Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Dancing Silanols: Stereospecific Rearrangements of Silanol Epoxides into Silanoxy-Tetrahydrofurans and Silanoxy-Tetrahydropyrans

Harshit Joshi[a], Annu Anna Thomas[a], Joel T. Mague[b], and Shyam Sathyamoorthi[a,*]

[a] University of Kansas, Department of Medicinal Chemistry, Lawrence, KS, USA (66047)

[b] Tulane University, Department of Chemistry, New Orleans, LA, USA (70118)

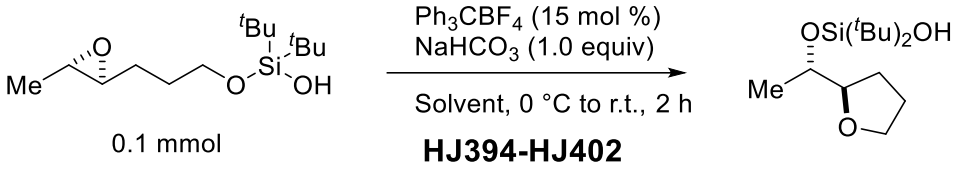
*ssathyam@ku.edu

I. General Considerations

All reagents were obtained commercially unless otherwise noted. Solvents were purified by passage under 10 psi N_2 through activated alumina columns. Infrared (IR) spectra were recorded on a Thermo Scientific™ Nicolet™ iS™5 FT-IR Spectrometer; data are reported in frequency of absorption (cm^{-1}). ^1H NMR spectra were recorded at 400, 500, or 600 MHz. Data are recorded as: chemical shift in ppm referenced internally using residual solvent peaks, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or overlap of nonequivalent resonances), integration, coupling constant (Hz). ^{13}C NMR spectra were recorded at 101 or 126 MHz. Exact mass spectra were recorded using an electrospray ion source (ESI) either in positive mode or negative mode and with a time-of-flight (TOF) analyzer on a Waters LCT PremierTM mass spectrometer and are given in m/z. Thin Layer Chromatography (TLC) was performed on pre-coated glass plates (Merck) and visualized either with a UV lamp (254 nm) or by dipping into a solution of $KMnO_4$–K_2CO_3 or of ceric ammonium molybdate in water followed by heating. Flash chromatography was performed on silica gel (230-400 mesh) or Florisil (60-100 mesh). *Note on compound numbering for the Supporting Information*: Silanol starting materials are numbered according to the corresponding manuscript epoxide. Thus, silanol **S1** was the starting material for epoxide substrate **1**, silanol **S3** was the starting material for epoxide substrate **3**, etc.

II. Additional Optimization Experiments

Solvent Screening:



Reaction No.	Lewis acid	SM remaining (%)	Product yield (%) (by ^{1}HNMR)
HJ394	THF	93	0
HJ395	DME	100	0
HJ396	Dioxane	100	0
HJ397	MeOH	94	0
HJ398	EtOAc	97	0
HJ399	Acetone	84	03
HJ400	MeCN	79	16
HJ401	$MeNO_2$	0	75
HJ402	Toluene	80	15

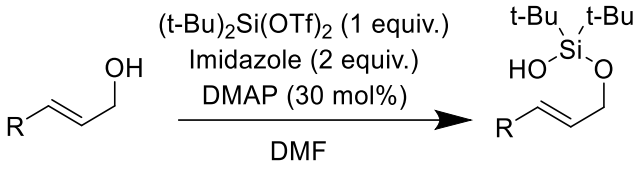
Note: Catalyst Loading of less than 15% leads to lower product yields.

III. Preparation of Alkenyl Silanols

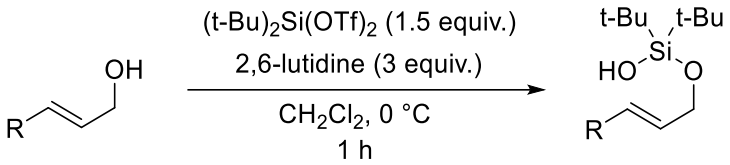
General Procedure A

Silanols were prepared according to the procedures disclosed in *Org. Lett.* **2020**, *22*, 8665–8669 and in *Org. Lett.* **2022**, *24*, 6202–6207. In general, the procedures give comparable yields of silanol products, but the latter procedure is operationally simpler on scale as it avoids the use of DMF.

Representative Schemes for Starting Material Preparation:

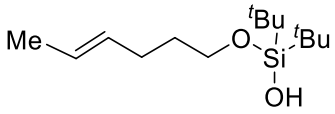


(*Org. Lett.* **2020**, *22*, 8665–8669)



(*Org. Lett.* **2022**, *24*, 6202–6207)

IV. Characterization of Alkenyl Silanols



(*E*)-di-*tert*-butyl(hex-4-en-1-yloxy)silanol

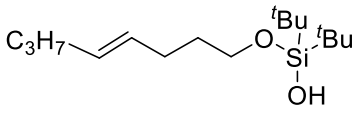
Compound S1: Synthesized using General Procedure A on a 10 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 2.12 g, 8.2 mmol, 82% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.52 – 5.32 (m, 2H), 3.78 (t, *J* = 6.4 Hz, 2H), 2.17 – 1.99 (m, 2H), 1.78 (s, 1H), 1.67 – 1.53 (m, 5H), 1.01 (s, 18H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 131.1, 125.2, 63.0, 32.9, 29.0, 27.6, 20.6, 18.1.

IR ν 3501, 2934, 2860, 1471, 1106, 827 cm^{-1}

HRMS (ESI) m/z = [M + Na$^+$] Calcd for $C_{14}H_{30}O_2SiNa^+$ 281.1913, found 281.1909 (1.4 ppm error).



(*E*)-di-*tert*-butyl(oct-4-en-1-yloxy)silanol

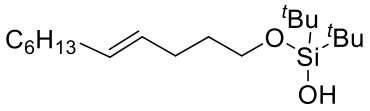
Compound S3: Synthesized using General Procedure A on a 2.0 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 349 mg, 1.22 mmol, 61% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.52 – 5.30 (m, 2H), 3.79 (t, *J* = 6.4 Hz, 2H), 2.11 – 2.02 (m, 2H), 1.99 – 1.91 (m, 2H), 1.80 (s, 1H), 1.65 – 1.55 (m, 2H), 1.36 (h, *J* = 7.3 Hz, 2H), 1.01 (s, 18H), 0.88 (t, *J* = 7.3 Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 130.8, 130.0, 63.0, 34.9, 32.9, 29.0, 27.6, 22.9, 20.6, 13.8.

IR ν 3502, 2933, 2859, 1473, 1107, 827, 648 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{16}H_{34}O_2SiNa^+$ 309.2226, found 309.2224 (0.6 ppm error).



(*E*)-di-*tert*-butyl(undec-4-en-1-yloxy)silanol

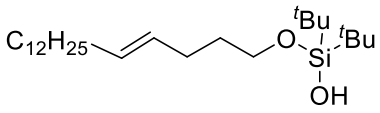
Compound S5: Synthesized using General Procedure A on a 2.0 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 415 mg, 1.26 mmol, 63% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.48 – 5.29 (m, 2H), 3.79 (t, *J* = 6.4 Hz, 2H), 2.11 – 2.02 (m, 2H), 2.01 – 1.93 (m, 2H), 1.64 – 1.56 (m, 2H), 1.35 – 1.23 (m, 8H), 1.01 (s, 18H), 0.88 (t, *J* = 6.8 Hz, 3H).

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 131.1, 129.8, 63.0, 32.9, 32.8, 31.9, 29.7, 29.01, 28.96, 27.6, 22.8, 20.6, 14.3.

IR ν 3496, 2930, 2857, 1471, 1107, 828, 647 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H$^+$] Calcd $C_{19}H_{41}O_2Si^+$ 329.2876, found 329.2869 (2.1 ppm error).



(*E*)-di-*tert*-butyl(heptadec-4-en-1-yloxy)silanol

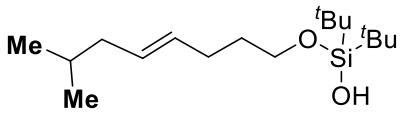
Compound S7: Synthesized using General Procedure A on a 1.0 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 322 mg, 0.78 mmol, 78% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.48 – 5.32 (m, 2H), 3.79 (t, *J* = 6.4 Hz, 2H), 2.11 – 2.01 (m, 2H), 2.01 – 1.92 (m, 2H), 1.81 (s, 1H), 1.64 – 1.56 (m, 2H), 1.34 – 1.24 (m, 20H), 1.01 (s, 18H), 0.90 – 0.86 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 131.1, 129.8, 63.0, 32.9, 32.8, 32.1, 29.84, 29.81, 29.79, 29.70, 29.5, 29.4, 29.0, 27.6, 22.8, 20.6, 14.3.

IR ν 3498, 2926, 2856, 1470, 1109, 828, 647 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{25}H_{52}O_2SiNa^+$ 435.3634, found 435.3660 (6.0 ppm error).



(*E*)-di-*tert*-butyl((7-methyloct-4-en-1-yl)oxy)silanol

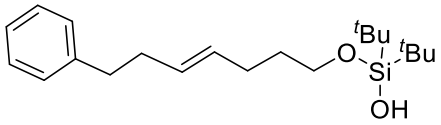
Compound S9: Synthesized using General Procedure A on a 4.0 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 664 mg, 2.21 mmol, 55% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.47 – 5.31 (m, 2H), 3.79 (t, *J* = 6.4 Hz, 2H), 2.12 – 2.02 (m, 2H), 1.89 – 1.83 (m, 2H), 1.79 (s, 1H), 1.65 – 1.51 (m, 3H), 1.01 (s, 18H), 0.87 (d, *J* = 6.6 Hz, 6H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 131.0, 129.7, 63.0, 42.2, 33.0, 29.0, 28.6, 27.6, 22.4, 20.6.

IR ν 3499, 2954, 2933, 2897, 2860, 1471, 1107, 968, 827, 647 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H^+] Calcd $C_{17}H_{37}O_2Si^+$ 301.2563, found 301.2556 (2.3 ppm error).



(*E*)-di-*tert*-butyl((7-phenylhept-4-en-1-yl)oxy)silanol

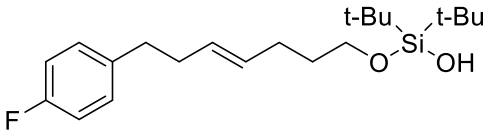
Compound S11: Synthesized using General Procedure A on a 4.0 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 782 mg, 2.24 mmol, 56% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.32 (m, 2H), 7.28 – 7.19 (m, 3H), 5.60 – 5.47 (m, 2H), 3.84 (t, *J* = 6.4 Hz, 2H), 2.74 (dd, *J* = 9.0, 6.7 Hz, 2H), 2.43 – 2.33 (m, 2H), 2.20 – 2.09 (m, 2H), 1.86 (s, 1H), 1.72 – 1.62 (m, 2H), 1.09 (s, 18H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 142.3, 130.6, 129.9, 128.6, 128.4, 125.8, 62.9, 36.2, 34.6, 32.9, 28.9, 27.6, 20.6.

IR ν 3499, 2933, 2857, 1471, 1109, 827, 698, 645 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H^+] Calcd $C_{21}H_{37}O_2Si^+$ 349.2563, found 349.2550 (3.7 ppm error).



(*E*)-di-*tert*-butyl((7-(4-fluorophenyl)hept-4-en-1-yl)oxy)silanol

Compound S13: Synthesized using General Procedure A on a 2 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 461 mg, 1.26 mmol, 63% yield).

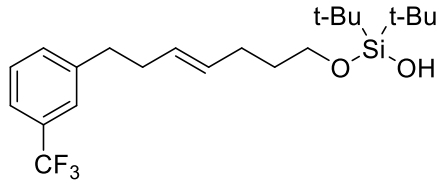
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 – 7.06 (m, 2H), 7.01 – 6.90 (m, 2H), 5.47 – 5.40 (m, 2H), 3.77 (t, J = 6.4 Hz, 2H), 2.64 (dd, J = 8.8, 6.7 Hz, 2H), 2.33 – 2.23 (m, 2H), 2.12-2.01 (m, 2H), 1.64 – 1.53 (m, 2H), 1.02 (s, 18H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 161.3 (d, J = 243.2 Hz), 137.8 (d, J = 3.5 Hz), 130.9, 129.9 (d, J = 7.5 Hz), 129.6, 115.1 (d, J = 21.2 Hz), 62.9, 35.4, 34.7, 32.8, 28.9, 27.6, 20.6.

^{19}F NMR (377 MHz, $CDCl_3$) δ -118.51 – -118.76 (m, J = 5.5 Hz).

IR ν 2965, 2857, 1510, 1471, 1224, 1107, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{21}H_{35}FO_2SiNa^+$ 389.2288, found 389.2284 (1.0 ppm error).



(*E*)-di-*tert*-butyl((7-(3-(trifluoromethyl)phenyl)hept-4-en-1-yl)oxy)silanol

Compound S15: Synthesized using General Procedure A on a 2 mmol scale.; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 541 mg, 1.30 mmol, 65% yield).

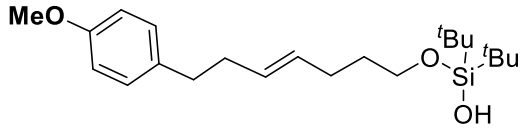
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, J = 1.8 Hz, 2H), 7.39 – 7.35 (m, 2H), 5.49 – 5.40 (m, 2H), 3.79 (t, J = 6.4 Hz, 2H), 2.73 (dd, J = 8.7, 6.8 Hz, 2H), 2.33 (dddd, J = 8.9, 6.6, 4.2, 1.4 Hz, 2H), 2.14 – 2.04 (m, 2H), 1.65 – 1.54 (m, 2H), 1.03 (s, 18H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 143.0, 132.0, 131.3, 130.6 (q, *J* = 31.9 Hz), 129.1, 128.7, 125.3 (q, *J* = 3.7 Hz), 123.1, (q, *J* = 273 Hz), 122.7 (q, *J* = 4.0 Hz), 62.9, 35.9, 34.2, 32.8, 28.9, 27.6, 20.6.

^{19}F NMR (377 MHz, $CDCl_3$) δ -63.2.

IR ν 3566, 2934, 2859, 1471, 1166, 1073, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{22}H_{35}F_3O_2SiNa^+$ 439.2256, found 439.2274 (4.1 ppm error).



(*E*)-di-*tert*-butyl((7-(4-methoxyphenyl)hept-4-en-1-yl)oxy)silanol

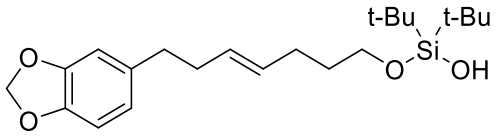
Compound S17: Synthesized using General Procedure A on a 2.94 mmol scale; Purified using a gradient of 10-55% CH_2Cl_2 in hexane on silica gel; (colorless oil, 870 mg, 2.30 mmol, 78% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 – 7.06 (m, 2H), 6.85 – 6.79 (m, 2H), 5.54 – 5.36 (m, 2H), 3.80 – 3.74 (m, 5H), 2.61 (dd, *J* = 8.9, 6.7 Hz, 2H), 2.32 – 2.22 (m, 2H), 2.12 – 2.00 (m, 2H), 1.79 (s, 1H), 1.64 – 1.54 (m, 2H), 1.02 (s, 18H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 157.8, 134.4, 130.6, 130.0, 129.5, 113.8, 63.0, 55.4, 35.3, 34.8, 32.9, 28.9, 27.6, 20.6.

IR ν 3499, 2933, 2857, 1612, 1512, 1471, 1246, 1107, 827, 647 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H$^+$] Calcd $C_{22}H_{39}O_3Si^+$ 379.2668, found 379.2669 (0.3 ppm error).



(*E*)-((7-(benzo[*d*][1,3]dioxol-5-yl)hept-4-en-1-yl)oxy)di-*tert*-butylsilanol

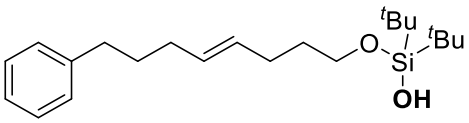
Compound S19: Synthesized using General Procedure A on a 2 mmol scale.; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 471 mg, 1.20 mmol, 60% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.73 (d, J = 7.9 Hz, 1H), 6.68 (d, J = 1.7 Hz, 1H), 6.62 (dd, J = 7.9, 1.7 Hz, 1H), 5.91 (s, 2H), 5.49 – 5.41 (m, 2H), 3.78 (t, J = 6.4 Hz, 2H), 2.59 (dd, J = 8.9, 6.7 Hz, 2H), 2.31 – 2.21 (m, 2H), 2.12 – 1.91 (m, 2H), 1.66 – 1.54 (m, 2H), 1.03 (s, 18H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 147.5, 145.6, 136.1, 130.7, 129.7, 121.3, 109.1, 108.2, 100.8, 62.9, 35.9, 34.8, 32.8, 28.9, 27.6, 20.6.

IR ν 3607, 2934, 2860, 1505, 1488, 1246, 1040, 828 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{22}H_{36}O_4SiNa^+$ 415.2281, found 415.2279 (0.5 ppm error).



(*E*)-di-*tert*-butyl((8-phenyloct-4-en-1-yl)oxy)silanol

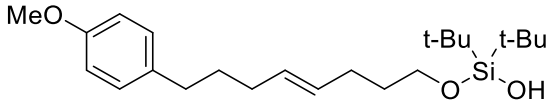
Compound S21: Synthesized using General Procedure A on a 2.0 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 556 mg, 1.53 mmol, 77% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.25 (m, 2H), 7.21 – 7.14 (m, 3H), 5.51 – 5.35 (m, 2H), 3.80 (t, *J* = 6.4 Hz, 2H), 2.64 – 2.57 (m, 2H), 2.15 – 1.96 (m, 4H), 1.81 (s, 1H), 1.73 – 1.65 (m, 2H), 1.65 – 1.57 (m, 2H), 1.02 (s, 18H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 142.8, 130.45, 130.40, 128.6, 128.4, 125.8, 63.0, 35.5, 32.9, 32.3, 31.4, 29.0, 27.6, 20.6.

IR ν 3500, 2931, 2859, 1604, 1495, 1471, 1107, 968, 828, 698, 647 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H$^+$] Calcd $C_{22}H_{39}O_2Si^+$ 363.2719, found 363.2704 (4.1 ppm error)



(*E*)-di-*tert*-butyl((8-(4-methoxyphenyl)oct-4-en-1-yl)oxy)silanol

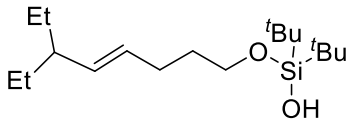
Compound S23: Synthesized using General Procedure A on a 2 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 549 mg, 1.40 mmol, 70% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 – 7.05 (m, 2H), 6.86 – 6.79 (m, 2H), 5.47 – 5.40 (m, 2H), 3.81 – 3.77 (m, 5H), 2.59 – 2.51 (m, 2H), 2.13 – 1.97 (m, 4H), 1.64 (tq, J = 14.9, 6.5 Hz, 4H), 1.02 (s, 18H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 157.8, 134.9, 130.5, 130.4, 129.4, 113.8, 63.0, 55.4, 34.6, 32.9, 32.2, 31.7, 29.0, 27.6, 20.6.

IR ν 2933, 2857, 1512, 1471, 1246, 1107, 828 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{23}H_{40}O_3SiNa^+$ 415.2644, found 415.2634 (2.4 ppm error).



(*E*)-di-*tert*-butyl((6-ethyloct-4-en-1-yl)oxy)silanol

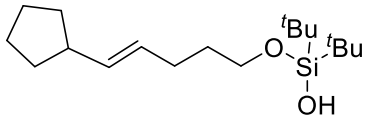
Compound S25: Synthesized using General Procedure A on a 2.0 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 388 mg, 1.23 mmol, 62% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.35 (dt, *J* = 15.3, 6.7 Hz, 1H), 5.11 (ddt, *J* = 15.2, 8.7, 1.4 Hz, 1H), 3.80 (t, *J* = 6.5 Hz, 2H), 2.14 – 2.03 (m, 2H), 1.82 (s, 1H), 1.70 (dp, *J* = 13.1, 4.5 Hz, 1H), 1.65 – 1.57 (m, 2H), 1.44 – 1.31 (m, 2H), 1.26 – 1.13 (m, 2H), 1.02 (s, 18H), 0.82 (t, *J* = 7.4 Hz, 6H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 135.1, 129.9, 63.0, 46.5, 33.1, 29.0, 27.9, 27.6, 20.6, 11.9.

IR ν 3506, 2963, 2933, 2859, 1471, 1107, 969, 828, 647 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{18}H_{38}O_2SiNa^+$ 337.2539, found 337.2561 (6.5 ppm error).



(*E*)-di-*tert*-butyl((5-cyclopentylpent-4-en-1-yl)oxy)silanol

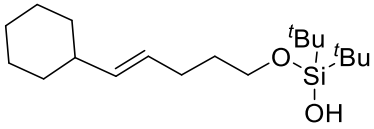
Compound S27: Synthesized using General Procedure A on a 2.0 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 398 mg, 1.27 mmol, 64% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.47 – 5.32 (m, 2H), 3.78 (t, *J* = 6.4 Hz, 2H), 2.44 – 2.30 (m, 1H), 2.12 – 2.00 (m, 2H), 1.87 (s, 1H), 1.78 – 1.69 (m, 2H), 1.65 – 1.47 (m, 6H), 1.31 – 1.19 (m, 2H), 1.01 (s, 18H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 135.7, 127.9, 63.0, 43.5, 33.4, 32.9, 28.9, 27.6, 25.3, 20.6.

IR ν 3499, 2934, 2859, 1471, 1109, 965, 828, 647 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H$^+$] Calcd $C_{18}H_{37}O_2Si^+$ 313.2563, found 313.2559 (1.3 ppm error).



(*E*)-di-*tert*-butyl((5-cyclohexylpent-4-en-1-yl)oxy)silanol

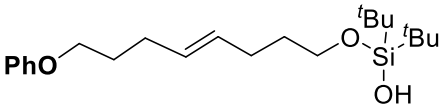
Compound S29: Synthesized using General Procedure A on a 2.0 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 435 mg, 1.33 mmol, 67% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.43 – 5.28 (m, 2H), 3.78 (t, *J* = 6.4 Hz, 2H), 2.10 – 2.00 (m, 2H), 1.95 – 1.79 (m, 2H), 1.75 – 1.55 (m, 7H), 1.32 – 1.03 (m, 5H), 1.01 (s, 18H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 137.1, 127.2, 62.9, 40.9, 33.4, 32.9, 29.0, 27.6, 26.4, 26.3, 20.6.

IR ν 3499, 2930, 2856, 1471, 1107, 827, 645 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{19}H_{38}O_2SiNa^+$ 349.2539, found 349.2561 (6.3 ppm error).



(*E*)-di-*tert*-butyl((8-phenoxyoct-4-en-1-yl)oxy)silanol

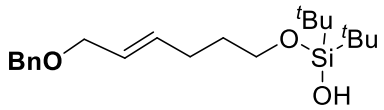
Compound S31: Synthesized using General Procedure A on a 1.9 mmol scale; Purified using a gradient of 30-100% CH_2Cl_2 in hexane on silica gel; (colorless oil, 371 mg, 0.98 mmol, 52% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.25 (m, 2H), 6.96 – 6.87 (m, 3H), 5.54 – 5.40 (m, 2H), 3.96 (t, *J* = 6.5 Hz, 2H), 3.79 (t, *J* = 6.4 Hz, 2H), 2.23 – 2.13 (m, 2H), 2.14 – 2.05 (m, 2H), 1.91 – 1.80 (m, 3H), 1.64 – 1.56 (m, 2H), 1.02 (s, 18H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.2, 130.9, 129.6, 129.5, 120.6, 114.7, 67.3, 62.9, 32.9, 29.3, 29.1, 28.9, 27.6, 20.6.

IR ν 3499, 2933, 2857, 1601, 1497, 1471, 1246, 1106, 827, 752, 691, 647 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{22}H_{38}O_3SiNa^+$ 401.2488, found 401.2493 (1.2 ppm error).



(*E*)-((6-(benzyloxy)hex-4-en-1-yl)oxy)di-*tert*-butylsilanol

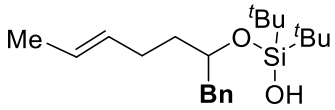
Compound S33: Synthesized using General Procedure A on a 2.0 mmol scale; Purified using a gradient of 30-100% CH_2Cl_2 in hexane on silica gel; (colorless oil, 583 mg, 1.6 mmol, 80% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.17 (m, 5H), 5.73 – 5.61 (m, 1H), 5.60 – 5.51 (m, 1H), 4.43 (s, 2H), 3.94 – 3.84 (m, 2H), 3.73 (t, *J* = 6.3 Hz, 2H), 2.14 – 2.03 (m, 2H), 1.87 (s, 1H), 1.63 – 1.52 (m, 2H), 0.94 (s, 18H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 138.5, 134.6, 128.5, 127.9, 127.7, 126.7, 72.1, 71.0, 62.8, 32.3, 28.8, 27.6, 20.6.

IR ν 3450, 2931, 2857, 1471, 1363, 1107, 969, 827, 738, 697, 647 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{21}H_{36}O_3SiNa^+$ 387.2331, found 387.2340 (2.3 ppm error).



(*E*)-di-*tert*-butyl((1-phenylhept-5-en-2-yl)oxy)silanol

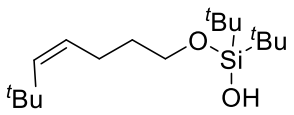
Compound S35: Synthesized using General Procedure A on a 4.9 mmol scale (Note modification of the procedure outlined in *Org. Lett.* **2022**, *24*, 6202–6207: $(t\text{-Bu})_2Si(OTf)_2$/2,6-lutidine, reaction time 18 h, reaction temperature 0 °C to 23 °C); Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 1.29 g, 3.70 mmol, 75% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.25 (m, 2H), 7.24 – 7.18 (m, 3H), 5.50 – 5.34 (m, 2H), 4.29 – 4.19 (m, 1H), 2.86 – 2.75 (m, 2H), 2.20 – 2.04 (m, 2H), 1.67 – 1.54 (m, 5H), 1.32 (s, 1H), 1.00 (s, 9H), 0.94 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 139.6, 131.3, 129.8, 128.4, 126.4, 125.1, 74.1, 43.6, 36.9, 28.2, 27.9, 27.8, 20.7, 20.6, 18.1.

IR ν 3644, 2931, 2859, 1497, 1471, 1454, 1364, 1012, 965, 827, 750, 645 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{21}H_{36}O_2SiNa^+$ 371.2382, found 371.2409 (7.3 ppm error).



(*Z*)-di-*tert*-butyl((6,6-dimethylhept-4-en-1-yl)oxy)silanol

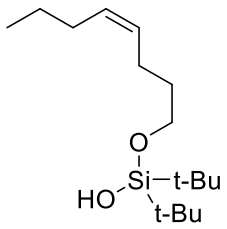
Compound S39: Synthesized using General Procedure A on a 2.69 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 494 mg, 1.64 mmol, 61% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.33 (dt, *J* = 11.9, 1.7 Hz, 1H), 5.16 (dt, *J* = 11.9, 7.4 Hz, 1H), 3.81 (t, *J* = 6.4 Hz, 2H), 2.26 (qd, *J* = 7.5, 1.7 Hz, 2H), 1.83 (s, 1H), 1.65 – 1.55 (m, 2H), 1.11 (s, 9H), 1.02 (s, 18H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 140.3, 128.5, 63.2, 33.7, 33.3, 31.3, 27.6, 24.9, 20.6.

IR ν 3499, 2960, 2934, 2860, 1473, 1363, 1109, 827, 750, 647 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H^+] Calcd $C_{17}H_{37}O_2Si^+$ 301.2563, found 301.2566 (1.0 ppm error).



(*Z*)-di-*tert*-butyl(oct-4-en-1-yloxy)silanol

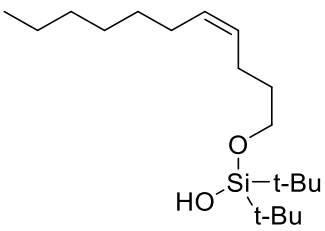
Compound S41: Synthesized using General Procedure A on a 2 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 430 mg, 1.50 mmol, 75% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.47 – 5.35 (m, 2H), 3.83 (t, J = 6.4 Hz, 2H), 2.20 – 2.10 (m, 2H), 2.10 – 2.00 (m, 2H), 1.68 – 1.56 (m, 2H), 1.39 (h, J = 7.3 Hz, 2H), 1.05 (s, 18H), 0.92 (t, J = 7.4 Hz, 3H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 130.3, 129.5, 63.1, 33.2, 29.4, 27.6, 23.7, 23.0, 20.6, 13.9.

IR ν 3649, 2933, 2860, 1558, 1473, 1106, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{16}H_{34}O_2SiNa^+$ 309.2226, found 309.2213 (4.2 ppm error).



(*Z*)-di-*tert*-butyl(undec-4-en-1-yloxy)silanol

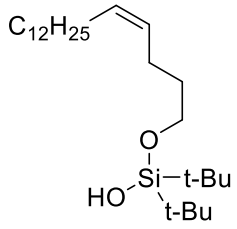
Compound S43: Synthesized using General Procedure A on a 2 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 519 mg, 1.58 mmol, 79% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.44 – 5.32 (m, 2H), 3.80 (t, J = 6.4 Hz, 2H), 2.07 (t, 2H), 2.07 – 1.98 (m, 2H), 1.65 – 1.51 (m, 2H), 1.38 – 1.22 (m, 8H), 1.02 (s, 18H), 0.92 – 0.84 (m, 3H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 130.6, 129.3, 63.1, 33.2, 31.9, 29.9, 29.2, 27.6, 27.4, 23.7, 22.8, 20.6, 14.2.

IR ν 3566, 2930, 2859, 1588, 1471, 1106, 828 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] $C_{19}H_{40}O_2SiNa^+$ 351.2695, found 351.2710 (4.3 ppm error).



(*Z*)-di-*tert*-butyl(heptadec-4-en-1-yloxy)silanol

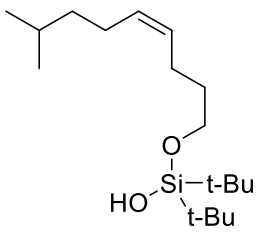
Compound S45: Synthesized using General Procedure A on a 2 mmol scale.; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 563 mg, 1.36 mmol, 68% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.47 – 5.34 (m, 2H), 3.83 (t, J = 6.4 Hz, 2H), 2.15 (td, J = 7.7, 6.0 Hz, 2H), 2.10 – 2.02 (m, 2H), 1.68 – 1.57 (m, 2H), 1.29 (s, 20H), 1.05 (s, 18H), 0.91 (t, J = 6.8 Hz, 3H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 130.6, 129.3, 63.1, 33.2, 32.1, 29.9, 29.8 (2C), 29.7, 29.5, 27.6, 27.4, 23.7, 22.9, 20.6, 14.3.

IR ν 3566, 2933, 2856, 1558, 1471, 1106, 828 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] $C_{25}H_{52}O_2SiNa^+$ 435.3634, found 435.3642 (1.8 ppm error).



(*Z*)-di-*tert*-butyl((8-methylnon-4-en-1-yl)oxy)silanol

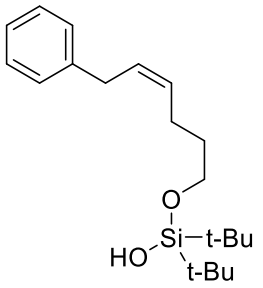
Compound S47: Synthesized using General Procedure A on a 2 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 459 mg, 1.46 mmol, 73% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.43 – 5.33 (m, 2H), 3.83 (t, J = 6.4 Hz, 2H), 2.19 – 2.12 (m, 2H), 2.10 – 1.99 (m, 2H), 1.68 – 1.51 (m, 3H), 1.31 – 1.20 (m, 2H), 1.05 (s, 18H), 0.91 (d, J = 6.6 Hz, 6H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 130.7, 129.1, 63.1, 39.1, 33.2, 27.8, 27.6, 25.3, 23.7, 22.7, 20.6.

IR ν 3649, 2933, 1652, 1507, 1473, 1107, 828 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] $C_{18}H_{38}O_2SiNa^+$ 337.2539, found 337.2532 (2.1 ppm error).



(*Z*)-di-*tert*-butyl((6-phenylhex-4-en-1-yl)oxy)silanol

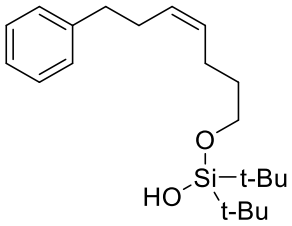
Compound S49: Synthesized using General Procedure A on a 2 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 401 mg, 1.20 mmol, 60% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (s, 2H), 7.29 – 7.20 (m, 3H), 5.72 – 5.54 (m, 2H), 3.89 (q, J = 6.6 Hz, 2H), 3.48 (d, J = 6.8 Hz, 2H), 2.37 – 2.27 (m, 2H), 1.78 – 1.66 (m, 2H), 1.12 – 1.07 (m, 18H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 141.3, 130.4, 128.7, 128.52, 128.45, 125.9, 62.9, 33.6, 33.0, 27.6, 23.7, 20.6.

IR ν 3647, 2931, 1652, 1471, 1012, 828 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] $C_{20}H_{34}O_2SiNa^+$ 357.2226, found 357.2242 (4.5 ppm error).



(*Z*)-di-*tert*-butyl((7-phenylhept-4-en-1-yl)oxy)silanol

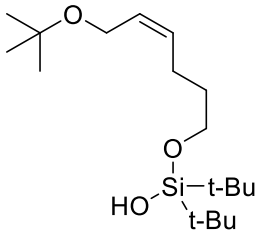
Compound S51: Synthesized using General Procedure A on a 2 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 513 mg, 1.47 mmol, 73% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.29 (m, 2H), 7.28 – 7.22 (m, 3H), 5.57 – 5.41 (m, 2H), 3.83 (t, J = 6.4 Hz, 2H), 2.72 (dd, J = 8.7, 6.9 Hz, 2H), 2.48 – 2.36 (m, 2H), 2.14 (ddd, J = 8.2, 6.4, 4.7 Hz, 2H), 1.60 (dq, J = 8.2, 6.5 Hz, 2H), 1.08 (s, 18H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 142.2, 130.2, 129.3, 128.6, 128.4, 125.9, 63.0, 36.1, 33.0, 29.3, 27.6, 23.7, 20.6.

IR ν 3566, 2931, 1471, 1455, 1075, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{21}H_{36}O_2SiNa^+$ 371.2382, found 371.2369 (3.5 ppm error).



(*Z*)-((6-(*tert*-butoxy)hex-4-en-1-yl)oxy)di-*tert*-butylsilanol

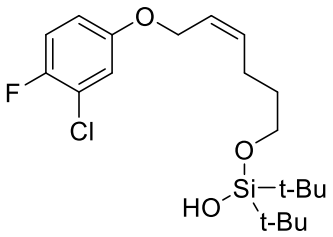
Compound S53: Synthesized using General Procedure A on a 2 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 397 mg, 1.20 mmol, 60% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.60 – 5.43 (m, 2H), 4.00 – 3.93 (m, 2H), 3.80 (t, J = 6.1 Hz, 2H), 2.18 (dt, J = 7.4, 6.2 Hz, 2H), 1.62 (tt, J = 7.3, 6.0 Hz, 2H), 1.23 (s, 9H), 1.02 (s, 18H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 132.0, 128.1, 73.4, 62.4, 57.7, 32.6, 27.69, 27.65, 23.8, 20.5.

IR ν 3566, 2968, 2857, 1558, 1471, 1106, 828 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] $C_{18}H_{38}O_3SiNa^+$ 353.2488, found 353.2495 (2 ppm error).



(*Z*)-di-*tert*-butyl((6-(3-chloro-4-fluorophenoxy)hex-4-en-1-yl)oxy)silanol

Compound S55: Synthesized using General Procedure A on a 2 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 564 mg, 1.40 mmol, 70% yield).

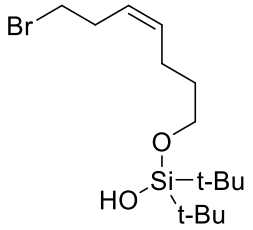
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.02 (td, J = 8.9, 1.0 Hz, 1H), 6.92 (dd, J = 6.0, 3.0 Hz, 1H), 6.75 (dt, J = 9.1, 3.4 Hz, 1H), 5.76 – 5.61 (m, 2H), 4.54 (d, J = 5.2 Hz, 2H), 3.82 (t, J = 6.1 Hz, 2H), 2.30 – 2.18 (m, 2H), 1.65 (tt, J = 7.4, 6.1 Hz, 2H), 1.01 (s, 18H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 155.0 (d, J = 2.3 Hz), 152.9 (d, J = 241.3 Hz), 134.6, 124.6, 121.1 (d, J = 19.1 Hz), 116.8 (d, J = 22.7 Hz), 116.5, 114.6 (d, J = 6.7 Hz), 64.9, 62.5, 32.6, 27.6, 24.2, 20.6.

^{19}F NMR (377 MHz, $CDCl_3$) δ -127.1 (tt, J = 10.0, 5.4 Hz).

IR ν 3566, 2933, 2859, 1558, 1497, 1106, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{20}H_{32}FClO_3SiNa^+$ 425.1691, found 425.1693 (0.5 ppm error).



(*Z*)-((7-bromohept-4-en-1-yl)oxy)di-*tert*-butylsilanol

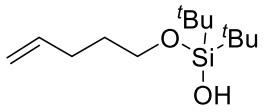
Compound S57: Synthesized using General Procedure A on a 2 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 491 mg, 1.40 mmol, 70% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.67 – 5.46 (m, 1H), 5.50 – 5.28 (m, 1H), 3.83 (t, J = 6.3 Hz, 2H), 3.39 (t, J = 7.1 Hz, 2H), 2.66 (qd, J = 7.2, 1.5 Hz, 2H), 2.18 (qd, J = 7.4, 1.6 Hz, 2H), 1.64 (tt, J = 7.3, 6.3 Hz, 2H), 1.05 (s, 18H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$)) δ 132.5, 126.4, 62.7, 32.8, 32.6, 30.9, 27.6, 23.8, 20.6.

IR ν 3566, 2933, 1558, 1471, 1107, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{15}H_{31}BrO_2SiNa^+$ 373.1174, found 373.1189 (4.0 ppm error).



di-*tert*-butyl(pent-4-en-1-yloxy)silanol

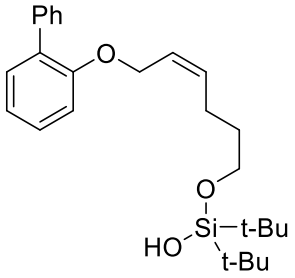
Compound S59: Synthesized using General Procedure A on a 4.0 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 655.0 mg, 2.68 mmol, 67% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.84 (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 5.08 – 4.92 (m, 2H), 3.80 (t, *J* = 6.4 Hz, 2H), 2.19 – 2.09 (m, 2H), 1.82 (s, 1H), 1.69 – 1.59 (m, 2H), 1.02 (s, 18H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 138.8, 114.7, 62.9, 32.2, 30.2, 27.6, 20.6.

IR ν 3512, 2933, 2859, 1641, 1471, 1105, 827, 645 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H$^+$] Calcd $C_{13}H_{29}O_2Si^+$ 245.1937, found 245.1937 (0.0 ppm error).



(*Z*)-((6-([1,1'-biphenyl]-2-yloxy)hex-4-en-1-yl)oxy)di-*tert*-butylsilanol

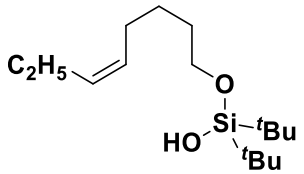
Compound S61: Synthesized using General Procedure A on a 2 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 597 mg, 1.40 mmol, 70% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 – 7.55 (m, 2H), 7.48 – 7.27 (m, 5H), 7.11 – 6.98 (m, 2H), 5.73 – 5.57 (m, 2H), 4.64 (d, J = 5.2 Hz, 2H), 3.82 (td, J = 6.3, 4.3 Hz, 2H), 2.27 – 2.12 (m, 2H), 1.71 – 1.59 (m, 2H), 1.05 (s, 18H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 155.7, 138.7, 133.1, 131.3, 131.1, 129.7, 128.5, 128.0, 126.9, 125.7, 121.2, 113.3, 64.8, 62.6, 32.6, 27.6, 24.2, 20.5.

IR ν 3647, 2931, 2857, 1558, 1471, 1106, 828 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{26}H_{38}O_3SiNa^+$ 449.2488, found 449.2507 (4.2 ppm error).



(*Z*)-di-*tert*-butyl(oct-5-en-1-yloxy)silanol

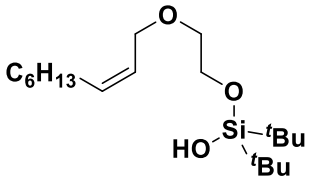
Compound S63: Synthesized using General Procedure A on a 5.0 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 1.25 g, 4.36 mmol, 87% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.44 – 5.26 (m, 2H), 3.79 (t, *J* = 6.4 Hz, 2H), 2.10 – 1.96 (m, 4H), 1.80 (s, 1H), 1.60 – 1.51 (m, 2H), 1.46 – 1.37 (m, 2H), 1.01 (s, 18H), 0.95 (t, *J* = 7.5 Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 131.9, 129.2, 63.5, 32.7, 27.6, 27.0, 26.1, 20.7, 20.6, 14.5.

IR ν 3510, 2933, 2859, 1471, 1107, 828, 750, 647 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H$^+$] Calcd $C_{16}H_{35}O_2Si^+$ 287.2406, found 287.2420 (4.9 ppm error).



(*Z*)-di-*tert*-butyl(2-(non-2-en-1-yloxy)ethoxy)silanol

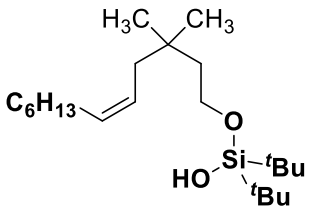
Compound S65: Synthesized using General Procedure A on a 4.0 mmol scale; Purified using a gradient of 30-100% CH_2Cl_2 in hexane on silica gel; (colorless oil, 965 mg, 2.8 mmol, 70% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.65 – 5.56 (m, 1H), 5.56 – 5.47 (m, 1H), 4.05 (dd, *J* = 6.4, 1.2 Hz, 2H), 4.00 – 3.94 (m, 2H), 3.54 – 3.48 (m, 2H), 2.09 – 1.98 (m, 2H), 1.39 – 1.23 (m, 8H), 1.01 (s, 18H), 0.91 – 0.85 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 134.7, 125.0, 72.5, 67.0, 63.3, 31.8, 29.6, 29.0, 27.7, 27.5, 22.8, 20.7, 14.2.

IR ν 3469, 2931, 2857, 1473, 1136, 1097, 869, 828, 750, 648 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{19}H_{40}O_3SiNa^+$ 367.2644, found 367.2645 (0.3 ppm error).



(*Z*)-di-*tert*-butyl((3,3-dimethyldodec-5-en-1-yl)oxy)silanol

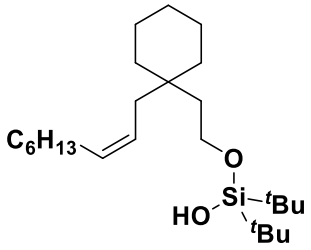
Compound S67: Synthesized using General Procedure A on a 2.0 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 618 mg, 1.67 mmol, 83% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.51 – 5.35 (m, 2H), 3.89 – 3.81 (m, 2H), 2.06 – 1.97 (m, 2H), 1.95 (d, *J* = 6.5 Hz, 2H), 1.78 (s, 1H), 1.56 – 1.47 (m, 2H), 1.37 – 1.23 (m, 8H), 1.01 (s, 18H), 0.91 – 0.85 (m, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 131.9, 126.0, 60.6, 44.5, 39.9, 33.1, 32.0, 29.8, 29.2, 27.6, 27.5, 27.4, 22.8, 20.6, 14.3.

IR ν 3506, 2928, 2857, 1470, 1364, 1097, 828, 647 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H$^+$] Calcd $C_{22}H_{47}O_2Si^+$ 371.3345, found 371.3356 (3.0 ppm error).



(*Z*)-di-*tert*-butyl(2-(1-(non-2-en-1-yl)cyclohexyl)ethoxy)silanol

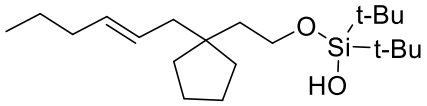
Compound S69: Synthesized using General Procedure A on a 4.0 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 1.20 g, 2.93 mmol, 73% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.54 – 5.32 (m, 2H), 3.89 – 3.75 (m, 2H), 2.08 – 1.97 (m, 4H), 1.75 (s, 1H), 1.61 – 1.53 (m, 2H), 1.48 – 1.39 (m, 5H), 1.38 – 1.24 (m, 13H), 1.01 (s, 18H), 0.92 – 0.86 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 131.9, 125.6, 59.9, 39.8, 36.1, 35.4, 35.3, 31.9, 29.9, 29.2, 27.7, 27.6, 26.6, 22.8, 21.9, 20.6, 14.3.

IR ν 2928, 2857, 1471, 1095, 827, 647 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{25}H_{50}O_2SiNa^+$ 433.3478, found 433.3484 (1.4 ppm error).



(*E*)-di-*tert*-butyl(2-(1-(hex-2-en-1-yl)cyclopentyl)ethoxy)silanol

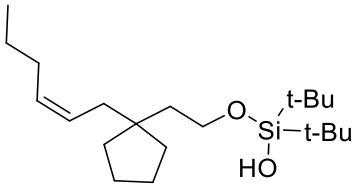
Compound S71: Synthesized using General Procedure A on a 2 mmol scale; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 425 mg, 1.20 mmol, 60% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.44 – 5.34 (m, 2H), 3.87 – 3.79 (m, 2H), 2.02 – 1.92 (m, 4H), 1.65 – 1.51 (m, 6H), 1.48 – 1.30 (m, 6H), 1.01 (s, 18H), 0.89 (t, J = 7.4 Hz, 3H).

$^{13}C\{^{1}H\}$ (101 MHz, $CDCl_3$) δ 132.7, 127.6, 60.9, 44.2, 41.9, 41.8, 37.6, 34.9, 27.6, 24.6, 22.8, 20.5, 13.8.

IR ν 3446, 2933, 1558, 1507, 1471, 1106, 828 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{21}H_{42}O_2SiNa^+$ 377.2852, found 377.2863 (2.9 ppm error).



(*Z*)-di-*tert*-butyl(2-(1-(hex-2-en-1-yl)cyclopentyl)ethoxy)silanol

Compound S73: Synthesized using General Procedure A on a 2 mmol scale.; Purified using a gradient of 10-30% CH_2Cl_2 in hexane on silica gel; (colorless oil, 461 mg, 1.30 mmol, 65% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.54 – 5.39 (m, 2H), 3.90 – 3.81 (m, 2H), 2.03 (tq, J = 6.5, 2.3 Hz, 4H), 1.69 – 1.56 (m, 6H), 1.49 – 1.36 (m, 6H), 1.04 (s, 18H), 0.93 (t, J = 7.4 Hz, 3H).

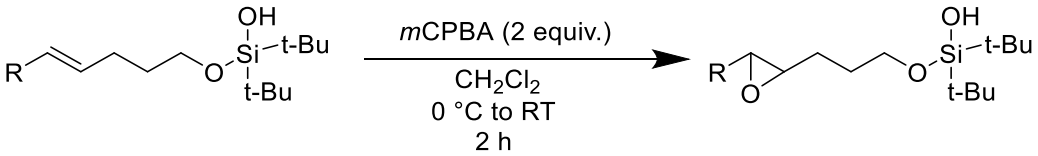
$^{13}C\{^{1}H\}$ (101 MHz, $CDCl_3$) δ 131.4, 126.9, 61.1, 44.3, 41.9, 37.7, 35.9, 29.7, 27.6, 24.7, 23.0, 20.6, 14.0.

IR ν 3446, 2938, 1558, 1507, 1471, 1107, 828 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{21}H_{42}O_2SiNa^+$ 377.2852, found 377.2867 (4.0 ppm error).

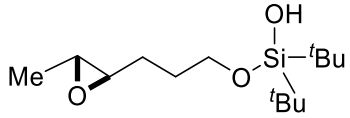
V. Preparation of Silanol Epoxides

General Procedure B



m-CPBA (2.0 equiv) was added to an ice-cold solution of alkenyl silanol substrate (1.0 equiv) in CH_2Cl_2 (0.1 M concentration). The reaction mixture was allowed to warm to room temperature over a period of two hours. Next, the reaction was quenched with saturated aqueous $Na_2S_2O_3$ solution and transferred to a separatory funnel. The aqueous layer was removed, and the organic layer was washed with one portion of 0.5 M aqueous NaOH solution. The organic layer was collected, dried with Na_2SO_4, and concentrated under reduced pressure. The resulting residue was purified by silica gel column chromatography (specific conditions are associated with each compound).

VI. Characterization of Silanol Epoxides



di-*tert*-butyl(3-((2*R**,3*R**)-3-methyloxiran-2-yl)propoxy)silanol

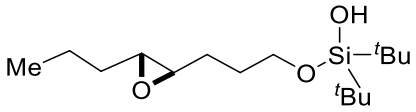
Compound 1: Synthesized using General Procedure B on a 3.87 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 1.03 g, 3.75 mmol, 97% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.92 – 3.78 (m, 2H), 2.82 – 2.70 (m, 2H), 2.67 (ddd, *J* = 6.6, 3.9, 2.3 Hz, 1H), 1.79 – 1.62 (m, 3H), 1.62 – 1.49 (m, 1H), 1.29 (d, *J* = 5.2 Hz, 3H), 1.00 (s, 9H), 0.99 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 62.6, 60.2, 54.6, 29.8, 28.2, 27.7, 27.6, 20.8, 20.6, 17.7.

IR ν 3437, 2933, 2860, 1473, 1130, 650 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{14}H_{30}O_3SiNa^+$ 297.1862, Found 297.1871 (3.0 ppm error).



di-*tert*-butyl(3-((2*R**,3*R**)-3-propyloxiran-2-yl)propoxy)silanol

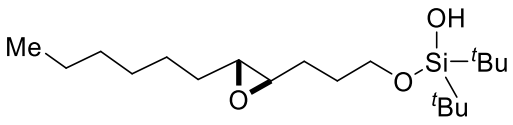
Compound 3: Synthesized using General Procedure B on a 1.1 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 312 mg, 1.03 mmol, 94% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.94 – 3.78 (m, 2H), 2.75 – 2.67 (m, 2H), 2.66 (s, 1H), 1.77 – 1.65 (m, 3H), 1.61 – 1.38 (m, 5H), 1.01 (s, 9H), 1.00 (s, 9H), 0.95 (t, *J* = 7.1 Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 62.6, 59.2, 58.6, 34.2, 29.8, 28.3, 27.7, 27.6, 20.8, 20.6, 19.4, 14.1.

IR ν 3442, 2961, 2933, 2859, 1473, 1106, 827, 647 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{16}H_{34}O_3SiNa^+$ 325.2175, Found 325.2185 (3.1 ppm error).



di-*tert*-butyl(3-((2*R**,3*R**)-3-hexyloxiran-2-yl)propoxy)silanol

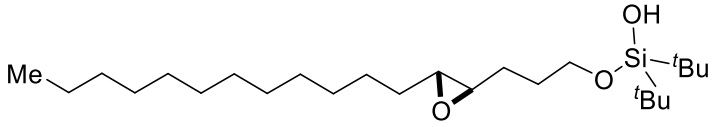
Compound 5: Synthesized using General Procedure B on a 1.26 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 380 mg, 1.10 mmol, 87% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.89 – 3.75 (m, 2H), 3.03 (s, 1H), 2.72 – 2.61 (m, 2H), 1.73 – 1.61 (m, 3H), 1.59 – 1.34 (m, 5H), 1.33 – 1.19 (m, 6H), 0.97 (s, 9H), 0.96 (s, 9H), 0.87 – 0.82 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 62.6, 59.3, 58.8, 32.2, 31.9, 29.9, 29.3, 28.3, 27.7, 27.6, 26.1, 22.7, 20.8, 20.6, 14.2.

IR ν 3475, 2931, 2857, 1473, 1103, 827, 648 cm^{-1}

HRMS (ESI) m/z = [M + H$^+$] calculated for $C_{19}H_{41}O_3Si^+$ 345.2825, Found 345.2822 (error 0.9 ppm).



di-*tert*-butyl(3-((2*R**,3*R**)-3-dodecyloxiran-2-yl)propoxy)silanol

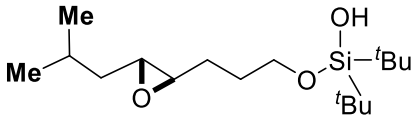
Compound 7: Synthesized using General Procedure B on a 0.75 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 288 mg, 0.672 mmol, 90% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.94 – 3.77 (m, 2H), 2.75 – 2.54 (m, 3H), 1.77 – 1.64 (m, 3H), 1.61 – 1.47 (m, 3H), 1.47 – 1.36 (m, 2H), 1.34 – 1.19 (m, 18H), 1.01 (s, 9H), 1.00 (s, 9H), 0.87 (t, *J* = 6.8 Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 62.6, 59.3, 58.8, 32.2, 32.1, 29.9, 29.8, 29.78, 29.70, 29.6, 29.5, 28.3, 27.7, 27.6, 26.1, 22.8, 20.8, 20.6, 14.3.

IR ν 2927, 2856, 1473, 1105, 827, 647 cm^{-1}.

HRMS (ESI) m/z = [M + Na$^+$] calculated for $C_{25}H_{52}O_3SiNa^+$ 451.3578, Found 451.3583 (1.1 ppm error).



di-*tert*-butyl(3-((2*R**,3*R**)-3-isobutyloxiran-2-yl)propoxy)silanol

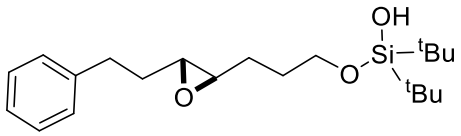
Compound 9: Synthesized using General Procedure B on a 1.0 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 300.0 mg, 0.948 mmol, 95% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.94 – 3.79 (m, 2H), 2.75 – 2.58 (m, 3H), 1.86 – 1.66 (m, 4H), 1.56 (ddt, *J* = 14.7, 7.0, 5.2 Hz, 1H), 1.47 (dt, *J* = 13.9, 6.1 Hz, 1H), 1.33 (ddd, *J* = 13.6, 7.7, 5.4 Hz, 1H), 1.01 (s, 9H), 1.00 (s, 9H), 0.96 (d, *J* = 4.5 Hz, 3H), 0.94 (d, *J* = 4.4 Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 62.6, 59.4, 57.7, 41.2, 29.8, 28.3, 27.7, 27.6, 26.5, 23.2, 22.6, 20.8, 20.6.

IR ν 3480, 2960, 2933, 2859, 1471, 1106, 827, 648 cm^{-1}.

HRMS (ESI) m/z = [M + Na$^+$] calculated for $C_{17}H_{36}O_3SiNa^+$ 339.2331, Found 339.2340 (2.7 ppm error).



di-*tert*-butyl(3-((2*R**,3*R**)-3-phenethyloxiran-2-yl)propoxy)silanol

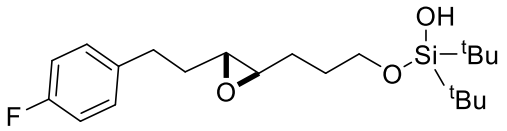
Compound 11: Synthesized using General Procedure B on a 2.24 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 547 mg, 1.5 mmol, 67% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.26 (m, 2H), 7.23 – 7.15 (m, 3H), 3.92 – 3.77 (m, 2H), 2.82 (ddd, *J* = 14.4, 8.1, 6.6 Hz, 1H), 2.77 – 2.63 (m, 3H), 2.60 – 2.51 (m, 1H), 1.93 – 1.78 (m, 2H), 1.72 – 1.49 (m, 4H), 1.02 (s, 9H), 1.00 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 141.4, 128.6, 128.5, 126.2, 62.6, 59.5, 58.1, 33.9, 32.4, 29.7, 28.2, 27.7, 27.6, 20.8, 20.6.

IR ν 3478, 2933, 2857, 1471, 1104, 827, 699, 648 cm^{-1}

HRMS (ESI) m/z = [M + H$^+$] calculated for $C_{21}H_{37}O_3Si^+$ 365.2512, Found 365.2506 (1.6 ppm error).



di-*tert*-butyl(3-(-3-(4-fluorophenethyl)oxiran-2-yl)propoxy)silanol

Compound 13: Synthesized using General Procedure B on a 1.25 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 443 mg, 1.16 mmol, 92% yield).

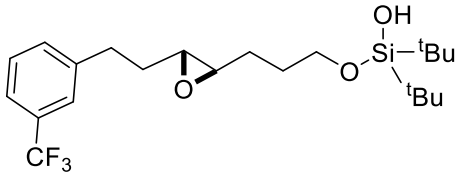
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 – 7.09 (m, 2H), 7.02 – 6.92 (m, 2H), 3.90 – 3.77 (m, 2H), 2.78 (dt, J = 14.4, 7.0 Hz, 1H), 2.72 (m, 4H), 1.82 (tdd, J = 7.5, 5.6, 2.4 Hz, 2H), 1.74 – 1.57 (m, 3H), 1.58 – 1.45 (m, 1H), 1.01 (s, 9H), 1.00 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 161.5 (d, J = 243.9 Hz), 137.0 (d, J = 3.5 Hz), 129.8 (d, J = 8.0 Hz), 115.3 (d, J = 21.3 Hz), 62.6, 59.4, 58.0, 34.0, 31.6, 29.7, 28.2, 27.67, 27.61, 20.7, 20.6.

^{19}F NMR (377 MHz, $CDCl_3$) δ -118.0 (ddd, J = 14.1, 9.1, 5.6 Hz).

IR ν 3487, 2965, 2859, 1510, 1106, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{21}H_{35}FO_3SiNa^+$ 405.2237, found 405.2240 (0.7 ppm error).



di-*tert*-butyl(3-(3-(4-(trifluoromethyl)phenethyl)oxiran-2-yl)propoxy)silanol

Compound 15: Synthesized using General Procedure B on a 1.3 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 393 mg, 0.908 mmol, 70% yield).

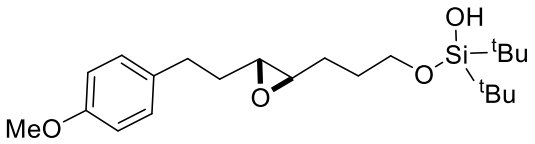
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, J = 8.6 Hz, 2H), 7.41 – 7.32 (m, 2H), 3.84 (td, J = 5.7, 2.4 Hz, 2H), 2.93 – 2.66 (m, 4H), 1.96 – 1.77 (m, 2H), 1.73 – 1.50 (m, 4H), 1.01 (s, 9H), 1.00 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 142.2, 131.94, 130.9 (q, *J* = 31.9 Hz), 129.0, 125.7 (q, *J* = 271.7 Hz), 125.2 (q, *J* = 3.7 Hz), 123.1 (q, *J* = 3.9 Hz), 62.6, 59.4, 57.9, 33.7, 32.2, 29.6, 28.2, 27.6, 27.5, 20.7, 20.5.

^{19}F NMR (377 MHz, $CDCl_3$) δ -63.2.

IR ν 3499, 2958, 2860, 1471, 1330, 1075, 828 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{22}H_{35}F_3O_3SiNa^+$ 455.2205, found 455.2183 (4.8 ppm error).



di-*tert*-butyl(3-((2*R**,3*R**)-3-(4-methoxyphenethyl)oxiran-2-yl)propoxy)silanol

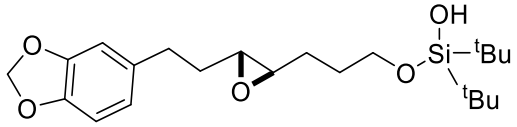
Compound 17: Synthesized using General Procedure B on a 1.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 417 mg, 1.06 mmol, 88% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.11 (d, *J* = 8.5 Hz, 2H), 6.83 (d, *J* = 8.6 Hz, 2H), 3.90 – 3.80 (m, 2H), 3.79 (s, 3H), 2.80 – 2.61 (m, 4H), 2.58 (s, 1H), 1.81 (td, *J* = 7.6, 5.4 Hz, 2H), 1.73 – 1.50 (m, 4H), 1.02 (s, 9H), 1.00 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 158.0, 133.4, 129.4, 114.0, 62.6, 59.4, 58.1, 55.4, 34.1, 31.4, 29.7, 28.2, 27.7, 27.6, 20.7, 20.6.

IR ν 3480, 2933, 2859, 1652, 1507, 1473, 1246, 1106, 827, 647 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calcd for $C_{22}H_{38}O_4SiNa^+$ 417.2437, found 417.2434 (0.7 ppm error).



(3-(3-(2-(benzo[*d*][1,3]dioxol-5-yl)ethyl)oxiran-2-yl)propoxy)di-*tert*-butylsilanol

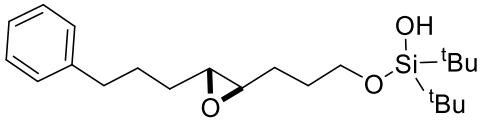
Compound 19: Synthesized using General Procedure B on a 1.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 302 mg, 0.739 mmol, 62% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.75 – 6.58 (m, 3H), 5.91 (s, 2H), 3.83 (tt, J = 6.7, 3.8 Hz, 2H), 2.81 – 2.56 (m, 4H), 1.79 (td, J = 7.7, 5.5 Hz, 2H), 1.71 – 1.60 (m, 3H), 1.58 – 1.50 (m, 1H), 1.01 (s, 9H), 1.00 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 147.7, 145.9, 135.2, 121.2, 108.9, 108.3, 100.9, 62.6, 59.4, 58.0, 34.1, 32.1, 29.7, 28.2, 27.65, 27.60, 20.7, 20.6.

IR ν 3497, 2961, 2857, 1445, 1246, 1040, 855 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{22}H_{36}O_5SiNa^+$ 431.2230, found 431.2237 (1.6 ppm error).



di-*tert*-butyl(3-((2*R**,3*R**)-3-(3-phenylpropyl)oxiran-2-yl)propoxy)silanol

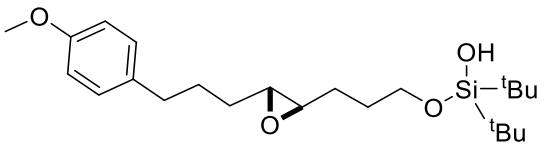
Compound 21: Synthesized using General Procedure B on a 1.53 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 522 mg, 1.38 mmol, 90% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.26 (m, 2H), 7.22 – 7.13 (m, 3H), 3.95 – 3.78 (m, 2H), 2.75 – 2.68 (m, 2H), 2.66 (t, *J* = 7.7 Hz, 2H), 2.60 (s, 1H), 1.85 – 1.65 (m, 5H), 1.62 – 1.48 (m, 3H), 1.02 (s, 9H), 1.00 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 142.1, 128.53, 128.48, 126.0, 62.6, 59.1, 58.6, 35.8, 31.7, 29.8, 28.3, 27.9, 27.7, 27.6, 20.8, 20.6.

IR ν 2933, 2859, 1558, 1507, 1473, 1457, 1106, 827, 698, 647 cm^{-1}.

HRMS (ESI) m/z = [M + H$^+$] calculated for $C_{22}H_{39}O_3Si^+$ 379.2668, Found 379.2662 (1.6 ppm error).



di-*tert*-butyl(3-(3-(3-(4-methoxyphenyl)propyl)oxiran-2-yl)propoxy)silanol

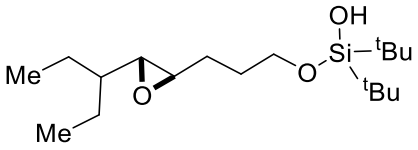
Compound 23: Synthesized using General Procedure B on a 1.4 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 490 mg, 1.20 mmol, 86% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 – 7.05 (m, 2H), 6.86 – 6.78 (m, 2H), 3.90 – 3.82 (m, 2H), 3.78 (s, 3H), 2.75 – 2.67 (m, 2H), 2.59 (t, J = 7.6 Hz, 2H), 1.85 – 1.61 (m, 5H), 1.55 (dddd, J = 9.0, 6.9, 5.2, 3.6 Hz, 3H), 1.01 (s, 9H), 1.00 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 157.9, 134.2, 129.4, 113.9, 62.6, 59.1, 58.6, 55.4, 34.8, 31.6, 29.8, 28.3, 28.1, 27.68, 27.62, 20.8, 20.6.

IR ν 3503, 2857, 1612, 1511, 1038, 1177, 828 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd for $C_{22}H_{38}O_4SiNa^+$ 417.2437, found 417.2450 (3.1 ppm error).



di-*tert*-butyl(3-((2*R**,3*R**)-3-(pentan-3-yl)oxiran-2-yl)propoxy)silanol

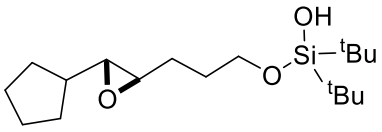
Compound 25: Synthesized using General Procedure B on a 1.13 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 345 mg, 1.04 mmol, 92% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.90 – 3.78 (m, 2H), 2.86 (s, 1H), 2.71 (ddd, *J* = 7.4, 3.8, 2.3 Hz, 1H), 2.45 (dd, *J* = 8.3, 2.4 Hz, 1H), 1.78 – 1.62 (m, 3H), 1.59 – 1.36 (m, 4H), 1.35 – 1.22 (m, 1H), 1.04 – 0.94 (m, 19H), 0.93 – 0.86 (m, 6H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 62.6, 62.4, 58.8, 44.2, 29.9, 28.3, 27.7, 27.6, 25.3, 23.8, 20.8, 20.6, 12.0, 11.2.

IR ν 3445, 2963, 2933, 2859, 1473, 1106, 827, 750, 647 cm^{-1}.

HRMS (ESI) m/z = [M + Na$^+$] calculated for $C_{18}H_{38}O_3SiNa^+$ 353.2488, Found 353.2501 (3.7 ppm error).



di-*tert*-butyl(3-((2*R**,3*R**)-3-cyclopentyloxiran-2-yl)propoxy)silanol

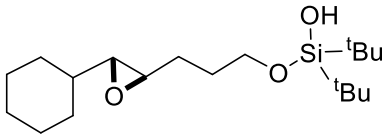
Compound 27: Synthesized using General Procedure B on a 1.0 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 296.6 mg, 0.903 mmol, 90% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.92 – 3.80 (m, 2H), 2.73 (ddd, *J* = 6.3, 3.9, 2.4 Hz, 1H), 2.67 – 2.52 (m, 2H), 1.83 – 1.51 (m, 11H), 1.47 – 1.30 (m, 2H), 1.01 (s, 9H), 1.00 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 62.6, 62.2, 58.6, 41.5, 29.9, 29.4, 28.9, 28.3, 27.7, 27.6, 25.5, 20.8, 20.6.

IR ν 2954, 2944, 2860, 1473, 1106, 827, 648 cm^{-1}.

HRMS (ESI) m/z = [M + Na$^+$] calculated for $C_{18}H_{36}O_3SiNa^+$ 351.2331, Found 351.2341 (2.8 ppm error).



di-*tert*-butyl(3-((2*R**,3*R**)-3-cyclohexyloxiran-2-yl)propoxy)silanol

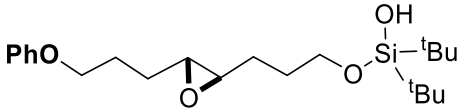
Compound 29: Synthesized using General Procedure B on a 2.14 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 705 mg, 2.06 mmol, 96% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.93 – 3.76 (m, 2H), 2.76 (ddd, *J* = 6.7, 4.1, 2.4 Hz, 1H), 2.61 (s, 1H), 2.48 (dd, *J* = 6.8, 2.3 Hz, 1H), 1.88 – 1.81 (m, 1H), 1.76 – 1.56 (m, 8H), 1.28 – 1.05 (m, 6H), 1.01 (s, 9H), 1.00 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 63.2, 62.6, 58.1, 40.2, 29.9, 29.8, 29.1, 28.4, 27.7, 27.6, 26.4, 25.8, 25.7, 20.8, 20.6.

IR ν 2928, 2856, 1473, 1097, 827, 647 cm^{-1}.

HRMS (ESI) m/z = [M + Na$^+$] calculated for $C_{19}H_{38}O_3SiNa^+$ 365.2488, Found 365.2493 (1.4 ppm error).



di-*tert*-butyl(3-((2*R**,3*R**)-3-(3-phenoxypropyl)oxiran-2-yl)propoxy)silanol

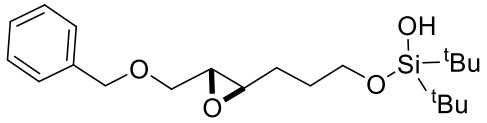
Compound 31: Synthesized using General Procedure B on a 0.978 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 303 mg, 0.768 mmol, 79% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 – 7.22 (m, 2H), 6.94 – 6.82 (m, 3H), 4.03 – 3.91 (m, 2H), 3.89 – 3.76 (m, 2H), 2.81 – 2.68 (m, 2H), 2.58 (s, 1H), 2.00 – 1.83 (m, 2H), 1.82 – 1.51 (m, 6H), 0.99 (s, 9H), 0.97 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.0, 129.6, 120.8, 114.6, 67.3, 62.6, 59.2, 58.3, 29.8, 28.9, 28.2, 27.7, 27.6, 26.0, 20.8, 20.6.

IR ν 2933, 2859, 2358, 2330, 1473, 1276, 1260, 827, 750 cm^{-1}.

HRMS (ESI) m/z = [M + Na$^+$] calculated for $C_{22}H_{38}O_4SiNa^+$ 417.2437, Found 417.2435 (0.5 ppm error).



(3-((2*R**,3*R**)-3-((benzyloxy)methyl)oxiran-2-yl)propoxy)di-*tert*-butylsilanol

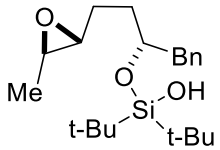
Compound 33: Synthesized using General Procedure B on a 1.6 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 408 mg, 1.07 mmol, 67% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.16 (m, 5H), 4.56 – 4.41 (m, 2H), 3.85 – 3.72 (m, 2H), 3.60 (dd, *J* = 11.4, 3.5 Hz, 1H), 3.43 (dd, *J* = 11.4, 5.4 Hz, 1H), 2.90 (ddd, *J* = 5.6, 3.5, 2.3 Hz, 1H), 2.80 (ddd, *J* = 6.6, 4.1, 2.3 Hz, 1H), 2.41 (s, 1H), 1.73 – 1.59 (m, 3H), 1.58 – 1.48 (m, 1H), 0.93 (s, 9H), 0.92 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 138.0, 128.6, 127.9 (2C), 73.5, 70.4, 62.6, 57.0, 56.5, 29.6, 28.0, 27.7, 27.6, 20.7, 20.6.

IR ν 2933, 2859, 2358, 1473, 1110, 827, 647 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{21}H_{36}O_4SiNa^+$ 403.2281, Found 403.2289 (2.0 ppm error).



di-*tert*-butyl(((*R**)-4-((2*R**,3*R**)-3-methyloxiran-2-yl)-1-phenylbutan-2-yl)oxy)silanol

Compound 35: Synthesized using General Procedure B on a 2.0 mmol scale; Purified by preparative TLC using 10% ether in hexane on silica gel; single diastereomer; (colorless oil, 253 mg, 0.694 mmol, 35% yield).

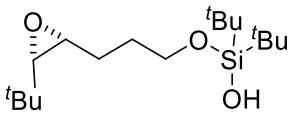
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.25 (m, 2H), 7.24 – 7.15 (m, 3H), 4.38 – 4.24 (m, 1H), 2.90 – 2.70 (m, 3H), 2.70 – 2.60 (m, 1H), 1.85 – 1.58 (m, 5H), 1.28 (d, *J* = 5.1 Hz, 3H), 1.00 (s, 9H), 0.94 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 139.2, 129.8, 128.4, 126.4, 73.8, 60.0, 54.8, 43.4, 32.6, 27.82, 27.78, 27.0, 20.7, 20.6, 17.8.

IR ν 3460, 2933, 2857, 1473, 1095, 827, 700, 645 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{21}H_{36}O_3SiNa^+$ 387.2331, Found 387.2350 (4.9 ppm error).

Compound 37: Previously characterized in *Org. Lett.* **2022**, *24*, 939–943.



di-*tert*-butyl(3-((2*R**,3*S**)-3-(*tert*-butyl)oxiran-2-yl)propoxy)silanol

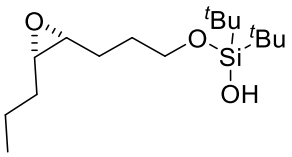
Compound 39: Synthesized using General Procedure B on a 1.64 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 507 mg, 1.6 mmol, 98% yield).

^{1}H NMR (400 MHz, $CDCl_3$) ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.89 (t, *J* = 5.8 Hz, 2H), 2.87 (dt, *J* = 6.9, 4.7 Hz, 1H), 2.67 (d, *J* = 4.5 Hz, 1H), 2.53 (s, 1H), 1.86 – 1.78 (m, 2H), 1.78 – 1.68 (m, 2H), 1.05 – 0.96 (m, 27H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 65.7, 62.7, 59.5, 31.8, 31.4, 27.9, 27.7, 27.6, 24.8, 20.8, 20.6.

IR ν 3471, 2961, 2933, 2859, 1471, 1106, 827, 648 cm^{-1}.

HRMS (ESI) m/z = [M + H$^+$] calculated for $C_{17}H_{37}O_3Si^+$ 317.2512, Found 317.2513 (0.3 ppm error).



di-*tert*-butyl(3-(3-propyloxiran-2-yl)propoxy)silanol

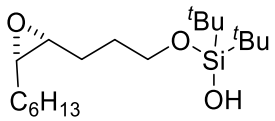
Compound 41: Synthesized using Procedure B on a 1.5 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 363 mg, 1.20 mmol, 80% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.94 – 3.86 (m, 2H), 2.96 (ddd, J = 6.0, 4.2, 2.4 Hz, 2H), 1.85 – 1.62 (m, 4H), 1.60 – 1.46 (m, 4H), 1.04 (s, 9H), 1.03 (s, 9H), 1.01 – 0.97 (m, 3H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 62.6, 57.5, 57.1, 30.3, 29.9, 27.68, 27.61, 24.2, 20.8, 20.5, 20.0, 14.1.

IR ν 3463, 2963, 2857, 1386, 1012, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{16}H_{34}O_3SiNa^+$ 325.2175, found 325.2166 (2.8 ppm error).



di-*tert*-butyl(3-(3-hexyloxiran-2-yl)propoxy)silanol

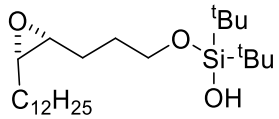
Compound 43: Synthesized using Procedure B on a 1.58 mmol scale.; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 457 mg, 1.33 mmol, 84% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.95 – 3.85 (m, 2H), 3.01 – 2.90 (m, 2H), 1.80 – 1.62 (m, 4H), 1.57 – 1.44 (m, 3H), 1.39 – 1.22 (m, 7H), 1.03 (s, 9H), 1.02 (s, 9H), 0.95 – 0.86 (m, 3H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 62.6, 57.6, 57.4, 31.9, 30.3, 29.3, 27.9, 27.68, 27.61, 26.6, 24.2, 22.7, 20.7, 20.5, 14.2.

IR ν 3480, 2961, 2859, 1471, 1012, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{19}H_{40}O_3SiNa^+$ 367.2644, found 367.2633 (3 ppm error).



di-*tert*-butyl(3-(3-dodecyloxiran-2-yl)propoxy)silanol

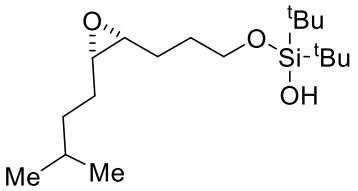
Compound 45: Synthesized using General Procedure B on a 1.36 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 524 mg, 1.22 mmol, 90% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.92 – 3.82 (m, 2H), 2.98 – 2.87 (m, 2H), 1.78 – 1.37 (m, 8H), 1.27 – 1.23 (m, 18H), 1.00 (s, 9H), 0.99 (s, 9H), 0.91 – 0.81 (m, 3H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 62.6, 57.6, 57.4, 32.1, 30.3, 29.80, 29.77, 29.70, 29.68, 29.5, 27.9, 27.69, 27.61, 26.7, 24.1, 22.8, 20.7, 20.5, 14.2.

IR ν 3436, 2954, 1470, 1386, 1103, 828 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H$^+$] Calcd $C_{25}H_{53}O_3Si^+$ 429.3764, found 429.3778 (3.3 ppm error).



di-*tert*-butyl(3-(3-isopentyloxiran-2-yl)propoxy)silanol

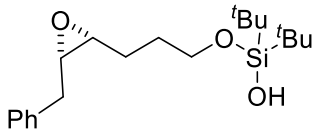
Compound 47: Synthesized using General Procedure B on a 1.46 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 434 mg, 1.31 mmol, 90% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.95 – 3.85 (m, 2H), 2.95 – 2.88 (m, 2H), 1.83 – 1.21 (m, 9H), 1.03 (s, 9H), 1.02 (s, 9H), 0.91 (d, J = 6.6 Hz, 6H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 62.6, 57.6, 57.5, 35.6, 30.3, 28.1, 27.68, 27.60, 25.8, 24.1, 22.64, 22.53, 20.7, 20.5.

IR ν 3472, 2960, 2857, 1470, 1386, 1012, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{18}H_{38}O_3SiNa^+$ 353.2488, found 353.2504 (4.5 ppm error).



(3-(3-benzyloxiran-2-yl)propoxy)di-*tert*-butylsilanol

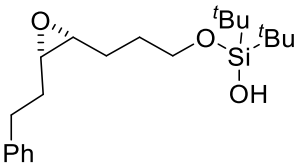
Compound 49: Synthesized using General Procedure B on a 1.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 274 mg, 0.782 mmol, 65% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.22 (m, 5H), 3.95 (ddd, J = 5.7, 4.5, 1.5 Hz, 2H), 3.23 (td, J = 6.3, 4.2 Hz, 1H), 3.07 (dt, J = 7.4, 4.3 Hz, 1H), 3.03 – 2.91 (m, 1H), 2.91 – 2.80 (m, 1H), 1.92 – 1.68 (m, 4H), 1.07 (s, 9H), 1.06 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 137.9, 128.9, 128.8, 126.7, 62.6, 57.7, 57.5, 34.4, 30.3, 27.68, 27.63, 24.4, 20.7, 20.6.

IR ν 3482, 2963, 2857, 1473, 1386, 1012, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{20}H_{34}O_3SiNa^+$ 373.2175, found 373.2158 (4.6 ppm error).



di-*tert*-butyl(3-(3-phenethyloxiran-2-yl)propoxy)silanol

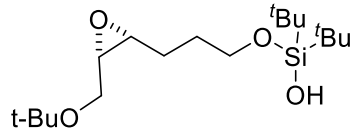
Compound 51: Synthesized using General Procedure B on a 1.47 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 455 mg, 1.25 mmol, 85% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.15 (m, 5H), 3.92 – 3.80 (m, 2H), 3.03 – 2.91 (m, 2H), 2.90 – 2.81 (m, 1H), 2.79 – 2.69 (m, 1H), 1.93 – 1.79 (m, 2H), 1.75 – 1.65 (m, 2H), 1.63 – 1.52 (m, 2H), 1.02 (s, 9H), 1.01 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 141.4, 128.59, 128.56, 126.2, 62.6, 57.8, 56.7, 33.0, 30.3, 29.9, 27.69, 27.62, 24.1, 20.8, 20.5.

IR ν 3499, 2965, 1471, 1386, 1012, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{21}H_{36}O_3SiNa^+$ 387.2331, found 387.2350 (4.9 ppm error).



(3-(3-(*tert*-butoxymethyl)oxiran-2-yl)propoxy)di-*tert*-butylsilanol

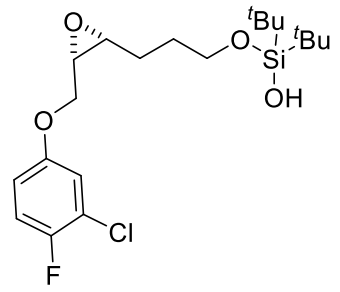
Compound 53: Synthesized using General Procedure B on a 1.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; (colorless oil, 298 mg, 0.860 mmol, 71% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.86 (t, J = 5.8 Hz, 2H), 3.55 – 3.38 (m, 2H), 3.09 (td, J = 5.5, 4.3 Hz, 1H), 3.00 (dt, J = 7.3, 4.5 Hz, 1H), 1.80 – 1.53 (m, 4H), 1.19 (s, 9H), 0.99 (s, 9H), 0.98 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 73.5, 62.6, 60.0, 57.0, 56.0, 30.2, 27.7, 27.6, 27.5, 24.3, 20.7, 20.5.

IR ν 3445, 2967, 2860, 1471, 1364, 1013, 828 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{18}H_{38}O_4SiNa^+$ 369.2437, found 369.2428 (2.4 ppm error).



di-*tert*-butyl(3-(3-((3-chloro-4-fluorophenoxy)methyl)oxiran-2-yl)propoxy)silanol

Compound 55: Synthesized using General Procedure B on a 1.4 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 469 mg, 1.12 mmol, 80% yield).

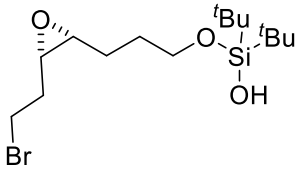
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 (t, J = 8.8 Hz, 1H), 6.94 (dd, J = 5.9, 3.1 Hz, 1H), 6.77 (dt, J = 9.0, 3.4 Hz, 1H), 4.13 (dd, J = 10.8, 4.2 Hz, 1H), 3.99 (dd, J = 10.8, 6.3 Hz, 1H), 3.87 (td, J = 5.2, 1.9 Hz, 2H), 3.31 (dt, J = 6.3, 4.3 Hz, 1H), 3.16 – 3.07 (m, 1H), 1.81 – 1.61 (m, 4H), 1.00 (s, 9H), 0.99 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 154.8 (d, *J* = 2.5 Hz), 153.1 (d, *J* = 241.8 Hz), 121.2 (d, *J* = 19.2 Hz), 116.8 (d, *J* = 22.8 Hz), 116.5, 114.4 (d, *J* = 6.6 Hz), 67.3, 62.5, 56.6, 54.4, 30.0, 27.6, 27.5, 24.6, 20.6, 20.5.

^{19}F NMR (377 MHz, $CDCl_3$) δ -126.4 (d, J = 5.3 Hz).

IR ν 2933, 2859, 1558, 1498, 1202, 1107, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{20}H_{32}FClO_4SiNa^+$ 441.1640, found 441.1644 (0.9 ppm error).



(3-(3-(2-bromoethyl)oxiran-2-yl)propoxy)di-*tert*-butylsilanol

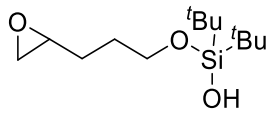
Compound 57: Synthesized using General Procedure B on a 1.4 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 360 mg, 0.980 mmol, 70% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.86 (dd, J = 6.9, 4.9 Hz, 2H), 3.60 – 3.43 (m, 2H), 3.14 – 2.97 (m, 2H), 2.20 – 1.99 (m, 2H), 1.77 – 1.66 (m, 3H), 1.65 – 1.54 (m, 1H), 1.00 (s, 9H), 0.99 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 62.6, 57.4, 55.6, 31.3, 30.1, 29.6, 27.6, 27.5, 24.4, 20.7, 20.5.

IR ν 3499, 2964, 2859, 1471, 1386, 1012, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{15}H_{31}BrO_3SiNa^+$ 389.1124, found 389.1120 (1.0 ppm error).



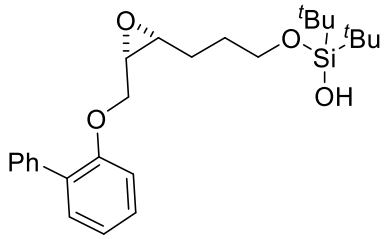
Compound 59: Synthesized using General Procedure B on a 2.66 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; (colorless oil, 545 mg, 2.09 mmol, 79% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.89 – 3.84 (m, 2H), 3.00 – 2.91 (m, 1H), 2.80 – 2.73 (m, 1H), 2.53 – 2.46 (m, 2H), 1.76 – 1.67 (m, 3H), 1.63 – 1.53 (m, 1H), 1.01 (s, 9H), 1.00 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 62.7, 52.7, 47.1, 29.6, 28.8, 27.7, 27.6, 20.7, 20.6.

IR ν 3474, 2933, 2857, 1473, 1104, 826, 648 cm^{-1}.

HRMS (ESI) m/z = [M + H$^+$] calculated for $C_{13}H_{29}O_3Si^+$ 261.1886, Found 261.1886 (error 0.0 ppm).



(3-(3-(([1,1'-biphenyl]-2-yloxy)methyl)oxiran-2-yl)propoxy)di-*tert*-butylsilanol

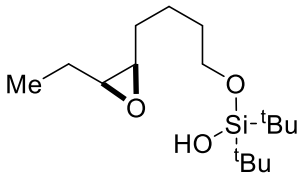
Compound 61: Synthesized using General Procedure B on a 1.4 mmol scale.; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; (colorless oil, 526 mg, 1.19 mmol, 85% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 – 7.47 (m, 2H), 7.39 – 7.23 (m, 5H), 7.07 – 6.90 (m, 2H), 4.06 (d, J = 5.3 Hz, 2H), 3.80 (td, J = 5.9, 4.1 Hz, 2H), 3.22 (td, J = 5.2, 4.3 Hz, 1H), 3.00 (ddd, J = 6.9, 5.1, 4.2 Hz, 1H), 1.76 – 1.50 (m, 4H), 0.98 (s, 9H), 0.97 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 155.5, 138.4, 131.5, 131.1, 129.7, 128.7, 128.1, 127.0, 121.8, 113.4, 67.2, 62.5, 56.7, 54.7, 30.0, 27.63, 27.59, 24.5, 20.67, 20.64.

IR ν 3486, 2964, 1505, 1473, 1230, 1009, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H$^+$] Calcd $C_{26}H_{39}O_4Si^+$ 443.2618, found 443.2606 (2.7 ppm error).



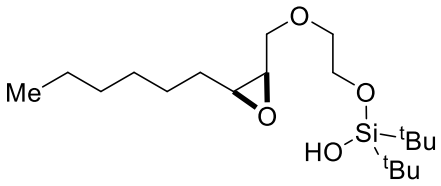
Compound 63: Synthesized using General Procedure B on a 4.35 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 1.21 g, 4.0 mmol, 92% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.81 (t, *J* = 6.0 Hz, 2H), 3.00 – 2.83 (m, 2H), 2.19 (s, 1H), 1.65 – 1.57 (m, 3H), 1.56 – 1.46 (m, 5H), 1.05 – 0.99 (m, 21H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 63.1, 58.7, 57.6, 32.7, 27.63, 27.62, 27.4, 22.9, 21.2, 20.64, 20.59, 10.7.

IR ν 3480, 2967, 2933, 2859, 1471, 1103, 827, 647 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calcd for $C_{16}H_{34}O_3SiNa^+$ 325.2175, found 325.2179 (1.2 ppm error)



di-*tert*-butyl(2-(((2*R**,3*S**)-3-hexyloxiran-2-yl)methoxy)ethoxy)silanol

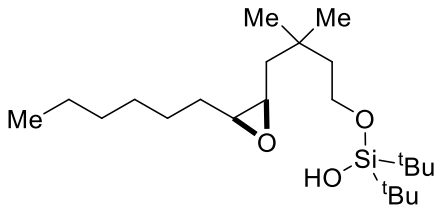
Compound 65: Synthesized using General Procedure B on a 2.8 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 650 mg, 1.8 mmol, 64% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.04 – 3.92 (m, 2H), 3.75 (dd, *J* = 11.1, 4.0 Hz, 1H), 3.67 – 3.55 (m, 2H), 3.52 – 3.46 (m, 1H), 3.30 (s, 1H), 3.14 (dt, *J* = 6.7, 4.2 Hz, 1H), 2.98 (td, *J* = 6.0, 4.3 Hz, 1H), 1.57 – 1.46 (m, 3H), 1.46 – 1.38 (m, 1H), 1.38 – 1.24 (m, 6H), 1.02 (s, 18H), 0.92 – 0.85 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 73.5, 69.4, 63.0, 56.4, 55.0, 31.8, 29.2, 28.2, 27.5, 26.8, 22.7, 20.69, 20.66, 14.2.

IR ν 3480, 2931, 2857, 1473, 1457, 1103, 828, 648 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{19}H_{40}O_4SiNa^+$ 383.2594, Found 383.2600 (1.6 ppm error).



di-*tert*-butyl(4-((2*R**,3*S**)-3-hexyloxiran-2-yl)-3,3-dimethylbutoxy)silanol

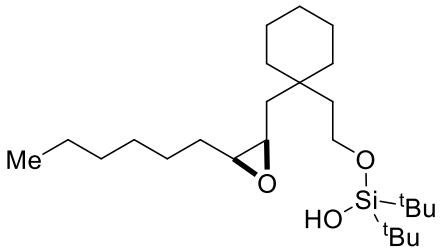
Compound 67: Synthesized using General Procedure B on a 0.6 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 216.5 mg, 0.56 mmol, 93% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.94 – 3.76 (m, 2H), 3.04 (ddd, *J* = 8.0, 4.4, 3.2 Hz, 1H), 2.91 (td, *J* = 5.9, 4.4 Hz, 1H), 2.78 (s, 1H), 1.71 – 1.58 (m, 2H), 1.57 – 1.39 (m, 5H), 1.39 – 1.26 (m, 7H), 1.03 (s, 3H), 1.01 (s, 9H), 1.00 (s, 9H), 0.98 (s, 3H), 0.92 – 0.86 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 60.3, 56.7, 54.4, 44.3, 39.4, 32.4, 31.9, 29.4, 28.5, 28.3, 28.1, 27.7, 27.6, 26.7, 22.7, 20.8, 20.3, 14.2.

IR ν 3480, 2960, 2931, 2857, 1471, 1096, 827, 647 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{22}H_{46}O_3SiNa^+$ 409.3114, Found 409.3116 (0.5 ppm error).



di-*tert*-butyl(2-(1-(((2*R**,3*S**)-3-hexyloxiran-2-yl)methyl)cyclohexyl)ethoxy)silanol

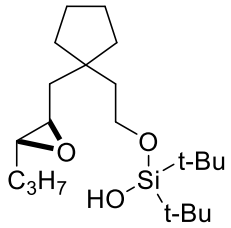
Compound 69: Synthesized using General Procedure B on a 1.7 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 662 mg, 1.55 mmol, 91% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.89 (td, *J* = 10.5, 4.7 Hz, 1H), 3.76 (td, *J* = 10.3, 6.2 Hz, 1H), 3.46 (s, 1H), 3.06 (ddd, *J* = 9.2, 4.5, 2.2 Hz, 1H), 2.98 – 2.87 (m, 1H), 1.88 – 1.77 (m, 1H), 1.69 – 1.61 (m, 2H), 1.55 – 1.42 (m, 9H), 1.41 – 1.21 (m, 12H), 1.01 (s, 9H), 1.01 (s, 9H), 0.92 – 0.86 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 59.6, 56.7, 54.1, 39.5, 36.8, 36.7, 36.2, 34.7, 31.9, 29.3, 28.1, 27.7, 27.5, 26.7, 26.5, 22.7, 21.7, 21.0, 20.2, 14.2.

IR ν 3496, 2930, 2856, 1471, 1092, 827, 647 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{25}H_{50}O_3SiNa$ 449.3427, Found 449.3444 (3.8 ppm error).



di-*tert*-butyl(2-(1-(3-propyloxiran-2-yl)methyl)cyclopentyl)ethoxy)silanol

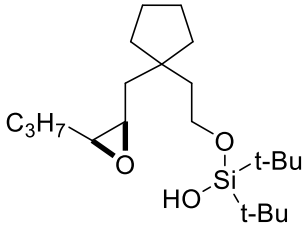
Compound 71: Synthesized using General Procedure B on a 1.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; (colorless oil, 351 mg, 0.947 mmol, 79% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.86 (td, J = 10.1, 5.2 Hz, 1H), 3.72 (td, J = 9.9, 6.4 Hz, 1H), 2.72 (ddd, J = 7.6, 3.7, 2.4 Hz, 1H), 2.64 (td, J = 5.4, 2.5 Hz, 1H), 1.80 – 1.64 (m, 2H), 1.63 – 1.53 (m, 5H), 1.50 – 1.34 (m, 9H), 0.97 (s, 18H), 0.91 (t, J = 7.2 Hz, 3H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 60.6, 58.4, 57.1, 43.7, 41.6, 40.7, 38.4, 38.0, 34.2, 27.64, 27.55, 24.4, 24.2, 20.8, 20.3, 19.4, 14.1.

IR ν 3544, 2941, 2856, 1471, 1386, 1097, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{21}H_{42}O_3SiNa^+$ 393.2801, found 393.2809 (2.0 ppm error).



di-*tert*-butyl(2-(1-(3-propyloxiran-2-yl)methyl)cyclopentyl)ethoxy)silanol

Compound 73: Synthesized using General Procedure B on a 1.3 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; (colorless oil, 458 mg, 1.24 mmol, 95% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.95 (td, J = 10.4, 4.9 Hz, 1H), 3.77 (td, J = 10.3, 6.1 Hz, 1H), 3.05 (ddd, J = 8.8, 4.5, 2.4 Hz, 1H), 2.95 (td, J = 5.8, 4.4 Hz, 1H), 1.89 – 1.77 (m, 1H), 1.75 – 1.60 (m, 6H), 1.58 – 1.47 (m, 7H), 1.46 – 1.37 (m, 2H), 1.08 – 0.93 (m, 21H).

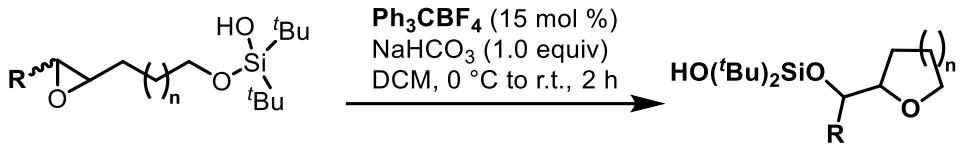
^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 60.6, 56.5, 55.3, 43.7, 41.7, 38.6, 38.2, 36.2, 30.1, 27.7, 27.5, 24.4, 24.1, 21.0, 20.1, 20.0, 14.2.

IR ν 3493, 2941, 2861, 1471, 1386, 1095, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H^+] Calcd $C_{21}H_{43}O_3Si^+$ 371.2981, found 371.2996 (4.0 ppm error).

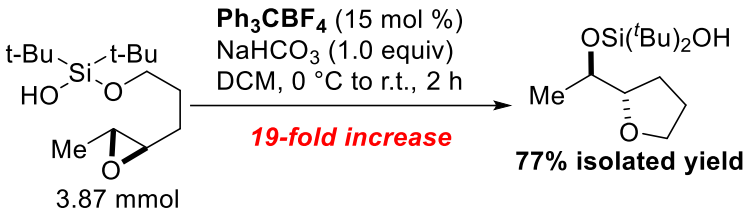
VII. Rearrangement Reaction Procedures

General Procedure C



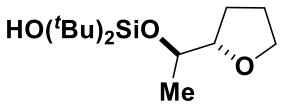
An oven-dried microwave vial equipped with a stir bar was cooled under nitrogen and charged with silanol epoxide (0.2 mmol), $NaHCO_3$ (1.0 equiv, 0.2 mmol, 16.8 mg) and CH_2Cl_2 (1.0 mL). The resulting mixture was cooled to 0 °C using an ice-water bath and stirred at this temperature for 10 minutes. Ph_3CBF_4 (0.15 equiv, 0.03 mmol, 10.0 mg) was added to the heterogeneous mixture in one portion, and the sides of the reaction vial were rinsed with an additional 1 mL of CH_2Cl_2 (Final concentration: 0.1 M). The reaction mixture was allowed to warm to room temperature over a period of two hours. Next, the reaction mixture was diluted with CH_2Cl_2, and transferred to a separatory funnel. The organic layer was washed with saturated aqueous $NaHCO_3$, collected, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The resulting residue was purified by chromatography on silica gel (specific conditions are associated with each product).

Scale-up Procedure



An oven-dried 100 mL round bottom flask equipped with a stir bar was cooled under nitrogen and charged with di-tert-butyl(3-(3-methyloxiran-2-yl)propoxy)silanol (1.0 equiv, 1.062 g, 3.87 mmol), $NaHCO_3$ (1.0 equiv, 3.87 mmol, 325.0 mg) and CH_2Cl_2 (0.1 M, 40.0 mL). The resulting mixture was cooled to 0 °C using an ice-water bath and stirred at this temperature for 10 minutes. Then, Ph_3CBF_4 (0.15 equiv, 0.58 mmol, 192.0 mg) was added to the reaction mixture in one portion. The reaction mixture was removed from the ice-water bath and allowed to warm to room temperature over a period of two hours. Next, the reaction mixture was diluted with CH_2Cl_2, and transferred to a separatory funnel. The organic layer was washed with saturated aqueous $NaHCO_3$ solution, collected, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The resulting residue was purified by chromatography on silica gel using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 as eluent. The desired product was obtained as a colorless oil in a 77% yield (818.2 mg, 2.98 mmol).

VIII. Characterization of Rearrangement Products



di-*tert*-butyl((*R**)-1-((*S**)-tetrahydrofuran-2-yl)ethoxy)silanol

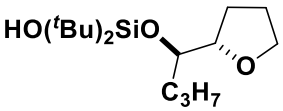
Compound 2: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 42.8 mg, 0.156 mmol, 78% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.40 (qd, *J* = 6.5, 2.5 Hz, 1H), 3.97 – 3.89 (m, 1H), 3.78 – 3.70 (m, 1H), 3.65 (ddd, *J* = 8.6, 6.0, 2.5 Hz, 1H), 1.98 – 1.84 (m, 3H), 1.82 – 1.70 (m, 1H), 1.15 (d, *J* = 6.5 Hz, 3H), 1.03 (s, 9H), 0.99 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 84.1, 68.1, 67.7, 27.9, 27.7, 25.2, 23.2, 21.2, 20.5, 20.3.

IR ν 3436, 2963, 2860, 1475, 650 cm^{-1}.

HRMS (ESI) m/z = [M + Na$^+$] calculated for $C_{14}H_{30}O_3SiNa^+$ 297.1862, Found 297.1881 (6.4 ppm error).



di-*tert*-butyl((*R**)-1-((*S**)-tetrahydrofuran-2-yl)butoxy)silanol

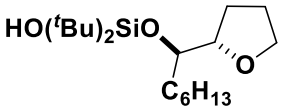
Compound 4: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 46.6 mg, 0.154 mmol, 77% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.32 (td, *J* = 6.4, 2.2 Hz, 1H), 4.14 (s, 1H), 4.01 – 3.91 (m, 1H), 3.76 – 3.67 (m, 2H), 1.99 – 1.82 (m, 3H), 1.77 – 1.67 (m, 1H), 1.54 – 1.30 (m, 4H), 1.04 (s, 9H), 0.99 (s, 9H), 0.93 (t, *J* = 6.9 Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 83.0, 71.4, 67.9, 37.6, 28.0, 27.8, 25.0, 23.0, 21.5, 20.6, 19.5, 14.6.

IR ν 3425, 2963, 2934, 2859, 1473, 1089, 827, 647 cm^{-1}.

HRMS (ESI) m/z = [M + Na$^+$] calculated for $C_{16}H_{34}O_3SiNa^+$ 325.2175, Found 325.2197 (6.8 ppm error).



di-*tert*-butyl(((*R**)-1-((*S**)-tetrahydrofuran-2-yl)heptyl)oxy)silanol

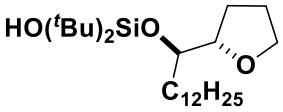
Compound 6: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 53.6 mg, 0.156 mmol, 78% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.30 (td, *J* = 6.5, 2.3 Hz, 1H), 4.13 (s, 1H), 4.01 – 3.91 (m, 1H), 3.77 – 3.63 (m, 2H), 1.99 – 1.82 (m, 3H), 1.78 – 1.66 (m, 1H), 1.57 – 1.37 (m, 3H), 1.33 – 1.22 (m, 7H), 1.04 (s, 9H), 0.99 (s, 9H), 0.91 – 0.85 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 82.9, 71.6, 67.9, 35.3, 31.9, 29.7, 28.0, 27.8, 26.1, 25.0, 23.0, 22.7, 21.5, 20.6, 14.2.

IR ν 3409, 2933, 2857, 1473, 1100, 827, 648 cm^{-1}.

HRMS (ESI) m/z = [M + Na$^+$] calculated for $C_{19}H_{40}O_3SiNa^+$ 367.2644, Found 367.2672 (7.6 ppm error).



di-*tert*-butyl(((*R**)-1-((*S**)-tetrahydrofuran-2-yl)tridecyl)oxy)silanol

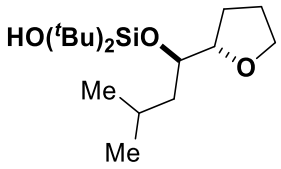
Compound 8: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 68.6 mg, 0.160 mmol, 80% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.30 (td, *J* = 6.4, 2.3 Hz, 1H), 4.13 (s, 1H), 4.00 – 3.91 (m, 1H), 3.76 – 3.67 (m, 2H), 1.99 – 1.82 (m, 3H), 1.79 – 1.66 (m, 1H), 1.56 – 1.38 (m, 3H), 1.26 (s, 19H), 1.04 (s, 9H), 0.99 (s, 9H), 0.91 – 0.85 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 82.9, 71.6, 67.9, 35.3, 32.1, 30.0, 29.82, 29.78, 29.71, 29.67, 29.5, 28.0, 27.8, 26.2, 25.1, 23.0, 22.8, 21.5, 20.6, 14.3.

IR ν 3442, 2927, 2856, 1473, 1105, 875, 827, 648 cm^{-1}.

HRMS (ESI) m/z = [M + Na$^+$] calculated for $C_{25}H_{52}O_3SiNa^+$ 451.3583, Found 451.3609 (5.8 ppm error).



di-*tert*-butyl((*R**)-3-methyl-1-((*S**)-tetrahydrofuran-2-yl)butoxy)silanol

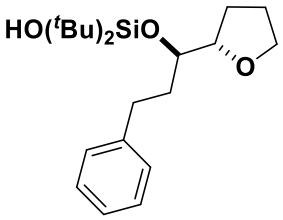
Compound 10: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 45.0 mg, 0.142 mmol, 71% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.44 – 4.33 (m, 1H), 4.13 (s, 1H), 4.01 – 3.91 (m, 1H), 3.77 – 3.65 (m, 2H), 1.99 – 1.84 (m, 3H), 1.76 – 1.60 (m, 2H), 1.42 (ddd, *J* = 14.1, 8.1, 6.3 Hz, 1H), 1.32 (ddd, *J* = 13.9, 7.7, 6.2 Hz, 1H), 1.04 (s, 9H), 0.99 (s, 9H), 0.92 (d, *J* = 5.0 Hz, 3H), 0.91 (d, *J* = 4.9 Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 82.7, 69.7, 68.0, 44.7, 28.0, 27.8, 25.0, 24.8, 23.7, 22.81, 22.77, 21.4, 20.6.

IR ν 3437, 2958, 2933, 2857, 1471, 1090, 827, 648 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] calculated for $C_{17}H_{36}O_3SiNa^+$ 339.2331, found 339.2331 (0.0 ppm error).



di-*tert*-butyl((*R**)-3-phenyl-1-((*S**)-tetrahydrofuran-2-yl)propoxy)silanol

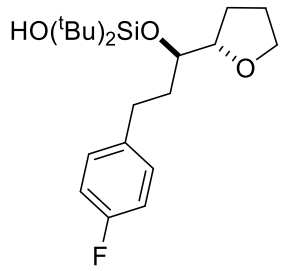
Compound 12: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; (colorless oil, 51.8 mg, 0.142 mmol, 71% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.26 (m, 2H), 7.23 – 7.15 (m, 3H), 4.40 (td, *J* = 6.5, 2.3 Hz, 1H), 4.03 – 3.95 (m, 1H), 3.83 – 3.70 (m, 2H), 2.81 (ddd, *J* = 13.6, 10.7, 6.3 Hz, 1H), 2.66 (ddd, *J* = 13.6, 10.7, 6.0 Hz, 1H), 2.01 – 1.86 (m, 3H), 1.86 – 1.71 (m, 3H), 1.05 (s, 9H), 1.03 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 142.2, 128.6, 128.4, 126.1, 82.9, 71.4, 68.0, 37.2, 32.7, 28.0, 27.8, 25.1, 23.3, 21.5, 20.6.

IR ν 3409, 2933, 2857, 1473, 1072, 827, 648 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{21}H_{36}O_3SiNa^+$ 387.2331, found 387.2330 (0.3 ppm error)



di-*tert*-butyl(3-(4-fluorophenyl)-1-tetrahydrofuran-2-yl)propoxy)silanol

Compound 14: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3 % acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 56 mg, 0.146 mmol, 73% yield).

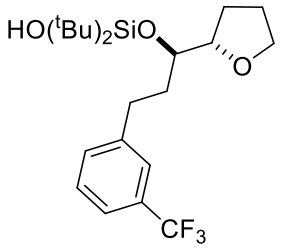
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 – 7.09 (m, 2H), 7.02 – 6.91 (m, 2H), 4.37 (td, J = 6.4, 2.3 Hz, 1H), 4.03 – 3.92 (m, 1H), 3.75 (dqd, J = 14.4, 6.1, 2.6 Hz, 2H), 2.77 (ddd, J = 13.7, 10.4, 6.5 Hz, 1H), 2.63 (ddd, J = 13.8, 10.3, 6.3 Hz, 1H), 1.99 – 1.88 (m, 3H), 1.91 – 1.68 (m, 3H), 1.04 (s, 9H), 1.02 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 161.4 (d, J = 243.5 Hz), 137.8 (d, J = 3.2 Hz), 129.7 (d, J = 7.5 Hz), 115.3 (d, J = 21.3 Hz), 82.8, 71.3, 68.0, 37.3, 31.8, 28.0, 27.8, 25.0, 23.3, 21.5, 20.6.

^{19}F NMR (377 MHz, $CDCl_3$) δ -118.2 – -118.4 (m).

IR ν 3443, 2965, 2859, 1510, 1110, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{21}H_{35}FO_3SiNa^+$ 405.2237, found 405.2240 (0.7 ppm error).



di-*tert*-butyl(1-tetrahydrofuran-2-yl)-3-(3-(trifluoromethyl)phenyl)propoxy)silanol

Compound 16: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; (colorless oil, 58 mg, 0.134 mmol, 67% yield).

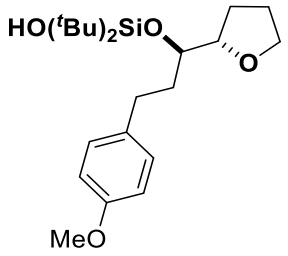
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.33 (m, 4H), 4.39 (td, J = 6.4, 2.4 Hz, 1H), 4.04 – 3.93 (m, 1H), 3.76 (ddtd, J = 12.2, 8.4, 6.1, 2.5 Hz, 2H), 2.87 (ddd, J = 13.7, 10.3, 6.6 Hz, 1H), 2.73 (ddd, J = 13.7, 10.2, 6.3 Hz, 1H), 2.06 – 1.88 (m, 3H), 1.88 – 1.68 (m, 3H), 1.05 (s, 9H), 1.02 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 143.1, 131.8, 130.9 (q, *J* = 32.3 Hz), 128.9, 125.7 (q, *J* = 273 Hz), 125.0 (q, *J* = 3.7 Hz), 122.9 (q, *J* = 3.7 Hz), 82.8, 71.3, 68.0, 36.8, 32.4, 28.0, 27.8, 25.0, 23.4, 21.4, 20.6.

^{19}F NMR (377 MHz, $CDCl_3$) δ -63.2.

IR ν 3400, 2936, 2891, 1471, 1163, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{22}H_{35}F_3O_3SiNa^+$ 455.2205, found 455.2217 (2.6 ppm error).



di-*tert*-butyl((*R**)-3-(4-methoxyphenyl)-1-((*S**)-tetrahydrofuran-2-yl)propoxy)silanol

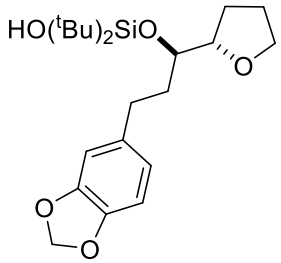
Compound 18: Synthesized using General Procedure C on a 0.2 mmol scale.; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 56.8 mg, 0.144 mmol, 72% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.11 (d, *J* = 8.7 Hz, 2H), 6.83 (d, *J* = 8.7 Hz, 2H), 4.38 (td, *J* = 6.5, 2.2 Hz, 1H), 4.02 – 3.94 (m, 1H), 3.84 – 3.66 (m, 5H), 2.74 (ddd, *J* = 13.7, 10.7, 6.2 Hz, 1H), 2.60 (ddd, *J* = 13.7, 10.7, 5.9 Hz, 1H), 2.03 – 1.85 (m, 3H), 1.85 – 1.69 (m, 3H), 1.05 (s, 9H), 1.02 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 158.0, 134.3, 129.3, 114.0, 82.9, 71.4, 68.0, 55.4, 37.4, 31.7, 28.0, 27.8, 25.1, 23.3, 21.5, 20.6.

IR ν 3411, 2933, 2857, 1512, 1473, 1246, 1070, 1035, 827, 648 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{22}H_{38}O_4SiNa$ 417.2437, Found 417.2458 (5.0 ppm error).



(3-(benzo[*d*][1,3]dioxol-5-yl)-1-tetrahydrofuran-2-yl)propoxy)di-*tert*-butylsilanol

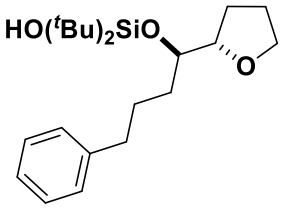
Compound 20: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 59 mg, 0.144 mmol, 72% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.77 – 6.57 (m, 3H), 5.91 (s, 2H), 4.36 (td, J = 6.4, 2.3 Hz, 1H), 4.03 – 3.92 (m, 1H), 3.74 (dddd, J = 14.5, 8.5, 4.8, 1.9 Hz, 2H), 2.71 (ddd, J = 13.7, 10.6, 6.3 Hz, 1H), 2.58 (ddd, J = 13.7, 10.6, 6.0 Hz, 1H), 1.93 (dddt, J = 12.8, 10.9, 7.9, 6.2 Hz, 3H), 1.81 – 1.56 (m, 3H), 1.04 (s, 9H), 1.01 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 147.8, 145.8, 136.0, 121.1, 108.9, 108.3, 100.9, 82.8, 71.3, 68.0, 37.5, 32.4, 28.01, 27.80, 25.1, 23.3, 21.5, 20.6.

IR ν 3566, 2960, 2859, 1504, 1244, 1038, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{22}H_{36}O_5SiNa^+$ 431.2230, found 431.2245 (3.5 ppm error).



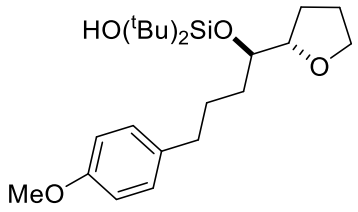
Compound 22: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; (colorless oil, 60.6 mg, 0.160 mmol, 80% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.30 (m, 2H), 7.26 – 7.18 (m, 3H), 4.39 (td, *J* = 6.5, 2.2 Hz, 1H), 4.05 – 3.97 (m, 1H), 3.82 – 3.72 (m, 2H), 2.78 – 2.60 (m, 2H), 2.04 – 1.81 (m, 4H), 1.79 – 1.68 (m, 2H), 1.67 – 1.49 (m, 2H), 1.09 (s, 9H), 1.05 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 142.2, 128.5, 128.4, 125.9, 82.9, 71.5, 67.9, 36.1, 34.8, 28.0, 27.8, 27.6, 25.0, 23.1, 21.5, 20.6.

IR ν 3410, 2933, 2857, 1473, 1075, 827, 647 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{22}H_{38}O_3SiNa^+$ 401.2488, Found 401.2508 (5.0 ppm error).



di-*tert*-butyl((4-methoxyphenyl)-1-tetrahydrofuran-2-yl)butoxy)silanol

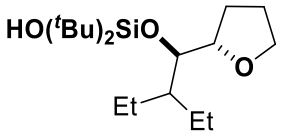
Compound 24: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 66 mg, 0.162 mmol, 81% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 – 7.04 (m, 2H), 6.87 – 6.78 (m, 2H), 4.33 (td, J = 6.4, 2.3 Hz, 1H), 4.01 – 3.91 (m, 1H), 3.79 (s, 3H), 3.76 – 3.65 (m, 2H), 2.66 – 2.49 (m, 2H), 1.98 – 1.82 (m, 3H), 1.77 – 1.35 (m, 5H), 1.04 (s, 9H), 1.00 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 157.9, 134.3, 129.3, 113.9, 82.9, 71.5, 67.9, 55.4, 35.2, 34.7, 28.0, 27.84, 27.80, 25.0, 23.1, 21.5, 20.6.

IR ν 3524, 2934, 2857, 1512, 1246, 1038, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H^+] Calcd $C_{23}H_{41}O_4Si^+$ 409.2774, found 409.2787 (3.2 ppm error).



di-*tert*-butyl((*R**)-2-ethyl-1-((*S**)-tetrahydrofuran-2-yl)butoxy)silanol

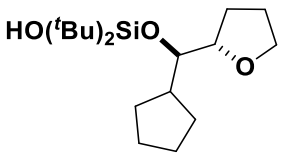
Compound 26: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 29.1 mg, 0.088 mmol, 44% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.43 – 4.34 (m, 1H), 4.25 (s, 1H), 3.98 – 3.89 (m, 1H), 3.82 – 3.72 (m, 1H), 3.70 – 3.62 (m, 1H), 2.05 – 1.83 (m, 3H), 1.74 – 1.60 (m, 2H), 1.48 – 1.33 (m, 3H), 1.05 (s, 10H), 1.01 (s, 9H), 0.95 – 0.88 (m, 6H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 81.4, 72.8, 67.1, 46.5, 28.1, 27.9, 25.5, 24.0, 23.2, 22.5, 21.6, 20.7, 12.6, 12.3.

IR ν 3429, 2950, 2859, 1473, 1102, 827, 647 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{18}H_{38}O_3SiNa^+$ 353.2488, Found 353.2496 (2.3 ppm error).



di-*tert*-butyl((*R**)-cyclopentyl((*S**)-tetrahydrofuran-2-yl)methoxy)silanol

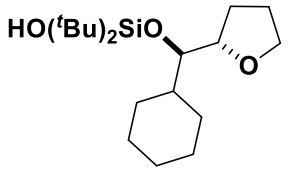
Compound 28: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 26.2 mg, 0.080 mmol, 40% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.48 (s, 1H), 4.21 (dd, *J* = 8.1, 2.0 Hz, 1H), 4.03 – 3.95 (m, 1H), 3.76 (ddd, *J* = 9.4, 6.7, 2.1 Hz, 1H), 3.69 (td, *J* = 8.5, 6.6 Hz, 1H), 2.01 – 1.71 (m, 6H), 1.65 – 1.44 (m, 6H), 1.27 – 1.21 (m, 1H), 1.06 (s, 9H), 1.00 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 83.1, 76.1, 67.8, 45.4, 30.6, 29.4, 28.2, 27.9, 25.4, 25.1, 24.9, 23.1, 21.9, 20.7.

IR ν 3409, 2947, 2859, 1473, 1115, 827, 647 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{18}H_{36}O_3SiNa^+$ 351.2331, found 351.2335 (1.1 ppm error).



di-*tert*-butyl((*R**)-cyclohexyl((*S**)-tetrahydrofuran-2-yl)methoxy)silanol

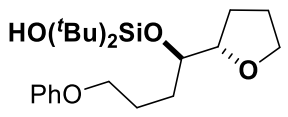
Compound 30: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 34.1 mg, 0.1 mmol, 50% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.36 (s, 1H), 4.14 (dd, *J* = 7.2, 2.0 Hz, 1H), 4.01 – 3.93 (m, 1H), 3.82 (ddd, *J* = 9.2, 6.8, 2.0 Hz, 1H), 3.65 (td, *J* = 8.4, 6.7 Hz, 1H), 2.01 – 1.84 (m, 4H), 1.80 – 1.64 (m, 5H), 1.46 – 1.35 (m, 1H), 1.28 – 1.06 (m, 4H), 1.05 (s, 9H), 1.00 (s, 10H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 81.9, 75.9, 67.5, 43.2, 30.2, 29.8, 28.1, 27.9, 26.71, 26.69, 25.2, 23.4, 21.8, 20.7.

IR ν 3445, 2924, 2851, 1448, 1060, 891 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{19}H_{38}O_3SiNa^+$ 365.2488, found 365.2492 (1.1 ppm error).



di-*tert*-butyl((*R**)-4-phenoxy-1-((*S**)-tetrahydrofuran-2-yl)butoxy)silanol

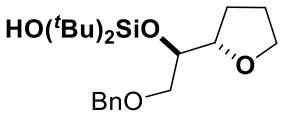
Compound 32: Synthesized using General Procedure C on a 0.2 mmol scale.; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; (colorless oil, 40.0 mg, 0.101 mmol, 50% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.25 (m, 2H), 6.94 (tt, *J* = 7.4, 1.1 Hz, 1H), 6.91 – 6.86 (m, 2H), 4.39 (td, *J* = 6.4, 2.5 Hz, 1H), 4.11 (s, 1H), 4.04 – 3.91 (m, 3H), 3.79 – 3.70 (m, 2H), 2.00 – 1.83 (m, 5H), 1.83 – 1.74 (m, 1H), 1.72 – 1.64 (m, 2H), 1.05 (s, 9H), 1.01 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.1, 129.6, 120.8, 114.6, 82.9, 71.6, 68.01, 67.96, 31.7, 28.0, 27.8, 25.9, 25.1, 23.5, 21.5, 20.6.

IR ν 3410, 2933, 2857, 1497, 1473, 1246, 1079, 827, 752, 647 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{22}H_{38}O_4SiNa^+$ 417.2437, Found 417.2446 (2.2 ppm error).



((*R**)-2-(benzyloxy)-1-((*S**)-tetrahydrofuran-2-yl)ethoxy)di-*tert*-butylsilanol

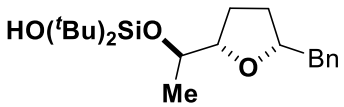
Compound 34: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 65.4 mg, 0.172 mmol, 86% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.18 (m, 5H), 4.52 – 4.42 (m, 2H), 4.25 (dt, *J* = 7.8, 4.0 Hz, 1H), 4.03 (s, 1H), 3.84 – 3.77 (m, 1H), 3.76 – 3.70 (m, 1H), 3.70 – 3.63 (m, 1H), 3.51 (dd, *J* = 9.8, 3.7 Hz, 1H), 3.37 (dd, *J* = 9.8, 7.6 Hz, 1H), 1.92 – 1.72 (m, 4H), 0.95 (s, 9H), 0.93 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 137.4, 128.7, 128.1, 80.5, 73.9, 73.6, 72.9, 68.3, 27.7, 27.6, 25.9, 25.7, 21.0, 20.8.

IR ν 3456, 2933, 2857, 1473, 1072, 871, 827, 748, 698, 648 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{21}H_{36}O_4SiNa^+$ 403.2281, Found 403.2277 (1.0 ppm error).



((*R**)-1-((2*S**,5*R**)-5-benzyltetrahydrofuran-2-yl)ethoxy)di-*tert*-butylsilanol

Compound 36: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 43.6 mg, 0.120 mmol, 60% yield).

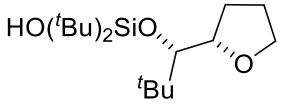
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34 – 7.26 (m, 2H), 7.25 – 7.17 (m, 3H), 4.41 (qd, *J* = 6.5, 2.4 Hz, 1H), 3.99 (dq, *J* = 9.2, 6.6 Hz, 1H), 3.67 (ddd, *J* = 8.7, 7.2, 2.3 Hz, 1H), 3.08 (dd, *J* = 13.5, 6.4 Hz, 1H), 2.78 (dd, *J* = 13.5, 7.1 Hz, 1H), 2.08 – 1.98 (m, 1H), 1.94 – 1.85 (m, 1H), 1.76 – 1.67 (m, 1H), 1.63 – 1.54 (m, 1H), 1.15 (d, *J* = 6.5 Hz, 3H), 1.06 (s, 9H), 1.06 (s, 9H).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 138.7, 129.1, 128.6, 126.5, 83.9, 81.0, 67.8, 41.5, 30.6, 27.93, 27.90, 22.7, 21.2, 20.5, 20.3.

IR ν 3460, 2933, 2857, 1473, 1075, 827, 700, 648 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{21}H_{36}O_3SiNa^+$ 387.2331, Found 387.2349 (error 4.6 ppm).

Compound 38: Previously characterized in *Org. Lett.* **2022**, *24*, 939–943.



di-*tert*-butyl((*S**)-2,2-dimethyl-1-((*S**)-tetrahydrofuran-2-yl)propoxy)silanol

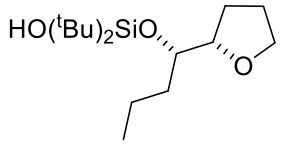
Compound 40: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; (colorless oil, 38.0 mg, 0.120 mmol, 60% yield).

^{1}H NMR (400 MHz, $CDCl_3$) ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.90 (s, 1H), 3.84 – 3.64 (m, 3H), 3.60 (d, *J* = 8.4 Hz, 1H), 2.06 – 1.85 (m, 3H), 1.59 – 1.44 (m, 1H), 1.04 (s, 9H), 1.01 (s, 9H), 0.98 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 84.2, 82.7, 66.1, 35.8, 30.1, 28.2, 27.8, 27.4, 26.7, 21.9, 20.8.

IR ν 3440, 2964, 2933, 2857, 1473, 1115, 879, 827, 647 cm^{-1}.

HRMS (ESI) m/z = [M + H^+] calculated for $C_{17}H_{37}O_3Si^+$ 317.2512, Found 317.2505 (2.2 ppm error).



di-*tert*-butyl(1-(tetrahydrofuran-2-yl)butoxy)silanol

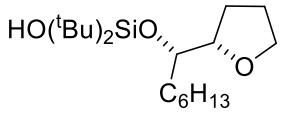
Compound 42: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 45 mg, 0.149 mmol, 74% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.89 – 3.74 (m, 3H), 3.66 (dd, J = 8.3, 5.7 Hz, 1H), 1.98 – 1.78 (m, 3H), 1.60 – 1.30 (m, 5H), 1.02 (s, 9H), 0.98 (s, 9H), 0.91 (t, J = 7.2 Hz, 3H).

$^{13}C\{^{1}H\}$ (101 MHz, $CDCl_3$) δ 84.4, 76.4, 67.7, 36.9, 28.5, 27.9, 27.5, 26.0, 21.4, 20.4, 18.3, 14.7.

IR ν 3453, 2958, 2886, 1386, 1013, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{16}H_{34}O_3SiNa^+$ 325.2175, found 325.2164 (3.4 ppm error).



di-*tert*-butyl((1-tetrahydrofuran-2-yl)heptyl)oxy)silanol

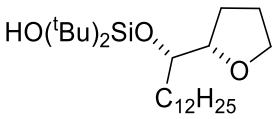
Compound 44: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 60 mg, 0.174 mmol, 87% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.87 – 3.75 (m, 3H), 3.67 (td, J = 8.7, 5.5 Hz, 1H), 1.96 – 1.82 (m, 3H), 1.53 – 1.32 (m, 5H), 1.30 – 1.20 (m, 6H) 1.02 (s, 9H), 0.98 (s, 9H), 0.91 – 0.84 (m, 3H).

$^{13}C\{^{1}H\}$ (101 MHz, $CDCl_3$) δ 84.3, 76.5, 67.7, 34.5, 31.9, 29.8, 28.5, 27.8, 27.5, 26.0, 24.8, 22.7, 21.4, 20.4, 14.2.

IR ν 3459, 2954, 2860, 1471, 1012, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{19}H_{40}O_3SiNa^+$ 367.2644, found 367.2628 (4.4 ppm error).



di-*tert*-butyl((1-tetrahydrofuran-2-yl)tridecyl)oxy)silanol

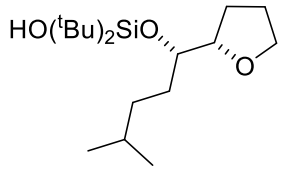
Compound 46: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 68.6 mg, 0.160 mmol, 80% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.88 – 3.74 (m, 3H), 3.67 (td, J = 8.6, 5.4 Hz, 1H), 1.98 – 1.78 (m, 3H), 1.48 – 1.35 (m, 5H), 1.31 – 1.18 (m, 18H), 1.01 (s, 9H), 0.98 (s, 9H), 0.91 – 0.82 (m, 3H).

$^{13}C\{^{1}H\}$ (101 MHz, $CDCl_3$) δ 84.4, 76.5, 67.7, 34.5, 32.1, 30.1, 29.83, 29.79, 29.72, 29.66, 29.5, 28.5, 27.9, 27.5, 26.0, 24.9, 22.8, 21.4, 20.4, 14.2.

IR ν 3445, 2941, 1471, 1386, 1013, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H^+] Calcd $C_{25}H_{53}O_3Si^+$ 429.3764, found 429.3770 (1.4 ppm error).



di-*tert*-butyl((4-methyl-1-(tetrahydrofuran-2-yl)pentyl)oxy)silanol

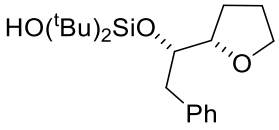
Compound 48: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2; single diastereomer; (colorless oil, 53 mg, 0.160 mmol, 80% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.89 – 3.74 (m, 3H), 3.70 – 3.64 (m, 1H), 1.98 – 1.78 (m, 3H), 1.58 – 1.29 (m, 6H), 1.02 (s, 9H), 0.99 (s, 9H), 0.88 (d, *J* = 3.6 Hz, 3H), 0.87 (d, *J* = 3.7 Hz, 3H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 84.3, 76.5, 67.7, 33.9, 32.2, 28.4, 28.3, 27.9, 27.5, 26.0, 22.9, 22.5, 21.4, 20.4.

IR ν 3443, 2931, 2857, 1471, 1386, 1013, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{18}H_{38}O_3SiNa^+$ 353.2488, found 353.2487 (0.3 ppm error).



di-*tert*-butyl(2-phenyl-1-tetrahydrofuran-2-yl)ethoxy)silanol

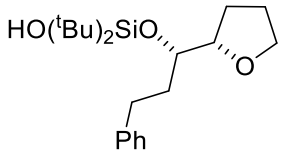
Compound 50: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 53 mg, 0.15 mmol, 75% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.15 (m, 5H), 4.04 (td, J = 8.0, 3.4 Hz, 1H), 3.93 – 3.76 (m, 2H), 3.69 (ddd, J = 9.5, 8.1, 6.0 Hz, 1H), 2.86 (dd, J = 13.5, 3.4 Hz, 1H), 2.61 (dd, J = 13.5, 8.0 Hz, 1H), 2.09 – 1.82 (m, 3H), 1.54 (dq, J = 11.9, 9.2 Hz, 1H), 0.99 (s, 9H), 0.77 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 138.2, 130.3, 128.1, 126.3, 84.0, 77.5, 67.8, 40.8, 28.6, 27.5, 27.4, 26.0, 21.0, 20.4.

IR ν 3440, 2964, 2859, 1471, 1386, 1012, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{20}H_{34}O_3SiNa^+$ 373.2175, found 373.2177 (0.5 ppm error).



di-*tert*-butyl(3-phenyl-1-(tetrahydrofuran-2-yl)propoxy)silanol

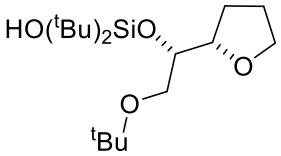
Compound 52: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 59 mg, 0.162 mmol, 81% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.09 (m, 5H), 3.99 (td, J = 7.8, 3.3 Hz, 1H), 3.96 – 3.88 (m, 2H), 3.85 – 3.79 (m, 1H), 2.95 (ddd, J = 13.7, 11.7, 5.3 Hz, 1H), 2.81 (ddd, J = 13.6, 11.6, 5.2 Hz, 1H), 2.04 – 1.72 (m, 5H), 1.54 – 1.45 (m, 1H), 1.14 (s, 9H), 1.12 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 142.8, 128.54, 128.46, 125.9, 84.1, 76.3, 67.8, 36.8, 31.5, 28.4, 27.9, 27.6, 26.0, 21.4, 20.5.

IR ν 3445, 2957, 1471, 1386, 1013, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{21}H_{36}O_3SiNa^+$ 387.2331, found 387.2327 (1.0 ppm error).



(2-(*tert*-butoxy)-1-tetrahydrofuran-2-yl)ethoxy)di-*tert*-butylsilanol

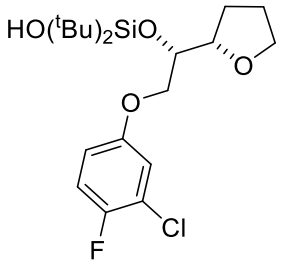
Compound 54: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 59 mg, 0.170 mmol, 85% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.04 (ddd, J = 6.8, 4.6, 3.3 Hz, 1H), 3.90 (td, J = 7.0, 4.6 Hz, 1H), 3.85 – 3.73 (m, 2H), 3.47 – 3.43 (m, 2H), 1.97 – 1.77 (m, 4H), 1.21 (s, 9H), 1.02 (s, 9H), 1.00 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 81.2, 75.3, 74.2, 68.6, 65.0, 27.7, 27.6, 27.5, 27.4, 26.2, 21.2, 20.6.

IR ν 3419, 2965, 2859, 1473, 1363, 1013, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H^+] Calcd $C_{18}H_{39}O_4Si^+$ 347.2618, found 347.2634 (4.6 ppm error).



di-*tert*-butyl(2-(3-chloro-4-fluorophenoxy)-1-tetrahydrofuran-2-yl)ethoxy)silanol

Compound 56: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 54 mg, 0.129 mmol, 64% yield).

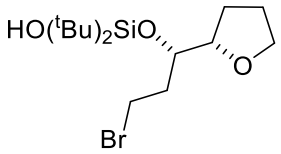
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.08 – 6.92 (m, 2H), 6.77 (dt, J = 9.0, 3.3 Hz, 1H), 4.20 (ddd, J = 7.2, 5.3, 4.1 Hz, 1H), 4.02 – 3.92 (m, 2H), 3.91 – 3.79 (m, 3H), 2.04 – 1.84 (m, 3H), 1.73 – 1.59 (m, 1H), 1.03 (s, 9H), 1.00 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 155.2 (d, J = 2.8 Hz), 153.1 (d, J = 241.7 Hz), 121.3 (d, J = 19.2 Hz), 116.9 (d, J = 22.2 Hz), 116.4, 114.4 (d, J = 6.6 Hz), 81.8, 74.8, 71.6, 68.0, 27.8, 27.6, 27.4, 26.1, 21.0, 20.6.

^{19}F NMR (377 MHz, $CDCl_3$) δ -126.7 (t, J = 9.2 Hz).

IR ν 2933, 2859, 1558, 1473, 1203, 1072, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{20}H_{32}FClO_4SiNa^+$ 441.1640, found 441.1659 (4.3 ppm error).



((3-bromo-1-tetrahydrofuran-2-yl)propoxy)di-*tert*-butylsilanol

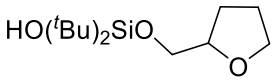
Compound 58: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 47 mg, 0.128 mmol, 64% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.96 (td, J = 7.8, 3.9 Hz, 1H), 3.91 – 3.78 (m, 2H), 3.75 – 3.70 (m, 1H), 3.61 – 3.50 (m, 2H), 2.06 – 1.81 (m, 5H), 1.51 – 1.36 (m, 1H), 1.04 (s, 9H), 0.98 (s, 9H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 83.7, 75.2, 68.0, 38.2, 29.8, 28.5, 27.9, 27.5, 25.9, 21.4, 20.4.

IR ν 3427, 2964, 2859, 1471, 1386, 1013, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{15}H_{31}BrO_3SiNa^+$ 389.1124, found 389.1136 (3.1 ppm error).



di-*tert*-butyl((tetrahydrofuran-2-yl)methoxy)silanol

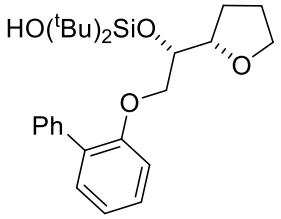
Compound 60: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0.1 to 2.0% acetone in CH_2Cl_2 on silica gel; (colorless oil, 31.3 mg, 0.120 mmol, 60% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.96 (ddt, *J* = 11.0, 6.3, 3.0 Hz, 1H), 3.91 – 3.84 (m, 2H), 3.82 – 3.76 (m, 2H), 1.98 – 1.79 (m, 3H), 1.68 – 1.54 (m, 1H), 1.01 (s, 9H), 1.01 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 80.7, 68.3, 66.1, 27.6, 27.5, 27.0, 25.8, 20.9, 20.6.

IR ν 3410, 2933, 2859, 1473, 1078, 862, 827, 648 cm^{-1}.

HRMS (ESI) m/z = [M + Na$^+$] calculated for $C_{13}H_{28}O_3SiNa^+$ 283.1705, Found 283.1705 (0.0 ppm error).



(2-([1,1'-biphenyl]-2-yloxy)-1-(tetrahydrofuran-2-yl)ethoxy)di-*tert*-butylsilanol

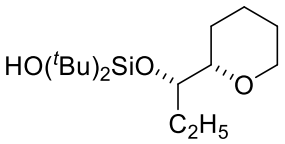
Compound 62: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 44 mg, 0.099 mmol, 50% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 – 7.50 (m, 2H), 7.43 – 7.25 (m, 5H), 7.10 – 6.98 (m, 2H), 4.11 – 3.98 (m, 3H), 3.85 – 3.68 (m, 3H), 1.87 – 1.65 (m, 3H), 1.49 – 1.26 (m, 1H), 0.99 (s, 18H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 156.0, 138.5, 131.4, 131.0, 129.8, 128.7, 128.0, 127.0, 121.4, 112.9, 82.2, 75.1, 71.7, 67.7, 27.7, 27.5, 27.4, 26.0, 21.0, 20.5.

IR ν 3649, 2965, 1505, 1473, 1233, 1009, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na$^+$] Calcd $C_{26}H_{38}O_4SiNa^+$ 465.2437, found 465.2429 (1.7 ppm error).



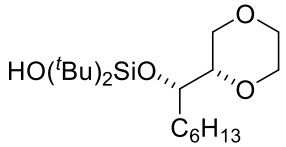
Compound 64: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 1-4% diethyl ether in hexane on silica gel; (colorless oil, 6.0 mg, 0.02 mmol, 10% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.37 (s, 1H), 4.01 (ddt, *J* = 11.4, 4.4, 1.8 Hz, 1H), 3.79 (ddd, *J* = 7.9, 5.5, 4.0 Hz, 1H), 3.38 (td, *J* = 11.6, 2.5 Hz, 1H), 3.19 (ddd, *J* = 11.4, 7.9, 2.0 Hz, 1H), 1.89 – 1.82 (m, 1H), 1.65 – 1.39 (m, 6H), 1.24 – 1.14 (m, 1H), 1.02 (s, 9H), 0.98 (s, 9H), 0.94 (t, *J* = 7.4 Hz, 3H).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 82.4, 75.8, 68.5, 27.7, 27.6, 27.4, 25.9, 25.7, 23.4, 21.2, 20.5, 8.4.

IR ν 3447, 2933, 2856, 1473, 1080, 878, 827, 647 cm^{-1}.

HRMS (ESI) m/z = [M + H^+] calculated for $C_{16}H_{35}O_3Si^+$ 303.2355, Found 303.2343 (error 4.0 ppm).



(((*S**)-1-((*S**)-1,4-dioxan-2-yl)heptyl)oxy)di-*tert*-butylsilanol

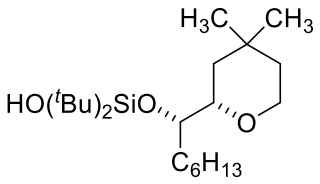
Compound 66: Synthesized using General Procedure C on a 0.2 mmol scale.; Purified using a gradient of 1-4% diethyl ether in hexane on silica gel; single diastereomer; (colorless oil, 11.0 mg, 0.030 mmol, 15% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.89 – 3.79 (m, 3H), 3.77 – 3.67 (m, 3H), 3.60 (dd, *J* = 11.9, 2.7 Hz, 1H), 3.56 – 3.49 (m, 1H), 3.30 (dd, *J* = 11.3, 10.4 Hz, 1H), 1.50 – 1.41 (m, 2H), 1.32 – 1.23 (m, 8H), 1.02 (s, 9H), 0.99 (s, 9H), 0.90 – 0.86 (m, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 80.0, 72.6, 67.8, 67.0, 66.2, 33.0, 31.8, 29.8, 27.7, 27.4, 23.9, 22.7, 21.1, 20.5, 14.2.

IR ν 3490, 2931, 2857, 1473, 1127, 827, 647 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{19}H_{40}O_4SiNa^+$ 383.2594, Found 383.2596 (0.5 ppm error).



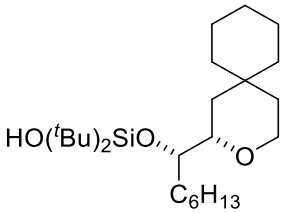
Compound 68: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 1-4% diethyl ether in hexane on silica gel; single diastereomer; (colorless oil, 54.1 mg, 0.140 mmol, 70% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 3.85 (ddd, *J* = 11.5, 5.1, 1.5 Hz, 1H), 3.79 (ddd, *J* = 7.8, 5.4, 3.6 Hz, 1H), 3.52 (ddd, *J* = 13.0, 11.7, 2.2 Hz, 1H), 3.31 (ddd, *J* = 11.9, 7.9, 2.0 Hz, 1H), 1.54 – 1.38 (m, 4H), 1.38 – 1.17 (m, 10H), 1.01 (s, 9H), 1.00 (s, 3H), 0.98 (s, 9H), 0.96 (s, 3H), 0.90 – 0.87 (m, 3H).

^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 78.3, 75.7, 64.3, 40.7, 38.5, 33.4, 33.2, 31.9, 30.0, 28.8, 27.7, 27.5, 24.2, 23.7, 22.8, 21.2, 20.5, 14.2.

IR ν 3462, 2954, 2931, 2857, 1471, 1102, 1060, 871, 827, 647 cm^{-1}.

HRMS (ESI) m/z: [M + Na^+] calculated for $C_{22}H_{46}O_3SiNa^+$ 409.3114, Found 409.3136 (error 5.4 ppm).



(((*S**)-1-((*S**)-3-oxaspiro[5.5]undecan-2-yl)heptyl)oxy)di-*tert*-butylsilanol

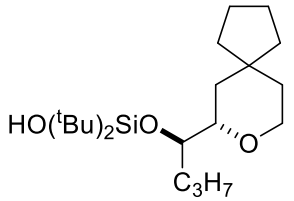
Compound 70: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 1-4% diethyl ether in hexane on silica gel; single diastereomer; (colorless oil, 70.0 mg, 0.164 mmol, 82% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.33 (s, 1H), 3.85 – 3.75 (m, 2H), 3.54 (ddd, *J* = 13.8, 11.5, 2.2 Hz, 1H), 3.34 (ddd, *J* = 11.9, 7.8, 1.8 Hz, 1H), 1.61 – 1.36 (m, 14H), 1.35 – 1.23 (m, 10H), 1.01 (s, 9H), 0.98 (s, 9H), 0.89 (t, *J* = 6.6 Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 77.4, 75.7, 63.7, 42.3, 38.8, 36.0, 33.3, 32.0, 31.9, 31.3, 30.0, 27.7, 27.5, 26.8, 23.7, 22.7, 21.6, 21.4, 21.2, 20.5, 14.2.

IR ν 3446, 2928, 2856, 1471, 1107, 1093, 827, 647 cm^{-1}.

HRMS (ESI) m/z: [M + H^+] calculated for $C_{25}H_{51}O_3Si^+$ 427.3607, Found 427.3639 (7.5 ppm error).



(oxaspiro[4.5]decan-7-yl)butoxy)di-*tert*-butylsilanol

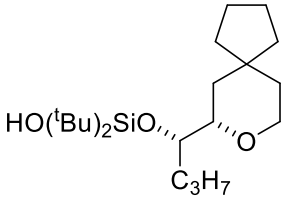
Compound 72: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 37 mg, 0.100 mmol, 50% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.19 (td, J = 6.7, 2.2 Hz, 1H), 3.96 (ddd, J = 11.5, 4.9, 1.5 Hz, 1H), 3.60 (ddd, J = 13.6, 11.6, 2.1 Hz, 1H), 3.35 (dt, J = 11.8, 2.1 Hz, 1H), 1.83 – 1.22 (m, 16H), 1.06 (s, 9H), 1.05 (s, 9H), 0.98 – 0.93 (m, 3H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 77.9, 74.5, 66.0, 42.8, 40.4, 37.7, 36.3, 34.6 (2C), 28.0, 27.9, 25.0, 23.8, 21.5, 20.6, 19.5, 14.5.

IR ν 3443, 2941, 2864, 1471, 1387, 1062, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{21}H_{42}O_3SiNa^+$ 393.2801, found 393.2809 (2.0 ppm error).



(oxaspiro[4.5]decan-7-yl)butoxy)di-*tert*-butylsilanol

Compound 74: Synthesized using General Procedure C on a 0.2 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 49 mg, 0.132 mmol, 66% yield).

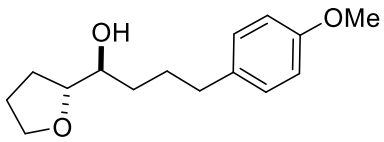
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.90 (ddd, J = 11.4, 4.9, 1.5 Hz, 1H), 3.81 (ddd, J = 7.8, 5.6, 3.5 Hz, 1H), 3.45 (ddd, J = 12.9, 11.5, 2.1 Hz, 1H), 3.24 (ddd, J = 11.8, 7.8, 2.0 Hz, 1H), 1.71 – 1.25 (m, 16H), 1.00 (s, 9H), 0.97 (s, 9H), 0.91 (t, J = 7.0 Hz, 3H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) 79.8, 75.5, 65.6, 42.7, 40.8, 39.8, 37.5, 35.7, 34.7, 27.7, 27.5, 24.9, 23.8, 21.2, 20.5, 17.3, 14.9.

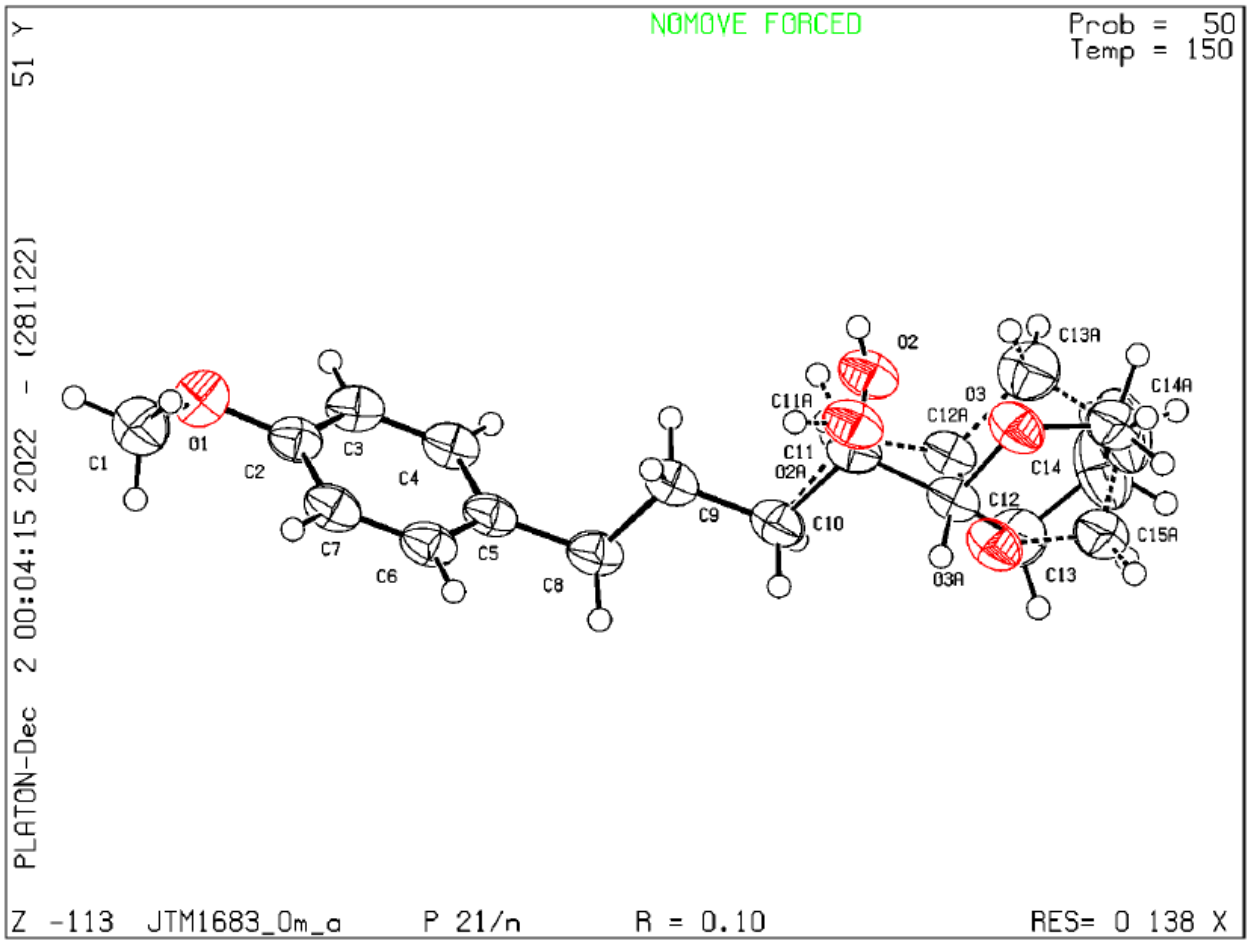
IR ν 3445, 2954, 2857, 1471, 1386, 1097, 827 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + H^+] Calcd $C_{21}H_{43}O_3Si^+$ 371.2981, found 371.2963 (4.9 ppm error).

IX. X-ray Crystallography



CCDC 2233482



Crystals grown from ethanol.

Crystal Structure Report for JTM1683

A colorless, plate-like specimen of $C_{15}H_{22}O_3$, approximate dimensions 0.112 mm x 0.188 mm x 0.272 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured on a Bruker D8 VENTURE PHOTON 3 CPAD

system equipped with a INCOATEC IμS micro--focus source (Cu-Kα, λ = 1.54178 Å) and a mirror monochromator.

Table 1: Data collection details for JTM1683.

Axis	dx/mm	2θ/°	ω/°	φ/°	χ/°	Width/°	Frames	Time/s	Wavelength/Å	Voltage/kV	Current/mA	Temperature/K
Omega	44.970	110.11	-67.70	96.00	54.76	0.50	352	15.00	1.54184	50	1.0	n/a
Omega	44.970	110.11	-67.70	-136.00	54.76	0.50	352	15.00	1.54184	50	1.0	n/a
Omega	44.970	45.19	43.38	0.00	54.76	0.50	352	5.00	1.54184	50	1.0	n/a
Omega	44.970	110.11	-67.70	32.00	54.76	0.50	352	15.00	1.54184	50	1.0	n/a
Omega	44.970	80.11	-97.70	180.00	54.76	0.50	352	8.40	1.54184	50	1.0	n/a
Omega	44.970	110.11	-67.70	128.00	54.76	0.50	352	15.00	1.54184	50	1.0	n/a
Omega	44.970	110.11	-67.70	-168.00	54.76	0.50	352	15.00	1.54184	50	1.0	n/a
Omega	44.970	110.11	-67.70	64.00	54.76	0.50	352	15.00	1.54184	50	1.0	n/a
Omega	44.970	110.11	-67.70	160.00	54.76	0.50	352	15.00	1.54184	50	1.0	n/a
Phi	44.970	50.11	-128.07	0.00	54.76	0.50	720	5.30	1.54184	50	1.0	n/a
Phi	44.970	95.11	-83.07	0.00	54.76	0.50	720	11.20	1.54184	50	1.0	n/a

Axis	dx/mm	2θ/°	ω/°	φ/°	χ/°	Width/°	Frames	Time/s	Wavelength/Å	Voltage/kV	Current/mA	Temperature/K
Omega	44.970	110.11	-67.70	0.00	54.76	0.50	352	15.00	1.54184	50	1.0	n/a
Omega	44.970	110.11	-67.70	-40.00	54.76	0.50	352	15.00	1.54184	50	1.0	n/a
Omega	44.970	65.11	63.30	90.00	54.76	0.50	352	6.50	1.54184	50	1.0	n/a

A total of 5664 frames were collected. The total exposure time was 18.45 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 22882 reflections to a maximum θ angle of 72.50° (0.81 Å resolution), of which 2709 were independent (average redundancy 8.447, completeness = 99.0%, R_{int} = 5.56%, R_{sig} = 3.19%) and 2427 (89.59%) were greater than 2σ(F^2). The final cell constants of a = 8.6600(5) Å, b = 5.9392(3) Å, c = 26.8603(14) Å, β = 93.256(3)°, volume = 1379.29(13) $Å^3$, are based upon the refinement of the XYZ-centroids of 9658 reflections above 20 σ(I) with 6.592° < 2θ < 144.6°. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.819. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.8410 and 0.9300.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P 1 21/n 1, with Z = 4 for the formula unit, $C_{15}H_{22}O_3$. The final anisotropic full-matrix least-squares refinement on F^2 with 190 variables converged at R1 = 9.70%, for the observed data and wR2 = 25.52% for all data. The goodness-of-fit was 1.051. The largest peak in the final difference electron density synthesis was 0.873 $e^-/Å^3$ and the largest hole was -0.484 $e^-/Å^3$ with an RMS deviation of 0.064 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.205 g/cm^3 and F(000), 544 e^-.

Table 2. Sample and crystal data for JTM1683.

Identification code	JTM1683
Chemical formula	$C_{15}H_{22}O_3$
Formula weight	250.32 g/mol
Temperature	150(2) K

Wavelength	1.54178 Å	
Crystal size	0.112 x 0.188 x 0.272 mm	
Crystal habit	colorless plate	
Crystal system	monoclinic	
Space group	P 1 21/n 1	
Unit cell dimensions	a = 8.6600(5) Å	α = 90°
	b = 5.9392(3) Å	β = 93.256(3)°
	c = 26.8603(14) Å	γ = 90°
Volume	1379.29(13) $Å^3$	
Z	4	
Density (calculated)	1.205 g/cm^3	
Absorption coefficient	0.660 mm^{-1}	
F(000)	544	

Table 3. Data collection and structure refinement for JTM1683.

Diffractometer	Bruker D8 VENTURE PHOTON 3 CPAD
Radiation source	INCOATEC IμS micro--focus source (Cu-Kα, λ = 1.54178 Å)
Theta range for data collection	3.30 to 72.50°
Index ranges	-10<=h<=10, -7<=k<=7, -33<=l<=32
Reflections collected	22882
Independent reflections	2709 [R(int) = 0.0556]
Coverage of independent reflections	99.0%
Absorption correction	multi-scan
Max. and min. transmission	0.9300 and 0.8410

Structure solution technique	direct methods	
Structure solution program	*SHELXT* (Sheldrick, 2015*a*)	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	*SHELXL* (Sheldrick, 2015*b*)	
Function minimized	$\Sigma\ w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	2709 / 65 / 190	
Goodness-of-fit on F^2	1.051	
Final R indices	2427 data; $I>2\sigma(I)$	R1 = 0.0970, wR2 = 0.2500
	all data	R1 = 0.1036, wR2 = 0.2552
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.1106P)^2+2.3061P]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	0.873 and -0.484 eÅ^{-3}	
R.M.S. deviation from mean	0.064 eÅ^{-3}	

Table 4. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2) for JTM1683.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
O1	0.6271(3)	0.1831(4)	0.81928(8)	0.0529(6)

	x/a	y/b	z/c	U(eq)
C1	0.7245(4)	0.9928(7)	0.82238(15)	0.0591(9)
C2	0.5424(3)	0.2152(5)	0.77488(11)	0.0423(7)
C3	0.4513(4)	0.4079(5)	0.77173(12)	0.0455(7)
C4	0.3610(4)	0.4549(5)	0.72875(12)	0.0447(7)
C5	0.3587(3)	0.3114(5)	0.68757(11)	0.0427(7)
C6	0.4512(4)	0.1201(5)	0.69169(12)	0.0466(8)
C7	0.5428(4)	0.0700(5)	0.73434(12)	0.0462(7)
C8	0.2645(4)	0.3649(6)	0.64041(12)	0.0481(8)
C9	0.3494(4)	0.5236(5)	0.60616(12)	0.0464(7)
C10	0.2635(4)	0.5657(5)	0.55617(13)	0.0484(8)
O2	0.3230(4)	0.9498(5)	0.54393(11)	0.0538(8)
O3	0.3796(3)	0.8720(5)	0.43982(11)	0.0525(8)
C11	0.3433(4)	0.7304(6)	0.52314(14)	0.0426(9)
C12	0.2851(4)	0.7285(6)	0.46887(14)	0.0429(8)
C13	0.1155(5)	0.8130(10)	0.4583(2)	0.0649(14)
C14	0.1384(7)	0.0302(12)	0.4312(2)	0.0837(17)
C15	0.2858(5)	0.0222(8)	0.41014(17)	0.0551(10)
O2A	0.4607(11)	0.7026(18)	0.5091(4)	0.0538(8)
O3A	0.1538(13)	0.7506(16)	0.4596(4)	0.0525(8)
C11A	0.3288(11)	0.7767(12)	0.5343(3)	0.0426(9)
C12A	0.2149(10)	0.8966(14)	0.4989(3)	0.0429(8)
C13A	0.2801(16)	0.1035(18)	0.4709(5)	0.0649(14)
C14A	0.207(3)	0.078(3)	0.4193(5)	0.0837(17)
C15A	0.1085(17)	0.886(2)	0.4175(4)	0.0551(10)

Table 5. Bond lengths (Å) for JTM1683.

O1-C2	1.377(4)	O1-C1	1.410(4)
C1-H1A	0.980000	C1-H1B	0.980000
C1-H1C	0.980000	C2-C7	1.389(4)
C2-C3	1.390(4)	C3-C4	1.385(5)
C3-H3	0.950000	C4-C5	1.396(4)

C4-H4	0.950000	C5-C6	1.391(5)
C5-C8	1.501(5)	C6-C7	1.389(5)
C6-H6	0.950000	C7-H7	0.950000
C8-C9	1.534(4)	C8-H8A	0.990000
C8-H8B	0.990000	C9-C10	1.519(5)
C9-H9A	0.990000	C9-H9B	0.990000
C10-C11A	1.508(5)	C10-C11	1.514(4)
C10-H10A	0.990000	C10-H10B	0.990000
O2-C11	1.433(4)	O2-H2	0.841(10)
O3-C15	1.420(5)	O3-C12	1.441(4)
C11-C12	1.515(5)	C11-H11	1.000000
C12-C13	1.563(6)	C12-H12	1.000000
C13-C14	1.499(8)	C13-H13A	0.990000
C13-H13B	0.990000	C14-C15	1.426(7)
C14-H14A	0.990000	C14-H14B	0.990000
C15-H15A	0.990000	C15-H15B	0.990000
O2A-C11A	1.430(5)	O2A-H2A	0.840000
O3A-C15A	1.426(5)	O3A-C12A	1.443(5)
C11A-C12A	1.509(5)	C11A-H11A	1.000000
C12A-C13A	1.562(6)	C12A-H12A	1.000000
C13A-C14A	1.497(8)	C13A-H13C	0.990000
C13A-H13D	0.990000	C14A-C15A	1.426(7)
C14A-H14C	0.990000	C14A-H14D	0.990000

C15A-H15C	0.990000	C15A-H15D	0.990000

Table 6. Bond angles (°) for JTM1683.

C2-O1-C1	116.7(3)	O1-C1-H1A	109.500000
O1-C1-H1B	109.500000	H1A-C1-H1B	109.500000
O1-C1-H1C	109.500000	H1A-C1-H1C	109.500000
H1B-C1-H1C	109.500000	O1-C2-C7	124.6(3)
O1-C2-C3	116.1(3)	C7-C2-C3	119.3(3)
C4-C3-C2	120.6(3)	C4-C3-H3	119.700000
C2-C3-H3	119.700000	C3-C4-C5	121.3(3)
C3-C4-H4	119.300000	C5-C4-H4	119.300000
C6-C5-C4	117.0(3)	C6-C5-C8	121.5(3)
C4-C5-C8	121.5(3)	C7-C6-C5	122.7(3)
C7-C6-H6	118.600000	C5-C6-H6	118.600000
C6-C7-C2	119.1(3)	C6-C7-H7	120.400000
C2-C7-H7	120.400000	C5-C8-C9	112.3(3)
C5-C8-H8A	109.100000	C9-C8-H8A	109.100000
C5-C8-H8B	109.100000	C9-C8-H8B	109.100000
H8A-C8-H8B	107.900000	C10-C9-C8	113.8(3)
C10-C9-H9A	108.800000	C8-C9-H9A	108.800000
C10-C9-H9B	108.800000	C8-C9-H9B	108.800000
H9A-C9-H9B	107.700000	C11A-C10-C9	107.8(4)
C11-C10-C9	114.1(3)	C11-C10-H10A	108.700000
C9-C10-H10A	108.700000	C11-C10-H10B	108.700000

C9-C10-H10B	108.700000	H10A-C10-H10B	107.600000
C11-O2-H2	95.(4)	C15-O3-C12	110.6(3)
O2-C11-C10	106.8(3)	O2-C11-C12	109.9(3)
C10-C11-C12	114.9(3)	O2-C11-H11	108.300000
C10-C11-H11	108.300000	C12-C11-H11	108.300000
O3-C12-C11	110.4(3)	O3-C12-C13	105.6(3)
C11-C12-C13	115.5(3)	O3-C12-H12	108.400000
C11-C12-H12	108.400000	C13-C12-H12	108.400000
C14-C13-C12	102.4(4)	C14-C13-H13A	111.300000
C12-C13-H13A	111.300000	C14-C13-H13B	111.300000
C12-C13-H13B	111.300000	H13A-C13-H13B	109.200000
C15-C14-C13	108.0(4)	C15-C14-H14A	110.100000
C13-C14-H14A	110.100000	C15-C14-H14B	110.100000
C13-C14-H14B	110.100000	H14A-C14-H14B	108.400000
O3-C15-C14	107.1(4)	O3-C15-H15A	110.300000
C14-C15-H15A	110.300000	O3-C15-H15B	110.300000
C14-C15-H15B	110.300000	H15A-C15-H15B	108.500000
C11A-O2A-H2A	109.500000	C15A-O3A-C12A	108.3(4)
O2A-C11A-C10	104.8(8)	O2A-C11A-C12A	111.0(4)

C10-C11A-C12A	113.1(6)	O2A-C11A-H11A	109.300000
C10-C11A-H11A	109.300000	C12A-C11A-H11A	109.300000
O3A-C12A-C11A	112.1(4)	O3A-C12A-C13A	104.4(4)
C11A-C12A-C13A	115.7(4)	O3A-C12A-H12A	108.100000
C11A-C12A-H12A	108.100000	C13A-C12A-H12A	108.100000
C14A-C13A-C12A	102.8(4)	C14A-C13A-H13C	111.200000
C12A-C13A-H13C	111.200000	C14A-C13A-H13D	111.200000
C12A-C13A-H13D	111.200000	H13C-C13A-H13D	109.100000
C15A-C14A-C13A	109.4(5)	C15A-C14A-H14C	109.800000
C13A-C14A-H14C	109.800000	C15A-C14A-H14D	109.800000
C13A-C14A-H14D	109.800000	H14C-C14A-H14D	108.200000
O3A-C15A-C14A	106.7(5)	O3A-C15A-H15C	110.400000
C14A-C15A-H15C	110.400000	O3A-C15A-H15D	110.400000
C14A-C15A-H15D	110.400000	H15C-C15A-H15D	108.600000

Table 7. Torsion angles (°) for JTM1683.

C1-O1-C2-C7	2.4(4)	C1-O1-C2-C3	-177.7(3)
O1-C2-C3-C4	-179.6(3)	C7-C2-C3-C4	0.3(4)
C2-C3-C4-C5	0.0(5)	C3-C4-C5-C6	-0.2(4)
C3-C4-C5-C8	-178.3(3)	C4-C5-C6-C7	0.0(4)
C8-C5-C6-C7	178.1(3)	C5-C6-C7-C2	0.3(5)
O1-C2-C7-C6	179.4(3)	C3-C2-C7-C6	-0.4(4)
C6-C5-C8-C9	-94.2(3)	C4-C5-C8-C9	83.9(4)
C5-C8-C9-C10	175.1(3)	C8-C9-C10-C11A	161.1(5)
C8-C9-C10-C11	177.4(3)	C9-C10-C11-O2	-73.5(4)
C9-C10-C11-C12	164.3(3)	C15-O3-C12-C11	-129.8(3)
C15-O3-C12-C13	-4.3(4)	O2-C11-C12-O3	65.9(4)
C10-C11-C12-O3	-173.6(3)	O2-C11-C12-C13	-53.7(4)
C10-C11-C12-C13	66.8(4)	O3-C12-C13-C14	-10.9(5)
C11-C12-C13-C14	111.4(4)	C12-C13-C14-C15	22.5(6)
C12-O3-C15-C14	18.9(5)	C13-C14-C15-O3	-26.1(6)
C9-C10-C11A-O2A	85.3(6)	C9-C10-C11A-C12A	-153.7(5)
C15A-O3A-C12A-C11A	-153.7(8)	C15A-O3A-C12A-C13A	-27.8(9)
O2A-C11A-C12A-O3A	62.8(9)	C10-C11A-C12A-O3A	-54.7(8)
O2A-C11A-C12A-C13A	-56.8(10)	C10-C11A-C12A-C13A	-174.3(8)

O3A-C12A-C13A-C14A	15.6(13)	C11A-C12A-C13A-C14A	139.3(12)
C12A-C13A-C14A-C15A	1.5(17)	C12A-O3A-C15A-C14A	29.5(13)
C13A-C14A-C15A-O3A	-18.5(17)		

Table 8. Anisotropic atomic displacement parameters (Å^2) for JTM1683.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	0.0495(12)	0.0574(14)	0.0522(12)	-0.0081(11)	0.0064(10)	0.0082(11)
C1	0.0552(19)	0.057(2)	0.066(2)	0.0019(17)	0.0059(16)	0.0060(17)
C2	0.0399(15)	0.0416(15)	0.0467(15)	-0.0041(13)	0.0134(12)	-0.0053(12)
C3	0.0446(16)	0.0418(16)	0.0516(17)	-0.0128(13)	0.0145(13)	-0.0020(13)
C4	0.0444(16)	0.0373(15)	0.0540(17)	-0.0073(13)	0.0155(13)	-0.0014(13)
C5	0.0434(15)	0.0380(15)	0.0483(16)	-0.0027(12)	0.0157(12)	-0.0093(12)
C6	0.0566(18)	0.0363(15)	0.0486(16)	-0.0091(13)	0.0169(14)	-0.0071(13)
C7	0.0506(17)	0.0339(15)	0.0556(17)	-0.0051(13)	0.0168(14)	0.0008(13)
C8	0.0476(17)	0.0443(17)	0.0536(17)	-0.0038(14)	0.0116(13)	-0.0110(13)
C9	0.0478(17)	0.0389(16)	0.0531(17)	0.0001(13)	0.0091(13)	-0.0058(13)
C10	0.0429(16)	0.0393(16)	0.0635(19)	0.0059(14)	0.0068(14)	-0.0042(13)
O2	0.0611(17)	0.0369(14)	0.0647(17)	-0.0059(12)	0.0140(14)	-0.0035(13)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O3	0.0488(15)	0.0467(15)	0.0632(17)	0.0084(13)	0.0130(12)	-0.0042(12)
C11	0.0385(16)	0.0337(18)	0.056(2)	-0.0044(15)	0.0080(15)	-0.0019(14)
C12	0.0424(18)	0.0348(17)	0.0524(19)	-0.0002(15)	0.0112(15)	-0.0037(15)
C13	0.041(2)	0.067(3)	0.086(3)	0.002(2)	-0.001(2)	0.006(2)
C14	0.083(3)	0.094(4)	0.077(3)	0.032(3)	0.033(3)	0.028(3)
C15	0.059(2)	0.043(2)	0.062(2)	0.0031(18)	-0.0024(19)	-0.0040(18)
O2A	0.0611(17)	0.0369(14)	0.0647(17)	-0.0059(12)	0.0140(14)	-0.0035(13)
O3A	0.0488(15)	0.0467(15)	0.0632(17)	0.0084(13)	0.0130(12)	-0.0042(12)
C11A	0.0385(16)	0.0337(18)	0.056(2)	-0.0044(15)	0.0080(15)	-0.0019(14)
C12A	0.0424(18)	0.0348(17)	0.0524(19)	-0.0002(15)	0.0112(15)	-0.0037(15)
C13A	0.041(2)	0.067(3)	0.086(3)	0.002(2)	-0.001(2)	0.006(2)
C14A	0.083(3)	0.094(4)	0.077(3)	0.032(3)	0.033(3)	0.028(3)
C15A	0.059(2)	0.043(2)	0.062(2)	0.0031(18)	-0.0024(19)	-0.0040(18)

Table 9. Hydrogen atomic coordinates and isotropic atomic displacement parameters ($Å^2$) for JTM1683.

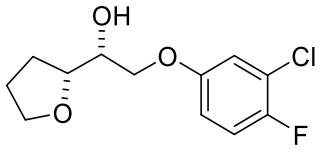
	x/a	y/b	z/c	U(eq)
H1A	0.6637	-0.1429	0.8143	0.089000
H1B	0.7718	-0.0198	0.8563	0.089000
H1C	0.8058	0.0087	0.7987	0.089000
H3	0.4509	0.5081	0.7993	0.055000

	x/a	y/b	z/c	U(eq)
H4	0.2995	0.5874	0.7273	0.054000
H6	0.4516	0.0196	0.6642	0.056000
H7	0.6050	-0.0619	0.7358	0.055000
H8A	0.1659	0.4356	0.6490	0.058000
H8B	0.2393	0.2231	0.6223	0.058000
H9A	0.3666	0.6695	0.6234	0.056000
H9B	0.4521	0.4586	0.6003	0.056000
H10A	0.2506	0.4205	0.5383	0.058000
H10B	0.1590	0.6238	0.5621	0.058000
H2	0.418(2)	0.983(10)	0.545(2)	0.081000
H11	0.4563	0.6949	0.5249	0.051000
H12	0.2925	0.5712	0.4560	0.051000
H13A	0.0543	0.7048	0.4372	0.078000
H13B	0.0633	0.8379	0.4897	0.078000
H14A	0.0557	1.0503	0.4046	0.100000
H14B	0.1343	1.1584	0.4547	0.100000
H15A	0.2748	0.9678	0.3753	0.066000
H15B	0.3330	1.1740	0.4103	0.066000
H2A	0.5169	0.6220	0.5284	0.081000
H11A	0.3632	0.8813	0.5619	0.051000
H12A	0.1267	0.9496	0.5183	0.051000
H13C	0.2490	1.2468	0.4863	0.078000
H13D	0.3943	1.0975	0.4708	0.078000
H14C	0.1465	1.2150	0.4102	0.100000
H14D	0.2886	1.0605	0.3952	0.100000
H15C	-0.0009	0.9326	0.4188	0.066000
H15D	0.1203	0.8009	0.3862	0.066000

Table 10. Hydrogen bond distances (Å) and angles (°) for JTM1683.

	Donor-H	Acceptor-H	Donor-Acceptor	Angle
O2^{a-} H2a···O3^a#1	0.841(10)	1.98(2)	2.796(4)	163.(6)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1, -y+2, -z+1



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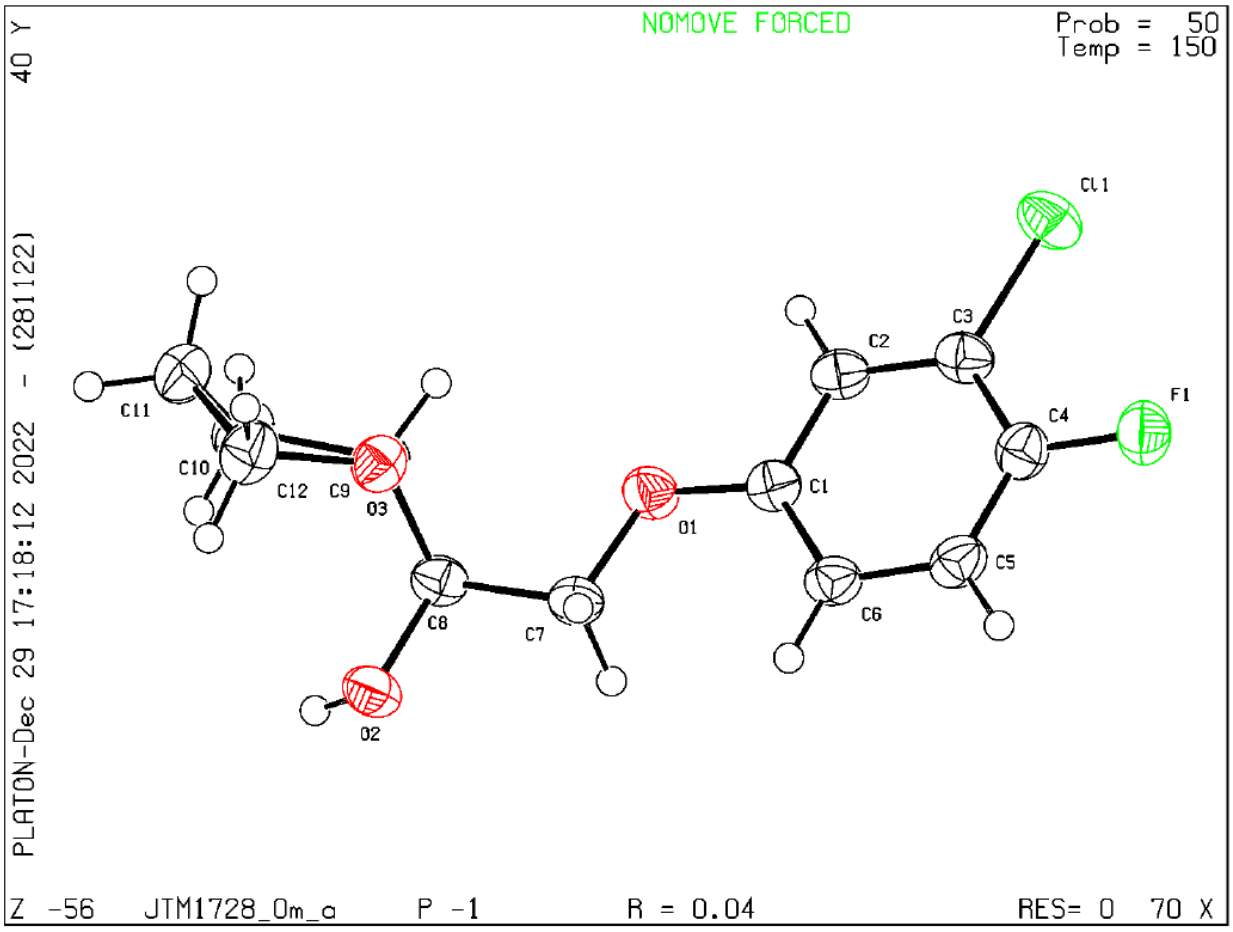


Crystals grown from pentane.

Crystal Structure Report for JTM1728

A colorless, block-like specimen of $C_{12}H_{14}ClFO_3$, approximate dimensions 0.135 mm x 0.136 mm x 0.219 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured on a Bruker D8 VENTURE PHOTON 3 CPAD system equipped with a INCOATEC IμS micro--focus source (Cu-Kα, λ = 1.54178 Å) and a mirror monochromator.

Table 1: Data collection details for JTM1728.

Axis	dx/mm	2θ/°	ω/°	φ/°	χ/°	Width/°	Frames	Time/s	Wavelength/Å	Voltage/kV	Current/mA	Temperature/K
Phi	39.968	48.14	40.70	0.00	54.76	0.50	720	4.00	1.54184	50	1.0	n/a
Omega	39.968	107.55	-64.89	0.00	54.76	0.50	330	15.00	1.54184	50	1.0	n/a
Omega	39.968	107.55	-64.89	252.00	54.76	0.50	330	15.00	1.54184	50	1.0	n/a
Omega	39.968	107.55	100.11	160.00	54.76	0.50	330	15.00	1.54184	50	1.0	n/a
Omega	39.968	107.55	-64.89	80.00	54.76	0.50	330	15.00	1.54184	50	1.0	n/a
Omega	39.968	62.55	55.11	0.00	54.76	0.50	330	5.30	1.54184	50	1.0	n/a
Omega	39.968	107.55	-64.89	120.00	54.76	0.50	330	15.00	1.54184	50	1.0	n/a
Omega	39.968	107.55	-64.89	-160.00	54.76	0.50	330	15.00	1.54184	50	1.0	n/a
Omega	39.968	107.55	100.11	-80.00	54.76	0.50	330	15.00	1.54184	50	1.0	n/a
Omega	39.968	107.55	100.11	40.00	54.76	0.50	330	15.00	1.54184	50	1.0	n/a
Omega	39.968	77.55	70.11	180.00	54.76	0.50	330	7.40	1.54184	50	1.0	n/a
Omega	39.968	107.55	-64.89	-40.00	54.76	0.50	330	15.00	1.54184	50	1.0	n/a
Omega	39.968	107.55	-64.89	-120.00	54.76	0.50	330	15.00	1.54184	50	1.0	n/a

A total of 4680 frames were collected. The total exposure time was 15.71 hours. The frames were integrated with the Bruker SAINT software package using a narrow-

frame algorithm. The integration of the data using a triclinic unit cell yielded a total of 11951 reflections to a maximum θ angle of 74.47° (0.80 Å resolution), of which 2412 were independent (average redundancy 4.955, completeness = 98.6%, R_{int} = 3.18%, R_{sig} = 2.49%) and 2327 (96.48%) were greater than 2σ(F^2). The final cell constants of a = 4.9861(2) Å, b = 11.0524(5) Å, c = 11.1934(5) Å, α = 94.348(2)°, β = 97.8150(10)°, γ = 99.1760(10)°, volume = 600.30(5) $Å^3$, are based upon the refinement of the XYZ-centroids of 9936 reflections above 20 σ(I) with 8.012° < 2θ < 148.9°. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.839. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.5680 and 0.6950.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P -1, with Z = 2 for the formula unit, $C_{12}H_{14}ClFO_3$. The final anisotropic full-matrix least-squares refinement on F^2 with 158 variables converged at R1 = 3.59%, for the observed data and wR2 = 9.91% for all data. The goodness-of-fit was 1.056. The largest peak in the final difference electron density synthesis was 0.415 $e^-/Å^3$ and the largest hole was -0.182 $e^-/Å^3$ with an RMS deviation of 0.053 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.442 g/cm^3 and F(000), 272 e^-.

Table 2. Sample and crystal data for JTM1728.

Identification code	JTM1728	
Chemical formula	$C_{12}H_{14}ClFO_3$	
Formula weight	260.68 g/mol	
Temperature	150(2) K	
Wavelength	1.54178 Å	
Crystal size	0.135 x 0.136 x 0.219 mm	
Crystal habit	colorless block	
Crystal system	triclinic	
Space group	P -1	
Unit cell dimensions	a = 4.9861(2) Å	α = 94.348(2)°
	b = 11.0524(5) Å	β = 97.8150(10)°
	c = 11.1934(5) Å	γ = 99.1760(10)°
Volume	600.30(5) $Å^3$	
Z	2	
Density (calculated)	1.442 g/cm^3	
Absorption coefficient	2.911 mm^{-1}	

F(000)	272

Table 3. Data collection and structure refinement for JTM1728.

Diffractometer	Bruker D8 VENTURE PHOTON 3 CPAD
Radiation source	INCOATEC IμS micro--focus source (Cu-Kα, λ = 1.54178 Å)
Theta range for data collection	4.01 to 74.47°
Index ranges	-6<=h<=6, -13<=k<=13, -13<=l<=13
Reflections collected	11951
Independent reflections	2412 [R(int) = 0.0318]
Coverage of independent reflections	98.6%
Absorption correction	multi-scan
Max. and min. transmission	0.6950 and 0.5680
Structure solution technique	direct methods
Structure solution program	*SHELXT* (Sheldrick, 2015*a*)
Refinement method	Full-matrix least-squares on F^2
Refinement program	*SHELXL* (Sheldrick, 2015*b*)
Function minimized	$\Sigma\ w(F_o^2 - F_c^2)^2$
Data / restraints / parameters	2412 / 0 / 158
Goodness-of-fit on F^2	1.056
Δ/σ_{max}	0.001

Final R indices	2327 data; I>2σ(I)	R1 = 0.0359, wR2 = 0.0979
	all data	R1 = 0.0368, wR2 = 0.0991
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0605P)^2+0.1513P]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	0.415 and -0.182 eÅ^{-3}	
R.M.S. deviation from mean	0.053 eÅ^{-3}	

Table 4. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2) for JTM1728.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
Cl1	0.11344(7)	0.44586(3)	0.27570(3)	0.04120(14)
F1	0.00488(18)	0.19015(8)	0.16785(7)	0.0415(2)
O1	0.3988(2)	0.28176(9)	0.52755(9)	0.0342(2)
O2	0.9420(2)	0.13053(9)	0.71267(10)	0.0360(2)
O3	0.50551(19)	0.19029(9)	0.81613(8)	0.0338(2)
C1	0.5391(3)	0.25236(12)	0.43576(12)	0.0303(3)
C2	0.7256(3)	0.34910(12)	0.40552(12)	0.0319(3)
C3	0.8799(3)	0.32749(12)	0.31519(12)	0.0320(3)
C4	0.8498(3)	0.21031(13)	0.25565(12)	0.0337(3)
C5	0.6636(3)	0.11510(13)	0.28351(12)	0.0347(3)
C6	0.5043(3)	0.13532(12)	0.37345(12)	0.0326(3)
C7	0.2221(3)	0.18251(12)	0.56752(12)	0.0308(3)
C8	0.1175(3)	0.23268(12)	0.67909(12)	0.0313(3)
C9	0.3515(3)	0.28599(12)	0.78107(12)	0.0321(3)
C10	0.2631(3)	0.33720(14)	0.89774(14)	0.0412(3)

	x/a	y/b	z/c	U(eq)
C11	0.4650(3)	0.30015(14)	0.99740(13)	0.0410(3)
C12	0.4972(3)	0.17501(14)	0.94184(13)	0.0415(3)

Table 5. Bond lengths (Å) for JTM1728.

Cl1-C3	1.7335(14)	F1-C4	1.3573(16)
O1-C1	1.3669(17)	O1-C7	1.4327(16)
O2-C8	1.4212(16)	O2-H2A	0.81(2)
O3-C12	1.4352(17)	O3-C9	1.4498(16)
C1-C2	1.3950(19)	C1-C6	1.3959(19)
C2-C3	1.380(2)	C2-H2	0.950000
C3-C4	1.387(2)	C4-C5	1.372(2)
C5-C6	1.391(2)	C5-H5	0.950000
C6-H6	0.950000	C7-C8	1.5178(19)
C7-H7A	0.990000	C7-H7B	0.990000
C8-C9	1.5259(19)	C8-H8	1.000000
C9-C10	1.5311(19)	C9-H9	1.000000
C10-C11	1.524(2)	C10-H10A	0.990000
C10-H10B	0.990000	C11-C12	1.514(2)
C11-H11A	0.990000	C11-H11B	0.990000
C12-H12A	0.990000	C12-H12B	0.990000

Table 6. Bond angles (°) for JTM1728.

C1-O1-C7	116.80(10)	C8-O2-H2A	111.2(17)
C12-O3-C9	109.25(10)	O1-C1-C2	115.37(11)
O1-C1-C6	124.29(12)	C2-C1-C6	120.33(13)

C3-C2-C1	119.41(12)	C3-C2-H2	120.300000
C1-C2-H2	120.300000	C2-C3-C4	120.05(12)
C2-C3-Cl1	120.46(10)	C4-C3-Cl1	119.48(11)
F1-C4-C5	119.71(12)	F1-C4-C3	119.40(12)
C5-C4-C3	120.89(13)	C4-C5-C6	119.91(12)
C4-C5-H5	120.000000	C6-C5-H5	120.000000
C5-C6-C1	119.37(12)	C5-C6-H6	120.300000
C1-C6-H6	120.300000	O1-C7-C8	107.75(10)
O1-C7-H7A	110.200000	C8-C7-H7A	110.200000
O1-C7-H7B	110.200000	C8-C7-H7B	110.200000
H7A-C7-H7B	108.500000	O2-C8-C7	104.76(10)
O2-C8-C9	111.87(11)	C7-C8-C9	112.02(11)
O2-C8-H8	109.400000	C7-C8-H8	109.400000
C9-C8-H8	109.400000	O3-C9-C8	109.49(10)
O3-C9-C10	105.50(11)	C8-C9-C10	115.33(12)
O3-C9-H9	108.800000	C8-C9-H9	108.800000
C10-C9-H9	108.800000	C11-C10-C9	103.44(12)
C11-C10-H10A	111.100000	C9-C10-H10A	111.100000
C11-C10-H10B	111.100000	C9-C10-H10B	111.100000
H10A-C10-H10B	109.000000	C12-C11-C10	100.62(12)
C12-C11-H11A	111.600000	C10-C11-H11A	111.600000
C12-C11-H11B	111.600000	C10-C11-H11B	111.600000
H11A-C11-H11B	109.400000	O3-C12-C11	104.59(12)
O3-C12-H12A	110.800000	C11-C12-H12A	110.800000

O3-C12-H12B	110.800000	C11-C12-H12B	110.800000
H12A-C12-H12B	108.900000		

Table 7. Torsion angles (°) for JTM1728.

C7-O1-C1-C2	-175.09(11)	C7-O1-C1-C6	5.27(18)
O1-C1-C2-C3	178.95(11)	C6-C1-C2-C3	-1.39(19)
C1-C2-C3-C4	-0.27(19)	C1-C2-C3-Cl1	-179.96(10)
C2-C3-C4-F1	-179.33(12)	Cl1-C3-C4-F1	0.36(17)
C2-C3-C4-C5	1.3(2)	Cl1-C3-C4-C5	-178.98(10)
F1-C4-C5-C6	179.96(12)	C3-C4-C5-C6	-0.7(2)
C4-C5-C6-C1	-1.0(2)	O1-C1-C6-C5	-178.36(12)
C2-C1-C6-C5	2.0(2)	C1-O1-C7-C8	172.83(10)
O1-C7-C8-O2	179.24(10)	O1-C7-C8-C9	-59.29(13)
C12-O3-C9-C8	-120.45(12)	C12-O3-C9-C10	4.24(14)
O2-C8-C9-O3	56.78(14)	C7-C8-C9-O3	-60.51(14)
O2-C8-C9-C10	-61.99(15)	C7-C8-C9-C10	-179.27(11)
O3-C9-C10-C11	21.01(14)	C8-C9-C10-C11	141.97(12)
C9-C10-C11-C12	-36.61(15)	C9-O3-C12-C11	-28.16(15)

C10-C11-C12-O3	39.93(15)

Table 8. Anisotropic atomic displacement parameters (Å^2) for JTM1728.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl1	0.0422(2)	0.0298(2)	0.0535(2)	0.01125(15)	0.01379(16)	0.00312(14)
F1	0.0485(5)	0.0415(5)	0.0365(4)	0.0033(3)	0.0130(4)	0.0084(4)
O1	0.0386(5)	0.0247(5)	0.0399(5)	0.0030(4)	0.0112(4)	0.0028(4)
O2	0.0320(5)	0.0286(5)	0.0477(6)	0.0007(4)	0.0131(4)	0.0015(4)
O3	0.0331(5)	0.0346(5)	0.0345(5)	0.0004(4)	0.0049(4)	0.0102(4)
C1	0.0318(6)	0.0272(6)	0.0315(6)	0.0048(5)	0.0020(5)	0.0057(5)
C2	0.0341(6)	0.0237(6)	0.0371(7)	0.0035(5)	0.0022(5)	0.0050(5)
C3	0.0328(6)	0.0264(6)	0.0363(7)	0.0091(5)	0.0017(5)	0.0039(5)
C4	0.0374(7)	0.0339(7)	0.0305(6)	0.0047(5)	0.0042(5)	0.0080(5)
C5	0.0423(7)	0.0266(6)	0.0328(6)	0.0004(5)	0.0016(5)	0.0039(5)
C6	0.0362(7)	0.0252(6)	0.0343(6)	0.0038(5)	0.0020(5)	0.0012(5)
C7	0.0320(6)	0.0249(6)	0.0348(6)	0.0039(5)	0.0044(5)	0.0024(5)
C8	0.0319(6)	0.0238(6)	0.0384(7)	0.0033(5)	0.0057(5)	0.0055(5)
C9	0.0345(6)	0.0230(6)	0.0380(7)	0.0022(5)	0.0061(5)	0.0028(5)
C10	0.0465(8)	0.0340(7)	0.0428(8)	-0.0067(6)	0.0056(6)	0.0113(6)
C11	0.0468(8)	0.0384(8)	0.0363(7)	-0.0033(6)	0.0078(6)	0.0052(6)
C12	0.0523(9)	0.0374(8)	0.0354(7)	0.0035(6)	0.0051(6)	0.0112(6)

Table 9. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å^2) for JTM1728.

	x/a	y/b	z/c	U(eq)
H2A	-0.183(5)	0.152(2)	0.743(2)	0.060(6)
H2	0.7463	0.4292	0.4467	0.038000
H5	0.6435	0.0355	0.2414	0.042000
H6	0.3731	0.0701	0.3923	0.039000
H7A	0.3248	0.1153	0.5875	0.037000
H7B	0.0660	0.1493	0.5028	0.037000
H8	0.0077	0.2981	0.6570	0.038000
H9	0.4770	0.3529	0.7509	0.038000
H10A	0.2780	0.4279	0.9017	0.049000
H10B	0.0717	0.3001	0.9037	0.049000
H11A	0.6417	0.3583	1.0121	0.049000
H11B	0.3879	0.2942	1.0741	0.049000
H12A	0.6691	0.1508	0.9800	0.050000
H12B	0.3399	0.1114	0.9515	0.050000

Table 10. Hydrogen bond distances (Å) and angles (°) for JTM1728.

	Donor-H	Acceptor-H	Donor-Acceptor	Angle
O2-H2A···O3#1	0.81(2)	1.94(2)	2.7464(14)	175.(2)
C5-H5···O3#5	0.950000	2.500000	3.4078(17)	160.900000
C6-H6···O2#4	0.950000	2.600000	3.3789(16)	139.500000
C9-H9···Cl1#6	1.000000	2.830000	3.8118(14)	166.400000
C11-H11B···F1#2	0.990000	2.470000	3.3217(18)	144.000000
C12-H12A···F1#3	0.990000	2.470000	3.3007(18)	141.700000

Symmetry transformations used to generate equivalent atoms:

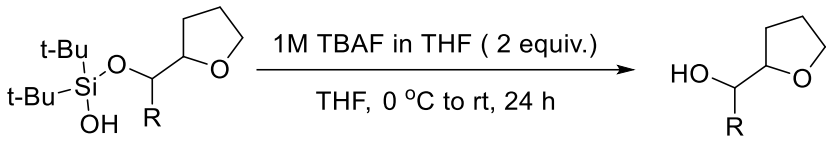
#1 x-1, y, z
#2 x-1, y, z+1
#3 x, y, z+1
#4 -x, -y, -z+1
#5 -x+1, -y, -z+1
#6 -x+2, -y+1, -z+1

X. Structural Reasoning

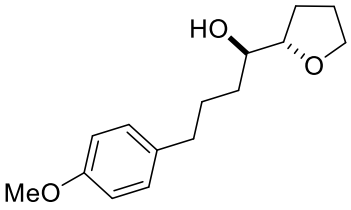
1. For silanoxy-tetrahydrofurans

We were unable to grow a crystal structure of the silanoxy-products; the presence of the silyl group makes most compounds oils or semi-solids. We thus removed the silyl group from two of the products and grew crystals of the corresponding alcohols (See X-ray crystallography section). The other product identities were assigned by analogy to these.

General Procedure D



A 25 mL round-bottom flask was charged with a stir-bar, silanoxy-tetrahydrofuran substrate (0.2 mmol), and anhydrous THF (2 mL). The reaction flask was cooled to 0 °C using an ice-water bath. Subsequently, TBAF (1M solution in THF, 0.4 mL, 0.4 mmol, 2 equiv.) was added dropwise. The reaction mixture was allowed to warm to room temperature over a period of 24 h. After this time, the reaction was diluted with EtOAc (1 mL), transferred to a separatory funnel, and washed once with saturated aqueous NH_4Cl solution (1 mL). The organic layer was separated, and the aqueous layer was extracted with two additional portions of EtOAc (2 x 5 mL). The combined organic layers were washed with brine solution (2 mL), dried over anhydrous $MgSO_4$, and the solvent was removed *in vacuo*. The resulting residue was purified through silica gel column chromatography (hexane/ethyl acetate = 30:70) to afford the corresponding product.



(*R**)-4-(4-methoxyphenyl)-1-((*S**)-tetrahydrofuran-2-yl)butan-1-ol

Compound S74: Synthesized using General Procedure D on a 0.2 mmol scale; Purified using 30% EtOAc/hexanes on silica gel; single diastereomer; (white solid, 30 mg, 0.120 mmol, 60% yield).

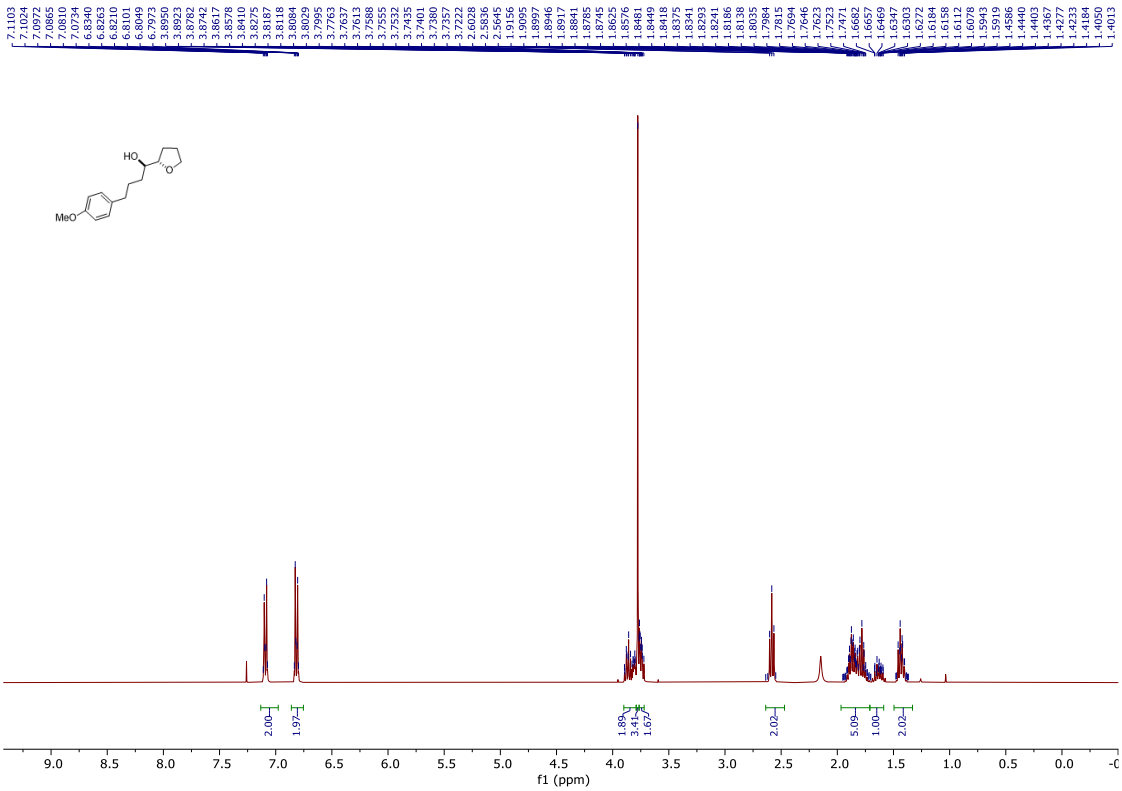
^{1}H NMR (400 MHz, $CDCl_3$) 7.13 – 7.05 (m, 2H), 6.86 – 6.77 (m, 2H), 3.89 – 3.80 (m, 2H), 3.78 (s, 3H), 3.77 – 3.71 (m, 2H), 2.58 (t, J = 7.7 Hz, 2H), 1.95 – 1.69 (m, 5H), 1.69 – 1.58 (m, 1H), 1.50 – 1.36 (m, 2H).

^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 157.8, 134.5, 129.4, 113.8, 82.3, 71.9, 68.6, 55.3, 35.0, 32.4, 28.1, 26.2, 24.6.

IR ν 3435, 2938, 2864, 1611, 1512, 1464, 1246 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{15}H_{22}O_3Na^+$ 273.1467, found 273.1454 (4.8 ppm error).

Compound S74 ($CDCl_3$, ^{1}H NMR: 400 MHz, ^{13}C{^{1}H} NMR: 101 MHz)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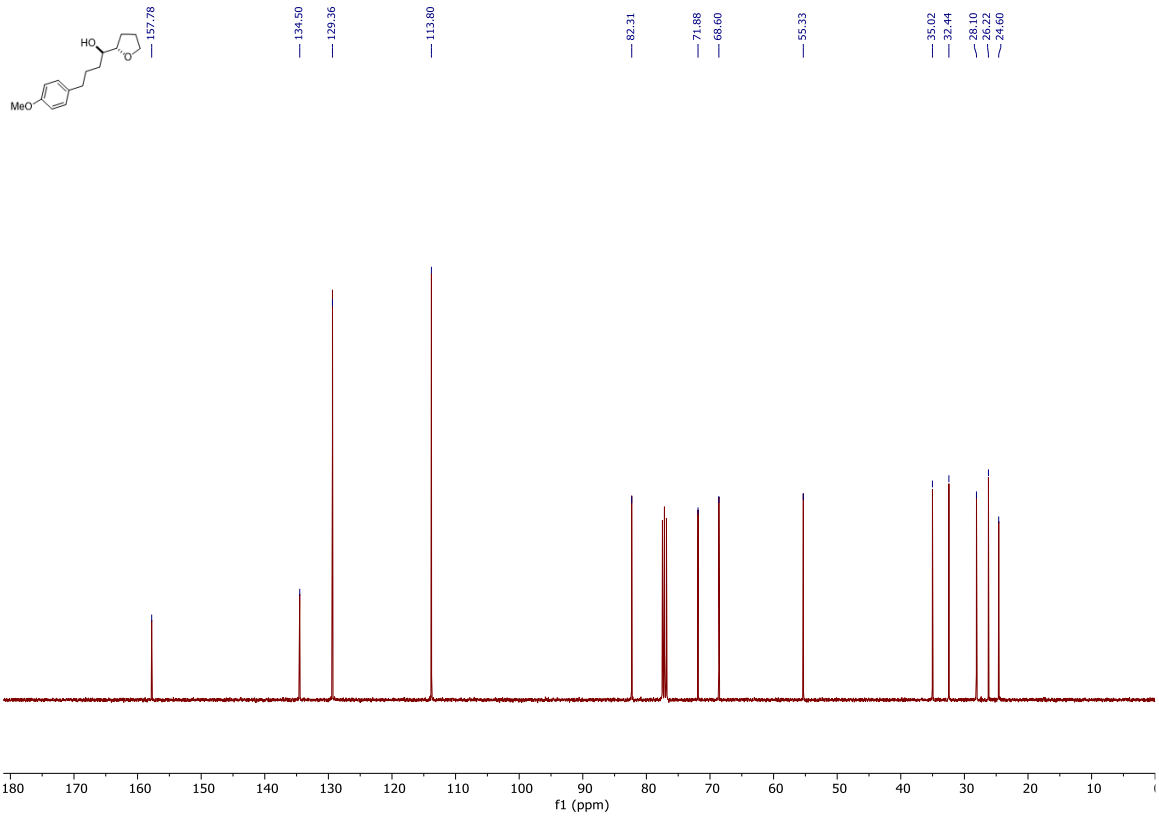
2.02
5.09
1.00
2.02
f1 (ppm)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MeO
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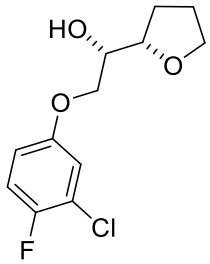
26.22
24.60
f1 (ppm)



(*S**)-2-(3-chloro-4-fluorophenoxy)-1-((*S**)-tetrahydrofuran-2-yl)ethan-1-ol

Compound S75: Synthesized using General Procedure D on a 0.2 mmol scale; Purified using 30% ethyl acetate in hexanes on silica gel; (White solid, 36 mg, 0.14 mmol, 70% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 (t, J = 8.8 Hz, 1H), 6.95 (dd, J = 5.9, 3.0 Hz, 1H), 6.77 (dt, J = 9.1, 3.4 Hz, 1H), 4.04 – 3.94 (m, 3H), 3.92 – 3.78 (m, 3H), 2.02 – 1.90 (m, 3H), 1.90 – 1.79 (m, 1H).

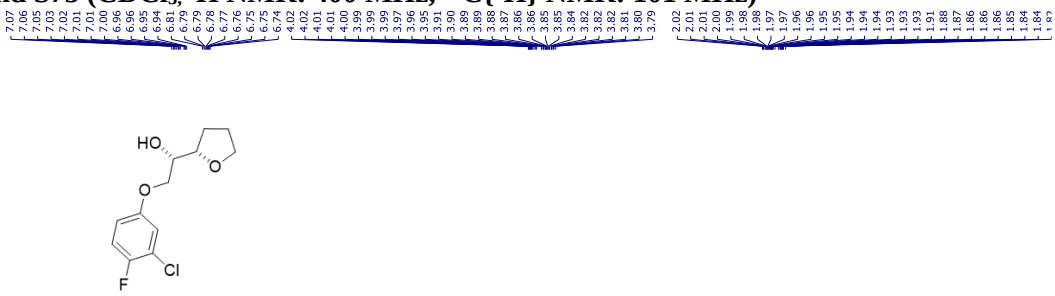
^{13}C{^{1}H} (101 MHz, $CDCl_3$) δ 155.1 (d, J = 2.6 Hz), 153.1 (d, J = 241.2 Hz), 121.2 (d, J = 19.1 Hz), 116.9 (d, J = 22.3 Hz), 116.5, 114.3 (d, J = 6.6 Hz), 79.0, 71.9, 70.7, 68.7, 27.9, 26.3.

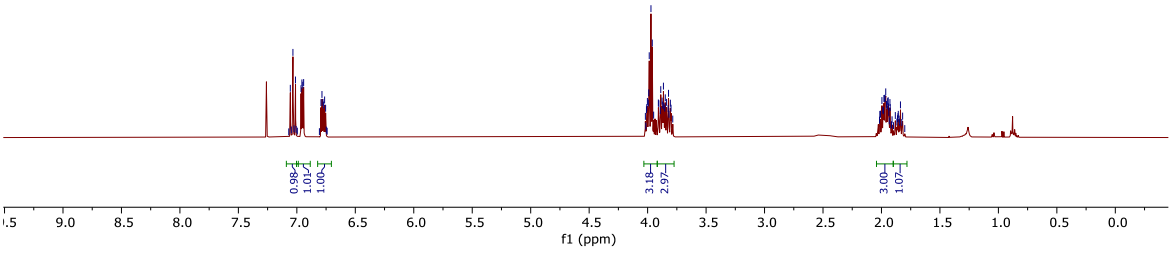
^{19}F NMR (377 MHz, $CDCl_3$) δ -126.7 – -126.8 (m).

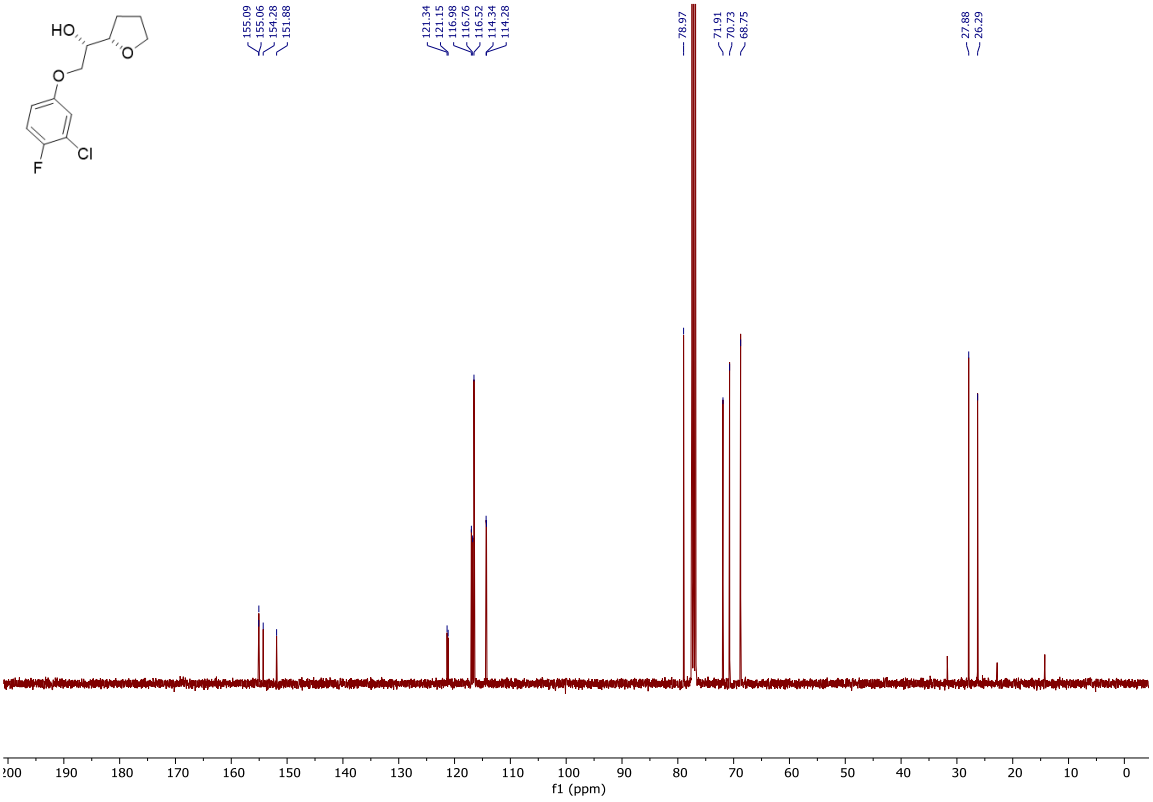
IR ν 3438, 2940, 2860, 1600, 1502, 1450, 1246 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_{12}H_{14}ClFO_3Na^+$ 283.0513, found 283.0522 (3.2 ppm error).

Compound S75 ($CDCl_3$, ^{1}H NMR: 400 MHz, ^{13}C{^{1}H} NMR: 101 MHz)



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O
Cl
F
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154.28
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121.34
121.15
116.98
116.76
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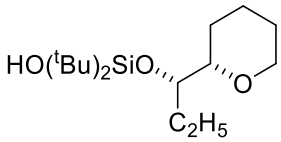
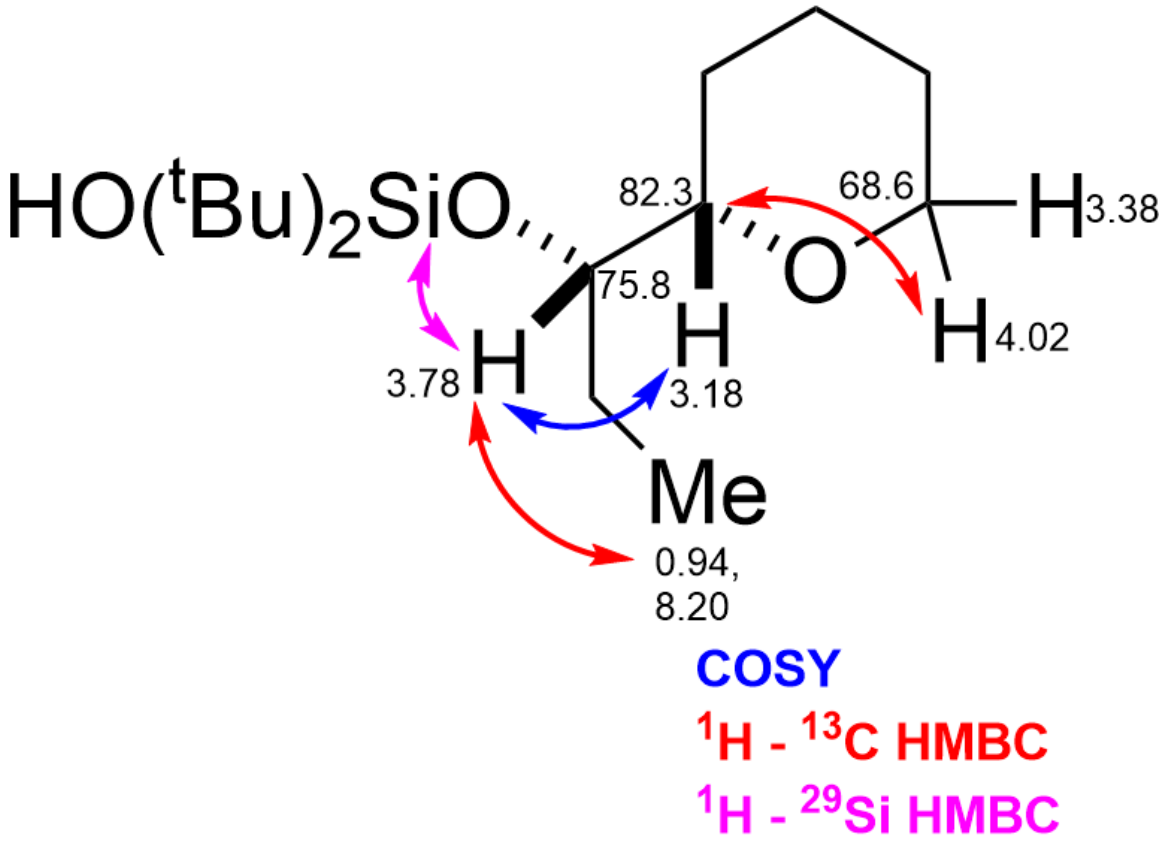
114.28
78.97
71.91
70.73
68.75
27.88
26.29
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

For silanoxy-tetrahydropyrans

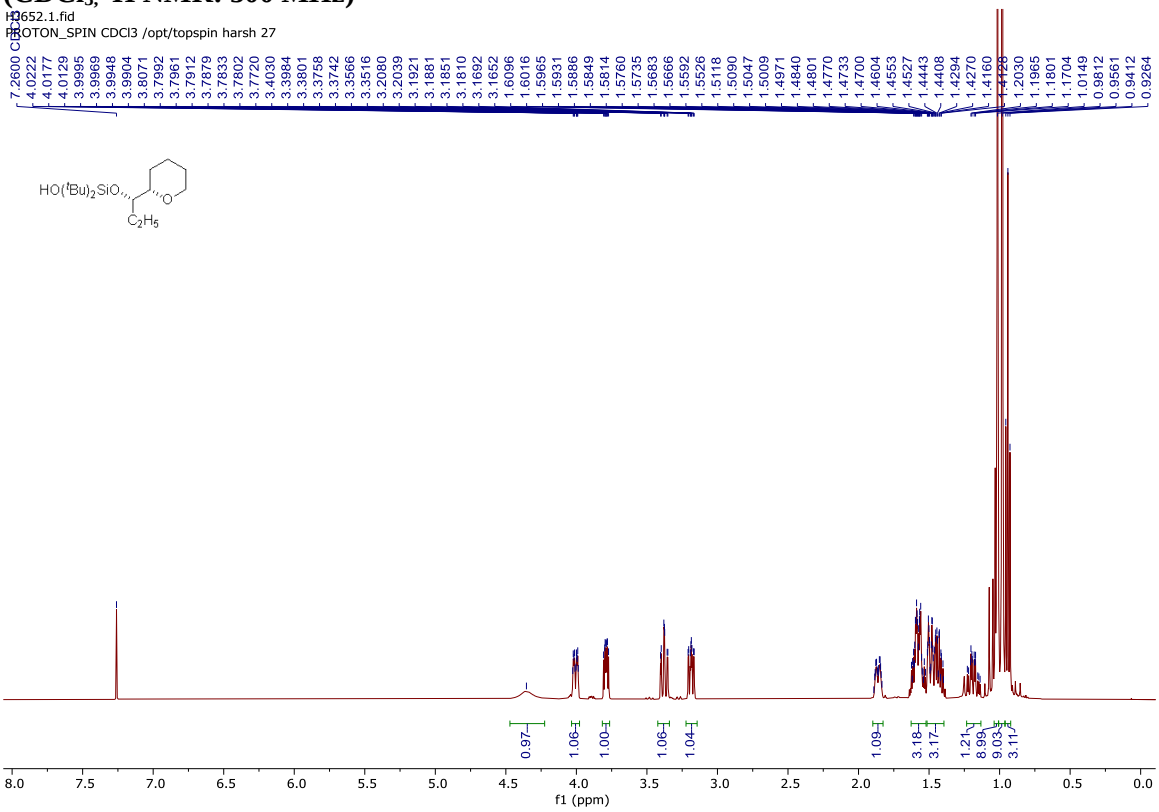
We have acquired 2-D NMR data for **Compound 64**. We have assigned other product identities by analogy to this.



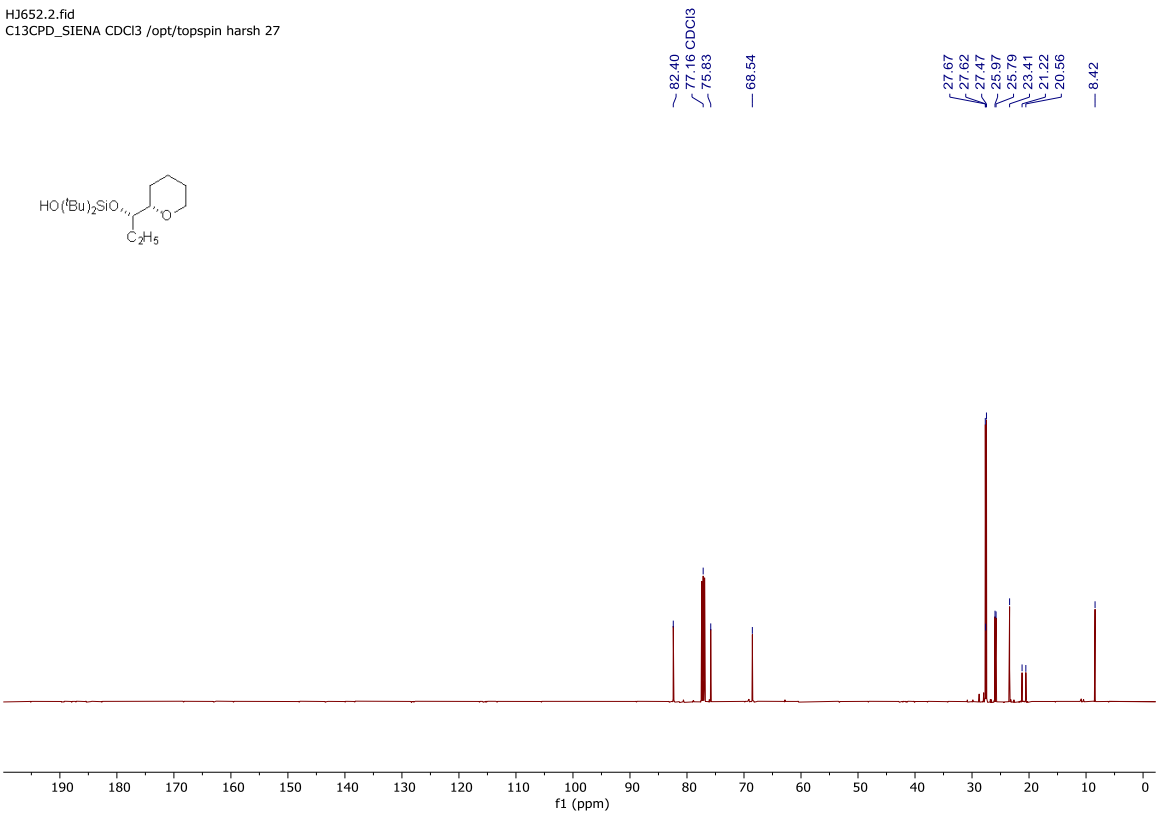
Compound 64



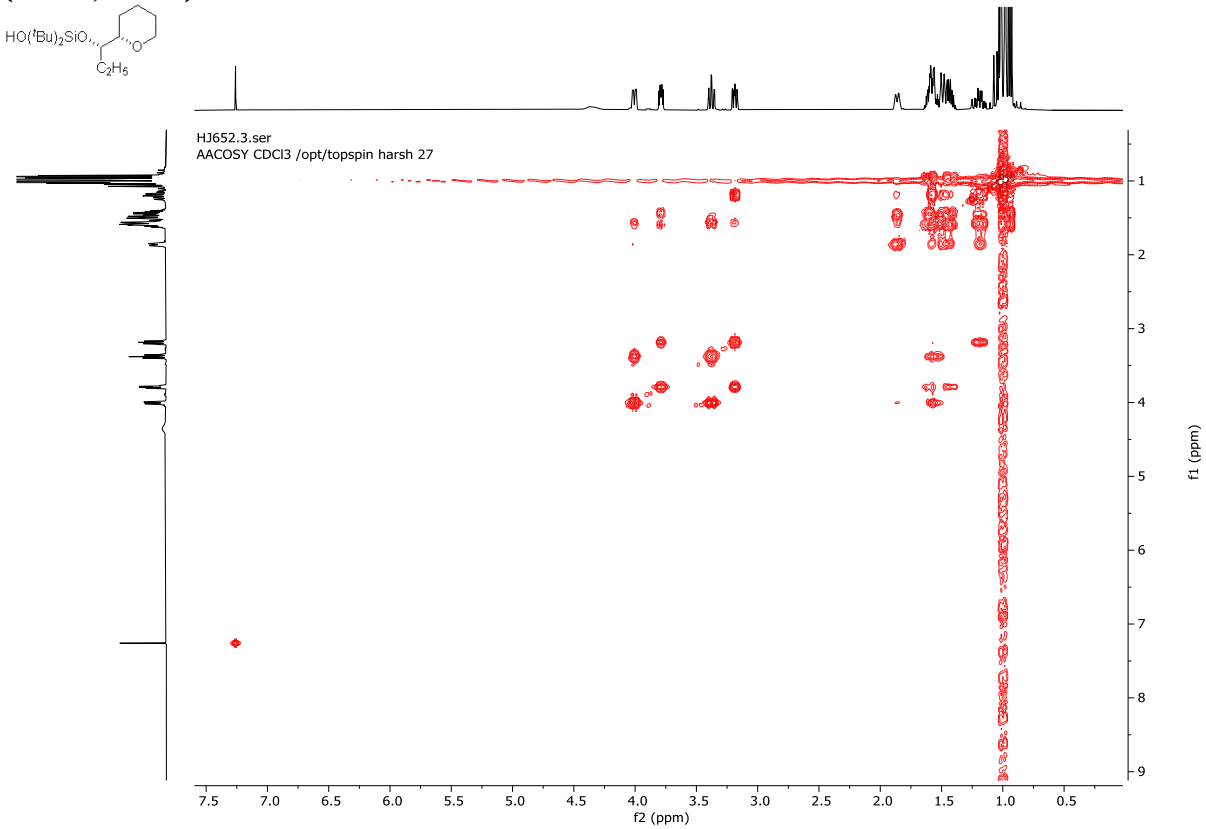
($CDCl_3$, ^1H NMR: 500 MHz)



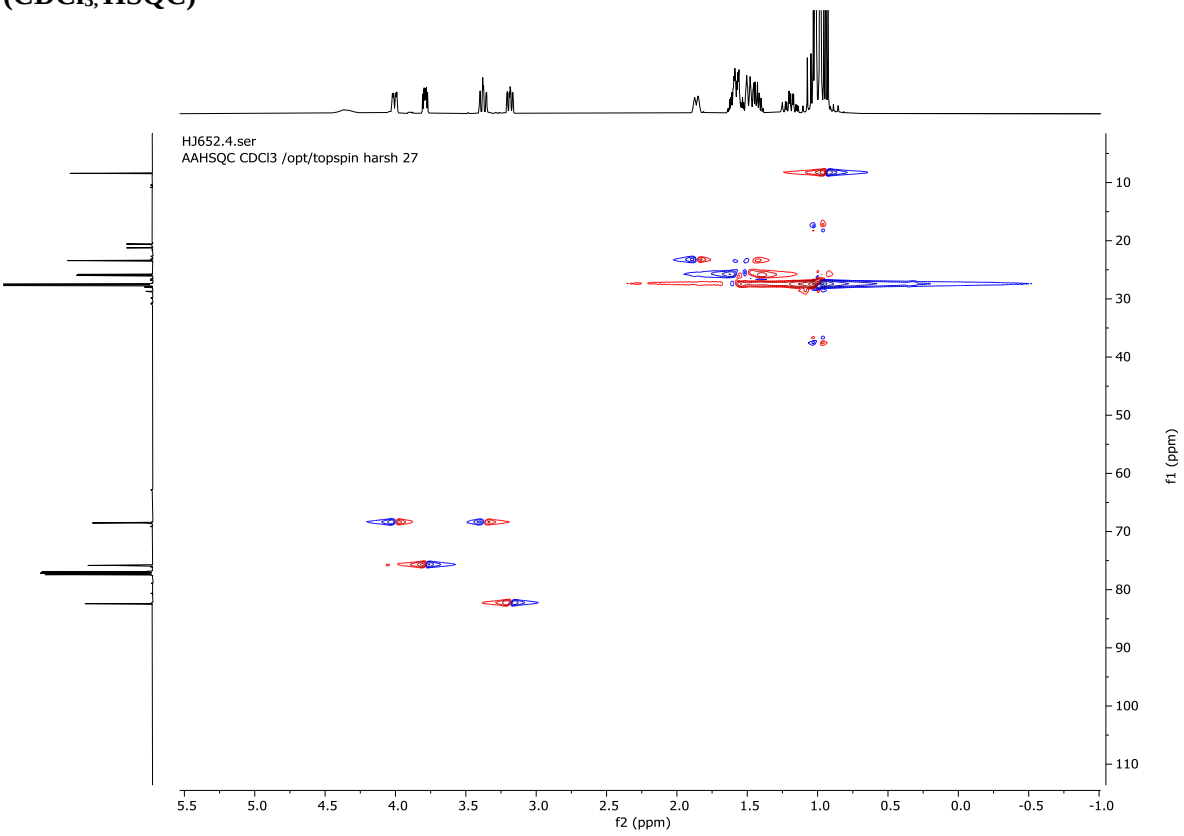
($CDCl_3$, $^{13}C\{^1H\}$ NMR: 126 MHz)



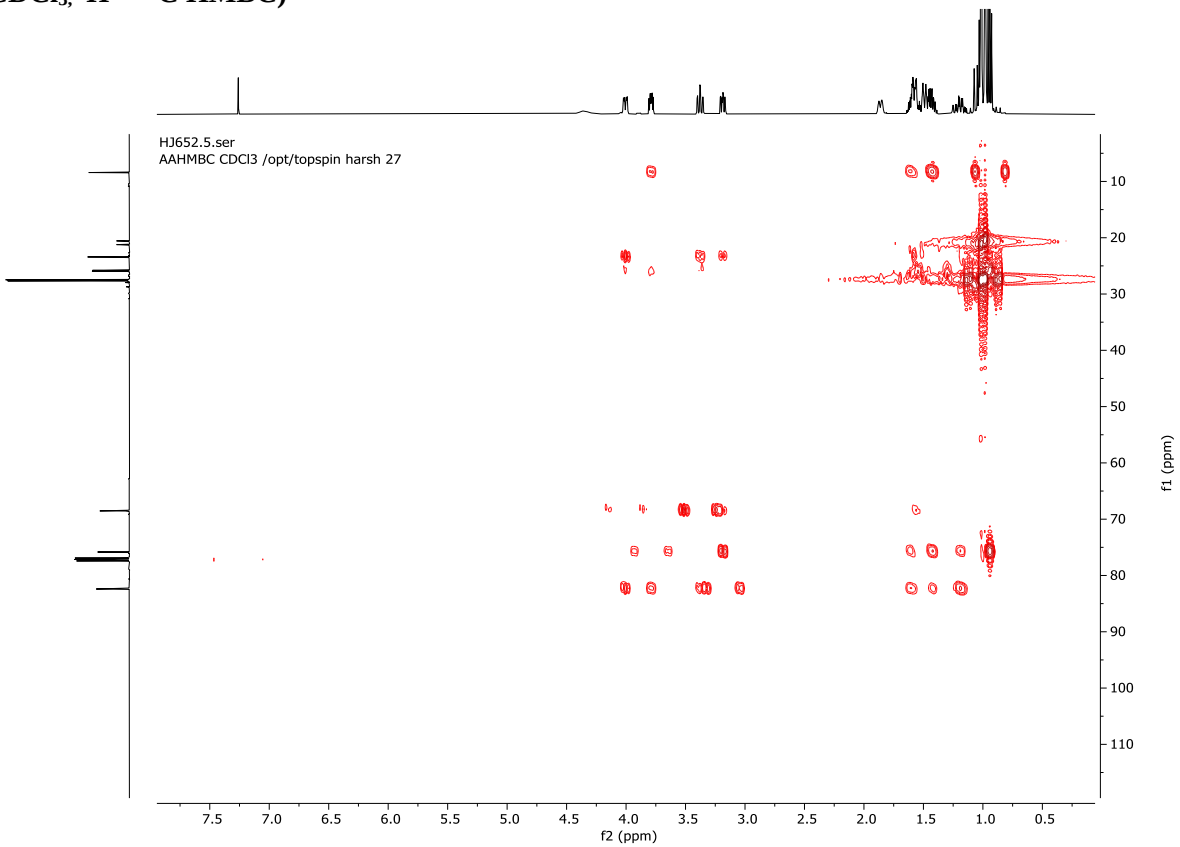
($CDCl_3$, COSY)



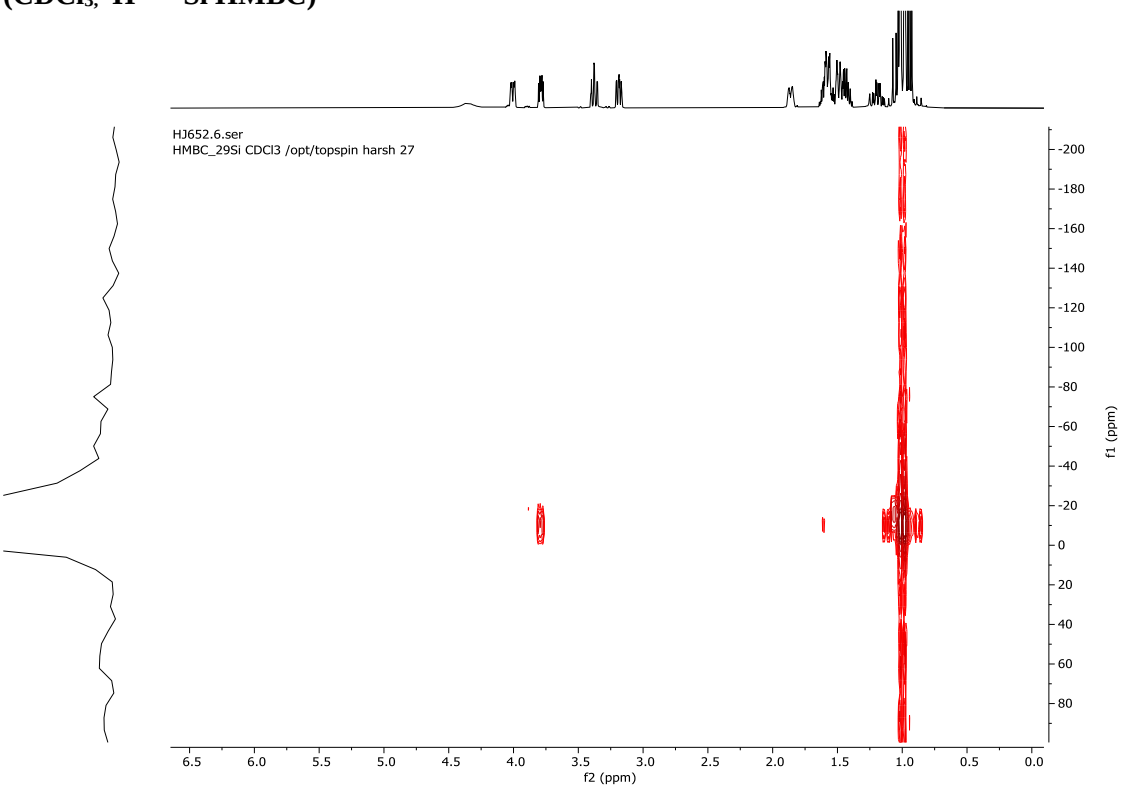
($CDCl_3$, HSQC)



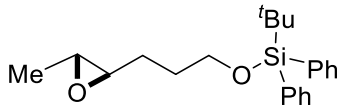
($CDCl_3$, ^{1}H – ^{13}C HMBC)



($CDCl_3$, ^{1}H – ^{29}Si HMBC)



XI. Control Experiments



tert-butyl(3-((2*R**,3*R**)-3-methyloxiran-2-yl)propoxy)diphenylsilane

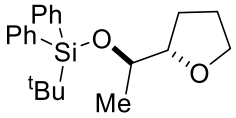
Compound 75: Synthesized using General Procedure B on a 1.8 mmol scale; Purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 407 mg, 1.15 mmol, 64% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.58 (m, 4H), 7.50 – 7.29 (m, 6H), 3.76 – 3.62 (m, 2H), 2.73 (qd, *J* = 5.2, 2.3 Hz, 1H), 2.66 – 2.56 (m, 1H), 1.79 – 1.55 (m, 4H), 1.27 (d, *J* = 5.2 Hz, 3H), 1.06 (s, 9H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 135.7, 134.0, 129.7, 127.8, 63.5, 59.6, 54.8, 29.1, 28.7, 27.0, 19.4, 17.8.

IR ν 3060, 2958, 2930, 2857, 1473, 1112, 701, 504 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{22}H_{30}O_2SiNa^+$ 377.1913, Found 377.1941 (7.4 ppm error).



tert-butyldiphenyl((*R**)-1-((*S**)-tetrahydrofuran-2-yl)ethoxy)silane

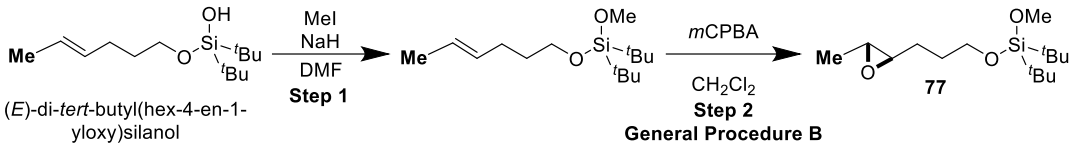
Compound 76: Synthesized using General Procedure C on a 0.2 mmol scale.; Purified using a gradient of 1-4% diethyl ether in hexane on silica gel; single diastereomer; (colorless oil, 21.2 mg, 0.060 mmol, 30% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 – 7.67 (m, 4H), 7.44 – 7.33 (m, 6H), 3.88 (qd, *J* = 6.2, 4.7 Hz, 1H), 3.85 – 3.78 (m, 1H), 3.77 – 3.66 (m, 2H), 1.96 – 1.75 (m, 4H), 1.07 (s, 9H), 1.00 (d, *J* = 6.3 Hz, 3H).

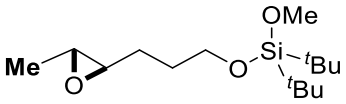
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 136.2, 136.1, 135.0, 134.2, 129.6 (2C), 127.6, 127.5, 83.8, 71.6, 68.6, 27.2, 27.0, 26.1, 20.6, 19.5.

IR ν 3070, 3050, 2930, 2857, 1473, 1112, 1080, 945, 822, 738, 701, 610, 509 cm^{-1}.

HRMS (ESI) m/z: [M + Na^+] calculated for $C_{22}H_{30}O_2SiNa^+$ 377.1913, Found 377.1941 (error 7.4 ppm).



Sodium hydride (50.0 mg, 2.0 mmol, 2.0 equiv) was added to the ice-cold solution of (E)-di-tert-butyl(hex-4-en-1-yloxy)silanol (258 mg, 1.0 mmol, 1.0 equiv) in dimethylformamide (2.0 mL). The mixture was stirred for 20 minutes at the same temperature then MeI (426 mg, 3.0 mmol, 3.0 equiv) was slowly added to it. The reaction mixture was allowed to warm to room temperature and stirred for 16 h. Next, the reaction was quenched with water and diluted with diethyl ether (15 mL). The organic layer was separated, washed with water (10 mL x 2), and dried over anhydrous Na_2SO_4. The solvent was removed *in vacuo* and the crude product was directly subjected to epoxidation following General Procedure B.



di-*tert*-butyl(methoxy)(3-((2*R**,3*R**)-3-methyloxiran-2-yl)propoxy)silane

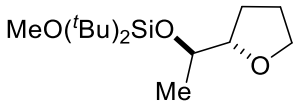
Compound 77: The title compound was purified using a gradient of 0-3% acetone in CH_2Cl_2 on silica gel; single diastereomer; (colorless oil, 224 mg, 0.777 mmol, 78% yield over two steps).

^{1}H NMR (600 MHz, $CDCl_3$) δ 3.91 – 3.82 (m, 2H), 3.63 (s, 3H), 2.77 (qd, *J* = 5.2, 2.2 Hz, 1H), 2.68 (ddd, *J* = 6.5, 4.8, 2.2 Hz, 1H), 1.77 – 1.64 (m, 3H), 1.62 – 1.57 (m, 1H), 1.30 (d, *J* = 5.2 Hz, 3H), 1.00 (s, 18H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 63.5, 59.7, 54.8, 52.3, 29.4, 28.7, 28.0, 21.3, 17.8.

IR ν 2965, 2934, 2860, 1473, 1105, 827, 651 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{15}H_{32}O_3SiNa^+$ 311.2018, Found 311.2042 (7.7 ppm error).



di-*tert*-butyl(methoxy)((*R**)-1-((*S**)-tetrahydrofuran-2-yl)ethoxy)silane

Compound 78: Synthesized using General Procedure C on a 0.2 mmol scale.; Purified using a gradient of 1-4% diethyl ether in hexane on silica gel; single diastereomer; (colorless oil, 34.6 mg, 0.120 mmol, 60% yield).

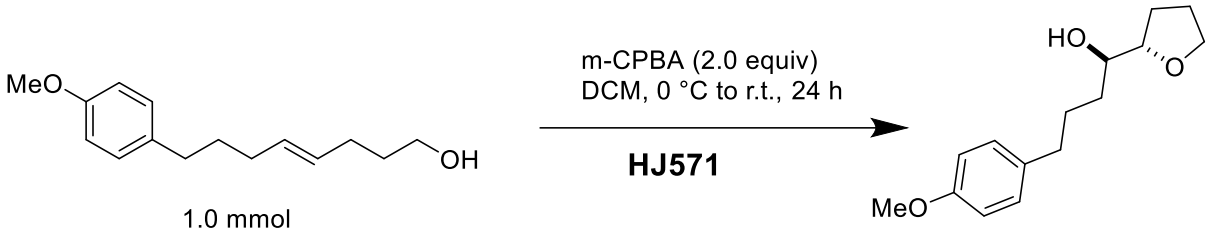
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.15 (qd, *J* = 6.3, 4.6 Hz, 1H), 3.88 – 3.82 (m, 1H), 3.78 – 3.69 (m, 2H), 3.66 (s, 3H), 1.94 – 1.80 (m, 4H), 1.23 (d, *J* = 6.2 Hz, 3H), 1.03 (s, 9H), 1.01 (s, 9H).

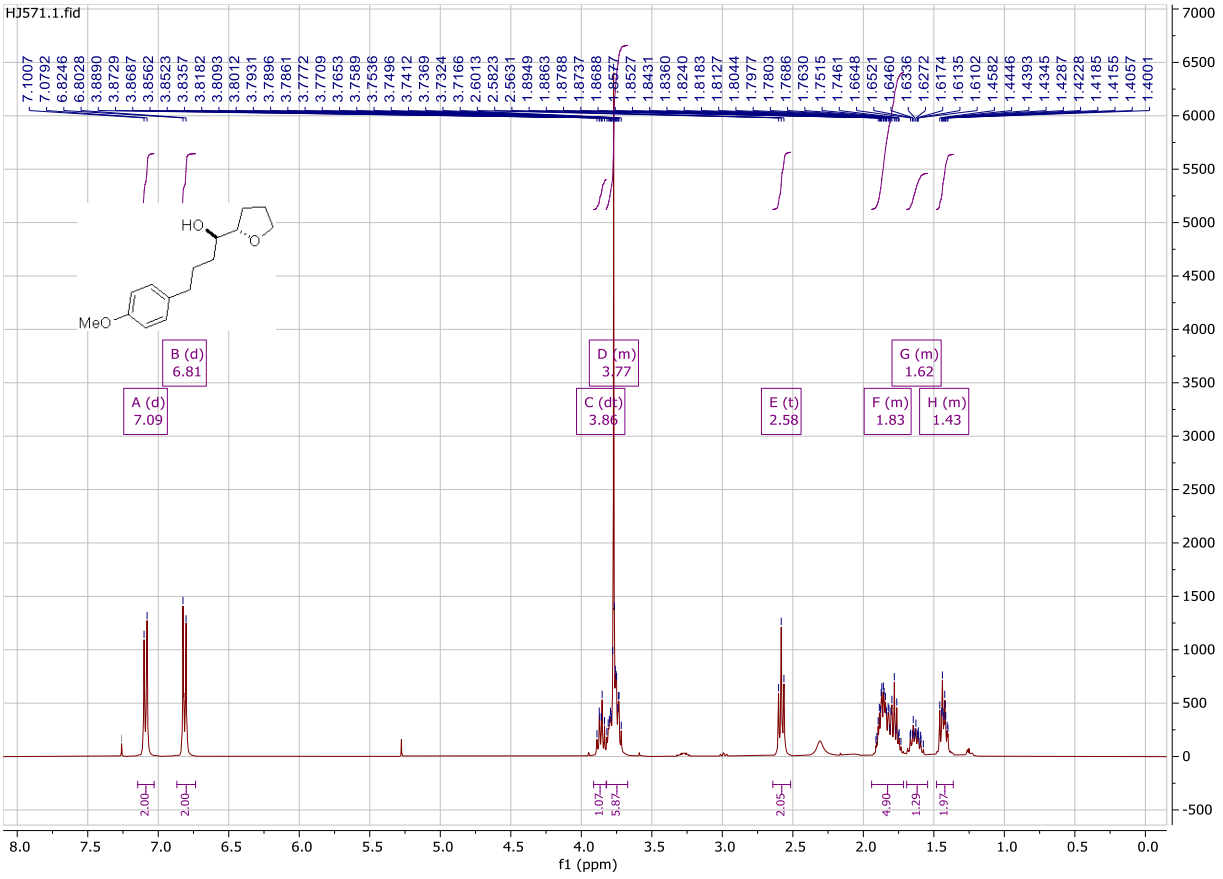
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 84.1, 71.1, 68.6, 52.4, 28.1, 26.6, 26.1, 21.5, 21.2, 20.9.

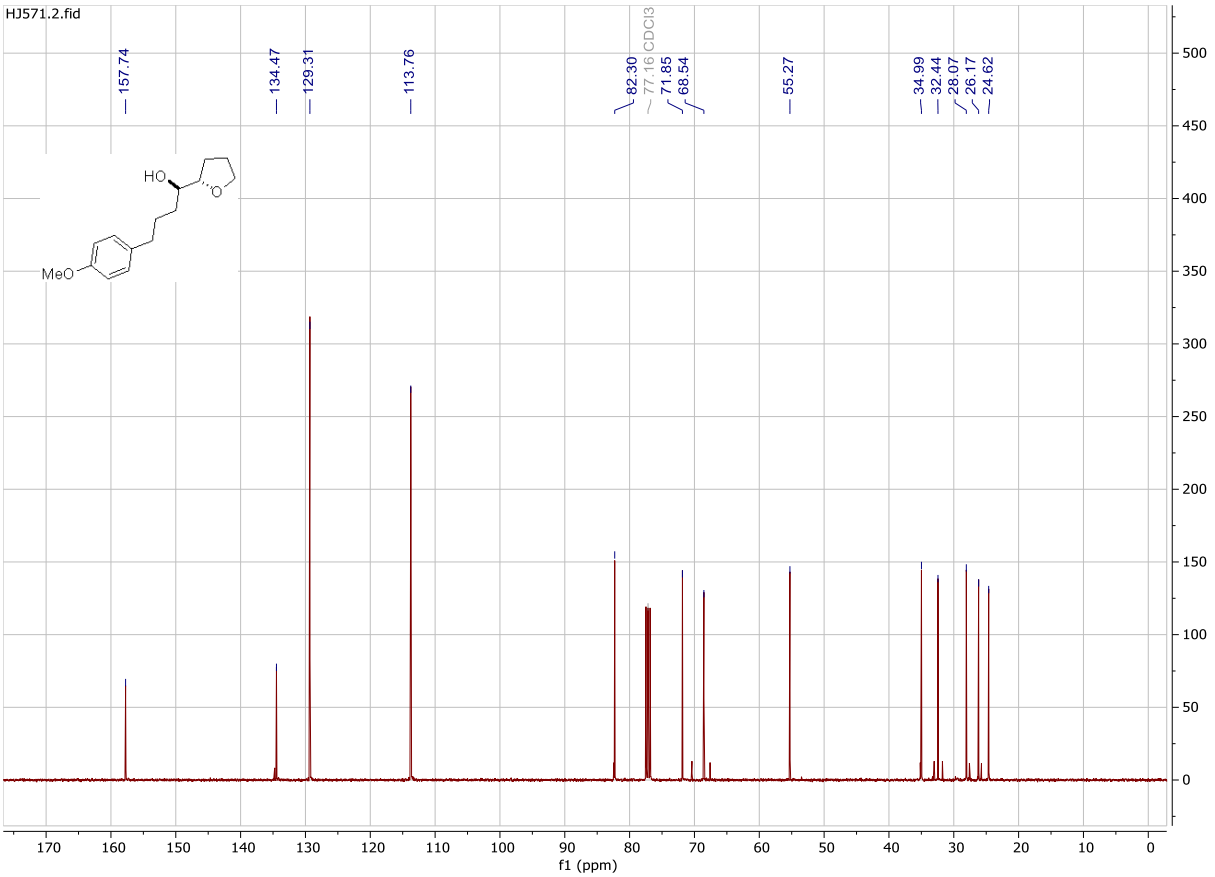
IR ν 2970, 2888, 2859, 1473, 1088, 827, 650 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{15}H_{32}O_3SiNa^+$ 311.2018, Found 311.2036 (5.8 ppm error).

We have also found that one-pot epoxidation-cyclization reactions of alkenyl alcohols are possible using *m*CPBA, but the mass balance and product yields of these reactions are generally poor. For a representative example:

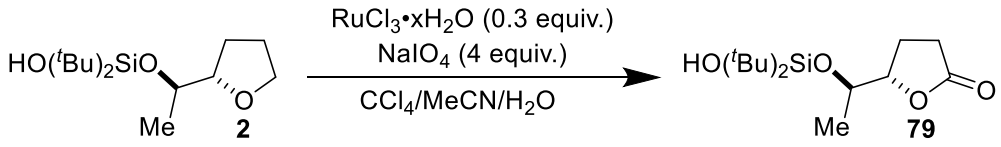




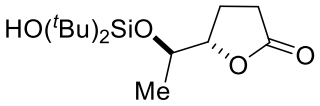


XII. Natural Products

For (±)-Solerone



An oven-dried 20 mL microwave vial was charged with a stir bar, silanoxy-tetrahydrofuran substrate **2** (548.4 mg, 2.0 mmol, 1.0 equiv), $NaIO_4$ (1.72 g, 8.0 mmol, 4.0 equiv) and a solvent mixture of CCl_4 (6.0 mL), MeCN (5.0 mL), and H_2O (4.0 mL) (Final concentration: 0.13 M). The resulting heterogeneous mixture was stirred vigorously at room temperature for 5 minutes, and $RuCl_3$•xH_2O (125.0 mg, 0.6 mmol, 0.3 equiv) was added to it in one portion. Upon stirring for 1 hour at room temperature, the reaction mixture was treated with 2-propanol (4.0 mL) and stirred vigorously for an additional 1 h. The mixture was transferred to a separatory funnel and extracted with CH_2Cl_2. The organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The resulting residue was purified by chromatography on silica gel to obtain the desired product using a gradient of 2.0 to 6.0% acetone in CH_2Cl_2 on silica gel).



(*S**)-5-((*R**)-1-((di-*tert*-butyl(hydroxy)silyl)oxy)ethyl)dihydrofuran-2(3*H*)-one

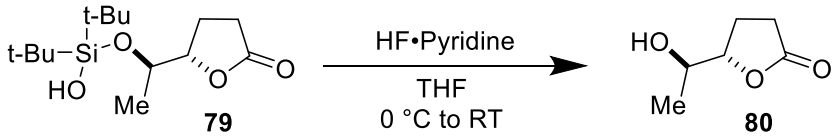
Compound 79: single diastereomer; (colorless oil, 263.2 mg, 0.912 mmol, 46% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.44 (qd, *J* = 6.4, 2.8 Hz, 1H), 4.37 (td, *J* = 7.3, 2.8 Hz, 1H), 2.83 (s, 1H), 2.60 – 2.44 (m, 2H), 2.33 (dddd, *J* = 12.6, 10.2, 8.5, 7.2 Hz, 1H), 2.12 (dddd, *J* = 12.9, 9.4, 7.5, 5.6 Hz, 1H), 1.19 (d, *J* = 6.5 Hz, 3H), 1.00 (s, 9H), 0.97 (s, 9H).

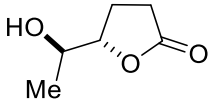
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 177.7, 84.0, 68.3, 28.7, 27.63, 27.58, 20.7, 20.6, 20.4, 19.1.

IR ν 3485, 2934, 2857, 1762, 1473, 1200, 1154, 1084, 1012, 826, 648 cm^{-1}.

HRMS (ESI) m/z = [M + Na^+] calculated for $C_{14}H_{28}O_4SiNa^+$ 311.1655, Found 311.1664 (2.9 ppm error).



HF-pyridine (~70% HF-30% pyridine, 0.240 mL of solution, 0.264 g of solution, 0.185 g HF, 9.2 mmol, 32 equiv.) was carefully added to an ice-cold solution of compound **79** (84.0 mg, 0.29 mmol, 1 equiv.) in THF (3 mL,~0.1 M). The homogenous mixture was allowed to warm to room temperature and stirred for 36 h. After completion, the reaction was quenched with saturated aqueous $NaHCO_3$ solution, transferred to a separatory funnel, and extracted with ethyl acetate (15 mL x 3). The combined organic layers were dried with anhydrous Na_2SO_4 and concentrated *in vacuo*. The residue was purified using a gradient of 15 to 50% EtOAc in hexane on silica gel to give **80** (colorless oil, 27.4 mg, 0.21 mmol, 73% yield).



(*S**)-5-((*R**)-1-hydroxyethyl)dihydrofuran-2(3*H*)-one

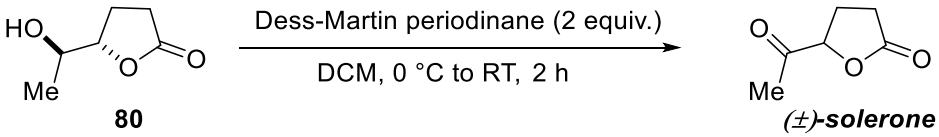
Compound 80:

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.40 (td, *J* = 7.3, 3.3 Hz, 1H), 4.11 (qd, *J* = 6.6, 3.3 Hz, 1H), 2.65 – 2.45 (m, 2H), 2.34 – 2.10 (m, 3H), 1.18 (d, *J* = 6.6 Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 177.7, 83.7, 67.5, 28.8, 21.1, 17.8.

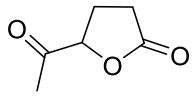
IR ν 3435, 2976, 2936, 1767, 1460, 1197, 916 cm^{-1}.

HRMS (ESI) m/z = [M + H^+] Calcd for $C_6H_{11}O_3^+$ 131.0703, found 131.0693 (7.6 ppm error).



80 ***(±)-solerone***

A 20 mL round-bottom flask was charged with a stir-bar, **80** (0.049 g, 0.38 mmol, 1 equiv.), and anhydrous DCM (5 mL). The reaction flask was cooled to 0 °C using an ice-water bath. Subsequently, Dess-Martin periodinane (326 mg, 0.769 mmol, 2 equiv.) was added in a single portion. The reaction mixture was allowed to warm to room temperature over a period of 2 hours. After this time, the reaction was quenched with saturated aqueous sodium thiosulfate solution, and this mixture was stirred for 1 hour. Following this time, the reaction mixture was transferred to a separatory funnel and extracted with DCM (2 x 10 mL). The combined organic layers were dried over anhydrous $MgSO_4$, and the solvent was removed *in vacuo*. The resulting residue was purified through silica gel column chromatography (gradient of 0 to 50% ethyl acetate/hexanes) to give **(±)-solerone** as a colorless oil (40 mg, 0.31 mmol, 82% yield).



5-acetyldihydrofuran-2(3*H*)-one

(±)-Solerone:

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.87 – 4.79 (m, 1H), 2.62 – 2.44 (m, 3H), 2.29 (s, 3H), 2.28 – 2.21 (m, 1H).

$^{13}C\{^1H\}$ (101 MHz, $CDCl_3$) δ 205.5, 176.0, 82.0, 27.4, 26.3, 24.5.

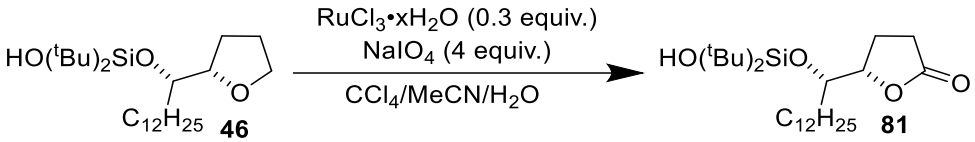
IR ν 2961, 1789, 1715, 1361, 1100, 850 cm^{-1}.

HRMS (ESI-TOF) m/z = [M + Na^+] Calcd $C_6H_8O_3Na^+$ 151.0371, found 151.0378 (4.6 ppm error).

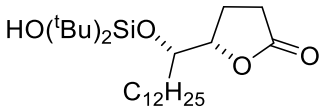
<u>Comparison of shifts with what is reported in *Org. Biomol. Chem*. 2014, *12*, 5601 – 5610.</u>

^{1}H NMR (400 MHz, $CDCl_3$)	Lit. ^{1}H NMR (500 MHz, $CDCl_3$)	^{13}C NMR (101 MHz)	Lit. ^{13}C NMR (125 MHz)
4.87 – 4.79 (m, 1H)	4.80 (m, 1H)	205.5	205.2
2.62 – 2.44 (m, 3H)	2.54–2.43 (m, 3H)	176.0	175.9
2.29 (s, 3H)	2.23 (s, 3H)	82.0	81.8
2.28 – 2.21 (m, 1H)	2.22–2.15 (m, 1H)	27.4	27.1
		26.3	26.0
		24.5	24.3

<u>For (±)-muricatacin</u>



An oven-dried 20 mL microwave vial was charged with a stir bar, silanoxy-tetrahydrofuran substrate **46** (300.0 mg, 0.7 mmol, 1.0 equiv), $NaIO_4$ (600.0 mg, 2.8 mmol, 4.0 equiv), and a mixed solvent system of CCl_4 (2.2 mL)/MeCN (1.8 mL)/H_2O (1.5 mL) (Final concentration: 0.13 M). The resulting heterogeneous mixture was stirred vigorously at room temperature for 5 minutes, and $RuCl_3$•xH_2O (44.0 mg, 0.21 mmol, 0.3 equiv) was added to it in one portion. Upon stirring for 1 hour at room temperature, the reaction mixture was treated with 2-propanol (1.5 mL) and stirred vigorously for an additional 1 h. The mixture was transferred to a separatory funnel and extracted with CH_2Cl_2. The organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The resulting residue was purified by chromatography on silica gel to obtain the desired product using a gradient of 2.0 to 6.0% acetone in CH_2Cl_2 on silica gel.



(*S**)-5-((*S**)-1-((di-*tert*-butyl(hydroxy)silyl)oxy)tridecyl)dihydrofuran-2(3*H*)-one

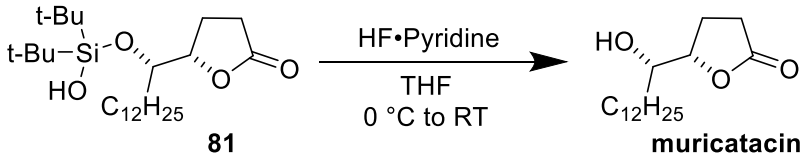
Compound 81: single diastereomer; (colorless oil, 152.5 mg, 0.346 mmol, 49% yield)

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.47 (dt, *J* = 8.3, 6.7 Hz, 1H), 4.01 (q, *J* = 5.5 Hz, 1H), 2.67 – 2.35 (m, 3H), 2.28 – 2.17 (m, 1H), 2.08 – 1.93 (m, 1H), 1.68 – 1.57 (m, 1H), 1.56 – 1.34 (m, 3H), 1.31 – 1.22 (m, 18H), 1.02 (s, 9H), 1.01 (s, 9H), 0.87 (t, *J* = 6.7 Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 176.7, 83.1, 75.0, 32.8, 32.1, 30.0, 29.8 (3C), 29.7, 29.6, 29.5, 29.0, 27.7 (2C), 24.5, 24.3, 22.8, 20.8, 20.7, 14.3.

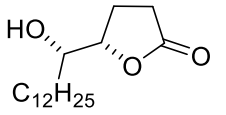
IR ν 3485, 2927, 2856, 1765, 1468, 1191, 1136, 1113, 1012, 827, 646 cm^{-1}.

HRMS (ESI) m/z: [M + Na^+] calculated for $C_{25}H_{50}O_4SiNa^+$ 465.3376, Found 465.3396 (4.3 ppm error).



HF-pyridine (~70% HF-30% pyridine, 0.290 mL, 0.319 g, 15.9 mmol, 46 equiv.) was carefully added to an ice-cold solution of **81** (153 mg, 0.346 mmol, 1 equiv.) in THF (3.5 mL,~0.1 M). The homogenous mixture was allowed to warm to room temperature and stirred for 72 h. After completion, the reaction was quenched with saturated aqueous $NaHCO_3$ solution, transferred to a separatory funnel, and extracted with

ethyl acetate (20 mL x 3). The combined organic layers were concentrated *in vacuo* after drying over anhydrous Na_2SO_4. The residue was purified using a gradient of 15 to 50% EtOAc in hexane on silica gel to give **muricatacin** (white solid, 90.0 mg, 0.316 mmol, 91% yield).



muricatacin

Muricatacin:

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.41 (td, *J* = 7.4, 4.6 Hz, 1H), 3.64 – 3.49 (m, 1H), 2.68 – 2.46 (m, 2H), 2.25 (dddd, *J* = 12.6, 9.6, 7.3, 5.0 Hz, 1H), 2.18 – 2.04 (m, 1H), 1.89 (s, 1H), 1.61 – 1.43 (m, 3H), 1.43 – 1.09 (m, 19H), 0.87 (t, *J* = 6.8 Hz, 3H).

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 177.5, 83.1, 73.7, 33.1, 32.0, 29.8, 29.75, 29.73, 29.6, 29.4, 28.8, 25.6, 24.2, 22.8, 14.2.

IR ν 3412, 2954, 2914, 2849, 1732, 1471, 1199 cm^{-1}.

HRMS (ESI) m/z = [M + H^+] calcd for $C_{17}H_{33}O_3^+$ 285.2430, found 285.2456 (9.1 ppm error).

^{1}H NMR comparison with literature values **(***Tetrahedron Lett.* **1991**, *32*, 1137−1140)

Our synthetic sample, 400 MHz, $CDCl_3$	Literature values 500 MHz, $CDCl_3$
δ 4.41 (td, *J* = 7.4, 4.6 Hz, 1H)	4.43 (1H dt)
3.64 – 3.49 (m, 1H)	3.58 (1H m)
2.68 – 2.46 (m, 2H)	2.63, 1H (dt), 2.54, 1H (dt)
2.25 (dddd, *J* = 12.6, 9.6, 7.3, 5.0 Hz, 1H)	2.25 (1H ddt)
2.18 – 2.04 (m, 1H)	2.12, 1 H (ddt)
1.89 (s, 1H)	
1.61 – 1.43 (m, 3H)	1.52 (2H m)
1.43 – 1.09 (m, 19H)	1.4-1.3 (20H m)
0.87 (t, *J* = 6.8 Hz, 3H)	0.87 (3H t 6.8)

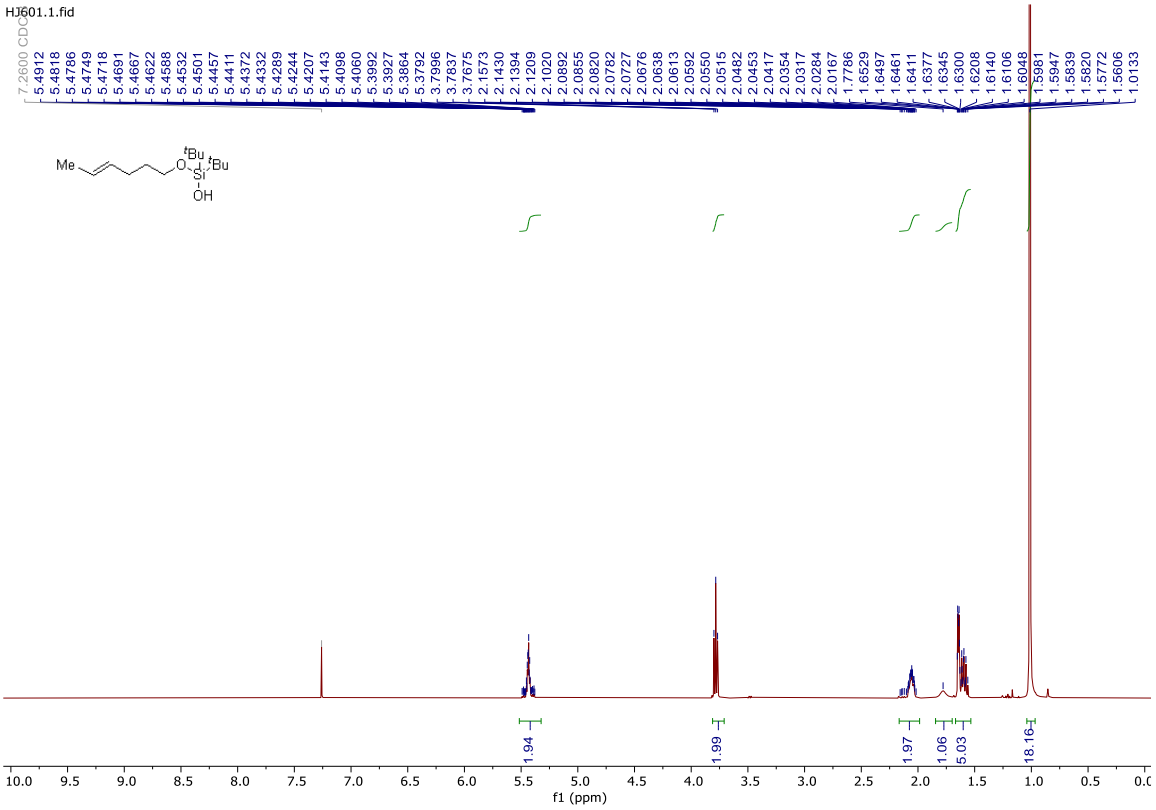
^{13}C {1H} NMR comparison with literature values **(***Tetrahedron Lett.* **1991**, *32*, 1137−1140)

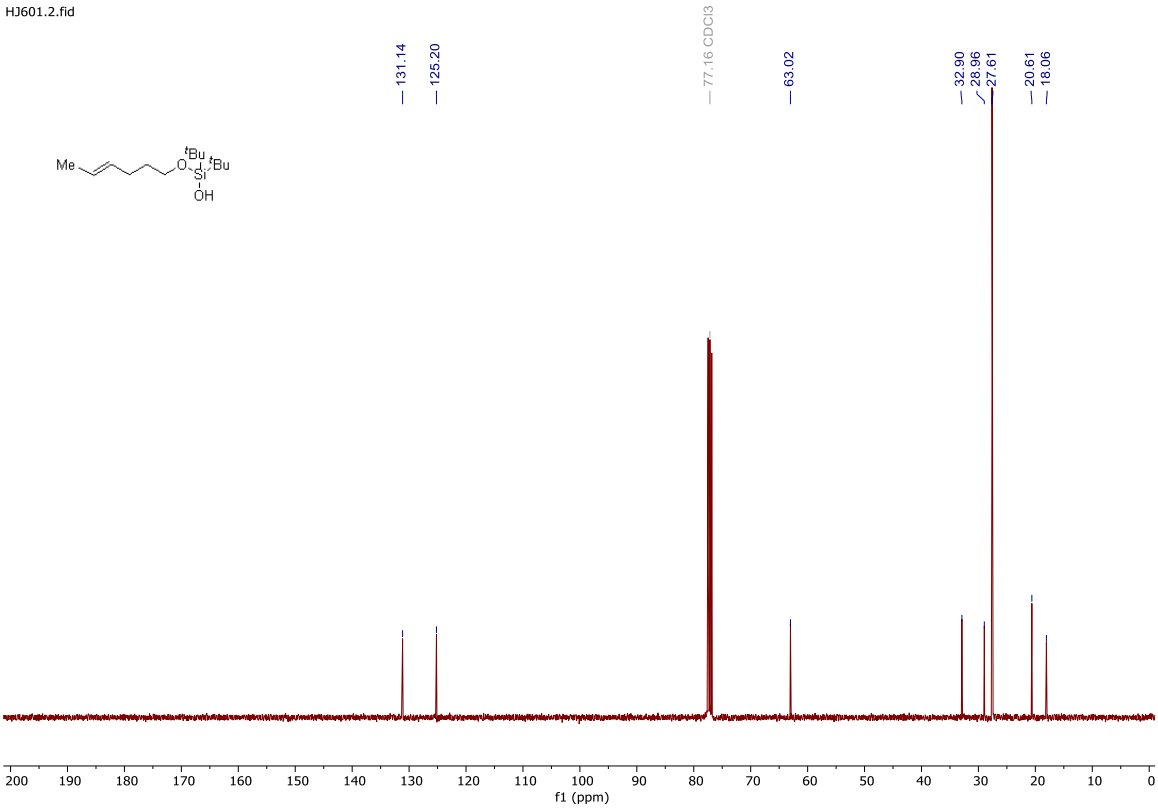
Our synthetic sample, 101 MHz, $CDCl_3$	Literature values 125 MHz, $CDCl_3$
177.5	177.14
83.1	82.92
73.7	73.65
33.1	32.92
32.0	32.88-22.65
29.8	

29.75	
29.73	
29.6	
29.4	
28.8	28.67
25.6	32.88-22.65
24.2	24.07
22.8	22.65
14.2	14.07

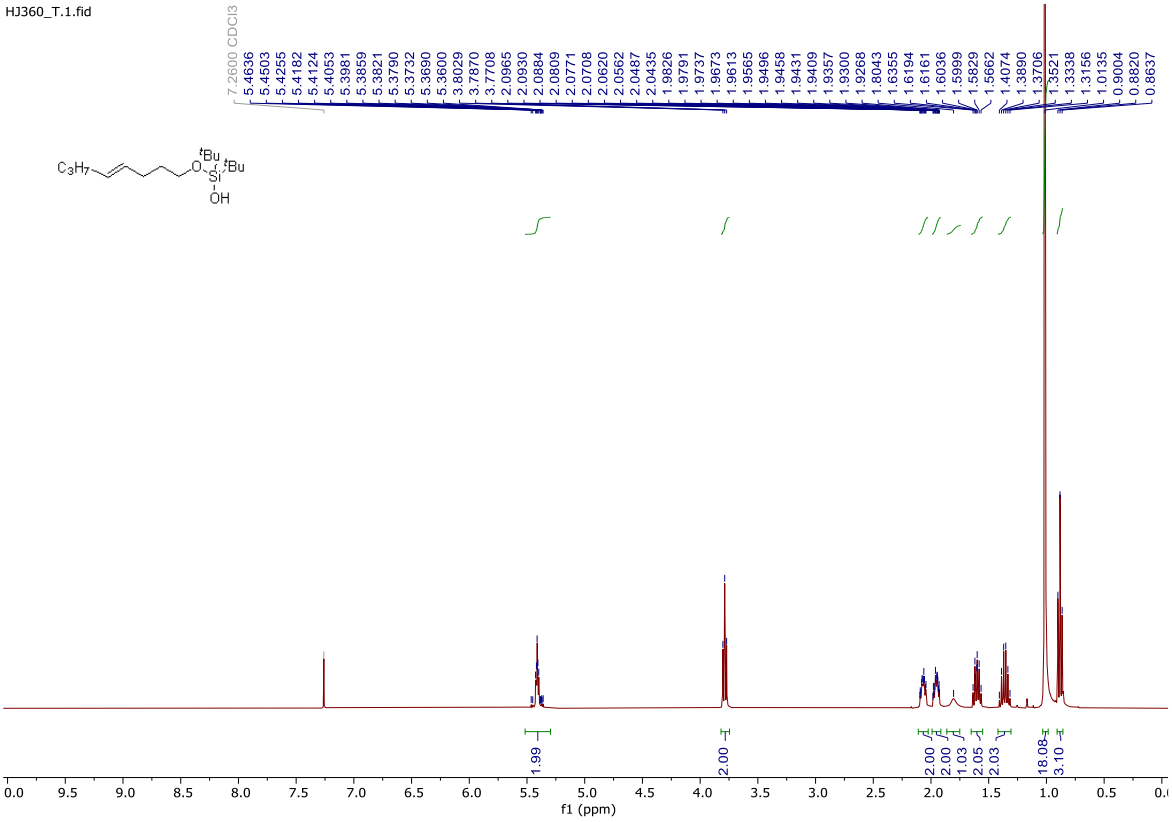
XIII. NMR Spectra

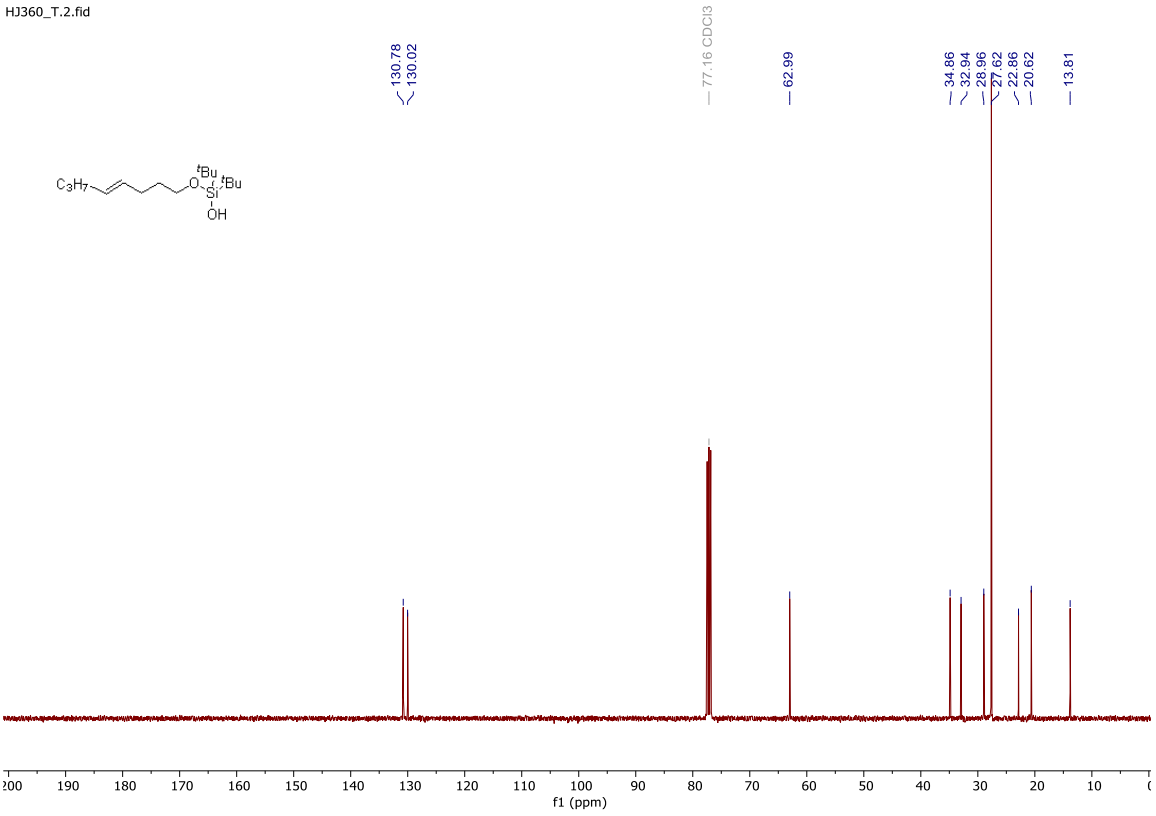
Compound S1 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



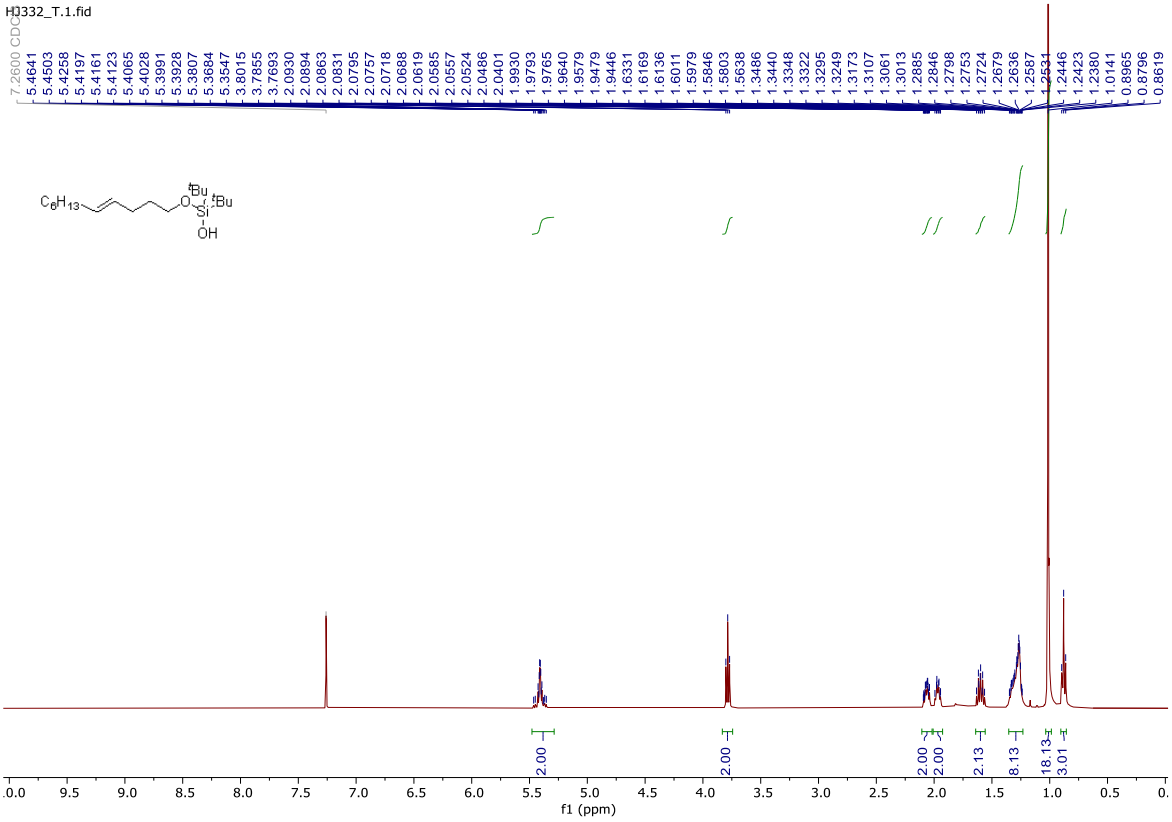


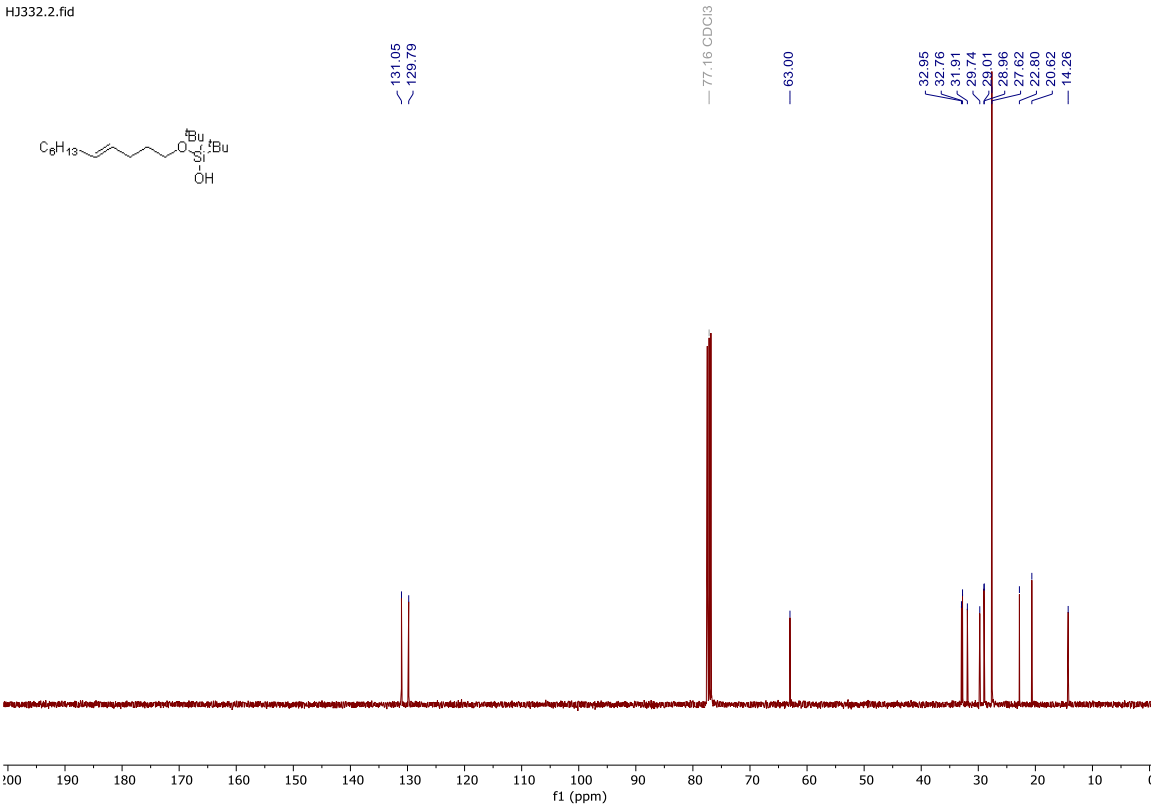
Compound S3 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



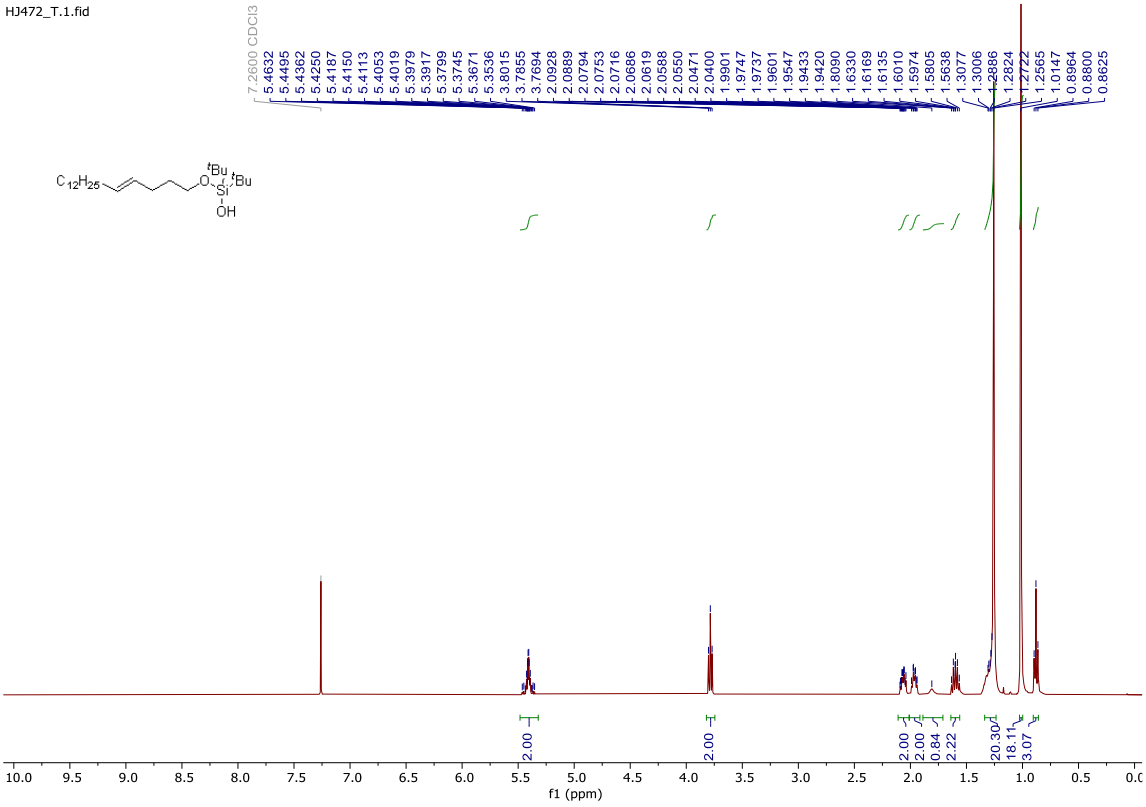


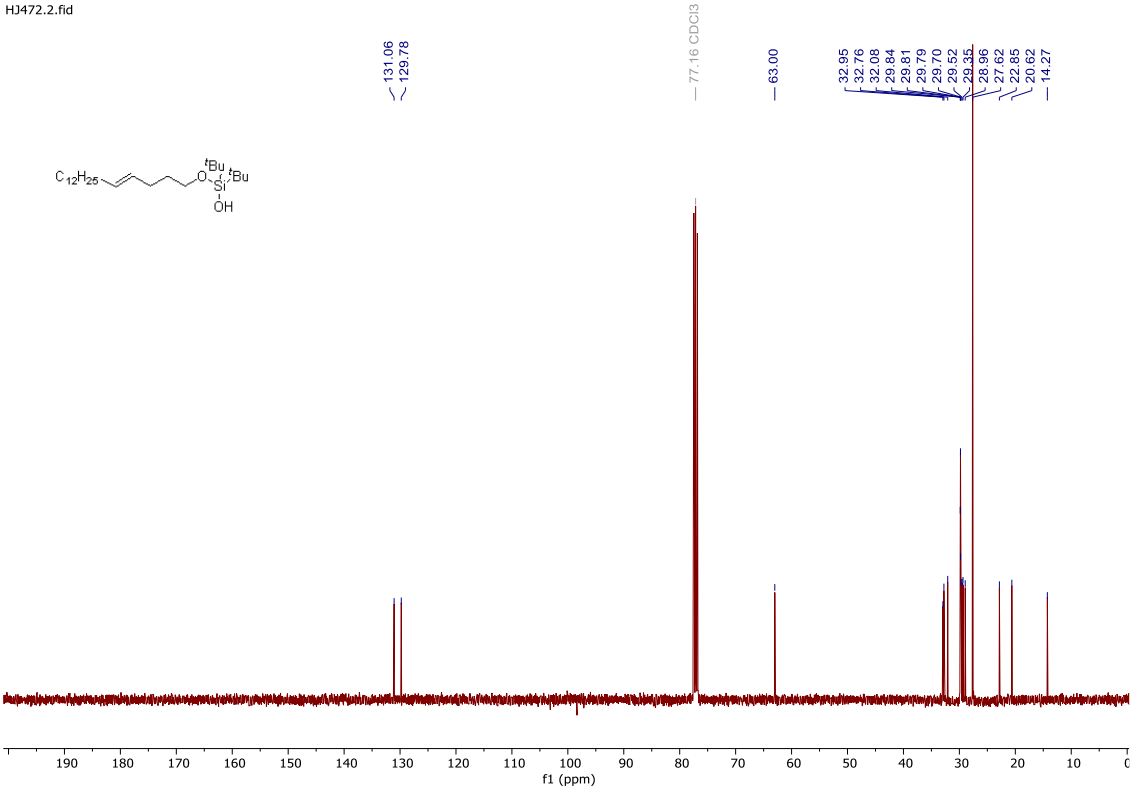
Compound S5 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



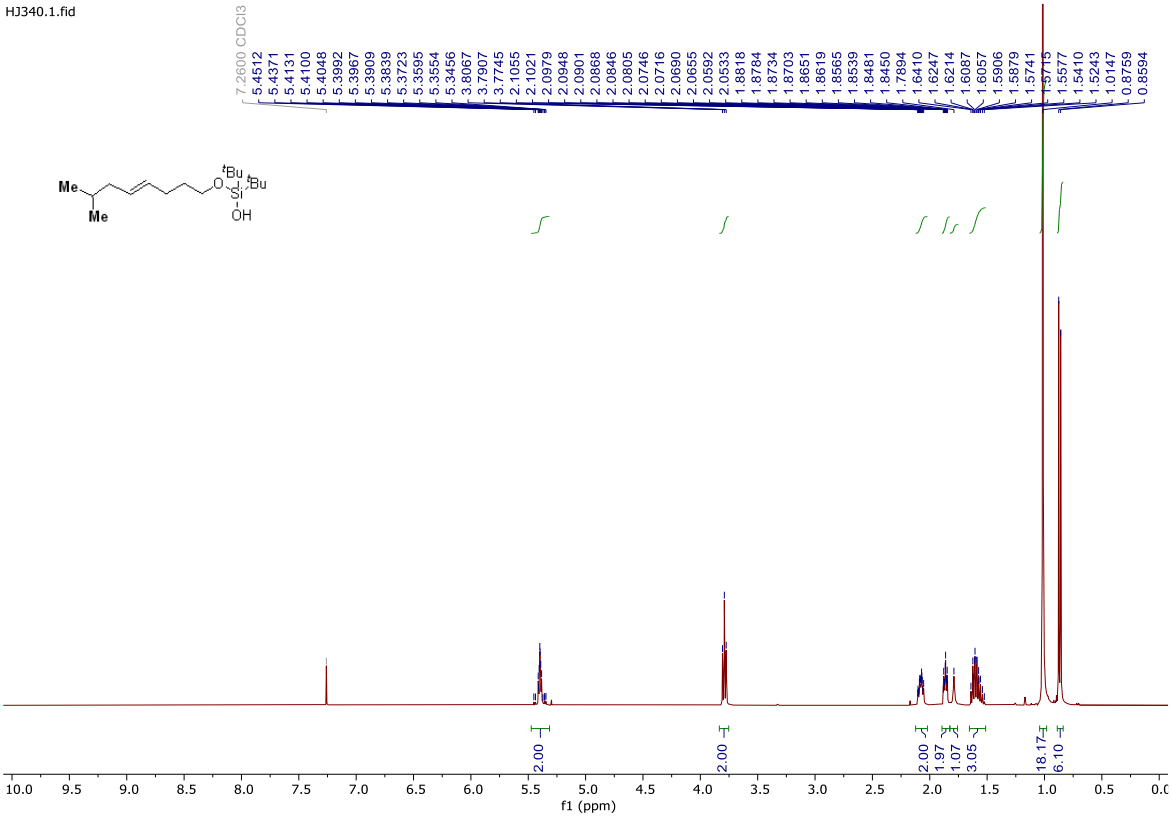


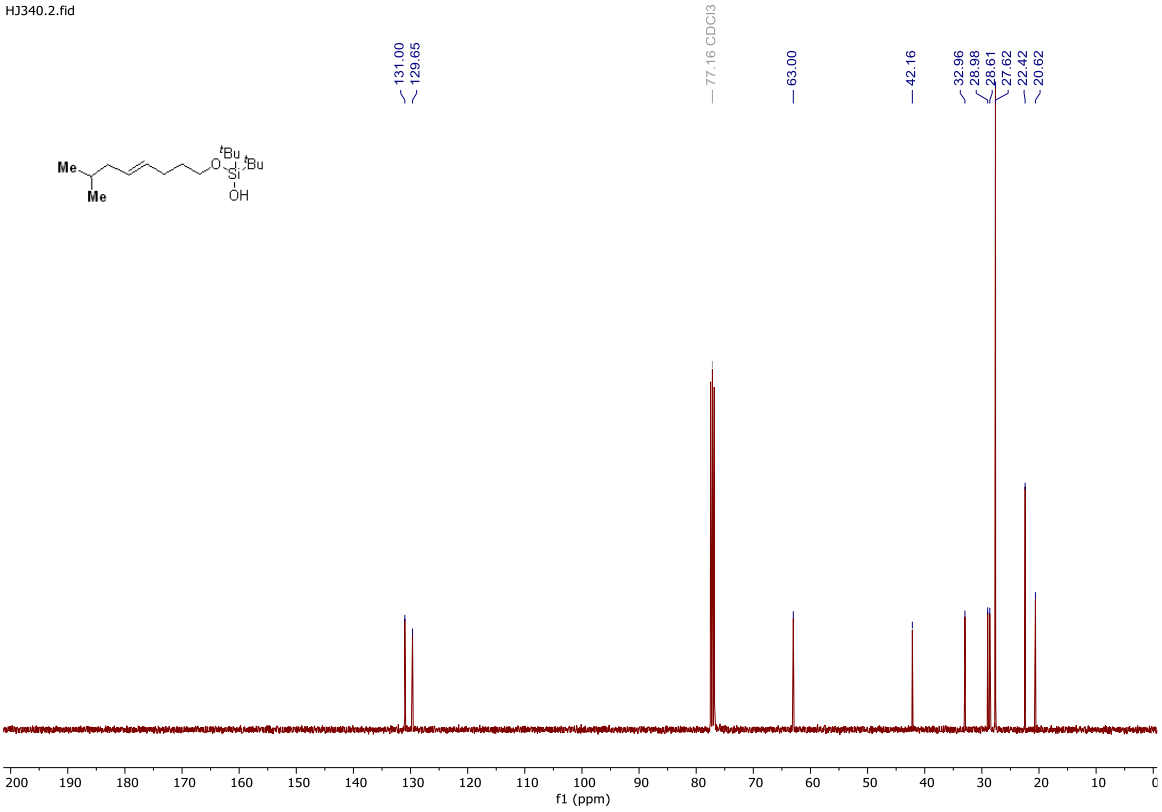
Compound S7 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



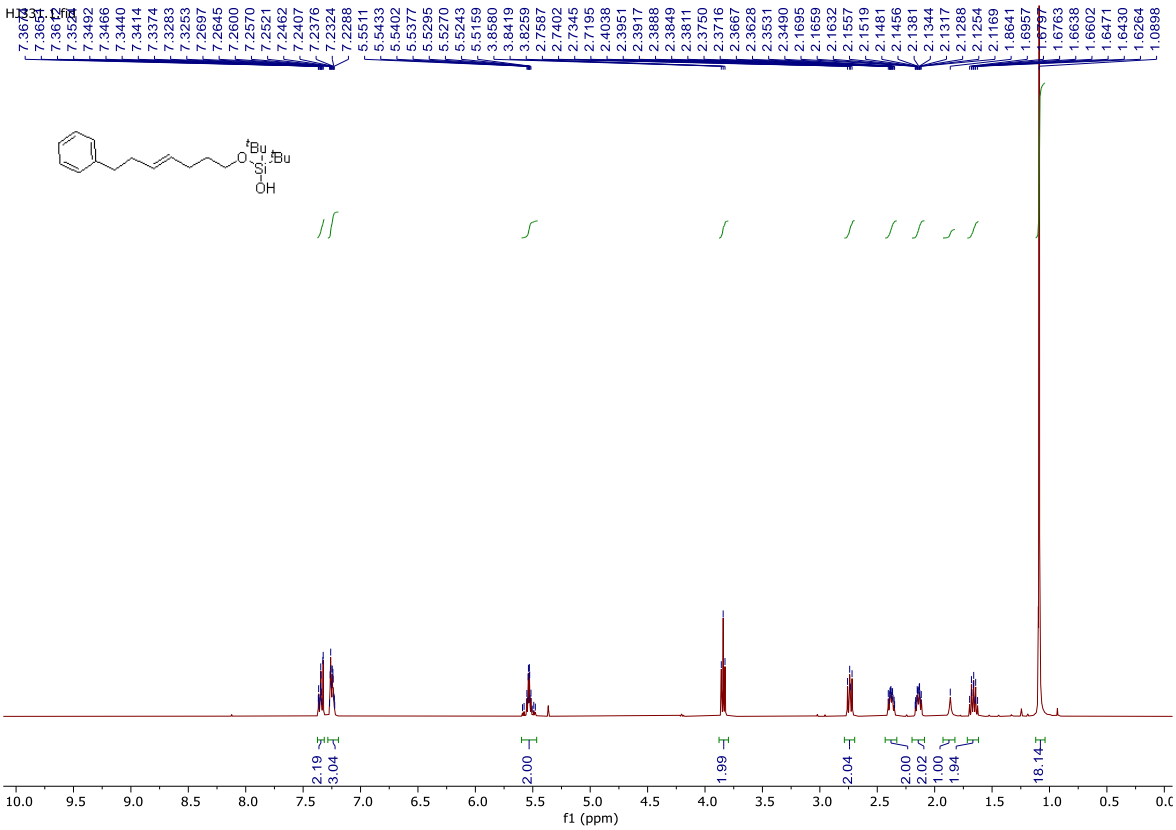


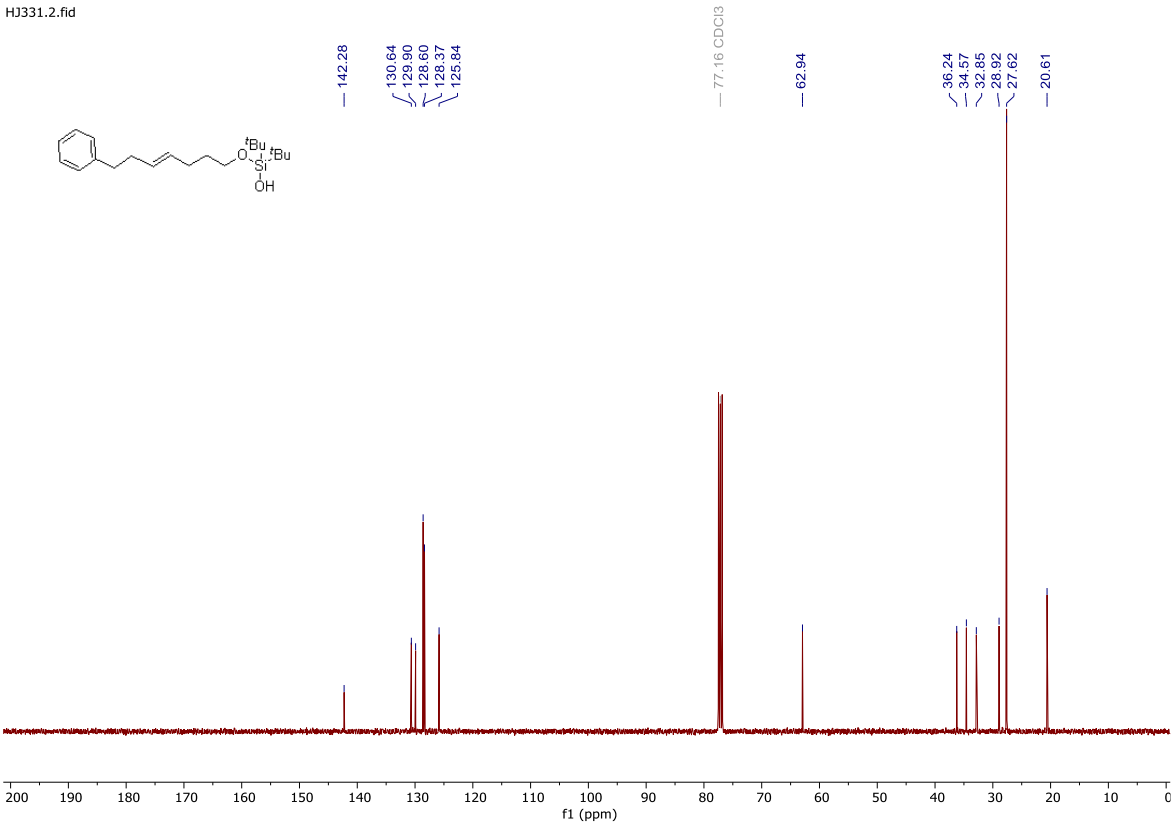
Compound S9 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



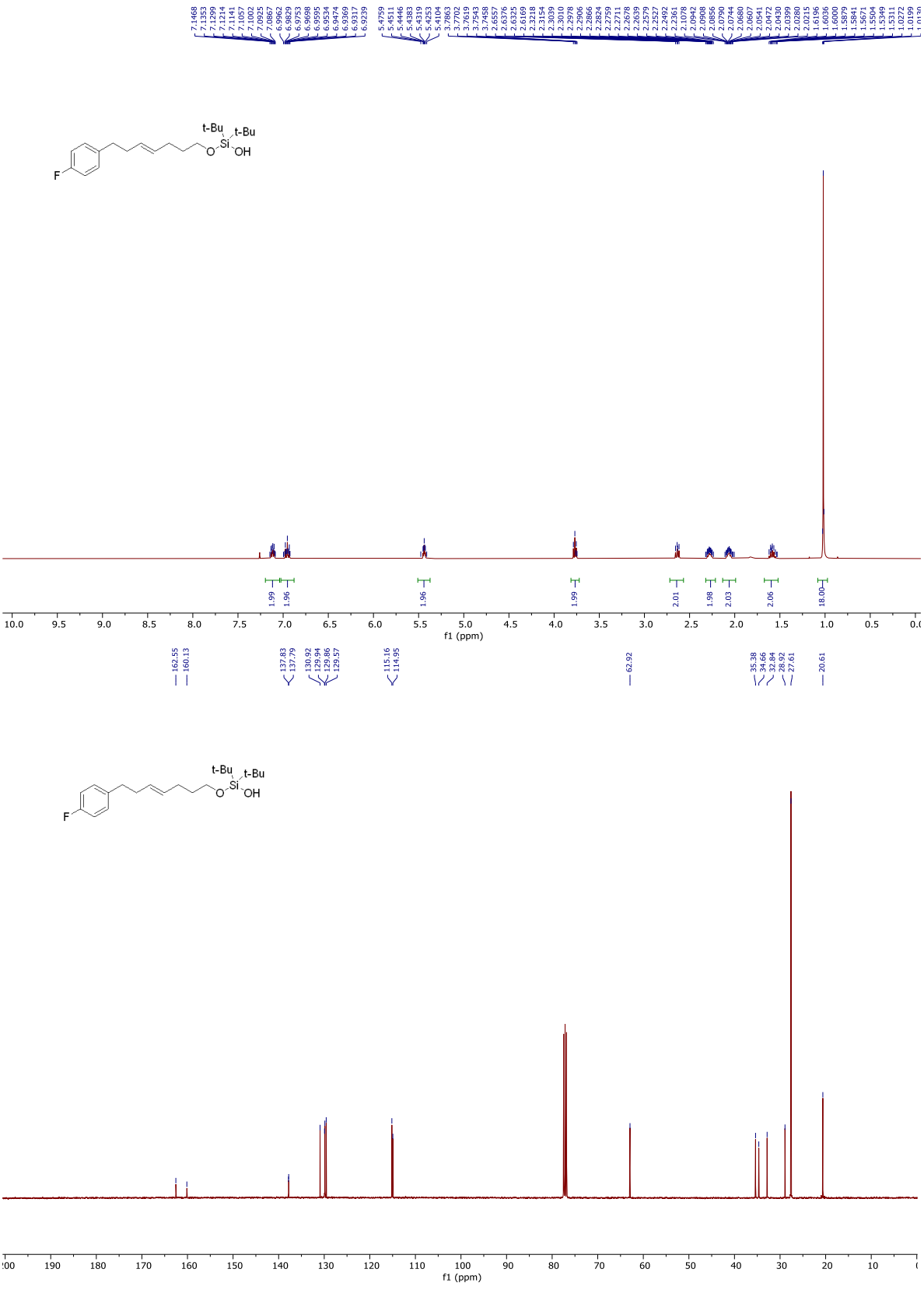


Compound S11 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

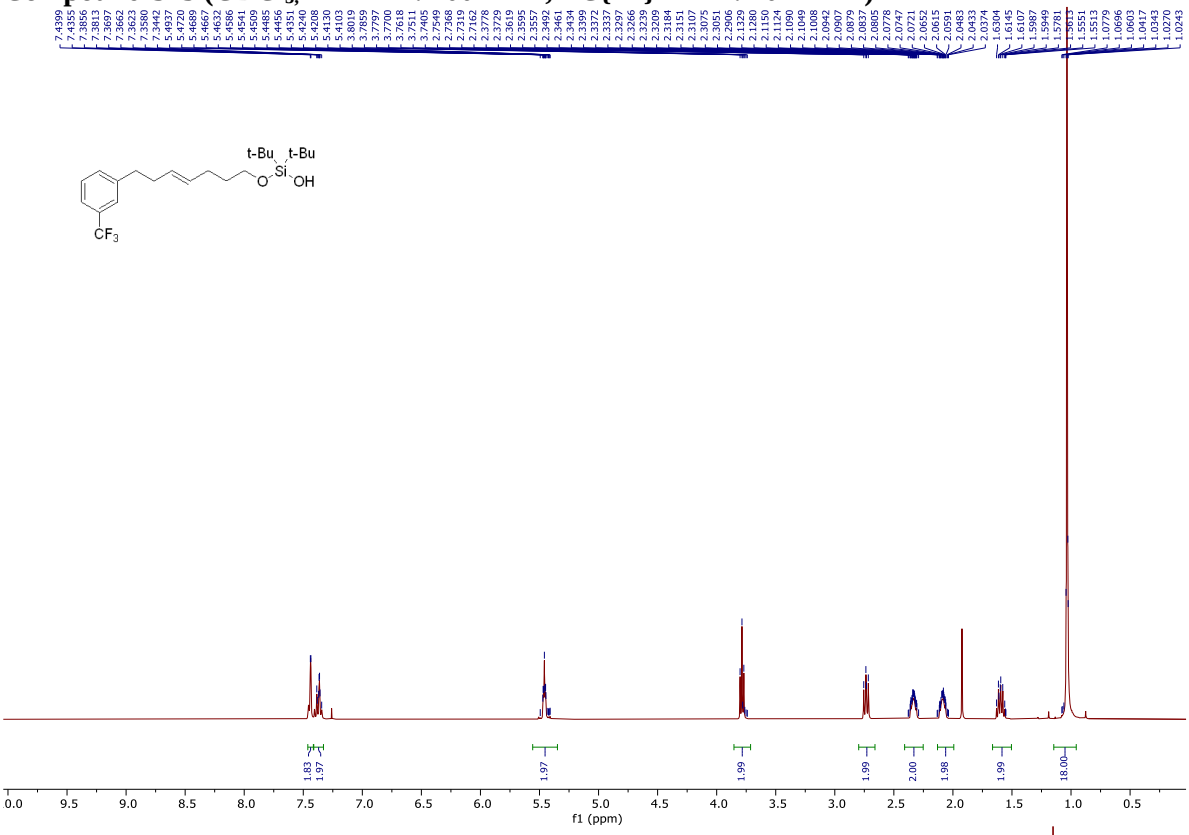


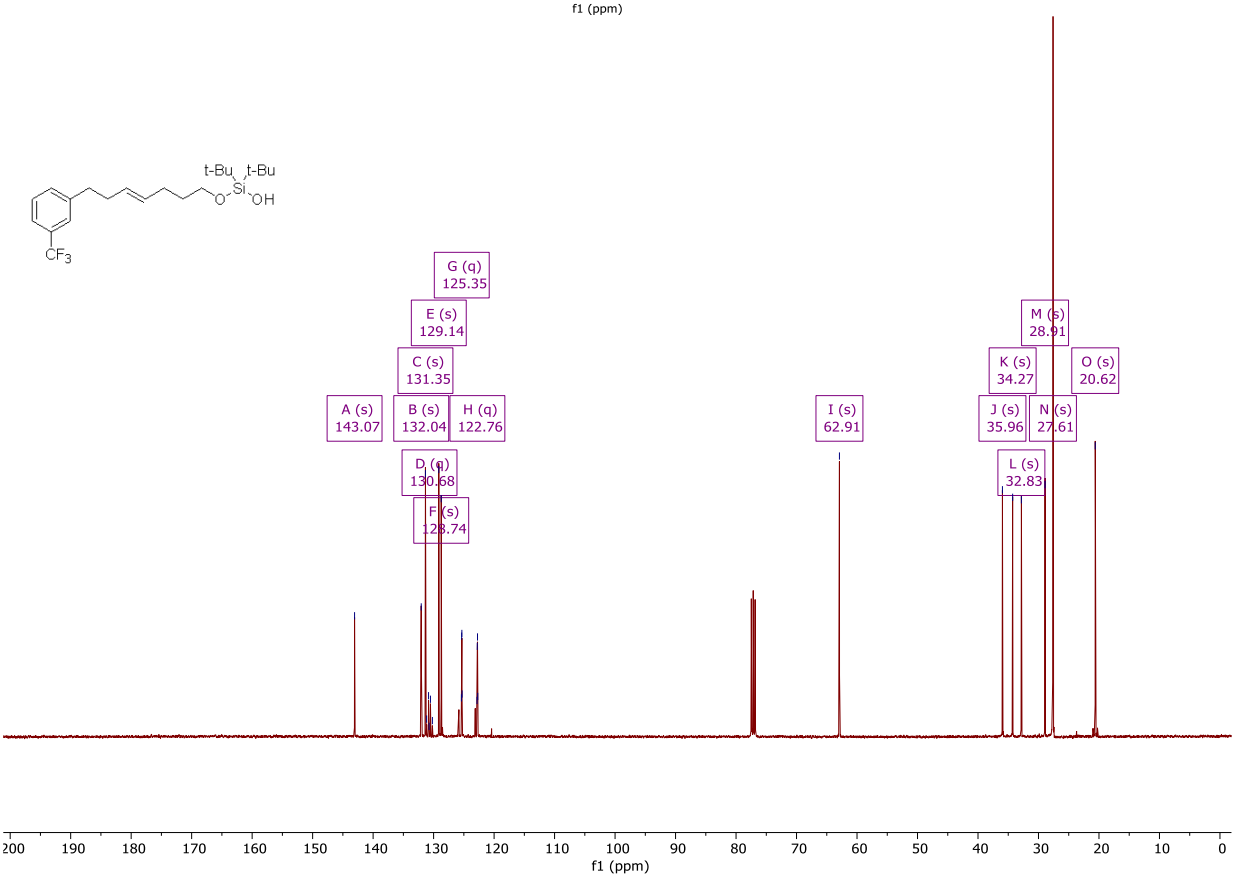


Compound S13 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

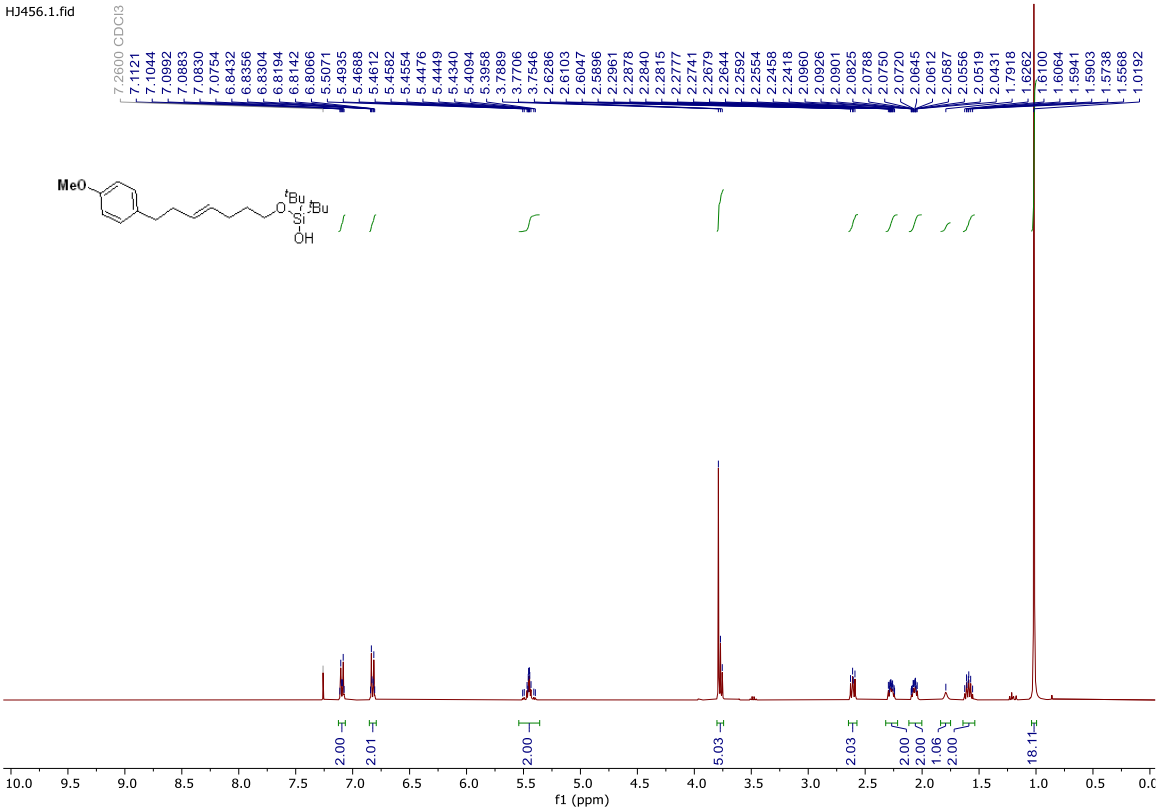


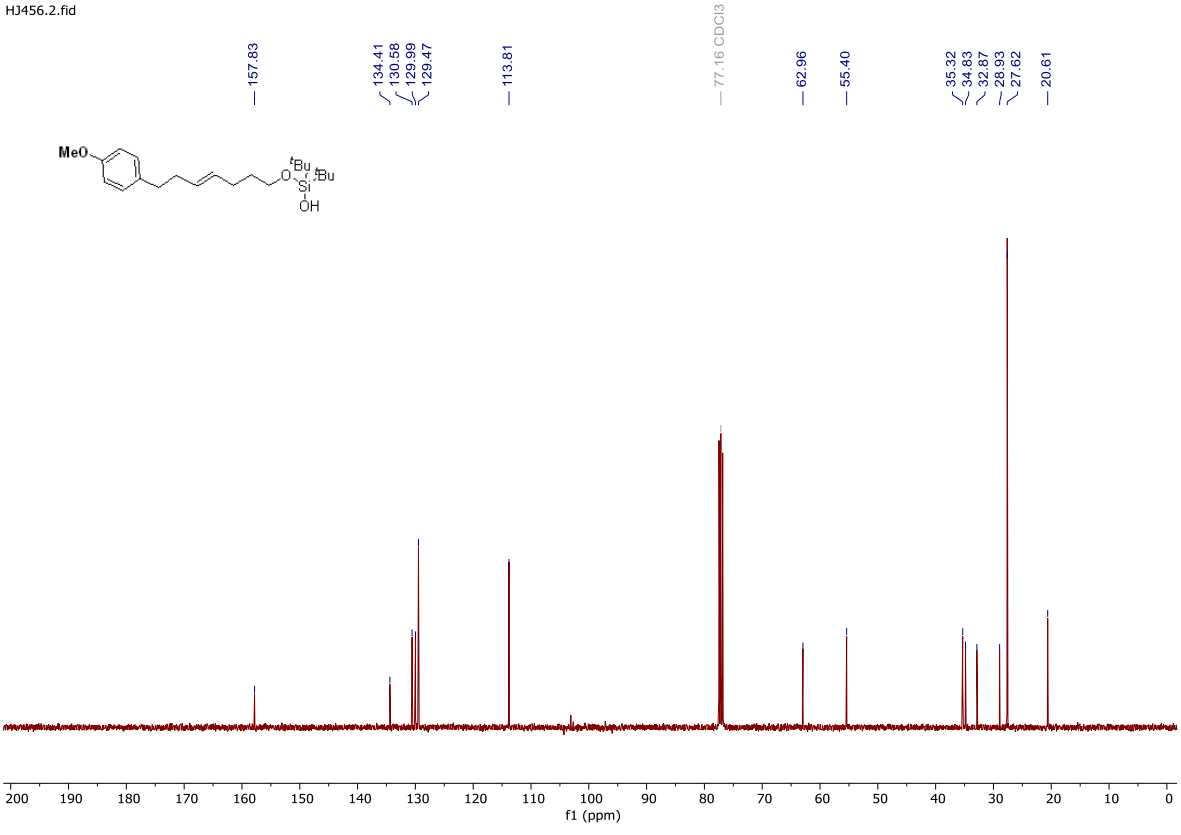
Compound S15 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



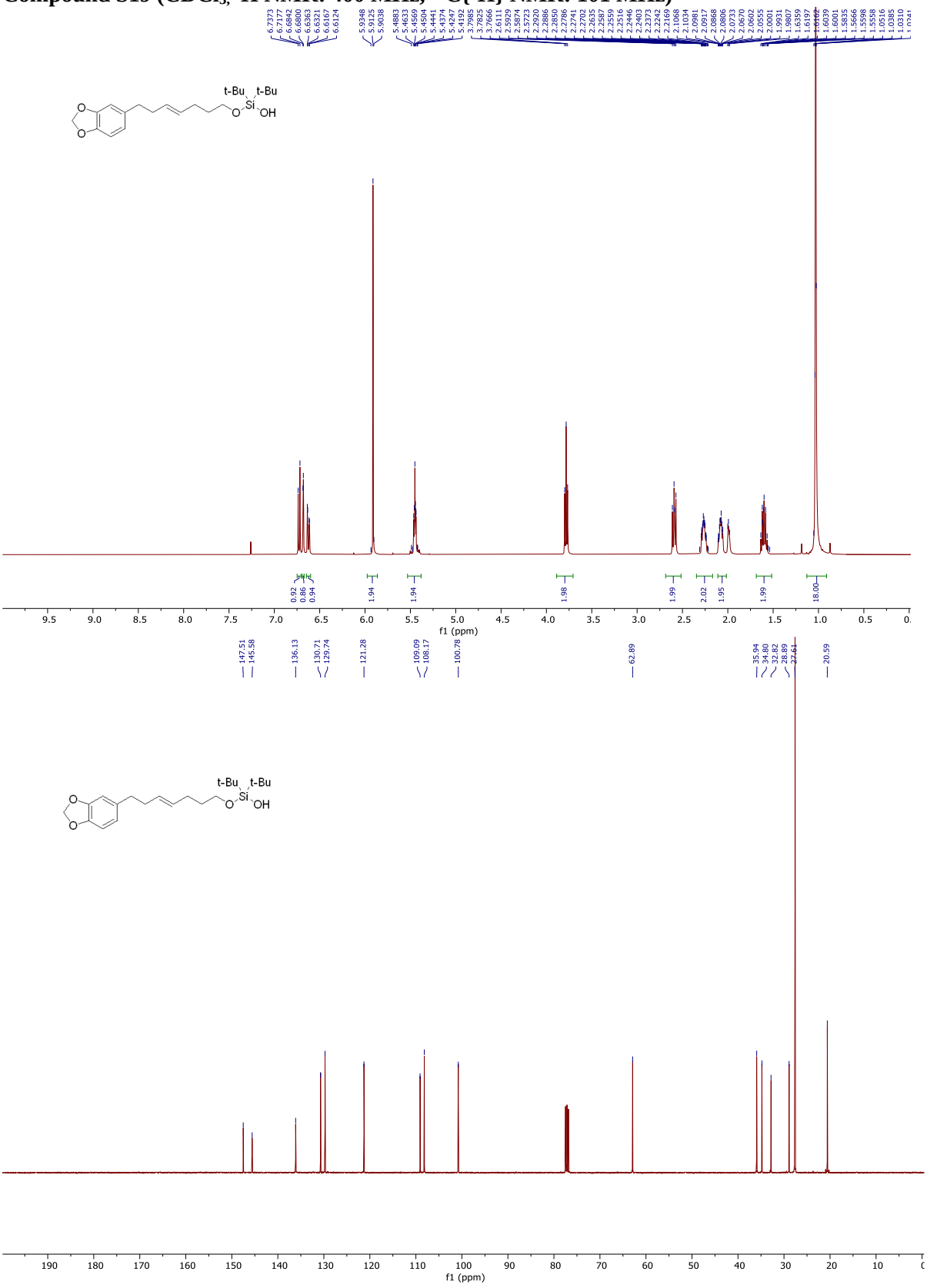


Compound S17 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

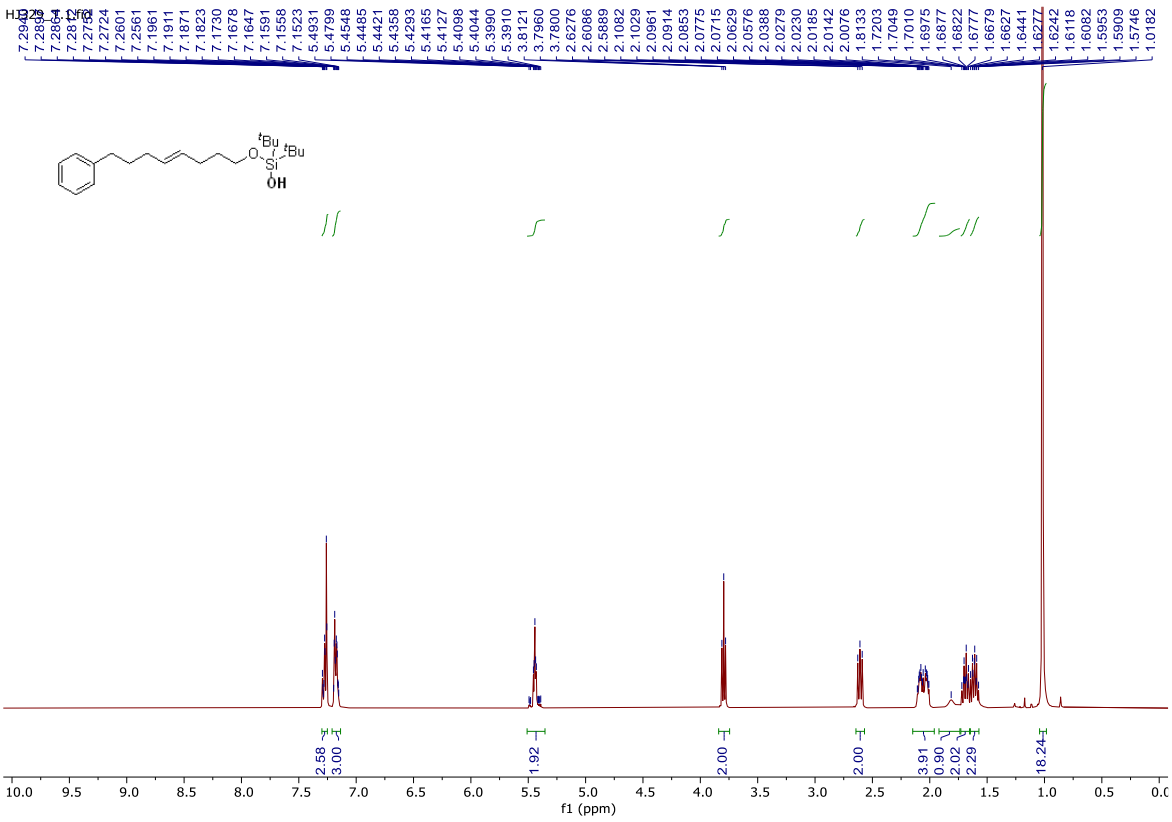


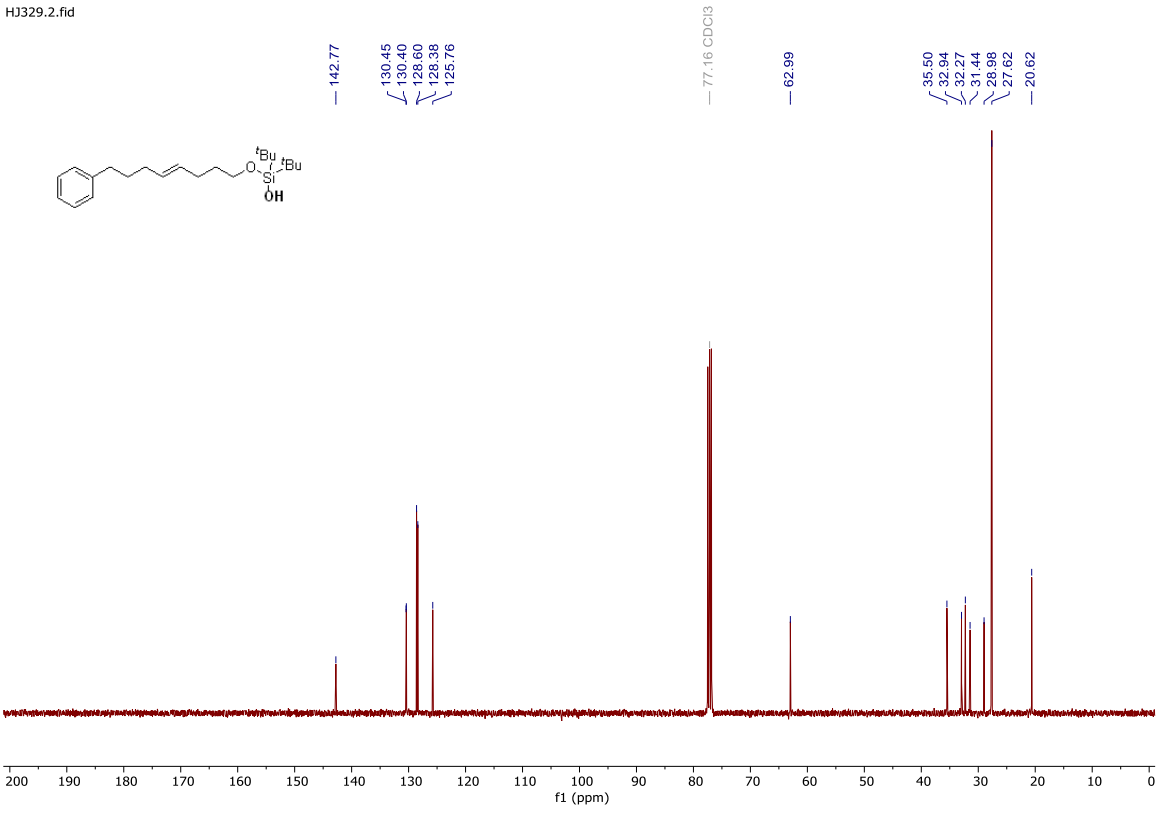


Compound S19 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

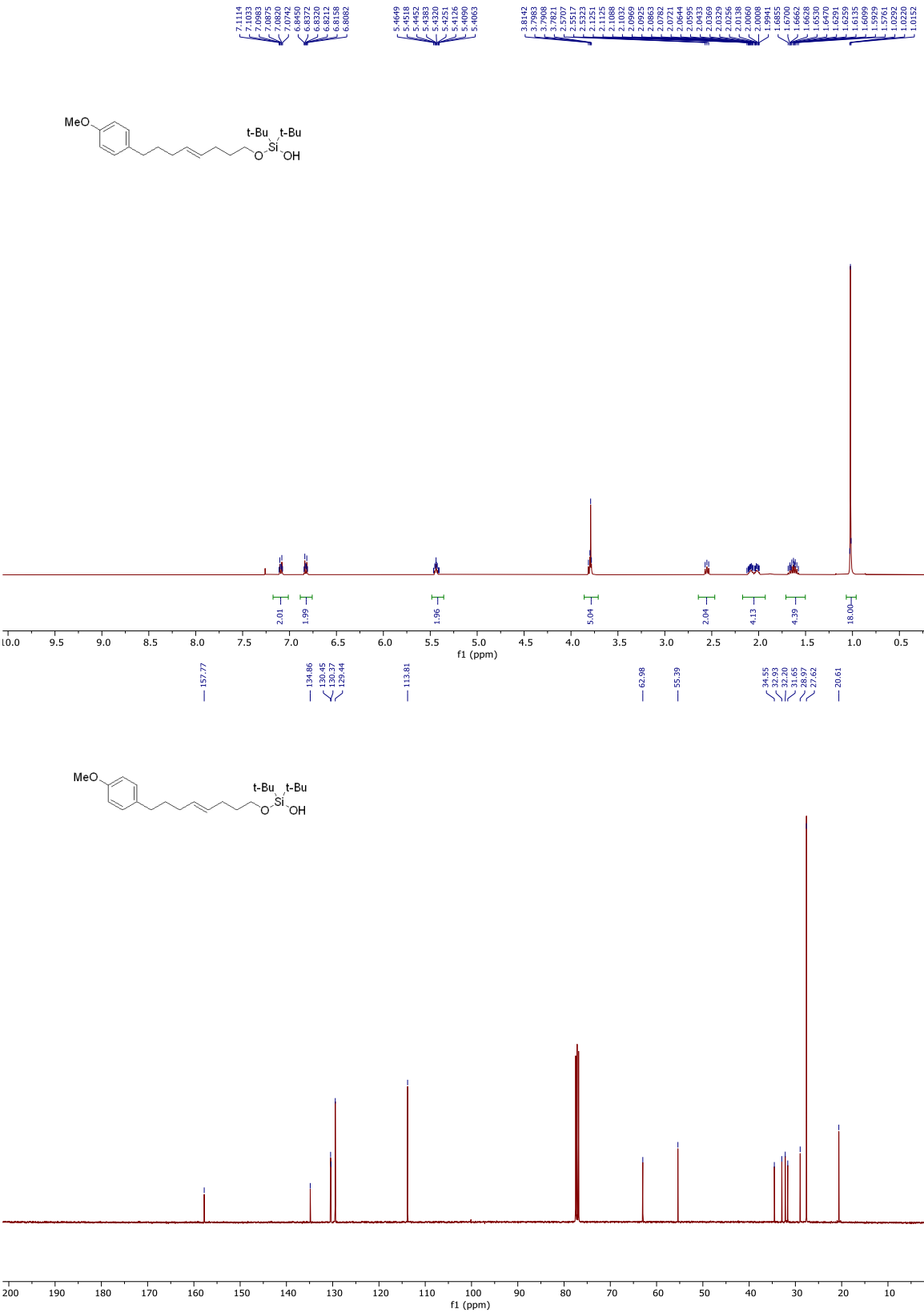


Compound S21 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

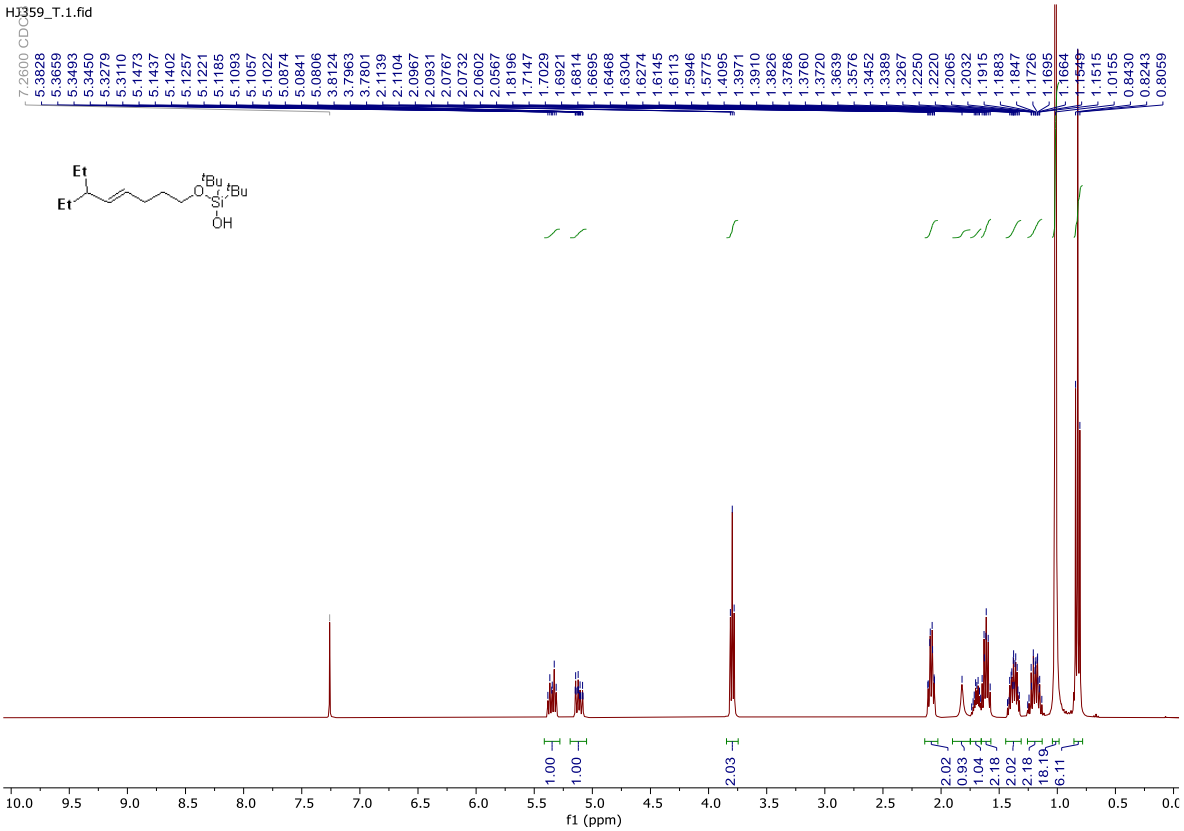


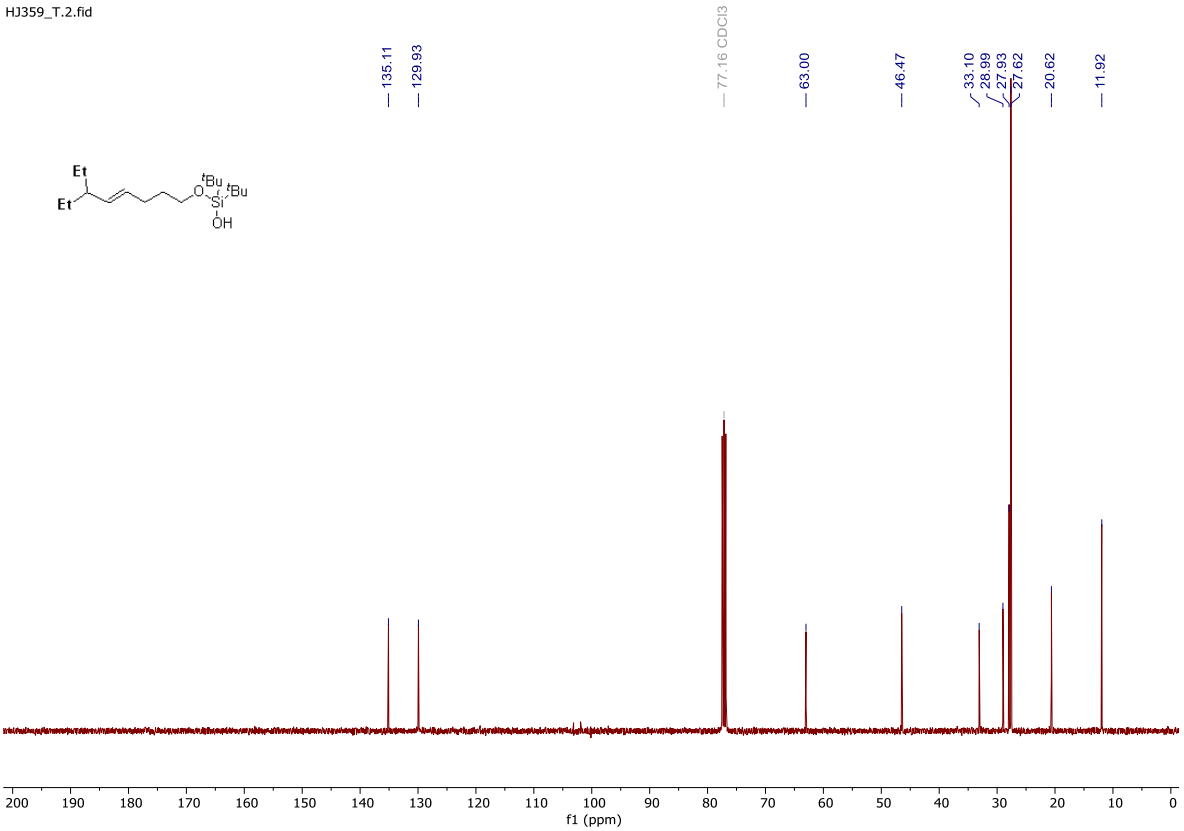


Compound S23 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

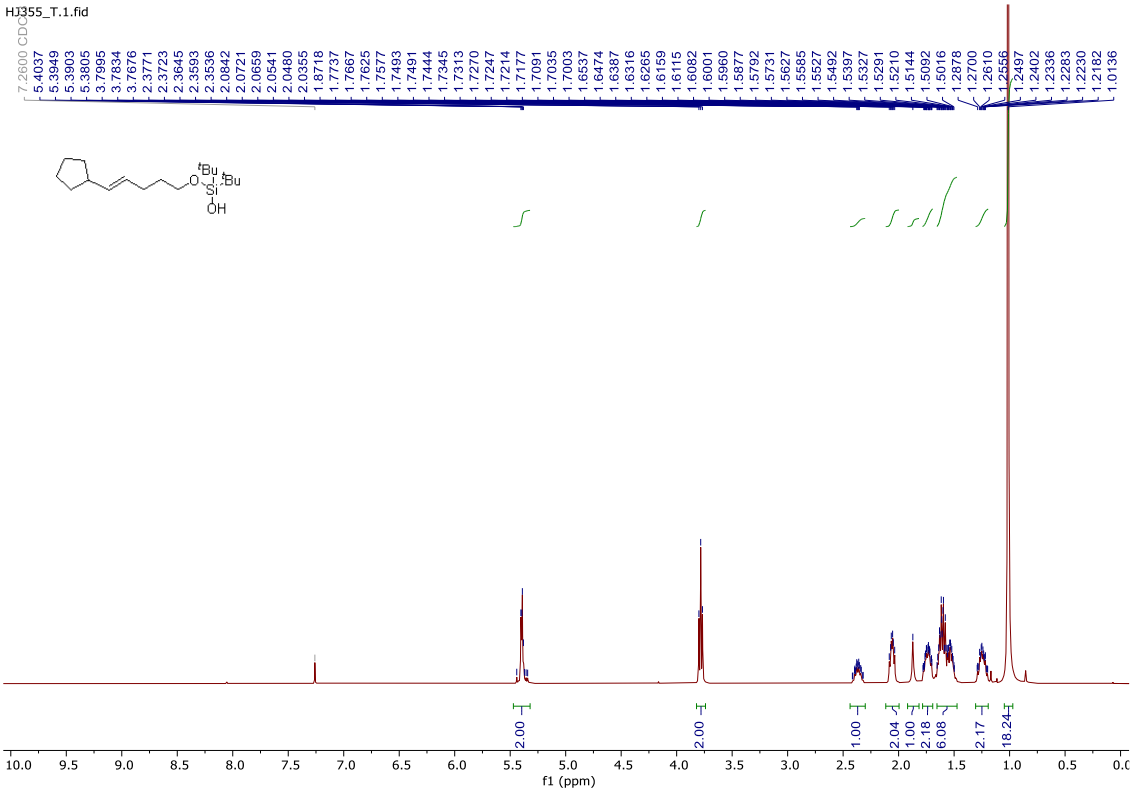


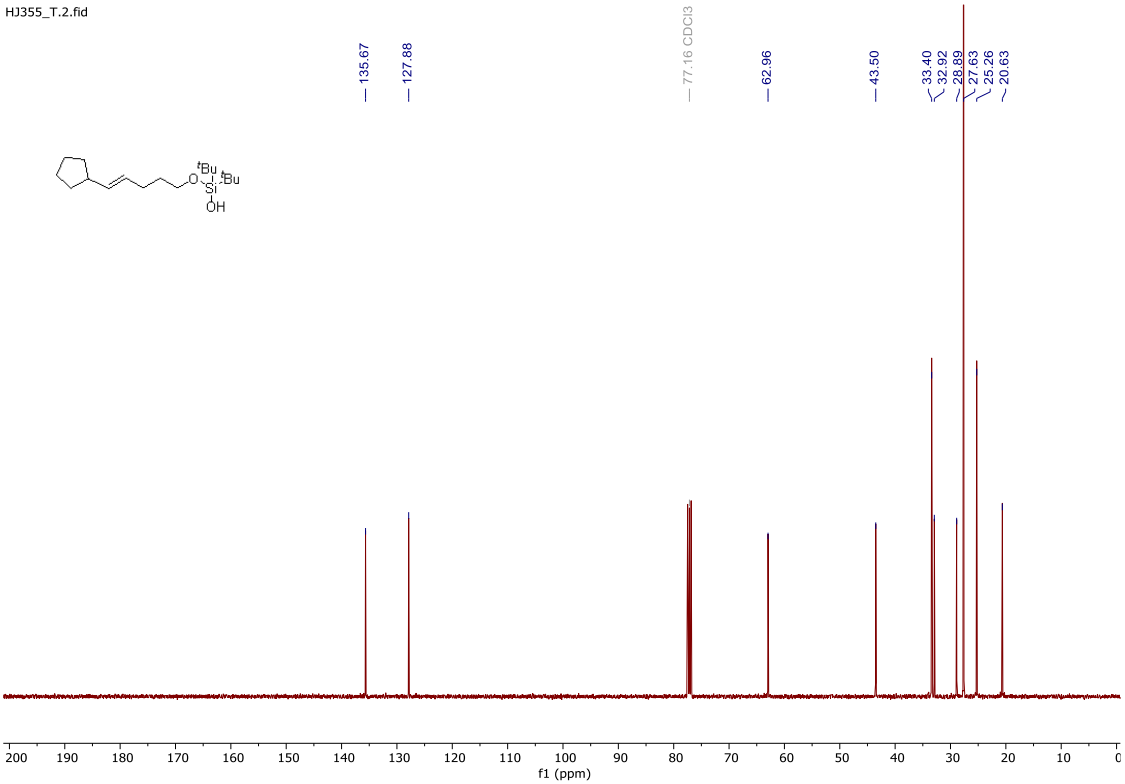
Compound S25 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



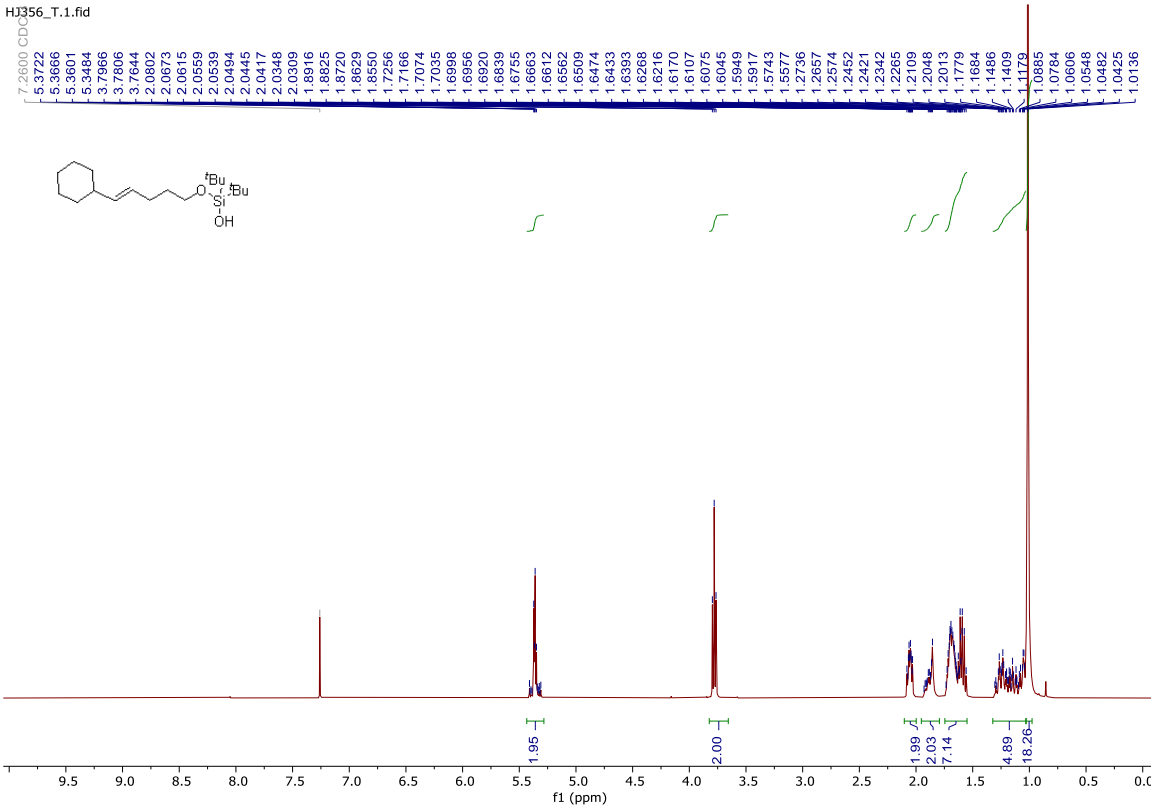


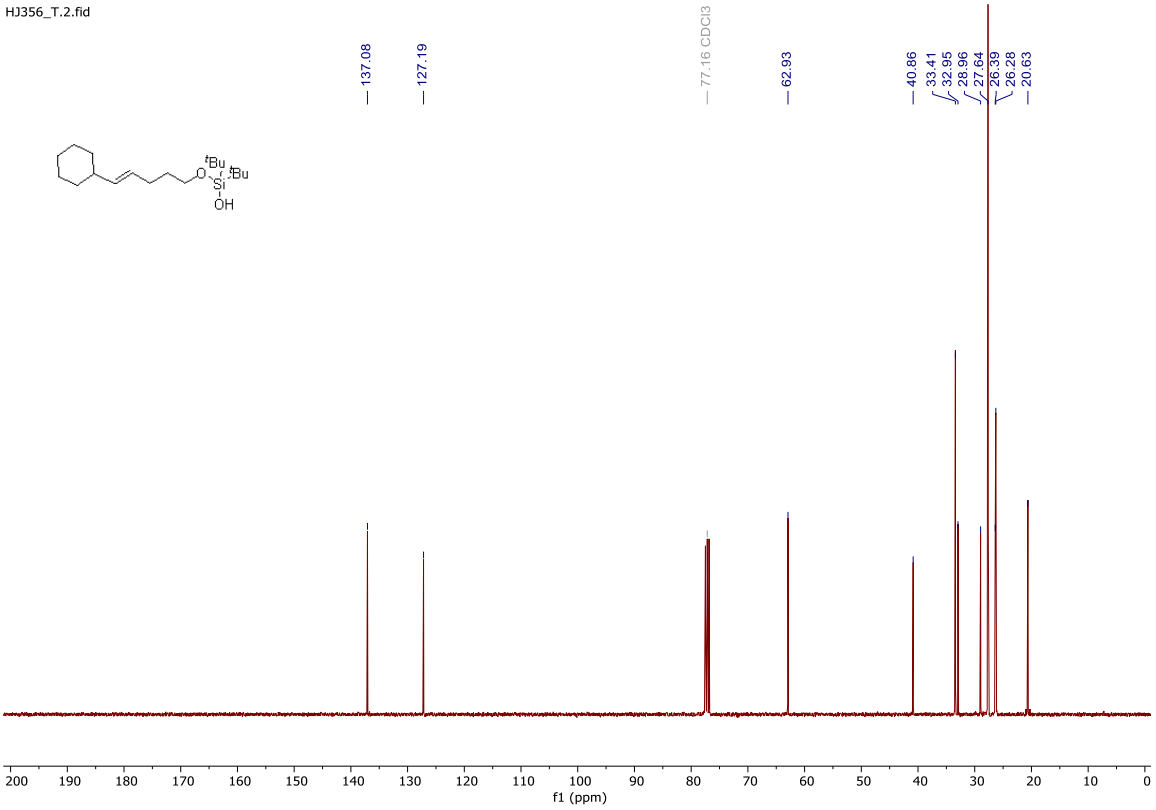
Compound S27 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



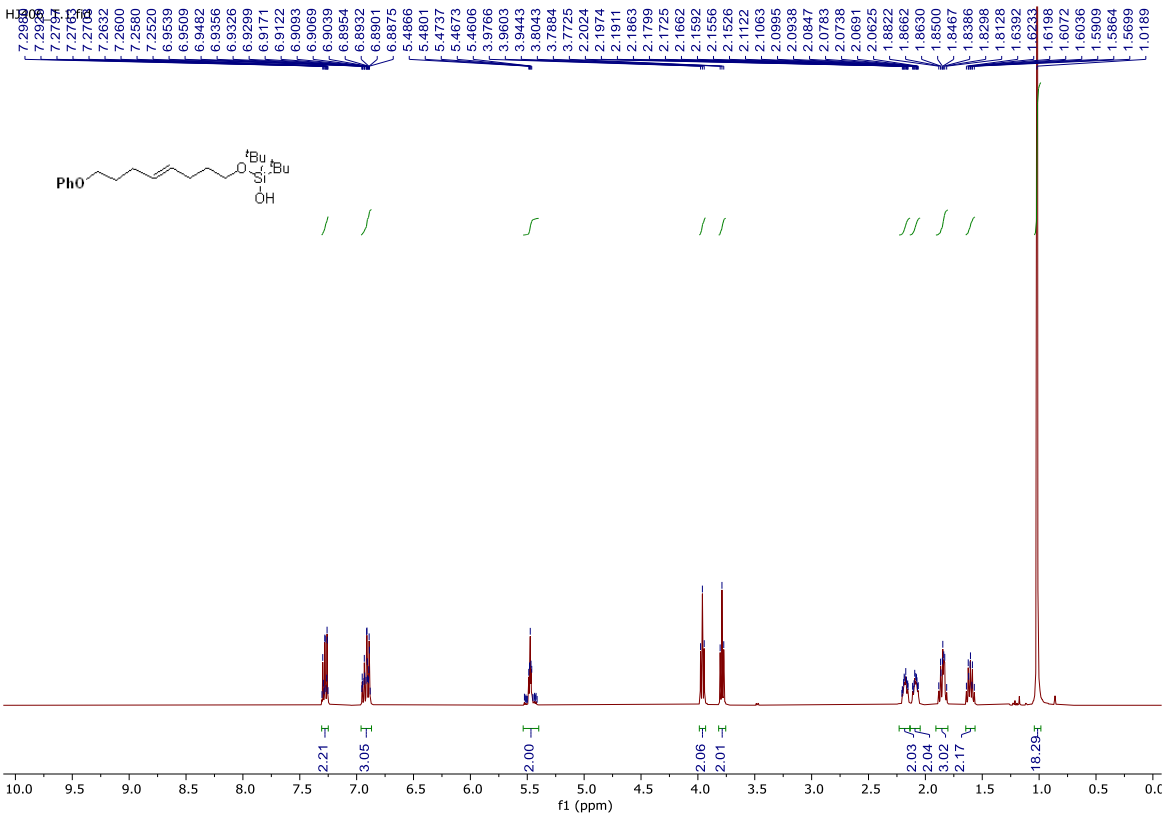


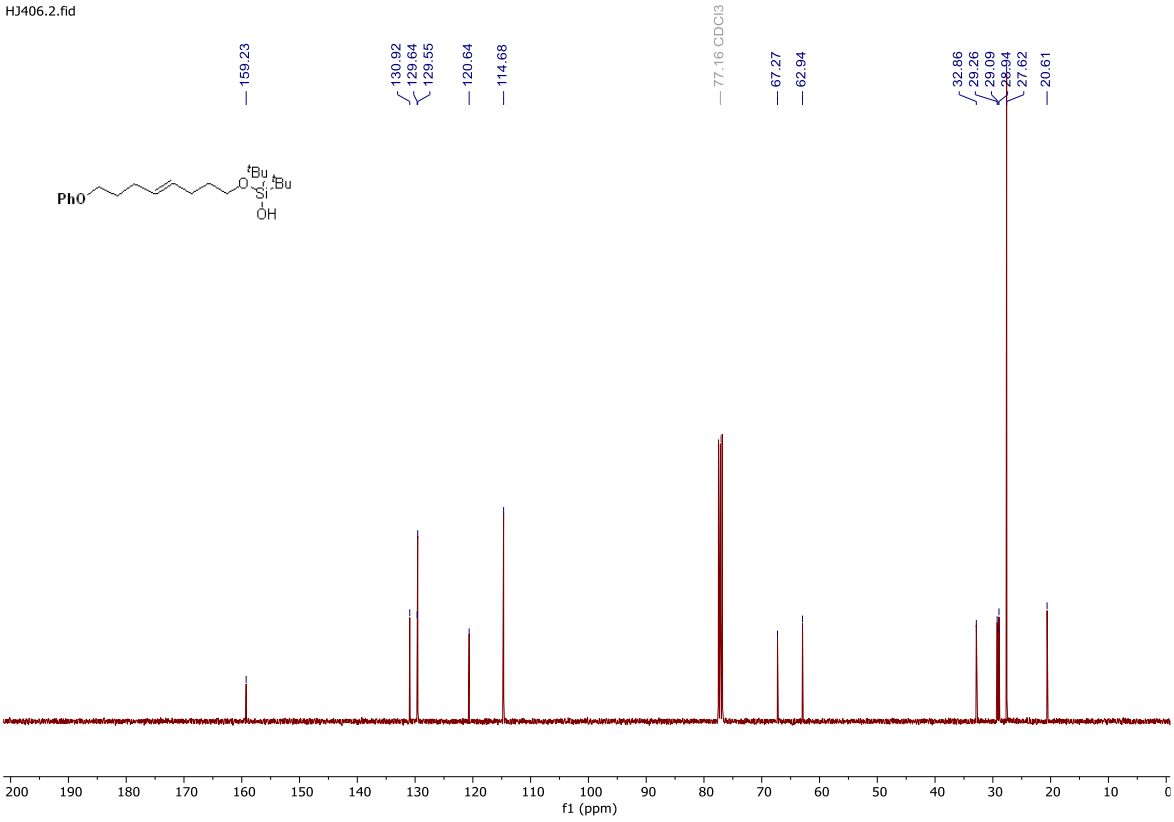
Compound S29 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



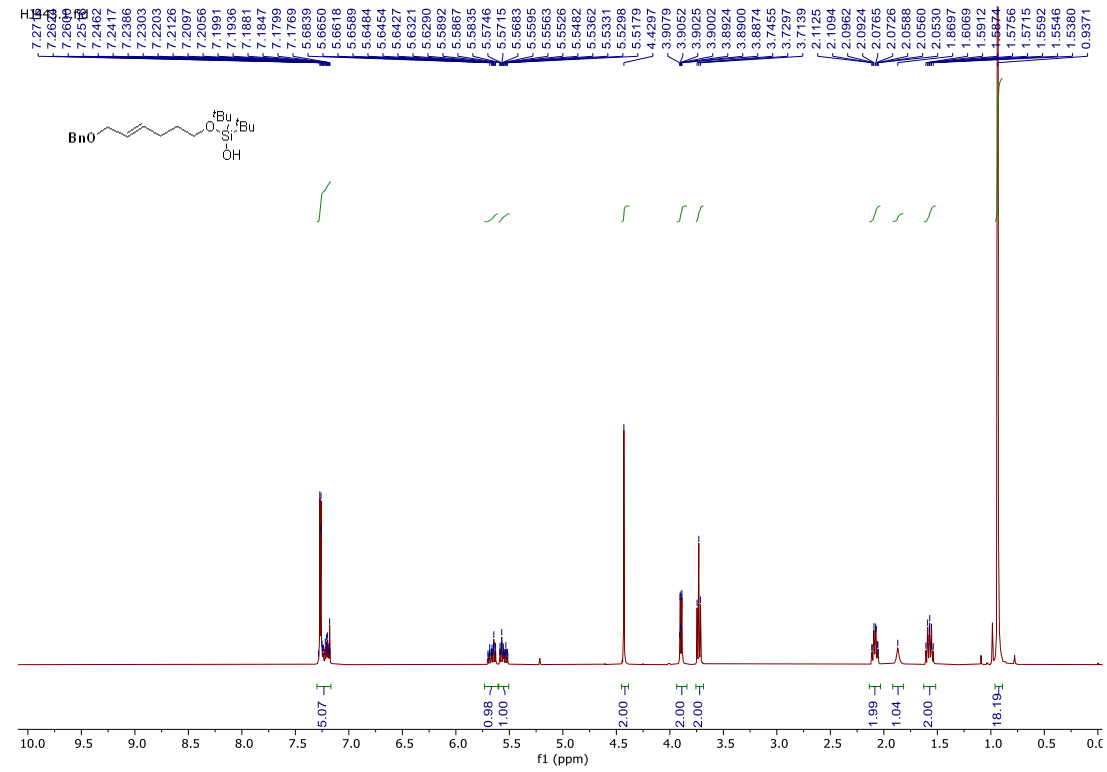


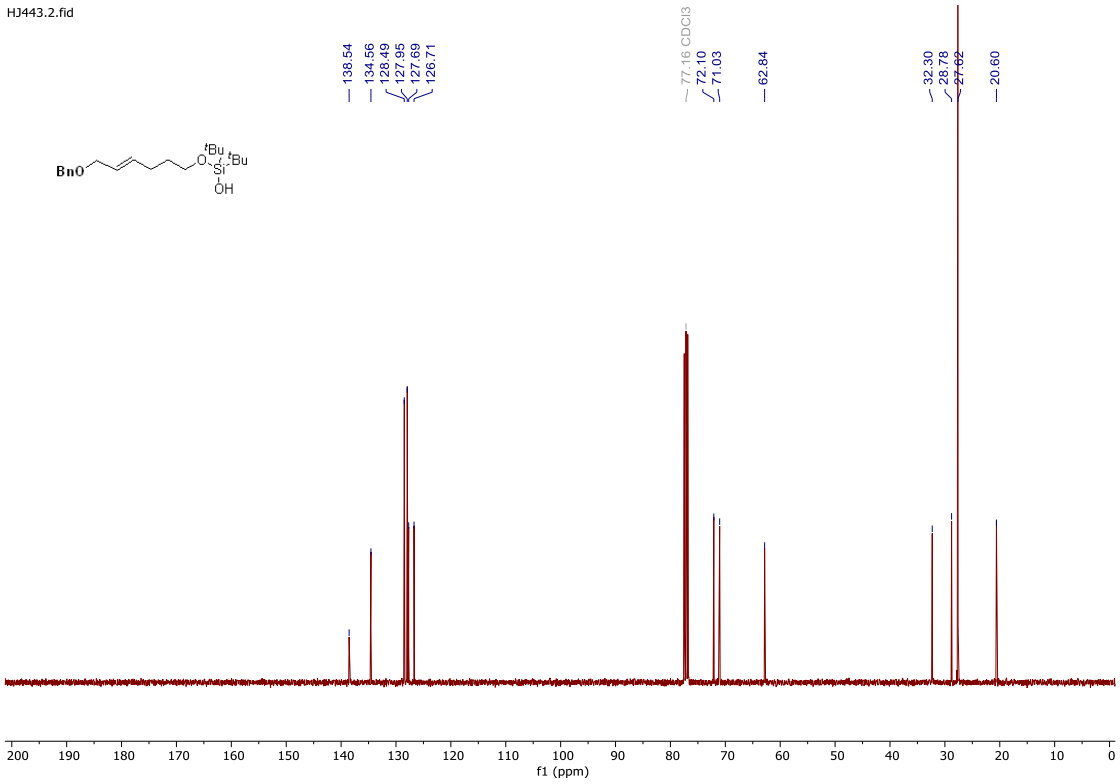
Compound S31 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



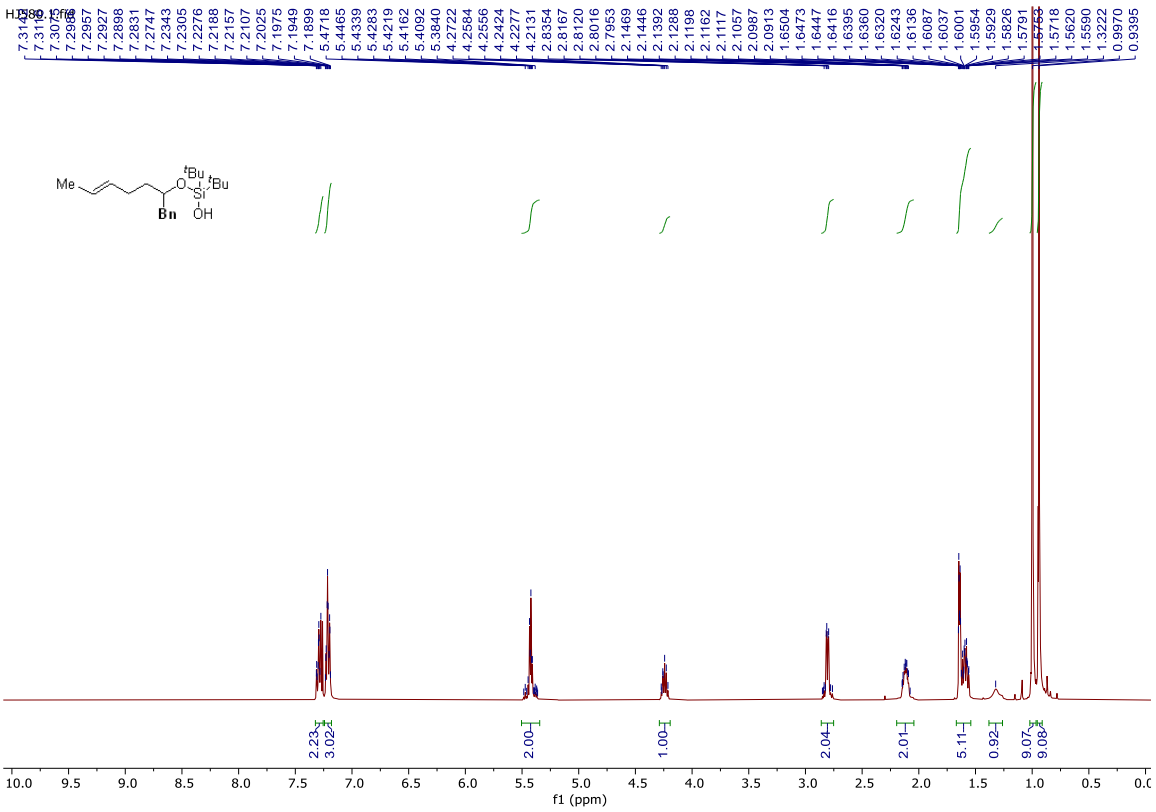


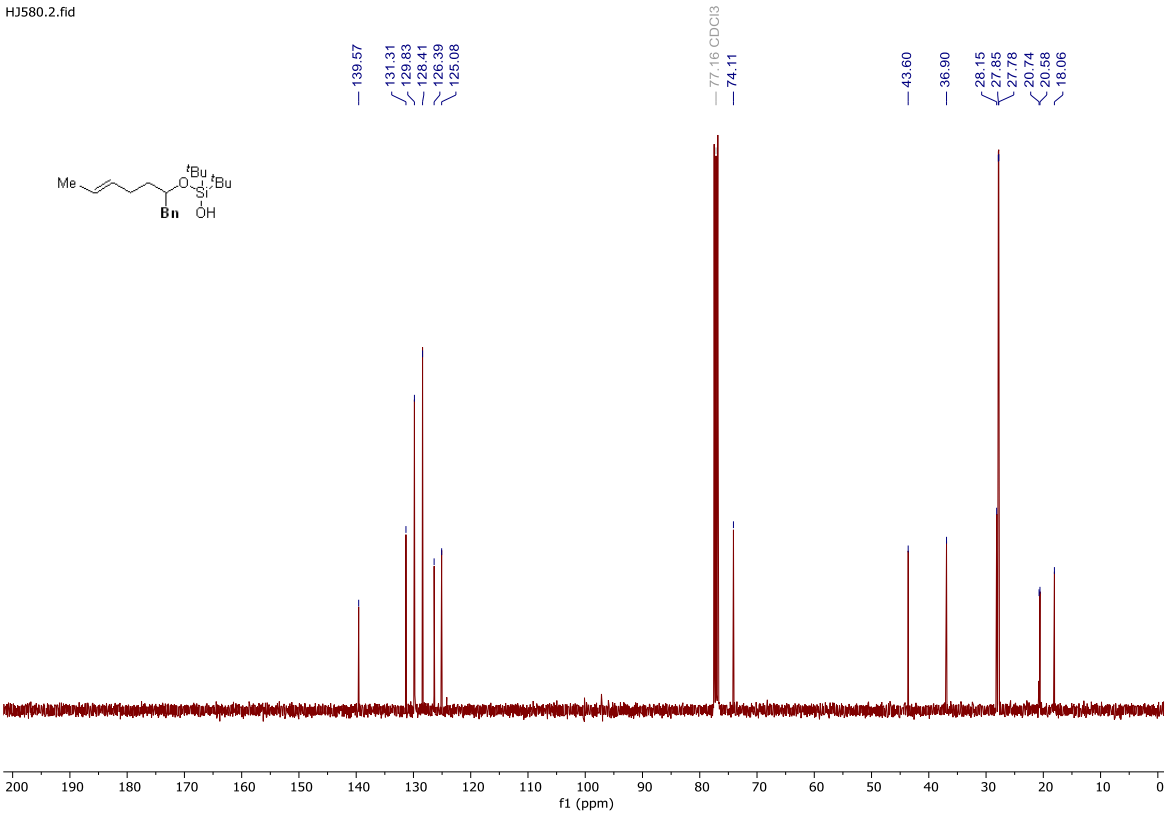
Compound S33 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



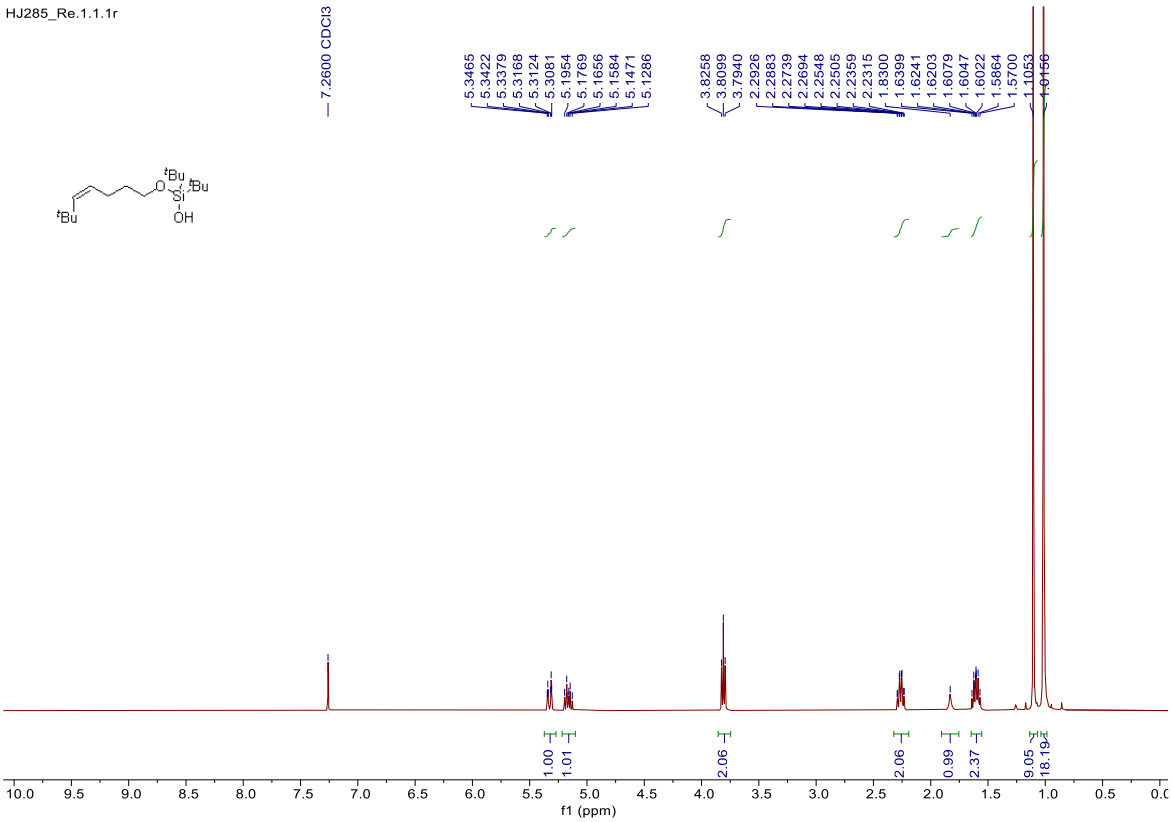


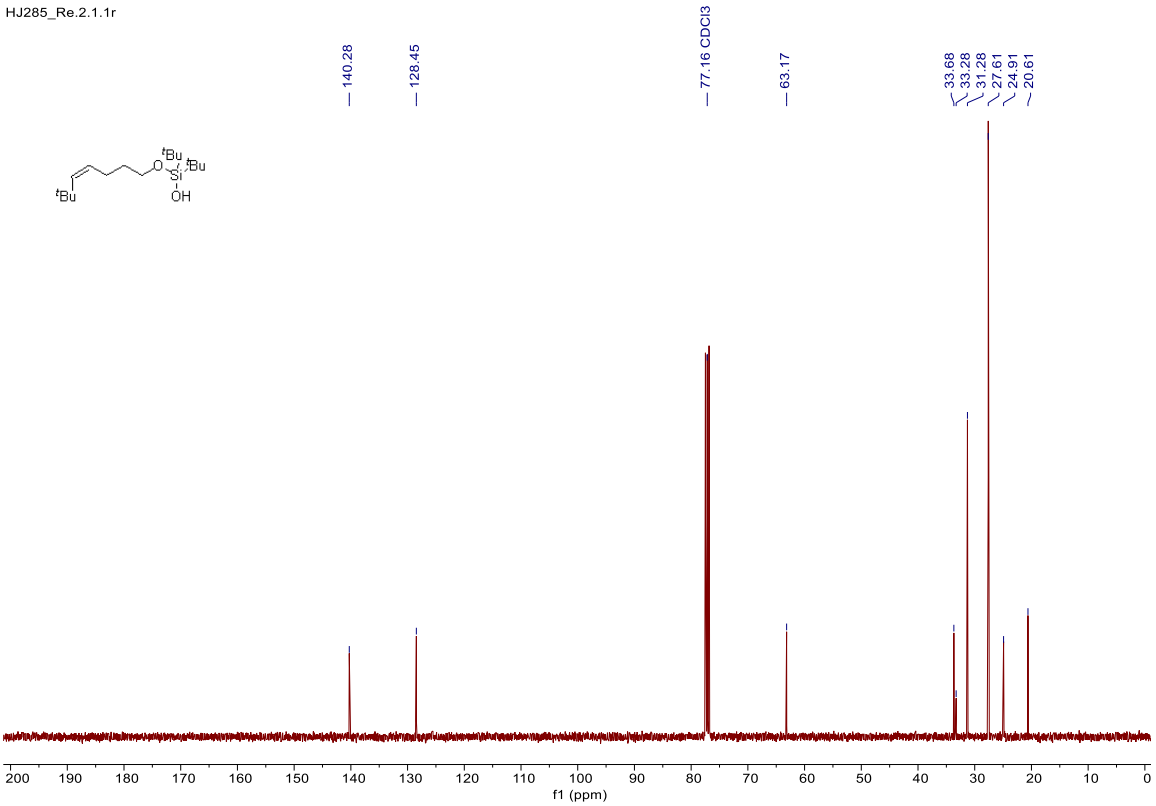
Compound S35 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



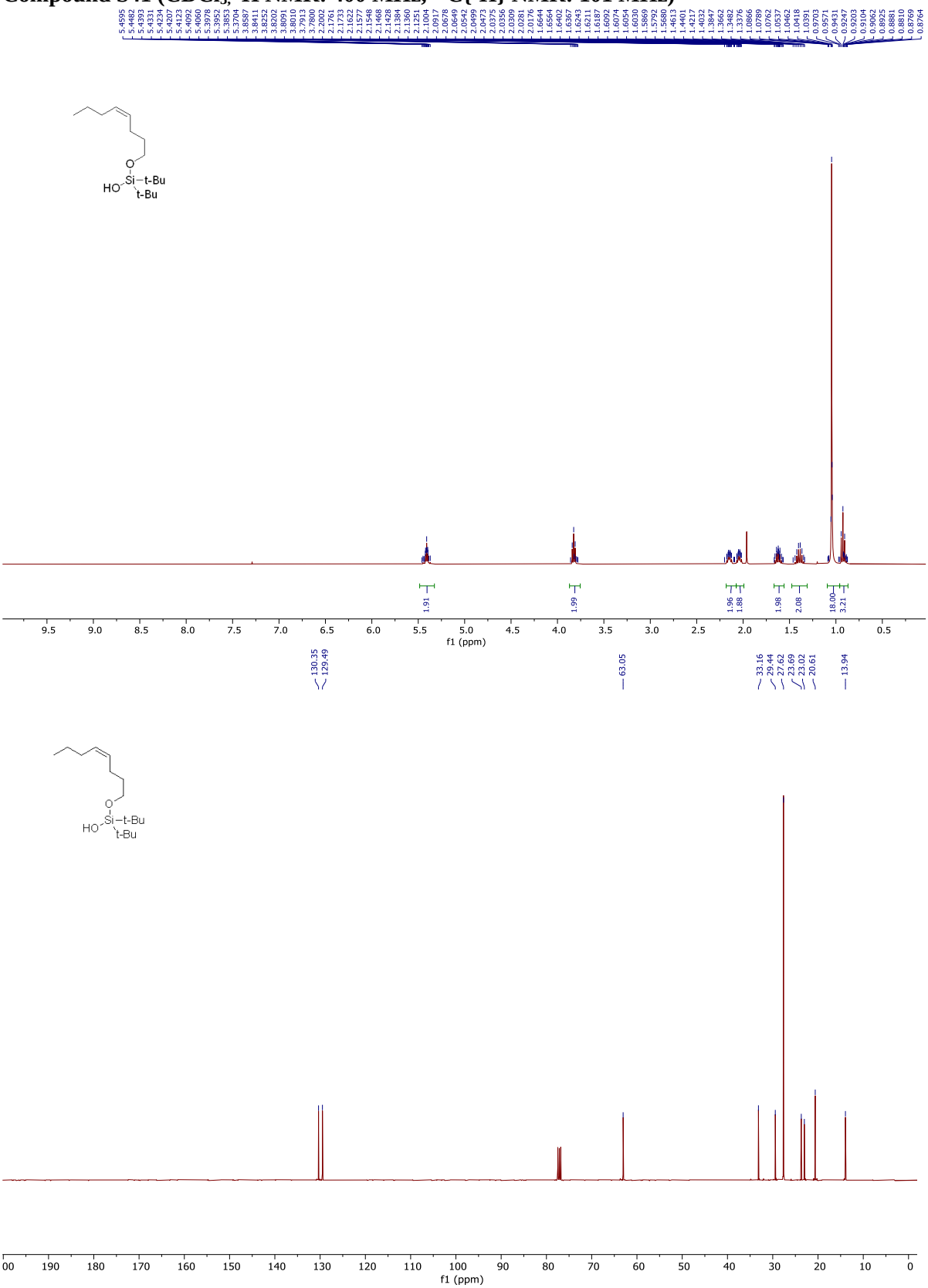


Compound S39 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

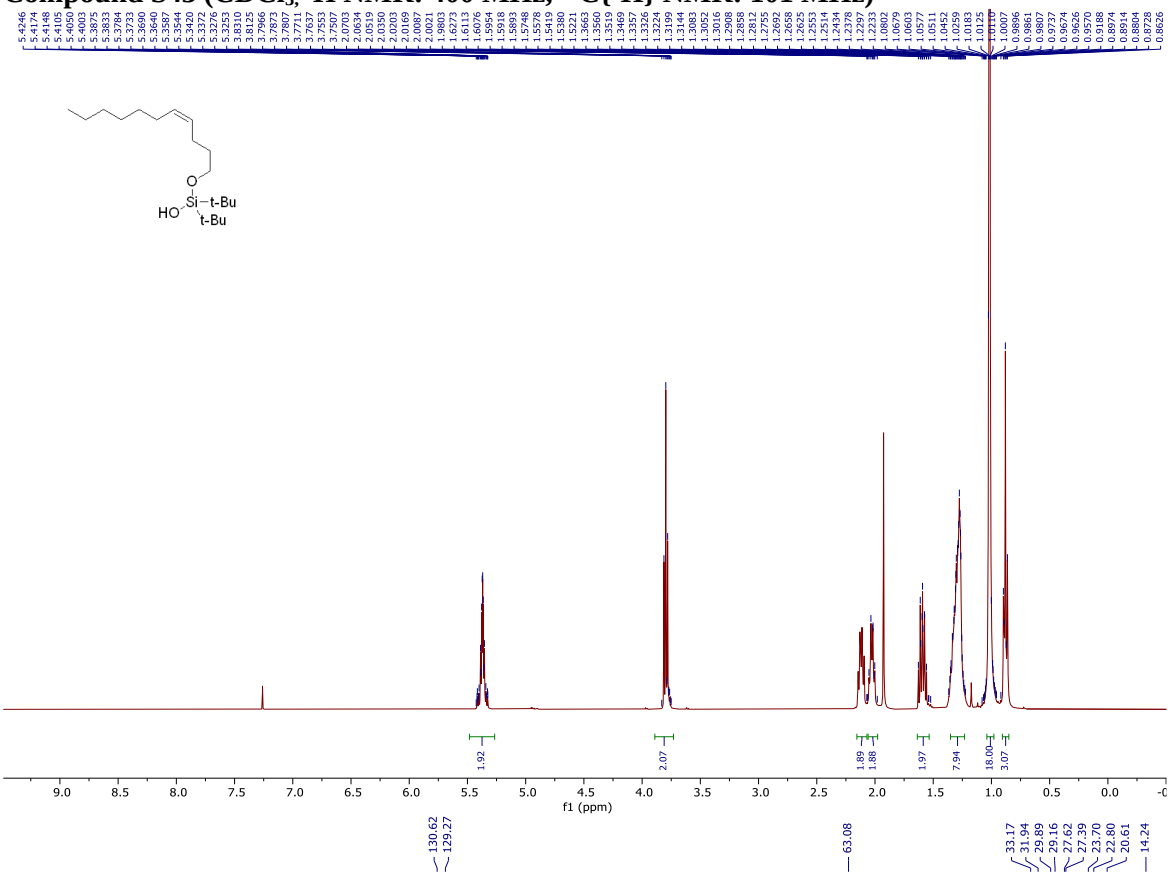


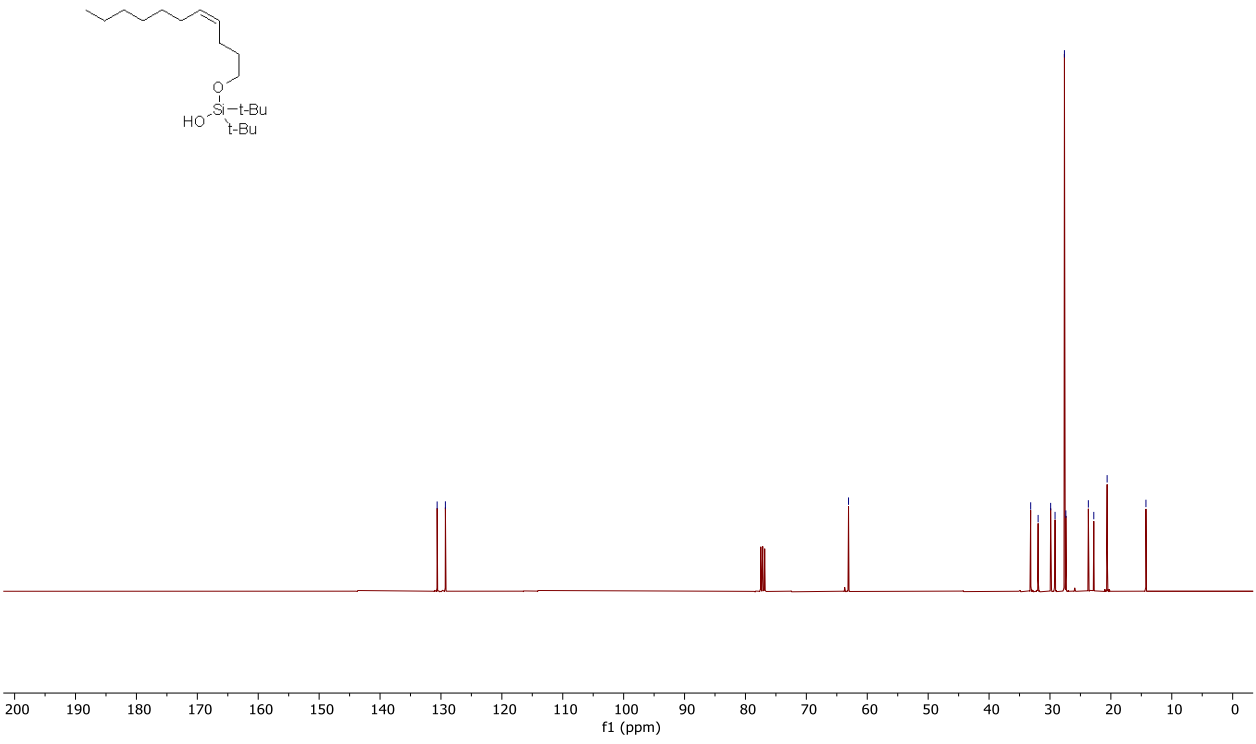


Compound S41 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

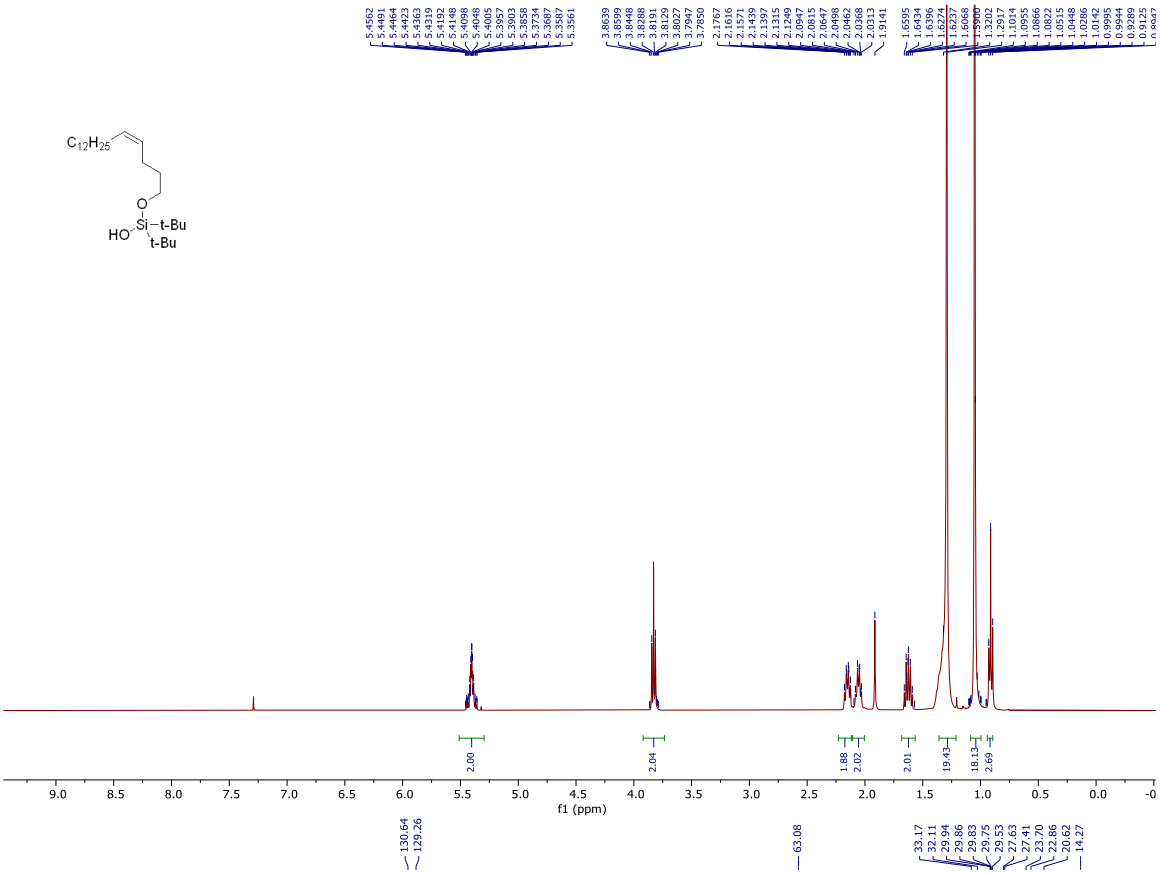


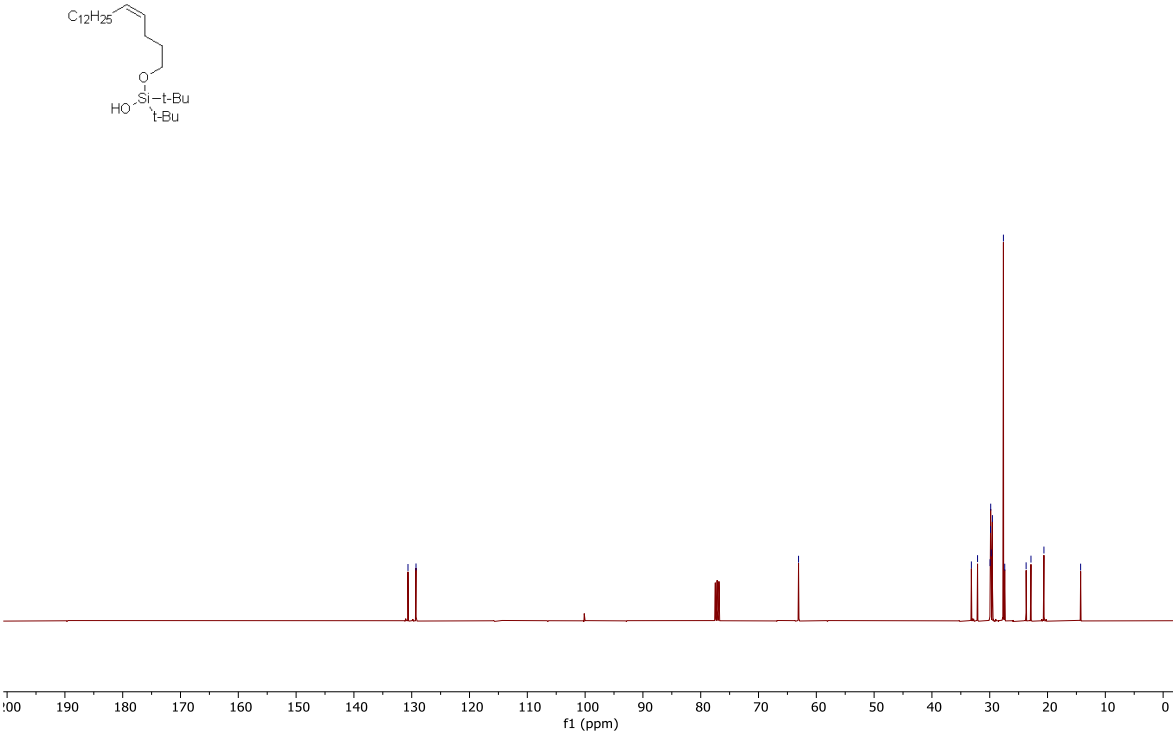
Compound S43 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



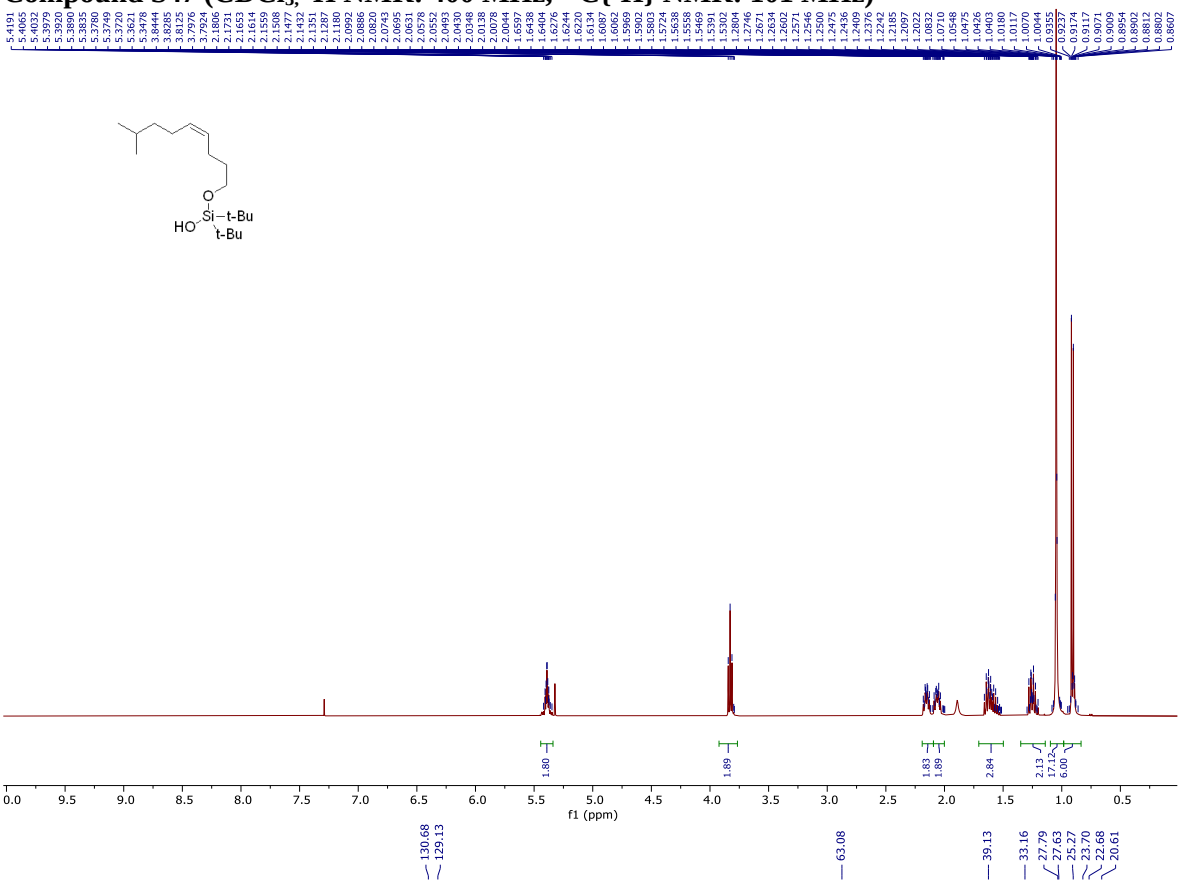


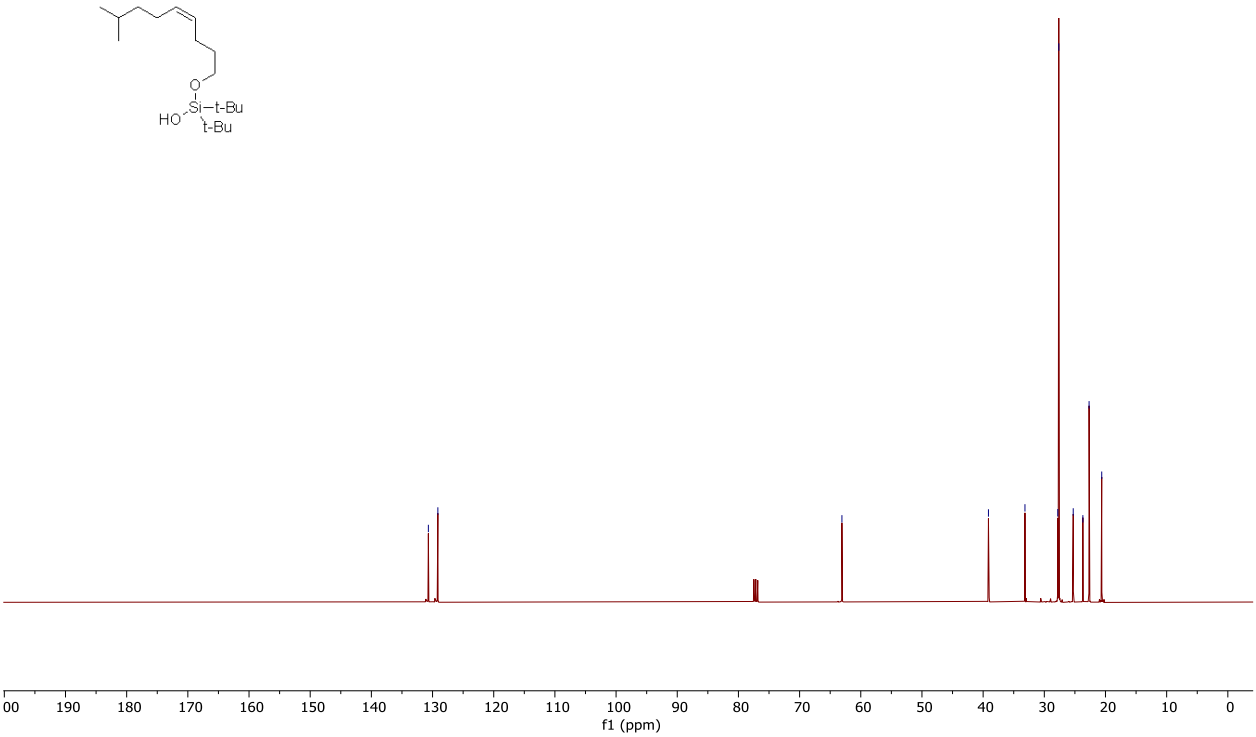
Compound S45 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



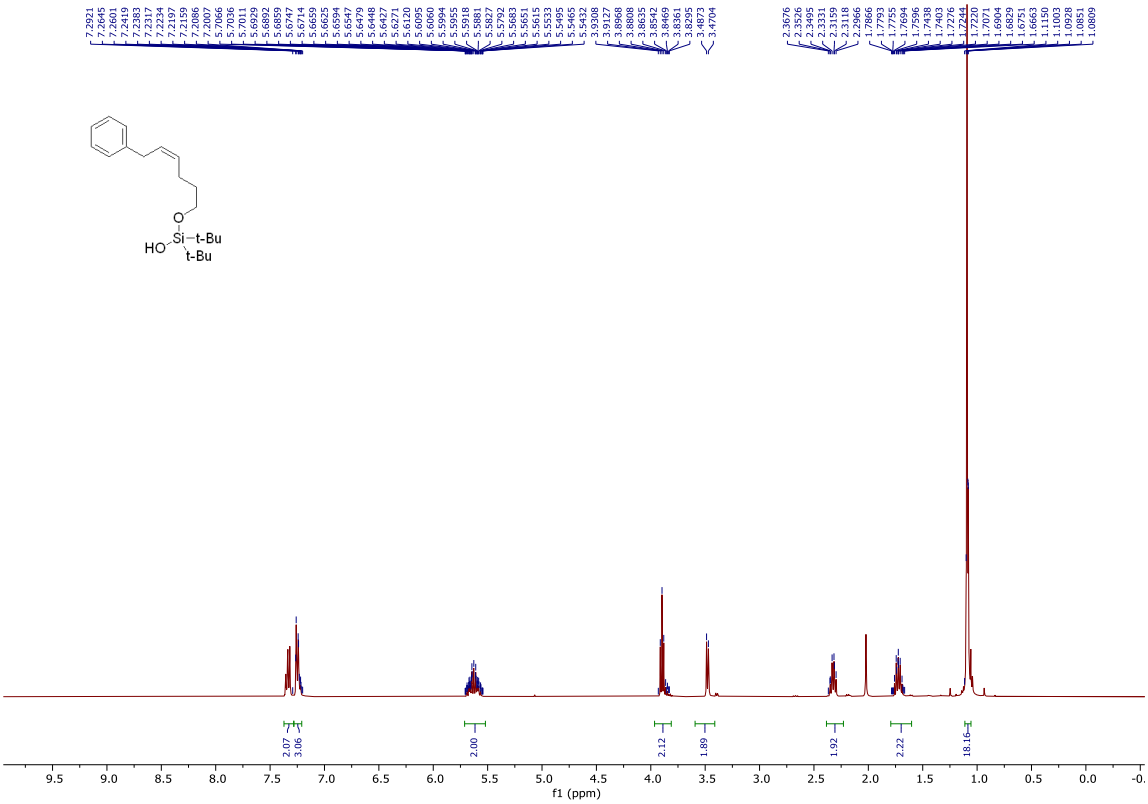


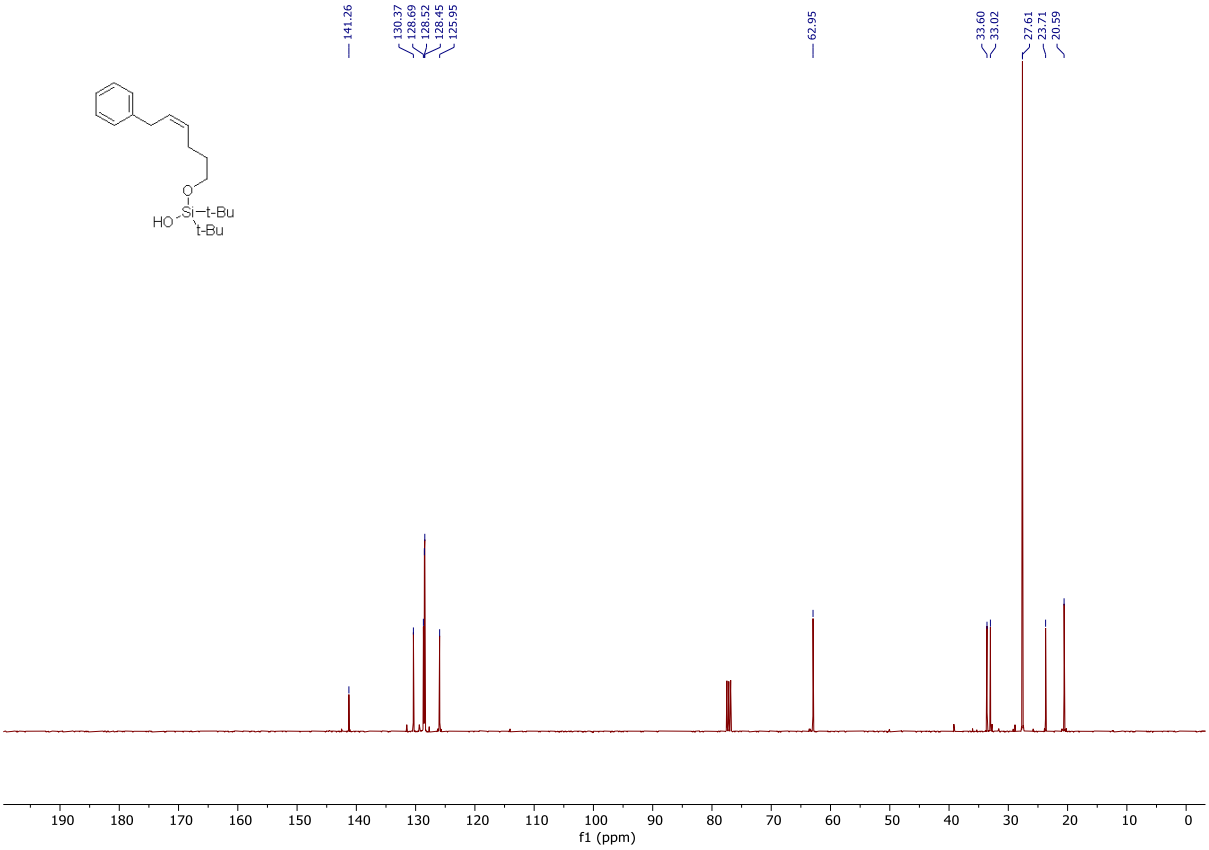
Compound S47 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



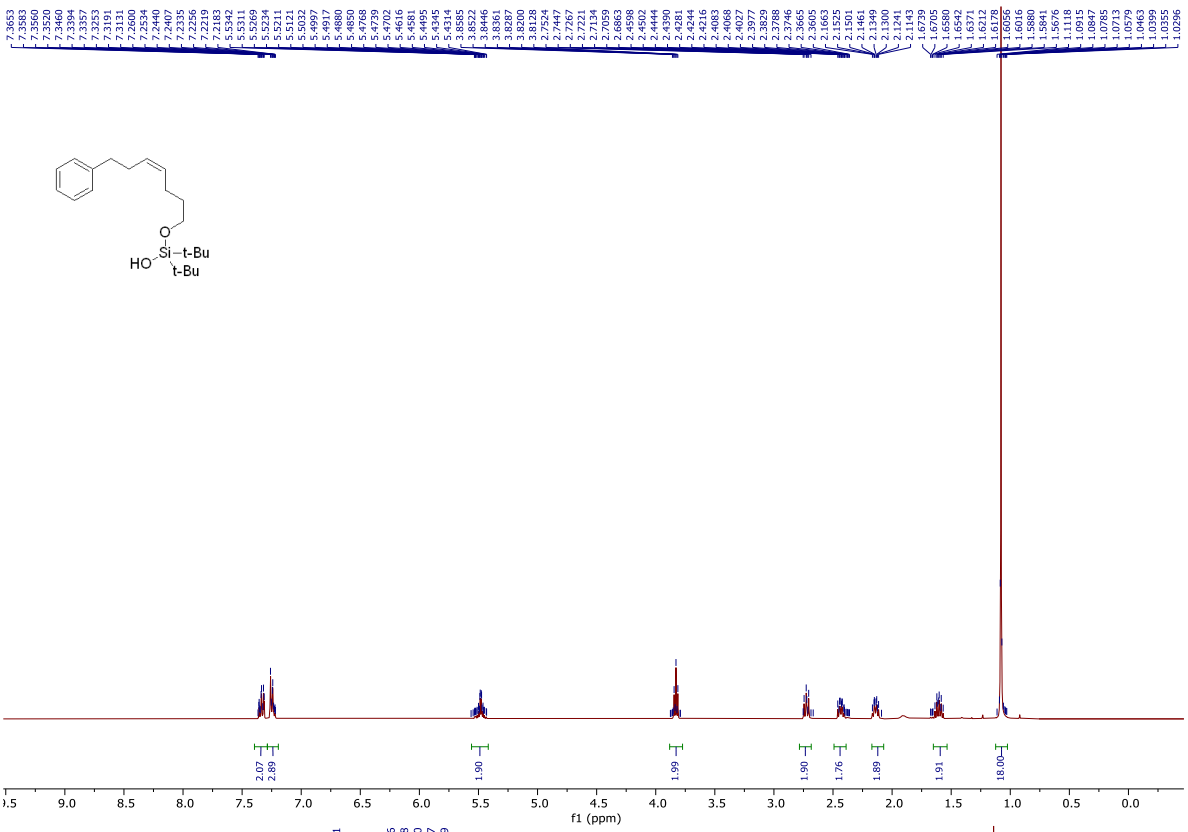


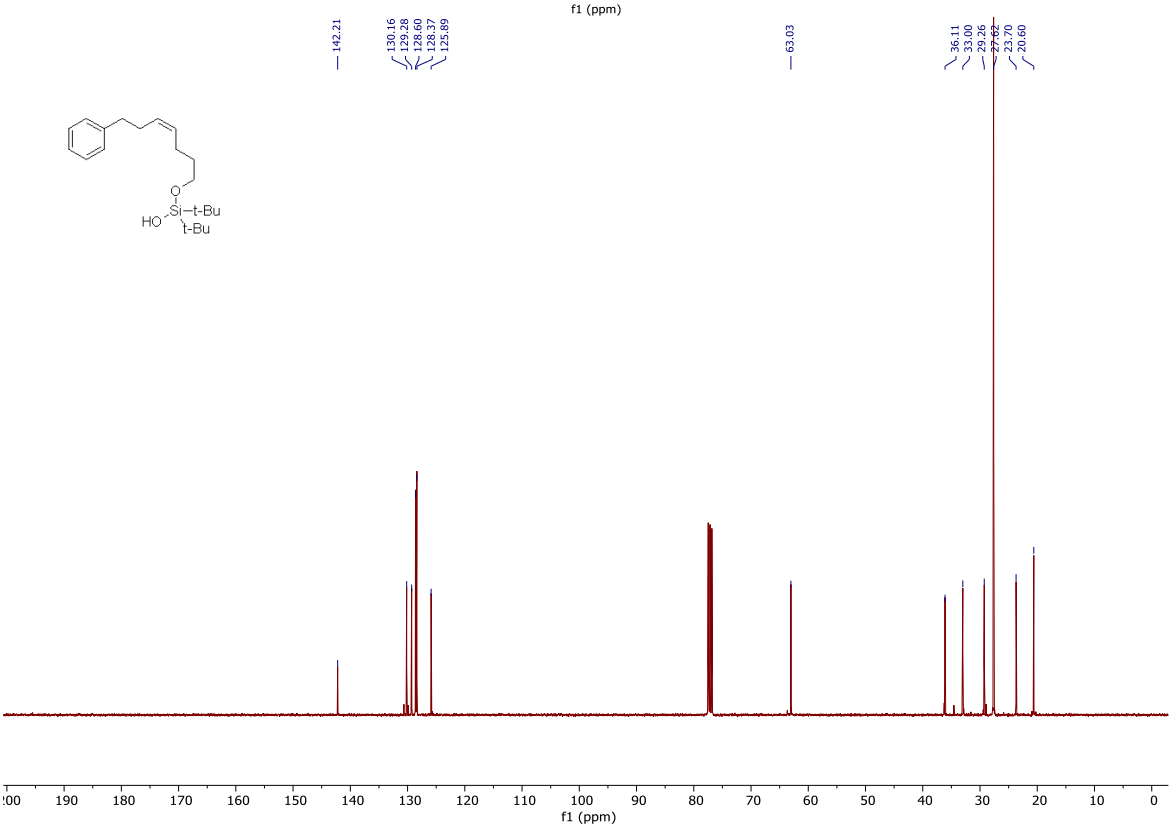
Compound S49 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



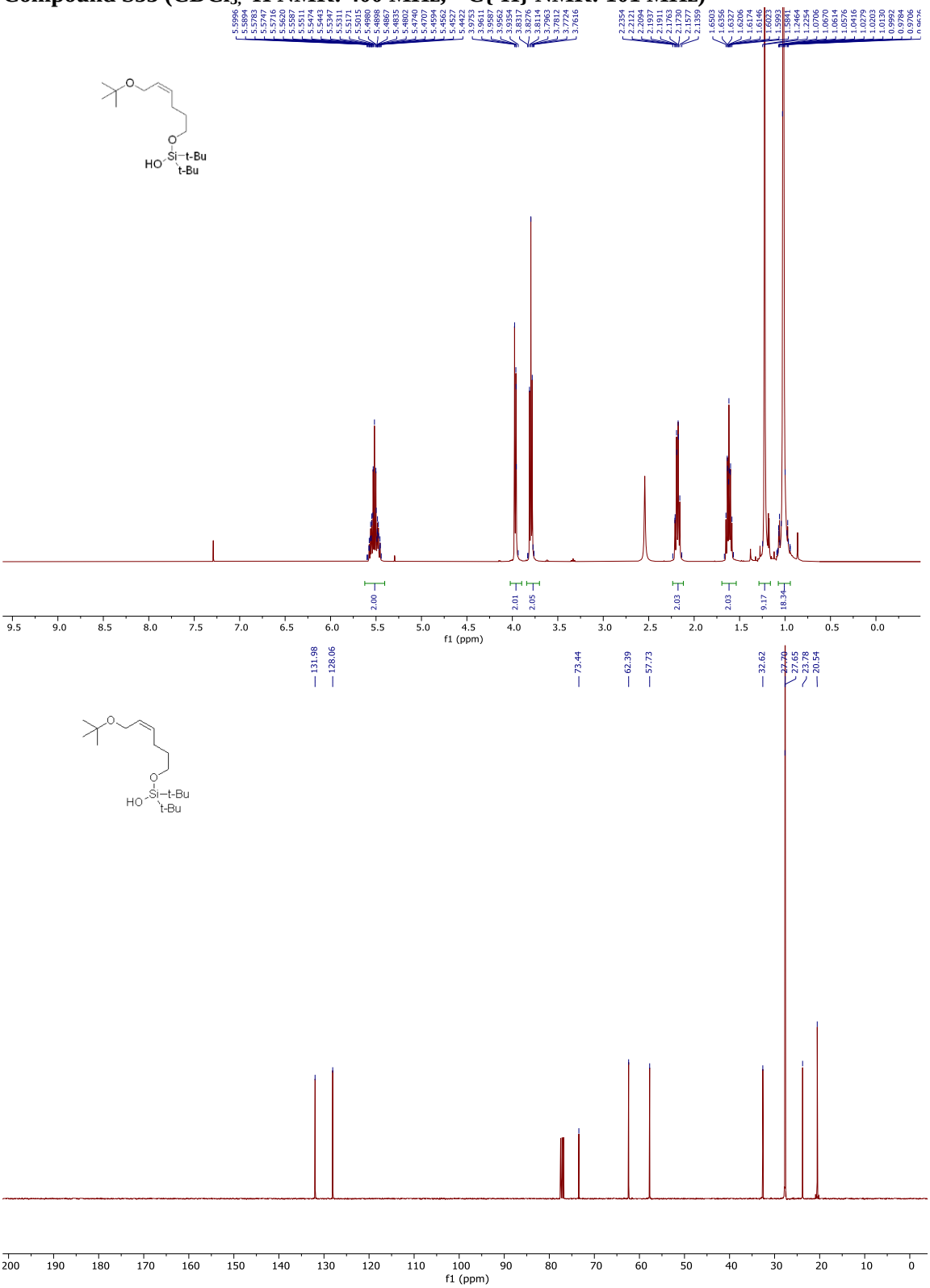


Compound S51 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

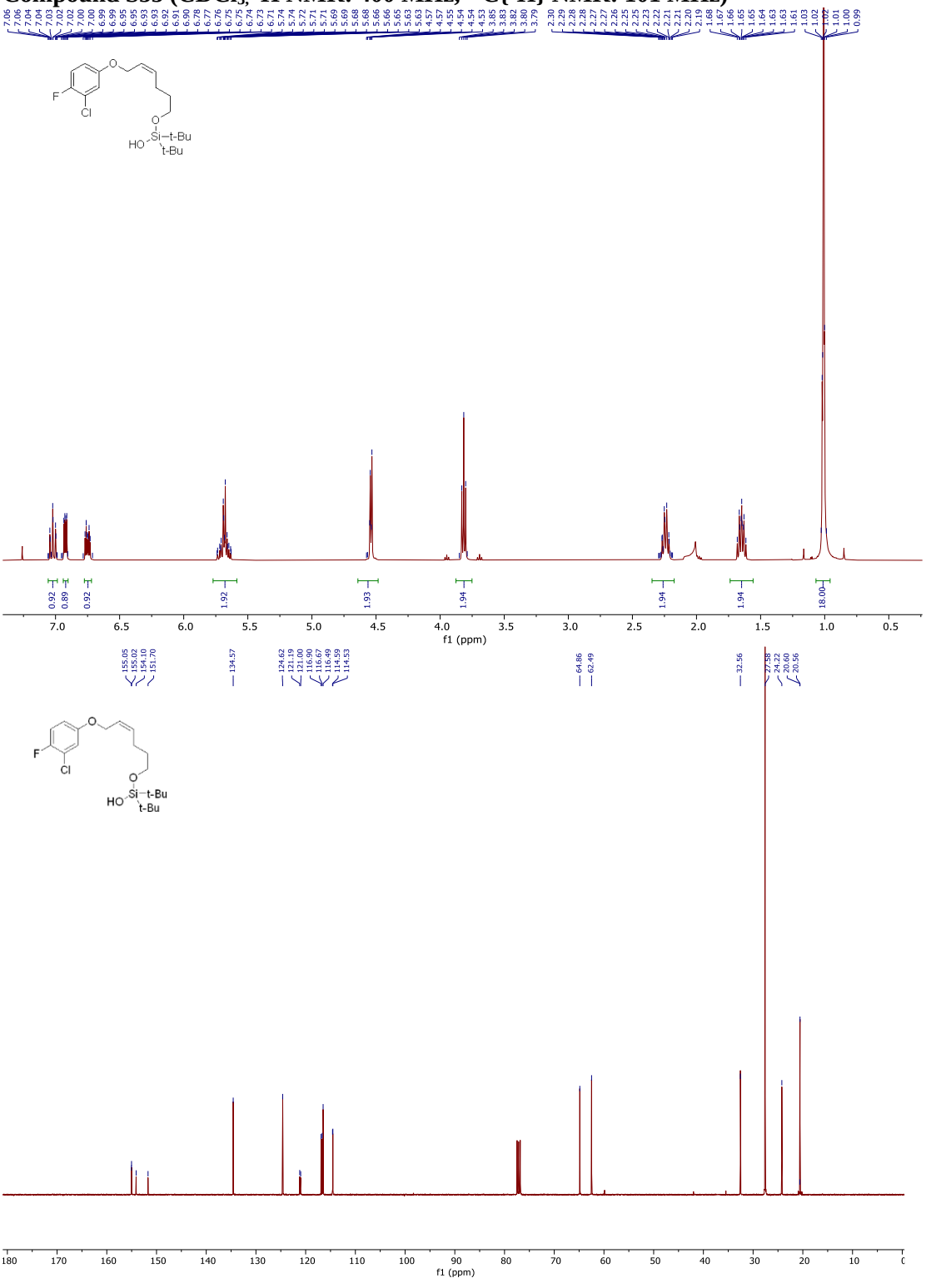




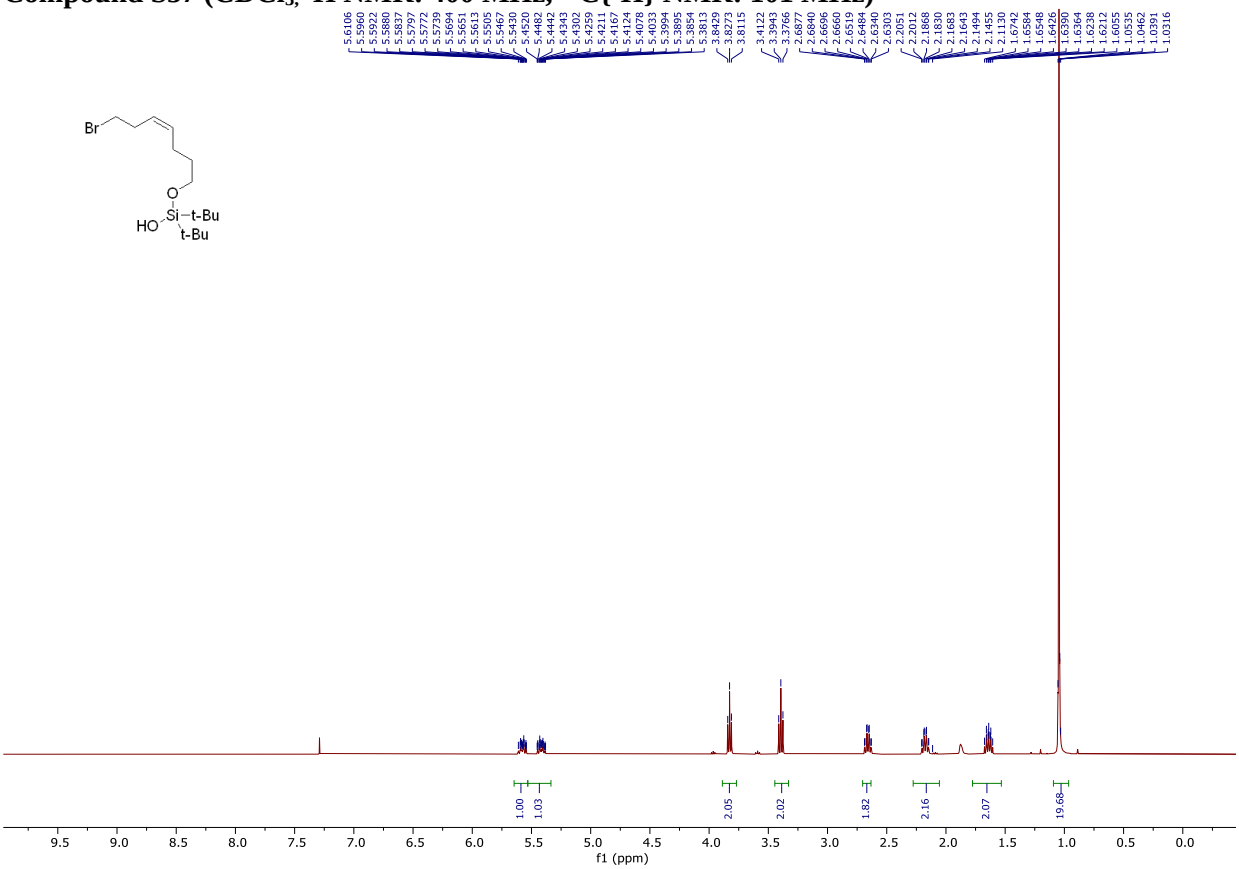
Compound S53 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

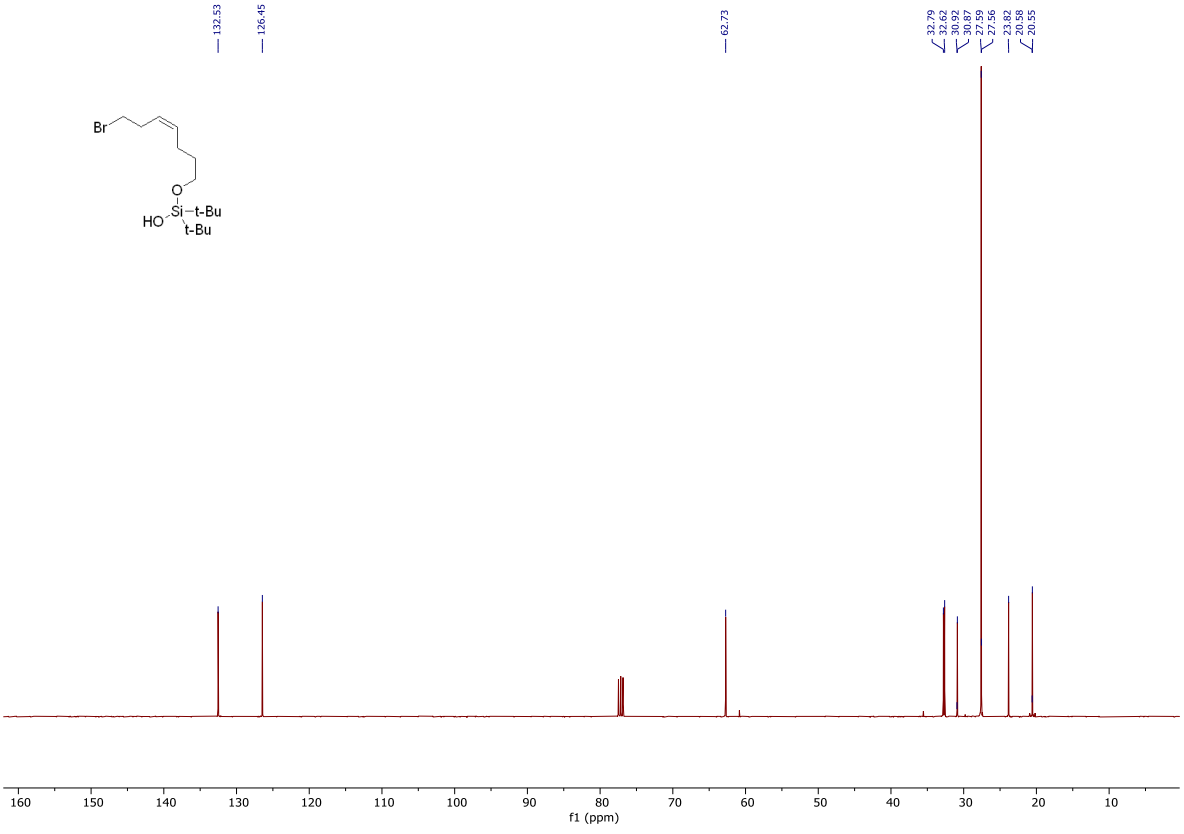


Compound S55 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

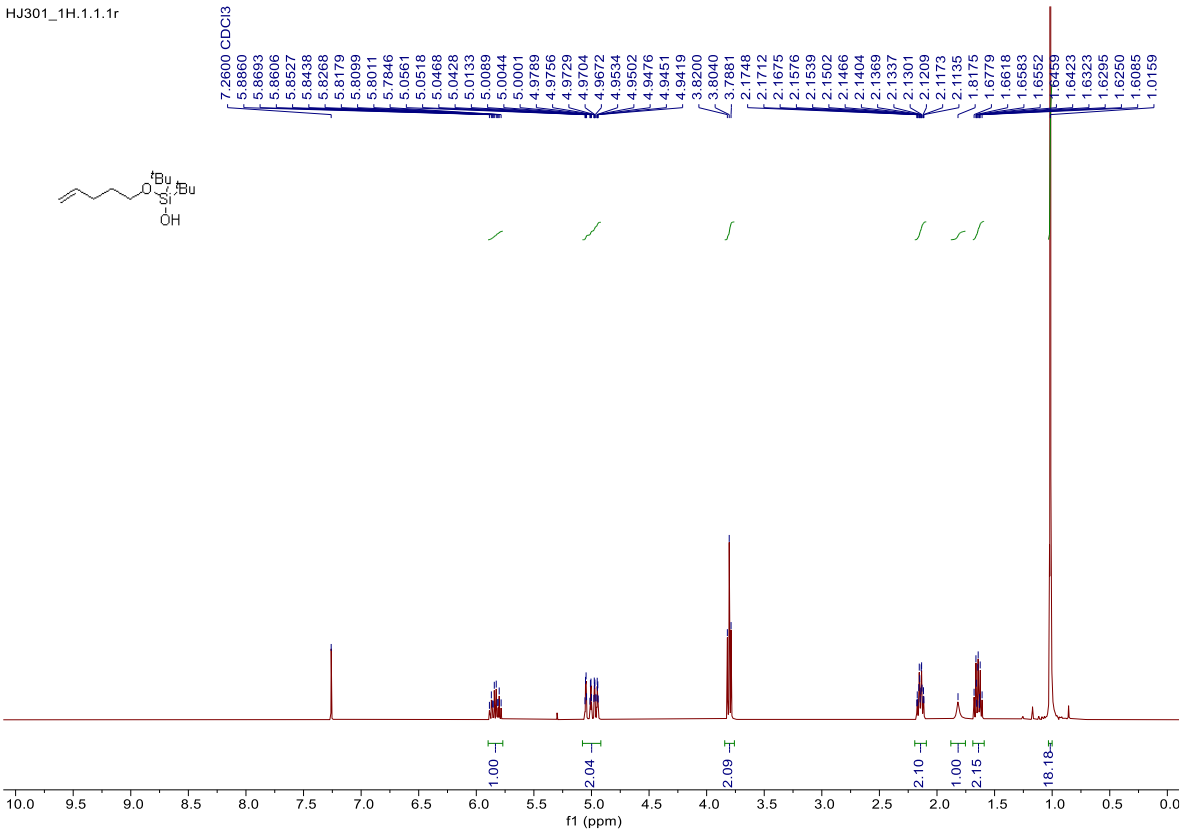


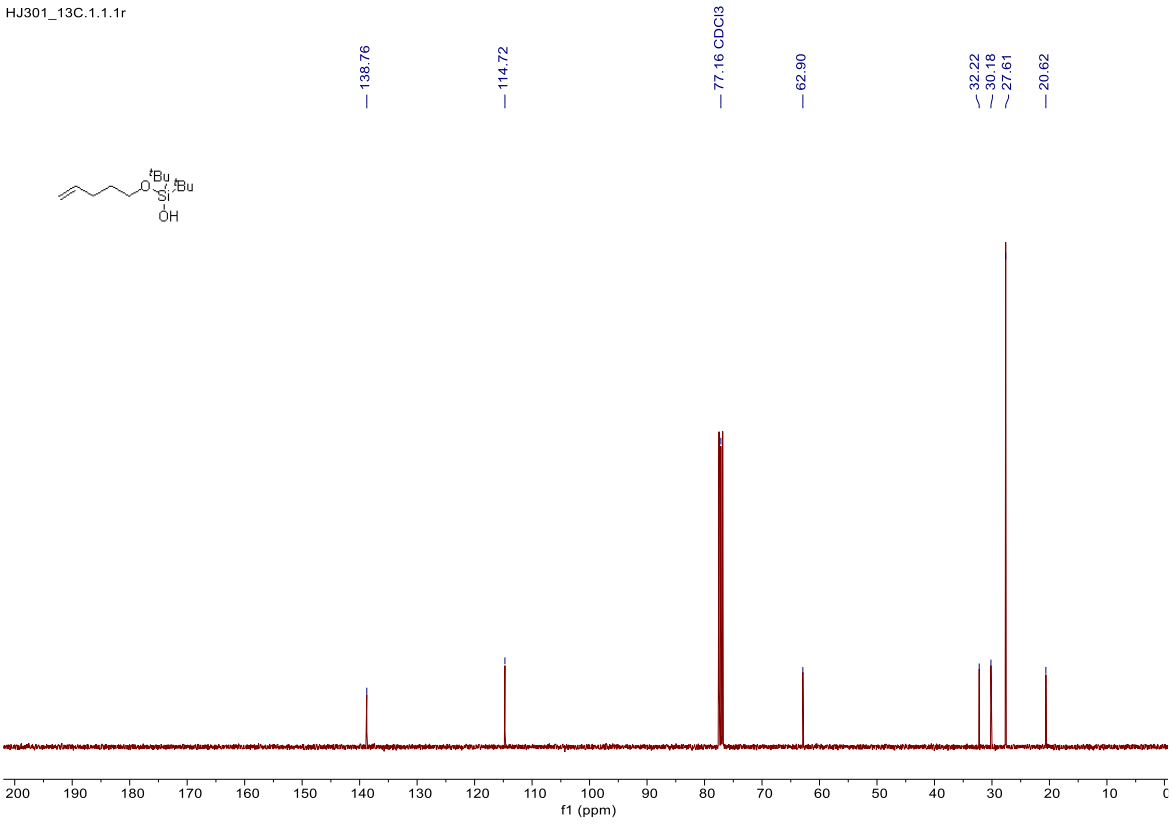
Compound S57 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



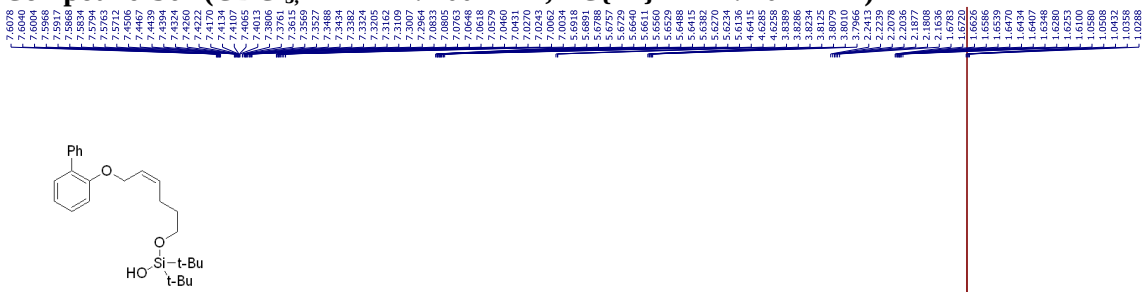


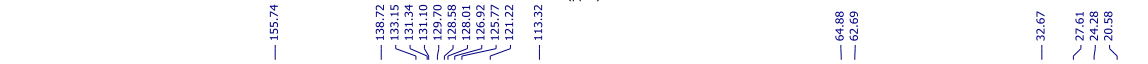
Compound S59 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

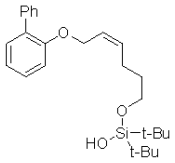


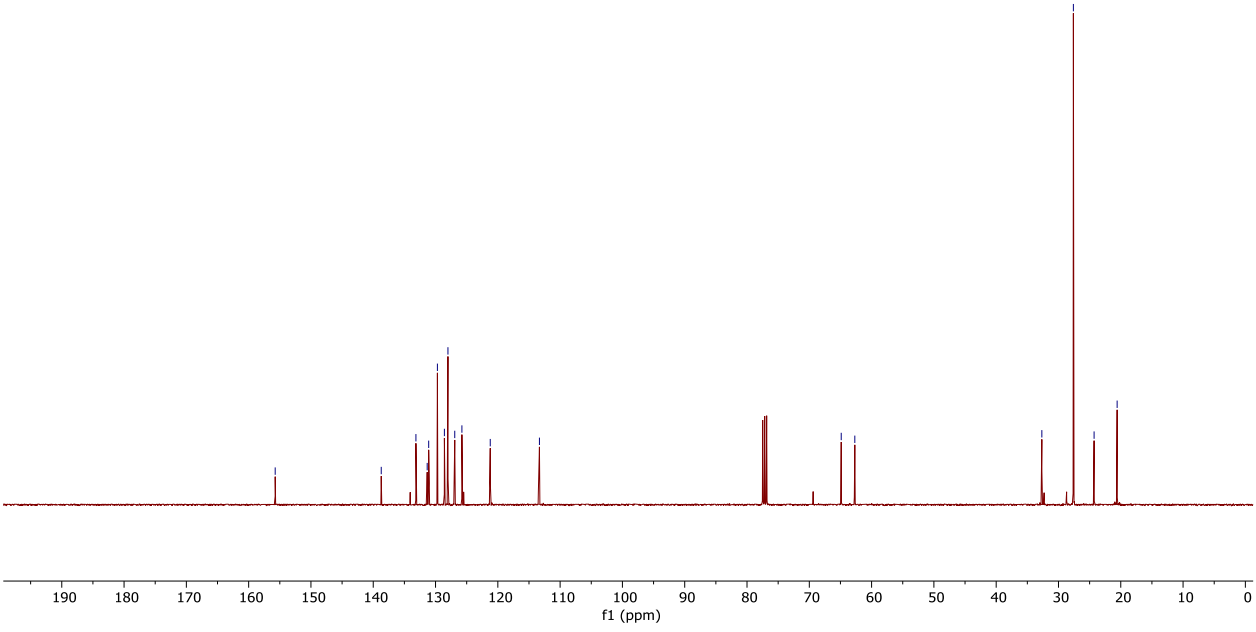


Compound S61 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

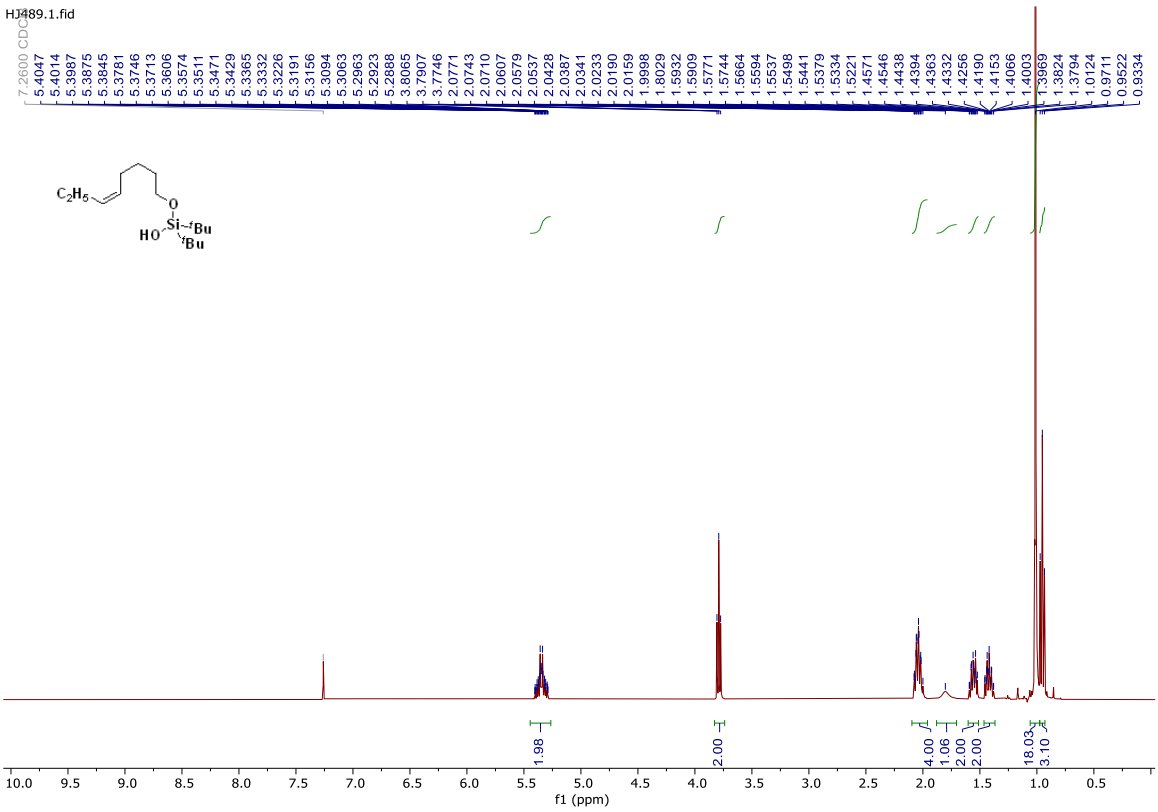


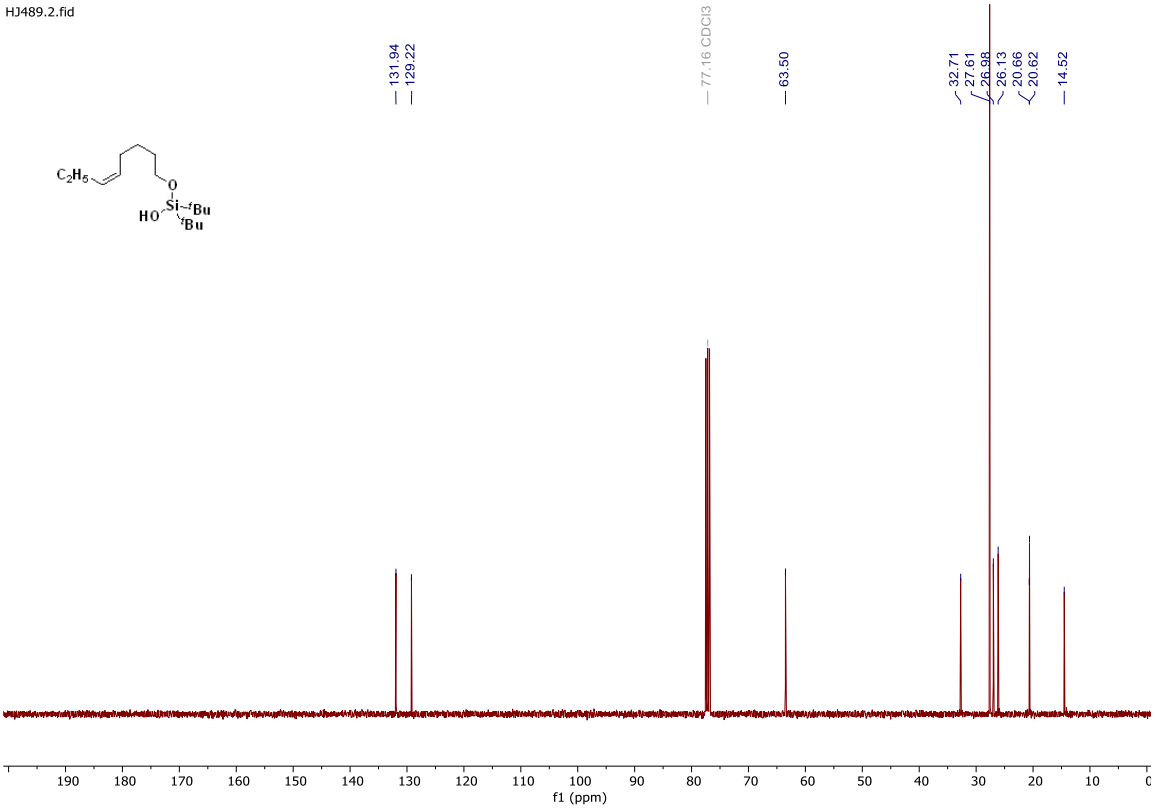




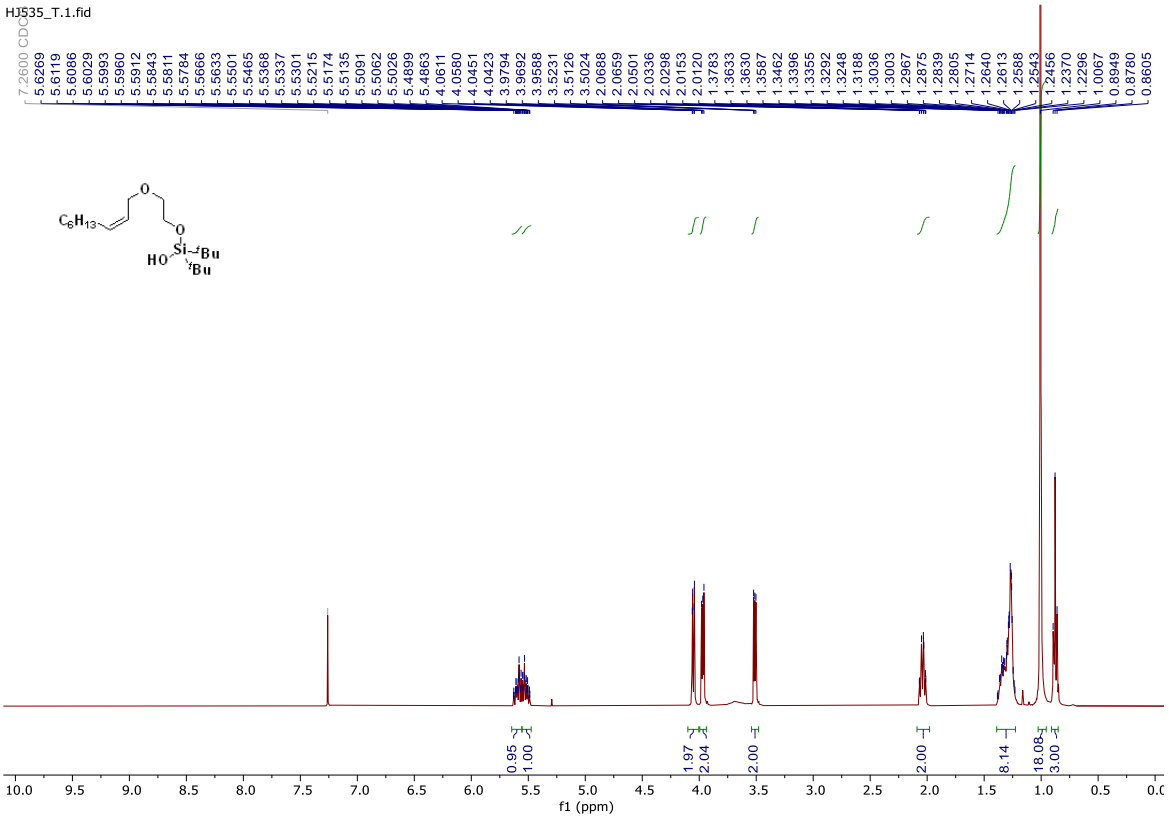


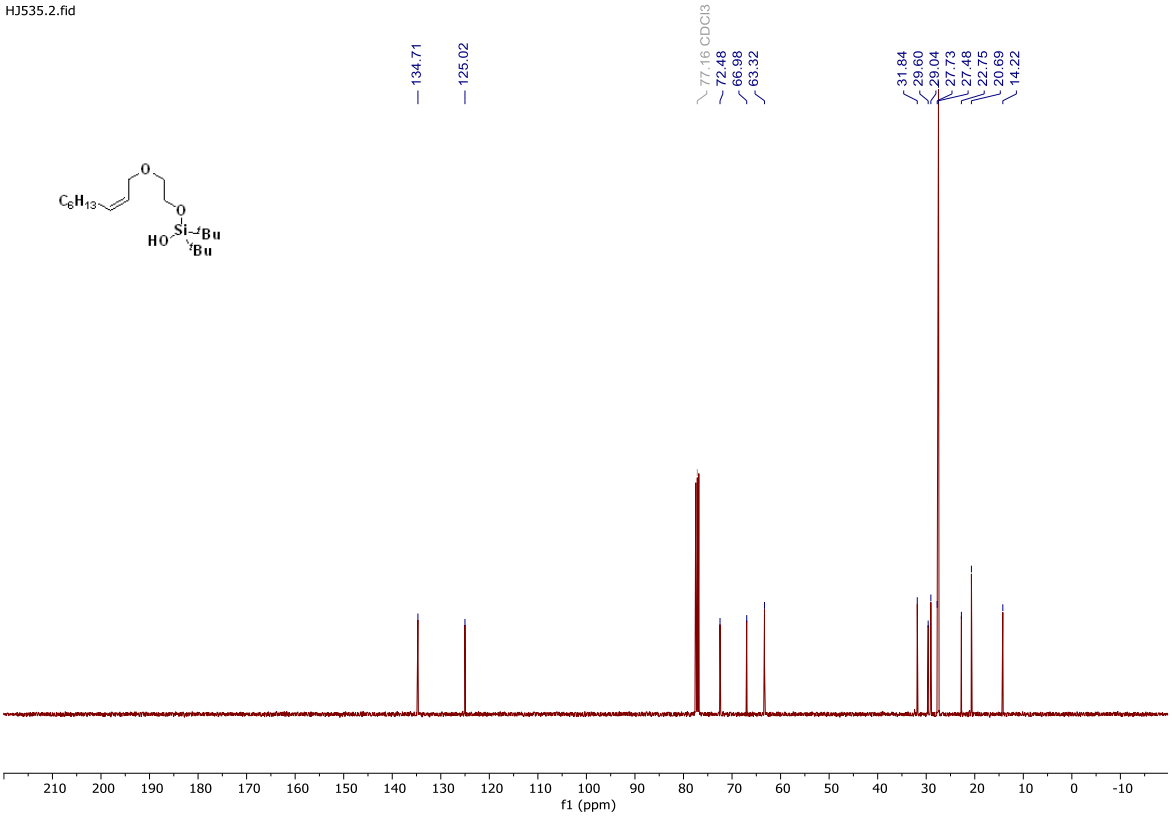
Compound S63 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



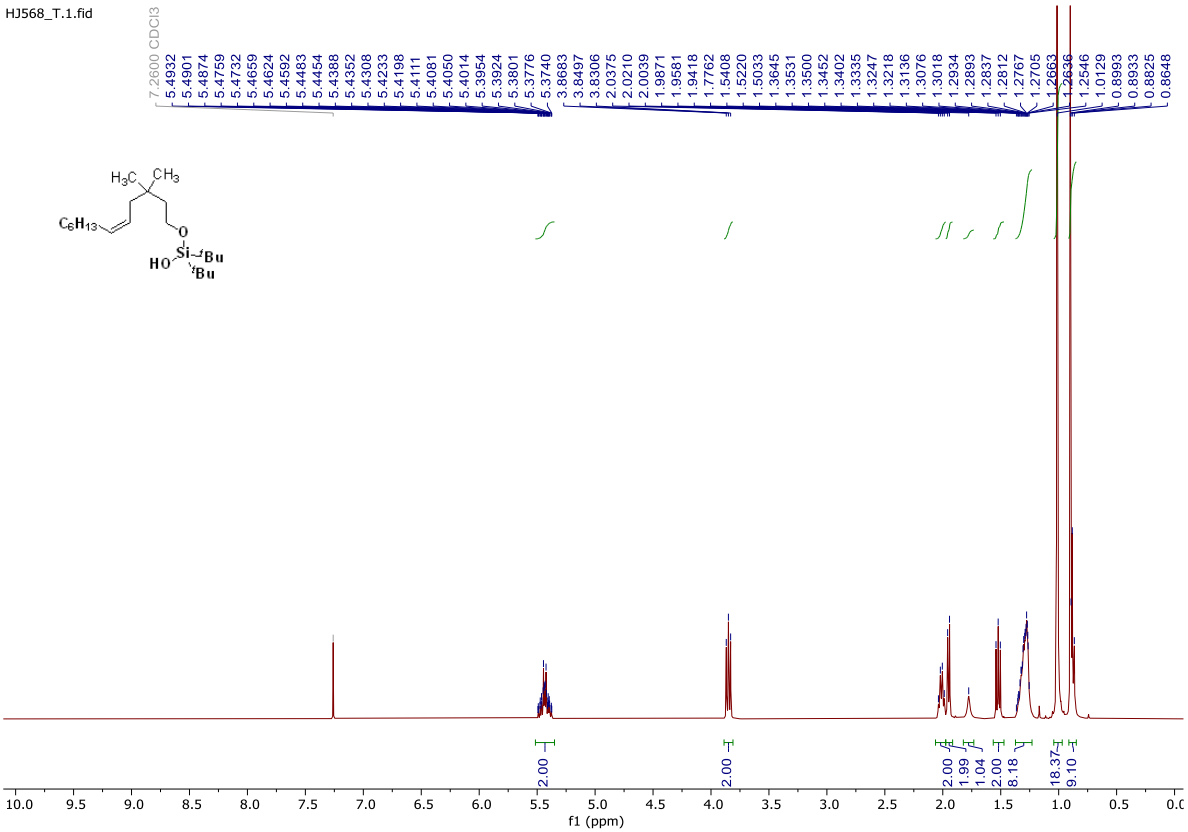


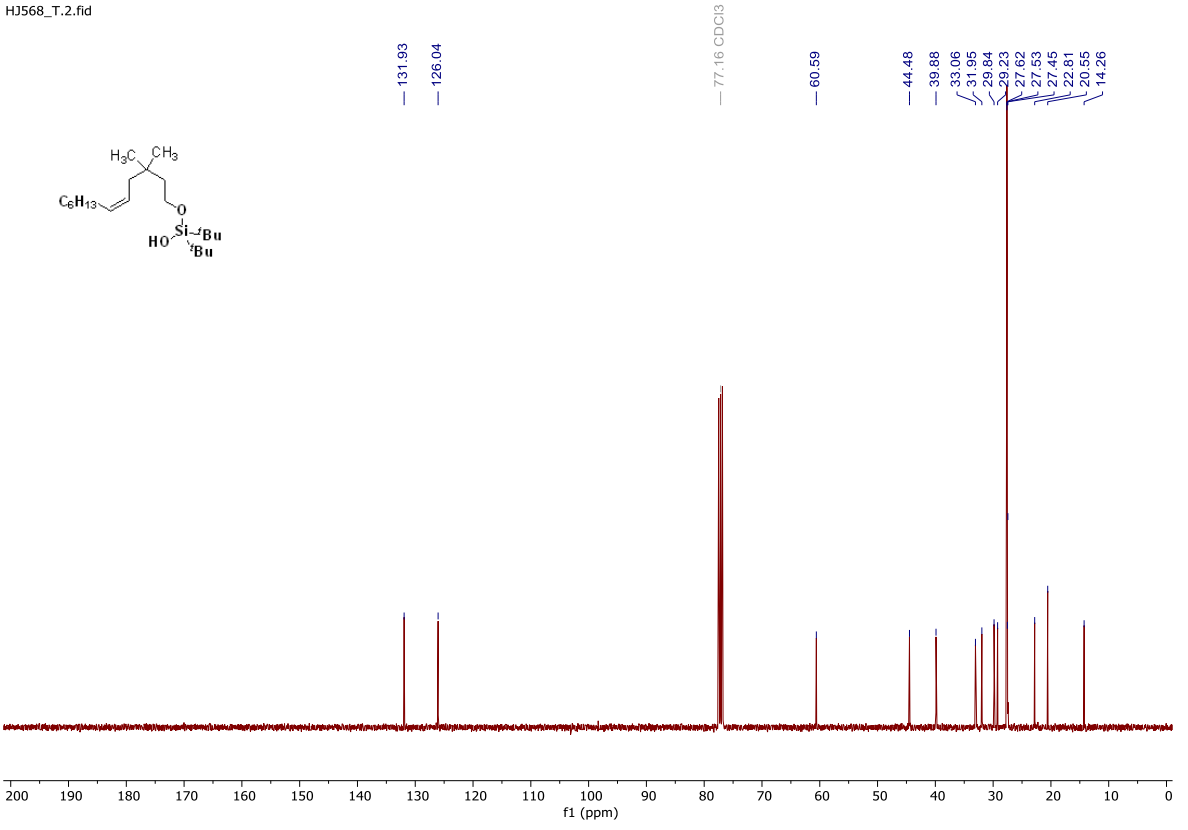
Compound S65 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



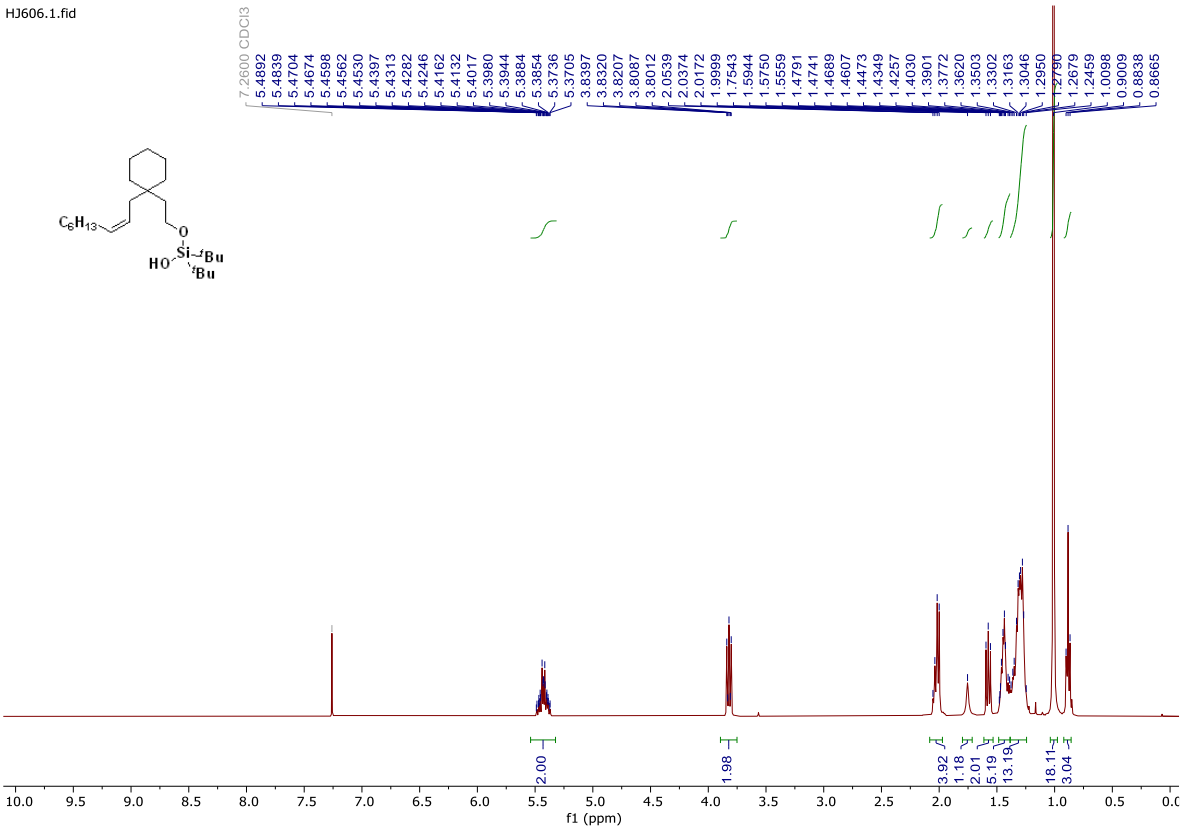


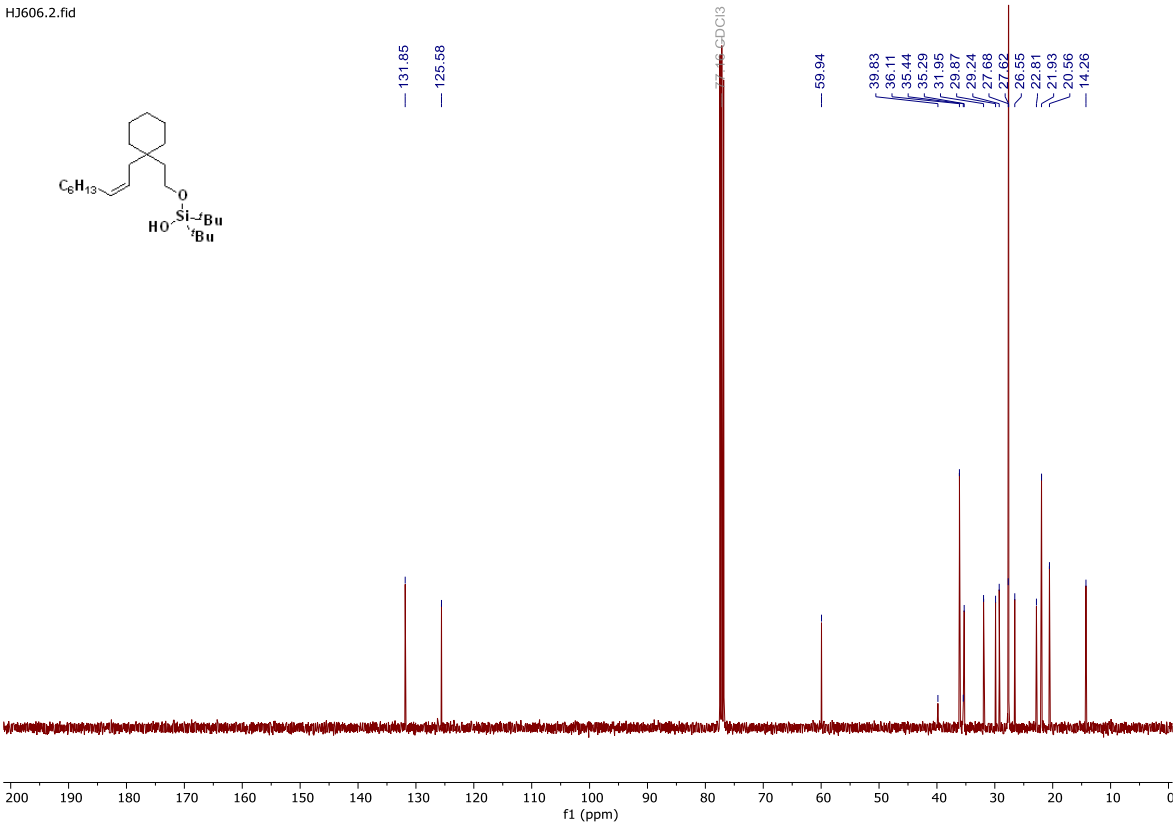
Compound S67 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



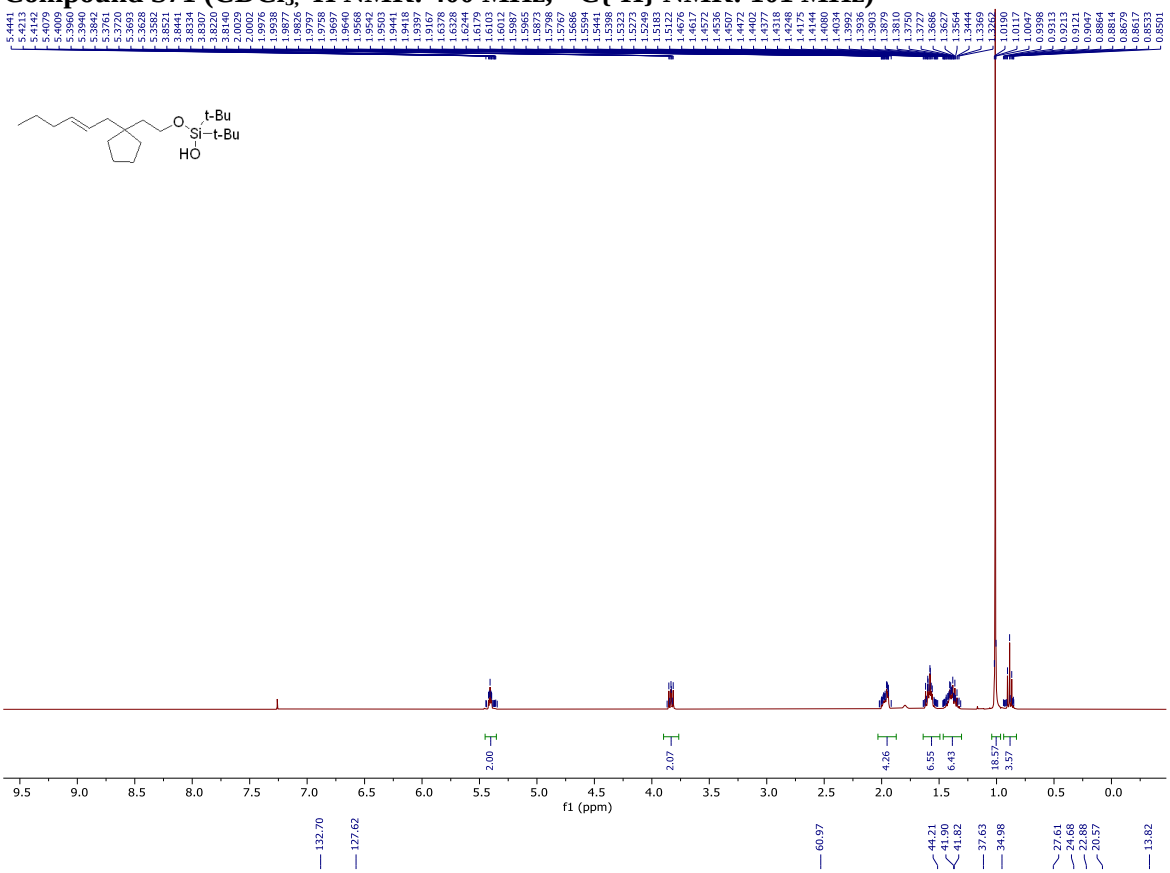


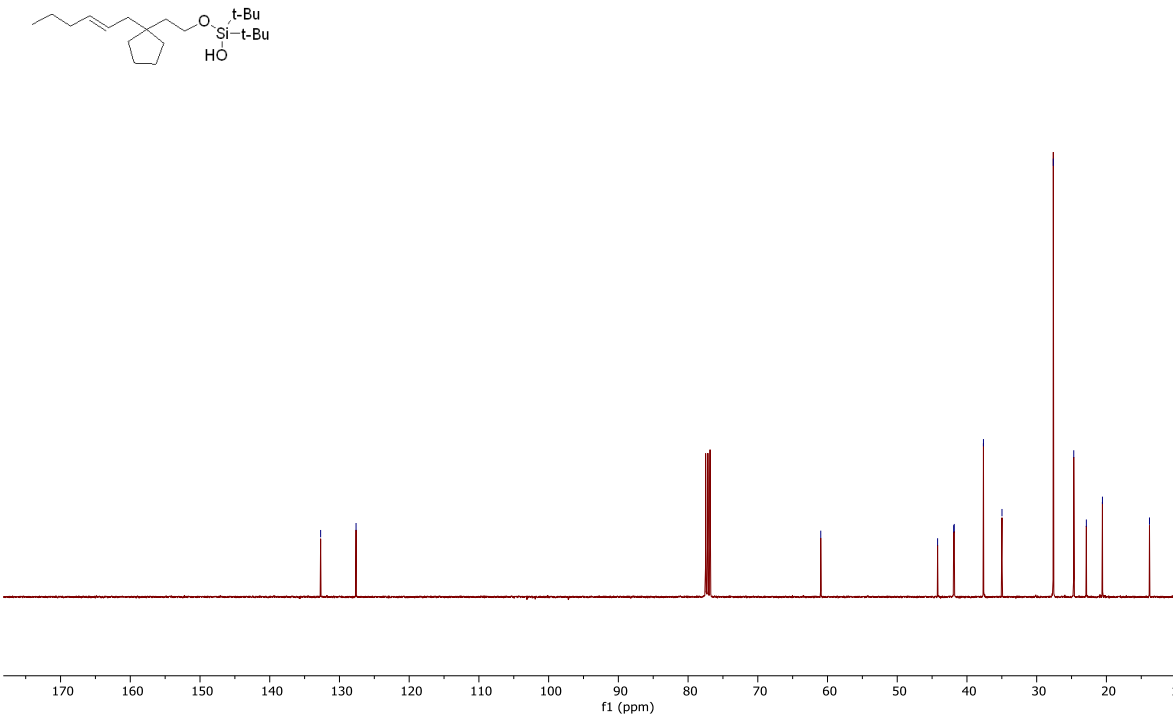
Compound S69 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



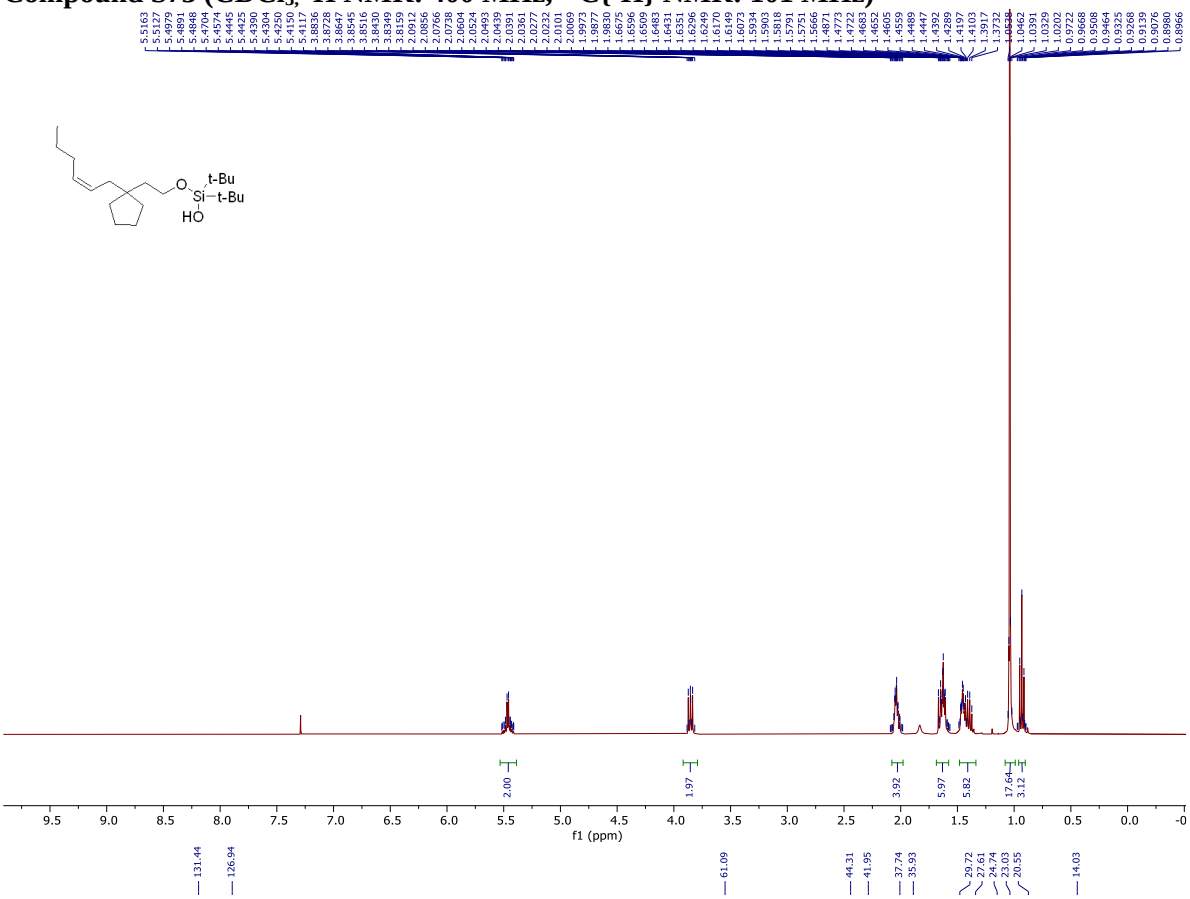


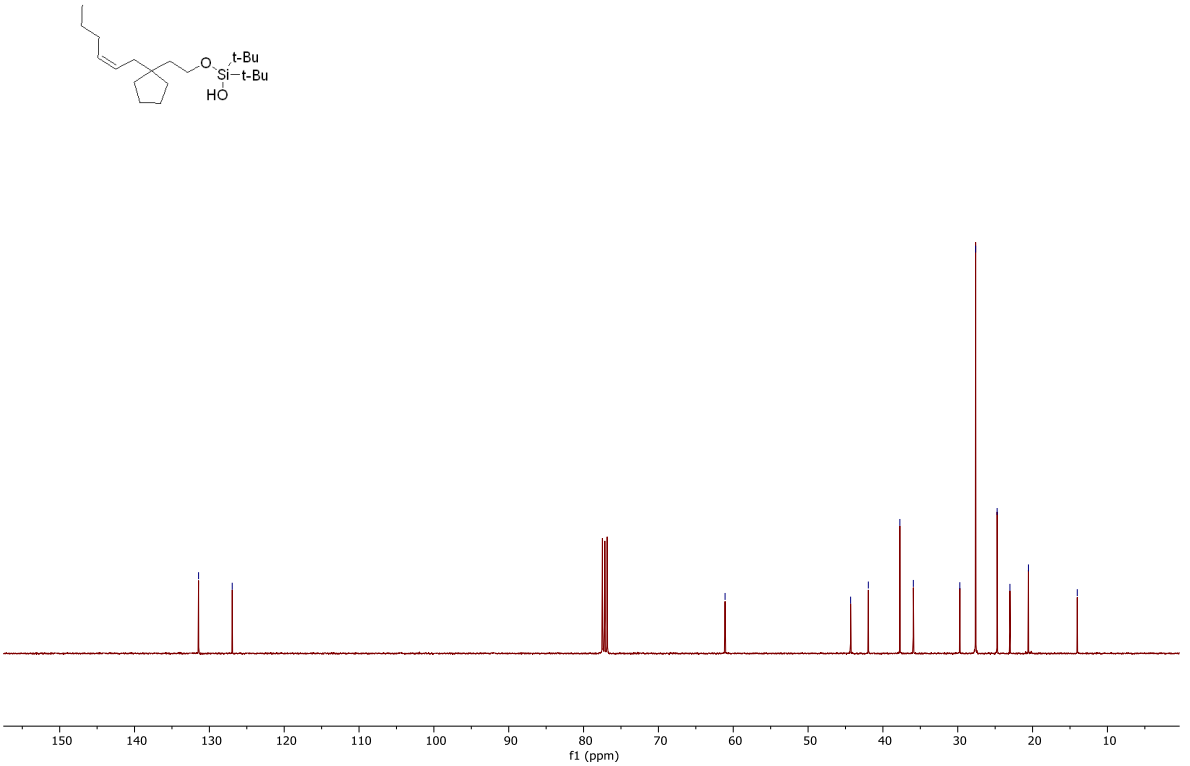
Compound S71 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



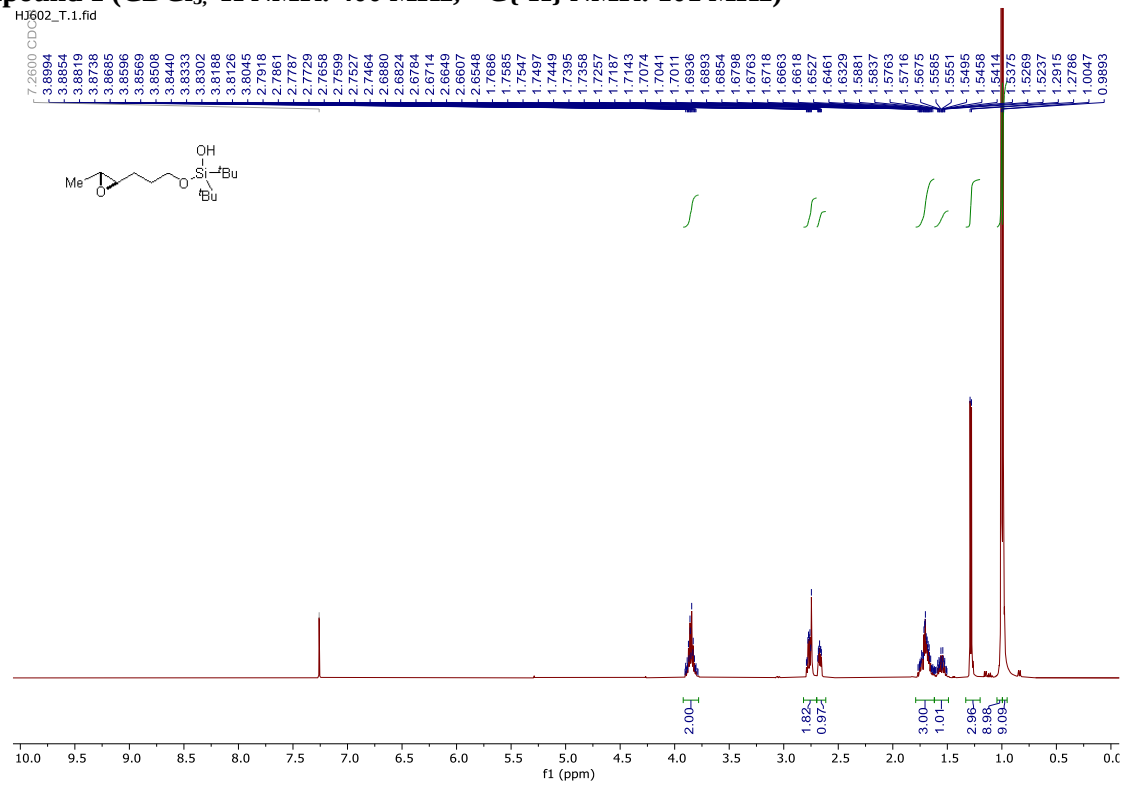


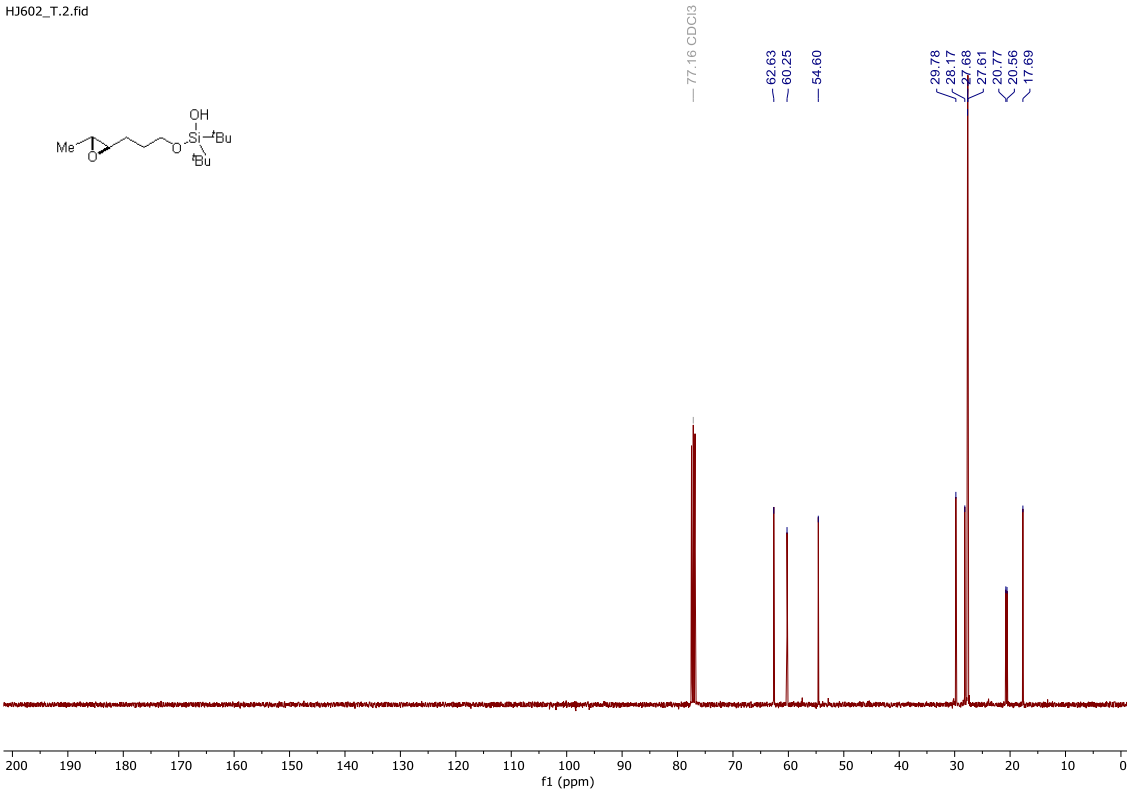
Compound S73 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



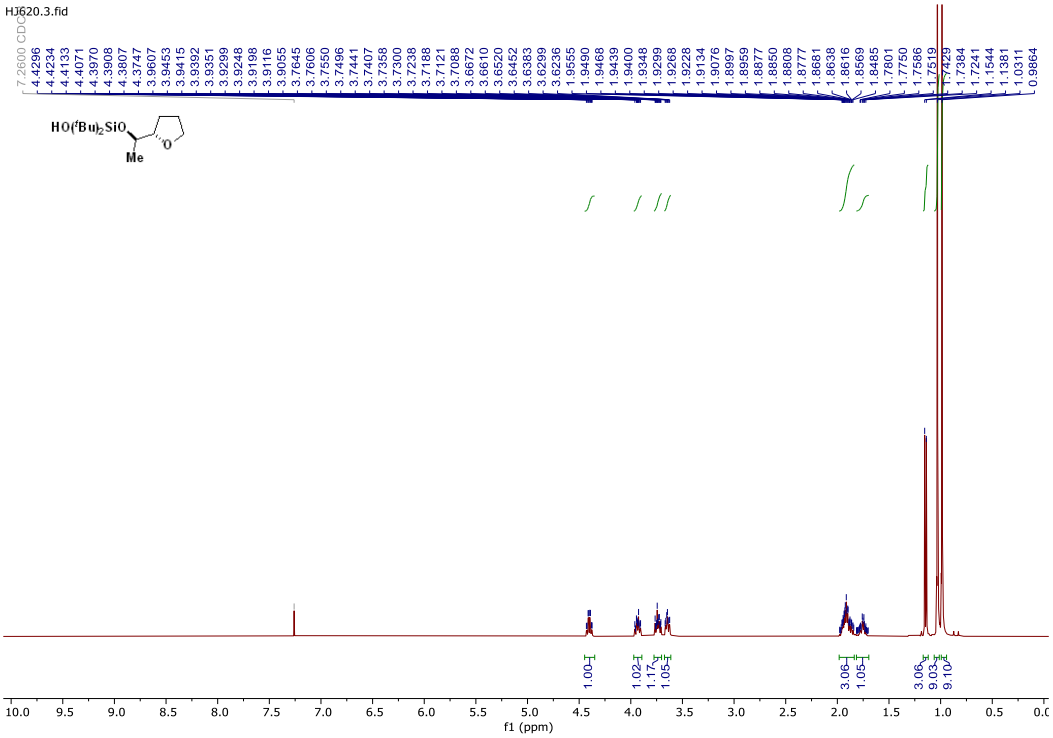


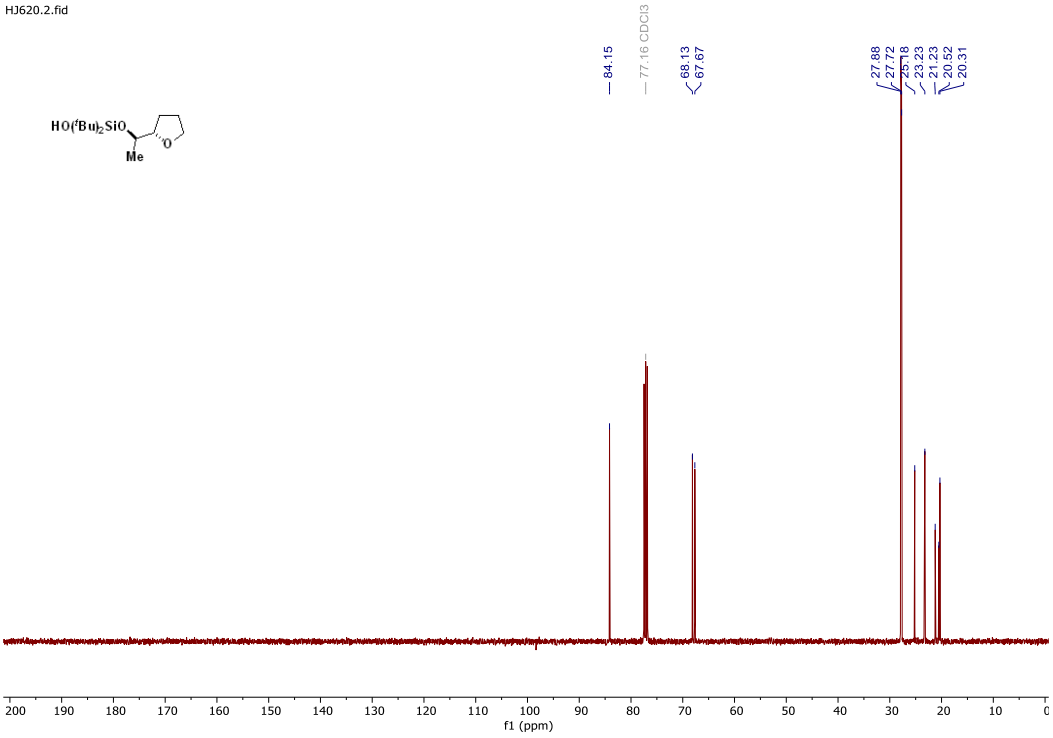
Compound 1 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



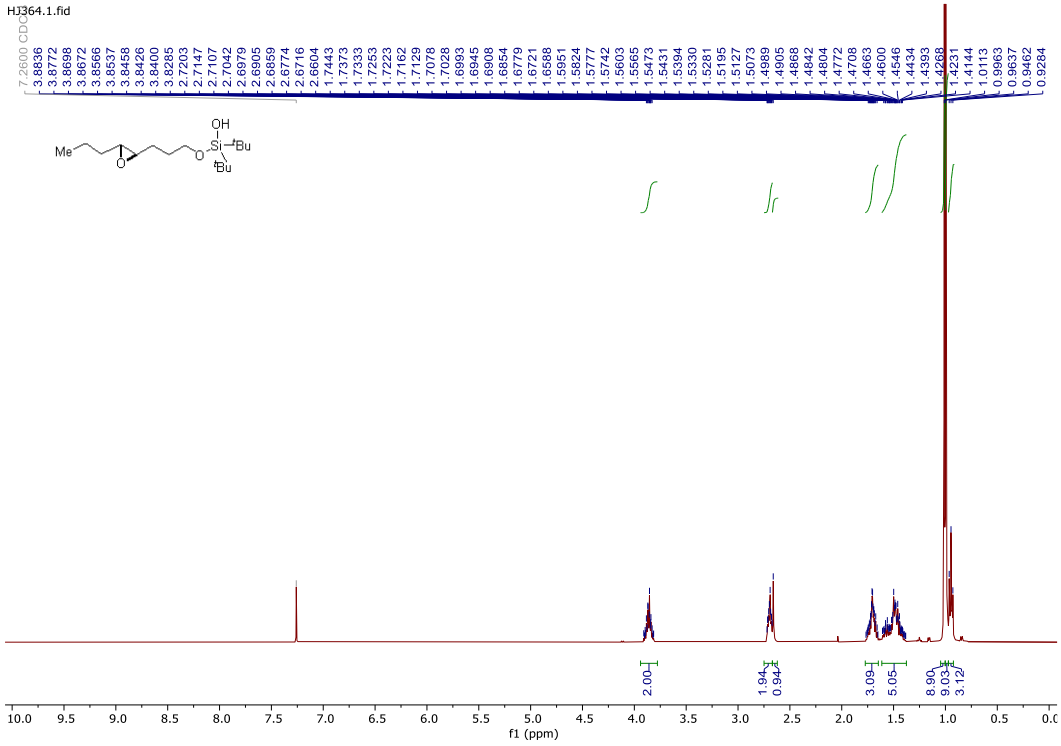


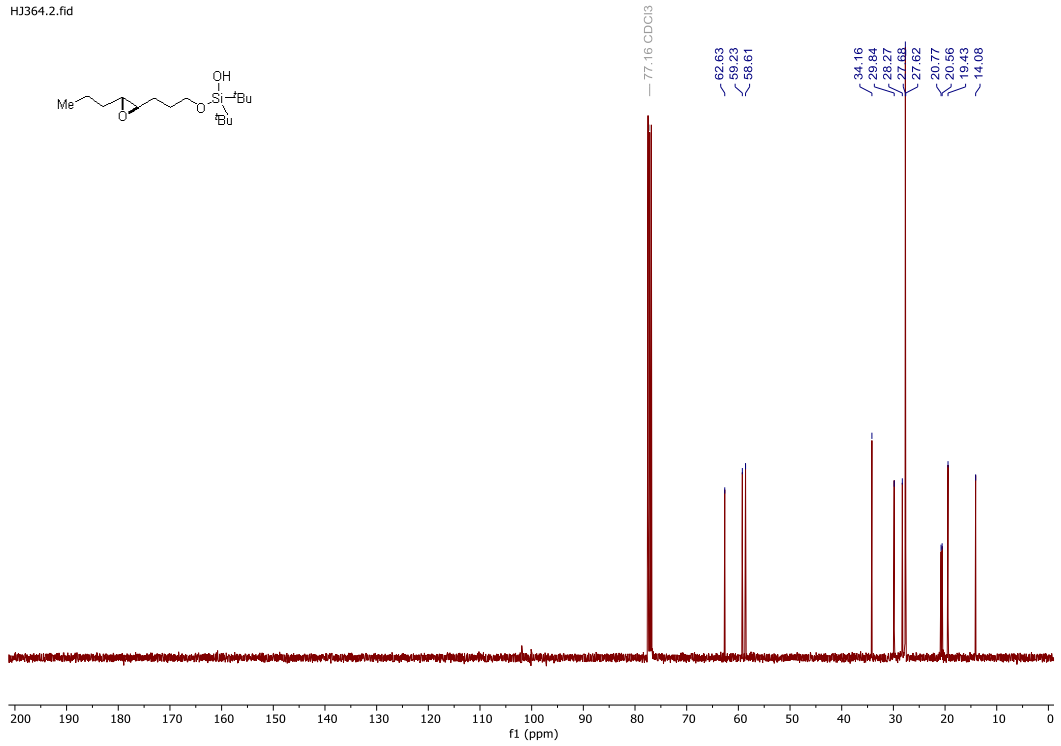
Compound 2 ($CDCl_3$, ^{1}H NMR: 400 MHz, $^{13}C\{^{1}H\}$ NMR: 101 MHz)



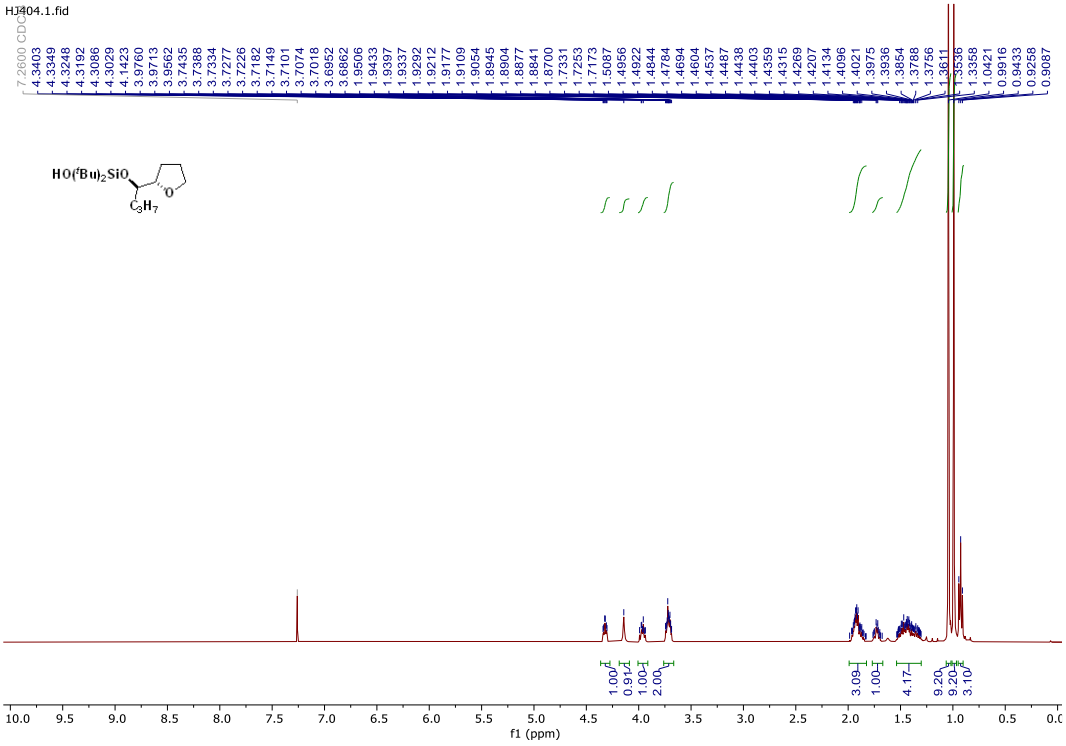


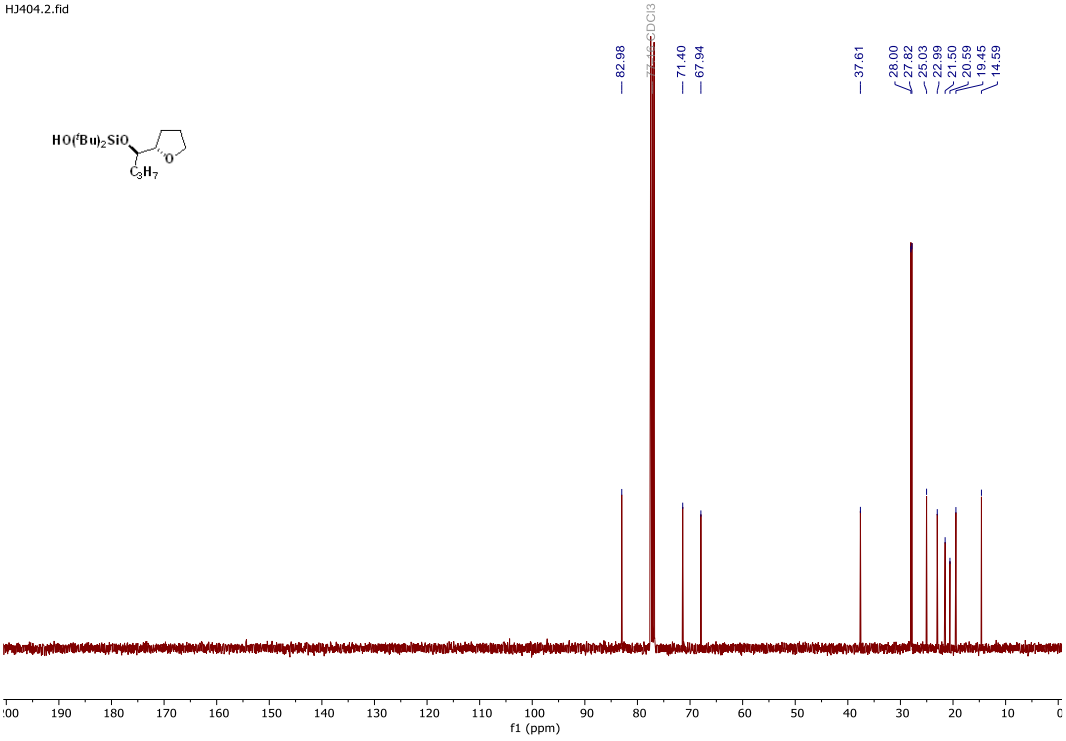
Compound 3 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



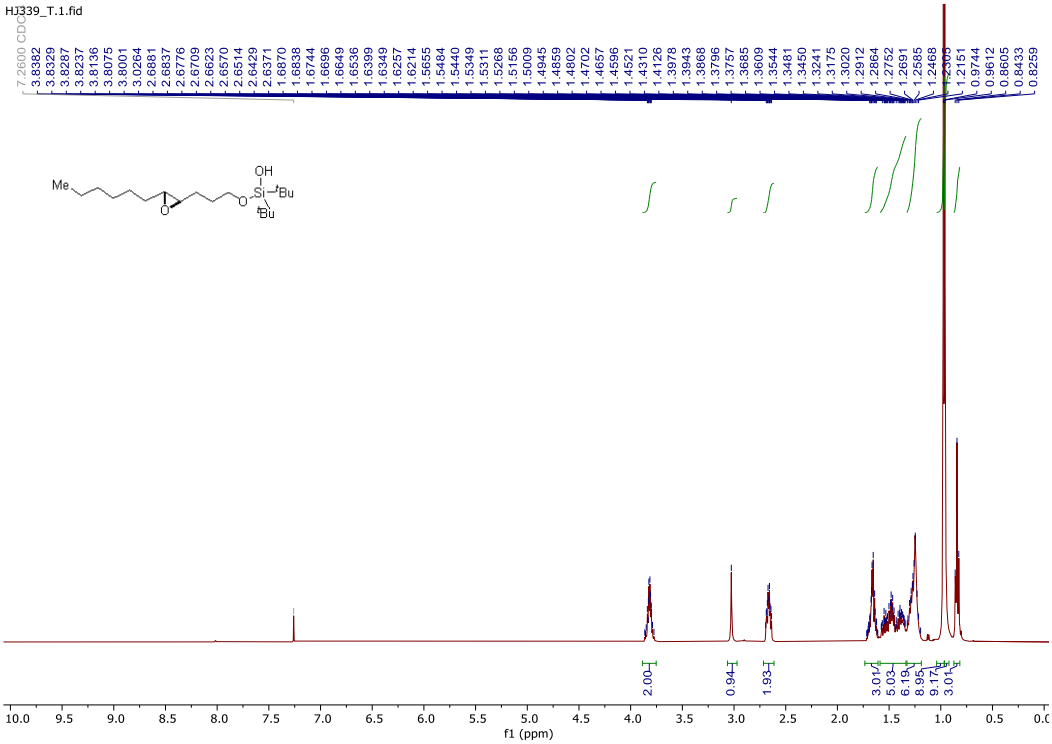


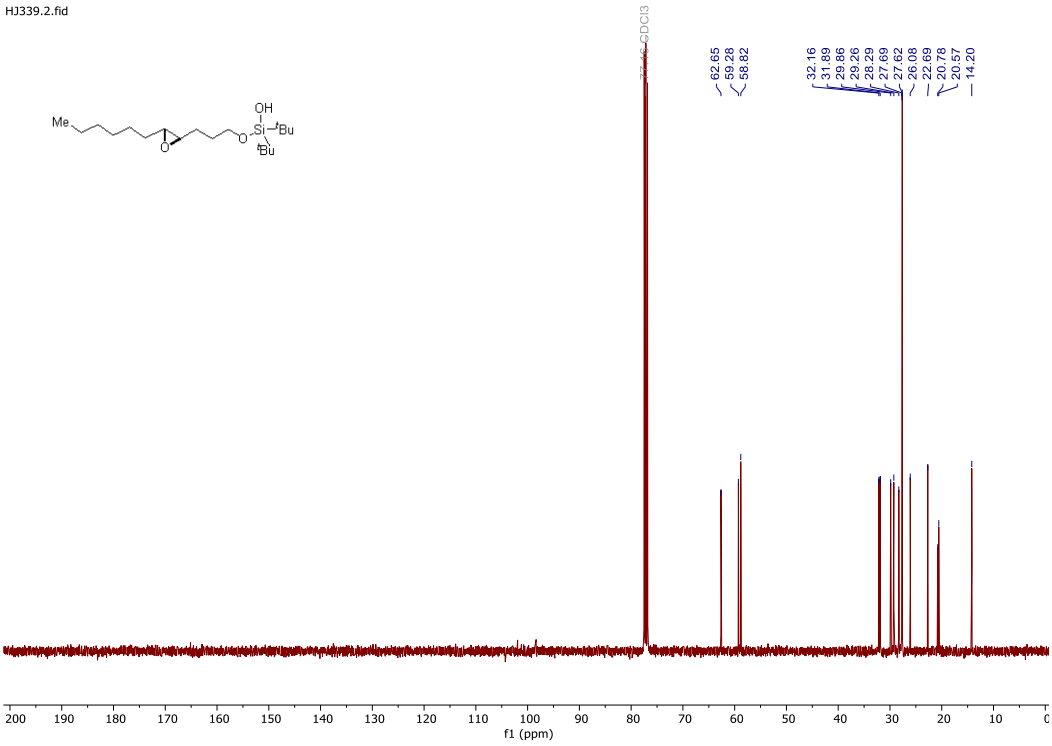
Compound 4 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



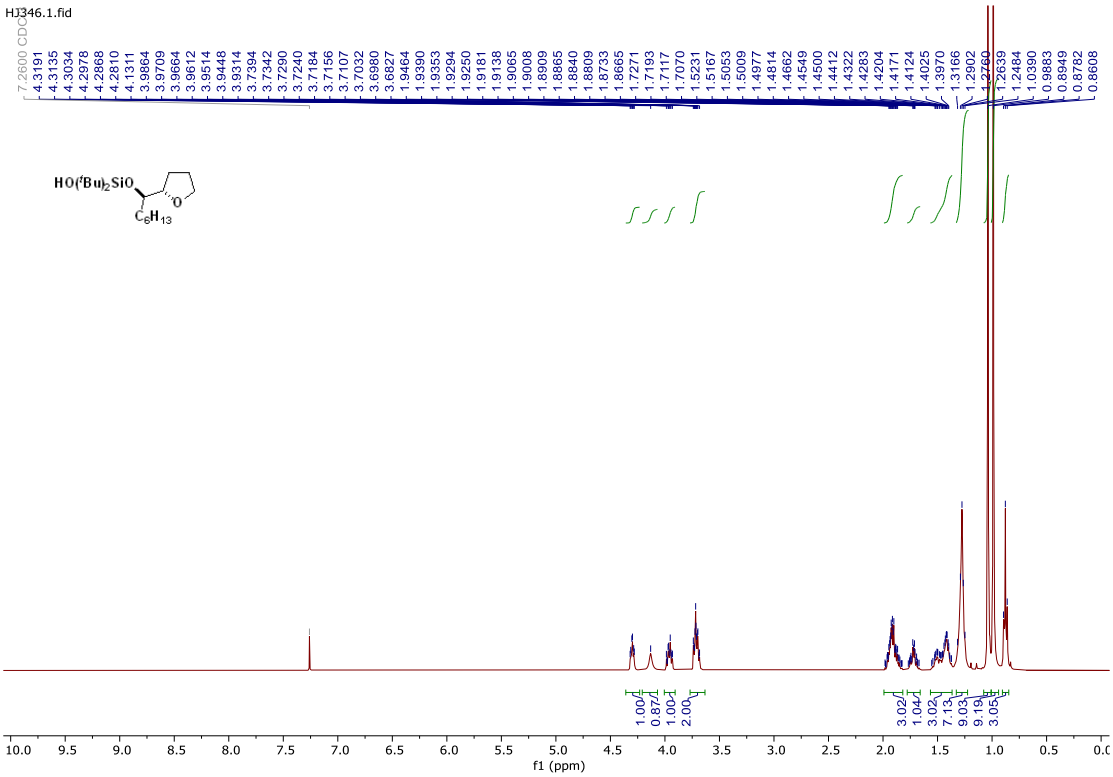


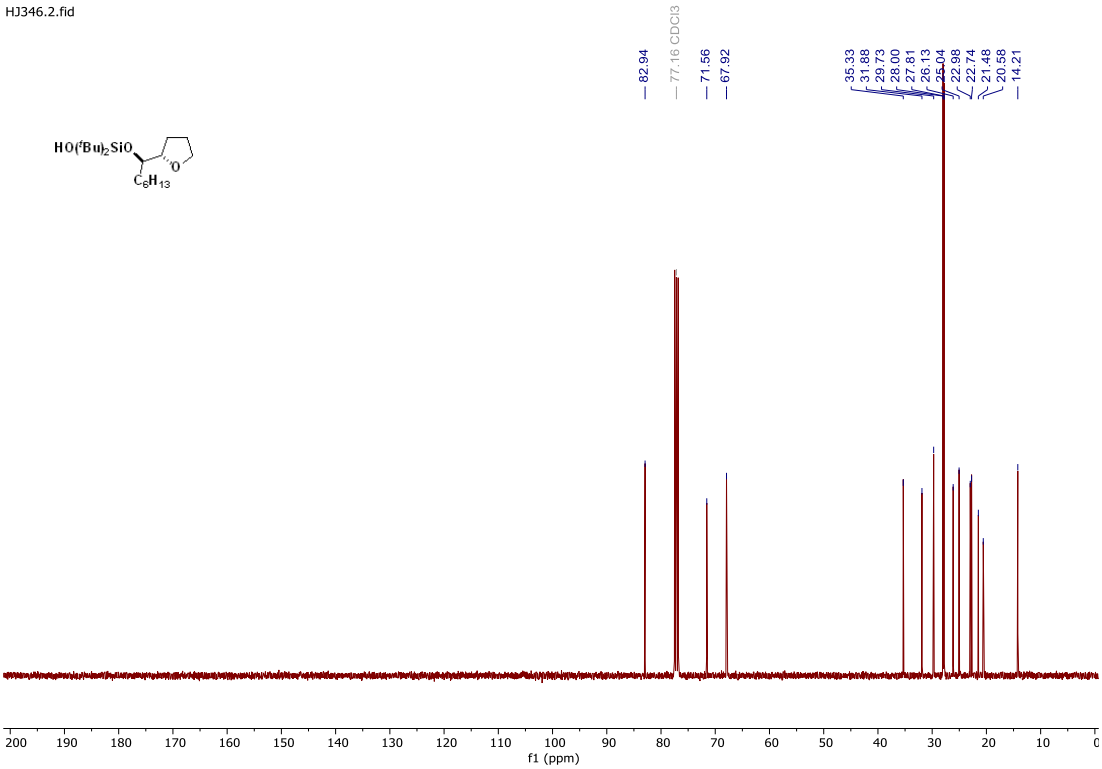
Compound 5 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



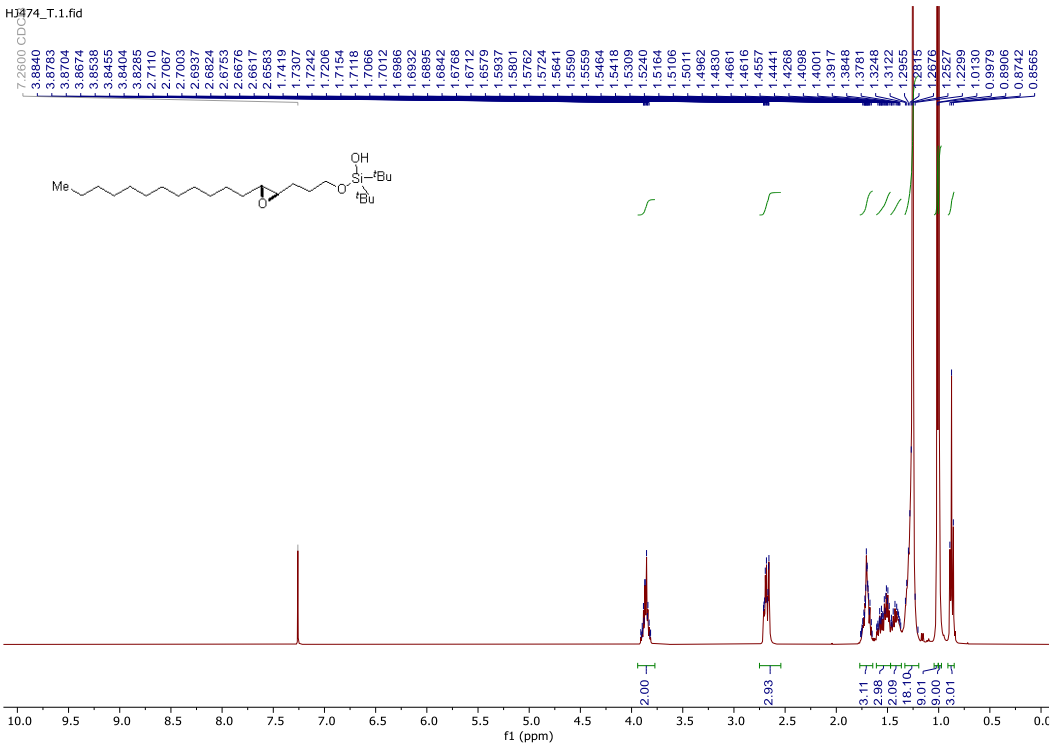


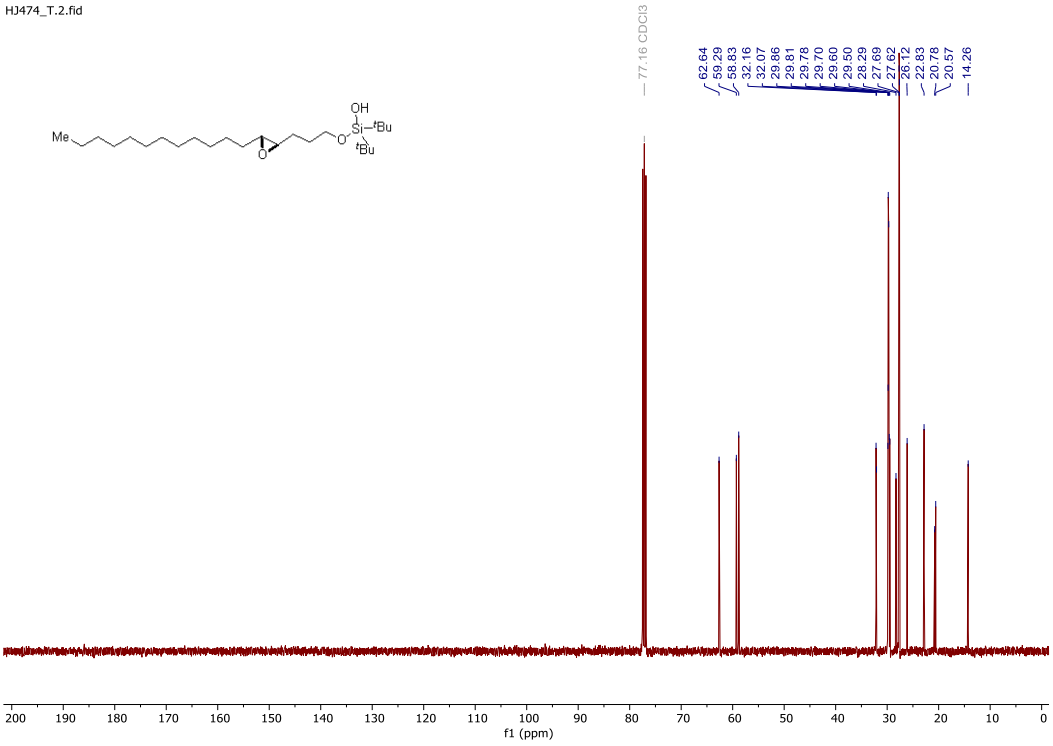
Compound 6 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



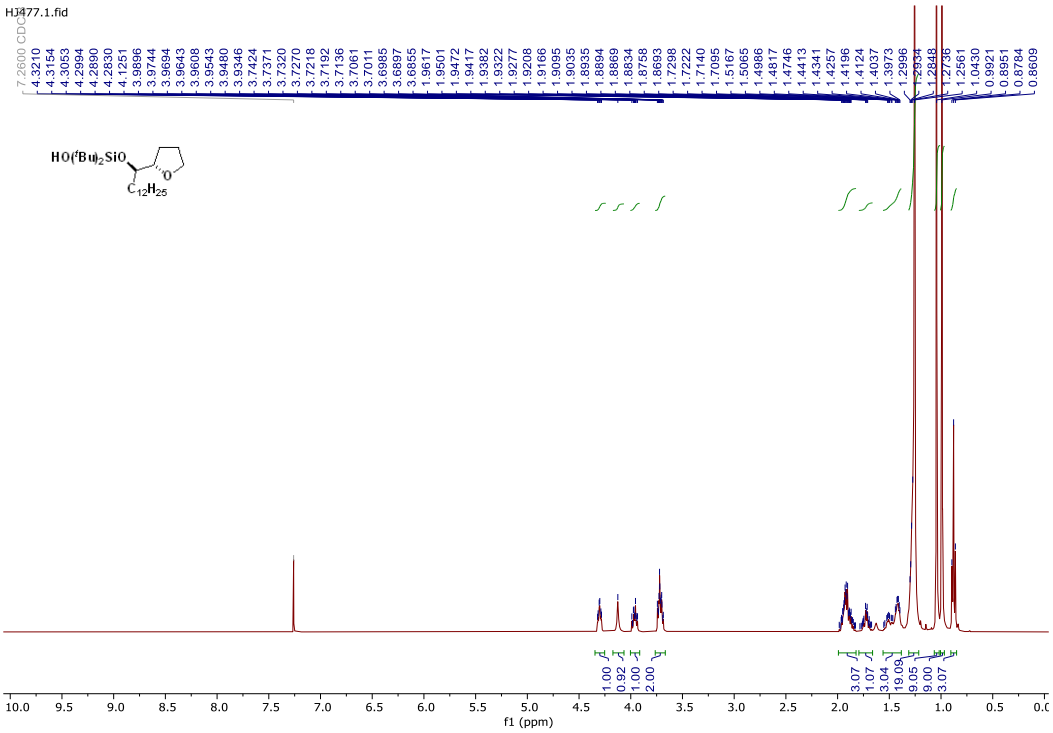


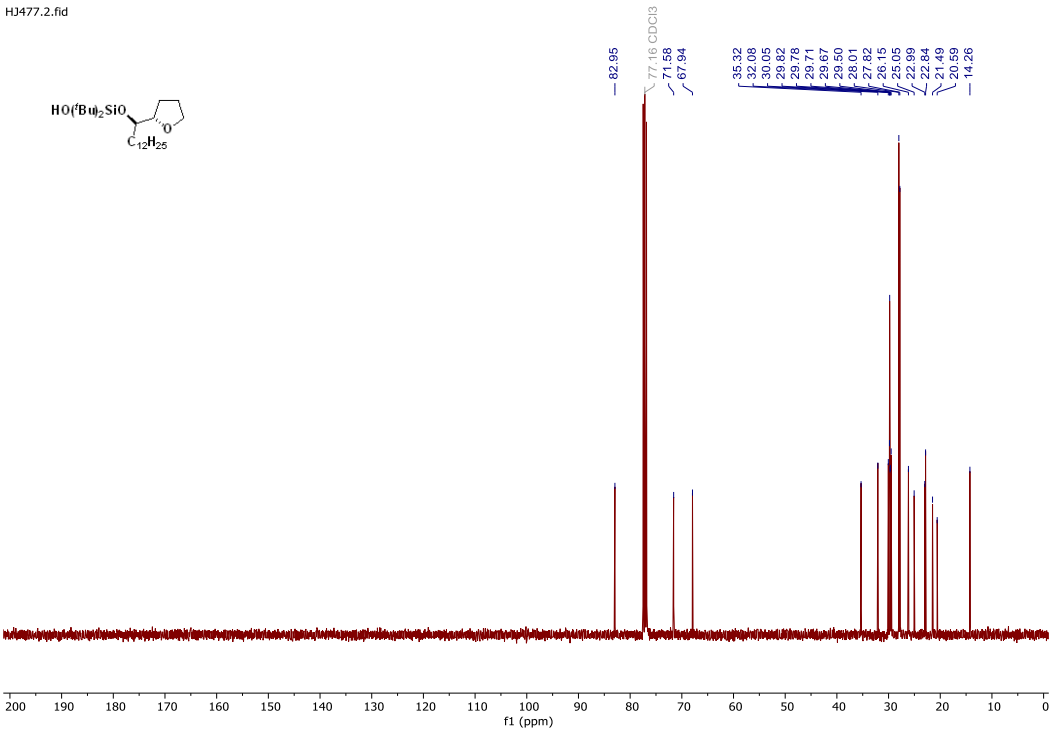
Compound 7 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



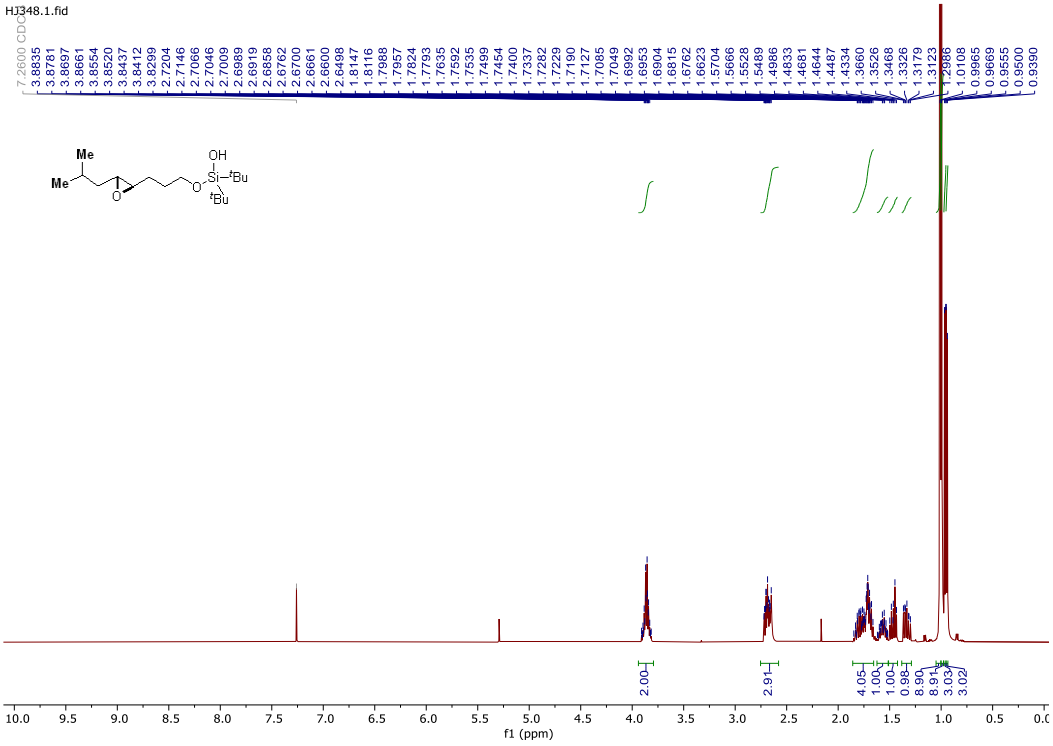


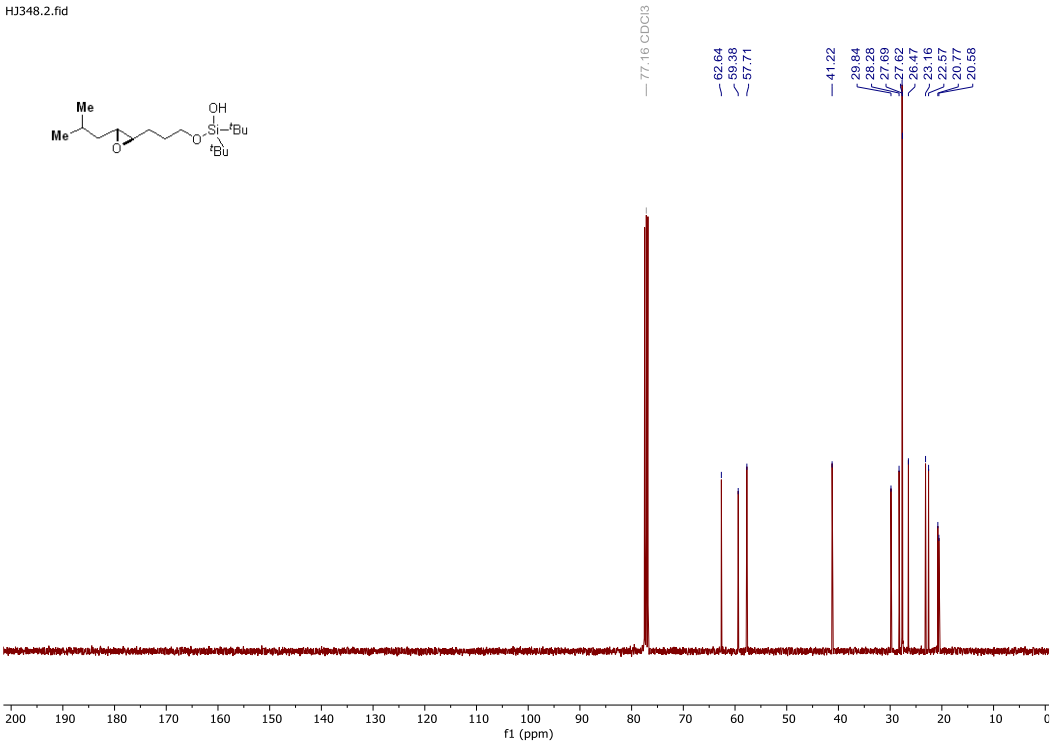
Compound 8 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



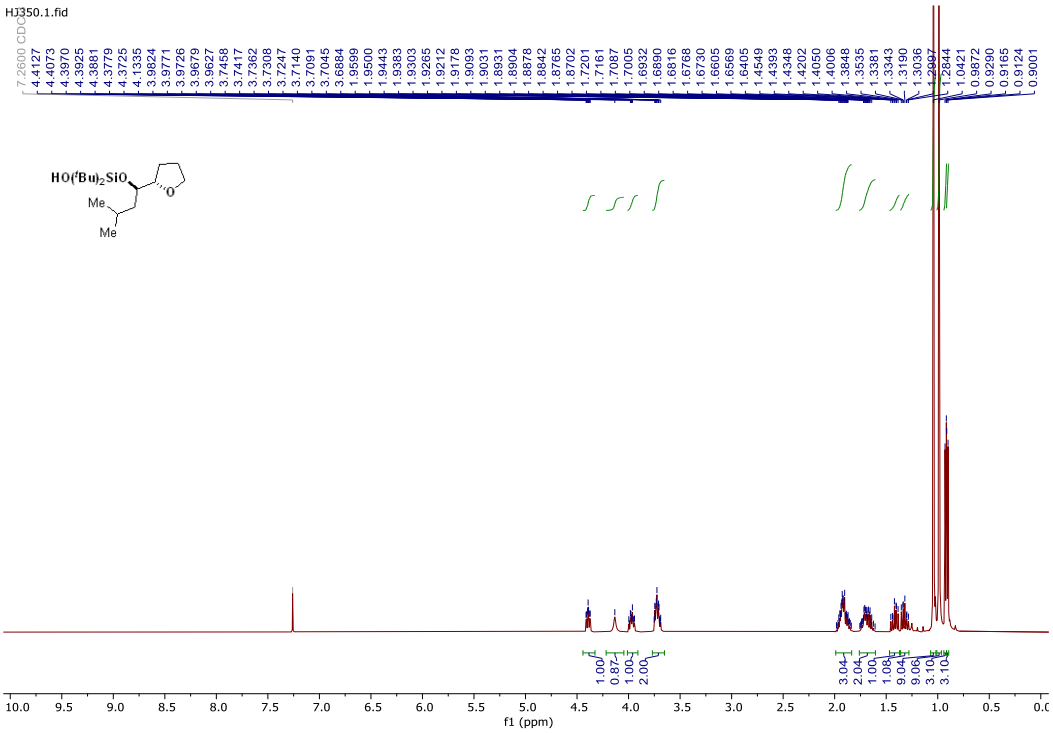


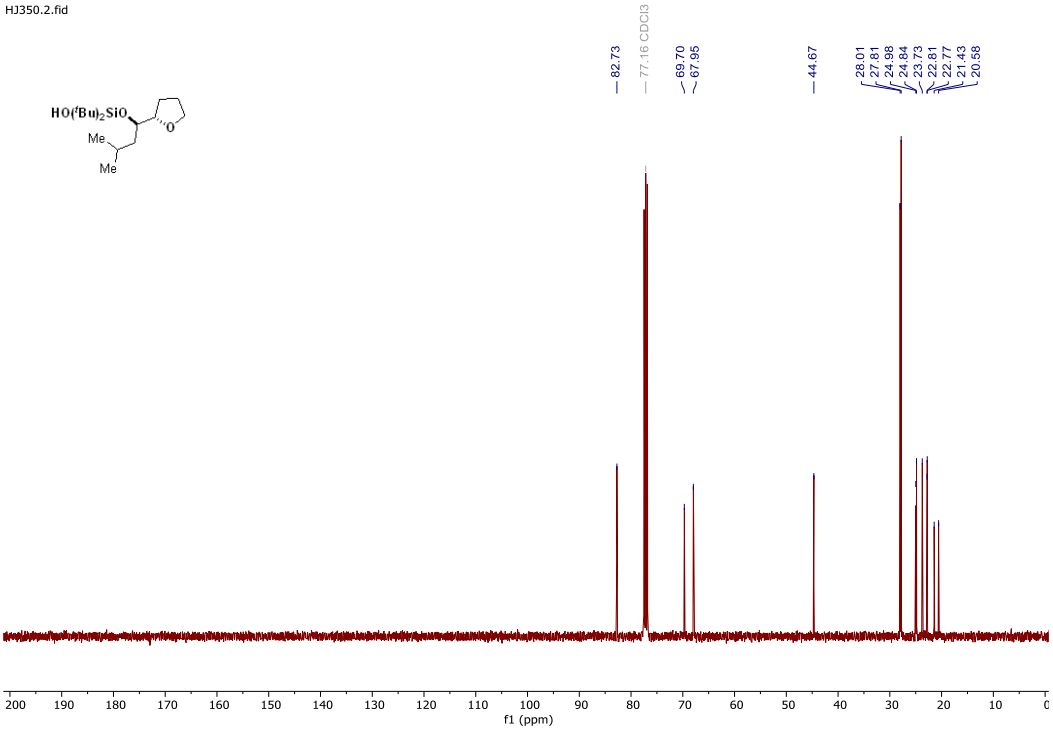
Compound 9 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



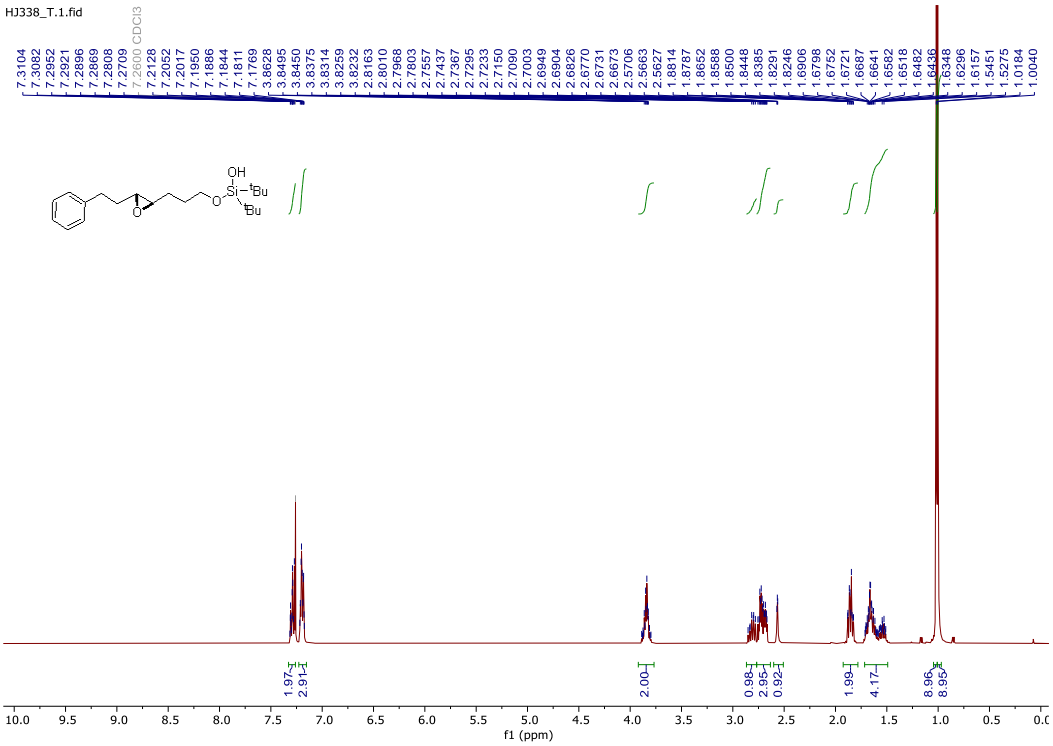


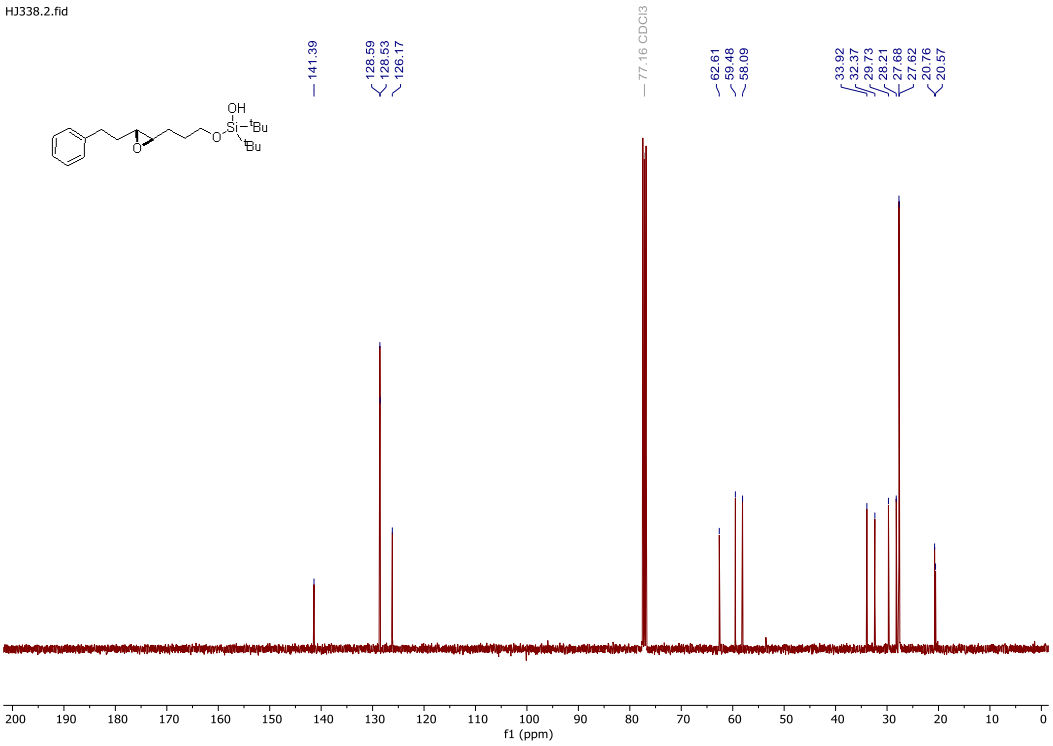
Compound 10 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



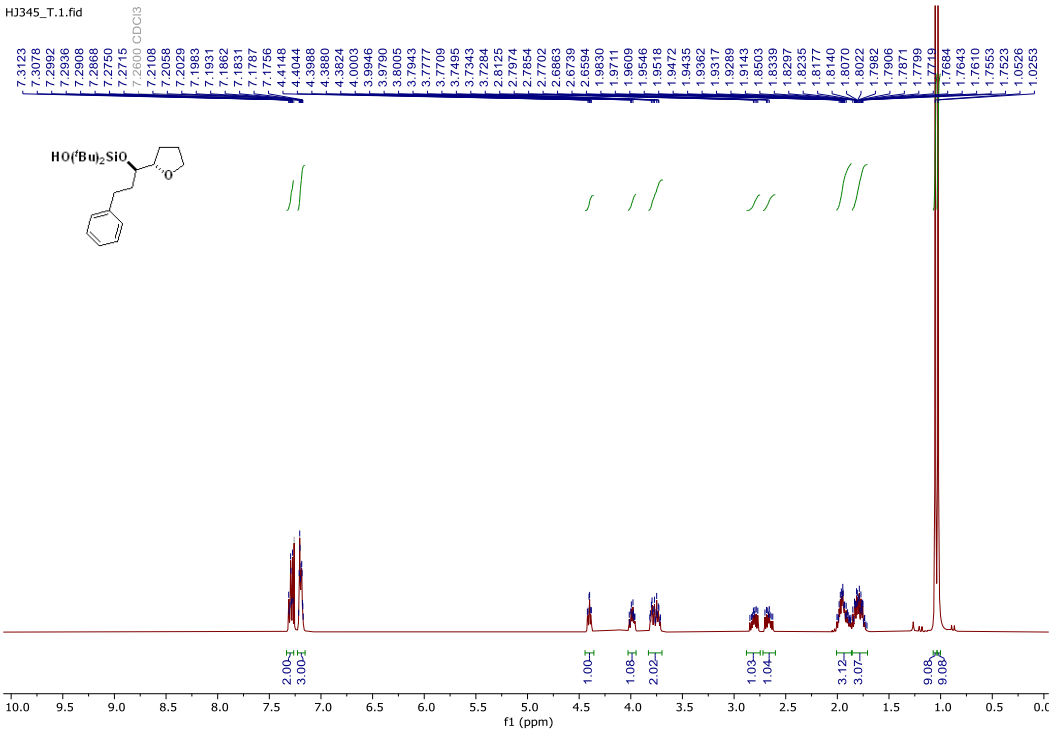


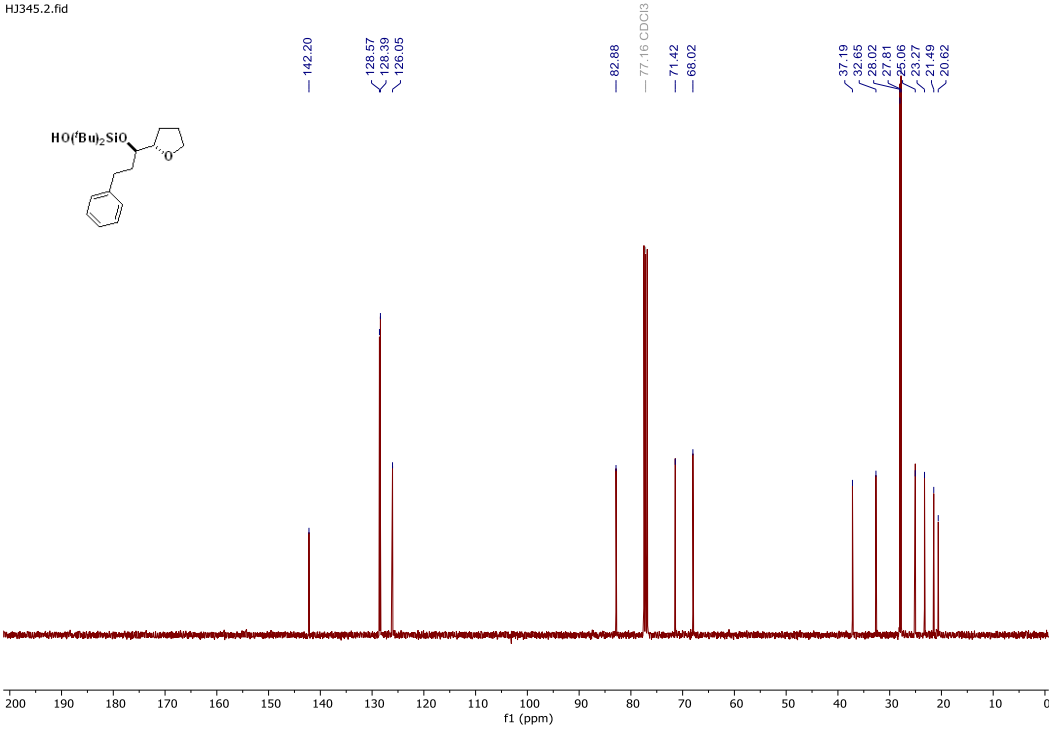
Compound 11 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



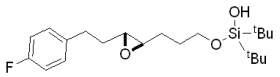


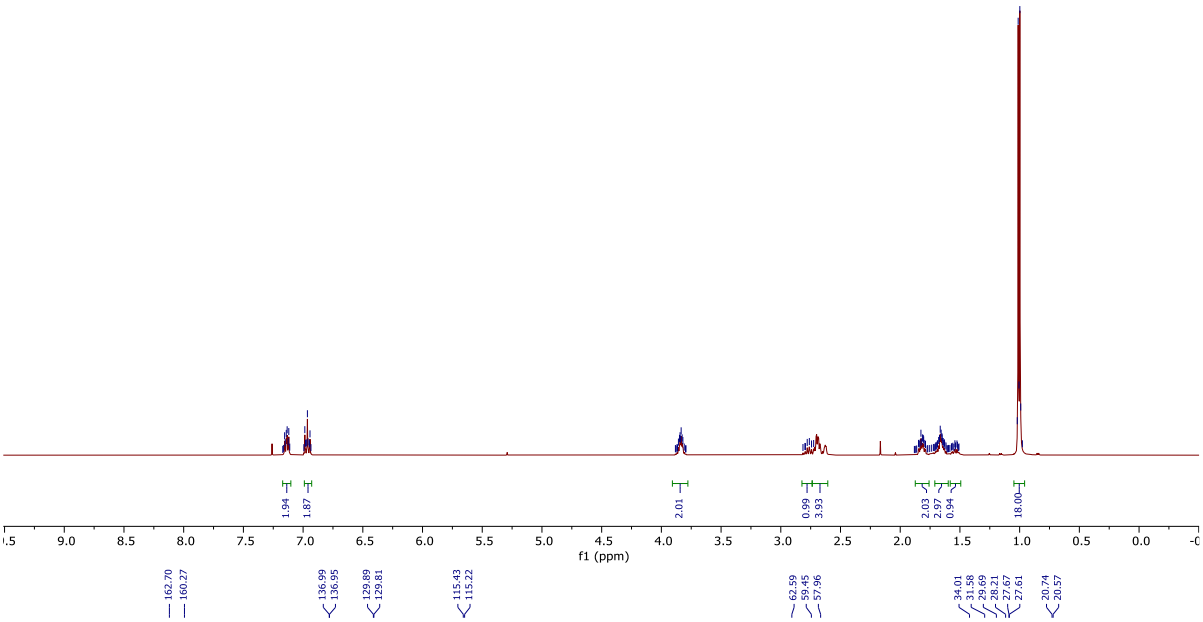
Compound 12 ($CDCl_3$, ^{1}H NMR: 400 MHz, $^{13}C\{^{1}H\}$ NMR: 101 MHz)

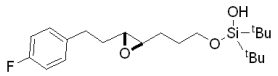


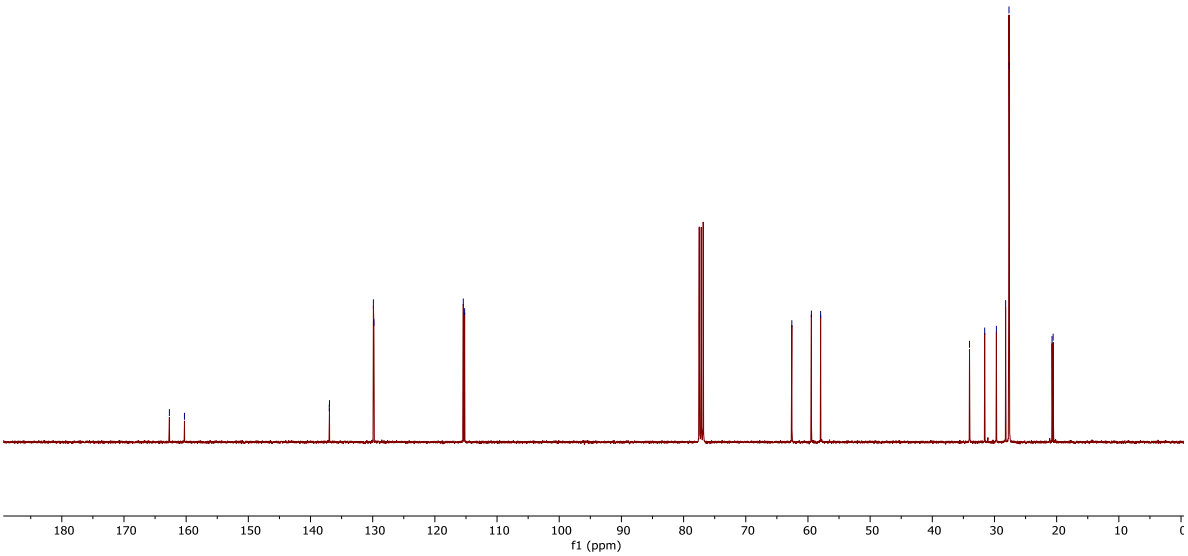


Compound 13 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

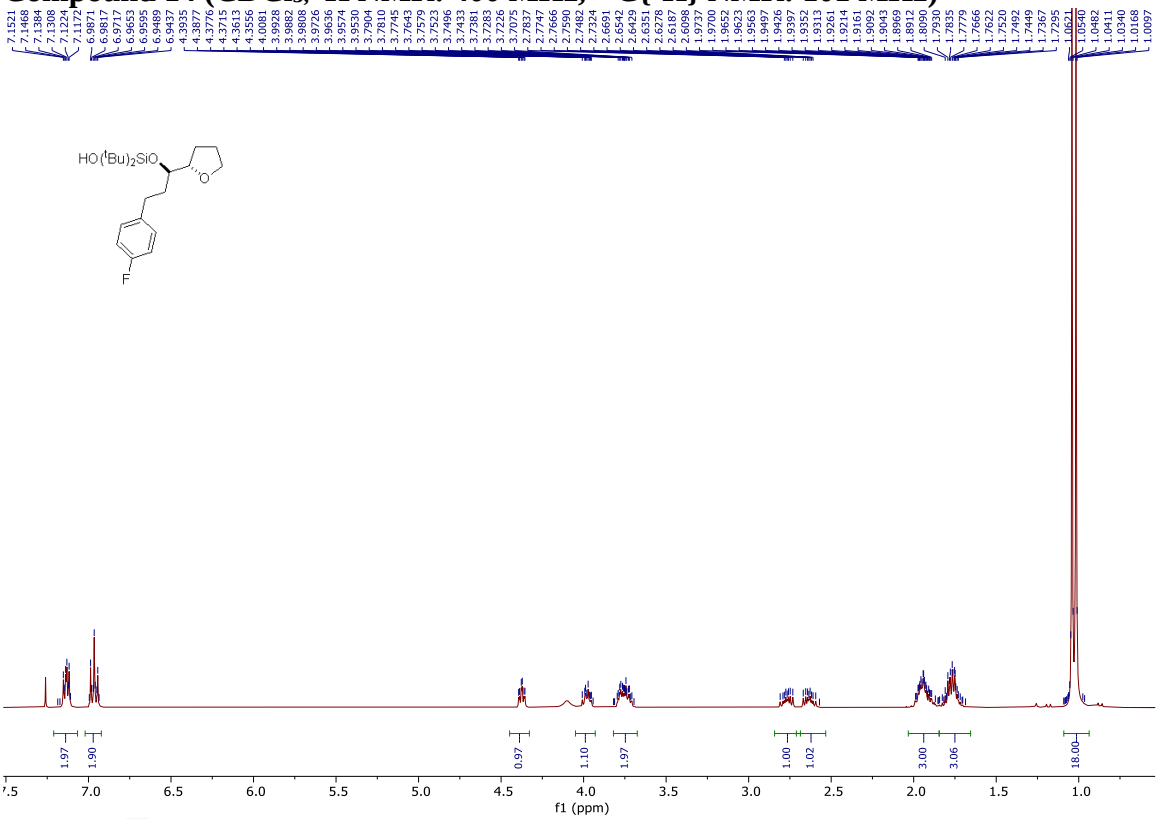


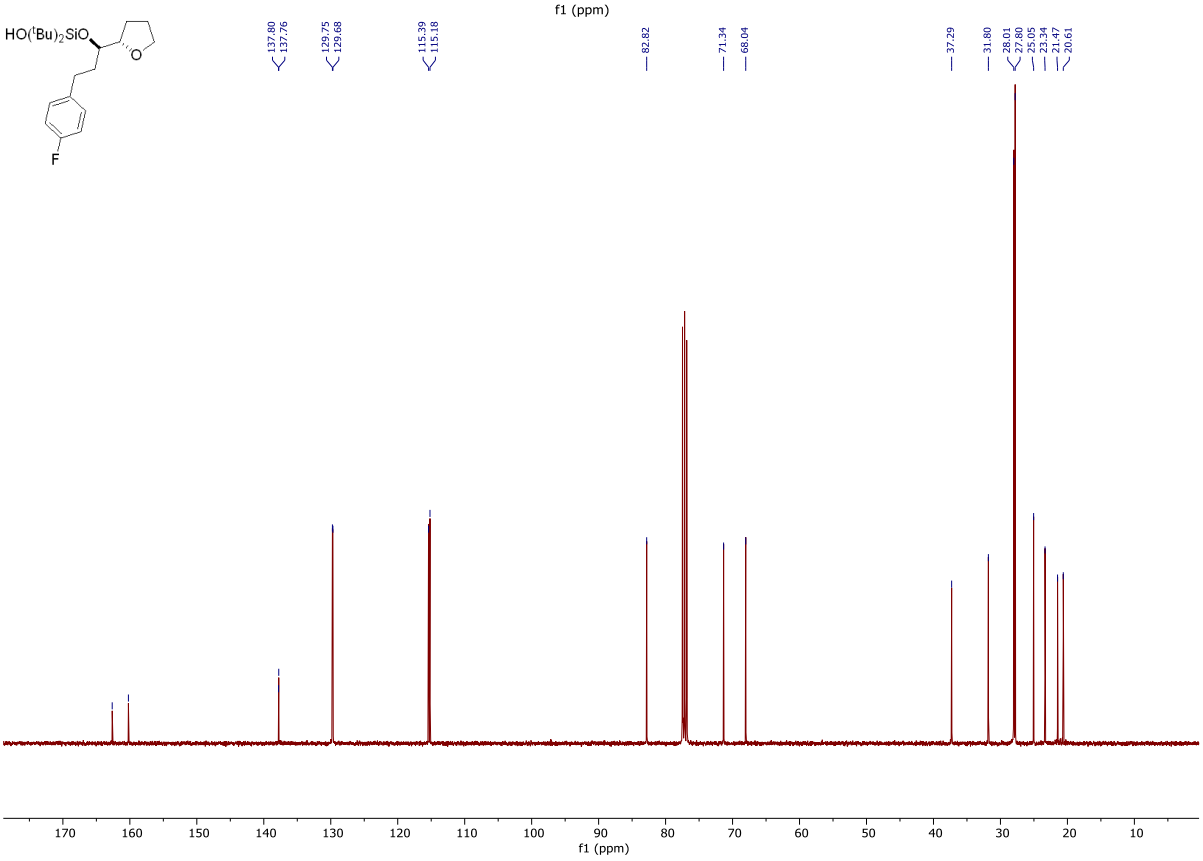




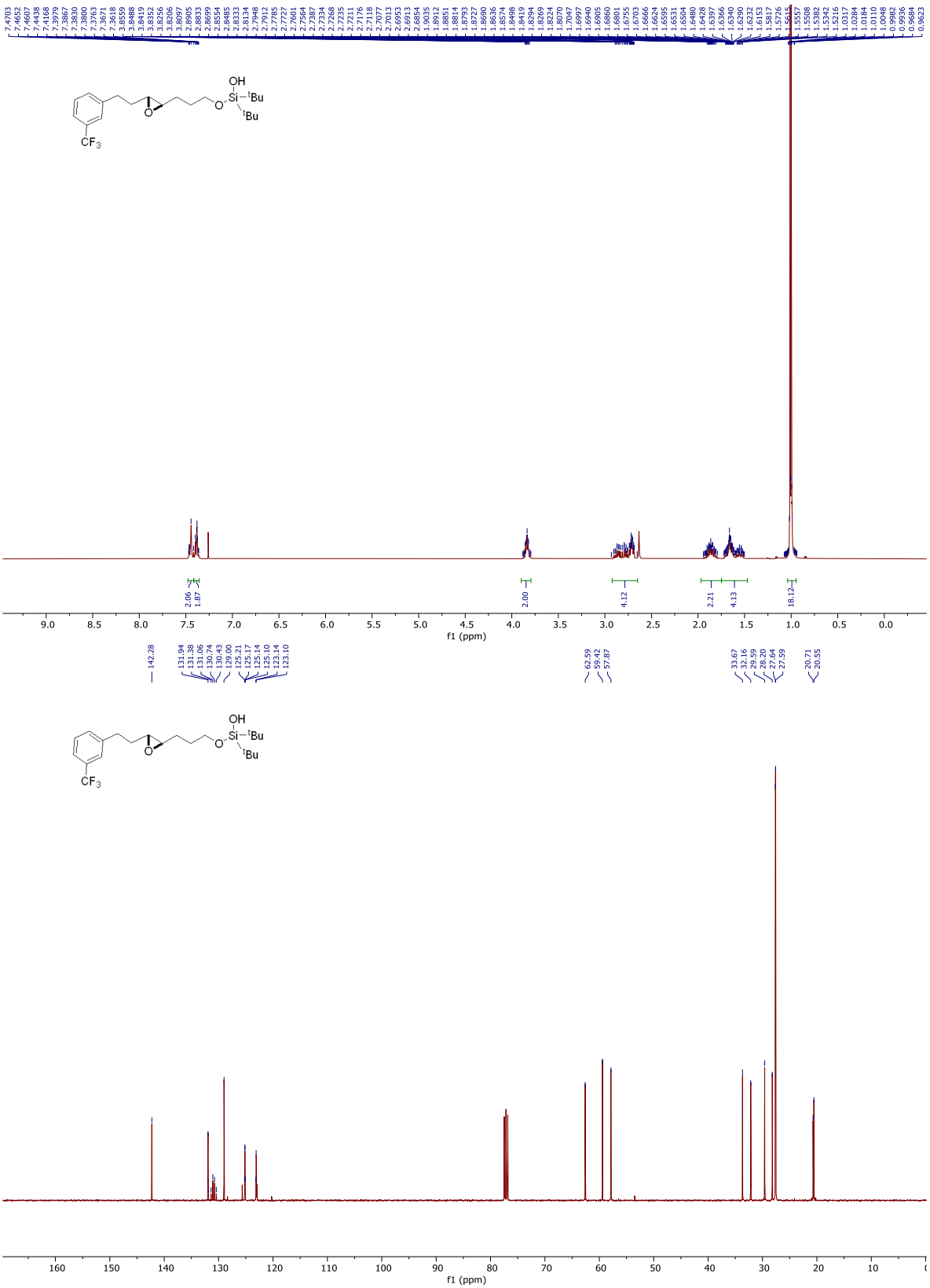


Compound 14 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

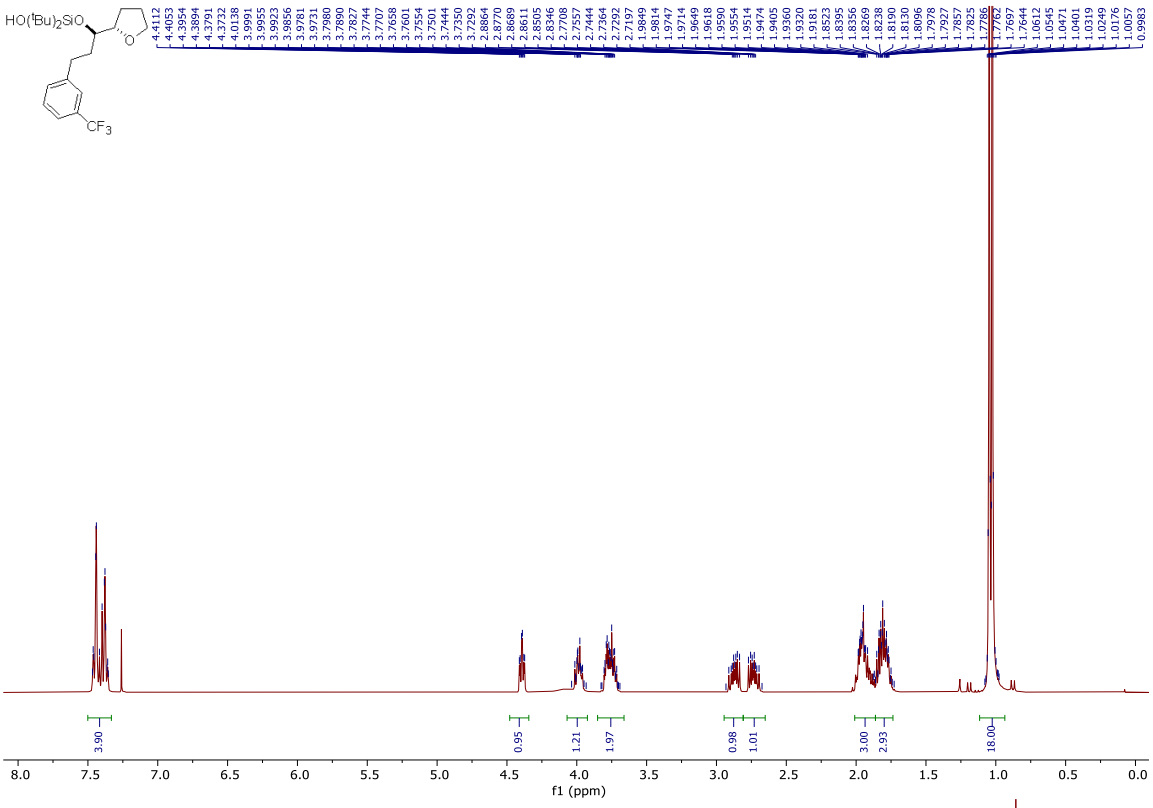


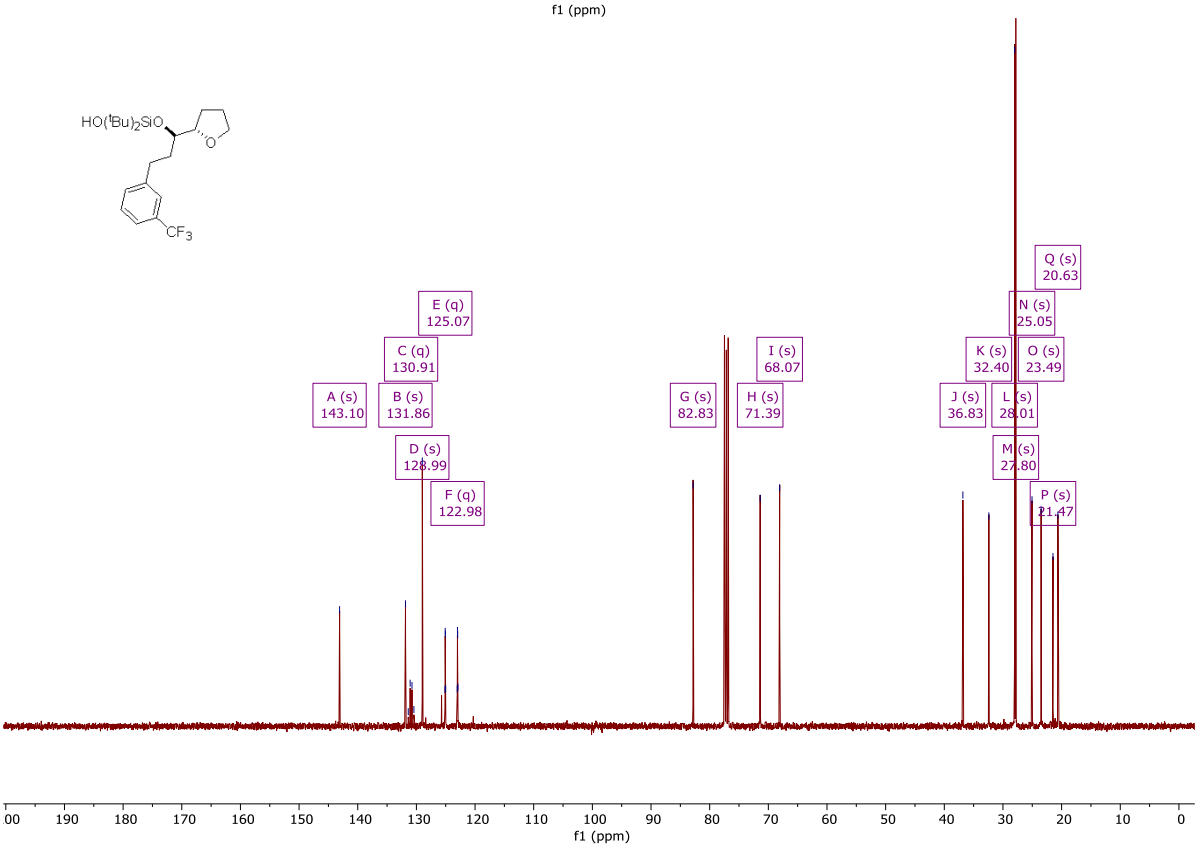


Compound 15 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

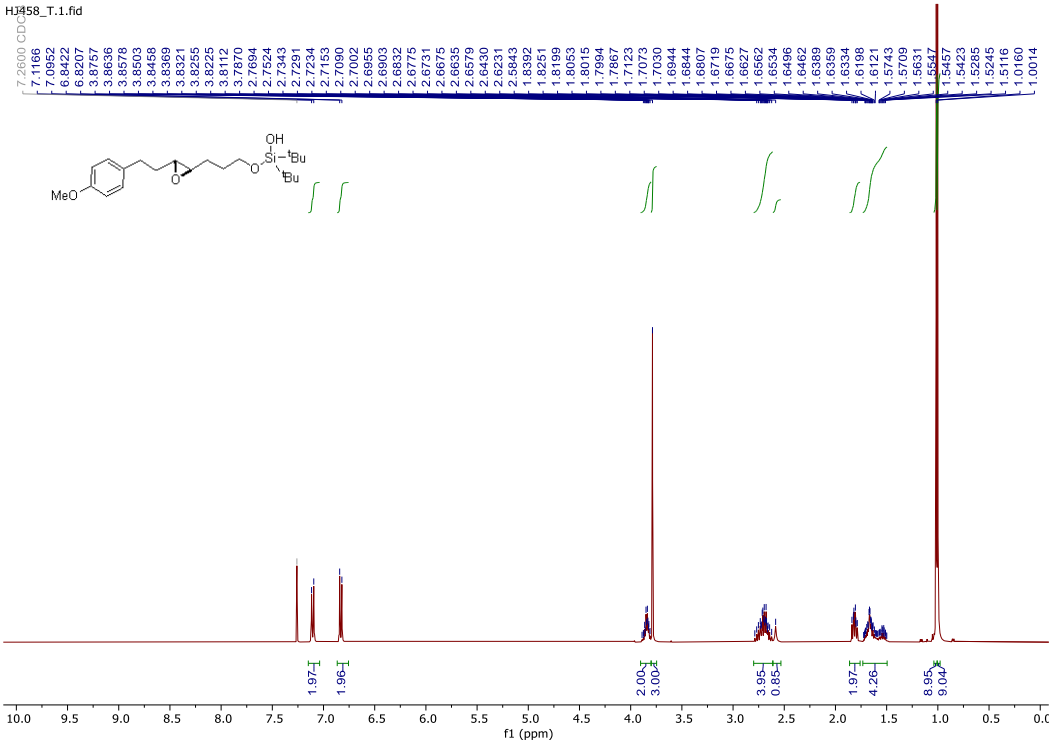


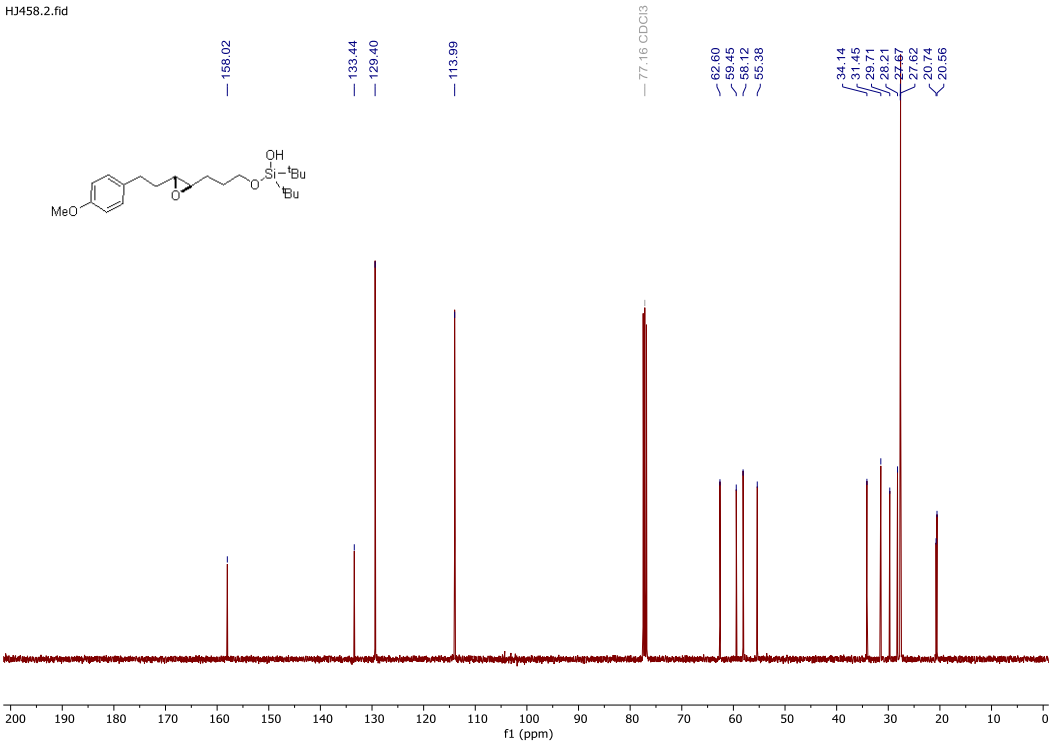
Compound 16 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



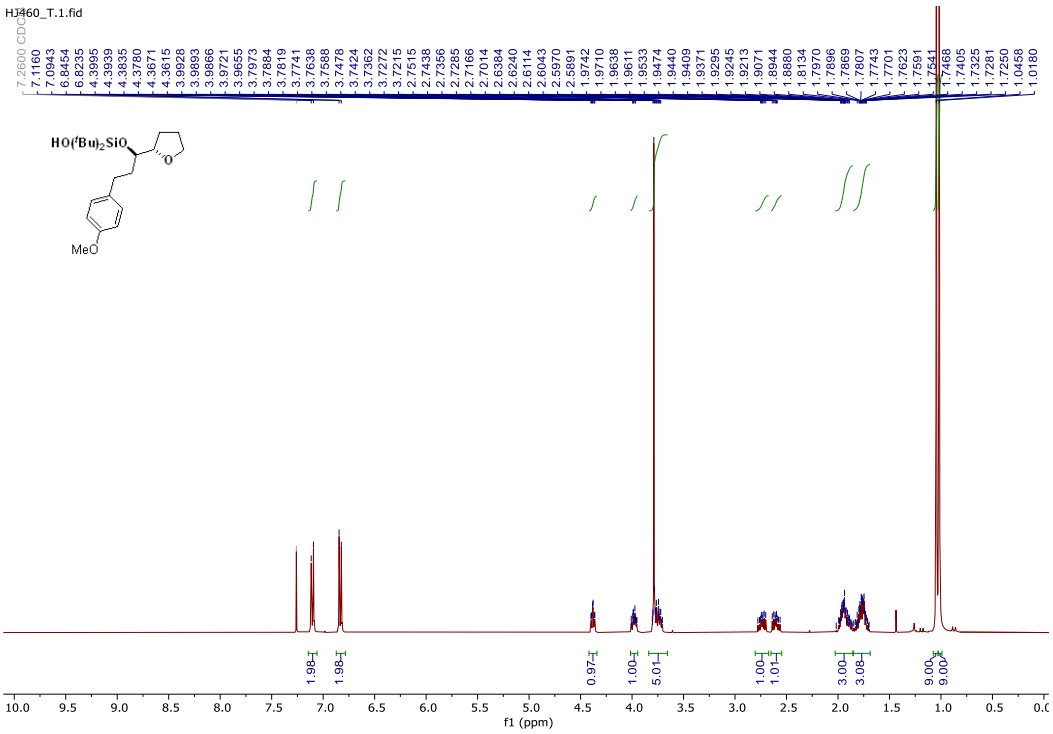


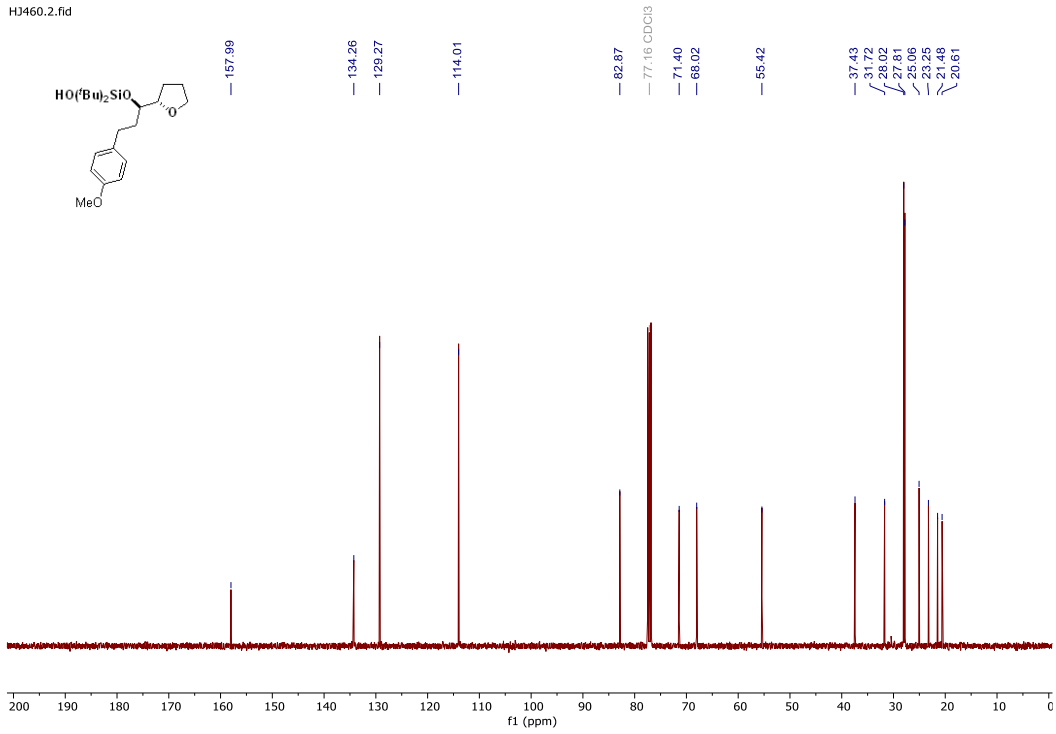
Compound 17 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



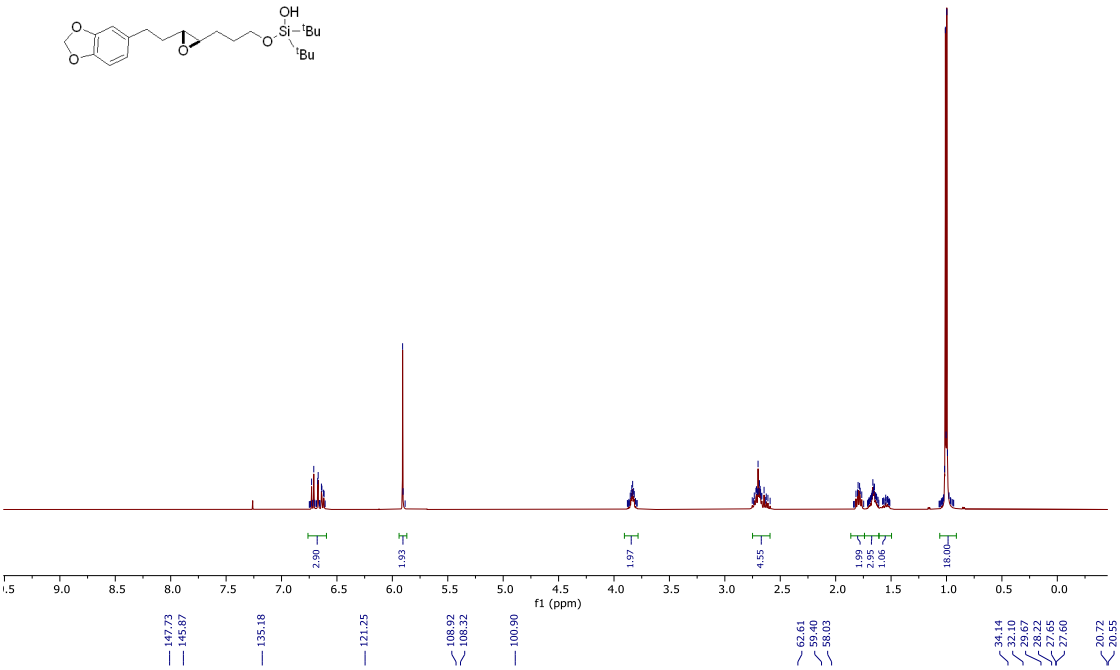


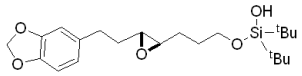
Compound 18 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

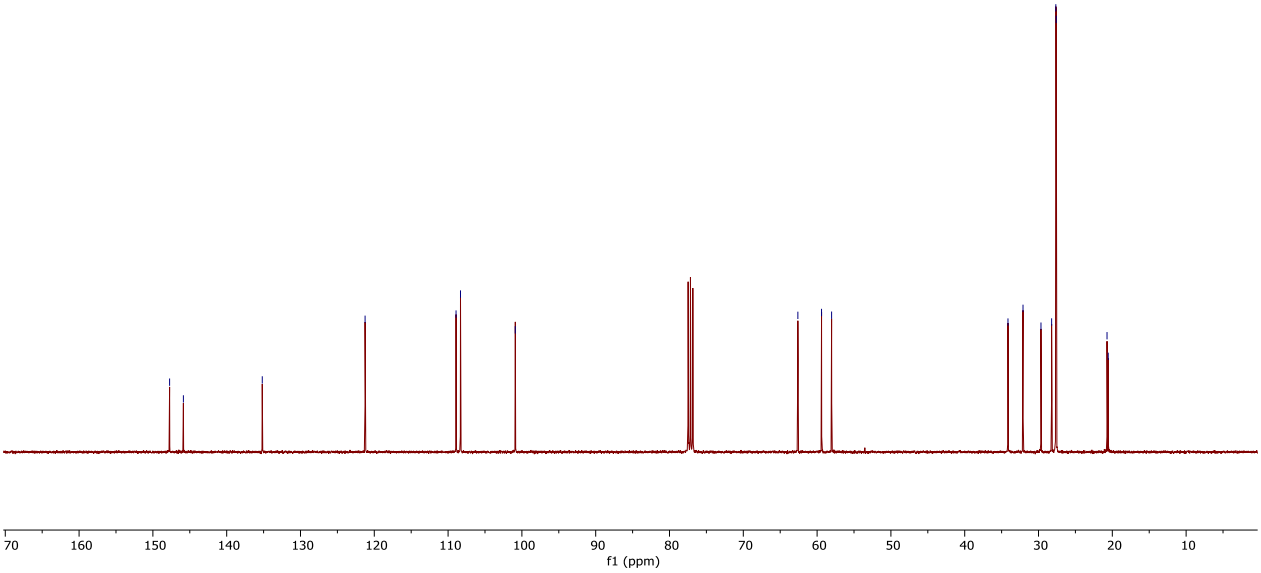




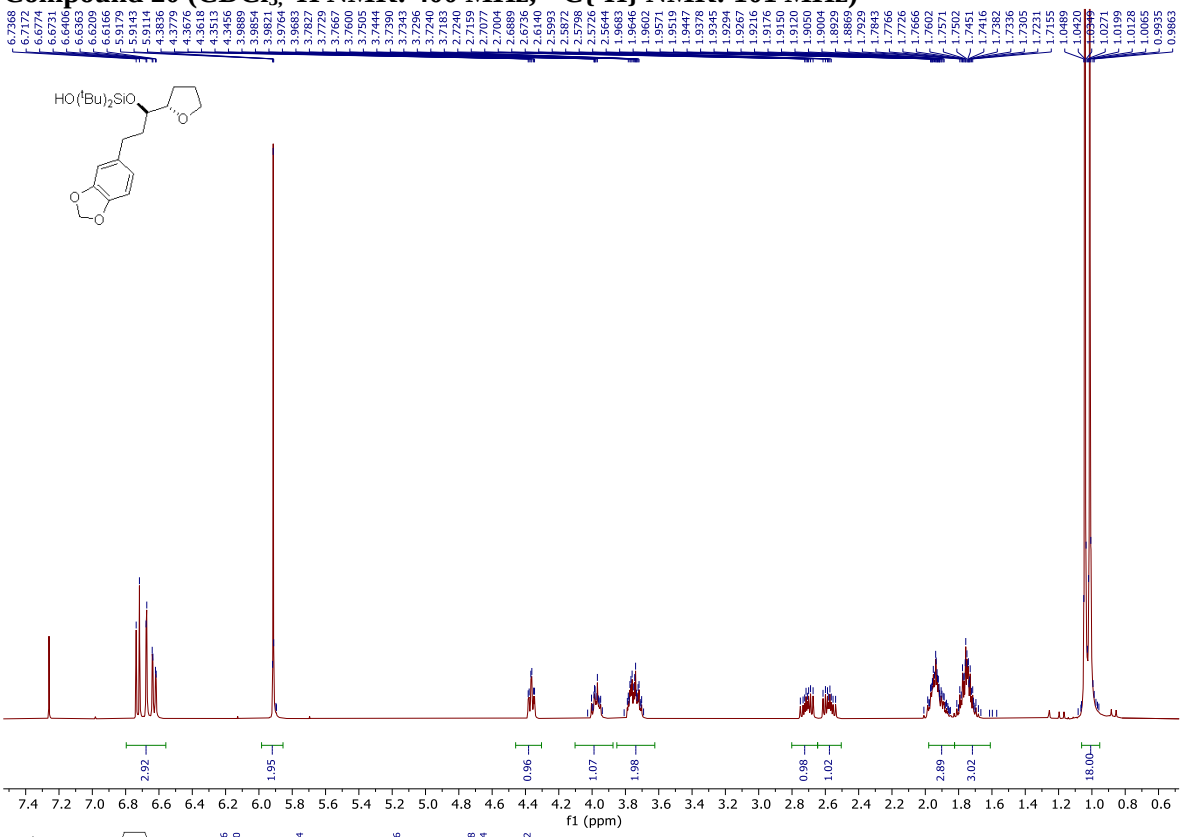
Compound 19 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

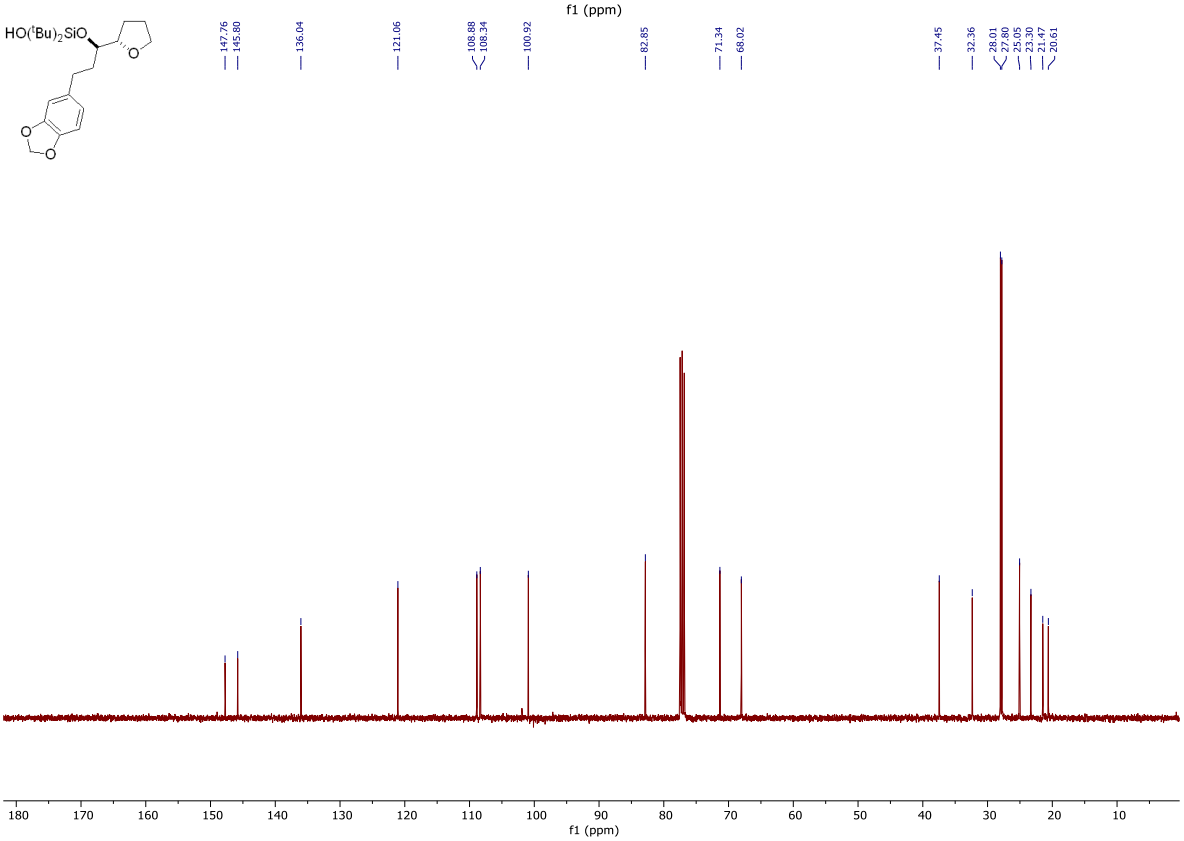




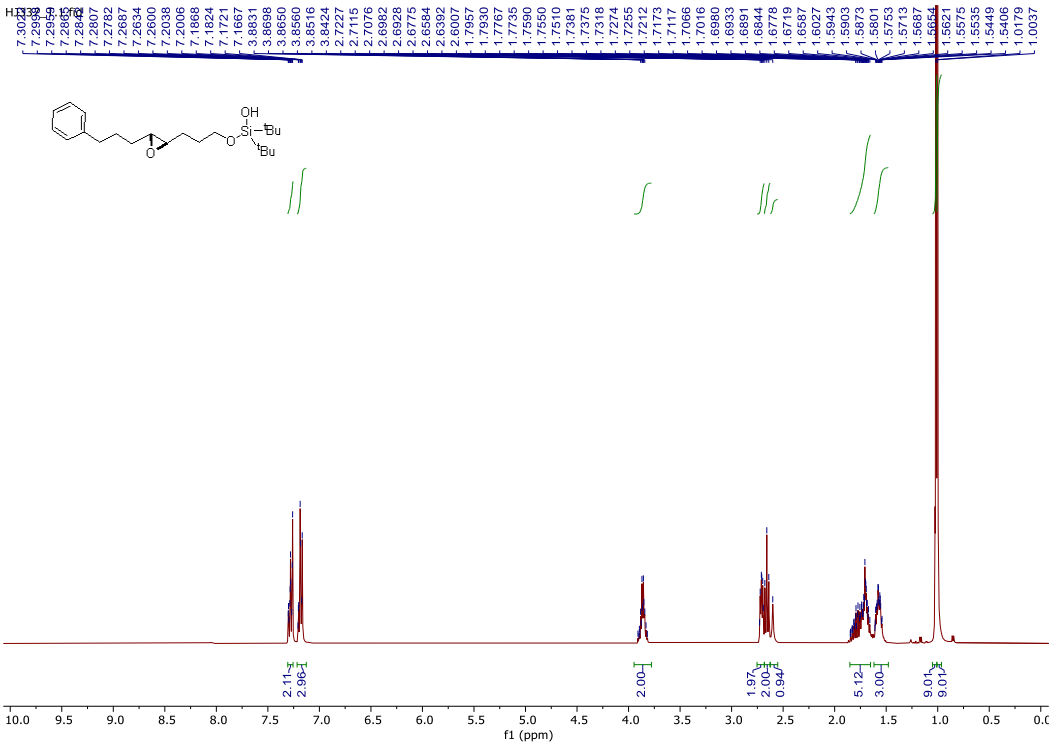


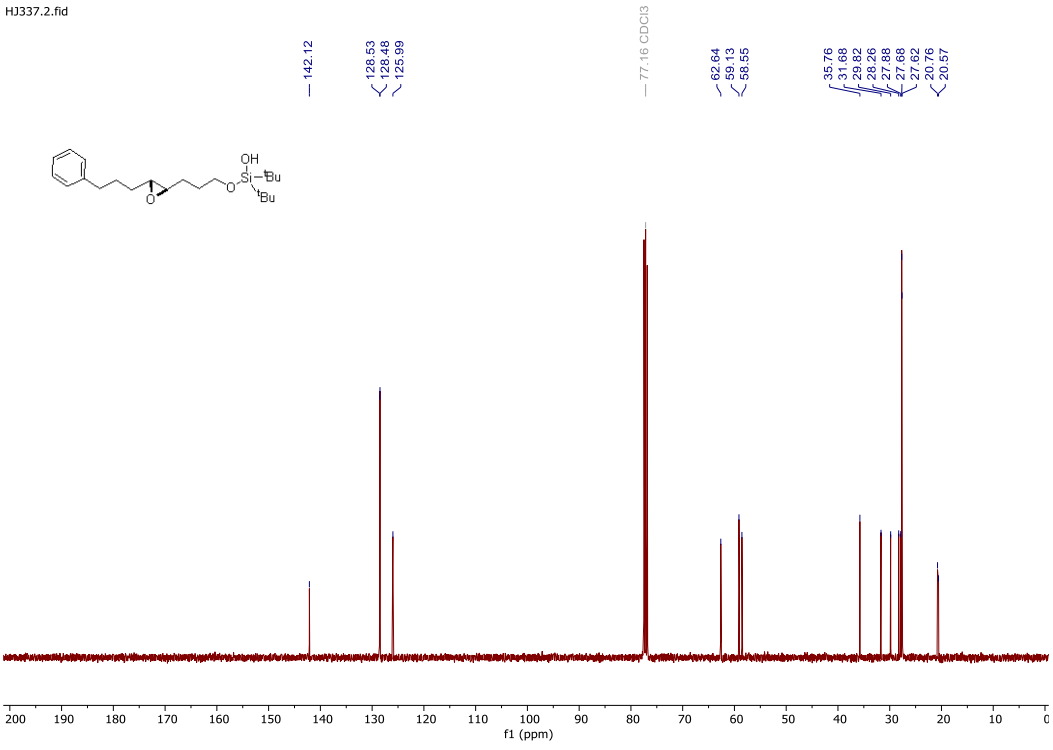
Compound 20 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



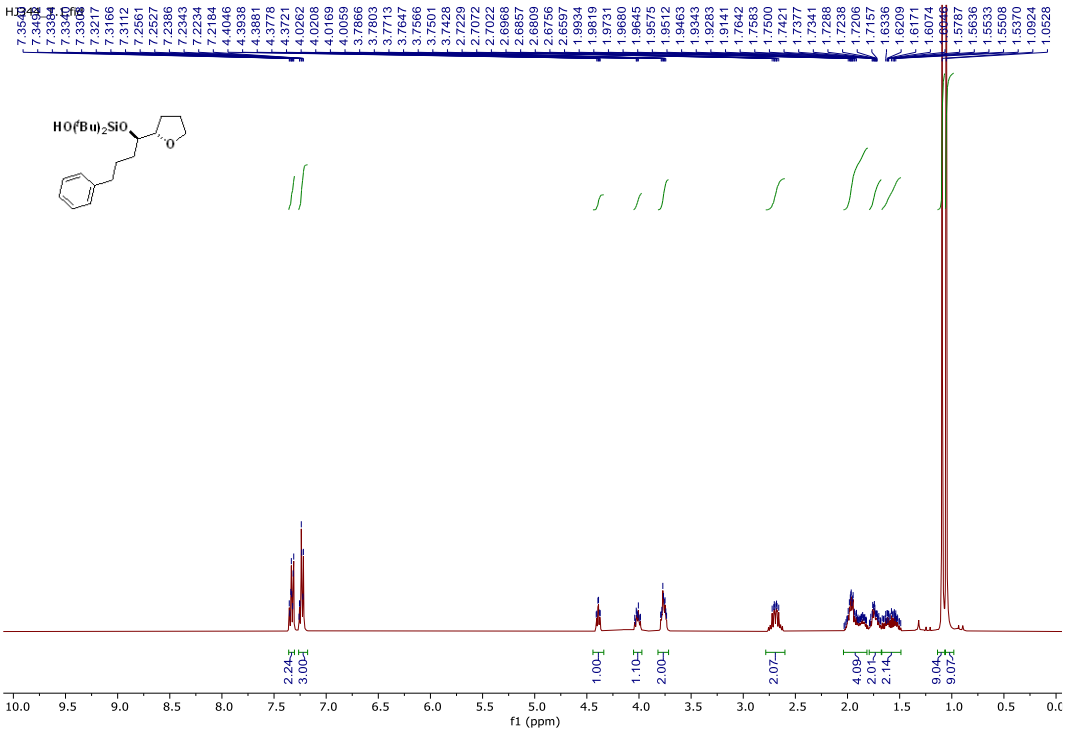


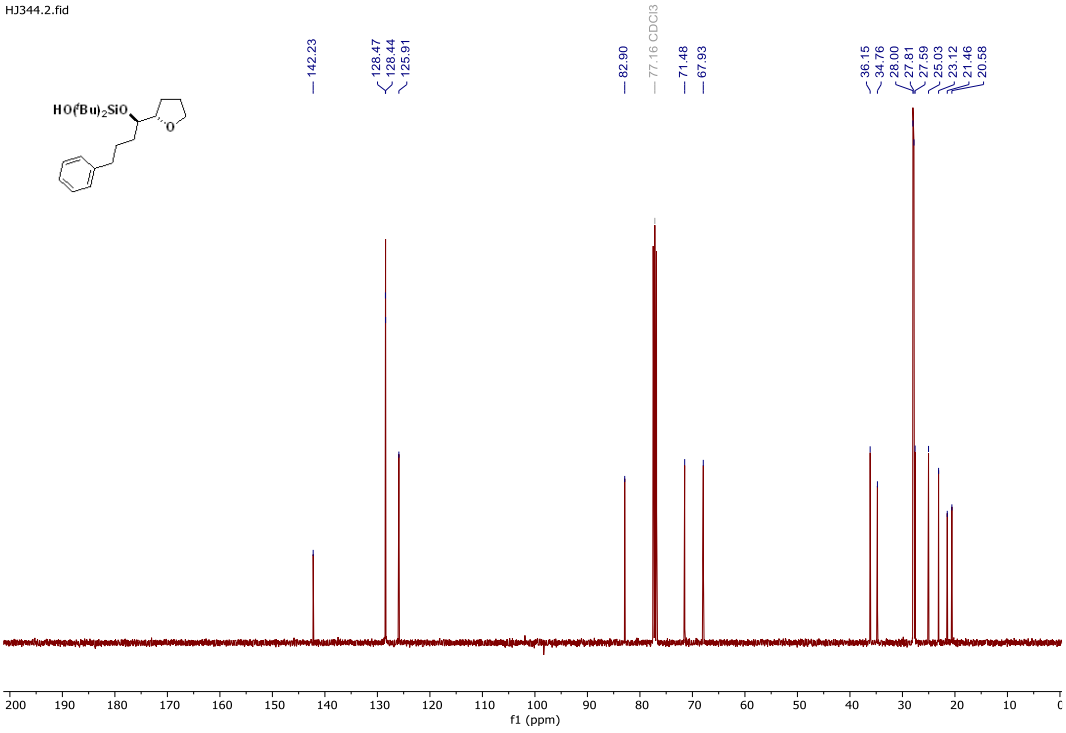
Compound 21 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



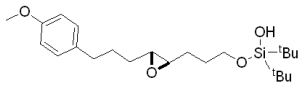


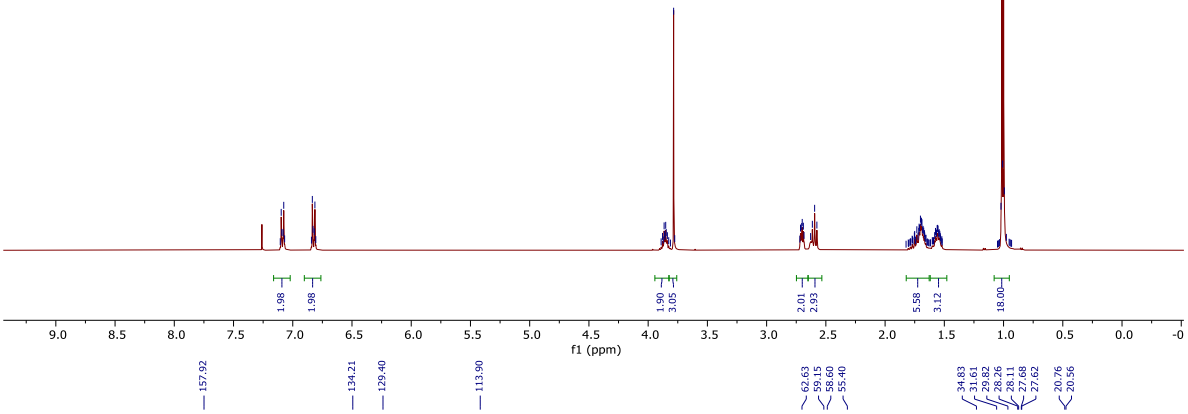
Compound 22 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



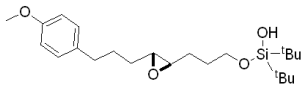


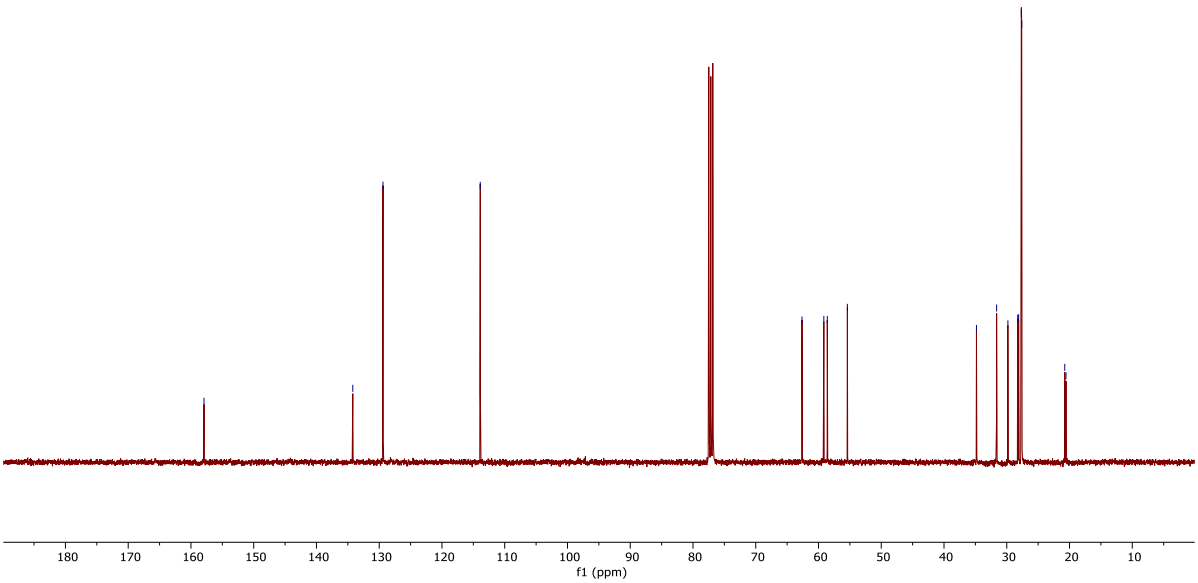
Compound 23 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



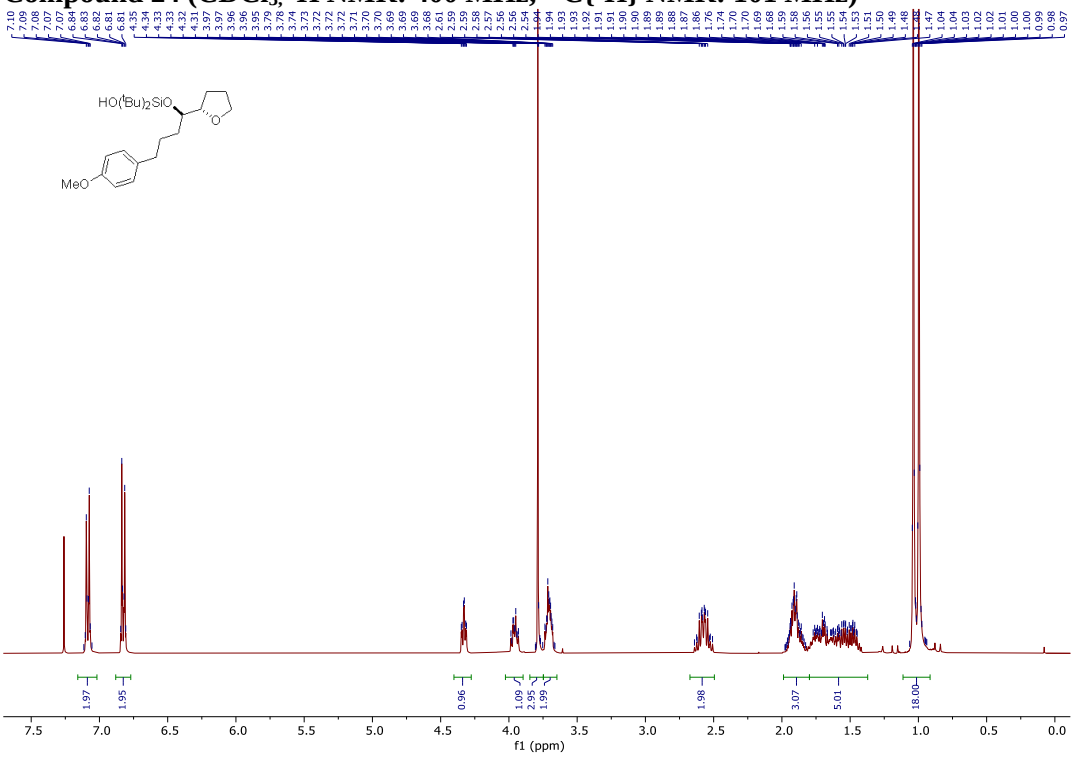


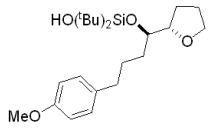
157.92
134.21
129.40
113.90
62.63
59.15
58.60
55.40
34.83
31.61
29.82
28.26
28.11
27.68
27.62
20.76
20.56

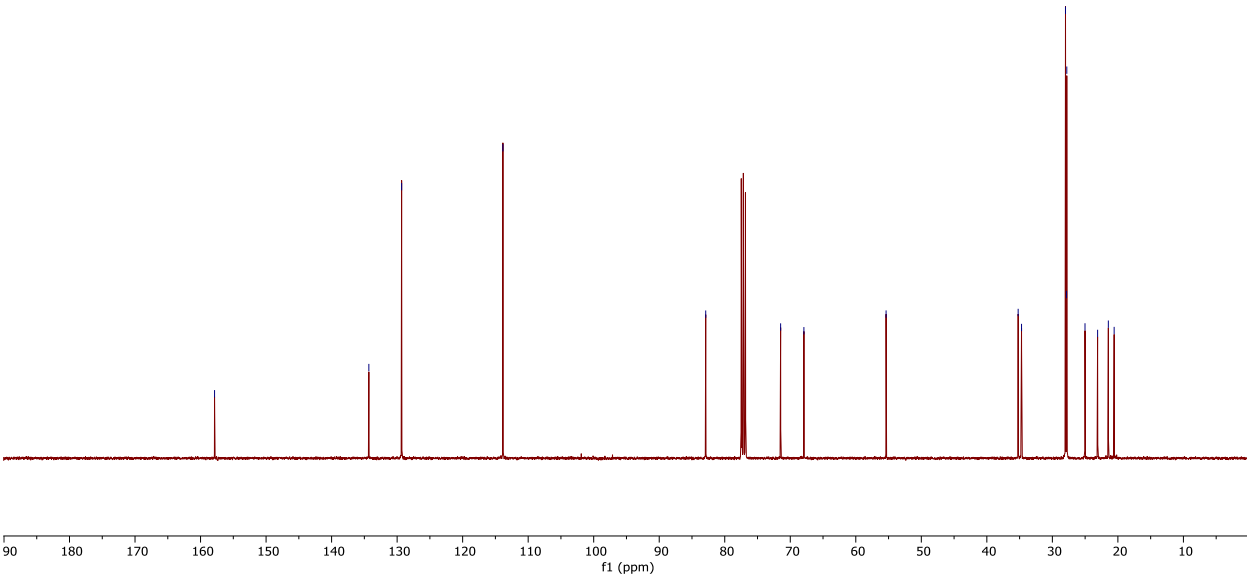




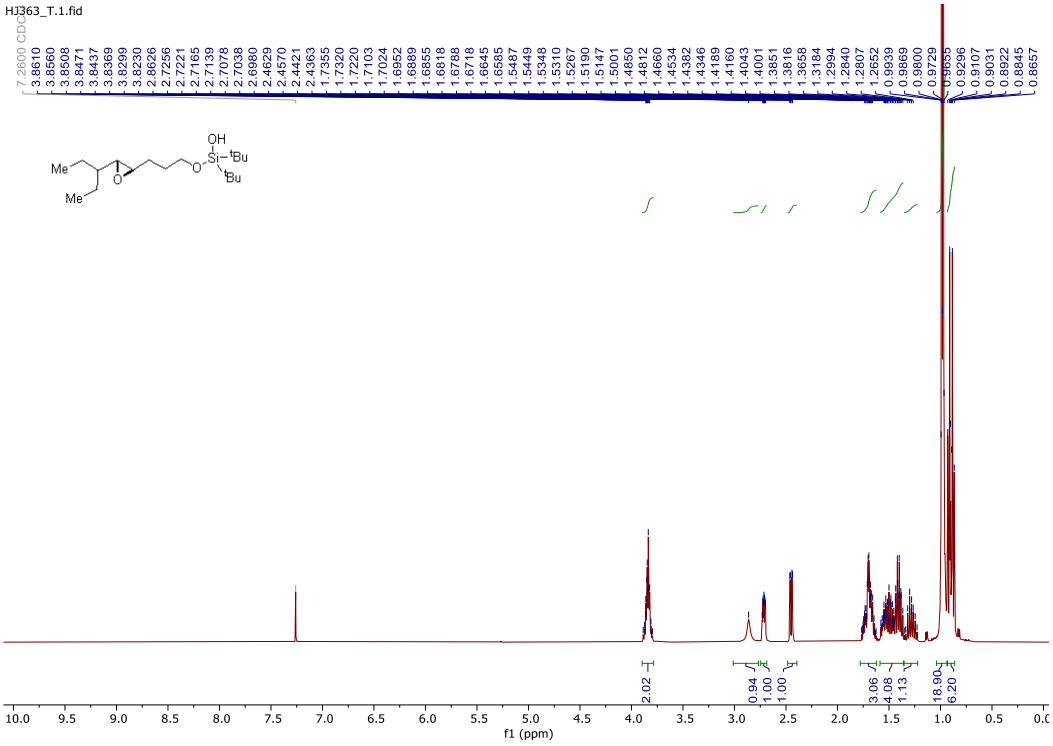
Compound 24 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

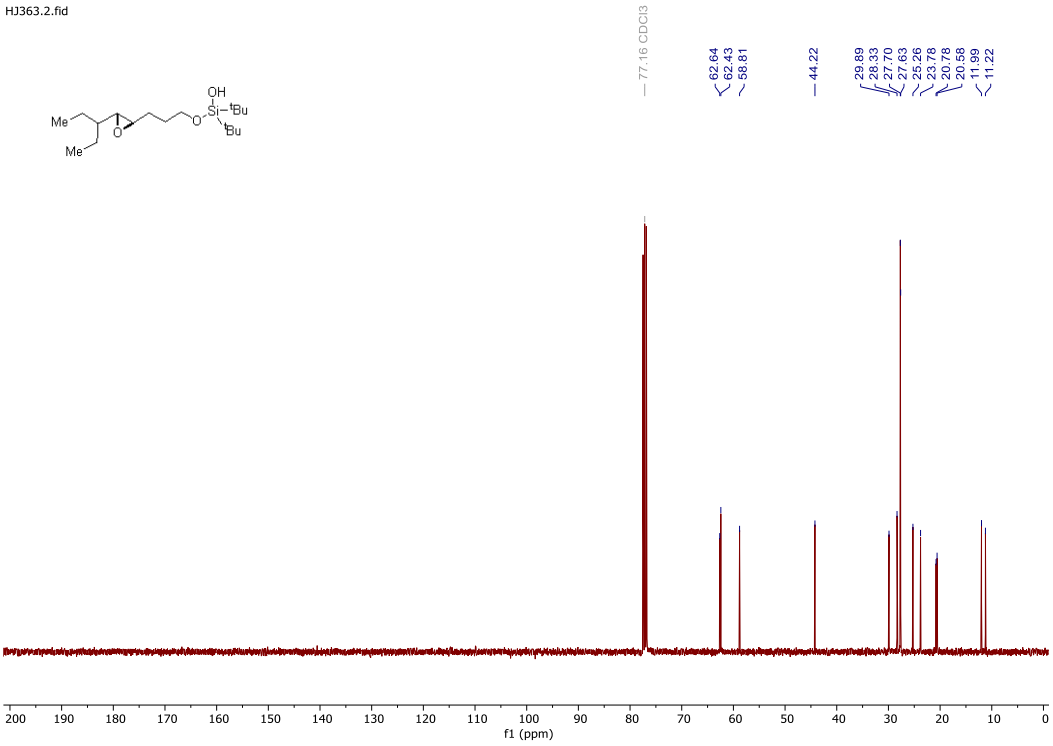




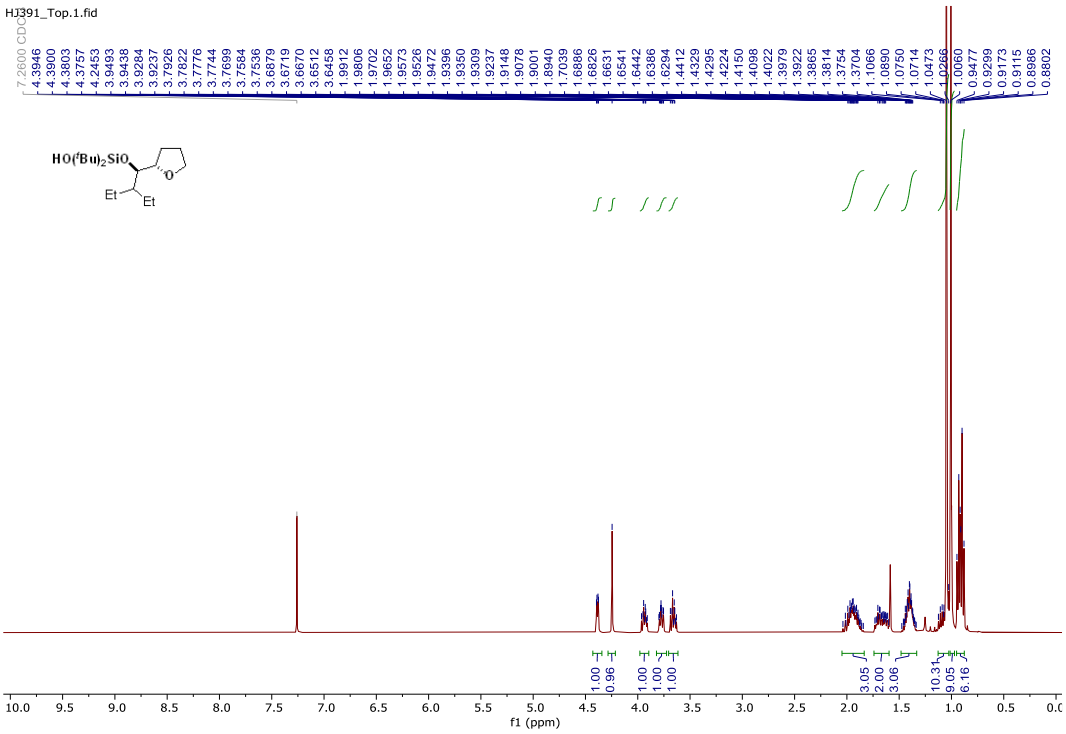


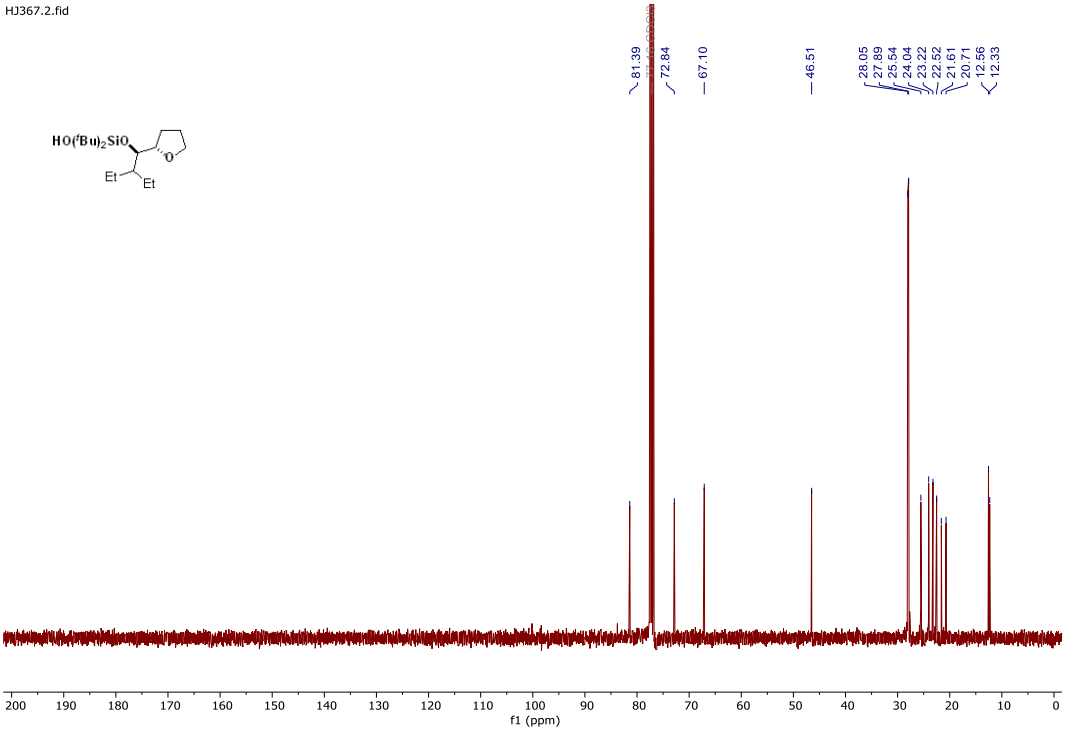
Compound 25 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



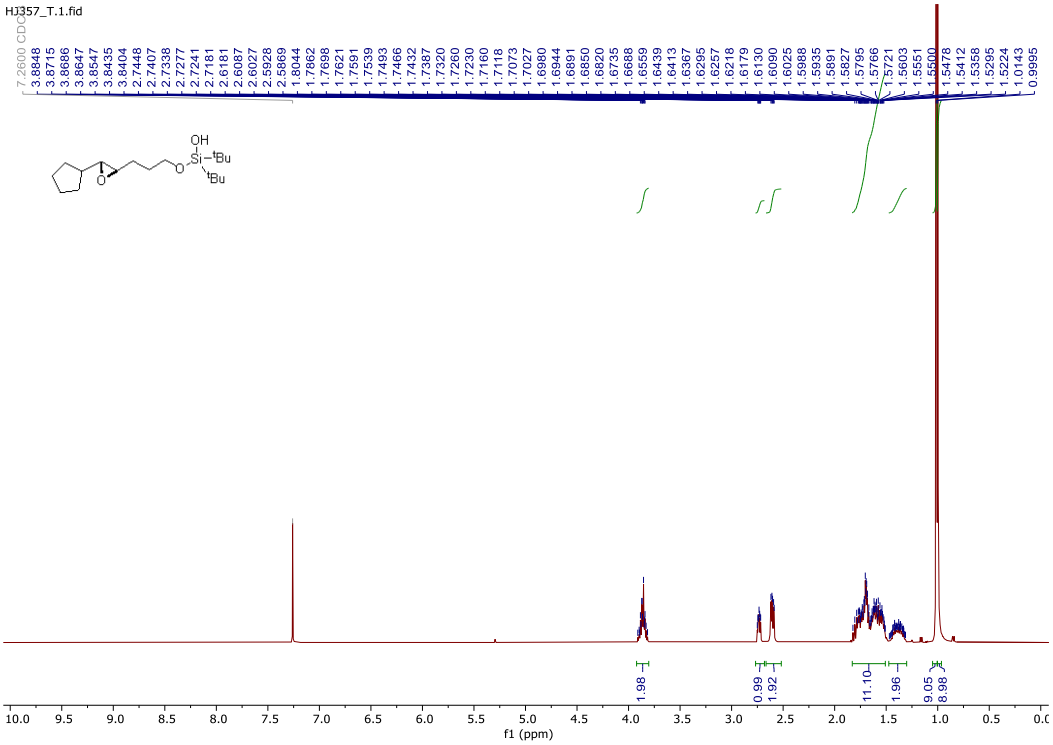


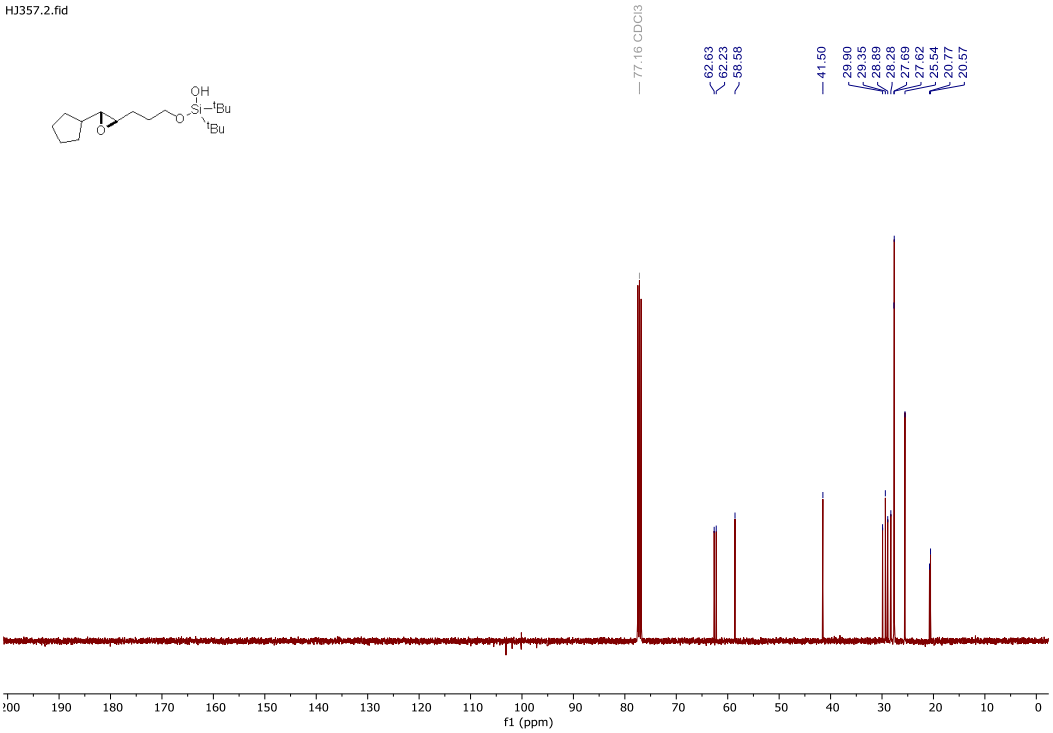
Compound 26 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



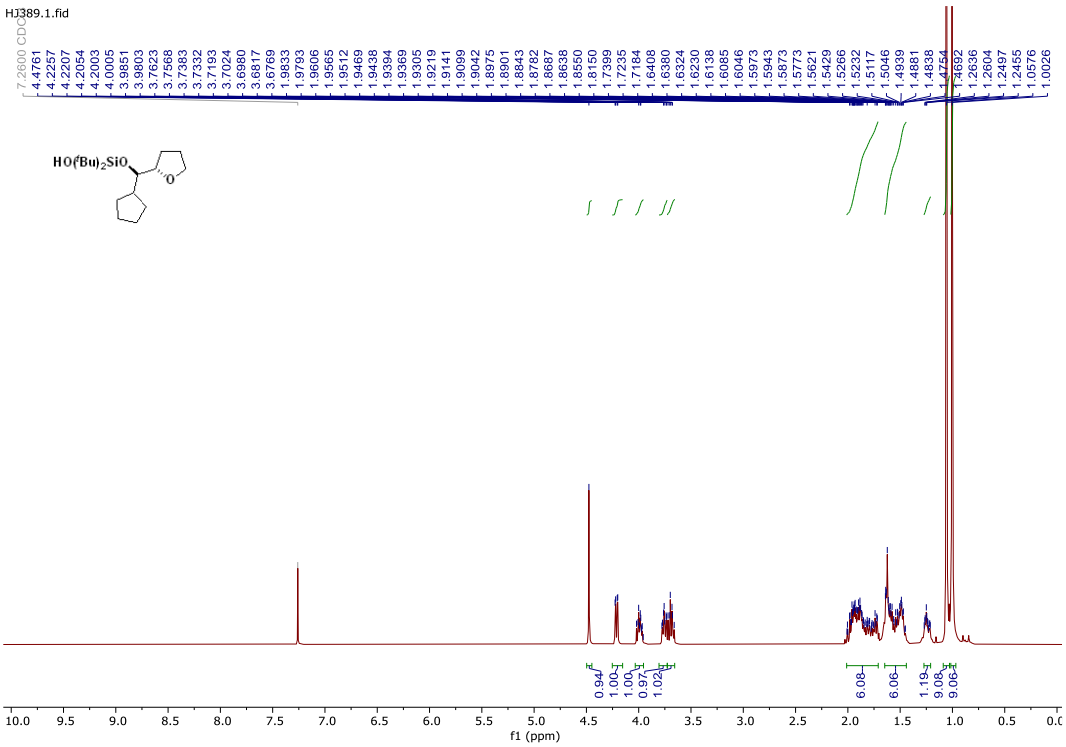


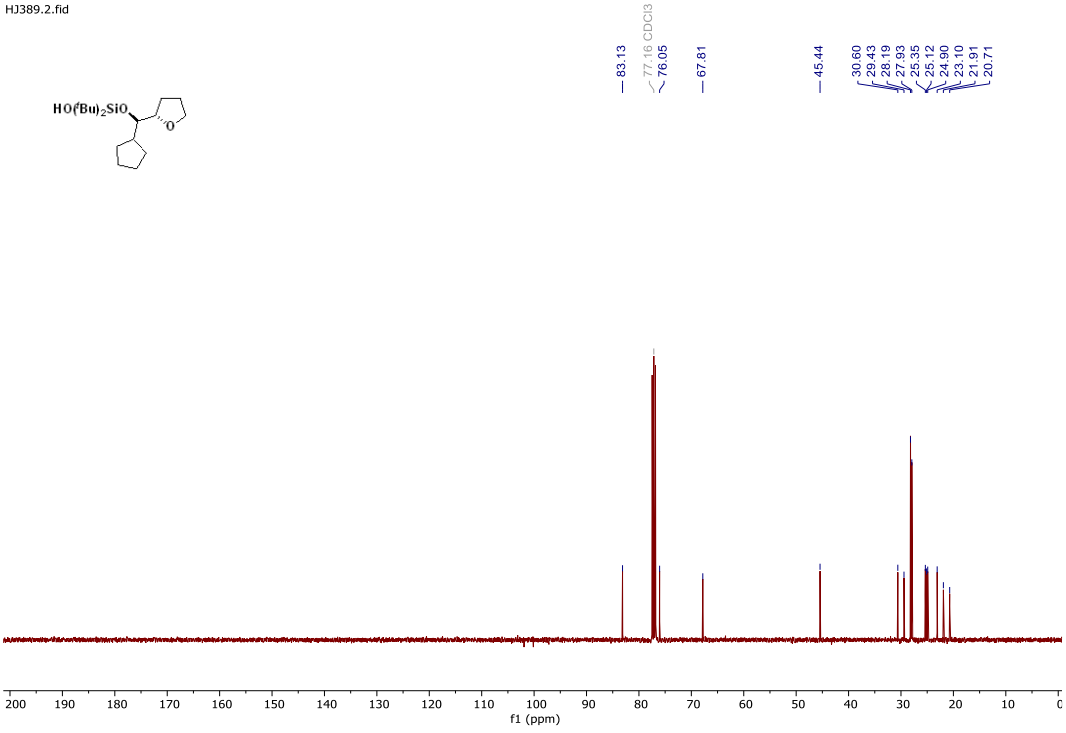
Compound 27 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



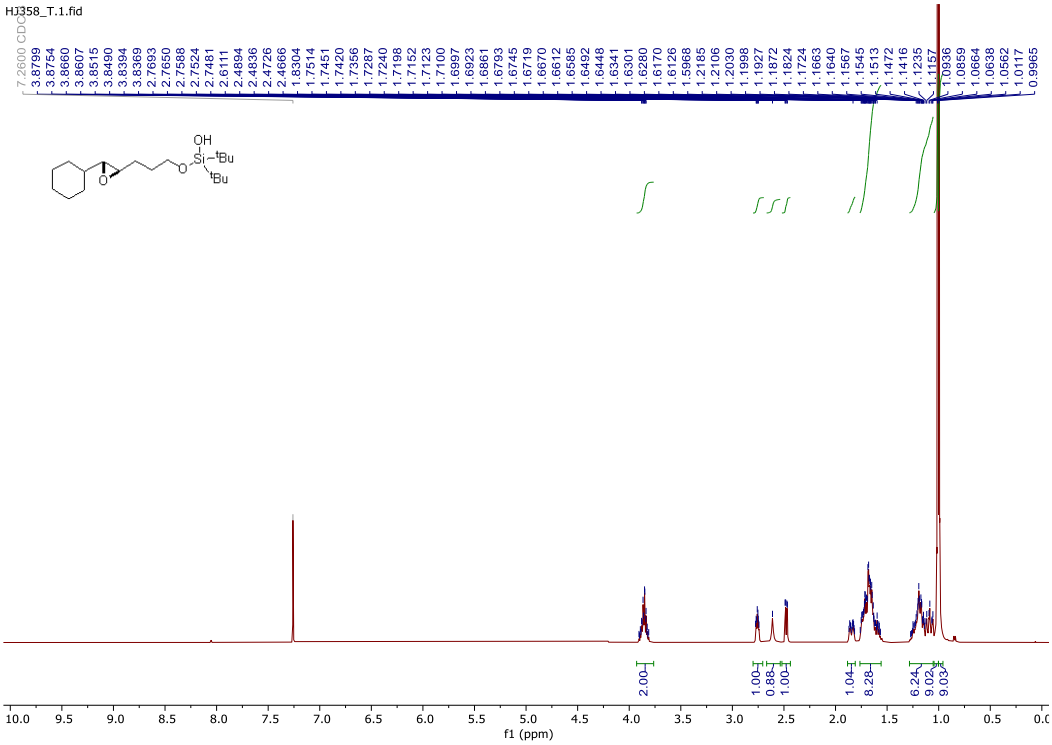


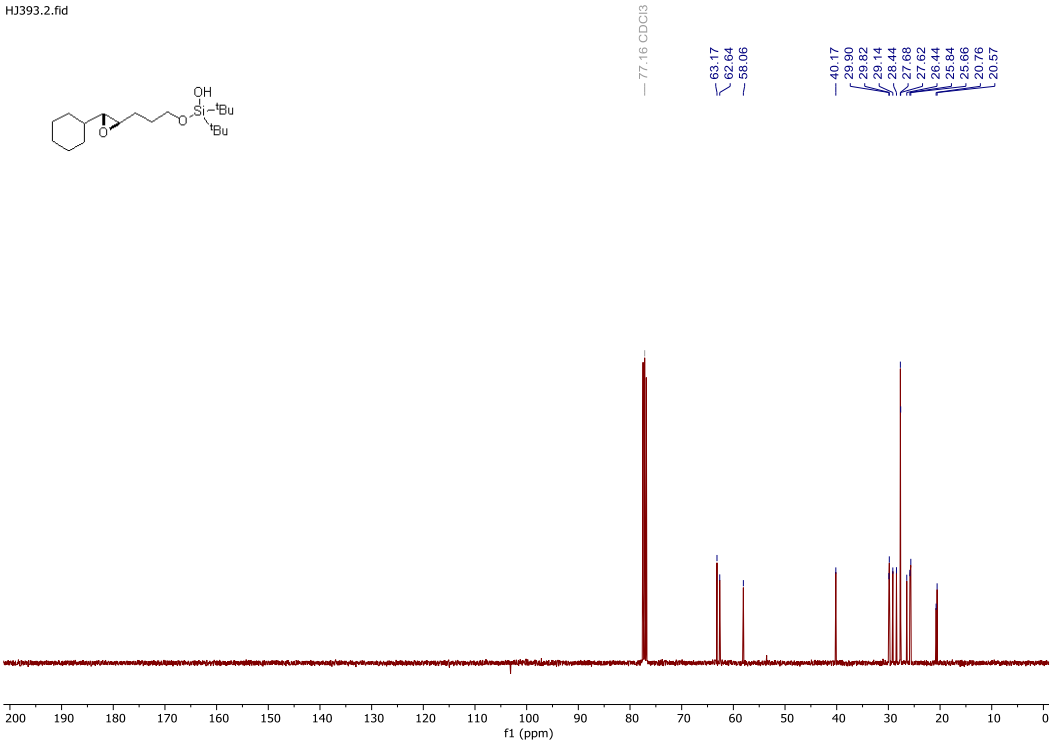
Compound 28 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



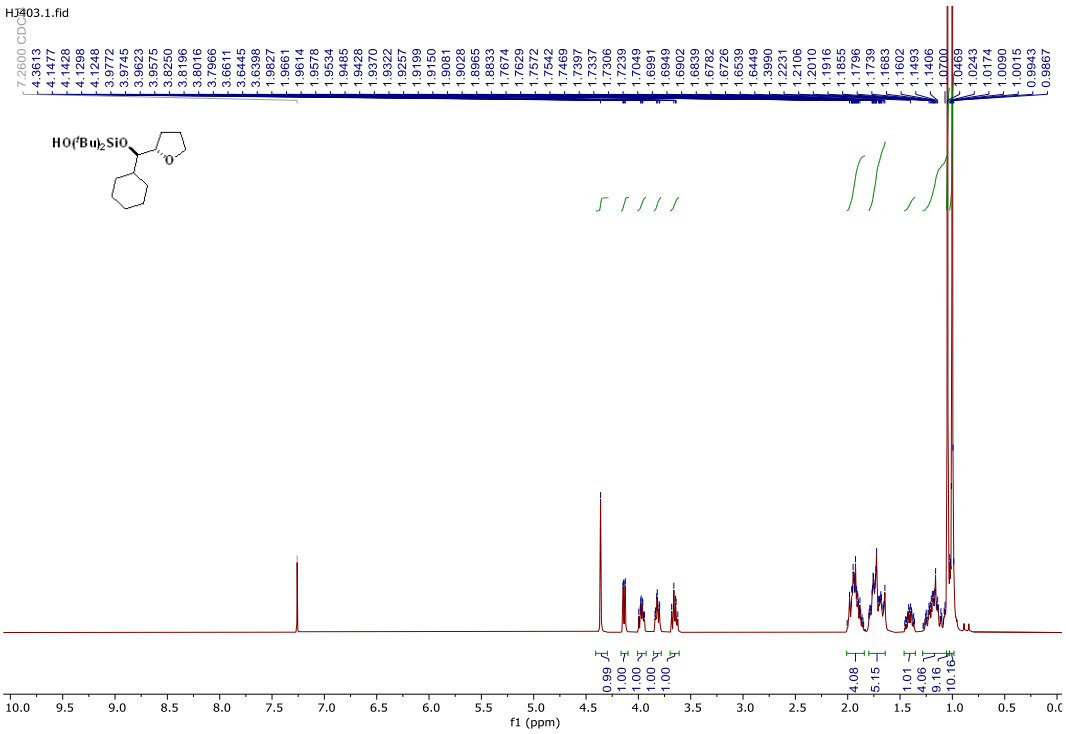


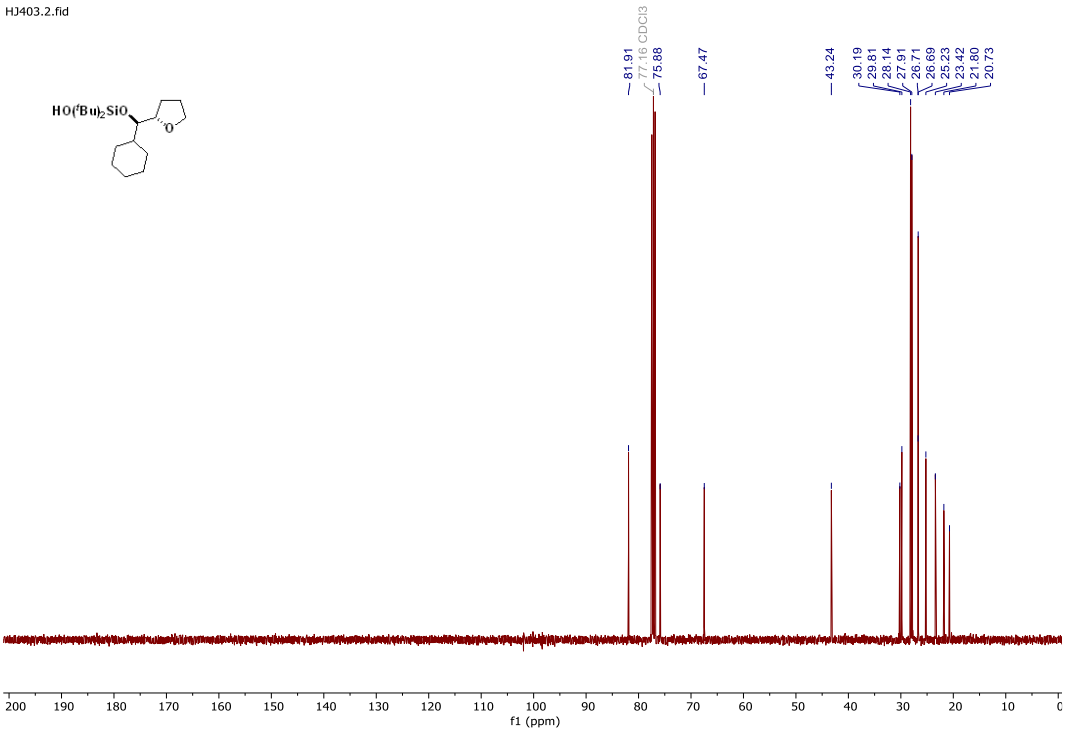
Compound 29 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



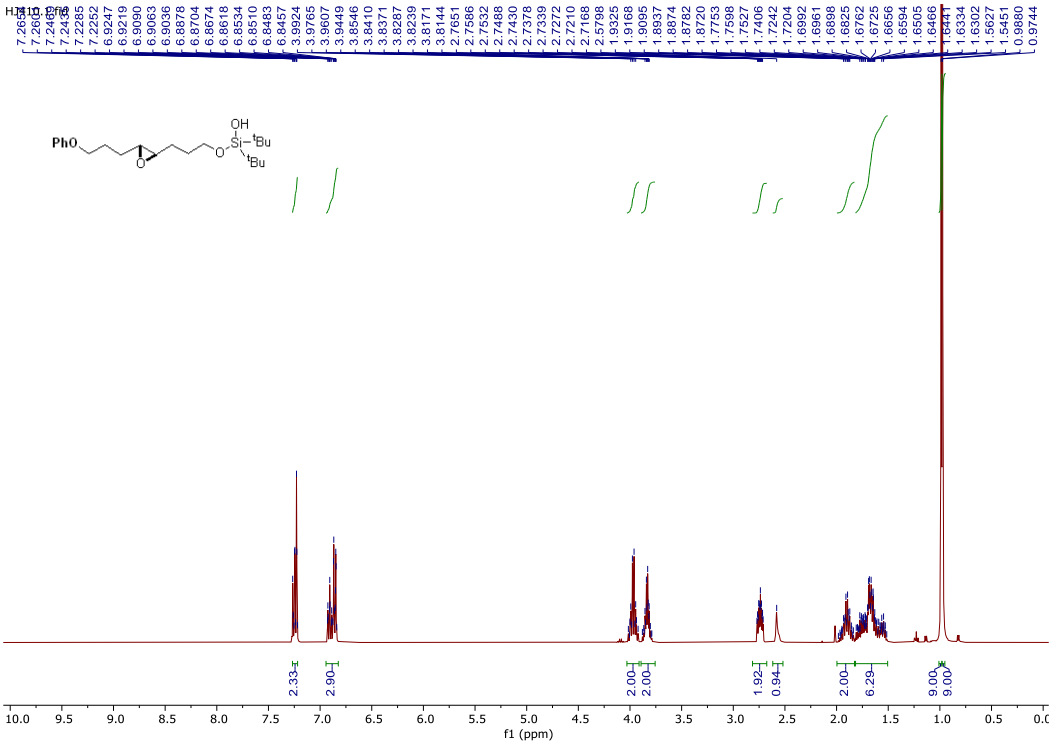


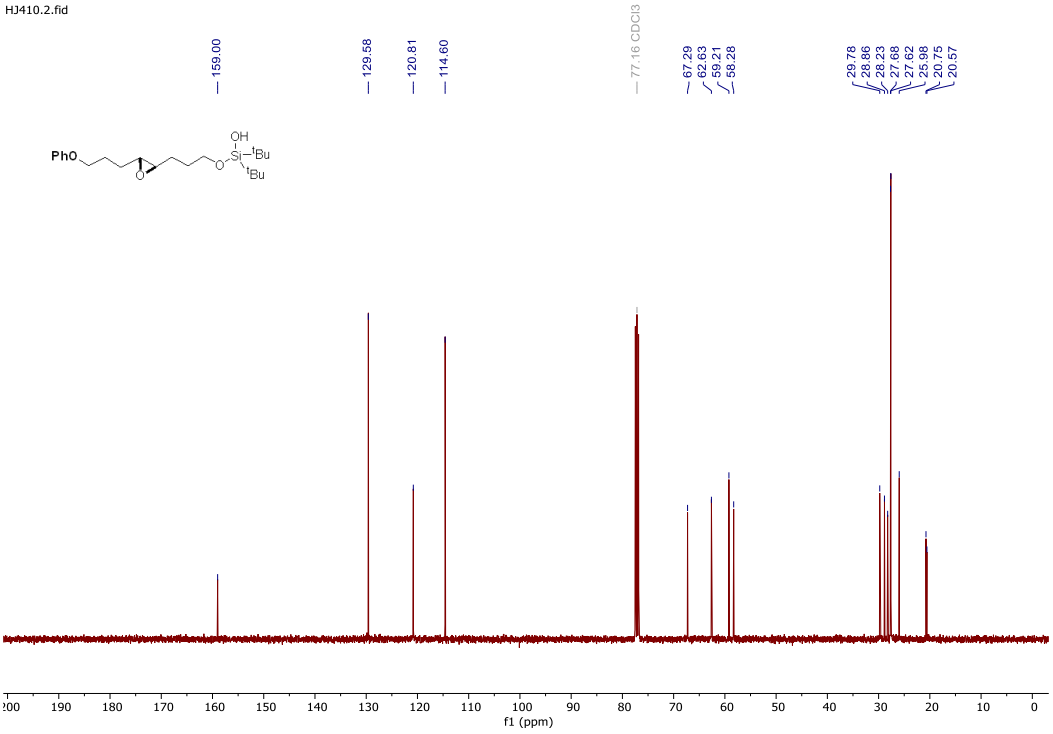
Compound 30 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



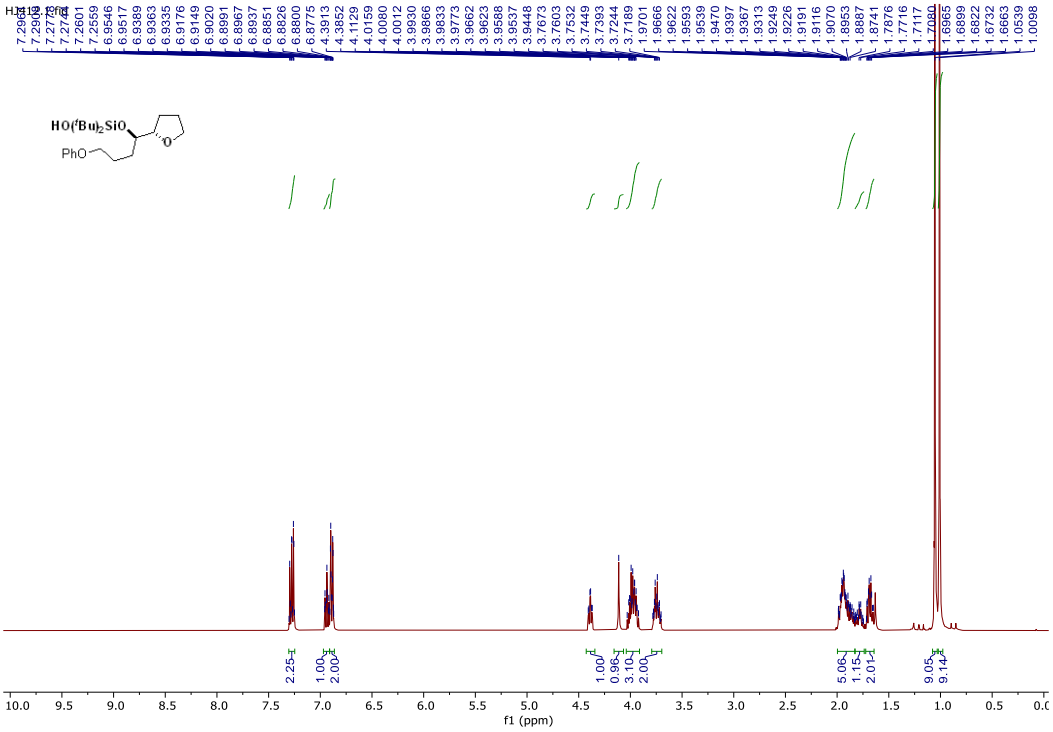


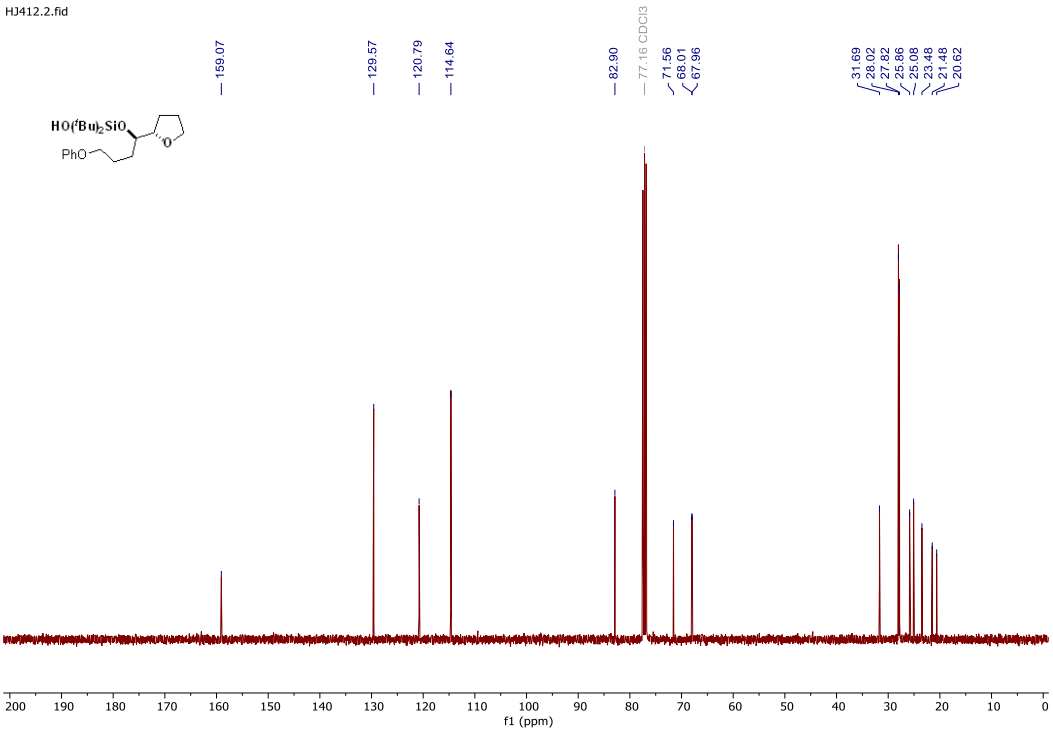
Compound 31 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



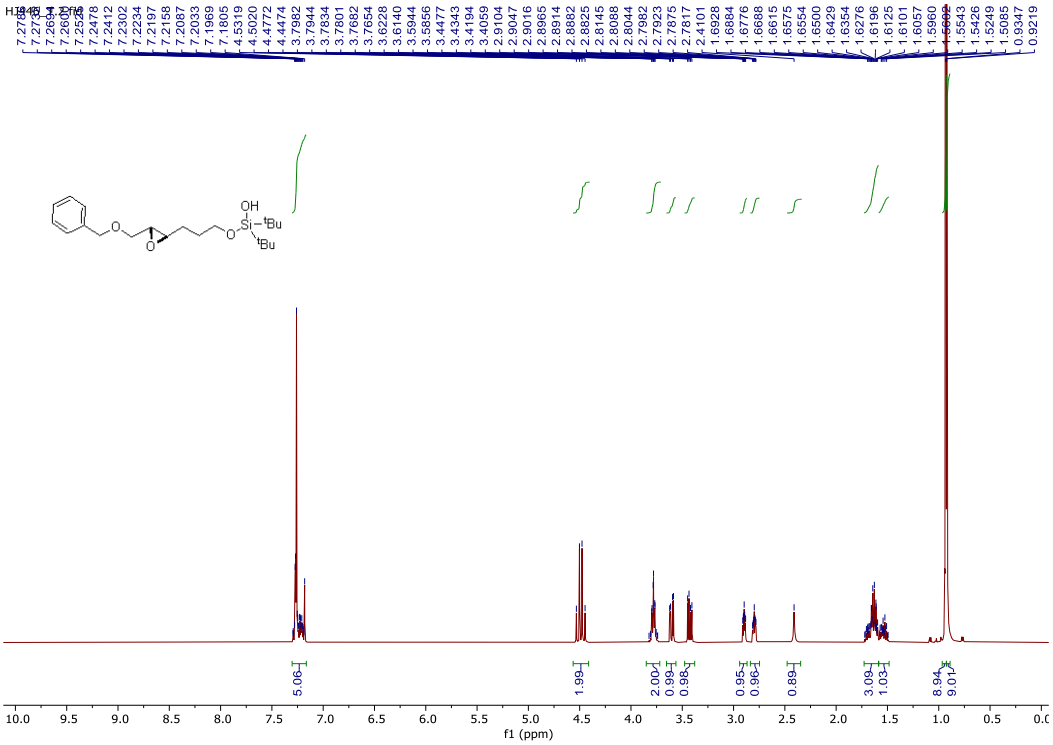


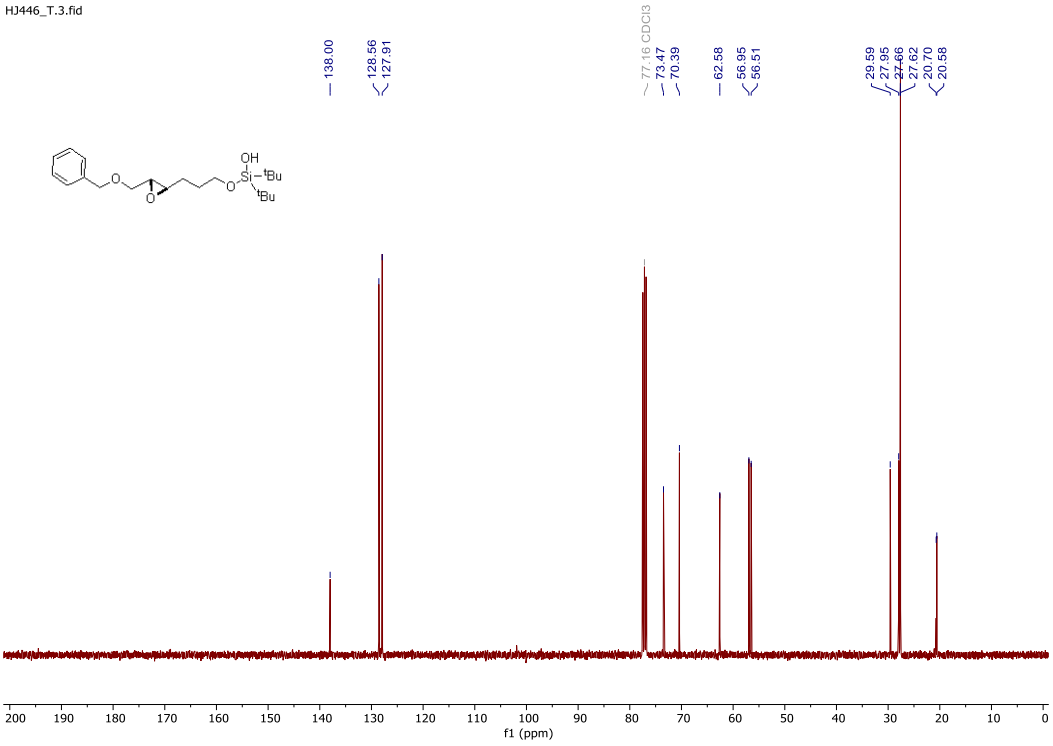
Compound 32 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



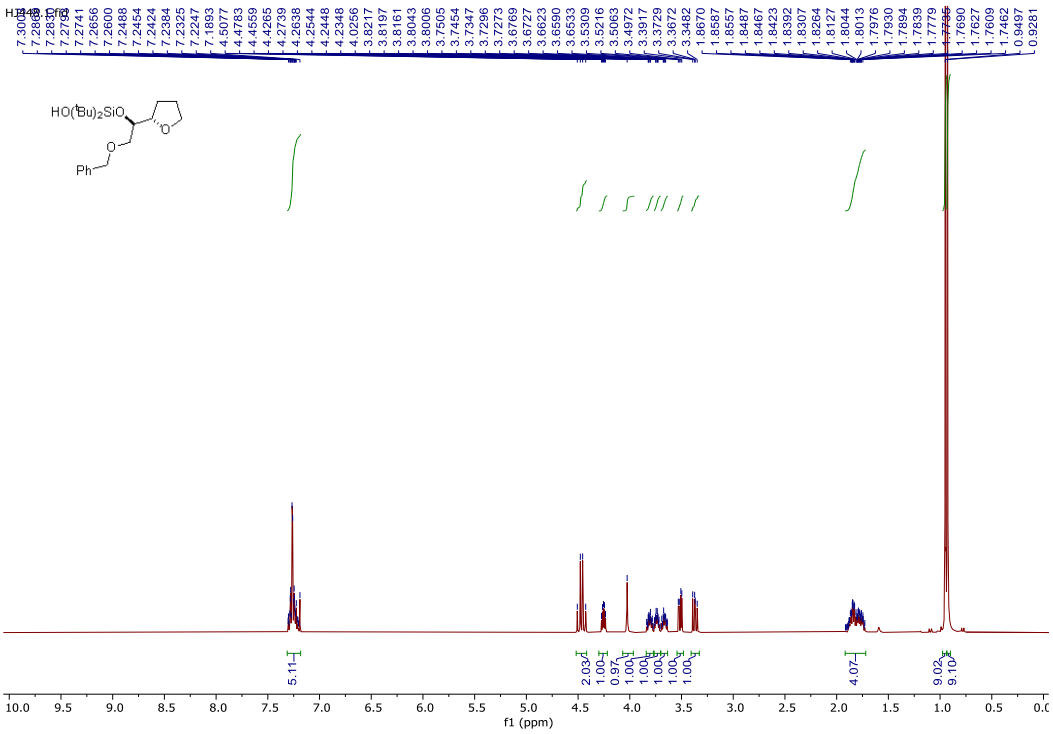


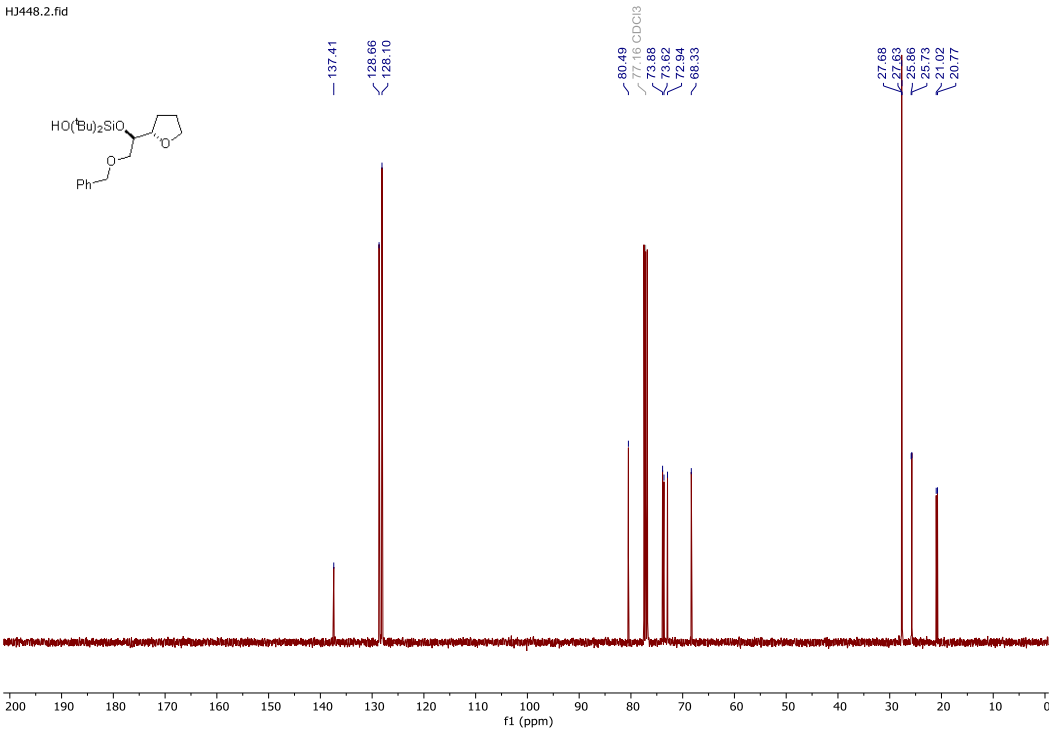
Compound 33 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



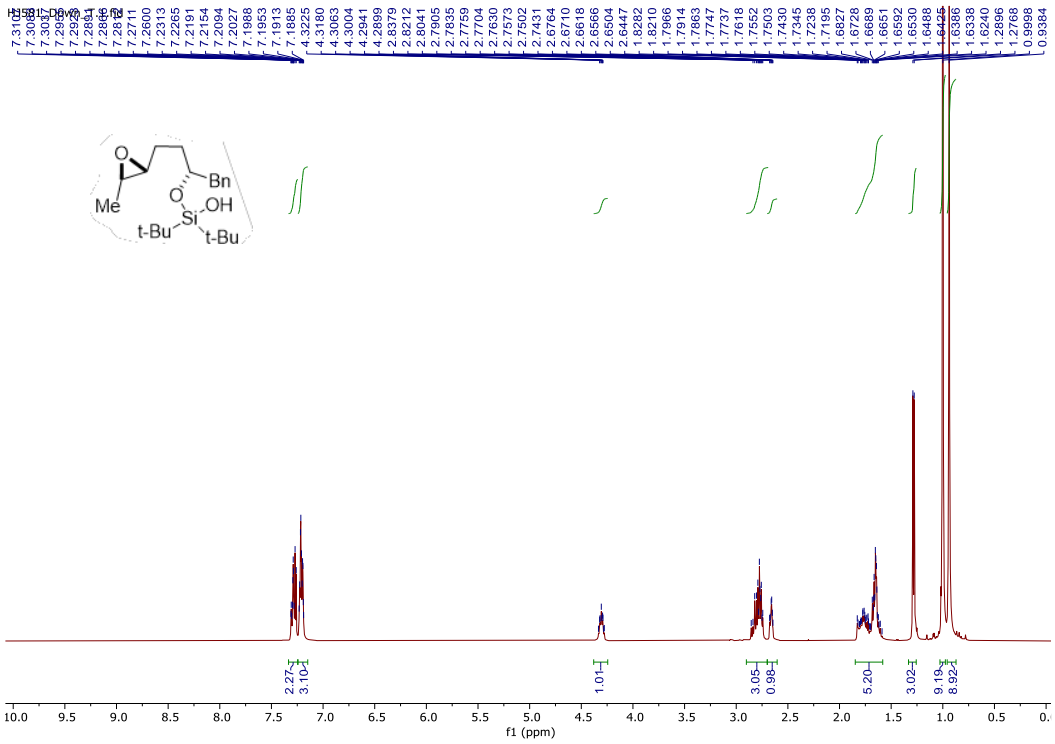


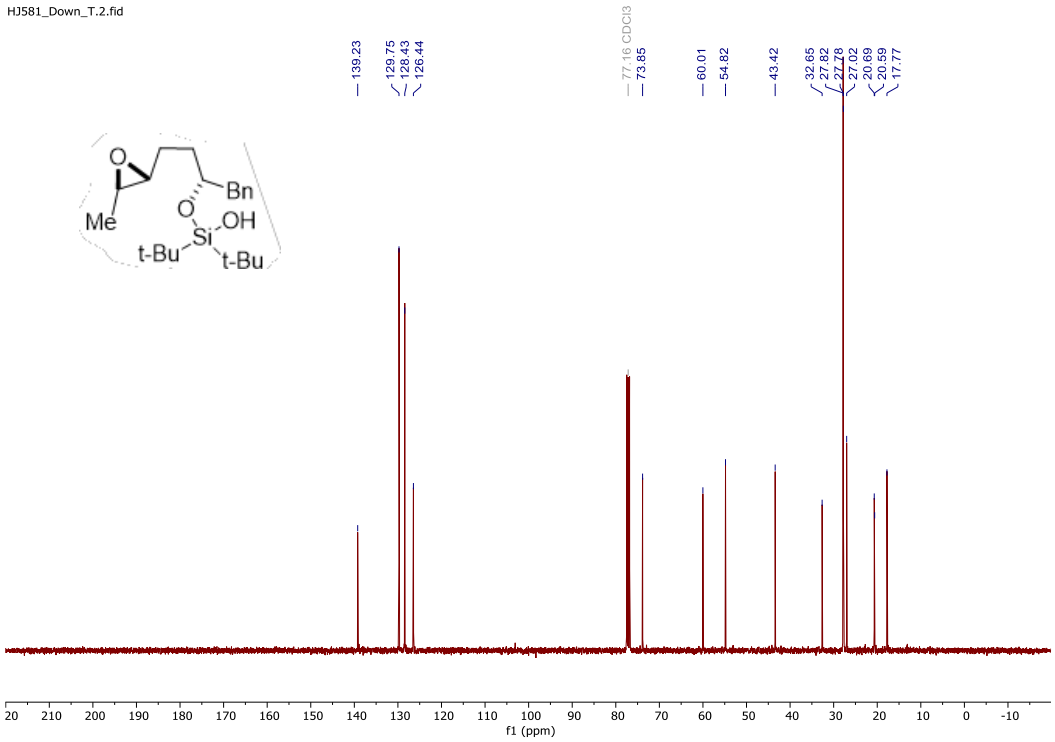
Compound 34 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



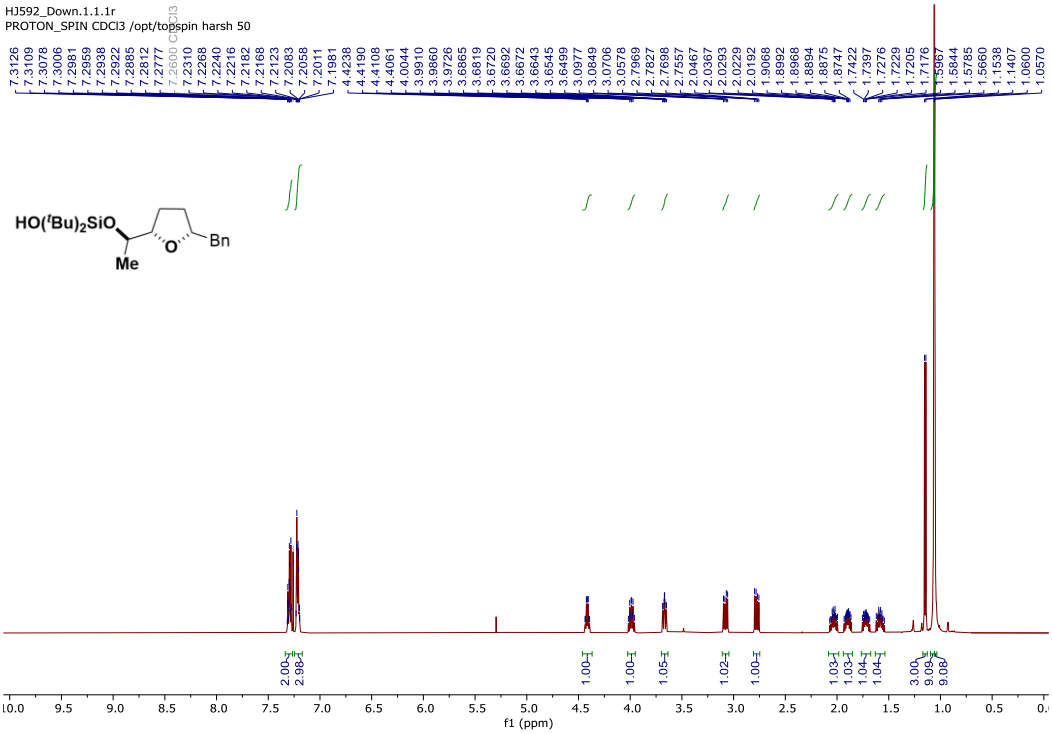


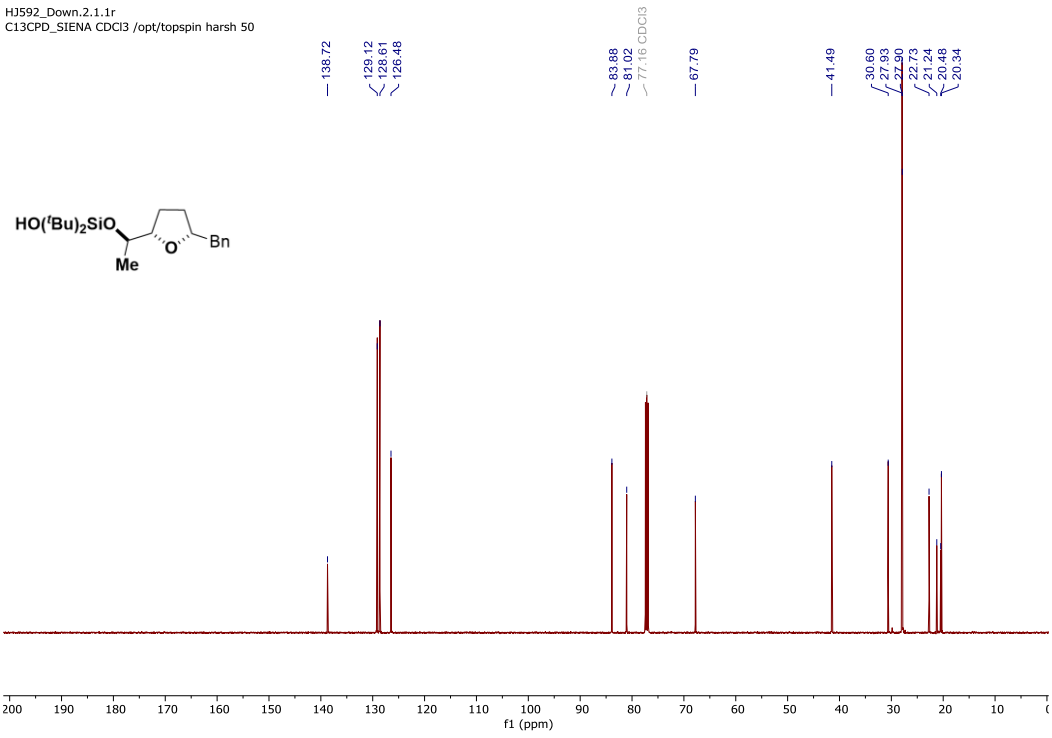
Compound 35 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



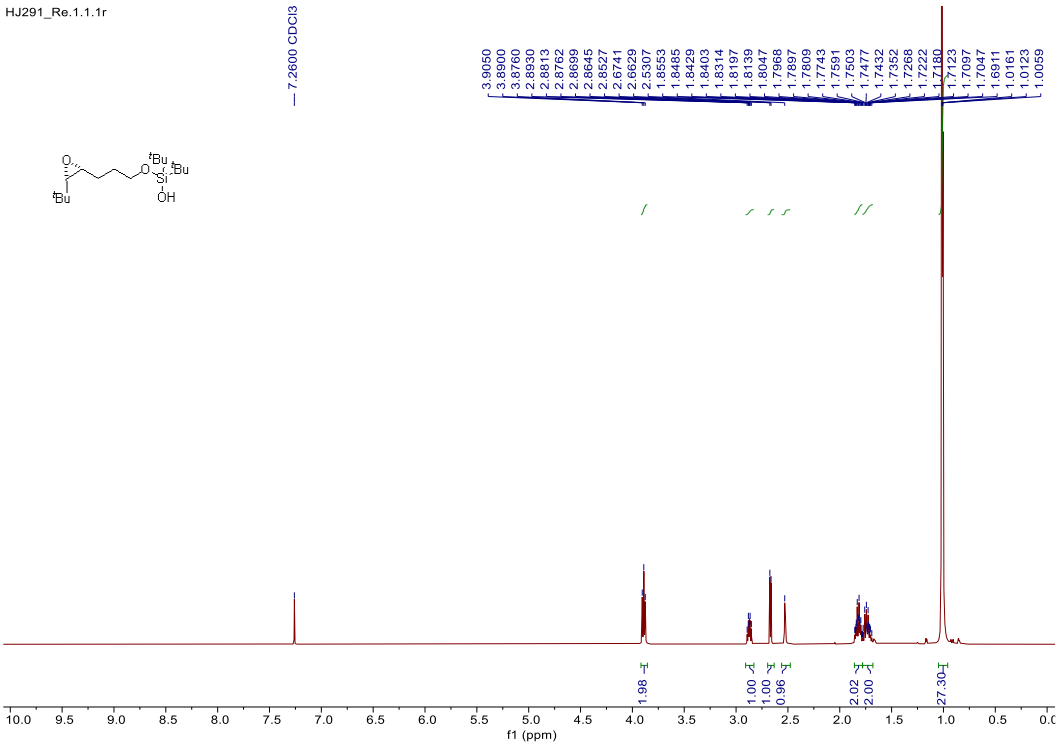


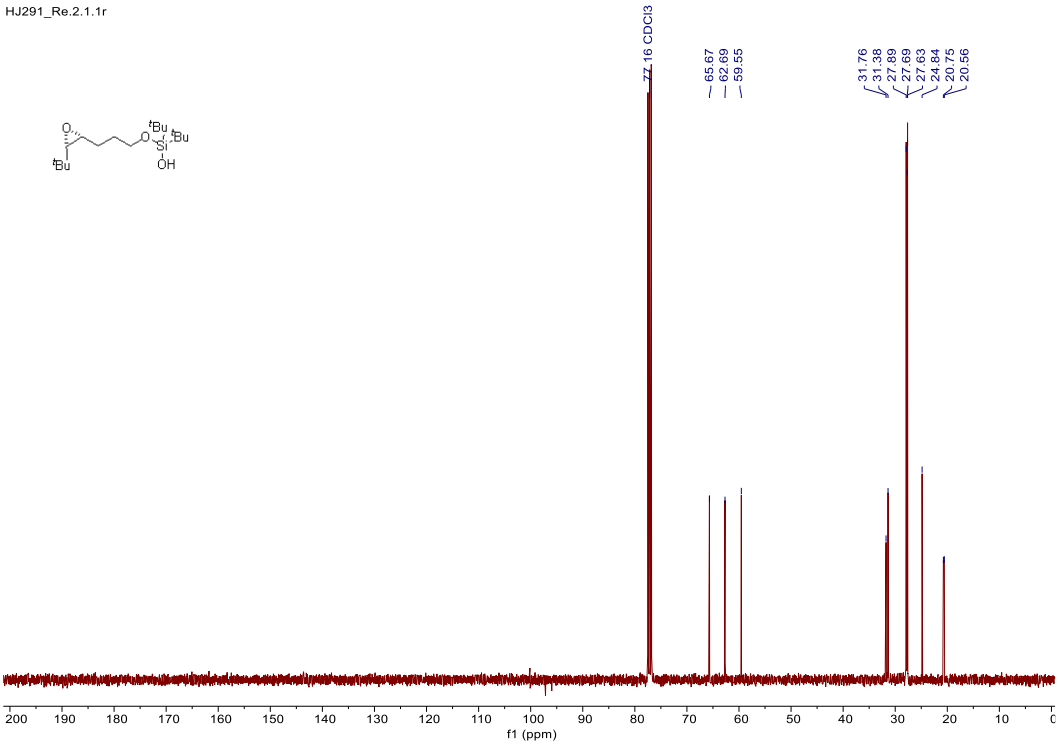
Compound 36 ($CDCl_3$, ^1H NMR: 500 MHz, $^{13}C\{^1H\}$ NMR: 126 MHz)



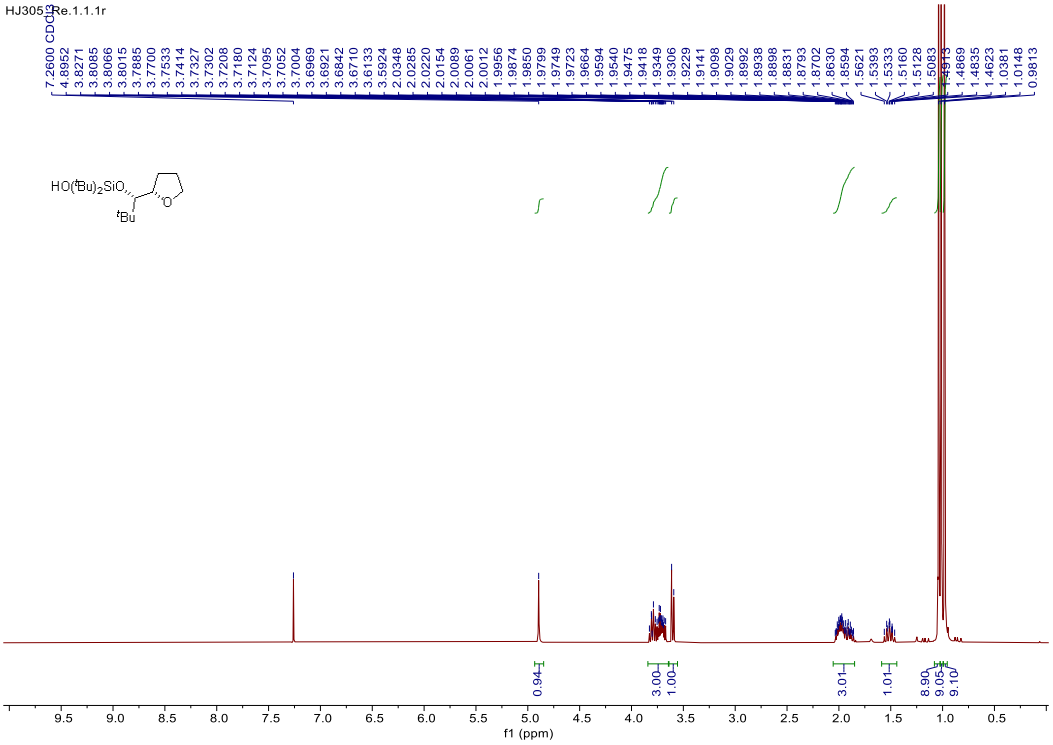


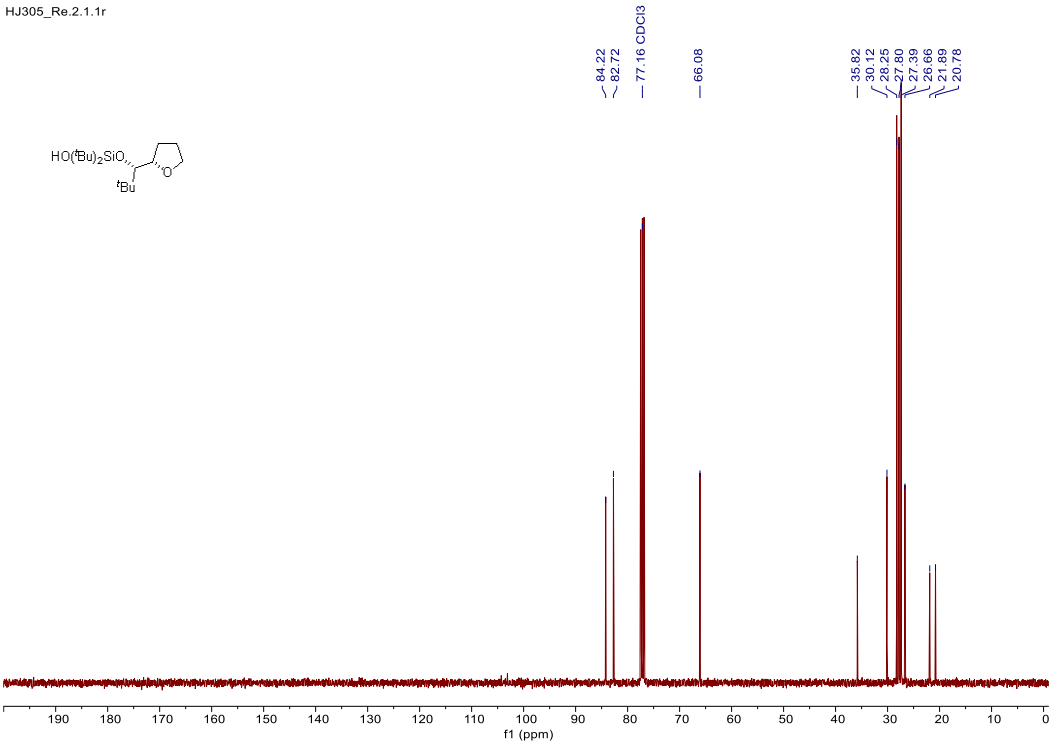
Compound 39 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



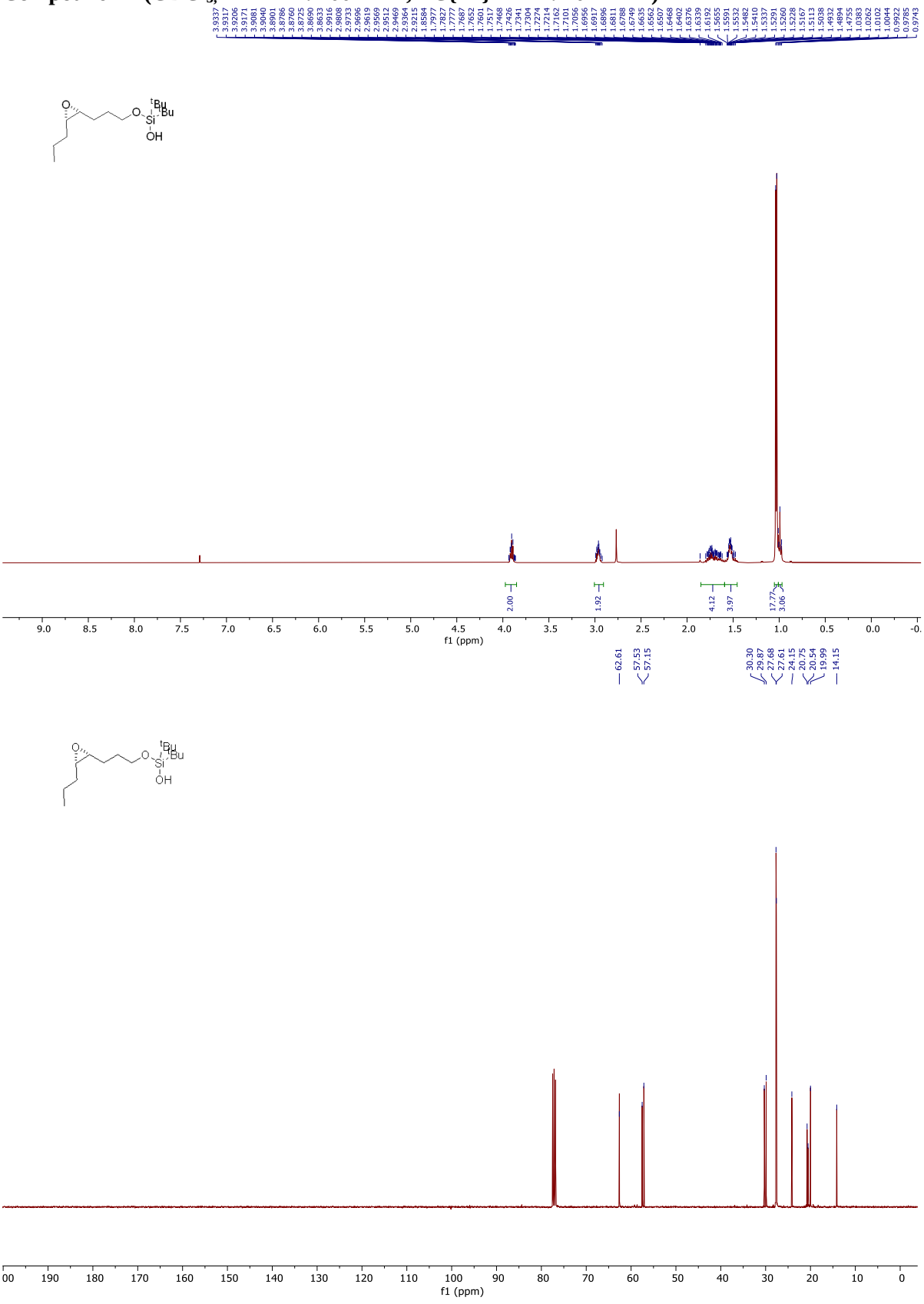


Compound 40 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

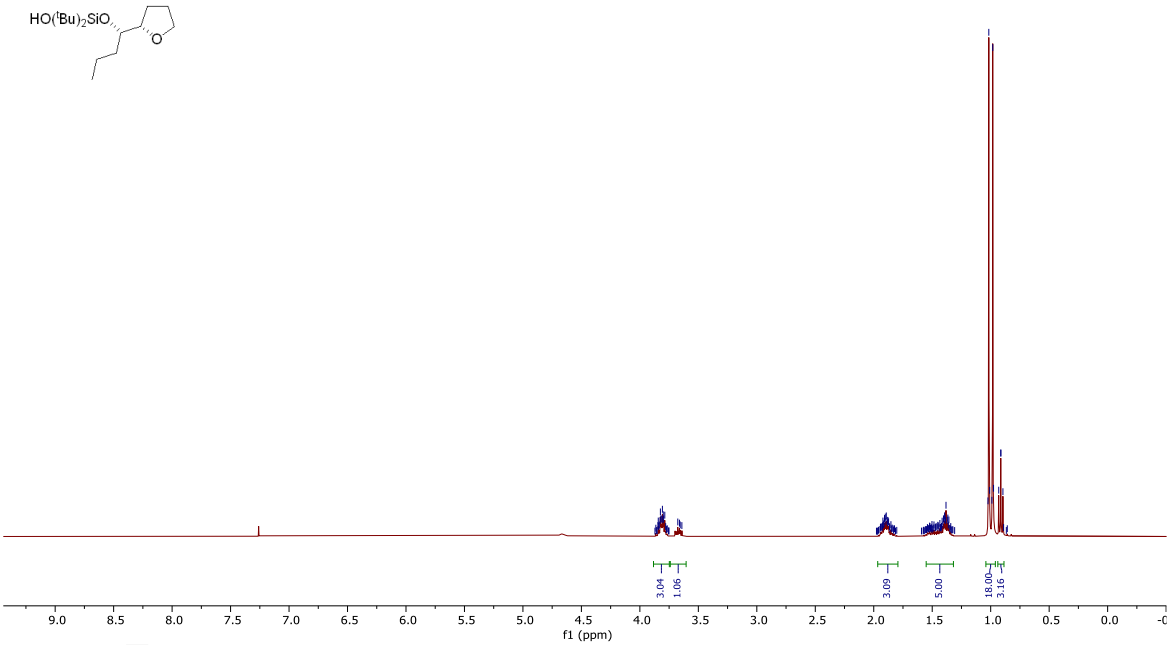


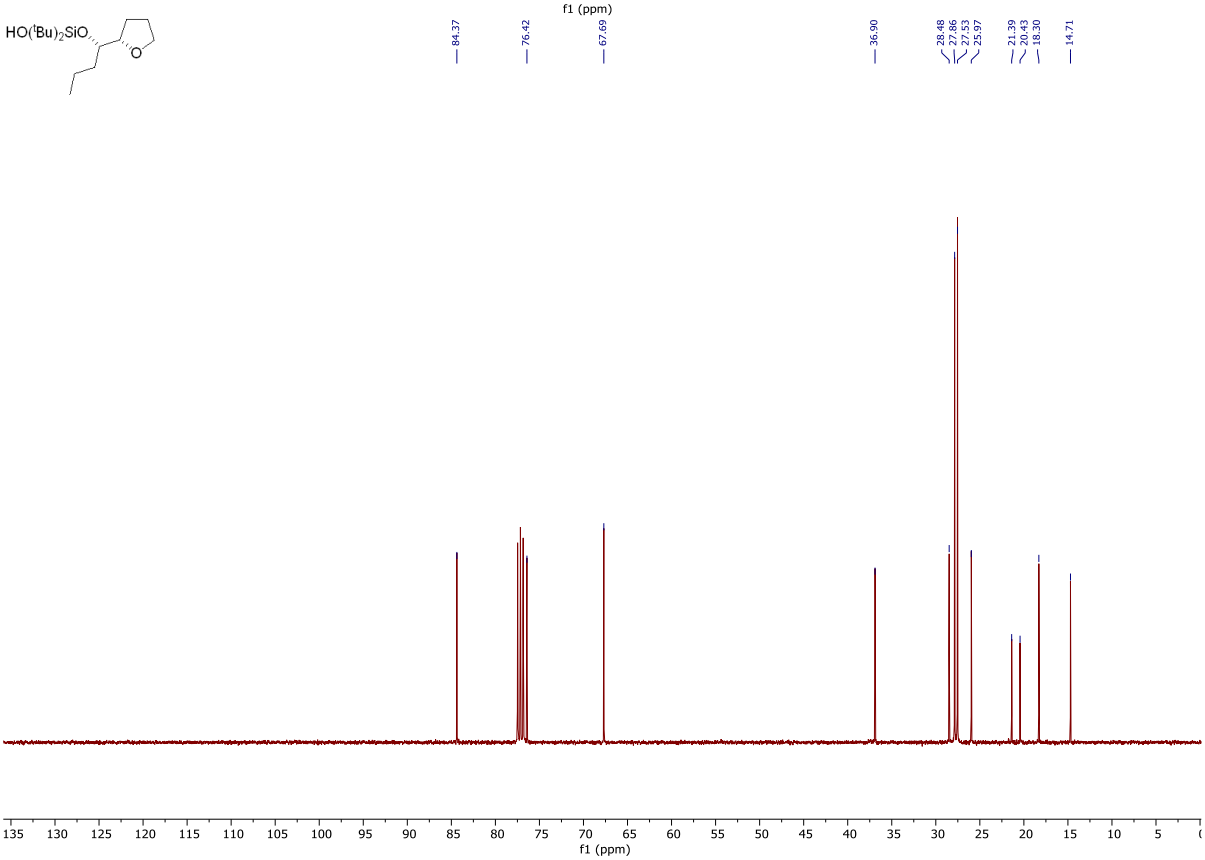


Compound 41 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

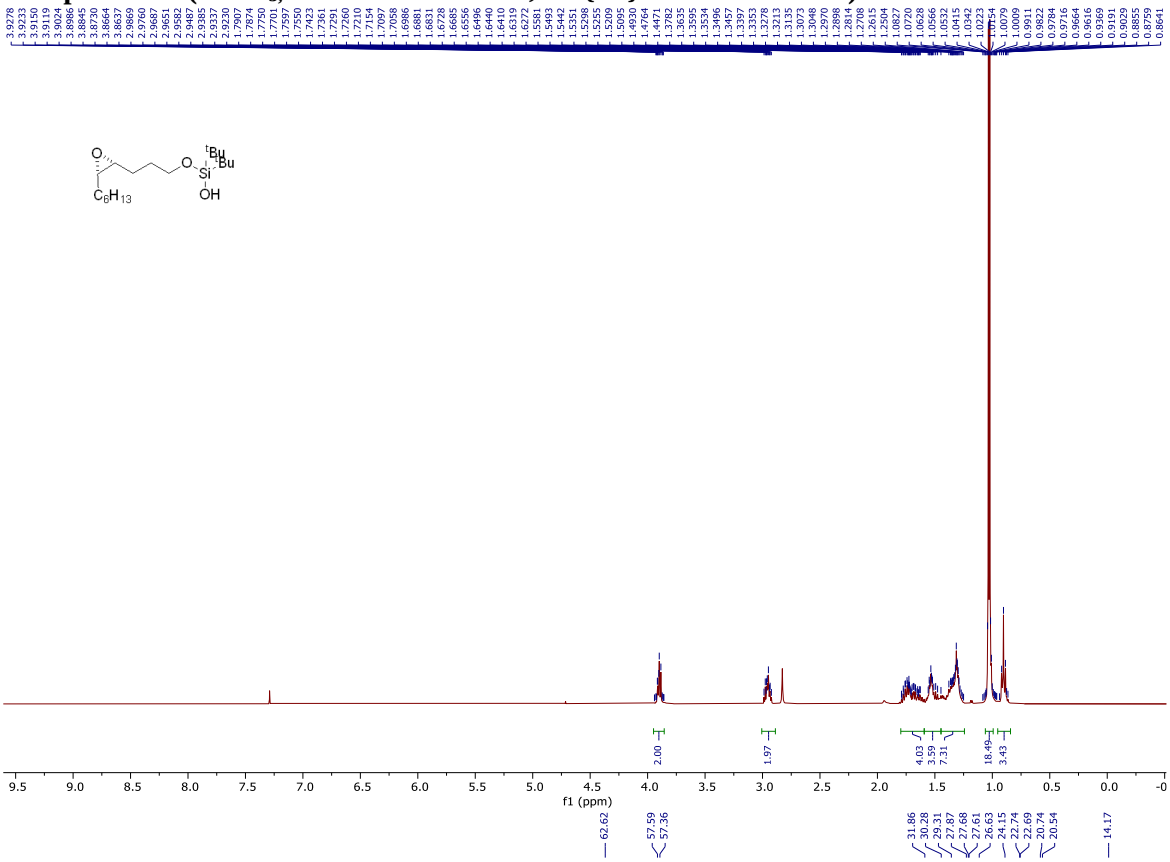


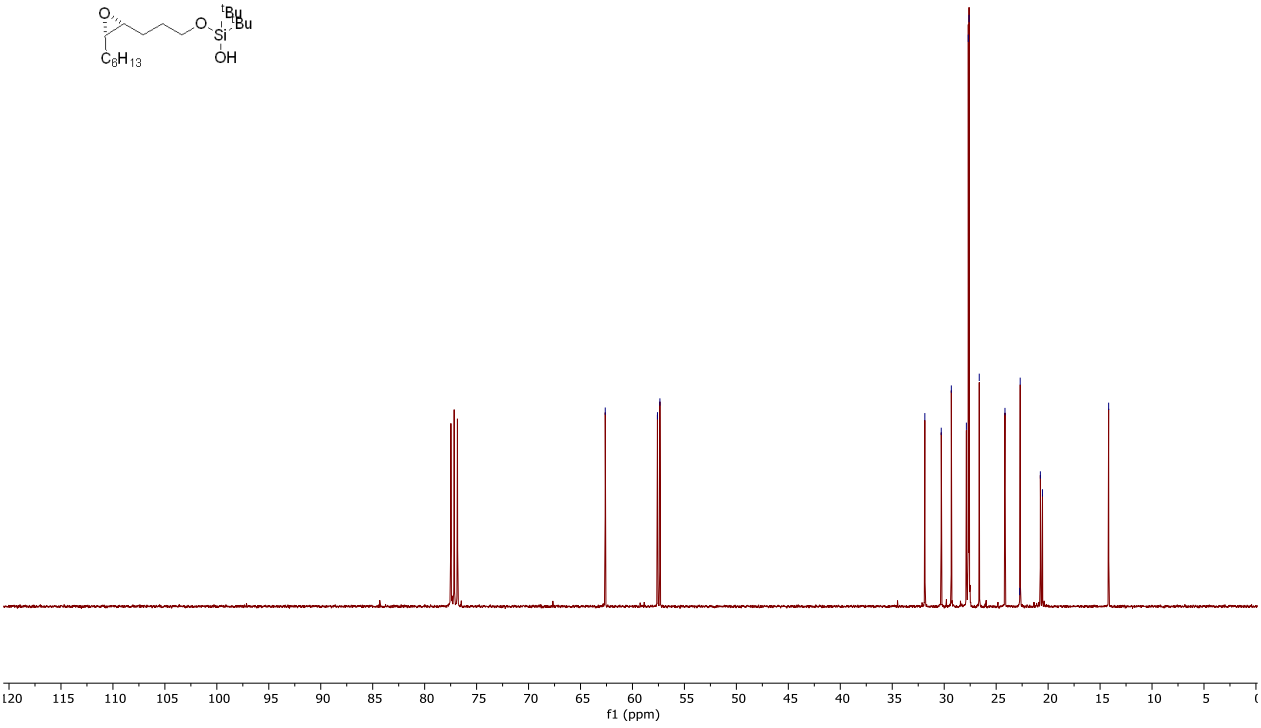
Compound 42 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



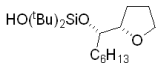


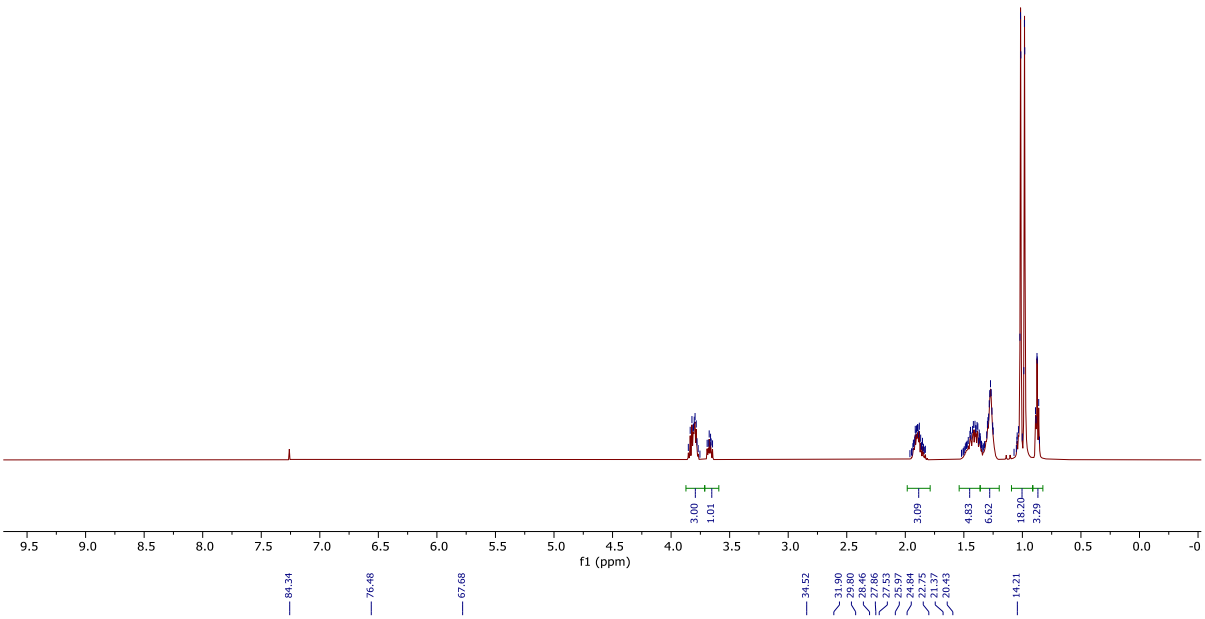
Compound 43 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

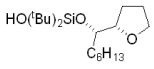


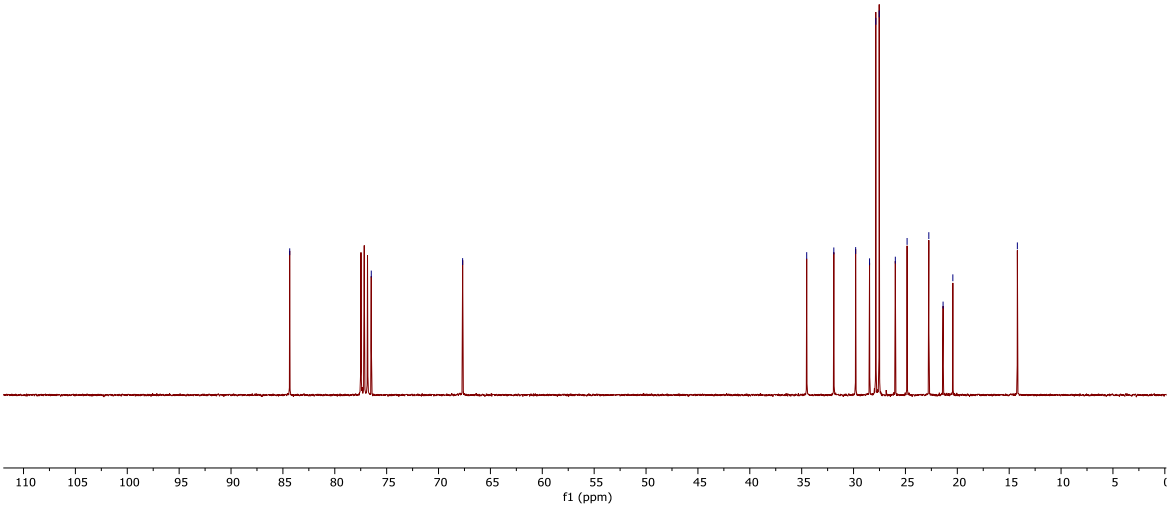


Compound 44 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

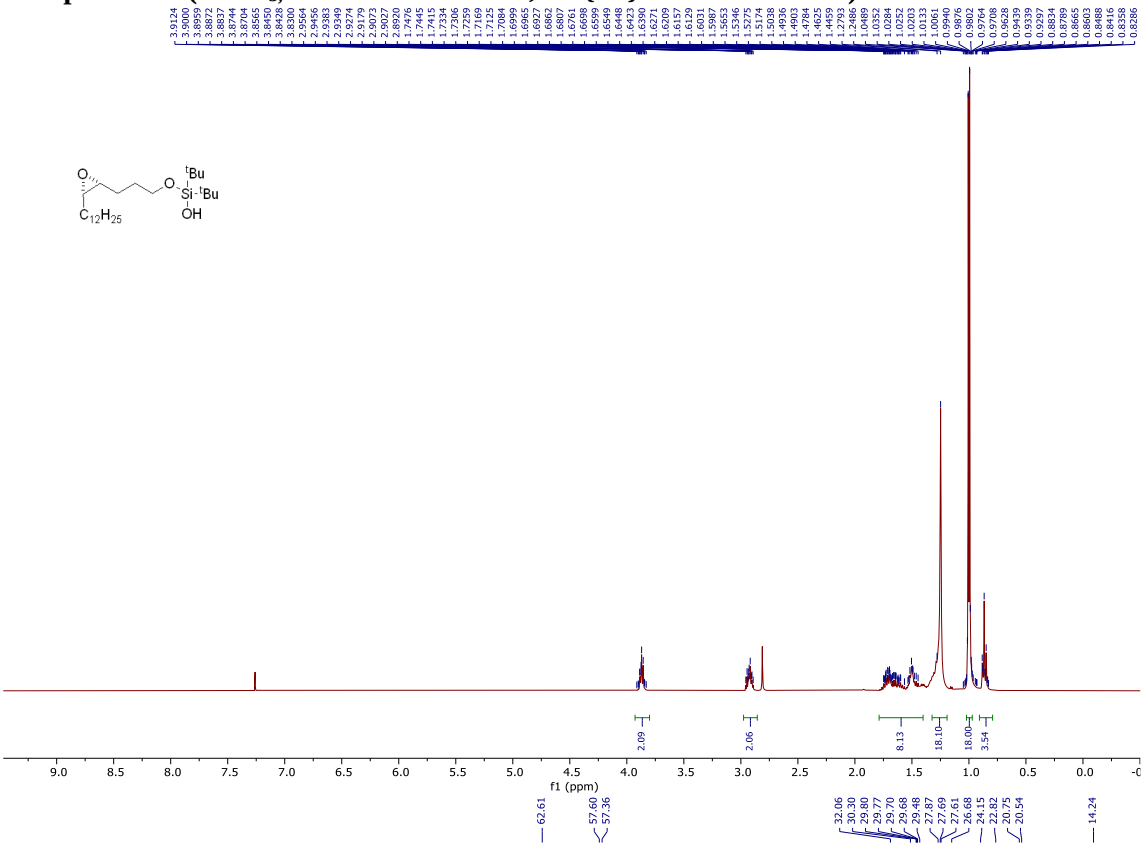


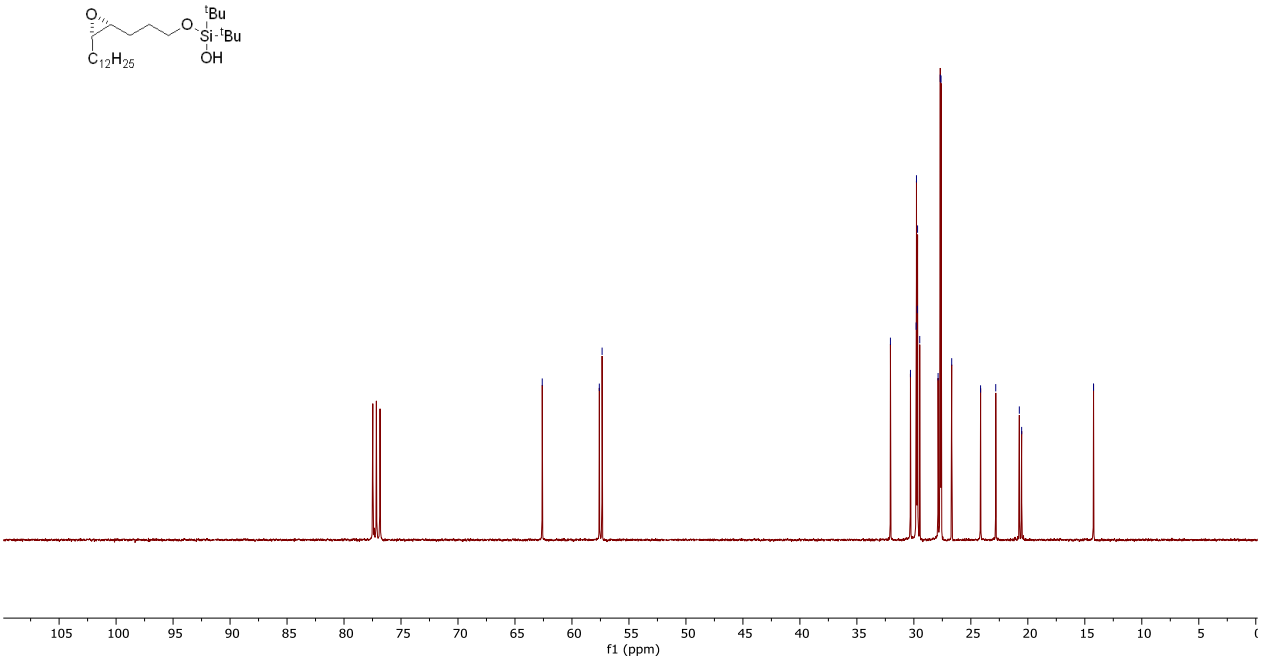




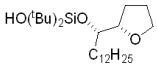


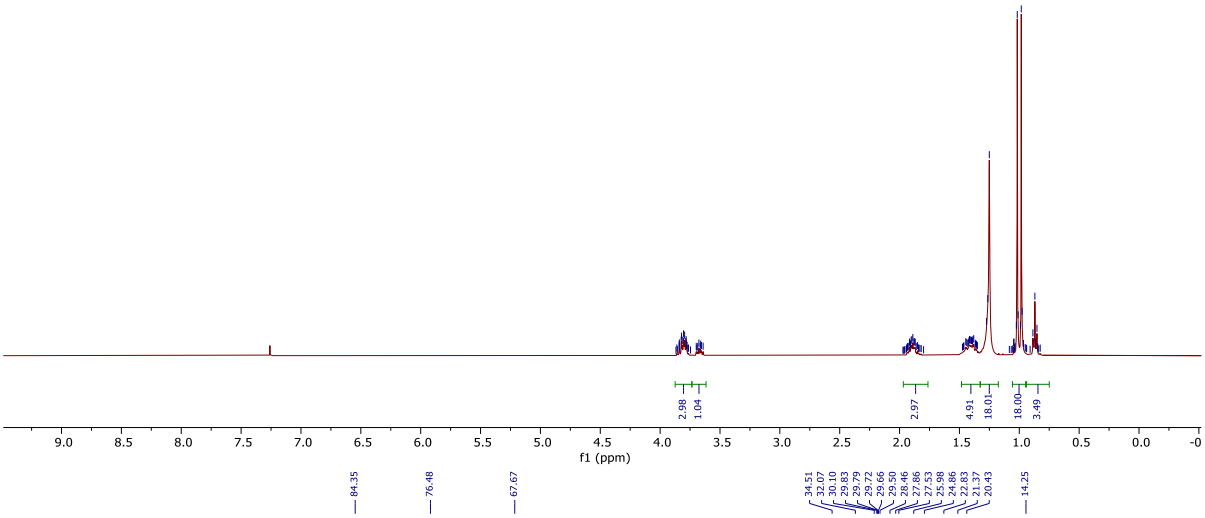
Compound 45 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

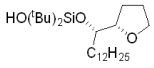


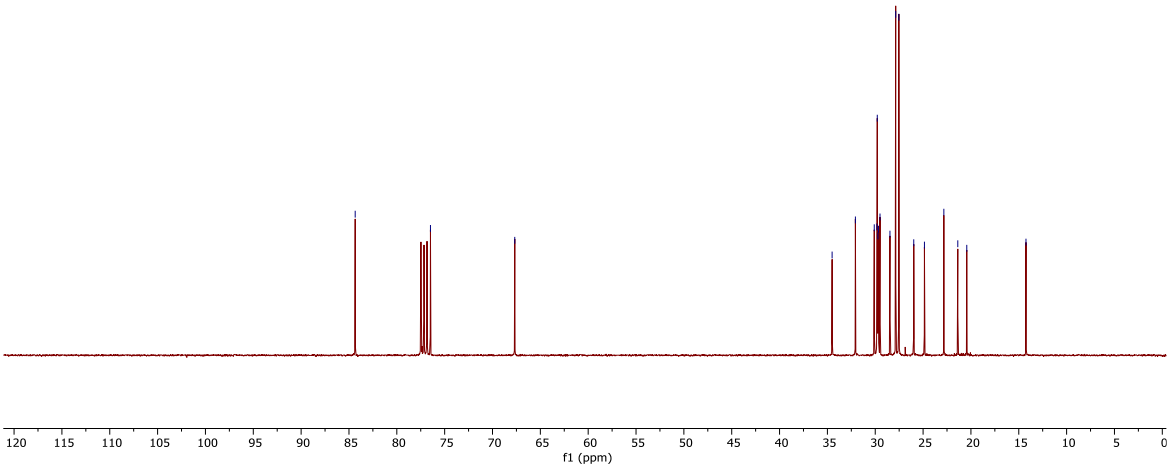


Compound 46 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

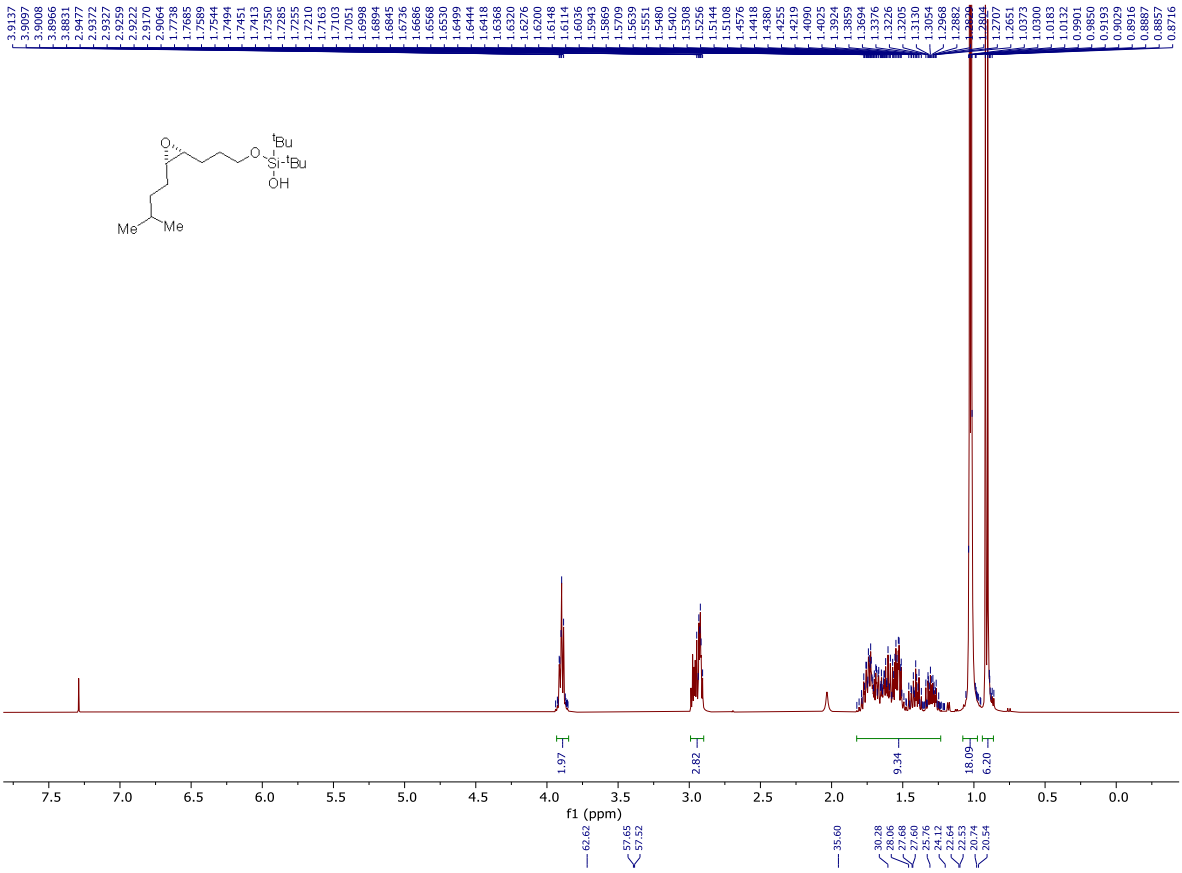


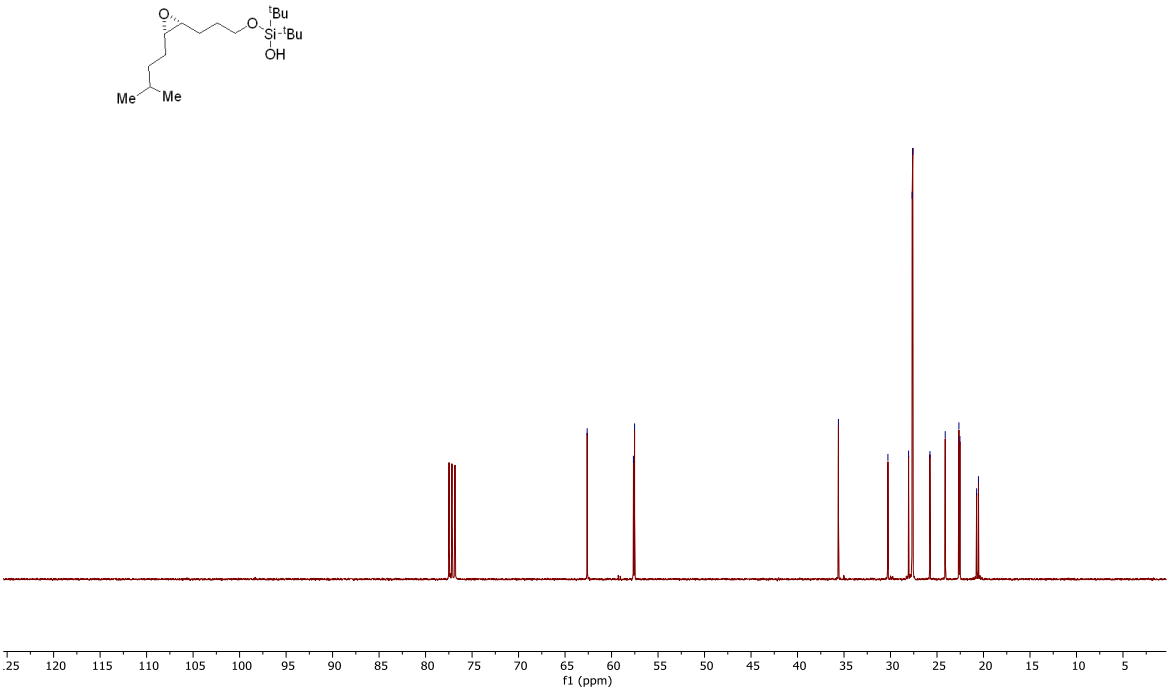




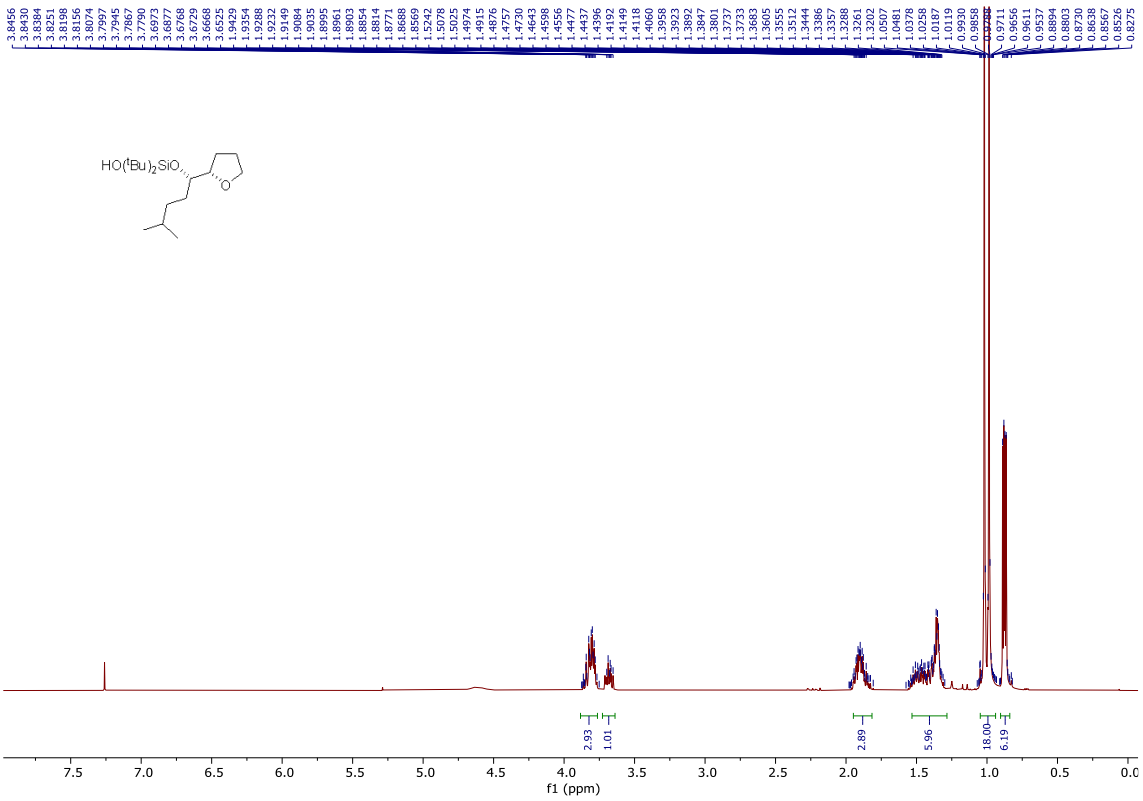


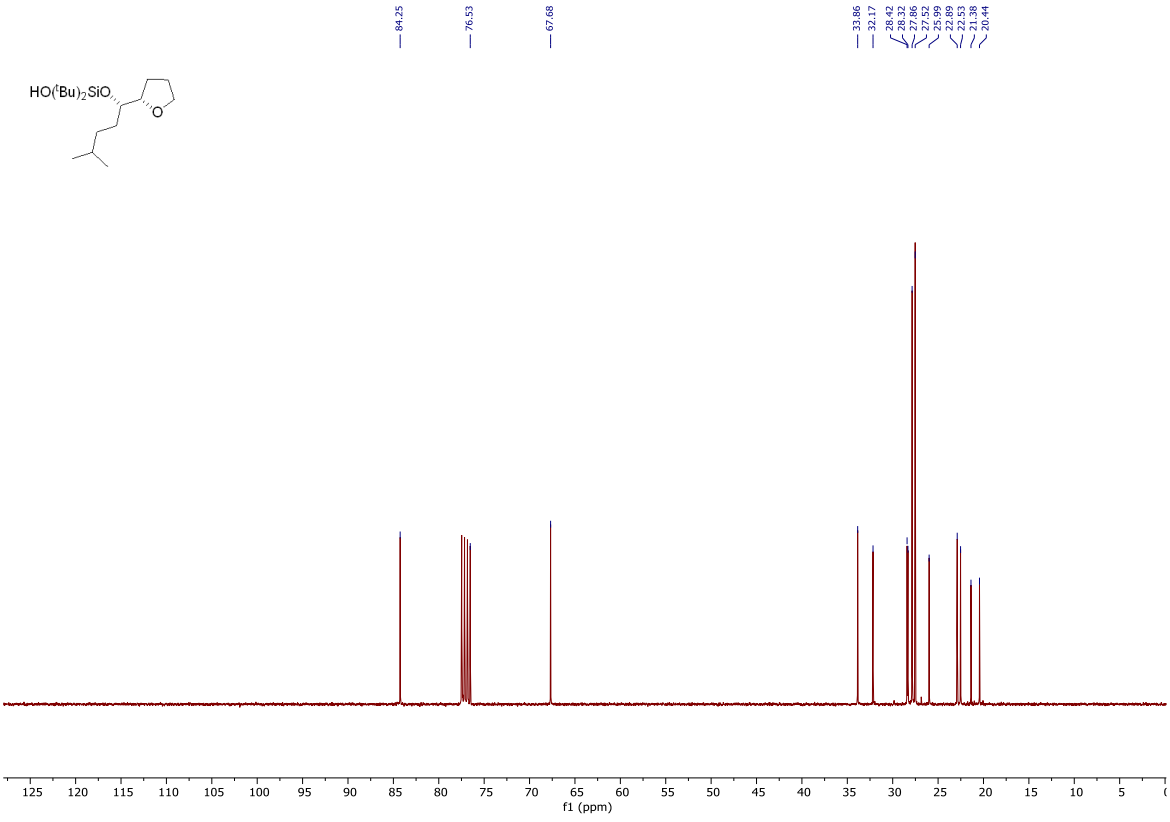
Compound 47 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



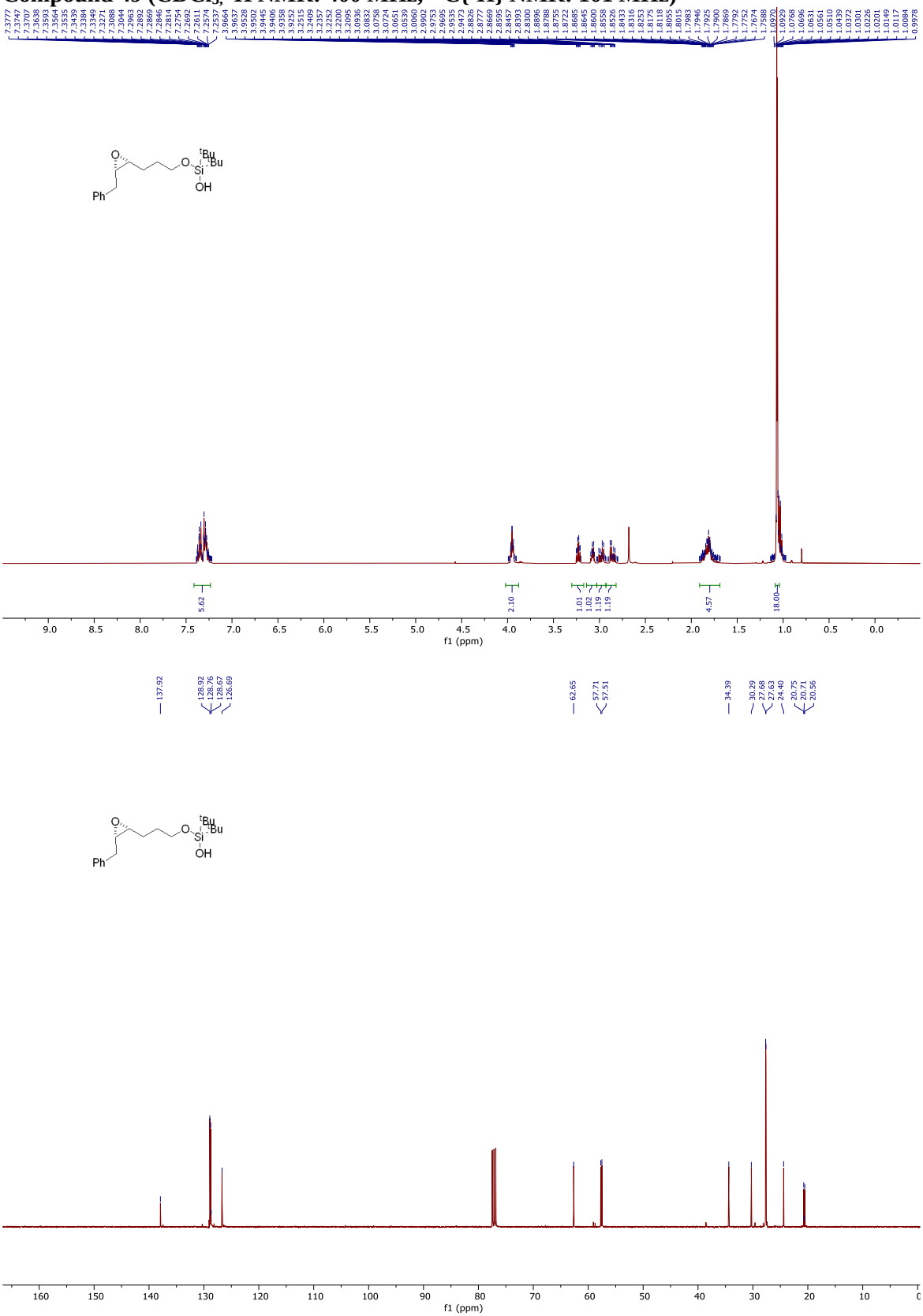


Compound 48 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

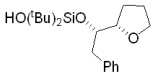


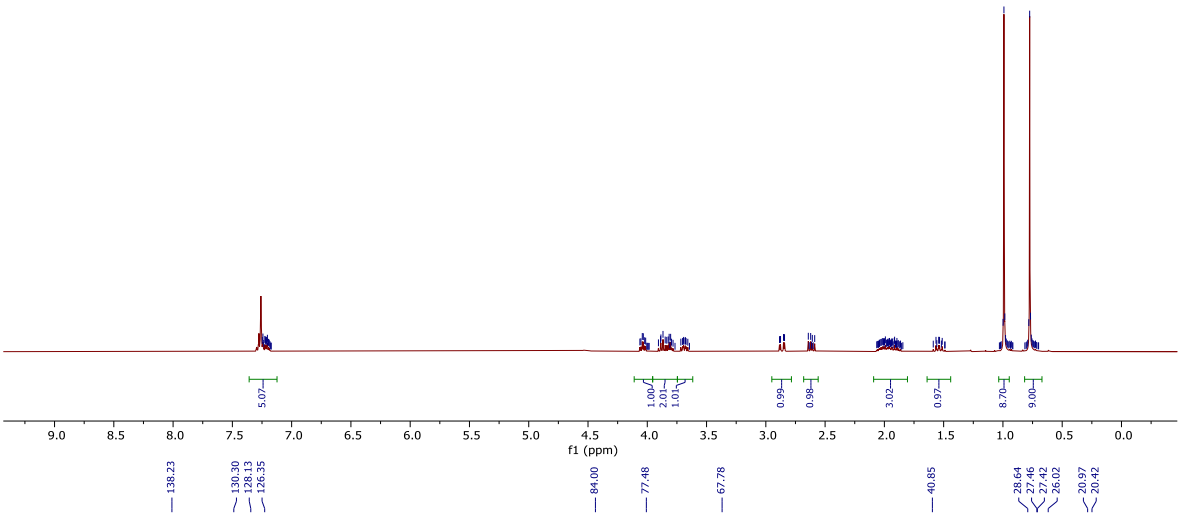


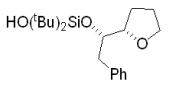
Compound 49 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

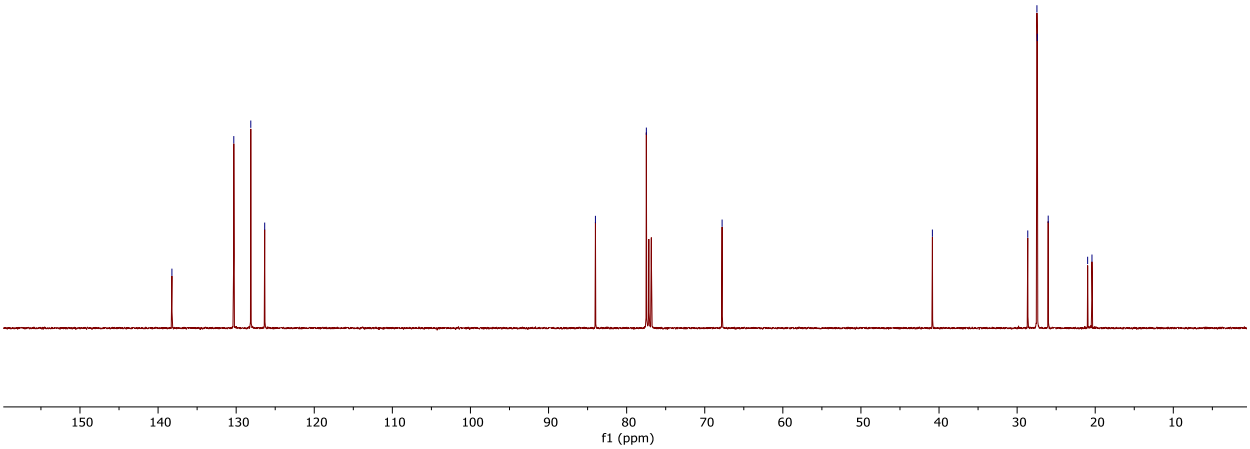


Compound 50 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

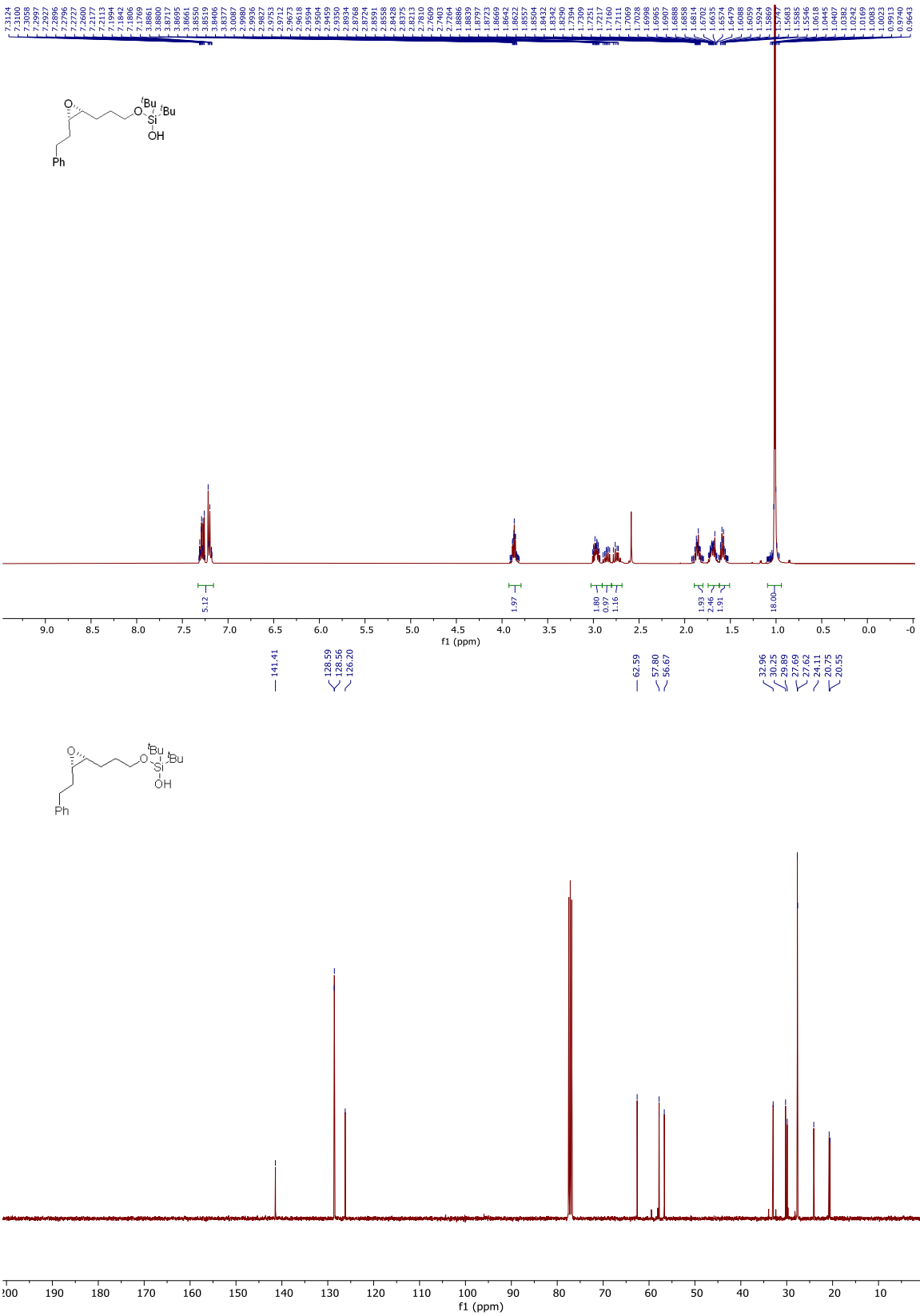




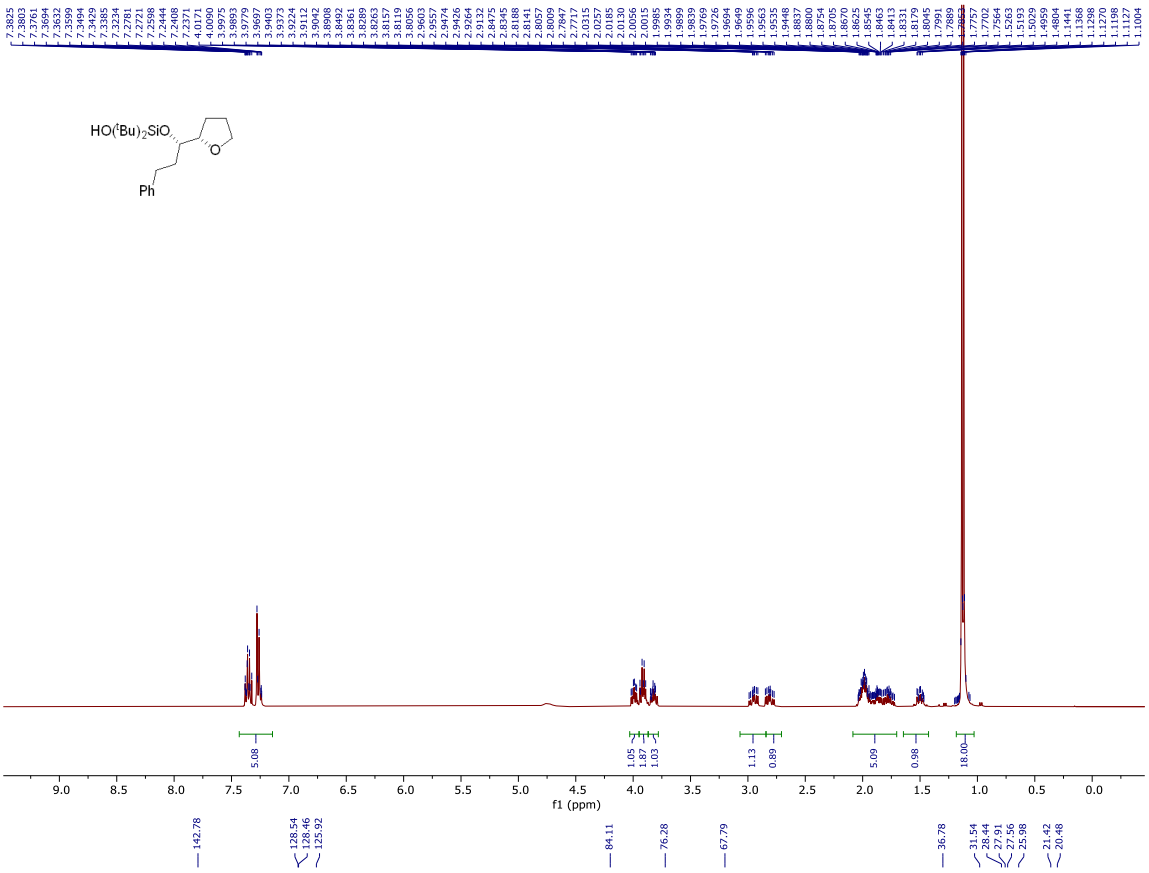


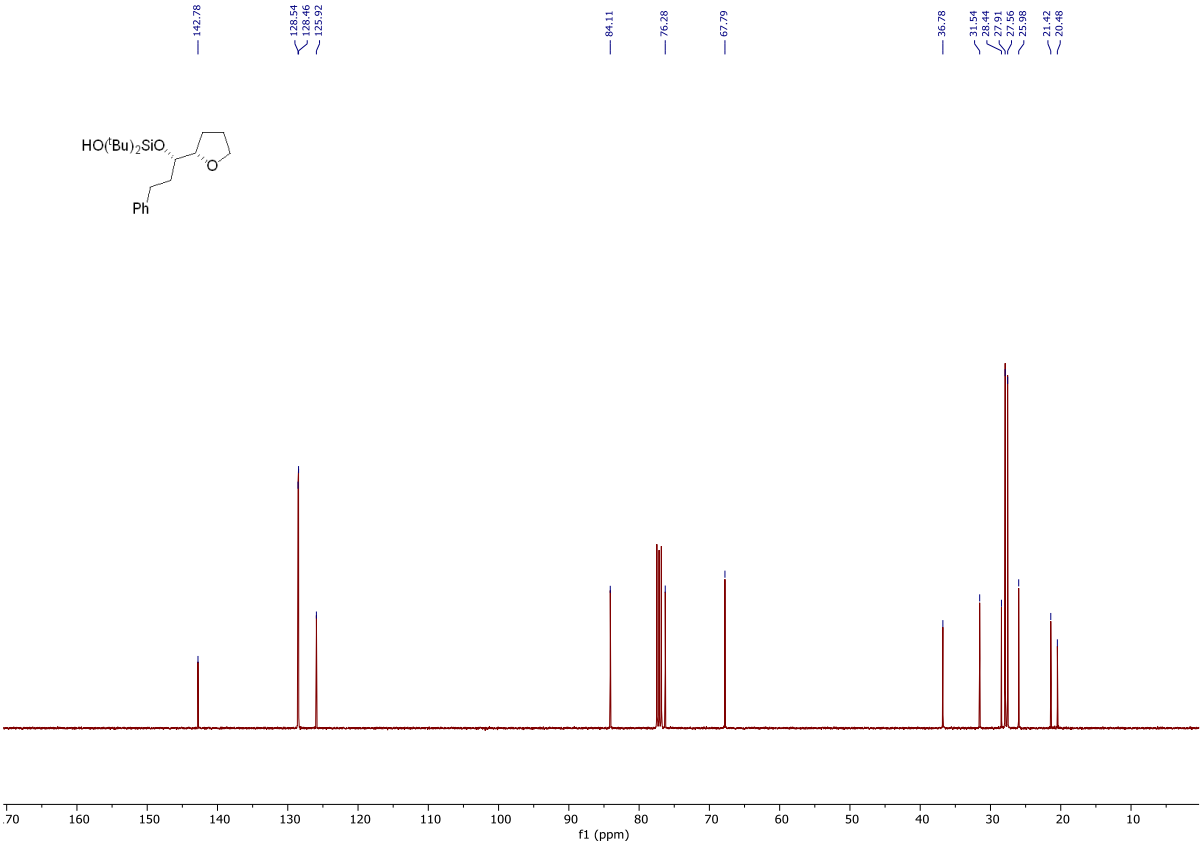


Compound 51 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

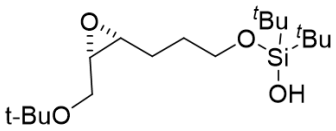


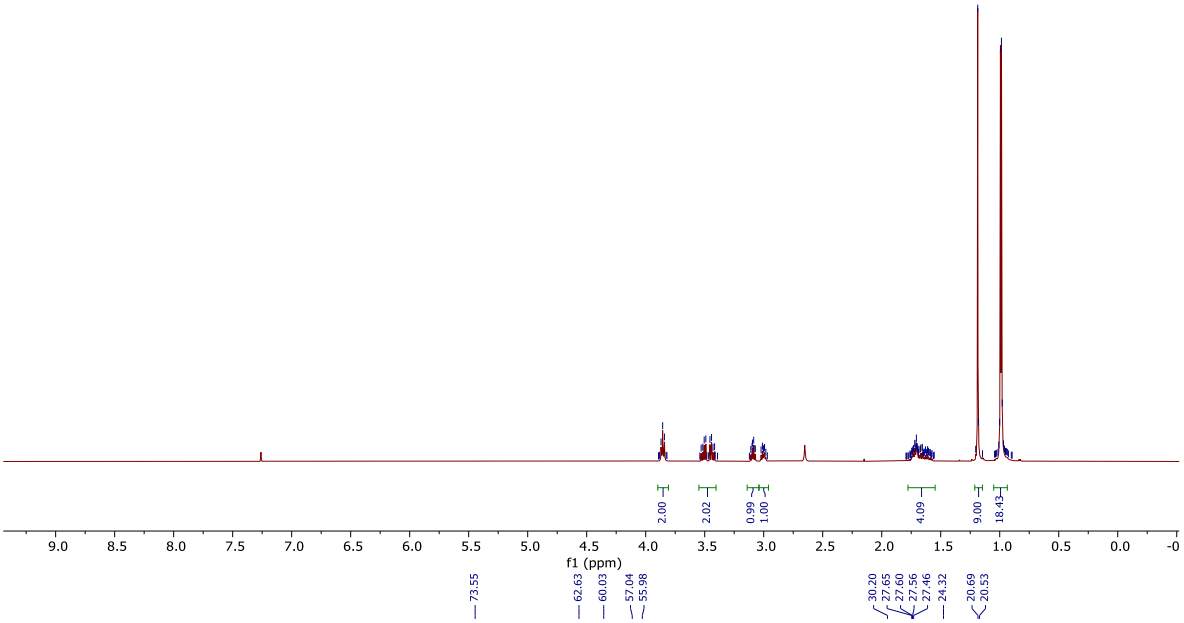
Compound 52 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

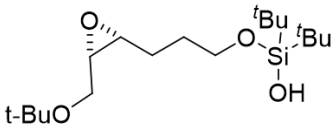




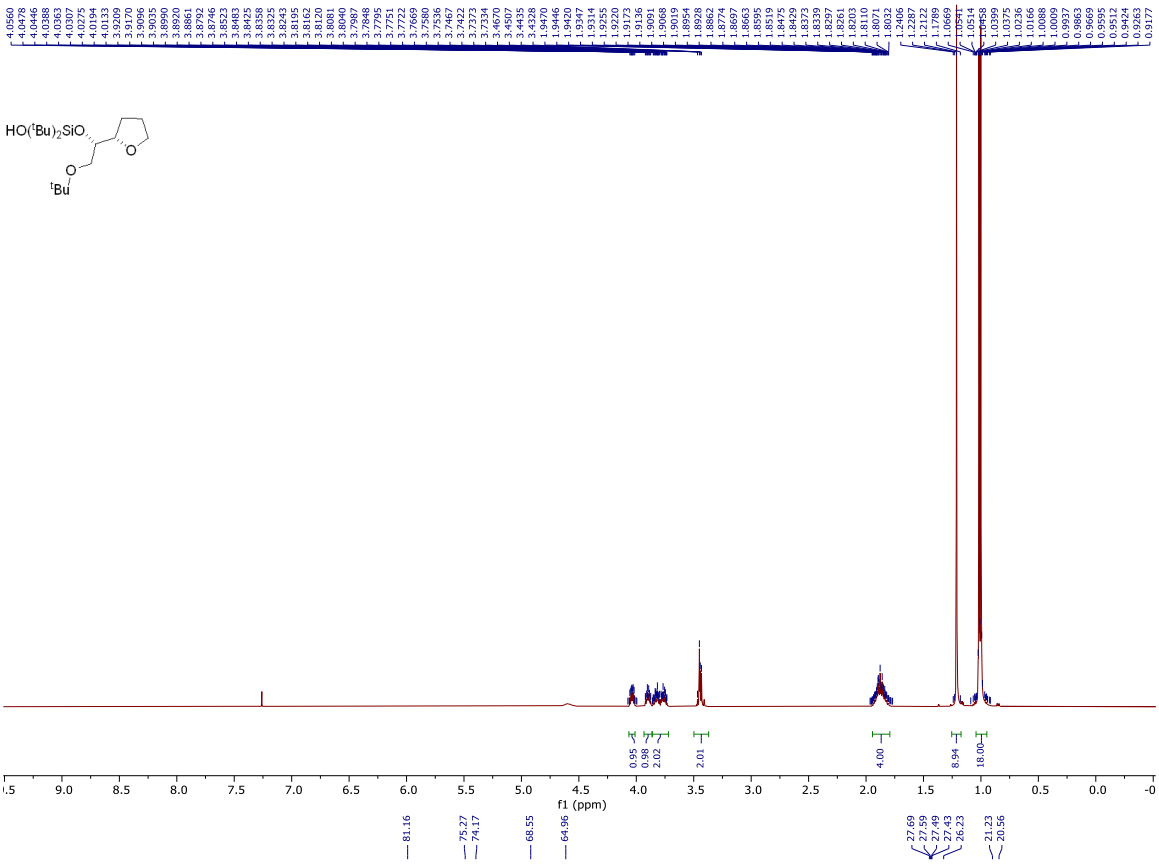
Compound 53 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

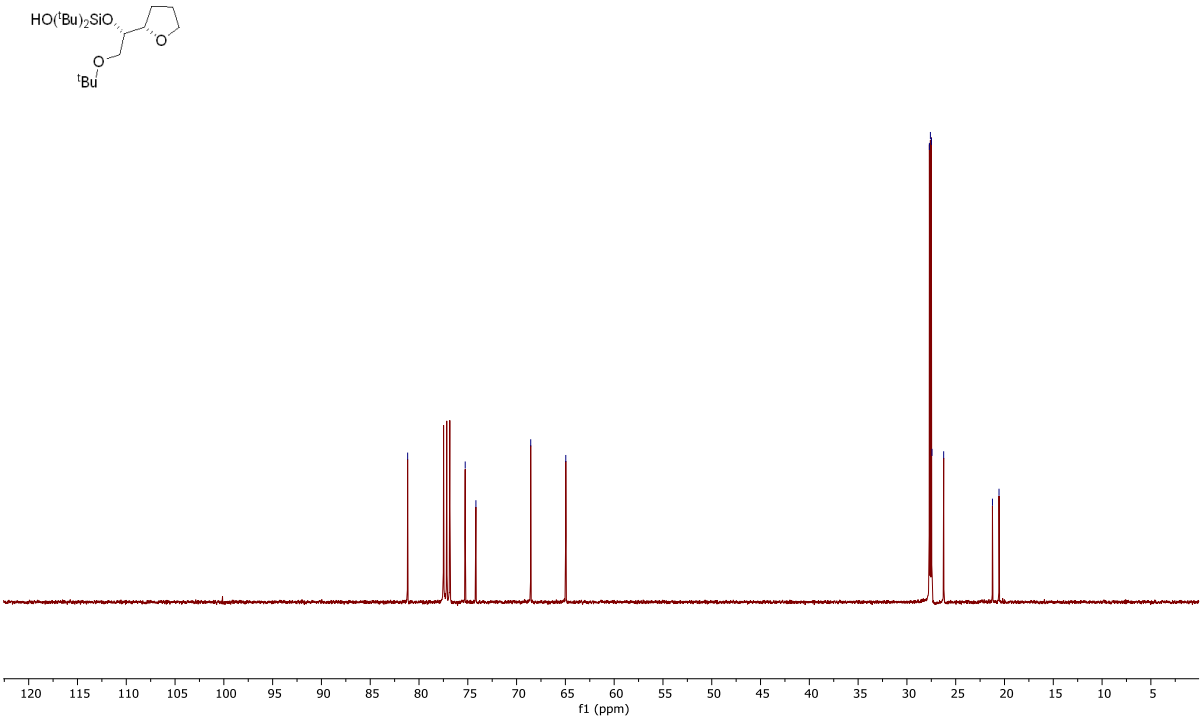




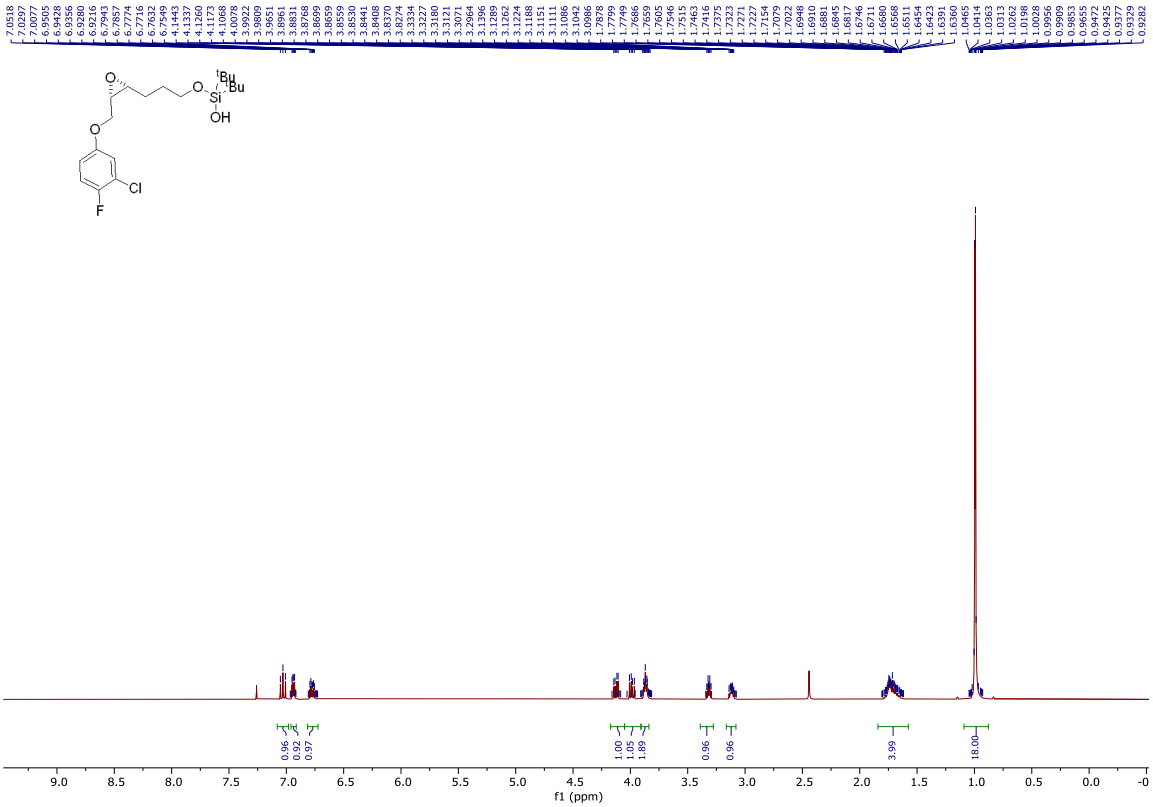


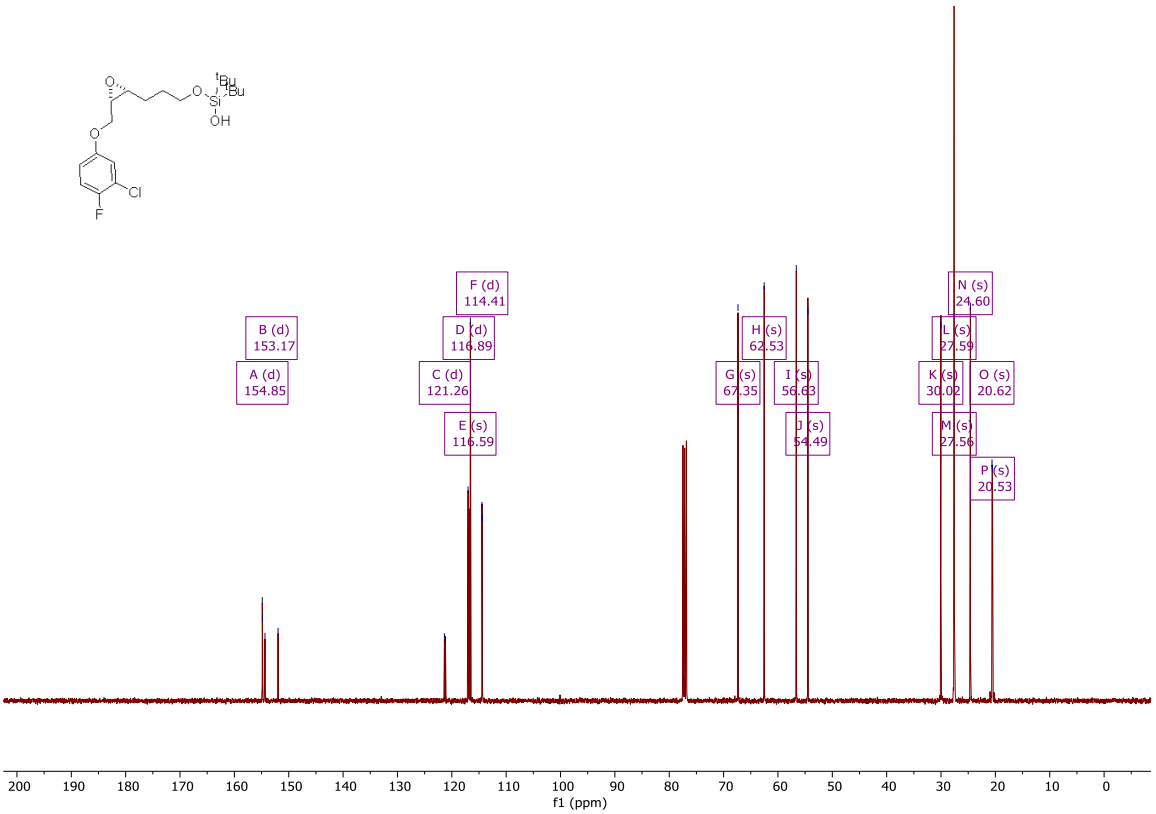
Compound 54 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



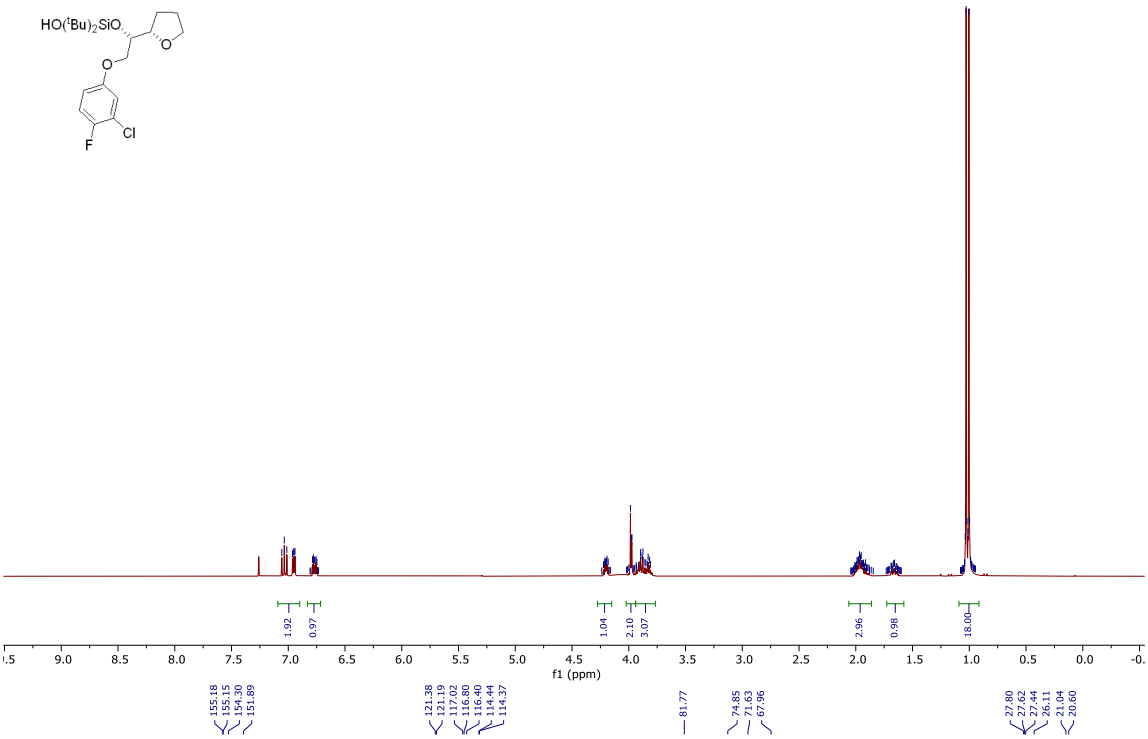


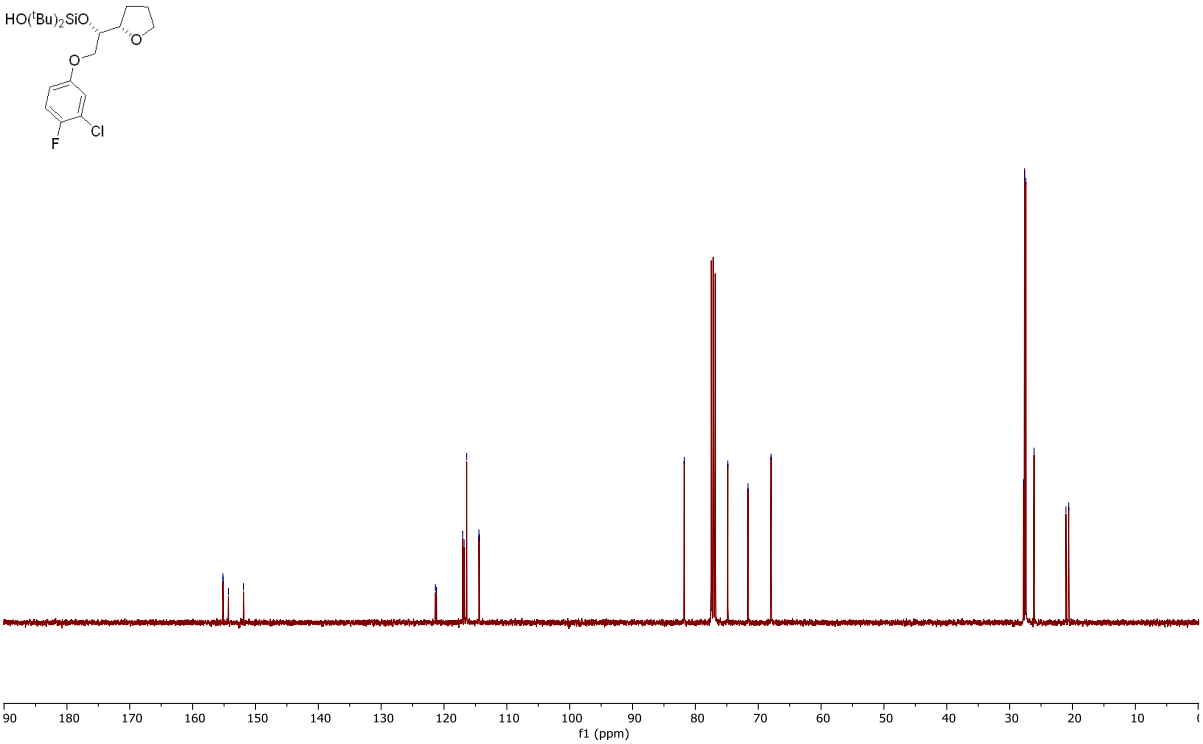
Compound 55 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



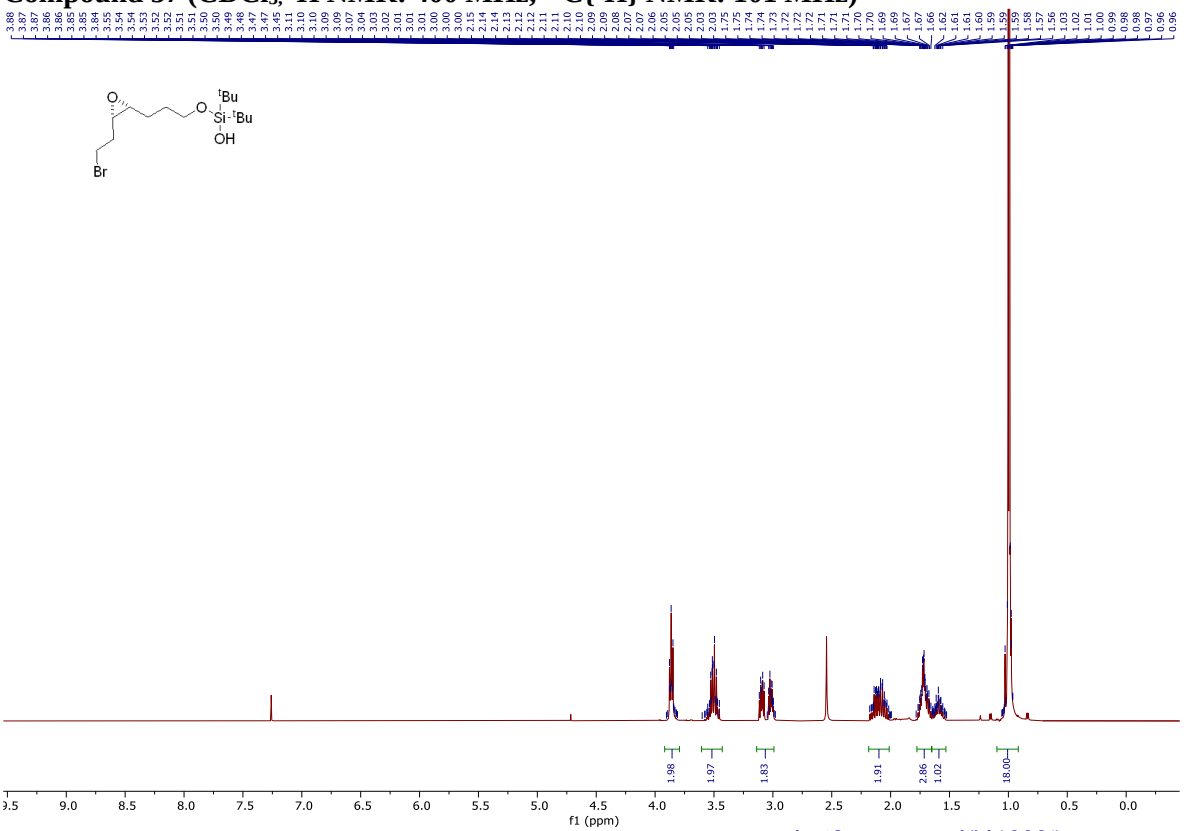


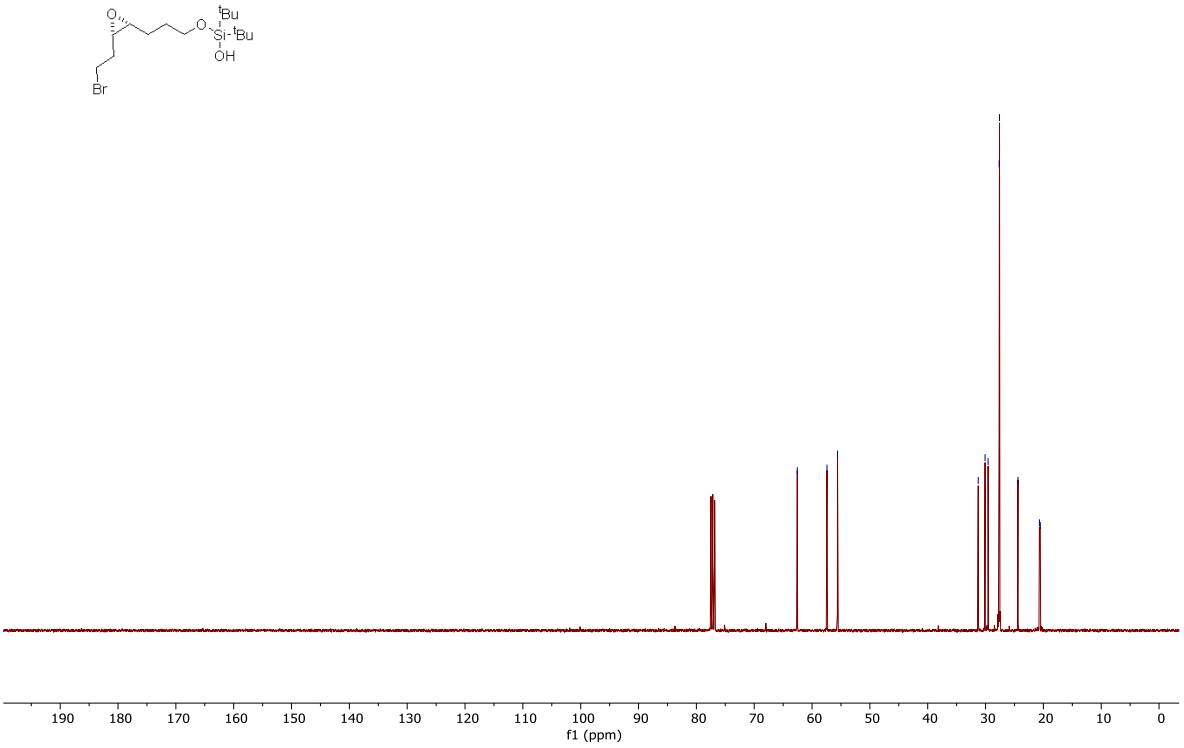
Compound 56 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



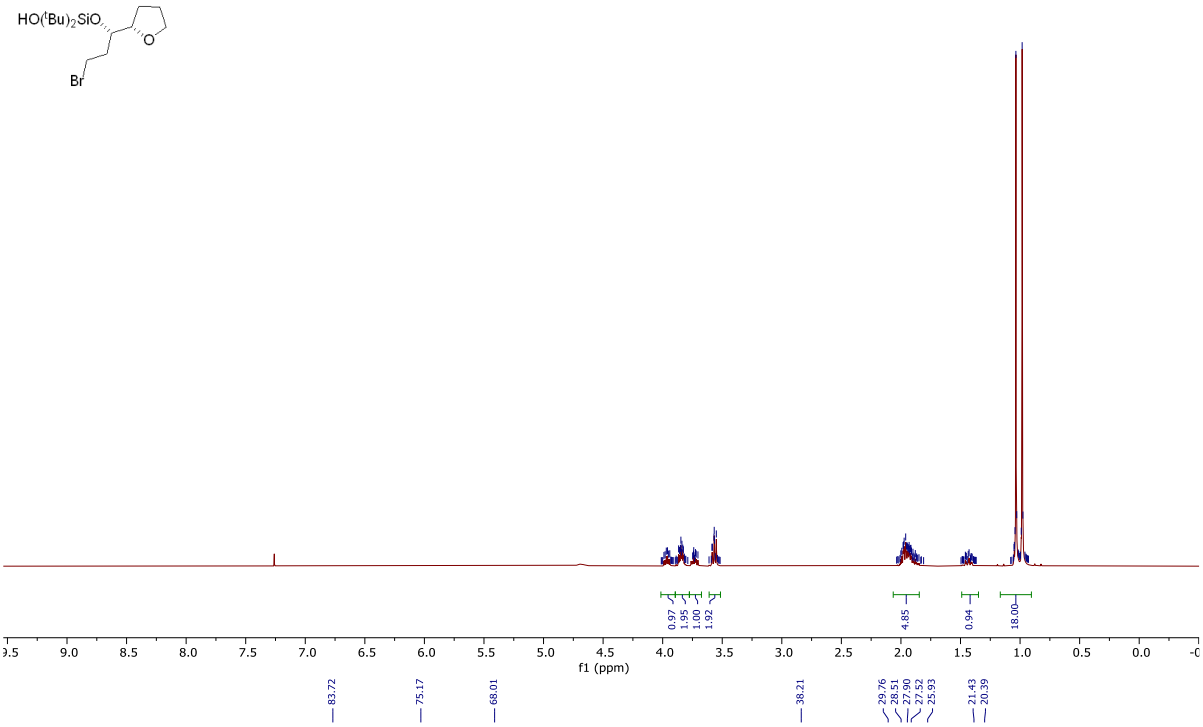


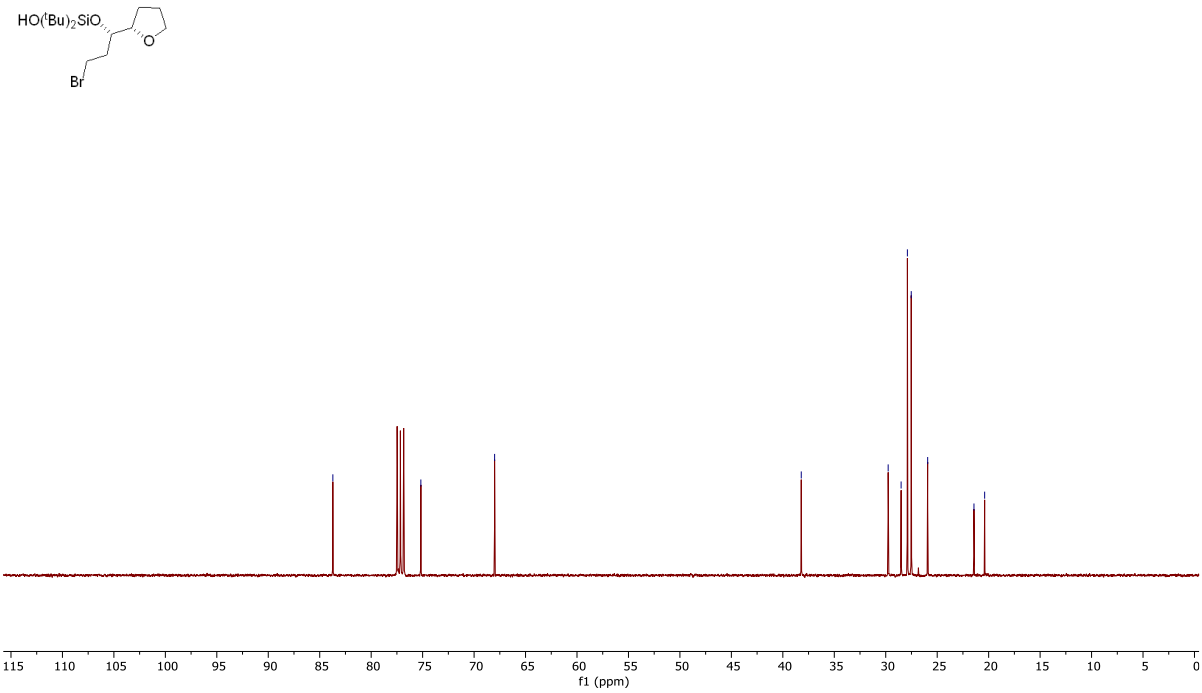
Compound 57 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



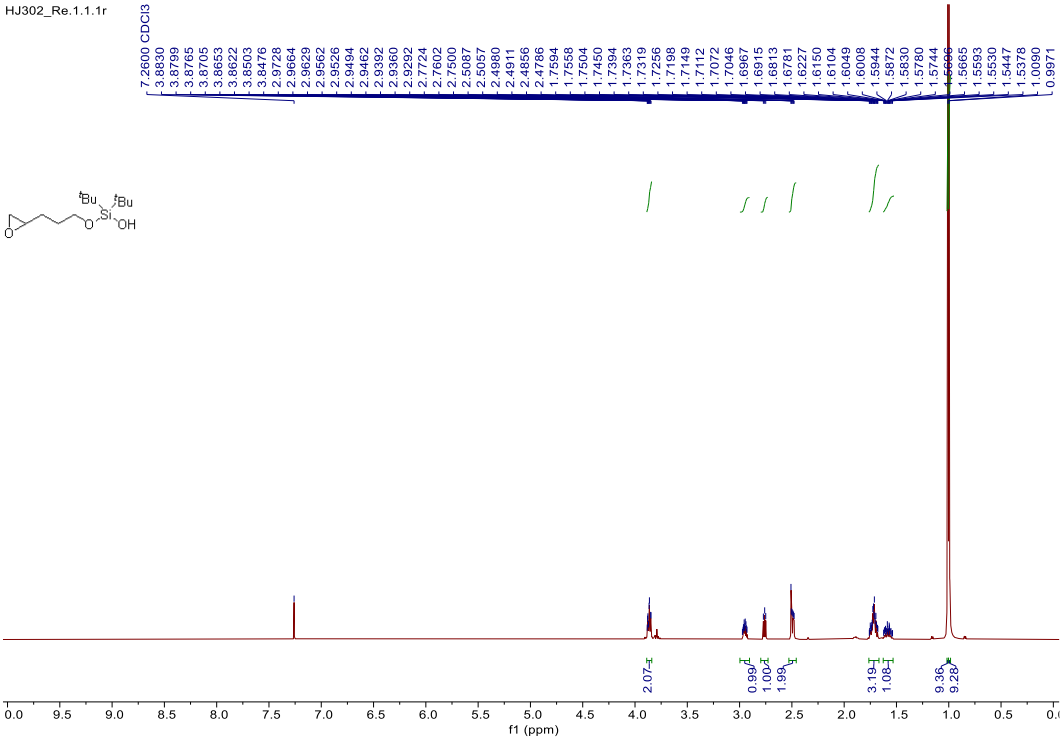


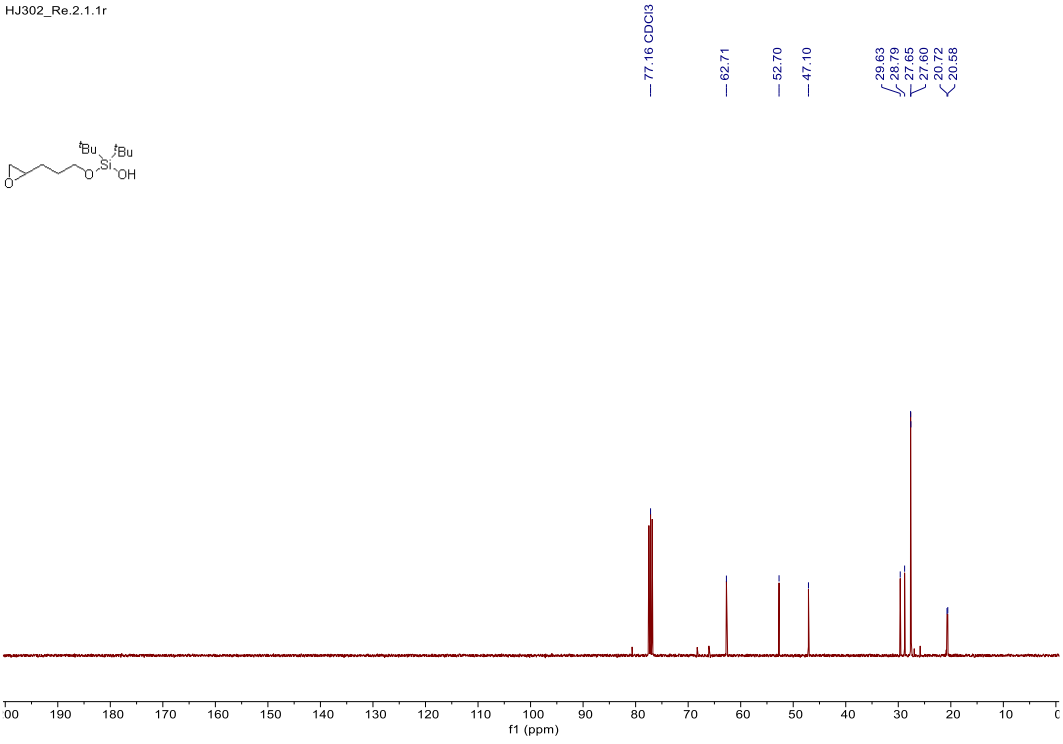
Compound 58 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



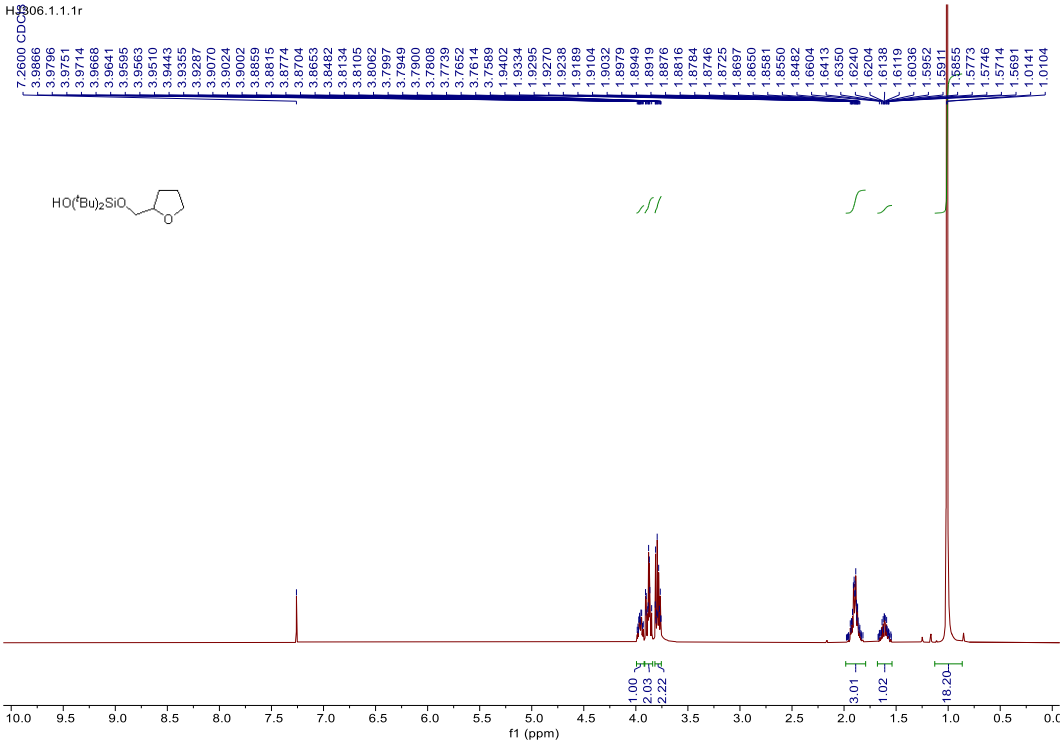


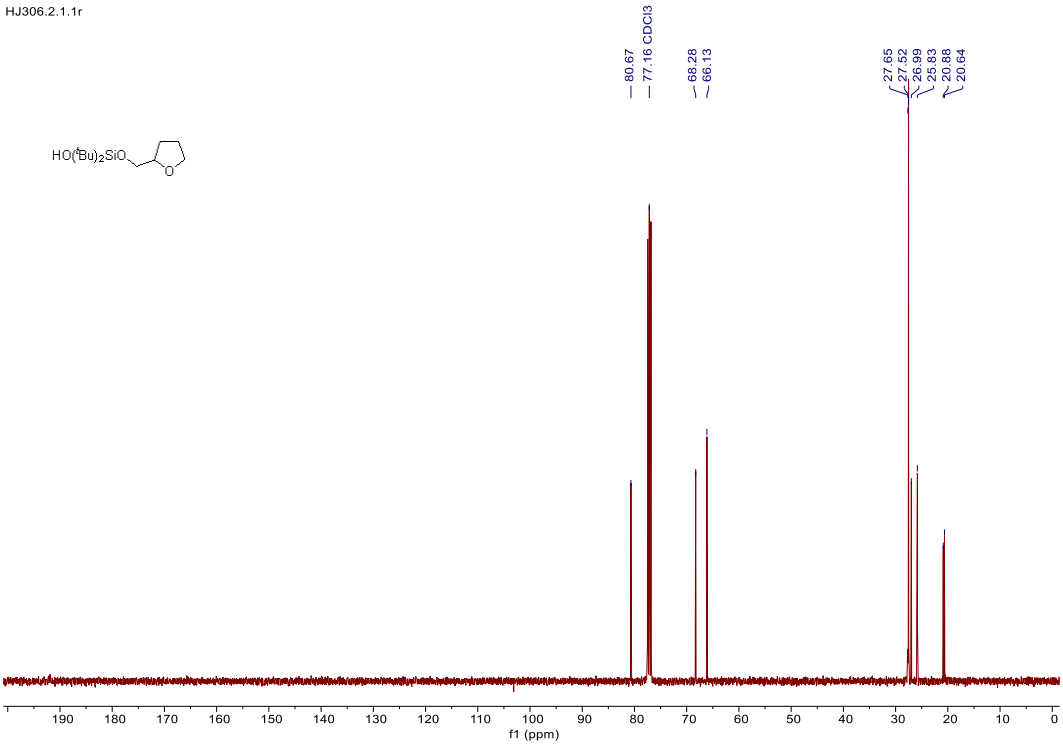
Compound 59 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



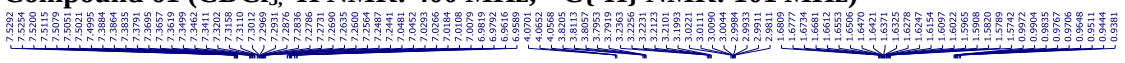


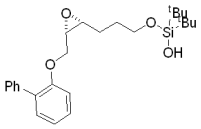
Compound 60 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

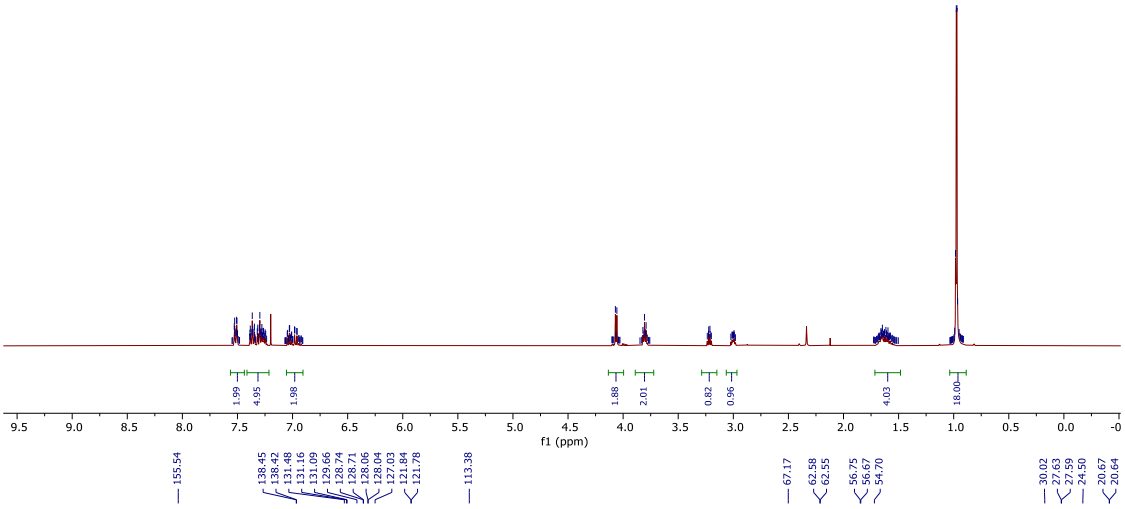


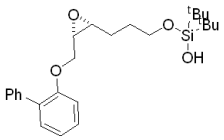


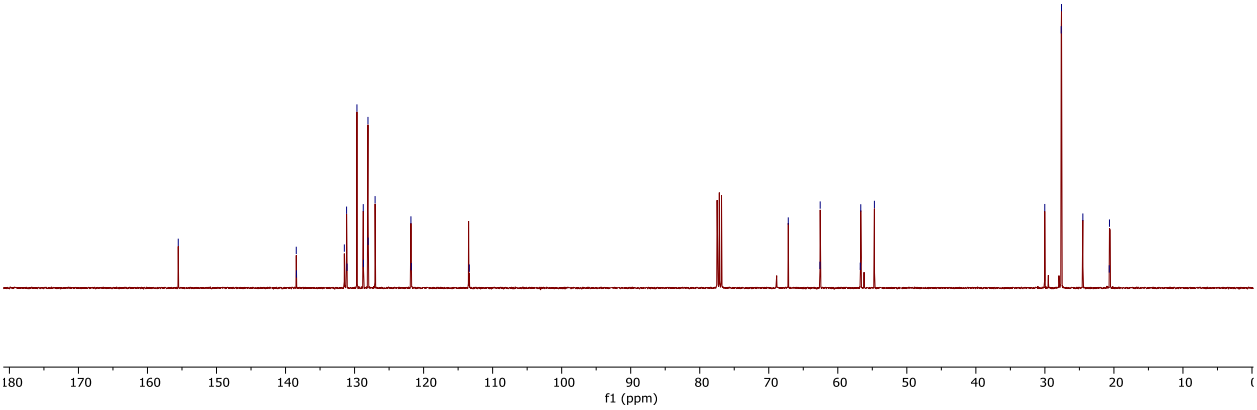
Compound 61 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



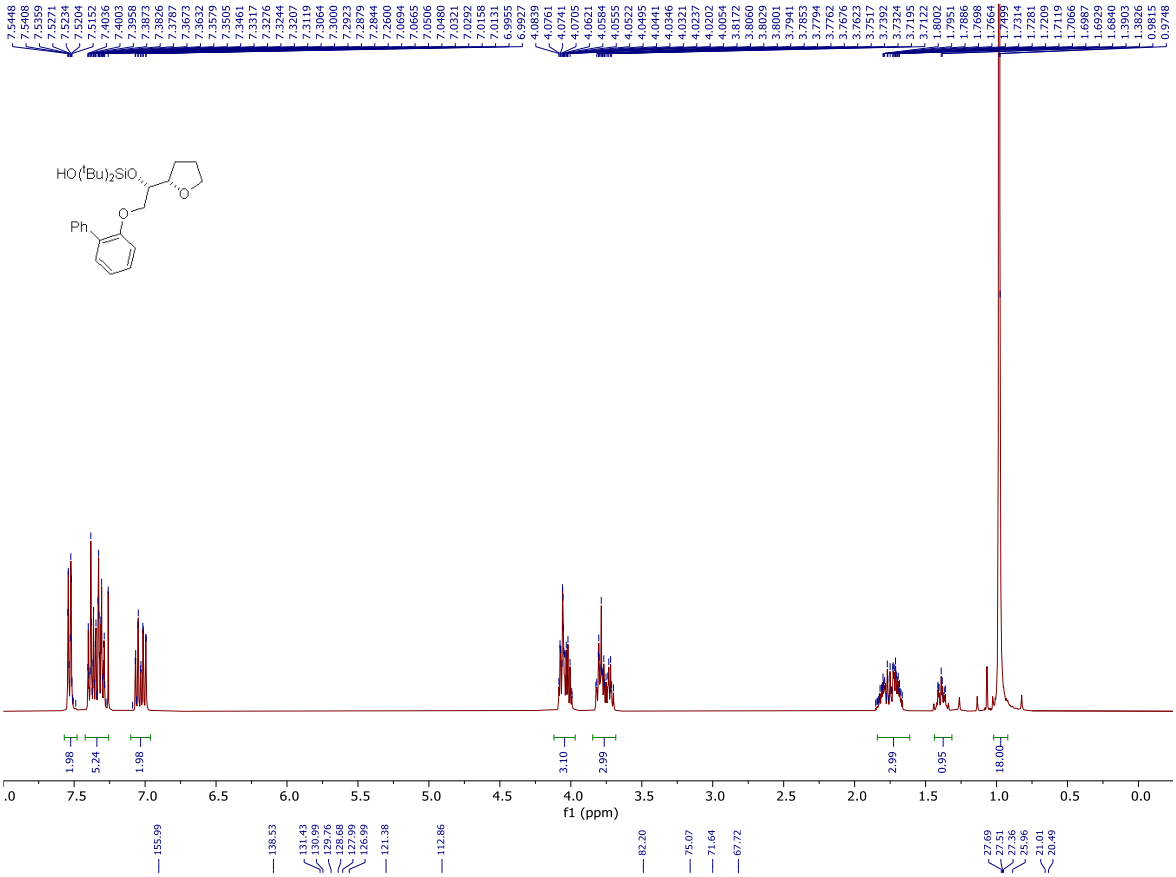


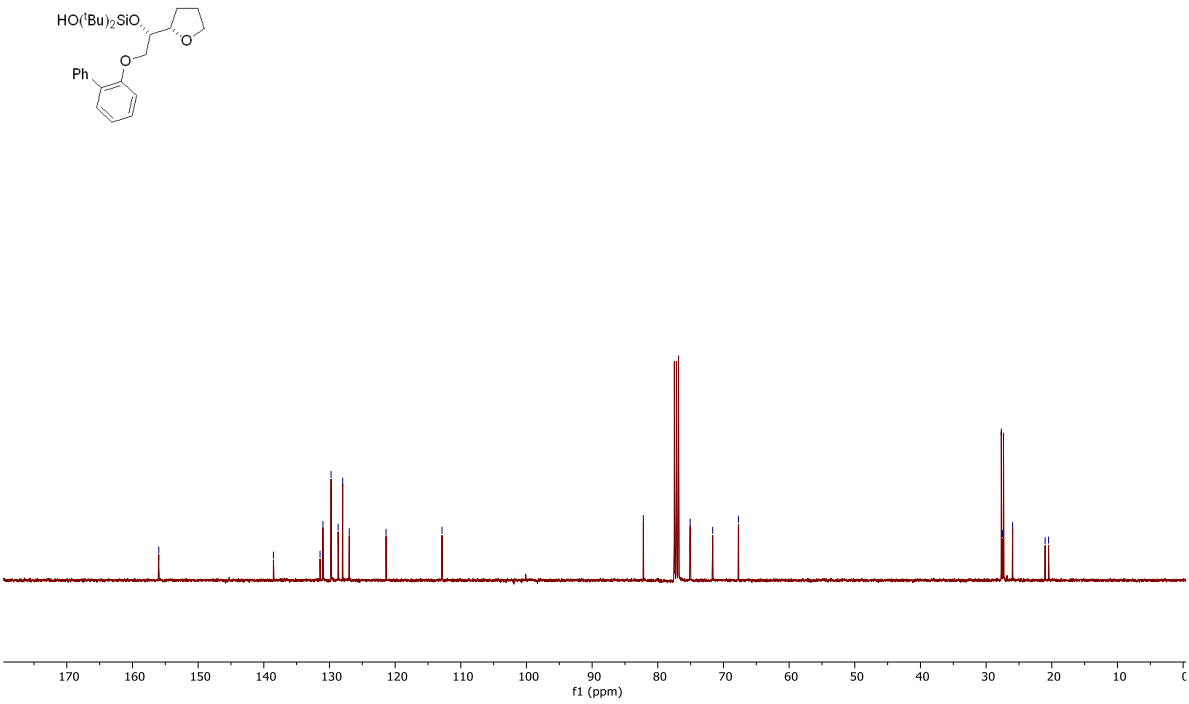




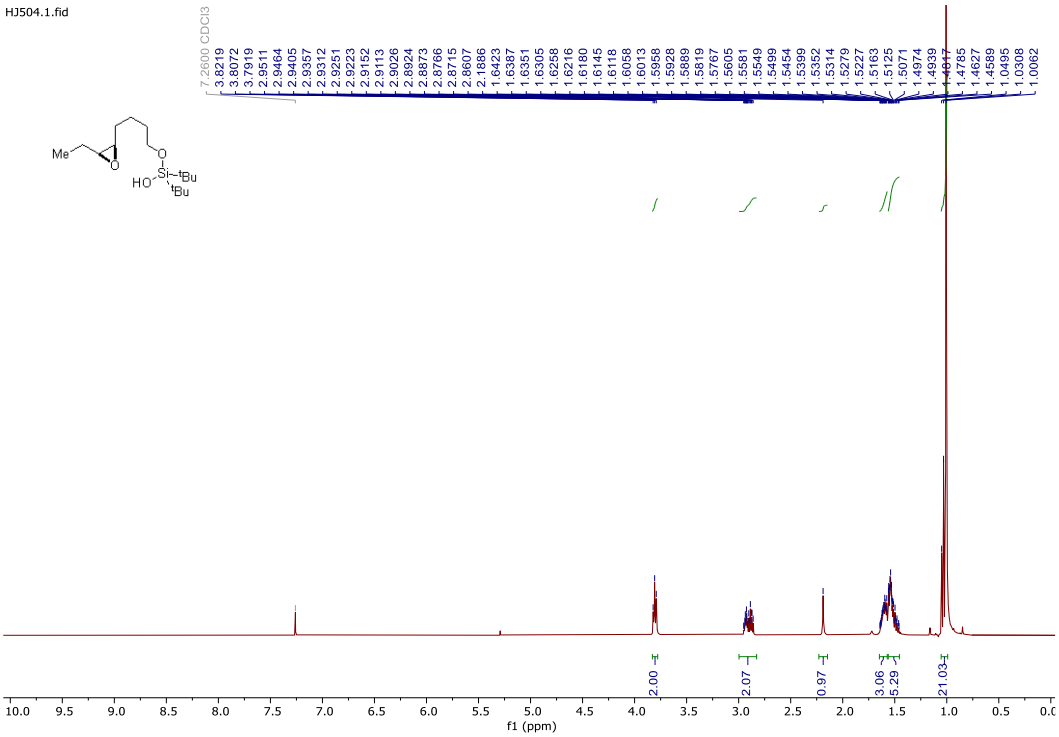


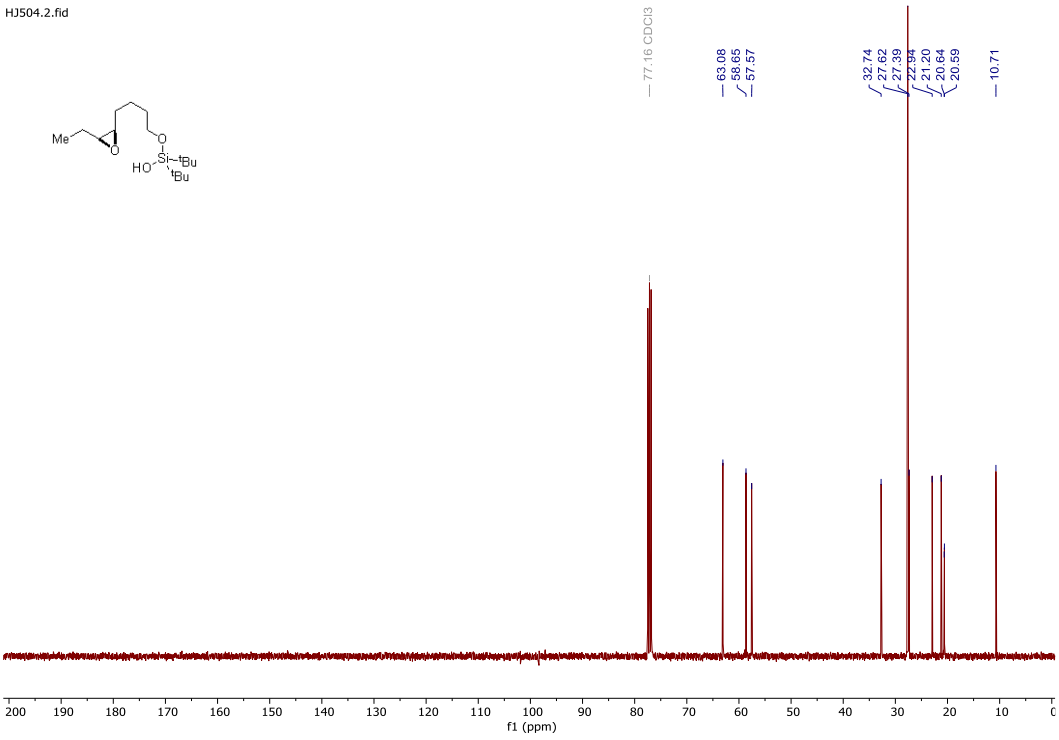
Compound 62 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



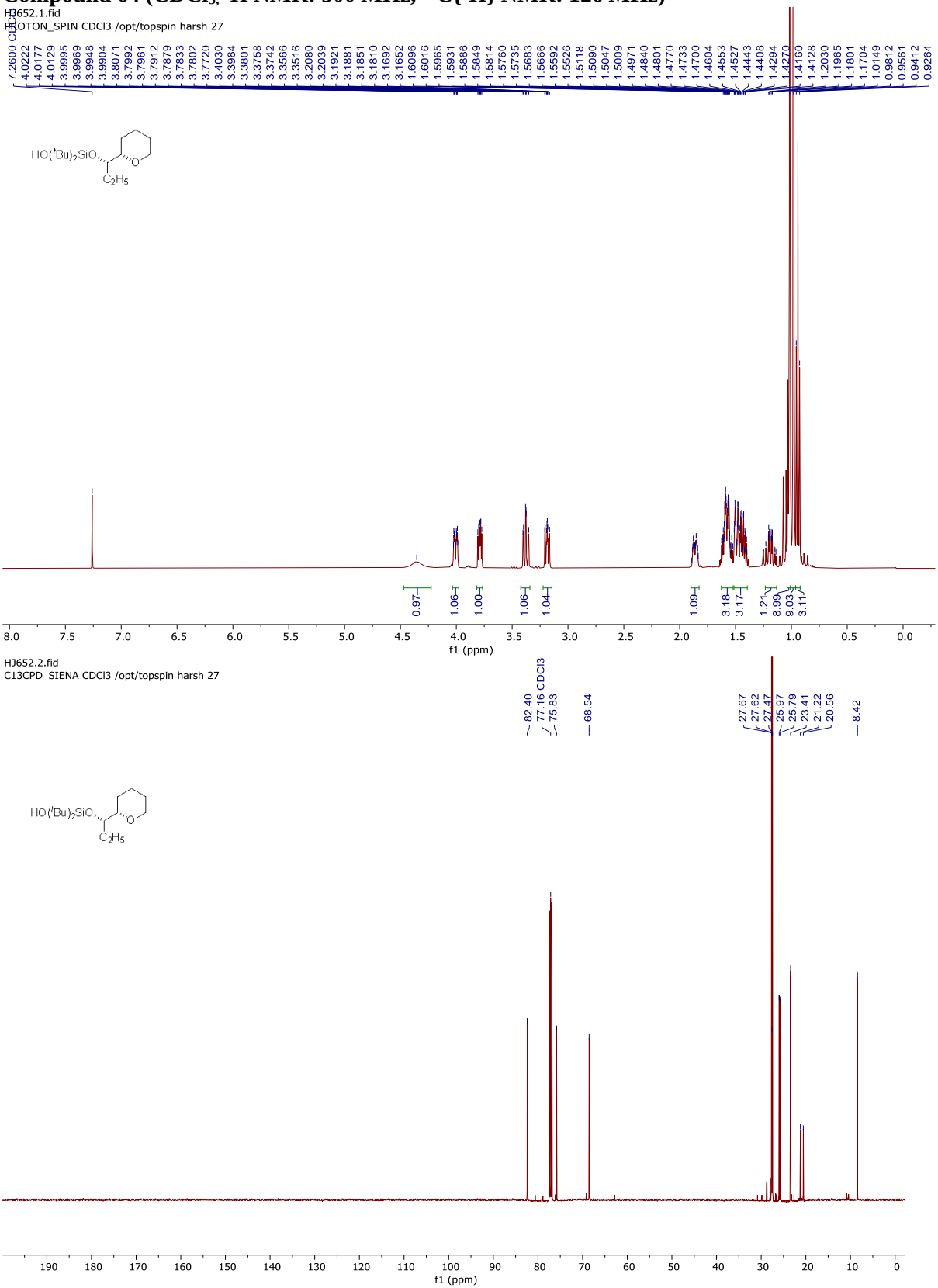


Compound 63 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

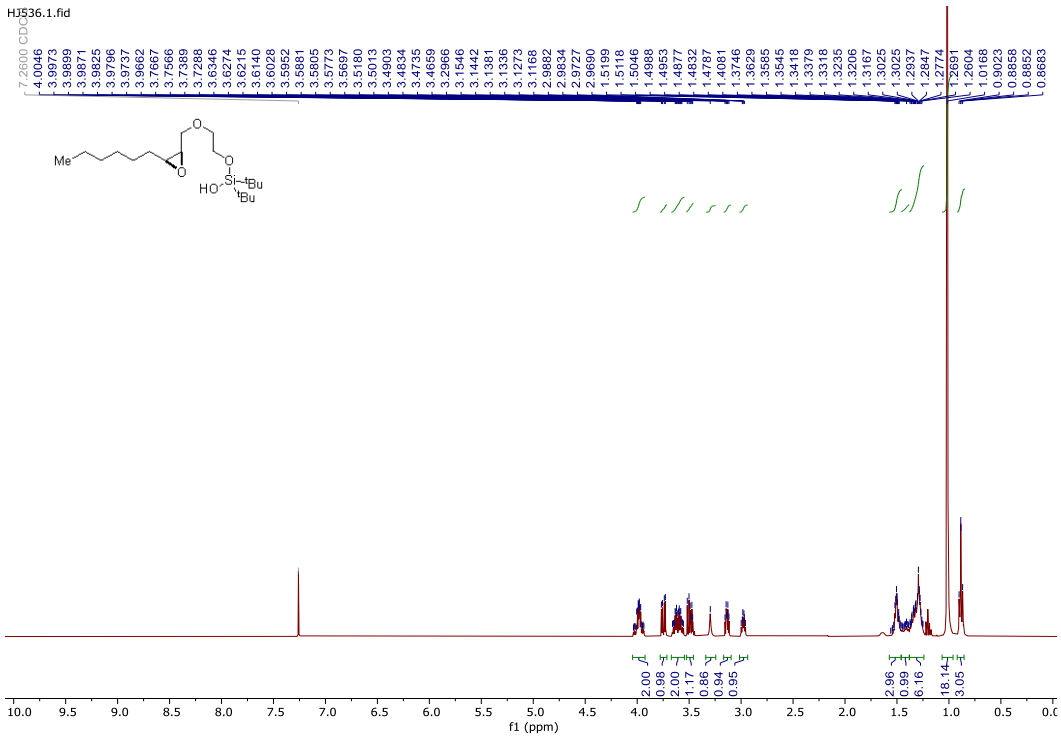


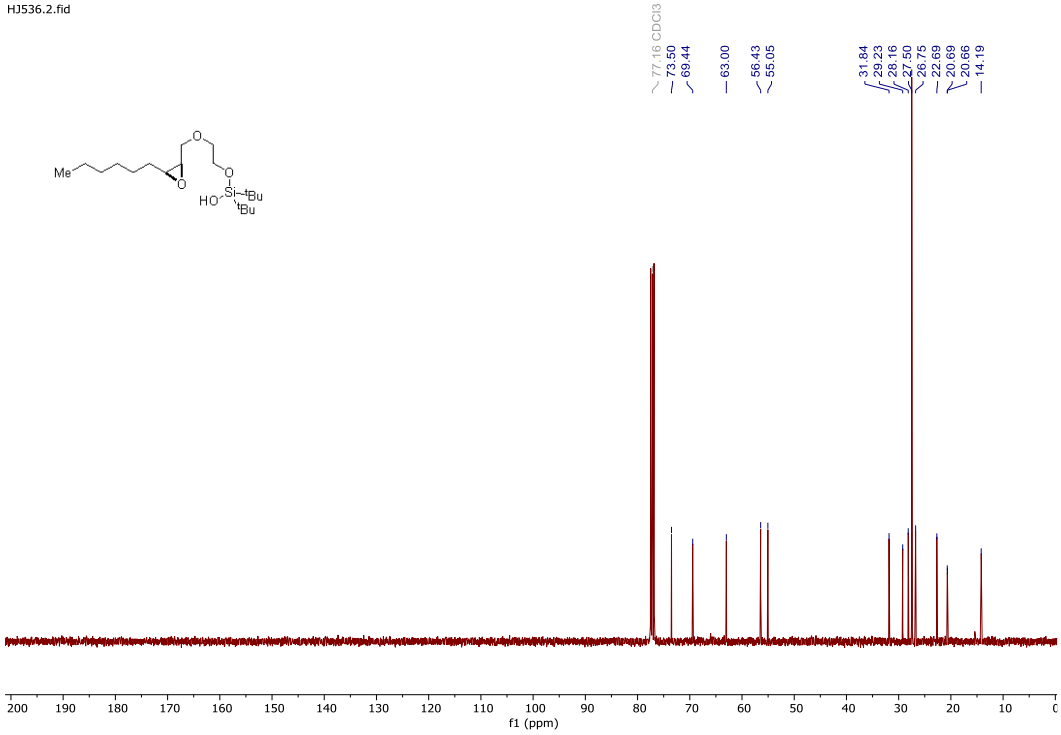


Compound 64 ($CDCl_3$, ^1H NMR: 500 MHz, $^{13}C\{^1H\}$ NMR: 126 MHz)

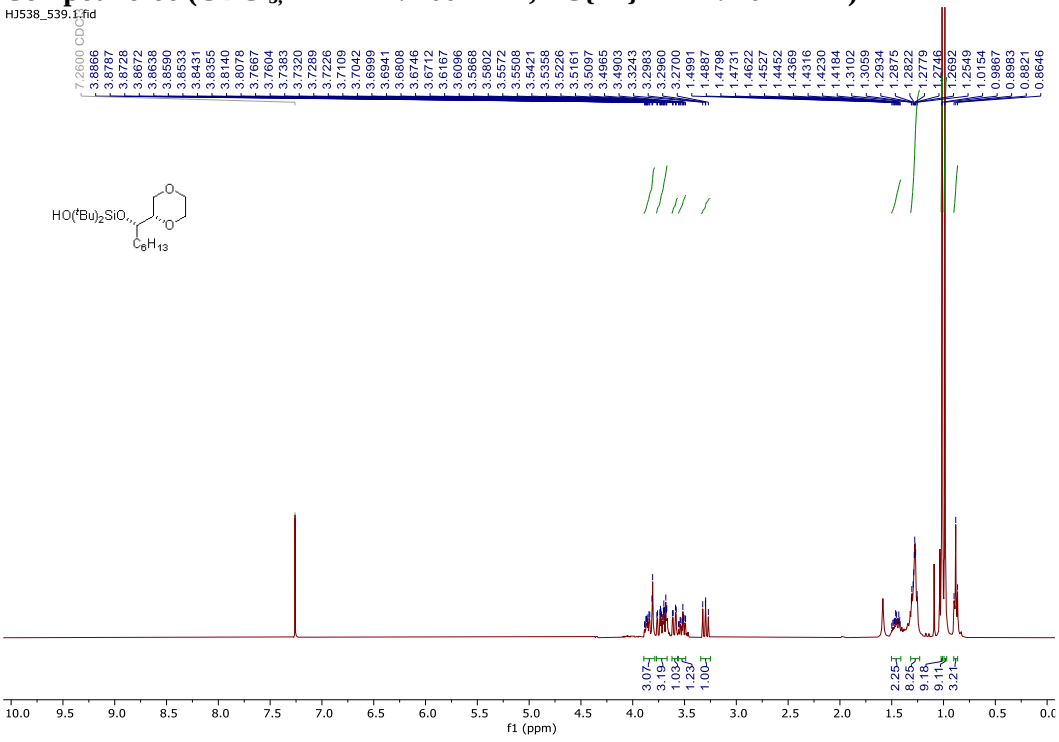


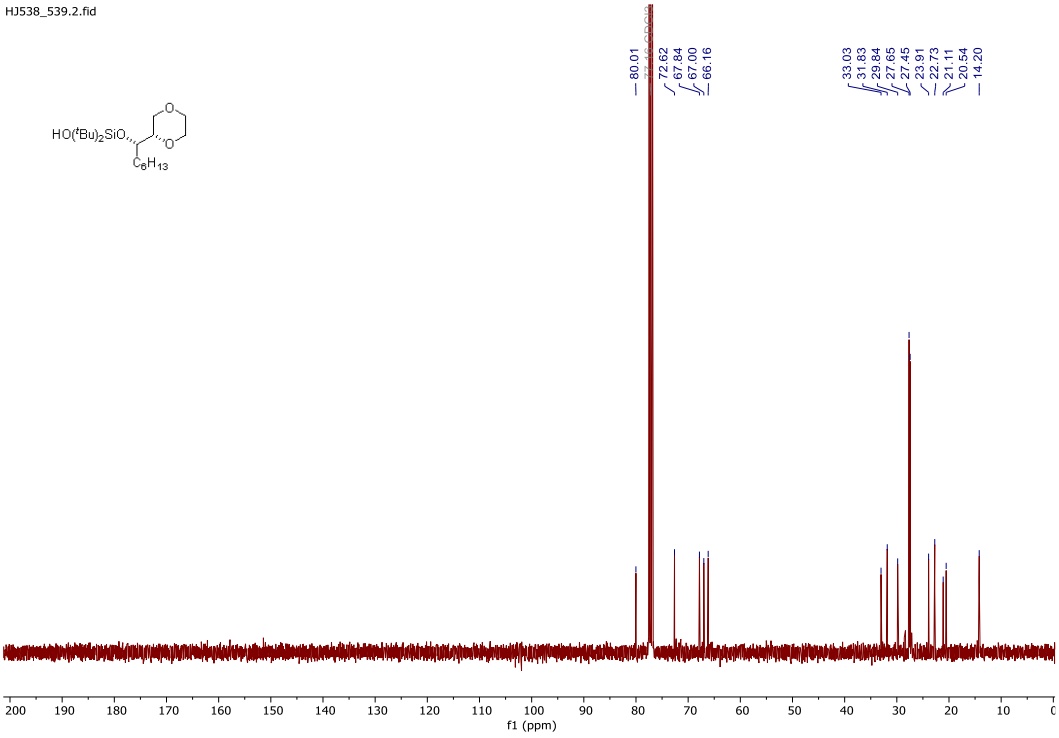
Compound 65 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



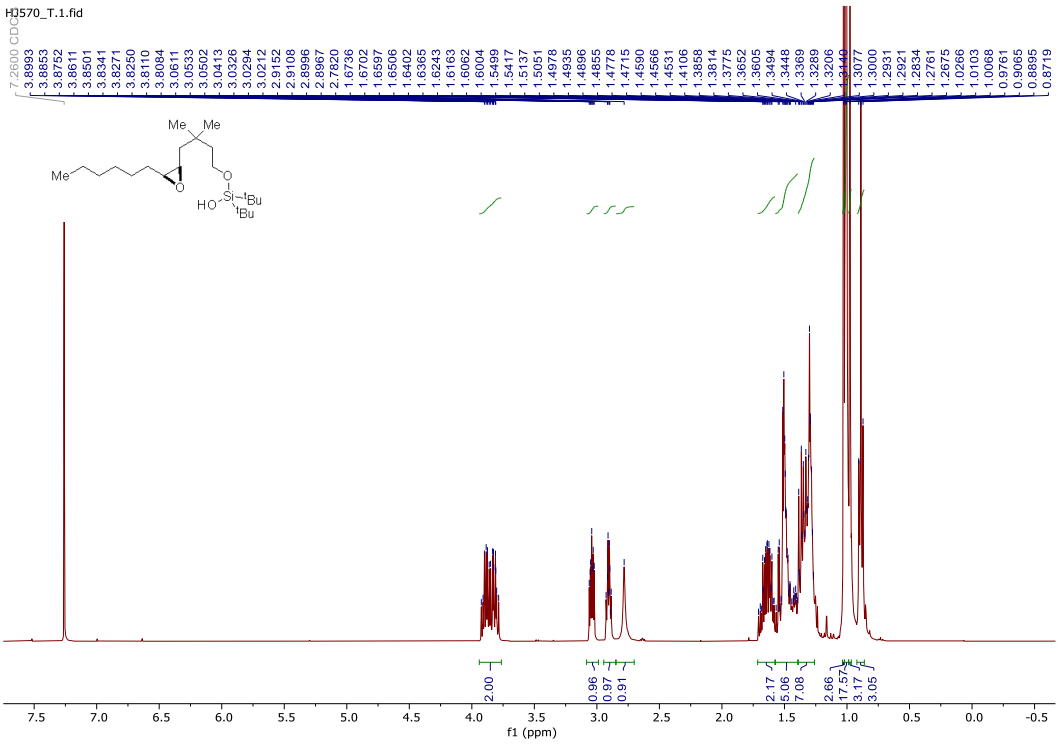


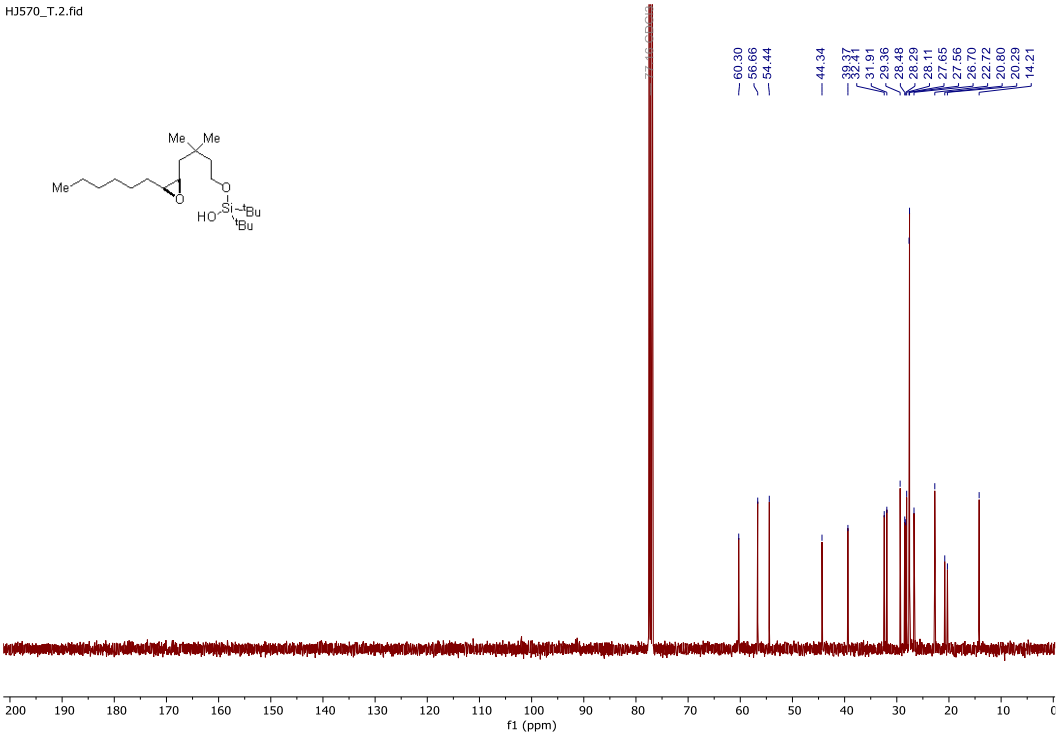
Compound 66 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



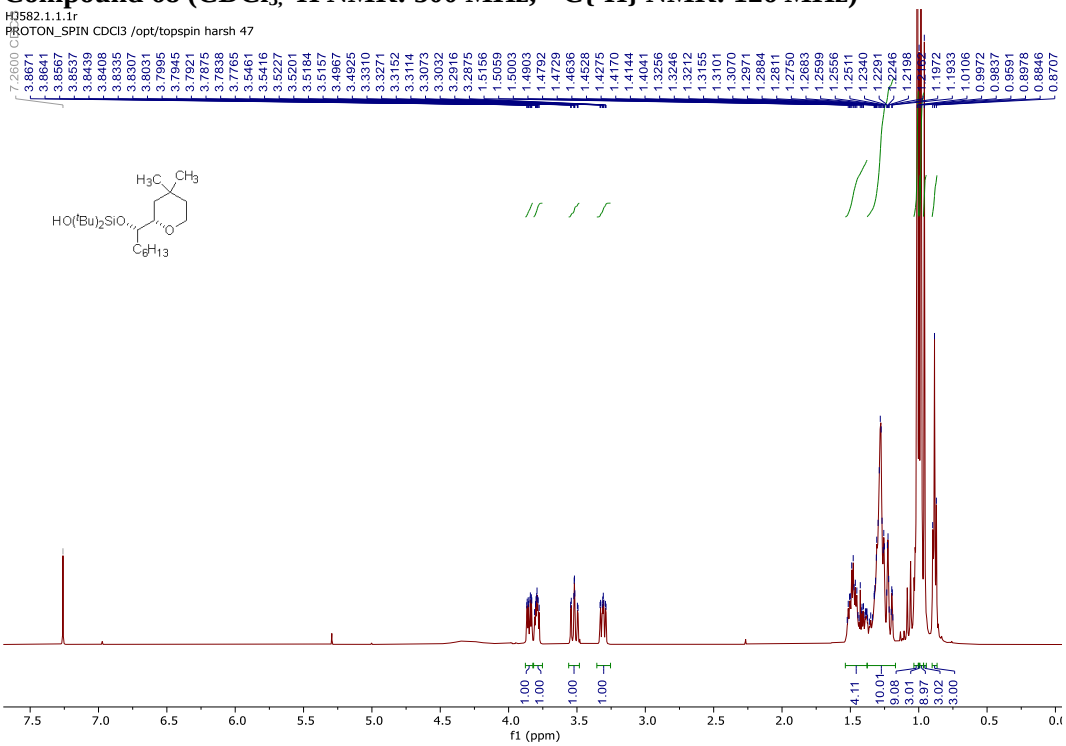


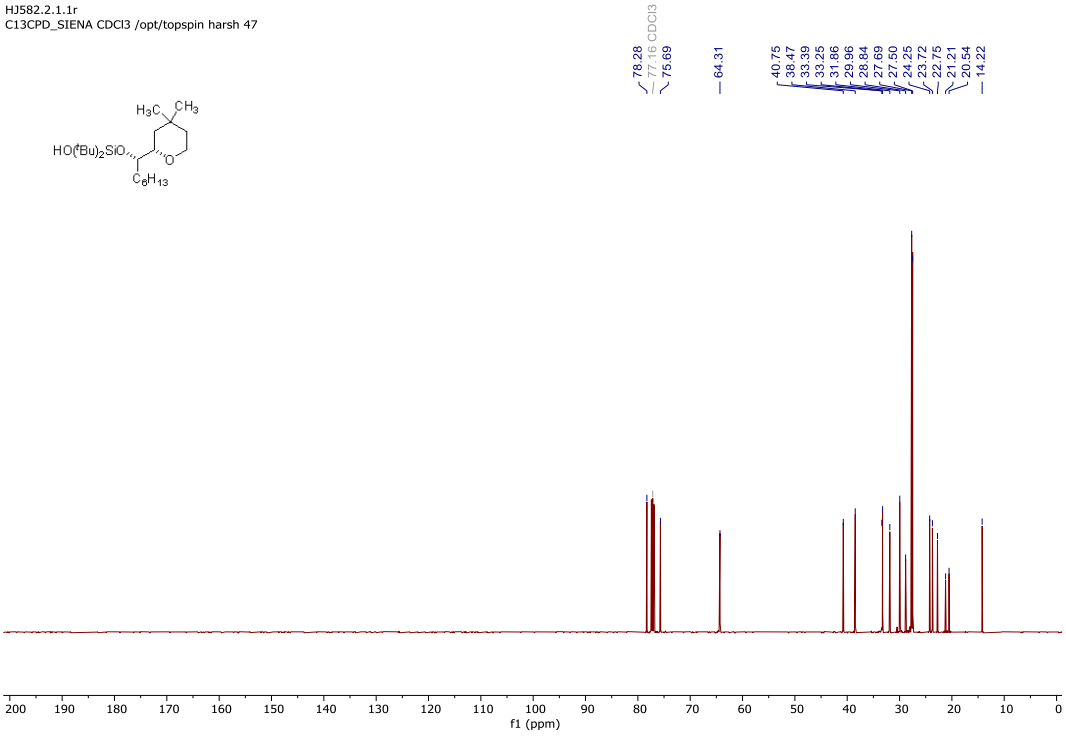
Compound 67 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



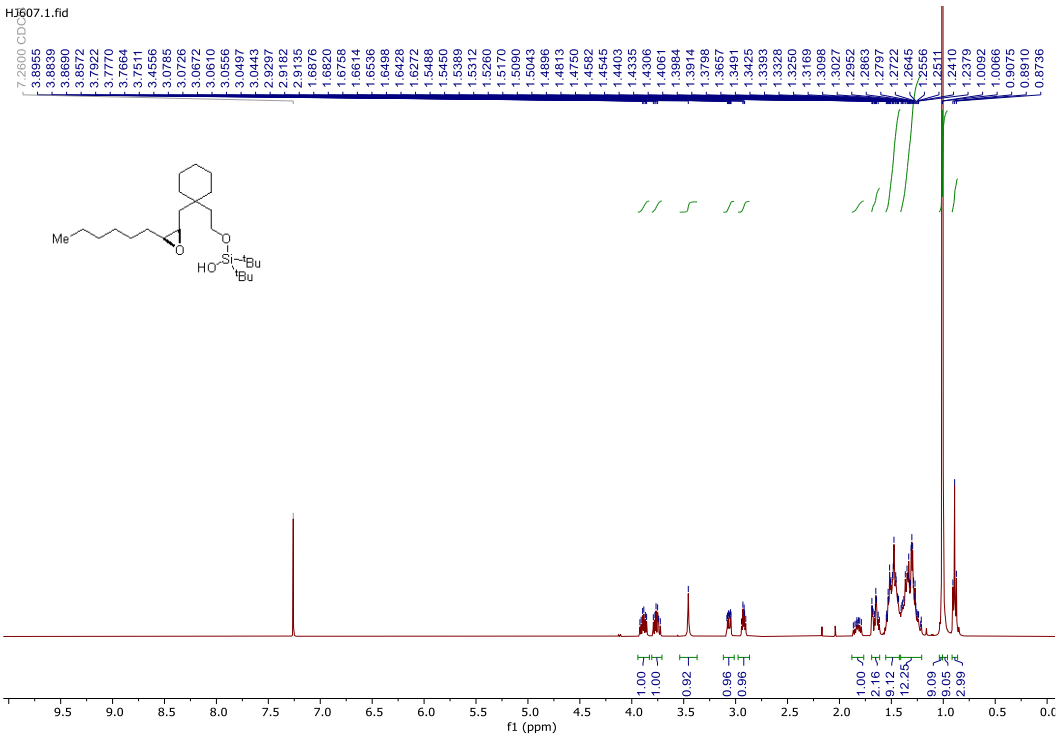


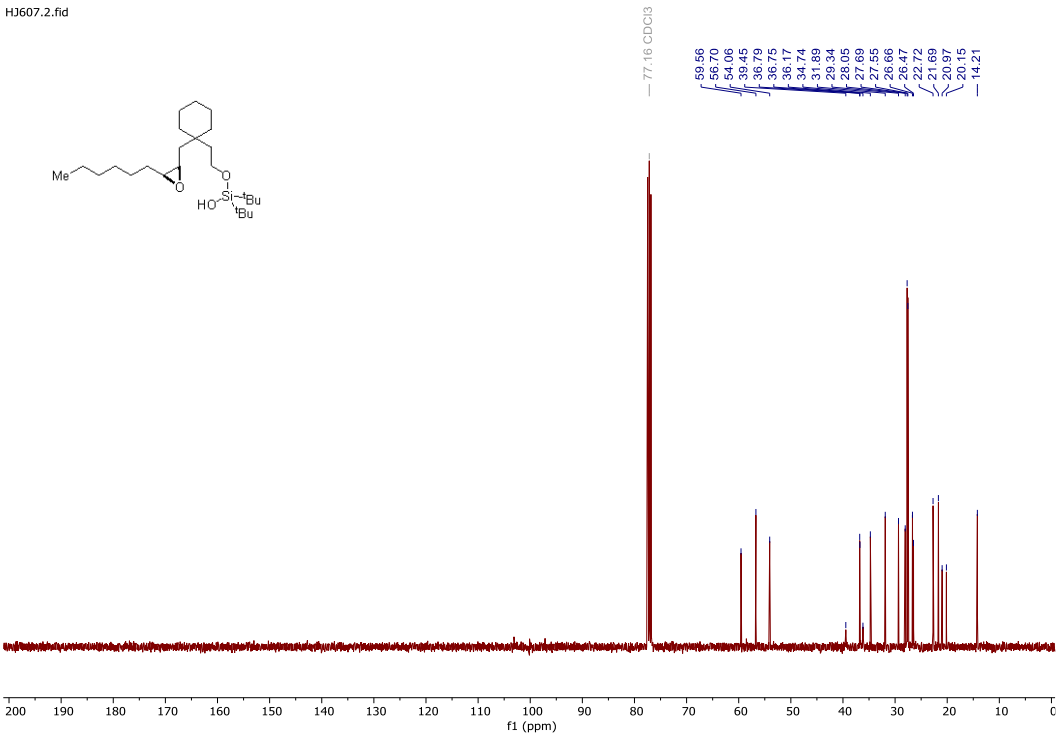
Compound 68 ($CDCl_3$, ^1H NMR: 500 MHz, $^{13}C\{^1H\}$ NMR: 126 MHz)



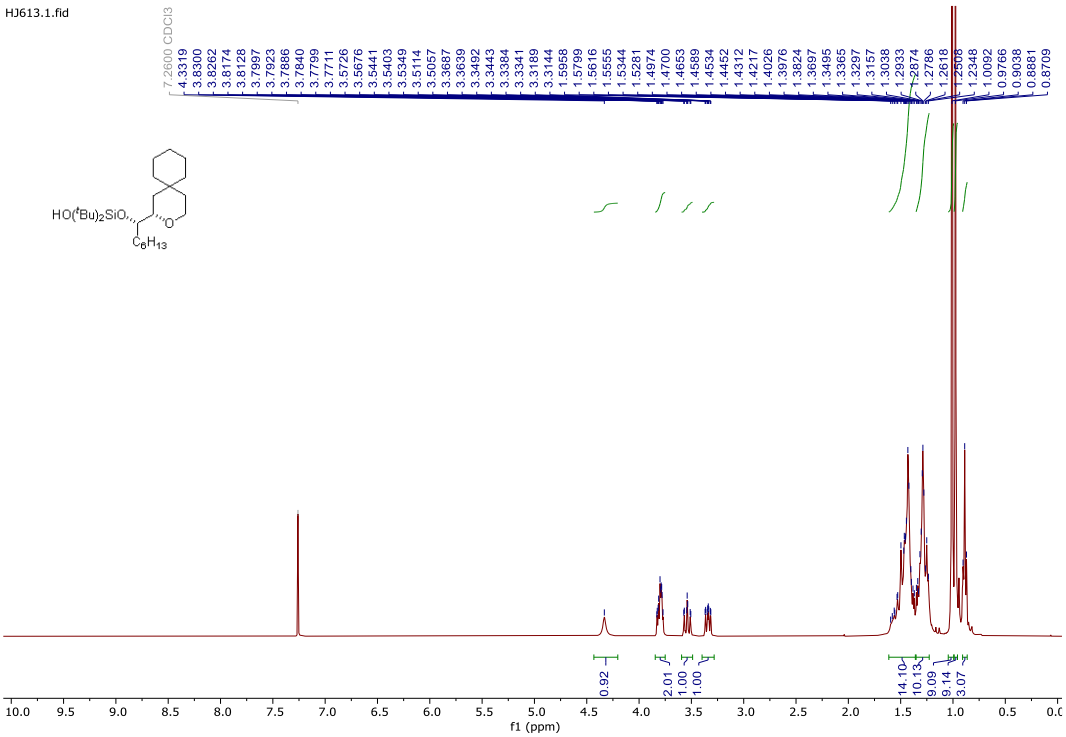


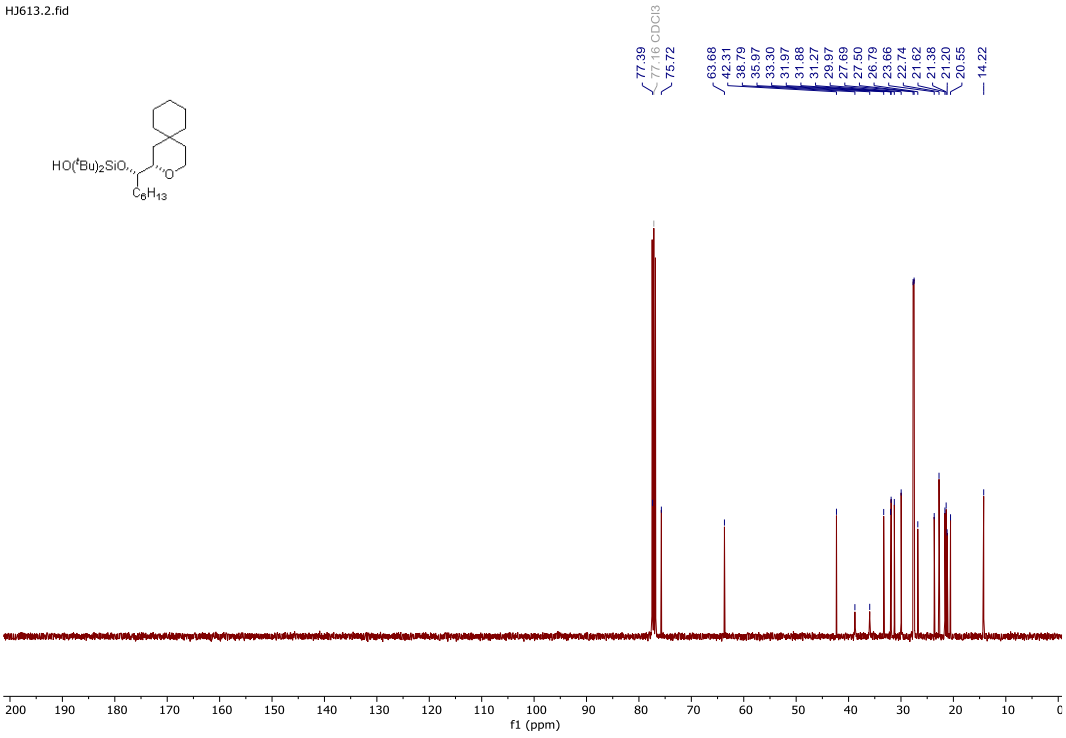
Compound 69 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



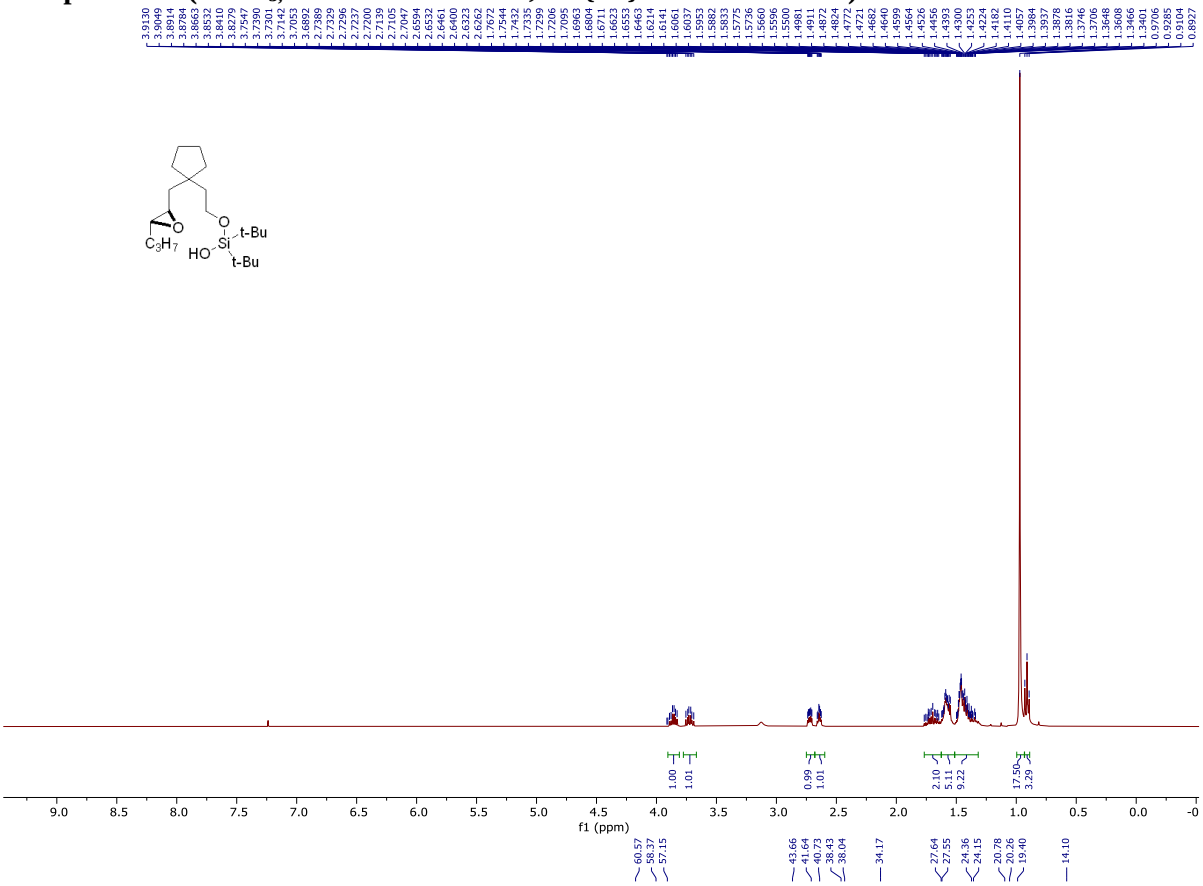


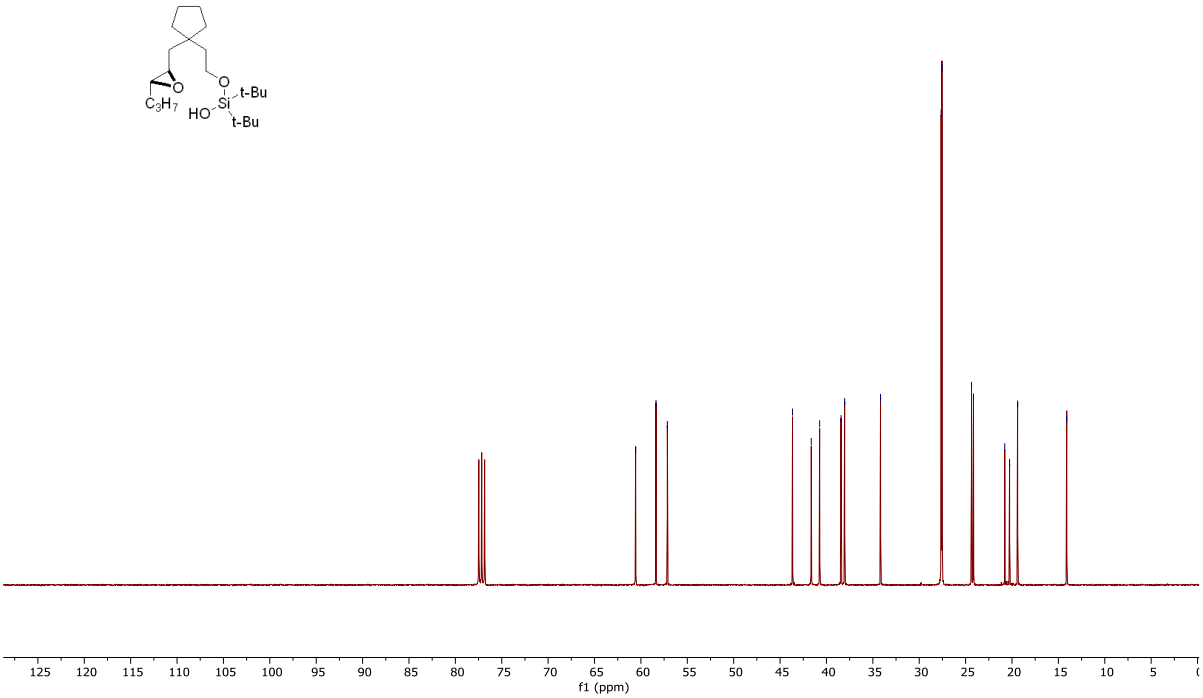
Compound 70 ($CDCl_3$, ^1H NMR: 500 MHz, $^{13}C\{^1H\}$ NMR: 126 MHz)



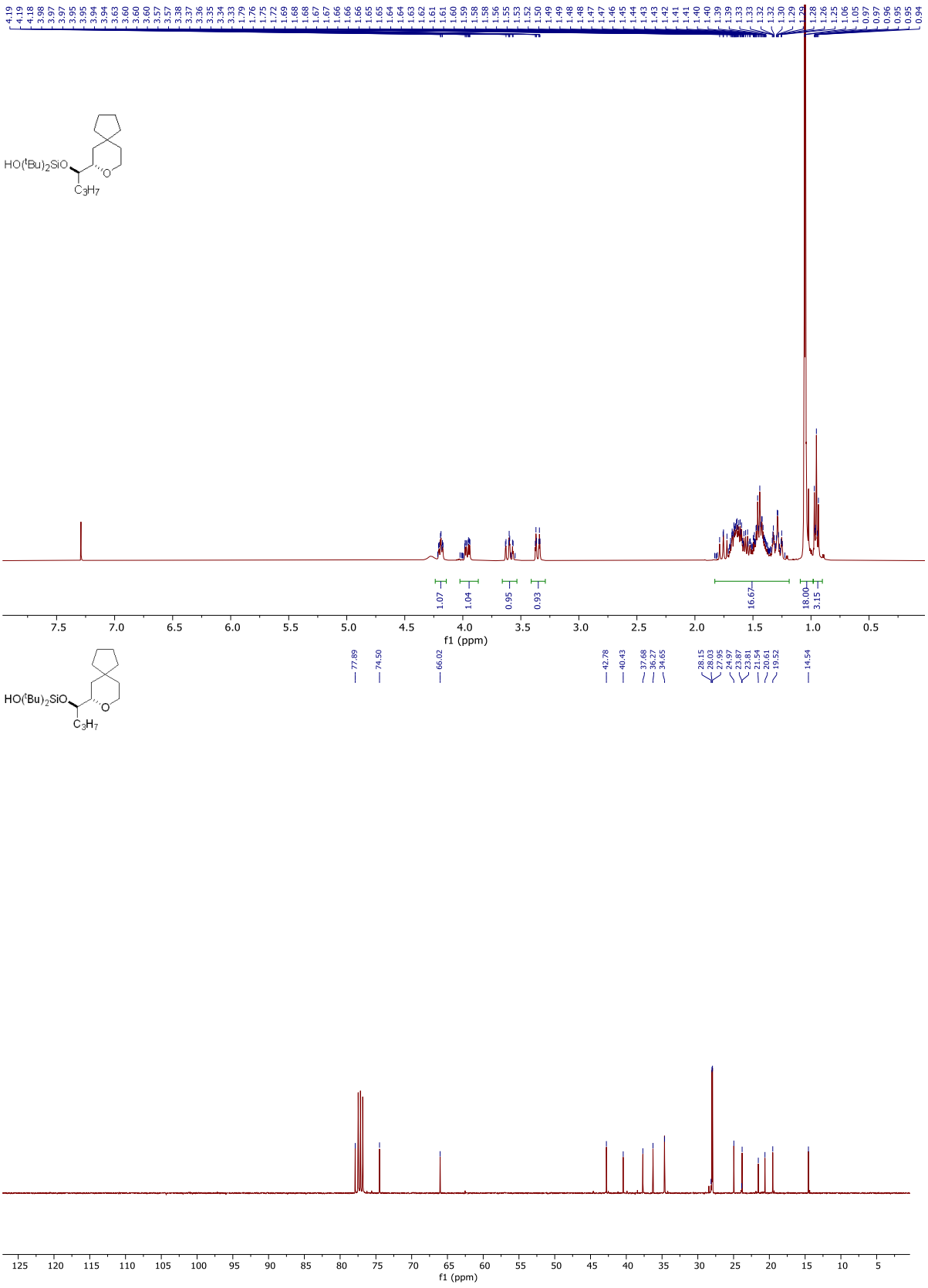


Compound 71 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

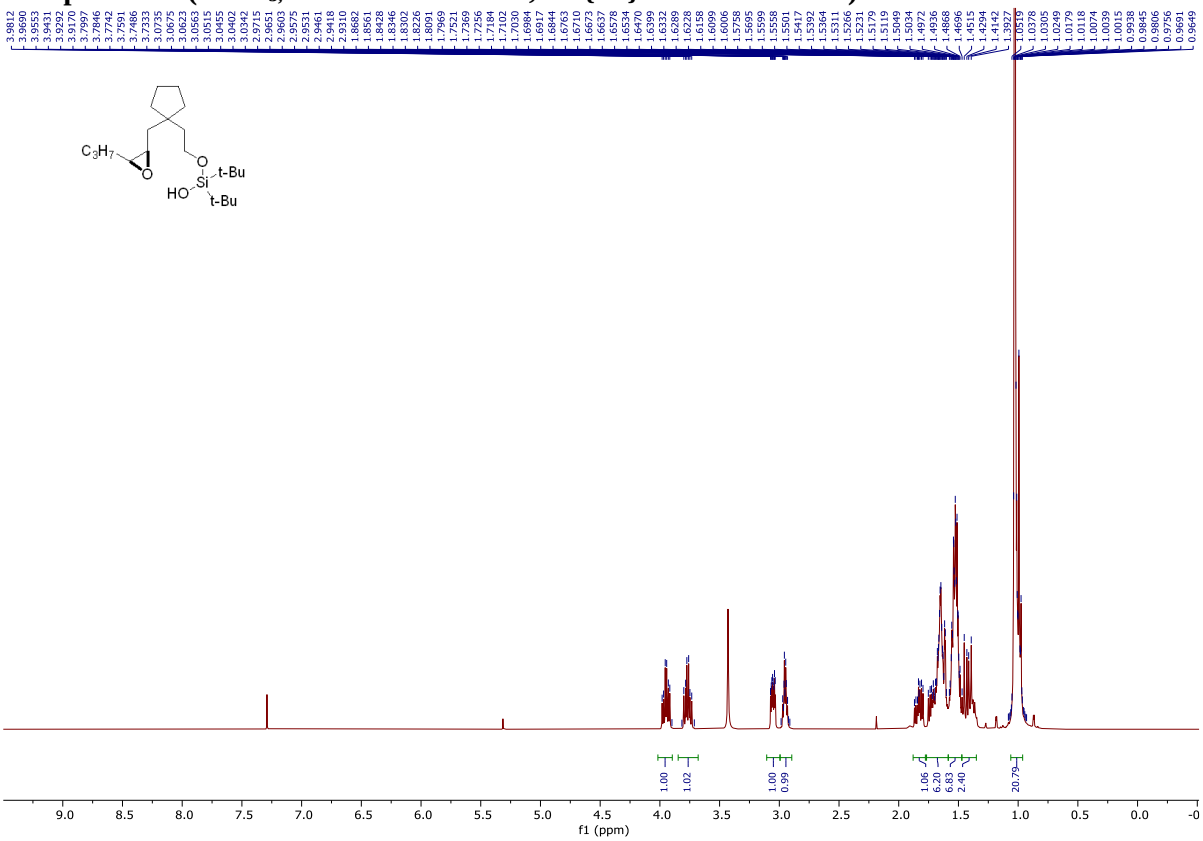


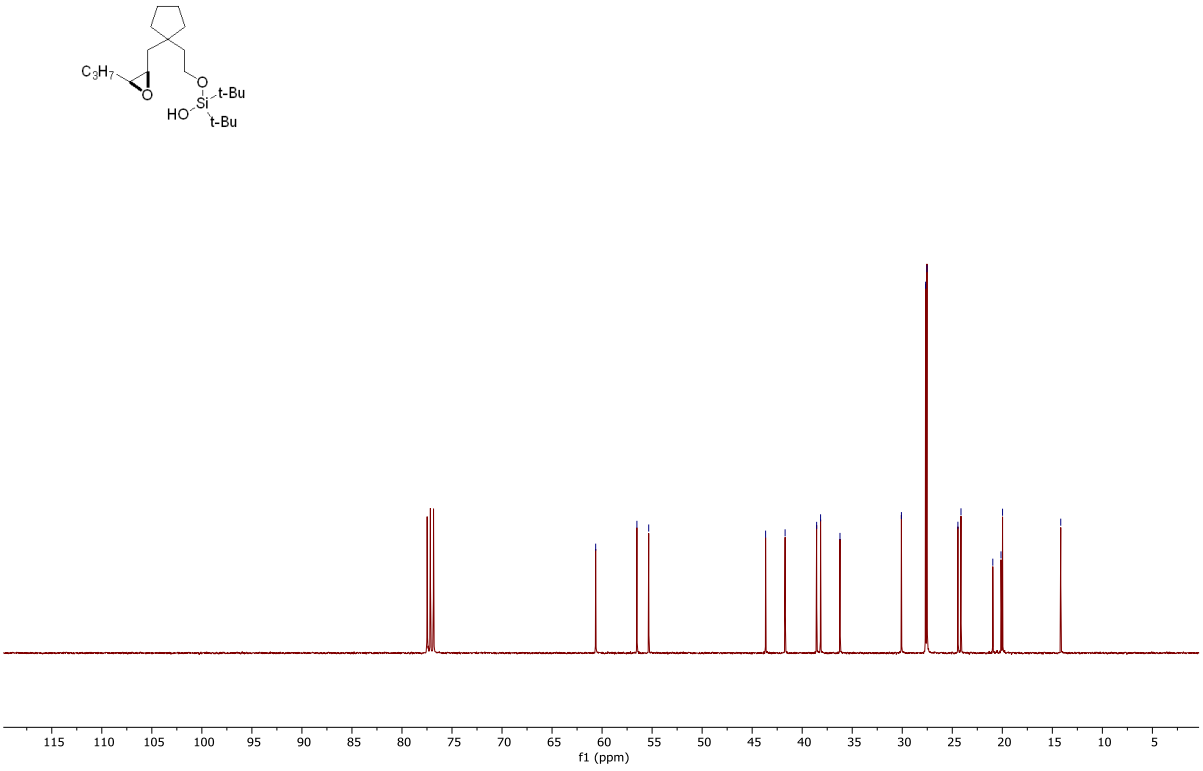


Compound 72 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

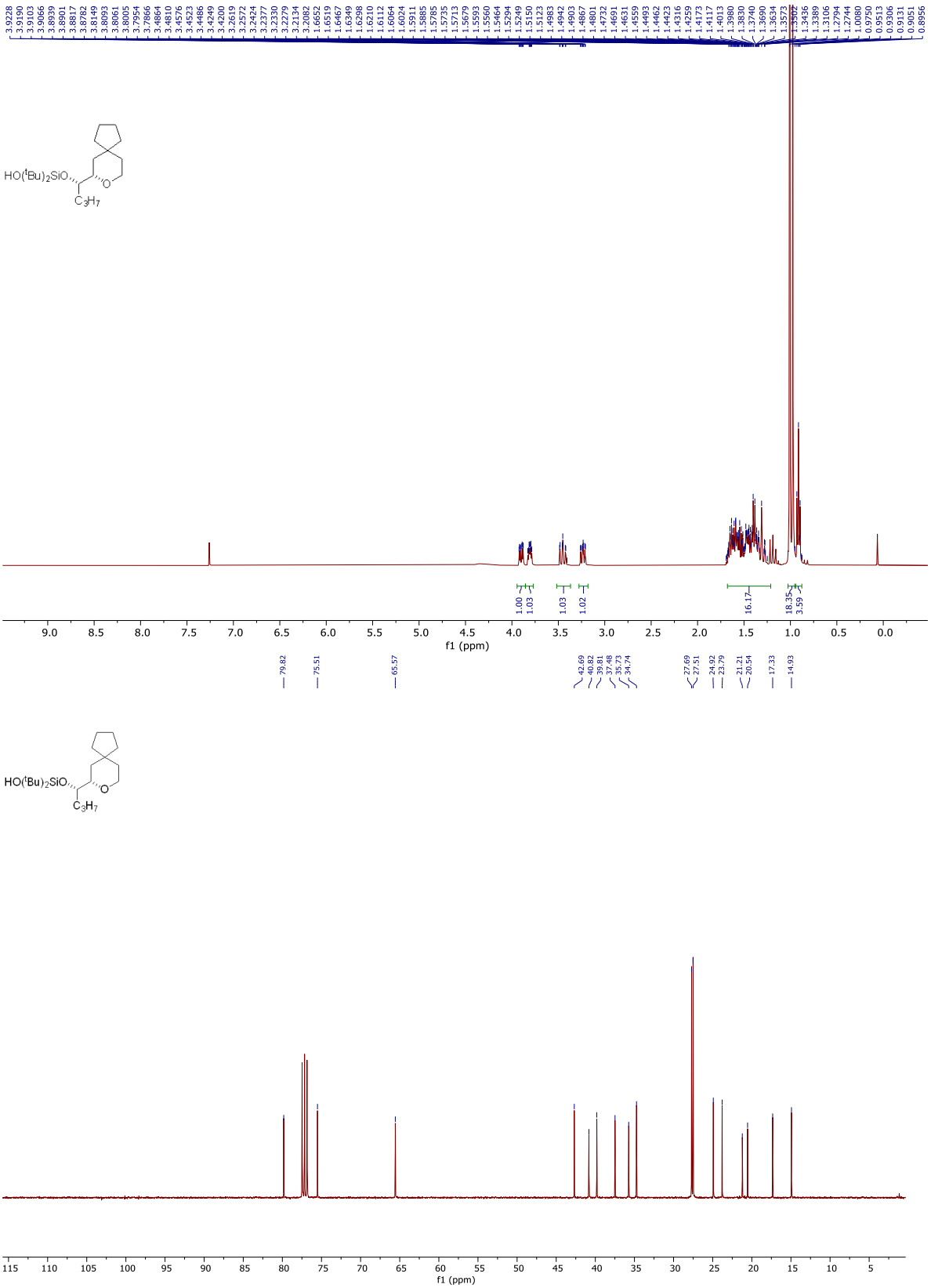


Compound 73 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

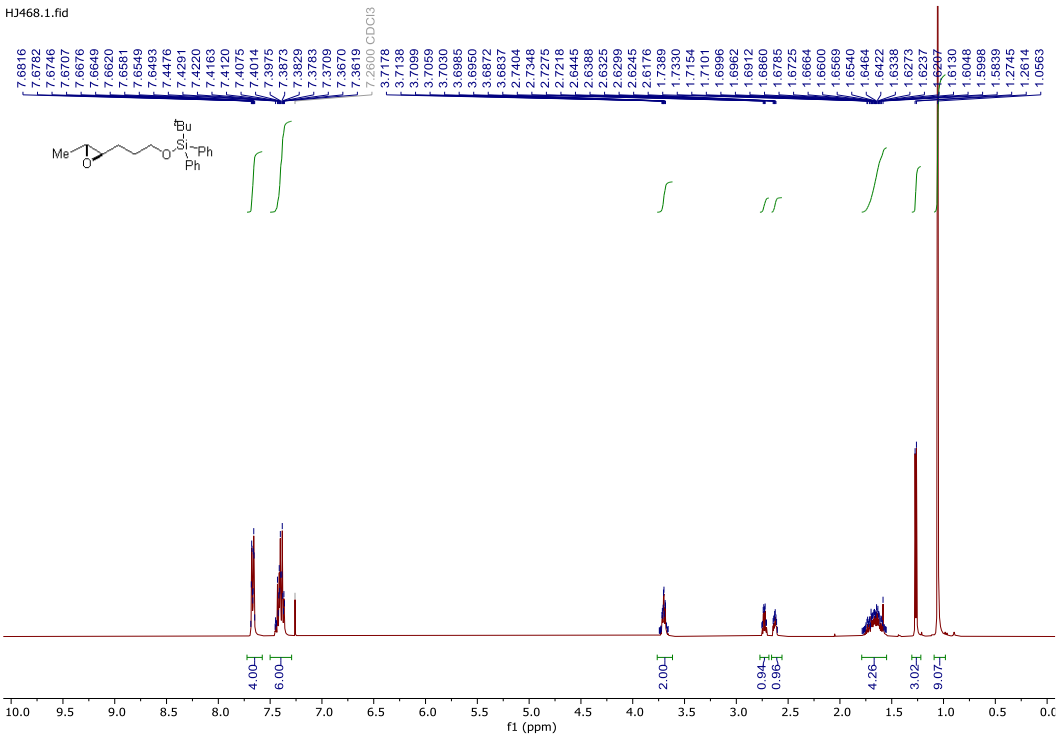


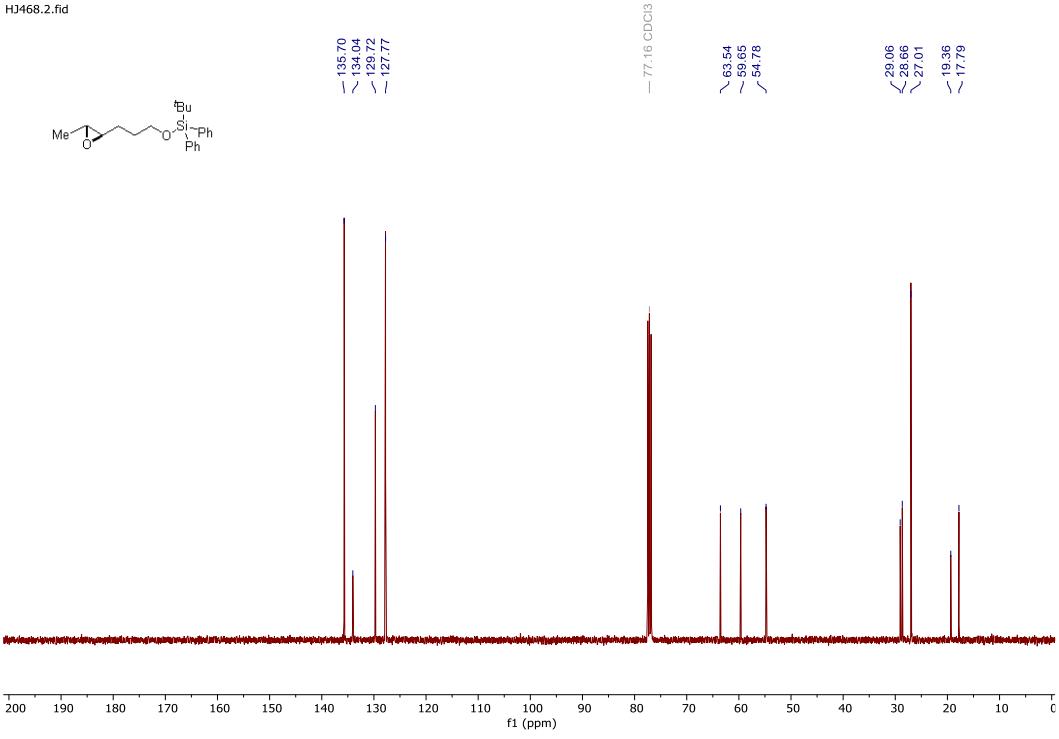


Compound 74 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)

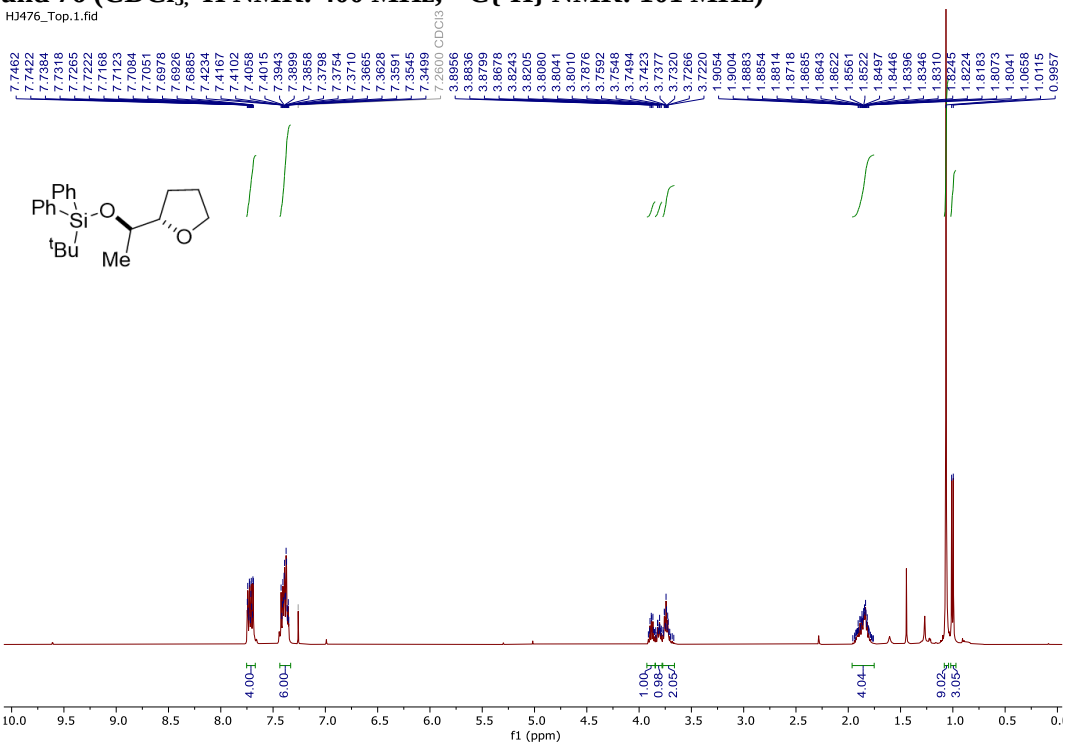


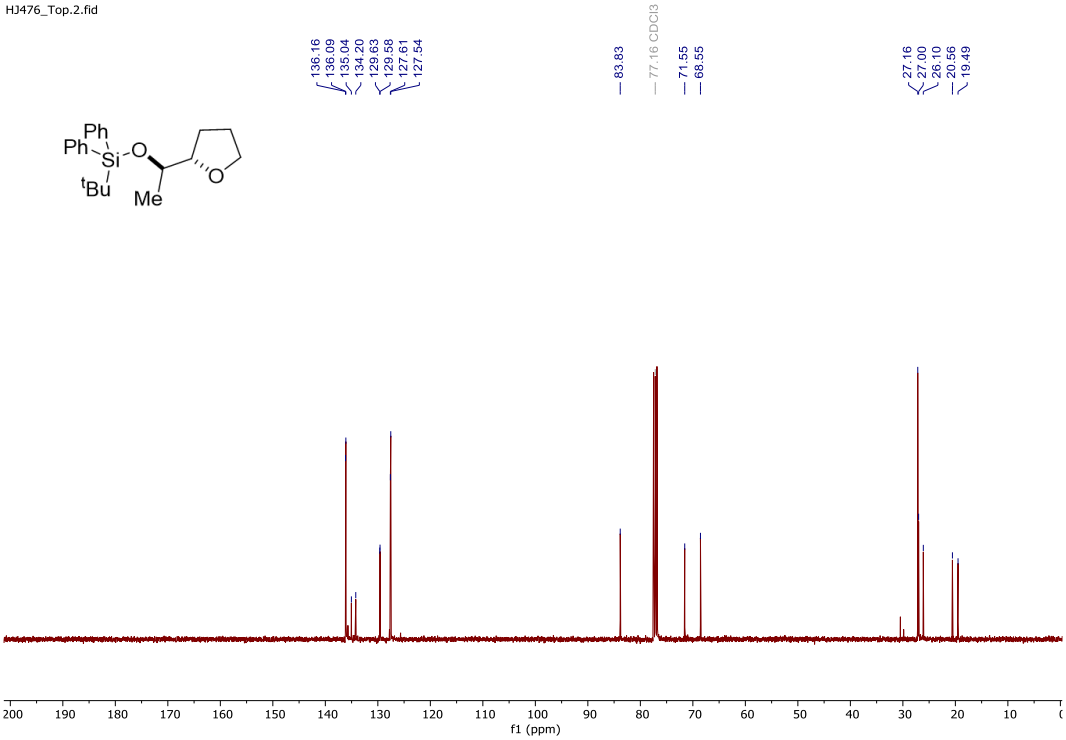
Compound 75 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



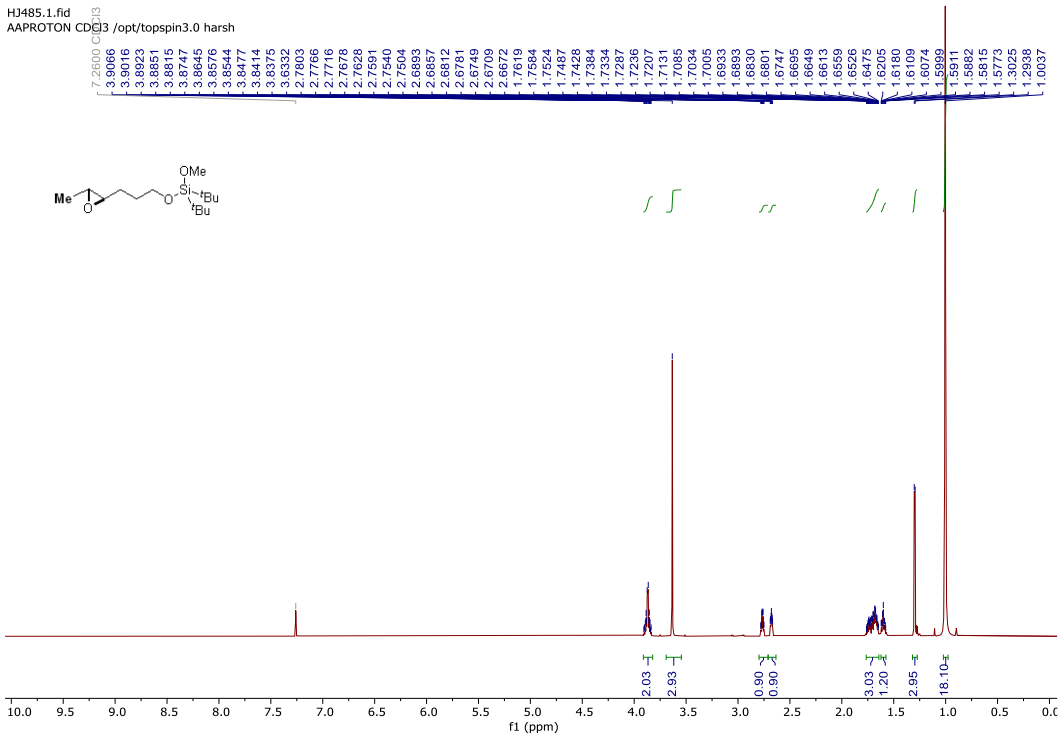


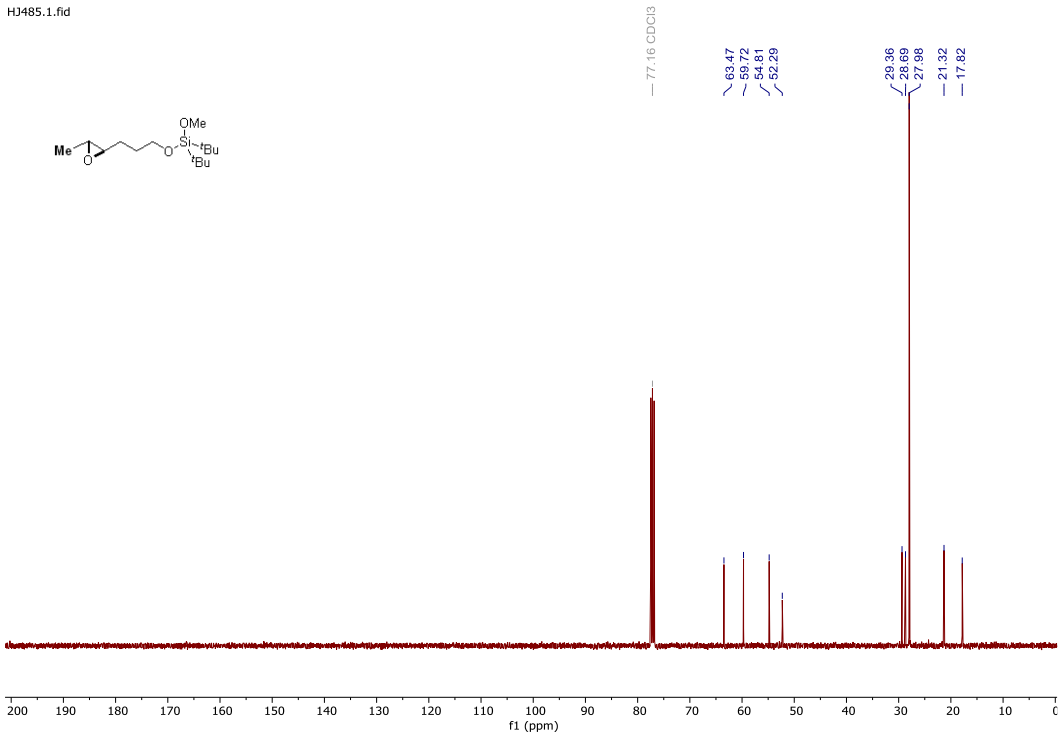
Compound 76 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



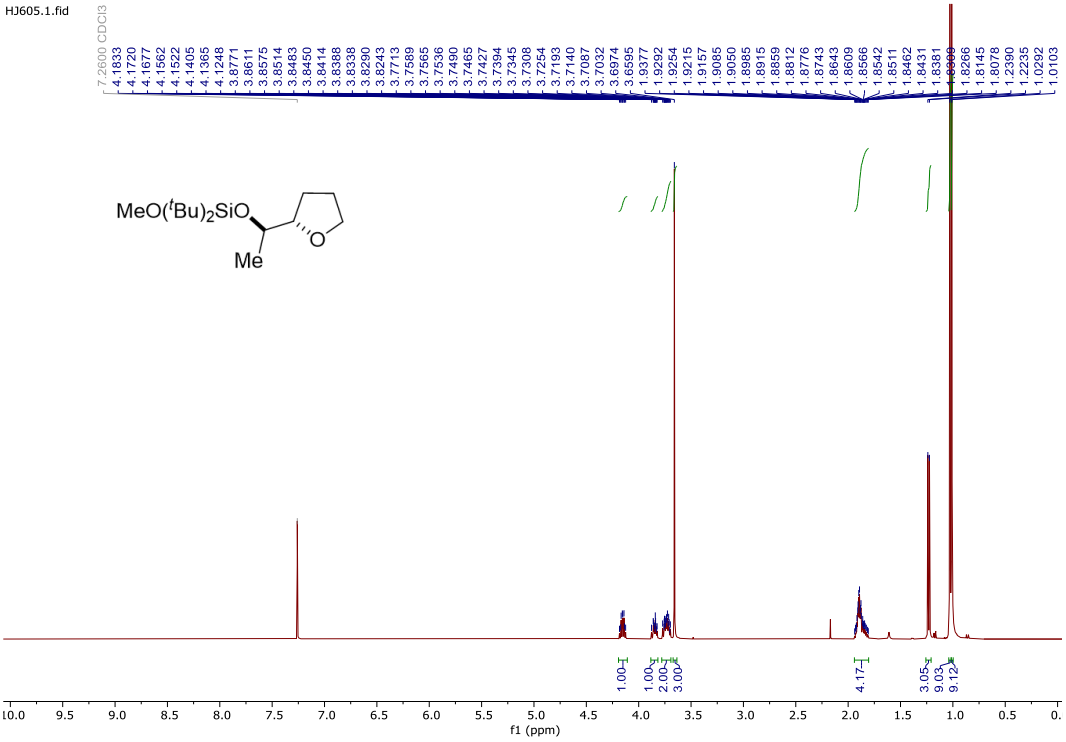


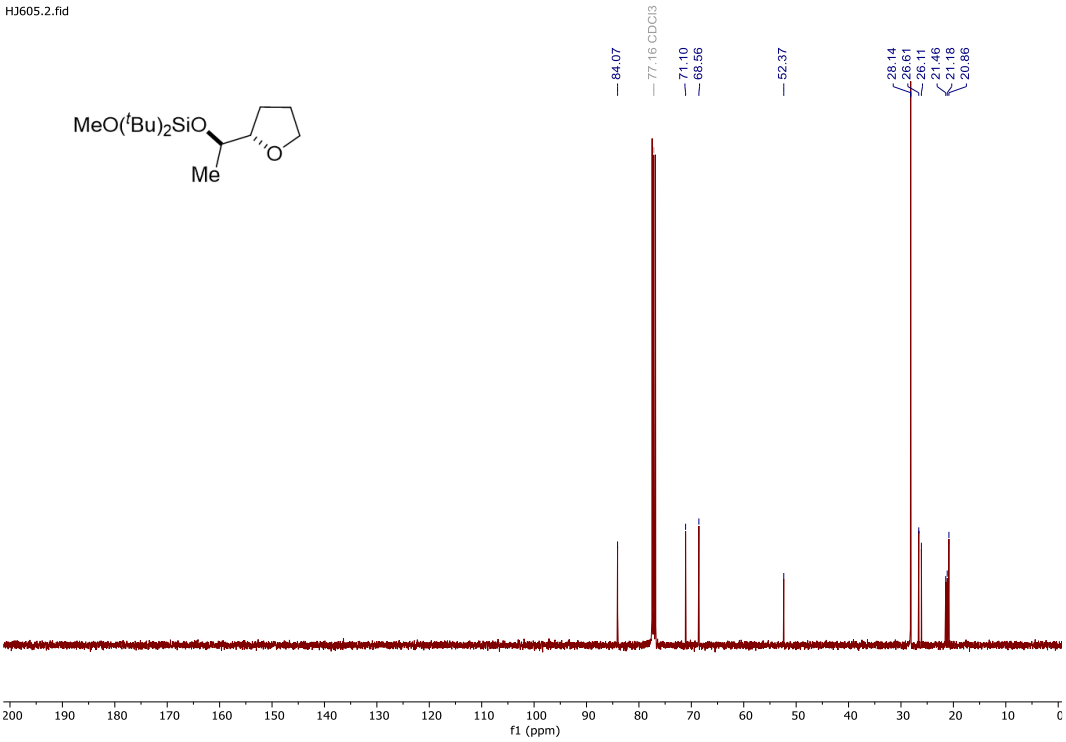
Compound 77 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



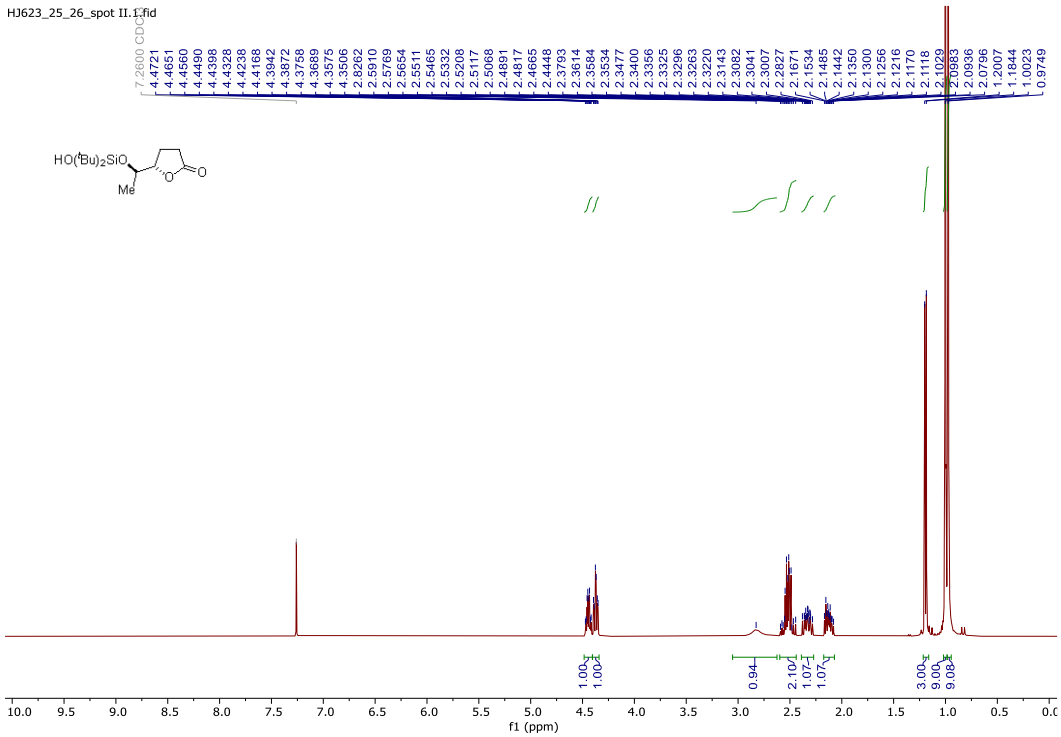


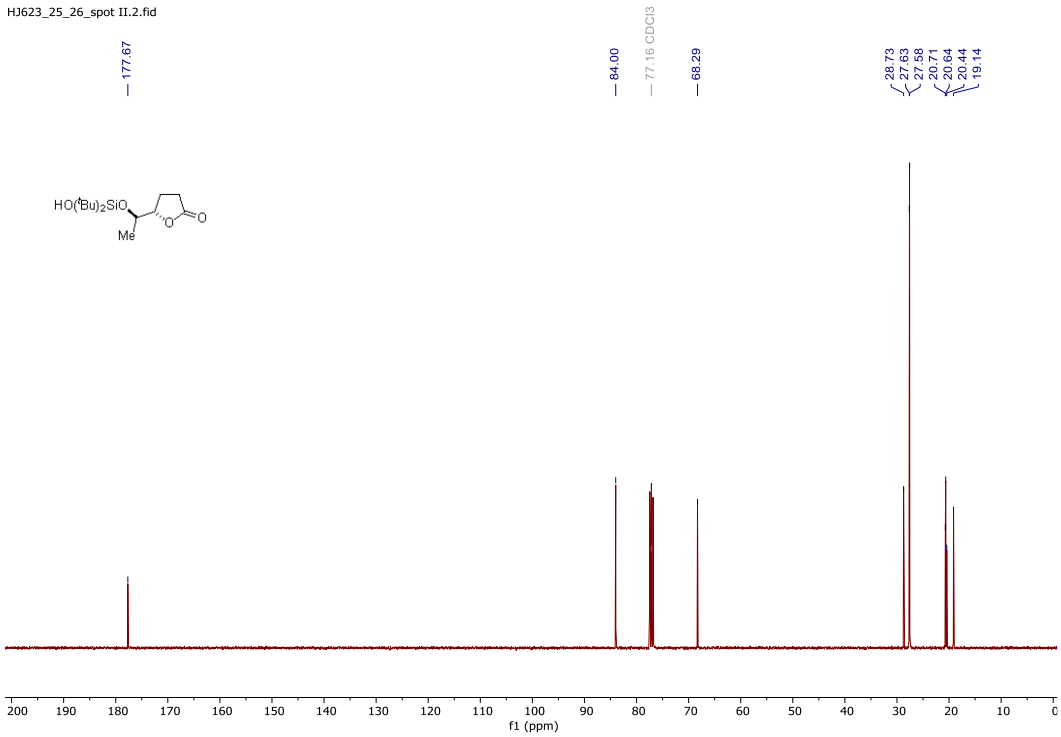
Compound 78 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



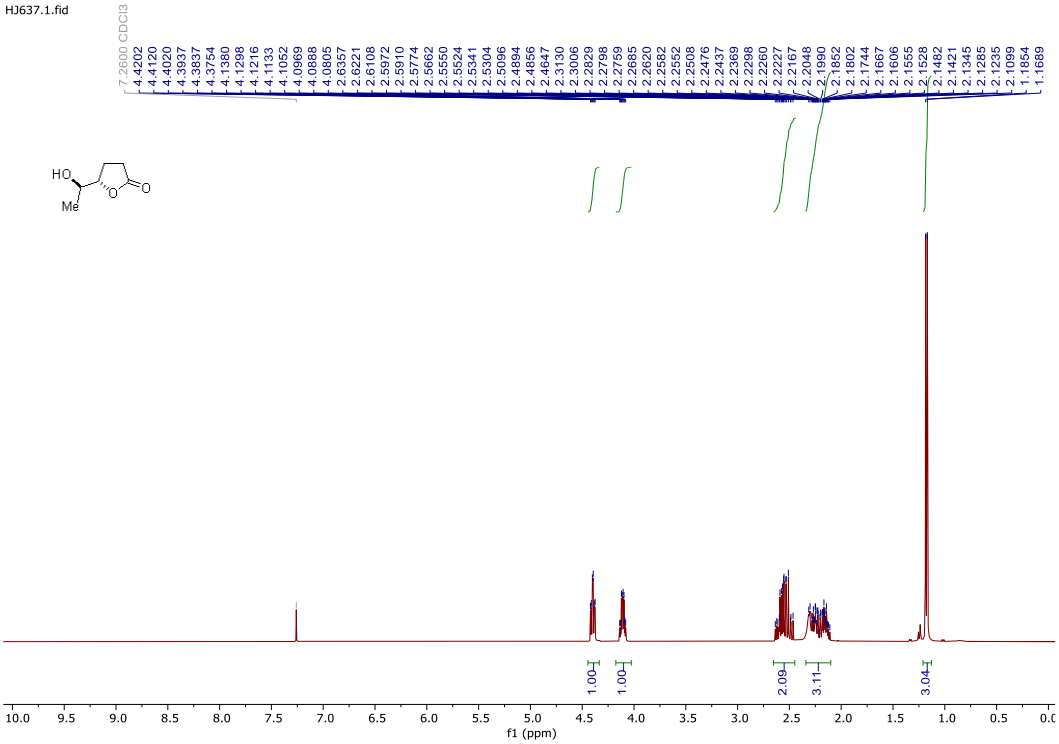


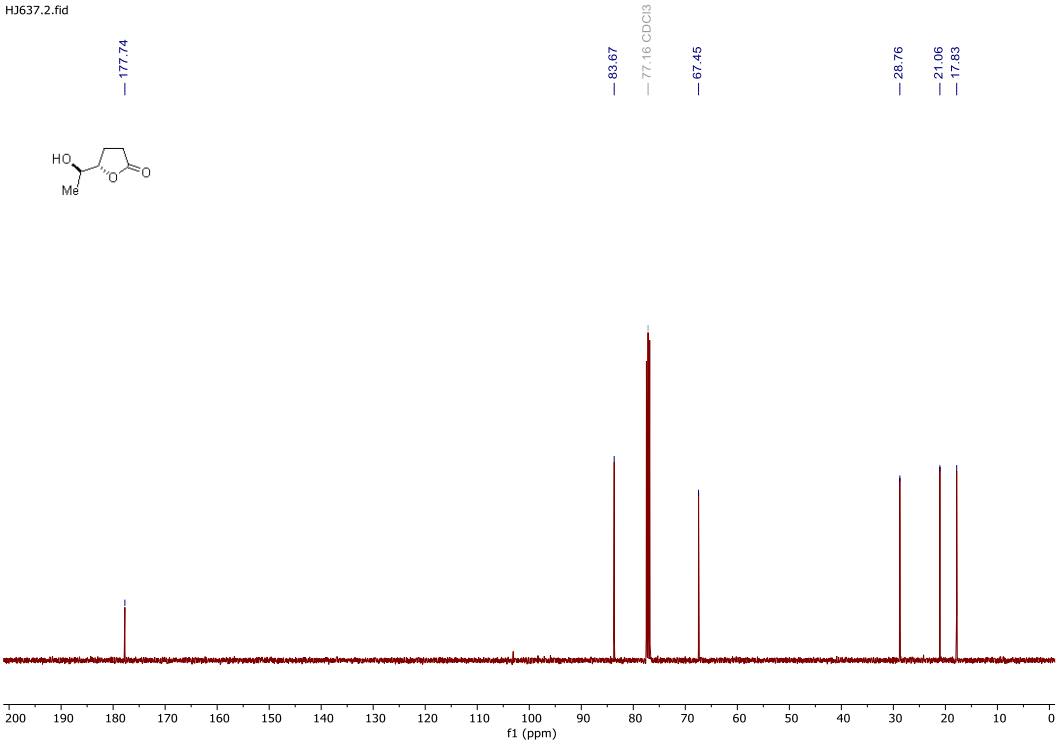
Compound 79 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)



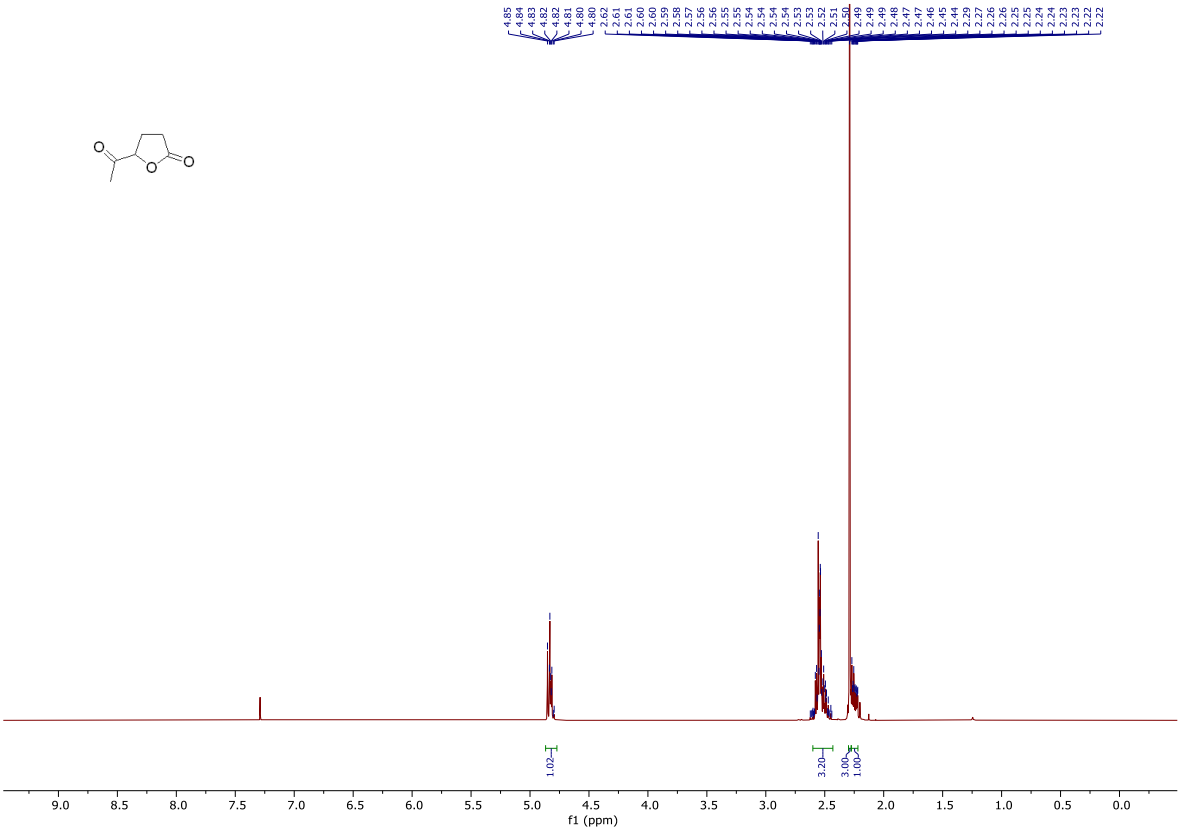


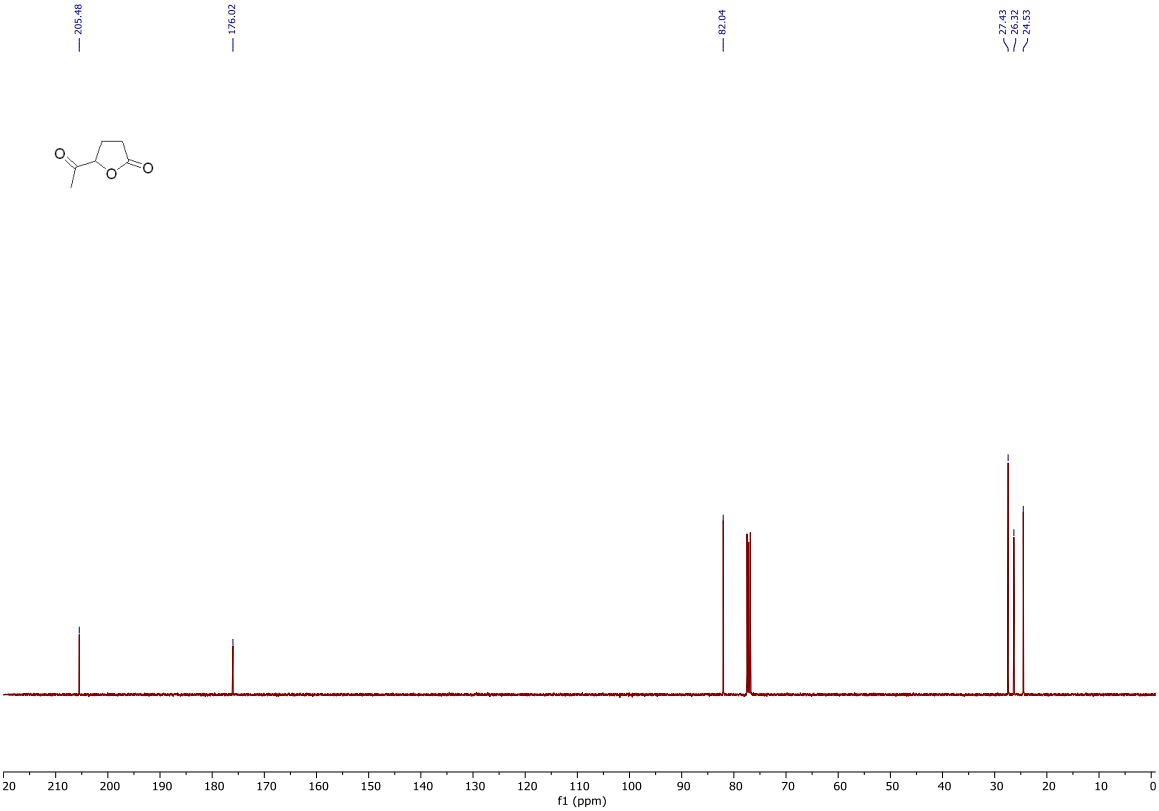
Compound 80 ($CDCl_3$, ^{1}H NMR: 400 MHz, $^{13}C\{^{1}H\}$ NMR: 101 MHz)



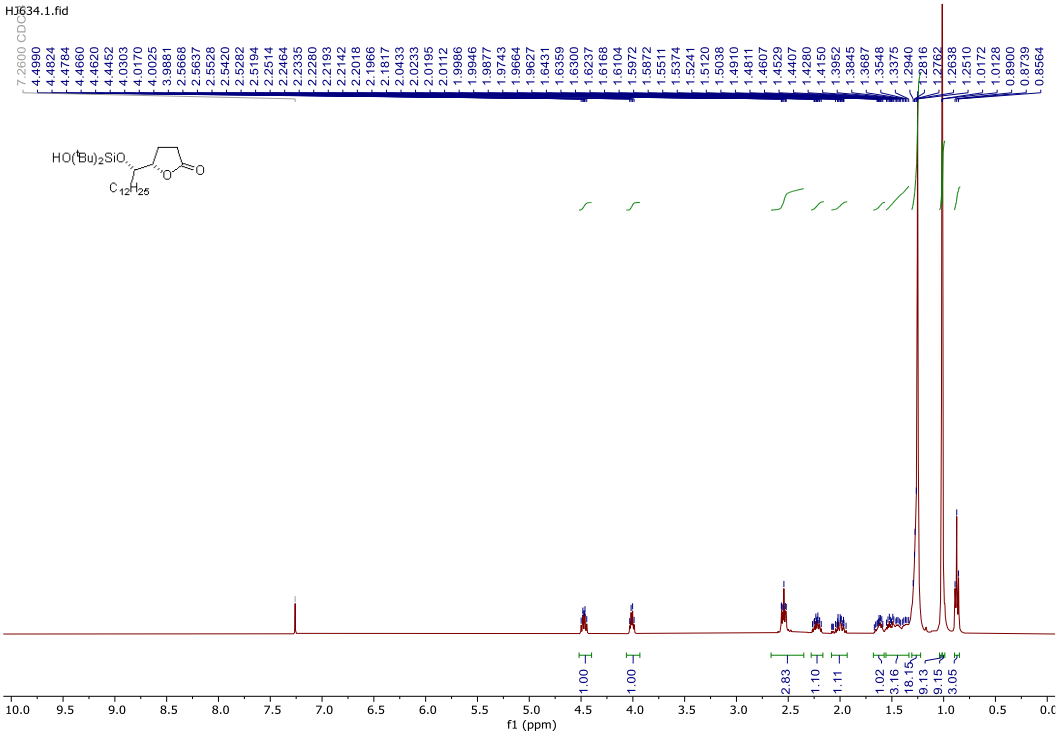


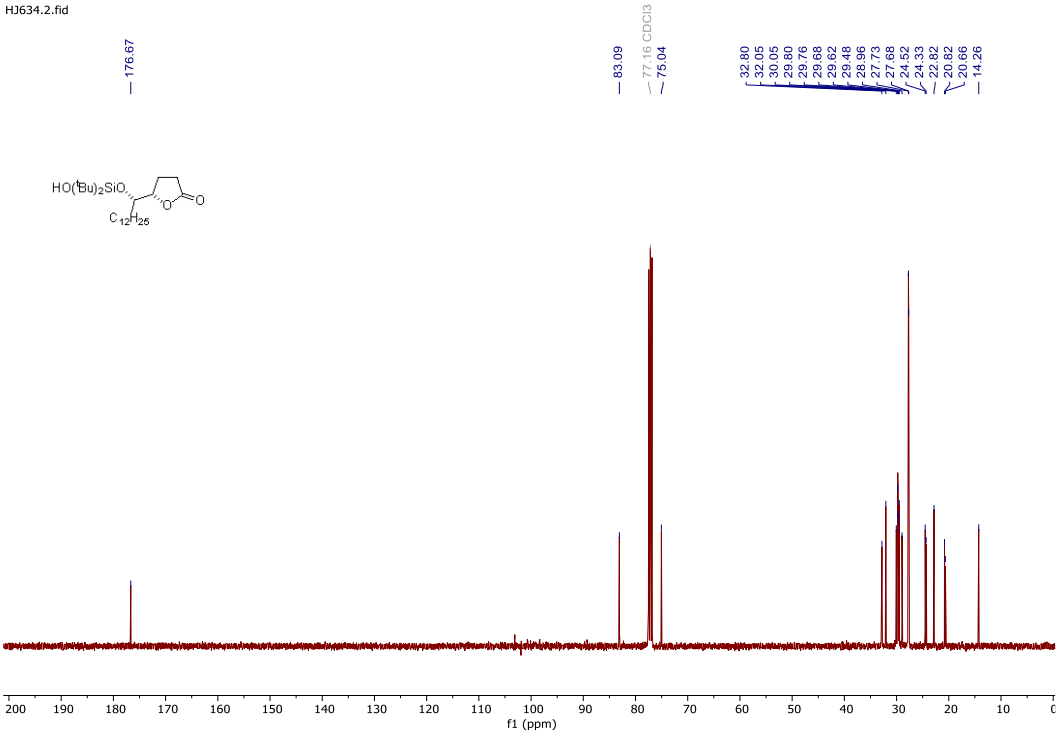
(±)-solerone ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)





Compound 81 ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)





(±)-muricatacin ($CDCl_3$, ^1H NMR: 400 MHz, $^{13}C\{^1H\}$ NMR: 101 MHz)