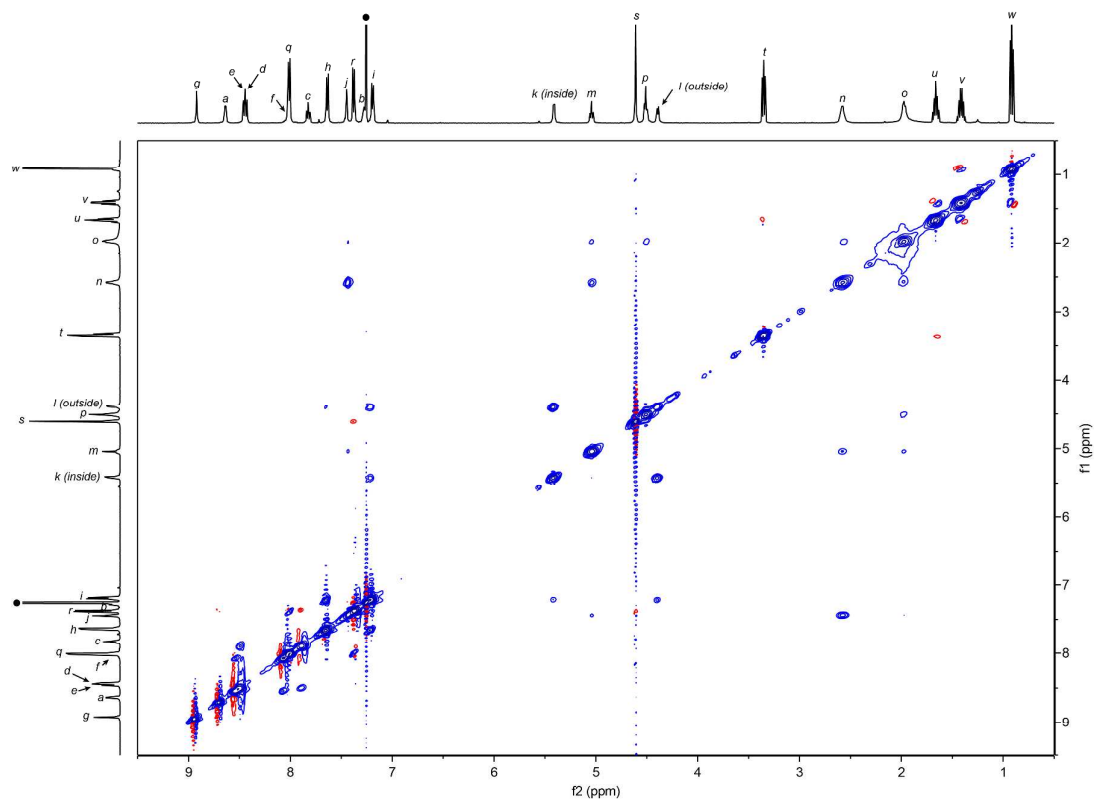
**Figure S3.**  $^1\text{H}$  (300 MHz,  $\text{chloroform-}d_1$ , 293 K) and  $^{13}\text{C}$  (75 MHz,  $\text{chloroform-}d_1$ , 293 K) NMR spectra of **6**.



**Figure S4.**  $^1\text{H}$  (300 MHz,  $\text{chloroform-}d_1$ , 293 K) and  $^{13}\text{C}$  (75 MHz,  $\text{chloroform-}d_1$ , 293 K) NMR spectra of **1**.

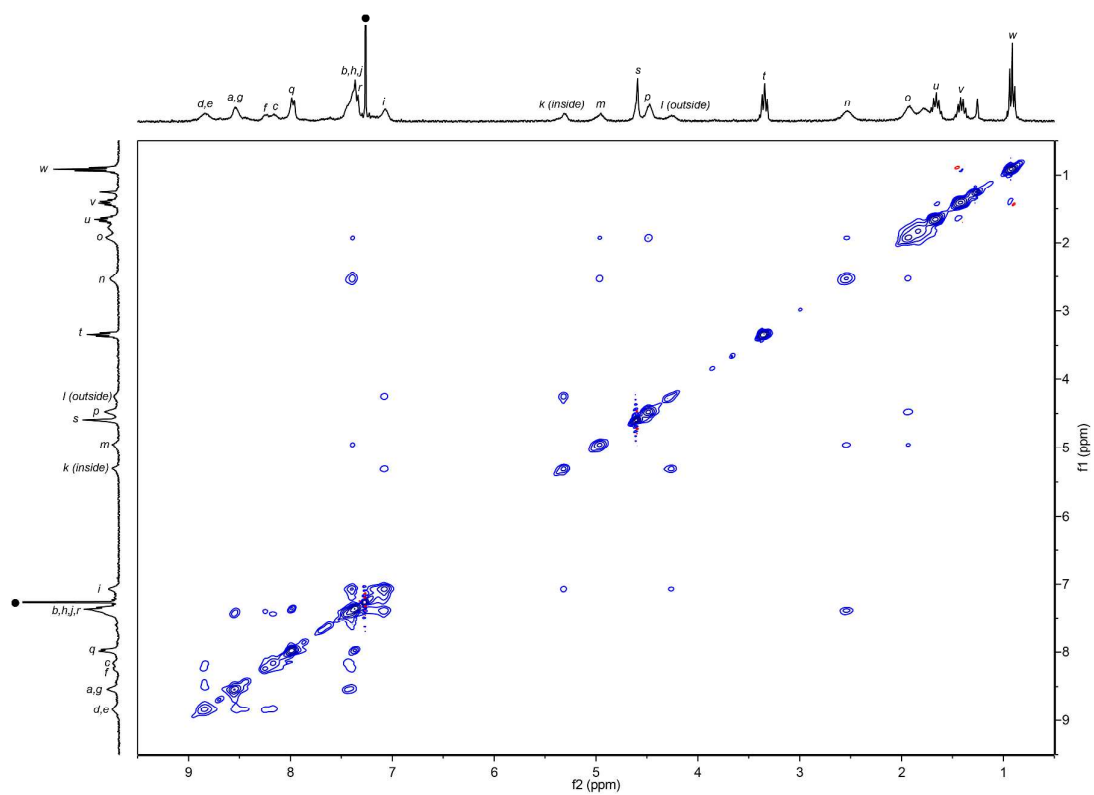


**Figure S5.** DQF-COSY spectrum (300 MHz, chloroform- $d_1$ , 293 K) of **1**.

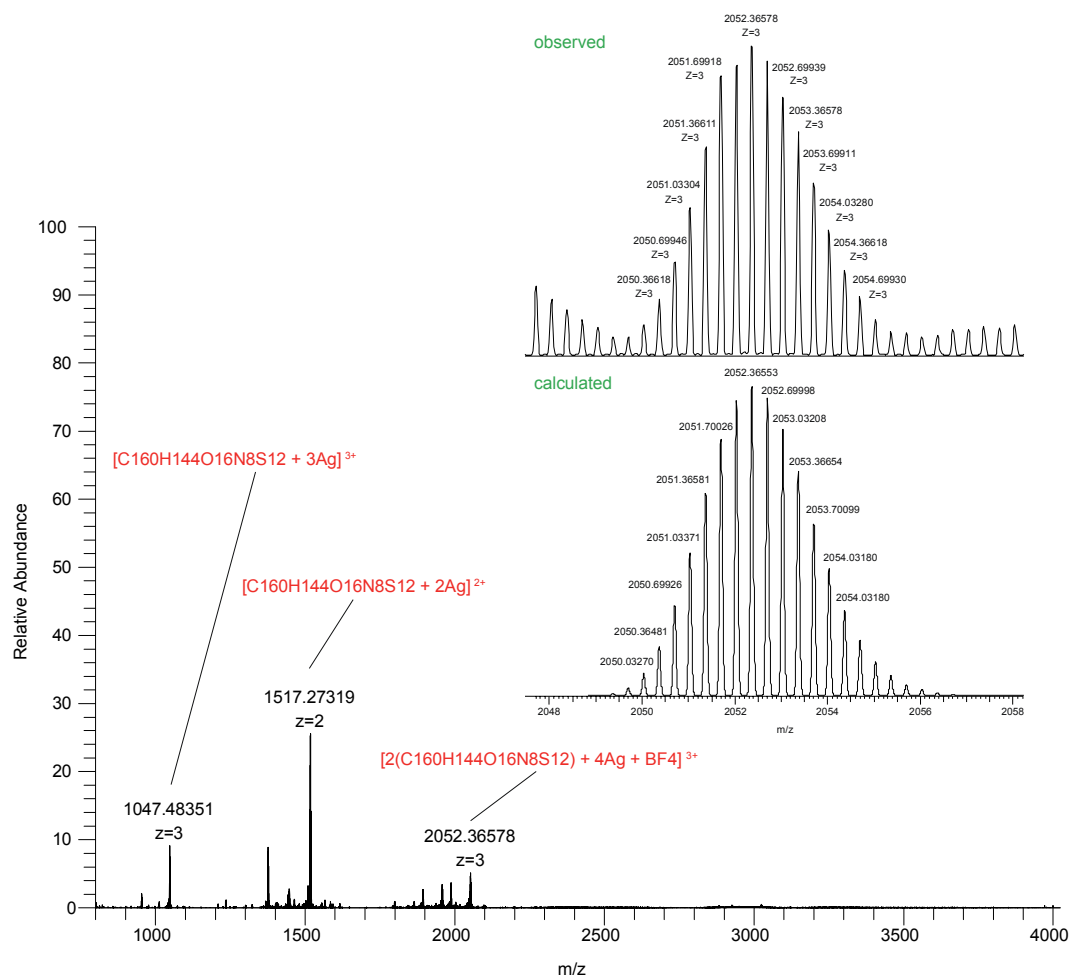


**Figure S6.** NOESY spectrum (300 MHz, chloroform- $d_1$ , 293 K) of **1**. Mixing time = 400 ms.



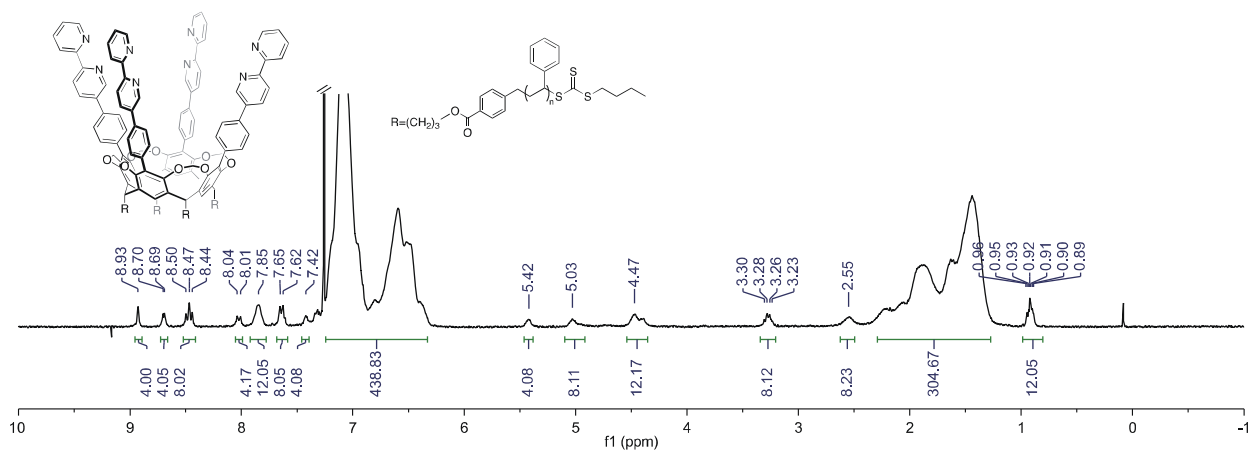


**Figure S9.** NOESY spectrum (300 MHz, chloroform- $d_1$ , 293 K) of **2** Mixing time = 400 ms.

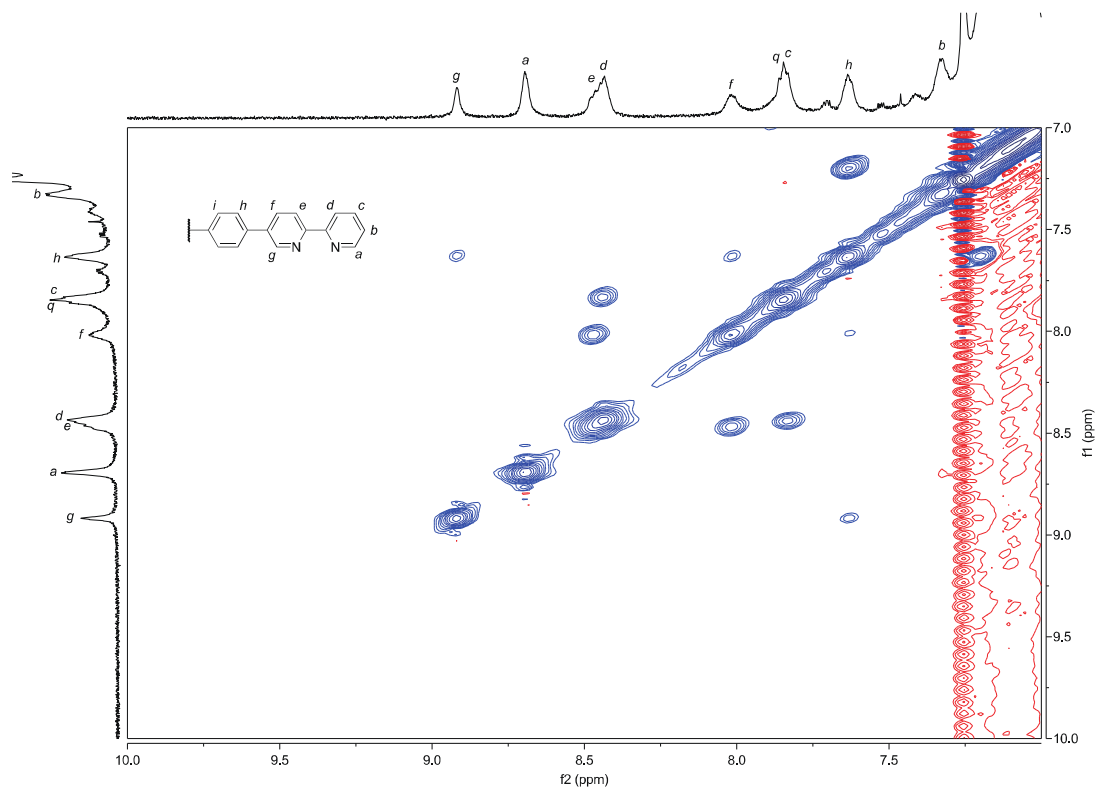


**Figure S10.** ESI-MS spectrum of **2**. The inset shows observed and calculated isotope distributions for  $[1_2 + Ag_4 + BF_4]^{3+}$ .

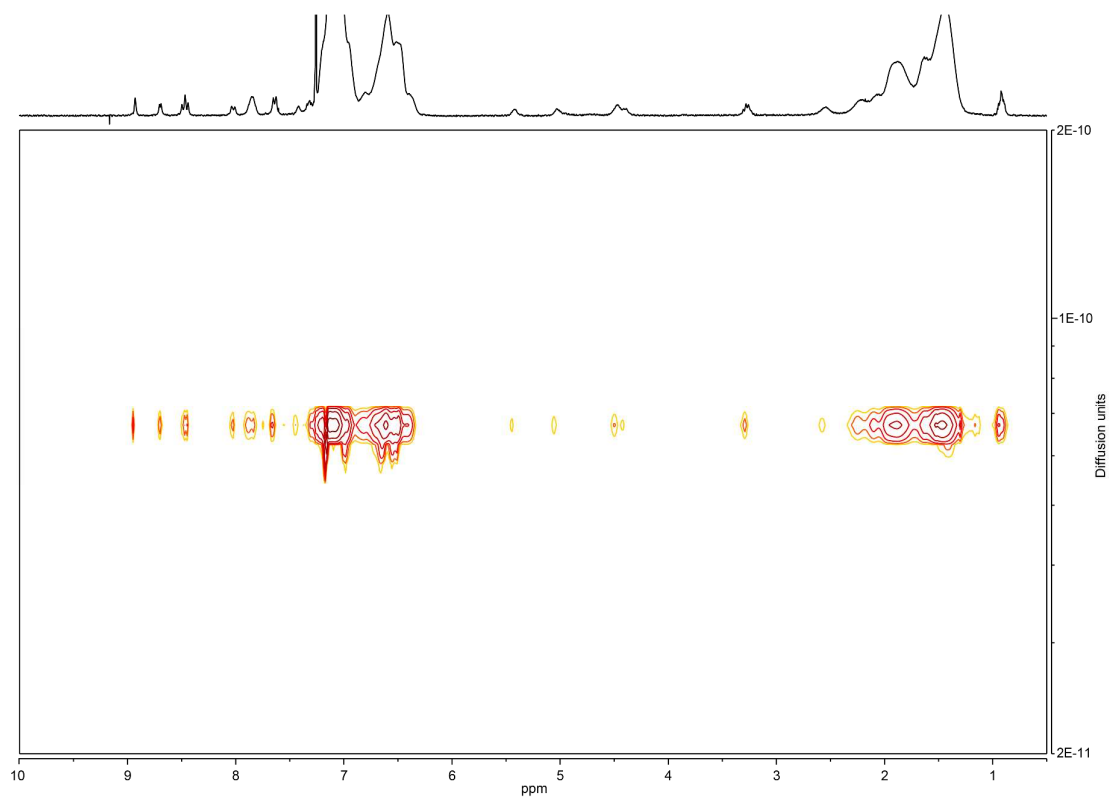
## Supporting Figures and Tables



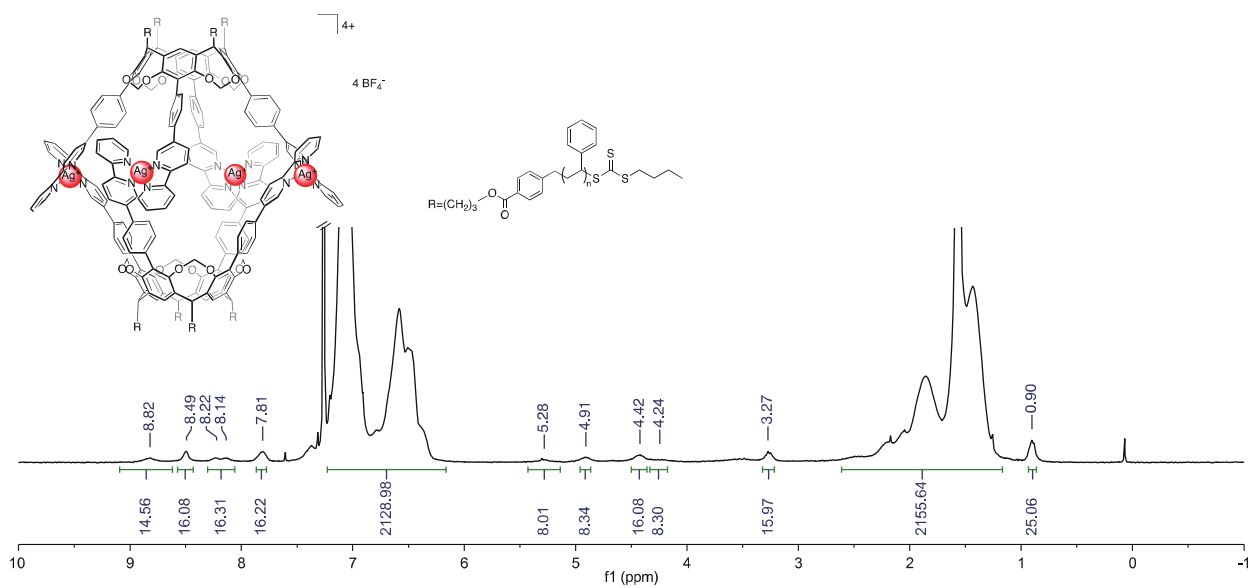
**Figure S11.**  $^1\text{H}$  (300 MHz,  $\text{CHCl}_3-d_1$ , 293 K) and NMR spectra of *poly-1*.



**Figure S12.** Partial NOESY spectrum (500 MHz,  $\text{CHCl}_3-d_1$ , 293 K) of *poly-1*. Mixing time = 500 ms.

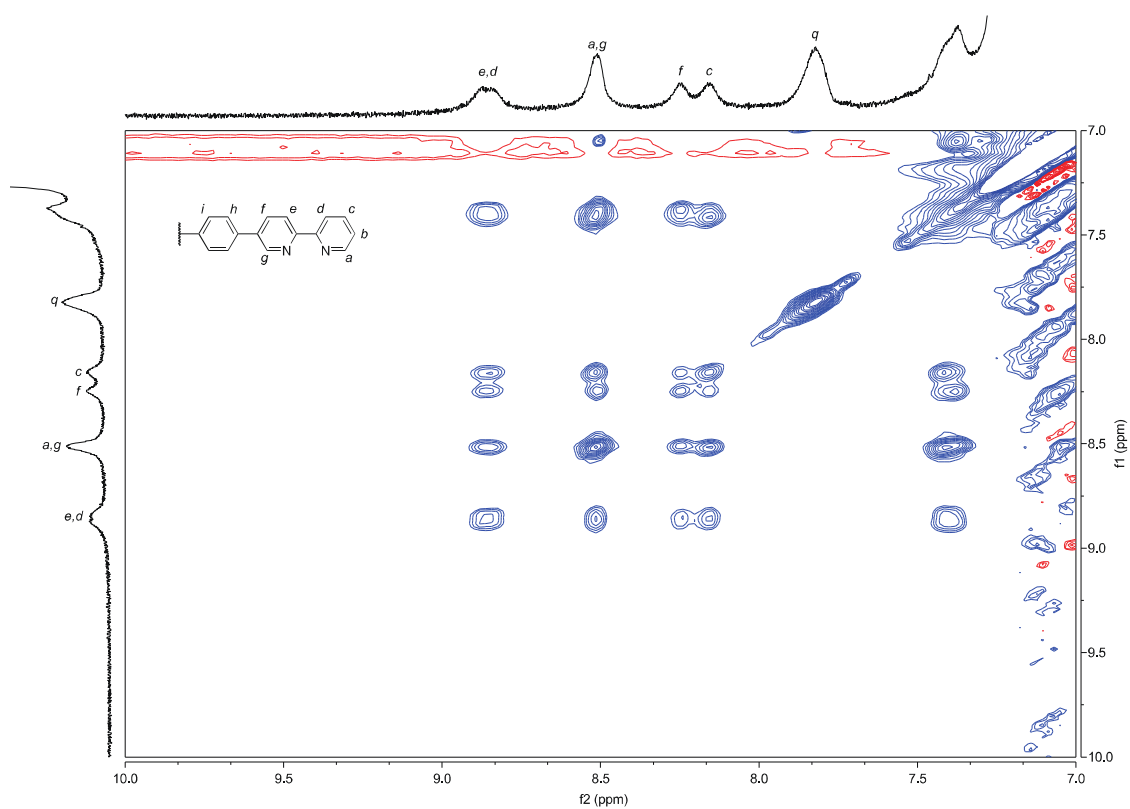


**Figure S13** DOSY spectrum (500 MHz, chloroform- $d_1$ , 293 K) of *poly-1*. The diffusion coefficient of *poly-1* was calculated to be  $6.23(2) \times 10^{-11} \text{ m}^2 \text{ s}^{-1}$ .

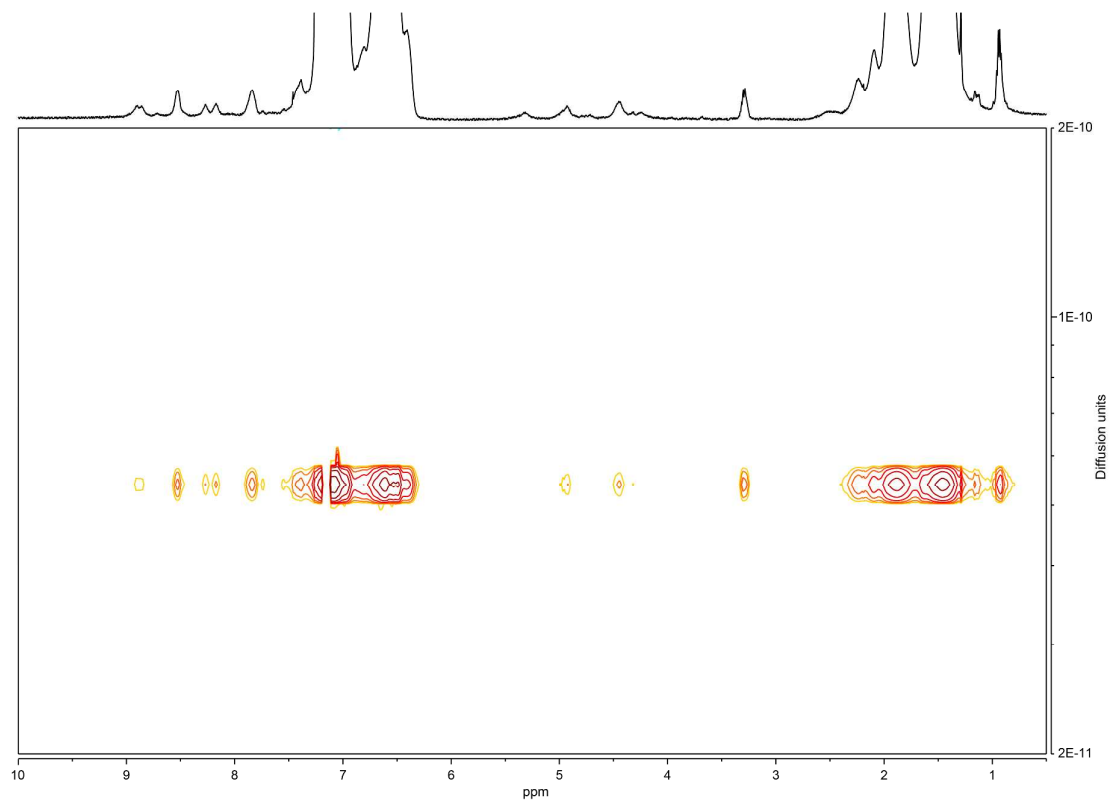


**Figure S14.**  $^1\text{H}$  (300 MHz, chloroform- $d_1$ , 293 K) NMR spectra of *poly-2*.

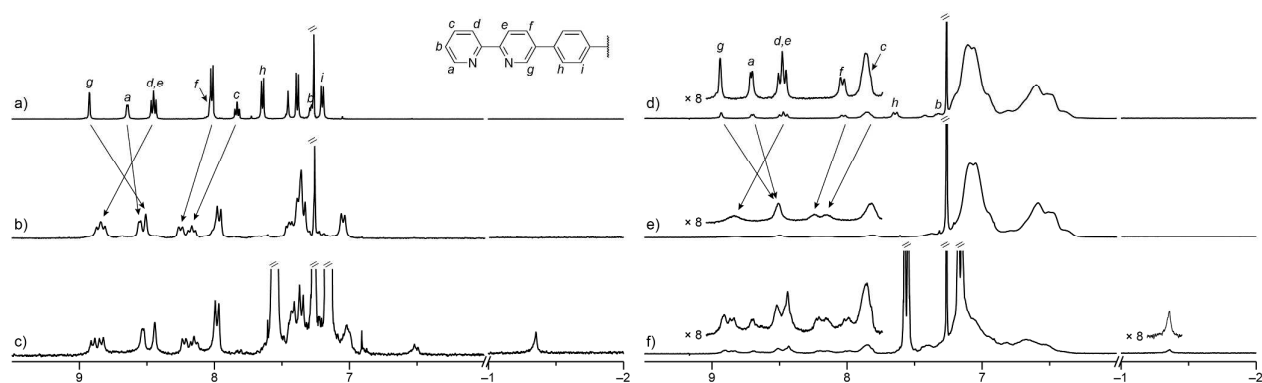




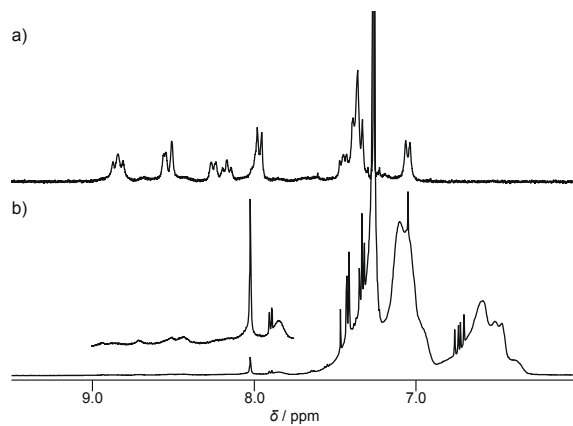
**Figure S15.** Partial NOESY spectrum (500 MHz, chloroform- $d_1$ , 293 K) of *poly-2*. Mixing time = 500 ms.



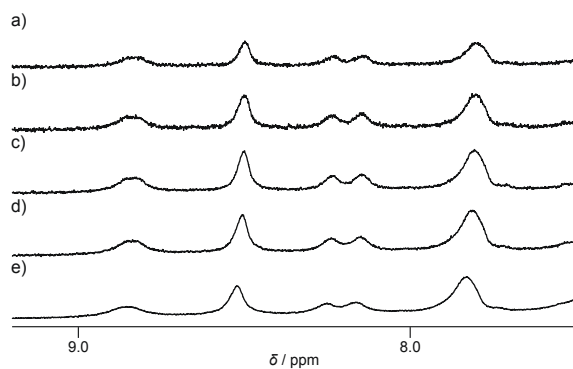
**Figure S16** DOSY spectrum (500 MHz, chloroform- $d_1$ , 293 K) of *poly-2*. The diffusion coefficient of *poly-2* was calculated to be  $5.14(5) \times 10^{-11} \text{ m}^2 \text{ s}^{-1}$ .



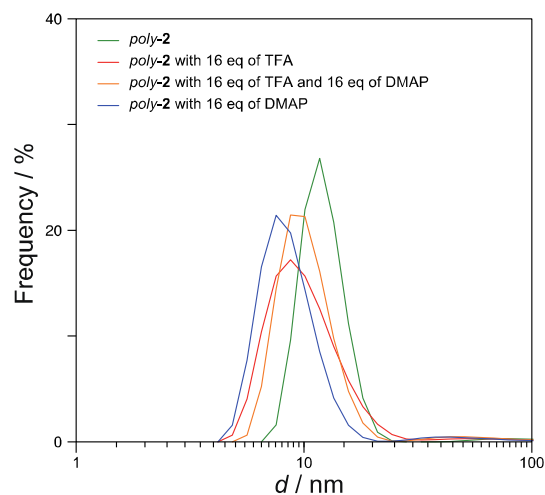
**Figure S17.** Partial  $^1\text{H}$  NMR spectra (300 MHz, chloroform- $d_1$ , 293 K) of (a) **1**, (b) **2**, (c) **2** with 20 equivalents of **G1**, (d) *poly-1*, (e) *poly-2* and (f) *poly-2* with 40 equivalents of **G1**. A new peak at  $\delta = -1.2$  ppm is assignable to the bound acetyl methyl group in **G1**.



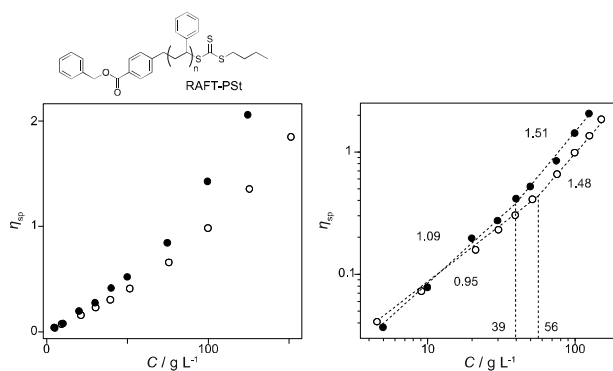
**Figure S18.**  $^1\text{H}$  NMR spectra (300 MHz, chloroform- $d_1$ ) of a) **2** and b) after the RAFT polymerization of **2** with styrene.



**Figure S19.**  $^1\text{H}$  NMR spectra of *poly-2* at 25 °C in chloroform- $d_1$ . The concentrations of *poly-2* are a) 0.10, b) 0.21, c) 0.41, d) 1.02, e) 2.05 mmol L $^{-1}$ .



**Figure S20.** DLS analysis of *poly-2* ( $0.5 \text{ mmol L}^{-1}$ ) with TFA and/or DMAP in chloroform.



**Figure S21.** Specific viscosities (left) of linear RAFT-PSt (white) and linear RAFT-PSt with 1 equiv. of  $\text{AgBF}_4$  (black) in 1,2-dichloromethane, and log-log plots (right) of the specific viscosities at 298 K

## References

- [1] Y. Tsunoda, K. Fukuta, T. Imamura, R. Sekiya, T. Furuyama, N. Kobayashi, and T. Haino, *Angew. Chem. Int. Ed.*, **2014**, *53*, 7243–7247.
- [2] Y. Tsunoda, M. Takatsuka, R. Sekiya, and T. Haino, *Angew. Chem. Int. Ed.*, **2017**, *56*, 2613–2618.