

SUPPORTING INFORMATION
FOR

**A library of new bifunctional alkenes obtained by
a highly regiodivergent silylation of 1,5-hexadiene**

Rafał Januszewski*^{1,2}, Bartosz Orwat^{1,3}, Jan Merna⁴, Ireneusz Kownacki*^{1,2}

¹ Faculty of Chemistry, Adam Mickiewicz University in Poznan, Uniwersytetu Poznanskiego 8, 61-614, Poznan, Poland

² Center for Advanced Technology, Adam Mickiewicz University in Poznan, Uniwersytetu Poznanskiego 10, 61-614, Poznan, Poland

³ Department of Molecular Physics, Lodz University of Technology, 90-924 Lodz,
Zeromskiego 116, Poland

⁴ University of Chemistry and Technology in Prague, Technická 5, 166 28, Prague 6, Czech Republic

Correspondence:

*Rafał Januszewski e-mail: r.janusz@amu.edu.pl

*Ireneusz Kownacki e-mail: ireneusz.kownacki@amu.edu.pl

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1. General remarks

1.1. Methods

NMR analysis - ^1H , ^{13}C and ^{29}Si NMR spectra were recorded at 25 °C on Bruker UltraShield 300 or 400 MHz. Chemical shifts were reported in ppm with the reference to the residue portion solvent peak. In all experiments d¹-chloroform was used as a solvent.

GC-MS analysis - The mass spectrum of the products were obtained by GC-MS analysis on a Bruker Scion 436-GC with a 30m Varian DB-5 0.25mm capillary column and a Scion SQ-MS mass spectrometry detector. Two temperature programs were used a) 60 °C (3 min), 10°C/min, 250 °C (30 min), b) 100 °C (3 min), 10°C/min, 280 °C (44.5 min).

1.2. Materials

1,5-hexadiene, 1,1,3,3-tetramethyldisiloxane, dimethylphenylsilane, anhydrous toluene, dimethylcyclohexylsilane, dimethyl(*tert*-butyl)silane, vinyltrimethylsilane, Karstedt's complex were purchased from Sigma-Aldrich. Platinum-octanal/octanol complex (2-2.5% Platinum concentration, dimethylbenzylsialne, dimethylbutylsilane were obtained from Fluorochem. 1,1,3,3,5-pentamethyldisiloxane, H₂PtCl₆, Rhodium (III) chloride were purchased from ABCR. Unsymmetrical disiloxanes were synthesized with the use of previously reported procedures.¹⁻³ 3-chloropropyldimethylsialne was prepared by method published by Daiss and co-workers.⁴ The [{Rh(μ-Cl)(cod)}₂] was prepared according to published method.⁵

¹R. Januszewski, I. Kownacki, H. Maciejewski, B. Marciniec, A. Szymanska *European Journal of Inorganic Chemistry* **2017**, 851-856.

²R. Januszewski, I. Kownacki, H. Maciejewski, B. Marciniec, *Journal of Organometallic Chemistry* **2017**, 846, 263-268.

³R. Januszewski, M. Grzelak, B. Orwat, M. Dutkiewicz, I. Kownacki, *Journal of Catalysis* **2020**, 390, 103-108.

⁴J. O. Daiss, S. Duda-Johner, C. Burschka, U. Holzgrabe, K. Mohr and R. Tacke, *Organometallics*, **2002**, 21, 803–811.

⁵S. Komiya, *Synthesis of Organometallic Compounds: A Practical Guide* (Ed. S. Komiya), Wiley, New York, 1997, p. 442.

1.3. General procedures for functionalization of 1,5-hexadiene

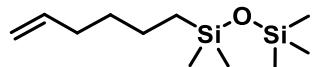
Hydrosilylation of 1,5-hexadiene: 1.68 mmol of silane and 16.8 mmol of 1,5-hexadiene were placed in a glass reactor and stirred at room temperature. To prepared mixture Karstedt's complex was added ([Pt] : [HSi] = 2x10⁻⁵ : 1). The reaction was monitored with GC analysis. After the complete conversion of the organosilicon compound the mixture was filtered through silica to separate the catalyst and was washed with diethyl ether or hexane. Evaporation of the solvent and 1,5-hexadiene excess gave the product as a pale yellow or transparent liquid.

Dehydrogenative silylation of 1,5-hexadiene: 1.68 mmol of silane and 8.4 mmol of 1,5-hexadiene were placed in a glass reactor and dissolved in 2mL of toluene. Prepared mixture was heated up to 50 °C, then the catalyst [2x10⁻⁴ Rh/ per 1 mol of H-Si] [{Rh(μ-Cl)(cod)}₂]

was added. The reaction was monitored with GC analysis. After the complete conversion of silane the mixture was then filtered through silica to separate the catalyst and was washed with diethyl ether or hexane. Evaporation of the solvent and 1,5-hexadiene excess gave the product as a pale yellow or transparent liquid.

2. Analytical data and NMR spectra of isolated products

2.1. Product 2A



¹H NMR (300 MHz, CDCl₃) δ 5.81 CH₂=CHR (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.09 – 4.87 CH₂=CHR (m, 2H), 2.05 (q, *J* = 6.8 Hz, 2H), 1.36 (m, 4H), 0.60 – 0.43 CH₂Si (m, 2H), 0.06 SiMe₃ (s, 9H), 0.04 SiMe₂ (s, 6H).

¹³C NMR (75 MHz, CDCl₃) δ 139.30, 114.28, 33.71, 32.74, 22.94, 18.35, 2.12, 0.49.

²⁹Si NMR (79 MHz, CDCl₃) δ 7.50, 7.07.

MS (EI, m/z): 215.0 [M-15]⁺ (3.5), 149.0 (7.5), 148.1 (14.7), 146.9 (100), 132.9 (44.5), 72.9 (14.1),

Product contains 2% of disubstituted derivative

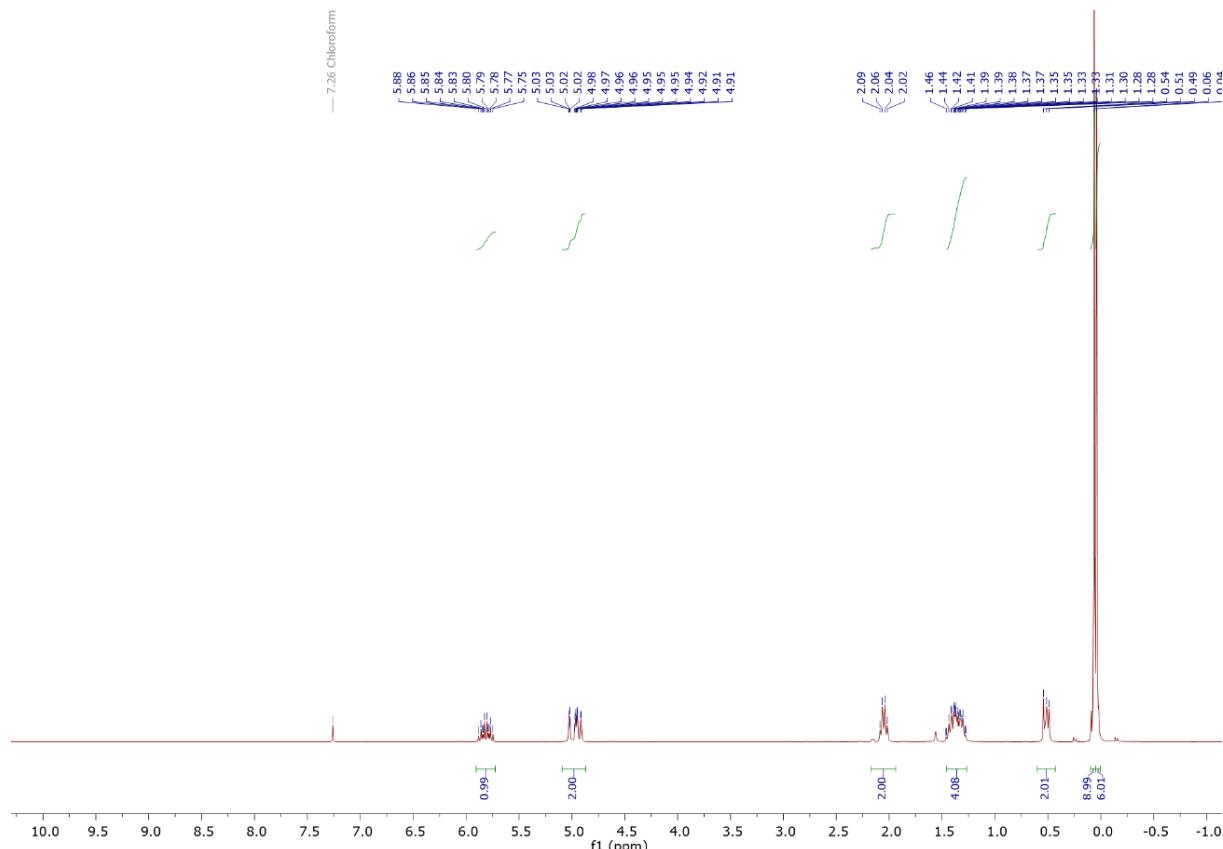


Figure S1. ¹H NMR spectrum of 2A

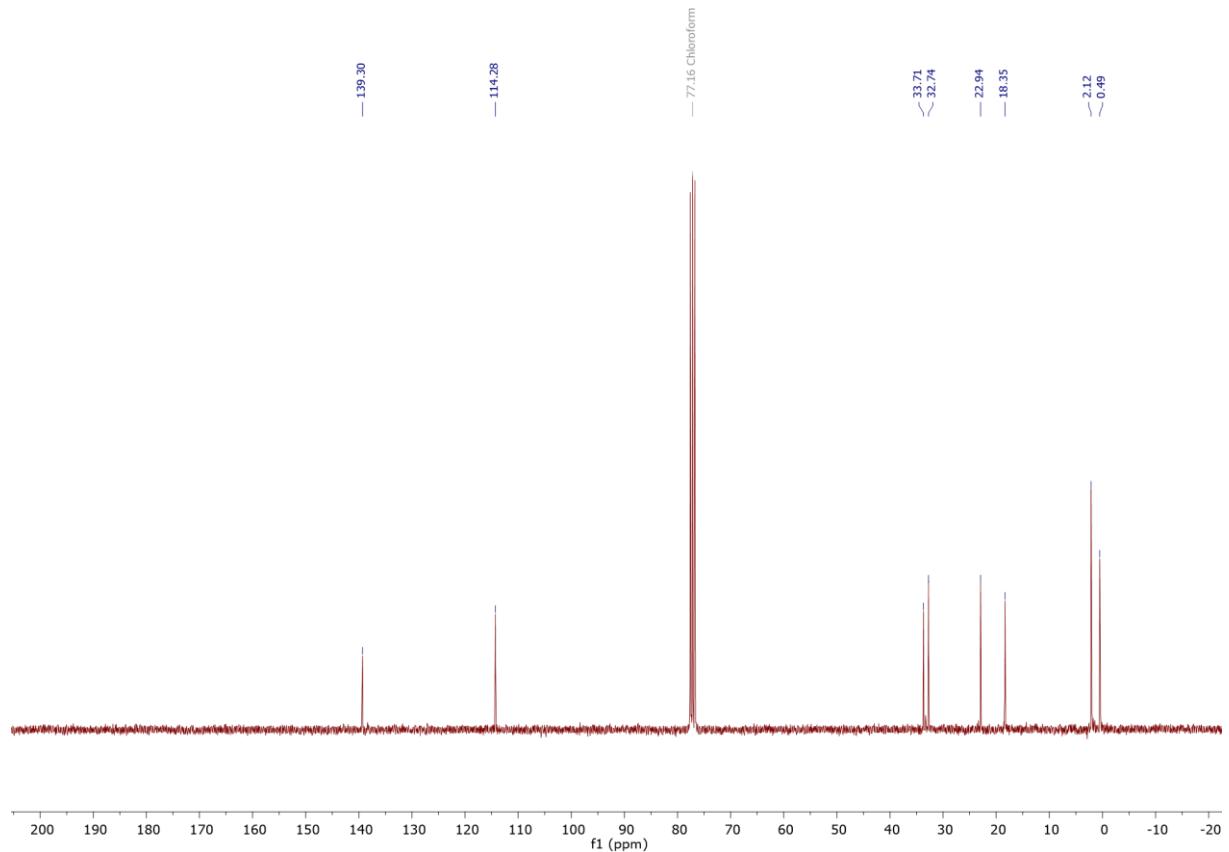


Figure S2. ^{13}C NMR spectrum of 2A

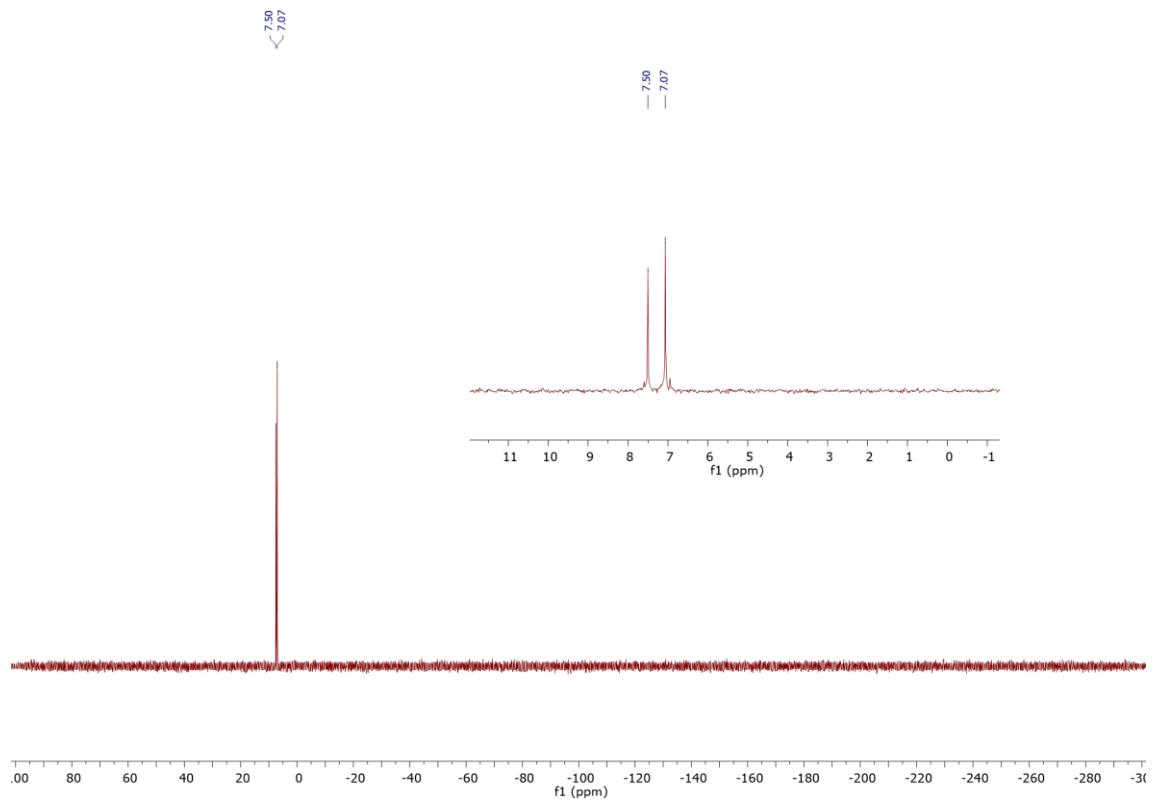
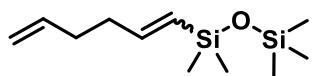


Figure S3. ^{29}Si NMR spectrum of 2A

2.2. Product 2C



¹H NMR (300 MHz, CDCl₃) δ, 6.10 RCH=CHSi (dt, *J* = 18.6, 5.8 Hz, 1H), 5.94 – 5.72 CH₂=CHR (m, 1H), 5.63 RCH=CHSi (d, *J* = 18.7 Hz, 1H), 5.13 – 4.79 CH₂=CHR (m, 2H), 2.19 (m, 4H), 0.11 SiMe₂ (s, 6H), 0.07 SiMe₃ (s, 9H). Isomer β-Z: 6.30 RCH=CHSi (dt, *J* = 14.3, 7.2 Hz, H) 5.47 RCH=CHSi (d, *J* = 14.2 Hz).

¹³C NMR (75 MHz, CDCl₃) δ 147.12, 138.36, 130.11, 114.81, 35.87, 32.94, 2.16, 0.91.

²⁹Si NMR (79 MHz, CDCl₃) δ 7.91, -4.10.

MS (EI, m/z): 213.0 [M-15]⁺ (3.9), 156.9 (5.6), 148.1 (6.5), 146.9 (44.6), 135.0 (7.1), 134.1 (13.6), 132.9 (100), 116.9 (5.2), 72.9 (15.0), 58.9 (5.3)

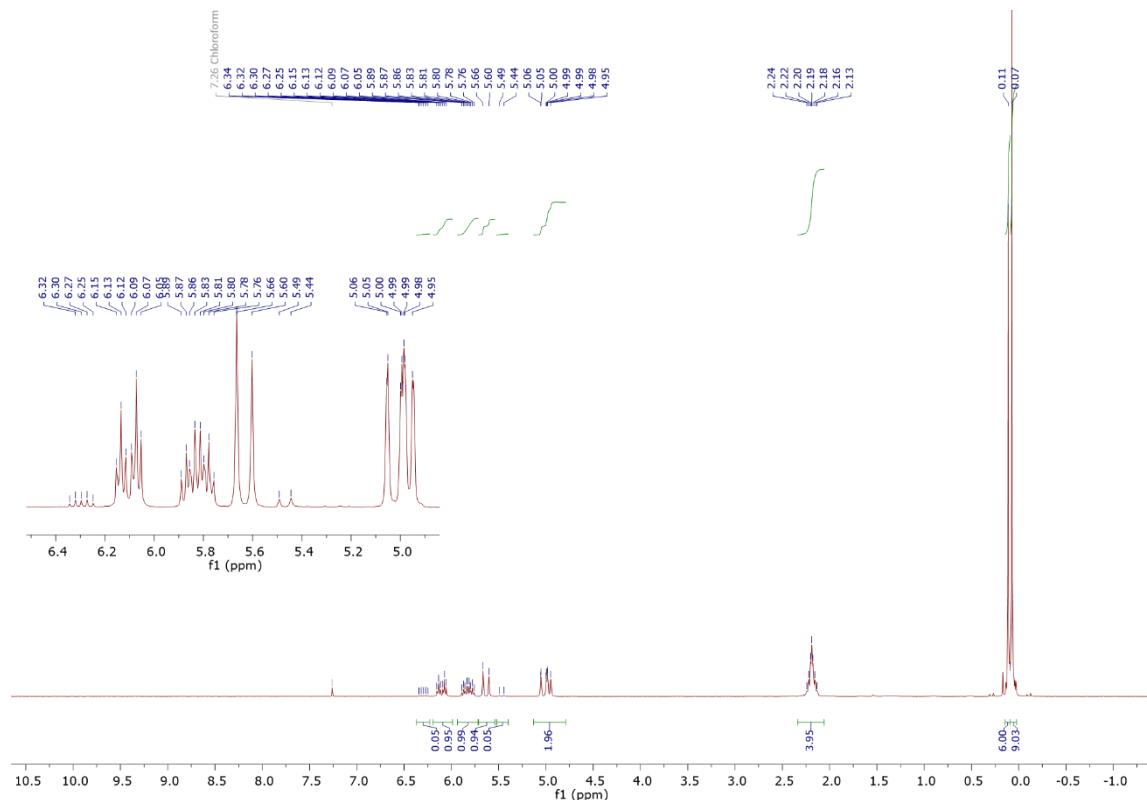


Figure S4. ¹H NMR spectrum of 2C

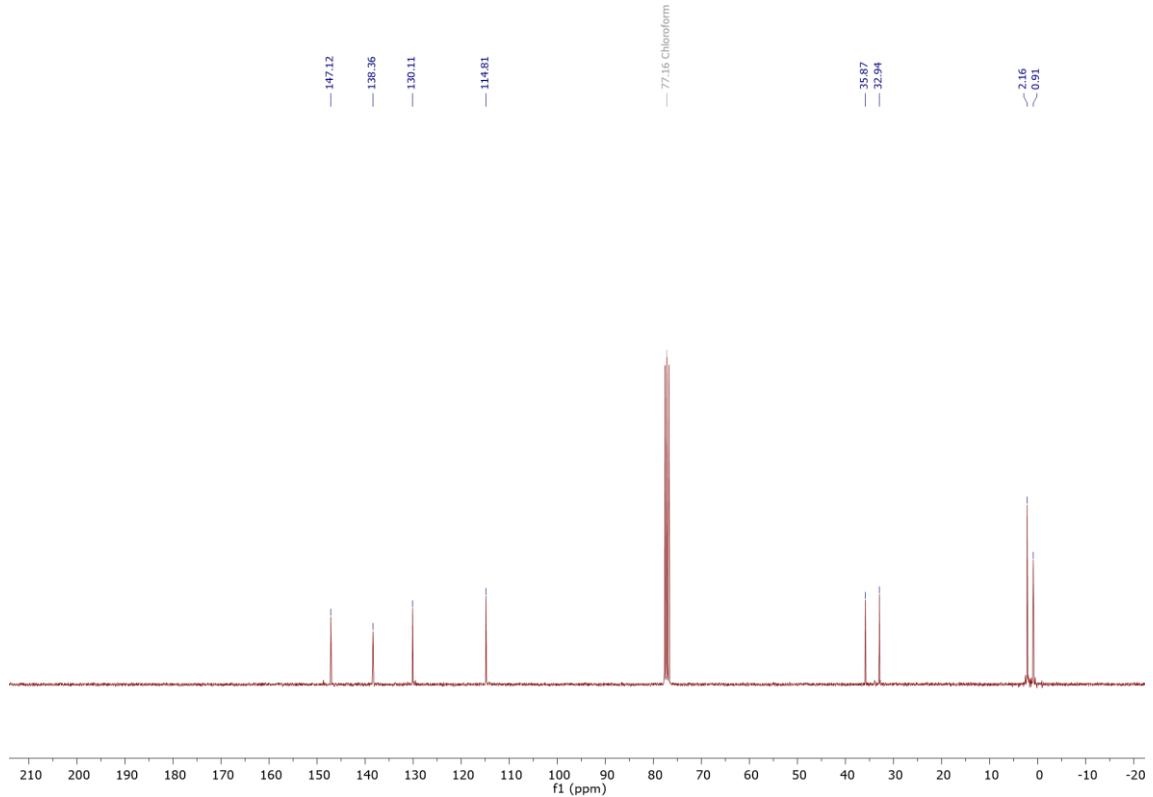


Figure S5. ^{13}C NMR spectrum of 2C

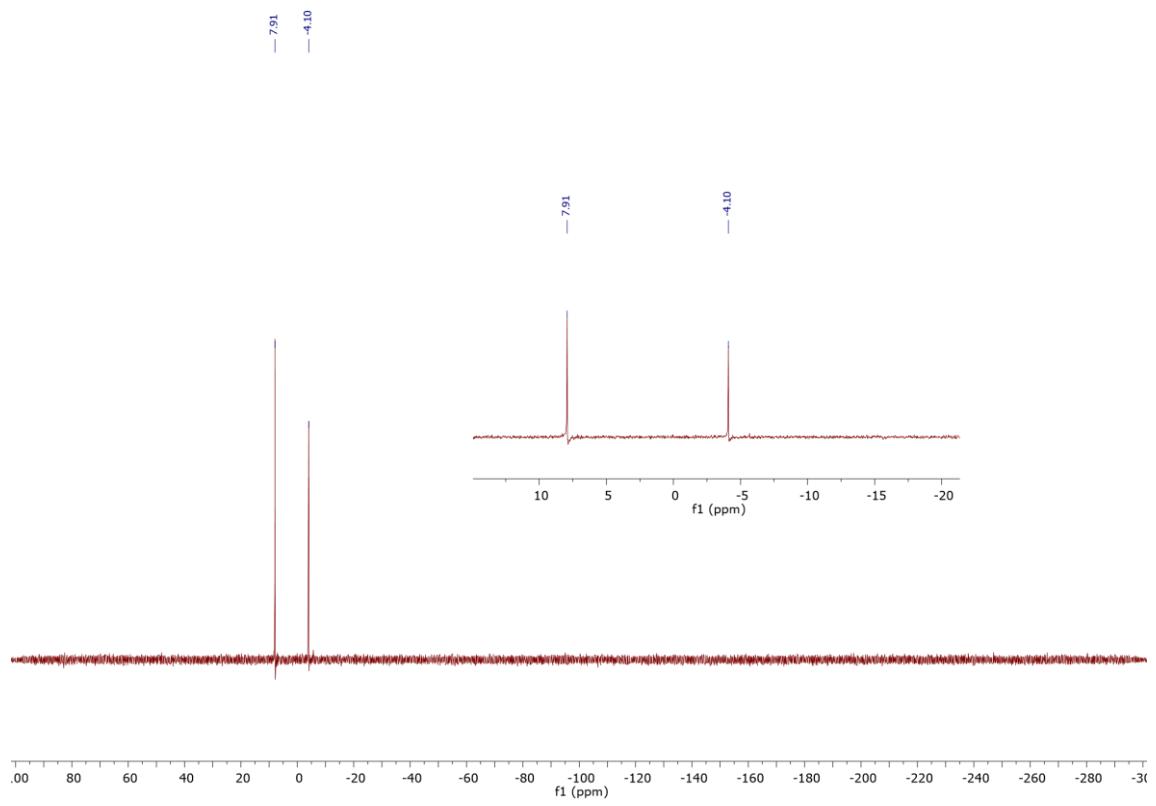
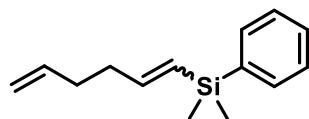


Figure S6. ^{29}Si NMR spectrum of 2C

2.3. Product 3a



$^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.59 – 7.50 (m, 2H), 7.40 – 7.32 (m, 3H), 6.14 $\text{RCH}=\text{CHSi}$ (dt, $J = 18.6, 5.8$ Hz, 1H), 5.92 – 5.73 $\text{CH}_2=\text{CHR}$, $\text{RCH}=\text{CHSi}$ (m, 2H), 5.13 – 4.89 $\text{CH}_2=\text{CHR}$ (m, 2H), 2.23 (m, 4H), 0.34 SiMe_2 (s, 6H), Isomer β -Z: 6.44 $\text{RCH}=\text{CHSi}$ (dt, $J = 14.1, 7.1$ Hz), 5.69 $\text{RCH}=\text{CHSi}$ (d, $J = 14.9$ Hz).

$^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 148.44, 139.36, 138.32, 133.97, 128.97, 128.03, 127.85, 114.85, 36.14, 32.99, -2.31.

$^{29}\text{Si NMR}$ (79 MHz, CDCl_3) δ -11.92.

MS (EI, m/z): 216.1 M^+ (2.2), 201.0 [M-15] $^+$ (16.0), 175.0 (23.3), 173.0 (5.7), 158.9 (13.5), 144.9 (15.4), 137.9 (19.1), 136.1 (8.3), 134.9 (59.0), 130.9 (5.6), 123.1 (9.9), 122.1 (9.8), 120.9 (100), 104.8 (13.1), 98.9 (46.0), 94.9 (7.8), 80.1 (7.4) 73.0 (11.1), 58.9 (29.5).

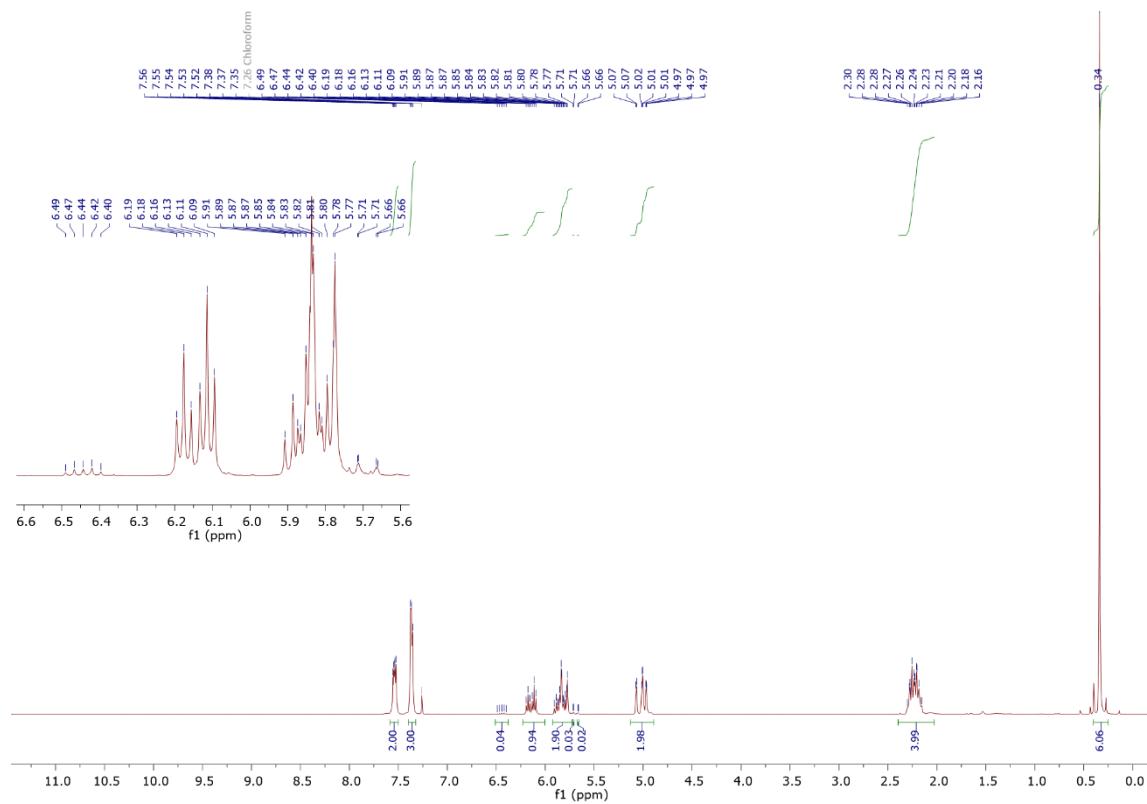


Figure S7. $^1\text{H NMR}$ spectrum of 3a

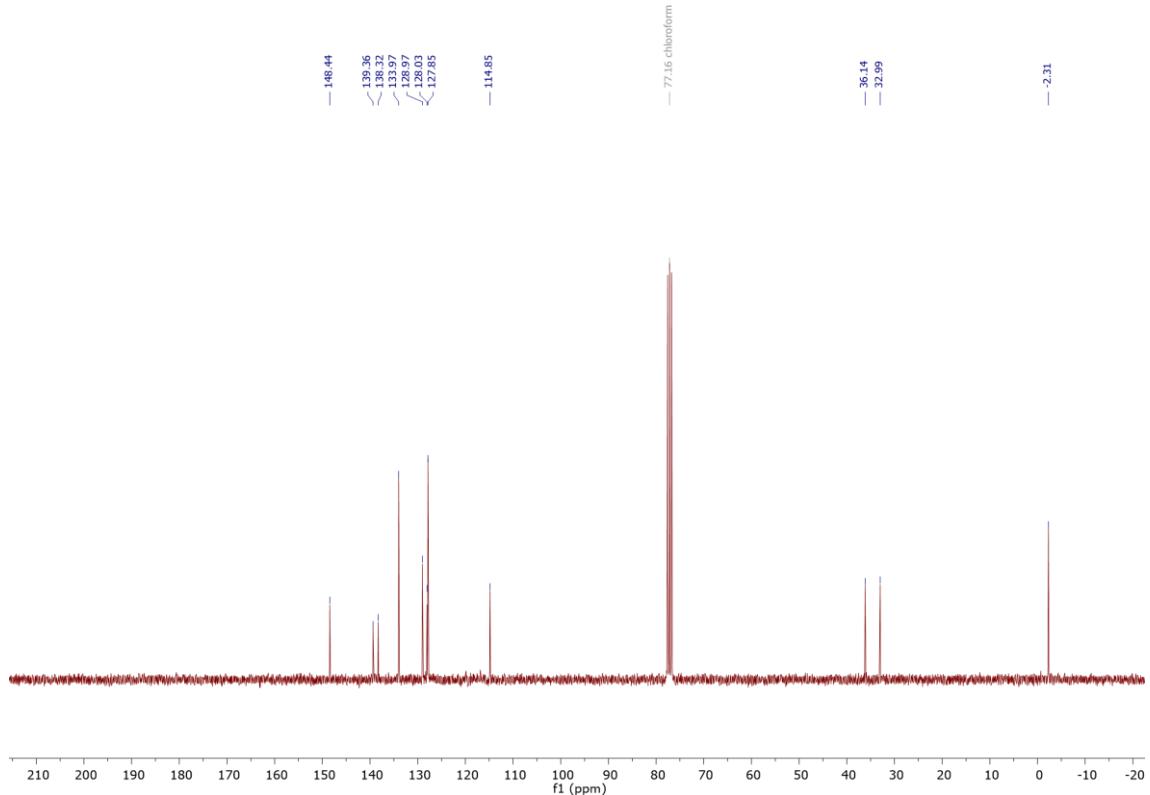


Figure S8. ¹³C NMR spectrum of 3a

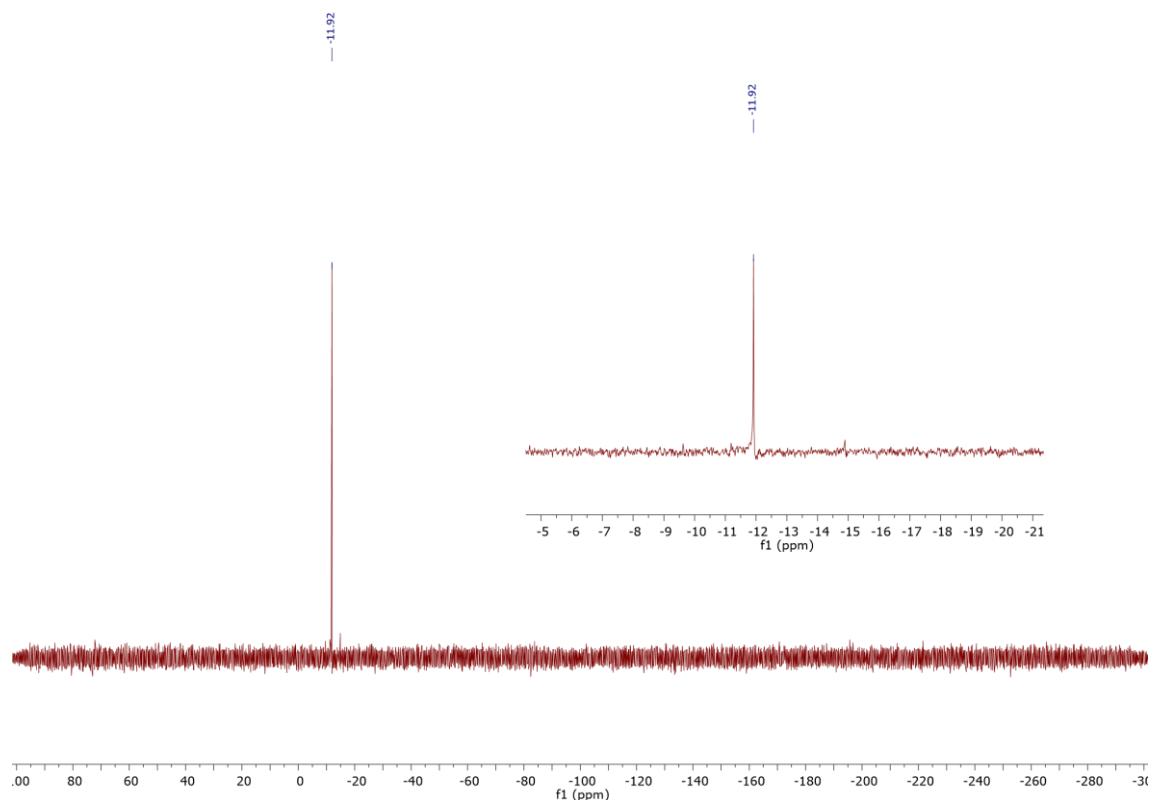
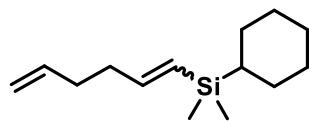


Figure S9. ^{29}Si NMR spectrum of 3a

2.4. Product 3b



¹H NMR (300 MHz, CDCl₃) δ 6.01 RCH=CHSi (dt, *J* = 18.6, 5.8 Hz, 1H), 5.82 (ddt, *J* = 16.6, 10.4, 6.3 Hz, 1H), 5.61 RCH=CHSi (d, *J* = 18.7 Hz, 1H), 5.10 – 4.85 (m, 2H), 2.18 (m, 4H), 1.68 (m, 6H), 1.27 – 0.98 (m, 5H), 0.67 – 0.50 SiCH (m, 1H), -0.02 SiMe₂ (s, 6H). Isomer β -Z: 6.33 RCH=CHSi (dt, *J* = 13.9, 6.9 Hz,), 5.47 RCH=CHSi (d, *J* = 13.1 Hz).

¹³C NMR (101 MHz, CDCl₃) δ 147.20, 138.45, 128.17, 114.72, 36.25, 33.18, 28.27, 27.60, 27.17, 25.91, -4.90.

²⁹Si NMR (79 MHz, CDCl₃) δ -5.79.

MS (EI, m/z): 207.0 [M-15]⁺ (4.8), 141.0 (12.0), 140.1 (13.8), 138.9 (86.6), 110.9 (16.0), 97.0 (14.1), 84.9 (9.7), 82.9 (9.9), 79.0 (57.5), 73.0 (34.8), 60.1 (6.8), 58.9 (100)

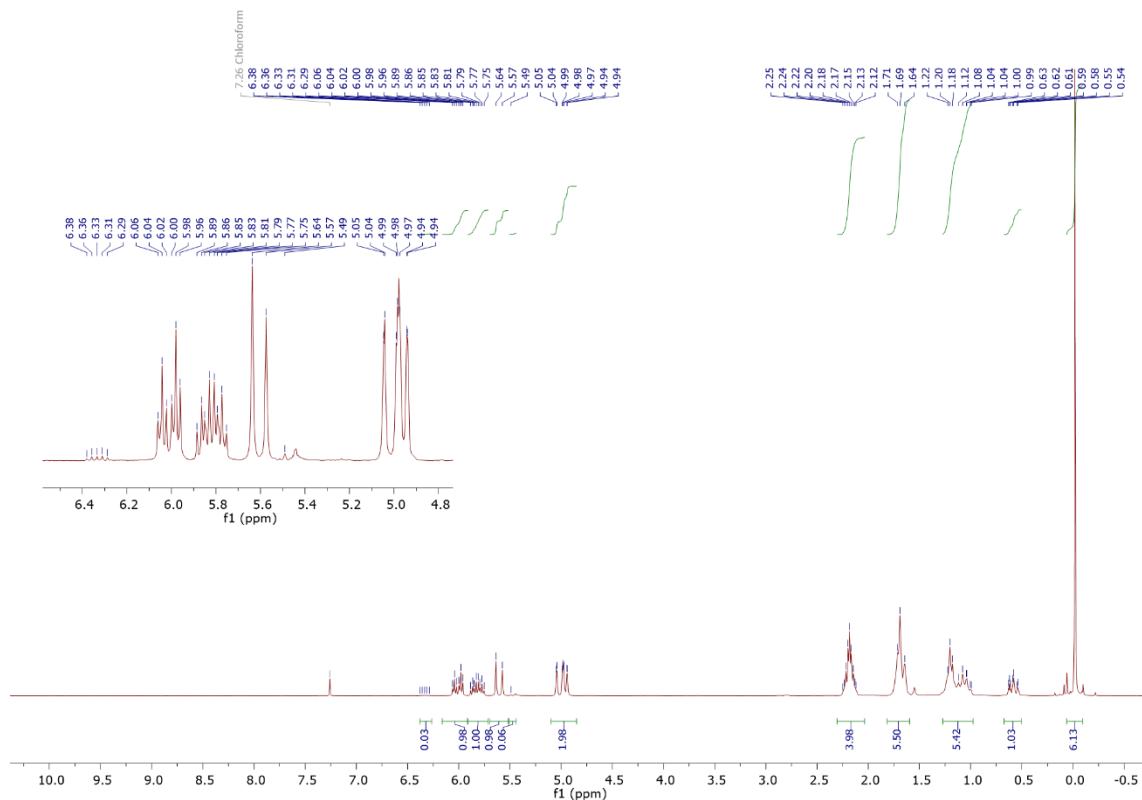


Figure S10. ^1H NMR spectrum of 3b

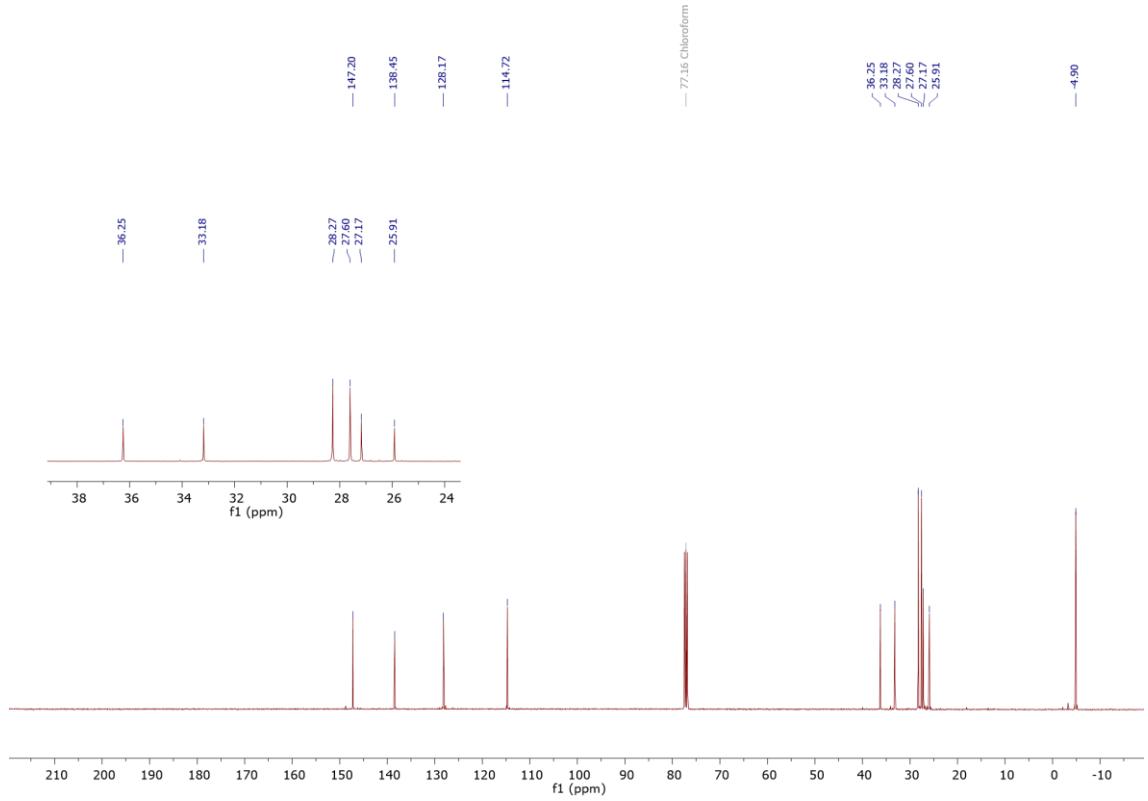


Figure S11. ^{13}C NMR spectrum of 3b

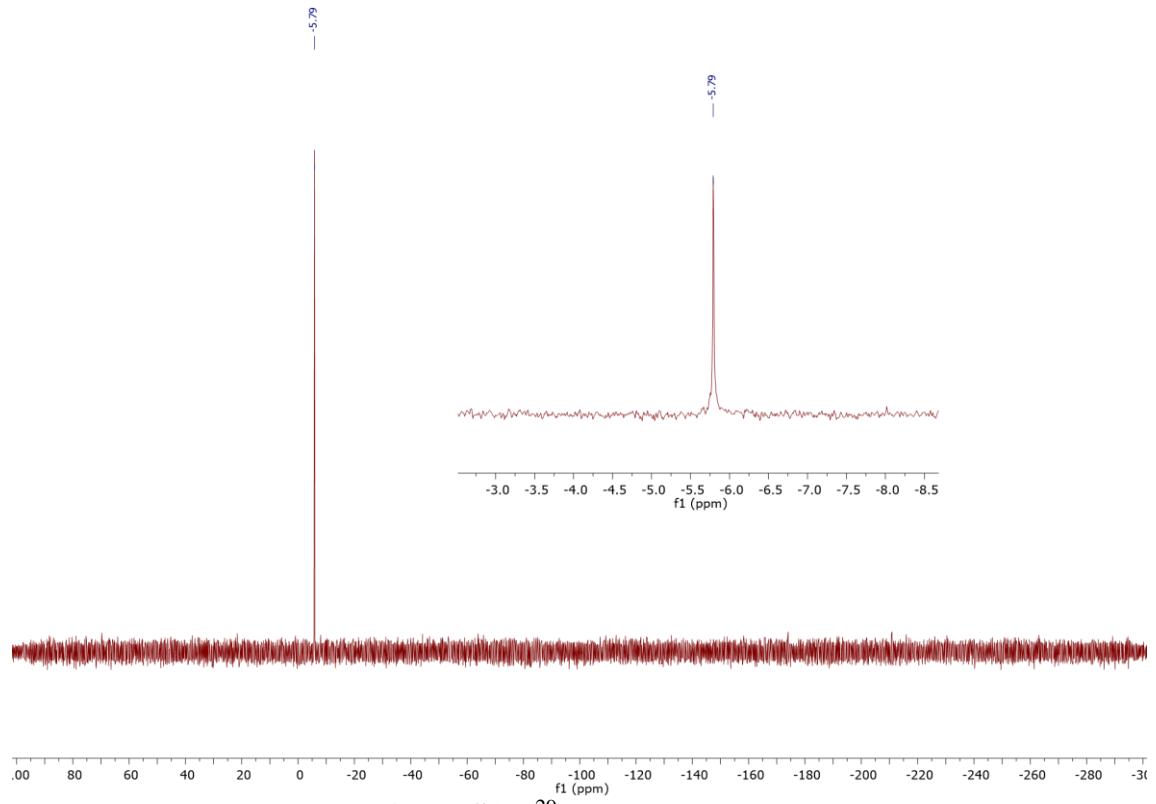
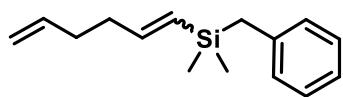


Figure S12. ^{29}Si NMR spectrum of 3b

2.5. Product 3c

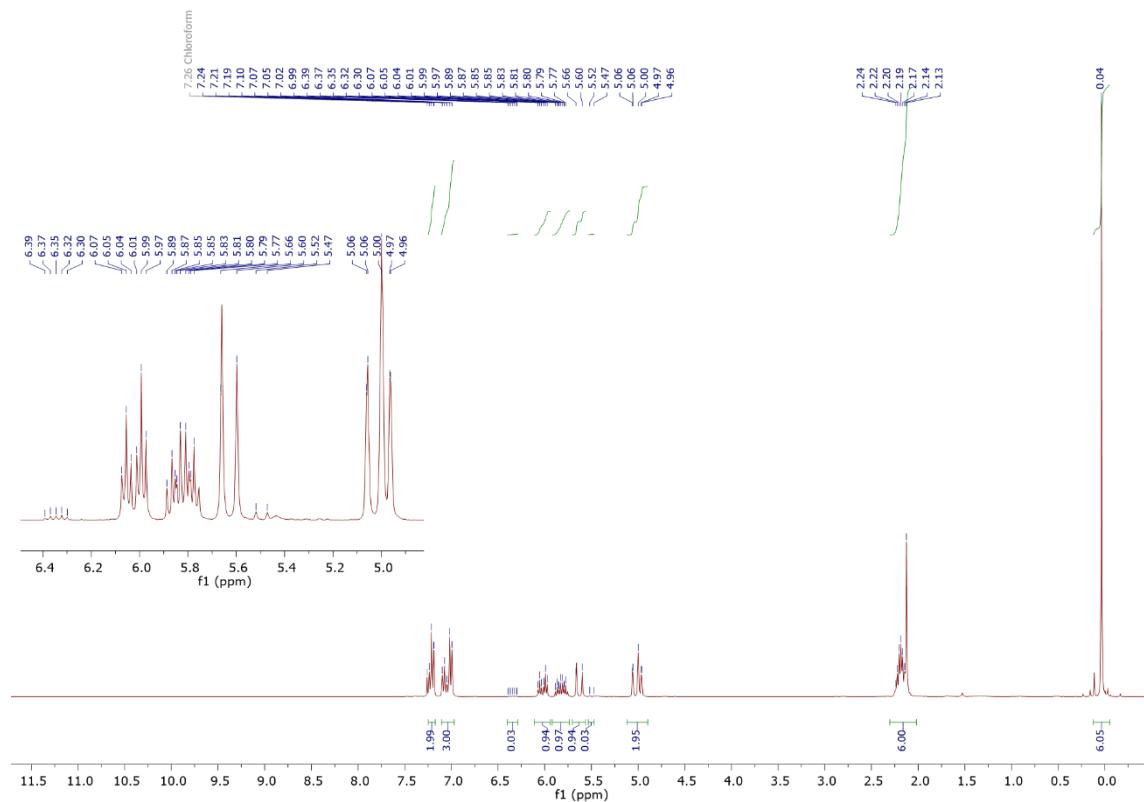


¹H NMR (300 MHz, CDCl₃) δ 7.21 (m, 2H), 7.05 (m, 3H), 6.02 RCH=CHSi (dt, *J* = 18.6, 5.8 Hz, 1H), 5.92 – 5.74 CH₂=CHR (m, 1H), 5.63 RCH=CHSi (d, *J* = 18.6 Hz, 1H), 5.12 – 4.90 CH₂=CHR (m, 2H), 2.30 – 2.02 (m, 6H), 0.04 SiMe₂ (s, 6H).

¹³C NMR (75 MHz, CDCl₃) δ 147.71, 140.32, 138.34, 128.40, 128.24, 128.17, 124.01, 114.80, 36.13, 32.98, 26.37, -3.16. Isomer β-Z: 6.35 RCH=CHSi (dt, *J* = 14.0, 6.9 Hz), 5.49 (d, *J* = 14.0 Hz)

²⁹Si NMR (79 MHz, CDCl₃) δ -7.40.

MS (EI, m/z): 230.0 (2.7) M⁺, 215.0 [M-15]⁺ (2.4), 140.1 (10.4), 139.0 (78.4), 120.9 (5.9), 110.9 (14.4), 84.9 (9.2), 82.9 (10.3), 79.0 (47.3), 73.0 (26.6), 60.1 (6.9), 59.0 (100),



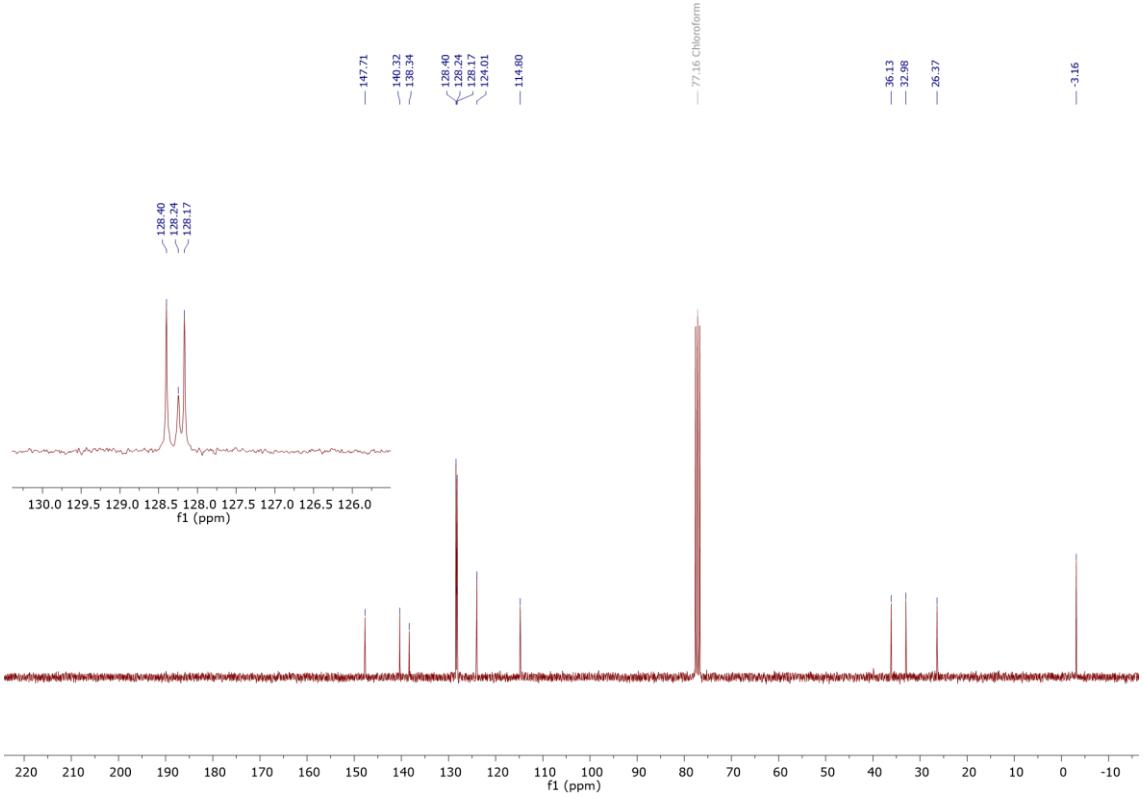


Figure S14. ^{13}C NMR spectrum of 3c

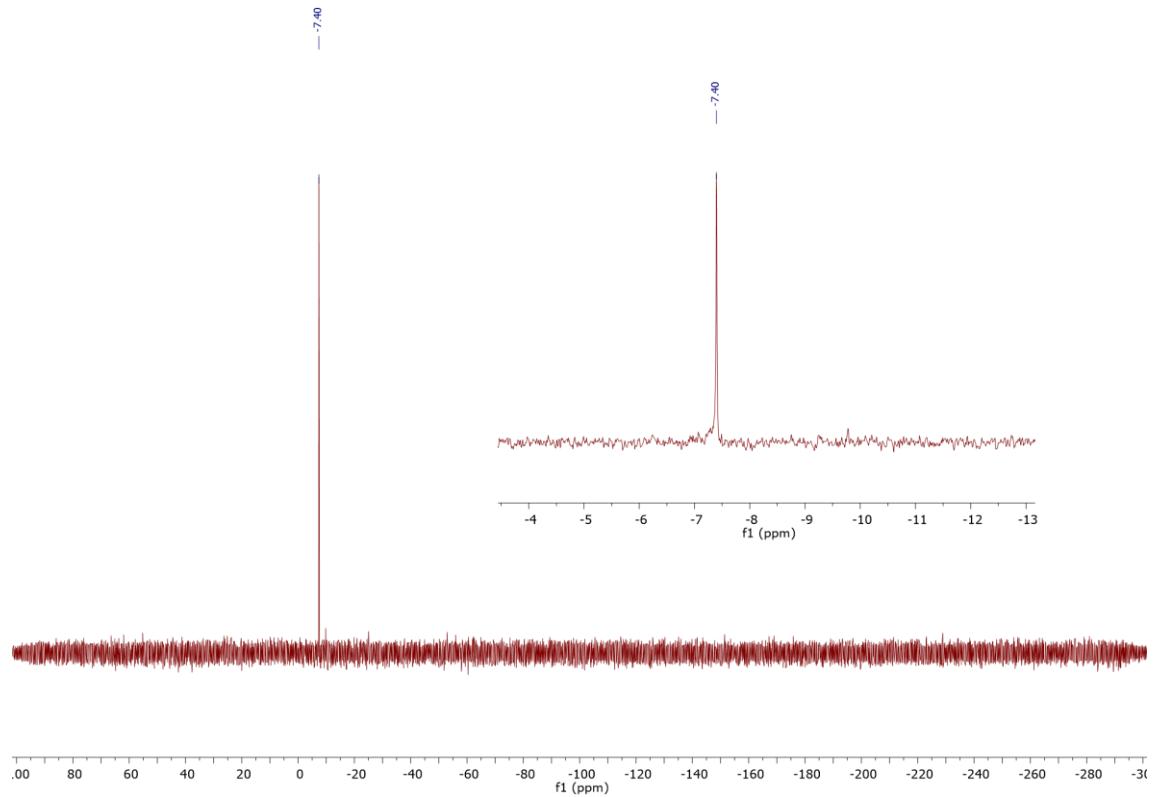
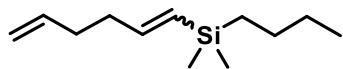


Figure S15. ^{29}Si NMR spectrum of 3c

2.6. Product 3d

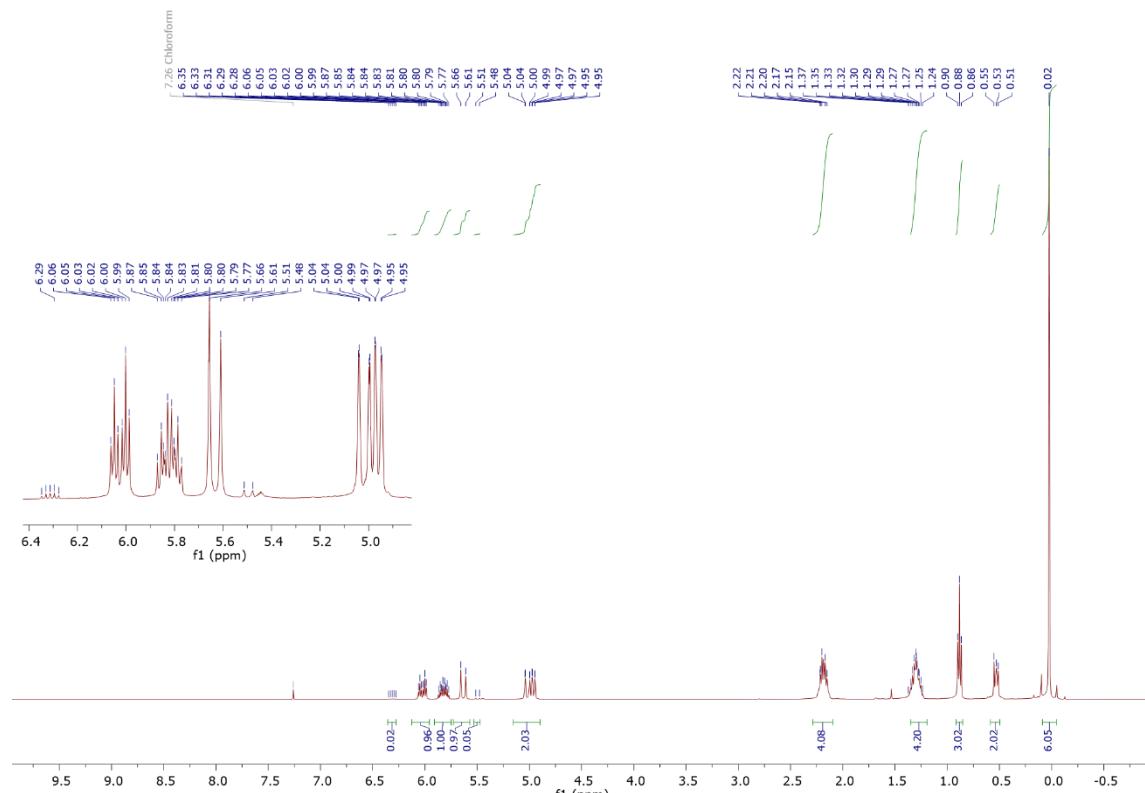


$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 6.02 $\text{RCH}=\text{CHSi}$ (dt, $J = 18.6, 5.8$ Hz, 1H), 5.82 $\text{CH}_2=\text{CHR}$ (ddt, $J = 16.4, 10.3, 6.2$ Hz, 1H), $\text{RCH}=\text{CHSi}$ 5.63 (d, $J = 18.6$ Hz, 1H), 5.15 – 4.90 $\text{CH}_2=\text{CHR}$ (m, 2H), 2.29 – 2.10 (m, 4H), 1.29 (m, 4H), 0.88 CH_3 (t, $J = 7.0$ Hz, 3H), 0.59 – 0.50 SiCH_2 (m, 2H), 0.02 SiMe_2 (s, 6H), Isomer β -Z: 6.31 $\text{RCH}=\text{CHSi}$ (dt, $J = 14.2, 7.1$ Hz), 5.50 $\text{RCH}=\text{CHSi}$ (d, $J = 14.0$ Hz)

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 146.72, 138.44, 129.42, 114.72, 36.18, 33.13, 26.70, 26.29, 15.63, 13.97, -2.82.

$^{29}\text{Si NMR}$ (79 MHz, CDCl_3) δ -6.84.

MS (EI, m/z): 181.0 [$\text{M}-15$]⁺ (5.1), 140.2 (6.1), 139.0 (41.5), 124.9 (5.6), 114.9 (22.7), 110.9 (13.0), 99.2 (6.9), 96.9 (26.7), 82.9 (9.2), 80.1 (14.4), 79.0 (45.7), 73.0 (39.5), 70.9 (5.6), 60.1 (6.9), 59.0 (100).



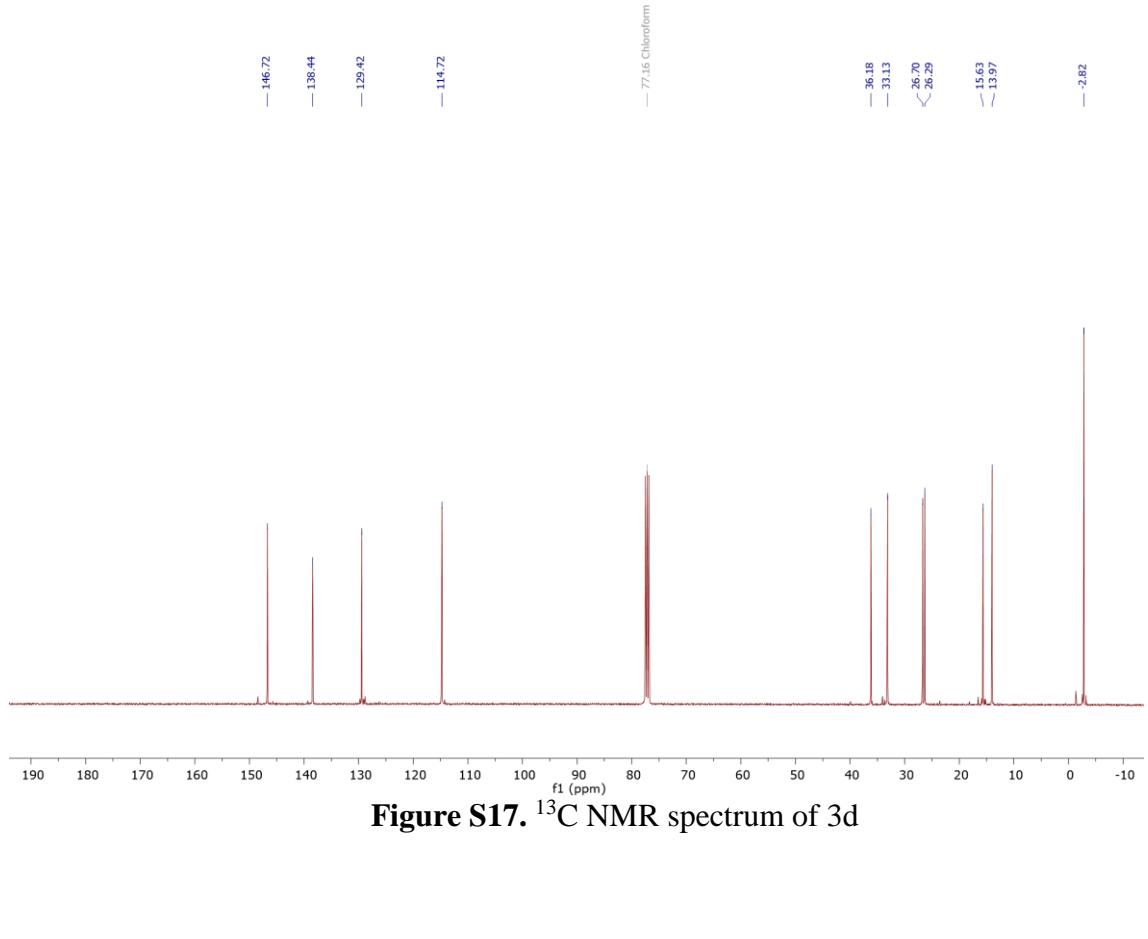


Figure S17. ^{13}C NMR spectrum of 3d

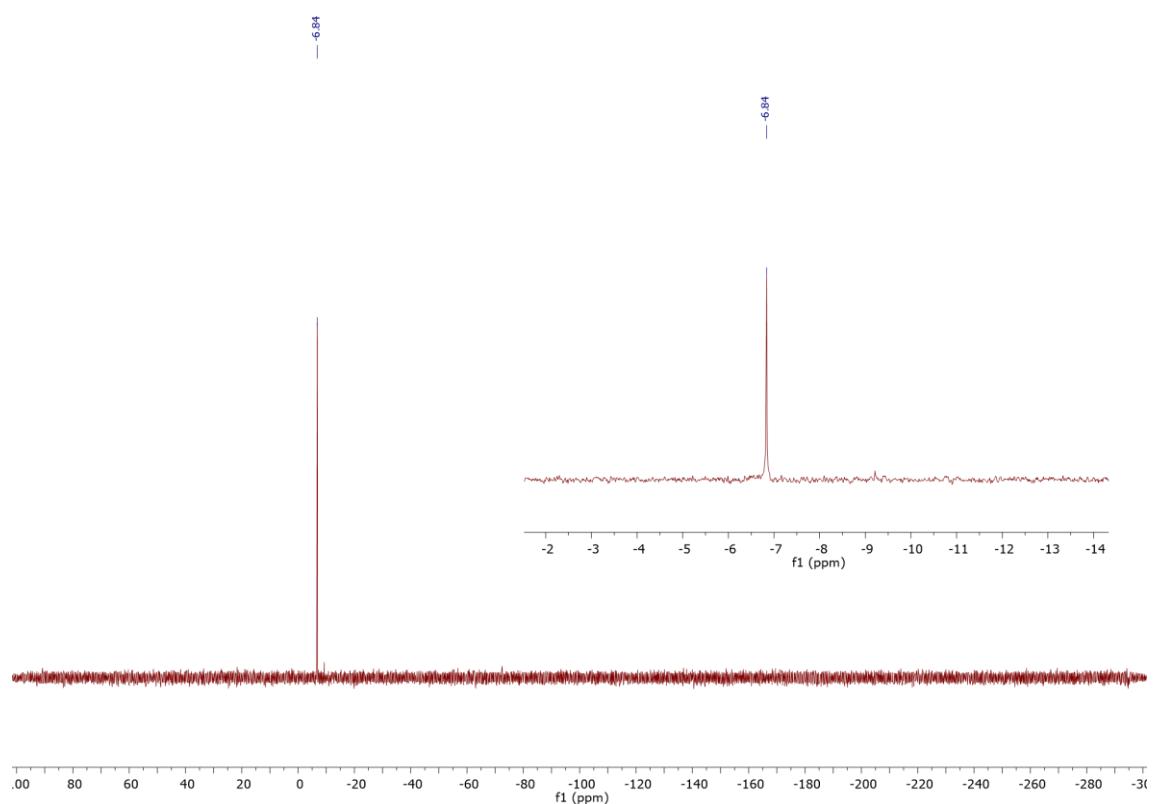
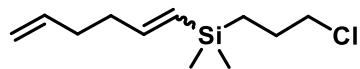


Figure S18. ^{29}Si NMR spectrum of 3d

2.7. Product 3f



¹H NMR (300 MHz, CDCl₃) δ 6.04 RCH=CHSi (dt, *J* = 18.6, 5.8 Hz, 1H), 5.81 CH₂=CHR (ddt, *J* = 16.6, 10.3, 6.3 Hz, 1H), 5.61 RCH=CHSi (d, *J* = 18.6 Hz, 1H), 5.13 – 4.83 CH₂=CHR (m, 2H), 3.50 CH₂Cl (t, *J* = 7.0 Hz, 2H), 2.18 (m, 4H), 1.76 (m, 2H), 0.69 – 0.56 SiCH₂ (m, 2H), 0.05 SiMe₂ (s, 6H), Isomer β-Z: 6.34 RCH=CHSi (dt, *J* = 14.0, 7.0 Hz), 5.47 RCH=CHSi (d, *J* = 14.1 Hz).

¹³C NMR (75 MHz, CDCl₃) δ 147.58, 138.28, 128.38, 114.85, 48.16, 36.12, 33.02, 27.84, 13.61, -2.97.

²⁹Si NMR (79 MHz, CDCl₃) δ -6.49.

MS (EI, m/z): 201.0 [M-15]⁺ (5.6), 158.9 (21.4), 138.9 (17.2), 134.8 (6.6), 130.9 (5.2), 122.9 (17.1), 110.9 (11.0), 104.9 (8.6), 97.1 (9.3), 94.8 (42.1), 93.9 (5.9), 92.9 (100), 84.9 (7.4), 82.9 (6.9), 81.1 (13.4), 80.1 (44.7), 79.0 (70.4), 73.0 (20.6), 66.9 (5.6), 64.9 (6.3), 60.1 (6.7), 59.0 (86.7)

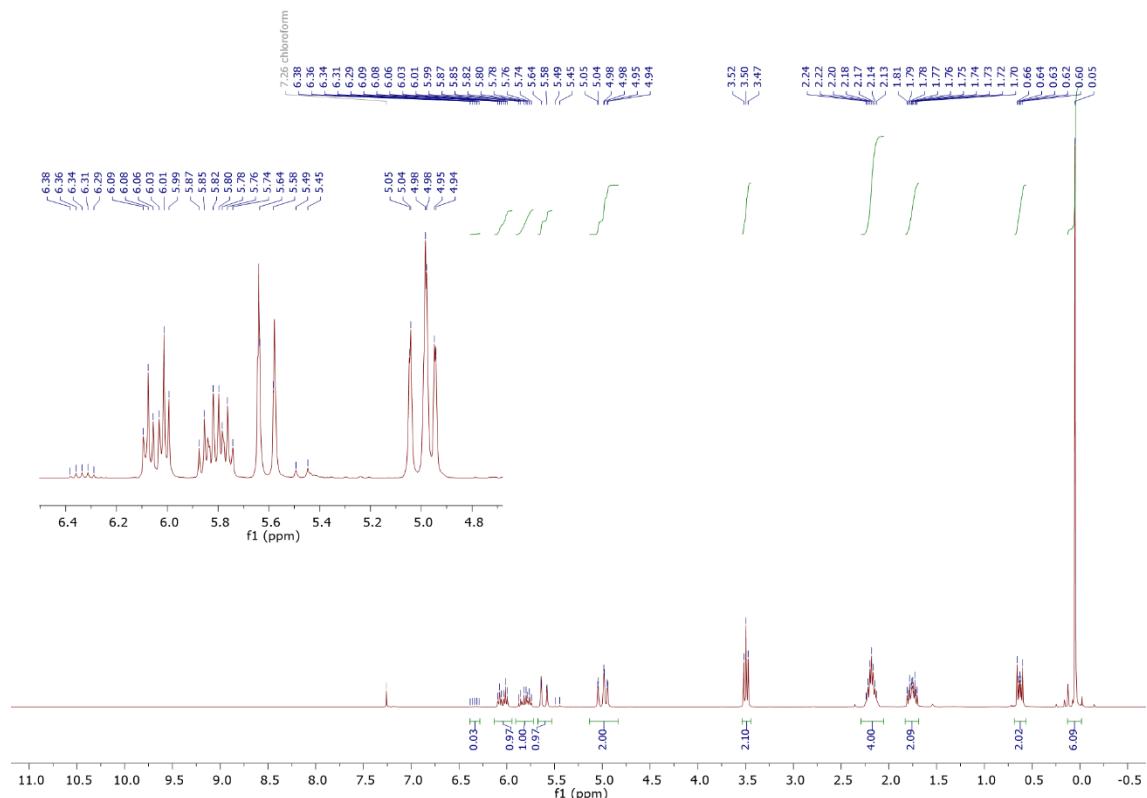


Figure S19. ^1H NMR spectrum of 3f

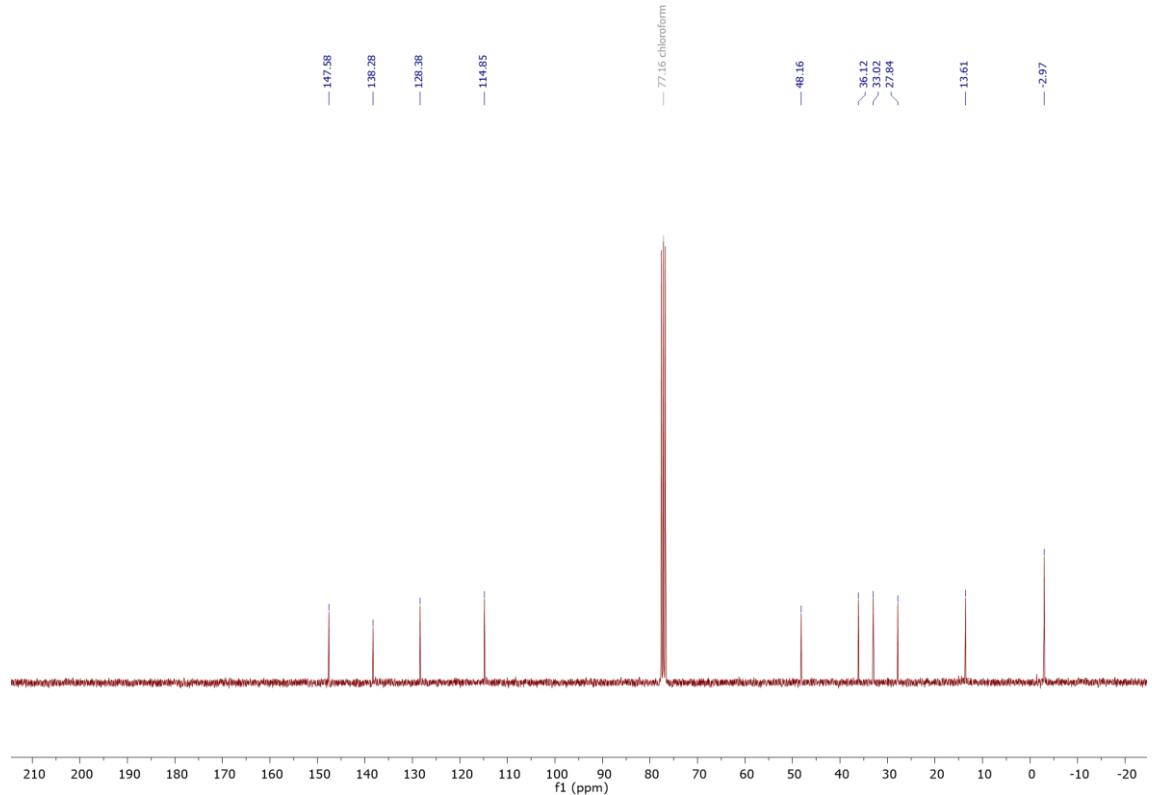


Figure S20. ^{13}C NMR spectrum of 3f

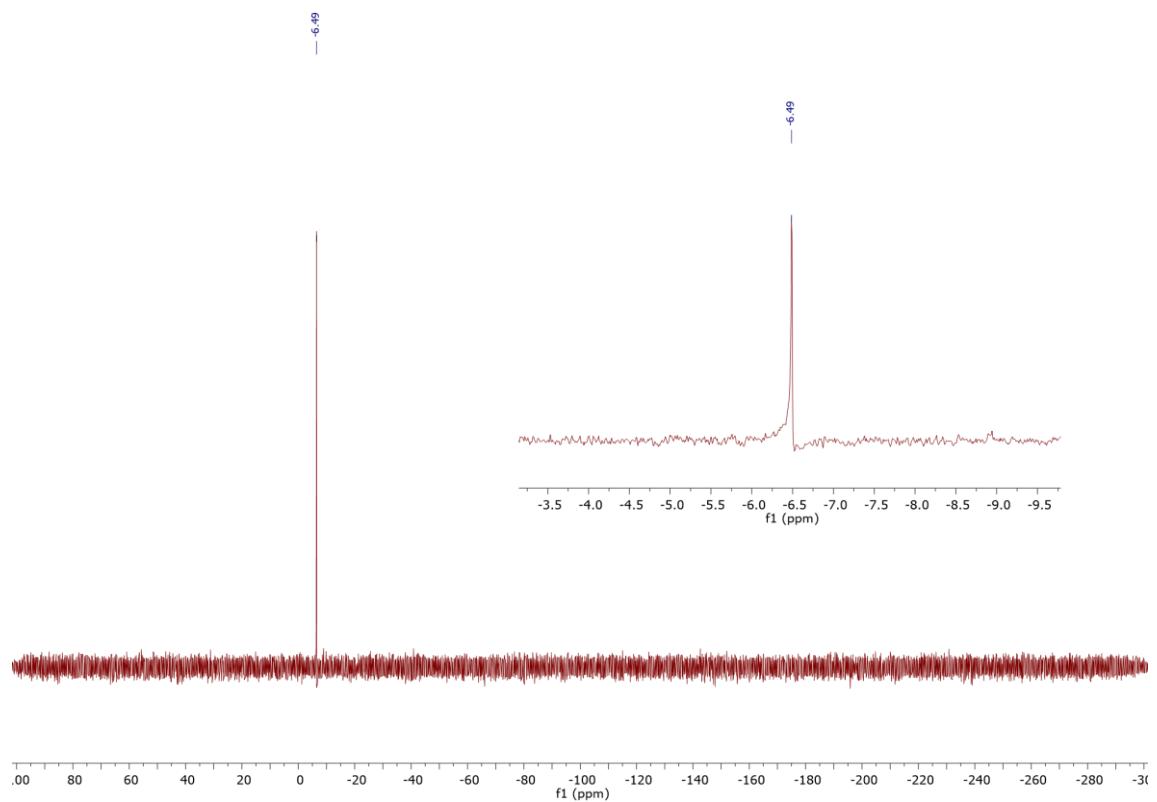
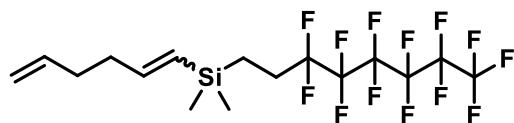


Figure S21. ^{29}Si NMR spectrum of 3f

2.8. Product 3g



¹H NMR (300 MHz, CDCl₃) δ 6.07 RCH=CHSi (dt, *J* = 18.7, 5.9 Hz, 1H), 5.80 CH₂=CHR (ddt, *J* = 16.5, 10.2, 6.3 Hz, 1H), 5.59 RCH=CHSi (d, *J* = 18.7 Hz, 1H), 5.08 – 4.88 CH₂=CHR (m, 2H), 2.32 – 2.10 (m, 4H), 2.09 – 1.86 (m, 2H), 0.85 – 0.70 SiCH₂ (m, 2H), 0.09 SiMe₂ (s, 6H), Isomer β-Z: 6.38 RCH=CHSi (dt, *J* = 14.4, 7.3 Hz), 5.45 RCH=CHSi (d, *J* = 14.0 Hz).

¹³C NMR (75 MHz, CDCl₃) δ 148.62, 138.11, 127.28, 114.95, 36.10, 32.94, 26.04, 5.20, -3.28.

²⁹Si NMR (79 MHz, CDCl₃) δ -5.54.

MS (EI, m/z): 288.9 (4.5), 244.9 (6.8), 238.9 (11.3), 140.2 (8.8), 138.9 (67.4), 114.9 (9.4), 110.9 (18.0), 88.9 (18.4), 84.9 (9.0), 80.9 (26.3), 79.0 (79.7), 77.9 (7.4), 76.9 (81.9), 73.0 (36.4), 68.9 (10.2), 62.9 (16.6), 59.0 (100)

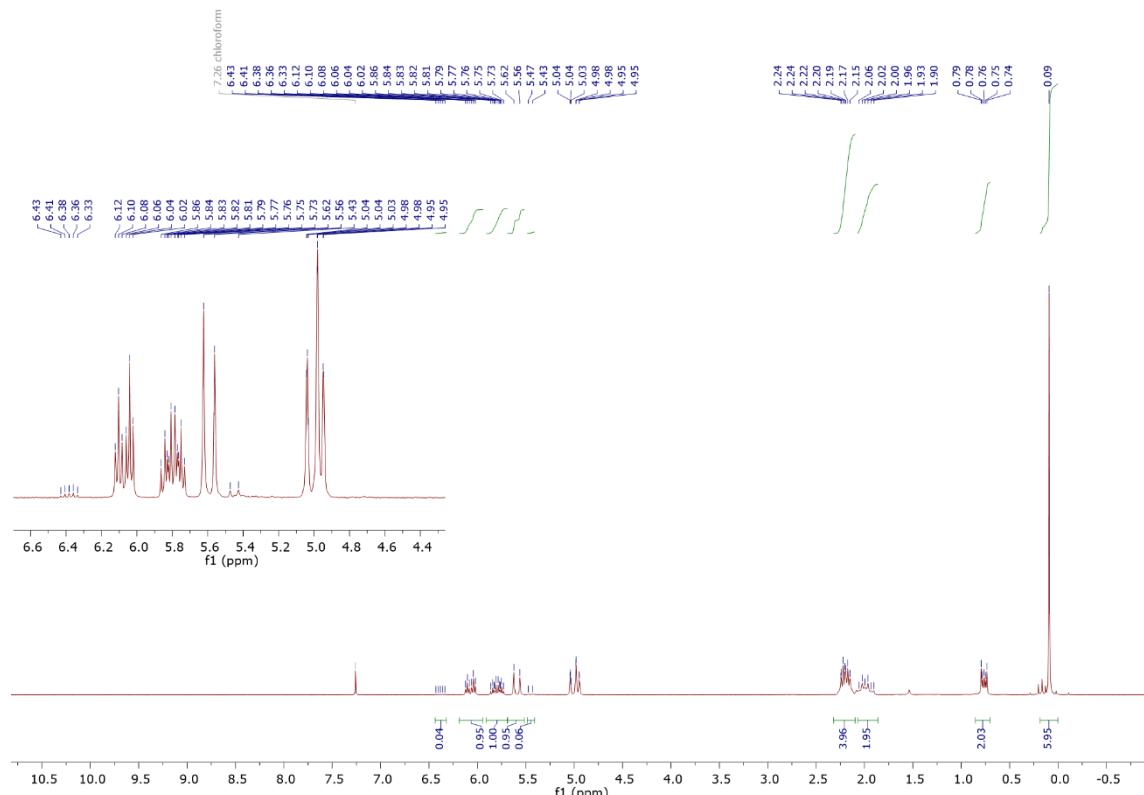


Figure S22. ^1H NMR spectrum of 3g

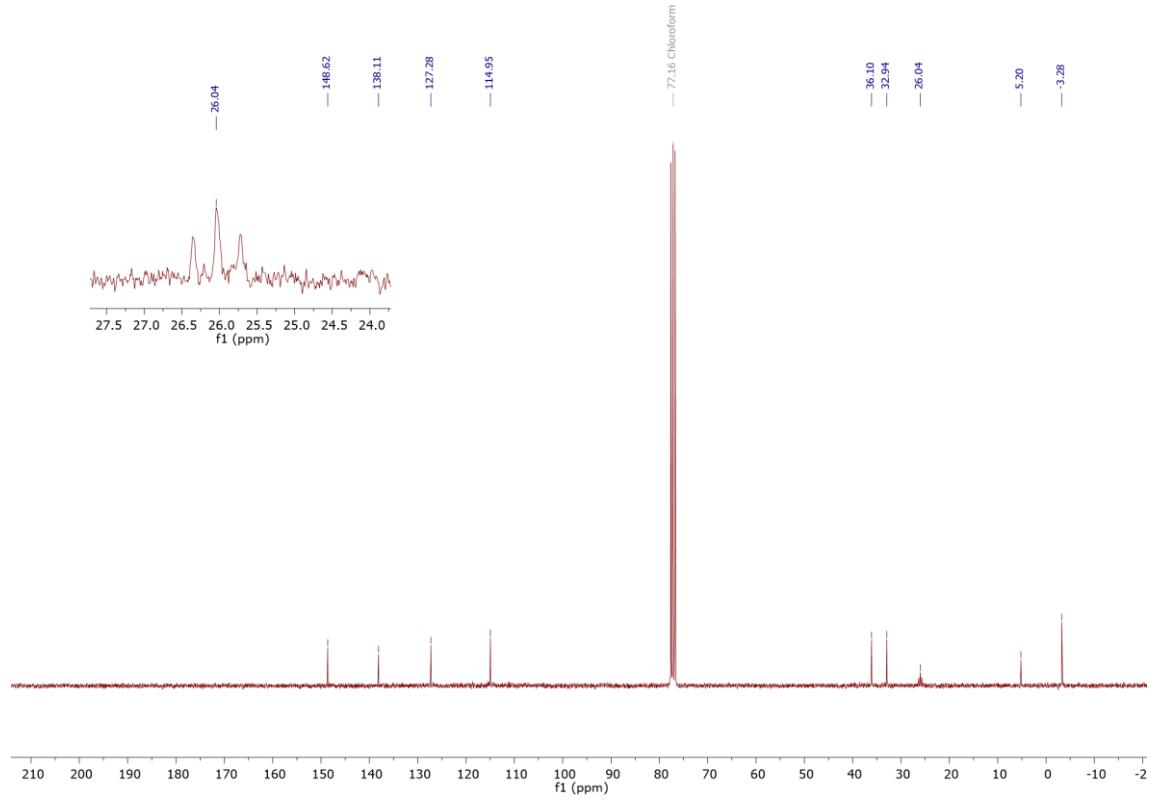


Figure S23. ^{13}C NMR spectrum of 3g

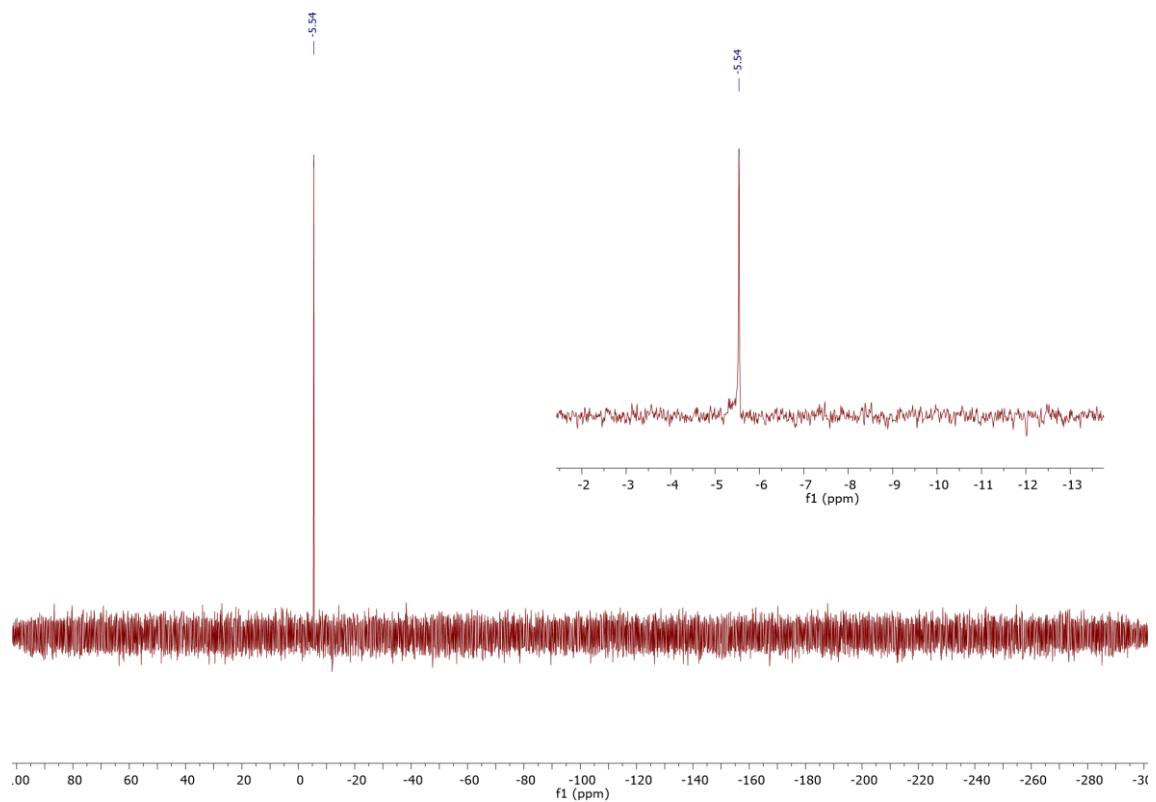
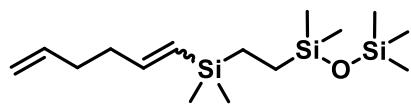


Figure S24. ^{29}Si NMR spectrum of 3g

2.9. Product 3h

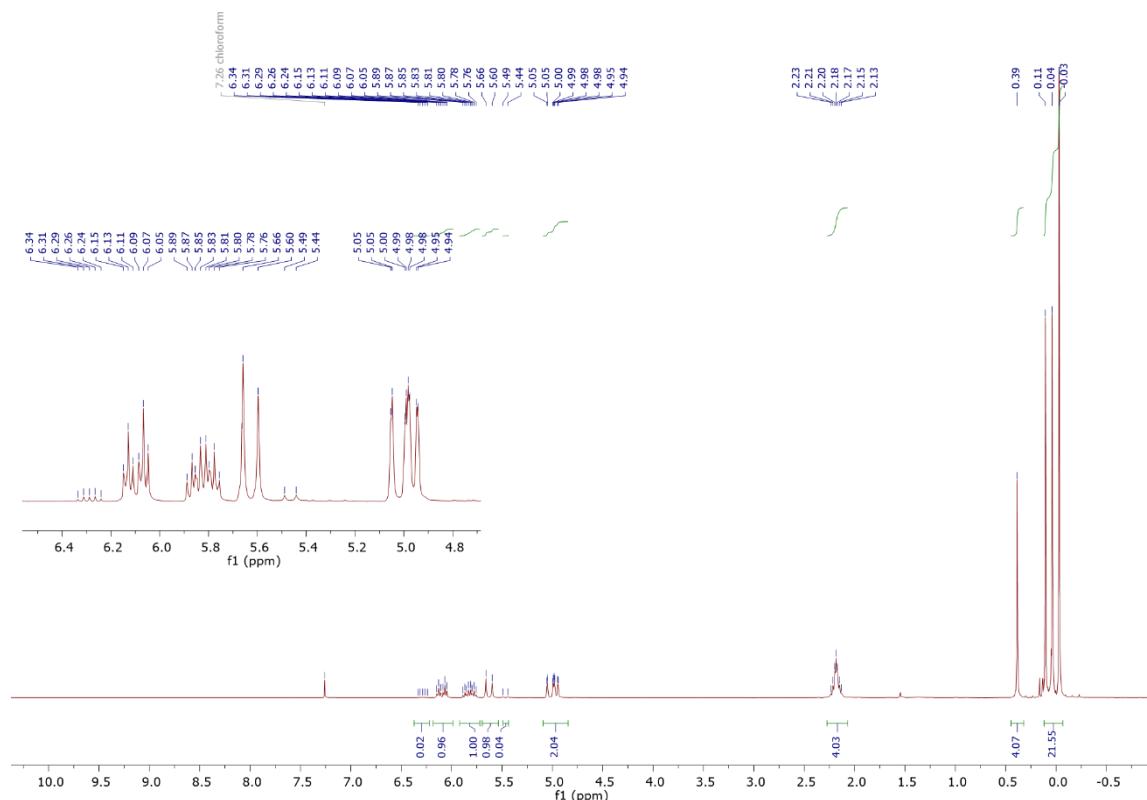


¹H NMR (300 MHz, CDCl₃) δ 6.10 RCH=CHSi (dt, *J* = 18.7, 5.8 Hz, 1H), 5.82 CH₂=CHR (ddt, *J* = 16.6, 10.3, 6.3 Hz, 1H), 5.63 RCH=CHSi (d, *J* = 18.7 Hz, 1H), CH₂=CHR 5.09 – 4.84 (m, 2H), 2.18 (m, 4H), 0.39 SiCH₂ (s, 4H), 0.12 – (-0.07) SiCH₃ (m, 21H), Isomer β-Z: 6.30 RCH=CHSi (dt, *J* = 14.3, 7.2 Hz), 5.46 RCH=CHSi (d, *J* = 14.2 Hz).

¹³C NMR (75 MHz, CDCl₃) δ 147.04, 138.36, 130.18, 114.80, 35.87, 32.94, 10.45, 8.19, 0.96, -0.18, -2.06.

²⁹Si NMR (79 MHz, CDCl₃) δ 8.94, 3.07, -4.41.

MS (EI, m/z): 299.1 [M-15]⁺ (4.7), 233.1 (5.5), 232.0 (4.9), 213.0 (18.8), 144.9 (9.1), 139.0 (8.7), 134.1 (13.8), 132.9 (100), 130.9 (5.1), 72.9 (27.0), 58.9 (12.0)



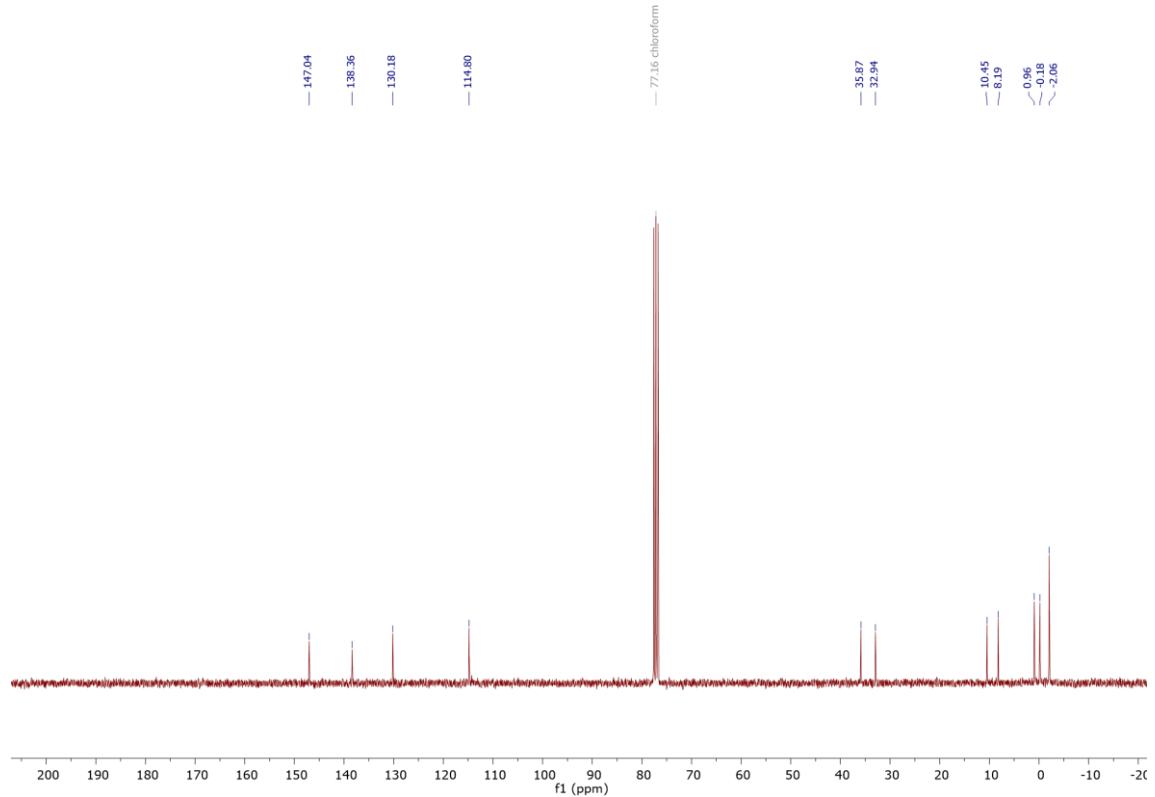


Figure S26. ^{13}C NMR spectrum of 3h

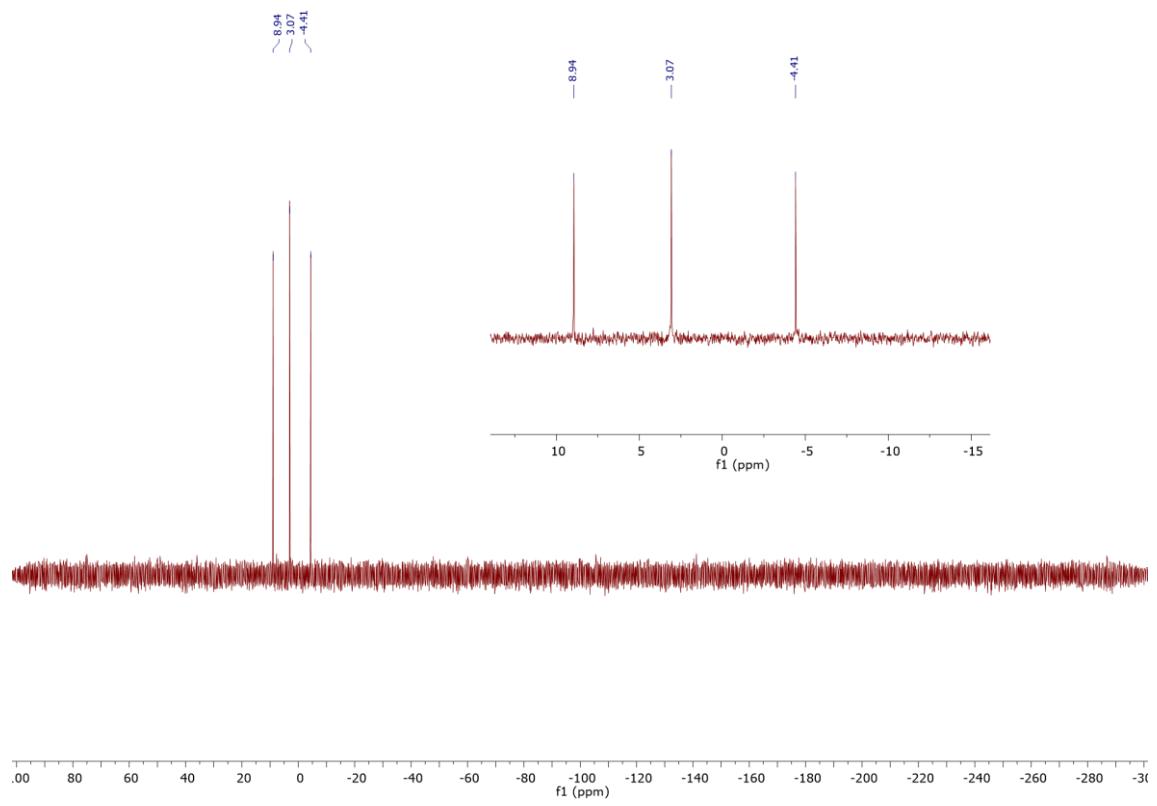
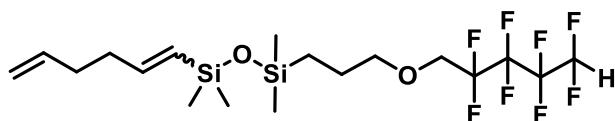


Figure S27. ^{29}Si NMR spectrum of 3h

2.10. Product 3i



¹H NMR (300 MHz, CDCl₃) δ 6.34 – 5.71 CF₂H, RCH=CHSi, CH₂=CHR (m, 3H), 5.62 RCH=CHSi (d, *J* = 18.7 Hz, 1H), 5.46 RCH=CHSi (β -Z isomer) (d, *J* = 14.2 Hz), 5.08 – 4.90 CH₂=CHR (m, 2H), 3.91 CH₂O (t, *J* = 14.0 Hz, 2H), 3.55 CH₂O (t, *J* = 6.9 Hz, 2H), 2.18 (m, 4H), 1.68 – 1.58 (m, 2H), 0.56 – 0.45 SiCH₂ (m, 2H), 0.10 SiMe₂ (s, 6H), 0.06 SiMe₂ (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 147.27, 138.32, 129.96, 114.80, 75.98, 67.64, 35.83, 32.90, 23.46, 14.10, 0.85, 0.42.

²⁹Si NMR (79 MHz, CDCl₃) δ 8.00, -3.82.

MS (EI, m/z): 471.1 (3.7) [M-15]⁺, 331. (3.5), 213.0 (7.4), 160.9 (6.6), 156.9 (6.5), 154.8 (6.6), 153.0 (9.1), 152.1 (13.5), 150.9 (100), 138.1 (11.4), 136.9 (91.4), 135.1 (9.9), 134.2 (13.5), 132.9 (97.9), 78.9 (7.0), 73.0 (11.9), 58.9 (5.6).

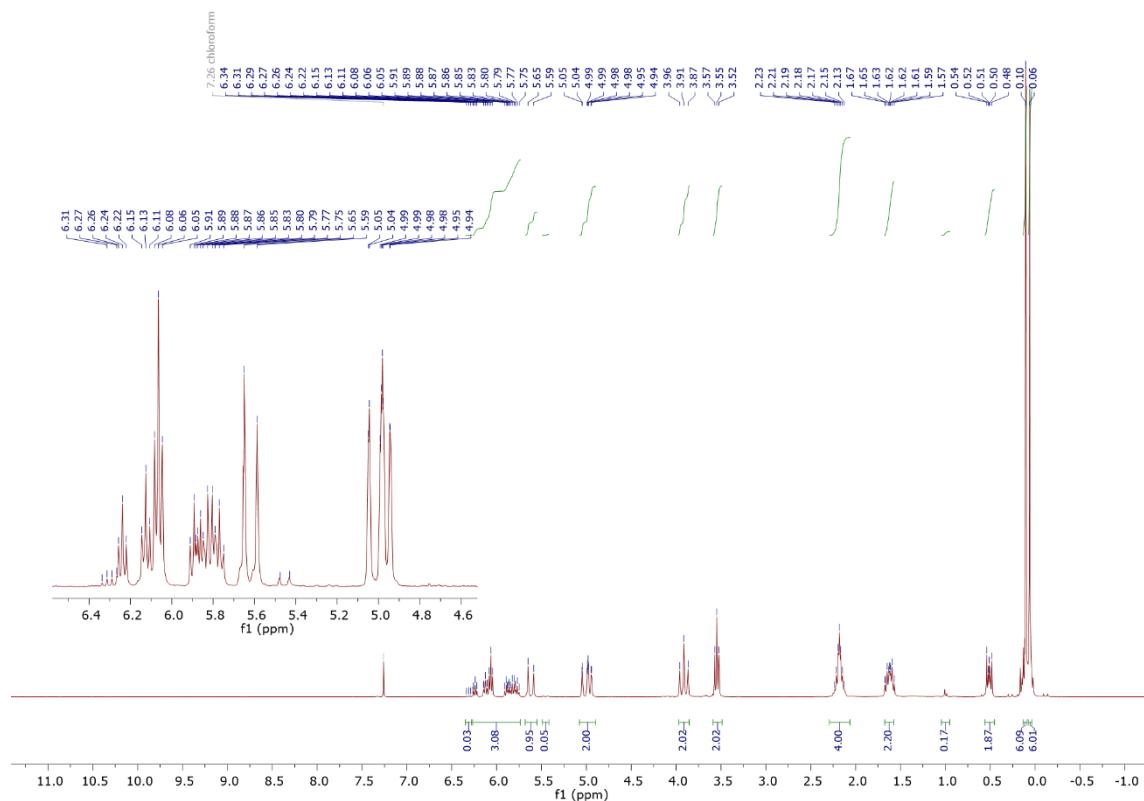


Figure S28. ^1H NMR spectrum of 3i

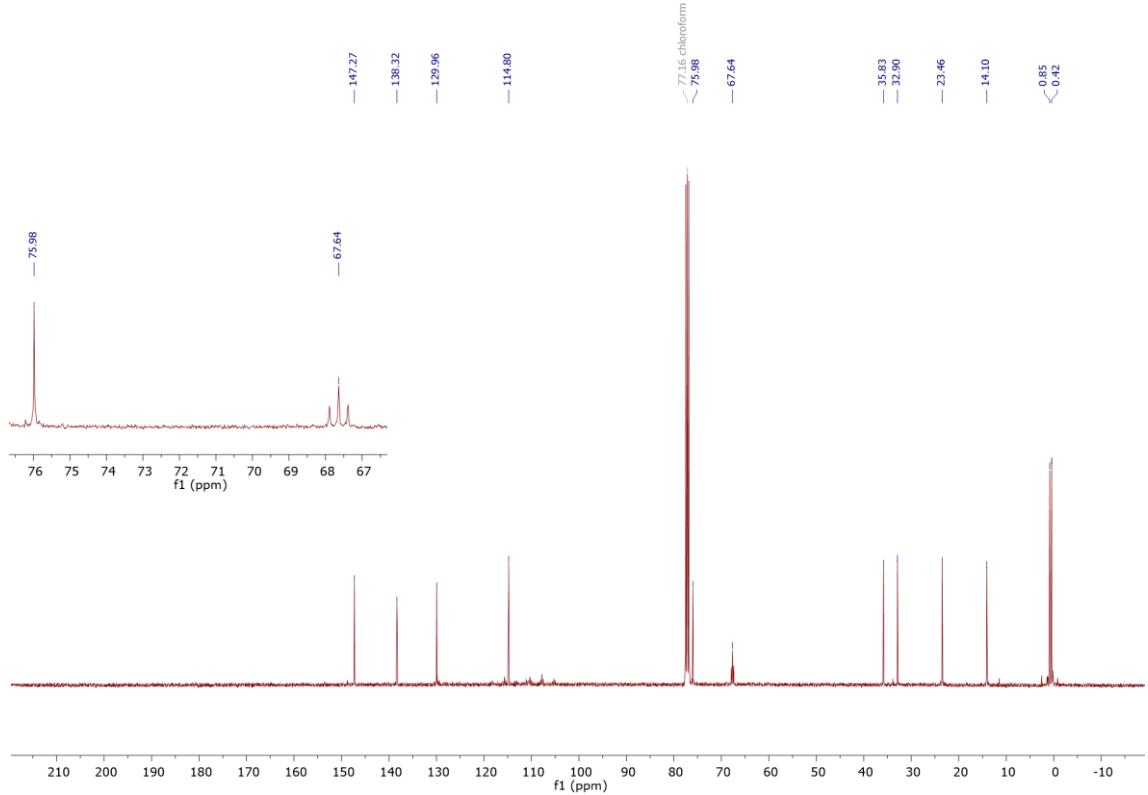


Figure S29. ¹³C NMR spectrum of 3i

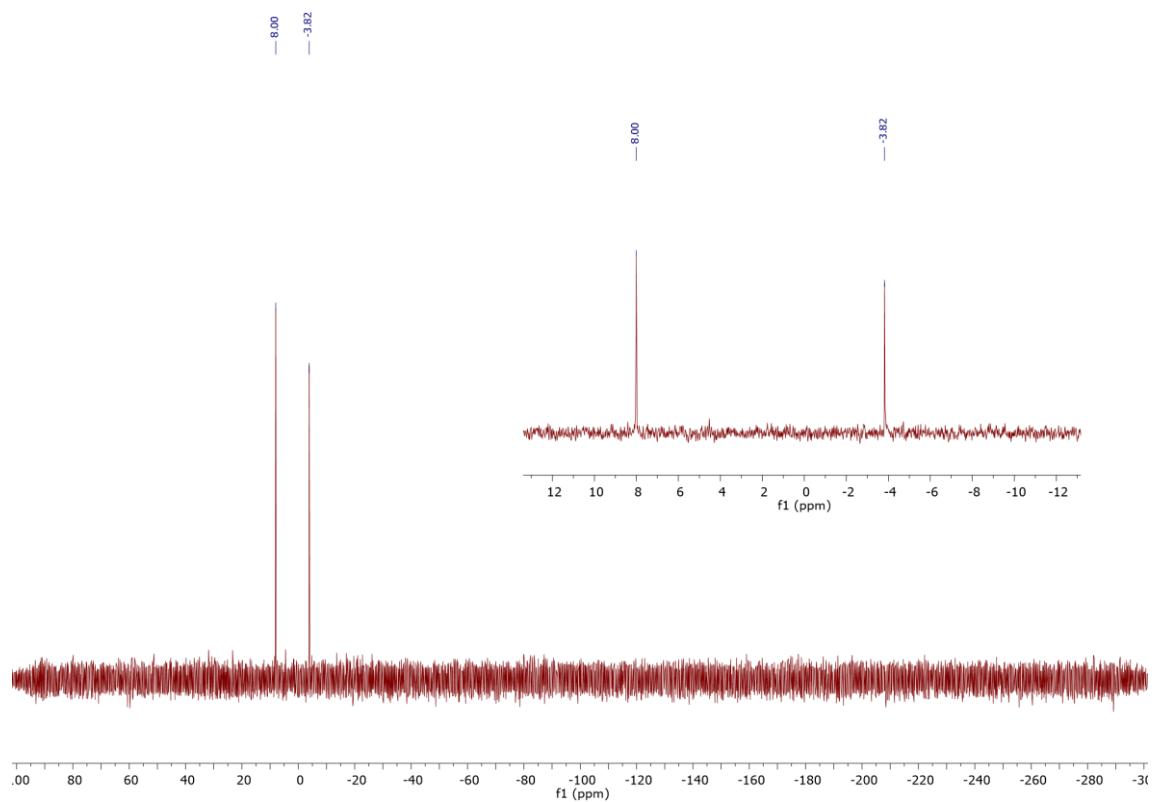
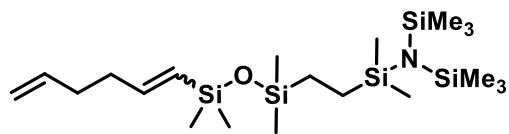


Figure S30. ^{29}Si NMR spectrum of 3i

2.11. Product 3j



¹H NMR (300 MHz, CDCl₃) δ 6.10 RCH=CHSi (dt, *J* = 18.6, 5.8 Hz, 1H), 5.82 CH₂=CHR (ddt, *J* = 16.5, 10.2, 6.3 Hz, 1H), 5.63 RCH=CHSi (d, *J* = 18.7 Hz, 1H), 5.11 – 4.86 (m, 2H), 2.18 (m, 4H), 0.55 – 0.47 SiCH₂ (m, 2H), 0.43 – 0.35 SiCH₂ (m, 2H), 0.17 SiMe₃, SiMe₂ (24H), 0.11 SiMe₂ (s, 6H), 0.05 SiMe₂ (s, 6H), Isomer β-Z: 6.29 RCH=CHSi (dt, *J* = 14.4, 7.3 Hz), 5.47 RCH=CHSi (d, *J* = 14.2 Hz).

¹³C NMR (75 MHz, CDCl₃) δ 147.04, 138.37, 130.16, 114.79, 35.87, 32.95, 12.11, 10.76, 5.74, 3.19, 0.99, -0.13.

²⁹Si NMR (79 MHz, CDCl₃) δ 8.89, 5.08, 2.23, -4.34.

MS (EI, m/z): 459.2 (0.2) M⁺, 220.1 (12.0), 219.2 (22.7), 218.0 (100), 216.0 (11.2), 202.0 (6.7), 132.9 (18.4), 129.9 (7.7), 72.9 (6.5).

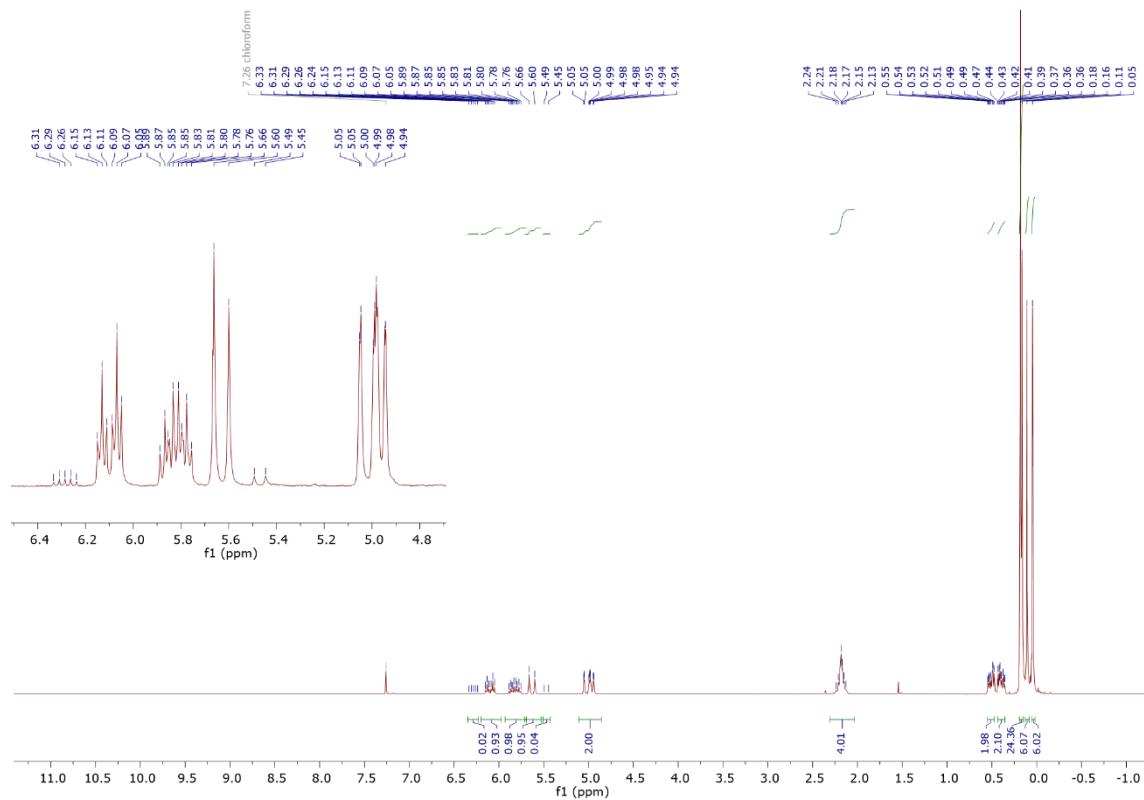
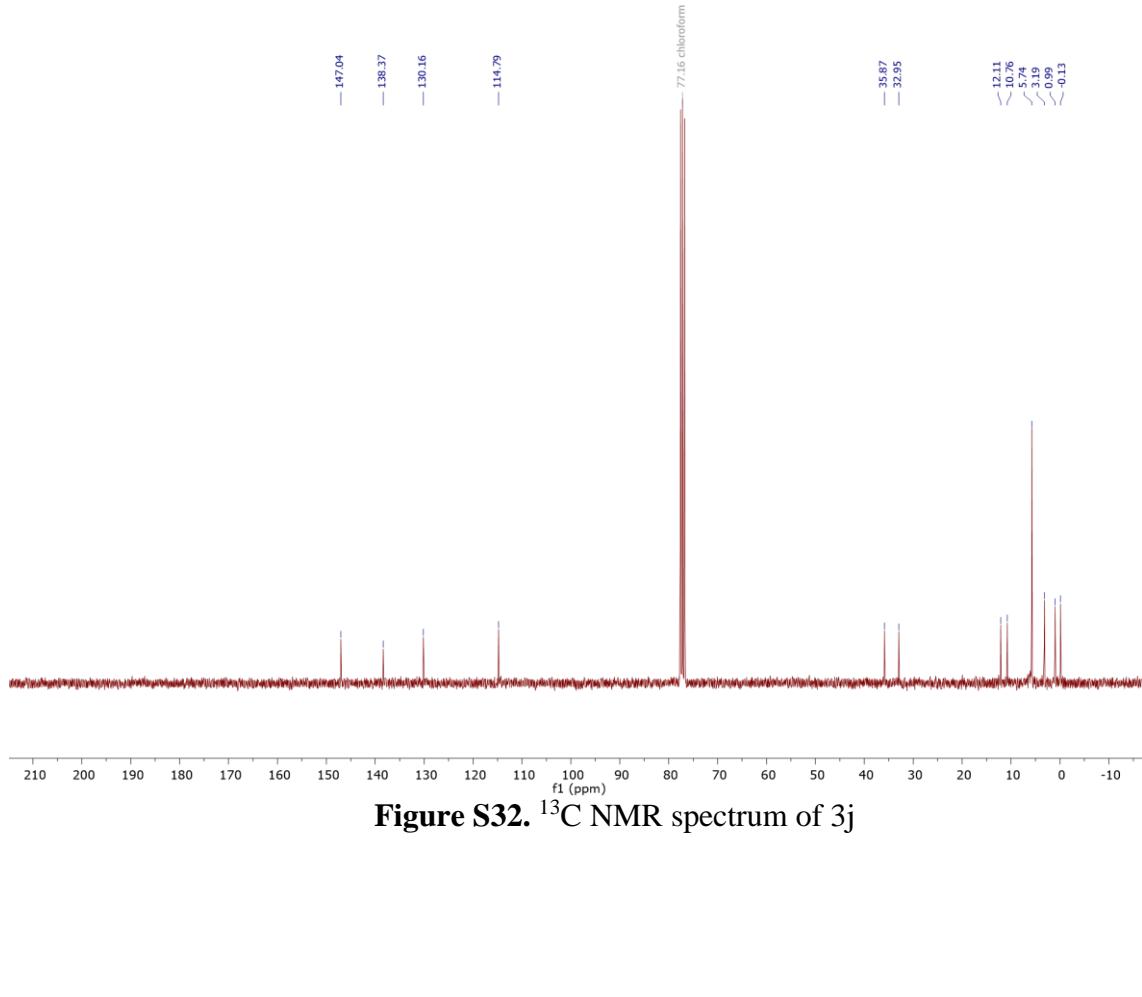


Figure S31. ¹H NMR spectrum of 3j



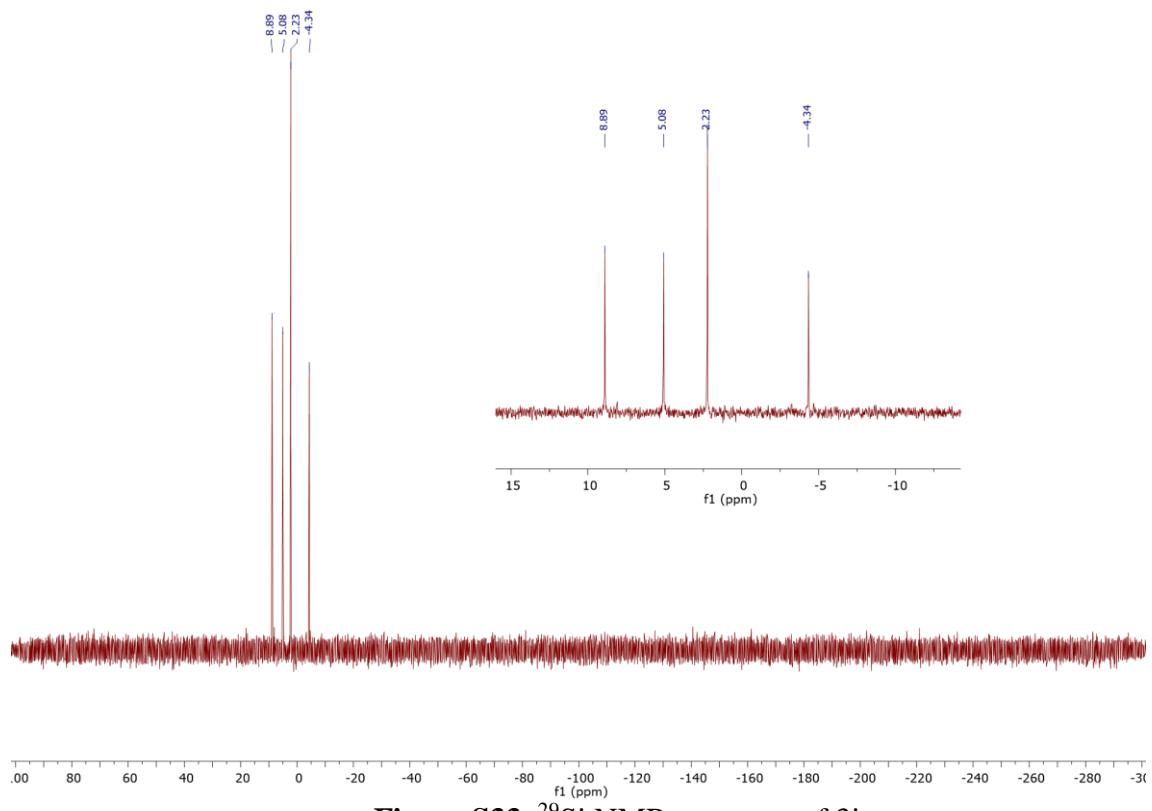
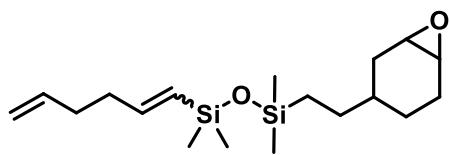


Figure S33. ^{29}Si NMR spectrum of 3j

2.12. Product 3k



¹H NMR (300 MHz, CDCl₃) δ 6.08 RCH=CHSi (dt, *J* = 18.6, 5.8 Hz, 1H), 5.82 CH₂=CHR (ddt, *J* = 16.6, 10.3, 6.3 Hz, 1H), RCH=CHSi 5.61 (d, *J* = 18.7 Hz, 1H), 5.10 – 4.87 CH₂=CHR (m, 2H), 3.14 CH(O) (m, 2H), 2.26 – 1.94 (m, 6H), 1.87 – 1.66 (m, 1H), 1.56 – 1.26 (m, 3H), 1.22 – 1.04 (m, 3H), 0.93 – 0.78 (m, 1H), 0.46 SiCH₂ (m, 2H), 0.09 SiMe₂ (s, 6H), 0.03 SiMe₂ (s, 6H), Isomer β-Z: 6.28 RCH=CHSi (dt, *J* = 14.5, 7.3 Hz), 5.45 RCH=CHSi (d, *J* = 14.0 Hz).

¹³C NMR (75 MHz, CDCl₃) δ 147.10, 138.31, 130.07, 114.82, 53.44, 52.92, 52.17, 52.13, 35.84, 35.53, 32.91, 32.42, 31.70, 30.56, 30.31, 29.75, 26.90, 25.54, 24.21, 23.74, 15.39, 15.26, 0.93, 0.40.

²⁹Si NMR (79 MHz, CDCl₃) δ 8.30, -4.23.

MS (EI, m/z): 150.1 (7.1), 148.9 (52.4), 135.2 (7.6), 134.5 (6.6), 132.9 (100), 118.9 (9.7), 116.9 (5.2), 108.9 (7.3), 92.9 (8.3), 81.0 (14.7), 80.0 (6.6), 79.0 (11.9), 72.9 (9.0), 67.0 (24.6).

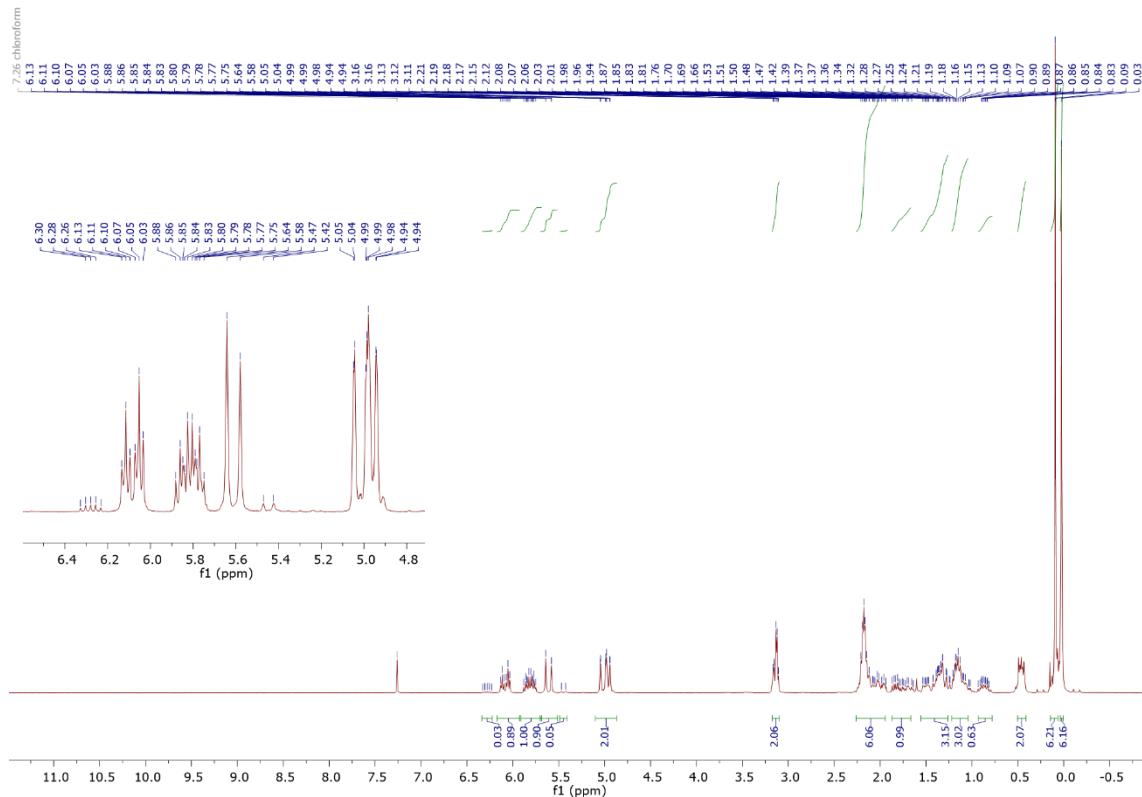


Figure S34. ¹H NMR spectrum of 3k

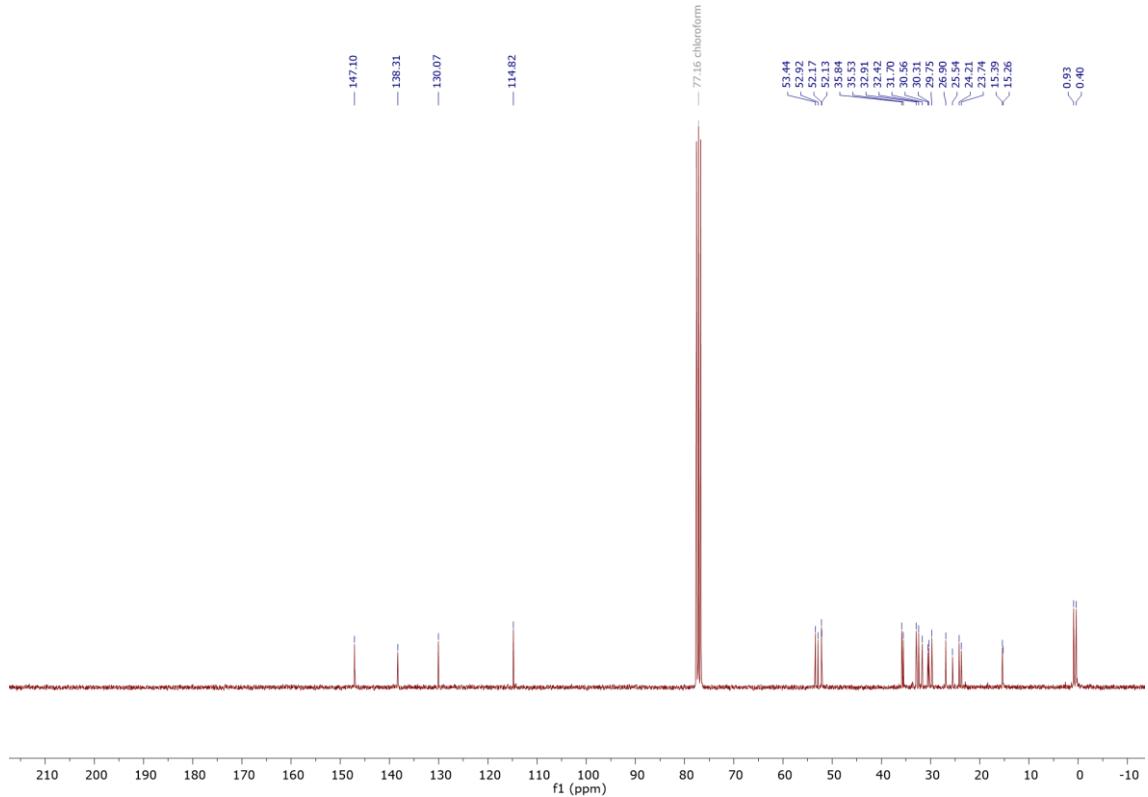


Figure S35. ^1H NMR spectrum of 3k

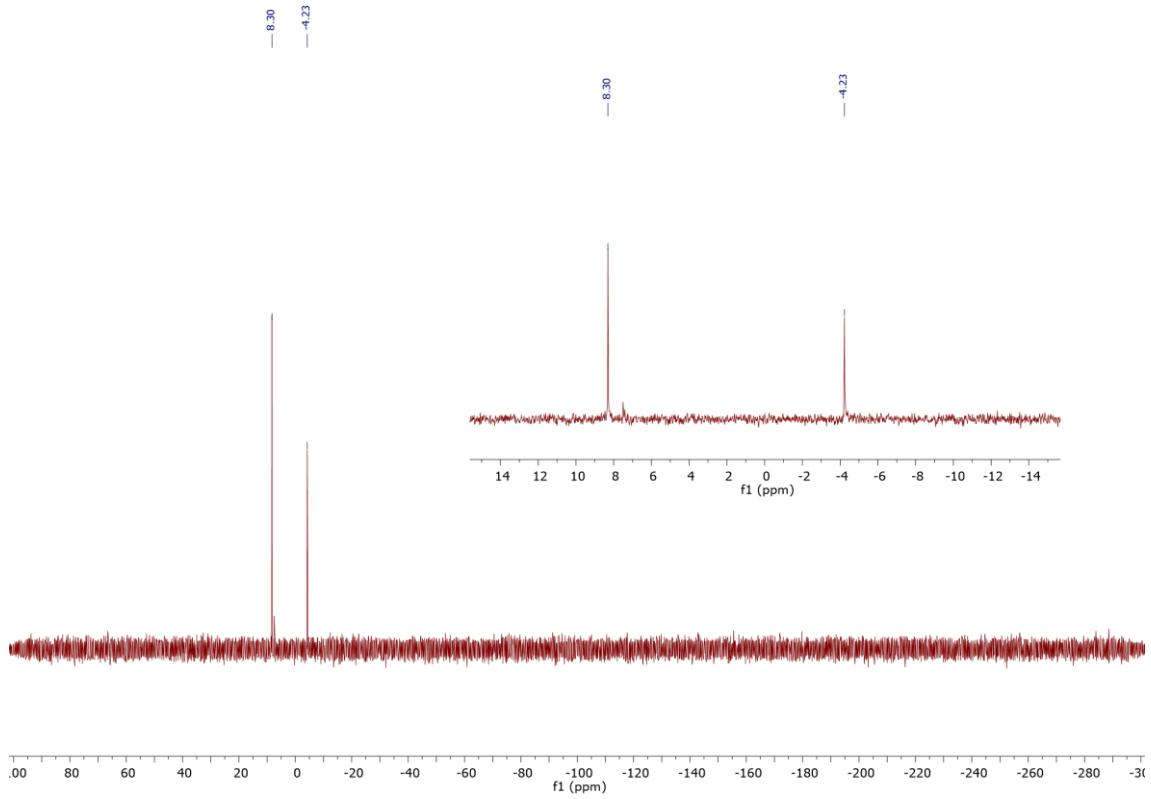
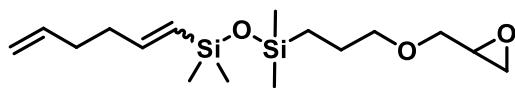


Figure S36. ^{29}Si NMR spectrum of 3k

2.13. Product 3l



¹H NMR (300 MHz, CDCl₃) δ 6.09 RCH=CHSi (dt, *J* = 18.7, 5.9 Hz, 1H), 5.81 CH₂=CHR (ddt, *J* = 16.6, 10.3, 6.3 Hz, 1H), 5.61 RCH=CHSi (d, *J* = 18.7 Hz, 1H), 5.08 – 4.85 CH₂=CHR (m, 2H), 3.70 (dd, *J* = 11.5, 3.1 Hz, 1H), 3.52 – 3.30 (m, 3H), 3.15 (m, 1H), 2.82 – 2.76 (m, 1H), 2.61 (dd, *J* = 5.0, 2.7 Hz, 1H), 2.26 – 2.09 (m, 4H), 1.66 – 1.55 (m, 2H), 0.56 – 0.45 SiCH₂ (m, 2H), 0.10 SiMe₂ (s, 6H), 0.05 SiMe₂ (s, 6H), Isomer β-Z: 6.28 RCH=CHSi (dt, *J* = 14.4, 7.2 Hz), 5.45 RCH=CHSi (d, *J* = 14.1 Hz).

¹³C NMR (101 MHz, CDCl₃) δ 147.16, 138.32, 130.03, 114.80, 74.52, 71.57, 51.02, 44.52, 35.83, 32.90, 23.62, 14.37, 0.90, 0.45.

²⁹Si NMR (79 MHz, CDCl₃) δ 8.16, -4.01.

MS (EI, m/z): 313.1 [M-15]⁺, 174.9 (29.7), 148.9 (8.3), 135.0 (14.4), 134.1 (13.8), 132.9 (100), 130.9 (5.5), 118.9 (7.4), 92.9 (5.3), 78.9 (7.0), 67.0 (14.5).

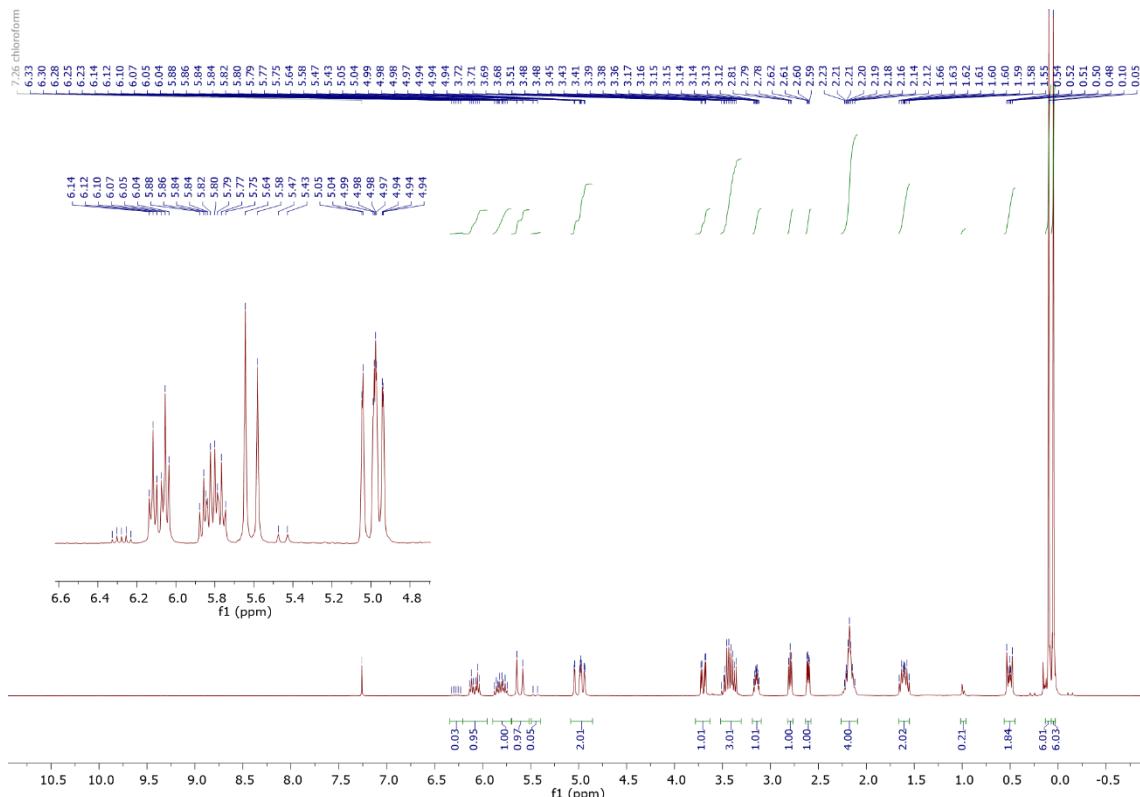


Figure S37. ^1H NMR spectrum of 31

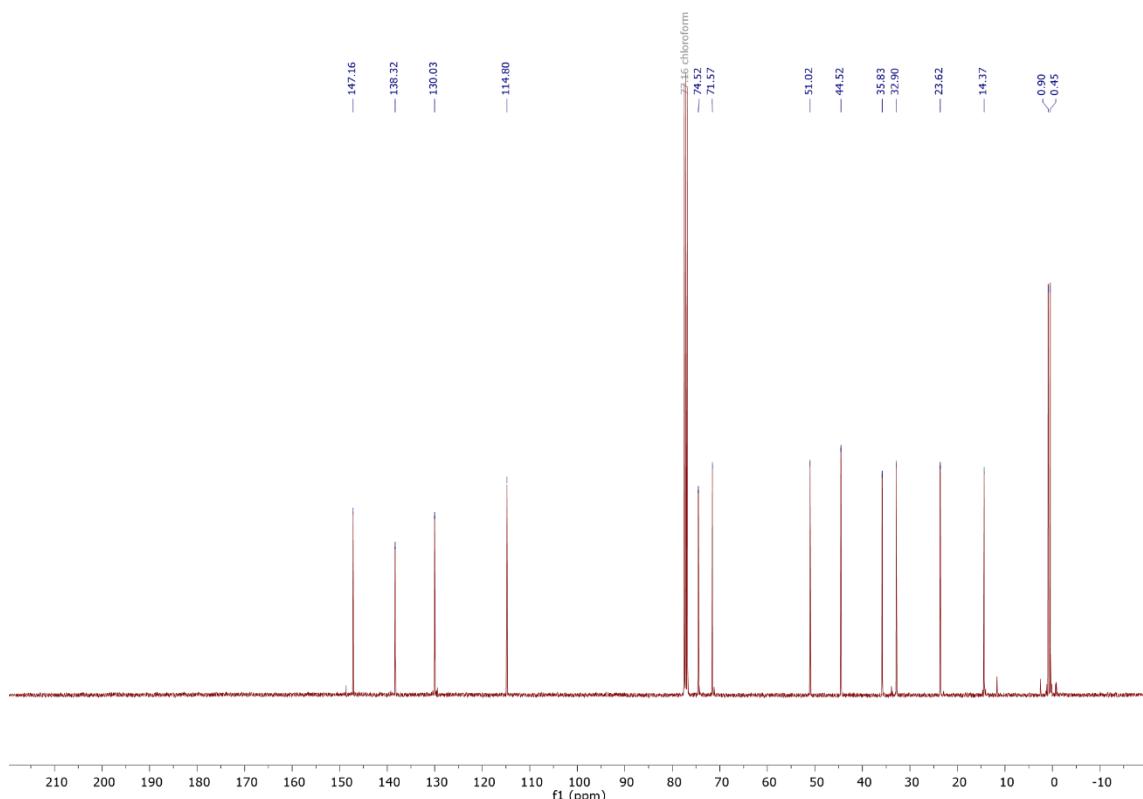


Figure S38. ^{13}C NMR spectrum of 31

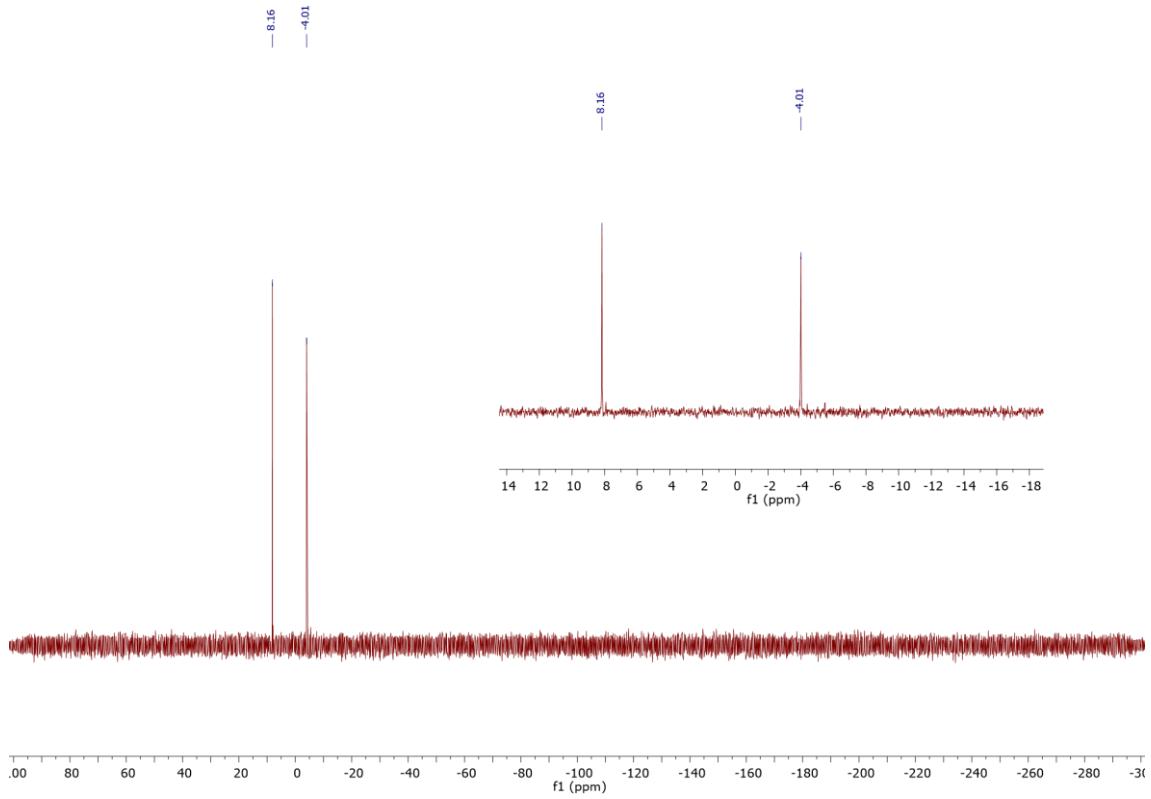
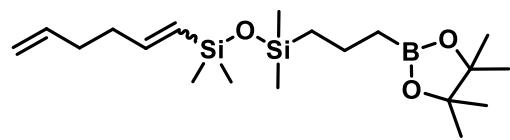


Figure S39. ^{29}Si NMR spectrum of 31

2.14. Product 3m



¹H NMR (300 MHz, CDCl₃) δ 6.08 RCH=CHSi (dt, *J* = 18.7, 5.9 Hz, 1H), 5.81 CH₂=CHR (ddt, *J* = 16.6, 10.2, 6.3 Hz, 1H), 5.62 RCH=CHSi (d, *J* = 18.7 Hz, 1H), 5.07 – 4.88 CH₂=CHR (m, 2H), 2.34 – 2.00 (m, 4H), 1.50 – 1.38 (m, 2H), 1.24 C(CH₃)₂ (s, 12H), 0.82 BCH₂ (t, *J* = 7.6 Hz, 2H), 0.53 SiCH₂ (m, 2H), 0.09 SiMe₂ (s, 6H), 0.03 SiMe₂ (s, 6H), Isomer β-Z: 6.27 RCH=CHSi (dt, *J* = 14.3, 7.2 Hz), 5.45 RCH=CHSi (d, *J* = 14.3 Hz).
¹³C NMR (75 MHz, CDCl₃) δ 147.01, 138.40, 130.18, 114.78, 82.94, 35.85, 32.93, 24.98, 21.84, 18.08, 0.95, 0.58.

¹³C NMR (75 MHz, CDCl₃) δ 147.01, 138.40, 130.18, 114.78, 82.94, 35.85, 32.93, 24.98, 21.84, 18.08, 0.95, 0.58.

SI NMR (¹H NMR, CDCl₃) δ 7.80, -4.42.
MS (EI, m/z): 367.1 [M-15]⁺, 267.0 (8.5), 227.1 (5.8), 225.0 (5.9), 212.9 (5.3), 203.1 (7.1), 202.2 (8.3), 201.0 (43.9), 199.9 (12.2), 187.0 (11.8), 175.1 (16.1), 174.4 (8.2), 172.9 (15.4), 151.0 (6.8), 158.9 (24.8), 156.9 (7.9), 144.9 (20.5), 135.1 (7.9), 134.1 (12.7), 132.9 (100), 130.9 (9.6), 116.9 (8.8), 84.0 (36.2), 83.0 (35.6), 73.0 (9.7), 69.0 (5.6)

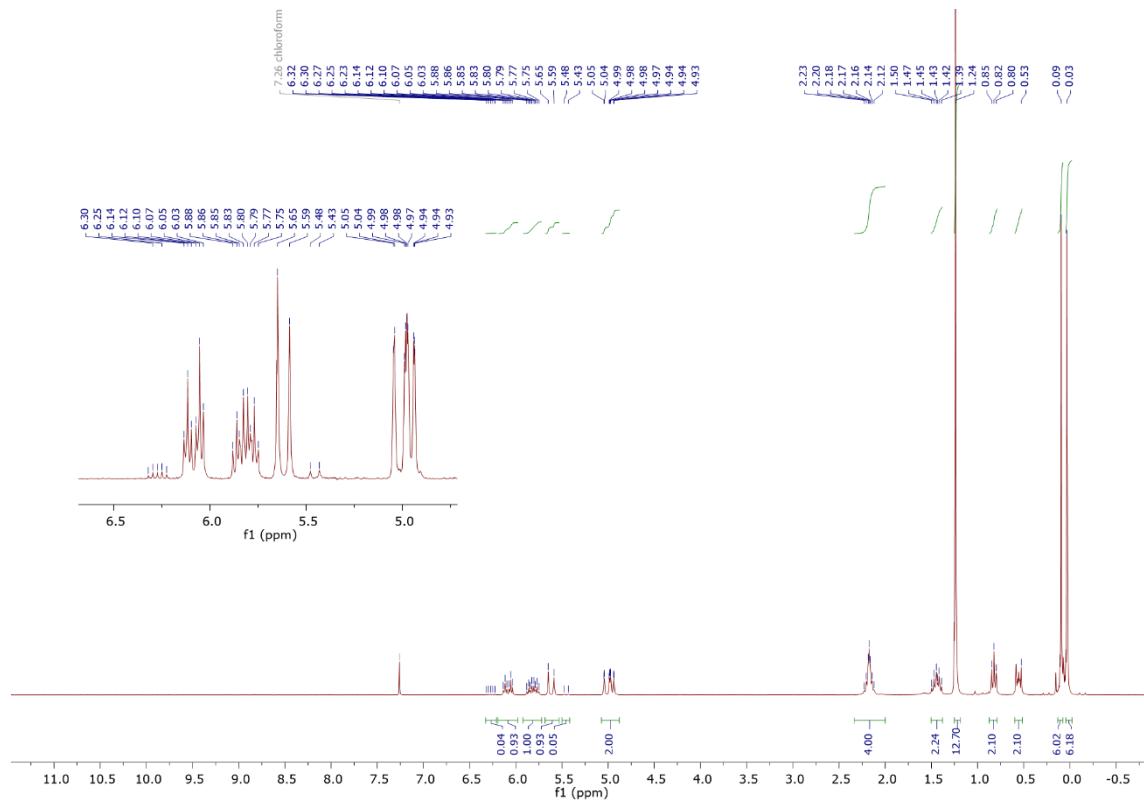


Figure S40. ^1H NMR spectrum of 3m

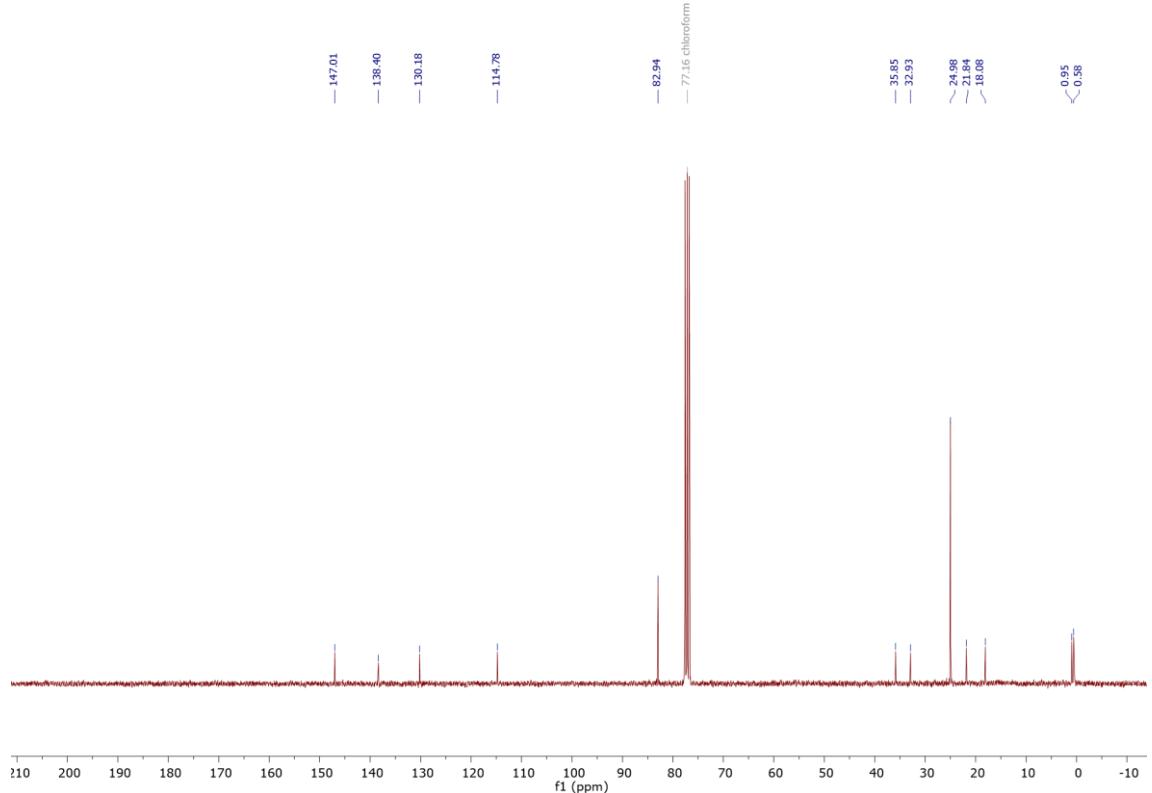


Figure S41. ^{13}C NMR spectrum of 3m

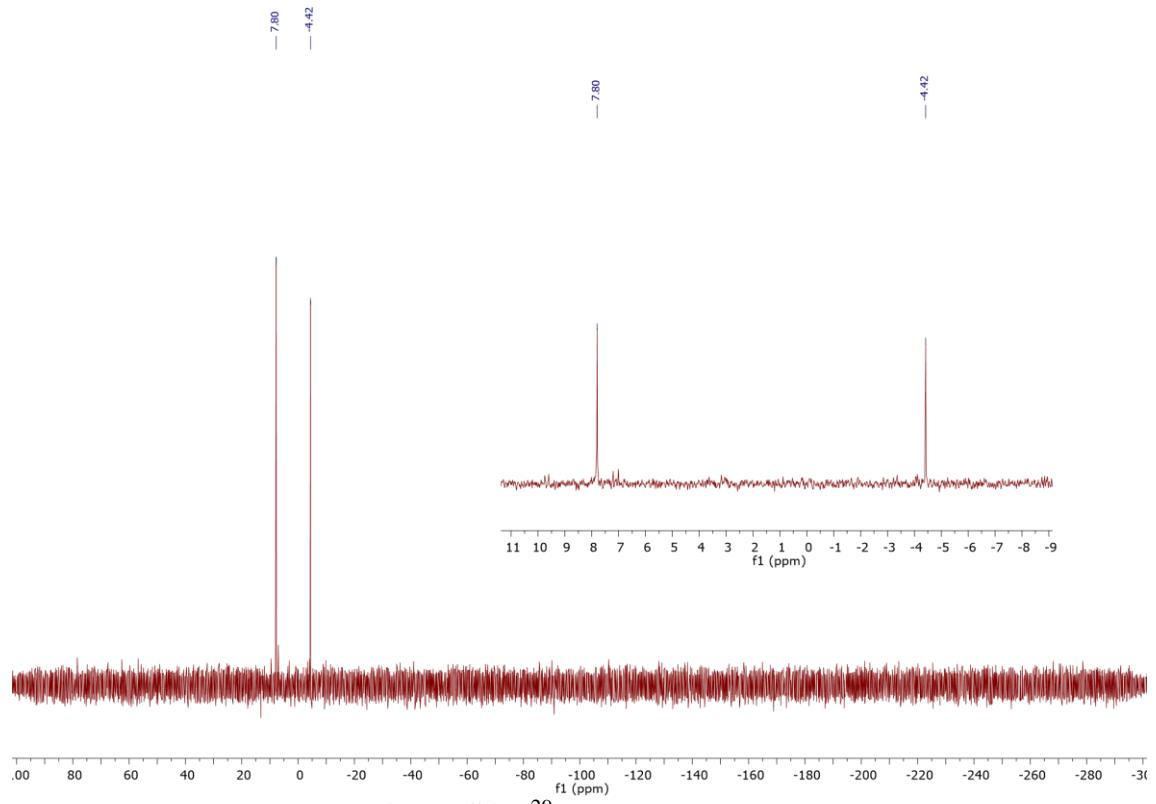
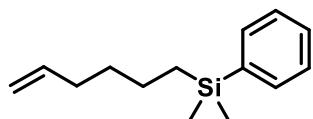


Figure S42. ^{29}Si NMR spectrum of 3m

2.15. Product 4a



¹H NMR (300 MHz, CDCl₃) δ 7.56 – 7.50 (m, 2H), 7.42 – 7.34 (m, 3H), 5.81 CH₂=CHR (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 5.12 – 4.76 CH₂=CHR (m, 2H), 2.06 (q, *J* = 6.8 Hz, 2H), 1.48 – 1.28 (m, 4H), 0.84 – 0.72 SiCH₂ (m, 2H), 0.28 SiMe₂ (s, 6H).

¹³C NMR (75 MHz, CDCl₃) δ 139.78, 139.21, 133.69, 128.90, 127.84, 114.30, 33.60, 32.92, 23.51, 15.69, -2.88.

²⁹Si NMR (79 MHz, CDCl₃) δ -3.08.

MS (EI, m/z): 218.0 M⁺, 203.0 (1.4) [M-15]⁺, 139.9 (22.4), 136.1 (13.6), 134.9 (100), 124.9 (8.7), 120.9 (19.9), 111.9 (6.3), 106.9 (5.4), 104.8 (6.1).

Product contains 4% of disubstituted derivative

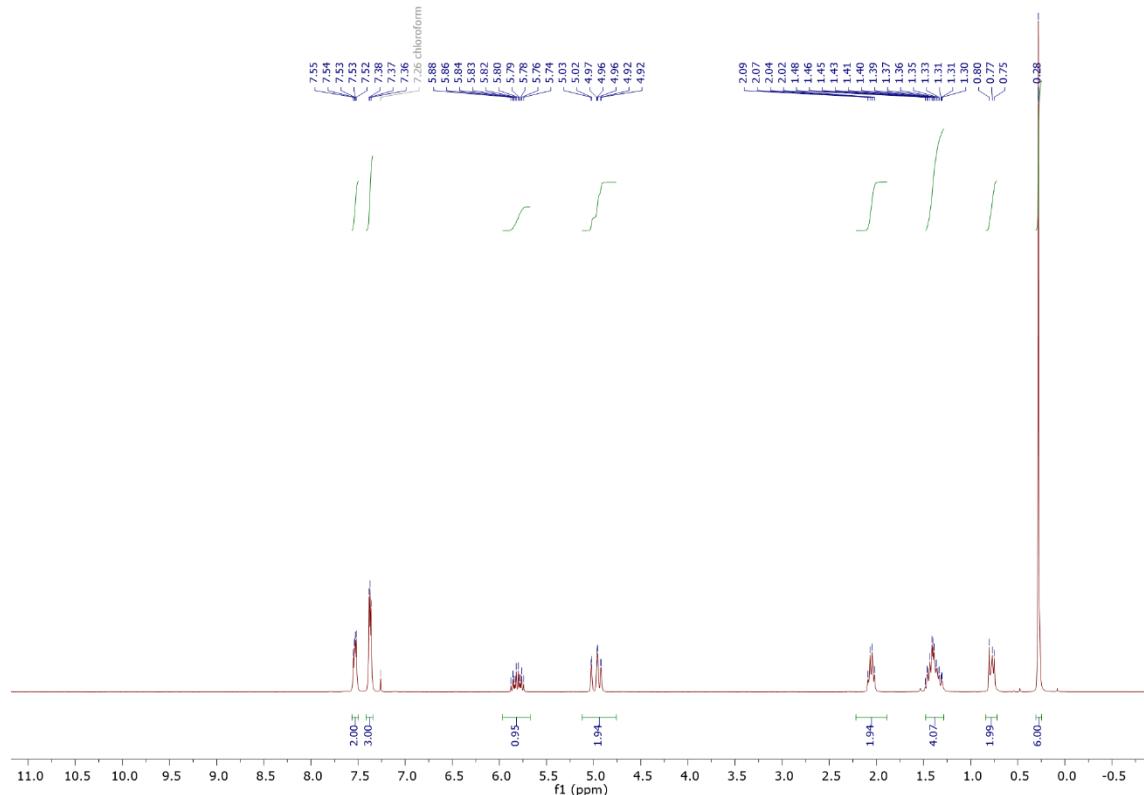


Figure S43. ¹H NMR spectrum of 4a

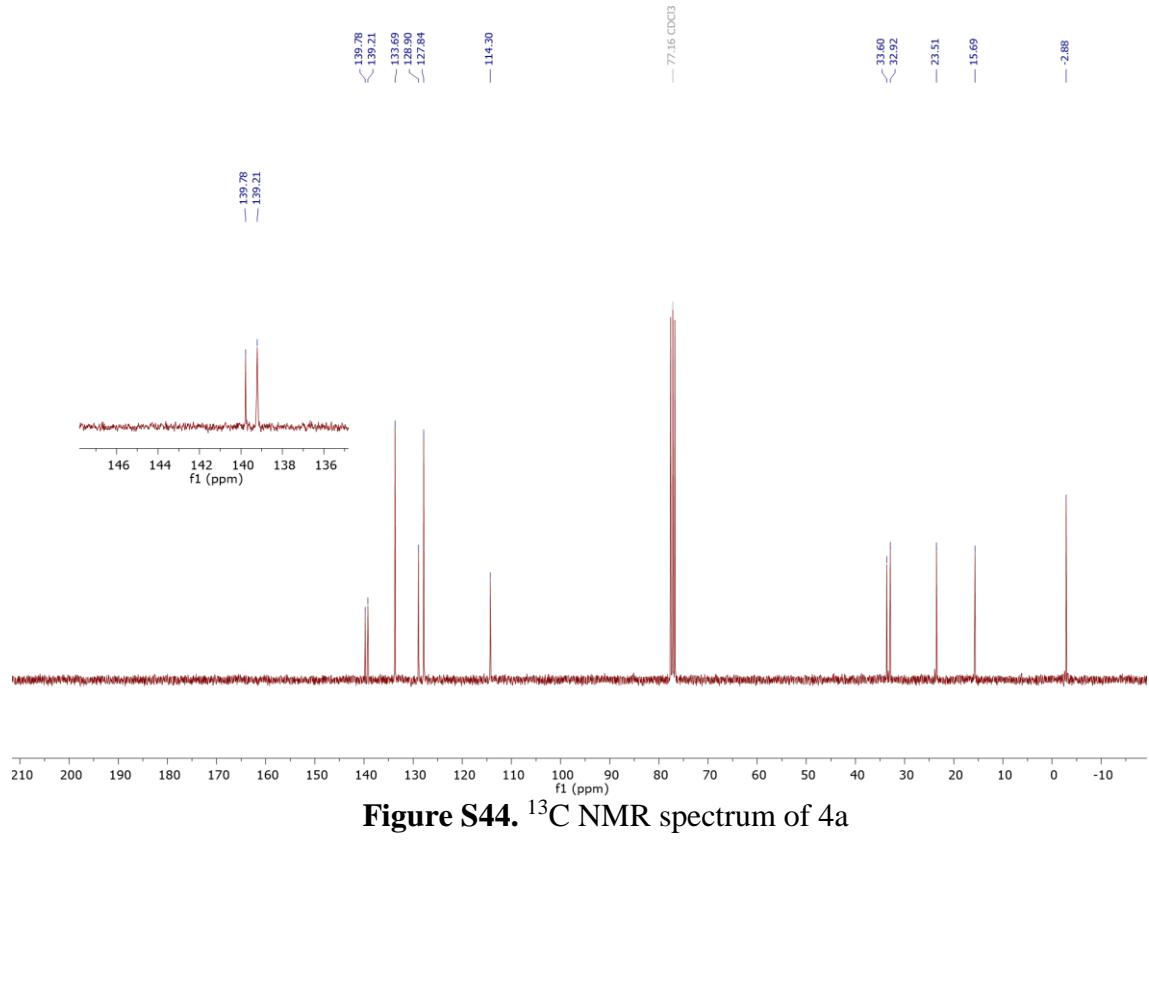


Figure S44. ^{13}C NMR spectrum of 4a

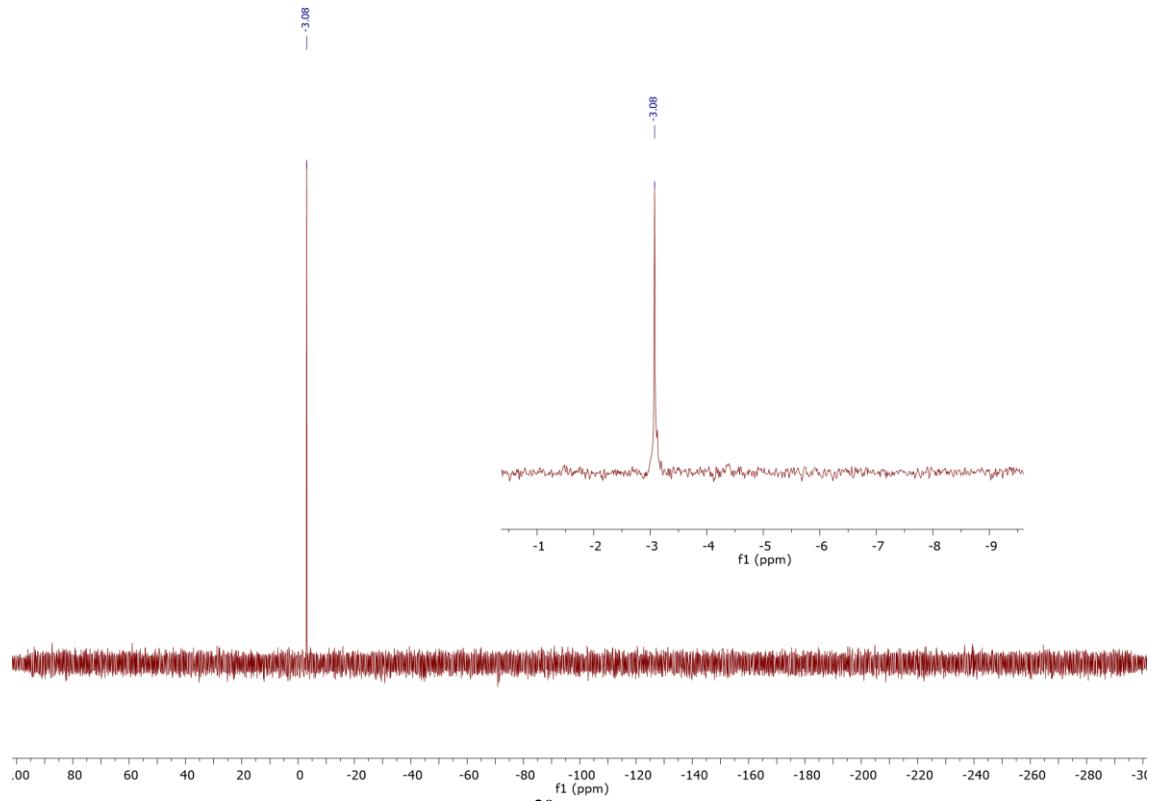
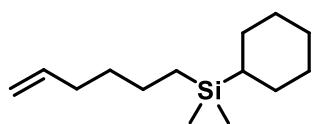


Figure S45. ^{29}Si NMR spectrum of 4a

2.16. Product 4b



¹H NMR (300 MHz, CDCl₃) δ 5.81 CH₂=CHR (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 5.10 – 4.71 CH₂=CHR (m, 2H), 2.05 (q, *J* = 6.9 Hz, 2H), 1.68 (m, 5H), 1.42 – 1.00 (m, 9H), 0.66 – 0.37 SiCH, SiCH₂ (m, 3H), -0.10 SiMe₂ (s, 6H).

¹³C NMR (75 MHz, CDCl₃) δ 139.34, 114.24, 33.68, 33.16, 28.32, 27.63, 27.20, 25.48, 23.60, 13.50, -5.19.

²⁹Si NMR (79 MHz, CDCl₃) δ 2.98.

MS (EI, m/z): 142.1 (9.2), 141.0 (71.9), 112.9 (32.3), 98.9 (32.8), 84.9 (11.8), 80.9 (24.7), 73.0 (87.5), 60.1 (7.1), 58.9 (100)

Product contains 4% of disubstituted derivative

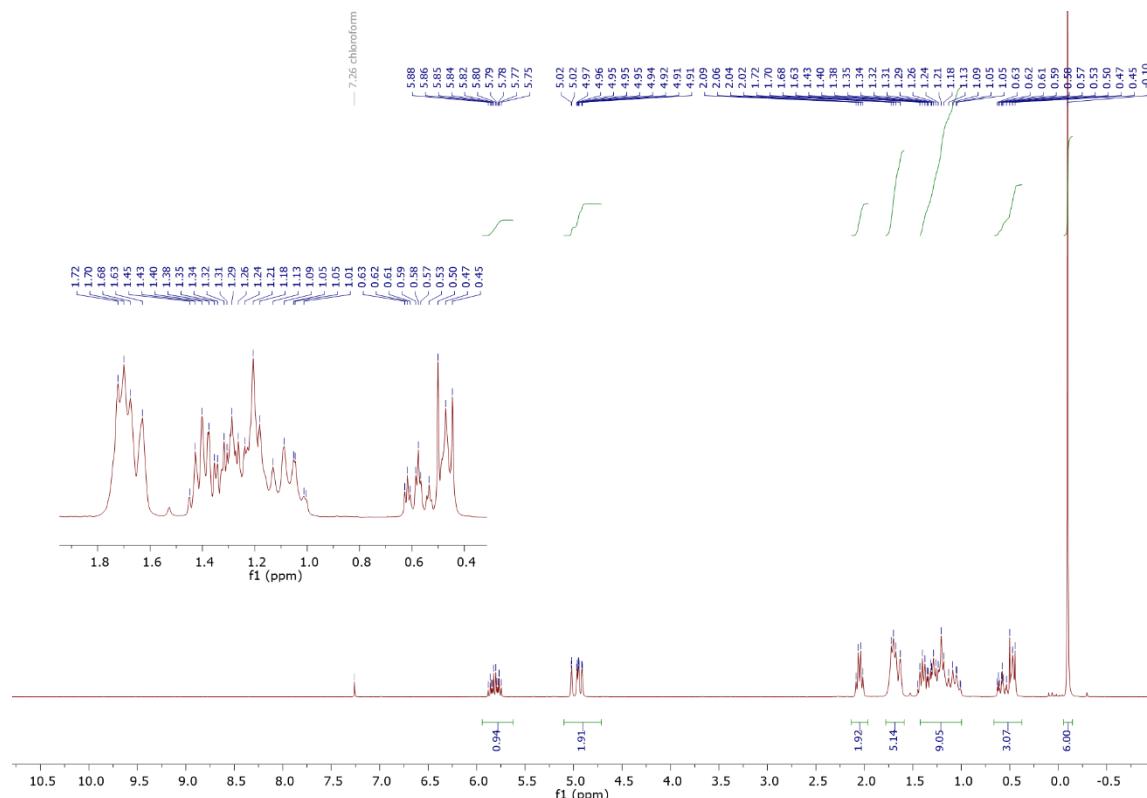


Figure S46. ¹H NMR spectrum of 4b

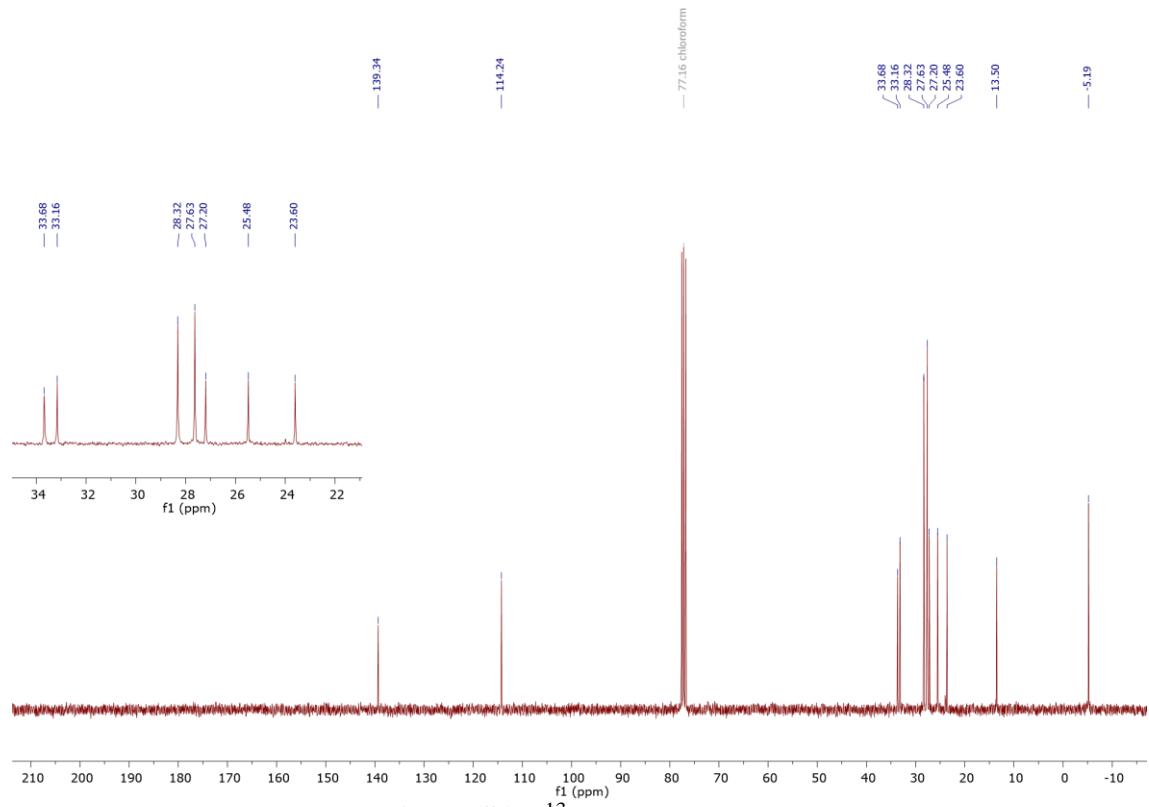


Figure S47. ^{13}C NMR spectrum of 4b

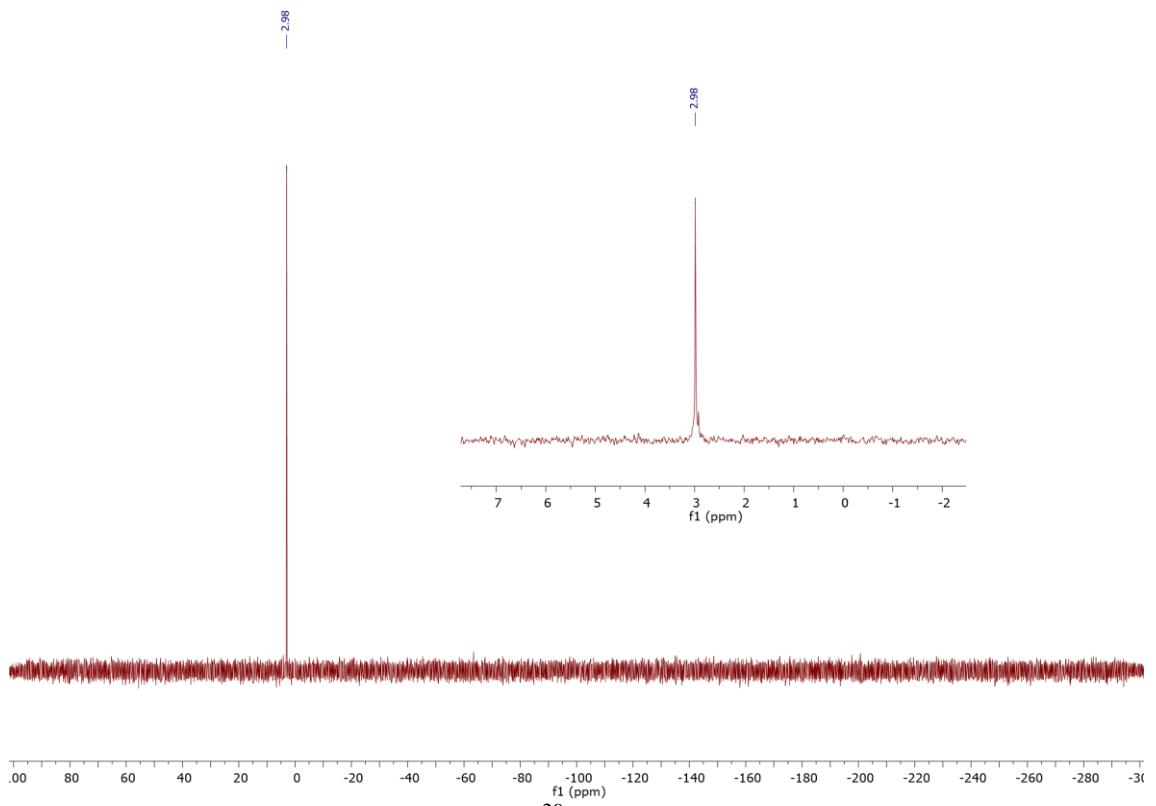
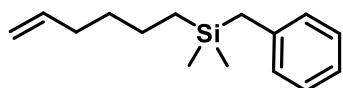


Figure S48. ^{29}Si NMR spectrum of 4b

2.17. Product 4c



$^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.22 (m, 2H), 7.05 (m, 3H), 5.82 $\text{CH}_2=\text{CHR}$ (ddt, $J = 16.9, 10.2, 6.7$ Hz, 1H), 5.09 – 4.88 $\text{CH}_2=\text{CHR}$ (m, 2H), 2.14 – 1.97 (m, 4H), 1.47 – 1.27 (m, 4H), 0.56 – 0.44 SiCH_2 (m, 2H), -0.02 SiMe_2 (s, 6H).

$^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 140.60, 139.21, 128.26, 128.20, 123.97, 114.34, 33.62, 32.92, 25.75, 23.38, 14.73, -3.46.

$^{29}\text{Si NMR}$ (79 MHz, CDCl_3) δ 2.29.

MS (EI, m/z): 216.9 [$\text{M}-15\text{I}^+$], 142.2 (8.9), 141.0 (70.3), 134.9 (6.5), 120.9 (16.2), 112.9 (32.1), 98.9 (25.2), 90.9 (5.8), 84.9 (15.3), 80.9 (14.6), 74.2 (6.0), 73.0 (80.4), 60.1 (7.0), 58.9 (100).

Product contains 3% of disubstituted derivative

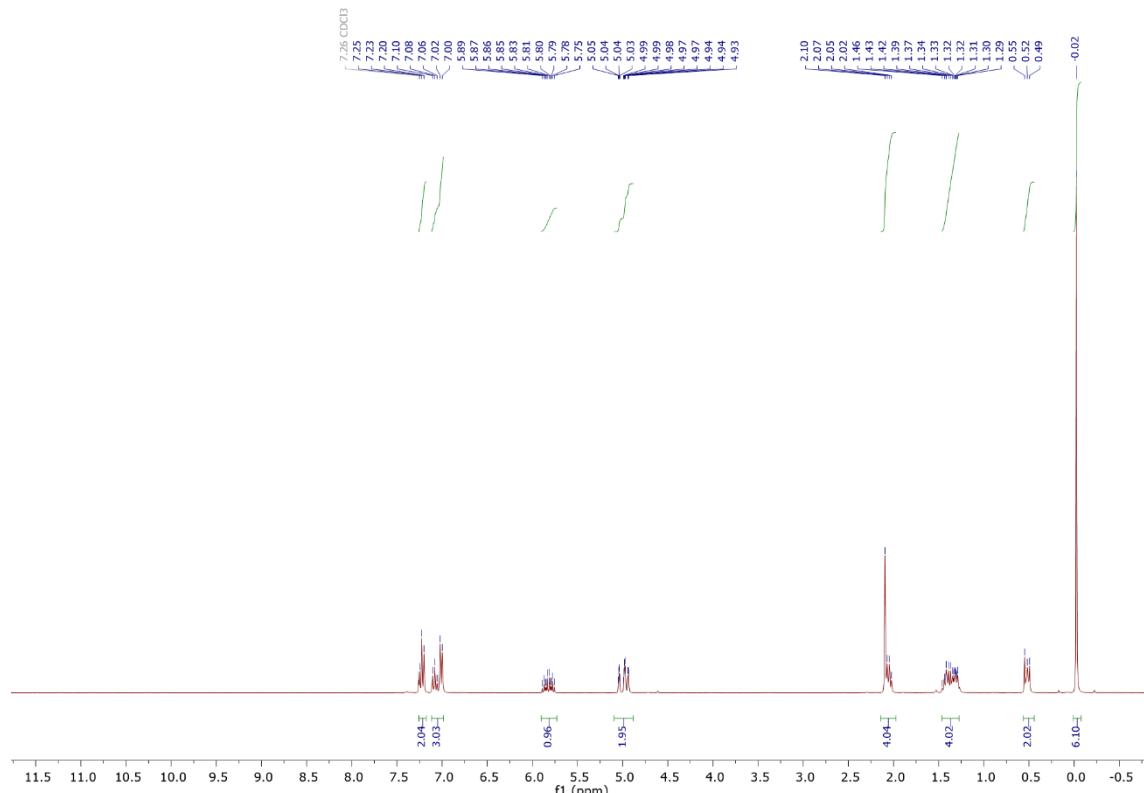


Figure S49. $^1\text{H NMR}$ spectrum of 4c

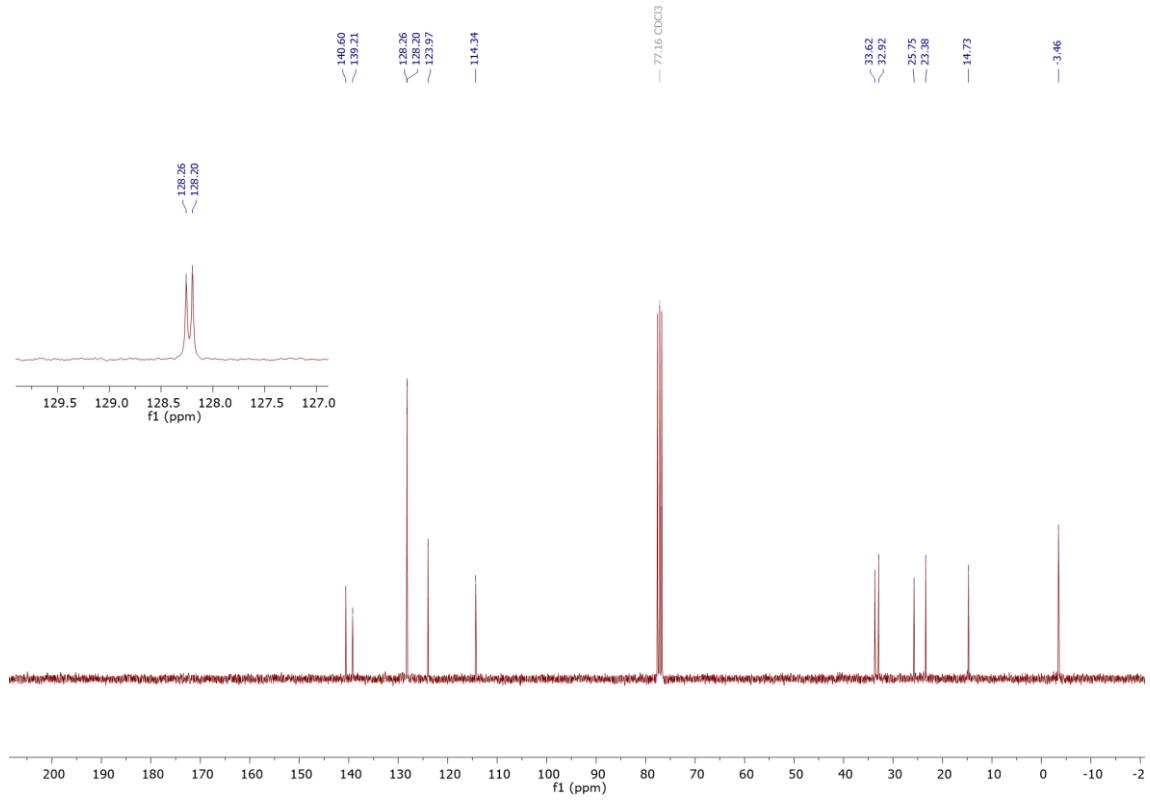


Figure S50. ^{13}C NMR spectrum of 4c

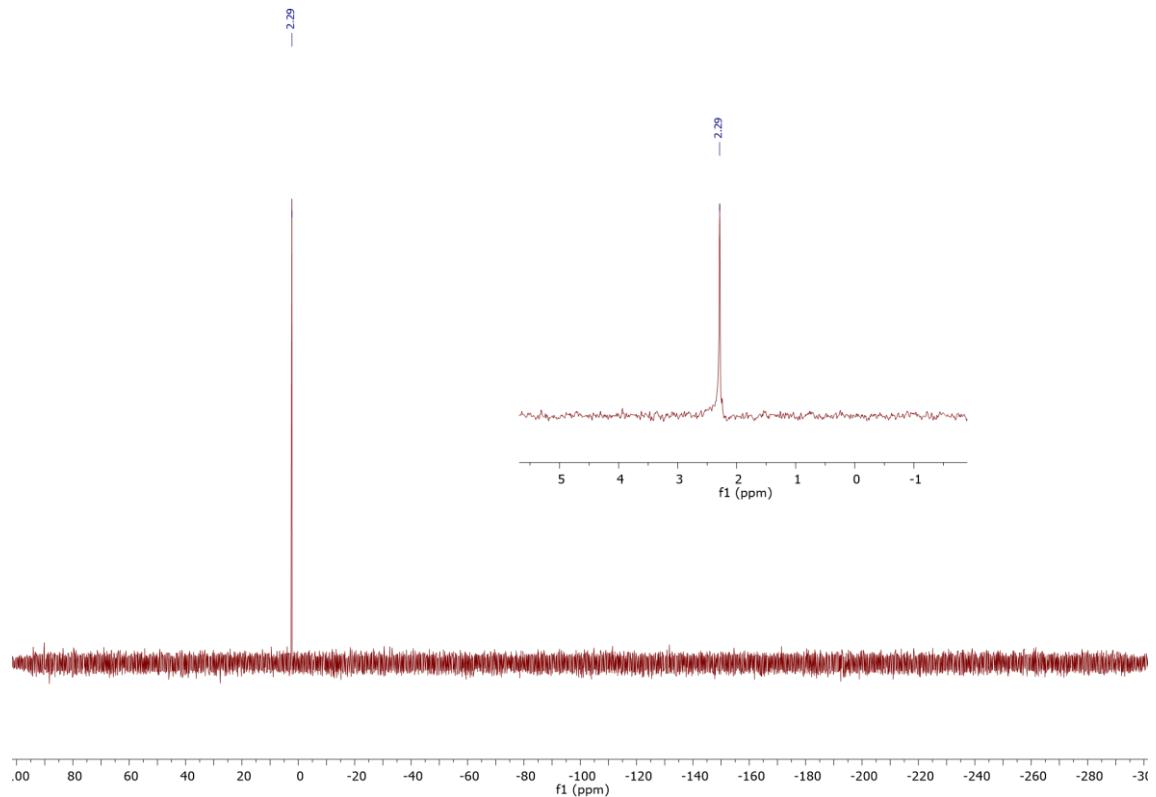
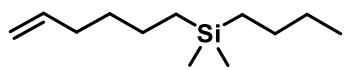


Figure S51. ^{29}Si NMR spectrum of 4c

2.18. Product 4d



¹H NMR (300 MHz, CDCl₃) δ 5.81 CH₂=CHR (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.07 – 4.83 CH₂=CHR (m, 2H), 2.05 (q, *J* = 6.9 Hz, 2H), 1.45 – 1.20 (m, 8H), 0.88 CH₃ (t, *J* = 6.9 Hz, 3H), 0.55 – 0.44 SiCH₂ (m, 4H), -0.05 SiMe₂ (s, 6H).

¹³C NMR (75 MHz, CDCl₃) δ 139.34, 114.24, 33.69, 33.05, 26.82, 26.31, 23.56, 15.28, 15.13, 13.99, -3.23.

^{29}Si NMR (79 MHz, CDCl_3) δ 2.27.

MS (EI, m/z): 142.2 (5.1), 141.0 (40.9), 126.9 (4.8), 114.9 (21.0), 112.9 (17.2), 98.9 (23.3), 84.9 (8.7), 80.9 (7.5), 74.2 (7.2), 73.0 (89.4), 60.1 (6.8), 58.9 (100).

Product contains 3% of disubstituted derivative

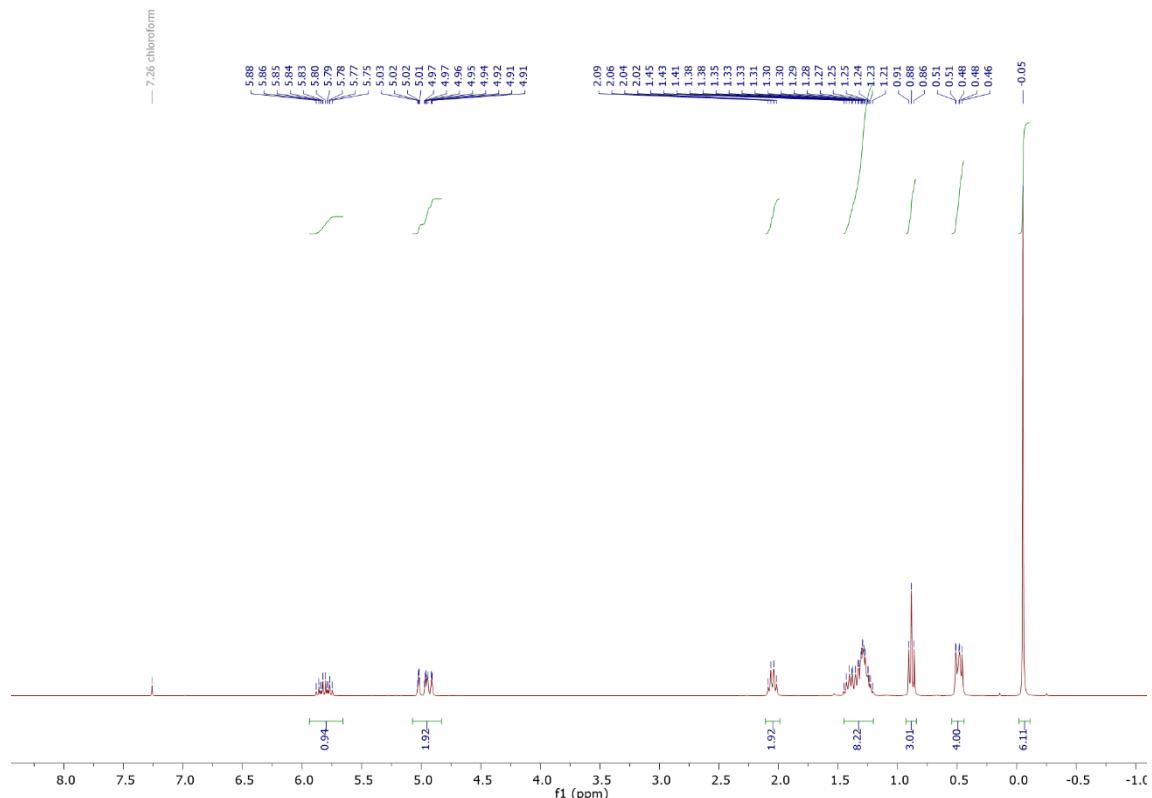


Figure S52. ^1H NMR spectrum of 4d

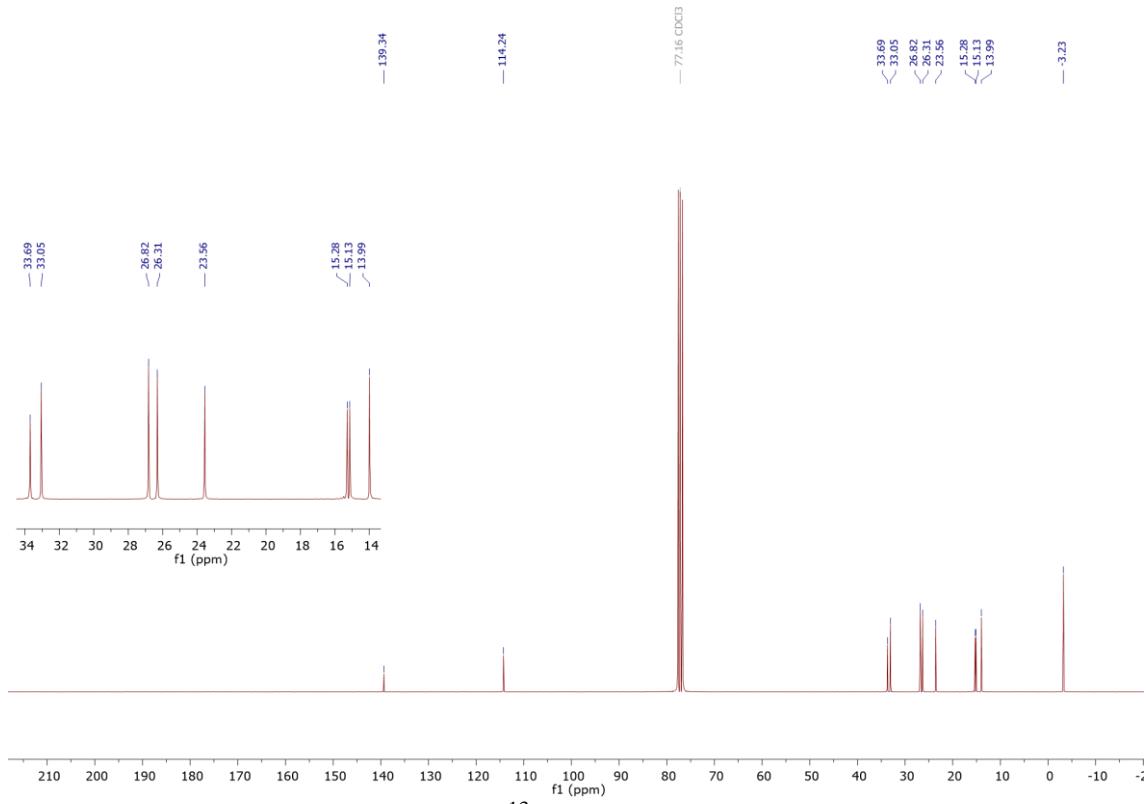


Figure S53. ^{13}C NMR spectrum of 4d

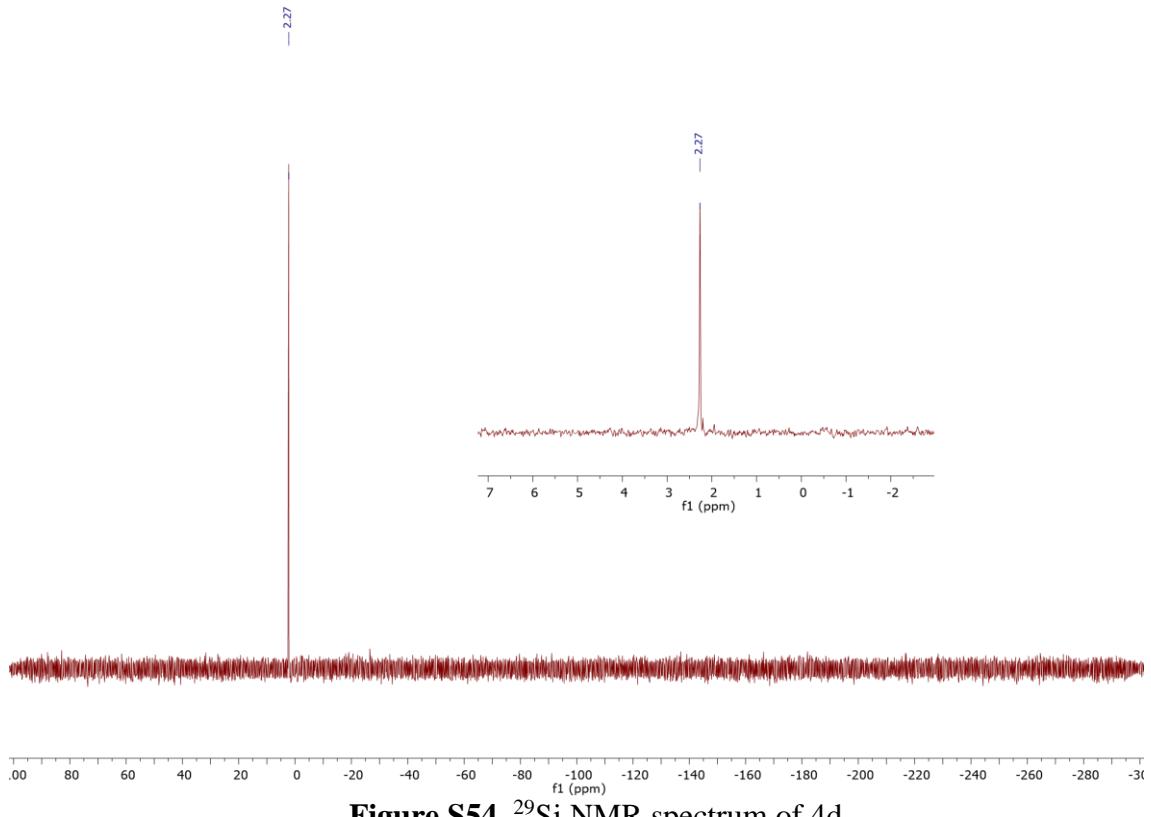
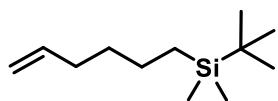


Figure S54. ^{29}Si NMR spectrum of 4d

2.19. Product 4e

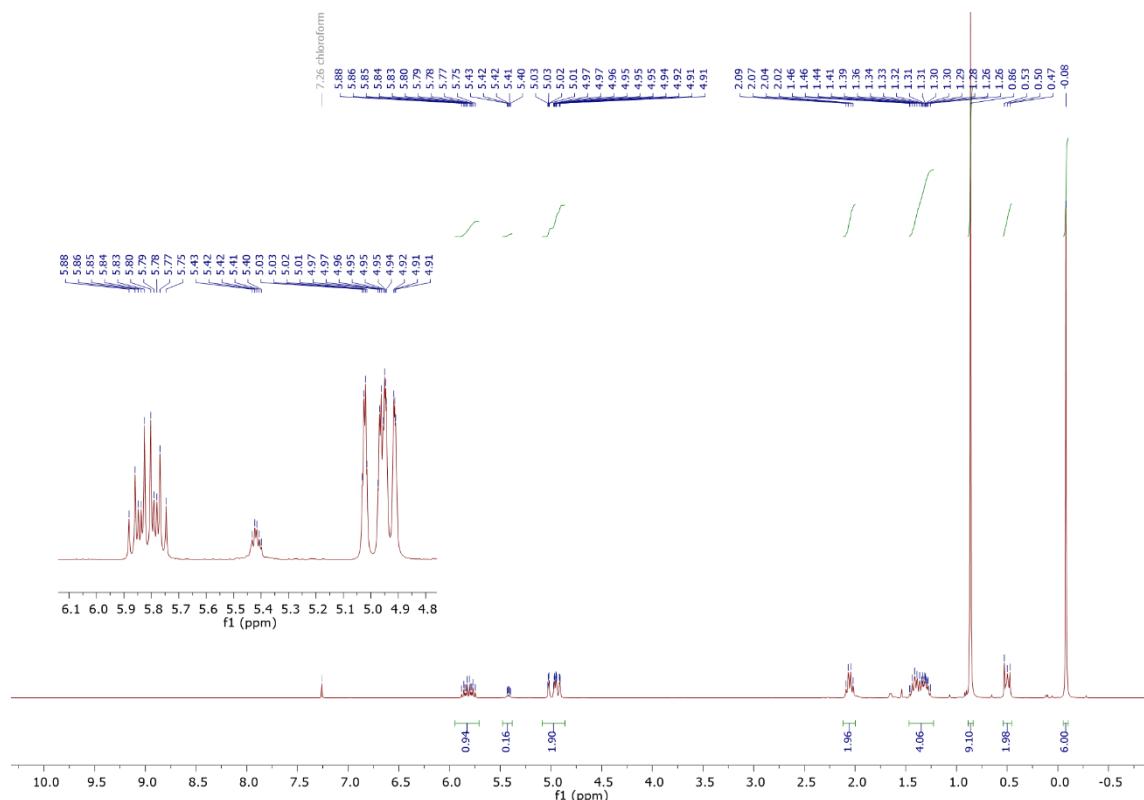


¹H NMR (300 MHz, CDCl₃) δ 5.81 CH₂=CHR (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 5.42 CH₃CH=CHR isomer (m, 2H) 5.09 – 4.86 CH₂=CHR (m, 2H), 2.05 (q, *J* = 6.9 Hz, 2H), 1.47 – 1.23 (m, 4H), 0.86 C(CH₃)₃ (s, 9H), 0.54 – 0.46 SiCH₂ (m, 2H), -0.08 SiMe₂ (s, 6H).

¹³C NMR (75 MHz, CDCl₃) δ 139.32, 114.27, 33.67, 33.26, 26.76, 23.94, 16.69, 12.46, -6.14.

²⁹Si NMR (79 MHz, CDCl₃) δ 8.16.

MS (EI, m/z): 142.1 (5.2), 141.0 (36.0), 112.9 (29.1), 98.9 (23.7), 84.9 (15.2), 80.9 (13.0), 74.1 (7.7), 72.9 (100), 60.1 (6.5), 58.9 (88.3)



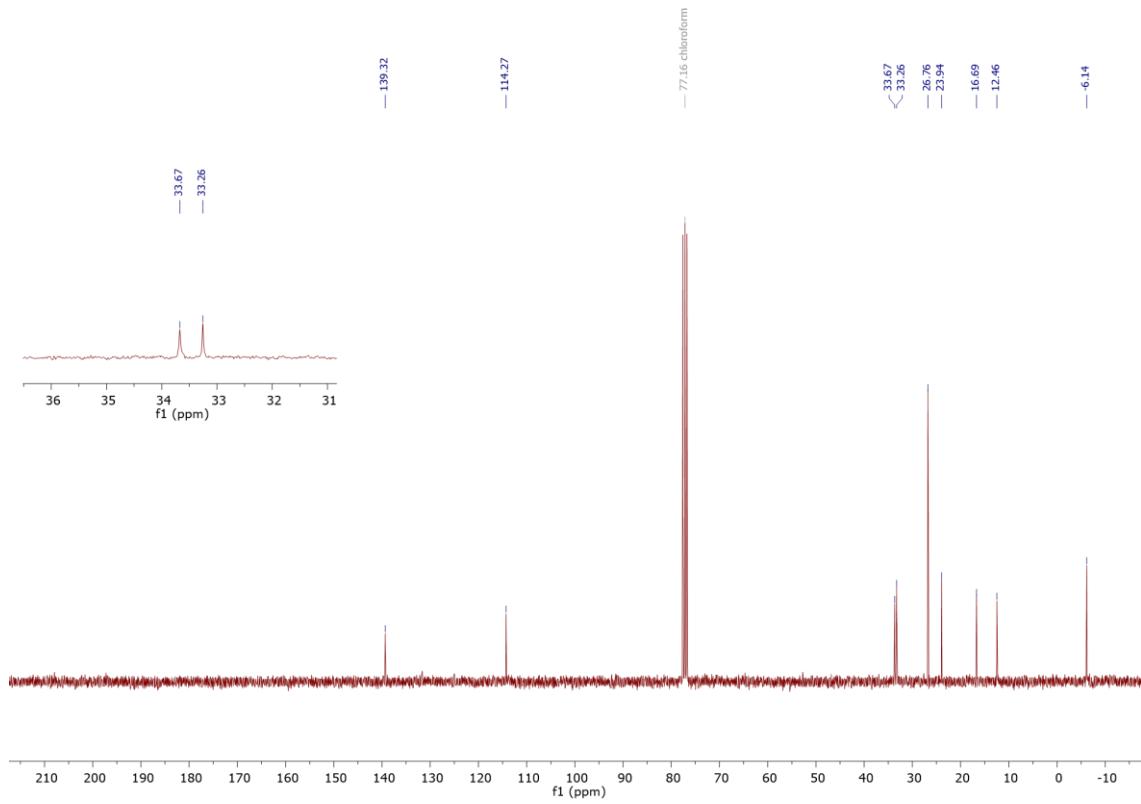


Figure S56. ^{13}C NMR spectrum of 4e

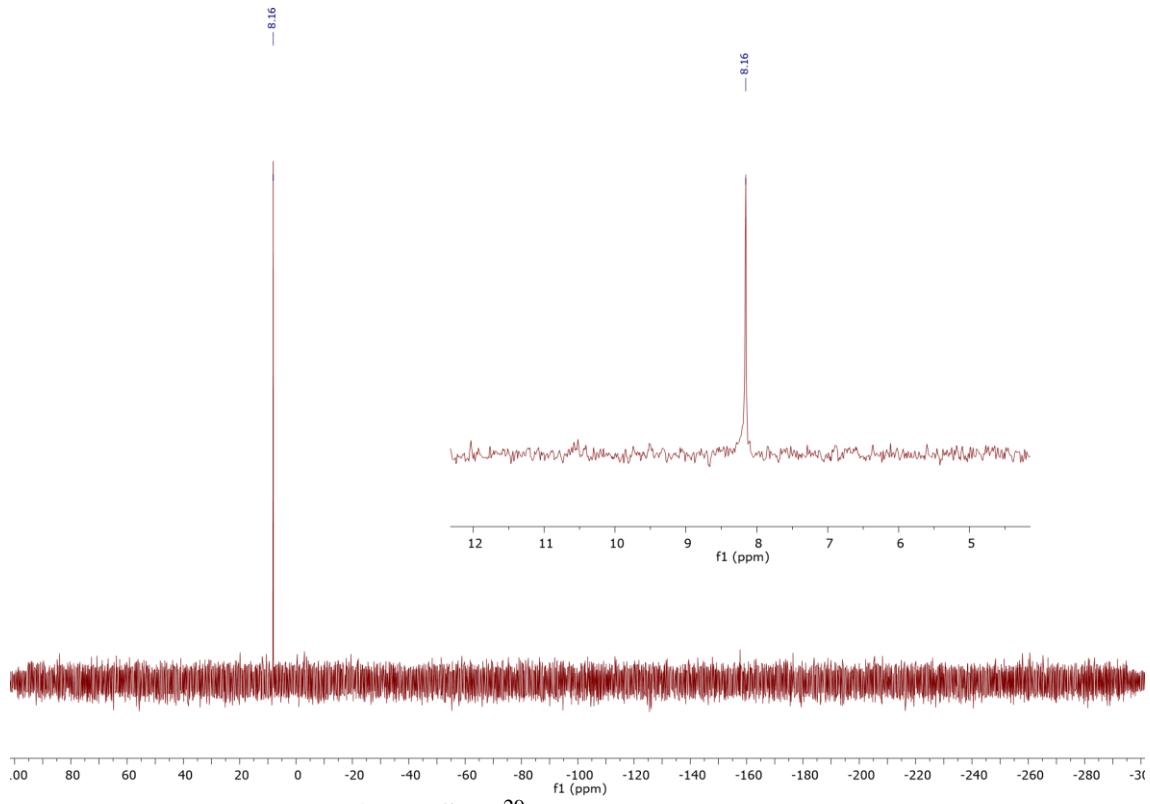
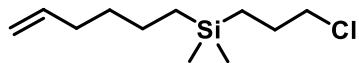


Figure S57. ^{29}Si NMR spectrum of 4e

2.20. Product 4f



¹H NMR (300 MHz, CDCl₃) δ 5.80 CH₂=CHR (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.11 – 4.80 CH₂=CHR (m, 2H), 3.50 CH₂Cl (t, *J* = 7.0 Hz, 2H), 2.05 (q, *J* = 6.9 Hz, 2H), 1.82 – 1.69 (m, 2H), 1.45 – 1.25 (m, 4H), 0.64 – 0.55 SiCH₂ (m, 2H), 0.54 – 0.46 SiCH₂ (m, 2H), -0.02 SiMe₂ (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 139.06, 114.21, 48.07, 33.48, 32.80, 27.74, 23.28, 14.93, 12.96, -3.49.

²⁹Si NMR (79 MHz, CDCl₃) δ 2.78.

MS (EI, m/z): 137.1 (4.1), 134.9 (15.4), 118.8 (4.0), 112.9 (4.2), 94.8 (34.6), 93.9 (6.9), 92.8 (100), 80.9 (4.8), 72.9 (8.7), 58.9 (24.3)

Product contains 3% of disubstituted derivative

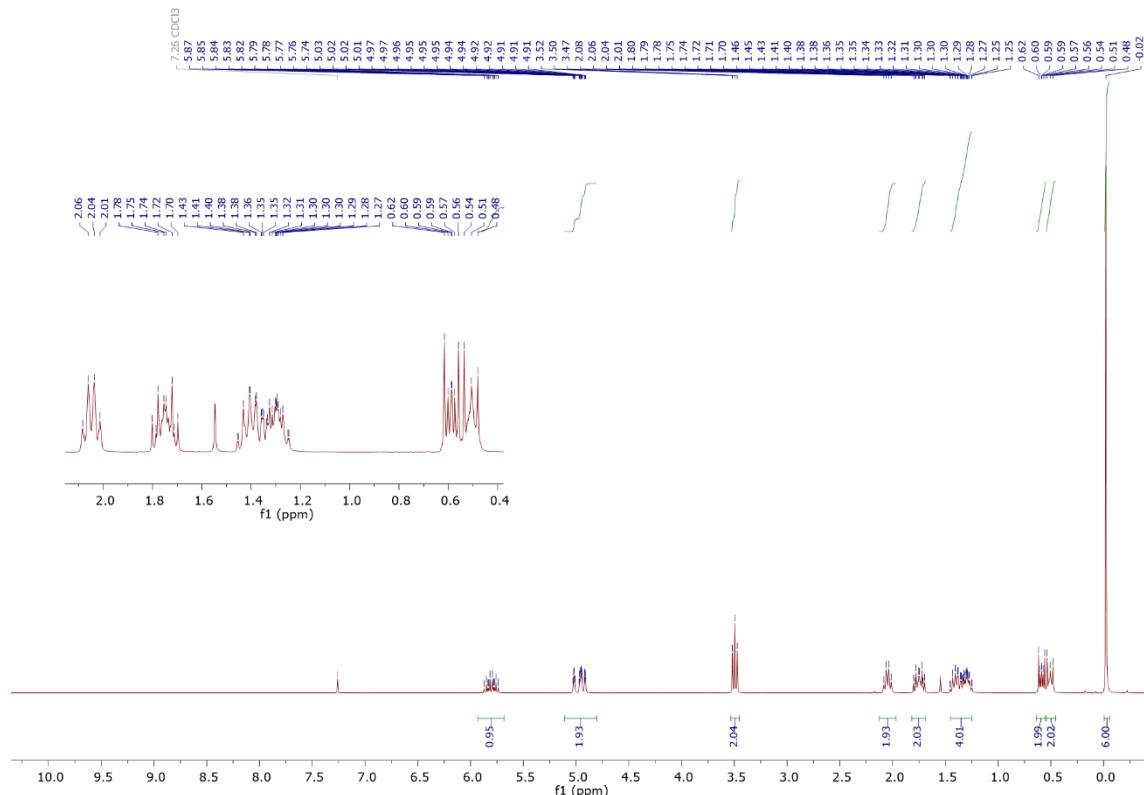


Figure S58. ^1H NMR spectrum of 4f

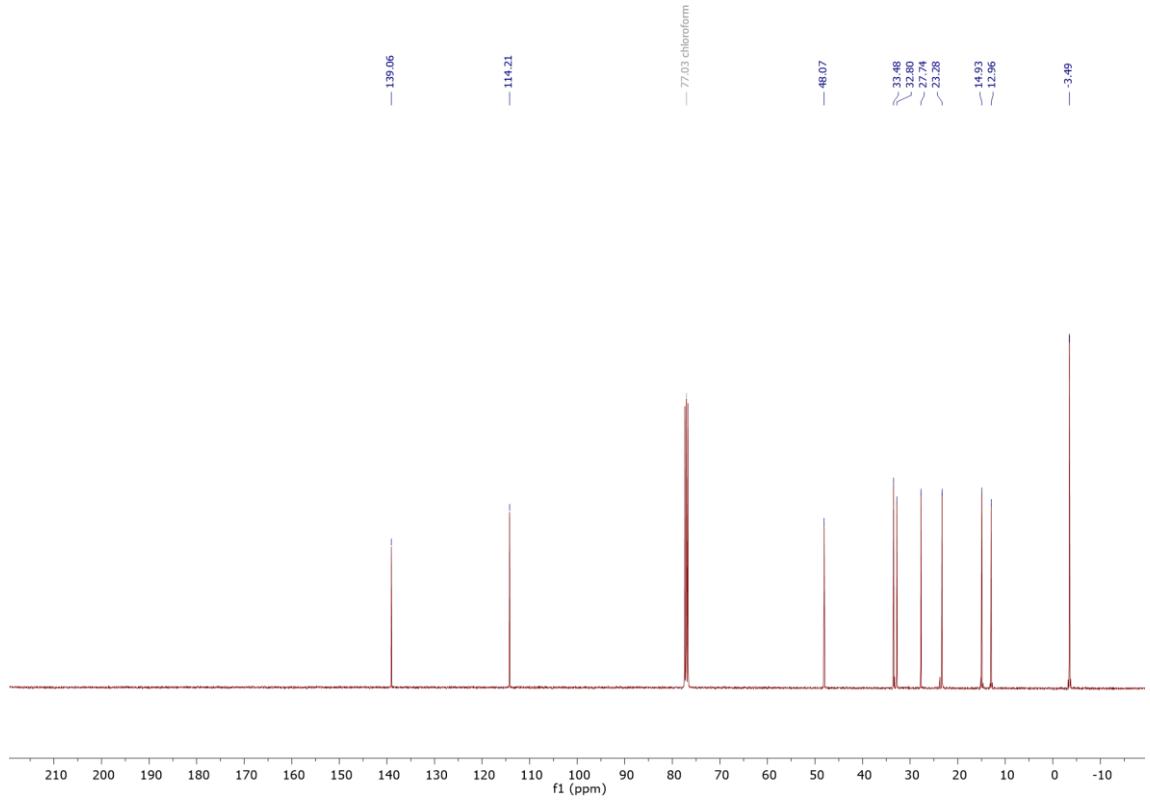


Figure S59. ^{13}C NMR spectrum of 4f

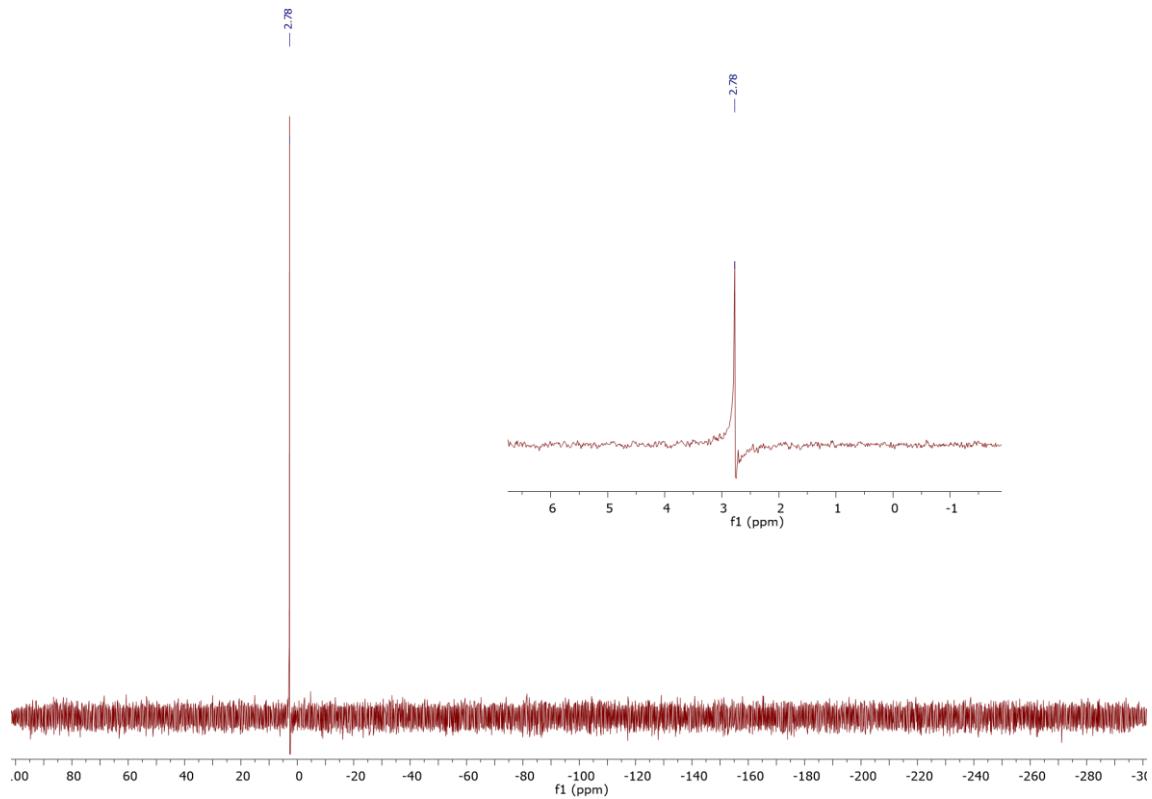
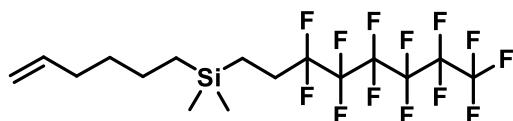


Figure S60. ^{29}Si NMR spectrum of 4f

2.21. Product 4g



¹H NMR (300 MHz, CDCl₃) δ 5.80 CH₂=CHR (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.06 – 4.88 CH₂=CHR (m, 2H), 2.13 – 1.90 (m, 4H), 1.47 – 1.26 (m, 4H), 0.79 – 0.68 SiMe₂ (m, 2H), 0.59 – 0.49 SiMe₂ (m, 2H), 0.02 SiMe₂ (s, 6H).

¹³C NMR (75 MHz, CDCl₃) δ 139.04, 114.47, 33.56, 32.82, 25.98, 23.23, 14.69, 4.54, -3.68.

²⁹Si NMR (79 MHz, CDCl₃) δ 3.76.

MS (EI, m/z): 308.9 (10.4), 288.9 (7.9), 262.9 (7.0), 244.9 (13.8), 238.9 (20.4), 194.9 (11.9), 138.9 (5.4), 116.9 (8.8), 112.9 (16.5), 102.9 (7.3), 98.9 (9.8), 88.9 (8.9), 84.9 (5.1), 80.9 (13.3), 78.2 (6.1), 76.9 (100), 73.0 (31.6) 68.9 (10.3), 62.9 (7.8), 58.9 (48.1), 55.0 (14.4), 51.0 (14.4).

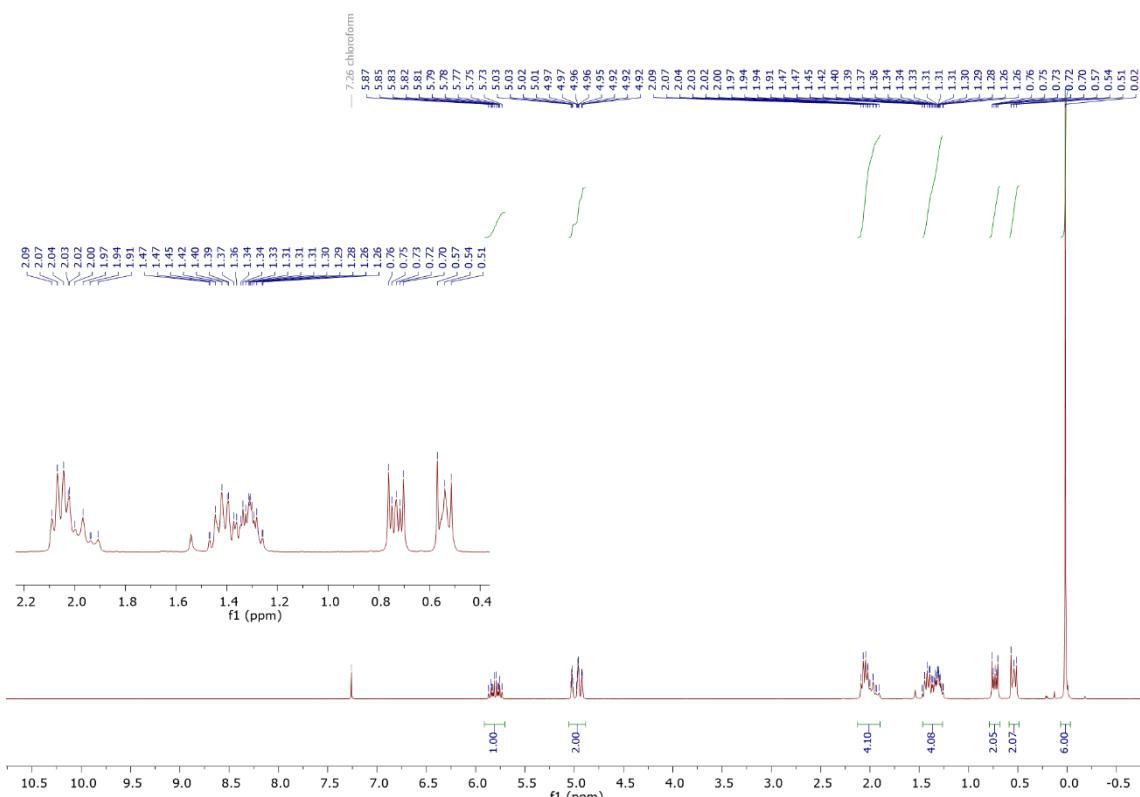


Figure S61. ¹H NMR spectrum of 4g

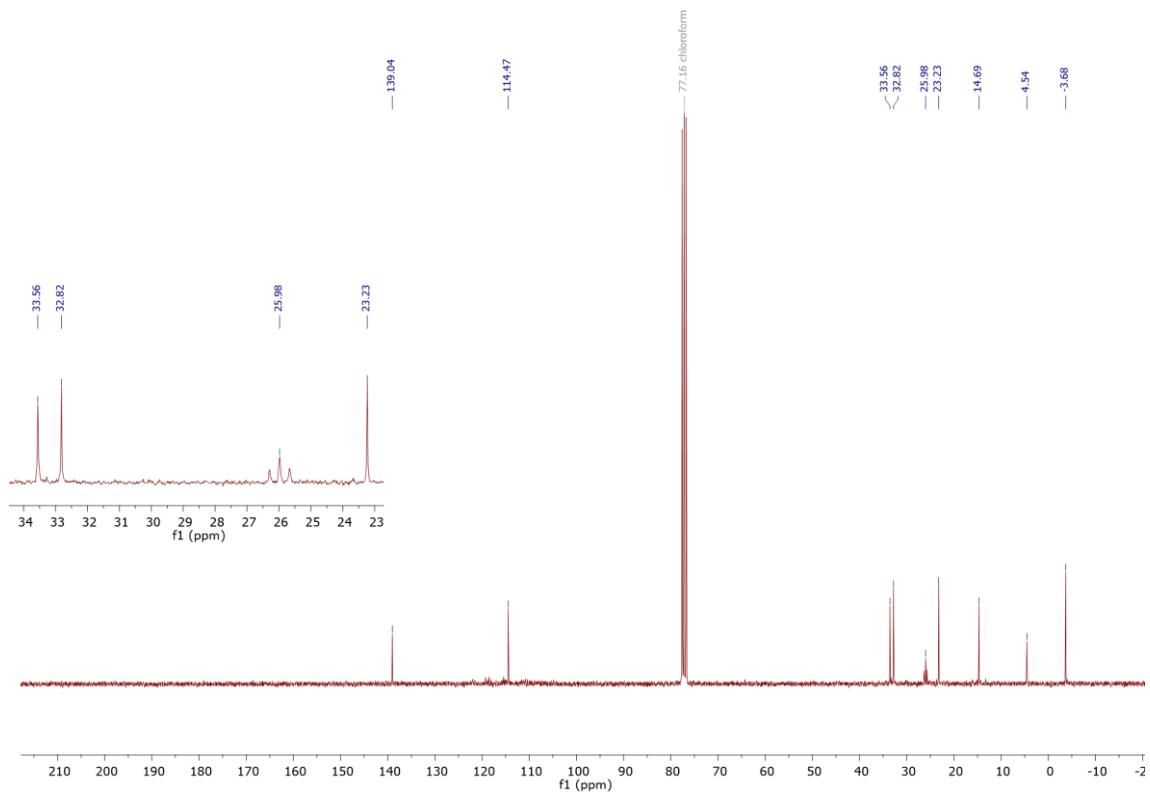


Figure S62. ^{13}C NMR spectrum of 4g

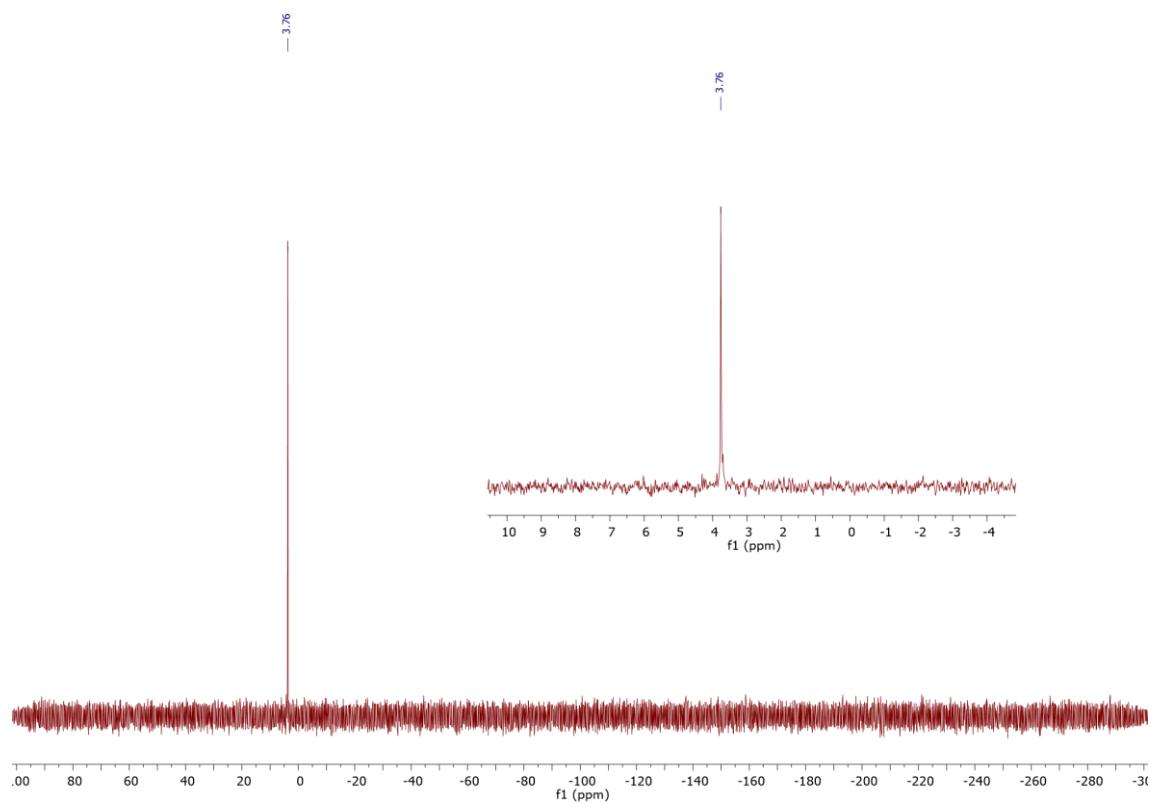
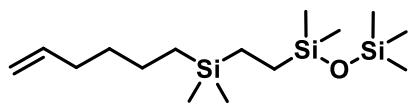


Figure S63. ^{29}Si NMR spectrum of 4g

2.22. Product 4h

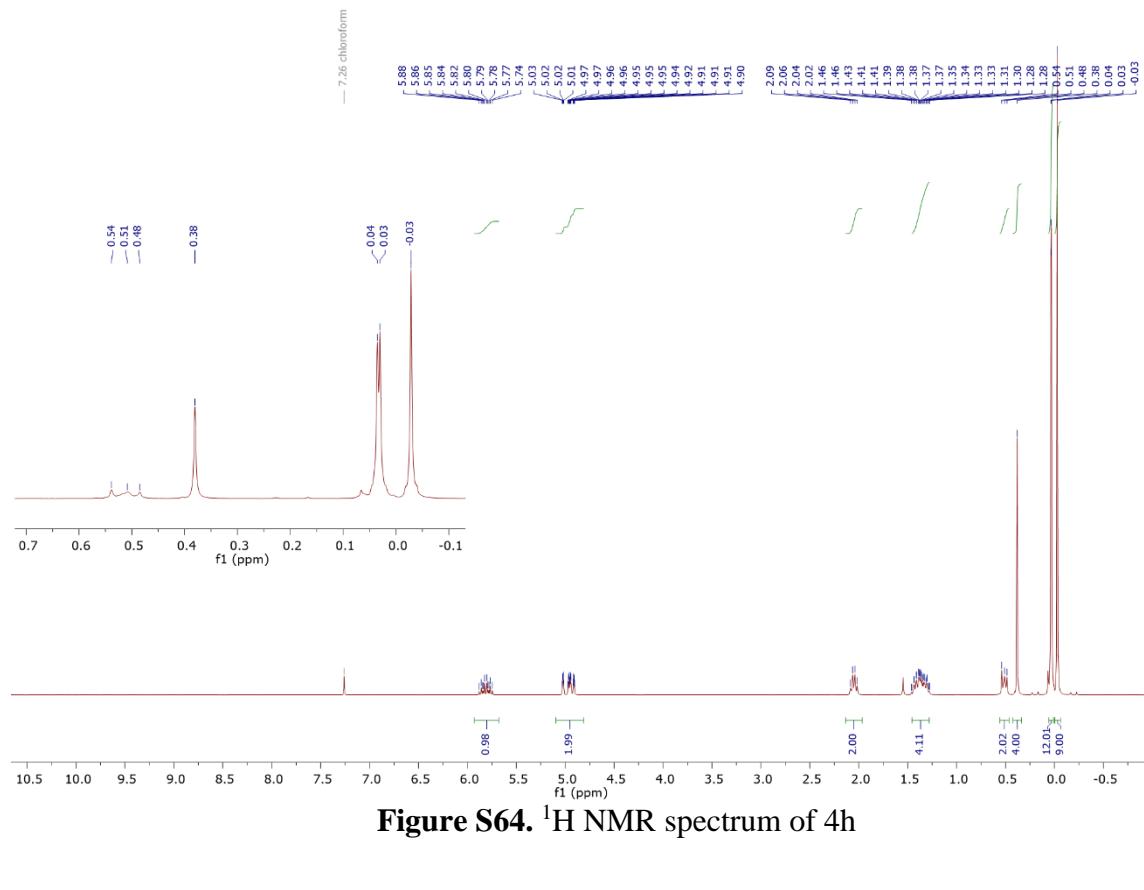


$^1\text{H NMR}$ (300 MHz, CDCl_3) δ 5.81 $\text{CH}_2=\text{CHR}$ (ddt, $J = 16.9, 10.1, 6.7$ Hz, 1H), 5.10 – 4.81 $\text{CH}_2=\text{CHR}$ (m, 2H), 2.05 (q, $J = 6.8$ Hz, 2H), 1.46 – 1.28 (m, 4H), 0.56 – 0.46 SiCH_2 (m, 2H), 0.38 SiCH_2 (s, 4H), 0.03 SiMe_2 (s, 12H), -0.03 SiMe_3 (s, 9H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 139.30, 114.27, 33.72, 32.78, 22.98, 18.42, 10.47, 8.22, 0.56, -0.21, -2.07.

$^{29}\text{Si NMR}$ (79 MHz, CDCl_3) δ 8.12, 7.18, 3.07.

MS (EI, m/z): 316.1 M^+ , 301.1 [$\text{M}-15$] $^+$, 233.0 (5.8), 215.0 (23.4), 146.9 (6.5), 144.9 (23.6), 141.0 (7.9), 135.0 (7.3), 134.1 (14.0), 132.9 (100), 116.9 (5.3), 72.9 (39.3), 58.9 (9.7)



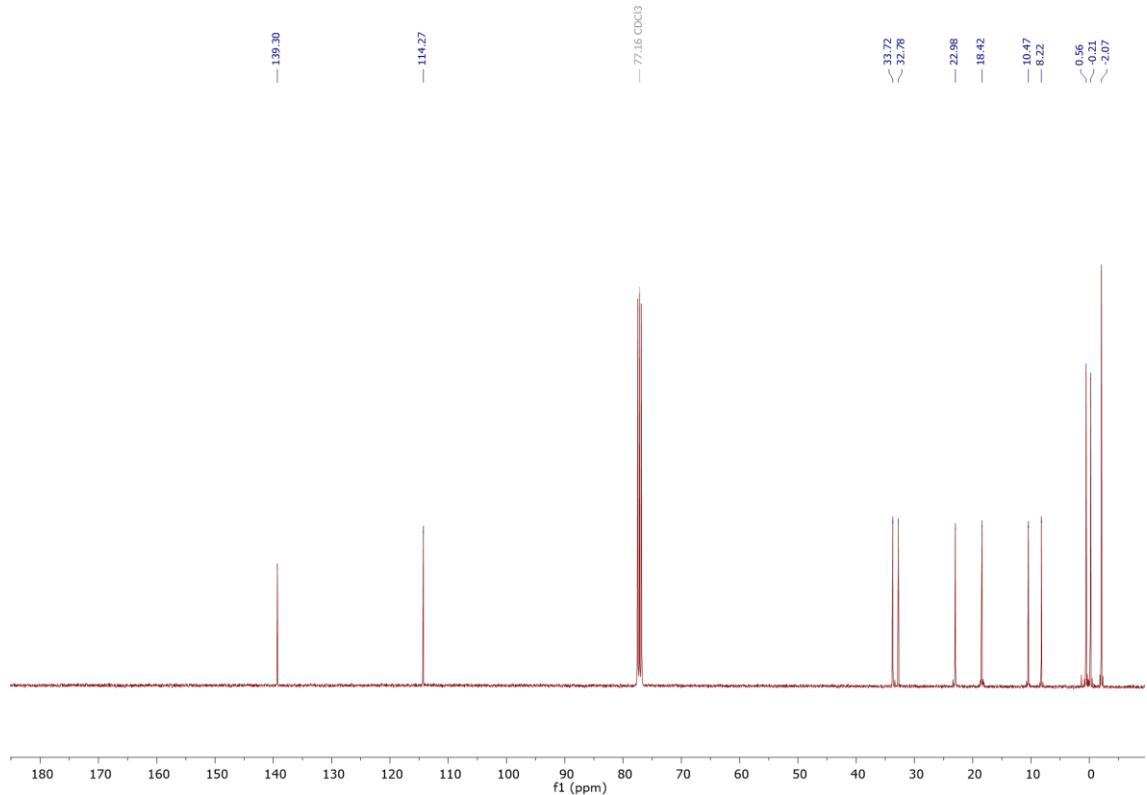


Figure S65. ¹³C NMR spectrum of 4h

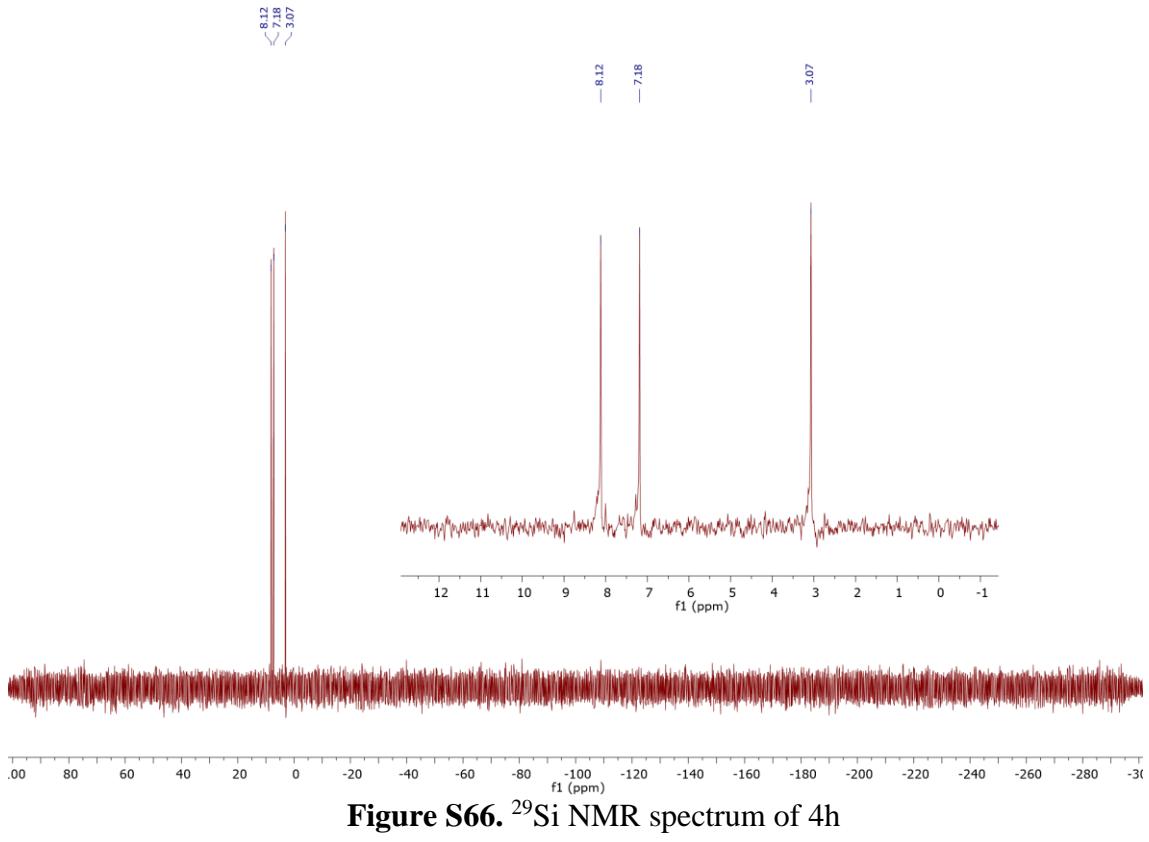
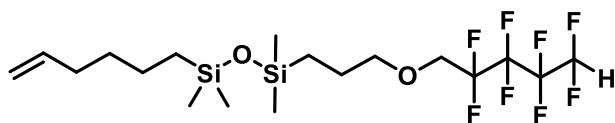


Figure S66. ^{29}Si NMR spectrum of 4h

2.23. Product 4i



¹H NMR (300 MHz, CDCl₃) δ 6.35 – 5.63 CF₂H, CH₂=CHR (m, 2H), 5.05 – 4.88 CH₂=CHR (m, 2H), 3.92 (t, *J* = 14.0 Hz, 2H), 3.55 (t, *J* = 6.9 Hz, 2H), 2.05 (q, *J* = 6.9 Hz, 2H), 1.68 – 1.56 (m, 2H), 1.45 – 1.28 (m, 4H), 0.58 – 0.44 (m, 4H), 0.06 (s, 6H), 0.04 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 139.26, 115.69, 114.27, 113.15, 110.32, 107.80, 105.28, 75.98, 67.64, 33.67, 32.72, 23.48, 22.92, 18.33, 14.12, 0.45, 0.38.

²⁹Si NMR (79 MHz, CDCl₃) δ 7.87, 7.23

MS (EI, m/z): 473.1 [M-15]⁺, 153.0 (7.5), 152.1 (13.5), 150.9 (100), 136.9 (13.5), 132.9 (32.5), 72.9 (6.5).

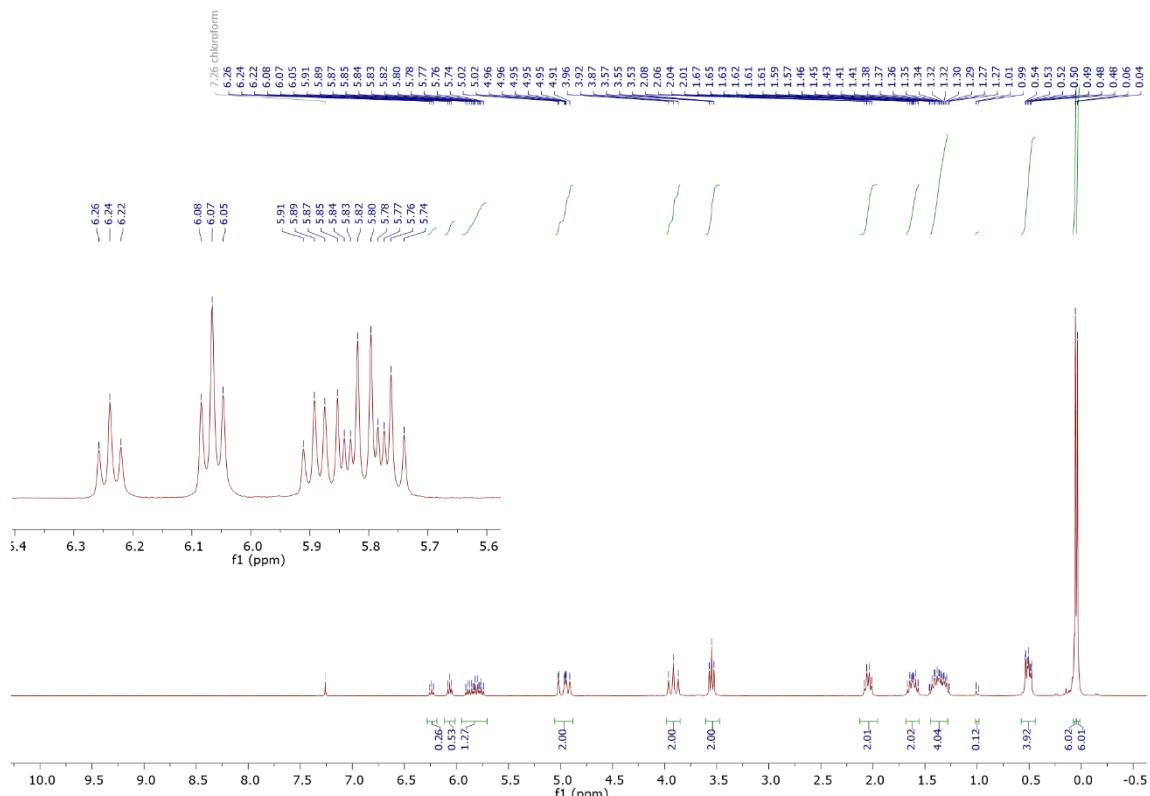


Figure S67. ^1H NMR spectrum of 4i

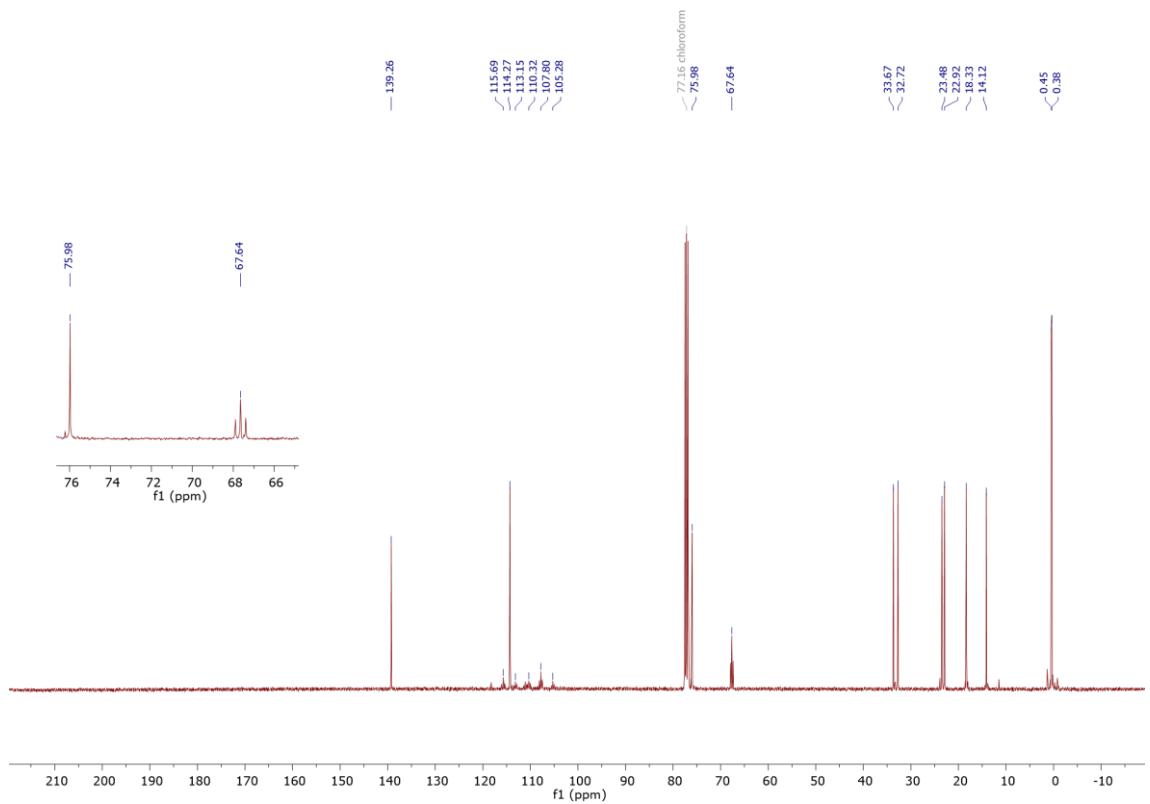


Figure S68. ^{13}C NMR spectrum of 4i

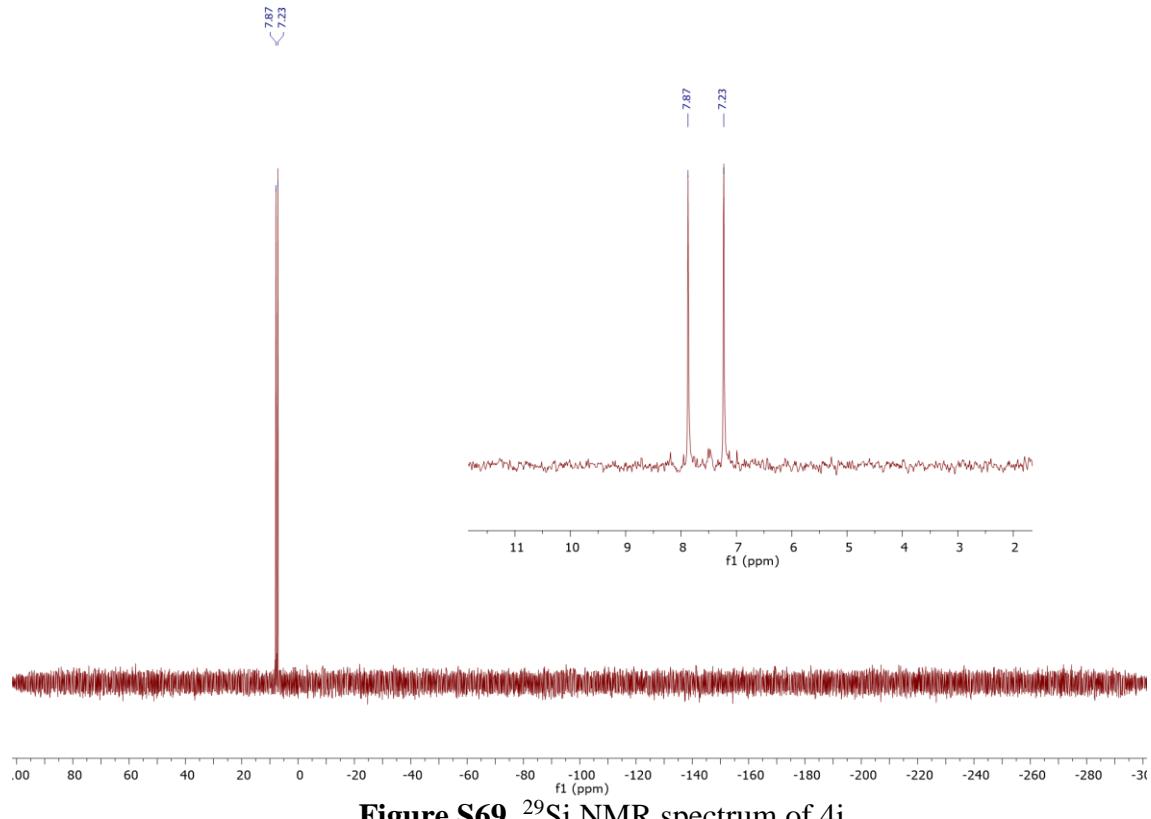
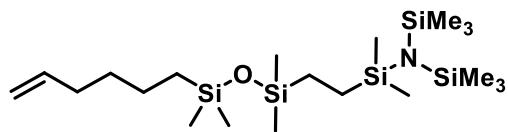


Figure S69. ${}^{29}\text{Si}$ NMR spectrum of 4i

2.24. Product 4j



¹H NMR (300 MHz, CDCl₃) δ 5.81 CH₂=CHR (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.05 – 4.88 CH₂=CHR (m, 2H), 2.05 (q, *J* = 6.8 Hz, 2H), 1.45 – 1.29 (m, 4H), 0.55 – 0.46 SiCH₂ (m, 4H), 0.44 – 0.35 SiCH₂ (m, 2H), 0.18 SiMe₃ (s, 18H), 0.16 SiMe₂ (s, 6H), 0.04 SiMe₂ (s, 12H).

¹³C NMR (75 MHz, CDCl₃) δ 139.30, 114.27, 33.72, 32.79, 22.99, 18.43, 12.14, 10.79, 5.74, 3.19, 0.57, -0.15.

²⁹Si NMR (79 MHz, CDCl₃) δ 8.11, 7.27, 5.07, 2.23.

MS (EI, m/z): 461.2 M⁺, 220.1 (11.7), 219.1 (23.0), 218.0 (100), 216.0 (11.8), 202.0 (6.1), 132.9 (17.2), 129.9 (6.8), 72.9 (6.8), .

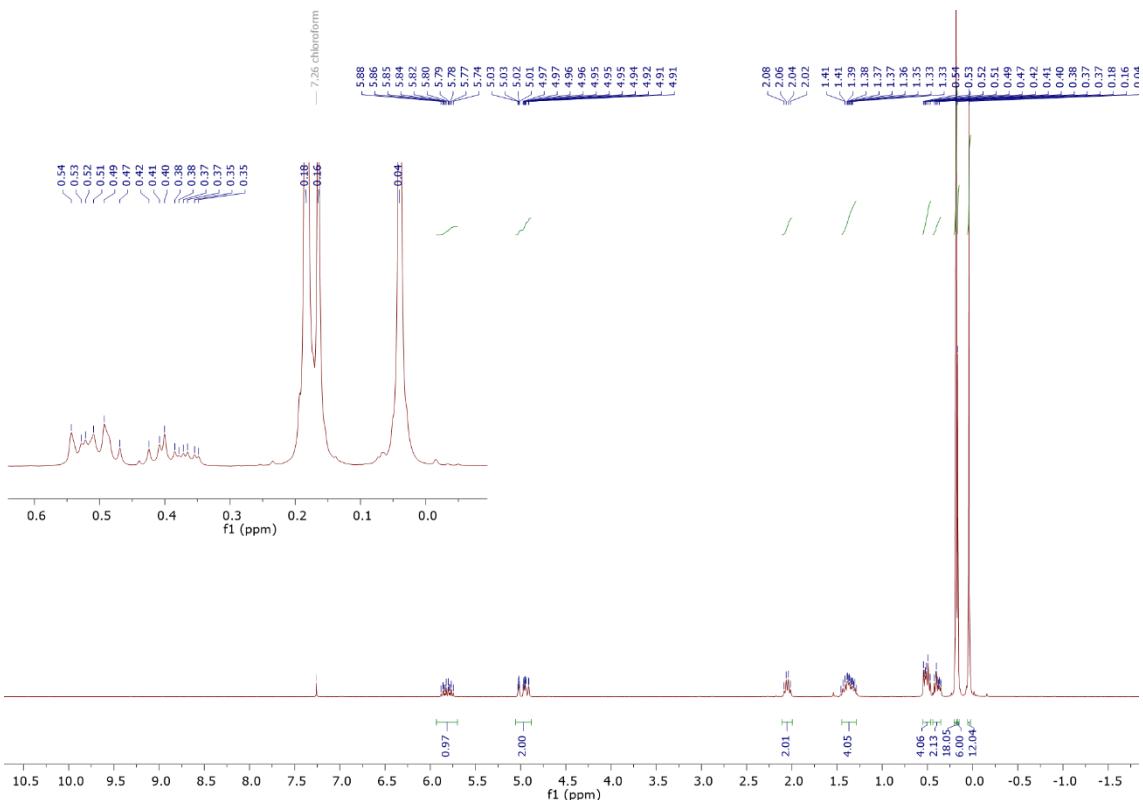


Figure S70. ^1H NMR spectrum of 4j

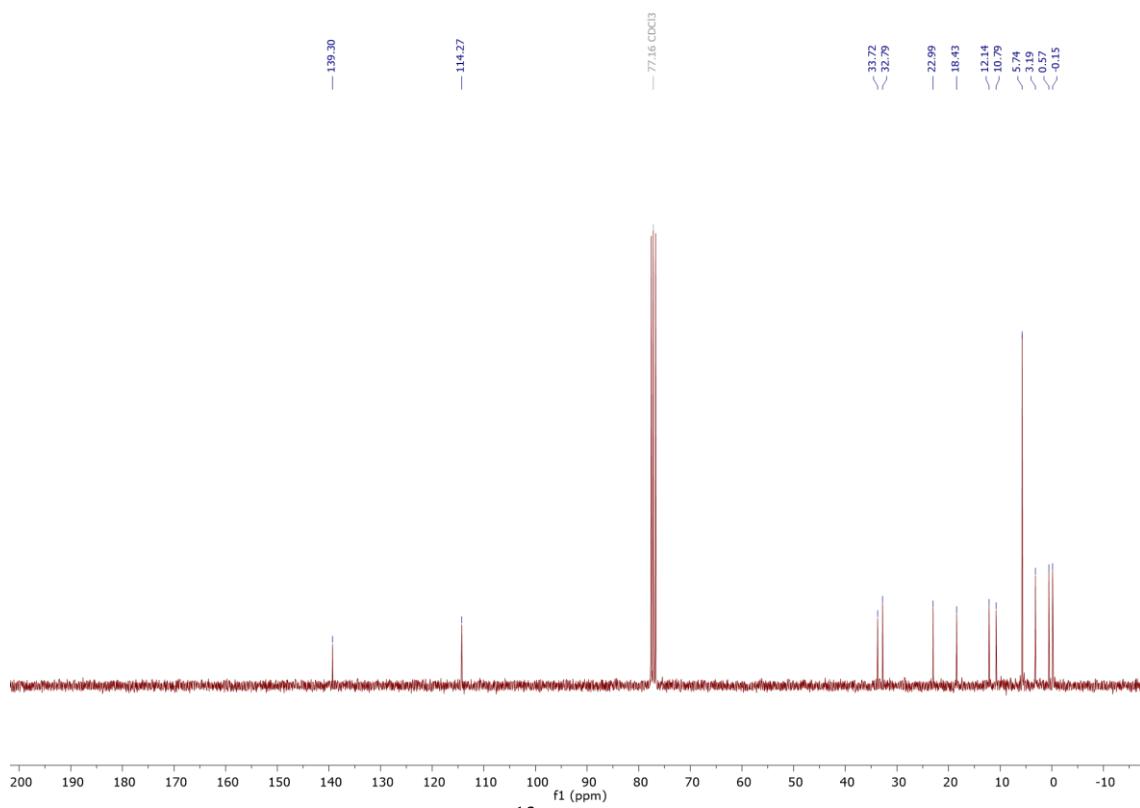


Figure S71. ¹³C NMR spectrum of 4j

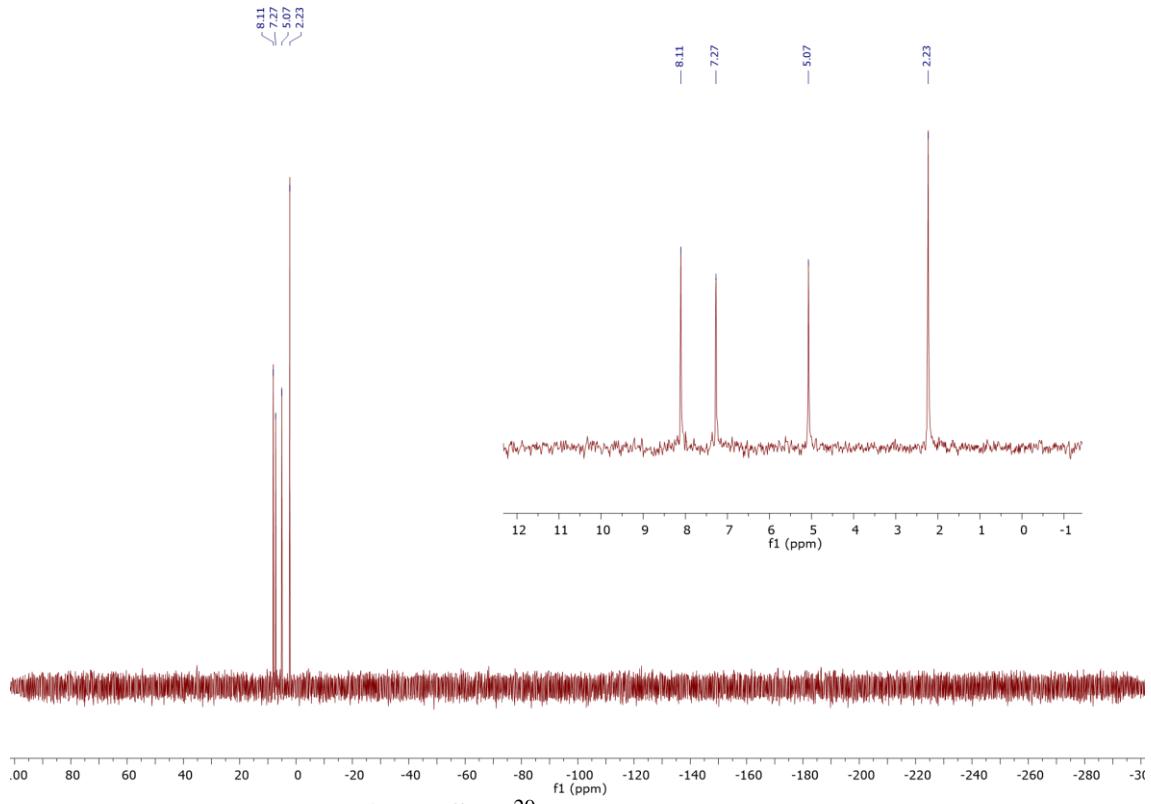
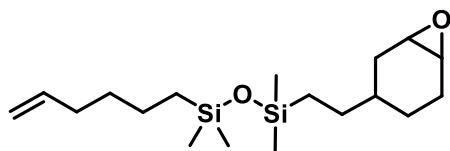


Figure S72. ${}^{29}\text{Si}$ NMR spectrum of 4j

2.25. Product 4k



¹H NMR (300 MHz, CDCl₃) δ 5.81 CH₂=CHR (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 5.06 – 4.86 CH₂=CHR (m, 2H), 3.21 – 3.05 (m, 2H), 2.22 – 1.64 (m, 5H), 1.54 – 0.82 (m, 10H), 0.58 – 0.37 SiCH₂ (m, 4H), 0.02 SiMe₂ (s, 12H).

¹³C NMR (75 MHz, CDCl₃) δ 139.25, 114.29, 53.44, 52.92, 52.17, 52.13, 35.54, 33.69, 32.73, 32.42, 31.70, 30.56, 30.33, 29.77, 26.91, 25.54, 24.21, 23.74, 22.93, 18.36, 15.40, 15.27, 0.53, 0.38.

²⁹Si NMR (79 MHz, CDCl₃) δ 7.50, 7.42, 7.40.

MS (EI, m/z): 325.0 [M-15]⁺, 151.0 (7.2), 150.1 (13.4), 148.9 (100), 134.9 (16.2), 134.0 (9.7), 132.9 (67.7), 118.8 (6.0), 108.9 (7.6), 80.9 (12.1), 80.0 (5.9), 78.9 (6.7), 72.9 (8.3), 66.9 (24.2).

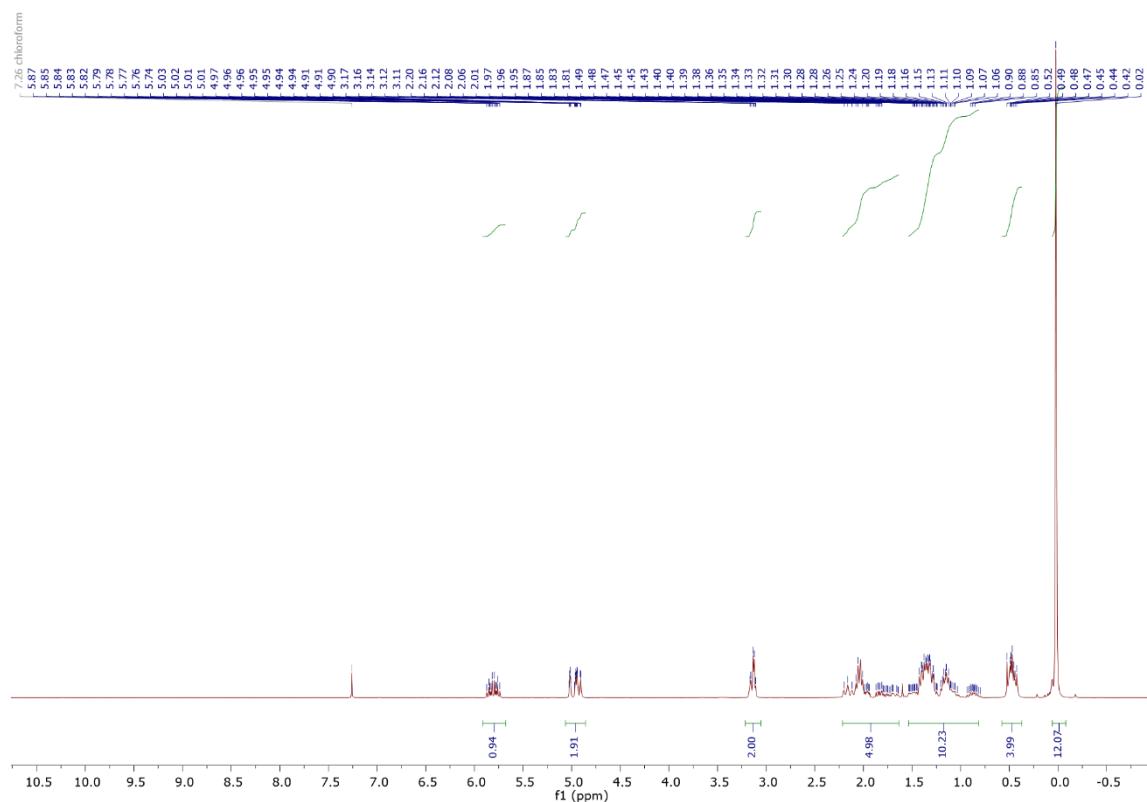


Figure S73. ¹H NMR spectrum of 4k

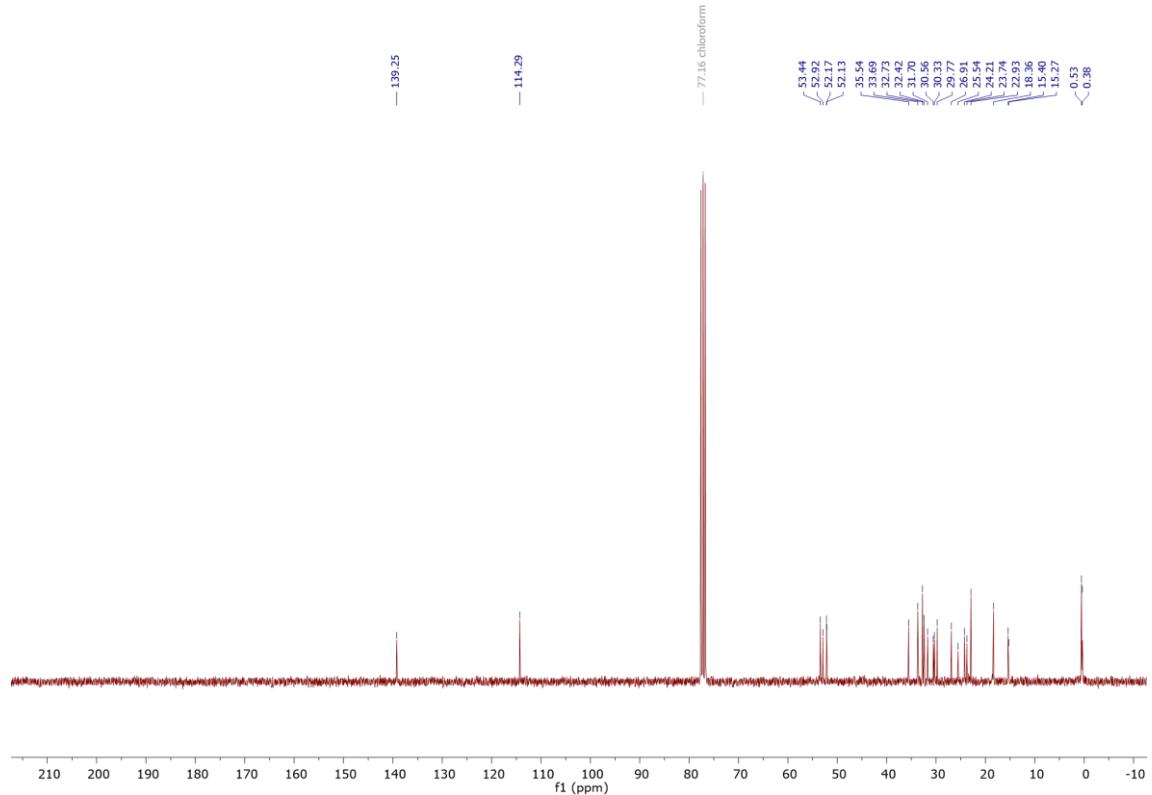


Figure S74. ^{13}C NMR spectrum of 4k

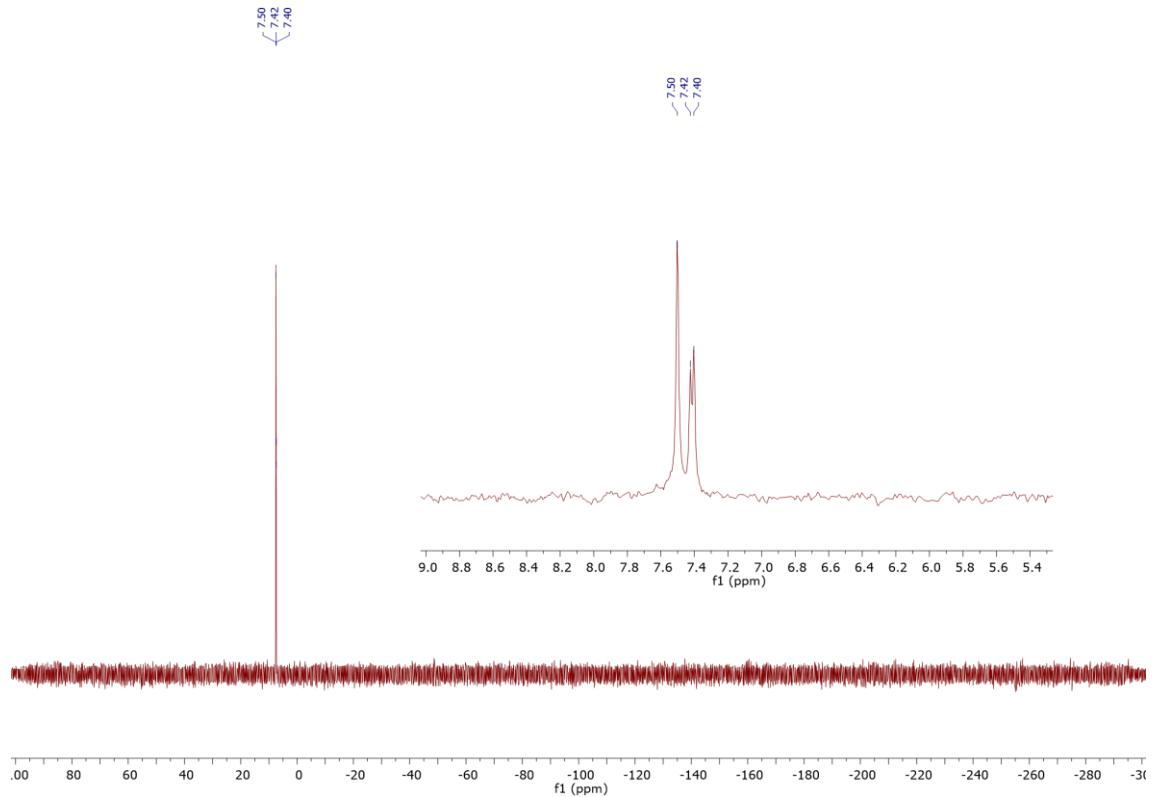
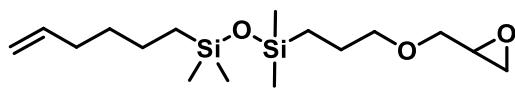


Figure S75. ^{29}Si NMR spectrum of 4k

2.26. Product 4l



¹H NMR (300 MHz, CDCl₃) δ 5.80 CH₂=CHR (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 5.05 – 4.86 CH₂=CHR (m, 2H), 3.70 (dd, *J* = 11.5, 3.1 Hz, 1H), 3.54 – 3.31 (m, 3H), 3.20 – 3.08 (m, 1H), 2.85 – 2.75 (m, 1H), 2.61 (dd, *J* = 5.0, 2.7 Hz, 1H), 2.04 (q, *J* = 6.8 Hz, 2H), 1.66 – 1.54 (m, 2H), 1.45 – 1.26 (m, 4H), 0.57 – 0.43 SiCH₂ (m, 4H), 0.04 SiMe₂ (s, 12H).

¹³C NMR (75 MHz, CDCl₃) δ 139.25, 114.28, 74.53, 71.57, 51.03, 44.52, 33.67, 32.71, 23.63, 22.91, 18.33, 14.37, 0.50, 0.42.

²⁹Si NMR (79 MHz, CDCl₃) δ 7.66, 7.38.

MS (EI, m/z): 315.1 [M-1]⁺, 177.1 (6.4), 176.1 (12.0), 174.9 (76.7), 148.9 (15.1), 135.0 (11.1), 134.1 (13.6), 132.9 (100), 130.9 (8.4), 118.8 (5.1), 116.9 (5.1), 72.9 (7.4), 57.0 (5.4).

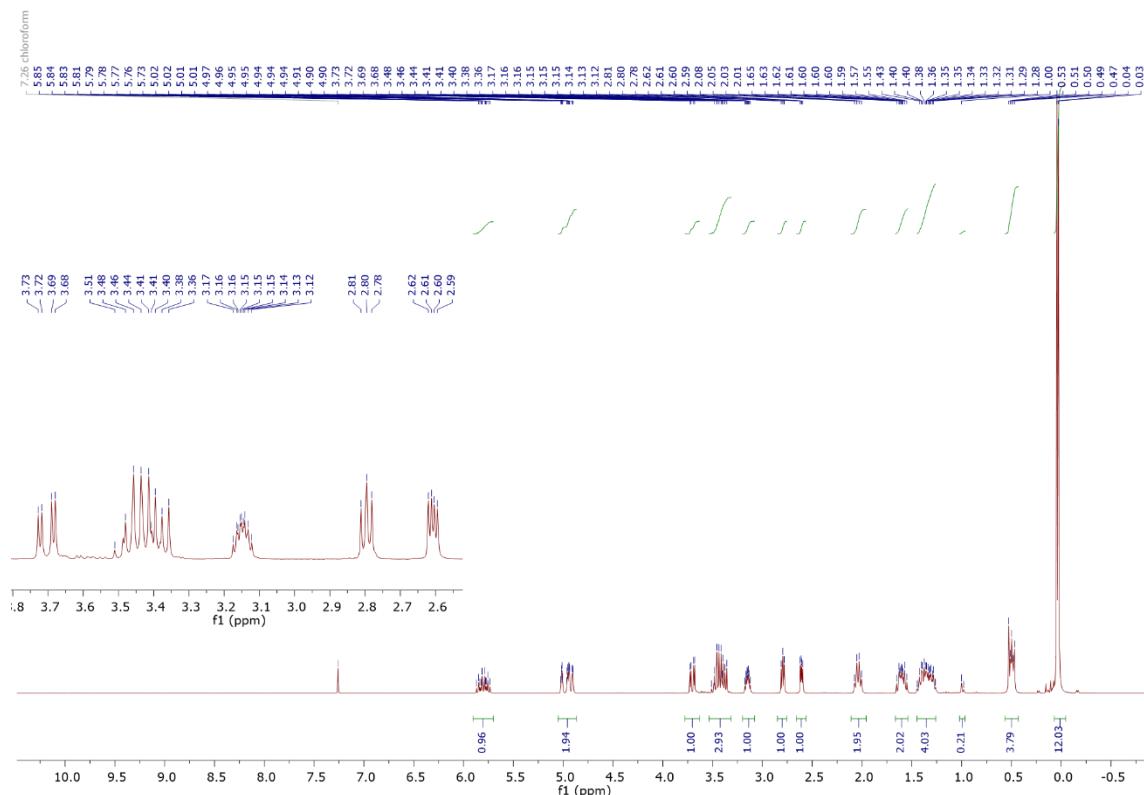


Figure S76. ^1H NMR spectrum of 41

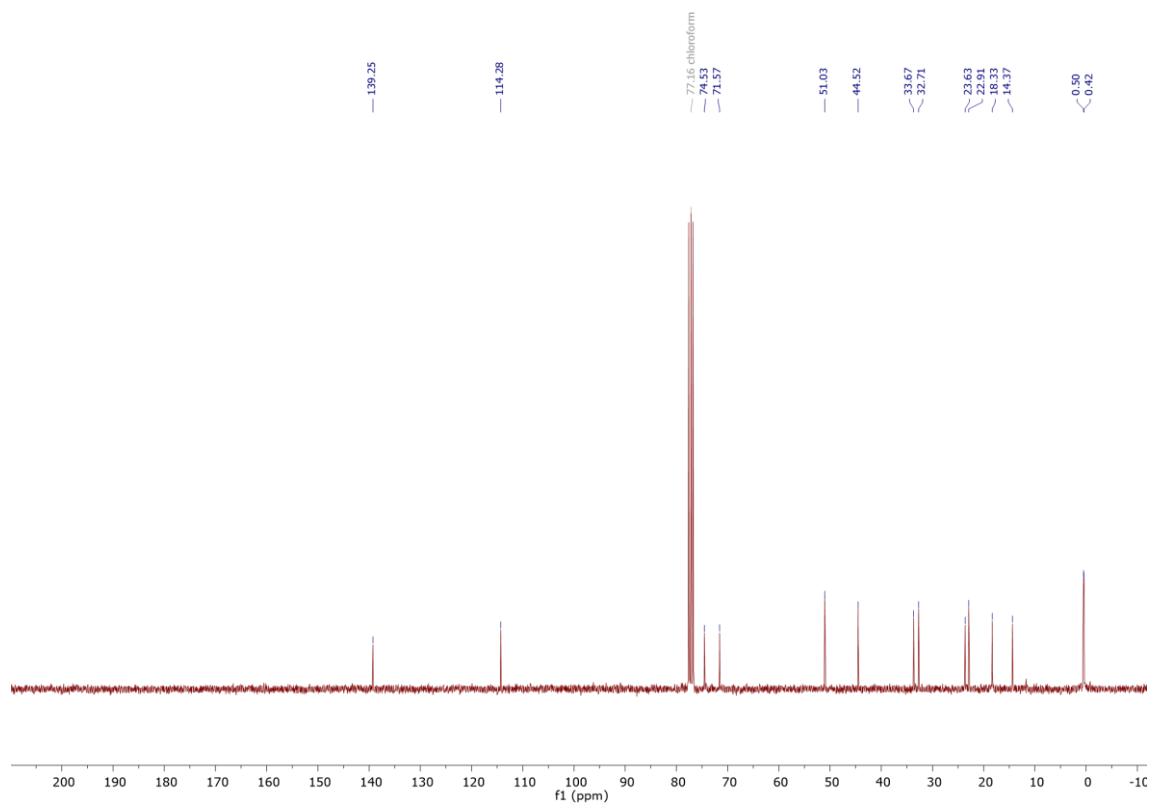


Figure S77. ^{13}C NMR spectrum of 41

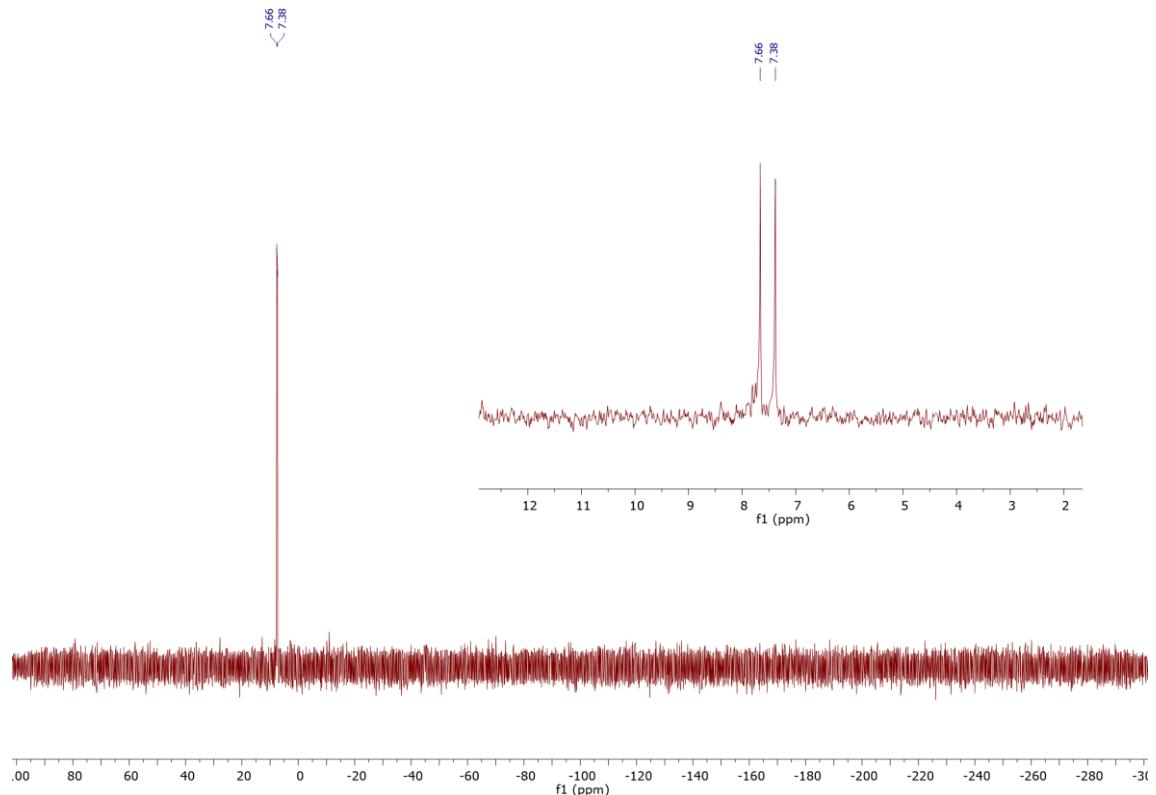
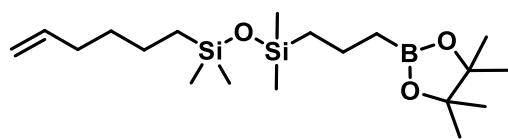


Figure S78. ^{29}Si NMR spectrum of 4l

2.27. Product 4m



¹H NMR (300 MHz, CDCl₃) δ 5.81 CH₂=CHR (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 5.09 – 4.81 CH₂=CHR (m, 2H), 2.04 (q, *J* = 7.0 Hz, 2H), 1.58 – 1.27 (m, 6H), 1.24 (s, 12H), 0.82 BCH₂ (t, *J* = 7.6 Hz, 2H), 0.53 SiCH₂ (m, 4H), 0.02 SiMe₂ (s, 12H).

¹³C NMR (75 MHz, CDCl₃) δ 139.32, 114.25, 82.94, 33.70, 32.74, 24.97, 22.94, 21.83, 18.36, 18.10, 0.56, 0.52.

²⁹Si NMR (79 MHz, CDCl₃) δ 7.20, 7.00.

MS (EI, m/z): 269.2 [M-15]⁺, 203.2 (8.9), 202.2 (18.0), 201.0 (100), 199.8 (23.1), 186.9 (7.5), 175.0 (12.4), 158.9 (30.1), 144.9 (7.2), 134.1 (7.6), 132.9 (56.1), 116.8 (5.5), 83.0 (12.9), 72.9 (8.1),

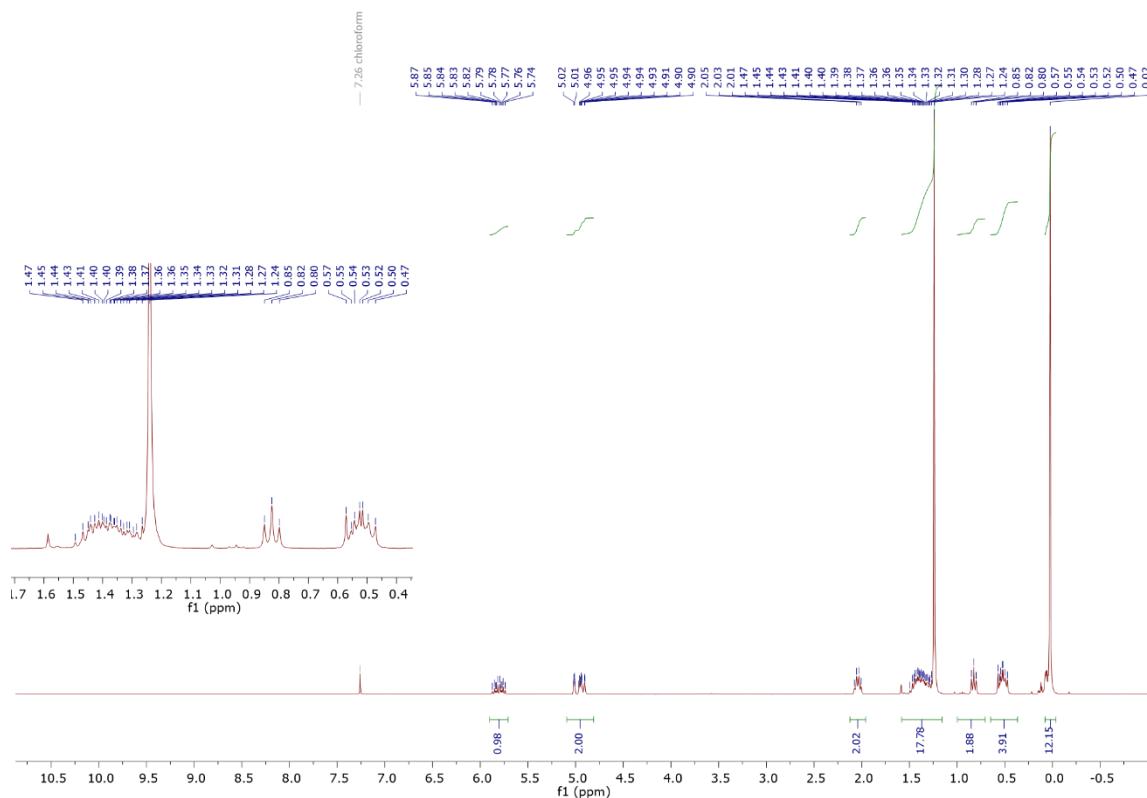


Figure S79. ¹H NMR spectrum of 4m

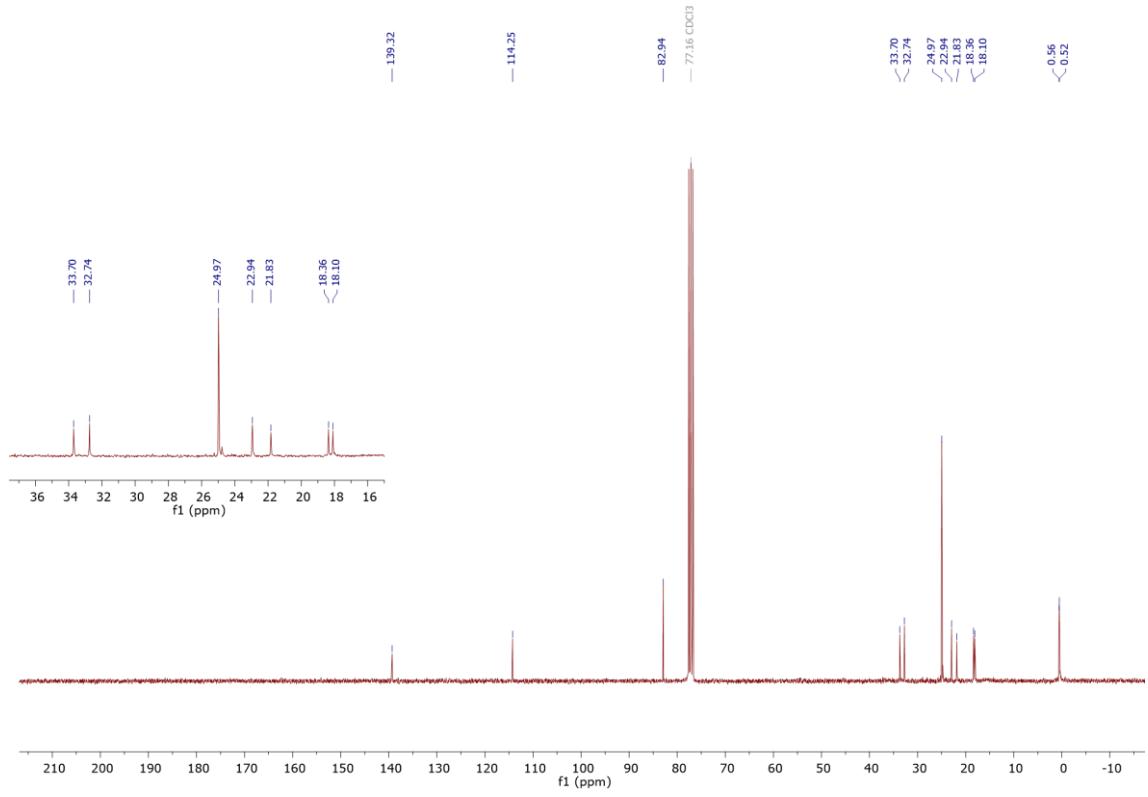


Figure S80. ^{13}C NMR spectrum of 4m

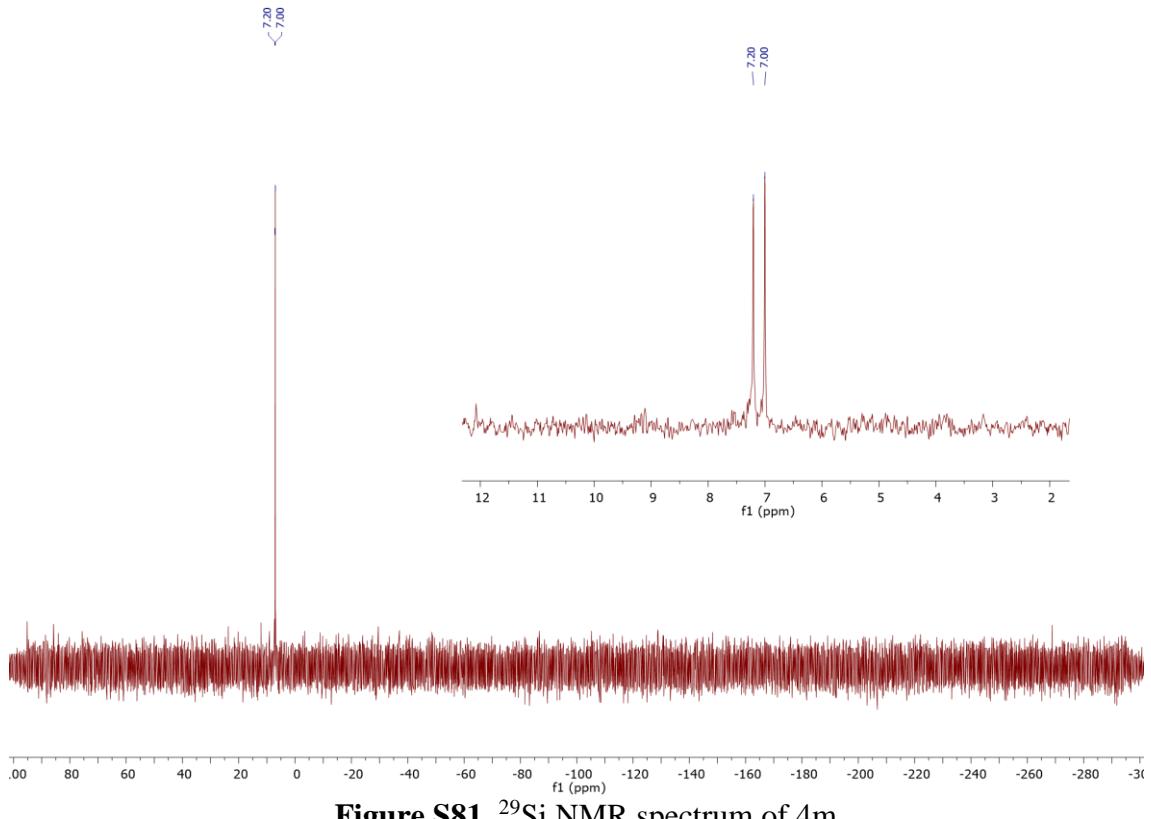


Figure S81. ^{29}Si NMR spectrum of 4m