

Supporting Information

Electrochemically mediated synthesis of trifluoromethylallenes

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Table of Contents

1 General Methods	2
2. Chemical Experiment procedure	3
2.1 Supplementary experiments.....	3
2.2 Synthesis of Substrates	5
2.3 General procedure for the preparation of products	5
2.4 Control Experiments	7
2.5 BHT or TEMPO trapped experiment.....	8
2.6 Copies of ¹ H NMR for the Product.....	9
2.7 Cyclic Voltammetry Studies	9
3.Characterization Data and Spectrum of Compounds	11
4.Copies of the NMR spectra	21

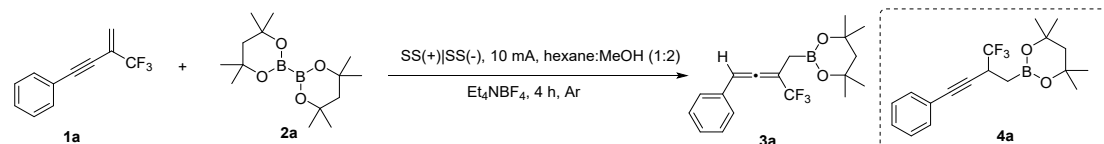
1 General Methods

Unless otherwise noted, all reagents and solvents were obtained commercially and used without further purification. Column chromatography on silica gel (300-400 mesh) was carried out using technical grade 60 - 90 °C petroleum ether and analytical grade EtOAc (without further purification). ^1H and ^{13}C and ^{19}F spectra were recorded on a 400 MHz or 500 MHz spectrometer. Chemical shifts were reported in ppm. ^1H and ^{19}F NMR spectra were referenced to CDCl_3 (7.26 ppm) or DMSO (2.5 ppm) or MeOD (4.87 ppm), and ^{13}C -NMR spectra were referenced to CDCl_3 (77.0 ppm) or DMSO (39.5 ppm) or MeOD (49.0 ppm). Peak multiplicities were designated by the following abbreviations: s, singlet; d, doublet; t, triplet; m, multiplet; brs, broad singlet and J, coupling constant in Hz. The HRMS spectrum was measured by micromass QTOF2 Quadrupole/Time of Flight Tandem mass spectrometer with electron spray ionization. Potentiostat was purchased from Shanghai Xinrui Company and the model is DJS-292B. Cyclic voltammograms were recorded on a CHI 660E potentiostat.

2. Chemical Experiment procedure

2.1 Supplementary experiments

Table S1 Screening of Reaction Conditions

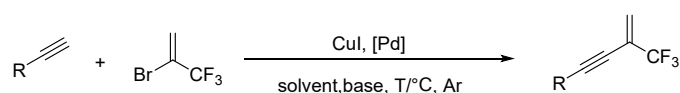


Entry	Variation from standard conditions	Yield/% ^[b]
1	none	81
2	CH ₃ CN instead of hexane:MeOH(1:2)	0
3	CH ₃ OH instead of hexane:MeOH(1:2)	66
4	THF/CH ₃ OH(1: 1) instead of hexane:MeOH(1:2)	0
5	THF instead of hexane:MeOH(1:2)	0
6	1,4-dioxane/CH ₃ OH(1:2) instead of hexane:MeOH(1:2)	50
7	CH ₃ CN/CH ₃ OH (9:1) instead of hexane:MeOH(1:2)	30
8	THF/CH ₃ OH(1:5) instead of hexane:MeOH(1:2)	47
9	HFIP instead of hexane:MeOH(1:2)	20
10	C (+) SS (-) instead of SS (+) SS (-)	0
11	SS (+) Pt (-) instead of SS (+) SS (-)	40
12	SS (+) C (-) instead of SS (+) SS (-)	0
13	Fe (+) SS (-) instead of SS (+) SS (-)	0
14	2.5 eq B ₂ oct ₂	79
15	1.5 eq B ₂ oct ₂	77
16	1 eq K ₂ CO ₃ as the base	54
17	1 eq CsF as the base	0
18	1 eq CH ₃ ONa as the base	20
19	1 eq ^t BuOK as the base	5
20	LiClO ₄ instead of Et ₄ NBF ₄	0
21	ⁿ Bu ₄ NBr instead of Et ₄ NBF ₄	20
22	ⁿ Bu ₄ NCl instead of Et ₄ NBF ₄	20
23	ⁿ Bu ₄ NI instead of Et ₄ NBF ₄	30
24	ⁿ Bu ₄ NPF ₆ instead of Et ₄ NBF ₄	54
25	5 mA instead of 10 mA	50
26	15 mA instead of 10 mA	56
27	6 h instead of 4 h	60
28	8 h instead of 4 h	50
29	no current	0

Standard conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), Et₄NBF₄ [0.05 M] in hexane:CH₃OH (1:2, 6 mL), rt, stainless steel electrodes (cathode and anode), 10 mA, 3 F·mol⁻¹, undivided cell, under Ar.
1a (0.1 mmol), **2a** (0.15 mmol), Et₄NBF₄ [0.05 M] in hexane:CH₃OH (1:2, 6 mL), rt, stainless steel

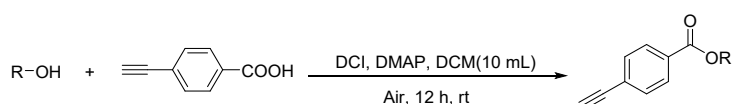
electrodes (cathode and anode), 10 mA, 3 F·mol⁻¹, undivided cell, under Ar. ^[a] Yield of isolated product .

2.2 Synthesis of Substrates



Method A: ¹Under argon atmosphere, PdCl₂(PPh₃)₂ (0.14 g, 2 mol %) and CuI (0.10 g, 5 mol %) were added into an oven dried Schlenk flask, followed by dry degassed triethylamine (30 mL) and 2-bromo-3,3,3-trifluoroprop-1-ene (1.75 g, 10 mmol, 1.0 equiv.). The corresponding 1-alkyne (11 mmol, 1.1 equiv.) was added dropwise via a syringe. The slurry was stirred at 50 °C (oil bath) overnight, and quenched with saturated NH₄Cl solution, extracted with hexane, dried over anhydrous Na₂SO₄. After concentration under vacuum, the residue was purified by distillation or column chromatography over silica gel (200-300 mesh) using ethyl acetate/petroleum ether as eluent.

Method B: ²Under argon atmosphere, Pd(PPh₃)₄ (0.0520 g, 0.3 mol %) and CuI (0.0571 g, 2 mol %) were added into an oven dried round bottom flask, followed by dry degassed diethylamine (7.5 mL) and THF (20 mL), sequentially. The corresponding 1-alkyne (15 mmol, 1.0 equiv.) and 2-bromo-3,3,3-trifluoroprop-1-ene (3.499 g, 1.3 equiv.) was added dropwise. The slurry was stirred at room temperature overnight, and quenched with saturated NH₄Cl solution at 0 °C, extracted with hexane/Et₂O (1:1), washed by 1 mol/L HCl, dried over anhydrous Na₂SO₄. After concentration under vacuum, the residue was purified by distillation.



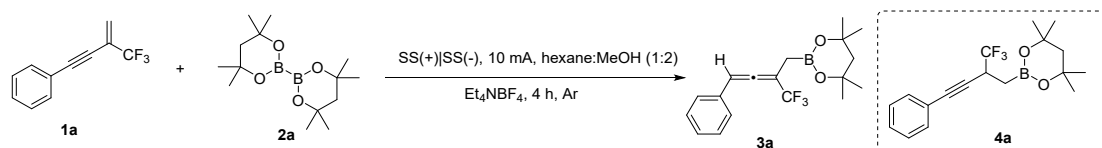
To a mixture of the 4-vinylbenzoic acid (1.0 equiv.), the corresponding alcohol or amine (1.5equiv.) and DMAP (0.2 equiv.) in CH₂Cl₂ (0.2 M) was added N, N'- Diisopropylcarbodiimide (1.2 equiv.) dropwise. The reaction was stirred at room temperature until its completion, which was monitored by TLC. The reaction was quenched with H₂O and extracted with CH₂Cl₂. The organic layers were washed with brine, dried over anhydrous Na₂SO₄, filtered, concentrated in vacuo. Flash chromatography (EtOAc/Hexane 6 = 1:5) afforded the product. The analytical data of the alkenes are in accordance with the reported data².

References:

1. C. Yang, Z.-L. Liu, D.-T. Dai, Q. Li, W.-W. Ma, M. Zhao and Y.-H. Xu, Catalytic Asymmetric Conjugate Protosilylation and Protoborylation of 2-Trifluoromethyl Enynes for Synthesis of Functionalized Allenes, *Org. Lett.*, 2020, **22**, 1360–1367;
2. S. Chen, J. Wang, L.-G. Xie, Transition Metal-Free Formal Hydro/Deuteromethylthiolation of Unactivated Alkenes, *Org. Biomol. Chem.* 2021, **19**, 4037–4042.

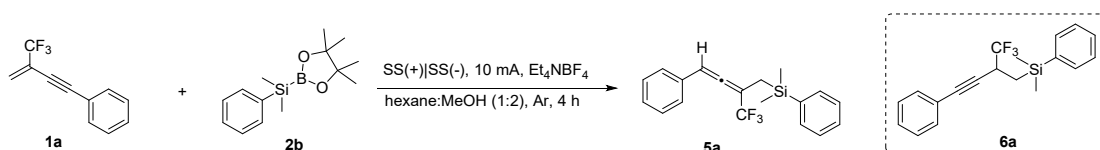
2.3 General procedure for the preparation of products

General procedure for synthesis of 4,4,6,6-tetramethyl-2-(4-phenyl-2-(trifluoromethyl)buta-2,3-dien-1-yl)-1,3,2-dioxaborinane

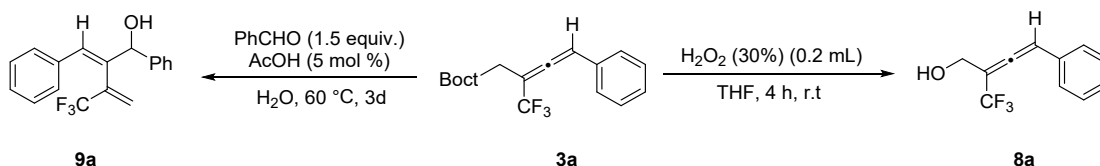


B₂pin₂ (112.7 mg, 0.2 mmol, 2 equiv) and Et₄NBF₄ (65.1 mg, 0.3 mmol, 0.05 M) were weighed under Argon in a glovebox and added in a 10 mL IKA[®] vial equipped with a magnetic stirrer. The 2-trifluoromethyl-1,3-enyne (0.2 mmol, 1 equiv) was added and all compounds were solubilized in 2 mL of hexane and 4 mL of methanol. Two stainless steel electrodes (SST) were immersed in the solution and the vial was set on the IKA ElectraSyn[®] device at 10 mA. The reaction is monitored by TLC and stopped when total conversion of the alkene substrate is observed. The crude mixture is filtered through a pad of silica and concentrated under reduced pressure. The residue was purified by flash chromatography, preparative thin layer chromatography (PTLC) or reversed-phase chromatography.

General procedure for synthesis of dimethyl(phenyl)(4-phenyl-2-(trifluoromethyl)buta-2,3-dien-1-yl)silane



PhMe₂Si-Bpin (39.4 mg, 0.15 mmol, 1.5 equiv) and Et₄NBF₄ (65.1 mg, 0.3 mmol, 0.05 M) were weighed under Argon in a glovebox and added in a 10 mL IKA[®] vial equipped with a magnetic stirrer. The 2-trifluoromethyl-1,3-enyne (0.1 mmol, 1 equiv) was added and all compounds were solubilized in 2 mL of hexane and 4 mL of methanol. Two stainless steel electrodes (SST) were immersed in the solution and the vial was set on the IKA ElectraSyn[®] device at 10 mA. The reaction is monitored by TLC and stopped when total conversion of the alkene substrate is observed. The crude mixture is filtered through a pad of silica and concentrated under reduced pressure. The residue was purified by flash chromatography, preparative thin layer chromatography (PTLC) or reversed-phase chromatography.



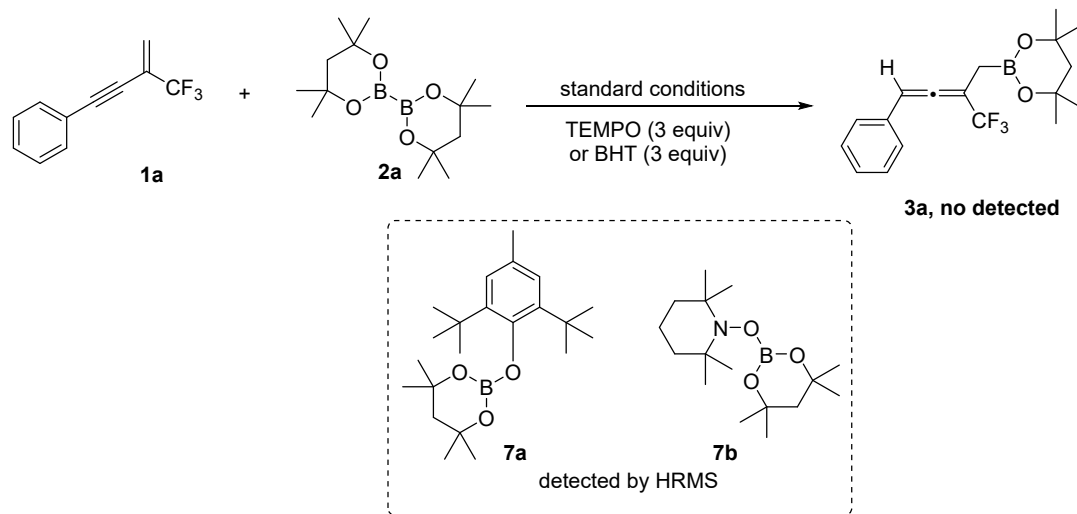
Under argon atmosphere, the starting material **3** (0.0338 g, 0.1 mmol), benzaldehyde (0.15 mmol, 1.5 equiv.) and H₂O (0.9 mL) were added sequentially into a Schlenk tube. Then AcOH (0.0003 g, 5 mol %) in H₂O (0.1 mL) was added into the mixture and stirred at 60 °C (oil bath) for 3 d. The mixture was cooled to room temperature, extracted with DCM (15 mL × 3). The combined organic layers dried over anhydrous Na₂SO₄. After concentration under vacuum, the residue was purified by column chromatography over silica gel (200-300 mesh) using ethyl acetate/petroleum ether (1:5) as eluent to afford the product **8a** as pale yellow oil (0.0234 g, 77% yield).

The starting material **3** (0.0338 g, 0.1 mmol) was dissolved in THF (2 mL) and cooled to 0 °C. Then, 30% H₂O₂ (0.2 mL) was added dropwise. The mixture was warmed to room temperature, diluted with 1 mL THF and stirred for 4 h in the air. When the reaction was over, the resulting solution was cooled to 0 °C again, saturated aqueous Na₂S₂O₃ (0.5 mL) was added slowly, stirred for a further 5 min. Warmed to room temperature and extracted with ethyl acetate (15 mL × 3), combined the organic layers and dried over anhydrous Na₂SO₄. After concentration under vacuum, the residue was purified by column chromatography over silica gel (200-300 mesh) using ethyl

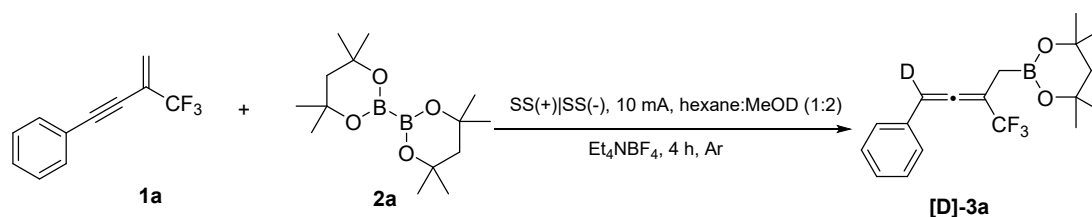
acetate/petroleum ether (1: 5) as eluent to afford the product as pale yellow oil (0.0214 g, 88% yield)

2.4 Control Experiments

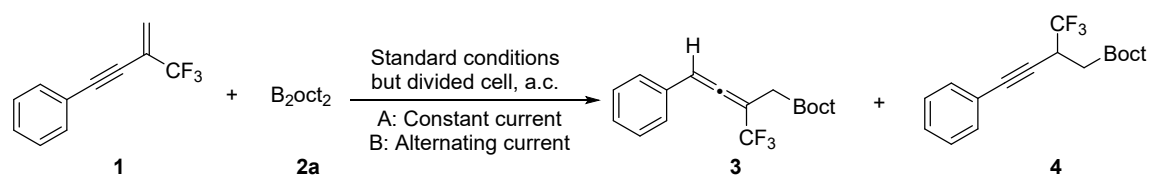
a) Radical inhibition experiments



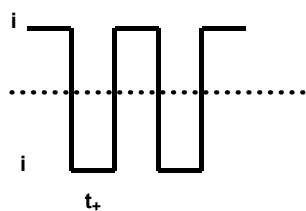
b) Labeling experiment



c) Divided electrolysis experiments



AC: $i = \pm 10 \text{ mA}$, $t_{\pm} = 1 \text{ s}$



Conditions	Yield of (3/4 ratio)
Method A	N.R
Method B	13%

Procedures for reaction b: A reaction was set-up according to the general procedure using a mixture of CH_3OD : hexane (2:1) as the solvent. The ratio of H- and D- products were determined by ^1H NMR.

2.5 BHT or TEMPO trapped experiment

Sample Name	BHT	Position	P1-A1	Instrument Name	Instrument 1
User Name		Inj Vol	0.2	InjPosition	
Sample Type	Sample	IRM Calibration Status	Success	Data Filename	BHT.d
ACQ Method	Yijing-8.m	Comment		Acquired Time	1/17/2024 9:35:08 PM

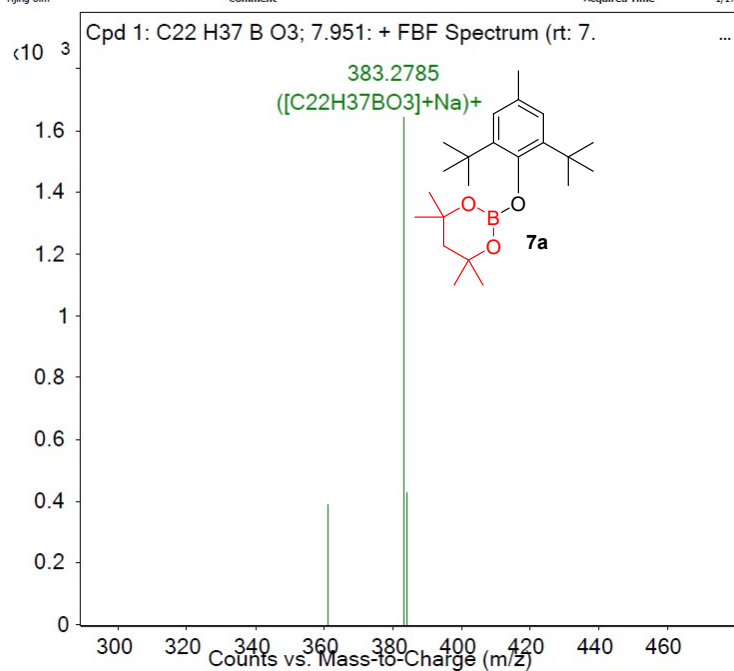


Figure S2 HRMS analysis compound **7a**. HRMS (m/z) [ESI]: calculated for C₂₂H₃₇BO₃ [M+Na]⁺: 383.2728, found 383.2785.

Sample Name	Unavailable	Position	Unavailable	Instrument Name	Unavailable
User Name	Unavailable	Inj Vol	Unavailable	InjPosition	Unavailable
Sample Type	Unavailable	IRM Calibration Status	Success	Data Filename	TEMPO.d
ACQ Method		Comment	Sample information is unavailable	Acquired Time	Unavailable

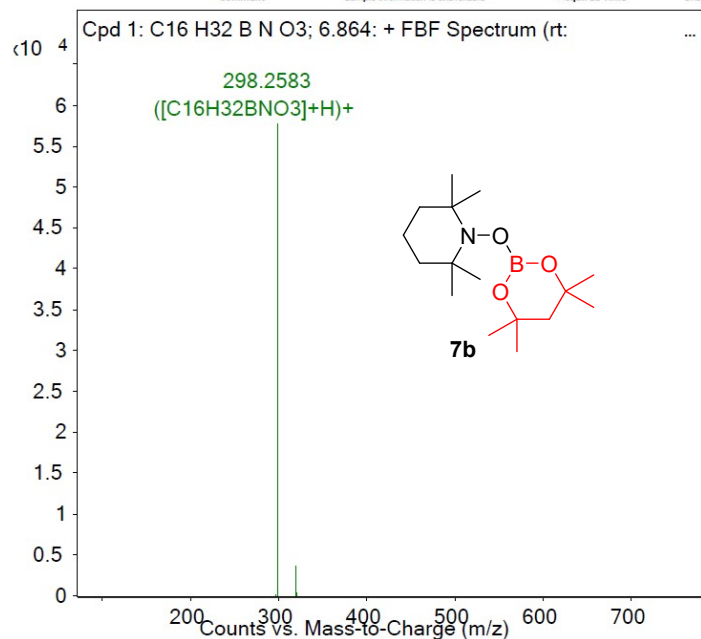
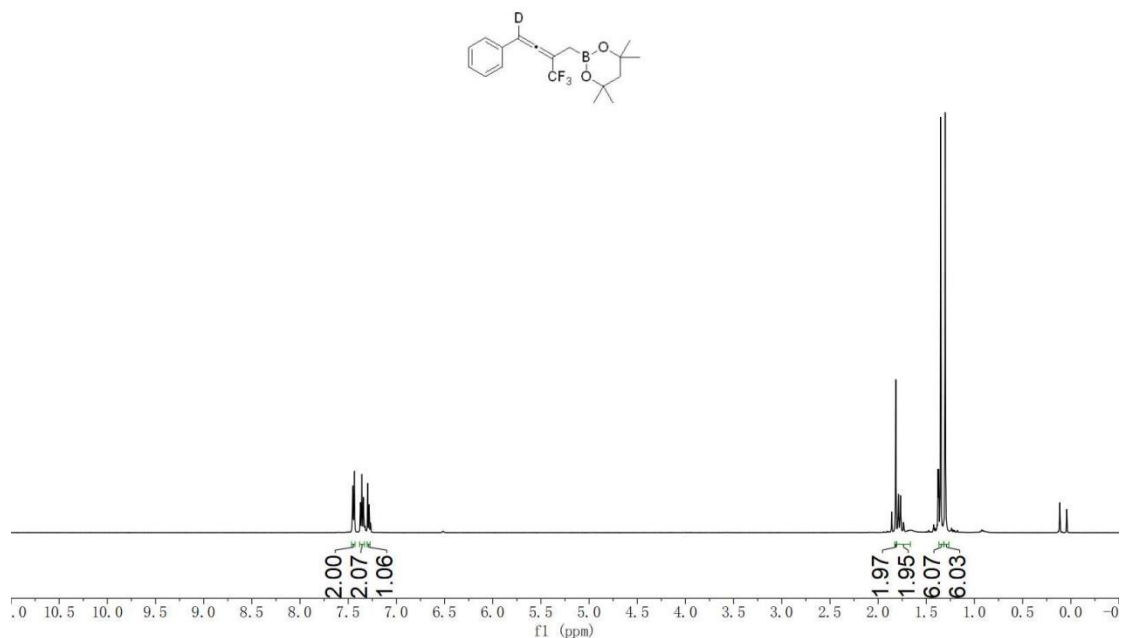


Figure S3 HRMS analysis compound **7b**. HRMS (m/z) [ESI]: calculated for C₁₆H₃₂BNO₃ [M+Na]⁺: 298.2548, found 298.2583.

2.6 Copies of ^1H NMR for the Product



2.7 Cyclic Voltammetry Studies

The cyclic voltammograms were recorded in an electrolyte solution of Et_4NBF_4 (0.05 M) in various solvents using a glassy carbon disk working electrode (diameter, 3 mm), a Pt wire auxiliary electrode and a Ag/AgCl reference electrode. The scan rate was 100 mV/s.

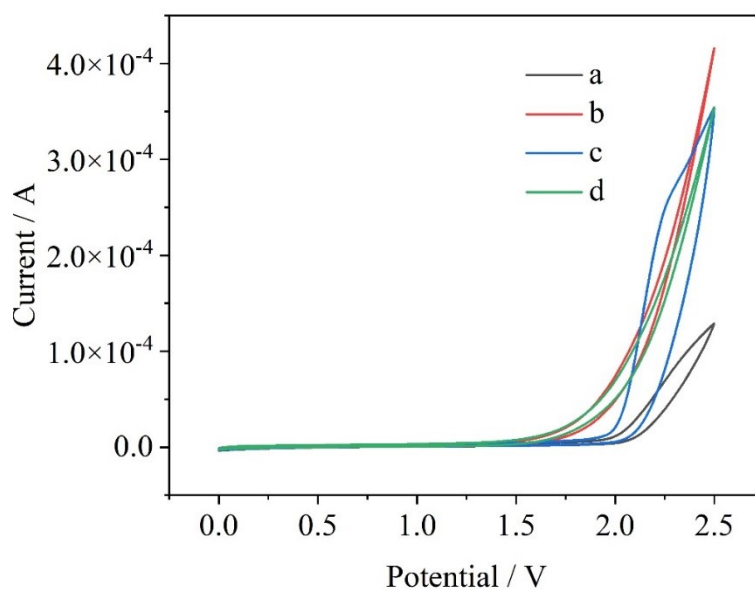


Figure S4-1 Cyclic voltammograms in 0.3 mmol B_2oct_2 + 0.05 M Et_4NBF_4 . a) CH_3CN , 6 mL. b) CH_3OH , 6 mL. c) CH_3ONa + CH_3CN , 6 mL. d) CH_3OH +Hexane, 6 mL.

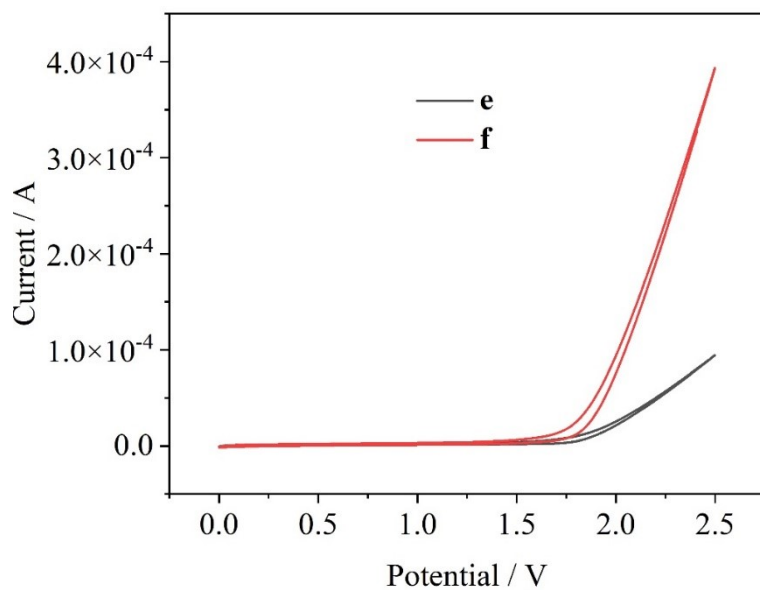


Figure S4-2 Cyclic voltammograms in 0.3 mmol enyne + 0.05 M Et_4NBF_4 + CH_3OH /hexane (1:2, 6 mL). e) no B_2oct_2 . f) with B_2oct_2

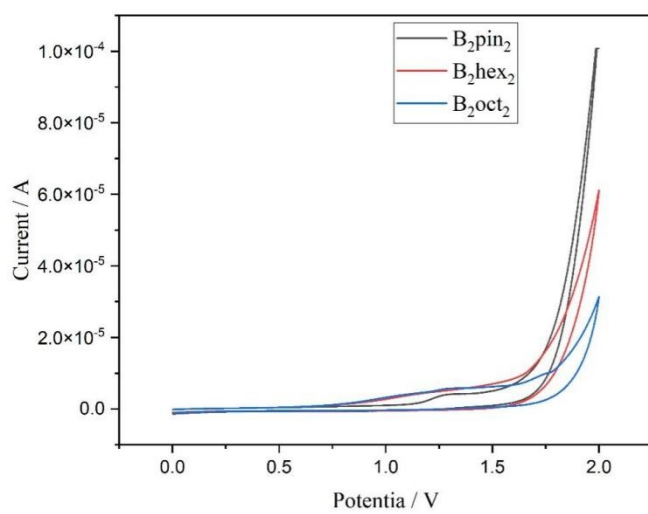
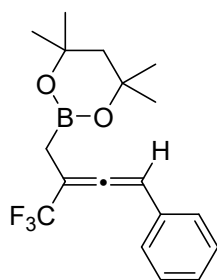


Figure S4-3 Cyclic voltammograms in 0.3 mmol Borane + 0.05 M Et_4NBF_4 + CH_3OH /hexane (1:2, 6 mL).

3.Characterization Data and Spectrum of Compounds

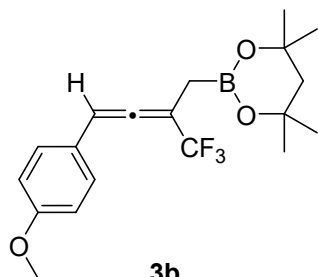


3a

4,4,6,6-tetramethyl-2-(4-phenyl-2-(trifluoromethyl)buta-2,3-dien-1-yl)-1,3,2-dioxaborinane(**3a**):

Pale yellow oil, yield = 81%, 0.0548 g

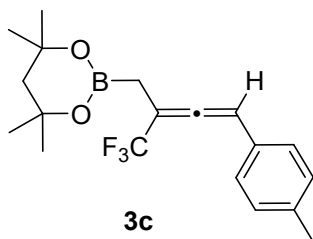
¹H NMR (400 MHz, Chloroform-d) δ 7.33-7.31 (m, 2H), 7.21-7.25 (m, 2H), 7.17-7.14 (m, 1H), 6.40-6.38(m, 1H), 1.69 (s, 2H), 1.67-1.59 (m, 2H), 1.23 (s, 6H), 1.18 (s, 6H); ¹³C NMR (101 MHz, Chloroform-d) δ 204.4 (q, $J = 4.1$ Hz), 132.8, 131.7, 128.6, 127.6, 123.9 (q, $J = 274.7$ Hz) , 100.2(q, $J = 30.3$ Hz), 99.8, 71.1, 48.7, 31.6, 31.5; ¹⁹F NMR (376 MHz, Chloroform-d) δ -64.85, -72.60; ¹¹B NMR (128 MHz, Chloroform-d) δ 28.66; HRMS (ESI) m/z calcd for C₁₈H₂₂BF₃O₂Na [M+Na]⁺ : 361.1557, found: 361.1563



3b

2-(4-(4-methoxyphenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)-4,4,6,6-tetramethyl-1,3,2-dioxaborinane(**3b**): Pale yellow oil, yield = 75%, 0.0552 g

¹H NMR (400 MHz, Chloroform-d) δ 7.35-7.30 (m, 2H), 6.88-6.84 (m, 2H), 6.45-6.43 (m, 1H), 3.81 (s, 3H), 1.78 (s, 2H), 1.74-1.68 (m, 2H), 1.31 (s, 7H), 1.27 (s, 8H); ¹³C NMR (126 MHz, Chloroform-d) δ 203.9 (q, $J = 3.8$ Hz), 159.4, 133.2, 128.8, 125.1, 123.9(q, $J = 274.7$ Hz), 114.1, 100.0(q, $J = 35.3$ Hz), 99.3, 71.1, 69.5, 55.4, 48.7, 31.7, 31.6; ¹⁹F NMR (376 MHz, Chloroform-d) δ -64.93, -72.68; ¹¹B NMR (128 MHz, Chloroform-d) δ 28.60; HRMS (ESI) m/z calcd for C₁₉H₂₄BF₃O₃Na [M+Na]⁺ : 391.1663, found: 391.1674



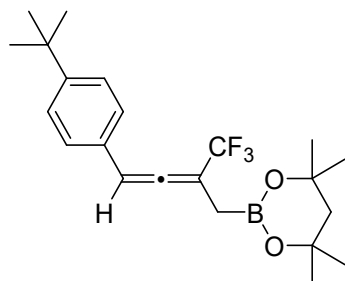
3c

4,4,6,6-tetramethyl-2-(4-(p-tolyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)-1,3,2-dioxaborinane(**3c**):

Pale yellow oil, yield = 78%, 0.0549 g

¹H NMR (500 MHz, Chloroform-d) δ 7.31-7.30 (m, 2H), 7.15-7.12 (m, 2H), 6.47-6.45 (m, 1H), 2.35 (s, 3H), 1.79 (s, 2H), 1.75-1.71 (m, 2H), 1.33 (s, 6H), 1.29 (s, 7H); ¹³C NMR (126 MHz,

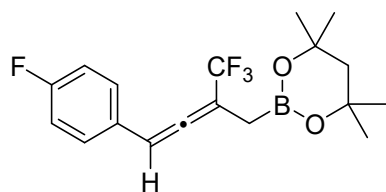
Chloroform-d) δ 204.2 (q, $J = 3.8$ Hz), 137.7, 131.7, 129.8, 129.4, 129.0, 127.5, 124.0 (q, $J = 274.7$ Hz), 119.9, 100.0 (q, $J = 35.3$ Hz), 99.6, 71.1, 48.8, 31.7, 31.6, 21.3; **^{19}F NMR** (471 MHz, Chloroform-d) δ -64.83, -72.61; **^{11}B NMR** (128 MHz, Chloroform-d) δ 28.69; **HRMS** (ESI) m/z calcd for $\text{C}_{19}\text{H}_{24}\text{BF}_3\text{O}_2\text{Na}$ $[\text{M}+\text{Na}]^+$: 375.1714, found: 375.1721



3d

2-(4-(4-(tert-butyl)phenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)-4,4,6,6-tetramethyl-1,3,2-dioxaborinane(**3d**): Pale yellow oil, yield = 69%, 0.0544 g

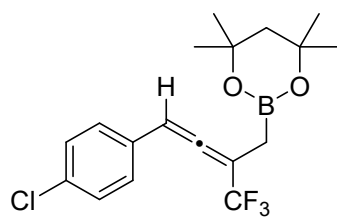
^1H NMR (400 MHz, Chloroform-d) δ 7.33 (s, 4H), 6.47-6.44 (m, 1H), 1.77 (s, 2H), 1.73-1.68 (m, 2H), 1.31 (s, 12H), 1.26 (s, 9H); **^{13}C NMR** (101 MHz, Chloroform-d) δ 204.4 (q, $J = 4.1$ Hz), 150.1, 131.5, 129.8, 127.3, 125.5, 125.2, 123.9 (q, $J = 275.7$ Hz), 99.9 (q, $J = 34.3$ Hz), 99.4, 71.1, 48.7, 34.6, 31.7, 31.5, 31.3; **^{19}F NMR** (376 MHz, Chloroform-d) δ -64.78, -72.65; **^{11}B NMR** (128 MHz, Chloroform-d) δ 28.48; **HRMS** (ESI) m/z calcd for $\text{C}_{22}\text{H}_{30}\text{BF}_3\text{O}_2\text{Na}$ $[\text{M}+\text{Na}]^+$: 417.2183, found: 417.2195



3e

2-(4-(4-fluorophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)-4,4,6,6-tetramethyl-1,3,2-dioxaborinane(**3e**): Yellow oil, yield = 71%, 0.0518 g

^1H NMR (400 MHz, Chloroform-d) δ 7.39-7.35 (m, 2H), 7.03-6.99 (m, 2H), 6.46-6.43 (m, 1H), 1.78 (s, 2H), 1.75-1.69 (m, 2H), 1.31 (s, 6H), 1.26 (s, 6H); **^{13}C NMR** (126 MHz, Chloroform-d) δ 204.26-204.16 (m), 162.5 (d, $J = 247.0$ Hz), 129.2 (d, $J = 8.8$ Hz), 123.8 (q, $J = 274.7$ Hz), 115.6 (d, $J = 21.4$ Hz), 100.4 (q, $J = 34.0$ Hz), 98.8, 71.2, 48.7, 31.7, 31.6; **^{19}F NMR** (471 MHz, Chloroform-d) δ -64.95, -72.60, -111.09, -114.03; **^{11}B NMR** (128 MHz, Chloroform-d) δ 28.63; **HRMS** (ESI) m/z calcd for $\text{C}_{18}\text{H}_{21}\text{BF}_4\text{O}_2\text{Na}$ $[\text{M}+\text{Na}]^+$: 379.1463, found: 379.1477

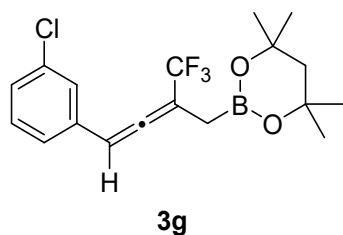


3f

2-(4-(4-chlorophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)-4,4,6,6-tetramethyl-1,3,2-

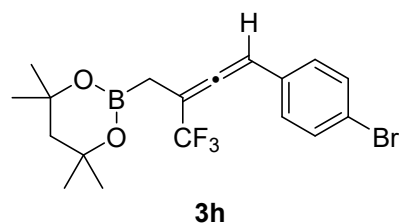
dioxaborinane(**3f**): Brownish-yellow oil, yield = 78%, 0.0580 g

¹H NMR (500 MHz, Chloroform-d) δ 7.39-7.36 (m, 2H), 7.34-7.30 (m, 2H), 6.49-6.45 (m, 1H), 1.83 (s, 2H), 1.79-1.73 (m, 2H), 1.35 (s, 6H), 1.31 (s, 6H); **¹³C NMR** (126 MHz, Chloroform-d) δ 204.5 (q, $J = 3.8$ Hz), 133.5, 131.4, 128.8 (d, $J = 3.8$ Hz), 123.8 (q, $J = 274.5$ Hz), 100.7 (q, $J = 35.3$ Hz), 98.9, 71.2, 48.7, 31.7, 31.6; **¹⁹F NMR** (471 MHz, Chloroform-d) δ -64.90, -72.54; **¹¹B NMR** (128 MHz, Chloroform-d) δ 28.47; **HRMS** (ESI) m/z calcd for $C_{18}H_{21}BClF_3O_2Na$ $[M+Na]^+$: 395.1167, found: 395.1173



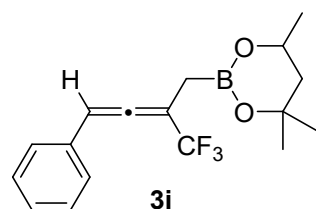
2-(4-(3-chlorophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)-4,4,6,6-tetramethyl-1,3,2-dioxaborinane(**3g**): Brownish-yellow oil, yield = 74%, 0.0551 g

¹H NMR (400 MHz, Chloroform-d) δ 7.37 (s, 1H), 7.27-7.22 (m, 3H), 6.42 (m, 1H), 1.79 (s, 2H), 1.76-1.69 (m, 2H), 1.32 (s, 6H), 1.27 (s, 6H); **¹³C NMR** (126 MHz, Chloroform-d) δ 204.63 (q, $J = 3.8$ Hz), 134.9, 134.7, 129.8, 127.9, 127.3, 125.8, 123.8 (q, $J = 275.9$ Hz), 100.8 (q, $J = 34.0$ Hz), 98.8, 71.2, 48.8, 31.7, 31.6; **¹⁹F NMR** (376 MHz, Chloroform-d) δ -64.86, -72.51; **¹¹B NMR** (128 MHz, Chloroform-d) δ 28.36; **HRMS** (ESI) m/z calcd for $C_{18}H_{21}BClF_3O_2Na$ $[M+Na]^+$: 395.1167, found: 395.1170



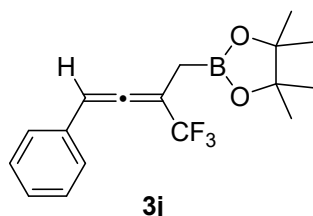
2-(4-(4-bromophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)-4,4,6,6-tetramethyl-1,3,2-dioxaborinane(**3h**): Yellow oil, yield = 63%, 0.0524 g

¹H NMR (400 MHz, Chloroform-d) δ 7.45-7.43 (m, 2H), 7.29-7.26 (m, 2H), 6.43-6.41 (m, 1H), 1.79 (s, 2H), 1.75-1.68 (m, 2H), 1.31 (s, 6H), 1.27 (s, 6H); **¹³C NMR** (101 MHz, Chloroform-d) δ 204.5 (q, $J = 4.2$ Hz), 131.7, 129.1, 123.7 (q, $J = 275.7$ Hz), 121.6, 100.7 (q, $J = 34.3$ Hz), 98.9, 71.2, 48.7, 31.7, 31.6; **¹⁹F NMR** (376 MHz, Chloroform-d) δ -64.90, -72.53; **¹¹B NMR** (128 MHz, Chloroform-d) δ 28.66; **HRMS** (ESI) m/z calcd for $C_{18}H_{21}BBBrF_3O_2Na$ $[M+Na]^+$: 439.0662, found: 439.0668



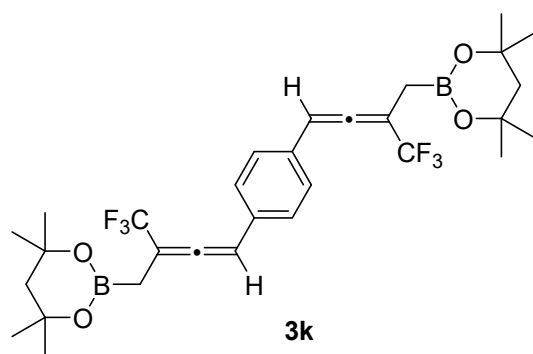
4,4,6-trimethyl-2-(4-phenyl-2-(trifluoromethyl)buta-2,3-dien-1-yl)-1,3,2-dioxaborinane(**3i**): Yellow oil, yield = 50%, 0.0324 g

¹H NMR (400 MHz, Chloroform-d) δ 7.40-7.37 (m, 2H), 7.33-7.30 (m, 2H), 7.26-7.22 (m, 1H), 6.48-6.46 (m, 1H), 4.21-4.09 (m, 1H), 1.75-1.71 (m, 3H), 1.45-1.38 (m, 1H), 1.24 (d, $J = 3.1$ Hz, 3H), 1.22 (s, 1H), 1.11-1.16 (m, 5H); **¹³C NMR** (101 MHz, Chloroform-d) δ 204.5-204. (m), 132.7, 128.6, 128.6, 127.8, 127.6, 127.5, 123.9 (q, $J = 275.7$ Hz), 99.8, 99.0 (q, $J = 35.1$ Hz), 71.3, 65.1, 65.1, 45.8, 45.7, 31.1, 31.0, 28.0, 27.9, 23.0, 22.9; **¹⁹F NMR** (376 MHz, Chloroform-d) δ -64.94, -72.64; **¹¹B NMR** (128 MHz, Chloroform-d) δ 28.82; **HRMS** (ESI) m/z calcd for C₁₇H₂₀BF₃O₂Na [M+Na]⁺: 347.1401, found: 347.1411



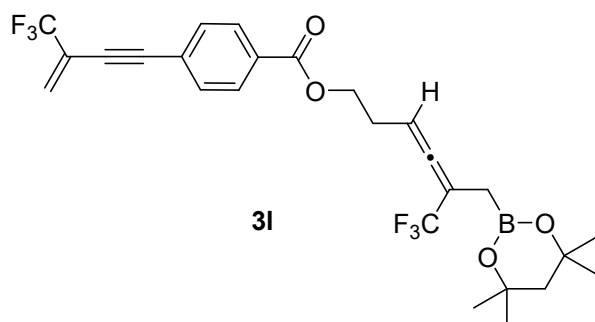
4,4,5,5-tetramethyl-2-(4-phenyl-2-(trifluoromethyl)buta-2,3-dien-1-yl)-1,3,2-dioxaborolane(**3j**): Yellow oil, yield = 57%, 0.0369 g

¹H NMR (400 MHz, Chloroform-d) δ 7.31-7.29 (m, 2H), 7.27-7.22 (m, 2H), 7.20-7.17 (m, 1H), 6.47-6.44 (m, 1H), 1.83-1.73 (m, 2H), 1.14 (s, 6H), 1.11 (s, 6H); **¹³C NMR** (101 MHz, Chloroform-d) δ 204.4 (q, $J = 4.0$ Hz), 131.8, 128.7, 128.2, 128.0, 127.7, 123.6 (q, $J = 274.0$ Hz), 100.5, 99.0 (q, $J = 35.1$ Hz), 83.9, 24.8, 24.7; **¹⁹F NMR** (376 MHz, Chloroform-d) δ -65.19, -72.95; **¹¹B NMR** (128 MHz, Chloroform-d) δ 32.07; **HRMS** (ESI) m/z calcd for C₁₇H₂₀BF₃O₂Na [M+Na]⁺: 347.1401, found: 347.1413



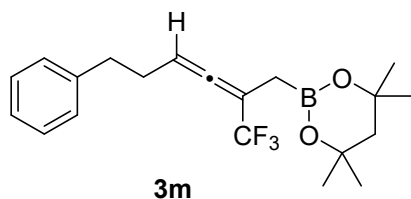
1,4-bis(4,4,4-trifluoro-3-((4,4,6,6-tetramethyl-1,3,2-dioxaborinan-2-yl)methyl)buta-1,2-dien-1-yl)benzene(**3k**): Pale yellow oil, yield = 55%, 0.0658 g

¹H NMR (400 MHz, Chloroform-d) δ 7.34-7.33 (m, 4H), 6.47-6.45 (m, 2H), 1.77 (d, $J = 2.7$ Hz, 4H), 1.74-1.68 (m, 4H), 1.30 (d, $J = 2.2$ Hz, 12H), 1.26 (d, $J = 3.3$ Hz, 12H); **¹³C NMR** (126 MHz, Chloroform-d) δ 204.7 (q, $J = 5.0$ Hz), 132.3, 127.8, 123.9 (q, $J = 274.7$ Hz), 100.3 (q, $J = 35.3$ Hz), 99.6, 71.1, 48.7, 31.7, 31.7; **¹⁹F NMR** (376 MHz, Chloroform-d) δ -64.89, -72.56; **¹¹B NMR** (128 MHz, Chloroform-d) δ 28.30; **HRMS** (ESI) m/z calcd for C₃₀H₃₈B₂F₆O₄Na [M+Na]⁺: 621.2753, found: 621.2766



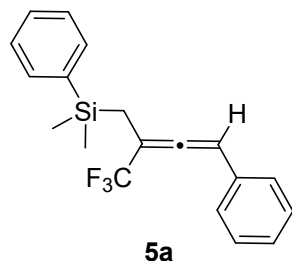
6,6,6-trifluoro-5-((4,4,6,6-tetramethyl-1,3,2-dioxaborinan-2-yl)methyl)hexa-3,4-dien-1-yl 4-(3-(trifluoromethyl)but-3-en-1-yn-1-yl)benzoate(**3l**): Pale yellow oil, yield = 50%, 0.0528 g

¹H NMR (500 MHz, Chloroform-*d*) δ 8.01-7.98 (m, 2H), 7.49-7.46 (m, 2H), 6.52-6.50 (m, 1H), 6.01 (d, J = 1.3 Hz, 1H), 5.81 (s, 1H), 4.46 (t, J = 6.7 Hz, 2H), 2.84 (t, J = 6.7 Hz, 2H), 1.79 (s, 2H), 1.77-1.70 (m, 2H), 1.31 (s, 6H), 1.27 (s, 6H); **¹³C NMR** (101 MHz, Chloroform-*d*) δ 205.4 (q, J = 4.0 Hz), 166.0, 138.1, 130.0, 128.9, 127.5, 126.7 (q, J = 4.5 Hz), 125.1, 123.8 (q, J = 275.7 Hz), 122.7 (q, J = 35.4 Hz), 121.3 (q, J = 274.7 Hz), 101.0, 99.2, 97.9 (q, J = 35.0 Hz), 90.3, 74.8 (q, J = 2.2 Hz), 71.2, 62.1, 48.7, 31.6, 31.6, 20.0; **¹⁹F NMR** (376 MHz, Chloroform-*d*) δ -64.83, -68.30, -72.45, -72.56; **¹¹B NMR** (128 MHz, Chloroform-*d*) δ 28.18; **HRMS** (ESI) m/z calcd for C₂₆H₂₇B₂F₆O₄Na [M+Na]⁺: 551.1799, found: 551.1821



4,4,6,6-tetramethyl-2-(6-phenyl-2-(trifluoromethyl)hexa-2,3-dien-1-yl)-1,3,2-dioxaborinane(**3m**): Pale yellow oil, yield = 66%, 0.0480 g

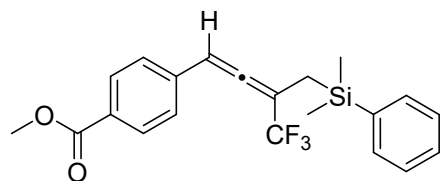
¹H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.26 (m, 2H), 7.22 – 7.18 (m, 3H), 5.58 – 5.55 (m, 0.4H), 2.82 – 2.69 (m, 2H), 2.46 – 2.35 (m, 2H), 1.80 (s, 2H), 1.62 – 1.52 (m, 1H), 1.3 (s, 12H); **¹³C NMR** (101 MHz, Chloroform-*d*) δ 202.7 (q, J = 4.0 Hz), 141.5, 140.7, 128.5, 128.5, 128.4, 128.4, 126.3, 126.0, 124.3 (q, J = 275.7 Hz), 96.7 (q, J = 35.4 Hz), 96.3, 82.6, 70.9, 70.9, 48.8, 48.8, 35.2, 34.7, 31.7, 31.7, 31.7, 20.9; **¹⁹F NMR** (376 MHz, Chloroform-*d*) δ -65.05, -72.95; **¹¹B NMR** (128 MHz, Chloroform-*d*) δ 28.72; **HRMS** (ESI) m/z calcd for C₂₀H₂₆BF₃O₂Na [M+Na]⁺: 389.1870, found: 389.1883



Dimethyl(phenyl)(4-phenyl-2-(trifluoromethyl)buta-2,3-dien-1-yl)silane(**5a**): Pale yellow oil, 64%, 0.0193 g

¹H NMR (400 MHz, Chloroform-*d*) δ 7.47-7.45 (m, 2H), 7.33 – 7.23 (m, 6H), 7.15 – 7.12 (m, 2H), 6.37-6.34 (m, 1H), 1.87 – 1.79 (m, 2H), 0.34 (s, 3H), 0.33 (s, 3H); **¹³C NMR** (101 MHz, Chloroform-*d*) δ 204.6 (q, J = 4.0 Hz), 137.6, 133.6, 132.1, 129.3, 128.8, 128.0, 127.8, 127.4, 123.7

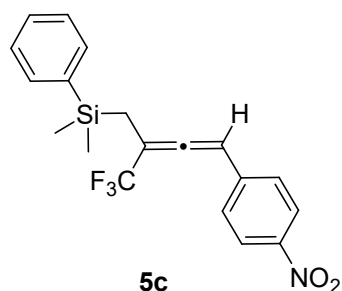
(q, $J = 274.7$ Hz), 101.0, 99.7 (q, $J = 34.3$ Hz), 14.5, -2.9; ^{19}F NMR (376 MHz, Chloroform-d) δ -64.73, -73.30; HRMS (ESI) m/z calcd for $\text{C}_{19}\text{H}_{19}\text{F}_3\text{Si}$ $[\text{M}+\text{H}]^+$:332.1203, found: 332.1210



5b

Methyl 4-(3-((dimethyl(phenyl)silyl)methyl)-4,4,4-trifluorobuta-1,2-dien-1-yl)benzoate (**5b**): Pale yellow oil, 68%, 0.0193 g

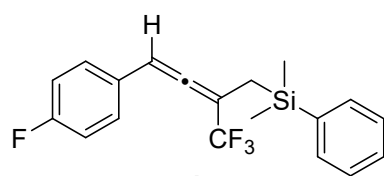
^1H NMR (400 MHz, Chloroform-d) δ 7.96 – 7.93 (m, 2H), 7.47 - 7.45 (m, 2H), 7.34 – 7.27 (m, 3H), 7.17 – 7.15 (m, 2H), 6.39-6.37 (m, 1H), 3.92 (s, 3H), 1.90 - 1.81 (m, 2H), 0.36 (s, 3H), 0.34 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-d) δ 205.4 (q, $J = 4.1$ Hz), 166.7, 137.3, 137.0, 133.6, 130.0, 129.5, 129.4, 127.9, 127.2, 123.5 (q, $J = 275.7$ Hz), 100.4, 100.3 (q, $J = 35.4$ Hz), 52.2, 14.4, -2.9, -2.9; ^{19}F NMR (376 MHz, Chloroform-d) δ -64.66, -73.15; HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{22}\text{F}_3\text{O}_2\text{Si}$ $[\text{M}+\text{H}]^+$: 391.1336, found: 391.1341



5c

Dimethyl(4-(4-nitrophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)(phenyl)silane (**5c**): Pale yellow oil, 64%, 0.0193 g

^1H NMR (400 MHz, Chloroform-d) δ 7.54 – 7.50 (m, 2H), 7.45-7.43 (m, 2H), 7.33 – 7.24 (m, 3H), 7.13 – 7.10 (m, 2H), 6.35-6.33 (m, 1H), 1.90-1.82 (m, 2H), 0.36 (s, 3H), 0.34 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-d) δ 205.63 (q, $J = 4.0$ Hz), 137.2, 137.0, 133.6, 132.5, 129.4, 127.9, 127.8, 123.4 (q, $J = 275.7$ Hz), 118.8, 111.2, 100.6 (q, $J = 35.4$ Hz), 99.9, 14.4, -2.8, -2.9; ^{19}F NMR (376 MHz, Chloroform-d) δ -64.61, -72.62; HRMS (ESI) m/z calcd for $\text{C}_{19}\text{H}_{19}\text{F}_3\text{NO}_2\text{Si}$ $[\text{M}+\text{H}]^+$: 378.1132, found: 378.1151

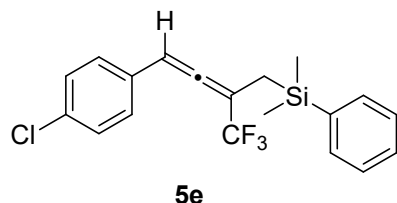


5d

(4-(4-fluorophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)dimethyl(phenyl)silane (**5d**): Yellow oil, yield = 80%, 0.0280 g

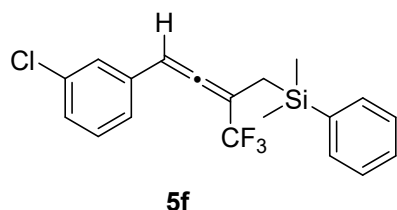
^1H NMR (500 MHz, Chloroform-d) δ 7.49-7.45 (m, 2H), 7.35-7.29 (m, 3H), 7.09 -7.06 (m, 2H), 7.00-6.95 (m, 2H), 6.35-6.33 (m, 1H), 1.88-1.82 (m, 2H), 0.36 (s, 3H), 0.34 (s, 3H); ^{13}C NMR (101

MHz, Chloroform-d) δ 206.6 (q, $J = 2.0$ Hz), 164.8 (d, $J = 248.5$ Hz), 139.8, 135.9, 131.6, 131.3 (d, $J = 8.1$ Hz), 130.2, 125.9 (q, $J = 274.7$ Hz), 118.1 (d, $J = 22.2$ Hz), 102.3, 102.3 (q, $J = 34.8$ Hz), 16.8, -0.52, -0.60; ^{19}F NMR (471 MHz, Chloroform-d) δ -64.79, -73.29, -110.47, -113.46; HRMS (ESI) m/z calcd for $\text{C}_{19}\text{H}_{19}\text{F}_4\text{Si}$ $[\text{M}+\text{H}]^+$: 351.1187, found: 351.1194



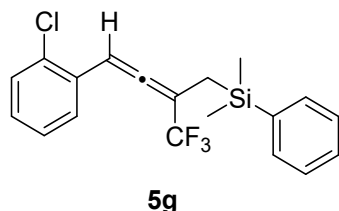
(4-(4-chlorophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)dimethyl(phenyl)silane(**5e**): Yellow oil, yield = 77%, 0.0281 g

^1H NMR (400 MHz, Chloroform-d) δ 7.48-7.44 (m, 2H), 7.35-7.27 (m, 3H), 7.26-7.22 (m, 2H), 7.04-7.00 (m, 2H), 6.32-6.30 (m, 1H), 1.88-1.80 (m, 2H), 0.35 (s, 3H), 0.33 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-d) δ 204.6 (q, $J = 4.0$ Hz), 137.4, 133.7, 133.6, 130.6, 129.3, 128.9, 128.5, 127.9, 123.5 (q, $J = 275.7$ Hz), 100.2 (q, $J = 35.4$ Hz), 100.0, 14.5, -2.8, -2.9; ^{19}F NMR (376 MHz, Chloroform-d) δ -64.77; HRMS (ESI) m/z calcd for $\text{C}_{19}\text{H}_{19}\text{ClF}_3\text{Si}$ $[\text{M}+\text{H}]^+$: 367.0891, found: 367.0923



(4-(3-chlorophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)dimethyl(phenyl)silane(**5f**): Pale yellow oil, yield = 99%, 0.0362 g

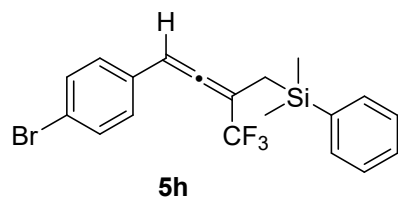
^1H NMR (400 MHz, Chloroform-d) δ 7.48-7.45 (m, 2H), 7.33-7.27 (m, 3H), 7.23-7.20 (m, 2H), 7.10-7.09 (m, 1H), 7.01 - 6.96 (m, 1H), 6.30-6.28 (m, 1H), 1.89-1.81 (m, 2H), 0.36 (s, 3H), 0.34 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-d) δ 204.7 (q, $J = 3.0$ Hz), 137.3, 134.7, 133.6, 129.9, 129.3, 128.1, 127.8, 127.2, 125.5, 123.5 (q, $J = 275.7$ Hz), 100.4 (q, $J = 34.3$ Hz), 100.0, 14.4, -2.9, -3.0; ^{19}F NMR (376 MHz, Chloroform-d) δ -64.76, -73.22; HRMS (ESI) m/z calcd for $\text{C}_{19}\text{H}_{19}\text{ClF}_3\text{Si}$ $[\text{M}+\text{H}]^+$: 367.0891, found: 367.0915



(4-(2-chlorophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)dimethyl(phenyl)silane(**5g**): Amber oil, yield = 96%, 0.0351 g

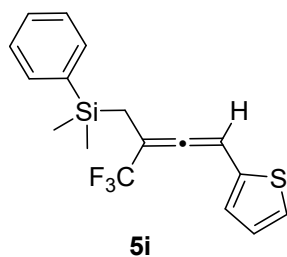
^1H NMR (400 MHz, Chloroform-d) δ 7.48-7.45 (m, 2H), 7.35 - 7.27 (m, 4H), 7.20 - 7.13 (m, 3H), 6.823-6.80 (m, 1H), 1.88-1.81 (m, 2H), 0.36 (s, 3H), 0.34 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-

d) δ 205.4 (q, $J = 4$ Hz), 137.3, 133.6, 132.6, 129.8, 129.3, 129.1, 128.9, 127.8, 127.0, 123.6 (q, $J = 275.7$ Hz), 100.4 (q, $J = 35.4$ Hz), 97.4, 14.5, -2.9, -3.0; ^{19}F NMR (376 MHz, Chloroform-d) δ -64.69; HRMS (ESI) m/z calcd for $\text{C}_{19}\text{H}_{19}\text{ClF}_3\text{Si}$ $[\text{M}+\text{H}]^+$: 367.0891, found: 367.0911



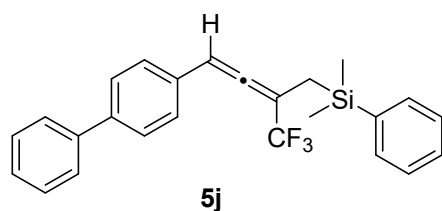
(4-(4-bromophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)dimethyl(phenyl)silane(**5h**): Pale yellow oil, yield = 77%, 0.0316 g

^1H NMR (400 MHz, Chloroform-d) δ 7.48-7.45 (m, 2H), 7.41 – 7.38 (m, 2H), 7.35-7.28 (m 3H), 6.98 – 6.95(m, 2H), 6.31-6.29 (m, 1H), 1.88-1.81 (m, 2H), 0.35 (s, 3H), 0.34 (s, 3H); ^{13}C NMR (126 MHz, Chloroform-d) δ 204.6 (q, $J = 3.0$ Hz), 137.4, 133.6, 131.9, 129.4, 128.9, 127.9, 123.5 (q, $J = 274.7$ Hz), 121.9, 100.3 (q, $J = 35.3$ Hz), 100.1, 14.5, -2.8, -2.9; ^{19}F NMR (376 MHz, Chloroform-d) δ -64.74 – -64.76 (m), -73.22; HRMS (ESI) m/z calcd for $\text{C}_{19}\text{H}_{19}\text{BrF}_3\text{Si}$ $[\text{M}+\text{H}]^+$: 411.0386, found: 411.0397



Dimethyl(phenyl)(4-(thiophen-2-yl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)silane(**5i**): Pale yellow oil, yield = 77%, 0.0260 g

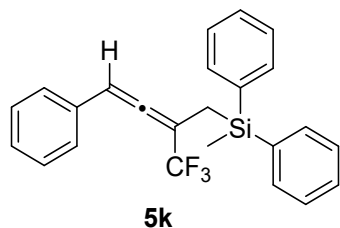
^1H NMR (500 MHz, Chloroform-d) δ 7.55 – 7.53 (m, 2H), 7.40 – 7.36 (m, 3H), 7.30 – 7.27 (m, 1H), 7.02-7.00 (m, 1H), 6.95-6.94 (m, 1H), 6.65-6.63 (m, 1H), 1.89-1.88 (m, 2H), 0.43 (s, 3H), 0.42 (s, 3H); ^{13}C NMR (126 MHz, Chloroform-d) δ 204.1 (q, $J = 3.8$ Hz), 137.7, 135.8, 133.6, 129.3, 127.9, 127.6, 126.7, 126.2, 123.4 (q, $J = 275.9$ Hz), 99.9 (q, $J = 35.3$ Hz), 95.10, 14.76, -2.92; ^{19}F NMR (471 MHz, Chloroform-d) δ -64.99, -73.09; HRMS (ESI) m/z calcd for $\text{C}_{17}\text{H}_{18}\text{F}_3\text{SSi}$ $[\text{M}+\text{H}]^+$: 339.0845, found: 339.0886.



(4-([1,1'-biphenyl]-4-yl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)dimethyl(phenyl)silane(**5j**): Pale yellow oil, yield = 66%, 0.0269 g

^1H NMR (400 MHz, Chloroform-d) δ 7.52 – 7.50 (m, 2H), 7.45 – 7.35 (m, 6H), 7.29 – 7.21 (m, 4H), 7.13 – 7.11 (m, 2H), 6.34 - 6.32 (m, 1H), 1.82 – 1.74 (m, 2H), 0.29 (s, 3H), 0.27 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-d) δ 204.8 (q, $J = 4.0$ Hz), 140.9, 140.6, 137.6, 133.6, 131.1, 129.3, 127.9, 127.8, 127.5, 127.0, 123.7 (q, $J = 275.7$ Hz), 100.6, 99.8 (q, $J = 35.4$ Hz), 14.5, -2.8, -2.9;

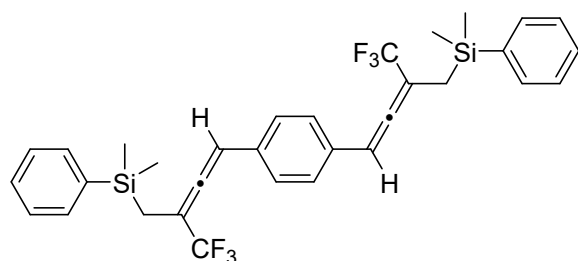
^{19}F NMR (376 MHz, Chloroform-d) δ -64.69; HRMS (ESI) m/z calcd for $\text{C}_{25}\text{H}_{23}\text{F}_3\text{Si}$ $[\text{M}+\text{H}]^+$: 408.1521, found: 408.1537



5k

Methyl(diphenyl(4-phenyl-2-(trifluoromethyl)buta-2,3-dien-1-yl)silane(**5k**): Pale yellow oil, yield = 50%, 0.0197 g

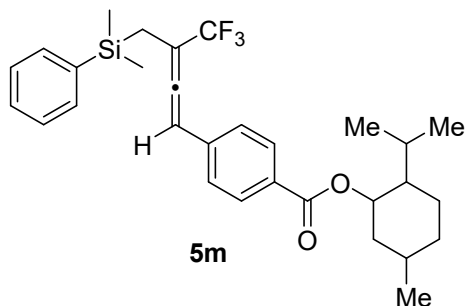
^1H NMR (400 MHz, Chloroform-d) δ 7.50 – 7.7 (m, 4H), 7.35 – 7.28 (m, 6H), 7.23 – 7.21 (m, 3H), 7.00 - 6.98 (m, 2H), 6.20 - 6.17 (m, 1H), 2.21 – 2.12 (m, 2H), 0.63 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-d) δ 204.9 (q, $J = 4.0$ Hz), 135.7, 134.5, 134.4, 131.9, 131.7, 129.5 (d, $J = 2.0$ Hz), 128.6, 127.9, 127.4, 123.7 (q, $J = 275.7$ Hz), 99.1 (q, $J = 35.4$ Hz), 12.80, -4.35; ^{19}F NMR (376 MHz, Chloroform-d) δ -64.94, -73.24; HRMS (ESI) m/z calcd for $\text{C}_{24}\text{H}_{22}\text{F}_3\text{Si}$ $[\text{M}+\text{H}]^+$: 395.1437, found: 395.1459



5l

1,4-bis(3-((dimethyl(phenyl)silyl)methyl)-4,4,4-trifluorobuta-1,2-dien-1-yl)benzene(**5l**): Pale yellow oil, yield = 73%, 0.0428 g

^1H NMR (400 MHz, Chloroform-d) δ 7.49-7.45 (m, 4H), 7.3 – 7.28 (m, 6H), 7.06 - 7.01 (m, 4H), 6.35-6.33(m, H), 1.88 – 1.81 (m, 4H), 0.36 (s, 6H), 0.34 (s, 3H), 0.34 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-d) δ 204.93 – 204.76 (m), 137.5, 137.5, 133.6, 132.1, 129.3, 127.9, 127.8, 127.7, 123.8(q, $J = 274.7$ Hz), 100.6, 99.9 (q, $J = 34.4$ Hz), 14.5, -2.9; ^{19}F NMR (376 MHz, Chloroform-d) δ -64.74, -73.25; HRMS (ESI) m/z calcd for $\text{C}_{32}\text{H}_{33}\text{F}_6\text{Si}_2$ $[\text{M}+\text{H}]^+$: 587.2019, found: 587.2033

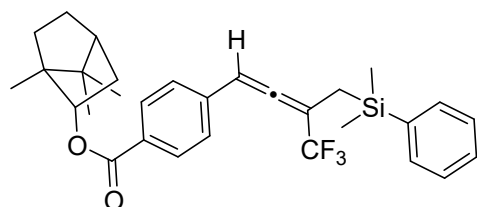


5m

2-isopropyl-5-methylcyclohexyl 4-(3-((dimethyl(phenyl)silyl)methyl)-4,4,4-trifluorobuta-1,2-dien-1-yl)benzoate(**5m**): Pale yellow oil, yield = 60%, 0.0308 g

^1H NMR (400 MHz, Chloroform-d) δ 7.97 – 7.93 (m, 2H), 7.47 – 7.45 (m, 2H), 7.33 – 7.27 (m, 2H), 7.19-7.14 (m, 2H), 6.40 - 6.37 (m, 1H), 4.96 - 4.90 (m, 1H), 2.16 – 2.12 (m, 1H), 1.98 – 1.85

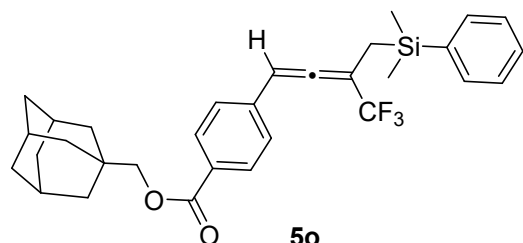
(m, 3H), 1.76-1.72 (m, 2H), 1.60 – 1.53 (m, 2H), 1.17 – 1.08 (m, 2H), 0.95 – 0.92 (m, 9H), 0.82-0.79 (m, 4H), 0.36 (s, 3H), 0.34 (s, 2H), 0.34 (s, 1H); ^{13}C NMR (126 MHz, Chloroform-d) δ 205.5 (d, $J = 3.7$ Hz), 165.8, 137.3, 136.8, 133.6, 130.0, 129.4, 127.9, 127.2, 126.4, 123.6 (q, $J = 272.5$ Hz), 100.4, 100.3 (q, $J = 35.3$ Hz), 75.0, 47.3, 41.0, 34.4, 31.5, 26.6, 23.7, 22.1, 20.8, 16.7, 14.5, -2.8, -2.9; ^{19}F NMR (376 MHz, Chloroform-d) δ -64.68, -72.38; HRMS (ESI) m/z calcd for $\text{C}_{30}\text{H}_{38}\text{F}_3\text{O}_2\text{Si}$ $[\text{M}+\text{H}]^+$:515.2588, found: 515.2594



5n

1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl 4-(3-((dimethyl(phenyl)silyl)methyl)-4,4,4-trifluorobuta-1,2-dien-1-yl)benzoate(**5n**): Pale yellow oil, yield = 55%, 0.0282 g

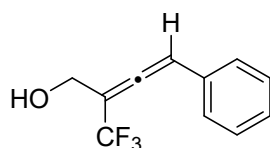
^1H NMR (500 MHz, Chloroform-d) δ 7.98-7.95 (m, 2H), 7.47 – 7.46 (m, 2H), 7.35 – 7.28 (m, 3H), 7.19-7.16 (m, 2H), 6.40-6.38 (m, 1H), 5.13-5.10 (m, 1H), 2.51-2.46 (m, 1H), 2.15 – 2.10 (m, 1H), 1.85 (t, $J = 2.9$ Hz, 2H), 1.80-1.74 (m, 2H), 1.36 – 1.28 (m, 2H), 1.14-1.10 (m, 1H), 0.97 (s, 3H), 0.94 – 0.92 (s, 6H), 0.36 (s, 3H), 0.34 (s, 3H); ^{13}C NMR (126 MHz, Chloroform-d) δ 205.5 (q, $J = 3.8$ Hz), 166.5, 137.3, 136.8, 133.6, 131.7, 130.0, 129.4, 127.9, 127.3, 126.4, 123.6 (q, $J = 274.7$ Hz), 100.5 (q, $J = 35.3$ Hz), 100.4, 80.7, 49.2, 48.0, 45.0, 37.0, 28.2, 27.5, 19.8, 19.0, 14.4, 13.7, -2.8, -2.9; ^{19}F NMR (471 MHz, Chloroform-d) δ -64.66, -72.35; HRMS (ESI) m/z calcd for $\text{C}_{30}\text{H}_{36}\text{F}_3\text{O}_2\text{Si}$ $[\text{M}+\text{H}]^+$:513.2431, found: 513.2437



5o

((1s,3s)-adamantan-1-yl)methyl 4-(3-((dimethyl(phenyl)silyl)methyl)-4,4,4-trifluorobuta-1,2-dien-1-yl)benzoate(**5o**): Pale yellow oil, yield = 77%, 0.0299 g

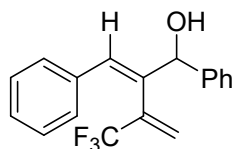
^1H NMR (400 MHz, Chloroform-d) δ 7.90 – 7.88 (m, 2H), 7.47-7.45 (m, 2H), 7.35 – 7.30 (m, 3H), 7.16 – 7.14 (m, 2H), 6.39-6.36 (m, 1H), 2.26 (s, 7H), 1.85-1.84 (m, 2H), 1.76 – 1.68 (m, 8H), 1.38 – 1.24 (m, 2H), 0.35 (s, 3H), 0.33 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-d) δ 205.3 (q, $J = 3.9$ Hz), 165.1, 137.4, 136.3, 133.6, 131.5, 129.9, 129.3, 127.9, 127.1, 123.5 (q, $J = 123.5$ Hz), 100.5, 100.2 (q, $J = 35.4$ Hz), 81.2, 41.4, 36.3, 30.9, 14.4, -2.9; ^{19}F NMR (376 MHz, Chloroform-d) δ -64.70, -72.39; HRMS (ESI) m/z calcd for $\text{C}_{31}\text{H}_{36}\text{F}_3\text{O}_2\text{Si}$ $[\text{M}+\text{H}]^+$:525.2431, found: 525.2449



8a

4-phenyl-2-(trifluoromethyl)buta-2,3-dien-1-ol(**8a**): pale yellow oil, yield = 88%, 0.0214 g

$^1\text{H NMR}$ (400 MHz, Chloroform- d) δ 7.39 – 7.29 (m, 5H), 6.76-6.74 (m, 1H), 4.45-4.38 (m, 2H), 1.85 (s, 1H); $^{13}\text{C NMR}$ (126 MHz, Chloroform- d) δ 204.32 – 204.09 (m), 130.99, 129.06, 128.78, 127.62, 123.0 (q, $J = 276.0$ Hz), 103.3 (q, $J = 33.1$ Hz), 102.75, 58.97; $^{19}\text{F NMR}$ (471 MHz, Chloroform- d) δ -62.16; **HRMS** (ESI) m/z calcd for $\text{C}_{11}\text{H}_{10}\text{F}_3\text{O} [\text{M}+\text{H}]^+$: 215.0678, found: 215.0684



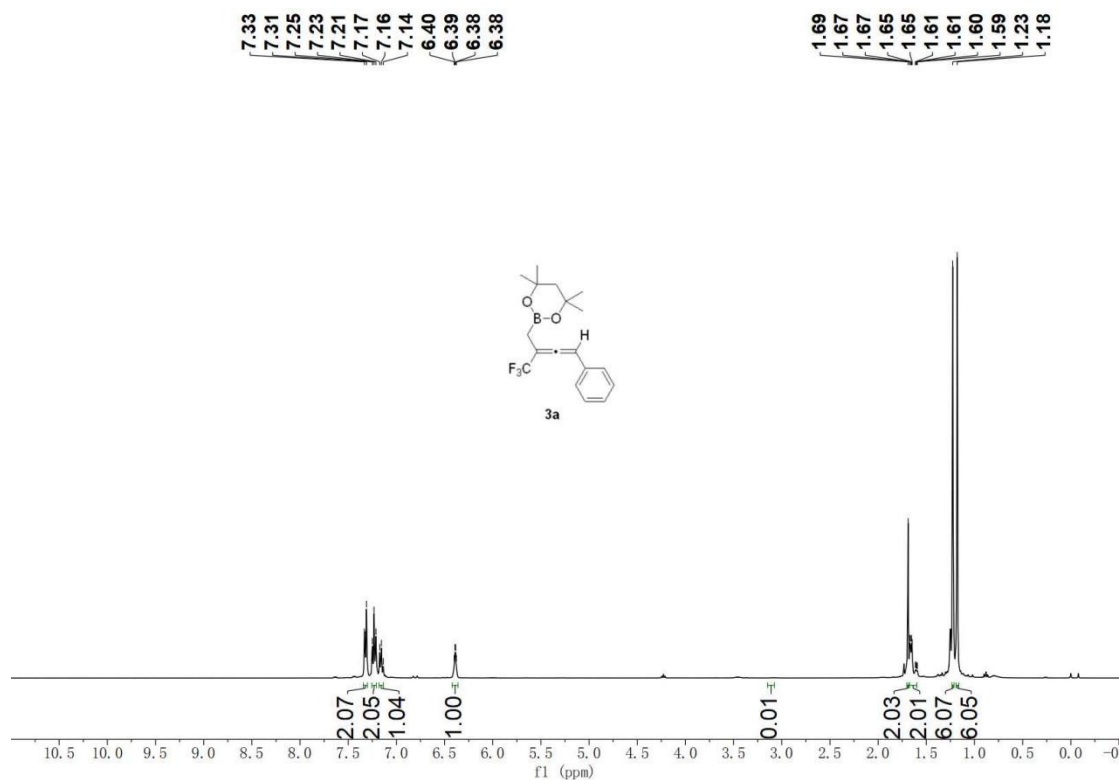
9a

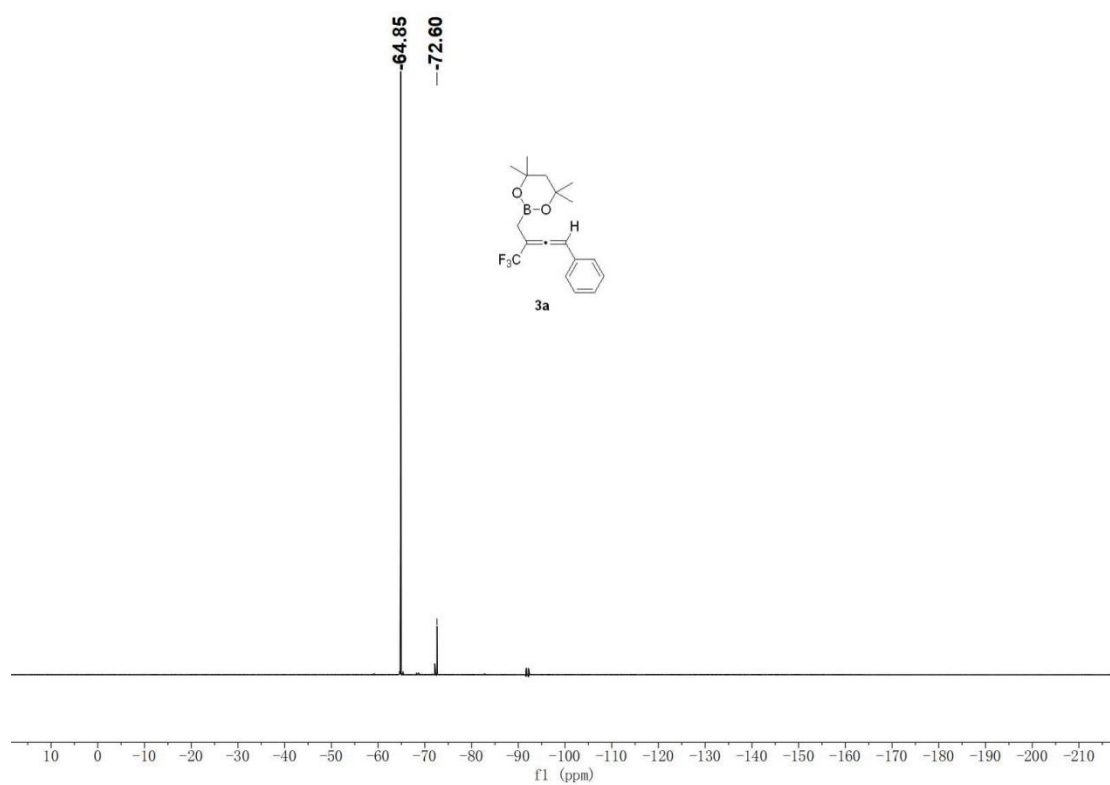
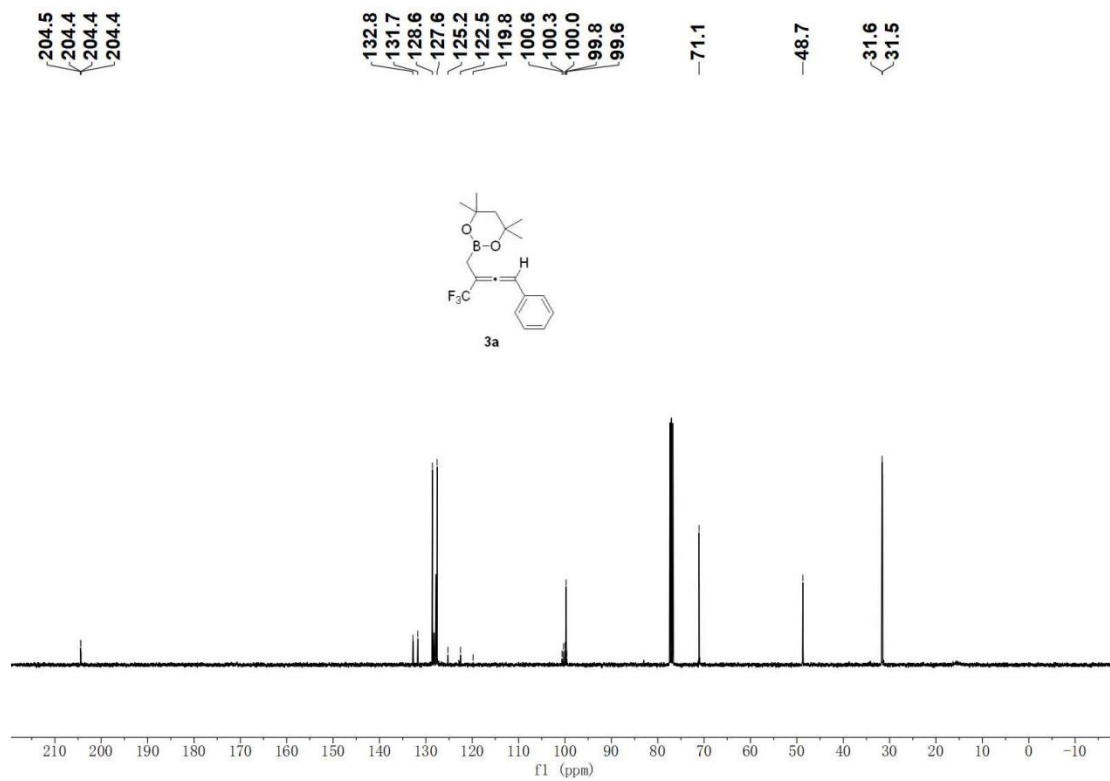
(*E*)-2-benzylidene-1-phenyl-3-(trifluoromethyl)but-3-en-1-ol (**9a**): Pale yellow oil, yield = 77%, 0.0234 g

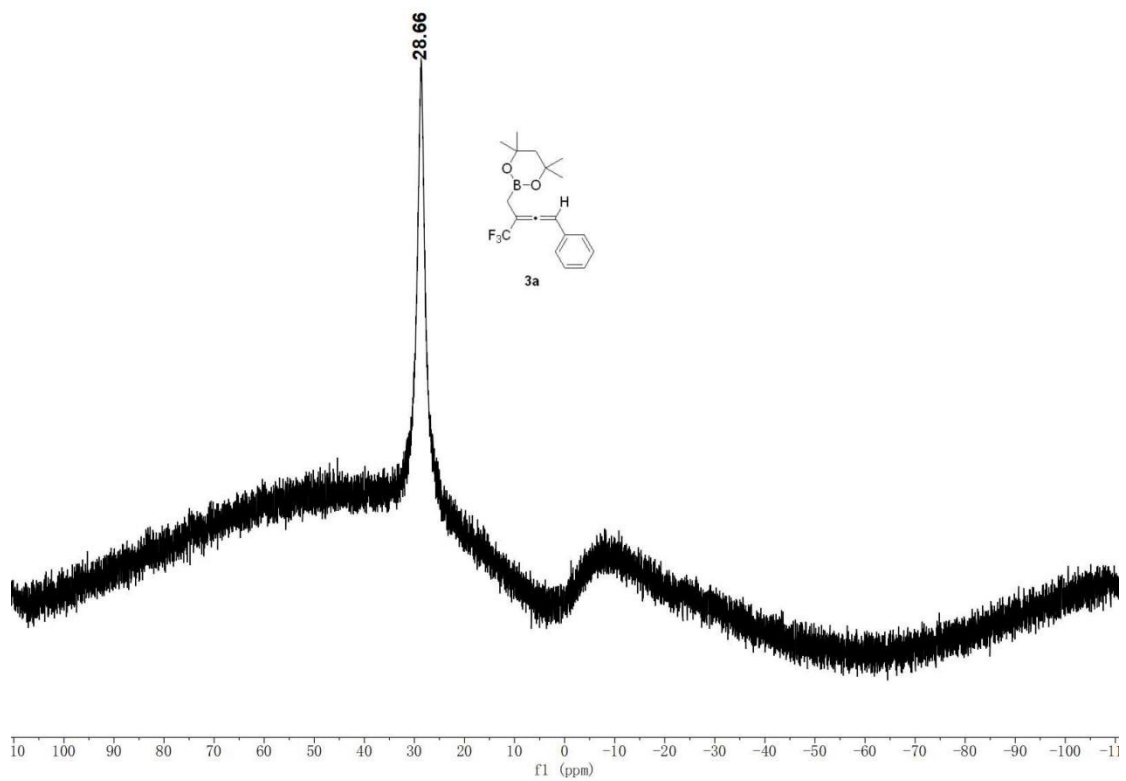
$^1\text{H NMR}$ (500 MHz, Chloroform- d) δ 7.42 – 7.33 (m, 6H), 7.32 – 7.27 (m, 3H), 7.25 – 7.21 (m, 1H), 7.06 (s, 1H), 5.78 – 5.77 (d, $J = 5$ Hz, 1H), 5.44 (s, 1H), 4.99 (s, 1H); $^{13}\text{C NMR}$ (126 MHz, Chloroform- d) δ 140.8, 136.8, 135.7, 135.3 (q, $J = 31.5$ Hz), 131.0, 129.0, 128.6, 128.2, 128.2, 127.7, 127.3, 126.0 (q, $J = 5.0$ Hz), 123.0 (q, $J = 274.7$ Hz), 76.5; $^{19}\text{F NMR}$ (471 MHz, Chloroform- d) δ -64.35; **HRMS** (ESI) m/z calcd for $\text{C}_{18}\text{H}_{15}\text{F}_3\text{ONa} [\text{M}+\text{Na}]^+$: 327.0667, found: 327.0688

4. Copies of the NMR spectra

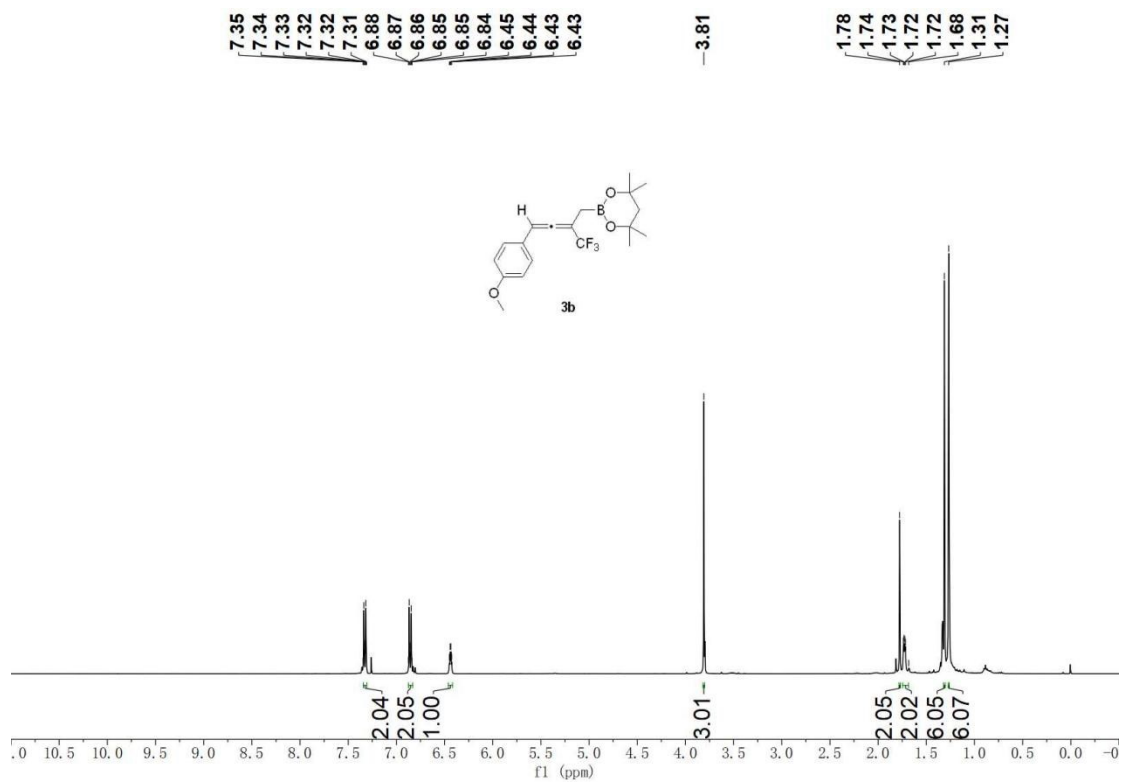
4,4,6,6-tetramethyl-2-(4-phenyl-2-(trifluoromethyl)buta-2,3-dien-1-yl)-1,3,2-dioxaborinane(3a)

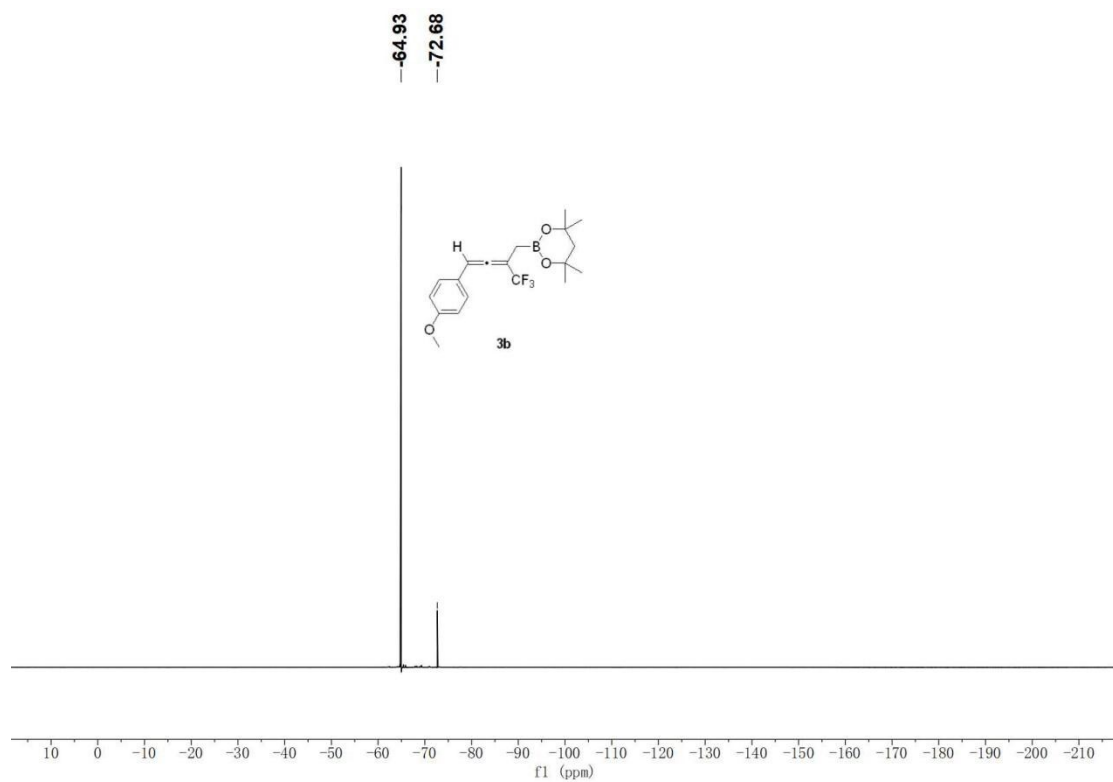
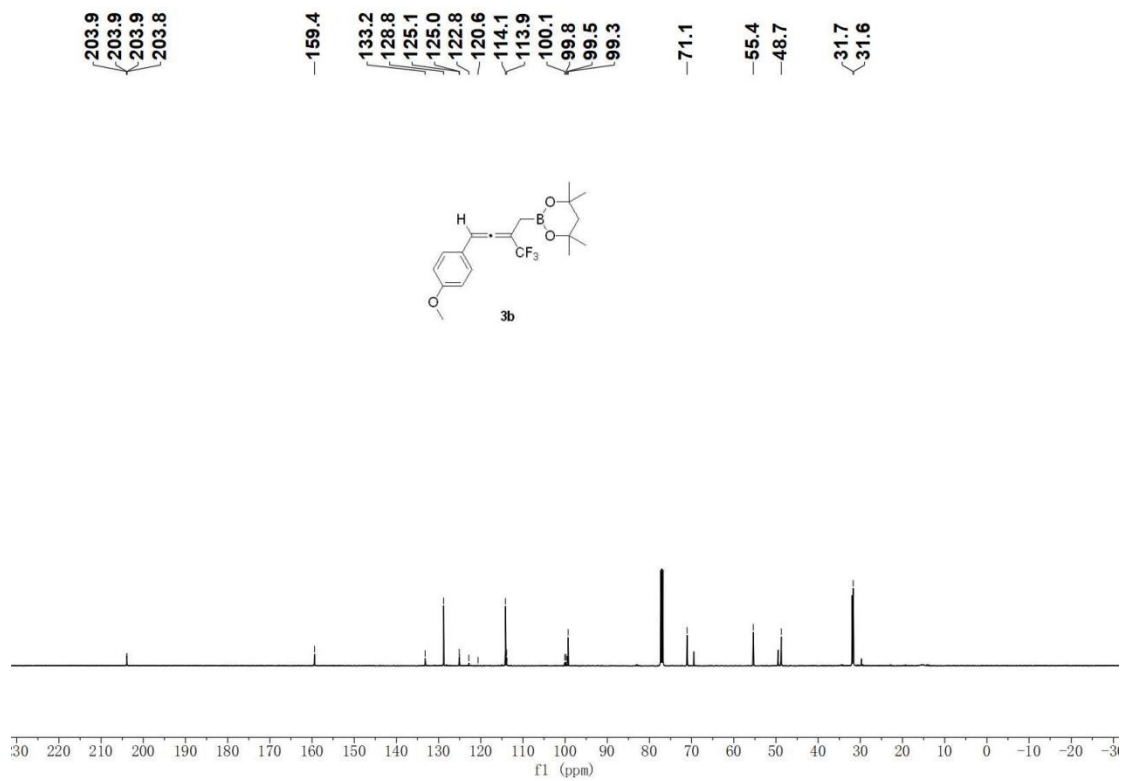


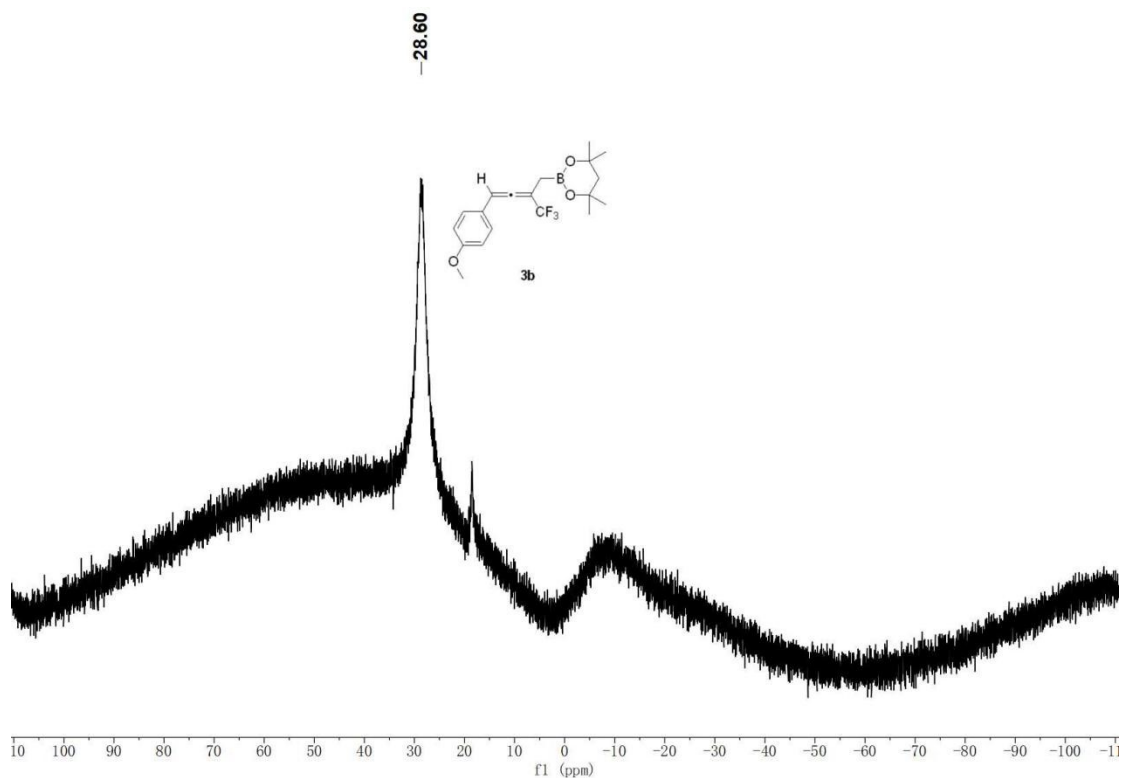




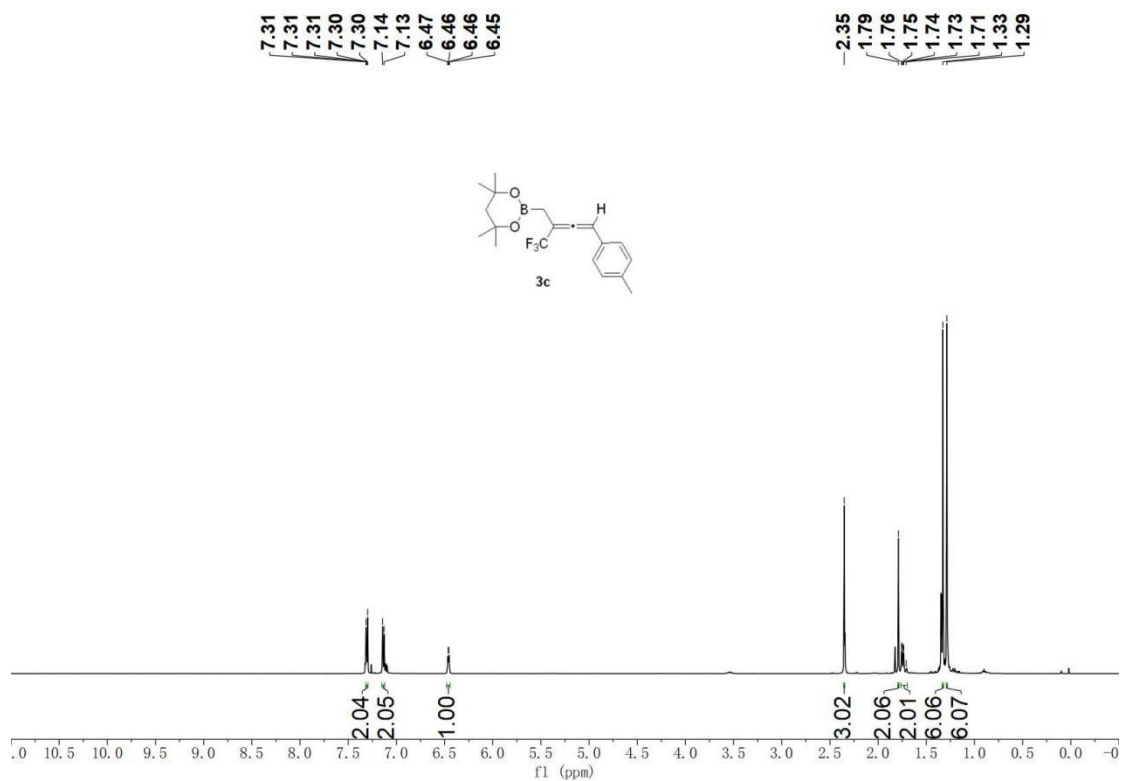
2-(4-(4-methoxyphenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)-4,4,6,6-tetramethyl-1,3,2-dioxaborinane(3b)

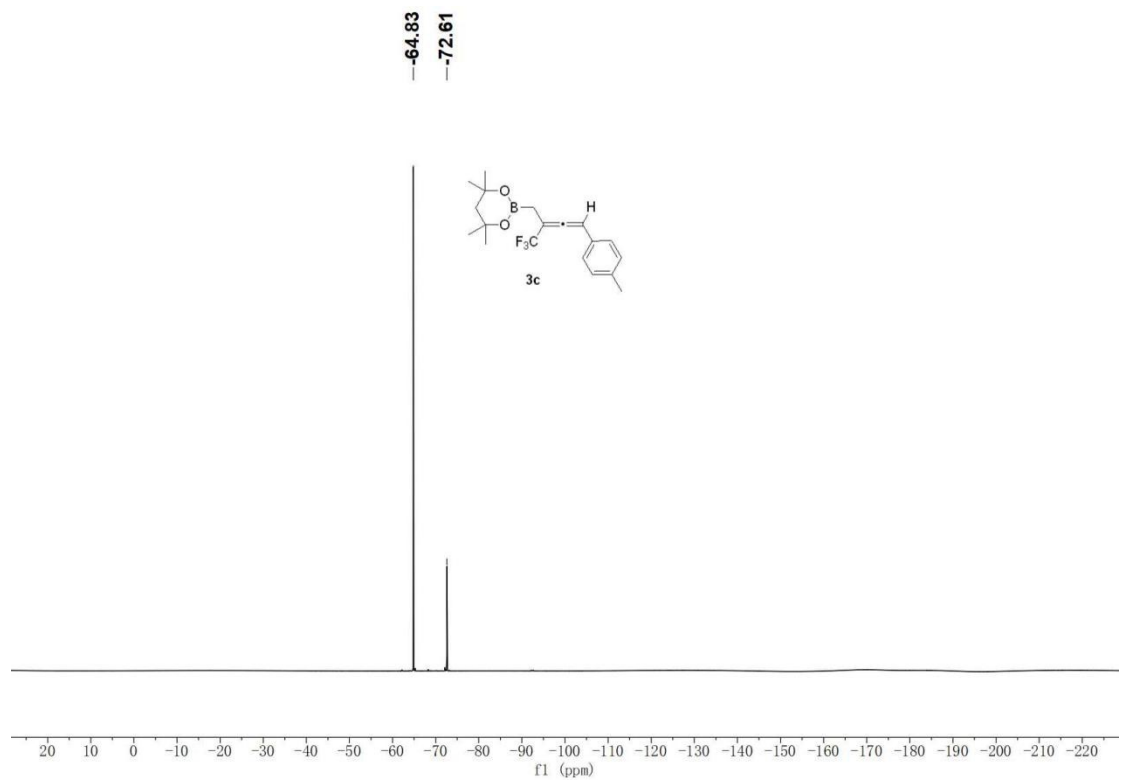
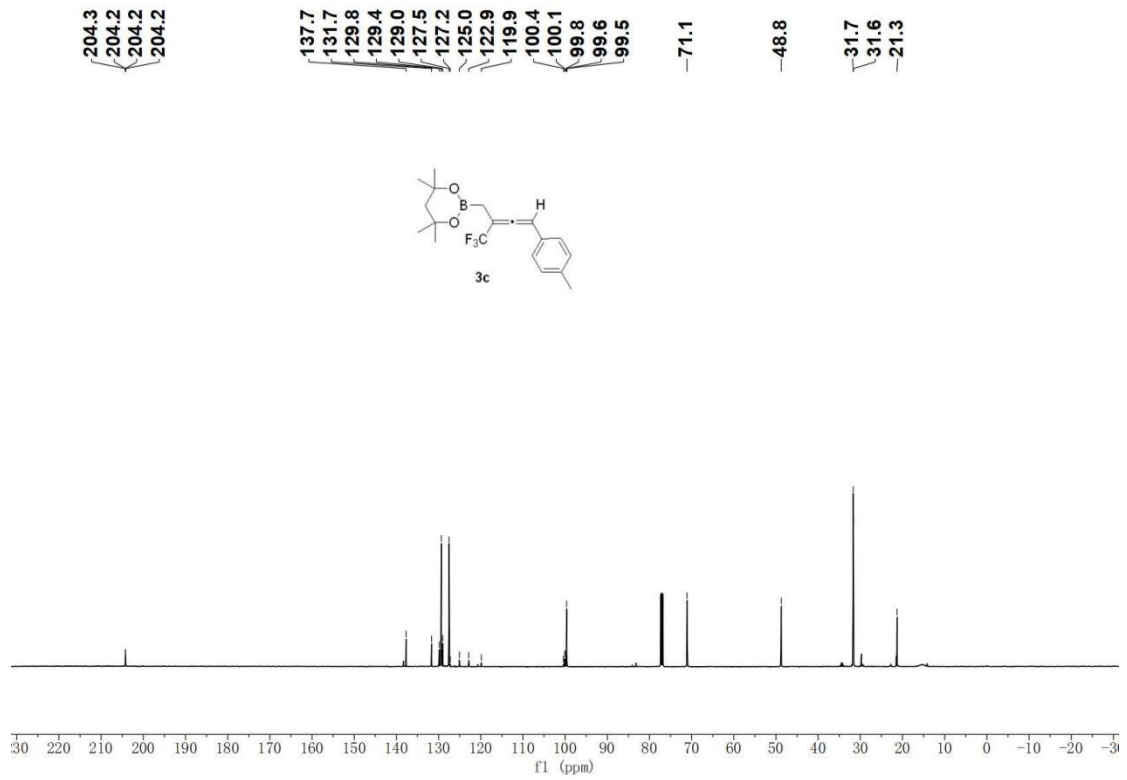


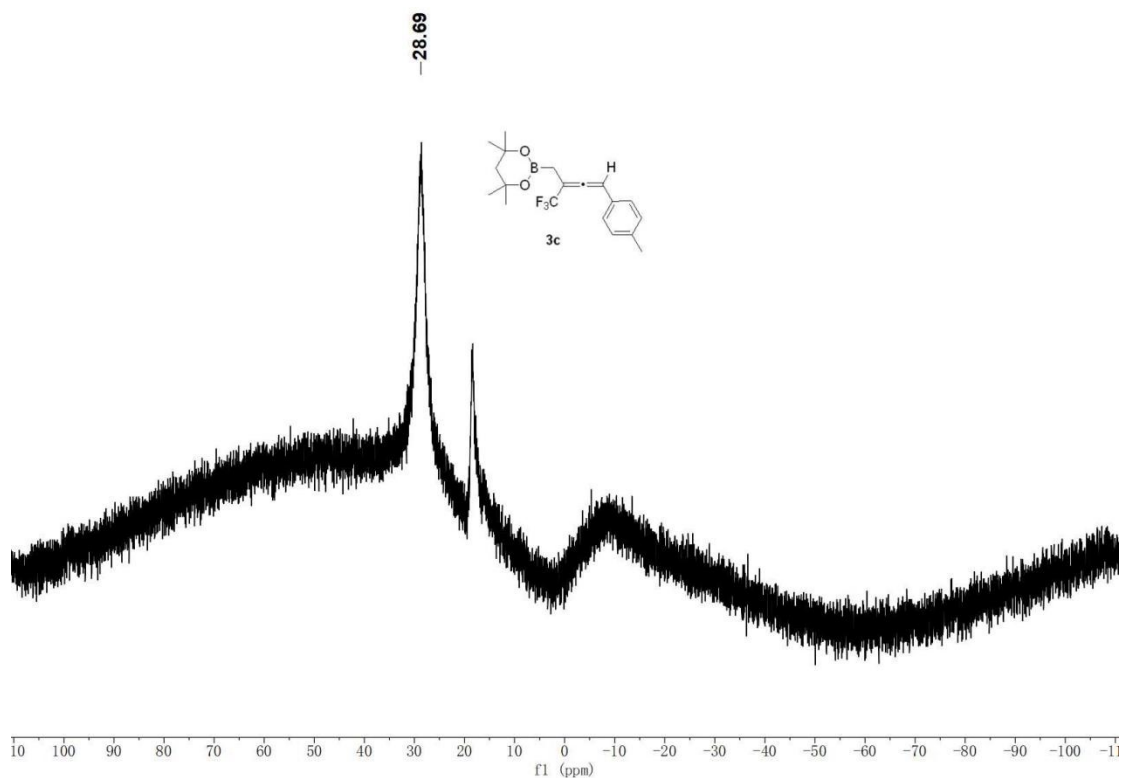




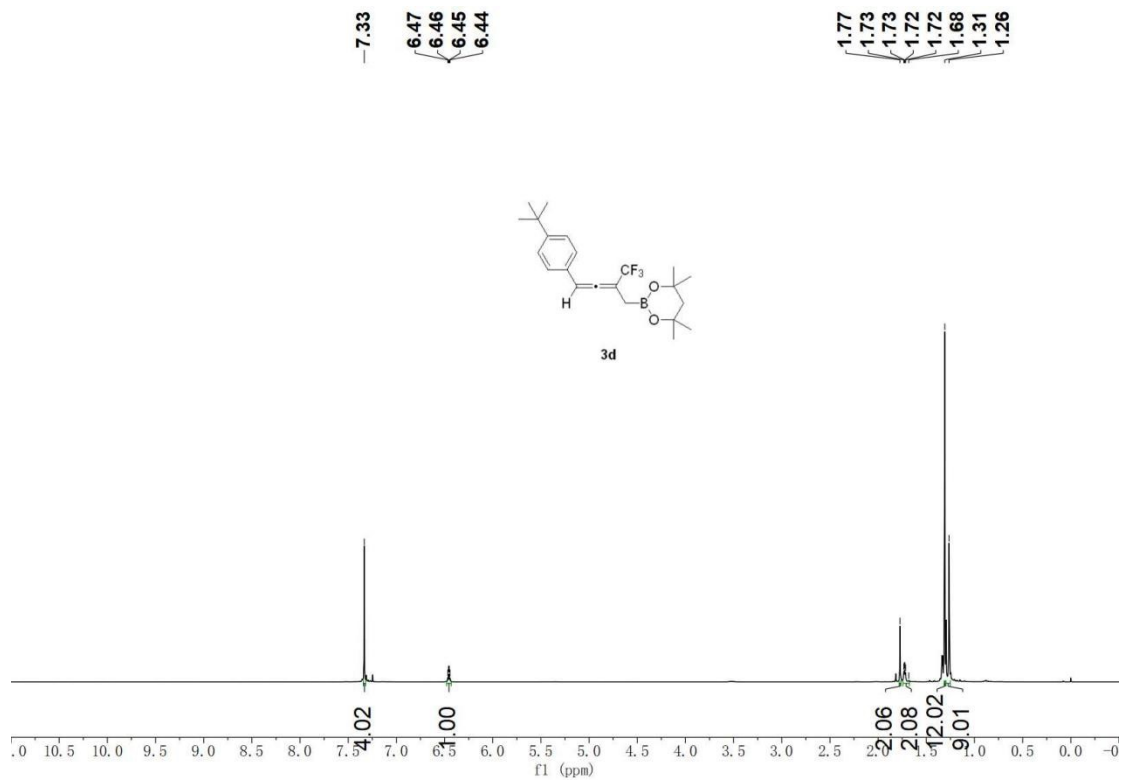
4,4,6,6-tetramethyl-2-(4-(p-tolyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)-1,3,2-dioxaborinane (3c)

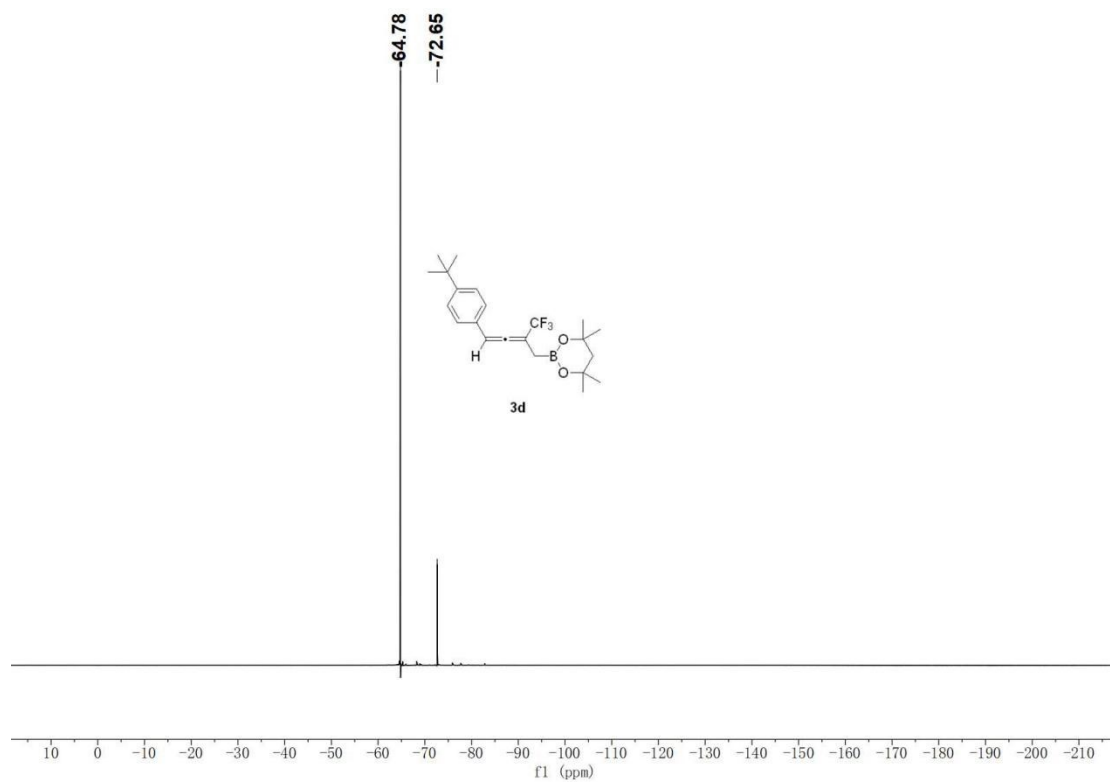
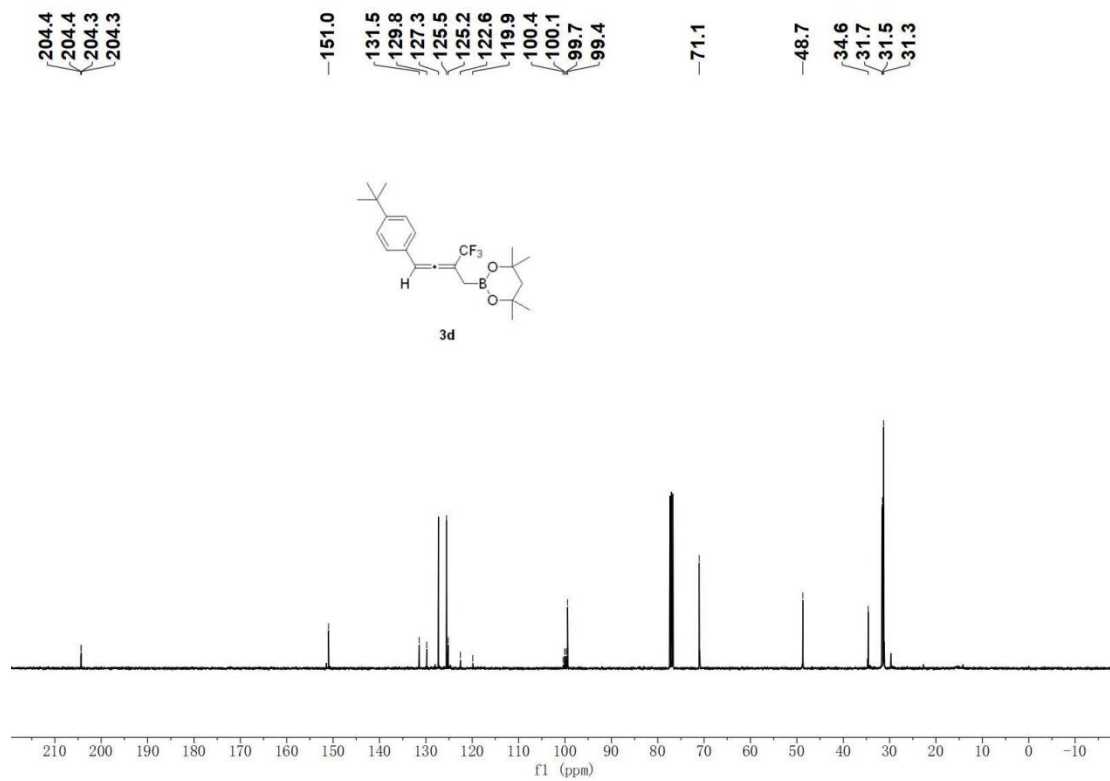


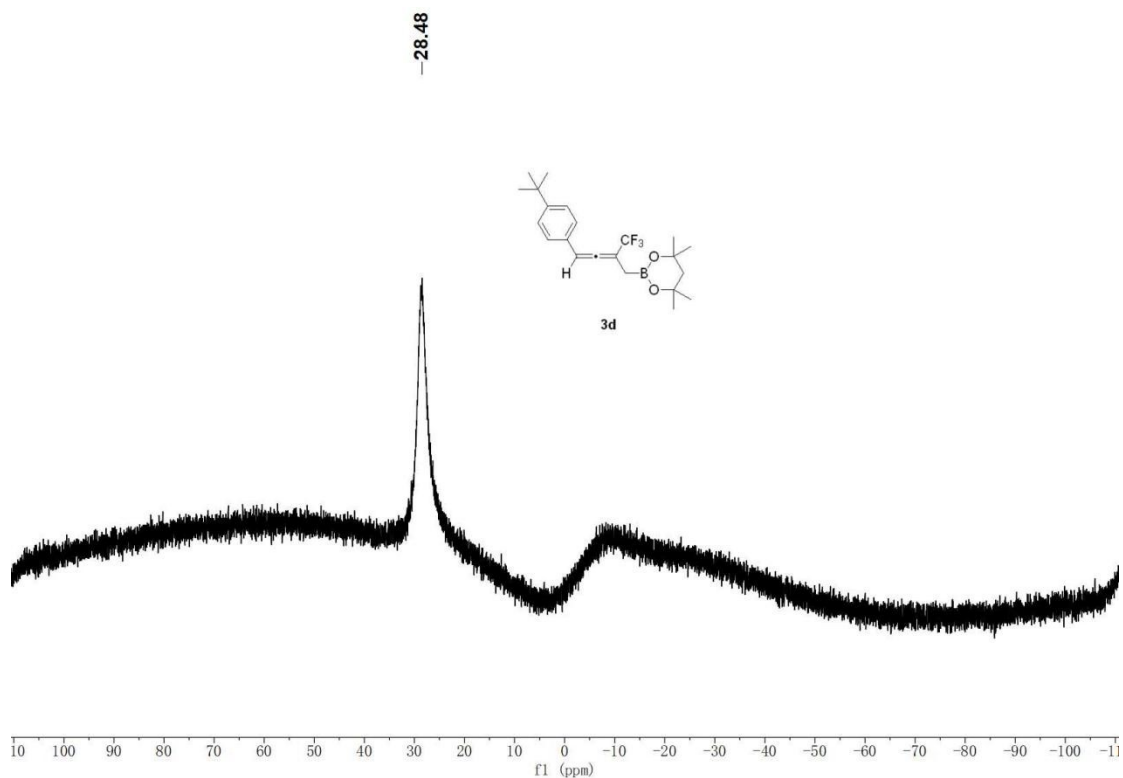




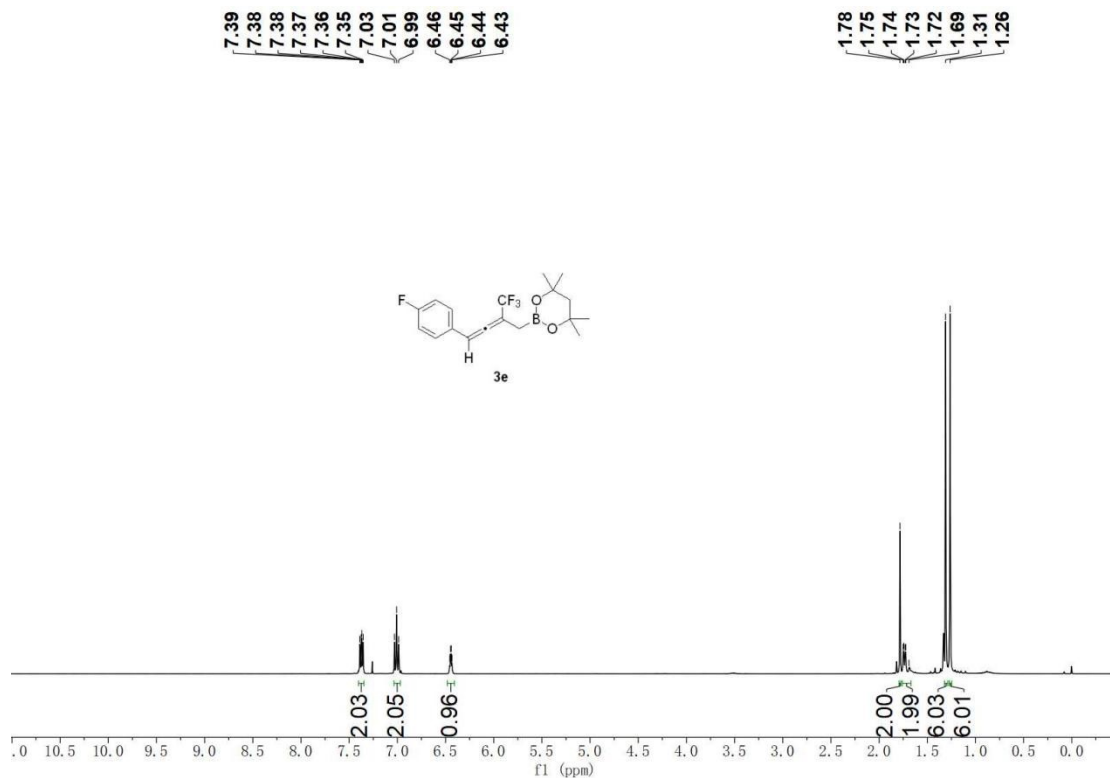
2-(4-(4-(*tert*-butyl)phenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)-4,4,6,6-tetramethyl-1,3,2-dioxaborinane (**3d**)

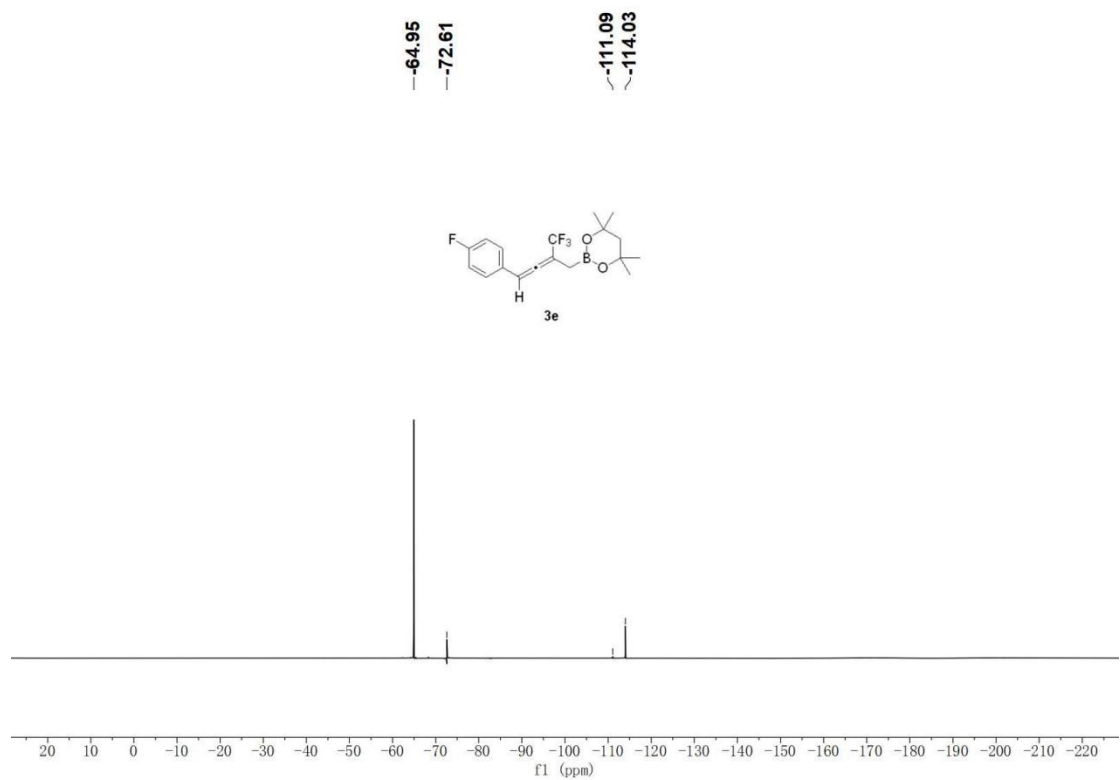
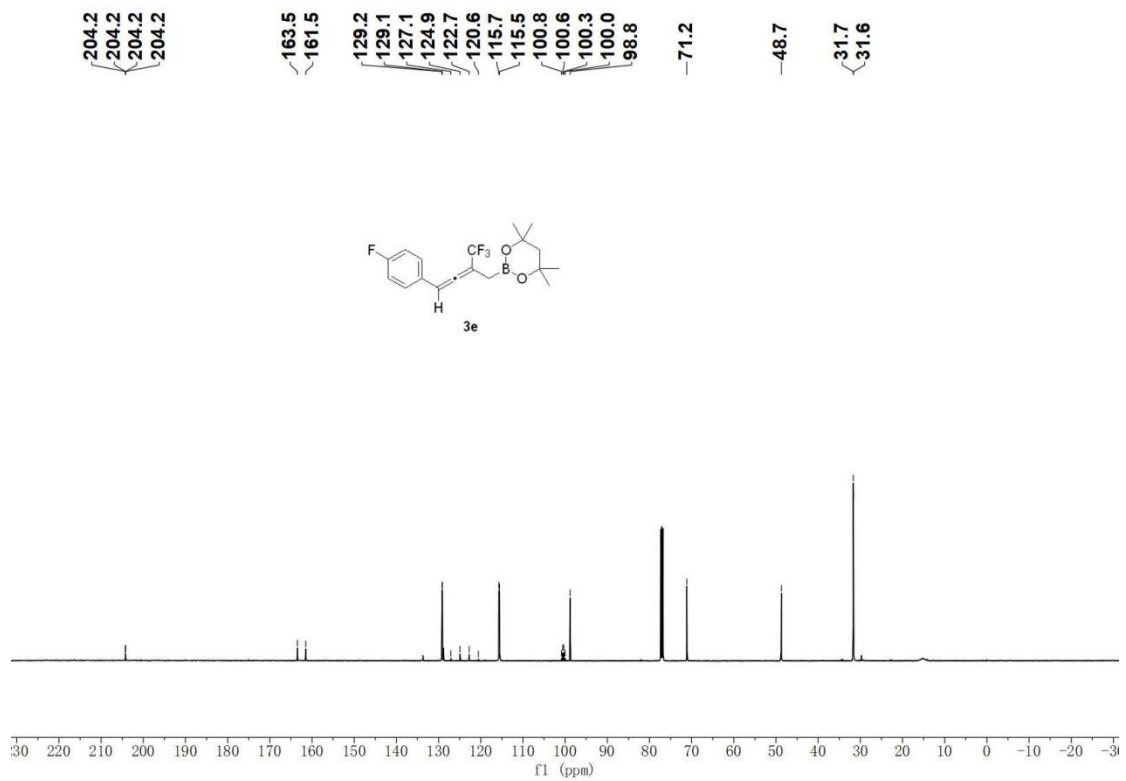


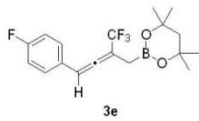
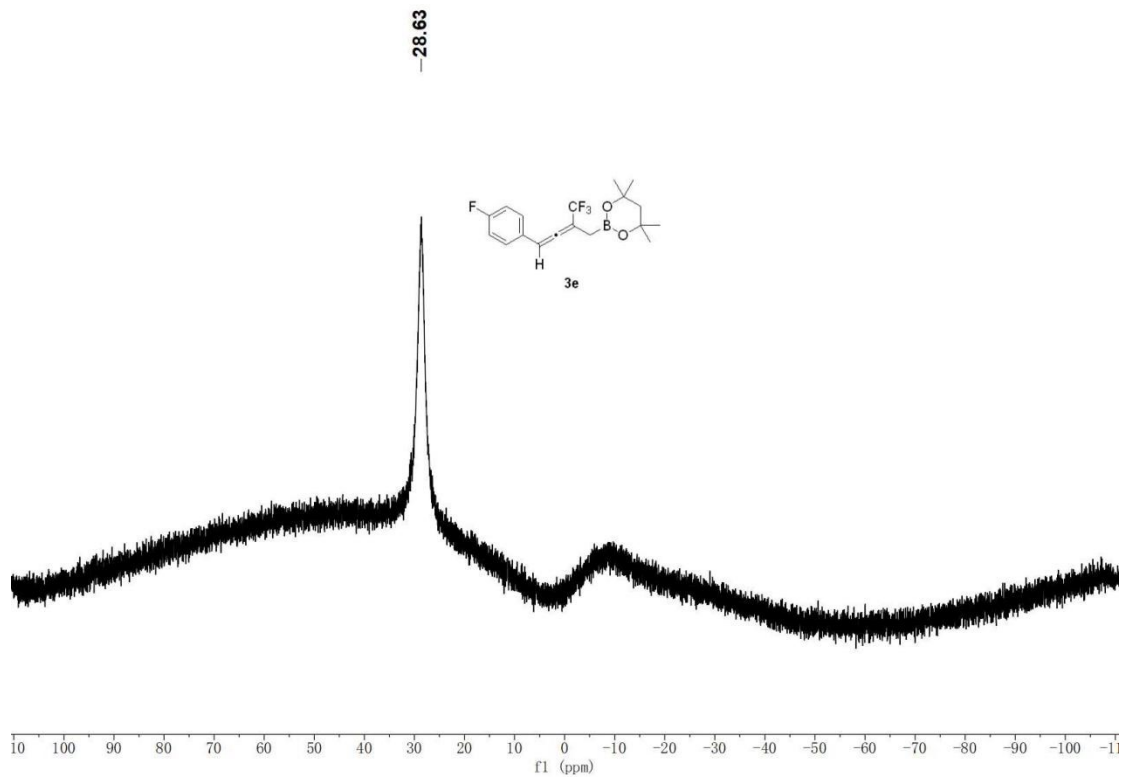




2-(4-(4-chlorophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)-4,4,6,6-tetramethyl-1,3,2-dioxaborinane (3e)



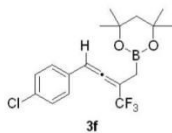
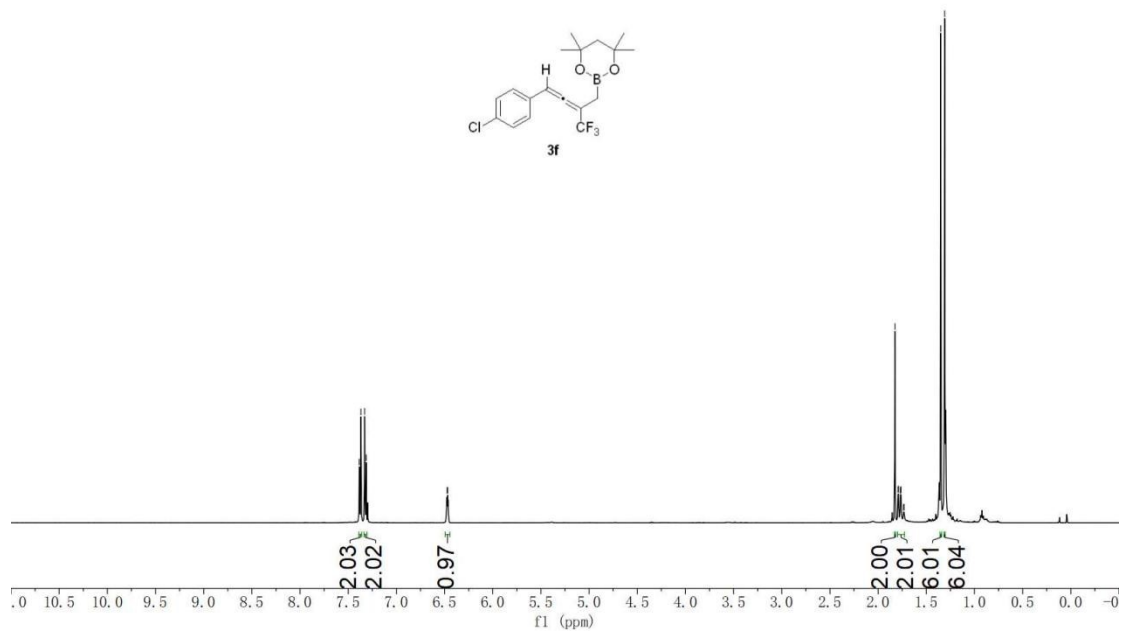


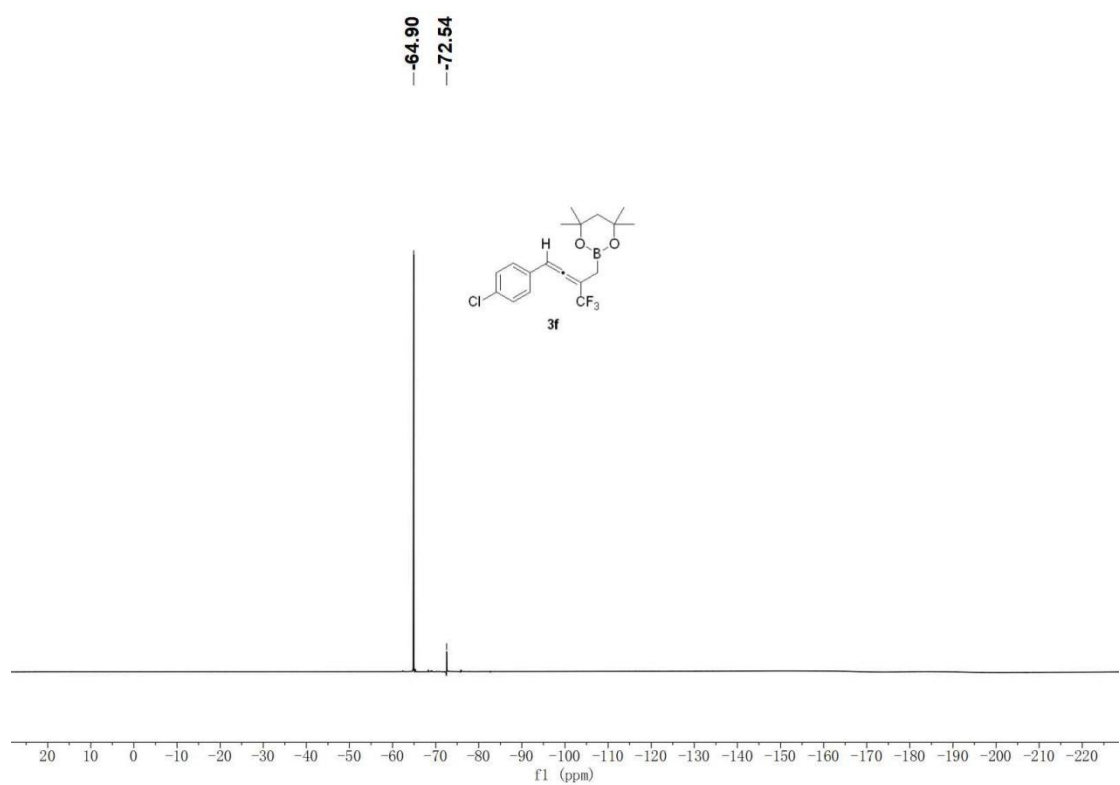
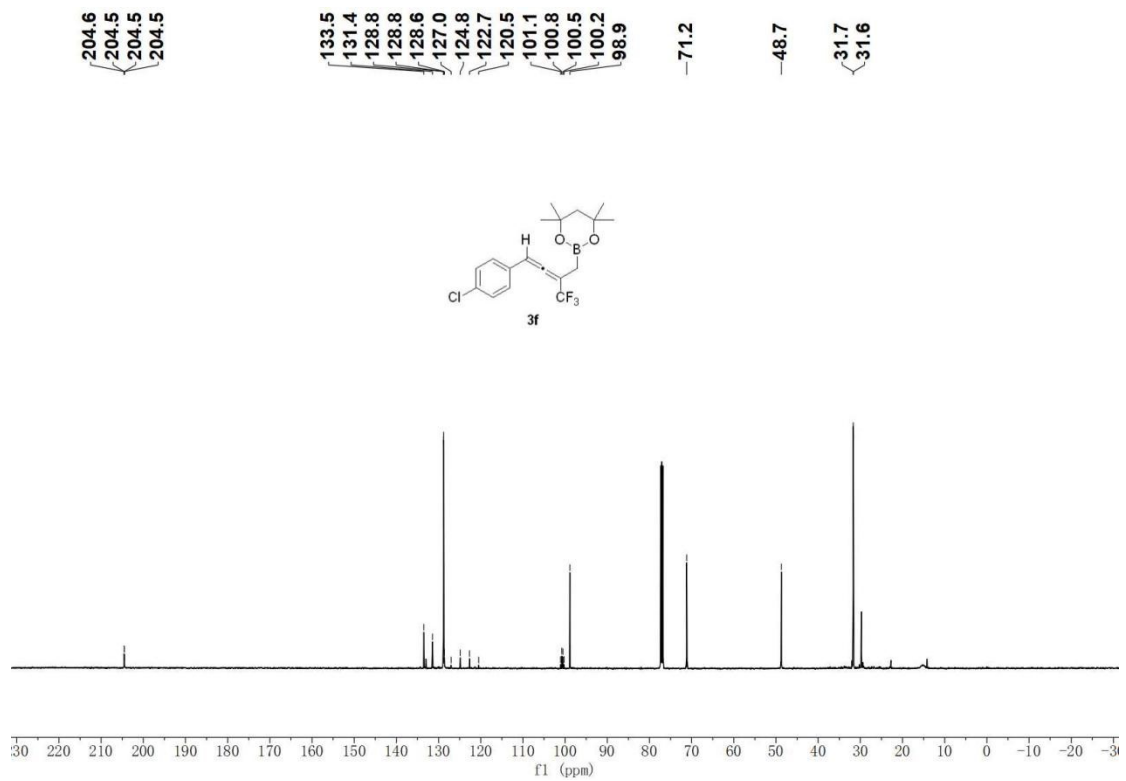


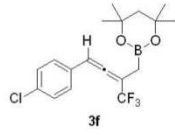
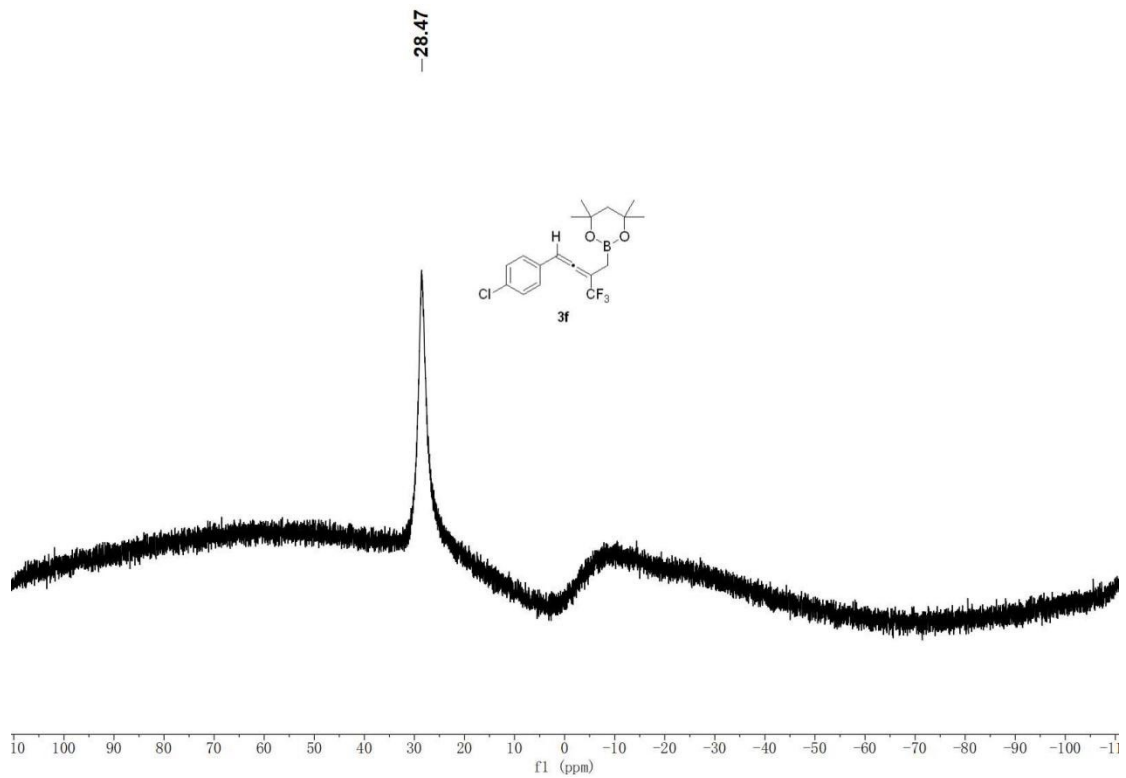
2-(4-(4-chlorophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)-4,4,6,6-tetramethyl-1,3,2-dioxaborinane(3f)

7.39
7.38
7.37
7.37
7.33
7.33
7.32
7.31
6.48
6.47
6.47
6.46

1.83
1.79
1.79
1.77
1.76
1.74
1.73
1.35
1.31



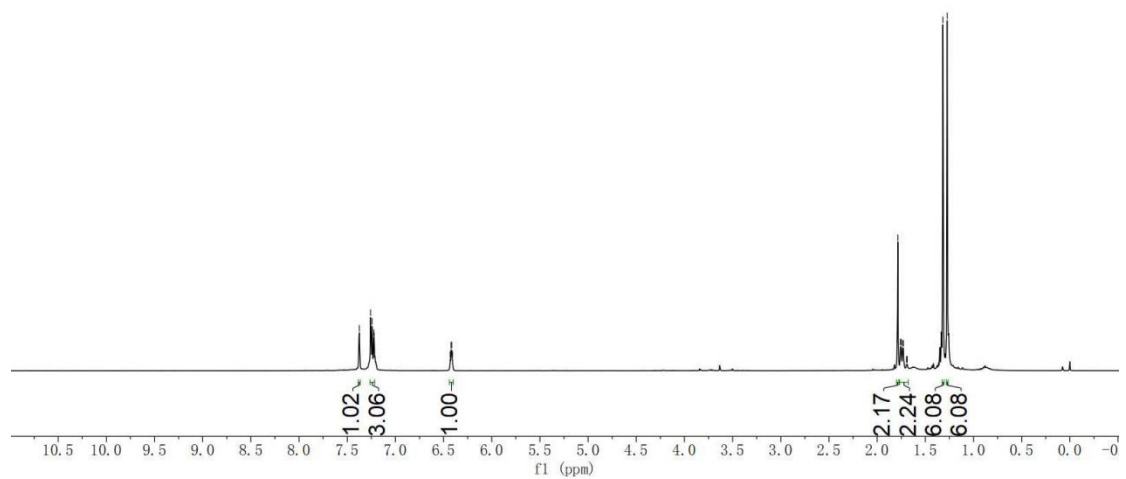
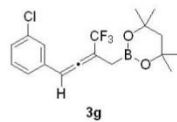


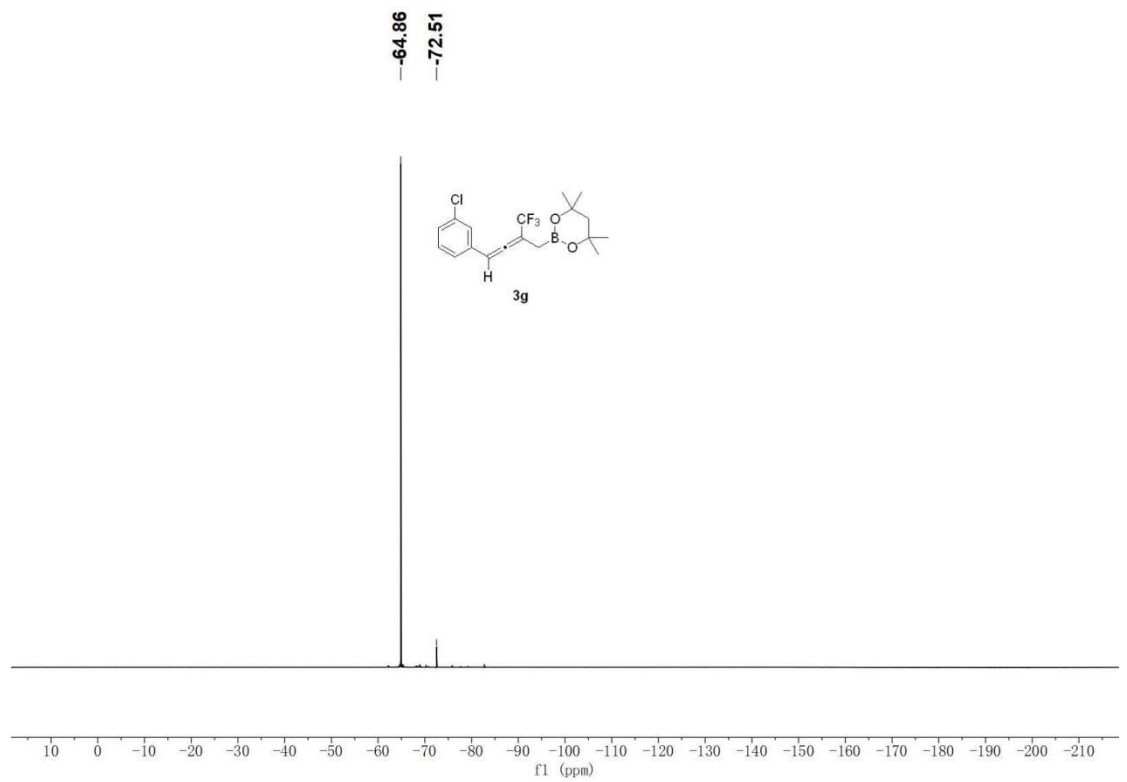
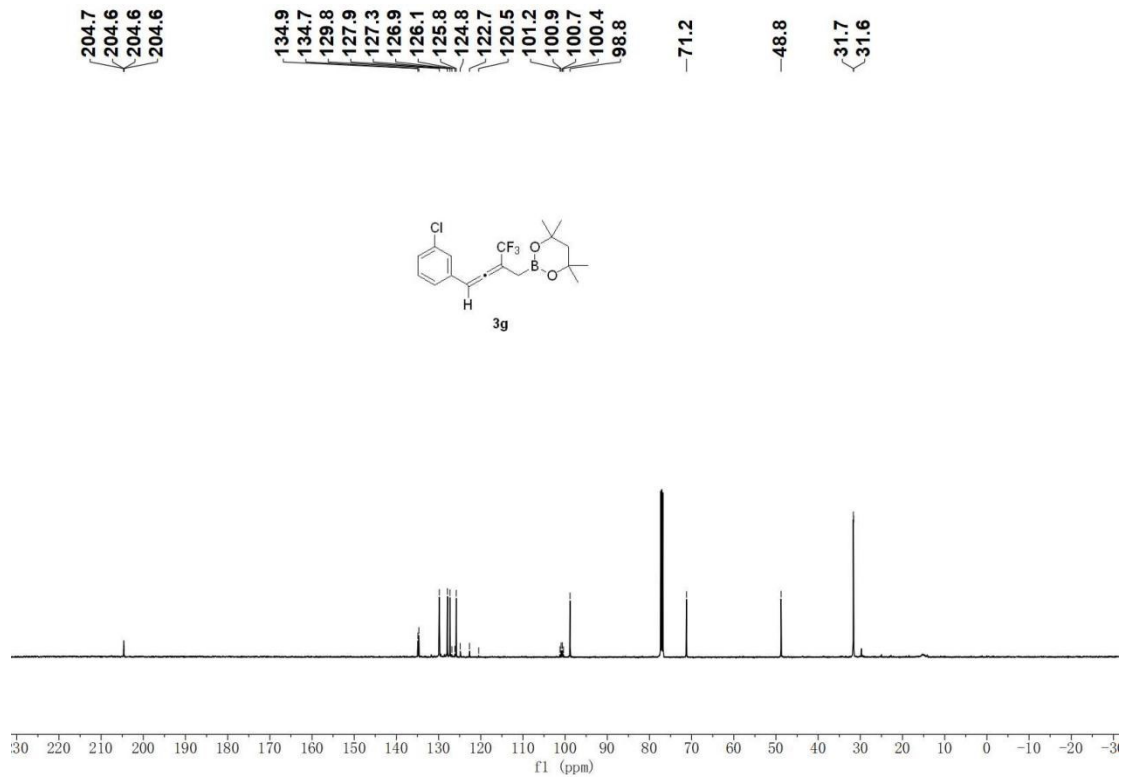


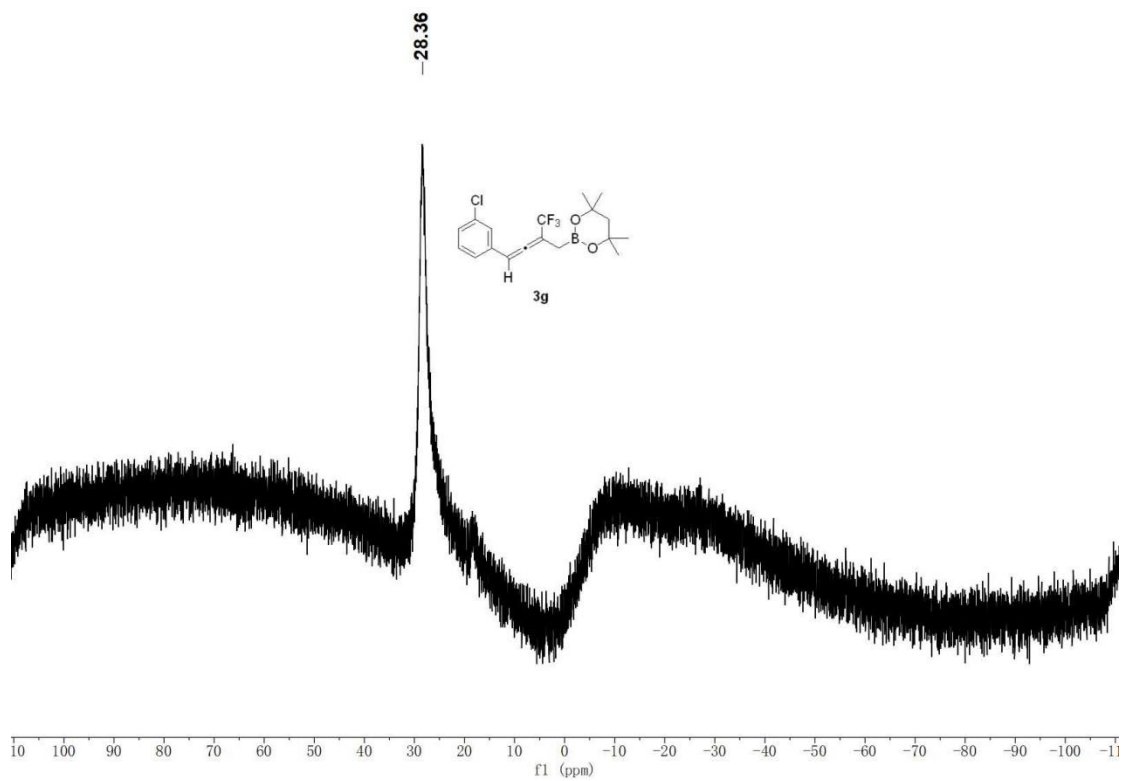
2-(4-(3-chlorophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)-4,4,6,6-tetramethyl-1,3,2-dioxaborinane(3g)

7.37
7.26
7.24
7.23
7.22
6.43
6.42
6.41

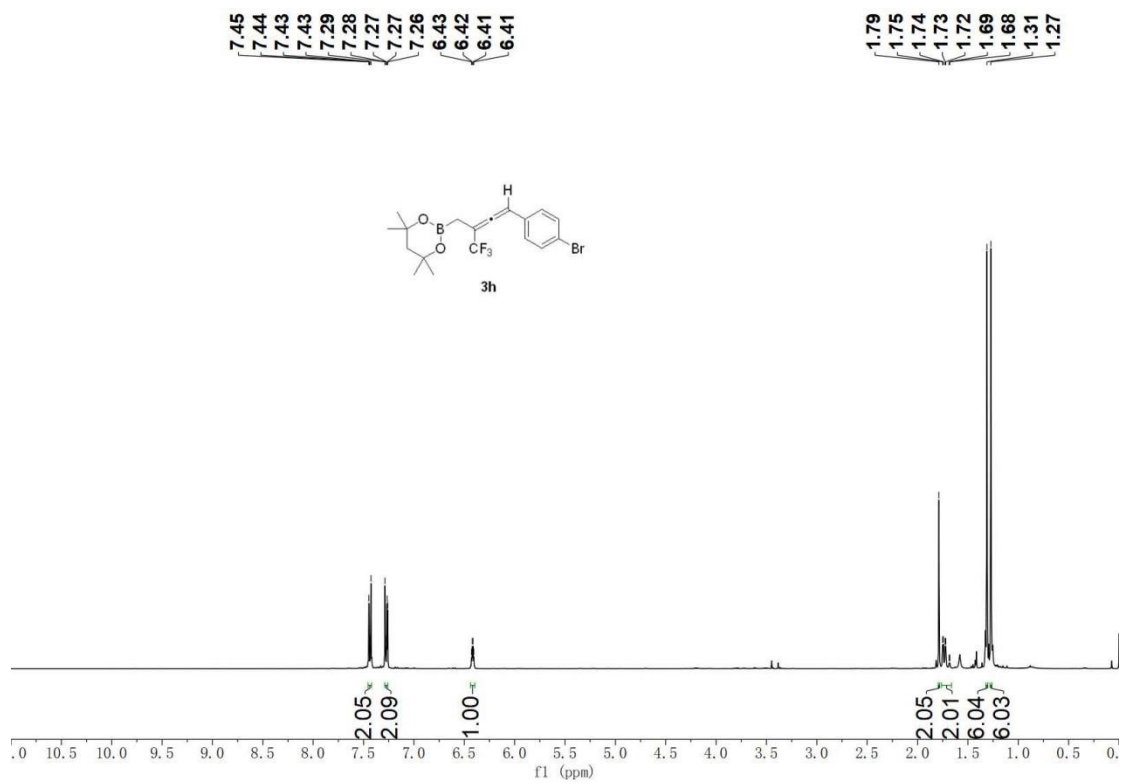
1.79
1.76
1.75
1.74
1.73
1.70
1.69
1.32
1.27

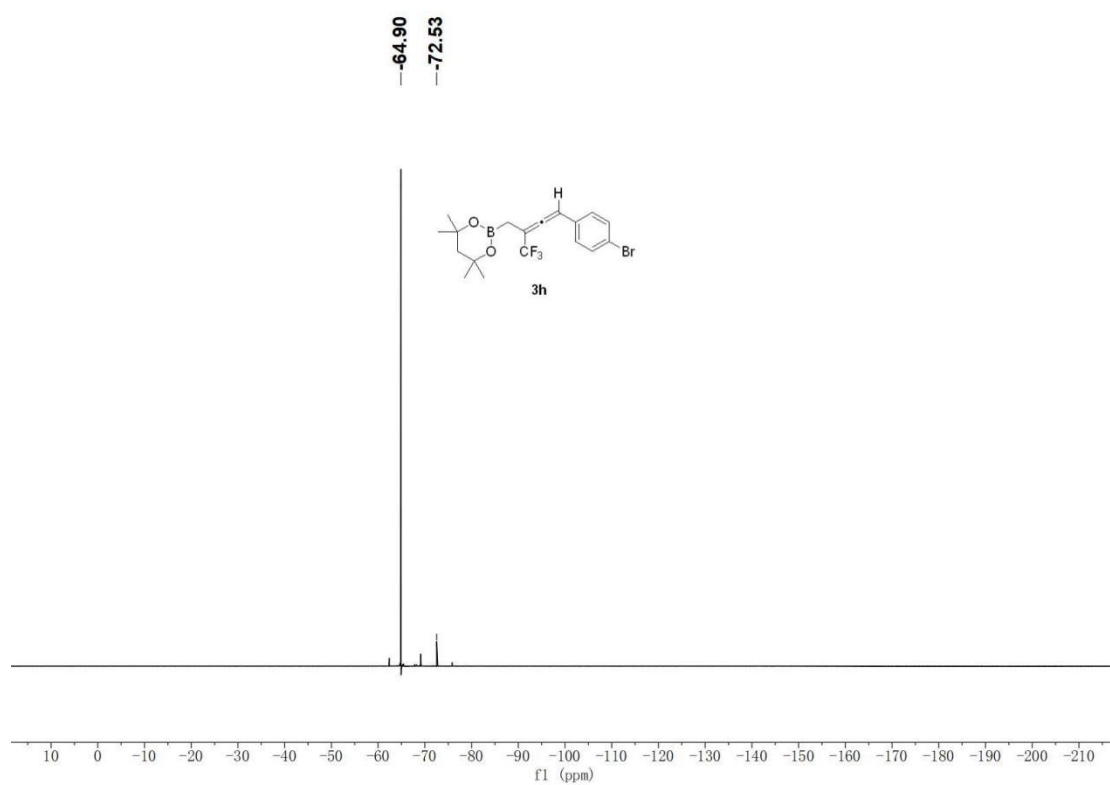
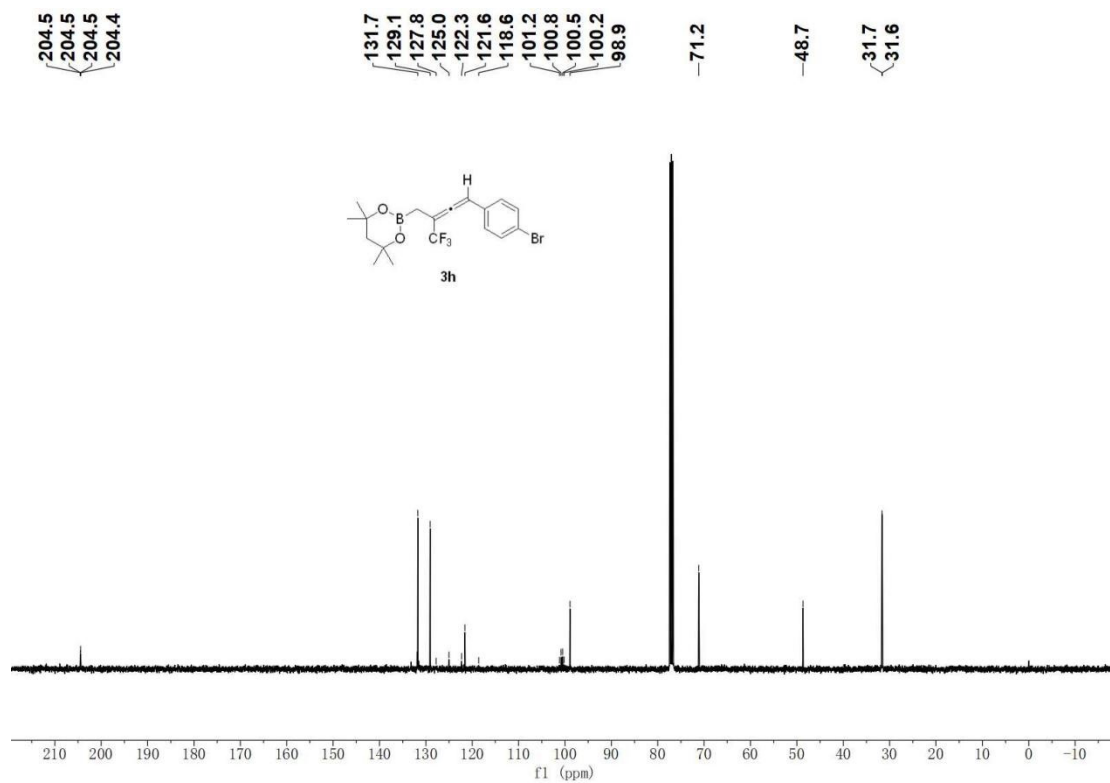


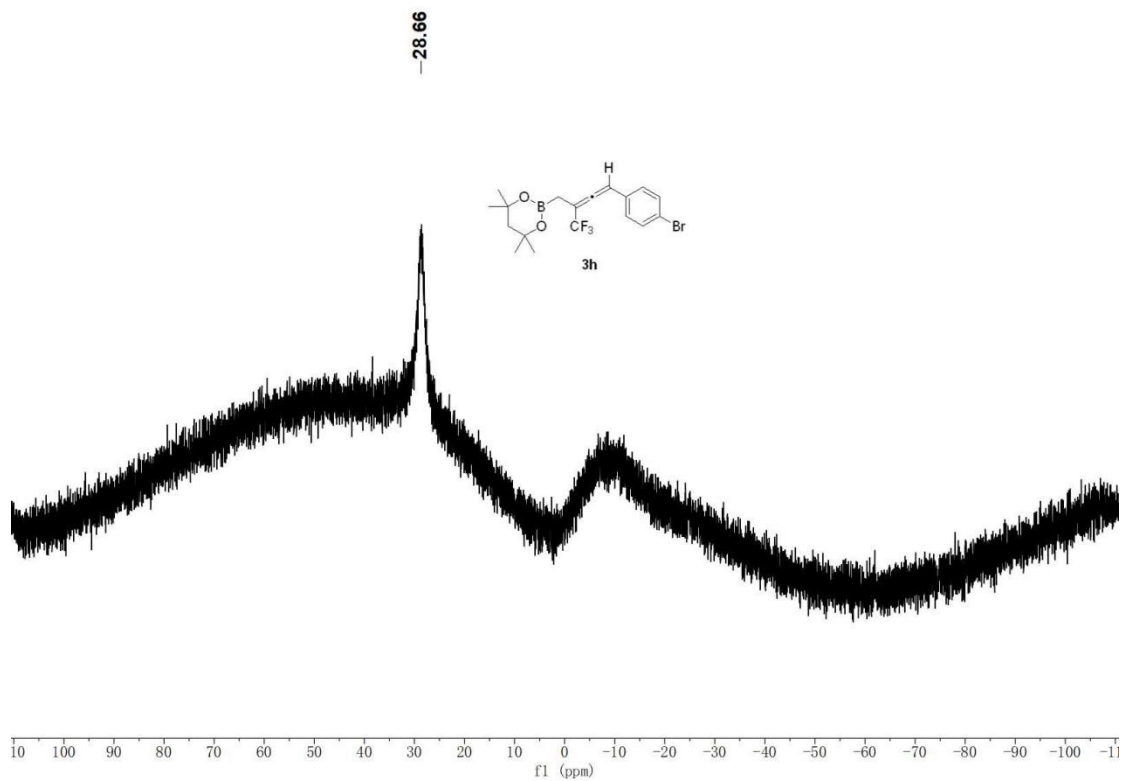




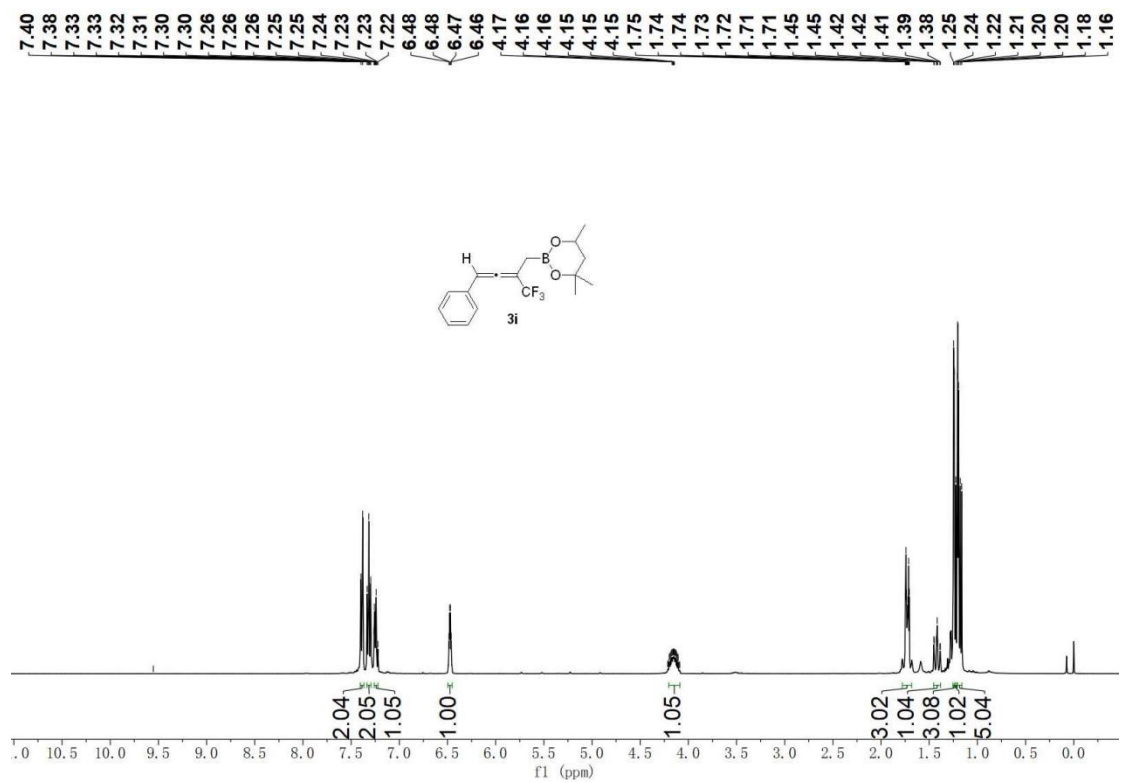
2-(4-(4-bromophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)-4,4,6,6-tetramethyl-1,3,2-dioxaborinane(3h)







4,4,6-trimethyl-2-(4-phenyl-2-(trifluoromethyl)buta-2,3-dien-1-yl)-1,3,2-dioxaborinane (3i)

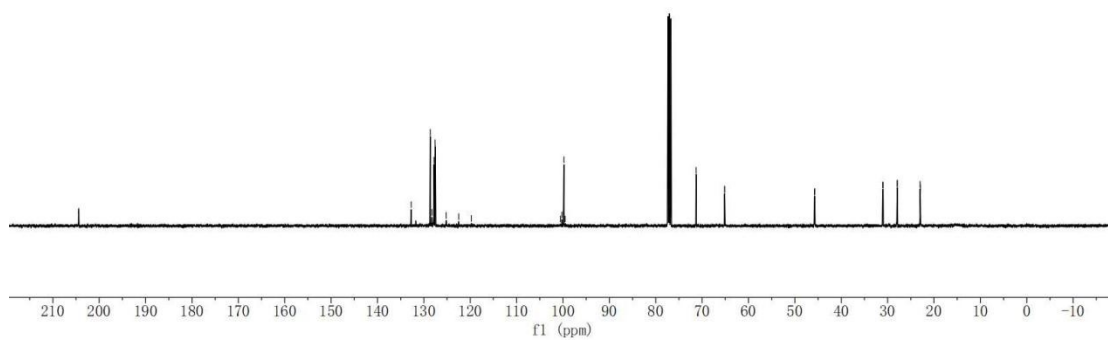
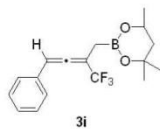


204.4
204.4
204.4

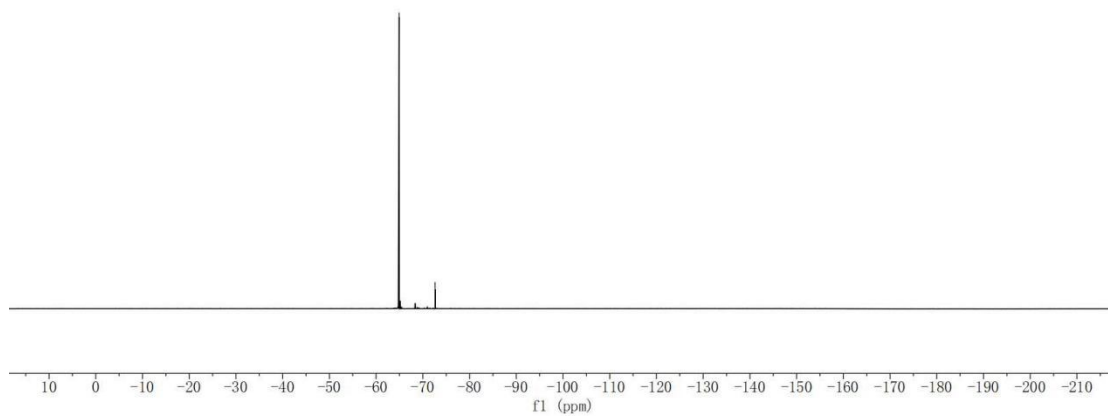
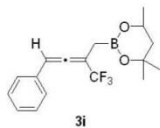
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99.8
99.5

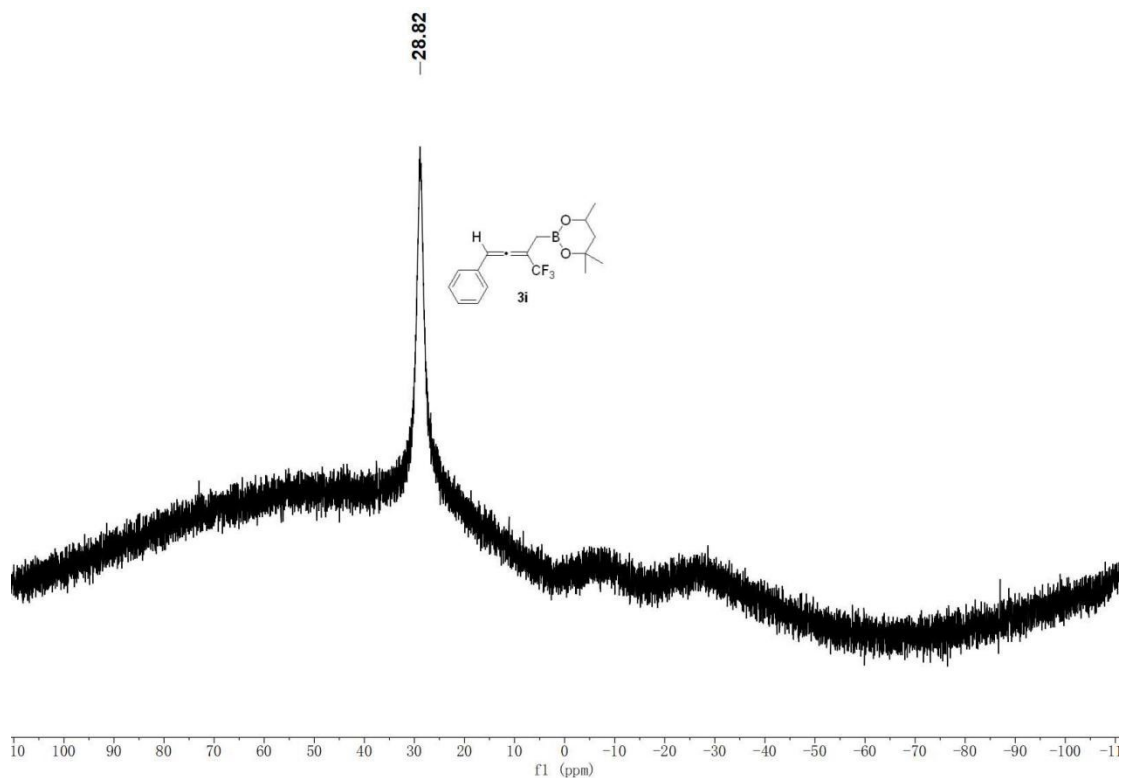
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31.0
28.0
27.9
23.0
22.9



-64.93
-72.62

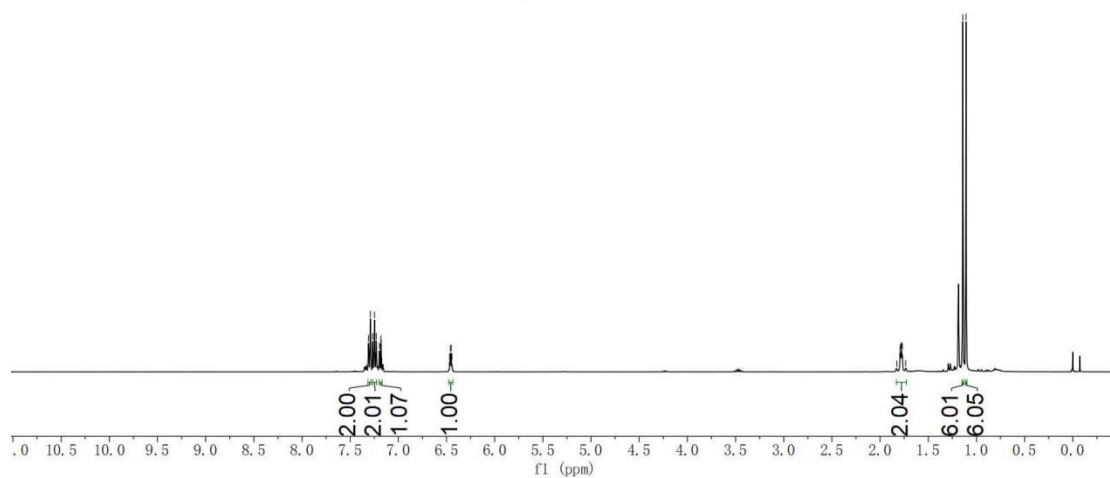
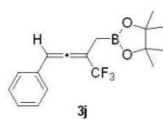


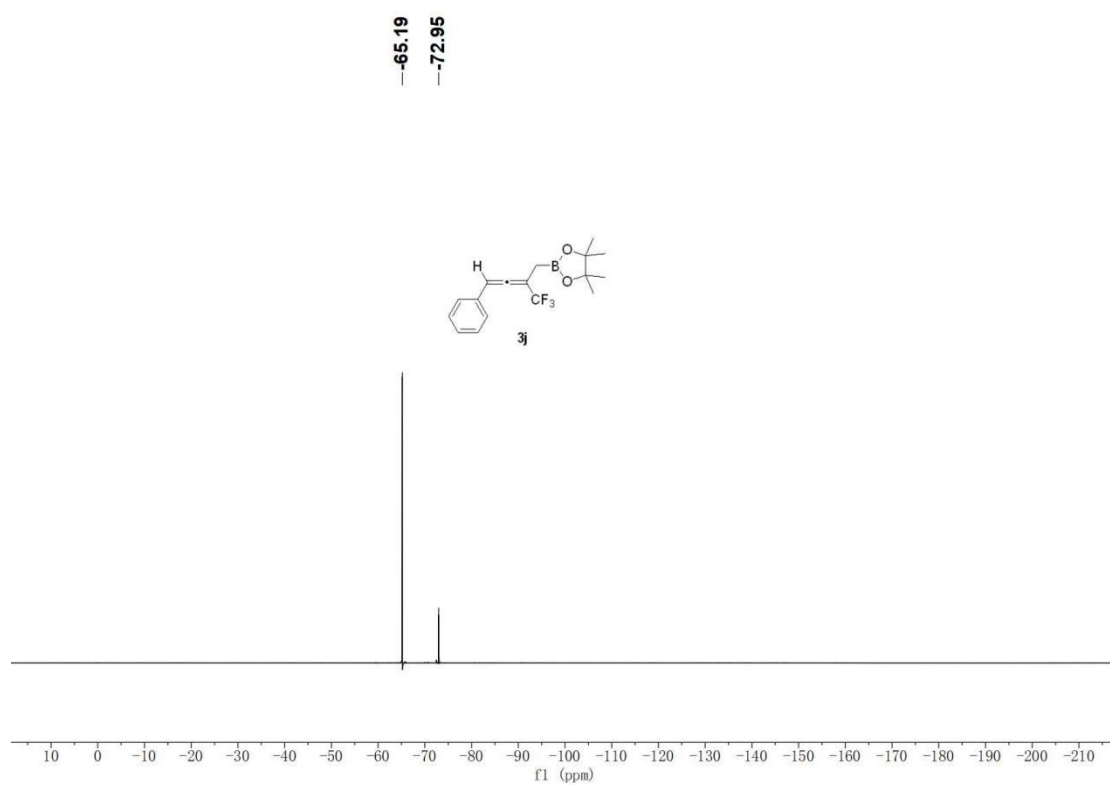
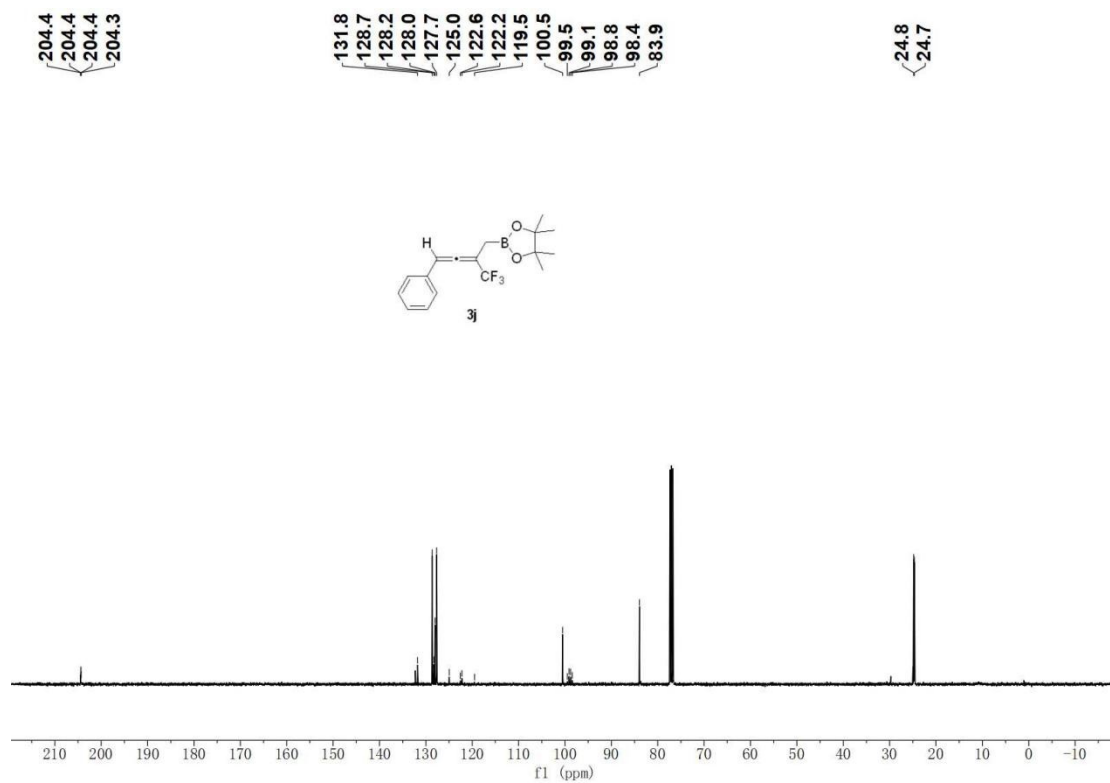


4,4,5,5-tetramethyl-2-(4-phenyl-2-(trifluoromethyl)buta-2,3-dien-1-yl)-1,3,2-dioxaborolane (3j)

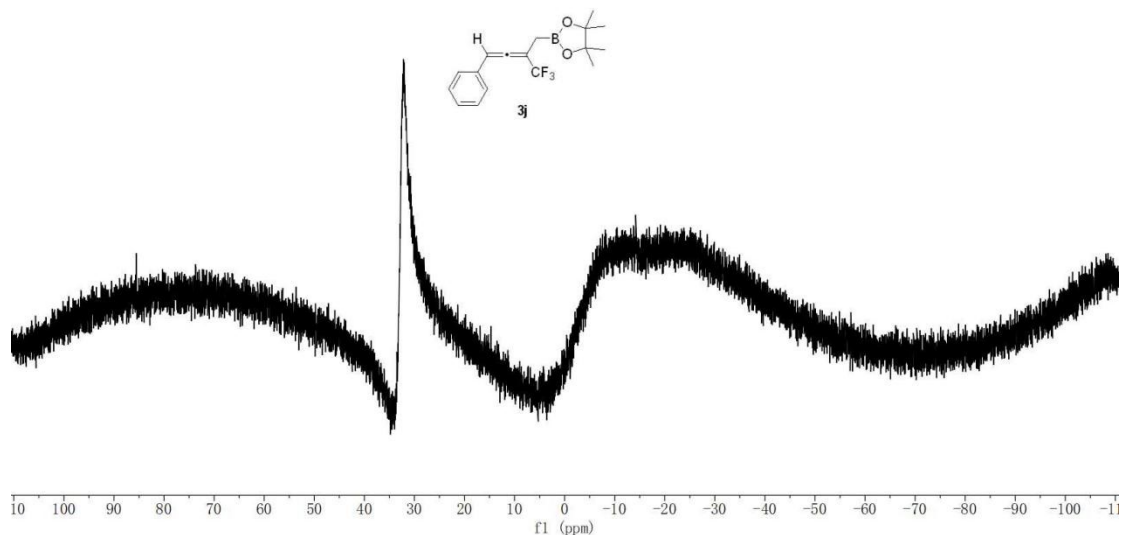
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7.19
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7.18
7.17
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6.44

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1.78
1.78
1.77
1.74
1.14
1.11



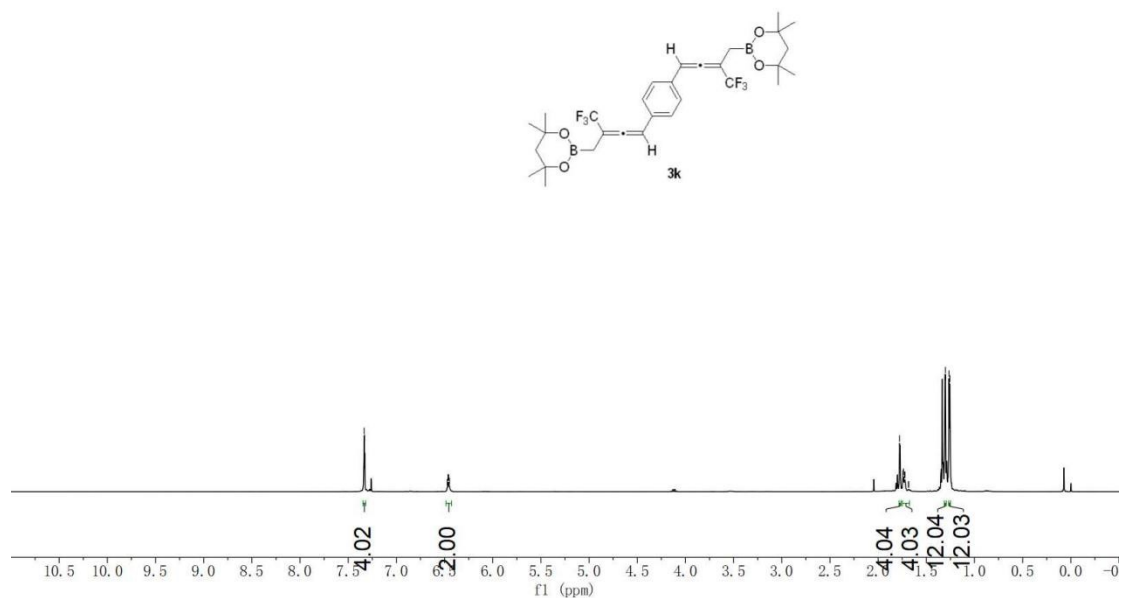


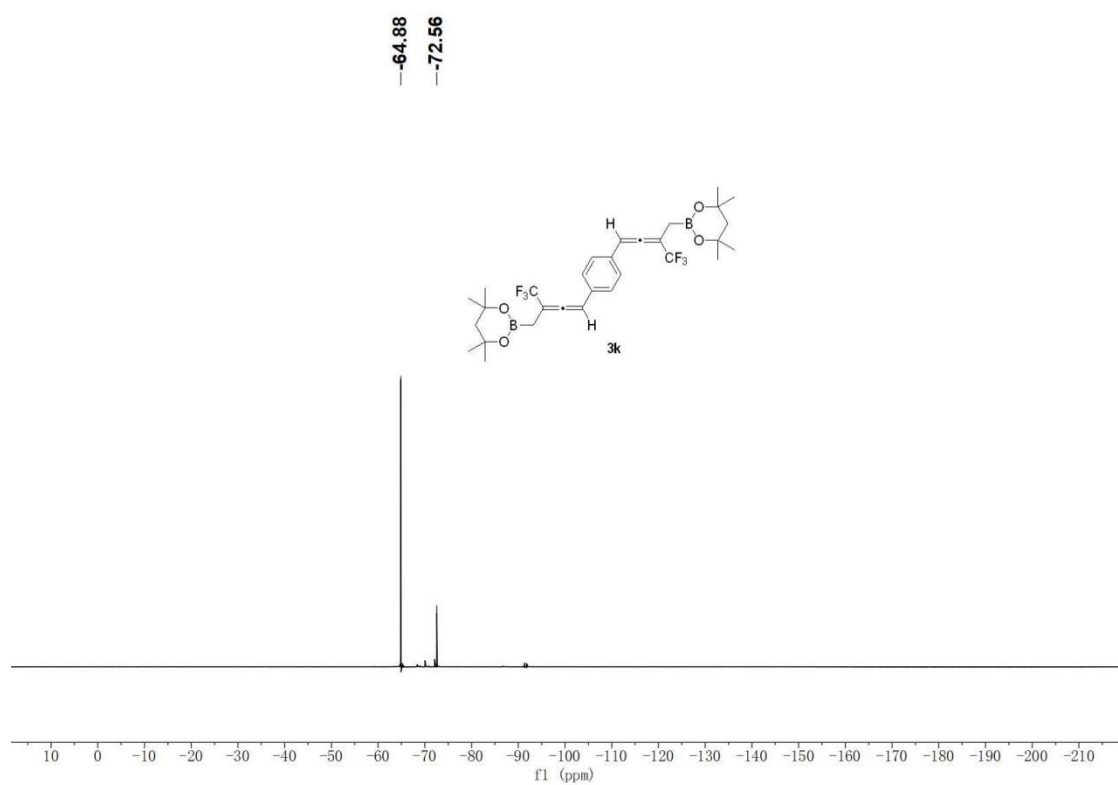
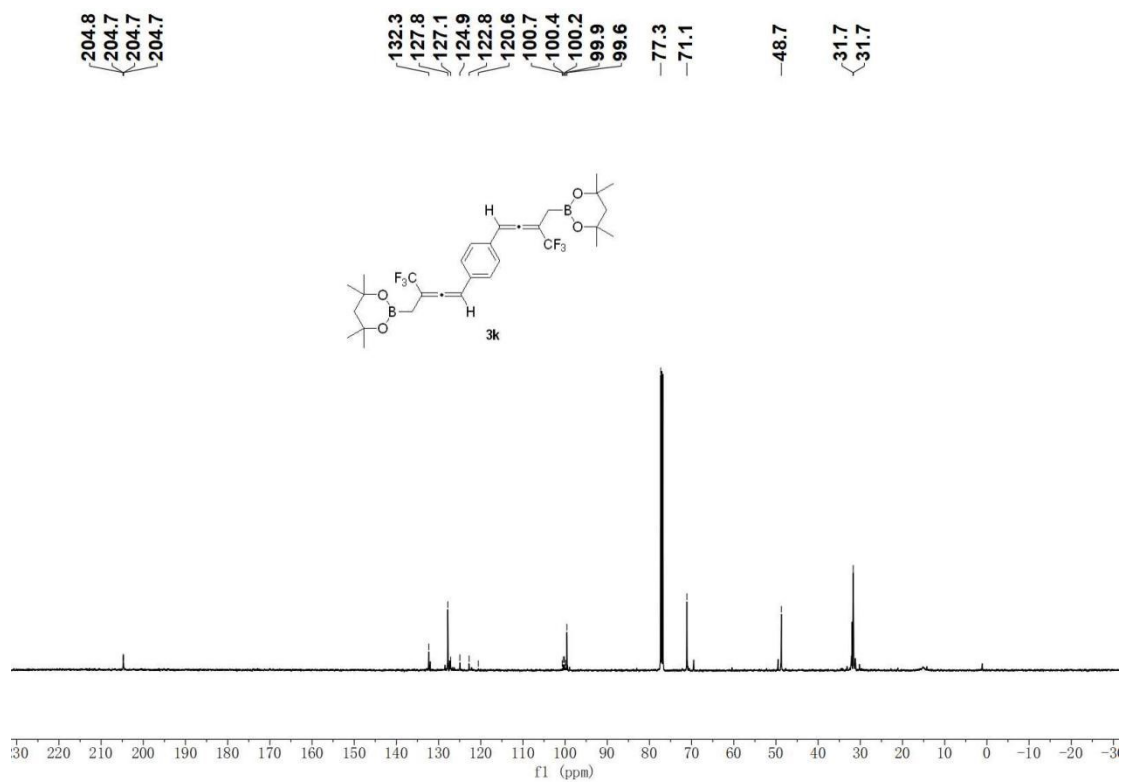
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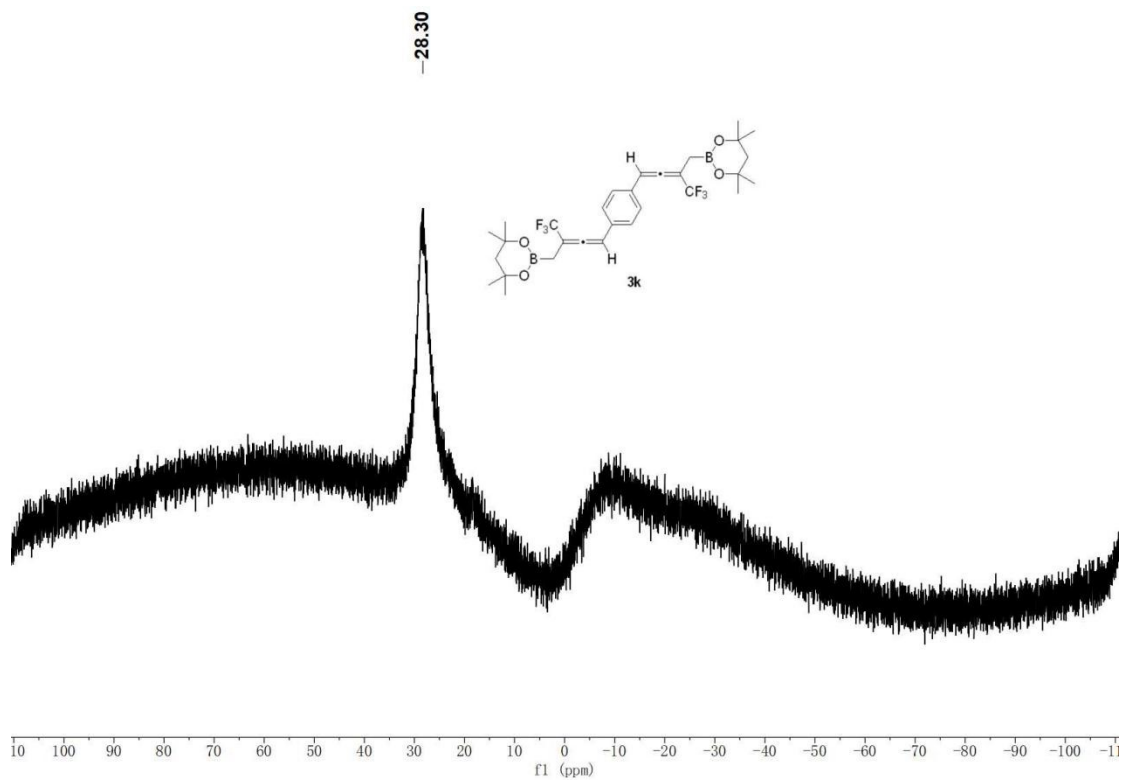


1,4-bis(4,4,4-trifluoro-3-((4,4,6,6-tetramethyl-1,3,2-dioxaborinan-2-yl)methyl)buta-1,2-dien-1-yl)benzene(3k)

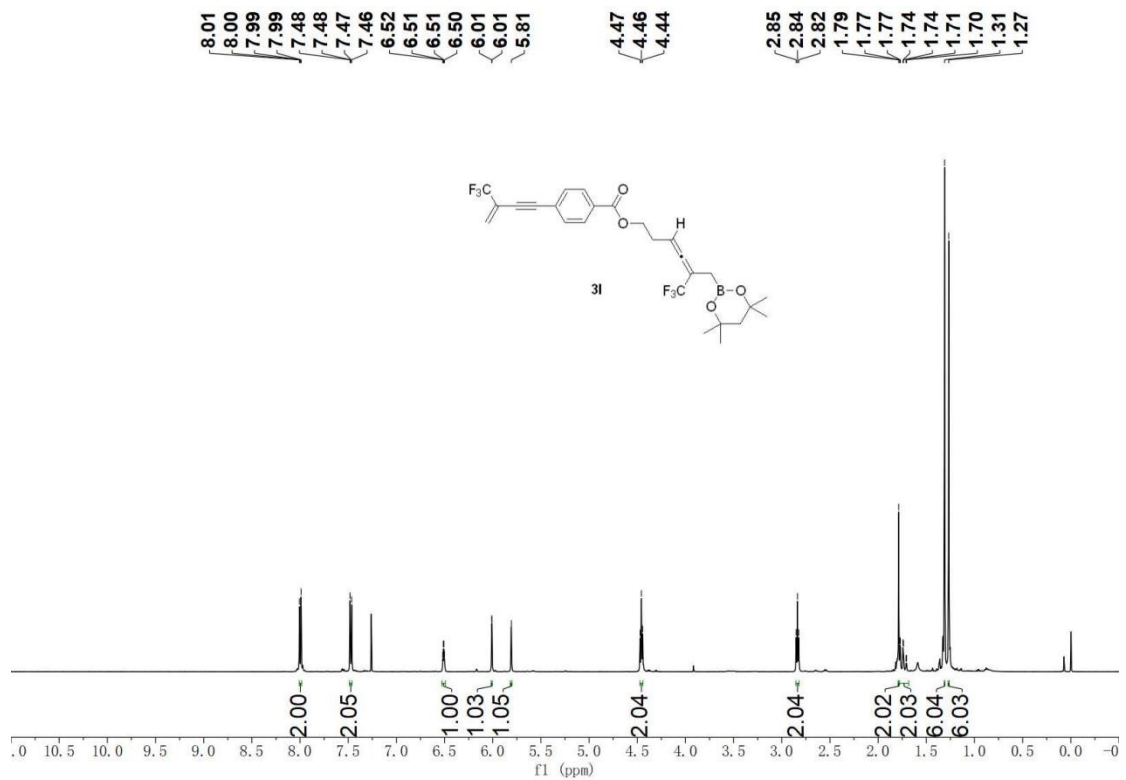
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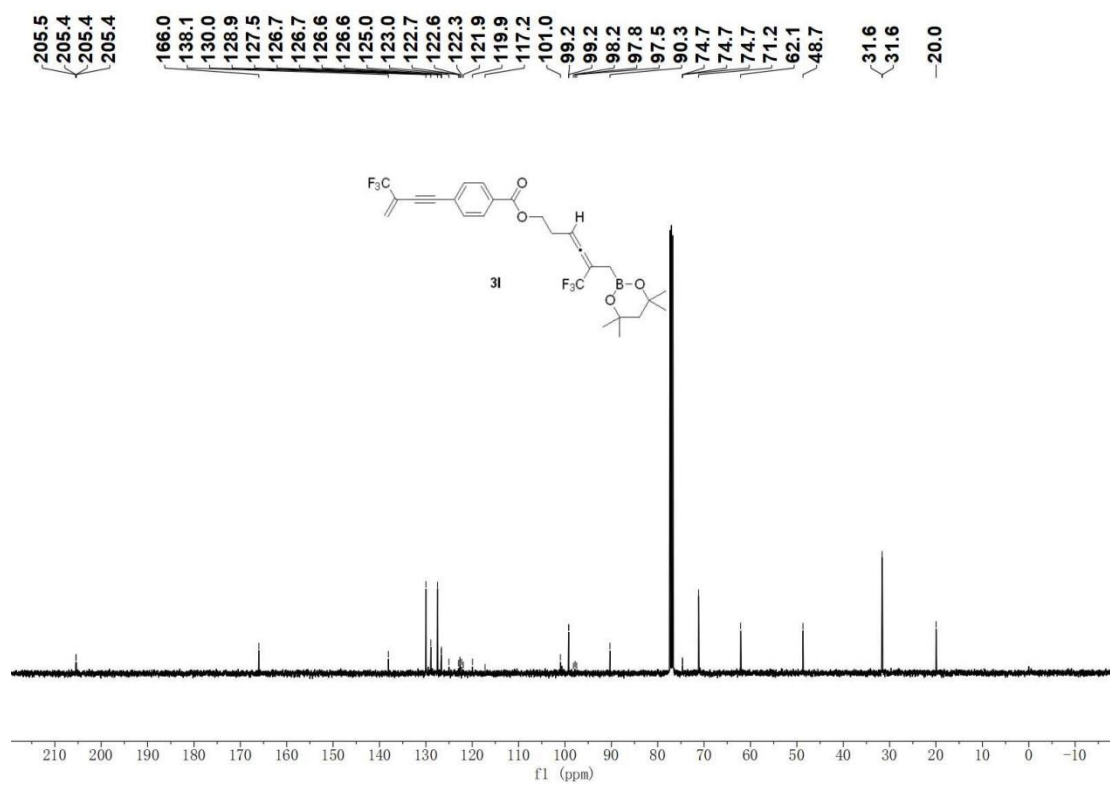




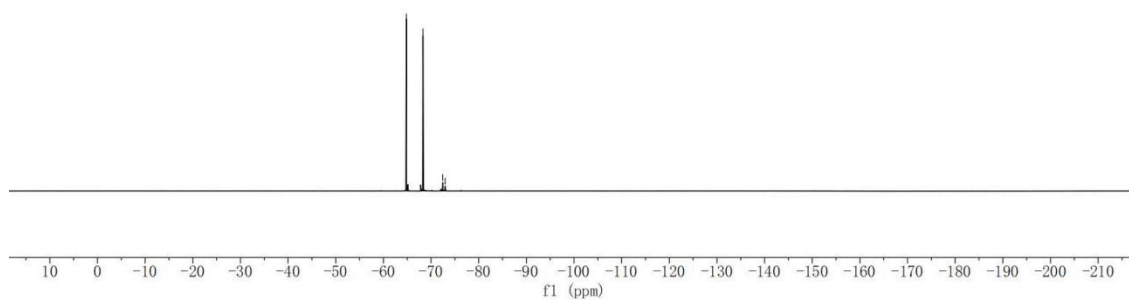
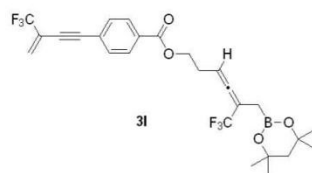


6,6,6-trifluoro-5-((4,4,6,6-tetramethyl-1,3,2-dioxaborinan-2-yl)methyl)hexa-3,4-dien-1-yl 4-(3-(trifluoromethyl)but-3-en-1-yn-1-yl)benzoate(3l)

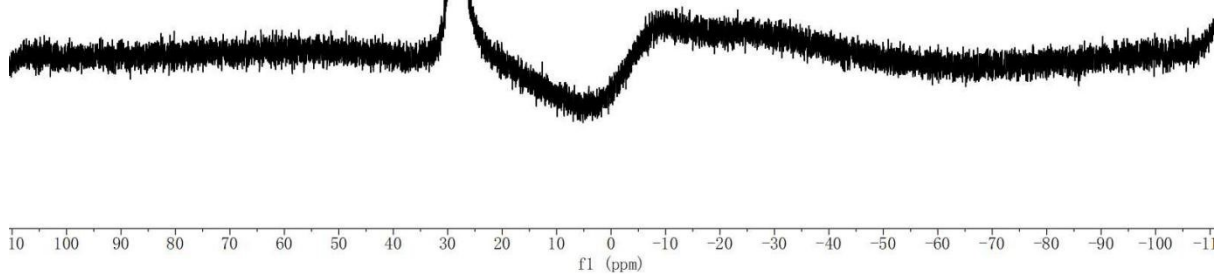
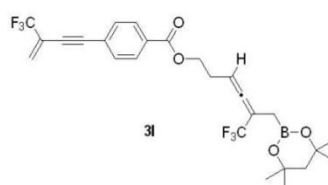




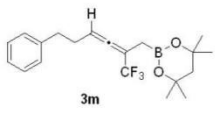
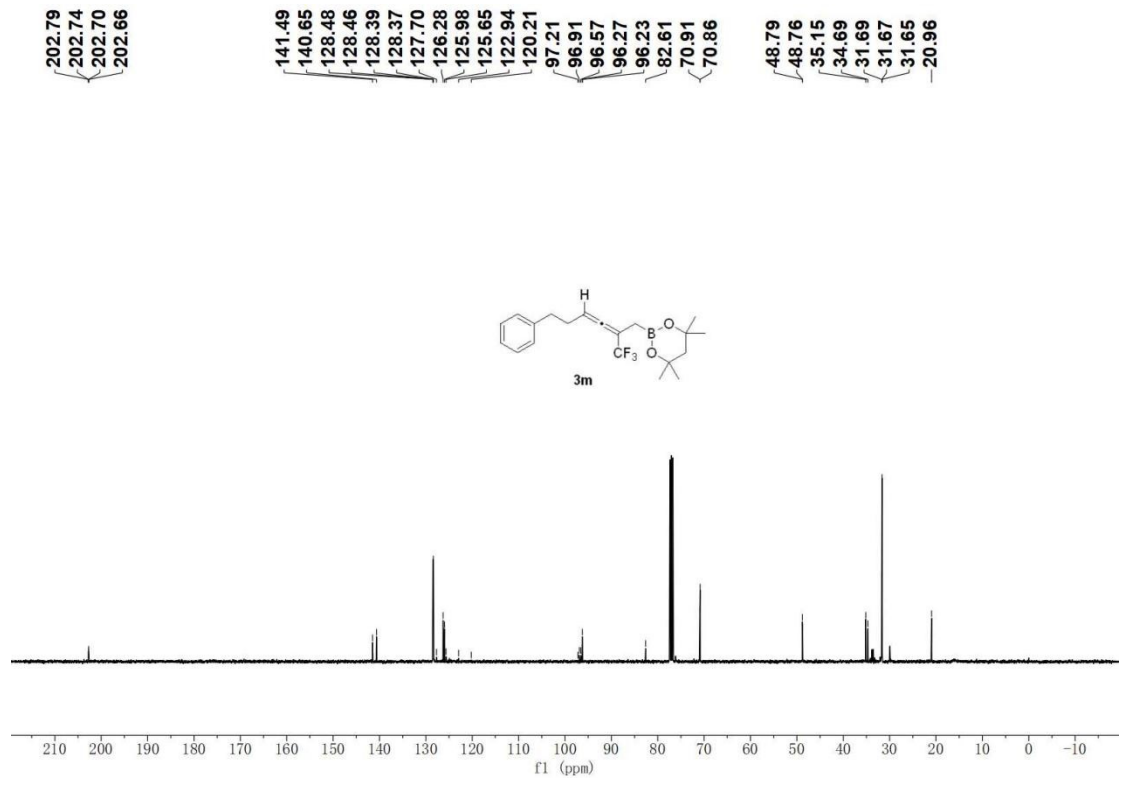
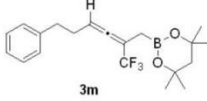
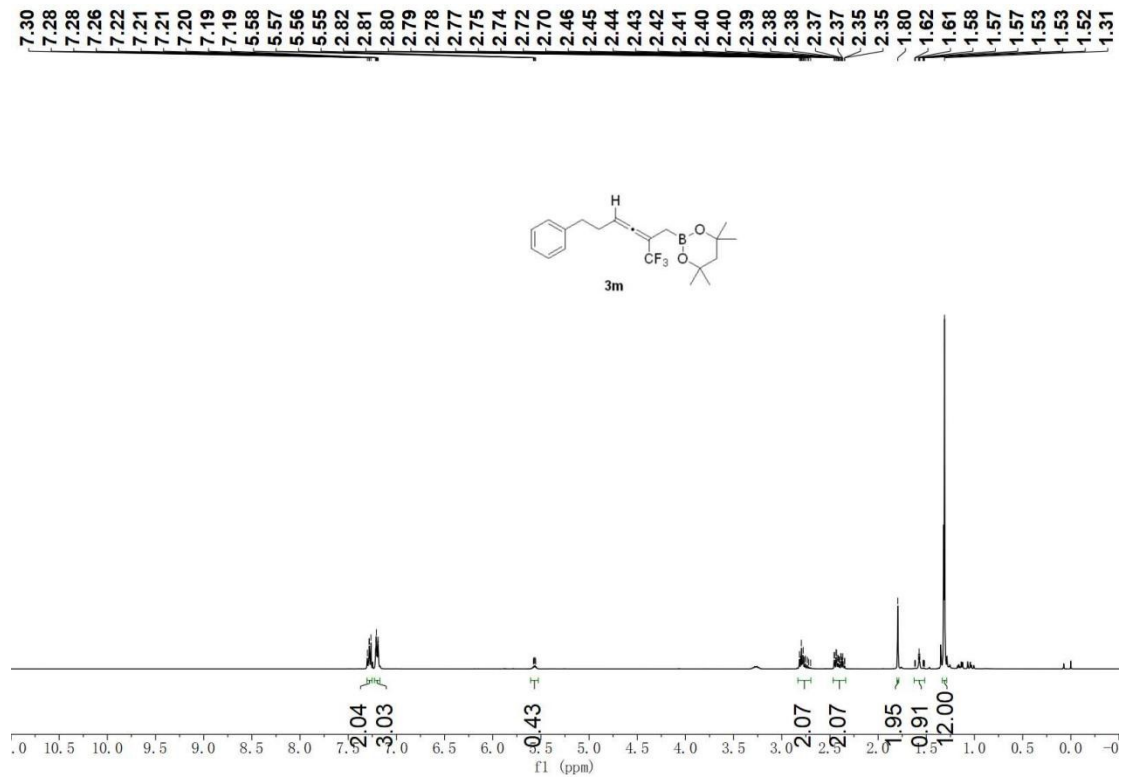
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-72.45
-72.96



-28.48

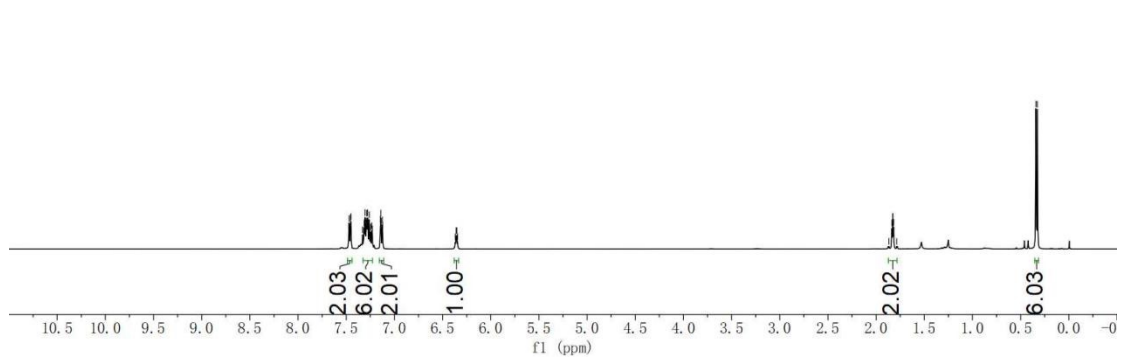
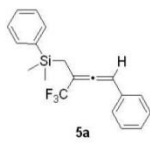


4,4,6,6-tetramethyl-2-(6-phenyl-2-(trifluoromethyl)hexa-2,3-dien-1-yl)-1,3,2-dioxaborinane(3m)



7.47
7.47
7.46
7.45
7.31
7.30
7.29
7.29
7.28
7.27
7.27
7.26
7.24
7.15
7.14
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1.83
1.82
1.79
0.34
0.33

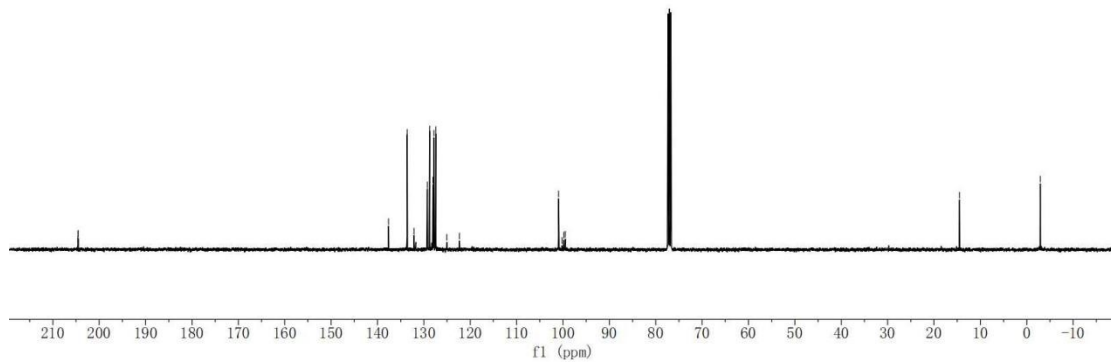
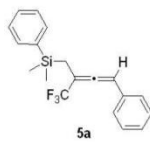


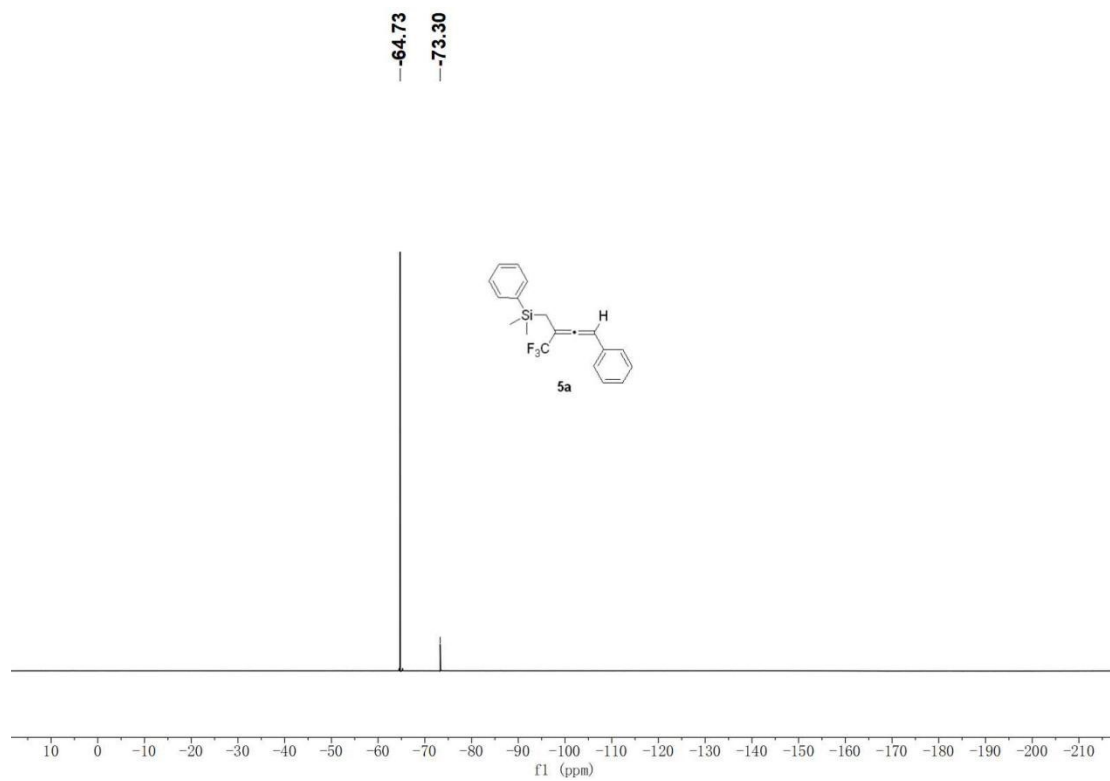
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204.6
204.5
204.5

137.6
133.6
132.1
129.3
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128.0
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127.4
125.1
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101.0
100.2
99.8
99.5

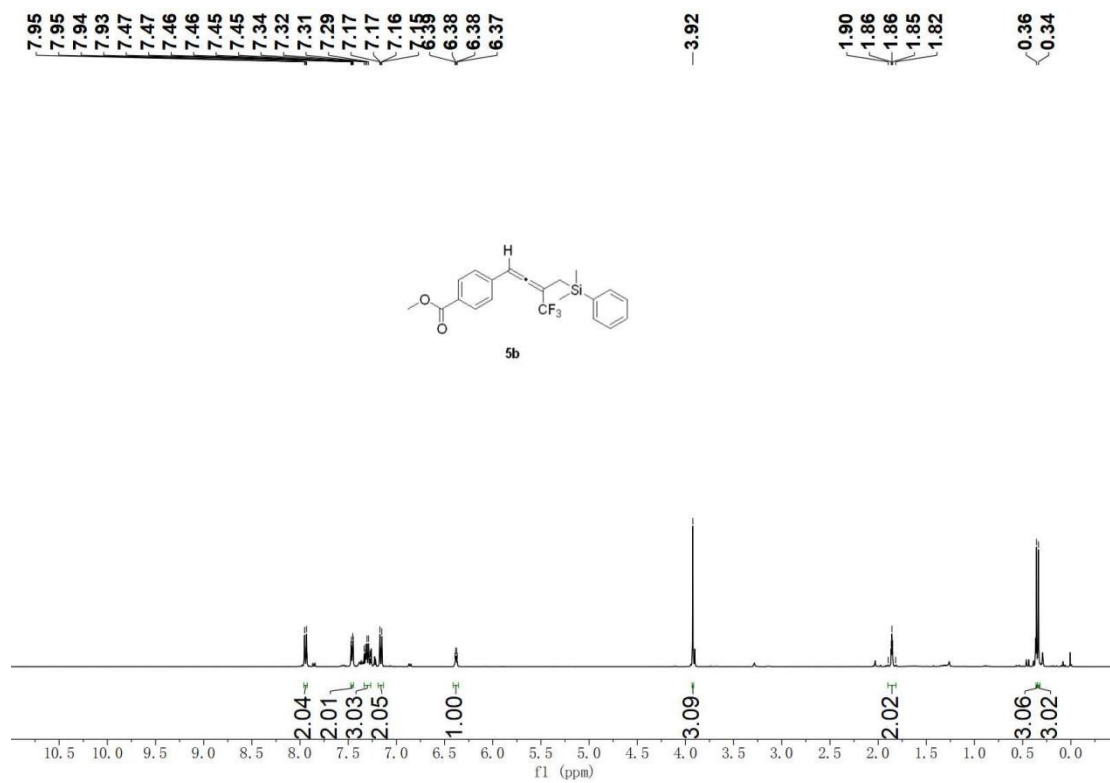
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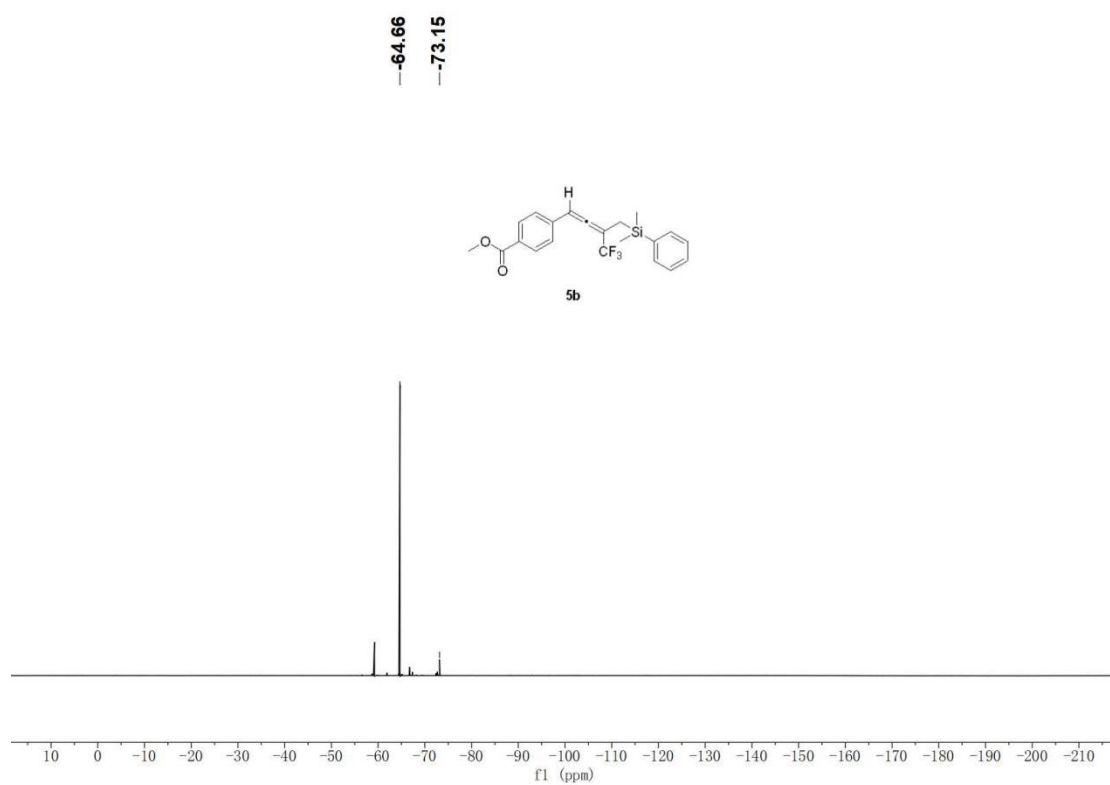
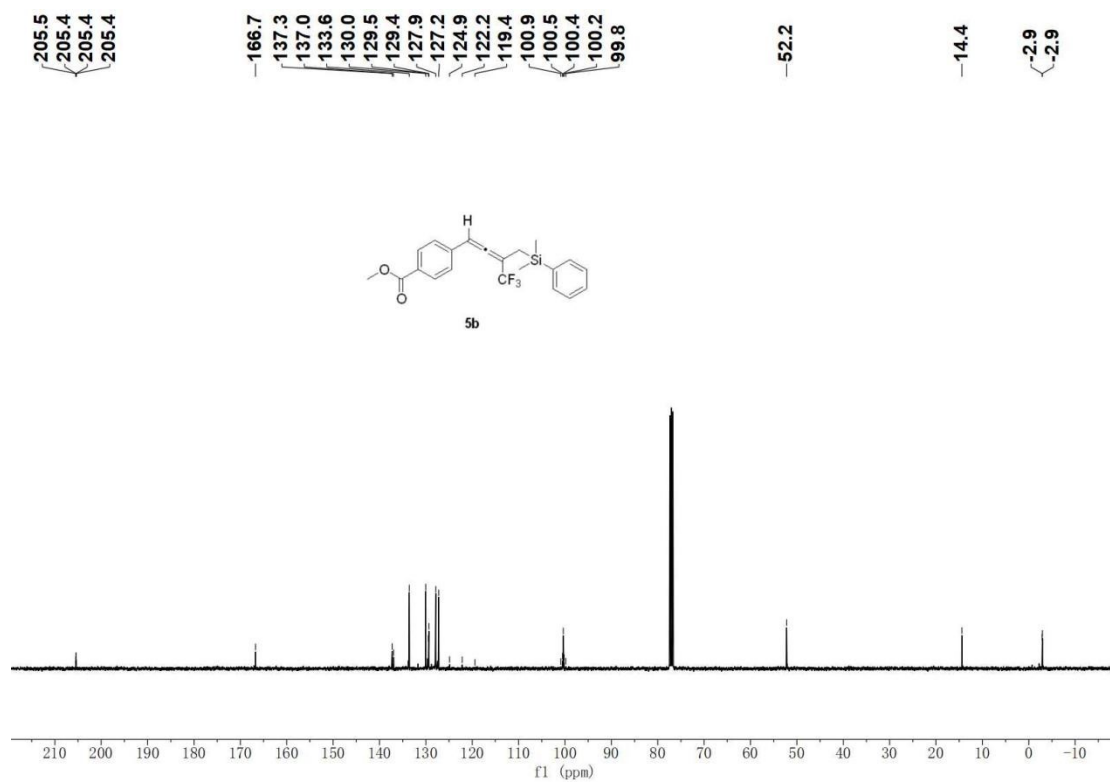
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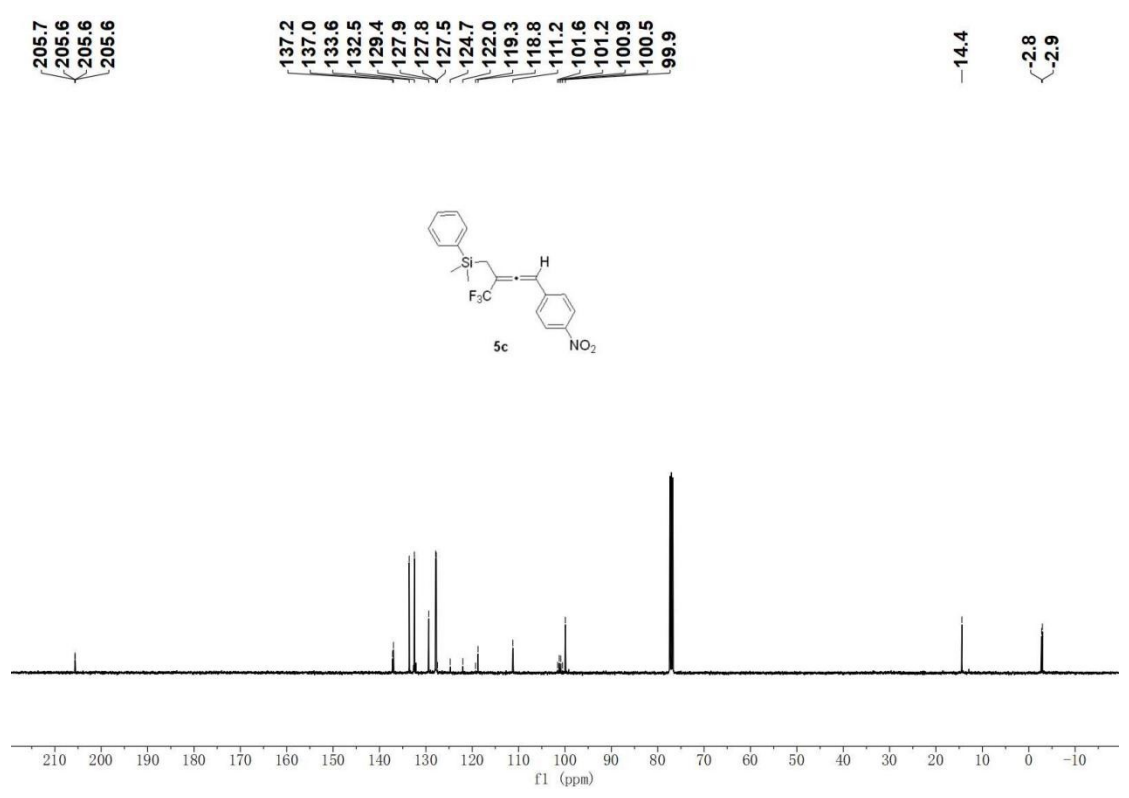
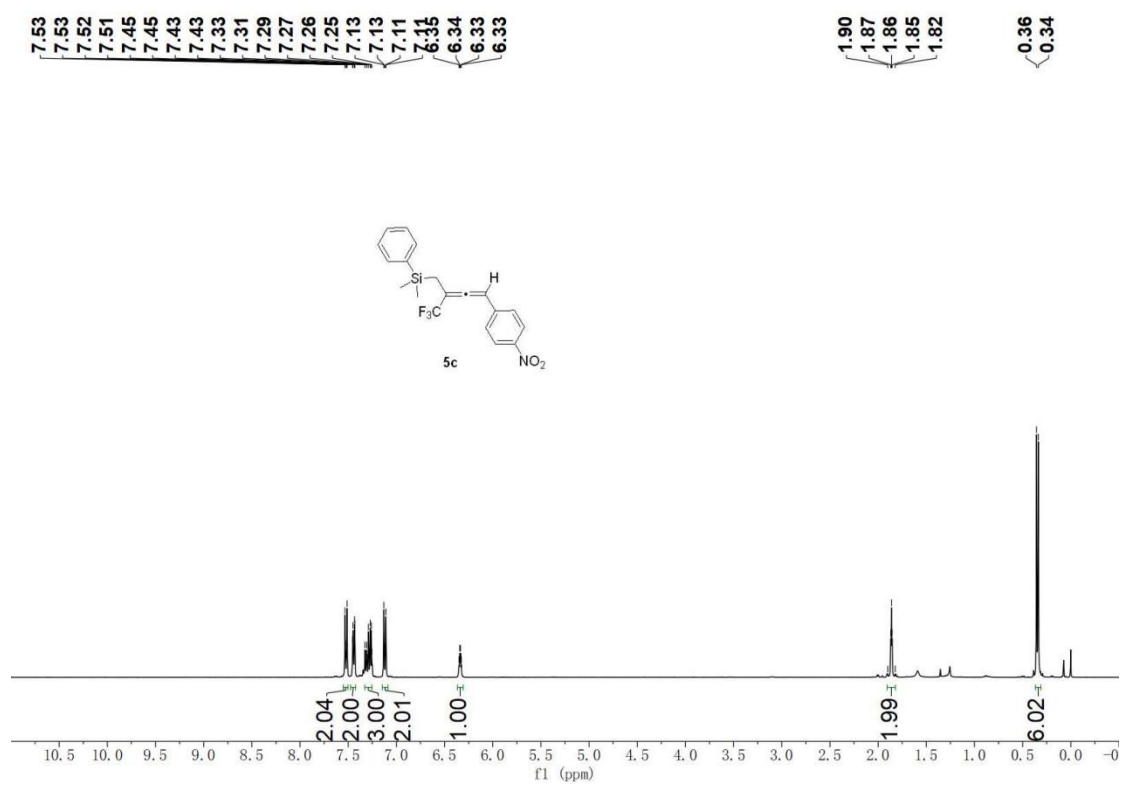


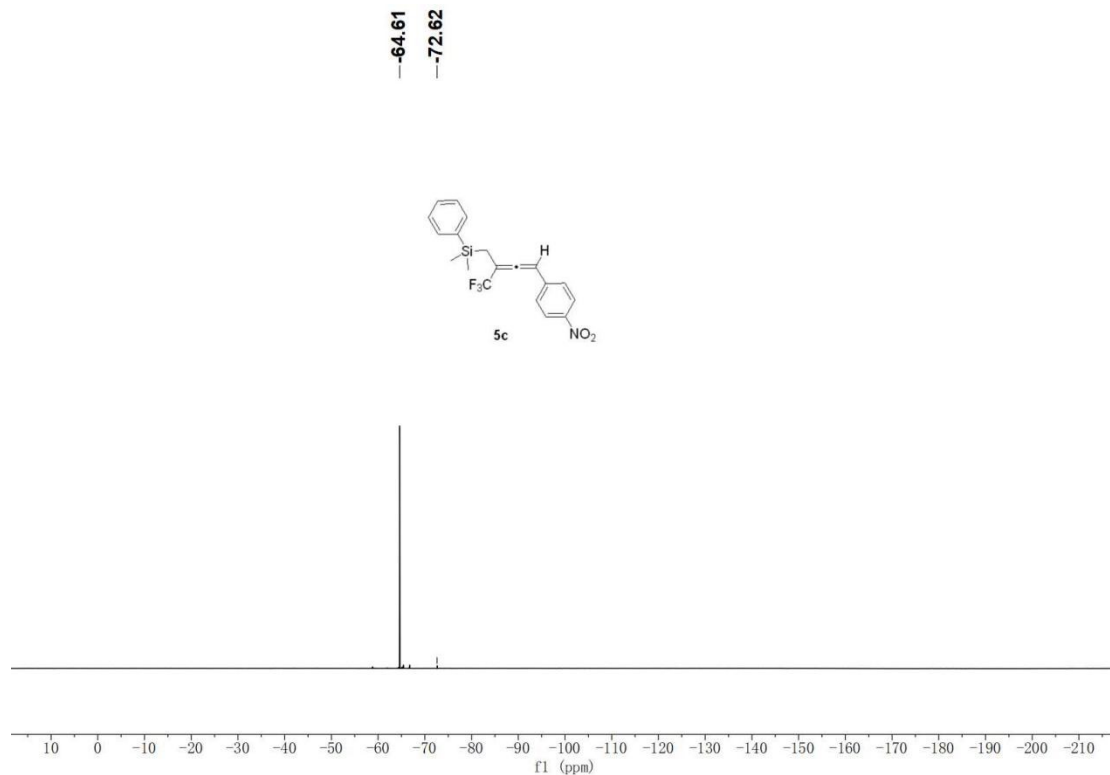
Methyl 4-(3-((dimethyl(phenyl)silyl)methyl)-4,4,4-trifluorobuta-1,2-dien-1-yl)benzoate(5b)



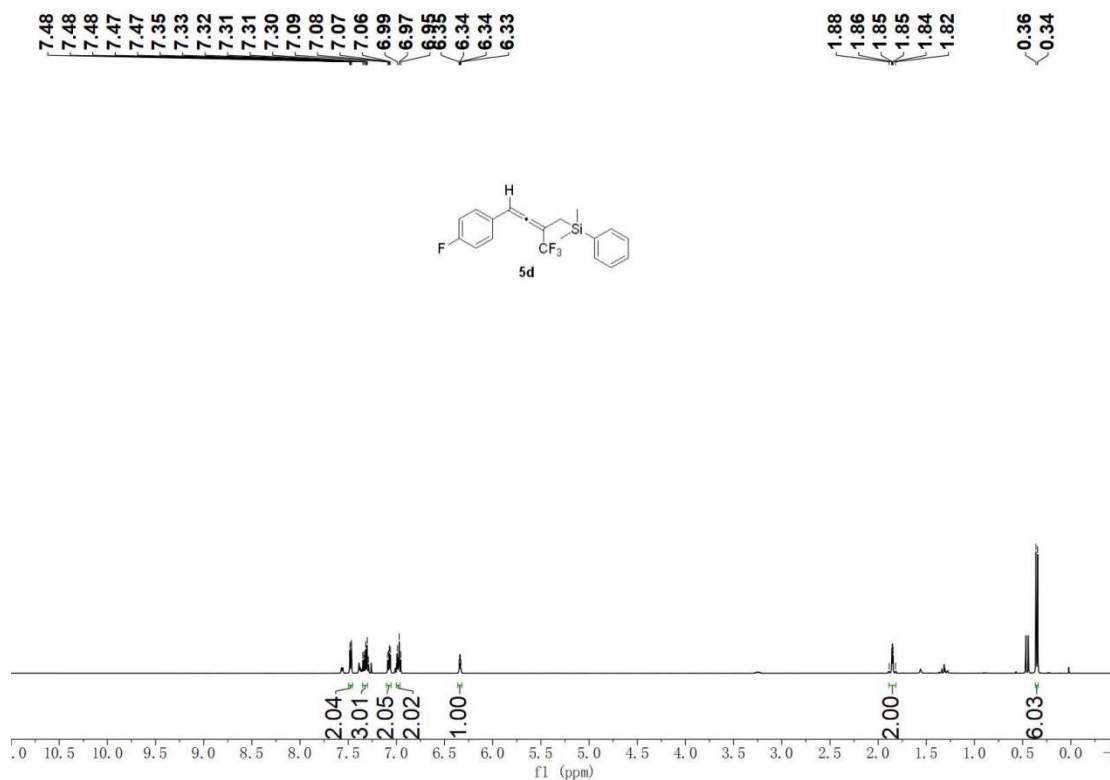


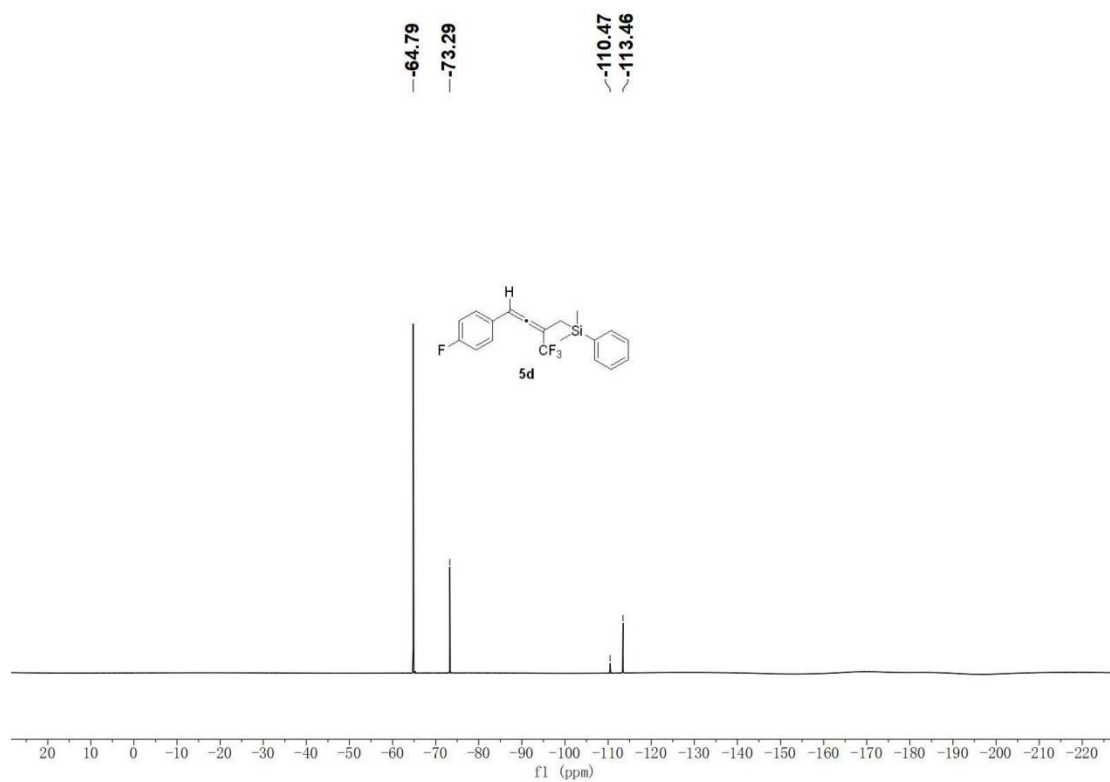
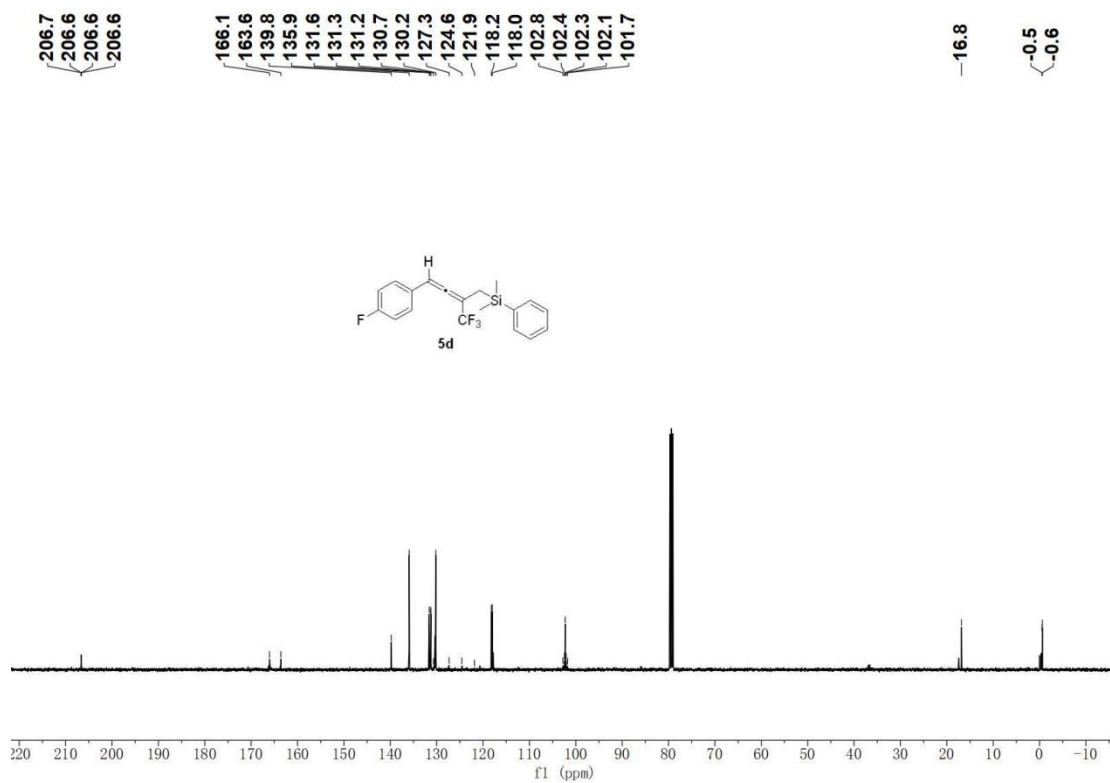
Dimethyl(4-(4-nitrophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)(phenyl)silane (5c)



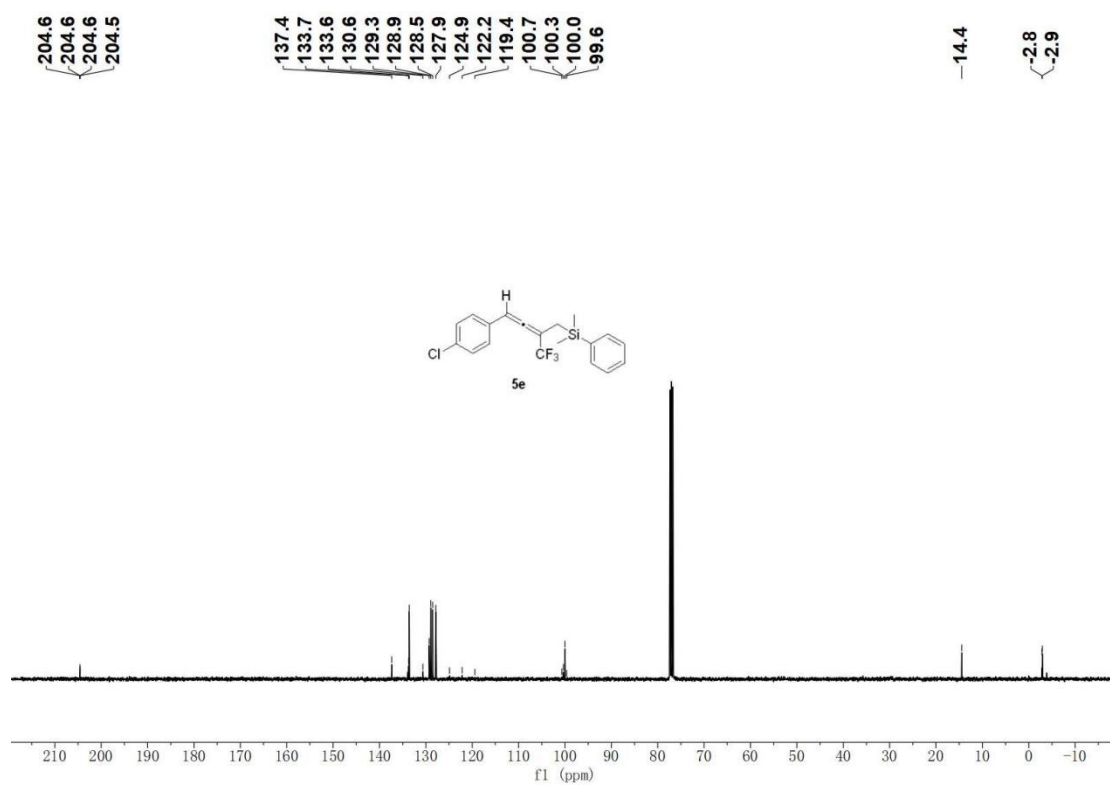
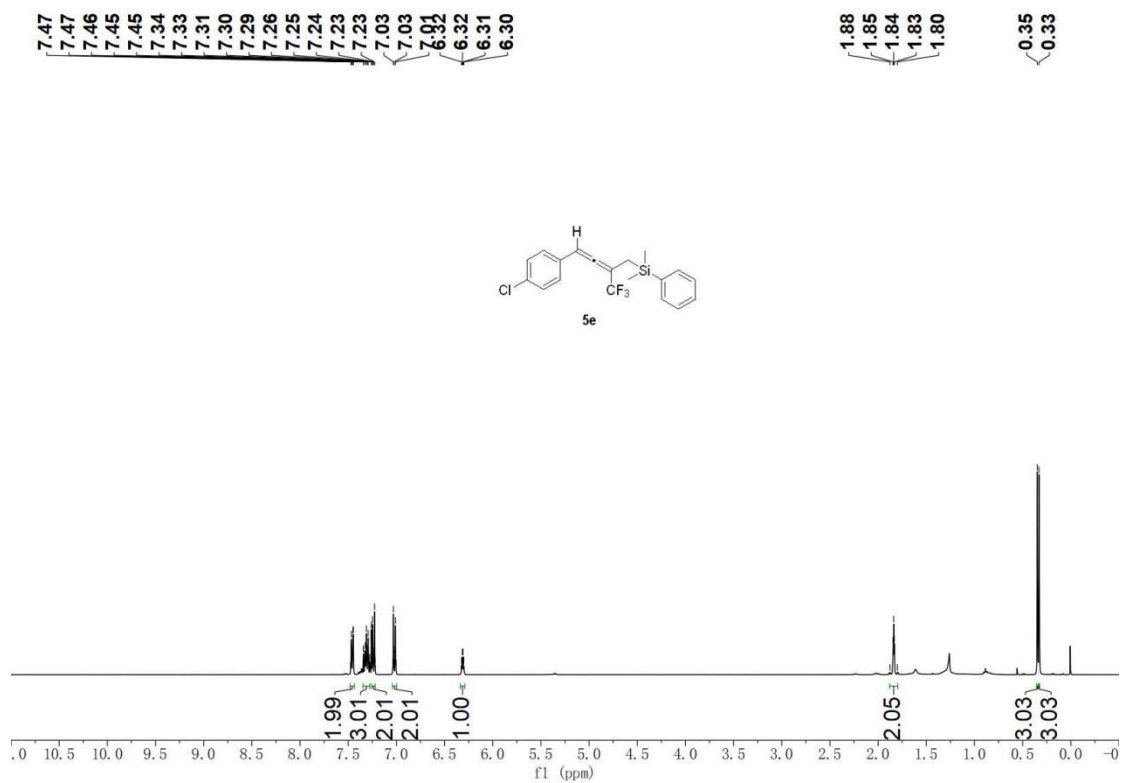


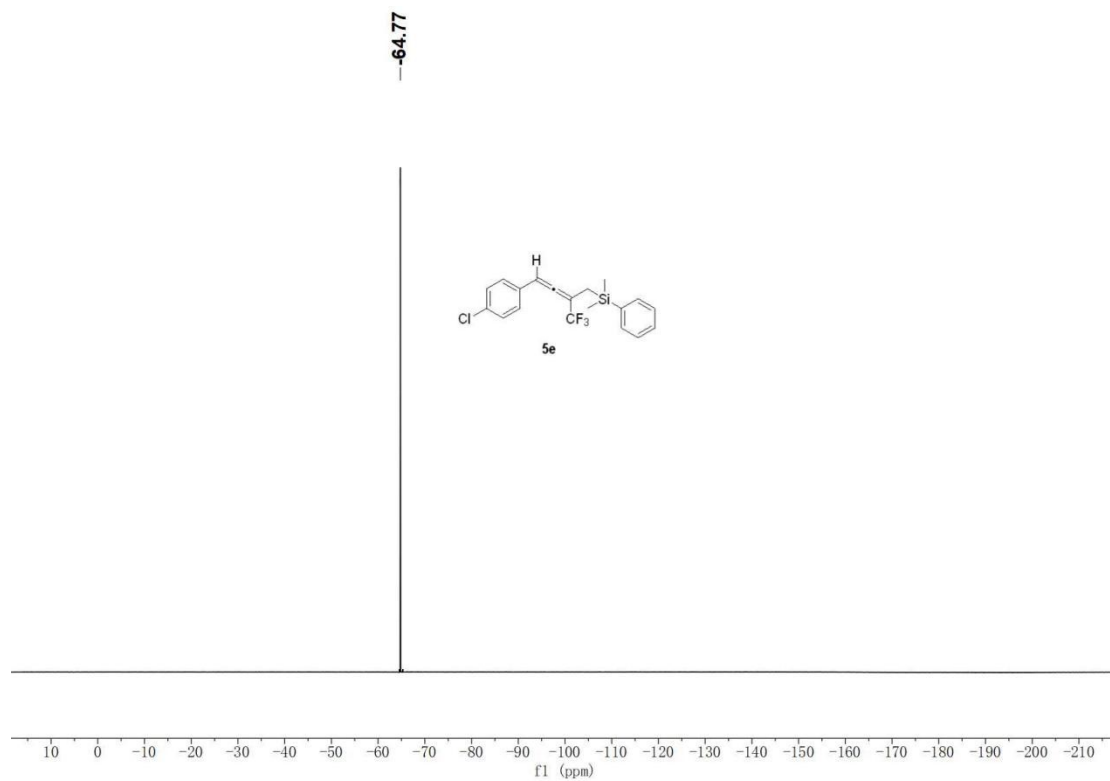
(4-(4-fluorophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)dimethyl(phenyl)silane(5d)



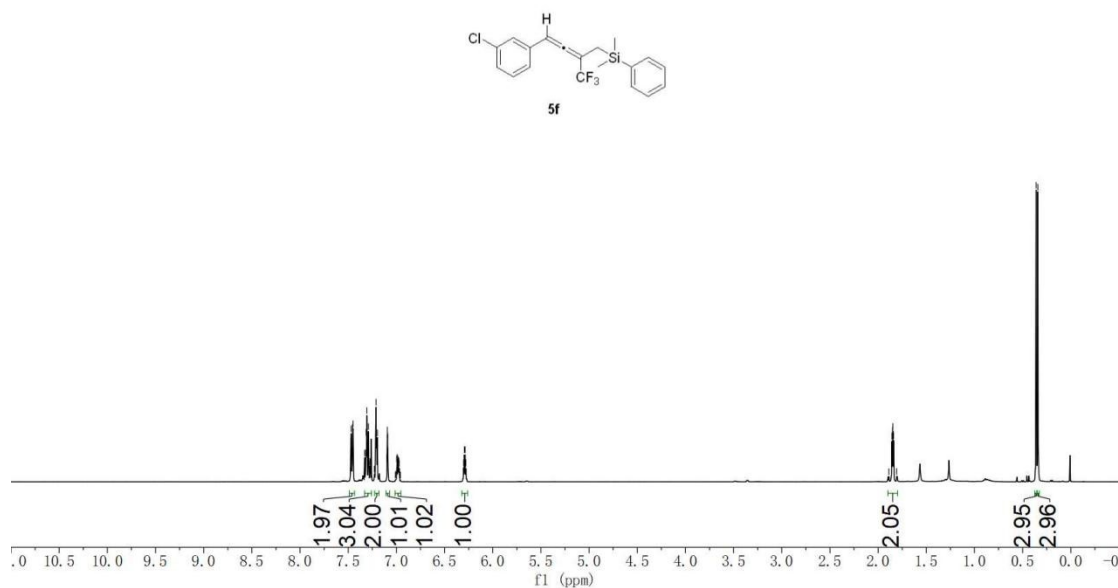


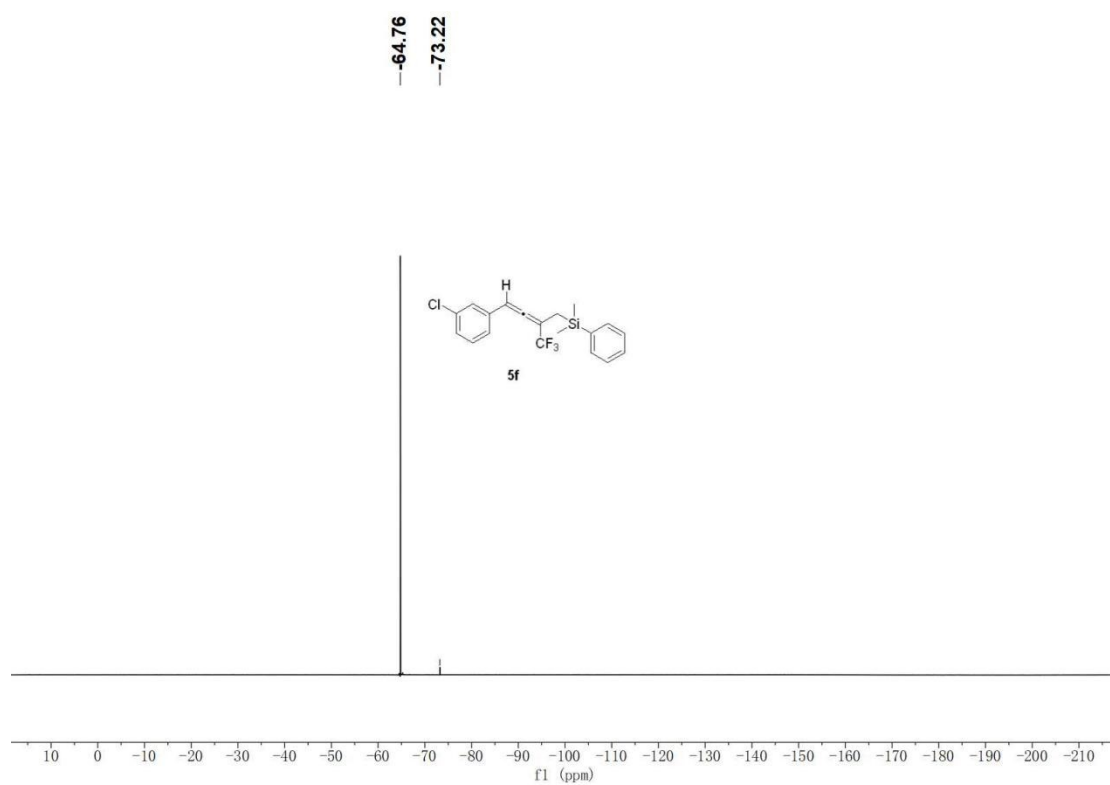
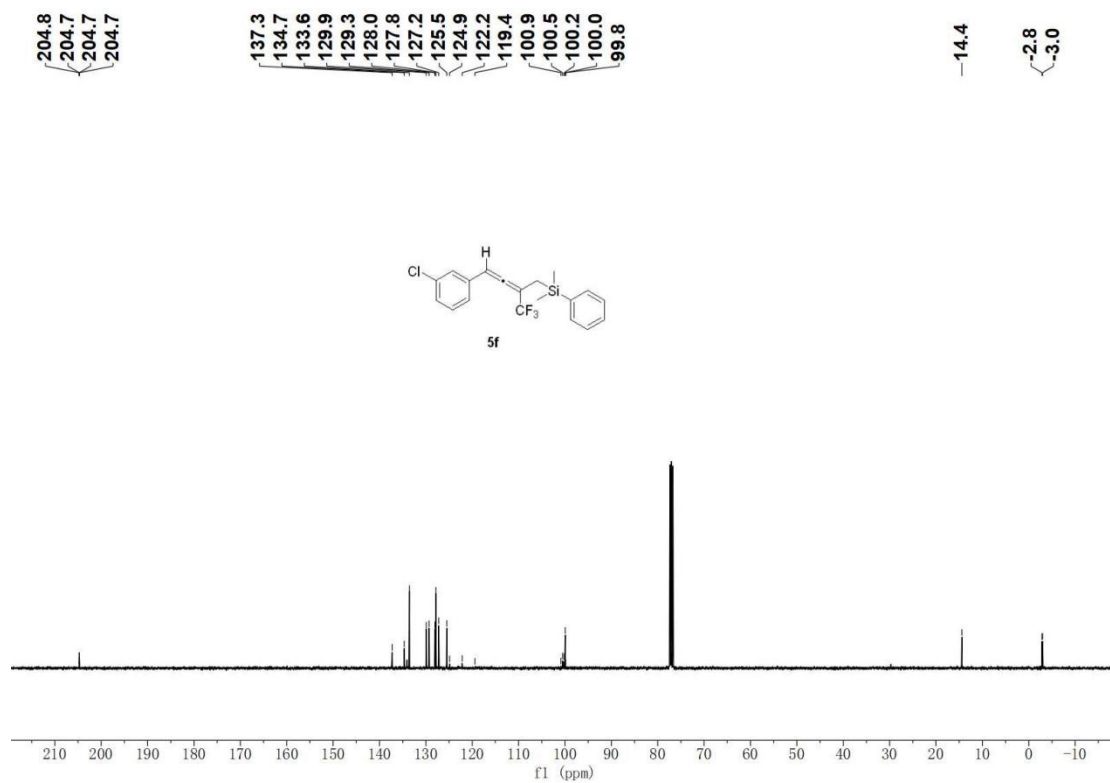
(4-(4-chlorophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)dimethyl(phenyl)silane(5e)



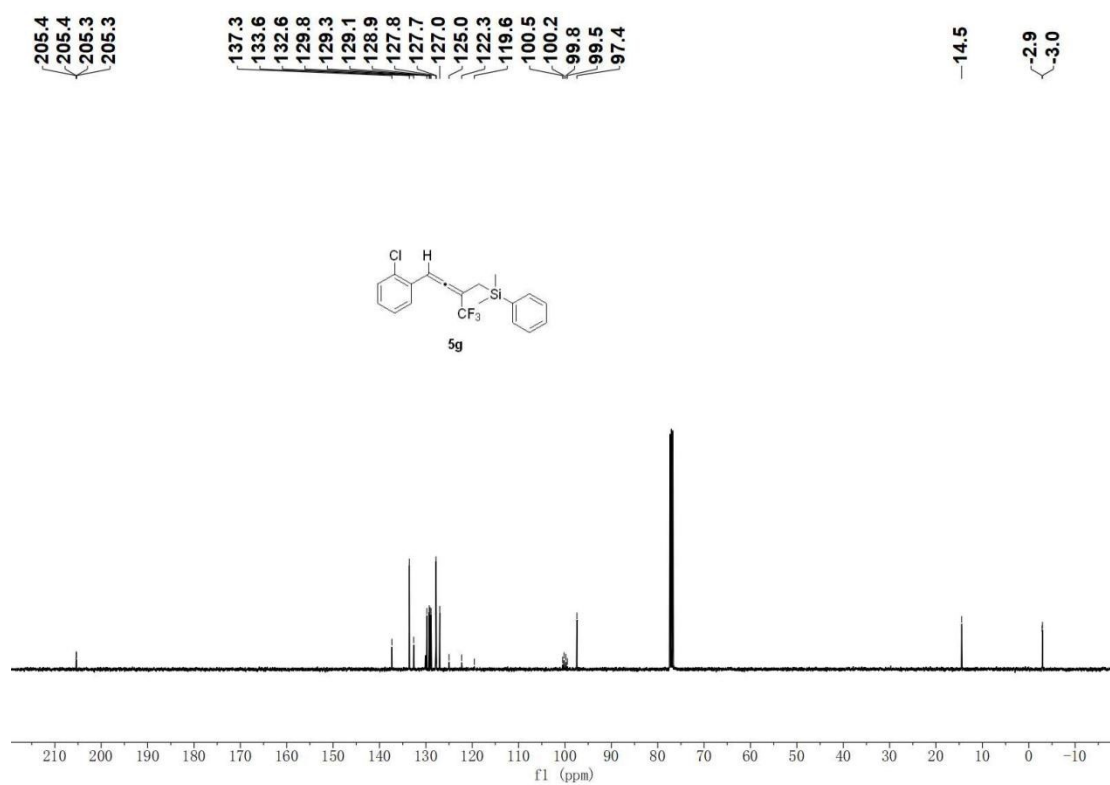
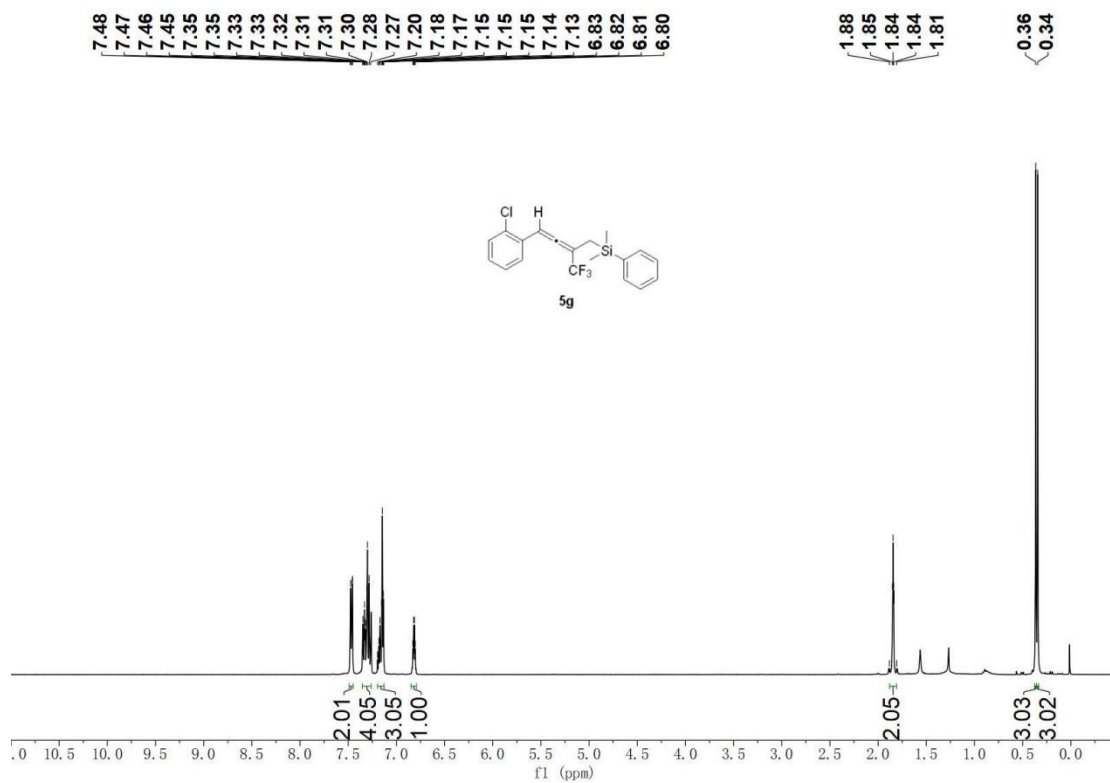


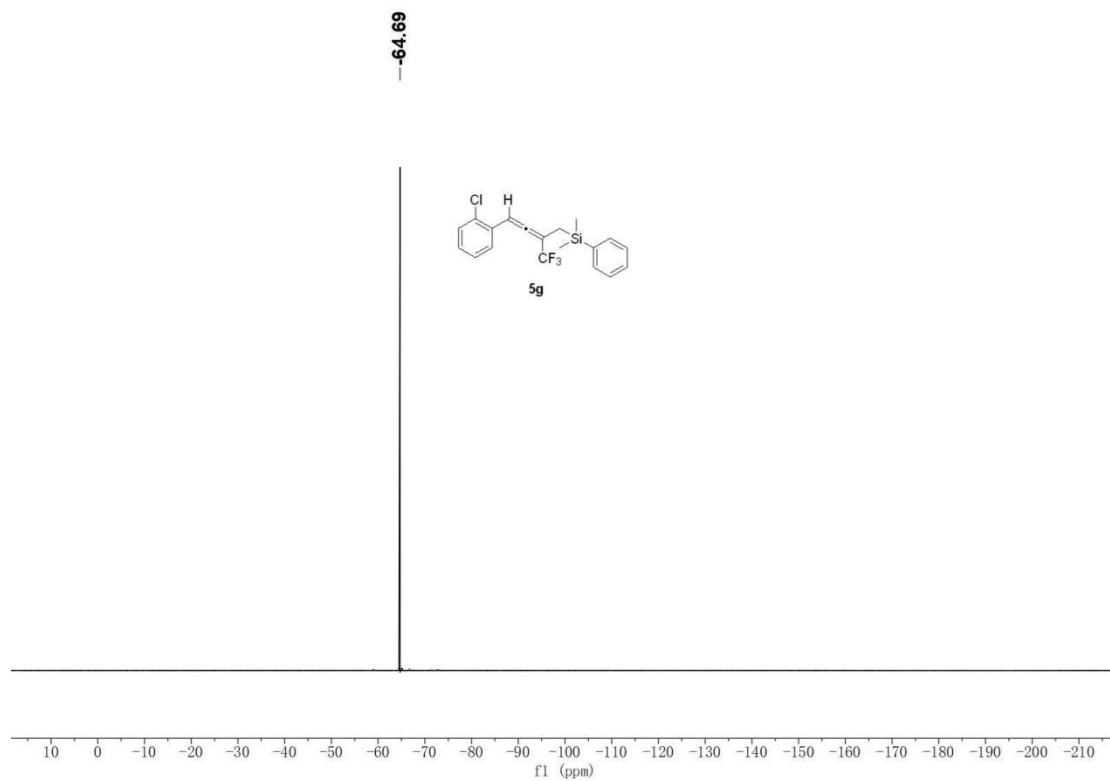
(4-(3-chlorophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)dimethyl(phenyl)silane(5f)





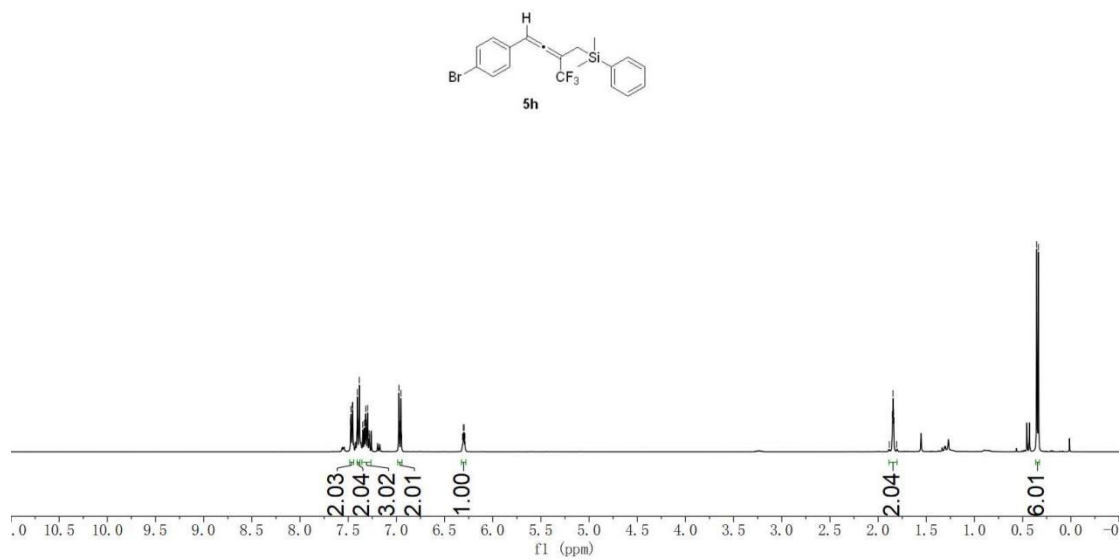
(4-(2-chlorophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)dimethyl(phenyl)silane(5g)

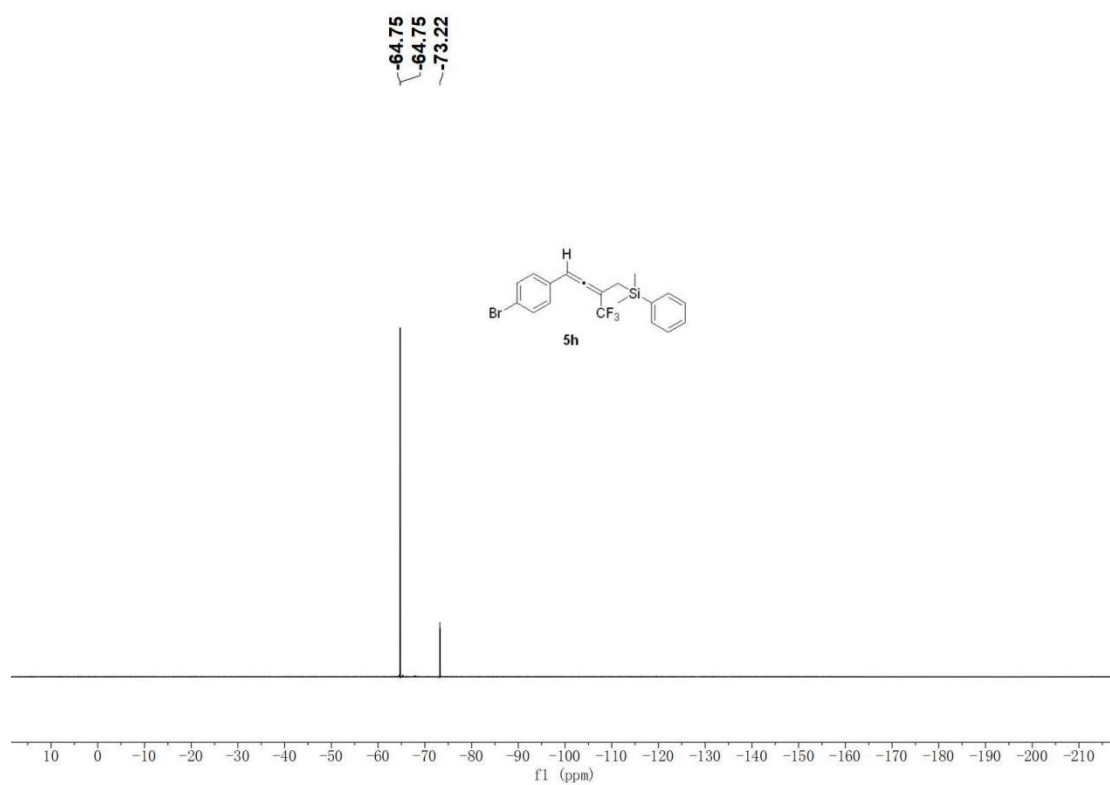
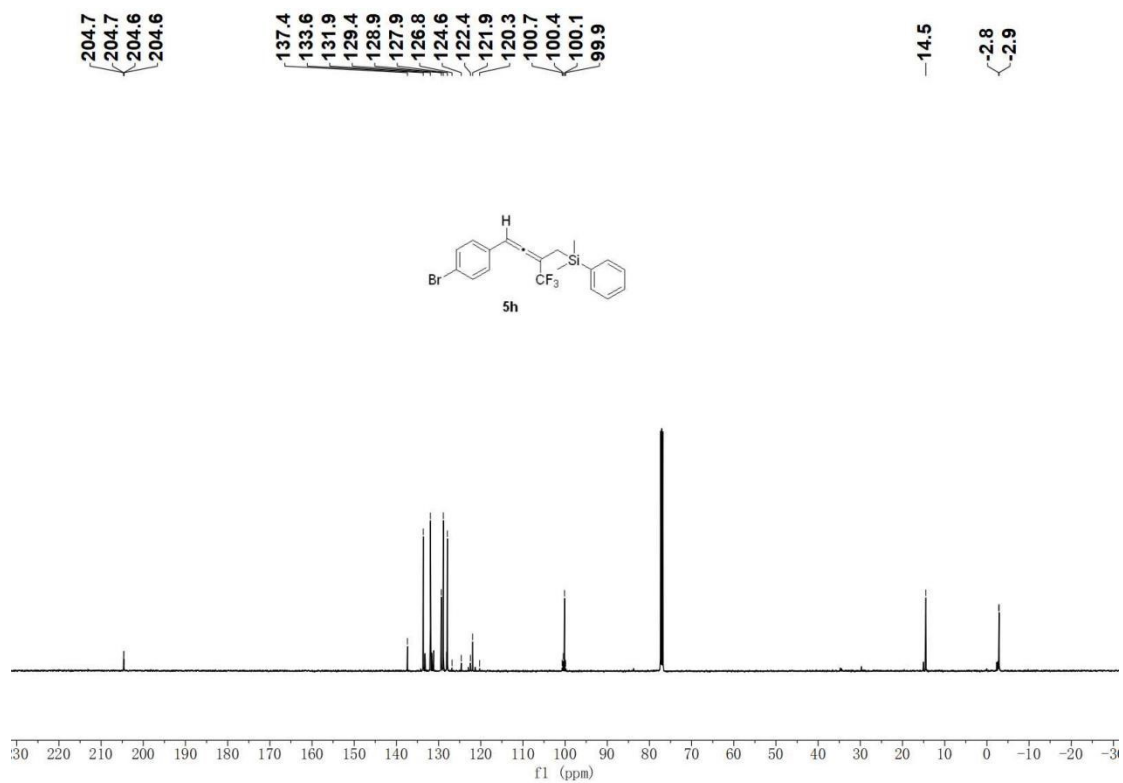




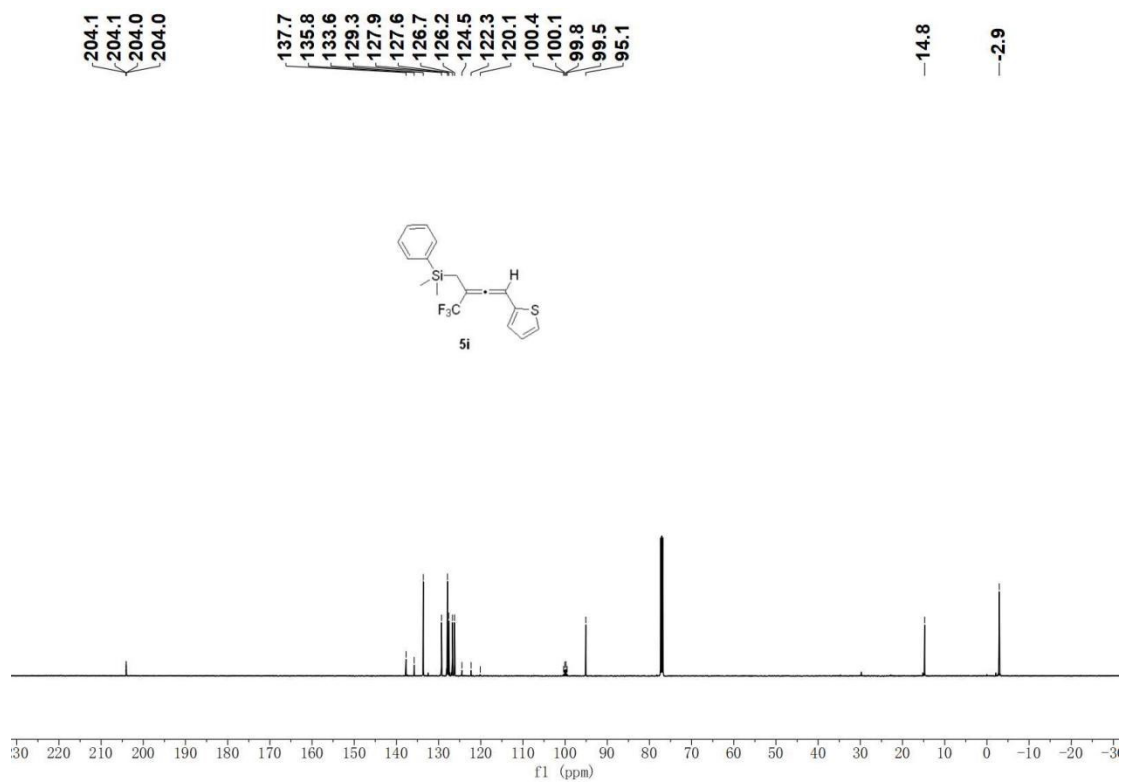
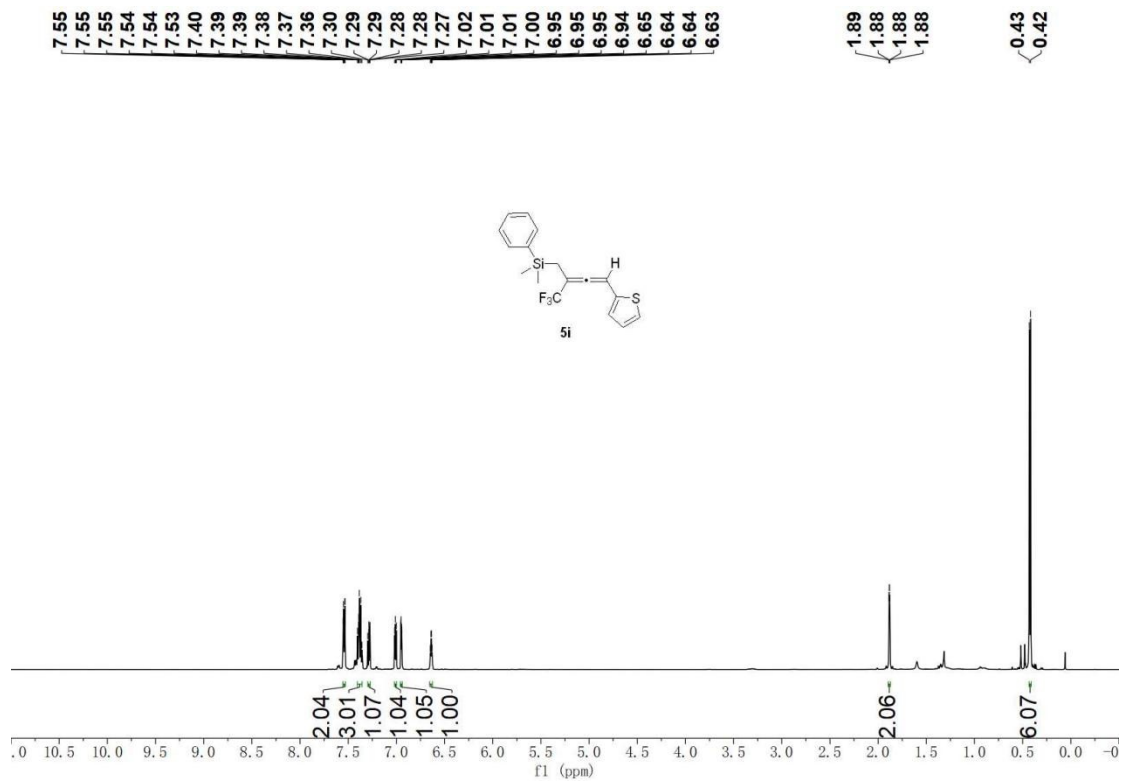
(4-(4-bromophenyl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)dimethyl(phenyl)silane(5h)

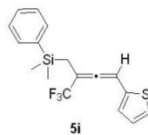
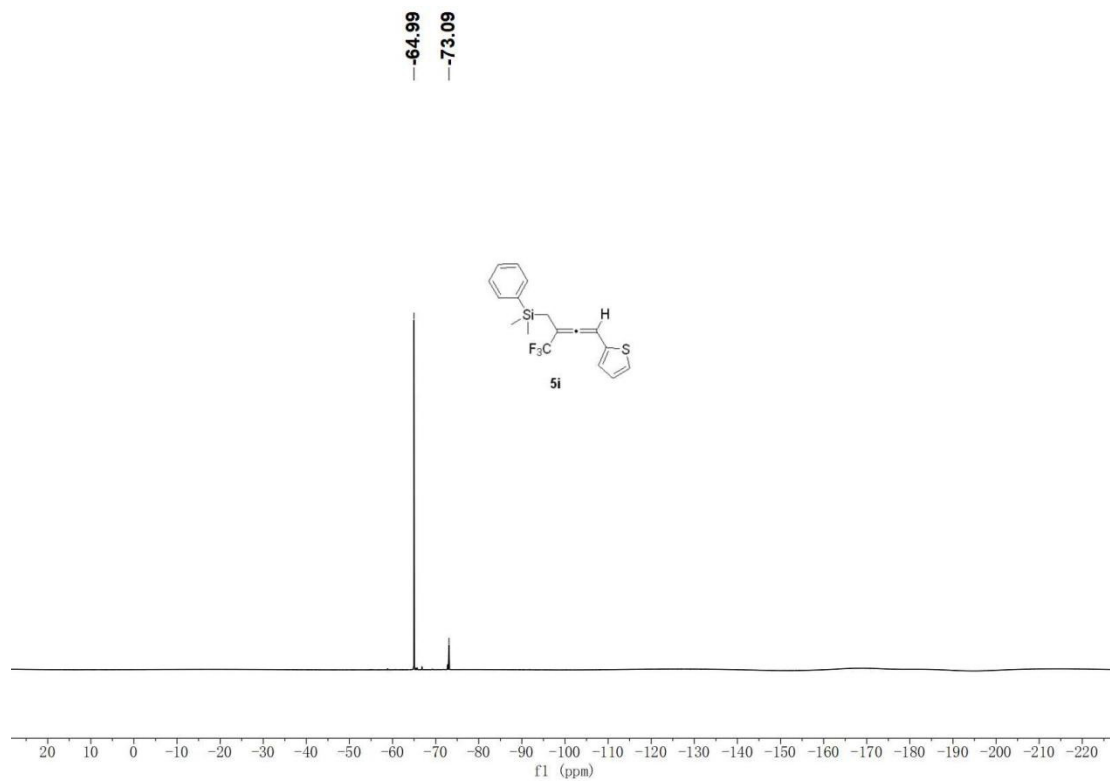
7.48 7.47 7.46 7.45 7.41 7.40 7.39 7.38 7.35 7.33 7.32 7.31 7.30 7.30 6.97 6.97 6.96 6.95 6.30 6.30 6.29 1.88 1.85 1.85 1.84 1.81 0.35 0.34



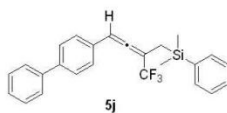
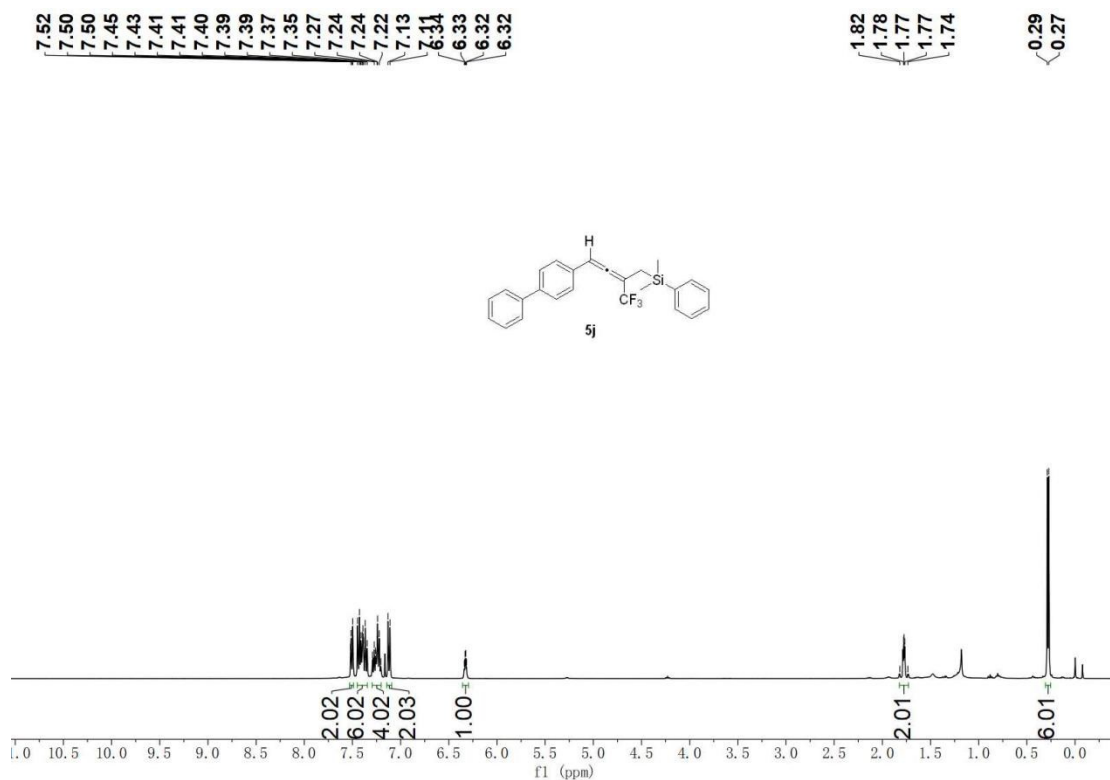


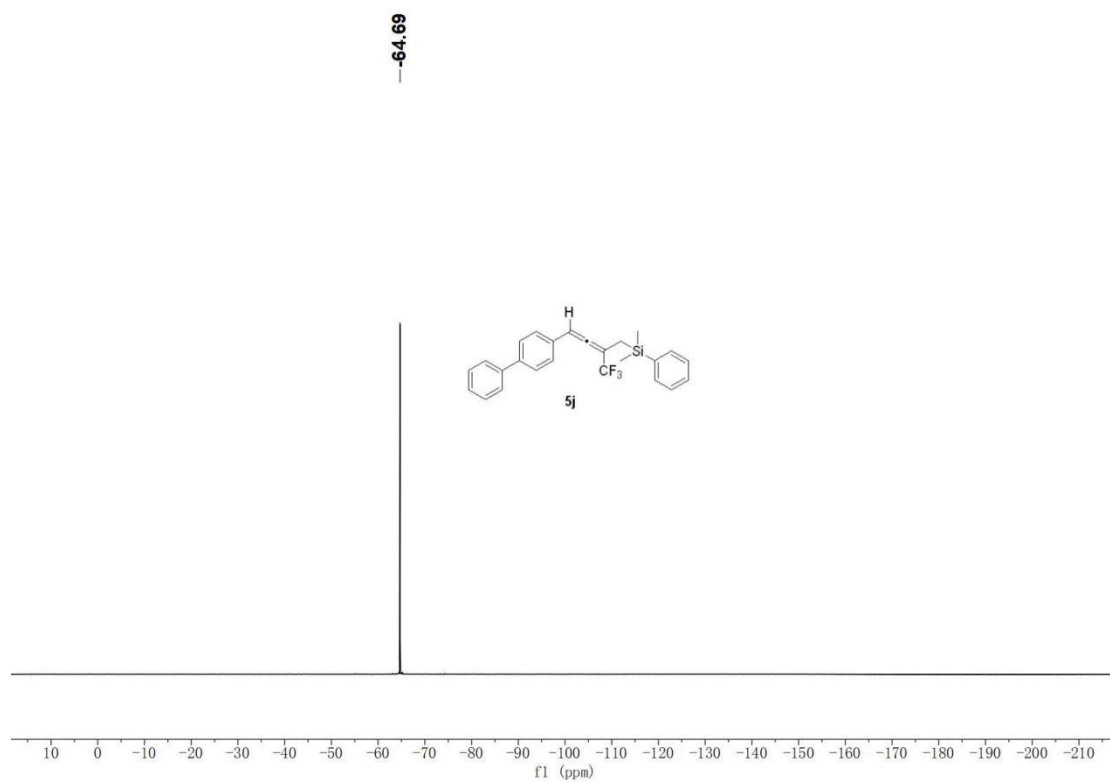
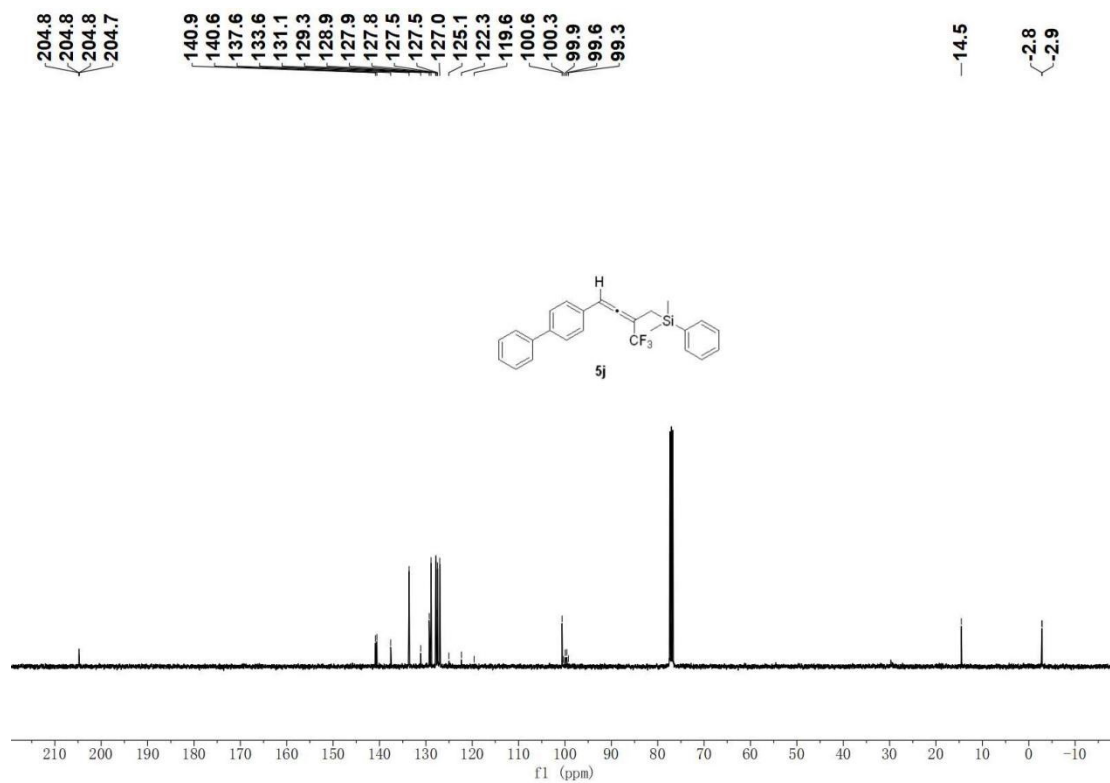
dimethyl(phenyl)(4-(thiophen-2-yl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)silane(5i)



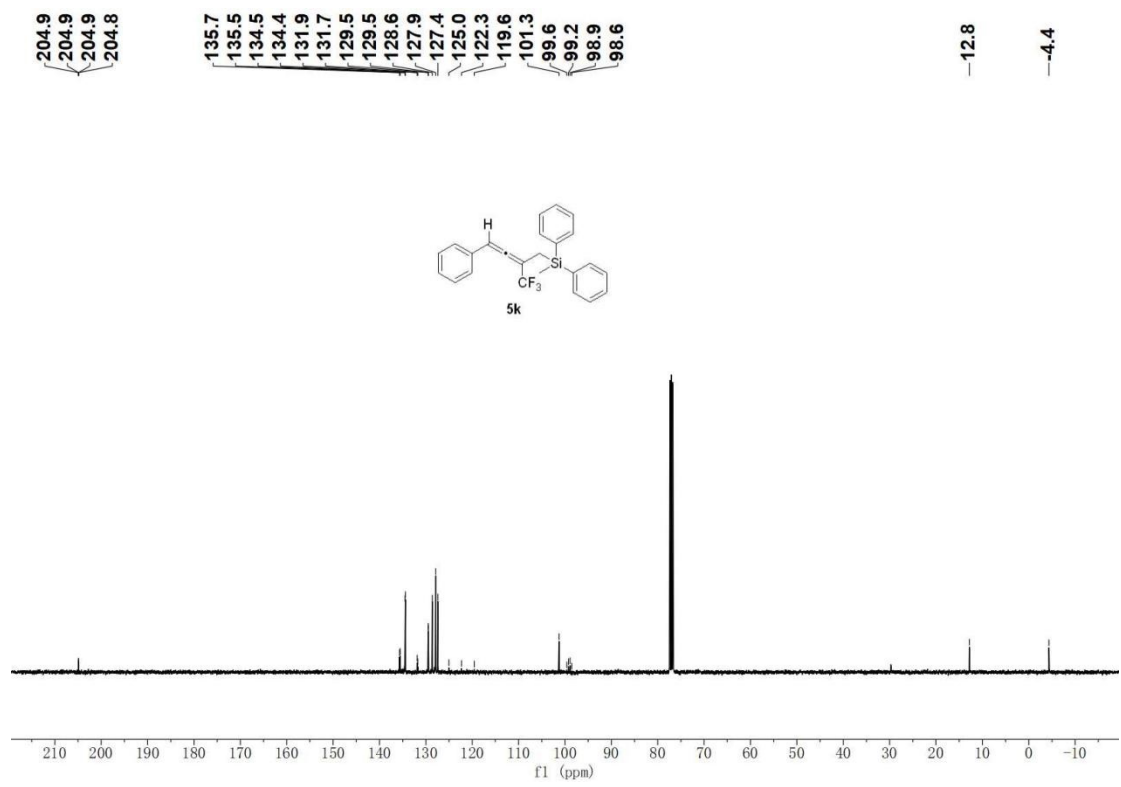
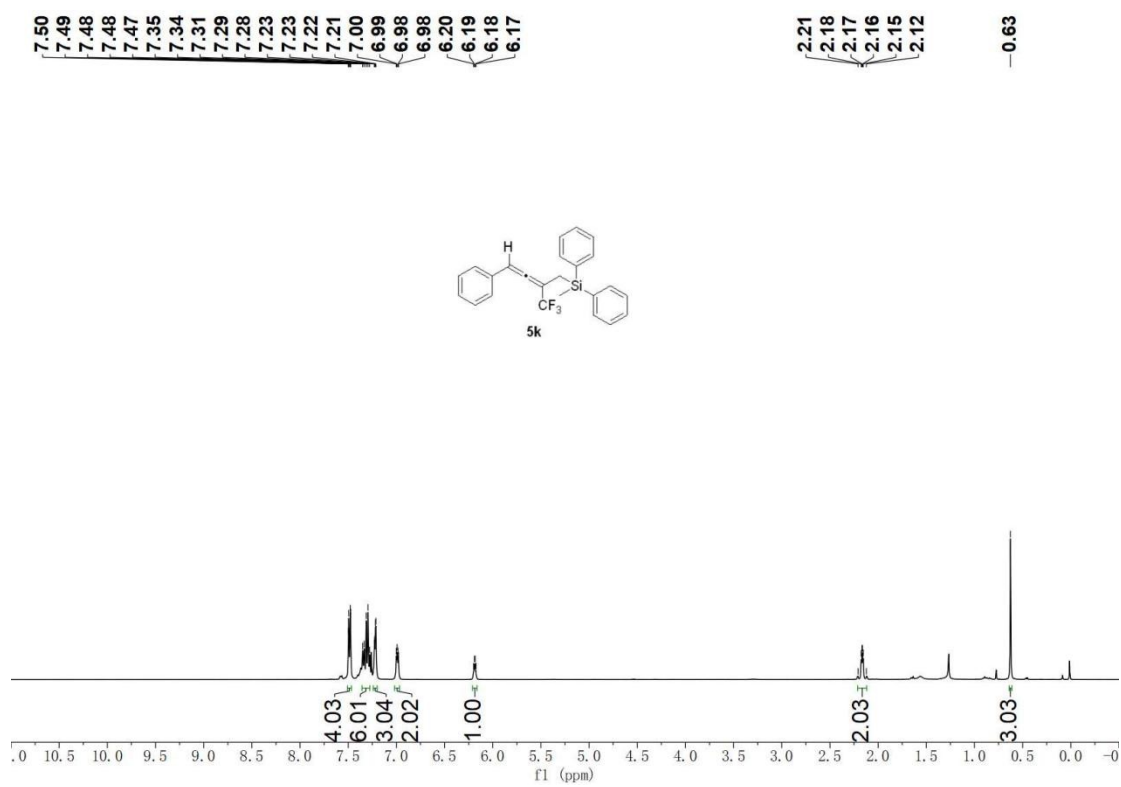


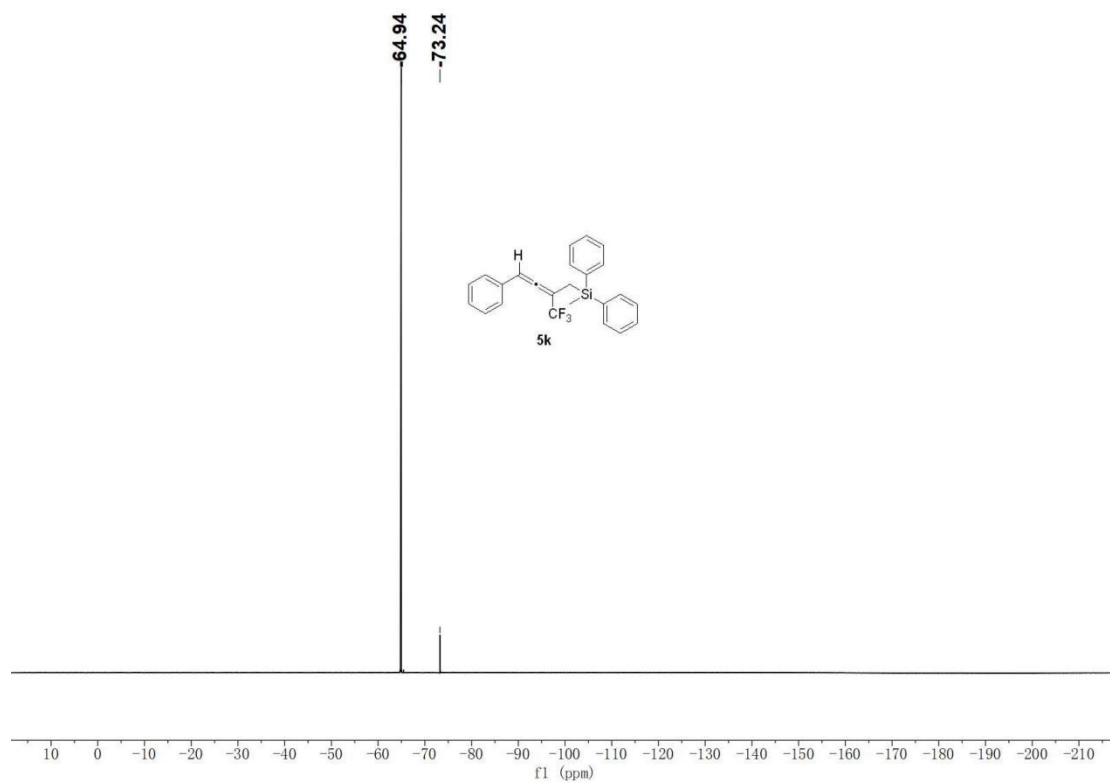
(4-([1,1'-biphenyl]-4-yl)-2-(trifluoromethyl)buta-2,3-dien-1-yl)dimethyl(phenyl)silane(5j)



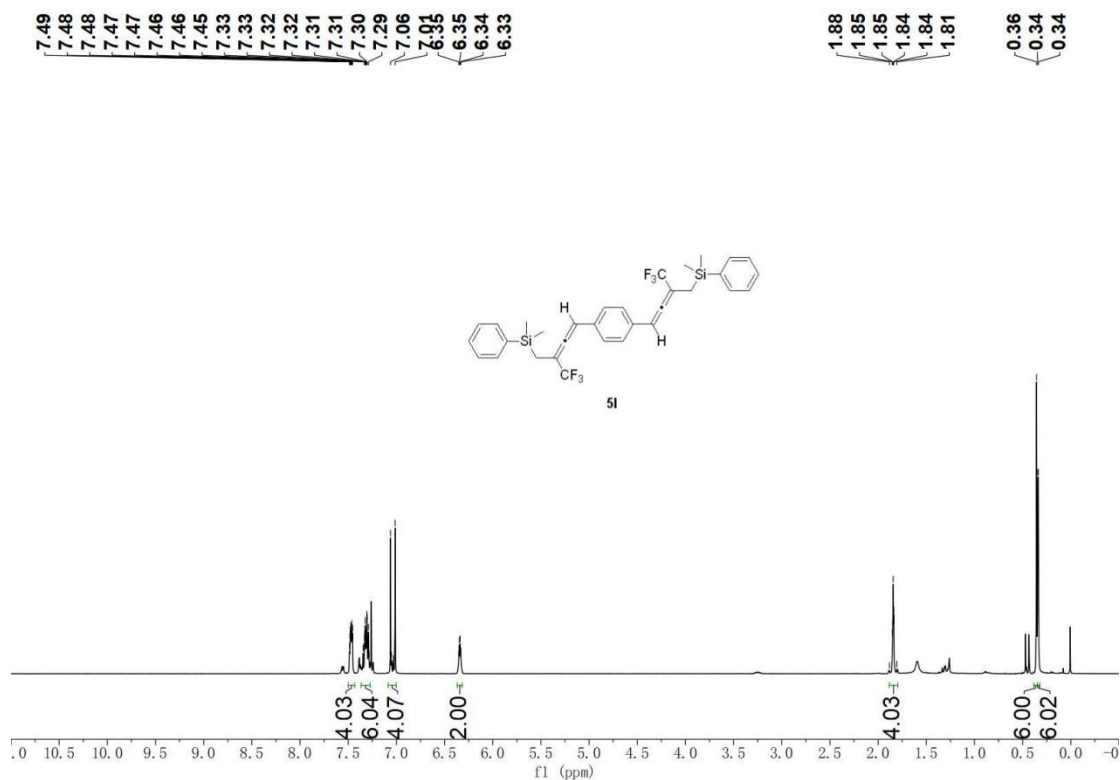


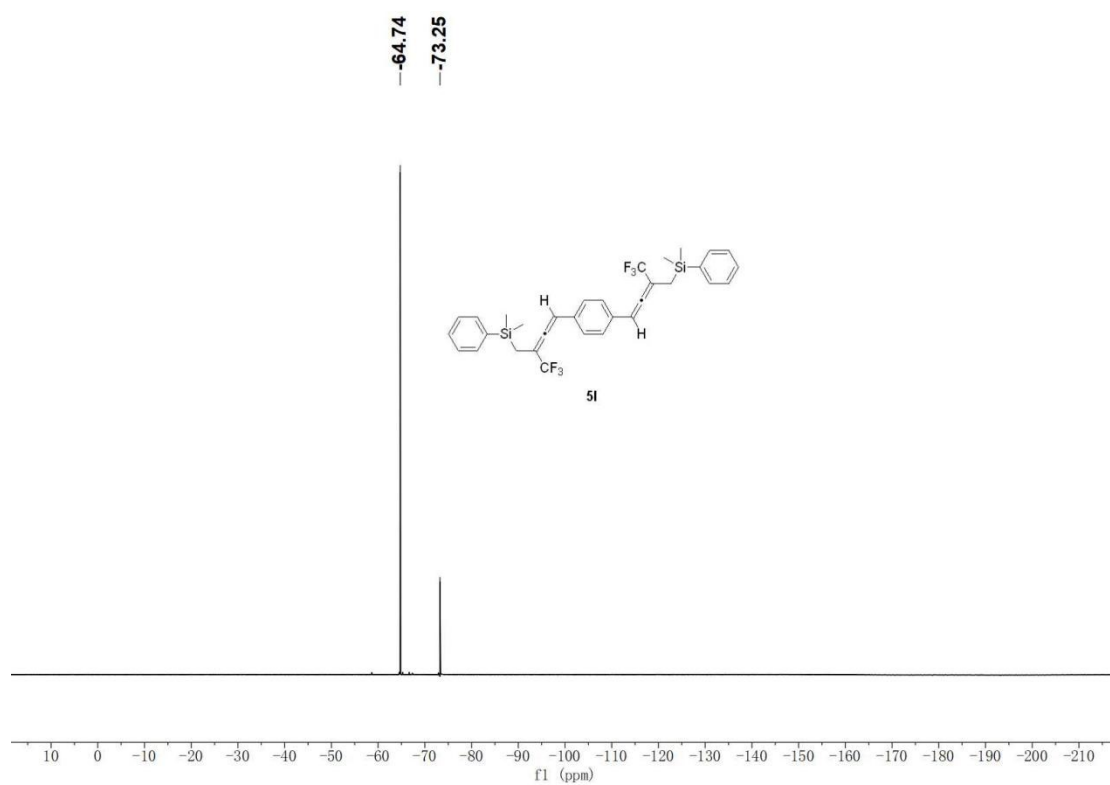
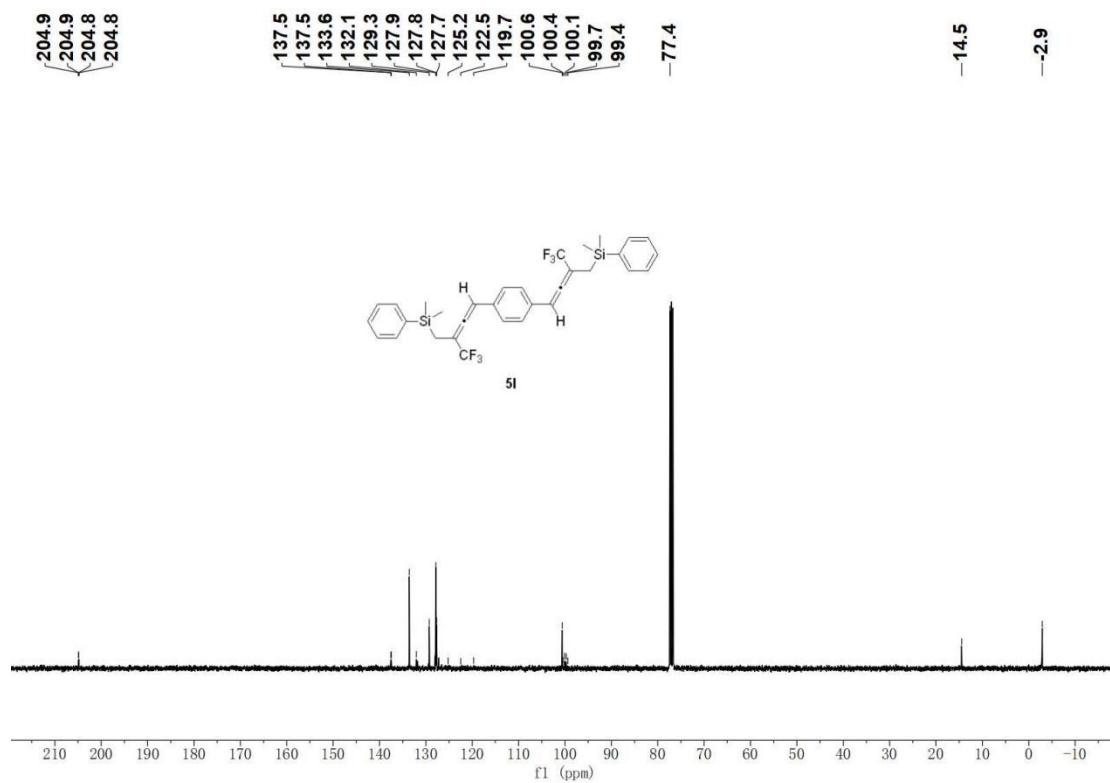
Methyldiphenyl(4-phenyl-2-(trifluoromethyl)buta-2,3-dien-1-yl)silane(5k)



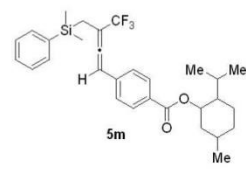
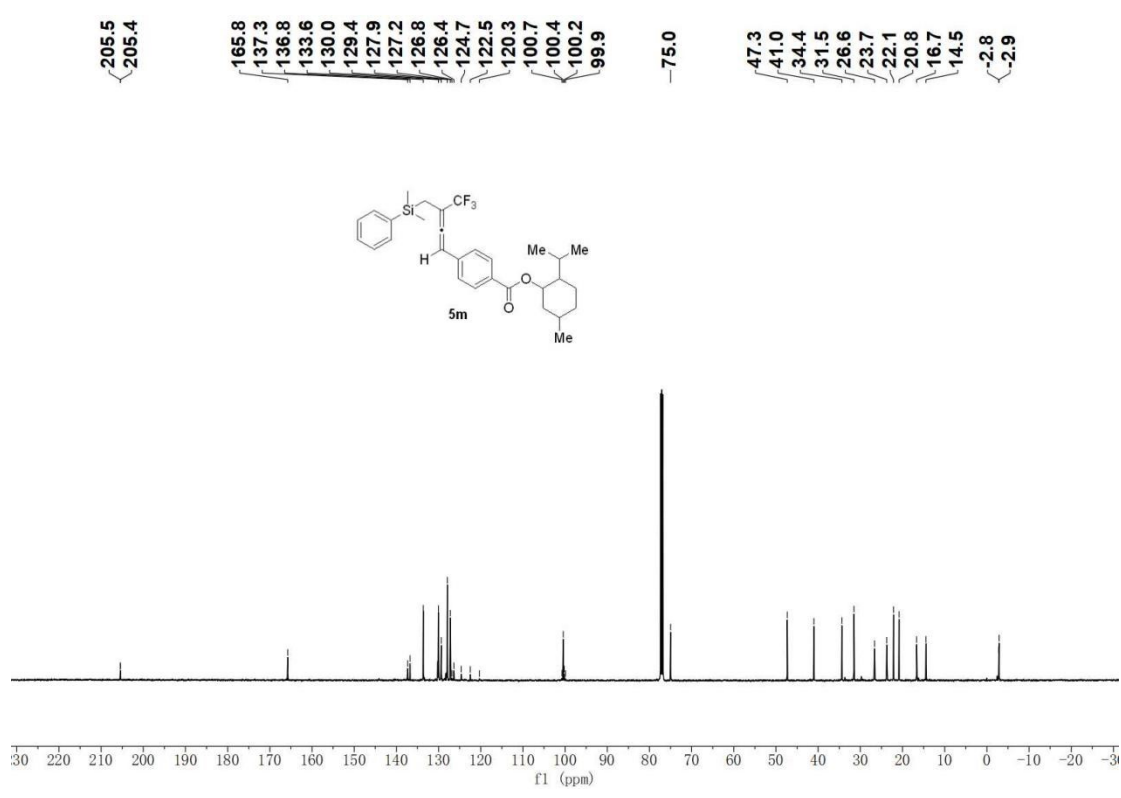
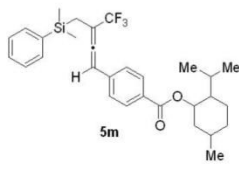
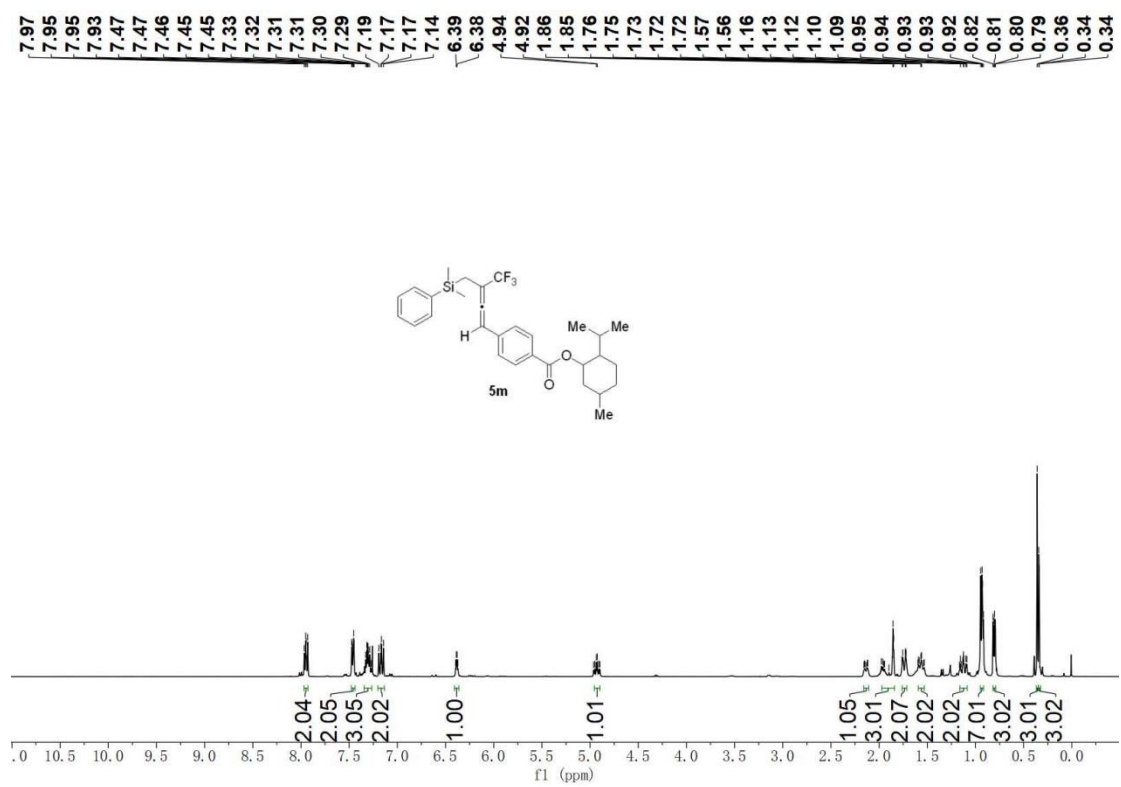


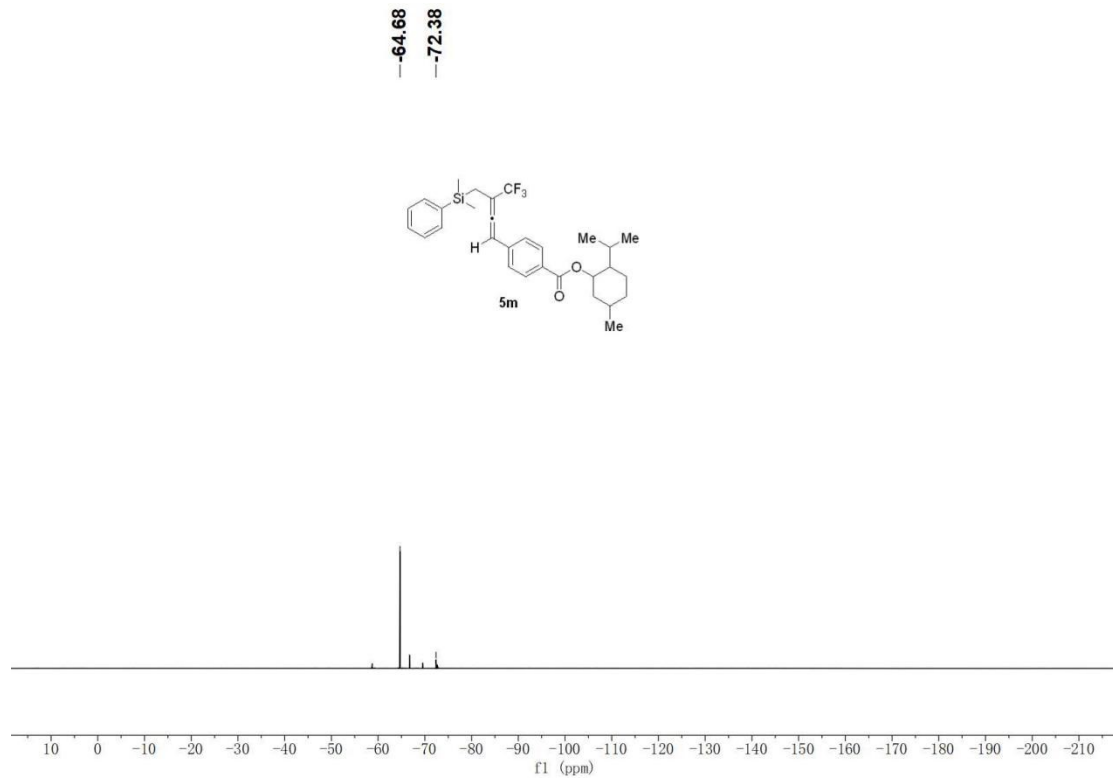
1,4-bis(3-((dimethyl(phenyl)silyl)methyl)-4,4,4-trifluorobuta-1,2-dien-1-yl)benzene(5l)



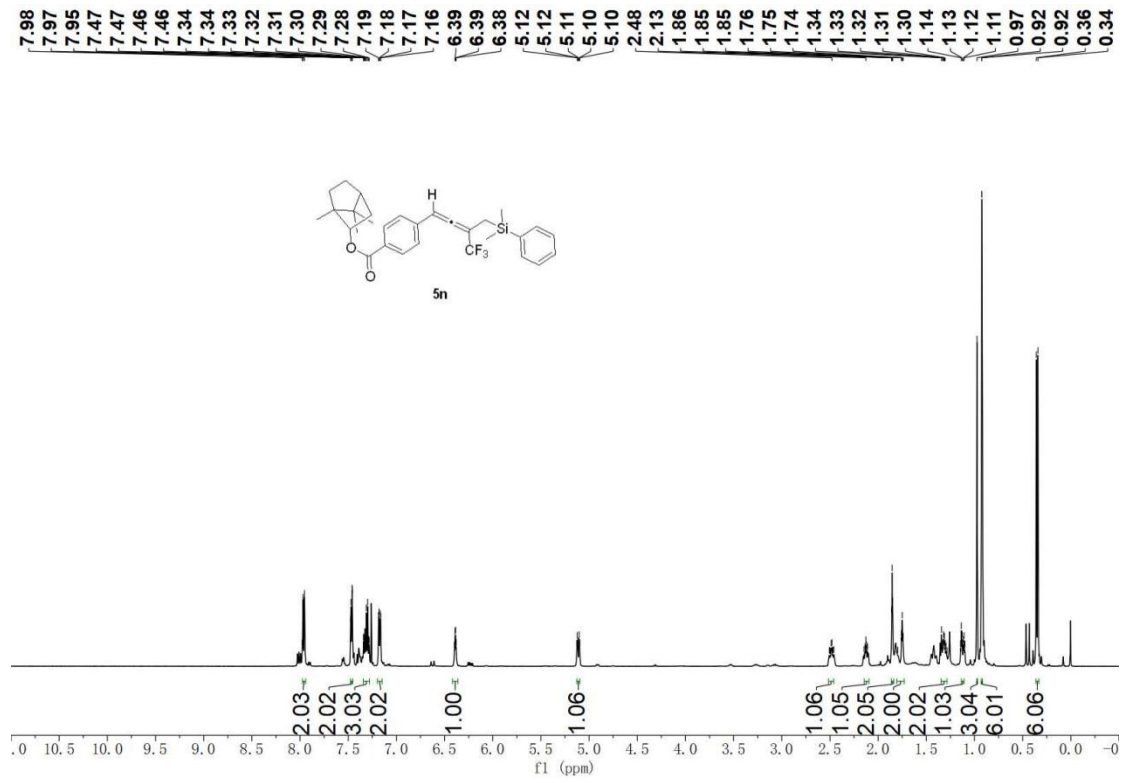


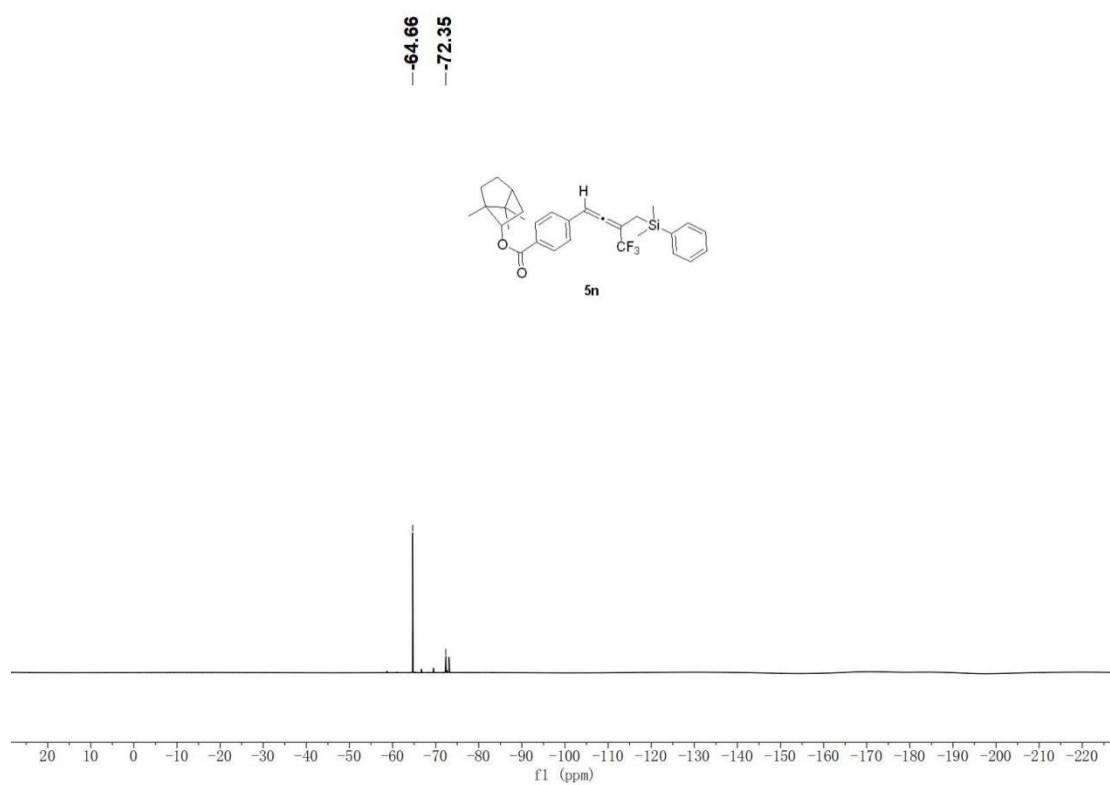
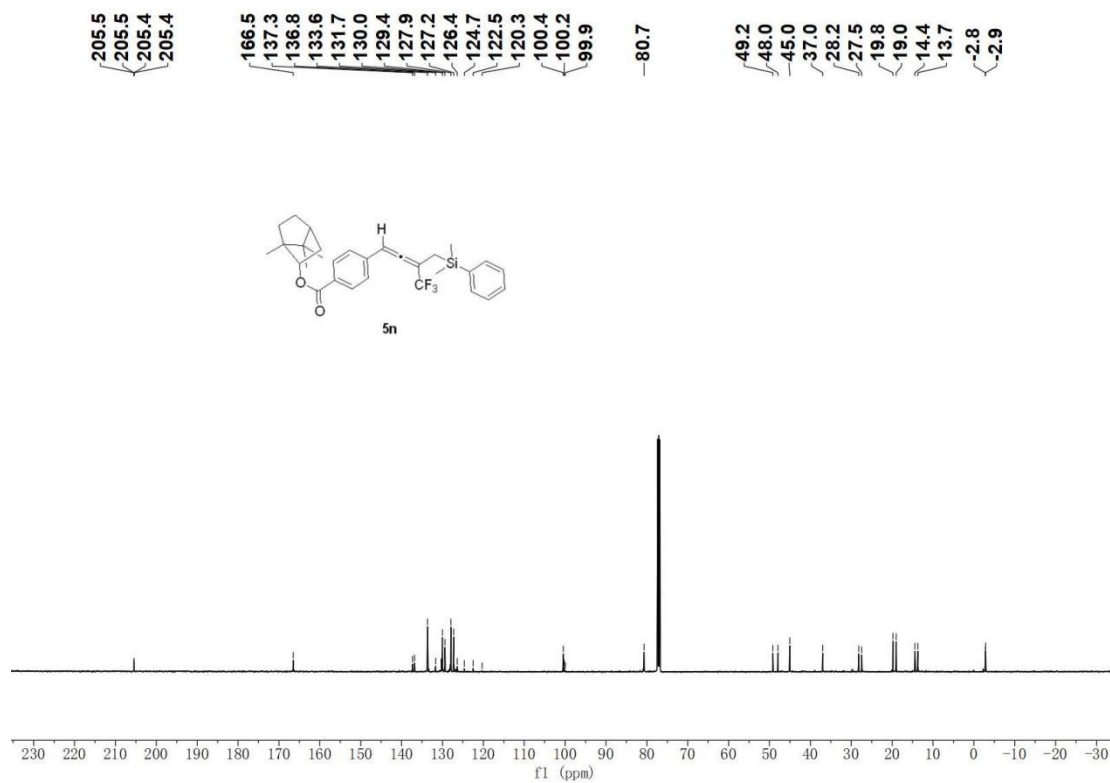
2-isopropyl-5-methylcyclohexyl 4-(3-((dimethyl(phenyl)silyl)methyl)-4,4,4-trifluorobuta-1,2-dien-1-yl)benzoate(5m)





1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl 4-(3-((dimethyl(phenyl)silyl)methyl)-4,4,4-trifluorobuta-1,2-dien-1-yl)benzoate(5n)

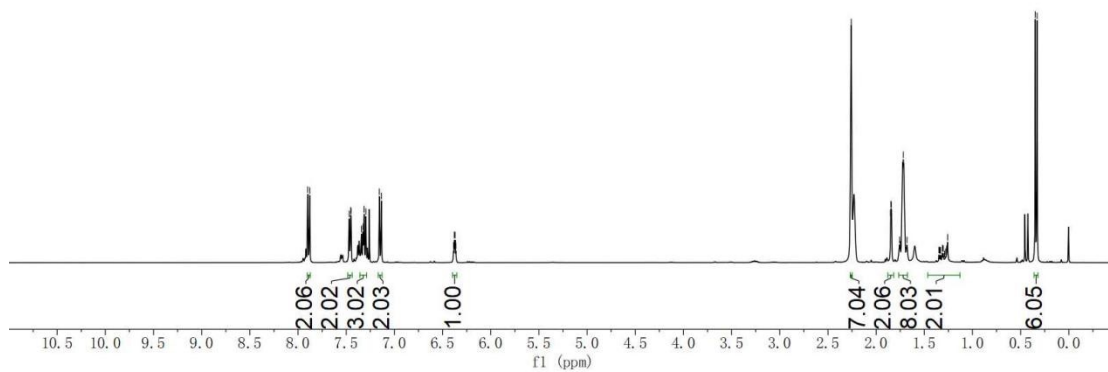
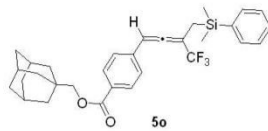




((1s,3s)-adamantan-1-yl)methyl 4-(3-((dimethyl(phenyl)silyl)methyl)-4,4,4-trifluorobuta-1,2-dien-1-yl)benzoate(5o)

7.90
7.88
7.88
7.47
7.47
7.45
7.34
7.33
7.32
7.31
7.30
7.16
7.15
7.14
7.14
6.39
6.38
6.37
6.36

2.26
1.85
1.85
1.84
1.84
1.76
1.75
1.73
1.72
1.71
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1.33
1.31
1.30
1.29
1.28
1.27
1.26
0.35
0.33



205.4
205.4
205.3
205.3

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127.1
124.9
122.2
119.4

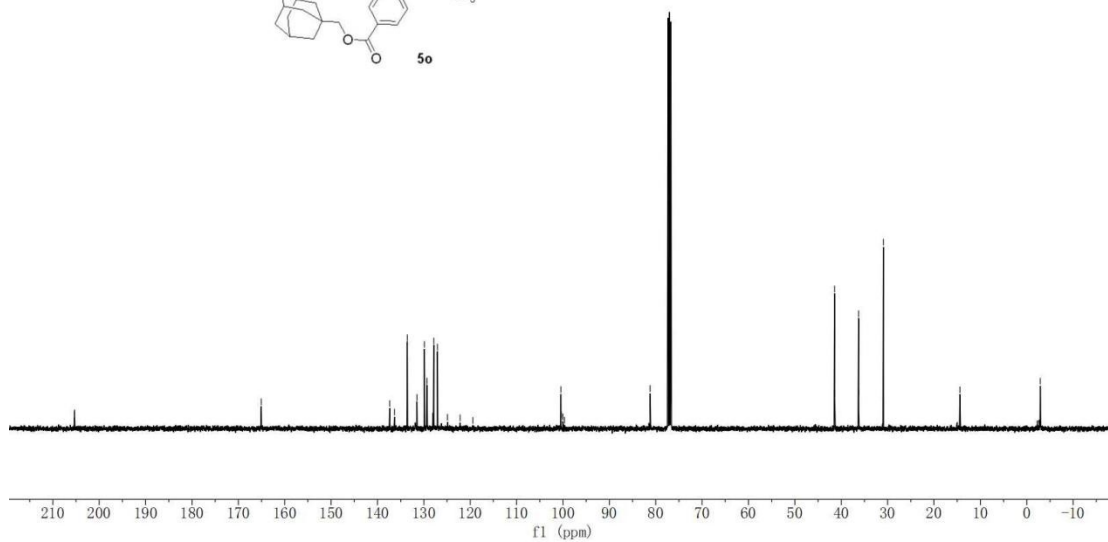
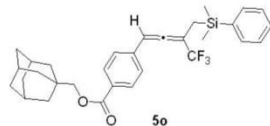
100.5
100.4
100.0
99.7

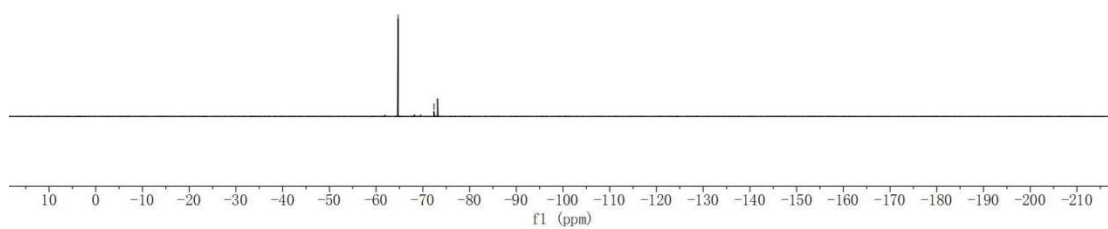
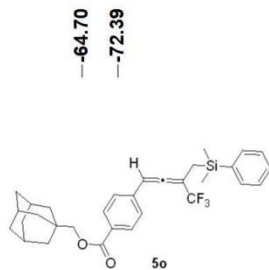
-81.2

41.4
41.4
36.3
36.2
30.9

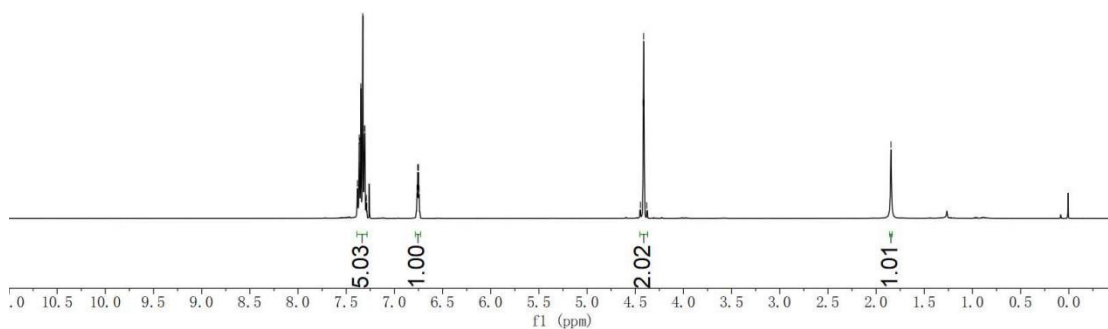
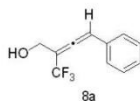
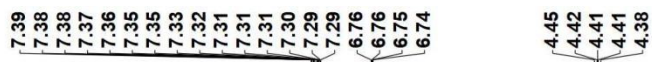
-14.4

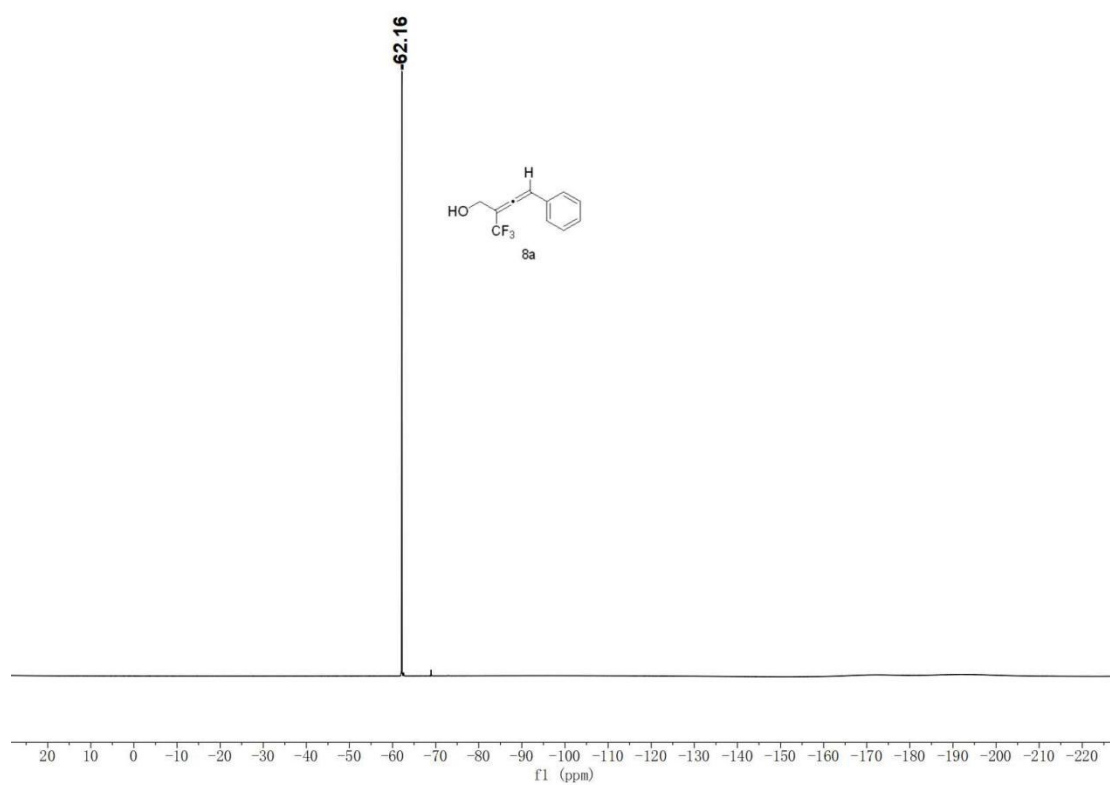
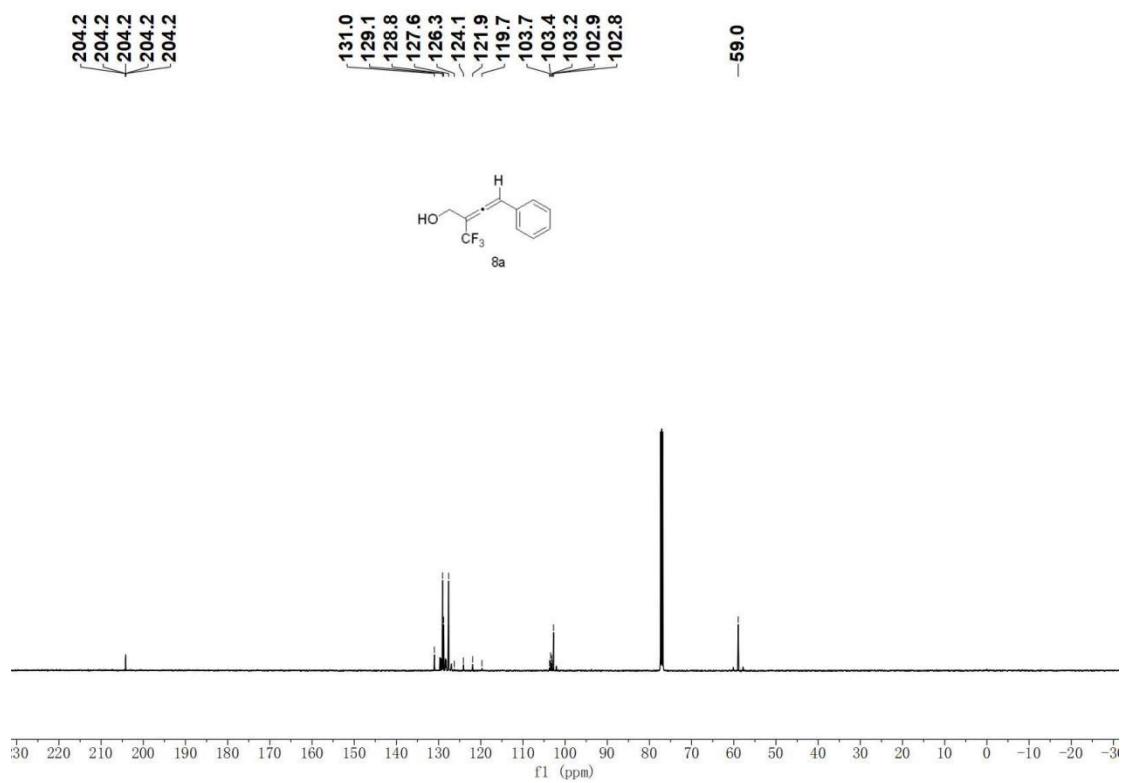
-2.9





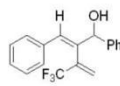
4-phenyl-2-(trifluoromethyl)buta-2,3-dien-1-ol(8a)



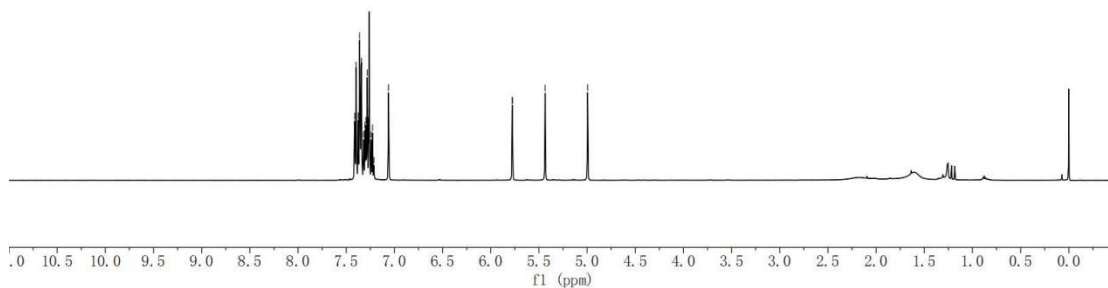


(E)-2-benzylidene-1-phenyl-3-(trifluoromethyl)but-3-en-1-ol(9a)

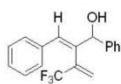
7.41
7.40
7.38
7.36
7.36
7.35
7.34
7.32
7.31
7.30
7.28
7.28
7.27
7.27
7.24
7.24
7.24
7.23
7.23
7.22
7.21
7.06
5.78
5.77
5.44
4.99



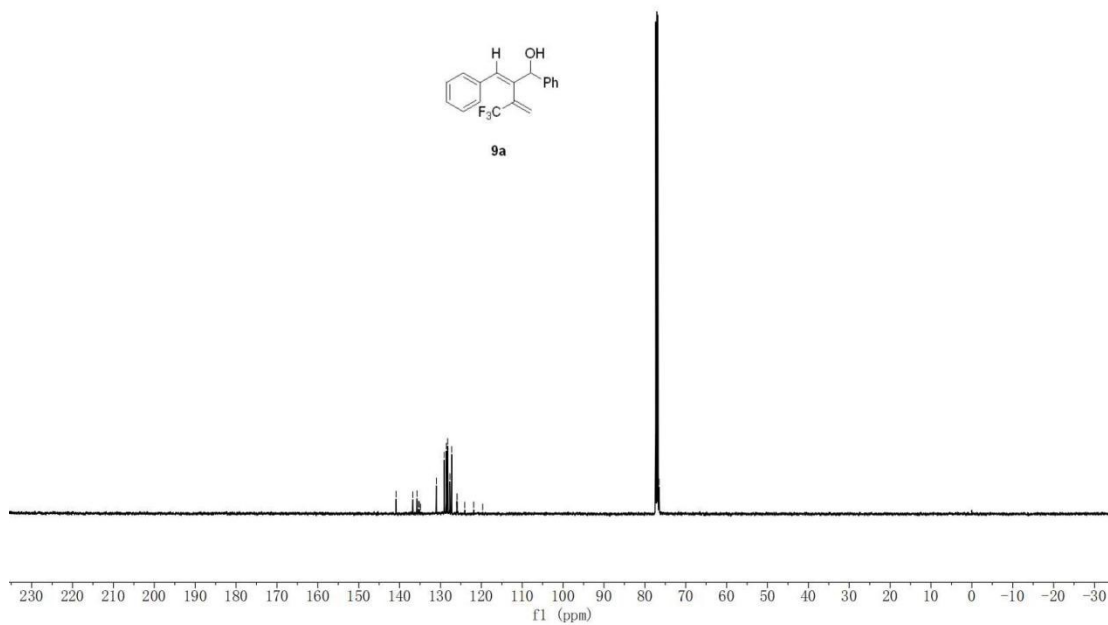
9a



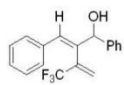
140.8
136.8
135.7
135.4
135.1
134.9
131.0
129.1
128.6
128.2
128.2
127.7
127.2
126.0
126.0
125.9
125.9
124.1
121.9
119.7
76.5



9a



-64.35



9a

