Fe-Catalyzed B–H and Si–H Insertion Reactions of *gem*-Dihaloalkanes

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Supporting Information

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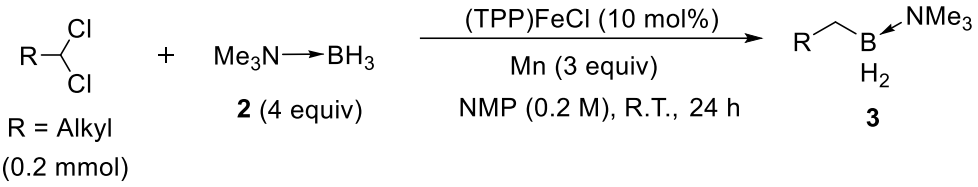
I. General Information

Unless otherwise noted, reagents were used as received from Sigma-Aldrich, Alfa, TCI, Bidepharm, Energy Chemical, J&K, Adamas. All reactions were performed under an atmosphere of dry nitrogen gas. Anhydrous DME was purchased from J&K and stored under nitrogen gas. Other solvents were purified with activated aluminum oxide using a solvent-purification system. *Gem*-dihaloalkanes were synthesized from corresponding aldehydes based on reported literatures.[1]

NMR spectra were recorded on a Bruker spectrometer with a Prodigy broadband cryoprobe (500 MHz for ^{1}H and 126 MHz for ^{13}C or 600 MHz for ^{1}H and 151 MHz for ^{13}C); chemical shifts (δ) are reported in ppm downfield from tertramethylsilane, using the solvent resonance as the internal standard. High resolution mass spectrometric analysis was performed on ultra-performance liquid chromatography-time-of-flight mass spectrometer (Synapt-G2-Si, Waters, USA) with electron spray ionization (ESI) resource, Atmospheric pressure chemical ionization (APCI) resource. Gas chromatography-mass spectrometry analysis was performed on Gas chromatography-mass spectrometry system (5977C GC/MSD, Agilent, USA).

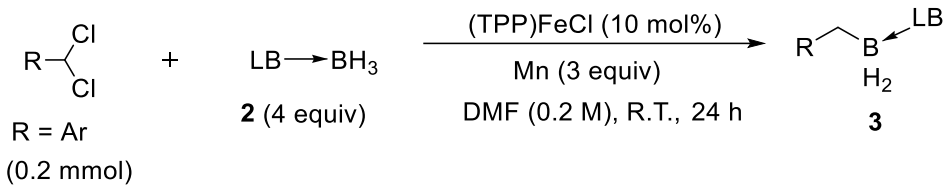
II. Catalytic carbene insertion Reactions

General procedure: Catalytic B-H carbene insertion Reactions.



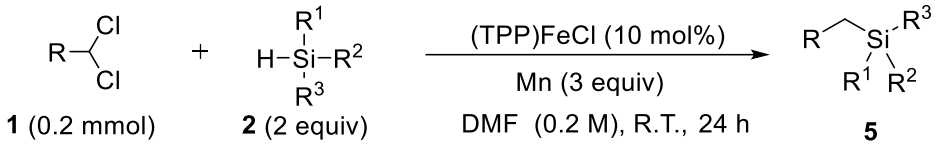
General procedure 1 (GP-1): Inside the glovebox under N_2 atmosphere, (TPP)FeCl (14.1 mg, 0.020 mmol, 0.10 equiv), Mn (33.2 mg, 0.6 mmol, 3.0 equiv) and N-Methylpyrrolidone (NMP) (1 mL) were added to an oven-dried 4 mL vial equipped with a magnetic stir bar. The reaction mixture was stirred (800 r/min) at room temperature for 0.5 hours. Next, the alkyl dichlorides (0.2 mmol, 1.0 equiv) and trimethylamine borane (58.4 mg, 0.80 mmol, 4.0 equiv) were added sequentially, The vial was quickly sealed with a PTFE septum cap. Then the vial was taken out of the glovebox, and stirred (800 r/min) at 25 °C for 24 hours.

Work-up: The reaction mixture was diluted with 1.0 mL ether,. then the reaction solvent was removed by concentration under reduced pressure. The residue was subjected to flash column chromatography with petroleum ether (PE) and ethyl acetate (EA) as the eluent to afford the desired borane products.



General procedure 2 (GP-2): Inside the glovebox under N_2 atmosphere, (TPP)FeCl (14.1 mg, 0.020 mmol, 0.10 equiv), Mn (33.2 mg, 0.6 mmol, 3.0 equiv) and N,N-dimethylformamide (DMF) (1 mL) were added to an oven-dried 4 mL vial equipped with a magnetic stir bar. The reaction mixture was stirred (800 r/min) at room temperature for 0.5 hours. Next, the aryl dichlorides (0.2 mmol, 1.0 equiv) and lewis base borane adduct (0.40 mmol, 2.0 equiv) were added sequentially, The vial was quickly sealed with a PTFE septum cap. Then the vial was taken out of the glovebox, and stirred (800 r/min) at 25 °C for 24 hours.

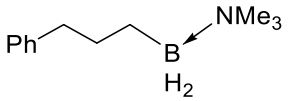
Work-up: The reaction mixture was diluted with 1.0 mL ether, then the reaction solvent was removed by concentration under reduced pressure. The residue was subjected to flash column chromatography with hexane and ethyl acetate (EA) as the eluent to afford the desired borane products.



General procedure 3 (GP-3): Inside the glovebox under N_2 atmosphere, (TPP)FeCl (14.1 mg, 0.020 mmol, 0.10 equiv), Mn (33.2 mg, 0.60 mmol, 3.0 equiv) and N,N-dimethylformamide (DMF) (1 mL) were added to an oven-dried 4 mL vial equipped with a magnetic stir bar. The reaction mixture was stirred (800 r/min) at room temperature for 0.5 hours. Next, the aryl dichlorides (0.2 mmol, 1.0 equiv) and substituted silane (0.40 mmol, 2.0 equiv) were added sequentially, The vial was quickly sealed with a PTFE septum cap. Then the vial was taken out of the glovebox, and stirred (800 r/min) at 25 °C for 24 hours.

Work-up: The reaction mixture was diluted with 1.0 mL ether, then the reaction solvent was removed by concentration under reduced pressure. The residue was subjected to flash column chromatography with petroleum ether (PE) and ethyl acetate (EA) as the eluent to afford the desired silane products.

Note: some silane products have been reported in literature[2-12]



Trimethylamine (3-phenylpropyl)borane (Figure 2, 3a). The title compound was prepared according to the **GP-1** from (3,3-dichloropropyl)benzene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, brown oil. 1st run: 26.3 mg, 69% yield; 2nd run: 26.4 mg, 70% yield.

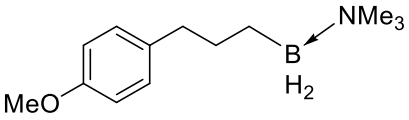
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.3 – 7.1 (m, 5H), 2.7 – 2.6 (m, 2H), 2.5 (s, 9H), 1.6 – 1.6 (m, 2H), 0.4 (p, *J* = 6.3 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 144.4, 128.5, 127.9, 125.0, 52.0, 39.6, 30.7.

HRMS (ESI) m/z $[M-H]^+$ calcd for $C_{12}H_{21}BN$: 190.1769, found:190.1775.

Gram-scale reaction： Inside the glovebox under N_2 atmosphere, Fe(TPP)Cl (781.5 mg, 1.110 mmol, 0.10 equiv), Mn (1.8 g, 33.3 mmol, 3.0 equiv) and N-Methylpyrrolidone (NMP) (55 mL) were added to an oven-dried 100 mL flask equipped with a magnetic stir bar. The reaction mixture was stirred (800 r/min) at room temperature for 0.5 hours. Next, (3,3-dichloropropyl)benzene (2.1 g, 11.1 mmol, 1.0 equiv) and trimethylamine borane (3.3 g, 44.4 mmol, 4.0 equiv) were added sequentially, The vial was quickly sealed with a PTFE septum cap. Then the vial was taken out of the glovebox, and stirred (800 r/min) at 25 °C for 24 hours.

Work-up: The reaction mixture was diluted with 55.0 mL ether,. then the reaction solvent was removed by concentration under reduced pressure. The residue was purified by flash column chromatography on silica gel: 10% to 20% EA in hexane, brown oil. 1.25 g, 59% yield.

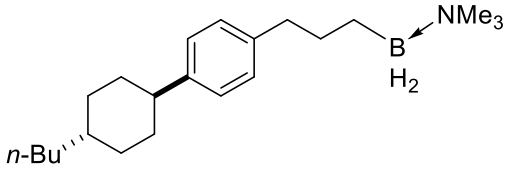


Trimethylamine (3-(4-methoxyphenyl)propyl)borane (Figure 2, 3b). The title compound was prepared according to the **GP-1** from 1-(3,3-dichloropropyl)-4-methoxybenzene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, yellow oil. 1st run: 22.6 mg, 51% yield; 2nd run: 23.0 mg, 52% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.12 (d, *J* = 8.5 Hz, 2H), 6.80 (d, *J* = 8.6 Hz, 2H), 3.78 (s, 3H), 2.61 – 2.56 (m, 2H), 2.54 (s, 9H), 1.58 – 1.50 (m, 2H, with water), 0.42 (dq, *J* = 11.9, 6.2 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 157.2, 136.6, 129.3, 113.4, 55.2, 52.0, 38.6, 31.0.

HRMS (ESI) m/z [M-H]$^{+}$ calcd for $C_{13}H_{23}BNO$: 220.1875, found:220.1867.

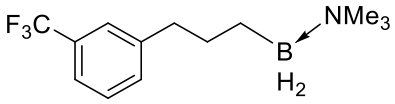


Trimethylamine (3-(4-((1s,4r)-4-butylcyclohexyl)phenyl)propyl)borane (Figure 2, 3c). The title compound was prepared according to the **GP-1** from 1-((1s,4r)-4-butylcyclohexyl)-4-(3,3-dichloropropyl)benzene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, yellow oil. 1st run: 38.2 mg, 58% yield; 2nd run: 37.5 mg, 57% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.15 – 7.04 (m, 4H), 2.63 – 2.57 (m, 2H), 2.54 (s, 9H), 2.41 (tt, *J* = 11.8, 3.3 Hz, 1H), 1.93 – 1.81 (m, 5H), 1.71 (p, *J* = 7.8 Hz, 1H), 1.63 – 1.18 (m, 15H), 1.10 – 0.98 (m, 2H), 0.93 – 0.86 (m, 3H), 0.45 (dq, *J* = 11.8, 6.2 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 144.5, 141.7, 128.3, 126.4, 52.0, 44.2, 39.2, 37.3, 37.2, 34.4, 33.7, 30.8, 29.3, 23.0, 14.2.

HRMS (ESI) m/z [M-H]$^{+}$ calcd for $C_{22}H_{39}BN$: 328.3180, found:328.3176.



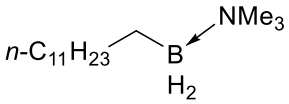
Trimethylamine (3-(3-(trifluoromethyl)phenyl)propyl)borane (Figure 2, 3d). The title compound was prepared according to the **GP-1** from 1-(3,3-dichloropropyl)-3-(trifluoromethyl)benzene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, yellow oil. 1st run: 31.6 mg, 61% yield; 2nd run: 31.1 mg, 60% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.46 (d, *J* = 1.7 Hz, 1H), 7.41 – 7.31 (m, 3H), 2.73 – 2.63 (m, 2H), 2.54 (s, 9H), 1.59 (tt, *J* = 9.5, 6.8 Hz, 2H), 0.42 (dq, *J* = 13.3, 6.4 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 131.9, 130.1 (q, *J* = 31.6 Hz), 128.3, 125.3 (q, *J* = 3.7 Hz), 124.5 (q, *J* = 272.2 Hz), 121.9 (q, *J* = 3.9 Hz), 52.0, 39.1, 30.5.

^{19}F NMR (565 MHz, Chloroform-*d*) δ -62.4.

HRMS (ESI) m/z [M-H]$^{+}$ calcd for $C_{13}H_{20}BF3N$: 258.1643, found:258.1655.

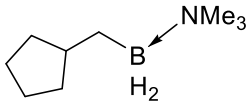


Trimethylamine dodecylborane (Figure 2, 3e). The title compound was prepared according to the **GP-1** from 1,1-dichlorododecane and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, colorless oil. 1st run: 24.1 mg, 50% yield; 2nd run: 24.6 mg, 51% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 2.55 (s, 9H), 1.32 – 1.21 (m, 22H), 0.87 (t, *J* = 7.0 Hz, 3H), 0.37 (dq, *J* = 12.1, 6.3 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 52.0, 33.6, 31.9, 30.0, 29.9, 29.8, 29.8, 29.7, 29.4, 28.8, 22.7, 14.1.

HRMS (ESI) m/z $[M-H]^+$ calcd for $C_{15}H_{35}BN$: 240.2865, found: 240.2865.

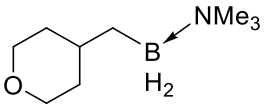


Trimethylamine (cyclopentylmethyl)borane (Figure 2, 3f). The title compound was prepared according to the **GP-1** from (dibromomethyl)cyclopentane and trimethylamine borane, purified by flash column chromatography on silica gel: 10% EA in hexane, colorless oil. 1st run: 25.4 mg, 82% yield; 2nd run: 25.8 mg, 83% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 2.54 (s, 9H), 1.79 – 1.40 (m, 9H), 1.13 – 0.99 (m, 2H), 0.45 (q, *J* = 6.5 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 51.9, 39.9, 35.7, 25.3.

HRMS (ESI) m/z $[M-H]^+$ calcd for $C_9H_{21}BN$: 154.1769, found: 154.1773.

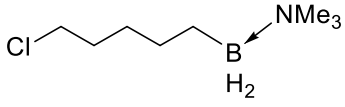


Trimethylamine ((tetrahydro-2H-pyran-4-yl)methyl)borane (Figure 2, 3g). The title compound was prepared according to the **GP-1** from 4-(dibromomethyl)tetrahydro-2*H*-pyran and trimethylamine borane, purified by flash column chromatography on silica gel: 40% EA in hexane, white soild. m.p.:72 – 73 °C. 1st run: 15.4 mg, 45% yield; 2nd run: 15.1 mg, 44% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 3.90 (dtd, *J* = 11.5, 2.5, 1.1 Hz, 2H), 3.36 (td, *J* = 11.7, 2.1 Hz, 2H), 2.54 (s, 9H), 1.65 (ddd, *J* = 13.0, 4.0, 1.9 Hz, 2H), 1.38 (dtq, *J* = 14.1, 6.7, 3.3 Hz, 1H), 1.30 – 1.16 (m, 2H), 0.34 (q, *J* = 6.4 Hz, 2H)

^{13}C NMR (126 MHz, Chloroform-*d*) δ 68.8, 52.0, 36.5, 34.9.

HRMS (ESI) m/z $[M-H]^+$ calcd for $C_9H_{21}BNO$: 170.1718, found: 170.1722.



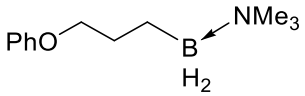
Trimethylamine (5-chloropentyl)borane (Figure 2, 3h). The title compound was

prepared according to the **GP-1** from 1,1-dibromo-5-chloropentane and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, colorless oil. 1st run: 22.0 mg, 62% yield; 2nd run: 22.3 mg, 63% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 3.53 (t, *J* = 7.0 Hz, 2H), 2.55 (s, 9H), 1.78 (p, *J* = 7.1 Hz, 2H), 1.48 – 1.41 (m, 2H), 1.32 – 1.21 (m, 2H), 0.38 (dq, *J* = 12.4, 6.5 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 52.0, 45.7, 33.0, 30.5, 27.9.

HRMS (ESI) m/z $[M-H]^+$ calcd for $C_8H_{20}BNCl$: 176.1379, found: 176.1400.

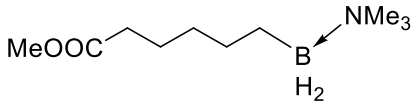


Trimethylamine (3-phenoxypropyl)borane (Figure 2, 3i). The title compound was prepared according to the **GP-1** from (3,3-dibromopropoxy)benzene and trimethylamine borane, purified by flash column chromatography on silica gel: 30% EA in hexane, colorless oil. 1st run: 21.5 mg, 52% yield; 2nd run: 21.1 mg, 51% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.28 – 7.23 (m, 2H), 6.94 – 6.86 (m, 3H), 3.97 (t, *J* = 7.0 Hz, 2H), 2.57 (s, 9H), 1.78 – 1.69 (m, 2H), 0.48 (dq, *J* = 12.3, 6.6 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 159.5, 129.2, 119.9, 114.6, 71.1, 52.0, 28.2.

HRMS (ESI) m/z $[M-H]^+$ calcd for $C_{12}H_{21}BNO$: 206.1718, found: 206.1723.

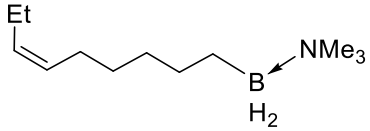


Trimethylamine methyl 6-boraneylhexanoate (Figure 2, 3j). The title compound was prepared according to the **GP-1** from methyl 6,6-dibromohexanoate and trimethylamine borane, purified by flash column chromatography on silica gel: 35% EA in hexane, yellow soild. m.p.:102 – 103 °C. 1st run: 36.3 mg, 63% yield; 2nd run: 36.9 mg, 64% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 3.65 (s, 3H), 2.54 (s, 9H), 2.35 – 2.20 (m, 2H), 1.66 – 1.59 (m, 2H), 1.38 – 1.28 (m, 2H), 1.28 – 1.13 (m, 2H), 0.36 (dq, *J* = 12.3, 6.3 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 174.8, 52.0, 51.3, 34.3, 32.8, 28.2, 25.2.

HRMS (ESI) m/z $[M-H]^+$ calcd for $C_{10}H_{23}BNO_2$: 200.1824, found: 200.1826.

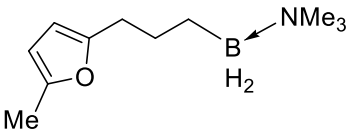


Trimethylamine (Z)-non-6-en-1-ylborane (Figure 2, 3k). The title compound was prepared according to the **GP-1** from (Z)-9,9-dichloronon-3-ene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, colorless oil. 1st run: 17.3 mg, 44% yield; 2nd run: 18.9 mg, 48% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 5.39 – 5.28 (m, 2H), 2.55 (s, 9H), 2.10 – 1.93 (m, 5H), 1.40 – 1.28 (m, 5H), 1.28– 1.17(m, 2H), 0.95 (t, *J* = 7.5 Hz, 3H), 0.37 (dq, *J* = 12.2, 6.3 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 131.1, 129.9, 52.0, 33.2, 30.1, 28.6, 27.3, 20.5, 14.4.

HRMS (ESI) m/z $[M-H]^+$ calcd for $C_{12}H_{27}BN$: 196.2239, found: 196.2236.

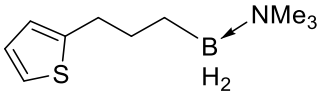


Trimethylamine (3-(5-methylfuran-2-yl)propyl)borane (Figure 2, 3l). The title compound was prepared according to the **GP-1** from 2-(3,3-dichloropropyl)-5-methylfuran and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, yellow oil. 1st run: 19.9 mg, 51% yield; 2nd run: 20.3 mg, 52% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 5.88 – 5.78 (m, 2H), 2.59 (t, *J* = 7.7 Hz, 2H), 2.55 (s, 9H), 2.24 (s, 3H), 1.60 – 1.53 (m, 2H), 0.44 (dq, *J* = 12.0, 6.2 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 156.5, 149.4, 105.6, 104.5, 52.0, 31.5, 27.3, 13.5.

HRMS (ESI) m/z $[M-H]^+$ calcd for $C_{11}H_{21}BNO$: 194.1718, found:197.1717 .

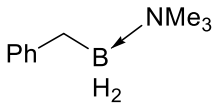


Trimethylamine (3-(thiophen-2-yl)propyl)borane (Figure 2, 3m). The title compound was prepared according to the **GP-1** from 2-(3,3-dichloropropyl)thiophene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, yellow oil. 1st run: 26.4 mg, 67% yield; 2nd run: 26.8 mg, 68% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.06 (dd, *J* = 5.1, 1.2 Hz, 1H), 6.89 (dd, *J* = 5.1, 3.4 Hz, 1H), 6.80 – 6.73 (m, 1H), 2.89 – 2.79 (m, 2H), 2.55 (s, 9H), 1.69 – 1.60 (m, 2H), 0.47 (dq, *J* = 12.1, 6.3 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 147.7, 126.4, 123.4, 122.2, 52.0, 33.4, 31.1.

HRMS (ESI) m/z $[M-H]^+$ calcd for $C_{10}H_{19}BNS$: 196.1333, found:196.1330.

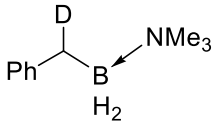


Trimethylamine benzylborane (Figure 2, 3n). The title compound was prepared according to the **GP-2** from (dichloromethyl)benzene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, colorless oil. 1st run: 24.1 mg, 75% yield; 2nd run: 24.0 mg, 74% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.22 – 7.12 (m, 4H), 6.98 (tt, *J* = 5.7, 2.6 Hz, 1H), 2.55 (s, 9H), 1.94 (t, *J* = 5.6 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 148.0, 128.5, 127.8, 122.9, 52.1.

HRMS (ESI) m/z $[M - H]^+$ calcd for $C_{10}H_{17}BN$: 162.1456, found: 162.1455

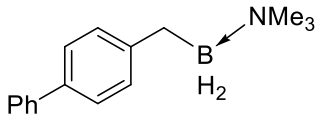


Trimethylamine (phenylmethyl-*d*)borane (Figure 2, 3o). The title compound was prepared according to the **GP-2** from (dichloromethyl-*d*)benzene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, colorless oil. 1st run: 25 mg, 75% yield; 2nd run: 24.2 mg, 74% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.21 – 7.12 (m, 4H), 7.03 – 6.94 (m, 1H), 2.55 (s, 9H), 2.29 – 1.98 (m, 1H), 1.92 (s, 1H), 1.87 – 1.60 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 148.0, 128.5, 127.8, 122.9, 52.1.

HRMS (ESI) m/z [M - H]$^{+}$ calcd for $C_{10}H_{16}$DBN: 163.1519, found: 163.1519

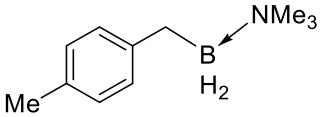


Trimethylamine ([1,1'-biphenyl]-4-ylmethyl)borane (Figure 2, 3p). The title compound was prepared according to the **GP-2** from 4-(dichloromethyl)-1,1'-biphenyl and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, white solid. m.p.:93 – 94 °C.1st run: 23.4 mg, 49% yield; 2nd run: 23.9 mg, 50% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.57 (dt, *J* = 7.7, 1.2 Hz, 2H), 7.46 – 7.36 (m, 4H), 7.30-7.24 (m, 3H), 2.58 (s, 9H), 1.99 (t, *J* = 5.7 Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 147.4, 141.8, 128.8, 128.5, 126.8, 126.6, 126.3, 77.0, 52.1.

HRMS (ESI) m/z [M - H]$^{+}$ calcd for $C_{16}H_{21}$BN: 238.1770, found: 238.1775

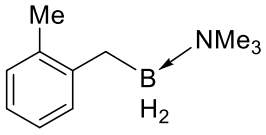


Trimethylamine (4-methylbenzyl)borane (Figure 2, 3q). The title compound was prepared according to the **GP-2** from 1-(dichloromethyl)-4-methylbenzene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, colorless oil. 1st run: 22.6 mg, 64% yield; 2nd run: 23.4 mg, 66% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.08 (d, *J* = 8.0 Hz, 2H), 6.99 (d, *J* = 7.7 Hz, 2H), 2.55 (s, 9H), 2.26 (s, 3H), 1.90 (t, *J* = 5.7 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 144.7, 132.0, 128.5, 128.3, 52.1, 20.9.

HRMS (ESI) m/z [M - H]$^{+}$ calcd for $C_{11}H_{19}$BN: 176.1613, found: 176.1610.



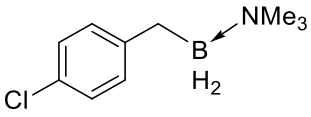
Trimethylamine (2-methylbenzyl)borane (Figure 2, 3r). The title compound was prepared according to the **GP-2** from 1-(dichloromethyl)-2-methylbenzene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, colorless oil. 1st run: 24.4 mg, 69% yield; 2nd run: 24.0 mg, 68% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.10 (dd, *J* = 7.5, 1.4 Hz, 1H), 7.06 – 7.00 (m, 2H), 6.91

(td, *J* = 7.4, 1.5 Hz, 1H), 2.61 (s, 9H), 2.33 (s, 3H), 1.88 (t, *J* = 5.9 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 145.7, 134.9, 129.6, 128.8, 125.4, 123.0, 51.9, 19.8.

HRMS (ESI) m/z [M - H]$^+$ calcd for $C_{11}H_{19}BN$: 176.1613, found: 176.1609.

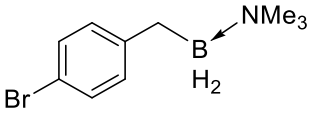


Trimethylamine (4-chlorobenzyl)borane (Figure 2, 3s). The title compound was prepared according to the **GP-2** from 1-chloro-4-(dichloromethyl)benzene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, colorless oil. 1st run: 28.8 mg, 73% yield; 2nd run: 29.6 mg, 75% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.15 – 7.07 (m, 4H), 2.55 (s, 9H), 1.89 (t, *J* = 5.0 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ δ 146.5, 129.8, 128.3, 127.7, 52.1.

HRMS (ESI) m/z [M - H]$^+$ calcd for $C_{10}H_{16}BNCl$: 197.1066, found:197.1066.

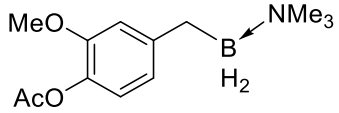


Trimethylamine (4-bromobenzyl)borane (Figure 2, 3t). The title compound was prepared according to the **GP-2** from 1-bromo-4-(dichloromethyl)benzene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, colorless oil. 1st run: 28.0 mg, 58% yield; 2nd run: 28.0 mg, 58% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.27 (d, *J* = 8.5 Hz, 2H, with $CHCl_3$), 7.08 – 7.02 (m, 2H), 2.55 (s, 9H), 1.88 (t, *J* = 5.1 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 147.1, 130.7, 130.3, 116.2, 52.1.

HRMS (ESI) m/z [M - H]$^+$ calcd for $C_{10}H_{16}BNBr$: 240.0561, found:240.0564.

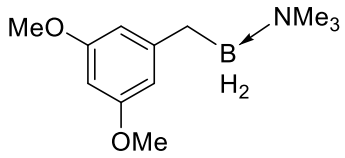


Trimethylamine 4-(boraneylmethyl)-2-methoxyphenyl acetate (Figure 2, 3u). The title compound was prepared according to the **GP-2** from 4-(dichloromethyl)-2-methoxyphenyl acetate and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, grey solid. m.p.:96 – 97 °C. 1st run: 17.6 mg, 35% yield; 2nd run: 16.6 mg, 33% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 6.87 – 6.77 (m, 2H), 6.72 (dd, *J* = 8.1, 1.9 Hz, 1H), 3.80 (s, 3H), 2.55 (s, 9H), 2.28 (s, 3H), 1.93 (t, *J* = 5.2 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ δ 169.5, 150.3, 147.2, 135.7, 121.6, 120.5, 112.7, 55.7, 52.1, 20.7.

HRMS (ESI) m/z [M - H]$^+$ calcd for $C_{13}H_{21}BNO_3$: 250.1617, found:250.1619.

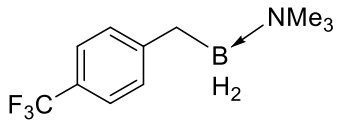


Trimethylamine (3,5-dimethoxybenzyl)borane (Figure 2, 3v). The title compound was prepared according to the **GP-2** from 1-(dichloromethyl)-3,5-dimethoxybenzene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, white solid. m.p.:89 – 90 °C.1st run: 28.6 mg, 64% yield; 2nd run: 29.0 mg, 65% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 6.39 (d, *J* = 2.3 Hz, 2H), 6.14 (s, 1H), 3.76 (s, 6H), 2.54 (s, 9H), 1.91 (t, *J* = 5.4 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 160.3, 150.9, 106.5, 95.5, 55.1, 52.1.

HRMS (ESI) m/z [M + H]$^{+}$ calcd for $C_{12}H_{22}BNO_2$: 224.1824, found:224.1827.



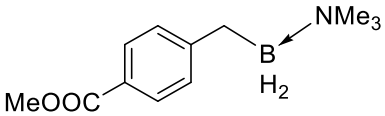
Trimethylamine (4-(trifluoromethyl)benzyl)borane (Figure 2, 3w). The title compound was prepared according to the **GP-2** from 1-(dichloromethyl)-4-(trifluoromethyl)benzene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, yellow oil. 1st run: 12.9 mg, 28% yield; 2nd run: 13.9 mg, 30% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.41 (d, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 8.2 Hz, 2H), 2.58 (s, 9H), 1.98 (brs, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 152.7, 128.5, 125.0 (q, *J* = 32.0 Hz), 124.9 (q, *J* = 271.2 Hz), 124.6 (q, *J* = 3.7 Hz), 52.1.

^{19}F NMR (565 MHz, Chloroform-*d*) δ -61.8.

HRMS (ESI) m/z [M - H]$^{+}$ calcd for $C_{11}H_{16}BF_3N$: 230.1330, found:230.1334.

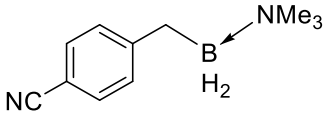


Trimethylamine methyl 4-(boraneylmethyl)benzoate (Figure 2, 3x). The title compound was prepared according to the **GP-2** from methyl 4-(dichloromethyl)benzoate and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, colorless oil. 1st run: 13.3 mg, 30% yield; 2nd run: 13.3 mg, 30% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.85 (d, *J* = 8.3 Hz, 2H), 7.22 (d, *J* = 8.3 Hz, 2H), 3.87 (s, 3H), 2.56 (s, 9H), 2.00 (t, *J* = 4.9 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 167.7, 154.9, 129.3, 128.4, 124.8, 52.1, 51.7.

HRMS (ESI) m/z [M + H]$^{+}$ calcd for $C_{12}H_{21}BNO_2$: 222.1668, found:222.1661.

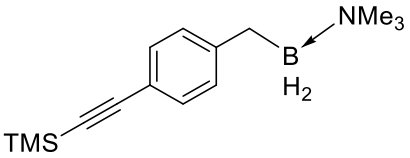


Trimethylamine 4-(boraneylmethyl)benzonitrile (Figure 2, 3y). The title compound was prepared according to the **GP-2** from 4-(dichloromethyl)benzonitrile and trimethylamine borane, purified by flash column chromatography on silica gel: 30% EA in hexane, colorless oil. 1st run: 15.8 mg, 42% yield; 2nd run: 14.3 mg, 38% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.47 – 7.40 (m, 2H), 7.25 – 7.19 (m, 2H), 2.58 (s, 9H), 1.98 (t, *J* = 5.7 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 154.8, 129.1, 120.1, 106.1, 52.1.

HRMS (ESI) m/z [M + H]$^{+}$ calcd for $C_{11}H_{18}BN$: 189.1565, found:189.1564 .

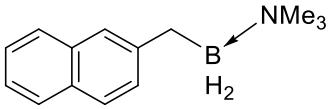


Trimethylamine ((4-(boraneylmethyl)phenyl)ethynyl)trimethylsilane (Figure 2, 3z). The title compound was prepared according to the **GP-2** from ((4-(dichloromethyl)phenyl)ethynyl)trimethylsilane and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, white solid. m.p.:97 – 98 °C. 1st run: 17.6 mg, 34% yield; 2nd run: 18.1 mg, 35% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.29 (d, *J* = 8.2 Hz, 2H), 7.11 (d, *J* = 8.1 Hz, 2H), 2.52 (s, 9H), 1.94 (t, *J* = 5.1 Hz, 4H), 0.23 (s, 9H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 149.7, 131.6, 128.4, 117.2, 106.4, 91.8, 52.1, 0.12.

HRMS (ESI) m/z [M + H]$^{+}$ calcd for $C_{11}H_{27}BNSi$: 260.2009, found:260.2007.

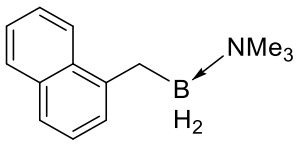


Trimethylamine (naphthalen-2-ylmethyl)borane (Figure 2, 3aa). The title compound was prepared according to the **GP-2** from 2-(dichloromethyl)naphthalene and trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, colorless oil. 1st run: 25.6 mg, 60% yield; 2nd run: 25.2 mg, 59% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.76 – 7.62 (m, 3H), 7.56 (s, 1H), 7.43 (dd, *J* = 8.4, 1.7 Hz, 1H), 7.36 (ddd, *J* = 8.2, 6.8, 1.3 Hz, 1H), 7.30 (ddd, *J* = 8.1, 6.8, 1.3 Hz, 1H), 2.56 (s, 9H), 2.12 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 146.0, 134.0, 130.8, 128.9, 127.4, 127.0, 126.9, 125.1, 125.1, 123.6, 52.2.

HRMS (ESI) m/z [M - H]$^{+}$ calcd for $C_{14}H_{19}BN$: 212.1613, found:212.1620.



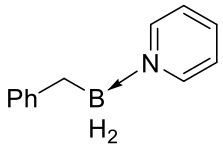
Trimethylamine (naphthalen-1-ylmethyl)borane (Figure 2, 3ab). The title compound was prepared according to the **GP-2** from 1-(dichloromethyl)naphthalene and

trimethylamine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, yellow oil. 1st run: 12.8 mg, 30% yield; 2nd run: 13.2 mg, 31% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.28 – 8.21 (m, 1H), 7.80 – 7.73 (m, 1H), 7.52 (dt, *J* = 7.8, 1.1 Hz, 1H), 7.40 (dddd, *J* = 18.9, 8.0, 6.7, 1.5 Hz, 2H), 7.35 – 7.27 (m, 2H), 2.63 (s, 9H), 2.40 (t, *J* = 5.7 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 144.6, 134.1, 132.3, 128.3, 125.7, 125.6, 125.0, 124.8, 124.3, 123.6, 52.0.

HRMS (ESI) m/z [M + H]$^+$ calcd for $C_{14}H_{21}BN$: 214.1770, found:214.1772.

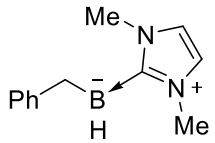


Pyridine benzylborane (Figure 2, 3ac). The title compound was prepared according to the **GP-2** from (dichloromethyl)benzene and pyridine borane, purified by flash column chromatography on silica gel: 20% EA in hexane, white soild. m.p.:74 – 75 °C. 1st run: 7.3 mg, 22% yield; 2nd run: 8.8 mg, 24% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.29 (d, *J* = 5.7 Hz, 2H), 7.88 (t, *J* = 7.7 Hz, 1H), 7.42 – 7.36 (m, 2H), 7.05 (t, *J* = 7.5 Hz, 2H), 6.93 (t, *J* = 7.3 Hz, 1H), 6.84 – 6.71 (m, 2H), 2.08 (t, *J* = 4.8 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 148.3, 146.8, 139.2, 127.8, 127.7, 125.0, 122.7.

HRMS (ESI) m/z [M-H]$^+$ calcd for $C_{12}H_{13}BN$: 182.1141, found: 182.1141.

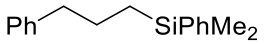


NHC benzylborane complex (Figure 2, 3ad). The title compound was prepared according to the **GP-2** from (dichloromethyl)benzene and (1,3-Dihydro-1,3-dimethyl-2H-imidazol-2-ylidene)trihydroboron, purified by flash column chromatography on silica gel: 10% EA in hexane, white soild. m.p.:92 – 93 °C. 1st run: 28.0 mg, 70% yield; 2nd run: 27.6 mg, 69% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.03 (t, *J* = 7.5 Hz, 2H), 6.89 (t, *J* = 7.3 Hz, 1H), 6.77 (d, *J* = 7.4 Hz, 2H), 6.72 (s, 2H), 3.42 (s, 6H), 1.95 (s, 2H), 1.79 – 1.22 (m, 2H)

^{13}C NMR (126 MHz, Chloroform-*d*) δ 151.8, 128.5, 127.6, 127.3, 127.0, 122.0, 119.9, 35.4, 27.3.

HRMS (ESI) m/z [M-H]$^+$ calcd for $C_{12}H_{16}BN_2$: 199.1409, found: 199.1410.



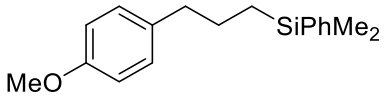
Dimethyl(phenyl)(3-phenylpropyl)silane(Figure 3, 5a).[2] The title compound was prepared according to the **GP-3** from (3,3-dichloropropyl)benzene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 49.3 mg, 97% yield; 2nd run: 49.8 mg, 98% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.51 – 7.47 (m, 2H), 7.34 (dd, *J* = 4.9, 1.9 Hz, 3H), 7.29 – 7.19 (m, 2H), 7.19 – 7.09 (m, 3H), 2.61 (t, *J* = 7.7 Hz, 2H), 1.68 – 1.57 (m, 2H), 0.86 – 0.65 (m, 2H), 0.25 (s, 6H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 142.5, 139.4, 133.5, 128.8, 128.5, 128.2, 127.7, 125.6, 39.8, 26.0, 15.6, -3.1.

Gram-scale reaction: Inside the glovebox under N_2 atmosphere, Fe(TPP)Cl (423.8 mg, 0.602 mmol, 0.10 equiv), Mn (1.0 g, 18.1 mmol, 3.0 equiv) and DMF (30 mL) were added to an oven-dried 100 mL flask equipped with a magnetic stir bar. The reaction mixture was stirred (800 r/min) at room temperature for 0.5 hours. Next, (3,3-dichloropropyl)benzene (1.1 g, 6.0 mmol, 1.0 equiv) and dimethylphenylsilane (1.6 g, 12.0 mmol, 2.0 equiv) were added sequentially, The vial was quickly sealed with a PTFE septum cap. Then the vial was taken out of the glovebox, and stirred (800 r/min) at 25 °C for 24 hours.

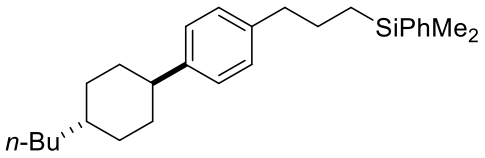
Work-up: The reaction mixture was diluted with 30.0 mL ether,. then the reaction solvent was removed by concentration under reduced pressure. The residue was purified by flash column chromatography on silica gel: hexane, colorless oil. 1.30 g, 83% yield.



(3-(4-methoxyphenyl)propyl)dimethyl(phenyl)silane (Figure 3, 5b).[3] The title compound was prepared according to the **GP-3** from 1-(3,3-dichloropropyl)-4-methoxybenzene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 48.8 mg, 86% yield; 2nd run: 48.3 mg, 85% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.57 – 7.43 (m, 2H), 7.35 (dd, *J* = 4.5, 2.2 Hz, 3H), 7.14 – 6.97 (m, 2H), 6.97 – 6.76 (m, 2H), 3.79 (s, 3H), 2.56 (t, *J* = 7.6 Hz, 2H), 1.72 – 1.58 (m, 2H), 0.84 – 0.73 (m, 2H), 0.26 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 157.7, 139.5, 134.7, 133.6, 129.4, 128.8, 127.7, 113.7, 55.3, 38.9, 26.2, 15.5, -3.0.

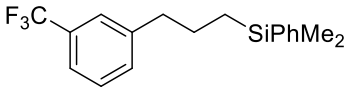


(3-(4-((1s,4r)-4-butylcyclohexyl)phenyl)propyl)dimethyl(phenyl)silane (Figure 3, 5c). The title compound was prepared according to the **GP-3** from ((1s,4r)-4-butylcyclohexyl)-4-(3,3-dichloropropyl)benzene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 59.6 mg, 76% yield; 2nd run: 58.8 mg, 75% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.53 – 7.45 (m, 2H), 7.37 – 7.30 (m, 3H), 7.11 (d, *J* = 8.1 Hz, 2H), 7.07 (d, *J* = 8.2 Hz, 2H), 2.58 (t, *J* = 7.7 Hz, 2H), 2.43 (tt, *J* = 12.2, 3.3 Hz, 1H), 1.96 – 1.78 (m, 4H), 1.69 – 1.59 (m, 2H), 1.44 (qd, *J* = 13.7, 13.2, 3.7 Hz, 2H), 1.37 – 1.19 (m, 7H), 1.05 (tdd, *J* = 12.8, 10.9, 3.7 Hz, 2H), 0.97 – 0.88 (m, 3H), 0.88 – 0.75 (m, 2H), 0.26 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 145.2, 139.9, 139.5, 133.6, 128.8, 128.3, 127.7, 126.6, 44.2, 39.5, 37.3, 37.1, 34.4, 33.7, 29.2, 26.0, 23.0, 15.7, 14.2, -3.0.

GCMS (EI) m/z $[M-CH_3]^+$ calcd for $C_{26}H_{37}Si$: 377.3, found: 377.1.



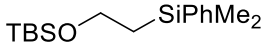
Dimethyl(phenyl)(3-(3-(trifluoromethyl)phenyl)propyl)silane (Figure 3, 5d). The title compound was prepared according to the **GP-3** from 1-(3,3-dichloropropyl)-3-(trifluoromethyl)benzene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 54.8 mg, 85% yield; 2nd run: 55.4 mg, 86% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.51 – 7.46 (m, 2H), 7.45 – 7.41 (m, 1H), 7.39 – 7.33 (m, 5H), 7.32 – 7.28 (m, 1H), 2.66 (t, *J* = 7.6 Hz, 2H), 1.71 – 1.56 (m, 2H), 0.84 – 0.71 (m, 2H), 0.26 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 143.3, 139.1, 133.5, 131.9, 130.6 (q, *J* = 31.7 Hz), 128.9, 128.6, 127.8, 125.1 (q, *J* = 3.8 Hz), 124.3 (q, *J* = 272.2 Hz),122.6 (q, *J* = 3.9 Hz), 39.4, 25.7, 15.5, -3.1F.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -62.5

GCMS (EI) m/z $[M-CH_3]^+$ calcd for $C_{17}H_{18}SiF_3$: 307.1, found: 307.1.



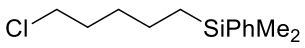
Methyl 6-(dimethyl(phenyl)silyl)hexanoate (Figure 3, 5e). The title compound was prepared according to the **GP-3** from tert-butyl(3,3-dibromopropoxy)dimethylsilane and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, yellow oil. 1st run: 54.7 mg, 93% yield; 2nd run: 54.1 mg, 92% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.54 – 7.45 (m, 2H), 7.39 – 7.30 (m, 3H), 3.55 (t, *J* = 6.9 Hz, 2H), 1.62 – 1.45 (m, 2H), 0.89 (s, 9H), 0.71 (d, *J* = 4.8 Hz, 2H), 0.26 (s, 6H), 0.03 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 139.3, 133.5, 128.8, 127.7, 66.0, 27.2, 18.4, 11.3, -3.1, -5.2.

GCMS (EI) m/z $[M-H]^+$ calcd for $C_{16}H_{29}OSi_2$: 293.2, found: 293.1

.

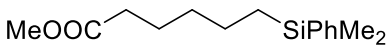


(5-chloropentyl)dimethyl(phenyl)silane (Figure 3, 5f). The title compound was prepared according to the **GP-3** from 1,1-dibromo-5-chloropentane and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 39.4 mg, 82% yield; 2nd run: 39.5 mg, 82% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.52 – 7.46 (m, 2H), 7.39 – 7.31 (m, 3H), 3.50 (t, *J* = 6.8 Hz, 2H), 1.75 (dq, *J* = 9.0, 6.9 Hz, 2H), 1.48 – 1.40 (m, 2H), 1.40 – 1.30 (m, 2H), 0.80 – 0.72 (m, 2H), 0.26 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 139.4, 133.5, 128.8, 127.7, 45.1, 32.3, 30.7, 23.2, 15.6, -3.1.

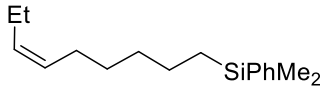
GCMS (EI) m/z $[M]^+$ calcd for $C_{13}H_{21}SiCl$: 225.1, found: 225.2.



Methyl 6-(dimethyl(phenyl)silyl)hexanoate (Figure 3, 5g).[4] The title compound was prepared according to the **GP-3** from methyl 6,6-dibromohexanoate and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, yellow oil. 1st run: 33.8mg, 64% yield; 2nd run: 33.3 mg, 63% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.54 – 7.45 (m, 2H), 7.37 – 7.31 (m, 3H), 3.65 (s, 3H), 2.27 (t, *J* = 7.6 Hz, 2H), 1.65 – 1.55 (m, 3H), 1.39 – 1.26 (m, 4H), 0.78 – 0.70 (m, 2H), 0.25 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 174.3, 139.5, 133.5, 128.8, 127.7, 51.4, 34.0, 33.0, 24.6, 23.5, 15.5, -3.1.

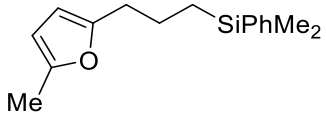


(Z)-dimethyl(non-6-en-1-yl)(phenyl)silane (Figure 3, 5h). The title compound was prepared according to the **GP-3** from 1-(3,3-dichloropropyl)-4-methoxybenzene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 26.7 mg, 51% yield; 2nd run: 26.9 mg, 51% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.54 – 7.47 (m, 2H), 7.37 – 7.31 (m, 3H), 5.42 – 5.17 (m, 2H), 2.08 – 1.94 (m, 4H), 1.32 (s, 6H), 0.95 (t, *J* = 7.5 Hz, 3H), 0.80 – 0.68 (m, 2H), 0.25 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 139.7, 133.5, 131.5, 129.3, 128.7, 127.7, 33.1, 29.3, 27.0, 23.7, 20.5, 15.7, 14.4, -3.0.

GCMS (EI) m/z $[M]^+$ calcd for $C_{17}H_{28}Si$: 260.2, found: 260.2.

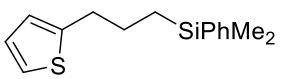


Dimethyl(3-(5-methylfuran-2-yl)propyl)(phenyl)silane (Figure 3, 5i). The title compound was prepared according to the **GP-3** from 2-(3,3-dichloropropyl)-5-methylfuran and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 40.8 mg, 79% yield; 2nd run: 42.9 mg, 83% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.56 – 7.46 (m, 2H), 7.37 – 7.30 (m, 3H), 5.84 – 5.79 (m, 2H), 2.57 (t, *J* = 7.5 Hz, 2H), 2.24 (s, 3H), 1.69 – 1.60 (m, 2H), 0.84 – 0.68 (m, 2H), 0.27 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 154.4, 150.1, 139.3, 133.5, 128.8, 127.7, 105.7, 105.4, 31.8, 22.8, 15.5, 13.5, -3.1.

GCMS (EI) m/z $[M]^+$ calcd for $C_{16}H_{22}SiO$: 258.1, found: 258.2.

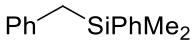


Dimethyl(phenyl)(3-(thiophen-2-yl)propyl)silane (Figure 3, 5j). The title compound was prepared according to the **GP-3** from 2-(3,3-dichloropropyl)thiophene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 40.6 mg, 78% yield; 2nd run: 41.1 mg, 79% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.54 – 7.42 (m, 2H), 7.38 – 7.26 (m, 3H), 7.10 (dd, *J* = 5.1, 1.2 Hz, 1H), 6.91 (dd, *J* = 5.1, 3.4 Hz, 1H), 6.75 (dq, *J* = 3.3, 1.0 Hz, 1H), 2.83 (td, *J* = 7.4, 0.9 Hz, 2H), 1.76 – 1.62 (m, 2H), 0.91 – 0.75 (m, 2H), 0.27 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 145.4, 139.2, 133.5, 128.8, 127.7, 126.6, 124.1, 122.8, 33.6, 26.4, 15.4, -3.1.

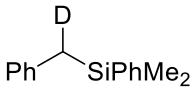
GCMS (EI) m/z $[M]^+$ calcd for $C_{15}H_{20}SiS$: 260.1, found: 260.2



Dimethyl(phenyl)(phenylmethyl-d)silane (Figure 3, 5k).[5] The title compound was prepared according to the **GP-3** from (dichloromethyl)benzene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 38.0 mg, 84% yield; 2nd run: 38.4 mg, 85% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.48 – 7.44 (m, 2H), 7.42 – 7.30 (m, 3H), 7.17 (t, *J* = 7.6 Hz, 2H), 7.09 – 7.04 (m, 1H), 6.97 – 6.90 (m, 2H), 2.30 (s, 2H), 0.24 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 139.7, 138.5, 133.7, 129.1, 128.3, 128.1, 127.7, 124.1, 26.2, -3.5.

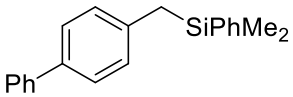


Dimethyl(phenyl)(phenylmethyl-d)silane (Figure 3, 5l). The title compound was prepared according to the **GP-3** from (dichloromethyl-*d*)benzene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 25.9 mg, 57% yield; 2nd run: 25.4 mg, 56% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.49 – 7.44 (m, 2H), 7.39 – 7.32 (m, 3H), 7.18 (dd, *J* = 8.3, 6.8 Hz, 2H), 7.10 – 7.03 (m, 1H), 6.96 – 6.88 (m, 2H), 2.29 (s, 1H), 0.25 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 139.6, 138.5, 133.7, 129.0, 128.3, 128.1, 127.7, 124.0, 25.8(t, *J* = 18.9 Hz), -3.5.

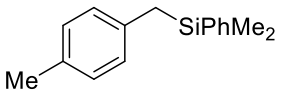
GCMS (EI) m/z $[M]^+$ calcd for $C_{15}H_{17}DSi$: 227.1, found: 227.2.



([1,1'-biphenyl]-4-ylmethyl)dimethyl(phenyl)silane (Figure 3, 5m).[6] The title compound was prepared according to the **GP-3** from 4-(dichloromethyl)-1,1'-biphenyl and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 36.9 mg, 61% yield; 2nd run: 34.4 mg, 57% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.61 – 7.54 (m, 2H), 7.51 – 7.46 (m, 2H), 7.45 – 7.28 (m, 8H), 7.03 – 6.97 (m, 2H), 2.34 (s, 2H), 0.28 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 141.1, 138.9, 138.4, 136.9, 133.7, 129.1, 128.7, 128.7, 127.7, 126.8, 25.9, -3.4.

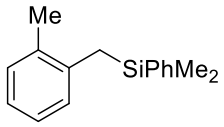


Dimethyl(4-methylbenzyl)(phenyl)silane (Figure 3, 5n).[7] The title compound was

prepared according to the **GP-3** from 1-(dichloromethyl)-4-methylbenzene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 29.8 mg, 62% yield; 2nd run: 30.3 mg, 63% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.49 – 7.45 (m, 2H), 7.40 – 7.32 (m, 3H), 7.03 – 6.94 (m, 2H), 6.86 – 6.79 (m, 2H), 2.29 (s, 3H), 2.27 (s, 2H), 0.24 (s, 6H).

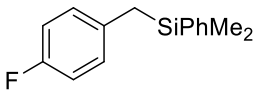
^{13}C NMR (126 MHz, Chloroform-*d*) δ 138.7, 136.3, 133.7, 133.4, 129.0, 128.8, 128.2, 127.7, 25.5, 20.9, -3.4.



Dimethyl(2-methylbenzyl)(phenyl)silane (Figure 3, 5o).[7] The title compound was prepared according to the **GP-3** from 1-(dichloromethyl)-2-methylbenzene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 28.9 mg, 60% yield; 2nd run: 26.9 mg, 56% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.48 – 7.42 (m, 2H), 7.39 – 7.31 (m, 3H), 7.22 – 7.19 (m, 1H), 7.09 – 6.96 (m, 3H), 6.90 (dd, *J* = 7.5, 1.5 Hz, 1H), 2.31 (s, 2H), 2.07 (s, 3H), 0.27 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 138.8, 138.1, 134.9, 133.6, 130.1, 129.0, 128.9, 127.7, 125.6, 124.2, 23.0, 20.2, -3.1.

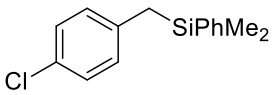


(4-fluorobenzyl)dimethyl(phenyl)silane (Figure 3, 5p).[7] The title compound was prepared according to the **GP-3** from 1-(dichloromethyl)-4-fluorobenzene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 42.0 mg, 86% yield; 2nd run: 41.5 mg, 85% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.47 – 7.41 (m, 2H), 7.40 – 7.31 (m, 3H), 6.89 – 6.81 (m, 4H), 2.26 (s, 2H), 0.25 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 160.4 (d, *J* = 241.4 Hz), 138.1, 135.1 (d, *J* = 3.1 Hz), 133.7, 129.3 (d, *J* = 7.5 Hz), 129.1, 127.8, 114.8 (d, *J* = 21.0 Hz), 25.2, -3.6.

^{19}F NMR (565 MHz, Chloroform-*d*) δ -120.2.

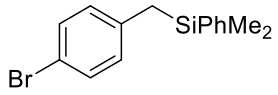


(4-chlorobenzyl)dimethyl(phenyl)silane (Figure 3, 5q). The title compound was prepared according to the **GP-3** from 1-chloro-4-(dichloromethyl)benzene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 29.1 mg, 56% yield; 2nd run: 29.1 mg, 56% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.46 – 7.31 (m, 5H), 7.16 – 7.11 (m, 2H), 6.86 – 6.81 (m, 2H), 2.27 (s, 2H), 0.25 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 138.2, 137.9, 133.7, 129.7, 129.5, 129.2, 128.1, 127.8, 25.7, -3.6.

GCMS (EI) m/z $[M]^+$ calcd for $C_{15}H_{17}ClSi$: 260.1 found: 260.1.

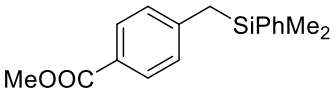


(4-bromobenzyl)dimethyl(phenyl)silane (Figure 3, 5r). The title compound was prepared according to the **GP-3** from 1-bromo-4-(dichloromethyl)benzene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 42.0 mg, 69% yield; 2nd run: 42.6 mg, 70% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.46 – 7.32 (m, 5H), 7.28 (d, *J* = 8.4 Hz, 2H), 6.78 (dt, *J* = 8.6, 0.5 Hz, 2H), 2.25 (s, 2H), 0.25 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 138.8, 137.9, 133.7, 131.1, 129.9, 129.2, 127.8, 117.6, 25.8, -3.6.

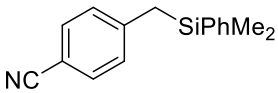
GCMS (EI) m/z $[M]^+$ calcd for $C_{15}H_{17}BrSi$: 304.0, found: 304.1.



Methyl 4-((dimethyl(phenyl)silyl)methyl)benzoate (Figure 3, 5s).[7] The title compound was prepared according to the **GP-3** from methyl 4-(dichloromethyl)benzoate and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 28.4 mg, 50% yield; 2nd run: 28.4 mg, 50% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.85 (d, *J* = 8.3 Hz, 2H), 7.45 – 7.41 (m, 2H), 7.41 – 7.32 (m, 3H), 6.97 (d, *J* = 8.3 Hz, 2H), 3.89 (s, 3H), 2.38 (s, 2H), 0.26 (s, 6H).

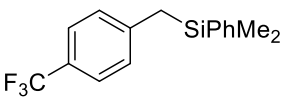
^{13}C NMR (126 MHz, Chloroform-*d*) δ 167.3, 146.0, 137.7, 133.7, 129.5, 129.3, 128.2, 127.8, 126.1, 51.9, 27.2, -3.5.



4-((dimethyl(phenyl)silyl)methyl)benzonitrile (Figure 3, 5t).[7] The title compound was prepared according to the **GP-3** from 4-(dichloromethyl)benzonitrile and dimethylphenylsilane, purified by flash column chromatography on silica gel: 5% EA in hexane, colorless oil. 1st run: 9.5 mg, 19% yield; 2nd run: 10.0 mg, 20% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.47 – 7.30 (m, 7H), 6.98 – 6.91 (m, 2H), 2.38 (s, 2H), 0.27 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 146.3, 137.1, 133.6, 131.9, 129.5, 128.8, 127.9, 119.4, 107.7, 27.7, -3.6.



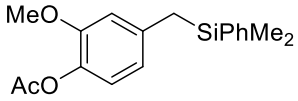
Dimethyl(phenyl)(4-(trifluoromethyl)benzyl)silane (Figure 3, 5u). The title compound was prepared according to the **GP-3** from 1-(dichloromethyl)-4-(trifluoromethyl)benzene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 20.0 mg, 34% yield; 2nd run: 19.4 mg, 33% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.46 – 7.30 (m, 7H), 6.99 (d, *J* = 8.0 Hz, 2H), 2.36 (s, 2H), 0.26 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 144.3, 137.6, 133.7, 129.3, 128.3, 127.9, 126.4 (q, *J* = 32.5 Hz), 125.0 (q, *J* = 3.8 Hz), 124.6 (q, *J* = 272.2 Hz)26.7, -3.6.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -62.0

GCMS (EI) m/z [M]$^{+}$ calcd for $C_{16}H_{17}SiF_3$: 294.1, found: 294.0.

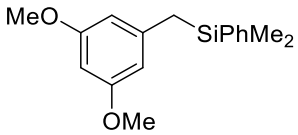


4-((dimethyl(phenyl)silyl)methyl)-2-methoxyphenyl acetate (Figure 3, 5v). The title compound was prepared according to the **GP-3** from 4-(dichloromethyl)-2-methoxyphenyl acetate and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, white soild. m.p.:82 – 83 °C. 1st run: 35.2 mg, 56% yield; 2nd run: 34.6 mg, 55% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.47 – 7.41 (m, 2H), 7.38 – 7.31 (m, 3H), 6.83 (d, *J* = 8.1 Hz, 1H), 6.51 (dd, *J* = 8.1, 2.0 Hz, 1H), 6.37 (d, *J* = 1.9 Hz, 1H), 3.61 (s, 3H), 2.28 (s, 3H), 2.27 (s, 2H), 0.27 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 169.3, 150.4, 138.6, 138.1, 136.5, 133.8, 129.1, 127.7, 122.2, 120.2, 112.5, 55.5, 26.3, 20.7, -3.5.

HRMS (APCI) m/z [M+H]$^{+}$ calcd for $C_{18}H_{23}SiO_3$: 315.1416, found: 315.1415.

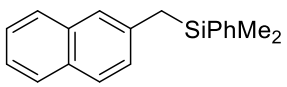


(3,5-dimethoxybenzyl)dimethyl(phenyl)silane (Figure 3, 5w) The title compound was prepared according to the **GP-3** from 1-(dichloromethyl)-3,5-dimethoxybenzene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 43.5 mg, 76% yield; 2nd run: 42.9 mg, 75% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.51 – 7.44 (m, 2H), 7.40 – 7.32 (m, 3H), 6.20 (t, *J* = 2.3 Hz, 1H), 6.08 (d, *J* = 2.3 Hz, 2H), 3.68 (s, 6H), 2.26 (s, 2H), 0.28 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 160.4, 142.0, 133.7, 129.0, 127.7, 106.3, 96.5, 55.1, 26.7, -3.4.

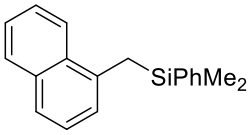
HRMS (APCI) m/z [M+H]$^{+}$ calcd for $C_{17}H_{23}SiO_2$: 287.1467, found: 287.1478.



Dimethyl(naphthalen-2-ylmethyl)(phenyl)silane (Figure3, 5x).[7] The title compound was prepared according to the **GP-3** from 2-(dichloromethyl)naphthalene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, white soild. m.p.:84 – 85 °C. 1st run: 34.8 mg, 63% yield; 2nd run: 34.2 mg, 62% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.76 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.69 – 7.63 (m, 2H), 7.50 – 7.46 (m, 2H), 7.44 – 7.32 (m, 6H), 7.08 (dd, *J* = 8.4, 1.8 Hz, 1H), 2.47 (s, 2H), 0.28 (s, 6H).

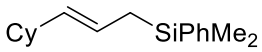
^{13}C NMR (126 MHz, Chloroform-*d*) δ 138.4, 137.4, 133.7, 131.1, 129.1, 128.0, 127.8, 127.5, 127.5, 127.0, 125.7, 125.6, 124.4, 26.5, -3.4.



Dimethyl(naphthalen-1-ylmethyl)(phenyl)silane (Figure 3, 5y).[7] The title compound was prepared according to the **GP-3** from 1-(dichloromethyl)naphthalene and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, white soild. m.p.:77 – 78 °C. 1st run: 16.6 mg, 30% yield; 2nd run: 17.1 mg, 31% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.90 – 7.76 (m, 2H), 7.61 (d, *J* = 8.2 Hz, 1H), 7.52 – 7.45 (m, 2H), 7.45 – 7.30 (m, 6H), 7.13 – 7.08 (m, 1H), 2.79 (s, 2H), 0.21 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 138.7, 136.4, 133.9, 133.6, 131.7, 129.1, 128.5, 127.8, 125.7, 125.4, 125.3, 125.0, 124.9, 124.7, 22.7, -2.9.

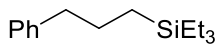


(*E*)-(3-cyclohexylallyl)dimethyl(phenyl)silane (Figure 3, 5z). The title compound was prepared according to the **GP-3** from the mixture of (*E*)-(3,3-dichloroprop-1-en-1-yl)cyclohexane and (1,3-dichloroallyl)cyclohexane (in 1:10 ratio), and dimethylphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 31.5 mg, 61% yield; 2nd run: 32.0 mg, 62% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.55 – 7.45 (m, 2H), 7.36 – 7.27 (m, 3H), 5.38 – 5.28 (m, 1H), 1.92 – 1.84 (m, 1H), 1.72 – 1.57 (m, 7H), 1.29 – 1.18 (m, 2H), 1.17 – 1.08 (m, 1H), 1.07 – 0.96 (m, 2H),0.25 (s, 6H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 139.1, 136.0, 133.7, 128.9, 127.7, 122.7, 41.0, 33.5, 26.3, 26.1, 21.6, -3.4.

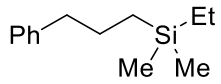
GCMS (EI) m/z $[M]^+$ calcd for $C_{17}H_{26}Si$: 258.2, found: 258.2.



Triethyl(3-phenylpropyl)silane Figure 3, 5ab).[6] The title compound was prepared according to the **GP-3** from (3,3-dichloropropyl)benzene and triethylsilane, purified by flash column chromatography on silica gel: hexane, yellow oil. 1st run: 45.9 mg, 98% yield; 2nd run: 45.4 mg, 97% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.31 – 7.25 (m, 2H), 7.21 – 7.13 (m, 3H), 2.62 (t, *J* = 7.7 Hz, 2H), 1.65 – 1.58 (m, 2H), 0.91 (t, *J* = 7.9 Hz, 9H), 0.59 – 0.54 (m, 2H), 0.50 (q, *J* = 7.9 Hz, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 142.8, 128.5, 128.2, 125.6, 40.3, 26.1, 11.3, 7.5, 3.3.

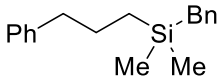


Ethyldimethyl(3-phenylpropyl)silane (Figure 3, 5ac).[8] The title compound was prepared

according to the **GP-3** from (3,3-dichloropropyl)benzene and ethyldimethylsilane, purified by flash column chromatography on silica gel: hexane, yellow oil. 1st run: 35.9 mg, 87% yield; 2nd run: 35.5 mg, 86% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.31 – 7.26 (m, 2H), 7.21 – 7.14 (m, 3H), 2.62 (t, *J* = 7.7 Hz, 2H), 1.66 – 1.56 (m, 2H), 0.90 (t, *J* = 7.9 Hz, 3H), 0.59 – 0.52 (m, 2H), 0.47 (q, *J* = 7.9 Hz, 2H), -0.06 (s, 6H).

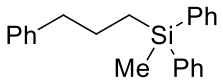
^{13}C NMR (126 MHz, Chloroform-*d*) δ 142.8, 128.5, 128.2, 125.6, 40.0, 26.1, 14.8, 7.4, 6.9, -3.9.



Benzyldimethyl(3-phenylpropyl)silane (Figure 3, 5ad).[9] The title compound was prepared according to the **GP-3** from (3,3-dichloropropyl)benzene and benzyldimethylsilane, purified by flash column chromatography on silica gel: hexane, yellow oil. 1st run: 52.6 mg, 98% yield; 2nd run: 53.1 mg, 99% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.30 – 7.26 (m, 2H), 7.21 – 7.11 (m, 5H), 7.10 – 7.02 (m, 1H), 6.98 – 6.92 (m, 2H), 2.60 (t, *J* = 7.6 Hz, 2H), 2.06 (s, 2H), 1.65 – 1.56 (m, 2H), 0.60 – 0.50 (m, 2H), -0.05 (s, 6H).

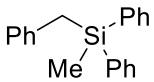
^{13}C NMR (126 MHz, Chloroform-*d*) δ 142.6, 140.3, 128.5, 128.2, 128.1, 128.0, 125.7, 123.9, 39.9, 26.0, 25.5, 14.6, -3.6.



Methyldiphenyl(3-phenylpropyl)silane (Figure 3, 5ae).[10] The title compound was prepared according to the **GP-3** from (3,3-dichloropropyl)benzene and methyldiphenylsilane, purified by flash column chromatography on silica gel: hexane, yellow oil. 1st run: 26.6 mg, 42% yield; 2nd run: 25.9 mg, 41% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.51 – 7.43 (m, 4H), 7.39 – 7.30 (m, 5H), 7.28 – 7.23 (m, 3H), 7.19 – 7.14 (m, 1H), 7.14 – 7.09 (m, 2H), 2.64 (t, *J* = 7.6 Hz, 2H), 1.76 – 1.63 (m, 2H), 1.19 – 1.07 (m, 2H), 0.53 (s, 3H).

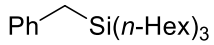
^{13}C NMR (126 MHz, Chloroform-*d*) δ 142.3, 137.2, 134.5, 129.1, 128.5, 128.2, 127.8, 125.7, 39.7, 25.8, 13.9, -4.5.



Benzyl(methyl)diphenylsilane (Figure 3, 5af).[5] The title compound was prepared according to the **GP-3** from (dichloromethyl)benzene and methyldiphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 39.2 mg, 68% yield; 2nd run: 38.6 mg, 67% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.50 – 7.44 (m, 4H), 7.41 – 7.30 (m, 6H), 7.15 – 7.10 (m, 2H), 7.08 – 7.02 (m, 1H), 6.92 – 6.85 (m, 2H), 2.63 (s, 2H), 0.48 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 138.9, 136.4, 134.7, 129.3, 128.7, 128.1, 127.8, 124.3, 24.5, -4.8.

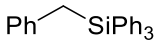


Benzyltrihexylsilane (Figure 3, 5ag). The title compound was prepared according to the **GP-3** from (dichloromethyl)benzene and trihexylsilane, purified by flash column chromatography on silica gel: hexane, yellow oil. 1st run: 71.1 mg, 95% yield; 2nd run: 71.9 mg, 96% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.20 (t, *J* = 7.7 Hz, 2H), 7.08 – 7.02 (m, 1H), 7.02 – 6.97 (m, 2H), 2.08 (s, 2H), 1.31 – 1.21 (m, 24H), 0.89 (t, *J* = 6.9 Hz, 9H), 0.52 – 0.44 (m, 6H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 140.8, 128.1, 123.7, 77.0, 33.5, 31.5, 23.7, 22.7, 22.6, 14.1, 12.0.

GCMS (EI) m/z [M]$^+$ calcd for $C_{25}H_{46}Si$: 374.3 found: 374.4

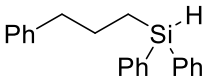


Benzyltriphenylsilane (Figure 3, 5ah). The title compound was prepared according to the **GP-3** from (dichloromethyl)benzene and triphenylsilane, purified by flash column chromatography on silica gel: hexane, white soild. m.p.:105 – 106 °C. 1st run: 41.3 mg, 59% yield; 2nd run: 42.0 mg, 60% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.45 – 7.37 (m, 9H), 7.36 – 7.31 (m, 6H), 7.09 – 7.00 (m, 3H), 6.89 – 6.80 (m, 2H), 2.93 (s, 2H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 138.2, 136.0, 134.2, 129.5, 129.2, 128.0, 127.7, 124.4, 23.4

GCMS (EI) m/z [M]$^+$ calcd for $C_{14}H_{16}Si$: 350.1, found: 350.2.

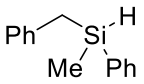


Diphenyl(3-phenylpropyl)silane (Figure 3, 5ai). The title compound was prepared according to the **GP-3** from (3,3-dichloropropyl)benzene and diphenylsilane, purified by flash column chromatography on silica gel: hexane, yellow oil. 1st run: 26.6 mg, 44% yield; 2nd run: 26.0 mg, 43% yield.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.55 – 7.51 (m, 4H), 7.41 – 7.36 (m, 2H), 7.37 – 7.31 (m, 5H), 7.28 – 7.23 (m, 3H), 7.18 – 7.15 (m, 1H), 7.14 – 7.10 (m, 2H), 4.86 (t, *J* = 3.7 Hz, 1H), 2.67 (t, *J* = 7.6 Hz, 2H), 1.78 (tt, *J* = 8.7, 6.8 Hz, 2H), 1.18 (dt, *J* = 11.8, 3.8 Hz, 2H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 142.1, 135.1, 134.3, 129.5, 128.5, 128.2, 128.0, 125.7, 39.2, 26.3, 11.8.

GCMS (EI) m/z [M]$^+$ calcd for $C_{21}H_{22}Si$: 302.1, found: 302.2

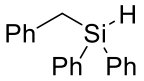


Benzyl(methyl)(phenyl)silane (Figure 3, 5aj). The title compound was prepared according to the **GP-3** from (dichloromethyl)benzene and methyl(phenyl)silane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 14.8 mg, 35% yield; 2nd run: 14.4 mg, 34% yield.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.50 – 7.44 (m, 2H), 7.41 – 7.32 (m, 3H), 7.23 – 7.17 (m, 2H), 7.13 – 7.04 (m, 1H), 7.04 – 6.97 (m, 2H), 4.53 – 4.29 (m, 1H), 2.43 (dd, *J* = 13.9, 3.0 Hz, 1H), 2.33 (dd, *J* = 13.9, 4.1 Hz, 1H), 0.31 (d, *J* = 3.7 Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 139.2, 135.5, 134.4, 129.5, 128.4, 128.3, 127.8, 124.4, 23.5, -6.2.

GCMS (EI) m/z $[M]^+$ calcd for $C_{14}H_{16}Si$: 212.1, found: 212.2.

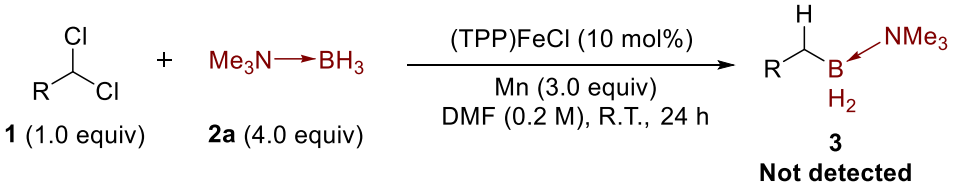


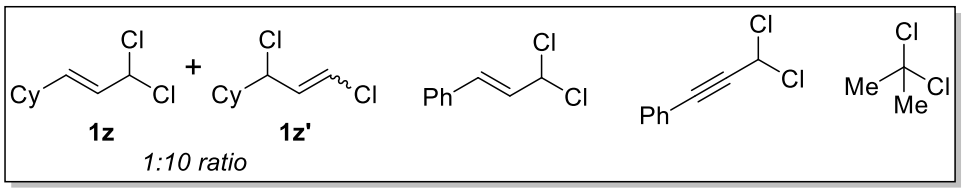
Benzyldiphenylsilane (Figure 3, 5ak).[11] The title compound was prepared according to the **GP-3** from (dichloromethyl)benzene and diphenylsilane, purified by flash column chromatography on silica gel: hexane, colorless oil. 1st run: 49.3 mg, 90% yield; 2nd run: 48.8 mg, 89% yield.

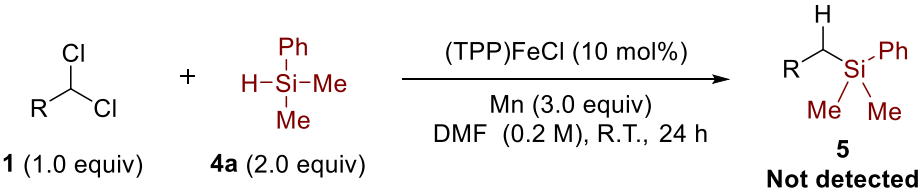
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.51 – 7.47 (m, 4H), 7.42 – 7.37 (m, 2H), 7.34 (dd, *J* = 7.9, 6.6 Hz, 4H), 7.16 (t, *J* = 7.6 Hz, 2H), 7.10 – 7.04 (m, 1H), 7.02 – 6.97 (m, 2H), 4.94 (t, *J* = 3.8 Hz, 1H), 2.68 (d, *J* = 3.8 Hz, 2H).

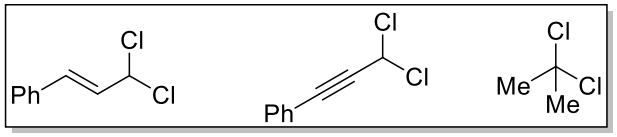
^{13}C NMR (151 MHz, Chloroform-*d*) δ 138.6, 135.3, 133.6, 129.7, 128.7, 128.3, 128.0, 124.6, 22.3.

Unsuccessful substrates for B–H or Si–H insertions:



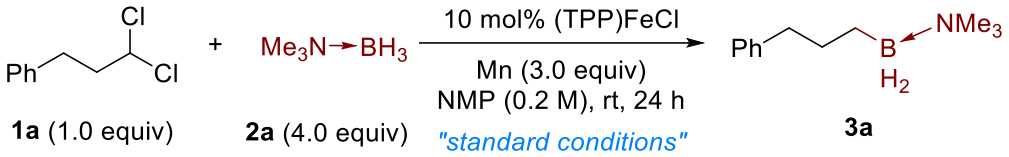






III. Effect of Reaction Parameters

Reactions in Table S1 were conducted based on the following procedure:

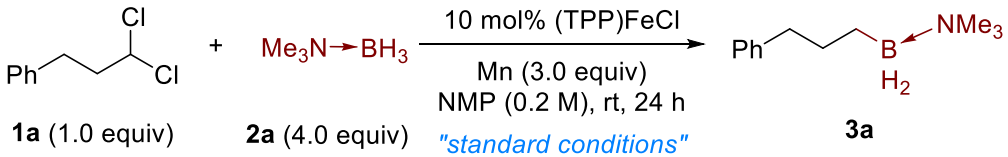


1a (1.0 equiv) **2a** (4.0 equiv) **3a**

Inside the glovebox under N_2 atmosphere, (TPP)FeCl (7.1 mg, 0.010 mmol, 0.10 equiv), Mn (16.6 mg, 0.3 mmol, 3.0 equiv) and *N*-Methylpyrrolidone (NMP) (0.5 mL) were added to an oven-dried 4 mL vial equipped with a magnetic stir bar. The reaction mixture was stirred (800 r/min) at room temperature for 0.5 hours. Next, (3,3-dichloropropyl)benzene (18.8 mg, 0.1 mmol, 1.0 equiv) and trimethylamine borane (29.2 mg, 0.40 mmol, 4.0 equiv) were added sequentially, The vial was quickly sealed with a PTFE septum cap. Then the vial was taken out of the glovebox, and stirred (800 r/min) at 25 °C for 24 hours.

Work-up: The reaction mixture was diluted with 0.5 mL ether, followed by the addition of the internal standard 1,3,5-trimethoxybenzene (0.10 mmol). The reaction solvent was removed by concentration under reduced pressure. The residue was subjected to flash column chromatography with petroleum ether (PE) and ethyl acetate (EA) as the eluent to afford the desired borane products.

Table S1. Effect of reaction parameters of B-H insertion[a]

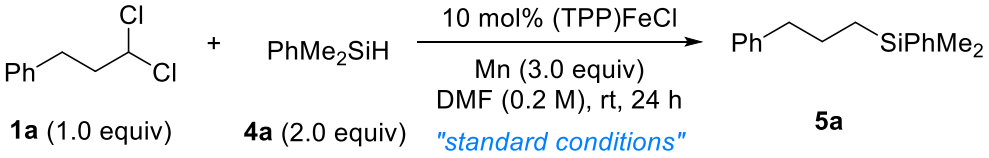


1a (1.0 equiv) **2a** (4.0 equiv) **3a**

entry	variations from *"standard condition"*	yield (%)[b]
1	none	70
2	no Fe(TPP)Cl	<2
3	no Mn	<2
4	Fe(*p*-OMeTPP)Cl, instead of Fe(TPP)Cl	68
5	Fe(*p*-ClTPP)Cl, instead of Fe(TPP)Cl	70
6	Fe(PC), instead of Fe(TPP)Cl	64
7	$FeCl_2$ or $FeBr_3$, instead of Fe(TPP)Cl	<2
8	Co(TPP), instead of Fe(TPP)Cl	15
9	Co(PC), instead of Fe(TPP)Cl	10
10	3.0 equiv Zn, instead of Mn	61
11	3.0 equiv Mg, instead of Mn	12
12	DMF, instead of NMP	56
13	DMAc, instead of NMP	39
14	THF or EA, instead of NMP	<2
15	2.0, instead of 3.0, equiv BH_3NMe_3	46
16	20 mol%, instead of 10 mol%, Fe(TPP)Cl	71
17	with 1.0 equiv DMAP	<2
18	with 1.0 equiv Et_3N	64

[a]Reaction conducted under N_2 on 0.10 mmol scale for 24 h, and all data are the average of two experiments.
[b]Yields were determined via ^1H NMR analysis with 1,3,5-trimethoxybenzene as the internal standard.

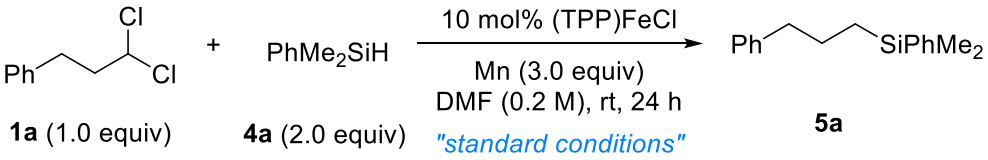
Reactions in Table S2 were conducted based on the following procedure:



Inside the glovebox under N_2 atmosphere, Fe(TPP)Cl (7.1 mg, 0.010 mmol, 0.10 equiv), Mn (16.5 mg, 0.3 mmol, 3.0 equiv) and *N*,*N*-dimethylformamide (DMF) (0.5 mL) were added to an oven-dried 4 mL vial equipped with a magnetic stir bar. The reaction mixture was stirred (800 r/min) at room temperature for 0.5 hours. Next, (3,3-dichloropropyl)benzene (18.8 mg, 0.1 mmol, 1.0 equiv) and dimethylphenyl silane (27.2 mg, 0.20 mmol, 2.0 equiv) were added sequentially, The vial was quickly sealed with a PTFE septum cap. Then the vial was taken out of the glovebox, and stirred (800 r/min) at 25 °C for 24 hours.

Work-up: The reaction mixture was diluted with 1.0 mL ether, followed by the addition of the internal standard 1,3,5-trimethoxybenzene (0.10 mmol). The reaction solvent was removed by concentration under reduced pressure. The residue was subjected to flash column chromatography with petroleum ether (PE) and ethyl acetate (EA) as the eluent to afford the desired silane products.

Table S2. Effect of reaction parameters of Si-H insertion[a]



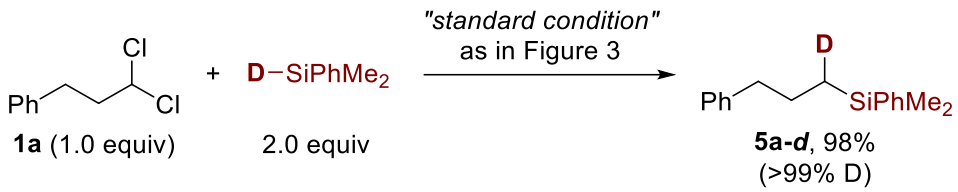
1a (1.0 equiv) **4a** (2.0 equiv) **5a**

entry	variations from *"standard condition"*	yield (%)[b]
1	none	70
2	no Fe(TPP)Cl	<2
3	no Mn	<2
4	Fe(*p*-OMeTPP)Cl, instead of Fe(TPP)Cl	93
5	Fe(*p*-ClTPP)Cl, instead of Fe(TPP)Cl	63
6	Co(TPP), instead of Fe(TPP)Cl	<2
7	Co(PC), instead of Fe(TPP)Cl	<2
8	3.0 equiv Zn, instead of Mn	26
9	3.0 equiv Mg, instead of Mn	<2
10	DMAc, instead of DMF	95
11	MeCN, instead of NMP	<2
12	THF or EA, instead of NMP	<2
13	Reaction at 0 ^{o}C	47
14	Reaction at 60 ^{o}C	67
15	(3,3-dibromopropyl)benzene instead of **1a**	80

[a]Reaction conducted under N_2 on 0.10 mmol scale for 24 h, and all data are the average of two experiments.
[b]Yields were determined via ^{1}H NMR analysis with 1,3,5-trimethoxybenzene as the internal standard.

IV. Preliminary Mechanistic Study

Deuterium-labelling experiment:

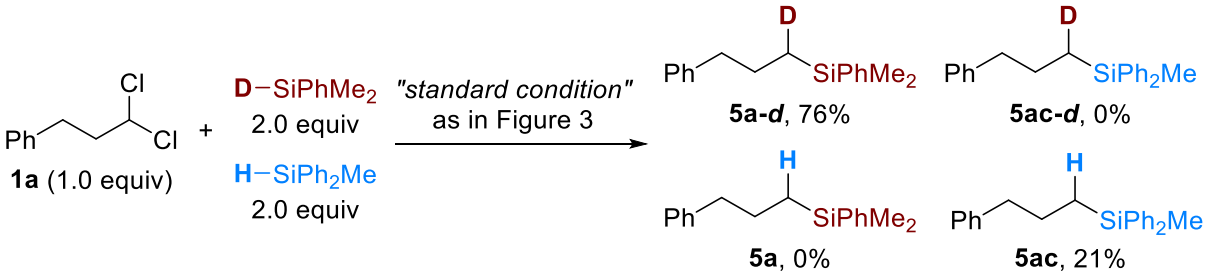


The product **dimethyl(phenyl)(3-phenylpropyl-1-d)silane** (**5a-*d***)[12] was prepared according to the **GP-3** from (dichloromethyl)benzene and dimethyl(phenyl)silane-*d*, purified by flash column chromatography on silica gel: hexane, white soild. 50.0 mg, 98% yield, >99%D.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.51 – 7.46 (m, 2H), 7.37 – 7.31 (m, 3H), 7.29 – 7.23 (m, 2H), 7.19 – 7.10 (m, 3H), 2.68 – 2.56 (m, 2H), 1.71 – 1.59 (m, 2H), 0.77 (tt, *J* = 8.5, 2.1 Hz, 1H), 0.25 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 142.5, 139.4, 133.5, 128.8, 128.5, 128.2, 127.7, 125.6, 39.8, 25.9, 15.1 (t, *J* = 18.0 Hz) -3.1.

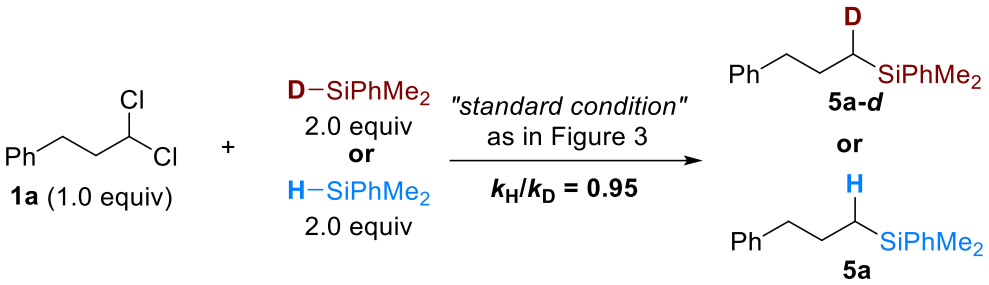
Exchange experiment:



Inside the glovebox under N_2 atmosphere, (TPP)FeCl (7.1 mg, 0.010 mmol, 0.10 equiv), **Mn** (16.5 mg, 0.3 mmol, 3.0 equiv) and N,N-dimethylformamide (DMF) (0.5 mL) were added to an oven-dried 4 mL vial equipped with a magnetic stir bar. The reaction mixture was stirred (800 r/min) at room temperature for 0.5 hours. Next, (3,3-dichloropropyl)benzene (18.8 mg, 0.1 mmol, 1.0 equiv), dimethyl(phenyl)silane-*d* (27.2 mg, 0.20 mmol, 2.0 equiv) and methyldiphenylsilane (39.6 mg, 0.20 mmol, 2.0 equiv) were added sequentially, The vial was quickly sealed with a PTFE septum cap. Then the vial was taken out of the glovebox, and stirred (800 r/min) at 25 °C for 24 hours.

Work-up: The reaction mixture was diluted with 1.0 mL ether, followed by the addition of the internal standard 1,3,5-trimethoxybenzene (0.10 mmol). The reaction solvent was removed by concentration under reduced pressure. The residue was subjected to flash column chromatography with PE as the eluent to afford **5a-d** (19.4 mg, 76%) and **5ac** (6.6 mg, 21%).

Parallel KIE experiment



Inside the glovebox under N_2 atmosphere, (TPP)FeCl (14.2 mg, 0.020 mmol, 0.10 equiv), Mn (33.3 mg, 0.6 mmol, 3.0 equiv) and N,N-dimethylformamide (DMF) (1 mL) were added to an oven-dried 4 mL vial equipped with a magnetic stir bar. The reaction mixture was stirred (800 r/min) at room temperature for 0.5 hours. Next, (3,3-dichloropropyl)benzene (38 mg, 0.2 mmol, 1.0 equiv), 1,3,5-trimethoxybenzene (0.10 mmol, 16.8 mg) as Internal standard, dimethyl(phenyl)silane (54.4 mg, 0.40 mmol, 2.0 equiv) or dimethyl(phenyl)silane-*d* (54.8 mg, 0.40 mmol, 2.0 equiv) were added sequentially, The vial was quickly sealed with a PTFE septum cap. The vial was stirred (800 r/min) at 25 °C in the glovebox.

Work-up: The 0.05 ml reaction solution was separated from vial with every 6 min, was diluted with 1.0 mL ether. The reaction solvent was removed by concentration under reduced pressure. The residue was subjected to short flash column chromatography with PE:EA (5:1) as the eluent to determine the ^{1}H NMR yield of silane product.

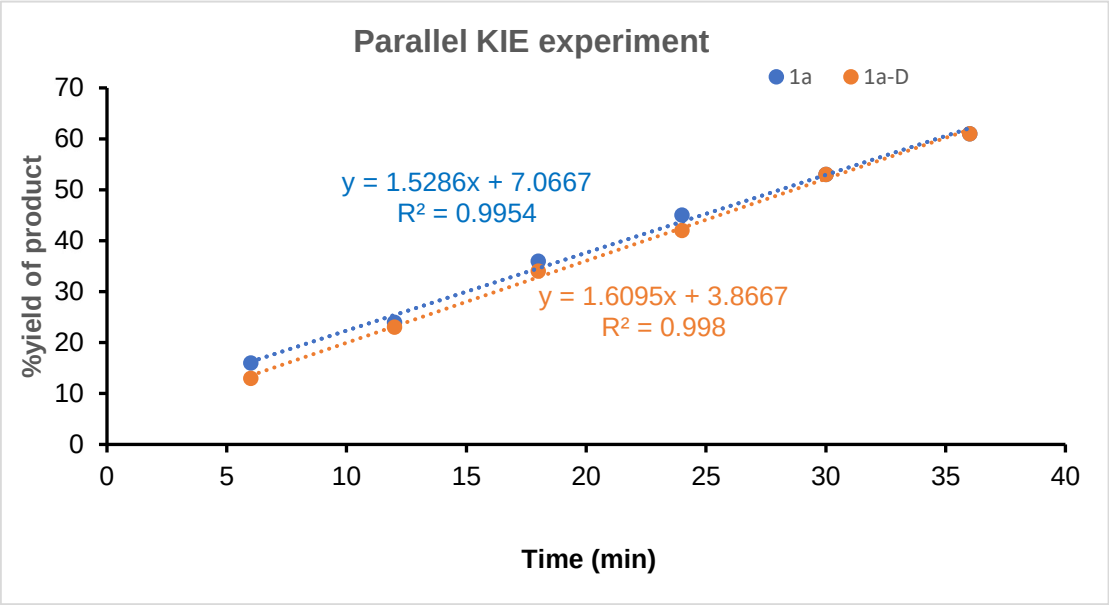
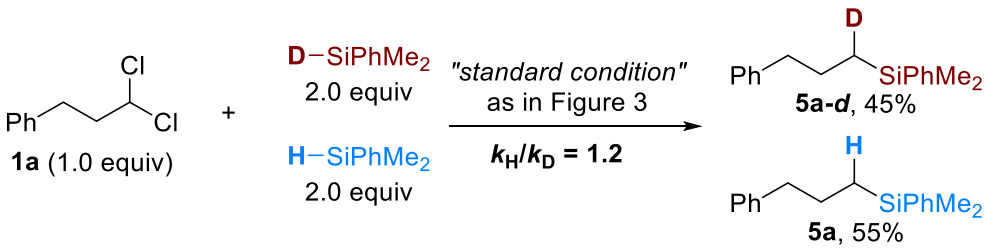


Figure S1. Parallel KIE experiment

k_H/k_D = 1.5286/1.6095 = 0.95

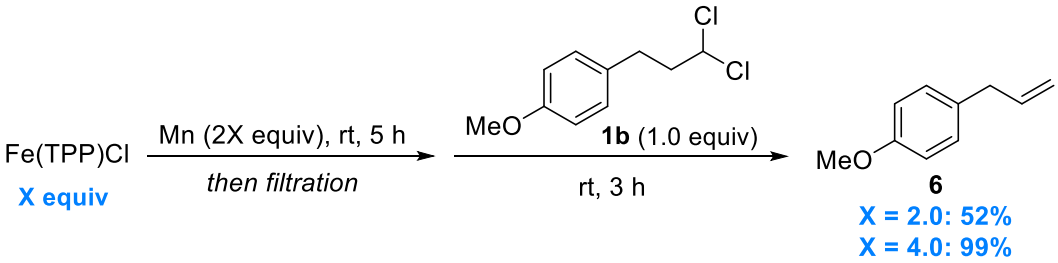
One-pot competition KIE experiment



Inside the glovebox under N_2 atmosphere, (TPP)FeCl (14.2 mg, 0.020 mmol, 0.10 equiv), Mn (33.3 mg, 0.6 mmol, 3.0 equiv) and N,N-dimethylformamide (DMF) (1 mL) were added to an oven-dried 4 mL vial equipped with a magnetic stir bar. The reaction mixture was stirred (800 r/min) at room temperature for 0.5 hours. Next, (3,3-dichloropropyl)benzene (38 mg, 0.2 mmol, 1.0 equiv), dimethyl(phenyl)silane (54.4 mg, 0.40 mmol, 2.0 equiv) and dimethyl(phenyl)silane-*d* (54.8 mg, 0.40 mmol, 2.0 equiv) were added sequentially, The vial was quickly sealed with a PTFE septum cap. The vial was stirred (800 r/min) at 25 °C in the glovebox for 24 h.

Work-up: The reaction mixture was diluted with 1.0 mL ether, followed by the addition of the internal standard 1,3,5-trimethoxybenzene (0.10 mmol). The reaction solvent was removed by concentration under reduced pressure. The residue was subjected to flash column chromatography with PE as the eluent to obtain the mixed product of **1a** and **1a-D**.

Control experiments:



Inside the glovebox under N_2 atmosphere, (TPP)FeCl (0.05x mmol, x equiv) or, **Mn** (0.1x mmol, 2x equiv) and N,N-dimethylformamide (DMF) (1 mL) were added to an oven-dried 4 mL vial equipped with a magnetic stir bar. The reaction mixture was stirred (800 r/min) at room temperature for 5 hours. Filter of the reaction mixture to remove excess Mn powder. Next, 1-(3,3-dichloropropyl)-4-methoxybenzene (11.1 mg, 0.05 mmol, 1.0 equiv) were added to the filtrate sequentially, the vial was quickly sealed with a PTFE septum cap. The vial was stirred (800 r/min) at 25 °C in the glovebox for 3 h. Finally, 2N HCl solution (0.5 ml) was added to the reaction mixture. which was subsequently extracted by diethyl ether (1 ml*3). The combined organic layers were dried over Na_2SO_4, evaporated under reduced pressure to determine product by crude ^1H NMR and purified by flash column chromatography on silica gel: hexane as eluent to obtain products.

V. References

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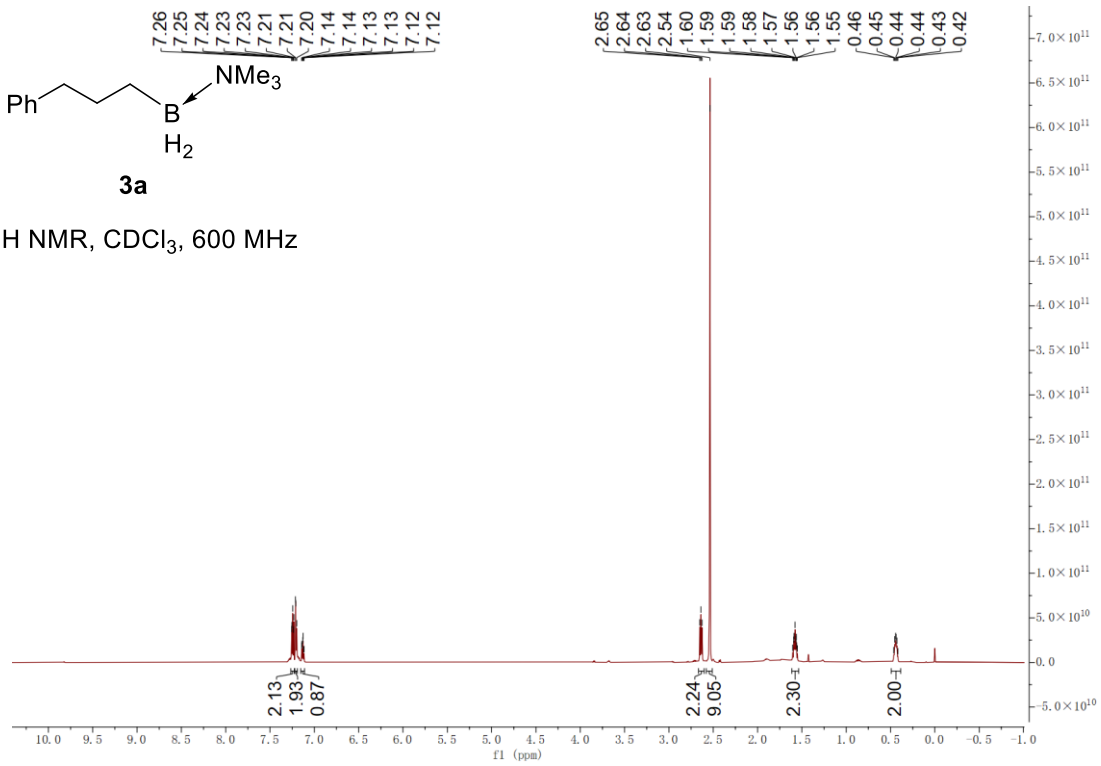
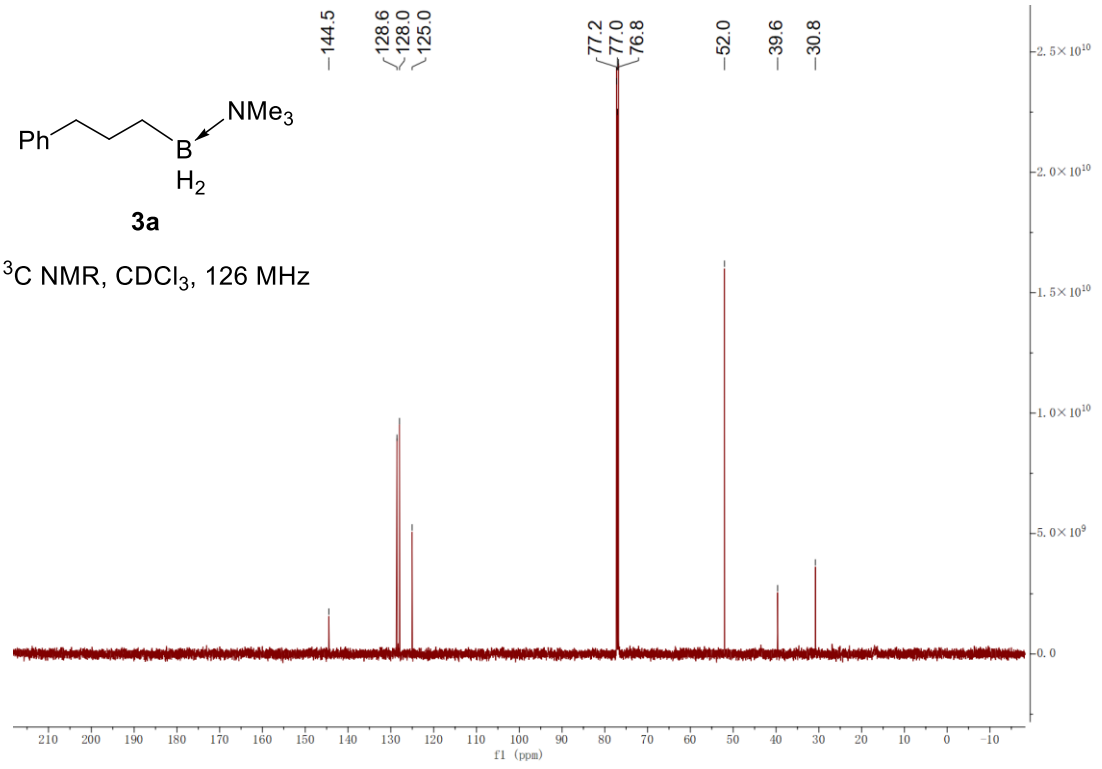
VI. NMR Spectra



3a

^{1}H NMR, $CDCl_3$, 600 MHz



3a

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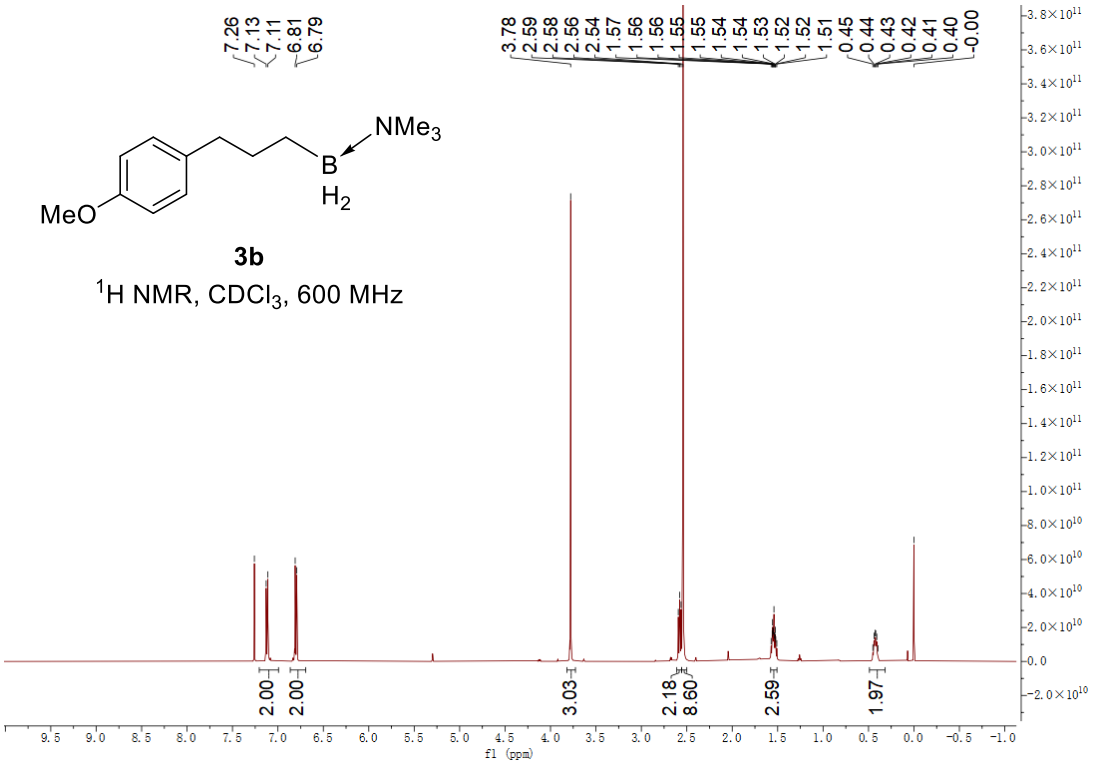

3b

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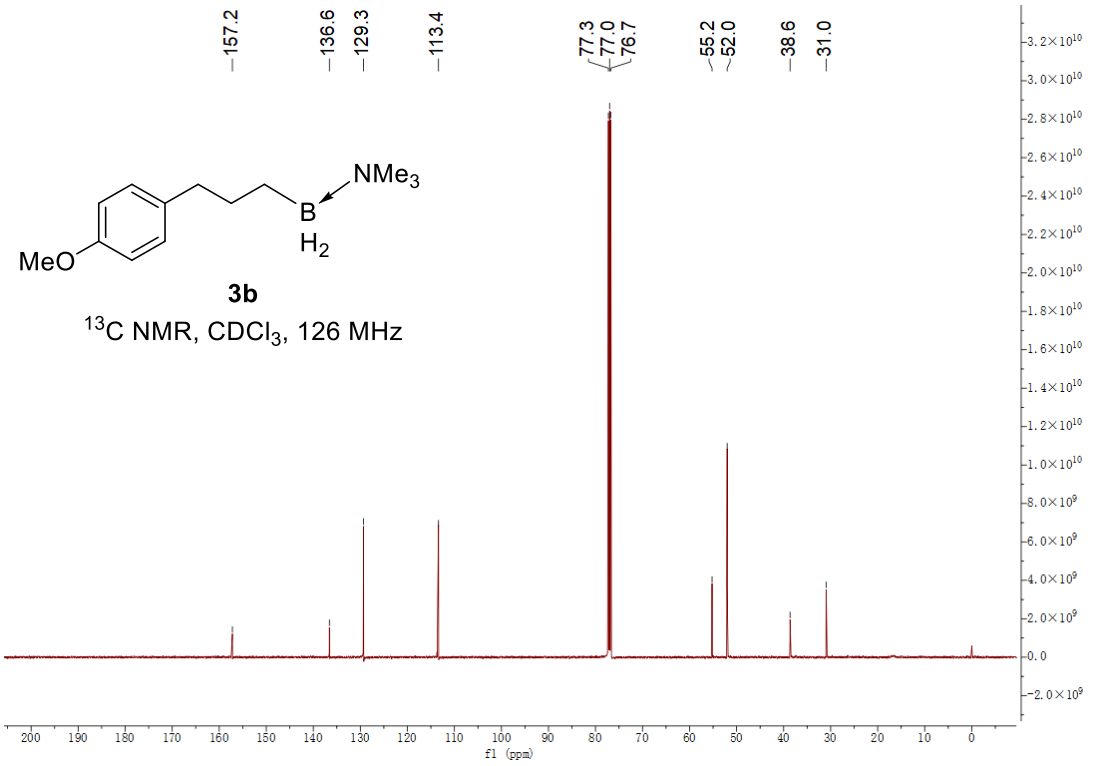
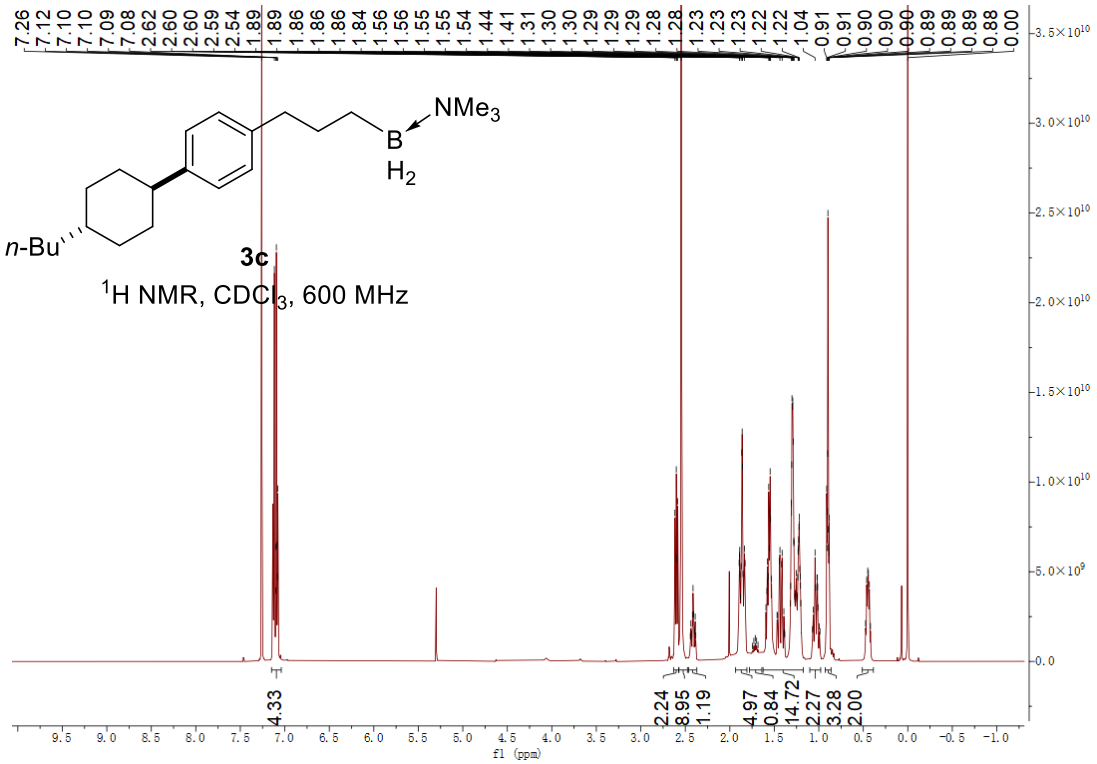


3b

^{13}C NMR, $CDCl_3$, 126 MHz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H2
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3c
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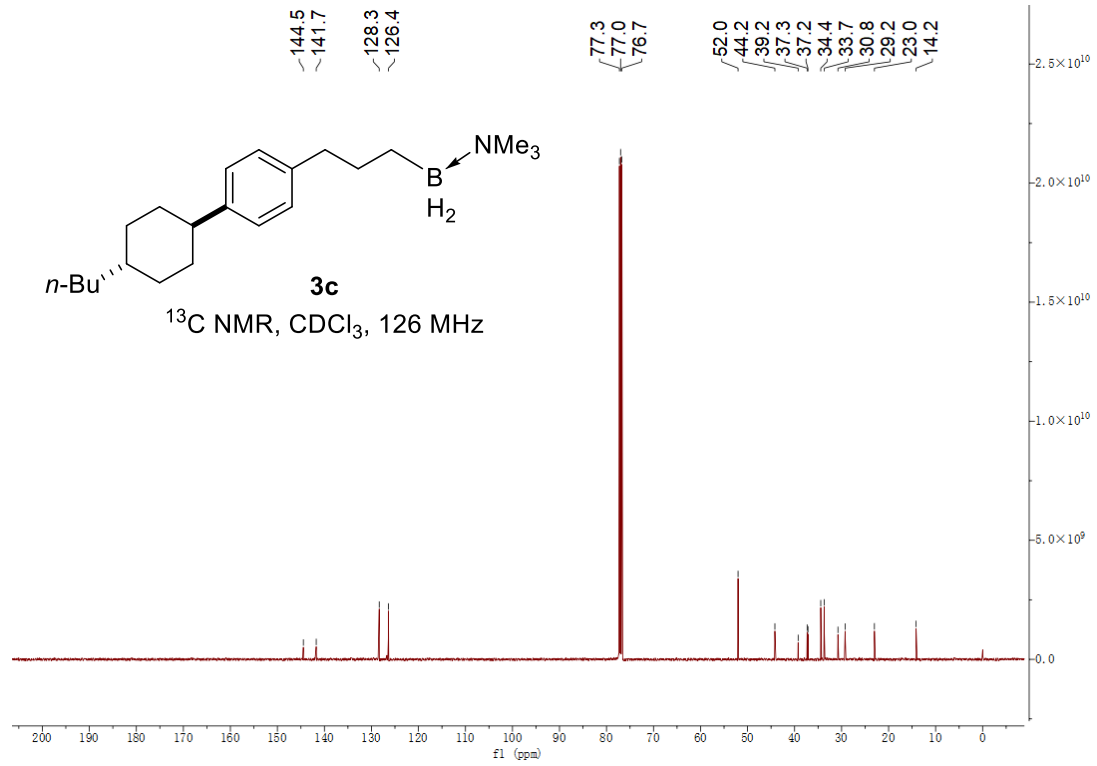
f1 (ppm)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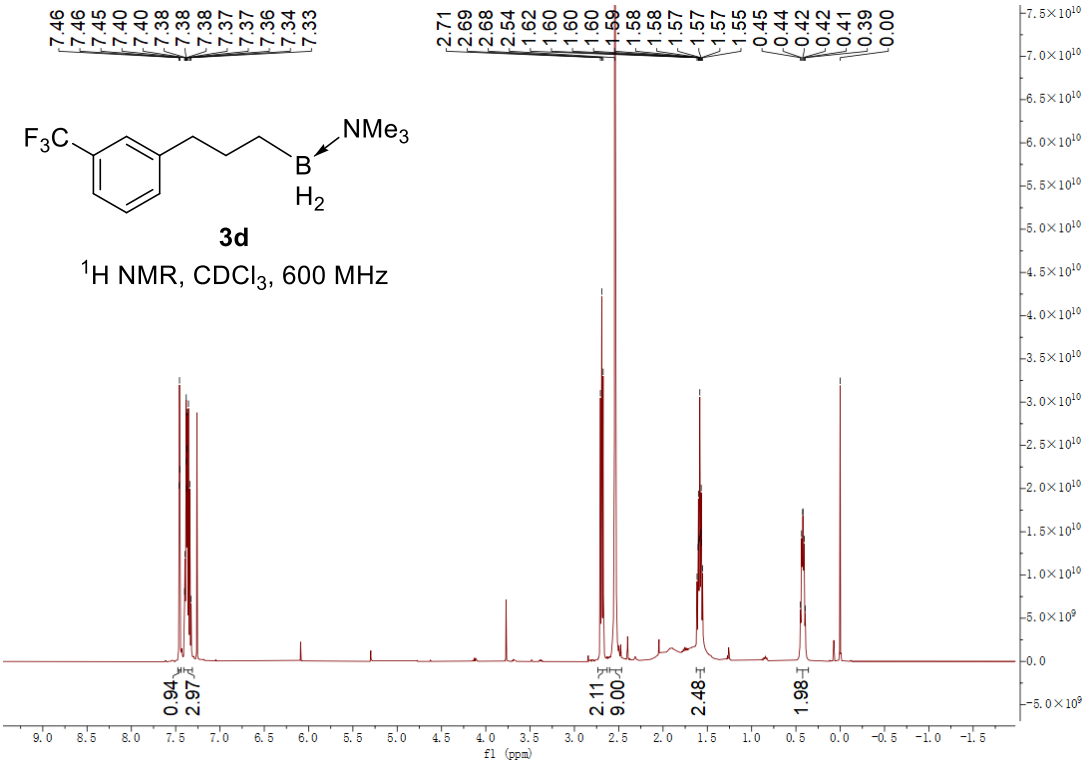
H2
n-Bu
3c
13C NMR, CDCl3, 126 MHz
f1 (ppm)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2.54
1.62
1.60
1.60
1.60
1.59
1.58
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1.57
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1.57
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0.42
0.41
0.39
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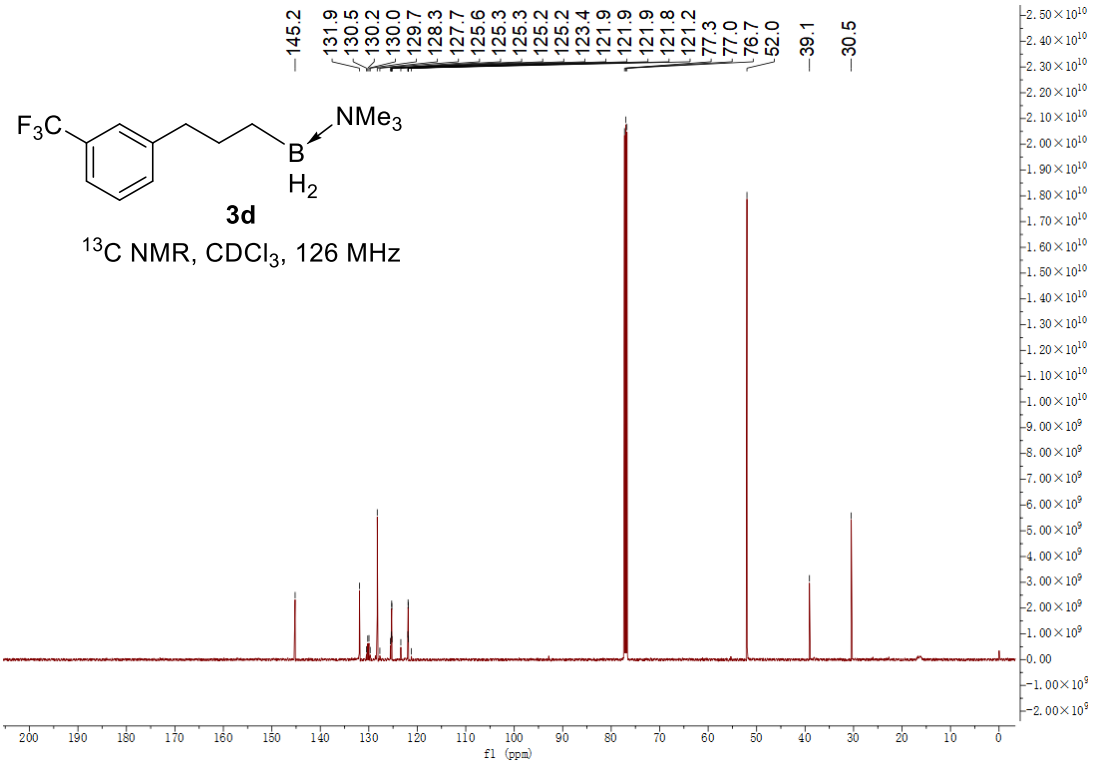
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121.8
121.2
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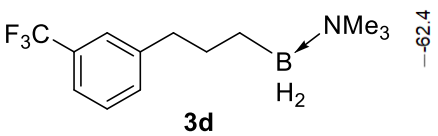
H2
3d
13C NMR, CDCl3, 126 MHz
f1 (ppm)



3d

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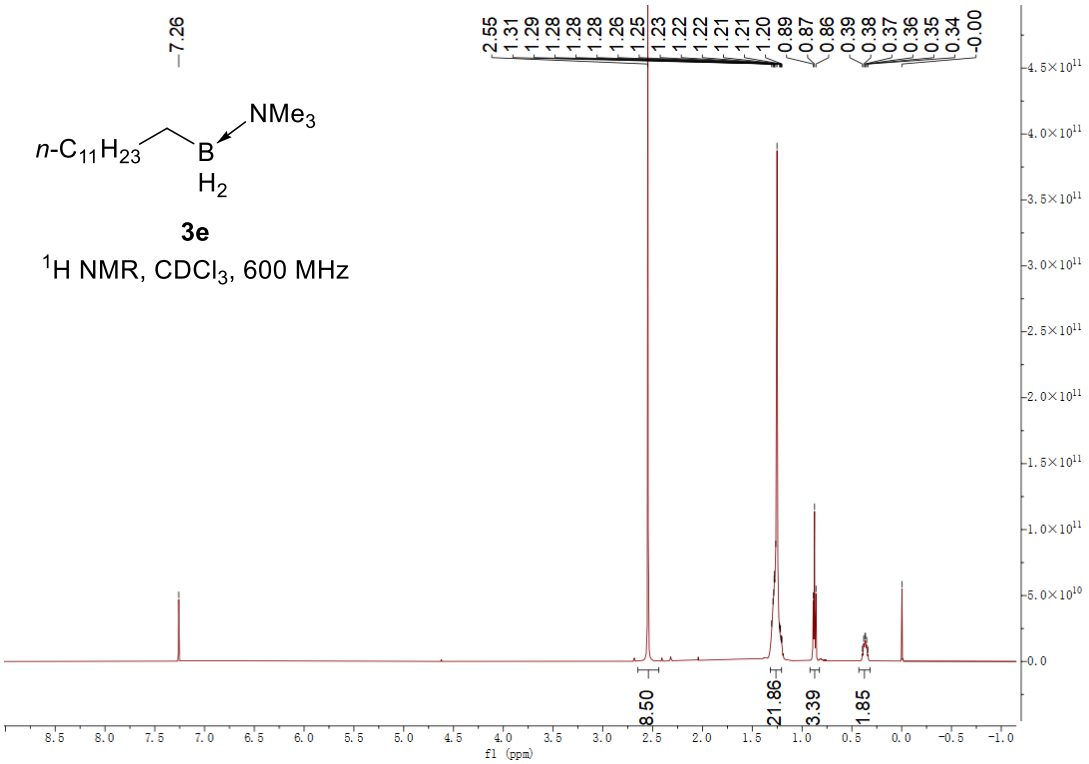

n-$C_{11}H_{23}$–B(NMe_3)H_2

3e

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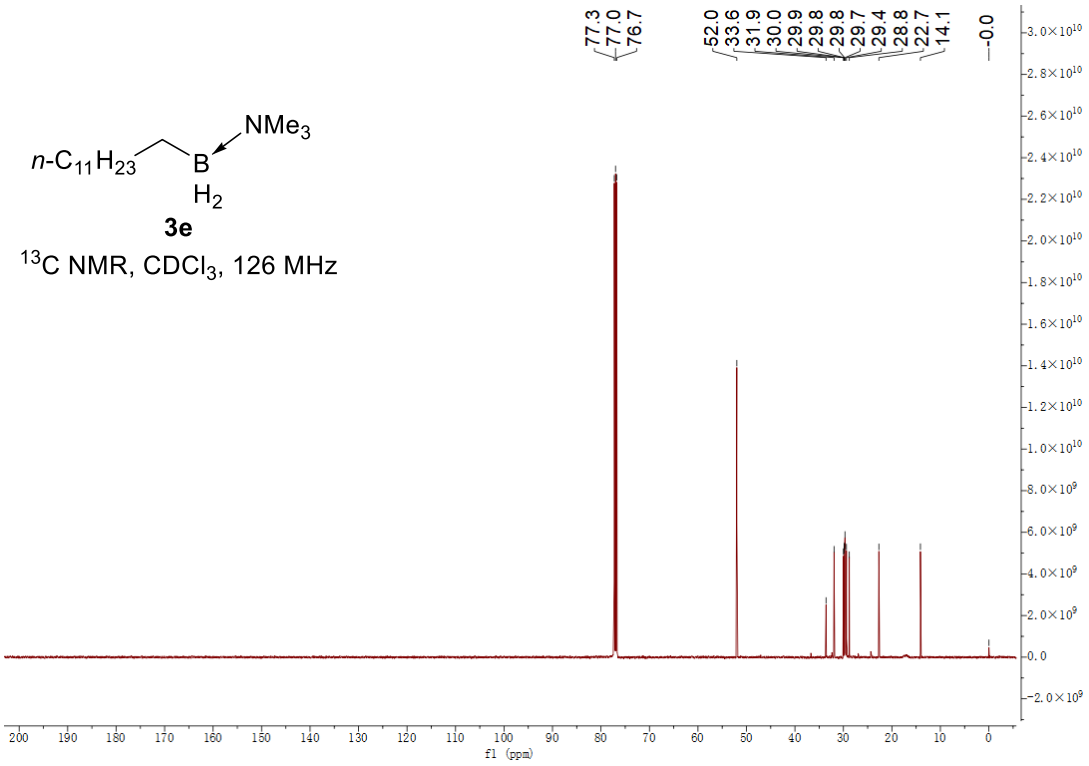
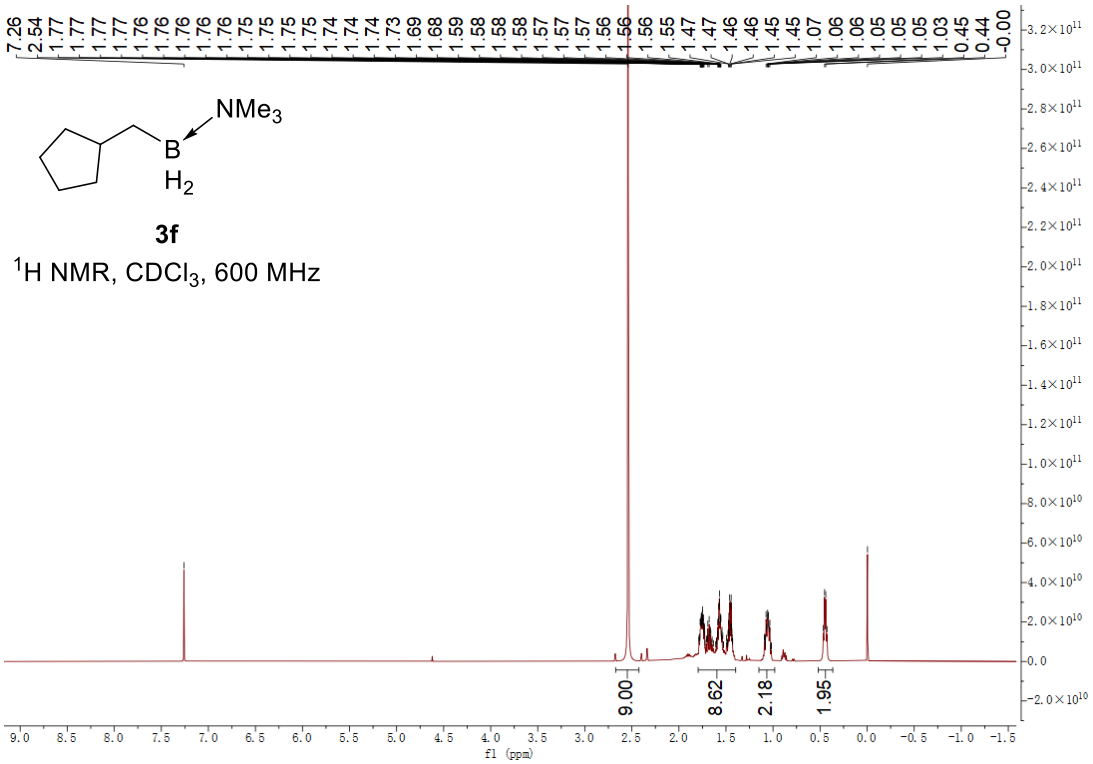


n-$C_{11}H_{23}$–B(NMe_3)H_2

3e

^{13}C NMR, $CDCl_3$, 126 MHz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B
H2
3f
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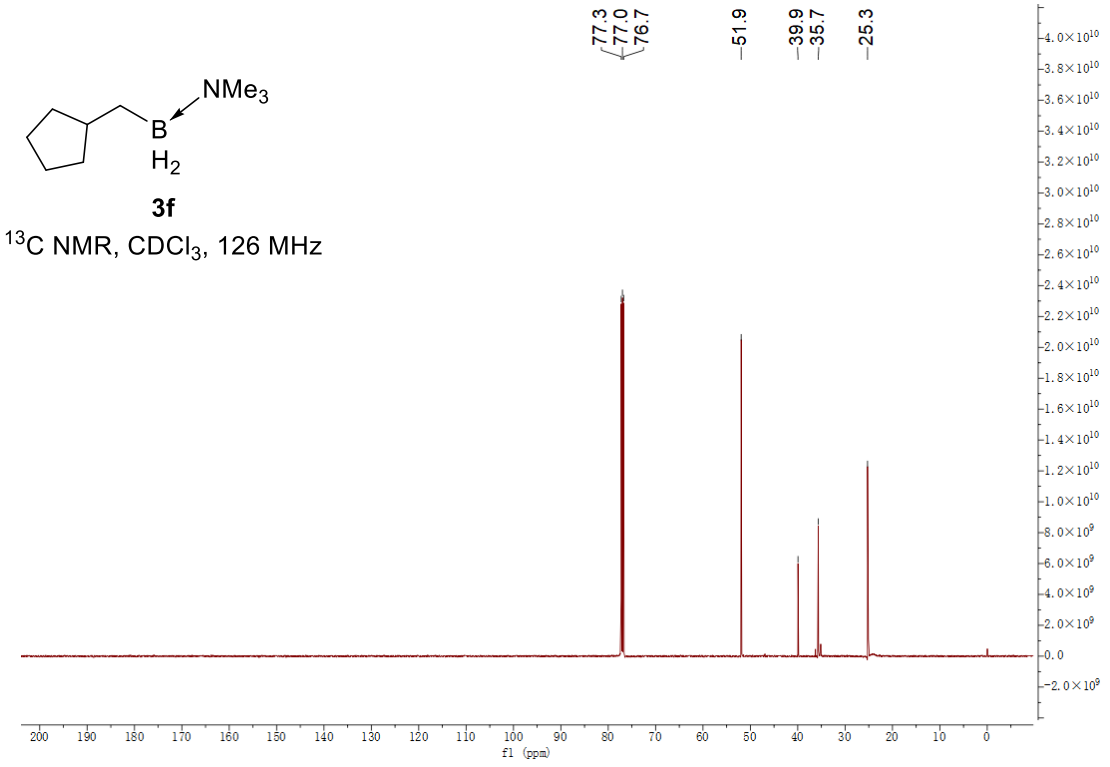
2.18
1.95
f1 (ppm)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H2
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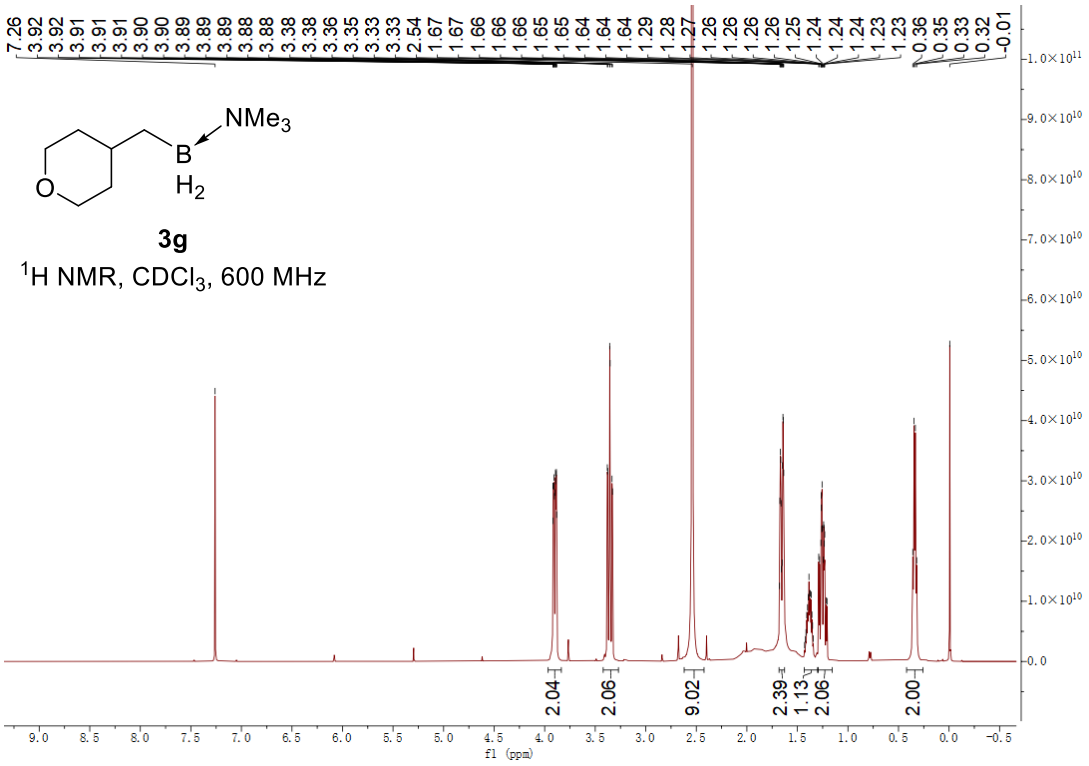
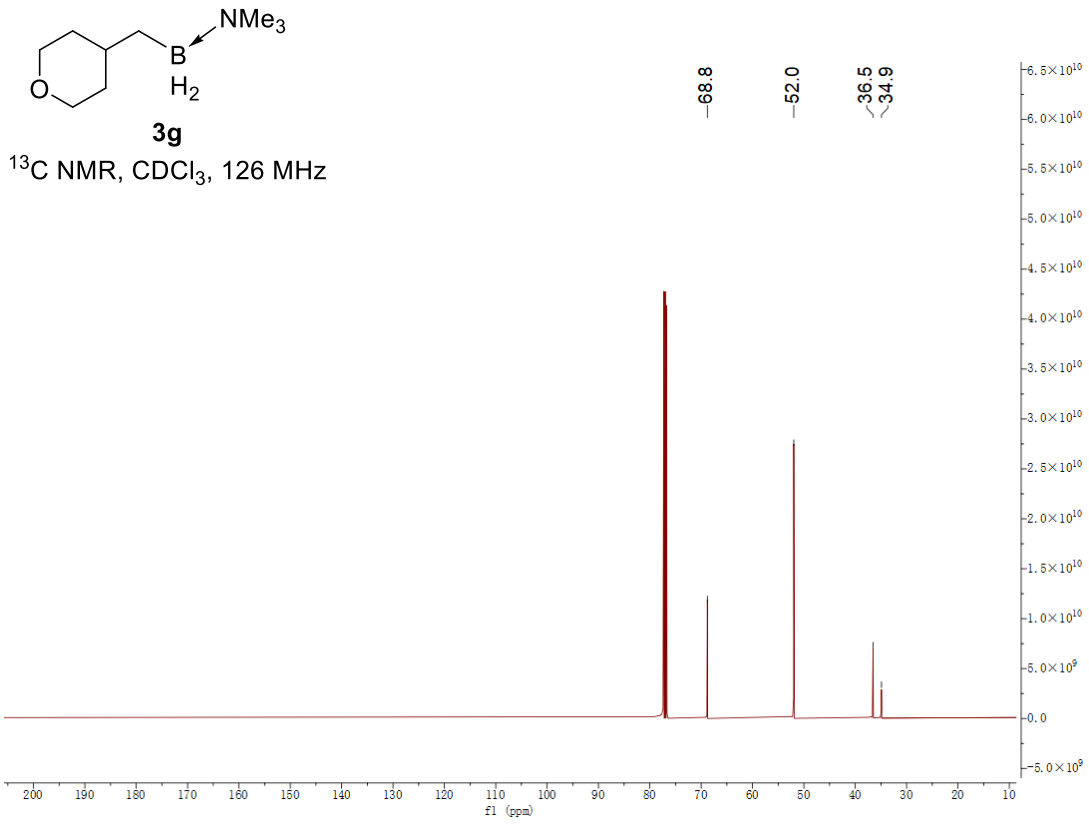
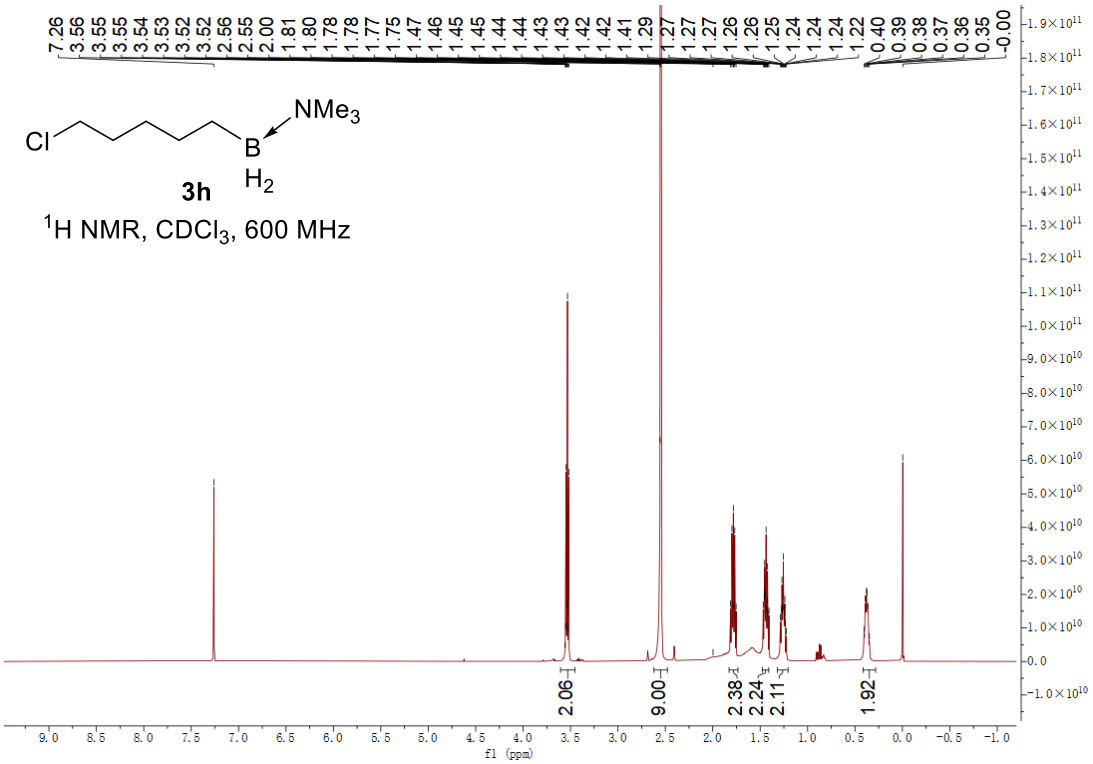
25.3
f1 (ppm)

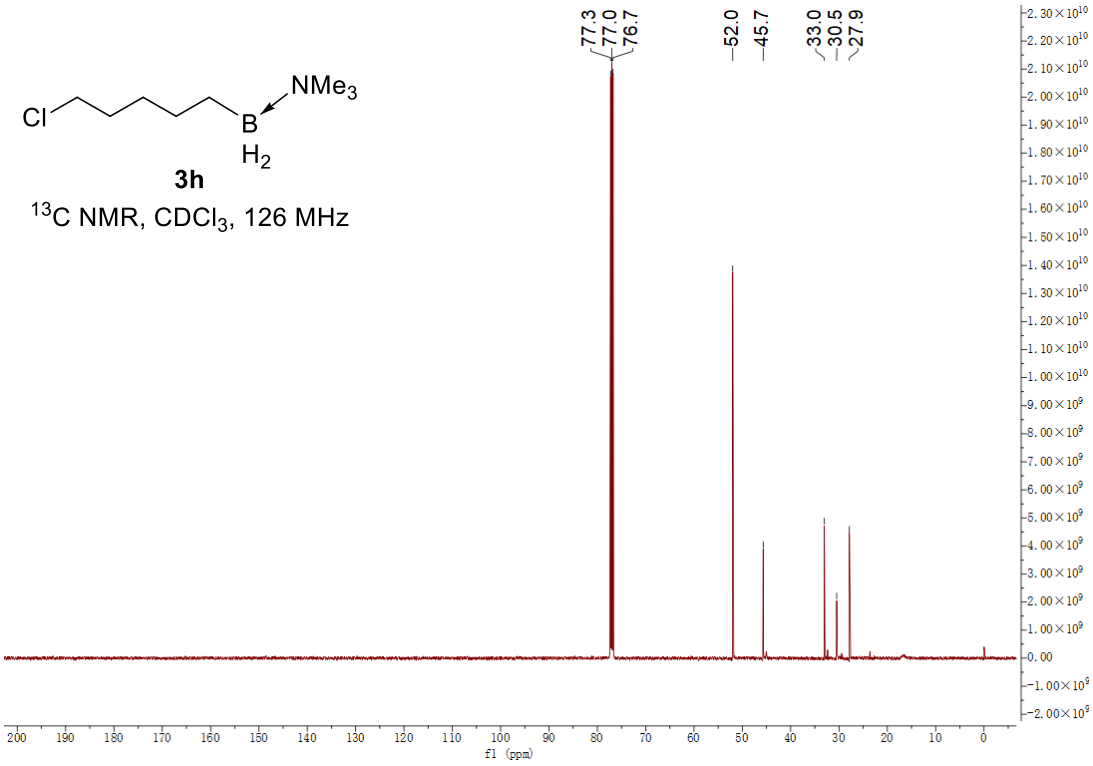

NMe3
B
H2
O
3g
1H NMR, CDCl3, 600 MHz
f1 (ppm)


NMe3
B
H2
O
3g
13C NMR, CDCl3, 126 MHz
68.8
52.0
36.5
34.9
f1 (ppm)



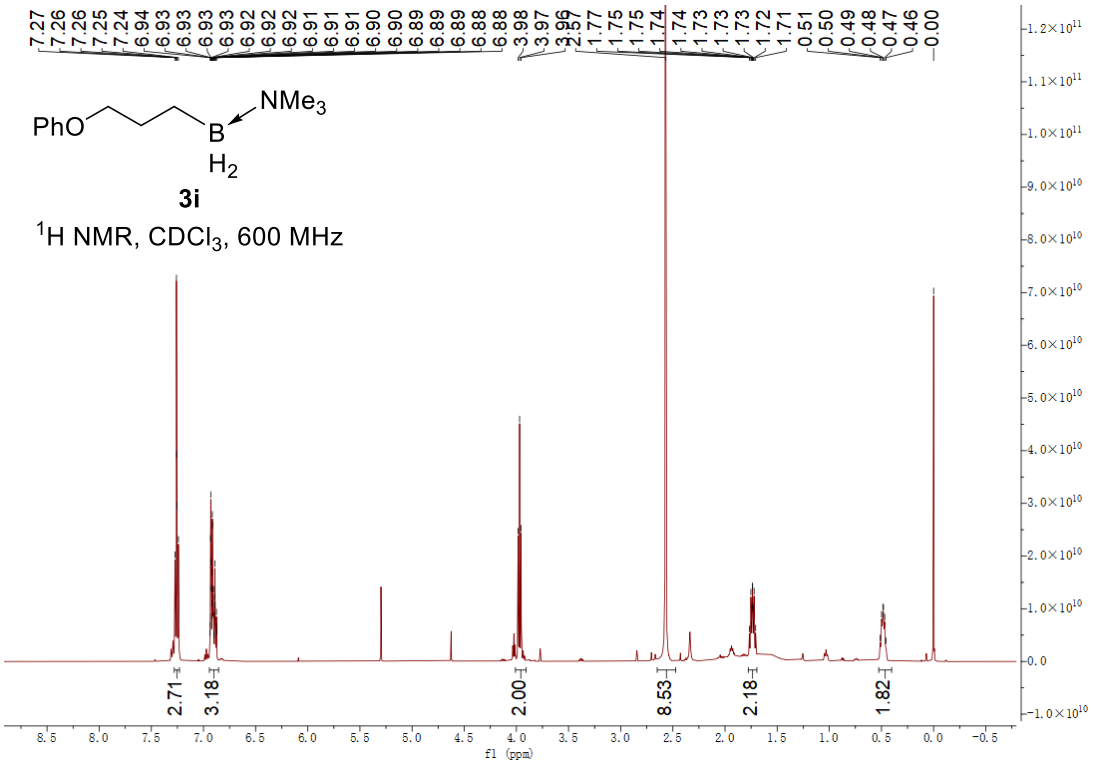
3h

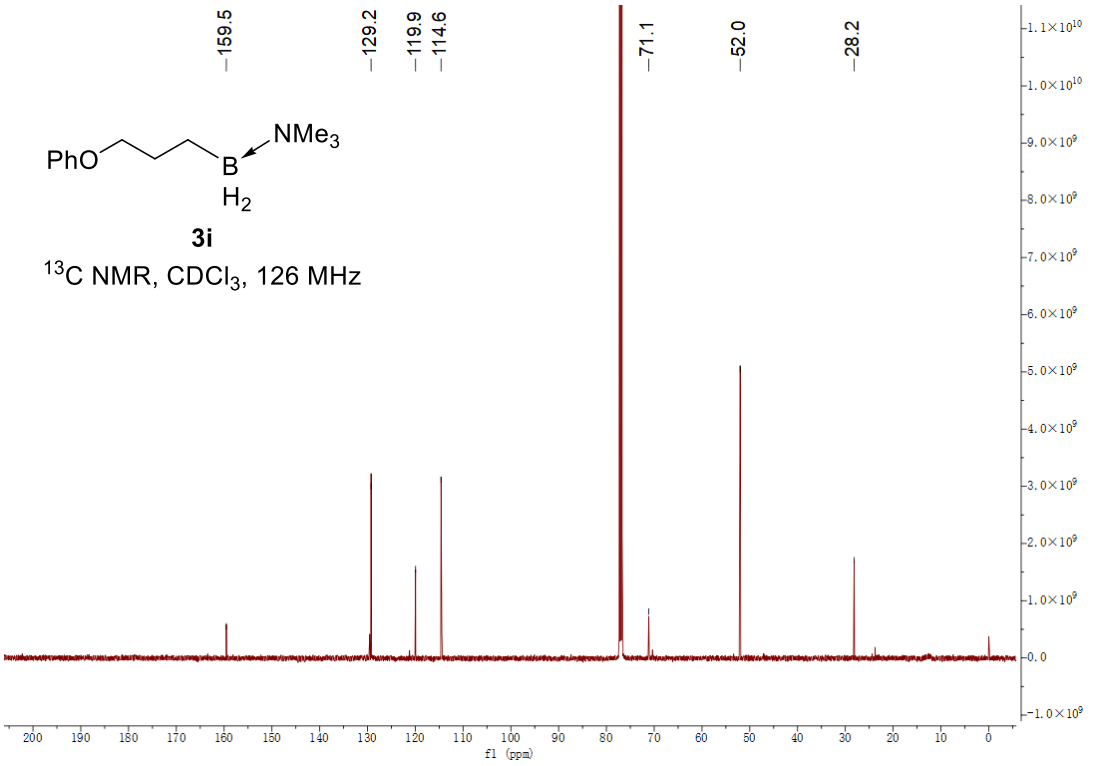
^{1}H NMR, $CDCl_3$, 600 MHz

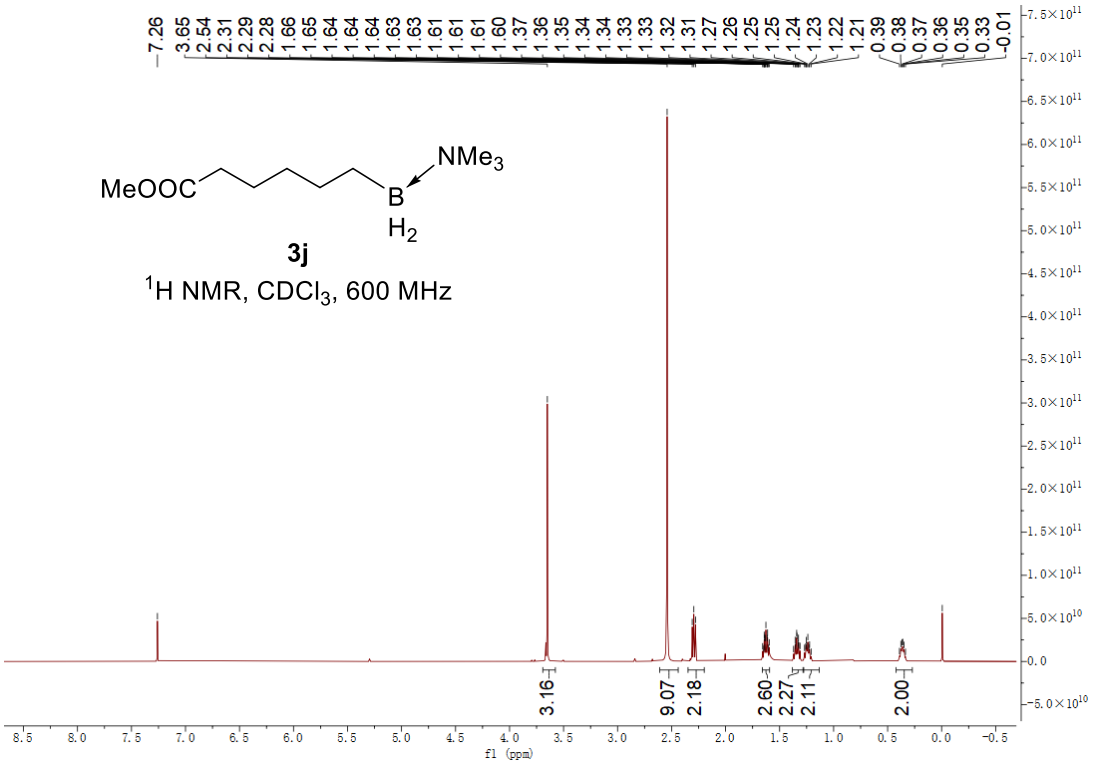


3h

^{13}C NMR, $CDCl_3$, 126 MHz

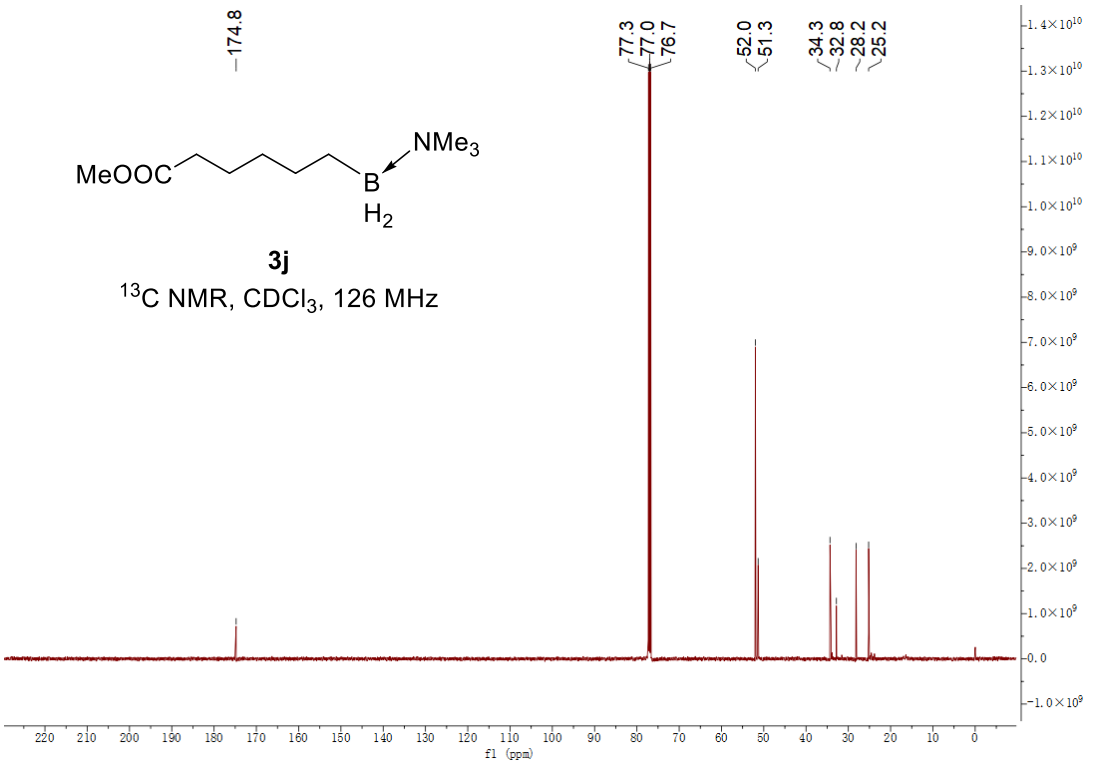

PhO
B
NMe3
H2
3i
1H NMR, CDCl3, 600 MHz
f1 (ppm)


PhO
B
NMe3
H2
3i
13C NMR, CDCl3, 126 MHz
159.5
129.2
119.9
114.6
71.1
52.0
28.2
f1 (ppm)



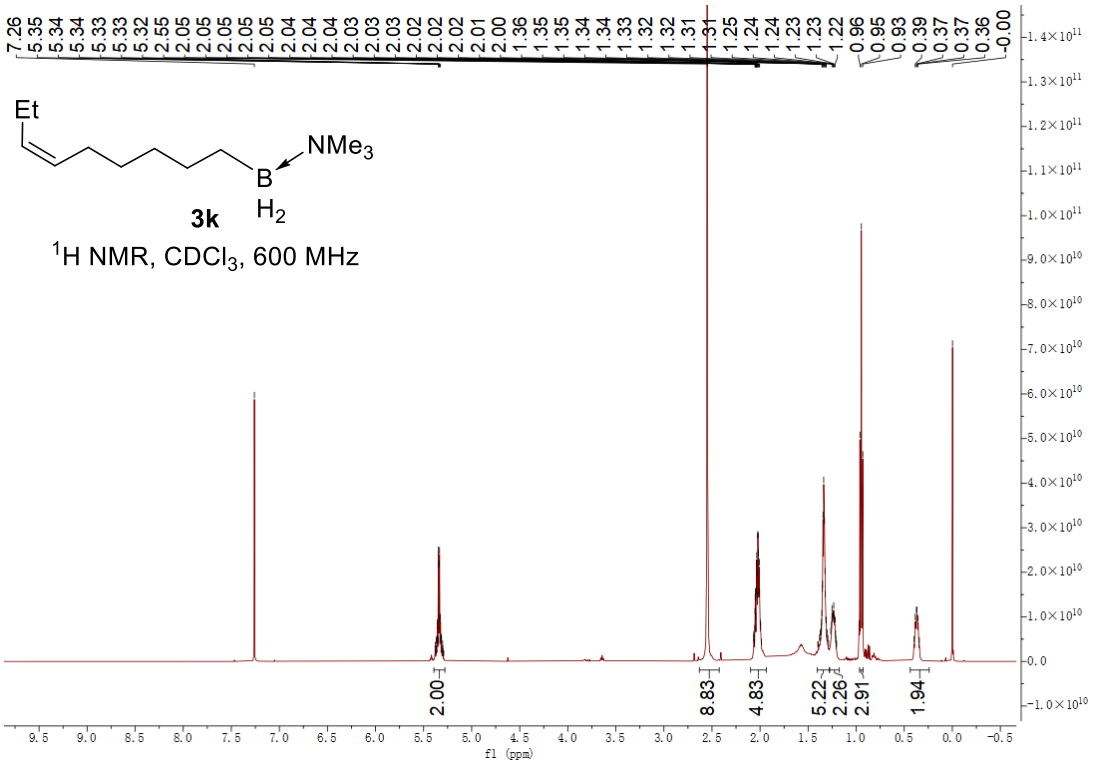
3j

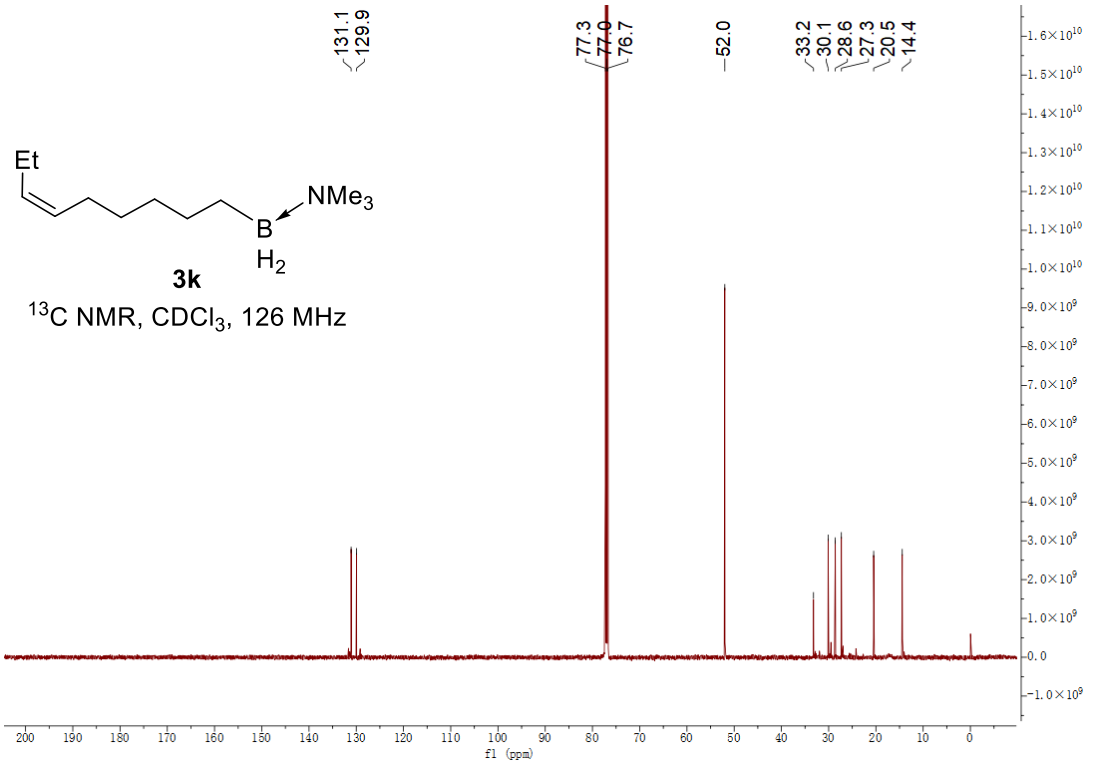
^{1}H NMR, $CDCl_3$, 600 MHz

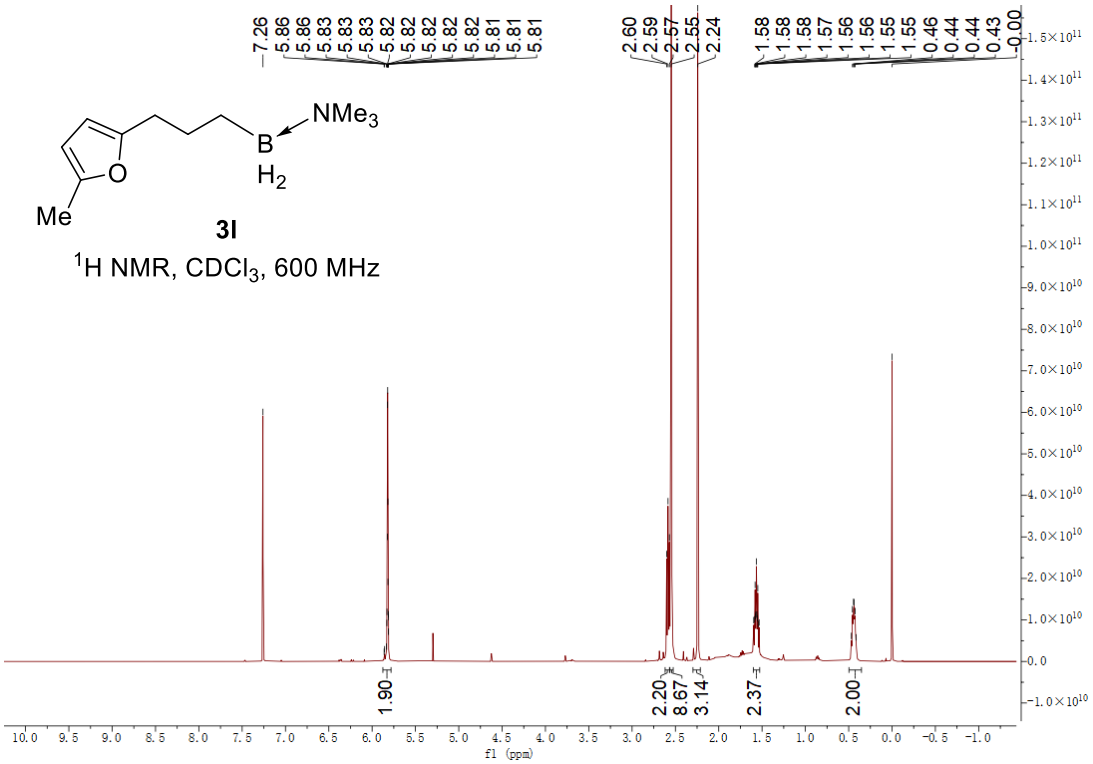


3j

^{13}C NMR, $CDCl_3$, 126 MHz

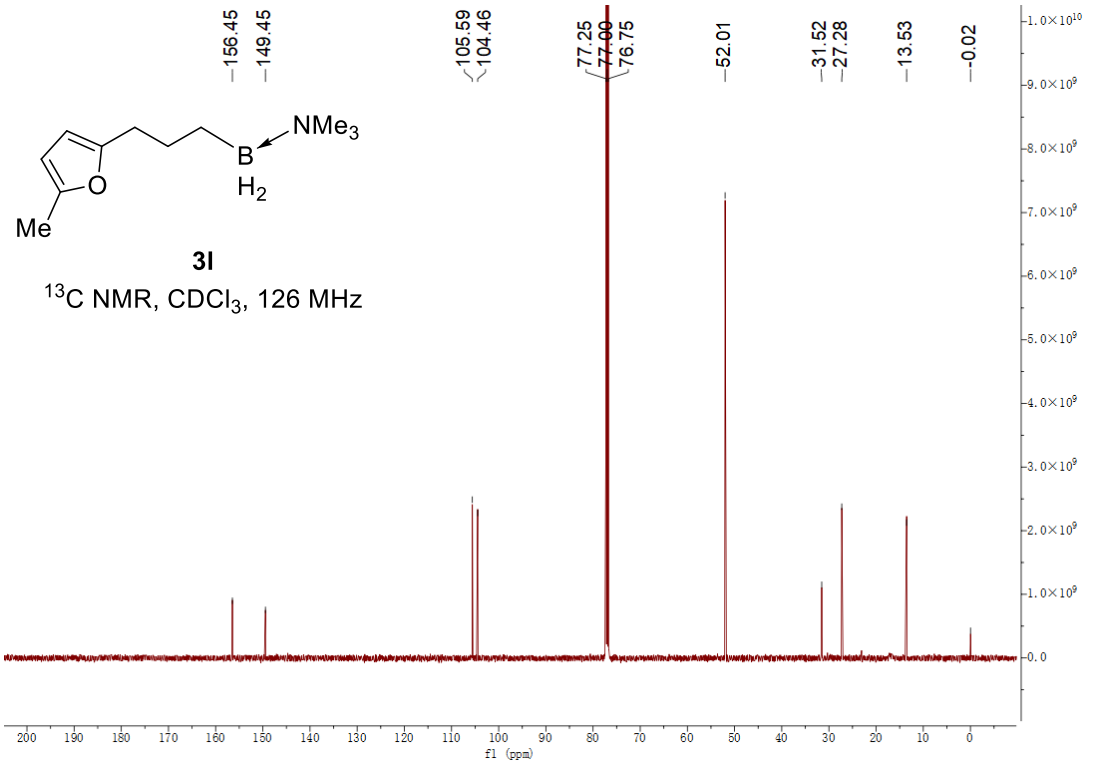

Et
NMe3
B
H2
3k
1H NMR, CDCl3, 600 MHz
f1 (ppm)


Et
NMe3
B
H2
3k
13C NMR, CDCl3, 126 MHz
f1 (ppm)



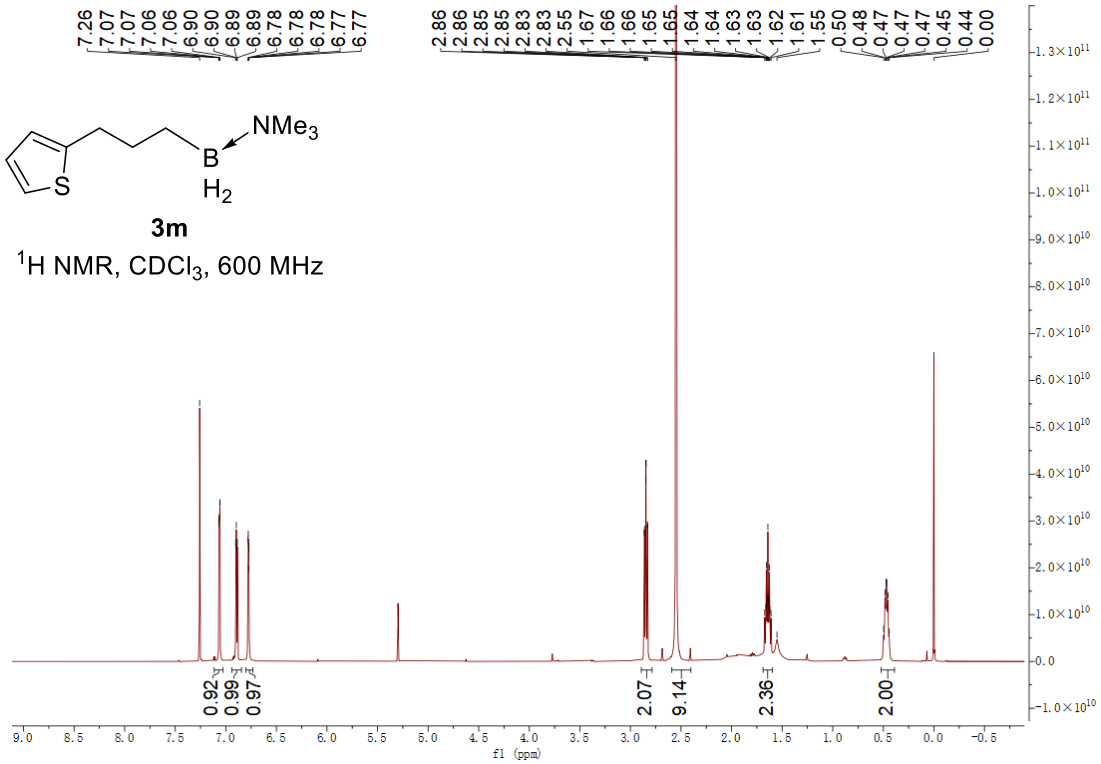
3l

^{1}H NMR, $CDCl_3$, 600 MHz



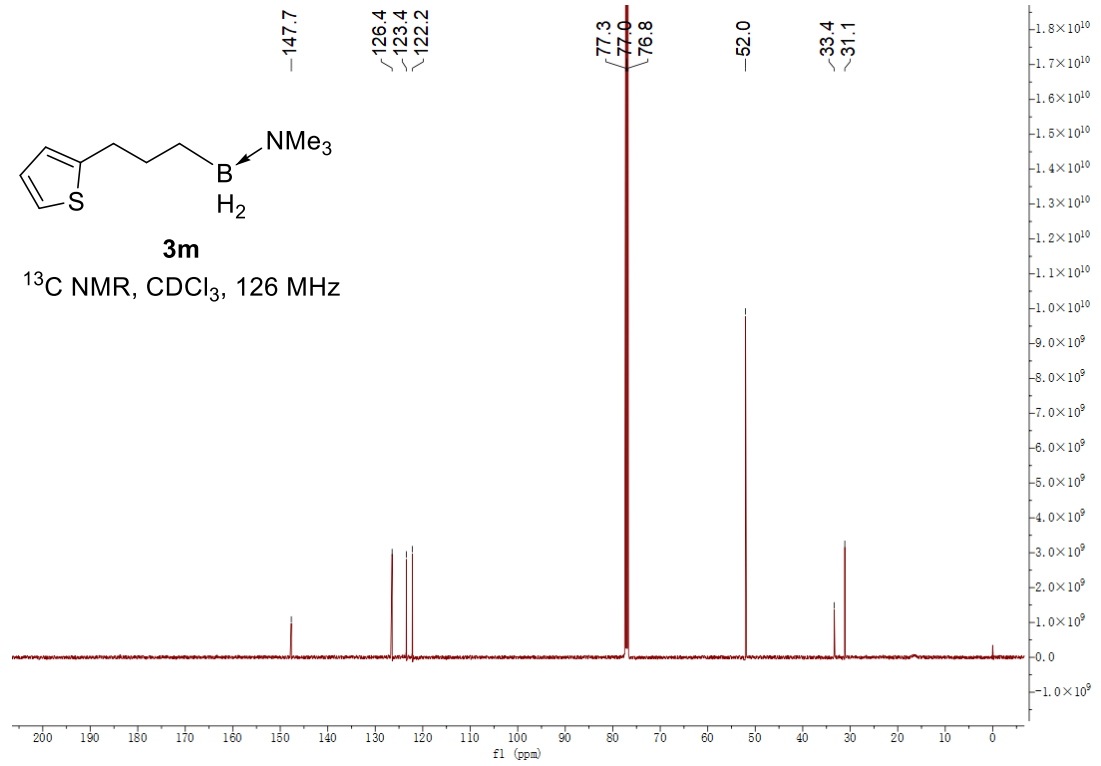
3l

^{13}C NMR, $CDCl_3$, 126 MHz



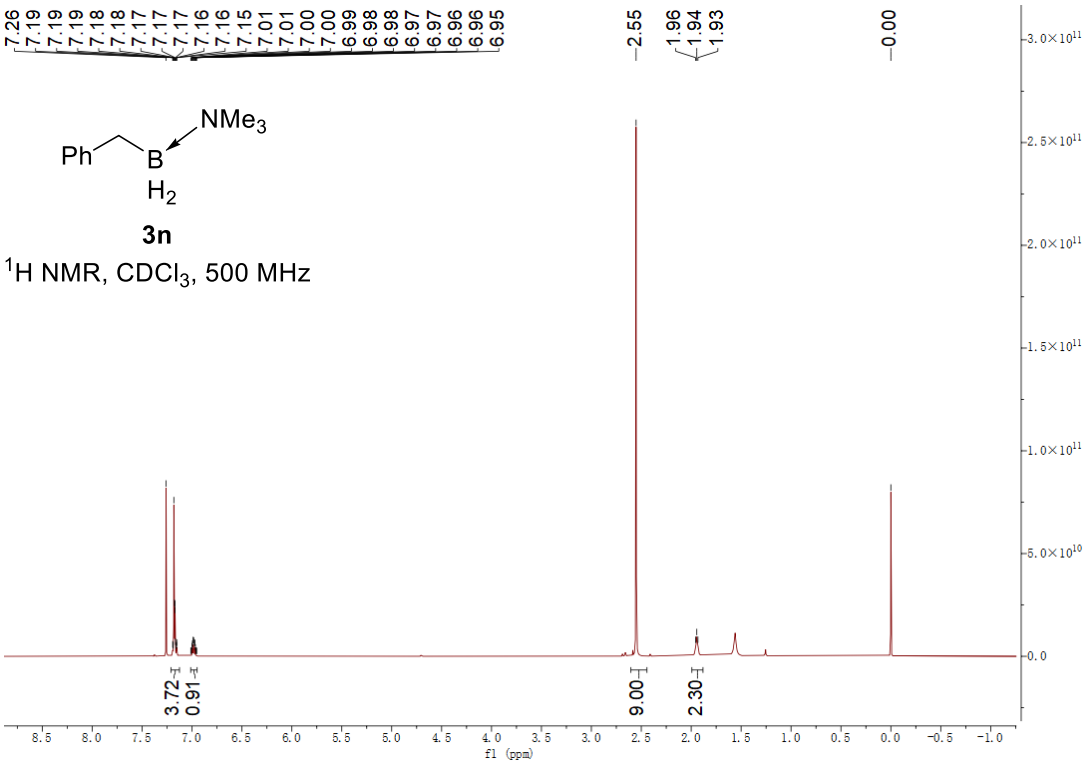
3m

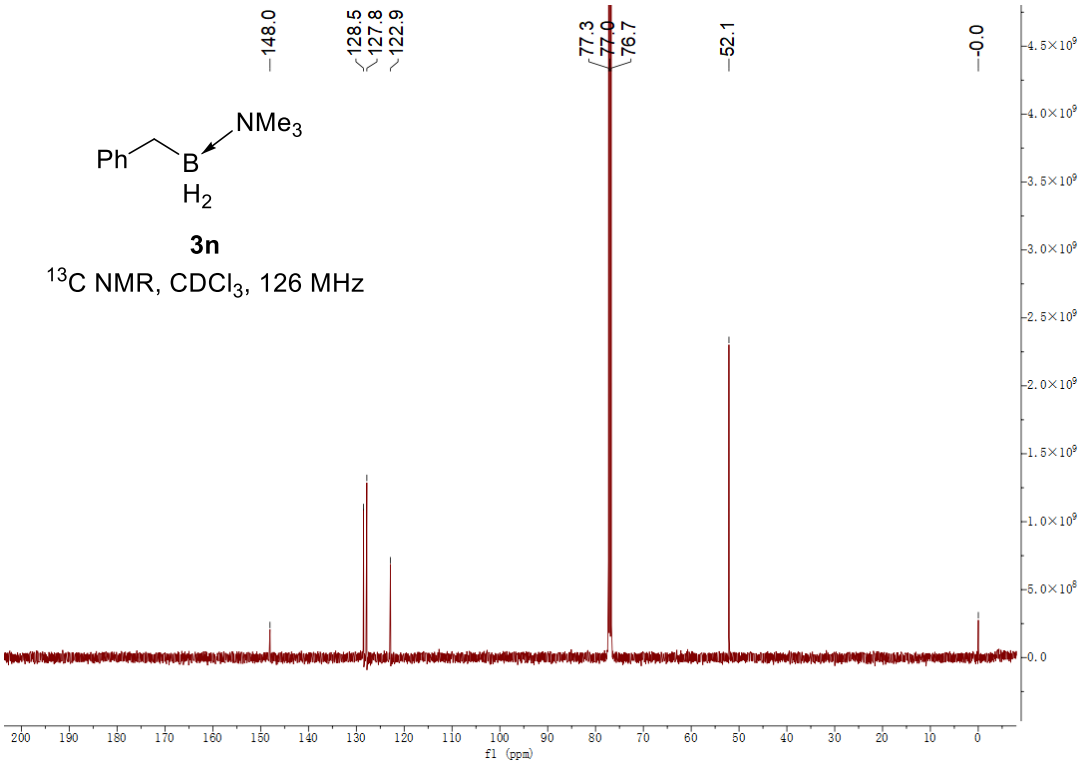
^{1}H NMR, $CDCl_3$, 600 MHz

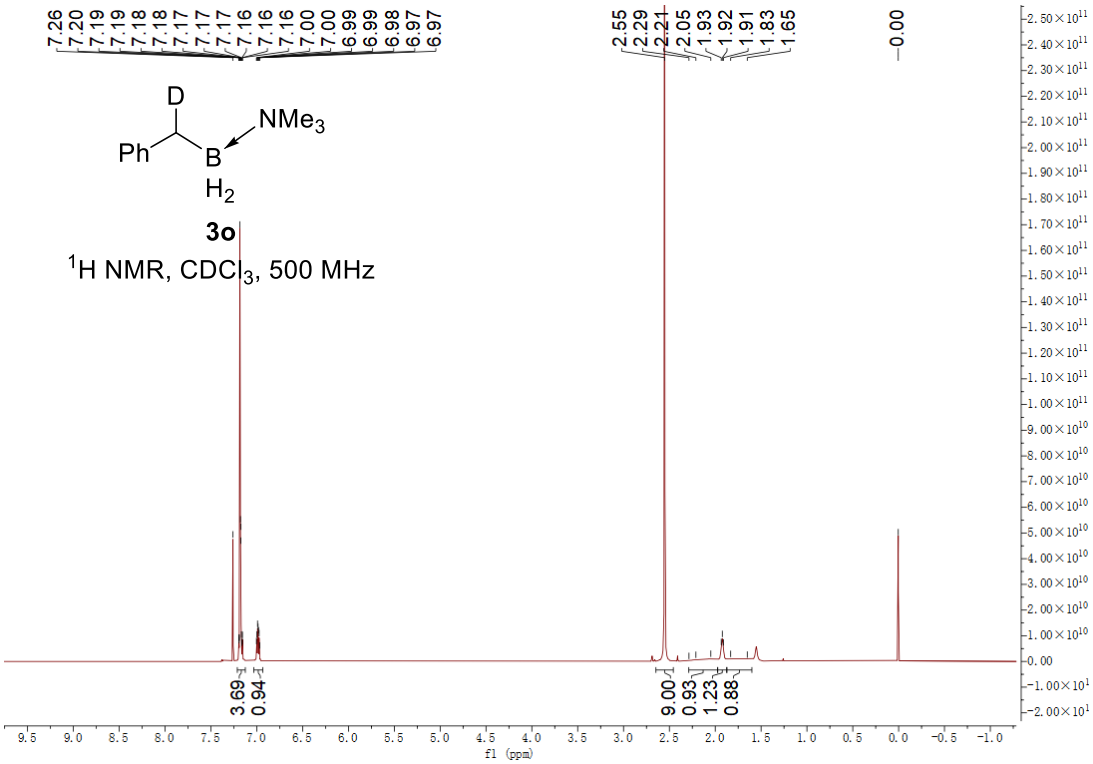


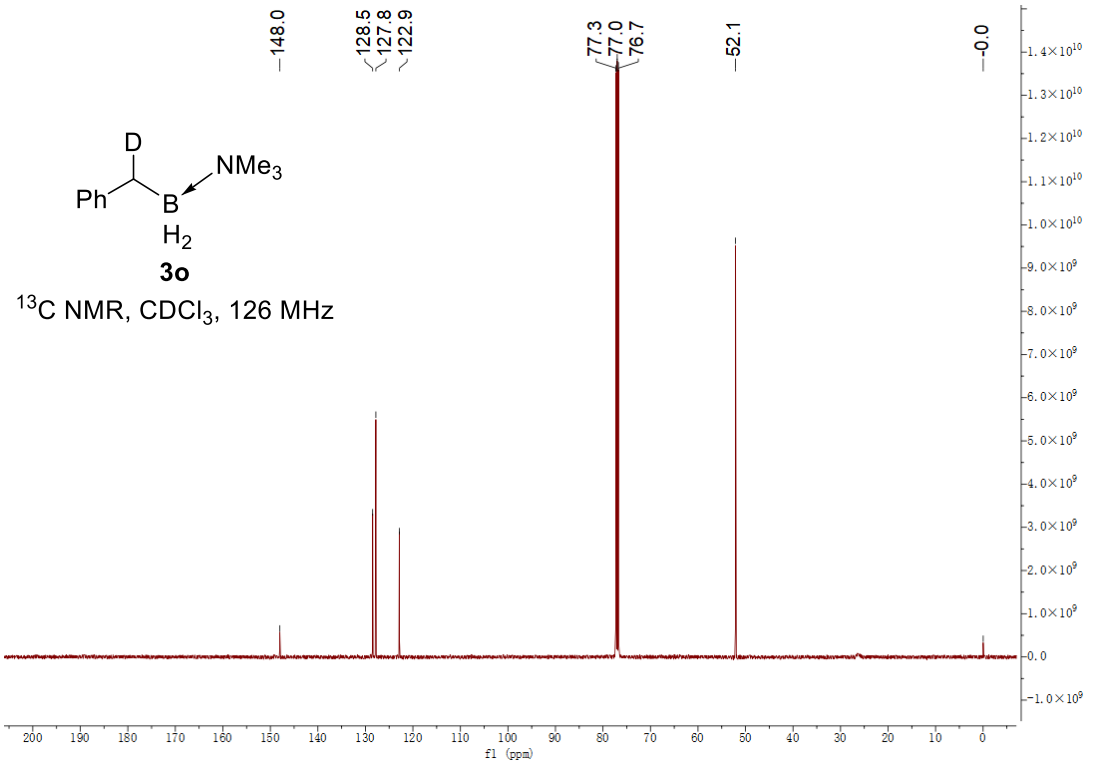
3m

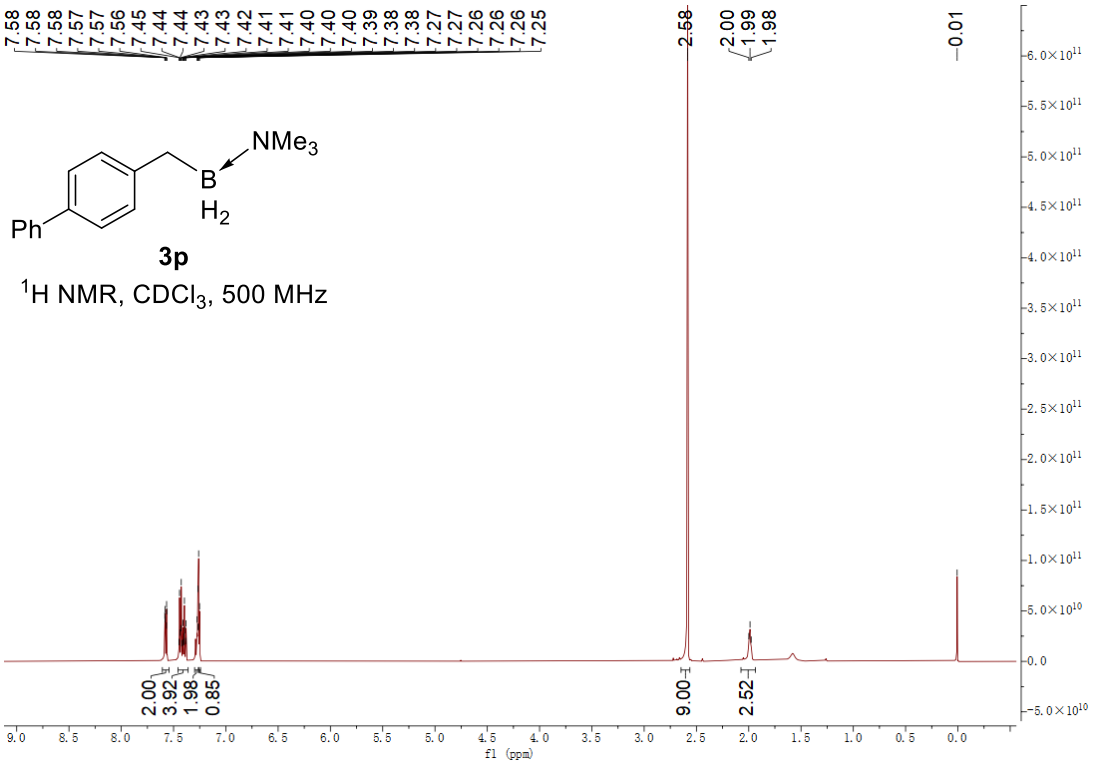
^{13}C NMR, $CDCl_3$, 126 MHz


7.26
7.19
7.19
7.19
7.18
7.18
7.17
7.17
7.17
7.16
7.16
7.15
7.01
7.01
7.00
7.00
6.99
6.98
6.98
6.97
6.97
6.96
6.96
6.95
2.55
1.96
1.94
1.93
0.00
NMe3
Ph
B
H2
3n
1H NMR, CDCl3, 500 MHz
3.72
0.91
9.00
2.30
f1 (ppm)


148.0
128.5
127.8
122.9
77.3
77.0
76.7
52.1
0.0
NMe3
Ph
B
H2
3n
13C NMR, CDCl3, 126 MHz
f1 (ppm)

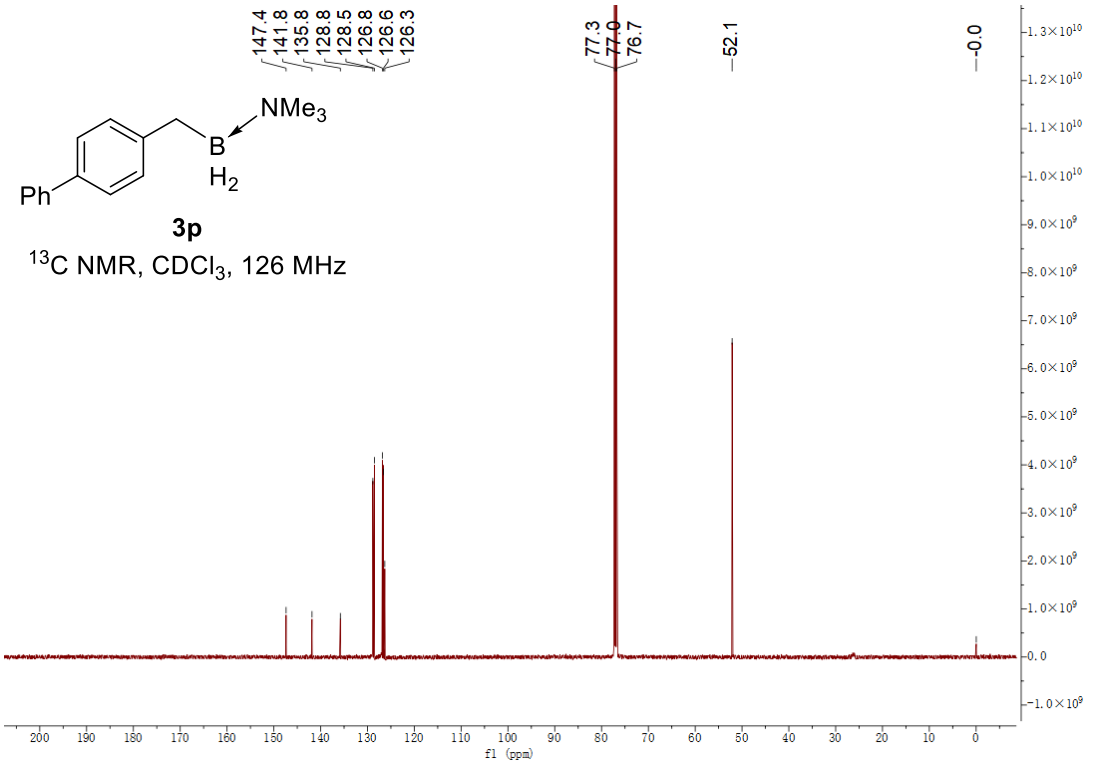

7.26
7.20
7.19
7.19
7.18
7.18
7.17
7.17
7.17
7.16
7.16
7.16
7.00
7.00
6.99
6.99
6.98
6.97
6.97
2.55
2.29
2.21
2.05
1.93
1.92
1.91
1.83
1.65
0.00
D
NMe3
Ph
B
H2
3o
1H NMR, CDCl3, 500 MHz
3.69
0.94
9.00
0.93
1.23
0.88
f1 (ppm)


148.0
128.5
127.8
122.9
77.3
77.0
76.7
52.1
0.0
D
NMe3
Ph
B
H2
3o
13C NMR, CDCl3, 126 MHz
f1 (ppm)



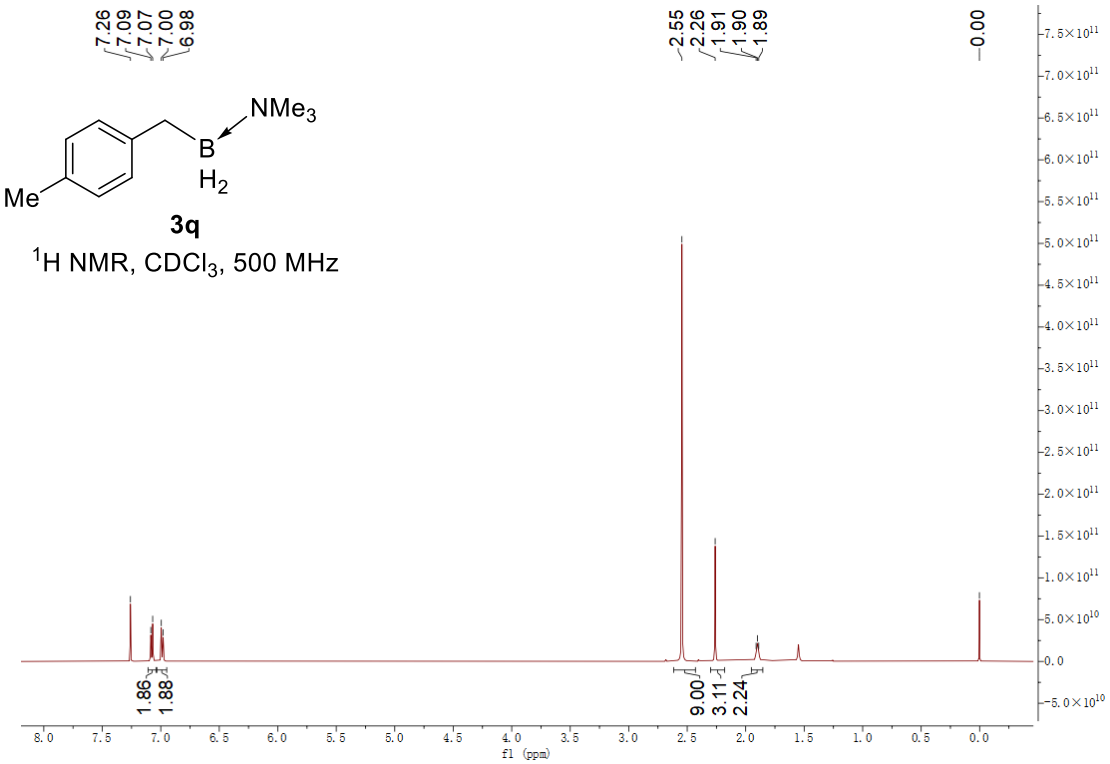
3p

^{1}H NMR, $CDCl_3$, 500 MHz



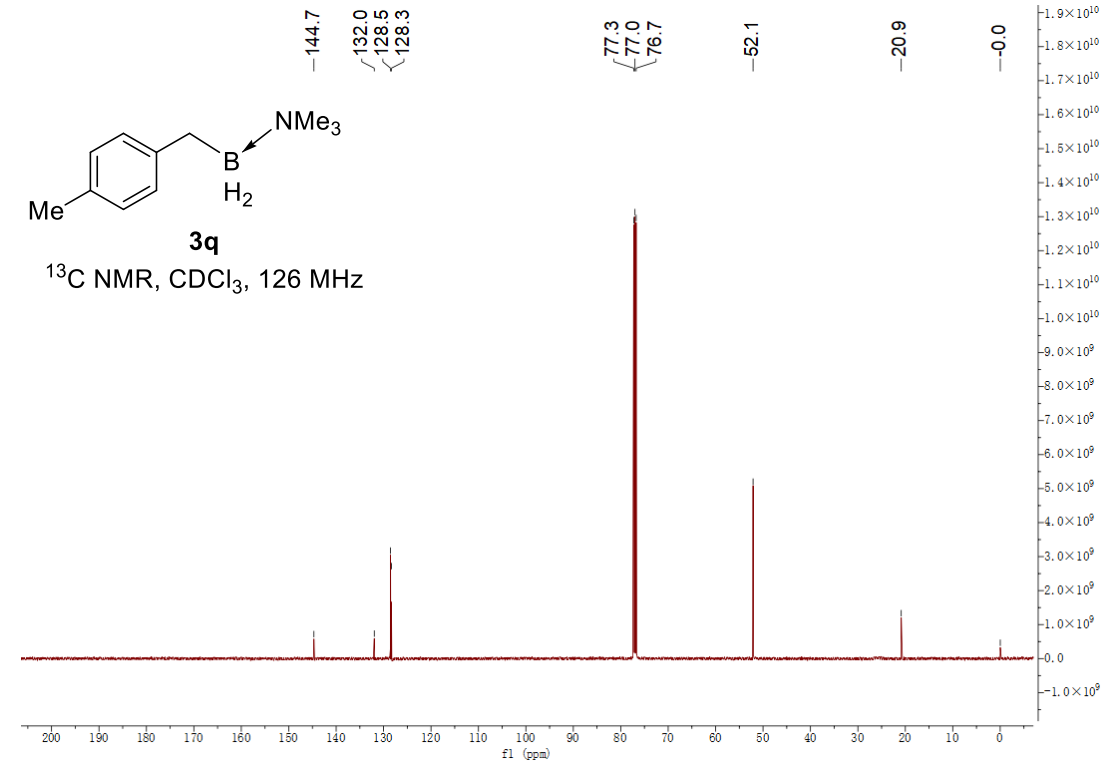
3p

^{13}C NMR, $CDCl_3$, 126 MHz



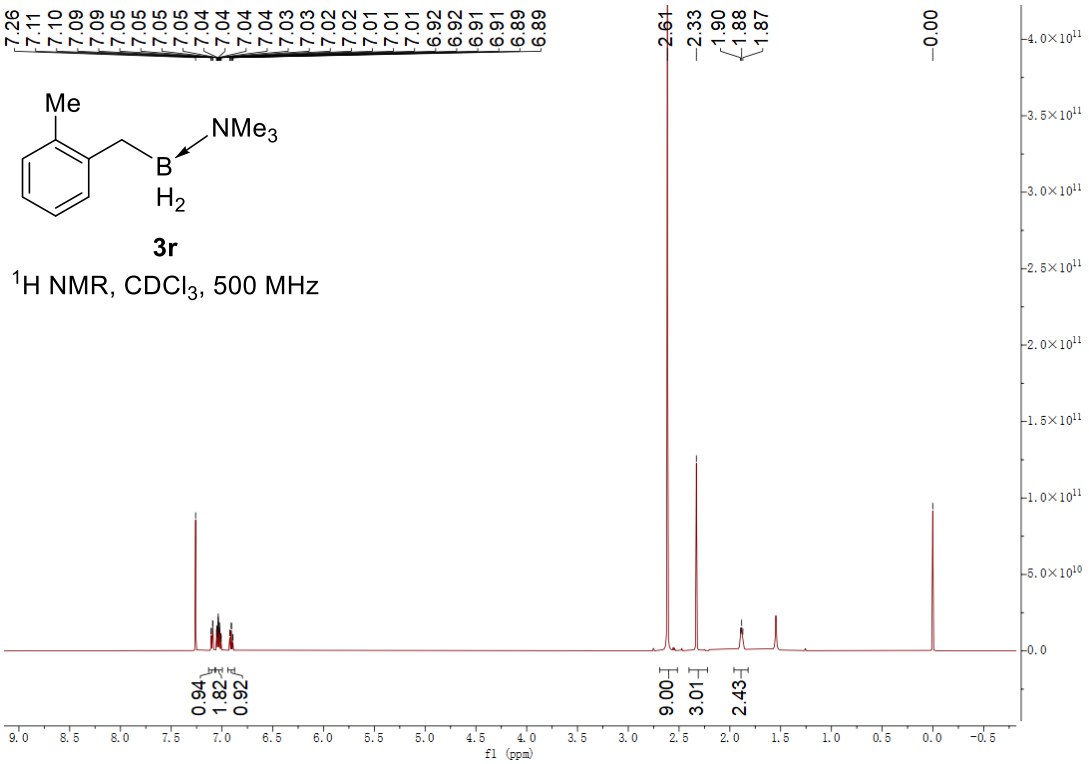
3q

^{1}H NMR, $CDCl_3$, 500 MHz



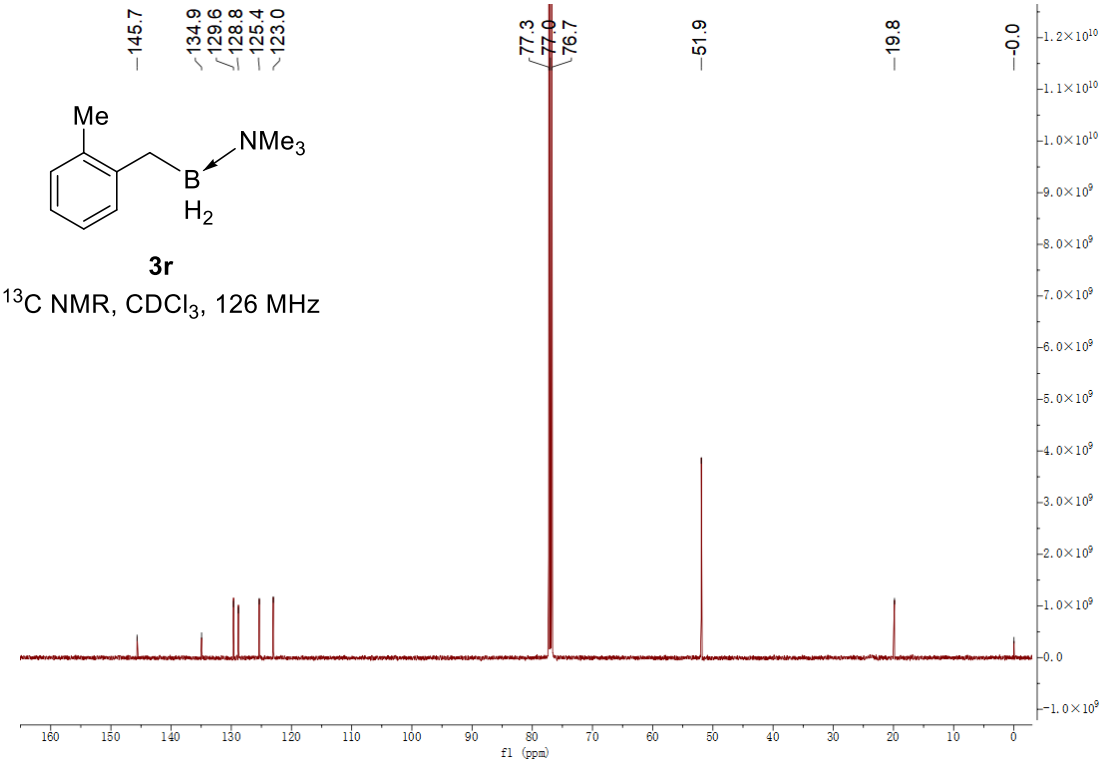
3q

^{13}C NMR, $CDCl_3$, 126 MHz



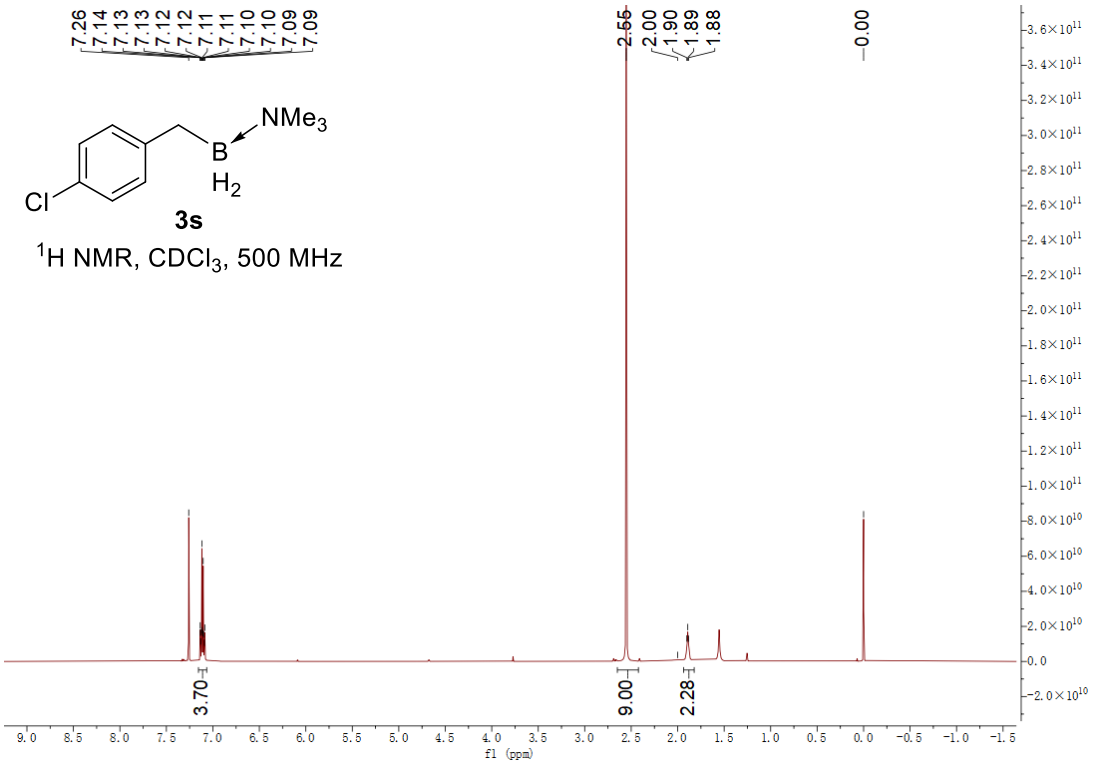
3r

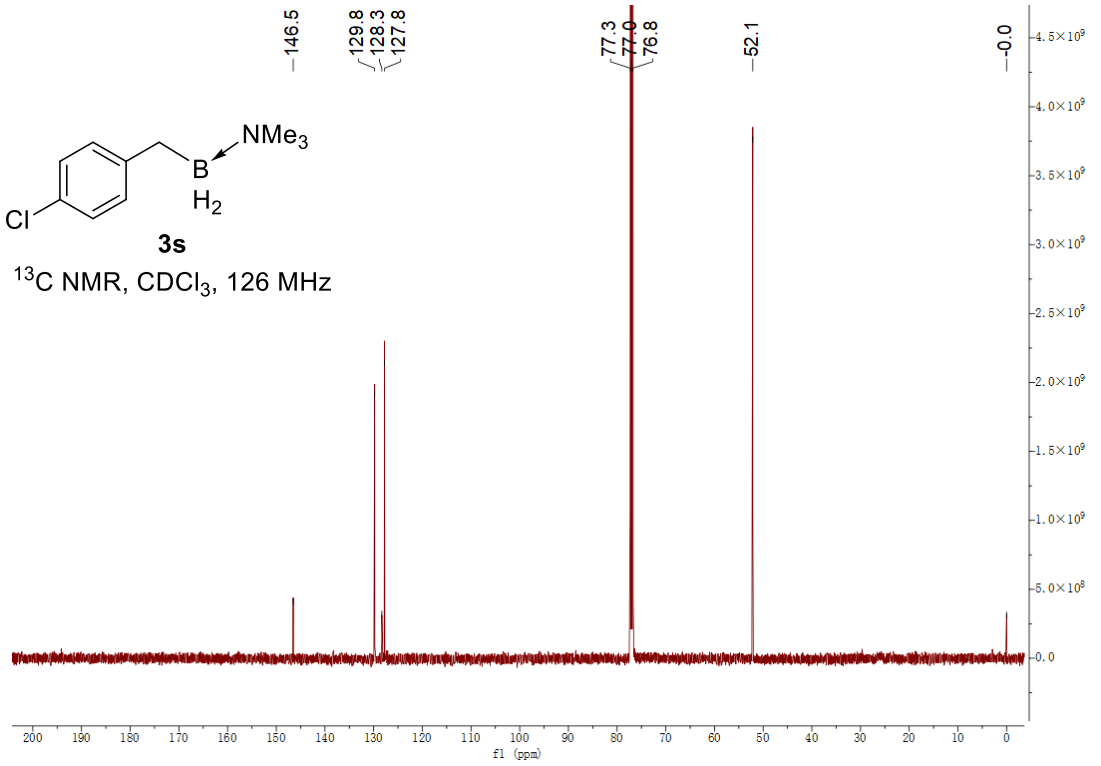
^{1}H NMR, $CDCl_3$, 500 MHz

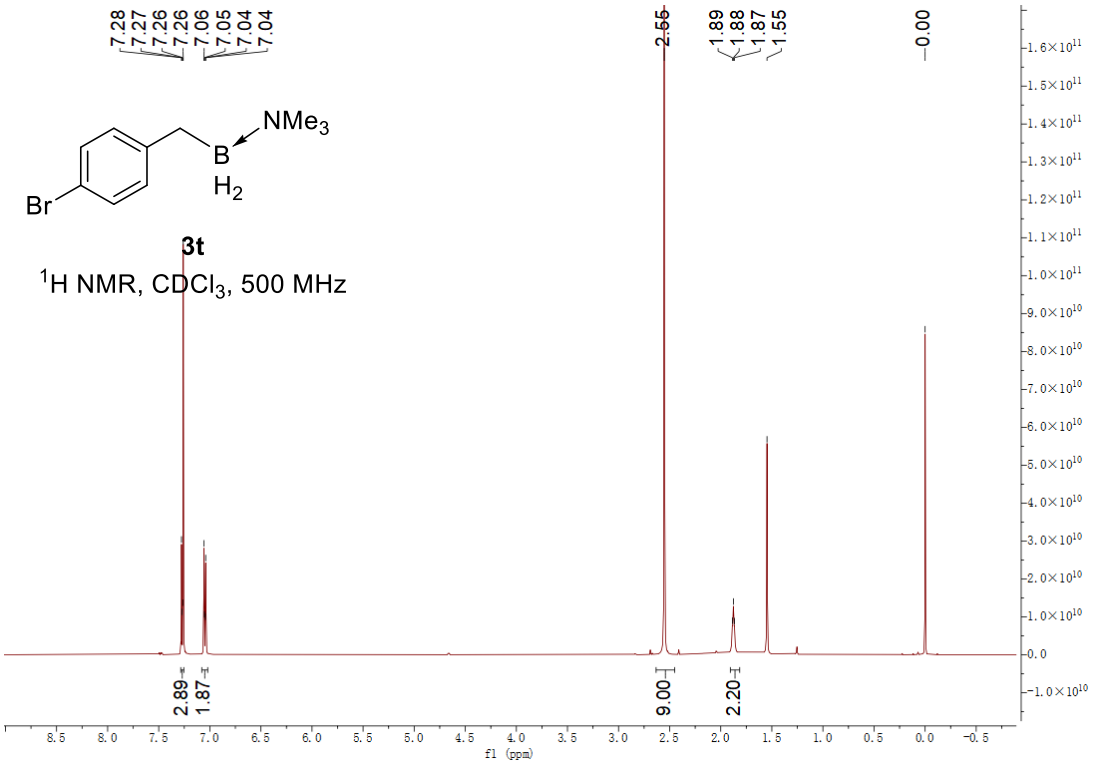


3r

^{13}C NMR, $CDCl_3$, 126 MHz

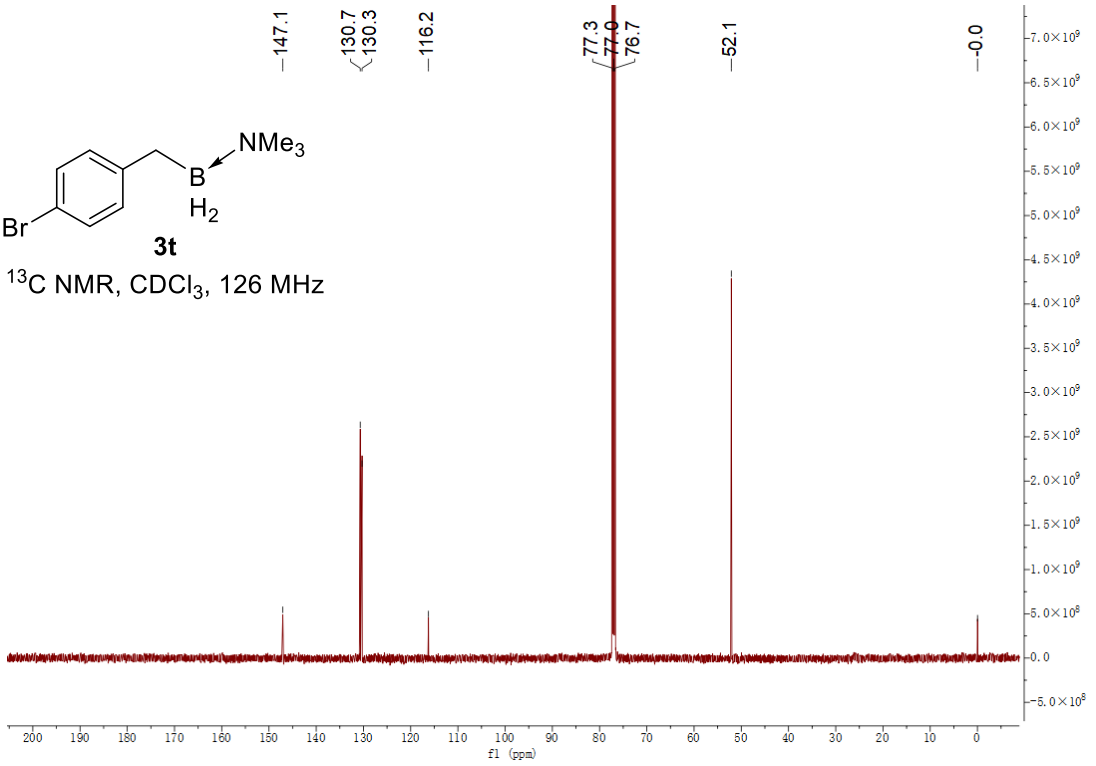

7.26
7.14
7.13
7.13
7.12
7.12
7.11
7.11
7.10
7.10
7.09
7.09
2.55
2.00
1.90
1.89
1.88
0.00
NMe3
B
H2
Cl
3s
1H NMR, CDCl3, 500 MHz
3.70
9.00
2.28
f1 (ppm)


146.5
129.8
128.3
127.8
77.3
77.0
76.8
52.1
0.0
NMe3
B
H2
Cl
3s
13C NMR, CDCl3, 126 MHz
f1 (ppm)



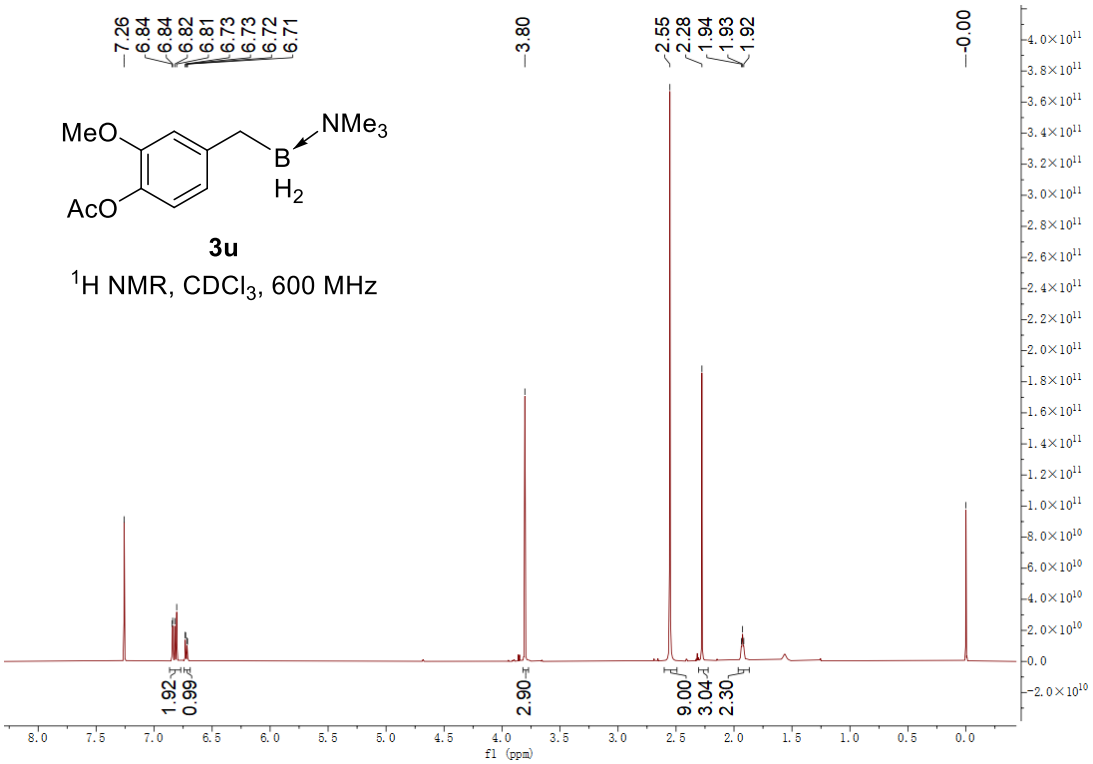
3t

^{1}H NMR, $CDCl_3$, 500 MHz



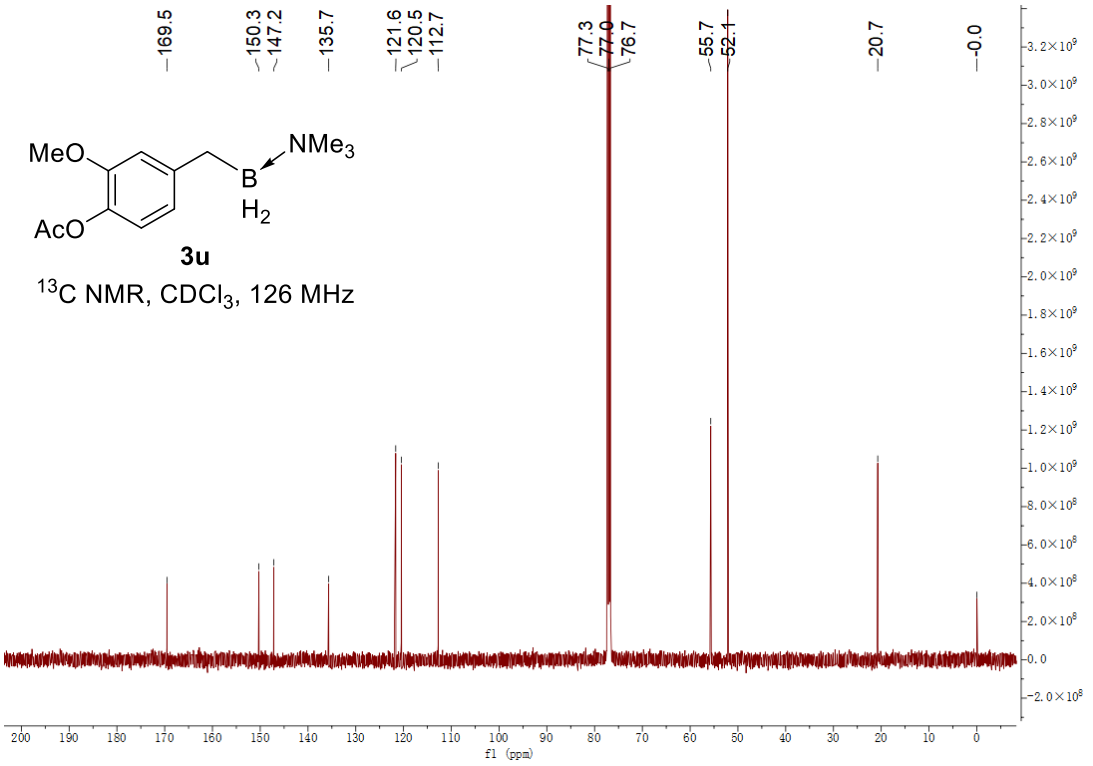
3t

^{13}C NMR, $CDCl_3$, 126 MHz



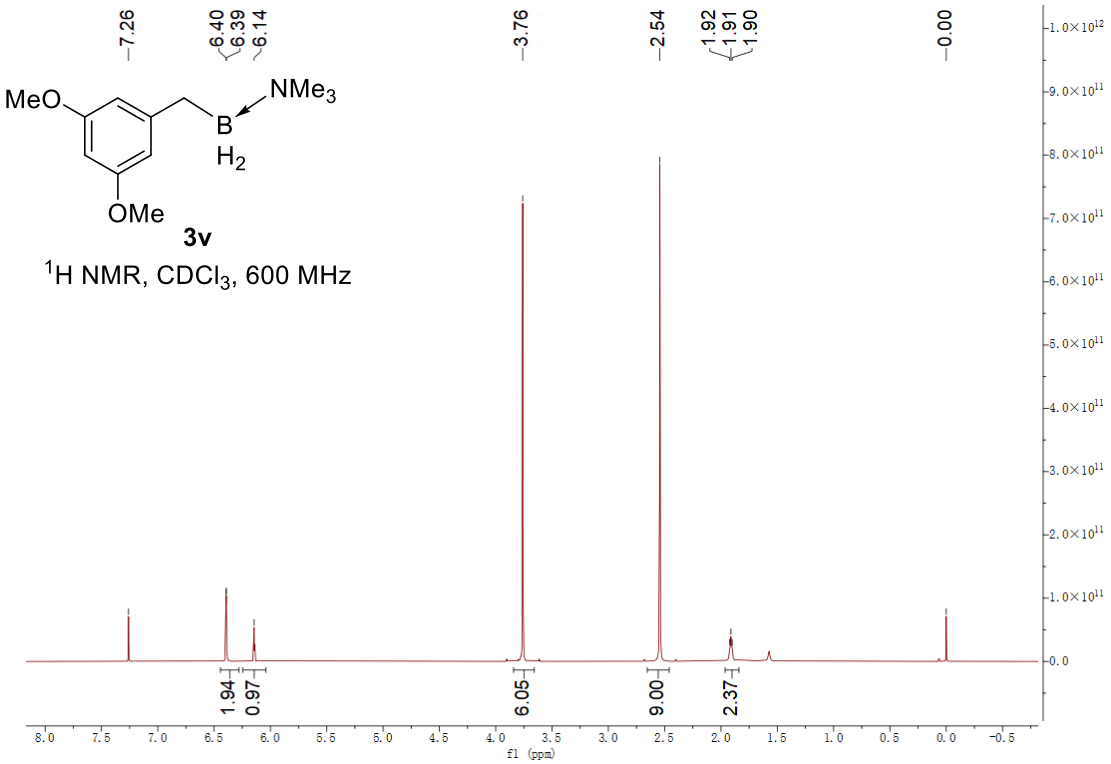
3u

^{1}H NMR, $CDCl_3$, 600 MHz



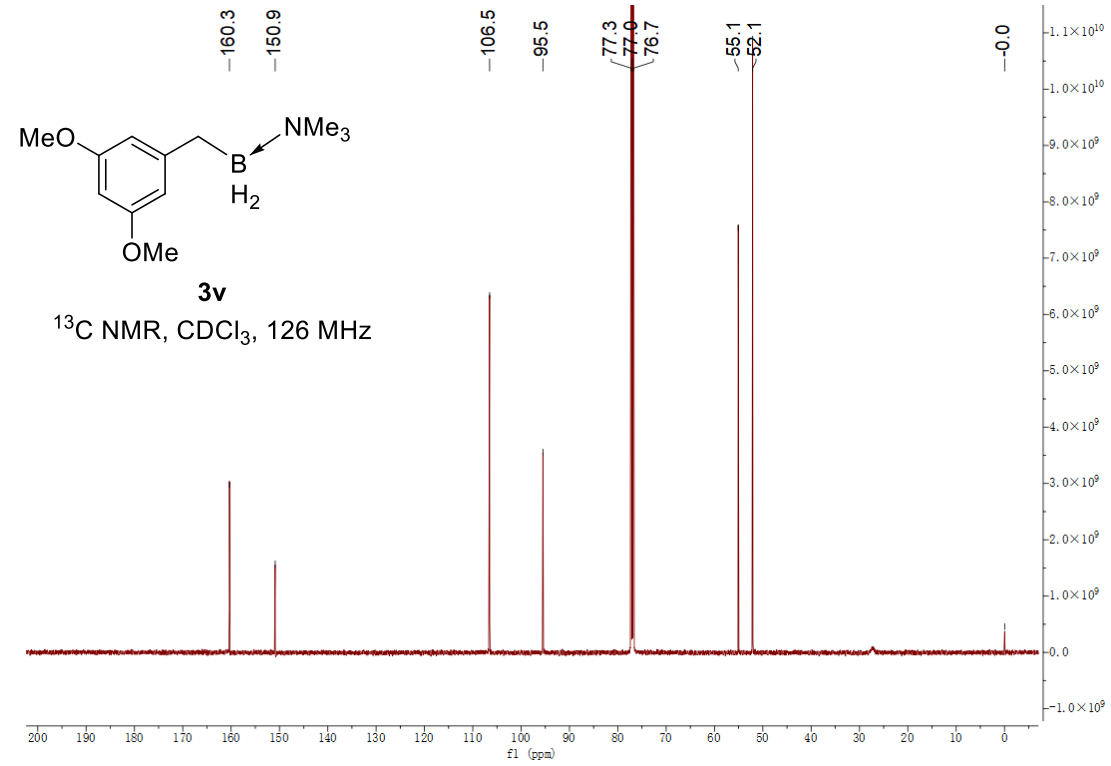
3u

^{13}C NMR, $CDCl_3$, 126 MHz



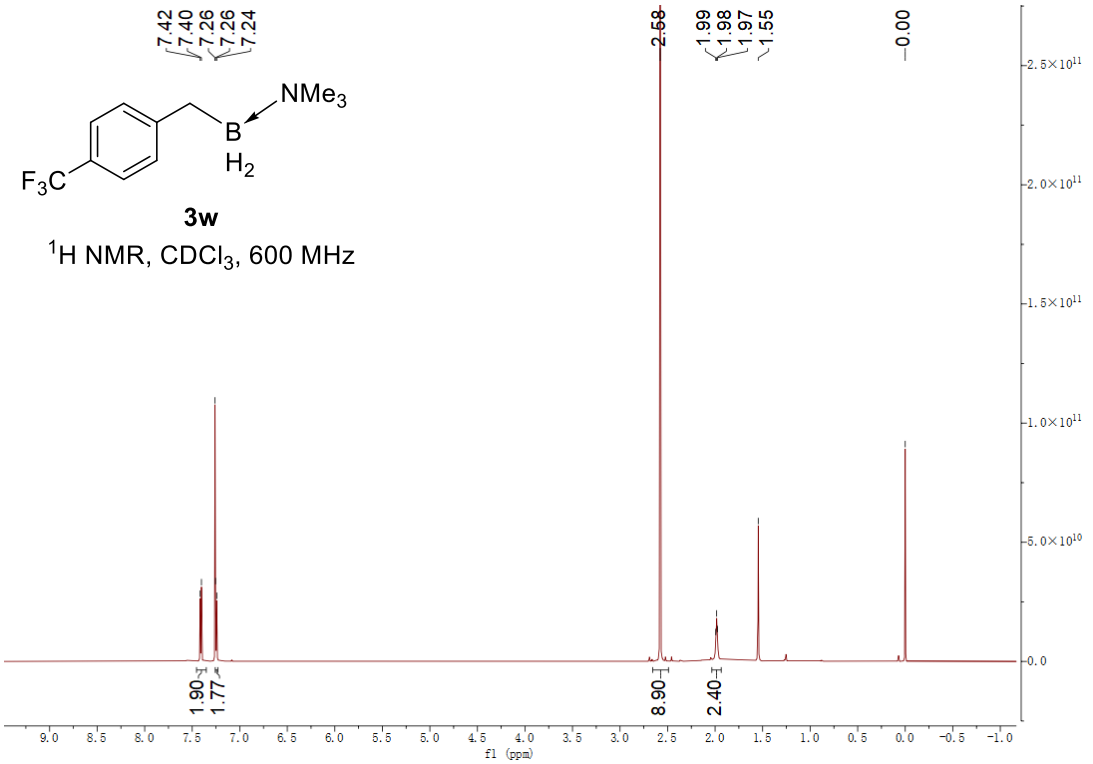
3v

^{1}H NMR, $CDCl_3$, 600 MHz



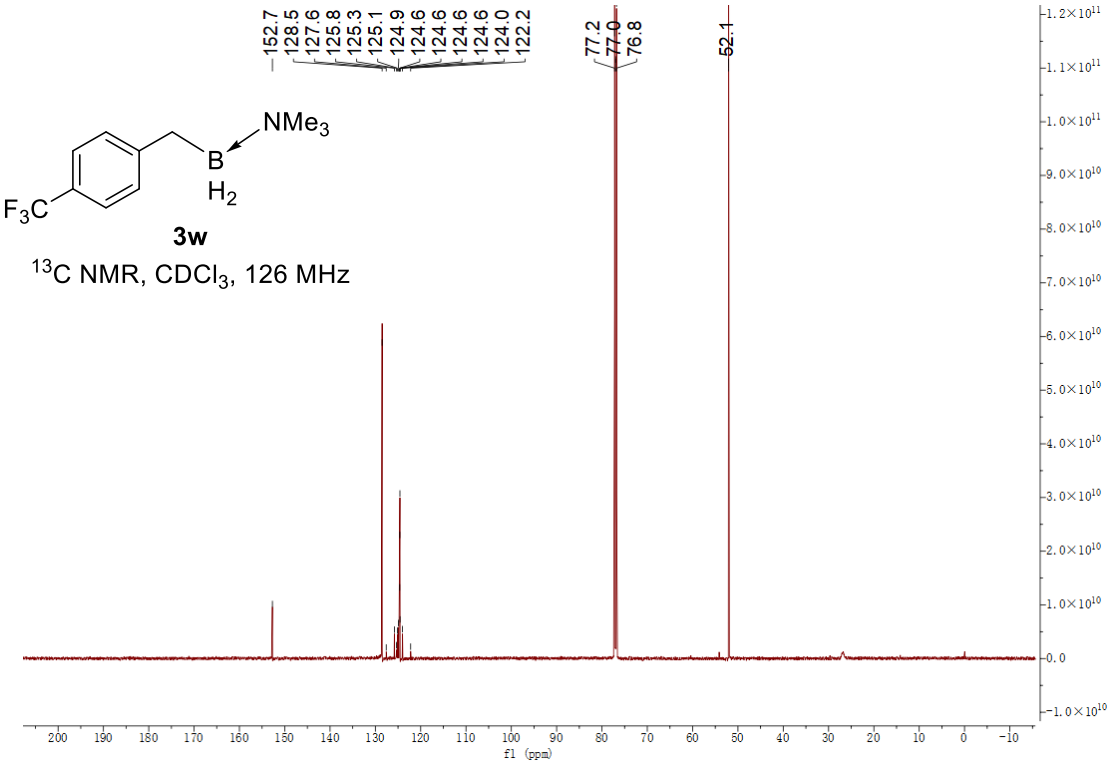
3v

^{13}C NMR, $CDCl_3$, 126 MHz



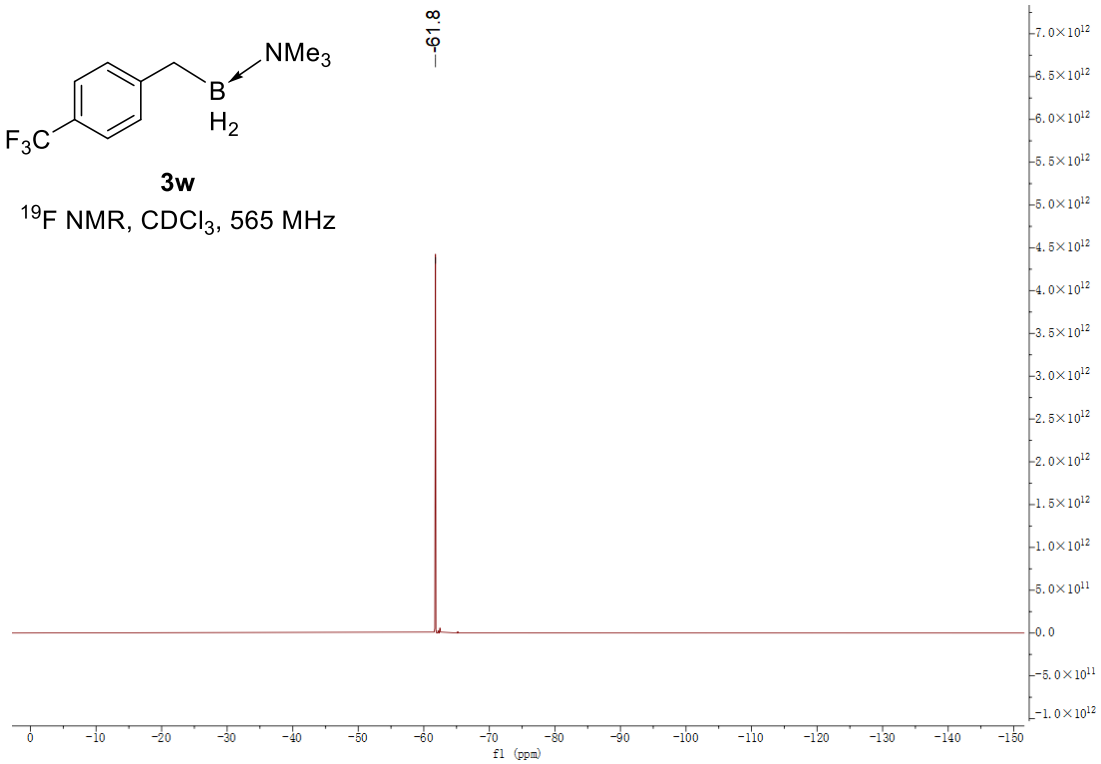
3w

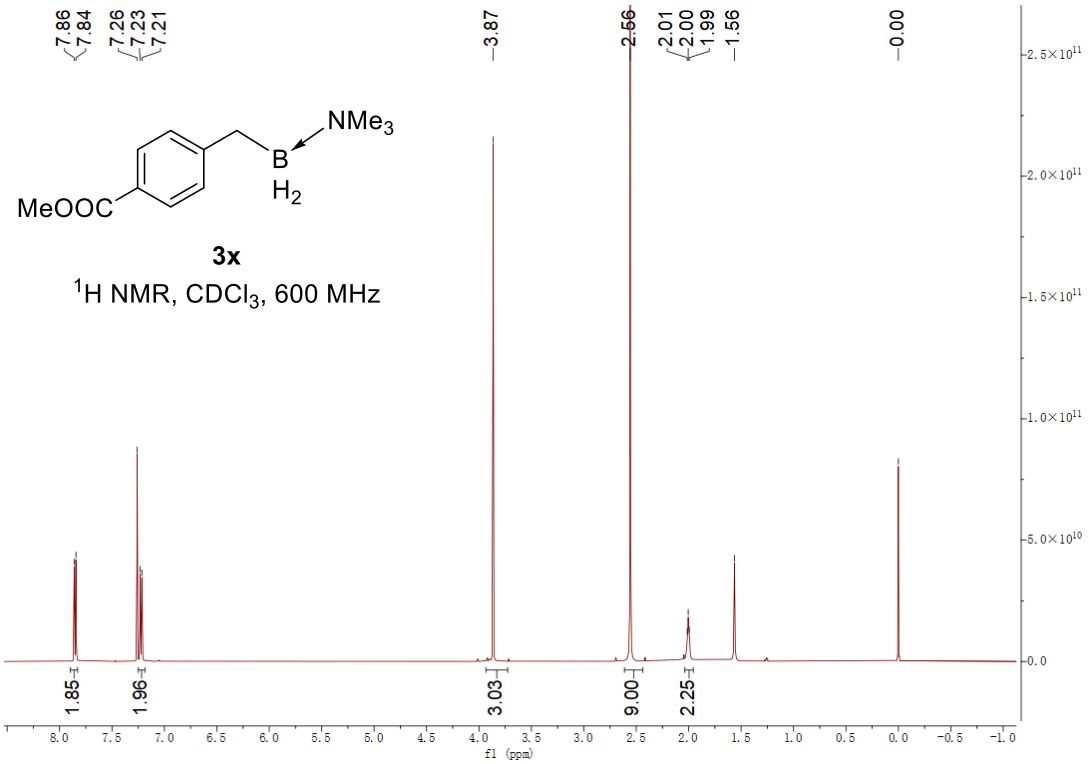
^{1}H NMR, $CDCl_3$, 600 MHz



3w

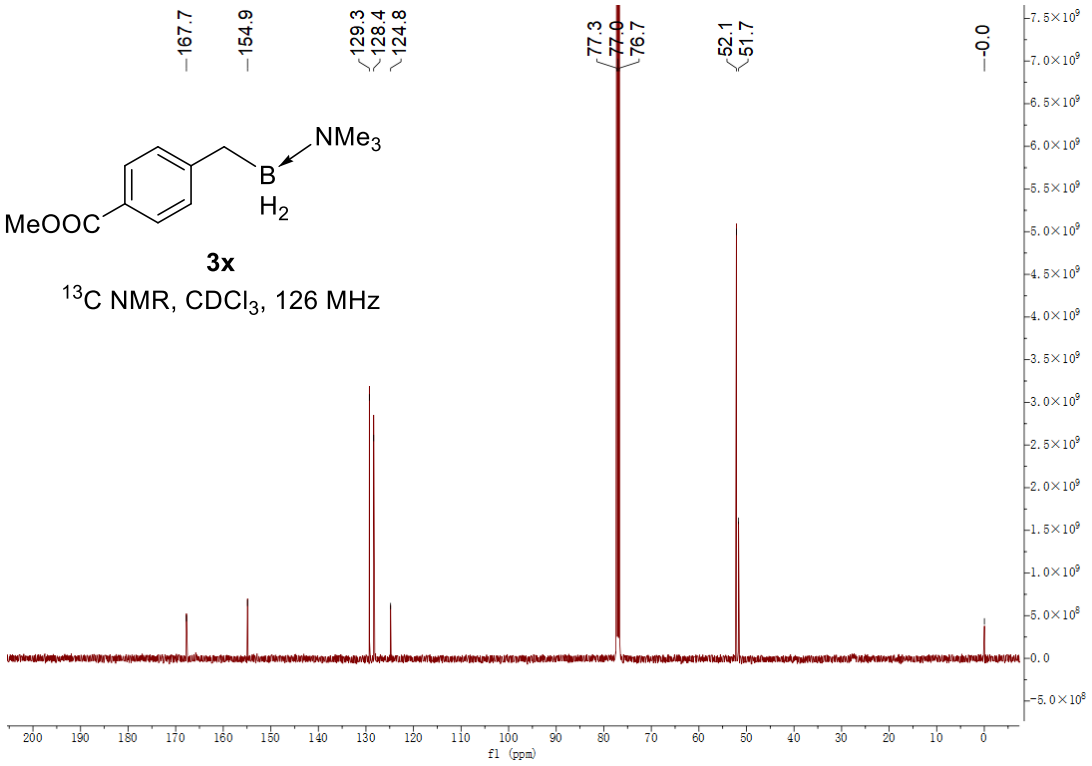
^{13}C NMR, $CDCl_3$, 126 MHz


NMe3
B
H2
F3C
3w
19F NMR, CDCl3, 565 MHz
-61.8
f1 (ppm)



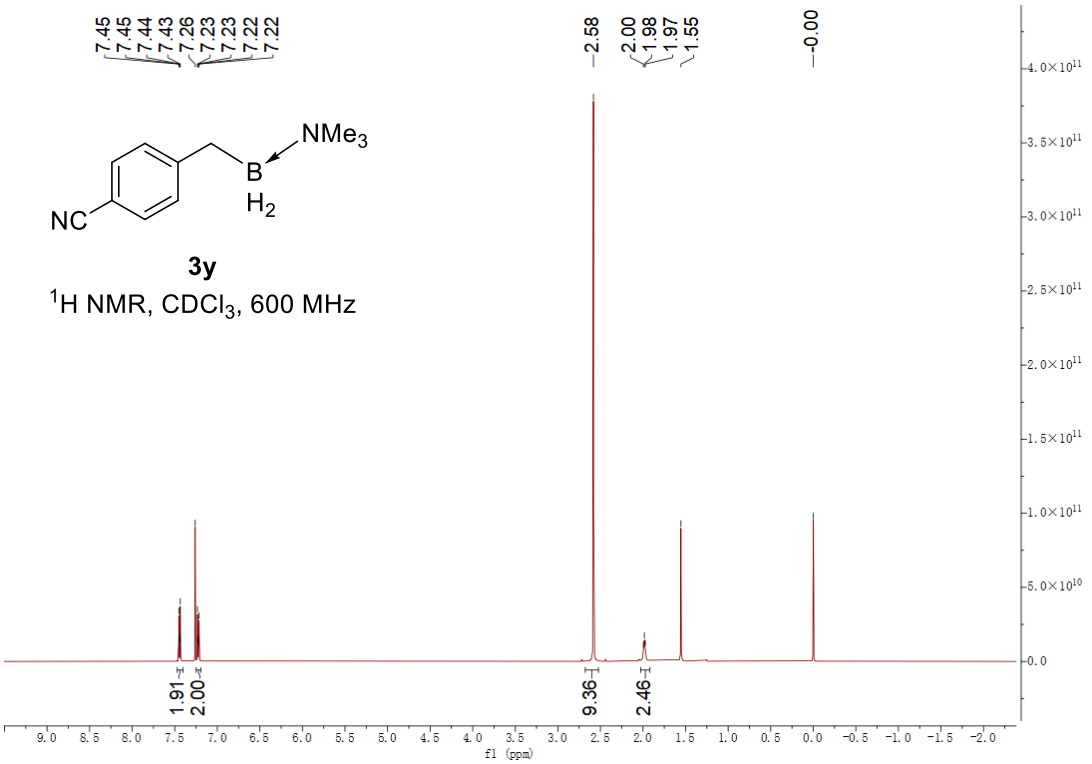
3x

^{1}H NMR, $CDCl_3$, 600 MHz



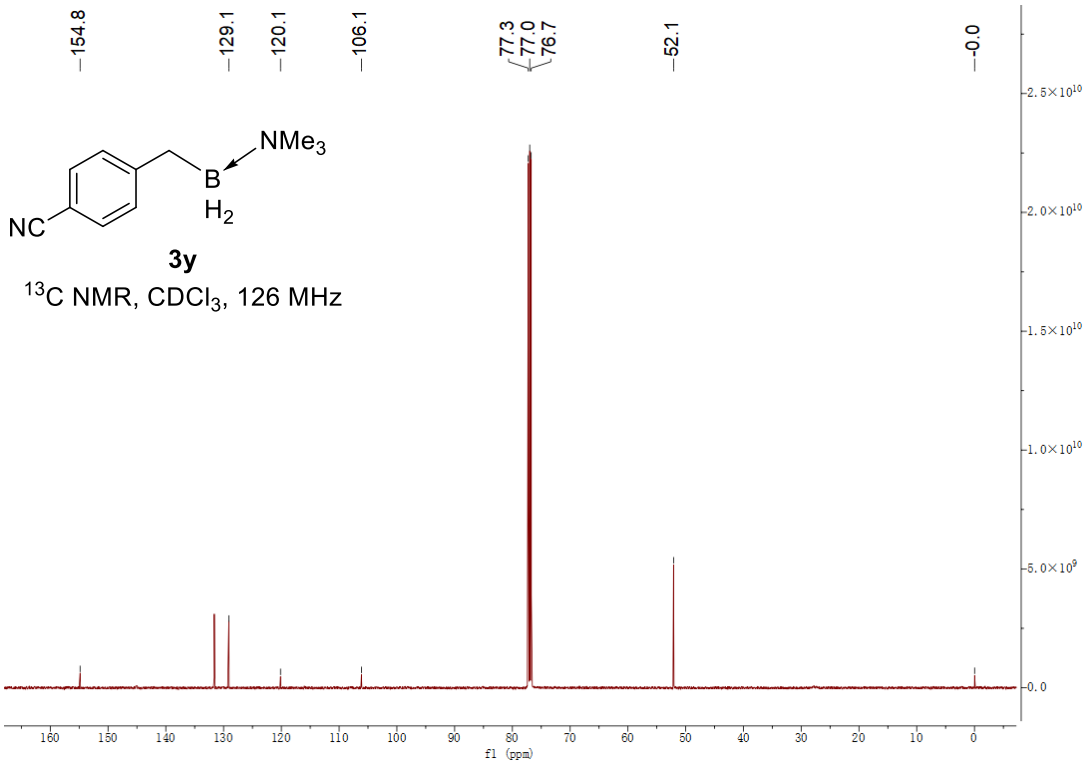
3x

^{13}C NMR, $CDCl_3$, 126 MHz



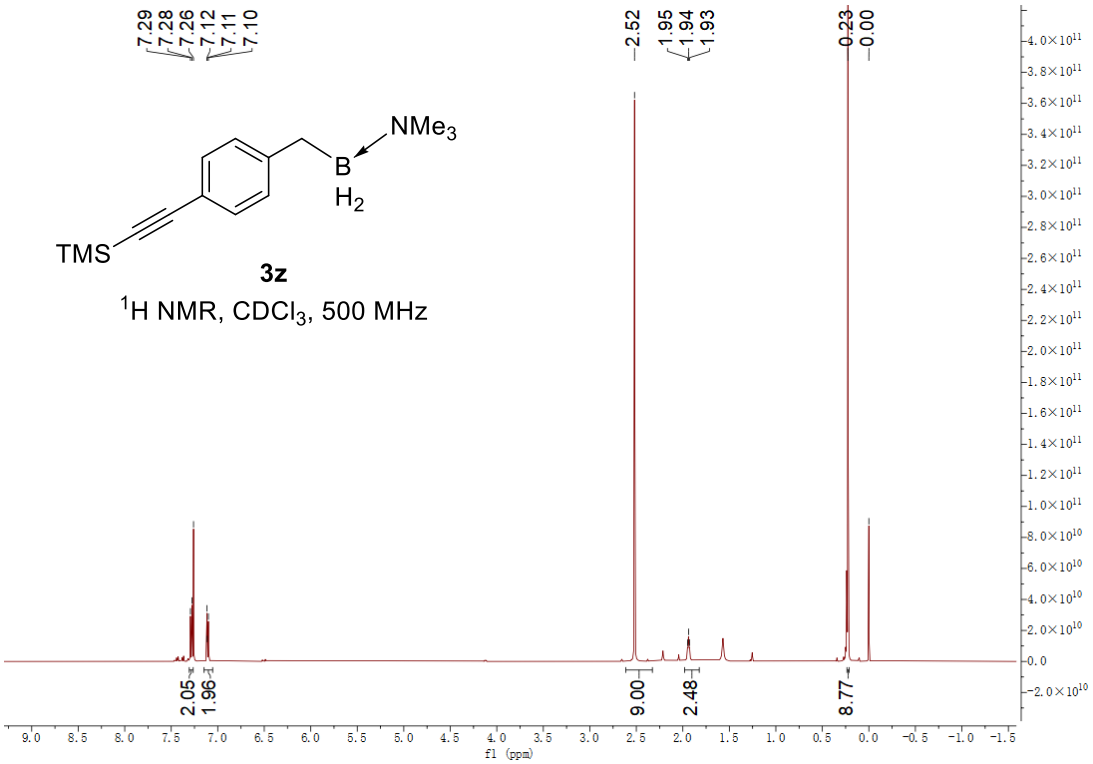
3y

^{1}H NMR, $CDCl_3$, 600 MHz



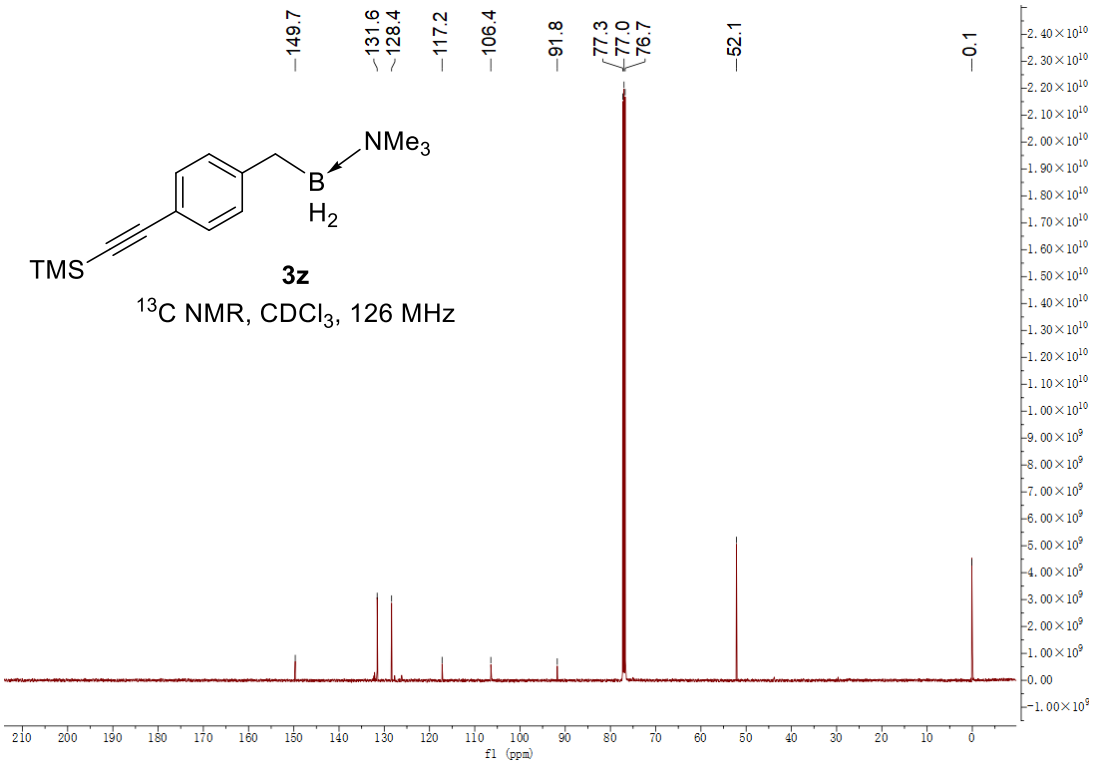
3y

^{13}C NMR, $CDCl_3$, 126 MHz



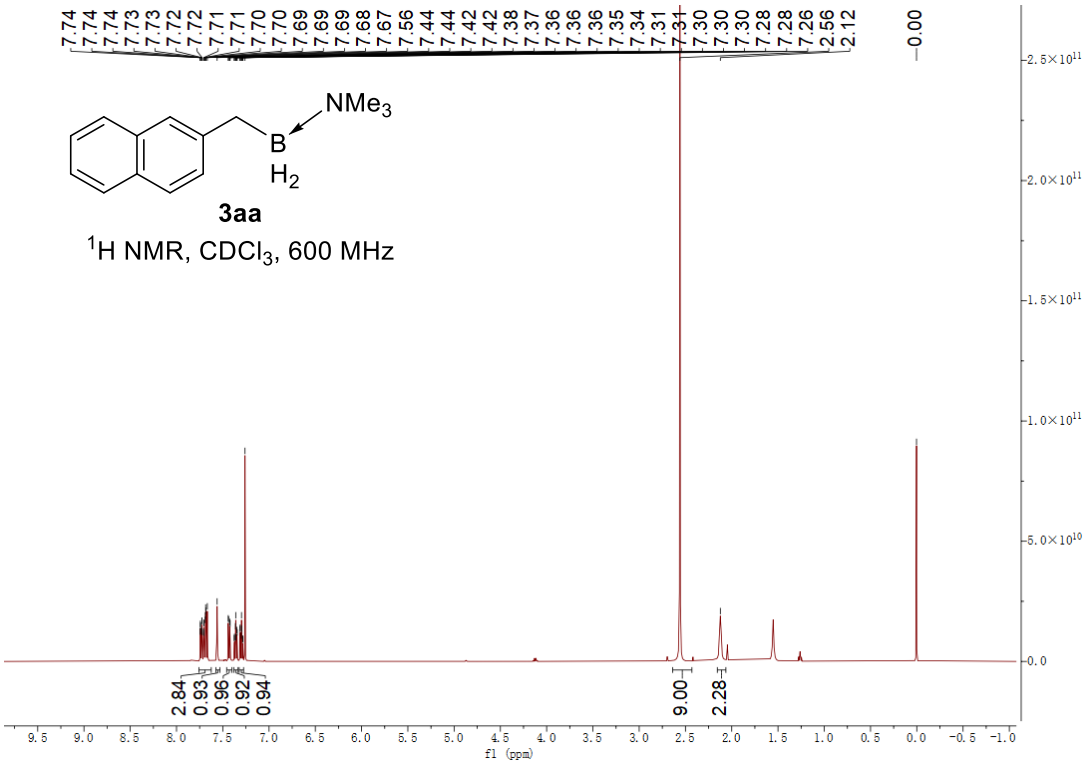
3z

^{1}H NMR, $CDCl_3$, 500 MHz



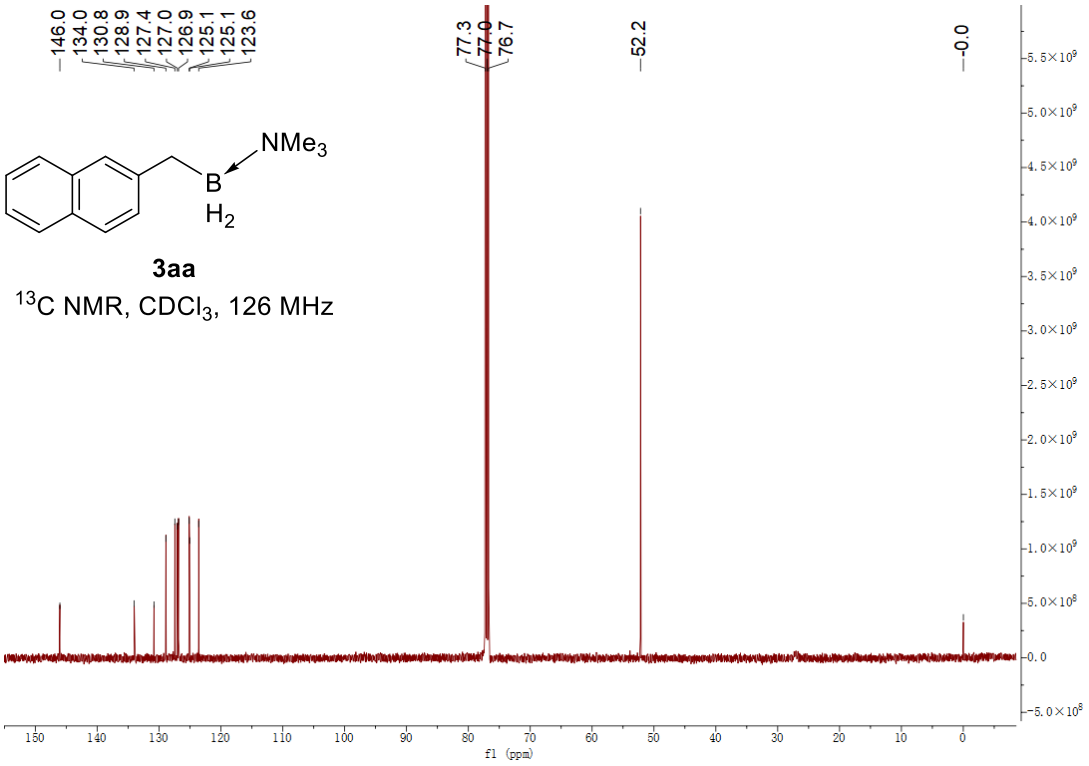
3z

^{13}C NMR, $CDCl_3$, 126 MHz



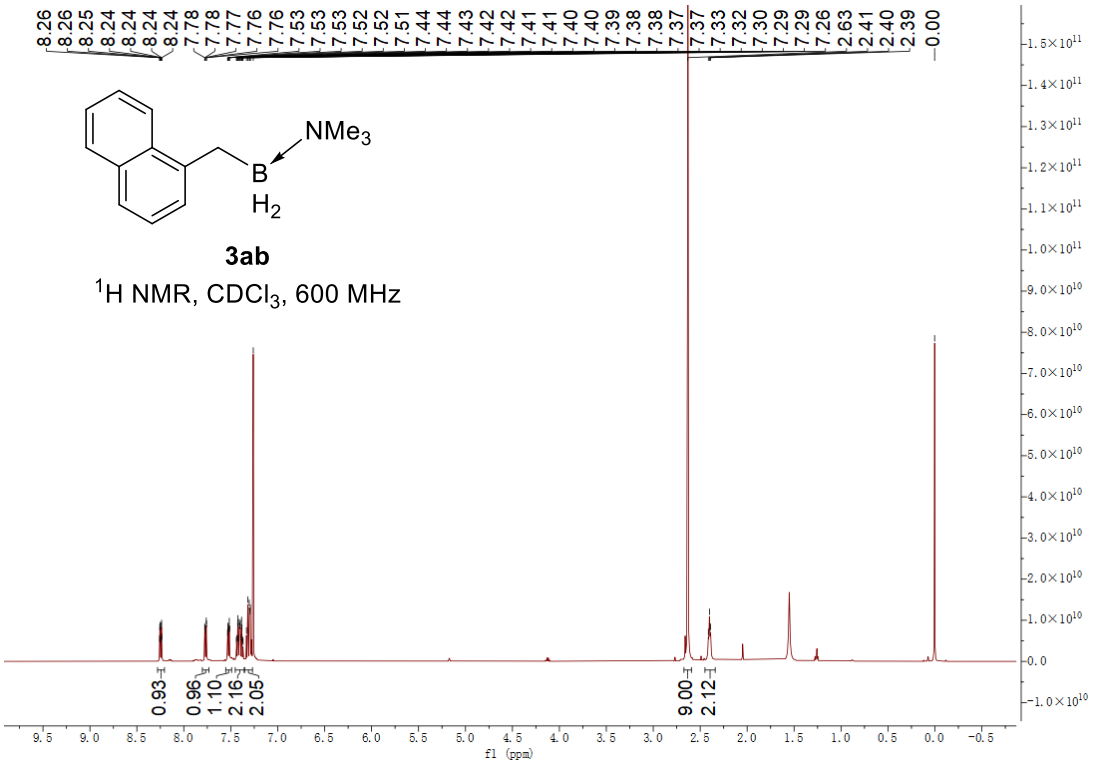
3aa

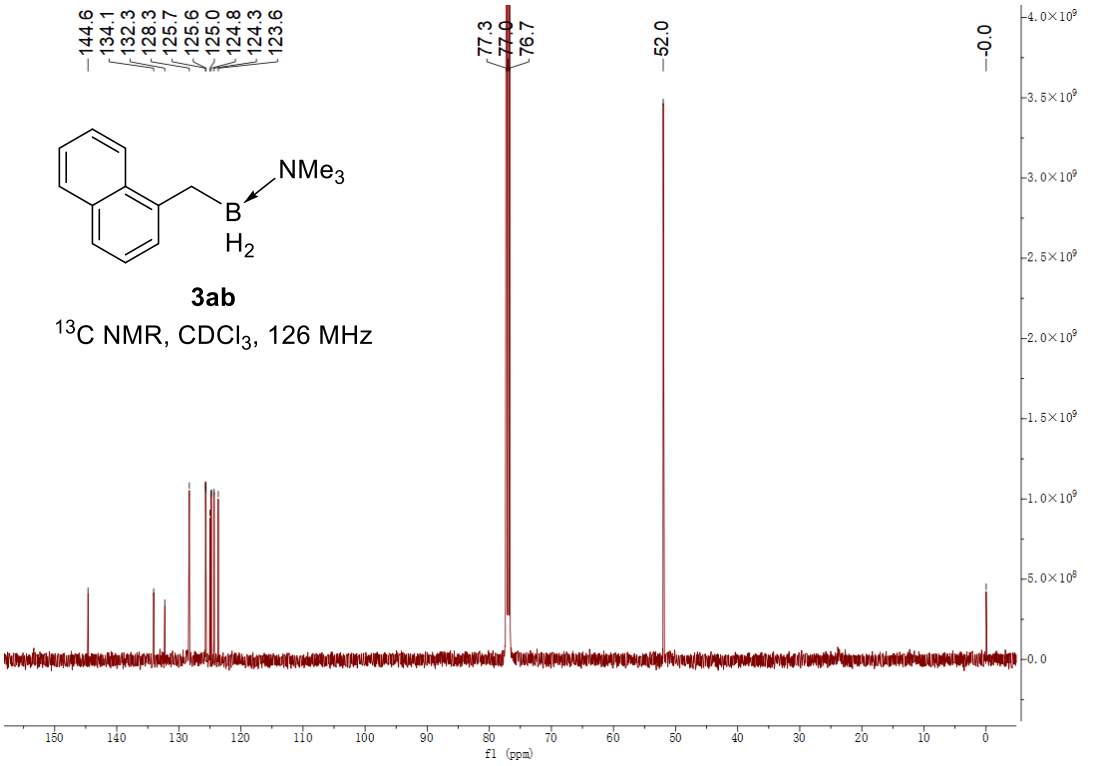
^{1}H NMR, $CDCl_3$, 600 MHz

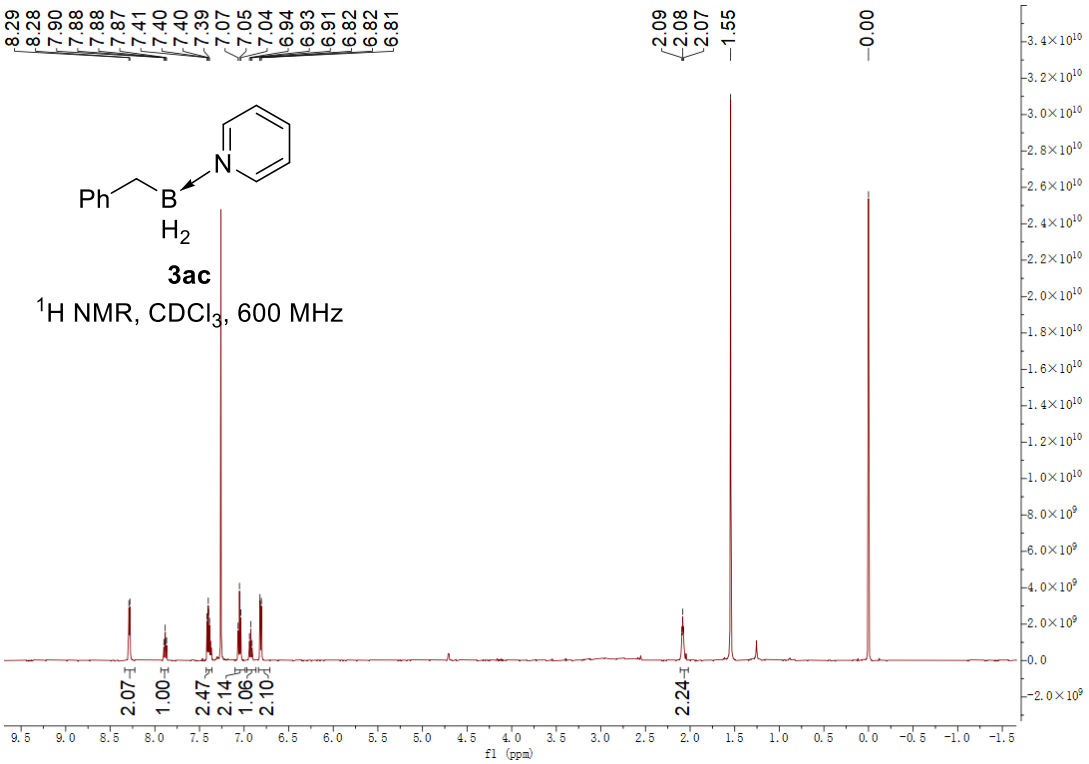


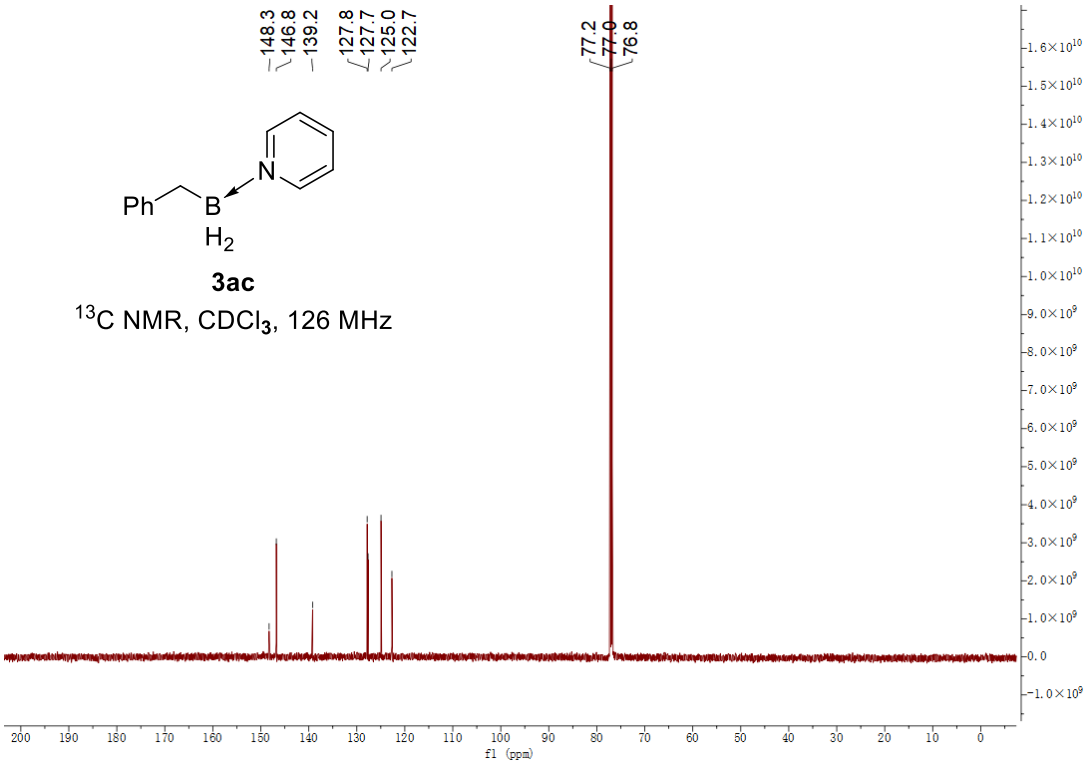
3aa

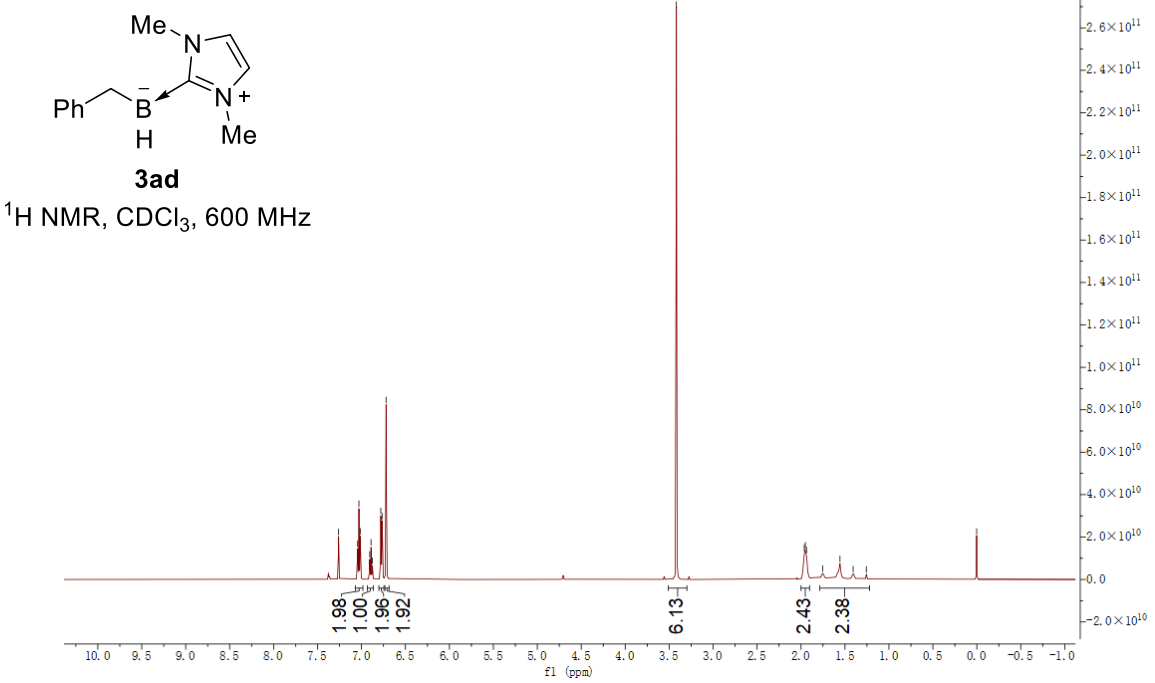
^{13}C NMR, $CDCl_3$, 126 MHz


8.26
8.26
8.25
8.24
8.24
8.24
8.24
7.78
7.78
7.77
7.76
7.76
7.53
7.53
7.52
7.52
7.51
7.44
7.44
7.43
7.42
7.42
7.41
7.41
7.40
7.40
7.39
7.38
7.38
7.37
7.37
7.33
7.32
7.30
7.29
7.29
7.26
2.63
2.41
2.40
2.39
0.00
NMe3
B
H2
3ab
1H NMR, CDCl3, 600 MHz
0.93
0.96
1.10
2.16
2.05
9.00
2.12
f1 (ppm)


144.6
134.1
132.3
128.3
125.7
125.6
125.0
124.8
124.3
123.6
77.3
77.0
76.7
52.0
0.0
NMe3
B
H2
3ab
13C NMR, CDCl3, 126 MHz
f1 (ppm)

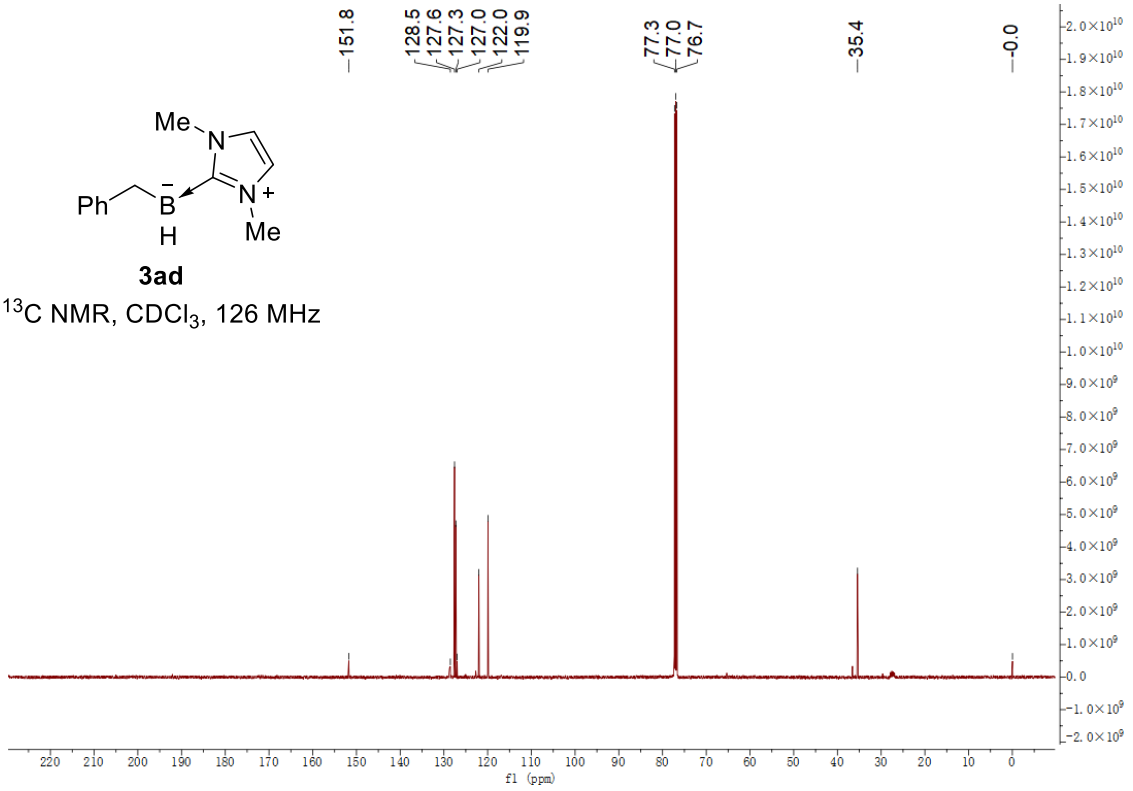

8.29
8.28
7.90
7.88
7.88
7.87
7.41
7.40
7.40
7.39
7.07
7.05
7.04
6.94
6.93
6.91
6.82
6.82
6.81
2.09
2.08
2.07
1.55
0.00
Ph
B
N
H2
3ac
1H NMR, CDCl3, 600 MHz
2.07
1.00
2.47
2.14
1.06
2.10
2.24
f1 (ppm)


148.3
146.8
139.2
127.8
127.7
125.0
122.7
77.2
77.0
76.8
Ph
B
N
H2
3ac
13C NMR, CDCl3, 126 MHz
f1 (ppm)



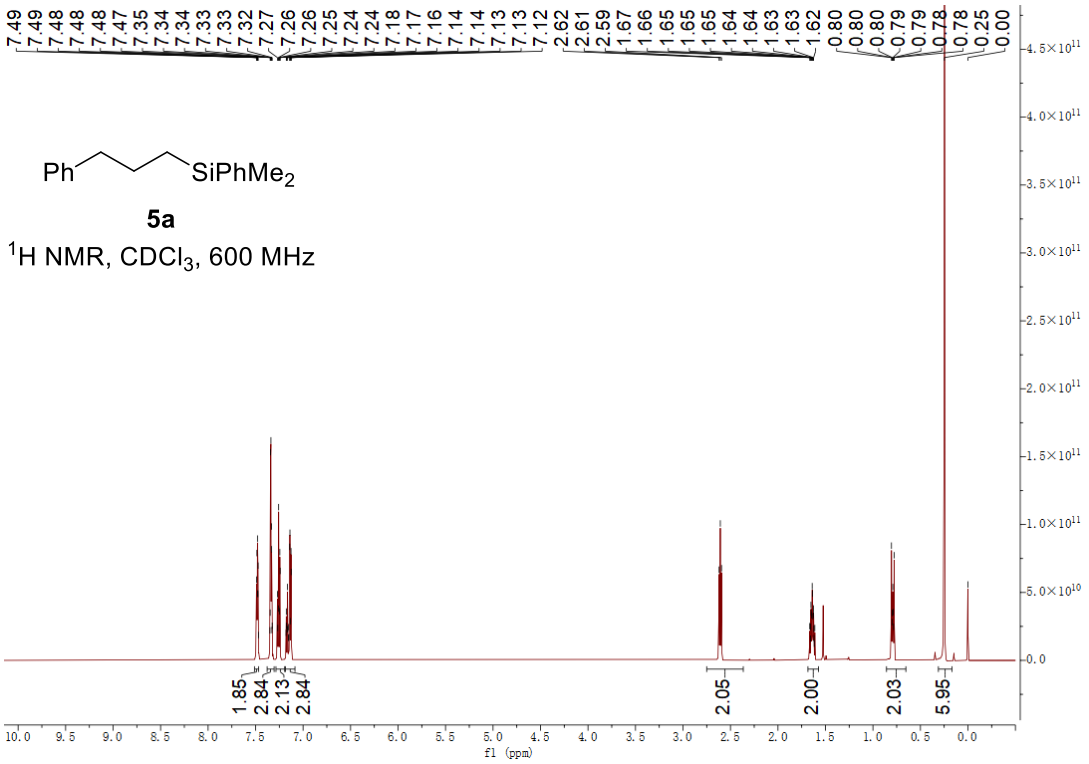
3ad

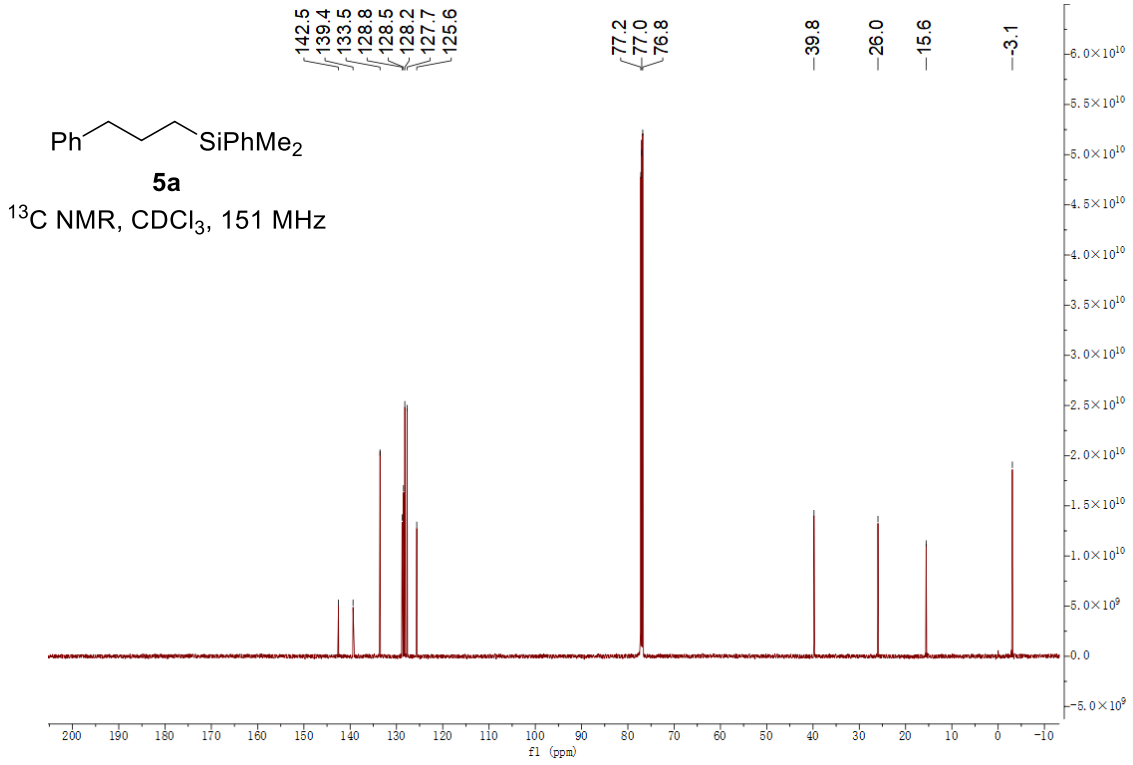
^{1}H NMR, $CDCl_3$, 600 MHz

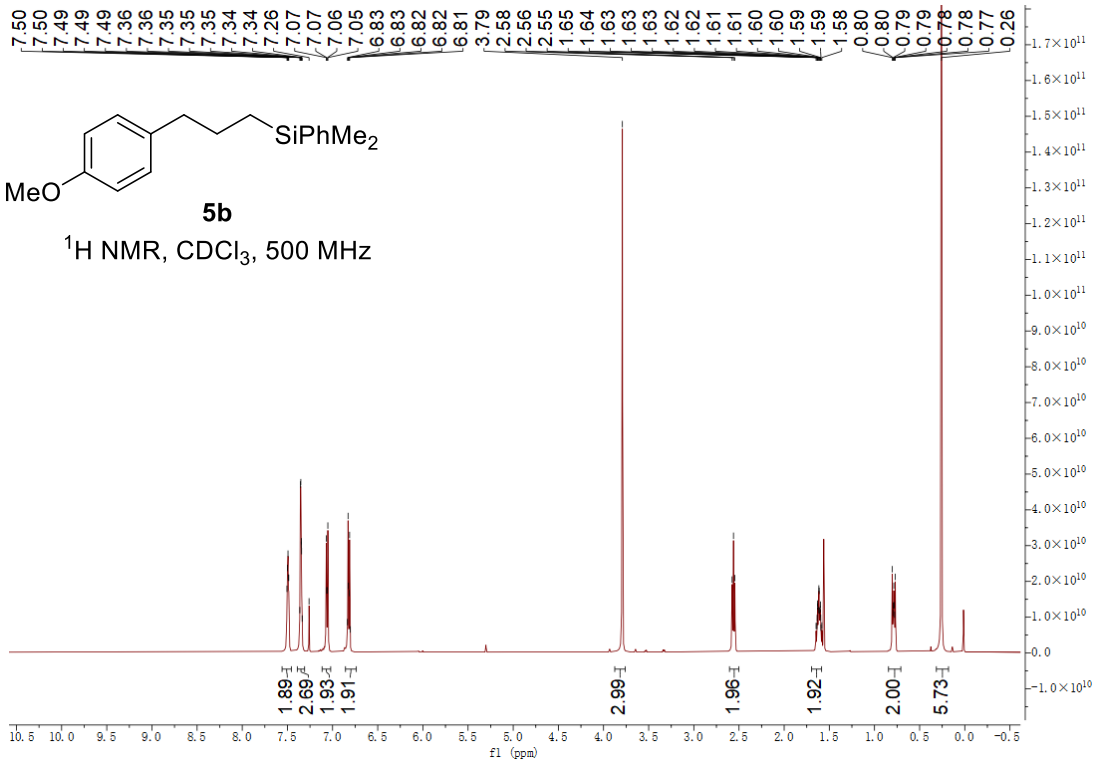


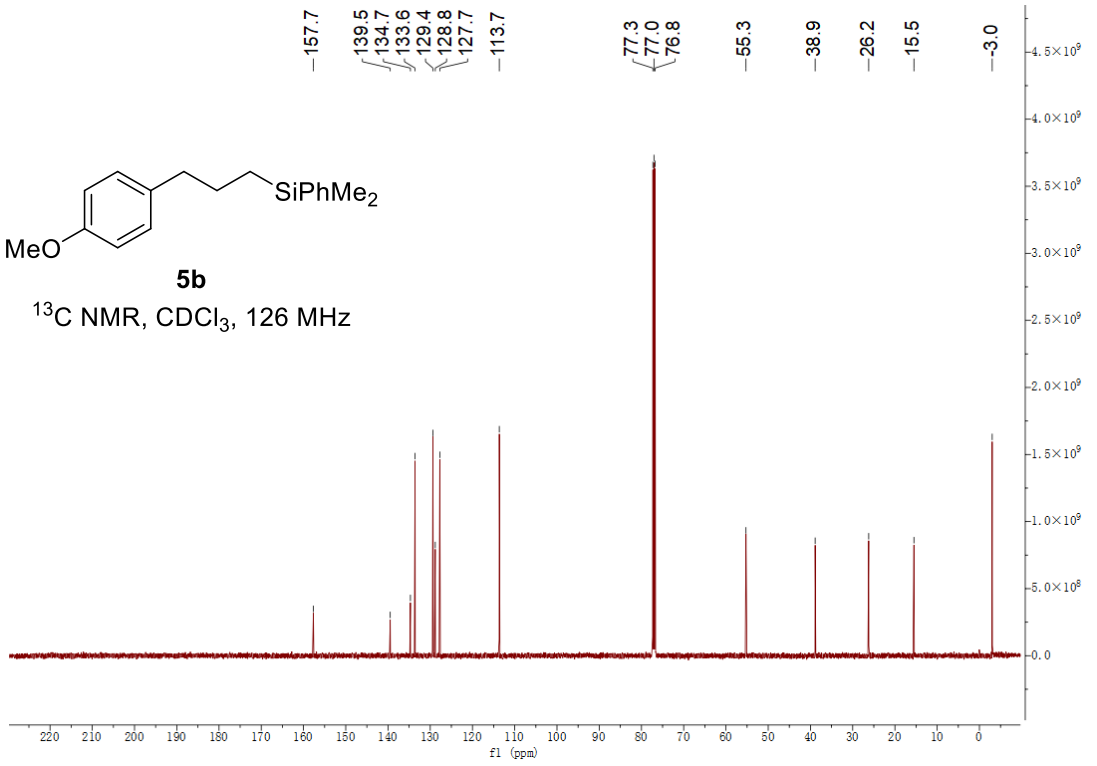
3ad

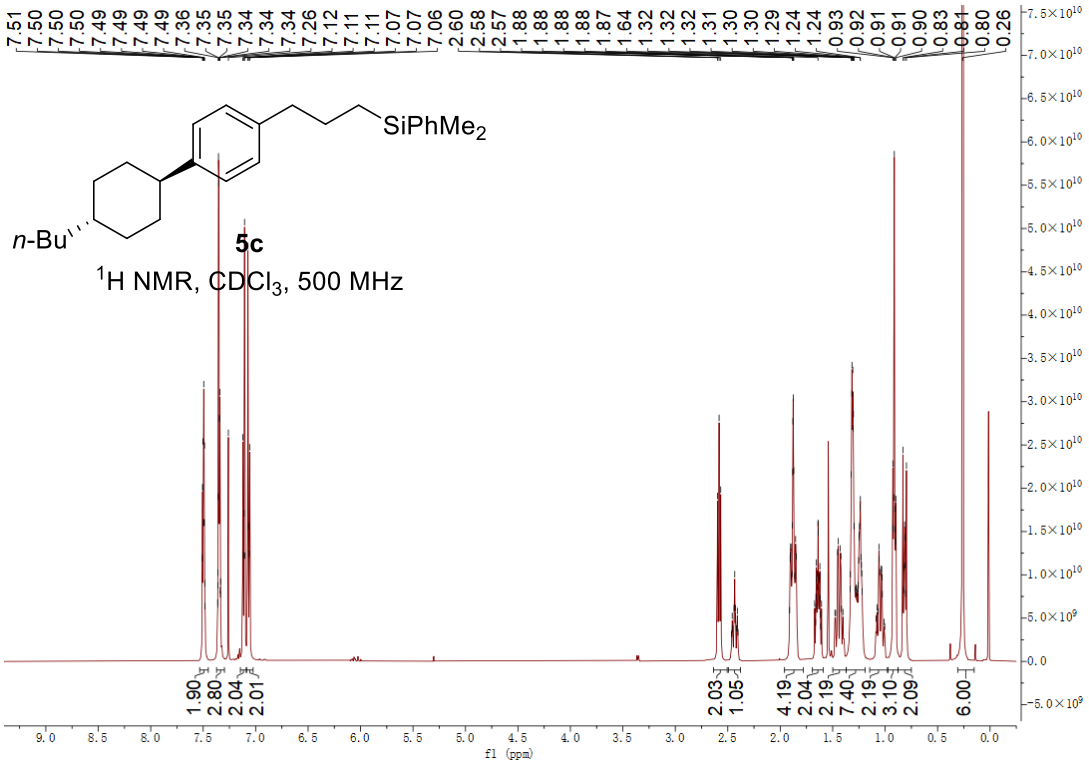
^{13}C NMR, $CDCl_3$, 126 MHz

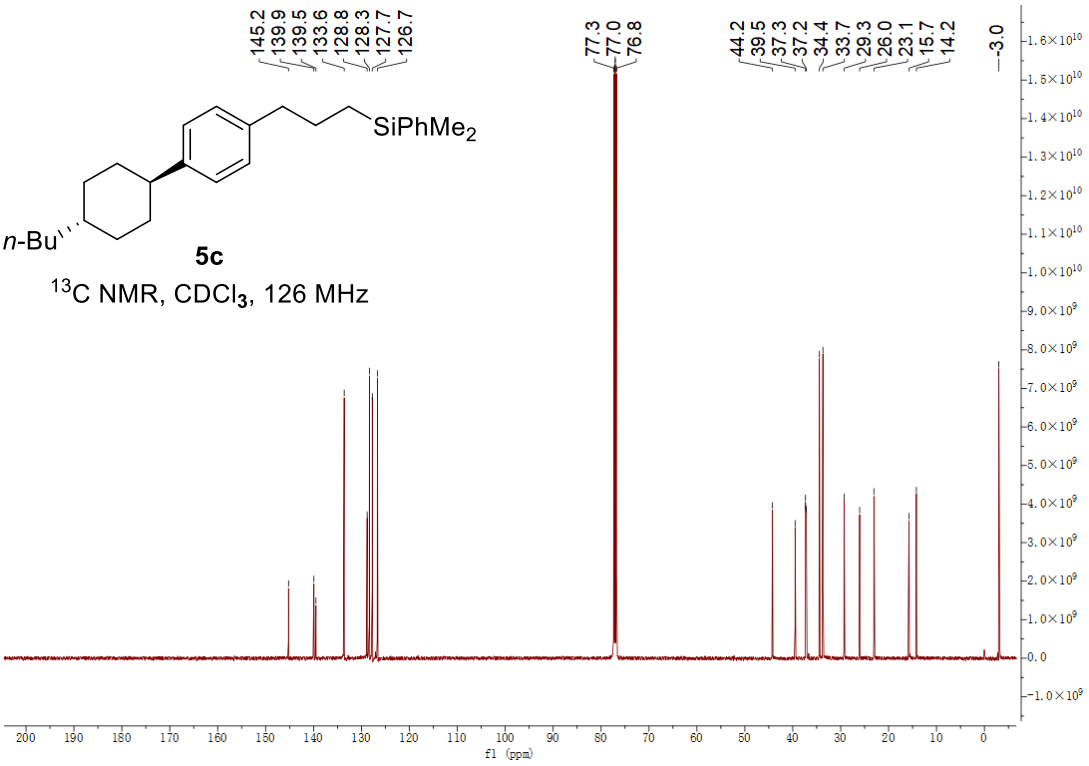

Ph
SiPhMe2
5a
1H NMR, CDCl3, 600 MHz
7.49 7.49 7.48 7.48 7.48 7.47 7.35 7.34 7.34 7.33 7.33 7.32 7.27 7.26 7.26 7.25 7.24 7.24 7.18 7.17 7.16 7.14 7.14 7.13 7.13 7.12 2.62 2.61 2.59 1.67 1.66 1.65 1.65 1.65 1.64 1.64 1.63 1.63 1.62 0.80 0.80 0.80 0.79 0.79 0.78 0.78 0.25 0.00
1.85 2.84 2.13 2.84 2.05 2.00 2.03 5.95
f1 (ppm)

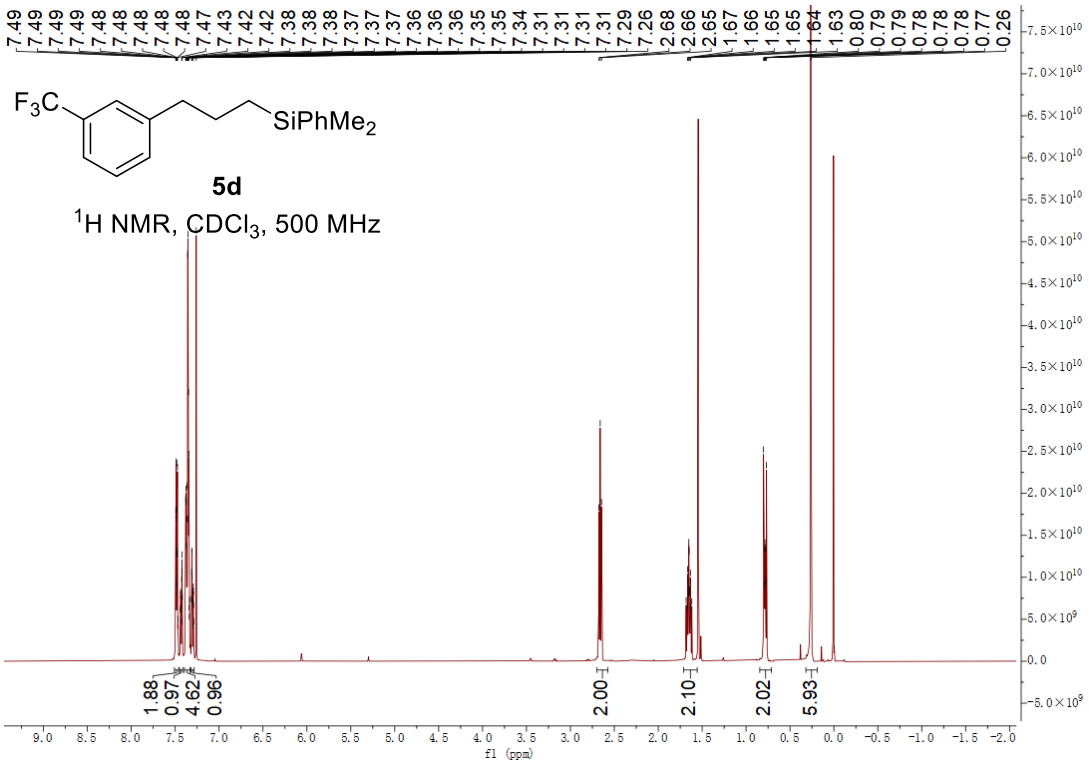

Ph
SiPhMe2
5a
13C NMR, CDCl3, 151 MHz
142.5 139.4 133.5 128.8 128.5 128.2 127.7 125.6 77.2 77.0 76.8 39.8 26.0 15.6 -3.1
f1 (ppm)

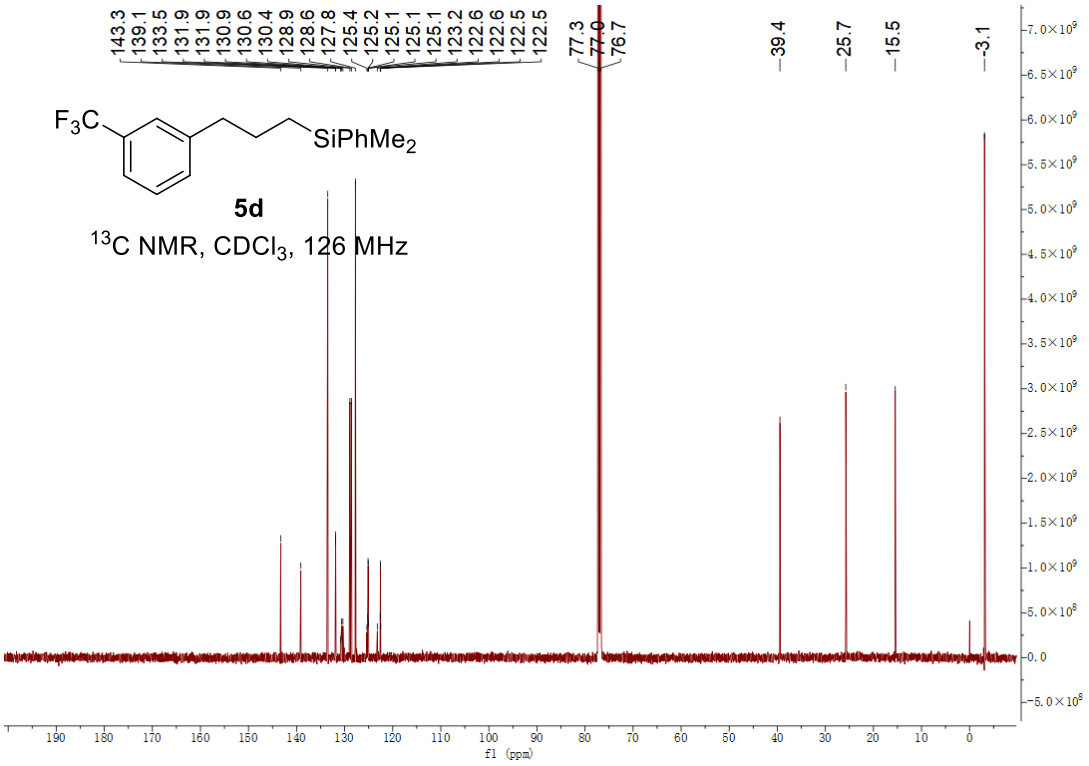

SiPhMe2
MeO
5b
1H NMR, CDCl3, 500 MHz
fl (ppm)

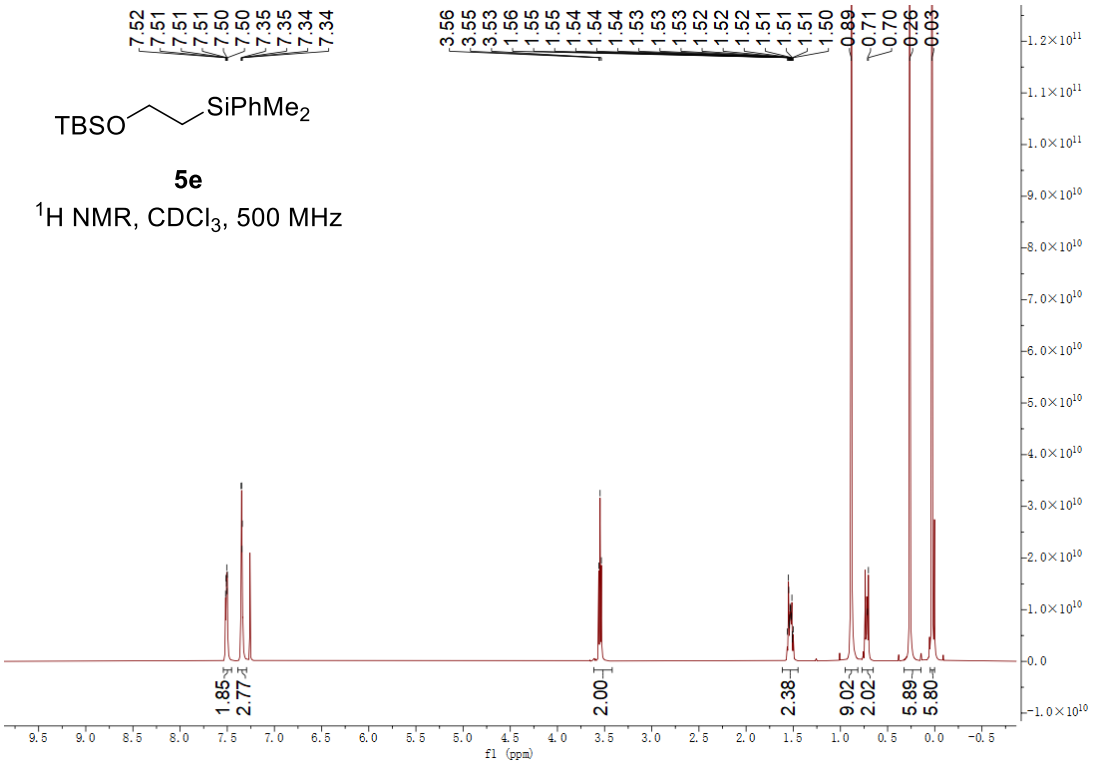

SiPhMe2
MeO
5b
13C NMR, CDCl3, 126 MHz
fl (ppm)


SiPhMe2
n-Bu
5c
1H NMR, CDCl3, 500 MHz
f1 (ppm)


SiPhMe2
n-Bu
5c
13C NMR, CDCl3, 126 MHz
f1 (ppm)

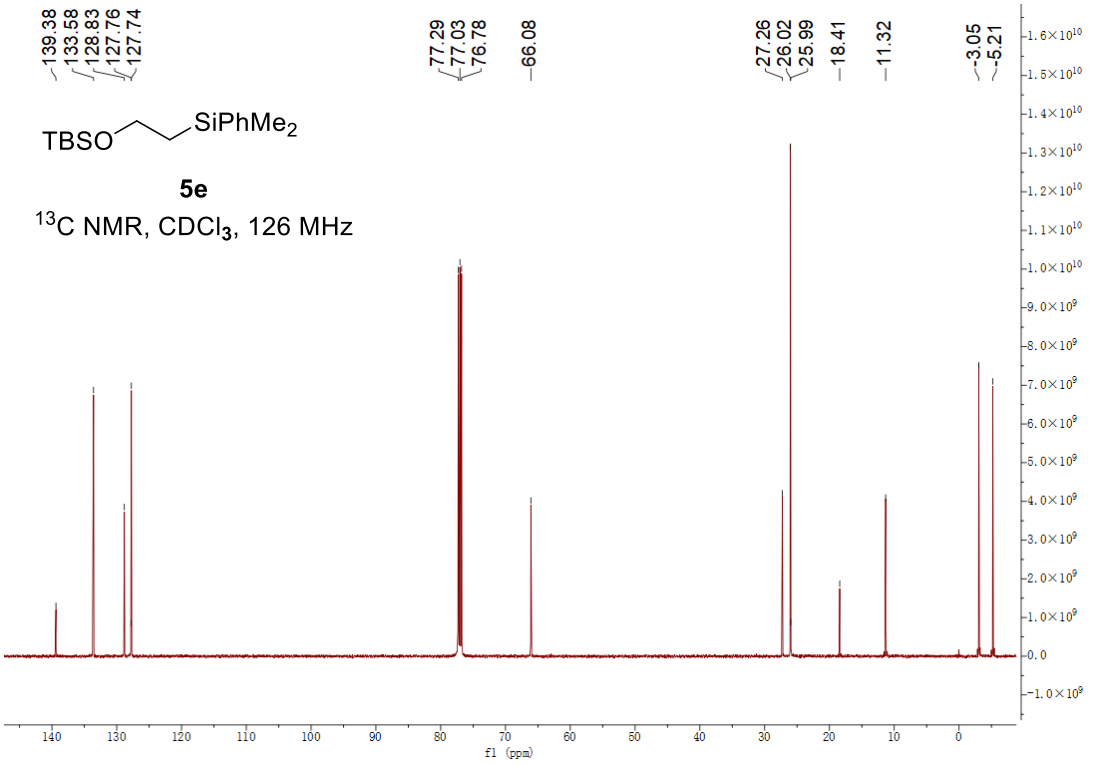

F3C
SiPhMe2
5d
1H NMR, CDCl3, 500 MHz
1.88
0.97
4.62
0.96
2.00
2.10
2.02
5.93
f1 (ppm)


F3C
SiPhMe2
5d
13C NMR, CDCl3, 126 MHz
39.4
25.7
15.5
-3.1
f1 (ppm)



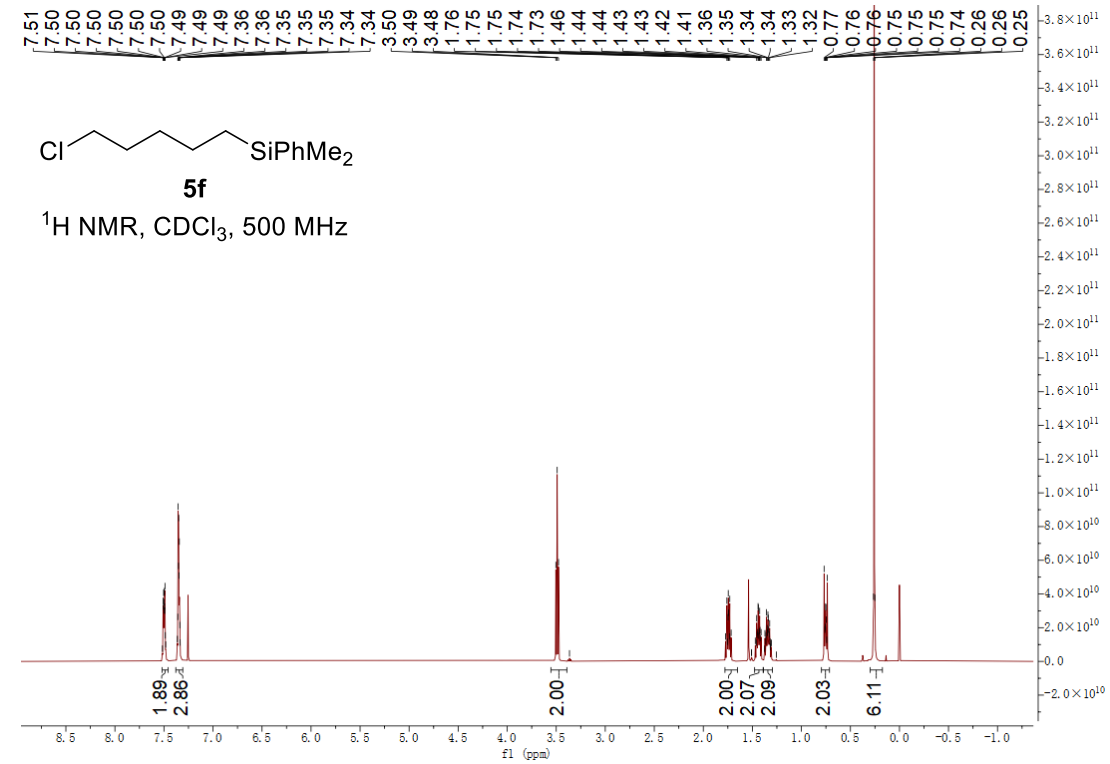
5e

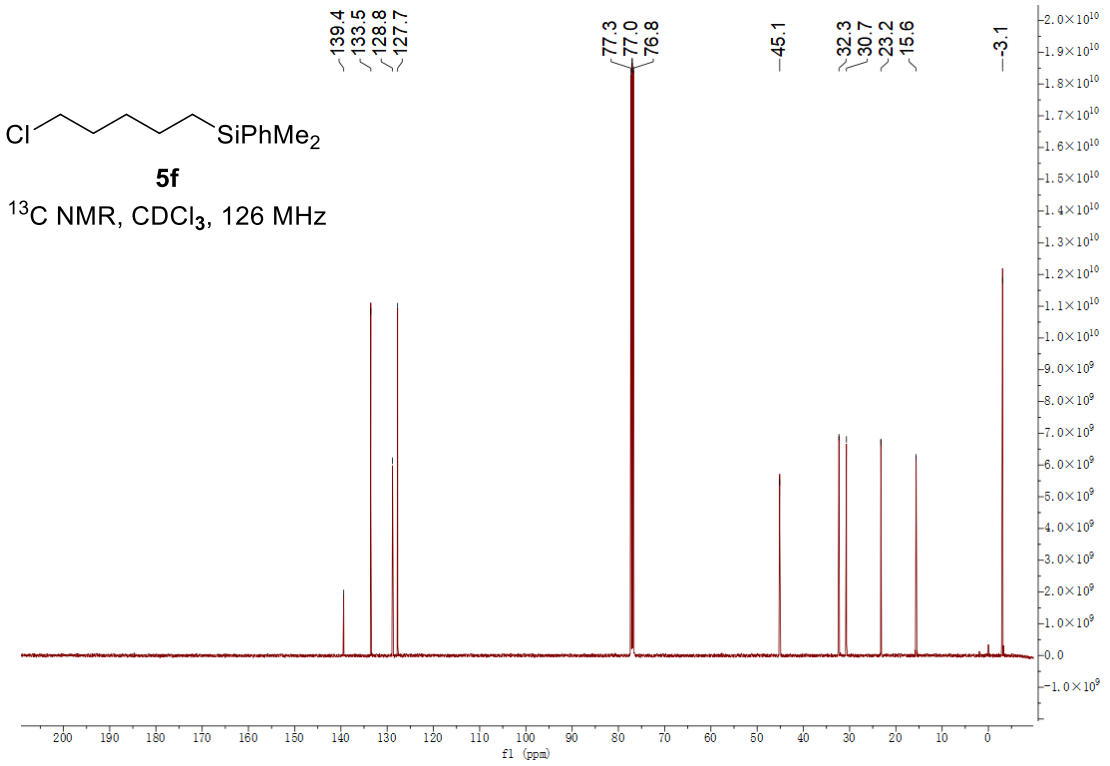
^{1}H NMR, $CDCl_3$, 500 MHz

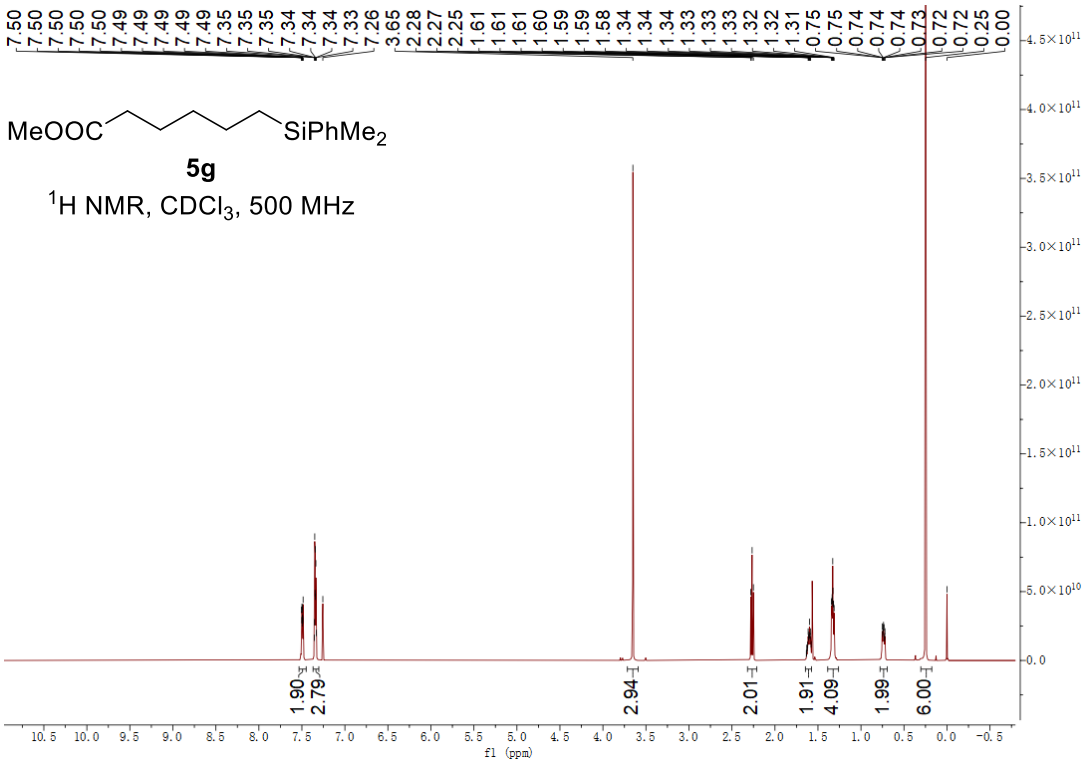


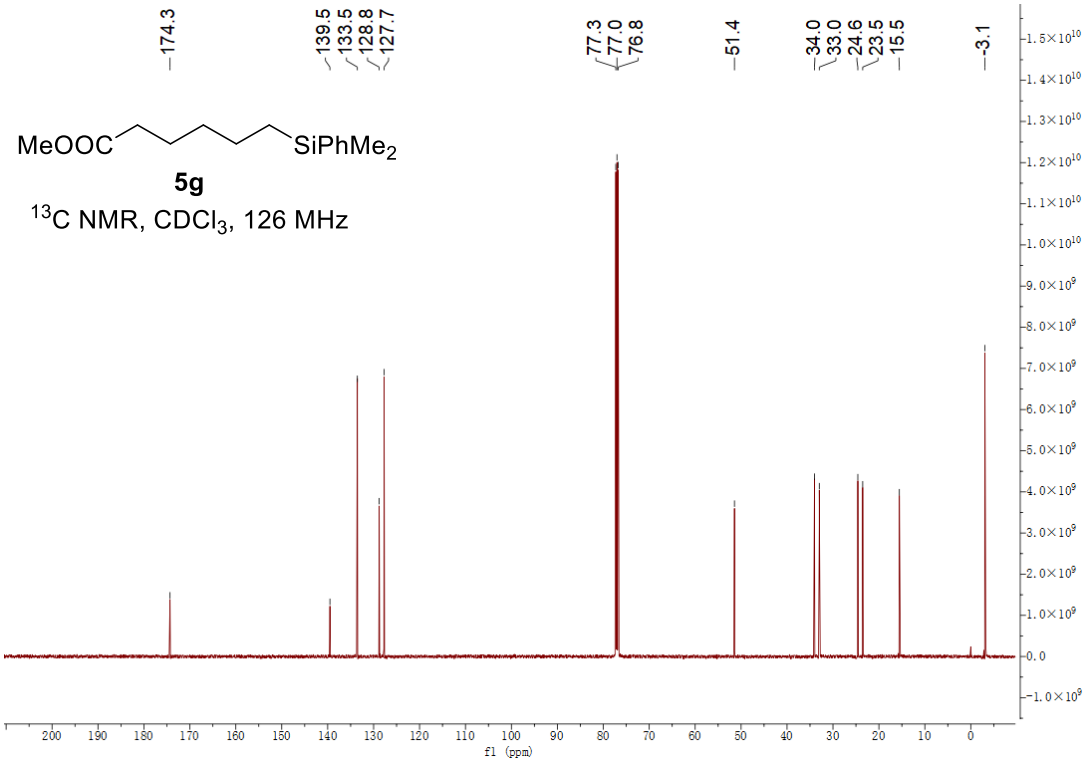
5e

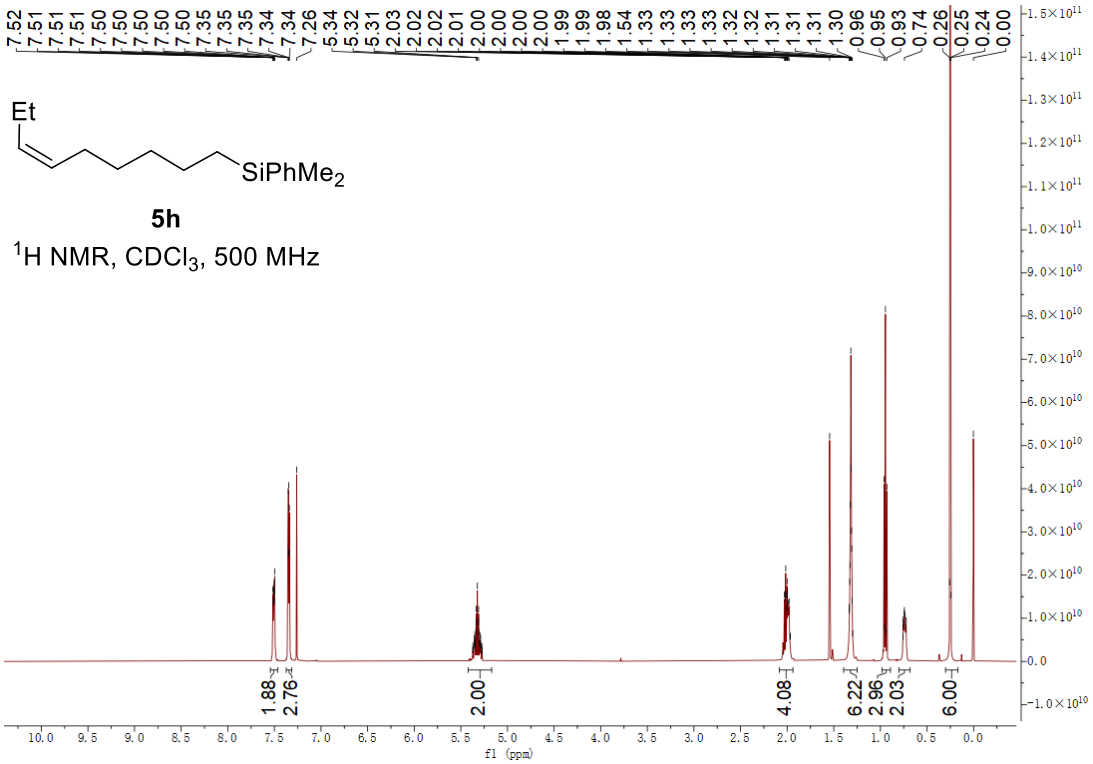
^{13}C NMR, $CDCl_3$, 126 MHz

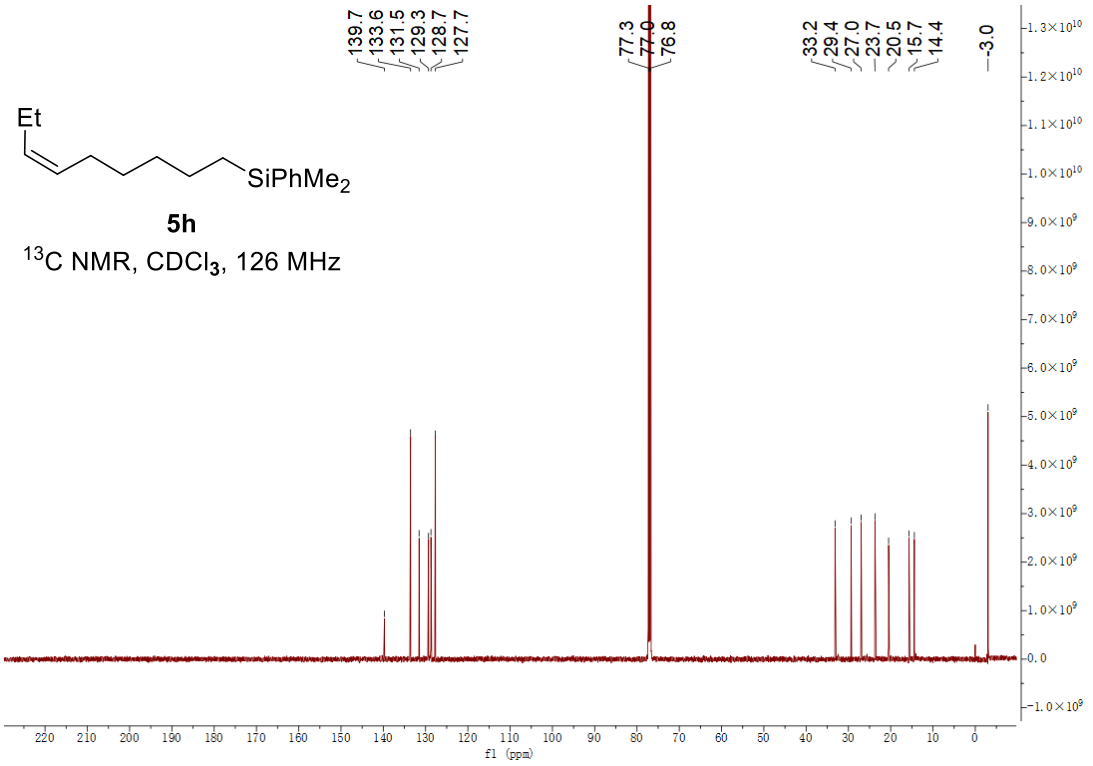

Cl
SiPhMe2
5f
1H NMR, CDCl3, 500 MHz
7.51 7.50 7.50 7.50 7.50 7.50 7.50 7.49 7.49 7.49 7.36 7.36 7.35 7.35 7.35 7.34 7.34
3.50 3.49 3.48 1.76 1.75 1.75 1.74 1.73 1.46 1.44 1.44 1.43 1.43 1.42 1.41 1.36 1.35 1.34 1.34 1.33 1.32
0.77 0.76 0.76 0.75 0.75 0.75 0.74 0.26 0.26 0.25
1.89 2.86 2.00 2.00 2.07 2.09 2.03 6.11
f1 (ppm)

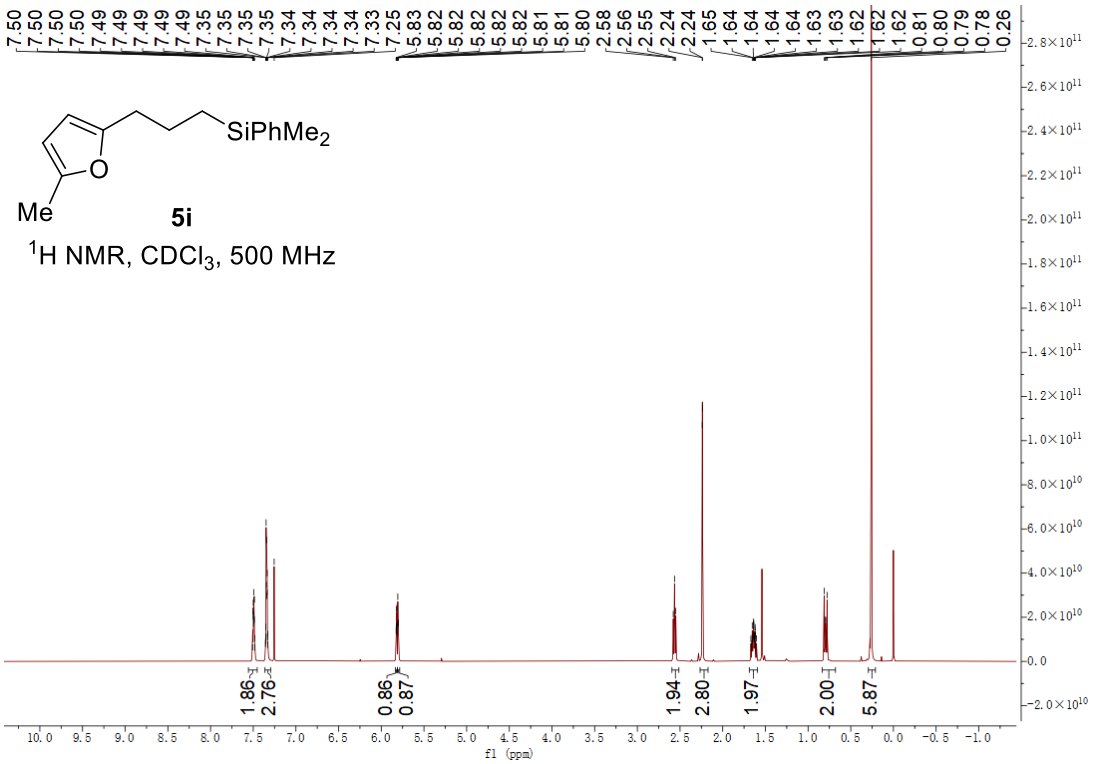

Cl
SiPhMe2
5f
13C NMR, CDCl3, 126 MHz
139.4 133.5 128.8 127.7
77.3 77.0 76.8
45.1
32.3 30.7 23.2 15.6
3.1
f1 (ppm)

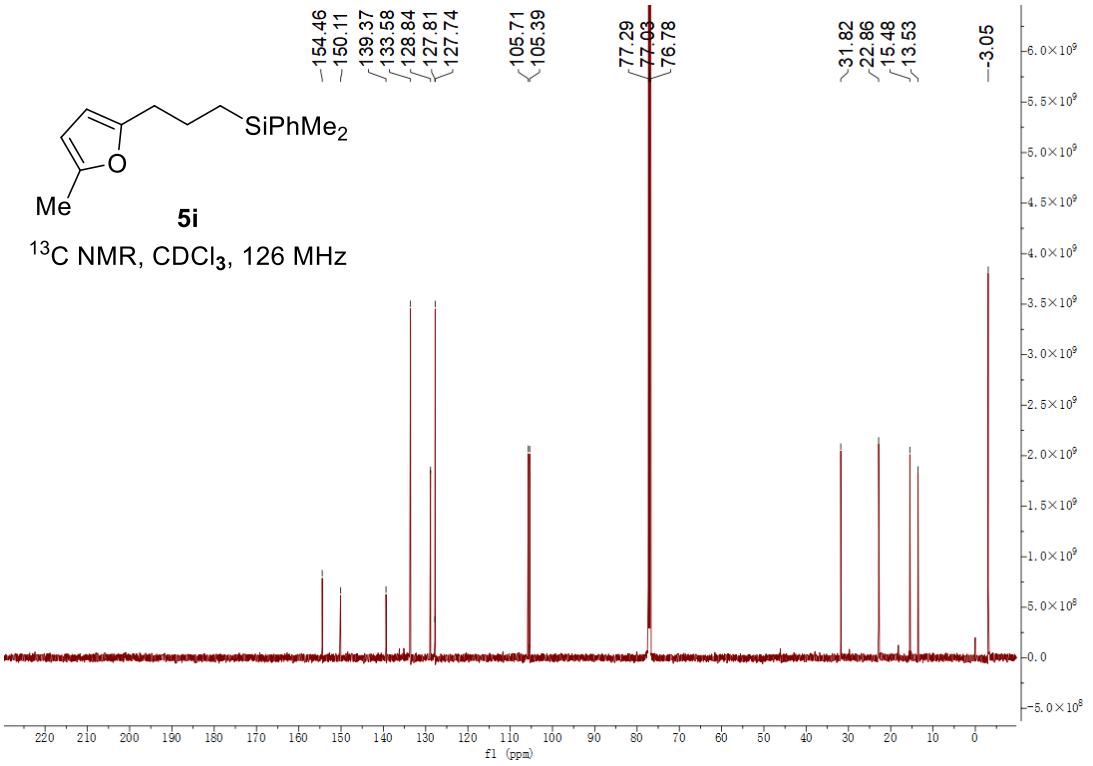

MeOOC
SiPhMe2
5g
1H NMR, CDCl3, 500 MHz
f1 (ppm)

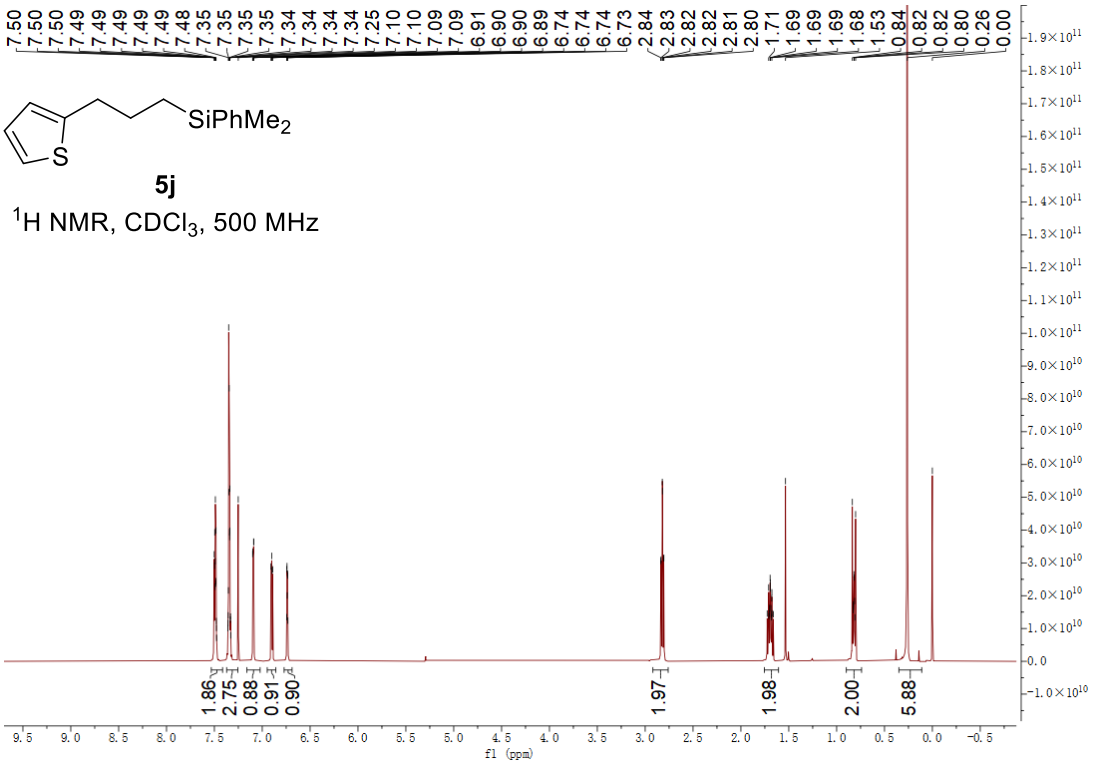

MeOOC
SiPhMe2
5g
13C NMR, CDCl3, 126 MHz
f1 (ppm)

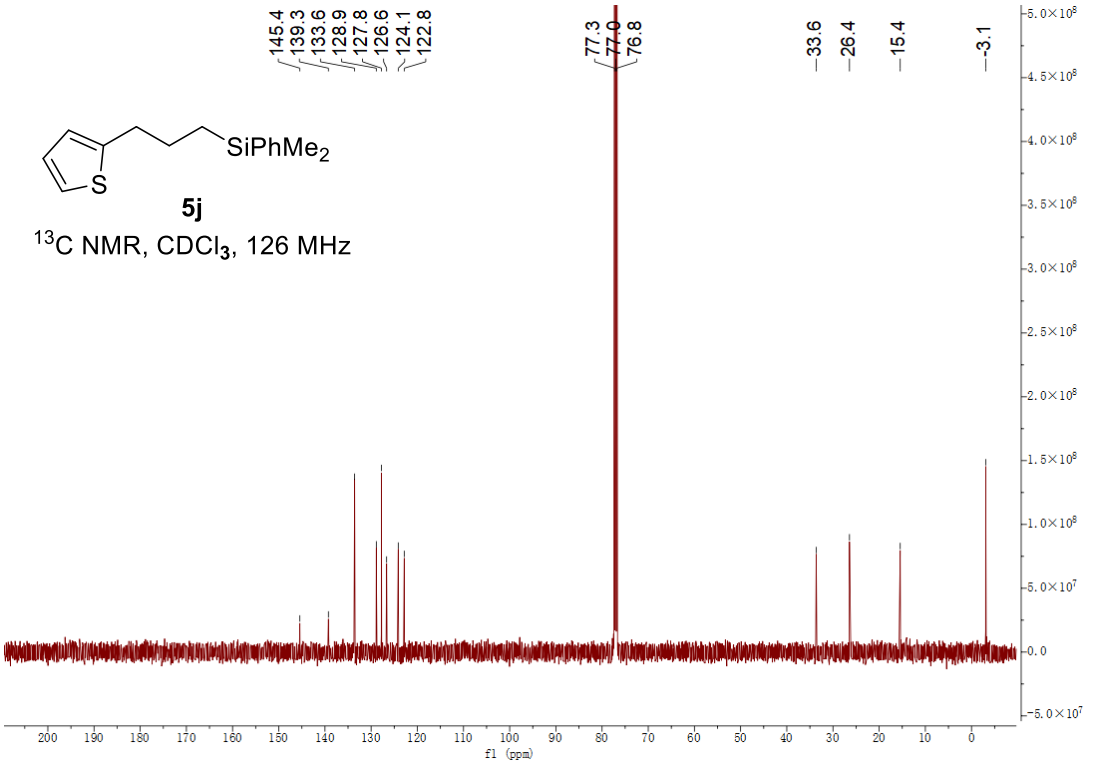

Et
SiPhMe2
5h
1H NMR, CDCl3, 500 MHz
7.52 7.51 7.51 7.51 7.50 7.50 7.50 7.50 7.50 7.35 7.35 7.35 7.34 7.34 7.26
5.34 5.32 5.31 2.03 2.02 2.02 2.01 2.00 2.00 2.00 2.00 1.99 1.99 1.98 1.54 1.33 1.33 1.33 1.32 1.32 1.31 1.31 1.31 1.30 0.96 0.95 0.93 0.74 0.26 0.25 0.24 0.00
1.88 2.76 2.00 4.08 6.22 2.96 2.03 6.00
f1 (ppm)

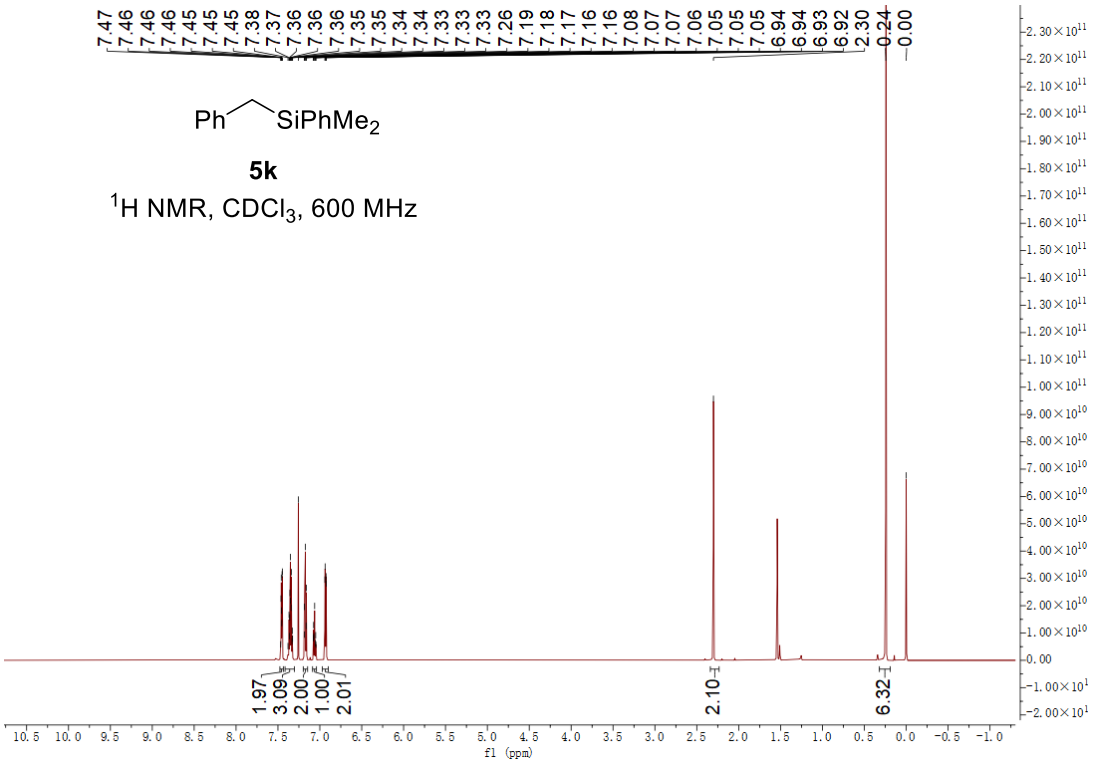

Et
SiPhMe2
5h
13C NMR, CDCl3, 126 MHz
139.7 133.6 131.5 129.3 128.7 127.7
77.3 77.0 76.8
33.2 29.4 27.0 23.7 20.5 15.7 14.4
-3.0
f1 (ppm)

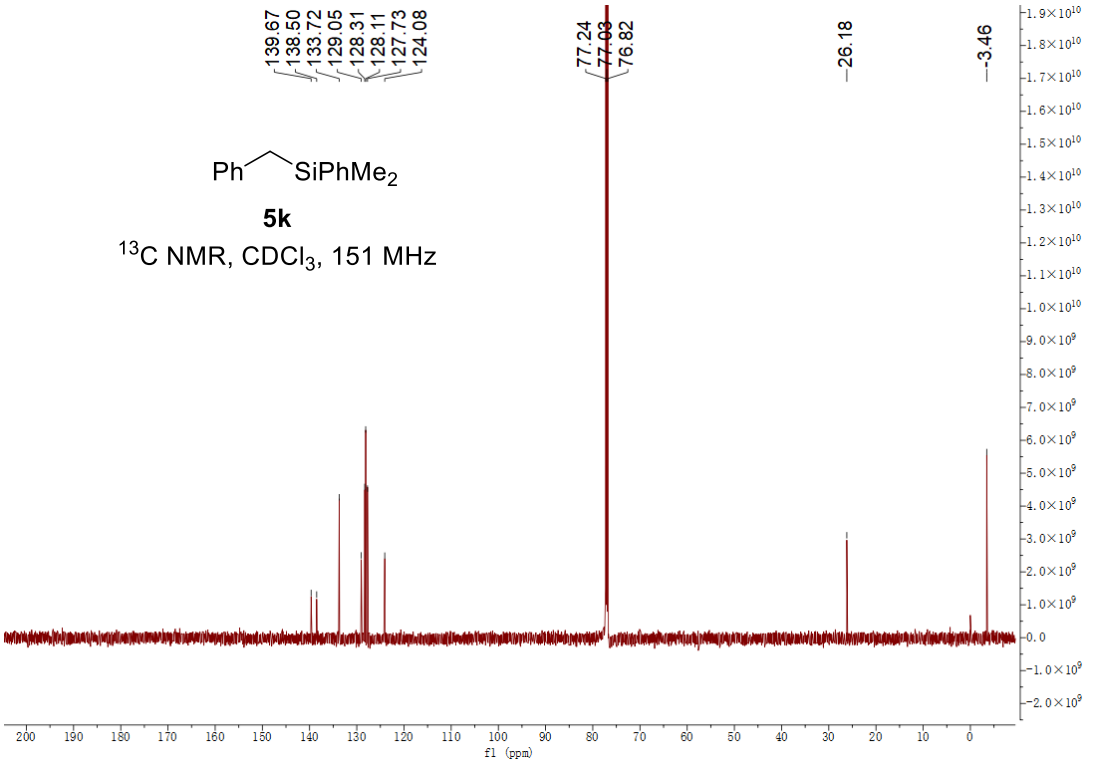

SiPhMe2
O
Me
5i
1H NMR, CDCl3, 500 MHz
f1 (ppm)

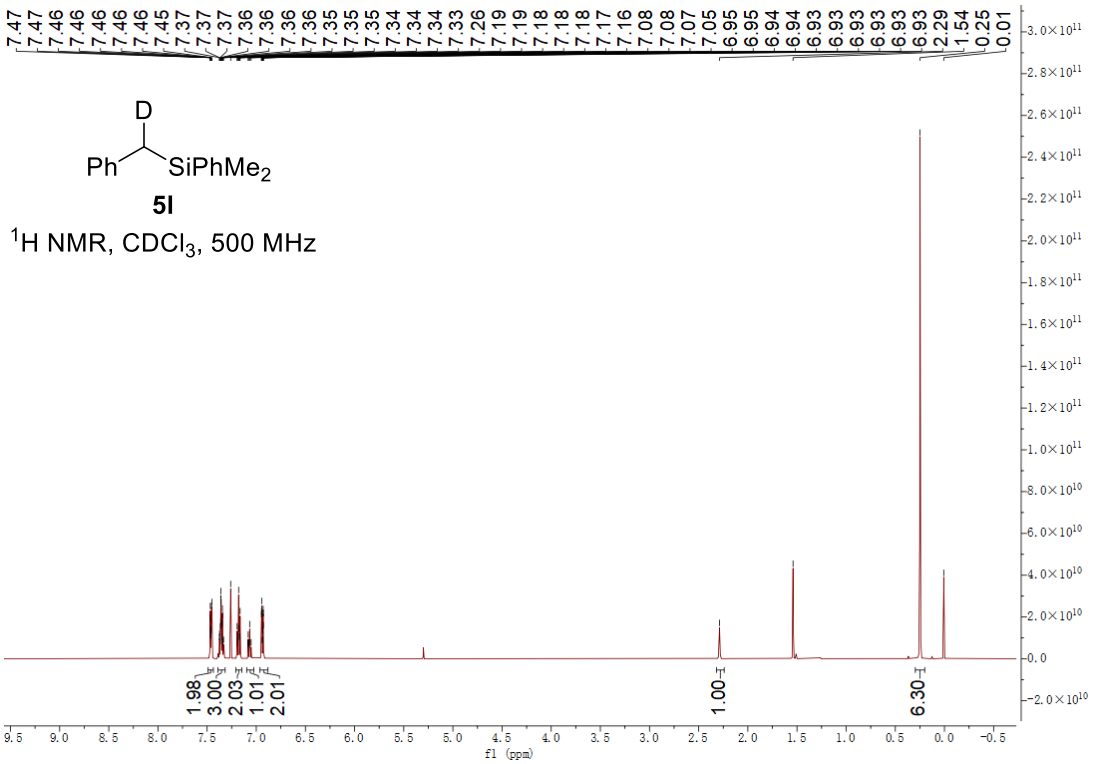

SiPhMe2
O
Me
5i
13C NMR, CDCl3, 126 MHz
f1 (ppm)

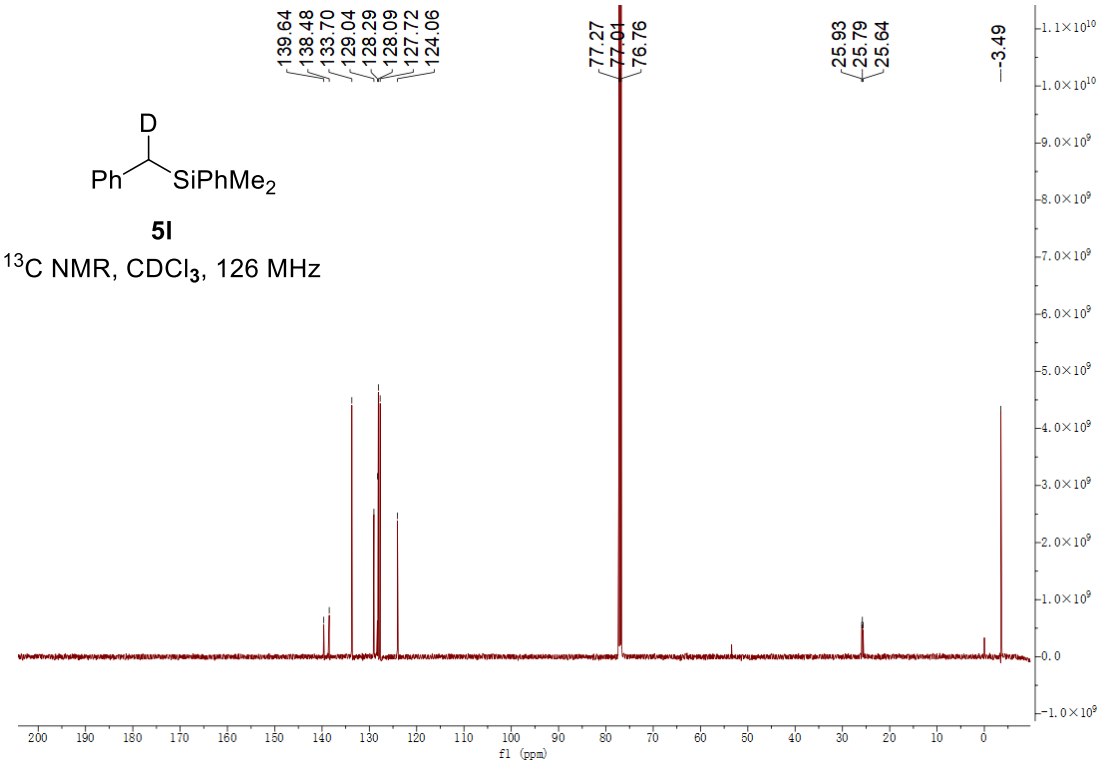

SiPhMe2
S
5j
1H NMR, CDCl3, 500 MHz
7.50 7.50 7.50 7.49 7.49 7.49 7.49 7.48 7.35 7.35 7.35 7.35 7.34 7.34 7.34 7.34 7.25 7.10 7.10 7.09 6.91 6.90 6.90 6.89 6.74 6.74 6.74 6.73 2.84 2.83 2.82 2.82 2.81 2.80 1.71 1.69 1.69 1.69 1.68 1.53 0.84 0.82 0.82 0.80 0.26 0.00
1.86 2.75 0.88 0.91 0.90 1.97 1.98 2.00 5.88
f1 (ppm)

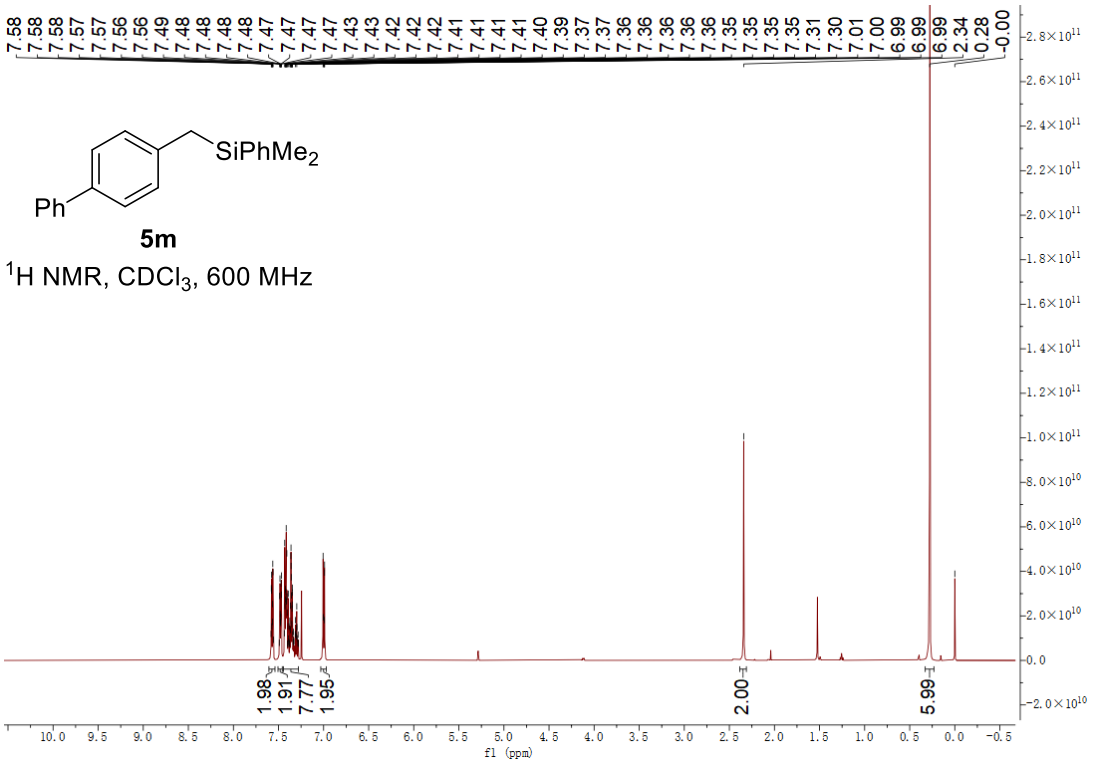

SiPhMe2
S
5j
13C NMR, CDCl3, 126 MHz
145.4 139.3 133.6 128.9 127.8 126.6 124.1 122.8
77.3 77.0 76.8
33.6 26.4 15.4 3.1
f1 (ppm)


7.47
7.46
7.46
7.46
7.45
7.45
7.45
7.38
7.37
7.36
7.36
7.36
7.35
7.35
7.34
7.34
7.33
7.33
7.33
7.26
7.19
7.18
7.17
7.16
7.16
7.08
7.07
7.07
7.06
7.05
7.05
7.05
6.94
6.94
6.93
6.92
2.30
0.24
0.00
Ph
SiPhMe2
5k
1H NMR, CDCl3, 600 MHz
1.97
3.09
2.00
1.00
2.01
2.10
6.32
f1 (ppm)


139.67
138.50
133.72
129.05
128.31
128.11
127.73
124.08
77.24
77.03
76.82
26.18
-3.46
Ph
SiPhMe2
5k
13C NMR, CDCl3, 151 MHz
f1 (ppm)

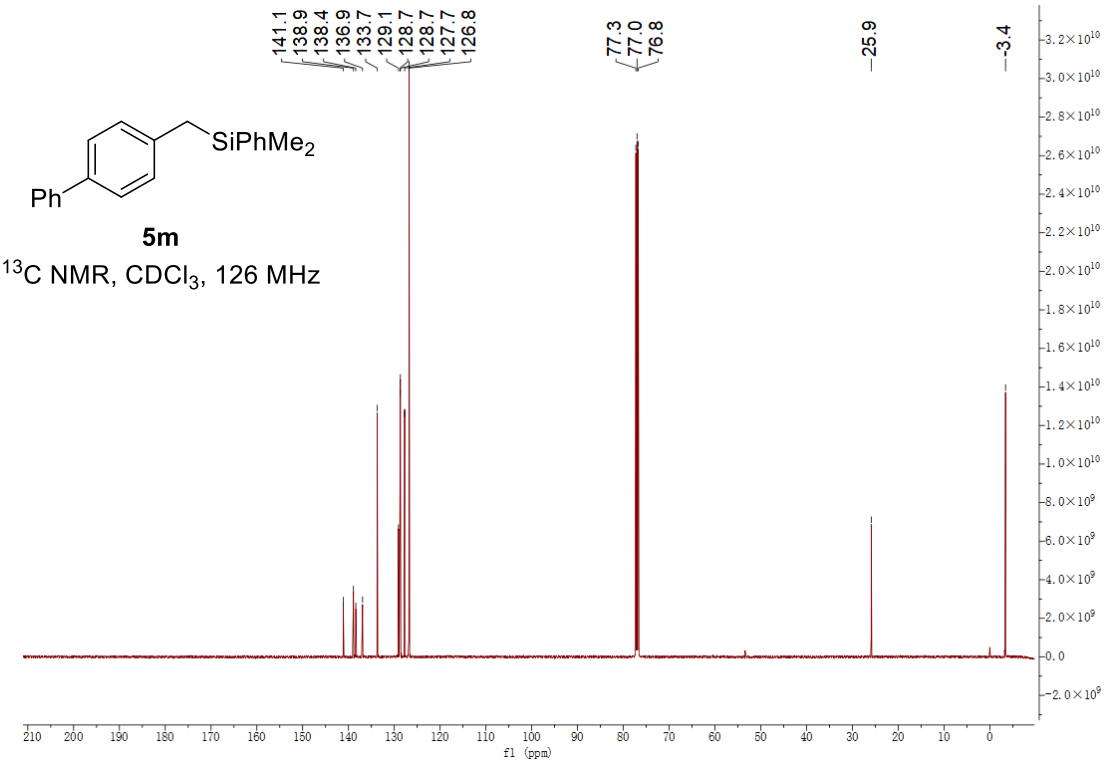

7.47 7.47 7.46 7.46 7.46 7.46 7.46 7.45 7.37 7.37 7.37 7.36 7.36 7.36 7.36 7.35 7.35 7.35 7.34 7.34 7.34 7.33 7.26 7.19 7.19 7.18 7.18 7.18 7.17 7.16 7.08 7.08 7.07 7.05 6.95 6.95 6.94 6.94 6.93 6.93 6.93 6.93 6.93 6.93 2.29 1.54 0.25 0.01
D
Ph
SiPhMe2
5l
1H NMR, CDCl3, 500 MHz
1.98 3.00 2.03 1.01 2.01 1.00 6.30
f1 (ppm)


139.64 138.48 133.70 129.04 128.29 128.09 127.72 124.06 77.27 77.01 76.76 25.93 25.79 25.64 -3.49
D
Ph
SiPhMe2
5l
13C NMR, CDCl3, 126 MHz
f1 (ppm)



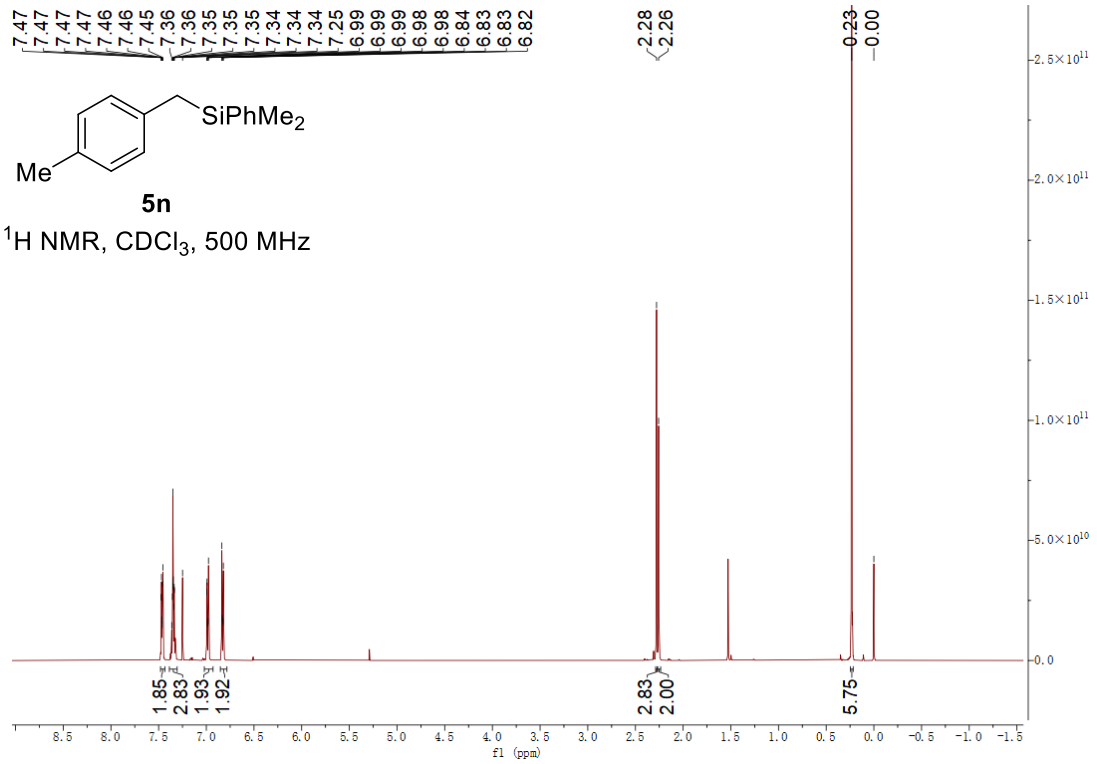
5m

^{1}H NMR, $CDCl_3$, 600 MHz



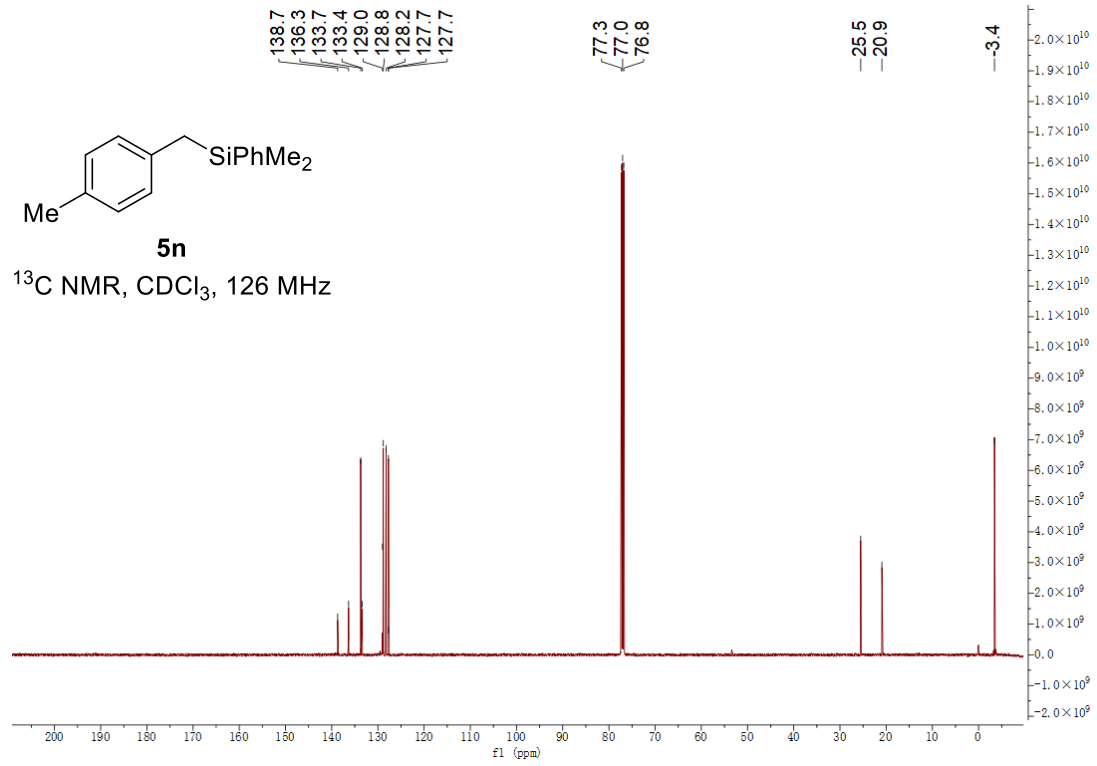
5m

^{13}C NMR, $CDCl_3$, 126 MHz



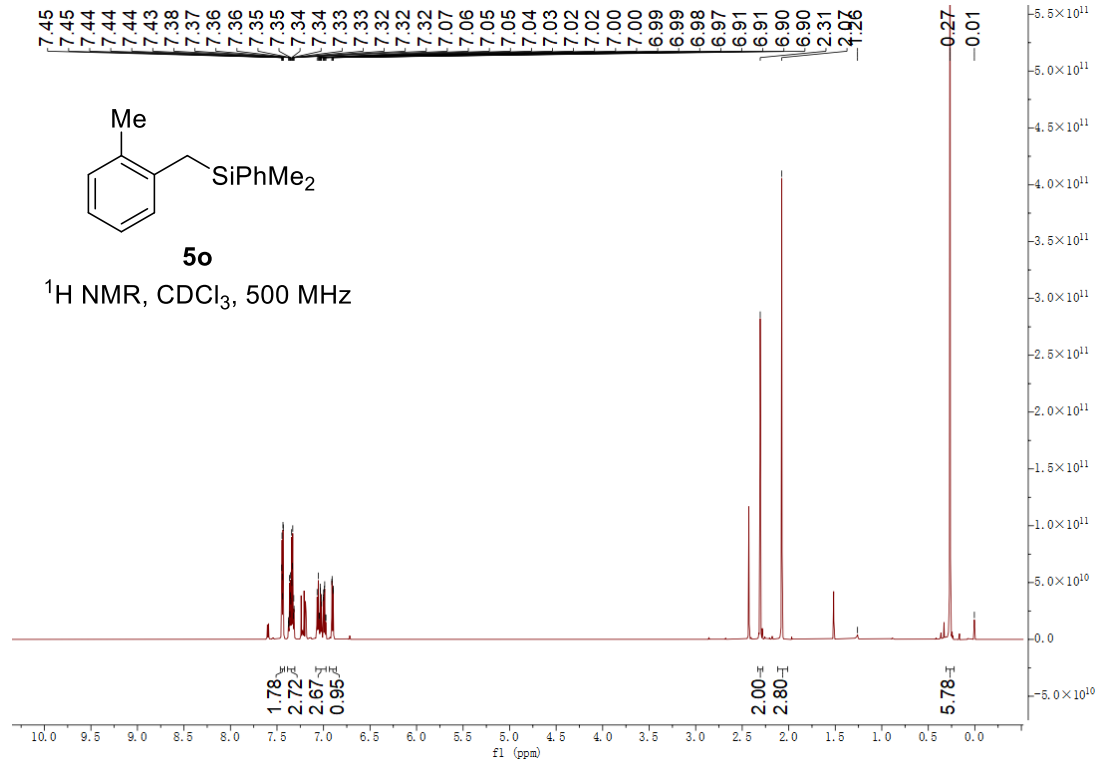
5n

^{1}H NMR, $CDCl_3$, 500 MHz



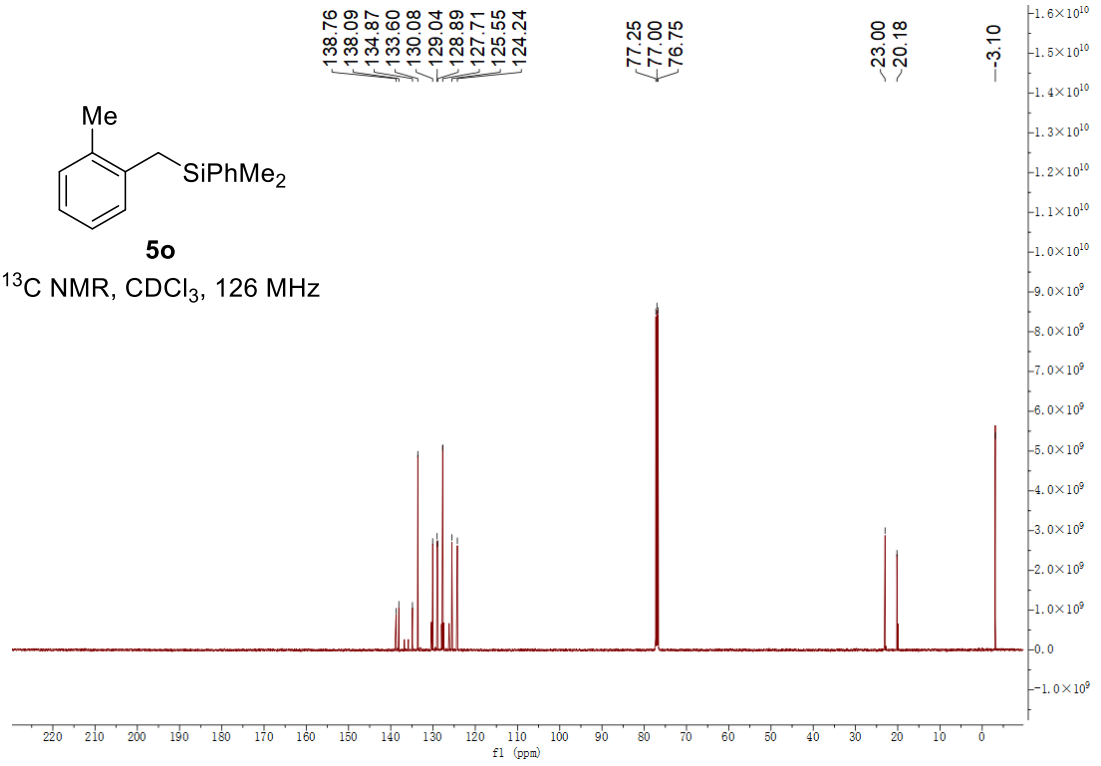
5n

^{13}C NMR, $CDCl_3$, 126 MHz



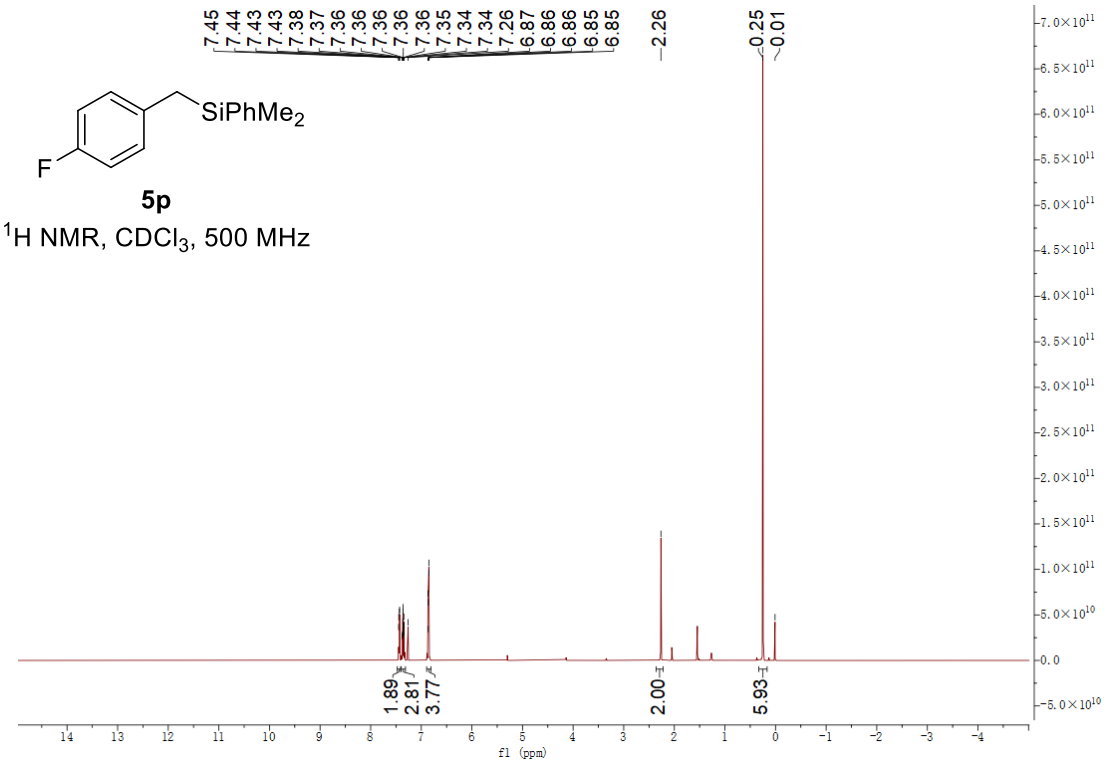
5o

^{1}H NMR, $CDCl_3$, 500 MHz



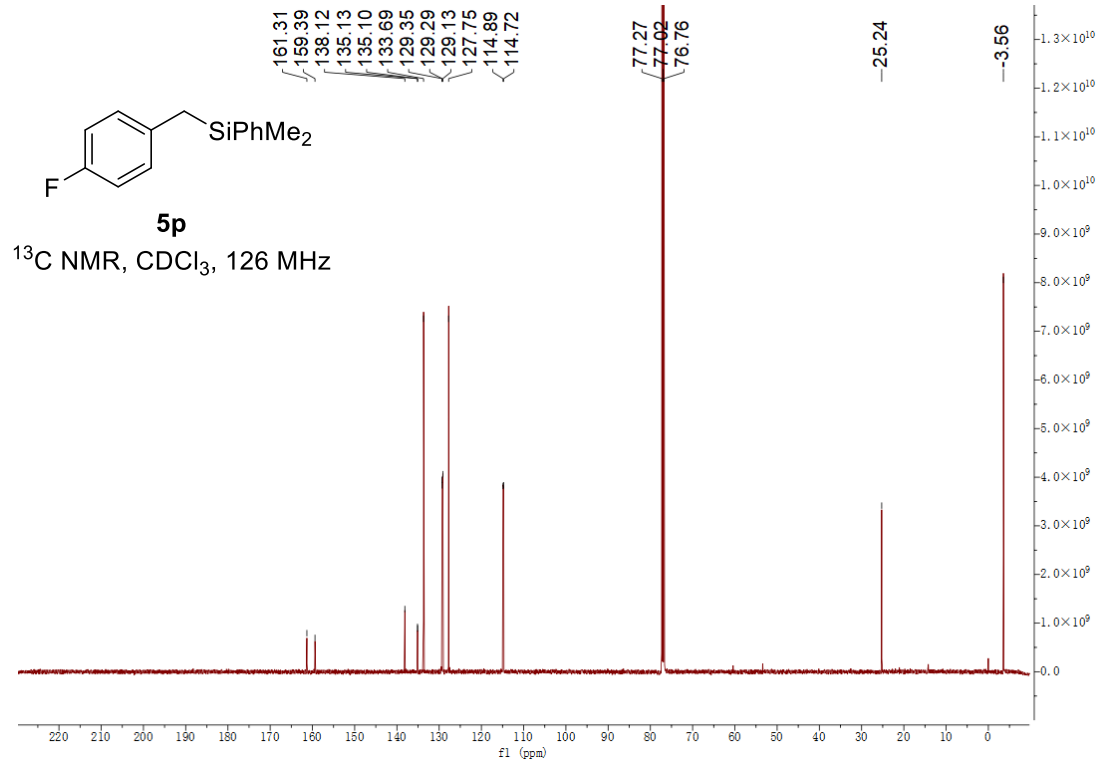
5o

^{13}C NMR, $CDCl_3$, 126 MHz



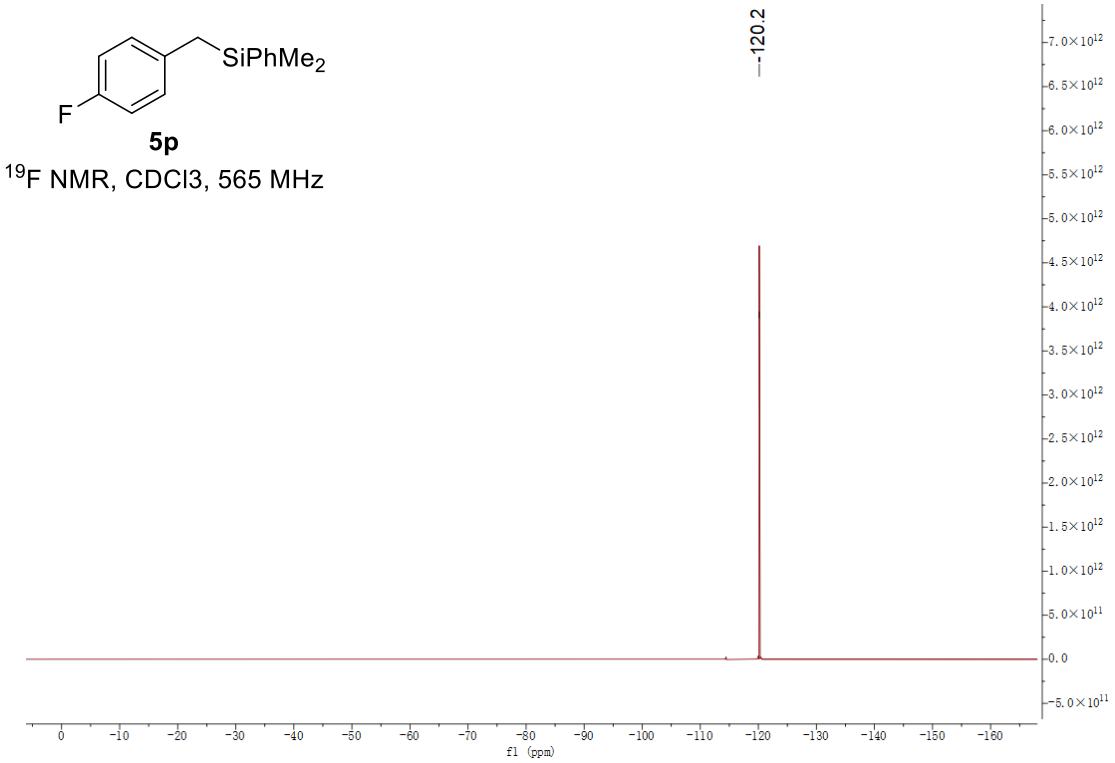
5p

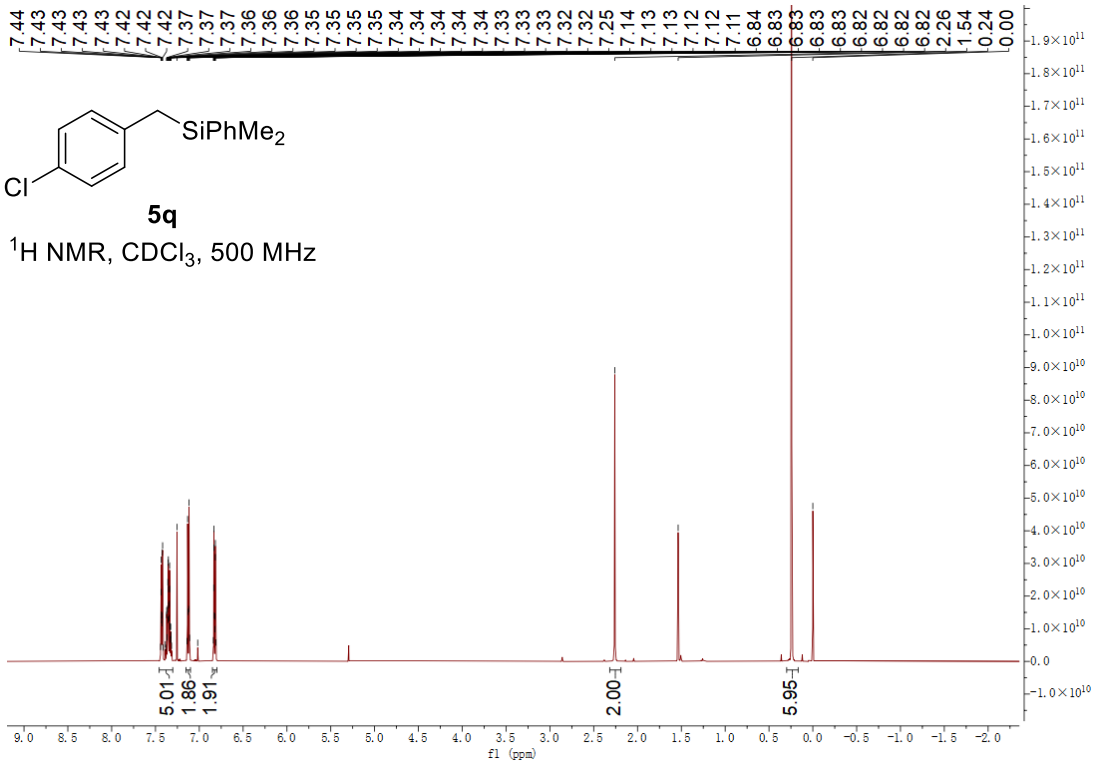
^{1}H NMR, $CDCl_3$, 500 MHz

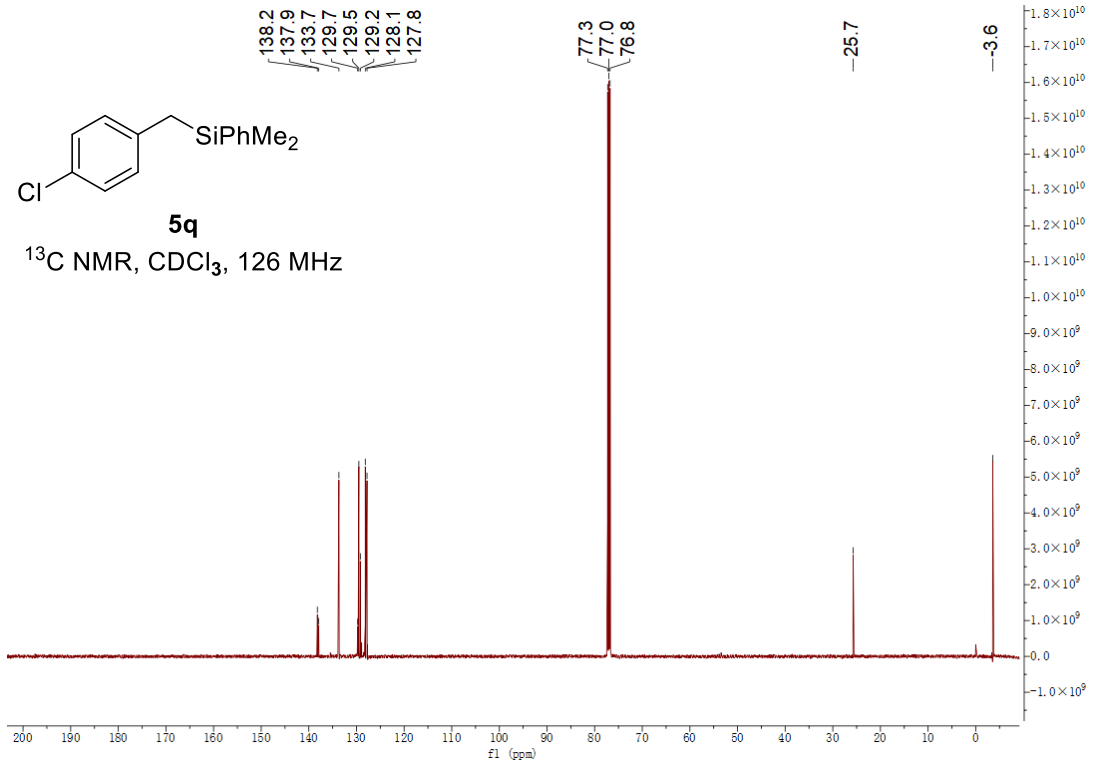


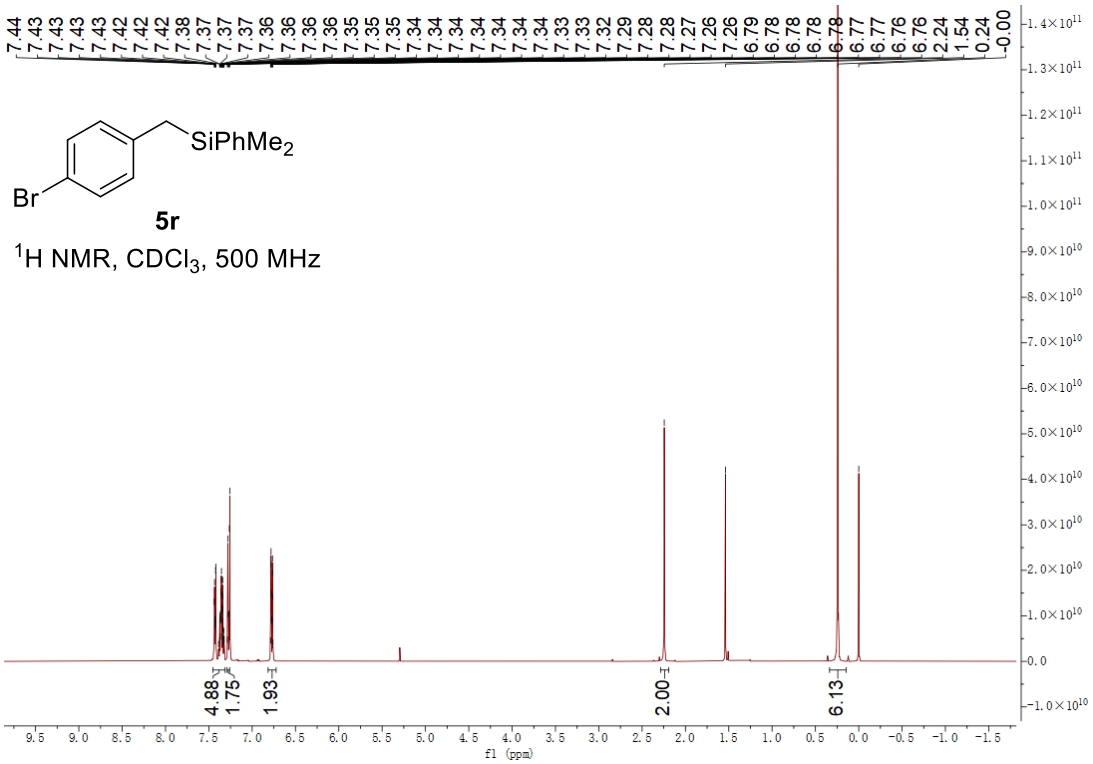
5p

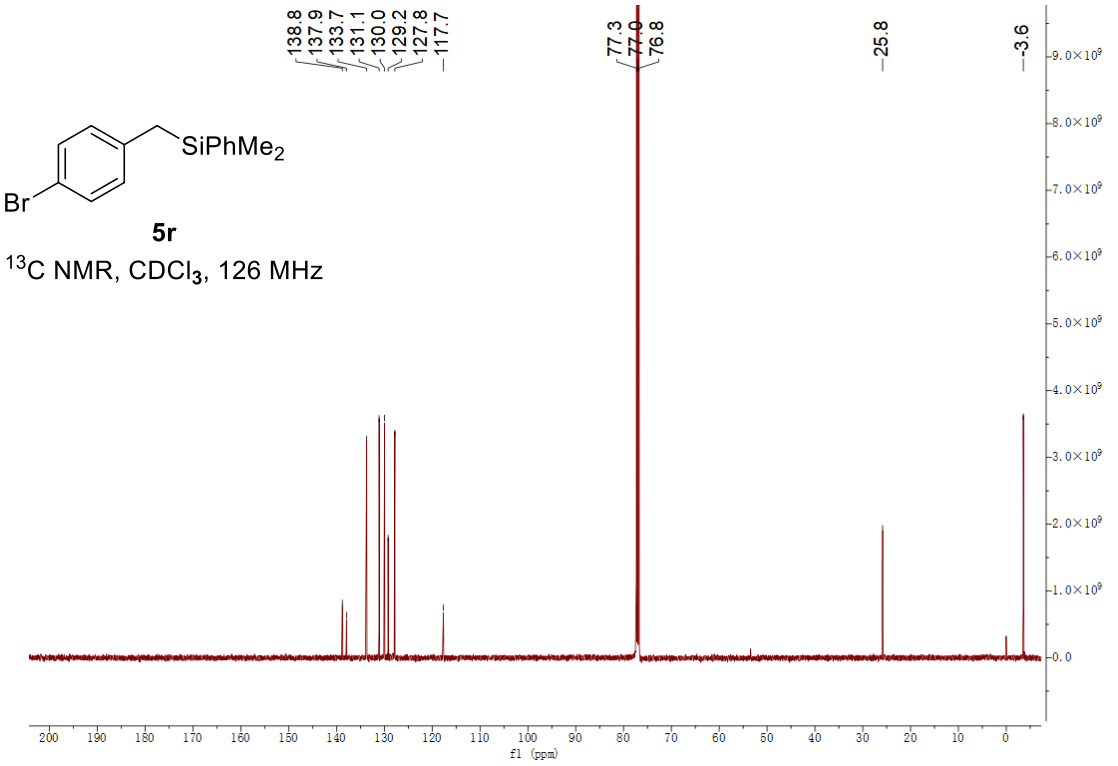
^{13}C NMR, $CDCl_3$, 126 MHz

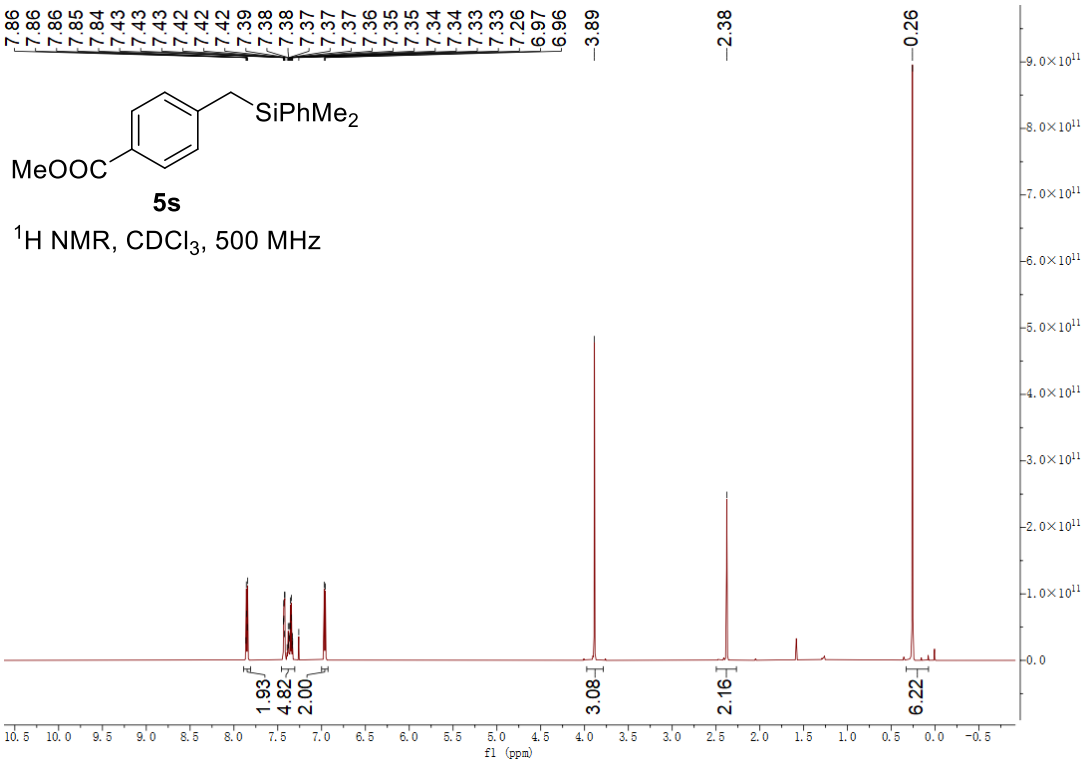

SiPhMe2
F
5p
19F NMR, CDCl3, 565 MHz
-120.2
7.0×10^12
6.5×10^12
6.0×10^12
5.5×10^12
5.0×10^12
4.5×10^12
4.0×10^12
3.5×10^12
3.0×10^12
2.5×10^12
2.0×10^12
1.5×10^12
1.0×10^12
5.0×10^11
0.0
-5.0×10^11
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)


SiPhMe2
Cl
5q
1H NMR, CDCl3, 500 MHz
7.44 7.43 7.43 7.43 7.43 7.42 7.42 7.42 7.37 7.37 7.37 7.36 7.36 7.36 7.35 7.35 7.35 7.35 7.34 7.34 7.34 7.34 7.34 7.33 7.33 7.33 7.32 7.32 7.25 7.14 7.13 7.13 7.12 7.12 7.11 6.84 6.83 6.83 6.83 6.83 6.82 6.82 6.82 6.82 2.26 1.54 0.24 0.00
5.01 1.86 1.91 2.00 5.95
f1 (ppm)


SiPhMe2
Cl
5q
13C NMR, CDCl3, 126 MHz
138.2 137.9 133.7 129.7 129.5 129.2 128.1 127.8
77.3 77.0 76.8
25.7
-3.6
f1 (ppm)

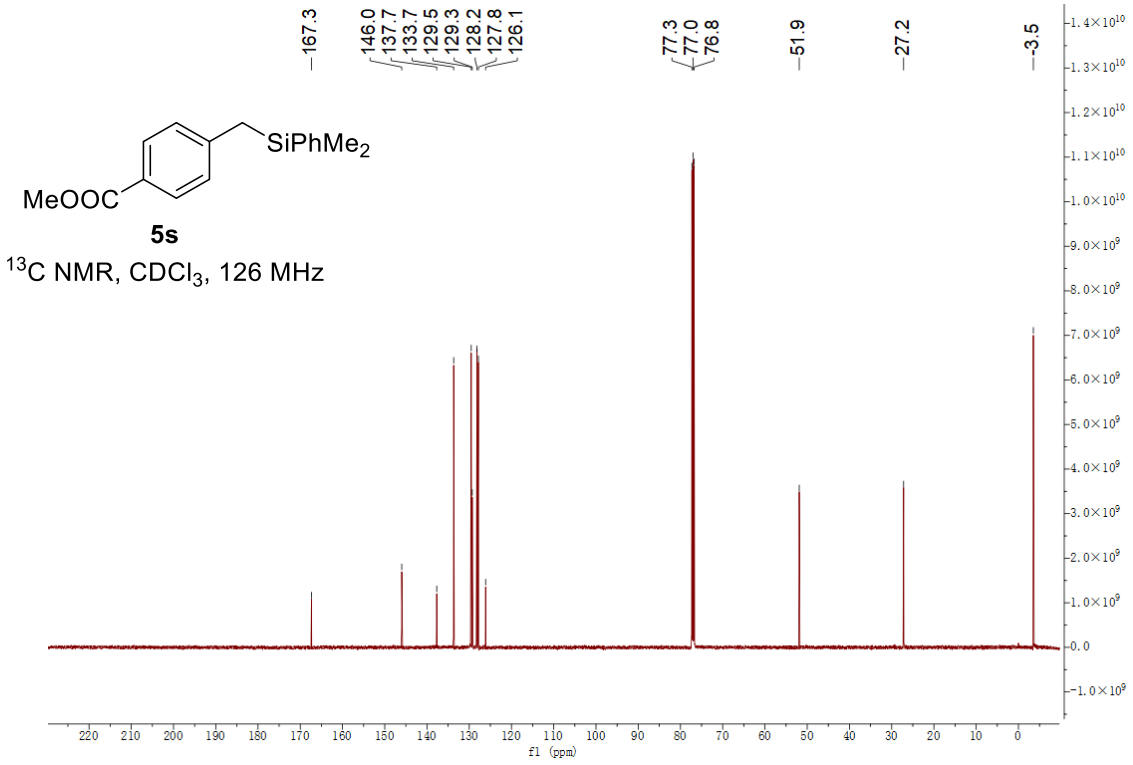

SiPhMe2
Br
5r
1H NMR, CDCl3, 500 MHz
4.88
1.75
1.93
2.00
6.13
f1 (ppm)


SiPhMe2
Br
5r
13C NMR, CDCl3, 126 MHz
138.8
137.9
133.7
131.1
130.0
129.2
127.8
117.7
77.3
77.0
76.8
25.8
-3.6
f1 (ppm)



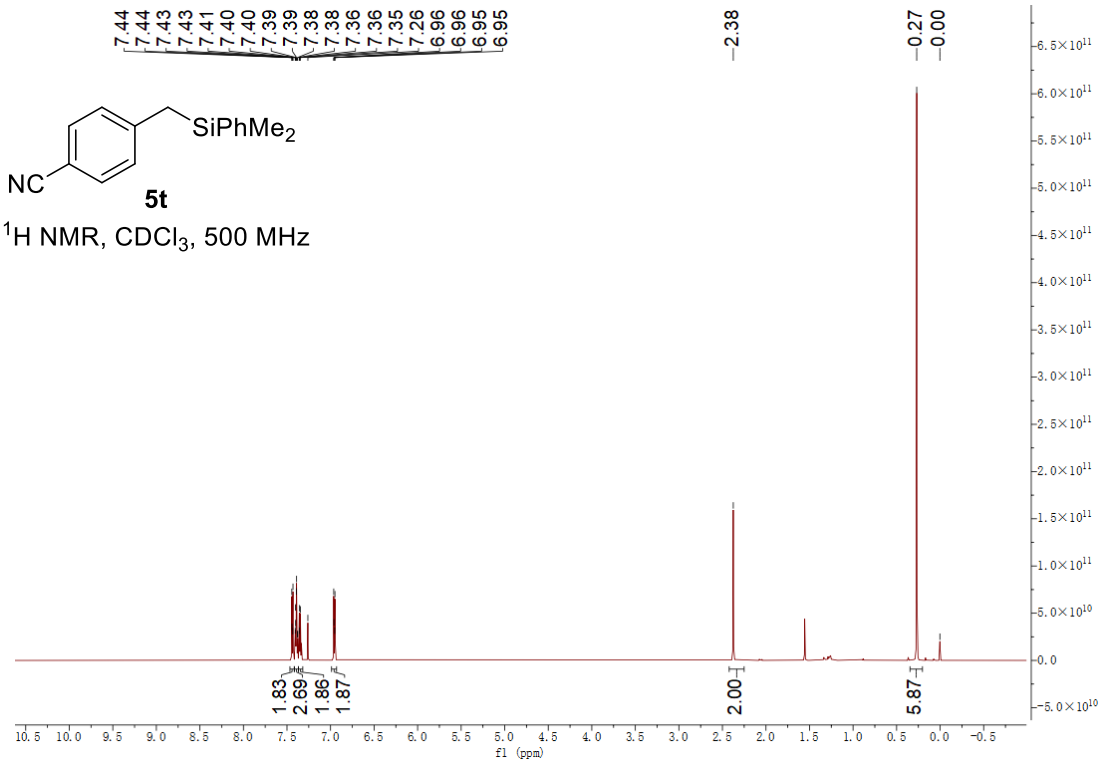
5s

^{1}H NMR, $CDCl_3$, 500 MHz



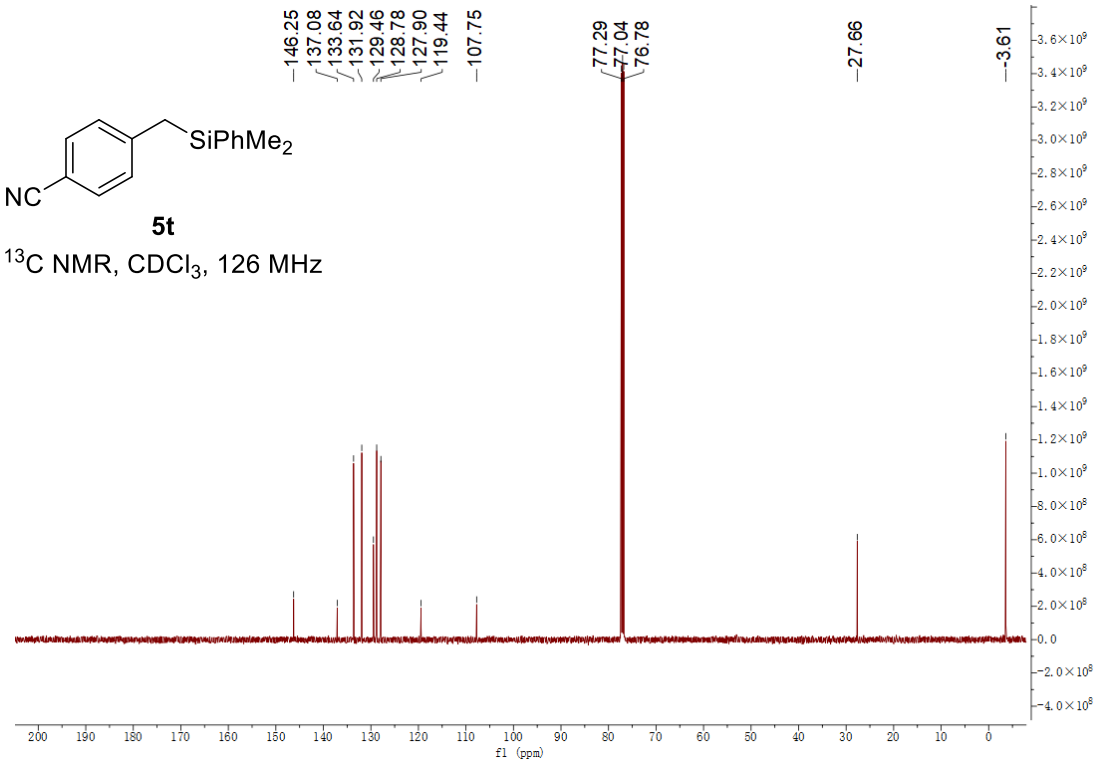
5s

^{13}C NMR, $CDCl_3$, 126 MHz



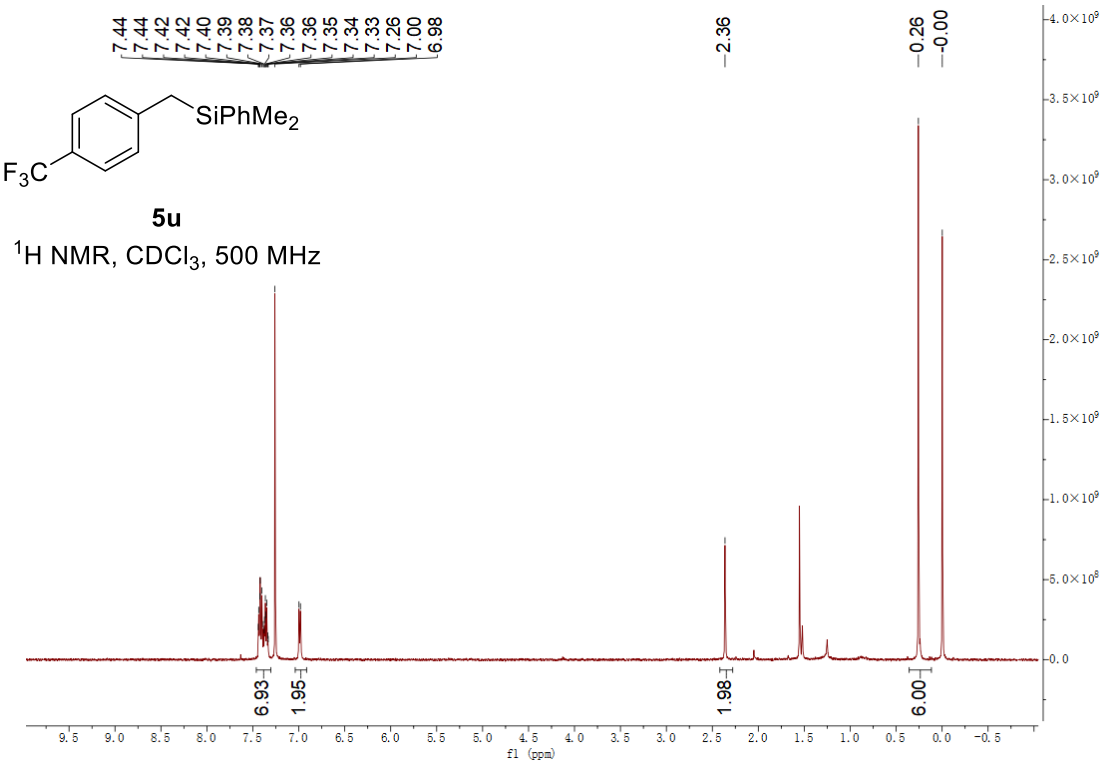
5t

^{1}H NMR, $CDCl_3$, 500 MHz



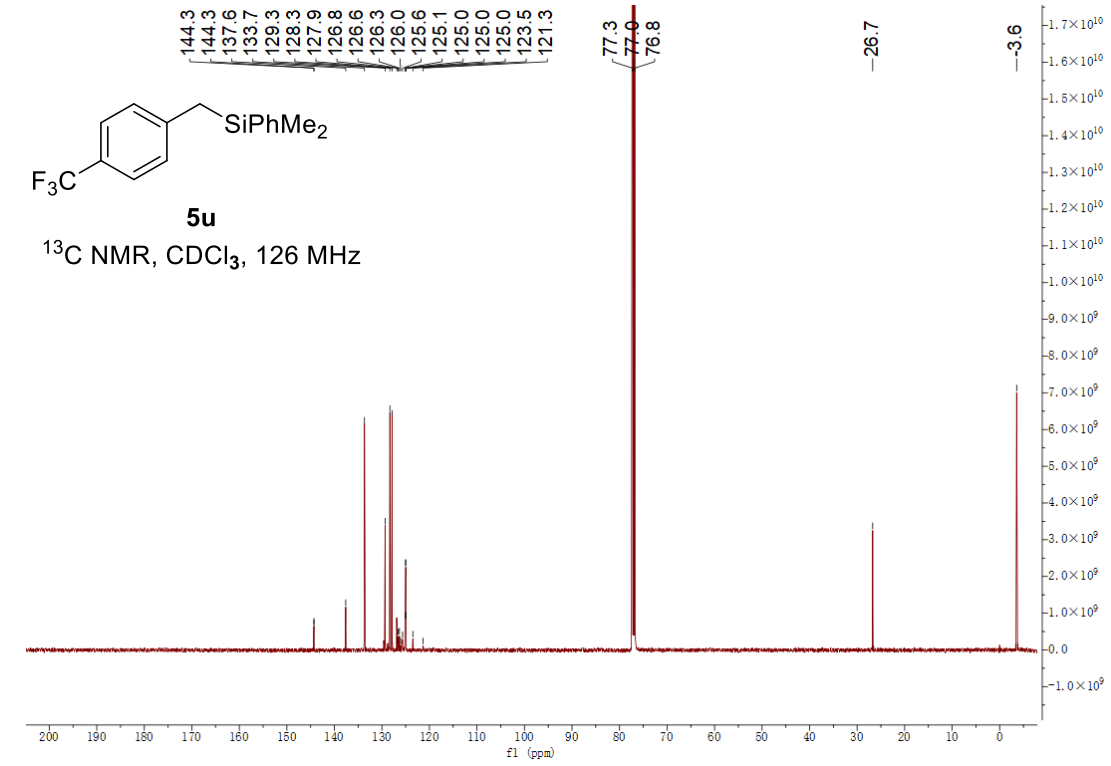
5t

^{13}C NMR, $CDCl_3$, 126 MHz



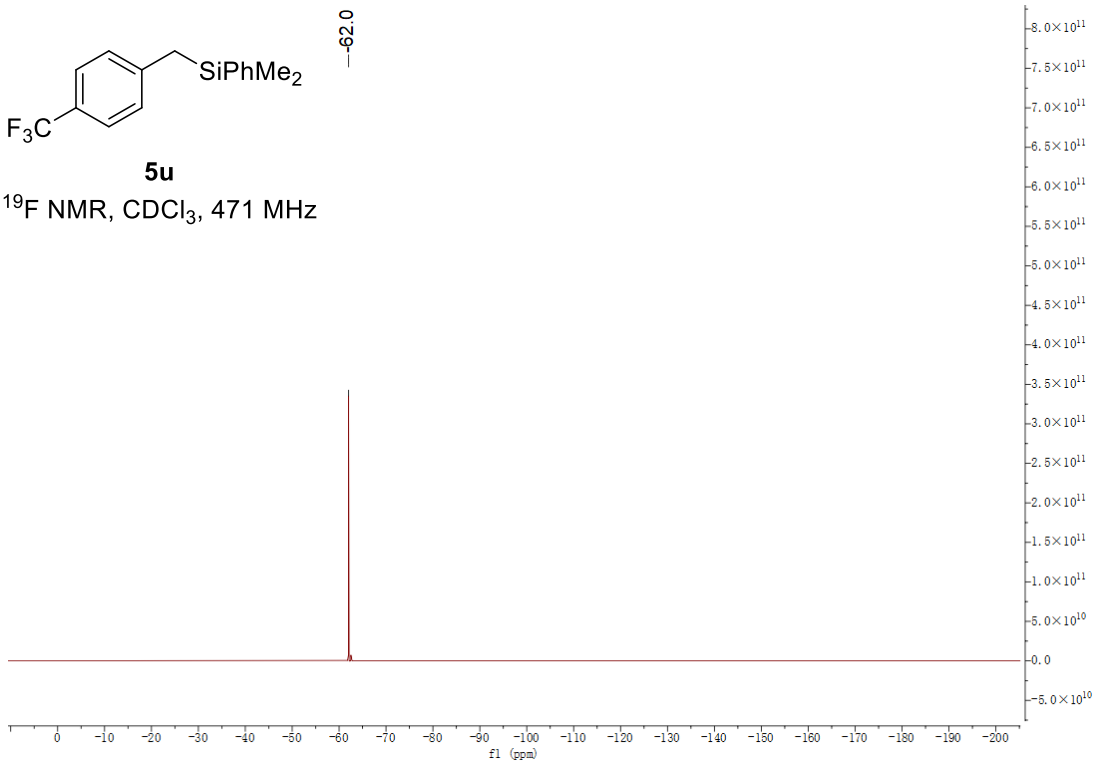
5u

^{1}H NMR, $CDCl_3$, 500 MHz



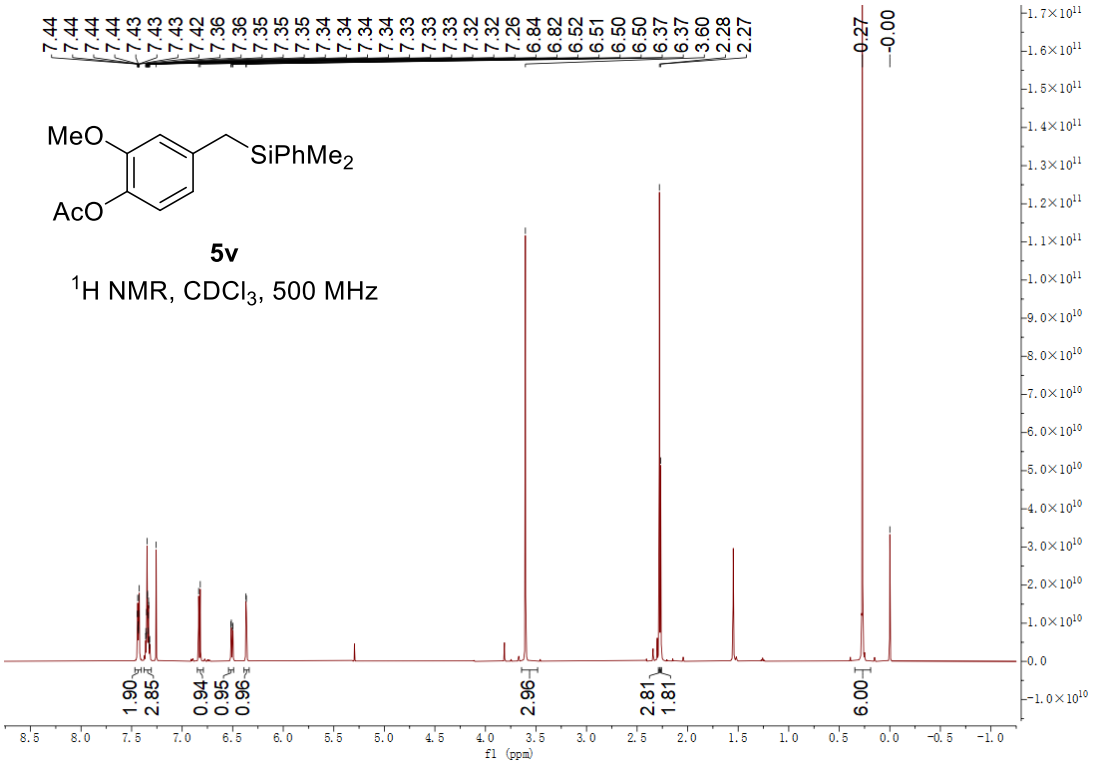
5u

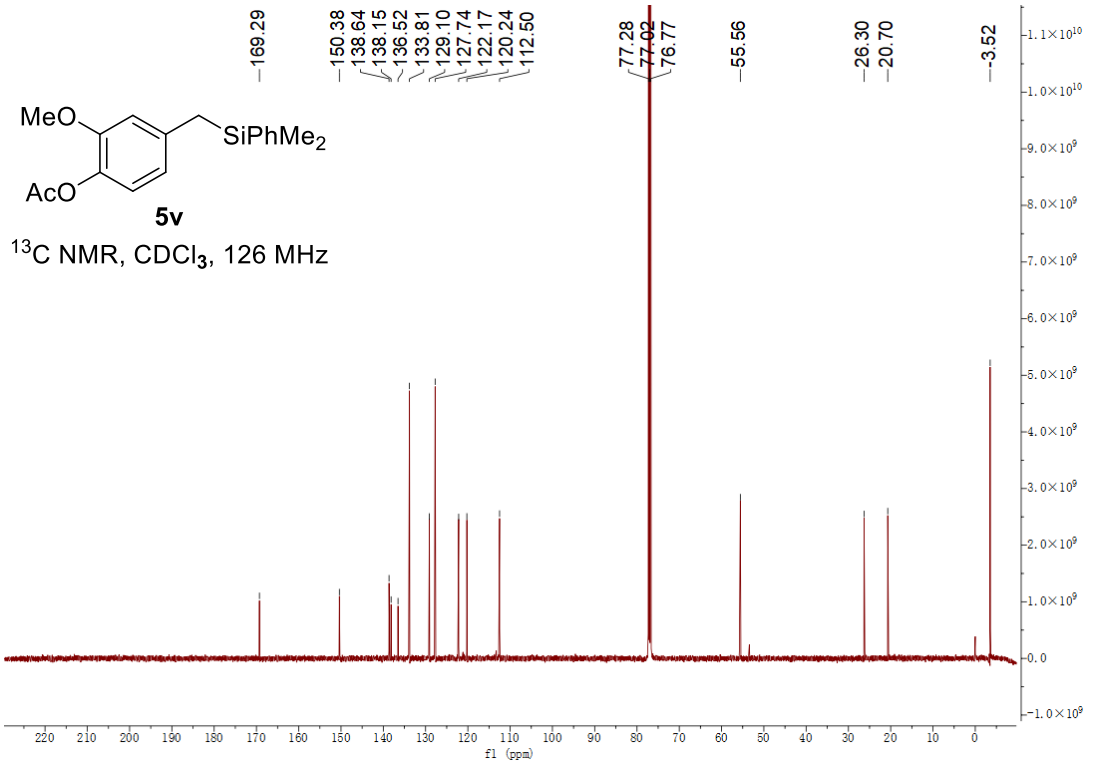
^{13}C NMR, $CDCl_3$, 126 MHz

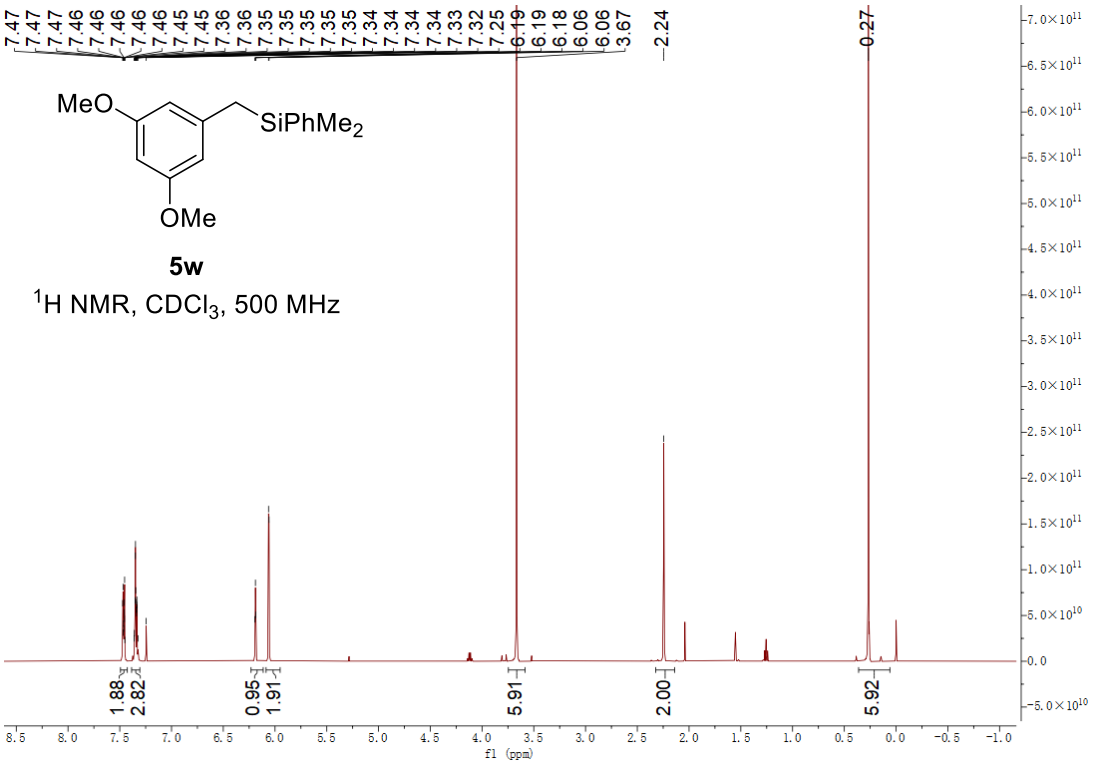


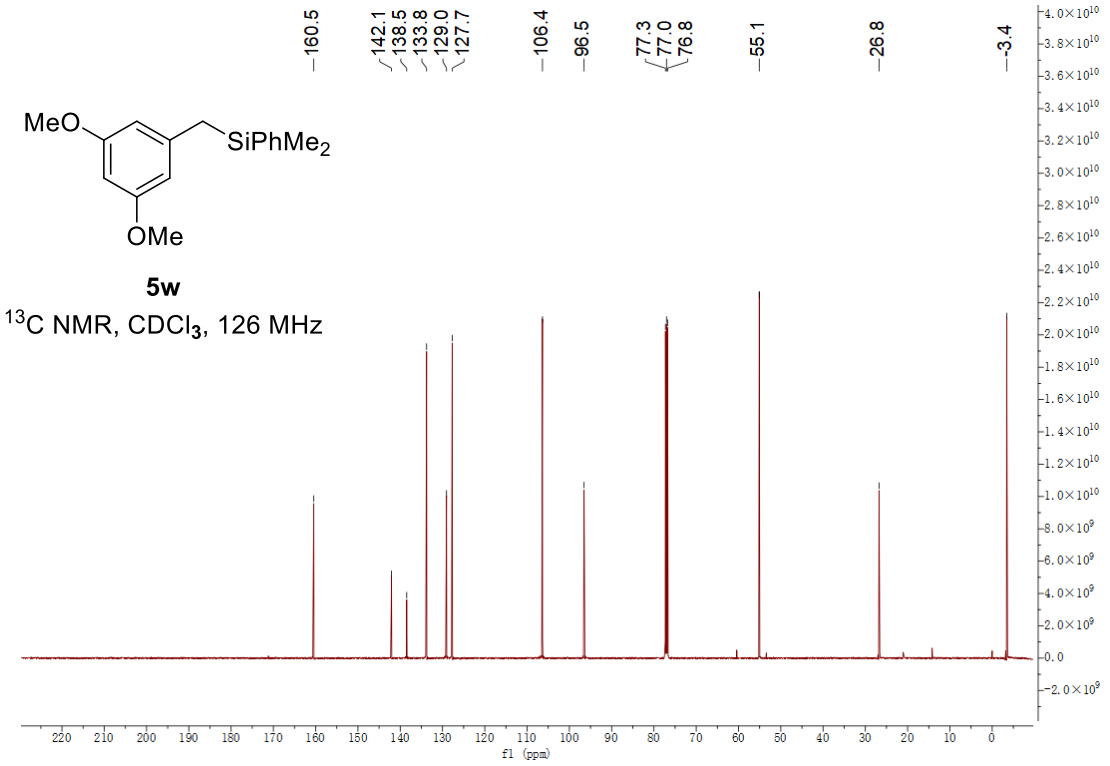
5u

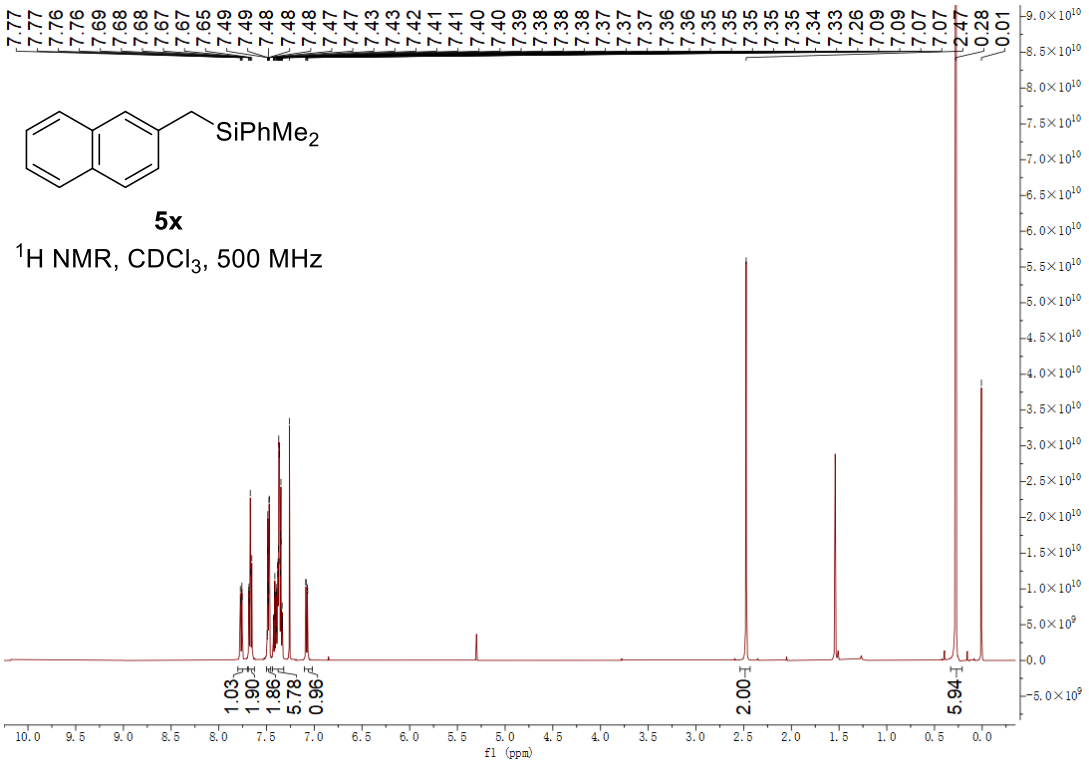
^{19}F NMR, $CDCl_3$, 471 MHz

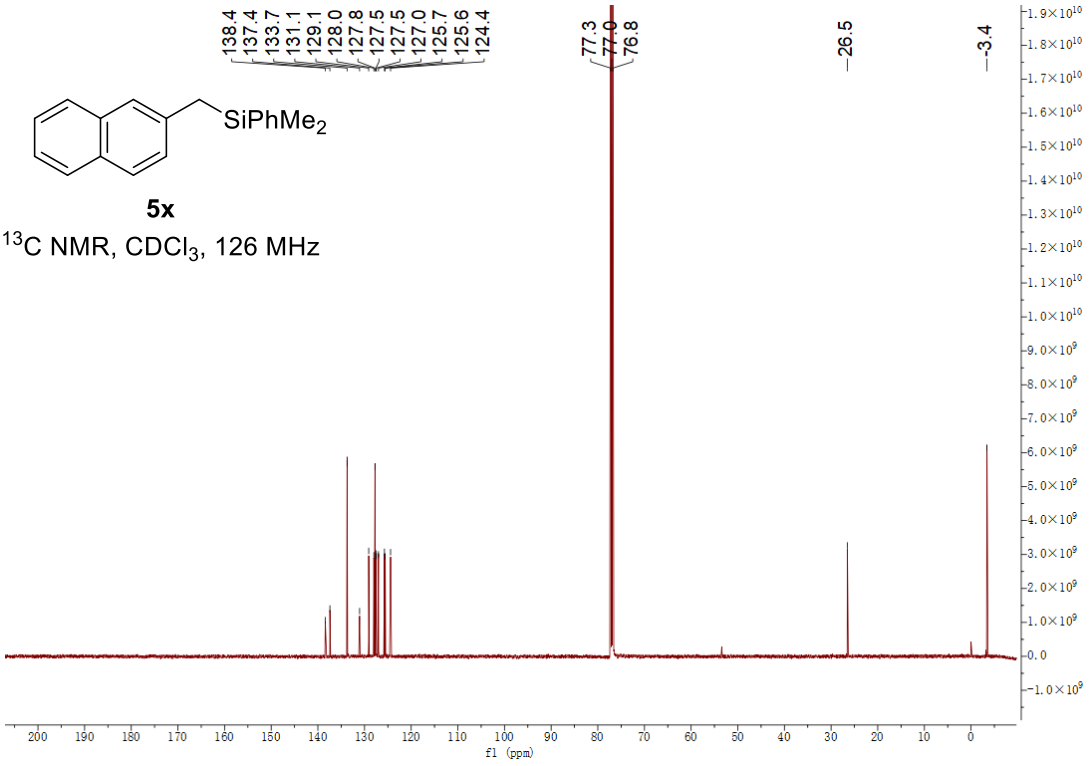

MeO
SiPhMe2
AcO
5v
1H NMR, CDCl3, 500 MHz

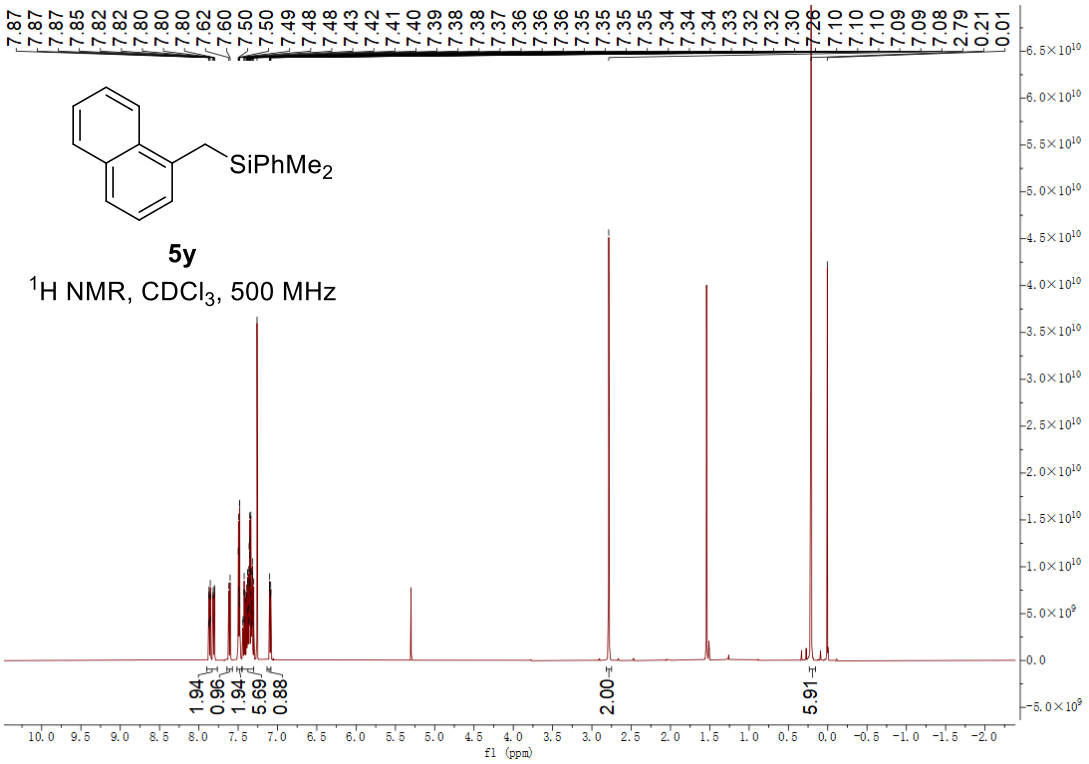

MeO
SiPhMe2
AcO
5v
13C NMR, CDCl3, 126 MHz

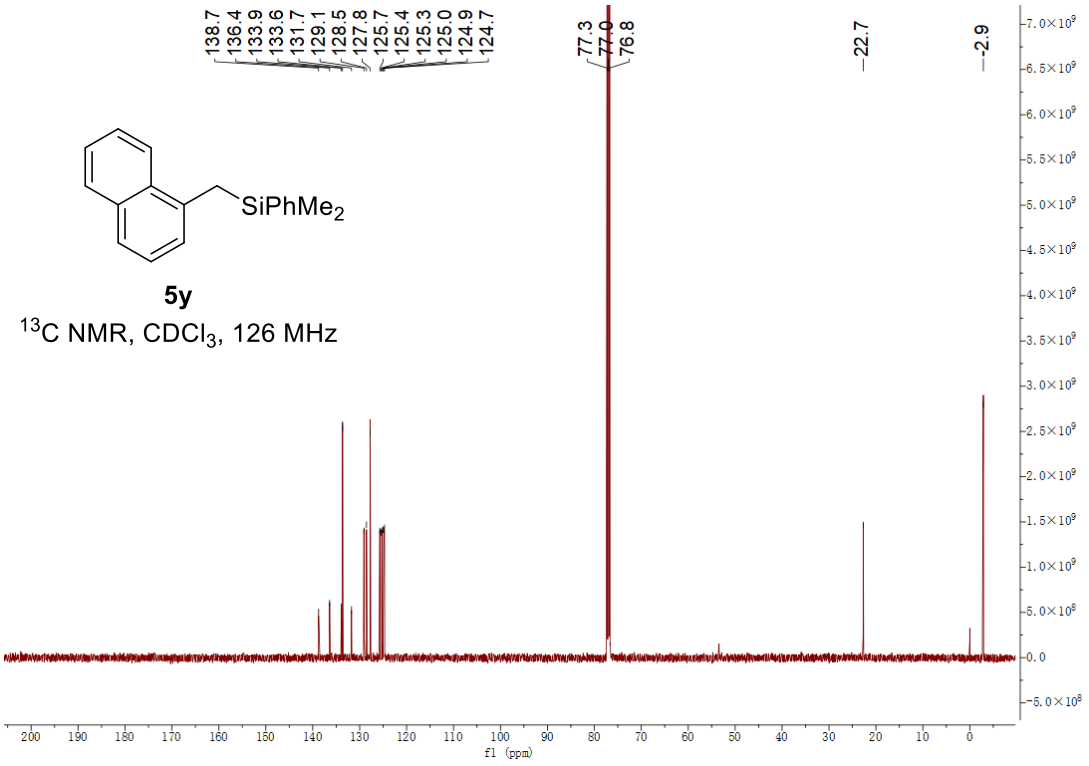

MeO
SiPhMe2
OMe
5w
1H NMR, CDCl3, 500 MHz
7.47
7.47
7.47
7.46
7.46
7.46
7.46
7.45
7.45
7.36
7.36
7.35
7.35
7.35
7.35
7.34
7.34
7.34
7.34
7.33
7.32
7.25
6.19
6.19
6.18
6.06
6.06
3.67
2.24
0.27
1.88
2.82
0.95
1.91
5.91
2.00
5.92
f1 (ppm)

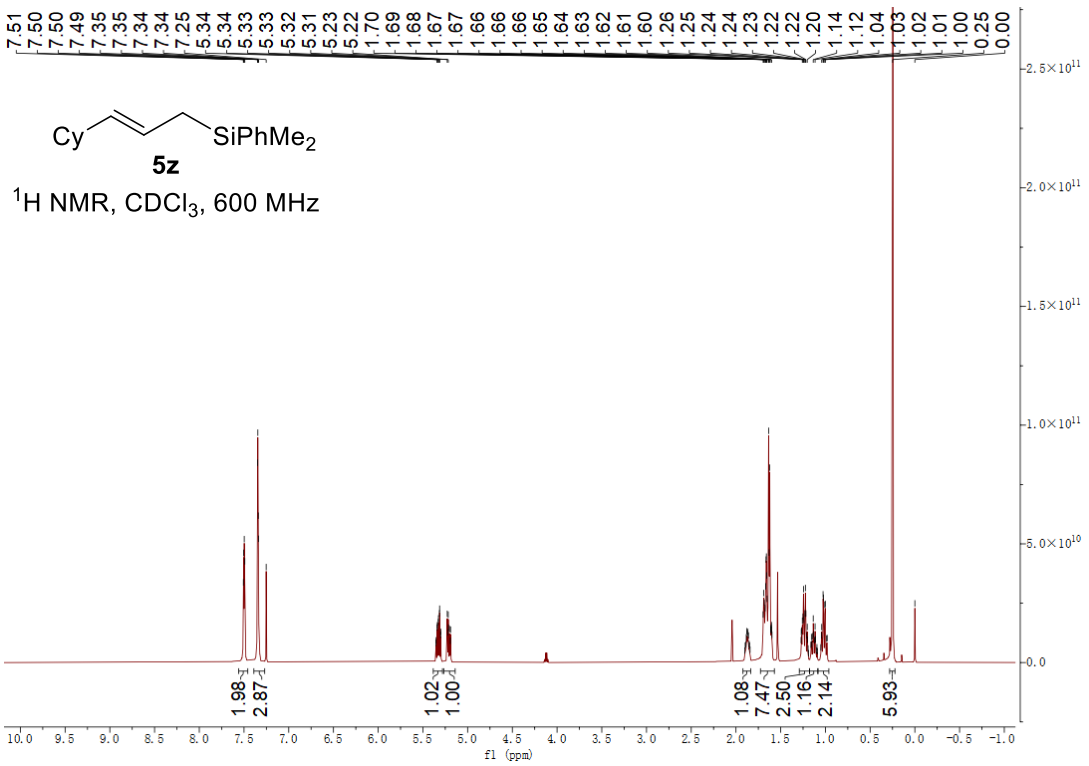

MeO
SiPhMe2
OMe
5w
13C NMR, CDCl3, 126 MHz
160.5
142.1
138.5
133.8
129.0
127.7
106.4
96.5
77.3
77.0
76.8
55.1
26.8
-3.4
f1 (ppm)

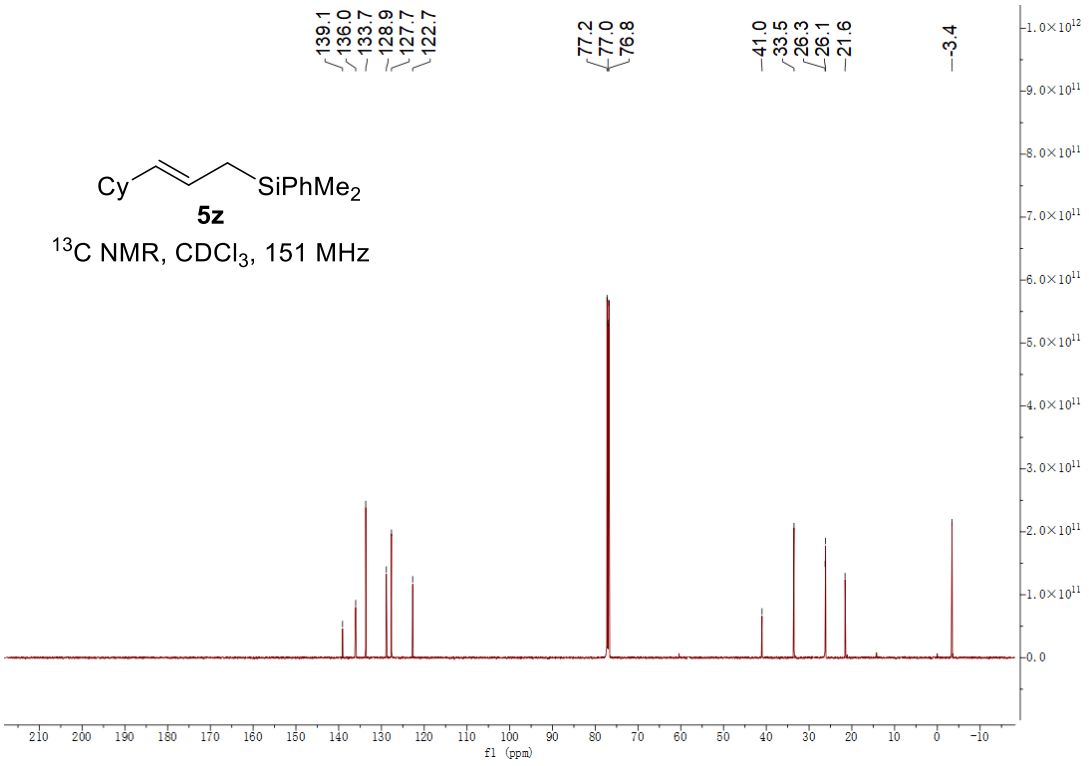

SiPhMe2
5x
1H NMR, CDCl3, 500 MHz
1.03
1.90
1.86
5.78
0.96
2.00
5.94
f1 (ppm)

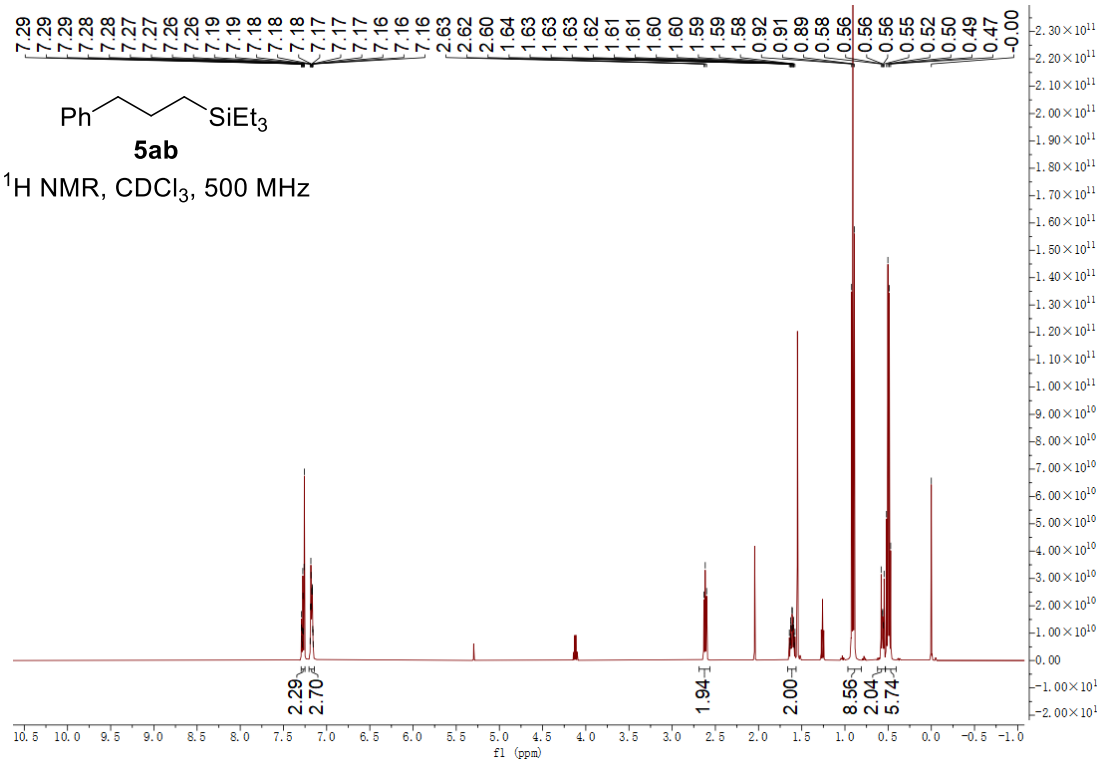

SiPhMe2
5x
13C NMR, CDCl3, 126 MHz
138.4
137.4
133.7
131.1
129.1
128.0
127.8
127.5
127.5
127.0
125.7
125.6
124.4
77.3
77.0
76.8
26.5
-3.4
f1 (ppm)

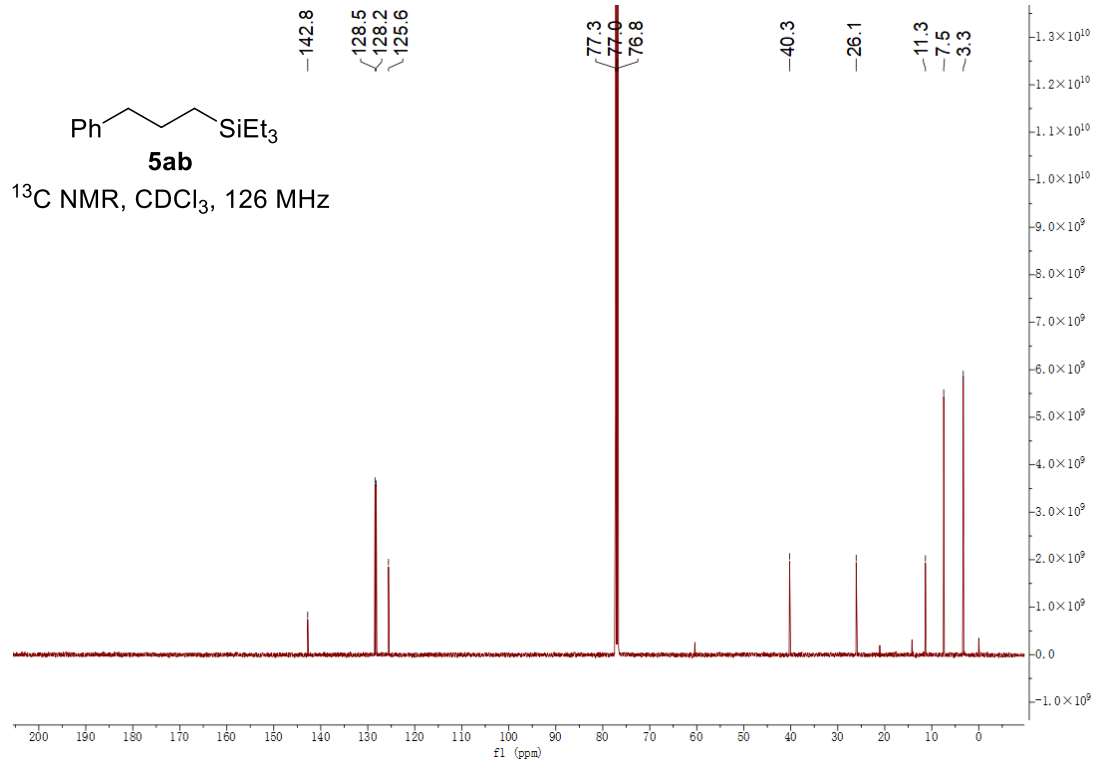

SiPhMe2
5y
1H NMR, CDCl3, 500 MHz
f1 (ppm)

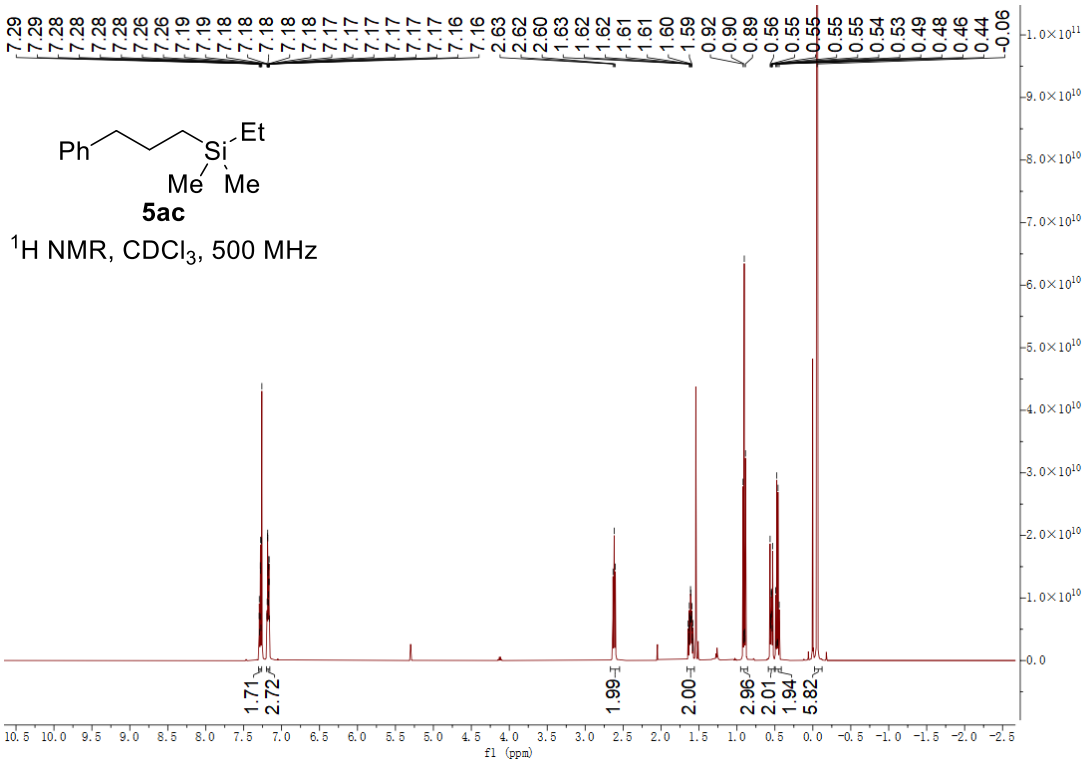

SiPhMe2
5y
13C NMR, CDCl3, 126 MHz
f1 (ppm)

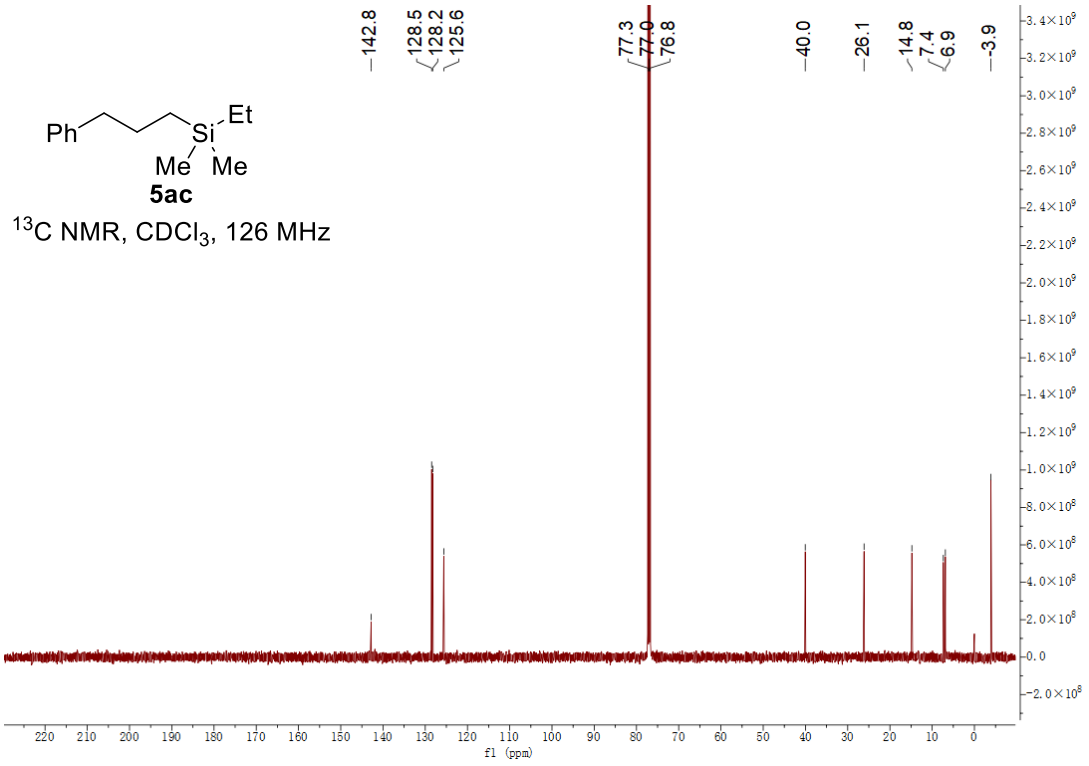

Cy
SiPhMe2
5z
1H NMR, CDCl3, 600 MHz
f1 (ppm)

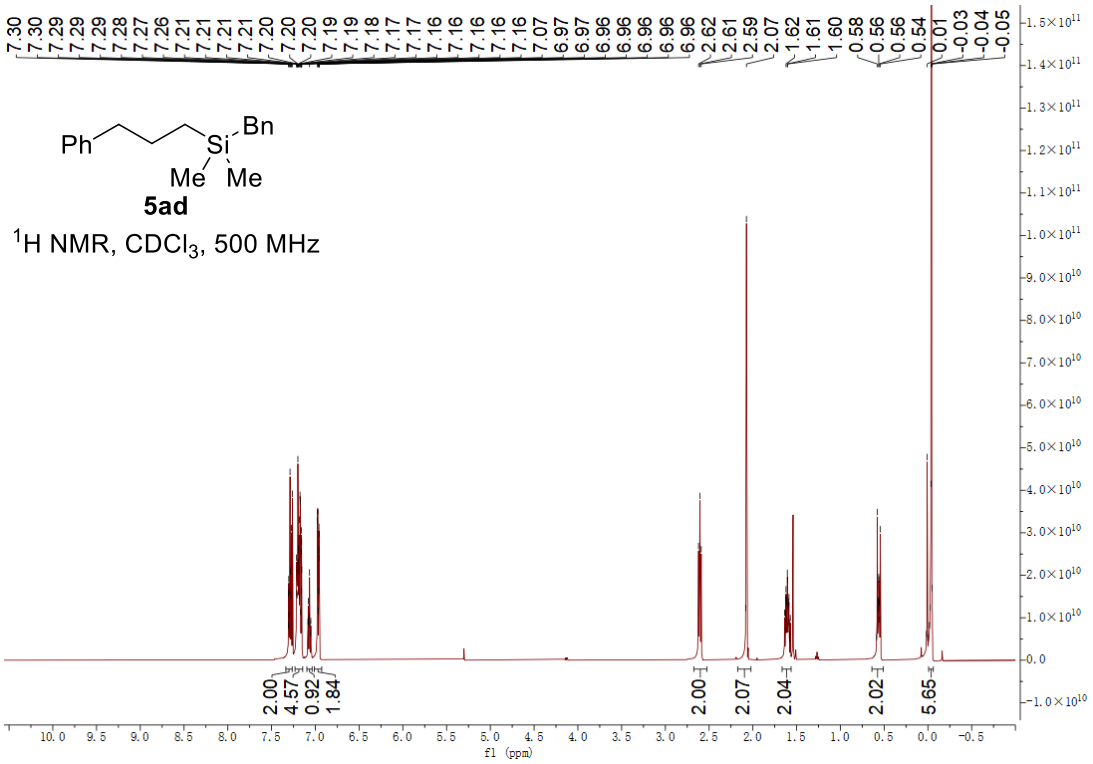

Cy
SiPhMe2
5z
13C NMR, CDCl3, 151 MHz
f1 (ppm)

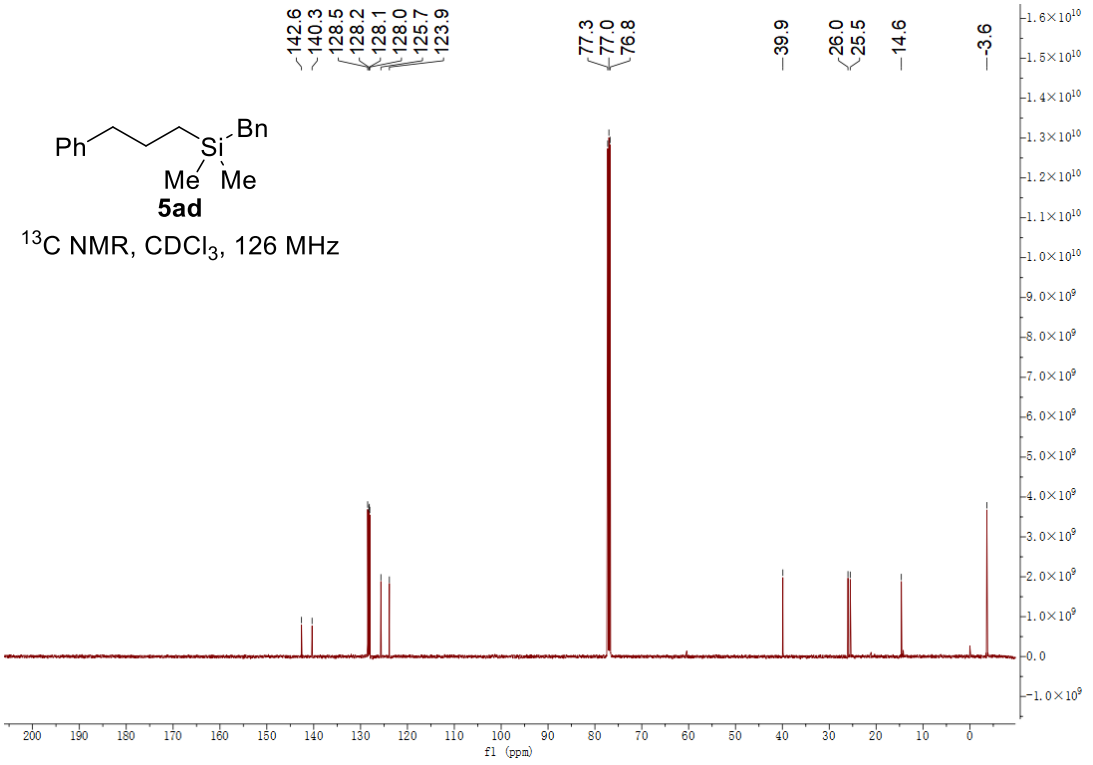

Ph
SiEt3
5ab
1H NMR, CDCl3, 500 MHz
f1 (ppm)

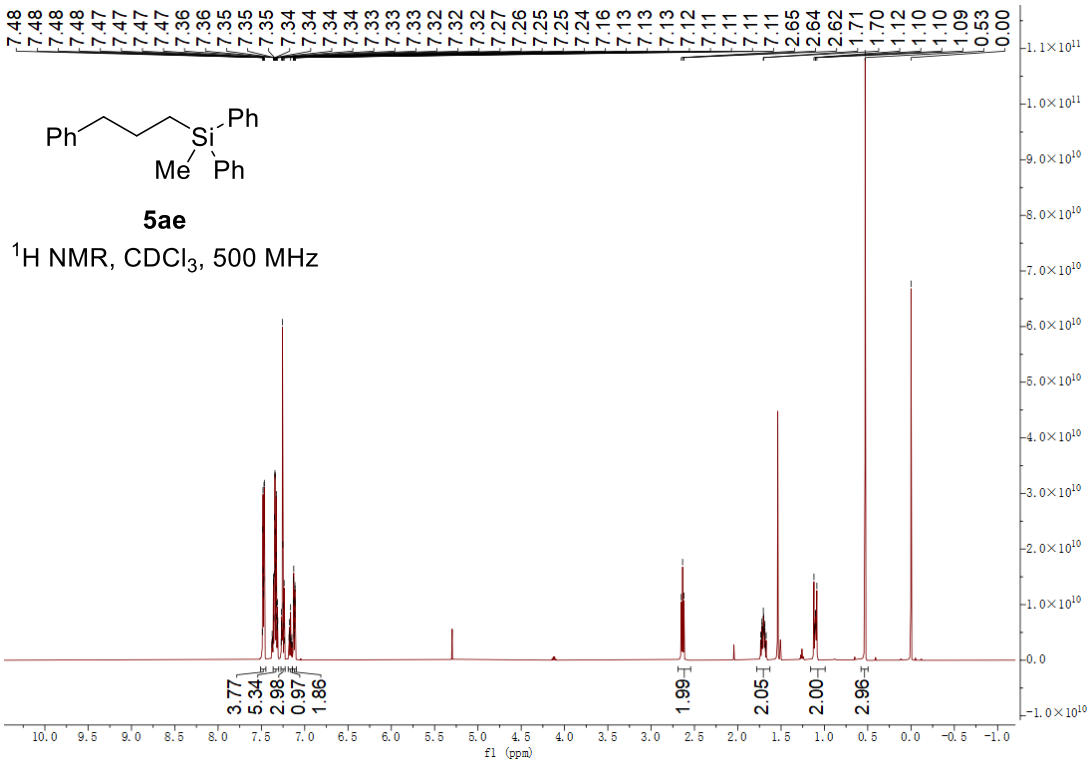

Ph
SiEt3
5ab
13C NMR, CDCl3, 126 MHz
f1 (ppm)


Ph
Si
Et
Me
Me
5ac
1H NMR, CDCl3, 500 MHz
f1 (ppm)


Ph
Si
Et
Me
Me
5ac
13C NMR, CDCl3, 126 MHz
f1 (ppm)

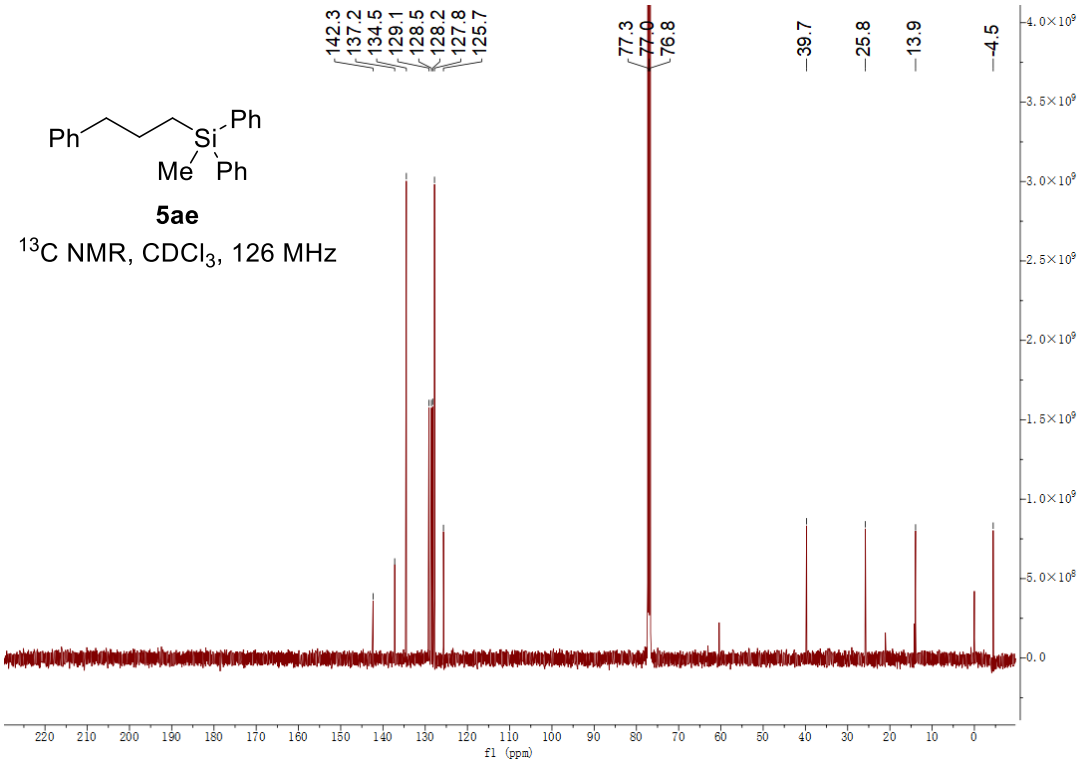

Ph
Si
Bn
Me
Me
5ad
1H NMR, CDCl3, 500 MHz
f1 (ppm)


Ph
Si
Bn
Me
Me
5ad
13C NMR, CDCl3, 126 MHz
f1 (ppm)



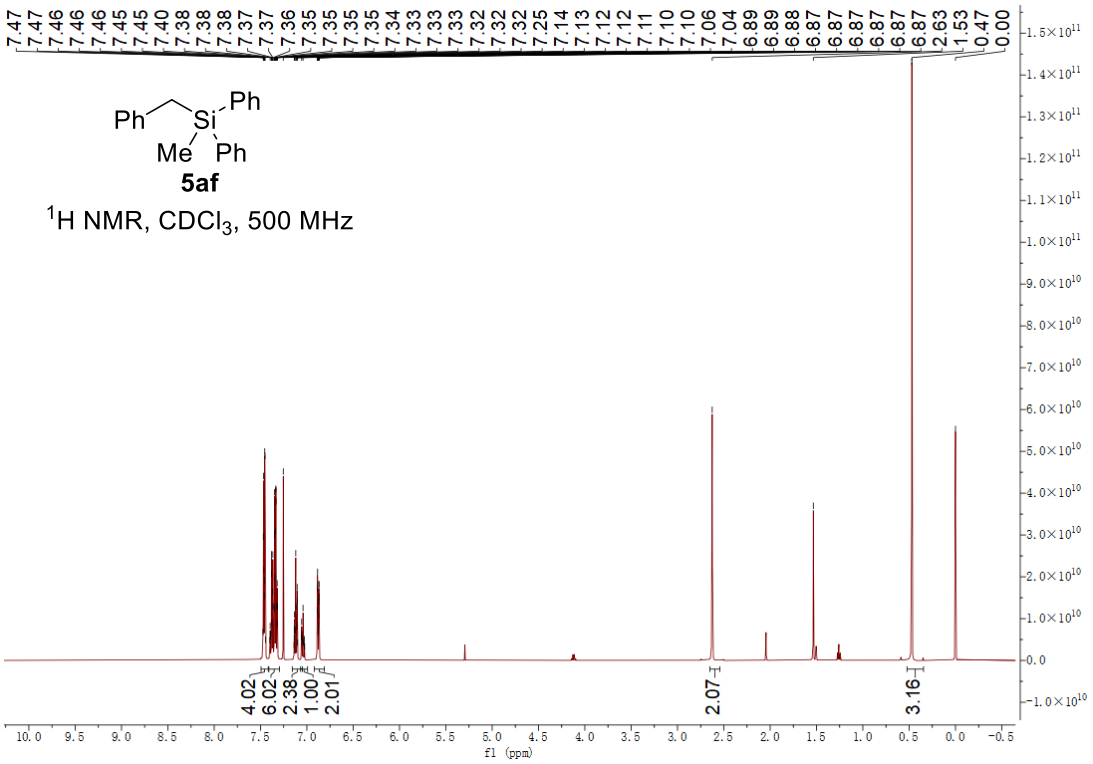
5ae

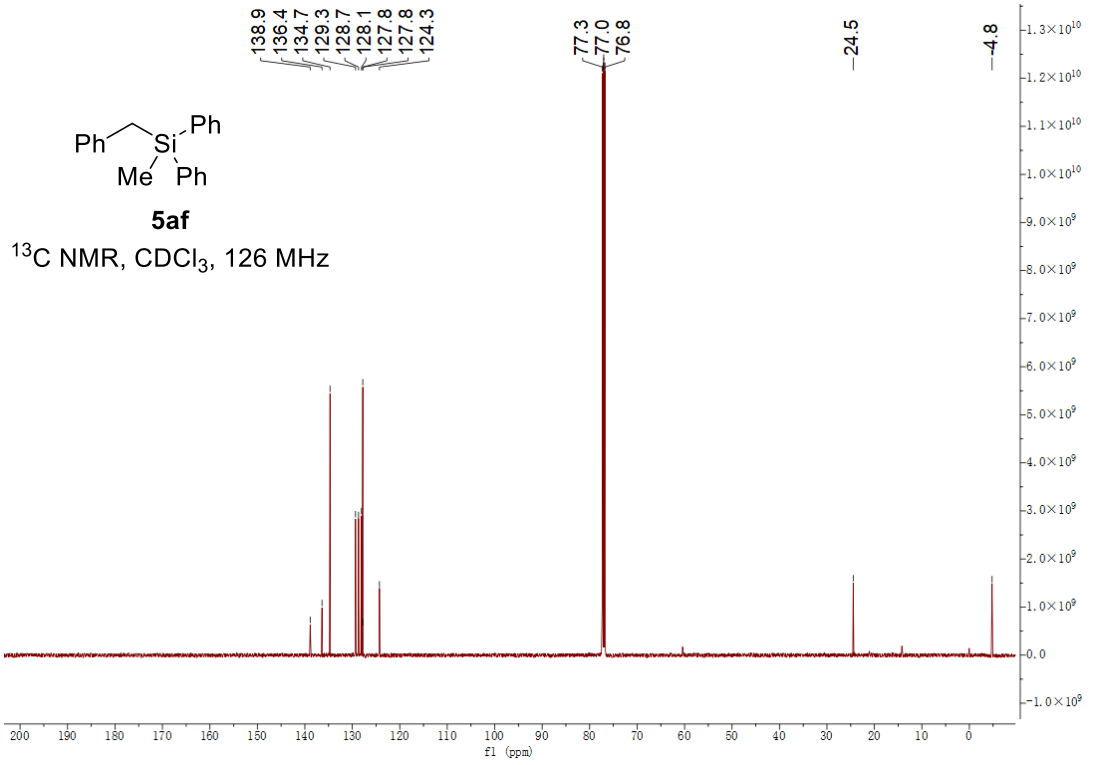
^{1}H NMR, $CDCl_3$, 500 MHz

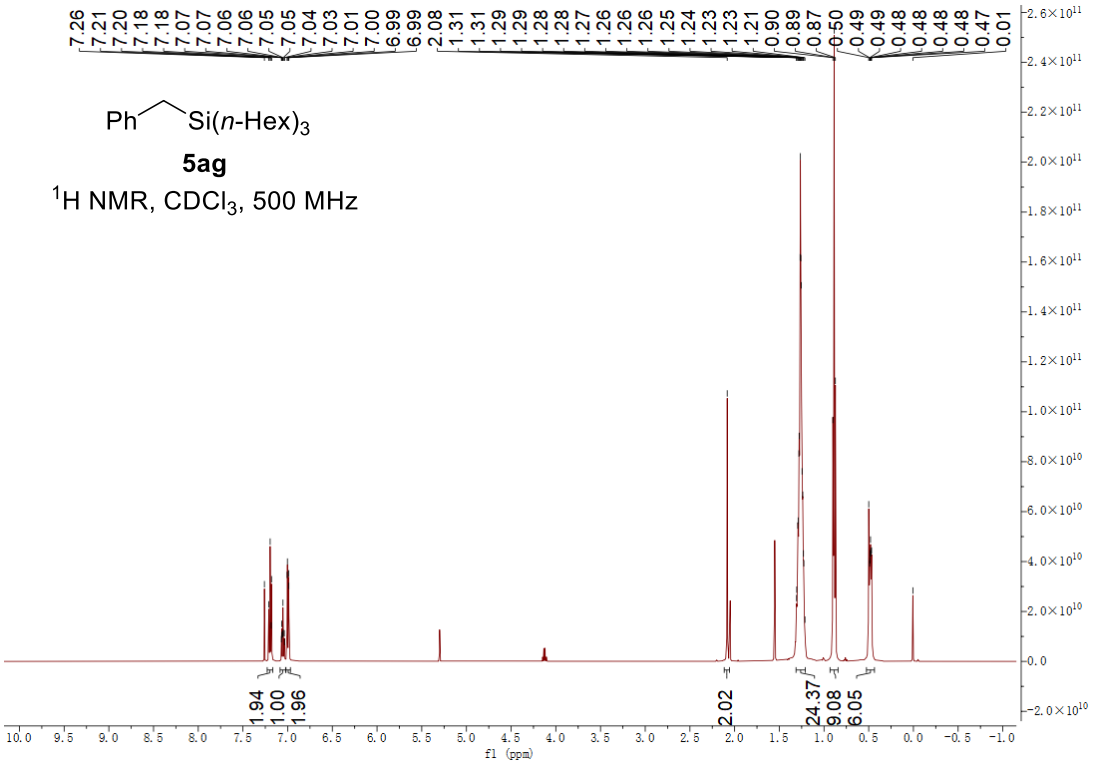


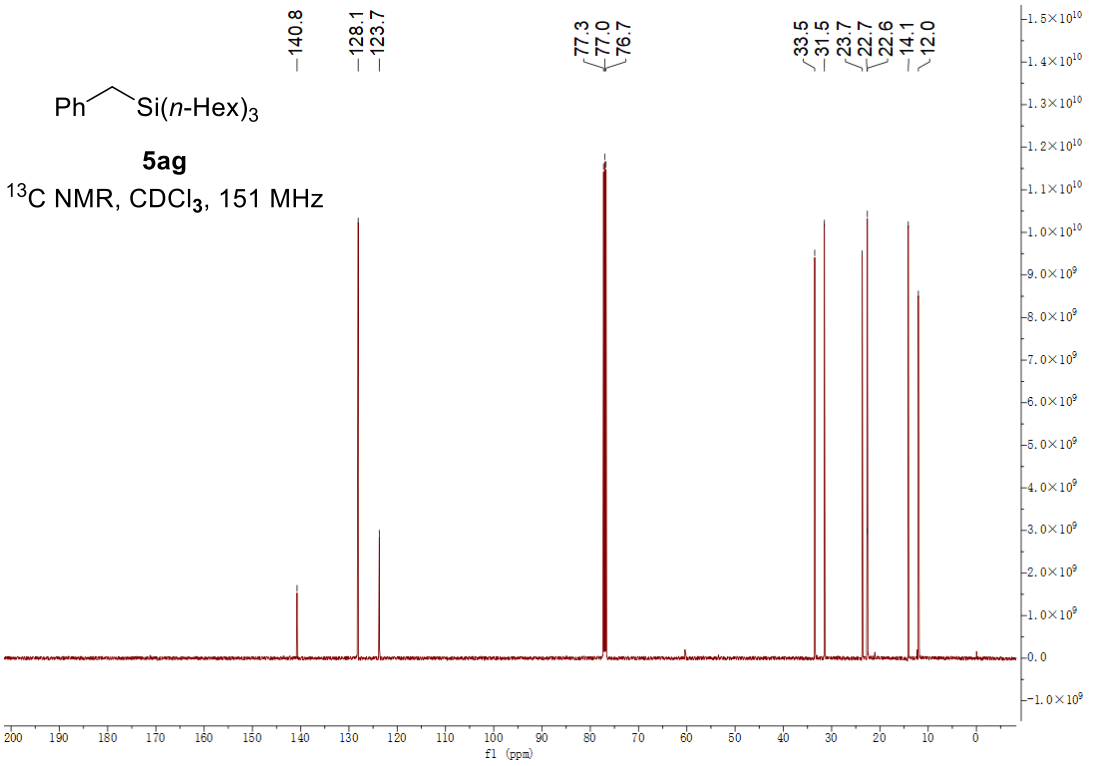
5ae

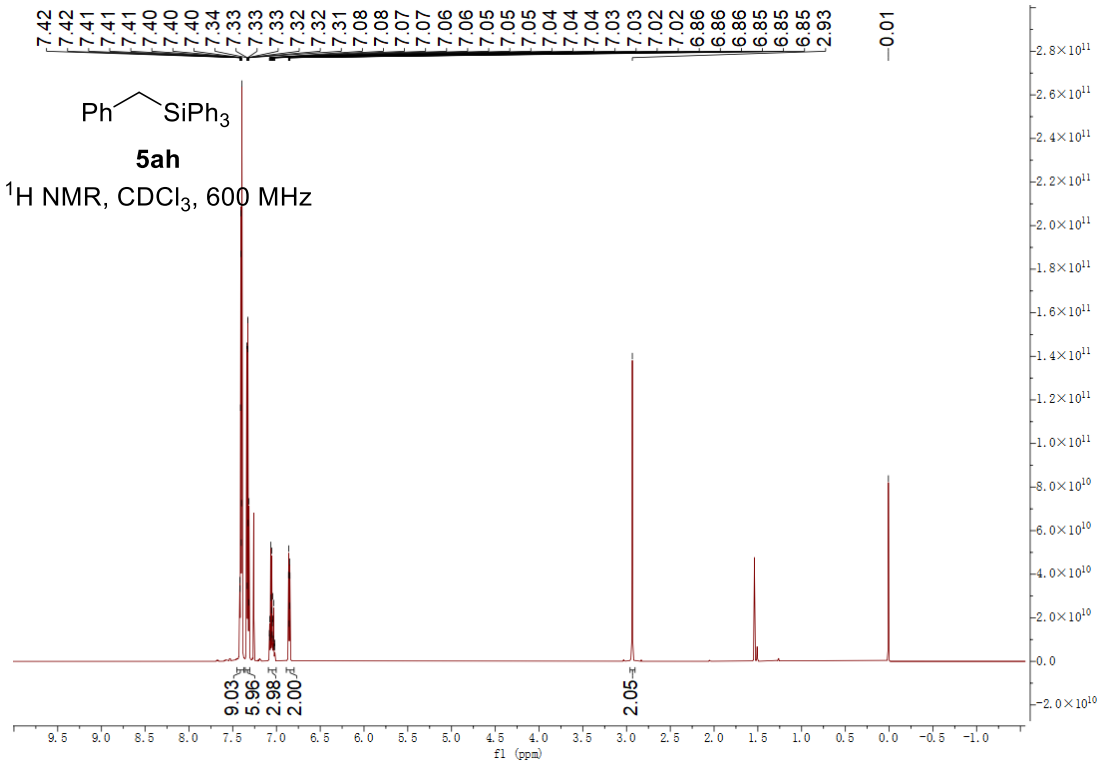
^{13}C NMR, $CDCl_3$, 126 MHz

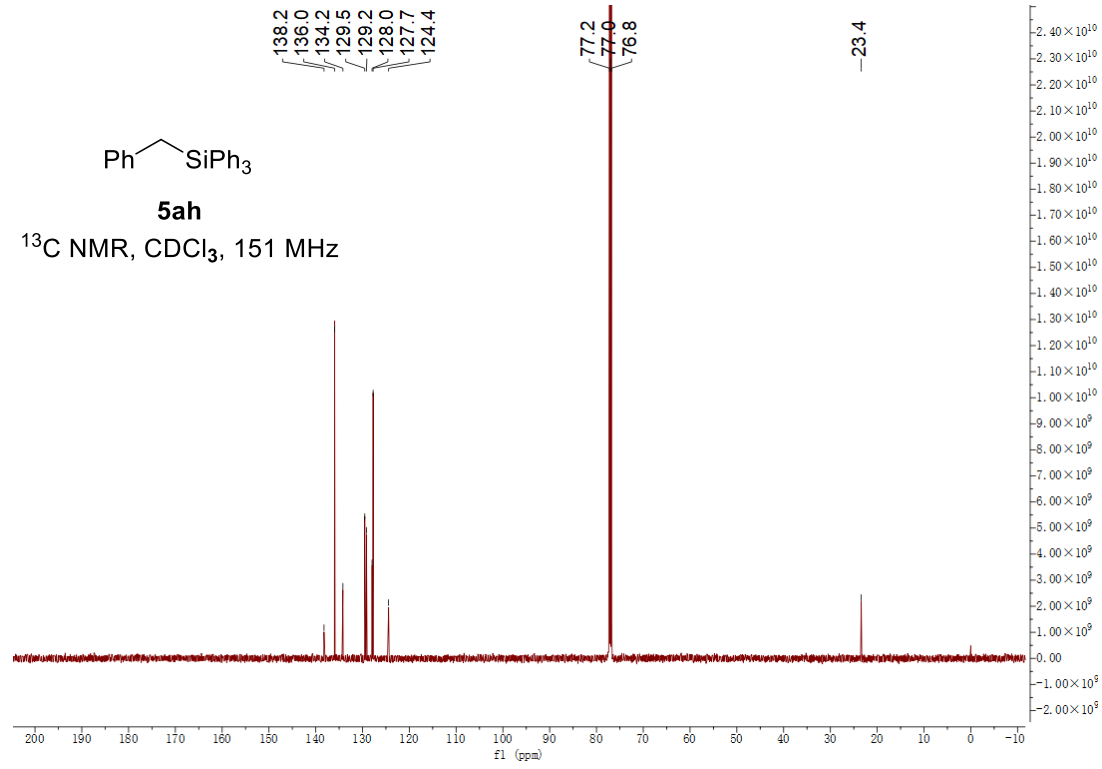

7.47 7.47 7.46 7.46 7.45 7.45 7.40 7.38 7.38 7.37 7.37 7.36 7.35 7.35 7.35 7.34 7.33 7.33 7.32 7.32 7.25 7.14 7.13 7.12 7.12 7.11 7.10 7.10 7.06 7.04 6.89 6.89 6.88 6.87 6.87 6.87 6.87 6.87 2.63 1.53 0.47 0.00
Ph Si Ph Me Ph
5af
1H NMR, CDCl3, 500 MHz
4.02 6.02 2.38 1.00 2.01 2.07 3.16
fl (ppm)

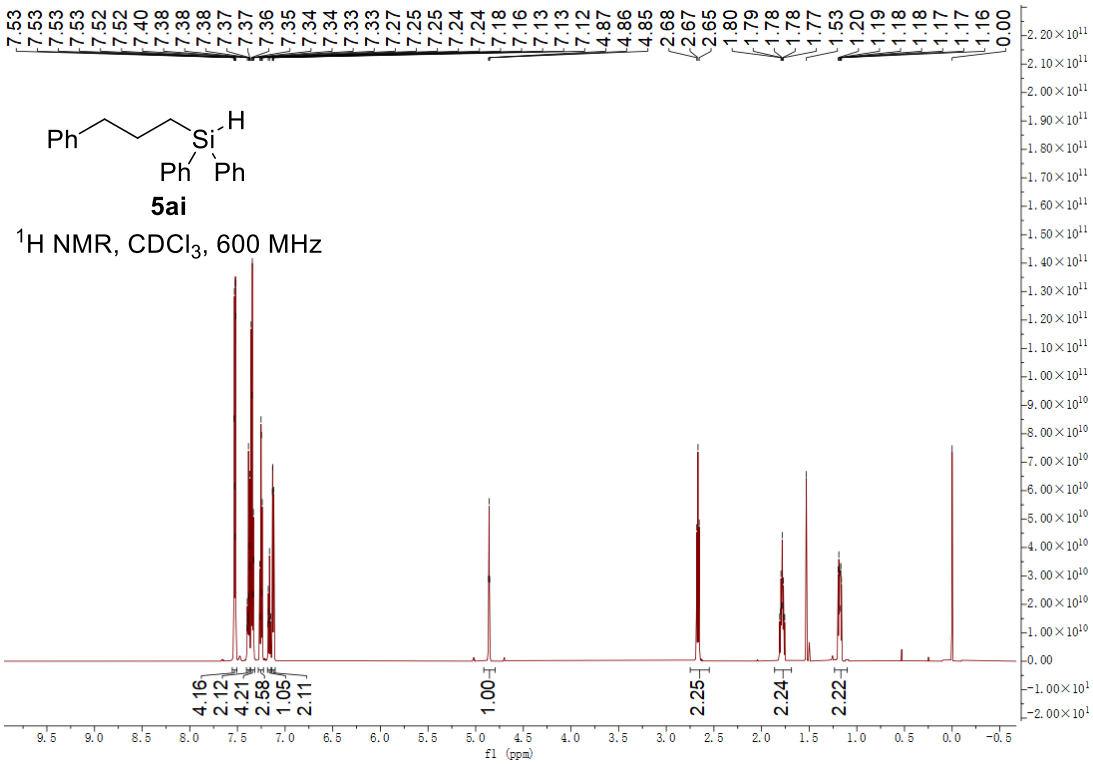

138.9 136.4 134.7 129.3 128.7 128.1 127.8 127.8 124.3 77.3 77.0 76.8 24.5 4.8
Ph Si Ph Me Ph
5af
13C NMR, CDCl3, 126 MHz
fl (ppm)

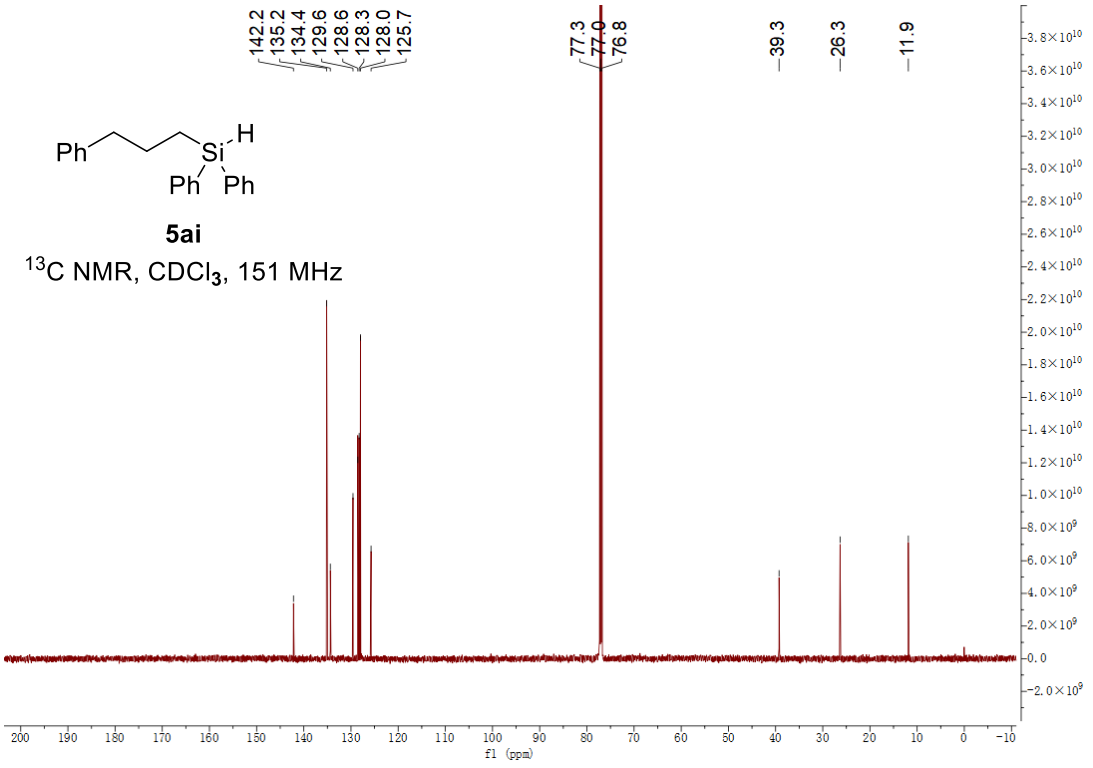

Ph
Si(n-Hex)3
5ag
1H NMR, CDCl3, 500 MHz
f1 (ppm)

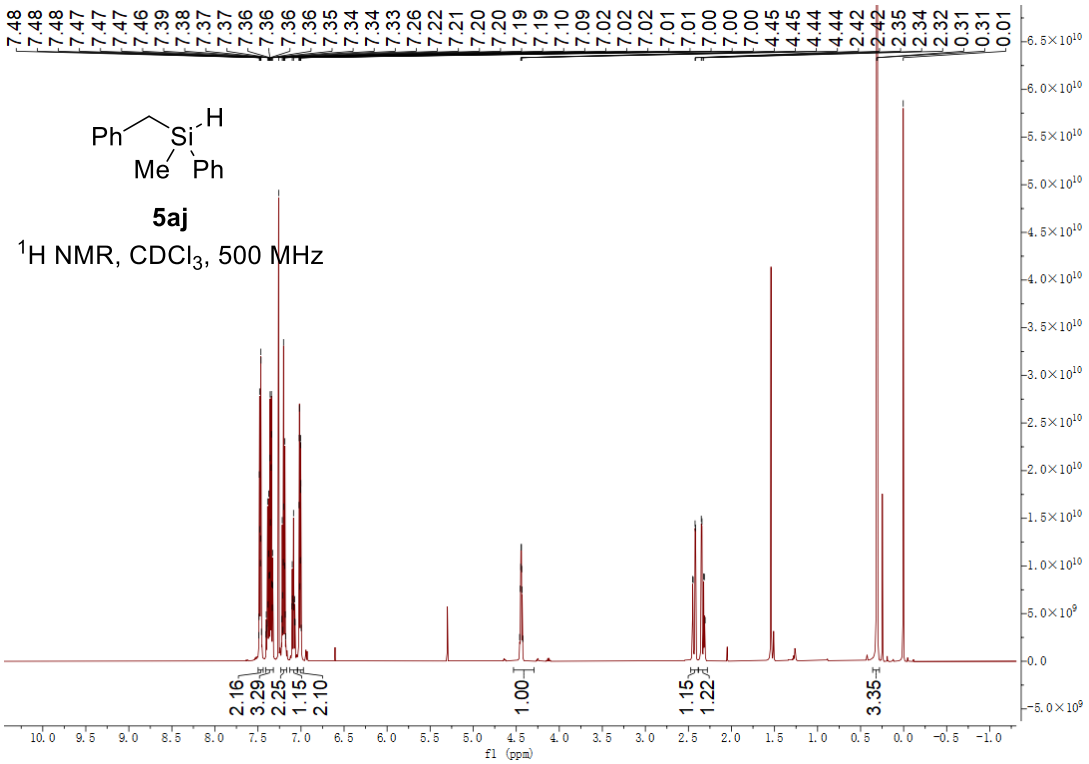

Ph
Si(n-Hex)3
5ag
13C NMR, CDCl3, 151 MHz
f1 (ppm)

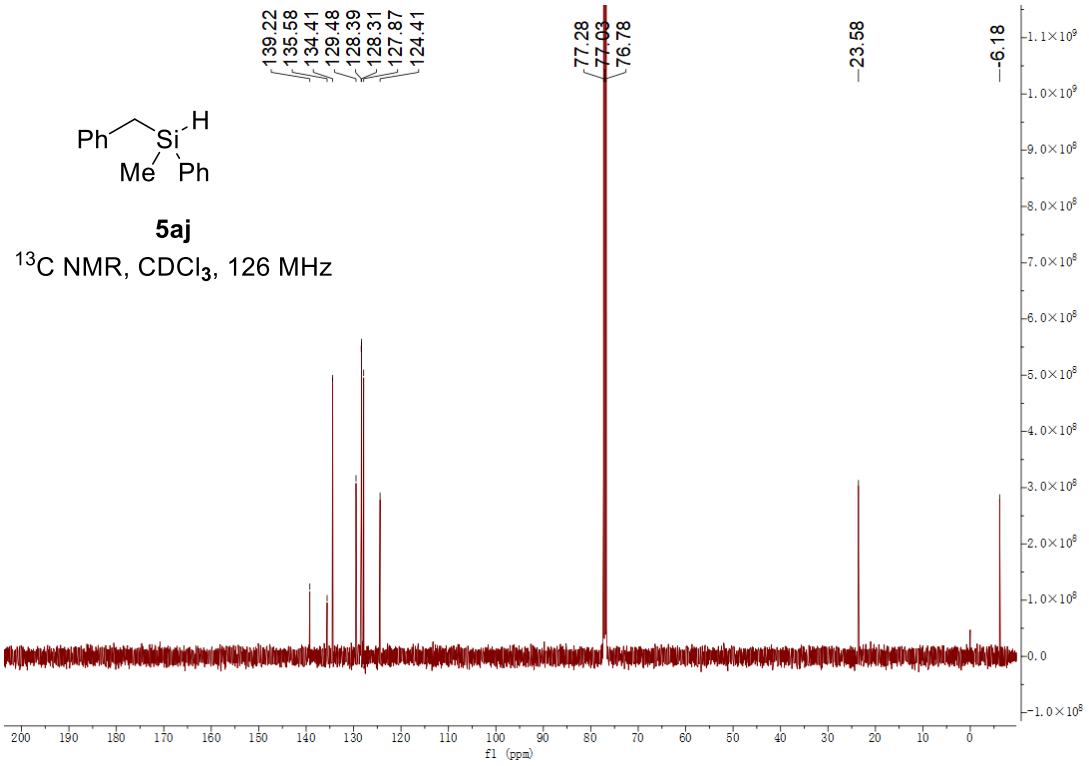

Ph
SiPh3
5ah
1H NMR, CDCl3, 600 MHz
9.03
5.96
2.98
2.00
2.05
f1 (ppm)

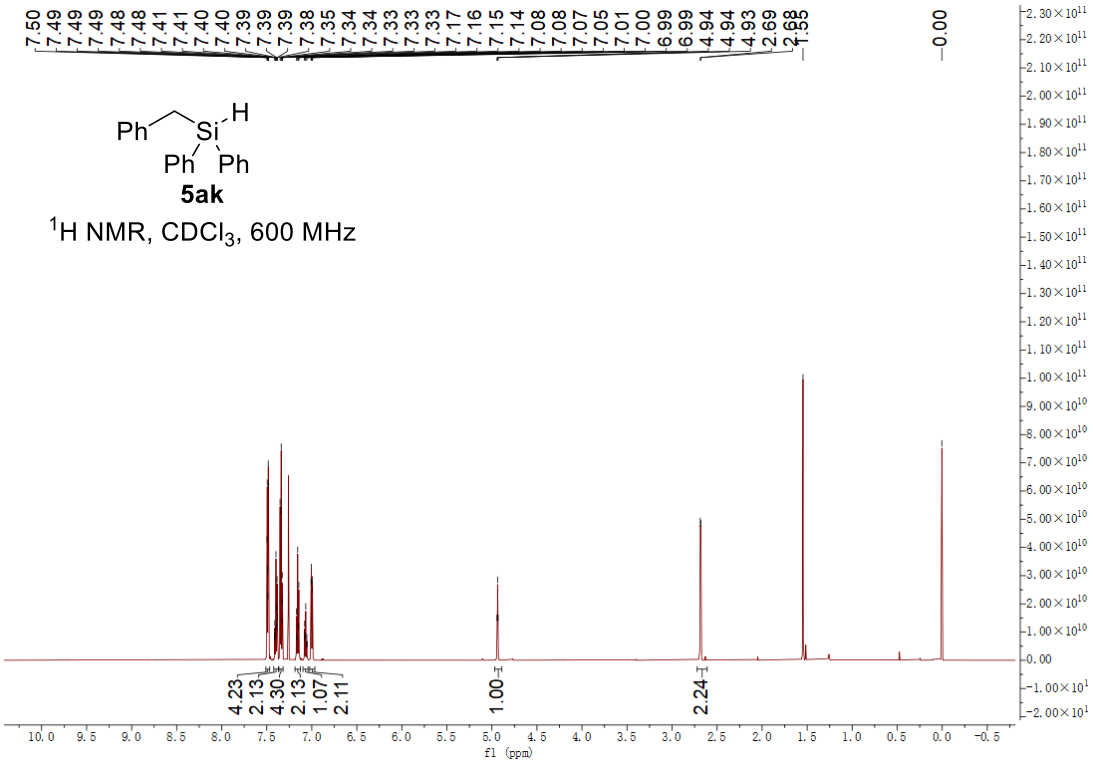

Ph
SiPh3
5ah
13C NMR, CDCl3, 151 MHz
f1 (ppm)

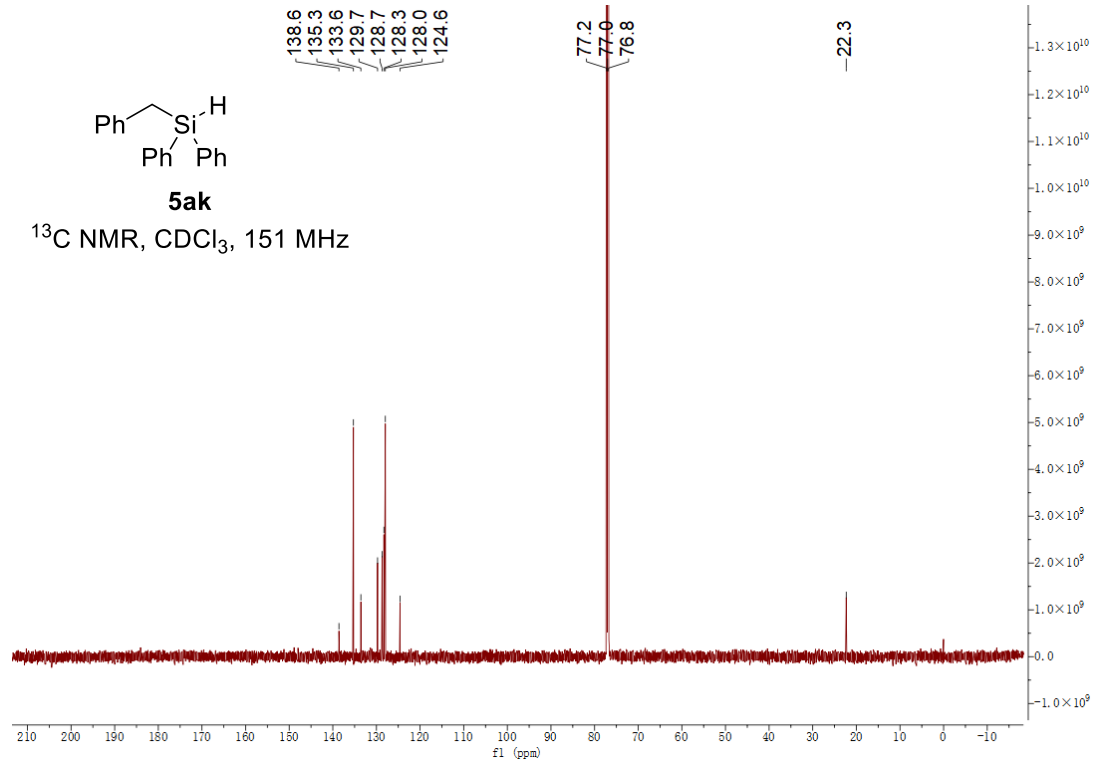

Ph
Si
H
Ph
Ph
5ai
1H NMR, CDCl3, 600 MHz
f1 (ppm)

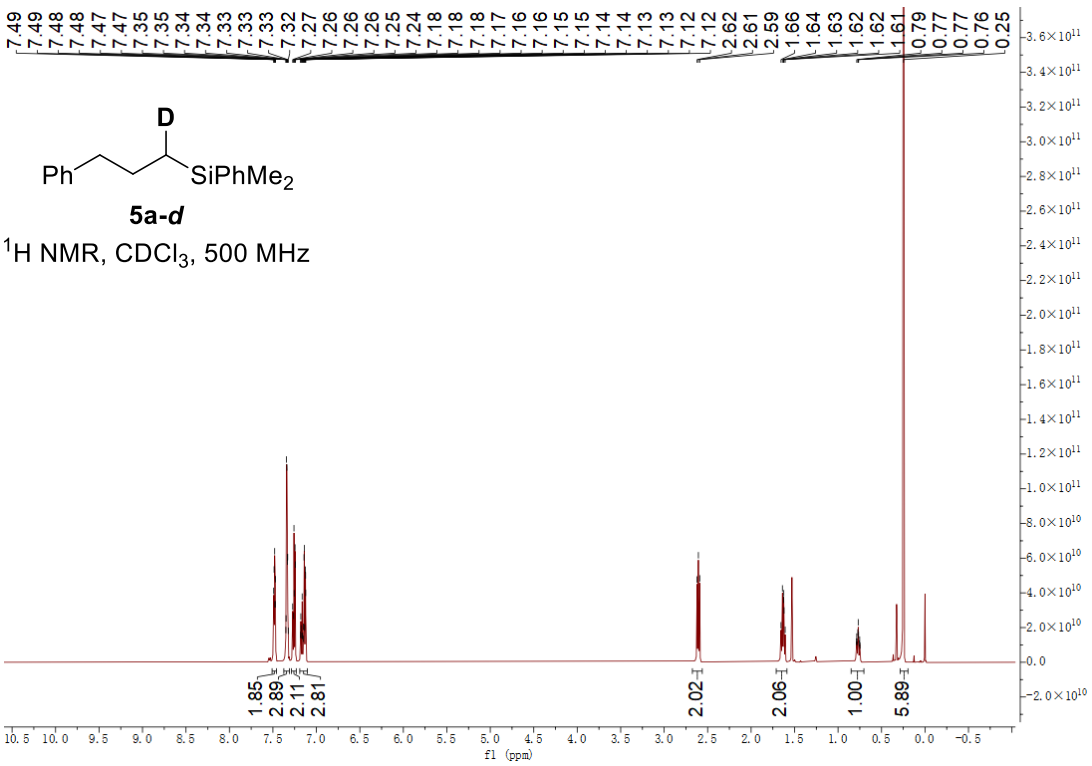

Ph
Si
H
Ph
Ph
5ai
13C NMR, CDCl3, 151 MHz
f1 (ppm)


Ph
Si
H
Me
Ph
5aj
1H NMR, CDCl3, 500 MHz
f1 (ppm)


Ph
Si
H
Me
Ph
5aj
13C NMR, CDCl3, 126 MHz
f1 (ppm)


7.50 7.49 7.49 7.48 7.48 7.41 7.41 7.40 7.40 7.39 7.39 7.39 7.38 7.35 7.34 7.34 7.33 7.33 7.33 7.17 7.16 7.15 7.14 7.08 7.08 7.07 7.05 7.01 7.00 6.99 6.99 4.94 4.94 4.93 2.69 2.68 1.55
0.00
Ph Si H Ph Ph
5ak
1H NMR, CDCl3, 600 MHz
4.23 2.13 4.30 2.13 1.07 2.11 1.00 2.24
f1 (ppm)


138.6 135.3 133.6 129.7 128.7 128.3 128.0 124.6
77.2 77.0 76.8
22.3
Ph Si H Ph Ph
5ak
13C NMR, CDCl3, 151 MHz
f1 (ppm)


7.49
7.49
7.48
7.48
7.47
7.47
7.35
7.35
7.34
7.34
7.33
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7.32
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7.26
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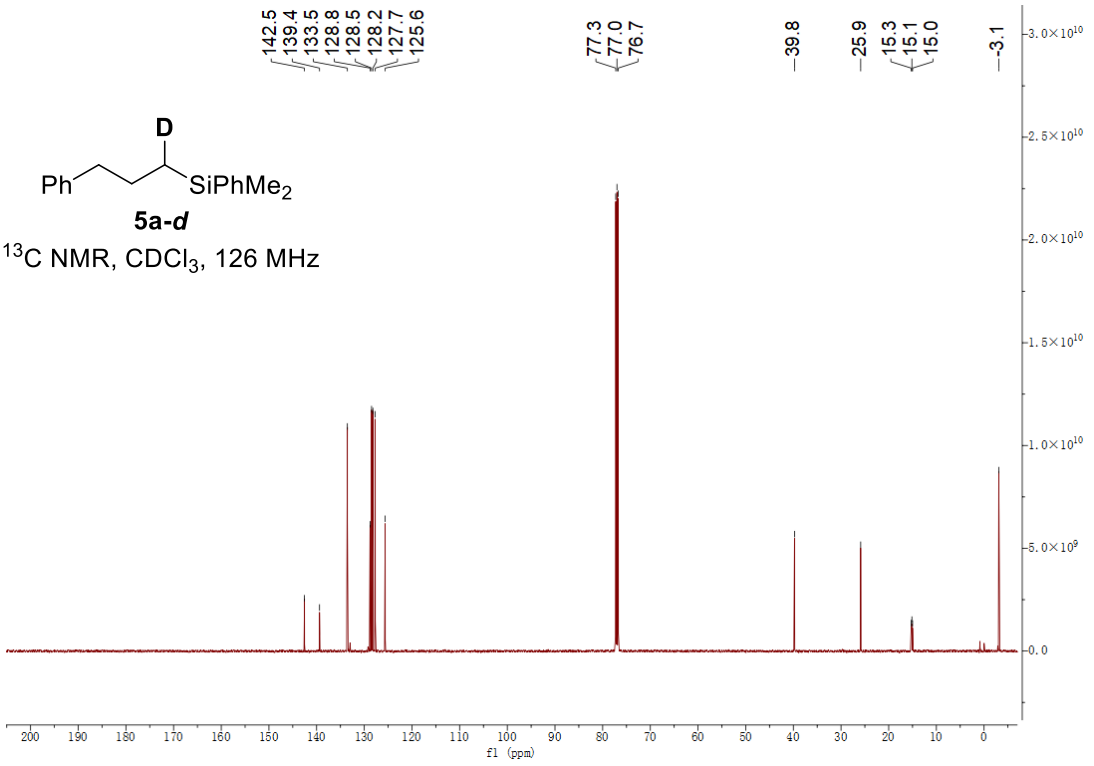
7.24
7.18
7.18
7.17
7.16
7.15
7.15
7.14
7.14
7.13
7.13
7.12
7.12
2.62
2.61
2.59
1.66
1.64
1.63
1.62
1.62
1.61
0.79
0.77
0.77
0.76
0.25
D
Ph
SiPhMe2
5a-d
1H NMR, CDCl3, 500 MHz
1.85
2.89
2.11
2.81
2.02
2.06
1.00
5.89
f1 (ppm)


142.5
139.4
133.5
128.8
128.5
128.2
127.7
125.6
77.3
77.0
76.7
39.8
25.9
15.3
15.1
15.0
3.1
D
Ph
SiPhMe2
5a-d
13C NMR, CDCl3, 126 MHz
f1 (ppm)