

## Supplementary Information

### Evolution of Nano- to Microsized Spherical Assemblies of Fluorogenic Biscalixarenes into Supramolecular Organogels

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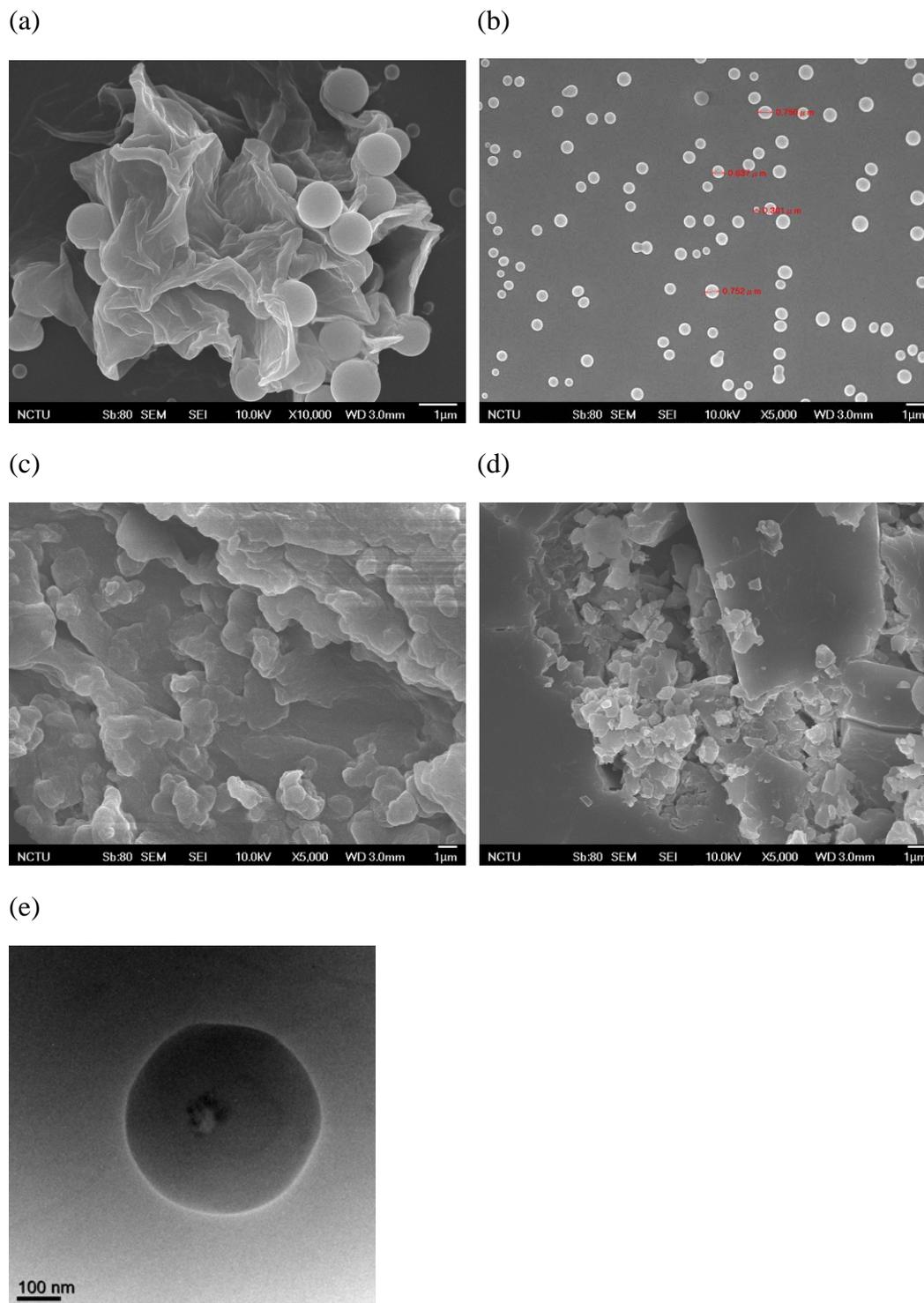
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#### Materials:

Materials: All solvents were purified according to reported procedures. Unless otherwise noted, reagents and materials were obtained from commercial suppliers and were used without further purification.

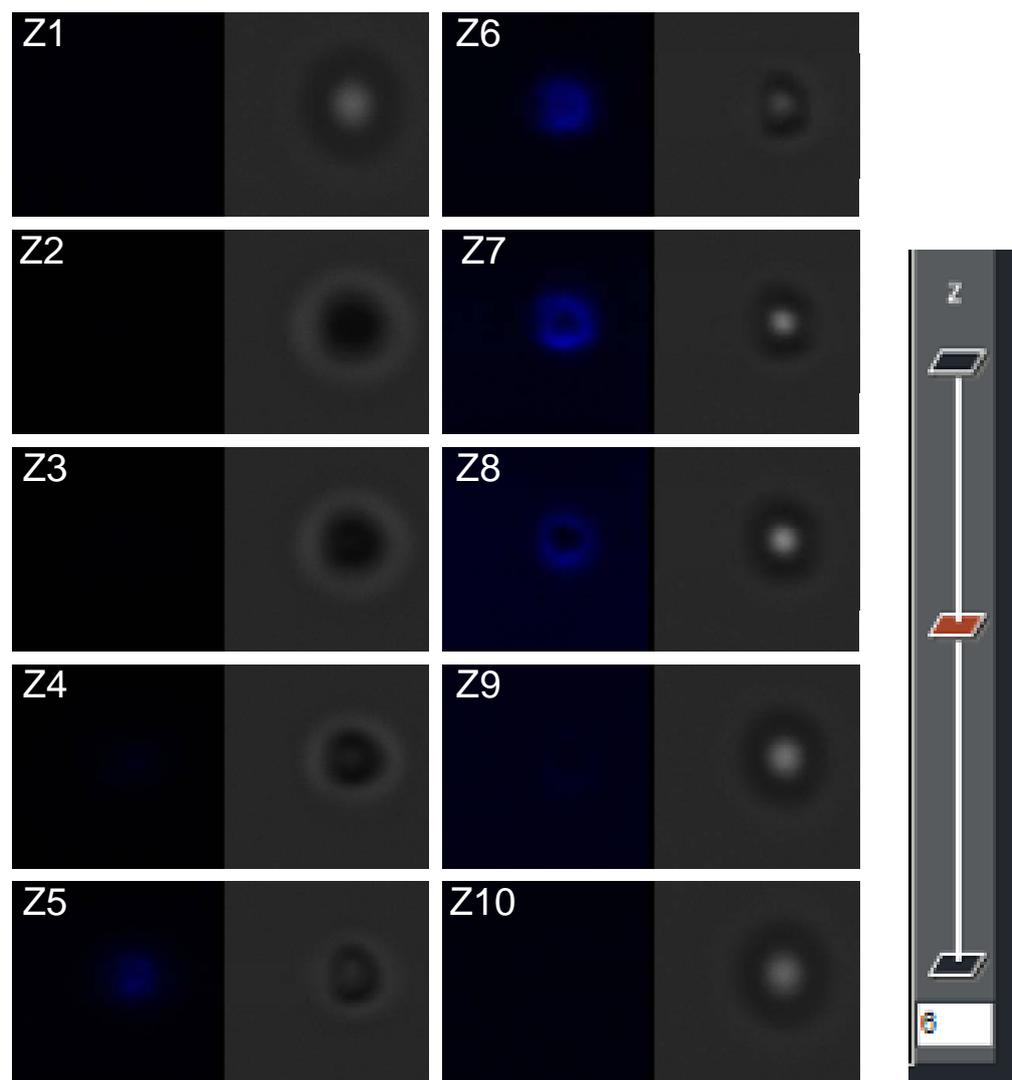
#### General Methods:

<sup>1</sup>H and <sup>13</sup>C NMR spectra were obtained on Varian VNMRS-600, UNITY INOVA-500, and Bruker DRX 300 NMR in CDCl<sub>3</sub>, CD<sub>3</sub>CN, or with TMS as internal standard. FE-SEM measurements were carried out on JEOL JSM-7401. Transmission Electron Micrographs (TEM) were recorded on a JEOL JEM-3000 F. CLSM images were recorded on Leica TCS SP5 X AOBs. UV-Vis spectra were obtained on a HP-8453 spectrophotometer. Fluorescence spectra were recorded on a HORIBA Fluoromax 4.

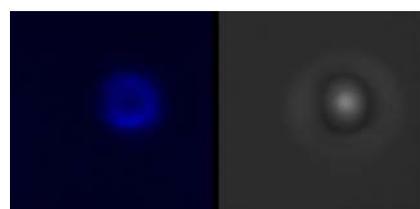


**Figure S1.** Morphology of self-assembled objects of **1**. FM-SEM images of the xerogels of **1** (a) and (b)  $5 \times 10^{-5}$  M, (c)  $1 \times 10^{-3}$  M in  $\text{CH}_3\text{CN}$ , (d)  $5 \times 10^{-3}$  M in  $\text{CHCl}_3$ , and (e) TEM image of a xerogels **1** in  $\text{CH}_3\text{CN}$  (0.5 mM).

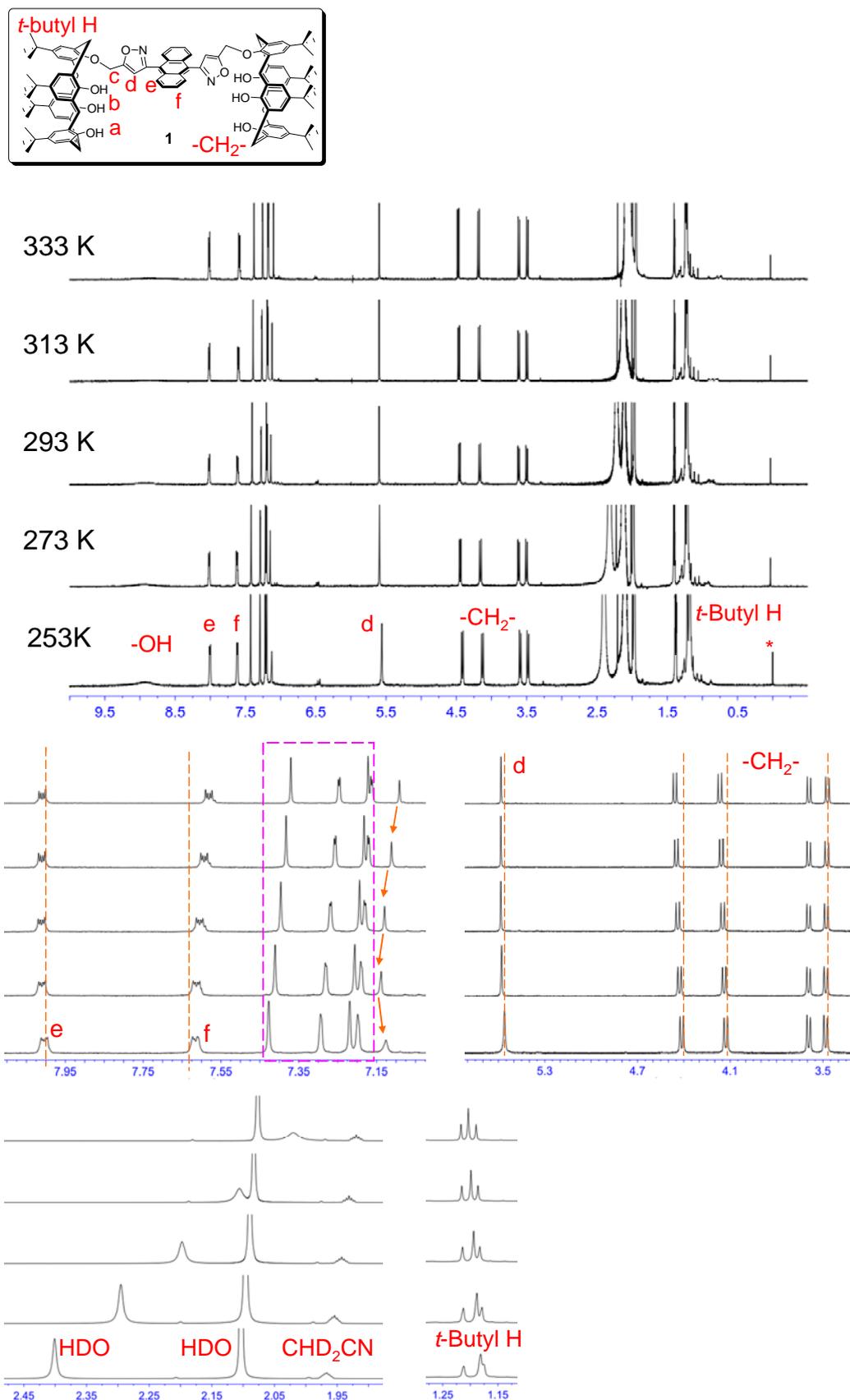
(a)



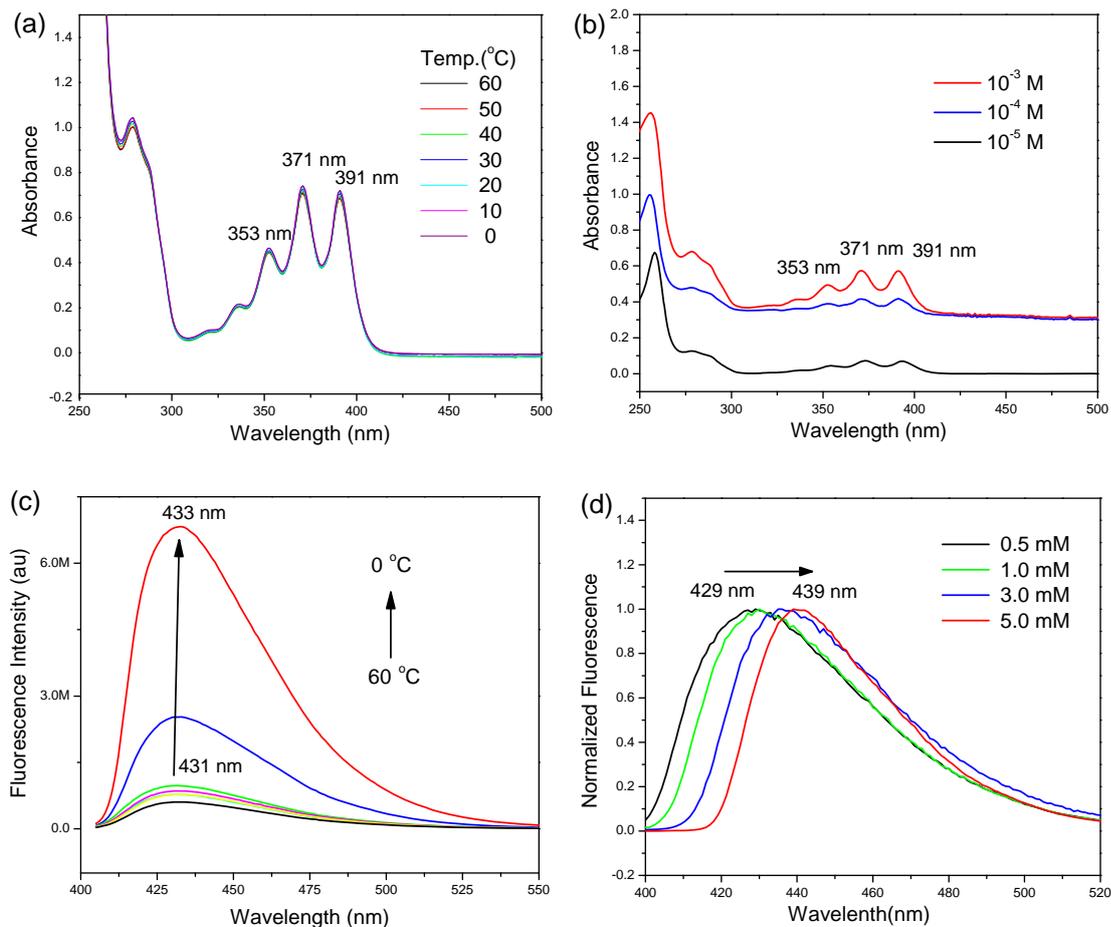
(b)



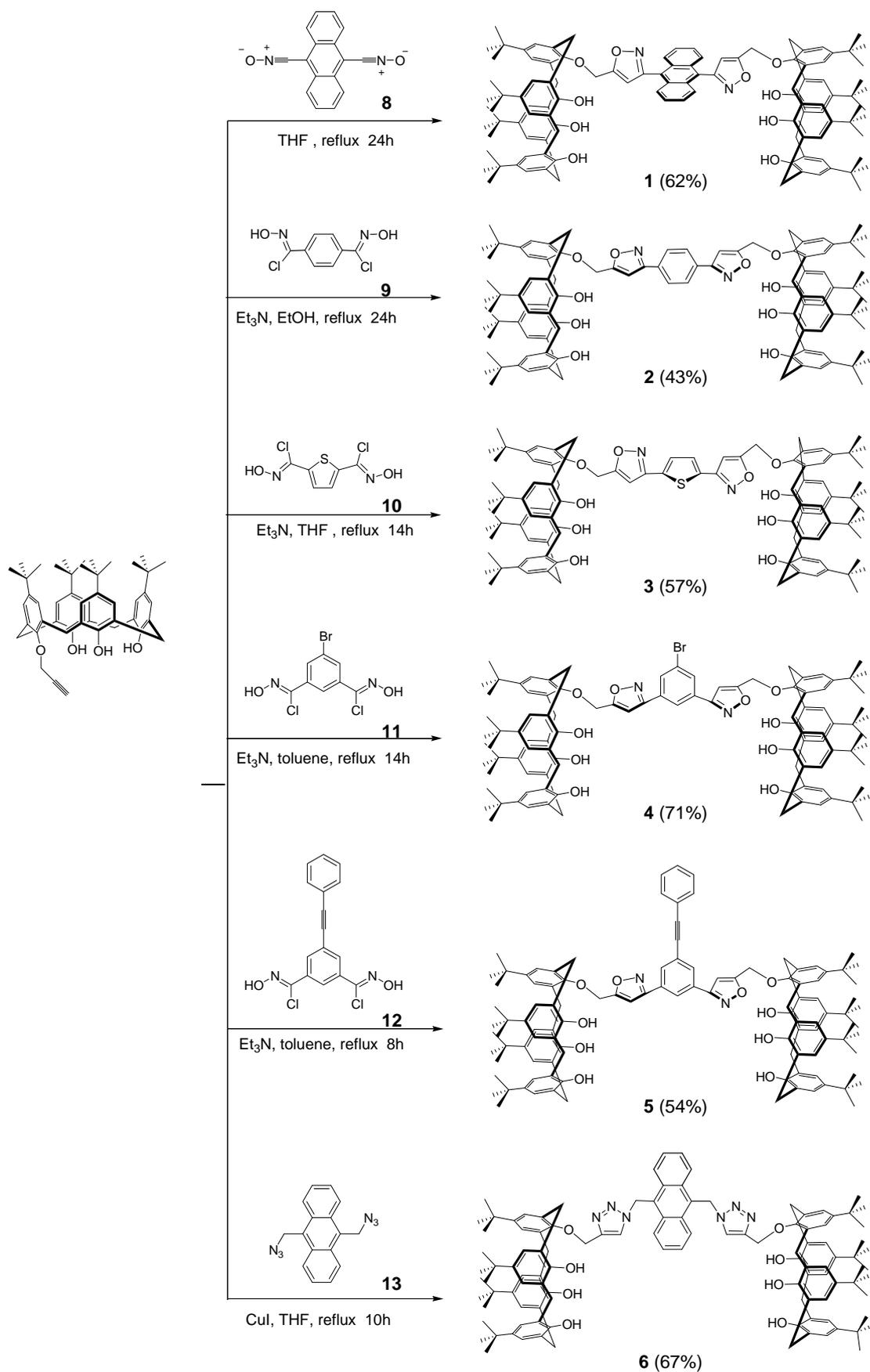
**Figure S2.** (a) CLSM images of organogel **1** with different depths in CH<sub>3</sub>CN (0.5 mM).  $\lambda_{\text{ex}} = 405 \text{ nm}$ ;  $\lambda_{\text{em}} = 415\text{--}500 \text{ nm}$ . (b) Entire image stack in Z axis.



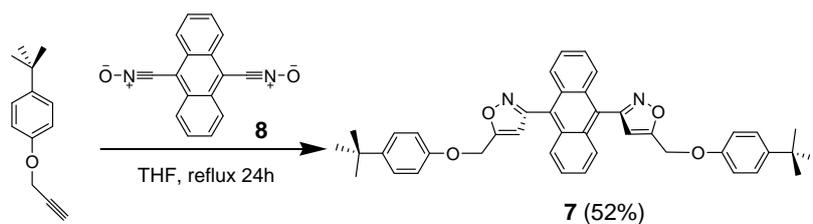
**Figure S3.** Variable-temperature  $^1\text{H-NMR}$  spectra of gelator **1** (3.0 mM) in  $\text{CD}_3\text{CN}$ , where \* denotes signal of an external standard TMS.



**Figure S4.** Changes in the UV-Vis and fluorescence spectra of biscalixarene **1** in  $\text{CH}_3\text{CN}$ . (a) Temperature-dependent absorption spectra (0.5 mM), (b) concentration-dependent absorption spectra, (c) temperature-dependent fluorescence spectra (2.0 mM), and (d) normalized fluorescence spectra for various concentrations of **1** at 25 °C. The excitation wavelength was 391 nm.



Scheme S1. Syntheses of biscalix[4]arenes **1-6**.



Scheme S2. Synthesis of control compound 7.

General procedures for the syntheses of biscalix[4]arenes **1-6**.

**9,10-Bis-isoxazolyllanthrylmethyl linked biscalix[4]arene, 1.** A mixture of 25-propargyloxy-26,27,28-trihydroxycalix[4]arene (0.20 g, 0.30 mmol) and **8** (0.04 g, 0.15 mmol) in THF (15 mL) was heated at reflux for 24 h under N<sub>2</sub> (g). After evaporation of the solvent, the mixture was washed with water and extracted with dichloromethane. The organic phase was dried over MgSO<sub>4</sub> and the solvent was removed under reduced pressure. The residue was purified by silica gel column chromatography with ethyl acetate/*n*-hexane as eluent to give 0.15 g (62.2%) of **1** as a yellow solid; mp 180–182 °C; *R<sub>f</sub>* = 0.35 (ethyl acetate/*n*-hexane (v/v, 1:5)). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ<sub>H</sub> 10.13 (s, 2H), 9.34 (s, 4H), 8.11–8.08 (m, 4H), 7.61–7.58 (m, 4H), 7.28–7.07 (m, 18H), 5.63 (s, 4H), 4.55(d, 4H, *J* = 13.2 Hz), 4.33 (d, 4H, *J* = 13.8 Hz), 3.61 (d, 4H, *J* = 13.2 Hz), 3.51 (d, 4H, *J* = 13.8 Hz), 1.33–1.29 (m, 72H) ppm. <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75.5 MHz) δ<sub>C</sub> 167.2 (Cq), 161.4 (Cq), 149.1 (Cq), 148.8 (Cq), 148.3 (Cq), 147.7 (Cq), 143.6 (Cq), 143.3 (Cq), 133.5 (Cq), 130.3 (Cq), 128.1 (Cq), 127.7 (Cq), 127.7(Cq), 126.8 (CH), 126.0 (CH), 125.9 (CH), 125.7 (CH), 125.7 (CH), 125.5 (Cq), 108.8 (CH) , 67.6 (CH<sub>2</sub>), 34.3 (Cq), 34.0 (Cq), 33.9 (Cq), 32.9 (CH<sub>2</sub>), 32.4 (CH<sub>2</sub>), 31.5 (CH<sub>3</sub>), 31.2 (CH<sub>3</sub>) ppm. FAB-MS *m/z* 1634 (M + H<sup>+</sup>), 1633 (M<sup>+</sup>); HRMS (FAB) calcd for C<sub>110</sub>H<sub>124</sub>O<sub>10</sub>N<sub>2</sub> 1632.9256; found 1632.9275.

**1,4-Bis-isoxazolyll-phenylmethyl linked biscalix[4]arene, 2.** Triethylamine (0.35 mmol) in

ethanol (1.9 mL) was slowly added to a well-stirred solution of 25-propargyloxy-26,27,28-trihydroxycalix[4]arene (0.40 g, 0.69 mmol) and hydroximoyl chloride **9** (0.07 g, 0.31 mmol) in ethanol (30 mL). The reaction mixture was stirred at reflux for 24 h under N<sub>2</sub> (g). After evaporation of the solvent, the mixture was washed with water and extracted with dichloromethane. The organic phase was dried over MgSO<sub>4</sub> and the solvent was removed under reduced pressure. The residue was purified by silica gel column chromatography using ethyl acetate/*n*-hexane as eluent to give 0.20 g (42.7%) of **2** as a yellow solid; mp 178–180 °C; *R<sub>f</sub>* = 0.45 (ethyl acetate/*n*-hexane = 1:4); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ<sub>H</sub> 10.00 (s, 2H), 9.16 (s, 4H), 8.03 (s, 4H), 7.12–6.99 (m, 16H), 5.39 (s, 4H), 4.33 (d, 4H, *J* = 13.2 Hz), 4.26 (d, 4H, *J* = 13.7 Hz), 3.46 (d, 4H, *J* = 13.2 Hz), 3.44 (d, 4H, *J* = 13.7 Hz), 1.22–1.20 (m, 72H) ppm. <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75.5 MHz): δ<sub>C</sub> 167.6 (Cq), 162.1 (Cq), 149.0 (Cq), 148.9 (Cq), 148.3 (Cq), 147.5 (Cq), 143.7 (Cq), 143.2 (Cq), 133.2 (Cq), 130.4 (Cq), 128.1 (Cq), 127.7 (Cq), 127.6 (CH), 127.4 (Cq), 126.7 (CH), 125.8 (CH), 125.7 (CH), 125.6 (CH), 102.8 (CH), 68.1 (CH<sub>2</sub>), 34.3 (Cq), 34.0 (Cq), 33.9 (Cq), 32.9 (CH<sub>2</sub>), 32.2 (CH<sub>2</sub>), 31.4 (CH<sub>3</sub>), 31.2 (CH<sub>3</sub>) ppm. FAB-MS *m/z* 1534 (M + H<sup>+</sup>), 1533 (M<sup>+</sup>); HRMS (FAB) calcd for C<sub>102</sub>H<sub>120</sub>O<sub>10</sub>N<sub>2</sub> 1532.8943; found: 1532.8916.

**2,5-Bis-isoxazolylthiophene-methyl linked biscalic[4]arene, 3.** Triethylamine (97.1 mg, 0.96 mmol) dissolved in toluene (3 mL) was slowly added to a well-stirred solution of 25-propargyloxy-26,27,28-trihydroxycalix[4]arene (0.7g, 1.02 mmol) and hydroxyimoyl chloride **10** (0.12g, 0.48 mmol) in toluene (7 mL). The reaction mixture was stirred at reflux for 14 h under N<sub>2</sub> (g). After evaporation of the solvent, the mixture was washed with water and extracted with ethyl acetate. The organic layer was dried over MgSO<sub>4</sub> and the solvent was removed under reduced pressure. The residue was purified by silica gel column chromatography with ethyl acetate/*n*-hexane to give **3** as a yellow solid in 57% (425 mg) yield. mp 163-168 °C; *R<sub>f</sub>* = 0.63 (ethyl acetate/*n*-hexane (v/v, 1:4)). <sup>1</sup>H NMR (300 MHz,

CDCl<sub>3</sub>)  $\delta_{\text{H}}$  9.99 (s, 2H), 9.12 (s, 4H), 7.59 (s, 2H), 7.11-6.99 (m, 18H), 5.37 (s, 4H), 4.31 (ABq,  $J = 13.2$  Hz, 4H), 4.26 (ABq,  $J = 14.4$  Hz, 4H), 3.45 (ABq,  $J = 13.2$  Hz, 4H), 3.43 (ABq,  $J = 14.4$  Hz, 4H), 1.29-1.19 (m, 72H) ppm. <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75.5 MHz)  $\delta_{\text{C}}$  167.7 (Cq), 157.6 (Cq), 148.9 (Cq), 148.8 (Cq), 147.5 (Cq), 143.7 (Cq), 143.3 (Cq), 133.2 (Cq), 132.5 (Cq), 128.3 (CH), 128.2 (Cq), 127.6 (Cq), 127.4 (Cq), 126.8 (CH), 125.9 (CH), 125.7 (CH), 125.6 (CH), 102.8 (CH), 77.2 (Cq), 68.0 (CH<sub>2</sub>), 34.4 (Cq), 34.0 (Cq), 33.9 (Cq), 32.9 (CH<sub>2</sub>), 32.2 (CH<sub>2</sub>), 31.5 (CH<sub>3</sub>), 31.4 (CH<sub>3</sub>), 31.2 (CH<sub>3</sub>) ppm. FAB-MS  $m/z$  1540 (M + H<sup>+</sup>); HRMS (FAB) calcd for C<sub>100</sub>H<sub>118</sub>O<sub>10</sub>N<sub>2</sub>S 1538.8507; found 1538.8536.

**1,3-(5-Bromo)-bis-isoxazolybenzen-methyl linked biscalix[4]arene, 4.** Triethylamine (0.03 g, 0.32 mmol) dissolved in toluene (1 mL) was slowly added to a well-stirred solution of 25-propargyloxy-26,27,28-trihydroxycalix[4]arene (0.22 g, 0.32 mmol) and hydroxyimoyl chloride **11** (0.05 g, 0.16 mmol) in toluene (4 mL). The reaction mixture was stirred at reflux for 14 h under N<sub>2</sub>(g). After evaporation of the solvent, the mixture was washed with water and extracted with ethyl acetate. The organic layer was dried over MgSO<sub>4</sub> and the solvent was removed under reduced pressure. The residue was purified by silica gel column chromatography with ethyl acetate /*n*-hexane to give **4** as a white solid in 71% (0.18 g) yield. mp 164-167 °C;  $R_f = 0.71$  (ethyl acetate/*n*-hexane (v/v, 1:5)). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta_{\text{H}}$  9.98 (s, 2H), 9.12 (s, 4H), 8.31 (t,  $J = 0.9$  Hz, 1H), 8.18 (d,  $J = 0.9$  Hz, 2H), 7.12-6.99 (m, 18H), 5.39 (s, 4H), 4.31 (ABq,  $J = 13.2$  Hz, 4H), 4.26 (ABq,  $J = 14.4$  Hz, 4H), 3.45 (ABq,  $J = 13.2$  Hz, 4H), 3.43 (ABq,  $J = 14.4$  Hz, 4H), 1.29-1.19 (m, 72H) ppm. <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75.5 MHz)  $\delta_{\text{C}}$  167.9 (Cq), 161.1 (Cq), 148.9 (Cq), 148.3 (Cq), 147.5 (Cq), 143.6 (Cq), 143.2 (Cq), 133.2 (Cq), 131.3 (CH), 128.1 (Cq), 127.6 (Cq), 127.4 (Cq), 126.8 (CH), 125.9 (CH), 125.7 (CH), 125.6 (CH), 124.2 (CH), 123.7 (Cq), 102.9 (CH), 67.8 (CH<sub>2</sub>), 34.3 (Cq), 34.0 (Cq), 33.9 (Cq), 32.9 (CH<sub>2</sub>), 32.2 (CH<sub>2</sub>), 31.5 (CH<sub>3</sub>), 31.2 (CH<sub>3</sub>) ppm. FAB-MS  $m/z$  1612 (M + H<sup>+</sup>); HRMS (FAB) calcd for C<sub>102</sub>H<sub>119</sub>O<sub>10</sub>N<sub>2</sub>Br 1610.8048; found 1610.8043.

**1,3-(5-Phenylethynyl)-bis-isoxazolybenzen-methyl linked biscalix[4]arene, 5.** Triethylamine (0.012g, 0.12 mmol) dissolved in toluene (1 mL) was slowly added to a well-stirred solution of 25-propargyloxy-26,27,28-trihydroxycalix[4]arene (0.08g, 0.12 mmol) and hydroxyimoyl chloride **12** (0.02 g, 0.06 mmol) in toluene (4 mL). The reaction mixture was stirred at reflux for 8 h under N<sub>2(g)</sub>. After evaporation of the solvent, the mixture was washed with water and extracted with dichloromethane. The organic layer was dried over MgSO<sub>4</sub> and the solvent was removed under reduced pressure. The residue was purified by silica gel column chromatography with ethyl acetate/*n*-hexane to give **5** as a white solid in 54.0% (0.05g) yield. mp 135-140 °C; *R*<sub>f</sub> = 0.56 (*n*-hexane (v/v, 1:5)). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ<sub>H</sub> 10.01 (s, 2H), 9.13 (s, 4H), 8.35 (t, *J* = 1.5 Hz, 1H), 8.17 (d, *J* = 1.5 Hz, 2H), 7.60-7.56 (m, 2H), 7.47-7.44 (m, 3H), 7.25-6.98 (m, 18H), 5.39 (s, 4H), 4.31 (d, 4H, *J* = 13.5 Hz), 4.26 (d, 4H, *J* = 13.7 Hz), 3.45 (d, 4H, *J* = 13.5 Hz), 3.42 (d, 4H, *J* = 13.7 Hz), 1.22-1.20 (m, 72H) ppm. <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75.5 MHz) δ<sub>C</sub> 167.7 (Cq), 161.7 (Cq), 148.9 (Cq), 148.8 (Cq), 148.3 (Cq), 147.6 (Cq), 143.6 (Cq), 143.2 (Cq), 133.3 (Cq), 131.8 (CH), 131.4 (CH), 129.9 (Cq), 128.7 (CH), 128.4 (CH), 128.2(Cq), 127.7 (Cq), 127.5(Cq), 126.8(CH), 125.9 (CH), 125.7 (CH), 125.6 (CH), 125.2 (Cq), 125.0 (CH), 122.6 (Cq), 103.0 (CH), 91.3 (Cq), 87.9 (Cq), 67.8 (CH<sub>2</sub>), 34.4 (Cq), 34.0 (Cq), 33.9 (Cq), 32.9 (CH<sub>2</sub>), 32.2 (CH<sub>2</sub>), 31.5 (CH<sub>3</sub>), 31.2 (CH<sub>3</sub>). FAB-MS *m/z* 1634 (M + H<sup>+</sup>); HRMS (FAB) calcd for C<sub>110</sub>H<sub>124</sub>O<sub>10</sub>N<sub>2</sub> 1632.9256, found 1632.9218.

**9,10-(Bis-triazole)-methylantrhryl methyl linked biscalix[4]arene, 6.** To a well stirred solution of 25-propargyloxy-26,27,28-trihydroxycalix[4]arene (476 mg, 0.7 mmol) and anthracene derivative **13** (100 mg, 0.34 mmol) in 10 mL THF was added CuI (13.2 mg, 0.07 mmol). The reaction mixture was reflux for 10 h. After evaporation of solvent, the residue was dissolved in CH<sub>2</sub>Cl<sub>2</sub> and extracted with H<sub>2</sub>O. The organic layer was dried over MgSO<sub>4</sub> and the filtrate was concentrated under reduced pressure. The solid product was purified by

column chromatography to afford **6** (394 mg, 67%) as a yellow powder. mp 198-200 °C;  $R_f = 0.33$  (*n*-hexane (v/v, 1:4)).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta_{\text{H}}$  9.15 (s, 2H), 8.70 (s, 4H), 8.49–8.46 (m, 4H), 7.91 (s, 2H), 7.68–7.64 (m, 4H), 6.98–6.83 (m, 16H), 6.37 (s, 4H), 5.12 (s, 4H), 4.11 (d, 4H,  $J = 13.2$  Hz), 3.36 (d, 4H,  $J = 13.5$  Hz), 3.24 (d, 4H,  $J = 13.2$  Hz), 3.16 (d, 4H,  $J = 13.5$  Hz), 1.15–1.14 (m, 72H) ppm.  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75.5 MHz)  $\delta_{\text{C}}$  148.6 (Cq), 148.4 (Cq), 147.8 (Cq), 147.4 (Cq), 143.6 (Cq), 143.1 (Cq), 133.6 (Cq), 130.8 (Cq), 127.9 (Cq), 127.7 (CH), 127.6 (Cq), 127.5 (Cq), 127.4 (Cq), 126.7 (Cq), 126.3 (CH), 125.7 (CH), 125.5 (CH), 124.7 (Cq), 124.2 (CH), 123.8 (CH), 69.4 ( $\text{CH}_2$ ), 46.3 ( $\text{CH}_2$ ), 34.2 (Cq), 34.0 (Cq), 33.8 (Cq), 32.6 ( $\text{CH}_2$ ), 32.0 ( $\text{CH}_2$ ), 31.5 ( $\text{CH}_3$ ), 31.4 ( $\text{CH}_3$ ), 31.1 ( $\text{CH}_3$ ). FAB-MS  $m/z$  1661 ( $\text{M}^+$ ); HRMS (FAB) calcd for  $\text{C}_{110}\text{H}_{128}\text{O}_8\text{N}_6$  1660.9794, found 1660.9790.

**3,3'-Anthracene-9,10-diylbis{5-[4-*tert*-butylphenoxy)methyl]isoxazole}, 7.** A mixture of 1-*tert*-butyl-4-(prop-2-ynoxy)benzene (0.20 g, 1.07 mmol) and **8** (0.12 g, 0.48 mmol) in THF (25 mL) was stirred and heated at reflux for 24 h under  $\text{N}_2$  system. The solvent was removed under vacuum and the residue was purified by silica gel column chromatography with ethyl acetate/*n*-hexane as eluent to give 0.16 g (52.1%) of **7** as a yellow solid; mp 231–233 °C;  $R_f = 0.45$  (ethyl acetate/*n*-hexane (v/v, 1:4)).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta_{\text{H}}$  7.88–7.85 (m, 4H), 7.49–7.45 (m, 4H), 7.38 (d, 4H,  $J = 8.7$  Hz), 7.00 (d, 4H,  $J = 8.7$  Hz), 6.64 (s, 2H), 5.37 (s, 4H), 1.31 (s, 18H) ppm.  $^{13}\text{C}$  NMR (75.5 MHz,  $\text{CDCl}_3$ )  $\delta_{\text{C}}$  168.9 (Cq), 160.8 (Cq), 155.5 (Cq), 144.7 (Cq), 130.1 (Cq), 126.5 (CH), 125.9 (CH), 125.5 (Cq), 114.4 (CH), 106.8 (CH), 61.7 ( $\text{CH}_2$ ), 34.1 (Cq), 31.5 ( $\text{CH}_3$ ) ppm. FABMS  $m/z$  637 ( $\text{M}+\text{H}^+$ ), 636 ( $\text{M}^+$ ); HRMS calcd for  $\text{C}_{42}\text{H}_{40}\text{O}_4\text{N}_2$  636.2988; found 636.2994.

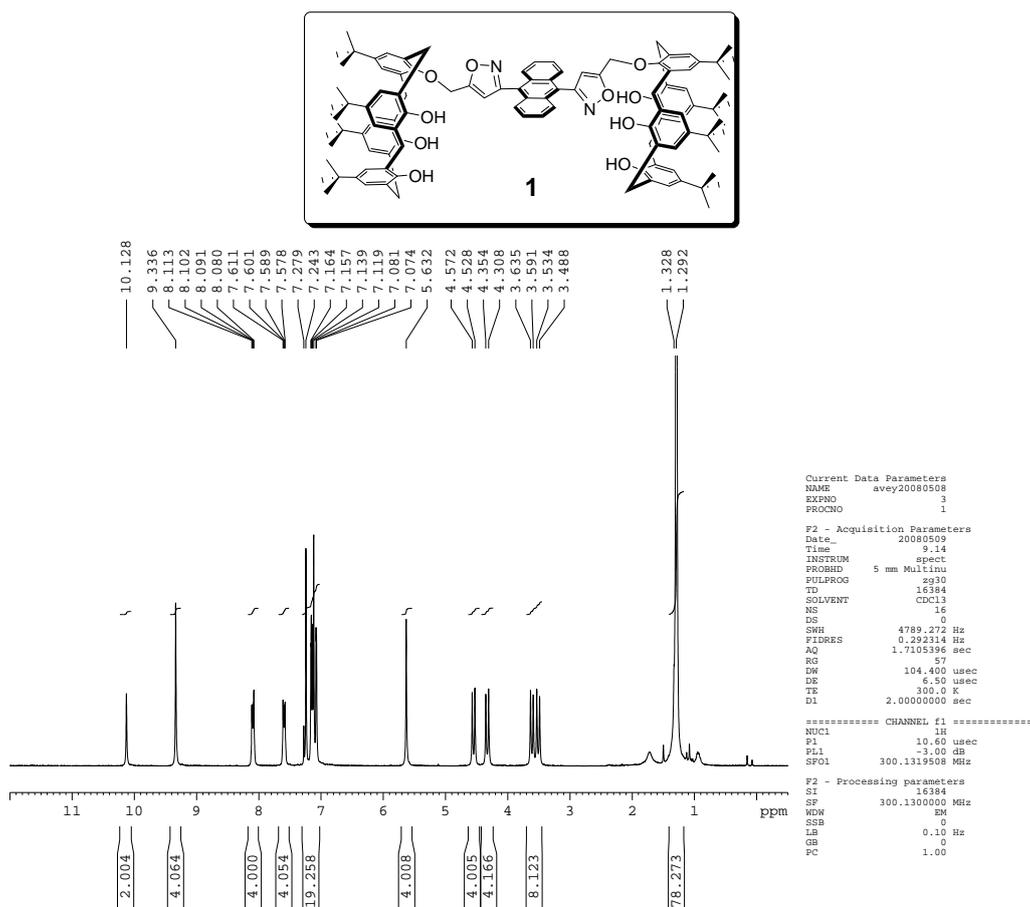


Figure S5. <sup>1</sup>H NMR (300 MHz) spectrum of **1** in CDCl<sub>3</sub>.

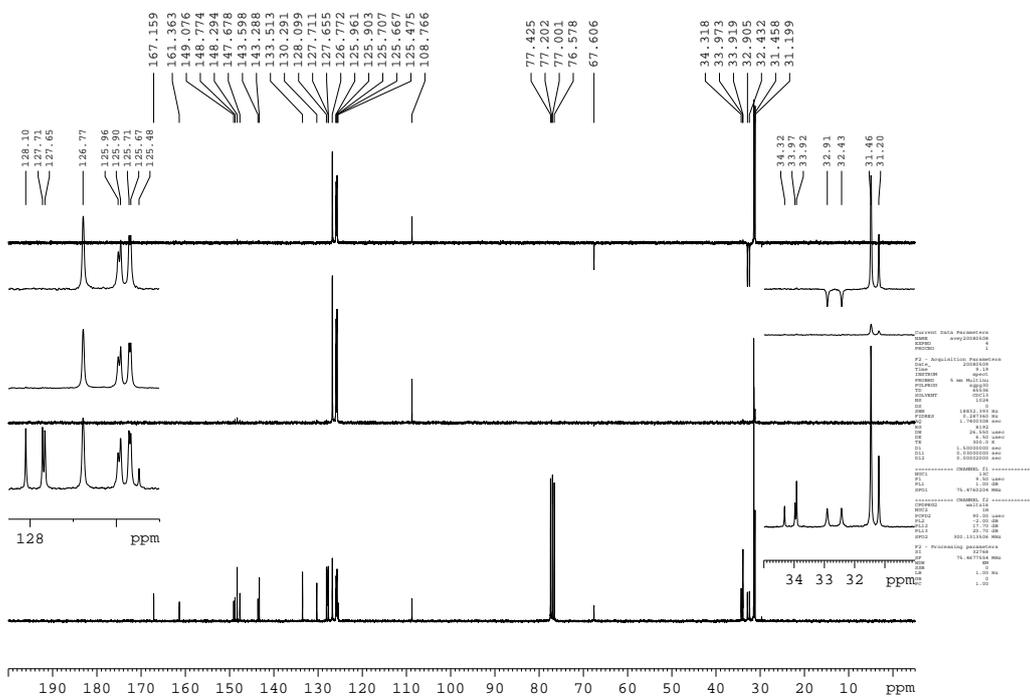


Figure S6. <sup>13</sup>C NMR (75.5 MHz) spectra of **1** in CDCl<sub>3</sub>.

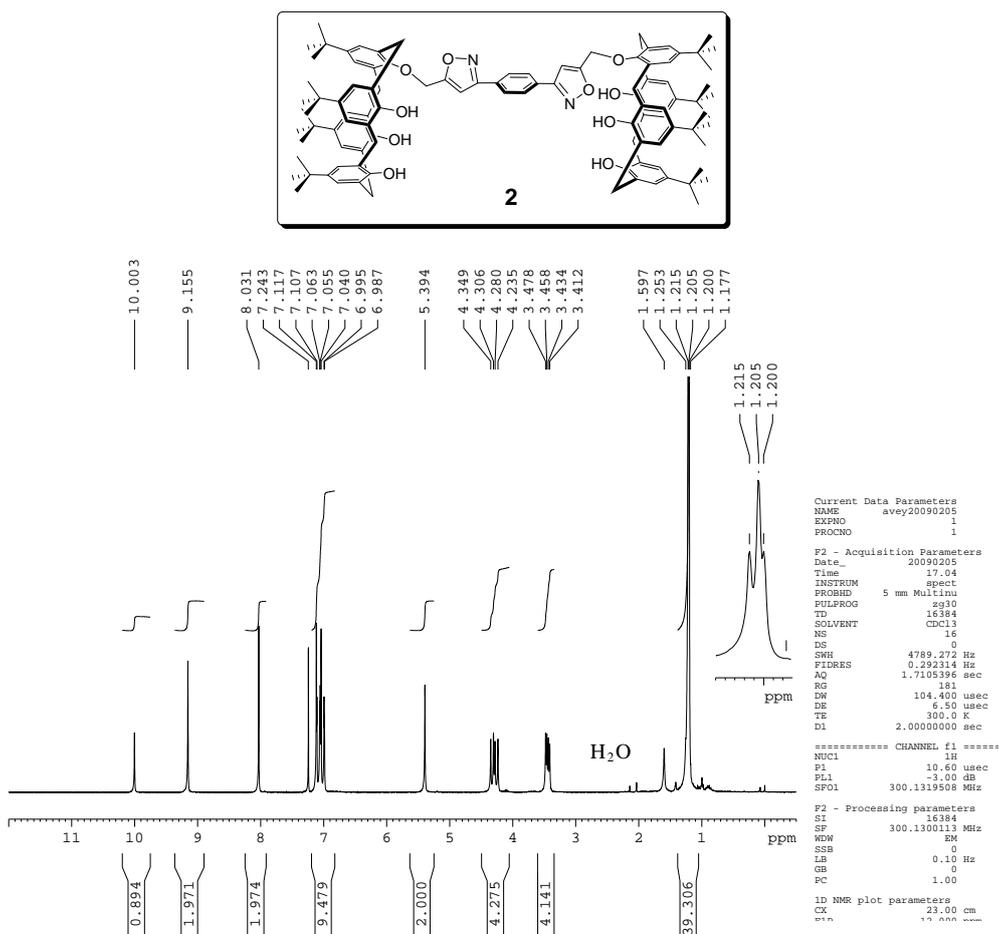


Figure S7. <sup>1</sup>H NMR (300 MHz) spectrum of **2** in CDCl<sub>3</sub>.

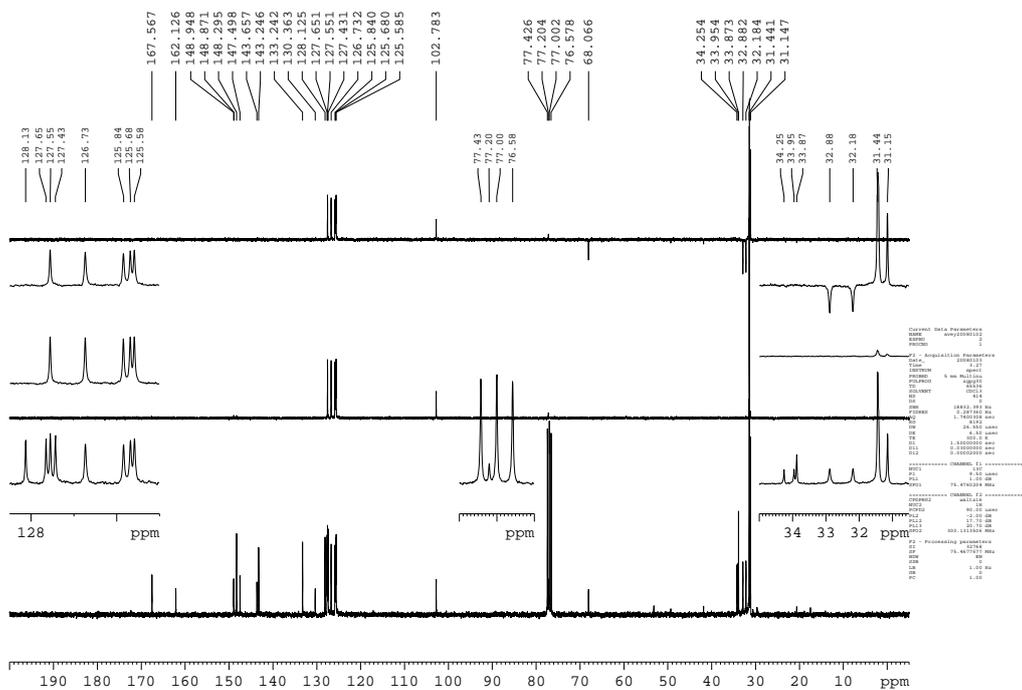


Figure S8. <sup>13</sup>C NMR (75.5 MHz) spectra of **2** in CDCl<sub>3</sub>.

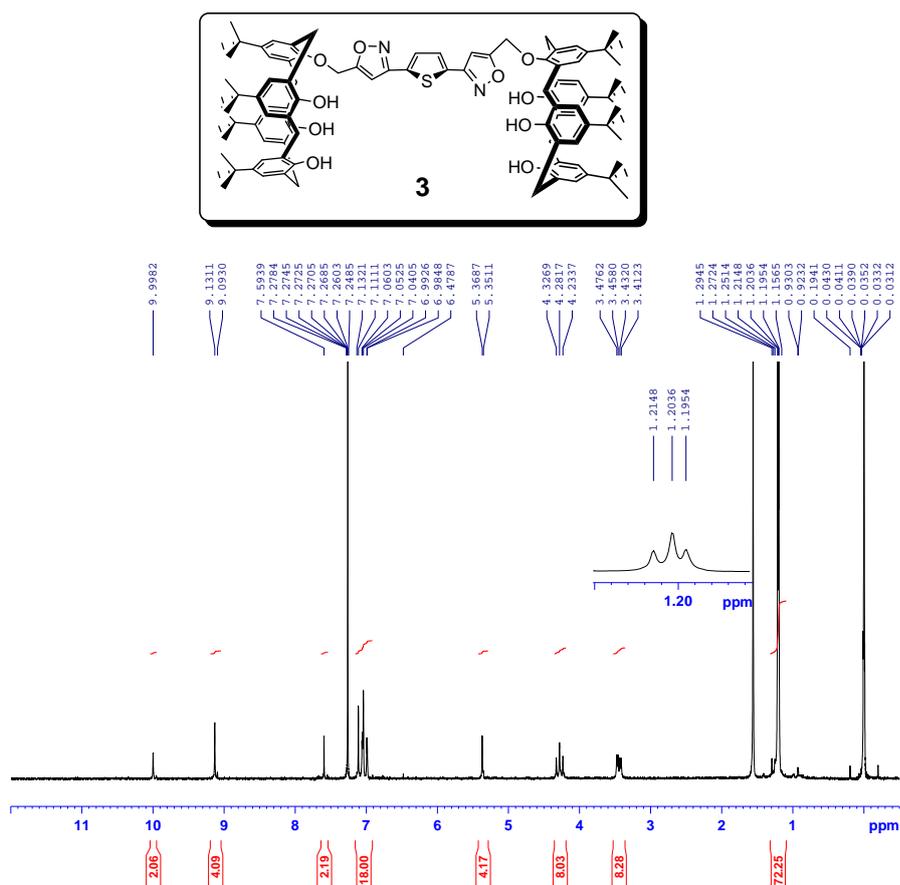


Figure S9. <sup>1</sup>H NMR (300 MHz) spectrum of **3** in CDCl<sub>3</sub>.

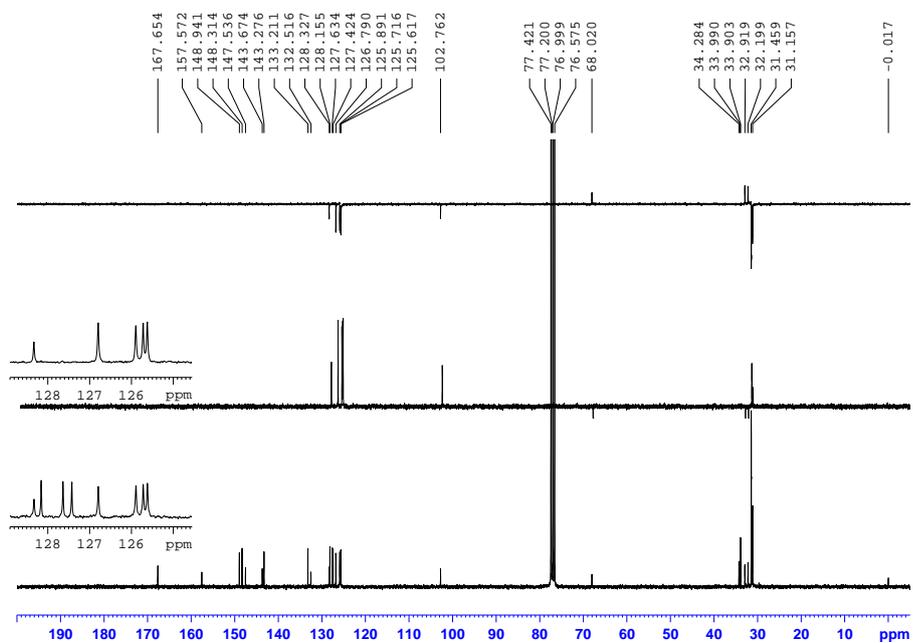


Figure S10. <sup>13</sup>C NMR (75.5 MHz) spectra of **3** in CDCl<sub>3</sub>.

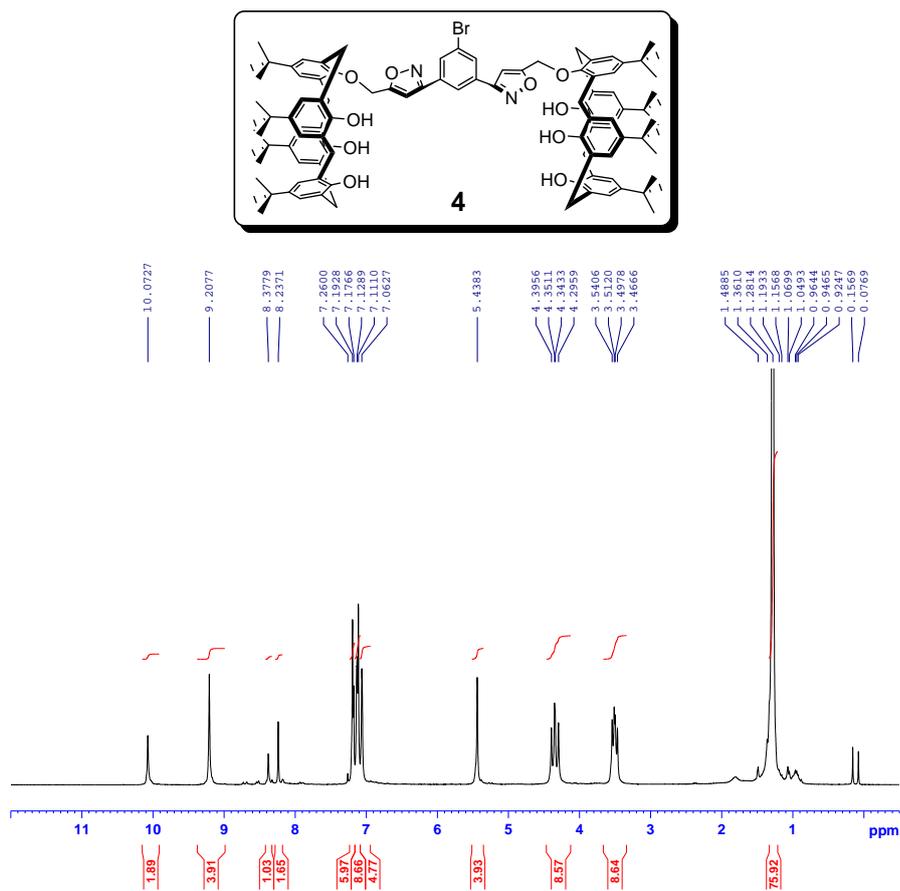


Figure S11. <sup>1</sup>H NMR (300 MHz) spectrum of **4** in CDCl<sub>3</sub>.

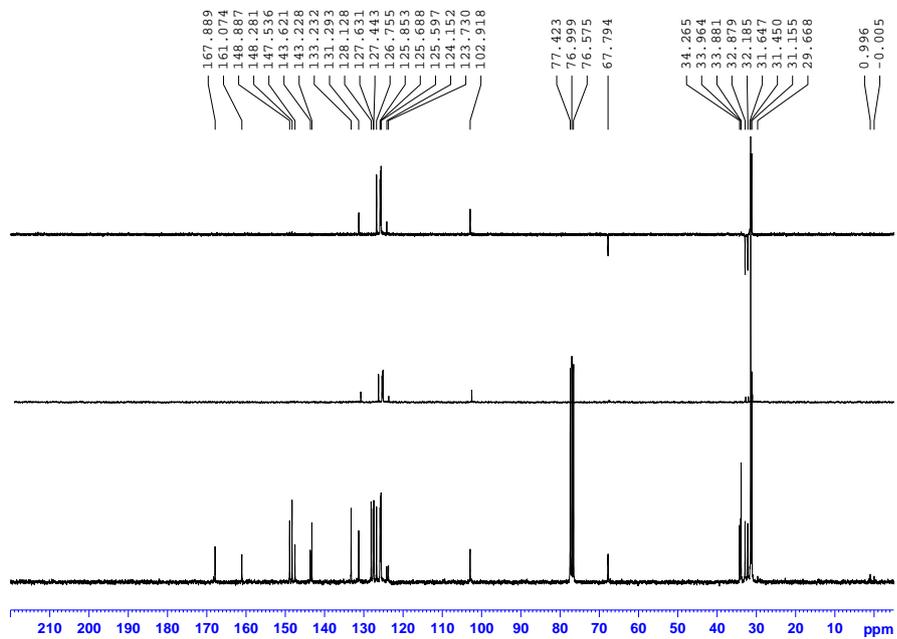


Figure S12. <sup>13</sup>C NMR (75.5 MHz) spectra of **4** in CDCl<sub>3</sub>.

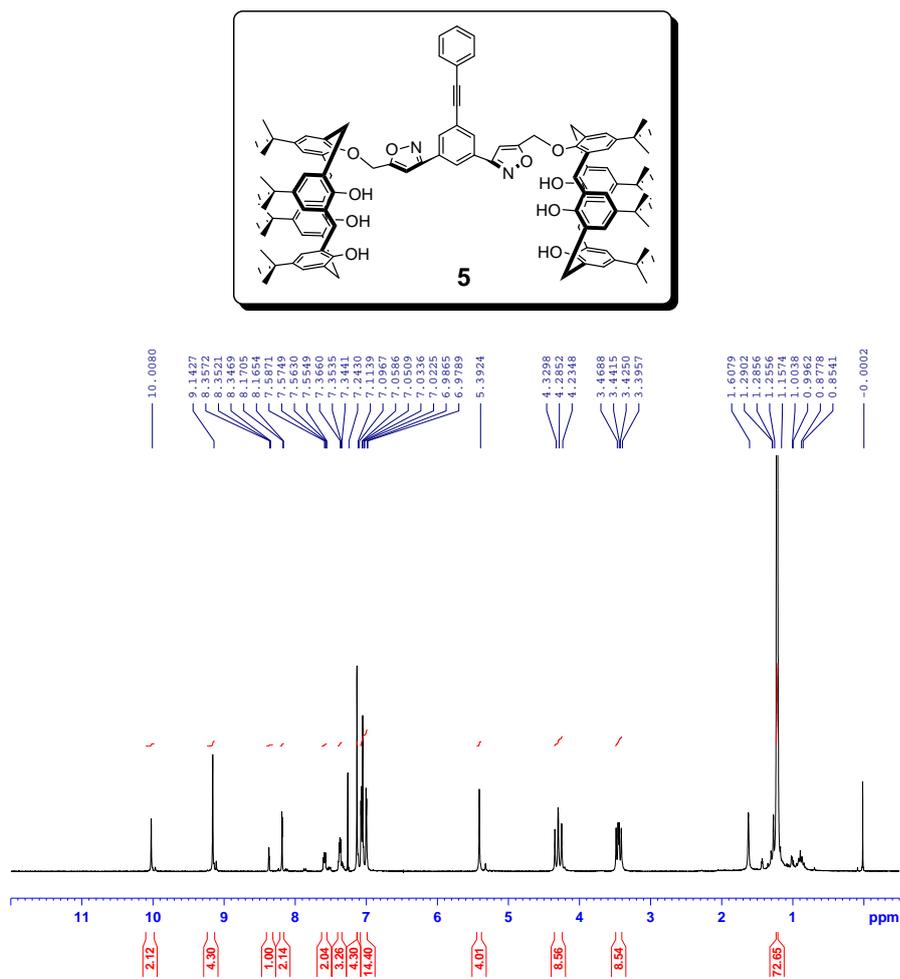


Figure S13.  $^1\text{H NMR}$  (300 MHz) spectrum of **5** in  $\text{CDCl}_3$ .

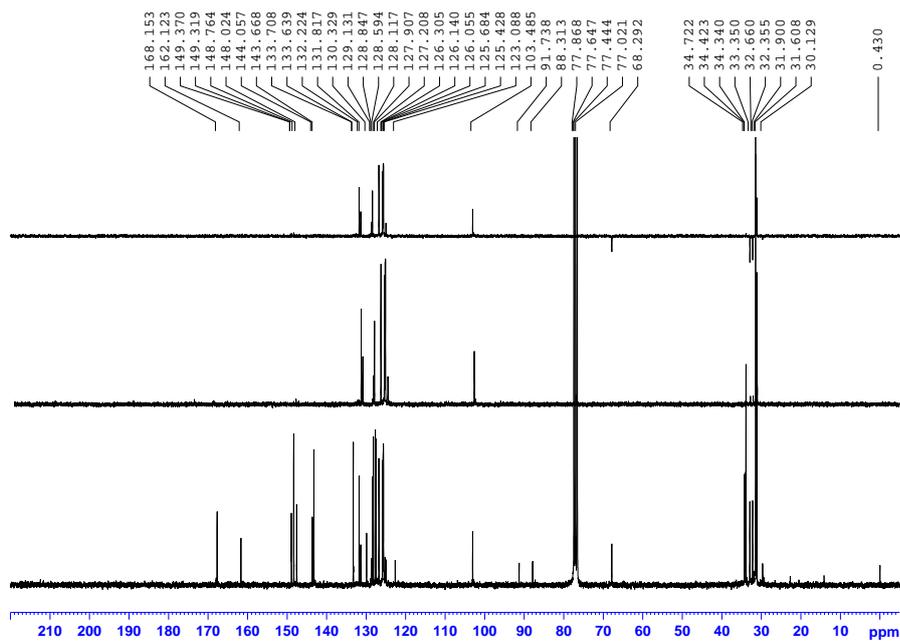


Figure S14.  $^{13}\text{C NMR}$  (75.5 MHz) spectra of **5** in  $\text{CDCl}_3$ .

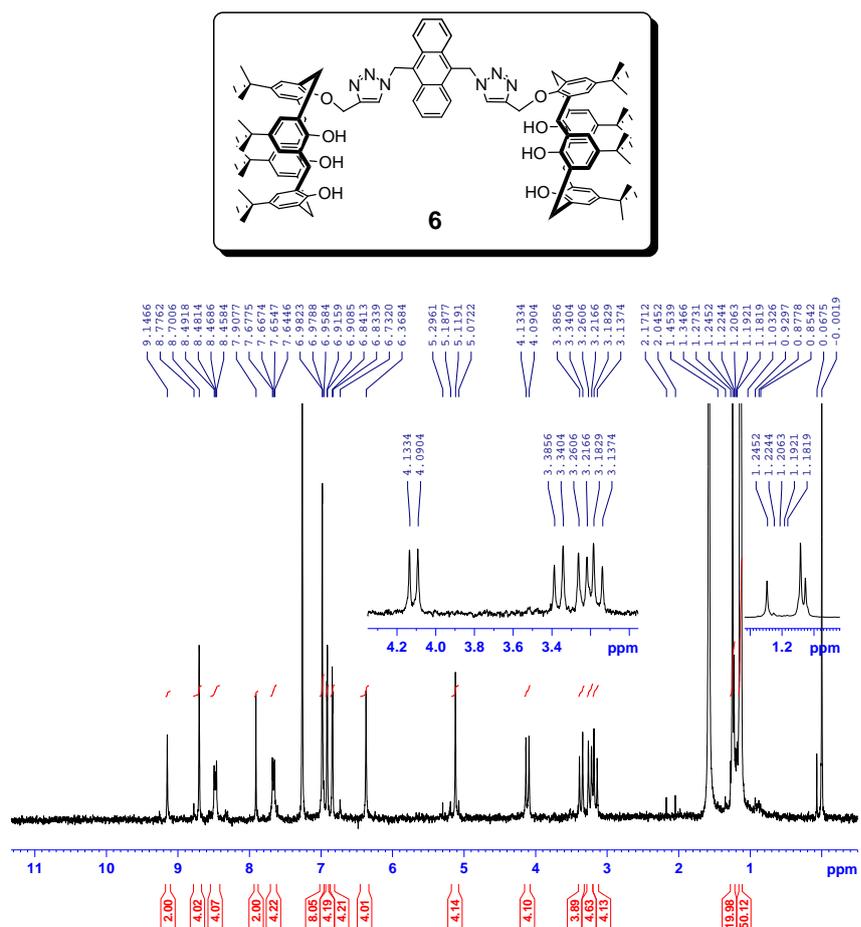


Figure S15. <sup>1</sup>H NMR (300 MHz) spectrum of 6 in CDCl<sub>3</sub>.

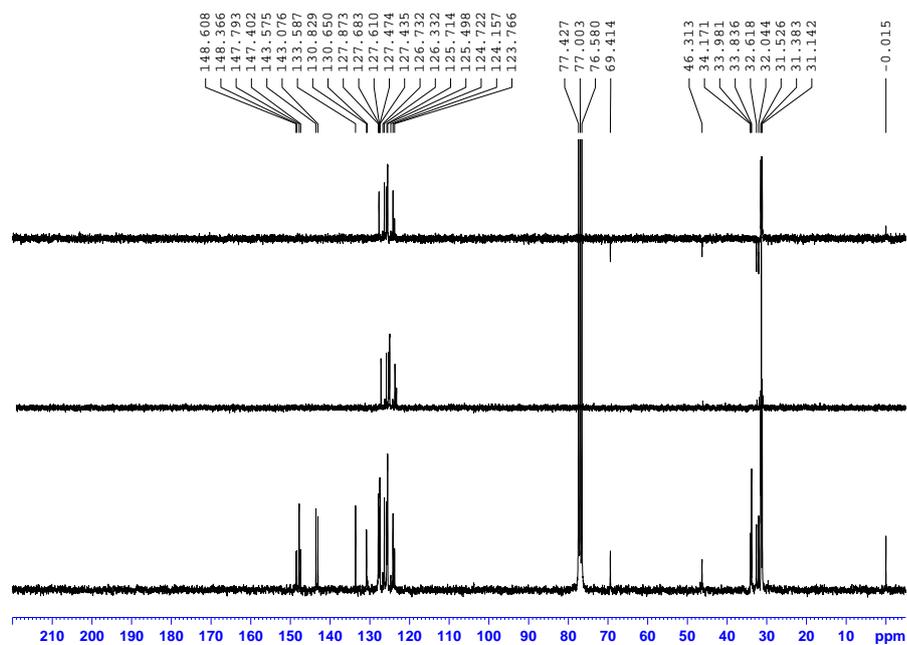


Figure S16. <sup>13</sup>C NMR (75.5 MHz) spectra of 6 in CDCl<sub>3</sub>.

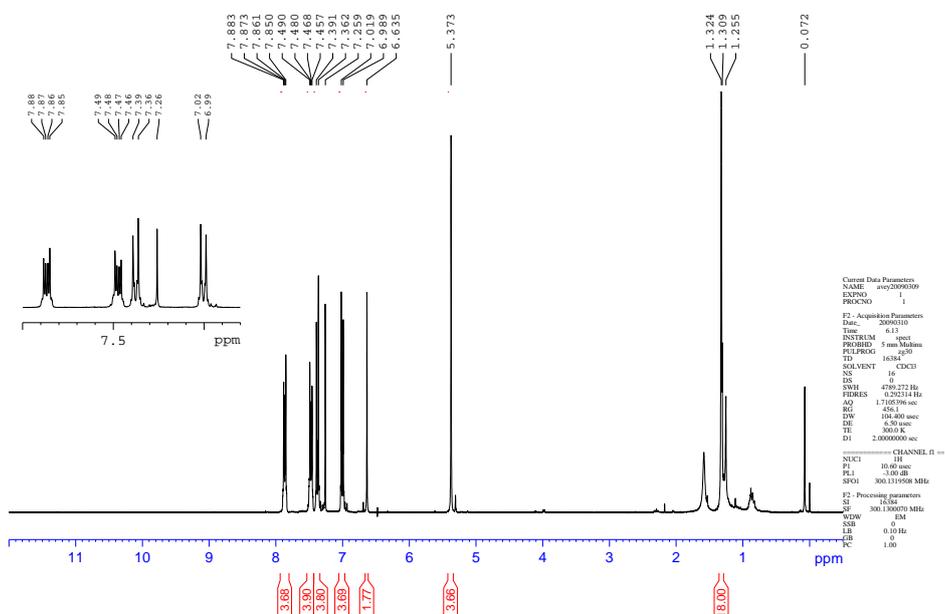
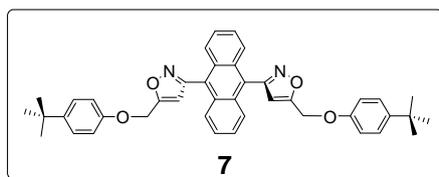


Figure S17.  $^1\text{H}$  NMR (300 MHz) spectrum of **7** in  $\text{CDCl}_3$ .

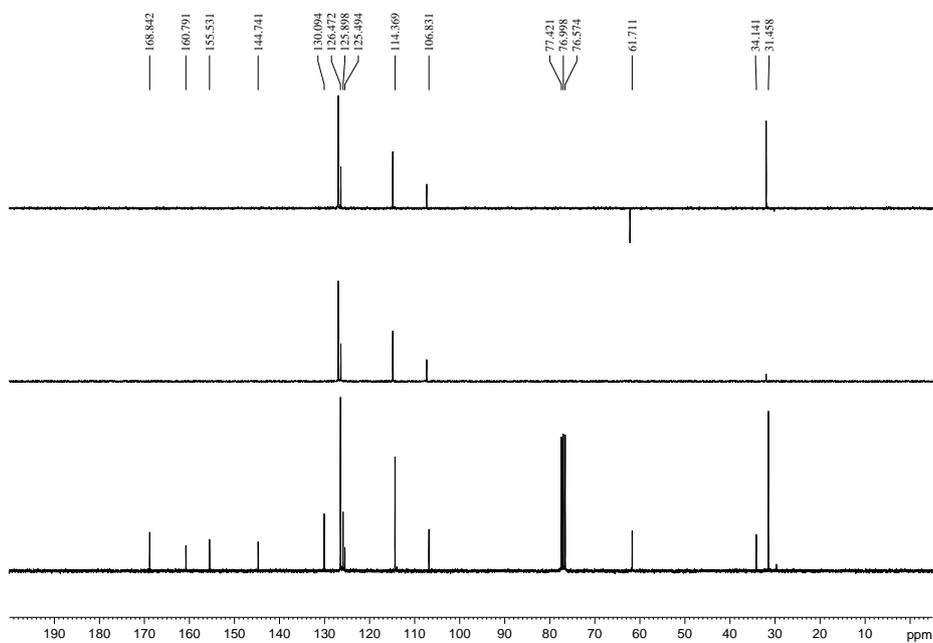


Figure S18.  $^{13}\text{C}$  NMR (75.5 MHz) spectra of **7** in  $\text{CDCl}_3$ .

**Table S1.** Gelation properties of compounds **1–7** in different solvents.<sup>a</sup>

solvent	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>	<b>7</b>
CH <sub>2</sub> Cl <sub>2</sub>	S	S	S	S	S	S	S
CHCl <sub>3</sub>	S	S	S	S	S	S	S
DMSO	S	S	S	S	S	S	S
DMF	S	S	S	S	S	S	S
THF	S	S	S	S	S	S	S
EA	S	S	S	S	S	S	S
toluene	S	S	S	S	S	S	S
acetone	S	S	S	S	S	S	S
CH <sub>3</sub> CN	G (5.0)	S	I	I	I	S	I
MeOH	I	I	I	I	I	I	I
EtOH	I	I	I	I	I	I	I
<i>n</i> -propanol	I	I	I	I	I	I	I
isopropanol	I	I	I	I	I	I	I
<i>n</i> -butanol	I	I	I	I	I	I	I
<i>t</i> -butanol	I	I	I	I	I	I	I
cyclohexane	I	I	I	I	I	I	I
hexane	I	I	I	I	I	I	I

<sup>a</sup> G = gel, I = insoluble, and S = solution. The minimum gelation concentration (mg/mL) is included in the parentheses.