

## **Slick Synthetic Approach to Various Fluoroalkyl Silsesquioxanes - - Assessment of their Dielectric Properties**

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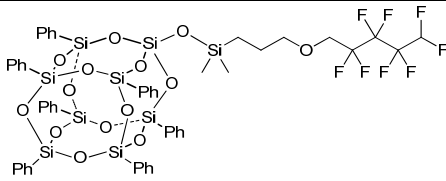
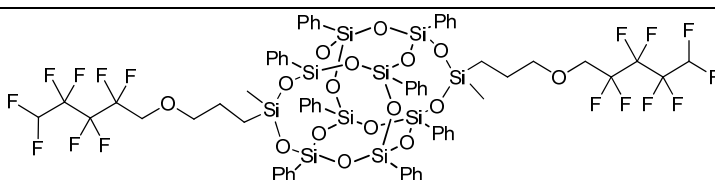
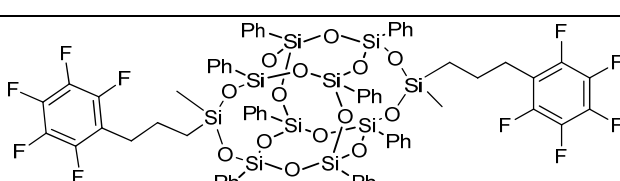
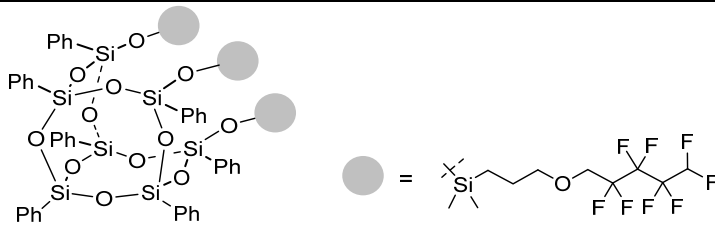
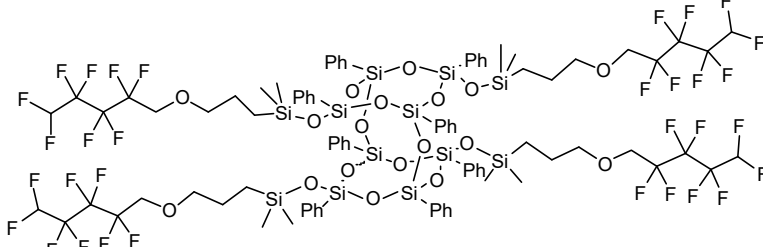
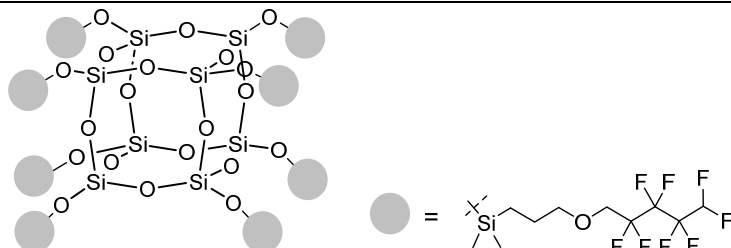
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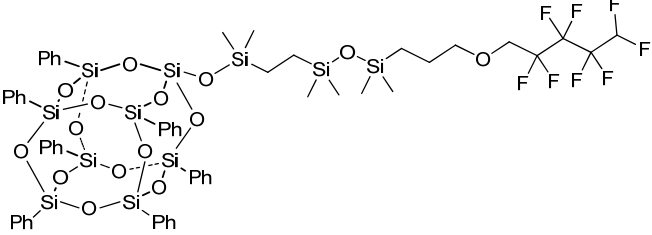
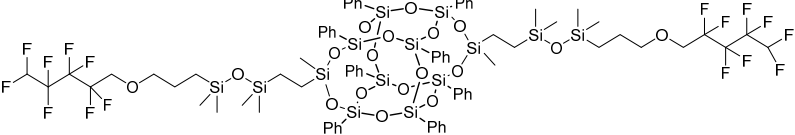
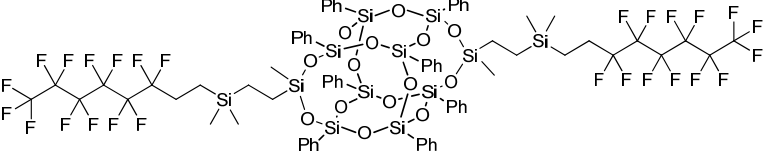
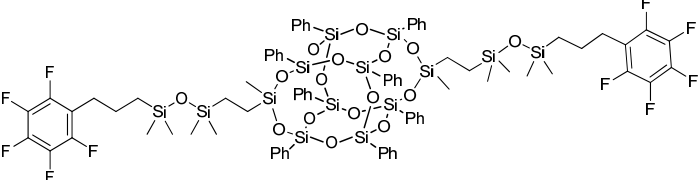
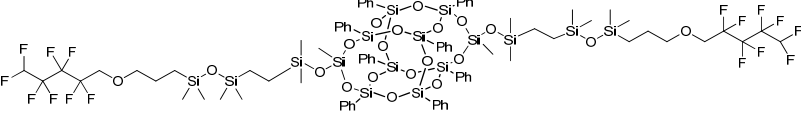
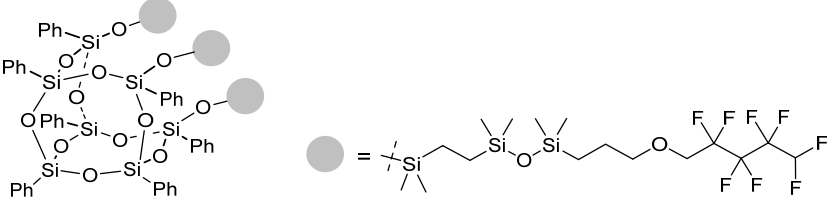
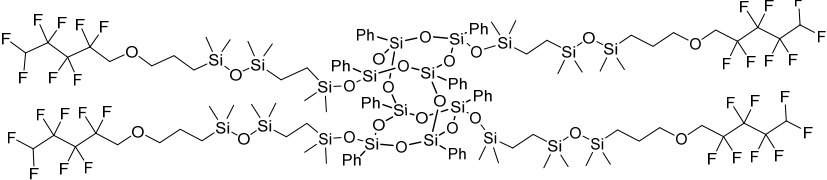
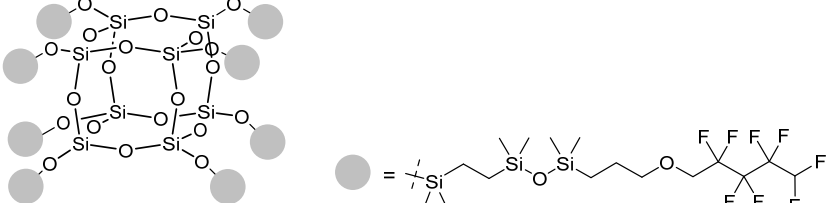
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**1. Table S1. The list of isolated fluorinated silsesquioxanes**

Structure	Compound Abbrev.	NMR spectra page:
	<b>monoT<sub>8</sub>(F1)</b>	4-5
	<b>DDSQ-2Si(F1)</b>	6-7
	<b>DDSQ-2Si(F2)</b>	8-9
	<b>triT<sub>7</sub>(F1)</b>	10-11
	<b>DDSQ-4Si(F1)</b>	12-13
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	<b>monoT<sub>8</sub>(F<sub>3</sub>)</b>	16-17
	<b>DDSQ-2Si(F<sub>3</sub>)</b>	18-19
	<b>DDSQ-2Si(F<sub>4</sub>)</b>	20-21
	<b>DDSQ-2Si(F<sub>5</sub>)</b>	22-23
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	<b>triT<sub>7</sub>(F<sub>3</sub>)</b>	26-27
	<b>DDSQ-4Si(F<sub>3</sub>)</b>	28-30
	<b>octaT<sub>8</sub>(F<sub>3</sub>)</b>	31-32



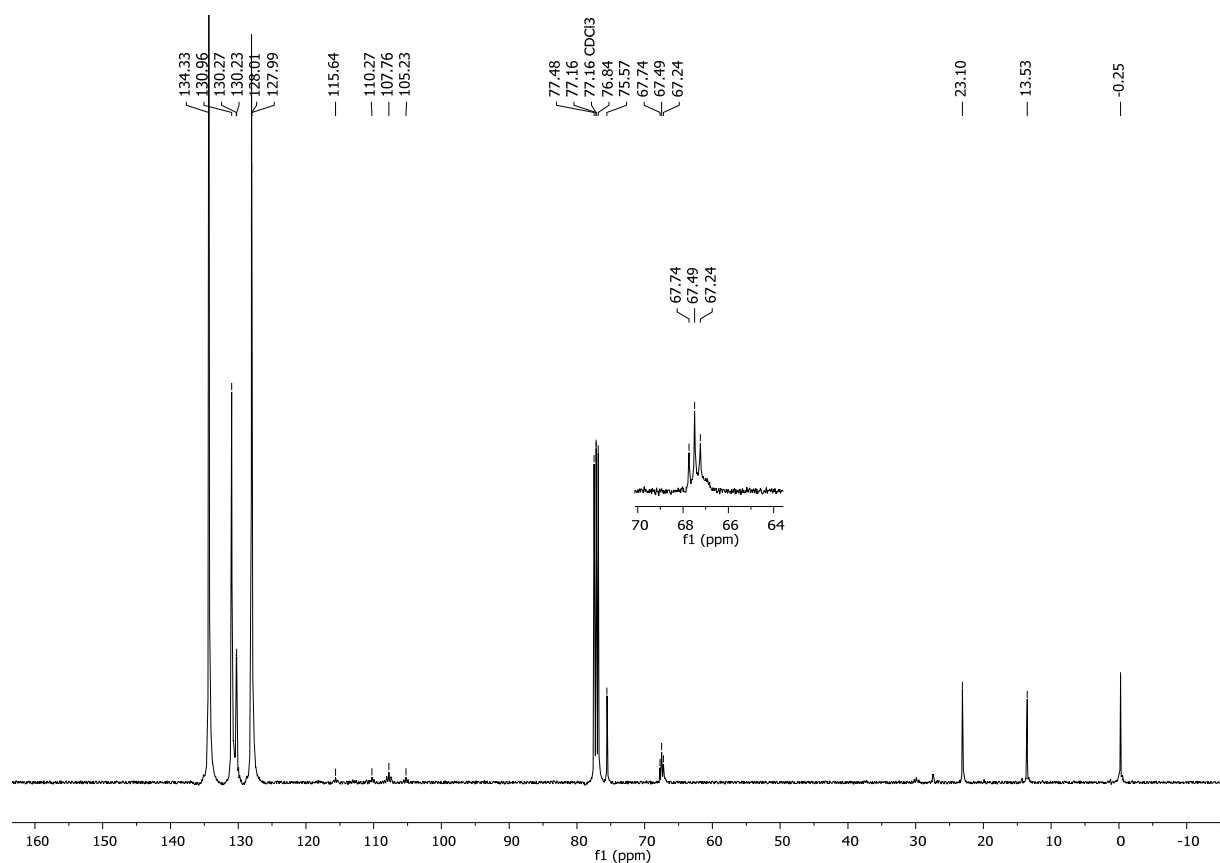


Figure S 2. <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>) spectrum of **monoT<sub>8</sub>(F1)**.

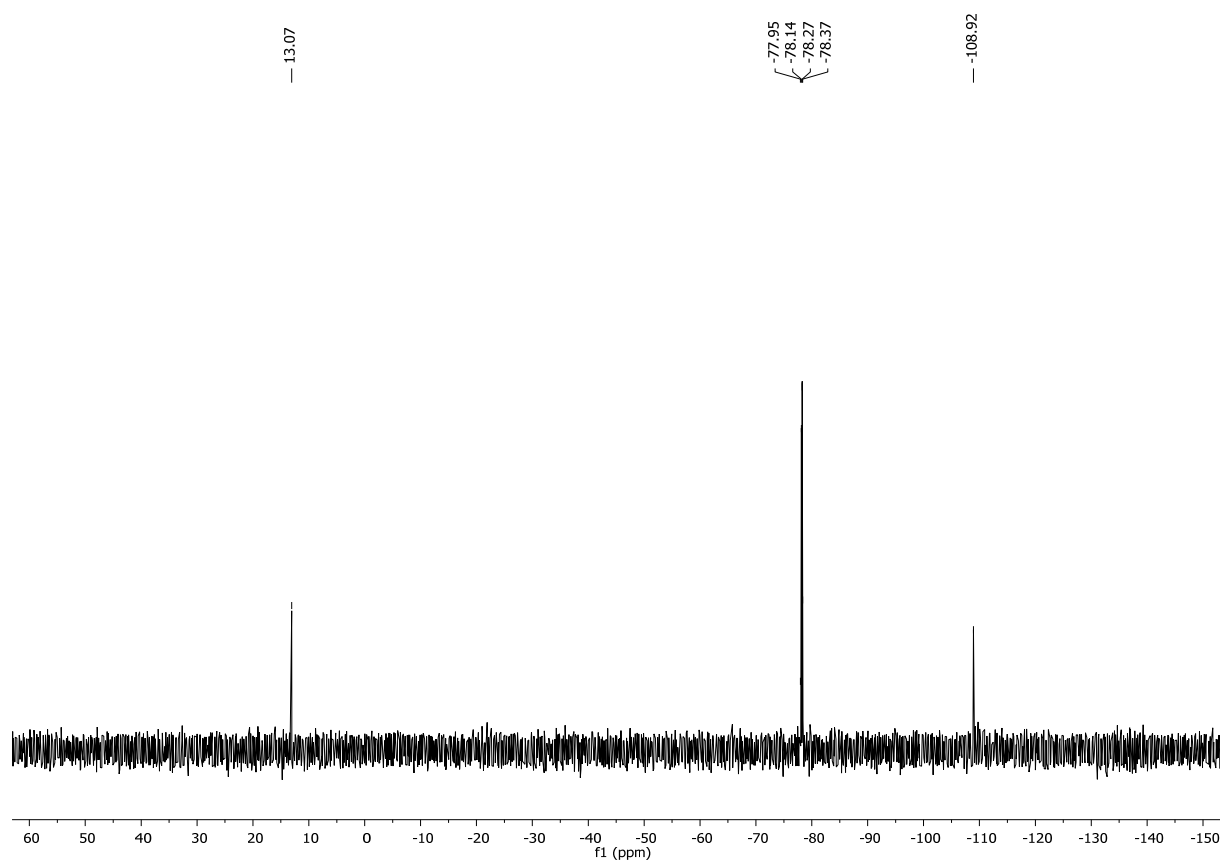
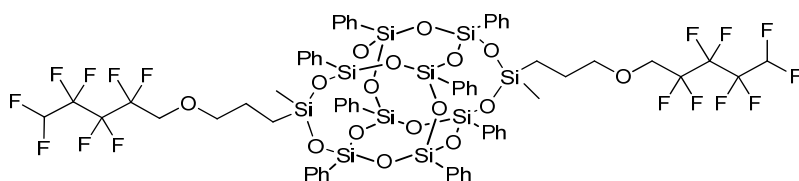


Figure S 3. <sup>29</sup>Si NMR (79.5 MHz, CDCl<sub>3</sub>) spectrum of **monoT<sub>8</sub>(F1)**.

## DDSQ-2Si(F1)



White solid. Isolated Yield 96%

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = 0.31 (s, 6H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.71-0.76 (m, 4H, Si-CH<sub>2</sub>-), 1.68 (dd, 4H, -CH<sub>2</sub>-), 3.41 (t,  $J_{H-H}$  = 6.7 Hz, 4H, -CH<sub>2</sub>-O), 3.64 (t,  $J_{H-H}$  = 14.0 Hz, 4H, -O-CH<sub>2</sub>-), 5.98 (tt,  $J_{H-H}$  = 52.0, 5.6 Hz, 2H, -CH-F<sub>2</sub>), 7.18-7.54 (m, 40H, Ph);

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = -0.82 (SiCH<sub>3</sub>), 12.65 (Si-CH<sub>2</sub>-), 22.89 (-CH<sub>2</sub>-), 67.17-67.68 (-CH<sub>2</sub>-O-), 75.22 (O-CH<sub>2</sub>-), 105.26 (-CF<sub>2</sub>-), 107.77 (-CF<sub>2</sub>-), 110.29 (-CHF<sub>2</sub>-), 115.61 (-CF<sub>2</sub>-), 127.85-128.00 (Ph), 130.61 (Ph), 131.11 (Ph), 131.11 (Ph), 132.03 (Ph), 134.04-134.17 (Ph);

**<sup>29</sup>Si NMR** (79 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = -17.64 (-Si-CH<sub>3</sub>-), -78.57, -79.43, -79.53, -79.62 (-Si-Ph);

**IR** (ATR, cm<sup>-1</sup>): 3073.45 (C-H phenyl), 2923.95 (C-H), 1594.03, 1430.90 (C=C phenyl), 1262.16 (Si-C), 1174.21 (C-F), 1076.40, 1028.14 (Si-O-Si), 997.48 (C-H phenyl).

**EA:** Anal. calcd for C<sub>66</sub>H<sub>64</sub>F<sub>16</sub>O<sub>16</sub>Si<sub>10</sub> (%): C, 46.68; H, 3.80; found: C, 46.80; H, 3.81.

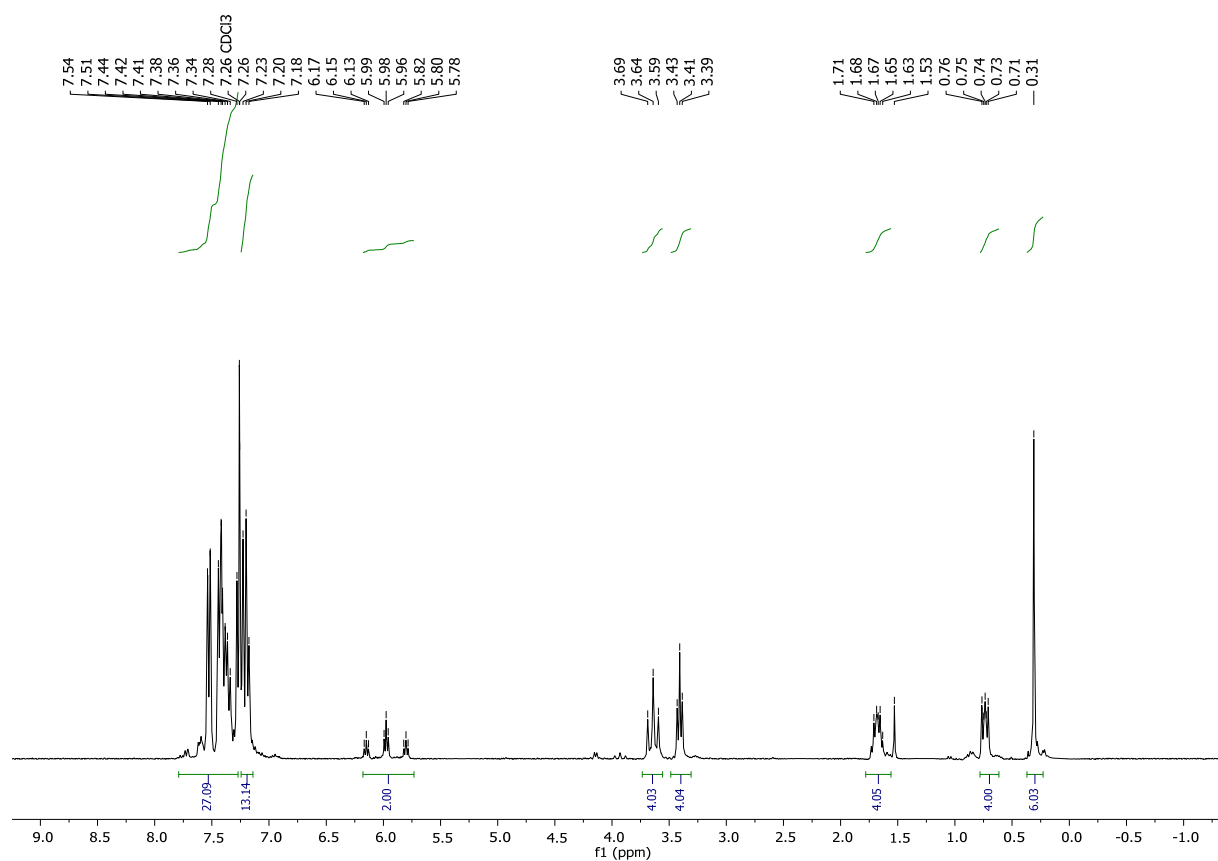


Figure S 4. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) spectrum of DDSQ-2Si(F1).

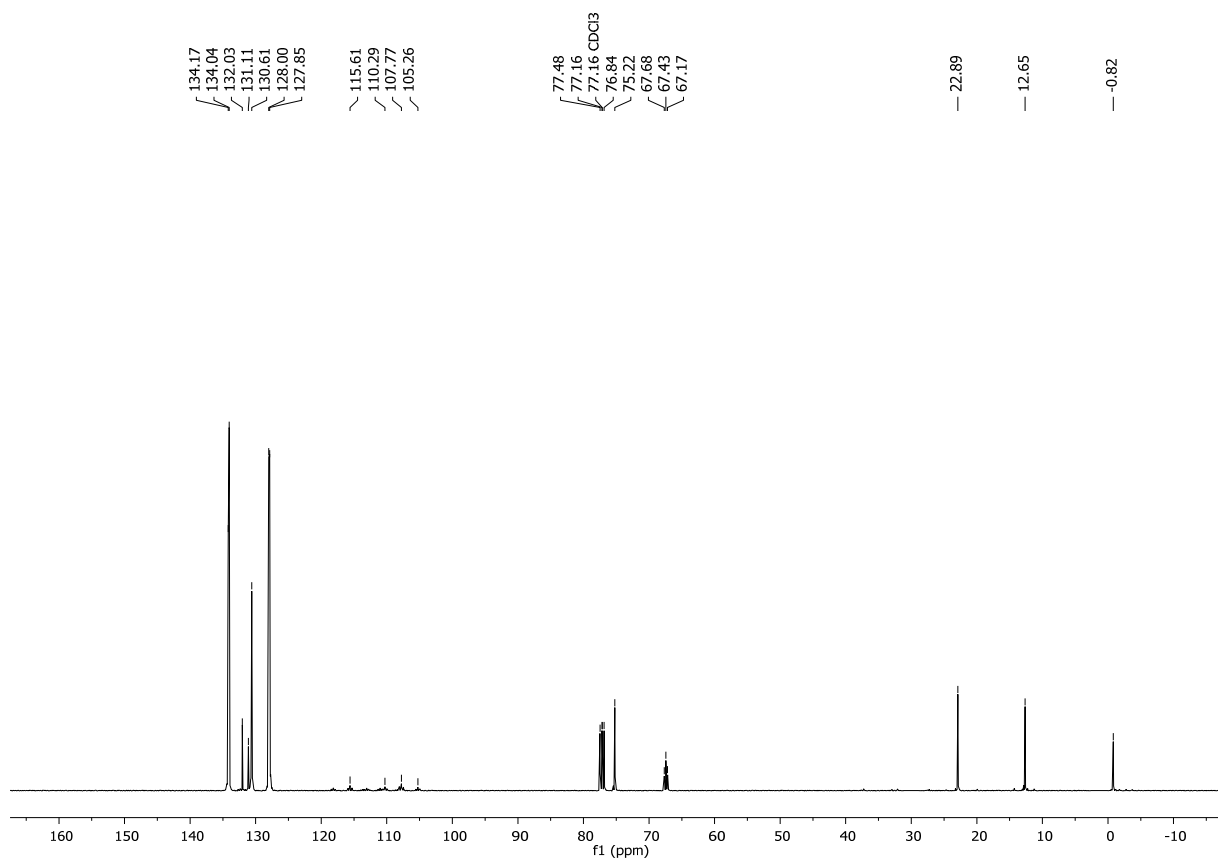


Figure S 5.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectrum of **DDSQ-2Si(F1)**.

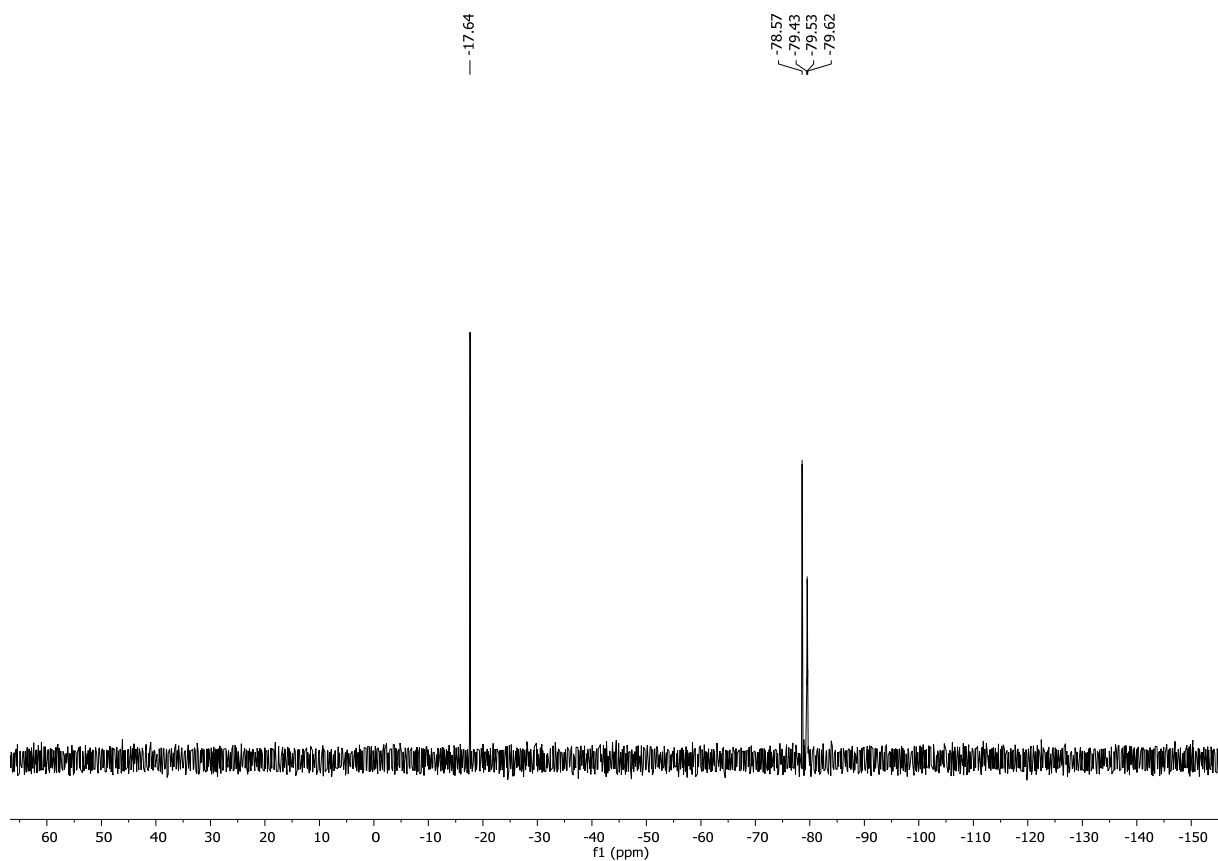
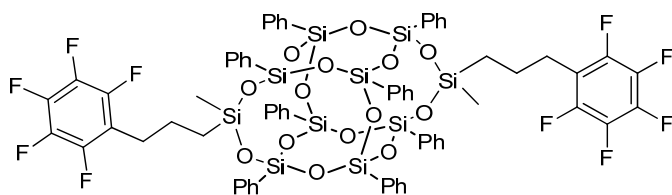


Figure S 6.  $^{29}\text{Si}$  NMR (79.5 MHz,  $\text{CDCl}_3$ ) spectrum of **DDSQ-2Si(F1)**.

## DDSQ-2Si(F2)



White solid. Isolated Yield 93%

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = 0.31 (s, 6H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.74-0.78 (m, 4H, Si-CH<sub>2</sub>-), 1.68 (td,  $J_{H-H}$  = 8.1, 7.3, 3.4 Hz 4H, -CH<sub>2</sub>-), 2.61 (t,  $J_{H-H}$  = 7.7 Hz, 4H, -CH<sub>2</sub>-), 7.20-7.54 (m, 40H, Ph);

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = -0.76 (SiCH<sub>3</sub>), 16.67 (Si-CH<sub>2</sub>-), 23.91 (-CH<sub>2</sub>-), 25.56 (-CH<sub>2</sub>-), 114.92-115.28 (-C-), 127.80-127.96 (Ph), 130.57 (Ph), 131.03 (Ph), 132.00 (Ph), 134.01-134.15 (Ph), 136.15 (-CF-), 138.64 (-CF-), 140.79 (-CF-), 143.84 (-CF-), 146.28 (-CF-);

**<sup>29</sup>Si NMR** (79 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = -18.38 (-Si- (CH<sub>3</sub>)-), -78.44, -79.36, -79.42, -79.47 (-Si-Ph);

**IR** (ATR, cm<sup>-1</sup>): 3072.92, 3051.06 (C-H phenyl), 2930.13 (C-H), 1655.26 (C=C phenyl), 1593.89, 1518.76, 1500.95 (-C-F-), 1429.98 (C=C phenyl), 1261.94 (Si-C), 1167.27 (C-F), 1027.64 (Si-O-Si), 997.19 (C-H phenyl).

**EA:** Anal. calcd for C<sub>68</sub>H<sub>58</sub>F<sub>10</sub>O<sub>14</sub>Si<sub>10</sub> (%): C, 52.02; H, 3.72; found: C, 51.91; H, 3.71.

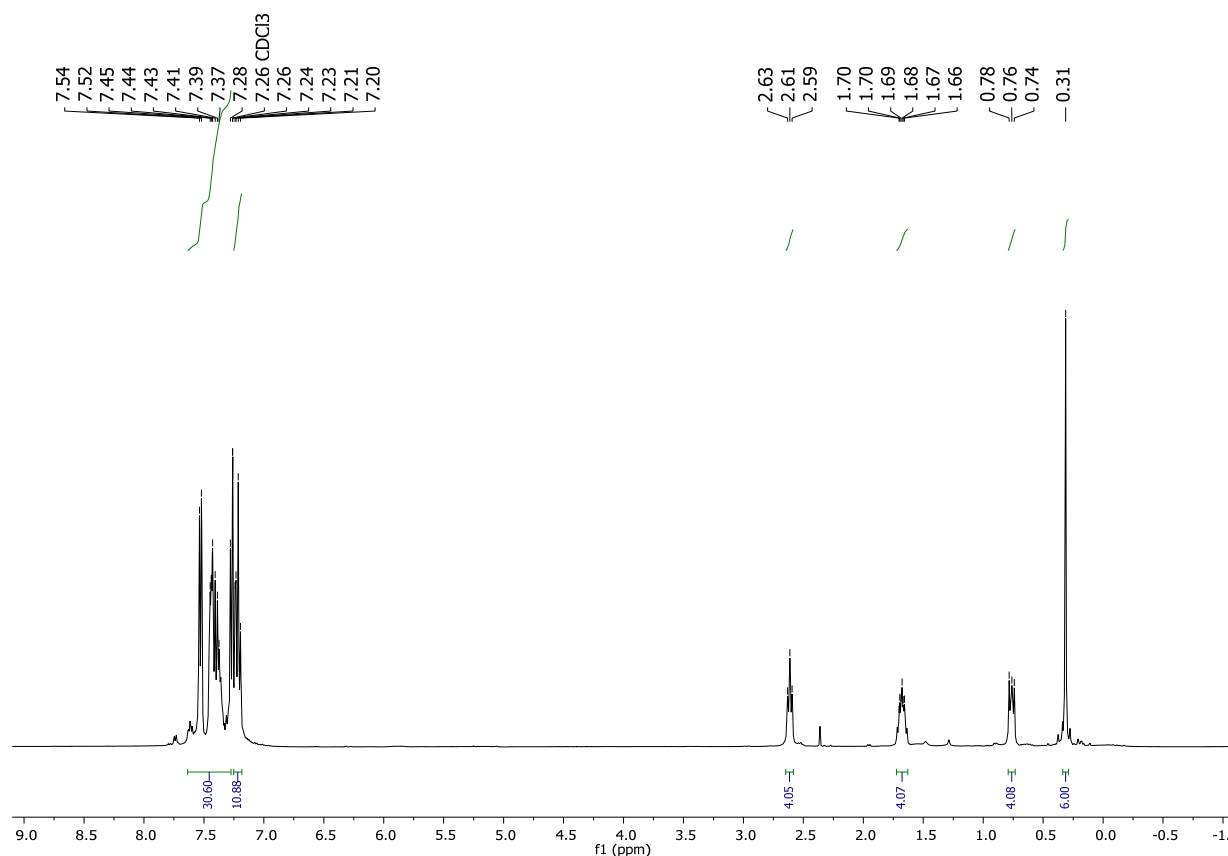


Figure S 7. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) spectrum of DDSQ-2Si(F2).



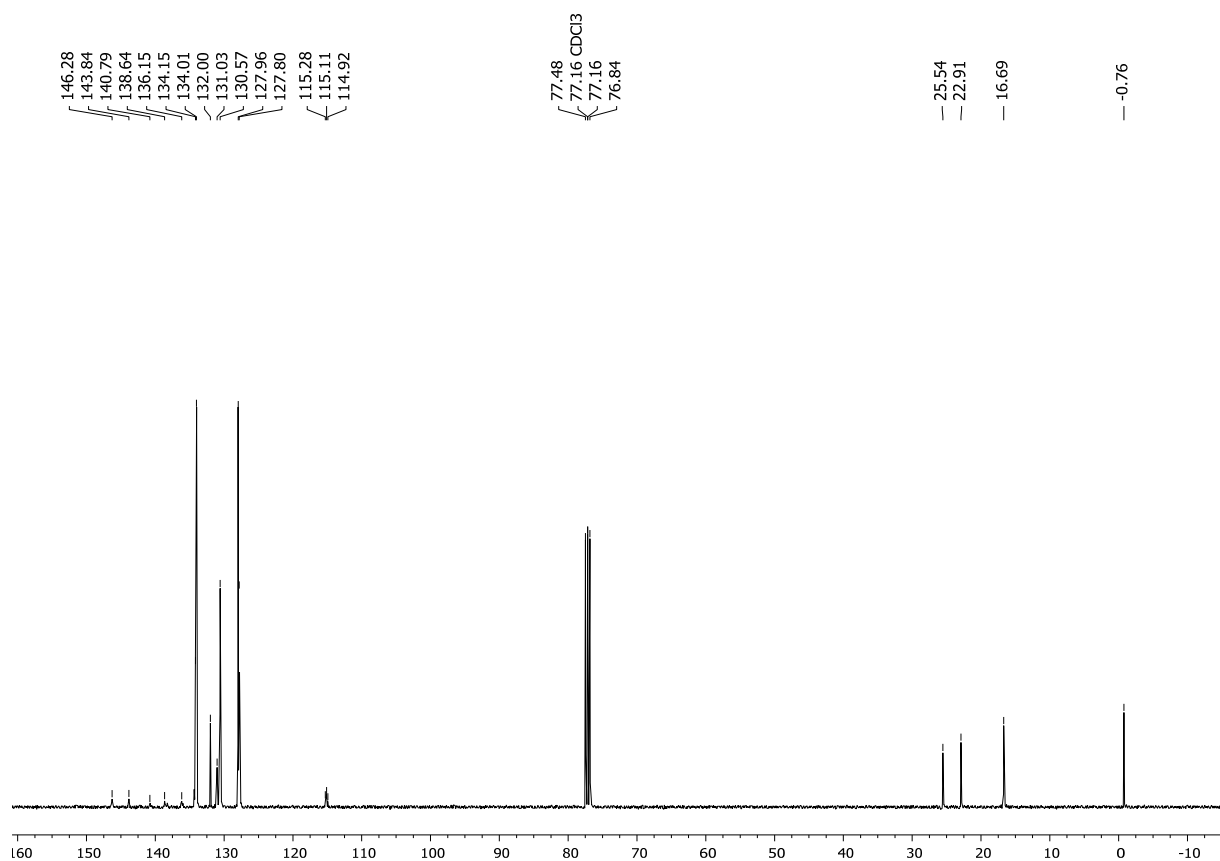


Figure S 8.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectrum of **DDSQ-2Si(F2)**.

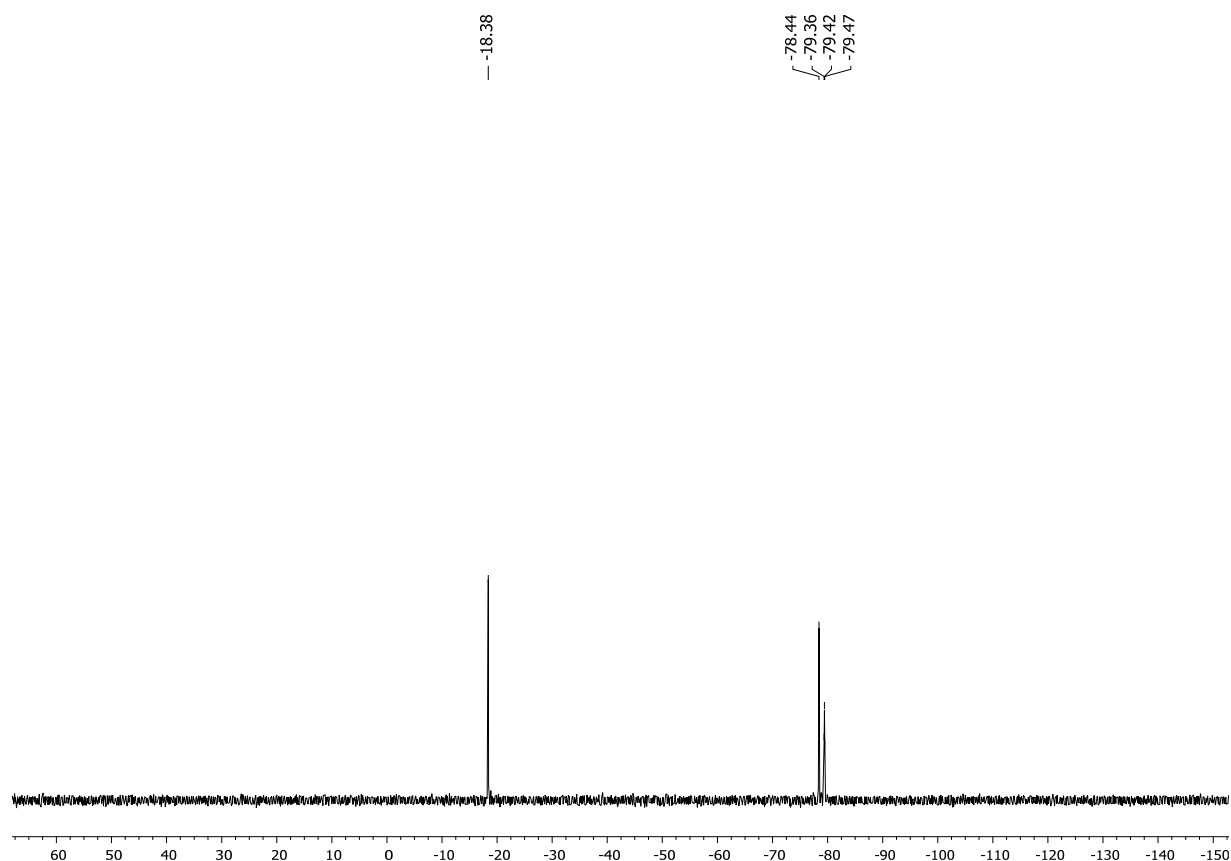
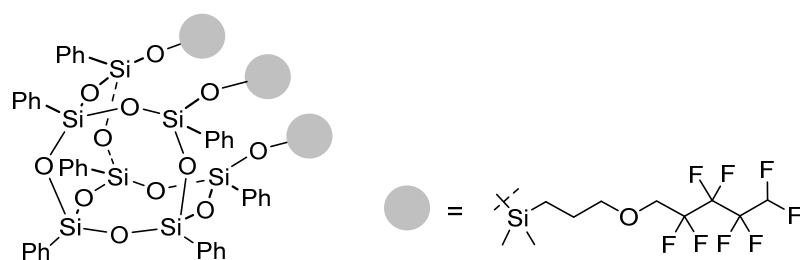


Figure S 9.  $^{29}\text{Si}$  NMR (79.5 MHz,  $\text{CDCl}_3$ ) spectrum of **DDSQ-2Si(F2)**.

**triT<sub>7</sub>(F1)**



Oil. Isolated Yield 94%

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = 0.23 (s, 18H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.56-0.61 (m, 6H, Si-CH<sub>2</sub>-), 1.59 (dq,  $J_{H-H}$  = 12.4, 7.1 Hz, 6H, -CH<sub>2</sub>-), 3.42 (t,  $J_{H-H}$  = 6.9 Hz, 6H, -CH<sub>2</sub>-O), 3.75 (t,  $J_{H-H}$  = 14.0 Hz, 4H, -O-CH<sub>2</sub>-), 6.00 (tt,  $J_{H-H}$  = 52.0, 5.5 Hz, 3H, -CH-F<sub>2</sub>), 7.06-7.52 (m, 40H, Ph);

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = 0.00 (SiCH<sub>3</sub>), 13.46 (Si-CH<sub>2</sub>-), 22.94 (-CH<sub>2</sub>-), 66.81-67.48 (-CH<sub>2</sub>-O-), 75.46 (O-CH<sub>2</sub>-), 104.07 (-CF<sub>2</sub>-), 107.44 (-CF<sub>2</sub>-), 115.34 (-CHF<sub>2</sub>-), 118.68 (-CF<sub>2</sub>-), 127.37-128.73 (Ph), 129.92 (Ph), 130.06 (Ph), 130.79 (Ph), 132.42 (Ph), 133.69-133.77 (Ph);

**<sup>29</sup>Si NMR** (79 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = 11.85 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), -77.36, -77.62, -77.98 (-Si-Ph);

**IR** (ATR, cm<sup>-1</sup>): 3073.78, 3051.97 (C-H phenyl), 2956.36, 2924.18 (C-H), 1594.24, 1430.50 (C=C phenyl), 1254.66 (Si-C), 1168.76 (C-F), 1107.87, 1047.64 (Si-O-Si), 997.68 (C-H phenyl).

**EA:** Anal. calcd for C<sub>72</sub>H<sub>80</sub>F<sub>24</sub>O<sub>15</sub>Si<sub>10</sub> (%): C, 44.99; H, 4.20; found: C, 45.07; H, 4.21.

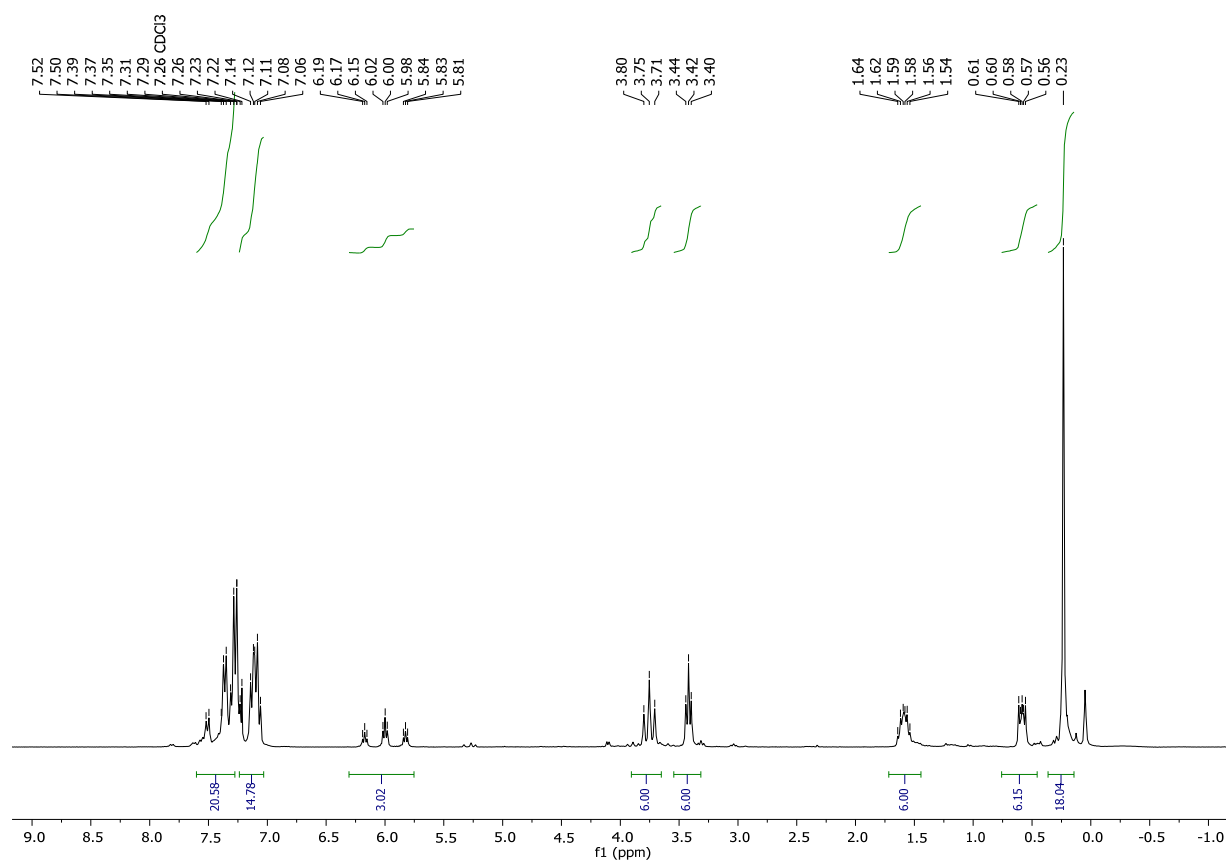


Figure S 10. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) spectrum of **triT<sub>7</sub>(F1)**.

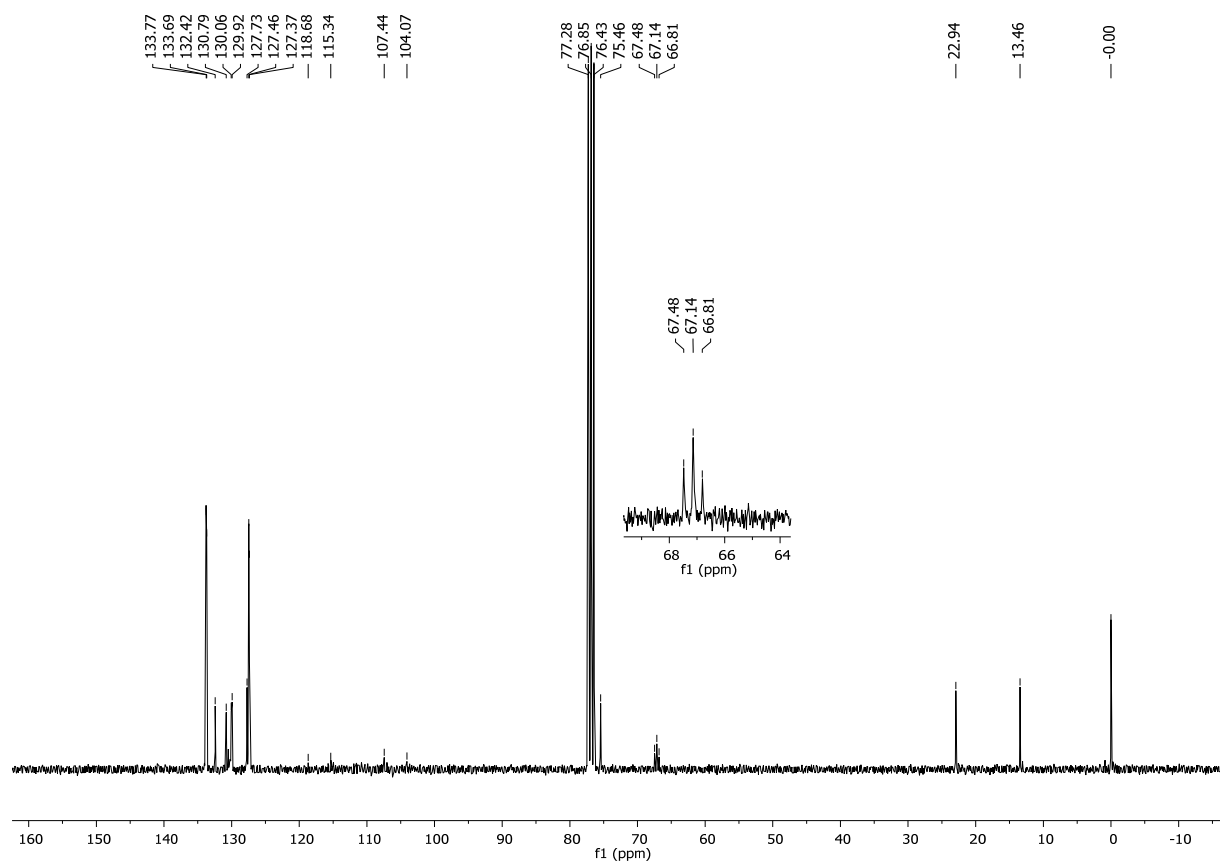


Figure S 11.  $^{13}\text{C}$  NMR (75.5 MHz,  $\text{CDCl}_3$ ) spectrum of **triT<sub>7</sub>(F1)**.

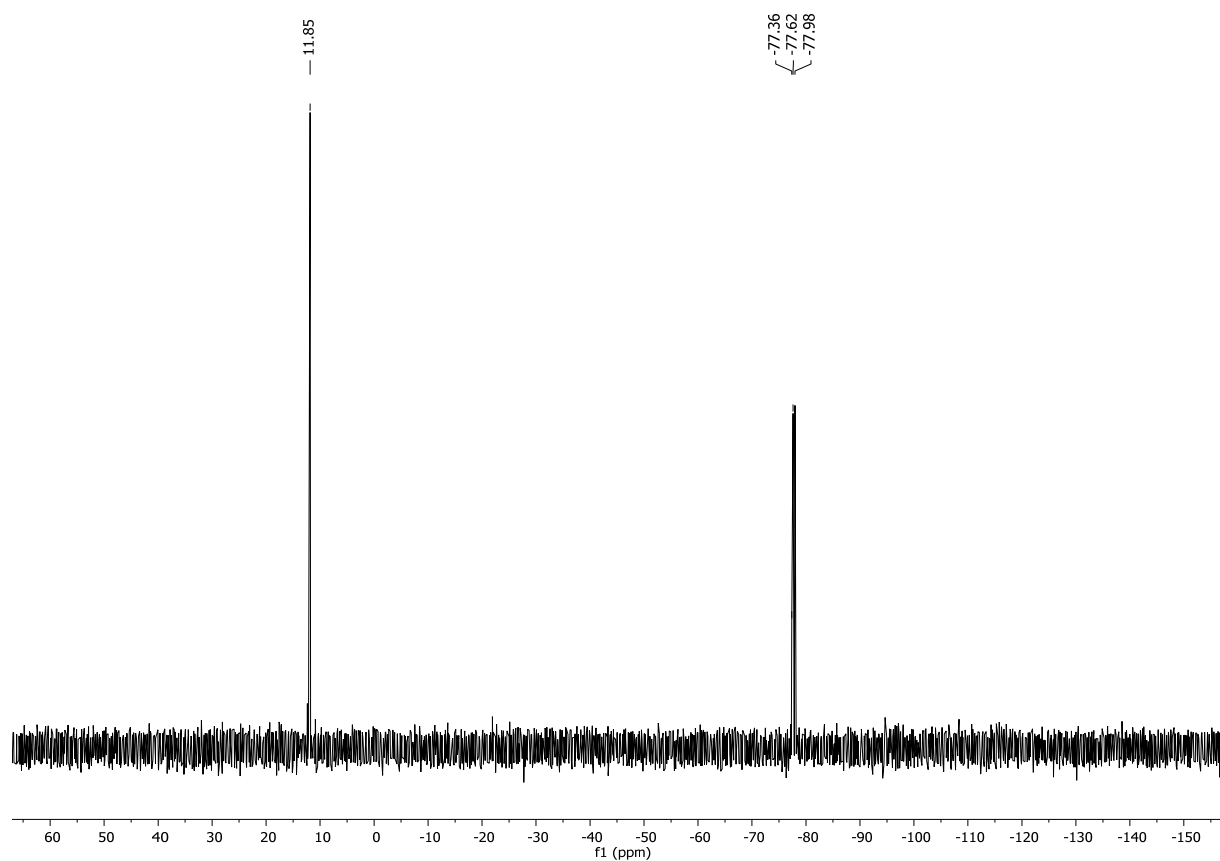
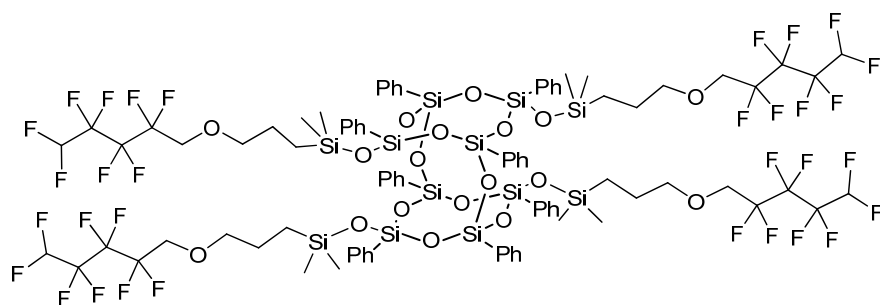


Figure S 12.  $^{29}\text{Si}$  NMR (79.5 MHz,  $\text{CDCl}_3$ ) spectrum of **triT<sub>7</sub>(F1)**.

## DDSQ-4Si(F1)



Waxy, white solid. Isolated Yield 95%, (0.186g).  $R_f=0.476$ .

**$^1\text{H}$  NMR** (300 MHz,  $\text{CDCl}_3$ , ppm):  $\delta$  = 0.04 (s, 24H,  $\text{Si}(\text{CH}_3)_2$ ), 0.44 (dd,  $J_{\text{H-H}} = 10.0$ , 6.2Hz 8H,  $\text{Si-CH}_2$ -), 1.32 (d,  $J_{\text{H-H}} = 5.9\text{Hz}$  8H,  $-\text{CH}_2$ -), 3.06 (m,  $J_{\text{H-H}} = 6.6$  Hz, 8H,  $-\text{CH}_2\text{-O}$ ), 3.61 (t,  $J_{\text{H-H}} = 14.1\text{Hz}$ , 8H,  $\text{-O-CH}_2$ -), 6.01 (tt,  $J_{\text{H-H}} = 5.5\text{Hz}$  4H,  $-\text{CH-F}_2$ ), 7.15-7.47 (m, 40H, Ph);

**$^{13}\text{C}$  NMR** (101 MHz,  $\text{CDCl}_3$ , ppm):  $\delta$  = 0.14 ( $\text{SiCH}_3$ ), 13.88 ( $\text{Si-CH}_2$ -), 23.07 ( $-\text{CH}_2$ -), 67.08-67.58 ( $-\text{CH}_2\text{-O-}$ ), 75.66 ( $\text{O-CH}_2$ -), 105.26 ( $-\text{CF}_2$ -), 107.77 ( $-\text{CF}_2$ -), 110.30 ( $-\text{CHF}_2$ -), 115.62 ( $-\text{CF}_2$ -), 127.70-128.38 (Ph), 129.19 (Ph), 130.17-130.48 (Ph), 131.67 (Ph), 133.32-134.45 (Ph);

**$^{29}\text{Si}$  NMR** (79 MHz,  $\text{CDCl}_3$ , ppm):  $\delta$  = 11.26 ( $-\text{Si}(\text{Me})_2$ -), -75.87, -78.69 ( $-\text{Si-Ph}$ );

**IR** (ATR,  $\text{cm}^{-1}$ ): 3074.47, 3053.89 (C-H phenyl), 2958.84, 2924.78 (C-H), 1594.27, 1430.62 (C=C phenyl), 1265.36 (Si-C), 1170.28 (C-F), 1127.99, 1052.45 (Si-O-Si), 998.50 (C-H phenyl).

**MALDI-ToF MS**: Calcd. for  $\text{C}_{88}\text{H}_{100}\text{F}_{32}\text{Na}^+\text{O}_{18}\text{Si}_{12}$ :  $m/z$  2411.3522  $[\text{M} + \text{Na}^+]$ . Found: 2411.3545.

The spectroscopic data are consistent with the literature.[64]

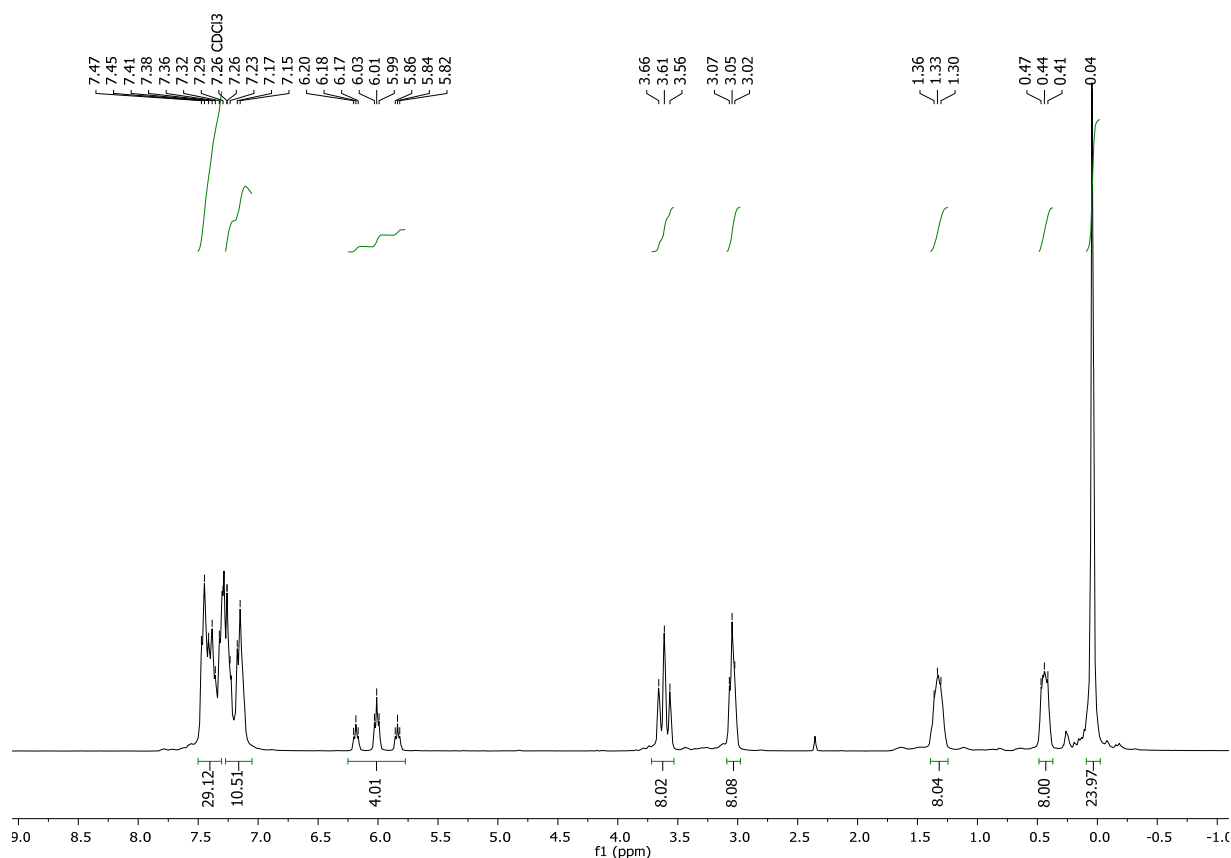


Figure S 13.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) spectrum of **DDSQ-4Si(F1)**.

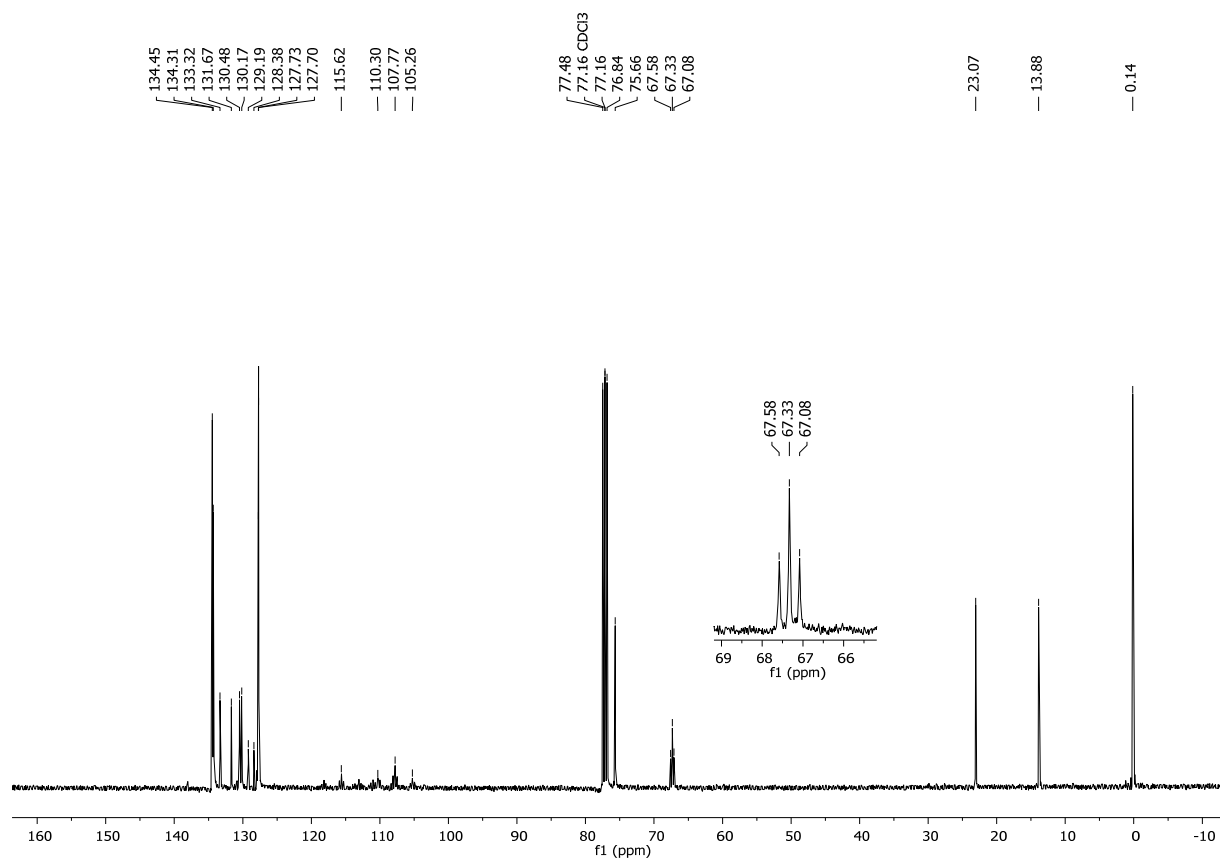


Figure S 14. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectrum of DDSQ-4Si(F1).

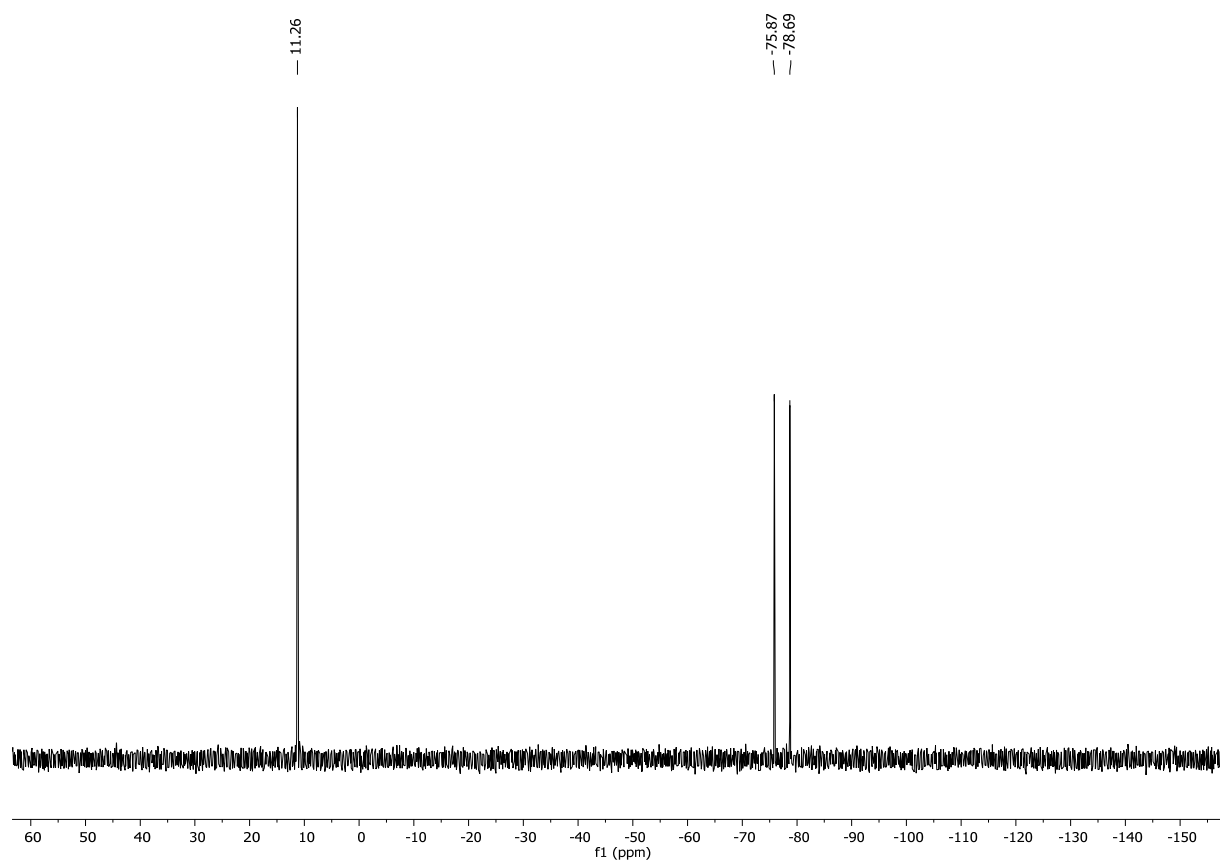
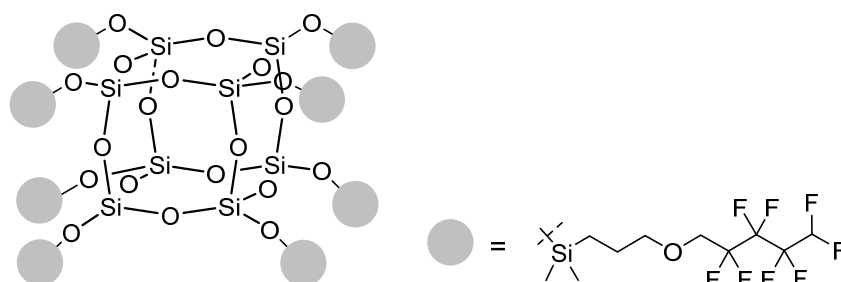


Figure S 15. <sup>29</sup>Si NMR (79.5 MHz, CDCl<sub>3</sub>) spectrum of DDSQ-4Si(F1).

**octaT<sub>8</sub>(F1)**



Oil. Isolated Yield 93%

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, ppm): δ = 0.14 (s, 48H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.56-0.62 (m, 16H, Si-CH<sub>2</sub>-), 1.64 (dq, *J*<sub>H-H</sub> = 12.7, 6.8 Hz 16H, -CH<sub>2</sub>-), 3.54 (t, *J*<sub>H-H</sub> = 6.7 Hz, 16H, -CH<sub>2</sub>-O), 3.9 (t, *J*<sub>H-H</sub> = 14.1Hz, 16H, -O-CH<sub>2</sub>-), 6.05 (tt, *J*<sub>H-H</sub> = 52.0, 5.5Hz 8H, -CH-F<sub>2</sub>);

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>, ppm): δ = -0.47 (SiCH<sub>3</sub>), 13.44 (Si-CH<sub>2</sub>-), 23.11 (-CH<sub>2</sub>-), 67.23-67.90 (-CH<sub>2</sub>-O-), 75.71 (O-CH<sub>2</sub>-), 104.37 (-CF<sub>2</sub>-), 107.75 (-CF<sub>2</sub>-), 111.09 (-CHF<sub>2</sub>-), 115.66 (-CF<sub>2</sub>-);

**<sup>29</sup>Si NMR** (79 MHz, CDCl<sub>3</sub>, ppm): δ = 13.00 (-Si-(Me)<sub>2</sub>-), -109.00 (-Si-);

**IR** (ATR, cm<sup>-1</sup>): 2958.18, 2927.81, 2880.96 (C-H), 1255.21 (Si-C), 1164.63 (C-F), 1072.06 (Si-O-Si).

The spectroscopic data are consistent with the literature.[69]

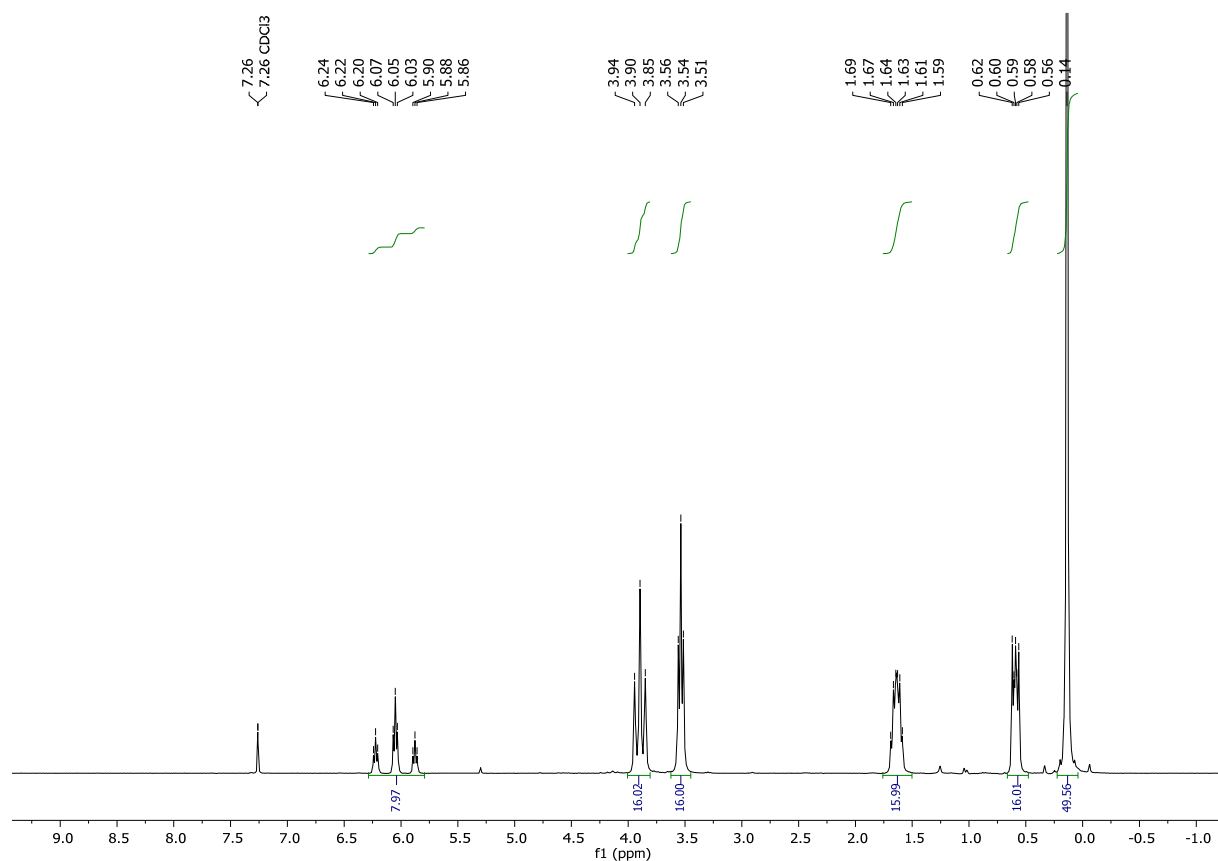


Figure S 16. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) spectrum of octaT<sub>8</sub>(F1).

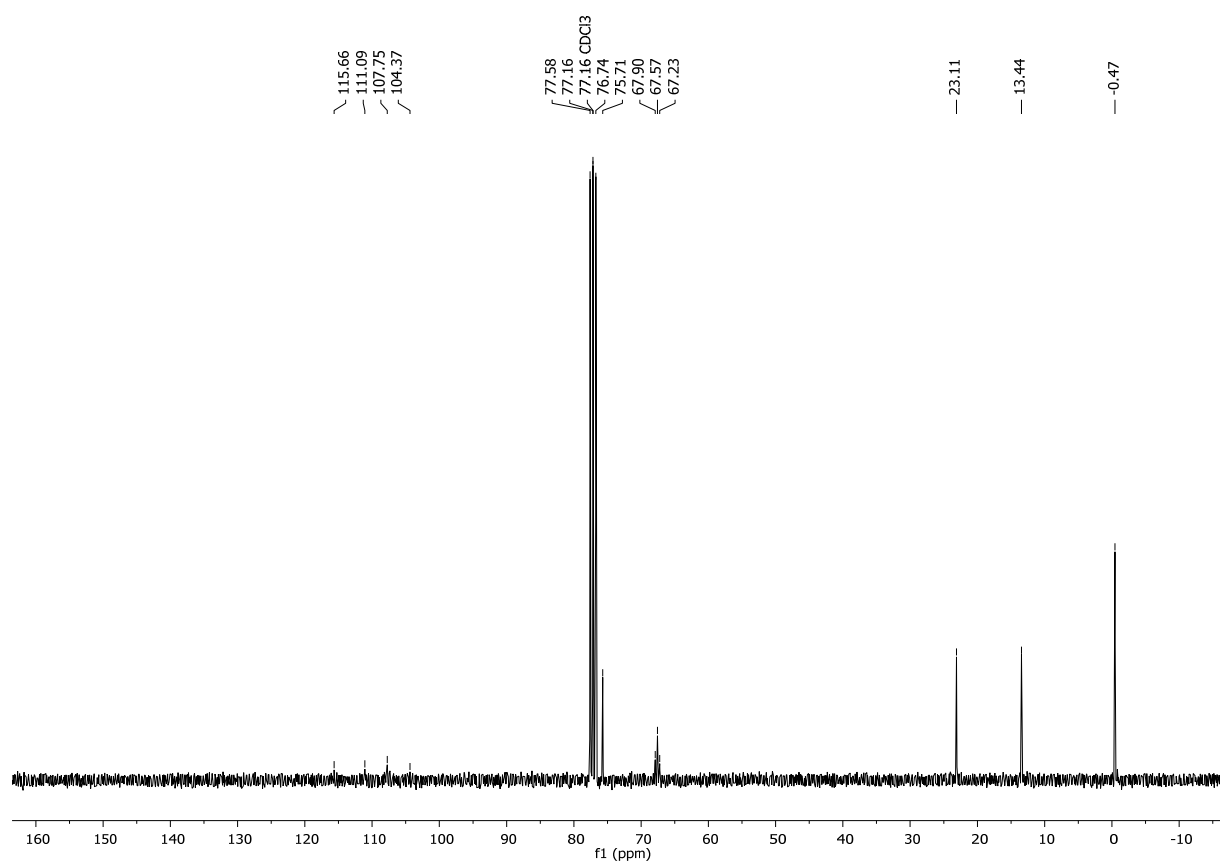


Figure S 17. <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>) spectrum of **octaT<sub>8</sub>(F1)**.

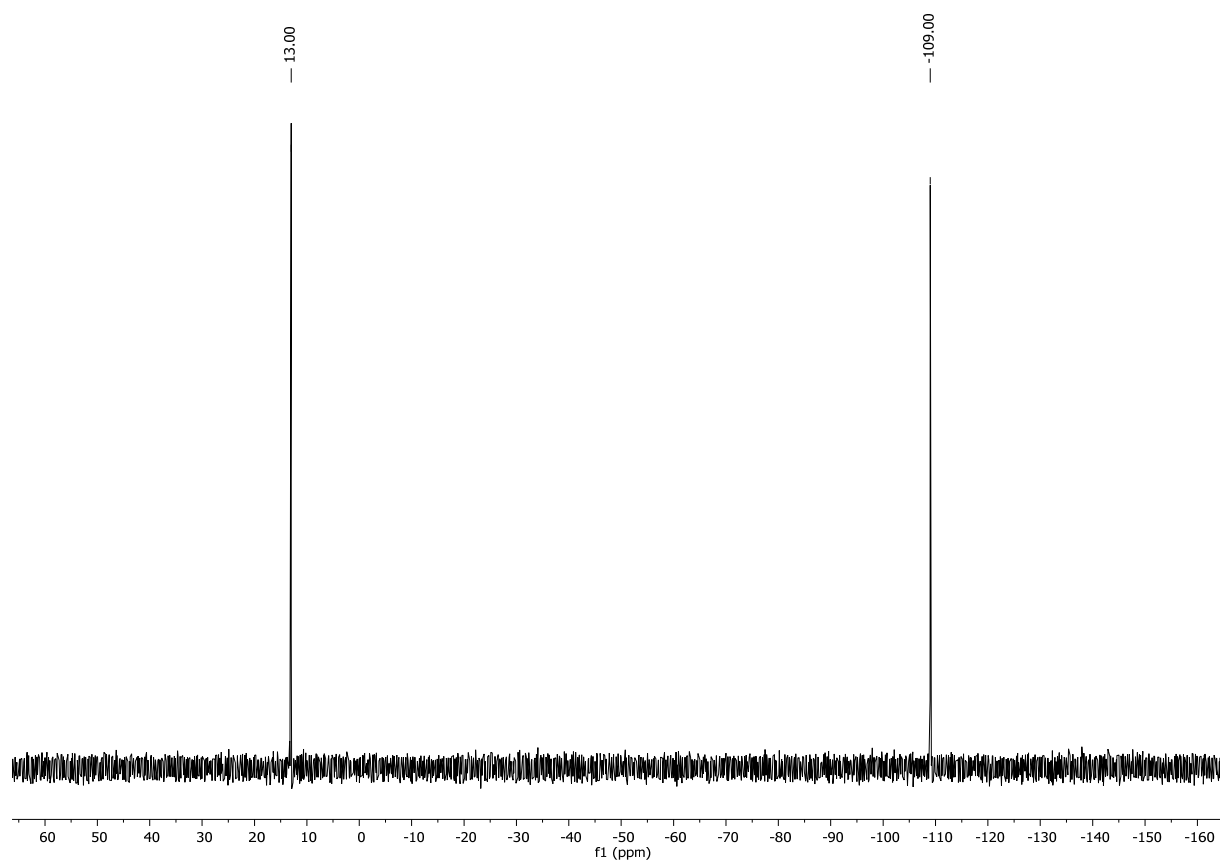
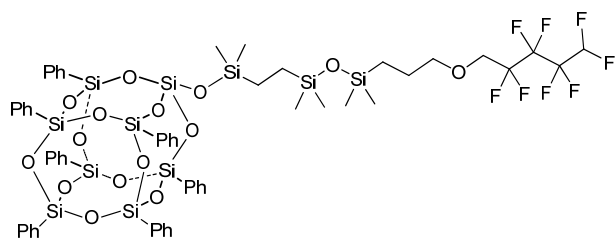


Figure S 18. <sup>29</sup>Si NMR (79.5 MHz, CDCl<sub>3</sub>) spectrum of **octaT<sub>8</sub>(F1)**.

## monoT<sub>8</sub>(F3)



Waxy, white solid. Isolated Yield 93%

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = -0.07(s, 6H, Si(CH<sub>3</sub>)<sub>2</sub>), -0.01 (s, 6H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.08 (s, 6H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.09-0.13 (m, 2H, Si-CH<sub>2</sub>-), 0.39-0.44 (m, 2H, -CH<sub>2</sub>-Si-), 0.47-0.57 (m, 2H, Si-CH<sub>2</sub>-), 1.52-1.65 (m, 2H, -CH<sub>2</sub>-), 3.51 (dt,  $J_{H-H}$  = 23.6, 6.8 Hz, 2H, -CH<sub>2</sub>-O), 3.89 (dt,  $J_{H-H}$  = 19.0, 13.9, Hz, 2H, -O-CH<sub>2</sub>-), 6.06 (tq,  $J_{H-H}$  = 52.00, 6.3 Hz, 1H, -CH-F<sub>2</sub>), 7.35-7.46, 7.73-7.77 (m, 35H, Ph);

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = -1.06 (-Si(CH<sub>3</sub>)<sub>2</sub>), -0.48 (Si(CH<sub>3</sub>)<sub>2</sub>), 0.23 (-Si(CH<sub>3</sub>)<sub>2</sub>), 9.02 (-Si-CH<sub>2</sub>-), 9.24 (-CH<sub>2</sub>-Si), 13.92 (-Si-CH<sub>2</sub>-), 23.29 (-CH<sub>2</sub>-), 67.19-67.72 (-CH<sub>2</sub>-O-), 75.76 (O-CH<sub>2</sub>-), 105.11 (-CF<sub>2</sub>-), 107.62 (-CF<sub>2</sub>-), 110.16 (-CHF<sub>2</sub>-), 115.53 (-CF<sub>2</sub>-), 127.67-127.83 (Ph), 130.16 (Ph), 130.19 (Ph), 130.75 (Ph), 134.00-134.19 (Ph);

**<sup>29</sup>Si NMR** (79 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = 7.24 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), 8.72 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), 13.66 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), -78.37, -78.32, -78.37 (-Si-Ph), -108.90 (-SiO<sub>4</sub>);

**IR** (ATR, cm<sup>-1</sup>): 3073.94, 3051.76 (C-H phenyl), 2956.72, 2924.13 (C-H), 1594.45, 1430.71 (C=C phenyl), 1254.19 (Si-C), 1131.06 (C-F), 1043.72 (Si-O-Si), 997.21 (C-H phenyl).

**EA:** Anal. calcd for C<sub>58</sub>H<sub>66</sub>F<sub>8</sub>O<sub>15</sub>Si<sub>11</sub> (%): C, 47.58; H, 4.54; found: C, 47.46; H, 4.53.

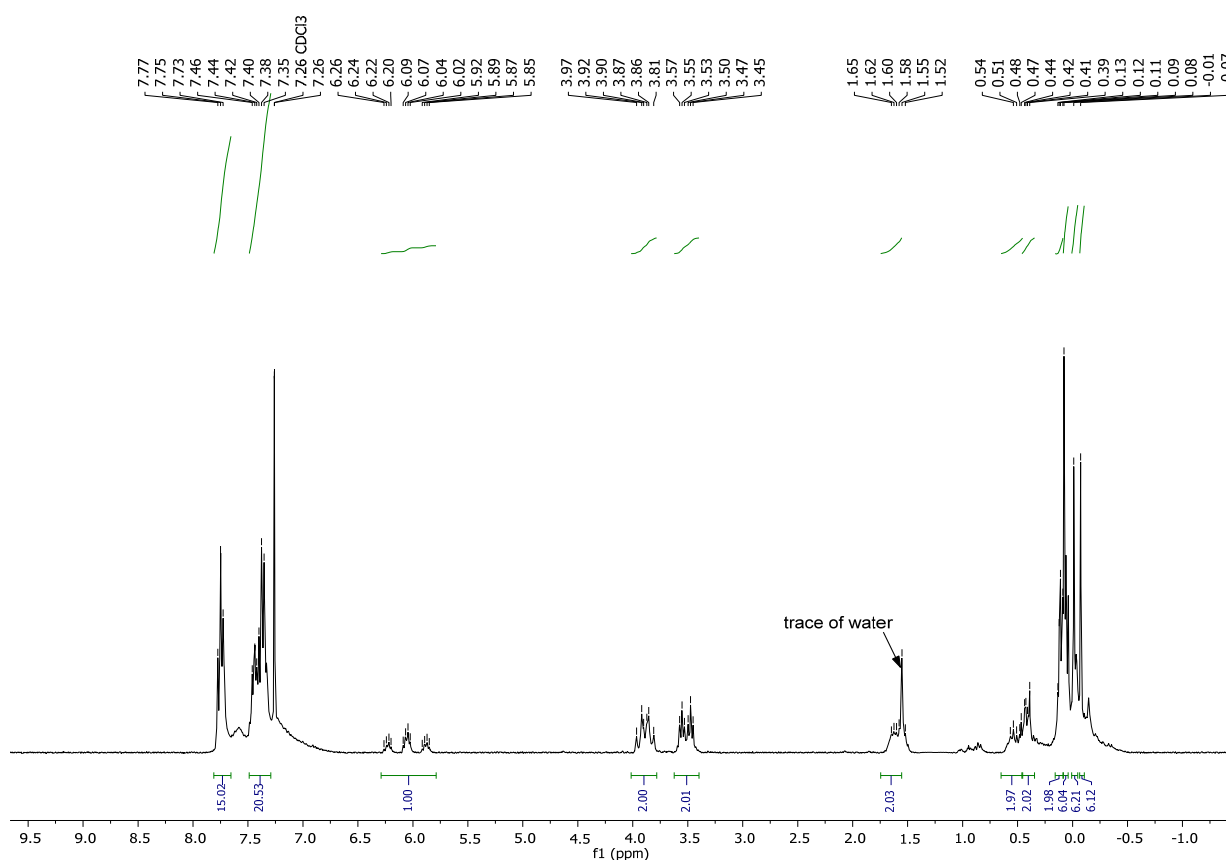


Figure S 19. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) spectrum of monoT<sub>8</sub>(F3).



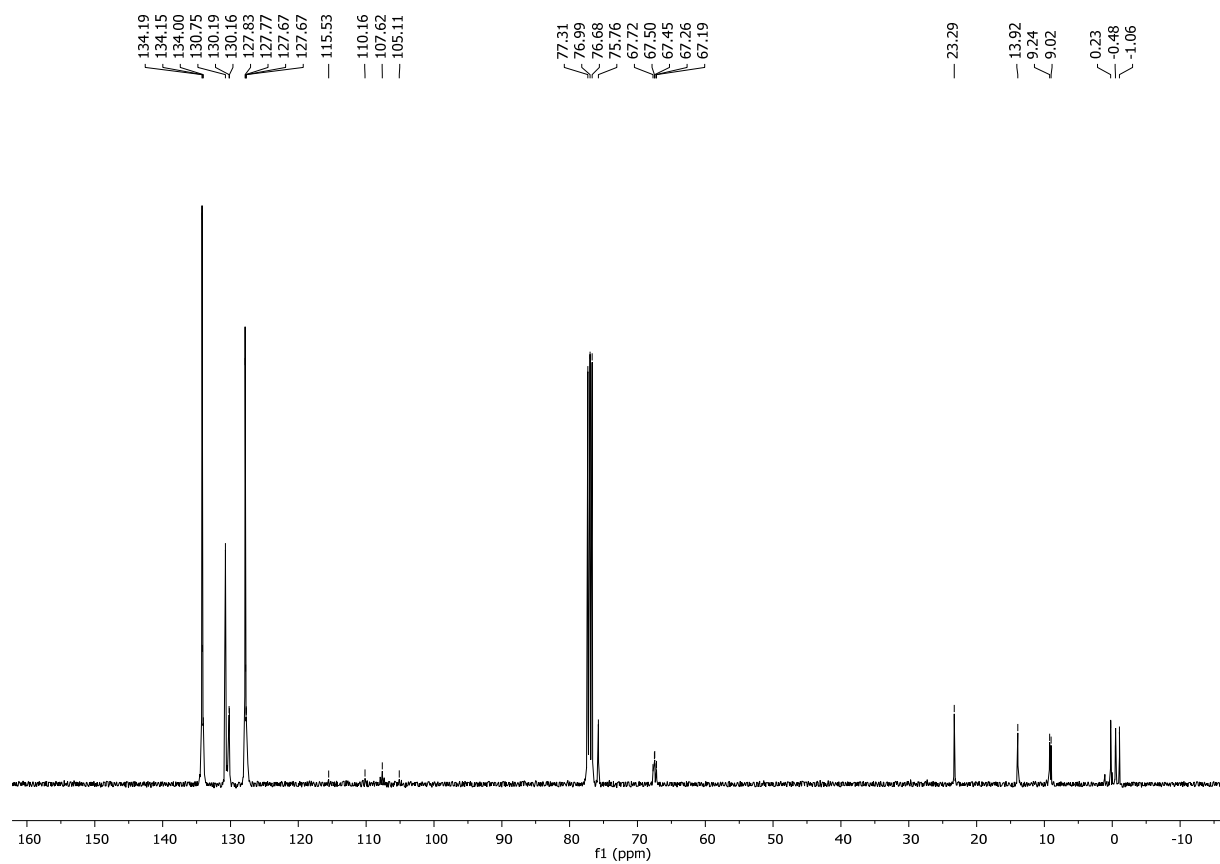


Figure S 20.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectrum of **monoT<sub>8</sub>(F<sub>3</sub>)**.

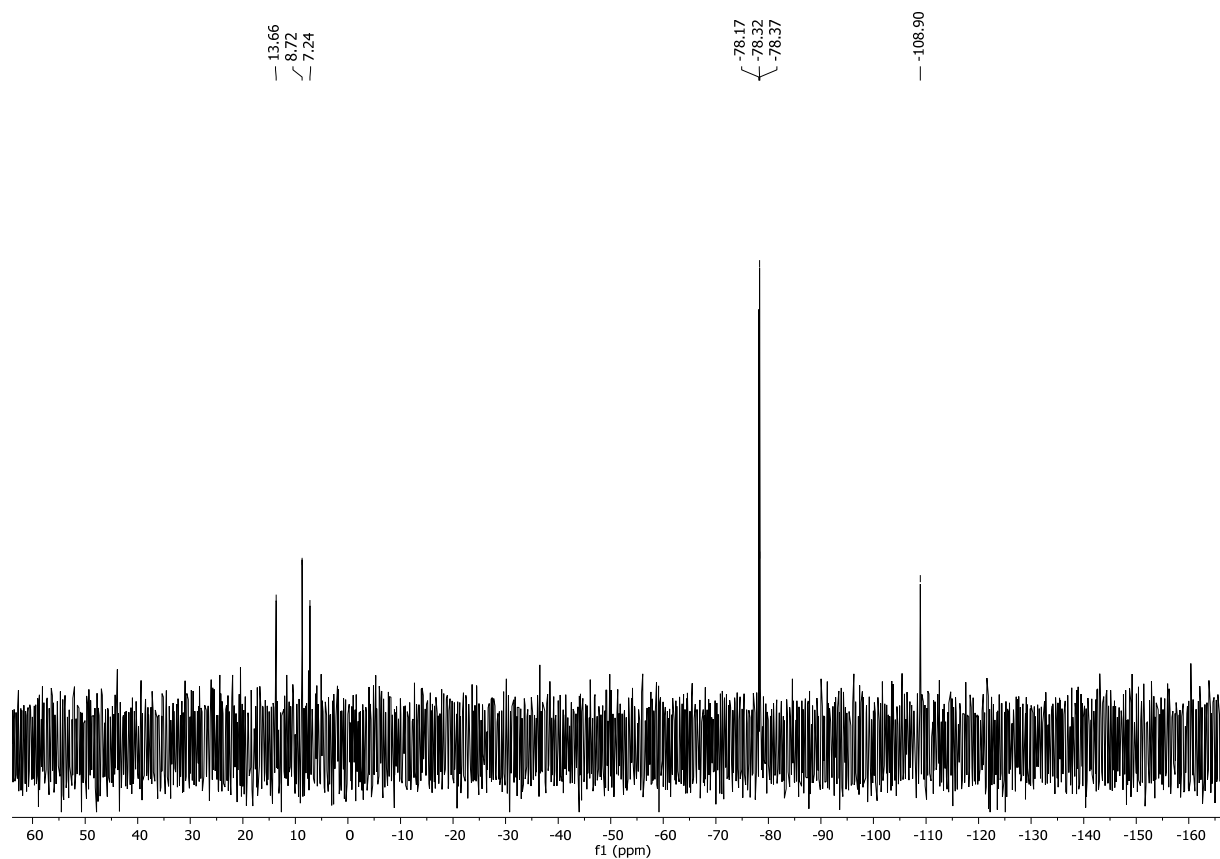
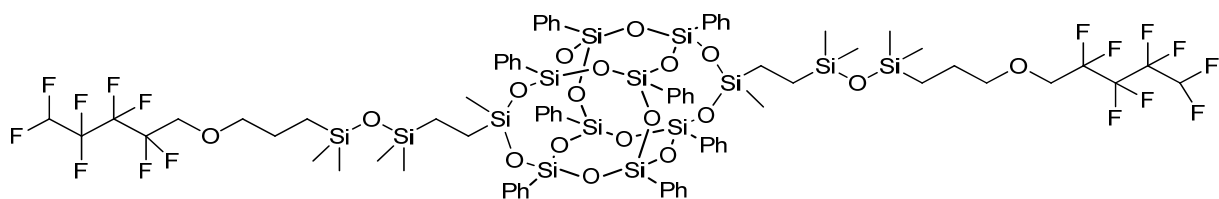


Figure S 21.  $^{29}\text{Si}$  NMR (79.5 MHz,  $\text{CDCl}_3$ ) spectrum of **monoT<sub>8</sub>(F<sub>3</sub>)**.

## DDSQ-2Si(F3)



White solid. Isolated Yield 91%

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = -0.09 (d,  $J_{H-H}$  = 2.8 Hz, 12H, Si(CH<sub>3</sub>)<sub>2</sub>), -0.03 (d,  $J_{H-H}$  = 2.6 Hz, 12H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.07-0.15 (m, 4H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.30 (d,  $J_{H-H}$  = 2.8 Hz, 6H, Si-CH<sub>2</sub>-), 0.40-0.54 (m, 4H, -CH<sub>2</sub>-Si-), 0.58-0.62 (m, 4H, Si-CH<sub>2</sub>-), 1.51-1.56 (m, 4H, -CH<sub>2</sub>-), 3.41-3.58 (m, 4H, -CH<sub>2</sub>-O-), 3.84 (t,  $J_{H-H}$  = 13.9 Hz, 4H, -O-CH<sub>2</sub>-), 6.04 (tt,  $J_{H-H}$  = 51.07, 6.0 Hz, 2H, -CH-F<sub>2</sub>), 7.18-7.57 (m, 40H, Ph);

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = -1.56 (-Si(CH<sub>3</sub>)<sub>2</sub>), -0.39 (Si(CH<sub>3</sub>)<sub>2</sub>), 0.35 (-Si(CH<sub>3</sub>)<sub>2</sub>), 8.43 (-Si-CH<sub>2</sub>-), 9.25 (-CH<sub>2</sub>-Si), 14.09 (-Si-CH<sub>2</sub>-), 23.45 (-CH<sub>2</sub>-), 67.35-67.85 (-CH<sub>2</sub>-O-), 75.89 (O-CH<sub>2</sub>-), 105.31 (-CF<sub>2</sub>-), 107.82 (-CF<sub>2</sub>-), 110.34 (-CHF<sub>2</sub>-), 115.70 (-CF<sub>2</sub>-), 127.80-127.92 (Ph), 130.47 (Ph), 131.32 (Ph), 132.38 (Ph), 134.13-134.23 (Ph);

**<sup>29</sup>Si NMR** (79 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = 8.69 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), 7.33 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), -17.08 (-Si-(CH<sub>3</sub>)<sub>2</sub>), -78.69, -79.64 (-Si-Ph);

**IR** (ATR, cm<sup>-1</sup>): 3073.49 (C-H phenyl), 2923.95 (C-H), 1594.23, 1430.48 (C=C phenyl), 1262.19 (Si-C), 1172.16 (C-F), 1028.14 (Si-O-Si), 997.48 (C-H phenyl).

**EA:** Anal. calcd for C<sub>78</sub>H<sub>96</sub>F<sub>16</sub>O<sub>18</sub>Si<sub>14</sub> (%): C, 46.41; H, 4.79; found: C, 46.50; H, 4.80.

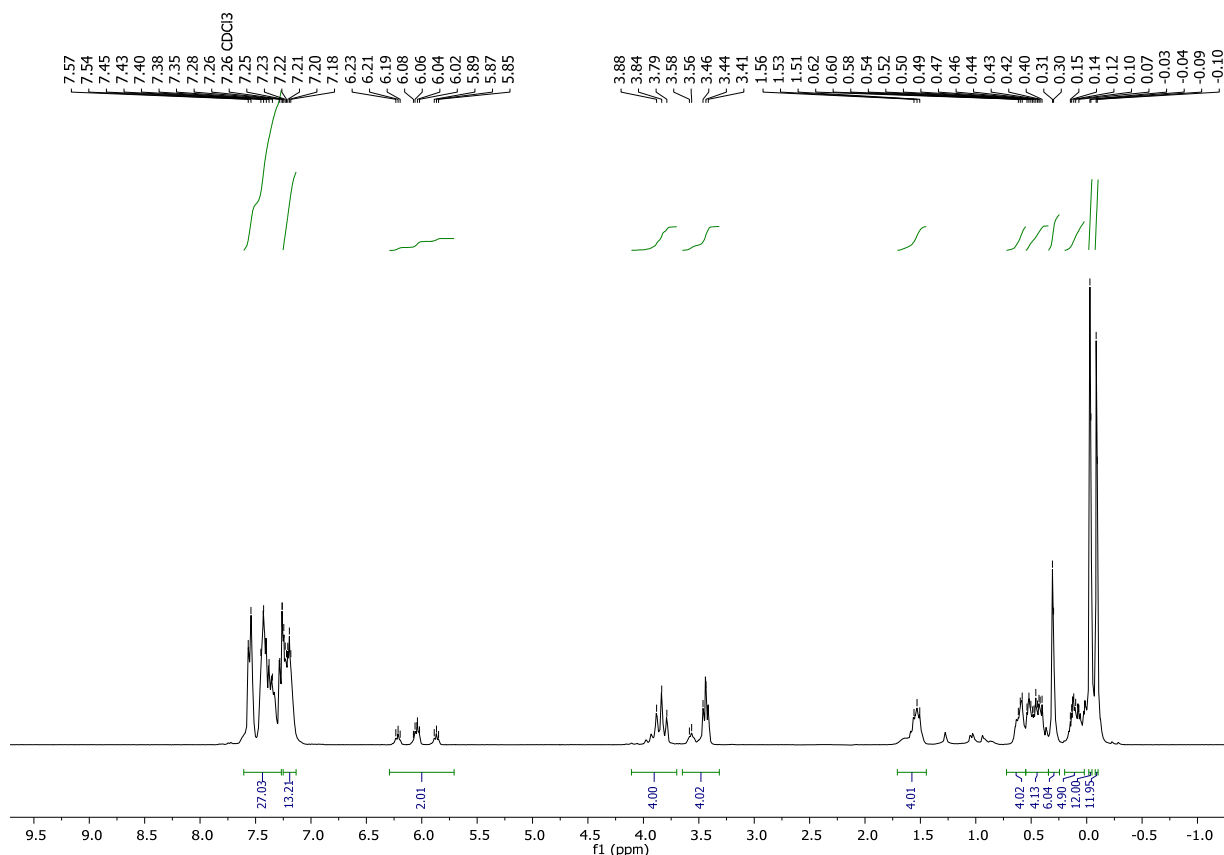


Figure S 22. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) spectrum of DDSQ-2Si(F3).

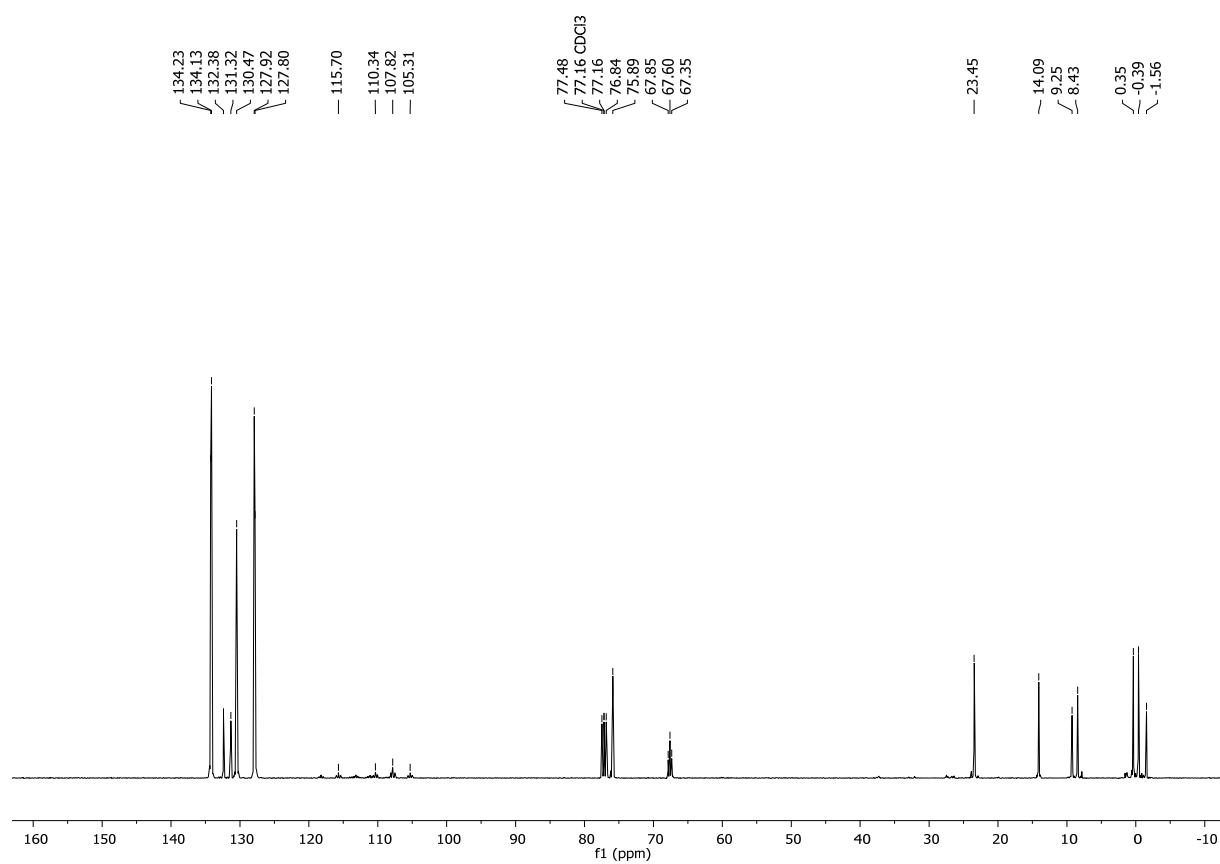


Figure S 23.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectrum of **DDSQ-2Si(F3)**.

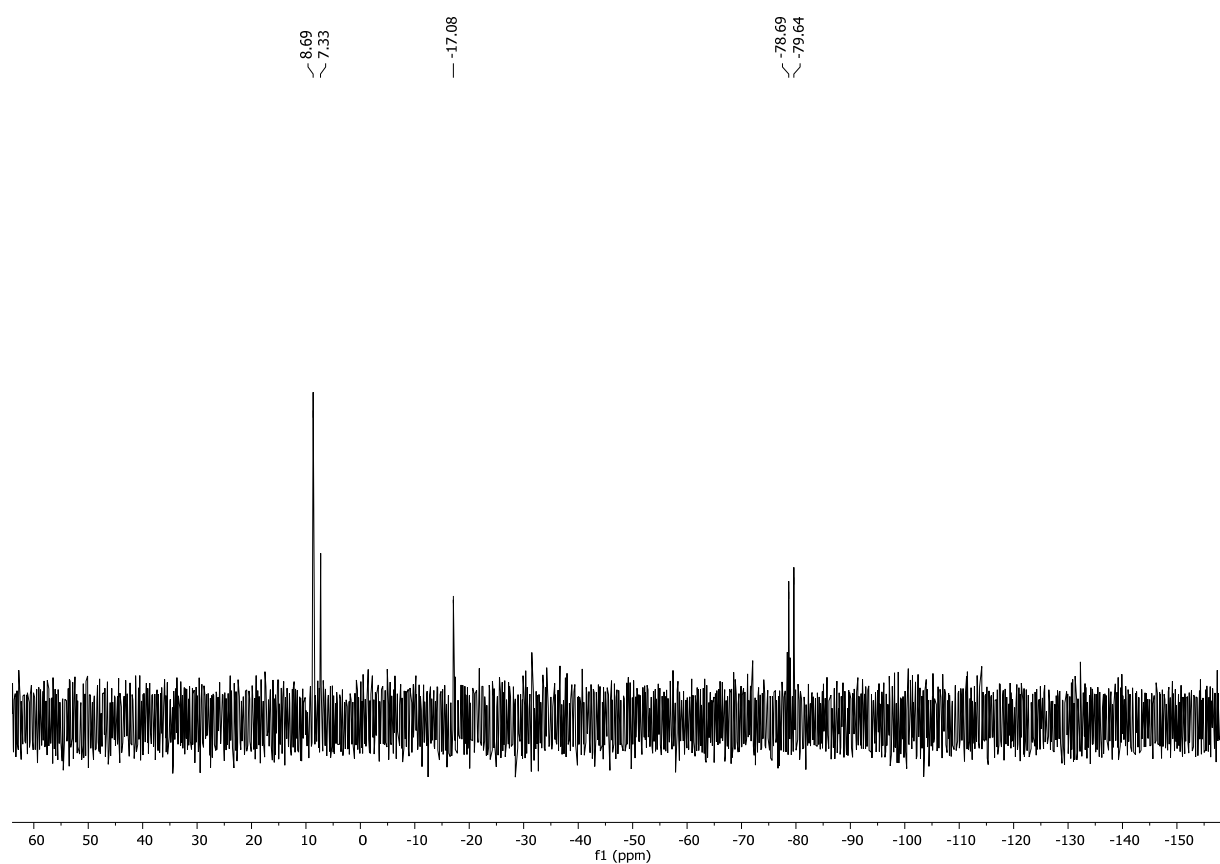
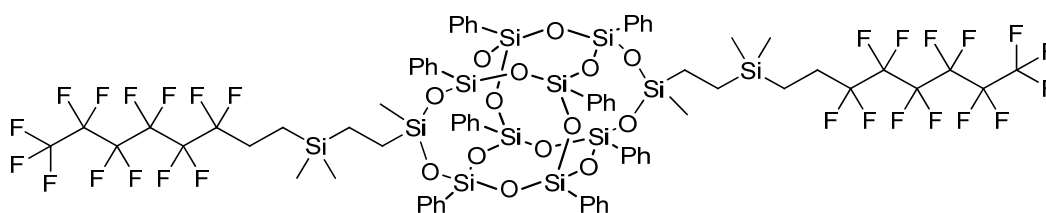


Figure S 24.  $^{29}\text{Si}$  NMR (79.5 MHz,  $\text{CDCl}_3$ ) spectrum of **DDSQ-2Si(F3)**.

## DDSQ-2Si(F4)



White solid. Isolated Yield 88%

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = -0.16 (s, 12H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.29 (s, 6H, SiCH<sub>3</sub>), 0.52-0.61 (m, 12H, Si-CH<sub>2</sub>-, -CH<sub>2</sub>-), 1.76-1.93 (m, 4H, -CH<sub>2</sub>-), 7.20-7.54 (m, 40H, Ph);

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = -4.46 (-Si(CH<sub>3</sub>)), -1.63-(Si(CH<sub>3</sub>)<sub>2</sub>), 3.84 (-CH<sub>2</sub>-), 5.67 (-CH<sub>2</sub>-), 8.66 (-CH<sub>2</sub>-), 25.49-25.97 (-CH<sub>2</sub>-), 115.82 (-CF-), 117.52 (-CF-), 118.11 (-CF-), 118.43 (-CF-), 118.71 (-CF-), 119.59 (-CF-), 125.32 (Ph), 127.67-128.25 (Ph), 129.05 (Ph), 130.40 (Ph), 131.06 (Ph), 132.05 (Ph), 133.91-134.04 (Ph);

**<sup>29</sup>Si NMR** (79 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = -5.59 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), -17.60 (-Si-(CH<sub>3</sub>)-), -78.60, -79.57, -79.64, -79.72 (-Si-Ph);

**IR** (ATR, cm<sup>-1</sup>): 3073.16, 3052.02, 3027.25 (C-H phenyl), 2954.81, 2909.70 (C-H), 1594.22, 1430.38 (C=C phenyl), 1239.96 (Si-C), 1207.93 (C-F), 1029.19 (Si-O-Si), 998.35 (C-H phenyl).

**EA:** Anal. calcd for C<sub>74</sub>H<sub>74</sub>F<sub>26</sub>O<sub>14</sub>Si<sub>12</sub> (%): C, 44.04; H, 3.70; found: C, 44.16; H, 3.71.

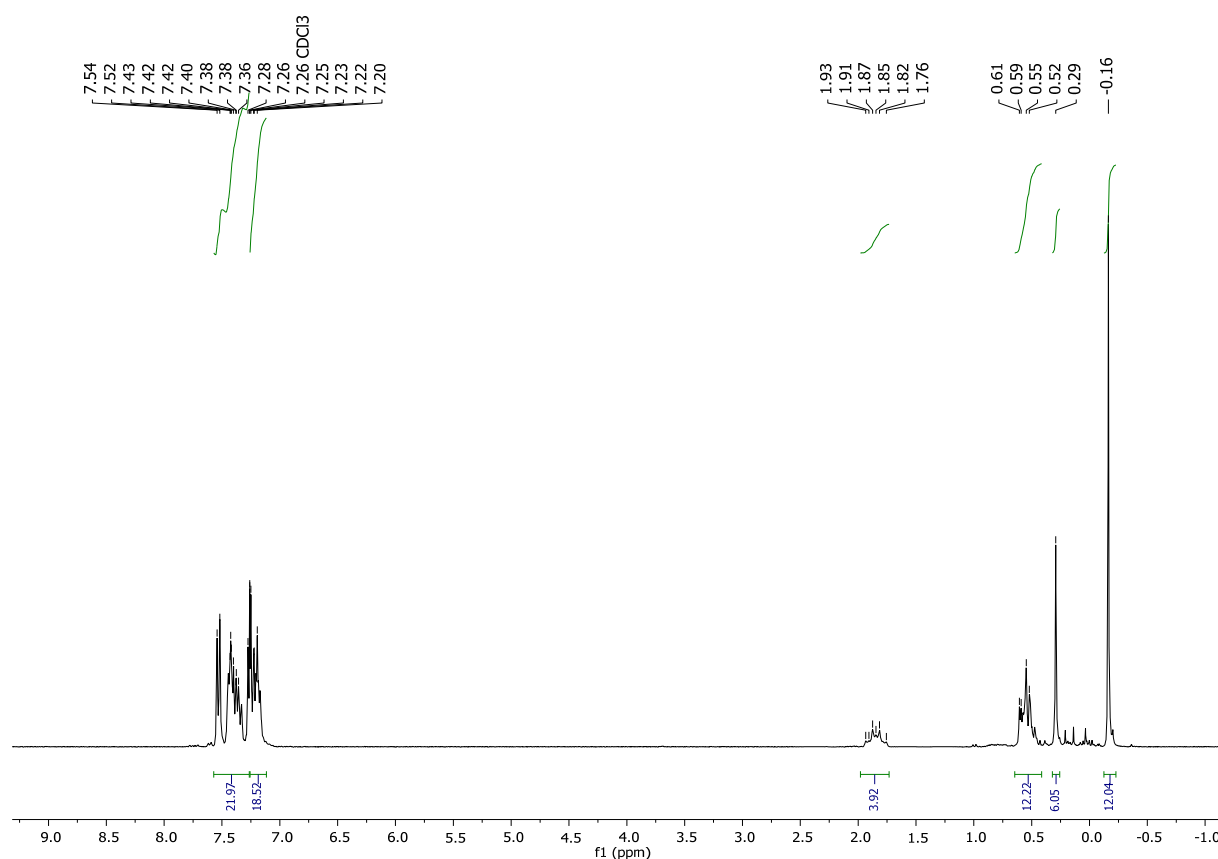


Figure S 25. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) spectrum of DDSQ-2Si(F4).

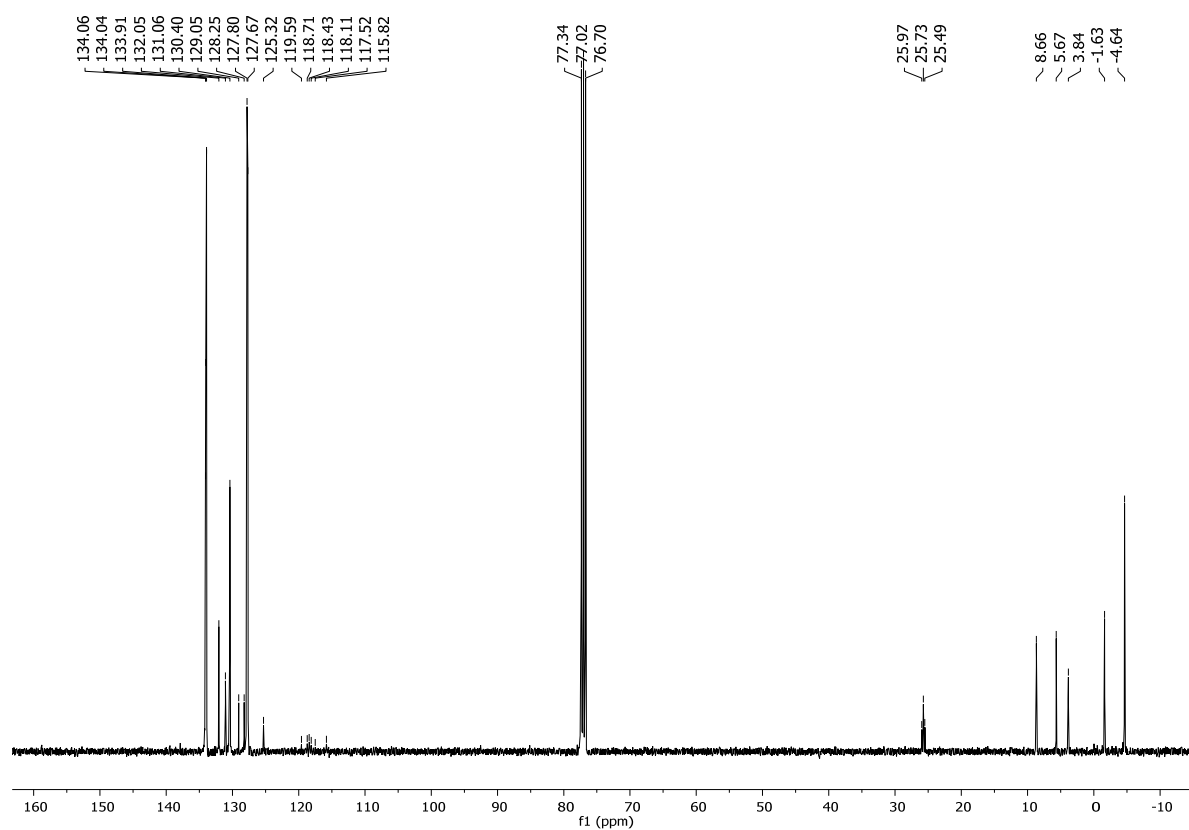


Figure S 26.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectrum of **DDSQ-2Si(F4)**.

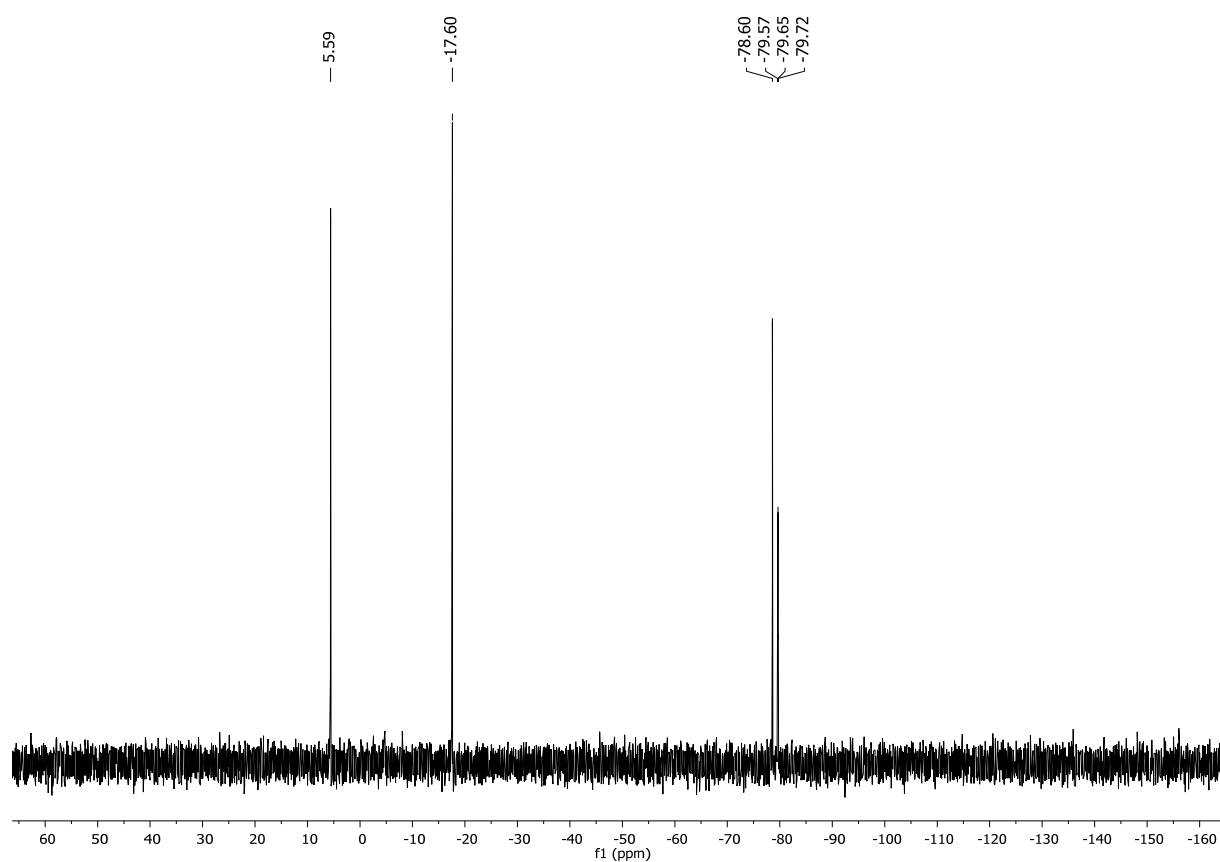
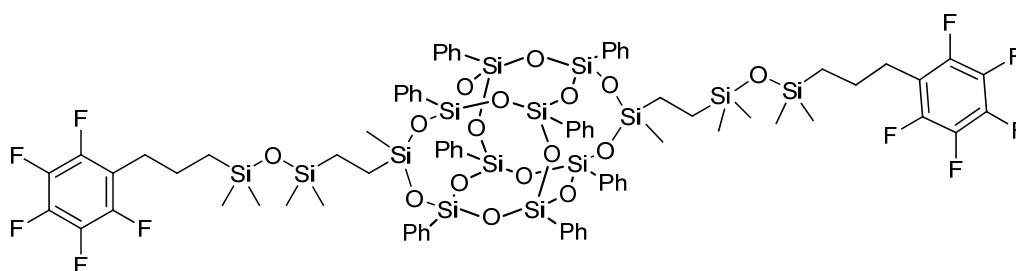


Figure S 27.  $^{29}\text{Si}$  NMR (79.5 MHz,  $\text{CDCl}_3$ ) spectrum of **DDSQ-2Si(F4)**.

## DDSQ-2Si(F5)



Waxy white solid. Isolated Yield 87%

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, ppm): δ = -0.09 (s, 12H, Si(CH<sub>3</sub>)<sub>2</sub>), -0.03 (s, 12H, SiCH<sub>3</sub>), 0.03-0.11 (m, 4H, Si-CH<sub>2</sub>-), 0.32 (s, 6H, Si(CH<sub>3</sub>)), 0.46-0.54 (m, 4H, -CH<sub>2</sub>-), 0.59-0.65 (m, 4H, -CH<sub>2</sub>-), 1.49-1.60 (m, 4H, -CH<sub>2</sub>-), 2.63 (t, *J*<sub>H-H</sub> = 7.7 Hz, 4H, -CH<sub>2</sub>-), 7.20-7.58 (m, 40H, Ph);

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>, ppm): δ = -1.59 (-Si(CH<sub>3</sub>)), 0.41(-Si(CH<sub>3</sub>)<sub>2</sub>), 0.37 (-Si(CH<sub>3</sub>)<sub>2</sub>), 8.37 (-Si-CH<sub>2</sub>-), 9.20 (-CH<sub>2</sub>-Si), 18.20 (-Si-CH<sub>2</sub>-), 23.54 (-CH<sub>2</sub>-), 25.86 (-CH<sub>2</sub>-), 115.47 (-C-), 127.73-127.88 (Ph), 130.43 (Ph), 131.28 (Ph), 132.34 (Ph), 134.09-134.20 (Ph), 136.24 (-CF-), 138.72 (-CF-), 140.74 (-CF-), 143.93 (-CF-), 146.37 (-CF-);

**<sup>29</sup>Si NMR** (79 MHz, CDCl<sub>3</sub>, ppm): δ = 8.77, 6.80 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), -17.08 (-Si-(CH<sub>3</sub>)<sub>3</sub>-), -78.67, -79.58, -79.63, -79.67 (-Si-Ph);

**IR** (ATR, cm<sup>-1</sup>): 3073.02, 3051.29, 3027.75 (C-H phenyl), 2956.08, 2920.51 (C-H), 1655.03, 1594.11 (C=C phenyl), 1253.73 (Si-C), 1084.53.82, 1028.21.36 (Si-O-Si), 997.91 (C-H phenyl);

**EA:** Anal. calcd for C<sub>80</sub>H<sub>90</sub>F<sub>10</sub>O<sub>16</sub>Si<sub>14</sub> (%): C, 50.82; H, 4.80; found: C, 50.80; H, 4.79.

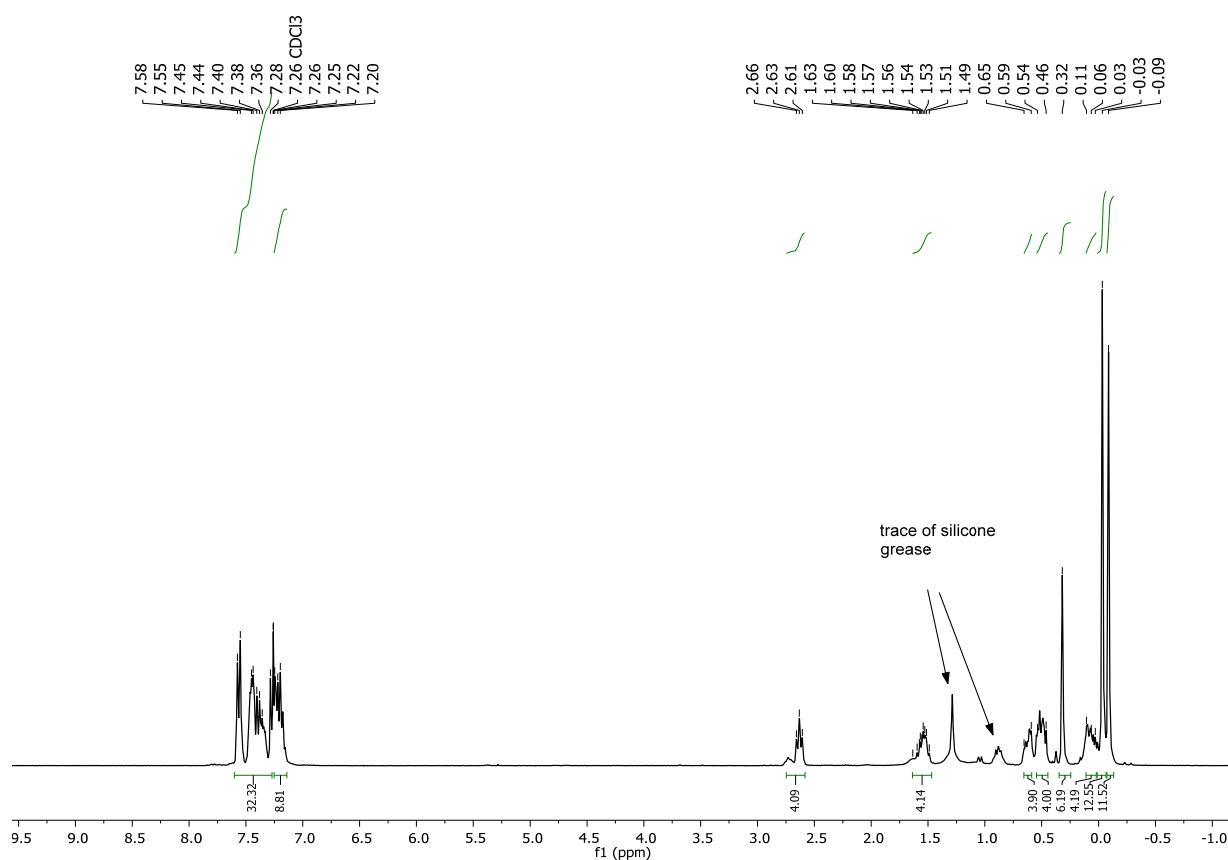


Figure S 28. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) spectrum of DDSQ-2Si(F5).

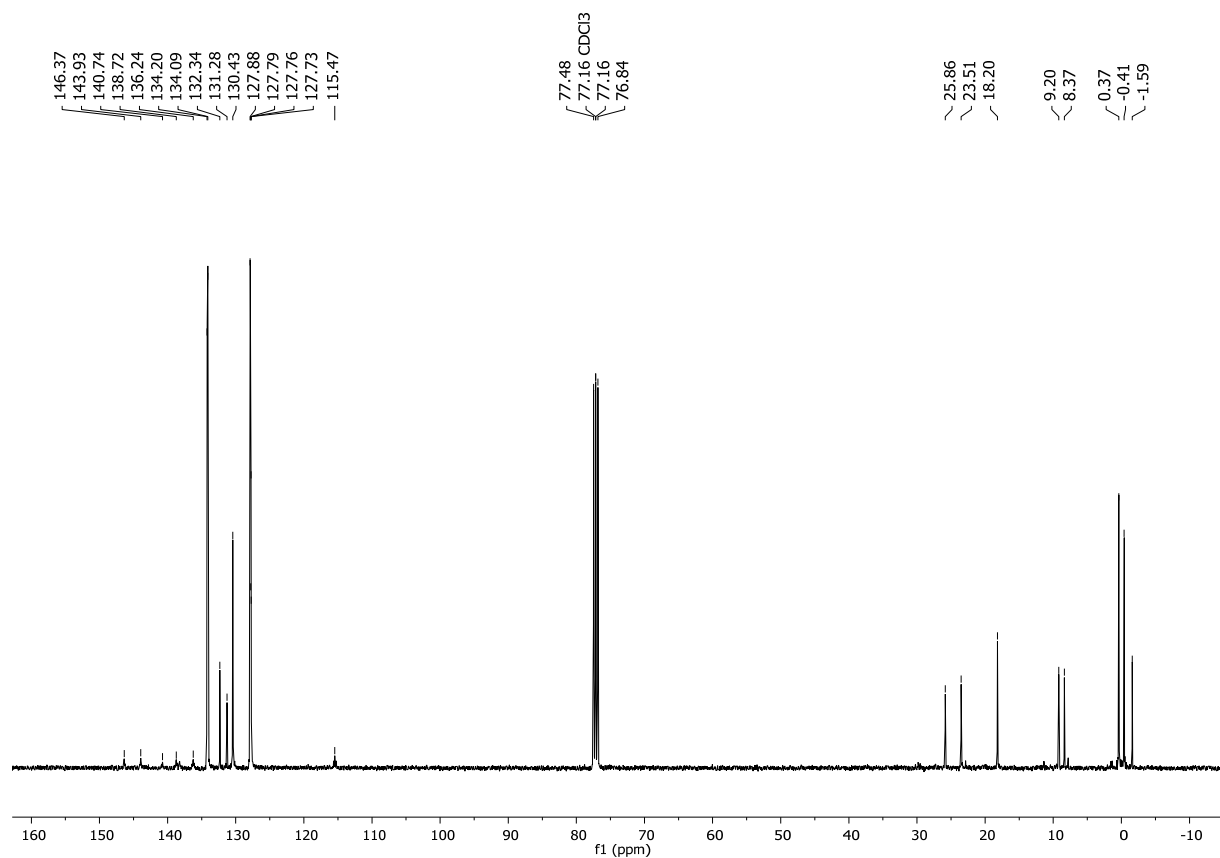


Figure S 29. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectrum of **DDSQ-2Si(F5)**.

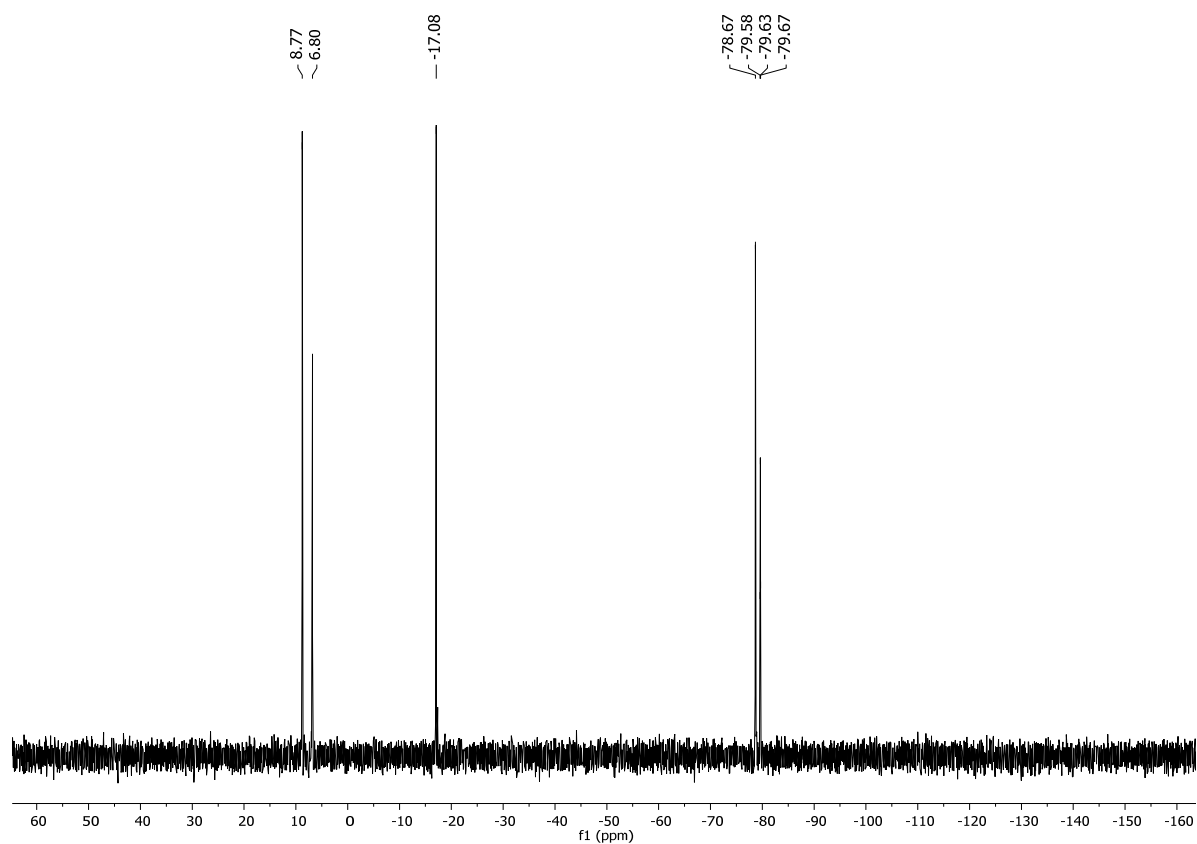
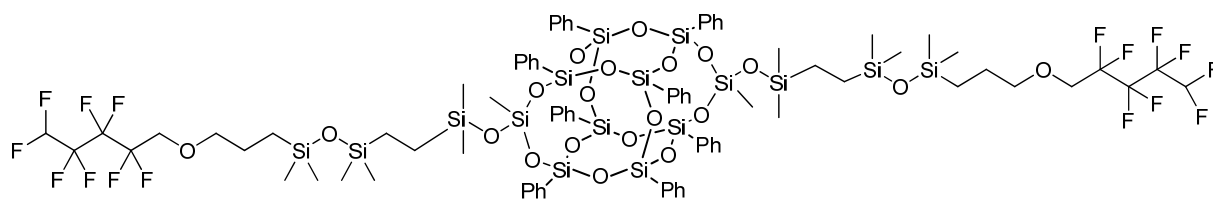


Figure S 30. <sup>29</sup>Si NMR (79.5 MHz, CDCl<sub>3</sub>) spectrum of **DDSQ-2Si(F5)**.

## DDSQ-2OSi(F3)



White solid. Isolated Yield 94%

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = -0.08 (s, 12H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.00 (s, 12H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.08 (s, 12H, Si-CH<sub>2</sub>-), 0.28 (s, 6H, Si-CH<sub>2</sub>-), 0.37-0.40 (d,  $J_{H-H}$  = 5.5 Hz, 4H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.43-0.49 (m, 4H, -CH<sub>2</sub>-Si-), 0.84-0.97 (td,  $J_{H-H}$  = 15.4, 7.7 Hz, 4H, Si-CH<sub>2</sub>-), 1.51-1.60 (m, 4H, -CH<sub>2</sub>-), 3.50 (t,  $J_{H-H}$  = 6.9 Hz, 4H, -CH<sub>2</sub>-O), 3.88 (t,  $J_{H-H}$  = 14.1 Hz, 4H, -O-CH<sub>2</sub>-), 6.05 (tt,  $J_{H-H}$  = 52.1, 5.7 Hz, 2H, -CH-F<sub>2</sub>), 7.16-7.58 (m, 40H, Ph);

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = -2.59 (-Si(CH<sub>3</sub>)), -0.61(-Si(CH<sub>3</sub>)<sub>2</sub>), -0.36 (-Si(CH<sub>3</sub>)<sub>2</sub>), 0.38 (-Si(CH<sub>3</sub>)<sub>2</sub>), 9.40 (-Si-CH<sub>2</sub>-), 9.53 (-CH<sub>2</sub>-Si), 14.09 (-Si-CH<sub>2</sub>-), 23.46 (-CH<sub>2</sub>-), 67.46-67.86 (-CH<sub>2</sub>-O-), 75.93 (O-CH<sub>2</sub>-), 105.27 (-CF<sub>2</sub>-), 107.79 (-CF<sub>2</sub>-), 110.31 (-CHF<sub>2</sub>-), 115.67 (-CF<sub>2</sub>-), 127.63-127.94 (Ph), 130.44-130.49 (Ph), 131.19 (Ph), 132.11 (Ph), 134.10-134.30 (Ph);

**<sup>29</sup>Si NMR** (79 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = 10.88 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), 8.80 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), 7.20 (-Si-(CH<sub>3</sub>)<sub>2</sub>), -64.23 (-SiCH<sub>3</sub>), -78.25, -79.31, -79.52 (-Si-Ph);

**IR** (ATR, cm<sup>-1</sup>): 3073.64, 3052.10 (C-H phenyl), 2956.20, 2922.43 (C-H), 1594.58, 1430.51 (C=C phenyl), 1253.28 (Si-C), 1170.38 (C-F), 1107.03, 1028.21 (Si-O-Si), 997.84 (C-H phenyl).

**EA:** Anal. calcd for C<sub>82</sub>H<sub>108</sub>F<sub>16</sub>O<sub>20</sub>Si<sub>16</sub> (%): C, 45.45; H, 5.02; found: C, 45.38; H, 5.01.

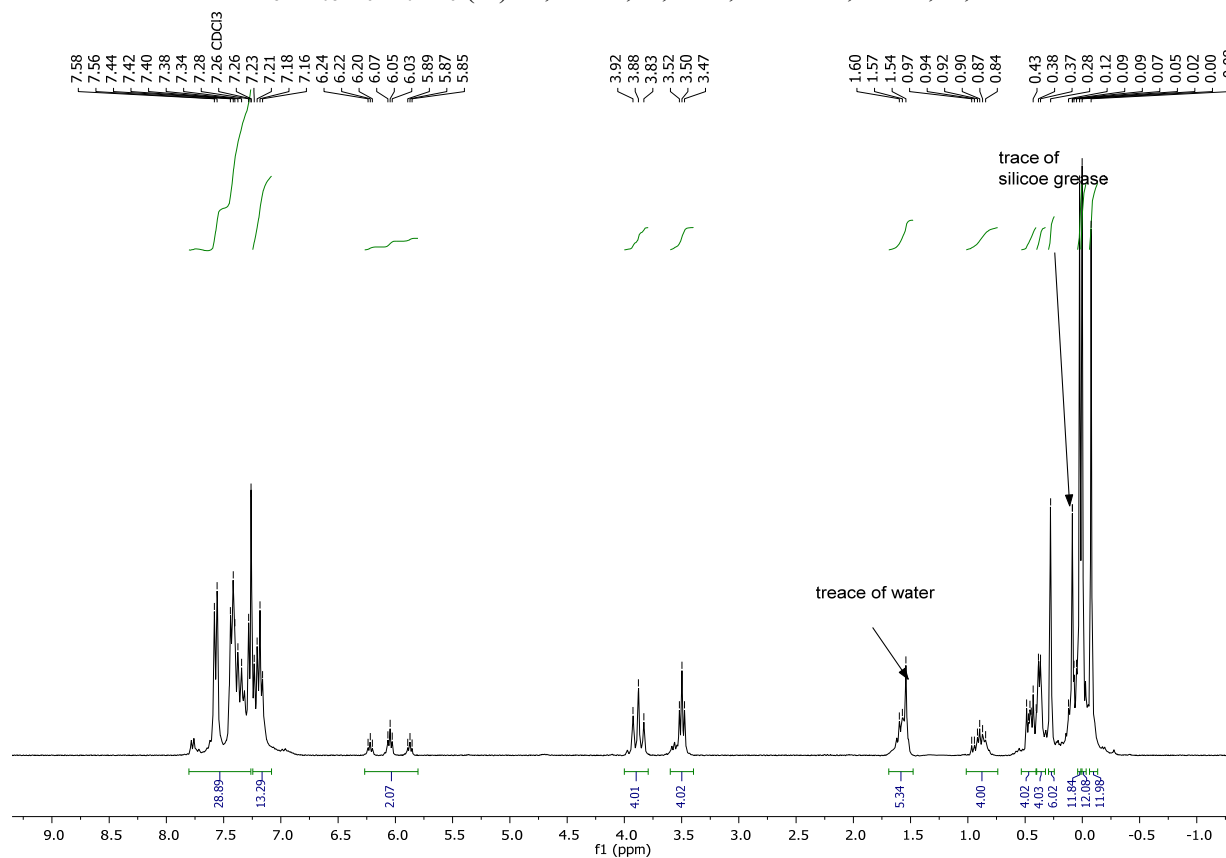


Figure S 31. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) spectrum of DDSQ-2OSi(F3).



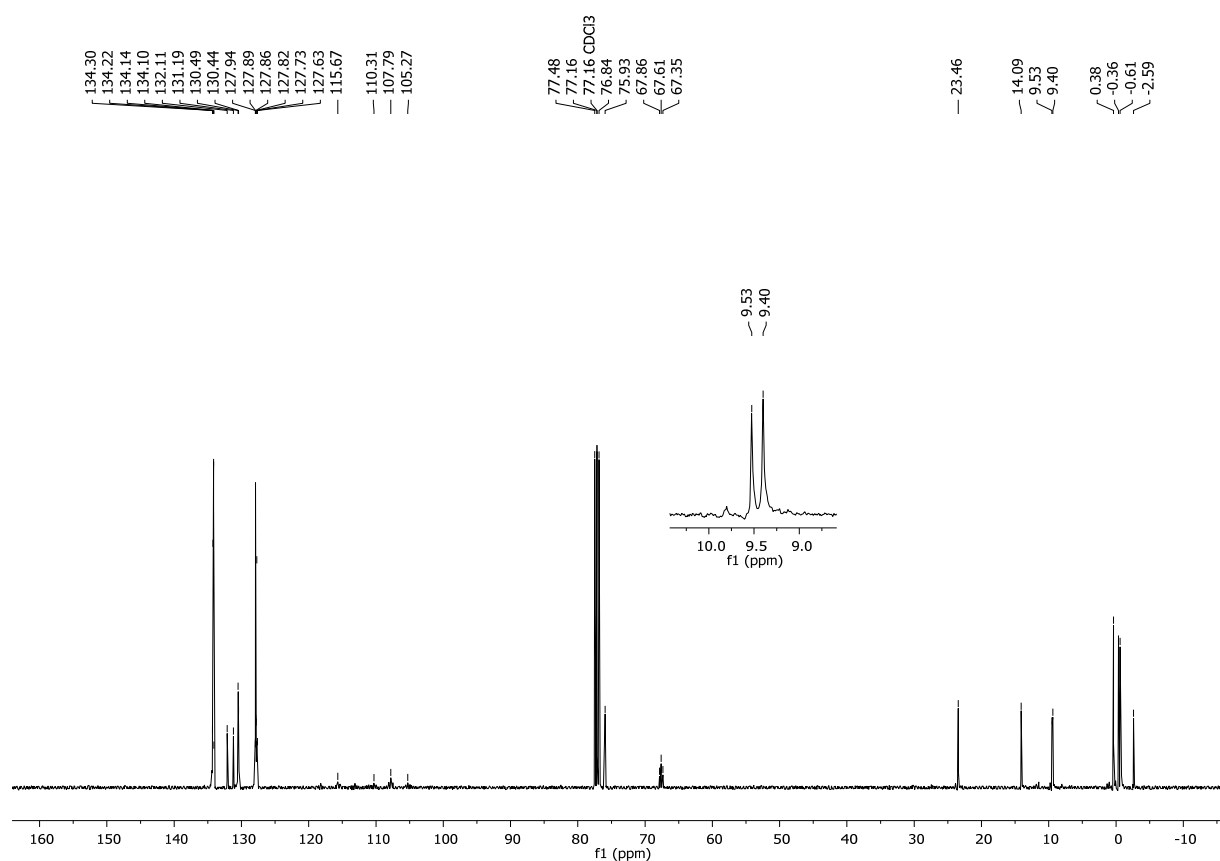


Figure S 32. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectrum of DDSQ-2OSi(F<sub>3</sub>).

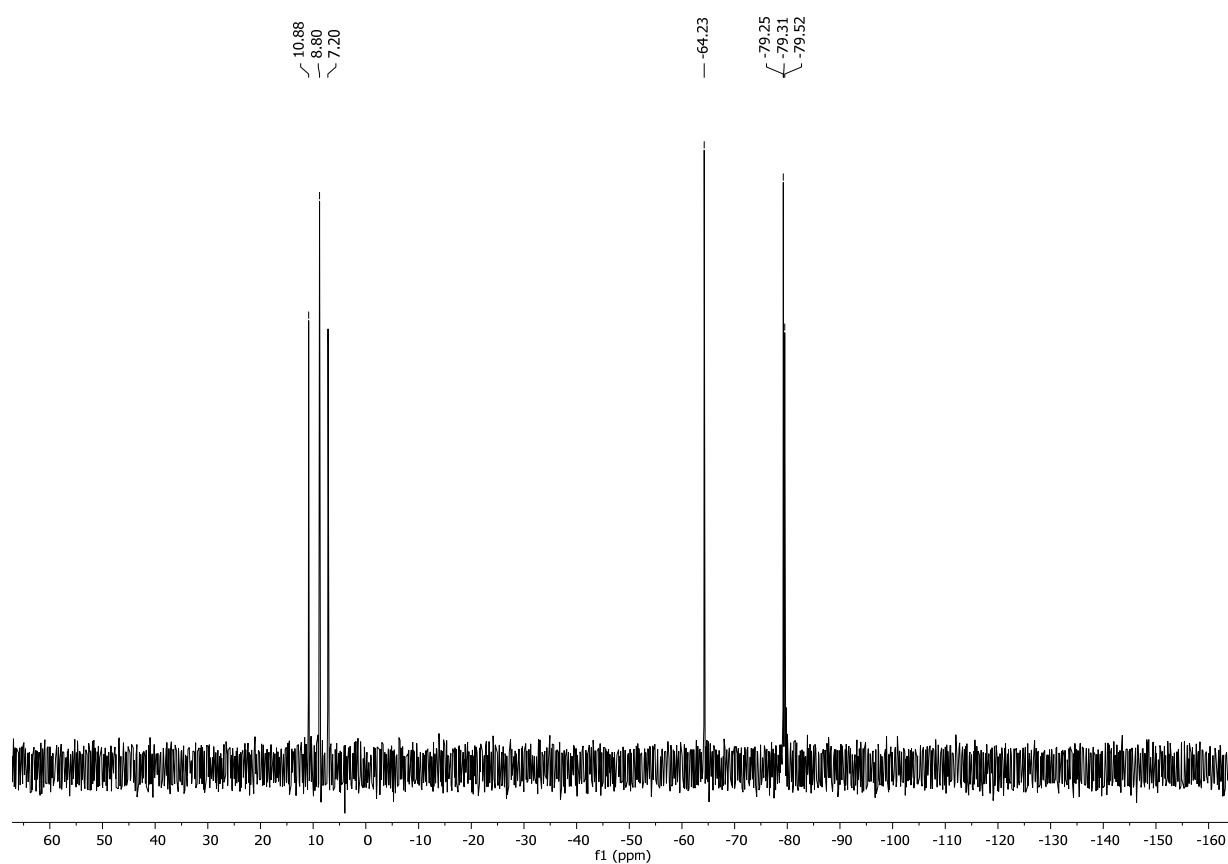
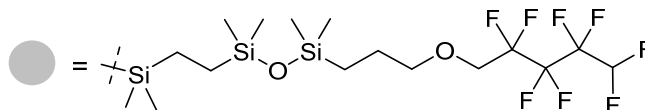


Figure S 33. <sup>29</sup>Si NMR (79.5 MHz, CDCl<sub>3</sub>) spectrum of DDSQ-2OSi(F<sub>3</sub>).

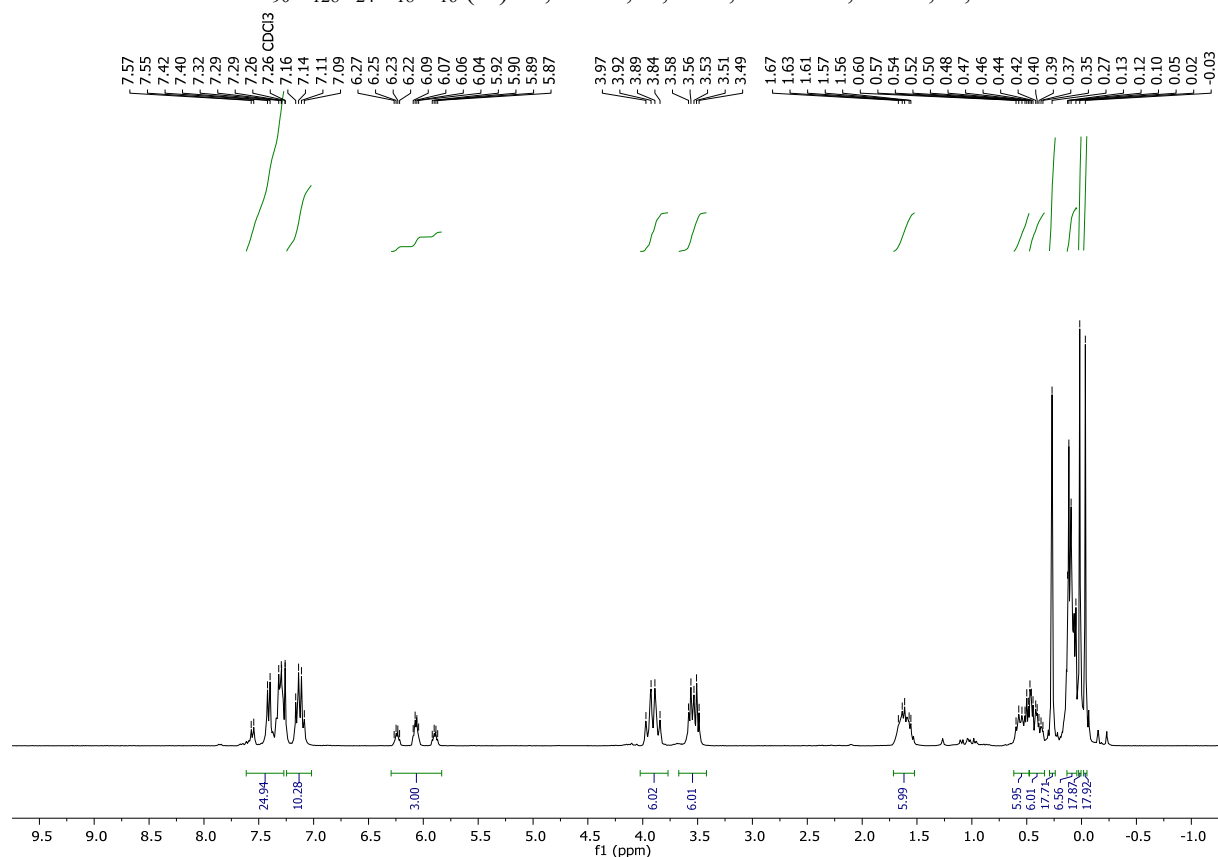
The diagram shows a cage-like structure of silicon (Si) and oxygen (O) atoms. The silicon atoms are arranged in a cage, with oxygen atoms bridging them. Each silicon atom is also bonded to a phenyl (Ph) group. There are three additional grey spheres attached to the cage via oxygen atoms, representing the connection points to the polymer network.



**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, ppm): δ = -0.03 (s, 18H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.02 (s, 18H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.15-0.13 (m, 6H, Si-CH<sub>2</sub>-), 0.27 (s, 18H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.35-0.48 (m, 6H, -CH<sub>2</sub>-Si-), 0.50-0.60 (m, 6H, Si-CH<sub>2</sub>-), 1.56-1.67 (m, 6H, -CH<sub>2</sub>-), 3.53 (dt, *J*<sub>H-H</sub> = 14.0, 6.6 Hz, 6H, -CH<sub>2</sub>-O), 3.91 (q, *J*<sub>H-H</sub> = 13.9 Hz, 6H, -O-CH<sub>2</sub>-), 6.07 (tq, *J*<sub>H-H</sub> = 52.00, 5.0 Hz, 3H, -CH-F<sub>2</sub>), 7.09-7.57 (m, 35H, Ph);

<sup>29</sup>Si NMR (79 MHz, CDCl<sub>3</sub>, ppm): δ = 12.49 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), 8.76 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), 7.17 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), -77.42, -77.91, -78.19 (-Si-Ph).

**EA:** Anal. calcd for  $C_{90}H_{128}F_{24}O_{18}Si_{16}$  (%): C, 44.98; H, 5.37; found: C, 45.11; H, 5.38.



S-26-

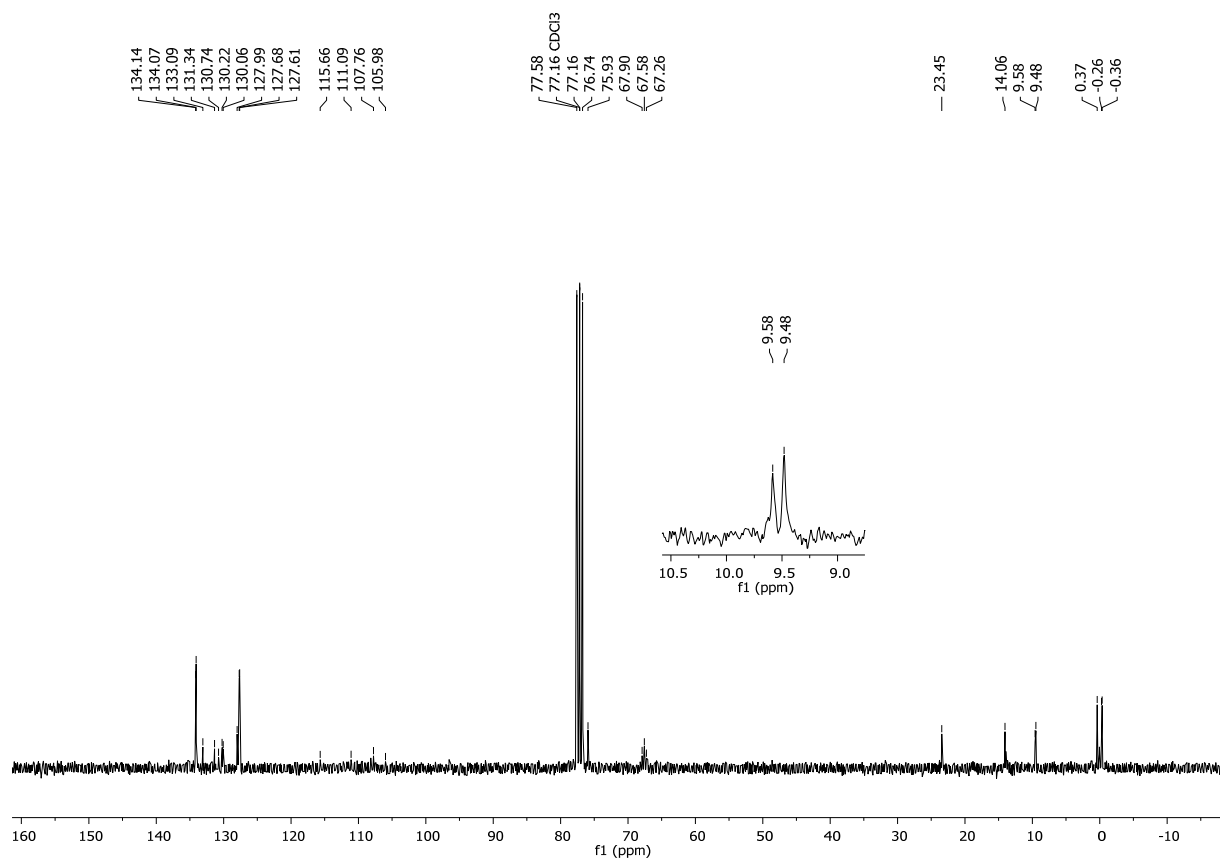


Figure S 35.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectrum of **triT<sub>7</sub>(F3)**.

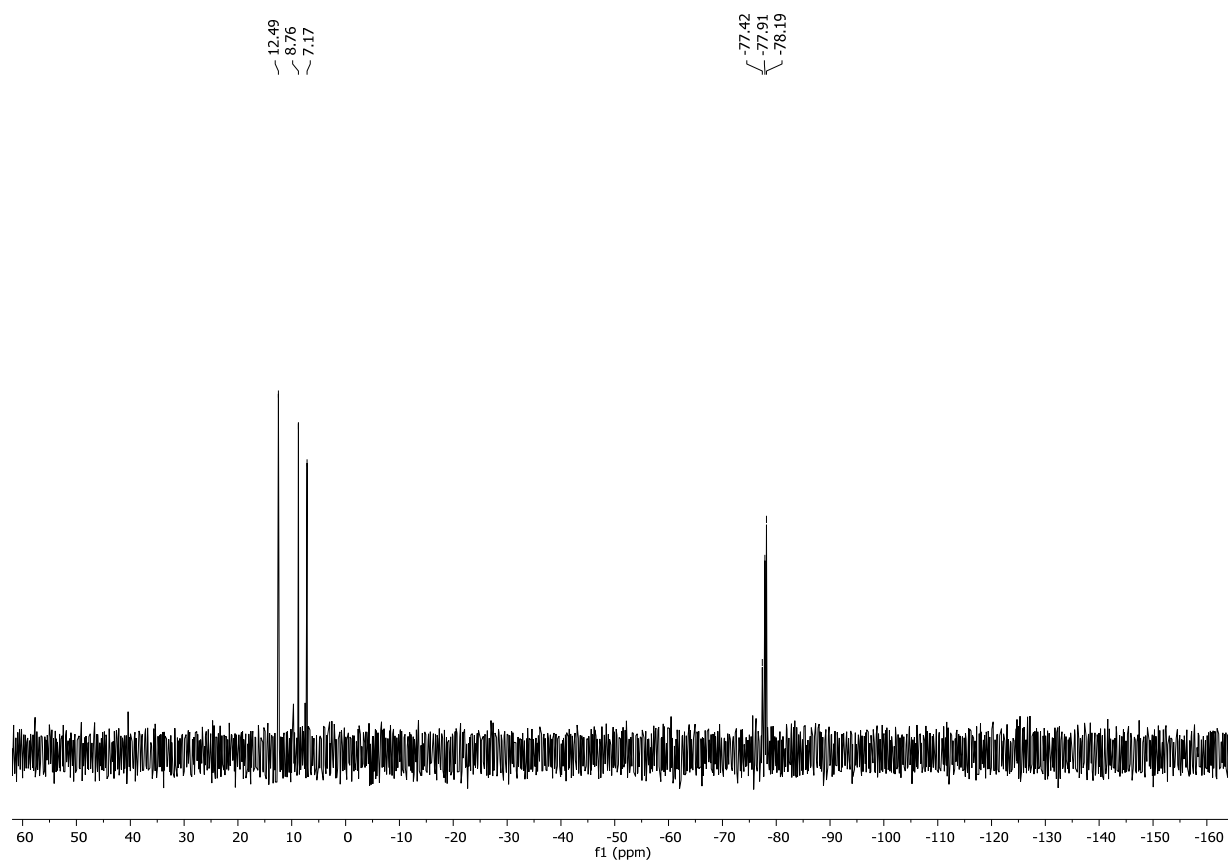
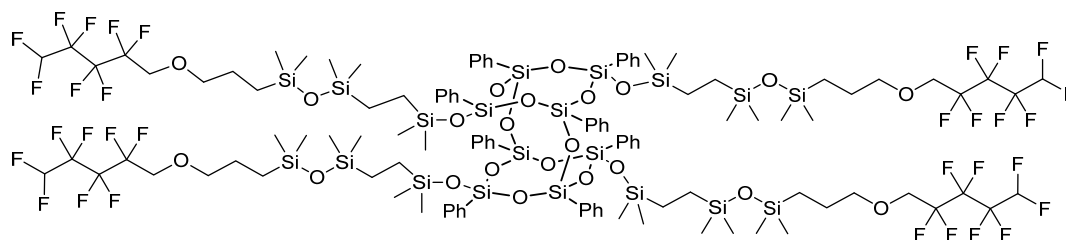


Figure S 36.  $^{29}\text{Si}$  NMR (79.5 MHz,  $\text{CDCl}_3$ ) spectrum of **triT<sub>7</sub>(F3)**.

## DDSQ-4Si(F3)



Waxy, white solid. Isolated Yield 94%

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>, ppm): δ = -0.22 (d, *J*<sub>H-H</sub> = 1.8 Hz, 24H, Si(CH<sub>3</sub>)<sub>2</sub>), -0.05 (d, *J*<sub>H-H</sub> = 1.6 Hz, 24H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.04 (d, *J*<sub>H-H</sub> = 1.8 Hz, 24H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.09-0.13 (m, 8H, Si-CH<sub>2</sub>-), 0.27-0.31 (m, 8H, -CH<sub>2</sub>-Si-), 0.39-0.43 (m, 8H, Si-CH<sub>2</sub>-), 1.50-1.69 (m, 8H, -CH<sub>2</sub>-), 3.49-3.58 (m, 8H, -CH<sub>2</sub>-O), 3.88 (t, *J*<sub>H-H</sub> = 14.7 Hz, 8H, -O-CH<sub>2</sub>-), 6.06 (tt, *J*<sub>H-H</sub> = 52.10, 5.6 Hz, 4H, -CH-F<sub>2</sub>), 7.13-7.47 (m, 40H, Ph);

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>, ppm): δ = -0.62 (-Si(CH<sub>3</sub>)<sub>2</sub>), -0.48 (Si(CH<sub>3</sub>)<sub>2</sub>), 0.30 (-Si(CH<sub>3</sub>)<sub>2</sub>), 9.55 (-Si-CH<sub>2</sub>-), 9.58 (-CH<sub>2</sub>-Si), 14.05 (-Si-CH<sub>2</sub>-), 23.44 (-CH<sub>2</sub>-), 67.44-67.77 (-CH<sub>2</sub>-O-), 75.94 (O-CH<sub>2</sub>-), 106.13 (-CF<sub>2</sub>-), 107.82 (-CF<sub>2</sub>-), 111.09 (-CHF<sub>2</sub>-), 115.69 (-CF<sub>2</sub>-), 127.59-127.62 (Ph), 129.89 (Ph), 130.25 (Ph), 131.95 (Ph), 133.28 (Ph), 134.28-134.50 (Ph);

**<sup>29</sup>Si NMR** (79 MHz, CDCl<sub>3</sub>, ppm): δ = 6.96 (-Si-(Me)<sub>2</sub>-), 8.71 (-Si-(Me)<sub>2</sub>-), 11.76 (-Si-(Me)<sub>2</sub>-), -76.10, -78.67 (-Si-Ph);

**IR** (ATR, cm<sup>-1</sup>): 3074.38, 3054.72 (C-H phenyl), 2957.00, 2924.57 (C-H), 1594.42, 1430.50 (C=C phenyl), 1252.59 (Si-C), 1170.08 (C-F), 1128.29, 1041.47 (Si-O-Si), 998.47 (C-H phenyl).

The spectroscopic data are consistent with the literature.[64]

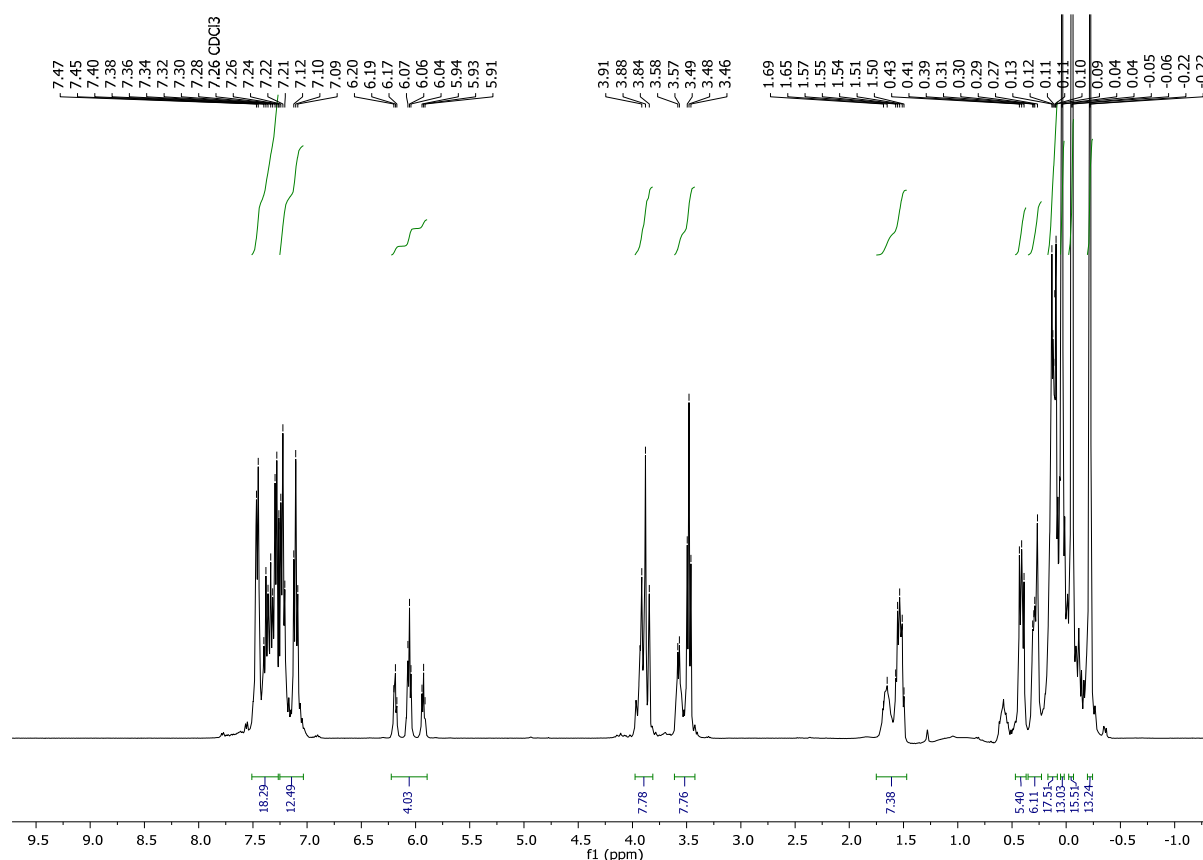


Figure S 37. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) spectrum of DDSQ-4Si(F3).

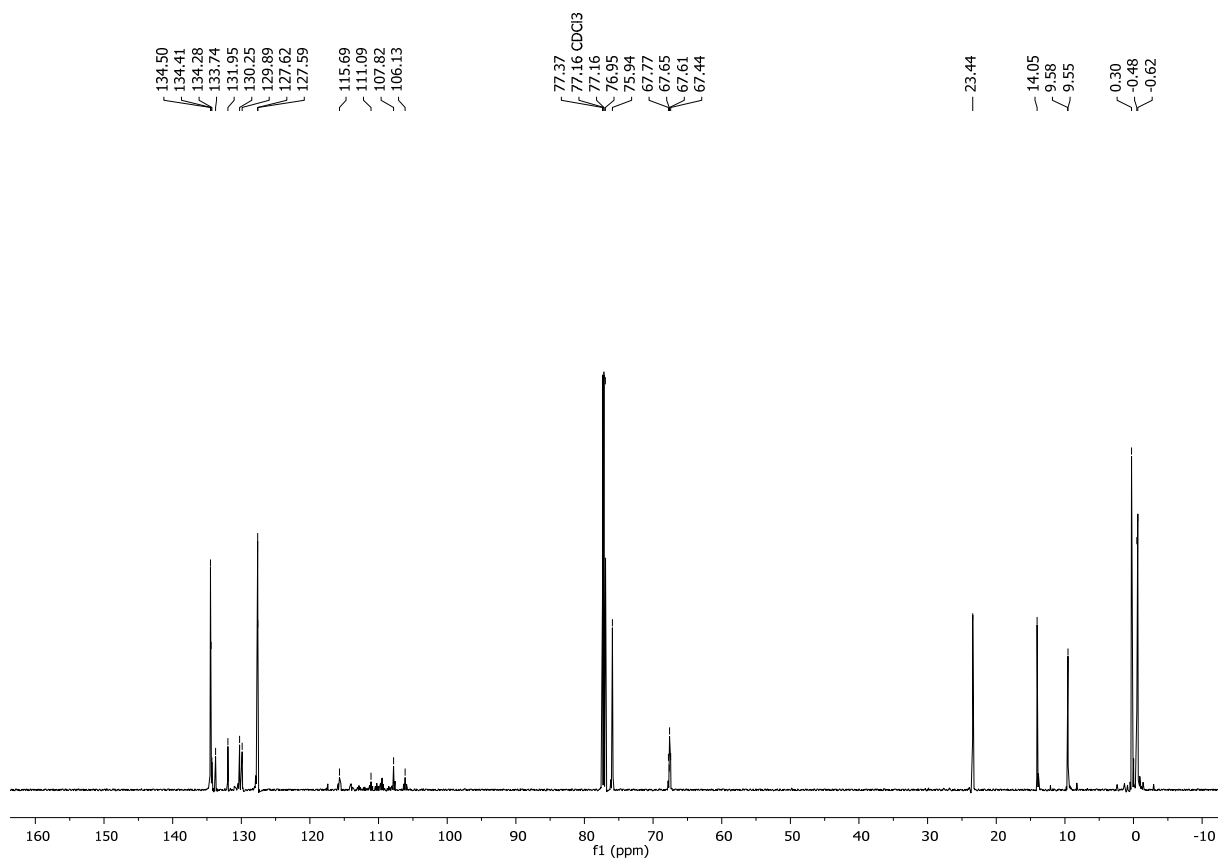


Figure S 38. <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>) spectrum of **DDSQ-4Si(F3)**.

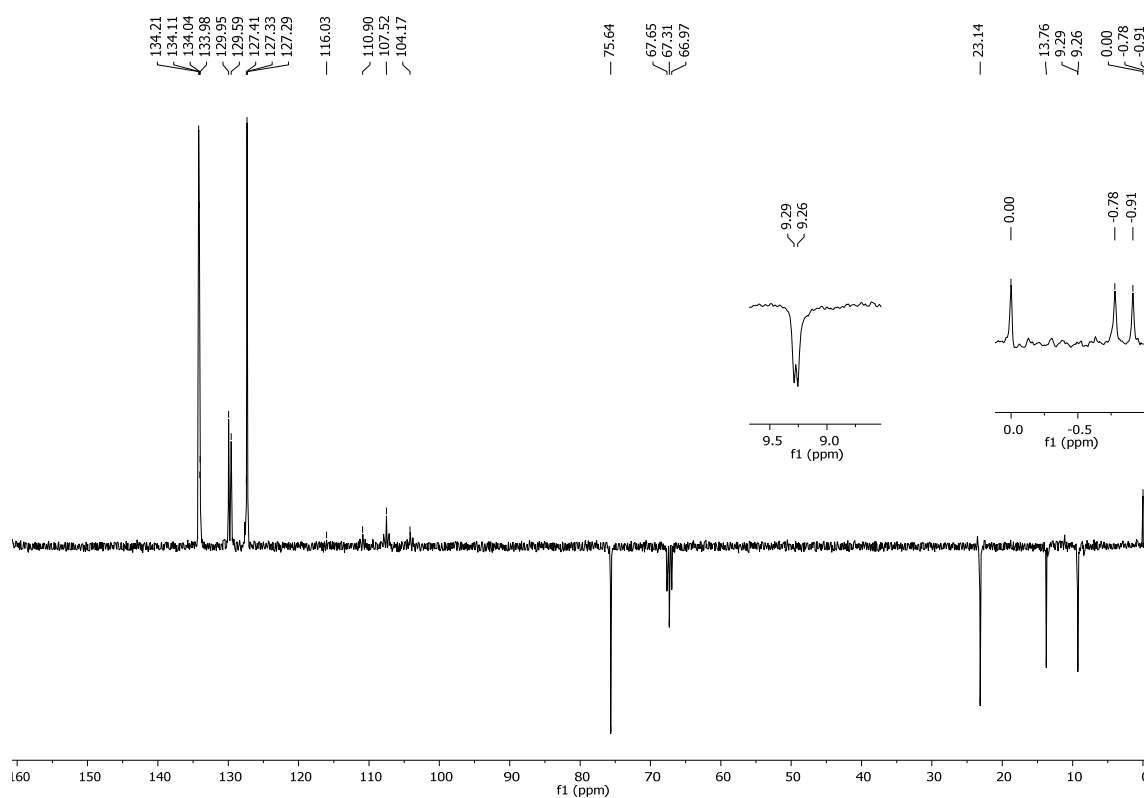


Figure S 39. <sup>13</sup>C DEPT 135 NMR (75.5 MHz, CDCl<sub>3</sub>) spectrum of **DDSQ-4Si(F3)**.

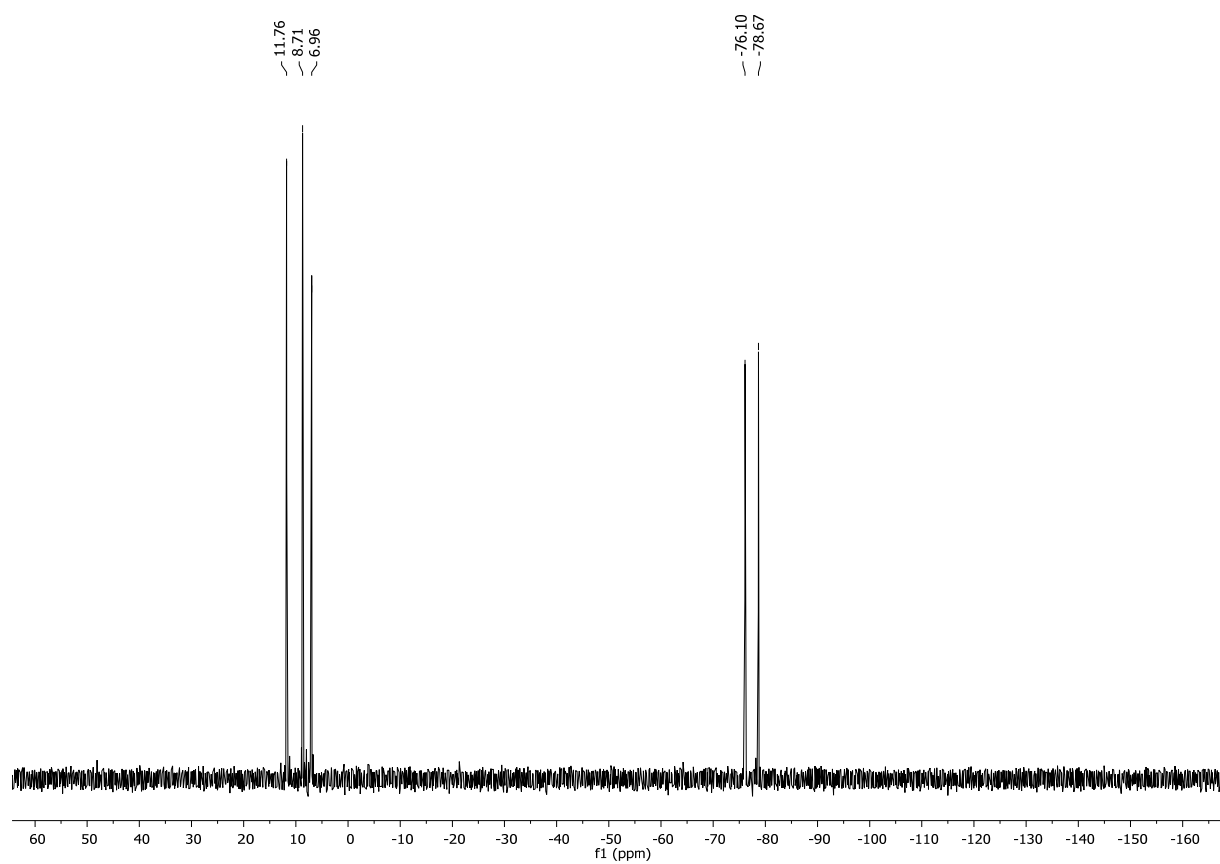
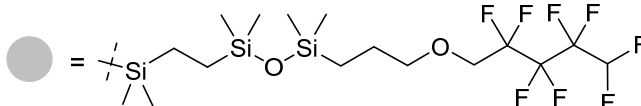
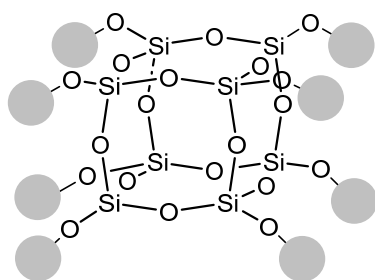


Figure S 40.  $^{29}\text{Si}$  NMR (79.5 MHz,  $\text{CDCl}_3$ ) spectrum of **DDSQ-4Si(F3)**.

**octaT<sub>8</sub>(F3)**

Oil. Isolated Yield 92%

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>, ppm): δ = 0.03 (s, 48H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.05 (s, 48H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.08-0.10 (m, 16H, Si-CH<sub>2</sub>-), 0.12 (s, 48H, Si(CH<sub>3</sub>)<sub>2</sub>), 0.41-0.45 (m, 16H, -CH<sub>2</sub>-Si-), 0.47-0.53 (m, 16H, Si-CH<sub>2</sub>-), 1.56-1.66 (m, 16H, -CH<sub>2</sub>-), 3.54 (t, *J*<sub>H-H</sub> = 6.9 Hz, 16H, -CH<sub>2</sub>-O), 3.91 (t, *J*<sub>H-H</sub> = 14.0 Hz, 16H, -O-CH<sub>2</sub>-), 6.06 (tt, *J*<sub>H-H</sub> = 52.00, 5.6 Hz, 8H, -CH-F<sub>2</sub>);

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>, ppm): δ = -0.96 (-Si(CH<sub>3</sub>)<sub>2</sub>), -0.28 (Si(CH<sub>3</sub>)<sub>2</sub>), 0.37(-Si(CH<sub>3</sub>)<sub>2</sub>), 9.07 (-Si-CH<sub>2</sub>-), 9.36 (-CH<sub>2</sub>-Si), 14.07 (-Si-CH<sub>2</sub>-), 23.46 (-CH<sub>2</sub>-), 67.24-67.90 (-CH<sub>2</sub>-O-), 75.94 (O-CH<sub>2</sub>-), 107.73 (-CF<sub>2</sub>-), 111.11 (-CF<sub>2</sub>-), 116.04 (-CHF<sub>2</sub>-), 119.05 (-CF<sub>2</sub>-);

**<sup>29</sup>Si NMR** (79 MHz, CDCl<sub>3</sub>, ppm): δ = 7.26 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), 8.70 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), 13.24 (-Si-(CH<sub>3</sub>)<sub>2</sub>-), -108.79 (-Si-);

**IR** (ATR, cm<sup>-1</sup>): 2956.83, 2910.07 (C-H), 1252.75 (Si-C), 1167.60 (C-F), 1131.30, 1045.49 (Si-O-Si).

**EA:** Anal. calcd for C<sub>12</sub>H<sub>24</sub>F<sub>64</sub>O<sub>36</sub>Si<sub>32</sub> (%): C, 34.33; H, 5.58; found: C, 34.43; H, 5.59.

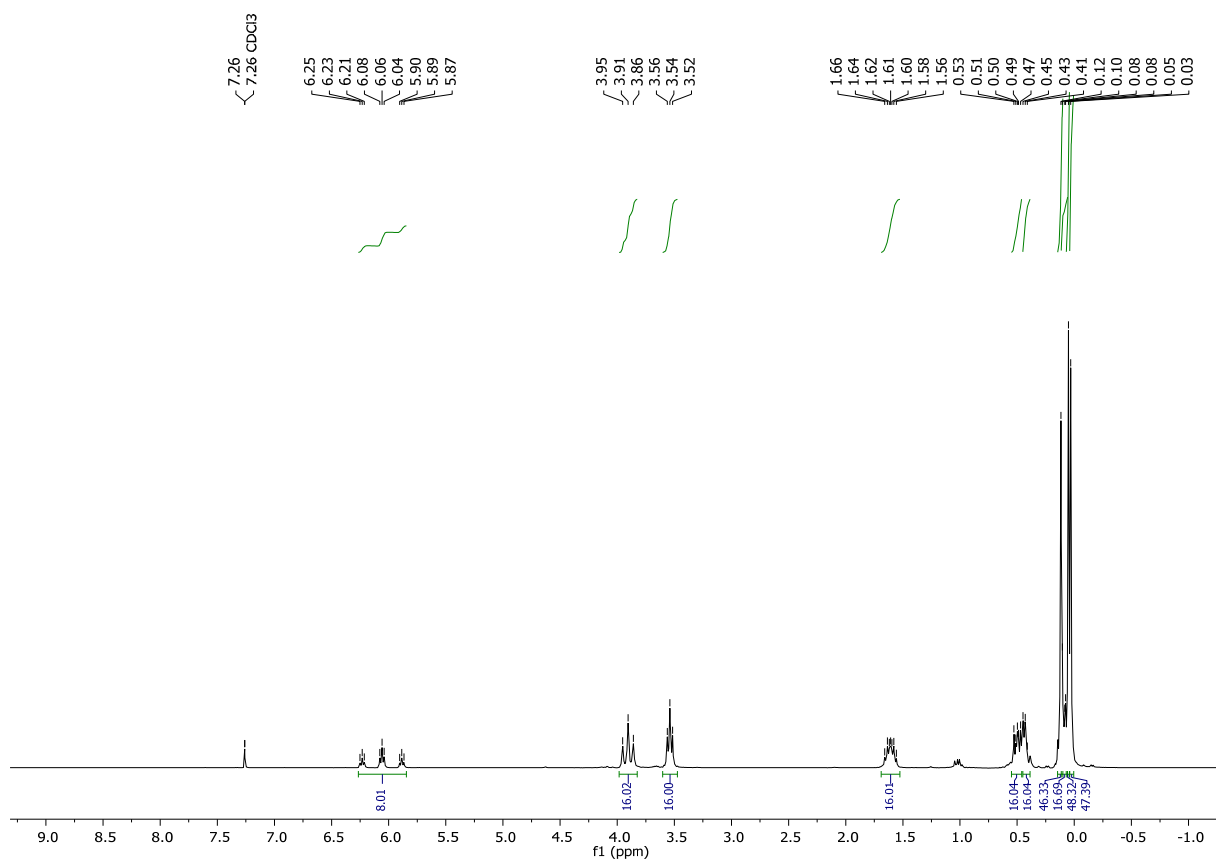


Figure S 41. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) spectrum of octaT<sub>8</sub>(F3).

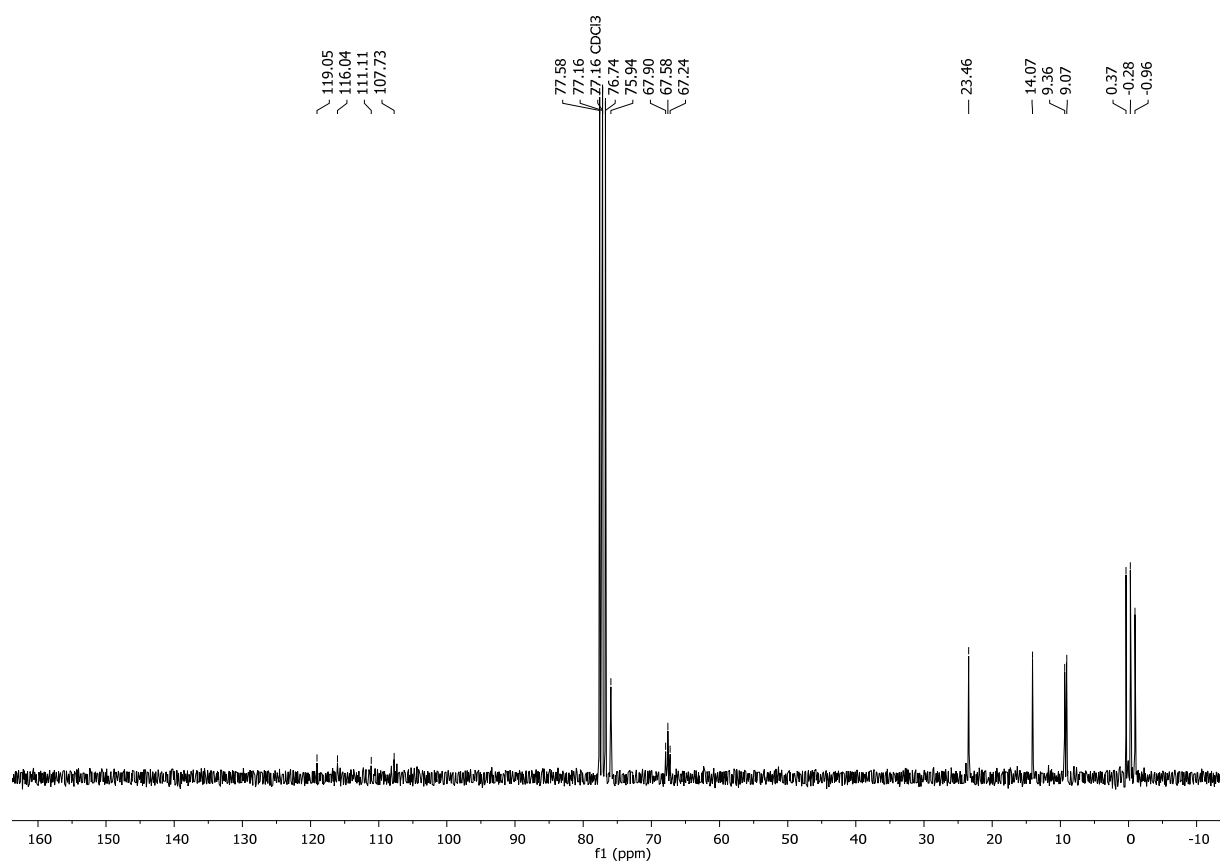


Figure S 42.  $^{13}\text{C}$  NMR (75.5 MHz,  $\text{CDCl}_3$ ) spectrum of **octaT<sub>8</sub>(F3)**.

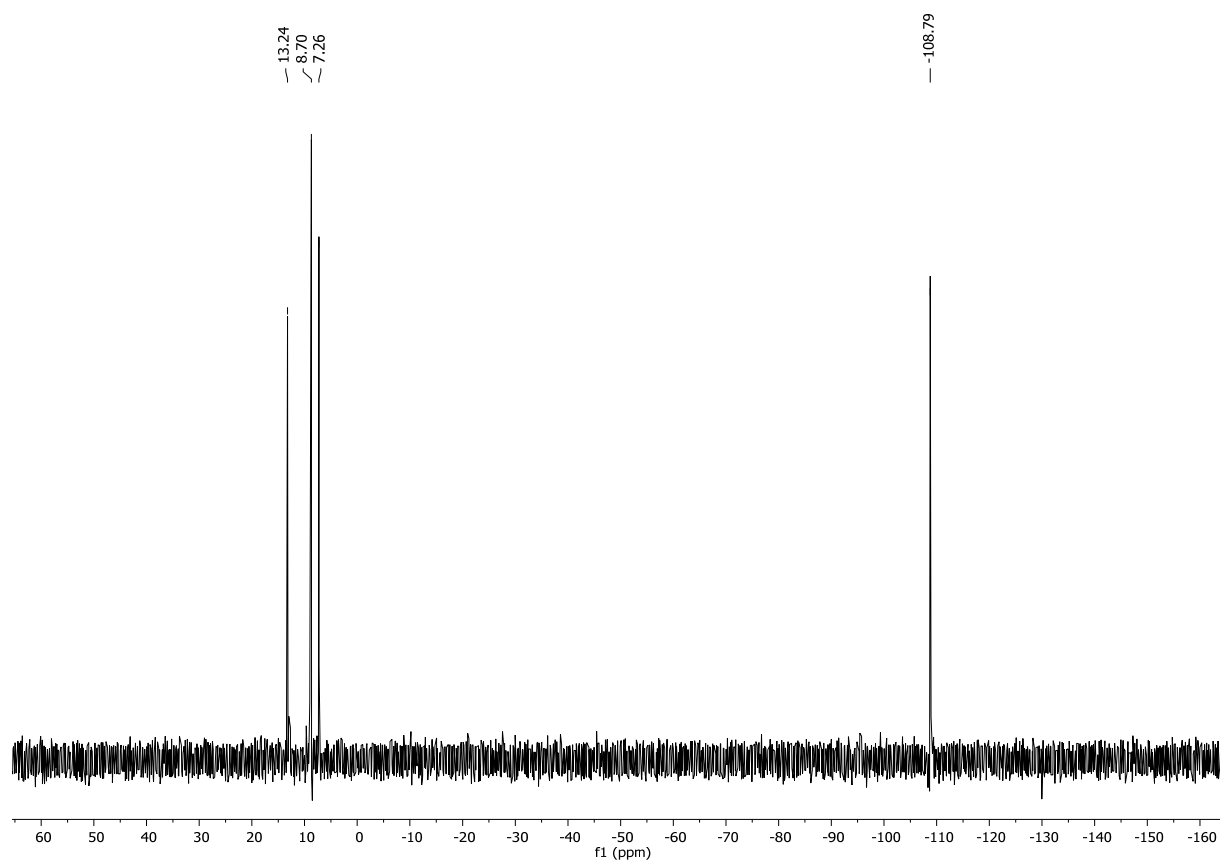


Figure S 43.  $^{29}\text{Si}$  NMR (79.5 MHz,  $\text{CDCl}_3$ ) spectrum of **octaT<sub>8</sub>(F3)**.



### 3. Additional $^{29}\text{Si}$ NMR spectra comparisons

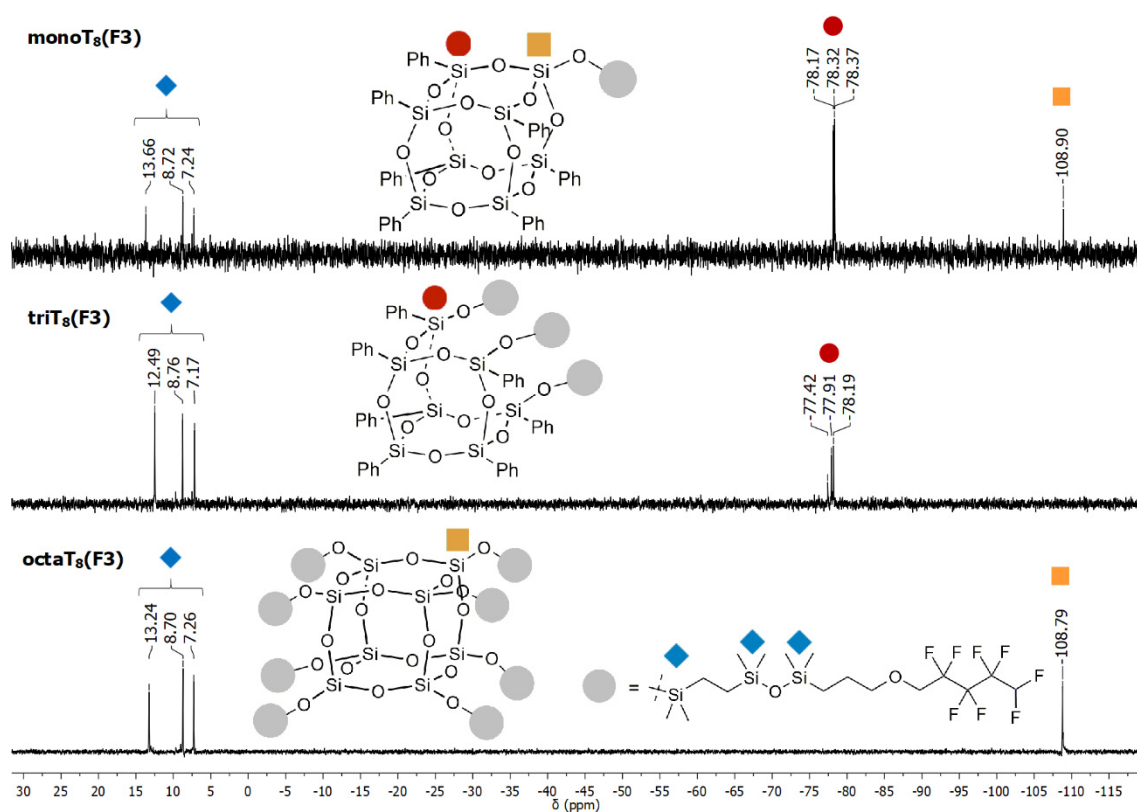


Figure S 44. Selected range of stacked  $^{29}\text{Si}$ -NMR spectra of **monoT<sub>8</sub>(F<sub>3</sub>)**, **triT<sub>8</sub>(F<sub>3</sub>)** and **octaT<sub>8</sub>(F<sub>3</sub>)**.

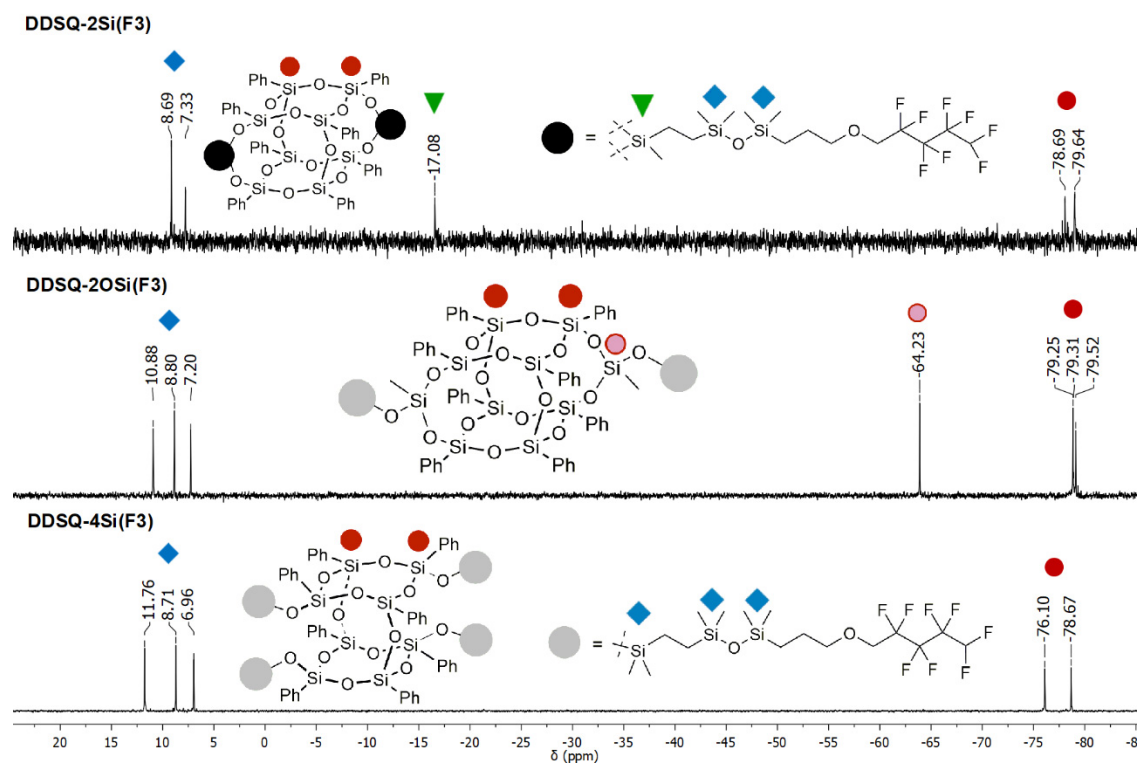


Figure S 45. Selected range of stacked  $^{29}\text{Si}$ -NMR spectra of **DDSQ-2Si(F<sub>3</sub>)**, **DDSQ-20Si(F<sub>3</sub>)** and **DDSQ-4Si(F<sub>3</sub>)**.

## 4. Dielectric Relaxation Spectra and Tables

**Table S2.** Values of dielectric permittivity, measured at -100°C at  $10^0 \times 10^6$  Hz

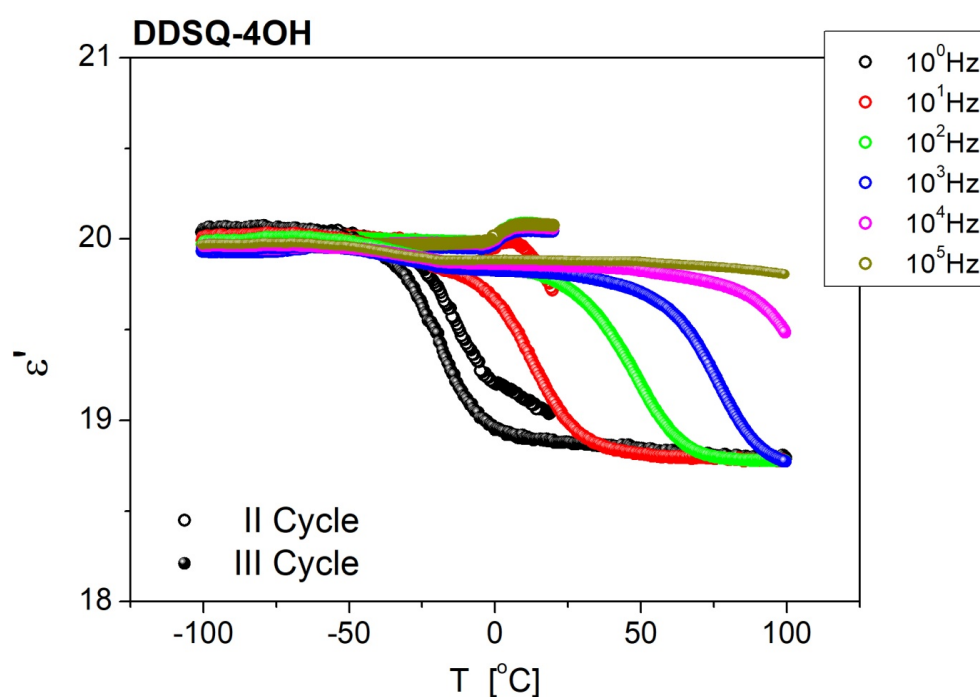
Sample	$\epsilon$ at -100 °C						
	1 Hz	10 Hz	100 Hz	1 kHz	10 kHz	100kHz	1 MHz
DDSQ-4OH	20.01	19.94	19.96	19.93	19.95	19.96	20.10
DDSQ-2SiVi	19.86	19.91	19.93	19.92	19.94	19.97	20.11
DDSQ-2SiH	17.36	17.35	17.36	17.36	17.37	17.37	17.44
DDSQ-2Si(F1)	18.26	18.23	18.17	18.05	17.89	17.67	17.52
DDSQ-2Si(F3)	15.71	16.08	16.12	16.09	16.09	16.11	16.30
DDSQ-2Si(F1)-f*	3.06	3.06	3.05	2.73	1.94	1.80	1.80
DDSQ-2Si(F3)-f*	2.86	2.86	2.84	2.82	2.80	2.78	2.76

For dielectric measurements, the powder material was formed into the form of pellet. \*Thin films in one layer prepared by a spin-coating technique using 5-10 wt% solutions of co-polymers in DCM (or DCM/THF (1:1)) deposited on the still plate.

**Table S3.** Values of dielectric permittivity, measured at 85°C at  $10^0 \times 10^6$  Hz.

Sample	$\epsilon$ at 85 °C						
	1 Hz	10 Hz	100 Hz	1 kHz	10 kHz	100kHz	1 MHz
DDSQ-4OH	18.80	18.77	18.77	18.82	19.65	19.85	20.06
DDSQ-2SiVi	18.99	18.94	18.96	18.93	18.95	19.29	22.58
DDSQ-2SiH	16.58	16.56	16.82	16.94	17.29	16.82	17.44
DDSQ-2Si(F1)	29.91	25.11	22.74	22.06	21.99	22.74	22.58
DDSQ-2Si(F3)	21.40	19.82	19.57	19.48	19.79	20.05	20.25
DDSQ-2Si(F1)-f*	3.03	3.03	2.66	1.95	1.84	1.83	1.83
DDSQ-2Si(F3)-f*	5.24	4.96	4.63	4.62	4.62	4.98	4.92

For dielectric measurements, the powder material was formed into the form of pellet. \*Thin films in one layer prepared by a spin-coating technique using 5-10 wt% solutions of co-polymers in DCM (or DCM/THF (1:1)) deposited on the still plate.



**Figure S 46.** Temperature dependence of dielectric permittivity, measured for **DDSQ-4OH** sample.

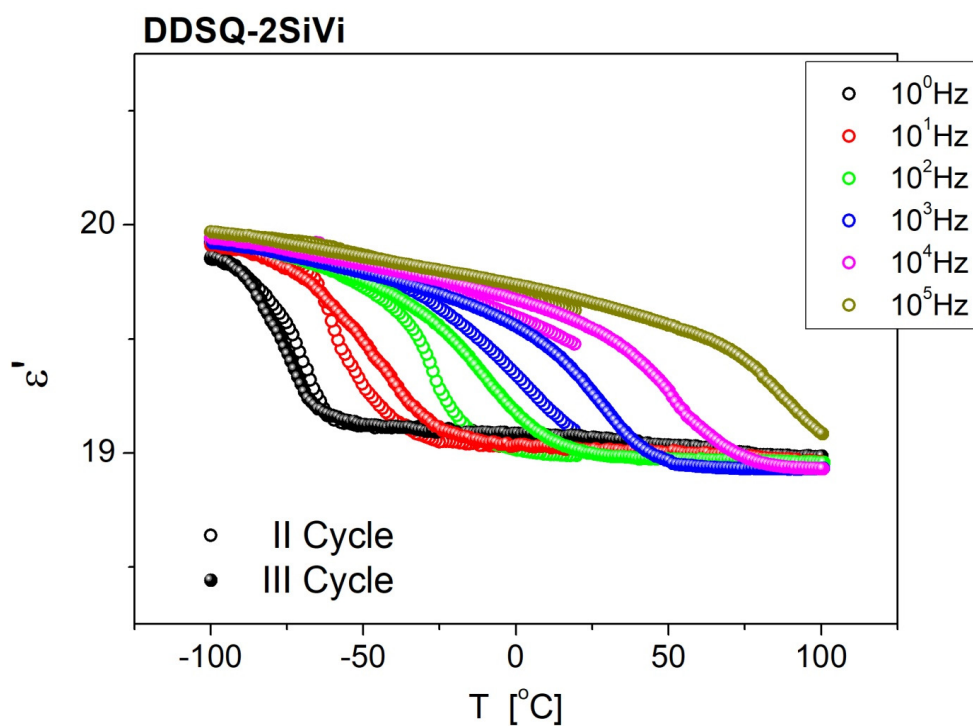


Figure S 47. Temperature dependence of dielectric permittivity, measured for **DDSQ-2SiVi** sample.

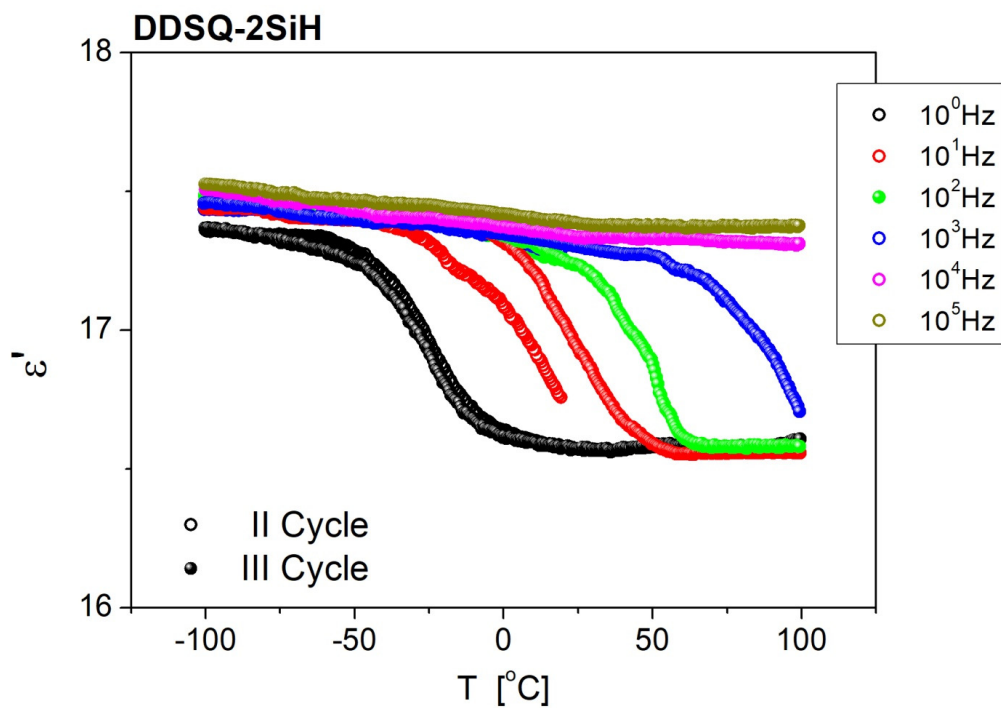


Figure S 48. Temperature dependence of dielectric permittivity, measured for **DDSQ-2SiH** sample.

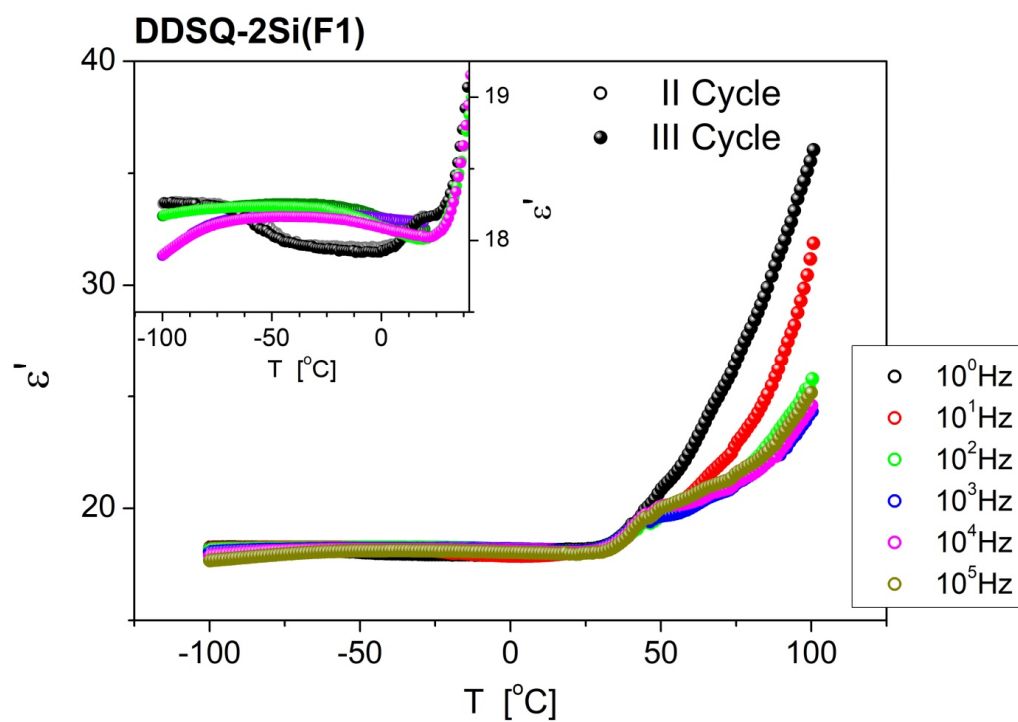


Figure S 49. Temperature dependence of dielectric permittivity, measured for **DDSQ-2Si(F1)** sample.

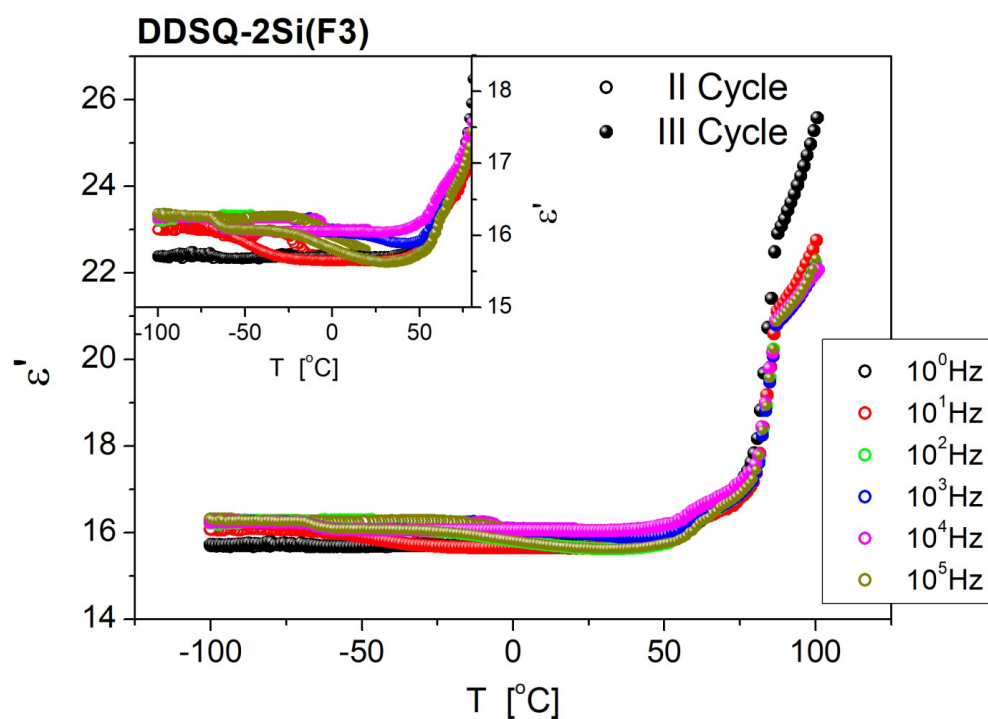


Figure S 50. Temperature dependence of dielectric permittivity, measured for **DDSQ-2Si(F3)** sample.

