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Supporting materials

A highly selective synthesis of new alkenylsilsesquioxanes by hydrosilylation of alkynes

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1. Materials and methods

Figure 1. Hydrosilylation of alkynes with POSS 1 monitored by in situ FT-IR spectroscopy.

1,3,5,7,9,11,14-heptaisobutyltricyclo[7.3.3.1^{5,11}]-heptasiloxane-endo-3,7,14-triol - TriSilanolIsobutyl POSS® ((*i*-Bu)$_7$Si$_7$O$_9$(OH)$_3$, Hybrid Plastics), chlorodimethylsilane (98%, Sigma-Aldrich), Silsesquioxane **1** was synthesized according to previously reported methods.[1] 1-heptyne (98%, Sigma-Aldrich), 1-bromo-4-ethynylbenzene (97%, Sigma-Aldrich), trimethyl-silylacetylene (98%, Sigma-Aldrich), phenyldimethyl-silylacetylene (98%, Sigma-Aldrich), dimethylisopropyl-silylacetylene (98%, Sigma-Aldrich), triethylsilylacetylene (97%, Sigma-Aldrich), tri(isopropyl)silylacetylene (97%, Sigma-Aldrich), triphenylsilylacetylene (98%, Sigma-Aldrich), 1,2-diphenylacetylene (98%, Sigma-Aldrich), 4,4′-(Acetylene-1,2-diyl)bis(phenylboronic acid pinacol ester) (95%, Sigma-Aldrich), bis(4-bromophenyl)acetylene (98%, Abcr), 4-octyne (98%, Sigma-Aldrich), 4-decyne (97%, Abcr), 1-phenyl-1-propyne (99%, Sigma-Aldrich), 1-phenyl-2-trimethylsilylacetylene (99%, Sigma-Aldrich), 4-[(trimethylsilyl)ethynyl]phenylboronic acid pinacol ester (97%, Sigma-Aldrich), platinum(0)-1,3-divinyl-1,1,3,3-tetramethyldisiloxane (Karstedt's catalyst, solution in xylene, Pt 2%, Sigma-Aldrich), petroleum ether (pure, POCh), methanol (pure, POCh), chloroform-d (99.96 atom% D, Sigma-Aldrich), calcium hydride (95%, chunks, +4 mesh, reagent grade, Sigma-Aldrich), (chunks, 4 mesh, reagent grade, 95%, Sigma-Aldrich), silica gel (high-purity grade, pore size ,60 Å, 60-100 mesh, Sigma-Aldrich) were used as received. Toluene (CHROMSOLV PLUS, for HPLC, 99.9%, Aldrich), THF (CHROMSOLV PLUS, for HPLC, ≥ 99.9%, inhibitor free, Aldrich) were used after purification by MBRAUN Solvent Purification Systems 500. Triethylamine (Et$_3$N, ≥ 99.5%, Sigma-Aldrich) was distilled from CaH$_2$ and stored in Schlenk's flask under argon atmosphere. Triethylgermane[2], tri(isopropyl)germane[3] and tri(n-butyl)germane[4] were obtained according to the previously published methods.

Real time infrared spectroscopy has been applied to monitor hydrosilylation of alkynes with $(HSiMe_2O)(i\text{-}Bu)_7Si_8O_{12}$, **1**.

In situ FT-IR measurements were performed on a Mettler-Toledo ReactIR 15 spectrometer equipped with 9.5 mm AgX DiComp (diamond) probe and a liquid nitrogen-cooled MCT detector. The spectra were taken with the resolution of 4 cm^{-1} collecting 50 scans for each spectrum at 15 s intervals. The reaction progress in the studied systems of parent compounds and catalysts was quantified by observing the rate of changes occurring with time in the area of the band at 904 cm^{-1} originating from stretching vibrations of Si-H bond.

Fourier Transform-Infrared (FT-IR) spectra were recorded on a Bruker Tensor 27 Fourier transform spectrophotometer equipped with a SPECAC Golden Gate, diamond ATR unit, with ensuring a resolution of 2cm^{-1}.

NMR spectra were recorded at 25^oC on a Brucker Ultra Shield (300 and 400 MHz) spectrometers. $CDCl_3$ was used as solvent and for internal deuterium lock. Chemical shifts are reported in ppm with reference to the residual solvent peaks for ^1H and ^{13}C NMR and to TMS for ^{29}Si NMR.

Mass spectrometry analyses were performed by using a Synapt G2-S mass spectrometer (Waters) equipped with the electrospray ion source and quadrupole-time-of-flight mass analyzer. Mass spectrometry analyses were performed using Synapt G2-S mass spectrometer (Waters) equipped with an Electrospray ion source and quadrupole-Time-of-flight mass analyzer. Methanol was used as a solvent. The measurements were performed in positive ion mode with the desolvation gas flow 600 L/h and capillary voltage set to 4500 V at the flow rate 100µl/min.

Elemental analyses were performed using a Vario EL III instrument.

2. Analytical data for obtained products

(1)

Isolated yield = 82% (8.77 g), white solid.

^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.23 (s, 6H, $OSiC\underline{H}_3$), 0.61 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.97 (m, 42H, $SiCH_2CHC\underline{H}_3$), 1.86 (m, 7H, $SiCH_2C\underline{H}CH_3$), 4.71 (m, 1H, SiH). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): 0.25 ($OSi\underline{C}H_3$), 22.53, 22.63, 22.68, ($Si\underline{C}H_2CHCH_3$), 23.99, 24.02 ($\underline{C}H$), 25.85, 25.87 ($SiCH_2CH\underline{C}H_3$). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -3.00 (1Si, $OSiCH_3$), -66.98 (3Si, $SiCH_2CHCH_3$), -67.86 (4Si, $SiCH_2CHCH_3$), -109.06 (1Si, SiO_4). **FT-IR** (cm^{-1}): 2953, 2927, 2906, 2870, 1465, 1401, 1383, 1366, 1332, 12253, 1228, 1169, 1070, 955, 890(Si-H), 837, 770, 739, 692,627, 560, 529, 472, 431. **Elemental Anal.** for $C_{30}H_{70}O_{13}Si_9$ (%): calcd.: C, 40.41; H, 7.91; found: C, 40.45; H, 8.00.

(3-4a)

Isolated yield = 92% (0.102 g), white solid.

^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.16, 0.20 (s, 6H, $OSiC\underline{H}_3$, isomers 3a and 4a), 0.61 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.92 (m, 3H, $CH_2CH_2CH_2CH_2C\underline{H}_3$), 0.96 (d, 42H, $SiCH_2CHC\underline{H}_3$), 1.17 (m, 6H, $CH_2C\underline{H}_2C\underline{H}_2C\underline{H}_2CH_3$), 1.87 (m, 7H, $SiCH_2C\underline{H}CH_3$), 2.10 (m, 2H, $C\underline{H}_2CH_2CH_2CH_2CH_3$), 5.41 (d, 1H, $\underline{H}_2C{=}C(Si(CH_3)_2O)$, isomer 4a), 5.58 (d, 1H, $\underline{H}_2C{=}C(Si(CH_3)_2O)$, isomer 4a), 5.61 (d, 1H, *J* (H,H) = 18.87 Hz, $Si(H)C{=}C(\underline{H})(Si(CH_3)_2O)$, isomer 3a), 6.16 (m, 1H, $(\underline{H})C{=}C(H)(Si(CH_3)_2O)$, isomer 3a). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): 0.07, 0.34 ($OSi\underline{C}H_3$, isomers 3a and 4a), 14.21, 14.28 ($CH_2CH_2CH_2CH_2\underline{C}H_3$),22.58, 22.63, 22.71 ($Si\underline{C}H_2CHCH_3$), 24.02 ($\underline{C}H$), 25.86 ($SiCH_2CH\underline{C}H_3$), 28.37, 28.75, 31.67, 31.97 ($CH_2\underline{C}H_2\underline{C}H_2\underline{C}H_2CH_3$, isomers 3a and 4a), 35.48, 36.68 ($\underline{C}H_2CH_2CH_2CH_2CH_3$, isomers 3a and 4a), 124.76 ($H_2C{=}\underline{C}(Si(CH_3)_2O$, isomer 4a), 128.12 ($H_2\underline{C}{=}C(Si(CH_3)_2O$, isomer 4a), 148.95 ($Si(H)C{=}\underline{C}(H)(Si(CH_3)_2O)$, isomer 3a), 151.49 ($Si(H)\underline{C}{=}C(H)(Si(CH_3)_2O)$, isomer 3a). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): 0.19, -0.85 (1Si, $OSiCH_3$, isomers 3a and 4a), -67.06 (3Si, $SiCH_2CHCH_3$, isomers 3a and 4a), -67.87, -67.88 (4Si, $SiCH_2CHCH_3$, isomers 3a and 4a), -109.61, -109.86 (1Si, SiO_4, isomer 3a and 4a). **FT-IR** (cm^{-1}): 2953, 2926, 2907, 2870, 1620, 1617, 1496, 1465, 1401, 1383, 1366, 1332, 1229, 1169, 1089, 1074, 953, 922, 836, 740, 728, 694, 564, 476, 432. **Elemental Anal.** for $C_{37}H_{82}O_{13}Si_9$ (%): calcd.: C, 44.99; H, 8.37; found: C, 44.83; H, 8.50.

(3-5 b)

Isolated yield = 90% (0.108 g), white solid.

^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.27, 0.28 (s, 6H, $OSiC\underline{H}_3$, isomer 3b and 4b), 0.61 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.96 (m, 42H, $SiCH_2CHC\underline{H}_3$), 1.87 (m, 7H, $SiCH_2C\underline{H}CH_3$), 5.74 (d, 1H, *J* (H,H) = 2.58

Hz, $\underline{H}_2C=C(Si(CH_3)_2O)$, isomer 4b), 5.89 (d, 1H, *J* (H,H) = 2.56 Hz, $\underline{H}_2C=C(Si(CH_3)_2O)$, isomer 4b), 6.41 (d, 1H, *J* (H,H) = 19.19 Hz, $Si(H)C=C(\underline{H})(Si(CH_3)_2O)$, isomer 3b), 6.91 (d, 1H, *J* (H,H) = 19.22 Hz, $(\underline{H})C=C(H)(Si(CH_3)_2O)$, isomer 3b), 6.98 (d, 1H, *J* (H,H) = 8.37 Hz, $Si(H)C=C(\underline{H})(Si(CH_3)_2O)$ isomer 5b) 7.16-7.47 (dd, 4H, C_6H_4Br, isomer 3b and 4b). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): 0.22, 0.31 ($OSi\underline{C}H_3$, isomer 3b and 4b), 22.51, 22.60, 22.64, ($Si\underline{C}H_2CHCH_3$), 24.02, ($\underline{C}H$), 25.85 ($SiCH_2CH\underline{C}H_3$), 120.68($H_2C=\underline{C}(Si(CH_3)_2O$, isomer 4b), 122.20 ($H_2\underline{C}=C(Si(CH_3)_2O$, isomer 4b), 128.22, 128.56, 128.63, 128.76, 137.22, 142.44 ($\underline{C}_6H_4Br$, isomer 3b and 4b), 143.46 ($Si(H)C=\underline{C}(H)(Si(CH_3)_2O)$, isomer 3b), 150.58 ($Si(H)\underline{C}=C(H)(Si(CH_3)_2O)$, isomer 3b). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): 0.11, -0.61 (1Si, $OSiCH_3$, isomer 3b and 4b), -66.99 (3Si, $SiCH_2CHCH_3$, isomer 3b and 4b), -67.85, -67.87 (4Si, $SiCH_2CHCH_3$, isomer 3b and 4b), -109.44, -109.75 (1Si, SiO_4, isomer 3b and 4b). **FT-IR** (cm^{-1}): 2953, 2925, 2906, 2869, 1607, 1590, 1487, 1464, 1400, 1383, 1366, 1332, 1253, 1229, 1169, 1069, 1010, 955, 838, 788, 740, 624, 561, 479, 433. **Elemental Anal.** for $C_{38}H_{75}BrO_{13}Si_9$ (%): calcd.: C, 42.55; H, 7.05; found: C, 42.43; H, 7.12.

(3c)

Isolated yield = 96% (0.106), white solid.

^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.07 (s, 9H, $SiC\underline{H}_3$), 0.18 (s, 6H, $OSiC\underline{H}_3$), 0.61 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.96 (m, 42H, $SiCH_2CHC\underline{H}_3$), 1.86 (m, 7H, $SiCH_2C\underline{H}CH_3$), 6.53 (d, 1H, *J* (H,H) = 22.7 Hz, $Si(H)C=C(\underline{H})(Si(CH_3)_2O)$), 6.70 (d, 1H, *J* (H,H) = 22.7 Hz, $Si(\underline{H})C=C(H)(Si(CH_3)_2O)$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): -1.50 ($Si\underline{C}H_3$), -0.06 ($OSi\underline{C}H_3$), 22.53, 22.58, 22.63, 22.68, 22.70 ($Si\underline{C}H_2CHCH_3$), 23.98, 24.03, 24.04 ($\underline{C}H$), 25.87 ($SiCH_2CH\underline{C}H_3$), 148.58 ($Si(H)C=\underline{C}(H)(Si(CH_3)_2O)$), 152.06 ($Si(H)\underline{C}=C(H)(Si(CH_3)_2O)$). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -2.06 (1Si, $OSiCH_3$), -7.31 (1Si, $SiCH_3$), -67.05 (3Si, $SiCH_2CHCH_3$), -67.87 (4Si, $SiCH_2CHCH_3$), -109.72 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 522.13, 889.26, 1011.32(100), 1012.32, 1013.32, 1014.32, 1015.32. **FT-IR** (cm^{-1}): 2953, 2907, 2873, 1464, 1380, 1366, 1332, 1251, 1229, 1169, 1084, 1011, 957, 904, 835, 798, 754, 563, 475, 432. **Elemental Anal.** for $C_{35}H_{80}O_{13}Si_{10}$ (%): calcd.: C, 42.47; H, 8.15; found: C, 42.45; H, 8.20.

(3d)

Isolated yield = 93% (0.106 g), white solid.

^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.03 (s, 6H, $SiC\underline{H}_3$), 0.18 (s, 6H, $OSiC\underline{H}_3$), 0.61 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.80 (m, 1H, $SiC\underline{H}(CH_3)$, 0.96 (m, 48H, $SiCH_2CHC\underline{H}_3$), 1.86 (m, 7H, $SiCH_2C\underline{H}CH_3$), 6.55 (d, 1H, *J* (H,H) = 22.8 Hz, $Si(H)C=C(\underline{H})(Si(CH_3)_2O)$), 6.68 (d, 1H, *J* (H,H) = 22.8 Hz, $Si(\underline{H})C=C(H)(Si(CH_3)_2O)$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): -5.53 ($Si(\underline{C}H_3)CHCH_3$), -0.01 ($OSi\underline{C}H_3$), 13.56 ($Si(CH_3)CH\underline{C}H_3$), 17.75 ($Si\underline{C}HCH_3$), 22.54, 22.60, 22.64 ($Si\underline{C}H_2CHCH_3$), 24.03 ($\underline{C}H$), 25.88 ($SiCH_2CH\underline{C}H_3$), 149.90 ($Si(H)C=\underline{C}(H)(Si(CH_3)_2O)$), 149.94 ($Si(H)\underline{C}=C(H)(Si(CH_3)_2O)$). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -2.32 (1Si, $OSiCH_3$), -2.56 (1Si, $SiCHCH_3$), -67.03 (3Si, $SiCH_2CHCH_3$), -67.84 (3Si, $SiCH_2CHCH_3$), -67.87 (1Si, $SiCH_2CHCH_3$), -109.52 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 889.26, 1034.39, 1039.35(100), 1040.35, 1041.35, 1042.35. **FT-IR** (cm^{-1}): 2953, 2928, 2906, 2868, 1464, 1401, 1383, 1366, 1332, 1251, 1229, 1169, 1070, 1012, 955, 832, 775, 739, 698, 561, 475, 432. **Elemental Anal.** for $C_{37}H_{84}O_{13}Si_{10}$ (%): calcd.: C, 43.66; H, 8.32; found: C, 44.00; H, 8.50.

(3e)

Isolated yield = 89% (0.105 g), white solid.

^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.22 (s, 6H, $OSiC\underline{H}_3$), 0.37 (s, 6H, $SiC\underline{H}_3(C_6H_5)$), 0.64 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.98 (m, 42H, $SiCH_2CHC\underline{H}_3$), 1.89 (m, 7H, $SiCH_2C\underline{H}CH_3$), 6.68 (d, 1H, *J* (H,H) = 22.7 Hz, $Si(H)C=C(\underline{H})(Si(CH_3)_2O)$), 6.84 (d, 1H, *J* (H,H) = 22.7 Hz, $Si(\underline{H})C=C(H)(Si(CH_3)_2O)$), 7.37 (m, 3H, $SiC_6\underline{H}_5$), 7.53 (m, 2H, $SiC_6\underline{H}_5$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): -2.76 ($Si\underline{C}H_3$), 0.01 ($OSi\underline{C}H_3$), 22.58, 22.65, 22.69 ($Si\underline{C}H_2CHCH_3$), 24.00, 24.04 ($SiCH_2\underline{C}HCH_3$), 25.88, 25.89 ($SiCH_2CH\underline{C}H_3$), 127.94, 129.11, 134.06, 138.65 ($Si\underline{C}_6H_5$), 149.42 ($Si(H)C=\underline{C}(H)(Si(CH_3)_2O)$), 150.85 ($Si(H)\underline{C}=C(H)(Si(CH_3)_2O)$). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -2.09 (1Si, $OSiCH_3$), -11.57 (1Si, $SiCH_3$), -66.99 (3Si, $SiCH_2CHCH_3$), -67.83 (3Si, $SiCH_2CHCH_3$), -67.86 (1Si, $SiCH_2CHCH_3$), -109.52 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 553.14, 889.26, 1073.33(100), 1074.34, 1075.33, 1076.34, 1077.33. **FT-IR** (cm^{-1}): 2953, 2928, 2906, 2870, 1464, 1428, 1401, 1383, 1366, 1332, 1250, 1229, 1169, 1074, 1012, 955, 833, 820, 780, 735, 698, 629, 561, 475, 432. **Elemental Anal.** for $C_{40}H_{82}O_{13}Si_{10}$ (%): calcd.: C, 45.67; H, 7.86; found: C, 45.60; H, 7.95.

(3f)

Isolated yield = 92% (0.106 g), white solid.

^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.18 (s, 6H, $OSiC\underline{H}_3$), 0.60 (m, 20H, $SiC\underline{H}_2CHCH_3$, $SiC\underline{H}_2CH_3$), 0.96 (m, 51H, $SiCH_2CHC\underline{H}_3$, $SiCH_2C\underline{H}_3$), 1.87 (m, 7H, $SiCH_2C\underline{H}CH_3$), 6.57 (d, 1H, *J* (H,H) = 22.9 Hz, $Si(H)C=C(\underline{H})(Si(CH_3)_2O)$), 6.64 (d, 1H, *J* (H,H) = 22.9 Hz, $Si(\underline{H})C=C(H)(Si(CH_3)_2O)$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): 0.00 ($OSi\underline{C}H_3$), 3.31 ($Si\underline{C}H_2CH_3$), 7.54 ($SiCH_2\underline{C}H_3$), 22.59, 22.66, 22.70 ($Si\underline{C}H_2CHCH_3$), 24.01, 24.06 ($SiCH_2\underline{C}HCH_3$), 25.87 ($SiCH_2CH\underline{C}H_3$), 148.44 ($Si(H)C=\underline{C}(H)(Si(CH_3)_2O)$), 150.60 ($Si(H)\underline{C}=C(H)(Si(CH_3)_2O)$). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -1.32 (1Si, $SiCH_2CH_3$), -2.44 (1Si, $OSiCH_3$), -67.05 (3Si, $SiCH_2CHCH_3$), -67.87 (3Si, $SiCH_2CHCH_3$), -67.89 (1Si, $SiCH_2CHCH_3$), -109.56 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 889.26, 1053.36 (100), 1054.37, 1055,37, 1056.36, 1057.36. **FT-IR** (cm^{-1}): 2953, 2907, 2873, 1464, 1383, 1366, 1332, 1251, 1229, 1169, 1073, 1011, 957, 904, 837, 798, 734, 563, 475, 432. **Elemental Anal.** for $C_{38}H_{86}O_{13}Si_{10}$ (%): calcd.: C, 44.23; H, 8.40; found: C, 44.53; H, 8.58.

(3g)

Isolated yield = 90% (0.108 g), white solid.

^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.19 (s, 6H, $OSiC\underline{H}_3$), 0.62 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.90 (m, 3H, $SiC\underline{H}(CH_3)$, 0.97 (d, 42H, $SiCH_2CHC\underline{H}_3$), 1.06 (d, 18H, $C\underline{H}_3$), 1.87 (m, 7H, $SiCH_2C\underline{H}CH_3$), 6.60 (d, 1H, *J* (H,H) = 23.1 Hz, $Si(H)C=C(\underline{H})(Si(CH_3)_2O)$), 6.65 (d, 1H, *J* (H,H) = 23.1 Hz, $Si(\underline{H})C=C(H)(Si(CH_3)_2O)$).

^{13}C NMR (75 MHz, $CDCl_3$, δ, ppm): 0.03 ($OSi\underline{C}H_3$), 10.88 ($SiCH\underline{C}H_3$), 14.31 ($SiCH\underline{C}H_3$), 18.63 ($Si\underline{C}HCH_3$), 22.61, 22.69, 22.73, 22.87 ($Si\underline{C}H_2CHCH_3$), 24.03, 24.07, 24.08 ($SiCH_2\underline{C}HCH_3$), 25.88, 25.90 ($SiCH_2CH\underline{C}H_3$), 146.42 ($Si(H)C=\underline{C}(H)(Si(CH_3)_2O)$), 151.89 ($Si(H)\underline{C}=C(H)(Si(CH_3)_2O)$). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -1.57 (1Si, $SiCHCH_3$), -2.75 (1Si, $OSiCH_3$), -67.05 (3Si, $SiCH_2CHCH_3$), -67.88 (3Si, $SiCH_2CHCH_3$), -67.89 (1Si, $SiCH_2CHCH_3$), -109.59 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 889.26, 1091.46, 1095.41(100), 1096.41, 1097.41, 1098.41, 1099.41. **FT-IR** (cm^{-1}): 2953, 2927, 2907, 2867, 1496, 1465, 1401, 1383, 1366, 1332, 1251, 1228, 1169, 1077, 1015, 953, 919, 882, 836, 795, 739, 694, 634, 561, 476, 432. **Elemental Anal.** for $C_{41}H_{92}O_{13}Si_{10}$ (%): calcd.: C, 45.85; H, 8.63; found: C, 45.83; H, 8.55.

(3h)

Isolated yield = 90% (0.119 g), white solid.

^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.22 (s, 6H, $OSiC\underline{H}_3$), 0.59 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.90 (m, 28H, $SiCH_2CHC\underline{H}_3$), 0.96 (m, 14H, $SiCH_2CHC\underline{H}_3$), 1.85 (m, 7H, $SiCH_2C\underline{H}CH_3$), 6.74 (d, 1H, *J* (H,H) = 22.6 Hz, $Si(H)C=C(\underline{H})(Si(CH_3)_2O)$), 7.19 (d, 1H, *J* (H,H) = 22.6 Hz, $Si(\underline{H})C=C(H)(Si(CH_3)_2O)$), 7.35-7.51 (m, 15H, $SiC_6\underline{H}_5$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): 0.04 ($OSi\underline{C}H_3$), 22.45, 22.62, 22.65, 22.68, 22.78 ($Si\underline{C}H_2CHCH_3$), 23.94, 24.01 ($SiCH_2\underline{C}HCH_3$), 25.78, 25.84, 25.86 ($SiCH_2CH\underline{C}H_3$), 127.99, 129.63, 134.46, 136.18 ($Si\underline{C}_6H_5$), 144.77 ($Si(H)C=\underline{C}(H)(Si(CH_3)_2O)$), 155.38 ($Si(H)\underline{C}=C(H)(Si(CH_3)_2O)$). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -2.33 (1Si, $OSiCH_3$), -18.09 (1Si, (1Si, SiC_6H_5), -66.98 (3Si, $SiCH_2CHCH_3$), -67.88 (4Si, $SiCH_2CHCH_3$), -109.62 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 889.26, 1193.41, 1197.37, 1198.37(100), 1199.37, 1200.37. **FT-IR** (cm^{-1}): 3069, 3050, 2953, 2926, 2906, 2870, 1590, 1465, 1429, 1401, 1383, 1366, 1332, 1251, 1229, 1169, 1077, 953, 837, 797, 734, 713, 698, 561, 506, 479, 433. **Elemental Anal.** for $C_{50}H_{86}O_{13}Si_{10}$ (%): calcd.: C, 51.06; H, 7.37; found: C, 51.13; H, 7.45.

(3i)

Isolated yield = 87% (0.105 g), white solid.

^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.07 (grease), 0.17 0.18 (s, 6H, $OSiC\underline{H}_3$), 0.60 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.79 (m, 6H, $GeC\underline{H}_2$), 0.96 (m, 42H, $SiCH_2CHC\underline{H}_3$), 1.02 (m, 9H, $GeCH_2C\underline{H}_3$), 1.85 (m, 7H, $SiCH_2C\underline{H}CH_3$), 6.46 (d, 1H, *J* (H,H) = 22.3 Hz, $Ge(H)C=C(\underline{H})(Si(CH_3)_2O)$), 6.79 (d, 1H, *J* (H,H) = 22.3 Hz, $Ge(\underline{H})C=C(H)(Si(CH_3)_2O)$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): 0.06 ($OSi\underline{C}H_3$, 3i), 1.17 ($OSi\underline{C}H_3$, 4i), 4.21 ($Ge\underline{C}H_2CH_3$), 9.06 ($SiCH_2\underline{C}H_3$), 22.52, 22.56, 22.63, 22.68 ($Si\underline{C}H_2CHCH_3$), 23.97, 24.01 ($SiCH_2\underline{C}HCH_3$), 25.84 ($SiCH_2CH\underline{C}H_3$), 148.74 ($Ge(H)C=\underline{C}(H)(Si(CH_3)_2O)$), 149.46 ($Ge(H)\underline{C}=C(H)(Si(CH_3)_2O)$). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -3.16 (1Si, $OSiCH_3$, 3i), -21.96 (1Si, $OSiCH_3$, 4i)), -67.06 (3Si, $SiCH_2CHCH_3$), -67.90 (3Si, $SiCH_2CHCH_3$), -67.91 (1Si, $SiCH_2CHCH_3$), -109.58 (1Si, SiO_4).

ESI MS – m/z ([M+Na/NH_4], (%)): 889.30, 1053.36 (100), 1054.37, 1055,37, 1056.36, 1057.36. **FT-IR** (cm^{-1}): 2953, 2929, 2906, 2871, 1464, 1401, 1383, 1366, 1332, 1250, 1229, 1169, 1074, 1011, 956, 837, 793, 740, 703, 567, 573, 475, 432. **Elemental Anal.** for $C_{38}H_{86}GeO_{13}Si_9$ (%): calcd.: C, 42.40; H, 8.05; found: C, 43.33; H, 8.00.

(3j)
Isolated yield = 90% (0.113 g), white solid.
^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.19 (s, 6H, $OSiC\underline{H}_3$), 0.62 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.97 (d, 42H, $SiCH_2CHC\underline{H}_3$), 1.15 (d, 18H, $C\underline{H}_3$), 1.40 (m, 3H, $GeC\underline{H}$), 1.87 (m, 7H, $SiCH_2C\underline{H}CH_3$), 6.52 (d, 1H, *J* (H,H) = 22.6 Hz, $Ge(H)C{=}C(\underline{H})(Si(CH_3)_2O)$), 6.75 (d, 1H, *J* (H,H) = 22.6 Hz, $Ge(\underline{H})C{=}C(H)(Si(CH_3)_2O)$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): 0.10 ($OSi\underline{C}H_3$), 14.04 ($GeCH\underline{C}H_3$), 20.13 ($Ge\underline{C}HCH_3$), 22.59, 22.67, 22.71, 22.86 ($Si\underline{C}H_2CHCH_3$), 24.02, 24.05 ($SiCH_2\underline{C}HCH_3$), 25.87, 25.89 ($SiCH_2CH\underline{C}H_3$), 147.60 ($Ge(H)C{=}\underline{C}(H)(Si(CH_3)_2O)$), 150.36 ($Si(H)\underline{C}{=}C(H)(Si(CH_3)_2O)$). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -3.56 (1Si, $OSiCH_3$), -67.06 (3Si, $SiCH_2CHCH_3$), -67.88 (3Si, $SiCH_2CHCH_3$),-67.90 (1Si, $SiCH_2CHCH_3$), -109.61 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 1139.36, 1140.36, 1141.36(100), 1142.36, 1143.36.
FT-IR (cm^{-1}): 2953, 2866, 1464, 1401, 1383, 1366, 1332, 1250, 1229, 1169, 1073, 1011, 955, 920, 878, 837, 793, 740, 640, 557, 475, 432. **Elemental Anal.** for $C_{41}H_{92}GeO_{13}Si_9$ (%): calcd.: C, 44.02; H, 8.29; found: C, 44.13; H, 8.10.

(3k)
Isolated yield = 91% (0.118 g), white solid.
^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.18 (s, 6H, $OSiC\underline{H}_3$), 0.62 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.80 (m, 6H, $GeC\underline{H}_2$), 0.90 (m, 12H, $C\underline{H}_2$), 0.97 (m, 42H, $SiCH_2CHC\underline{H}_3$), 1.36 (m, 9H, $CH_2C\underline{H}_3$), 1.87 (m, 7H, $SiCH_2C\underline{H}CH_3$), 6.45 (d, 1H, *J* (H,H) = 22.3 Hz, $Ge(H)C{=}C(\underline{H})(Si(CH_3)_2O)$), 6.81 (d, 1H, *J* (H,H) = 22.3 Hz, $Ge(\underline{H})C{=}C(H)(Si(CH_3)_2O)$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): 0.11 ($OSi\underline{C}H_3$), 12.74 ($Ge\underline{C}H_2CH_2CH_2CH_3$), 13.95 ($Ge\underline{C}H_2CH_2CH_2CH_3$), 22.59, 22.66, 22.71 ($Si\underline{C}H_2CHCH_3$), 24.03, 24.06 ($SiCH_2\underline{C}HCH_3$), 25.89 ($SiCH_2CH\underline{C}H_3$), 26.65 ($GeCH_2\underline{C}H_2CH_2CH_3$), 27.55 ($GeCH_2CH_2\underline{C}H_2CH_3$), 148.12 ($Ge(H)C{=}\underline{C}(H)(Si(CH_3)_2O)$), 150.71 ($Ge(H)\underline{C}{=}C(H)(Si(CH_3)_2O)$). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -3.20 (1Si, $SiCH_2CH_3$), -67.04 (3Si, $SiCH_2CHCH_3$), -67.86 (1Si, $SiCH_2CHCH_3$), -67.89 (1Si, $SiCH_2CHCH_3$), -109.63 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 524.08, 889.26, 1181.41, 1182.41, 1183.41(100), 1184.41, 1185.42, 1186. **FT-IR** (cm^{-1}): 3027, 2954, 2925, 2871, 1603, 1496, 1465, 1401, 1383, 1366, 1332, 1251, 1228, 1168, 1073, 1008, 953, 836, 793, 740, 727, 694, 560, 476, 432. **Elemental Anal.** for $C_{44}H_{98}GeO_{13}Si_9$ (%): calcd.: C, 45.53; H, 8.51; found: C, 45.63; H, 8.50.

(3l)
Isolated yield = 95% (0.114 g), white solid.
^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.27 (s, 6H, $OSiC\underline{H}_3$), 0.68 (m, 14H, $SiC\underline{H}_2CHCH_3$), 1.01 (m, 42H, $SiCH_2CHC\underline{H}_3$), 1.94 (m, 7H, $SiCH_2C\underline{H}CH_3$), 7.04-7.35 (s, 1H, $=C\underline{H}$, m, 10H, $C_6\underline{H}_5$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): -0.43, -0.40 ($OSi\underline{C}H_3$), 22.63, 22.69 ($Si\underline{C}H_2CHCH_3$), 24.05, 24.09 ($SiCH_2\underline{C}HCH_3$), 25.91 ($SiCH_2CH\underline{C}H_3$), 126.01, 127.36, 128.01, 128.81, 129.89, 137.49, 138.58 ($=C(Si)\underline{C}_6H_5$, $C(H)\underline{C}_6H_5$), 141.84 ($(H)C{=}\underline{C}(Si(CH_3)_2O)$), 145.07 ($(H)\underline{C}{=}C(Si(CH_3)_2O)$). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -1.00 (1Si, $OSiCH_3$), -66.92 (3Si, $SiCH_2CHCH_3$), -67.81 (4Si, $SiCH_2CHCH_3$), -109.64 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)):889.26, 890.27, 891.27, 1091.34, 1092.34, 1093.34.
FT-IR (cm^{-1}): 2953, 2927, 2906, 2870, 1610, 1465, 1399, 1359, 1333, 1260, 1229, 1168, 1084, 1021, 962, 890, 859, 836, 803, 739, 694, 656, 560, 475, 432. **Elemental Anal.** for $C_{44}H_{80}O_{13}Si_9$ (%): calcd.: C, 49.40; H, 7.54; found: C, 49.53; H, 7.50.

(3m)
Isolated yield = 93% (0.128 g), white solid.
^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.27 (s, 6H, $OSiC\underline{H}_3$), 0.67 (m, 14H, $SiC\underline{H}_2CHCH_3$), 1.01 (m, 42H, $SiCH_2CHC\underline{H}_3$), 1.91 (m, 7H, $SiCH_2C\underline{H}CH_3$), 6.89 (d, 2H, *J* (H,H) = 8.5 Hz, $C_6\underline{H}_4Br$), 6.95 (s, 1H, $=C\underline{H}$), 6.97 (d, 2H, *J* (H,H) = 8.4 Hz, $C_6\underline{H}_4Br$), 7.29 (d, 2H, *J* (H,H) = 8.4 Hz, $C_6\underline{H}_4Br$), 7.46 (d, 2H, *J* (H,H) = 8.4 Hz, $C_6\underline{H}_4Br$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): -0.52 ($OSi\underline{C}H_3$), 22.57, 22.61, 22.65 ($Si\underline{C}H_2CHCH_3$), 24.06 ($SiCH_2\underline{C}HCH_3$), 25.90 ($SiCH_2CH\underline{C}H_3$), 120.26, 121.61, 129.66, 131. 27, 131.32, 132.10, 135.84, 137.80 ($=C(Si)\underline{C}_6H_4Br$, $=C(H)\underline{C}_6H_4Br$), 140.29 ($(H)C{=}\underline{C}(Si(CH_3)_2O)$), 144.92

$((H)\underline{C}=C(Si(CH_3)_2O))$. **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -1.01 (1Si, $OSiCH_3$), -66.95 (3Si, $SiCH_2CHCH_3$), -67.80 (3Si, $SiCH_2CHCH_3$), -67.83 (1Si, $SiCH_2CHCH_3$), -109.63 (1Si, SiO_4).
ESI MS – m/z ([M+Na], (%)): 889.30, 921.30, 1014.30, 1244.20, 1245.30. **FT-IR** (cm^{-1}): 2953, 2926, 2906, 2869, 1603, 1585, 1485, 1465, 1401, 1383, 1366, 1332, 1253, 1229, 1169, 1081, 1011, 964, 935, 903, 837, 784, 740, 614, 565, 478, 433. **Elemental Anal.** for $C_{44}H_{78}Br_2O_{13}Si_9$ (%): calcd.: C, 43.05; H, 6.40; found: C, 42.93; H, 6.35.

(3n)
Isolated yield = 89% (0.132 g), white solid.
^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.22 (s, 6H, $OSiC\underline{H}_3$), 0.63 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.97 (m, 42H, $SiCH_2CHC\underline{H}_3$), 1.31 (s, 12H, $OCC\underline{H}_3$), 1.37 (s, 12H, $OCC\underline{H}_3$), 1.87 (m, 7H, $SiCH_2C\underline{H}CH_3$), 6.97 (d, 2H, *J* (H,H) = 8.0 Hz, $C_6\underline{H}_4B$), 7.00 (s, 1H, $=C\underline{H}$), 7.06 (d, 2H, *J* (H,H) = 8.0 Hz, $C_6\underline{H}_4B$), 7.53 (d, 2H, *J* (H,H) = 8.0 Hz, $C_6\underline{H}_4B$), 7.72 (d, 2H, *J* (H,H) = 8.0 Hz, $C_6\underline{H}_4B$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): -0.46 ($OSi\underline{C}H_3$), 22.55, 22.62, 22.68, 22.78 ($Si\underline{C}H_2CHCH_3$), 23.98, 24.02 ($SiCH_2\underline{C}HCH_3$), 25.00, 25.10 ($OC\underline{C}H_3$), 25.85, 25.87 ($SiCH_2CH\underline{C}H_3$), 83.78 ($O\underline{C}CH_3$), 127.28, 129.06, 134.44, 135.22, 138.52, 139.97 ($=C(Si)\underline{C}_6H_4B$, $=C(H)\underline{C}_6H_4B$), 146.07 ($((H)C=\underline{C}(Si(CH_3)_2O))$), 146.30 ($((H)\underline{C}=C(Si(CH_3)_2O))$). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -1.11 (1Si, $OSiCH_3$), -66.93 (3Si, $SiCH_2CHCH_3$), -67.84 (3Si, $SiCH_2CHCH_3$), -67.88 (1Si, $SiCH_2CHCH_3$), -109.63 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 889.30, 1342.51, 1343.51, 1344.51, 1345.52, 1346.51. **FT-IR** (cm^{-1}): 2953, 2927, 2906, 2870, 1608, 1465, 1399, 1359, 1333, 1260, 1229, 1168, 1084, 1021, 962, 890, 859, 836, 803, 739, 694, 656, 560, 475, 432. **Elemental Anal.** for $C_{56}H_{102}B_2O_{17}Si_9$ (%): calcd.: C, 50.89; H, 7.78; found: C, 50.83; H, 7.67.

(3o)
Isolated yield = 95% (0.107 g), white solid.
^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.17 (s, 6H, $OSiC\underline{H}_3$), 0.63 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.90 (m, 6H, $CH_2C\underline{H}_3$), 0.95 (m, 42H, $SiCH_2CHC\underline{H}_3$), 1.41 (m, 4H, $CH_2C\underline{H}_2CH_3$), 1.87 (m, 7H, $SiCH_2C\underline{H}CH_3$), 2.09 (m, 4H, $=CHC\underline{H}_2$, $=C(Si)C\underline{H}_2$), 5.84 (t, 1H, $=C\underline{H}CH_2$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): 0.30 ($OSi\underline{C}H_3$), 14.13, 14.29, 14.63 ($CH_2\underline{C}H_3$), 22.62, 22.66, 22.71 ($Si\underline{C}H_2CHCH_3$), 22.84, 23.42 ($\underline{C}H_2CH_3$), 24.00, 24.06 ($SiCH_2\underline{C}HCH_3$), 25.88 ($SiCH_2CH\underline{C}H_3$), 30.59, 31.35, 31.79 ($=CH\underline{C}H_2$, $=C(Si)\underline{C}H_2$), 140.30 ($((H)C=\underline{C}(Si(CH_3)_2O))$), 141.47 ($((H)\underline{C}=C(Si(CH_3)_2O))$). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): 0.58 (1Si, $OSiCH_3$), -67.11 (3Si, $SiCH_2CHCH_3$), -67.89 (4Si, $SiCH_2CHCH_3$), -109.89 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 889.26, 890.26, 891.26, 1023.37, 1024.37, 1025.37. **FT-IR** (cm^{-1}): 3018, 2953, 2920, 2906, 2869, 1608, 1493, 1465, 1401. 1383, 1366, 1332, 1229, 1169, 1078, 953, 904, 837, 780, 740, 561, 480, 433. **Elemental Anal.** for $C_{38}H_{84}O_{13}Si_9$ (%): calcd.: C, 45.56; H, 8.45; found: C, 45.70; H, 8.50.

(3-4p)
Isolated yield = 91% (0.105 g), white solid.
^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.17 (s, 6H, $OSiC\underline{H}_3$), 0.60 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.90 (m, 6H, $CH_2C\underline{H}_3$), 0.95 (d, 42H, $C\underline{H}_3$), 1.39 (m, 8H, $C\underline{H}_2$), 1.86 (m, 7H, $SiCH_2C\underline{H}CH_3$), 2.10 (m, 4H, $=CHC\underline{H}_2$, $=C(Si)C\underline{H}_2$), 5.83 (m, 1H, $=C\underline{H}CH_2$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): 0.29 ($OSi\underline{C}H_3$), 14.16, 14.27, 14.34, 14.66 ($CH_2\underline{C}H_3$), 22.58, 22. 63, 22.67 ($Si\underline{C}H_2CHCH_3$), 22.77, 22.79, 22.84, 23.44 ($\underline{C}H_2CH_3$), 24.01, 24.06 ($SiCH_2\underline{C}HCH_3$), 25.89 ($SiCH_2CH\underline{C}H_3$), 28.55, 29.11, 29.42, 29.98 ($\underline{C}H_2CH_2$, $\underline{C}H_2CH_2$) 30.52, 31.31, 31.95, 32.52 ($=CH\underline{C}H_2$, $=C(Si)\underline{C}H_2$), 139.94, 140.43 ($((H)C=\underline{C}(Si(CH_3)_2O))$), 141.23, 141.74 ($((H)\underline{C}=C(Si(CH_3)_2O))$). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): 0.57, 0.54 (1Si, $OSiCH_3$), -67.11 (3Si, $SiCH_2CHCH_3$), -67.89 (3Si, $SiCH_2CHCH_3$), -67.98 (1Si, $SiCH_2CHCH_3$), -109.88, -109.90 (1Si, Q^4). **ESI MS** – m/z ([M+Na], (%)): 889.26, 890.26, 891.26, 1051.41(100), 1052.40, 1053.40.
FT-IR (cm^{-1}): 2953, 2926, 2906, 2869, 1490, 1464, 1401, 1383, 1366, 1332, 1252, 1229, 1169, 1074, 959, 935, 919, 895, 836, 782, 739, 696, 568, 476, 432. **Elemental Anal.** for $C_{40}H_{88}O_{13}Si_9$ (%): calcd.: C, 46.65; H, 8.61; found: C, 46.83; H, 8.70.

(3-5q)
Isolated yield = 92% (0.104 g), white solid.
^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.31 (s, 6H, $OSiC\underline{H}_3$), 0.65 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.99 (m, 42H, $SiC\underline{H}_2CHCH_3$), 1.89 (m, 7H, $SiCH_2C\underline{H}CH_3$), 2.00 (s, 3H, CH_3), 6.88 (s, 1H, $=C\underline{H}$), 7.17-7.36 (m, 5H, $C_6\underline{H}_5$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): -0.72 ($OSi\underline{C}H_3$), 15.67, 15.69 (CH_3), 22.57, 22.63, ($Si\underline{C}H_2CHCH_3$), 24.01, 24.04 ($SiCH_2\underline{C}HCH_3$), 25.87 ($SiCH_2CH\underline{C}H_3$), 126.75, 128.14, 128.37, 129.27, 129.27, 137.12, 137.87 ($\underline{C}_6H_5$), 138.40 ($((Si)(H)C=\underline{C}(Si(CH_3)_2O))$), 138.82 ($((Si)(H)\underline{C}=C(Si(CH_3)_2O))$). **^{29}Si**

NMR (79 MHz, $CDCl_3$, δ, ppm): -1.22 (1Si, $OSiCH_3$), -66.93 (3Si, $SiCH_2CHCH_3$), -67.79 (3Si, $SiCH_2CHCH_3$), -67.81 (1Si, $SiCH_2CHCH_3$), -109.60 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 889.26, 1029.32(100), 1030.33, 1031.32, 1032.32. **FT-IR** (cm^{-1}):
2953, 2926, 2906, 2869, 1490, 1464, 1401, 1383, 1366, 1332, 1252, 1229, 1169, 1070, 959, 935, 919, 895, 836, 782, 739, 696, 568, 476, 432. **Elemental Anal.** for $C_{39}H_{78}O_{13}Si_9$ (%): calcd.: C, 46.48; H, 7.80; found: C, 46.33; H, 7.66.

(3r)
Isolated yield = 90% (0.107 g), white solid.
^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.14 (s, 9H, $SiC\underline{H}_3$), 0.19 (s, 6H, $OSiC\underline{H}_3$), 0.63 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.90 (m, 3H, $CH_2CH_2CH_2CH_2C\underline{H}_3$), 0.96 (m, 42H, $SiCH_2CHC\underline{H}_3$), 1.32 (m, 4H, $CH_2CH_2C\underline{H}_2C\underline{H}_2CH_3$), 1.40 (m, 4H, $CH_2C\underline{H}_2CH_2CH_2CH_3$), 1.87 (m, 7H, $SiCH_2C\underline{H}CH_3$), 2.29 (m, 2H, $\underline{C}H_2CH_2CH_2CH_2CH_3$), 6.07 (s, 1H, $=C\underline{H}$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): 0.63 ($Si\underline{C}H_3$), 0.67 ($OSi\underline{C}H_3$), 14.35 ($CH_2CH_2CH_2CH_2\underline{C}H_3$), 22.64, 22.68, 22.73, 22.82 ($Si\underline{C}H_2CHCH_3$, $CH_2CH_2CH_2\underline{C}H_2CH_3$), 24.03, 24.07 ($SiCH_2\underline{C}HCH_3$), 25.90, 25.92 ($SiCH_2CH\underline{C}H_3$), 30.74 ($CH_2\underline{C}H_2CH_2CH_2CH_3$), 32.73 ($CH_2CH_2\underline{C}H_2CH_2CH_3$), 36.25 ($\underline{C}H_2CH_2CH_2CH_2CH_3$), 141.82 ((Si)(H)C=$\underline{C}$($Si(CH_3)_2O$)), 164.41 ((Si)(H)$\underline{C}$=C($Si(CH_3)_2O$)). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -1.52 (1Si, $OSiCH_3$), -11.77 (1Si, $SiCH_3$), -67.09 (3Si, $SiCH_2CHCH_3$), -67.87 (3Si, $SiCH_2CHCH_3$), -67.89 (1Si, $SiCH_2CHCH_3$), -109.84 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 889.26, 1081.40(100), 1082.40, 1083.39, 1084.39, 1097.37. **FT-IR** (cm^{-1}): 2954, 2929, 2907, 2870, 1465, 1400, 1383, 1366, 1332, 1248, 1229, 1169, 1074, 1038, 956, 890, 859, 835, 803, 799, 740, 691, 561, 473, 432. **Elemental Anal.** for $C_{40}H_{90}O_{13}Si_{10}$ (%): calcd.: C, 45.32; H, 8.56; found: C, 45.53; H, 8.62.

(3s)
Isolated yield = 89% (0.112 g), white solid.
^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 0.22 (s, 6H, $OSiC\underline{H}_3$), 0.40 (s, 6H, $SiC\underline{H}_3$), 0.63 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.84 (m, 3H, $CH_2CH_2CH_2CH_2C\underline{H}_3$), 0.95 (m, 42H, $SiCH_2CHC\underline{H}_3$), 1.12 (m, 2H, $CH_2CH_2C\underline{H}_2CH_2CH_3$), 1.21 (m, 2H, $CH_2CH_2CH_2C\underline{H}_2CH_3$), 1.29 (m, 2H, $CH_2C\underline{H}_2CH_2CH_2CH_3$), 1.86 (m, 7H, $SiCH_2C\underline{H}CH_3$), 2.23 (m, 2H, $\underline{C}H_2CH_2CH_2CH_2CH_3$), 6.21 (s, 1H, $=C\underline{H}$), 7.35 (m, 3H, $SiC_6\underline{H}_5$), 7.55 (m, 2H, $SiC_6\underline{H}_5$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): -0.40 ($Si\underline{C}H_3$), 0.60 ($OSi\underline{C}H_3$), 14.27 ($CH_2CH_2CH_2CH_2\underline{C}H_3$), 22.58, 22.64, 22.69, 22.70 ($Si\underline{C}H_2CHCH_3$, $CH_2CH_2CH_2\underline{C}H_2CH_3$), 23.99, 24.04 ($SiCH_2\underline{C}HCH_3$), 25.88 ($SiCH_2CH\underline{C}H_3$), 30.36 ($CH_2\underline{C}H_2CH_2CH_2CH_3$), 32.55 ($CH_2CH_2\underline{C}H_2CH_2CH_3$), 36.59 ($\underline{C}H_2CH_2CH_2CH_2CH_3$), 127.87, 128.85, 133.95, 139.18 ($Si\underline{C}_6H_5$), 140.17 ((Si)(H)C=$\underline{C}$($Si(CH_3)_2O$)), 166.66 ((Si)(H)$\underline{C}$=C($Si(CH_3)_2O$)). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -1.13 (1Si, $OSiCH_3$), -16.24 (1Si, $SiCH_3$), -67.06 (3Si, $SiCH_2CHCH_3$), -67.86 (4Si, $SiCH_2CHCH_3$), -109.82 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 1139.46, 1143.42, 1144.41(100), 1145.41, 1146.41. **FT-IR** (cm^{-1}): 2954, 2929, 2907, 2870, 1465, 1428, 1401, 1383, 1366, 1332, 1249, 1229, 1169, 1081, 1038, 955, 920, 835, 782, 736, 699, 620, 559, 529, 475, 432. **Elemental Anal.** for $C_{45}H_{92}O_{13}Si_{10}$ (%): calcd.: C, 48.17; H, 8.26; found: C, 48.13; H, 8.20.

(3t)
Isolated yield = 92% (0.110 g), white solid.
^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): -0.17 (s, 9H, $SiC\underline{H}_3$), 0.16 (s, 6H, $OSiC\underline{H}_3$), 0.62 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.98 (m, 42H, $SiCH_2CHC\underline{H}_3$), 1.88 (m, 7H, $SiCH_2C\underline{H}CH_3$), 6.50 (s, 1H, $=C\underline{H}$), 7.01 (m, 2H, $C_6\underline{H}_5$), 7.18 (m, 1H, $C_6\underline{H}_5$), 7.25 (m, 2H, $C_6\underline{H}_5$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): -0.49 ($Si\underline{C}H_3$), 0.21 ($OSi\underline{C}H_3$), 22.64, 22.70, 22.74 ($Si\underline{C}H_2CHCH_3$), 24.04, 24.09 ($SiCH_2\underline{C}HCH_3$), 25.93 ($SiCH_2CH\underline{C}H_3$), 126.02, 127.81, 144.52 ($\underline{C}_6H_5$), 145.12 ((Si)(H)C=$\underline{C}$($Si(CH_3)_2O$)), 164.51 ((Si)(H)$\underline{C}$=C($Si(CH_3)_2O$)). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -3.46 (1Si, $OSiCH_3$), -9.63 (1Si, $SiCH_3$), -67.01 (3Si, $SiCH_2CHCH_3$), -67.83 (4Si, $SiCH_2CHCH_3$), -109.76 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 560.15, 889.26, 1087.35(100) 1088.35, 1089.35, 1090.35. **FT-IR** (cm^{-1}): 3026, 2953, 2927, 2906, 2870, 1605, 1488, 1465, 1401, 1383, 1366, 1332, 1228, 1249, 1228, 1169, 1081, , 952, 938, 836, 784, 762, 740, 701, 560, 478, 433. **Elemental Anal.** for $C_{41}H_{84}O_{13}Si_{10}$ (%): Calcd.: 46.20; H, 7.94; found: C, 46.18; H, 8.02.

(3u)
Isolated yield = 88% (0.118 g), white solid.

^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): -0.17 (s, 9H, $SiC\underline{H}_3$), 0.19 (s, 6H, $OSiC\underline{H}_3$), 0.63 (m, 14H, $SiC\underline{H}_2CHCH_3$), 0.98 (m, 42H, $SiC\underline{H}_2CHCH_3$), 1.36 (s, 12H, $OCCH_3$), 1.89 (m, 7H, $SiCH_2C\underline{H}CH_3$), 2.35(toluene), 6.50 (s, 1H, =C$\underline{H}$), 7.02 (d, 2H, *J* (H,H) = 7.8 Hz, $C_6\underline{H}_4B$), 7.17-7.24 (toluene), 7.71 (d, 2H, *J* (H,H) = 7.8 Hz, $C_6\underline{H}_4B$). **^{13}C NMR** (75 MHz, $CDCl_3$, δ, ppm): -0.52 ($Si\underline{C}H_3$), 0.28 ($OSi\underline{C}H_3$), 22.61, 22.68, 22.73 ($Si\underline{C}H_2CHCH_3$), 24.01, 24.07 ($SiCH_2\underline{C}HCH_3$), 25.15 ($OC\underline{C}H_3$), 25.91, 25.92 ($SiCH_2CH\underline{C}H_3$), 83.74 ($O\underline{C}CH_3$), 125.50, 127.18, 128.41, 129.19, 134.39, 146.01 ($\underline{C}_6H_4B$), 147.73 ((Si)(H)C=$\underline{C}$($Si(CH_3)_2O$)), 164.53 ((Si)(H)$\underline{C}$=C($Si(CH_3)_2O$)). **^{29}Si NMR** (79 MHz, $CDCl_3$, δ, ppm): -3.45 (1Si, $OSiCH_3$), -9.49 (1Si, $SiCH_3$), -67.00 (3Si, $SiCH_2CHCH_3$), -67.84 (3Si, $SiCH_2CHCH_3$), -67.87 (1Si, $SiCH_2CHCH_3$), -109.77 (1Si, SiO_4). **ESI MS** – m/z ([M+Na], (%)): 889.3, 1212.5(100), 1213.44, 1214.44, 1215.44, 1215.44, 1216.44. **FT-IR** (cm^{-1}): 2954, 2929, 2906, 2871, 1607, 1465, 1398, 1361, 1332, 1249, 1229, 1168, 1081, 1021, 962, 930, 858, 835, 801, 782, 740, 689, 659, 559, 475, 432. **Elemental Anal.** for $C_{47}H_{95}BO_{15}Si_{10}$ (%): calcd.: C, 47.36; H, 8.03; found: C, 47.32; H, 8.09.

3. Determination of hydrosilylation progress by in situ FT-IR and NMR spectra of silsesquioxane 1 and resulted products

3.1 Silsesquioxane 1

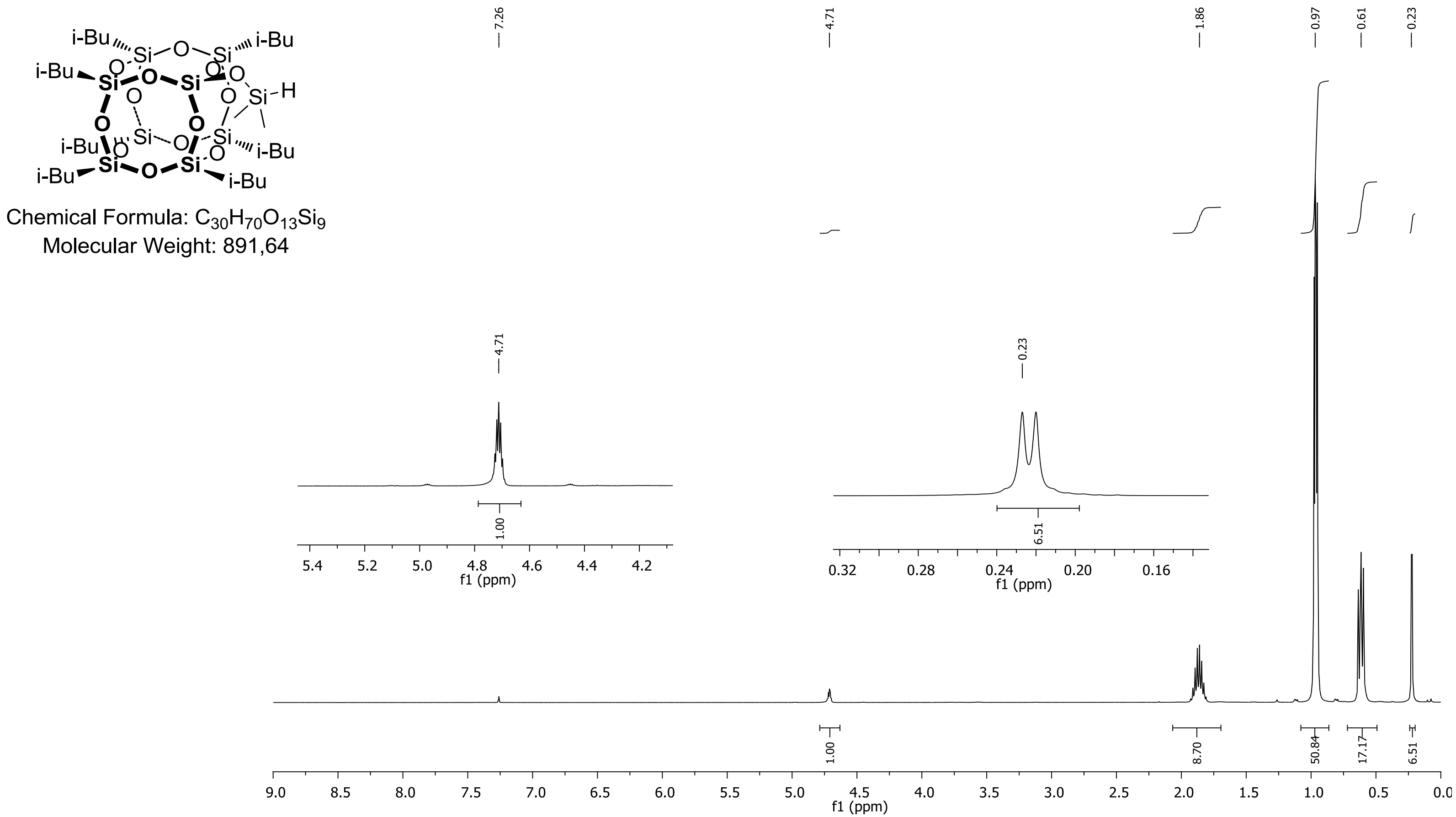


Figure 2. ^{1}H NMR of compound 1.

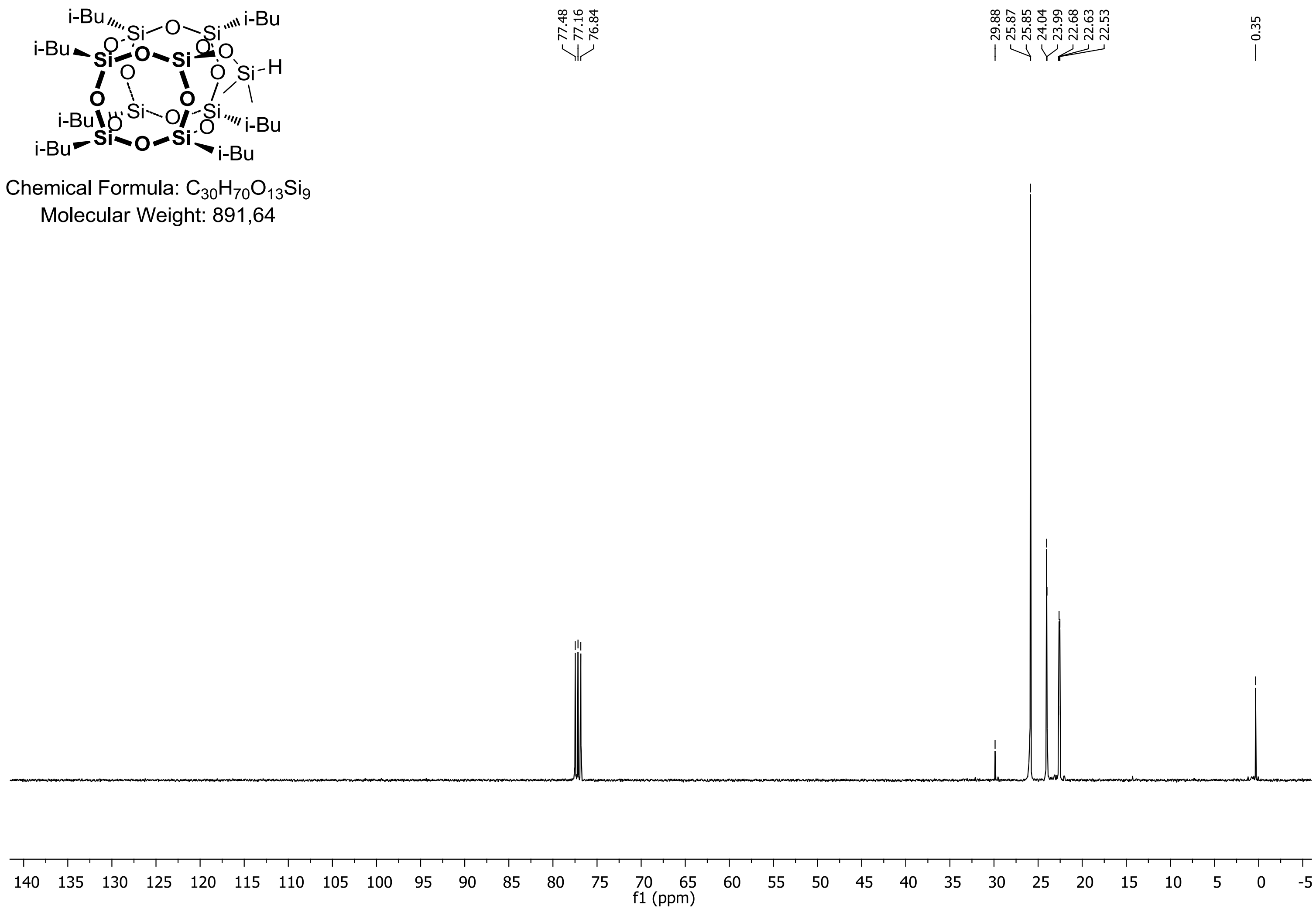


Figure 3. ^{13}C NMR of compound 1.

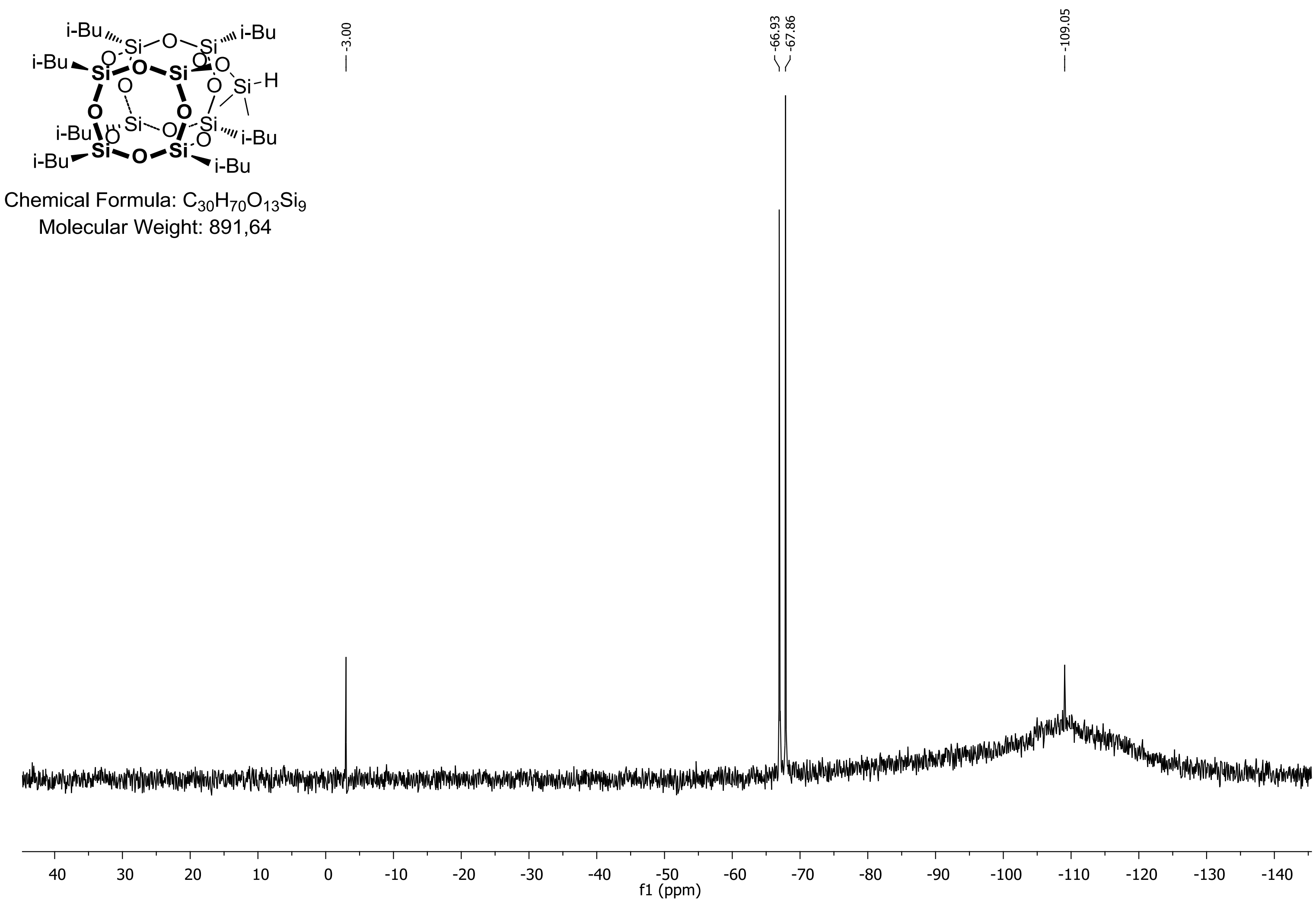


Figure 4. ^{29}Si NMR of compound 1.

3.2 Hydrosilylation of 2a with silsesquioxane 1

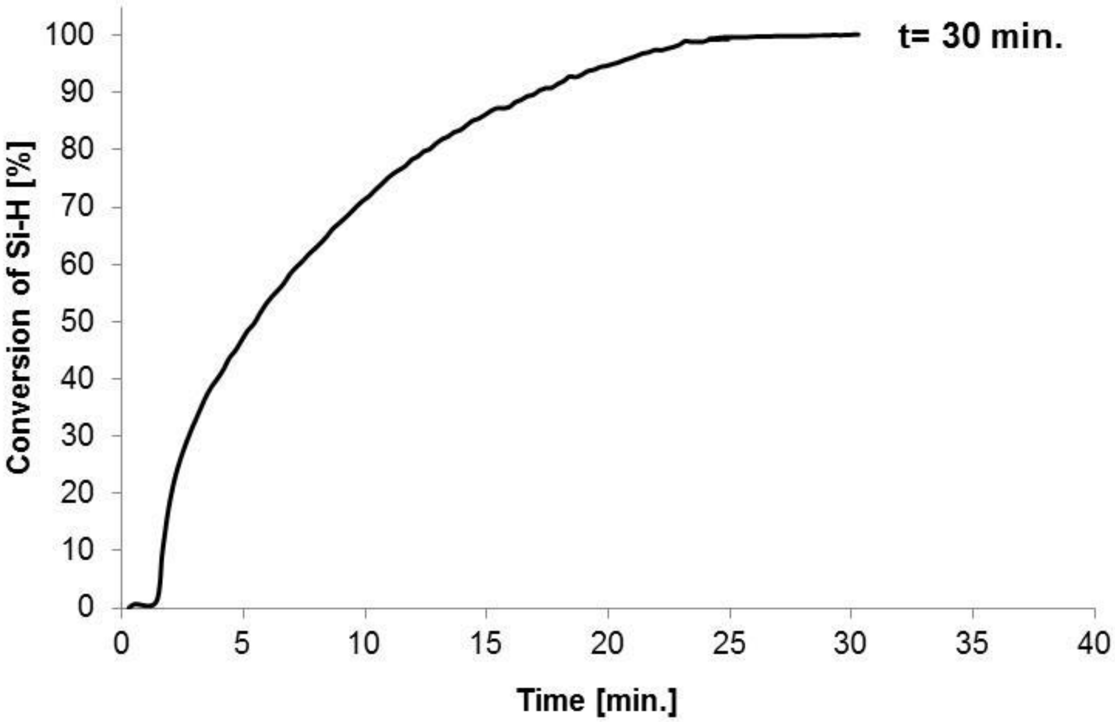
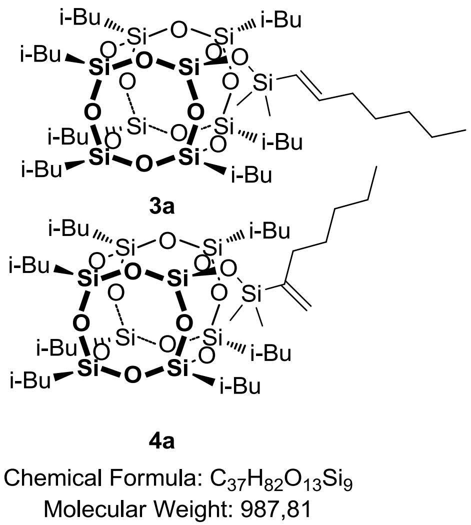


Figure 5. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2a with 1.



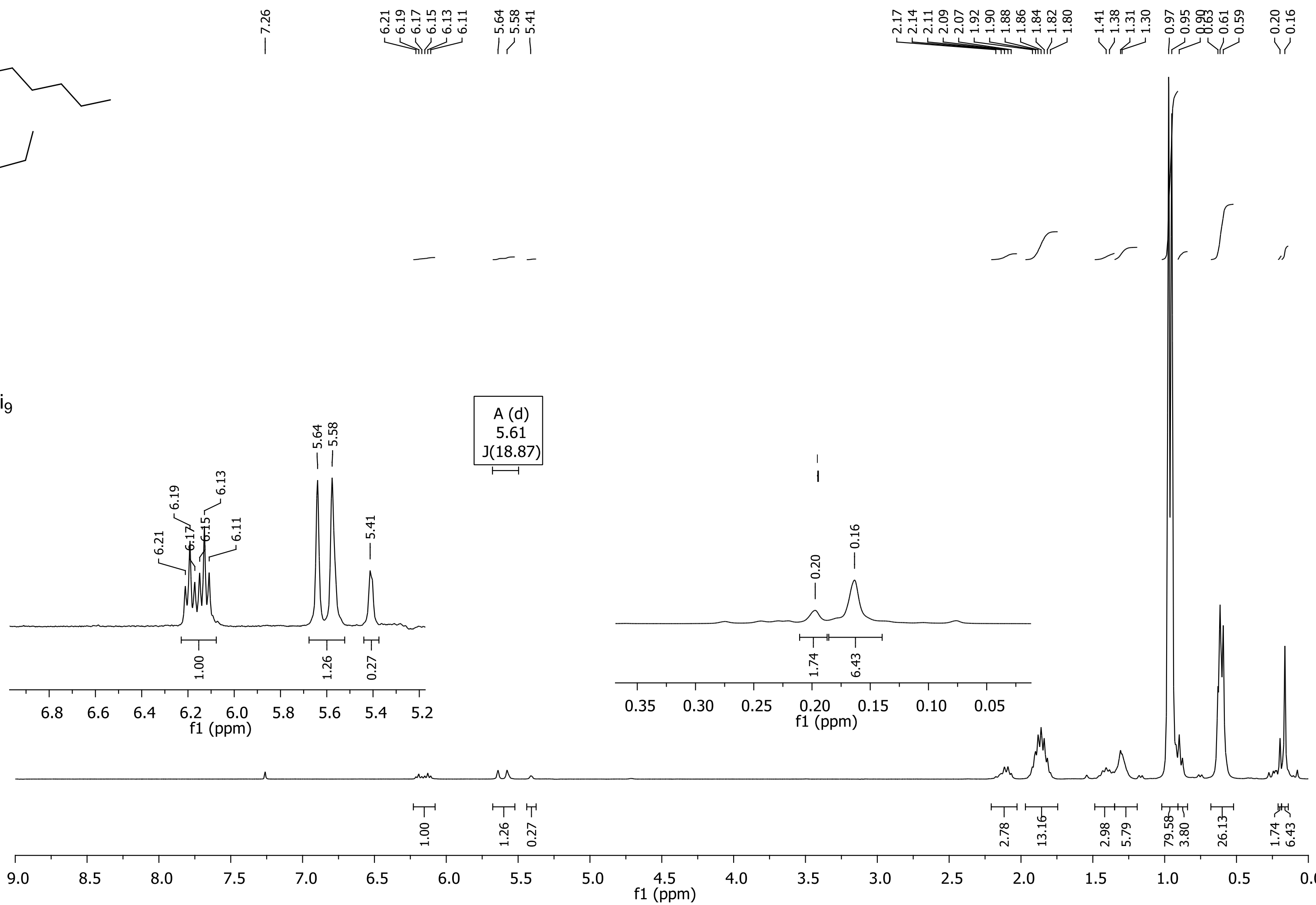
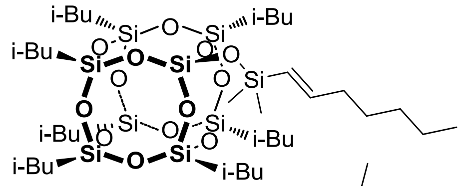
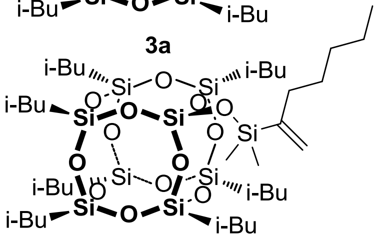


Figure 6. ^{1}H NMR of 3-4a mixture.



3a



4a

Chemical Formula: $C_{37}H_{82}O_{13}Si_9$
Molecular Weight: 987,81

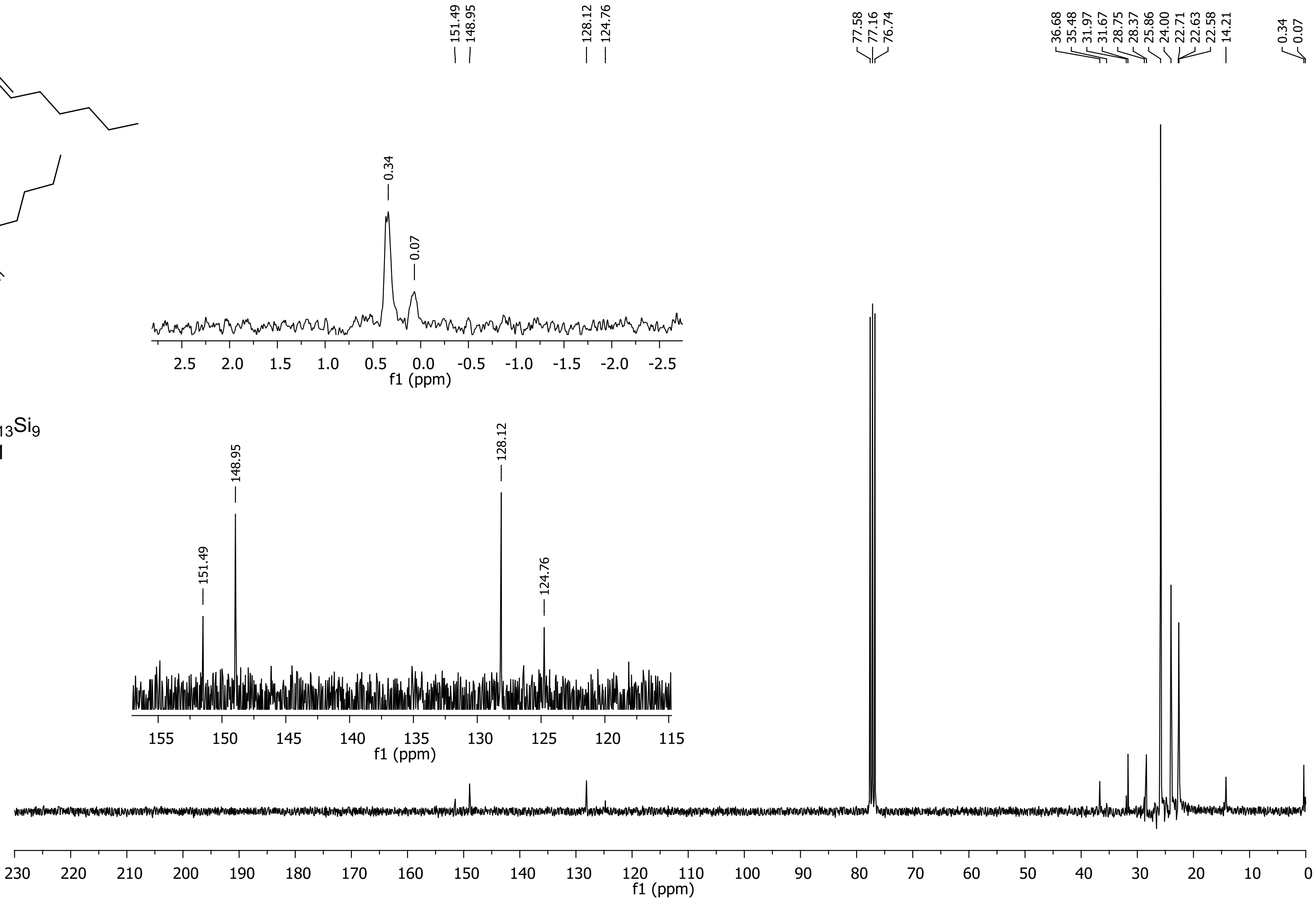
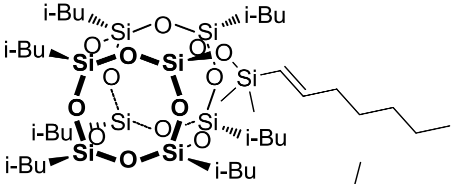
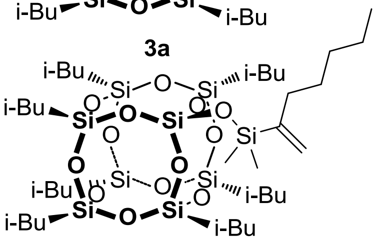


Figure 7. ^{13}C NMR of 3-4a mixture.



3a



4a

Chemical Formula: $C_{37}H_{82}O_{13}Si_9$
Molecular Weight: 987,81

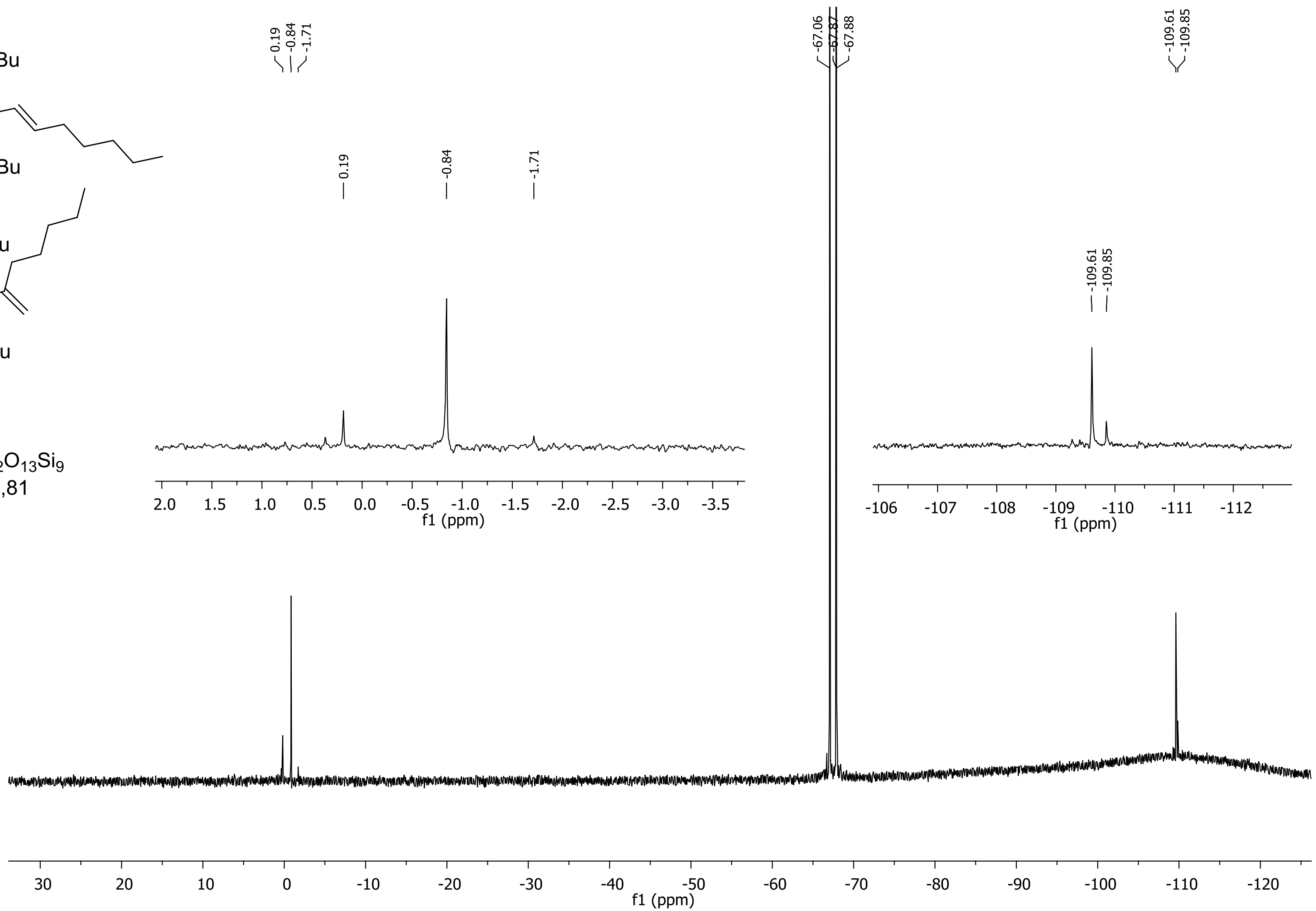


Figure 8. ^{29}Si NMR of 3-4a mixture.

3.3 Hydrosilylation of 2b with silsesquioxane 1

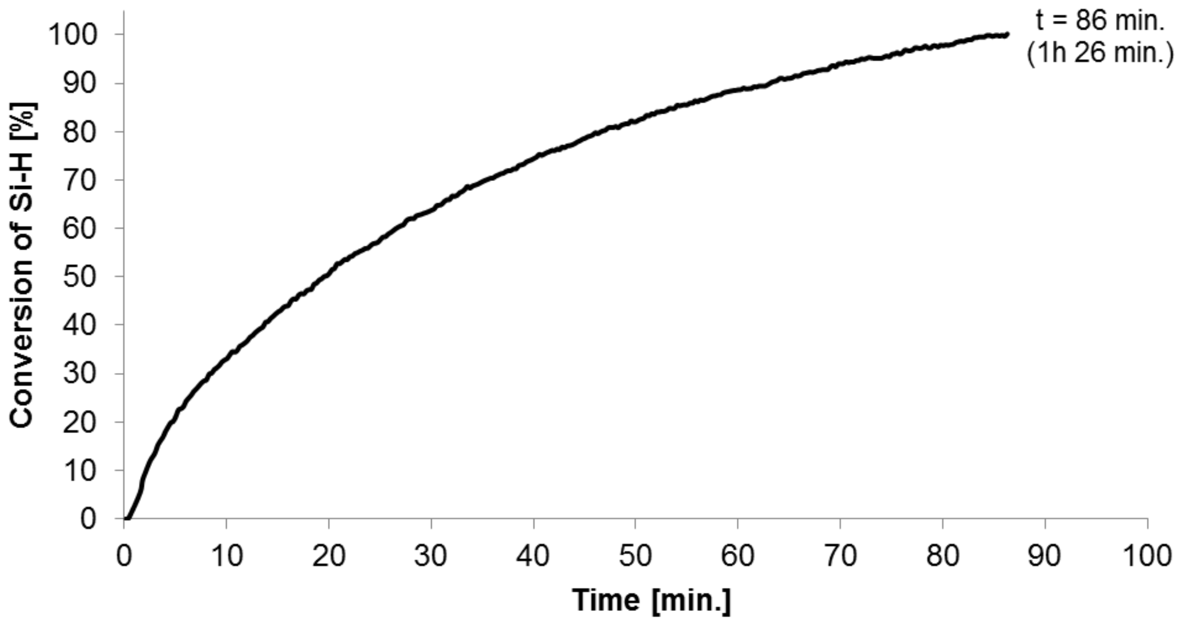


Figure 9. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2b with 1.

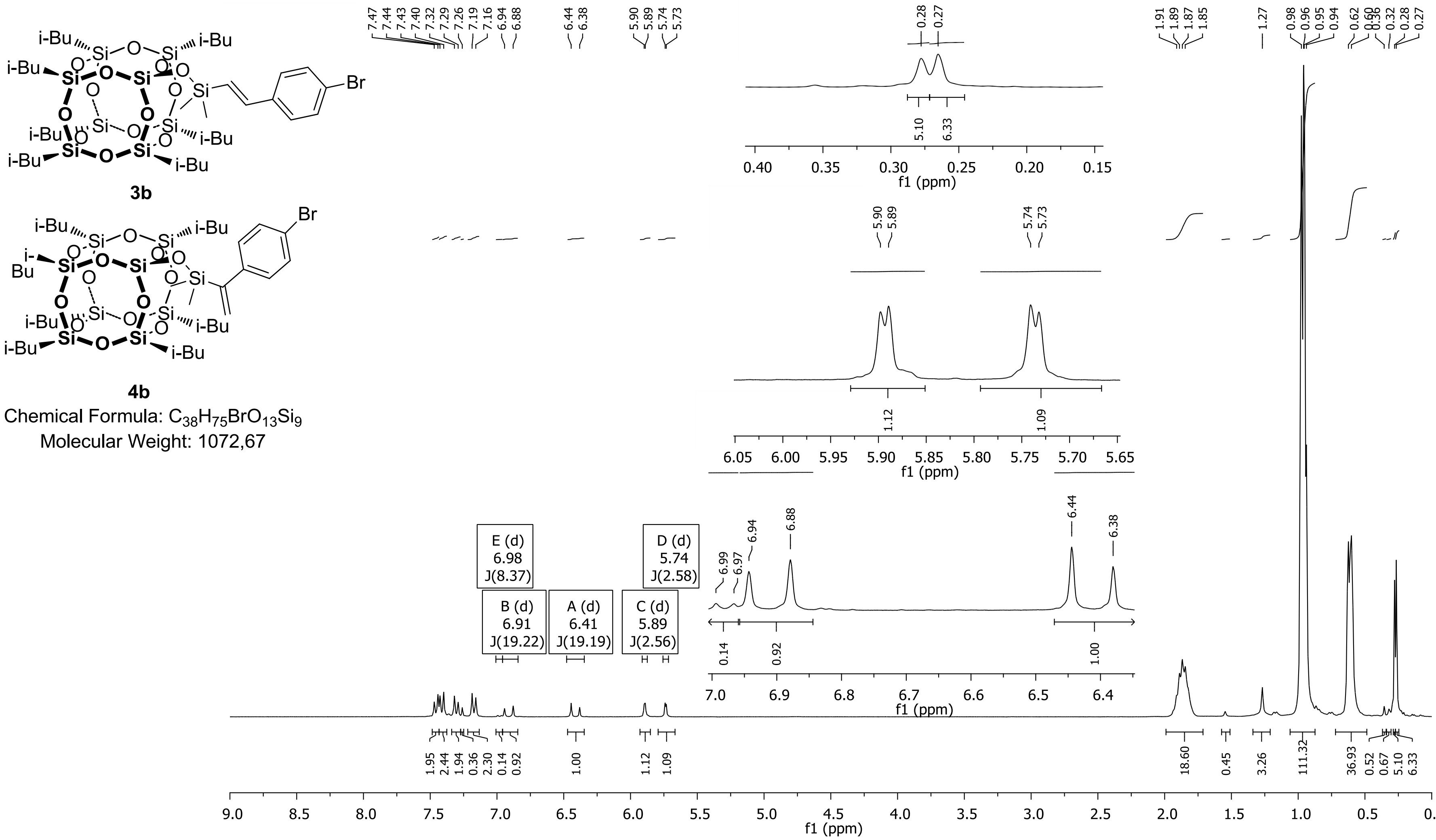
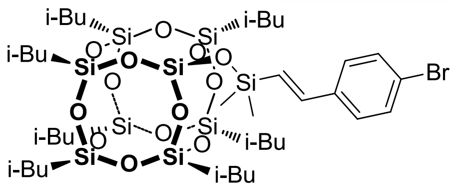
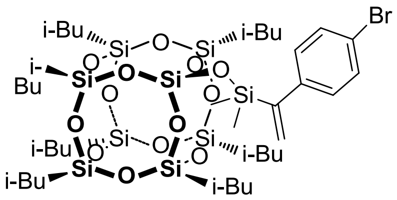


Figure 10. [1]H NMR of 3-5b mixture.



3b



4b

Chemical Formula: $C_{38}H_{75}BrO_{13}Si_9$

Molecular Weight: 1072,67

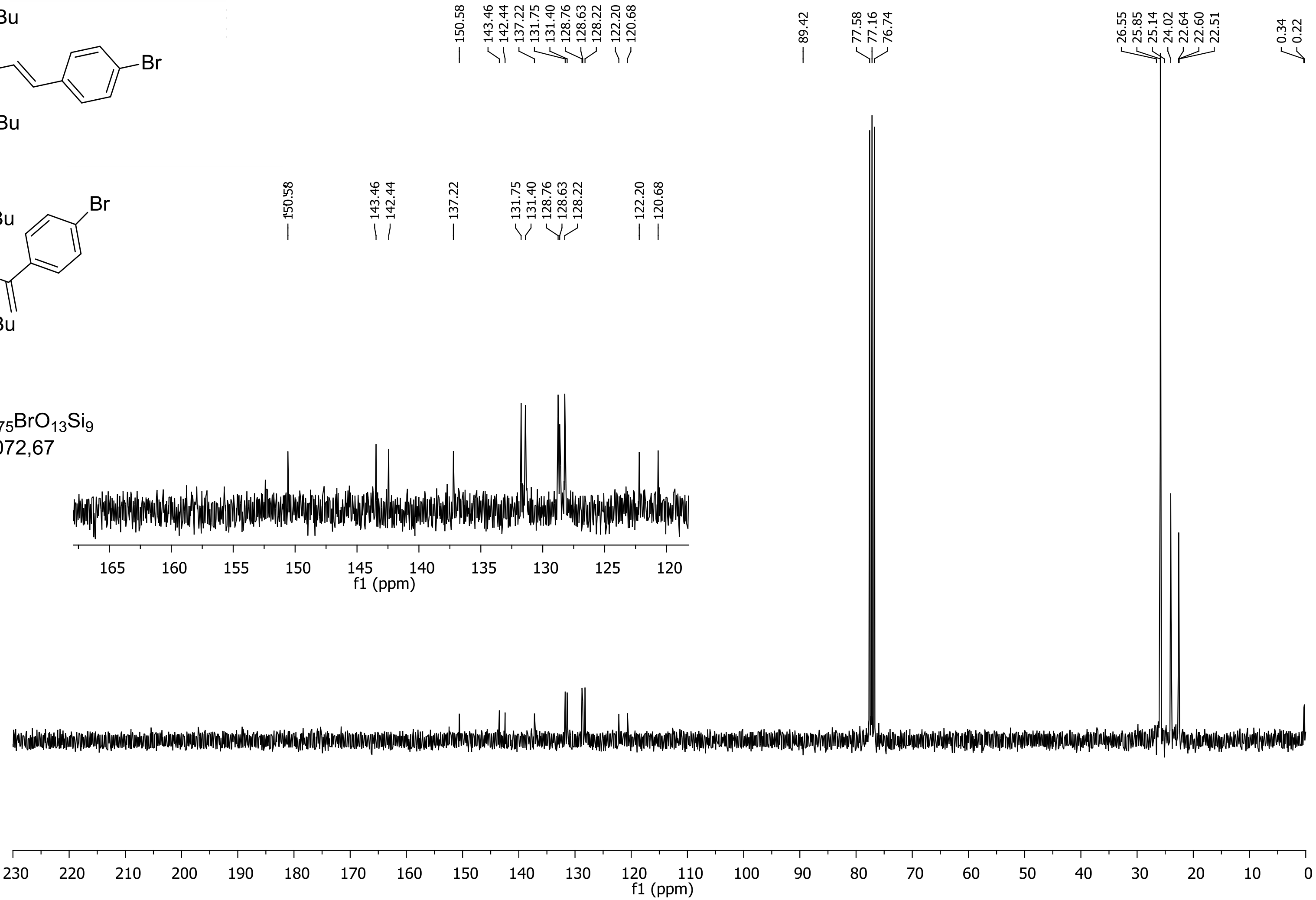


Figure 11. ^{13}C NMR of 3-5b mixture.

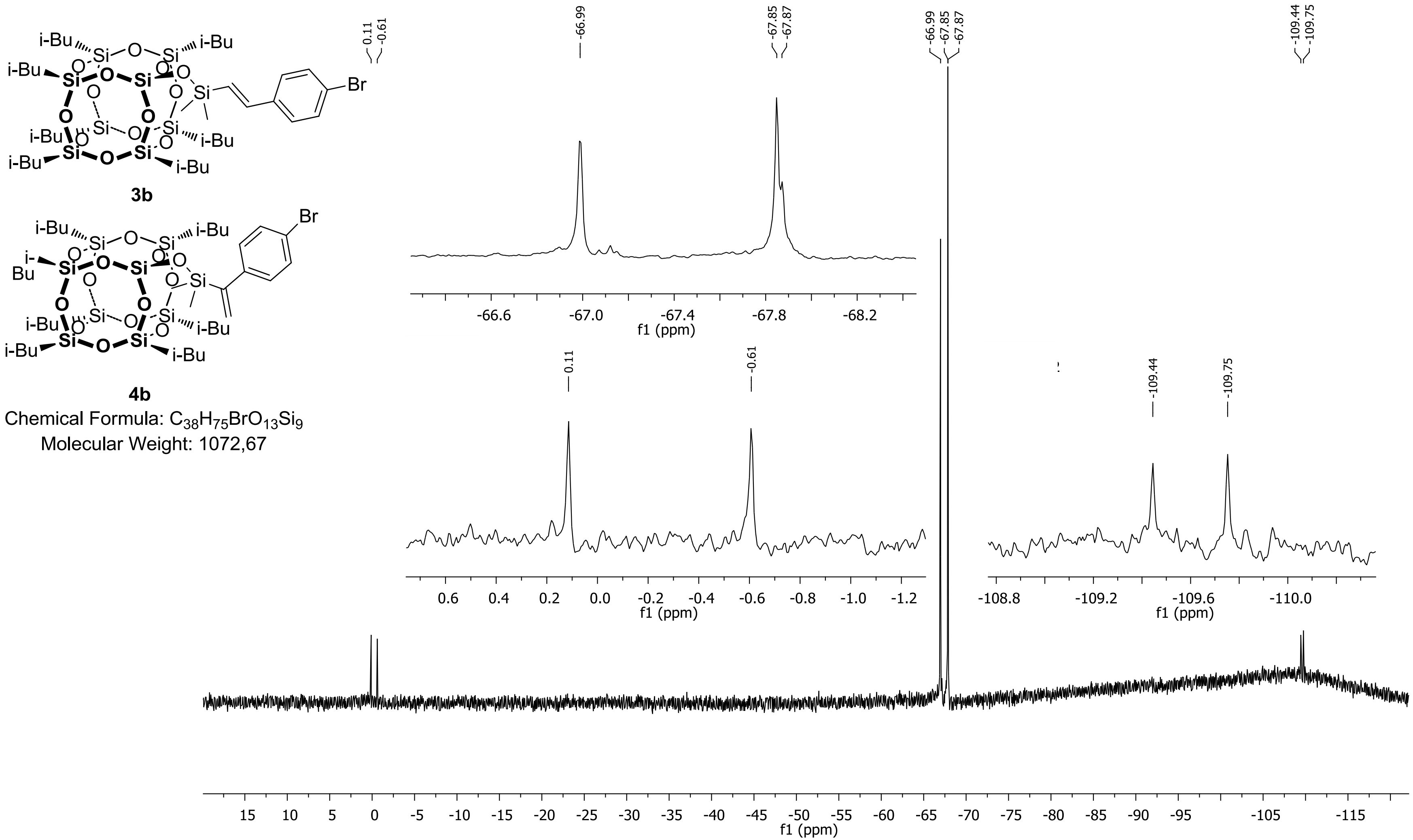
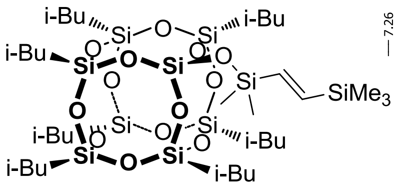


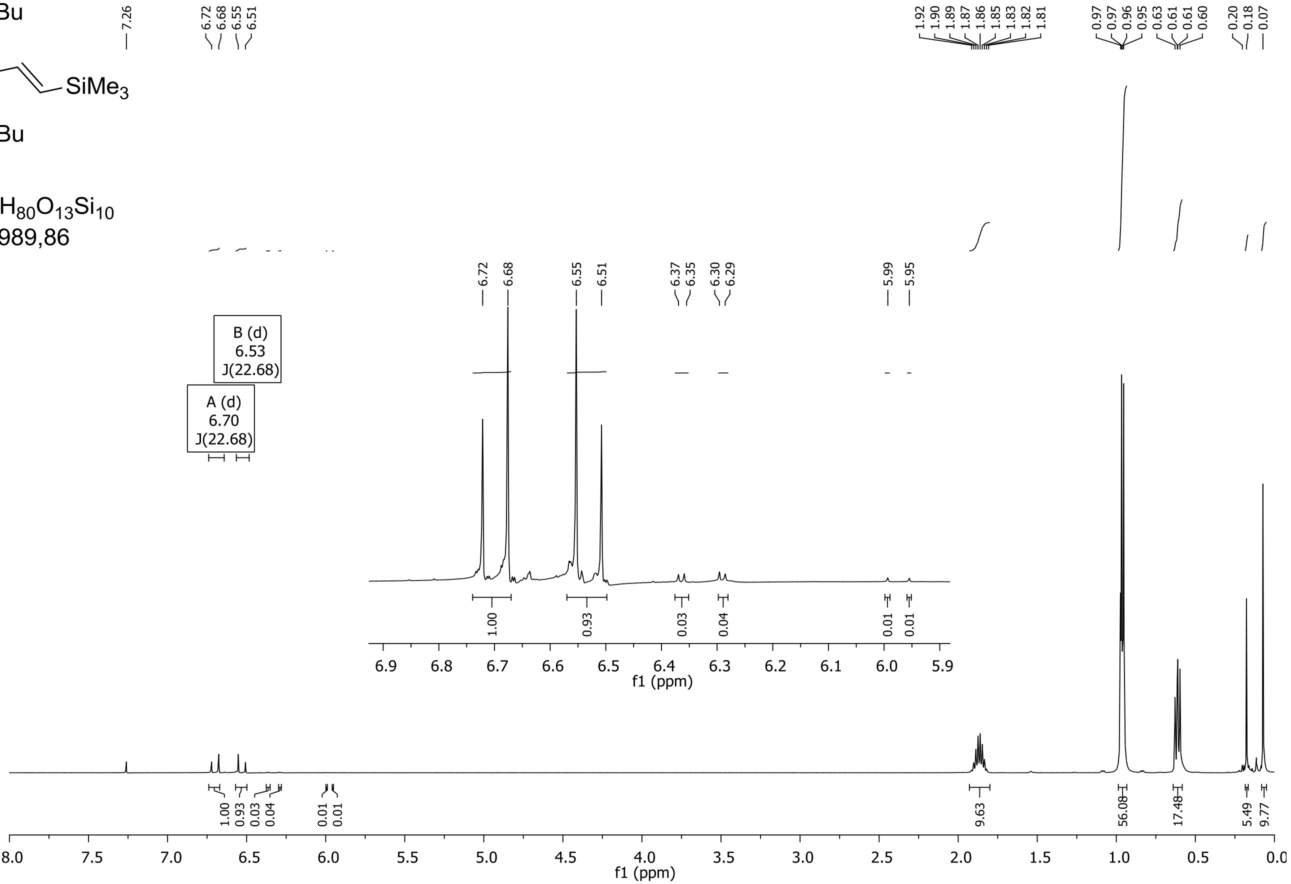
Figure 12. ^{29}Si NMR of 3-5b mixture.

3.4 Hydrosilylation of 2c with silsesquioxane 1

Since boiling point of trimethylsilylacetylene is 53°C, the reaction time was determined by ^{1}H NMR and FT-IR (ATR) spectroscopies and the reaction was performed in schlenk flask with Rotaflo® stopcock. Reaction time = 30 min.

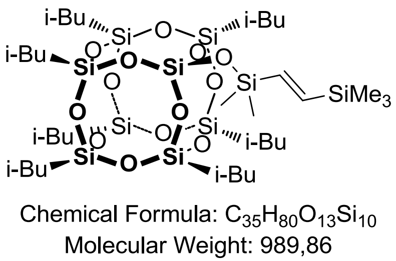


Chemical Formula: $C_{35}H_{80}O_{13}Si_{10}$
Molecular Weight: 989,86



.

Figure 13. ^{1}**H NMR of compound 3c.**



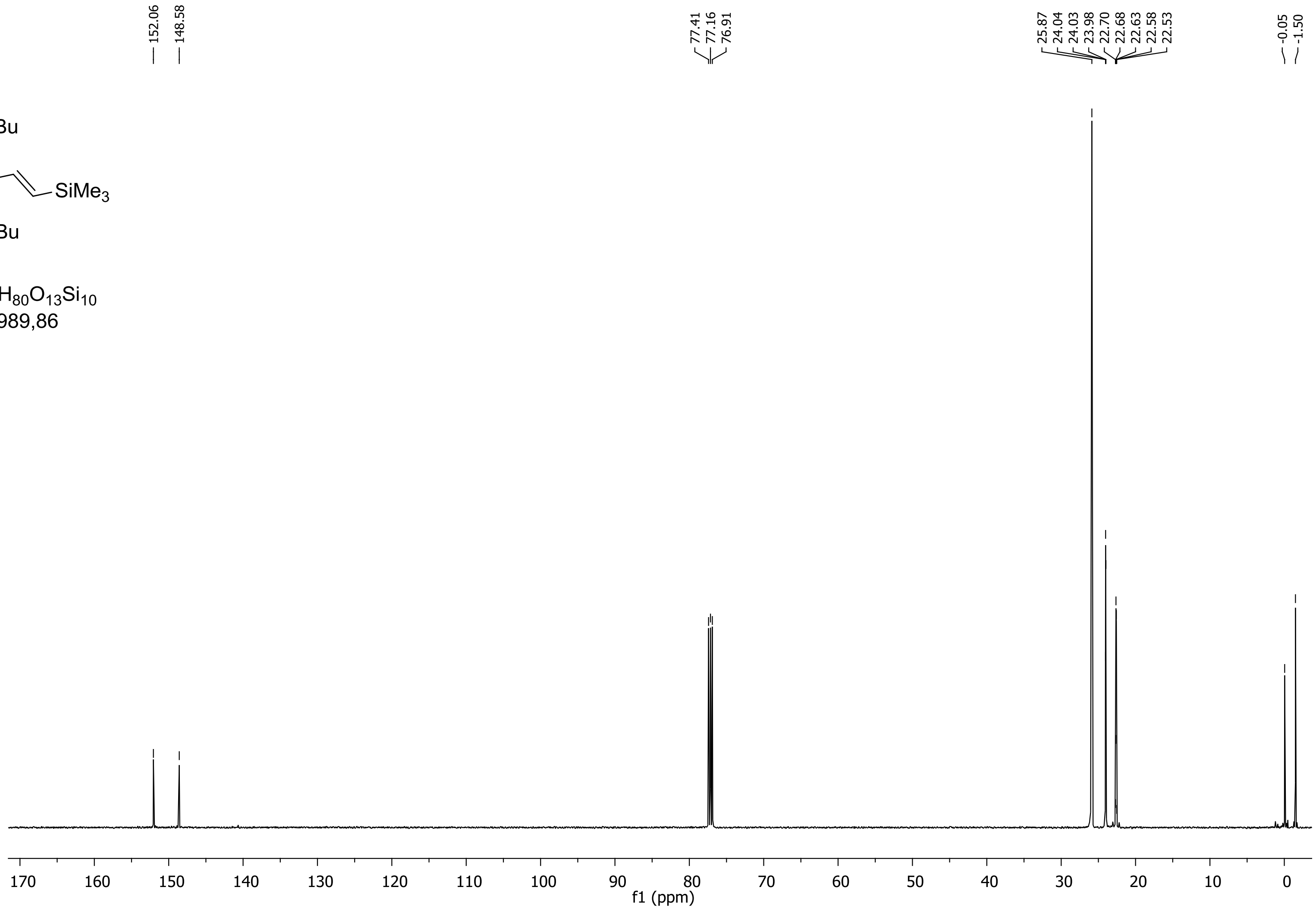
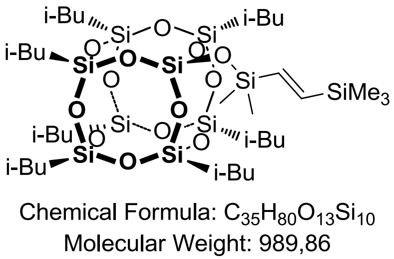


Figure 14. ^{13}**C NMR of compound 3c.**



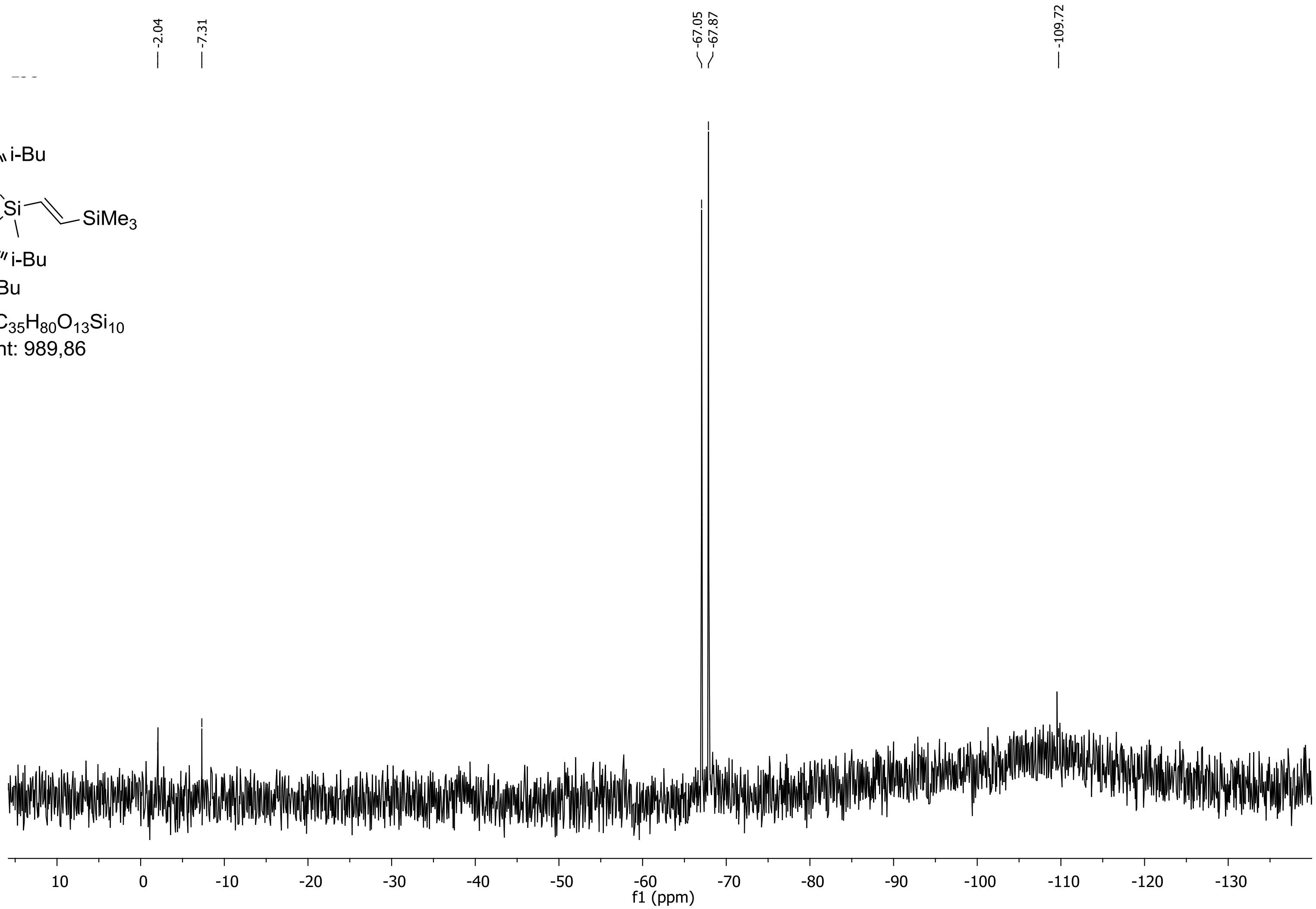


Figure 15. ^{29}Si NMR of compound 3c.

3.5 Hydrosilylation of 2d with silsesquioxane 1

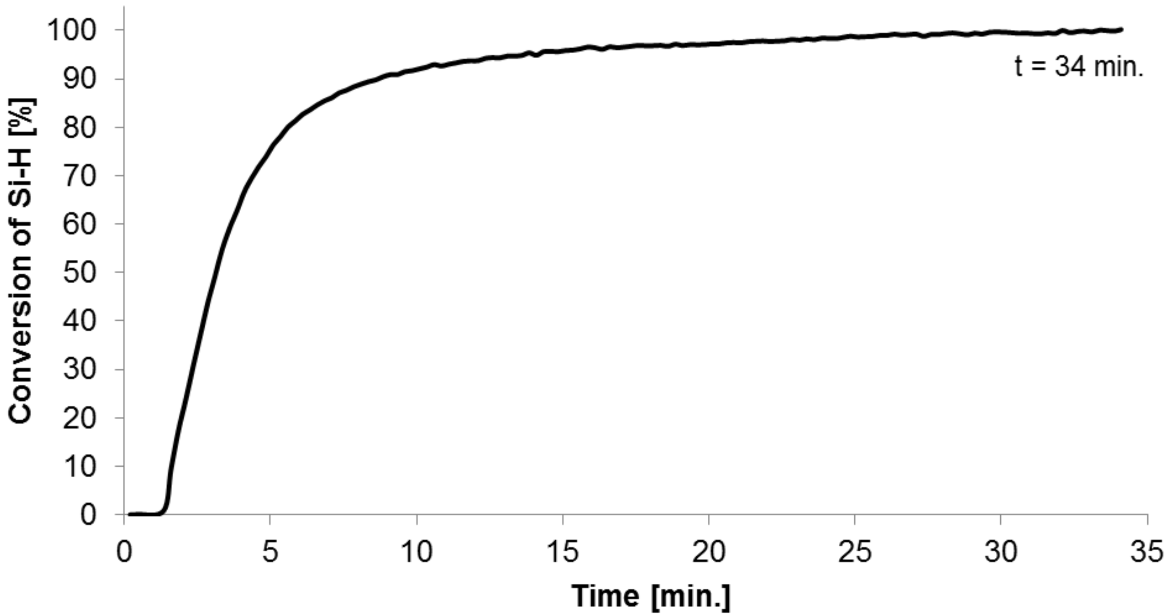


Figure 16. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2d with 1.

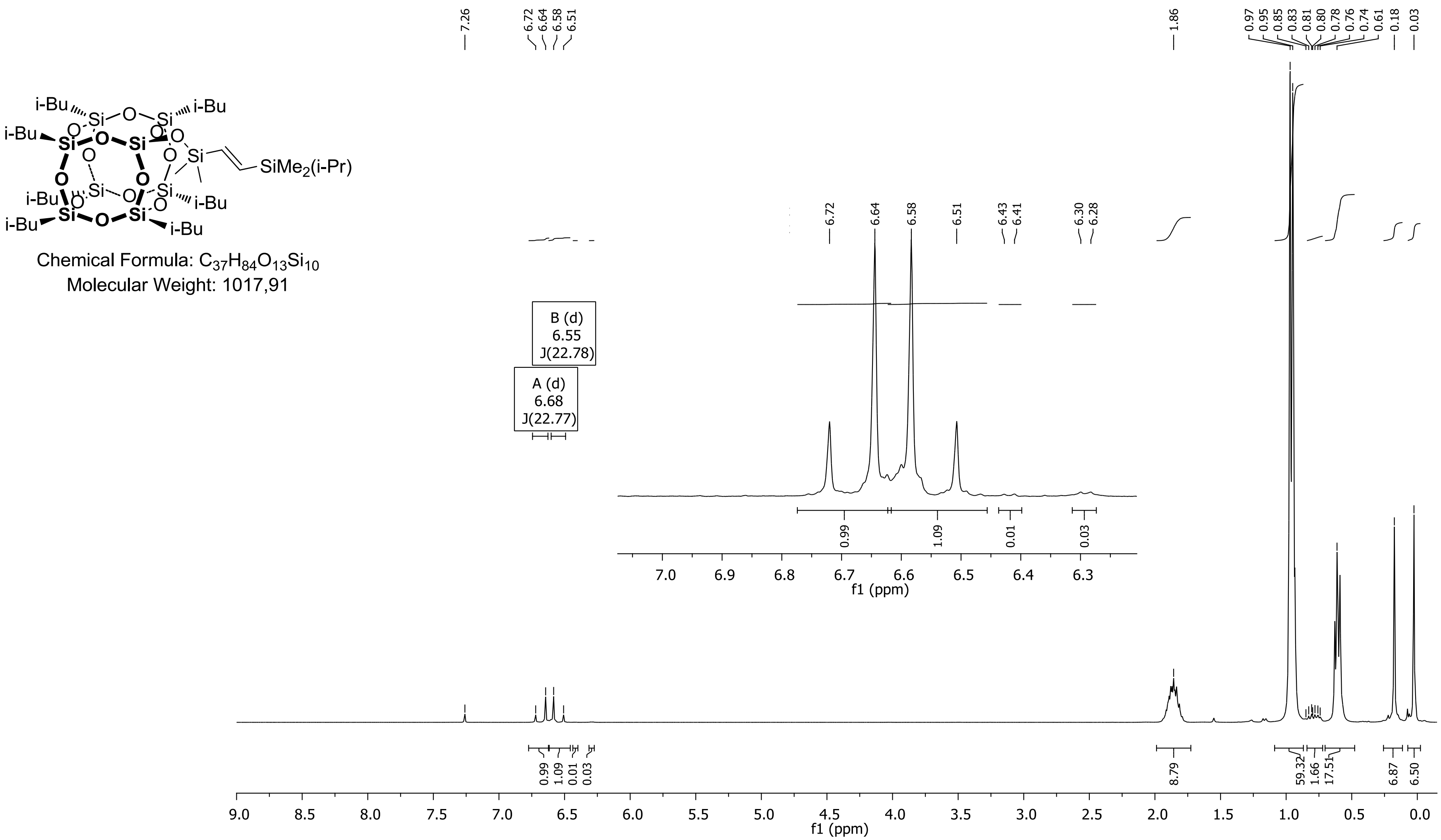
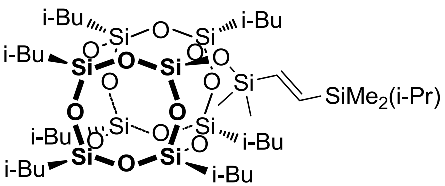


Figure 17. ^{1}H NMR of compound 3d.



Chemical Formula: $C_{37}H_{84}O_{13}Si_{10}$
Molecular Weight: 1017,91

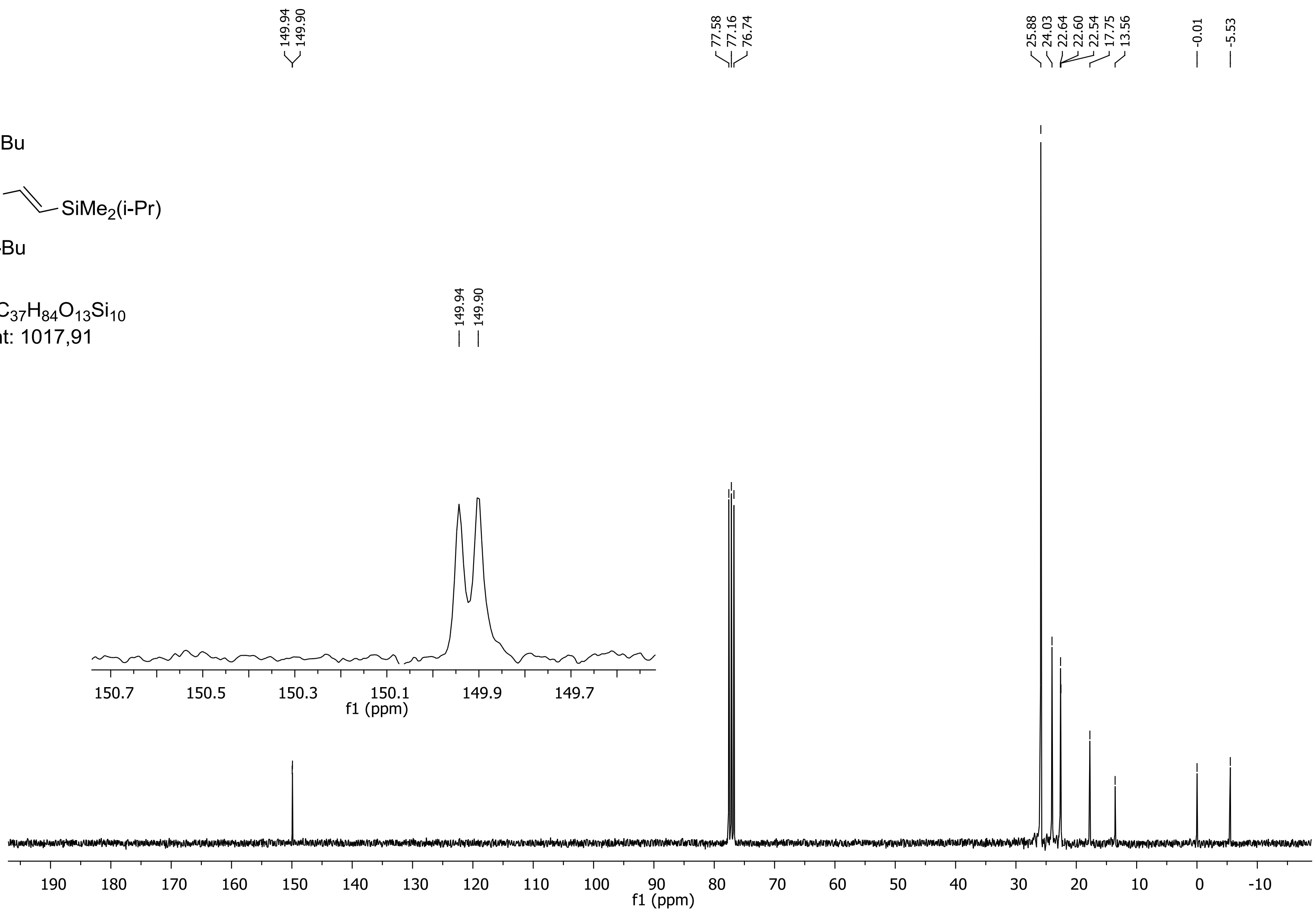


Figure 18. ^{13}C NMR of compound 3d.

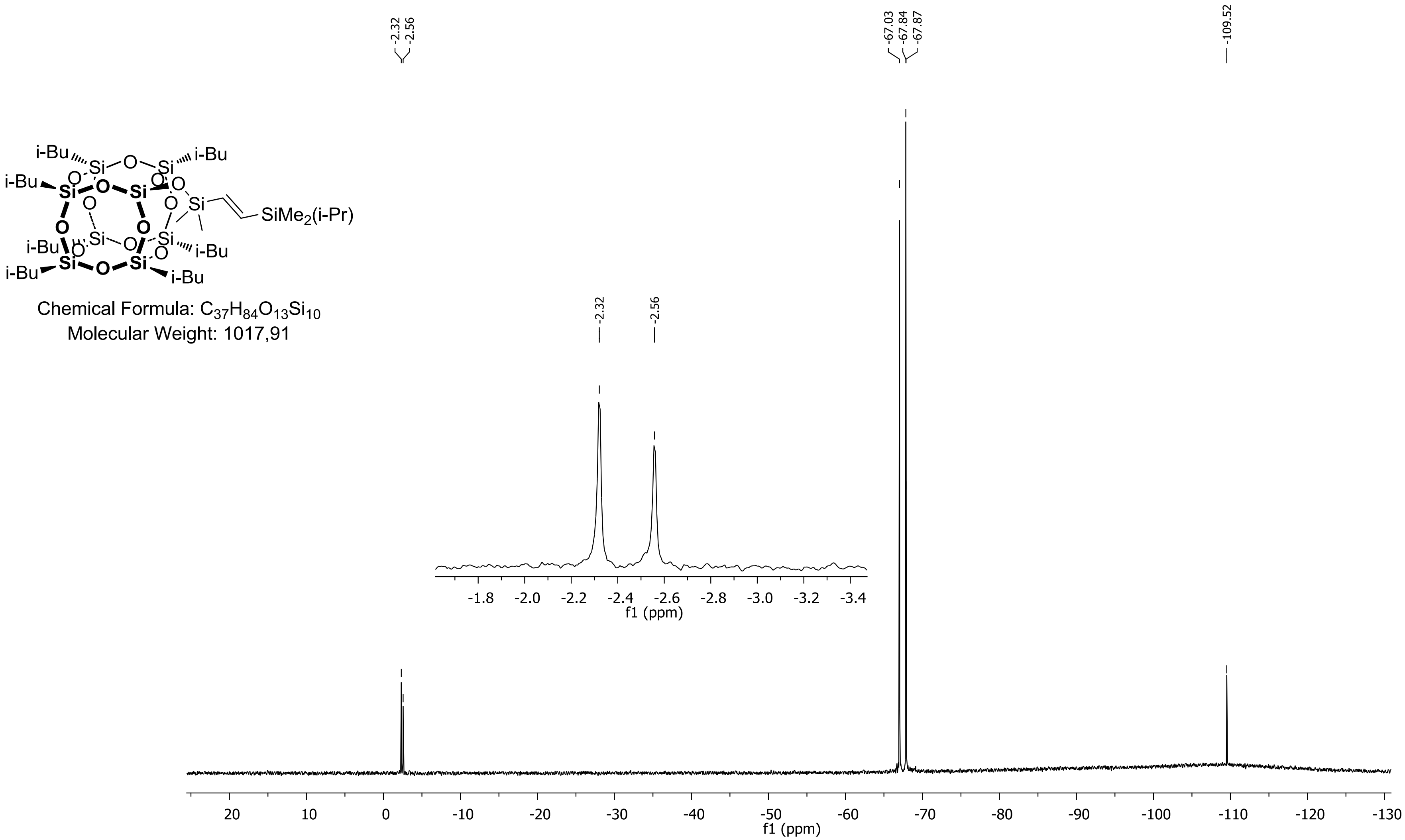


Figure 19. ^{29}Si NMR of compound 3d.

3.6 Hydrosilylation of 2e with silsesquioxane 1

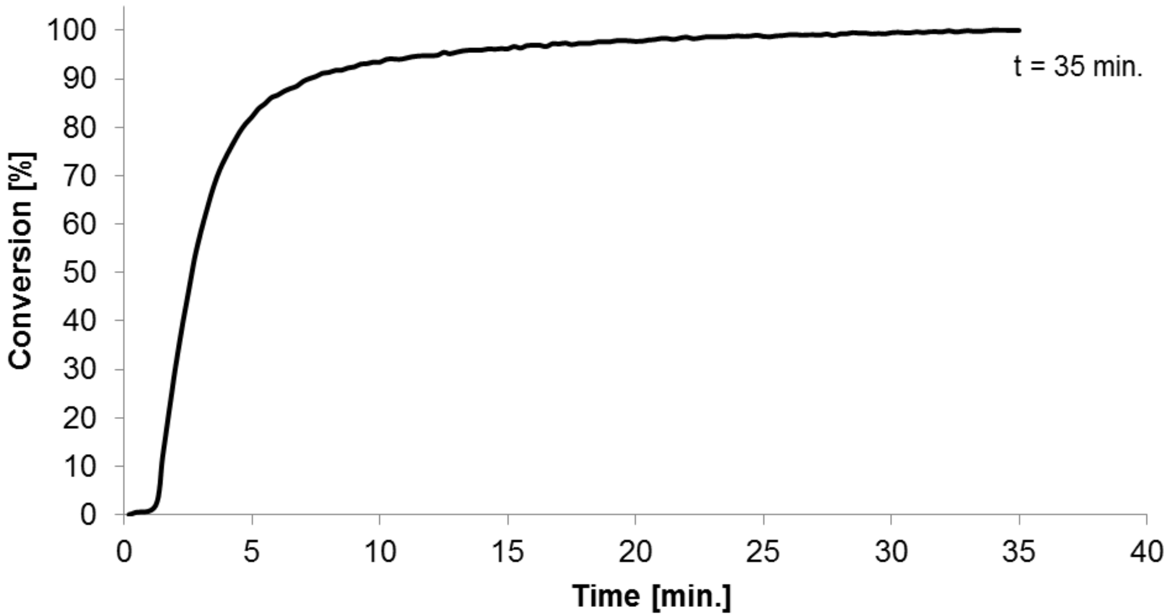
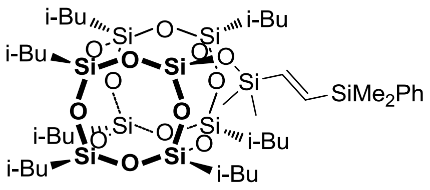


Figure 20. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2e with 1.



Chemical Formula: $C_{40}H_{82}O_{13}Si_{10}$
Molecular Weight: 1051,93

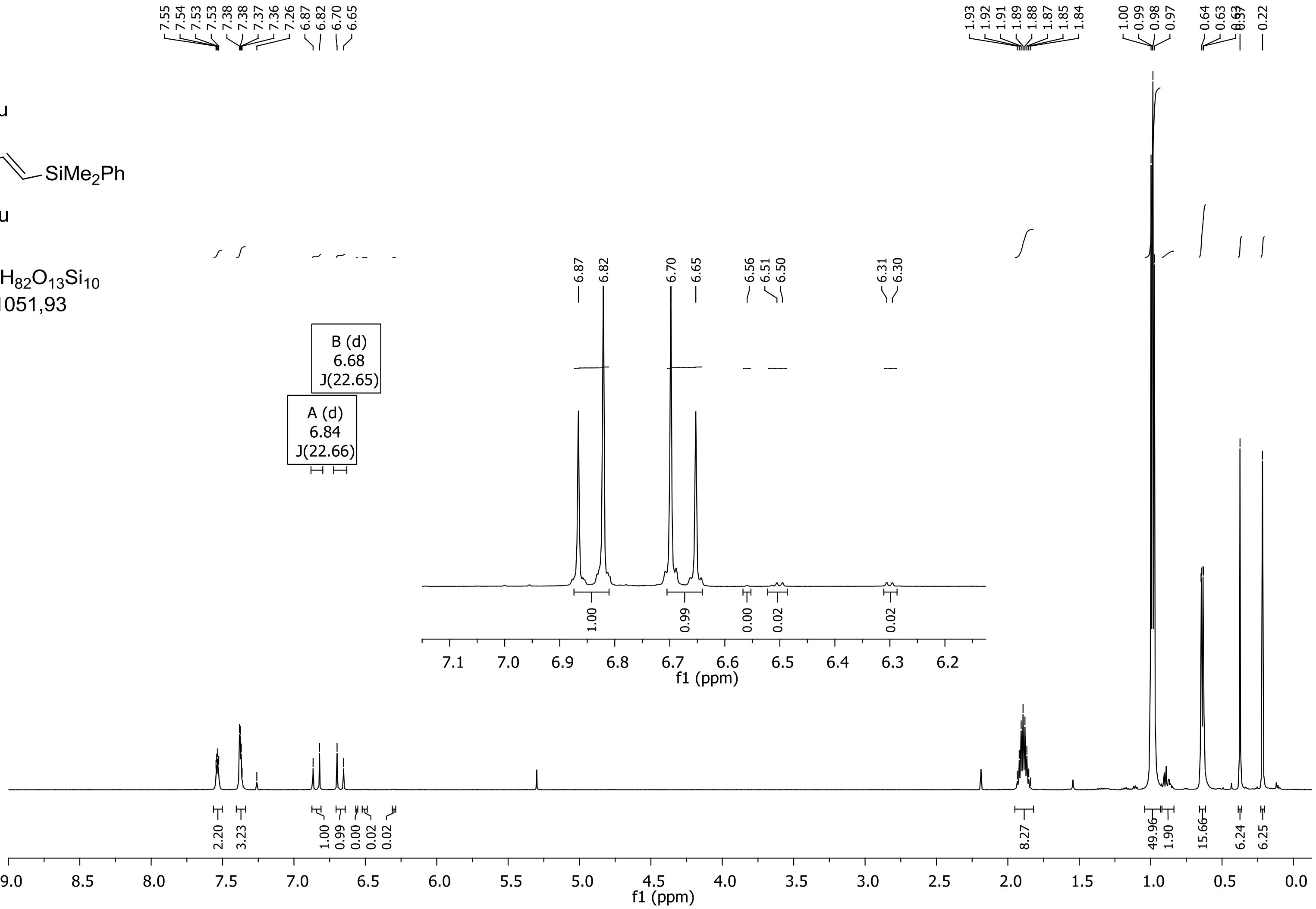
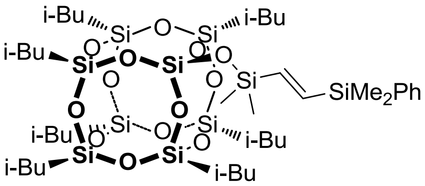


Figure 21. ^{1}H NMR of compound 3e.



Chemical Formula: $C_{40}H_{82}O_{13}Si_{10}$
Molecular Weight: 1051,93

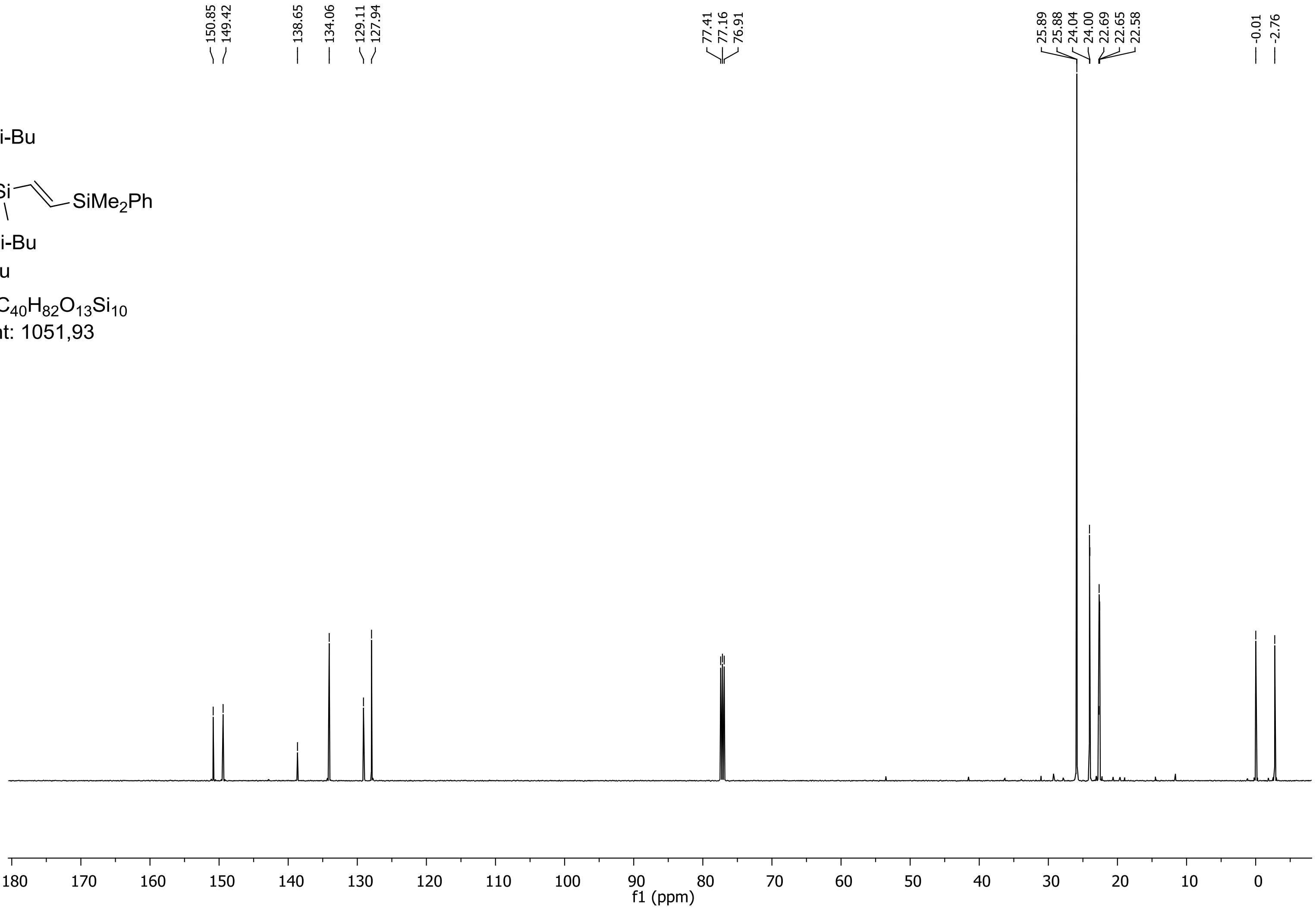


Figure 22. [13]C NMR of compound 3e.

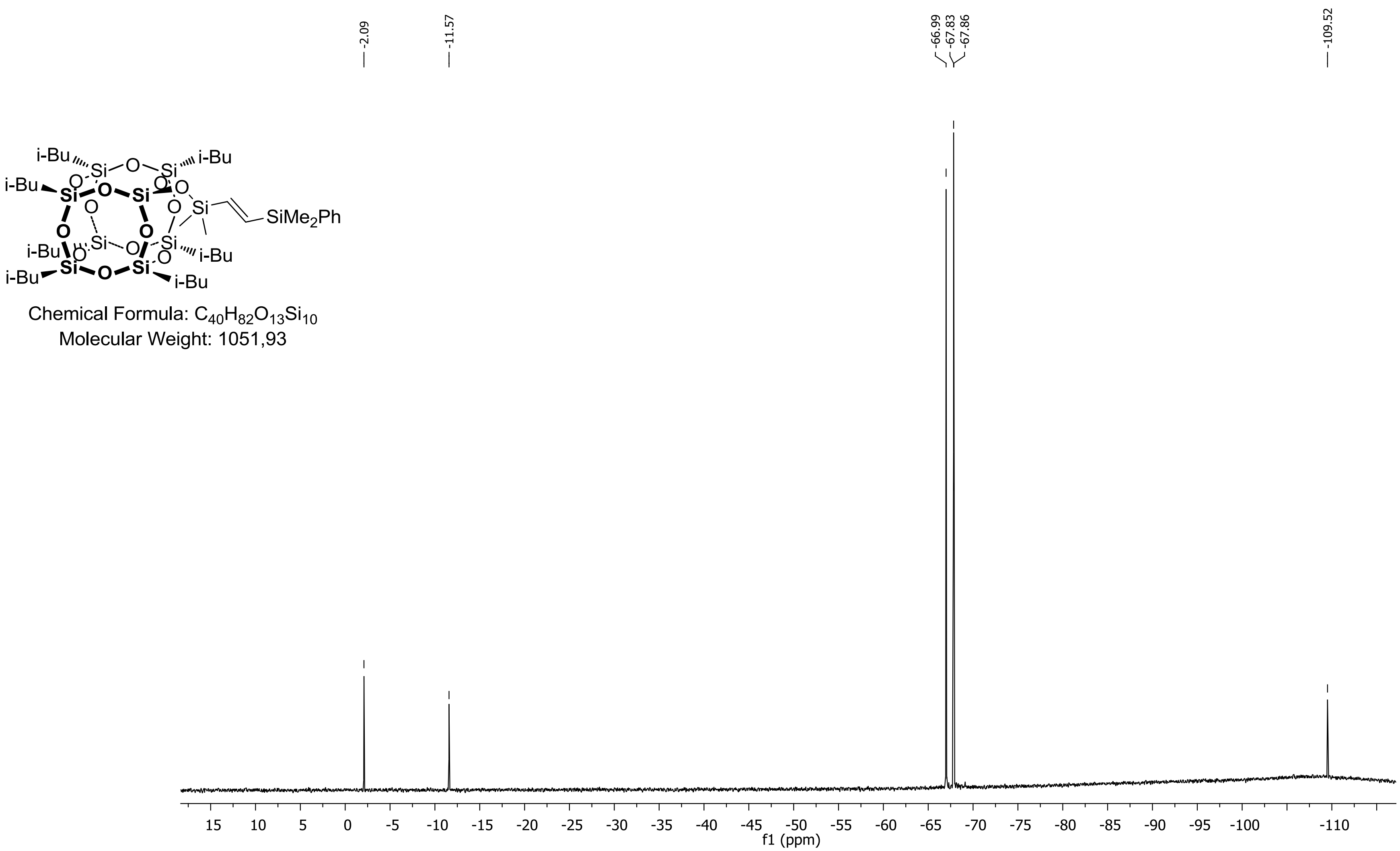


Figure 23. ^{29}**Si NMR of compound 3e.**

3.7 Hydrosilylation of 2f with silsesquioxane 1

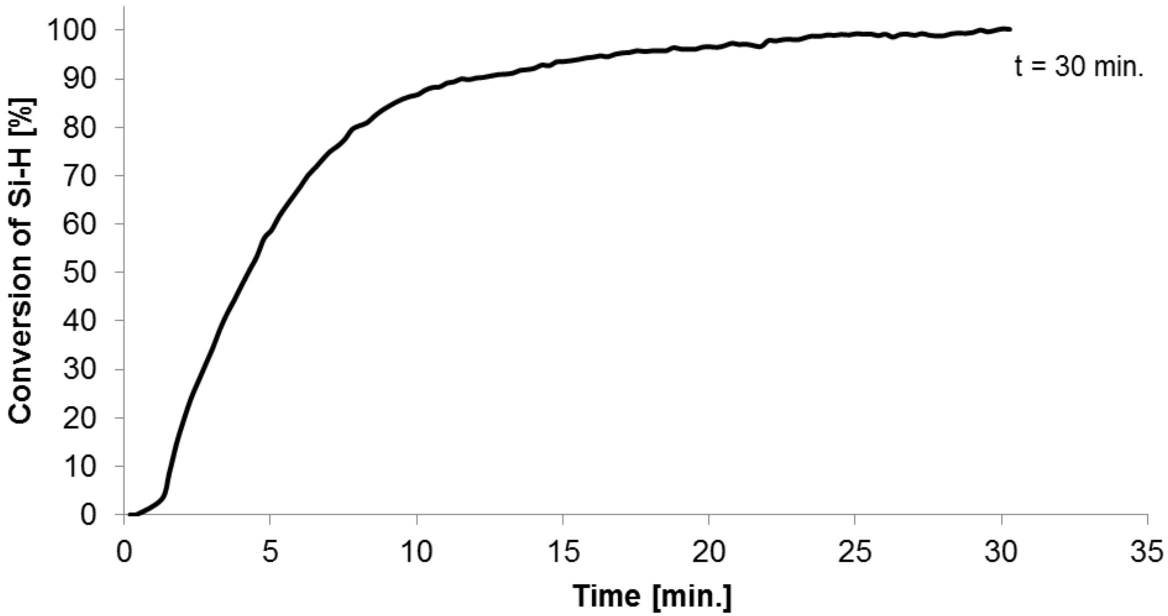
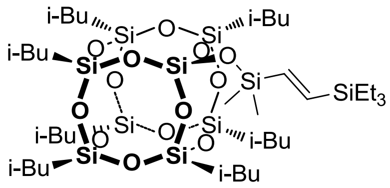


Figure 24. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2f with 1.



Chemical Formula: $C_{38}H_{86}O_{13}Si_{10}$
Molecular Weight: 1031,94

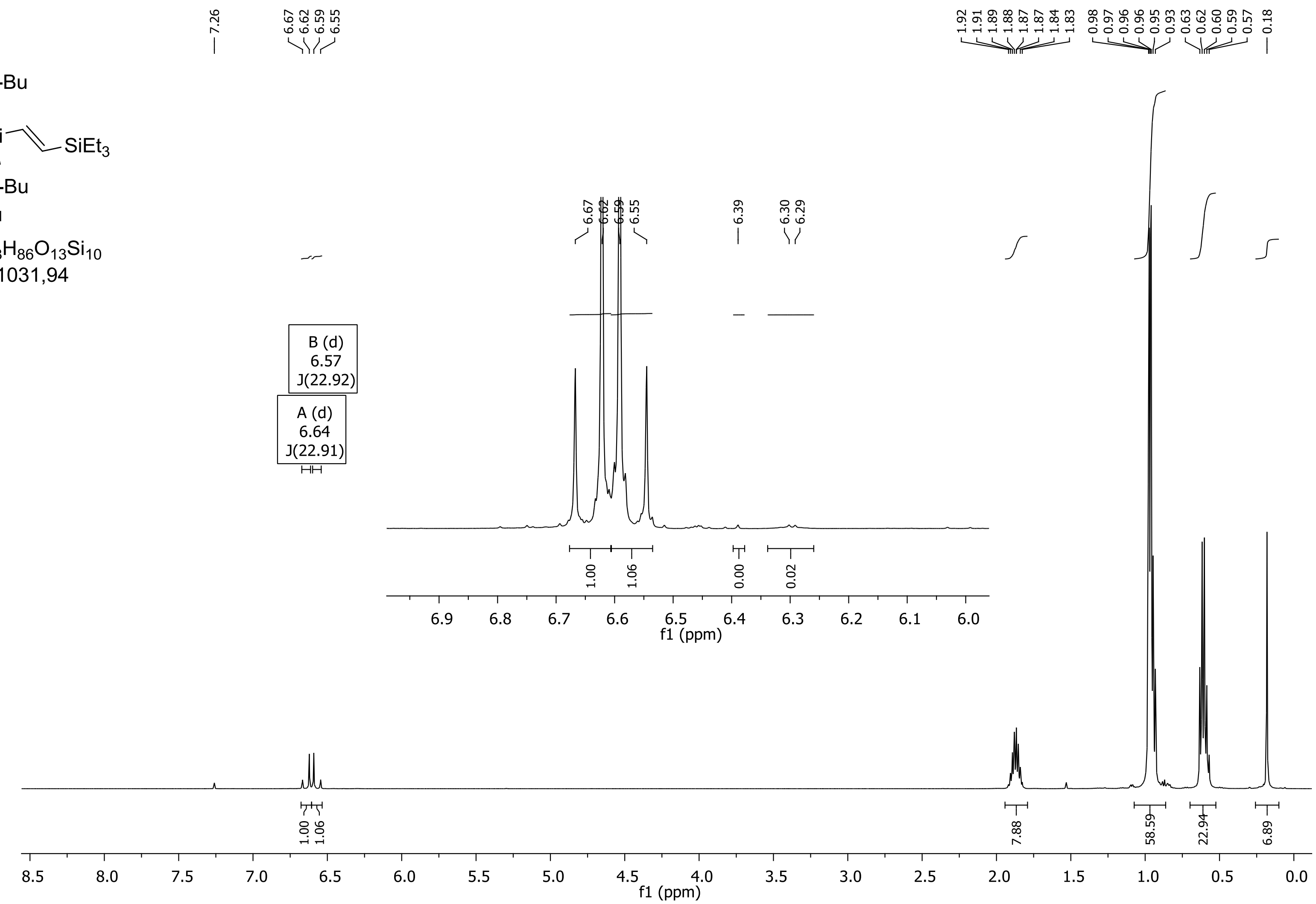
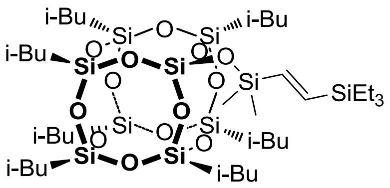


Figure 25. ^{1}**H NMR of compound 3f.**



Chemical Formula: $C_{38}H_{86}O_{13}Si_{10}$
Molecular Weight: 1031,94

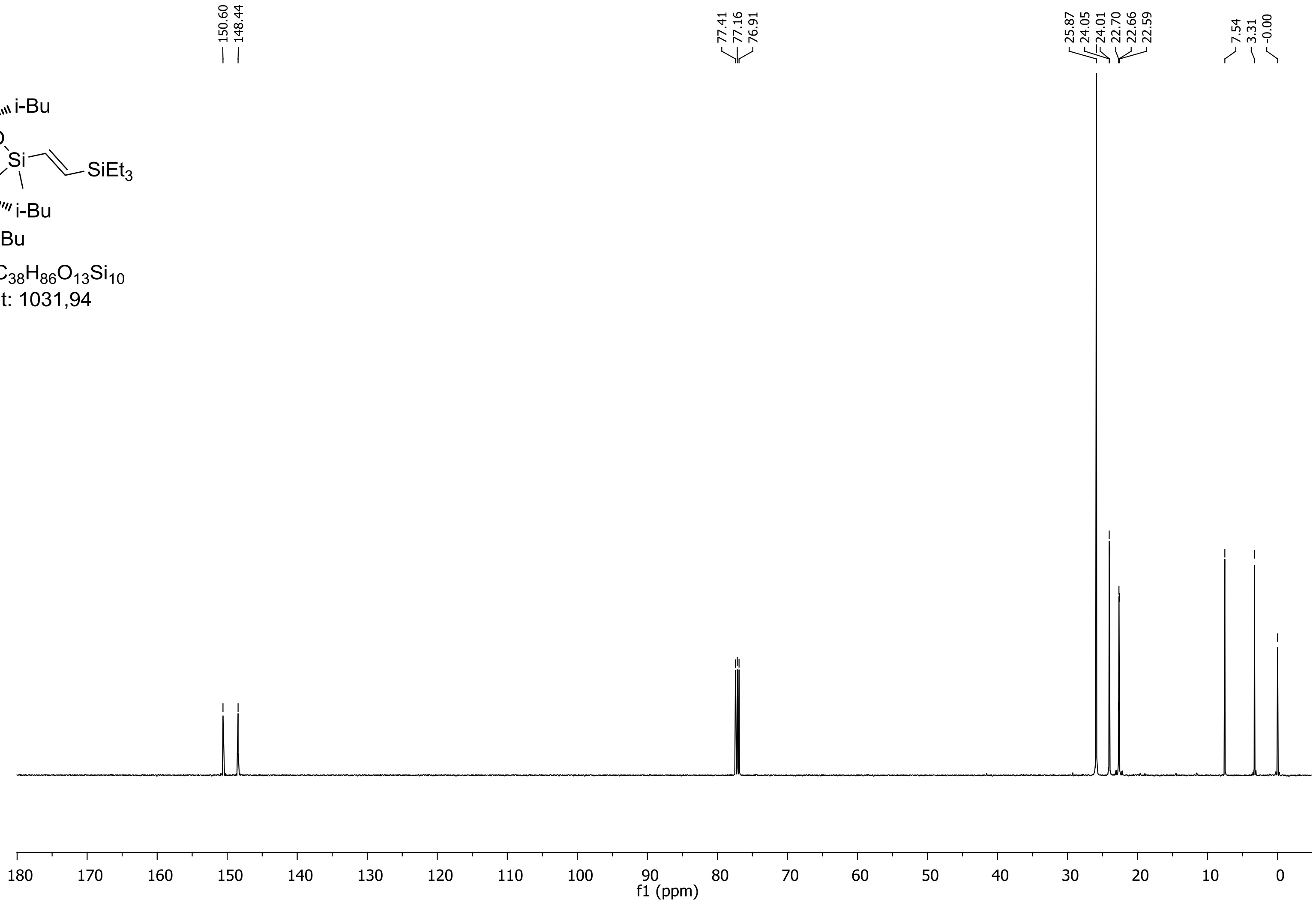
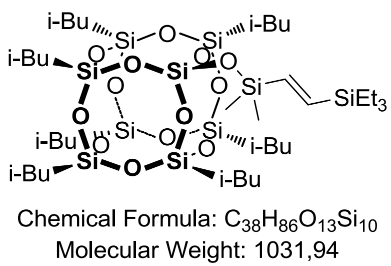


Figure 26. ^{13}C NMR of compound 3f.



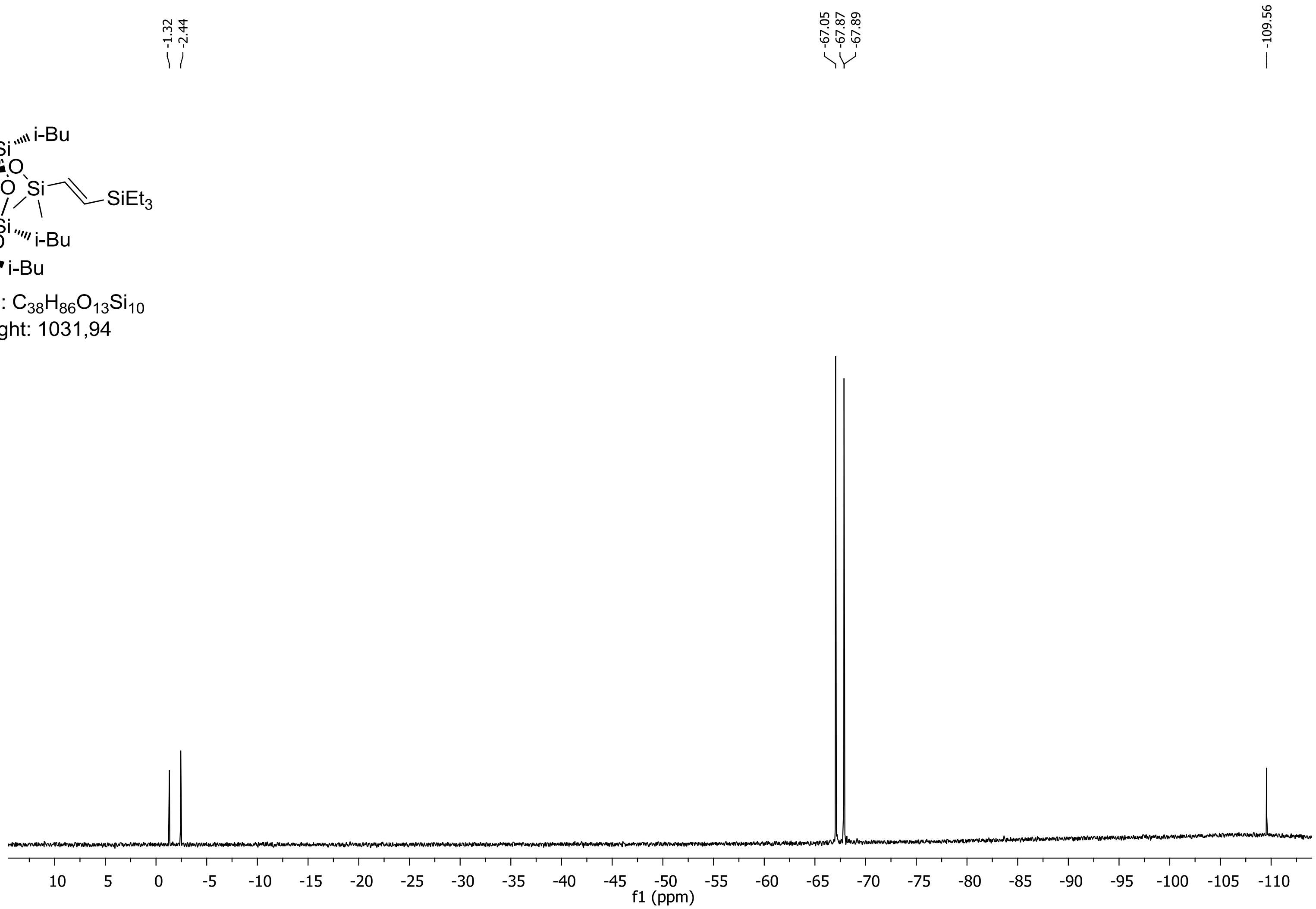


Figure 27. ^{29}**Si NMR of compound 3f.**

3.8 Hydrosilylation of 2g with silsesquioxane 1

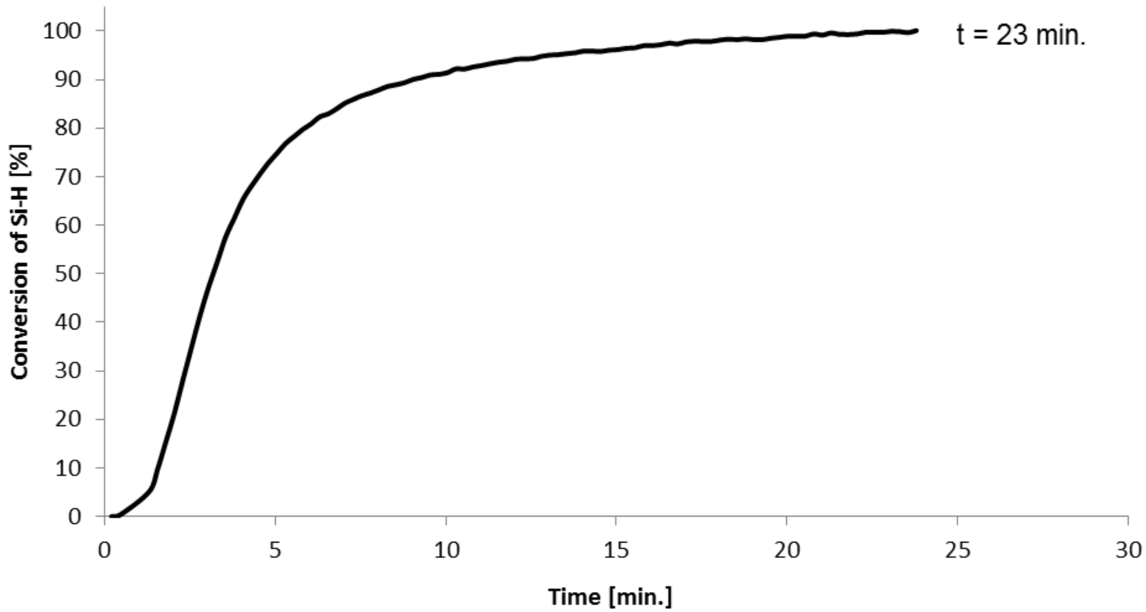


Figure 28. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2g with 1.

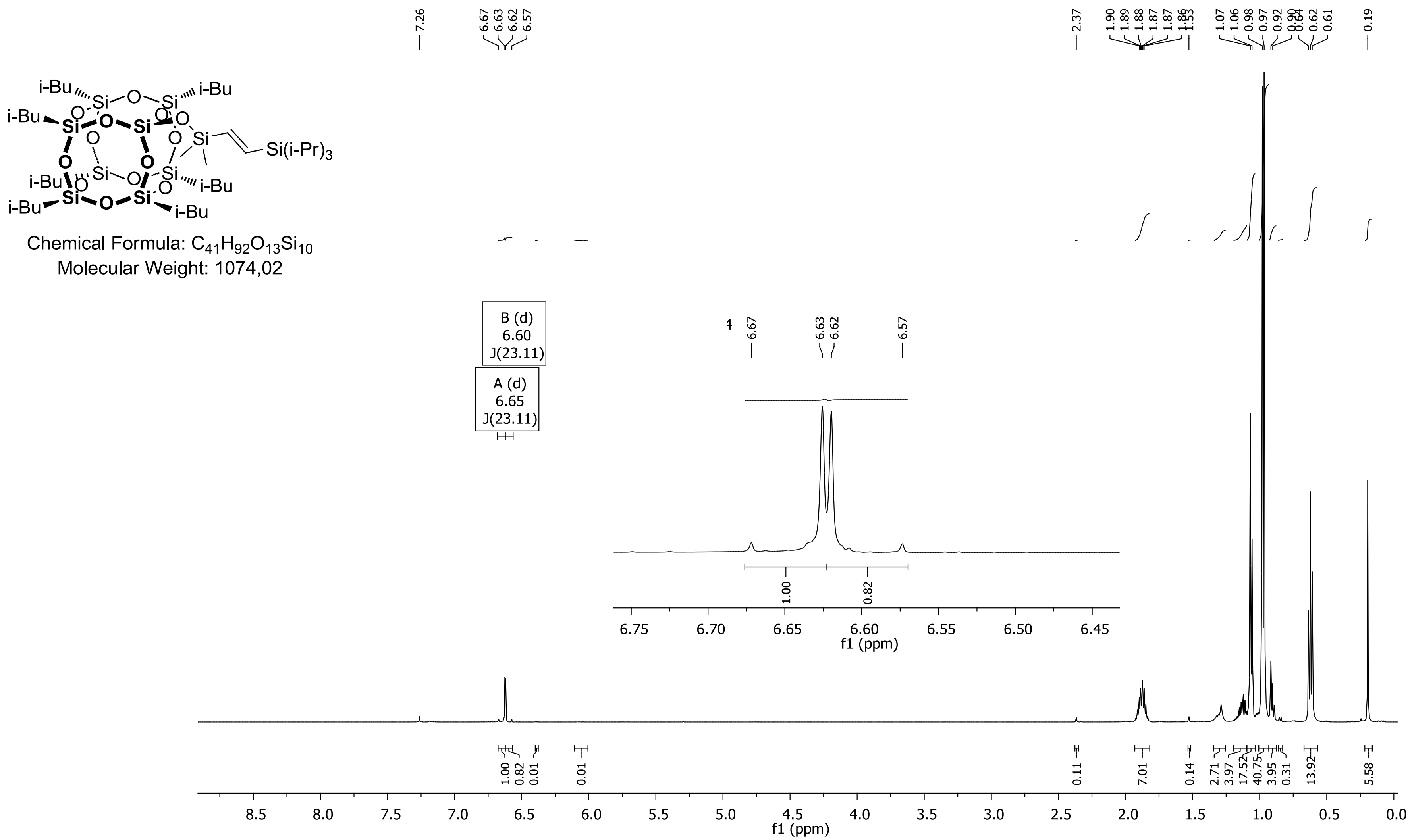
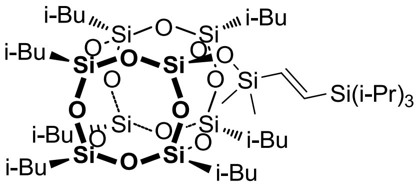


Figure 29. ^{1}H NMR of compound 3g.



Chemical Formula: $C_{41}H_{92}O_{13}Si_{10}$
Molecular Weight: 1074,02

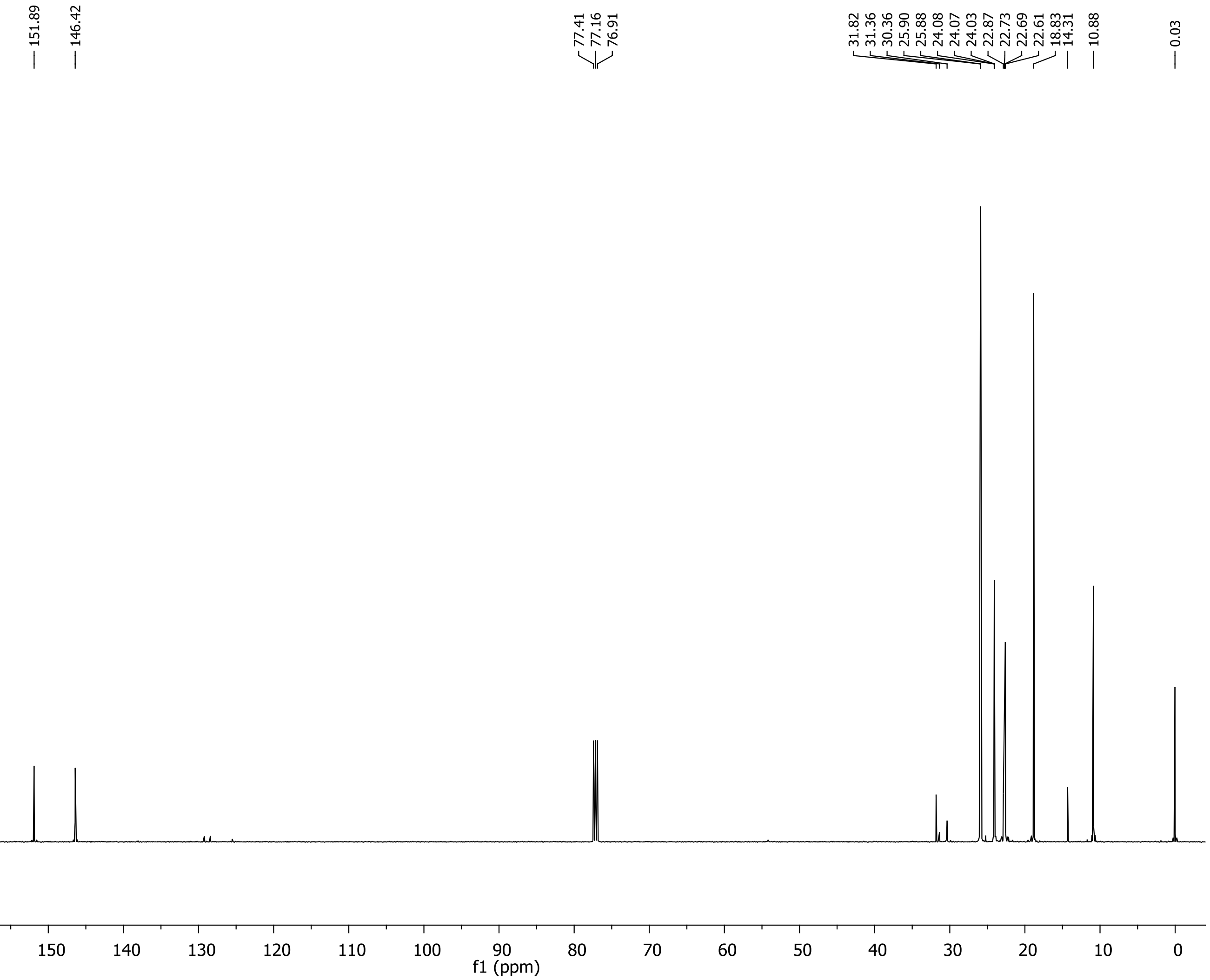
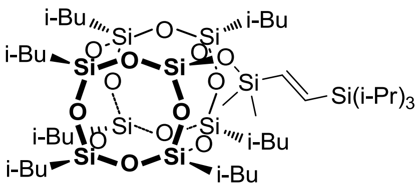


Figure 30. ^{13}C NMR of compound 3g.



Chemical Formula: $C_{41}H_{92}O_{13}Si_{10}$
Molecular Weight: 1074,02

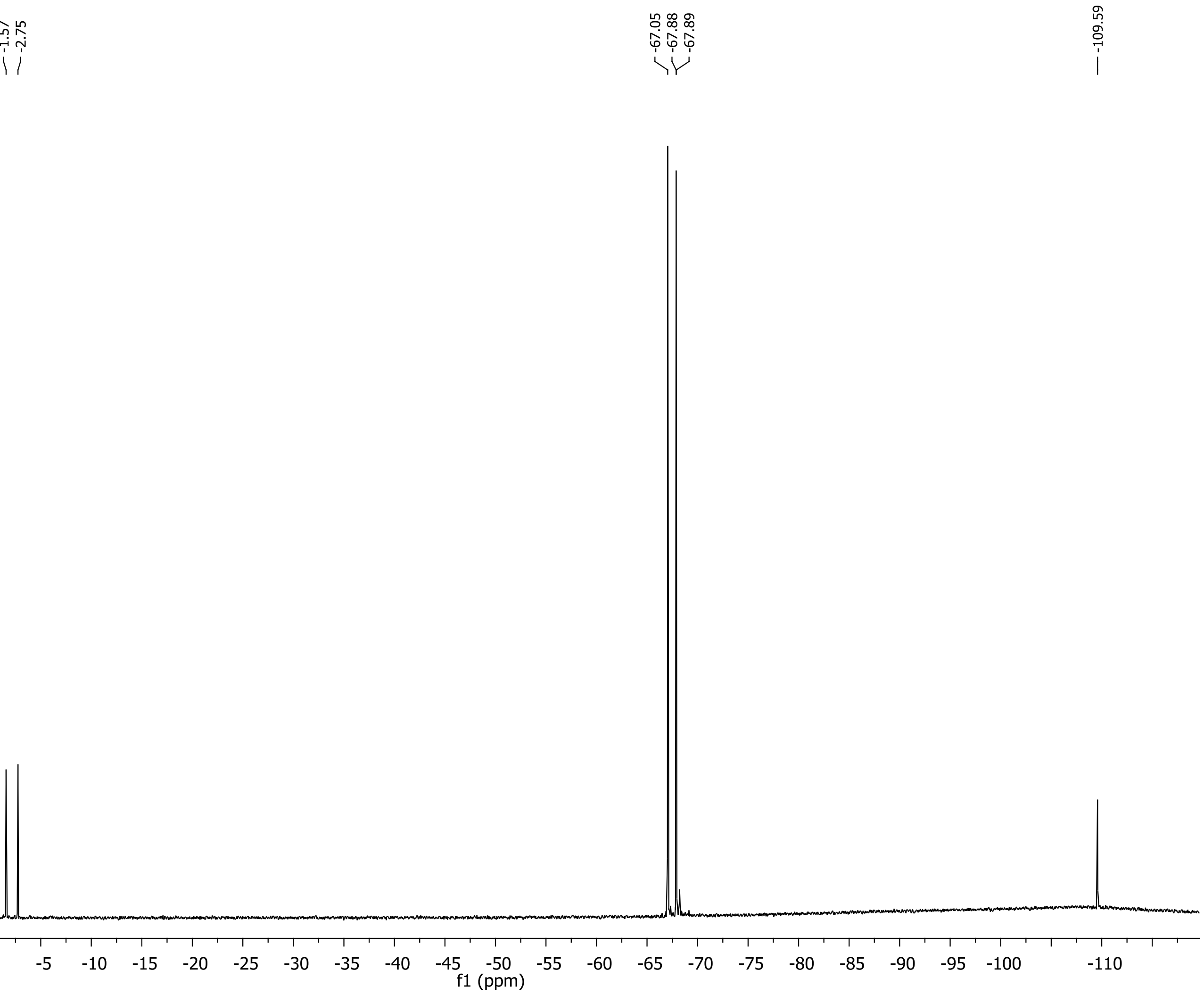


Figure 31. ^{29}Si NMR of compound 3g.

3.9 Hydrosilylation of 2h with silsesquioxane 1

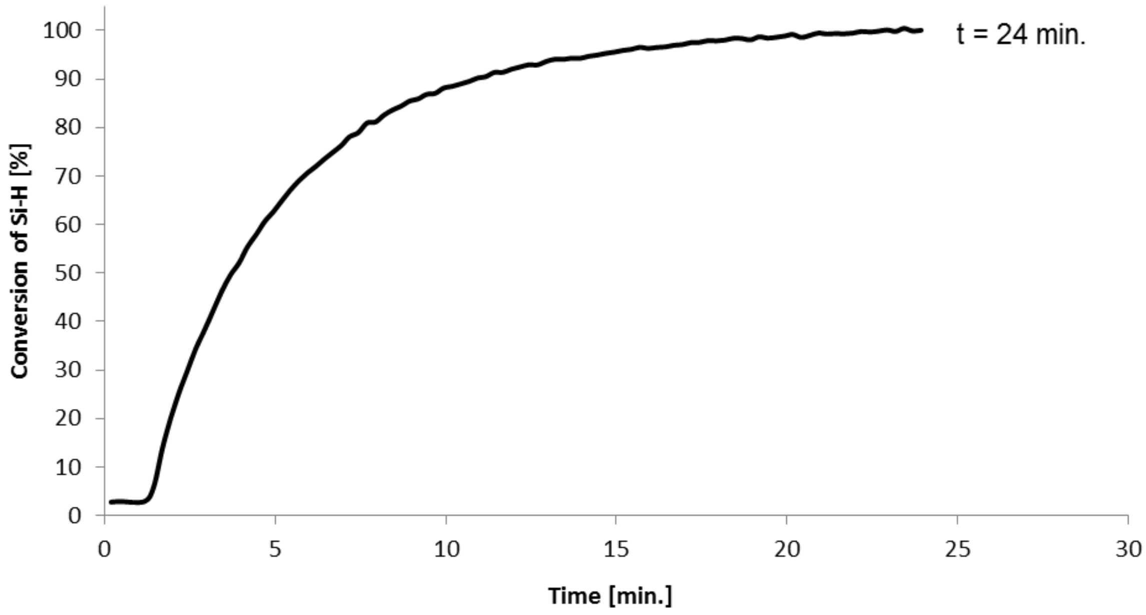
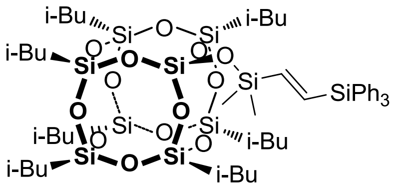


Figure 32. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2h with 1.



Chemical Formula: $C_{50}H_{86}O_{13}Si_{10}$
Molecular Weight: 1176,07

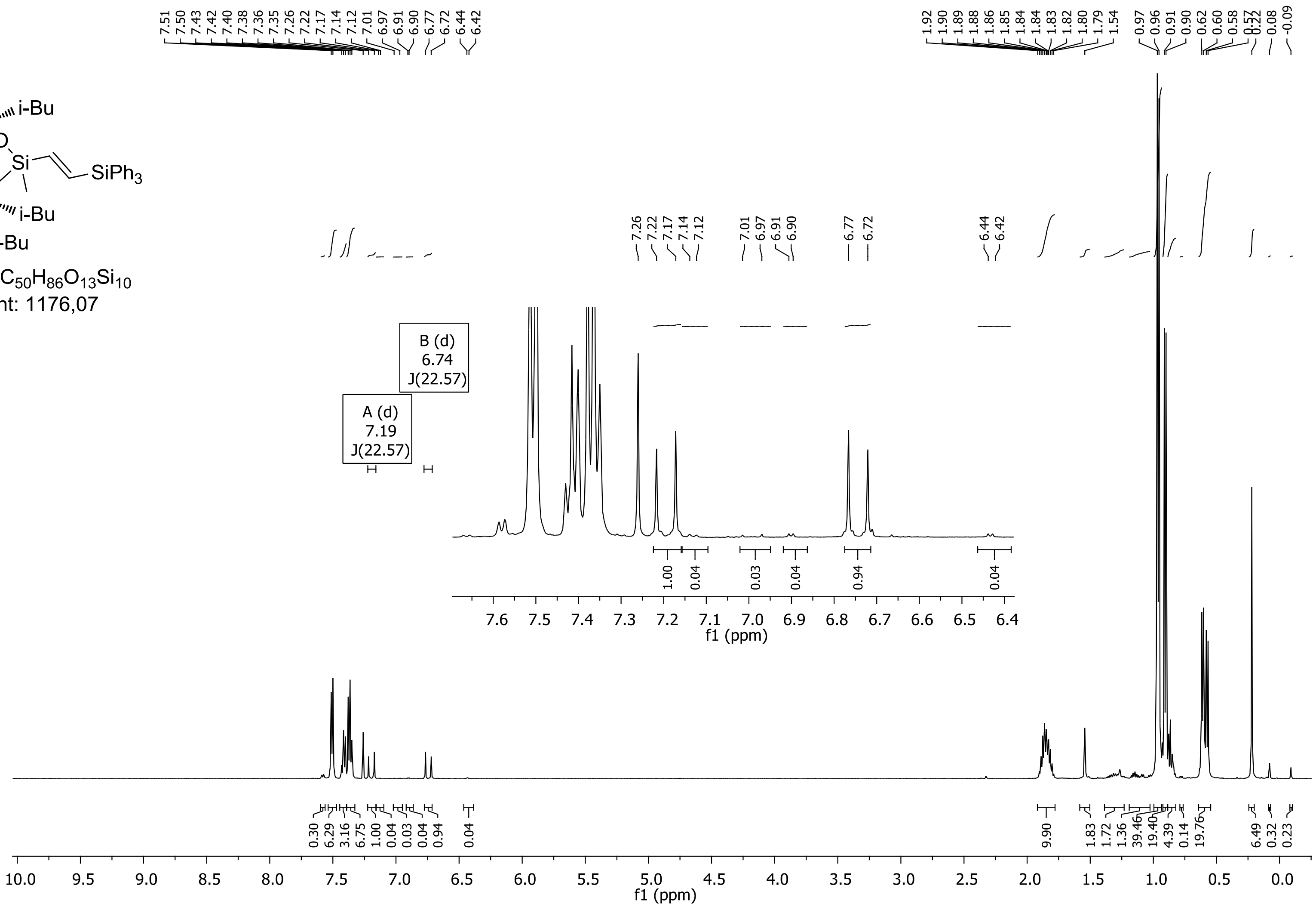
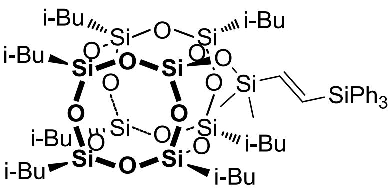


Figure 33. ^{1}H NMR of compound 3h.



Chemical Formula: $C_{50}H_{86}O_{13}Si_{10}$
Molecular Weight: 1176,07

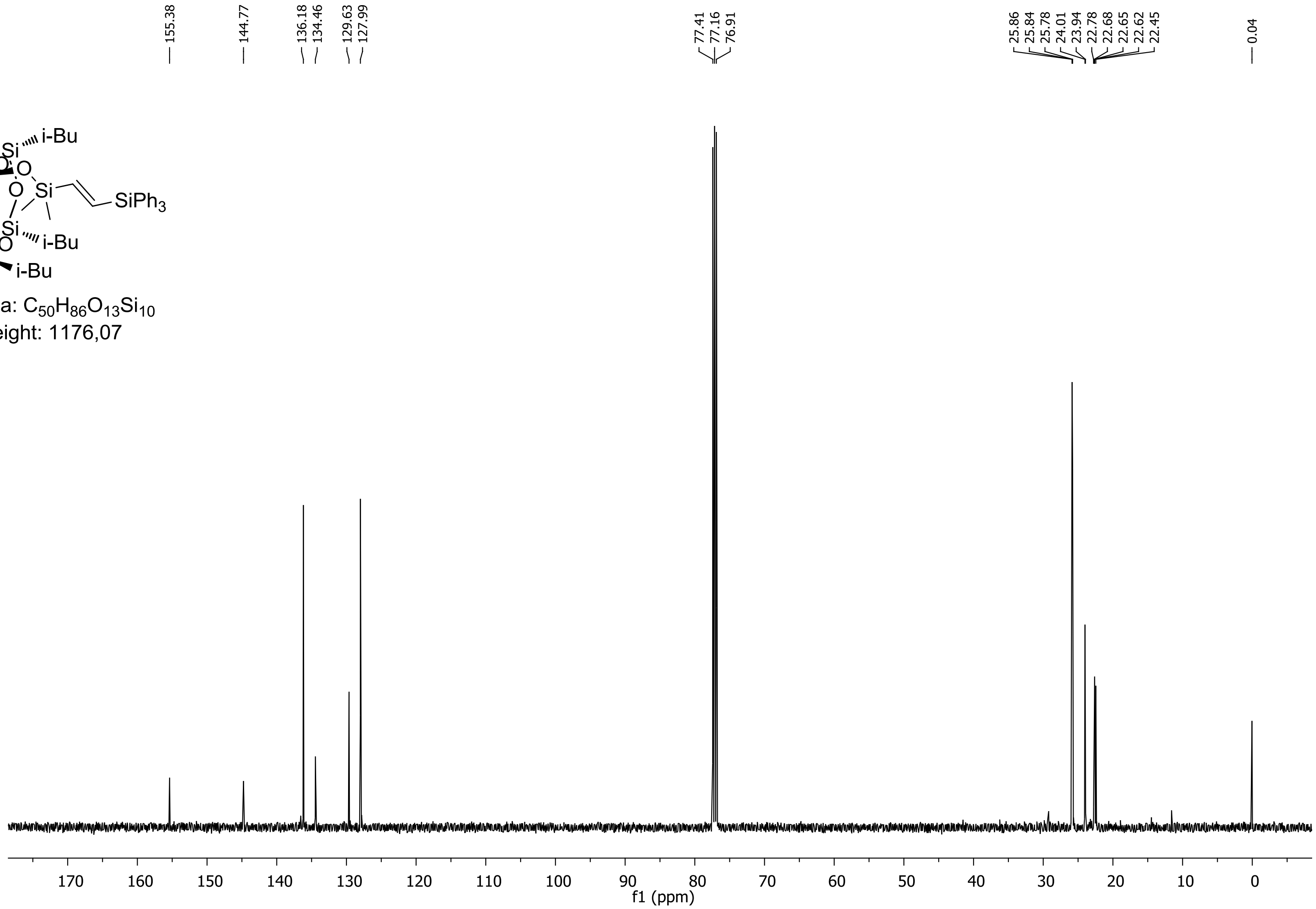


Figure 34. **[13]C NMR of compound 3h.**

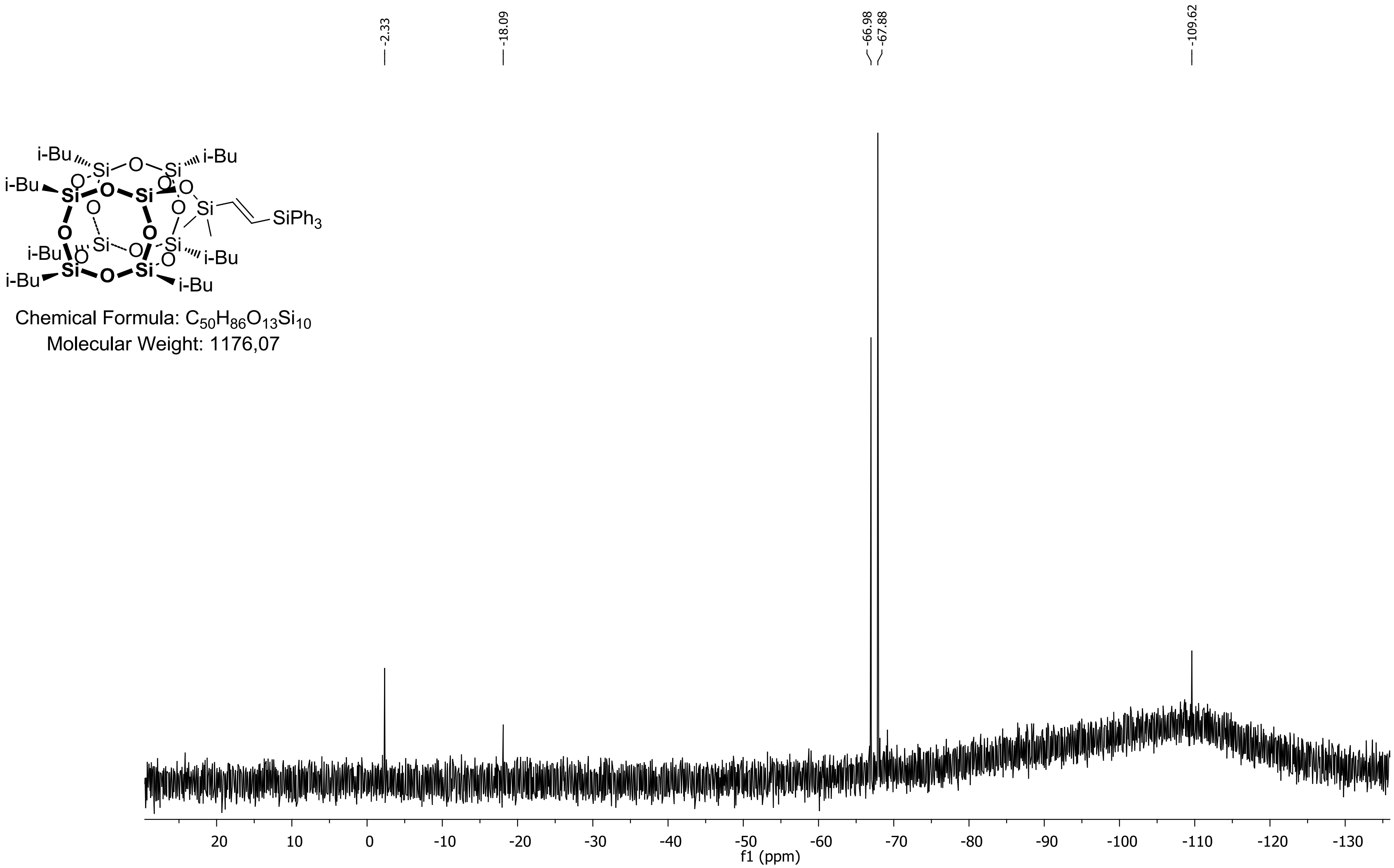


Figure 35. ^{29}Si NMR of compound 3h.

3.10 Hydrosilylation of 2i with silsesquioxane 1

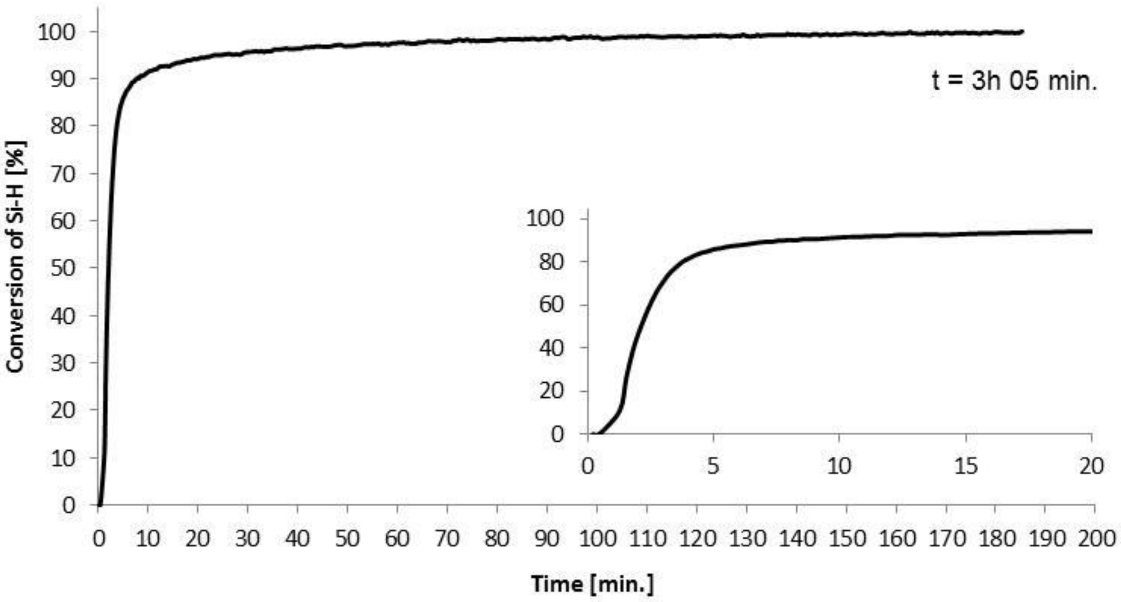
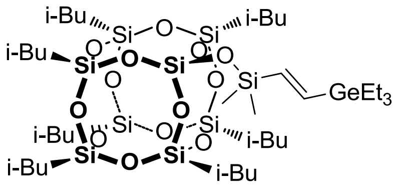


Figure 36. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2i with 1.



Chemical Formula: $C_{38}H_{86}GeO_{13}Si_9$
Molecular Weight: 1076,49

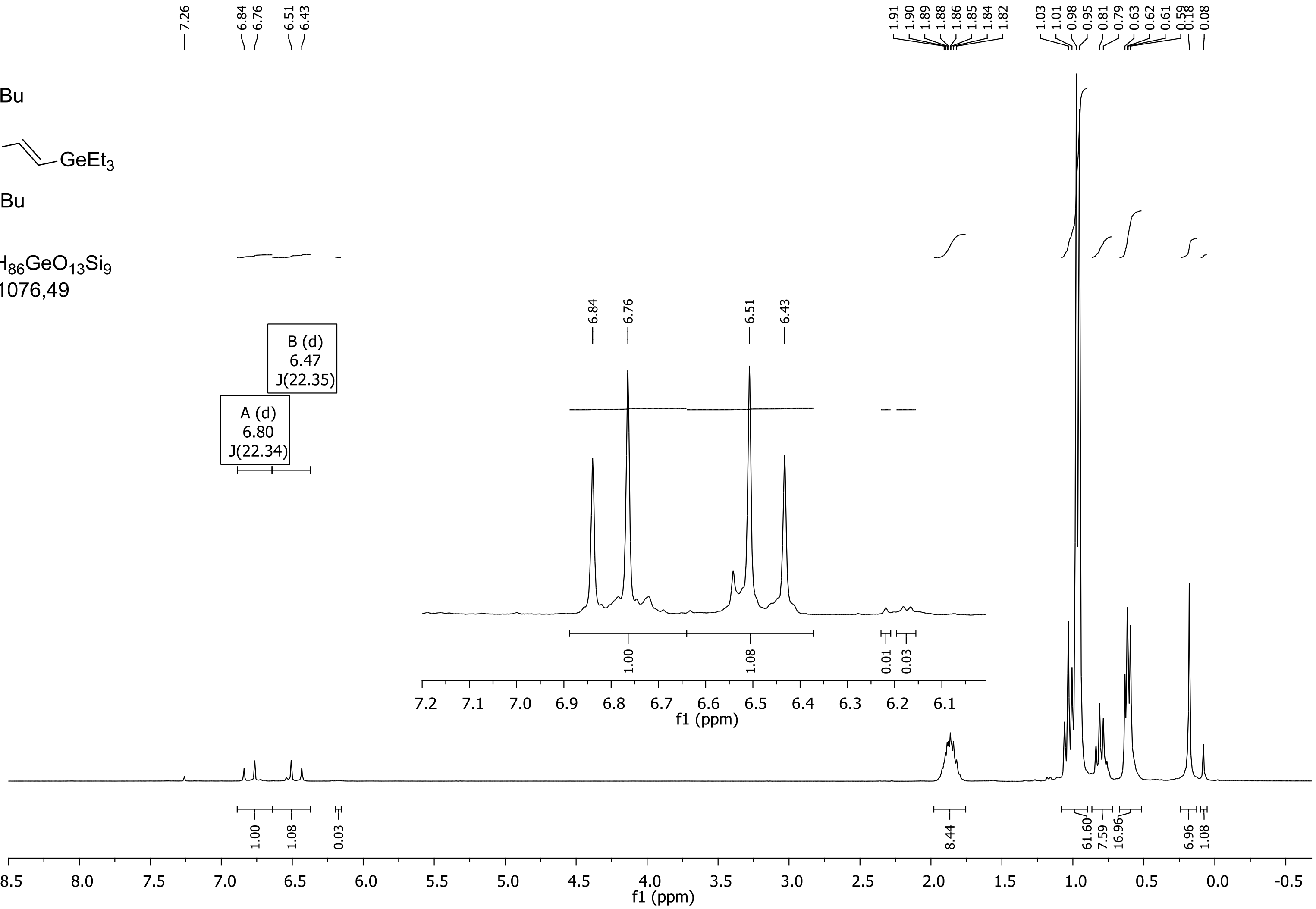


Figure 37. ^{1}H NMR of compound 3i.

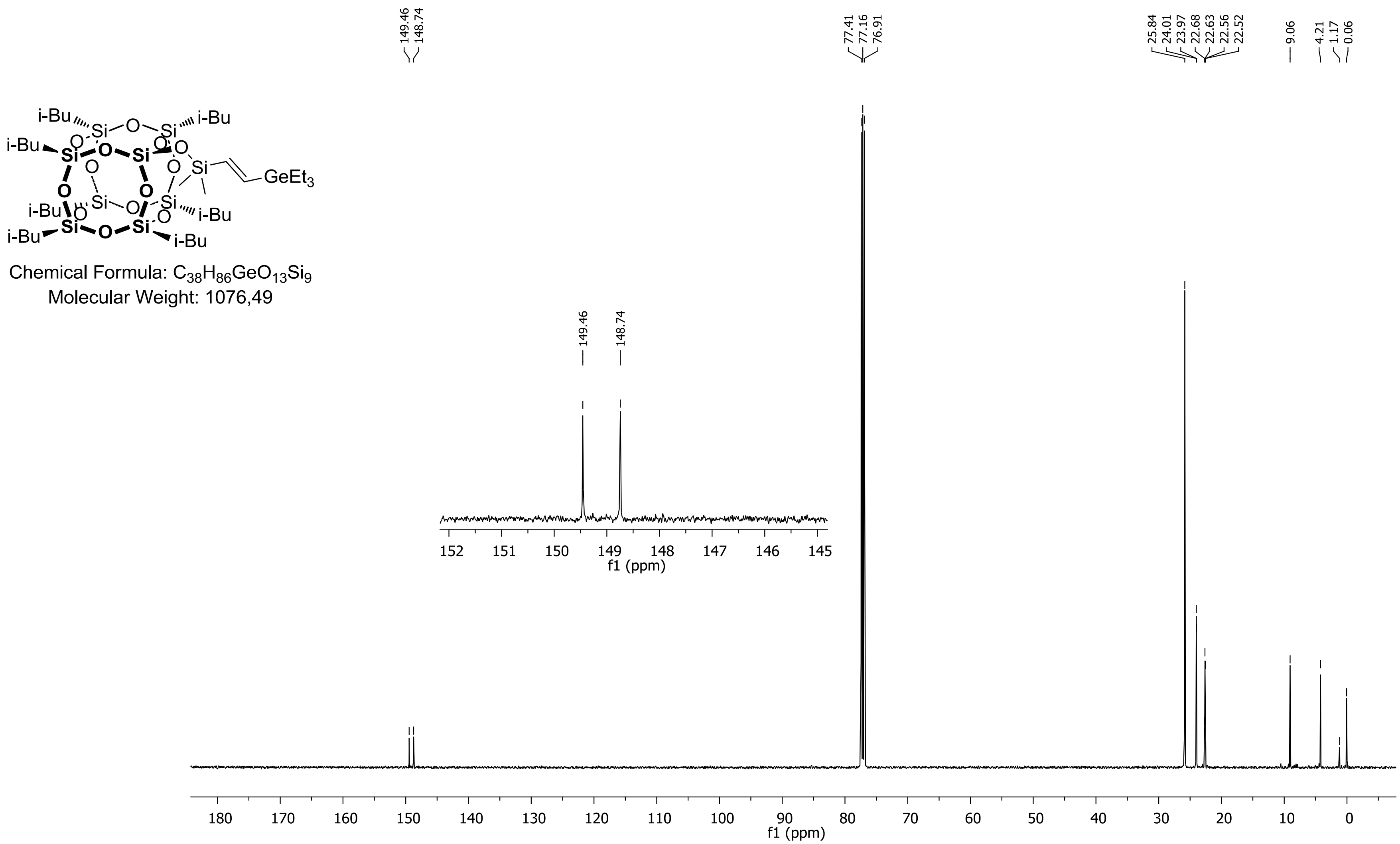


Figure 38. ^{13}**C NMR of compound 3i.**

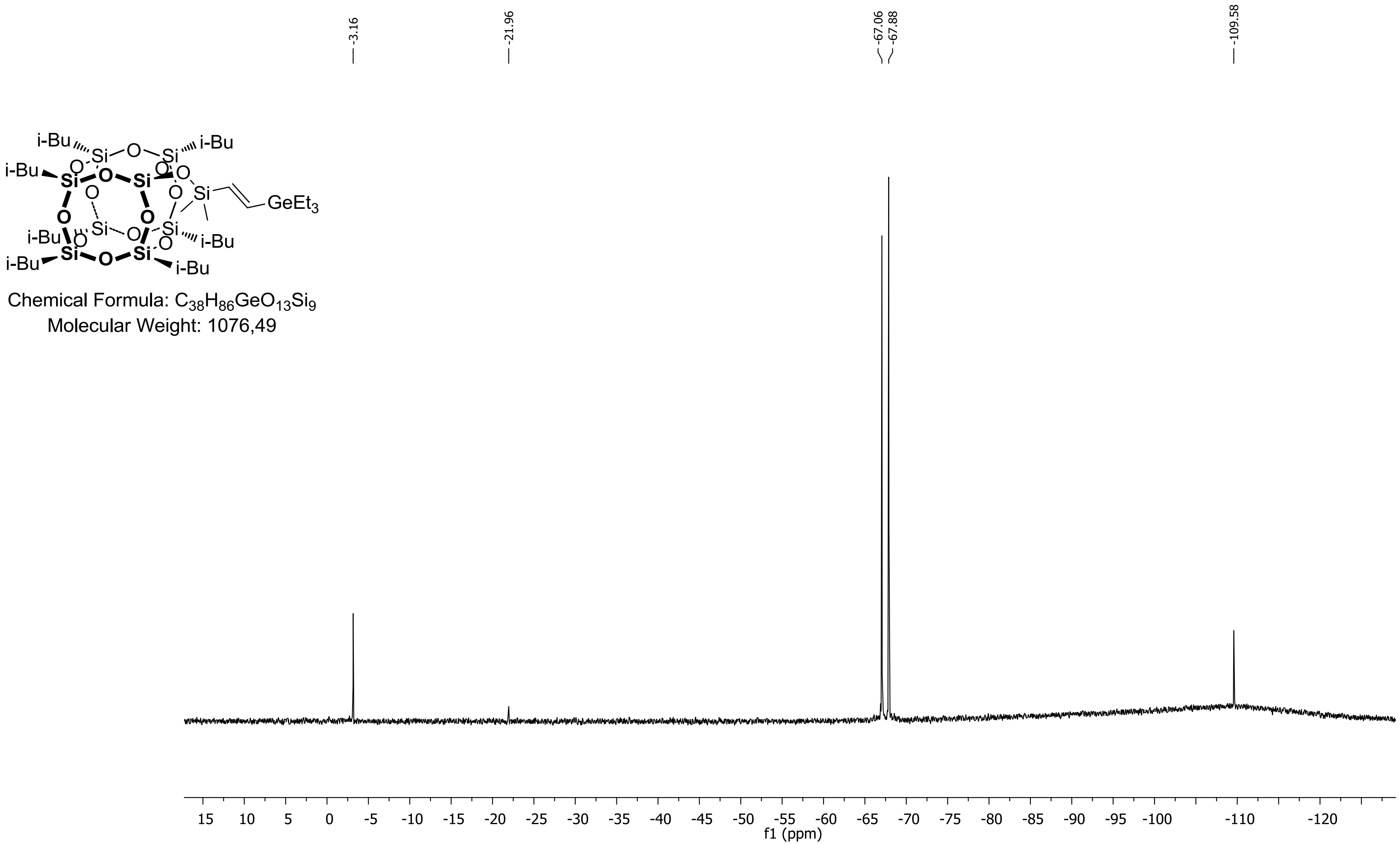


Figure 39. ^{29}**Si NMR of compound 3i.**

3.11 Hydrosilylation of 2j with silsesquioxane 1

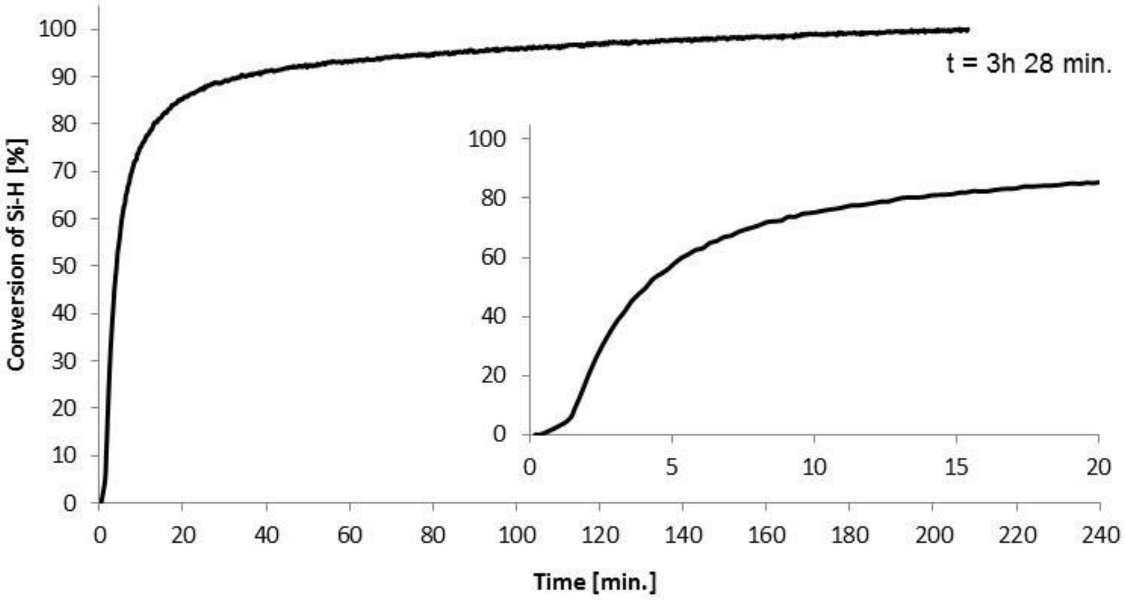
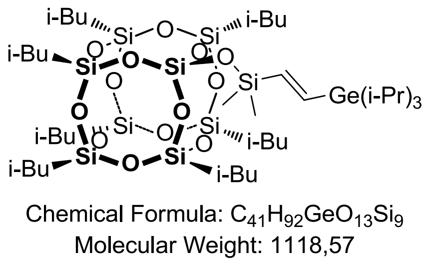


Figure 40. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2j with 1.



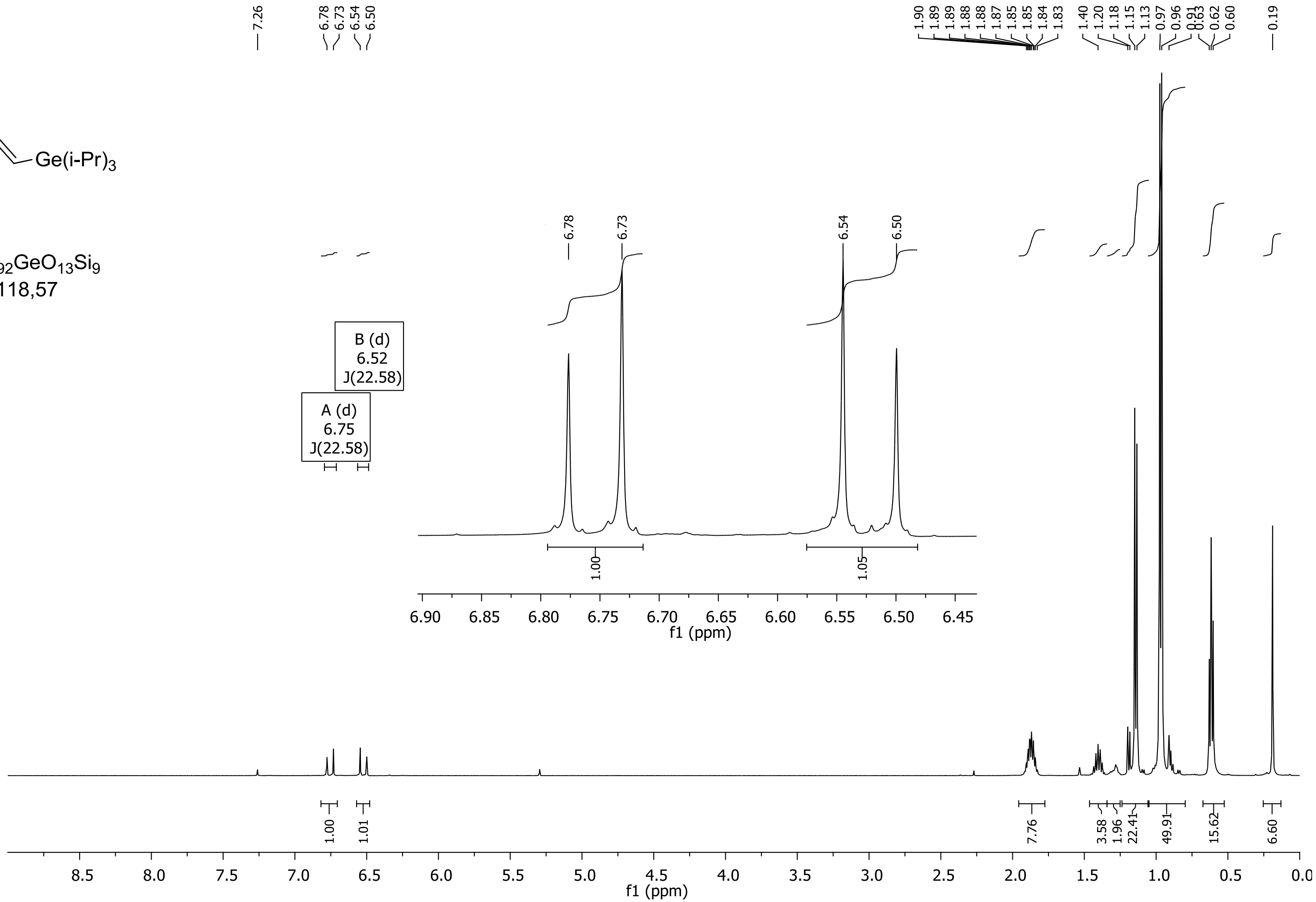


Figure 41. ^{1}H NMR of compound 3j.

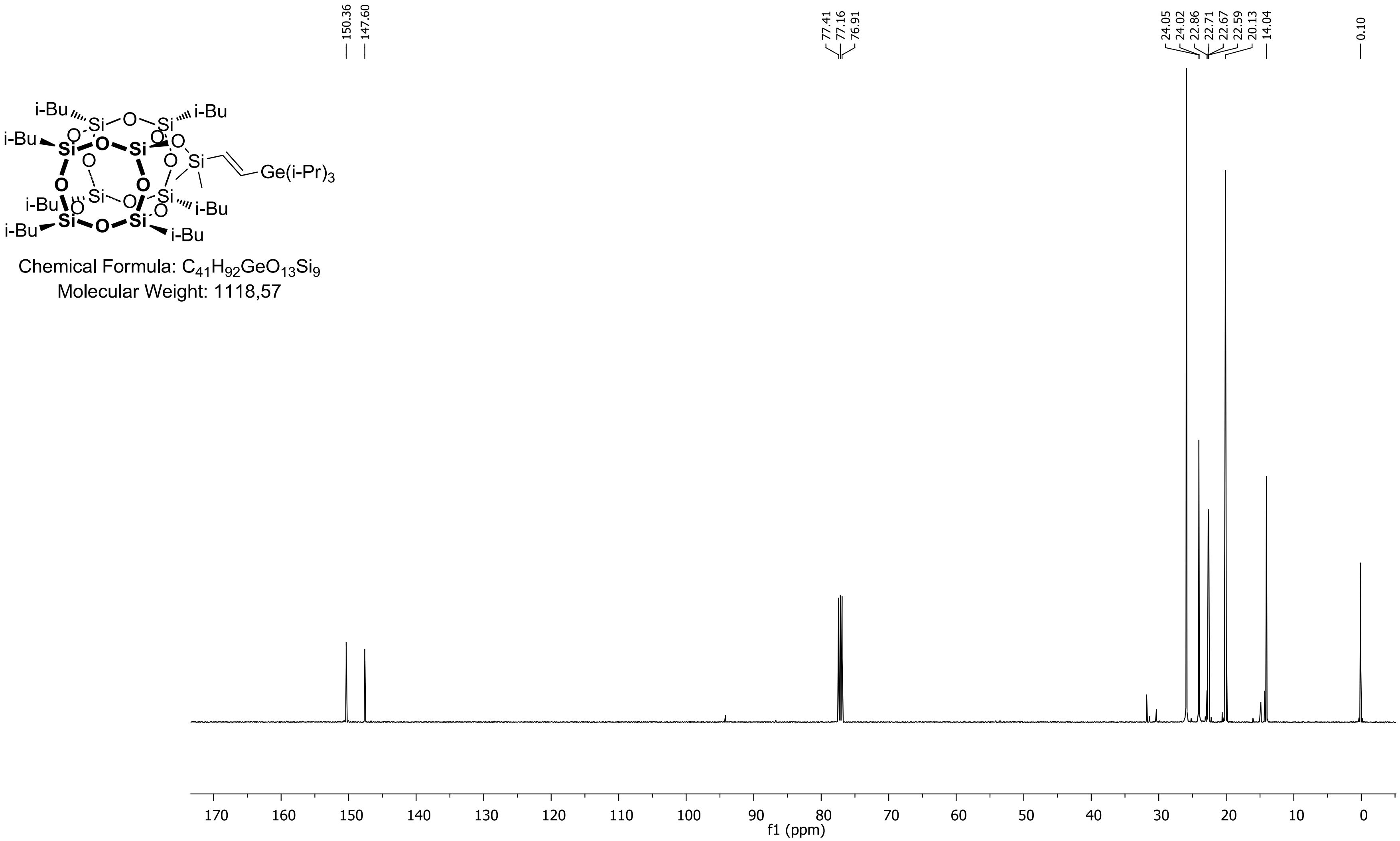


Figure 42. ^{13}**C NMR of compound 3j.**

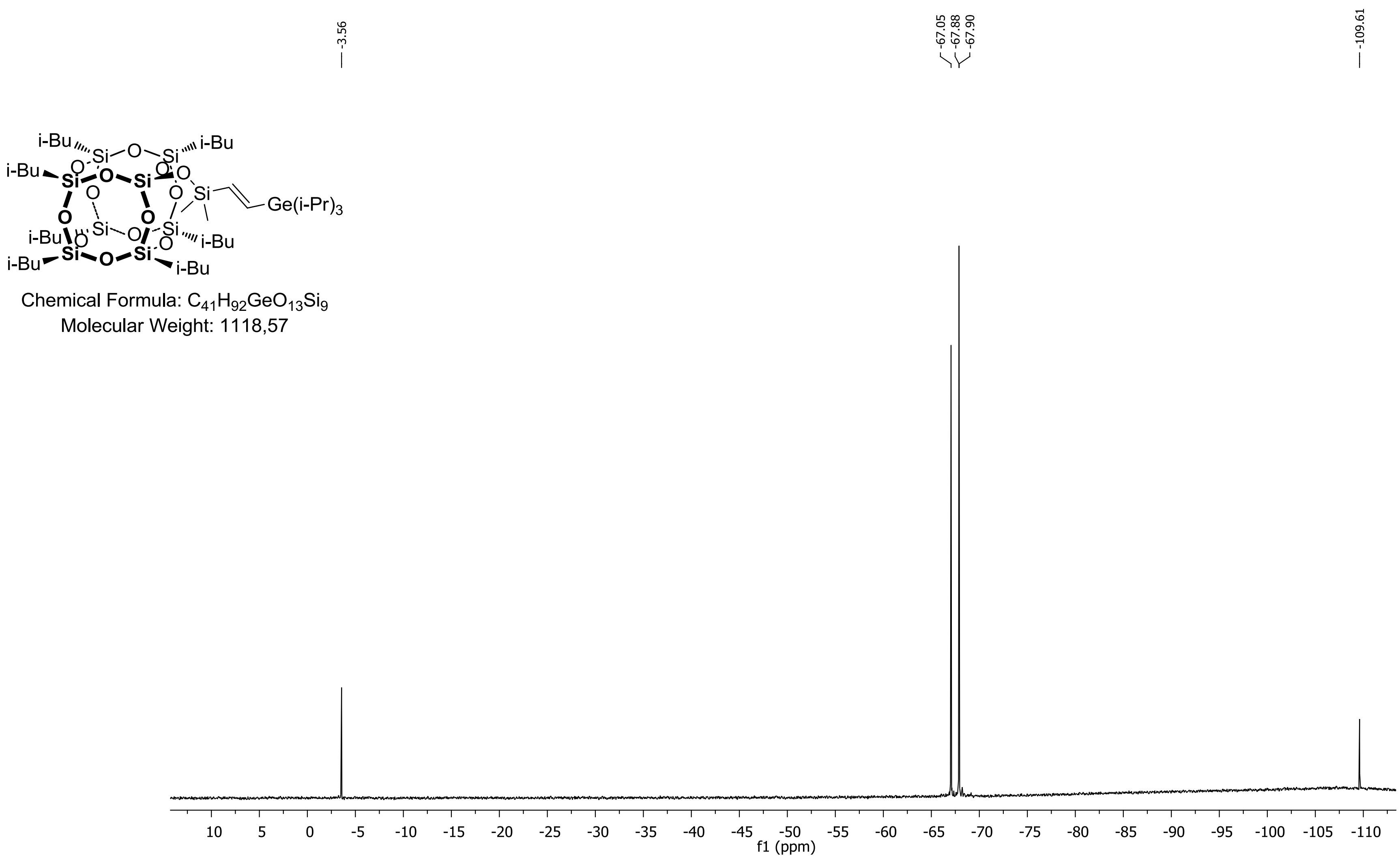


Figure 43. [29]Si NMR of compound 3j.

3.12 Hydrosilylation of 2k with silsesquioxane 1

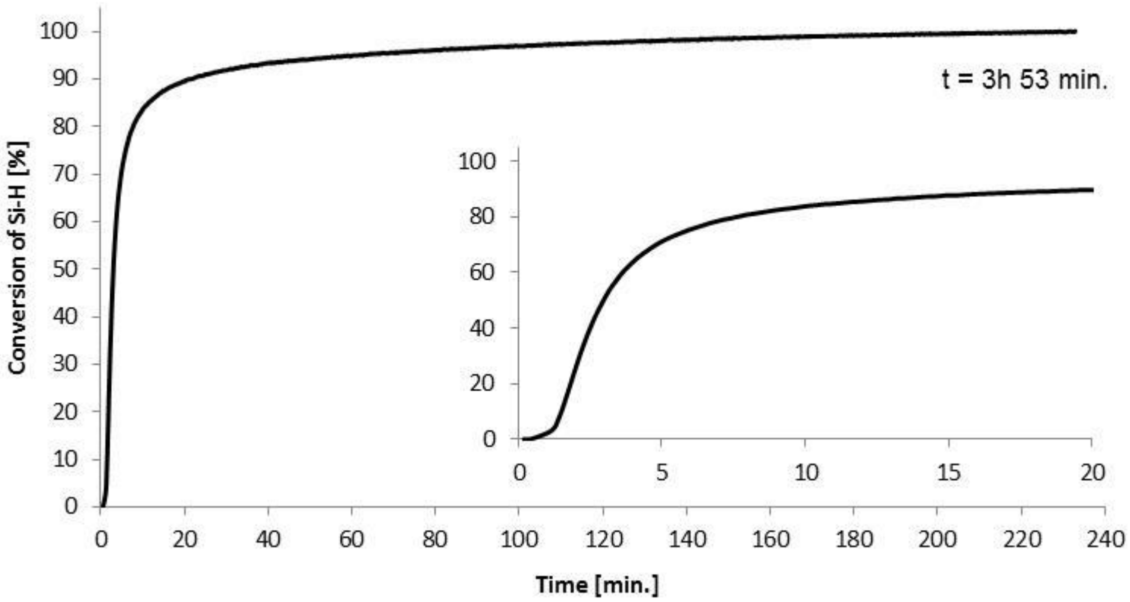
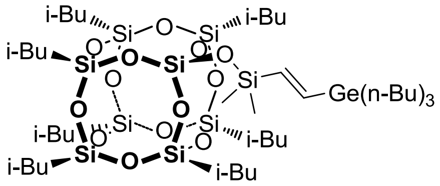


Figure 44. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2k with 1.



Chemical Formula: $C_{44}H_{98}GeO_{13}Si_9$
Molecular Weight: 1160,65

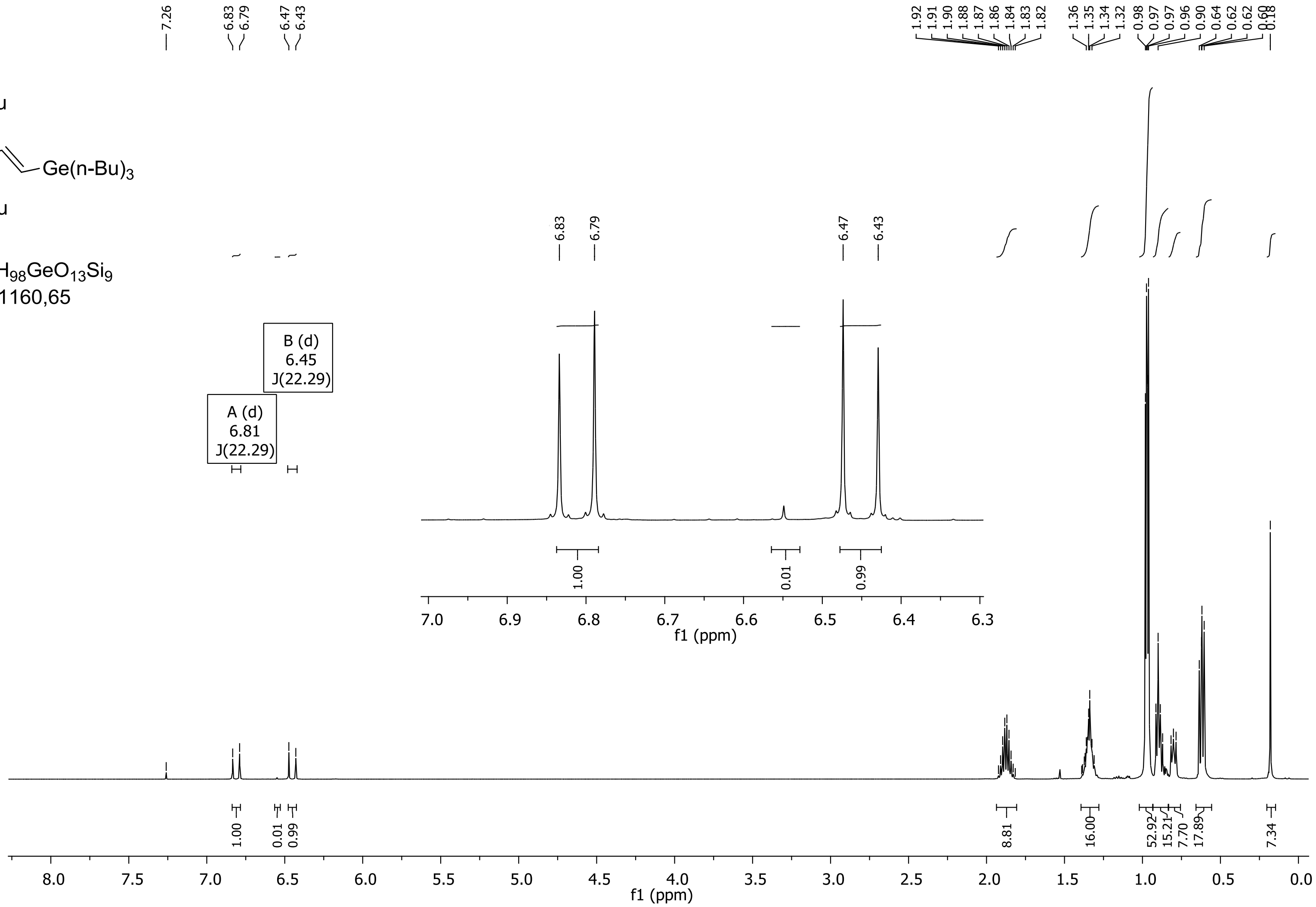
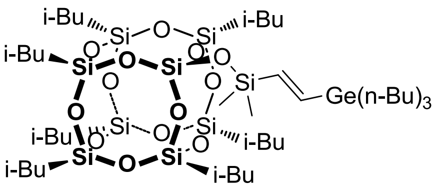


Figure 45. ^{1}H NMR of compound 3k.



Chemical Formula: $C_{44}H_{98}GeO_{13}Si_9$
Molecular Weight: 1160,65

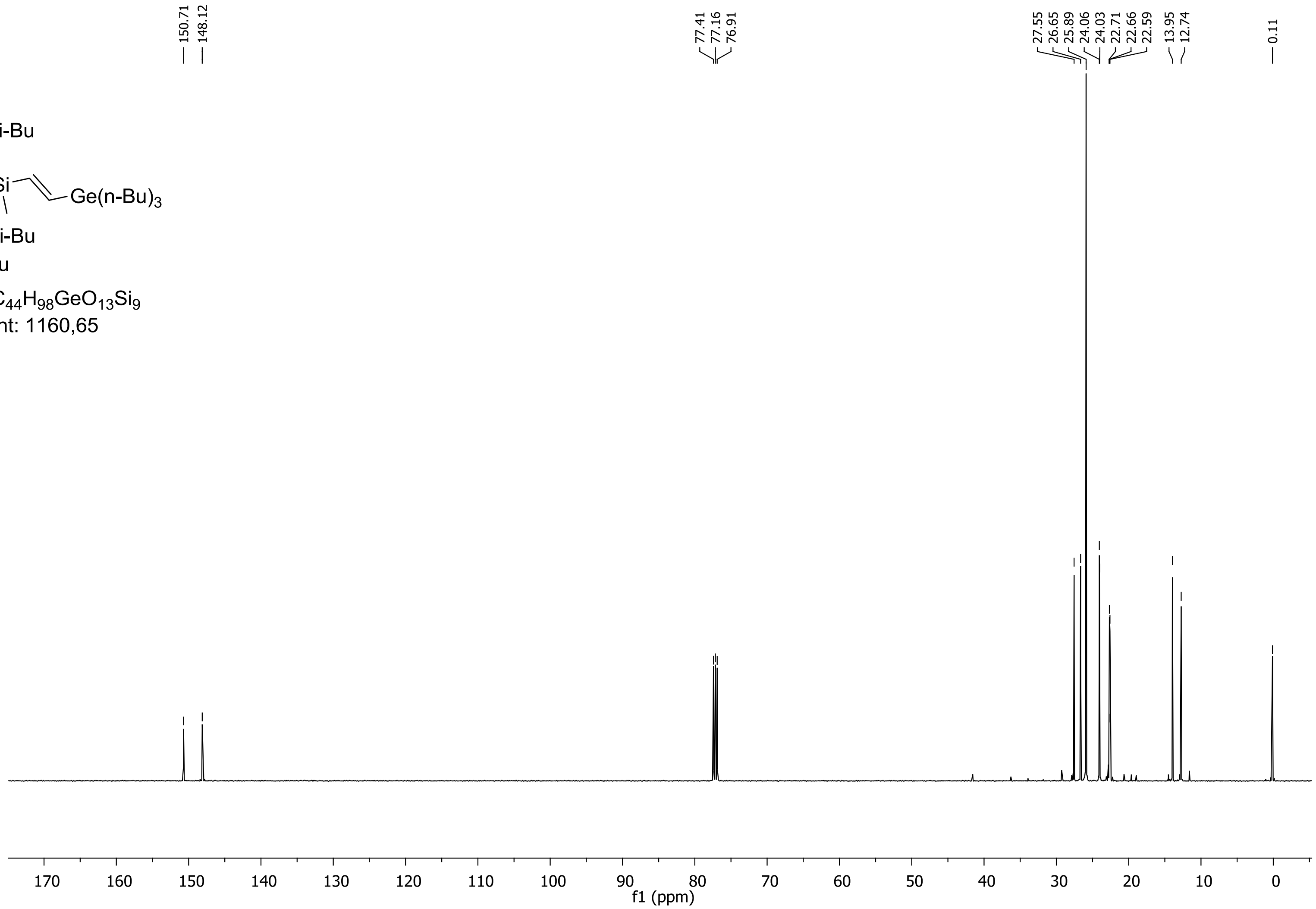


Figure 46. [13]C NMR of compound 3k.

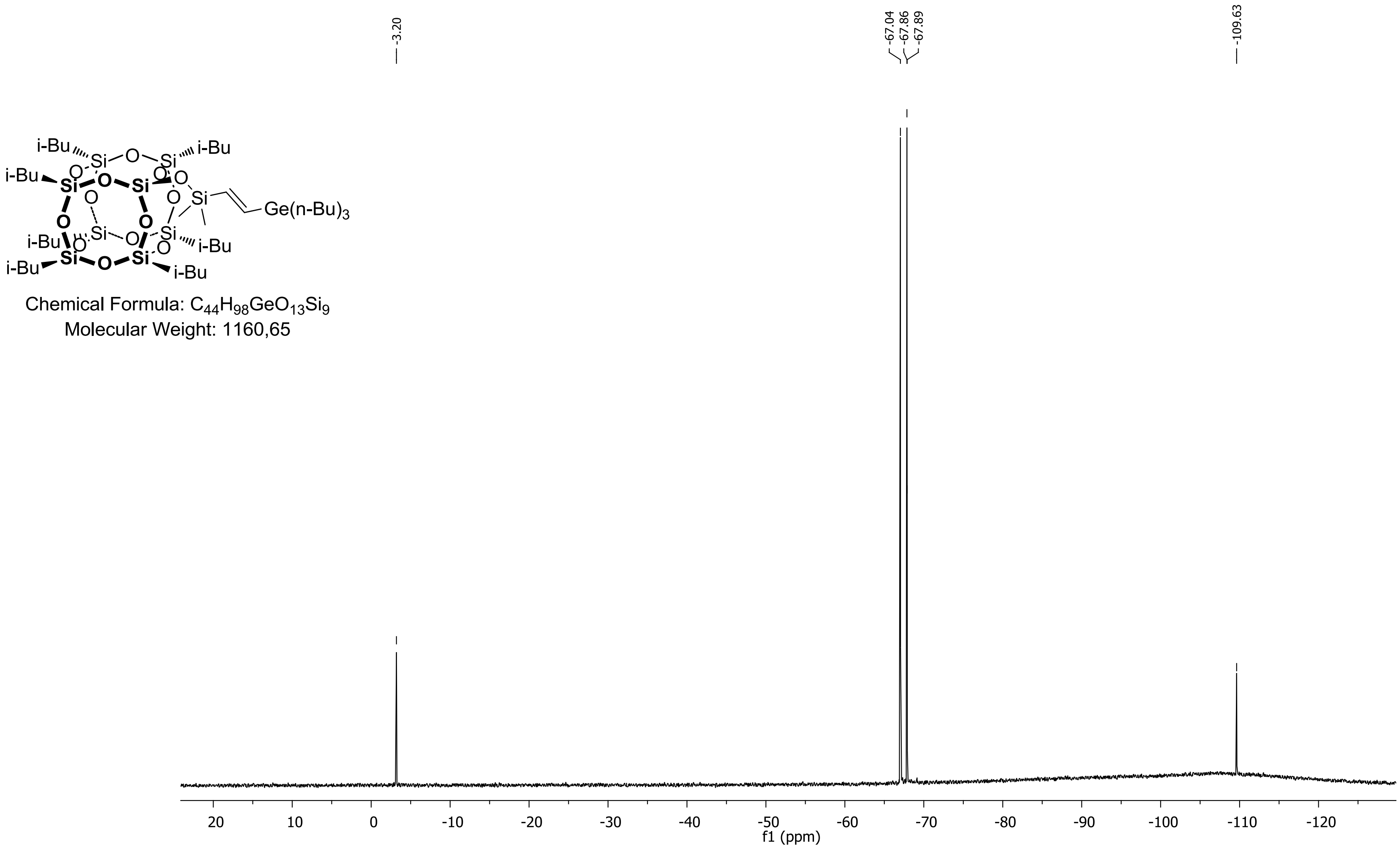


Figure 47. ^{29}Si NMR of compound 3k

3.13 Hydrosilylation of 2l with silsesquioxane 1

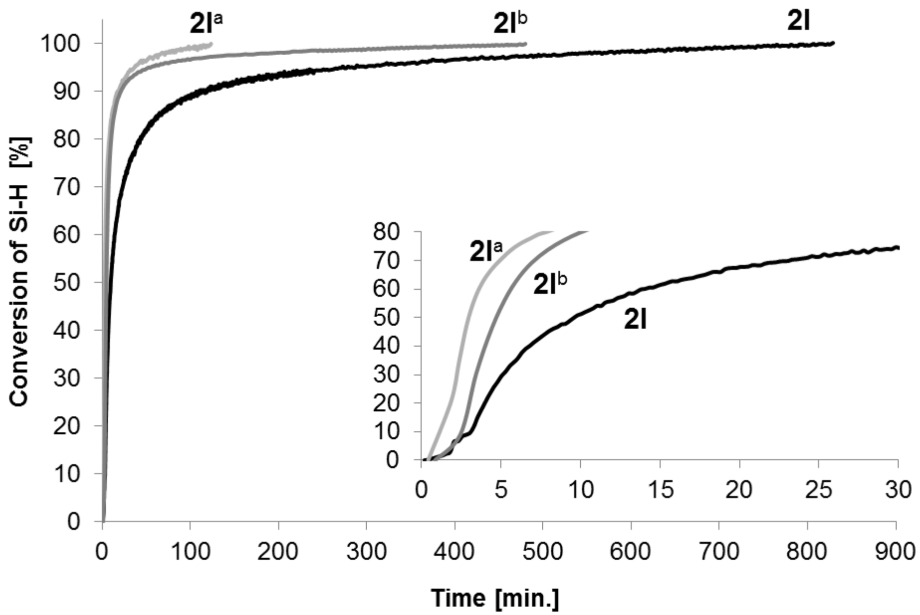


Figure 48. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2l with 1 - 10^{-4} mol of Pt-catalyst, $ms_1/V_{tol.}$ = 50mg/1ml, 100°C; [a] 10^{-3} mol of Pt-catalyst, $ms_1/V_{tol.}$ = 50mg/1ml. 100°C; [b] 10^{-4} mol of Pt-catalyst, $ms_1/V_{tol.}$ = 500mg/1ml, 100°C.

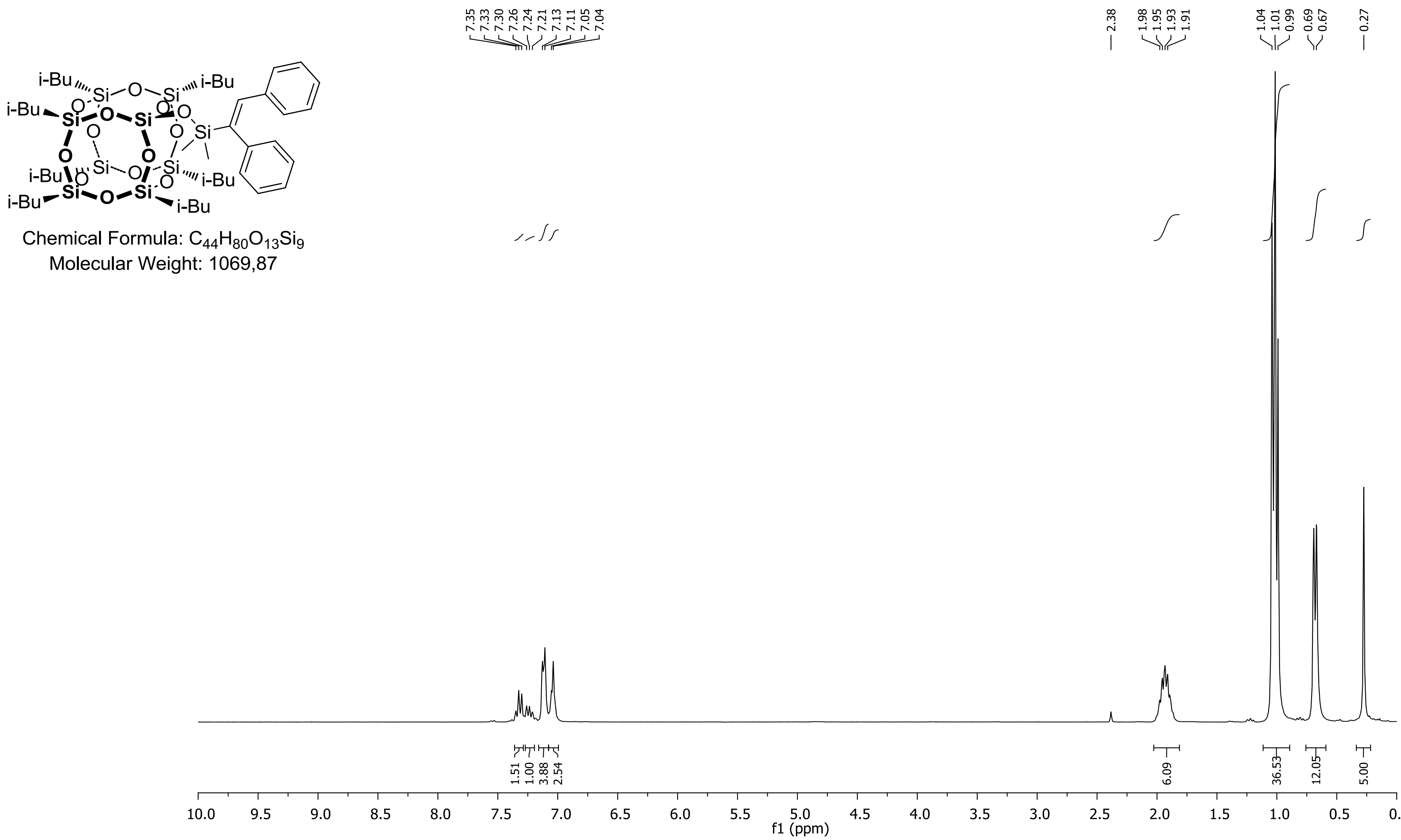
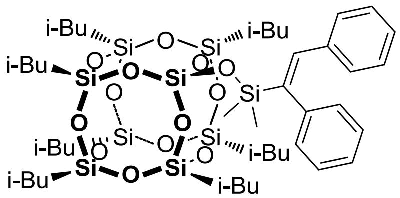


Figure 49. ^{1}H NMR of compound 3l.



Chemical Formula: $C_{44}H_{80}O_{13}Si_9$
Molecular Weight: 1069,87

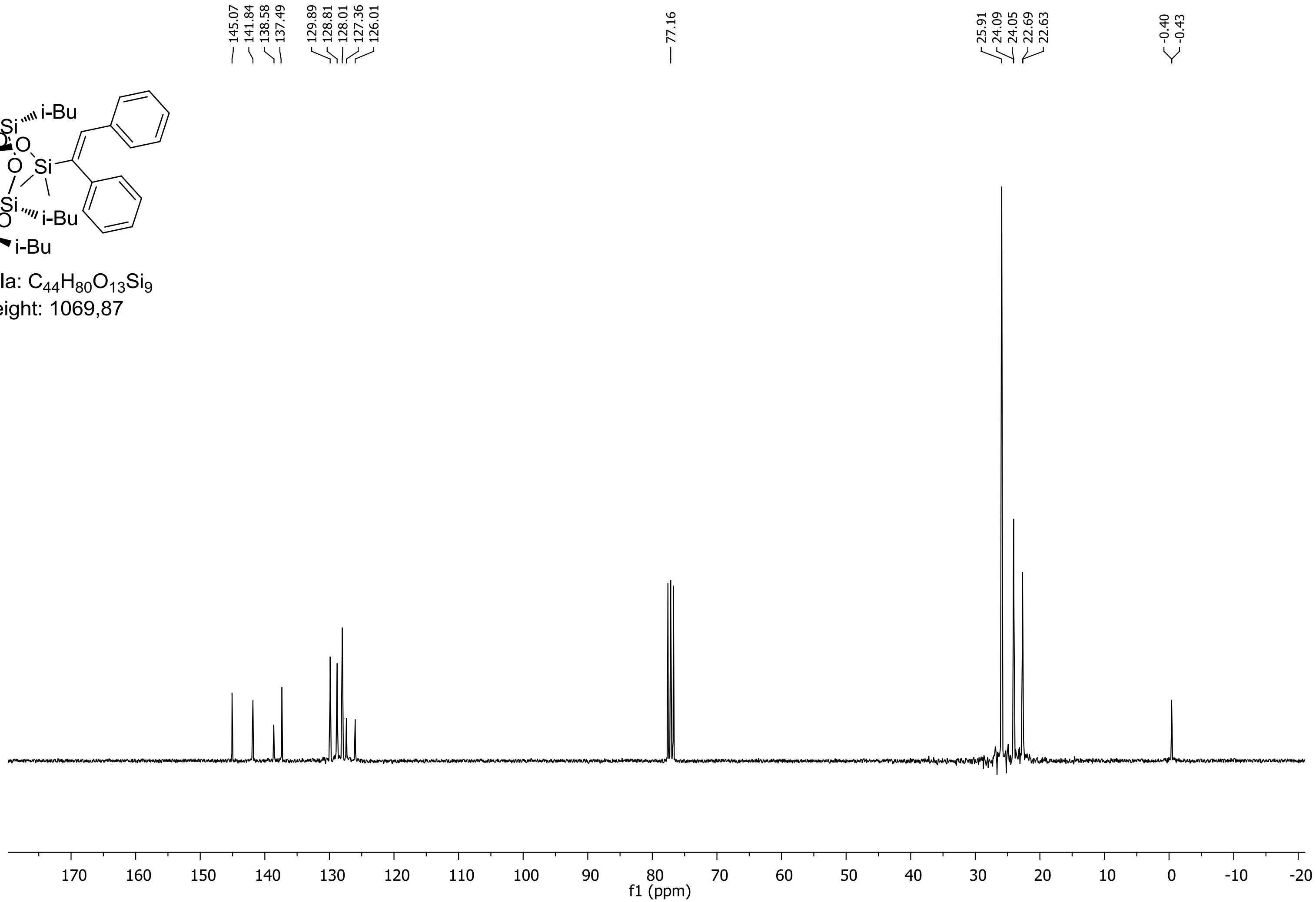


Figure 50. ^{13}C NMR of compound 3l.

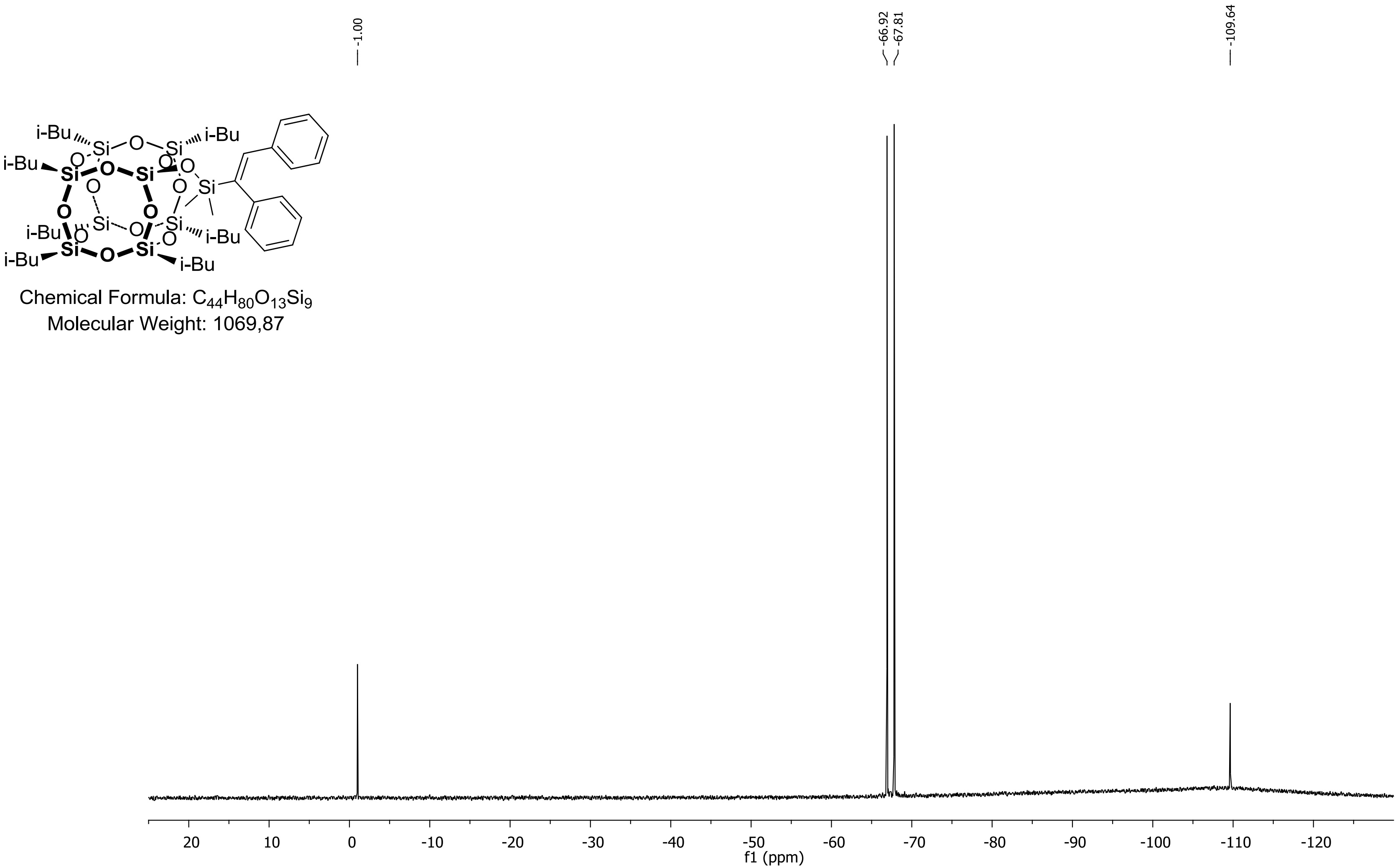


Figure 51. ^{29}**Si NMR of compound 3l.**

3.14 Hydrosilylation of 2m with silsesquioxane 1

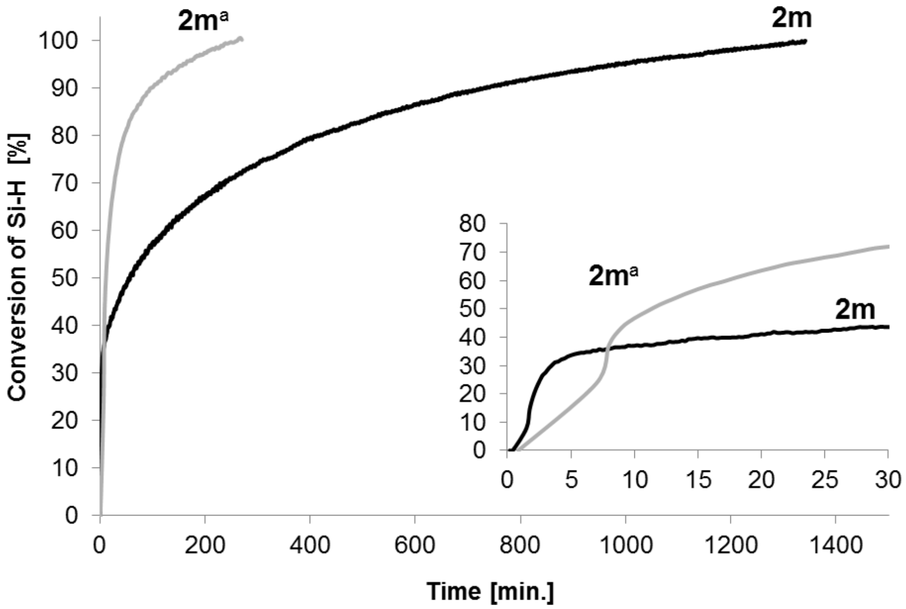
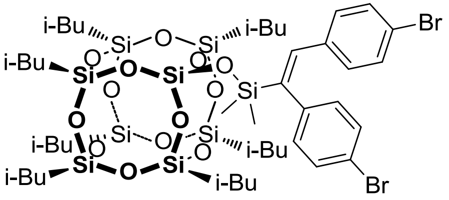


Figure 52. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2m with 1 - 10^{-4} mol of Pt-catalyst, $ms_1/V_{tol.}$ = 50mg/1ml, 100°C; [a] 10^{-3} mol of Pt-catalyst, $ms_1/V_{tol.}$ = 50mg/1ml, 100°C.



Chemical Formula: $C_{44}H_{78}Br_2O_{13}Si_9$

Molecular Weight: 1227,66

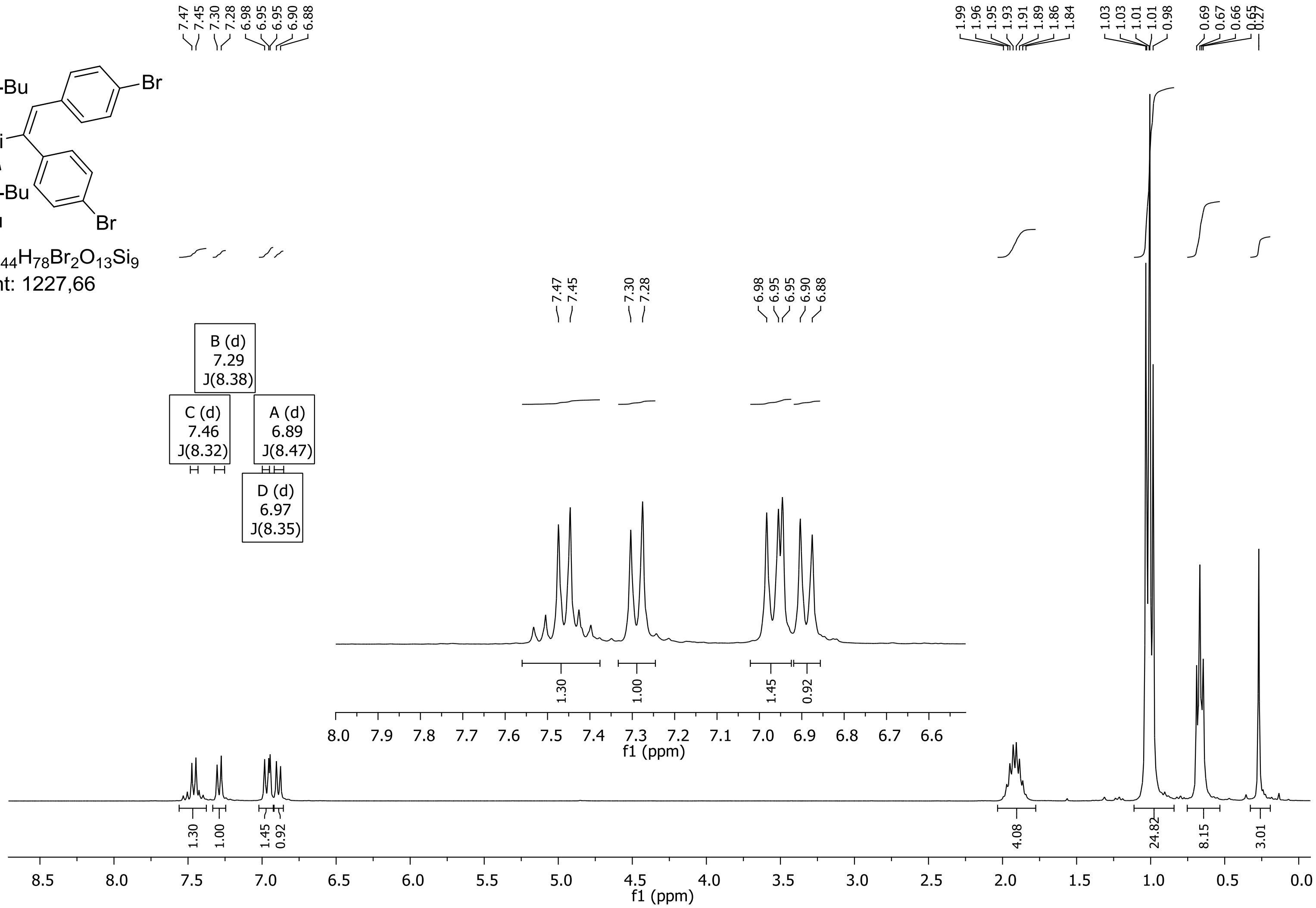
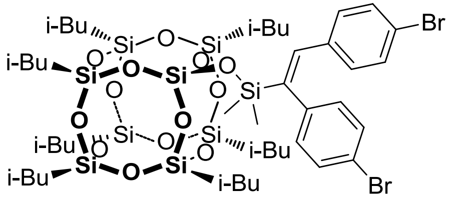


Figure 53. ^{1}H NMR of compound 3m.



Chemical Formula: $C_{44}H_{78}Br_2O_{13}Si_9$
Molecular Weight: 1227,66

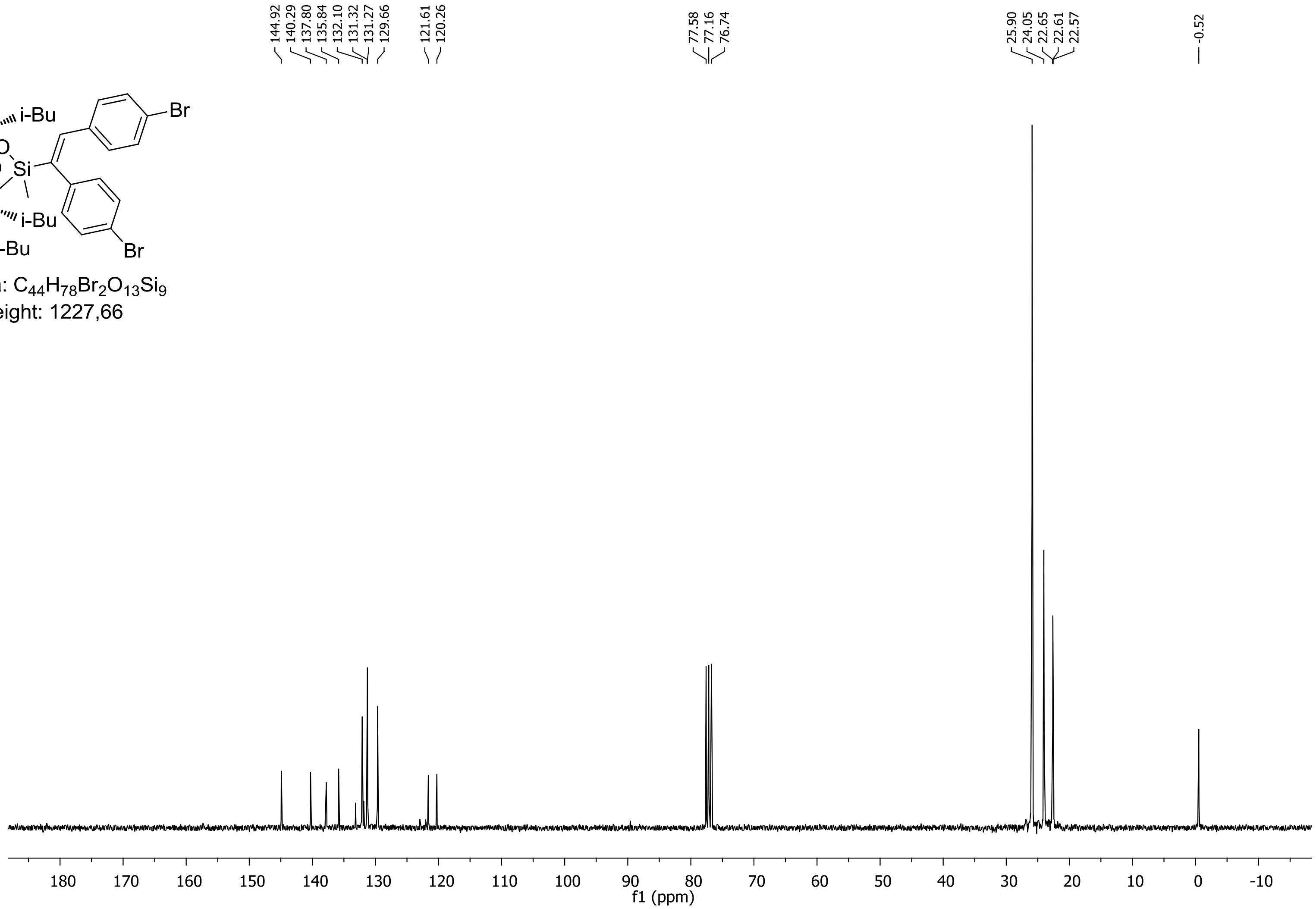


Figure 54. ^{13}C NMR of compound 3m.

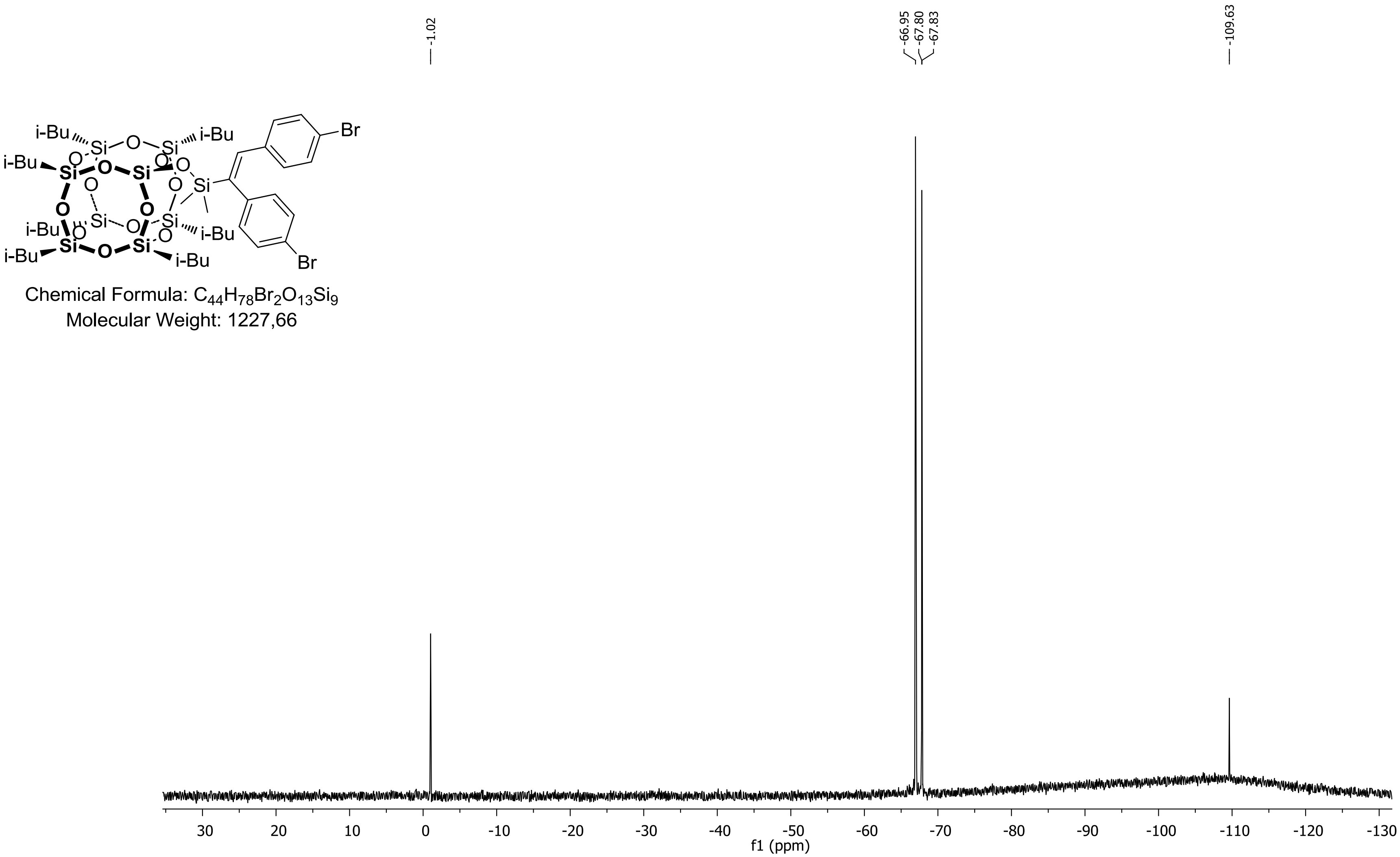


Figure 55. ^{29}Si NMR of compound 3m.

3.15 Hydrosilylation of 2n with silsesquioxane 1

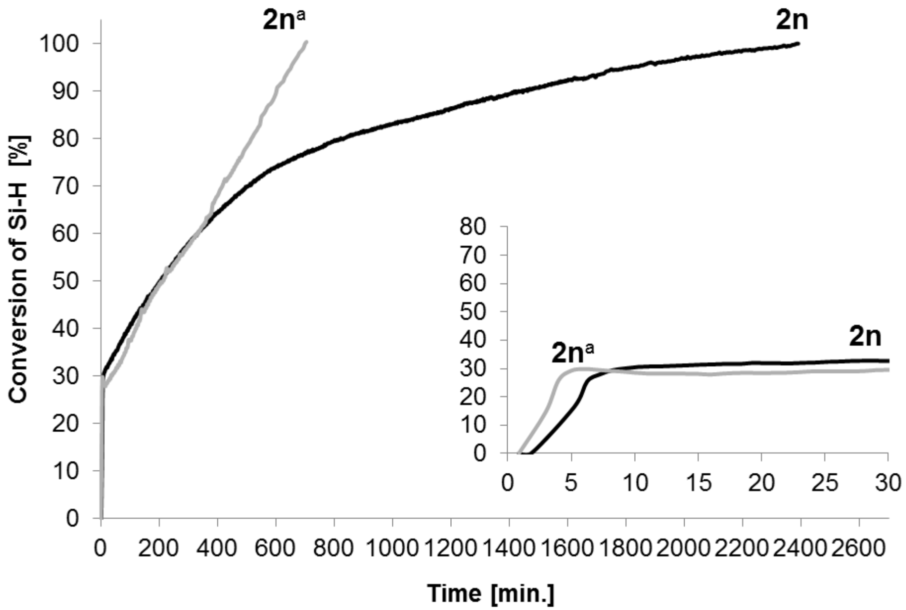


Figure 56. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2n with 1 - 10^{-4} mol of Pt-catalyst, $m_{S1}/V_{tol.}$ = 50mg/1ml, 100°C; [a] 10^{-3} mol of Pt-catalyst, $m_{S1}/V_{tol.}$ = 50mg/1ml, 100°C.

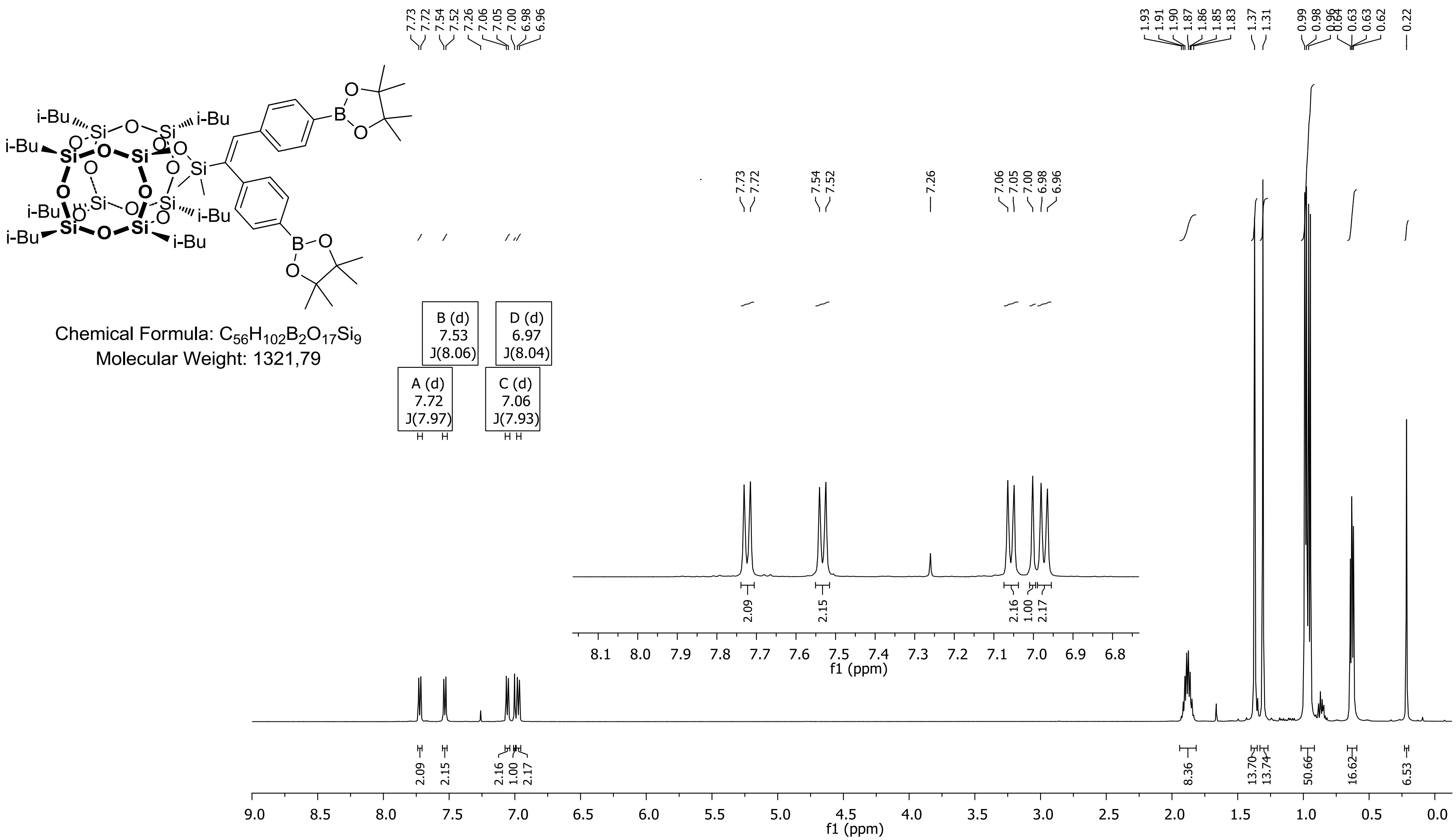


Figure 57. ^{1}H NMR of compound 3n.

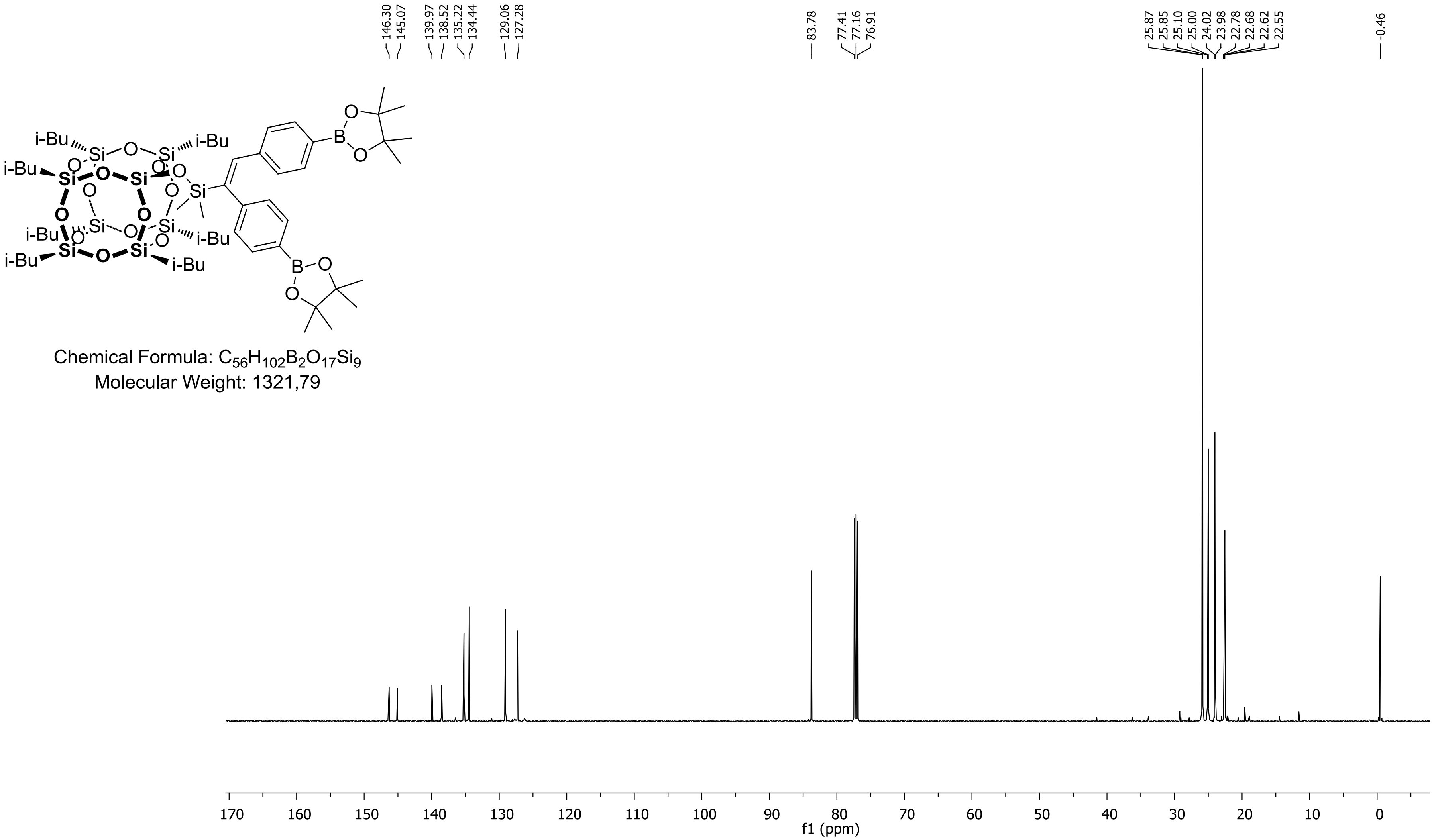


Figure 58. ^{13}**C NMR of compound 3n.**

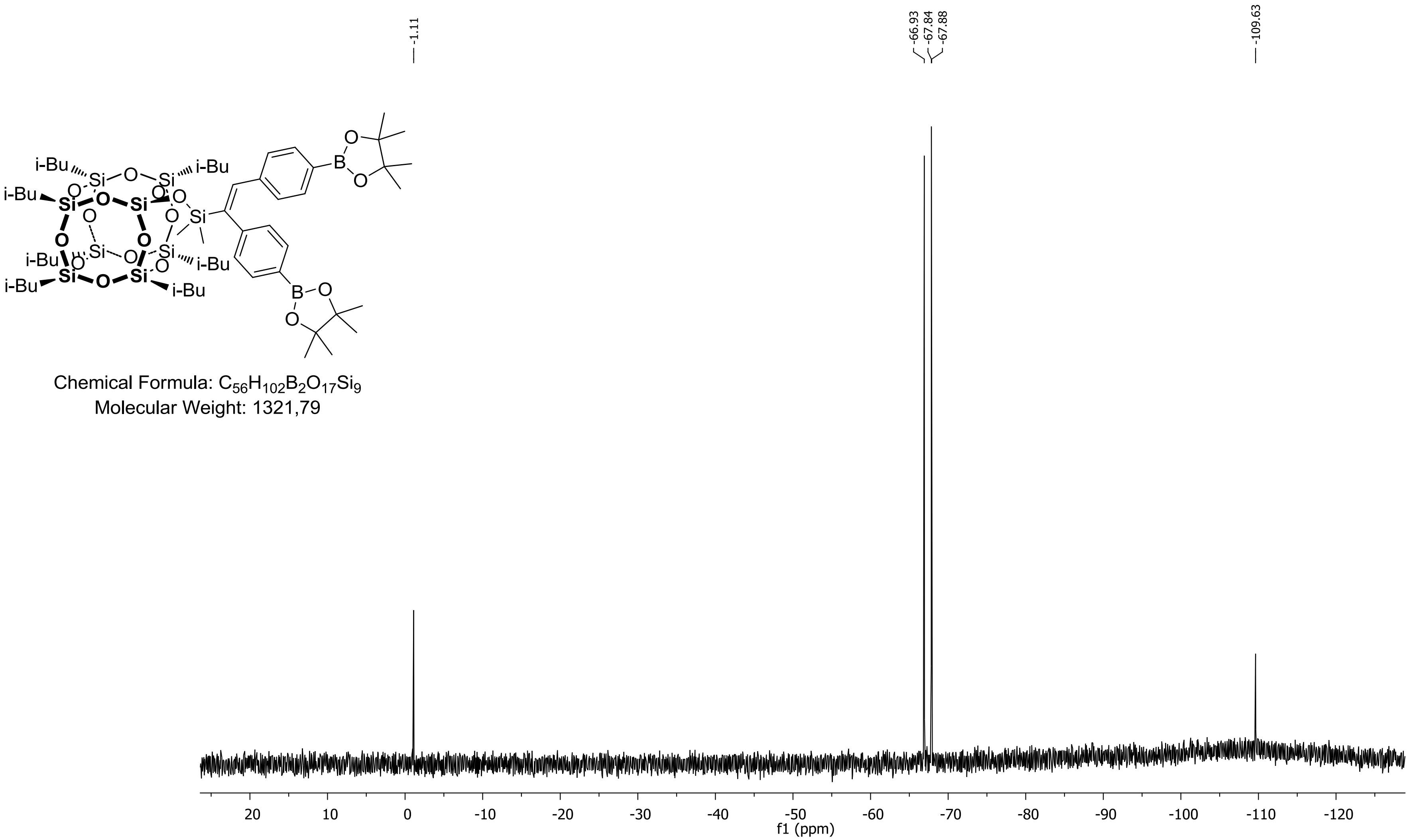


Figure 59. ^{29}Si NMR of compound 3n.

3.16 Hydrosilylation of 2o with silsesquioxane 1

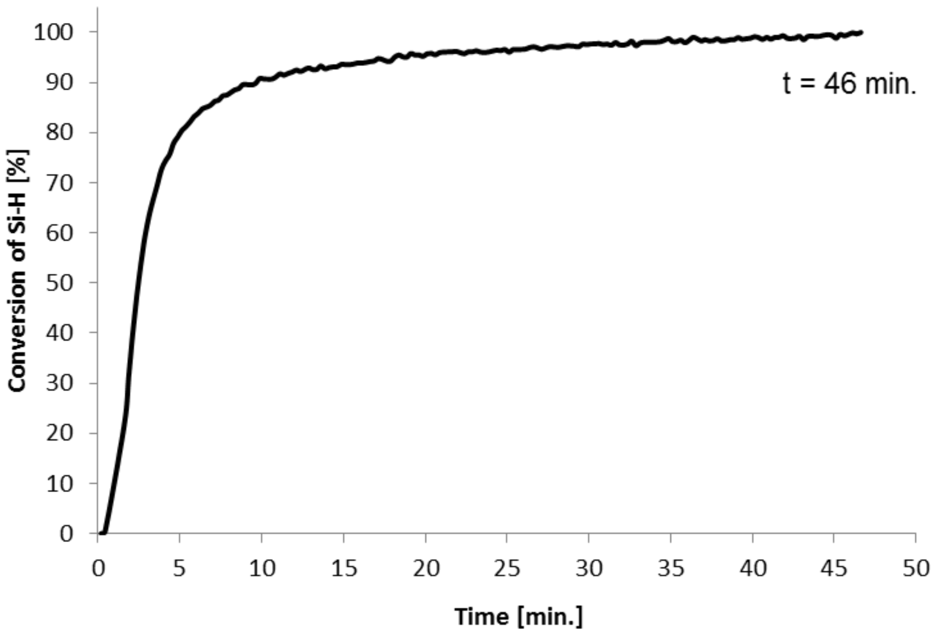


Figure 60. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2o with 1.

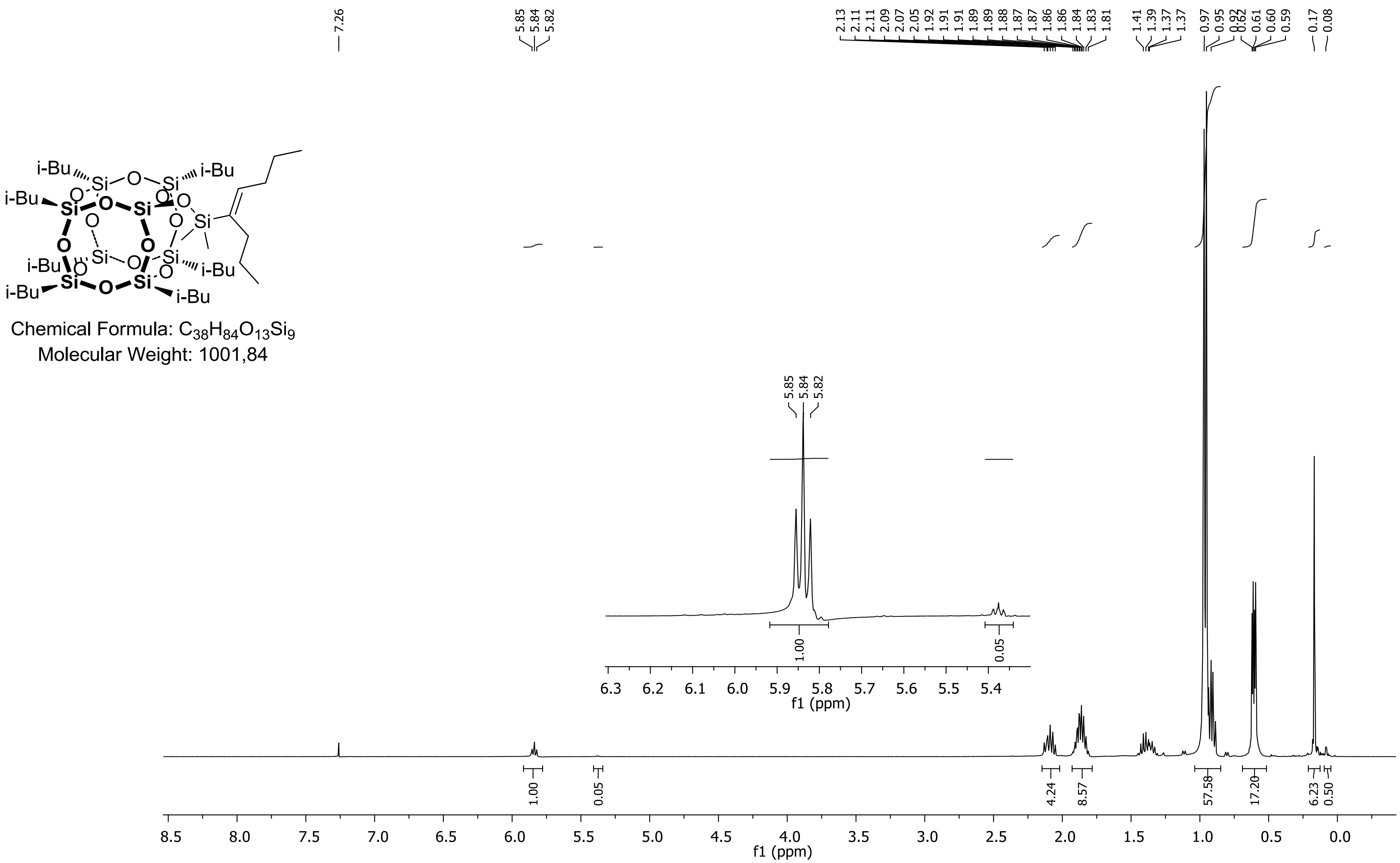
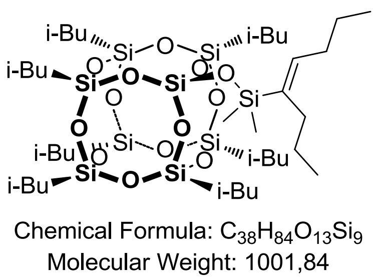


Figure 61. **^{1}H NMR of compound 3o.**



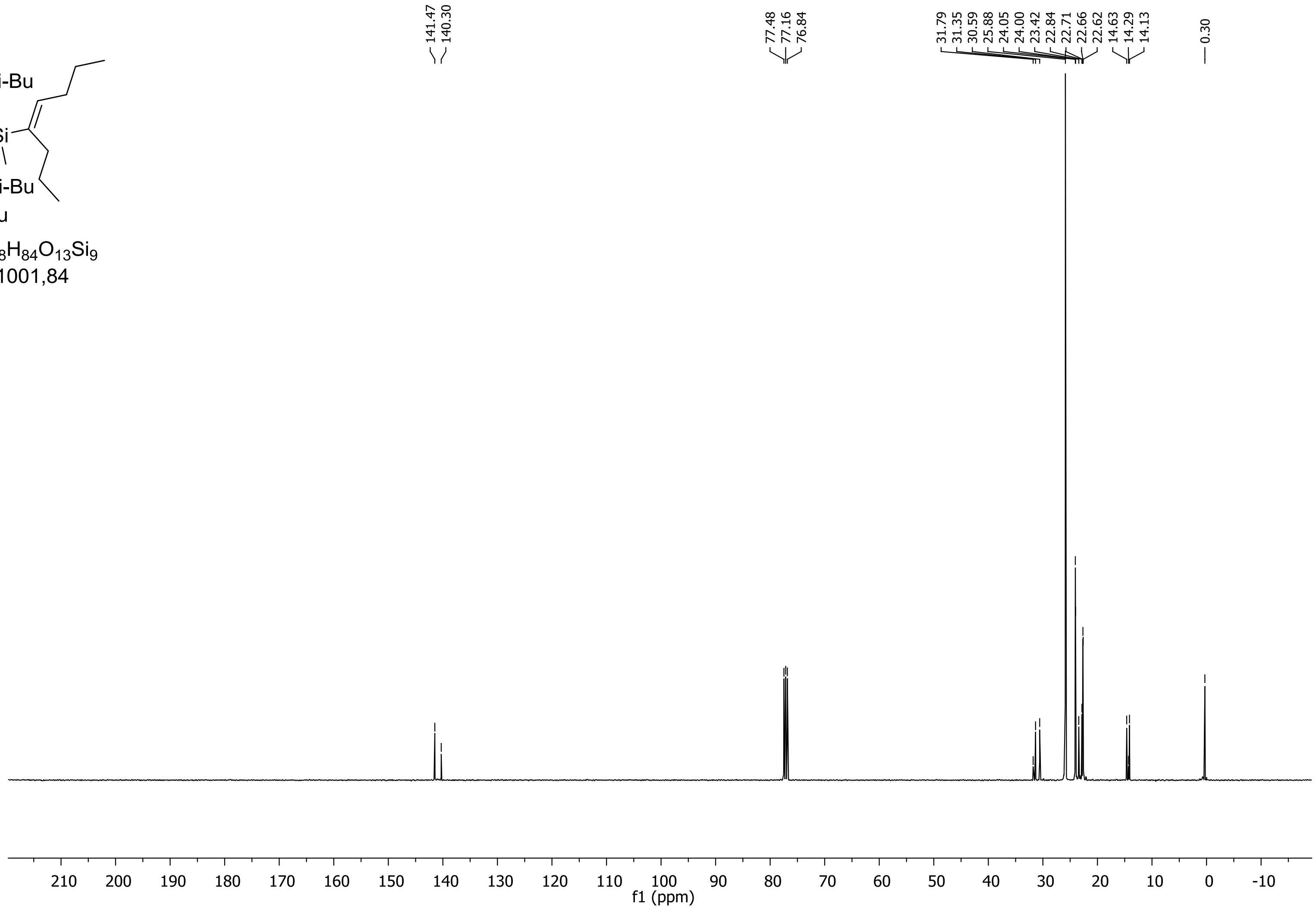


Figure 62. **^{13}C NMR of compound 3o.**

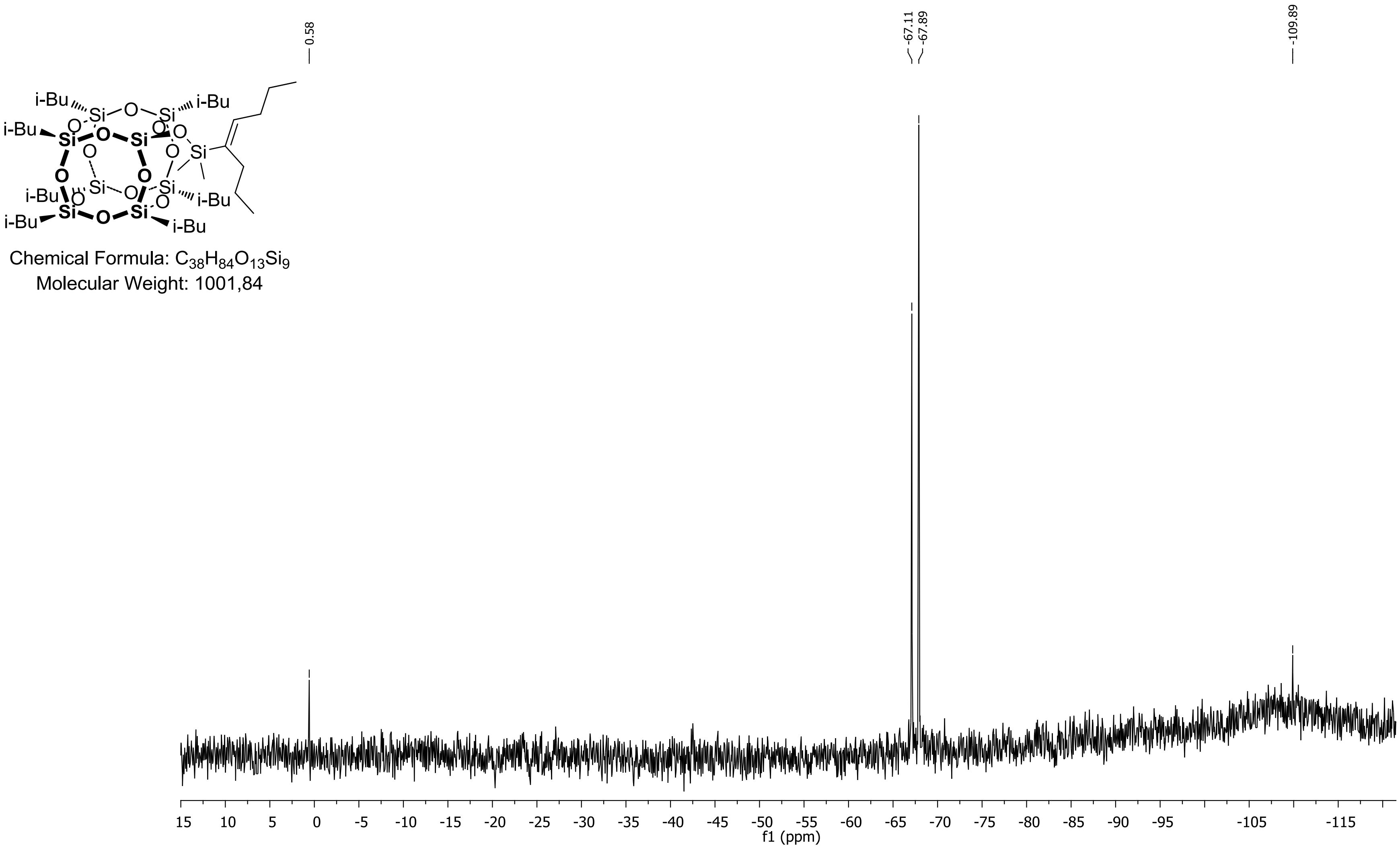


Figure 63. ^{29}Si NMR of compound 3o.

3.17 Hydrosilylation of 2p with silsesquioxane 1

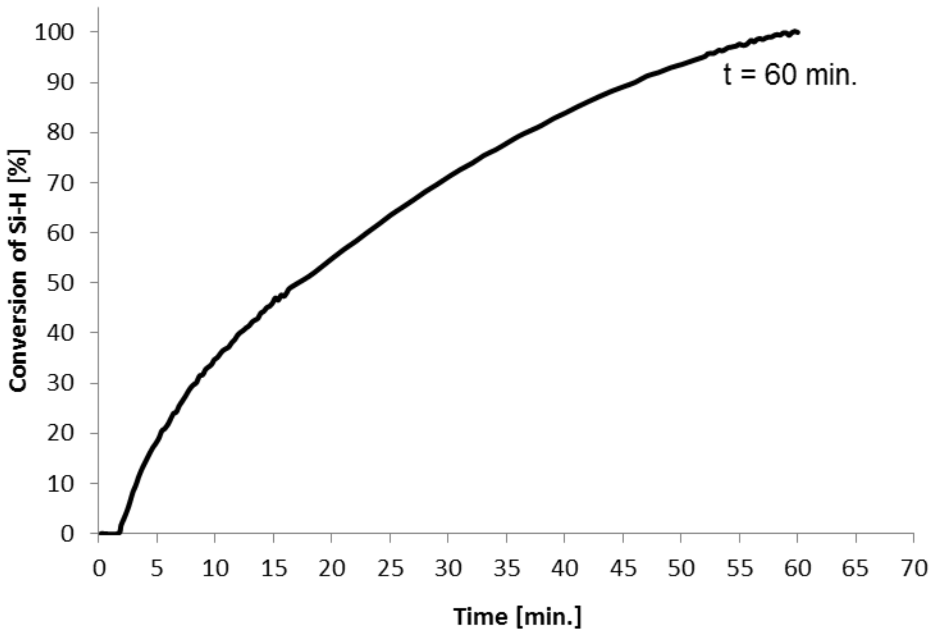
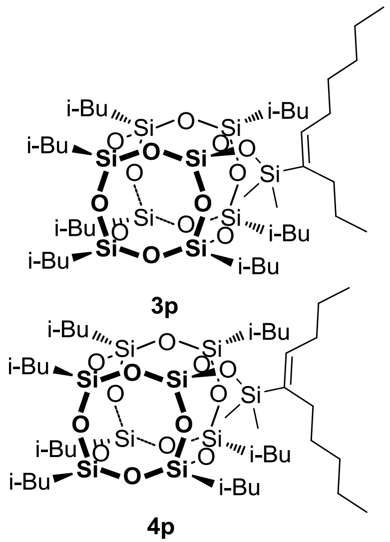


Figure 64. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2p with 1.



Chemical Formula: $C_{40}H_{88}O_{13}Si_9$
Molecular Weight: 1029,89

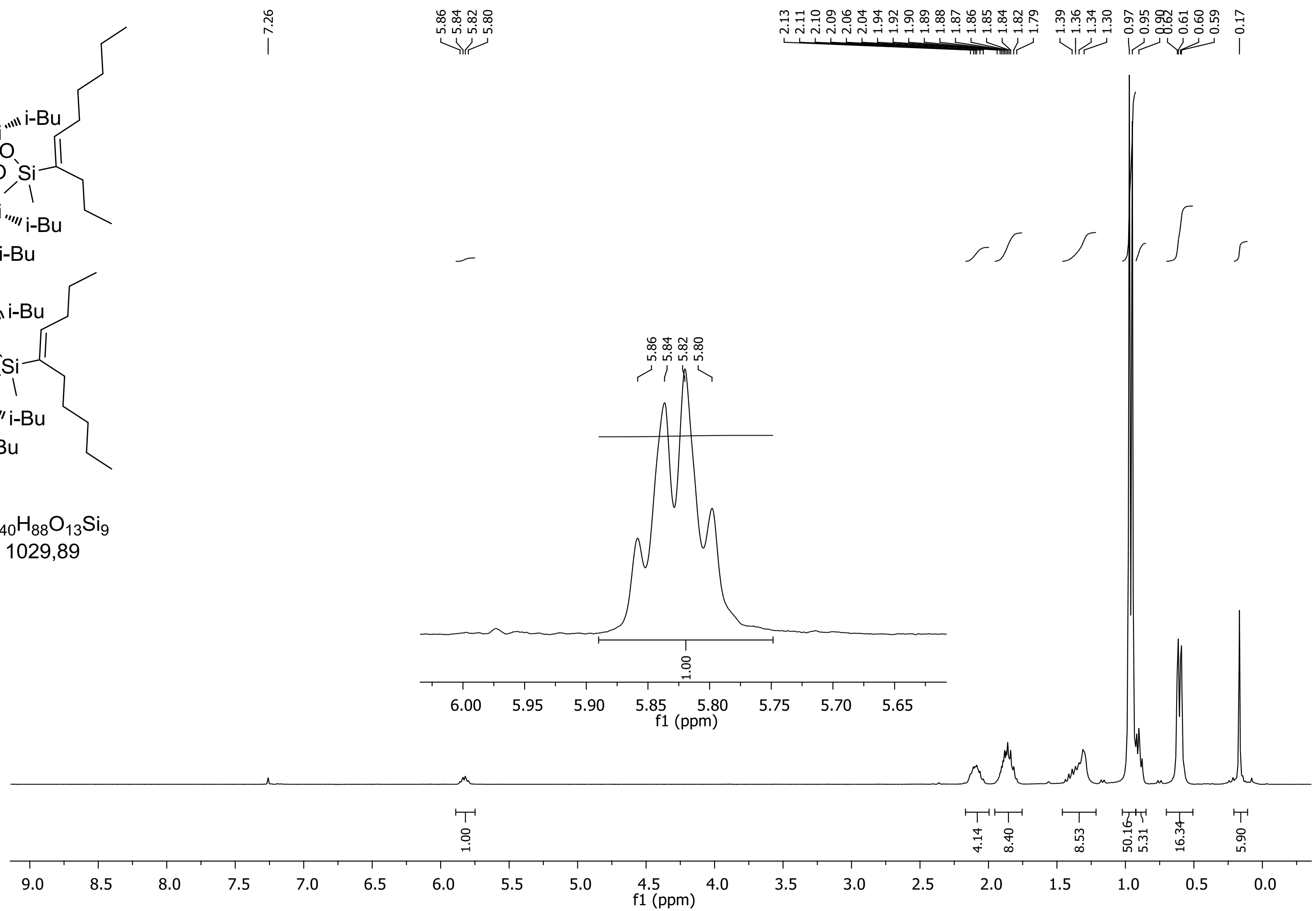
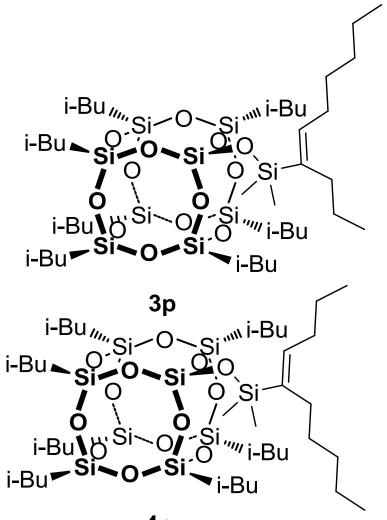


Figure 65. [1]H NMR of compound 3p.



3p

4p

Chemical Formula: $C_{40}H_{88}O_{13}Si_9$
Molecular Weight: 1029,89

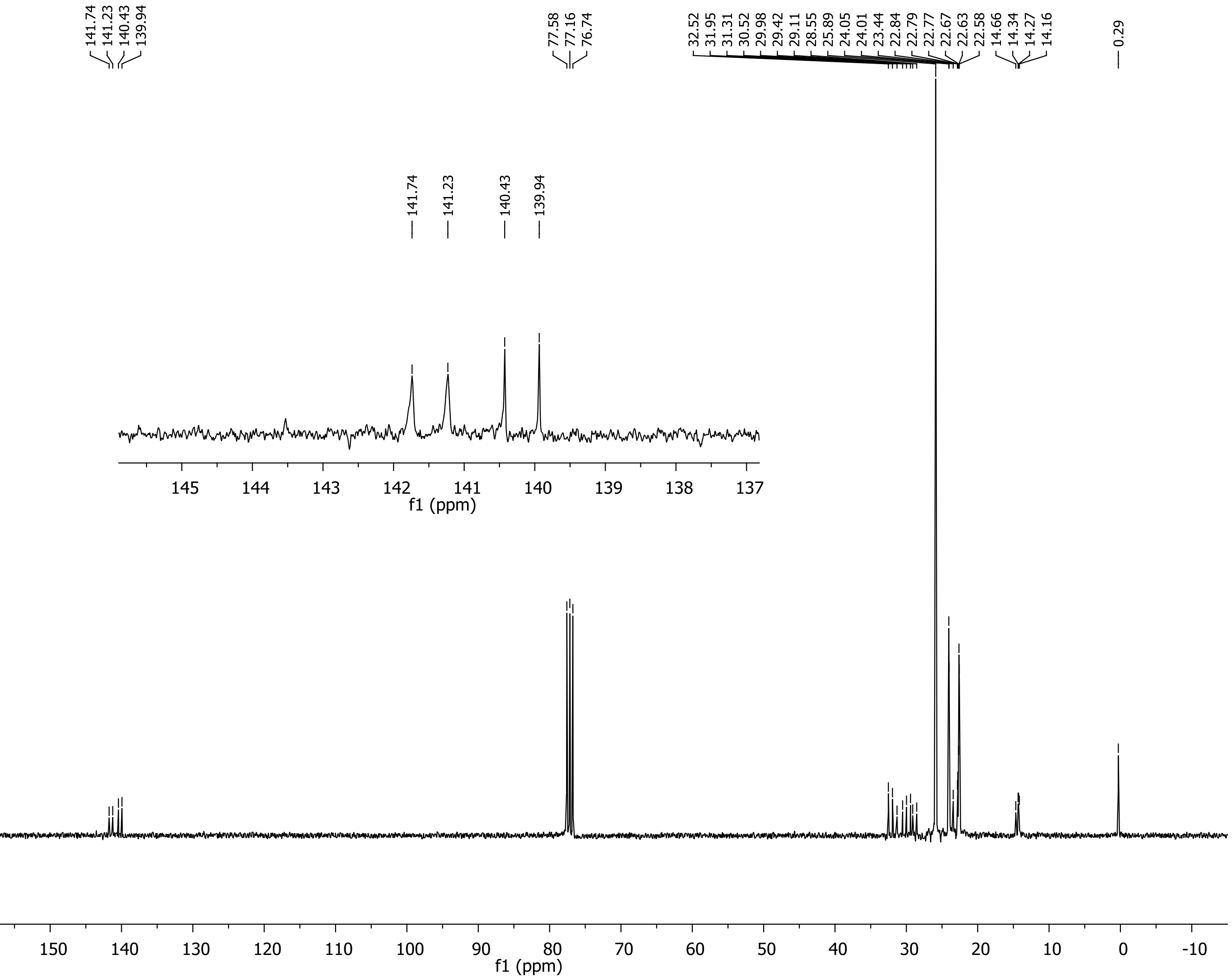
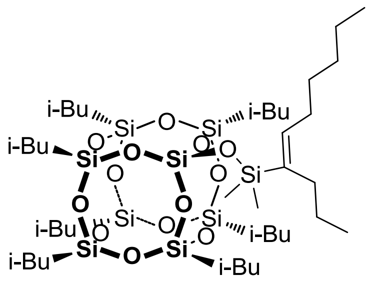
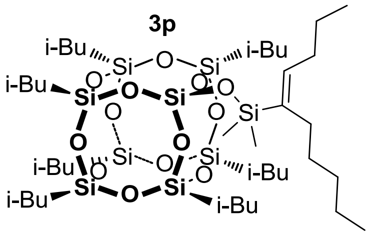


Figure 66. ^{13}C NMR of compound 3p.



3p



4p

Chemical Formula: $C_{40}H_{88}O_{13}Si_9$
Molecular Weight: 1029,89

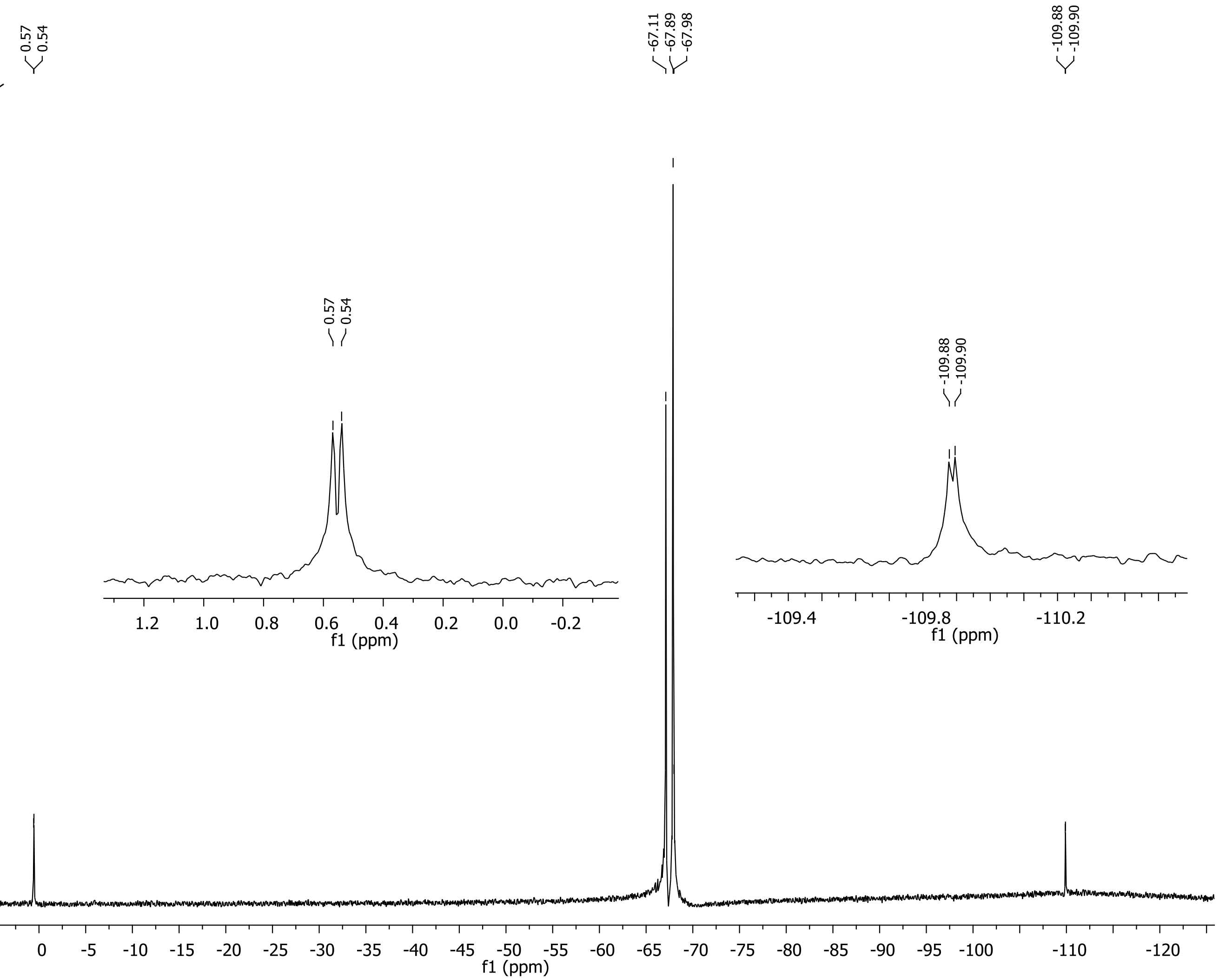


Figure 67. ^{29}Si NMR of compound 3p.

3.18 Hydrosilylation of 2q with silsesquioxane 1

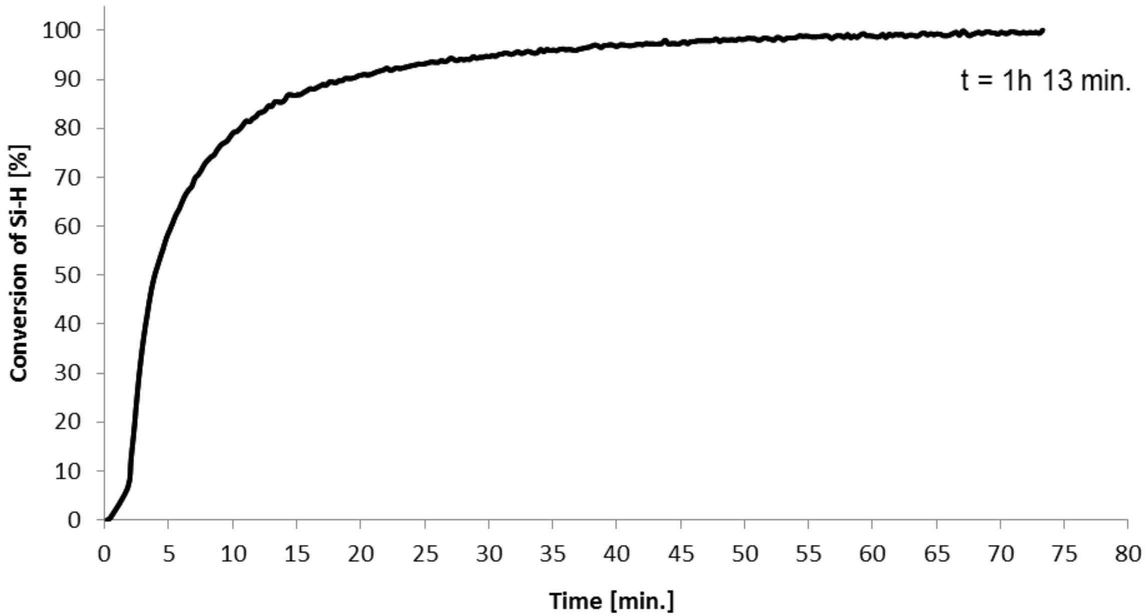
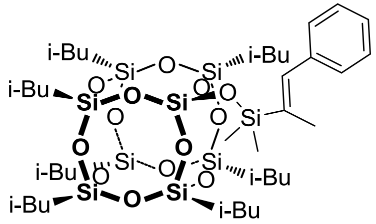
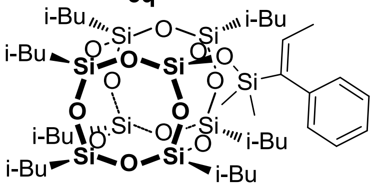


Figure 68. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2q with 1.



3q



4q

Chemical Formula: $C_{39}H_{78}O_{13}Si_9$
Molecular Weight: 1007,80

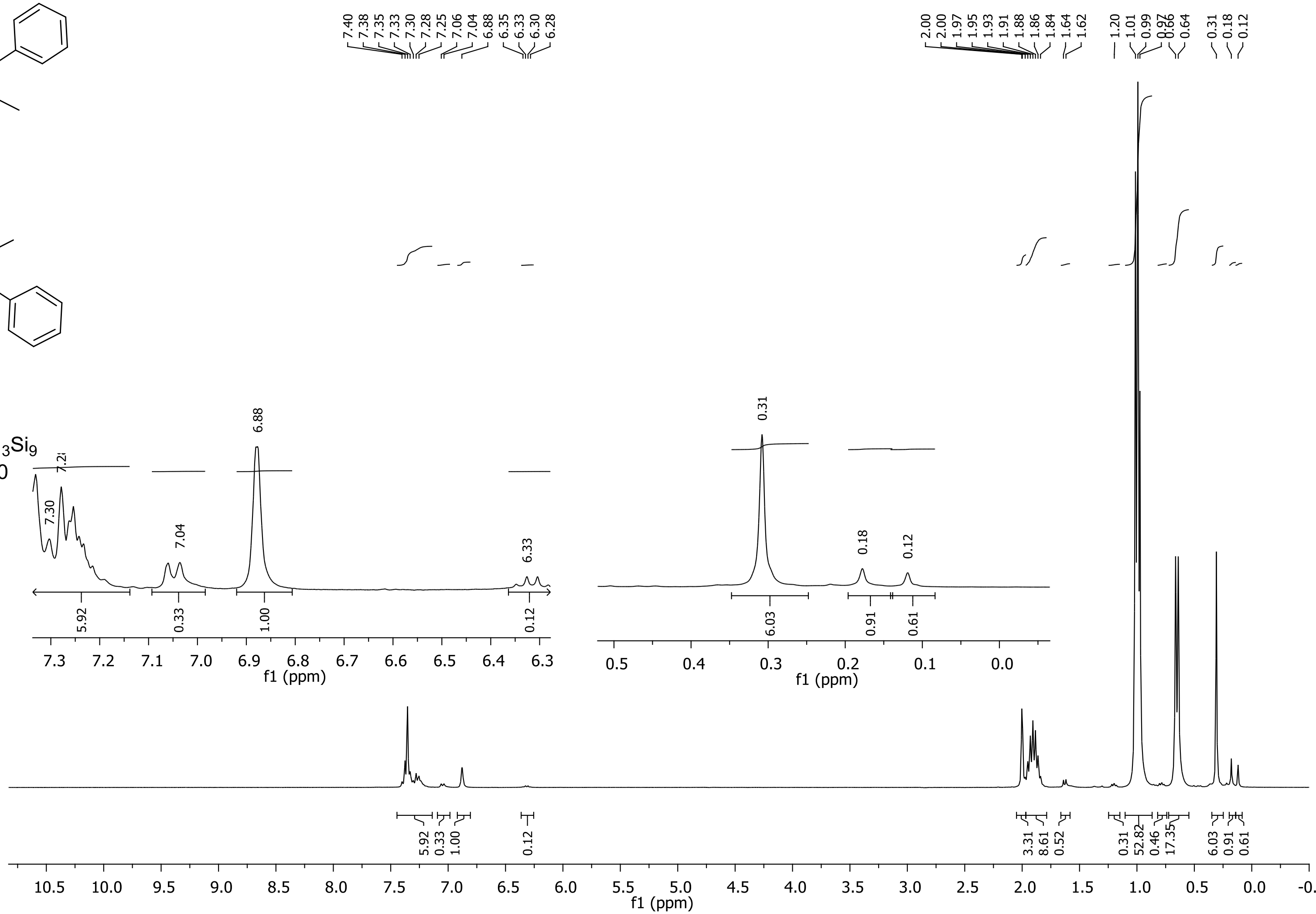
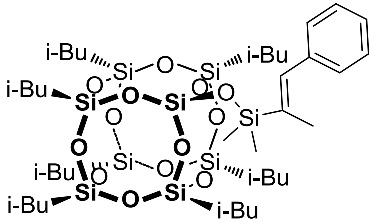
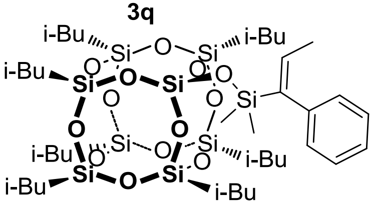


Figure 69. [1]H NMR of compound 3-4q.





Chemical Formula: $C_{39}H_{78}O_{13}Si_9$
Molecular Weight: 1007,80

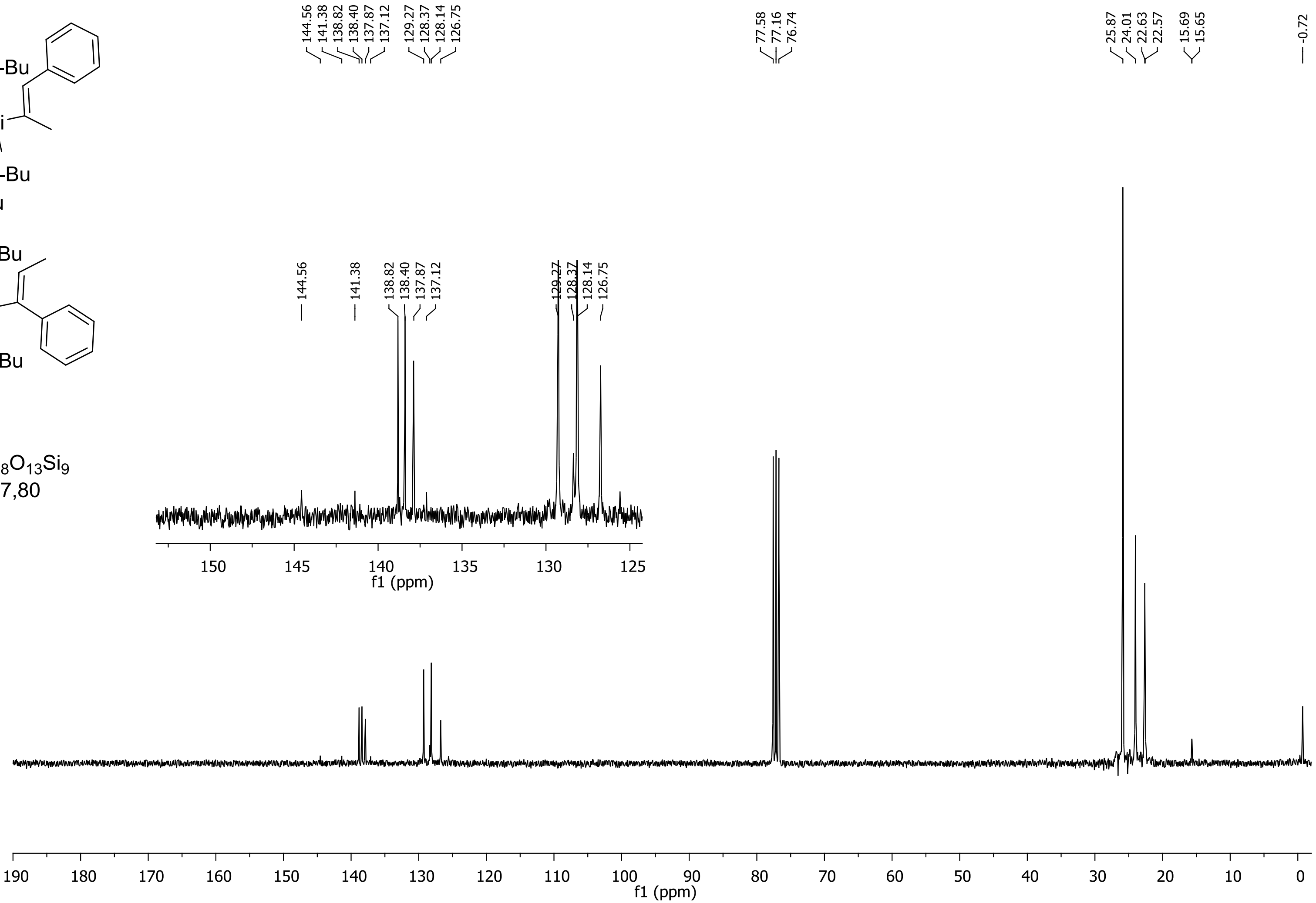
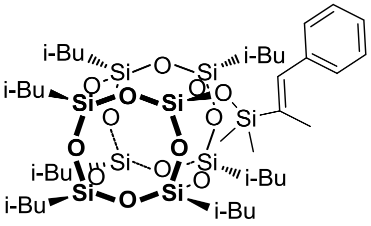
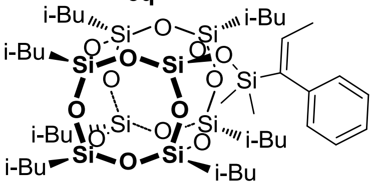


Figure 70. ^{13}C NMR of compound 3-4q.



3q



4q

Chemical Formula: $C_{39}H_{78}O_{13}Si_9$
Molecular Weight: 1007,80

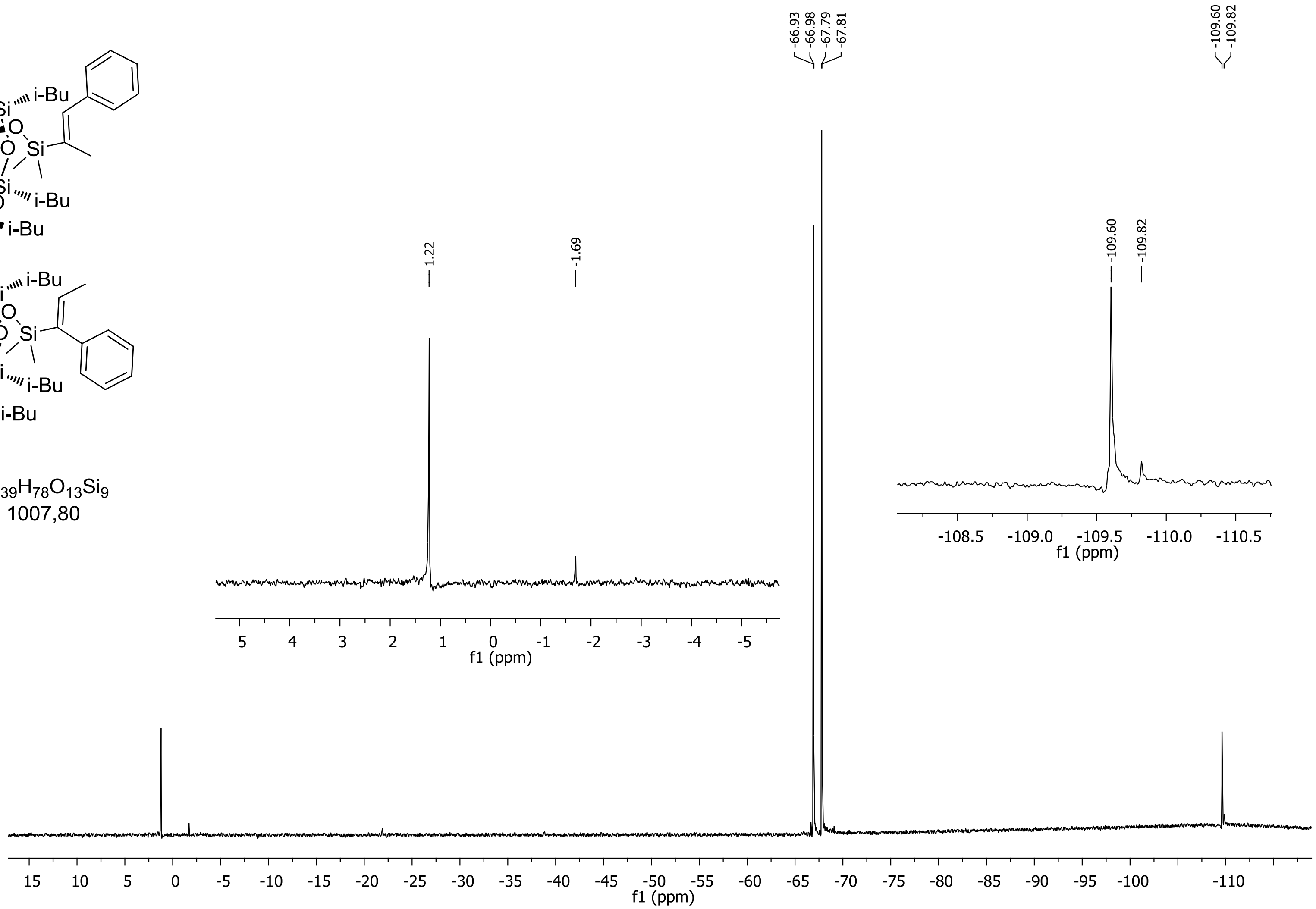


Figure 71. ^{29}Si NMR of compound 3-4q.

3.19 Hydrosilylation of 2r with silsesquioxane 1

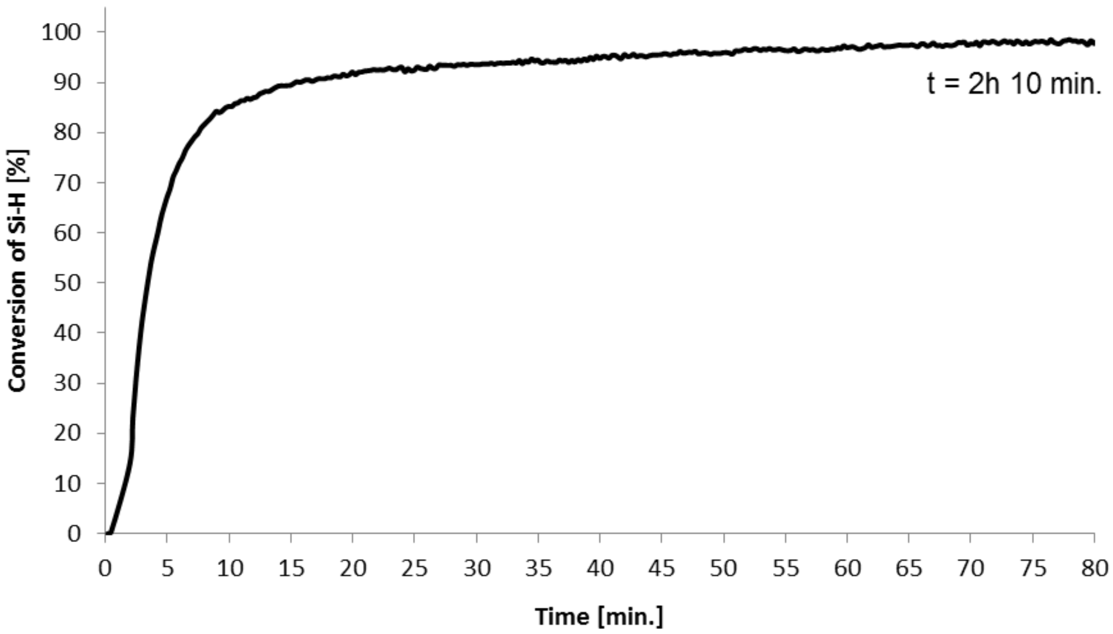
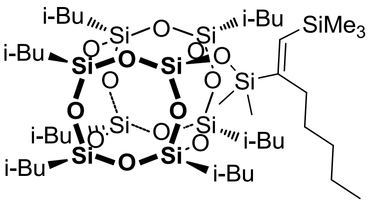


Figure 72. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2r with 1.



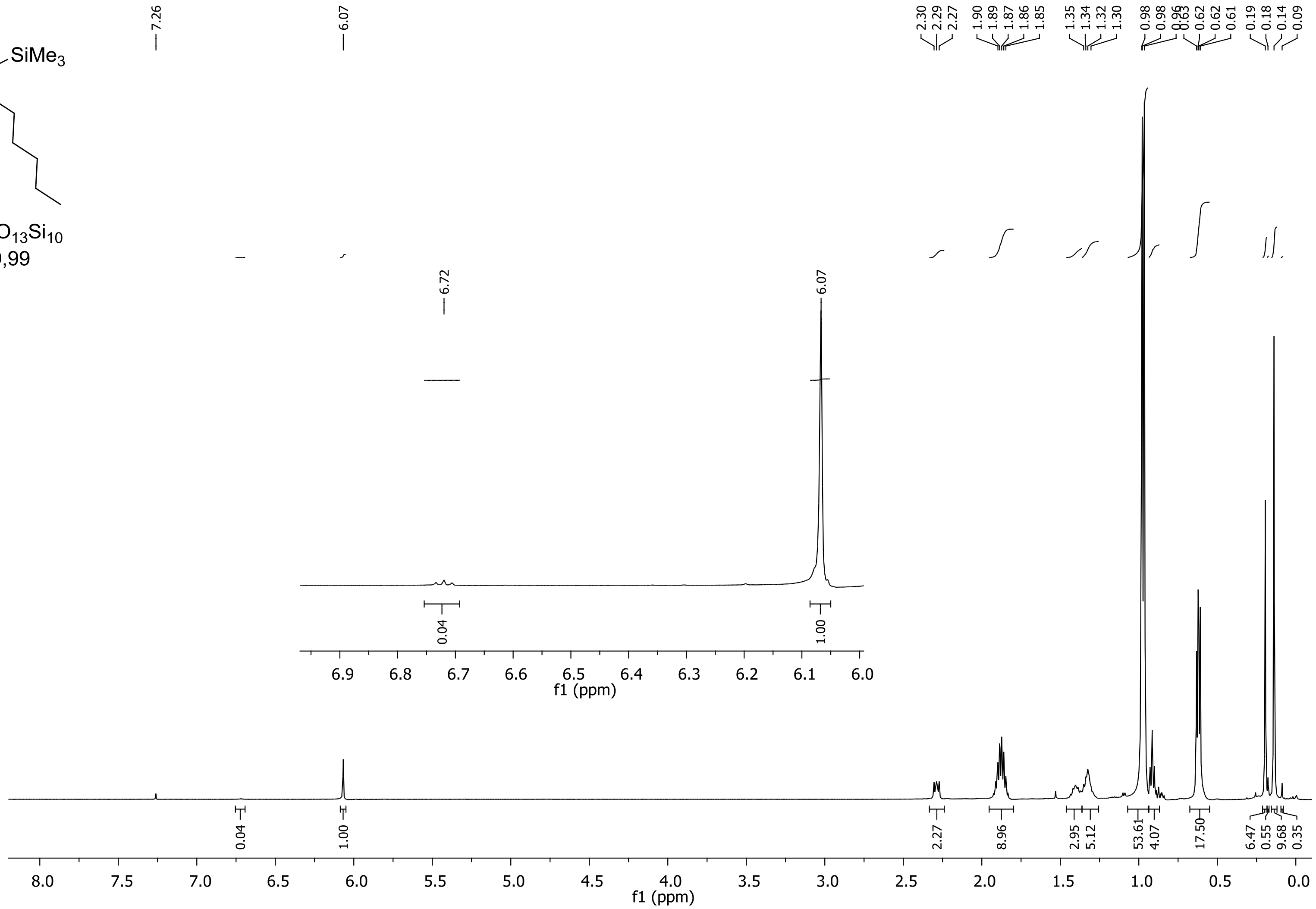
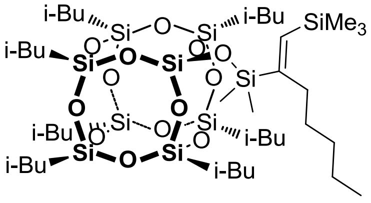


Figure 73. ^{1}H NMR of compound 3r.



Chemical Formula: $C_{40}H_{90}O_{13}Si_{10}$
Molecular Weight: 1059,99

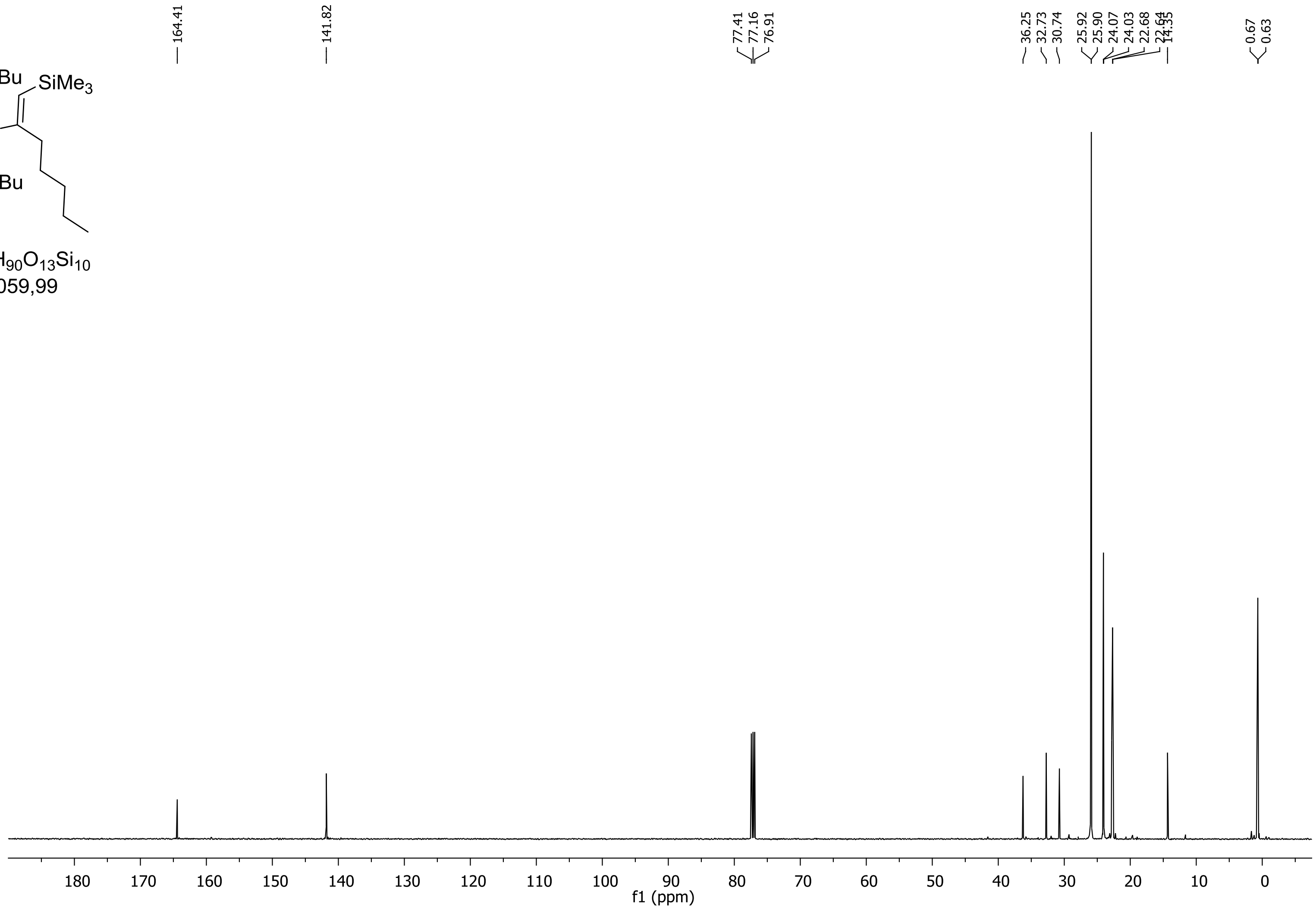


Figure 74. ^{13}C NMR of compound 3r.

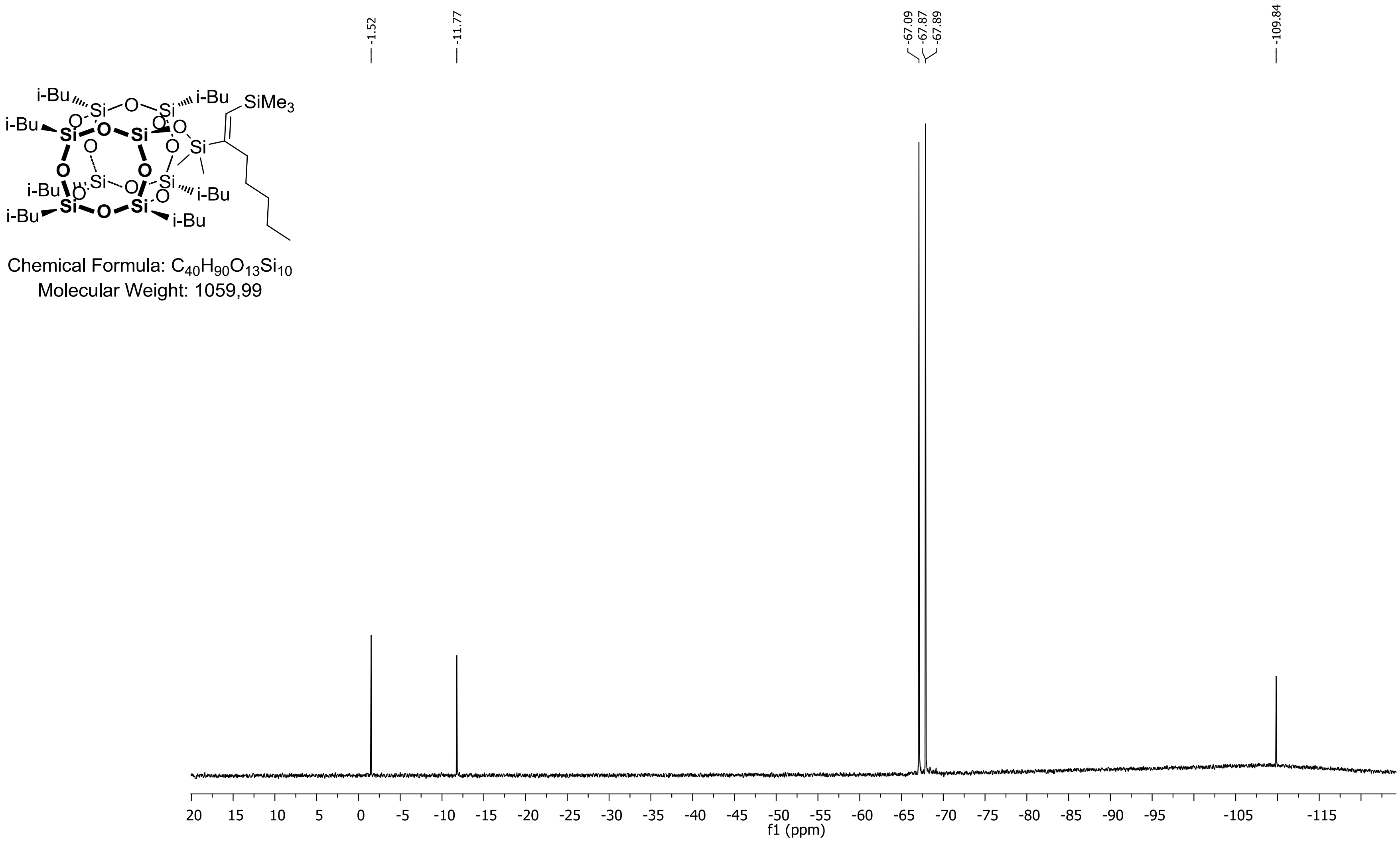


Figure 75. ^{29}Si NMR of compound 3r.

3.20 Hydrosilylation of 2s with silsesquioxane 1

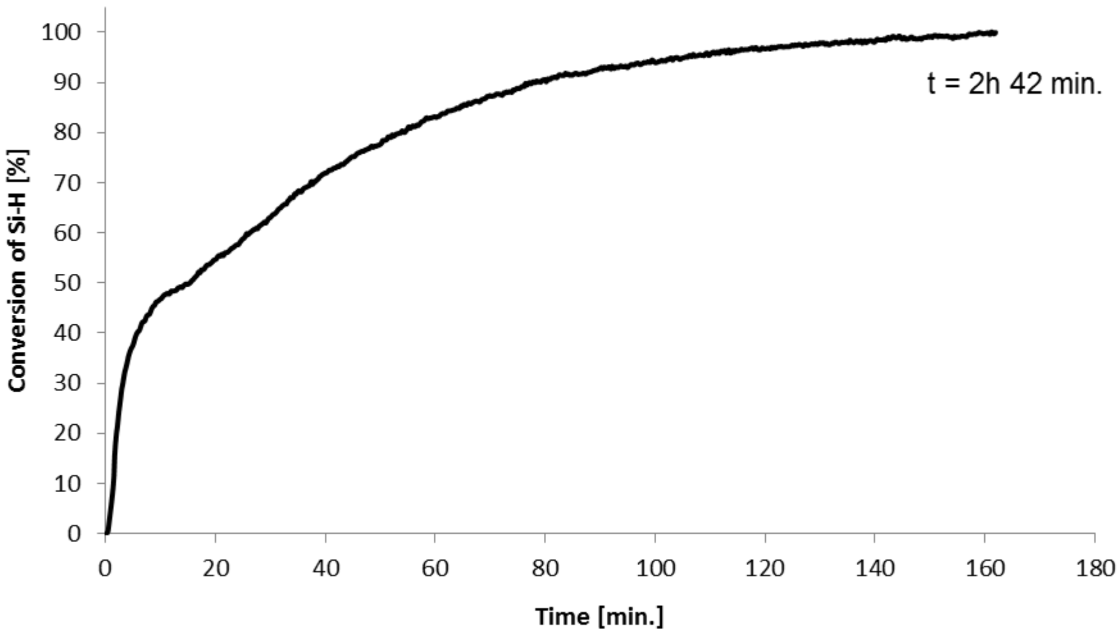
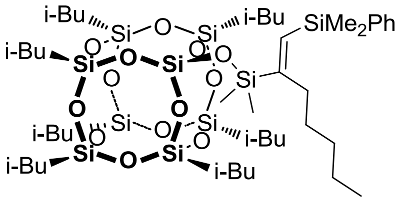


Figure 76. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2s with 1.



Chemical Formula: $C_{45}H_{92}O_{13}Si_{10}$
Molecular Weight: 1122,06

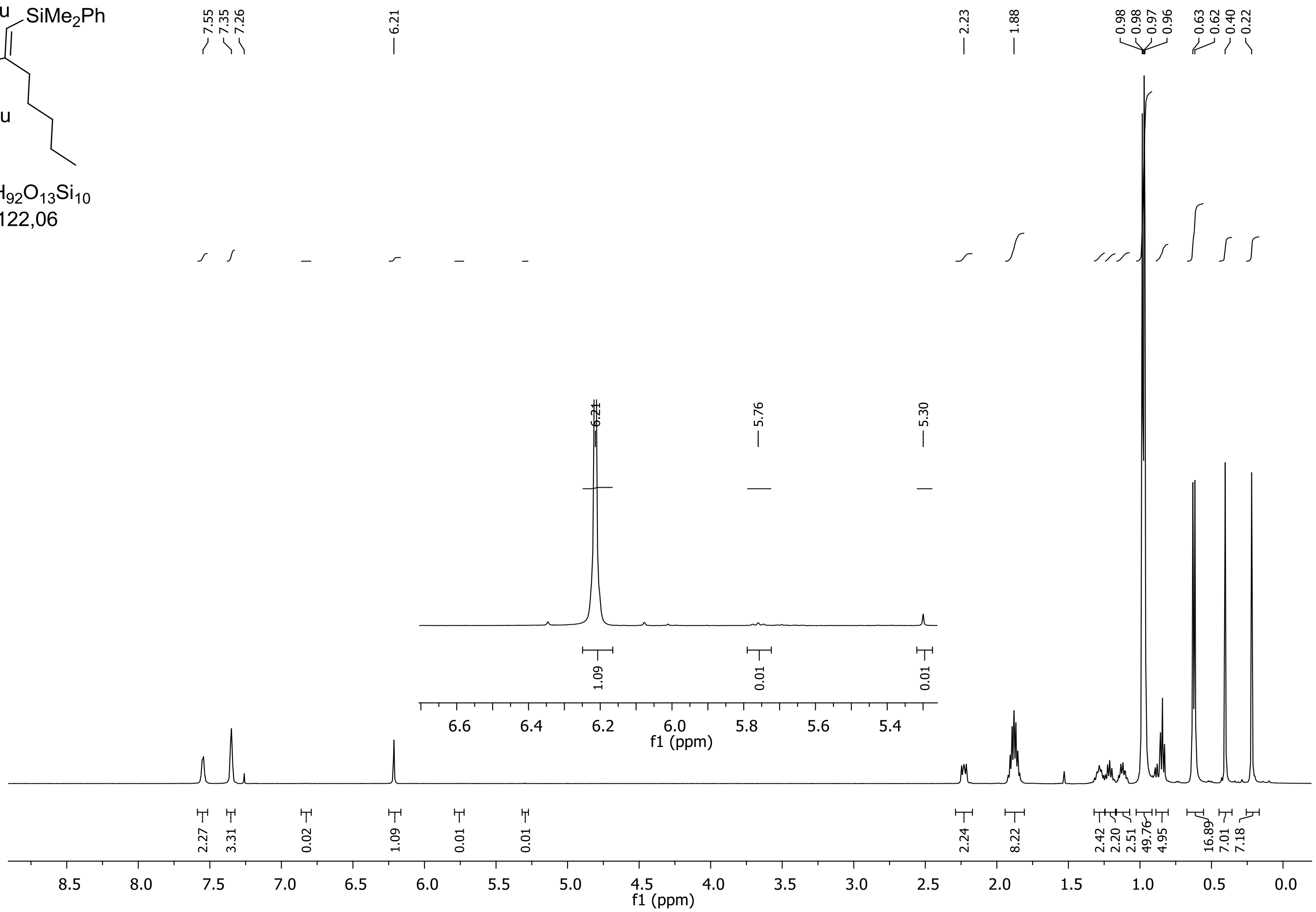
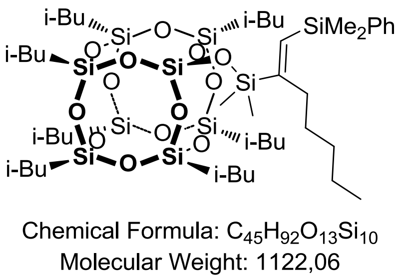


Figure 77. [1]H NMR of compound 3s.



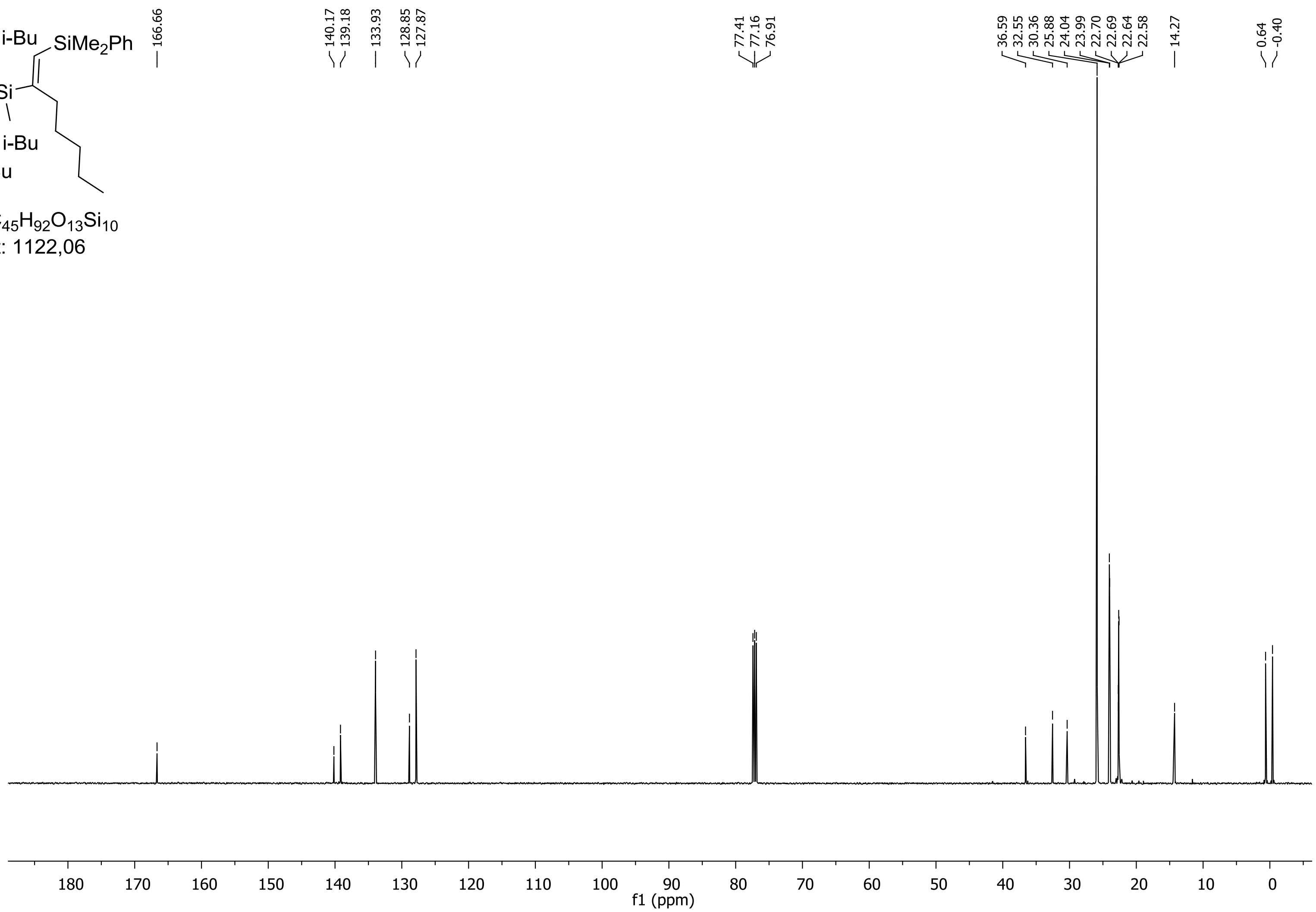


Figure 78. ^{13}**C NMR of compound 3s.**

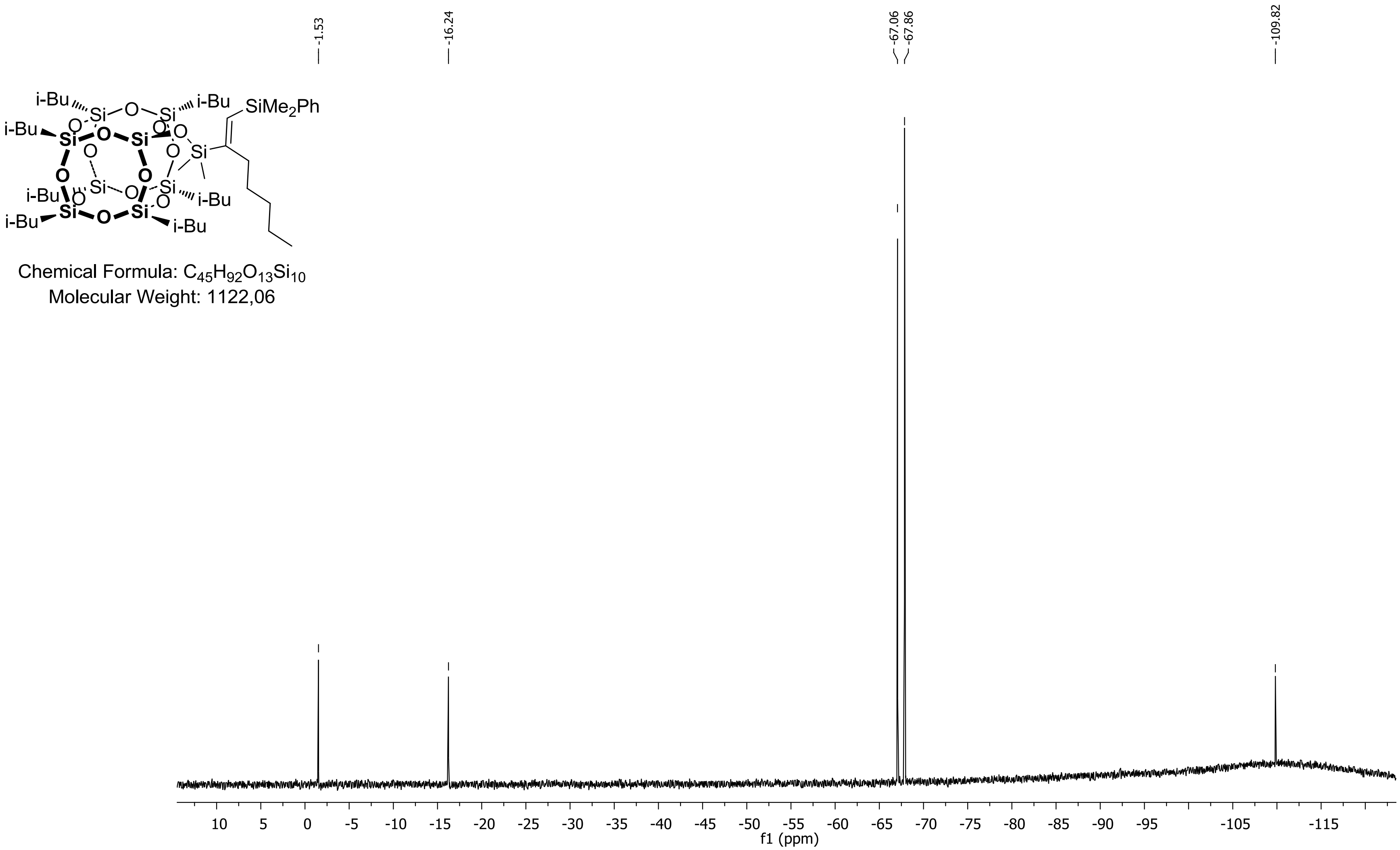


Figure 79. ^{29}**Si NMR of compound 3s.**

3.21 Hydrosilylation of 2t with silsesquioxane 1

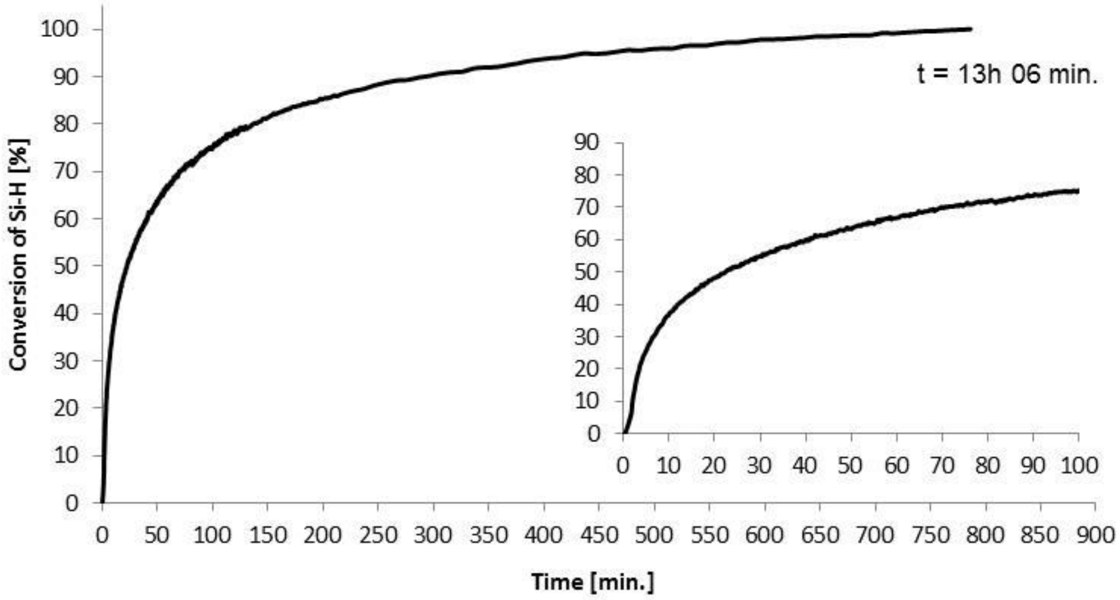
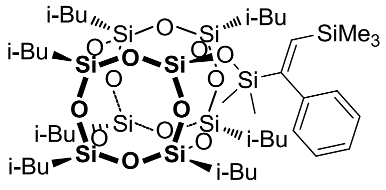


Figure 80. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2t with 1.



Chemical Formula: $C_{41}H_{84}O_{13}Si_{10}$
Molecular Weight: 1065,95

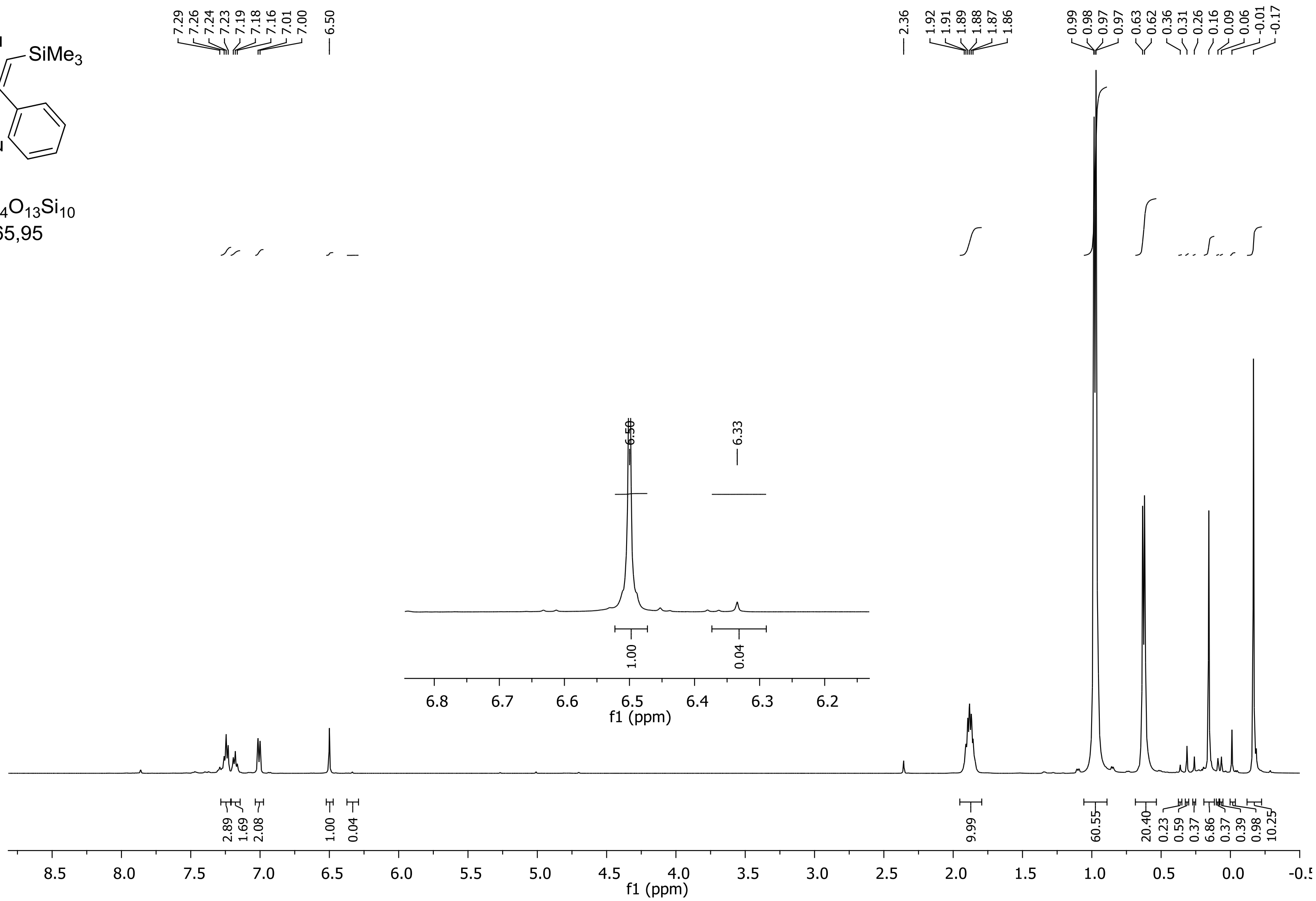
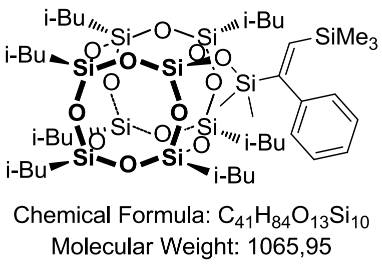


Figure 81. ^{1}H NMR of compound 3t.



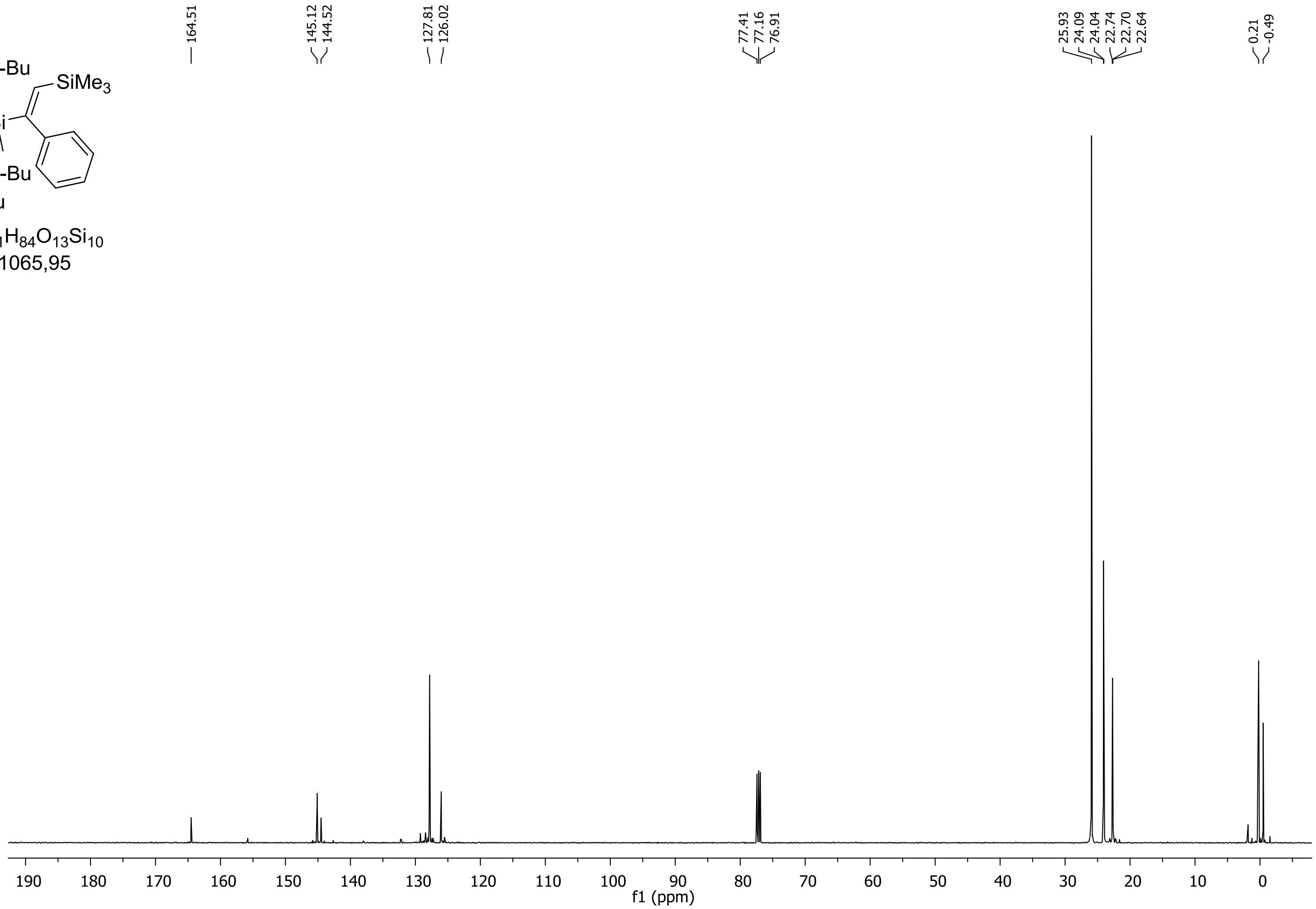
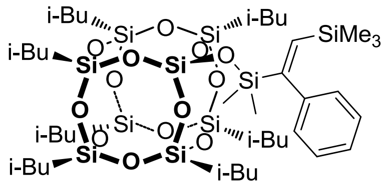


Figure 82. [13]C NMR of compound 3t.



Chemical Formula: $C_{41}H_{84}O_{13}Si_{10}$
Molecular Weight: 1065,95

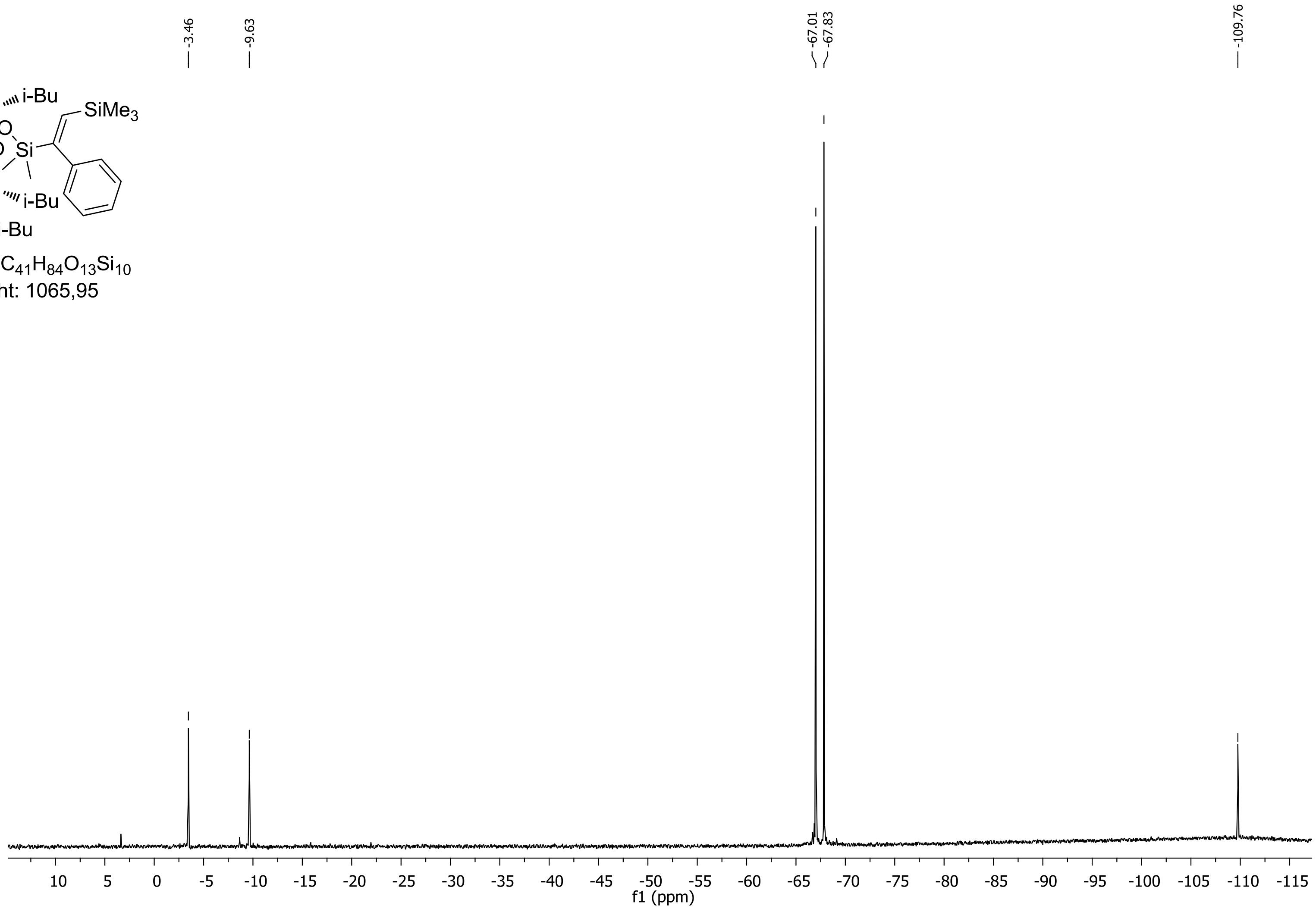


Figure 83. ^{29}**Si NMR of compound 3t.**

3.22 Hydrosilylation of 2u with silsesquioxane 1

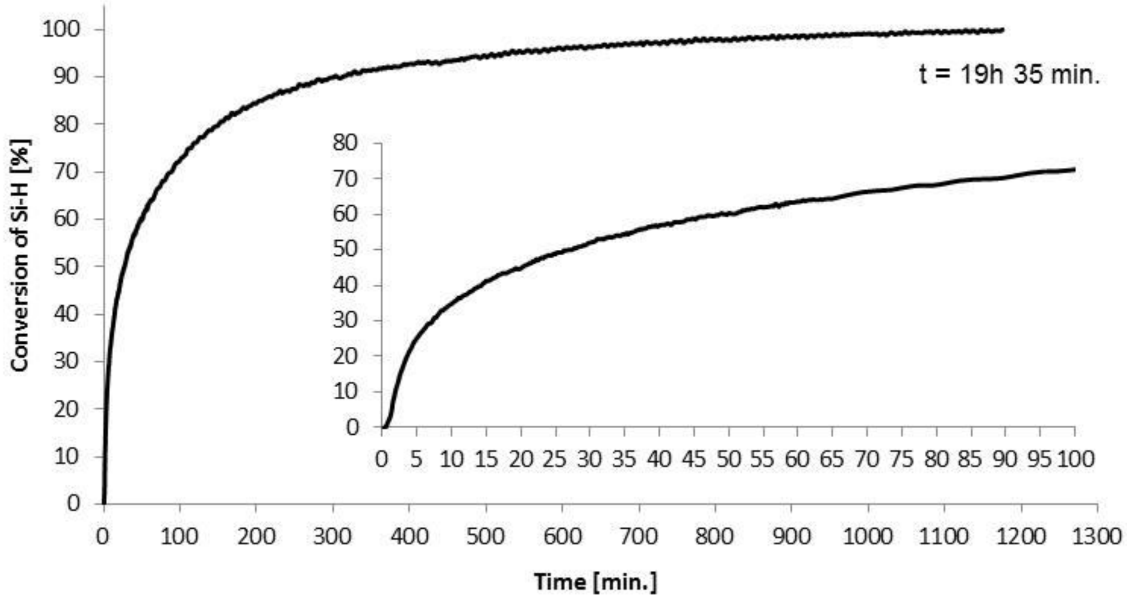
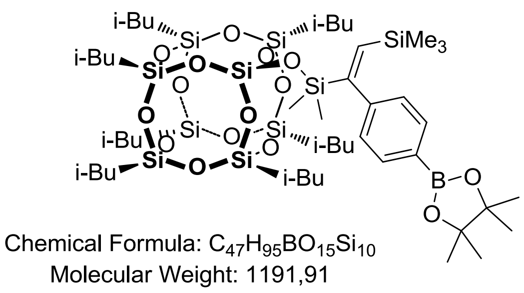


Figure 84. Plot of conversion vs time determined by in situ FT-IR for hydrosilylation of 2u with 1.



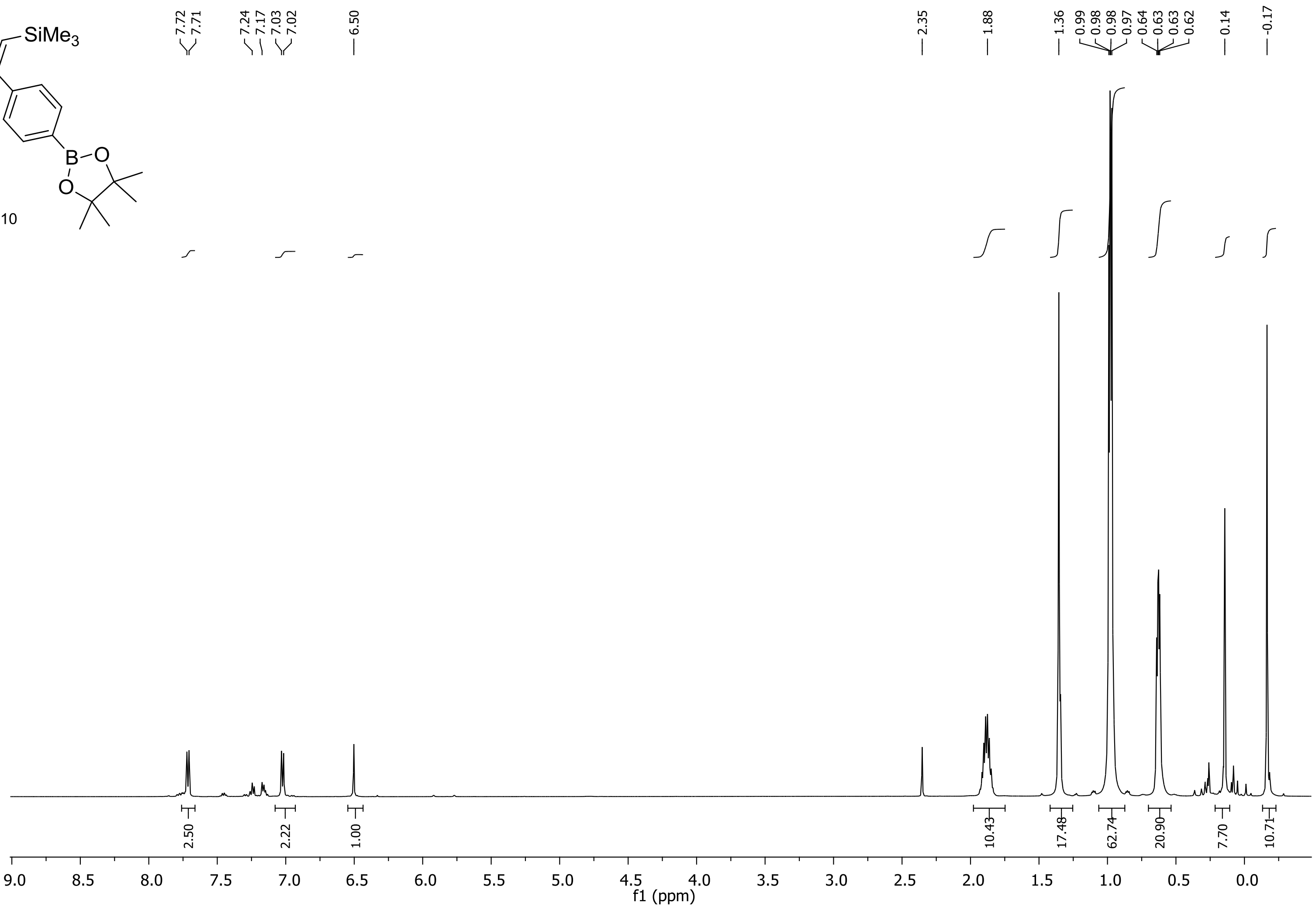


Figure 85. **[1]H NMR of compound 3u.**

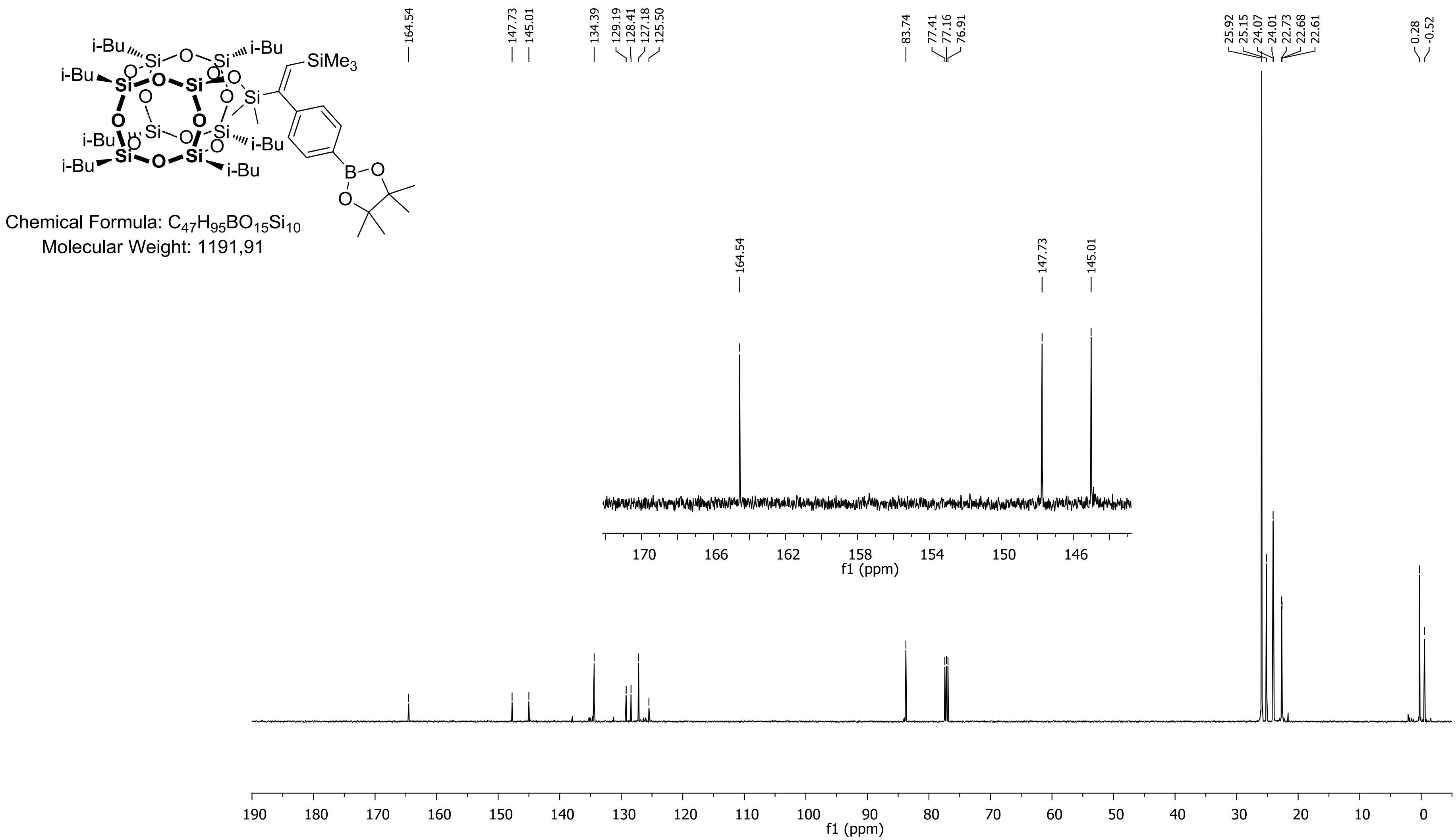


Figure 86. ^{13}C NMR of compound 3u.

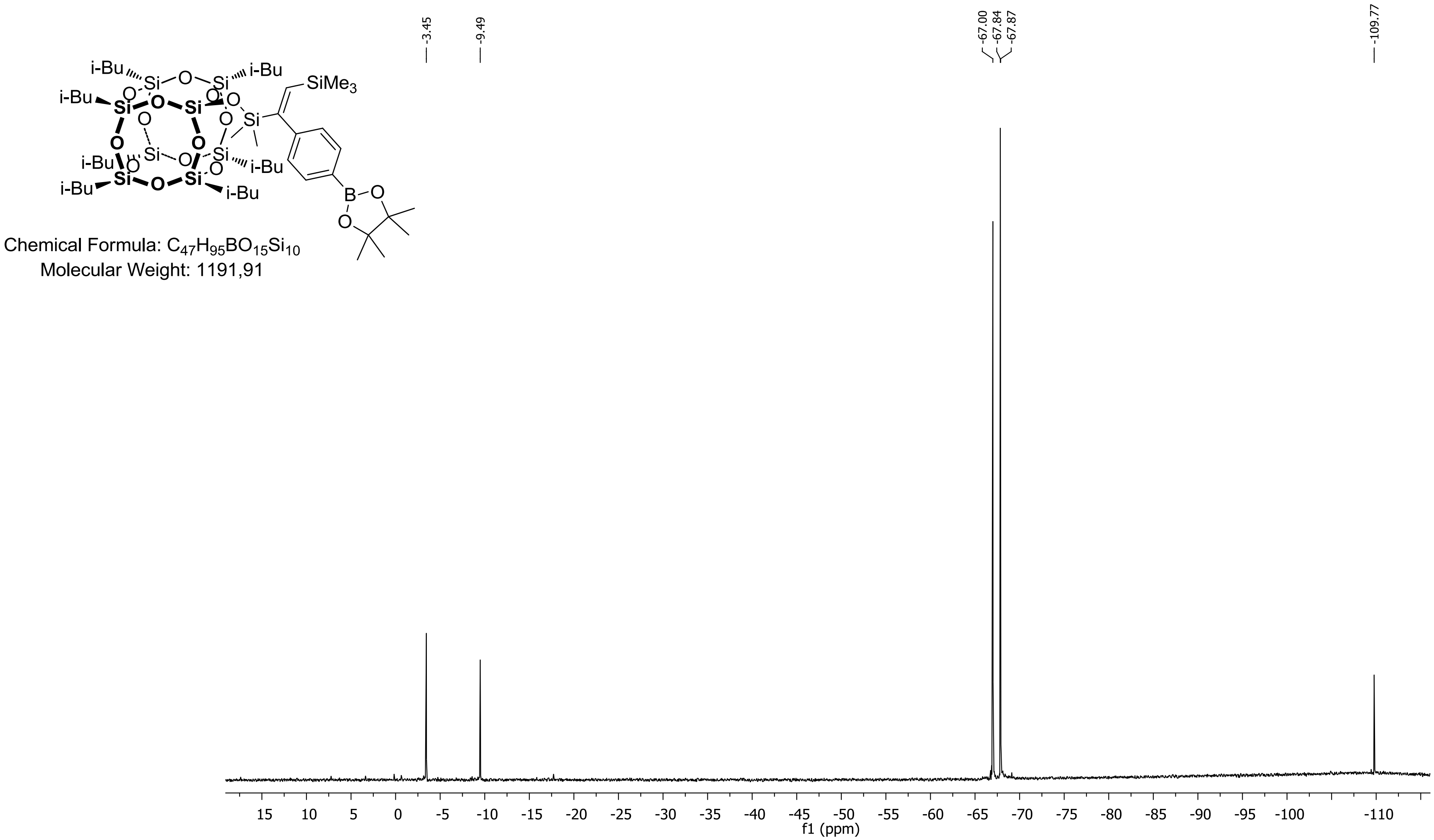


Figure 87. ^{29}Si NMR of compound 3u.

4. References

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[4] P. Mazerolles, *Bulletin de la Societe Chimique de France* 1960, 859.