

## **Supporting Information**

# **Attempted Synthesis of a Homocyclic Bis(silyl)silylene Leads to the Formation of a Tricyclo[3,1,1,1<sup>2,4</sup>]octasilane**

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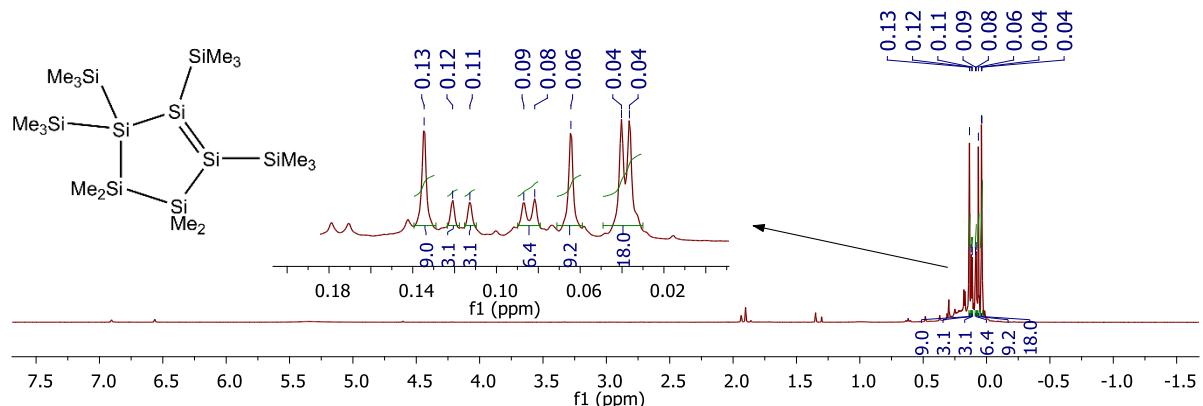
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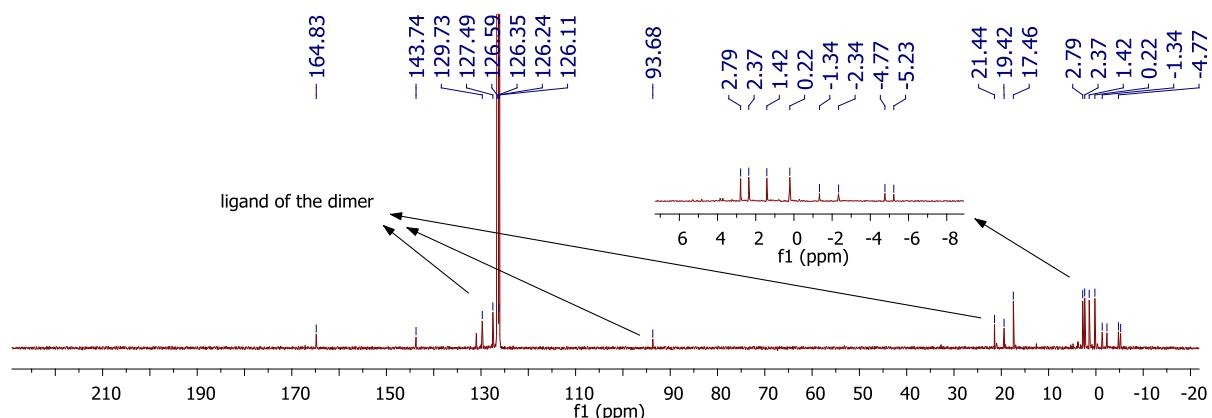
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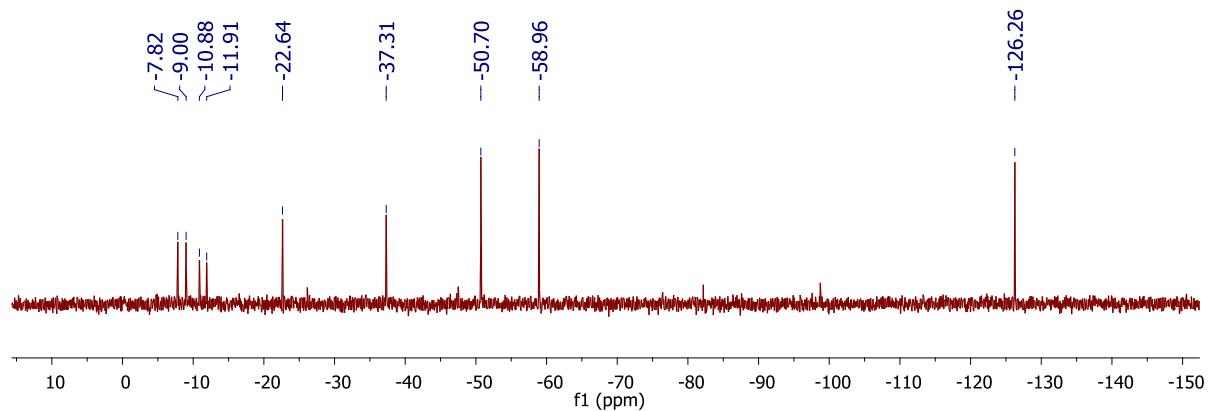
## 1. NMR-Spectroscopy



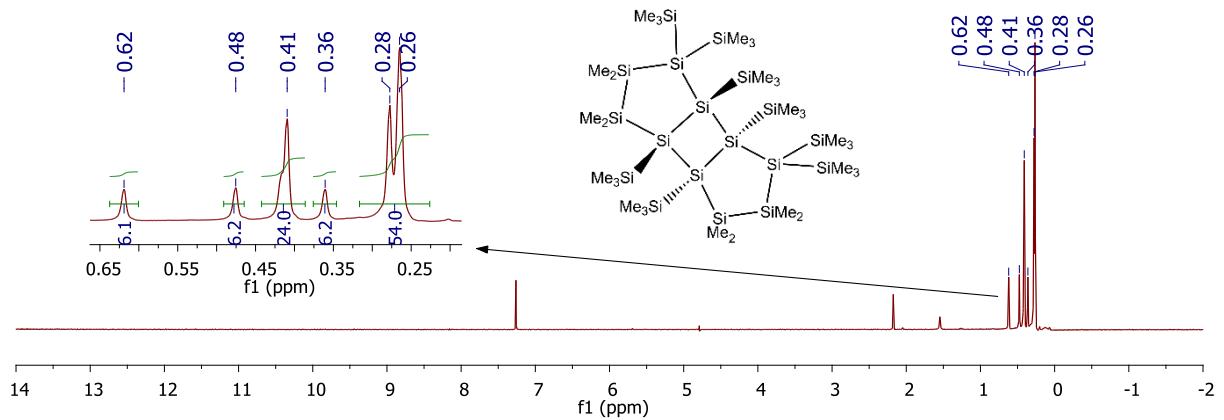
**Figure S1**  $^1\text{H}$ -NMR spectrum of **1** ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



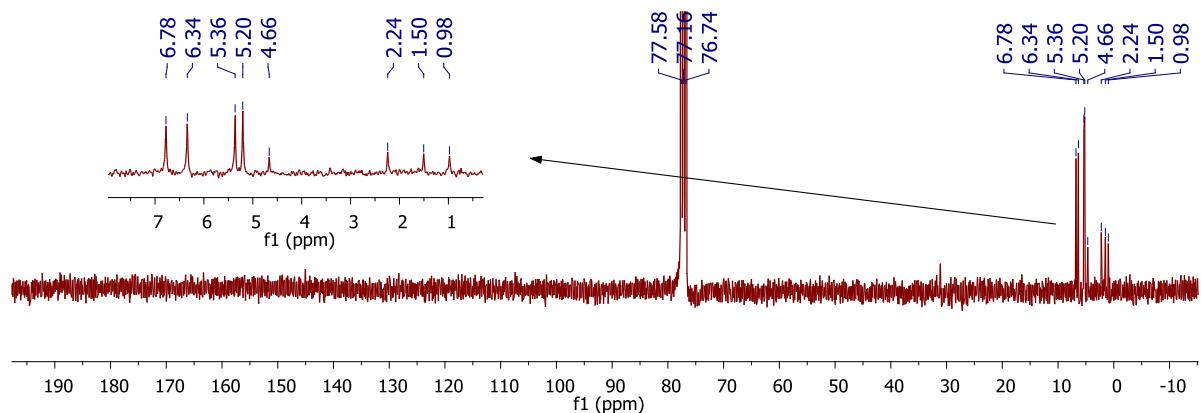
**Figure S2.**  $^{13}\text{C}$ -NMR spectrum of **1** ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



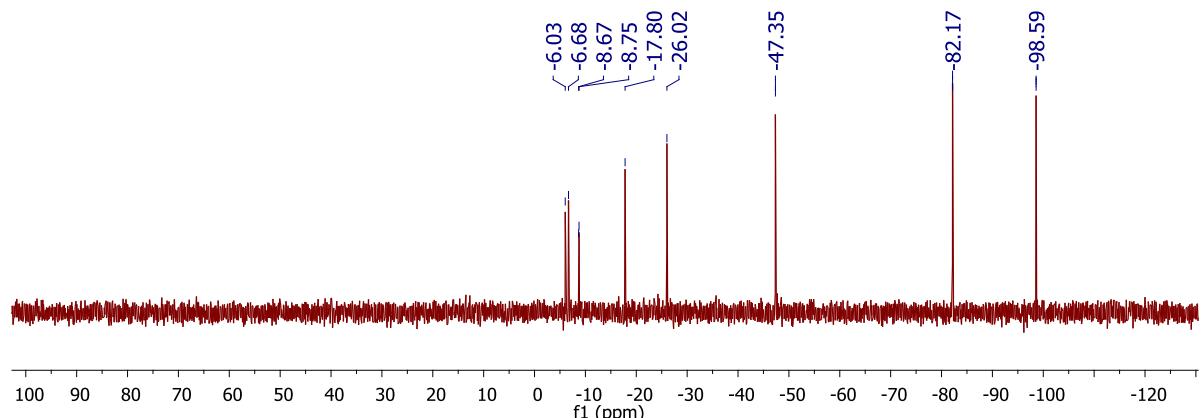
**Figure S3.**  $^{29}\text{Si}$ -NMR spectrum of **1** ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



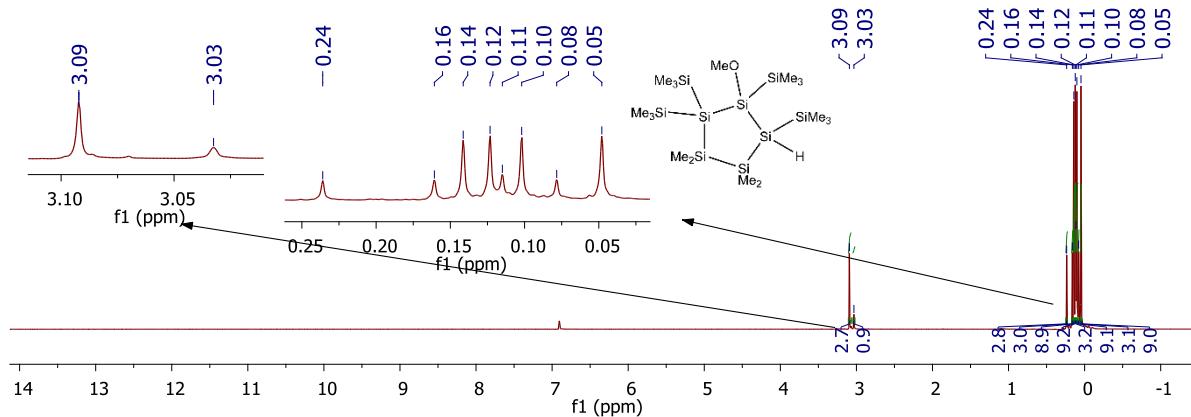
**Figure S4**  $^1\text{H}$ -NMR spectrum of 1-dimer ( $\text{CDCl}_3$  solution, vs. ext. TMS, ppm)



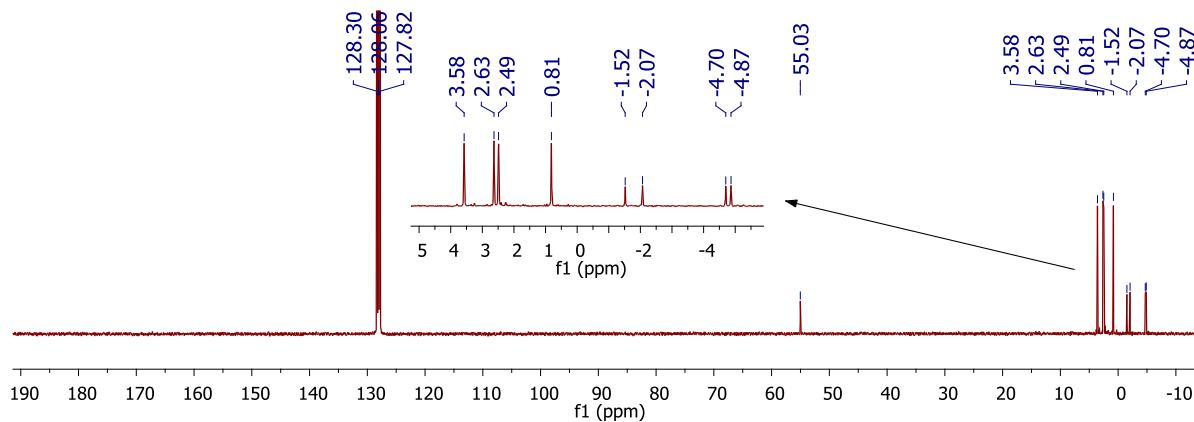
**Figure S5.**  $^{13}\text{C}$ -NMR spectrum of 1-dimer ( $\text{CDCl}_3$  solution, vs. ext. TMS, ppm)



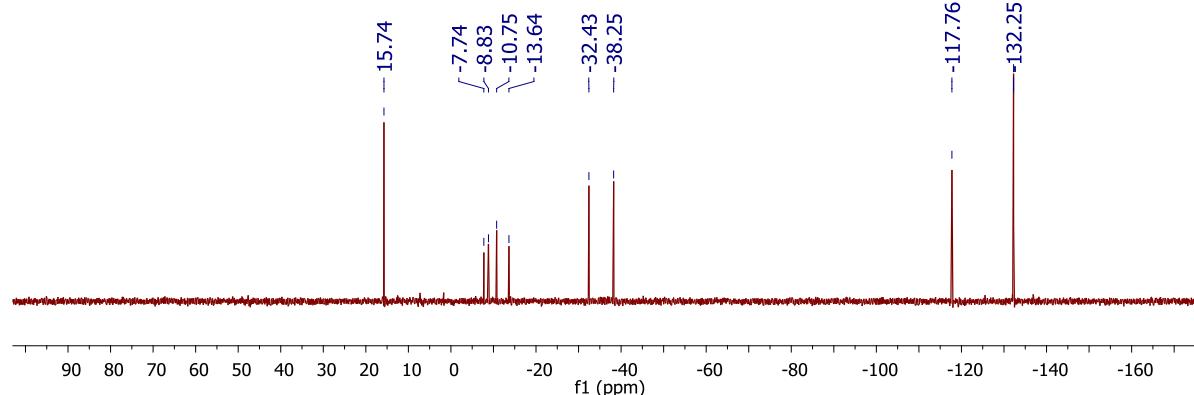
**Figure S6.**  $^{29}\text{Si}$ -NMR spectrum of 1-dimer ( $\text{CDCl}_3$  solution, vs. ext. TMS, ppm)



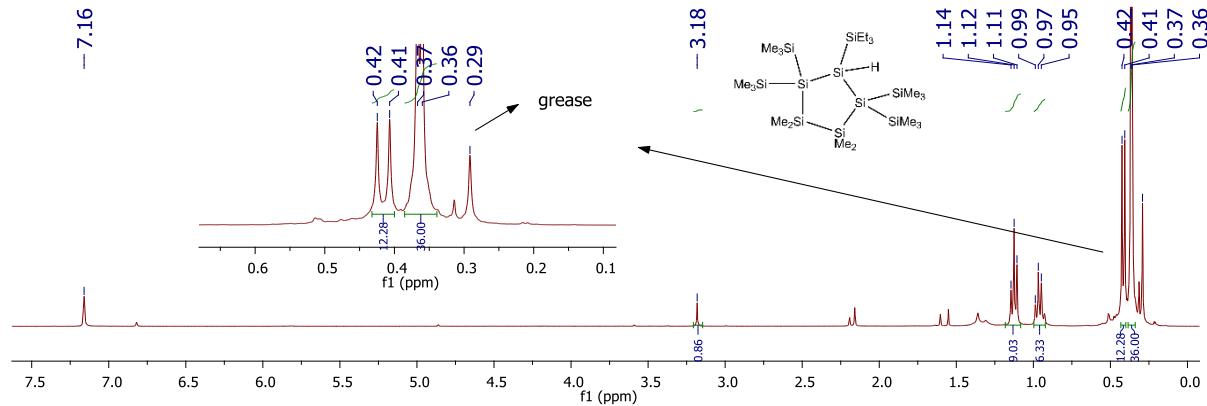
**Figure S7.**  $^1\text{H}$ -NMR spectrum of **2** ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



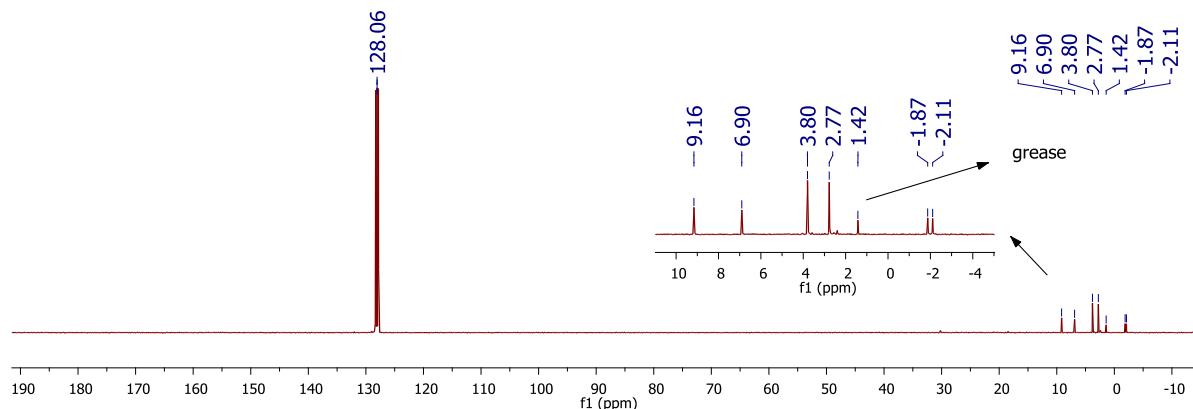
**Figure S8.**  $^{13}\text{C}$ -NMR spectrum of **2** ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



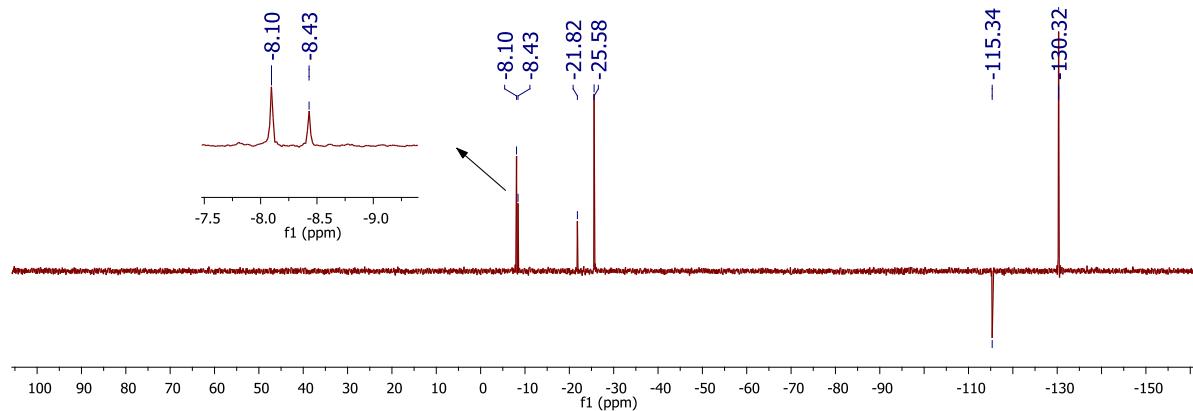
**Figure S9.**  $^{29}\text{Si}$ -NMR spectrum of **2** ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



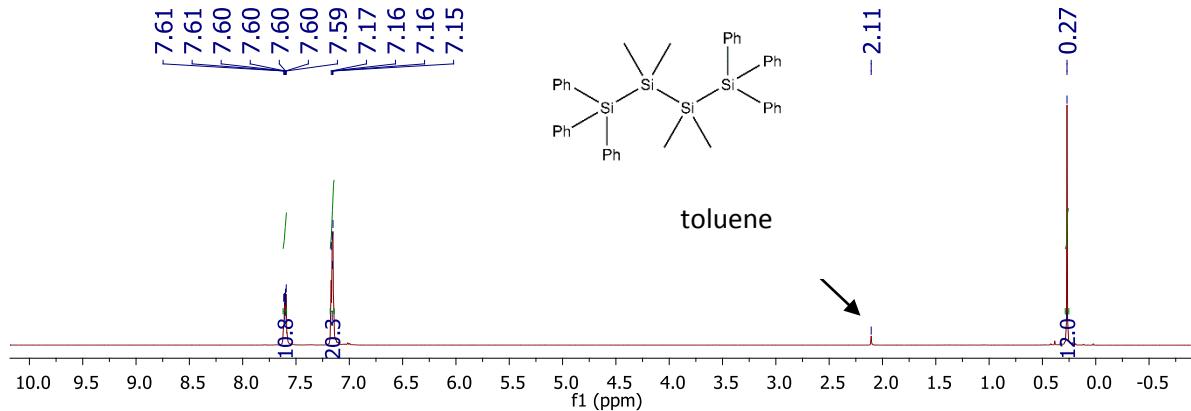
**Figure S10.**  $^1\text{H}$ -NMR spectrum of 3 ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



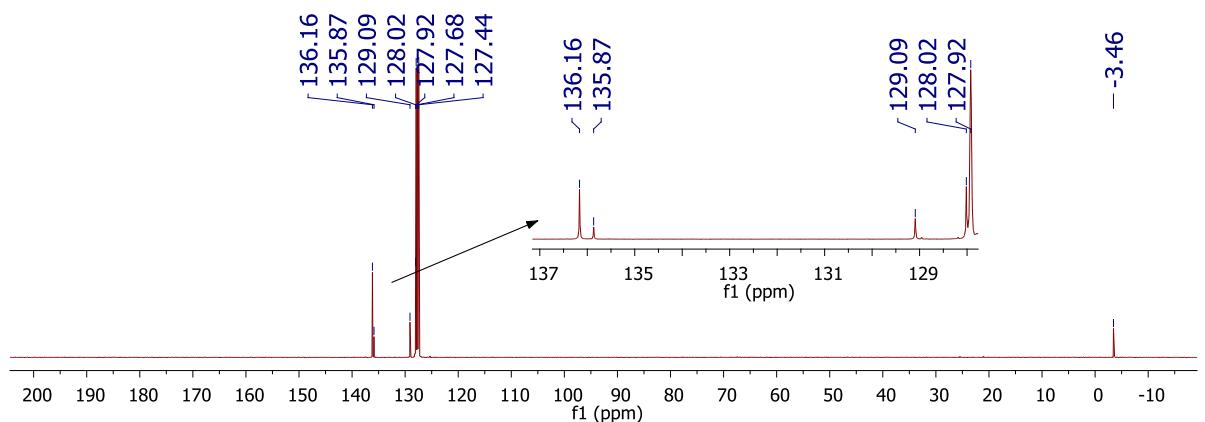
**Figure S11.**  $^{13}\text{C}$ -NMR spectrum of 3 ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



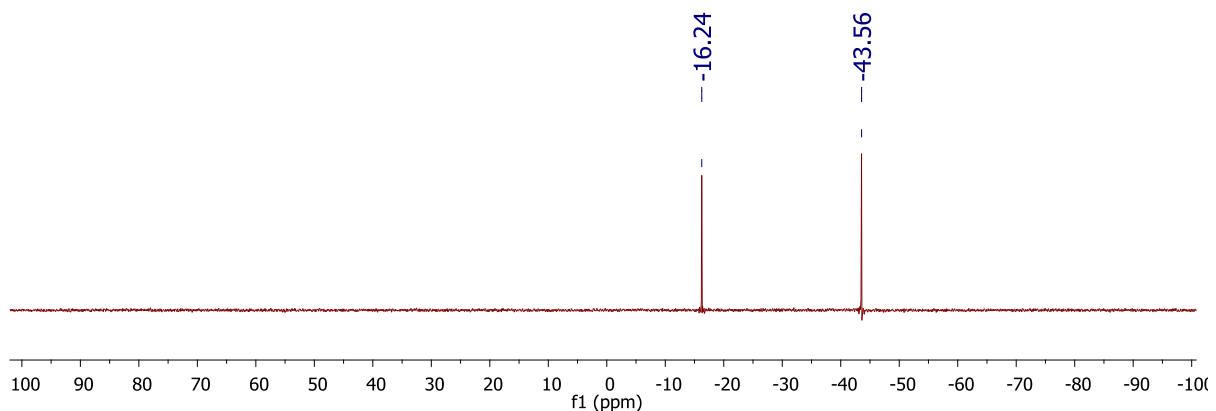
**Figure S12.**  $^{29}\text{Si}$ -NMR spectrum of 3 ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



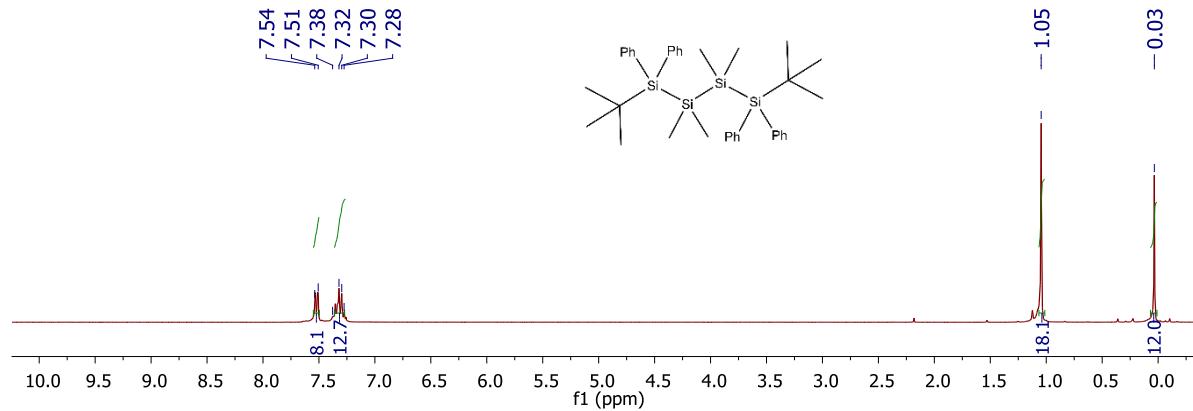
**Figure S13.**  $^1\text{H}$ -NMR spectrum of 4 ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



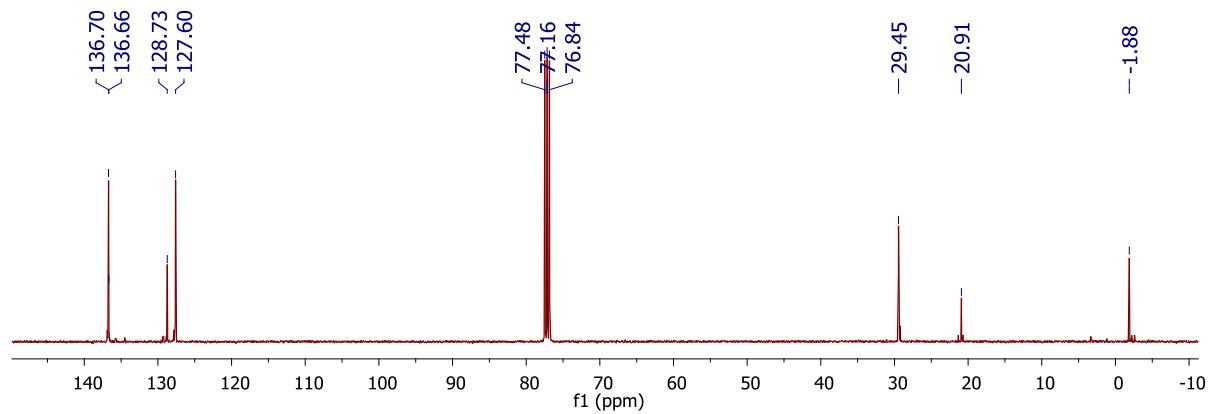
**Figure S14.**  $^{13}\text{C}$ -NMR spectrum of 4 ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



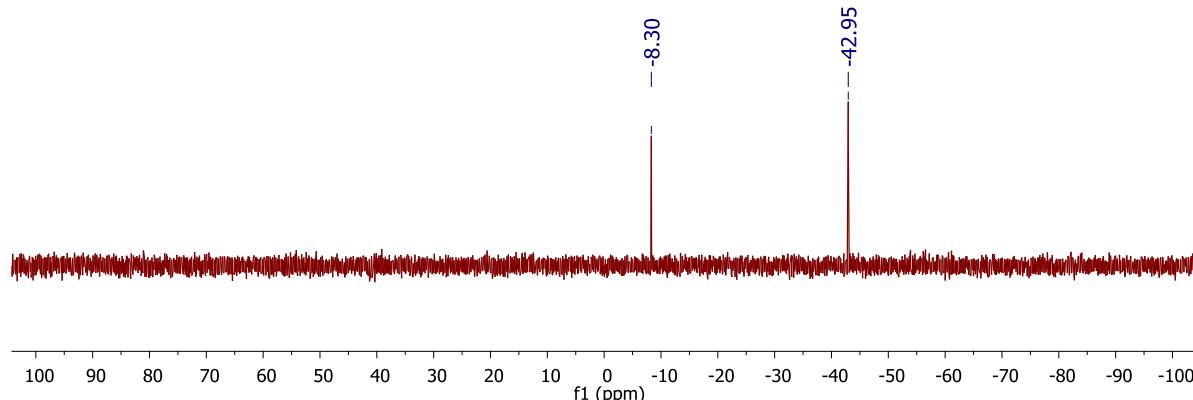
**Figure S15.**  $^{29}\text{Si}$ -NMR spectrum of 4 ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



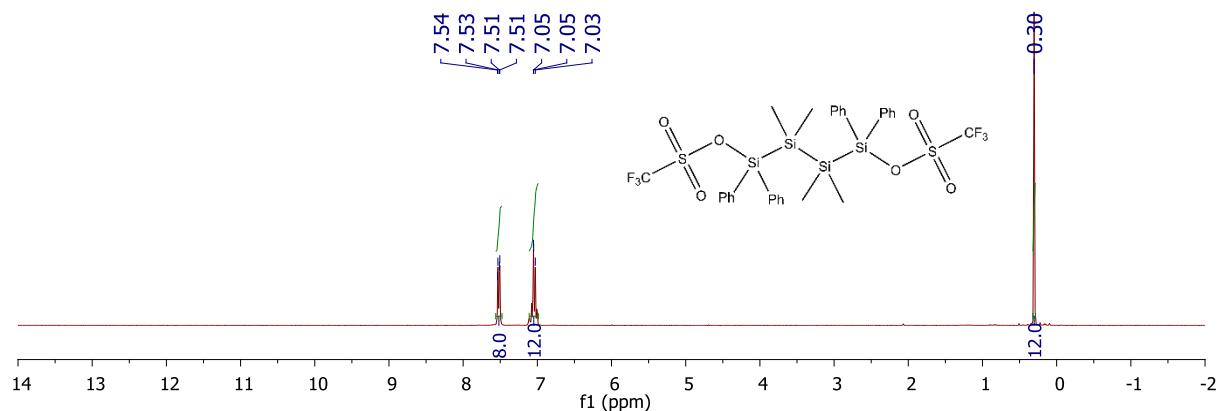
**Figure S16.**  $^1\text{H}$ -NMR spectrum of 5 ( $\text{CDCl}_3$  solution, vs. ext. TMS, ppm)



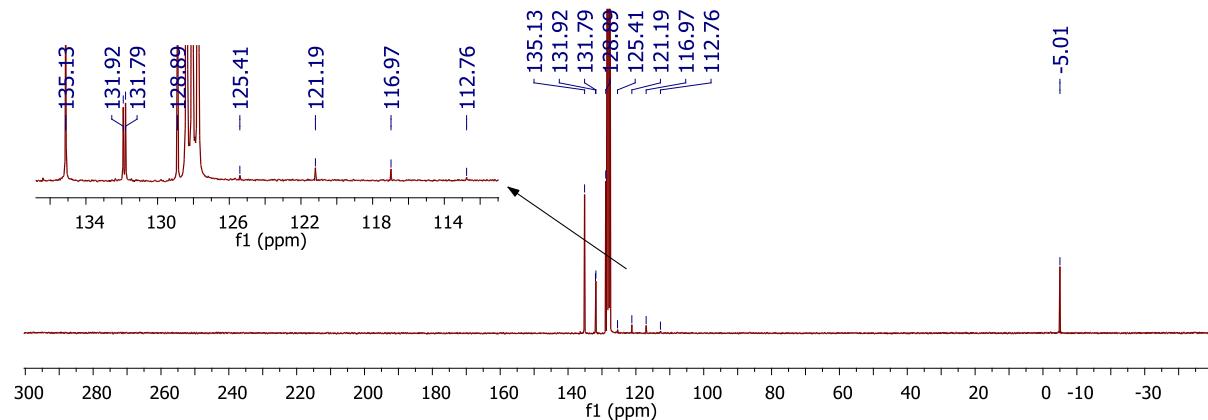
**Figure S17.**  $^{13}\text{C}$ -NMR spectrum of 5 ( $\text{CDCl}_3$  solution, vs. ext. TMS, ppm)



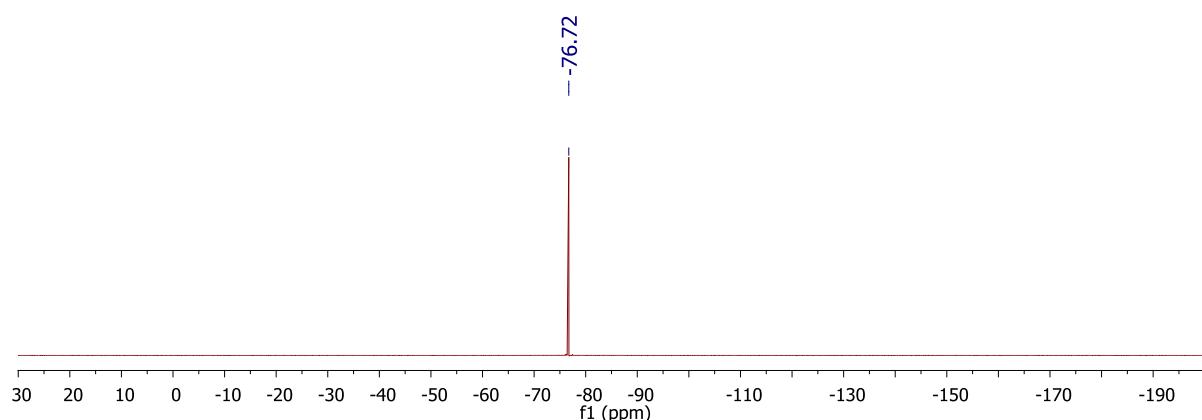
**Figure S18.**  $^{29}\text{Si}$ -NMR spectrum of 5 ( $\text{CDCl}_3$  solution, vs. ext. TMS, ppm)



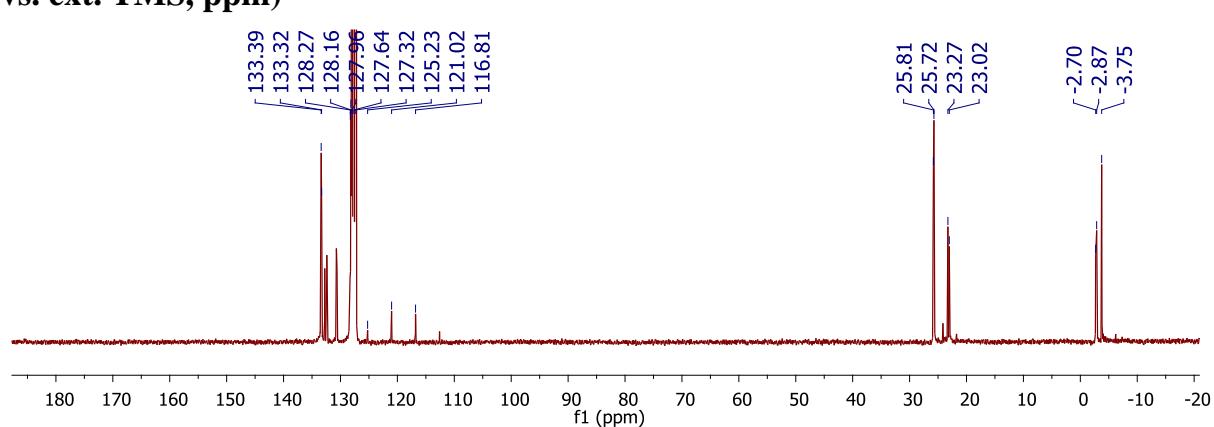
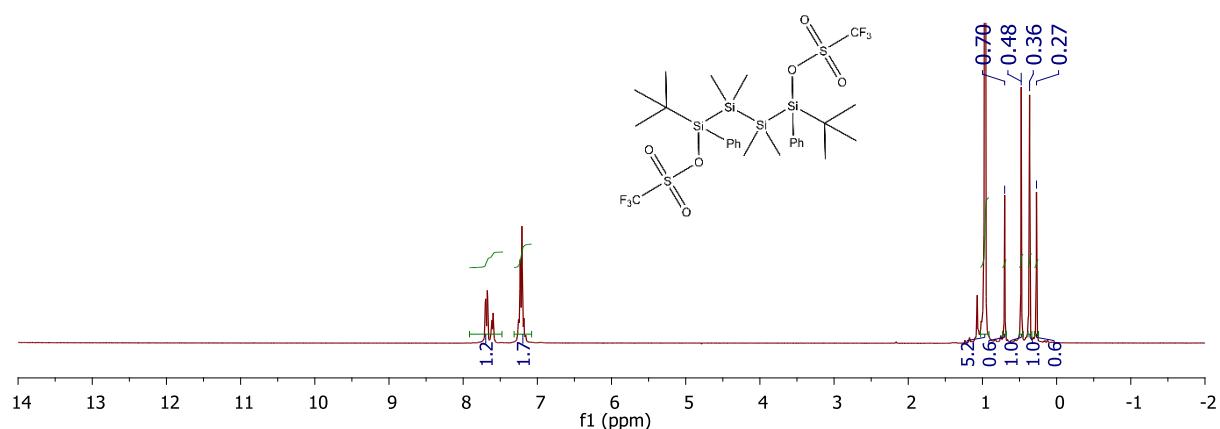
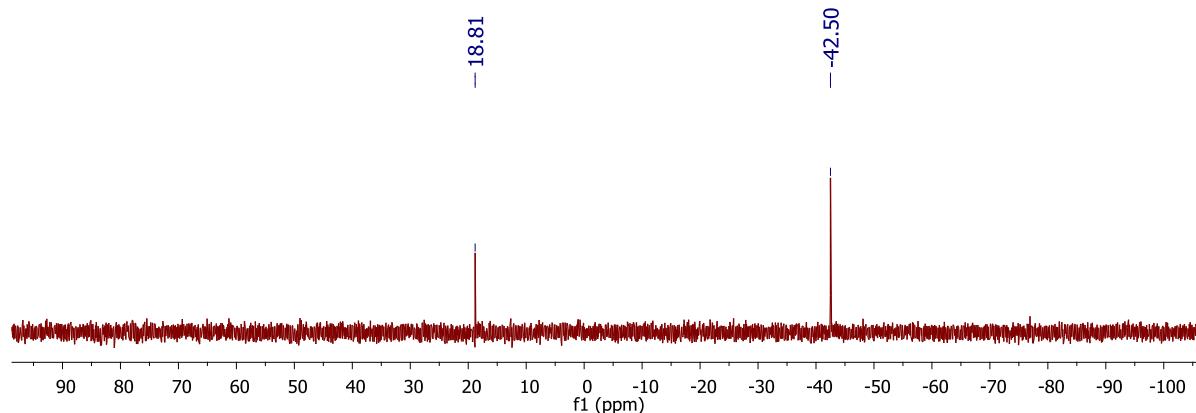
**Figure S19.**  $^1\text{H-NMR}$  spectrum of **6** ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)

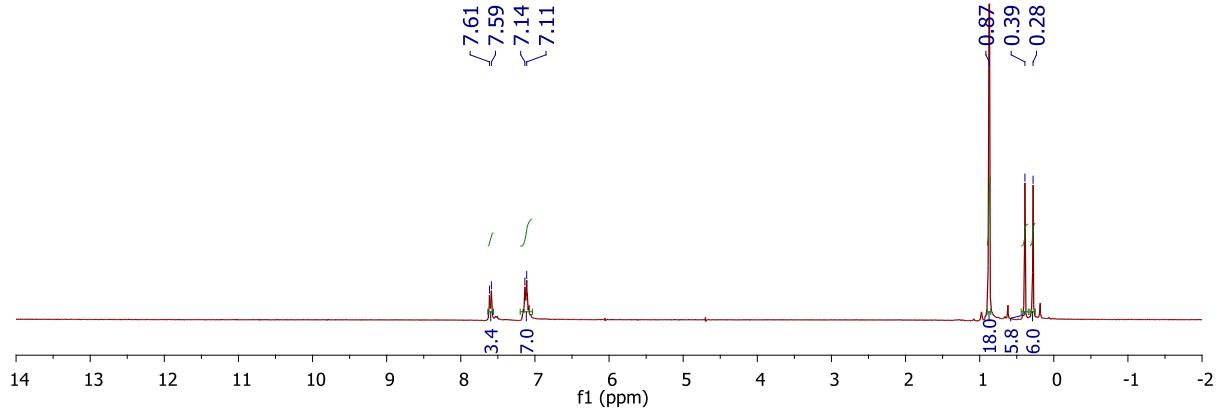
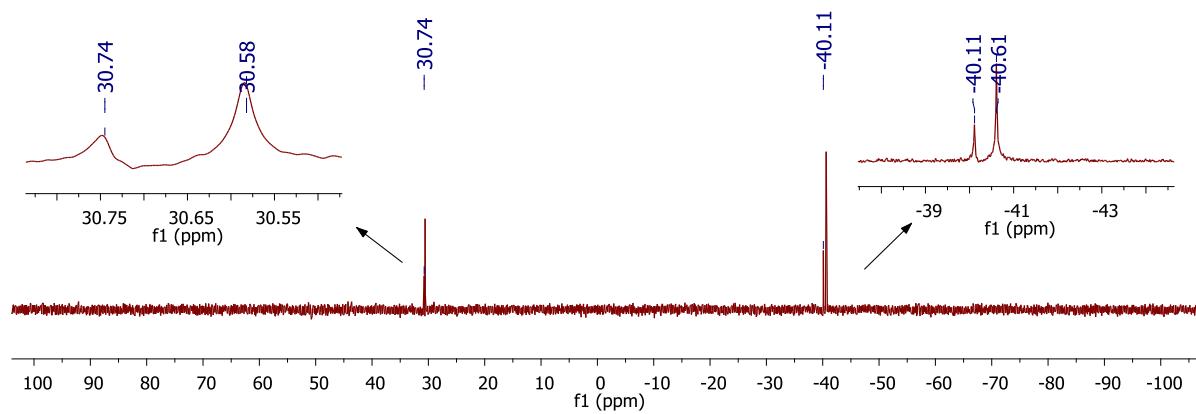
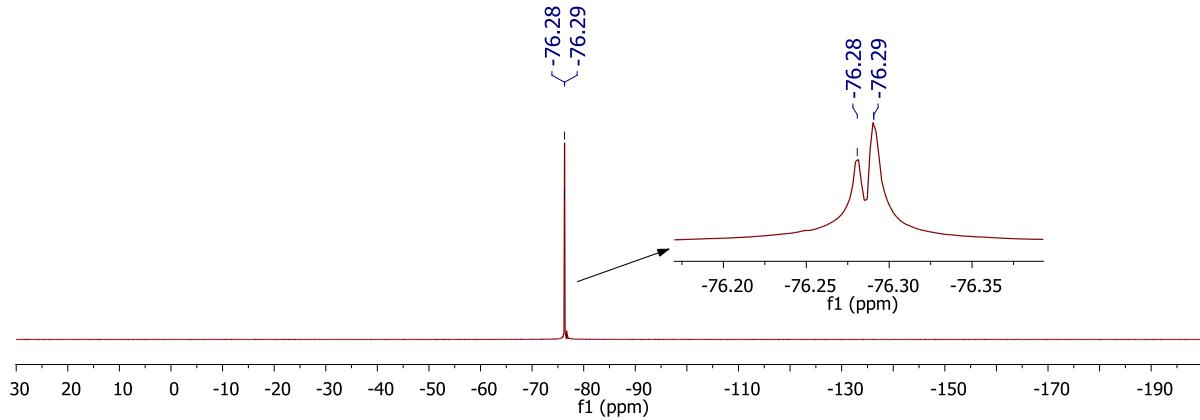


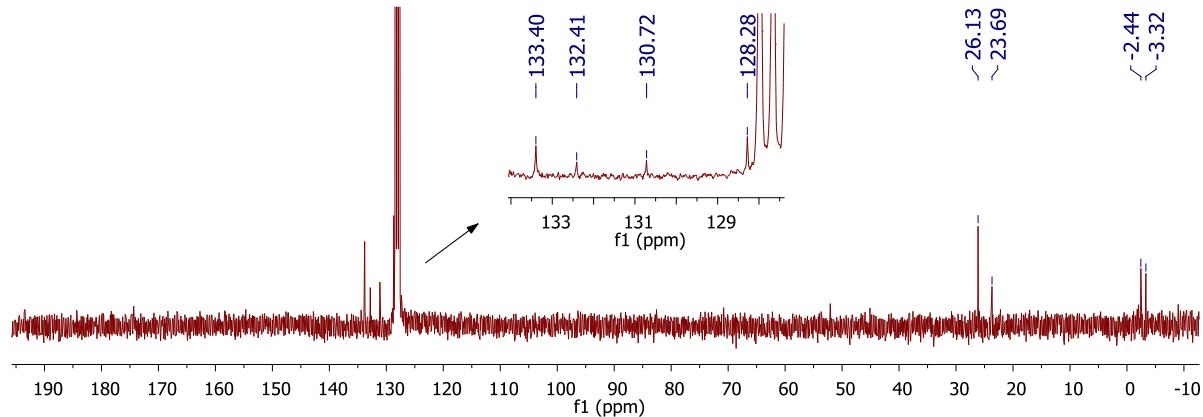
**Figure S20.**  $^{13}\text{C}$ -NMR spectrum of **6** ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



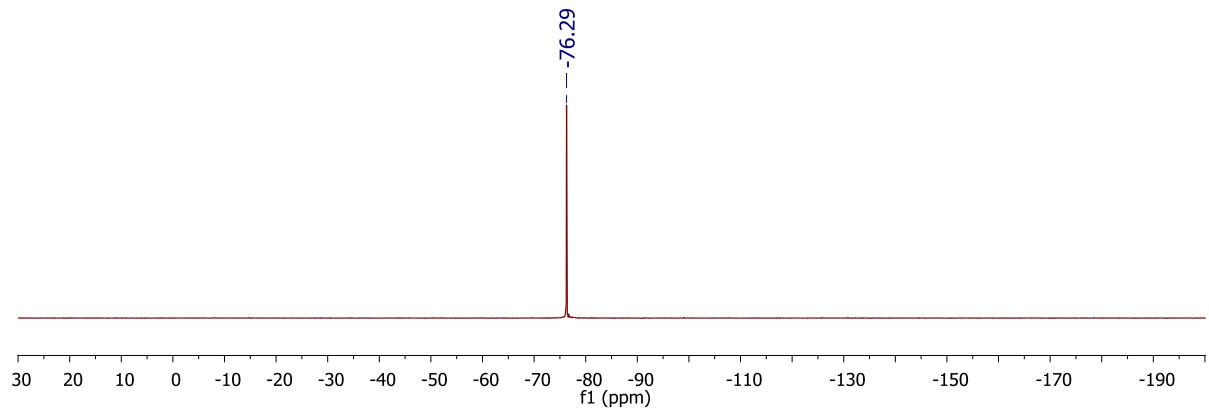
**Figure S21.**  $^{19}\text{F}$ -NMR spectrum of **6** ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



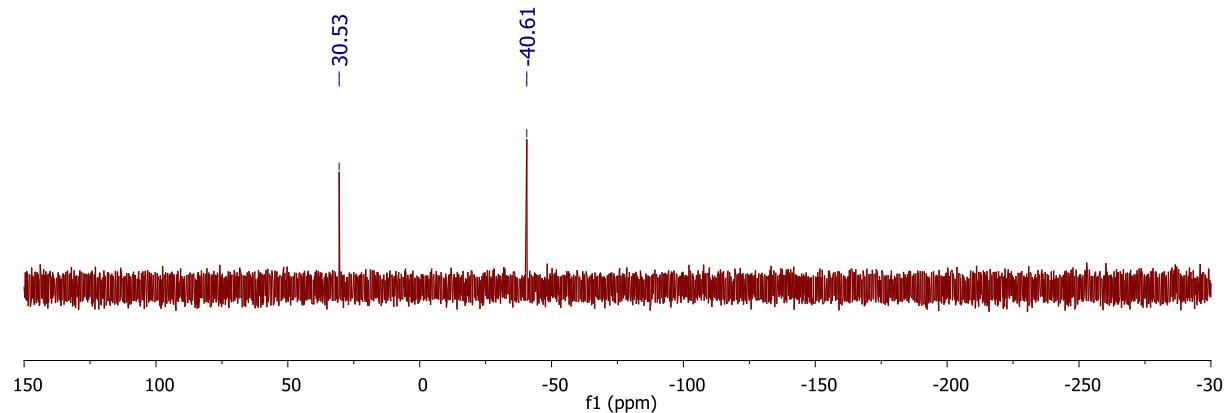




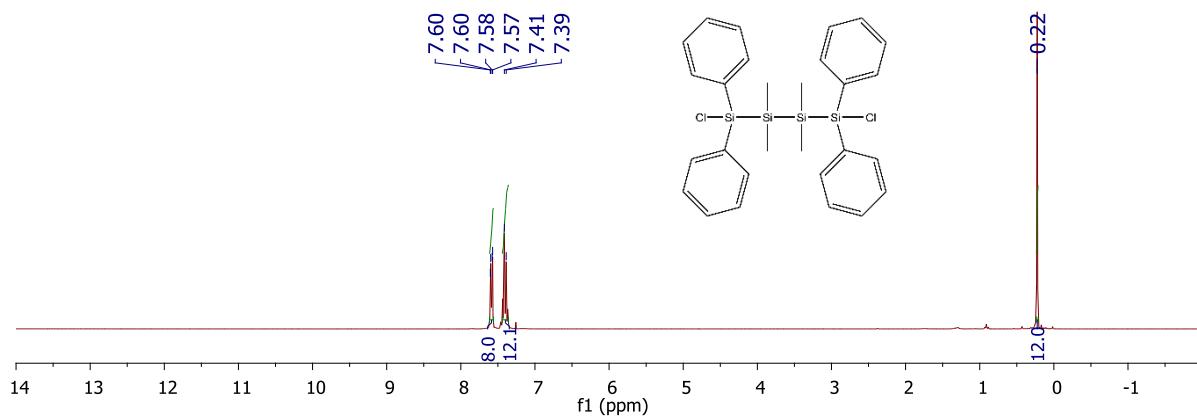
**Figure S28.** <sup>13</sup>C-NMR spectrum of 7 meso-diastereomer (C<sub>6</sub>D<sub>6</sub> solution, vs. ext. TMS, ppm)



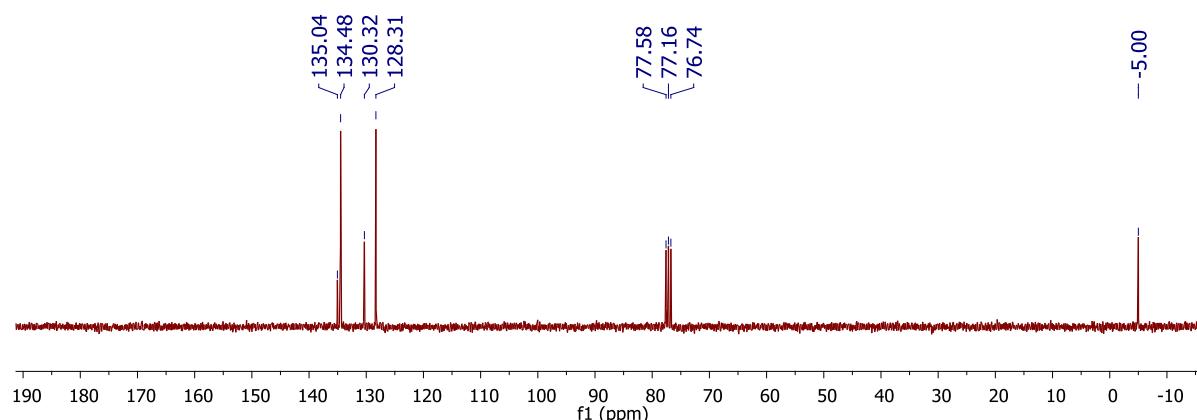
**Figure S29.** <sup>19</sup>F-NMR spectrum of 7 meso-diastereomer (C<sub>6</sub>D<sub>6</sub> solution, vs. ext. TMS, ppm)



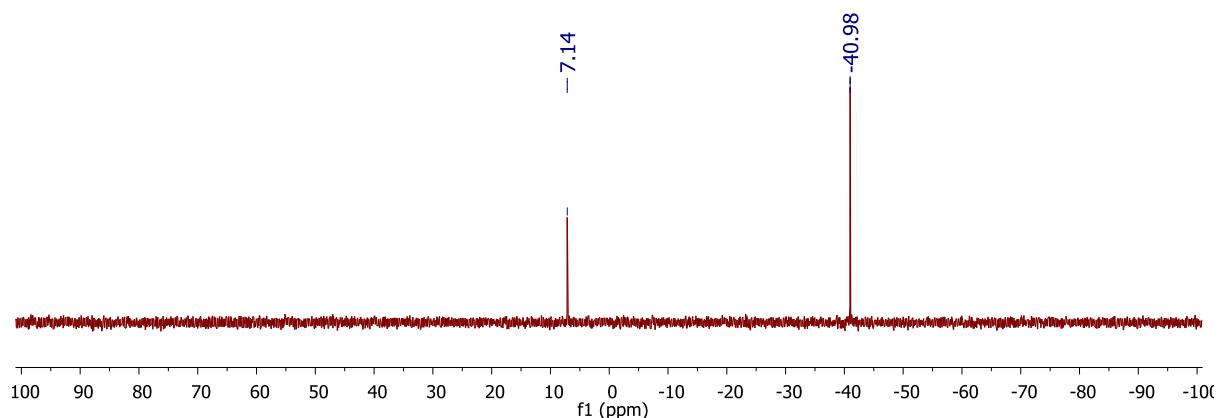
**Figure S30.** <sup>29</sup>Si-NMR spectrum of 7 meso-diastereomer (C<sub>6</sub>D<sub>6</sub> solution, vs. ext. TMS, ppm)



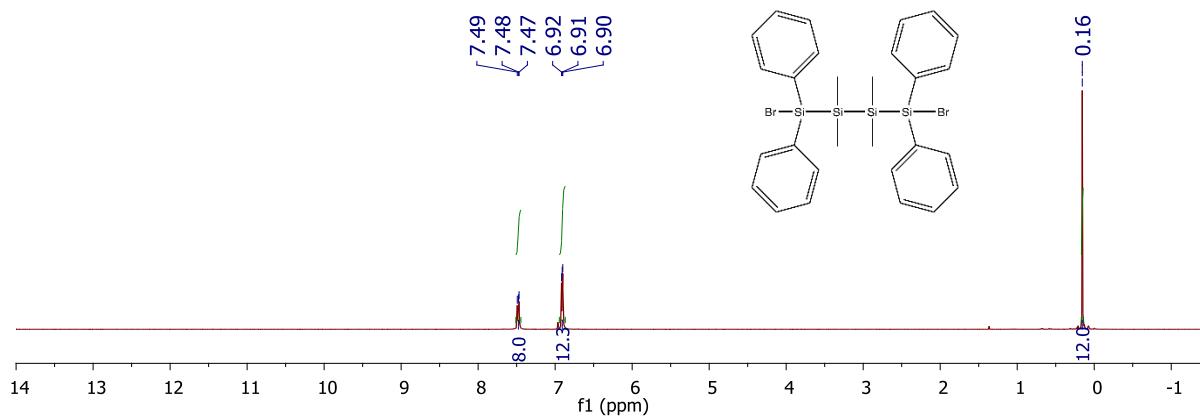
**Figure S31.** <sup>1</sup>H-NMR spectrum of 8a (CDCl<sub>3</sub> solution, vs. ext. TMS, ppm)



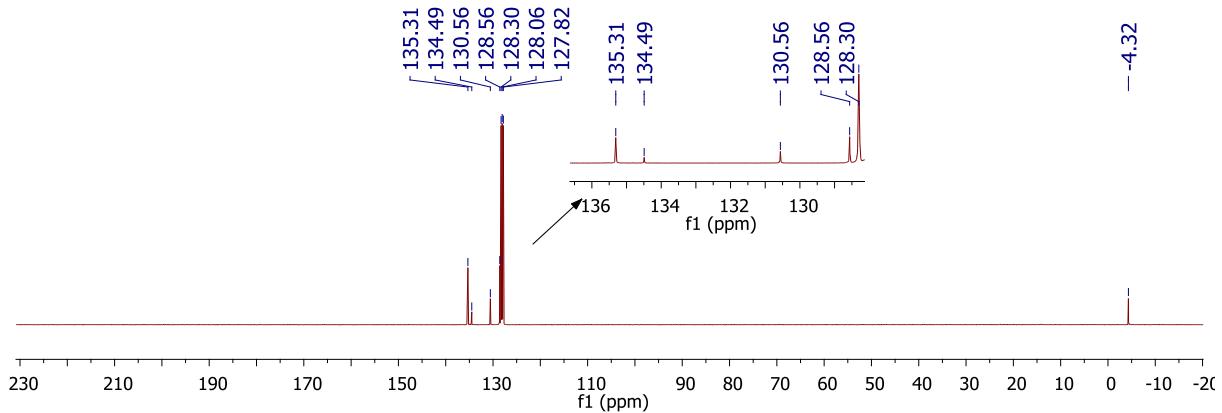
**Figure S32.** <sup>13</sup>C-NMR spectrum of 8a (CDCl<sub>3</sub> solution, vs. ext. TMS, ppm)



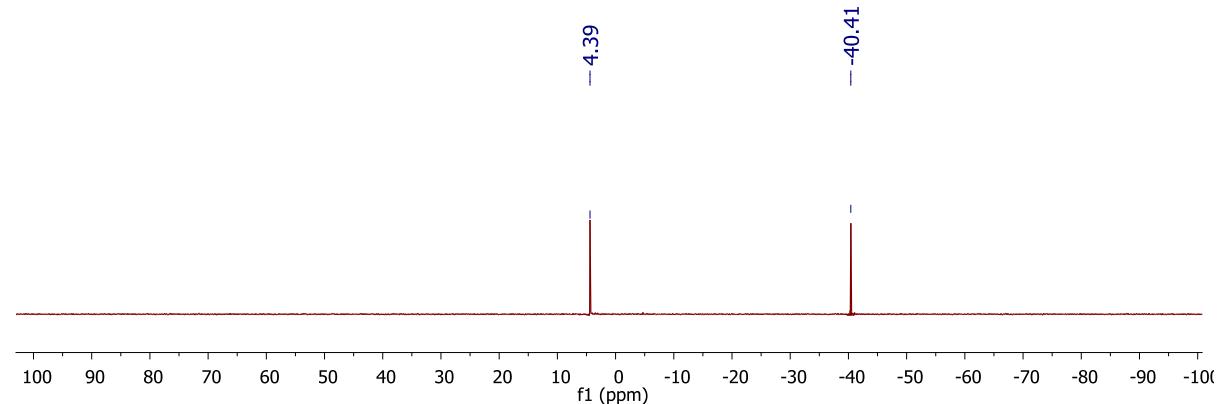
**Figure S33.** <sup>29</sup>Si-NMR spectrum of 8a (CDCl<sub>3</sub> solution, vs. ext. TMS, ppm)



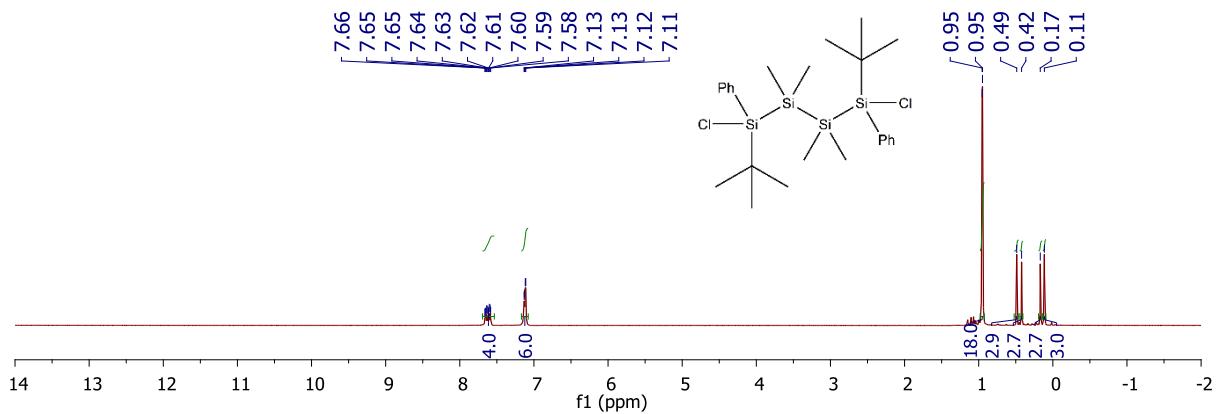
**Figure S34.**  $^1\text{H}$ -NMR spectrum of 8b ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



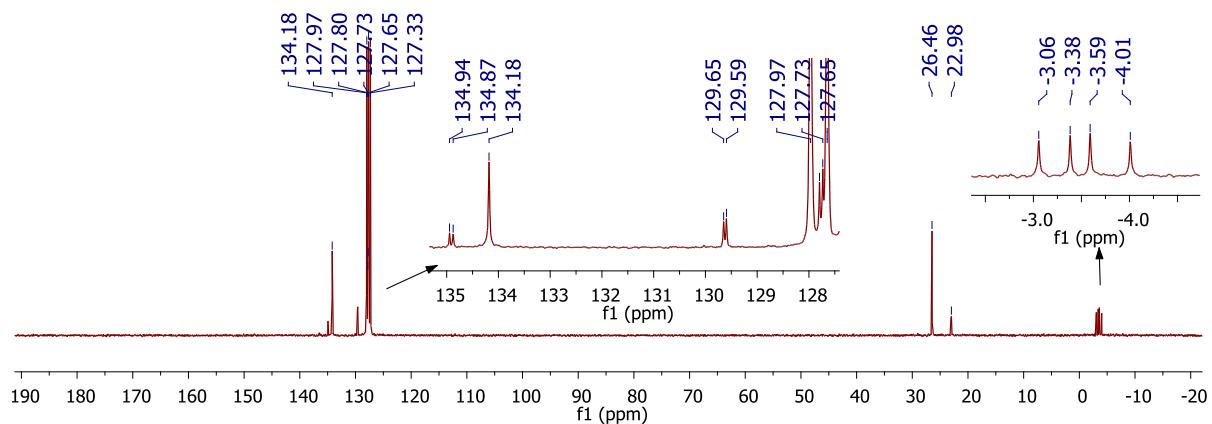
**Figure S35.**  $^{13}\text{C}$ -NMR spectrum of 8b ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



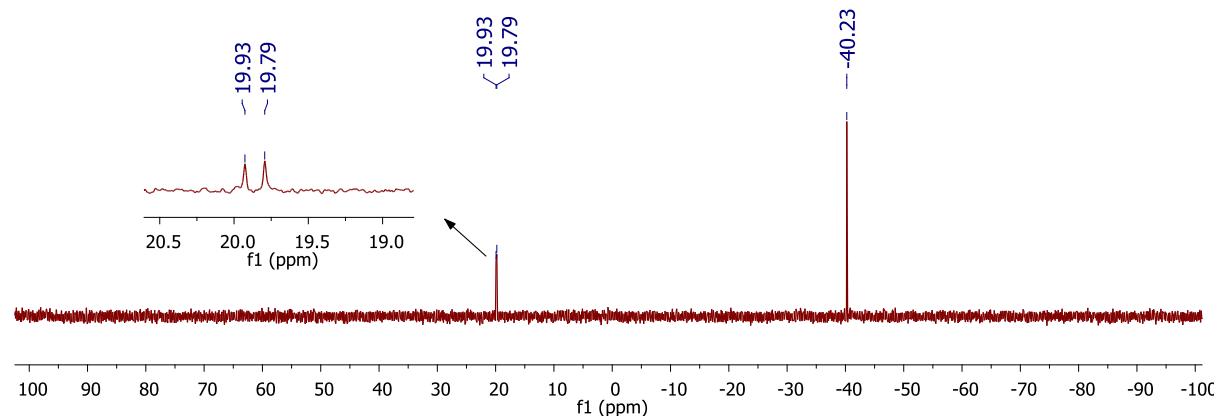
**Figure S36.**  $^{29}\text{Si}$ -NMR spectrum of 8b ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



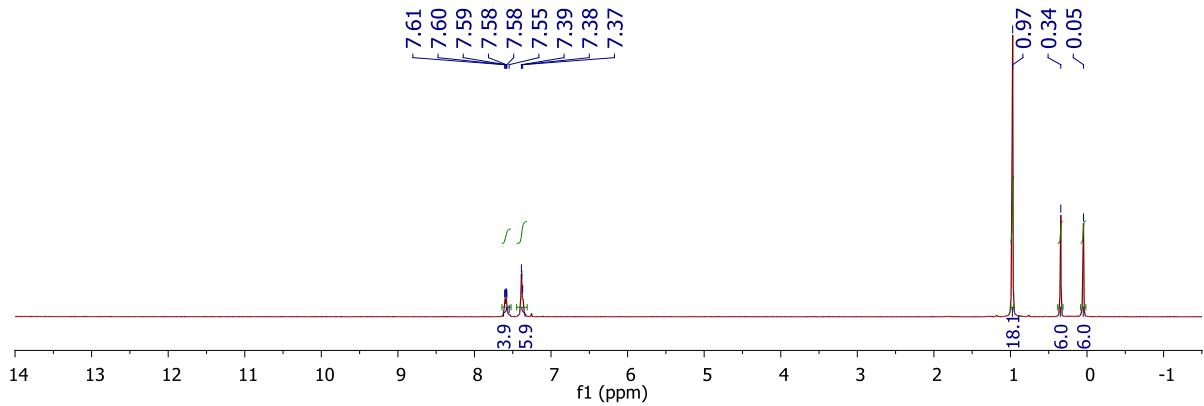
**Figure S37.**  $^1\text{H-NMR}$  spectrum of **9** as mixture of two diastereomers ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



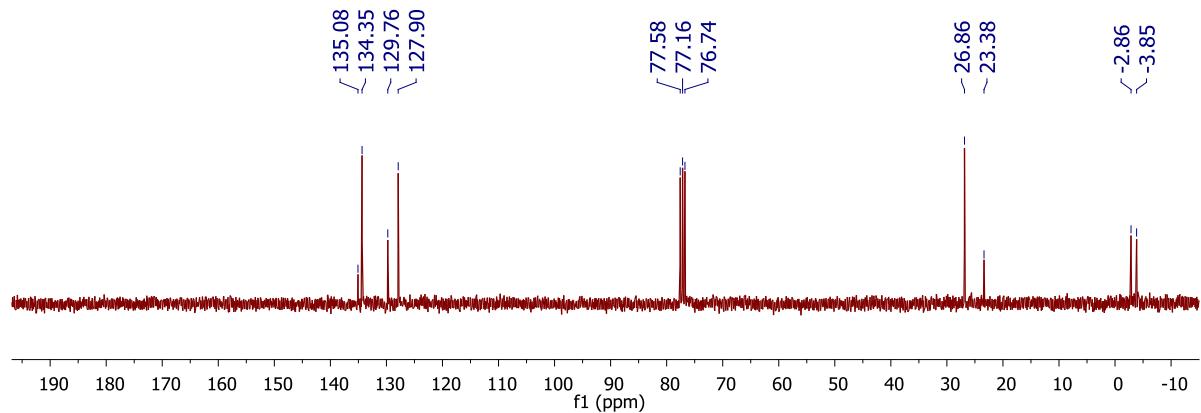
**Figure S38.**  $^{13}\text{C}$ -NMR spectrum of **9** as mixture of two diastereomers ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



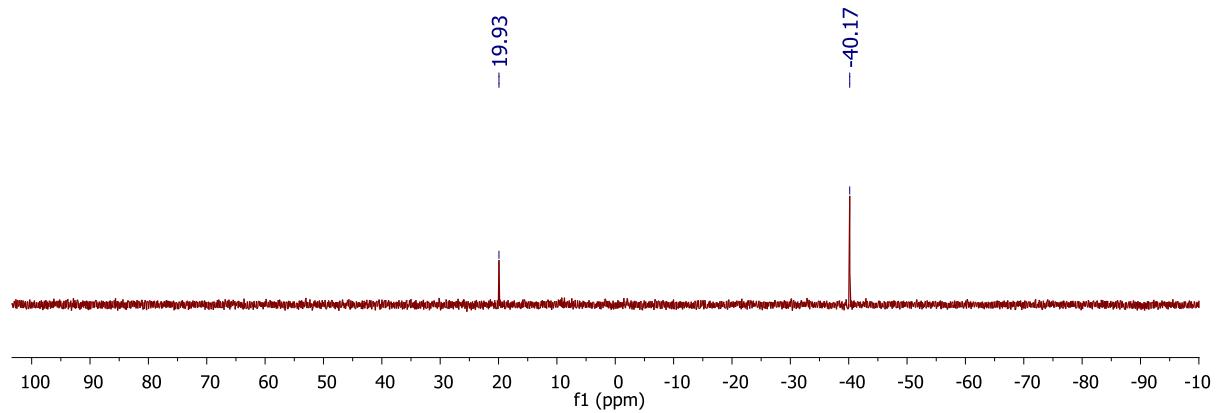
**Figure S39.**  $^{29}\text{Si}$ -NMR spectrum of **9** as mixture of two diastereomers ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



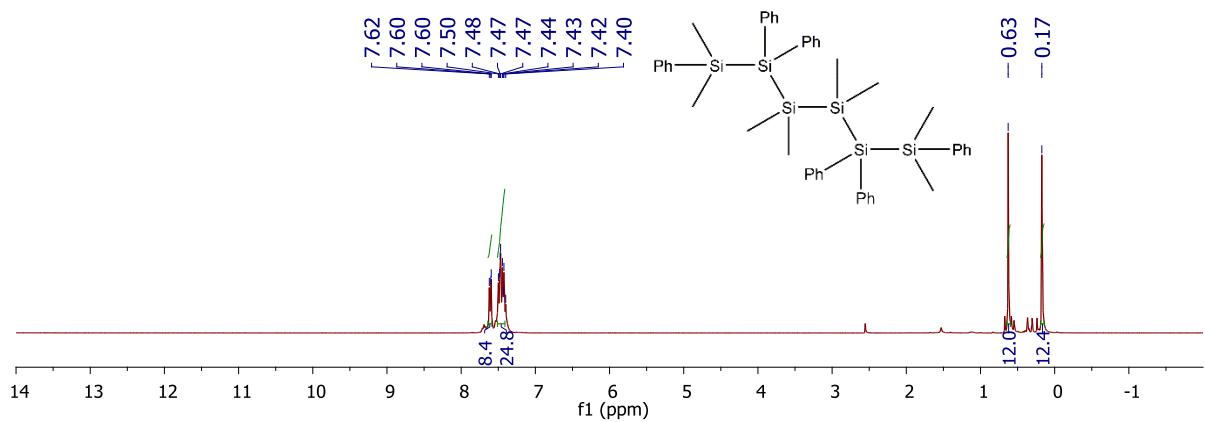
**Figure S40.**  $^1\text{H}$ -NMR spectrum of **9** (SS-RR-diastereomer) ( $\text{CDCl}_3$  solution, vs. ext. TMS, ppm)



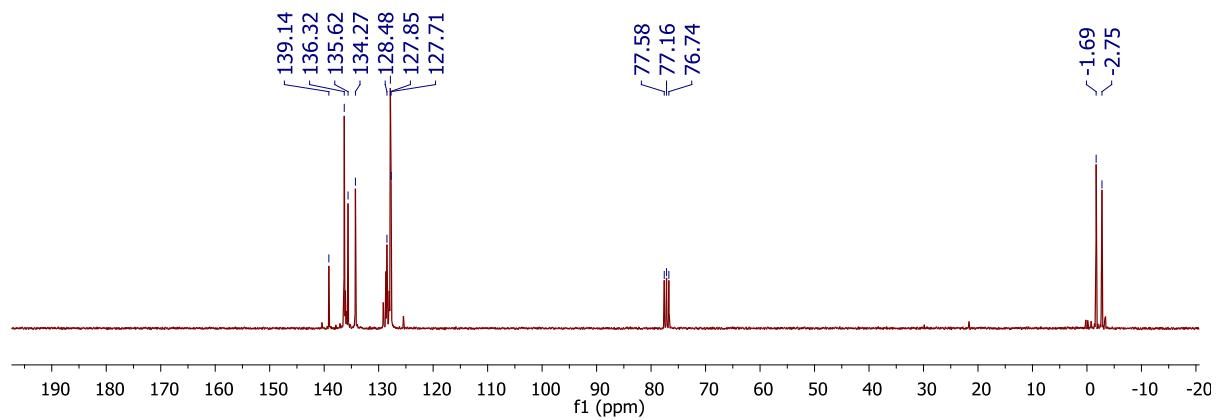
**Figure S41.**  $^{13}\text{C}$ -NMR spectrum of **9** (SS-RR-diastereomer) ( $\text{CDCl}_3$  solution, vs. ext. TMS, ppm)



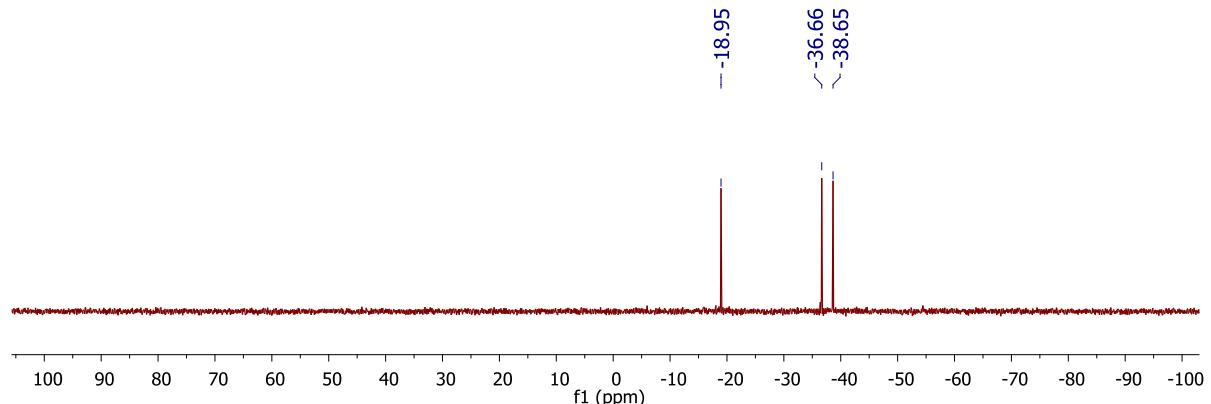
**Figure S42.**  $^{29}\text{Si}$ -NMR spectrum of **9** (SS-RR-diastereomer) ( $\text{CDCl}_3$  solution, vs. ext. TMS, ppm)



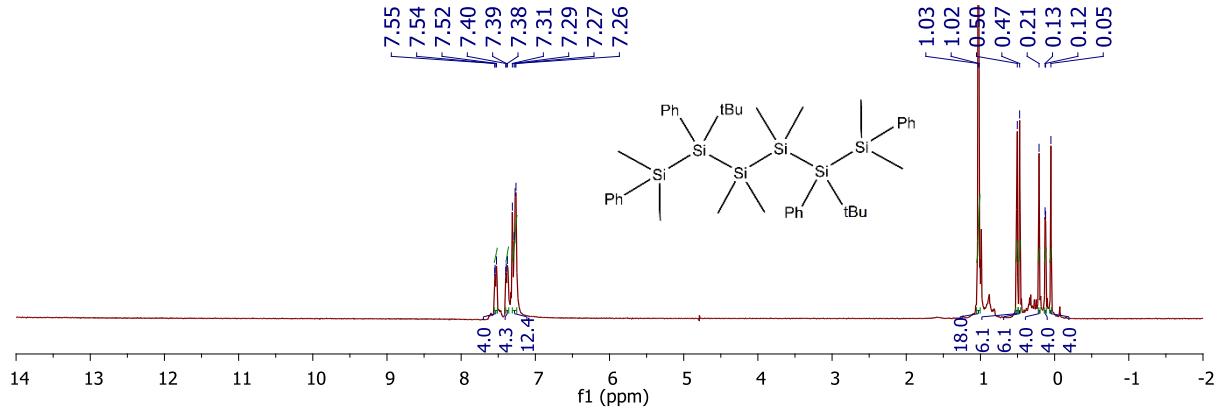
**Figure S43.**  $^1\text{H}$ -NMR spectrum of **10** ( $\text{CDCl}_3$  solution, vs. ext. TMS, ppm)



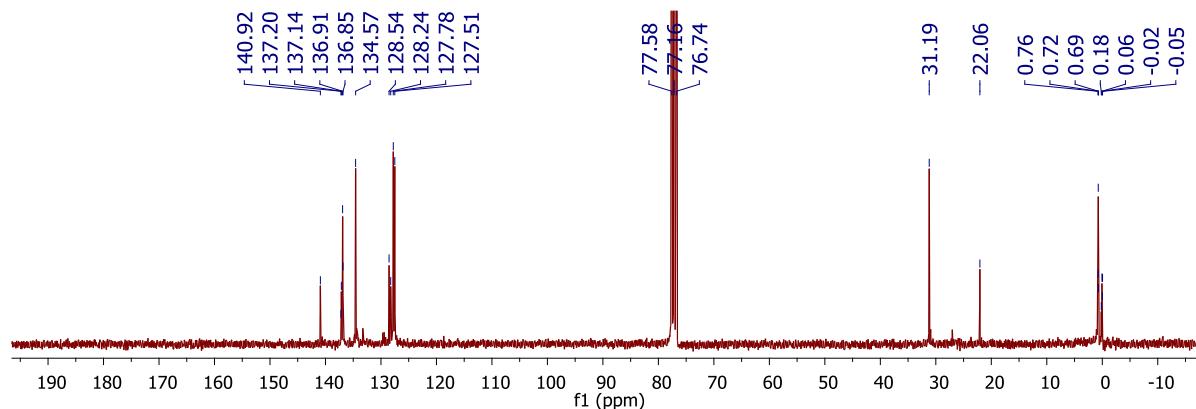
**Figure S44.**  $^{13}\text{C}$ -NMR spectrum of **10** ( $\text{CDCl}_3$  solution, vs. ext. TMS, ppm)



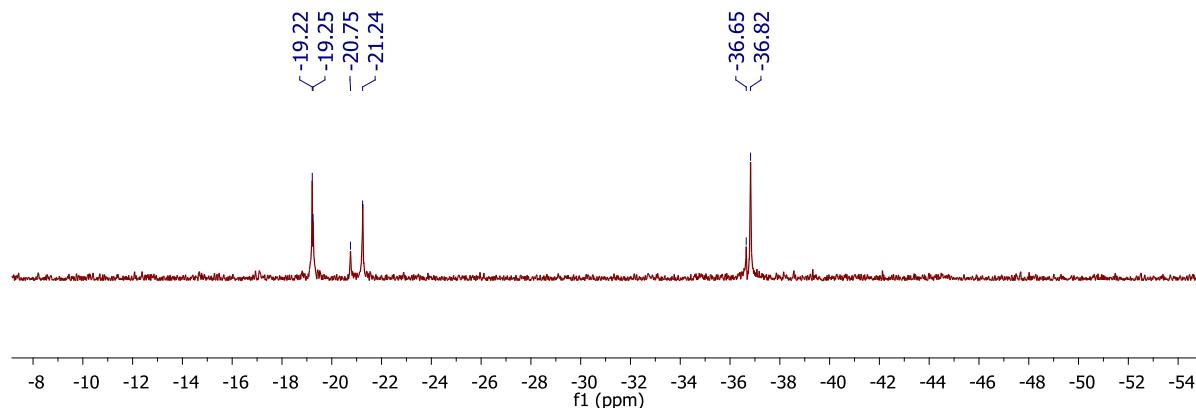
**Figure S45.**  $^{29}\text{Si}$ -NMR spectrum of **10** ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



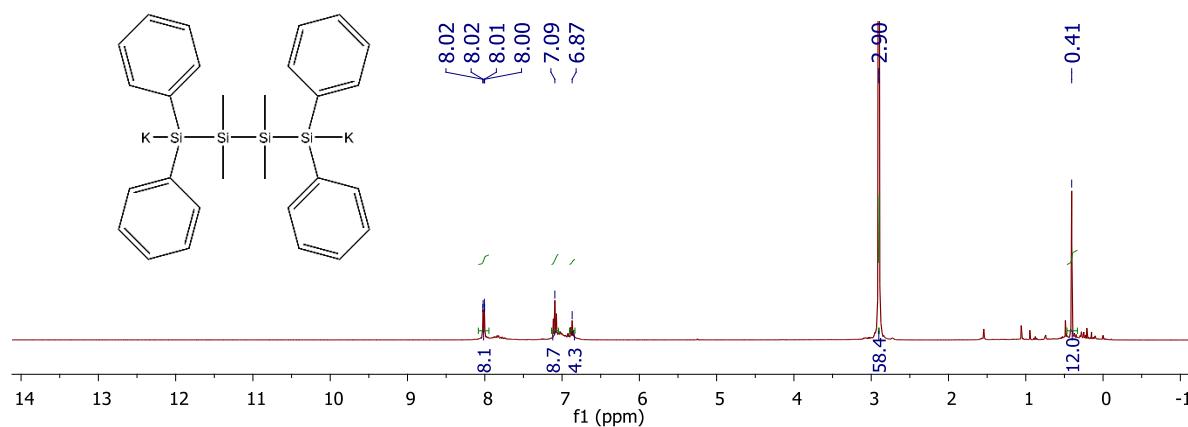
**Figure S46.** <sup>1</sup>H-NMR spectrum of 11 (CDCl<sub>3</sub> solution, vs. ext. TMS, ppm)



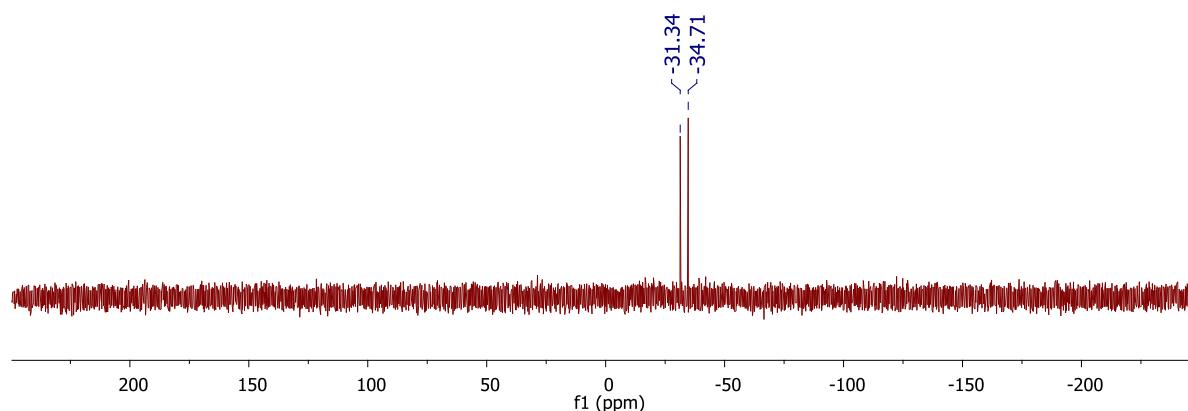
**Figure S47.** <sup>13</sup>C-NMR spectrum of 11 (CDCl<sub>3</sub> solution, vs. ext. TMS, ppm)



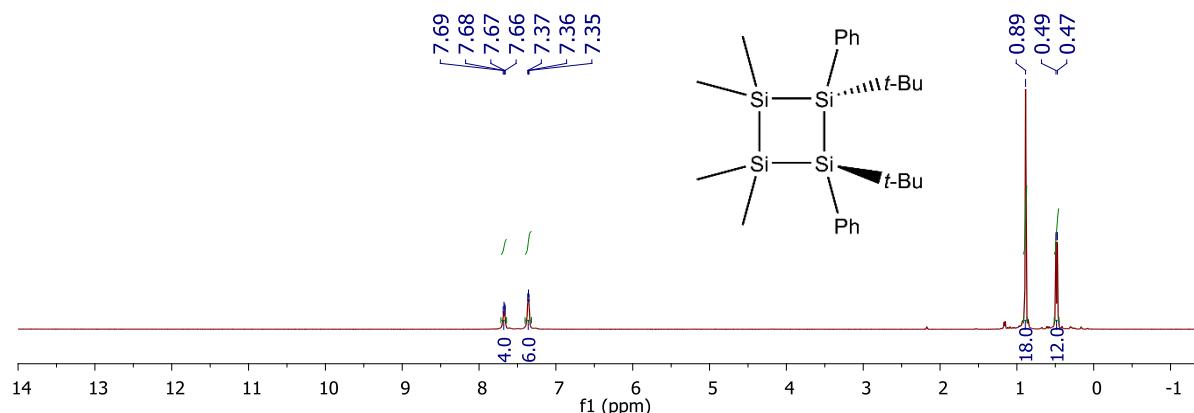
**Figure S48.** <sup>29</sup>Si-NMR spectrum of 11 (CDCl<sub>3</sub> solution, vs. ext. TMS, ppm)



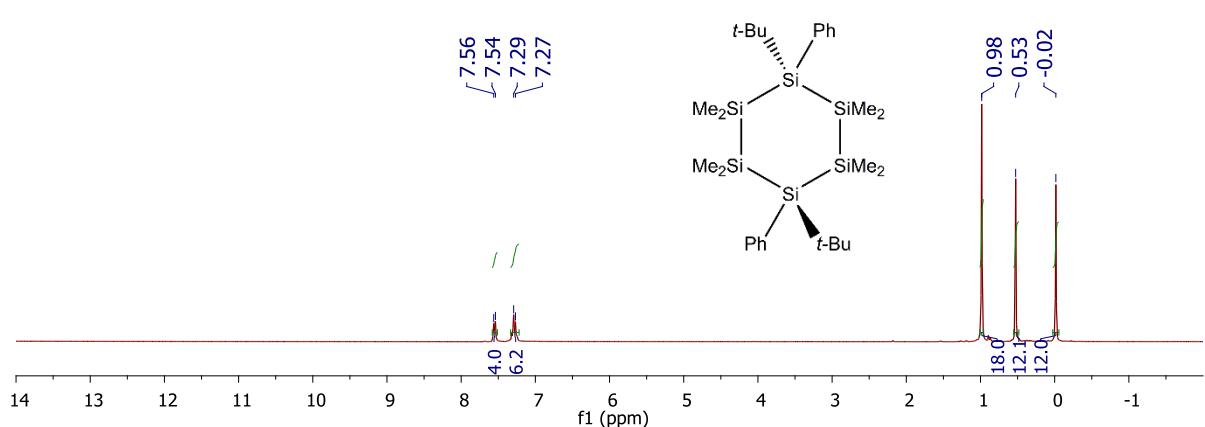
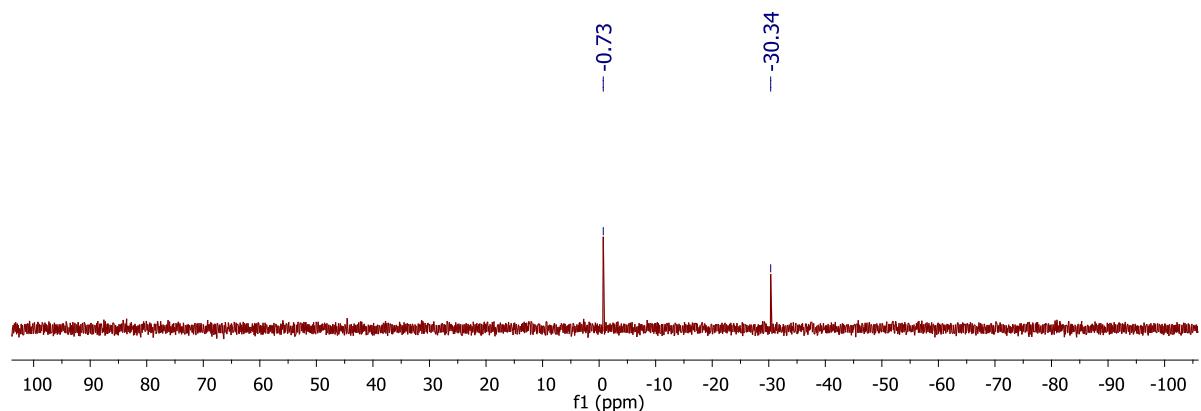
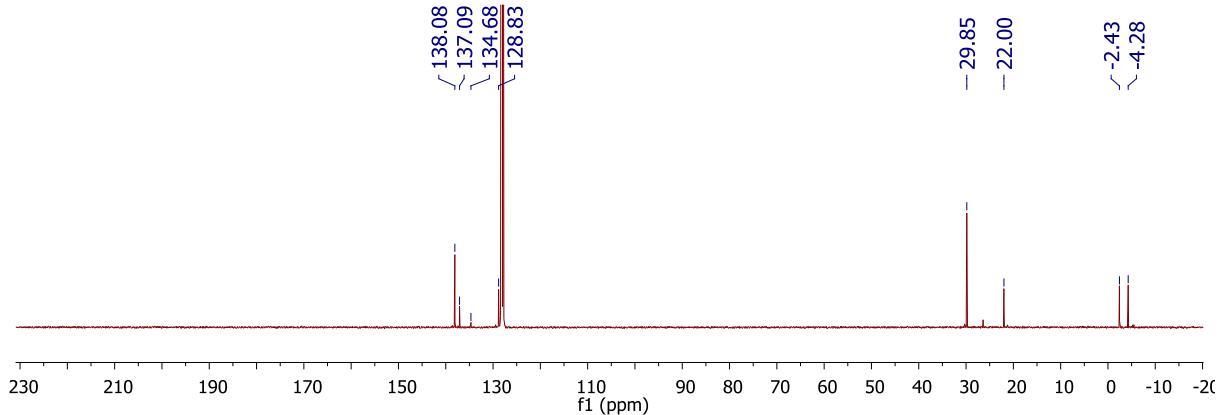
**Figure S49.**  $^1\text{H}$ -NMR spectrum of 12 ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)

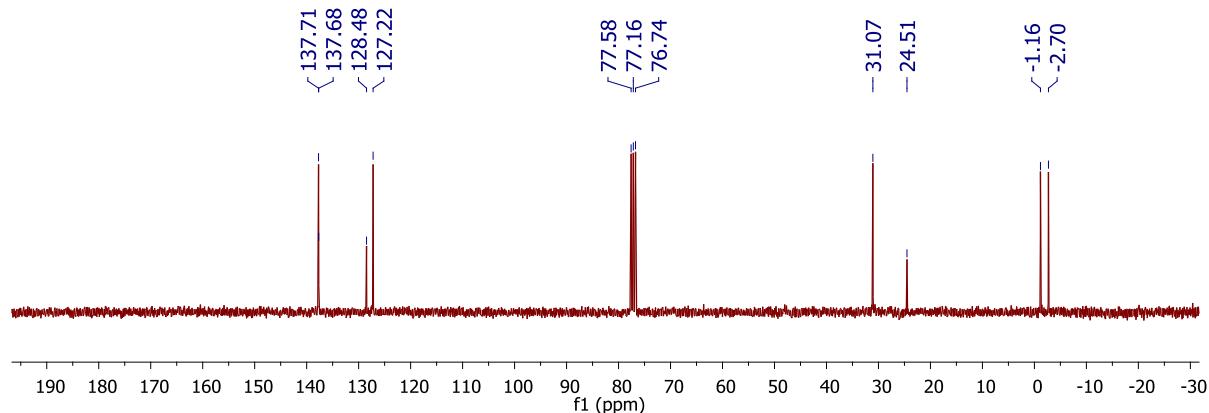


**Figure S50.**  $^{29}\text{Si}$ -NMR spectrum of 12 ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)

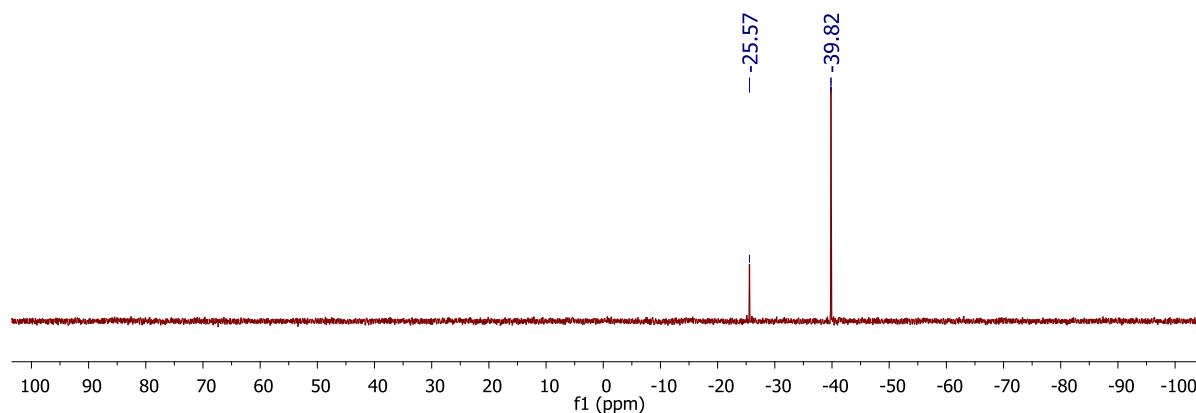


**Figure S51.**  $^1\text{H}$ -NMR spectrum of 14 ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)

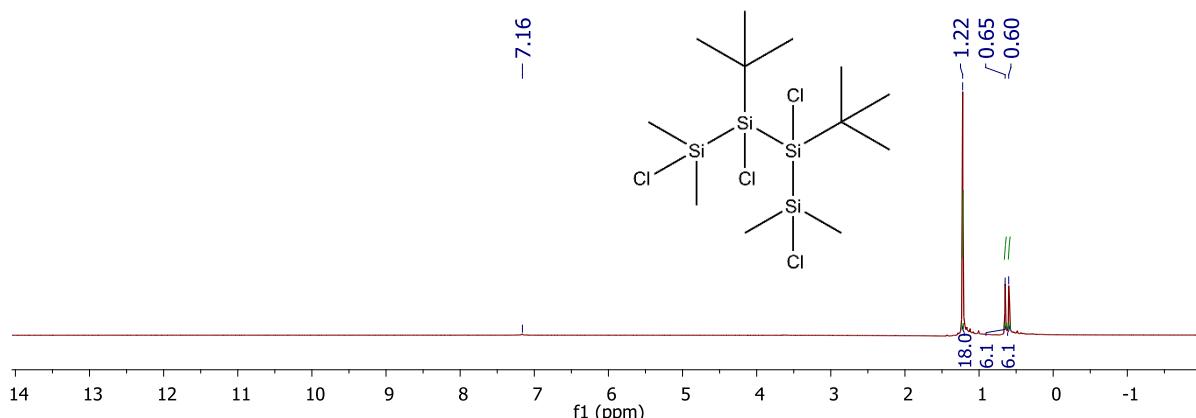




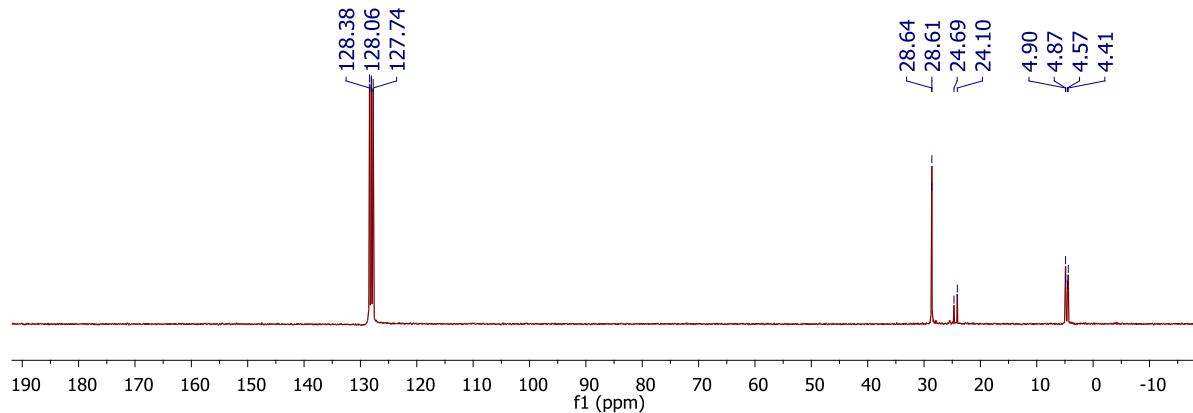
**Figure S55.**  $^{13}\text{C}$ -NMR spectrum of **15** ( $\text{CDCl}_3$  solution, vs. ext. TMS, ppm)



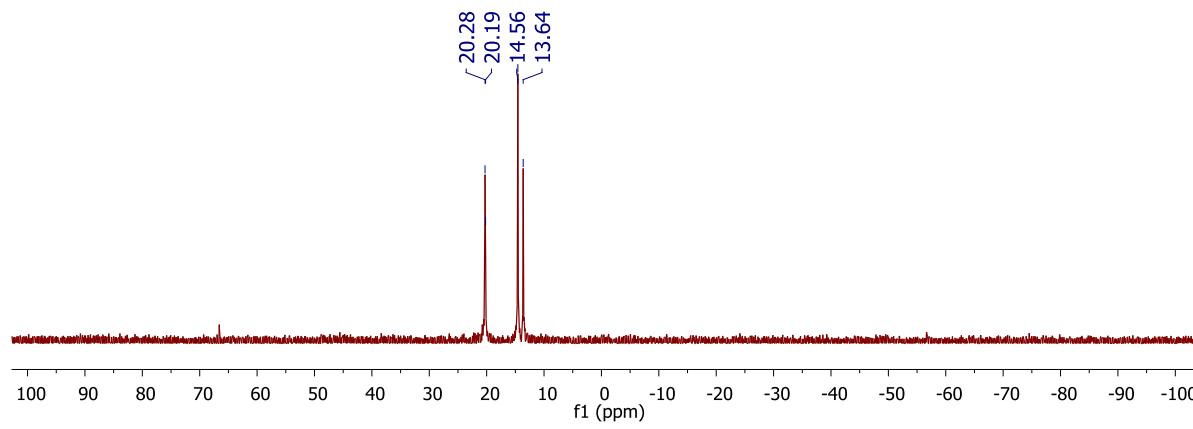
**Figure S56.**  $^{29}\text{Si}$ -NMR spectrum of **15** ( $\text{CDCl}_3$  solution, vs. ext. TMS, ppm)



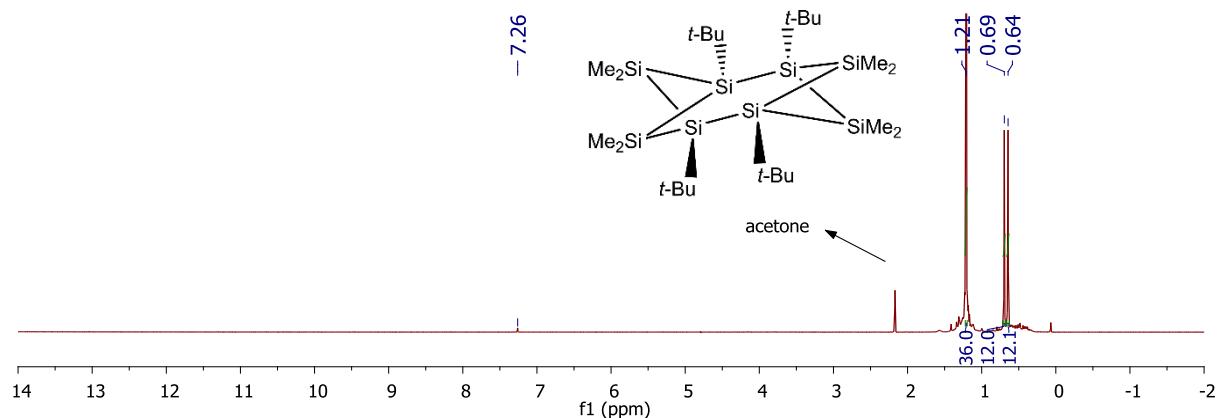
**Figure S57.**  $^1\text{H}$ -NMR spectrum of **16** ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



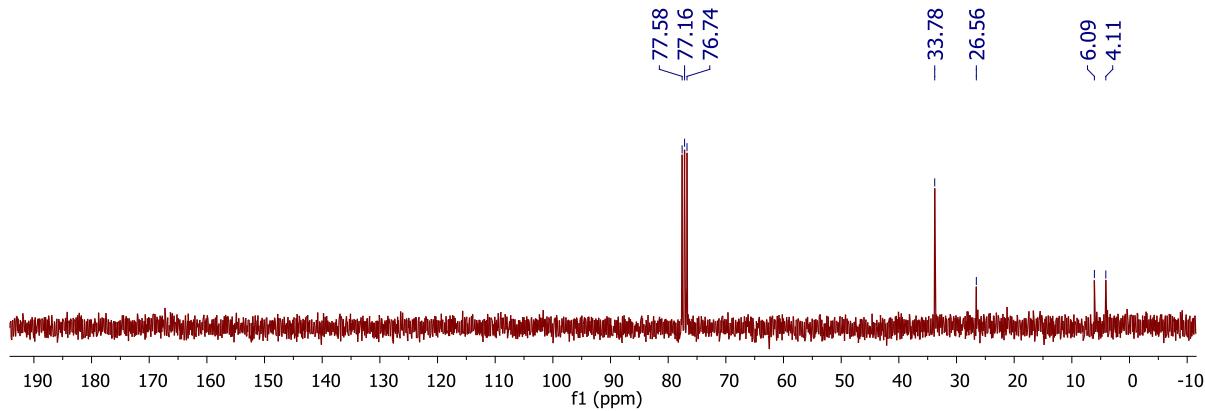
**Figure S58.**  $^{13}\text{C}$ -NMR spectrum of **16** ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



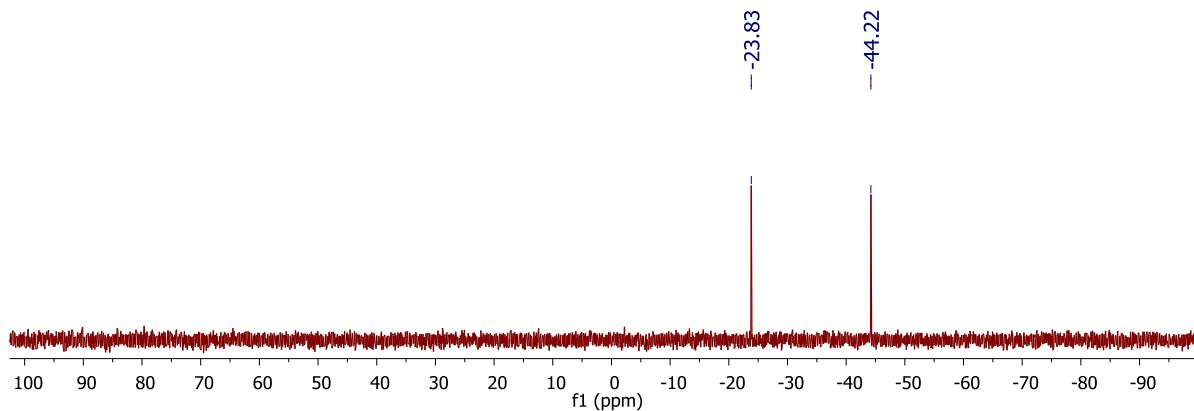
**Figure S59.**  $^{29}\text{Si}$ -NMR spectrum of **16** ( $\text{C}_6\text{D}_6$  solution, vs. ext. TMS, ppm)



**Figure S60.**  $^1\text{H}$ -NMR spectrum of **17** ( $\text{CDCl}_3$  solution, vs. ext. TMS, ppm)



**Figure S61.**  $^{13}\text{C}$ -NMR spectrum of 17 (CDCl<sub>3</sub> solution, vs. ext. TMS, ppm)



**Figure S62.**  $^{29}\text{Si}$ -NMR spectrum of 17 (CDCl<sub>3</sub> solution, vs. ext. TMS, ppm)

## 2. X-ray Crystallography

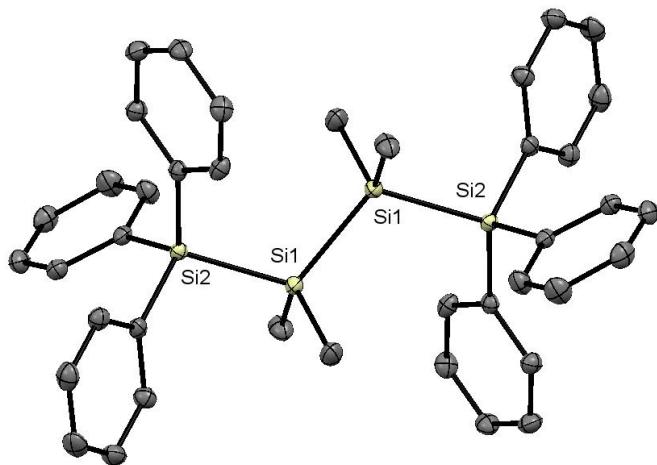
**Table S1:** Crystallographic data and details of measurements for compounds **3-7, 8b, 9** and **13-15** and **17**

Mo K $\alpha$  ( $\lambda=0.71073\text{\AA}$ ). R1 =  $\sum |F_o| - |F_c| / |\sum |F_d|$ ; wR2 =  $[\sum_w (F_o^2 - F_2^2)^2 / \sum_w (F_o^2)^2]^{1/2}$

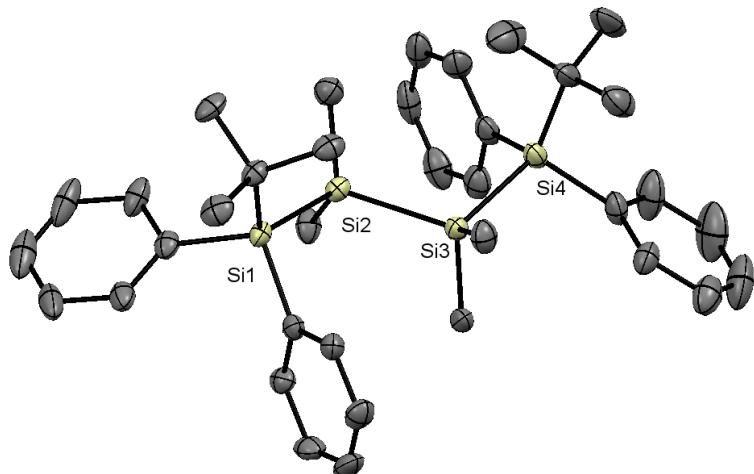
Compound	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>
Formula	C <sub>22</sub> H <sub>64</sub> Si <sub>10</sub>	C <sub>40</sub> H <sub>42</sub> Si <sub>4</sub>	C <sub>36</sub> H <sub>50</sub> Si <sub>4</sub>	C <sub>30</sub> H <sub>32</sub> F <sub>6</sub> O <sub>6</sub> S <sub>2</sub> Si <sub>4</sub>
Mw (g mol <sup>-1</sup> )	609.63	635.09	595.12	779.03
<i>a</i> (Å)	42.6458(19)	8.7630(18)	13.3342(12)	9.2952(10)
<i>b</i> (Å)	15.7965(8)	9.5690(19)	14.5271(14)	9.3399(9)
<i>c</i> (Å)	40.3481(19)	12.425(3)	18.2178(17)	11.2596(11)
$\alpha$ (°)	90	68.00(3)	90	80.046(5)
$\beta$ (°)	118.2280(10)	79.71(3)	93.459(2)	76.448(5)
$\gamma$ (°)	90	65.00(3)	90	71.373(5)
<i>V</i> (Å <sup>3</sup> )	23948(2)	875.3(4)	3522.5(6)	895.41(16)
<i>Z</i>	24	1	4	1
Crystal size (mm)	0.17 x 0.13 x 0.11	0.10 x 0.08 x 0.06	0.15 x 0.12 x 0.10	0.06 x 0.06 x 0.06
Crystal habit	Block, colourless	Block, colourless	Block, colourless	Block, colourless
Crystal system	Monoclinic	Triclinic	Monoclinic	Triclinic
Space group	<i>P</i> 1 21/c 1	<i>P</i> -1	<i>P</i> 2 <sub>1</sub> /n	<i>P</i> -1
<i>d</i> <sub>calc</sub> (mg/m <sup>3</sup> )	1.015	1.205	1.122	1.445
$\mu$ (mm <sup>-1</sup> )	0.340	0.197	0.191	0.35
<i>T</i> (K)	100(2)	100(2)	123(2)	100(2)
2θ range (°)	1.7-25.5	3.5 to 25.2	1.8 to 25.5	2.3-33.1
<i>F</i> (000)	8064	338	1288	402
<i>R</i> <sub>int</sub>	0.0947	0.0182	0.1138	0.052
independent reflns	44553	2836	6539	6849
No. of params	1990	201	371	219
R1, wR2 (all data)	R1 = 0.1963 wR2 = 0.2591	R1 = 0.0357 wR2 = 0.0855	R1 = 0.1317 wR2 = 0.1663	R1 = 0.0397 wR2 = 0.0808
R1, wR2 (>2σ)	R1 = 0.1105 wR2 = 0.2093	R1 = 0.0338 wR2 = 0.0842	R1 = 0.0681 wR2 = 0.1390	R1 = 0.0397 wR2 = 0.0745

Compound	<b>7</b>	<b>8b</b>	<b>9</b>	<b>13</b>
Formula	C <sub>26</sub> H <sub>40</sub> F <sub>6</sub> O <sub>6</sub> S <sub>2</sub> Si <sub>4</sub>	C <sub>28</sub> H <sub>32</sub> Br <sub>2</sub> Si <sub>4</sub>	C <sub>24</sub> H <sub>40</sub> Cl <sub>2</sub> Si <sub>4</sub>	C <sub>48</sub> H <sub>88</sub> K <sub>2</sub> O <sub>12</sub> Si <sub>4</sub>
Mw (g mol <sup>-1</sup> )	739.06	640.71	511.82	523.87
<i>a</i> (Å)	15.4562(5)	13.1505(7)	27.1714(18)	10.8607(5)
<i>b</i> (Å)	9.3014(3)	14.5941(6)	28.8435(18)	11.3721(5)
<i>c</i> (Å)	25.6438(9)	8.1017(4)	7.3180(4)	14.0989(6)
$\alpha$ (°)	90	90	90	81.357(2)
$\beta$ (°)	105.499(2)	105.6720(10)	90	69.123(2)
$\gamma$ (°)	90	90	90	61.886(2)
<i>V</i> (Å <sup>3</sup> )	3552.6(2)	1497.07(13)	5735.3(6)	1434.72(11)
<i>Z</i>	4	1	8	1
Crystal size (mm)	0.18 x 0.13 x 0.12	0.16 x 0.13 x 0.10	0.28 x 0.25 x 0.13	0.18 x 0.15 x 0.1
Crystal habit	Block, colourless	Block, colourless	Block, colourless	Block, colourless
Crystal system	Monoclinic	Monoclinic	Orthorhombic	Triclinic
Space group	<i>P</i> 2 <sub>1</sub> / <i>n</i>	<i>P</i> 2 <sub>1</sub> / <i>c</i>	<i>F</i> dd2	<i>P</i> -1
<i>d</i> <sub>calc</sub> (mg/m <sup>3</sup> )	1.382	1.421	1.186	1.213
$\mu$ (mm <sup>-1</sup> )	0.353	2.883	0.404	0.302
<i>T</i> (K)	100(2)	123(2)	100(2)	100(2)
2θ range (°)	1.4 to 26.0	2.1 to 26.2	2.8 to 29.0	2.3 to 29.0
<i>F</i> (000)	1544	652	2192	566
<i>R</i> <sub>int</sub>	0.0468	0.0810	0.1074	0.0701
independent reflns	6987	2962	3799	6903
No. of params	465	156	141	303
R1, wR2 (all data)	R1 = 0.0428 wR2 = 0.0915	R1 = 0.0325 wR2 = 0.0741	R1 = 0.0496 wR2 = 0.1073	R1 = 0.0651 wR2 = 0.0805
R1, wR2 (>2σ)	R1 = 0.0363 wR2 = 0.0846	R1 = 0.0267 wR2 = 0.0728	R1 = 0.0437 wR2 = 0.1037	R1 = 0.0371 wR2 = 0.0725

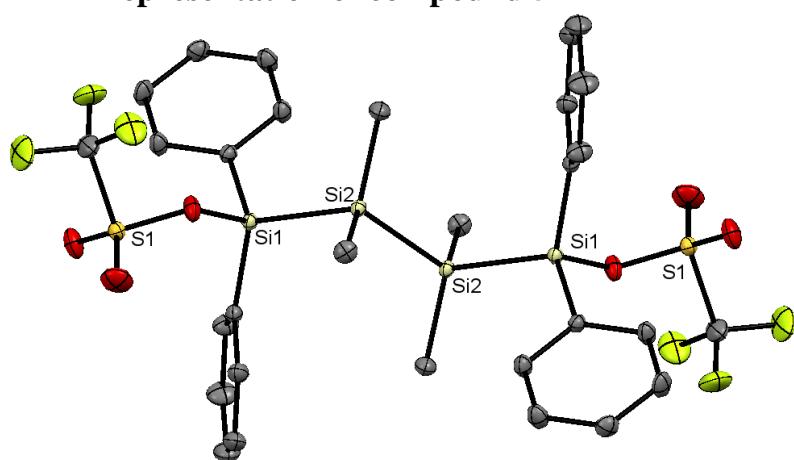
Compound	<b>14</b>	<b>15</b>	<b>17</b>
Formula	C <sub>24</sub> H <sub>40</sub> Si <sub>4</sub>	C <sub>28</sub> H <sub>52</sub> Si <sub>6</sub>	C <sub>24</sub> H <sub>60</sub> Si <sub>8</sub>
Fw (g mol <sup>-1</sup> )	440.92	557.23	573.44
<i>a</i> (Å)	17.6483(7)	9.5598(3)	21.299(2)
<i>b</i> (Å)	9.4015(3)	11.7937(4)	21.299(2)
<i>c</i> (Å)	16.6150(6)	14.7457(5)	15.9202(19)
$\alpha$ (°)	90	90	90
$\beta$ (°)	105.776(4)	94.929(2)	90
$\gamma$ (°)	90	90	90
<i>V</i> (Å <sup>3</sup> )	2652.93(17)	1656.36(10)	7222.2(18)
<i>Z</i>	4	2	8
Crystal size (mm)	0.13 x 0.12 x 0.10	0.35 x 0.28 x 0.26	0.16 x 0.13 x 0.1
Crystal habit	Block, colourless	Block, colourless	Block, colourless
Crystal system	Monoclinic	Monoclinic	Tetragonal
Space group	<i>C</i> 2/c	<i>P</i> 1 21/n 1	<i>P</i> -421c
<i>d</i> <sub>calc</sub> (mg/m <sup>3</sup> )	1.104	1.117	1.055
$\mu$ (mm <sup>-1</sup> )	0.232	0.267	0.309
<i>T</i> (K)	123(2)	100(2)	100(2)
2θ range (°)	2.5 to 26.0	2.2 to 28.0	2.3 to 28.0
<i>F</i> (000)	960	608	2528
<i>R</i> <sub>int</sub>	0.0555	0.0518	0.046
independent reflns	2610	4004	16248
No. of params	132	161	309
R1, wR2 (all data)	R1 = 0.0400 wR2 = 0.0915	R1 = 0.0284 wR2 = 0.0665	R1 = 0.1391 wR2 = 0.1480
R1, wR2 (>2σ)	R1 = 0.0339 wR2 = 0.0876	R1 = 0.0247 wR2 = 0.0635	R1 = 0.0678 wR2 = 0.1205



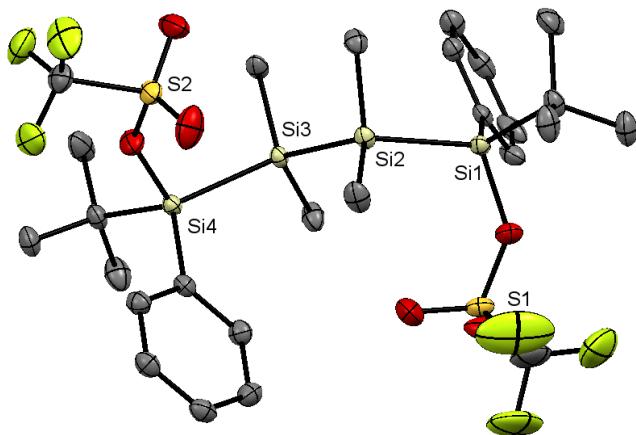
**Figure S63.** ORTEP representation of compound 4



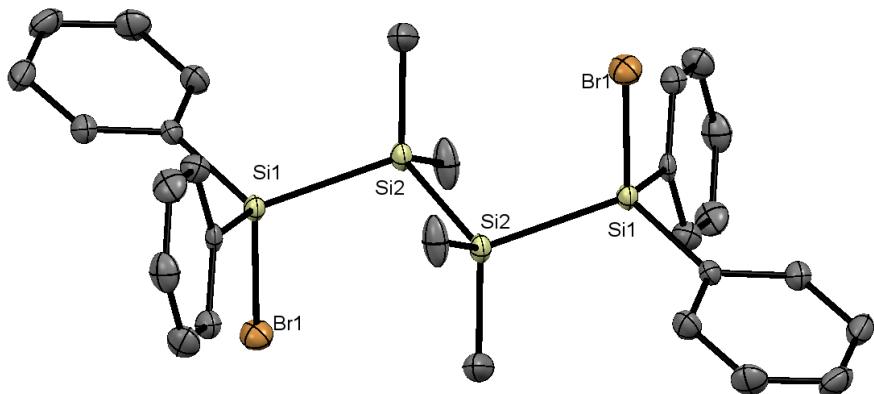
**Figure S64.** ORTEP representation of compound 5



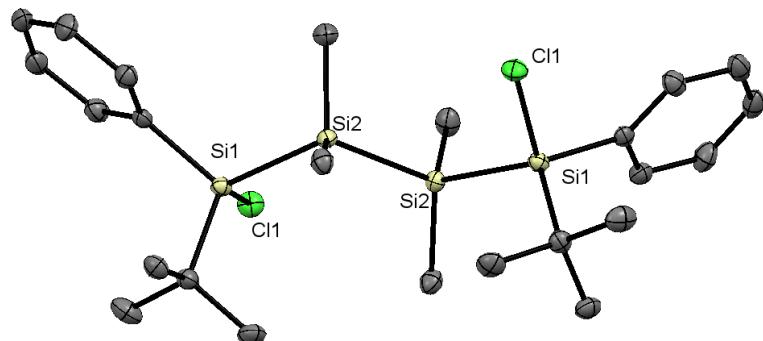
**Figure S65.** ORTEP representation of compound 6



**Figure S66.** ORTEP representation of compound 7



**Figure S67.** ORTEP representation of compound 8b



**Figure S68.** ORTEP representation of compound 9

