Electronic Supplementary Information

Synthesis of Vinyl-Substituted Alcohols Using Acetylene as C2 Building Block

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General Information

^{1}H, ^{13}C, ^{19}F NMR spectra were recorded on a Bruker AVANCE 400 (400 MHz for ^{1}H; 101 MHz for ^{13}C; 376 MHz for ^{19}F) or Bruker AVANCE 500 (500 MHz for ^{1}H; 126 MHz for ^{13}C; 470 MHz for ^{19}F), ^{1}H NMR and ^{13}C NMR chemical shifts were determined relative to internal standard TMS at δ 0.0 and ^{19}F NMR chemical shifts were determined relative to $CFCl_3$ as external standard. Chemical shifts (δ) are reported in ppm, and coupling constants (J) are in Hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Infrared (IR) spectra are recorded on a Nicolet 210 spectrophotometer and were recorded in potassium bromide (KBr) pellet. The following abbreviations were used to explain the intensity and shape of peaks: s = strong, m = middle, w = weak, br = broad. Mass spectra (MS) were obtained using ESI, DART mass spectrometer. Melting points were determined using a hot stage apparatus. All reactions that required heating were proceeded in oil bath. All reagents were used as received from commercial sources, unless specified otherwise, or prepared as described in the literature.

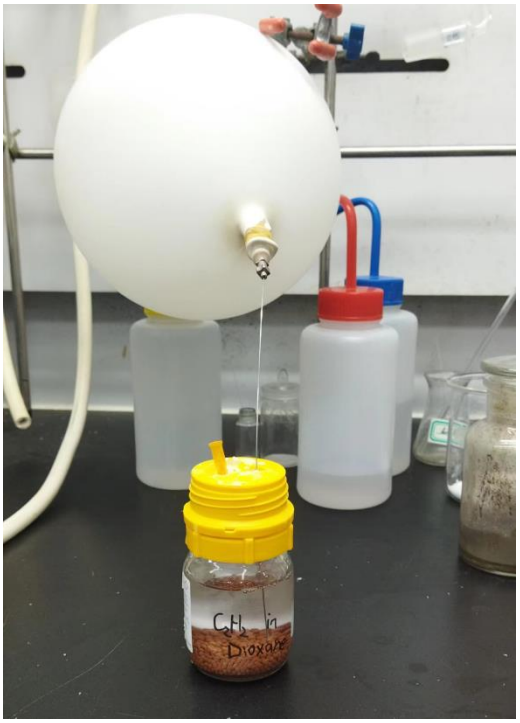
General Procedures

General procedure for the optimization of reaction involving gaseous acetylene (GP1):

1) Nickel catalyst and ligand were weighed out and transferred into a Schlenk tube in the glovebox.
2) The tube was then sealed with a hollow glass plug and taken out of the glovebox.
3) Putting the tube under nitrogen via 3 vacuum / N_2 backfill cycles and unplugged the hollow glass plug.
4) Add 1 mL solvent and base (if needed) and seal the tube with a three-way valve.
5) Stir the solution at room temperature until all the solid dissolved for 2 min.
6) Add 4 equiv silane via syringe after the same operation of step 3.
7) Stir the solution at room temperature for 2 min.
8) Attach the gaseous acetylene balloon to one valve of the three-way valve.
9) Putting the tube under acetylene via 10 vacuum / C_2H_2 backfill cycles with a water pump and sealed with a hollow glass plug (for the volatile solvent, it requires cooling to -20 ℃ in advance)
10) Unplugged the glass plug and add 4-biphenylcarboxaldehyde (0.3 mmol, 54.7 mg) dissolved in 0.5 mL solvent via syringe ASAP.
11) Stir the solution at 35 ℃ for 24 h.
12) After completion of reaction, filtrate the mixture by a short pad of silica gel and wash with EA and was then concentrated under reduced pressure.
13) Add the 4-nitrotoluene as internal for calculation of ^{1}H NMR yield.

General procedure for the preparation of saturated solution of acetylene in THF (GP2):

The procedure was carried out according to literature[1] with minor modification.



1) 80 mL anhydrous THF was filled in a 100mL Schlenk bottle. Vacuumize the system with oil pump

for about 60 seconds. (Note: the solvent will be boiling and should prevent the solvent from flushing into the pump by applying a surge flask.

2) Bubble the solution with acetylene through a long needle about 2 h after consumption of 2 acetylene balloon in size of roughly 10 cm in diameter for the initial preparation.

3) Draw the solution via syringe under protection or bubbling of acetylene while using.

General procedure for the optimization of reaction involving saturated solution of acetylene in THF (GP3):

1) Nickel catalyst and ligand were weighed out and transferred into a Schlenk tube in the glove box.

2) The tube was then sealed with a hollow glass plug and taken out of the glovebox.

3) Putting the tube under nitrogen via 3 vacuum / N_2 backfill cycles and unplugged the hollow glass plug.

4) Add 1 mL solvent and base (if needed) and seal the tube with a three-way valve.

5) Stir the solution at room temperature until all the solid dissolved (2 min).

6) Add 4 equiv silane via syringe after the same operation of step 3.

7) Stir the solution at room temperature for 2 min.

8) Add 4-biphenylcarboxaldehyde (0.2 mmol) dissolved in 800μL (4.4 equiv) saturated solution of acetylene in THF (0.55 mol/L).

9) Stir the solution at 35℃, monitored by TLC.

10) After completion of reaction, filtrate the mixture by a short pad of silica gel and wash with EA and was then concentrated under reduced pressure.

11) Add the 4-nitrotoluene as internal for calculation of ^{1}H NMR yield. Purify the crude via column chromatography if needed.

Note: Due to gas-liquid equilibrium, few acetylene gas desorbed from the liquid phase. As a result, a bit more amount of acetylene saturated solution added to ensure no less than 4 equiv acetylene employed in reaction.

General procedure for the synthesis of vinyl alcohol (GP4):

1) 11.0 mg $Ni(cod)_2$ (0.04 mmol, 20 mol%), 34.0 mg IPr·HCl (0.08 mmol, 40 mol%) were weighed out and transferred into a Schlenk tube in the glove box.

2) The tube was then sealed with a hollow glass plug and taken out of the glovebox.

3) Putting the tube under nitrogen via 3 vacuum / N_2 backfill cycles and unplugged the hollow glass plug.

4) Add 1.0 mL solvent and KO^{t}Bu 88μL (0.088 mmol, 44 mol%,1 mol/L in THF).

5) Stir the solution at room temperature until all the solid dissolved (2 min).

6) Add 93mg (128μL, ρ= 0.728 g/mL at 25℃) triethylsilane (0.8 mmol, 4 equiv) via syringe after the same operation of step 3.

7) Stir the solution at room temperature for 2 min.

8) Add aldehyde (0.2 mmol) dissolved in 800μL saturated solution of acetylene in THF (0.55 mol/L). if the aldehyde was sparingly soluble, add aldehyde directly into solution followed by rapid addition of saturated solution of acetylene.

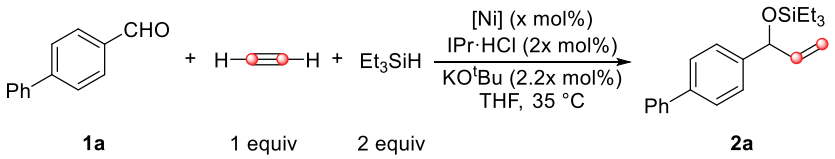
9) Stir the solution at 35 ℃, monitored by TLC.

10) After consumption of aldehyde, filtrate the mixture by a short pad of silica gel and wash with EA and was then concentrated under reduced pressure.

11) Purify the crude via column chromatography.

Optimization of Reaction Condition

Table S1. Optimization of Ni precatalysts[a]



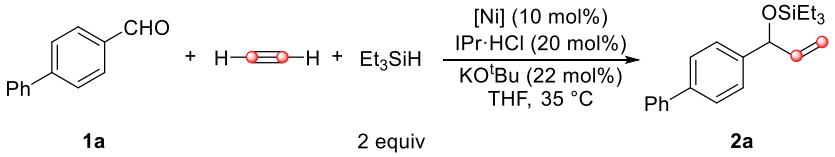
1a 1 equiv 2 equiv **2a**

Entry	[Ni] cat.	Ligand	Yield[b]
1	$Ni(acac)_2$ (20 mol%) + Zn (40 mol%.)	IPr·HCl (40 mol%)	N.D
2	Ni(cod)DQ (10 mol%)	IPr·HCl (20 mol%)	7%
3	$Ni^F(stb)_3$ (10 mol%)	IPr·HCl (20 mol%)	trace
4	**$Ni(cod)_2$ (10 mol%)**	**IPr·HCl (20 mol%)**	**28%**
5[b]	Ni(cod)DQ (20 mol%)	IPr·HCl (40 mol%)	26%

[a] all the solvents were anhydrous. acetylene saturated solution in THF was prepared according to GP2, the reaction was carried out by using GP3. Yield was determined by ^{1}H NMR spectroscopy with 4-nitrotoluene as internal standard. $Ni(acac)_2$: nickel(II) acetylacetonate. $Ni(cod)_2$: bis(1,5-cyclooctadiene)nickel(0). Ni(cod)DQ: (cycloocta-1,5-diene)(duroquinone)nickel (II). $Ni^F(stb)_3$: tris(trans-1,2-bis(4-(trifluoromethyl)phenyl)ethene)nickel(0) [b] the reaction was conducted in comparison to the optimized conditions shown in the article.

Conclusion : Ni^0 in situ generated from reduction of Ni^{II} by Zn dust seem incompatible in this reaction. $Ni(cod)_2$ was the best catalyst. Entry 5 was compared with standard condition.

Table S2. Comparison of saturated solution of acetylene in THF and acetylene balloon[a]



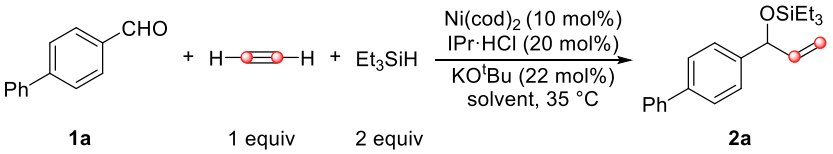
1a 2 equiv **2a**

Entry	HC≡CH	Yield[b]
1	**Saturated solution in THF**	**28%**
2	balloon	6%

[a] all the solvents were anhydrous. acetylene saturated solution in THF was prepared according to GP2, the reaction was carried out by using GP3. Yield was determined by ^{1}H NMR spectroscopy with 4-nitrotoluene as internal standard.

Conclusion: saturated solution of acetylene in THF was given a better result than gaseous acetylene balloon.

Table S3. Optimization of solvents[a]



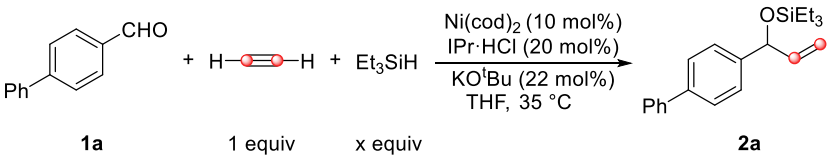
1a 1 equiv 2 equiv **2a**

Entry	solvent	Yield[b]
1	**THF**	**28%**
2	DCE	10%
3	DMF	28%
4	CH_3CN	7%
5	Et_3N	23%
6	Toluene	24%
7	^{i}PrOH	N.D
8[b]	DCE	21%
9[b]	Toluene	32%

[a] all the solvents were anhydrous. acetylene saturated solution in THF was prepared according to GP2, the reaction was carried out by using GP3. Yield was determined by ^{1}H NMR spectroscopy with 4-nitrotoluene as internal standard. [b] the reaction was conducted in the optimized conditions with the replacement of [Ni] cat.

Conclusion: THF was the best solvent. Entry 8,9 were compared with standard condition.

Table S4. Optimization of the loading of Et_3SiH[a]



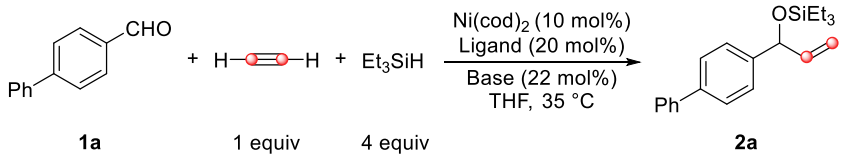
1a 1 equiv x equiv **2a**

Entry	Et_3SiH (x eq.)	Yield
1	1	16%
2	2	28%
3	3	30%
4	**4**	**34%**
5	8	26%
6[b]	2	49%

[a] all the solvents were anhydrous. acetylene saturated solution in THF was prepared according to GP2, the reaction was carried out by using GP3. Yield was determined by ^{1}H NMR spectroscopy with 4-nitrotoluene as internal standard. [b] the reaction was conducted in the optimized conditions with the replacement of the loading of Et_3SiH.

Conclusion: 4 equivalent Et_3SiH was the best. Entry 6 was compared with standard condition.

Table S5. Optimization of the ligand[a]

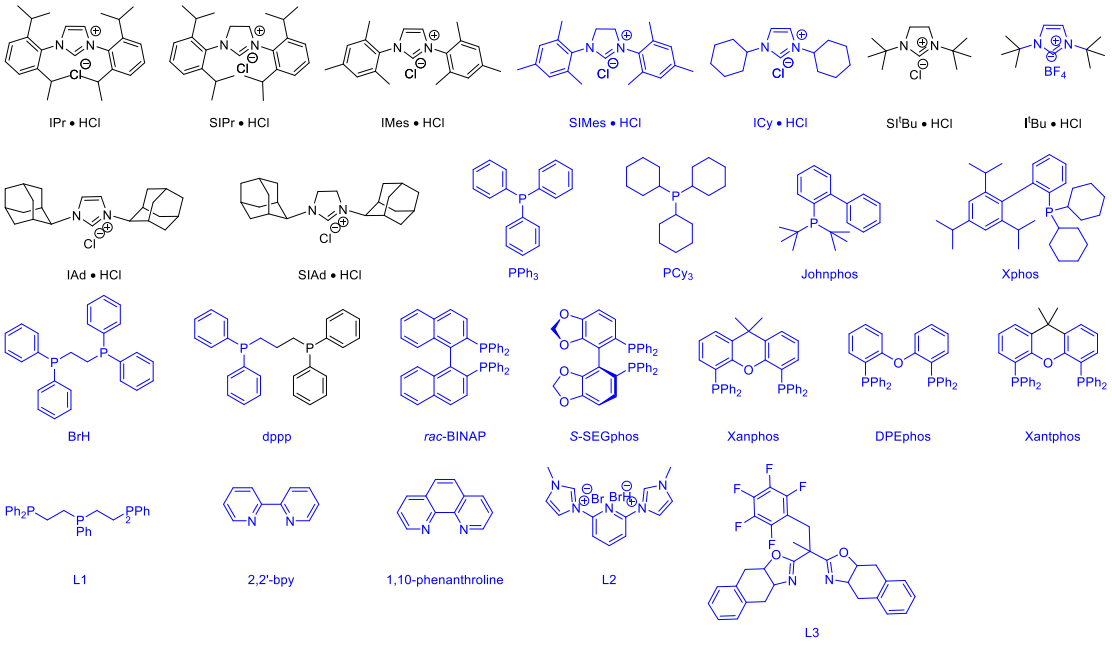


1a 1 equiv 4 equiv **2a**

Entry	Ligand (x mol%)	Base (1.1x mol%)	Yield of 2a
1	**IPr·HCl (20 mol%)**	**KO^{t}Bu (22 mol%)**	**34%**
2	SIPr·HCl (20 mol%)	KO^{t}Bu (22 mol%)	27%
3	IMes·HCl (20 mol%)	KO^{t}Bu (22 mol%)	7%
4	SIMes·HCl (20 mol%)	KO^{t}Bu (22 mol%)	trace
5	IAd·HBF$_4$ (20 mol%)[c]	KO^{t}Bu (22 mol%)	30%
6	SIAd·HBF$_4$ (20 mol%)	KO^{t}Bu (22 mol%)	9%
7	I^{t}Bu·HBF$_4$ (20 mol%)[c]	KO^{t}Bu (22 mol%)	25%
8	SI^{t}Bu·HCl (20 mol%)	KO^{t}Bu (22 mol%)	12%
9	ICy·HCl (20 mol%)	KO^{t}Bu (22 mol%)	N.D
10	PPh$_3$ (20 mol%)	/	6%
11	PCy$_3$ (20 mol%)	/	19%
12	Johnphos (20 mol%)	/	trace
13	Xphos (20 mol%)	/	6%
14	dppe (20 mol%)	/	N.D
15	dppp (20 mol%)	/	N.D
16	*rac*-BINAP (20 mol%)	/	N.D
17	*S*-SEGphos (20 mol%)	/	N.D
18	Xantphos (20 mol%)	/	N.D
19	DPEphos (20 mol%)	/	N.D
20	L1 (20 mol%)	/	N.D
21	2,2'-bpy (20 mol%)	/	N.D
22	1,10-phenanthroline (20 mol%)	/	N.D
23	L2 (20 mol%)	KO^{t}Bu (40 mol%)	N.D
24	L3 (20 mol%)	/	N.D
25[b]	IMes·HCl (40 mol%)	KO^{t}Bu (44 mol%)	17%
26[b]	IAd·HBF$_4$ (40 mol%)	KO^{t}Bu (44 mol%)	40%

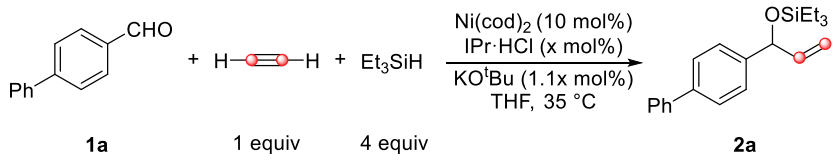
[a] all the solvents were anhydrous. acetylene saturated solution in THF was prepared according to GP2, the reaction was carried out by using GP3. Yield was determined by ^{1}H NMR spectroscopy with 4-nitrotoluene as internal standard. [b] the reaction was conducted in the optimized conditions with the replacement of ligand.

Conclusion: IPr·HCl was the best ligand. Entry 26,27 were compared with standard condition.



Scheme S1. Ligands which were trialed for the optimization studies.

Table S6. Optimization of the ratio of ligand and catalyst[a]



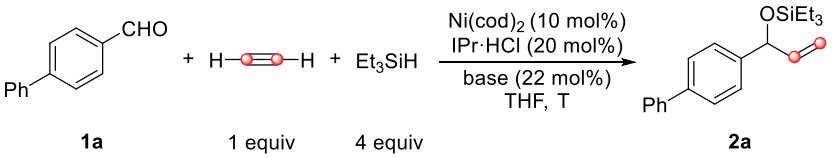
1a 1 equiv 4 equiv **2a**

Entry	Ni(cod)$_2$ (10 mol%)	Ligand (x mol%)	Yield
1	Ni(cod)$_2$ (10 mol%)	IPr·HCl (10 mol%)	17%
2	**Ni(cod)$_2$ (10 mol%)**	**IPr·HCl (20 mol%)**	**34%**
3	Ni(cod)$_2$ (10 mol%)	IPr·HCl (30 mol%)	21%
4	Ni(cod)$_2$ (10 mol%)	IPr·HCl (40 mol%)	25%
5	Ni(cod)$_2$ (10 mol%)	IPr·HCl (50 mol%)	31%

[a] all the solvents were anhydrous. acetylene saturated solution in THF was prepared according to GP2, the reaction was carried out by using GP3. Yield was determined by ^{1}H NMR spectroscopy with 4-nitrotoluene as internal standard.

Conclusion: 2 equivalents of IPr·HCl compared to Ni(cod)$_2$ was the most reasonable.

Table S7. Optimization of the base[a]



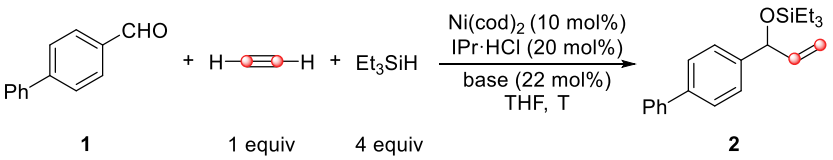
1a 1 equiv 4 equiv **2a**

Entry	base	Yield
1	**KO^{t}Bu (20 mol%)**	**34%**
2	NaO^{t}Bu (20 mol%)	11%
3	LiO^{t}Bu (20 mol%)	5%
4	^{n}BuLi (20 mol%)	7%
5[b]	NaO^{t}Bu (44 mol%)	39%

[b] all the solvents were anhydrous. acetylene saturated solution in THF was prepared according to GP2, the reaction was carried out by using GP3. Yield was determined by ^{1}H NMR spectroscopy with 4-nitrotoluene as internal standard. [b] the reaction was conducted in the optimized conditions with the replacement of the base.

Conclusion: KO^{t}Bu was the best base. Entry 5 was compared with standard condition.

Table S8. Optimization of the temperature[a]



1 1 equiv 4 equiv **2**

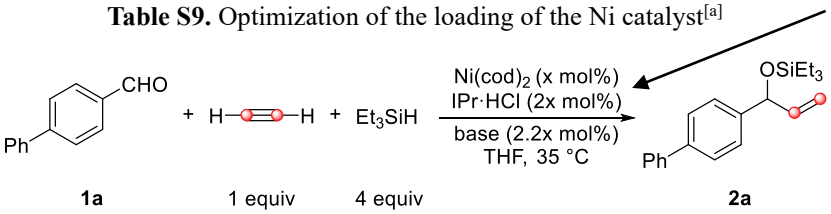
Entry	T (°C)	Yield
1	25	13%
2	**35**	**34%**
3	45	26%
4[b]	25	56%

[c] all the solvents were anhydrous. acetylene saturated solution in THF was prepared according to GP2, the reaction was carried out by using GP3. Yield was determined by ^{1}H NMR spectroscopy with 4-nitrotoluene as internal standard. [b] the reaction was conducted in the optimized conditions with the replacement of the base.

Conclusion: 35 °C was the most suitable reaction tempertature. Entry 5 was compared with standard condition. Either temperature higher than 45 °C or lower than 25 °C lead to less than trace amount of desired products.

The ligand/nickel ratio is 2:1, which we had wrote it 1:1 by mistake. And the base should be 1.1 equivalent to ligand.

Table S9. Optimization of the loading of the Ni catalyst[a]



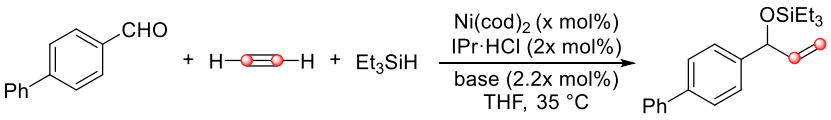
1a 1 equiv 4 equiv **2a**

Entry	Loading of $Ni(cod)_2$	Yield
1	5	15%
2	10	34%
3	15	44%
4	**20**	**53%**
5	25	56%

[d] all the solvents were anhydrous. acetylene saturated solution in THF was prepared according to GP2, the reaction was carried out by using GP3. Yield was determined by ^1H NMR spectroscopy with 4-nitrotoluene as internal standard. [b] the reaction was conducted in the optimized conditions with the replacement of the base.

Conclusion: while the loading of $Ni(cod)_2$ was 20% seemed to be the most reasonable choice.

Table S10. Optimization of the loading of the acetylene[a]



1a x equiv 4 equiv **2a**

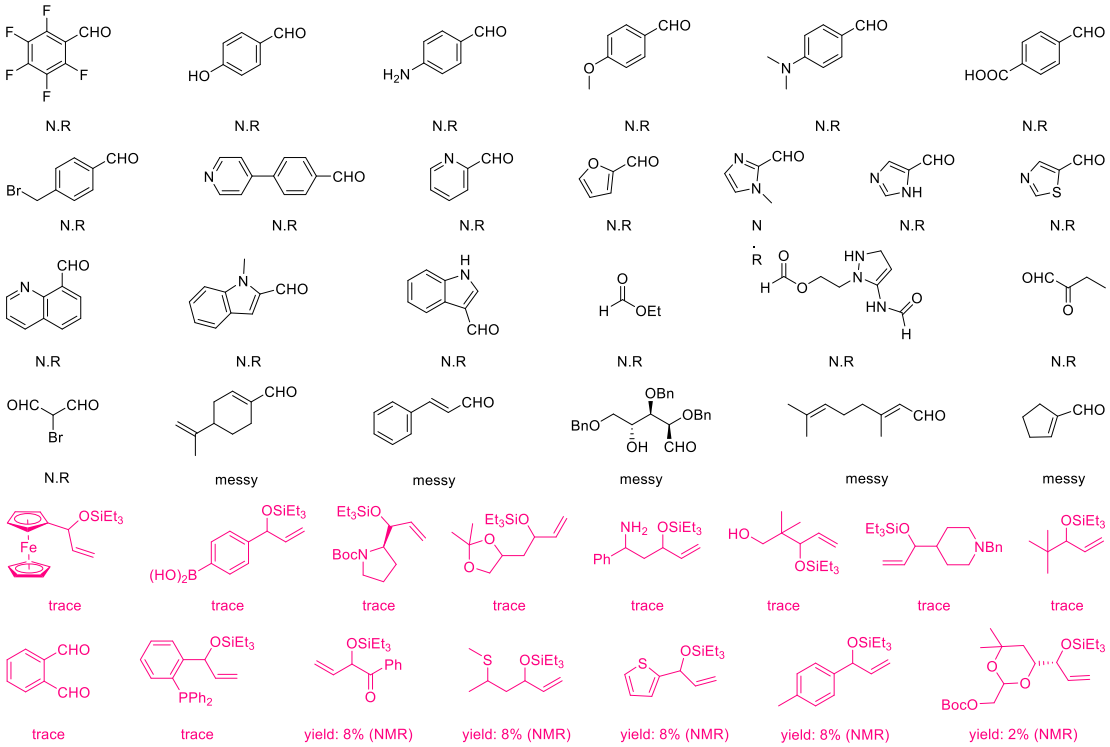
Entry	Loading of HC≡CH	Yield
1	1 equivalent	53%
2	**2 equivalent**	**74%**
3	3 equivalent	72%
4	4 equivalent	70%
5	Gaseous acetylene balloon	18%

[e] all the solvents were anhydrous. acetylene saturated solution in THF was prepared according to GP2, the reaction was carried out by using GP3. Yield was determined by ^1H NMR spectroscopy with 4-nitrotoluene as internal standard. [b] the reaction was conducted in the optimized conditions with the replacement of the base.

Conclusion: while the loading of acetylene was 2 equivalent compared to aldehyde seemed to be the most reasonable choice.

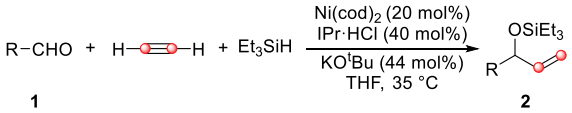
All the Incompatible Substrates

All the substrates that were tested but gave a poor yield or a messy result were listed below.

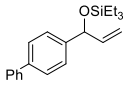


Scheme S2. Collection of failed substrates.

Sythesis of Vinyl Alcohol with Acetylene



2a ((1-([1,1'-biphenyl]-4-yl)allyl)oxy)triethylsilane



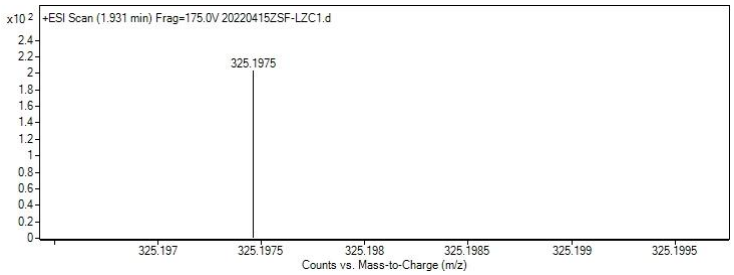
Followed GP4 with 54.7 mg (0.3 mmol) of **1a**, 72.5 mg **2a** was obtained as colorless liquid, R_f = 0.6 (PE), yield= 75%.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 – 7.53 (m, 4H), 7.42 (t, *J* = 7.8 Hz, 4H), 7.35 – 7.29 (m, 1H), 5.97 (ddd, *J* = 16.6, 10.2, 6.1 Hz, 1H), 5.31 (d, *J* = 16.9 Hz, 1H), 5.21 (d, *J* = 6.0 Hz, 1H), 5.10 (d, *J* = 10.2 Hz, 1H), 0.94 (t, *J* = 8.0 Hz, 9H), 0.62 (qd, *J* = 7.8, 2.9 Hz, 6H).

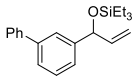
^{13}C NMR (126 MHz, $CDCl_3$) δ 142.9, 141.6, 141.0, 140.0, 128.7, 127.2, 127.1, 127.0, 126.4, 113.7, 75.5, 6.9, 5.0.

IR: 2955(w), 1764(m), 1459(w), 1377(m), 1243(s), 1057(m), 1008(w), 914(w), 813(w), 744(m).

HRMS: Calculation for $C_{21}H_{28}OSi$, $[M+H^+]^+$: 325.1982, Found: 325.1975.



2b ((1-([1,1'-biphenyl]-3-yl)allyl)oxy)triethylsilane



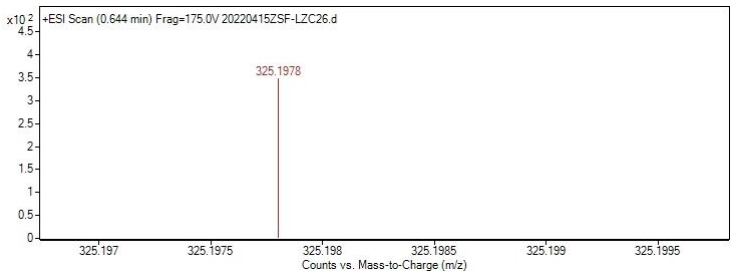
Followed GP4 with 36.4 mg (0.2 mmol) of **1b**, 55.6 mg **2b** was obtained as colorless liquid, R_f = 0.6 (PE), yield= 86%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 -7.63 (m, 3H), 7.54 – 7.37 (m, 6H), 6.10 – 5.96 (m, 1H), 5.36 (ddd, *J* = 17.0, 1.6 Hz, 1H), 5.27 (d, *J* = 6.0 Hz, 1H), 5.14 (dt, *J* = 10.2, 1.5 Hz, 1H), 0.99 (t, *J* = 7.9 Hz, 9H), 0.67 (qd, *J* = 7.8, 2.1 Hz, 6H).

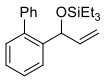
^{13}C NMR (101 MHz, $CDCl_3$) δ 144.3, 141.6, 141.4, 141.1, 128.7, 128.7, 127.2, 127.2, 126.0, 125.0, 124.9, 113.8, 75.8, 6.9, 5.0.

IR: 3463(s), 2956(m), 1763(w), 1637(s), 1242(m), 1070(w), 916(w), 738(m).

HRMS: Calculation for $C_{21}H_{28}OSi$, $[M+H^+]^+$: 325.1982, Found: 325.1978.



2c ((1-([1,1'-biphenyl]-2-yl)allyl)oxy)triethylsilane



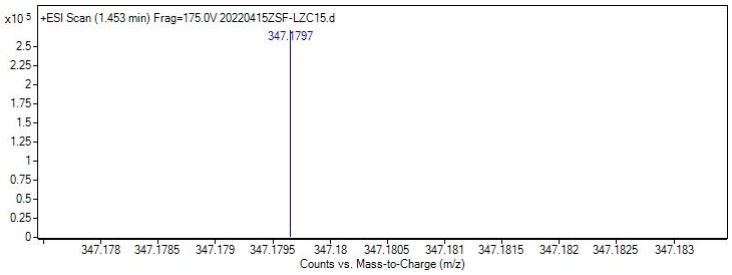
Followed GP4 with 36.4 mg (0.2 mmol) of **1c**, 56.3 mg **2c** was obtained as colorless liquid, R_f = 0.6 (PE), yield= 87%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.43 – 7.33 (m, 4H), 7.27 (tdd, *J* = 5.0, 3.3, 1.4 Hz, 3H), 7.17 (dd, *J* = 7.6, 1.5 Hz, 1H), 5.93 (ddd, *J* = 17.0, 10.3, 5.3 Hz, 1H), 5.25 (dt, *J* = 5.3, 1.5 Hz, 1H), 5.01 (dt, *J* = 12.3, 1.7 Hz, 1H), 4.97 (dt, *J* = 5.6, 1.7 Hz, 1H), 0.79 (t, *J* = 7.9 Hz, 9H), 0.39 (qd, *J* = 8.3, 1.2 Hz 6H).

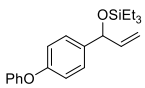
^{13}C NMR (101 MHz, $CDCl_3$) δ 141.5, 141.3, 141.1, 140.0, 129.4, 128.0, 128.0, 127.7, 127.2, 127.1, 126.8, 113.4, 71.5, 6.7, 4.8.

IR: 3455(w), 2955(w), 2880(w),1764(w), 1633(w), 1469(w), 1377(w), 1242(m), 1058(w),1009(w), 916(w), 845(w), 743(m), 701(m), 508(w).

HRMS: Calculation for $C_{21}H_{28}OSi$, $[M+Na^+]^+$: 347.1801, Found: 347.1797.



2d ((1-(4-phenoxyphenyl)allyl)oxy)triethylsilane



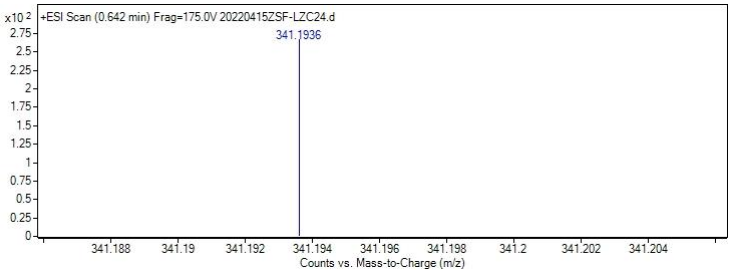
Followed GP4 with 39.6 mg (0.2 mmol) of **1d**, 41.3 mg **2d** was obtained as light-yellow liquid, R_f = 0.5 (PE), yield= 61%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.31 (m, 4H), 7.15 – 7.06 (m, 1H), 7.01 (ddd, *J* = 13.2, 7.6, 1.6 Hz, 4H), 5.98 (ddd, *J* = 16.6, 10.2, 5.9 Hz, 1H), 5.31 (dt, *J* = 17.0, 1.6 Hz, 1H), 5.18 (d, *J* = 5.9 Hz, 1H), 5.12 (dt, *J* = 10.2, 1.5 Hz, 1H), 0.96 (t, *J* = 7.9 Hz, 9H), 0.64 (qd, *J* = 7.9, 2.0 Hz, 6H).

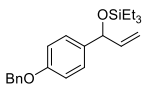
^{13}C NMR (101 MHz, $CDCl_3$) δ 157.4, 156.2, 141.6, 138.8, 129.7, 127.5, 123.1, 118.7, 118.7, 113.6, 75.3, 6.8, 4.9.

IR: 3513(m), 2956(m), 1765(m), 1648(w), 1591(w), 1490(w), 1377(w), 1241(s), 1057(m), 926(w), 846 (w), 744(w), 692(w).

HRMS: Calculation for $C_{21}H_{28}O_2Si$, $[M+H^+]^+$: 341.1932, Found: 341.1936.



2e ((1-(4-(benzyloxy)phenyl)allyl)oxy)triethylsilane



Followed GP4 with 42.4 mg (0.2 mmol) of **1e**, 25.8 mg **2e** was obtained as light-yellow liquid, R_f = 0.5 (PE), yield= 36%.

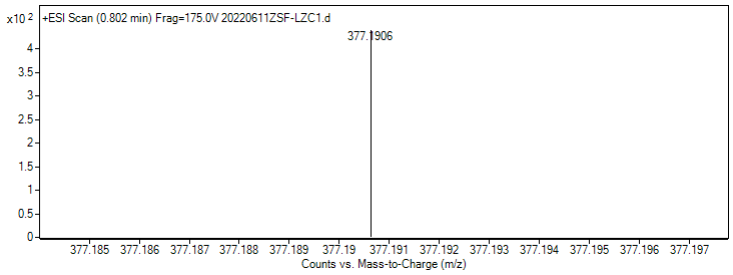
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.35 (m, 5H), 7.29 (d, *J* = 5.8 Hz, 2H), 6.99 – 6.94 (m, 2H), 5.96 (ddd, *J* = 16.5, 10.2, 5.9 Hz, 1H), 5.28 (dt, *J* = 17.0, 1.6 Hz, 1H), 5.15 (d, *J* = 6.0 Hz, 1H), 5.11 – 5.07 (m, 3H), 0.95 (t, *J* = 8.0 Hz, 9H), 0.62 (qd, *J* = 7.9, 2.2 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 158.0, 141.8, 137.2, 136.3, 128.6, 127.9, 127.5, 127.3, 114.5, 113.3, 75.3, 70.1, 6.8, 4.9.

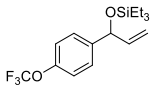
IR: 3407(m), 2954(w), 2878(m), 1614(w), 1508(w), 1460(w), 1237(w), 1171(w), 1080(w), 1015(w),

843(w), 735(w), 611(w).

HRMS: Calculation for $C_{22}H_{30}O_2Si$, $[M+Na^+]^+$: 377.1907, Found: 377.1906.



2f ((1-(4-(trifluoromethoxy)phenyl)allyl)oxy)triethylsilane



Followed GP4 with 38.0 mg (0.2 mmol) of **1f**, 42.4 mg **2f** was obtained as colorless liquid, R_f = 0.6 (PE), yield= 64%.

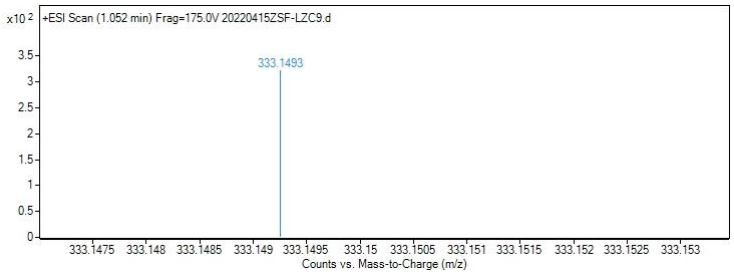
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.7 Hz, 2H), 7.16 (d, *J* = 8.2 Hz, 2H), 5.90 (ddd, *J* = 16.7, 10.2, 6.1 Hz, 1H), 5.28 (dt, *J* = 17.1, 1.5 Hz, 1H), 5.16 (d, *J* = 6.1 Hz, 1H), 5.10 (dt, *J* = 10.2, 1.4 Hz, 1H), 0.92 (t, *J* = 8.0 Hz, 9H), 0.60 (qd, *J* = 7.9, 2.0 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 148.22 (q, J = 2.0 Hz), 142.5, 141.2, 127.3, 123.6, 120.59 (q, J = 256.6 Hz), 120.7, 119.5, 117.7, 114.1, 75.1, 6.7, 4.9.

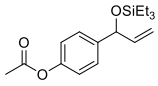
IR: 3449(s), 2957(m), 1743(w), 1640(w), 1457(w), 1380(w), 1243(m), 1084(w), 1047(m), 1009(w), 917(w), 840(w), 776(w), 737(m).

^{19}F NMR (471 MHz, Chloroform-*d*) δ -57.88.

HRMS: Calculation for $C_{16}H_{23}F_3O_2Si$, $[M+H^+]^+$: 333.1492, Found: 333.1493.



2g 4-(1-((triethylsilyl)oxy)allyl)phenyl acetate



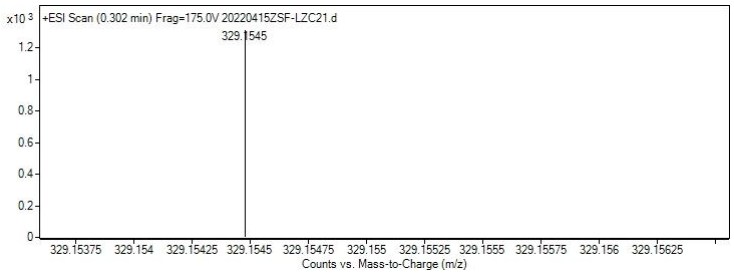
Followed GP4 with 32.8 mg (0.2 mmol) of **1g**, 41.3 mg **2g** was obtained as light-yellow liquid, R_f = 0.4 (PE:EA= 20:1), yield= 67%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.31 (m, 2H), 7.07 – 6.99 (m, 2H), 5.91 (ddd, *J* = 16.6, 10.2, 6.1 Hz, 1H), 5.27 (dt, *J* = 17.0, 1.6 Hz, 1H), 5.15 (d, *J* = 6.1 Hz, 1H), 5.08 (dt, *J* = 10.2, 1.5 Hz, 1H), 2.28 (s, 3H), 0.92 (t, *J* = 7.9 Hz, 9H), 0.60 (qd, *J* = 7.9, 1.9 Hz, 6H).

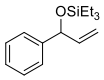
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.5, 149.6, 141.4, 141.3, 127.0, 121.2, 113.8, 75.2, 21.2, 6.8, 4.9.

IR: 3483(w), 2958(w), 2883(w), 1764(w), 1629(w), 1463(w), 1378(w), 1345(w), 1244(m), 1171(w), 1132(m), 1052(w), 915(w), 844(w), 742(m).

HRMS: Calculation for $C_{17}H_{26}O_3Si$, $[M+Na^+]^+$: 329.1543, Found: 329.1545.



2h ((1-phenylallyl)oxy)triethylsilane



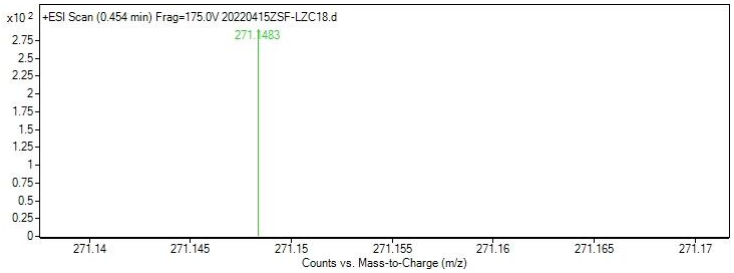
Followed GP4 with 21 μL (21.2mg, 0.2 mmol) of **1h**, 34.5 mg **2h** was obtained as colorless liquid, R_f = 0.6 (PE), yield= 69%.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.27 (m, 4H), 7.25 – 7.20 (m, 1H), 5.94 (ddd, *J* = 16.9, 10.2, 5.9 Hz, 1H), 5.27 (dt, *J* = 17.1, 1.6 Hz, 1H), 5.19 – 5.12 (m, 1H), 5.07 (dt, *J* = 10.2, 1.5 Hz, 1H), 0.92 (t, *J* = 8.0 Hz, 9H), 0.60 (qd, *J* = 7.9, 3.6 Hz, 6H).

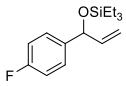
^{13}C NMR (126 MHz, $CDCl_3$) δ 143.8, 141.7, 128.2, 127.1, 126.0, 113.6, 75.7, 6.8, 4.9.

IR: 3452(s), 1762(w), 1636(m), 1379(w), 1243(w), 1055(w), 914(w), 712(w).

HRMS: Calculation for $C_{15}H_{24}OSiv$ $[M+Na^+]^+$: 271.1488, Found: 271.1483.



2i ((1-(4-fluorophenyl)allyl)oxy)triethylsilane



Followed GP4 with 24.8 mg (0.2 mmol) of **1i**, 29.8 mg **2i** was obtained as colorless liquid, R_f = 0.6 (PE), yield= 56%.

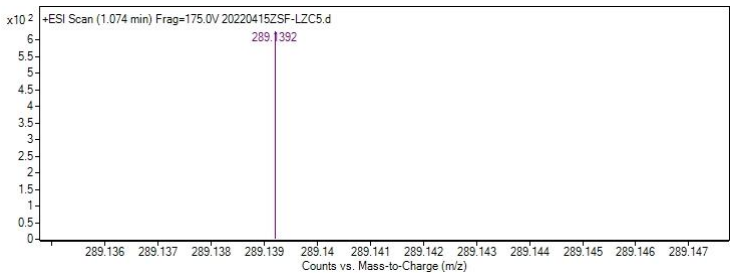
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (dd, *J* = 8.6, 5.6 Hz, 2H), 7.02 (t, *J* = 8.7 Hz, 2H), 5.93 (ddd, *J* = 17.0, 10.2, 5.9 Hz, 1H), 5.29 (dt, *J* = 17.0, 1.5 Hz, 1H), 5.17 (d, *J* = 5.9 Hz, 1H), 5.10 (dt, *J* = 10.2, 1.5 Hz, 1H), 0.95 (t, *J* = 8.0 Hz, 9H), 0.62 (qd, *J* = 7.9, 2.2 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 161.97 (d, *J* = 244.6 Hz), 141.5, 139.56 (d, *J* = 3.1 Hz), 127.61 (d, *J* = 7.9 Hz), 114.96 (d, *J* = 21.3 Hz), 113.7, 75.1, 6.8, 4.9.

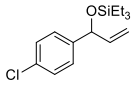
^{19}F NMR (471 MHz, $CDCl_3$) δ -116.04.

IR: 3441(m), 2960(m), 2881(m), 2228(w), 1738(s), 1456(w), 1375(w), 1245(s), 1083(m), 1047(s), 919(w), 808(w), 734(w), 641(s), 558(w).

HRMS: Calculation for $C_{15}H_{23}OFSi$, $[M+Na^+]^+$: 289.1394, Found: 289.1392.



2j ((1-(4-chlorophenyl)allyl)oxy)triethylsilane



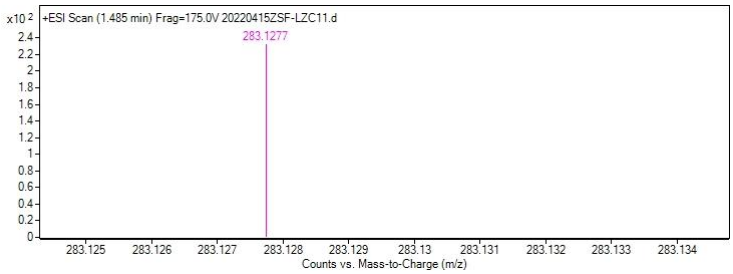
Followed GP4 with 29.1 mg (0.2 mmol) of **1j**, 40.0 mg **2j** was obtained as colorless liquid, R_f = 0.6 (PE), yield= 71%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (s, 4H), 5.92 (ddd, *J* = 17.0, 10.2, 6.0 Hz, 1H), 5.29 (dt, *J* = 17.0, 1.5 Hz, 1H), 5.15 (dt, *J* = 6.0, 1.4 Hz, 1H), 5.11 (dt, *J* = 10.1, 1.4 Hz, 1H), 0.95 (t, *J* = 7.9 Hz, 9H), 0.62 (qd, *J* = 7.9, 2.1 Hz, 6H).

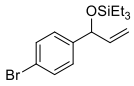
^{13}C NMR (101 MHz, $CDCl_3$) δ 142.4, 141.3, 132.7, 128.3, 127.4, 114.0, 6.8, 4.9.

IR: 3449(s), 2956(w), 1763(w), 1635(w), 1379(w), 1243(m), 1051(w), 917(w), 880(w), 736(m).

HRMS: Calculation for $C_{15}H_{23}ClOSi$, $[M+H^+]^+$: 283.1280, Found: 283.1277.



2k ((1-(4-bromophenyl)allyl)oxy)triethylsilane



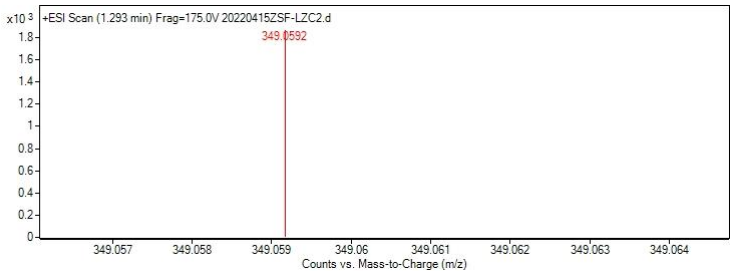
Followed GP4 with 37.0 mg (0.2 mmol) of **1k**, 30.9 mg **2k** was obtained as colorless liquid, R_f = 0.6 (PE), yield= 47%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.43 (m, 2H), 7.27 – 7.20 (m, 2H), 5.91 (ddd, *J* = 17.0, 10.2, 6.0 Hz, 1H), 5.29 (dt, *J* = 17.0, 1.5 Hz, 1H), 5.13 (d, *J* = 6.7 Hz, 1H), 5.11 (dt, *J* = 10.2, 1.4 Hz, 1H), 0.94 (t, *J* = 7.9 Hz, 9H), 0.62 (qd, *J* = 7.9, 2.1 Hz, 6H).

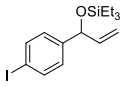
^{13}C NMR (101 MHz, $CDCl_3$) δ 142.9, 141.2, 131.3, 127.8, 120.9, 114.0, 75.1, 6.8, 4.9.

IR: 3426(s), 2956(m), 2880(m), 1733(w), 1641(m), 1480(w), 1408(w), 1077(w), 928(m), 841(w), 730(m).

HRMS: Calculation for $C_{15}H_{23}BrOSi$, $[M+Na^+]^+$: 349.0594, Found: 349.0592.



2l ((1-(4-iodophenyl)allyl)oxy)triethylsilane



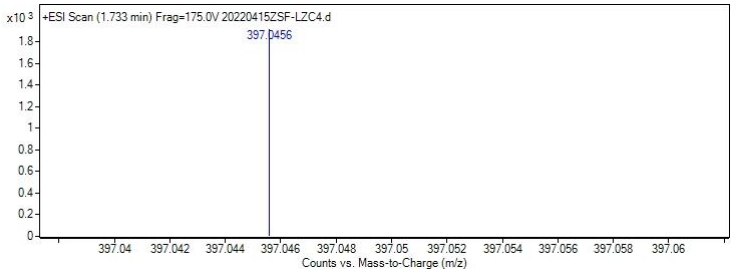
Followed GP4 with 46.4 mg (0.2 mmol) of **1l**, 34.5 mg **2l** was obtained as colorless liquid, R_f = 0.6 (PE), yield= 46%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.4 Hz, 2H), 7.12 (d, *J* = 8.3 Hz, 2H), 5.91 (ddd, *J* = 16.6, 10.2, 6.0 Hz, 1H), 5.29 (d, *J* = 17.0 Hz, 1H), 5.15 – 5.06 (m, 2H), 0.95 (t, *J* = 7.9 Hz, 9H), 0.62 (qd, *J* = 7.9, 2.1 Hz, 6H).

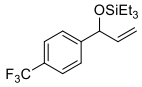
^{13}C NMR (126 MHz, $CDCl_3$) δ 143.6, 141.2, 137.3, 128.1, 114.0, 92.5, 75.2, 6.8, 4.9.

IR: 3774(m), 3664(s), 2953(s), 2878(s), 1595(w), 1473(w), 1404(w), 1237(w), 1074(m), 1004(m), 924(w), 840(m), 799(m), 733(s), 606(w), 509(w).

HRMS: Calculation for $C_{15}H_{23}OISi$, $[M+Na^+]^+$: 397.0455, Found: 397.0456.



2m ((1-(4-(trifluoromethyl)phenyl)allyl)oxy)triethylsilane



Followed GP4 with 34.8 mg (0.2 mmol) of **1m**, 39.5 mg **2m** was obtained as light-yellow liquid, R_f = 0.6 (PE), yield=62%.

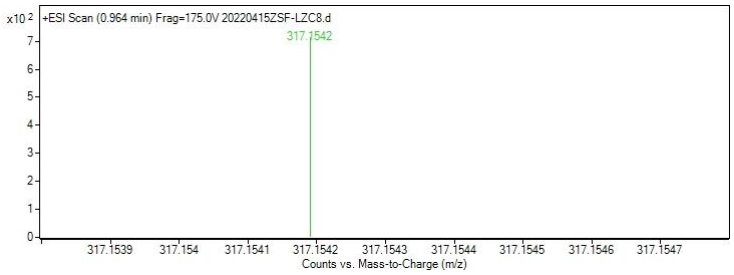
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.1 Hz, 2H), 7.51 – 7.40 (m, 2H), 5.90 (ddd, *J* = 17.0, 10.2, 6.1 Hz, 1H), 5.30 (dt, *J* = 17.0, 1.5 Hz, 1H), 5.21 (d, *J* = 6.1 Hz, 1H), 5.11 (dt, *J* = 10.2, 1.4 Hz, 1H), 0.93 (t, *J* = 7.9 Hz, 9H), 0.61 (qd, *J* = 8.4, 8.0, 2.2 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ (126 MHz, CDCl3) δ 147.8, 140.9, 129.31 (q, *J* = 32.2 Hz), 125.18 (q, *J* = 3.8 Hz), 124.56 (q, *J* = 271.4 Hz), 121.0, 114.4, 75.3, 6.7, 4.9.

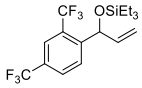
^{19}F NMR (471 MHz, $CDCl_3$) δ -62.37.

IR: 3427(s), 2976(w), 1736(w), 1644(w), 1380(w), 1261(m), 1167(w), 1084(w), 1047(m), 917(w), 737(w).

HRMS: Calculation for $C_{16}H_{23}F_4OSi$, $[M+H^+]^+$: 317.1543, Found: 317.1542.



2n ((1-(2,4-bis(trifluoromethyl)phenyl)allyl)oxy)triethylsilane



Followed GP4 with 48.4 mg (0.2 mmol) of **1n**, 42.2 mg **2n** was obtained as yellow liquid, R_f = 0.5 (PE), yield= 55%.

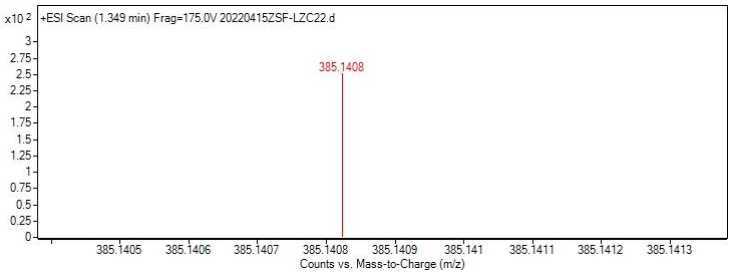
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.3 Hz, 1H), 7.90 – 7.76 (m, 2H), 5.92 (ddd, *J* = 16.9, 10.3, 4.7 Hz, 1H), 5.64 (dd, *J* = 4.9, 2.0 Hz, 1H), 5.38 (d, J = 17.0 Hz, 1H), 5.12 (dt, *J* = 10.3, 1.6 Hz, 1H), 0.92 (t, *J* = 7.9 Hz, 9H), 0.61 (qd, *J* = 7.9, 5.0 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 147.5, 139.9, 129.7, 129.67 (q, J = 33.5 Hz), 128.85 (d, J = 3.7 Hz), 126.78 (q, J = 31.6 Hz), 124.5, 123.49 (q, J = 274.0 Hz), 122.40 (q, J = 1.7 Hz), 114.1, 70.0, 6.5, 4.6.

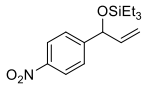
^{19}F NMR (376 MHz, $CDCl_3$) δ -58.54, -62.77.

IR: 3453(s), 2956(m), 2880(m), 1723(m), 1637(m), 1428(w), 1277(m), 1187(m), 1106(m), 1011(w), 920(w), 840(w), 739(m).

HRMS: Calculation for $C_{17}H_{22}F_6OSi$, $[M+H^+]^+$: 385.1417, Found: 385.1408.



2o triethyl((1-(4-nitrophenyl)allyl)oxy)silane



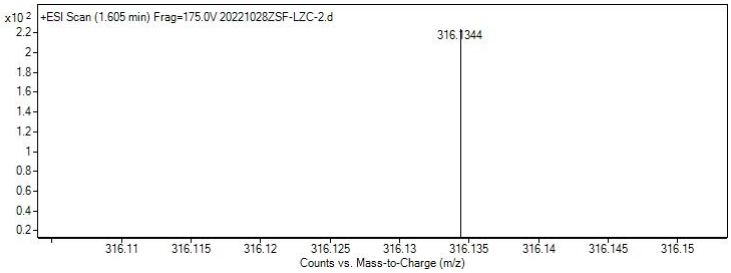
Followed GP4 with 21.2 mg (0.1 mmol) of **1o**, 12.5 mg **2o** was obtained as yellow liquid, R_f = 0.4 (PE:EA= 20:1), yield= 43%.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.12 (d, *J* = 8.8 Hz, 2H), 5.81 (ddd, *J* = 16.7, 10.2, 6.2 Hz, 1H), 5.26 (dt, *J* = 17.1, 1.4 Hz, 1H), 5.17 (d, *J* = 6.2 Hz, 1H), 5.08 (dt, *J* = 10.3, 1.3 Hz, 1H), 0.86 (t, *J* = 7.9 Hz, 9H), 0.55 (qd, *J* = 7.9, 2.9 Hz, 6H).

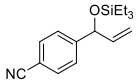
^{13}C NMR (126 MHz, $CDCl_3$) δ 150.2, 146.1, 139.3, 125.7, 122.6, 114.1, 74.1, 5.7, 3.8.

IR: 3452(s), 3162(s), 1646(s), 1403(s), 721(m), 559(m).

HRMS: Calculation for $C_{15}H_{23}NO_3Si$, $[M+Na^+]^+$: 316.1339, Found: 316.1344.



2p 4-(1-((triethylsilyl)oxy)allyl)benzonitrile



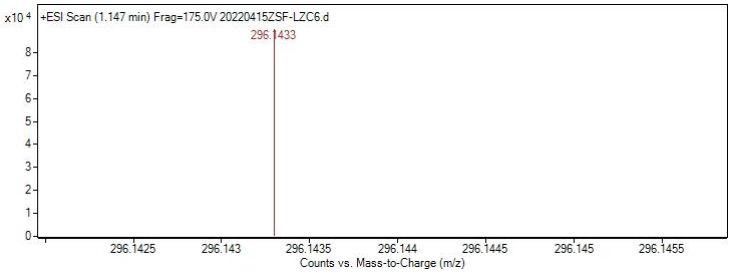
Followed GP4 with 26.2 mg (0.2 mmol) of **1p**, 18.9 mg **2p** was obtained as light-yellow liquid, R_f = 0.6 (PE), yield= 35%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 8.3 Hz, 2H), 7.46 (d, *J* = 8.3 Hz, 2H), 5.86 (ddd, *J* = 16.7, 10.2, 6.2 Hz, 1H), 5.31 (dt, *J* = 17.0, 1.4 Hz, 1H), 5.19 (d, *J* = 6.2 Hz, 1H), 5.13 (dt, *J* = 10.2, 1.3 Hz, 1H), 0.93 (t, *J* = 7.9 Hz, 9H), 0.61 (qd, *J* = 7.9, 2.1 Hz, 6H).

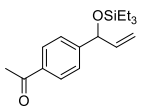
^{13}C NMR (126 MHz, $CDCl_3$) δ 149.2, 140.4, 132.1, 126.6, 119.0, 114.9, 110.9, 75.2, 6.7, 4.8.

IR: 3691(w), 2959(m), 1763(w), 1377(w), 1325(w), 1243(m), 1166(m), 1129(m), 1065(w), 916(w), 849(w), 742(w).

HRMS: Calculation for $C_{16}H_{23}NOSi$, $[M+Na^+]^+$: 296.1441, Found: 296.1433.



2q 1-(4-(1-((triethylsilyl)oxy)allyl)phenyl)ethan-1-one



Followed GP4 with 29.6 mg (0.2 mmol) of **1q**, 45.9 mg **2q** was obtained as yellow

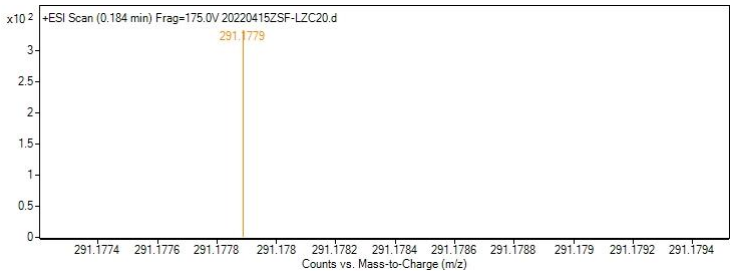
liquid, R_f = 0.4 (PE:EA= 20:1), yield= 79%.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34 (d, J = 8.5 Hz, 2H), 7.03 (d, J = 8.3 Hz, 2H), 5.91 (ddd, J = 16.6, 10.2, 6.1 Hz, 1H), 5.27 (d, J = 17.0 Hz, 1H), 5.15 (d, J = 6.1 Hz, 1H), 5.08 (d, J = 10.1 Hz, 1H), 2.28 (s, 3H), 0.92 (t, J = 7.9 Hz, 9H), 0.60 (qd, J = 7.9, 3.0 Hz, 6H).

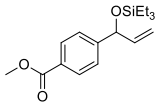
^{13}C NMR (126 MHz, $CDCl_3$) δ 169.5, 149.7, 141.4, 141.3, 127.0, 121.2, 113.8, 75.2, 21.2, 6.8, 4.9.

IR: 3450(w), 2956(w), 1763(m), 1685(w), 1607(w), 1375(w), 1243(s), 1054(m), 915(w), 846(w), 741(m).

HRMS: Calculation for $C_{17}H_{26}O_2Si$, $[M+H^+]^+$: 291.1775, Found: 291.1779.



2r methyl 4-(1-((triethylsilyl)oxy)allyl)benzoate



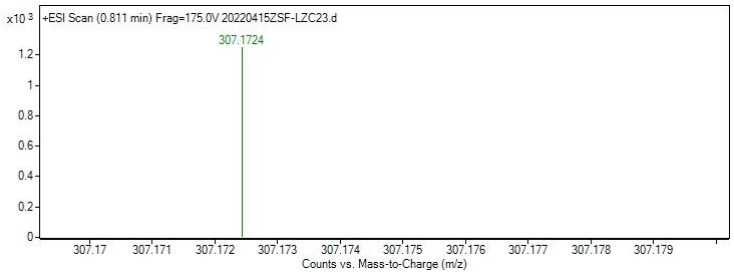
Followed GP4 with 32.8 mg (0.2 mmol) of **1r**, 54.2 mg **2r** was obtained as yellow liquid, R_f = 0.4 (PE:EA= 60:1), yield= 89%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.4 Hz, 2H), 7.44 (d, *J* = 8.3 Hz, 2H), 6.03 – 5.81 (m, 1H), 5.31 (ddd, J = J = 14.2, 1.5, 1.4 Hz, 1H), 5.22 (d, *J* = 6.0 Hz, 1H), 5.12 (dt, *J* = 10.2, 1.4 Hz, 1H), 3.93 (s, 3H), 0.94 (t, *J* = 7.9 Hz, 9H), 0.63 (qd, *J* = 8.0, 2.3 Hz, 6H).

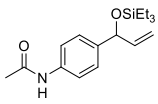
^{13}C NMR(101 MHz, $CDCl_3$) δ 167.1, 149.0, 140.9, 129.6, 129.0, 125.9, 114.3, 75.4, 52.0, 6.8, 4.9.

IR: 3455(s), 1763(w), 1637(m), 1243(w), 743(w).

HRMS: Calculation for $C_{17}H_{26}O_3Si$, $[M+H^+]^+$: 307.1724, Found: 307.1724.



2s N-(4-(1-((triethylsilyl)oxy)allyl)phenyl)acetamide



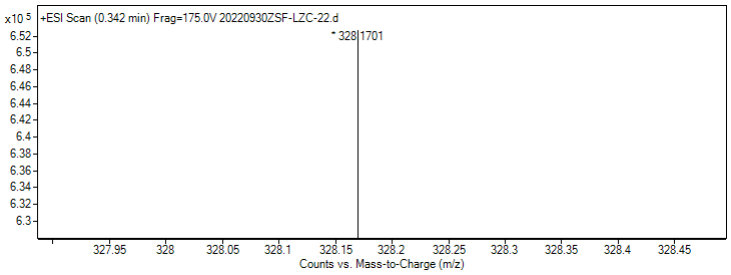
Followed GP4 with 32.6 mg (0.2 mmol) of **1s**, 25.0 mg **2s** was obtained as yellow liquid, R_f = 0.3 (PE:EA= 1:1), yield= 41%.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.1 Hz, 2H), 7.31 – 7.26 (m, 2H), 5.91 (ddd, *J* = 16.6, 10.2, 5.9 Hz, 1H), 5.25 (d, *J* = 17.0 Hz, 1H), 5.12 (d, *J* = 5.9 Hz, 1H), 5.06 (d, *J* = 10.2 Hz, 1H), 2.15 (s, 3H), 0.91 (t, *J* = 8.0 Hz, 9H), 0.59 (qd, *J* = 7.8, 3.2 Hz, 6H).

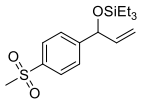
^{13}C NMR (126 MHz, $CDCl_3$) δ 168.4, 141.6, 139.8, 136.8, 126.7, 119.7, 113.5, 75.3, 24.5, 6.8, 4.9.

IR: 3478(s), 3415(s), 3131(m), 2957(m), 2878(m), 1765(m), 1668(m), 1606(m), 1540(m), 1460(w), 1409(w), 1374(w), 1317(w), 1243(s), 1055(m), 1010(w), 916(w), 846(w), 743(s), 607(w), 541(w).

HRMS: Calculation for $C_{17}H_{27}NO_2Si$, $[M+Na^+]^+$: 328.1703, Found: 328.1701.



2t ((1-(4-(methylsulfonyl)phenyl)allyl)oxy)triethylsilane



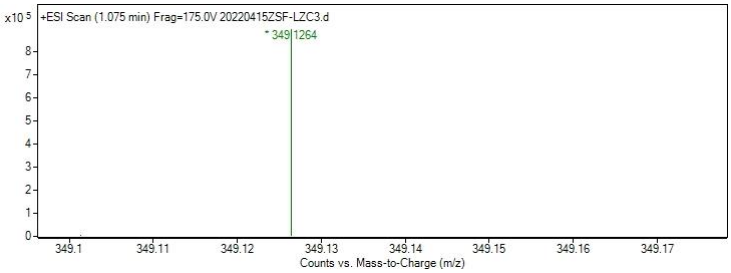
Followed GP4 with 36.8 mg (0.2 mmol) of **1t**, 30.0 mg **2t** was obtained as yellow liquid, R_f = 0.5 (PE:EA= 3:1), yield= 46%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.3 Hz, 2H), 7.57 (d, *J* = 8.1 Hz, 2H), 5.90 (ddd, *J* = 16.7, 10.2, 6.2 Hz, 1H), 5.34 (dt, *J* = 17.0, 1.4 Hz, 1H), 5.25 (d, *J* = 6.2 Hz, 1H), 5.15 (dt, *J* = 10.2, 1.3 Hz, 1H), 3.06 (s, 3H), 0.95 (t, *J* = 7.9 Hz, 9H), 0.64 (qd, *J* = 7.9, 1.7 Hz, 6H).

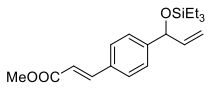
^{13}C NMR(101 MHz, $CDCl_3$) δ 150.2, 140.5, 139.2, 127.4, 126.8, 114.9, 75.2, 44.6, 6.7, 4.8.

IR: 3679(w), 2988(m), 1762(w), 1377(w), 1309(w), 1243(s), 1148(w), 1053(m), 917(w), 842(w), 740(m), 534(w).

HRMS: Calculation for $C_{16}H_{26}O_3SSi$, $[M+Na^+]^+$: 349.1264, Found: 349.1264.



2u (E)-4-(1-((triethylsilyl)oxy)allyl)styryl acetate



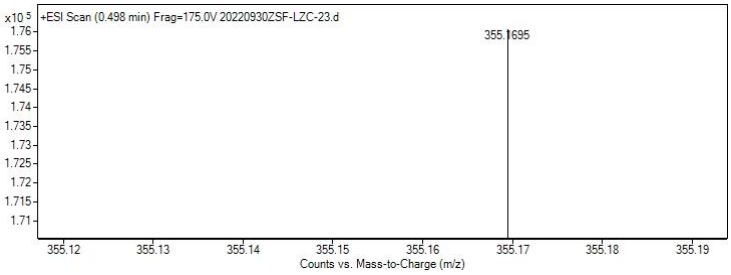
Followed GP4 with 38.0 mg (0.2 mmol) of **1u**, 43.8 mg **2u** was obtained as yellow liquid, R_f = 0.4 (PE:EA= 40:1), yield= 66%.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 (d, *J* = 16.0 Hz, 1H), 7.48 (d, *J* = 7.9 Hz, 2H), 7.37 (d, *J* = 7.9 Hz, 2H), 6.42 (d, *J* = 16.0 Hz, 1H), 5.91 (ddd, *J* = 16.6, 10.0, 6.0 Hz, 1H), 5.29 (dt, *J* = 17.0, 1.5 Hz, 1H), 5.17 (d, *J* = 6.1 Hz, 1H), 5.09 (dt, *J* = 10.1, 1.4 Hz, 1H), 3.80 (s, *J* = 1.3 Hz, 3H), 0.93 (t, *J* = 8.0 Hz, 9H), 0.60 (qd, *J* = 7.9, 3.1 Hz, 6H).

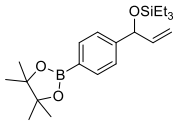
^{13}C NMR (126 MHz, $CDCl_3$) δ 167.5, 146.3, 144.7, 141.1, 133.2, 128.1, 126.5, 117.4, 114.1, 75.4, 51.7, 6.8, 4.9.

IR: 3444(s), 2963(w), 1718(m), 1641(m), 1408(w), 1319(w), 1270(w), 1170(w), 1063(w), 1048(w), 837(w), 735(w), 536(w).

HRMS: Calculation for $C_{19}H_{28}NO_2Si$, $[M+Na^+]^+$: 355.1700, Found: 355.1695.



2v ((1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)allyl)oxy)triethylsilane



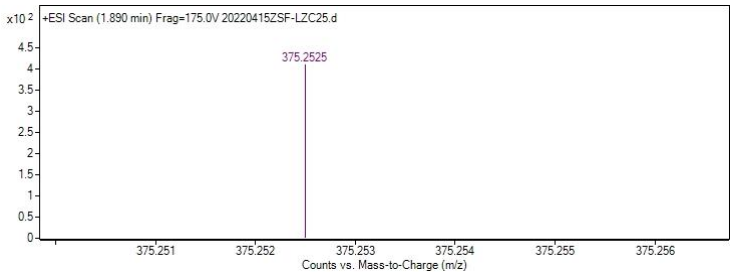
Followed GP4 with 46.4 mg (0.2 mmol) of **1v**, 52.9 mg **2v** was obtained as yellow liquid, R_f = 0.5 (PE:EA= 80:1), yield= 71%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.1 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 5.92 (ddd, *J* = 17.0, 10.2, 5.9 Hz, 1H), 5.26 (dt, *J* = 17.0, 1.5 Hz, 1H), 5.16 (d, *J* = 5.9 Hz, 1H), 5.06 (dt, *J* = 10.2, 1.5 Hz, 1H), 1.33 (s, 12H), 0.91 (t, *J* = 7.9 Hz, 9H), 0.60 (qd, *J* = 7.8, 2.4 Hz, 6H).

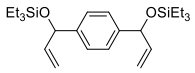
^{13}C NMR (126 MHz, $CDCl_3$) δ 147.0, 141.4, 134.8, 125.4, 113.7, 83.7, 75.7, 24.9, 6.8, 4.9.

IR: 3451(s), 2955(w), 1637(m), 1242(w), 1073(w), 744(m).

HRMS: Calculation for $C_{21}H_{35}BO_3Si$, $[M+H^+]^+$: 375.2522, Found: 375.2525.



2w 1,4-bis(1-((triethylsilyl)oxy)allyl)benzene



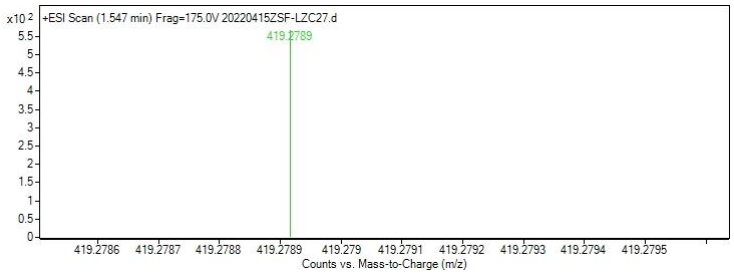
Followed GP4 with 13.4mg (0.1 mmol) of **1w**, 22.3mg **2w** was obtained as colorless liquid, Rf=0.4 (PE), yield= 53%.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (s, 4H), 5.93 (ddd, *J* = 16.6, 10.2, 5.9 Hz, 2H), 5.26 (dt, *J* = 17.1, 1.7 Hz, 2H), 5.14 (d, *J* = 5.9, 2H), 5.06 (dt, *J* = 10.2, 1.6 Hz, 2H), 0.91 (t, *J* = 8.0 Hz, 18H), 0.58 (qd, *J* = 7.8, 3.3 Hz, 12H).

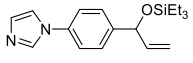
^{13}C NMR (126 MHz, $CDCl_3$) δ 142.6, 141.7, 125.9, 113.5, 75.6, 6.8, 4.9.

IR: 3450(s), 1764(w), 1636(m), 1379(w), 1242(m), 1060(w), 916(w), 740(w).

HRMS: Calculation for $C_{24}H_{42}O_2Si_2$, $[M+H^+]^+$: 419.2796, Found: 419.2789.



2x ((1-cyclohexylallyl)oxy)triethylsilane



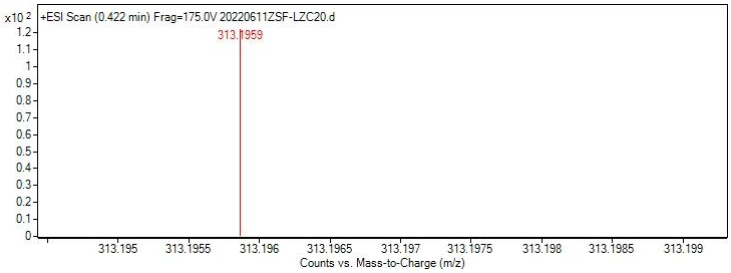
Followed GP4 with 34.4mg (0.2 mmol) of **1x**, 31.3mg **2x** was obtained as yellow liquid, R_f = 0.4 (PE:EA= 1:1), yield= 50%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (s, 1H), 7.51 – 7.46 (m, 2H), 7.36 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 1.7 Hz, 1H), 7.21 (s, 1H), 5.95 (ddd, *J* = 16.6, 10.2, 6.1 Hz, 1H), 5.33 (dt, *J* = 17.0, 1.5 Hz, 1H), 5.23 (d, *J* = 6.2 Hz, 1H), 5.14 (dt, *J* = 10.2, 1.4 Hz, 1H), 0.96 (t, *J* = 8.0 Hz, 9H), 0.64 (qd, *J* = 7.9, 2.0 Hz, 6H).

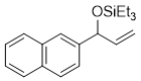
^{13}C NMR (126 MHz, $CDCl_3$) δ 143.4, 141.1, 136.5, 136.2, 130.3, 127.4, 121.3, 114.2, 75.1, 6.8, 4.9.

IR: 3411(s), 3176(s), 2927(s), 1630(s), 1455(m), 1401(s), 1240(w), 1146(w), 1065(m), 1010(m), 823 (m), 734(w), 611(w), 524(w).

HRMS: Calculation for $C_{18}H_{26}N_2OSi$: $[M+H^+]^+$: 313.1958, Found: 313.1959.



2y ((1-(naphthalen-1-yl)allyl)oxy)triethylsilane



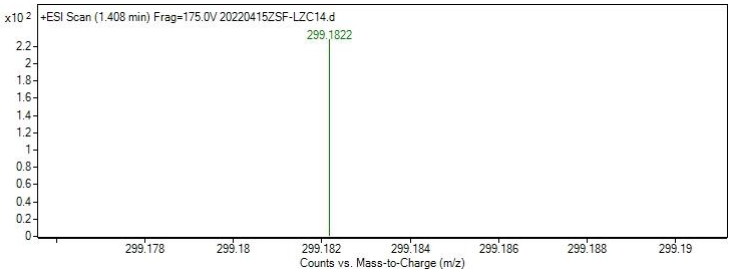
Followed GP4 with 31.2 mg (0.2 mmol) of **1z**, 33.2 mg **2z** was obtained as light-yellow liquid, R_f = 0.5 (PE), yield= 56%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.77 (m, 4H), 7.50 – 7.42 (m, 3H), 6.01 (ddd, *J* = 17.2, 10.2, 5.5 Hz, 1H), 5.39 – 5.29 (m, 2H), 5.11 (dt, *J* = 10.0, 1.3 Hz, 1H), 0.94 (t, *J* = 7.9 Hz, 9H), 0.62 (qd, *J* = 7.9, 3.0 Hz, 6H).

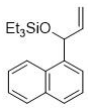
^{13}C NMR (101 MHz, $CDCl_3$) δ 141.6, 141.2, 133.3, 132.8, 128.0, 127.9, 127.7, 125.9, 125.6, 124.6, 124.4, 113.8, 75.8, 6.8, 4.9.

IR: 3455(w), 2955(w), 2880(w),1764(w), 1633(w), 1468(w), 1377(w), 1242(m), 1058(w),1009(w), 743(m), 701(w), 508(w).

HRMS: Calculation for $C_{19}H_{26}OSi$, $[M+H^+]^+$: 299.1826, Found: 299.1822.



2z ((1-(naphthalen-1-yl)allyl)oxy)triethylsilane



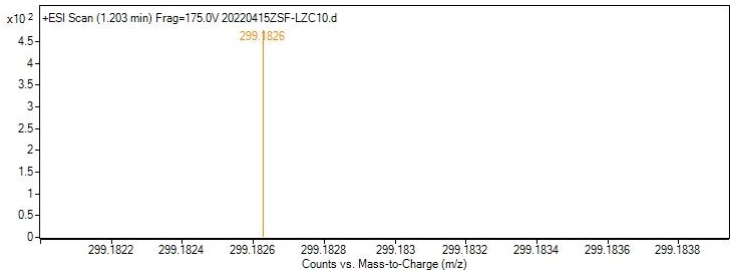
Followed GP4 with 31.2 mg (0.2 mmol) of **1y**, 33.1 mg **2y** was obtained as light-yellow liquid, R_f = 0.5 (PE), yield= 56%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.90 (dd, *J* = 7.6, 2.0 Hz, 1H), 7.81 (d, *J* = 8.3 Hz, 1H), 7.70 (dt, *J* = 7.2, 1.0 Hz, 1H), 7.54 – 7.49 (m, 3H), 6.18 (ddd, *J* = 17.1, 10.3, 5.2 Hz, 1H), 5.93 – 5.85 (m, 1H), 5.40 (dt, *J* = 17.1, 1.6 Hz, 1H), 5.14 (dt, *J* = 10.3, 1.6 Hz, 1H), 0.95 (t, *J* = 7.9 Hz, 9H), 0.64 (qd, *J* = 8.4, 8.0, 5.8 Hz, 6H).

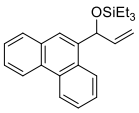
^{13}C NMR (126 MHz, $CDCl_3$) δ 141.0, 139.3, 133.9, 130.3, 128.8, 127.9, 125.6, 125.5, 125.4, 124.1, 124.0, 113.9, 73.6, 6.9, 4.9.

IR: 3461(s), 2955(m), 2879(m), 1764(m), 1643(m), 1487(w), 1408(m), 1242(s), 1080(m), 1009(m), 919(w), 843(w), 738(m), 518(w), 470(w).

HRMS: Calculation for $C_{19}H_{26}OSi$, $[M+H^+]^+$: 299.1826, Found: 299.1826.



2aa ((1-(phenanthren-9-yl)allyl)oxy)triethylsilane



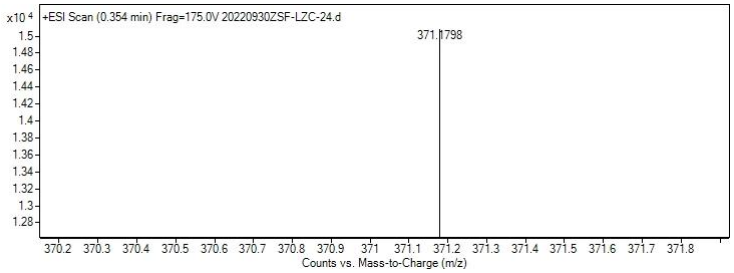
Followed GP4 with 41.2 mg (0.2 mmol) of **1aa**, 49.4 mg **2aa** was obtained as light-yellow liquid, R_f = 0.3 (PE), yield= 71%.

^{1}H NMR(400 MHz, $CDCl_3$) δ 8.76 (dd, *J* = 29.1, 8.0 Hz, 2H), 8.34 (d, *J* = 7.9 Hz, 1H), 8.05 – 7.91 (m, 2H), 7.68 (h, *J* = 6.9 Hz, 4H), 6.27 (ddd, *J* = 16.3, 10.3, 5.1 Hz, 1H), 5.94 (d, *J* = 5.1 Hz, 1H), 5.47 (d, *J* = 17.1 Hz, 1H), 5.22 (d, *J* = 10.3 Hz, 1H), 1.01 (t, *J* = 8.0 Hz, 9H), 0.72 (q, *J* = 7.3 Hz, 6H).

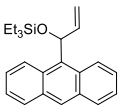
^{13}C NMR (101 MHz, $CDCl_3$) δ 140.8, 137.5, 131.7, 130.9, 130.2, 129.7, 128.8, 126.7, 126.5, 126.3, 126.2, 125.1, 124.9, 123.2, 122.5, 114.6, 74.0, 6.9, 5.0.

IR: 3449(s), 2957(m), 1743(w), 1640(w), 1458(w), 1381(w), 1243(m), 1048(m), 1009(w), 917(w), 840(w), 776(w),737(m).

HRMS: Calculation for $C_{23}H_{28}OSi$, $[M+H^+]^+$: 371.1801, Found: 371.1798.



2ab ((1-(anthracen-9-yl)allyl)oxy)triethylsilane



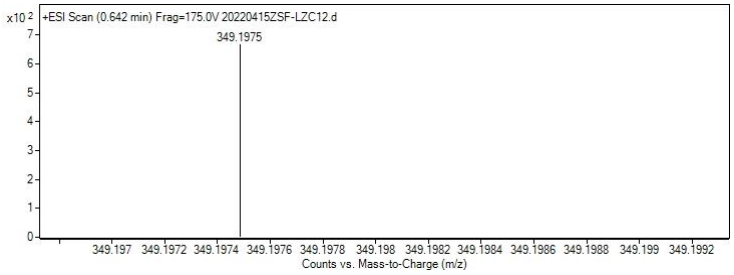
Followed GP4 with 41.2 mg (0.2 mmol) of **1ab**, 24.4 mg **2ab** was obtained as light-yellow liquid, R_f = 0.5 (PE), yield = 35%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.66 (s, 2H), 8.43 (s, 1H), 8.02 (dd, *J* = 7.5, 2.2 Hz, 2H), 7.59 – 7.38 (m, 4H), 6.76 (dt, *J* = 4.3, 2.1 Hz, 1H), 6.43 (ddd, *J* = 17.1, 10.4, 4.2 Hz, 1H), 5.43 (dt, *J* = 17.1, 1.8 Hz, 1H), 5.20 (dt, *J* = 10.4, 1.9 Hz, 1H), 0.79 (t, *J* = 7.9 Hz, 9H), 0.59 – 0.35 (m, 6H).

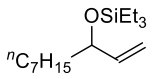
^{13}C NMR (126 MHz, $CDCl_3$) δ 140.9, 134.1, 131.7, 129.3, 129.1, 127.9, 127.3, 125.3, 124.7, 113.8, 70.9, 6.6, 4.7.

IR: 3452(s), 2990(w), 1763(s), 1637(m), 1377(m), 1243(s), 1055(m), 915(w), 743(m).

HRMS: Calculation for $C_{23}H_{28}OSi$, $[M+H^+]^+$: 349.1982, Found: 349.1975.



2ba (undec-1-en-3-yloxy)triethylsilane



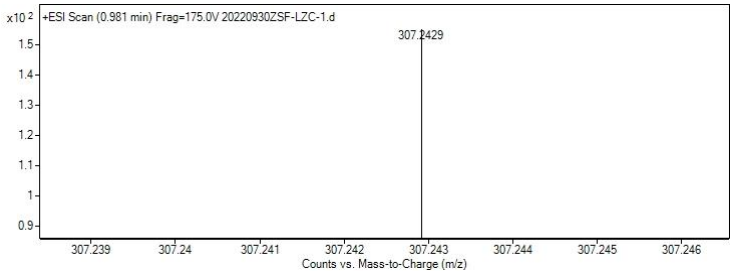
Followed GP4 with 25.6 mg (0.2 mmol) of **1ba**, 37.6 mg **2ba** was obtained as colorless liquid, R_f = 0.6 (PE), yield= 66%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.82 (ddd, *J* = 16.9, 10.3, 6.4 Hz, 1H), 5.15 (dt, *J* = 17.2, 1.6 Hz, 1H), 5.03 (dt, *J* = 10.4, 1.5 Hz, 1H), 4.08 (qt, *J* = 6.3, 1.3 Hz, 1H), 1.68 – 1.23 (m, 14H), 0.98 (t, *J* = 7.9 Hz, 9H), 0.90 (t, *J* = 6.7 Hz, 3H), 0.62 (q, *J* = 8.1 Hz, 6H).

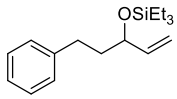
^{13}C NMR (101 MHz, $CDCl_3$) δ 141.9, 113.5, 73.9, 38.2, 31.9, 29.6, 29.3, 25.3, 22.7, 14.1, 6.9, 4.9.

IR: 3547(w), 3398(w), 3160(m), 2982(w), 1741(m), 1615(w), 1401(m), 1244(m), 1047(m), 851(w), 734 (m), 611(m).

HRMS: Calculation for $C_{17}H_{36}OSi$, $[M+Na^+]^+$: 307.2427, Found: 307.2429.



2bb ((5-phenylpent-1-en-3-yl)oxy)triethylsilane



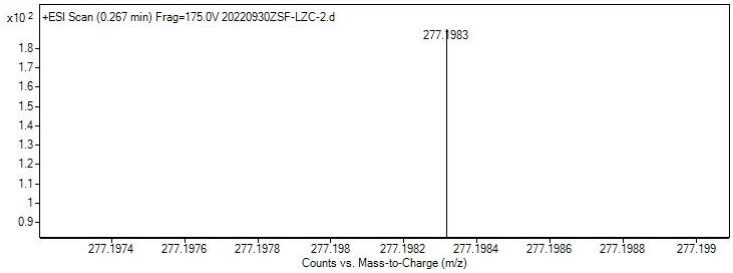
Followed GP4 with 26.8 mg (0.2 mmol) of **1bb**, 42.3 mg **2bb** was obtained as colorless liquid, R_f = 0.7 (PE), yield= 76%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (td, *J* = 7.1, 1.6 Hz, 2H), 7.24 – 7.20 (m, 3H), 5.89 (ddd, *J* = 16.9, 10.3, 6.4 Hz, 1H), 5.22 (dt, *J* = 17.1, 1.5 Hz, 1H), 5.11 (dt, *J* = 10.4, 1.4 Hz, 1H), 4.18 (qt, *J* = 6.2, 1.2 Hz, 1H), 2.78 – 2.62 (m, 2H), 1.86 (tt, *J* = 9.5, 6.3 Hz, 2H), 1.00 (t, *J* = 7.9 Hz, 9H), 0.64 (q, *J* = 7.7 Hz, 6H).

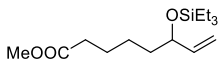
^{13}C NMR (126 MHz, $CDCl_3$) δ 142.4, 141.4, 128.4, 128.3, 125.7, 114.2, 73.4, 39.8, 31.5, 6.9, 5.0.

IR: 3689(w), 3478(w), 3409(w), 3104(s), 1401(s), 1164(m), 1050(m), 958(m), 617(w), 473(w).

HRMS: Calculation for $C_{17}H_{28}OSi$, $[M+Na^+]^+$: 277.1982, Found: 277.1983.



2bc methyl 6-((triethylsilyl)oxy)oct-7-enoate



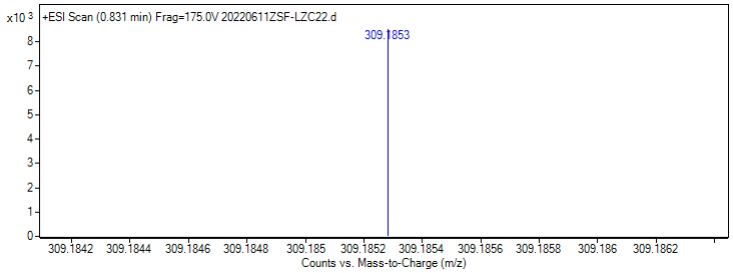
Followed GP4 with 28.8mg (0.2 mmol) of **1bc**, 44.1mg **2bc** was obtained as colorless liquid, R_f = 0.5 (PE:EA = 40:1), yield= 77%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.80 (ddd, *J* = 16.9, 10.3, 6.4 Hz, 1H), 5.14 (dt, *J* = 17.0, 1.4 Hz, 1H), 5.03 (ddd, *J* = 10.3, 1.9, 1.0 Hz, 1H), 4.08 (q, *J* = 6.2 Hz, 1H), 3.67 (s, 3H), 2.32 (t, *J* = 7.6 Hz, 2H), 1.64 (p, *J* = 7.5 Hz, 2H), 1.56 – 1.32 (m, 4H), 0.96 (t, *J* = 7.9 Hz, 9H), 0.60 (q, *J* = 7.7 Hz, 6H).

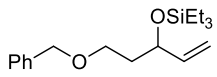
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.2, 141.6, 113.8, 73.6, 51.4, 37.7, 34.1, 25.0, 24.7, 6.8, 4.9.

IR: 3449(s), 2955(s), 1742(m), 1633(s), 1401(s), 1240(w), 1072(m), 1007(m), 923(m), 733(m).

HRMS: Calculation for $C_{15}H_{30}O_3Si$, $[M+Na^+]^+$: 309.1856, Found: 309.1853.



2bd ((5-(benzyloxy)pent-1-en-3-yl)oxy)triethylsilane



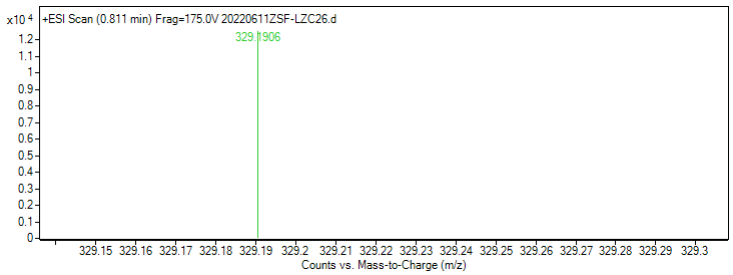
Followed GP4 with 32.8 mg (0.2 mmol) of **1bd**, 30.4 mg **2bd** was obtained as light-yellow liquid, R_f = 0.5 (PE:EA= 10:1), yield= 50%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.29 (m, 5H), 5.85 (ddd, *J* = 16.9, 10.3, 6.4 Hz, 1H), 5.18 (dt, *J* = 17.2, 1.5 Hz, 1H), 5.05 (dt, *J* = 10.4, 1.4 Hz, 1H), 4.52 (q, *J* = 11.9 Hz, 2H), 4.34 (q, *J* = 6.4 Hz, 1H), 3.58 (ddt, *J* = 30.9, 9.3, 6.4 Hz, 2H), 1.83 (qd, *J* = 6.2, 1.9 Hz, 2H), 0.98 (t, *J* = 8.0 Hz, 9H), 0.66 – 0.59 (m, 6H).

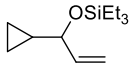
^{13}C NMR (101 MHz, $CDCl_3$) δ 141.6, 138.6, 128.3, 127.7, 127.5, 113.9, 73.0, 70.9, 66.7, 38.2, 6.8, 4.9.

IR: 3413(s), 3134(s), 1625(m), 1401(s), 1142(w), 953(w), 739(w), 619(w), 479(w).

HRMS: Calculation for $C_{18}H_{30}O_2Si$, $[M+Na^+]^+$: 329.1907, Found: 329.1906.



2be ((1-cyclopropylallyl)oxy)triethylsilane



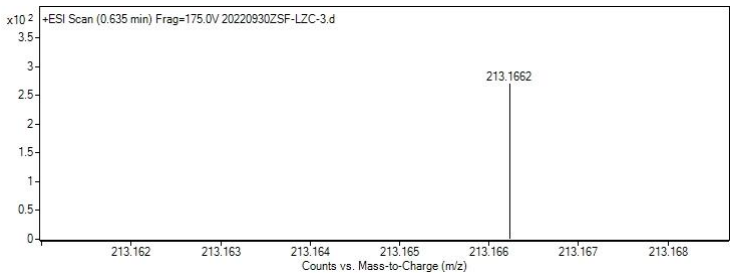
Followed GP4 with 14.0 mg (0.2 mmol) of **1be**, 27.8 mg **2be** was obtained as light-yellow liquid, R_f = 0.7 (PE), yield= 66%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.92 (ddd, *J* = 17.2, 10.4, 5.9 Hz, 1H), 5.19 (dt, *J* = 17.1, 1.6 Hz, 1H), 5.04 (ddd, *J* = 10.4, 1.9, 1.2 Hz, 1H), 3.60 (tt, *J* = 5.8, 1.4 Hz, 1H), 0.98 (t, *J* = 7.9 Hz, 10H), 0.62 (d, *J* = 7.9 Hz, 6H), 0.51 – 0.43 (m, 2H), 0.37 – 0.31 (m, 1H), 0.28 – 0.21 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 140.91, 113.31, 17.63, 6.82, 5.00, 3.14, 1.78.

IR: 3380(w), 2916(w), 1746(w), 1627(w), 1462(w), 1244(w), 1149(w), 1055(w), 958(w), 736(w), 616 (w), 527(w).

HRMS: Calculation for $C_{12}H_{24}OSi$, $[M+H^+]^+$: 213.1669, Found: 213.1662.



2bf ((1-cyclobutylallyl)oxy)triethylsilane

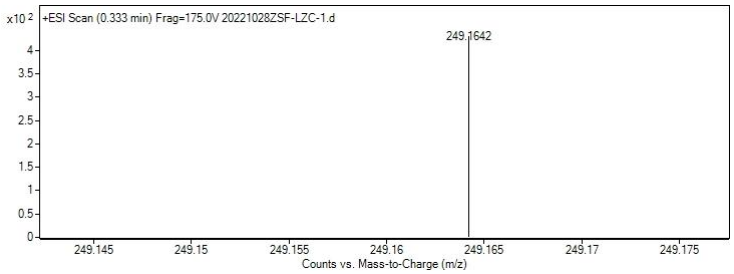
Followed GP4 with 16.8 mg (0.2 mmol) of **1bf**, 28.6 mg **2bf** was obtained as light-yellow liquid, R_f = 0.7 (PE), yield= 63%.

^{1}H NMR(400 MHz, $CDCl_3$) δ 5.75 (ddd, *J* = 17.0, 10.4, 6.4 Hz, 1H), 5.15 (dt, *J* = 17.2, 1.6 Hz, 1H), 5.03 (ddd, *J* = 10.4, 2.2, 1.1 Hz, 1H), 3.98 (t, *J* = 6.8 Hz, 1H), 2.45 – 2.27 (m, 1H), 1.98 (ddd, *J* = 11.5, 7.1, 4.2 Hz, 1H), 1.88 (ddt, *J* = 12.4, 7.5, 3.5 Hz, 3H), 1.78 (ddt, *J* = 6.8, 4.9, 3.5 Hz, 2H), 0.98 (t, *J* = 7.9 Hz, 9H), 0.63 (q, *J* = 7.7 Hz, 6H).

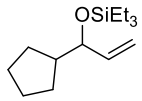
^{13}C NMR (126 MHz, $CDCl_3$) δ 139.8, 114.0, 41.4, 24.5, 24.0, 17.8, 6.9, 5.0.

IR: 3444(s), 2963(m), 1718 (m), 1641(m), 1408(w), 1319(w), 1270(w), 1170 (w), 1083(w), 1048 (w), 837(w), 735(m), 536(w).

HRMS: Calculation for $C_{13}H_{26}OSi$, $[M+Na^+]^+$: 249.1645, Found: 249.1642.



2bg ((1-cyclopentylallyl)oxy)triethylsilane



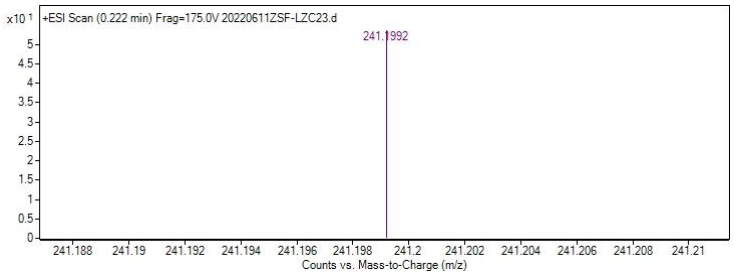
Followed GP4 with 19.6 mg (0.2 mmol) of **1bg**, 31.1 mg **2bg** was obtained as light-yellow liquid, R_f = 0.7 (PE), yield= 65%.

^{1}H NMR (400 MHz,$CDCl_3$) δ 5.83 (ddd, *J* = 17.2, 10.3, 6.9 Hz, 1H), 5.14 (ddd, *J* = 17.2, 1.9, 1.1 Hz, 1H), 5.04 (ddd, *J* = 10.3, 2.0, 1.0 Hz, 1H), 3.90 (tt, *J* = 7.0, 1.1 Hz, 1H), 1.94 (q, *J* = 7.8 Hz, 1H), 1.81 – 1.69 (m, 1H), 1.63 – 1.49 (m, 5H), 1.40 (ddd, *J* = 12.3, 10.0, 6.7 Hz, 1H), 1.27 – 1.17 (m, 1H), 0.97 (t, *J* = 7.9 Hz, 9H), 0.65 – 0.58 (m, 6H).

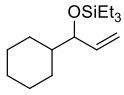
^{13}C NMR (101 MHz, $CDCl_3$) δ 141.3, 114.0, 78.0, 46.6, 28.8, 25.6, 6.9, 5.1.

IR: 3452(s), 3139(s), 2957(s), 1634(s), 1401(s), 1240(w), 1072(m), 1007(m), 923(m), 733(m).

HRMS: Calculation for $C_{14}H_{28}OSi$, $[M+H^+]^+$: 241.1982, Found: 241.1992.



2bh ((1-cyclohexylallyl)oxy)triethylsilane



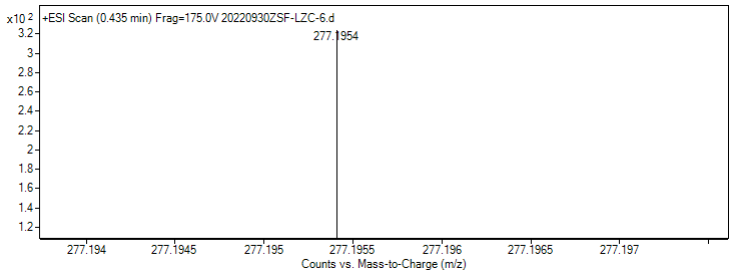
Followed GP4 with 22.4 mg (0.2 mmol) of **1bh**, 36.7 mg **2bh** was obtained as light-yellow liquid, R_f = 0.7 (PE), yield= 72%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.80 (ddd, *J* = 17.2, 10.3, 7.0 Hz, 1H), 5.15 – 5.00 (m, 2H), 3.80 (t, *J* = 6.6 Hz, 1H), 1.85 (dtd, *J* = 12.3, 3.7, 2.0 Hz, 1H), 1.79 – 1.64 (m, 5H), 1.35 (dtd, *J* = 11.7, 5.9, 5.4, 2.8 Hz, 1H), 1.26 – 1.05 (m, 4H), 0.98 (d, *J* = 7.9 Hz, 9H), 0.64 – 0.57 (m, 6H).

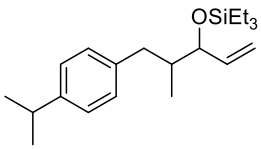
^{13}C NMR (101 MHz, $CDCl_3$) δ 140.5, 114.6, 78.7, 44.3, 28.8, 26.7, 26.3, 6.9, 5.0.

IR: 3411(s), 3176(s), 2927(s), 1630(s), 1455(m), 1401(s), 1240(w), 1146(w), 1065(m), 1010(m), 823 (m), 734(w), 611(w), 524(w).

HRMS: Calculation for $C_{15}H_{30}OSi$, $[M+Na^+]^+$: 277.1958, Found: 277.1954.



2ca ((5-(4-isopropylphenyl)-4-methylpent-1-en-3-yl)oxy)triethylsilane



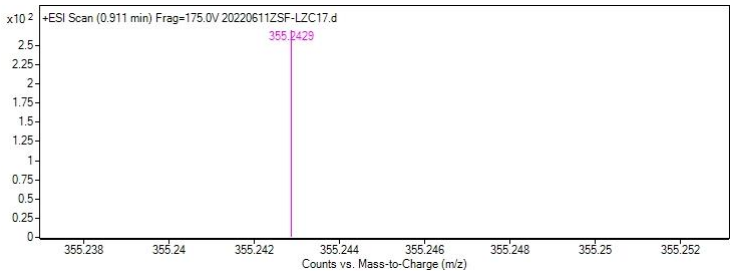
Followed GP4 with 38.0 mg (0.2 mmol) of **1ca**, 60.1 mg **2ca** was obtained as light-yellow liquid, R_f = 0.7 (PE), yield= 90%, 1.5:1 dr.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (dd, *J* = 8.1, 1.9 Hz, 2H), 7.12 (dd, *J* = 8.1, 3.9 Hz, 2H), 5.91 (ddd, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.22 (d, *J* = 17.1 Hz, 1H), 5.19 – 5.13 (m, 1H), 4.10 (dd, *J* = 6.5, 4.7 Hz, 1H), 3.01 – 2.84 (m, 2H), 2.22 (ddd, *J* = 23.6, 13.5, 10.0 Hz, 1H), 1.88 (ddd, *J* = 13.1, 6.7, 4.0 Hz, 1H), 1.29 (d, *J* = 6.9 Hz, 6H), 1.01 (dt, *J* = 13.8, 7.9 Hz, 9H), 0.85 (dd, *J* = 6.8, 2.5 Hz, 3H), 0.65 (dt, *J* = 15.3, 7.8 Hz, 6H).

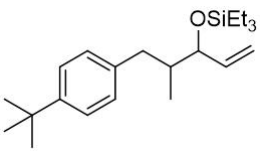
^{13}C NMR (101 MHz, $CDCl_3$) δ 146.2, 139.6, 138.9, 129.0, 126.2, 115.1, 77.6, 42.0, 38.4, 33.7, 24.1, 14.6, 7.0, 5.1.

IR: 3553(s), 3404(s), 3138(s), 2960(s), 1763(w), 1625(m), 1512(w), 1459(w), 1401(s), 1292(w), 1241(w), 1146(w), 1064(m), 1016(w), 955(w), 827(w), 735(w), 614(w), 524(w).

HRMS: Calculation for $C_{21}H_{36}OSi$, $[M+Na^+]^+$: 355.2428, Found: 355.2429.



2cb ((5-(4-(tert-butyl)phenyl)-4-methylpent-1-en-3-yl)oxy)triethylsilane



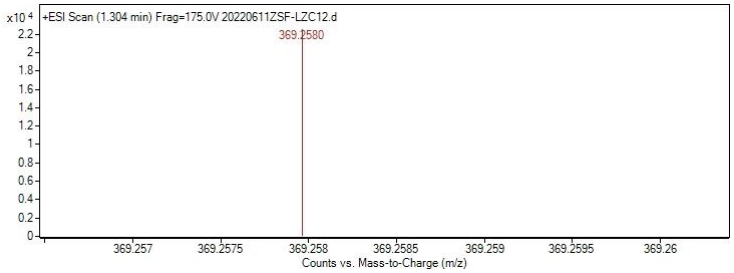
Followed GP4 with 40.8 mg (0.2 mmol) of **1cb**, 58.8 mg **2cb** was obtained as light-yellow liquid, R_f = 0.7 (PE), yield= 85%, 1.5:1 dr.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (dd, *J* = 8.3, 2.1 Hz, 2H), 7.12 (dd, *J* = 8.1, 4.2 Hz, 2H), 5.90 (dtd, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.22 (dt, *J* = 17.2, 1.5 Hz, 1H), 5.19 – 5.13 (m, 1H), 4.09 (t, *J* = 5.5 Hz, 1H), 2.92 (ddd, *J* = 24.3, 13.4, 4.5 Hz, 1H), 2.22 (ddd, *J* = 23.6, 13.4, 10.0 Hz, 1H), 1.88 (ddq, *J* = 10.6, 8.1, 3.8, 3.3 Hz, 1H), 1.35 (s, 9H), 1.05 – 0.98 (m, 9H), 0.84 (dd, *J* = 6.8, 2.7 Hz, 3H), 0.65 (dt, *J* = 15.6, 7.9 Hz, 6H).

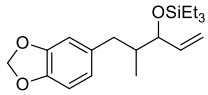
^{13}C NMR (126 MHz, $CDCl_3$) δ 148.4, 139.5, 138.5, 128.9, 125.1, 115.1, 77.6, 42.0, 38.2, 34.4, 31.4, 14.6, 6.9,5.0.

IR: 3370(m), 3128(m), 2961(m), 1627(m), 1460(w), 1401(m), 1293(w), 1241(w), 1149(w), 1063(w), 957(w), 829(w), 735(w), 613(w), 527(w).

HRMS: Calculation for $C_{22}H_{38}OSi$, $[M+Na^+]^+$: 369.2584, Found: 369.2580.



2cc ((5-(benzo[d][1,3]dioxol-5-yl)-4-methylpent-1-en-3-yl)oxy)triethylsilane.



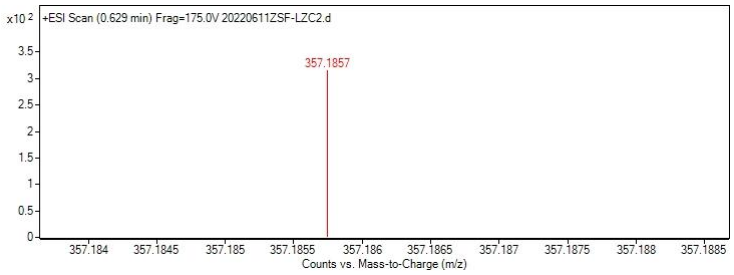
Followed GP3 with 40.8 mg (0.2 mmol) of **1cc**, 58.8 mg **2cc** was obtained as light-yellow liquid, R_f = 0.7 (PE), yield= 59%, 1.5:1 dr.

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.78 – 6.51 (m, 3H), 5.97 – 5.76 (m, 3H), 5.22 – 5.05 (m, 2H), 4.02 (t, *J* = 5.6 Hz, 1H), 2.83 (ddd, *J* = 25.8, 13.5, 4.5 Hz, 1H), 2.12 (ddd, *J* = 28.5, 13.5, 10.1 Hz, 1H), 1.77 (tdq, *J* = 10.6, 6.6, 3.9, 2.5 Hz, 1H), 0.96 (dt, *J* = 11.7, 7.9 Hz, 9H), 0.78 (dd, *J* = 6.9, 3.9 Hz, 3H), 0.65 – 0.55 (m, 6H).

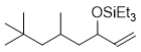
^{13}C NMR (126 MHz, $CDCl_3$) δ 147.4, 145.4, 139.5, 135.4, 121.9, 115.2, 109.4, 107.9, 100.7,, 77.5, 42.2, 38.4, 14.5, 6.9, 5.1.

IR: 3407(m), 2958(m), 2881(m), 1635(m), 1493(m), 1403(m), 1245(m), 1191(w), 1081(m), 1041(m), 932(w), 809(w), 734(m).

HRMS: Calculation for $C_{19}H_{30}O_3Si$, $[M+Na^+]^+$: 357.1856, Found: 357.1857.



2cd ((5,7,7-trimethyloct-1-en-3-yl)oxy)triethylsilane



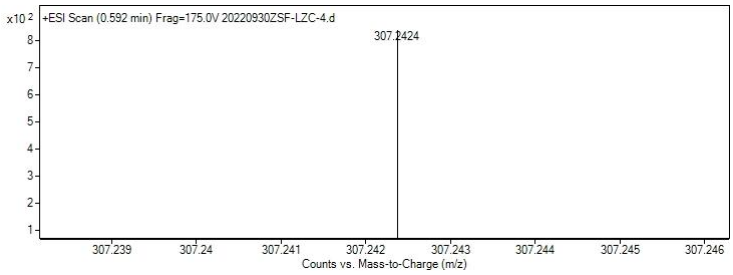
Followed GP4 with 28.4 mg (0.2 mmol) of **1cd**, 40.7 mg **2cd** was obtained as colorless liquid, R_f = 0.7 (PE), yield= 72%, 1.6:1 dr.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.82 (dddd, *J* = 17.2, 10.3, 8.2, 6.8 Hz, 1H), 5.20 – 5.12 (m, 1H), 5.02 (ddd, *J* = 10.3, 1.8, 1.0 Hz, 1H), 4.20 – 4.06 (m, 1H), 1.60 – 1.34 (m, 2H), 1.30 – 1.17 (m, 2H), 1.10 (t, *J* = 6.2 Hz, 1H), 1.01 – 0.90 (m, 21H), 0.66 – 0.59 (m, 6H).

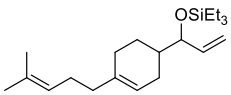
^{13}C NMR (126 MHz, $CDCl_3$) δ 142.1, 113.8, 72.5, 51.9, 48.2, 31.2, 30.0, 25.5, 22.6, 6.9, 5.0.

IR: 3407(s), 3174(s), 2961(s), 1627(s), 1401(s), 1293(w), 1241(w), 1148(w), 1061(w), 955(w), 735(w), 613(w), 525(w).

HRMS: Calculation for $C_{17}H_{36}OSi$, $[M+Na^+]^+$: 307.2427, Found: 307.2424.



2ce ((1-(4-(4-methylpent-3-en-1-yl)cyclohex-3-en-1-yl)allyl)oxy)triethylsilane



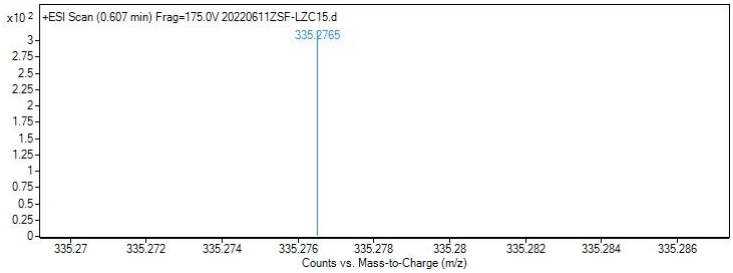
Followed GP4 with 38.4 mg (0.2 mmol) of **1ce**, 36.3 mg **2ce** was obtained as yellow liquid, R_f = 0.7 (PE), yield= 54%, 2:1 dr.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.82 (ddd, *J* = 17.3, 10.3, 7.1, Hz, 1H), 5.40 (q, *J* = 5.3, 4.2 Hz, 1H), 5.14 (dddt, *J* = 18.7, 14.5, 9.7, 2.0 Hz, 3H), 3.93 (td, *J* = 6.8, 3.1 Hz, 1H), 2.10 (p, *J* = 7.0, 6.5 Hz, 3H), 1.98 (q, *J* = 8.5, 7.8 Hz, 4H), 1.93 – 1.82 (m, 1H), 1.79 – 1.69 (m, 4H), 1.63 (s, 3H), 1.37 – 1.11 (m, 2H), 0.98 (td, *J* = 7.9, 2.6 Hz, 9H), 0.66 – 0.57 (m, 6H).

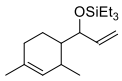
^{13}C NMR (101 MHz, $CDCl_3$) δ 140.4, 137.5, 131.2, 124.5, 120.3, 114.7, 78.0, 40.5, 37.8, 28.4, 27.5, 26.5, 25.4, 17.6, 6.9, 5.0.

IR: 3411(s), 3132(s), 2963(s), 1764(w), 1625(m), 1460(w), 1401(s), 1293(w), 1241(w), 1151(w), 1060(w), 952(w), 737(w), 616(w), 525(w).

HRMS: Calculation for $C_{21}H_{38}OSi$, $[M+H^+]^+$: 335.2765, Found: 335.2765.



2cf ((1-(2,4-dimethylcyclohex-3-en-1-yl)allyl)oxy)triethylsilane



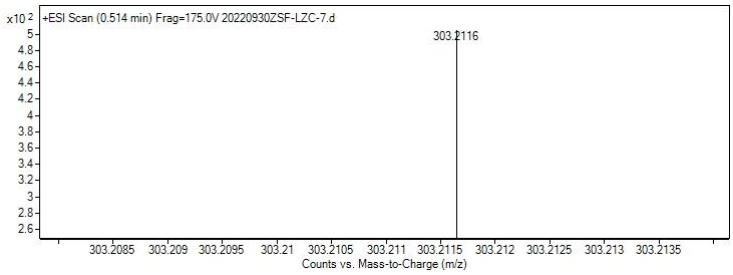
Followed GP4 with 27.6 mg (0.2 mmol) of **1cf**, 33.9 mg **2cf** was obtained as yellow liquid, R_f = 0.7 (PE), yield= 60%, 1:1 dr.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.82 (ddd, *J* = 17.7, 10.3, 7.7 Hz, 1H), 5.36 (dt, *J* = 5.6, 1.7 Hz, 1H), 5.21 (dd, *J* = 17.2, 1.8 Hz, 1H), 5.11 (dd, *J* = 10.3, 1.8 Hz, 1H), 3.87 (dd, *J* = 9.6, 7.7 Hz, 1H), 2.12 (d, *J* = 6.6 Hz, 1H), 1.95 (td, *J* = 13.8, 11.8, 7.6 Hz, 3H), 1.65 – 1.55 (m, 4H), 1.38 – 1.30 (m, 1H), 0.98 (t, *J* = 7.9 Hz, 9H), 0.84 (d, *J* = 6.9 Hz, 3H), 0.66 – 0.59 (m, 6H).

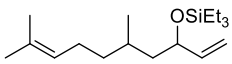
^{13}C NMR(101 MHz, $CDCl_3$) δ 140.7, 133.0, 127.7, 115.1, 76.0, 44.2, 31.0, 30.1, 23.5, 19.5, 15.0, 6.9, 5.1.

IR: 3451(s), 1635(s), 1401(s), 1240(w), 1062(m), 734(m), 520(m).

HRMS: Calculation for $C_{17}H_{32}OSi$, $[M+H^+]^+$: 303.2114, Found: 303.2116.



2cg ((4,9-dimethyldeca-1,8-dien-3-yl)oxy)triethylsilane



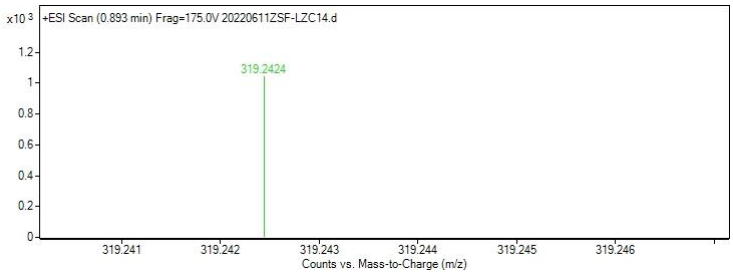
Followed GP4 with 30.8 mg (0.2 mmol) of **1cg**, 32.1 mg **2cg** was obtained as yellow liquid, R_f = 0.7 (PE), yield= 54%, 1:1 dr.

^{1}H NMR (500 MHz, $CDCl_3$) δ 5.79 (dddd, J = 17.2, 14.1, 10.3, 6.7 Hz, 1H), 5.15 – 5.07 (m, 2H), 5.01 (ddt, J = 10.1, 8.4, 1.4 Hz, 1H), 4.18 – 4.11 (m, 1H), 1.97 (tq, J = 15.7, 7.6 Hz, 2H), 1.68 (t, J = 1.4 Hz, 3H), 1.60 (s, 3H), 1.53 (ddd, J = 17.6, 8.7, 4.6 Hz, 1H), 1.44 – 1.31 (m, 2H), 1.21 – 1.03 (m, 2H), 0.98 – 0.93 (m, 9H), 0.89 (dd, J = 6.6, 1.3 Hz, 3H), 0.62 – 0.57 (m, 6H).

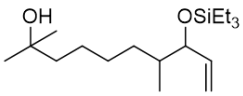
^{13}C NMR (126 MHz, $CDCl_3$) δ 142.2, 131.1, 124.9, 113.6, 72.3, 45.7, 37.4, 28.6, 25.5, 19.7, 17.6, 6.9, 5.0.

IR: 3414(s), 3182(s), 2961(s), 1757(m), 1629(m), 1458(w), 1400(s), 1242(s), 1149(w), 1055(m), 917(w), 839(w), 738(m), 610(w), 527(w).

HRMS: Calculation for $C_{18}H_{36}OSi$, $[M+Na^+]^+$: 319.2427, Found: 319.2424.



2ch 7-dimethyl-8-((triethylsilyl)oxy)dec-9-en-2-ol



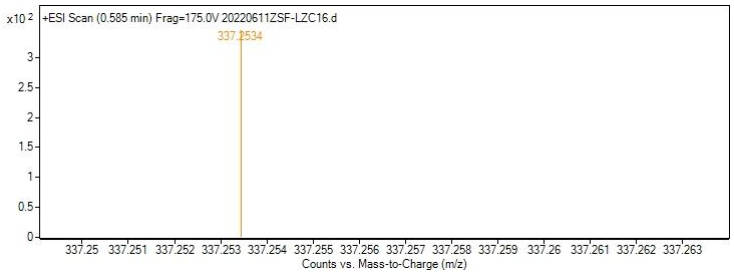
Followed GP4 with 38.4 mg (0.2mmol) of **1ch**, 48.4 mg **2ch** was obtained as yellow liquid, R_f = 0.7 (PE), yield= 72%, 1:1 dr.

^{1}H NMR (500 MHz, $CDCl_3$) δ 5.86 – 5.72 (m, 1H), 5.12 (d, *J* = 17.1 Hz, 1H), 5.01 (t, *J* = 10.9 Hz, 1H), 4.15 (t, *J* = 6.7 Hz, 1H), 1.62 (p, *J* = 6.5 Hz, 1H), 1.58 – 1.27 (m, 9H), 1.21 (s, 6H), 0.95 (t, *J* = 7.9 Hz, 9H), 0.90 (d, *J* = 6.6 Hz, 3H), 0.60 (q, *J* = 7.9 Hz, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 142.1, 113.7, 72.2, 71.0, 45.7, 44.2, 37.8, 29.1, 28.8, 21.7, 19.8, 6.9, 5.0.

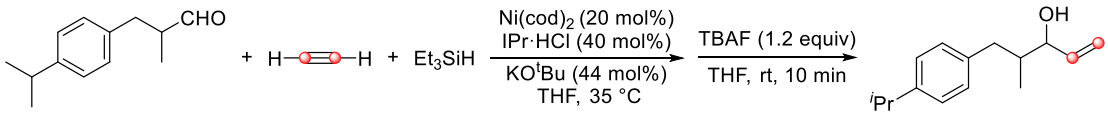
IR: 3419(s), 3187(s), 2963(m), 1764(w), 1630(s), 1400(s),1241(m), 1060(m), 739(w), 611(w), 528(w).

HRMS: Calculation for $C_{18}H_{38}O_2Si$, $[M+Na^+]^+$: 337.2533, Found: 337.2534.


x10 2
+ESI Scan (0.585 min) Frag=175.0V 20220611ZSF-LZC16.d
337.2534
3
2.5
2
1.5
1
0.5
0
337.25 337.251 337.252 337.253 337.254 337.255 337.256 337.257 337.258 337.259 337.26 337.261 337.262 337.263
Counts vs. Mass-to-Charge (m/z)

Gram-Scale Reaction

3ca 5-(4-isopropylphenyl)-4-methylpent-1-en-3-ol



1) 50 mg $Ni(cod)_2$ (2 mmol, 20 mol%), 1.7 g IPr·HCl (4 mmol, 40 mol%) were weighed out and transferred into a Schlenk tube in the glove box.

The tube was then sealed with a hollow glass plug and taken out of the glovebox.

2) Putting the tube under nitrogen via 3 vacuum / N_2 backfill cycles and unplugged the hollow glass plug.

3) Add 20 mL THF and 4.4 mL KO^{t}Bu (4.4 mmol, 44 mol%,1 mol/L in THF).

4) Stir the solution at room temperature until all the solid dissolved (2 min).

5) Add 4.65 g (6.4 mL, ρ= 0.728 g/mL at 25 ℃) triethylsilane (40 mmol, 4 equiv) via syringe after the same operation of step 3.

6) Stir the solution at room temperature for 2 min.

7) Add 1.9 g cyclamen aldehyde (10 mmol) dissolved in 37 mL (2.05 equiv) saturated solution of acetylene in THF (0.55 mol/L).

8) Stir the solution at 35 ℃ for about 18 h, monitored by TLC.

9) After completion of reaction, filtrate the mixture by a short pad of silica gel and wash with EA and was then concentrated under reduced pressure.

10) Purify the crude by column chromatography with PE (R_f = 0.6).

11) Add 12 mL TBAF (12 mmol, 1.2 equiv, 1 mol/L in THF) to the purified product in 25 mL round bottom flask.

12) Stir the solution for 10 min, monitored by TLC.

13) Quench the reaction by adding saturated NH_4Cl and extract with EA.

14) Concentrated the residue under reduced pressure.

15) Separate the residue by column chromatography with PE:EA= 10:1 (R_f = 0.4).

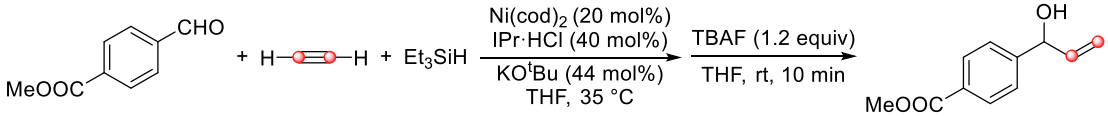
After the above procedures, 1.68 g **3ca** was obtained as light-yellow liquid, yield= 77%, 1.5:1 dr.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (qd, *J* = 8.3, 2.9 Hz, 4H), 6.04 – 5.93 (m, 1H), 5.39 – 5.19 (m, 2H), 4.13 (t, *J* = 5.0 Hz, 1H), 2.94 (tt, *J* = 13.0, 6.4 Hz, 2H), 2.43 (td, *J* = 13.0, 9.1 Hz, 1H), 1.98 (q, *J* = 6.7 Hz, 1H), 1.34 – 1.31 (m, 6H), 0.94 (dd, *J* = 8.3, 6.8 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 146.4, 146.3, 139.9, 139.0, 138.3, 138.1, 129.2, 129.1, 126.3, 126.3, 116.2, 115.2, 75.5, 40.7, 40.6, 38.8, 38.3, 33.7, 24.1, 14.9, 13.9.

Characterization data matched those reported in the literature.[2]

3q methyl 4-(1-hydroxyallyl)benzoate



1) 550 mg $Ni(cod)_2$ (2 mmol, 20 mol%), 1.7g IPr·HCl (4 mmol, 40 mol%) were weighed out and transferred into a Schlenk tube in the glove box.

The tube was then sealed with a hollow glass plug and taken out of the glovebox.

2) Putting the tube under nitrogen via 3 vacuum / N_2 backfill cycles and unplugged the hollow glass

plug.

3) Add 20 mL THF and 4.4mL KO^{t}Bu (4.4 mmol, 44 mol%,1 mol/L in THF).

4) Stir the solution at room temperature until all the solid dissolved (2min).

5) Add 4.65 g (6.4 mL, ρ= 0.728 g/mL at 25 ℃) triethylsilane (40 mmol, 4 equiv) via syringe after the same operation of step 3.

6) Stir the solution at room temperature for 2 min.

7) Add 1.64 g methyl 4-formylbenzoate (10 mmol) dissolved in 37 mL (2.05 equiv) saturated solution of acetylene in THF (0.55 mol/L).

8) Stir the solution at 35 ℃ for about 18 h, monitored by TLC.

9) After completion of reaction, filtrate the mixture by a short pad of silica gel and wash with EA and was then concentrated under reduced pressure.

10) Purify the crude by column chromatography with PE:EA= 100:1 (R_f = 0.3).

11) Add 12 mL TBAF (12 mmol, 1.2 equiv, 1 mol/L in THF) to the purified product in 25 mL round bottom flask.

12) Stir the solution for 10 min, monitored by TLC.

13) Quench the reaction by adding saturated NH_4Cl and extract with EA.

14) Concentrated the residue under reduced pressure.

15) Separate the residue by column chromatography with PE:EA= 5:1 (R_f = 0.4).

After the above procedures, 0.96 g **3q** was obtained as yellow liquid, yield= 50%.

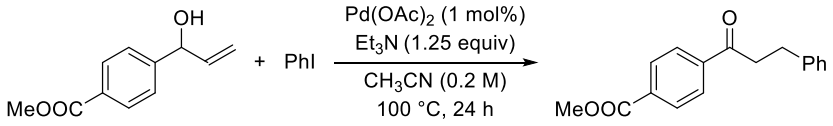
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.3 Hz, 2H), 7.45 (d, *J* = 8.3 Hz, 2H), 6.02 (ddd, *J* = 16.8, 10.3, 6.2 Hz, 1H), 5.36 (dt, *J* = 17.1, 1.3 Hz, 1H), 5.28 – 5.18 (m, 2H), 3.92 (s, 3H), 2.52 (s, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.0, 147.6, 139.7, 129.8, 129.4, 126.2, 115.9, 75.0, 52.1.

Characterization data matched those reported in the literature.[3]

Synthetic Applications of Products.

4 methyl 4-(3-phenylpropanoyl)benzoate



3q **4**

1) Add 40.4 mg iodobenzene (0.2 mmol), 48.0 mg **3q** (0.25 mmol) and 0.45 mg $Pd(OAc)_2$ (0.002 mmol, 1 mol%) to 25 ml Schlenk tube.

2) Evacuate the Schlenk tube with oil pump and refill the tube with N_2, repeat it 3 times.

3) Add 1 mL anhydrous CH_3CN to the Schlenk tube.

4) Stir the mixture for 24 h at 100 ℃.

5) Filtrate the mixture by thin layer of silica gel.

6) Concentrated the residue under reduced pressure.

7) Separate the residue by column chromatography with PE:EA= 40:1 (R_f = 0.4).

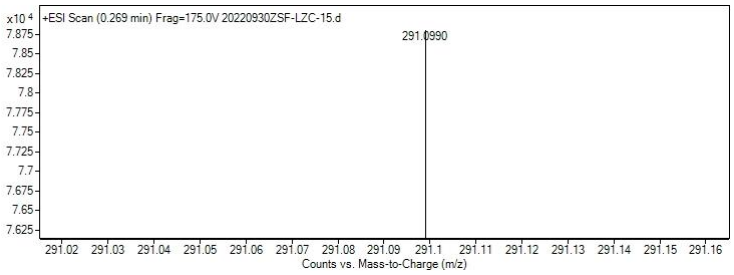
After the above procedures, 44.9 mg **4** was obtained as white solid, yield= 84%.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.1 Hz, 2H), 7.90 (d, *J* = 8.2 Hz, 2H), 7.27 – 7.07 (m, 5H), 3.85 (s, 3H), 3.23 (t, *J* = 7.6 Hz, 2H), 2.98 (t, *J* = 7.6 Hz, 2H).

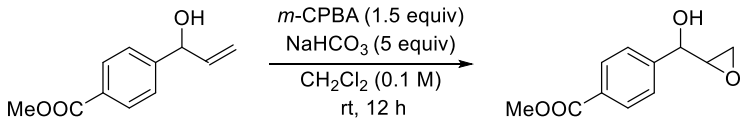
^{13}C NMR (126 MHz, $CDCl_3$) δ 198.7, 166.2, 141.0, 140.1, 133.9, 129.8, 128.6, 128.4, 127.9, 126.2, 52.4, 40.8, 30.0.

IR: 3410(s), 3132(s), 1766(w), 1621(w), 1401(s), 1243(m),1107(w),1055(w), 860(w), 757(w), 698(w), 624(w), 530(w).

HRMS: Calculation for $C_{17}H_{16}O$, $[M+Na^+]^+$: 291.0991, Found: 291.0990.



5 methyl 4-(hydroxy(oxiran-2-yl)methyl)benzoate



3q **5**

1) Add 25.9 mg 3-chloroperoxybenzoic acid (0.15 mmol), 19.2 mg **3q** (0.25 mmol), 42 mg $NaHCO_3$ (0.5 mmol, 1 mol%), to 25 ml round bottom flask.

2) Add 1mL CH_2Cl_2 to the flask.

3) Stir the mixture for 12 h at room temperature.

4) Quench the reaction by saturated $NaHCO_3$ solution and extract with CH_2Cl_2.

8) Concentrated the residue under reduced pressure.

5) Separate the residue by column chromatography with PE:EA= 1:1 (R_f = 0.5).

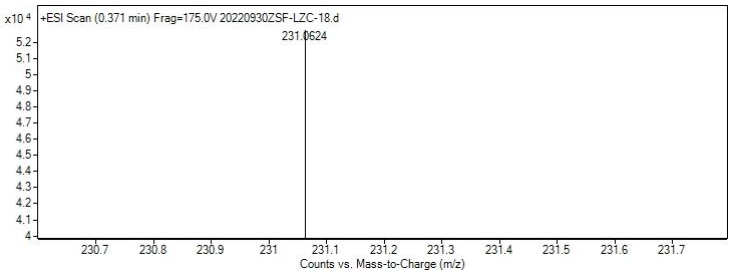
After the above procedures, 18.8 mg **5** was obtained as colorless liquid, yield= 90%, 1:1 dr.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05 (dd, J = 8.4, 2.2 Hz, 2H), 7.49 (t, J = 8.6 Hz, 2H), 4.99 (d, J = 3.1 Hz, 1H), 4.56 (d, J = 5.3 Hz, 1H), 3.92 (s, 3H), 3.28 – 3.18 (m, 1H), 2.91 – 2.83 (m, 1H).

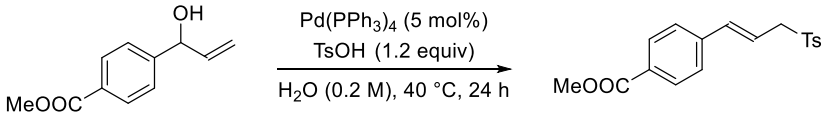
^{13}C NMR (126 MHz, $CDCl_3$) δ 166.8, 145.0, 144.5, 130.0, 130.0, 130.0, 129.9, 126.2, 126.2, 73.9, 70.4, 55.7, 54.8, 52.2, 45.4, 43.5.

IR: 3410(s), 3132(s), 1766(m), 1621(m), 1401(s), 1243(m),1107(w), 1055(w), 860(w), 757(w), 698(w), 624(w), 530(w).

HRMS: Calculation for $C_{11}H_{12}O$, $[M+Na^+]^+$: 231.0628, Found: 231.0624.



6 methyl (E)-4-(3-tosylprop-1-en-1-yl)benzoate



3q **6**

1) Add 24.6 mg *p*-toluenesulfonic acid (0.12 mmol), 19.2 mg **3q** (0.2 mmol) and 5.8 mg $Pd(PPh_3)_4$ (0.005 mmol, 5 mol%) to 25 ml Schlenk tube.

2) Evacuate the Schlenk tube with oil pump and refill the tube with N_2, repeat it 3 times.

3) Add 1 mL deionized water to the Schlenk tube.

4) Stir the mixture for 12 h at 40 ℃.

5) Extract the mixture with EA.

6) Wash the combined organic layer with brine, dry over Na_2SO_4.

7) Concentrated the residue under reduced pressure.

8) Separate the residue by column chromatography with PE:EA= 3:1 (R_f = 0.3).

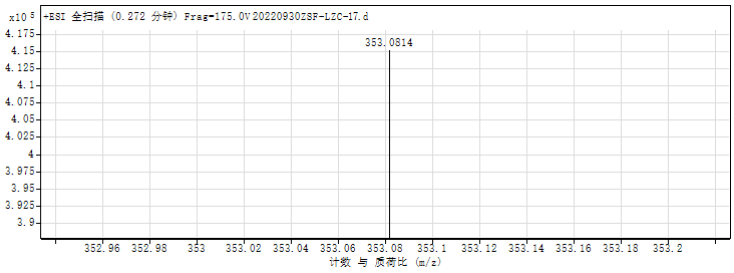
After the above procedures, 26.2 mg **6** was obtained as colorless liquid, yield= 79%.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.1 Hz, 2H), 7.75 (d, *J* = 8.0 Hz, 2H), 7.34 (t, *J* = 8.7 Hz, 4H), 6.44 (d, *J* = 15.9 Hz, 1H), 6.21 (dt, *J* = 15.5, 7.6 Hz, 1H), 3.95 (d, *J* = 7.6 Hz, 2H), 3.91 (s, 3H), 2.43 (s, 3H).

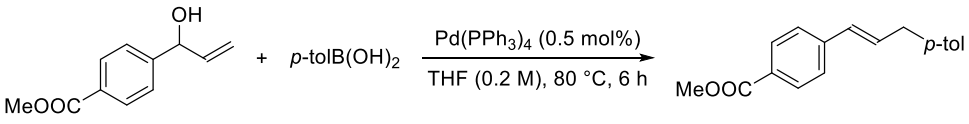
^{13}C NMR (126 MHz, $CDCl_3$) δ 166.6, 144.9, 140.1, 138.0, 135.6, 130.0, 129.9, 129.9, 129.8, 128.5, 126.5, 118.1, 60.5, 52.1, 21.6.

IR: 3413(s), 3131(s), 1765(w), 1621(w),1401(s), 1243(w), 1054(w),914(w), 744(w), 531(w).

HRMS: Calculation for $C_{18}H_{18}O_4S$, $[M+Na^+]^+$: 353.0818, Found: 353.0814.



7 methyl (E)-4-(3-(p-tolyl)prop-1-en-1-yl)benzoate



3q **7**

1) Add 32.6 mg 4-tolylboronic acid (0.24 mmol), 38.4 mg **3q** (0.2 mmol) and 1.15 mg $Pd(PPh_3)_4$ (0.001 mmol, 0.5 mol%) to 25 ml Schlenk tube.

2) Evacuate the Schlenk tube with oil pump and refill the tube with N_2, repeat it 3 times.

3) Add 1 mL anhydrous THF to the Schlenk tube.

4) Stir the mixture for 6 h at 80 ℃.

5) Filtrate the mixture by thin layer of silica gel.

6) Concentrated the residue under reduced pressure.

7) Separate the residue by column chromatography with PE:EA= 40:1 (R_f = 0.4).

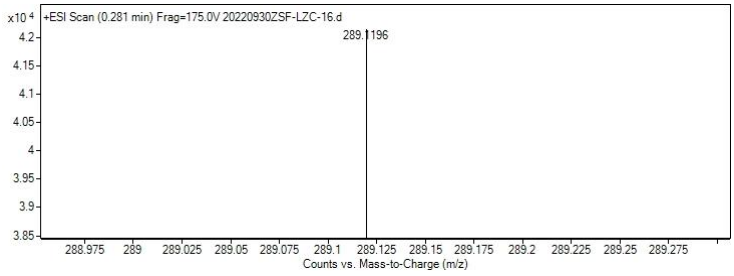
After the above procedures, 44.3 mg **7** was obtained as colorless liquid, yield= 83%.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.1 Hz, 2H), 7.38 (d, *J* = 8.1 Hz, 2H), 7.12 (s, 4H), 6.45 (d, *J* = 2.9 Hz, 2H), 3.88 (s, 3H), 3.52 (d, *J* = 4.7 Hz, 2H), 2.32 (s, 3H).

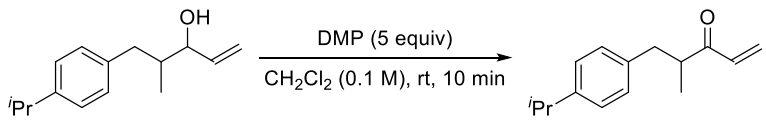
^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 142.1, 136.5, 135.9, 132.6, 130.0, 129.9, 129.3, 128.6, 128.5, 126.0, 52.0, 39.0, 21.1.

IR: 3417(s), 3129(s), 1637(m), 1621(m 1401(s), 1277(w),1177(w), 1104(w), 966(w), 762(w), 529(w).

HRMS: Calculation for $C_{18}H_{18}O_2$, $[M+Na^+]^+$: 289.1199, Found: 289.1196.



8 5-(4-isopropylphenyl)-4-methylpent-1-en-3-one



3ca **8ca**

1) Add 12.5 mg Dess-Martin periodinane (0.3 mmol), 43.6 mg **3ca** (0.2 mmol) to 25 ml round bottom flask.

2) Add 2 mL anhydrous CH_2Cl_2 to the flask.

3) Stir the mixture for 10 min at room temperature.

4) Quench the reaction by saturated solution of NaS_2O_3 and $NaHCO_3$ (1:1) and extract with CH_2Cl_2.

5) Wash the combined organic layer with brine, dry over Na_2SO_4.

6) Concentrated the residue under reduced pressure.

7) Separate the residue by column chromatography with PE:EA= 40:1 (R_f = 0.5).

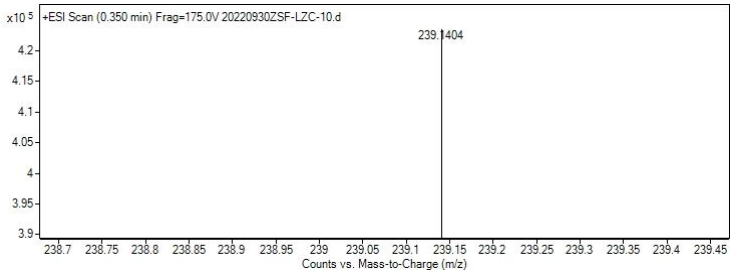
After the above procedures, 35.1 mg **8** was obtained as light-yellow liquid, yield= 81%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (d, *J* = 7.8 Hz, 2H), 7.11 (d, *J* = 7.7 Hz, 2H), 6.43 (dd, *J* = 17.5, 10.5 Hz, 1H), 6.31 – 6.17 (m, 1H), 5.83 – 5.71 (m, 1H), 3.13 (q, *J* = 6.9 Hz, 1H), 3.04 (dd, *J* = 13.6, 6.3 Hz, 1H), 2.94 – 2.85 (m, 1H), 2.59 (dd, *J* = 13.6, 7.8 Hz, 1H), 1.26 (d, *J* = 6.9 Hz, 6H), 1.13 (d, *J* = 6.9 Hz, 3H).

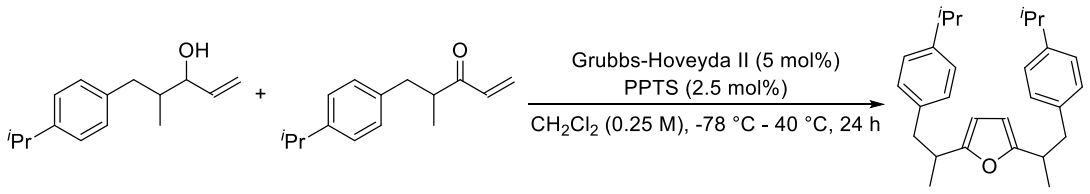
^{13}C NMR (101 MHz, $CDCl_3$) δ 203.6, 146.8, 137.0, 135.3, 128.9, 128.2, 126.4, 45.3, 38.5, 33.7, 24.0, 16.5.

IR: 3414(s), 3132(s), 2964(m), 1677(w), 1616(s), 1513(w),1401(s), 1052(w), 970(w), 816(w), 515(w).

HRMS: Calculation for $C_{15}H_{20}O$, $[M+Na^+]^+$: 239.1406, Found: 239.1404.



9 2,5-bis(1-(4-isopropylphenyl)propan-2-yl)furan



3ca **8ca** **9**

1) Add 1.3 mg pyridinium *p*-toluenesulfonate (PPTS) (0.005 mmol, 2.5 mol%) and 6.3 mg Grubbs-Hoveyda (0.01 mmol, 5 mol%) to 25 ml Schlenk tube.

2) Evacuate the Schlenk tube with oil pump and refill the tube with N_2, repeat it 3 times.

3) Add 1 mL anhydrous CH_2Cl_2 to the Schlenk tube.

4) Cool the solution to -78 ℃.

5) Add 43.6 mg **3ca** (0.2 mmol) and 95 mg **8ca** (1 mmol, 5 equiv) to the solution in dropwise.

6) Stir the mixture for 24 h at 40 ℃.

7) Filtrate the mixture by thin layer of silica gel.

8) Concentrated the residue under reduced pressure.

9) Separate the residue by column chromatography with PE:EA= 40:1 (R_f = 0.4).

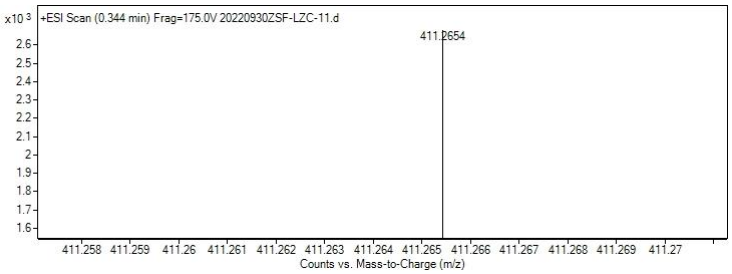
After the above procedures, 52.0 mg **9** was obtained as light-yellow liquid, yield= 67%.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.03 (d, *J* = 7.7 Hz, 4H), 6.94 (d, *J* = 7.7 Hz, 4H), 5.73 (s, 2H), 2.95 (dq, *J* = 15.3, 7.0 Hz, 4H), 2.78 (p, *J* = 7.0 Hz, 2H), 2.62 – 2.51 (m, 2H), 1.15 (d, *J* = 7.1 Hz, 12H), 1.09 (d, *J* = 6.4 Hz, 6H).

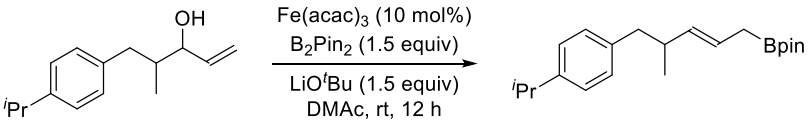
^{13}C NMR (126 MHz, $CDCl_3$) δ 158.2, 158.2, 146.4, 137.6, 129.2, 126.2, 104.1, 104.0, 41.9, 41.8, 35.0, 33.7, 24.1, 18.4, 18.3.

IR: 3414(s), 3131(s), 1765(w), 1621(w), 1400(s), 1243(w), 1054(w), 914(w), 744(w), 531(w).

HRMS: Calculation for $C_{28}H_{36}O$, $[M+Na^+]^+$: 411.2658, Found: 411.2654.



10 (E)-2-(5-(4-isopropylphenyl)-4-methylpent-2-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane



3ca **10**

1) Add 7.0 mg ferric acetylacetonate (0.02 mmol, 10 mol%), 76.2 mg bis(pinacolato)diborane (0.3 mmol, 1.5 equiv), 24.0 mg lithium tert-butoxide (0.3 mmol, 1.5 equiv) and 43.6 mg **3ca** (0.2 mmol) to 25 ml Schlenk tube.

2) Evacuate the Schlenk tube with oil pump and refill the tube with N_2, repeat it 3 times.

3) Add 1.0 mL DMAc to the Schlenk tube.

4) Stir the mixture for 12 h at room temperature.

5) Filtrate the mixture by thin layer of silica gel.

6) Concentrated the residue under reduced pressure.

7) Separate the residue by column chromatography with PE:EA= 60:1 (R_f = 0.4).

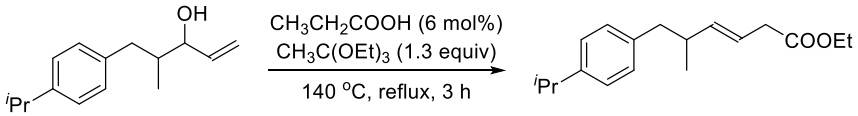
After the above procedures, 46.0 mg **12** was obtained as colorless liquid, yield= 70%.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.11 (d, J = 8.3 Hz, 2H), 7.06 (d, J = 7.8 Hz, 2H), 5.52 – 5.29 (m, 2H), 2.86 (p, J = 6.8 Hz, 1H), 2.64 (h, J = 6.3, 5.3 Hz, 1H), 2.49 – 2.32 (m, 2H), 1.62 (d, J = 5.8 Hz, 2H), 1.24 (d, J = 3.5 Hz, 18H), 0.93 (d, J = 6.2 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 146.0, 138.4, 136.4, 129.2, 126.0, 123.1, 83.1, 43.4, 38.2, 33.7, 24.8, 24.7, 24.1, 24.1, 20.0.

Characterization data matched those reported in the literature.[2]

11 ethyl (E)-6-(4-isopropylphenyl)-5-methylhex-3-enoate



3ca **11**

1) Evacuate the Schlenk tube with oil pump and refill the tube with N_2, repeat it 3 times.

2) Add 43.6 mg **3ca**, triethyl orthoacetate (0.26 mmol, 1.3 equiv) and 0.9 μL propionic acid (0.012 mmol, 6 mol%) to 25 ml Schlenk tube.

3) Reflux the mixture for 3 h at 140 ℃.

4) Concentrated the residue under reduced pressure.

5) Separate the residue by column chromatography with PE:EA= 100:1 (R_f = 0.5)

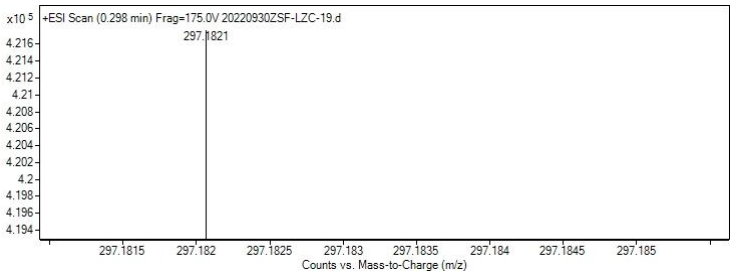
After the above procedures, 43.5 mg **11** was obtained as colorless liquid, yield= 75%.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (d, *J* = 7.8 Hz, 2H), 7.08 (d, *J* = 7.8 Hz, 2H), 5.54 – 5.21 (m, 2H), 4.16 (q, *J* = 7.1 Hz, 1H), 2.91 (p, *J* = 6.9 Hz, 1H), 2.64 (dd, *J* = 13.1, 6.5 Hz, 1H), 2.45 (ddd, *J* = 27.5, 13.4, 7.3 Hz, 5H), 1.28 (d, *J* = 6.9 Hz, 9H), 0.98 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 173.3, 146.2, 138.1, 137.0, 129.2, 126.4, 126.1, 60.2, 43.2, 38.3, 34.4, 33.7, 28.0, 24.1, 19.9, 14.3.

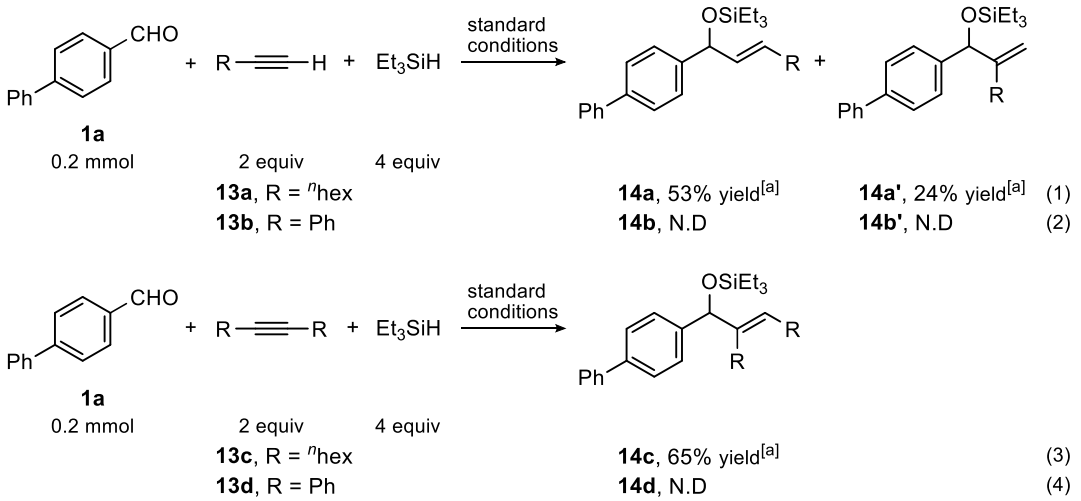
IR: 3413(s), 3126(s), 1741(w), 1620(w), 1400(s), 1242(w), 1178(w), 1054(w), 915(w), 744(w), 530(w).

HRMS: Calculation for $C_{18}H_{26}O_2$, $[M+Na^+]^+$: 297.1825, Found: 297.1821.



Comparative Studies of Substituted Alkynes Versus Acetylene

Ni-catalyzed Alkenylation of Different Alkynes with Aldehydes.



All the following operations were carried out in parallel in four separate flasks respectively.

1) 11.0 mg $Ni(cod)_2$ (0.04 mmol, 20 mol%), 34.0 mg IPr·HCl (0.08 mmol, 40 mol%) were weighed out and transferred into a Schlenk tube in the glove box.

2) The tube was then sealed with a hollow glass plug and taken out of the glovebox.

3) Putting the tube under nitrogen via 3 vacuum / N_2 backfill cycles and unplugged the hollow glass plug.

4) Add 1.0 mL solvent and KO^{t}Bu 88 μL (0.088 mmol, 44 mol%,1 mol/L in THF).

5) Stir the solution at room temperature until all the solid dissolved (2 min).

6) Add 93 mg (128 μL, ρ= 0.728 g/mL at 25 ℃) triethylsilane (0.8 mmol, 4 equiv) via syringe after the same operation of step 3.

7) Stir the solution at room temperature for 2 min.

8) Add aldehyde (0.2 mmol) and alkyne (0.4 mmol) dissolved in 800 μL THF.

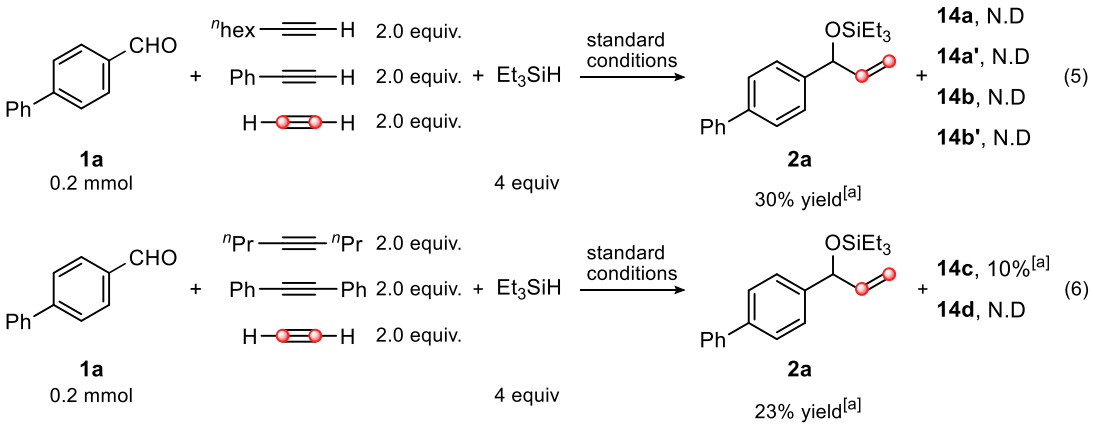
9) Stir the solution at 35 ℃, monitored by TLC.

10) After consumption of aldehyde, filtrate the mixture by a short pad of silica gel and wash with EA and was then concentrated under reduced pressure.

11) Add the 4-nitrotoluene as internal for calculation of ^{1}H NMR yield.

The results were listed above or in Scheme 4a in the main text.

Competition of Ni-Catalyzed Alkenylation among Different Alkynes.



All the following operations were carried out parallelly in four flasks respectively.

1) 11.0 mg $Ni(cod)_2$ (0.04 mmol, 20 mol%), 34.0 mg IPr·HCl (0.08 mmol, 40 mol%) were weighed out and transferred into a Schlenk tube in the glove box.

2) The tube was then sealed with a hollow glass plug and taken out of the glovebox.

3) Putting the tube under nitrogen via 3 vacuum / N_2 backfill cycles and unplugged the hollow glass plug.

4) Add 1.0 mL solvent and KOtBu 88 μL (0.088 mmol, 44 mol%,1 mol/L in THF).

5) Stir the solution at room temperature until all the solid dissolved (2 min).

6) Add 93 mg (128 μL, ρ= 0.728g/mL at 25 ℃) triethylsilane (0.8 mmol, 4 equiv) via syringe after the same operation of step 3.

7) Stir the solution at room temperature for 2 min.

8) Add the solution of substituted alkynes (0.4 mmol) and 36.4 mg 4-biphenylcarboxaldehyde (0.2 mmol) dissolved in 800μL saturated solution of acetylene in THF.

9) Stir the solution at 35 ℃, monitored by TLC.

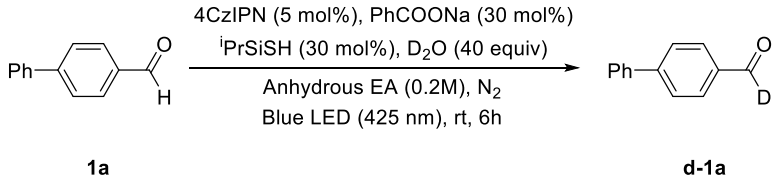
10) After completion of reaction, filtrate the mixture by a short pad of silica gel and wash with EA and was then concentrated under reduced pressure.

11) Add the 4-nitrotoluene as internal for calculation of ^1H NMR yield.

The results were listed above or in Scheme 4b in the main text.

Mechanistic Studies of Acetylene

d-1a [1,1'-biphenyl]-4-carbaldehyde-d



The deuterated substrate **d-1a** was prepared according to reported procedure.[4]

1) 7.9 mg 2,4,5,6-tetrakis(carbazol-9-yl)-4,6-dicyanobenzene (4CzIPN) (0.01 mmol, 5 mol%), 8.6 mg sodium benzoate (0.06 mmol, 30 mol%) and 36.4 mg 4-biphenylcarboxaldehyde (0.2 mmol) were weighed out and transferred into a Schlenk tube .

2) Putting the tube under nitrogen via 3 vacuum / N_2 backfill cycles and unplugged the hollow glass plug.

3) Add 60μL triisopropylsilanethiol (0.06 mmol, 30 mol%), 160μL D_2O (8 mmol, 40 equiv) and 2 mL anhydrous EA.

4) Stir the solution under irradiation of blue LED (425 nm, 10 W) for 6 h.

5) Extract the mixture with EA.

6) Wash the combined organic layer with brine, dry over Na_2SO_4。

7) Concentrated the residue under reduced pressure.

8) Separate the residue by column chromatography with PE:EA = 40:1 (R_f = 0.4).

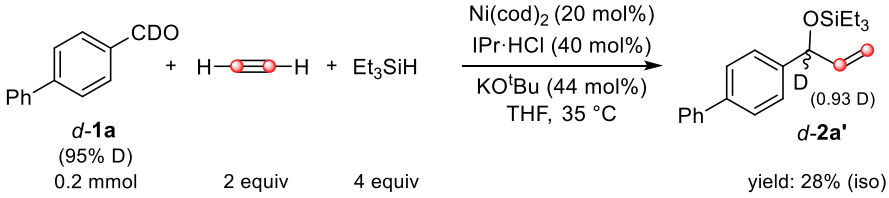
After the above procedures, 33.0 mg *d*-**1a** was obtained as yellow liquid, yield = 90%, The ratio of deuteration was determined by ^{1}H NMR, 95% - *D* ratio.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 7.9 Hz, 2H), 7.78 (d, *J* = 8.1 Hz, 2H), 7.72 – 7.59 (m, 2H), 7.48 (dt, *J* = 25.9, 7.4 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 191.9, 191.8, 191.6, 191.4, 147.2, 139.7, 135.2, 135.2, 135.1, 130.3, 129.0, 128.5, 127.7, 127.4.

Characterization data matched those reported in the literature.[4]

d-2a ((1-([1,1'-biphenyl]-4-yl)allyl-1-d)oxy)triethylsilane



1) 11.0 mg $Ni(cod)_2$ (0.04 mmol, 20 mol%), 34.0 mg IPr·HCl (0.08 mmol, 40 mol%) were weighed out and transferred into a Schlenk tube in the glove box.

2) The tube was then sealed with a hollow glass plug and taken out of the glovebox.

3) Putting the tube under nitrogen via 3 vacuum / N_2 backfill cycles and unplugged the hollow glass plug.

4) Add 1.0 mL solvent and KO^{t}Bu 88 μL (0.088 mmol, 44 mol%,1 mol/L in THF).

5) Stir the solution at room temperature until all the solid dissolved (2 min).

6) Add 93mg (128 μL, ρ =0.728 g/mL at 25 ℃) triethylsilane (0.8 mmol, 4 equiv) via syringe after the same operation of step 3.

7) Stir the solution at room temperature for 2 min.

8) Add [1,1'-biphenyl]-4-carbaldehyde-d, *d*-**1a**, (0.2 mmol) dissolved in 800 μL saturated solution of acetylene in THF (0.55 mol/L).

9) Stir the solution at 35 ℃, monitored by TLC.

10) After completion of reaction, filtrate the mixture by a short pad of silica gel and wash with EA and was then concentrated under reduced pressure.

11) Purify the crude via column chromatography with PE (R_f =0.4).

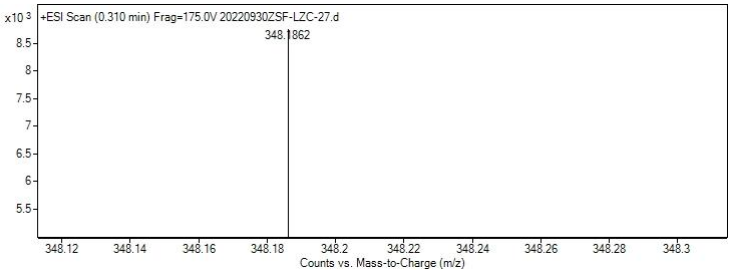
After the above procedures, 18.3mg *d*-**2a** was obtained as colorless liquid, yield= 28 %, The ratio of deuteration was determined by ^{1}H NMR, 93%-*D* ratio.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 – 7.56 (m, 4H), 7.50 – 7.42 (m, 4H), 7.39 – 7.33 (m, 1H), 6.01 (dd, *J* = 17.0, 10.2 Hz, 1H), 5.35 (dd, *J* = 17.1, 1.8 Hz, 1H), 5.14 (dd, *J* = 10.2, 1.7 Hz, 1H), 0.98 (t, *J* = 7.9 Hz, 9H), 0.66 (qd, *J* = 7.9, 1.9 Hz, 6H).

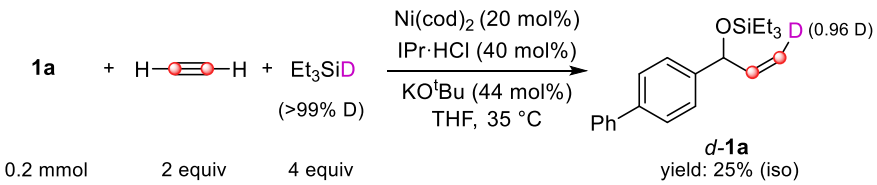
^{13}C NMR (101 MHz, $CDCl_3$) δ 142.8, 141.5, 141.0, 140.0, 128.7, 127.1, 127.1, 127.0, 126.4, 113.7, 6.8, 4.9.

IR: 3414(s), 3137(s), 1637(m), 1401(s), 1294(w), 1089(w), 1007(w), 846(w), 741(w), 695(w), 618(w), 514(w).

HRMS: Calculation for $C_{21}H_{27}DOSi$, $[M+Na^+]^+$: 348.1864, Found: 348.1862.



***d*-2a′ (Z)-((1-([1,1'-biphenyl]-4-yl)allyl-3-d)oxy)triethylsilane**



1) 11.0 mg $Ni(cod)_2$ (0.04 mmol, 20 mol%), 34.0 mg IPr·HCl (0.08 mmol, 40 mol%) were weighed out and transferred into a Schlenk tube in the glove box.

2) The tube was then sealed with a hollow glass plug and taken out of the glovebox.

3) Putting the tube under nitrogen via 3 vacuum / N_2 backfill cycles and unplugged the hollow glass plug.

4) Add 1.0 mL solvent and KO^{t}Bu 88 μL (0.088 mmol, 44 mol%,1 mol/L in THF).

5) Stir the solution at room temperature until all the solid dissolved (2 min).

6) Add 93 mg (128 μL, ρ= 0.737 g/mL at 25 ℃) triethylsilane-d (0.4 mmol, 4 equiv) via syringe after the same operation of step 3.

7) Stir the solution at room temperature for 2 min.

8) Add [1,1'-biphenyl]-4-carbaldehyde-d (0.2 mmol) dissolved in 800 μL saturated solution of acetylene in THF (0.55 mol/L).

9) Stir the solution at 35 ℃, monitored by TLC.

10) After completion of reaction, filtrate the mixture by a short pad of silica gel and wash with EA and was then concentrated under reduced pressure.

11) Purify the crude via column chromatography with PE (R_f = 0.4).

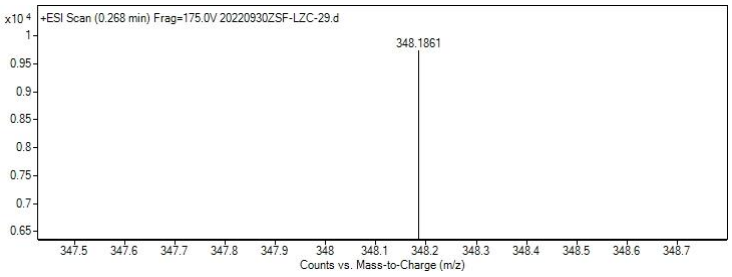
After the above procedures, 16.0 mg *d*-**2a′** was obtained as colorless liquid, yield= 25%, The ratio of deuteration was determined by ^{1}H NMR, 96%-*D* ratio.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 – 7.57 (m, 2H), 7.55 (d, *J* = 7.9 Hz, 2H), 7.45 – 7.39 (m, 4H), 7.32 (t, *J* = 7.4 Hz, 1H), 5.96 (ddt, *J* = 10.7, 5.5, 2.3 Hz, 1H), 5.21 (d, *J* = 6.0 Hz, 1H), 5.09 (dd, *J* = 10.1, 1.2 Hz, 1H), 0.94 (t, *J* = 7.9 Hz, 9H), 0.62 (qd, *J* = 7.9, 2.9 Hz, 6H).

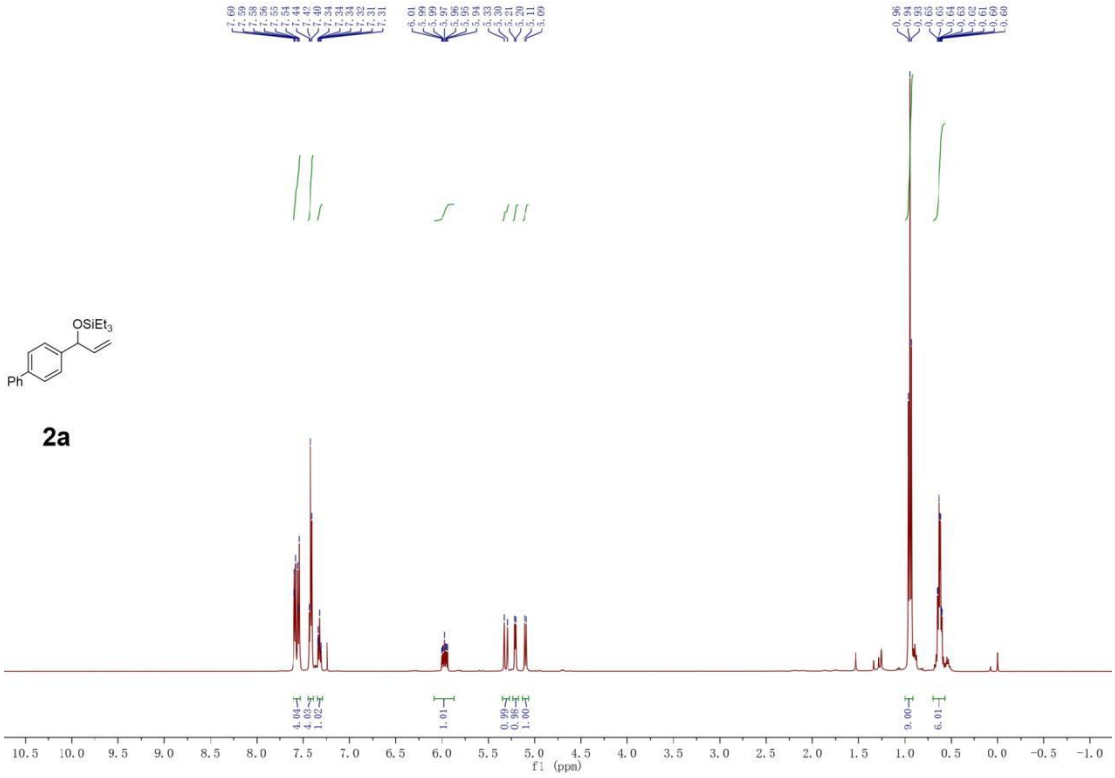
^{13}C NMR (126 MHz, $CDCl_3$) δ 142.9, 141.5, 141.0, 140.0, 128.7, 127.1, 127.0, 126.4, 113.6, 113.4, 113.2, 75.5, 6.8, 4.9.

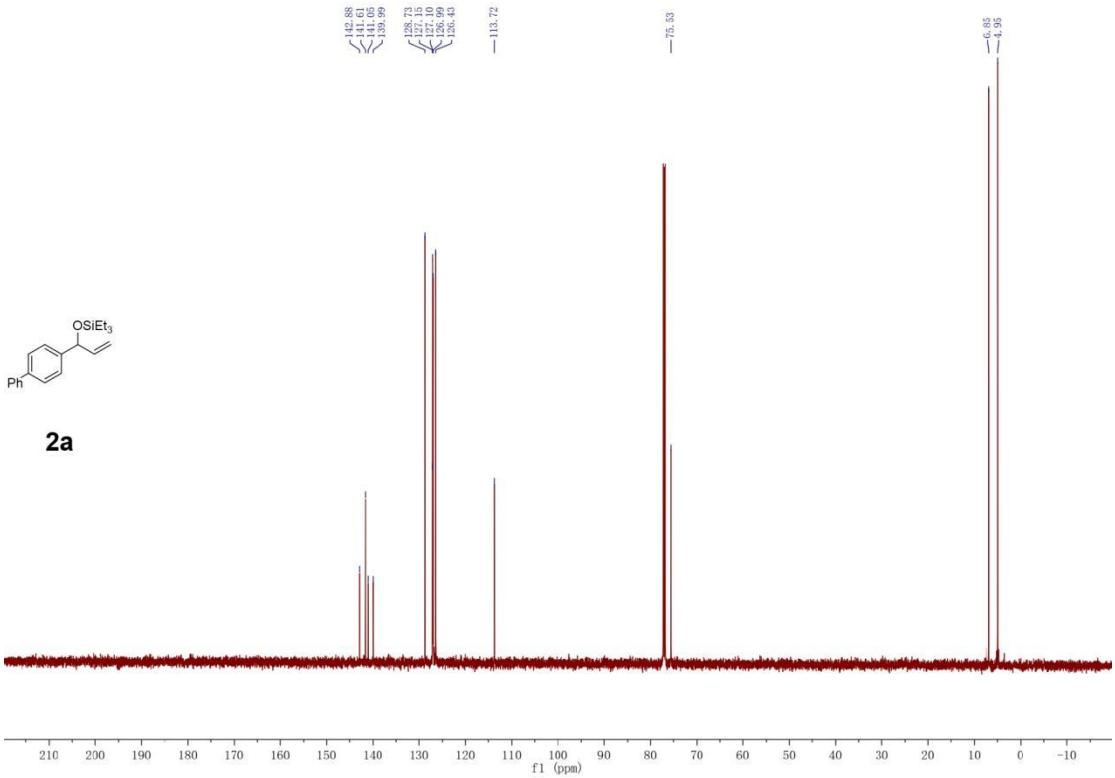
IR: 3417(s), 3135(s), 1621(m), 1401(s), 1067(w), 1007(w), 803(w), 737(w), 695(w), 621(w), 514(w).

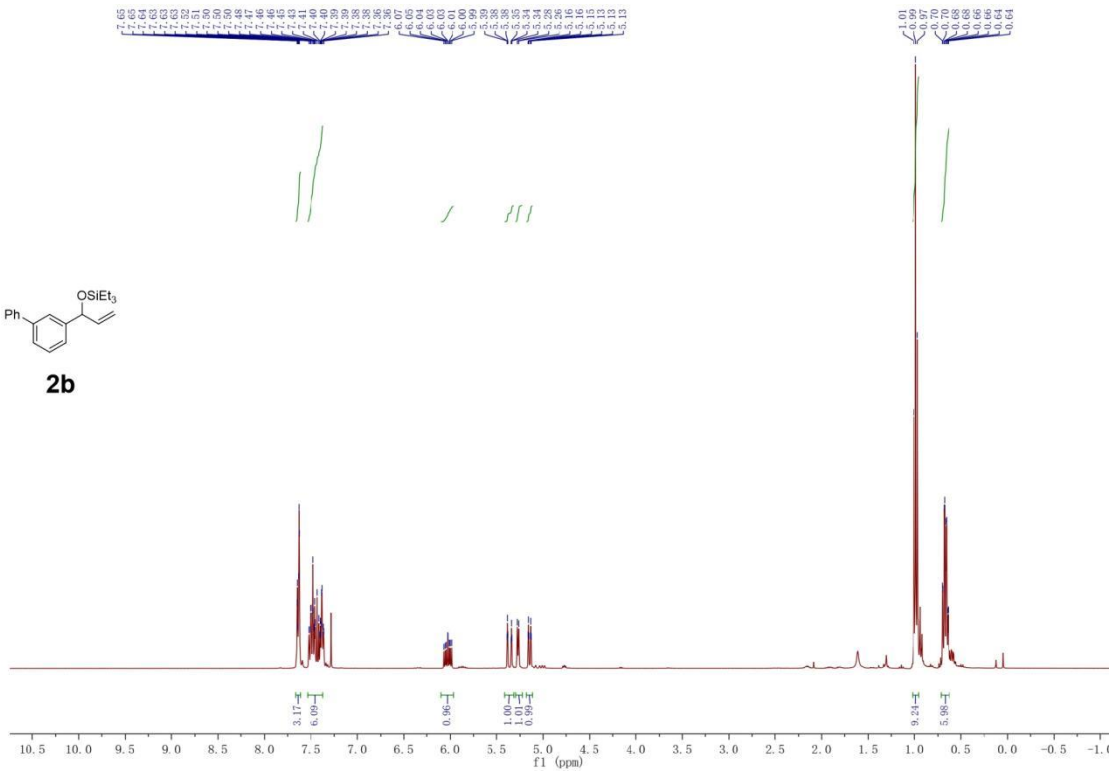
HRMS: Calculation for $C_{21}H_{27}DOSi$, $[M+Na^+]^+$:348.1864 , Found: 348.1861.



Original Spectrum



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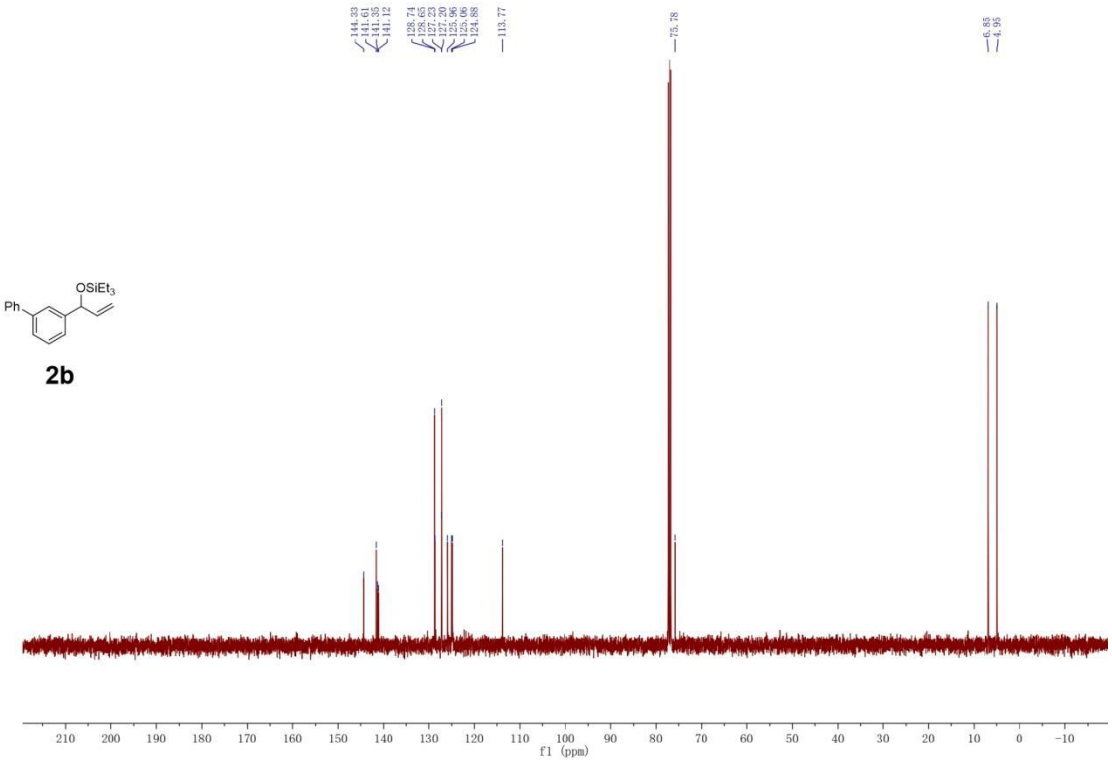
f1 (ppm)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Ph
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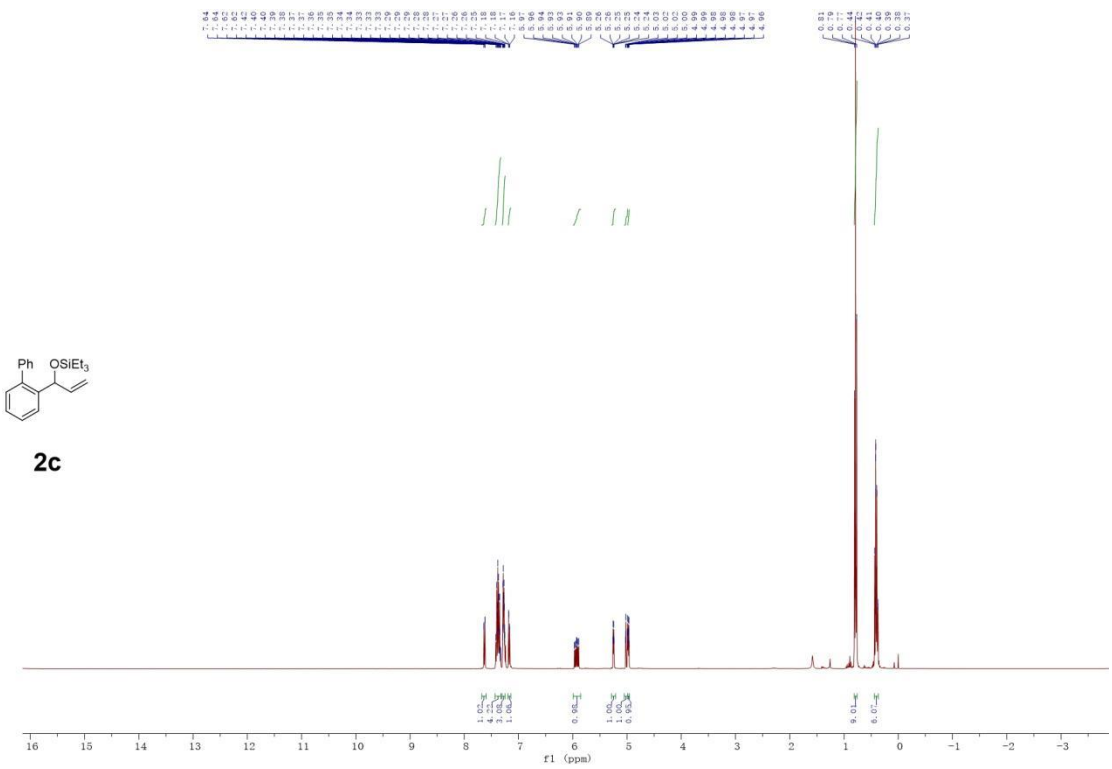
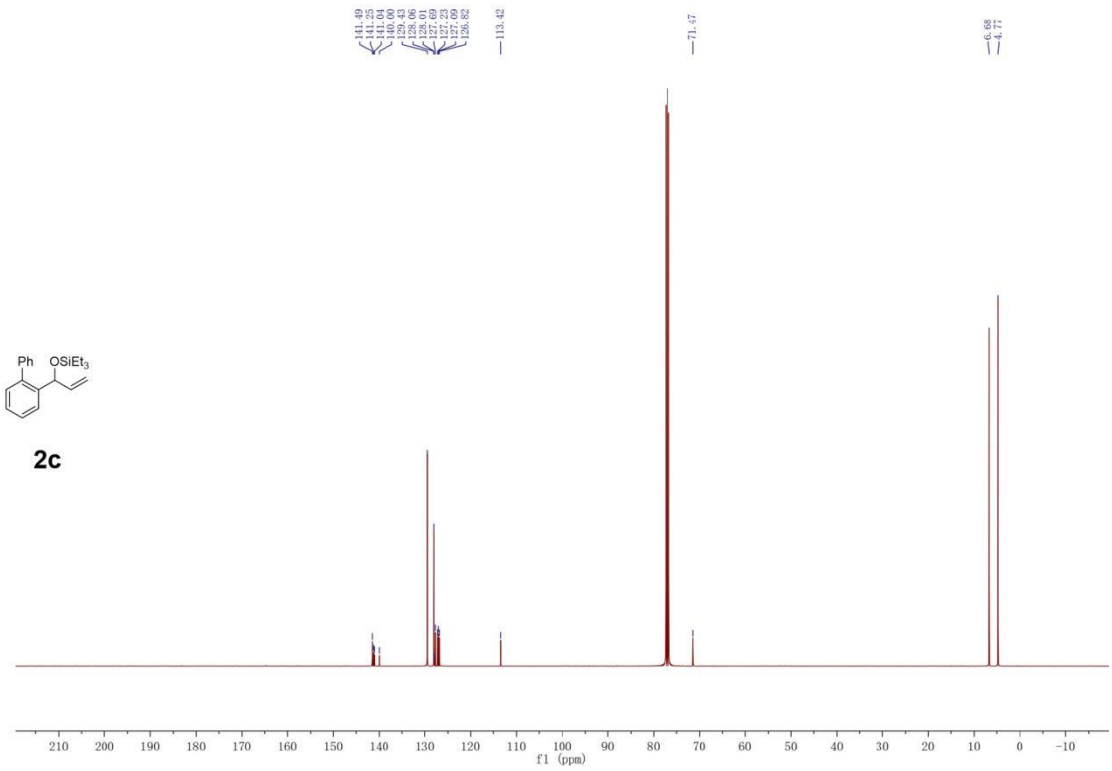
f1 (ppm)


Ph
OSiEt3
2c
f1 (ppm)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141.25
141.04
140.99
129.43
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128.01
127.69
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127.09
126.82
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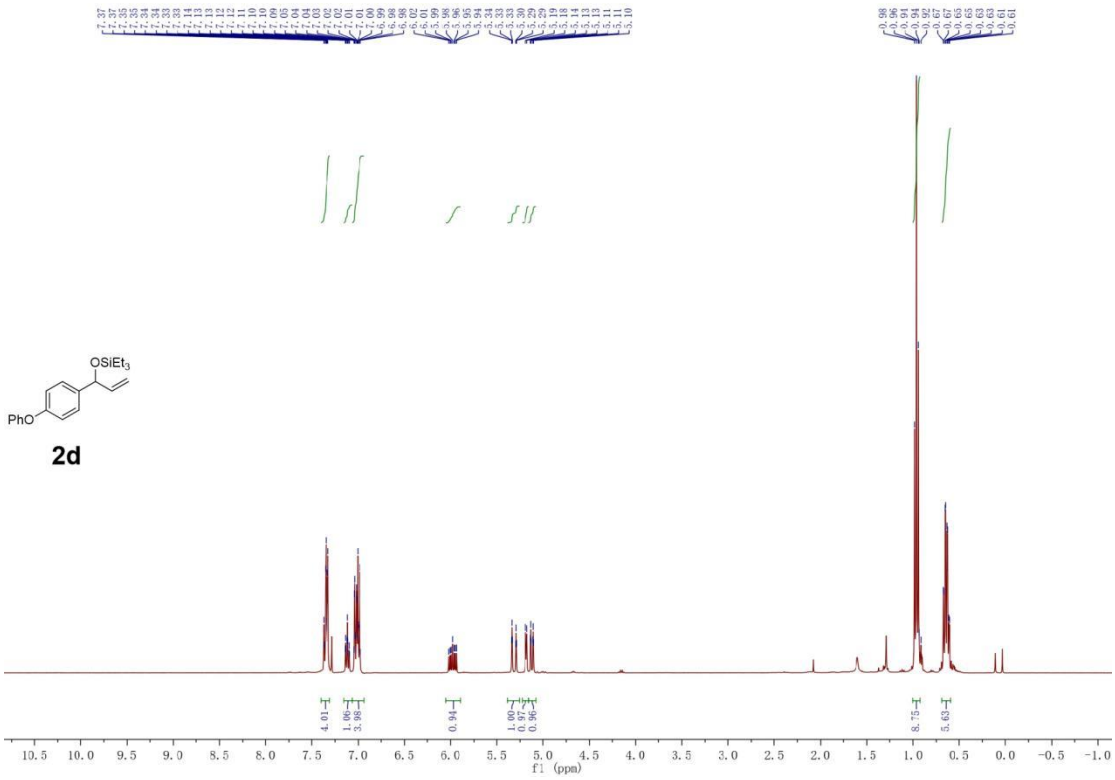
6.68
4.71
Ph
OSiEt3
2c
f1 (ppm)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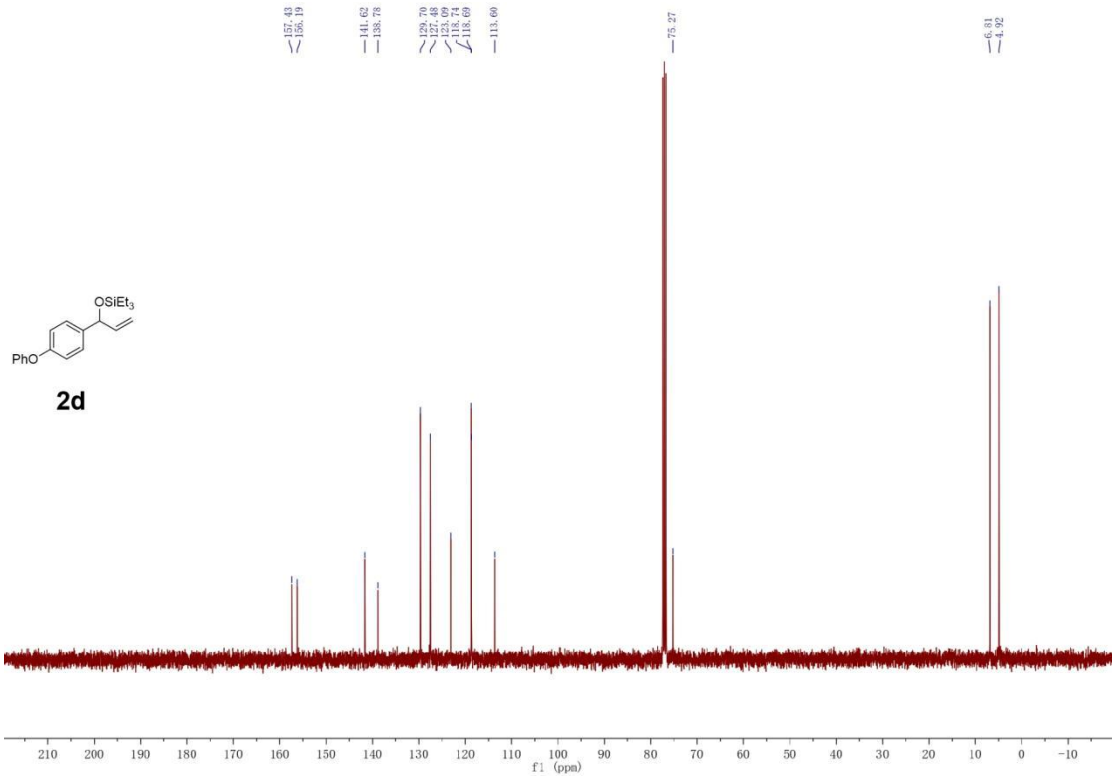
PhO
2d
f1 (ppm)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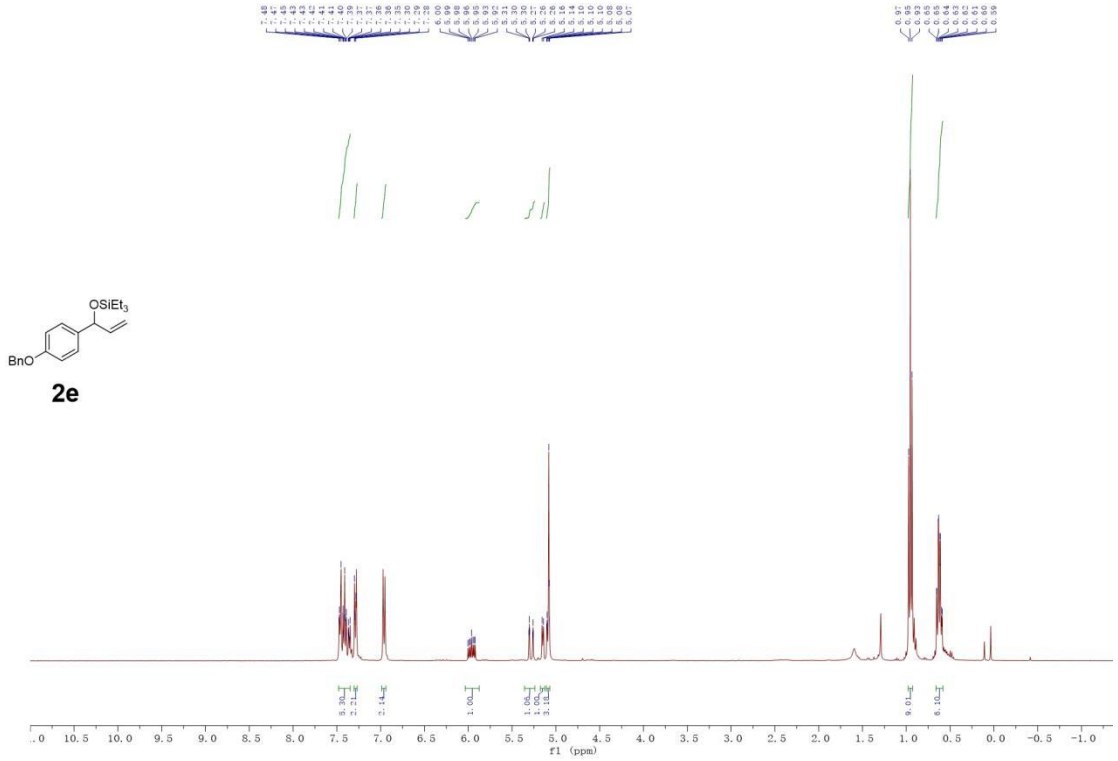
PhO
2d
f1 (ppm)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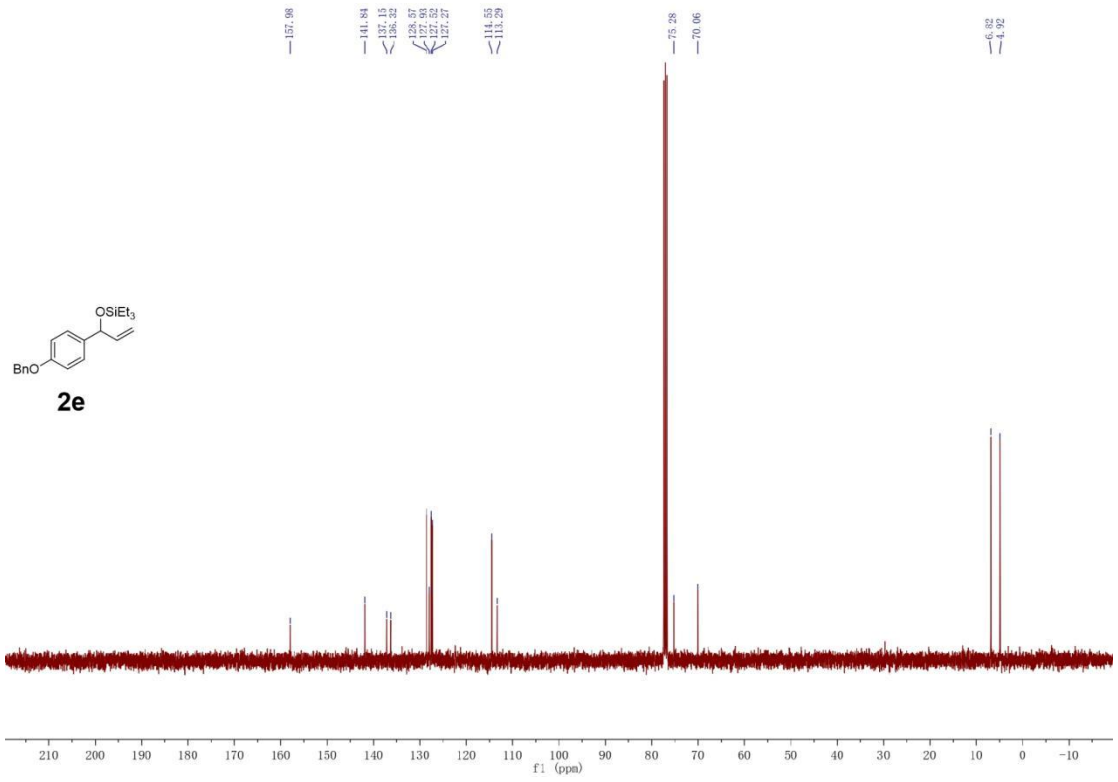
BnO
2e
f1 (ppm)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127.27
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113.29
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4.92
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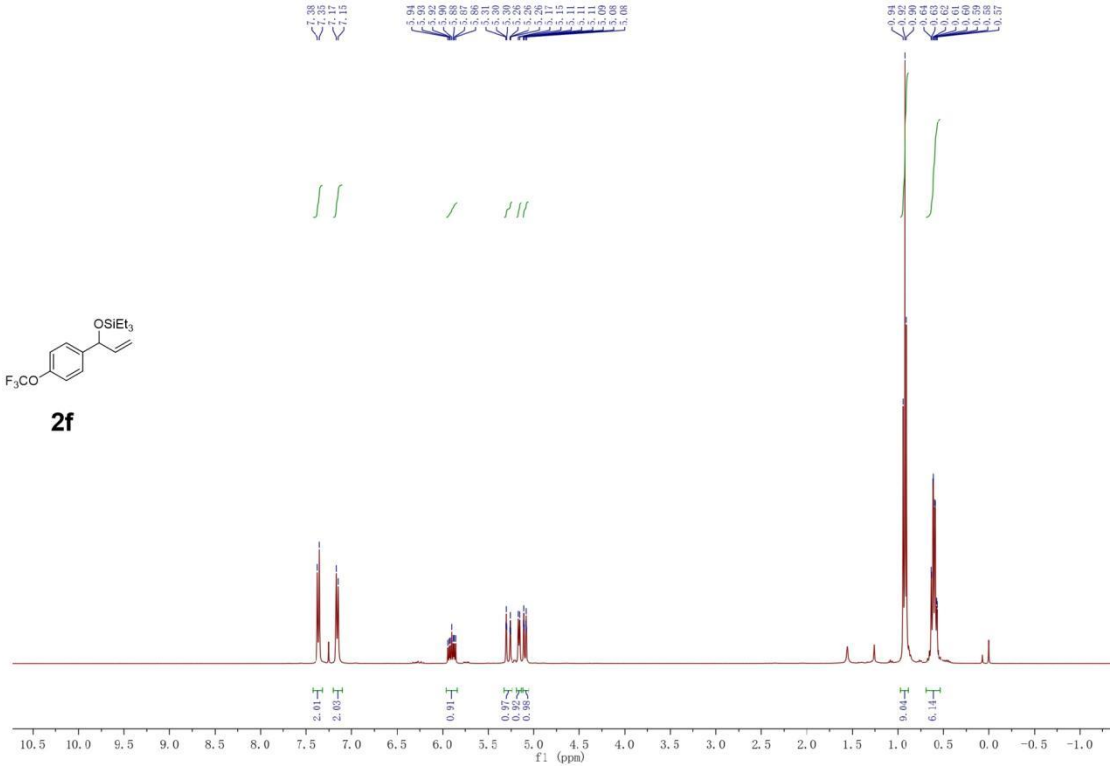
BnO
2e
f1 (ppm)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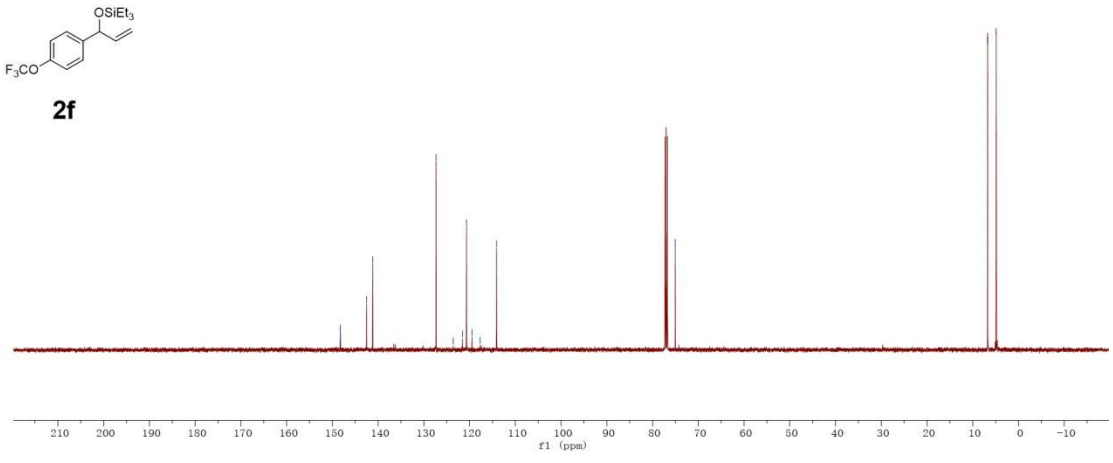
2f
f1 (ppm)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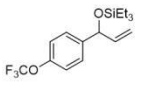
F3CO
2f
f1 (ppm)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F3CO

2f

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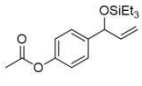
f1 (ppm)


OSiEt3

2g

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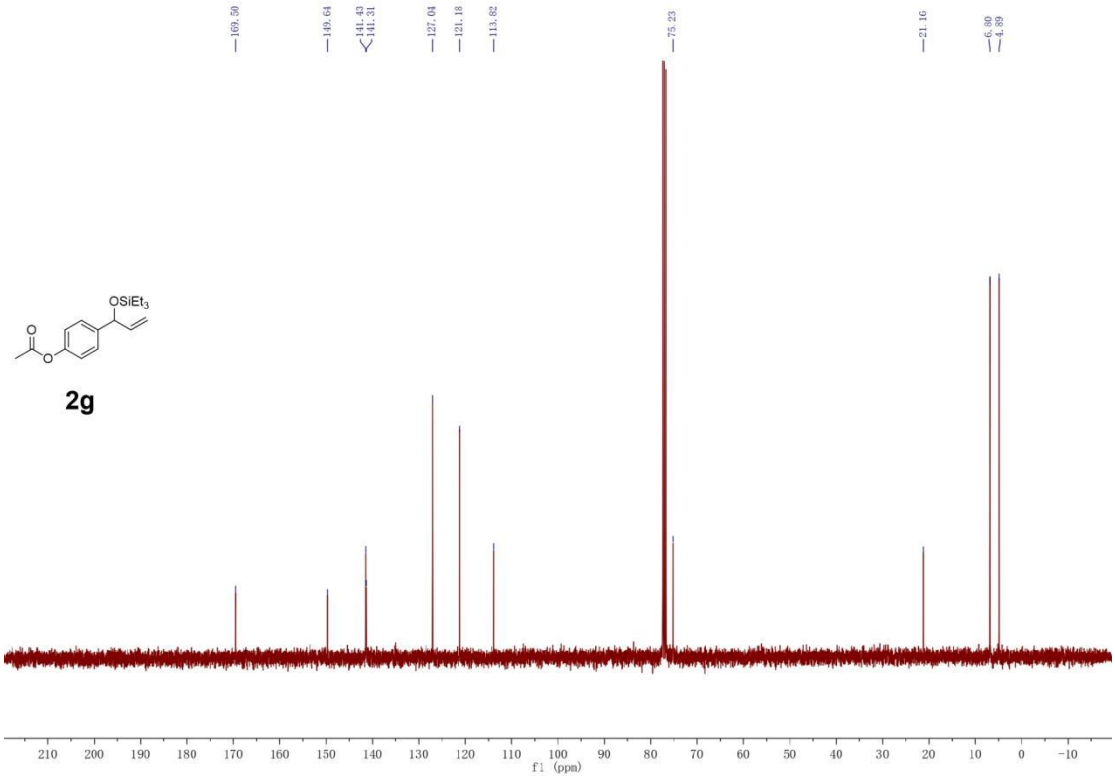
1.00
0.97
1.04
3.19
9.34
6.15
f1 (ppm)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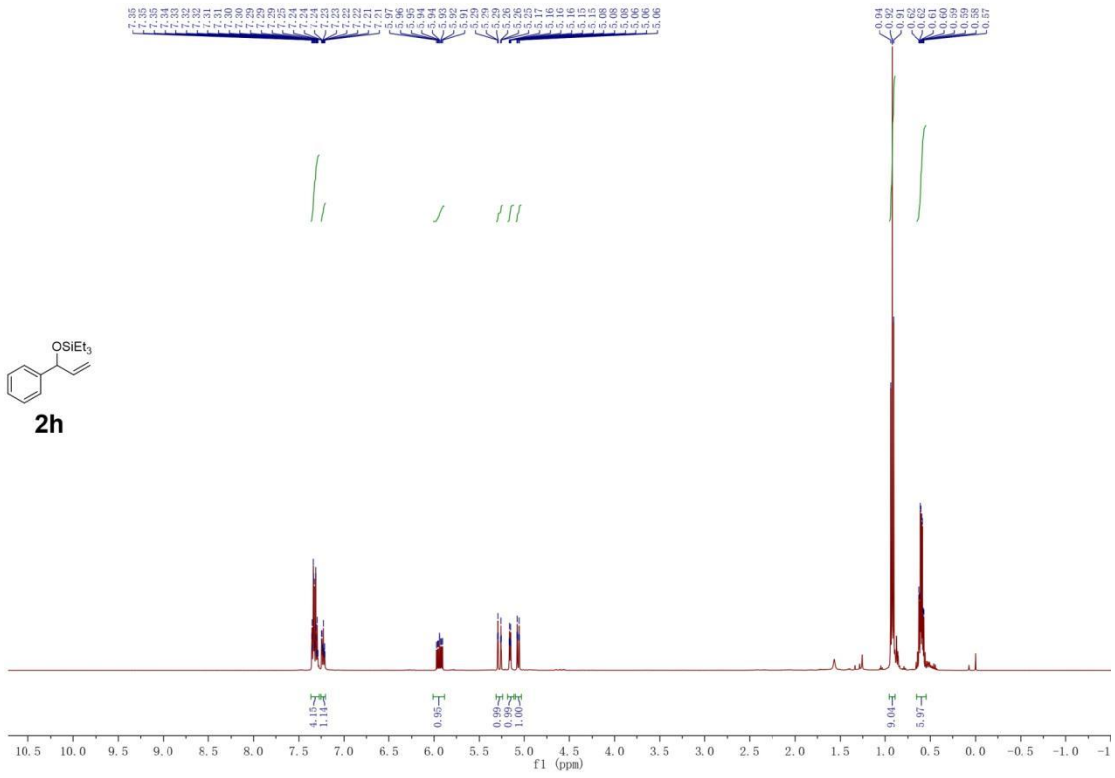
21.16
6.90
4.89
OSiEt3
2g
f1 (ppm)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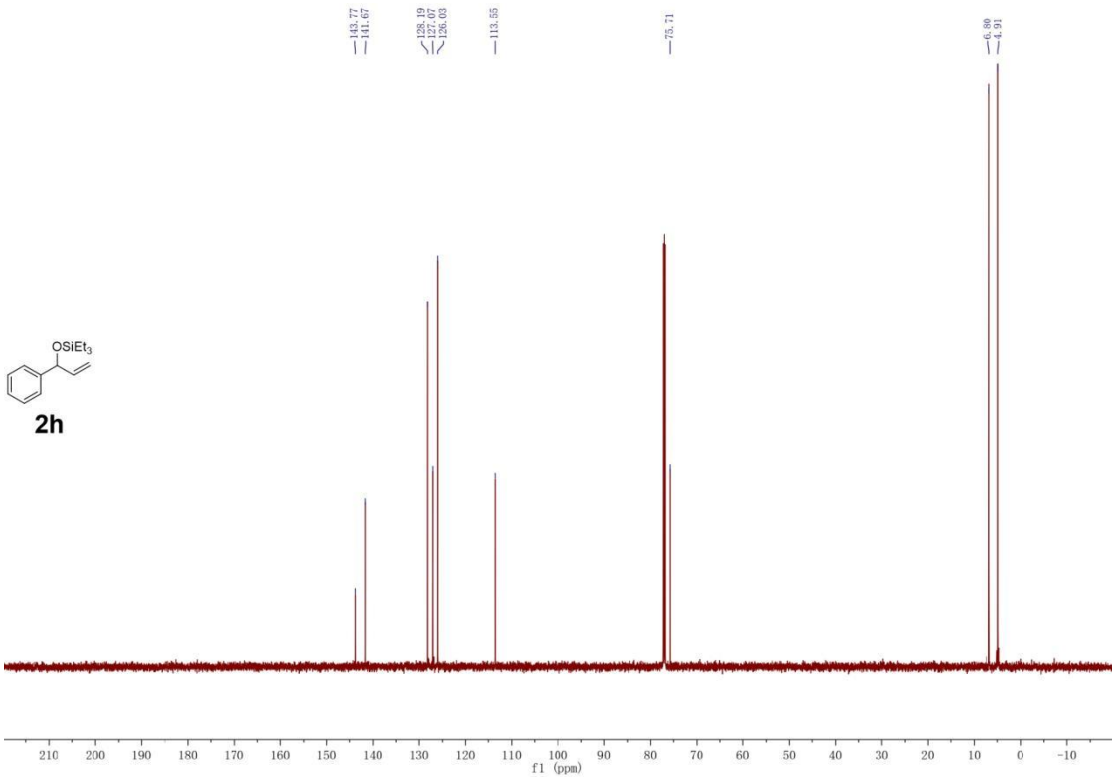
2h
f1 (ppm)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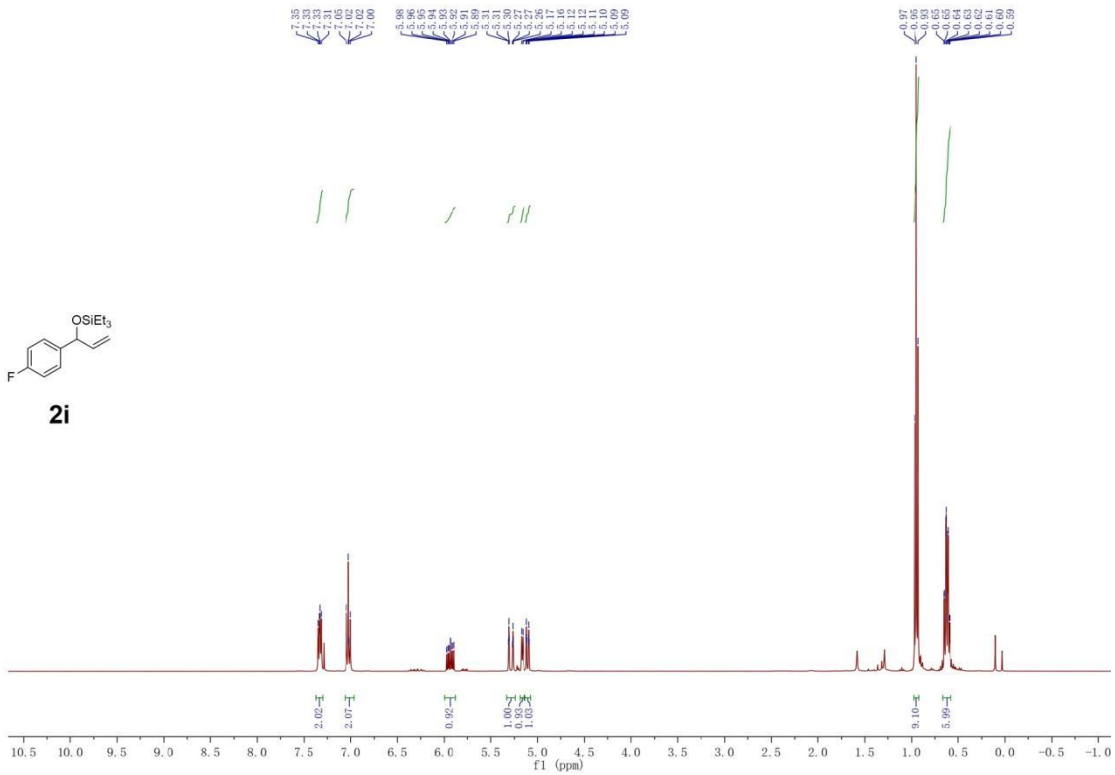
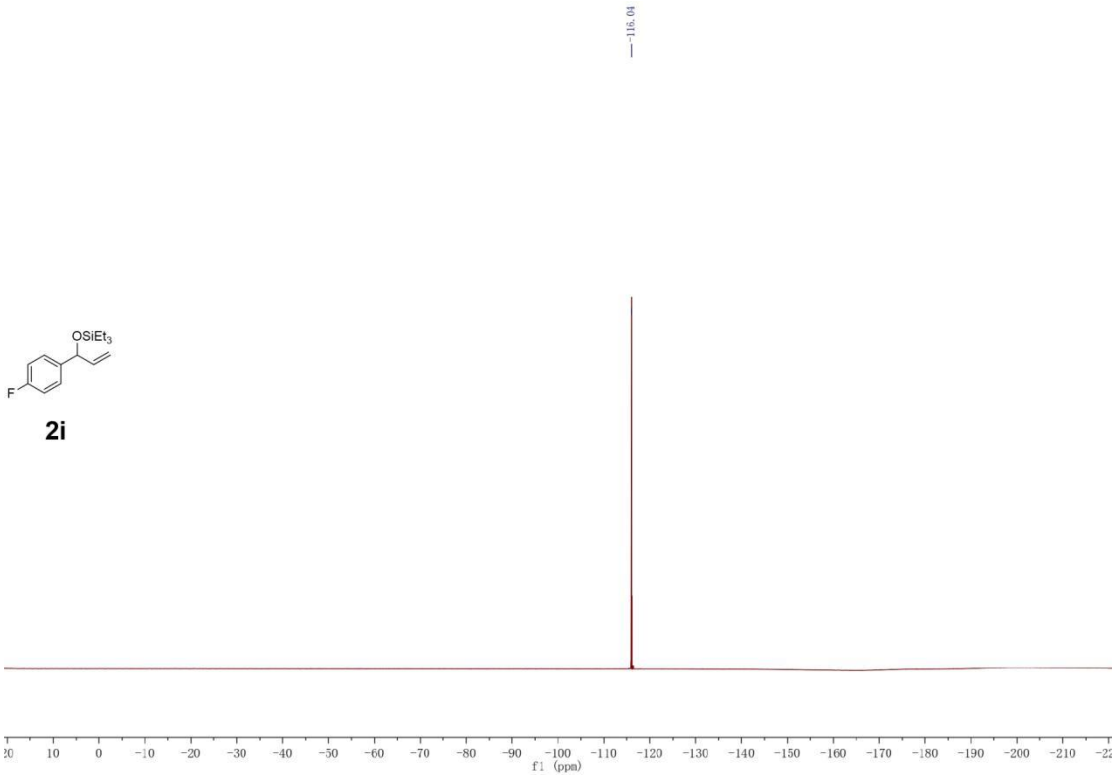
113.55
75.71
6.89
4.91
OSiEt3
2h
f1 (ppm)


OSiEt3
F
2i
f1 (ppm)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2i
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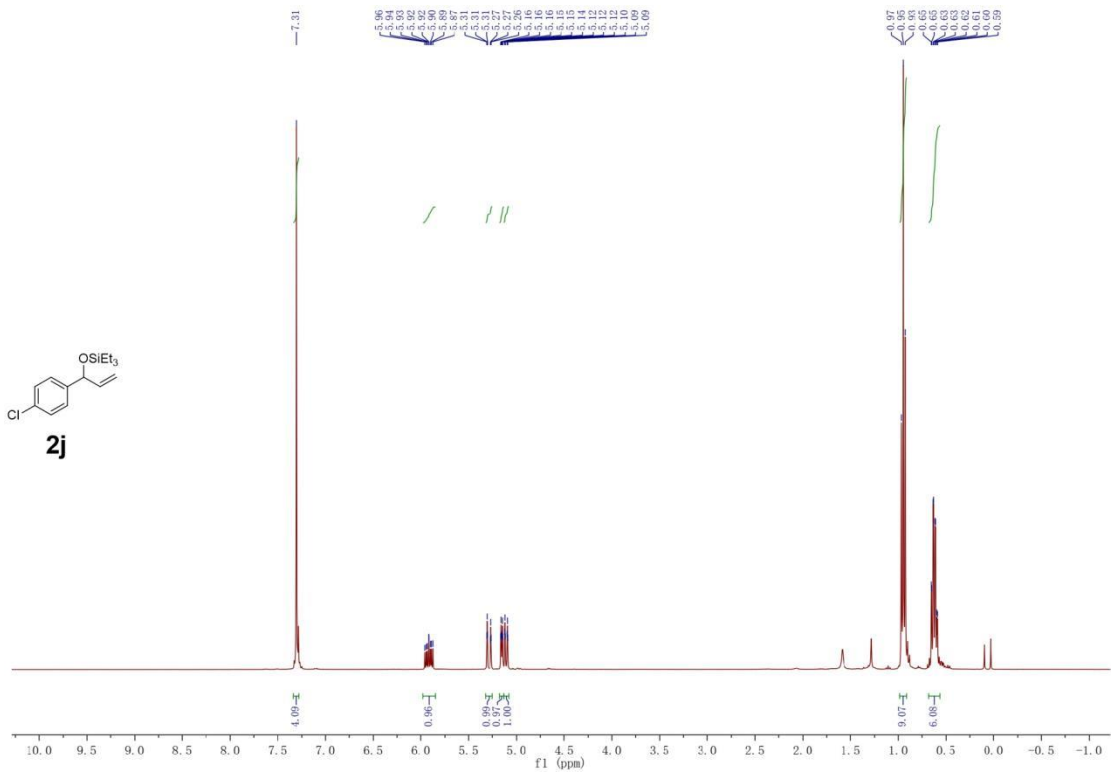
f1 (ppm)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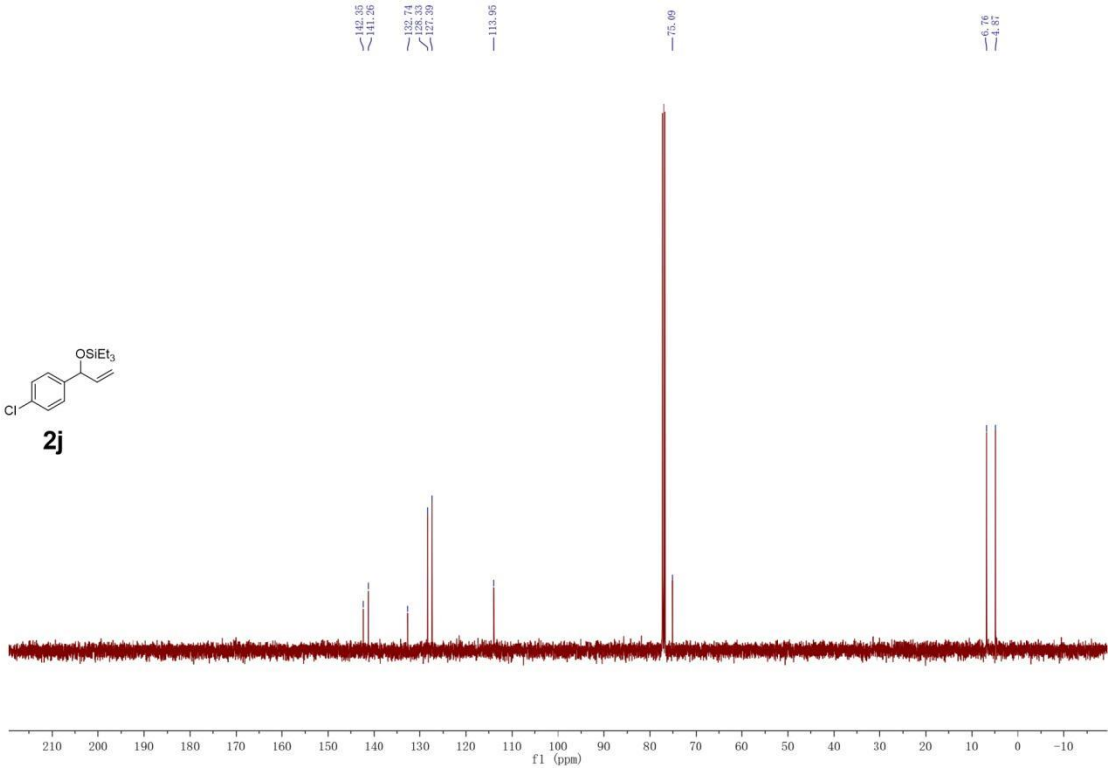
Cl
2j
7.31
f1 (ppm)


142.35
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4.87
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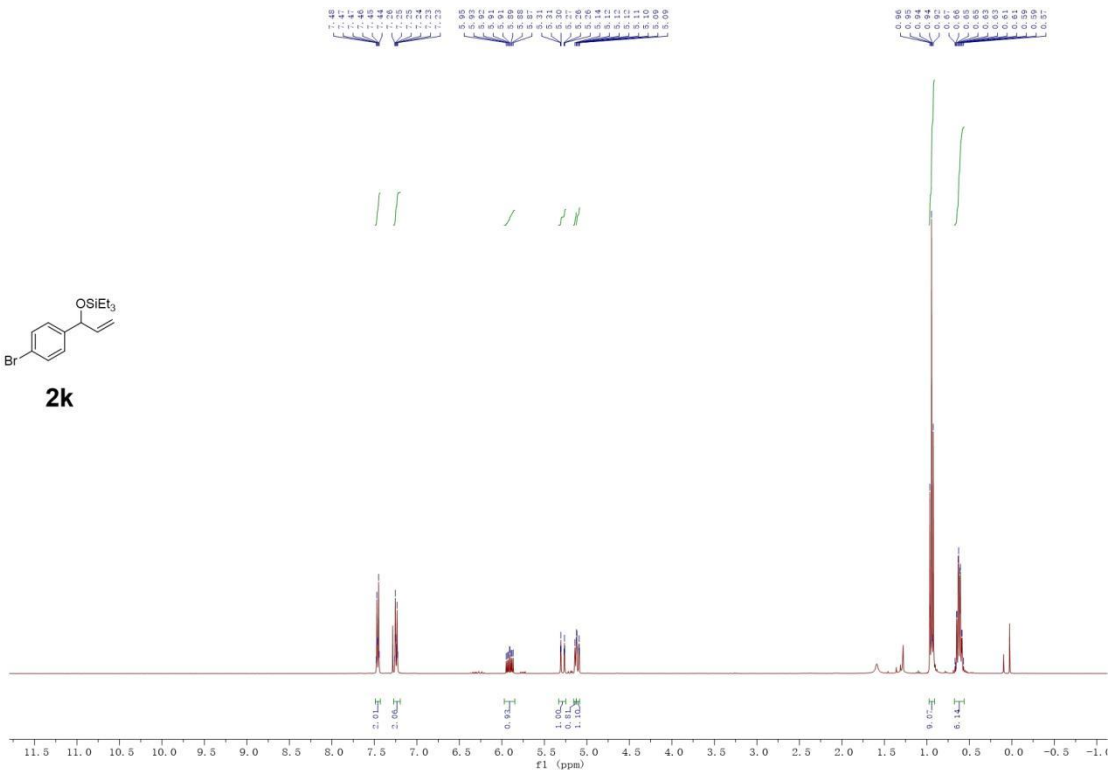
Cl
2j
f1 (ppm)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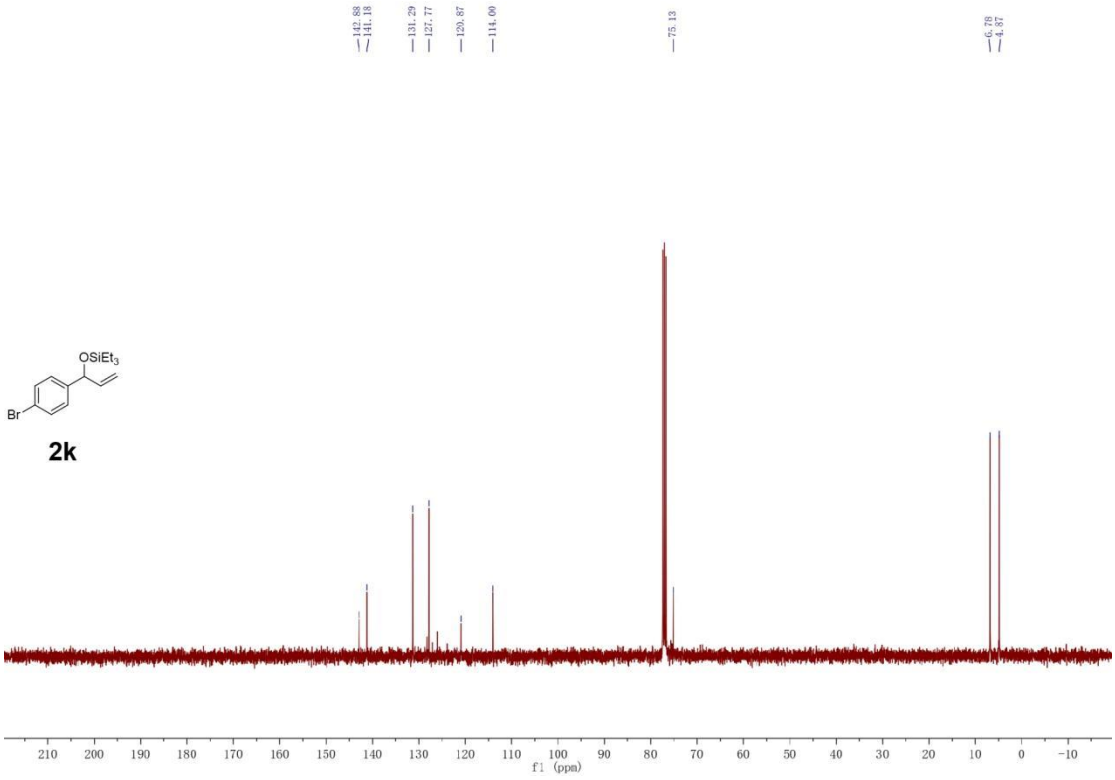
Br
2k
f1 (ppm)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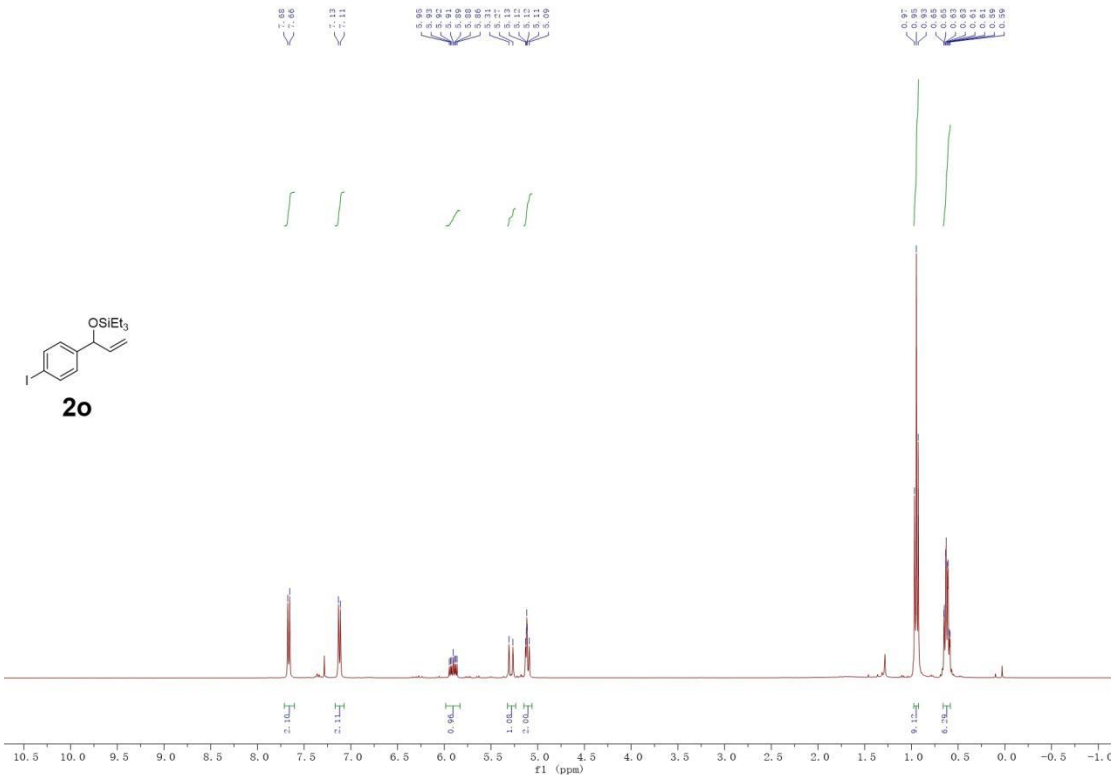
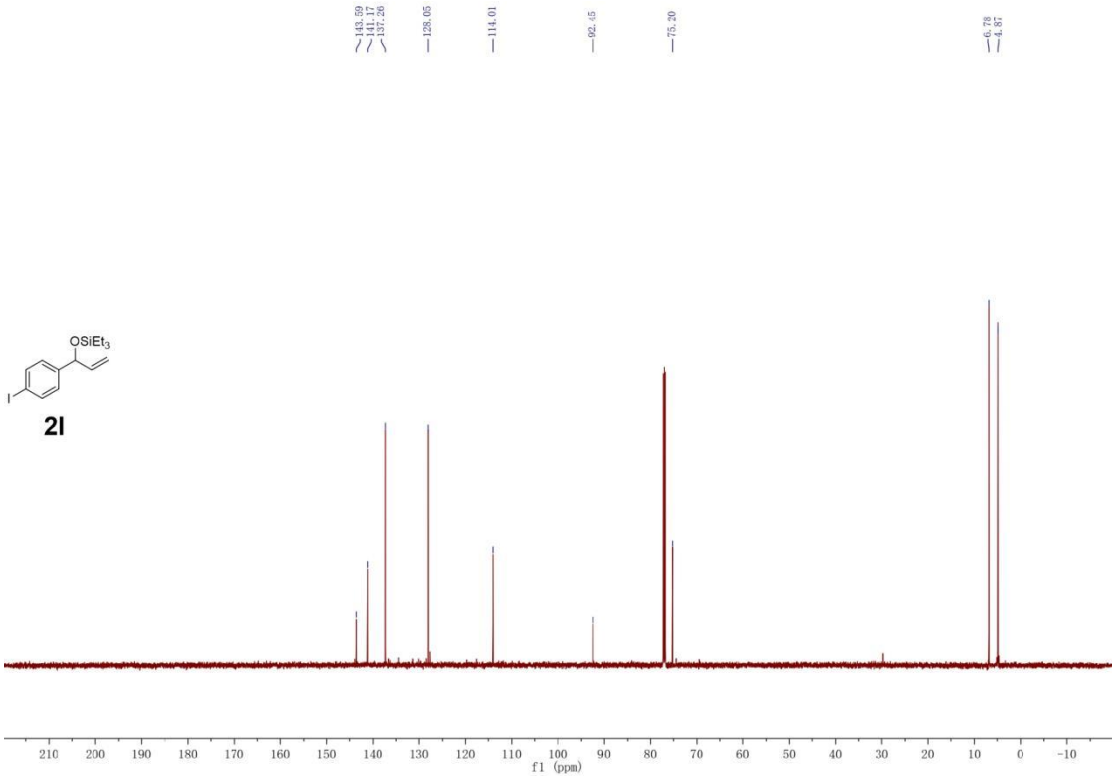
114.09
75.13
6.78
4.87
OSiEt3
Br
2k
f1 (ppm)


OSiEt3
I
2o
f1 (ppm)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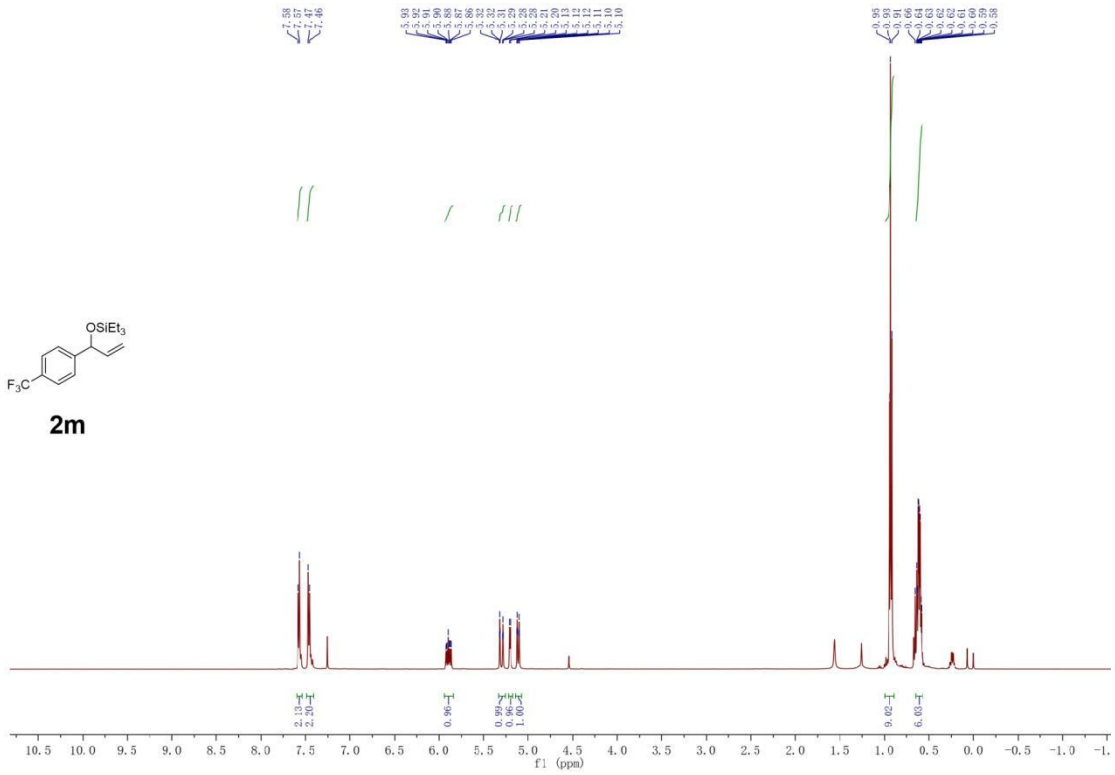
4.87
OSiEt3
I
2l
f1 (ppm)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5.92
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5.89
5.88
5.87
5.86
5.32
5.31
5.29
5.28
5.21
5.20
5.13
5.12
5.11
5.10
0.95
0.93
0.91
0.66
0.64
0.63
0.62
0.62
0.61
0.60
0.59
0.58
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F3C
2m
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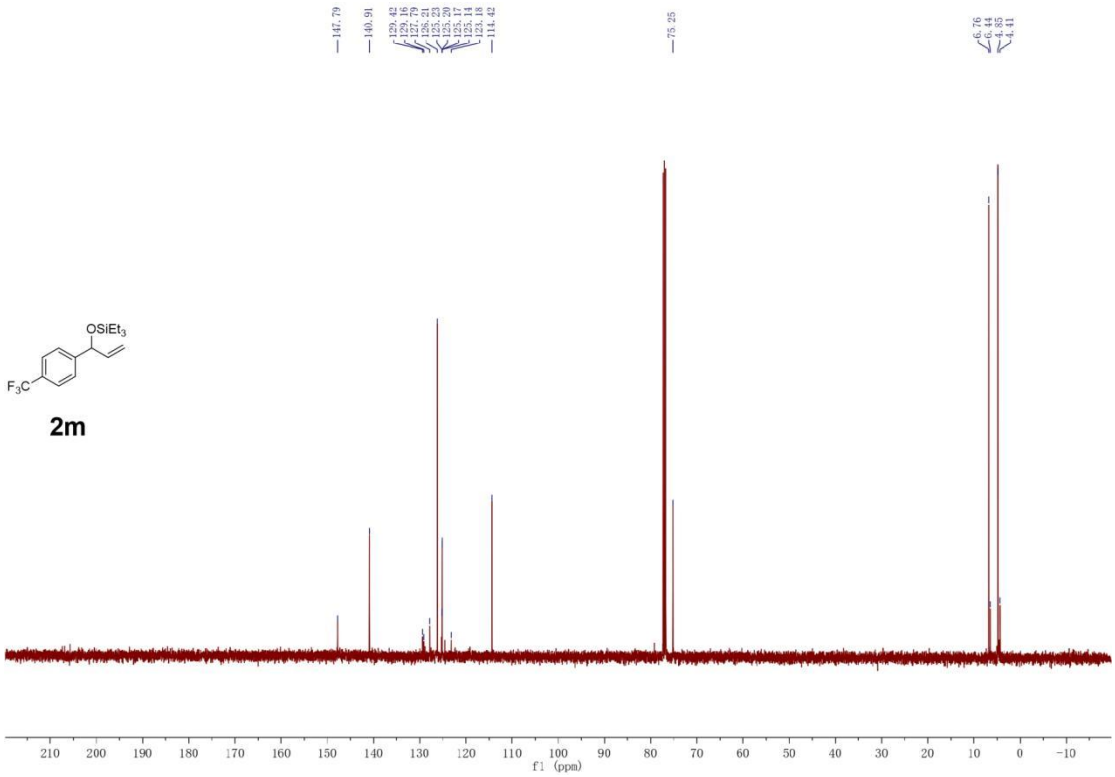
1.00
9.02
6.03
f1 (ppm)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129.16
127.79
126.21
125.23
125.20
125.17
125.14
125.18
114.42
75.25
6.76
6.44
4.65
4.41
OSiEt3
F3C
2m
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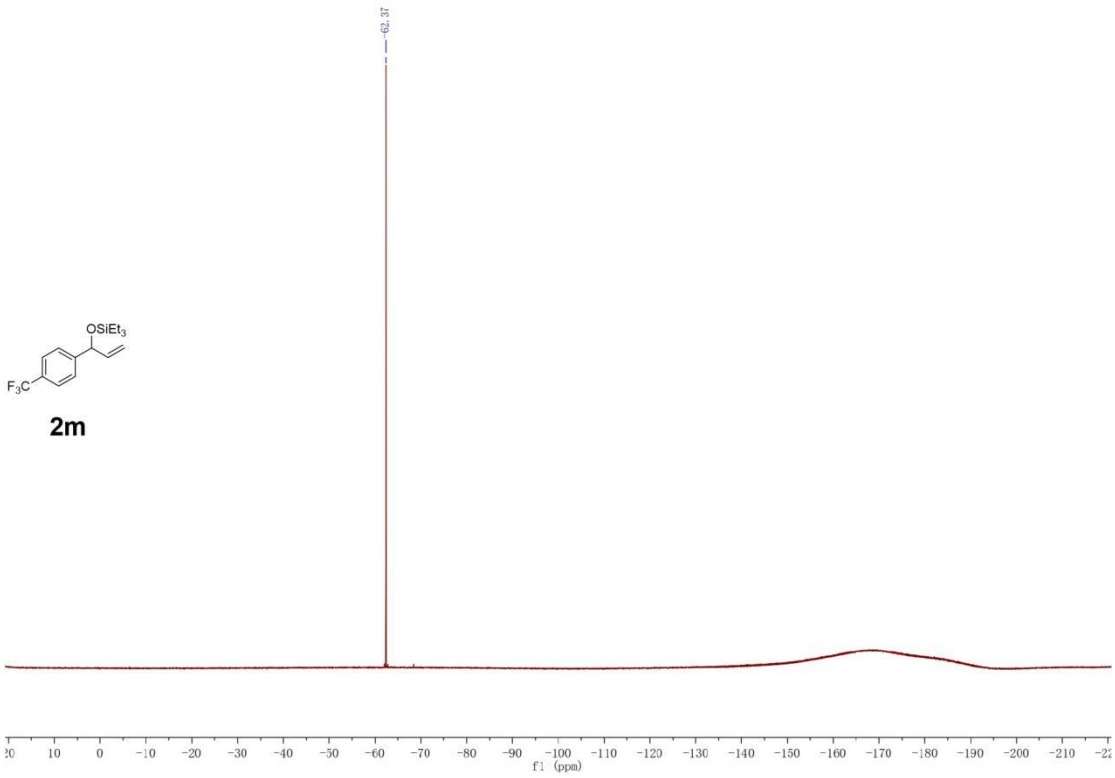
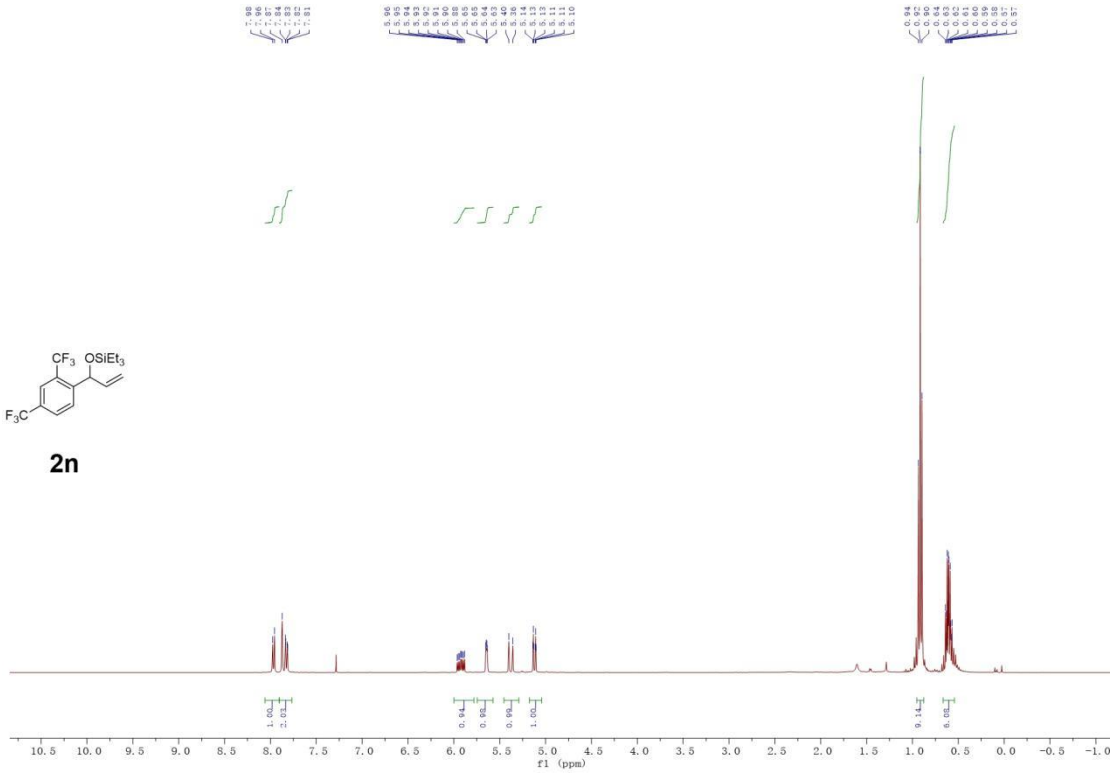
f1 (ppm)


62.37
OSiEt3
F3C
2m
20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210 −22
f1 (ppm)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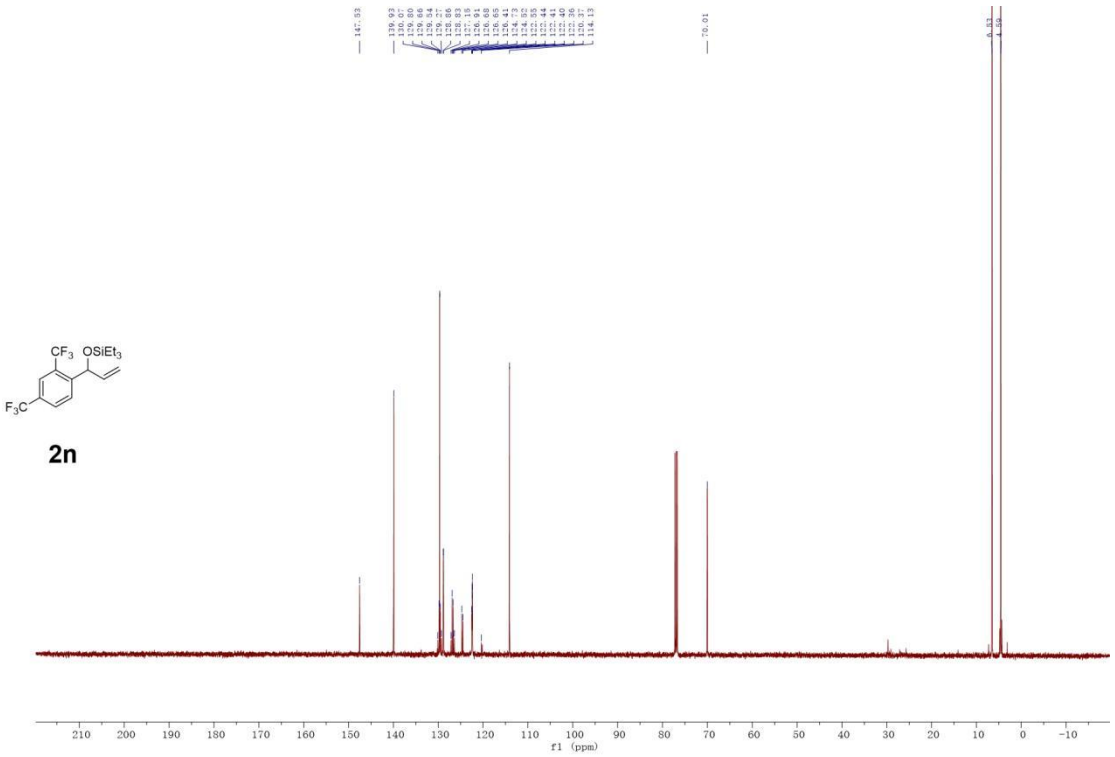
F3C
2n
f1 (ppm)


CF3
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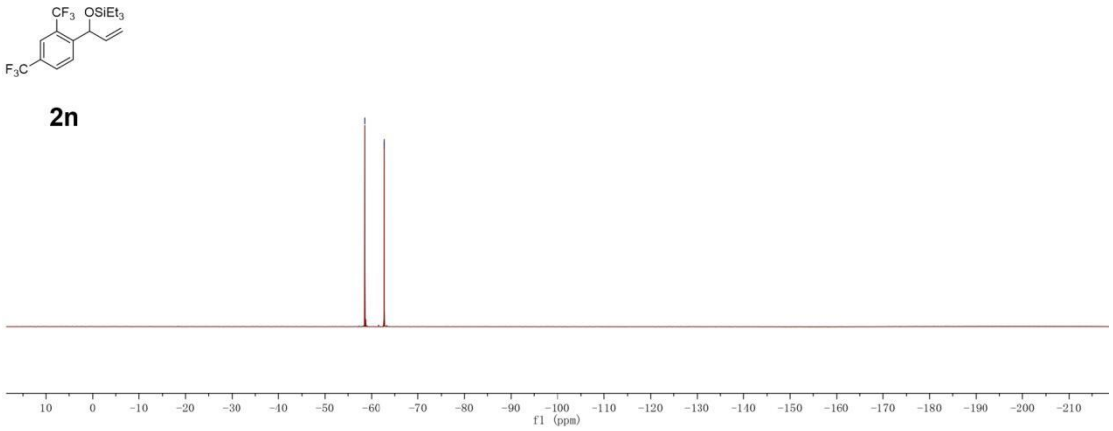
F3C
2n
f1 (ppm)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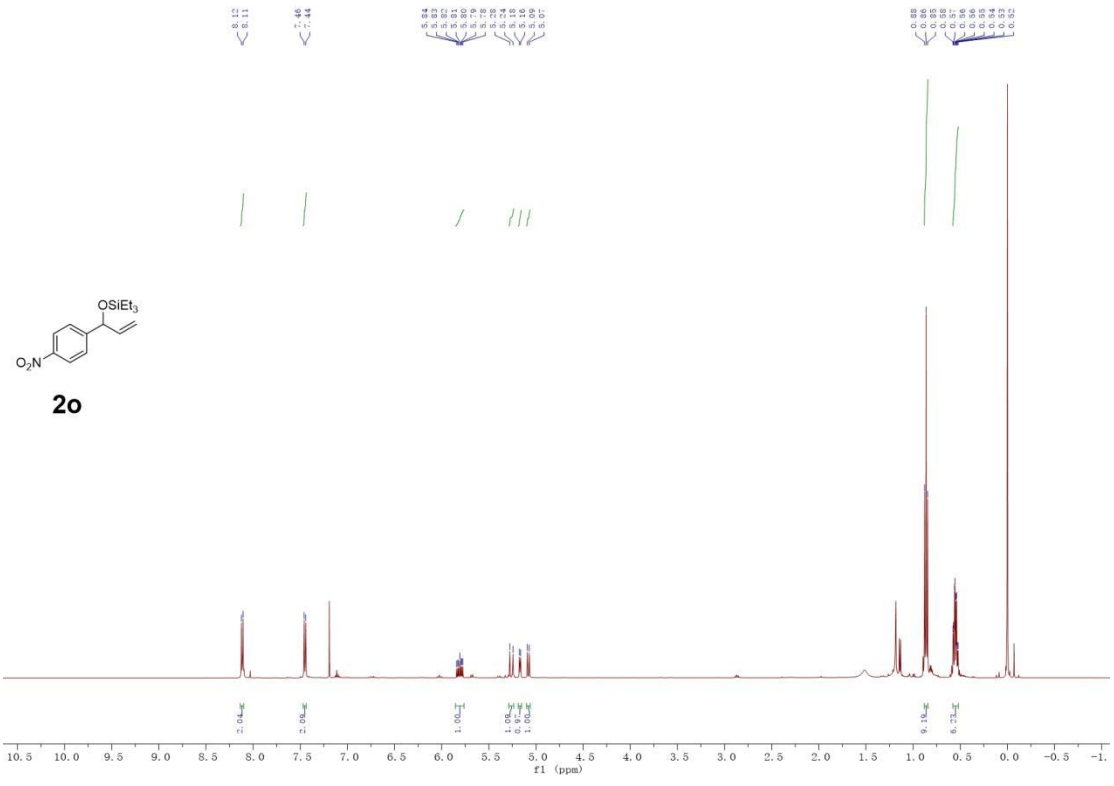
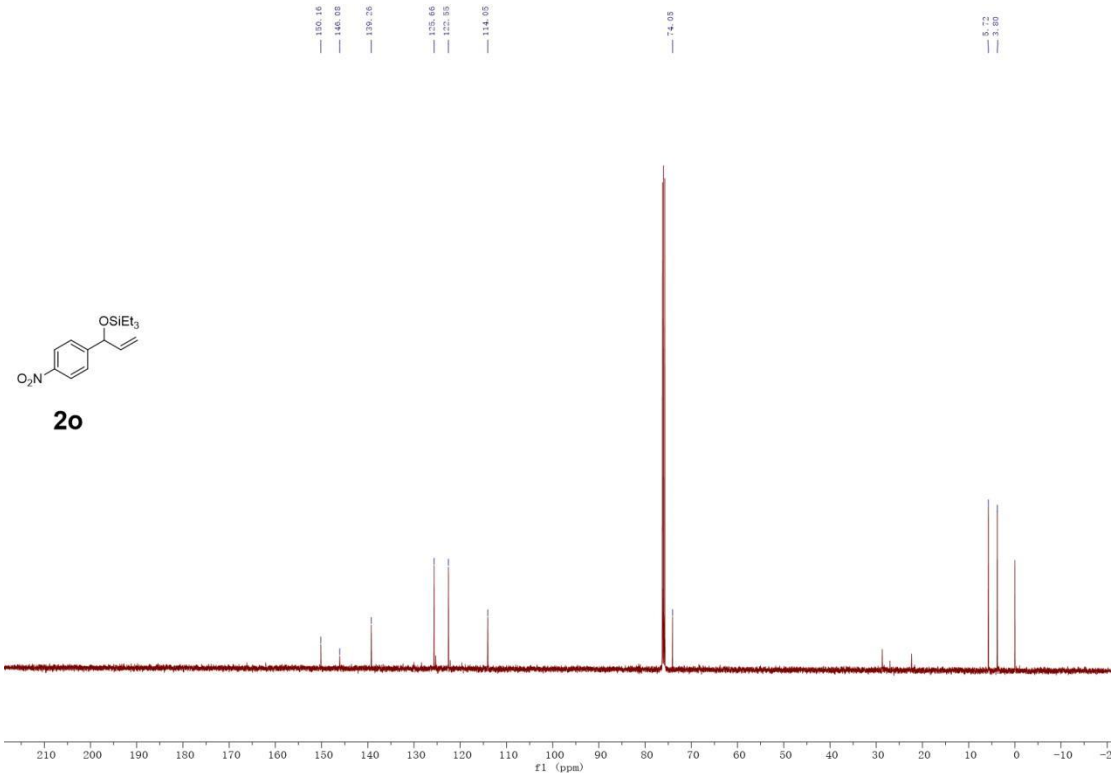
2n
f1 (ppm)


OSiEt3
O2N
2o
f1 (ppm)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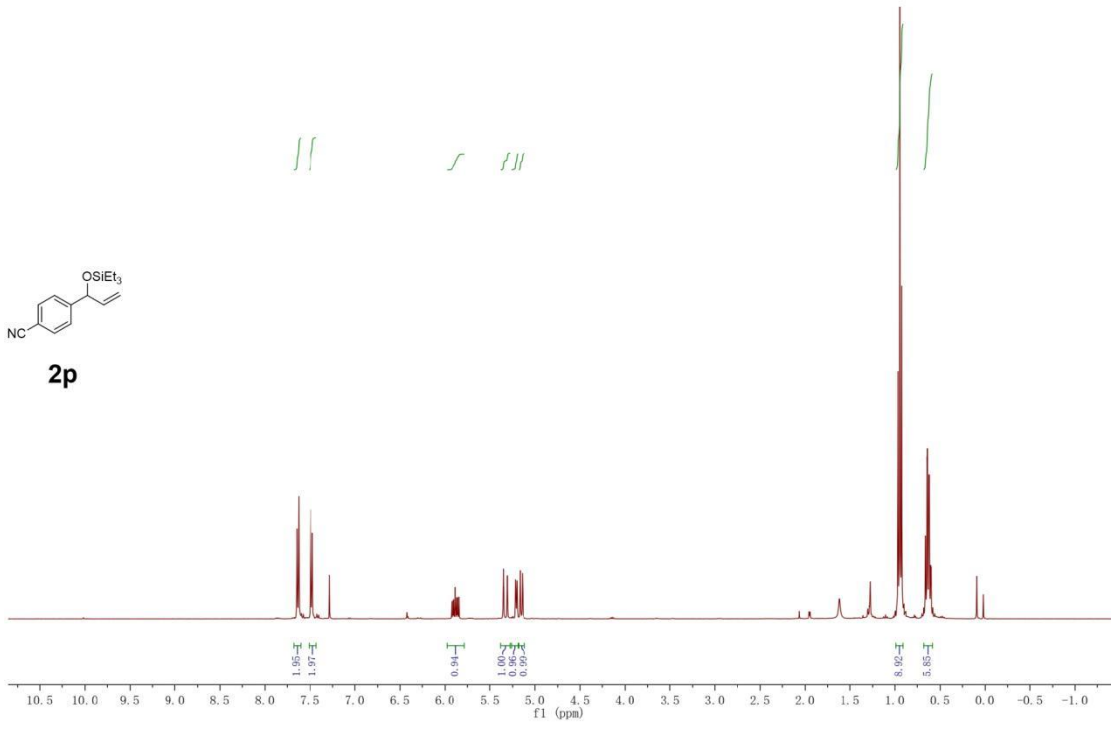
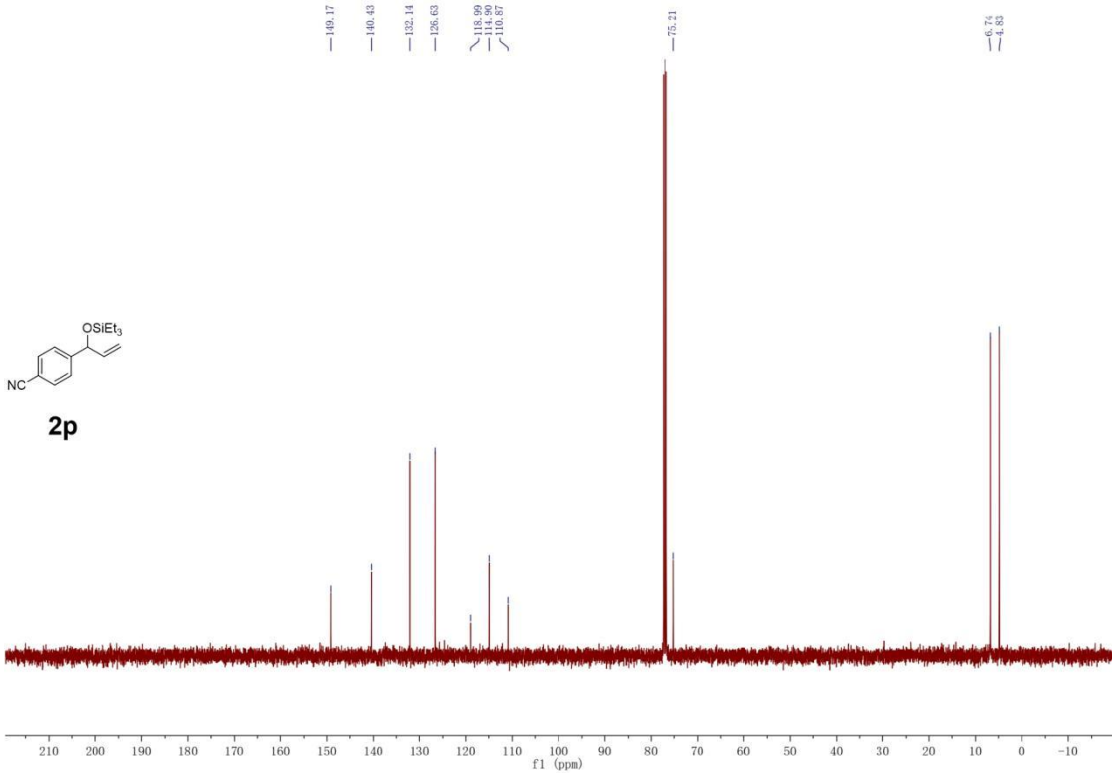
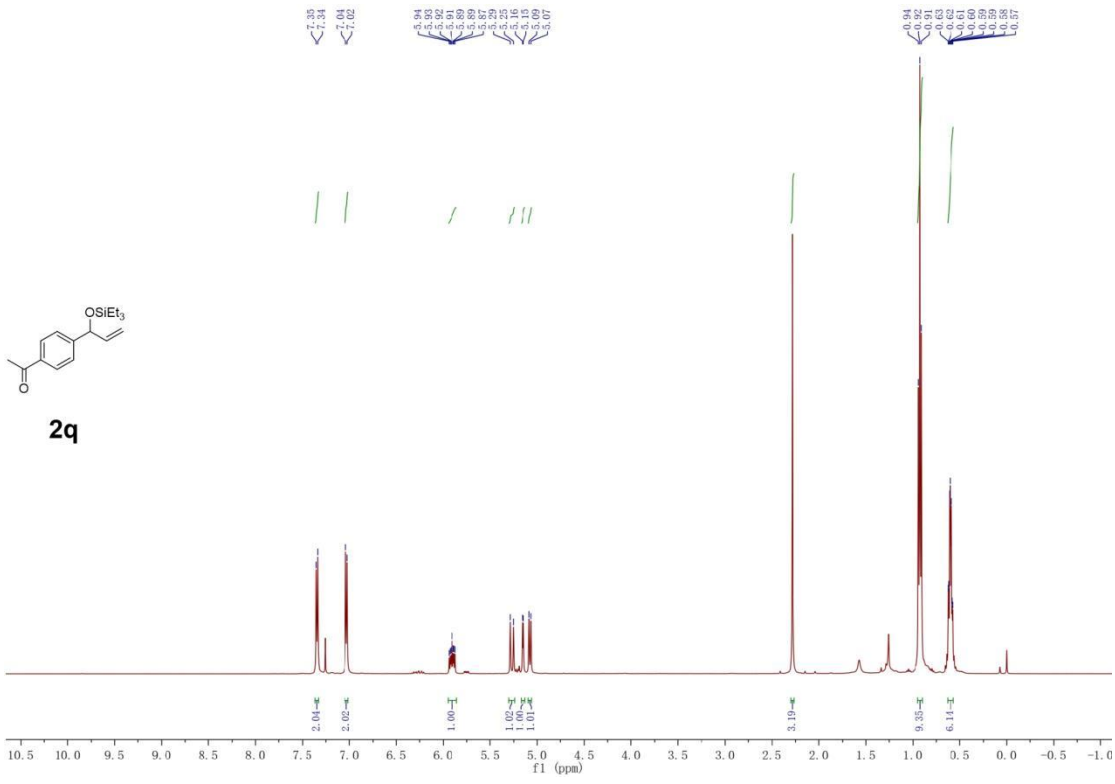
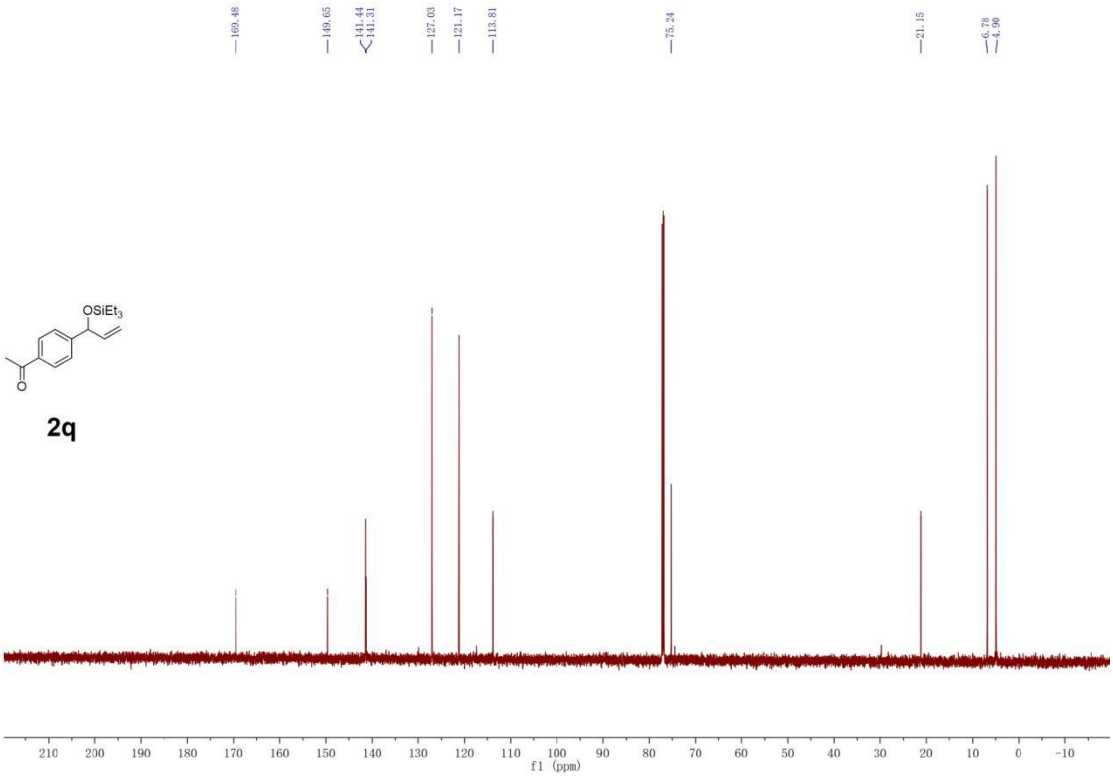
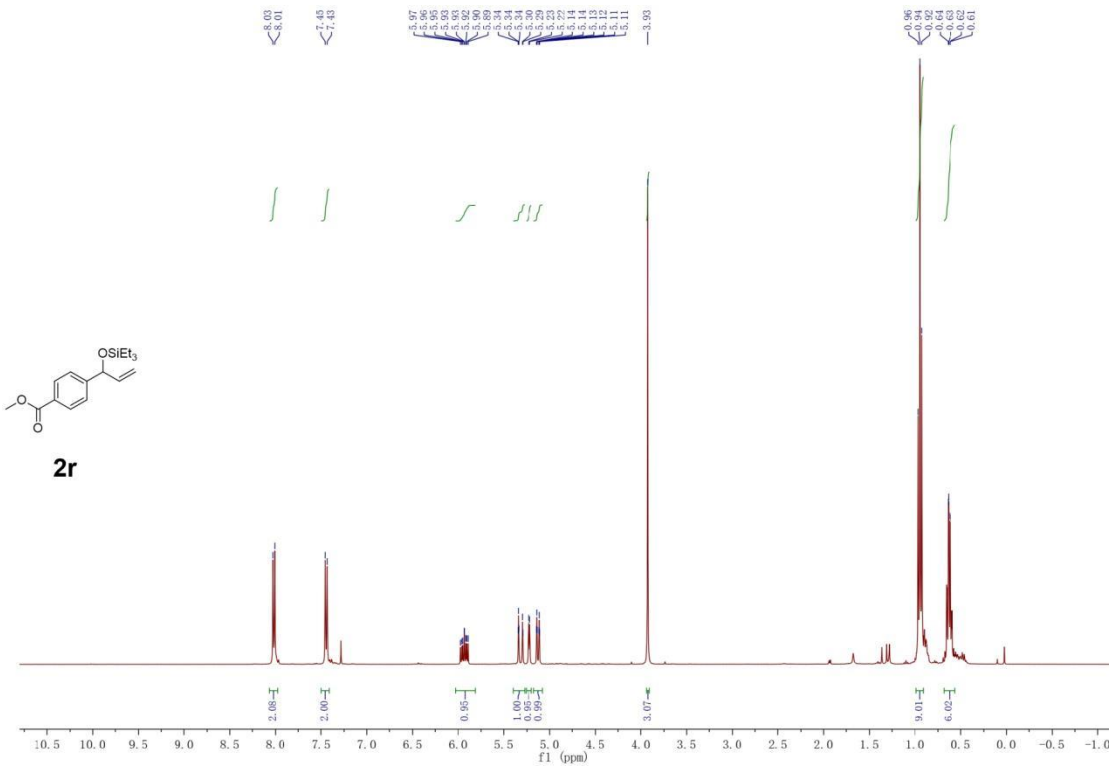
3.80
OSiEt3
O2N
2o
f1 (ppm)

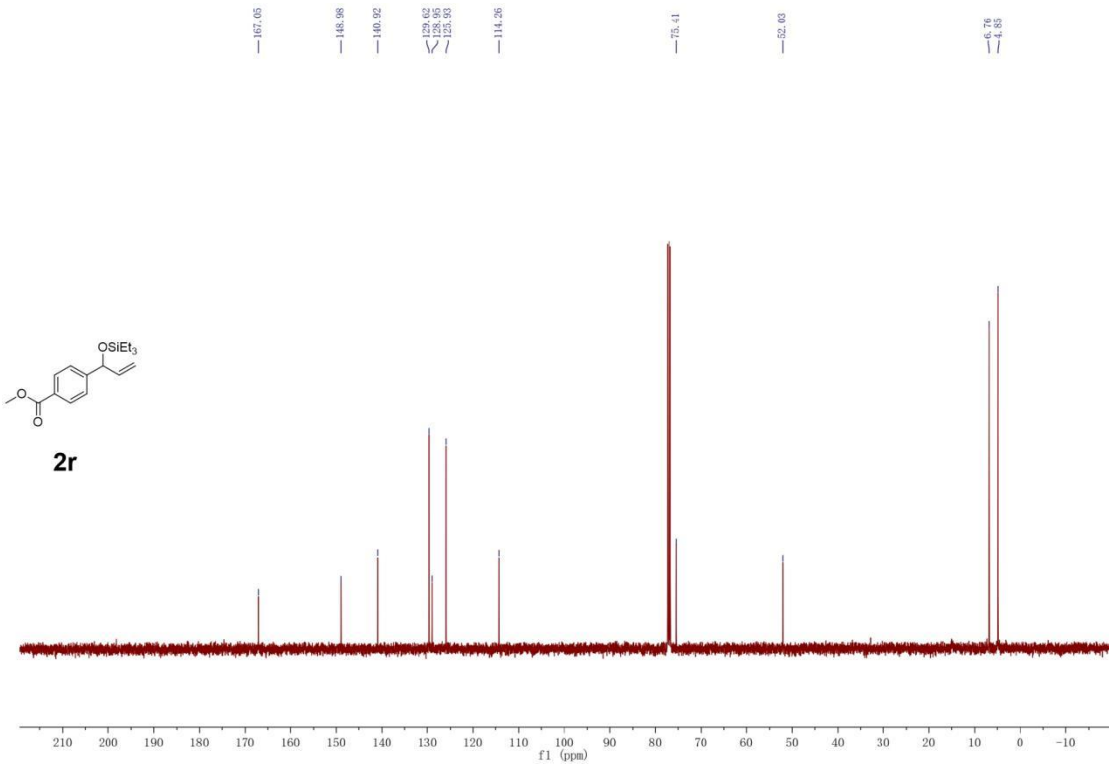

OSiEt3
NC
2p
1.95
1.97
0.94
1.00
0.96
0.99
8.92
5.85
f1 (ppm)

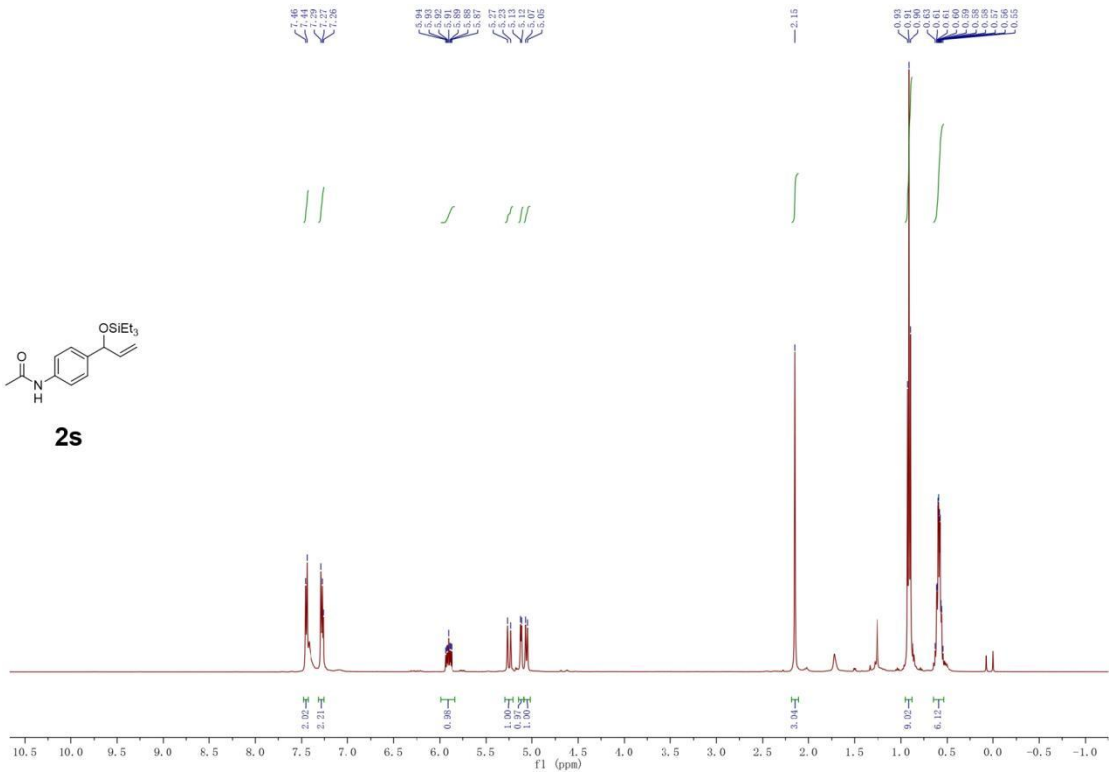

149.17
140.43
132.14
126.63
118.99
114.90
110.87
75.21
6.74
4.83
OSiEt3
NC
2p
f1 (ppm)

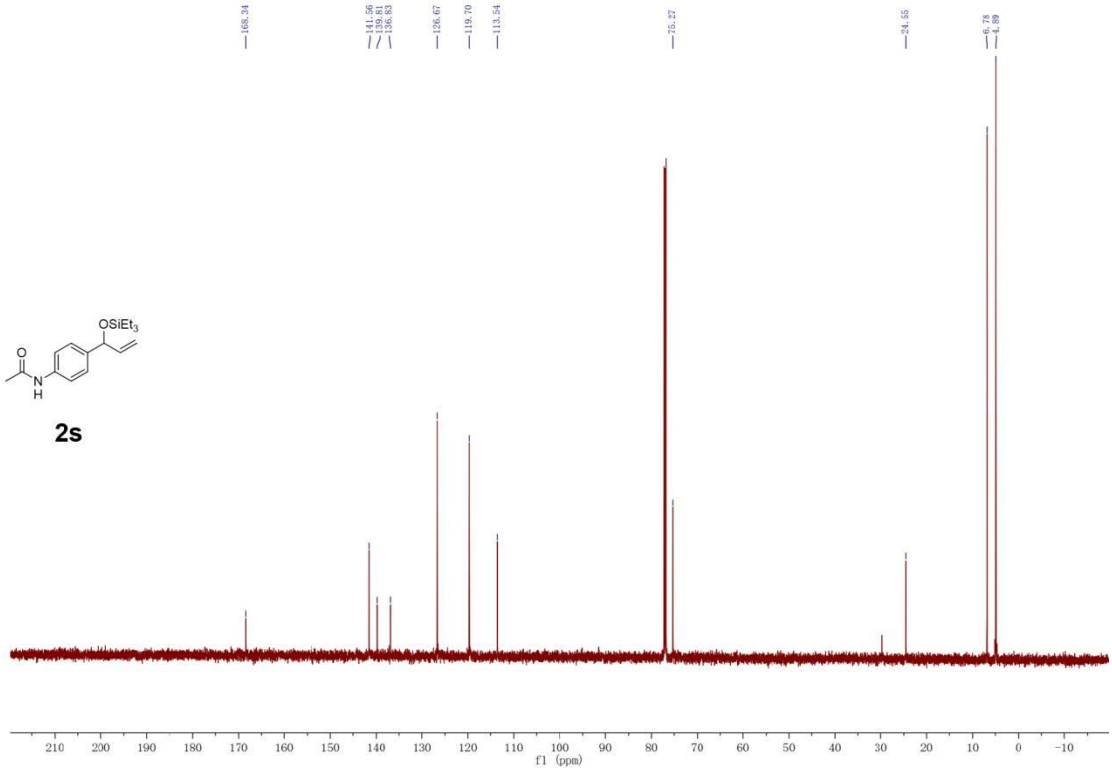

7.35
7.34
7.04
7.02
5.94
5.93
5.92
5.91
5.89
5.87
5.29
5.25
5.16
5.15
5.09
5.07
0.94
0.92
0.91
0.63
0.62
0.61
0.60
0.59
0.58
0.57
OSiEt3
O
2q
2.04
2.02
1.00
1.02
1.00
1.01
3.19
9.35
6.14
f1 (ppm)

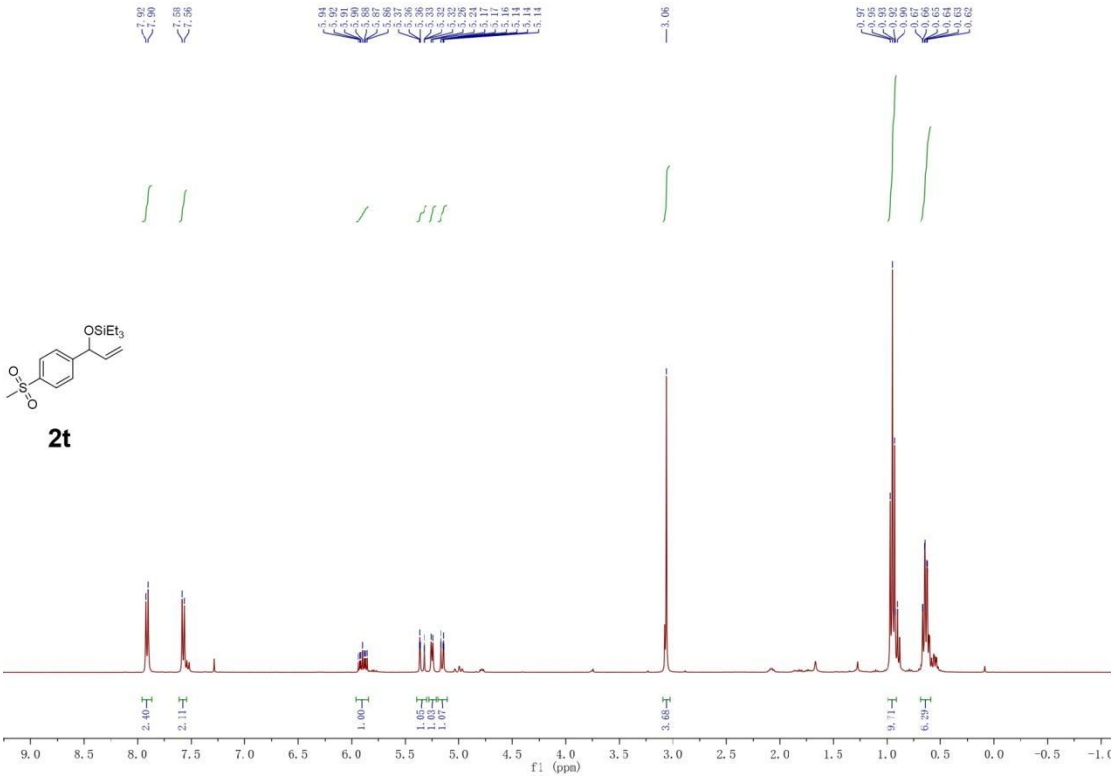

OSiEt3
O
2q
169.48
149.65
141.44
141.31
127.03
121.17
113.81
75.24
21.15
6.78
4.90
f1 (ppm)

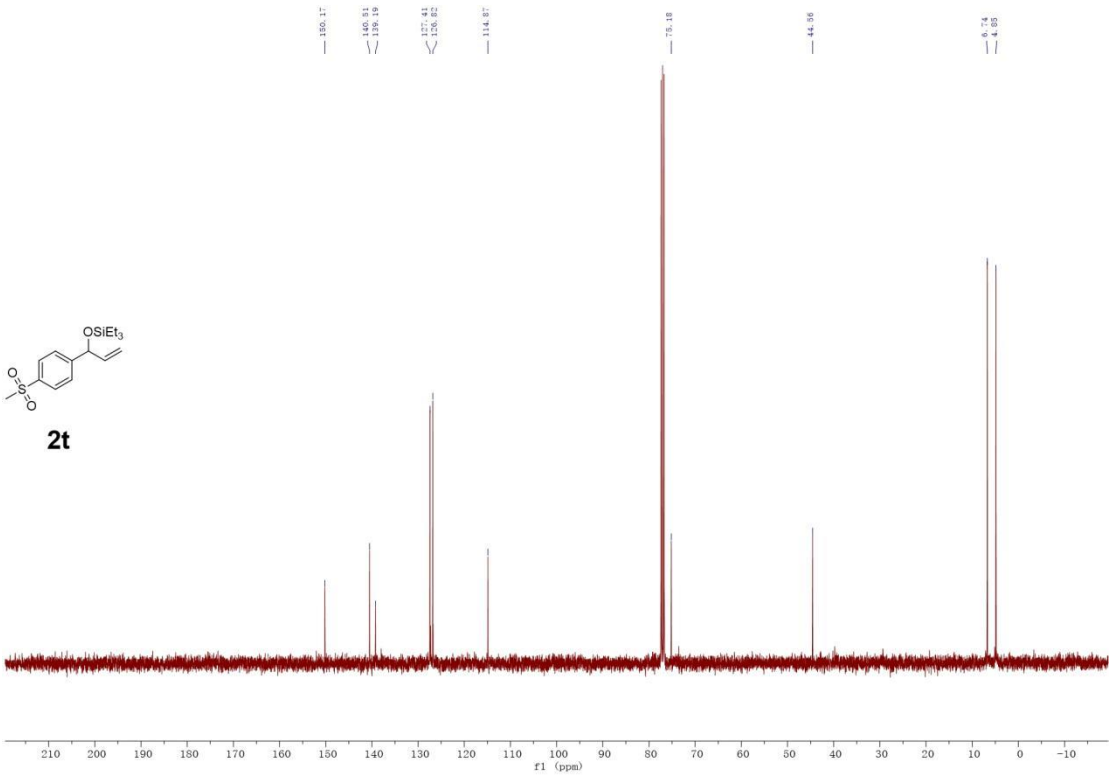

OSiEt3
O
O
2r
8.03
8.01
7.45
7.43
3.93
0.96
0.94
0.92
0.64
0.63
0.62
0.61
2.08
2.00
0.95
1.00
0.95
0.99
3.07
9.01
6.02
f1 (ppm)

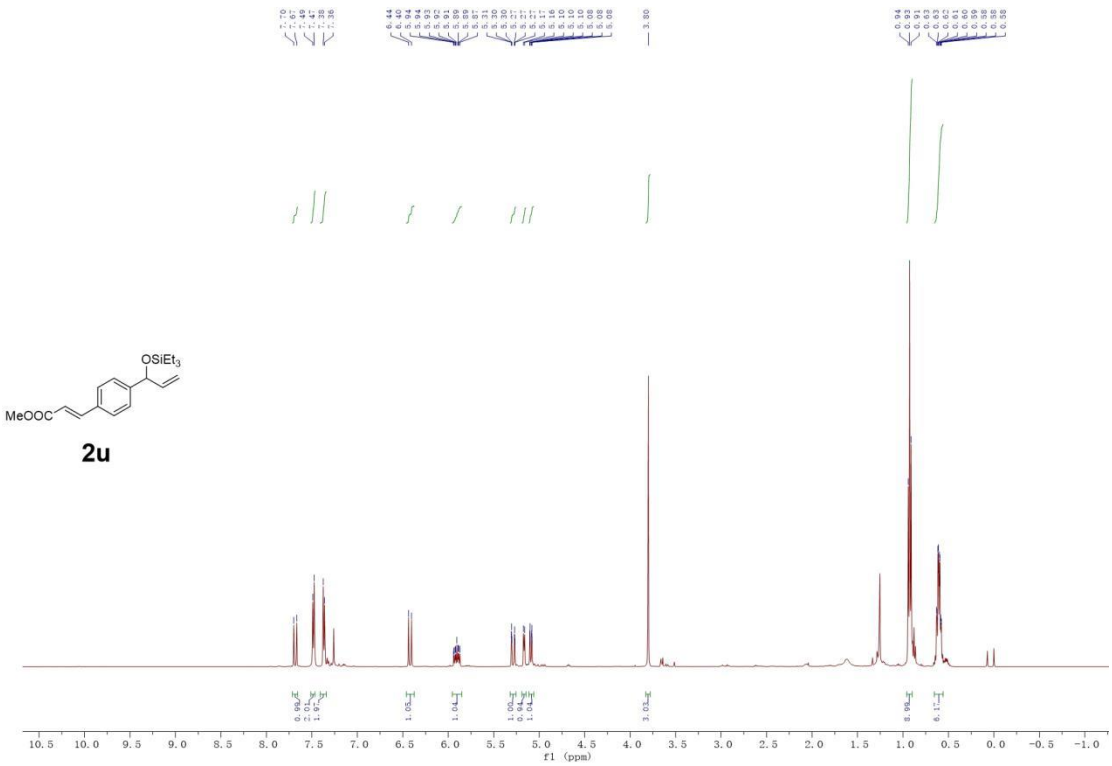

167.05
148.98
140.92
129.62
128.95
125.93
114.26
75.41
52.03
6.76
4.83
OSiEt3
O
O
2r
f1 (ppm)

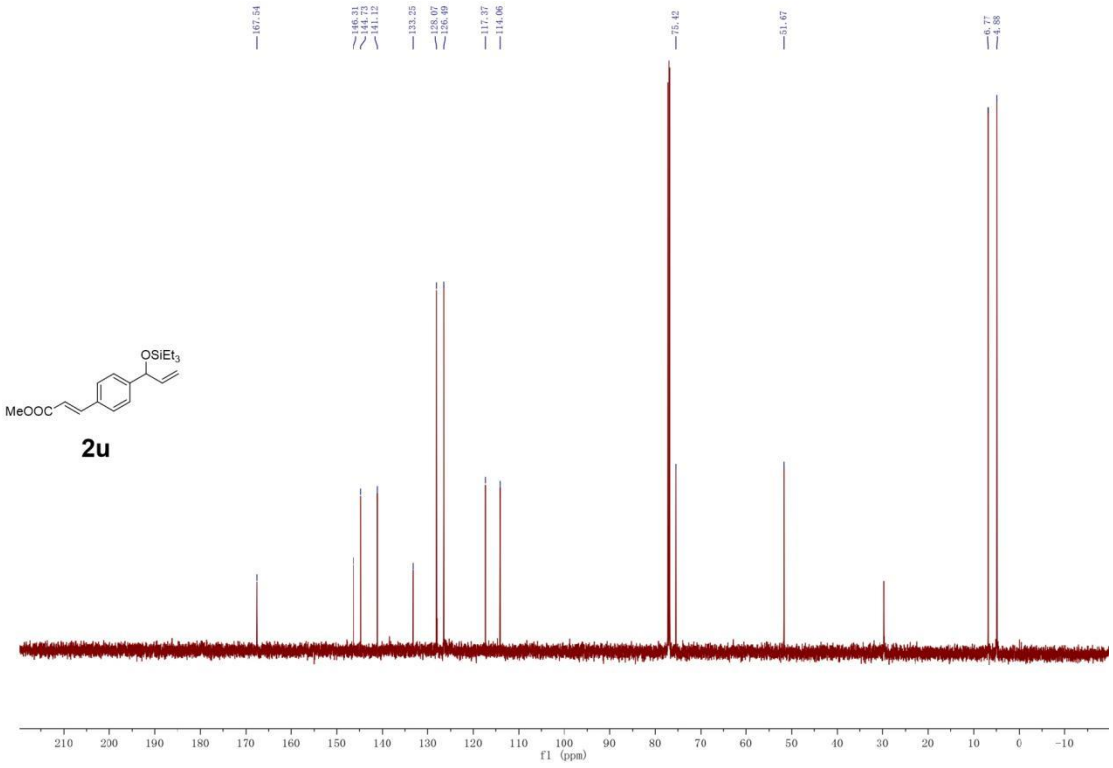

7.46
7.44
7.29
7.27
7.26
5.94
5.93
5.92
5.91
5.89
5.88
5.87
5.27
5.25
5.13
5.12
5.07
5.05
2.15
0.93
0.91
0.90
0.63
0.61
0.60
0.59
0.58
0.58
0.57
0.56
0.55
OSiEt3
O
N
H
2s
2.02
2.21
0.98
1.00
0.97
1.00
3.04
9.02
6.12
f1 (ppm)

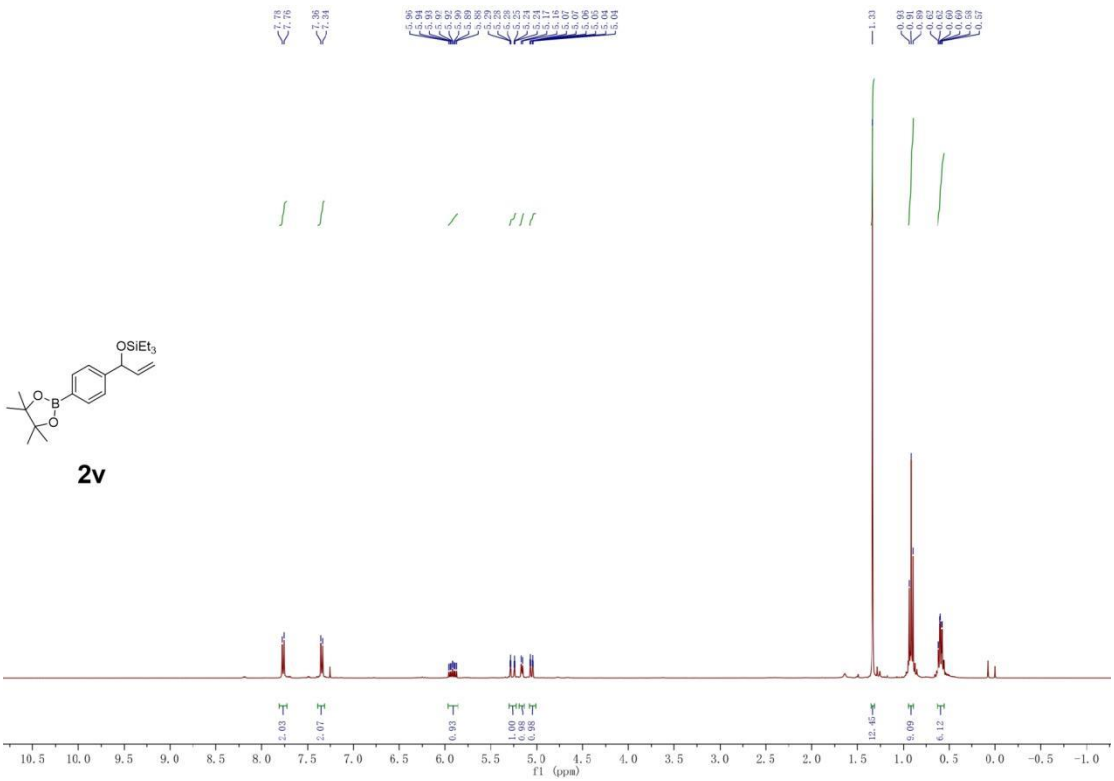

168.34
141.56
139.81
138.83
126.67
119.70
113.54
75.27
24.55
6.78
4.89
OSiEt3
O
N
H
2s
f1 (ppm)

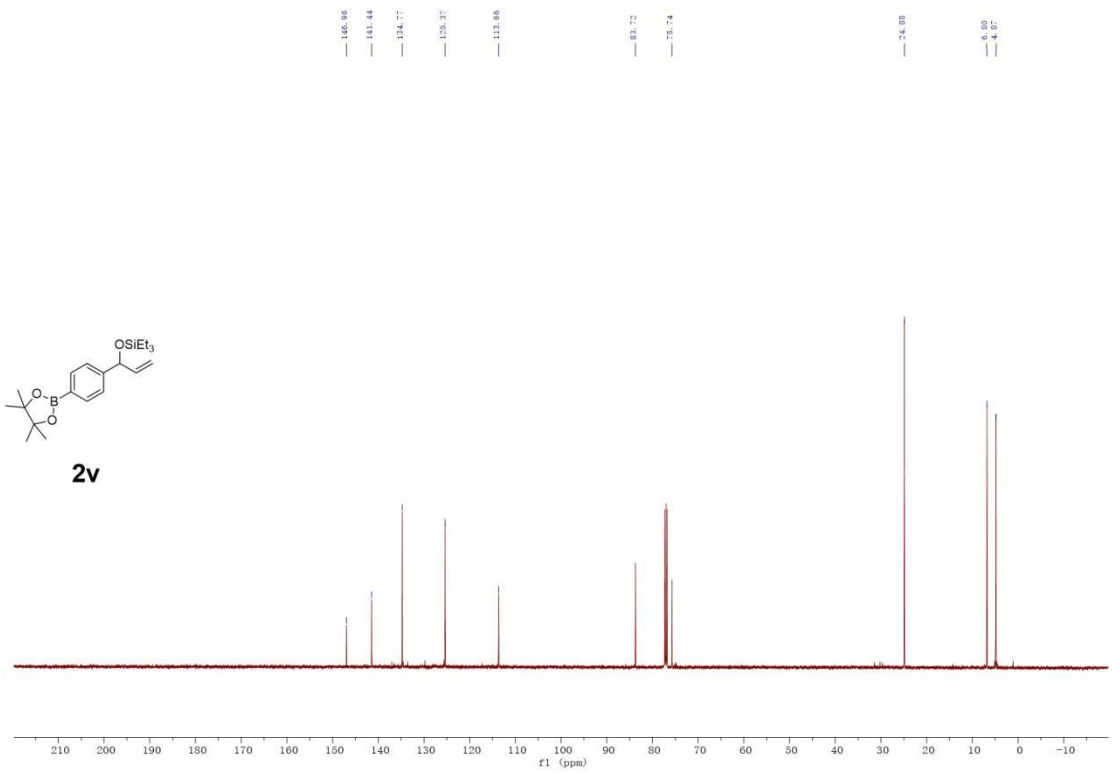

7.97
7.99
7.58
7.56
5.94
5.92
5.91
5.90
5.88
5.87
5.86
5.37
5.36
5.35
5.33
5.32
5.25
5.24
5.17
5.16
5.15
5.14
5.13
5.14
3.06
0.97
0.96
0.93
0.92
0.90
0.67
0.66
0.65
0.64
0.63
0.62
OSiEt3
O
S
O
2t
2.40
2.11
1.00
1.05
1.03
1.07
3.68
9.71
6.29
f1 (ppm)

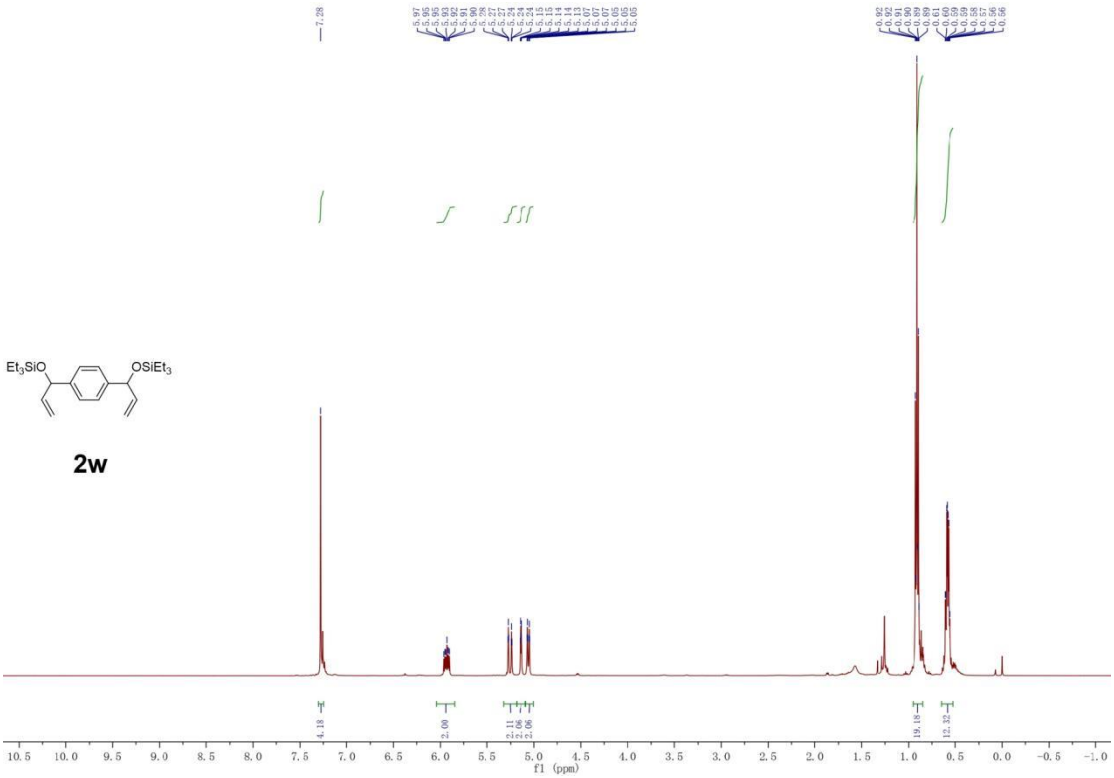

OSiEt3
O
S
O
2t
150.17
146.51
130.19
127.41
126.82
114.87
75.18
44.56
6.74
4.85
f1 (ppm)

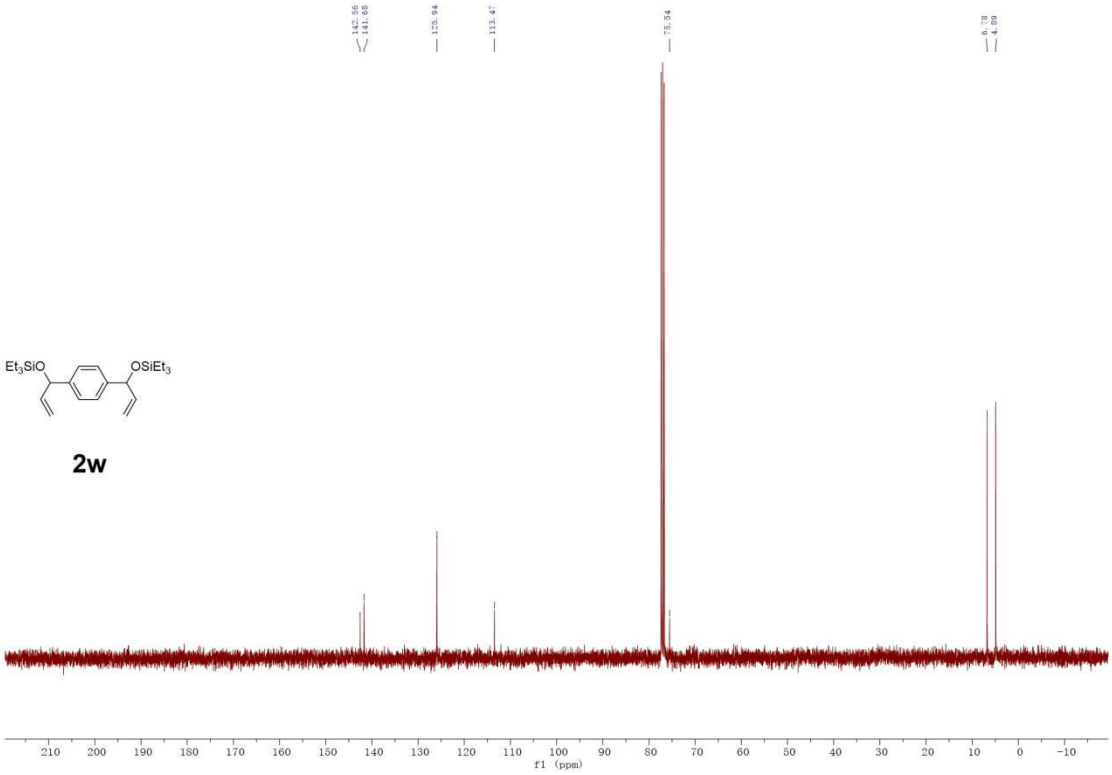

OSiEt3
MeOOC
2u
3.80
f1 (ppm)

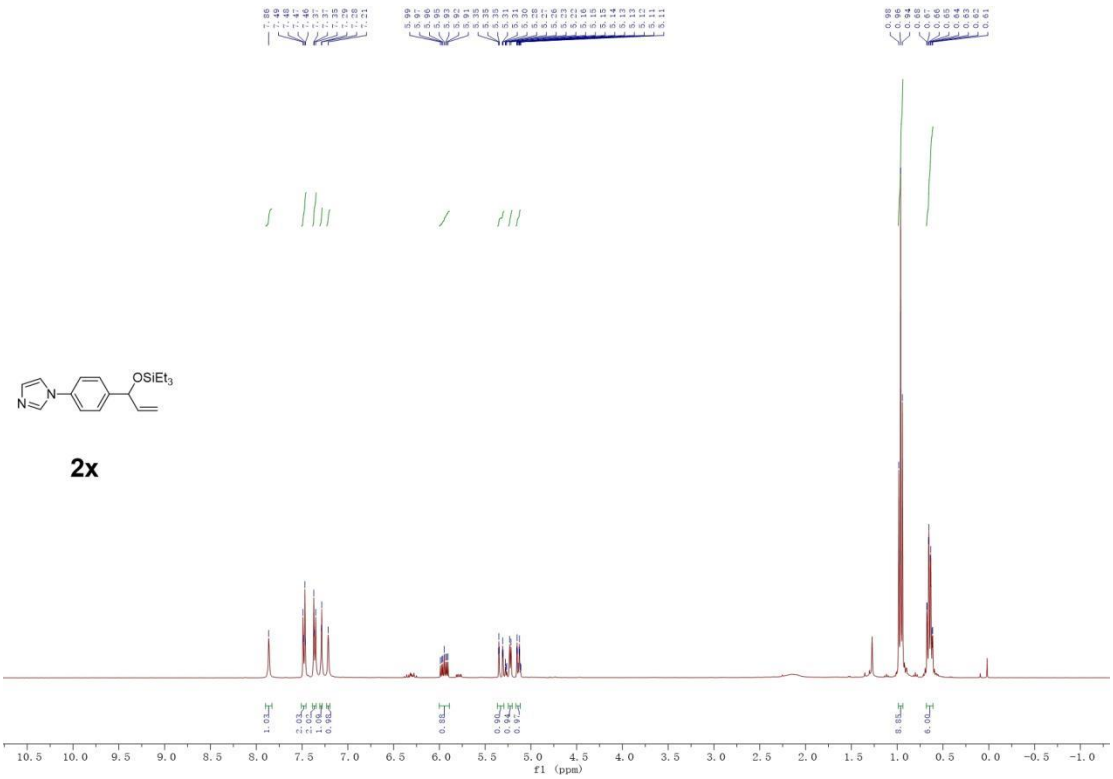

OSiEt3
MeOOC
2u
167.84
146.31
144.73
141.12
133.25
128.07
126.49
117.37
114.06
75.42
51.67
6.77
4.88
f1 (ppm)

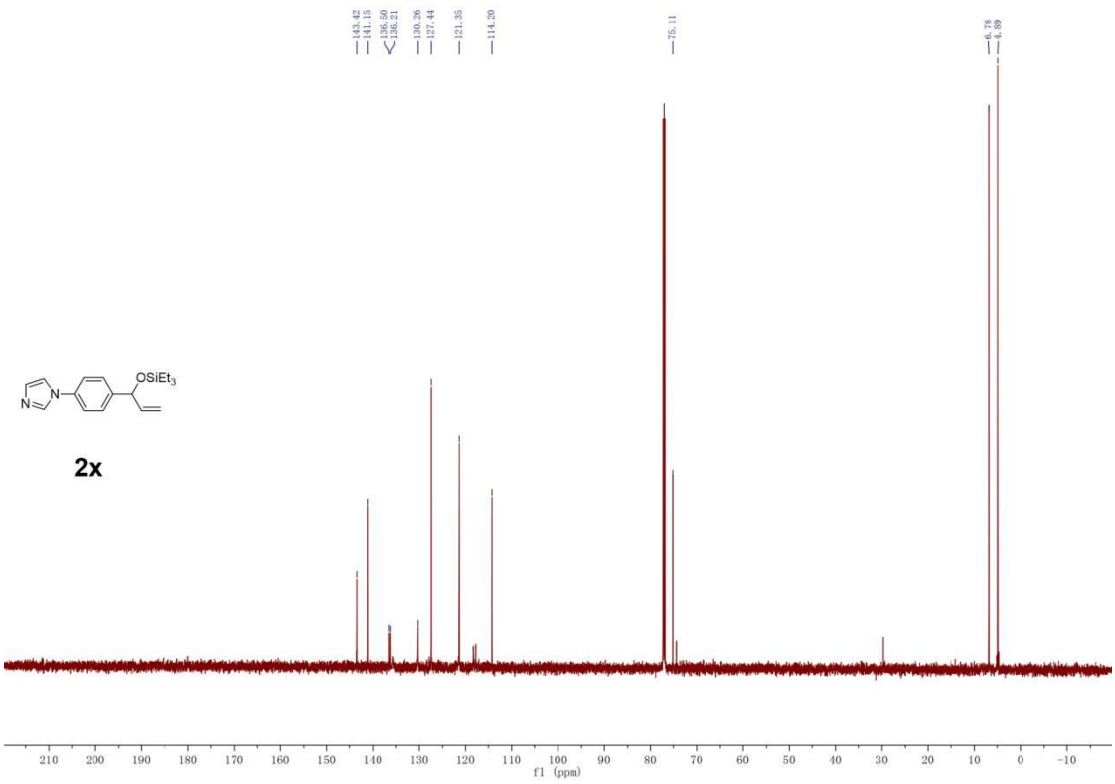

OSiEt3
2v
7.78
7.76
7.36
7.34
1.33
0.93
0.91
0.89
0.62
0.60
0.58
0.57
2.03
2.07
6.93
1.00
0.98
0.98
12.45
9.09
6.12
f1 (ppm)

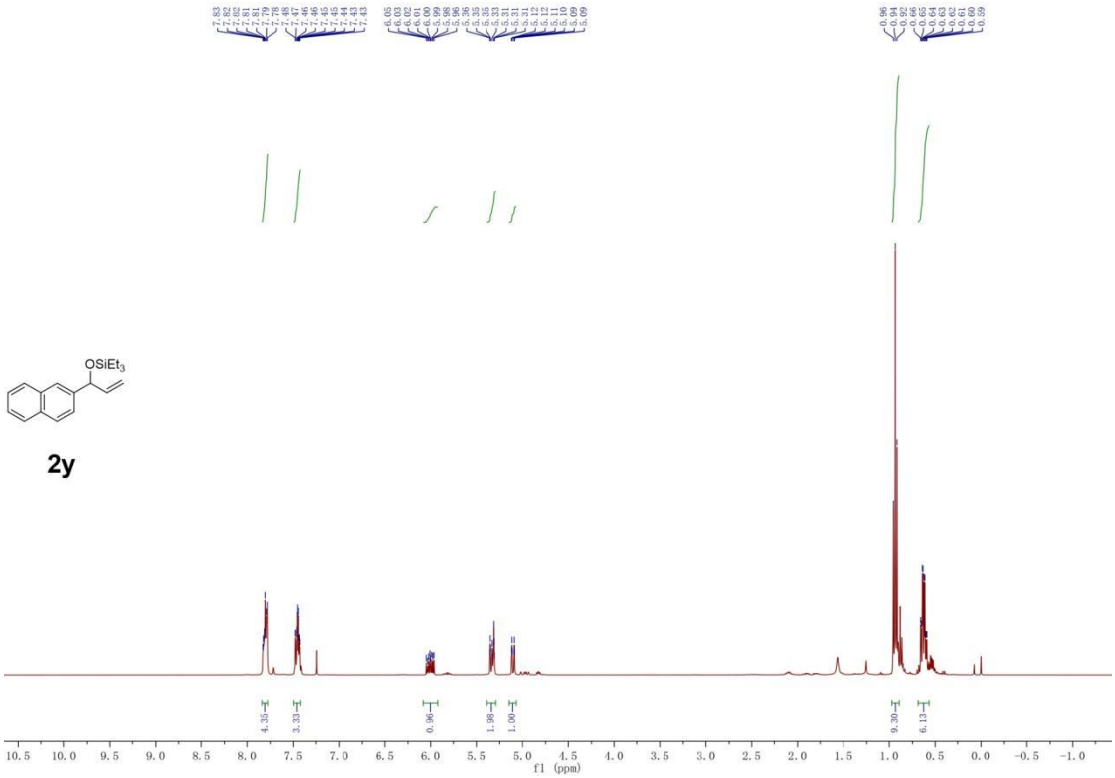

OSiEt3
B
O
O
2v
146.96
141.44
134.77
126.37
113.66
83.72
78.74
24.88
6.80
4.67
f1 (ppm)

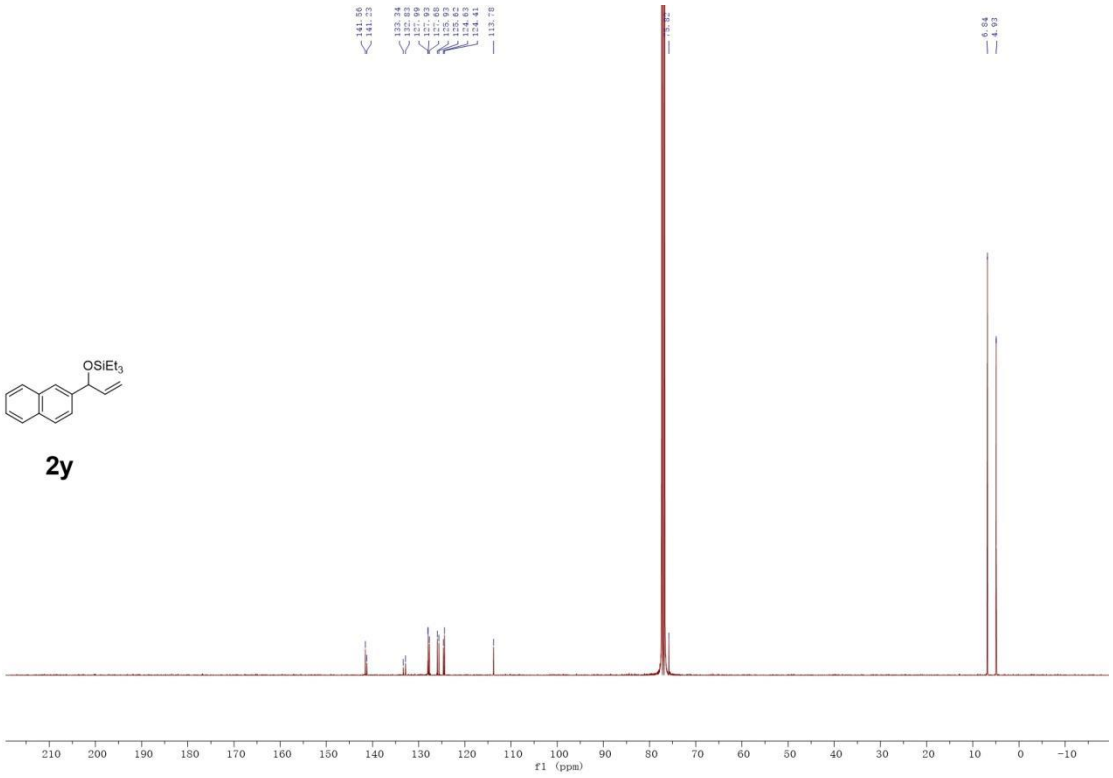

Et3SiO
OSiEt3
2w
7.28
f1 (ppm)


142.56
141.68
125.94
113.47
75.54
6.78
4.89
Et3SiO
OSiEt3
2w
f1 (ppm)

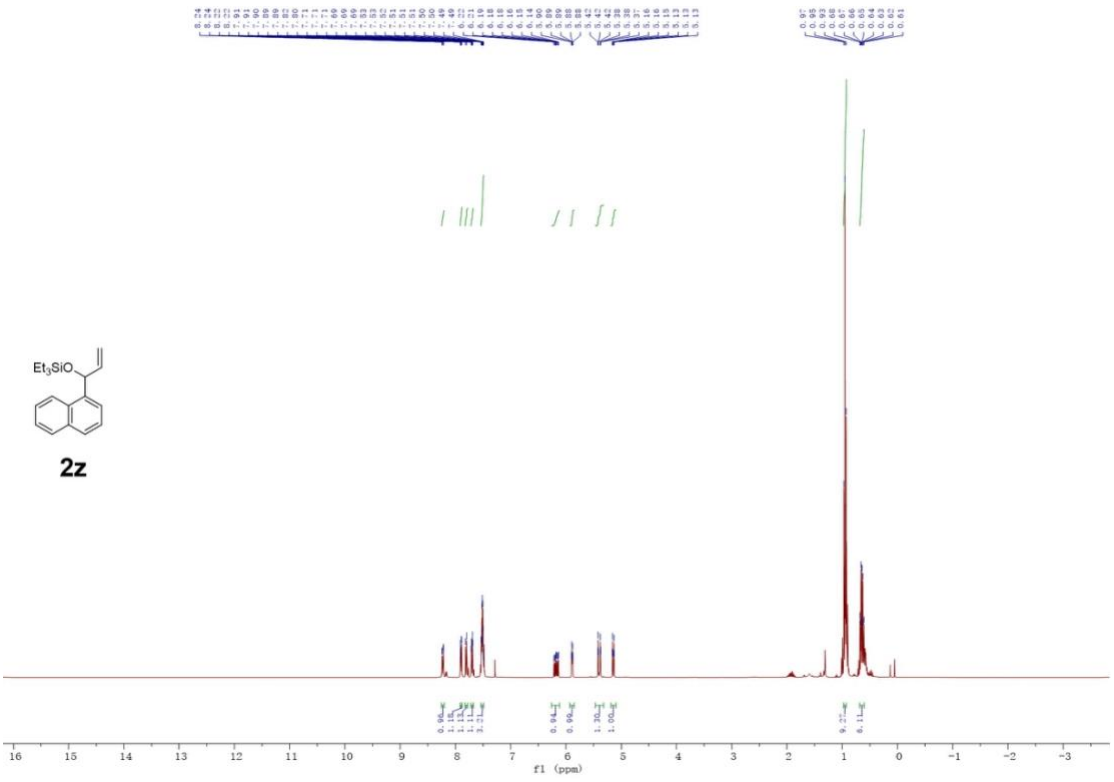

OSiEt3
2x
f1 (ppm)


143.42
141.15
136.50
136.21
130.26
127.44
121.35
114.20
75.11
6.78
4.89
OSiEt3
N
N
2x
f1 (ppm)

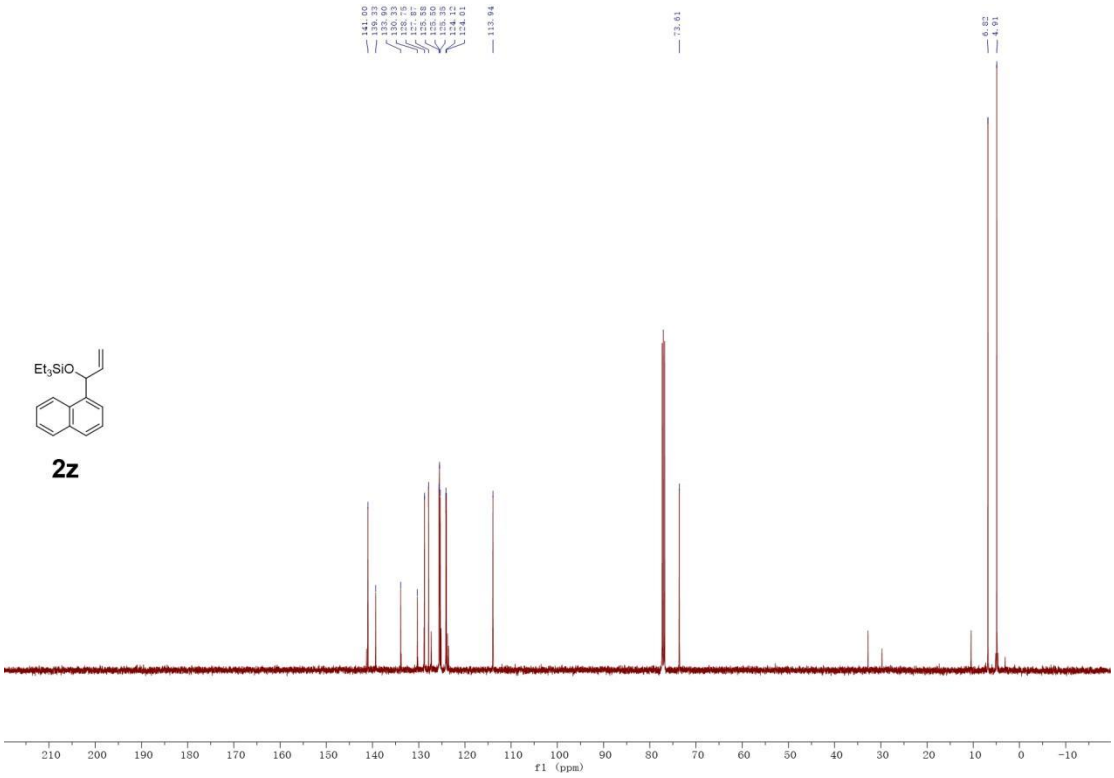

7.83
7.82
7.81
7.79
7.78
7.48
7.47
7.46
7.45
7.44
7.43
7.43
6.05
6.03
6.02
6.01
6.00
5.98
5.96
5.36
5.35
5.33
5.31
5.31
5.12
5.11
5.10
5.09
5.09
0.96
0.94
0.92
0.66
0.65
0.64
0.63
0.62
0.61
0.60
0.59
OSiEt3
2y
4.35
3.33
0.96
1.88
1.00
9.30
6.13
f1 (ppm)

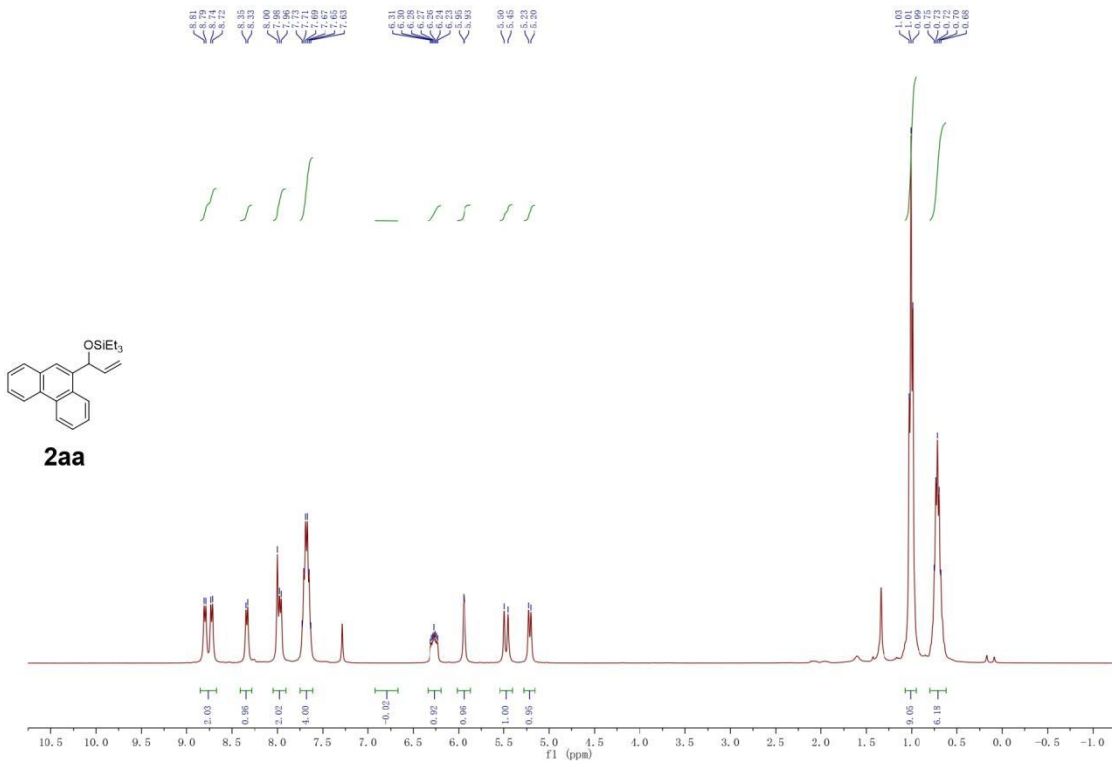

OSiEt3
141.56
141.23
133.24
132.03
131.99
127.93
125.68
125.50
125.62
124.63
124.41
113.78
78.82
6.84
4.93
f1 (ppm)

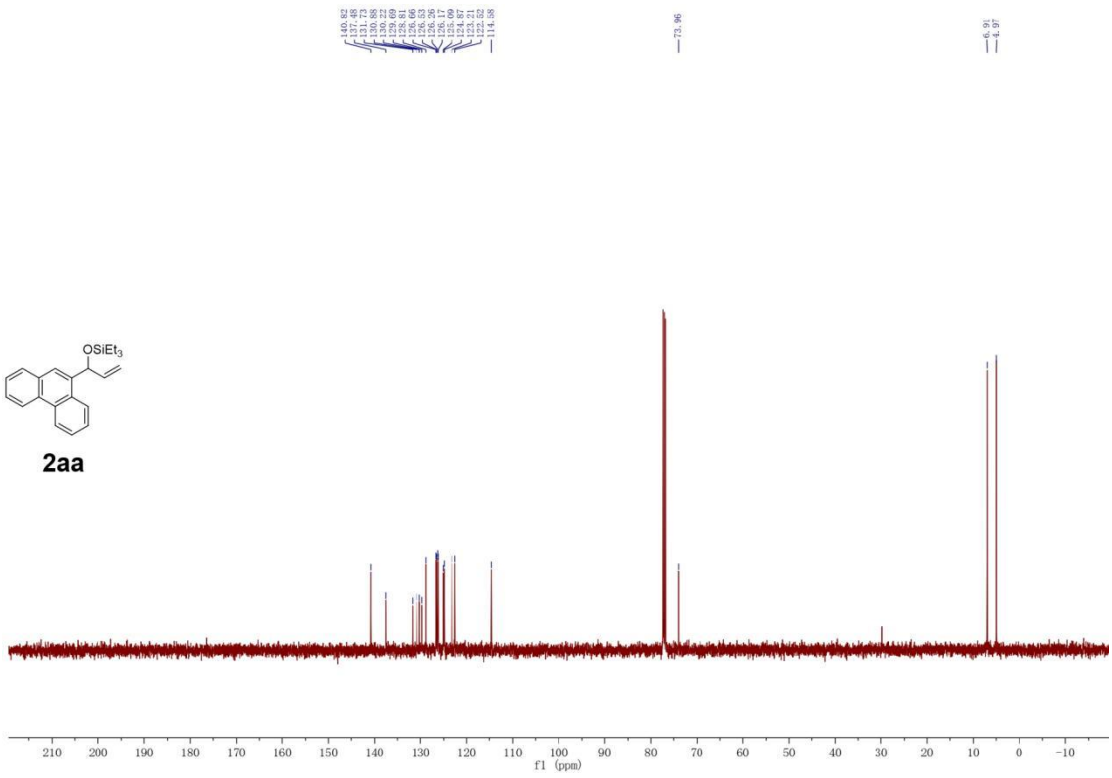
2y

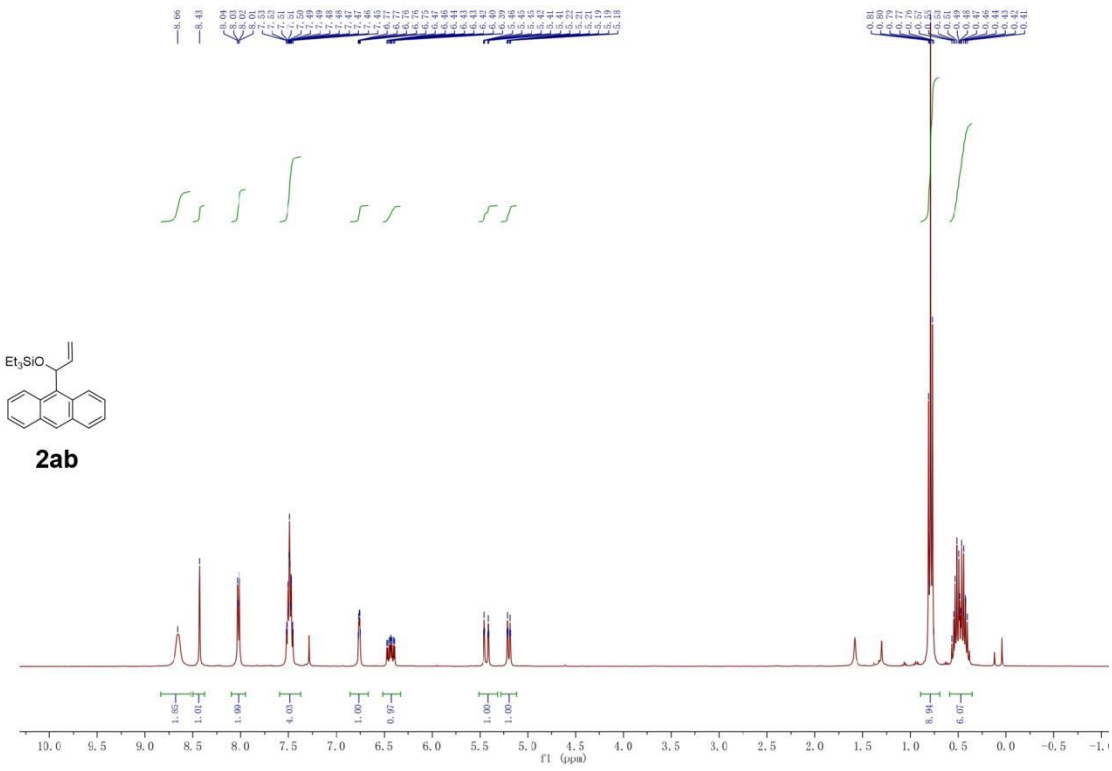

Et3SiO
f1 (ppm)

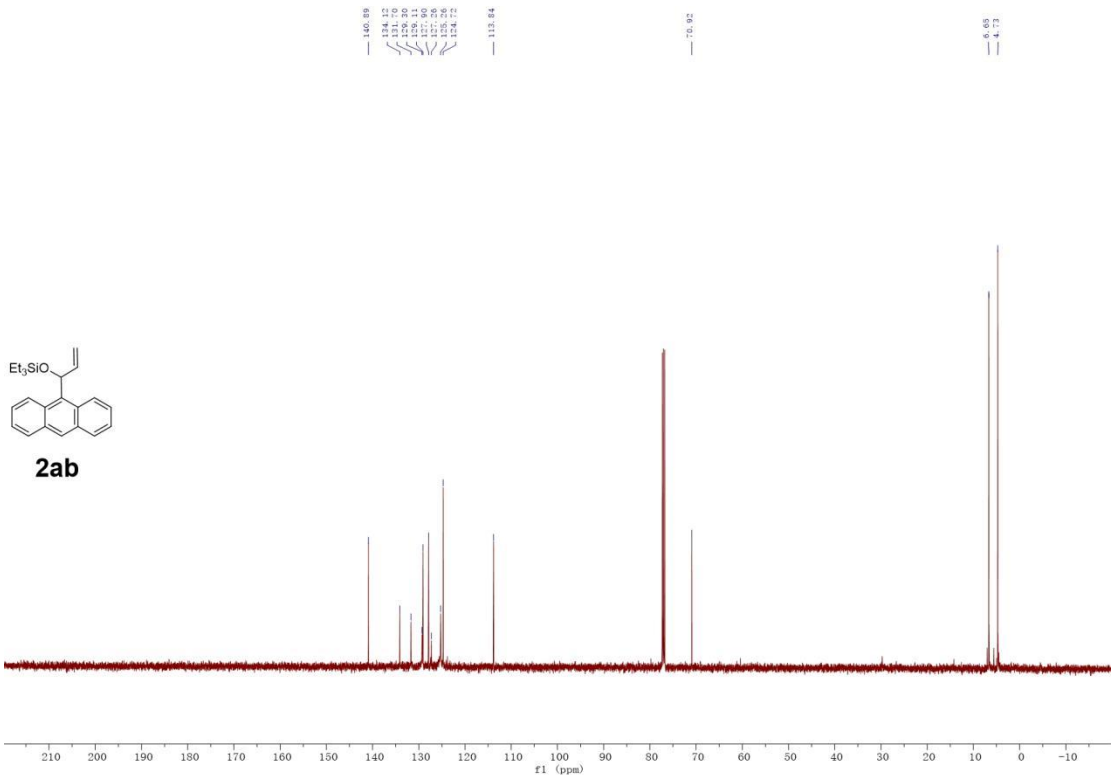
2z

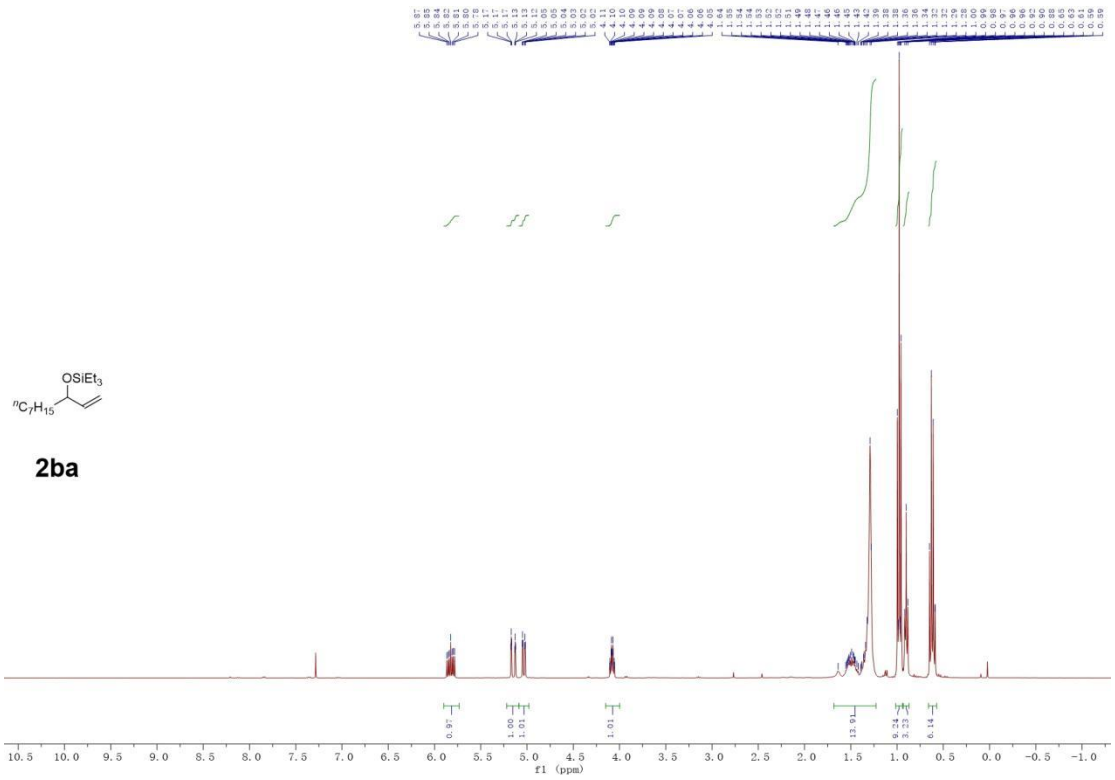

Et3SiO
2z
141.00
139.33
132.90
130.33
128.75
127.87
125.58
125.50
125.35
124.12
124.01
113.94
73.61
6.82
4.91
f1 (ppm)

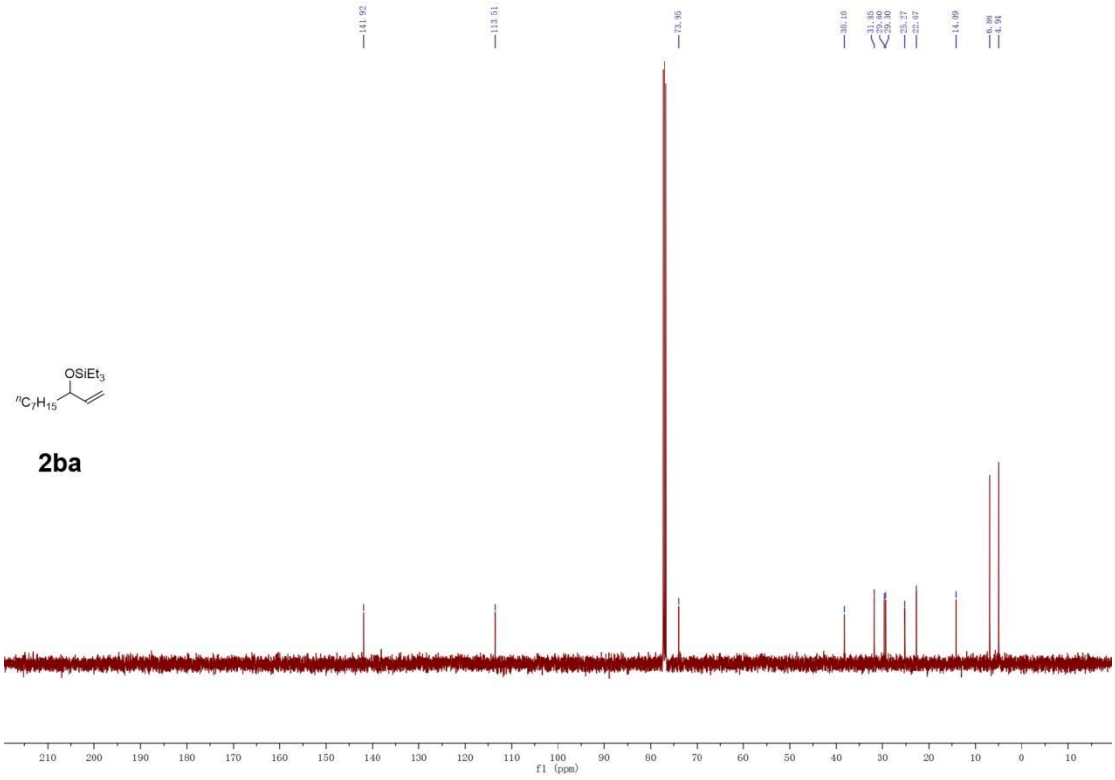

OSiEt3
2aa
8.81
8.79
8.74
8.72
8.35
8.33
8.00
7.98
7.96
7.73
7.71
7.69
7.67
7.65
7.63
6.31
6.30
6.28
6.27
6.26
6.24
6.23
5.95
5.93
5.60
5.45
5.23
5.20
1.03
1.01
0.99
0.73
0.72
0.70
0.68
2.03
0.96
2.02
4.00
0.02
0.92
0.86
1.00
0.95
9.05
6.18
f1 (ppm)


OSiEt3
2aa
f1 (ppm)

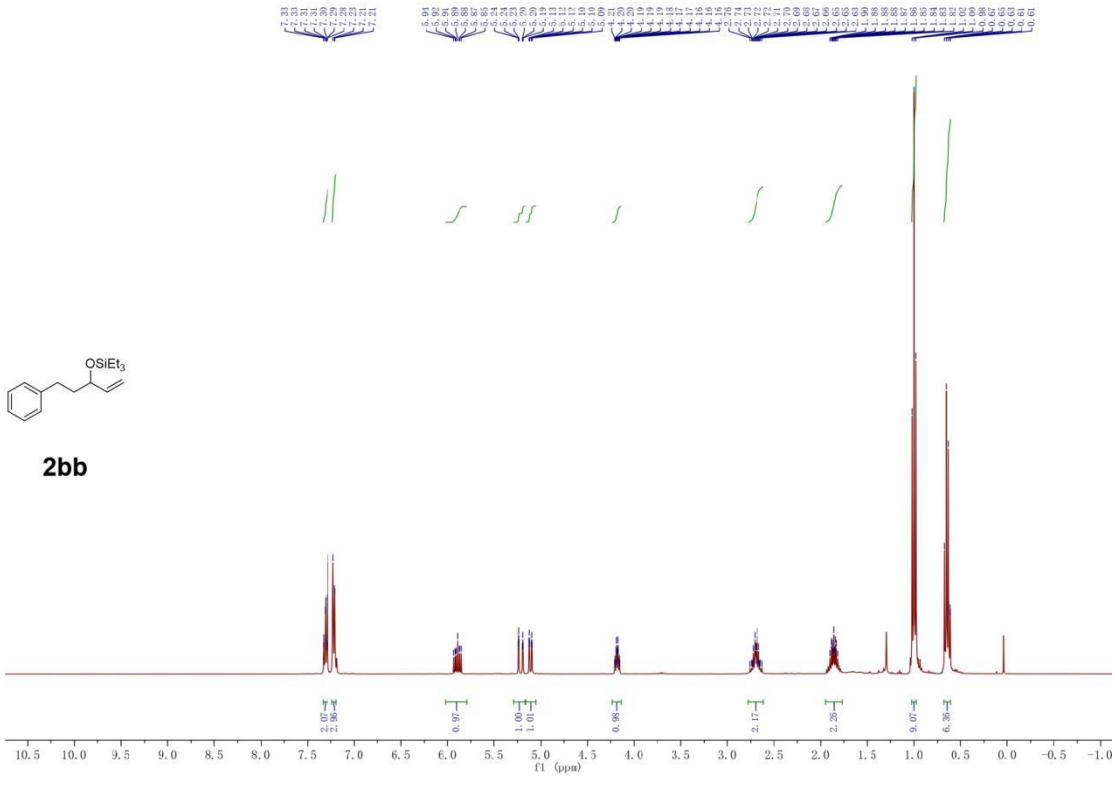

Et3SiO
2ab
f1 (ppm)


Et3SiO
2ab
f1 (ppm)

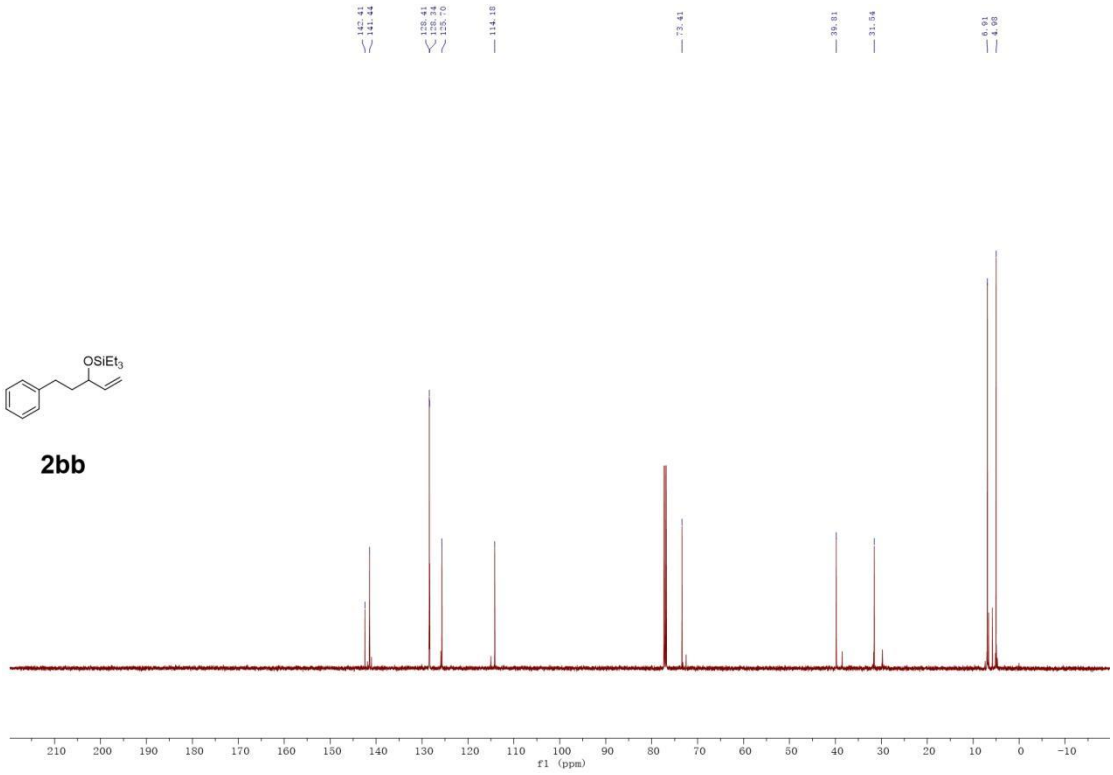

OSiEt3
nC7H15
2ba
f1 (ppm)

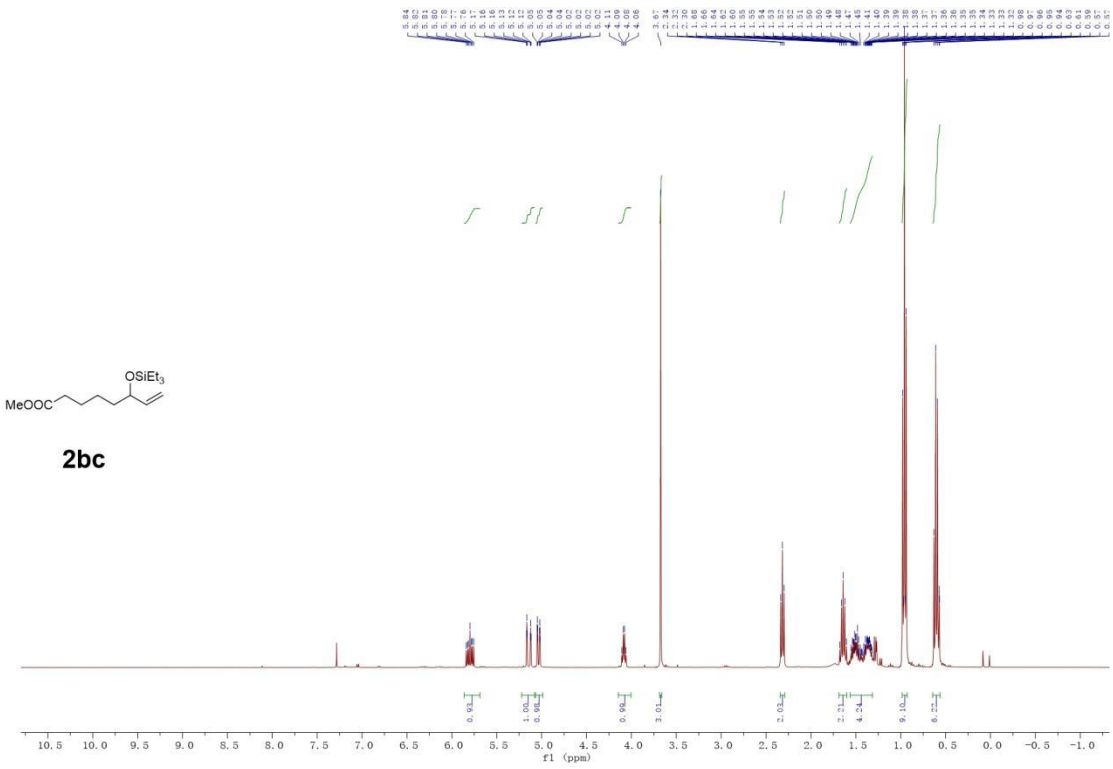

141.92
113.51
73.93
38.18
31.85
29.60
29.30
25.27
22.67
14.09
6.89
4.94
OSiEt3
nC7H15
f1 (ppm)

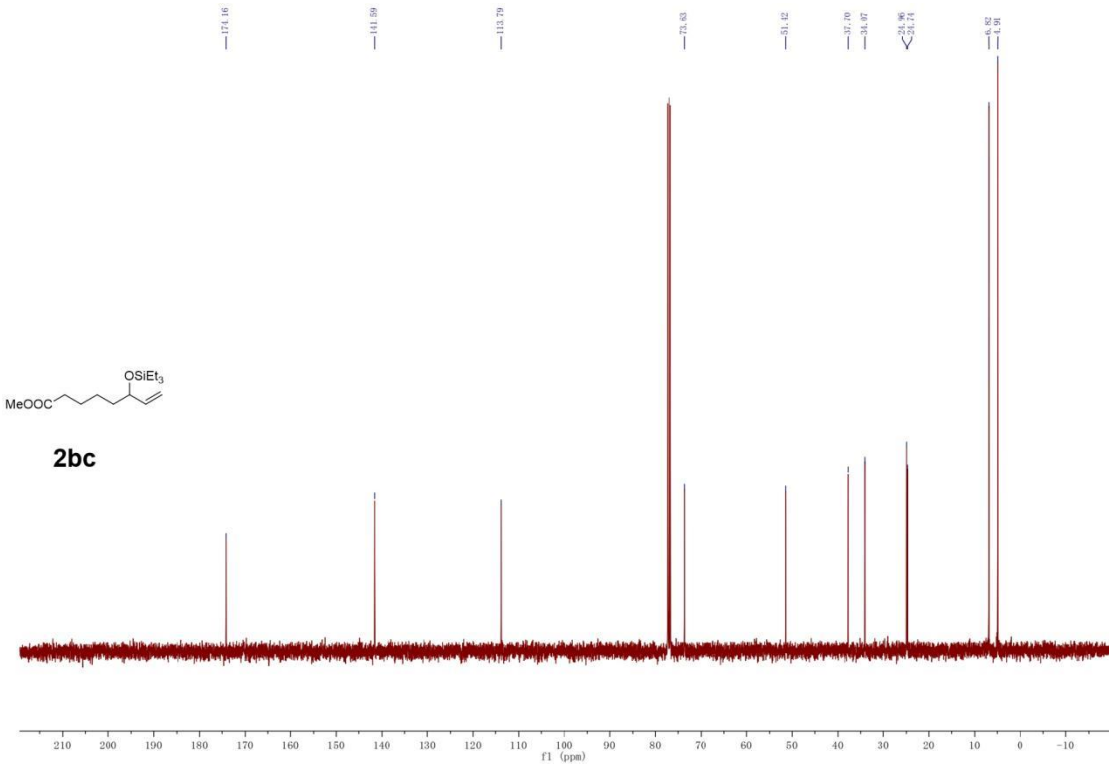
2ba

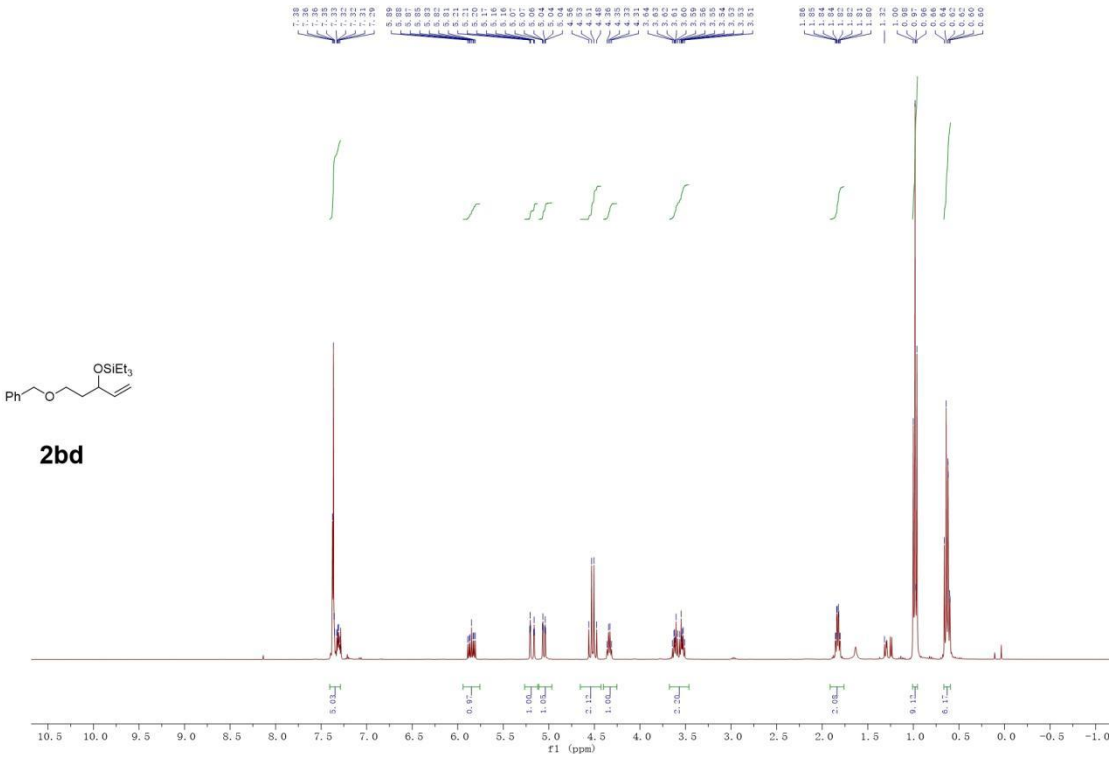

OSiEt3
f1 (ppm)

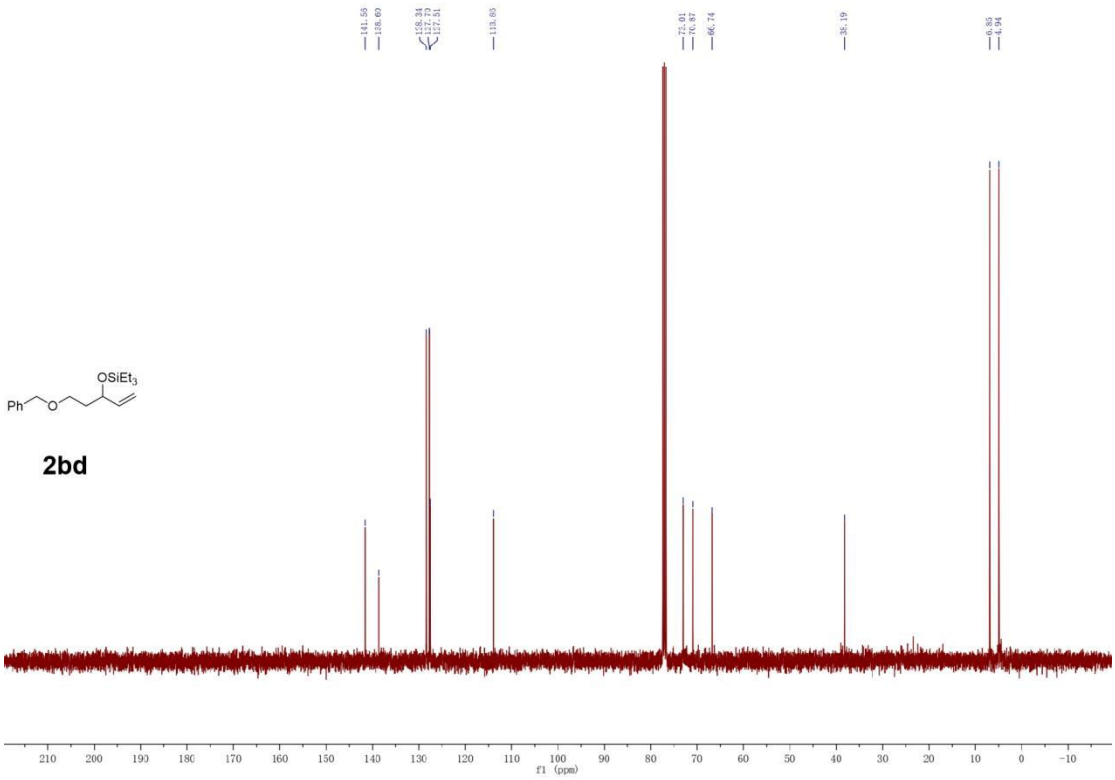
2bb

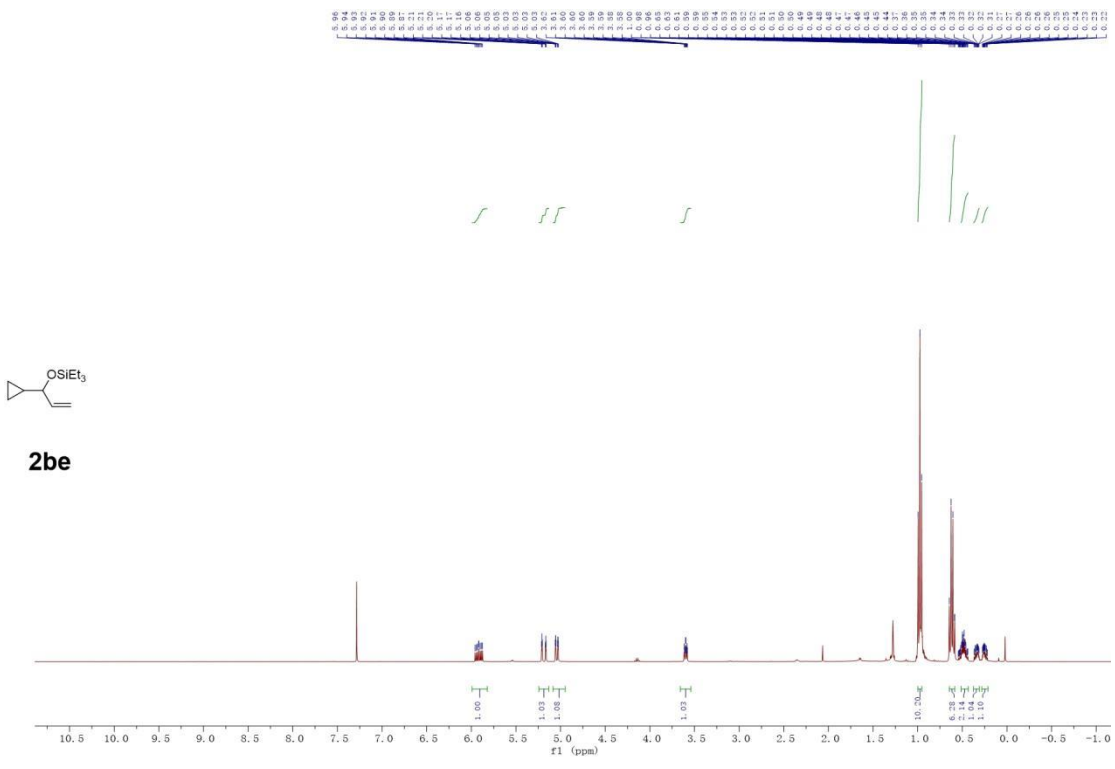

142.41
141.44
128.41
128.34
125.70
114.18
73.41
39.81
31.64
6.91
4.98
OSiEt3
2bb
f1 (ppm)

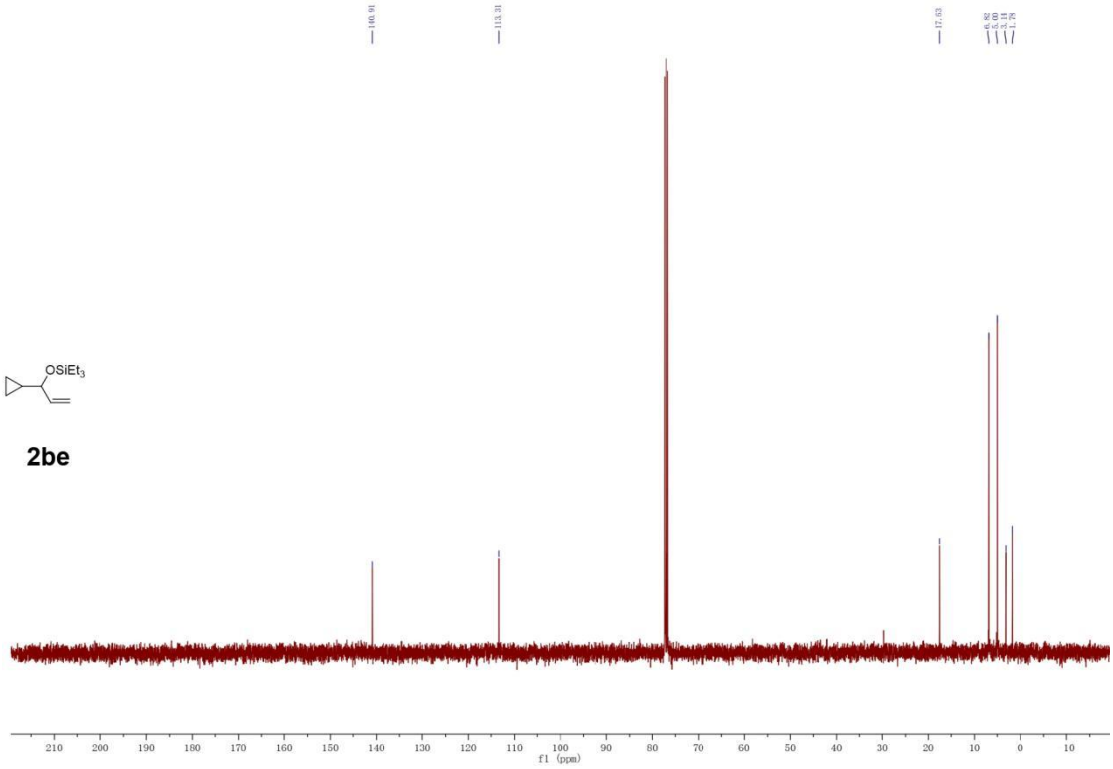

OSiEt3
MeOOC
2bc
f1 (ppm)

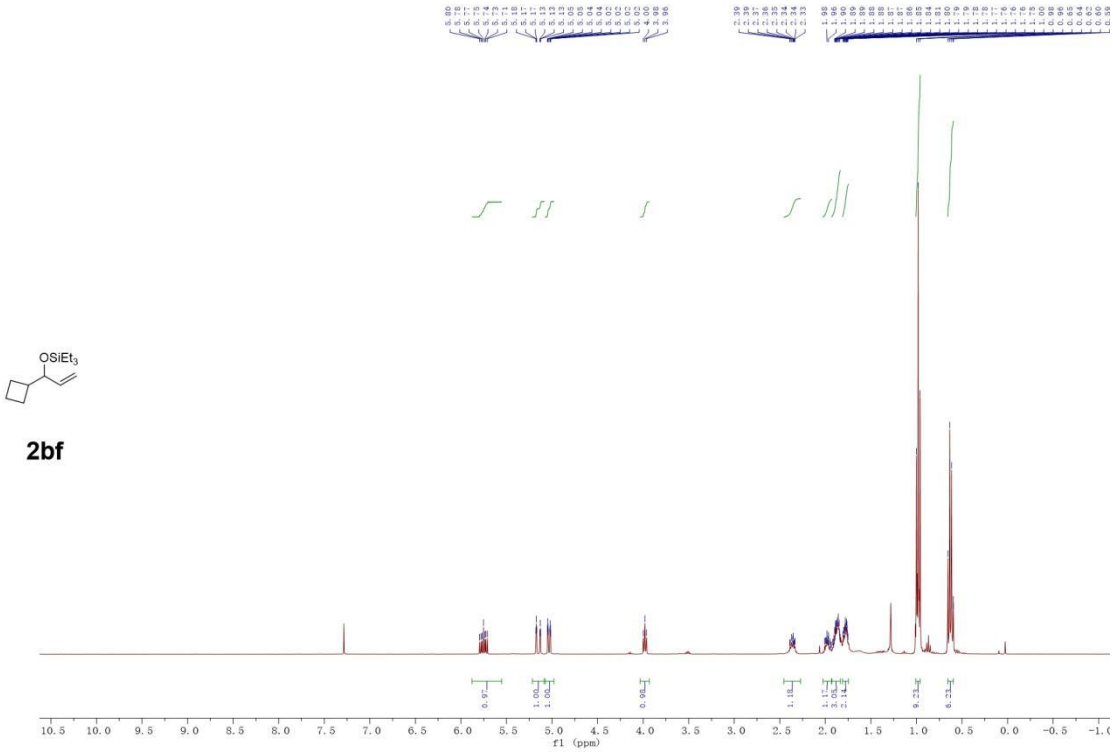

174.16
141.59
113.79
73.63
51.42
37.70
34.07
24.96
24.74
6.82
4.91
OSiEt3
MeOOC
2bc
f1 (ppm)

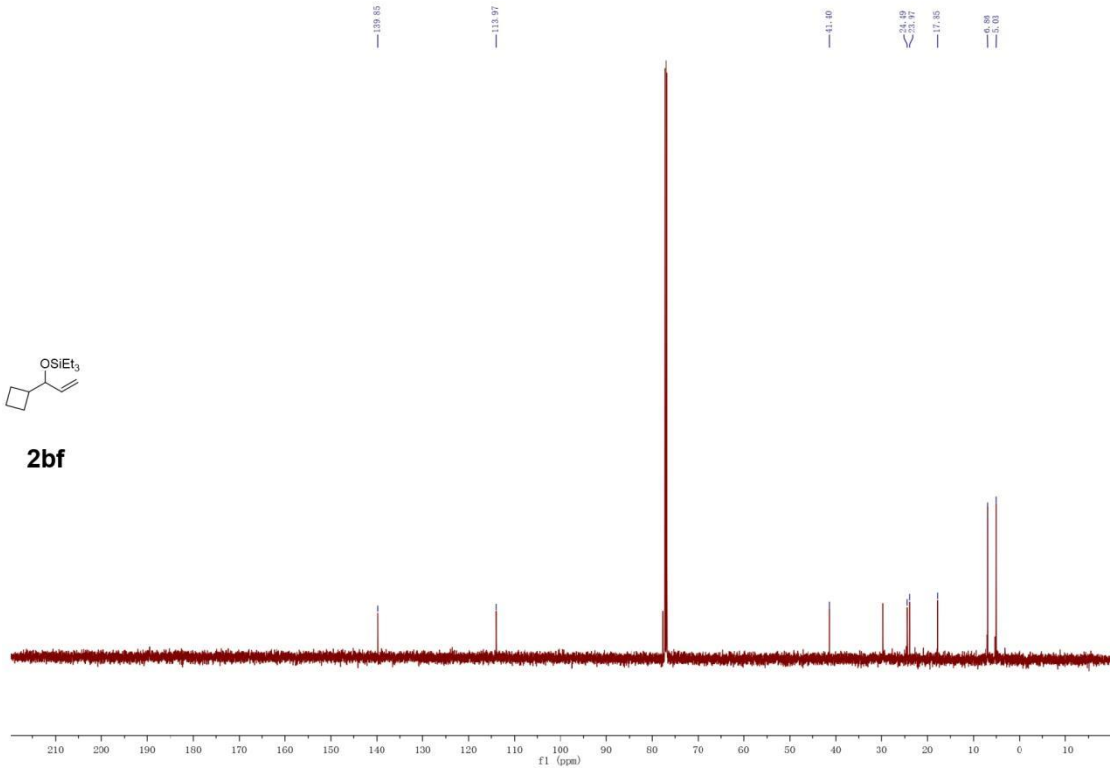

OSiEt3
Ph
O
2bd
f1 (ppm)

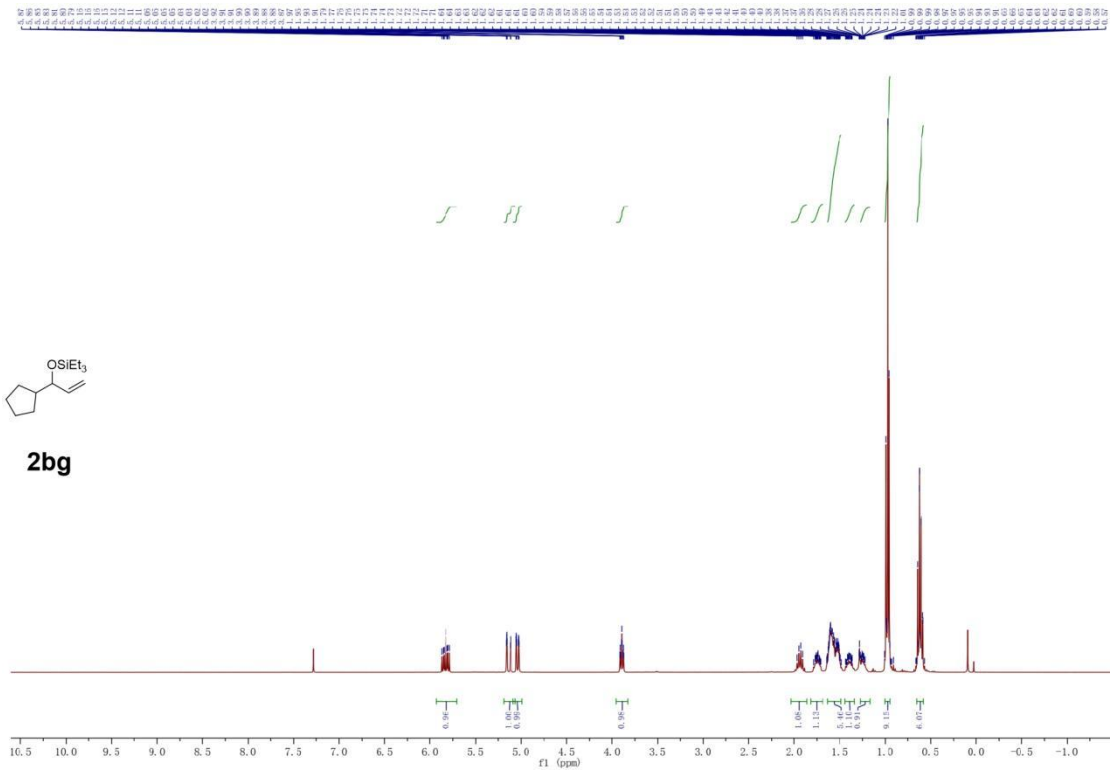

OSiEt3
Ph
O
2bd
141.53
126.60
128.34
127.73
127.51
113.85
73.01
70.87
66.74
36.19
6.85
4.94
f1 (ppm)

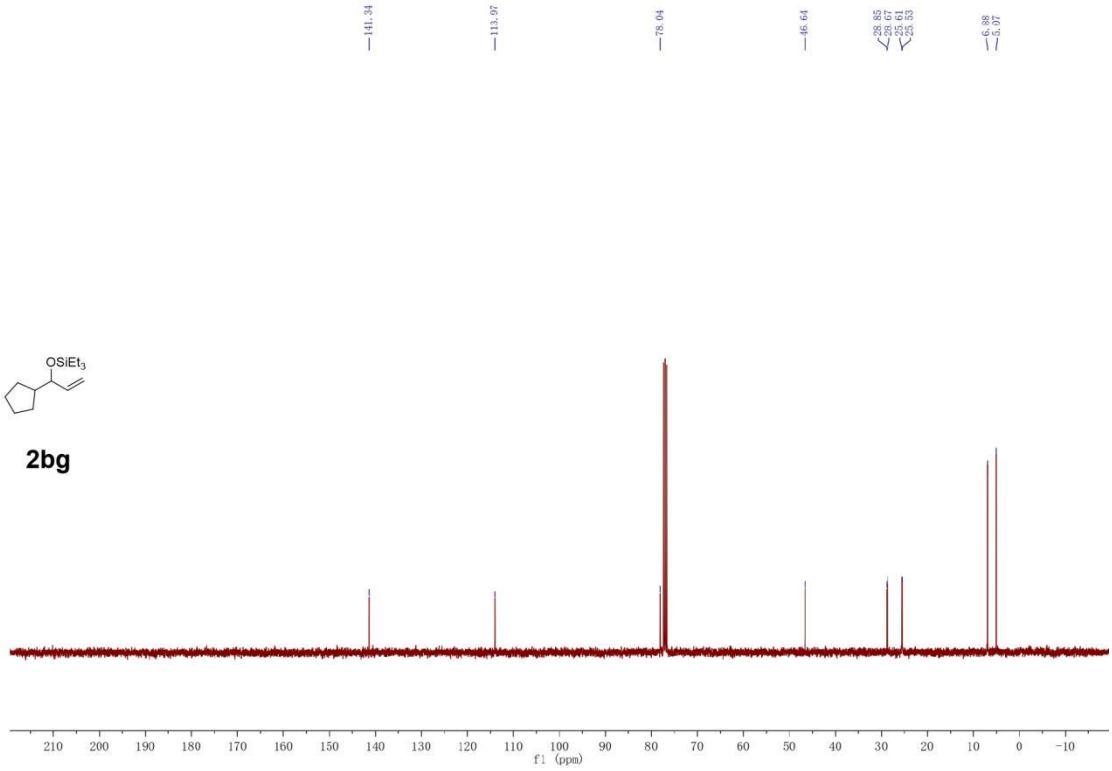

OSiEt3
2be
f1 (ppm)

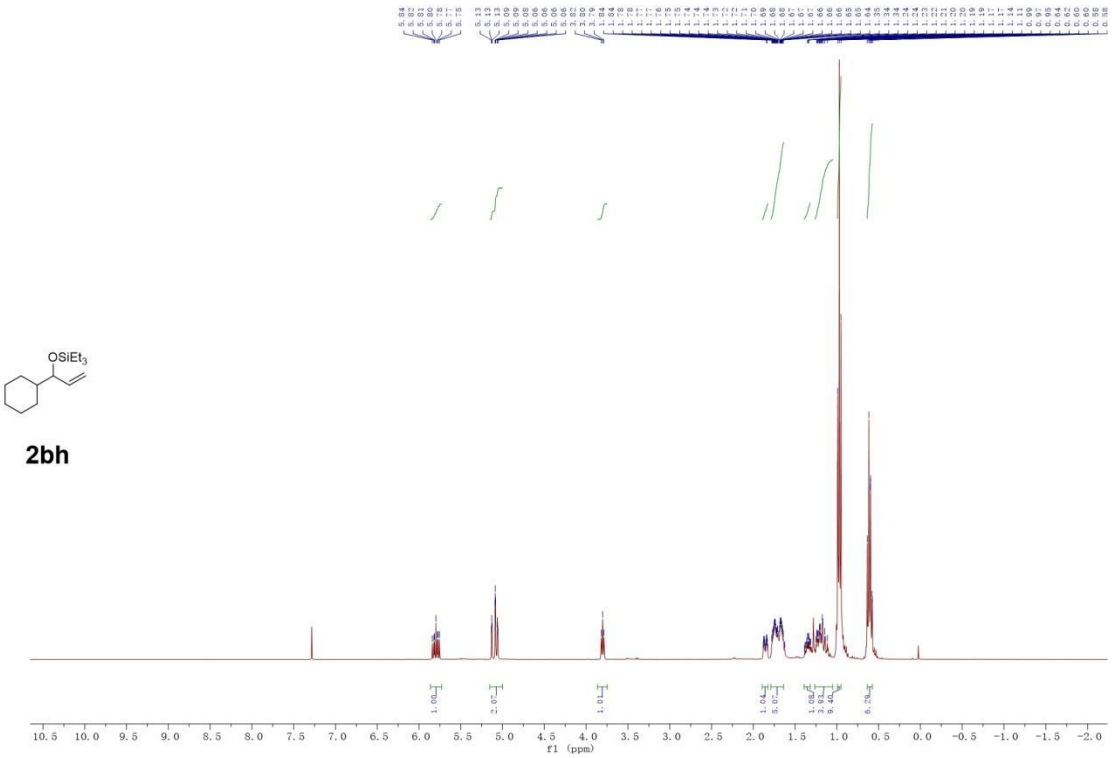

OSiEt3
2be
f1 (ppm)

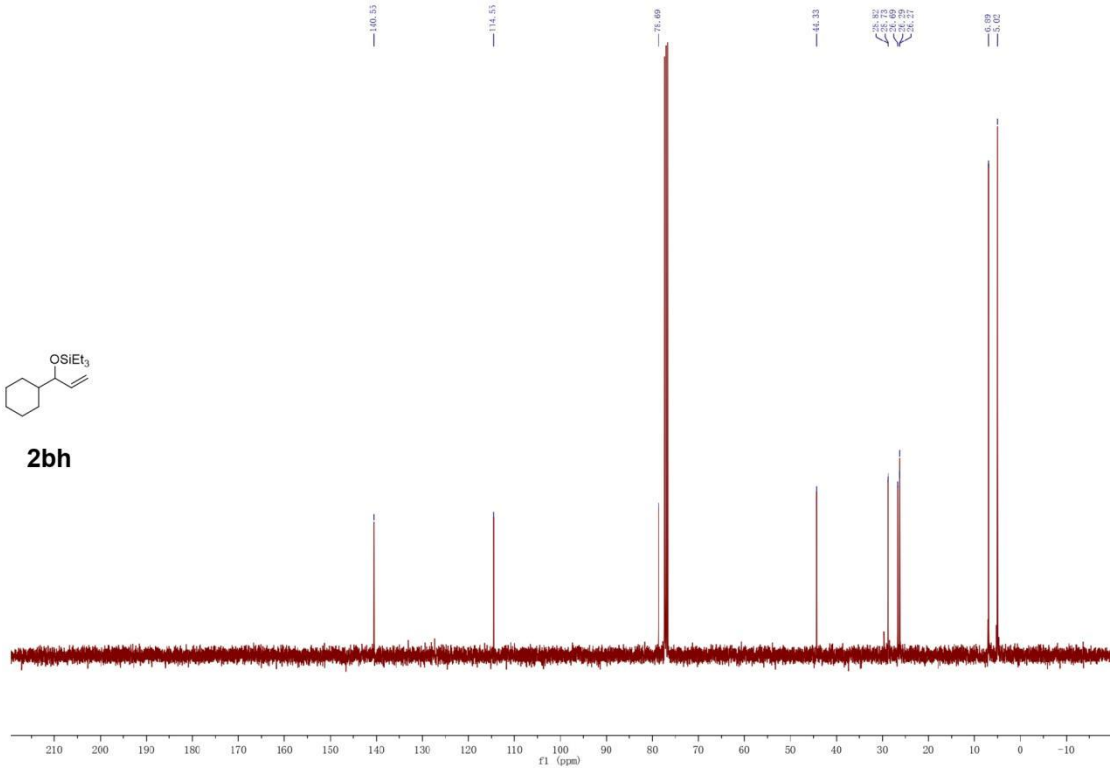

OSiEt3
2bf
f1 (ppm)

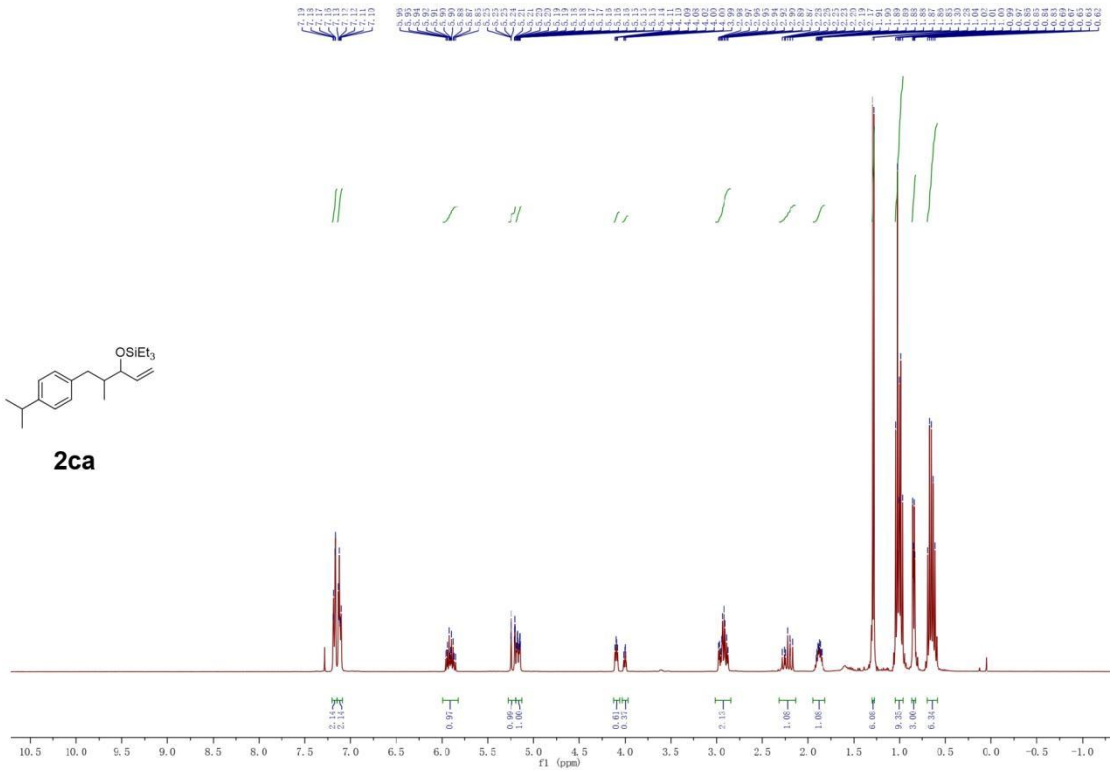

139.85
113.97
41.40
24.49
23.97
17.85
6.88
5.03
OSiEt3
2bf
f1 (ppm)

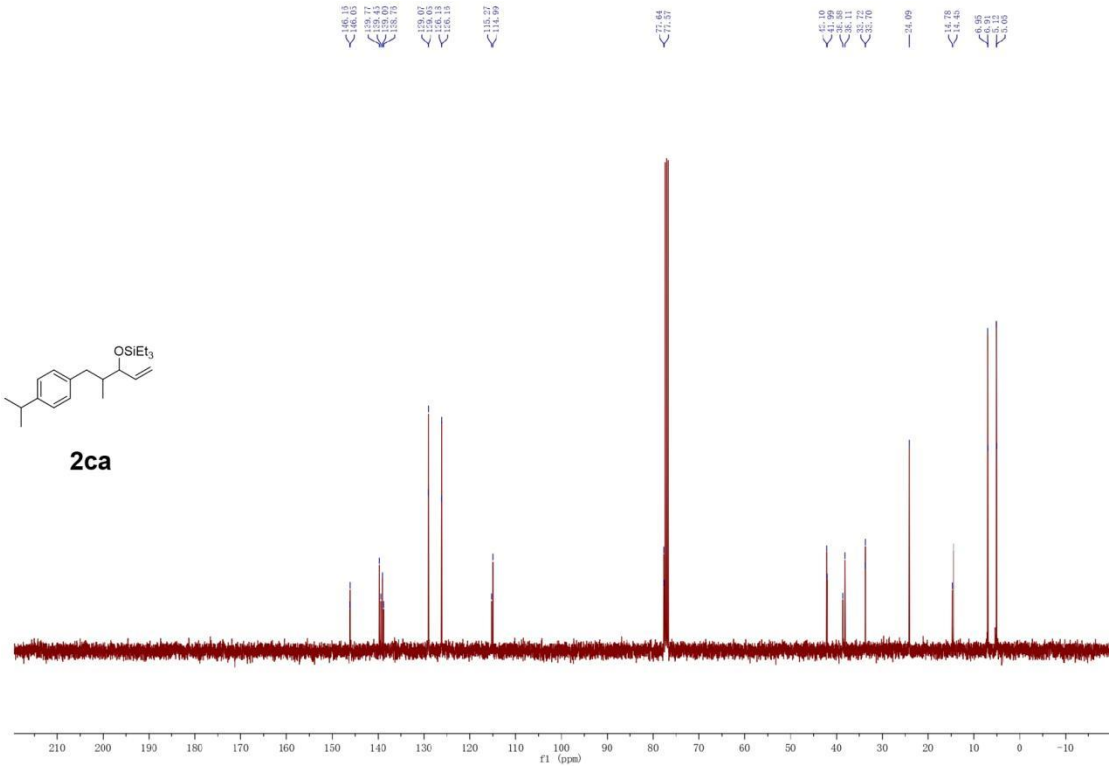

OSiEt3
2bg
f1 (ppm)

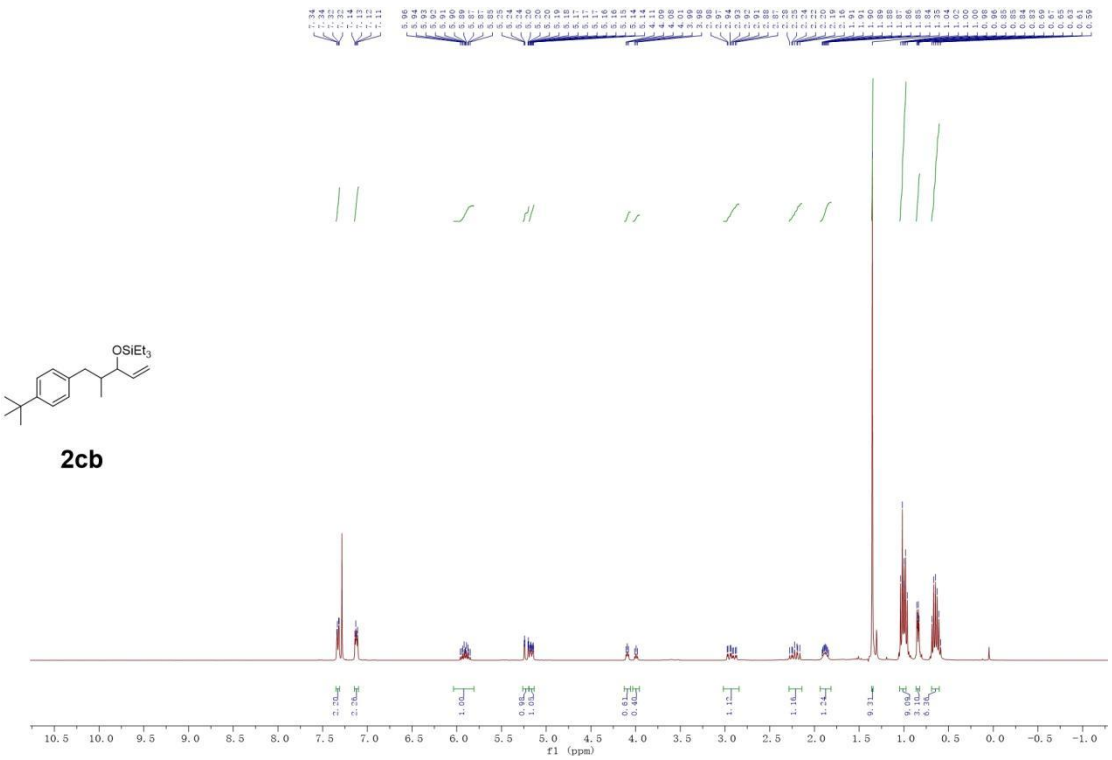

141.34
113.97
78.04
46.64
28.85
28.67
25.61
25.53
6.88
5.97
OSiEt3
2bg
f1 (ppm)

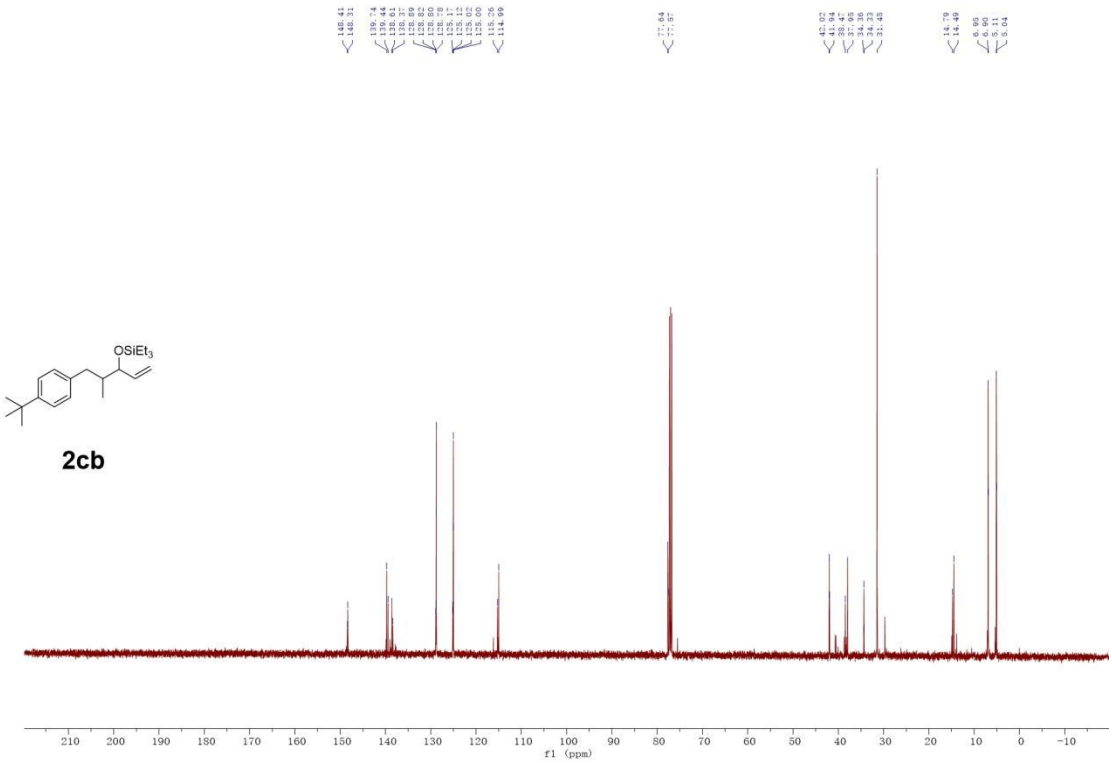

OSiEt3
2bh
f1 (ppm)

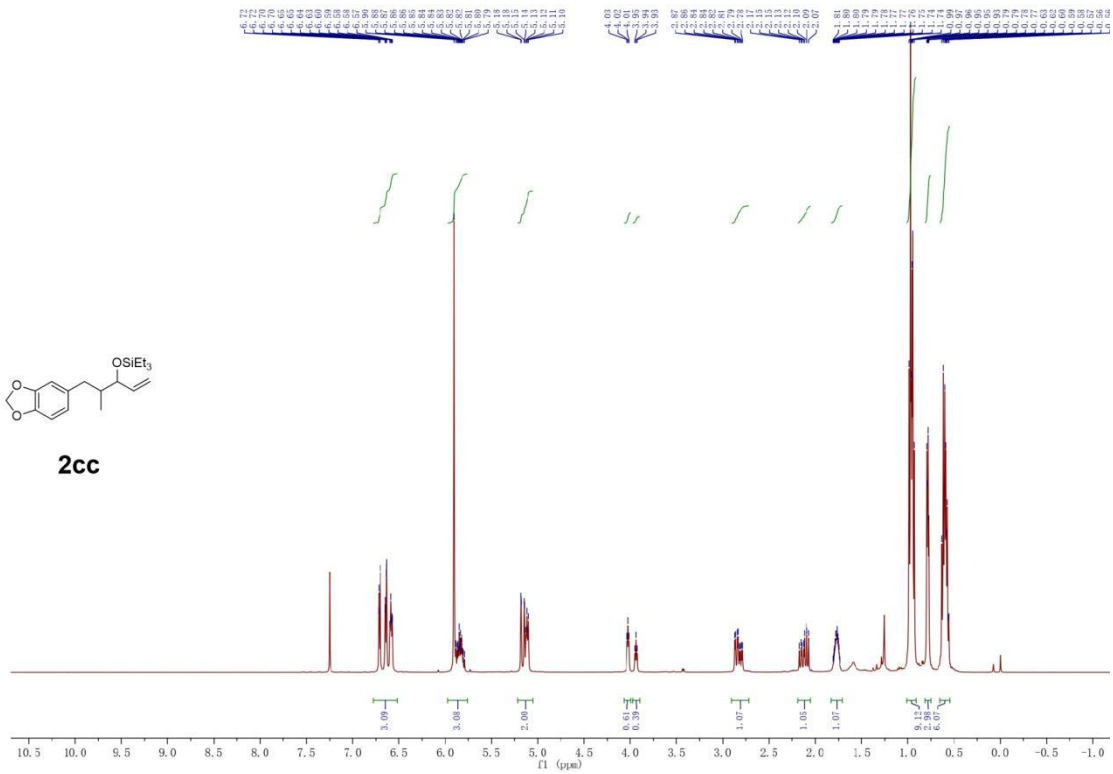

OSiEt3
2bh
140.55
114.55
76.69
44.33
28.82
25.73
25.69
25.29
24.27
6.89
5.02
f1 (ppm)

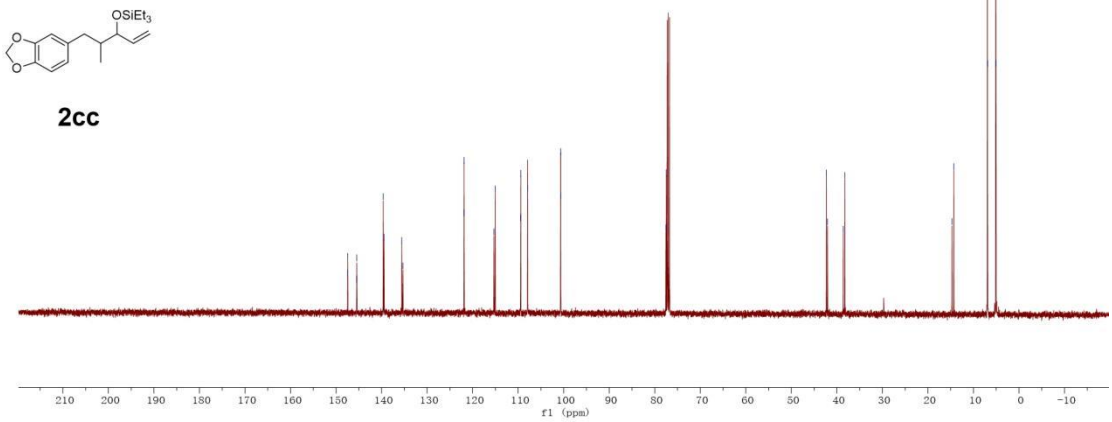

OSiEt3
2ca
f1 (ppm)

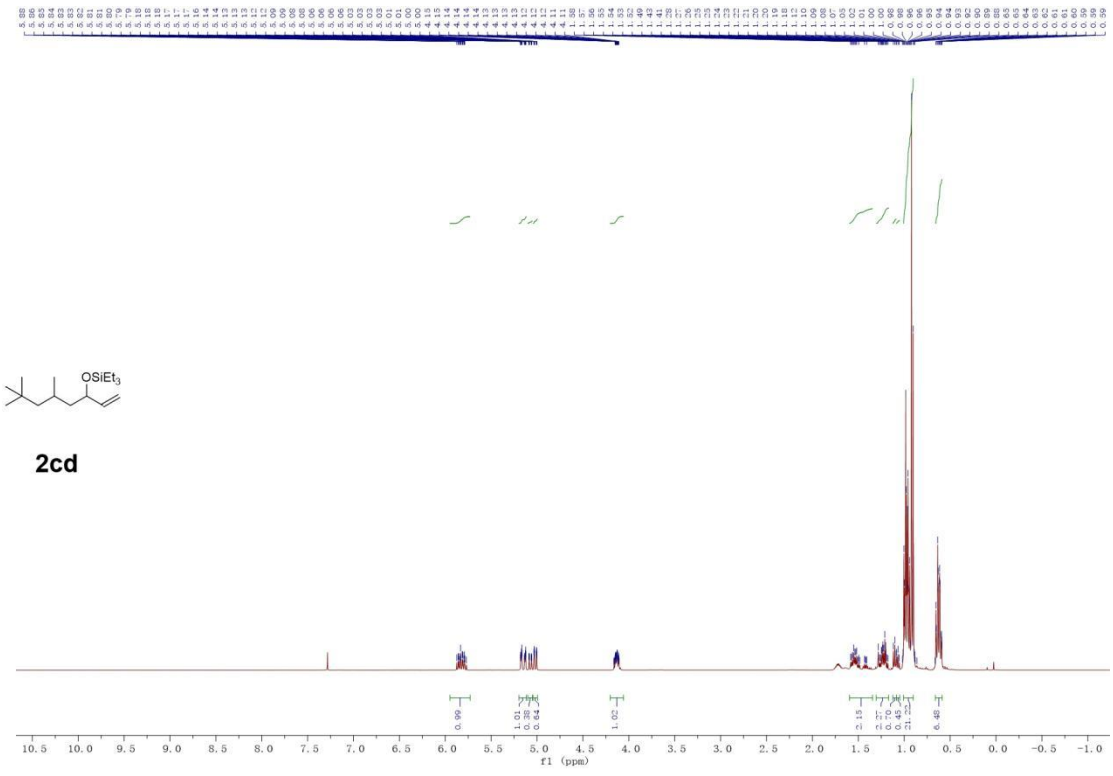

OSiEt3
2ca
f1 (ppm)

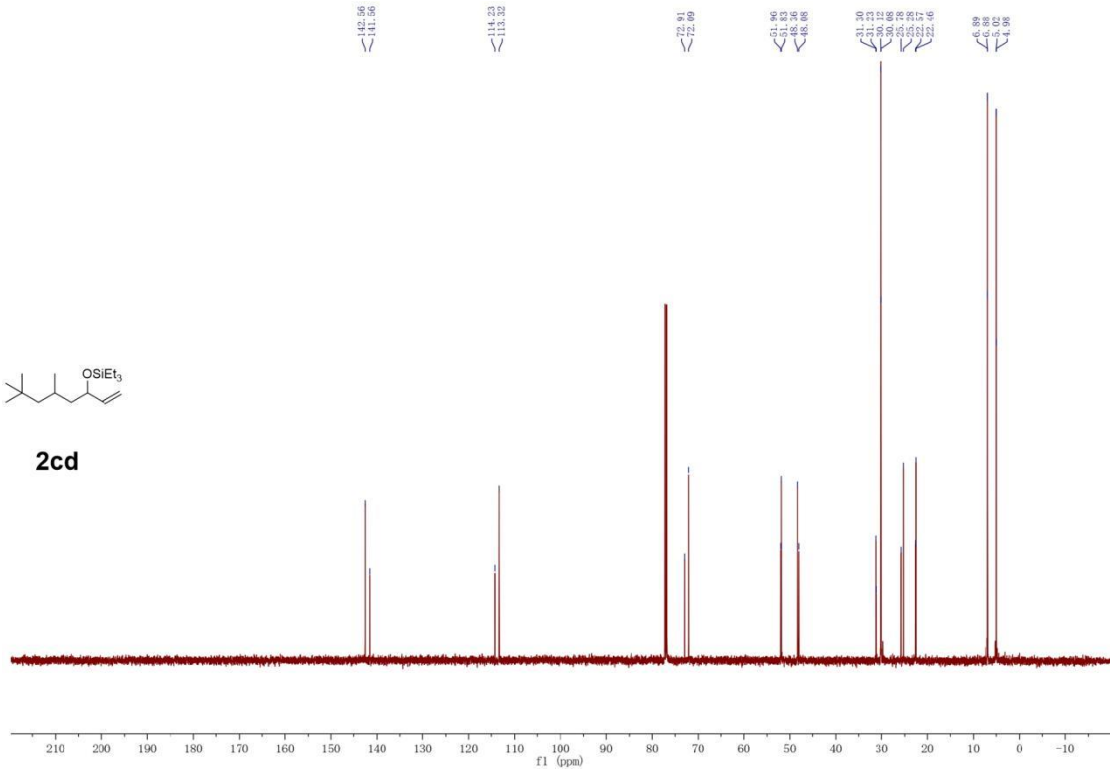

OSiEt3
2cb
f1 (ppm)

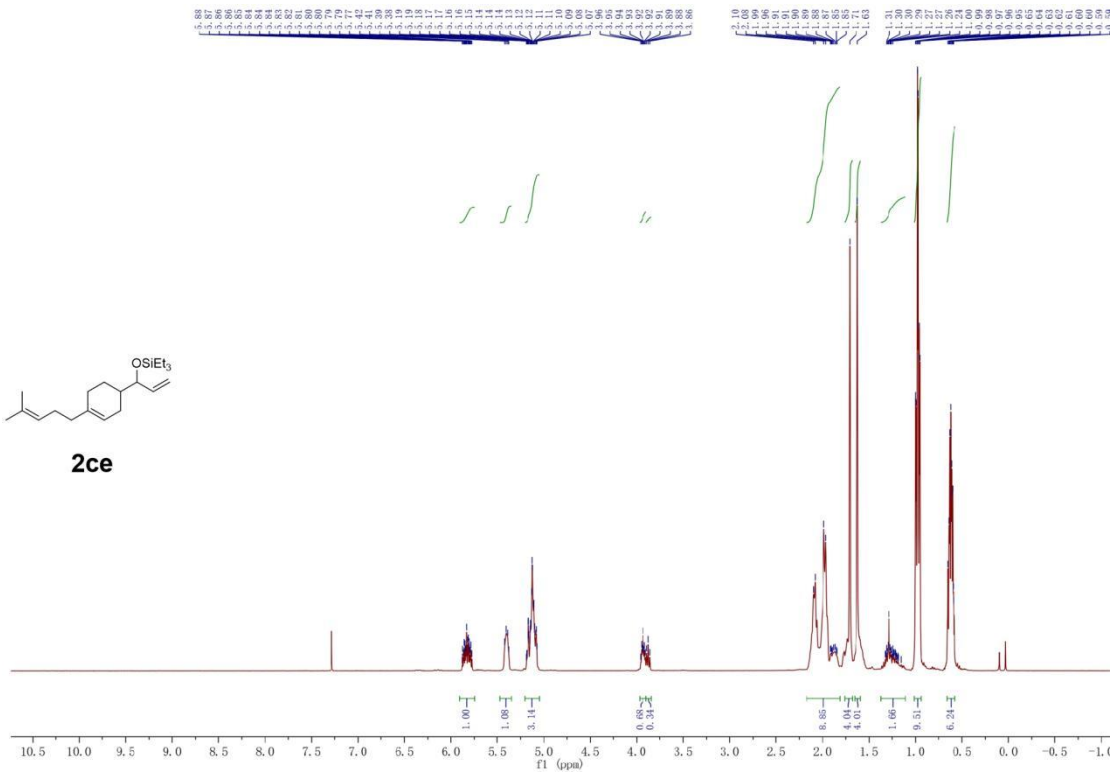

OSiEt3
2cb
f1 (ppm)

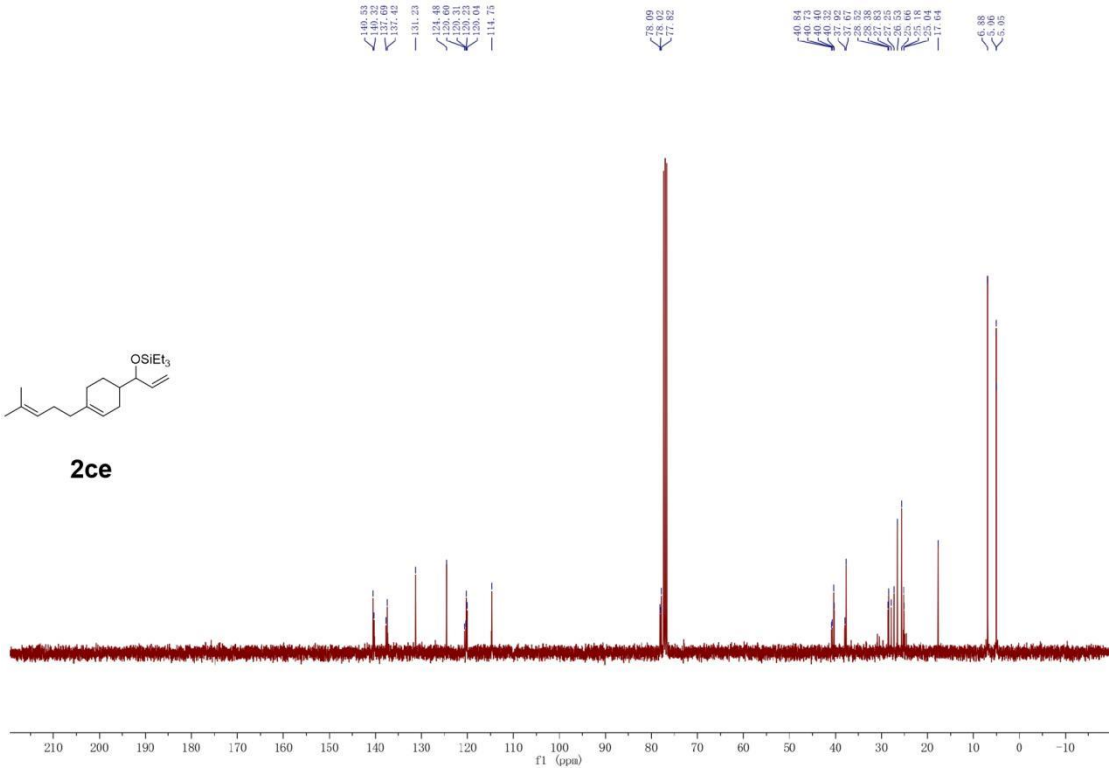

OSiEt3
2cc
f1 (ppm)

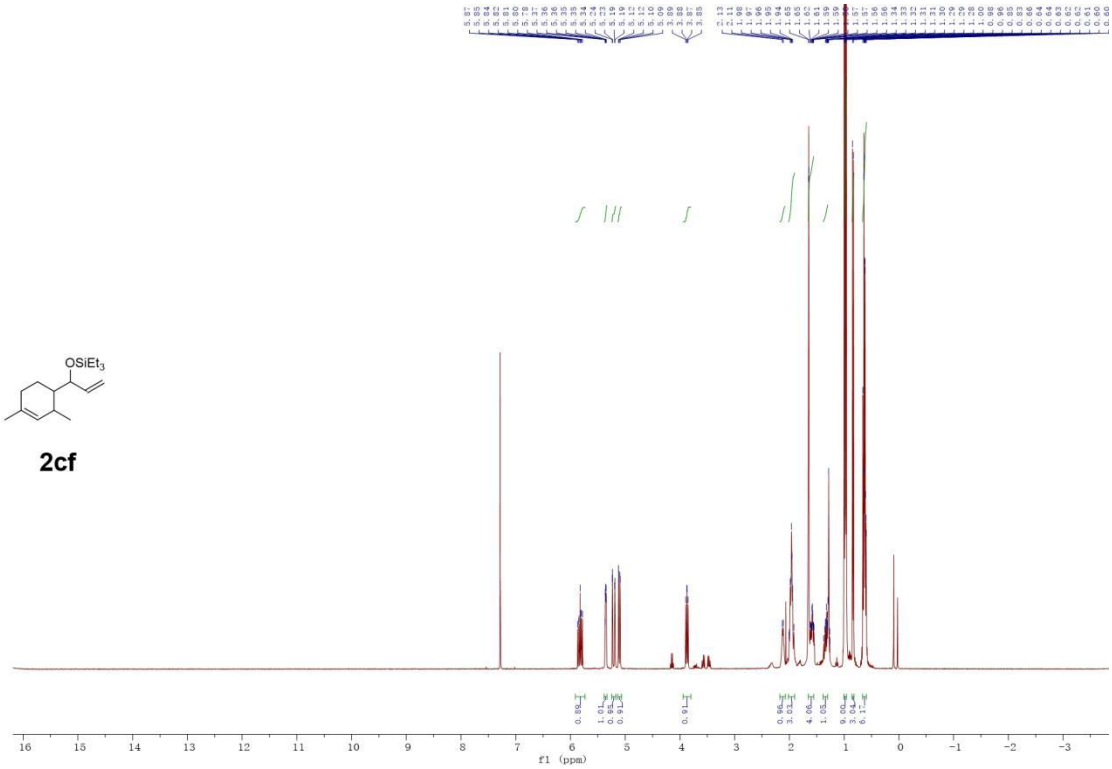

OSiEt3
2cc
f1 (ppm)

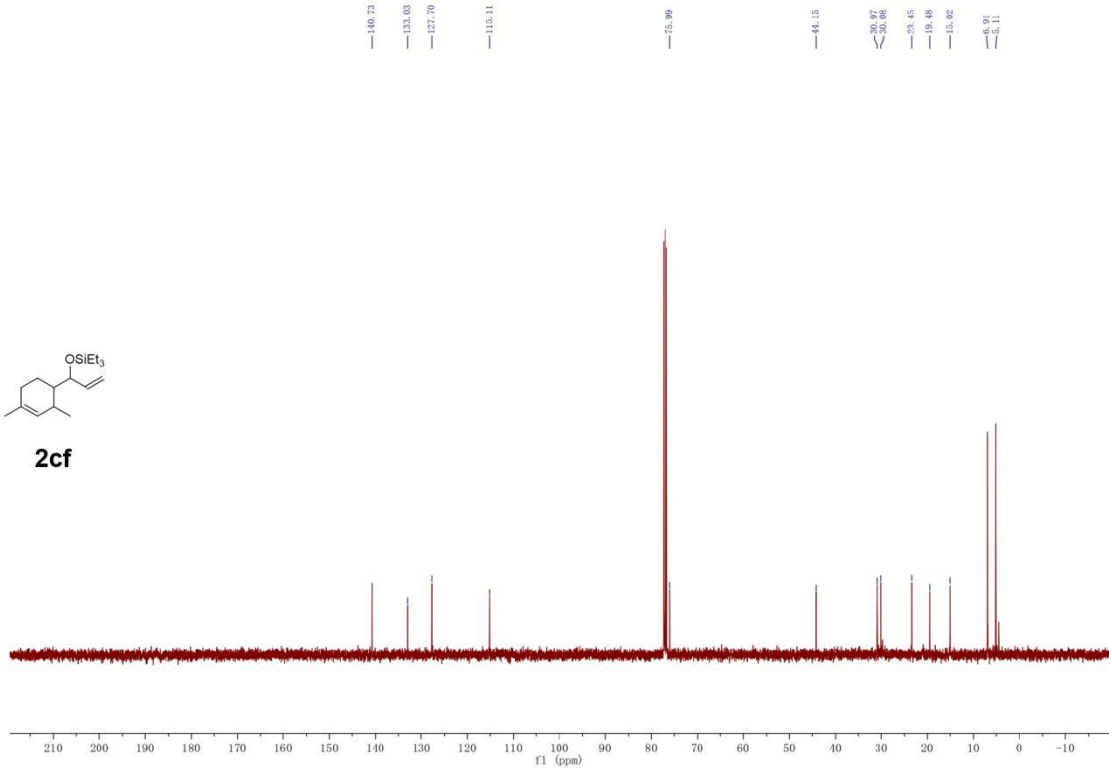

OSiEt3
2cd
f1 (ppm)

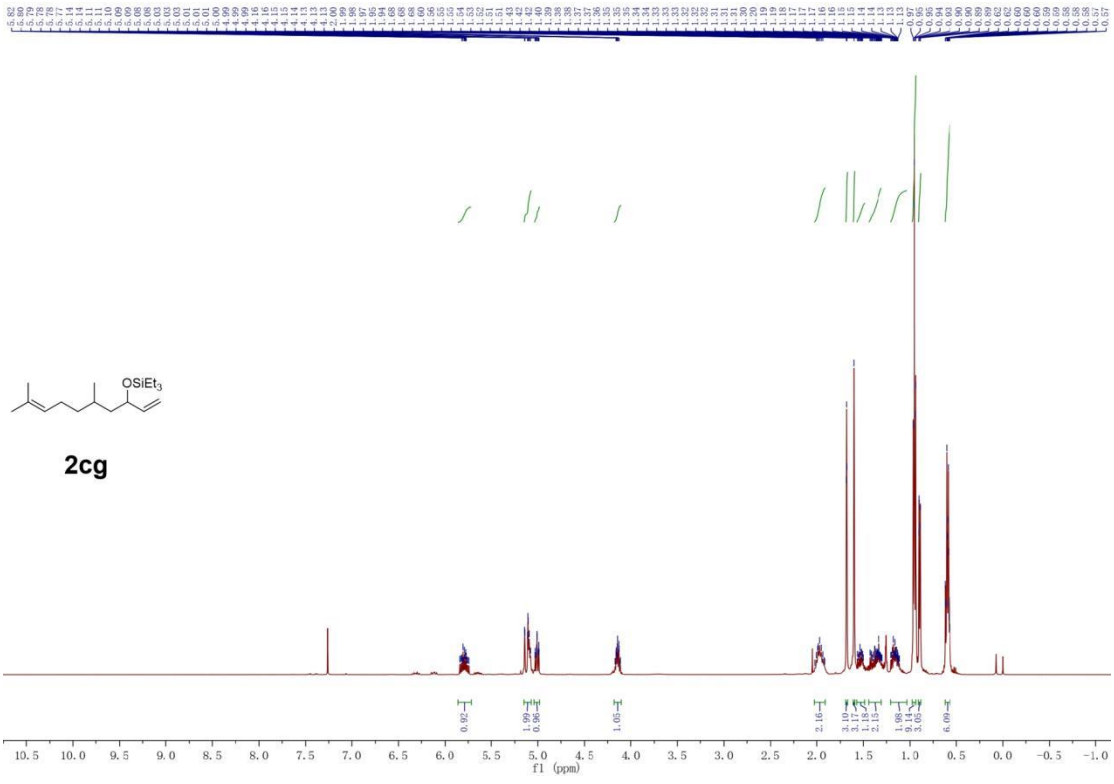

OSiEt3
2cd
142.56
141.56
114.23
113.32
72.91
72.69
61.96
61.83
48.36
48.08
31.20
31.23
30.32
30.08
25.78
25.38
22.57
22.46
6.89
6.88
6.02
4.98
f1 (ppm)

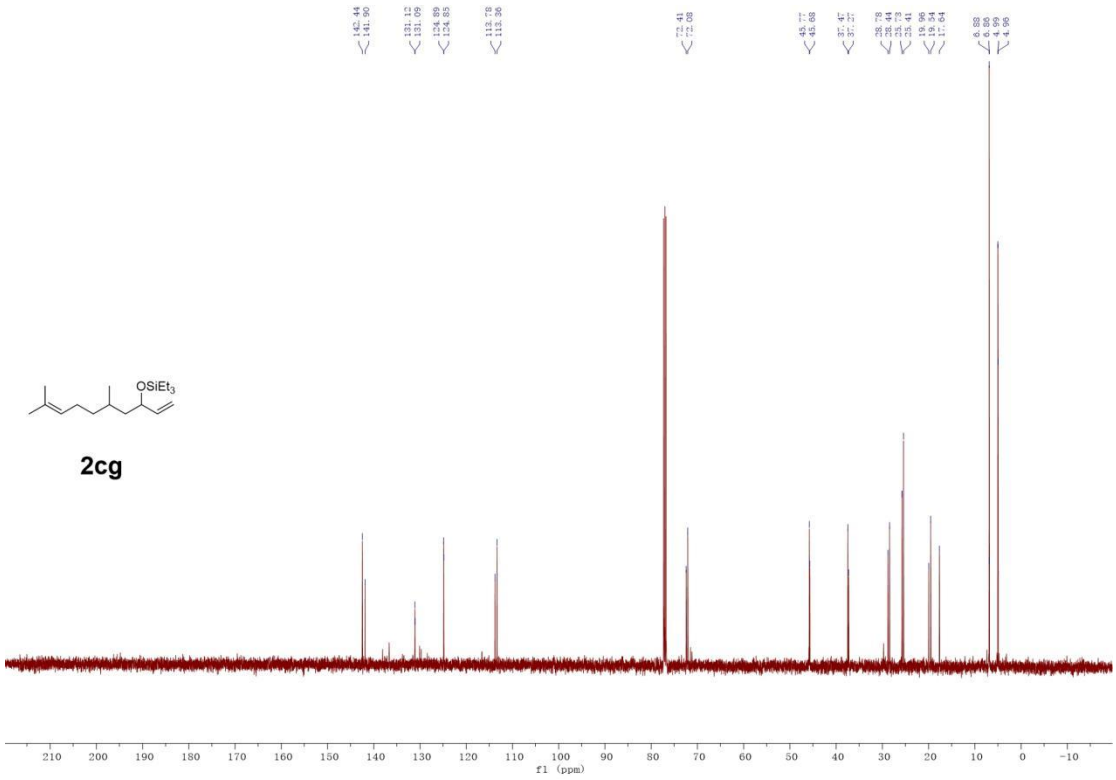

OSiEt3
2ce
1.00
1.08
3.14
0.68
0.34
8.85
4.04
4.01
1.66
9.51
6.24
f1 (ppm)

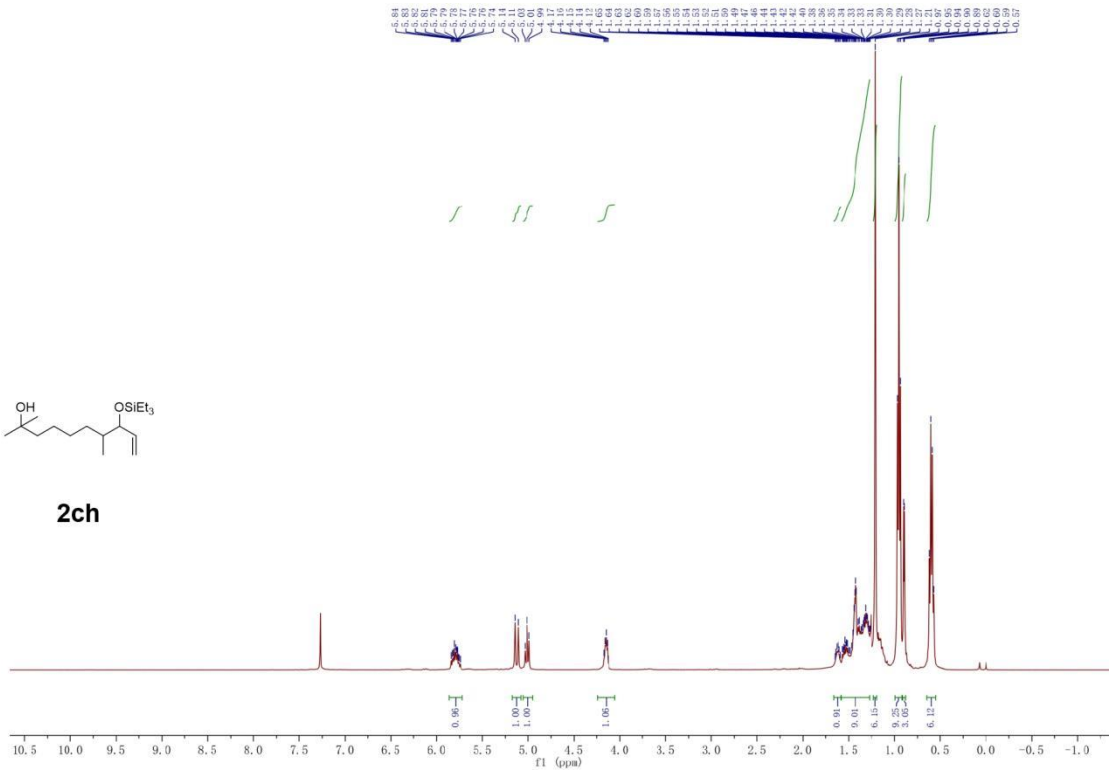

OSiEt3
2ce
f1 (ppm)

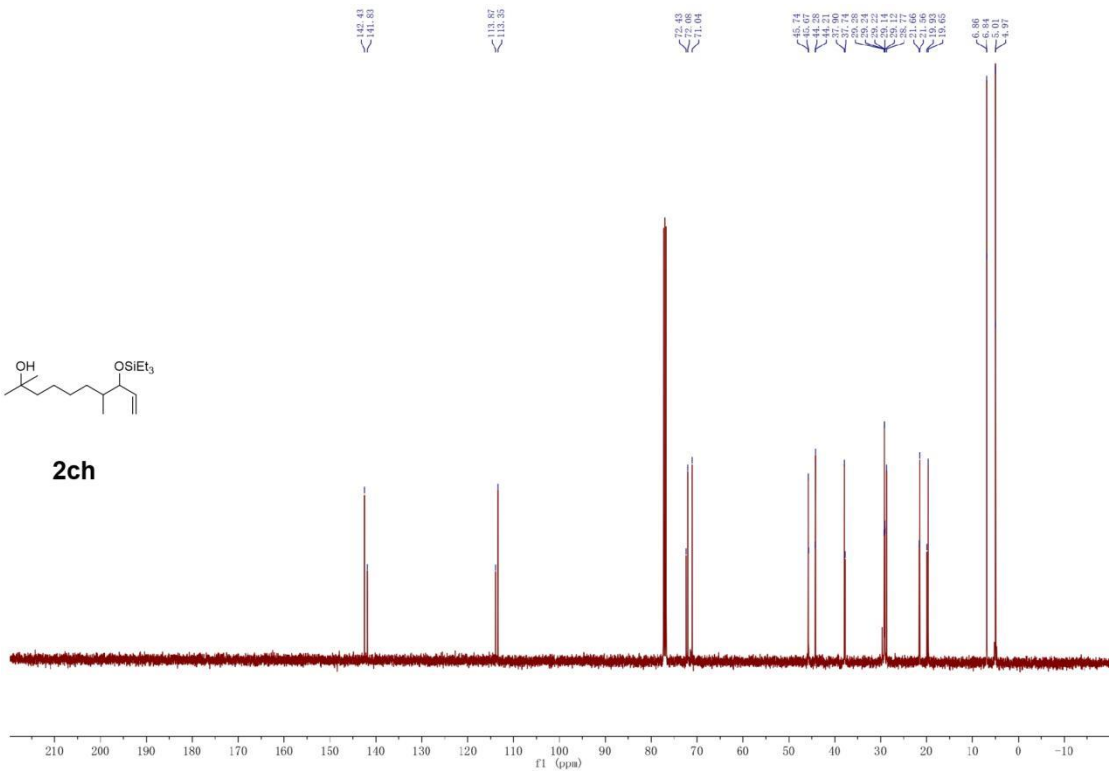

OSiEt3
2cf
f1 (ppm)

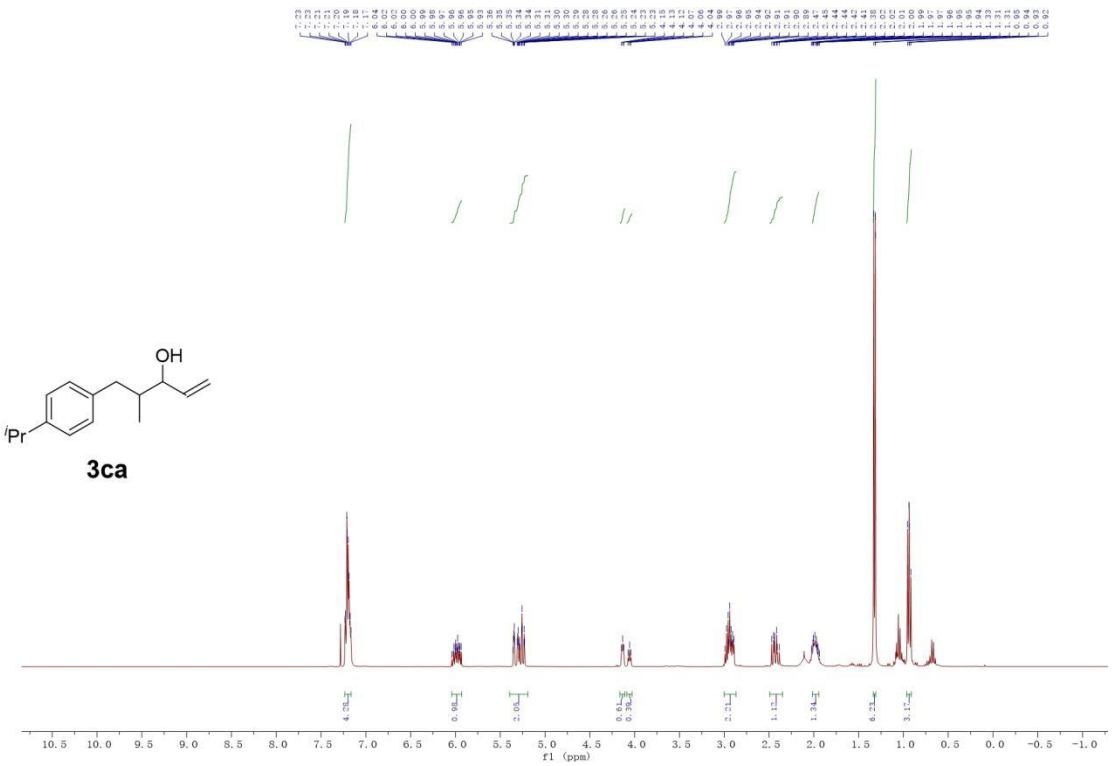

140.73
133.03
127.70
116.11
75.99
44.15
30.97
30.08
23.45
19.48
15.02
6.91
5.11
OSiEt3
2cf
f1 (ppm)

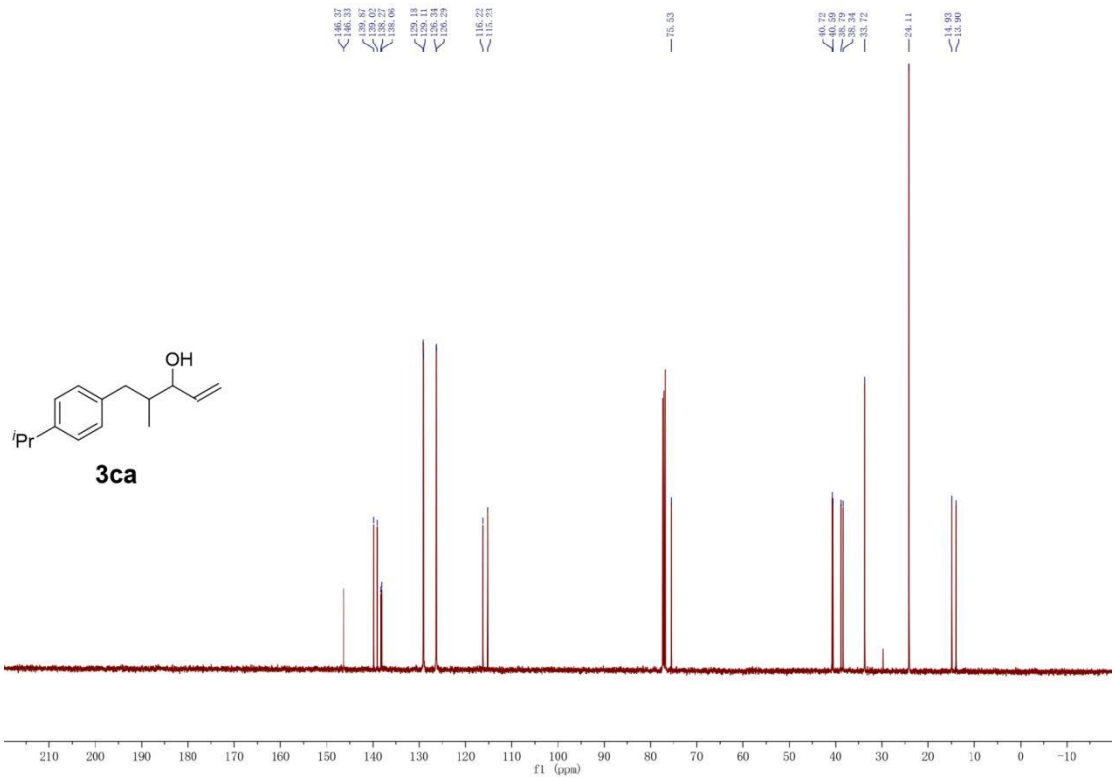

OSiEt3
2cg
f1 (ppm)

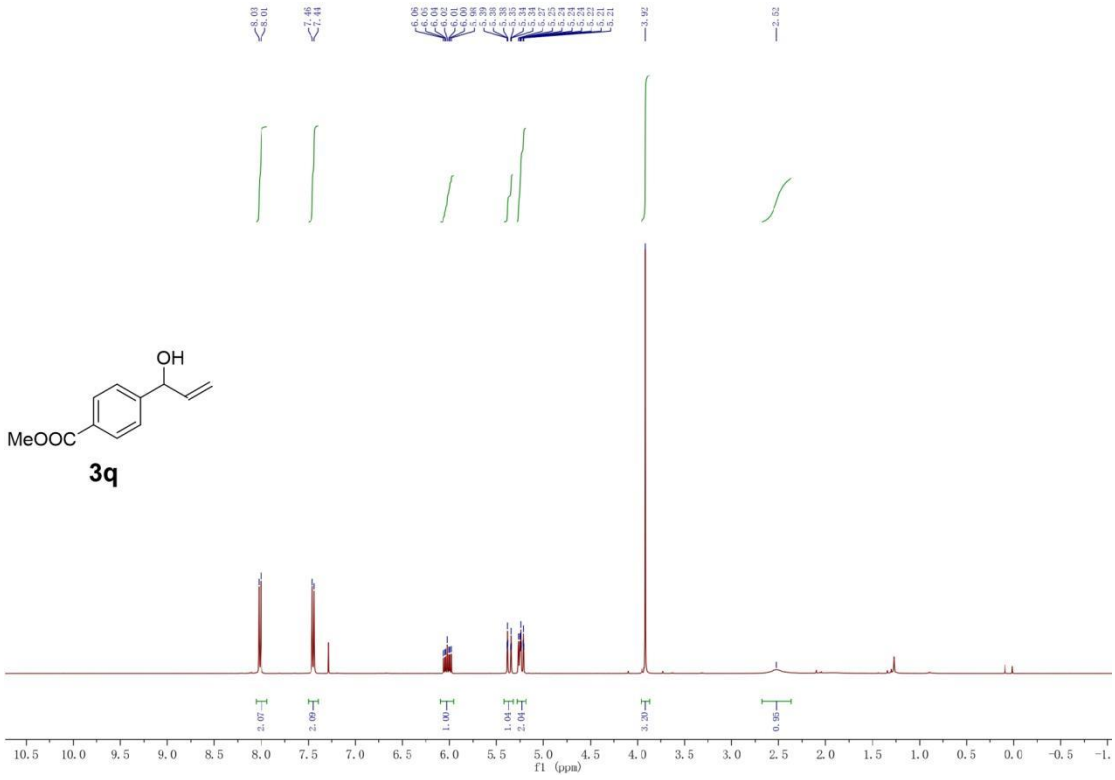

OSiEt3
2cg
f1 (ppm)

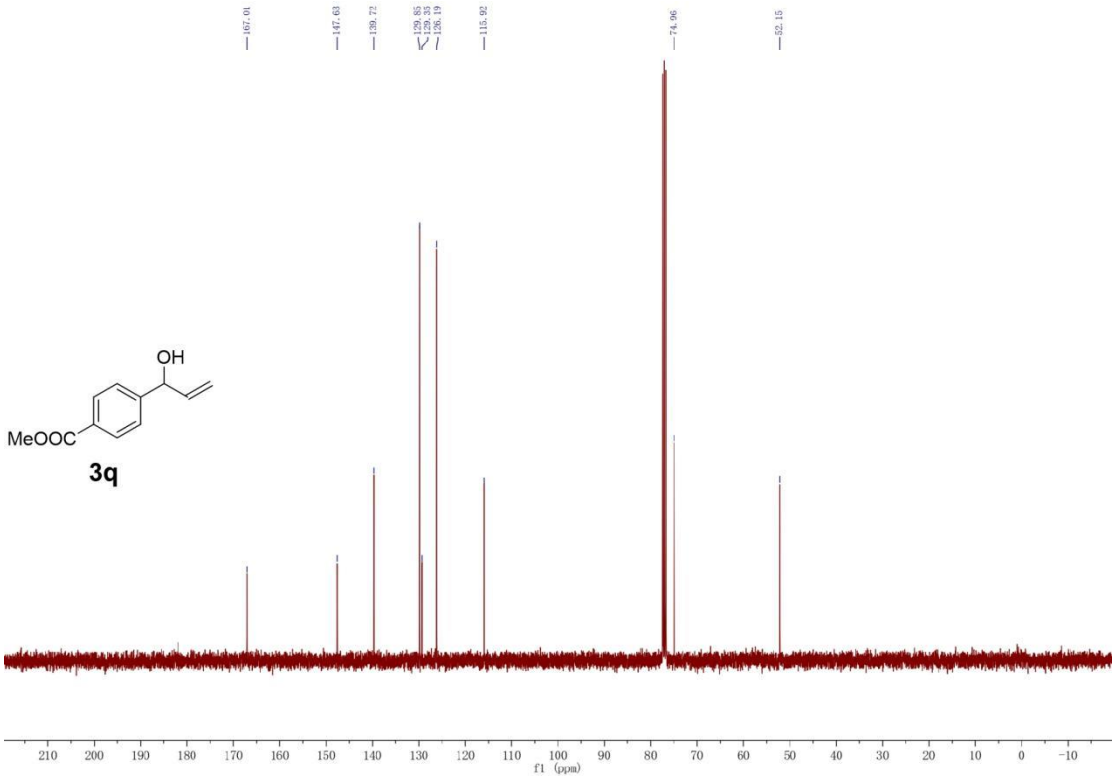

OH
OSiEt3
2ch
f1 (ppm)

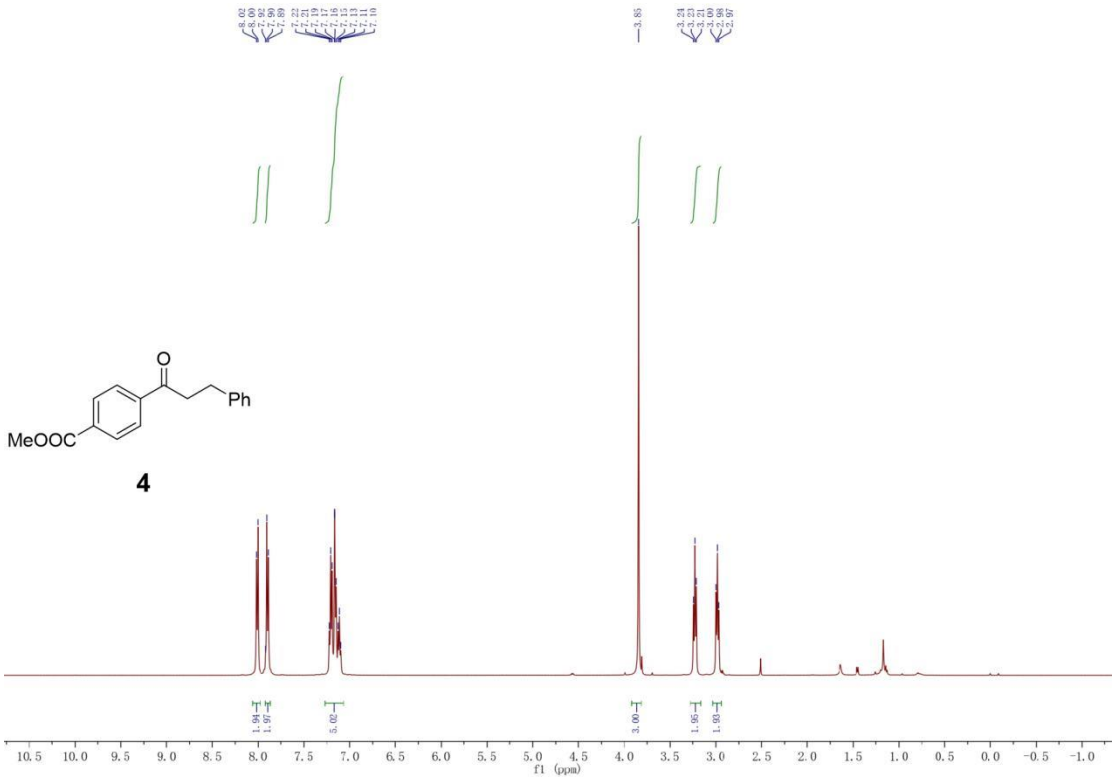

OH
OSiEt3
2ch
f1 (ppm)

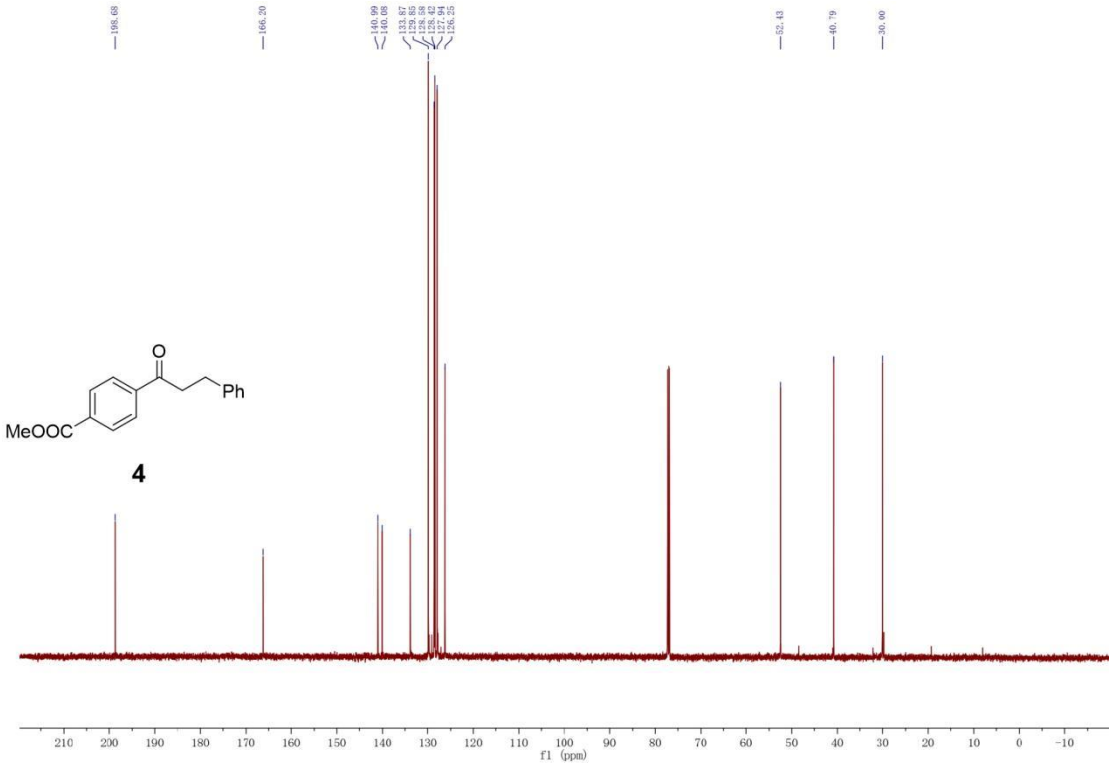

OH
iPr
3ca
f1 (ppm)

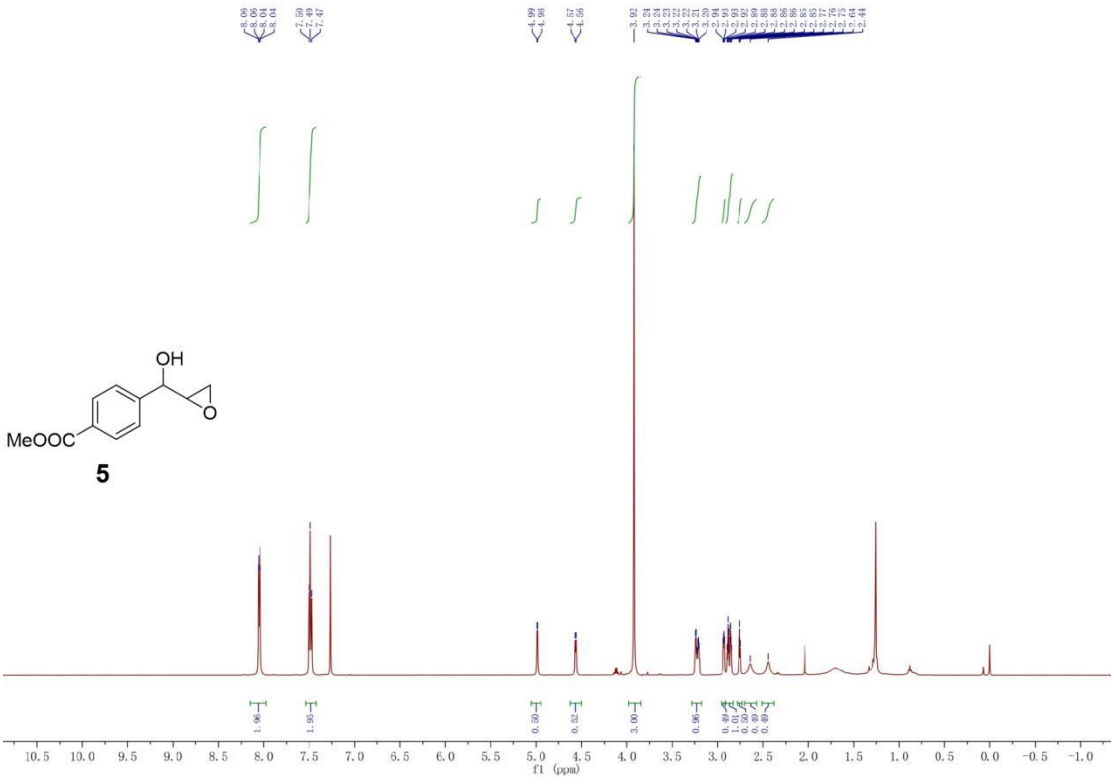

OH
iPr
3ca
f1 (ppm)

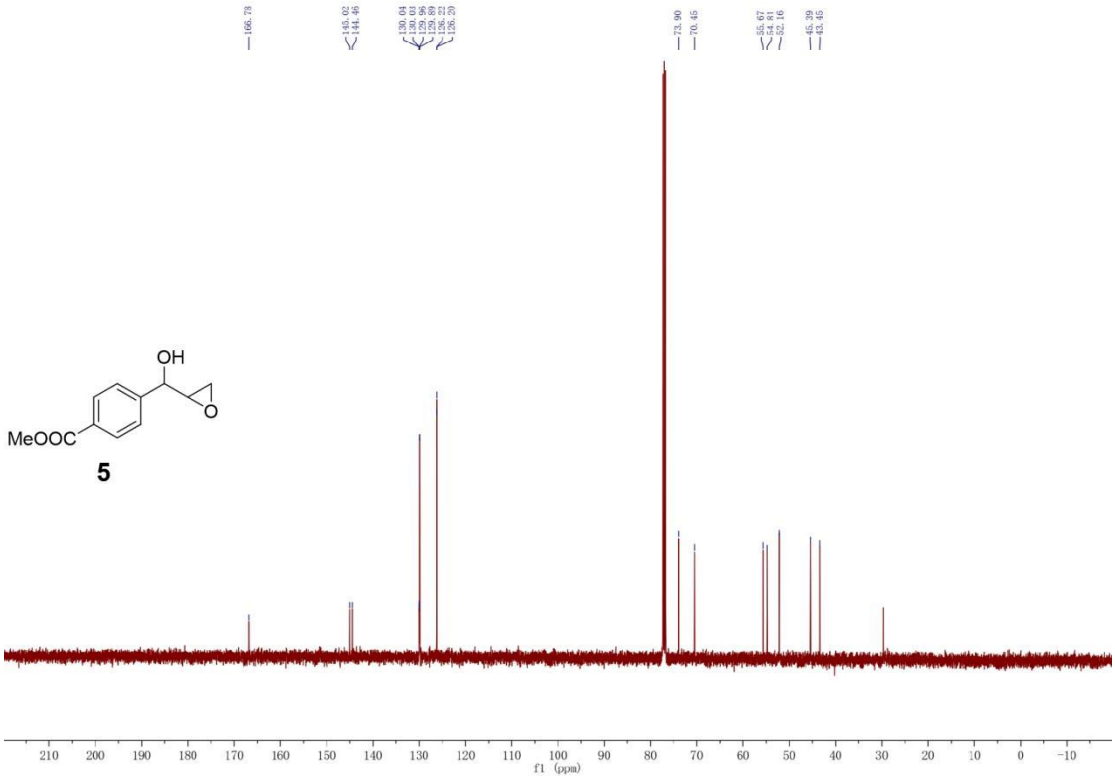

OH
MeOOC
3q
f1 (ppm)

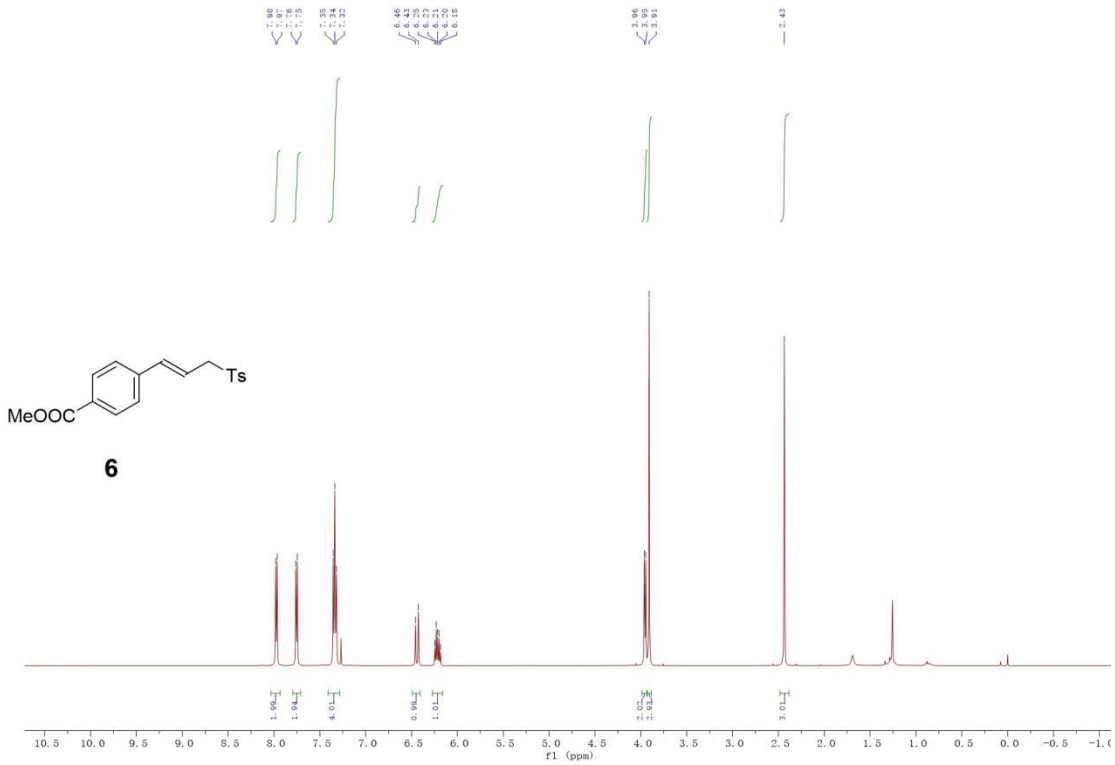

167.01
147.63
139.72
129.85
129.35
126.19
115.92
74.96
52.15
OH
MeOOC
3q
f1 (ppm)

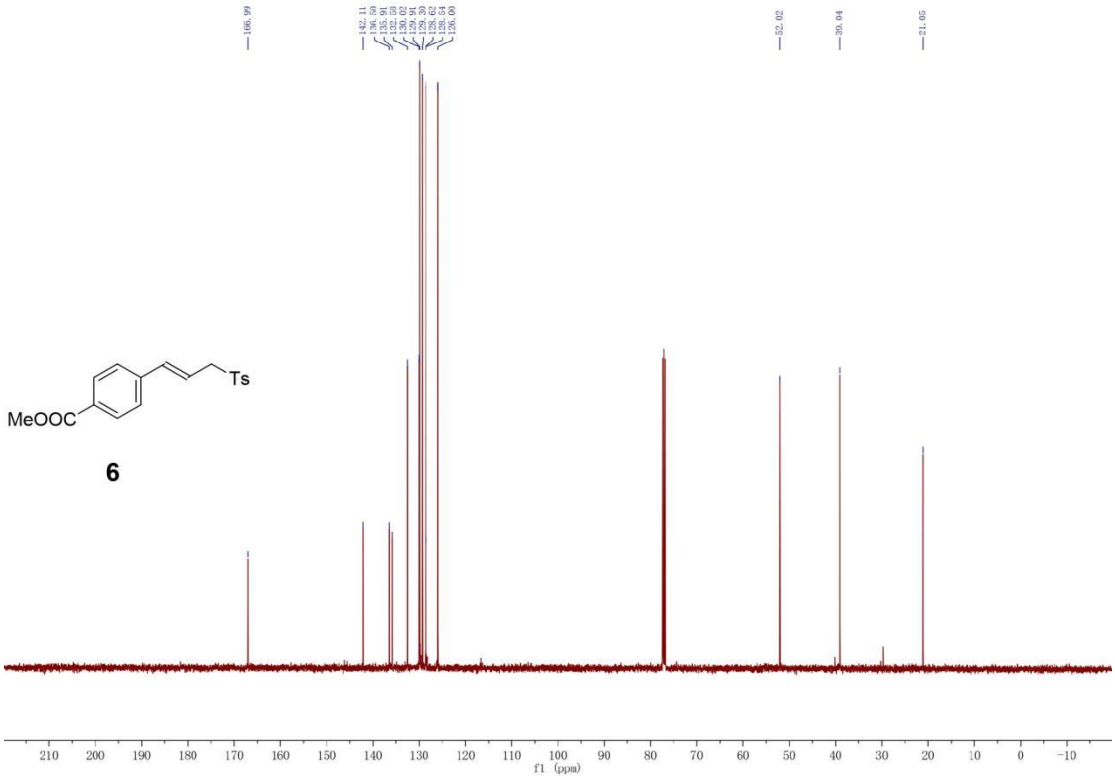

8.02
8.00
7.92
7.90
7.89
7.22
7.21
7.19
7.17
7.16
7.15
7.13
7.11
7.10
3.83
3.24
3.23
3.21
3.00
2.98
2.97
O
Ph
MeOOC
4
1.94
1.95
5.02
3.00
1.95
1.93
f1 (ppm)

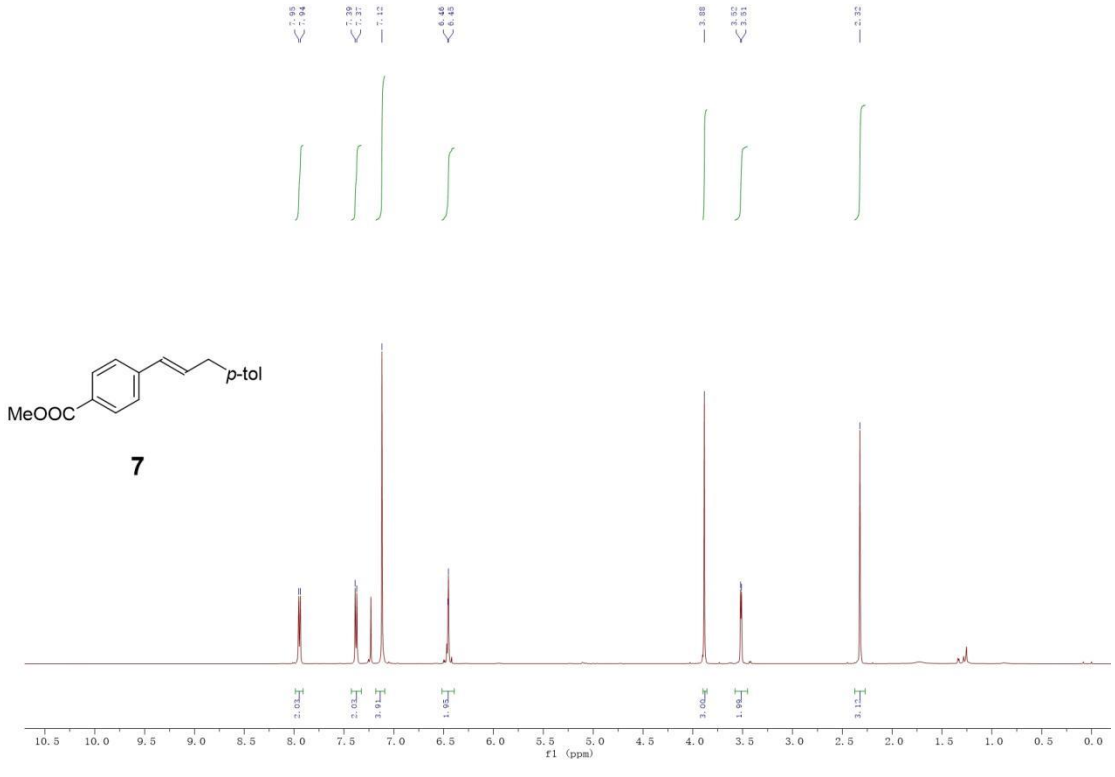

198.68
166.20
140.99
140.08
133.87
129.85
128.38
128.32
127.94
126.25
52.43
40.79
30.60
O
Ph
MeOOC
4
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

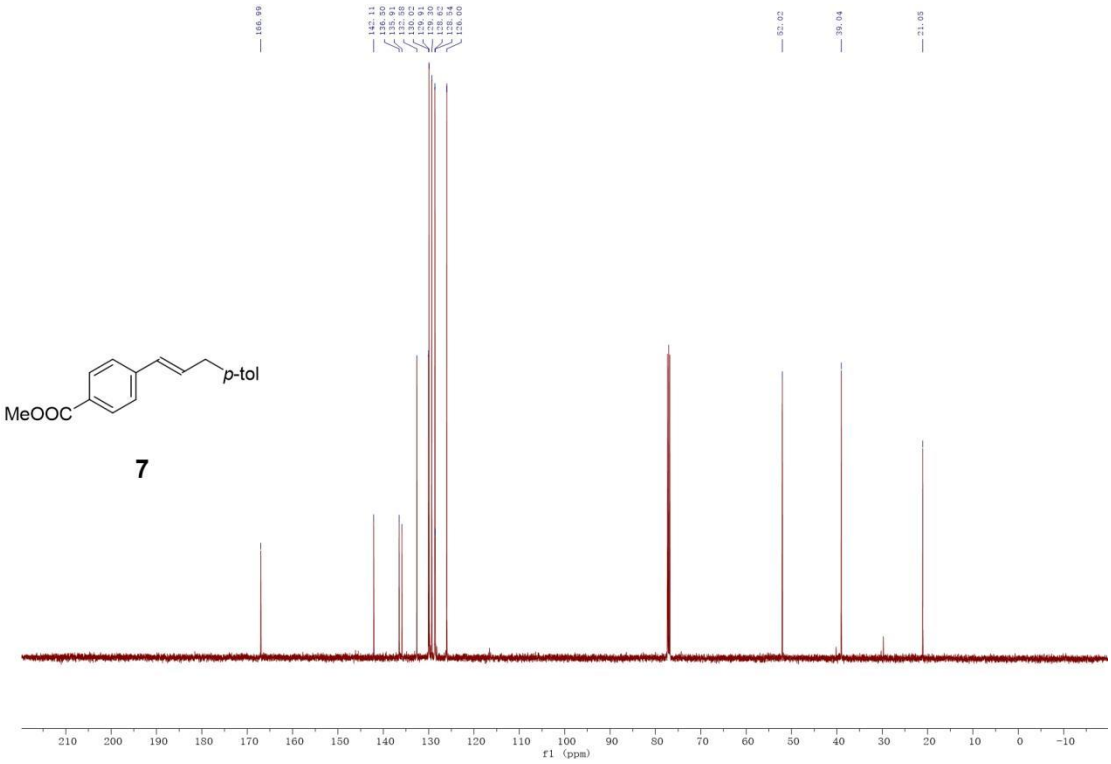

8.06
8.05
8.04
7.49
7.47
4.99
4.98
4.57
4.56
3.92
3.24
3.23
3.22
3.21
3.20
2.93
2.92
2.88
2.86
2.85
2.77
2.75
2.63
2.44
OH
O
MeOOC
5
1.96
1.95
0.50
0.82
3.00
0.96
0.49
1.01
0.50
0.49
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

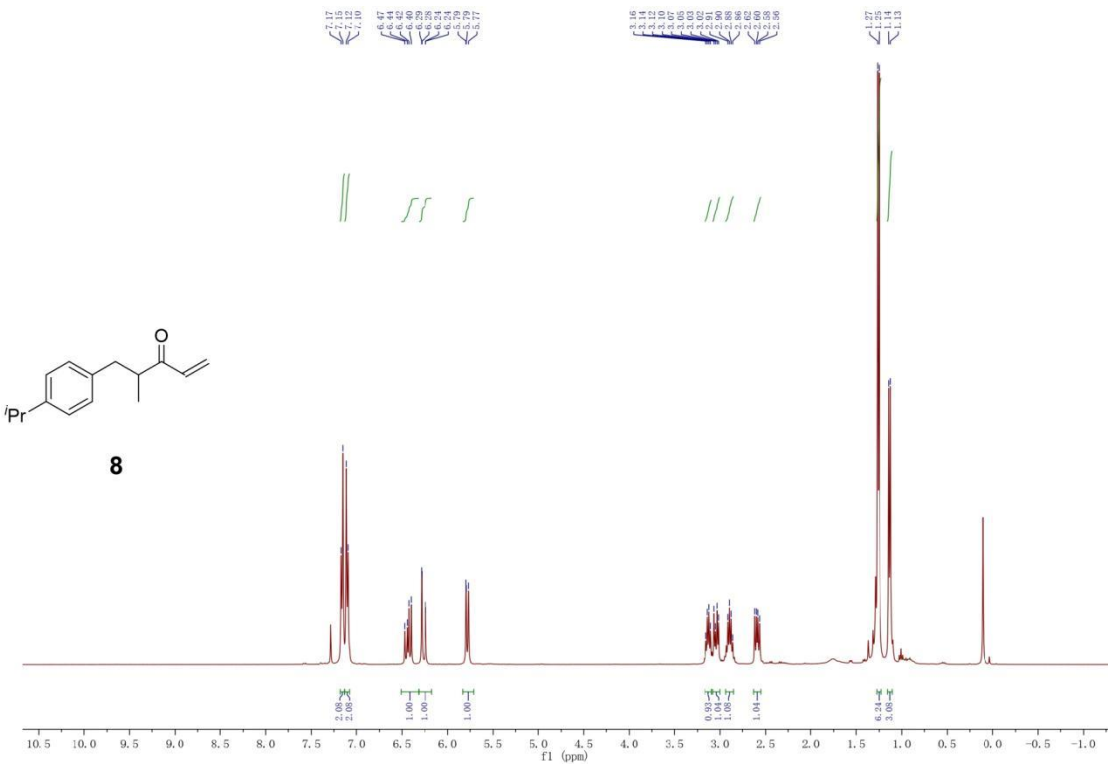

OH
O
MeOOC
5
166.78
145.02
144.46
130.04
130.03
129.89
129.89
126.22
126.20
73.90
70.45
55.67
54.81
52.16
45.39
43.46
f1 (ppm)

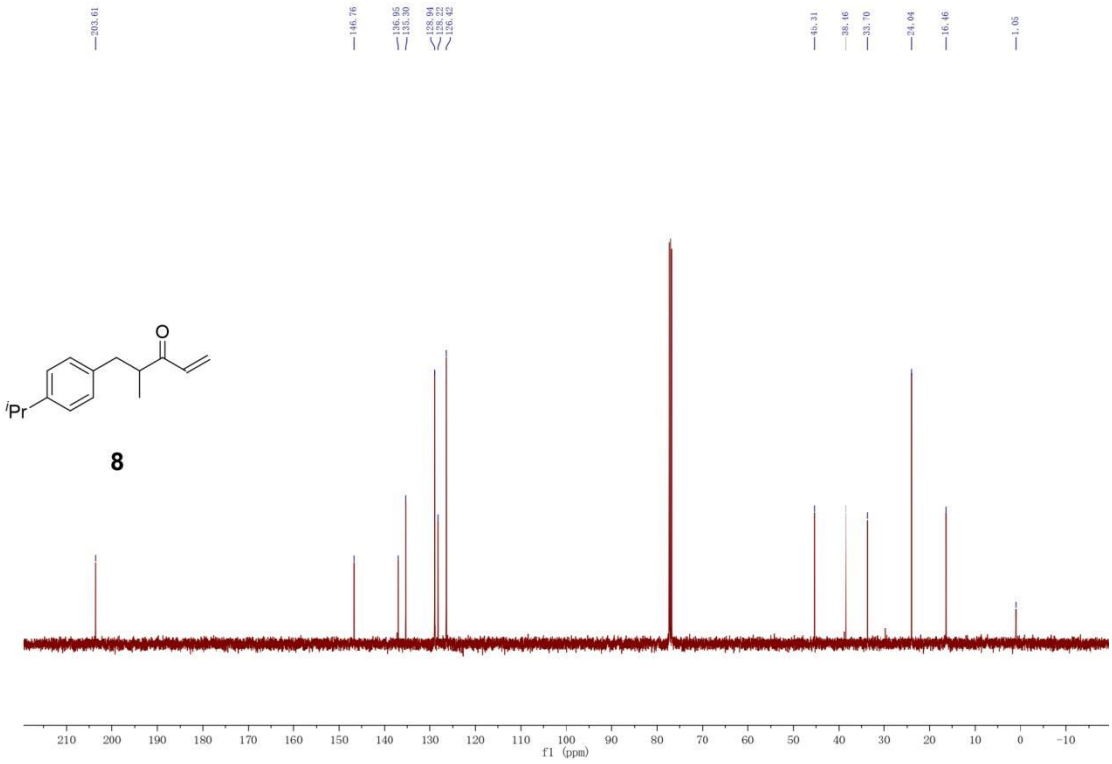

Ts
MeOOC
6
7.98
7.97
7.76
7.75
7.35
7.34
7.32
6.46
6.43
6.25
6.23
6.21
6.20
6.18
3.96
3.95
3.91
2.43
1.99
1.94
4.01
0.99
1.01
2.02
2.91
3.01
f1 (ppm)

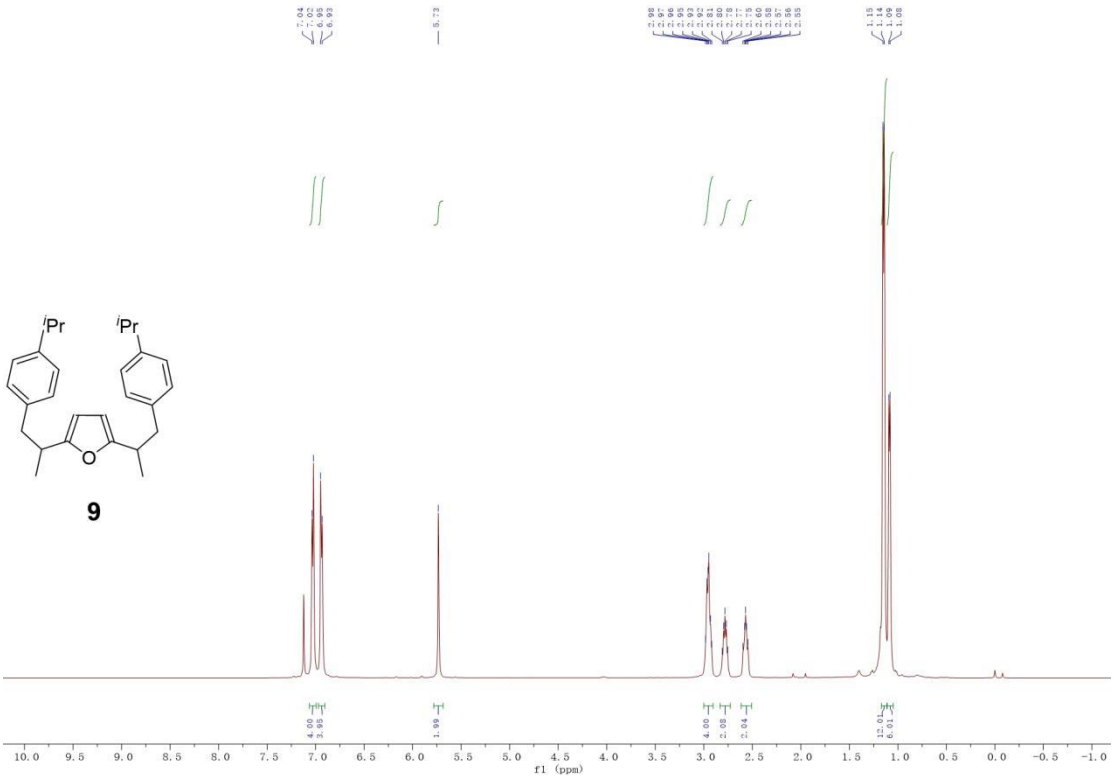

166.99
142.11
136.59
135.91
133.03
130.02
129.91
129.39
128.62
128.54
126.69
52.02
39.04
21.05
Ts
MeOOC
6
f1 (ppm)

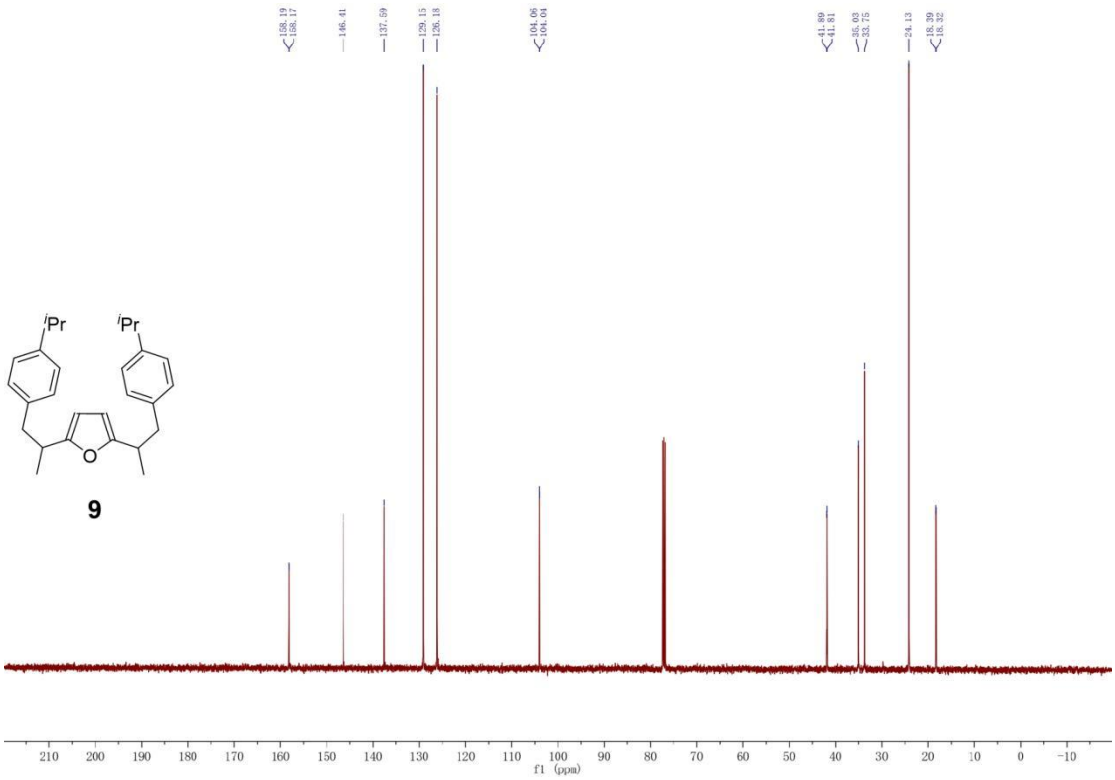

7.95
7.94
7.39
7.37
7.12
6.46
6.45
3.88
3.52
3.51
2.32
p-tol
MeOOC
7
2.01
2.01
3.01
1.95
3.00
1.99
3.11
f1 (ppm)

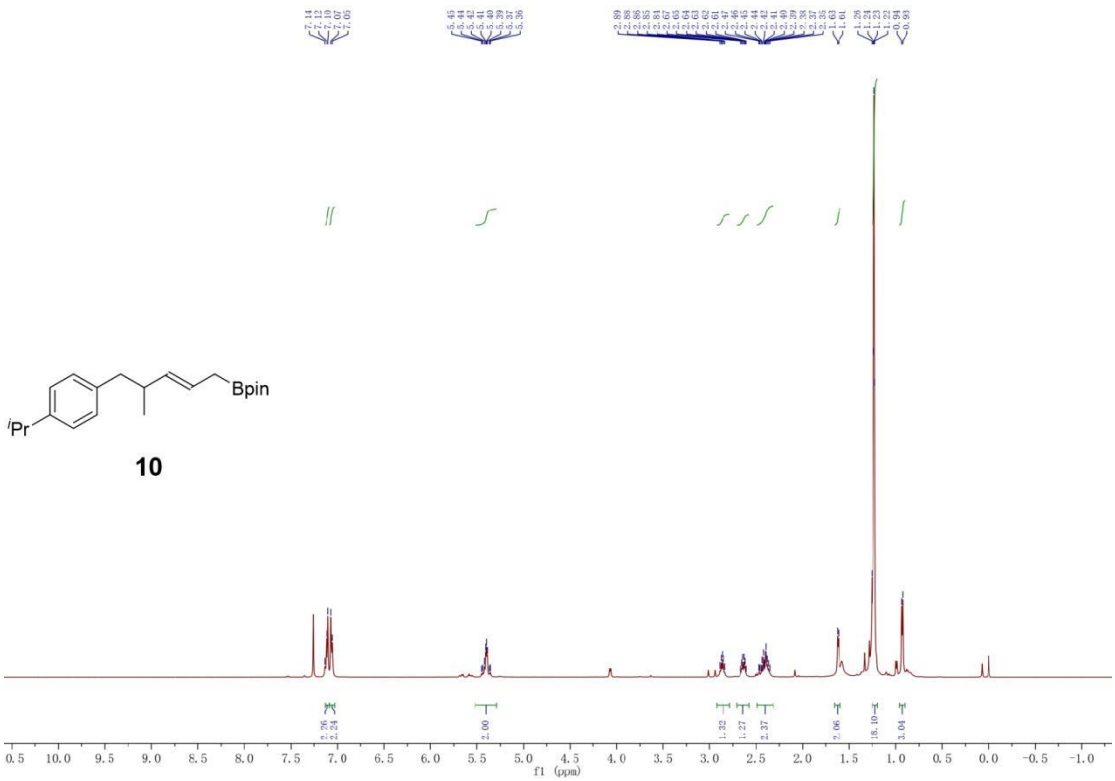

MeOOC
p-tol
7
166.99
142.11
136.90
135.91
132.38
130.02
129.91
129.30
128.62
128.04
126.00
52.02
39.04
21.05
f1 (ppm)

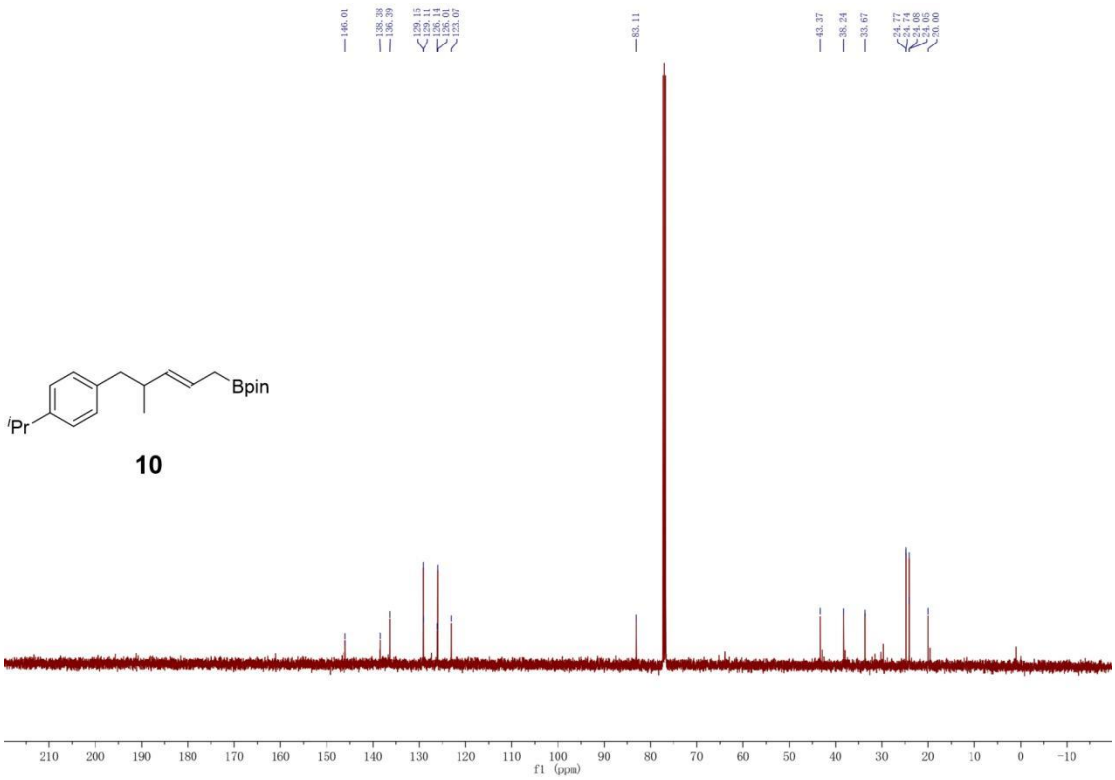

O
iPr
8
f1 (ppm)

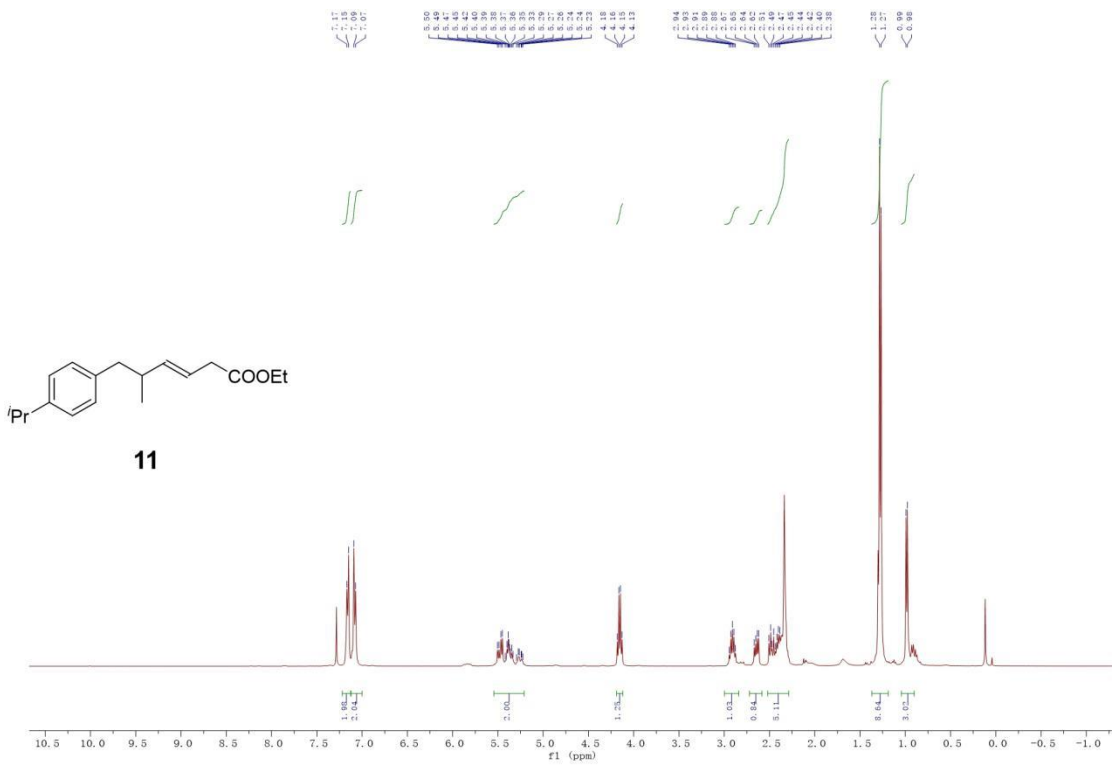

203.61
146.76
136.95
135.30
128.94
128.22
126.42
45.31
38.46
33.70
24.64
16.46
1.05
O
iPr
8
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

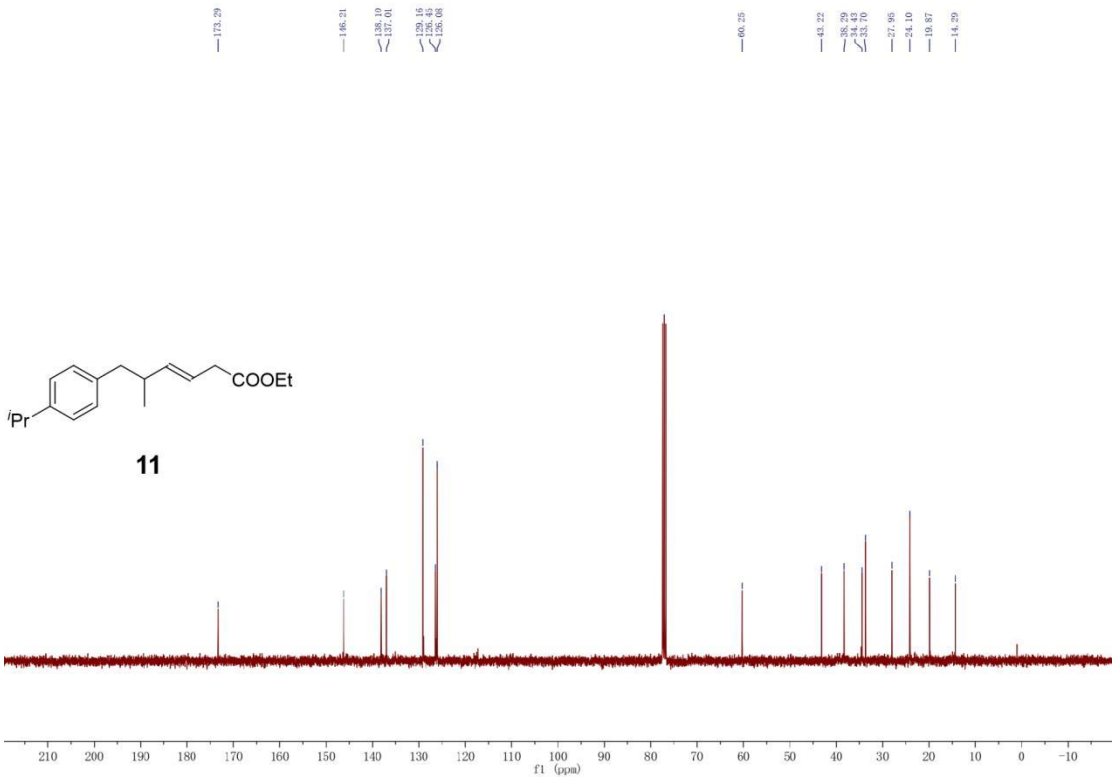

7.04
6.92
6.90
6.93
5.73
2.98
2.97
2.96
2.95
2.93
2.83
2.81
2.80
2.78
2.75
2.60
2.57
2.56
2.55
1.15
1.14
1.09
1.08
iPr
iPr
O
9
4.00
3.98
1.99
4.00
2.08
2.04
12.01
6.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)

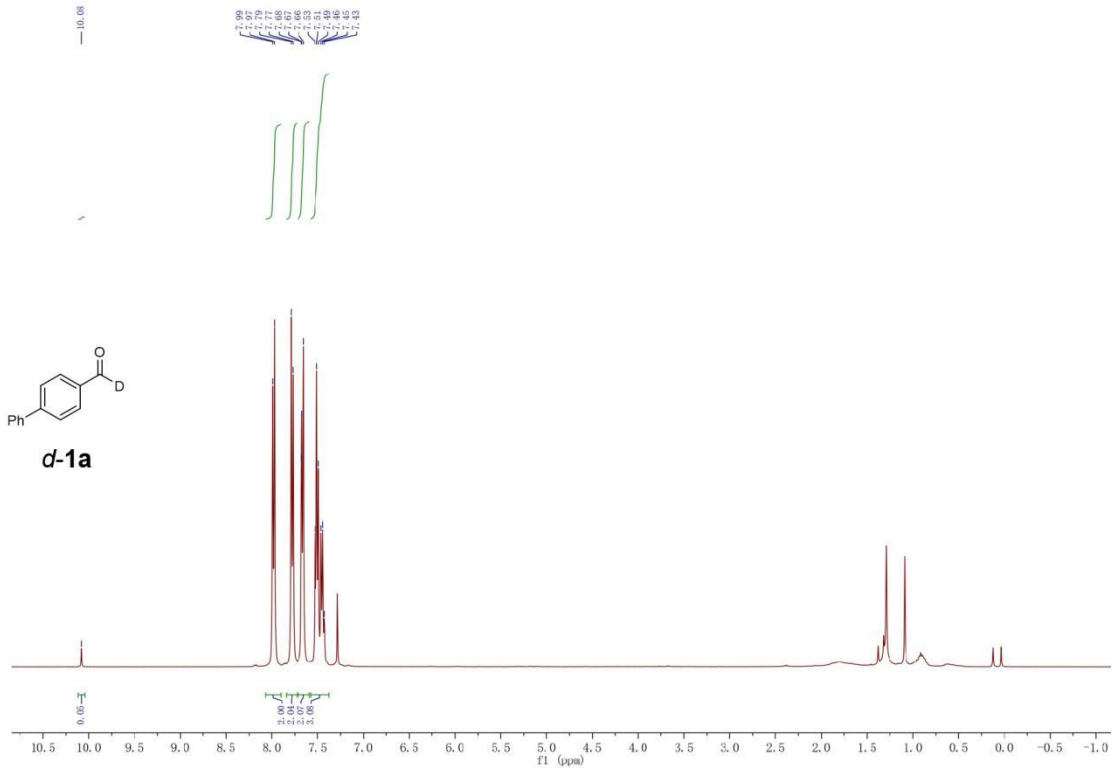

iPr
iPr
O
9
f1 (ppm)

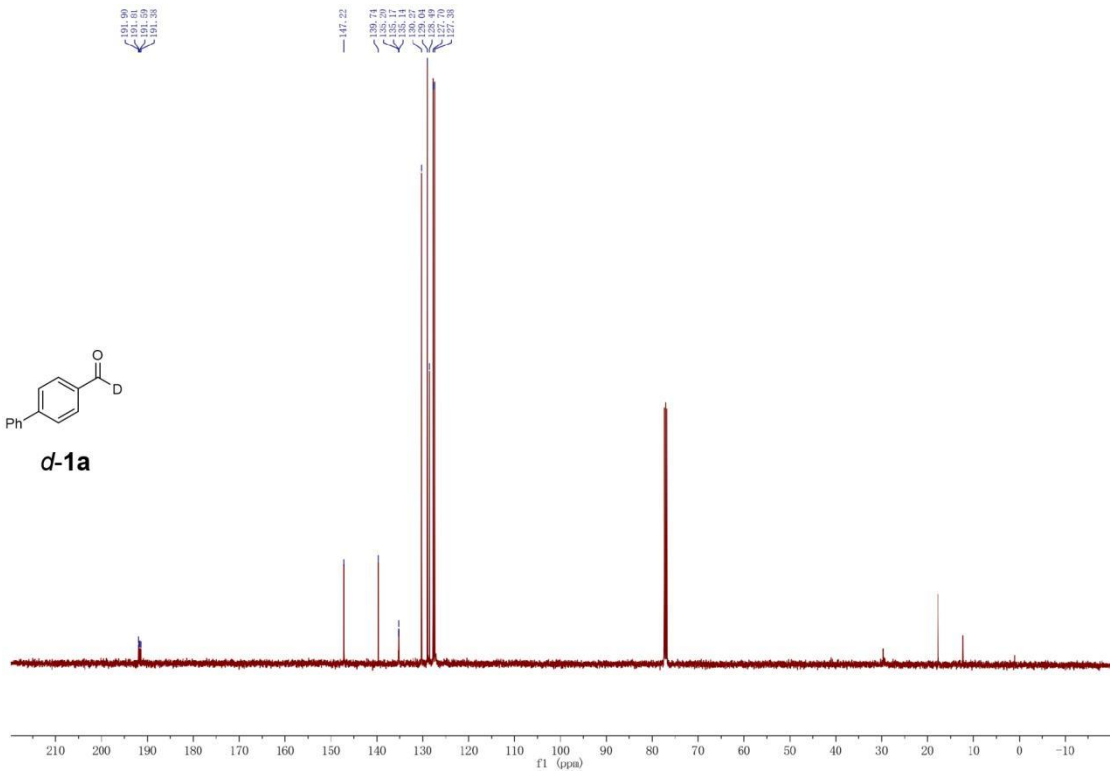

Bpin
iPr
10
f1 (ppm)


Bpin
iPr
10
f1 (ppm)

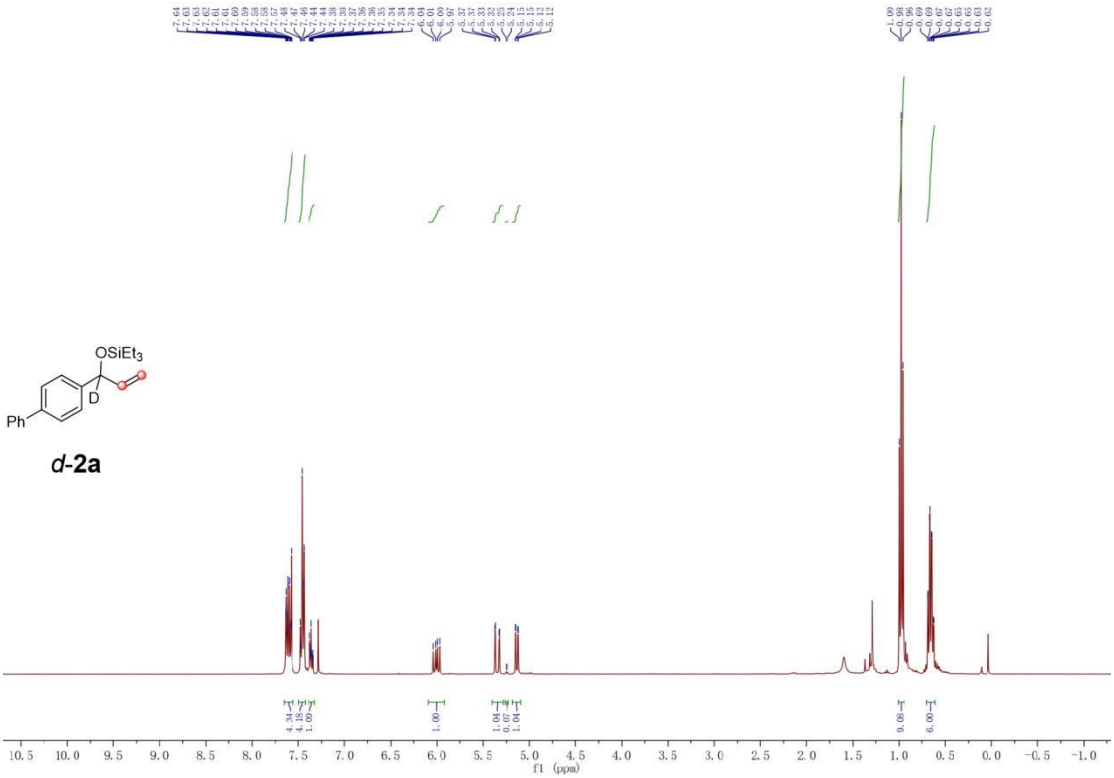

COOEt
iPr
11
f1 (ppm)


173.29
146.21
138.10
137.01
129.16
126.45
125.68
60.25
43.22
38.29
34.45
33.70
27.95
24.10
19.87
14.29
COOEt
iPr
11
f1 (ppm)

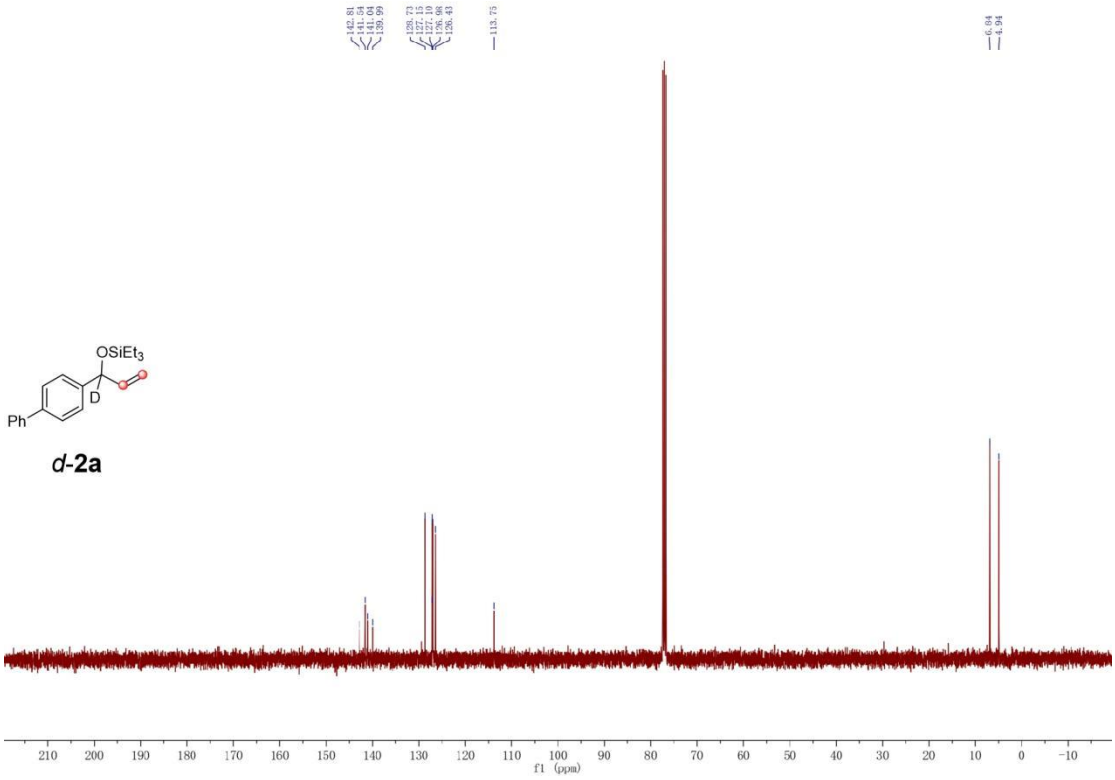

10.08
7.99
7.97
7.79
7.77
7.68
7.67
7.66
7.65
7.53
7.51
7.49
7.46
7.45
7.43
O
D
Ph
d-1a
0.05
2.00
2.04
2.07
3.08
f1 (ppm)


191.90
191.81
191.59
191.38
147.22
139.74
135.29
135.17
135.11
130.27
129.04
128.89
127.79
127.38
O
D
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

d-**1a**


7.64
7.63
7.63
7.62
7.61
7.61
7.60
7.59
7.58
7.58
7.57
7.48
7.47
7.46
7.44
7.44
7.38
7.38
7.37
7.36
7.35
7.34
7.34
6.04
6.01
6.00
5.97
5.37
5.33
5.32
5.25
5.24
5.15
5.15
5.12
1.09
0.98
0.98
0.69
0.69
0.67
0.65
0.65
0.63
0.62
OSiEt3
D
O
Ph
4.34
4.18
1.89
1.00
1.04
0.67
1.04
9.08
6.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

d-**2a**


142.81
141.54
141.04
139.99
129.73
127.15
127.10
126.98
126.43
113.75
6.84
6.94
OSiEt3
D
Ph
d-2a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

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