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Supporting Information

Graphene-Enhanced Platinum -Catalysed Hydrosilylation of Amides and Chalcones: A Sustainable Strategy Allocated with *in-situ* Heterogenization and Multi-Task Application of H_2PtCl_6

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1. General Information

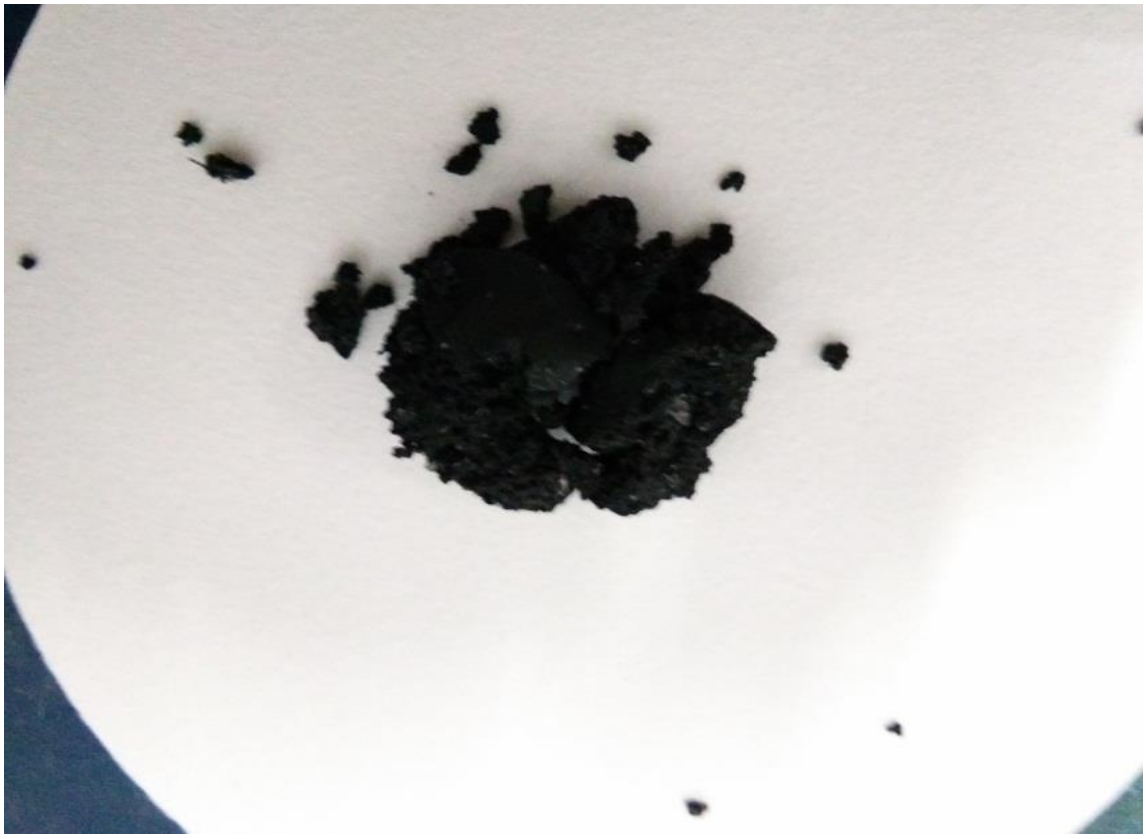
Unless specifically stated, all reagents were commercially obtained and where appropriate, purified prior to use. For example, all the aldehydes recrystallized or distilled prior to use. Dichloromethane, toluene, were freshly distilled from CaH_2, THF was freshly distilled from sodium metal prior to use. Other commercially available reagents and solvents were used directly without purification. Reactions were monitored by thin layer chromatography (TLC) using silica gel plates. Flash column chromatography was performed over silica (300-400 mesh). 1H, 13C, 19F, and 29Si spectra were recorded on a Bruker 400 MHz or 500 MHz spectrometer in $CDCl_3$. Multiplicities were given as: s(singlet); d (doublet); dd (doublets of doublet); t (triplet); q (quartet);or m (multiplets). High resolution mass spectra (HRMS) of the products were obtained on a Bruker Daltonics micro TOF-spectrometer. Infrared spectra were recorded on a Nexus 870 FTIR spectrometer. The morphology of the polymer fracture surface was characterized by scanning electron micrograph (SEM, Hitachi S3000N). The samples were frozen in liquid nitrogen and snapped immediately. Transmission electron microscopy (TEM) was carried out with the phosphotungstic acid staining method on a Hitachi H-7650 transmission electron microscope.

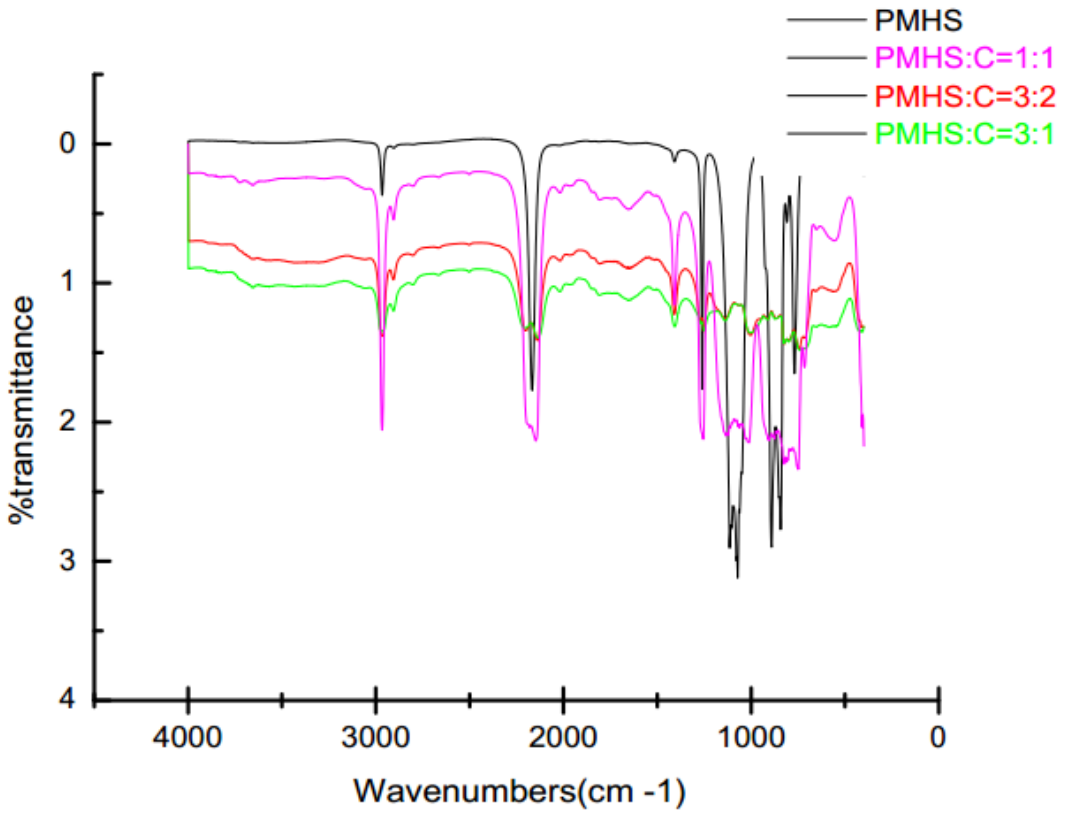
2. General Procedure for the synthesis of PMHS@graphene (or G@PMHS) and Pt@G@Si

2.1 General Procedure for synthesis of Graphene-dispersed PMHS.

A mixture of graphene (0.66 g, 27.6 mmol calculated to C=C) and $Co(acac)_2$ (5 mol%, 4.9 mg, 13.8 μmol) was treated with the PMHS (82.8 mmol, 5.34 g, 1.55% Si-H) under an argon atmosphere, and the solvent-free mixture was stirred at 100 ℃. After it was allowed to stand for 24 h at the same temperature, the residues were passed through a funnel, washed with the distilled water, dried for 24 h at 80 ℃ under vacuum to give the graphene-dispersed PMHS material (G@PMHS).

The preparation of graphite-dispersed PMHS (PMHS@graphite or Graphite@PMHS) is similarly to above process.

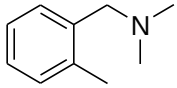




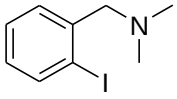
2.2 General procedure for synthesis of amines and Pt@G@Si catalyst in the platinum-catalyzed reduction of amides

2.2.1 General procedure for the preparation of amine (2)

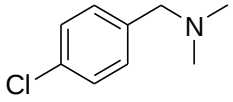
A solution of amide **1** (1 mmol) and G@PMHS (3.0 equiv Si-H) was treated with a THF solution of $H_2PtCl_6 \cdot 6H_2O$ (1 mol% based on the amide) at 60 ℃. The homogeneous solution became gradually viscous, and set to gel. After it was allowed to stand for 4 h, the reaction mixture was extracted with EtOAc. The extracts were passed through a funnel, dried for 24 h at 80 ℃ under vacuum to give the graphene and cross-linked organosilicon-supported platinum catalyst (Pt@G@Si). For the isolation of the corresponding product in the reduction of amide, after removal of EtOAc, purification of residue by alumina column chromatography gave the amine **2**. These amine products were known products and have been reported elsewhere.



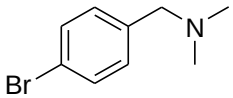
N,N-dimethyl-1-(o-tolyl) methanamine (2a): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23, 7.22, 7.19, 7.18, 7.15, 3.11, 2.79, 2.27. ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.3, 136.5, 133.7, 130.1, 128.5, 125.6, 38.1, 34.3, 18.6 ppm.



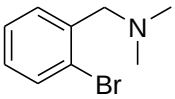
1-(2-iodophenyl)-N,N-dimethylmethanamine (2b): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (dd, J = 7.9, 0.9 Hz, 1H), 7.38 (dd, J = 7.6, 1.6 Hz, 1H), 7.31 (dd, J = 7.3, 0.8 Hz, 1H), 6.93 (td, J = 7.6, 1.7 Hz, 1H), 3.46 (s, 2H), 2.30 (s, 6H)**.** ^{13}CNMR (101 MHz, $CDCl_3$) δ 141.3, 139.6, 130.5, 128.8, 128.1, 100.8, 68.1, 45.6 ppm.



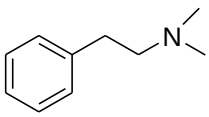
1-(4-chlorophenyl)-N,N-dimethylmethanamine (2c): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (s, 1H), 7.21 – 7.13 (m, 4H), 3.30 (s, 2H), 2.14 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.4, 132.8, 130.4, 128.8– 128.3, 68.0, 63.6, 45.3 ppm.



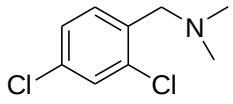
1-(4-bromophenyl)-N,N-dimethylmethanamine (2d): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.37 (m, 2H), 7.15 (d, *J* = 8.3 Hz, 2H), 3.32 (s, 2H), 2.18 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.9, 131.2 ,30.5, 120.7, 63.5, 45.2 ppm.



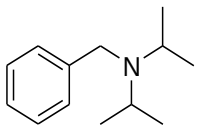
1-(2-bromophenyl)-N,N-dimethylmethanamine (2e): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.40 (d, *J* = 1.5 Hz, 1H), 7.26 (s, 1H), 7.08 (td, *J* = 7.8, 1.6 Hz, 1H), 3.52 (s, 2H), 2.29 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.1, 132.8, 131.0, 128.5, 127.3, 124.8, 63.3, 45.5 ppm.



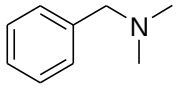
N,N-dimethyl-2-phenylethanamine (2f): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (dd, *J* = 12.1, 4.9 Hz, 2H), 7.20 (d, *J* = 7.4 Hz, 3H), 2.82 – 2.74 (m, 2H), 2.55 (dd, *J* = 9.7, 6.5 Hz, 2H), 2.30 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.5, 128.7, 126.2, 61.6, 45.5, 34.4 ppm.



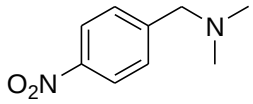
1-(2,4-dichlorophenyl)-N,N-dimethylmethanamine (2g): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (dd, *J* = 6.4, 5.5 Hz, 2H), 7.20 (dd, *J* = 8.3, 2.1 Hz, 1H), 3.48 (s, 2H), 2.27 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 135.3, 135.0, 133.2, 131.7, 129.3,127.0,60.2,45.5ppm.



N-benzyl-N-isopropylpropan-2-amine (2h): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (d, J = 7.2 Hz, 2H), 7.19 (dd, J = 15.8, 8.1 Hz, 2H), 7.11 (d, J = 7.2 Hz, 1H), 3.57 (s, 2H), 2.99 – 2.91 (m, 2H), 0.95 (d, J = 6.6 Hz, 12H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.3, 128.1, 126.3, 49.1, 48.0, 29.9, 20.9 ppm.



N,N-dimethyl-1-phenylmethanamine (2i): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (d, J = 4.5 Hz, 4H), 7.19 (s, 1H), 3.42 (s, 2H), 2.23 (d, J = 10.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 129.4, 128.5, 127.4, 64.2, 45.1, 29.8 ppm.

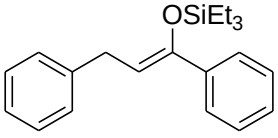


N,N-dimethyl-1-(4-nitrophenyl)methanamine (2j): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (t, J = 13.3 Hz, 2H), 6.55 (d, J = 8.5 Hz, 2H), 4.03 (s, 2H), 2.96 (s, 6H).

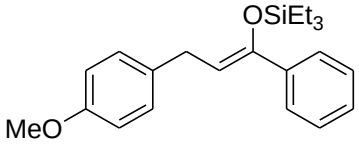
3. General procedure for the hydrosilylation reaction of chalcones

The triethylsilane (0.17 mL, 1.5 mmol, 3.0 equiv.), Pt@G@Si (10.2 mg, 25 μmol, 5 mol%), and chalcone (104 mg, 0.5 mmol, 1.0 equiv.) was dissolved in anhydrous THF (1.0 mL). And then the reaction mixture was heated to 60 ℃ monitored by TLC until total conversion of the starting materials (about 3 hours).The reaction mixture was cooled to room temperature. After filtration under diminished pressure, the organic layer was diluted with 10.0 mL EtOAc, and aqueous layer was extracted with EtOAc

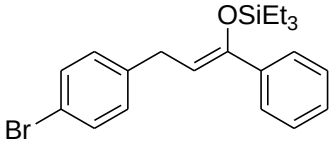
(5.0 mL×2). The combined organic layer was dried over Na_2SO_4 and concentrated. The purification by silica gel flash chromatography (using petroleum : EtOAc = 10:1) to give the desired products with good yields. All the products are confirmed by MS, and usual spectral methods (^{1}H-NMR, ^{13}C-NMR).



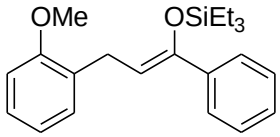
1,3-diphenylprop-1-enyloxy)triethylsilane (5a): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, J = 6.9 Hz, 2H), 7.19 (t, J = 5.2 Hz, 3H), 7.17 – 7.15 (m, 3H), 7.15 – 7.05 (m, 2H), 5.22 (t, J = 7.2 Hz, 1H), 3.50 (d, J = 7.2 Hz, 2H), 0.85 (t, J = 7.9 Hz, 9H), 0.54 (q, J = 7.9 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.3, 141.7, 139.55, 128.5, 128.1, 127.7, 125.9, 109.8, 77.4, 77.1, 76.8, 32.4, 6.8, 5.5, 5.2. HRMS (ESI): m/z: $[M+H]^+$ calculated for $C_{21}H_{29}OSi$: 325.1982, Found: 325.1971.



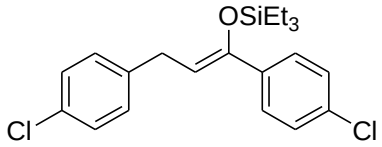
3-(4-methoxyphenyl)-1-phenylprop-1-enyloxy)triethylsilane (5b): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, J = 7.7 Hz, 2H), 7.25 (t, J = 7.5 Hz, 2H), 7.23 (s, 1H), 7.16 (d, J = 7.8 Hz, 2H), 6.80 (d, J = 7.3 Hz, 2H), 5.29 (t, J = 7.1 Hz, 1H), 3.70 (s, 4H), 3.53 (d, J = 7.1 Hz, 2H), 0.94 (t, J = 8.0 Hz, 9H), 0.63 (q, J = 7.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.1, 150.1, 139.7, 133.7, 129.4, 128.1, 125.8, 113.9, 110.3, 77.6, 77.3, 76.9, 55.2, 31.5, 6.9, 5.6, 5.2. HRMS(ESI): m/z: $[M+H]^+$ calculated for $C_{22}H_{31}O_2Si$: 355.2088, Found:355.2077.



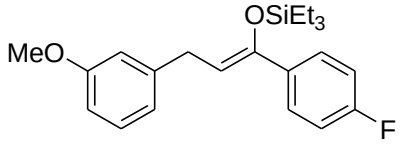
3-(4-bromophenyl)-1-phenylprop-1-enyloxy)triethylsilane (5c): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, J = 7.6 Hz, 2H), 7.28 (d, J = 7.3 Hz, 2H), 7.18 (s, 3H), 7.02 (d, J = 7.6 Hz, 2H), 5.14 (s, 1H), 3.42 (d, J = 7.2 Hz, 2H), 0.83 (t, J = 7.8 Hz, 9H), 0.53 (t, J = 7.8 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.8, 140.6, 139.4, 131.4, 130.2, 128.1, 127.9, 125.8, 119.6, 77.4, 77.1, 76.8, 31.7, 6.8, 6.5, 5.5. HRMS(ESI): m/z: $[M+H]^+$ calculated for $C_{22}H_{30}BrO_2Si$: 433.1193, Found: 435.1175.



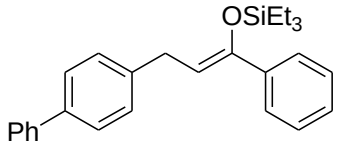
3-(2-methoxyphenyl)-1-phenylprop-1-enyloxy)triethylsilane (5d): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, J = 8.0 Hz, 2H), 7.48 – 7.42 (m, 1H), 7.39 (d, J = 8.6 Hz, 2H), 7.37 – 7.26 (m, 2H), 7.03 (t, J = 7.4 Hz, 1H), 6.97 (d, J = 8.3 Hz, 1H), 5.50 (t, J = 7.1 Hz, 1H), 3.95 (s, 3H), 3.74 (d, J = 7.1 Hz, 2H), 1.10 (t, J = 7.9 Hz, 9H), 0.79 (q, J = 7.9 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.5, 150.1, 139.8, 129.6, 128.0, 127.5, 127.1, 125.8, 120.6, 110.3, 109.5, 77.5, 77.2, 76.8, 55.4, 26.4, 6.8, 5.6, 5.2.^{29}Si NMR (99 MHz, $CDCl_3$) δ 20.82, 20.13. HRMS(ESI): m/z: $[M+H]^+$ calculated for $C_{22}H_{31}O_2Si$: 355.208, Found: 355.2077.



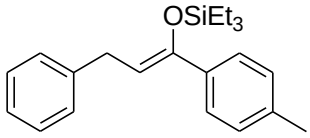
1,3-bis(4-chlorophenyl)prop-1-enyloxy)triethylsilane (5e): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (s, 2H), 7.27 (s, 2H), 7.23 (d, J = 8.8 Hz, 2H), 7.16 (s, 2H), 5.23 (s, 1H), 3.52 (d, J = 7.2 Hz, 2H), 0.93 (t, J = 7.9 Hz, 9H), 0.63 (t, J = 7.9 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.7, 139.8, 137.9, 133.6, 129.7, 128.5, 128.3, 127.0, 109.6, 77.4, 77.0, 76.7, 31.6, 6.7, 5.5, 5.1. ^{29}Si NMR (99 MHz, $CDCl_3$) δ 21.66. HRMS(ESI): m/z: $[M+H]^+$ calculated for $C_{21}H_{27}Cl_2OSi$: 393.1203, Found: 393.1198.



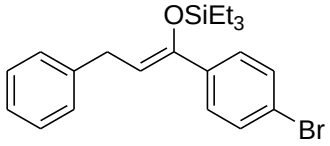
1-(4-fluorophenyl)-3-(3-methoxyphenyl)prop-1-enyloxy)triethylsilane (5f): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (dd, J = 8.8, 5.5 Hz, 2H), 7.07 (t, J = 7.9 Hz, 1H), 6.91 – 6.80 (m, 2H), 6.76 – 6.63 (m, 2H), 6.63 – 6.58 (m, 1H), 5.12 (dd, J = 9.9, 4.4 Hz, 1H), 3.64 (s, 3H), 3.44 (d, J = 7.2 Hz, 2H), 0.83 (t, J = 8.0 Hz, 9H), 0.52 (dd, J = 15.6, 7.7 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.9, 149.4, 143.2, 129.4, 127.5 (d, J = 8.0 Hz), 120.9, 115.0, 114.8, 114.3, 111.3, 77.5, 77.1, 76.8, 55.1, 32.4, 6.8, 5.5, 5.1. ^{29}Si NMR (99 MHz, $CDCl_3$) δ 21.18, 20.33.^{19}F NMR (471 MHz, $CDCl_3$) δ -113.26, -114.29. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{22}H_{29}FNaO_2Si$: 395.1813, Found: 395.1803.



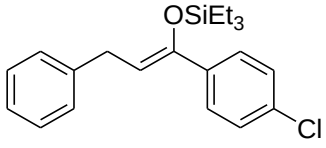
3-([1,1'-biphenyl]-4-yl)-1-phenylprop-1-en-1-yl)oxy)triethylsilane (5g): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 8.0 Hz, 2H), 7.78 (d, J = 7.9 Hz, 4H), 7.62 (dd, J = 18.7, 8.1 Hz, 4H), 7.56 – 7.44 (m, 4H), 5.61 (dt, J = 7.0, 3.5 Hz, 1H), 3.91 (d, J = 7.1 Hz, 2H), 1.24 (t, J = 8.0 Hz, 9H), 0.93 (q, J = 7.9 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.6, 141.4, 140.9, 139.7, 139.1, 129.0 (d, J = 15.5 Hz), 128.2, 127.9, 127.3 (d, J = 15.4 Hz), 126.0, 109.8, 77.6, 77.3, 77.0, 32.2, 7.0, 5.8, 5.4. HRMS(ESI): m/z: $[M+K]^+$ calculated for $C_{27}H_{32}KOSi$: 439.2080, Found: 439.2084.



3-phenyl-1-p-tolylprop-1-enyloxy)triethylsilane (5h): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 – 7.58 (m, 2H), 7.58 – 7.49 (m, 4H), 7.46 (dd, J = 12.1, 5.4 Hz, 2H), 7.33 (d, J = 7.4 Hz, 1H), 5.62 (dd, J = 7.1, 5.5 Hz, 1H), 3.91 (d, J = 6.9 Hz, 2H), 2.61 (s, 3H), 1.27 (dd, J = 8.5, 7.2 Hz, 9H), 1.02 – 0.90 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.6, 141.9, 137.6, 128.6 (d, J = 6.5 Hz), 128.2, 126.7, 126.0, 123.2, 109.7, 77.6, 77.3, 77.0, 32.6, 21.6, 7.0, 5.8, 5.4. HRMS(ESI): m/z: $[M+H]^+$ calculated for $C_{22}H_{31}OSi$: 339.2139, Found: 339.2143.

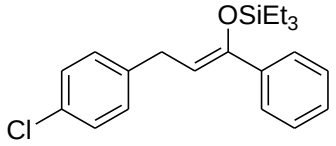


1-(4-bromophenyl)-3-phenylprop-1-enyloxy)triethylsilane (5i): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, J = 8.5 Hz, 2H), 7.25 (d, J = 8.5 Hz, 2H), 7.21 – 7.16 (m, 2H), 7.14 (d, J = 6.8 Hz, 2H), 7.08 (d, J = 6.8 Hz, 1H), 5.21 (t, J = 7.2 Hz, 1H), 3.47 (d, J = 7.2 Hz, 2H), 0.85 (t, J = 7.9 Hz, 9H), 0.54 (q, J = 8.0 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.3, 141.3, 138.5, 131.2, 128.5 (d, J = 3.1 Hz), 127.3, 126.0, 110.5, 77.4, 77.1, 76.8, 32.4, 6.8, 5.5, 5.1.^{29}SiNMR (99 MHz, $CDCl_3$) δ 21.52. HRMS(ESI): m/z: $[M+H]^+$ calculated for $C_{21}H_{27}BrNSi$: 400.1091, Found: 403.1059.

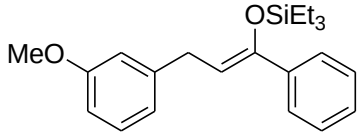


1-(4-chlorophenyl)-3-phenylprop-1-enyloxy)triethylsilane (5j): ^{1}H NMR (400 MHz, $CDCl_3$)^{1}H NMR (400 MHz,

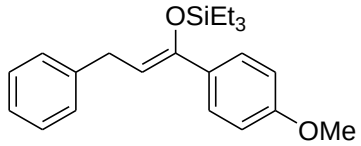
$CDCl_3$) δ 7.60 – 7.50 (m, 2H), 7.42 (dd, J = 14.6, 7.8 Hz, 5H), 7.39 – 7.27 (m, 2H), 5.91 – 5.00 (m, 1H), 3.75 (d, J = 7.1 Hz, 2H), 1.12 (t, J = 7.9 Hz, 9H), 0.81 (dd, J = 15.5, 7.7 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.3, 141.4, 138.1, 133.5, 129.7, 128.6 – 128.2 (m), 127.1, 126.0, 110.4, 77.5, 77.2, 76.9, 32.5, 6.8, 5.6, 5.2. ^{29}SiNMR (99 MHz, $CDCl_3$) δ 21.38. HRMS(ESI): m/z: $[M+H]^+$ calculated for $C_{21}H_{28}ClOSi$: 359.1592, Found: 359.1582.



3-(4-chlorophenyl)-1-phenylprop-1-enyloxy)triethylsilane (5k): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.38 (m, 2H), 7.28 (t, J = 7.1 Hz, 2H), 7.23 (dd, J = 7.6, 4.7 Hz, 3H), 7.16 (d, J = 8.5 Hz, 2H), 5.24 (t, J = 7.2 Hz, 1H), 3.53 (d, J = 7.2 Hz, 2H), 0.93 (t, J = 7.9 Hz, 9H), 0.61 (q, J = 7.9 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.8, 140.1, 139.4, 129.8, 128.5, 128.1, 127.9, 125.8, 109.0, 77.4, 77.1, 76.8, 6.8, 5.5, 5.2. HRMS(ESI): m/z: $[M+H]^+$ calculated for $C_{21}H_{28}ClOSi$: 359.1592, Found: 359.1582.

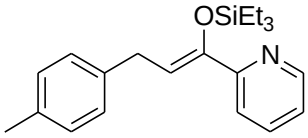


3-(3-methoxyphenyl)-1-phenylprop-1-enyloxy)triethylsilane (5L): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, J = 6.7 Hz, 2H), 7.43 (s, 1H), 7.42 – 7.35 (m, 2H), 7.34 (d, J = 8.6 Hz, 1H), 6.99 (d, J = 13.9 Hz, 2H), 6.86 (d, J = 8.0 Hz, 1H), 5.46 (t, J = 6.9 Hz, 1H), 3.89 (s, 3H), 3.72 (d, J = 6.8 Hz, 2H), 1.09 (t, J = 7.8 Hz, 9H), 0.78 (q, J = 7.5 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.9, 150.4, 143.3, 139.6, 129.4, 128.1, 125.8, 120.9, 114.3, 111.3, 109.7, 77.4, 77.5, 76.8, 55.1, 32.4, 6.8, 5.6. HRMS(ESI): m/z: $[M+H]^+$ calculated for $C_{22}H_{31}O_2Si$: 355.2088, Found: 355.2097.

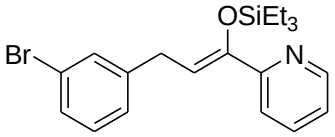


1-(4-methoxyphenyl)-3-phenylprop-1-enyloxy)triethylsilane (5m): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, J = 8.6 Hz, 2H), 7.20 – 7.10 (m, 4H), 7.10 – 7.03 (m, 1H), 6.72 (d, J = 8.6 Hz, 2H), 5.11 (t, J = 7.1 Hz, 1H), 3.67 (s, 3H), 3.47 (d, J = 7.1 Hz, 2H), 0.85 (t, J = 8.0 Hz, 9H), 0.54 (q, J = 7.9 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.4, 150.0, 141.9, 132.3, 128.4 (d, J = 7.5 Hz), 127.1, 125.8, 113.4, 108.3, 77.4,

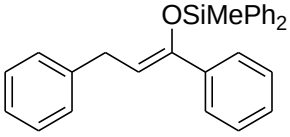
77.1, 76.8, 55.2, 32.3, 6.8, 5.5, 5.2. HRMS(ESI): m/z: $[M+H]^+$ calculated for $C_{22}H_{31}O_2Si$: 355.2088, Found: 355.2077.



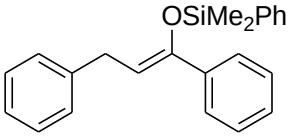
2-(3-(p-tolyl)-1-((triethylsilyl)oxy)prop-1-en-1-yl)pyridine (5n): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 – 8.34 (m, 1H), 7.44 (td, J = 7.8, 1.8 Hz, 1H), 7.37 (d, J = 8.0 Hz, 1H), 7.05 (d, J = 8.0 Hz, 2H), 6.95 (d, J = 8.1 Hz, 3H), 5.93 (t, J = 7.4 Hz, 1H), 3.49 (d, J = 7.4 Hz, 2H), 2.17 (s, 3H), 0.87 (t, J = 7.9 Hz, 9H), 0.61 (q, J = 7.9 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.2, 148.8, 138.1, 136.1, 135.3, 129.1, 128.5, 122.2, 119.4, 112.5, 77.6, 77.2, 76.9, 32.0, 21.0, 6.9, 5.8. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{21}H_{29}NNaOSi$: 362.1916, Found: 362.1920.



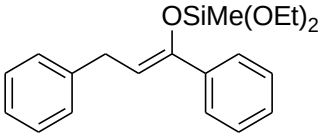
2-(3-(3-bromophenyl)-1-((triethylsilyl)oxy)prop-1-en-1-yl)pyridine (5o): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (ddd, J = 4.8, 1.7, 0.9 Hz, 1H), 7.57 (td, J = 7.8, 1.8 Hz, 1H), 7.43 (d, J = 8.0 Hz, 1H), 7.34 (s, 1H), 7.23 (d, J = 7.8 Hz, 1H), 7.12 (d, J = 7.7 Hz, 1H), 7.07 (d, J = 7.7 Hz, 2H), 5.90 (t, J = 7.4 Hz, 1H), 3.51 (d, J = 7.4 Hz, 2H), 0.90 (t, J = 7.9 Hz, 9H), 0.68 – 0.59 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 155.8, 149.4, 148.8, 143.5, 136.2, 131.6, 129.9, 129.0, 127.2, 122.3, 119.5, 110.9, 77.3, 77.0, 76.70, 31.9, 6.8, 5.7. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{20}H_{26}BrNNaOSi$: 426.0865, Found: 426.0862.



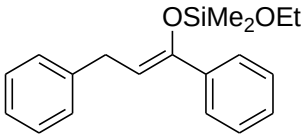
1,3-diphenylprop-1-enyloxy)(methyl)diphenylsilane (5p): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (dd, J = 7.8, 1.3 Hz, 5H), 7.34 – 7.27 (m, 5H), 7.23 (t, J = 7.1 Hz, 4H), 7.09 (dd, J = 8.5, 5.3 Hz, 4H), 6.94 (d, J = 7.2 Hz, 2H), 5.25 (t, J = 7.2 Hz, 1H), 3.27 (d, J = 7.2 Hz, 2H), 0.46 (d, J = 10.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.7, 143.5, 140.8, 137.7, 136.6 (d, J = 10.8 Hz), 136.6 (d, J = 10.8 Hz), 136.2, 132.2, 131.9, 130.5 (d, J = 11.1 Hz), 130.0 (dd, J = 16.7, 13.3 Hz), 128.2, 127.9, 79.5, 79.2, 78.9, 34.6, 20.6, -0.0. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{28}H_{26}NaOSi$: 429.1650, Found: 429.1652.



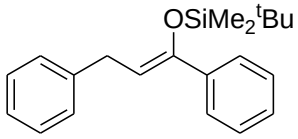
1,3-diphenylprop-1-enyloxy)dimethyl(phenyl)silane (5q): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 – 7.55 (m, 2H), 7.43 (d, J = 1.5 Hz, 2H), 7.38 (d, J = 7.2 Hz, 1H), 7.35 (d, J = 7.3 Hz, 2H), 7.26 (s, 1H), 7.23 (dd, J = 4.2, 3.3 Hz, 4H), 7.17 (d, J = 7.2 Hz, 1H), 7.15 – 7.11 (m, 2H), 5.37 (t, J = 7.2 Hz, 1H), 3.45 (d, J = 7.2 Hz, 2H), 0.38 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.5, 142.3, 139.7, 138.1, 134.3, 133.9, 130.7, 129.2 (d, J = 7.9 Hz), 128.9, 128.5 (d, J = 16.4 Hz), 128.5, 126.6, 111.1, 78.2, 77.9, 77.6, 33.2, 30.6, 0.0. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{23}H_{24}NaOSi$: 401.0682, Found: 401.0680.



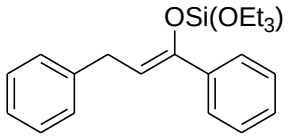
1,3-diphenylprop-1-enyloxy)diethoxy(methyl)silane (5r): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, J = 7.9 Hz, 1H), 7.42 (d, J = 7.6 Hz, 2H), 7.32 (t, J = 7.6 Hz, 1H), 7.15 (d, J = 4.1 Hz, 4H), 7.12 (d, J = 5.8 Hz, 2H), 5.33 (t, J = 7.2 Hz, 1H), 3.68 (d, J = 7.0 Hz, 4H), 3.50 (dd, J = 15.5, 7.2 Hz, 2H), 1.04 (t, J = 7.0 Hz, 6H), -0.00 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.2, 141.4, 128.5, 128.4, 128.1, 127.7, 125.8, 125.4, 110.2, 77.3, 77.0, 76.7, 58.7, 32.2, 29.7, 18.1, -6.5. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{20}H_{26}NaO_3Si$: 365.1543, Found: 365.1543.



1,3-diphenylprop-1-enyloxy)(ethoxy)dimethylsilane (5s): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 – 7.75 (m, 1H), 7.39 – 7.33 (m, 2H), 7.26 (t, J = 7.6 Hz, 1H), 7.12 – 7.10 (m, 4H), 7.08 (d, J = 7.1 Hz, 2H), 5.29 (t, J = 7.2 Hz, 1H), 3.57 (q, J = 7.0 Hz, 2H), 3.45 (d, J = 7.2 Hz, 2H), 0.99 (t, J = 7.0 Hz, 3H), -0.00 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.5, 151.3, 143.7, 140.9, 130.8 (dd, J = 14.3, 5.8 Hz), 130.4 (d, J = 6.7 Hz), 130.0, 127.8, 112.3, 79.7, 79.4, 79.1, 60.9, 34.6, 32.5, 20.6, 0.0. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{19}H_{24}NaO_2Si$: 335.1443, Found: 335.1442.



1,3-diphenylprop-1-enyloxy)(isopropyl)dimethylsilane (5t): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, J = 7.1 Hz, 2H), 7.27 (d, J = 4.3 Hz, 5H), 7.22 (dd, J = 14.5, 7.4 Hz, 2H), 7.17 (dd, J = 8.8, 4.5 Hz, 1H), 5.31 (t, J = 7.2 Hz, 1H), 3.59 (d, J = 7.2 Hz, 2H), 1.02 (s, 7H), 0.00 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.2, 141.7, 139.7, 128.6 (d, J = 6.4 Hz), 128.1, 127.8, 126.2, 126.0, 110.5, 77.5, 77.2, 76.9, 32.5, 26.1, 18.5, -3.7. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{20}H_{26}NaOSi$: 333.1654, Found: 333.1652.

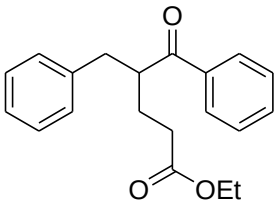


1,3-diphenylprop-1-enyloxy)triethoxysilane (5u): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, J = 7.4 Hz, 2H), 7.23 – 7.20 (m, 6H), 7.18 (d, J = 6.8 Hz, 2H), 5.39 (t, J = 7.2 Hz, 1H), 3.75 (q, J = 7.0 Hz, 6H), 3.60 (d, J = 7.2 Hz, 2H), 1.09 (t, J = 7.0 Hz, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.8, 141.5, 138.0, 128.6, 128.3, 128.0, 127.7, 125.4, 110.4, 77.4, 77.0, 76.7, 59.5, 32.1, 18.0. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{21}H_{29}NaO_4Si$: 395.1655, Found: 395.1650.

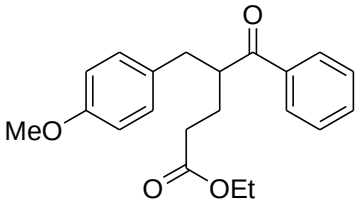
4. General Procedure for the conjugate addition of silyl enolate to ethyl acrylate:

To a solution of 1,3-diphenylprop-1-enyloxy)triethylsilane (1.0 mmol, l.0 equiv.) in anhydrous THF (1 mL), tetrabutylammonium fluoride (1M in THF, 0.1 mL, 10 mol%) was added dropwise under argon atmosphere, followed by addition of ethyl acrylate (0.16 mL, 1.0 mmol, 1.0 equiv.). The resulting solution was stirred for 4 h at 60 ℃. The reaction was detected with TLC, and the mixture was cooled to room temperature, filtration under diminished pressure. The organic layer was diluted with 10 mL EtOAc and water layer was extracted with EtOAc (5 mL × 2). The combined organic layer was dried over Na_2SO_4 and concentrated. After purification by silica gel flash chromatography (using petroleum: EtOAc = 4:1) to give moderate with good

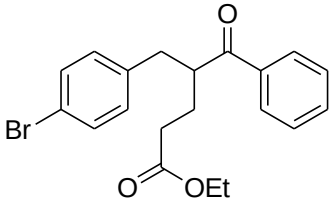
yield.



ethyl-4-benzyl-5-oxo-5-phenylpentanoate (7a): ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.89 – 7.84 (m, 2H), 7.64 – 7.57 (m, 1H), 7.51 (t, J = 7.4 Hz, 1H), 7.40 (t, J = 7.7 Hz, 2H), 7.20 (d, J = 7.2 Hz, 2H), 7.15 (d, J = 6.9 Hz, 2H), 4.05 (q, J = 7.1 Hz, 2H), 3.89 – 3.80 (m, 1H), 3.10 (dd, J = 13.7, 7.3 Hz, 1H), 2.75 (dd, J = 13.7, 6.8 Hz, 1H), 2.32 (dd, J = 8.4, 6.1 Hz, 1H), 2.18 (ddd, J = 30.1, 11.0, 6.7 Hz, 2H), 1.90 (dd, J = 10.7, 3.9 Hz, 1H), 1.16 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.2, 173.1, 139.3, 134.0, 133.1, 129.0, 128.6 (d, J = 19.0 Hz), 128.3, 127.9, 126.4, 77.4, 77.1, 76.8, 60.4, 47.0, 38.4, 31.8, 27.0, 14.2. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{20}H_{22}NaO_3$: 333.1461, Found: 333.1449.

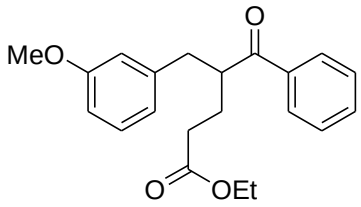


ethyl-4-(4-methoxybenzyl)-5-oxo-5-phenylpentanoate (7b): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, J = 7.3 Hz, 1H), 7.50 (t, J = 7.4 Hz, 1H), 7.44 – 7.20 (m, 2H), 7.07 (d, J = 8.5 Hz, 1H), 6.86 – 6.67 (m, 2H), 4.05 (q, J = 7.1 Hz, 2H), 3.82 (dd, J = 10.0, 4.6 Hz, 1H), 3.70 (s, 3H), 2.99 (ddd, J = 16.4, 13.3, 5.5 Hz, 1H), 2.70 (dd, J = 13.8, 6.7 Hz, 1H), 2.32 (dd, J = 8.9, 6.7 Hz, 1H), 2.22 (dd, J = 15.8, 8.4 Hz, 1H), 2.16 – 1.95 (m, 1H), 1.93 – 1.79 (m, 1H), 1.21 (t, J = 7.1 Hz, 1H), 1.16 (t, J = 7.1 Hz, 2H), 0.87 (t, J = 7.2 Hz, 1H). ^{13}C NMR (101 MHz, CDCl3) δ 203.2, 173.0, 158.1, 137.2, 133.0, 131.2, 129.9, 128.6, 128.2, 113.9, 77.5, 77.2, 76.9, 60.3, 55.1, 47.2, 37.5, 31.7, 26.9, 14.1. HRMS(ESI) : m/z: $[M+Na]^+$ calculated for $C_{21}H_{24}NaO_4$: 363.1567, Found: 363.1555.

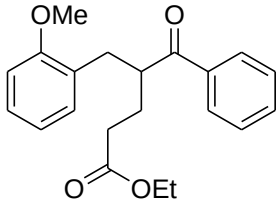


ethyl-4-(4-bromobenzyl)-5-oxo-5-phenylpentanoate (7c): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, J = 7.5 Hz, 2H), 7.48 – 7.41 (m, 1H), 7.35 (t, J = 6.2 Hz, 2H), 7.26 (d, J = 8.2 Hz, 2H), 7.12 (dd, J = 17.9, 7.4 Hz, 1H), 6.97 (d, J = 8.2 Hz, 1H), 4.00 (dd, J = 7.1, 4.3 Hz, 2H), 3.85 – 3.73 (m, 1H), 3.00 (dd, J = 13.7,

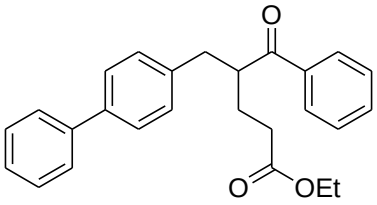
7.7 Hz, 1H), 2.66 (dd, J = 13.9, 6.3 Hz, 1H), 2.34 – 2.23 (m, 1H), 2.23 – 2.11 (m, 1H), 2.11 – 1.98 (m, 1H), 1.82 (dd, J = 10.6, 4.4 Hz, 1H), 1.11 (dd, J = 9.2, 5.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.7, 171.9, 137.4, 132.3, 130.6, 129.8, 128.0, 127.8, 127.3, 119.3, 76.5, 76.2, 75.9, 59.5, 45.7, 36.6, 30.6, 26.2, 13.2. HRMS(ESI) : m/z: $[M+Na]^+$ calculated for $C_{20}H_{21}BrNaO_3$: 411.0566, Found: 411.0556.



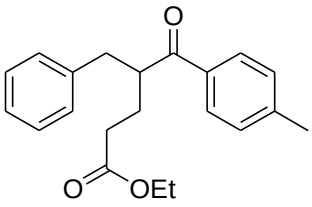
ethyl-4-(3-methoxybenzyl)-5-oxo-5-phenylpentanoate (7d): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, J = 7.4 Hz, 1H), 7.51 (t, J = 7.4 Hz, 1H), 7.40 (t, J = 7.6 Hz, 2H), 7.13 (t, J = 7.8 Hz, 1H), 6.75 (d, J = 7.6 Hz, 1H), 6.72 – 6.62 (m, 2H), 4.05 (q, J = 7.1 Hz, 2H), 3.85 (dd, J = 8.8, 4.3 Hz, 1H), 3.71 (s, 3H), 3.01 (ddd, J = 16.4, 13.2, 5.5 Hz, 1H), 2.80 – 2.68 (m, 1H), 2.32 (dd, J = 8.4, 6.4 Hz, 1H), 2.26 – 2.17 (m, 1H), 2.16 – 2.07 (m, 1H), 1.94 – 1.86 (m, 1H), 1.21 (t, J = 7.1 Hz, 1H), 1.17 (t, J = 7.2 Hz, 2H), 0.87 (t, J = 7.7 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.0, 173.0, 159.6, 140.8, 137.2, 133.1, 129.4, 128.6, 128.2, 121.3, 114.8, 111.7, 77.5, 77.1, 76.8, 60.3, 55.1, 46.8, 38.4, 31.7, 27.0, 14.1. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{21}H_{24}NaO_4$: 363.1567, Found: 363.1559.



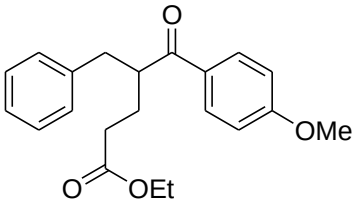
ethyl-4-(2-methoxybenzyl)-5-oxo-5-phenylpentanoate (7e): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, J = 7.4 Hz, 2H), 7.49 (t, J = 7.3 Hz, 1H), 7.39 (t, J = 7.6 Hz, 2H), 7.12 (dd, J = 11.1, 4.5 Hz, 1H), 6.98 (dd, J = 72.1, 7.2 Hz, 1H), 6.82 – 6.75 (m, 2H), 4.03 (q, J = 7.1 Hz, 2H), 3.79 (s, 3H), 3.61 (d, J = 7.7 Hz, 1H), 2.99 (ddd, J = 16.3, 13.0, 4.9 Hz, 1H), 2.72 (dd, J = 13.3, 7.5 Hz, 1H), 2.34 (dd, J = 12.2, 6.6 Hz, 1H), 2.25 – 2.10 (m, 2H), 1.89 – 1.77 (m, 1H), 1.20 (t, J = 7.1 Hz, 1H), 1.15 (t, J = 7.1 Hz, 3H), 1.07 (t, J = 7.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.3, 173.1, 157.4, 137.2, 132.9, 131.2, 128.5, 128.3, 127.8, 127.5, 120.4, 110.2, 77.5, 77.2, 76.9, 60.2, 55.1, 45.0, 34.0, 32.0, 26.6, 14.1. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{21}H_{24}NaO_4$: 363.1567, Found: 363.1559.



ethyl-4-(4-benzylbenzyl)-5-oxo-5-phenylpentanoate (7f): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, J = 7.6 Hz, 2H), 7.53 (d, J = 7.5 Hz, 3H), 7.48 – 7.37 (m, 6H), 7.31 (t, J = 7.3 Hz, 1H), 7.26 – 7.21 (m, 2H), 4.07 (q, J = 7.1 Hz, 2H), 3.15 (dd, J = 13.7, 7.3 Hz, 1H), 2.80 (dd, J = 13.7, 6.7 Hz, 1H), 2.36 (dd, J = 8.3, 6.3 Hz, 1H), 2.25 (dd, J = 15.8, 8.3 Hz, 1H), 2.19 – 2.09 (m, 1H), 1.98 – 1.87 (m, 1H), 1.18 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.2, 172.9, 140.9, 139.3, 138.4, 137.3, 133.8, 129.0, 128.8, 128.7, 128.4, 127.2, 127.1, 127.0, 77.6, 77.2, 76.9, 60.3, 47.0, 38.4, 31.7, 21.3, 14.2. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{26}H_{26}NaO_3$: 409.1774, Found: 409.1764.

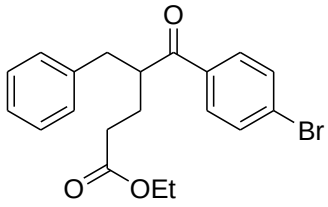


ethyl-4-benzyl-5-oxo-5-p-tolylpentanoate (7g): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, J = 6.8 Hz, 2H), 7.20 – 7.12 (m, 2H), 7.12 – 7.06 (m, 2H), 7.04 (d, J = 6.8 Hz, 2H), 7.00 (t, J = 7.1 Hz, 1H), 3.93 (q, J = 7.1 Hz, 2H), 3.73 (dd, J = 7.5, 5.6 Hz, 1H), 2.98 (dd, J = 13.7, 7.4 Hz, 1H), 2.64 (dd, J = 13.7, 6.7 Hz, 1H), 2.22 (s, 3H), 2.21 – 2.04 (m, 2H), 2.04 – 1.95 (m, 1H), 1.78 (dd, J = 6.6, 5.0 Hz, 1H), 1.04 (t, J = 7.1 Hz, 3H), 0.99 (t, J = 7.1 Hz, 1H), 0.83 (t, J = 7.9 Hz, 1H), 0.74 (t, J = 7.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.2, 172.9, 139.4, 138.3, 133.8, 129.0, 128.8, 128.5 (d, J = 7.4 Hz), 126.3, 125.5, 77.6, 77.2, 76.9, 60.3, 47.0, 38.4, 31.7, 27.1, 21.3, 14.2. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{21}H_{24}NaO_3$: 347.1618, Found: 347.1615.

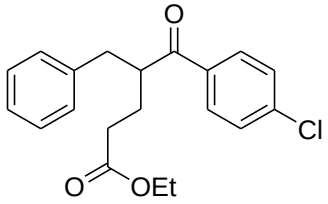


ethyl-4-benzyl-5-(4-methoxyphenyl)-5-oxopentanoat e (7h): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, J = 8.9 Hz, 2H), 7.23 (dd, J = 15.3, 7.9 Hz, 2H), 7.17 – 7.11 (m, 3H), 6.88 (d, J = 8.9 Hz, 2H), 4.06 (q, J = 7.1 Hz, 2H), 3.84 (s, 3H), 3.82 – 3.77 (m, 1H), 3.09 (dd, J = 13.7, 7.4 Hz, 1H), 2.75 (dd, J = 13.7, 6.8 Hz, 1H), 2.31 (dd, J = 8.4, 6.1 Hz, 1H), 2.25 – 2.15 (m, 1H), 2.14 – 2.05 (m, 1H), 1.92 – 1.83 (m, 1H), 1.18 (t, J = 7.1 Hz, 3H), 0.94 (t, J = 7.1 Hz, 1H). ^{13}C NMR (101

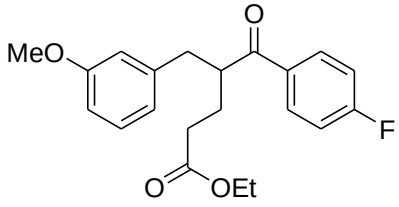
MHz, $CDCl_3$) δ 201.5, 173.1, 163.6, 139.5, 130.6, 130.3, 129.0, 128.4, 126.3, 113.8, 77.4, 77.0, 76.7, 60.3, 55.4, 46.5, 38.5, 31.8, 27.2, 14.12. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{21}H_{24}NaO_4$: 363.1567, Found: 363.1560.



ethyl-4-benzyl-5-(4-bromophenyl)-5-oxopentanoate (7i): ^1H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, J = 8.6 Hz, 2H), 7.44 (d, J = 8.6 Hz, 2H), 7.16 – 7.08 (m, 2H), 7.05 (dd, J = 7.1, 4.9 Hz, 3H), 3.98 (q, J = 7.1 Hz, 2H), 3.75 – 3.65 (m, 1H), 2.98 (dd, J = 13.7, 7.7 Hz, 1H), 2.68 (dd, J = 13.7, 6.4 Hz, 1H), 2.32 – 2.20 (m, 1H), 2.17 – 1.98 (m, 2H), 1.85 – 1.75 (m, 1H), 1.10 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 202.2, 172.9, 139.0, 136.0, 131.9, 129.8, 128.9, 128.5, 128.3, 126.4, 77.4, 77.1, 76.8, 60.4, 47.0, 38.4, 31.6, 27.1, 14.2. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{20}H_{21}BrNaO_3$: 414.0884, Found: 414.0848.

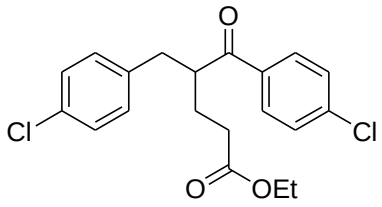


ethyl-4-benzyl-5-(4-chlorophenyl)-5-oxopentanoate (7j): ^1H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, J = 8.6 Hz, 2H), 7.26 (d, J = 8.6 Hz, 2H), 7.14 – 7.08 (m, 2H), 7.07 – 7.00 (m, 3H), 3.97 (q, J = 7.1 Hz, 2H), 3.75 – 3.65 (m, 1H), 2.98 (dd, J = 13.7, 7.7 Hz, 1H), 2.68 (dd, J = 13.7, 6.4 Hz, 1H), 2.31 – 2.20 (m, 1H), 2.17 – 1.98 (m, 2H), 1.85 – 1.75 (m, 1H), 1.09 (t, J = 7.1 Hz, 3H), 1.01 (t, J = 7.1 Hz, 1H), 0.83 (t, J = 7.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 202.0, 172.9, 139.5, 139.1, 135.6, 129.7, 129.0 (d, J = 1.7 Hz), 128.5, 128.1, 126.4, 77.4, 77.1, 76.8, 60.4, 47.0, 38.4, 31.6, 27.1, 14.2. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{20}H_{21}ClNaO_3$: 367.1072, Found: 367.1070.

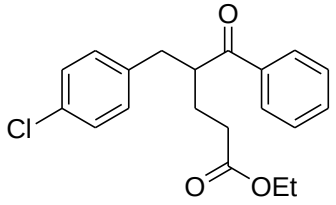


ethyl-4-(3-methoxybenzyl)-5-(4-fluorophenyl)-5-oxopentanoate (7k): ^1H NMR (400 MHz, $CDCl_3$) δ 7.81 (dd, J = 8.8, 5.4 Hz, 2H), 7.03 (t, J = 8.2 Hz, 1H), 6.97 (t, J = 8.6 Hz, 2H), 6.64 (d, J = 7.6 Hz, 1H), 6.59 (d, J = 6.0 Hz, 2H), 3.97 (q, J = 7.1 Hz, 2H), 3.78 – 3.68 (m, 1H), 3.63 (s, 3H),

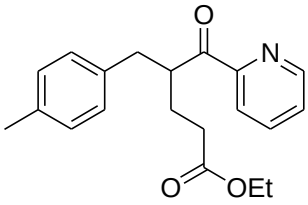
2.96 (dd, J = 13.7, 7.7 Hz, 1H), 2.65 (dd, J = 13.7, 6.5 Hz, 1H), 2.24 (dd, J = 8.0, 6.6 Hz, 1H), 2.08 (ddd, J = 20.2, 15.3, 7.0 Hz, 2H), 1.86 – 1.75 (m, 1H), 1.09 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.5, 172.9, 159.7, 140.7, 133.7, 130.9 (d, J = 9.3 Hz), 129.4, 121.3, 115.8, 115.5, 114.8, 111.7, 77.4, 77.1, 76.8, 60.4, 55.1, 46.8, 38.4, 31.6, 27.2, 14.1. ^{19}F NMR (471 MHz, $CDCl_3$) δ -105.17 (s). HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{21}H_{23}FNaO_4$: 381.1473, Found: 381.1462.



ethyl-4-(4-chlorobenzyl)-5-(4-chlorophenyl)-5-oxopentanoate (7l): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, J = 8.6 Hz, 2H), 7.29 (d, J = 8.6 Hz, 2H), 7.08 (d, J = 8.4 Hz, 2H), 6.98 (d, J = 8.4 Hz, 2H), 3.99 (q, J = 7.1 Hz, 2H), 3.71 (t, J = 5.9 Hz, 1H), 2.96 (dd, J = 13.7, 8.0 Hz, 1H), 2.65 (dd, J = 13.7, 6.1 Hz, 1H), 2.24 (dd, J = 7.6, 6.9 Hz, 1H), 2.18 – 2.08 (m, 1H), 2.05 – 1.94 (m, 1H), 1.83 – 1.72 (m, 1H), 1.10 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.6, 172.8, 139.7, 137.6, 135.3, 132.2, 130.3, 129.6, 129.0, 128.6, 77.4, 77.1, 76.8, 60.5, 46.8, 37.4, 31.4, 27.2, 14.2. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{20}H_{20}Cl_2NaO_3$: 401.0682, Found: 401.0680.

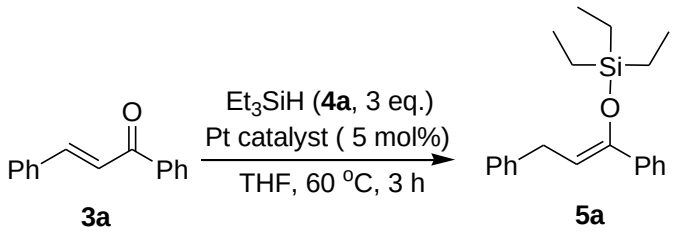


ethyl-4-(4-chlorobenzyl)-5-oxo-5-phenylpentanoate (7m): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, J = 7.2 Hz, 2H), 7.52 (t, J = 7.3 Hz, 1H), 7.41 (t, J = 7.7 Hz, 2H), 7.17 (d, J = 8.4 Hz, 2H), 7.08 (d, J = 8.4 Hz, 2H), 4.06 (q, J = 7.1 Hz, 2H), 3.85 (dd, J = 9.6, 3.8 Hz, 1H), 3.07 (dd, J = 13.7, 7.7 Hz, 1H), 2.74 (dd, J = 13.8, 6.4 Hz, 1H), 2.33 (dd, J = 8.2, 6.4 Hz, 1H), 2.24 (dd, J = 15.7, 8.3 Hz, 1H), 2.11 (dd, J = 13.9, 6.3 Hz, 1H), 1.92 – 1.81 (m, 1H), 1.17 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 202.7, 172.9, 137.8, 137.1, 133.2, 132.1, 130.4, 128.6 (d, J = 17.8 Hz), 128.7, 128.6, 77.5, 77.1, 76.8, 60.4, 46.7, 37.5, 31.6, 27.1, 14.1. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{20}H_{21}ClNaO_3$: 367.1072, Found: 367.1070.



ethyl-4-(4-methylbenzyl)-5-oxo-5-(pyridin-2-yl)pentanoate (7n): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.66 (ddd, J = 4.7, 1.6, 0.9 Hz, 1H), 8.02 – 7.96 (m, 1H), 7.82 – 7.74 (m, 1H), 7.46 – 7.36 (m, 1H), 7.10 (d, J = 8.0 Hz, 2H), 7.02 (d, J = 7.9 Hz, 2H), 4.56 – 4.42 (m, 1H), 4.04 (q, J = 7.1 Hz, 2H), 3.10 (dd, J = 13.7, 6.7 Hz, 1H), 2.69 (dd, J = 13.7, 7.6 Hz, 1H), 2.29 (dd, J = 9.6, 6.0 Hz, 1H), 2.26 (s, 3H), 2.25 – 2.18 (m, 1H), 2.09 (dd, J = 14.2, 5.5 Hz, 1H), 1.93 (dd, J = 4.7, 2.9 Hz, 1H), 1.16 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.9, 173.1, 153.2, 149.0, 136.8, 136.4, 135.5, 129.0 (d, J = 3.8 Hz), 128.9, 127.0, 122.3, 77.4, 77.1, 76.8, 60.2, 45.4, 37.3, 32.1, 26.1, 21.0, 14.1. HRMS(ESI): m/z: $[M+Na]^+$ calculated for $C_{20}H_{23}NNaO_3$: 348.1576, Found: 348.1572.

Table S1. Optimization of the platinum-catalyzed hydrosilylation of chalcone.[a]

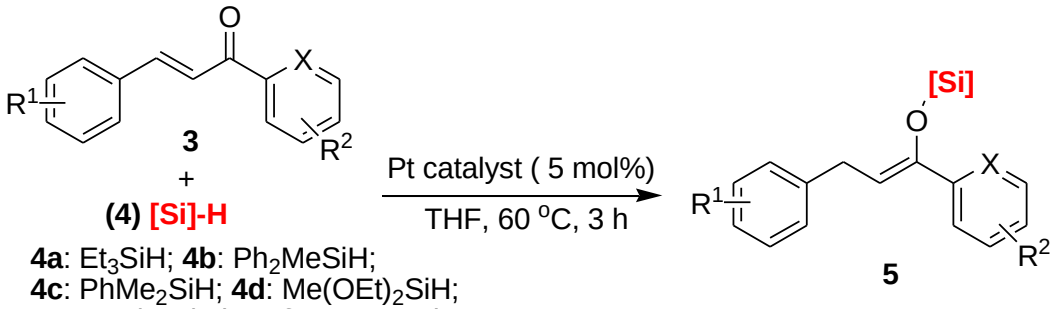


(1) Pt@G@Si, 94% yield, **Z/E** = 91:9;
(2) Pt@Graphite@Si, 56% yield, **Z/E** = 70:30;
(3) Pt@Si (without graphene), <5% yield
(4) H_2PtCl_6, no dersired product
(5) Karstedt catalyst, no dersired product

Entry	Pt catalyst	Temp. (°C)	Solvent	Z/E	Yield (%)[b]
1	Pt@Graphite@Si	RT	THF	ND	35
2	Pt@Graphite@Si	80	THF	ND	45
3	Pt@Graphite@Si	50	THF	ND	40
4	Pt@Graphite@Si	65	THF	67:33	45
5	Pt@Graphite@Si	60	THF	70:30	56
6	Pt@Graphite@Si	60	DCM	44:56	12
7	Pt@Graphite@Si	60	toluene	70:30	35
8	Pt@Graphite@Si	60	EA	85:15	22
9	Pt@Graphite@Si	60	MeCN	29:71	15
10	Pt@Graphite@Si	60	Et_2O	ND	35
11	Pt@Graphite@Si	60	dioxane	ND	30
12[c]	Pt@Graphite@Si	60	THF	ND	40
13[d]	Pt@Graphite@Si	60	THF	ND	30
14[e]	Pt@Graphite@Si	60	THF	ND	42
15	Pt@Si	60	THF	ND	<5
16	H_2PtCl_6	60	THF	ND	0
17	Karstedt catalyst	60	THF	ND	0
18	Pt@G@Si	60	THF	91:9	94

[a] Reactions were performed with chalcone (0.5 mmol), Et_3SiH (1.5 mmol, 3 equiv.), platinum catalyst (5 mol%), solvent (1 mL). Reaction time is 3 h. Pt@G@Si is obtained from graphene-modified PMHS, Pt@Graphite@Si is obtained from graphite-modified PMHS, and Pt@Si is directly recovered from the H_2PtCl_6-catayzed hydrosilylation of amide without graphene or graphite. The other conditions were shown in this table except special note. [b] Isolated yield. [c] With 1 mmol of Et_3SiH (2 equiv.). [d] With 0.5 mmol of Et_3SiH (1 equiv.). [e] With 2 mmol of Et_3SiH (4 equiv.).

Table S2. Pt@G@Si-catalyzed hydrosilylation of chalcones with various hydrosilanes.[a]



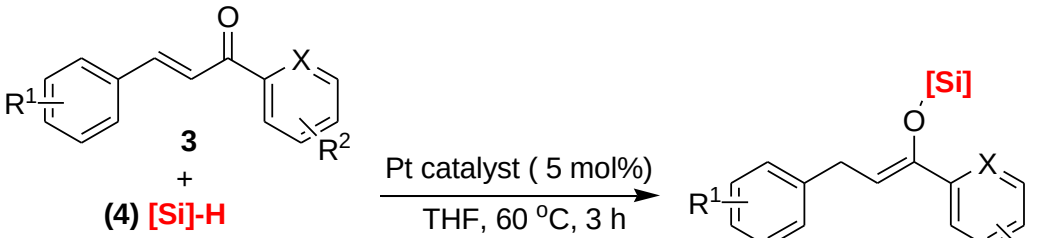
4a: Et_3SiH; **4b**: Ph_2MeSiH;
4c: $PhMe_2SiH$; **4d**: $Me(OEt)_2SiH$;
4e: $Me_2(OEt)SiH$; **4f**: *t*-$BuMe_2SiH$;
4g: $HSi(OEt)_3$;

Entry[a]	R^1/ R^2	[Si]-H	Ratio of Z/E[b]	Yield (%)[c]
1	H/ H	4a	91:9	5a: 94
2	4-OMe/ H	4a	79:21	5b: 83
3	4-Br/ H	4a	96:4	5c: 86
4	2-OMe/ H	4a	88:12	5d: 85
5	4-Cl/4-Cl	4a	80:20	5e: 89
6	3-OMe/4-F	4a	75:25	5f: 90
7	4-Ph/ H	4a	80:20	5g: 88
8	H/4-Me	4a	78:22	5h: 90
9	H/4-Br	4a	93:7	5i: 88
10	H/4-Cl	4a	82:18	5j: 91
11	4-Cl/ H	4a	89:11	5k: 90
12	3-OMe/ H	4a	77:23	5l: 89
13	H/4-OMe	4a	85:15	5m: 84
14	4-Me/ H (X = N)	4a	97:3	5n: 89
15	3-Br/ H (X = N)	4a	99:1	5o: 84
16	H/H	4b	90:10	5p: 76
17	H/H	4c	90:10	5q: 71
18	H/H	4d	91:9	5r: 81

19	**H/H**	**4e**	**90:10**	**5s: 73**
20	**H/H**	**4f**	**90:10**	**5t: 78**
21	**H/H**	**4g**	**99:1**	**5u: 83**

[a] Reactions were performed with chalcone (0.5 mmol), hydrosilane ([Si]-H, 1.5 mmol), Pt@G@Si catalyst (5 mol%), THF (1 mL). [b] The ratio of Z/E is determined by ^{1}H-NMR. [c] Isolated yield.

Table S3. (PPh3)2PtCl2 catalyzed hydrosilylation of chalcones with various hydrosilanes.[a]



4a: Et_3SiH; **4b**: Ph_2MeSiH;
4c: $PhMe_2SiH$; **4d**: $Me(OEt)_2SiH$;
4e: $Me_2(OEt)SiH$; **4f**: *t*-$BuMe_2SiH$;
4g: $HSi(OEt)_3$;

Entry[a]	R^1/R^2	[Si]-H	Ratio of Z/E[b]	Yield (%)[c]
1	H/H	4a	98:2	94
2	4-OMe/H	4a	95:5	87
3	4-Br/H	4a	96:4	85
4	2-OMe/H	4a	95:5	88
5	4-Cl/4-Cl	4a	99:1	86
6	3-OMe/4-F	4a	99:1	91
7	4-Ph/H	4a	97:3	86
8	H/4-Me	4a	>99:1	83
9	H/4-Br	4a	97:3	82
10	H/4-Cl	4a	99:1	89
11	4-Cl/H	4a	92:8	84
12	3-OMe/H	4a	95:5	84
13	H/4-OMe	4a	97:3	86
14	4-Me/ H (X = N)	4a	98:2	85
15	3-Br/ H (X = N)	4a	99:1	81
16	H/H	4b	97:3	81
17	H/H	4c	99:1	87
18	H/H	4f	99:1	83

[a] Reactions were performed with chalcone (0.5 mmol), hydrosilane ([Si]-H, 1.5 mmol), $(PPh_3)_2PtCl_2$ catalyst (5 mol%), THF (1 mL). [b] The ratio of Z/E is determined by ^{1}H-NMR. [c] Isolated yield.

Table S3. ICP-MS analysis of Pt@G@Si and other platinum catalysts.

Analyte	Mass	Intensity	RSD	Platinum/ppb
1	195	22846.6	0.6	4885830.60
2	195	16955.1	1.9	7310643.78
3	195	107898.6	7.8	46556429.15
4	195	10571.2	1.5	2350901.51

1. Pt@Graphite@Si (Pt:1 mol%)
2. Pt@G@Si (Pt:1 mol%)
3. Pt@G@Si (Pt:5 mol%)
4. Pt@G@Si (Pt:5 mol%, after recycling the first time).

5. Figure S1-S14: SEM, TEM, Raman, IR, XRD, XPS and ICP-MS spectra of Pt@G@Si and other platinum catalysts

Figure S1. SEM image of Pt@G@Si.

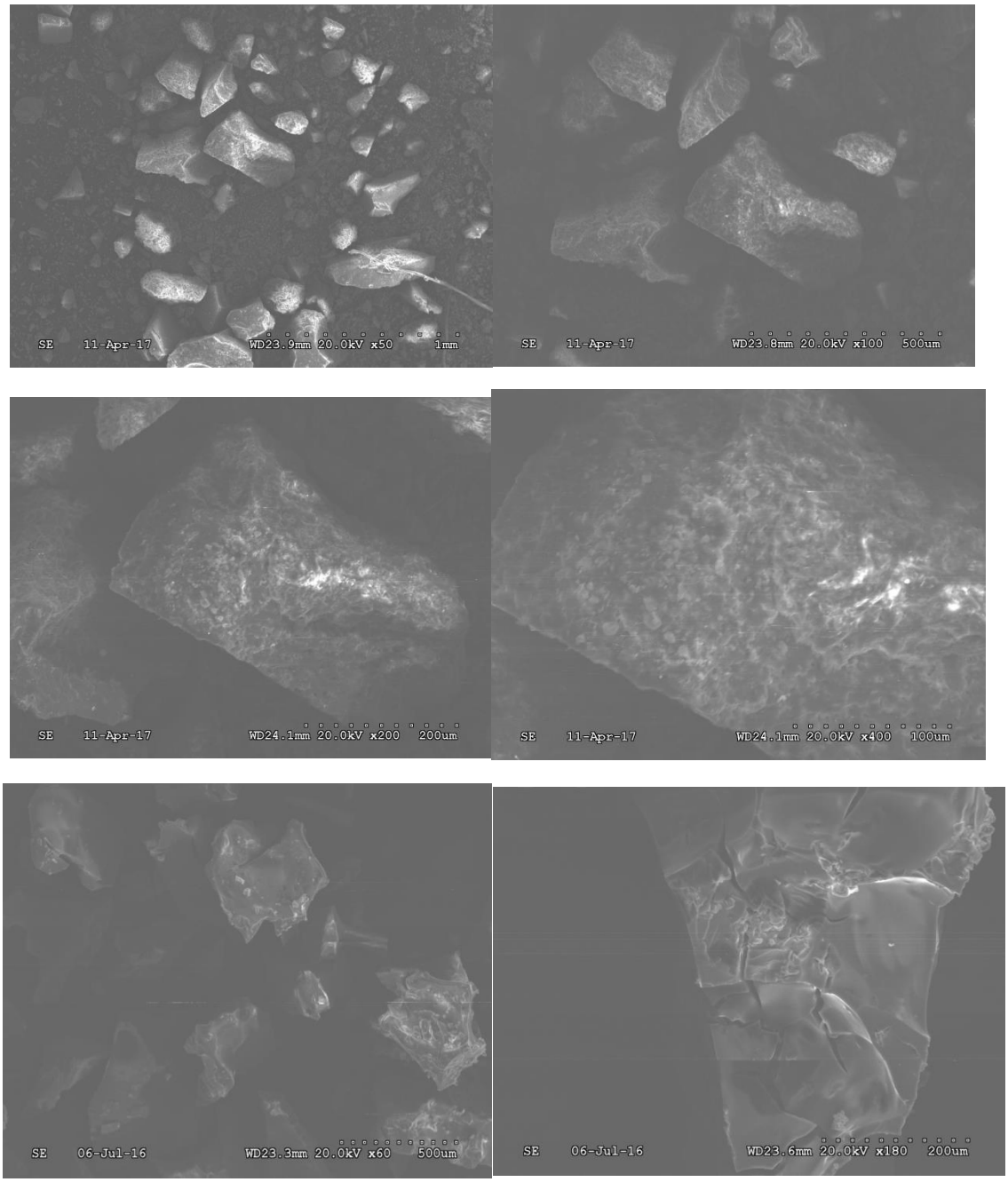


Figure S2. TEM image of Pt@G@Si.

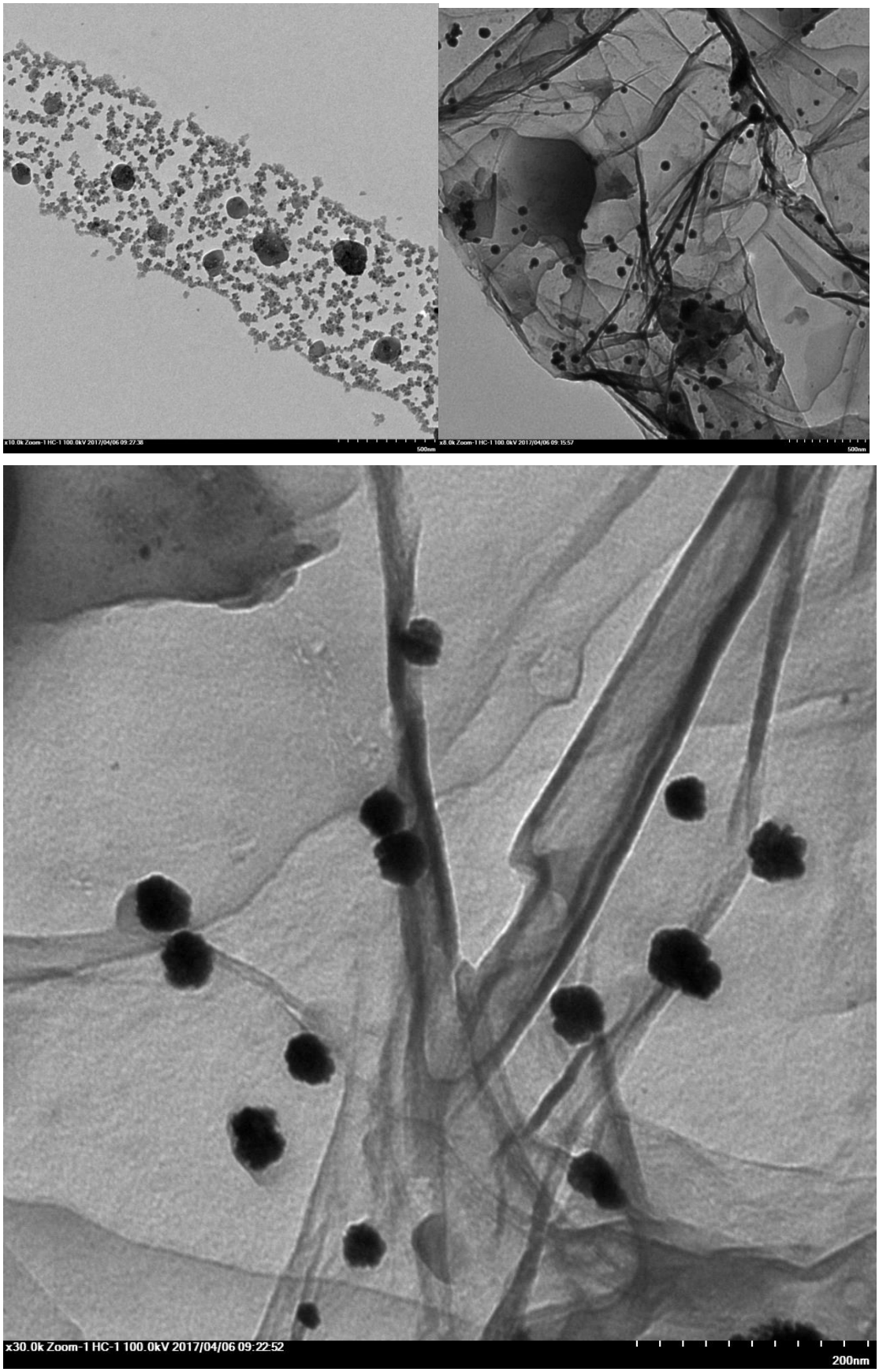


Figure S3. SEM image of PMHS@graphene.

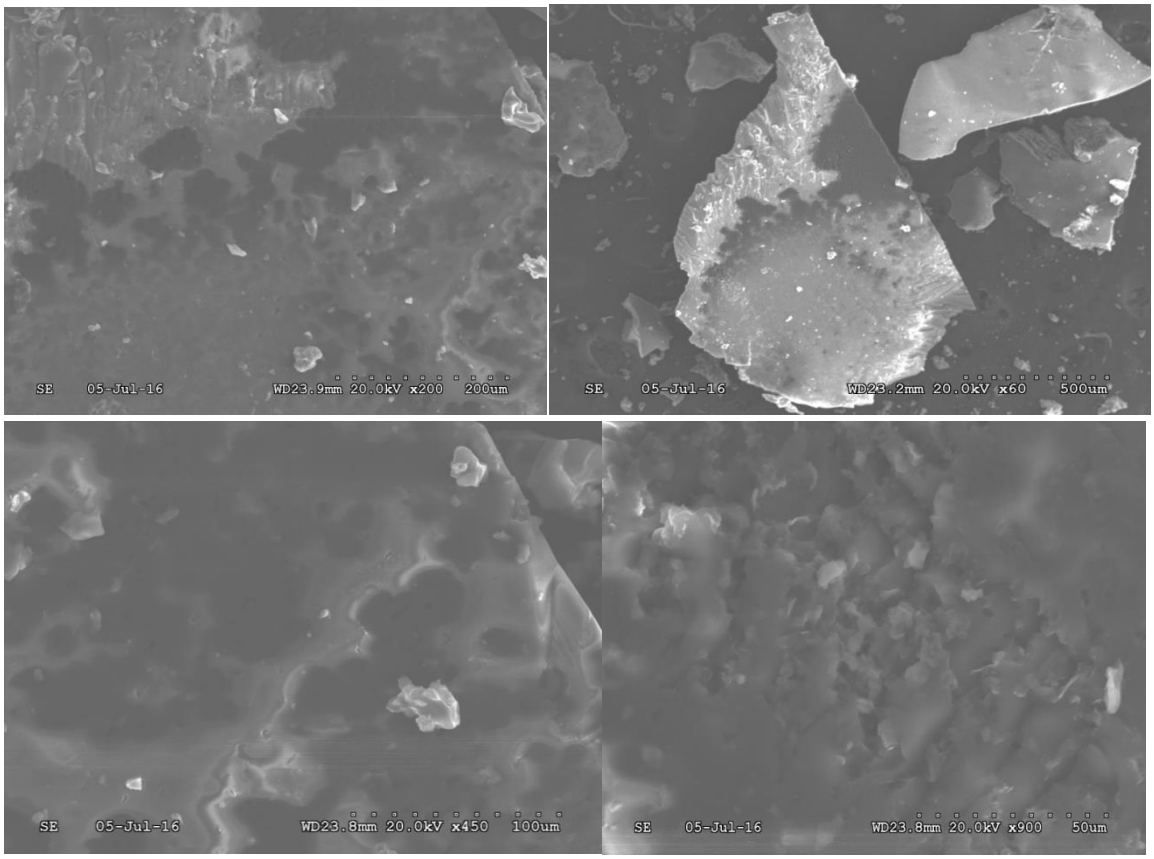


Figure S4. SEM image of PMHS@graphite.

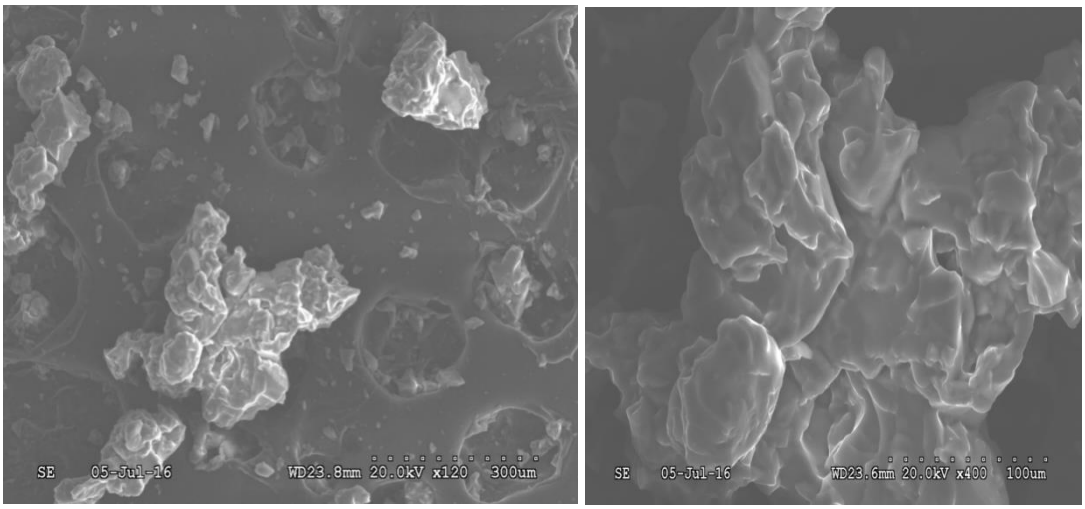


Figure S5. SEM image of Pt@Graphite@Si.

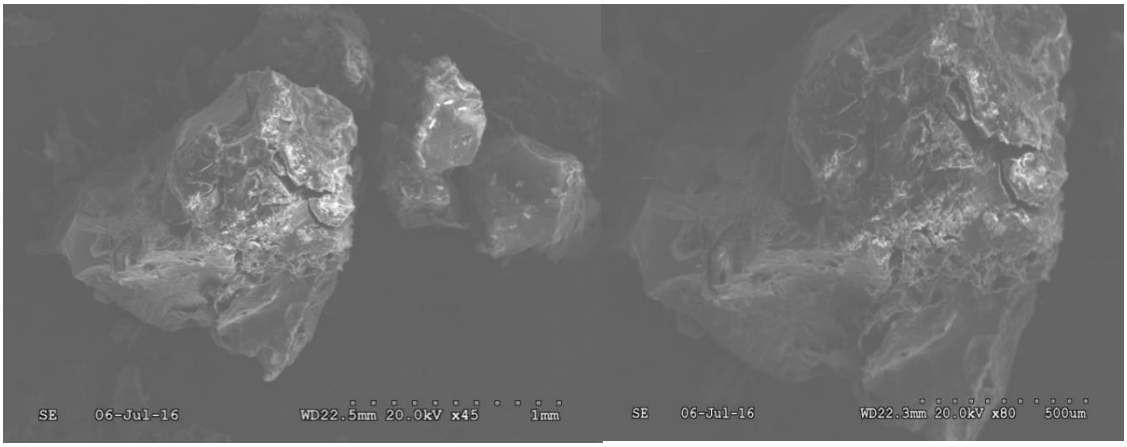


Figure S6. TEM image of Pt@Graphite@Si

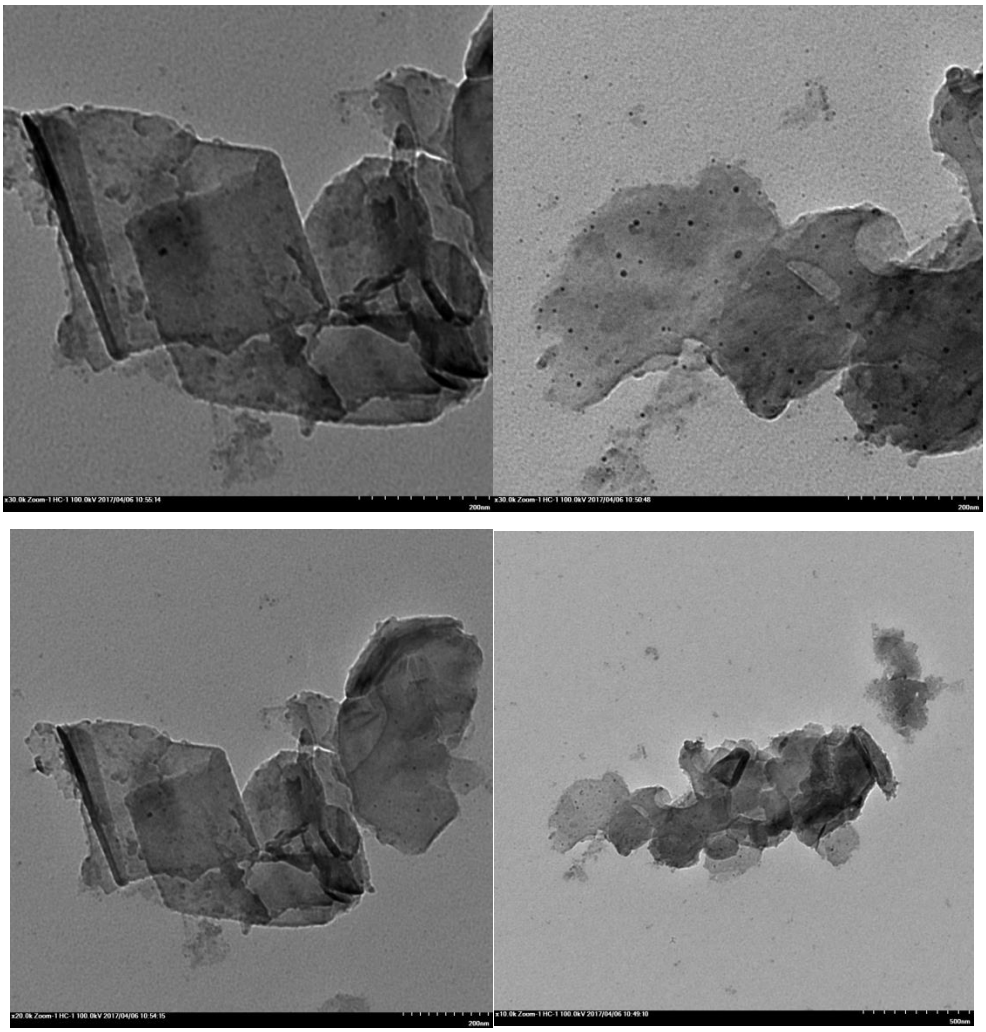


Figure S7. Raman spectra of Pt@G@Si.

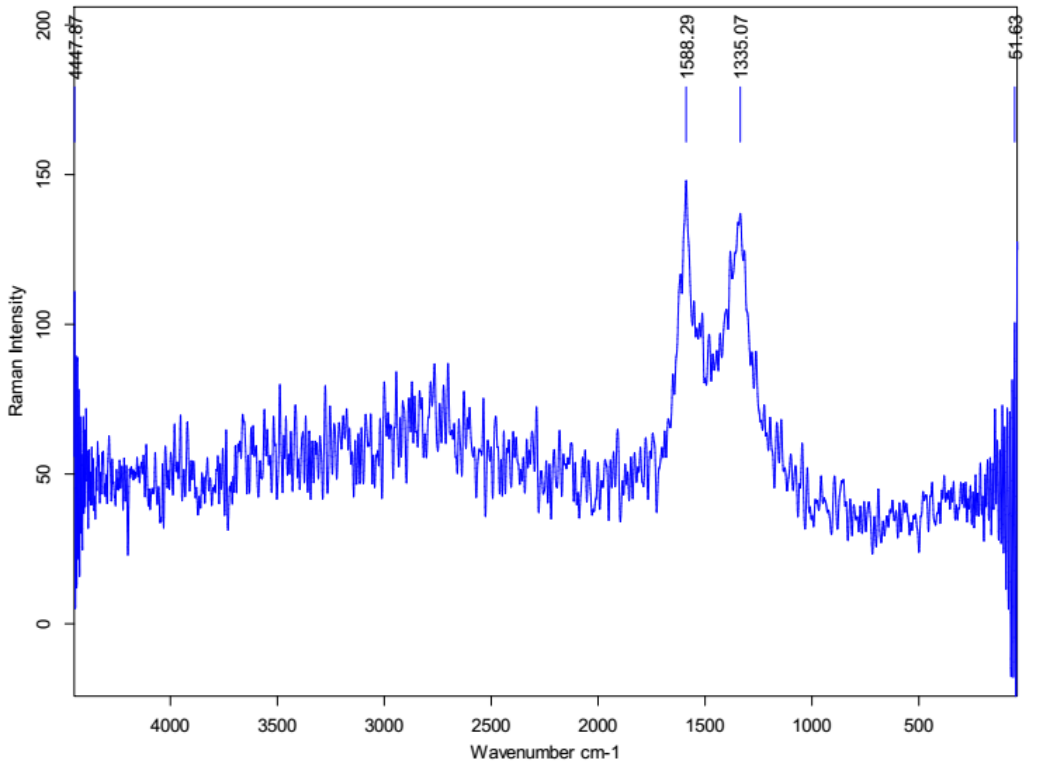
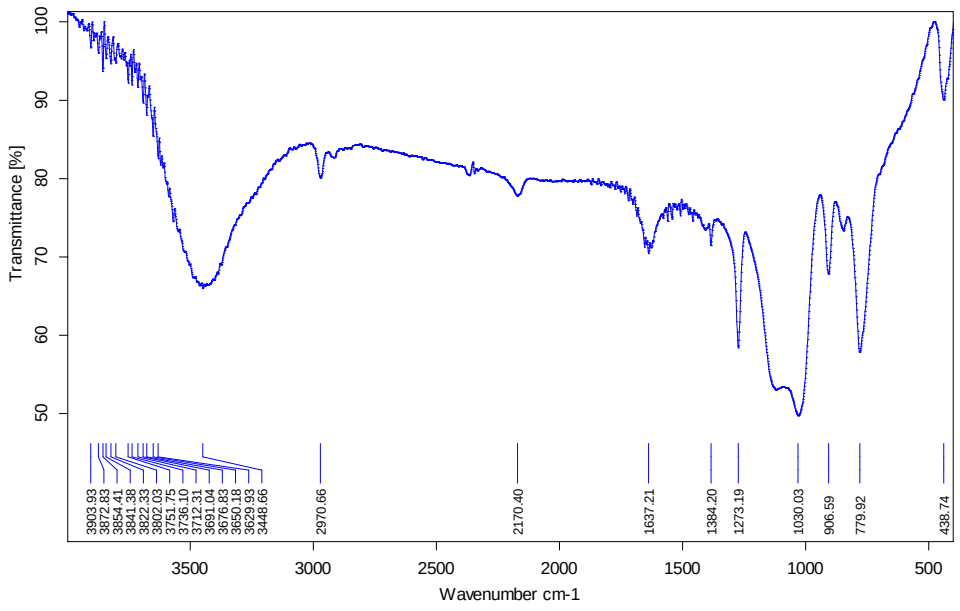
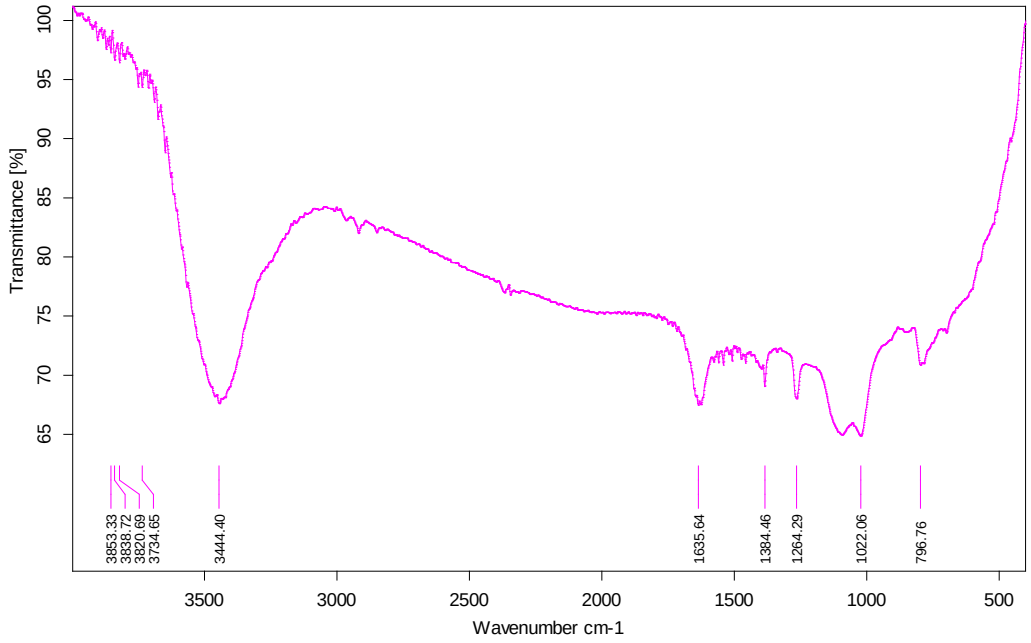


Figure S8. IR spectra of Pt@G@Si.





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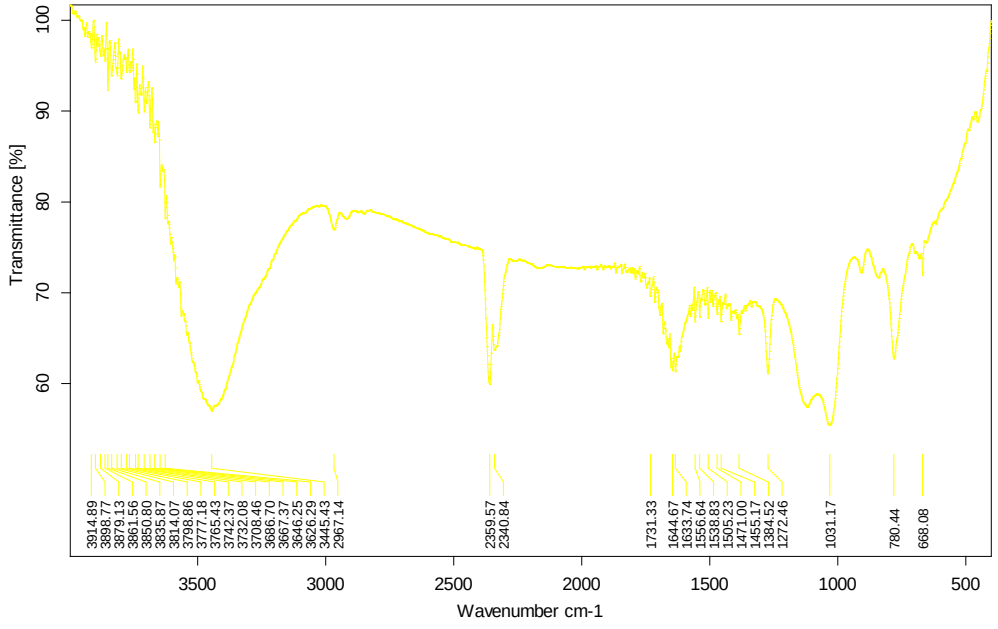

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Pt@G@Si (Pt: 5mol%, after recycling the first time)

Figure S9. XRD for Pt@Graphite@Si (PMHS@graphite powder@Pt) and Pt@G@Si (PMHS@graphene@Pt).

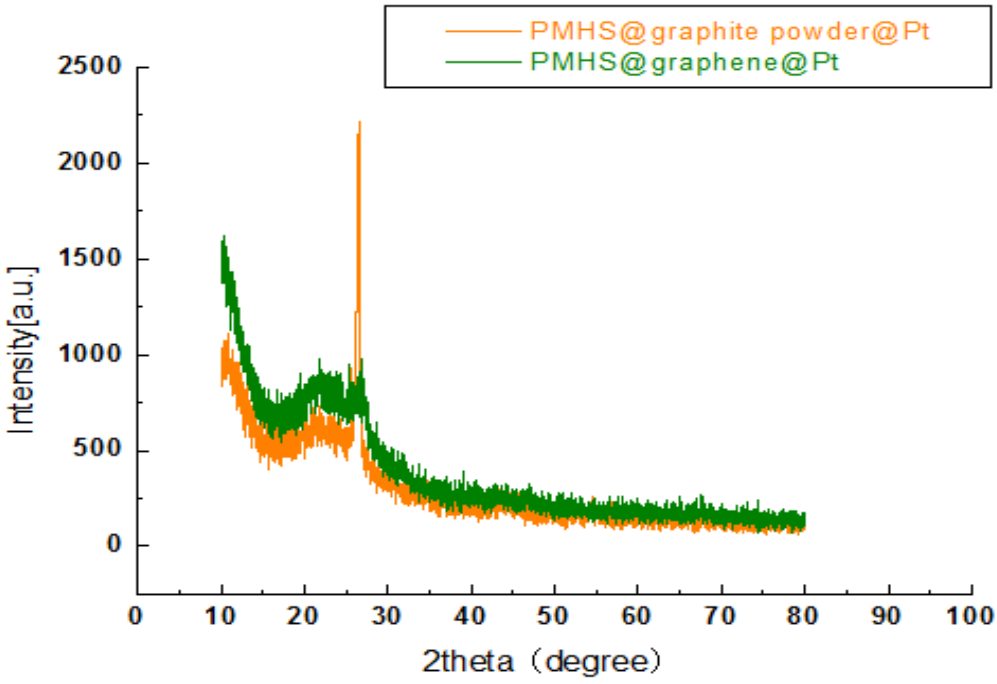


Figure S10. XRD for PMHS@graphene and PMHS@graphite.

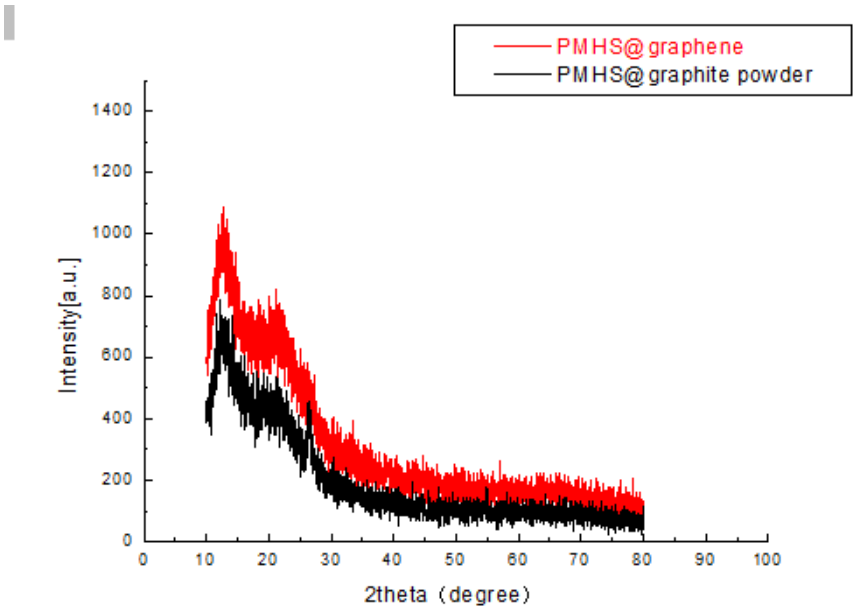
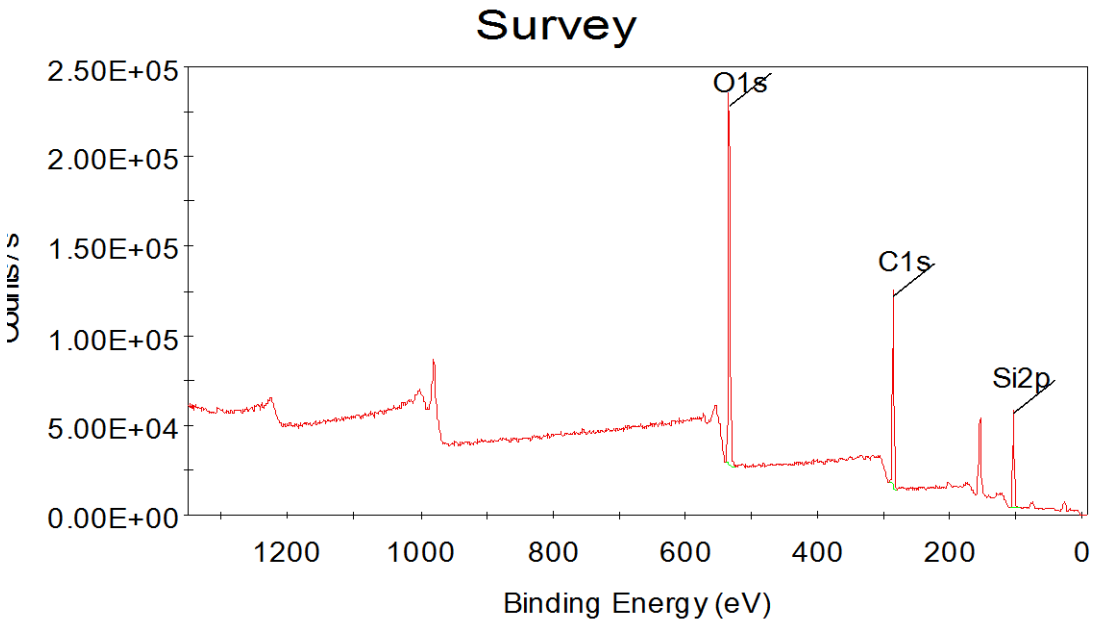
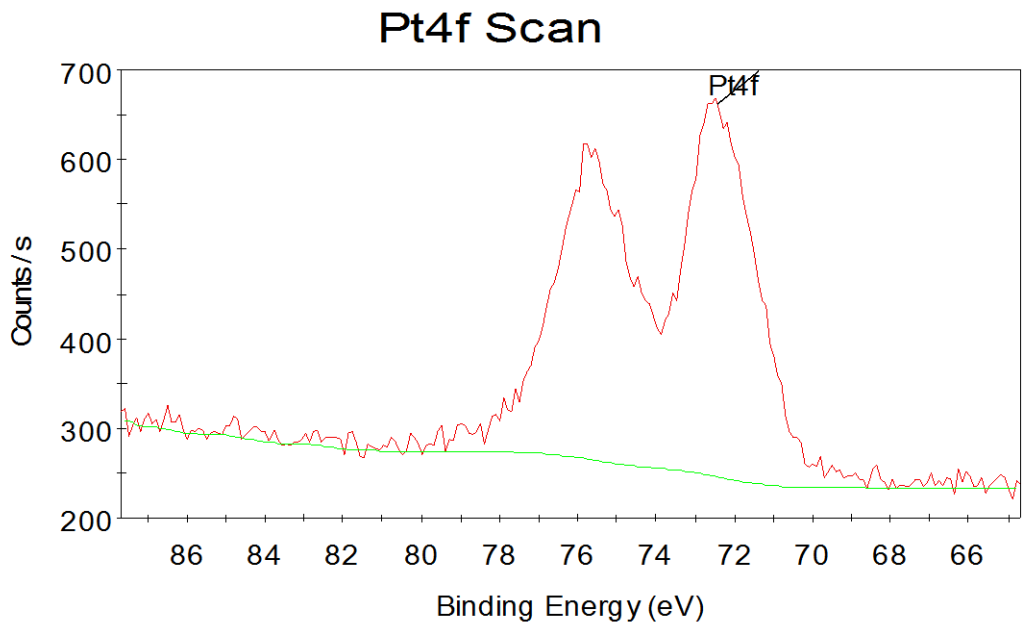
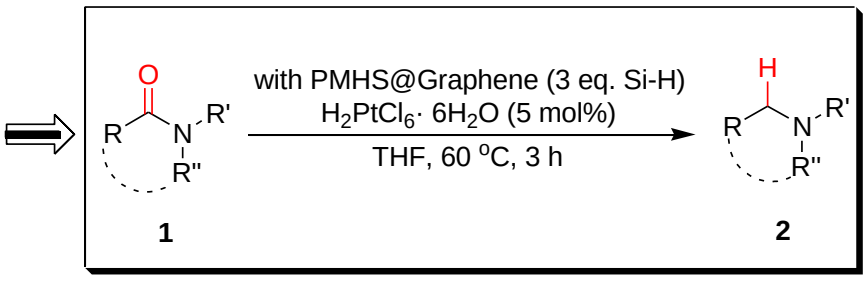
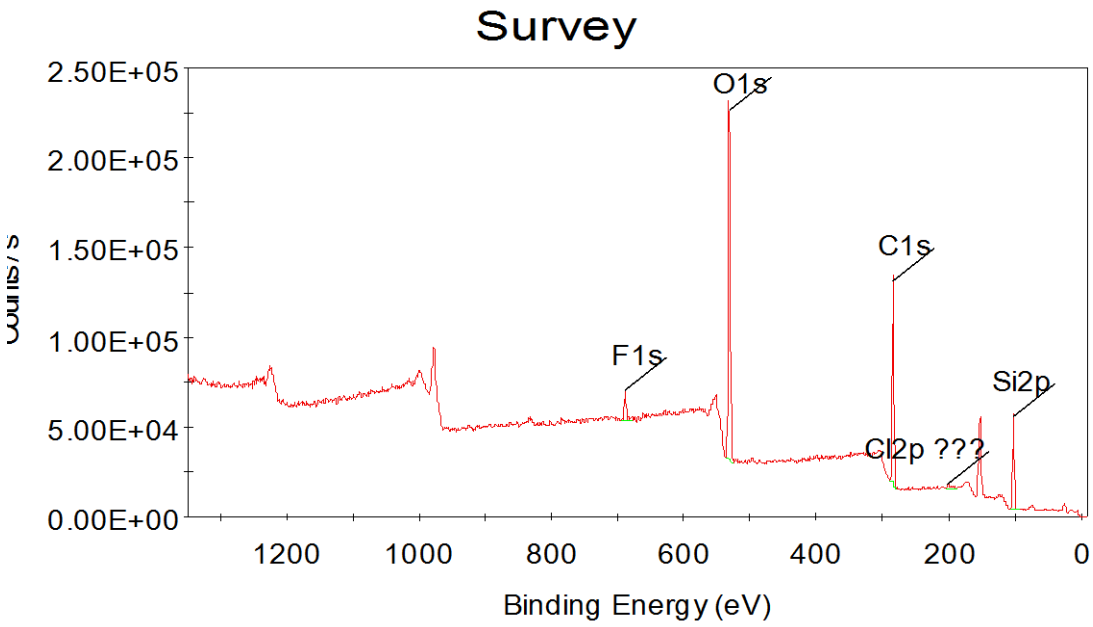


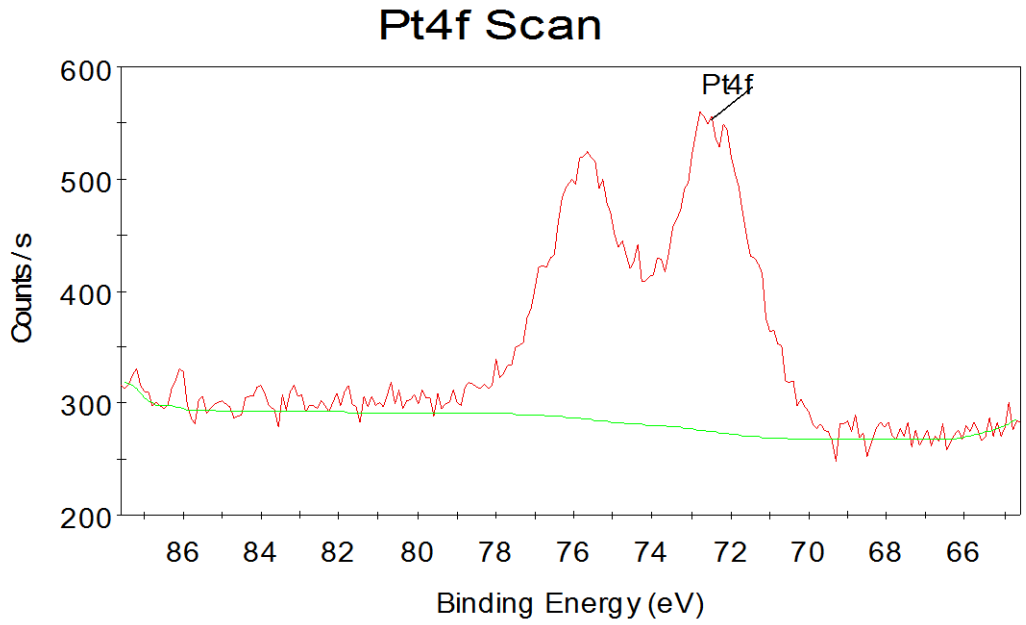
Figure S11. XPS analysis of Pt@G@Si.








Survey
2.50E+05
2.00E+05
1.50E+05
1.00E+05
5.00E+04
0.00E+00
Counts / s
O1s
C1s
F1s
Si2p
Cl2p ???
1200
1000
800
600
400
200
0
Binding Energy (eV)


Pt4f Scan
600
500
400
300
200
Counts / s
Pt4f
86
84
82
80
78
76
74
72
70
68
66
Binding Energy (eV)

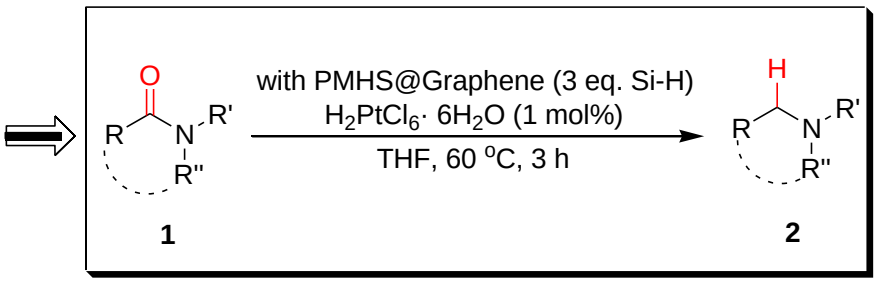
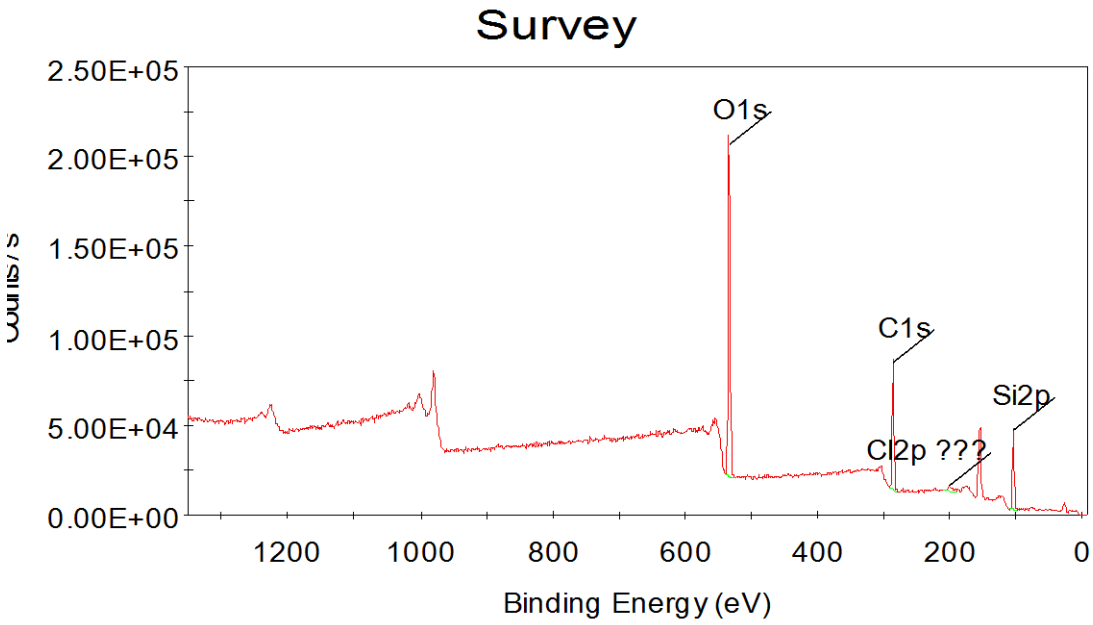
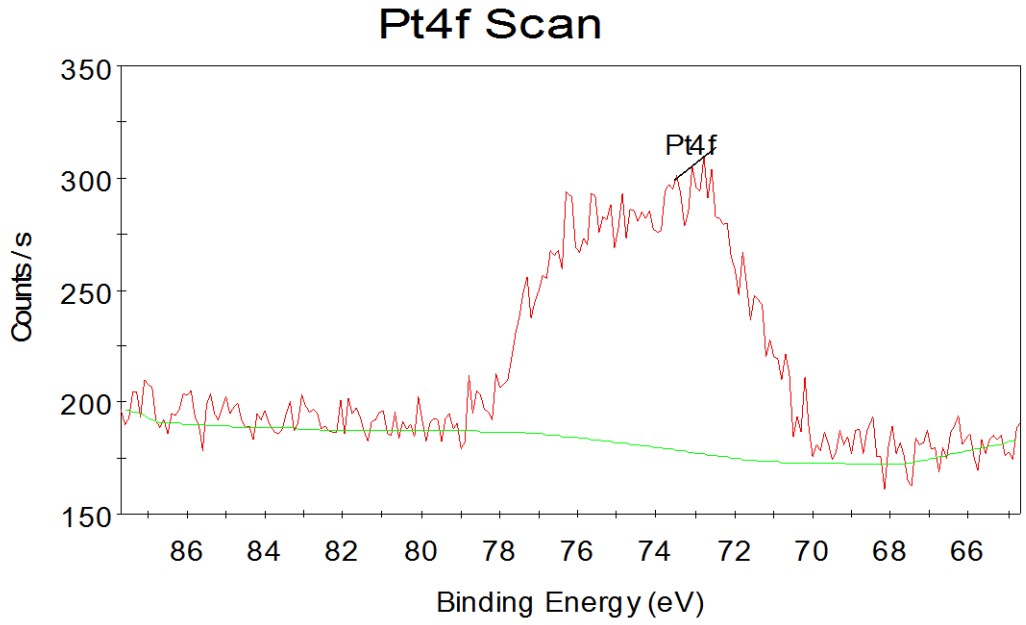

O
R
N
R'
R''
1
with PMHS@Graphene (3 eq. Si-H)
H2PtCl6· 6H2O (1 mol%)
THF, 60 °C, 3 h
H
R
N
R'
R''
2

Figure S12. XPS analysis of Pt@Graphite@Si.





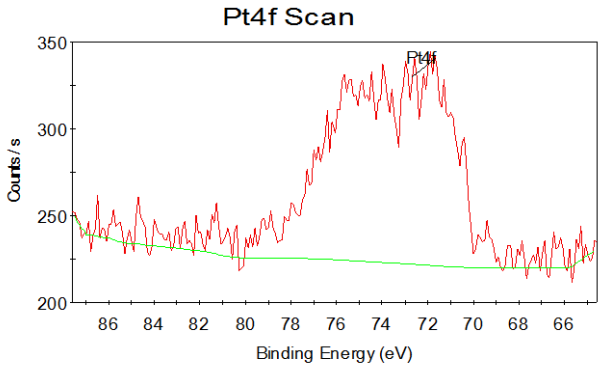
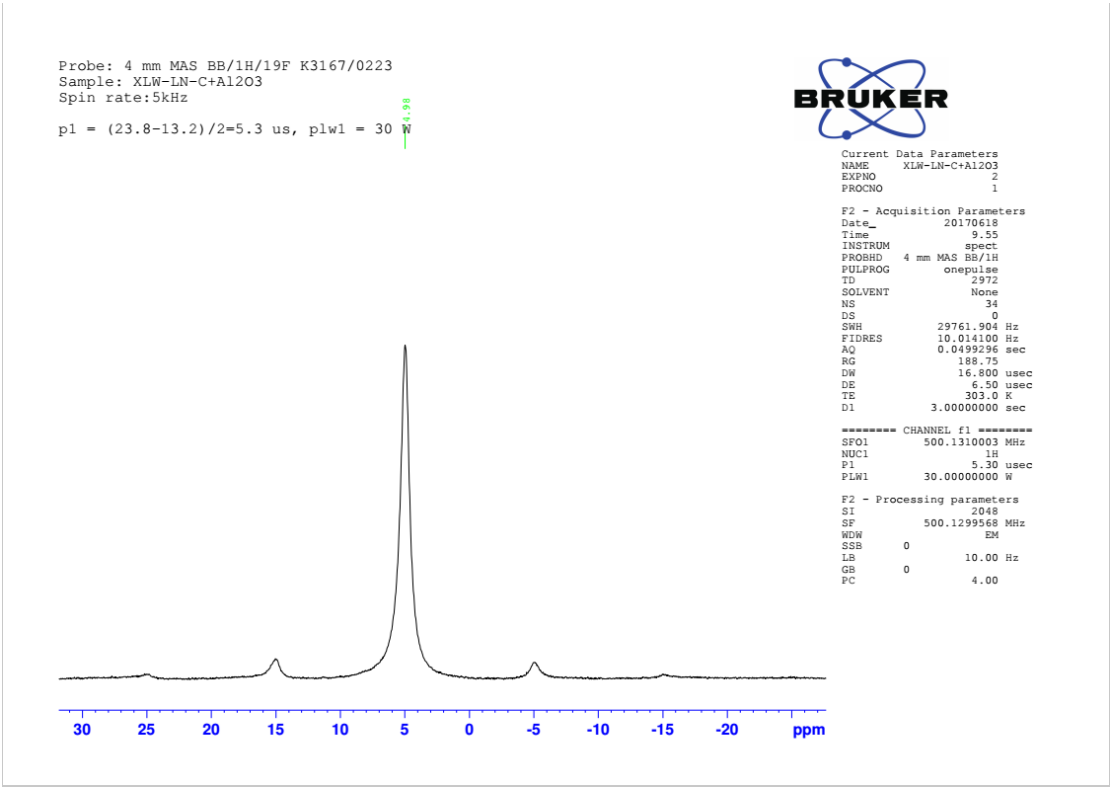
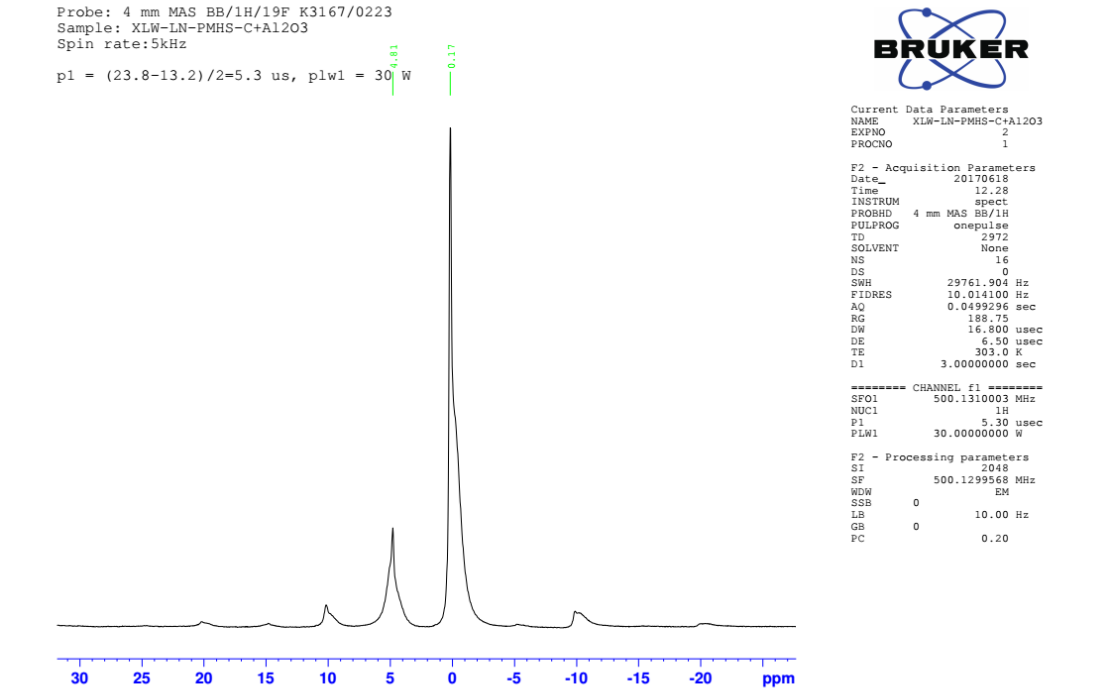


Figure S13. Solid-state NMR analysis of Pt@G@Si and graphene material.

(a). Solid-state NMR analysis of graphene



(b). Solid-state NMR analysis of G@PMHS



(c). Solid-state NMR analysis of Pt@G@Si

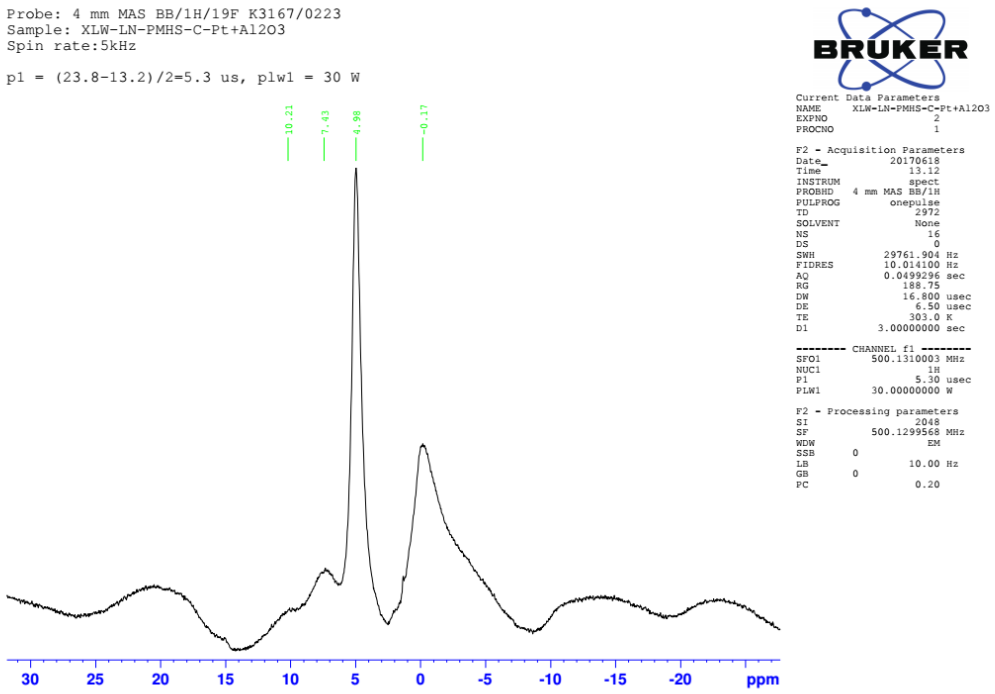
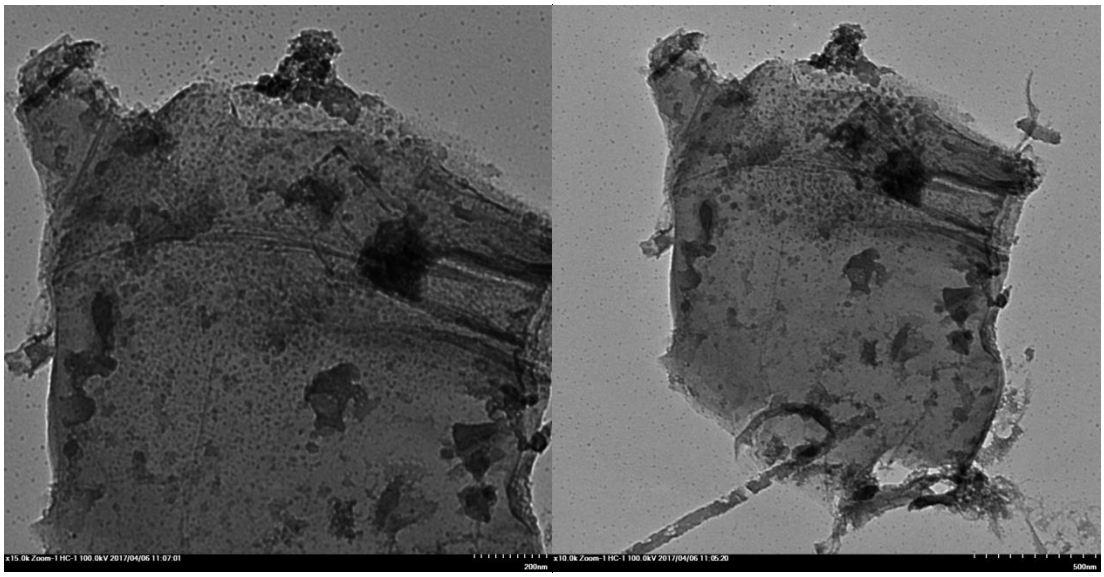
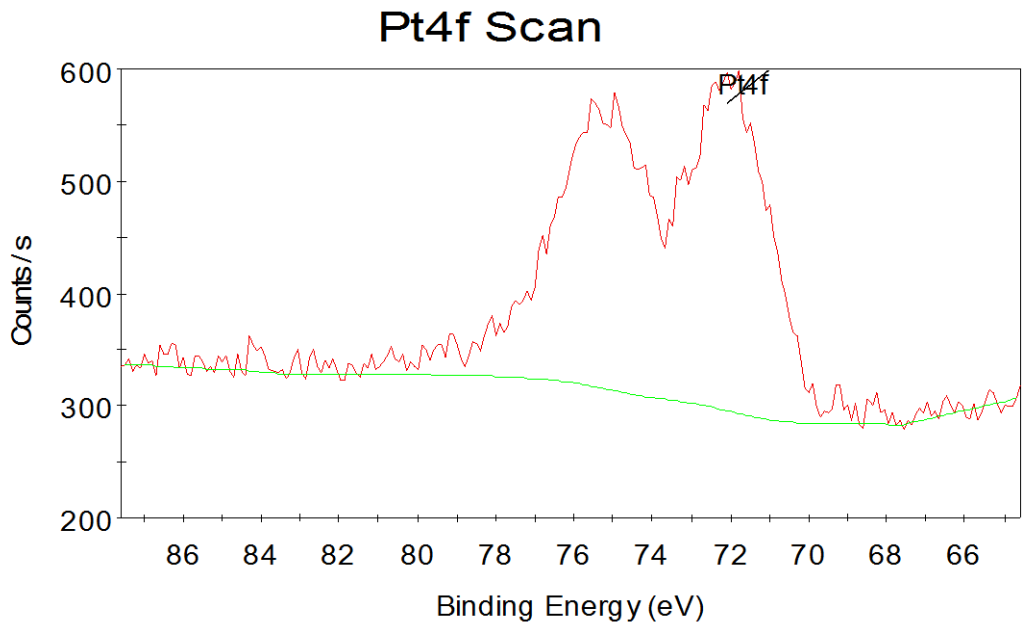


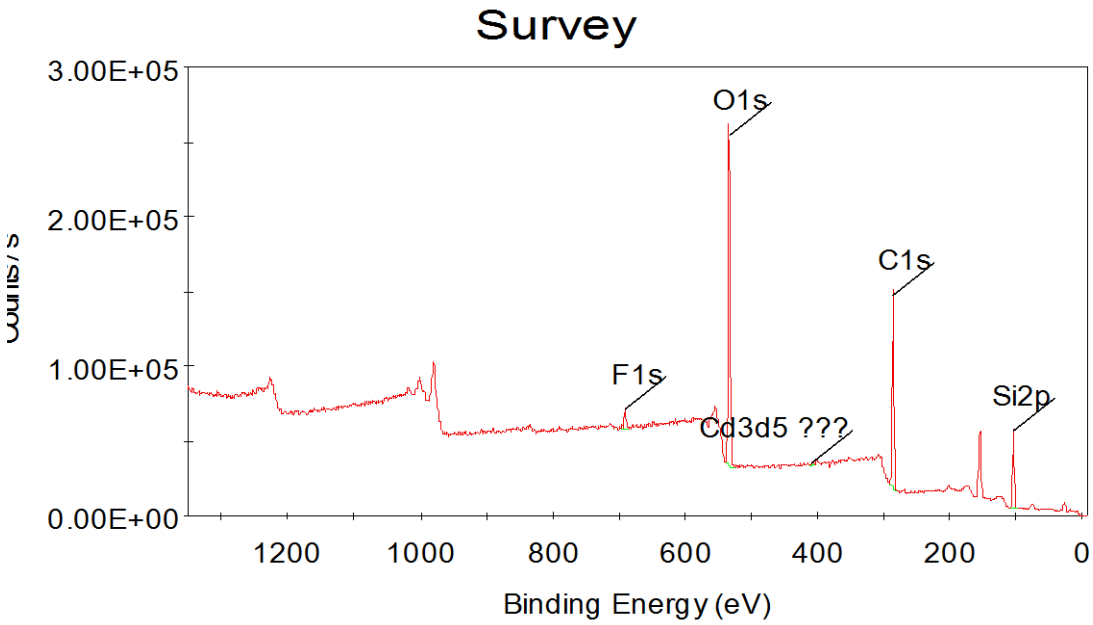
Figure S14. Spectra analysis of recovered Pt catalyst (Pt@G@Si) after hydrosilylation of chalcone.

(a) TEM image of recovered Pt@G@Si



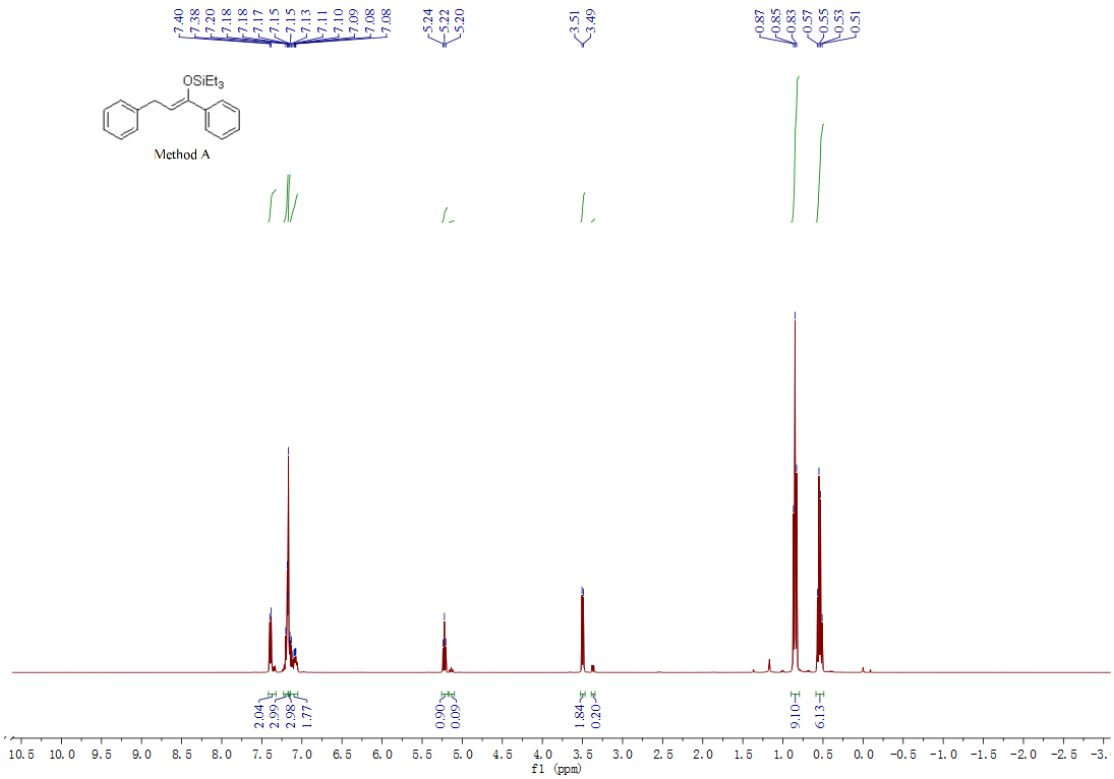
(b) XPS analysis of recovered Pt@Graphite@Si.

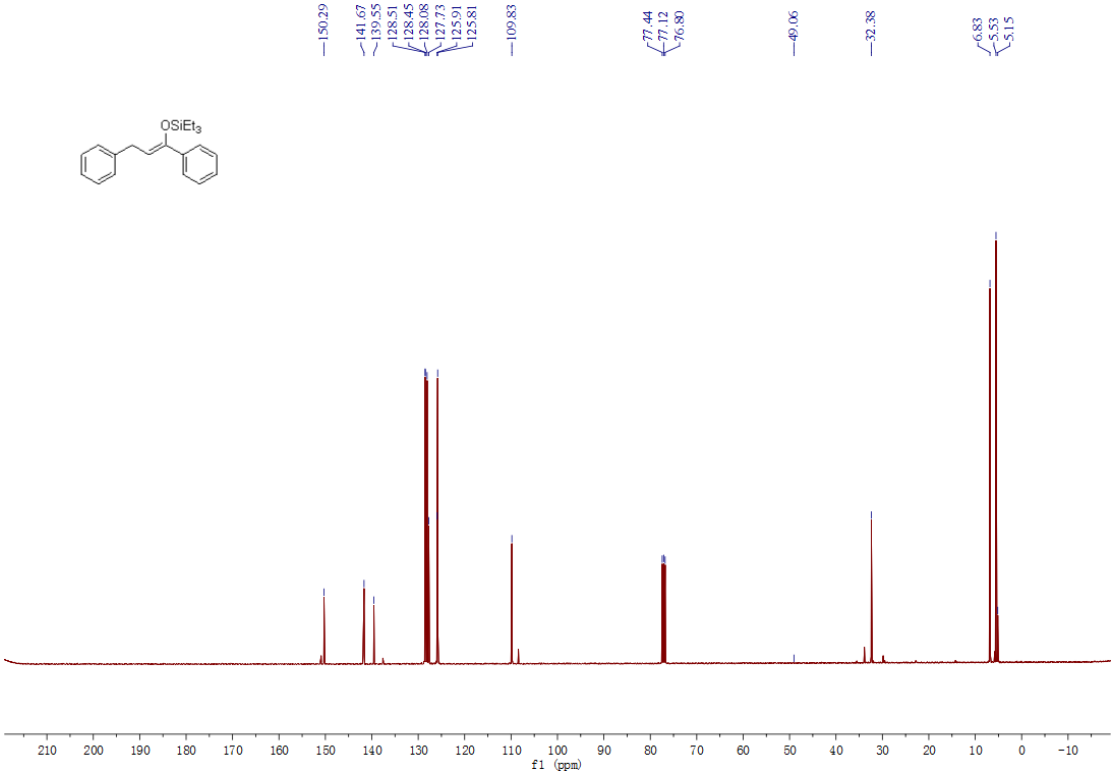


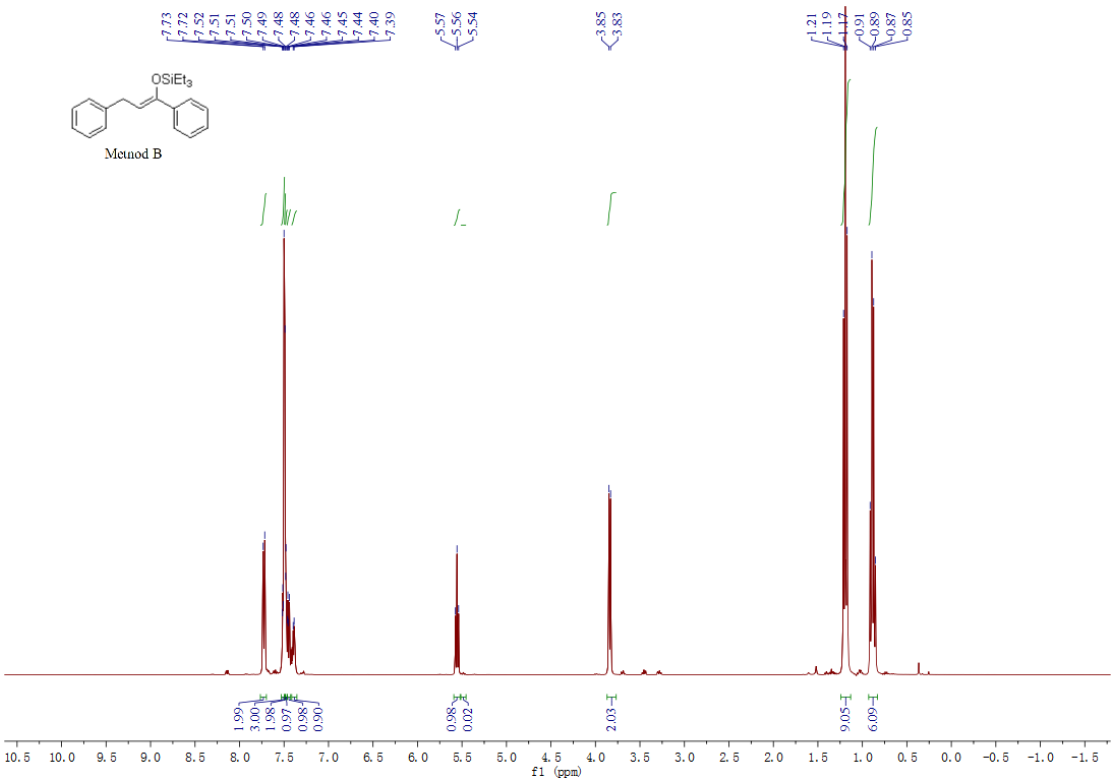


Name	Start BE	Peak BE	End BE	Height CPS	FWHM eV
O1s	538.08	532.92	524.58	210770.98	2.99
C1s	291.08	285.26	278.58	121917.21	2.98
Si2p	108.58	103.22	96.08	49979.67	2.99
F1s	697.08	690.26	684.08	13243.25	3.43
Cd3d5 ???	410.08	407.5	403.08	2118.46	1.59
Pt4f	87.48	72.07	64.68	296.24	2.8
C1s	295.48	284.76	279.68	16340.92	1.71

6. Copies of ^{1}H, ^{13}C, ^{29}Si and ^{19}F NMR spectra for the products.






OSiEt3
Metnod B
7.73
7.72
7.52
7.51
7.51
7.50
7.49
7.48
7.48
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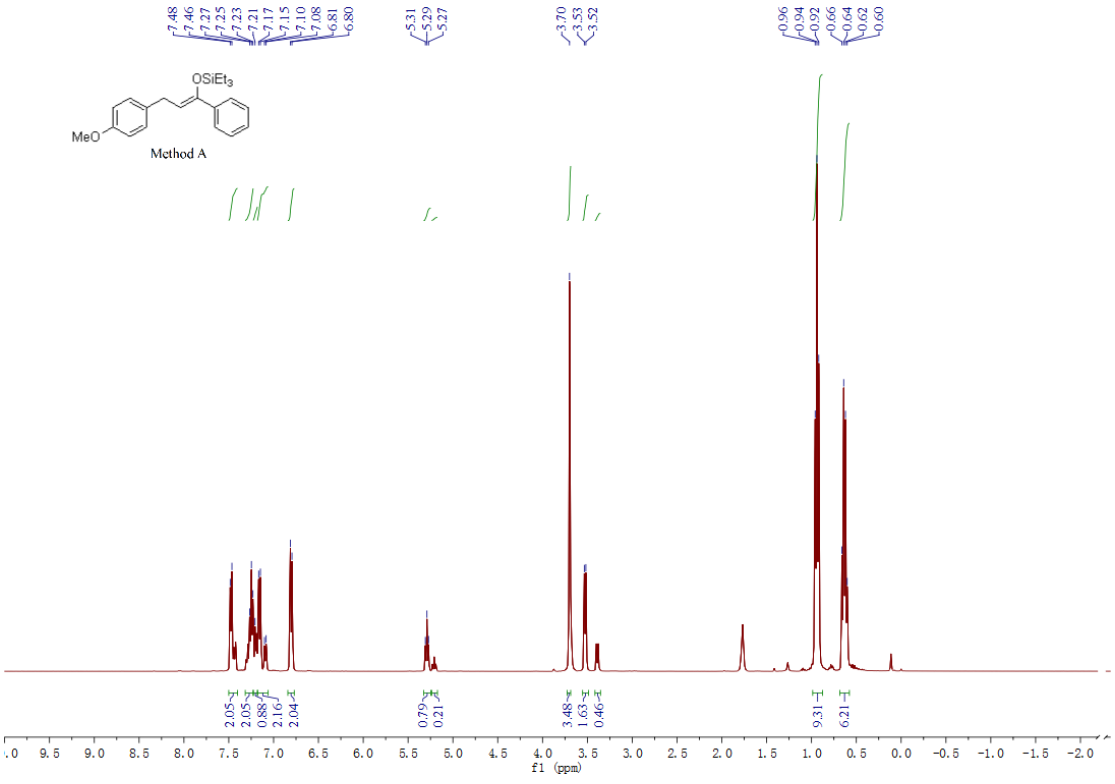
7.46
7.45
7.44
7.40
7.39
5.57
5.56
5.54
3.85
3.83
1.21
1.19
1.17
0.91
0.89
0.87
0.85
1.99
3.00
1.98
0.97
0.98
0.90
0.98
0.02
2.03
9.05
6.09
f1 (ppm)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MeO
Method A
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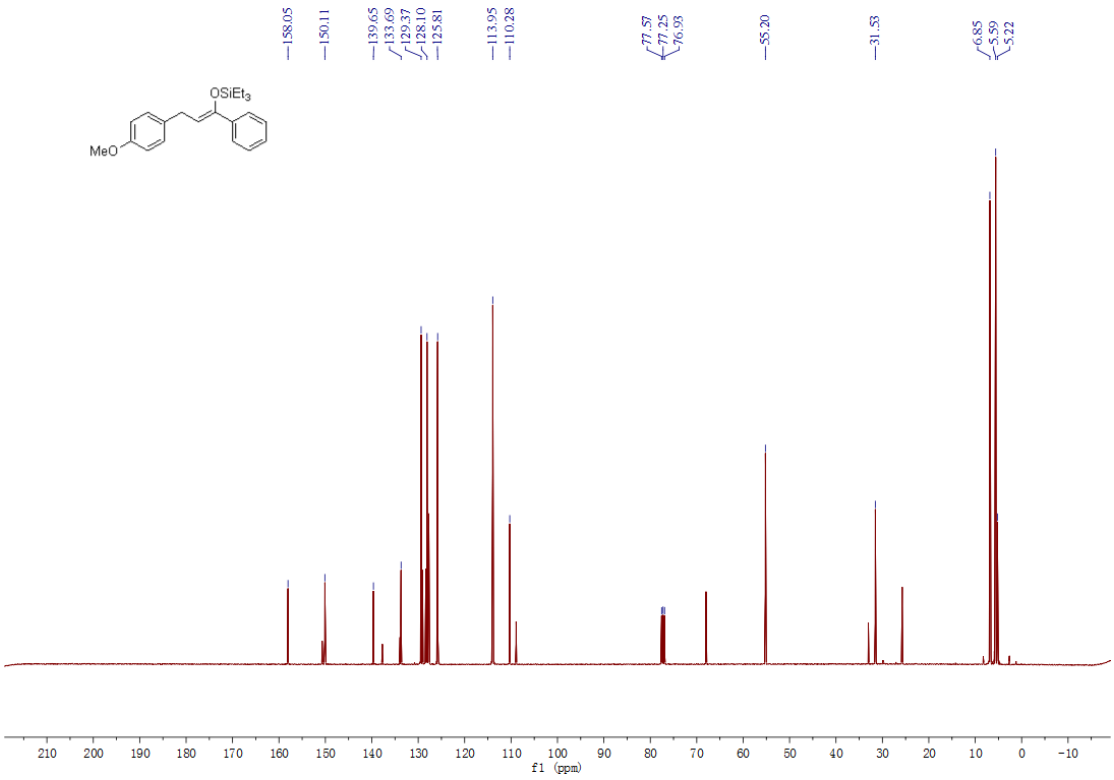
7.27
7.25
7.23
7.21
7.17
7.15
7.10
7.08
6.81
6.80
5.31
5.29
5.27
3.70
3.53
3.52
0.96
0.94
0.92
0.66
0.64
0.62
0.60
2.05
2.05
0.88
2.16
2.04
0.79
0.21
3.48
1.63
0.46
9.31
6.21
f1 (ppm)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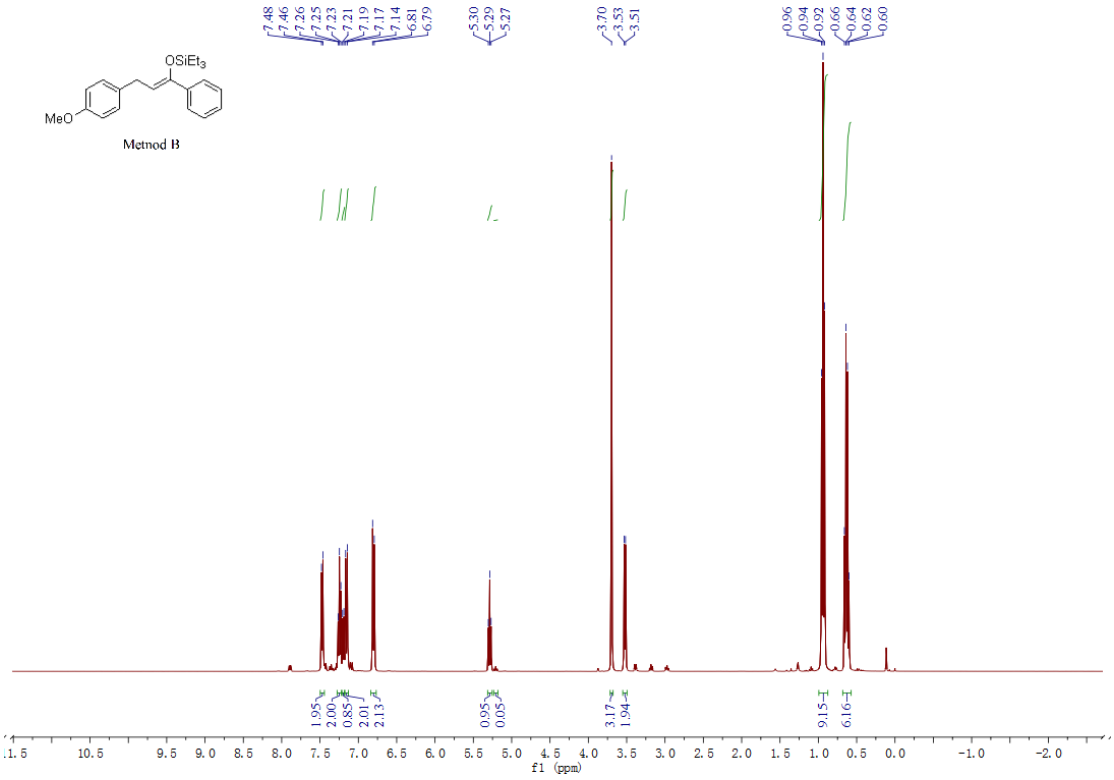
150.11
139.65
133.69
129.37
128.10
125.81
113.95
110.28
77.57
77.25
76.93
55.20
31.53
6.85
5.59
5.22
OSiEt3
MeO
f1 (ppm)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7.17
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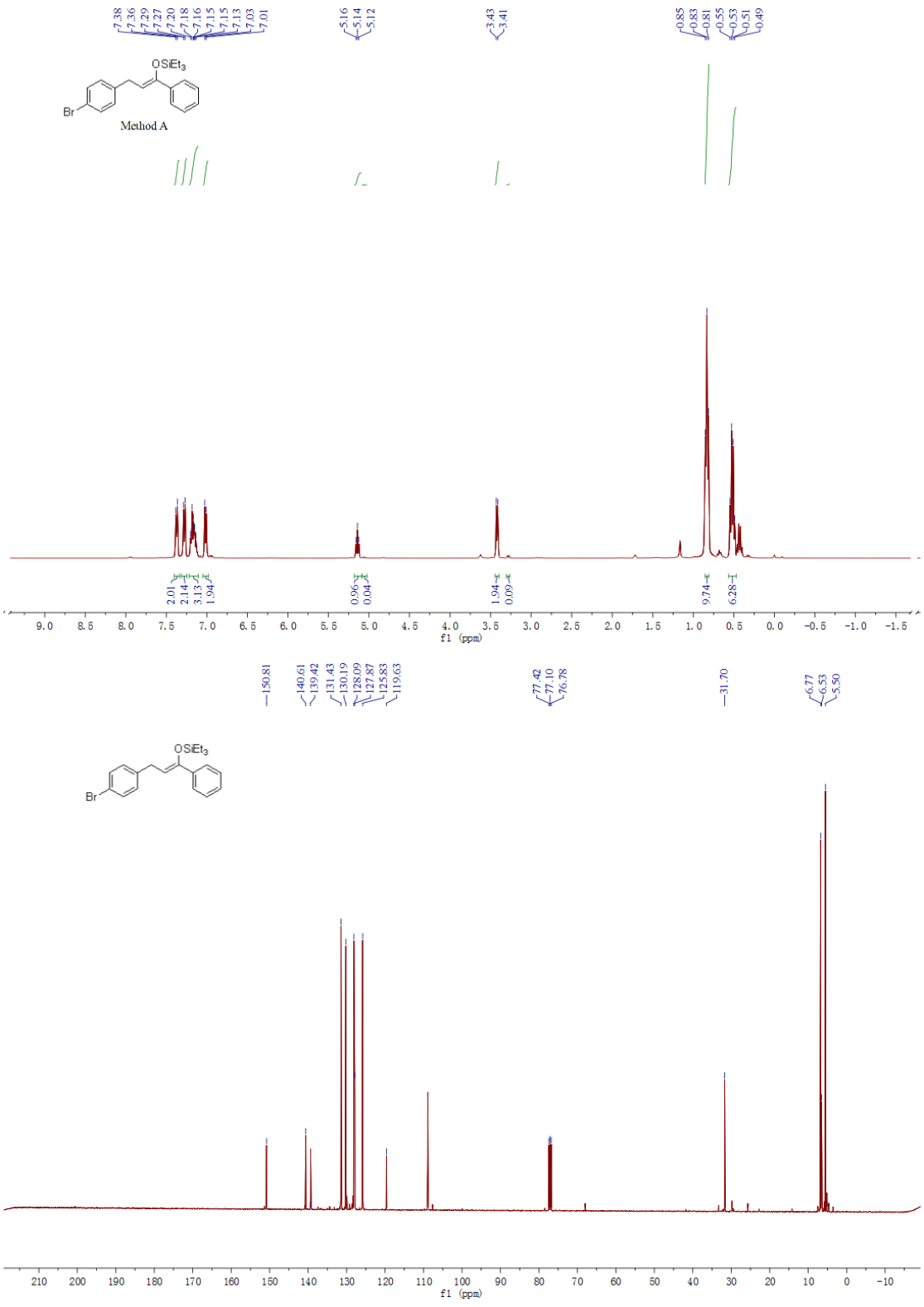
6.81
6.79
5.30
5.29
5.27
3.70
3.53
3.51
0.96
0.94
0.92
0.66
0.64
0.62
0.60
OSiEt3
MeO
Metnod B
1.95
2.00
0.85
2.01
2.13
0.95
0.05
3.17
1.94
9.15
6.16
f1 (ppm)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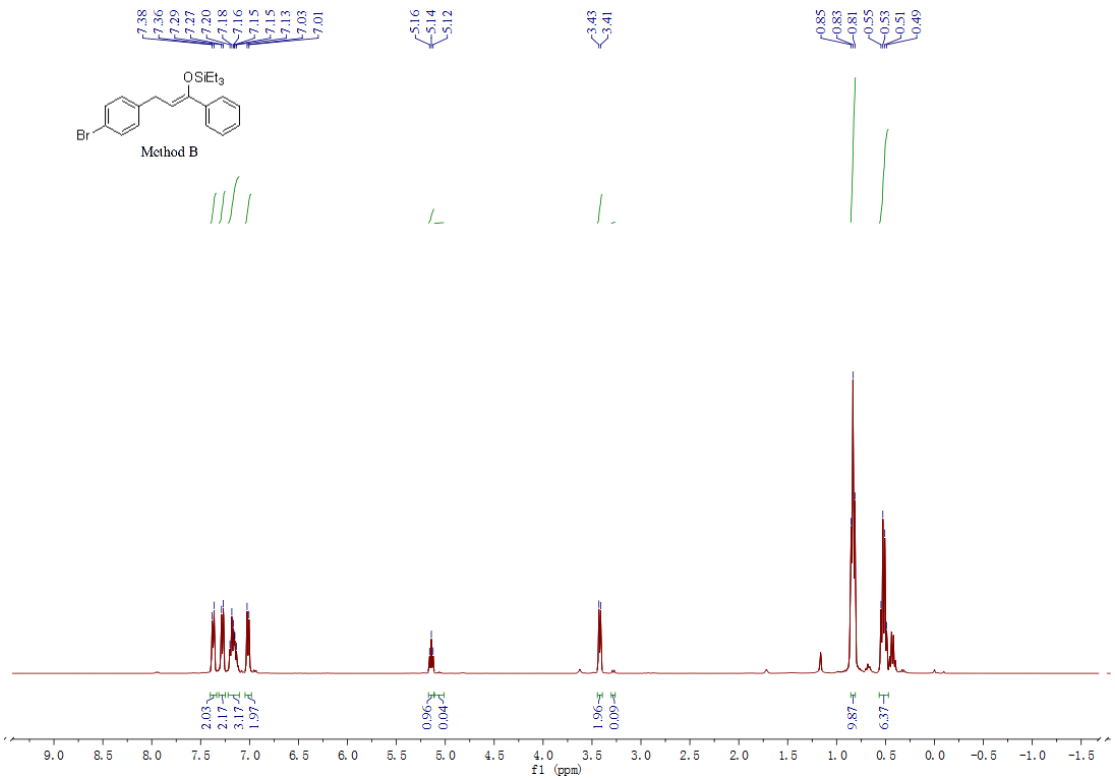
7.27
7.20
7.18
7.16
7.15
7.15
7.13
7.03
7.01
5.16
5.14
5.12
3.43
3.41
0.85
0.83
0.81
0.55
0.53
0.51
0.49
OSiEt3
Br
Method A
2.01
2.14
3.13
1.94
0.96
0.04
1.94
0.09
9.74
6.28
f1 (ppm)
150.81
140.61
139.42
131.43
130.19
128.09
127.87
125.83
119.63
77.42
77.10
76.78
31.70
6.77
6.53
5.50
OSiEt3
Br
f1 (ppm)


7.38
7.36
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7.27
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7.16
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7.01
5.16
5.14
5.12
3.43
3.41
0.85
0.83
0.81
0.55
0.53
0.51
0.49
OSiEt3
Br
Method B
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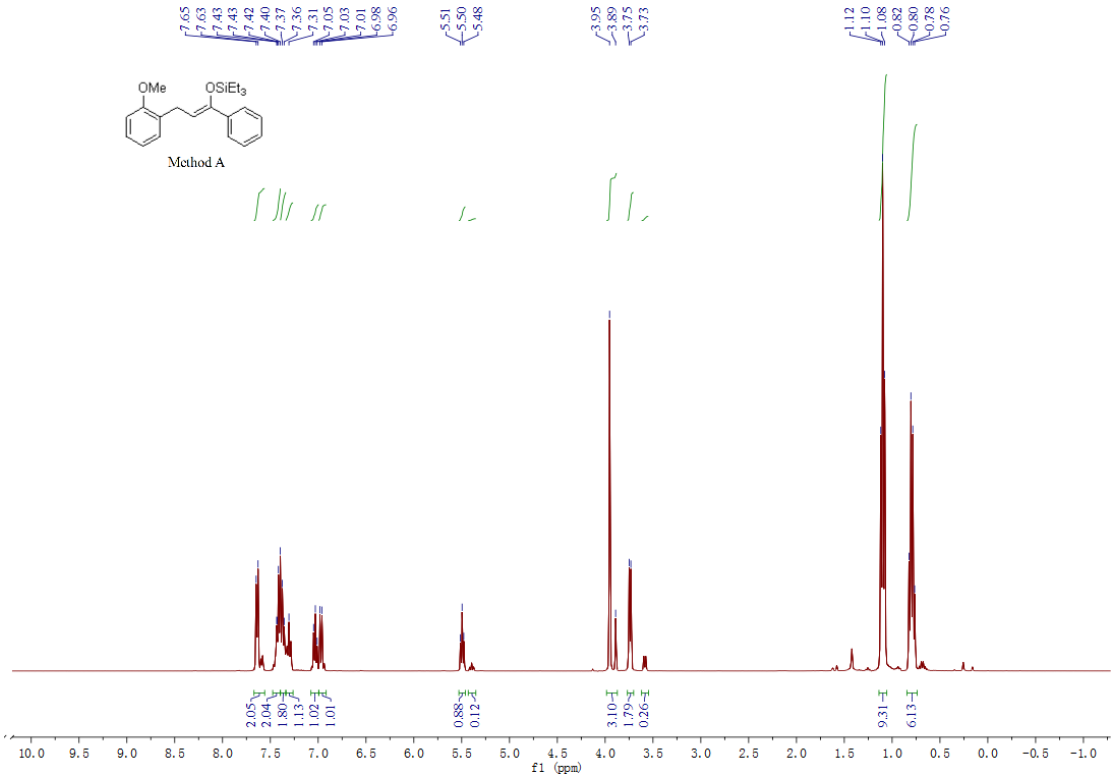
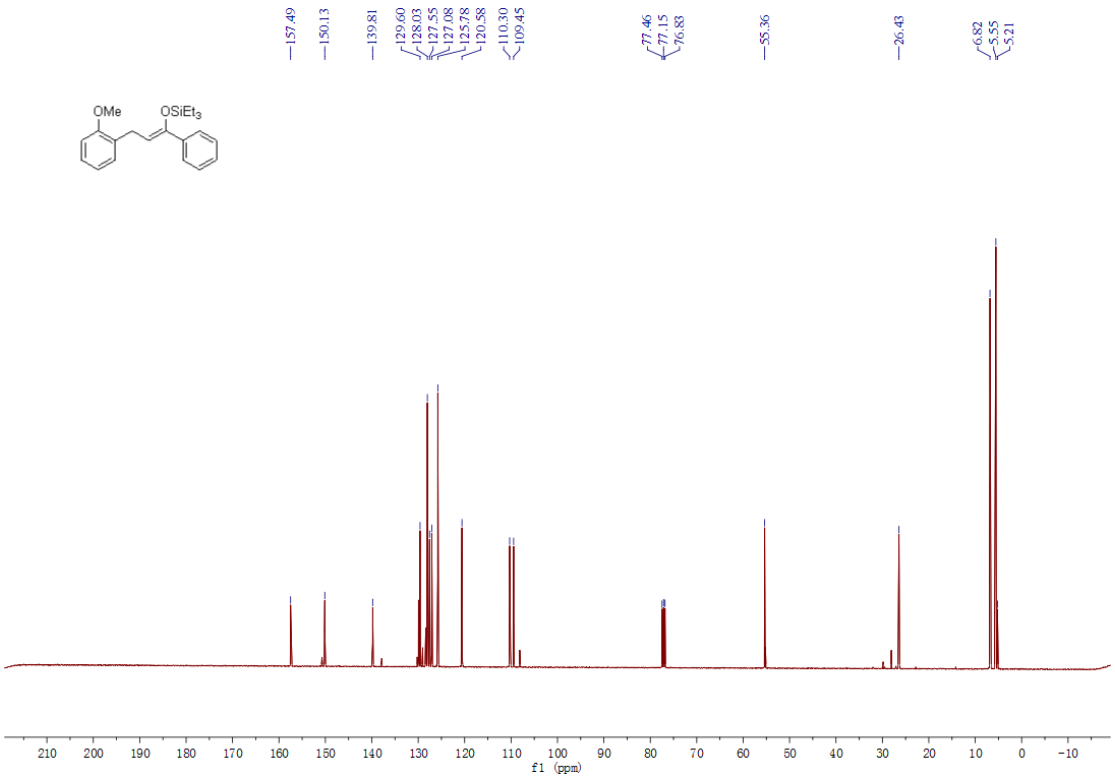
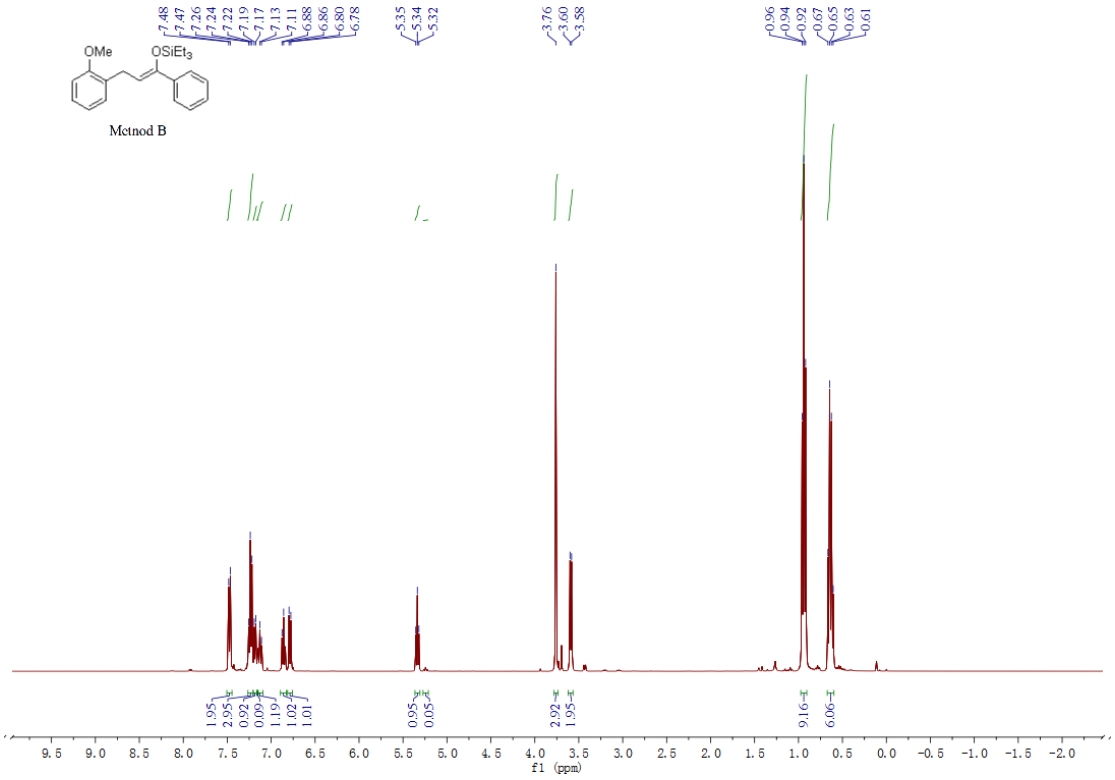
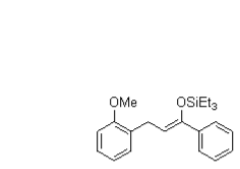
2.17
3.17
1.97
0.96
0.04
1.96
0.09
9.87
6.37
f1 (ppm)

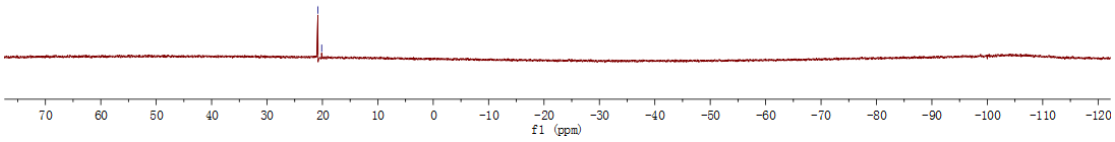

7.65
7.63
7.43
7.43
7.42
7.40
7.37
7.36
7.31
7.05
7.03
7.01
6.98
6.96
5.51
5.50
5.48
3.95
3.89
3.75
3.73
1.12
1.10
1.08
0.82
0.80
0.78
0.76
OMe
OSiEt3
Method A
2.05
2.04
1.80
1.13
1.02
1.01
0.88
0.12
3.10
1.79
0.26
9.31
6.13
f1 (ppm)

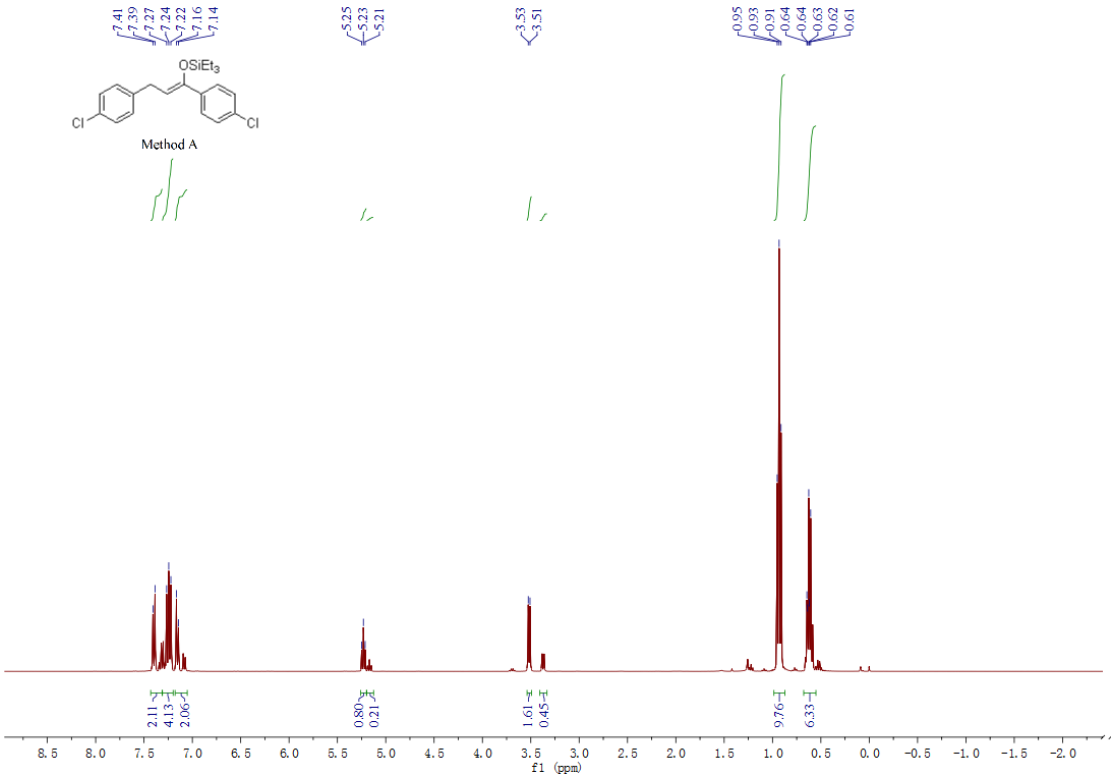

157.49
150.13
139.81
129.60
128.03
127.55
127.08
125.78
120.58
110.30
109.45
77.46
77.15
76.83
55.36
26.43
6.82
5.55
5.21
OMe
OSiEt3
f1 (ppm)

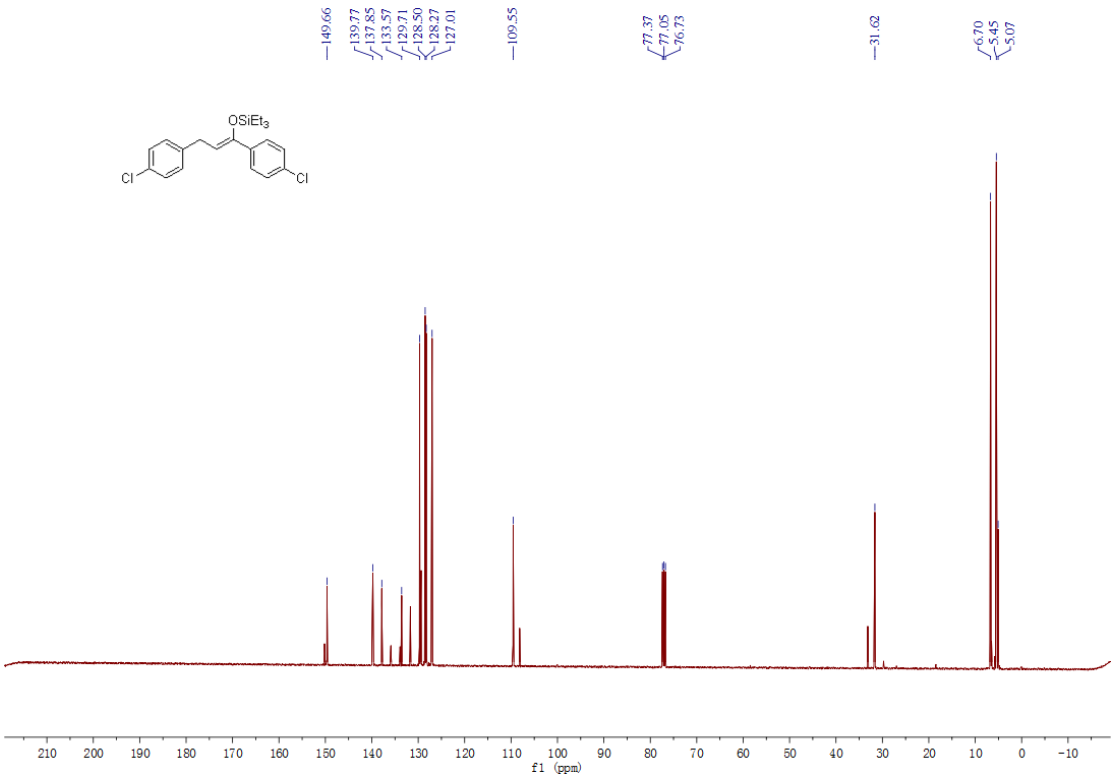

7.48
7.47
7.26
7.24
7.22
7.19
7.17
7.13
7.11
6.88
6.86
6.80
6.78
5.35
5.34
5.32
3.76
3.60
3.58
0.96
0.94
0.92
0.67
0.65
0.63
0.61
OMe
OSiEt3
Metnod B
1.95
2.95
0.92
0.09
1.19
1.02
1.01
0.95
0.05
2.92
1.95
9.16
6.06
f1 (ppm)

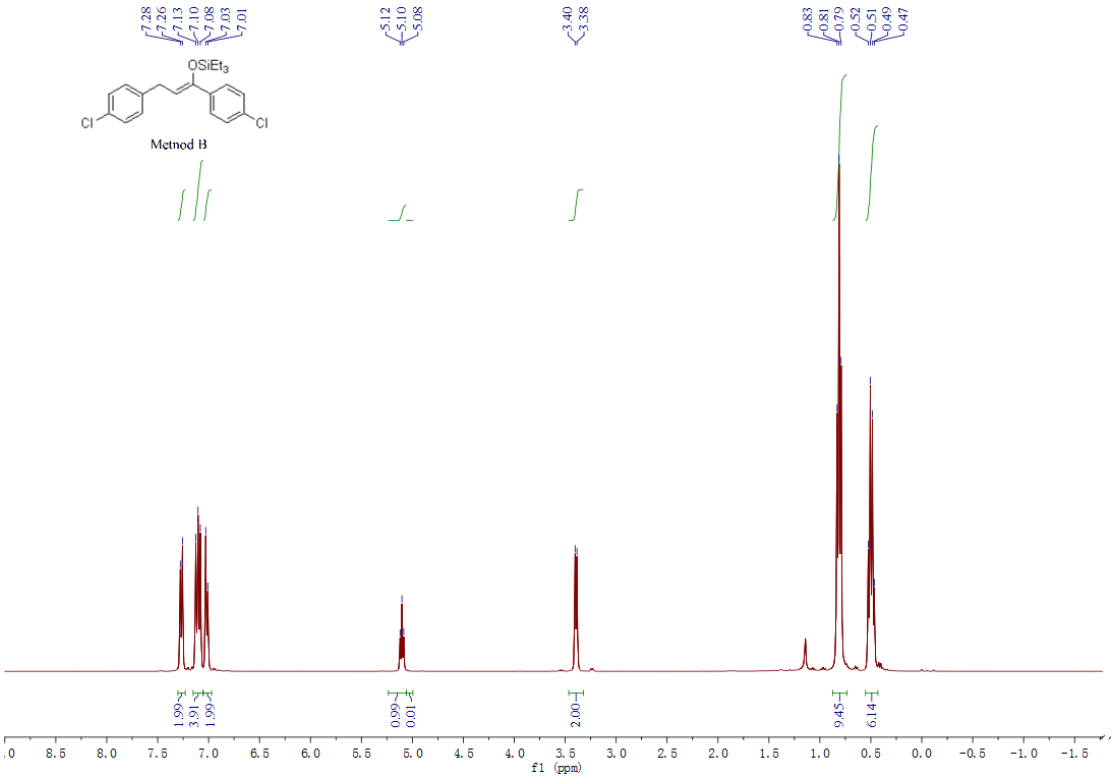


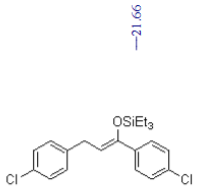
^{29}Si NMR (99 MHz, $CDCl_3$) δ 20.82 (s), 20.13 (s).



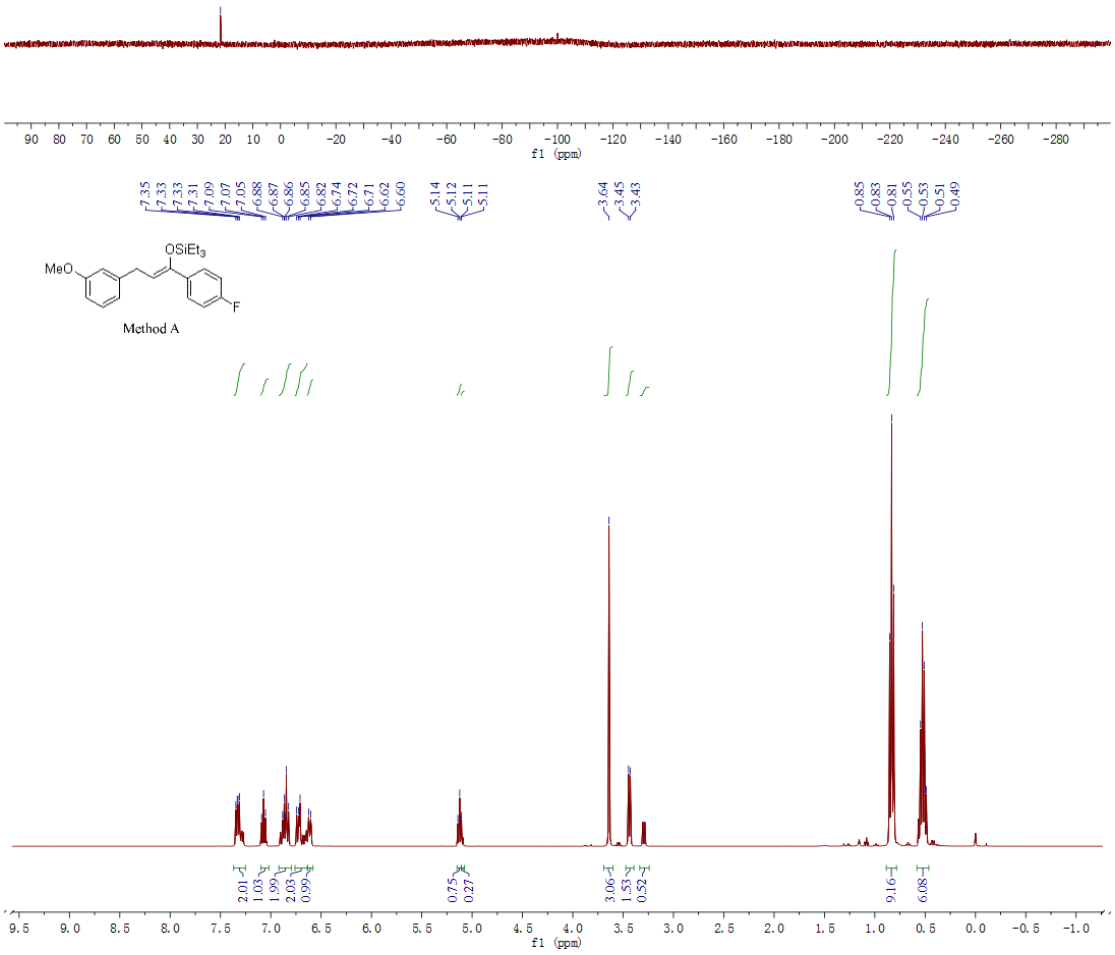


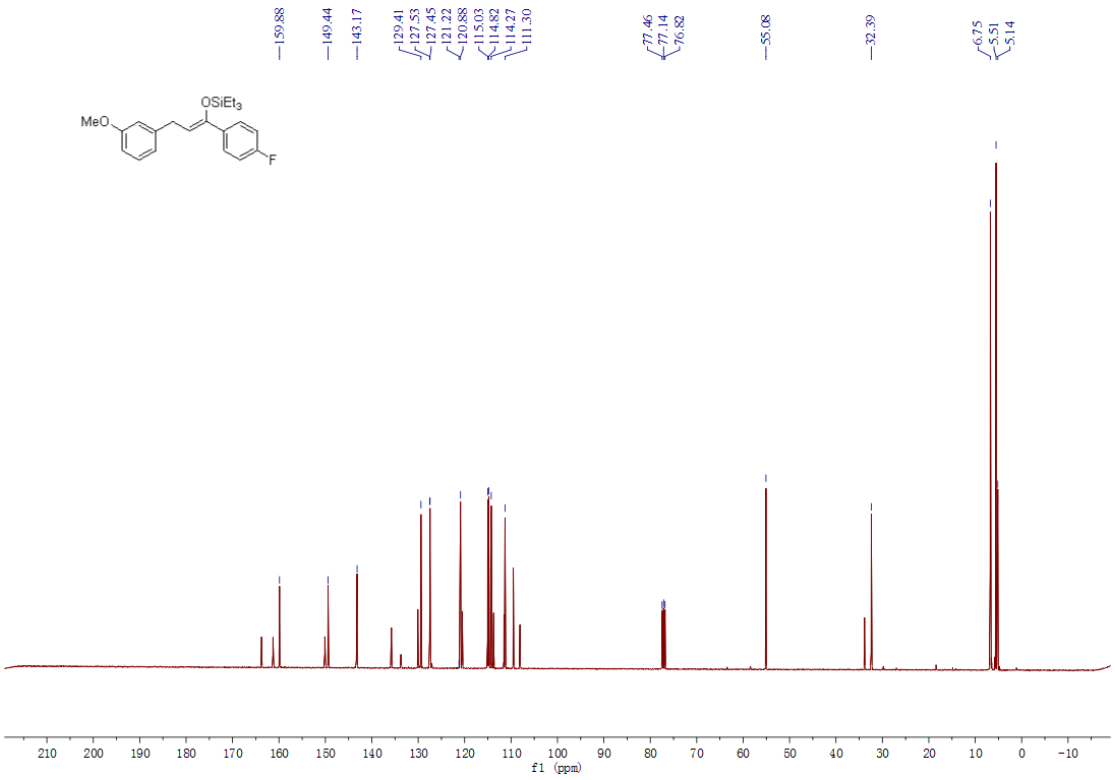

149.66
139.77
137.85
133.57
129.71
128.50
128.27
127.01
109.55
77.37
77.05
76.73
31.62
6.70
5.45
5.07
OSiEt3
Cl
Cl
f1 (ppm)

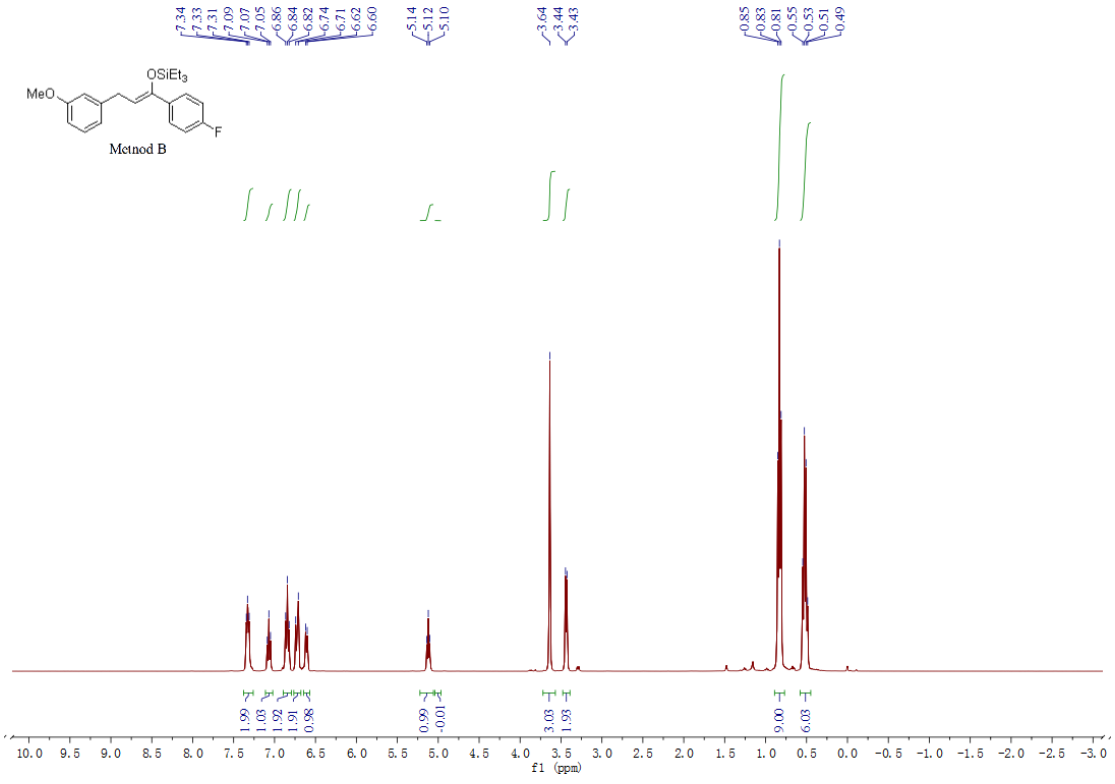

7.28
7.26
7.13
7.10
7.08
7.03
7.01
5.12
5.10
5.08
3.40
3.38
0.83
0.81
0.79
0.52
0.51
0.49
0.47
OSiEt3
Cl
Cl
Metnod B
1.99
3.91
1.99
0.99
0.01
2.00
9.45
6.14
f1 (ppm)

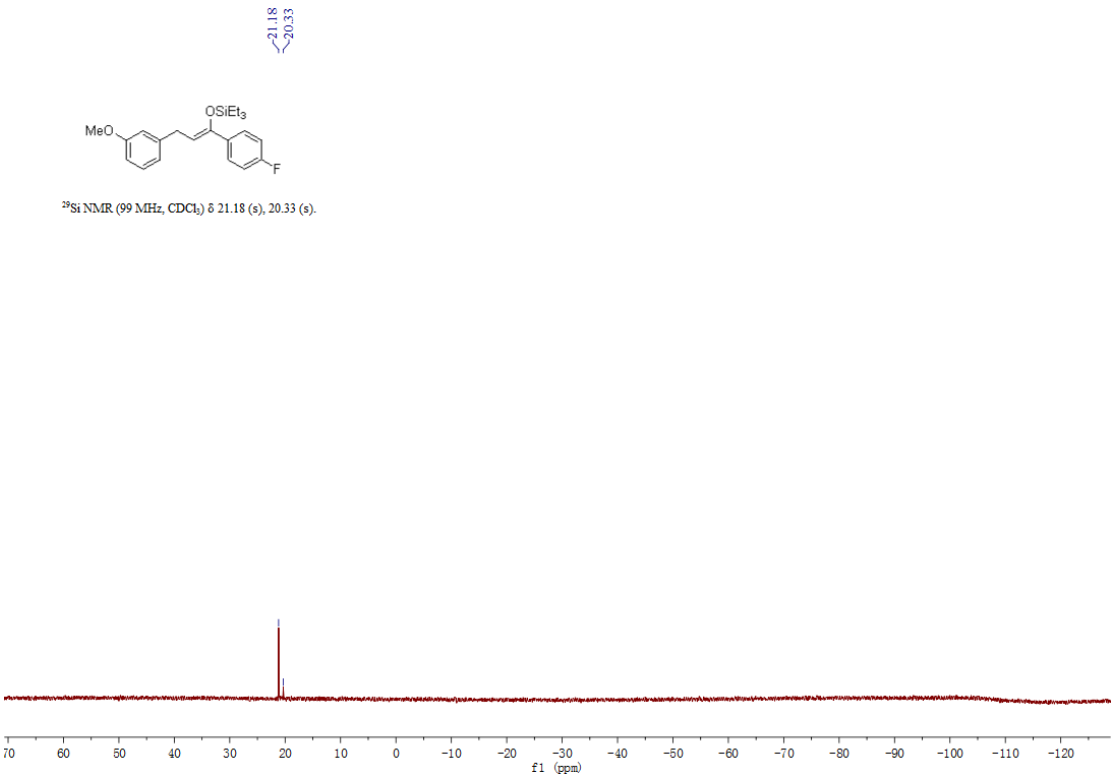


^{29}Si NMR (99 MHz, $CDCl_3$) δ 21.66 (s).

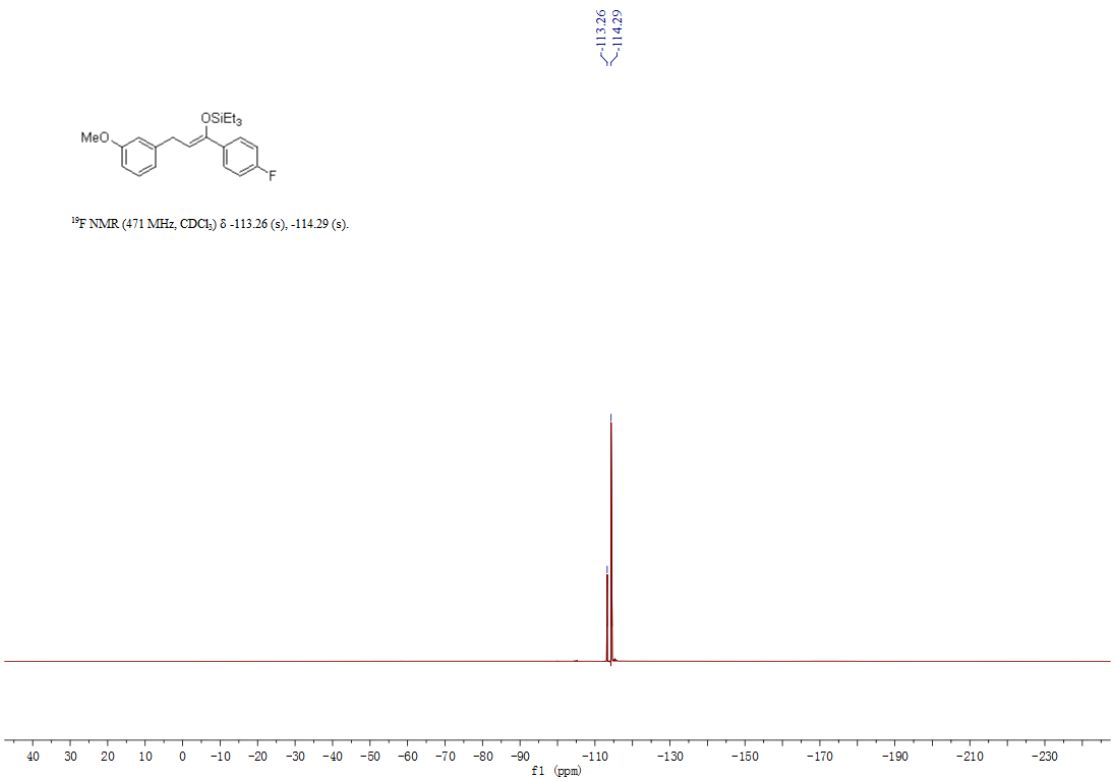



159.88
149.44
143.17
129.41
127.53
127.45
121.22
120.88
115.03
114.82
114.27
111.30
77.46
77.14
76.82
55.08
32.39
6.75
5.51
5.14
OSiEt3
MeO
F
f1 (ppm)

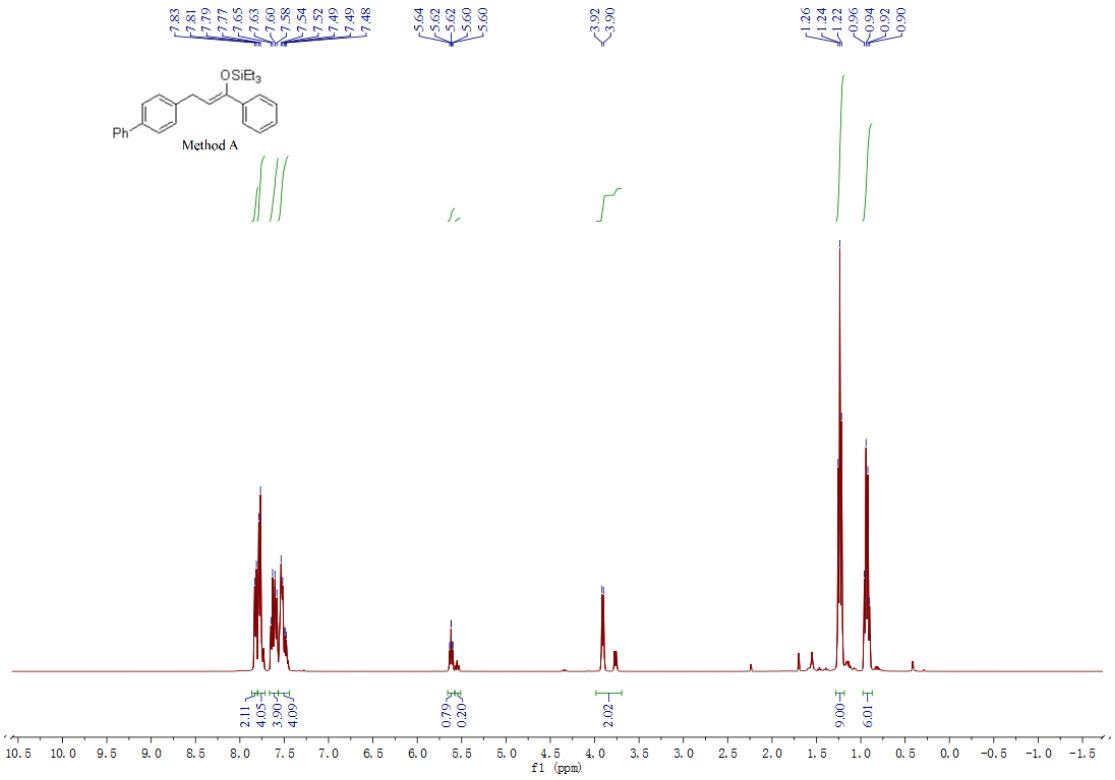

7.34
7.33
7.31
7.09
7.07
7.05
6.86
6.84
6.82
6.74
6.71
6.62
6.60
5.14
5.12
5.10
3.64
3.44
3.43
0.85
0.83
0.81
0.55
0.53
0.51
0.49
OSiEt3
MeO
F
Metnod B
1.99
1.03
1.92
1.91
0.88
0.99
-0.01
3.03
1.93
9.00
6.03
f1 (ppm)

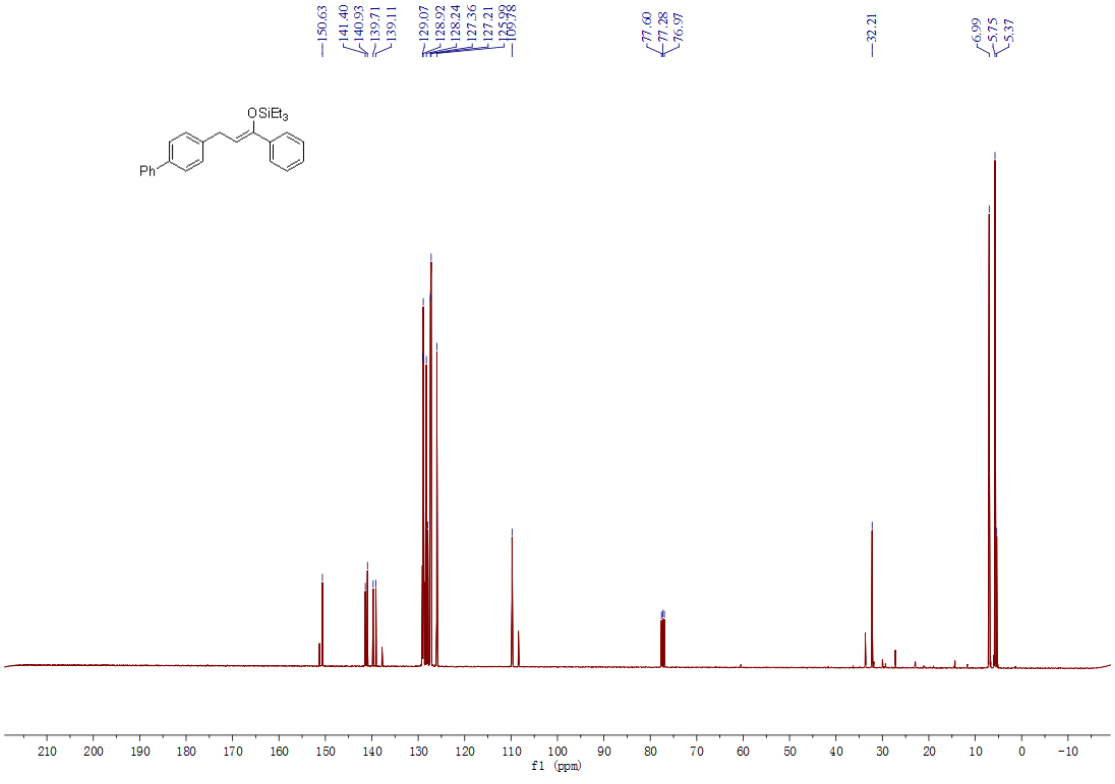


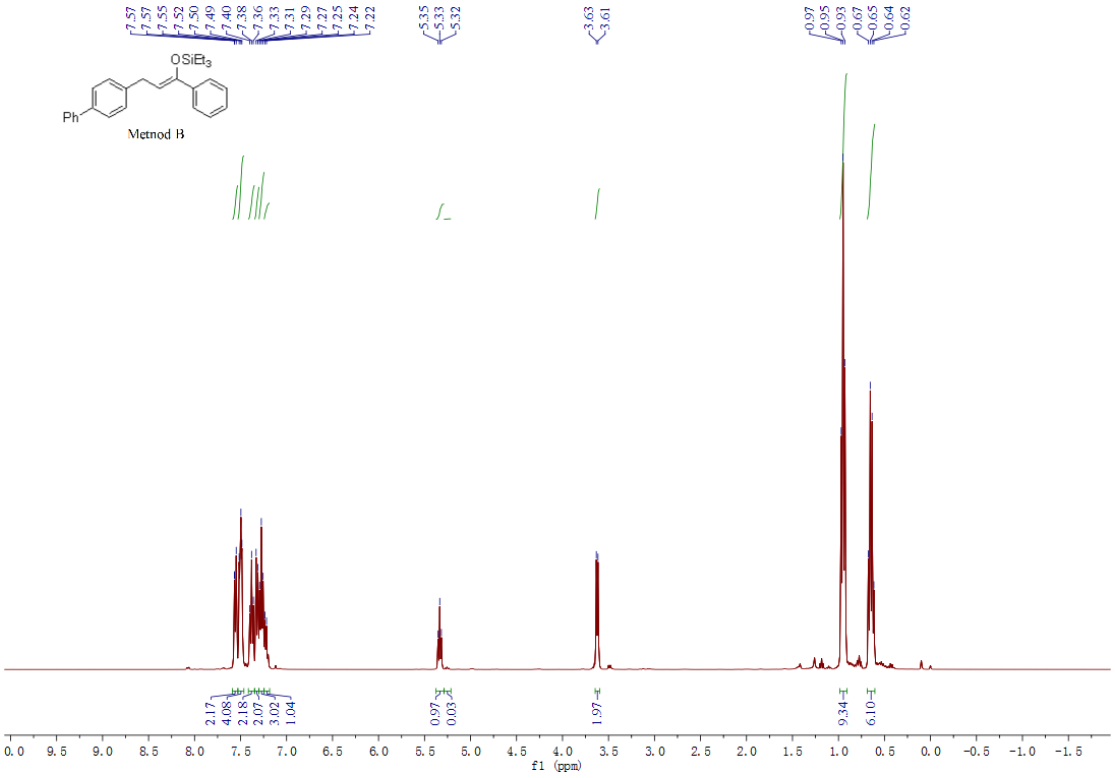
^{29}Si NMR (99 MHz, $CDCl_3$) δ 21.18 (s), 20.33 (s).

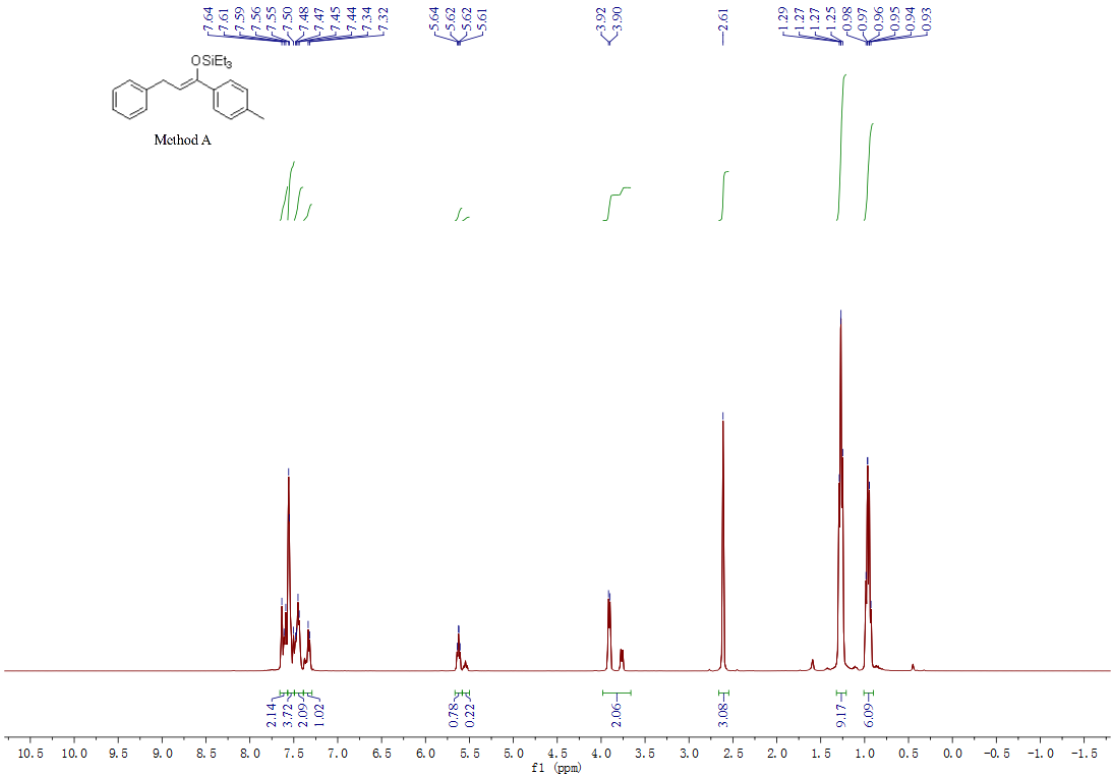


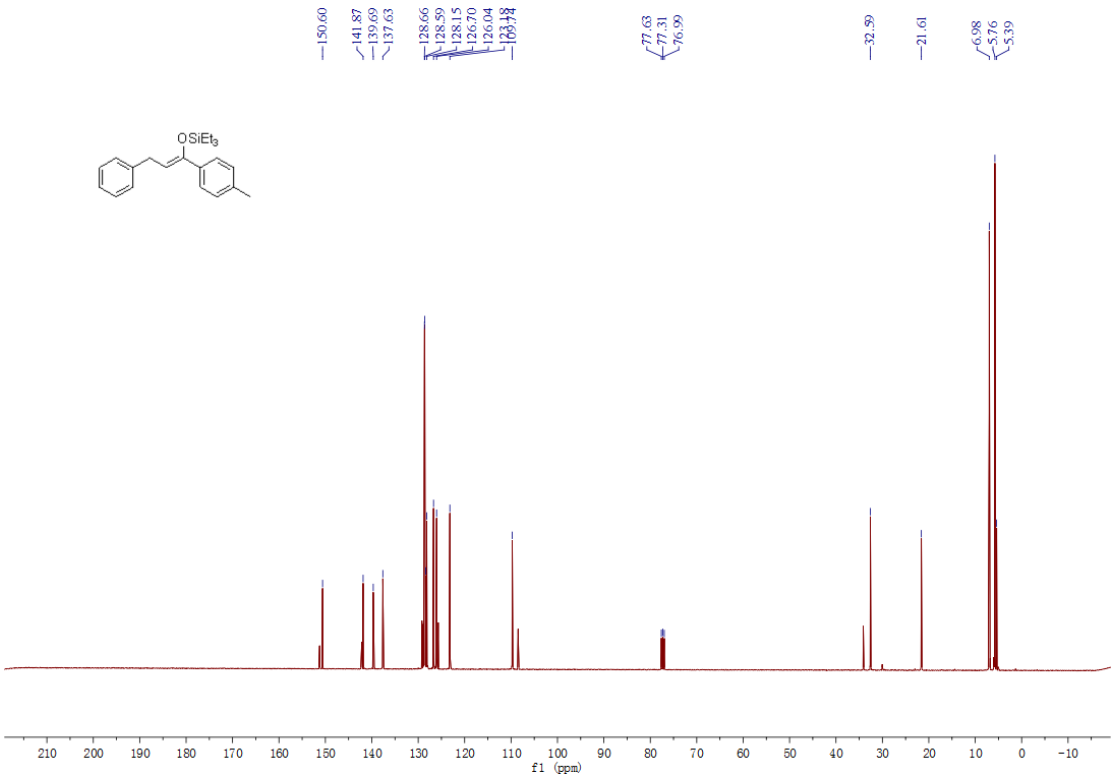
^{19}F NMR (471 MHz, $CDCl_3$) δ -113.26 (s), -114.29 (s).

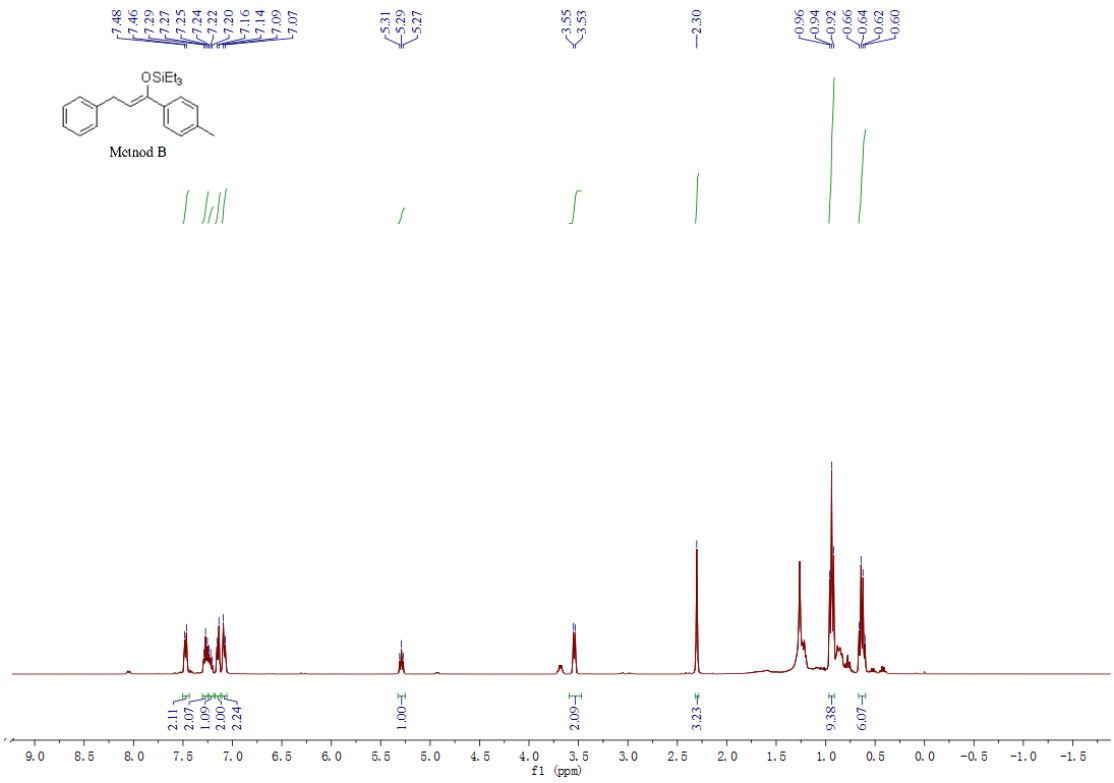

7.83
7.81
7.79
7.77
7.65
7.63
7.60
7.58
7.54
7.52
7.49
7.49
7.48
5.64
5.62
5.62
5.60
5.60
3.92
3.90
1.26
1.24
1.22
0.96
0.94
0.92
0.90
OSiEt3
Ph
Method A
2.11
4.05
3.90
4.09
0.79
0.20
2.02
9.00
6.01
f1 (ppm)

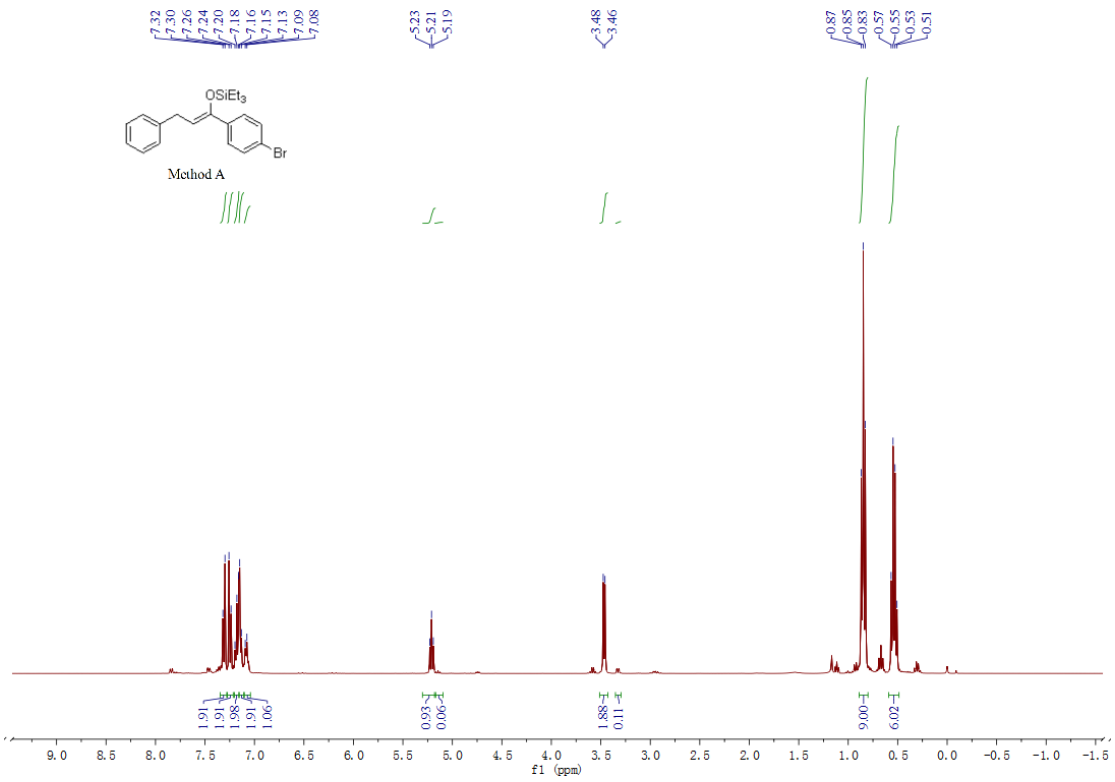

150.63
141.40
140.93
139.71
139.11
129.07
128.92
128.24
127.36
127.21
125.99
125.78
77.60
77.28
76.97
32.21
6.99
5.75
5.37
OSiEt3
Ph
f1 (ppm)

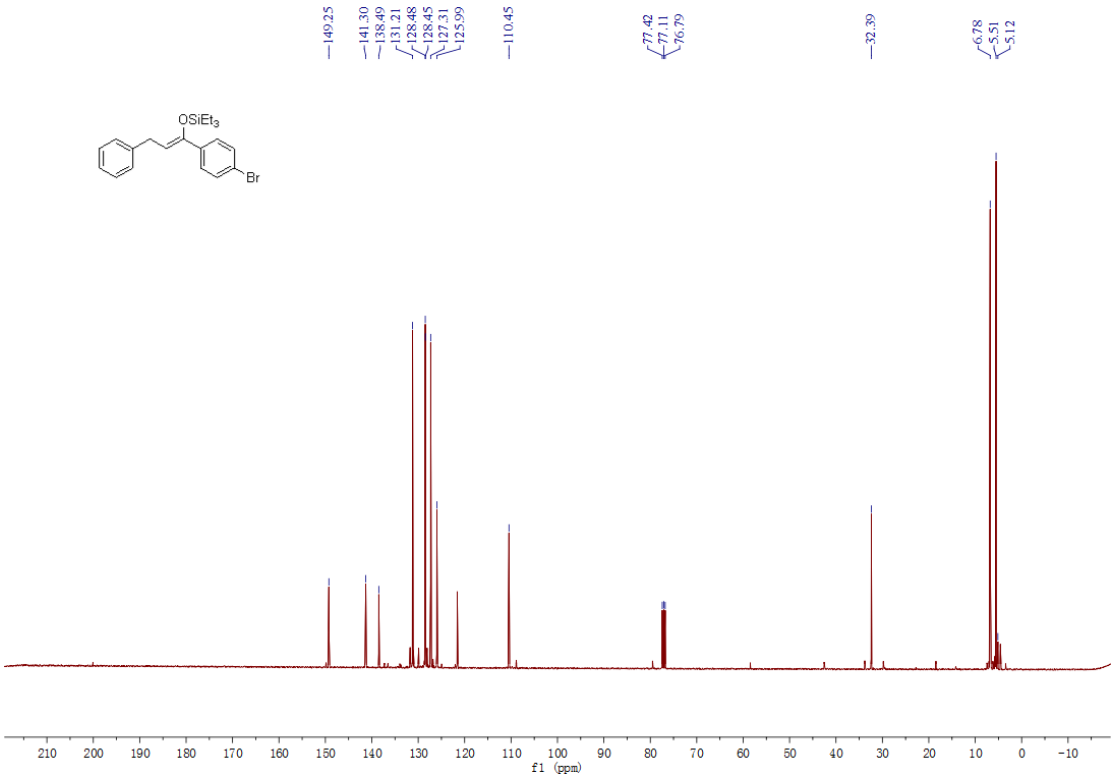

OSiEt3
Ph
Metnod B
f1 (ppm)

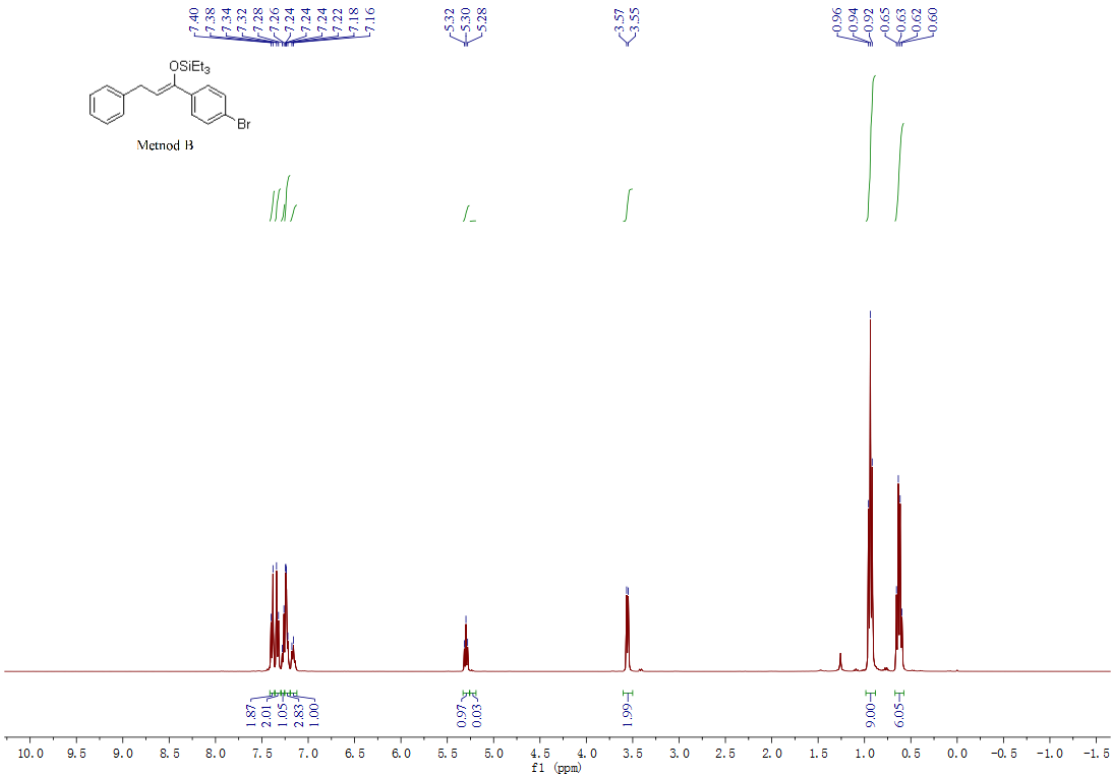

OSiEt3
Method A
f1 (ppm)


OSiEt3
150.60
141.87
139.69
137.63
128.66
128.59
128.15
126.70
126.04
77.63
77.31
76.99
32.59
21.61
6.98
5.76
5.39
f1 (ppm)

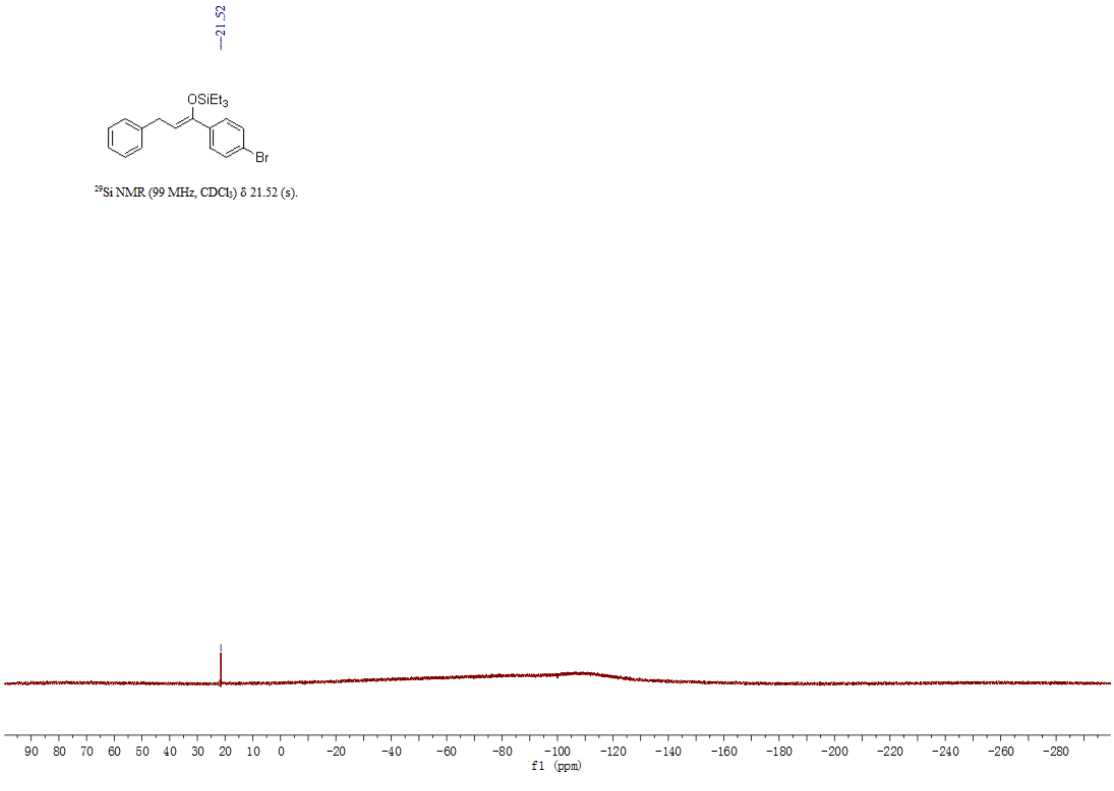

7.48
7.46
7.29
7.27
7.25
7.24
7.22
7.20
7.16
7.14
7.09
7.07
5.31
5.29
5.27
3.55
3.53
2.30
0.96
0.94
0.92
0.66
0.64
0.62
0.60
OSiEt3
Metnod B
2.11
2.07
1.09
2.00
2.24
1.00
2.09
3.23
9.38
6.07
f1 (ppm)


7.32
7.30
7.26
7.24
7.20
7.18
7.16
7.15
7.13
7.09
7.08
5.23
5.21
5.19
3.48
3.46
0.87
0.85
0.83
0.57
0.55
0.53
0.51
OSiEt3
Br
Method A
1.91
1.91
1.98
1.91
1.06
0.93
0.06
1.88
0.11
9.00
6.02
f1 (ppm)

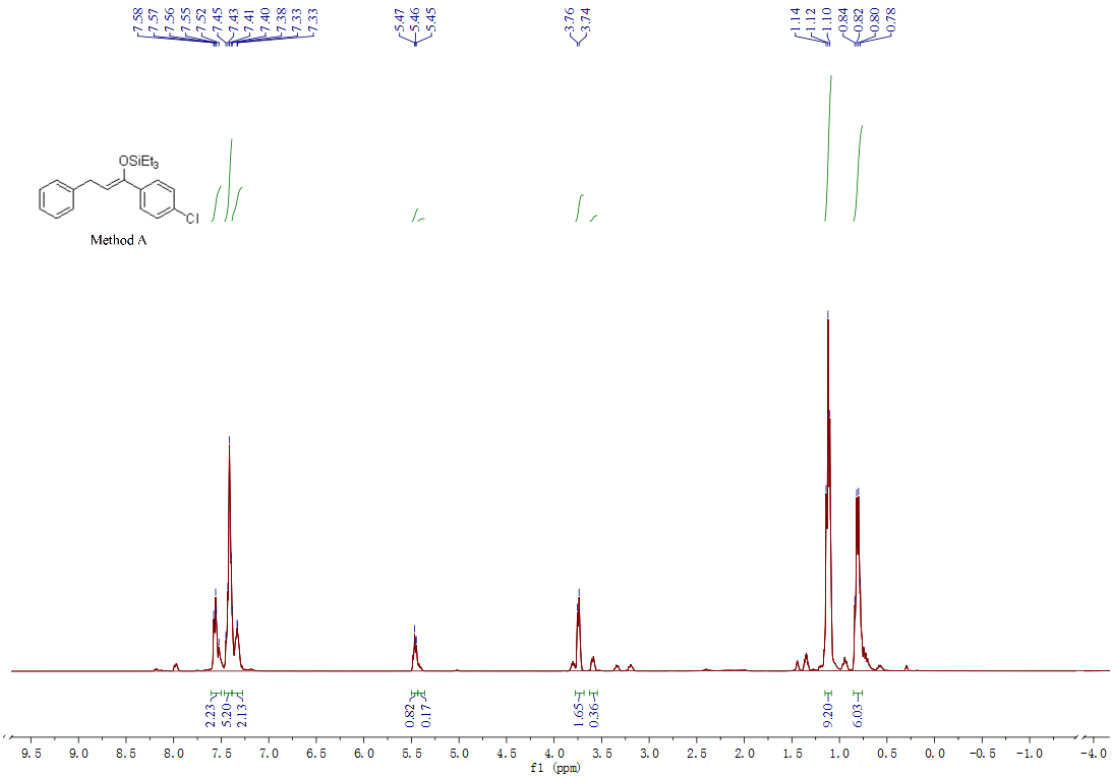

149.25
141.30
138.49
131.21
128.48
128.45
127.31
125.99
110.45
77.42
77.11
76.79
32.39
6.78
5.51
5.12
OSiEt3
Br
f1 (ppm)

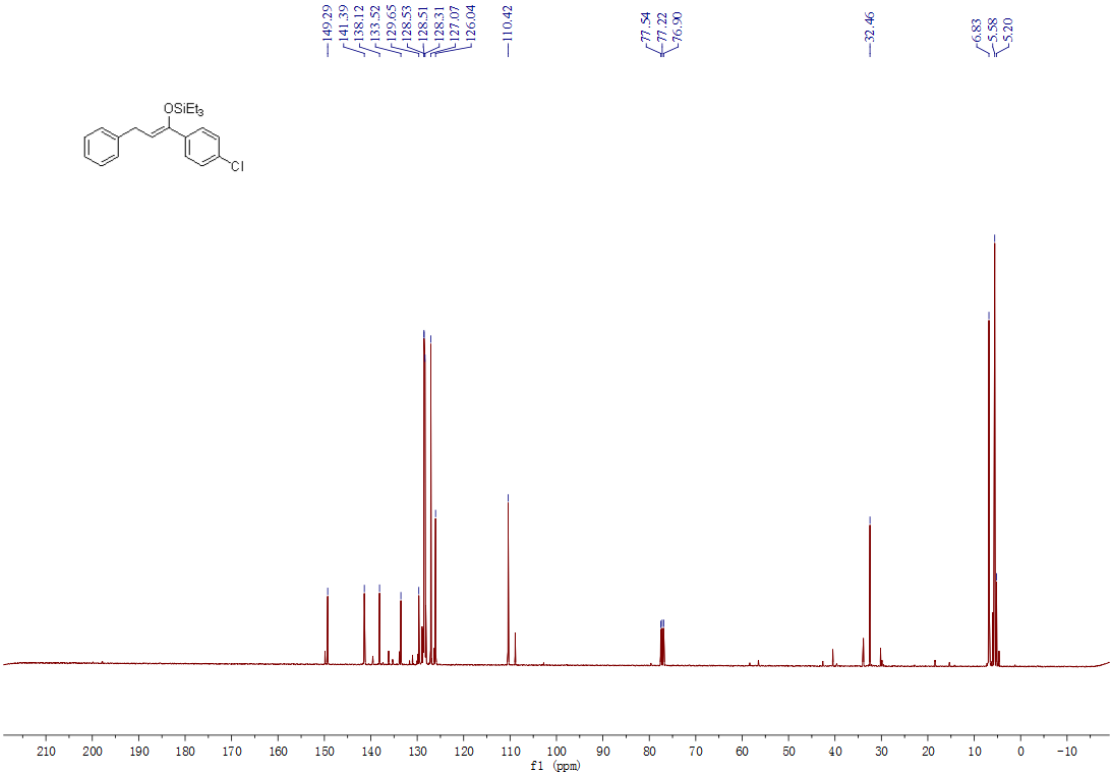


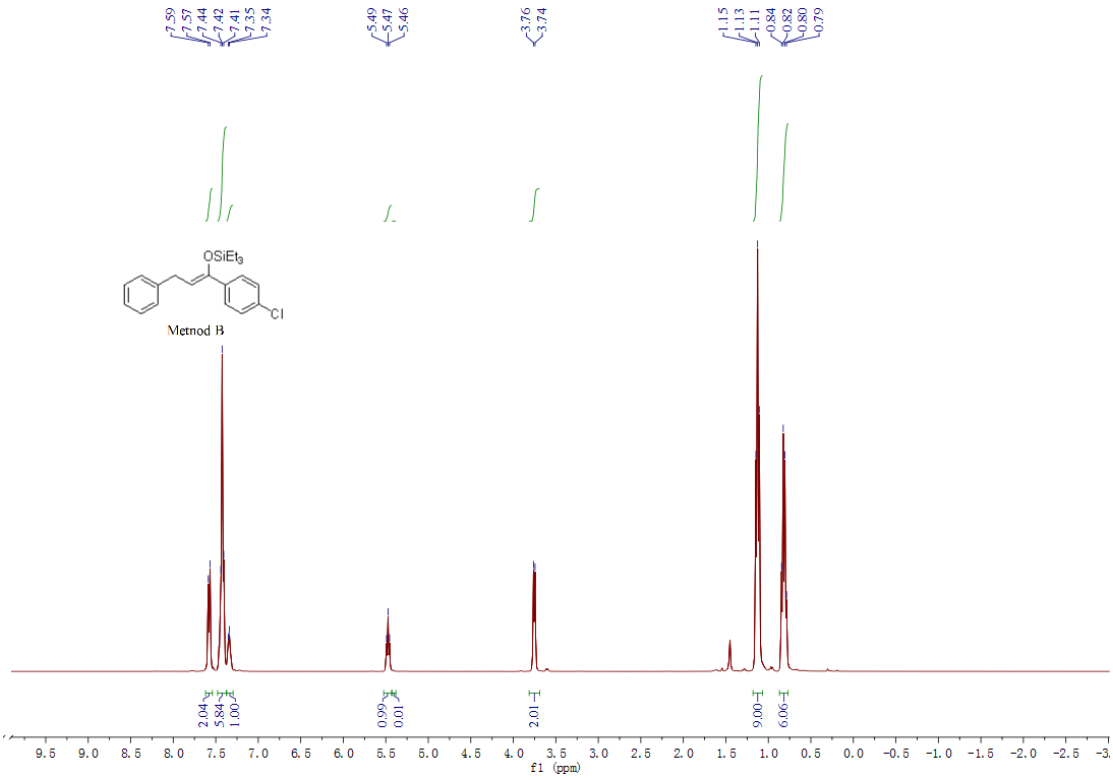
Metnod B

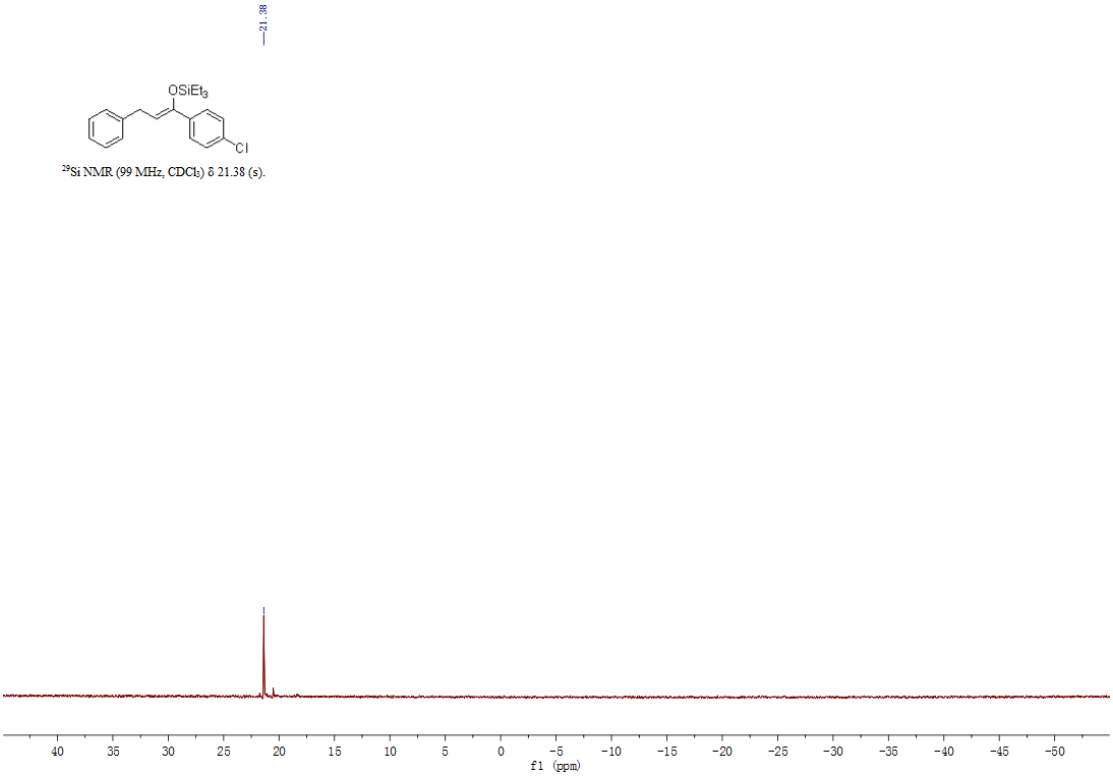


^{29}Si NMR (99 MHz, $CDCl_3$) δ 21.52 (s).

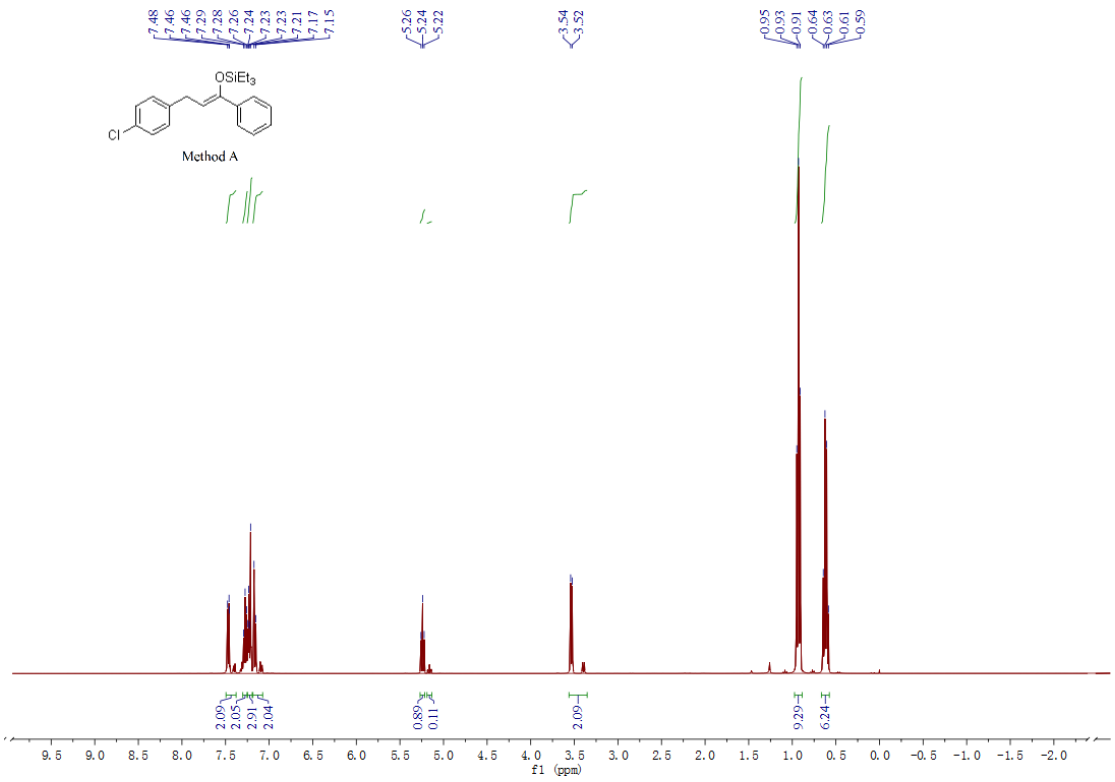

7.58
7.57
7.56
7.55
7.52
7.45
7.43
7.41
7.40
7.38
7.33
7.33
5.47
5.46
5.45
3.76
3.74
1.14
1.12
1.10
0.84
0.82
0.80
0.78
OSiEt3
Cl
Method A
2.23
5.20
2.13
0.82
0.17
1.65
0.36
9.20
6.03
f1 (ppm)

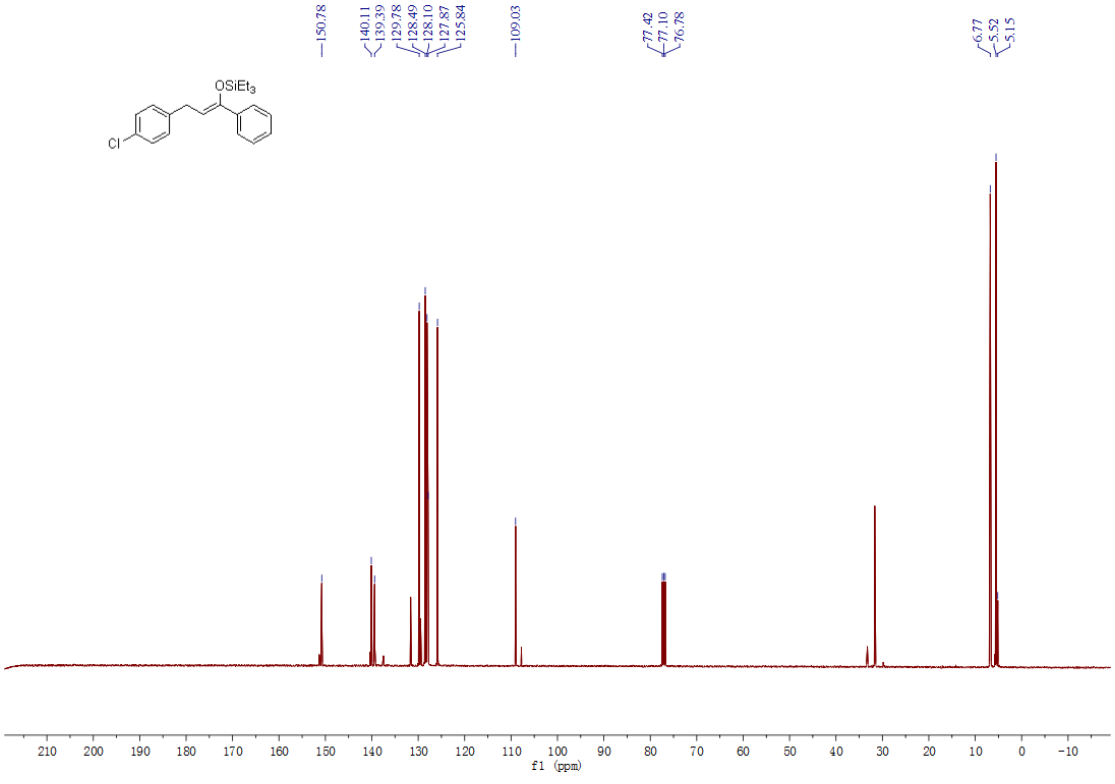

149.29
141.39
138.12
133.52
129.65
128.53
128.51
128.31
127.07
126.04
110.42
77.54
77.22
76.90
32.46
6.83
5.58
5.20
OSiEt3
Cl
f1 (ppm)

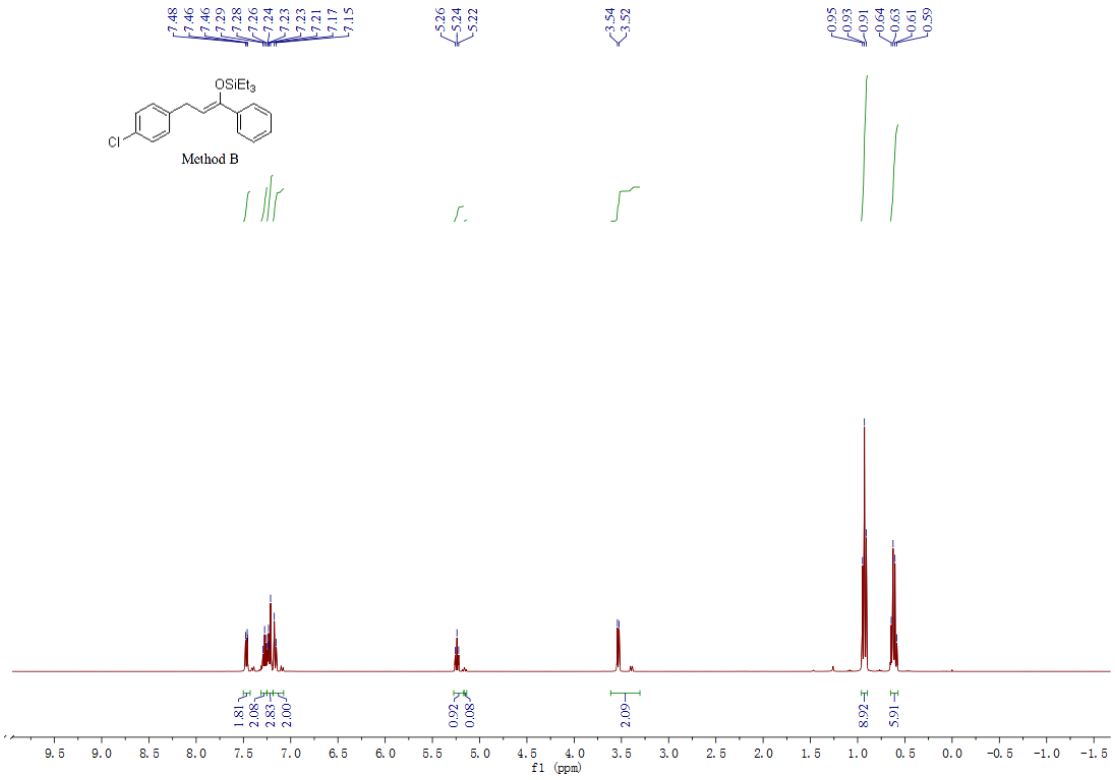


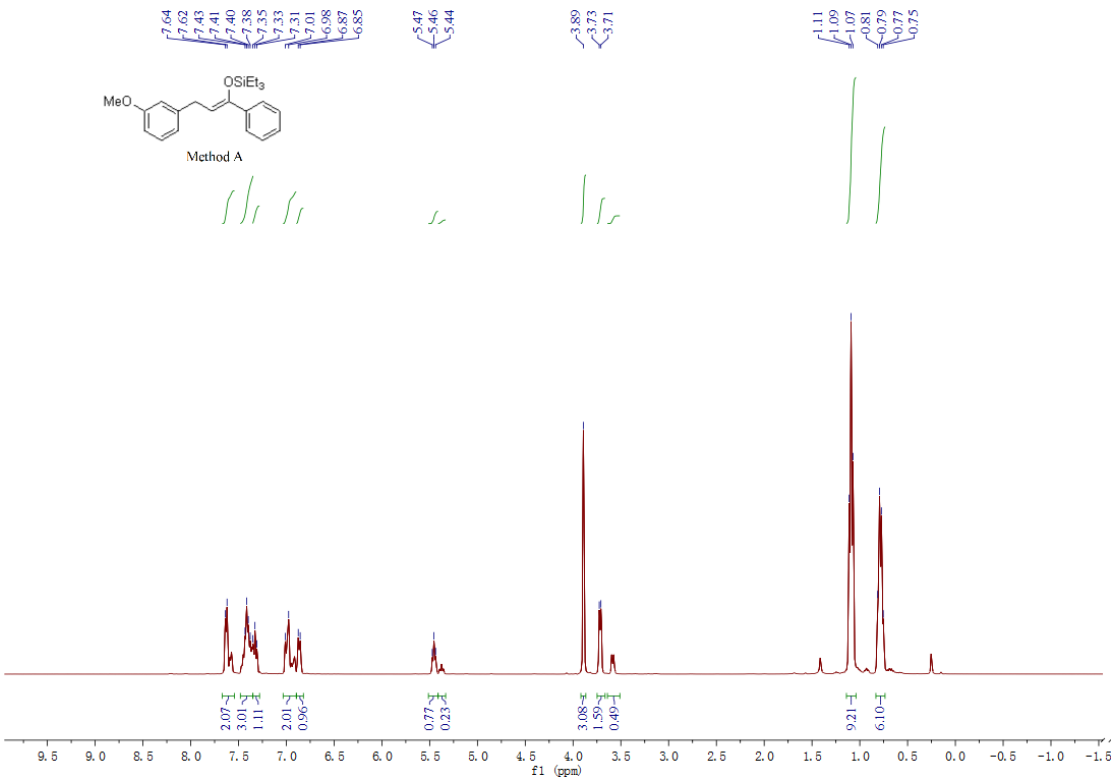


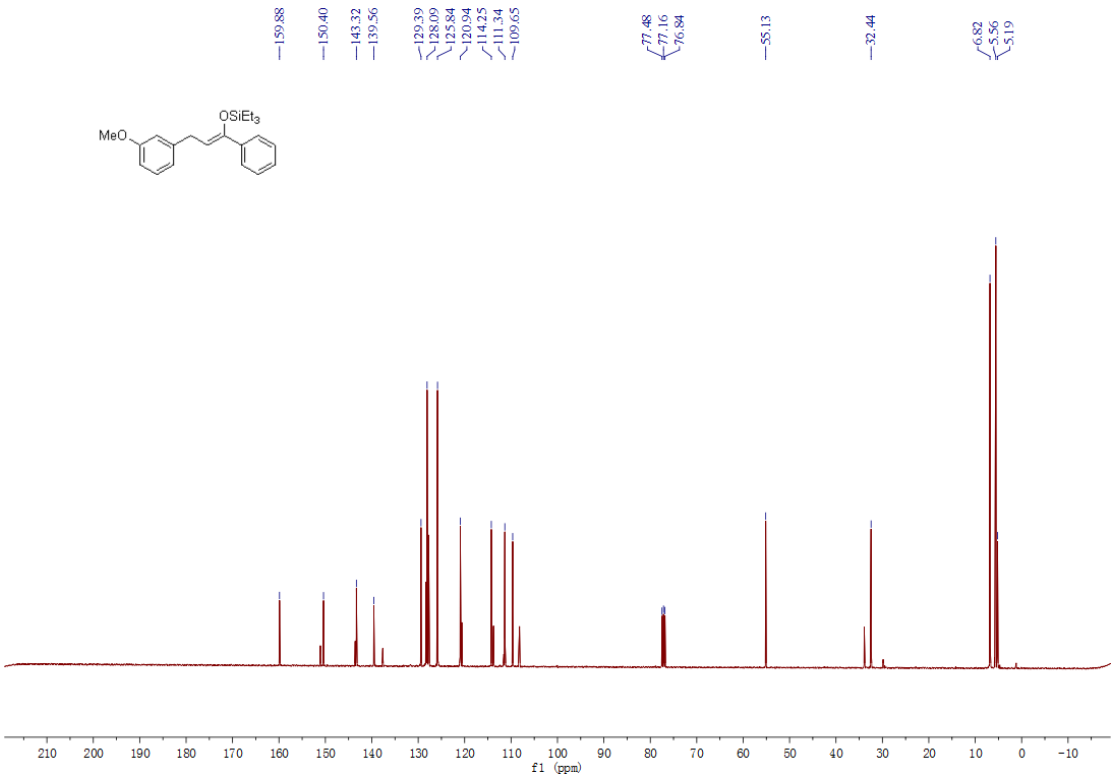
^{29}Si NMR (99 MHz, $CDCl_3$) δ 21.38 (s).

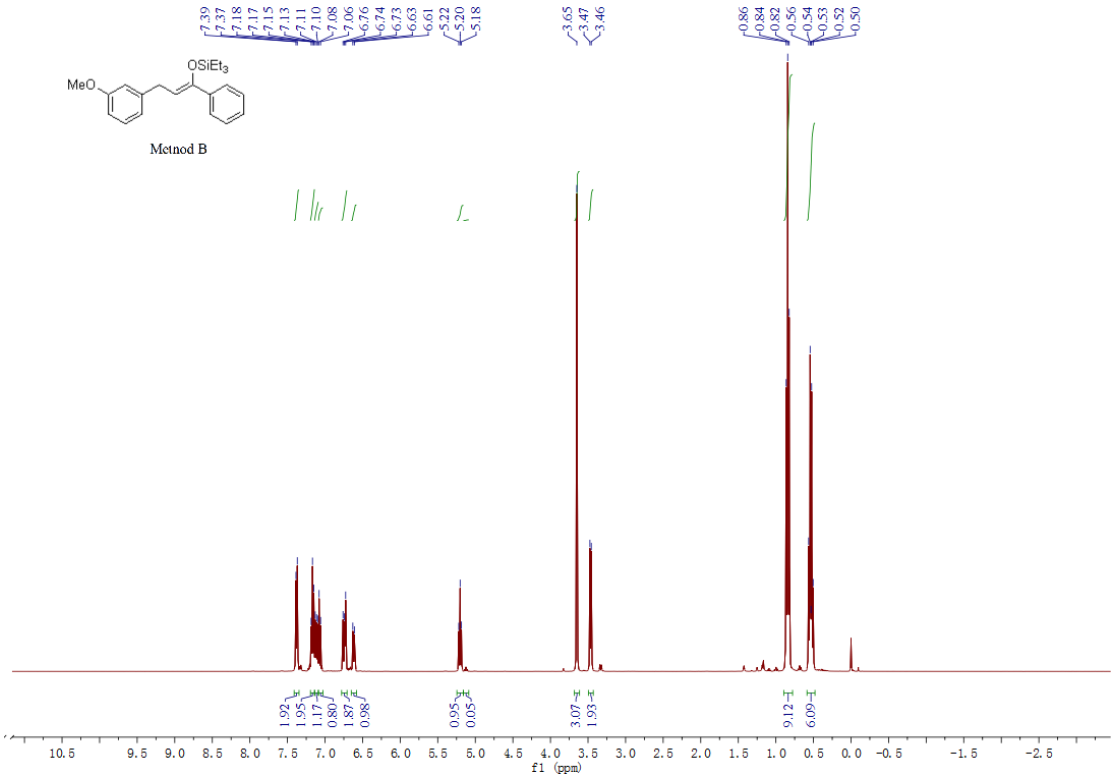

7.48
7.46
7.46
7.29
7.28
7.26
7.24
7.23
7.23
7.21
7.17
7.15
5.26
5.24
5.22
3.54
3.52
0.95
0.93
0.91
0.64
0.63
0.61
0.59
OSiEt3
Cl
Method A
2.09
2.05
2.91
2.04
0.89
0.11
2.09
9.29
6.24
f1 (ppm)

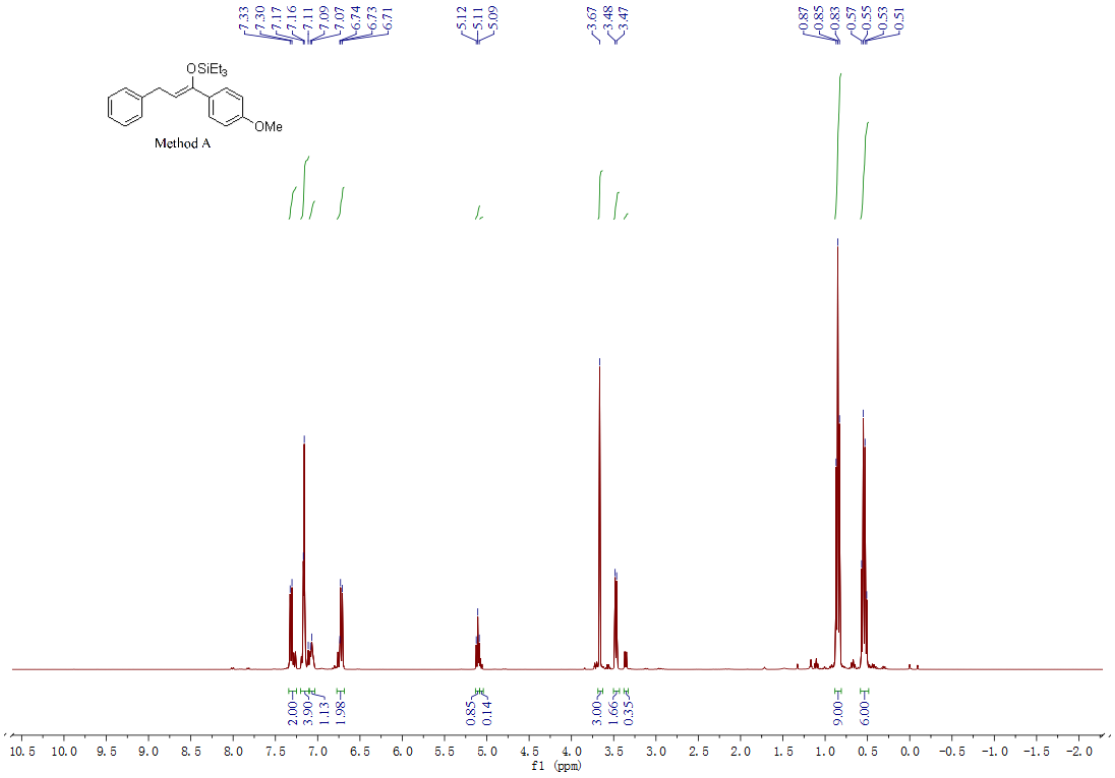

150.78
140.11
139.39
129.78
128.49
128.10
127.87
125.84
109.03
77.42
77.10
76.78
6.77
5.52
5.15
OSiEt3
Cl
f1 (ppm)

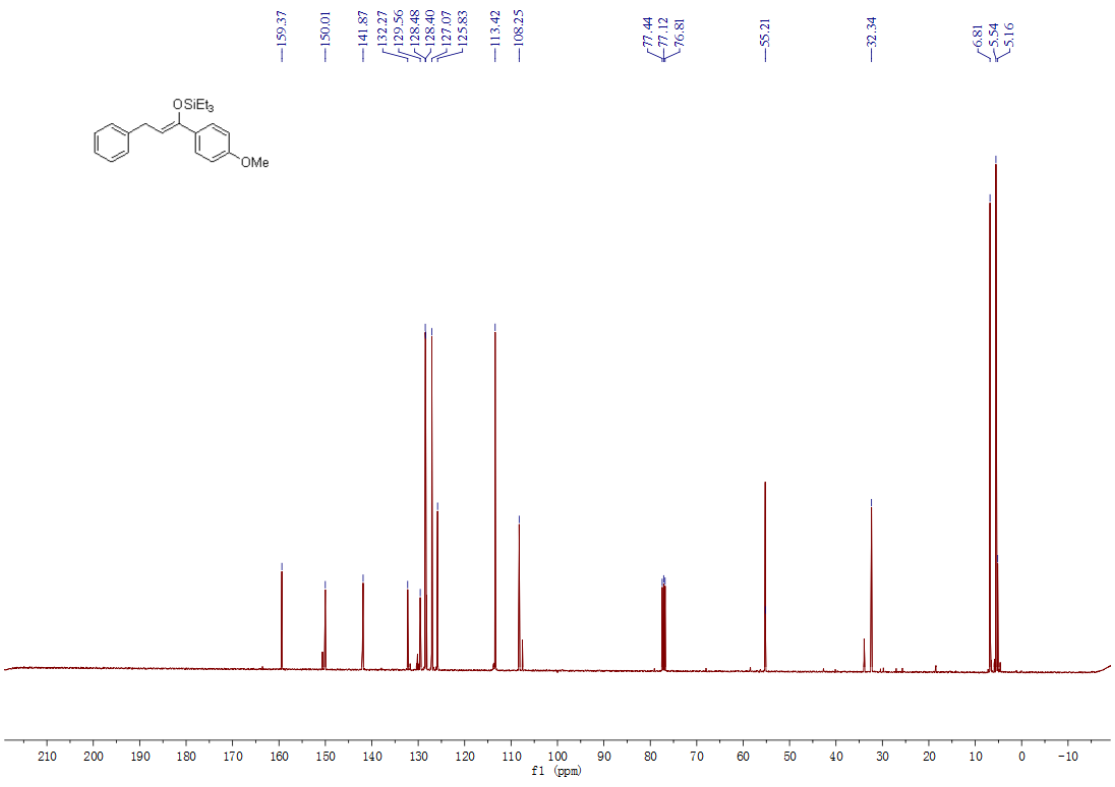

OSiEt3
Cl
Method B
f1 (ppm)

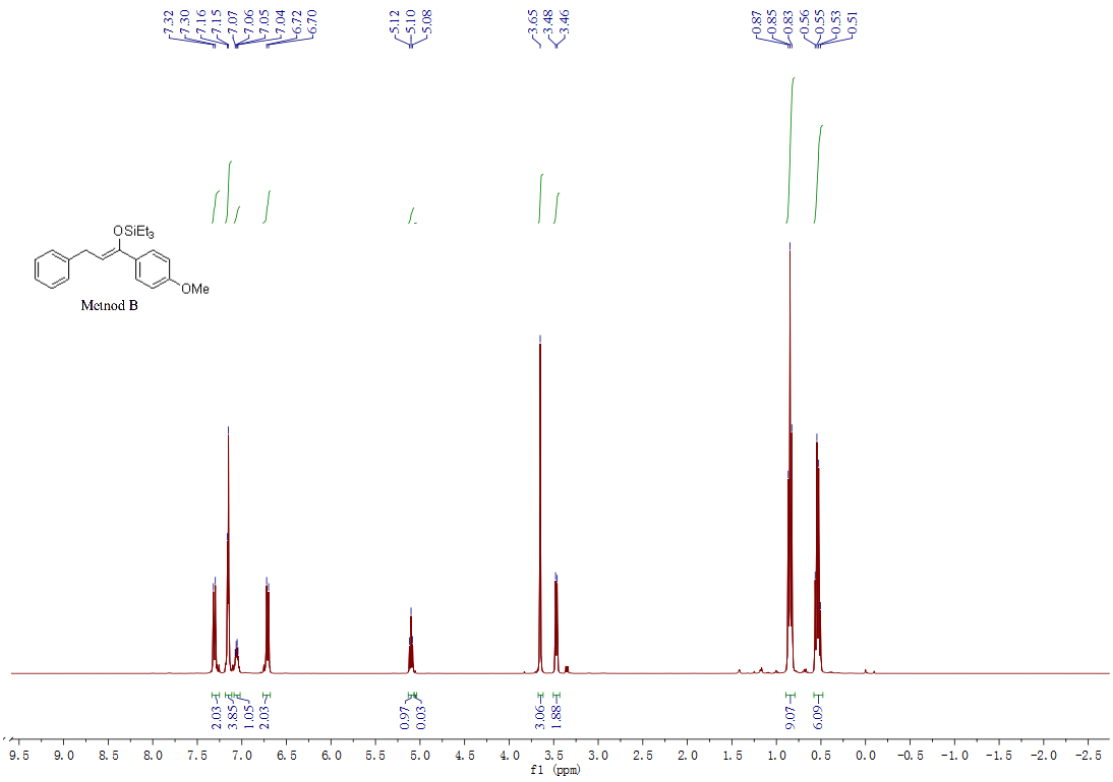

OSiEt3
MeO
Method A
f1 (ppm)

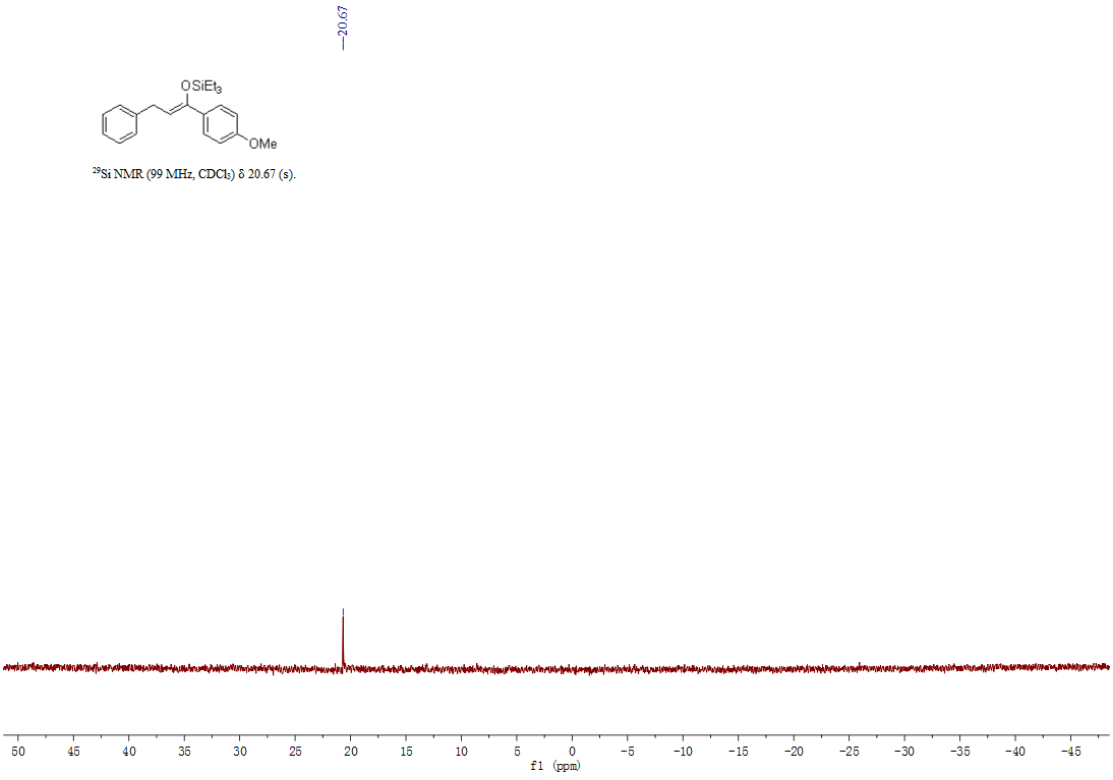

159.88
150.40
143.32
139.56
129.39
128.09
125.84
120.94
114.25
111.34
109.65
77.48
77.16
76.84
55.13
32.44
6.82
5.56
5.19
MeO
OSiEt3
f1 (ppm)


7.39
7.37
7.18
7.17
7.15
7.13
7.11
7.10
7.08
7.06
6.76
6.74
6.73
6.63
6.61
5.22
5.20
5.18
3.65
3.47
3.46
0.86
0.84
0.82
0.56
0.54
0.53
0.52
0.50
MeO
OSiEt3
Metnod B
1.92
1.95
1.17
0.80
1.87
0.98
0.95
0.05
3.07
1.93
9.12
6.09
f1 (ppm)

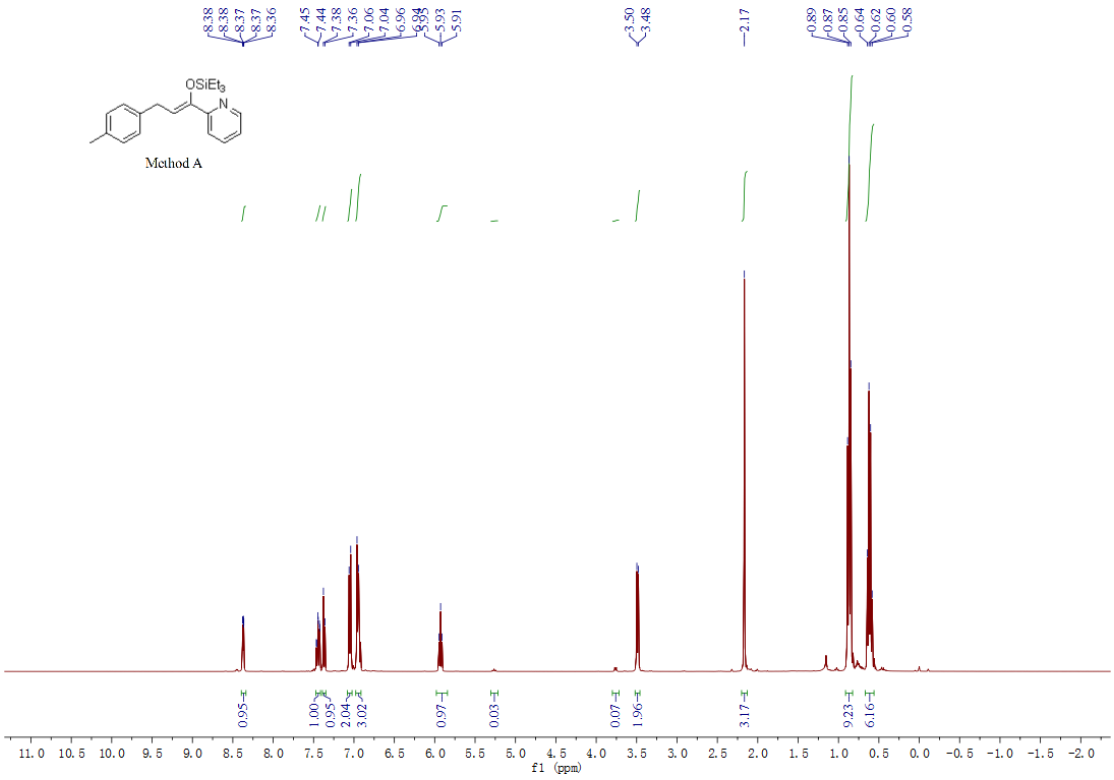

OSiEt3
OMe
Method A
7.33
7.30
7.17
7.16
7.11
7.09
7.07
6.74
6.73
6.71
5.12
5.11
5.09
3.67
3.48
3.47
0.87
0.85
0.83
0.57
0.55
0.53
0.51
2.00
3.90
1.13
1.98
0.85
0.14
3.00
1.66
0.35
9.00
6.00
f1 (ppm)

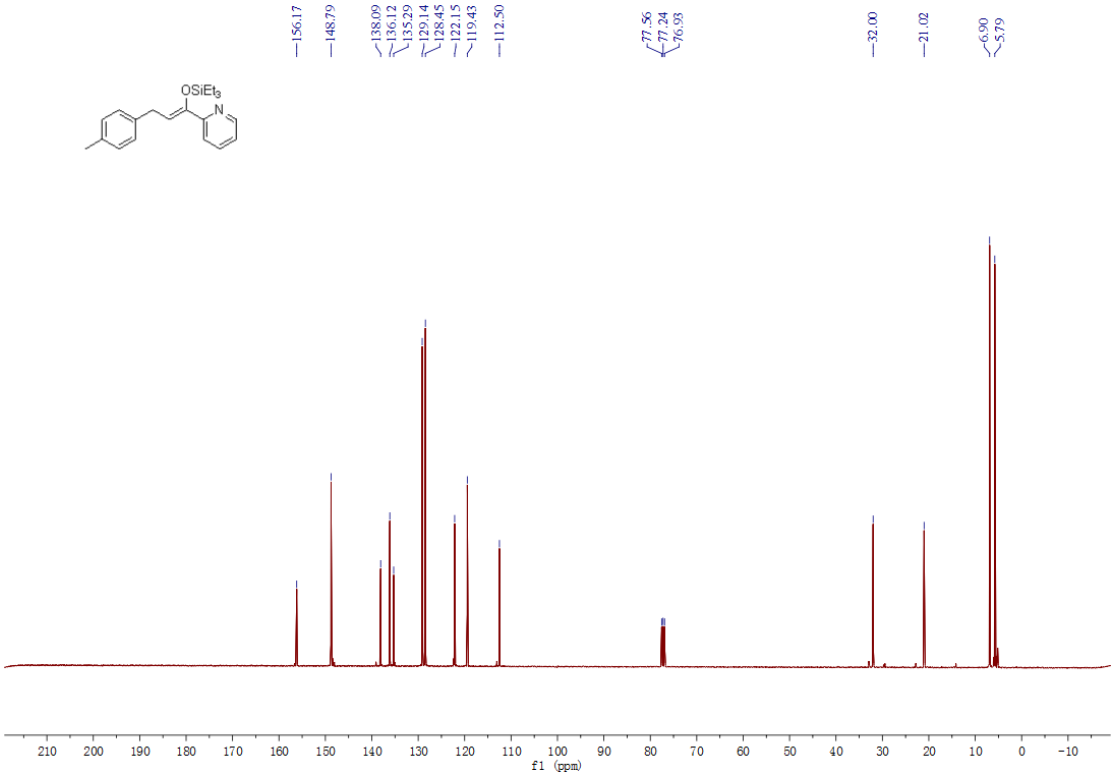

OSiEt3
OMe
159.37
150.01
141.87
132.27
129.56
128.48
128.40
127.07
125.83
113.42
108.25
77.44
77.12
76.81
55.21
32.34
6.81
5.54
5.16
f1 (ppm)

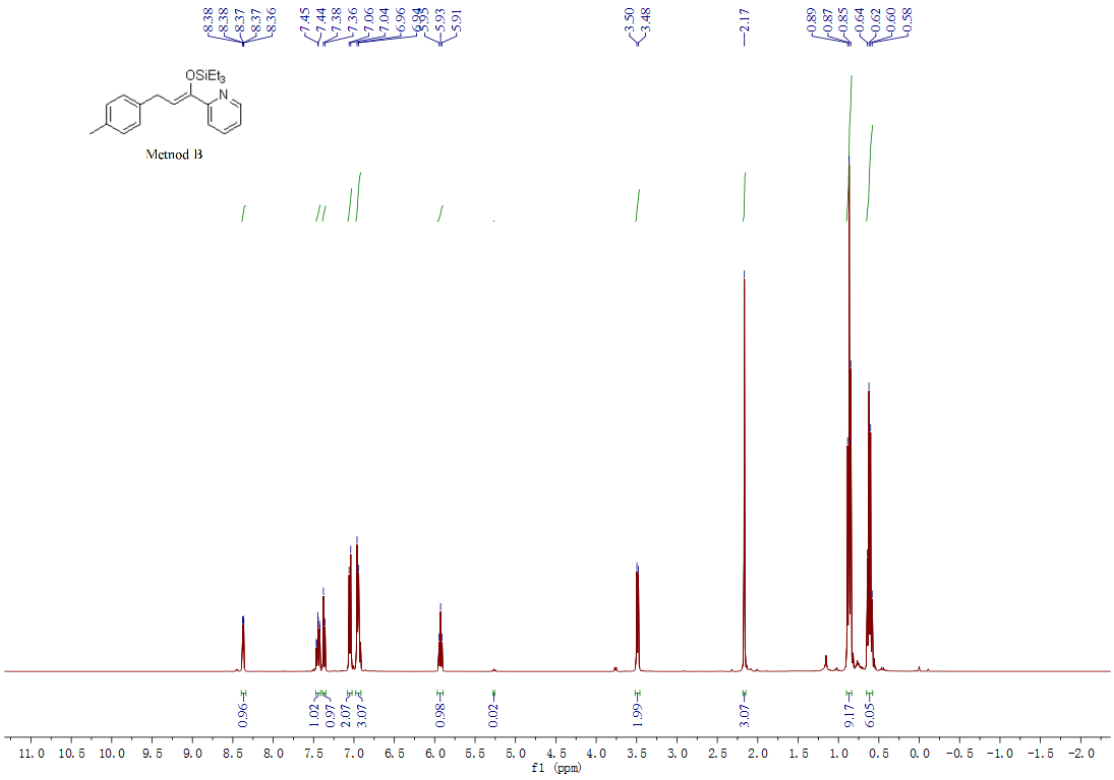


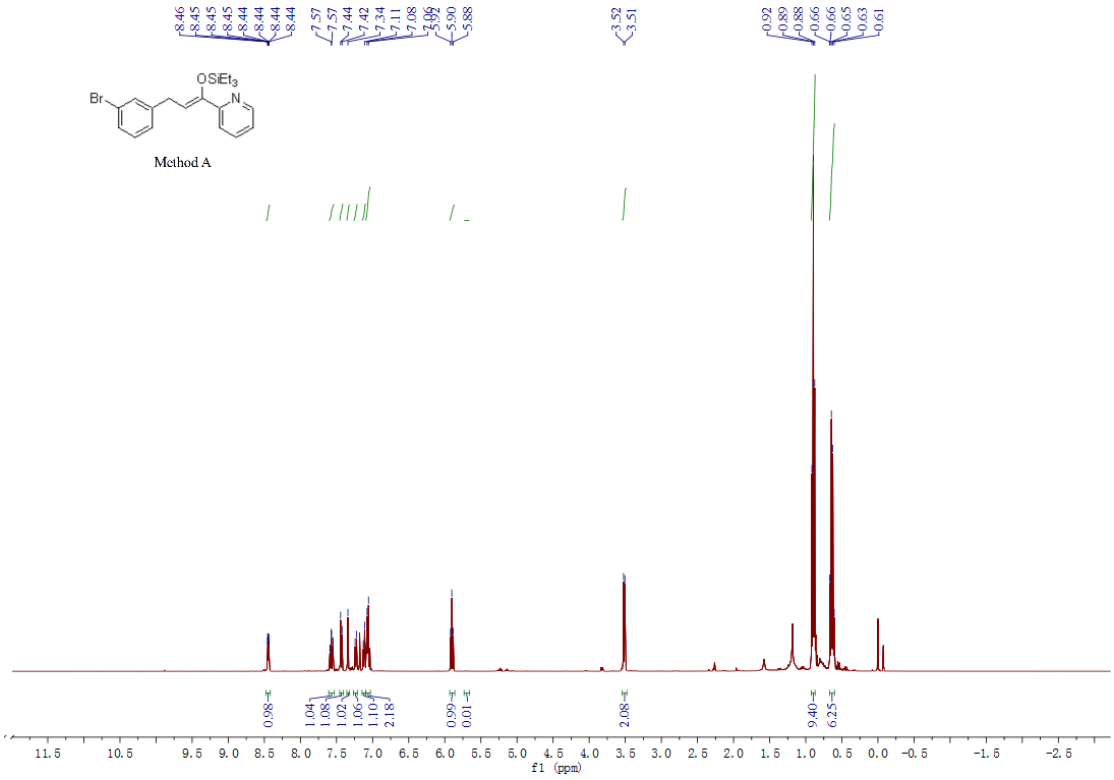


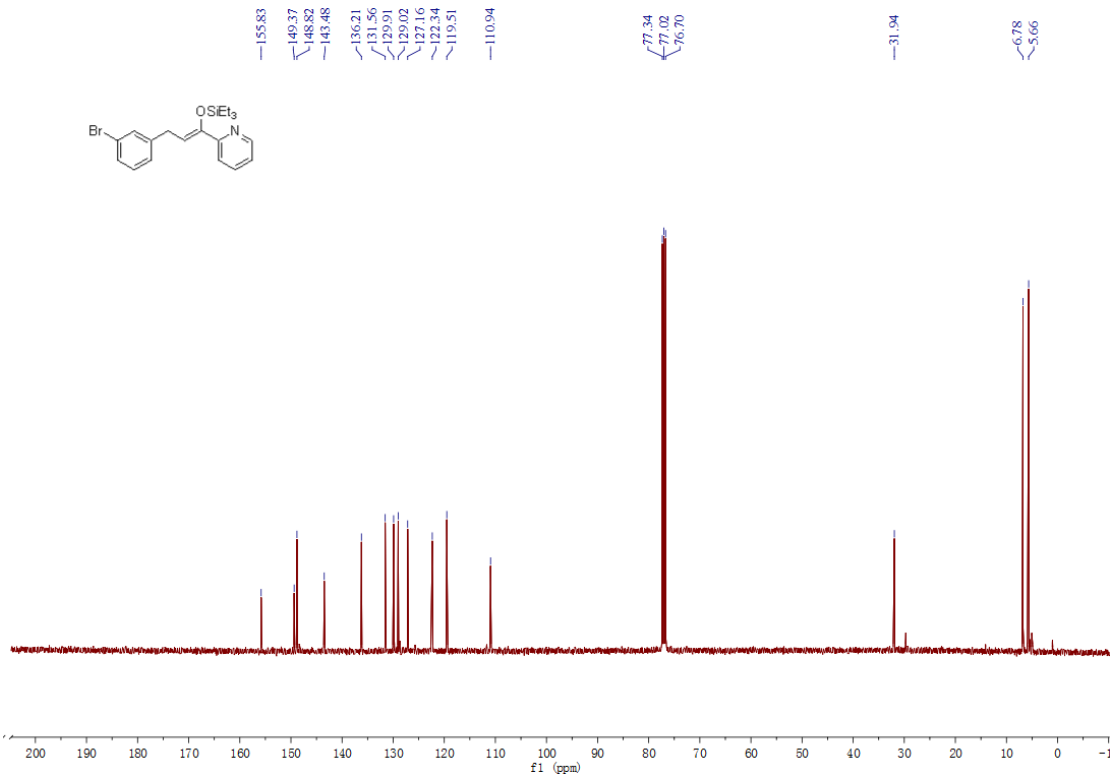
^{29}Si NMR (99 MHz, $CDCl_3$) δ 20.67 (s).

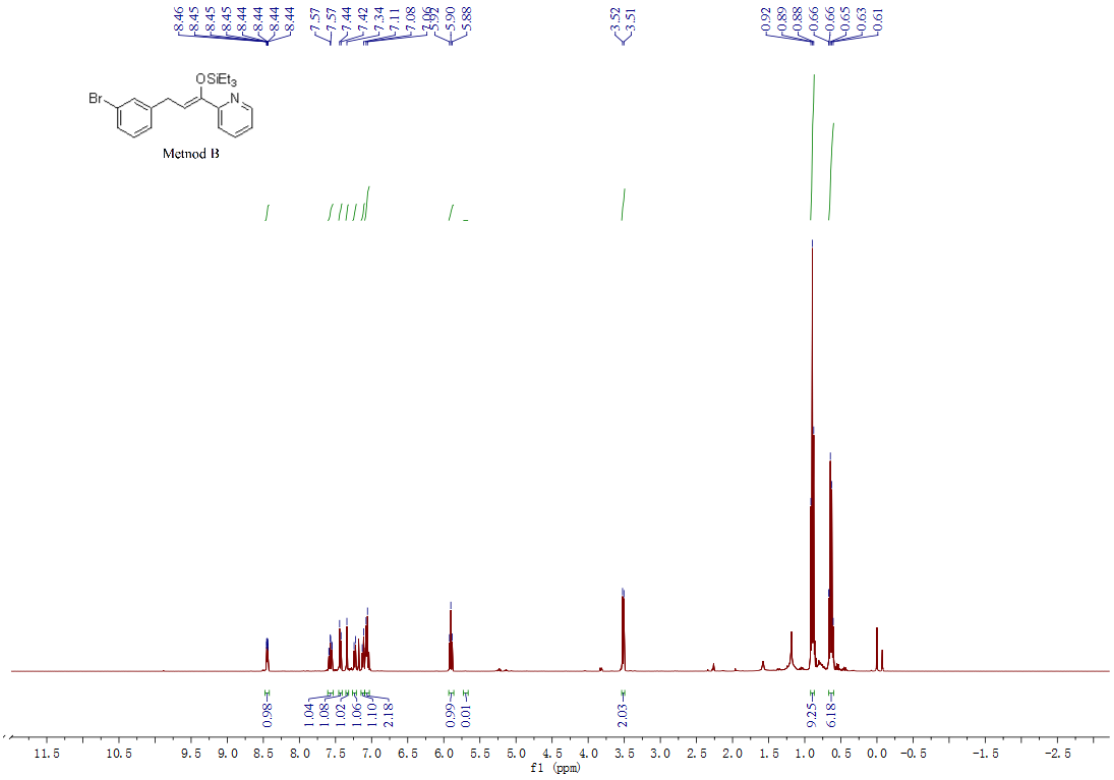

8.38
8.38
8.37
8.37
8.36
7.45
7.44
7.38
7.36
7.06
7.04
6.96
6.94
5.93
5.91
3.50
3.48
2.17
0.89
0.87
0.85
0.64
0.62
0.60
0.58
OSiEt3
N
Method A
0.95
1.00
0.95
2.04
3.02
0.97
0.03
0.07
1.96
3.17
9.23
6.16
f1 (ppm)

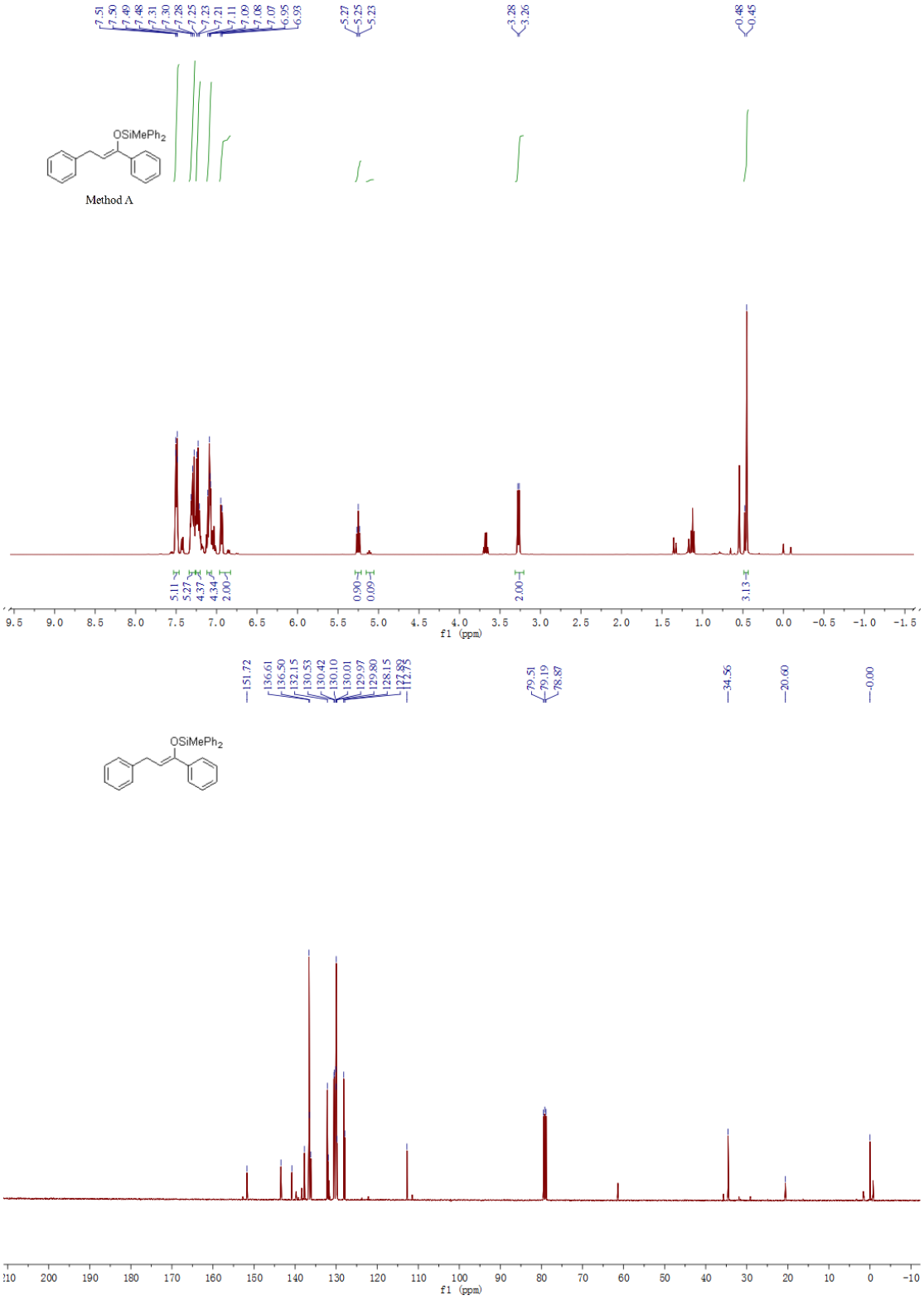

156.17
148.79
138.09
136.12
135.29
129.14
128.45
122.15
119.43
112.50
77.56
77.24
76.93
32.00
21.02
6.90
5.79
OSiEt3
N
f1 (ppm)

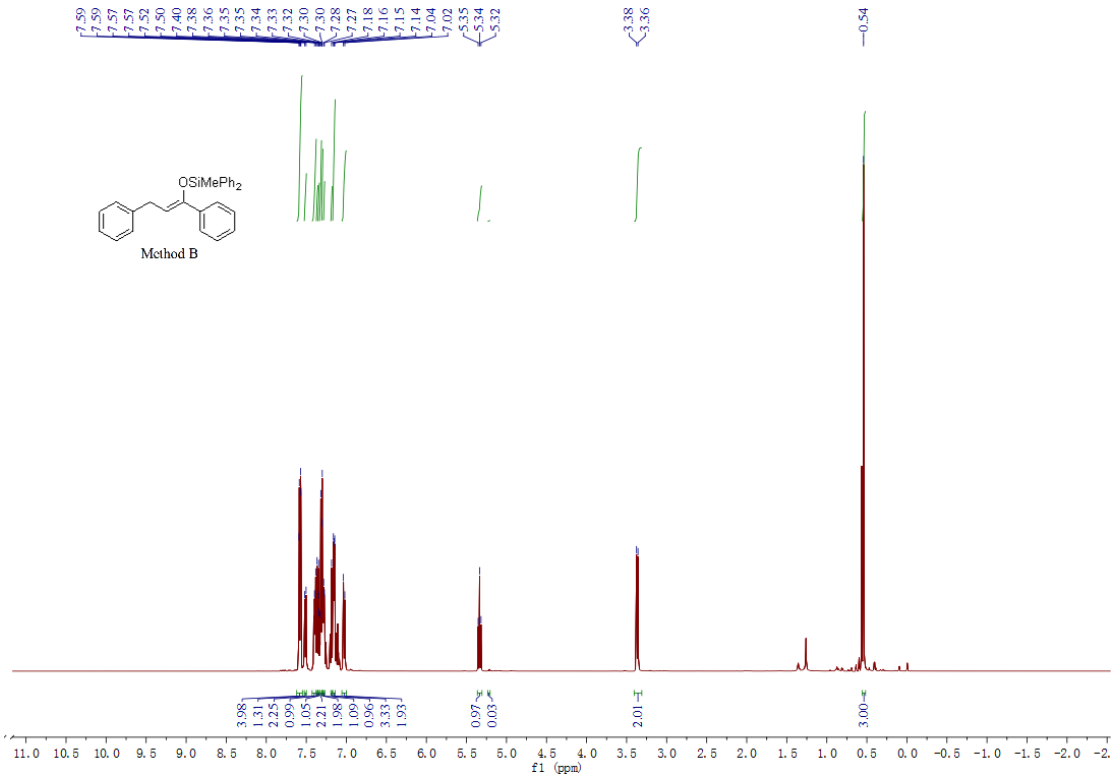

OSiEt3
N
Metnod B
8.38
8.38
8.37
8.37
8.36
7.45
7.44
7.38
7.36
7.06
7.04
6.96
5.93
5.91
3.50
3.48
2.17
0.89
0.87
0.85
0.64
0.62
0.60
0.58
0.96
1.02
0.97
2.07
3.07
0.98
0.02
1.99
3.07
9.17
6.05
f1 (ppm)

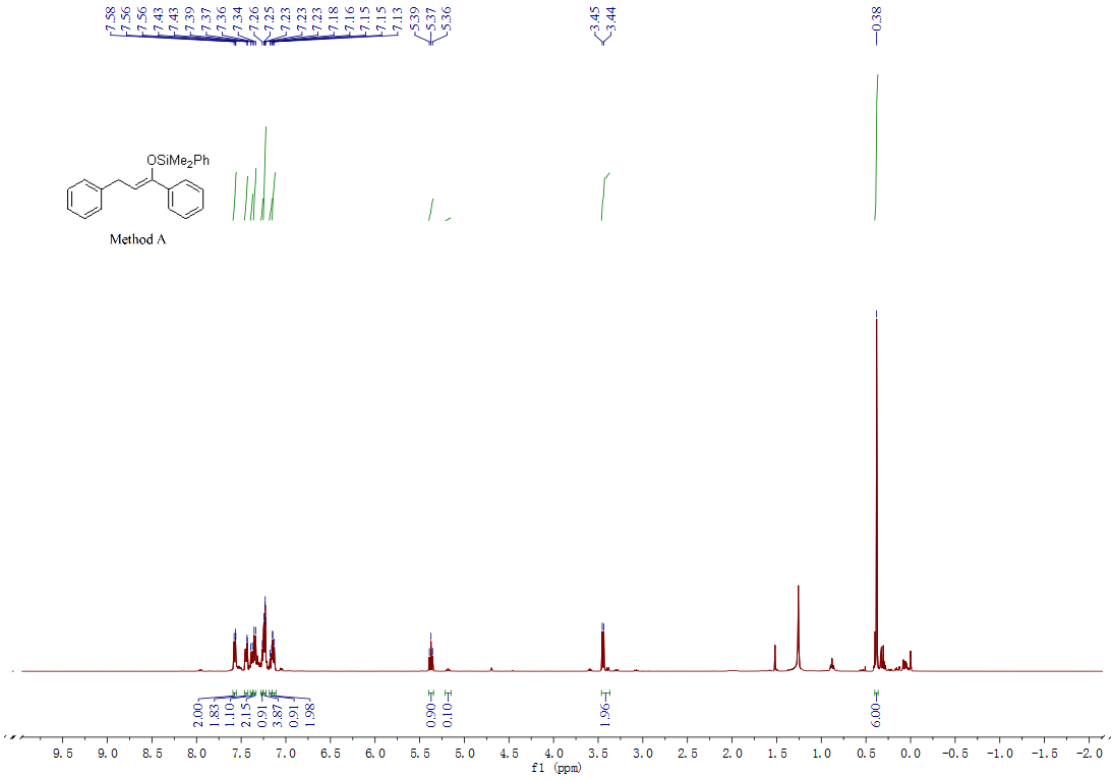

OSiEt3
Br
N
Method A
8.46
8.45
8.45
8.45
8.44
8.44
8.44
8.44
7.57
7.57
7.44
7.42
7.34
7.11
7.08
5.90
5.888
3.52
3.51
0.92
0.89
0.88
0.66
0.65
0.63
0.61
0.98
1.04
1.08
1.02
1.06
1.10
2.18
0.99
0.01
2.08
9.40
6.25
f1 (ppm)

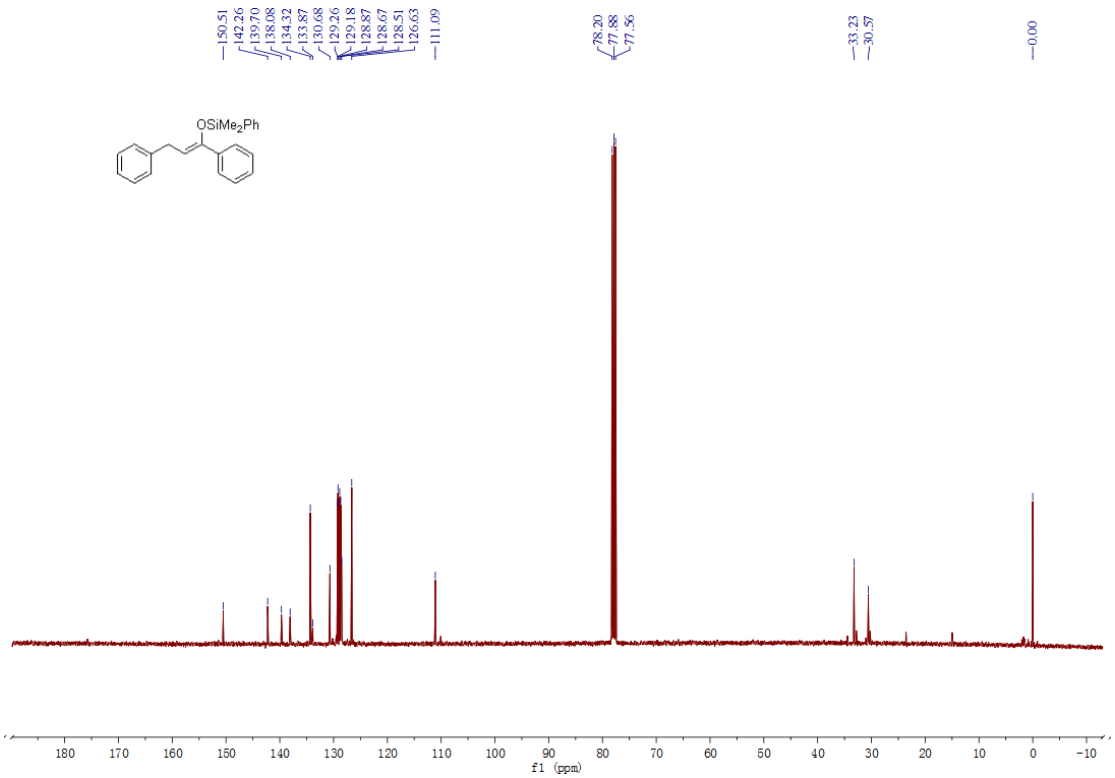

155.83
149.37
148.82
143.48
136.21
131.56
129.91
129.02
127.16
122.34
119.51
110.94
77.34
77.02
76.70
31.94
6.78
5.66
OSiEt3
Br
N
f1 (ppm)

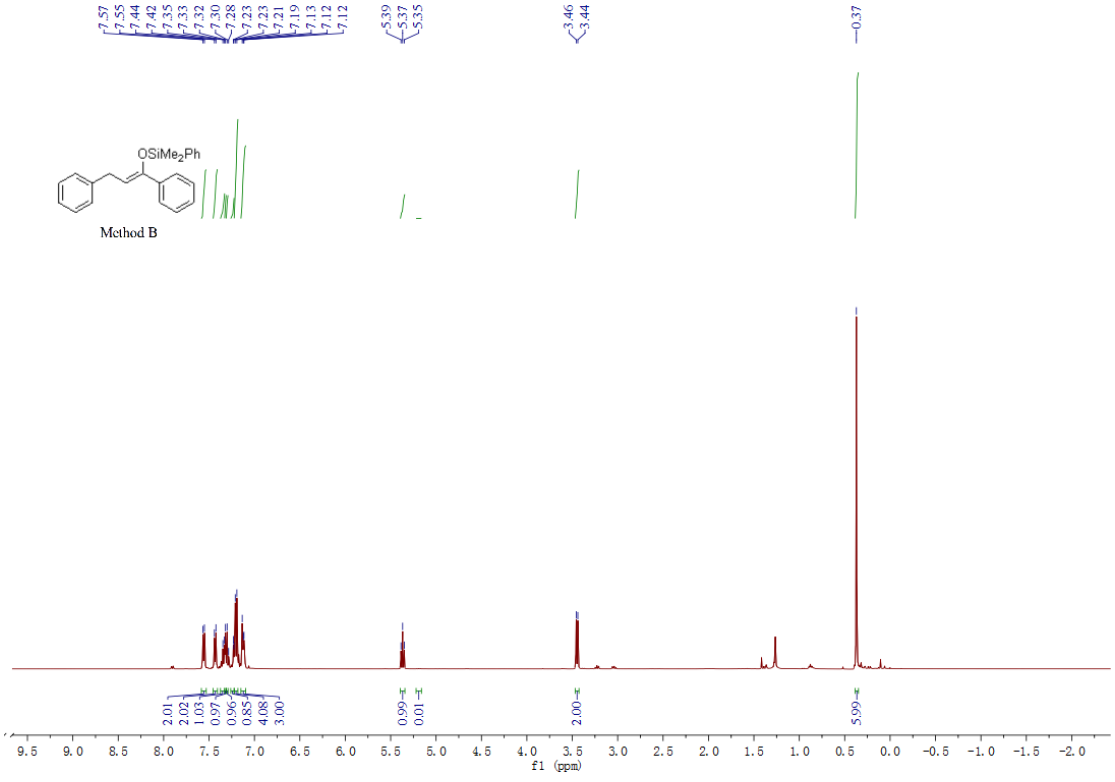

8.46
8.45
8.45
8.45
8.44
8.44
8.44
8.44
7.57
7.57
7.44
7.42
7.34
7.11
7.08
5.90
5.888
3.52
3.51
0.92
0.89
0.88
0.66
0.66
0.65
0.63
0.61
OSiEt3
Br
N
Metnod B
0.98
1.04
1.08
1.02
1.06
1.10
2.18
0.99
0.01
2.03
9.25
6.18
f1 (ppm)

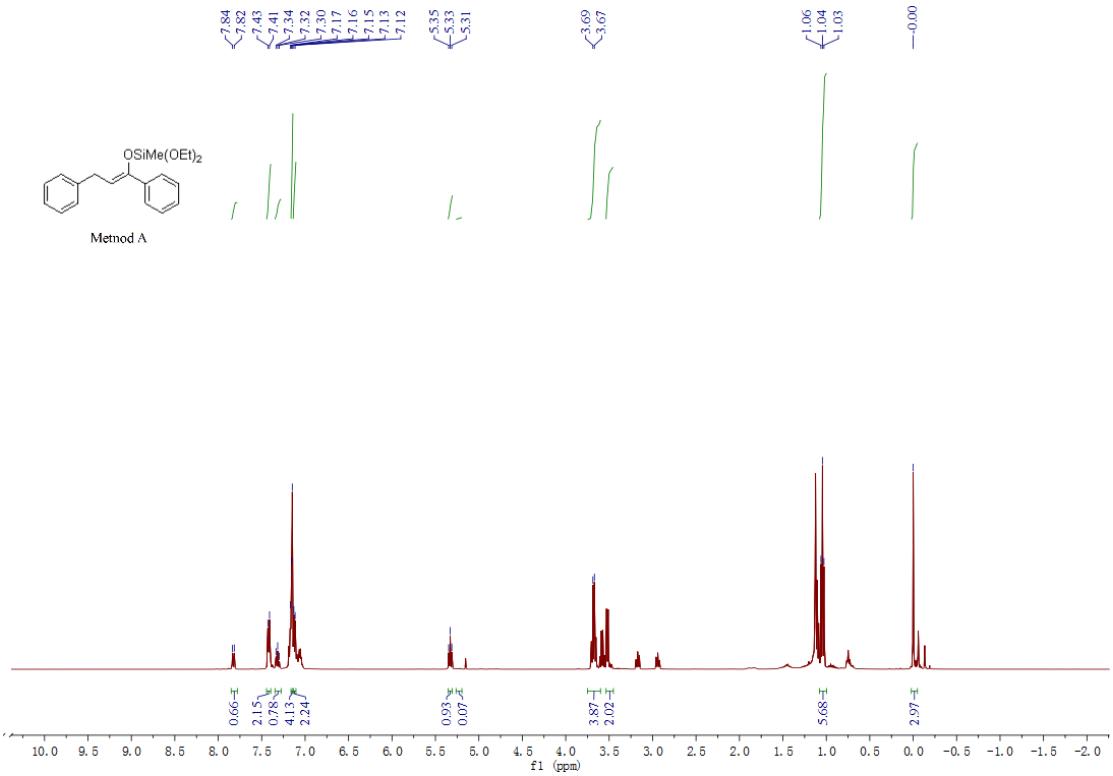

7.51
7.50
7.49
7.48
7.31
7.30
7.28
7.25
7.23
7.21
7.11
7.09
7.08
7.07
6.95
6.93
5.27
5.25
5.23
3.28
3.26
0.48
0.45
OSiMePh2
Method A
5.11
5.27
4.37
4.34
2.00
0.90
0.09
2.00
3.13
f1 (ppm)
151.72
136.61
136.50
132.15
130.53
130.42
130.10
130.01
129.97
129.80
128.15
79.51
79.19
78.87
34.56
20.60
0.00
OSiMePh2
f1 (ppm)


OSiMePh2
Method B
f1 (ppm)

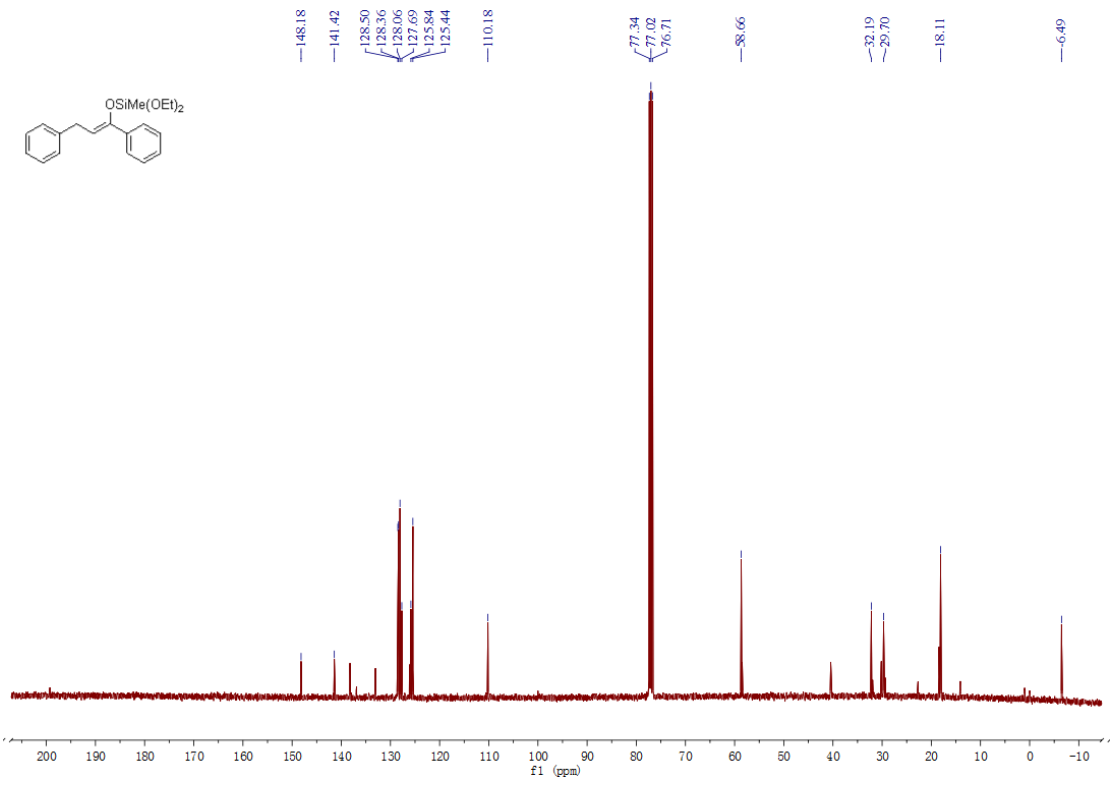

OSiMe2Ph
Method A
f1 (ppm)

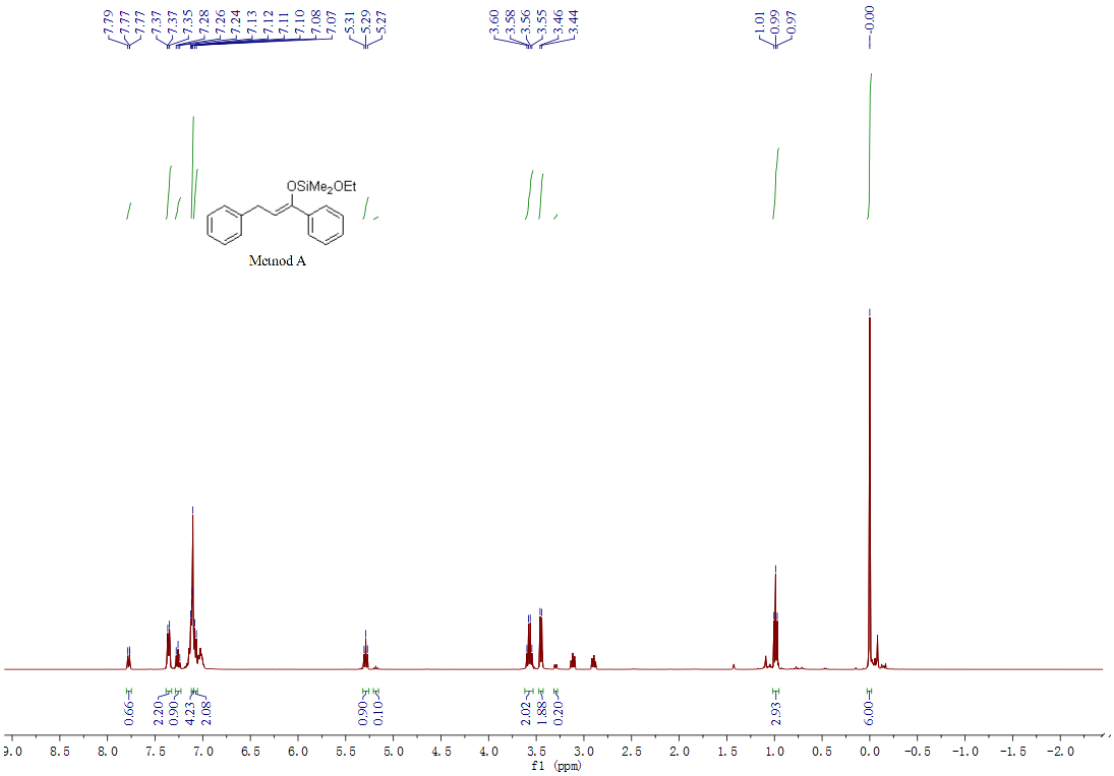

150.51
142.26
139.70
138.08
134.32
133.87
130.68
129.26
129.18
128.87
128.67
128.51
126.63
111.09
78.20
77.88
77.56
33.23
30.57
0.00
OSiMe2Ph
f1 (ppm)

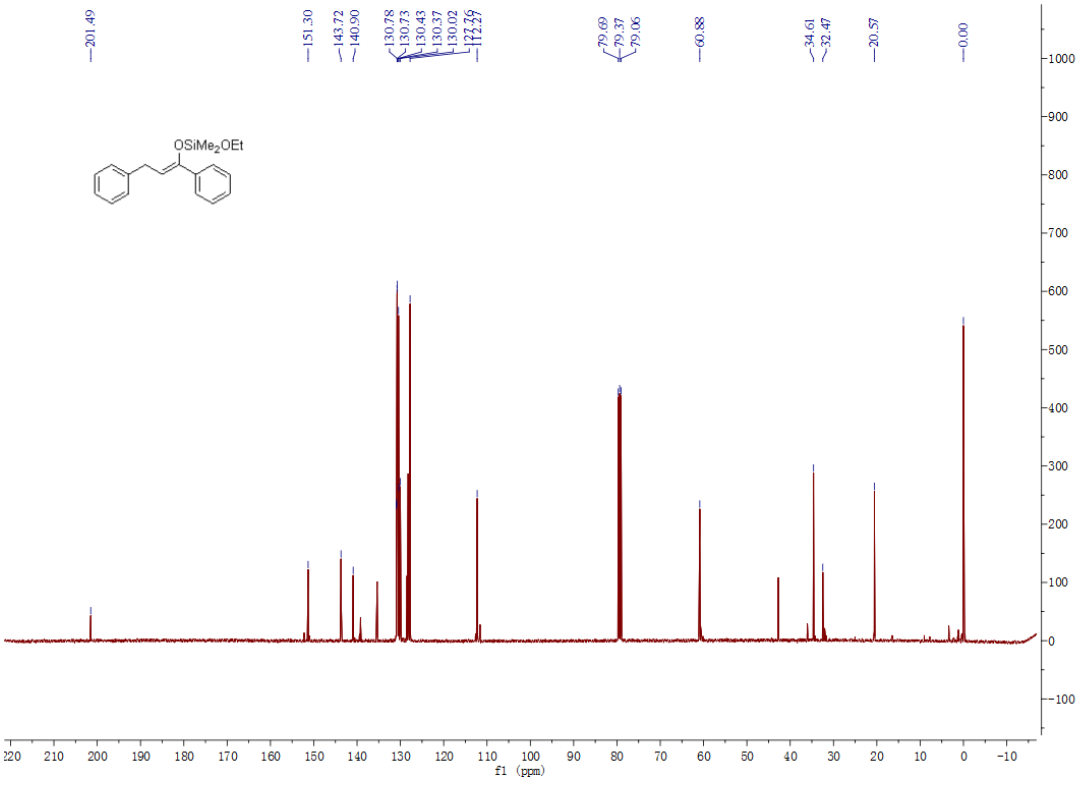

7.57
7.55
7.44
7.42
7.35
7.33
7.32
7.30
7.28
7.23
7.23
7.21
7.19
7.13
7.12
7.12
5.39
5.37
5.35
3.46
3.44
0.37
OSiMe2Ph
Method B
2.01
2.02
1.03
0.97
0.96
0.85
4.08
3.00
0.99
0.01
2.00
5.99
f1 (ppm)

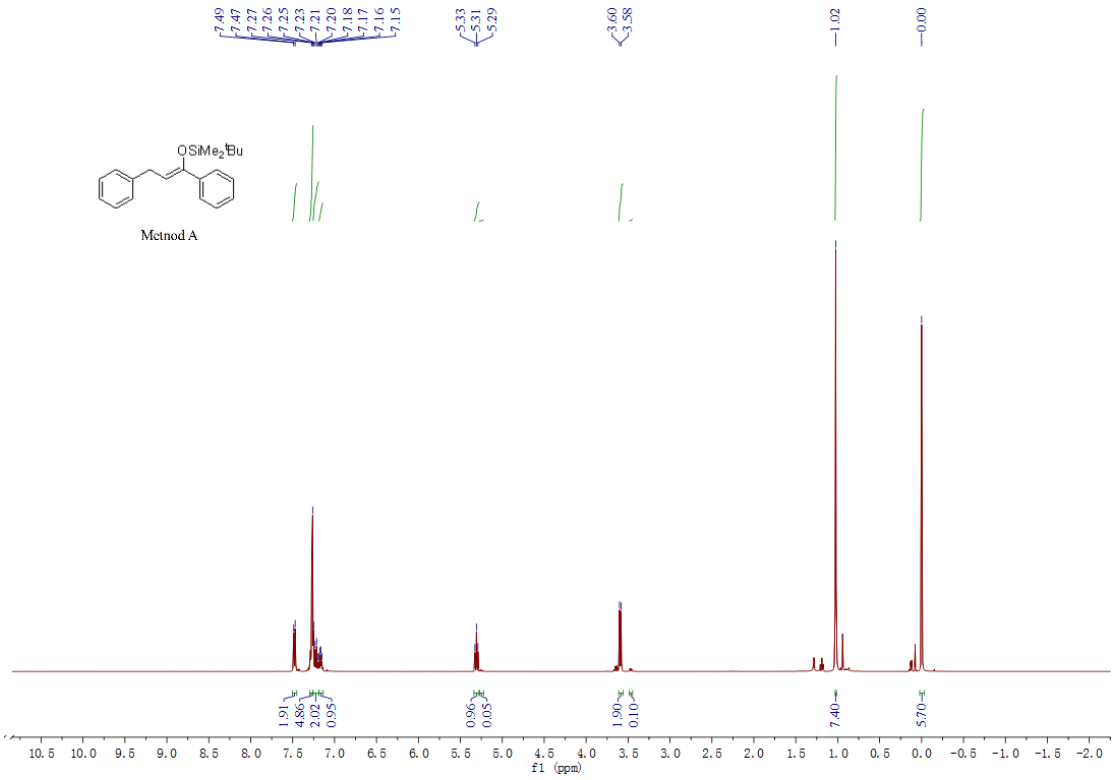

7.84
7.82
7.43
7.41
7.34
7.32
7.30
7.17
7.16
7.15
7.13
7.12
5.35
5.33
5.31
3.69
3.67
1.06
1.04
1.03
0.00
OSiMe(OEt)2
0.66
2.15
0.78
4.13
2.24
0.93
0.07
3.87
2.02
5.68
2.97
f1 (ppm)

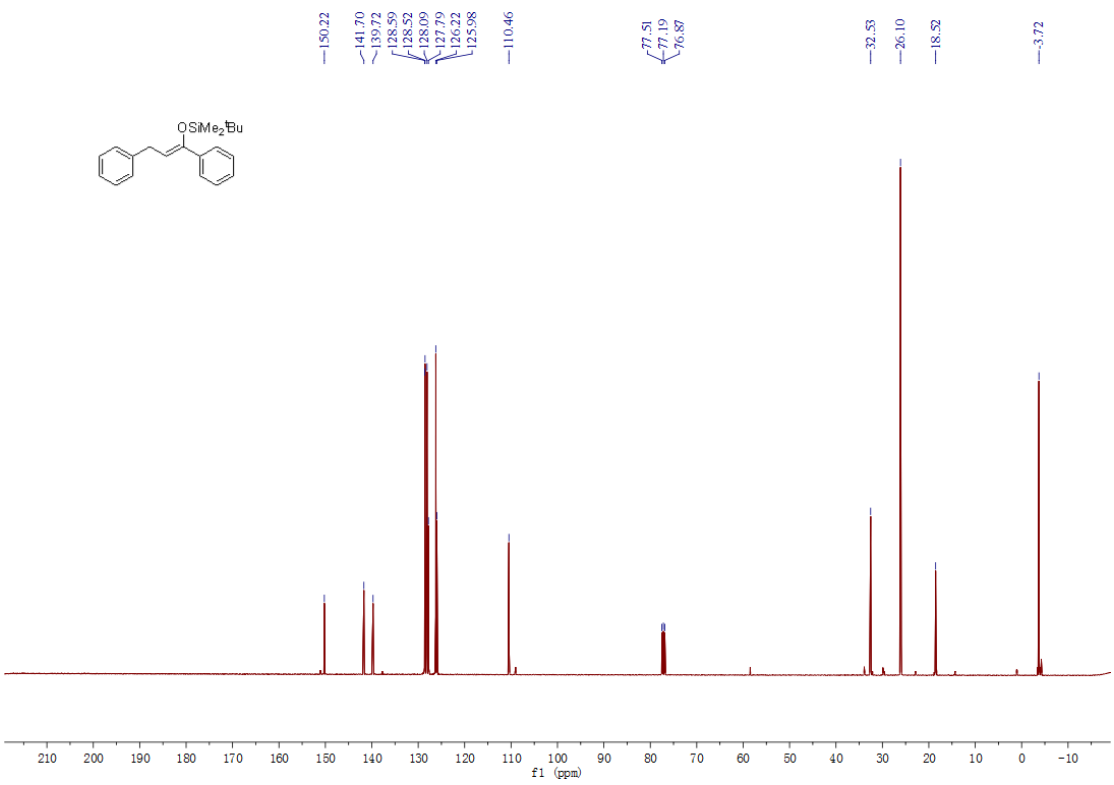
Metnod A

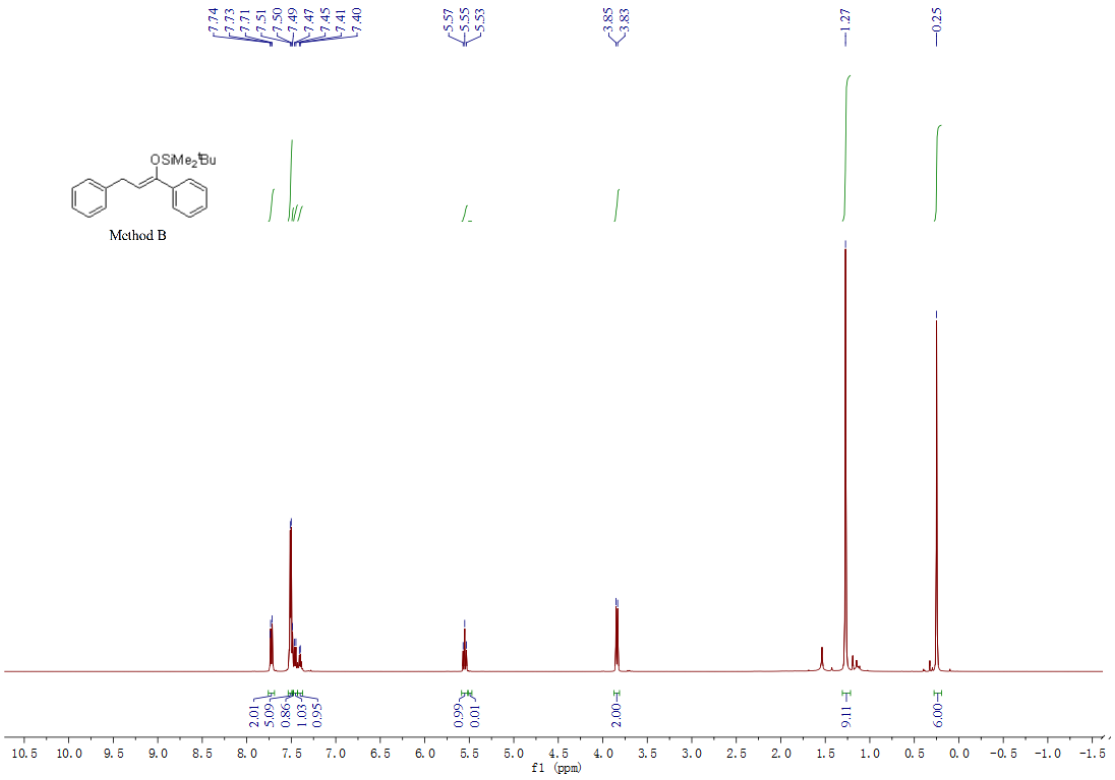

148.18
141.42
128.50
128.36
128.06
127.69
125.84
125.44
110.18
77.34
77.02
76.71
58.66
32.19
29.70
18.11
6.49
OSiMe(OEt)2
f1 (ppm)

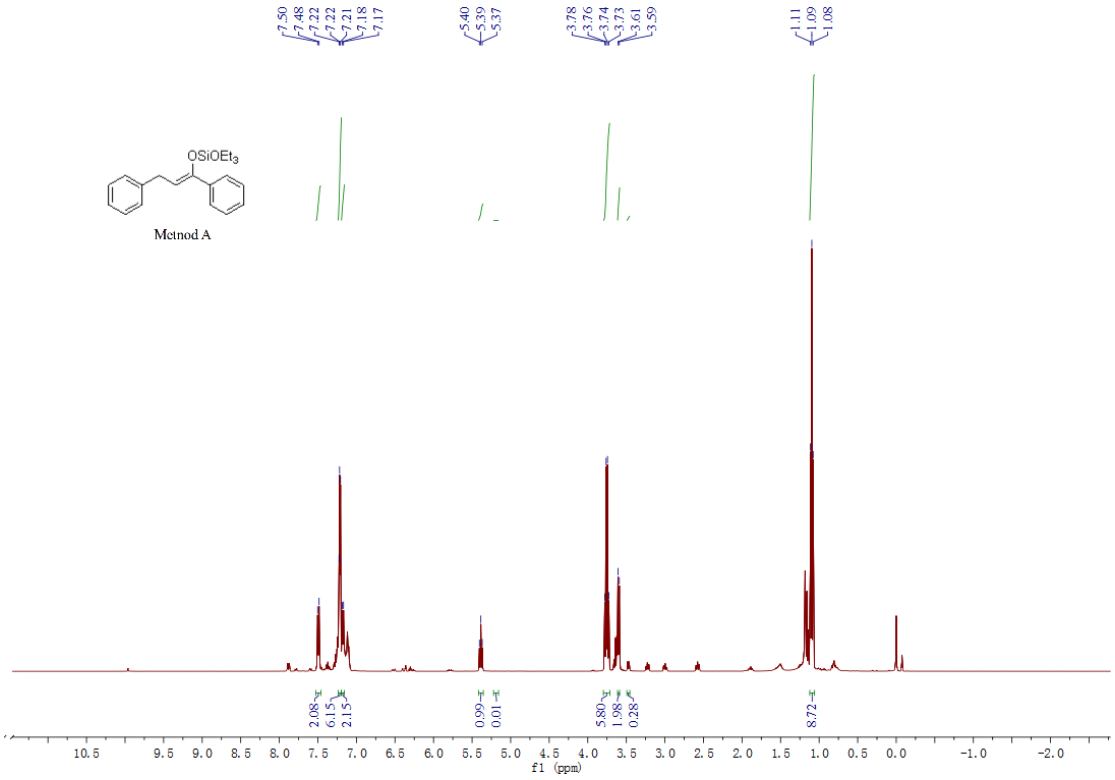

7.79
7.77
7.77
7.37
7.37
7.35
7.28
7.26
7.24
7.13
7.12
7.11
7.10
7.08
7.07
5.31
5.29
5.27
3.60
3.58
3.56
3.55
3.46
3.44
1.01
0.99
0.97
0.00
OSiMe2OEt
Metnod A
0.66
2.20
0.90
4.23
2.08
0.90
0.10
2.02
1.88
0.20
2.93
6.00
f1 (ppm)

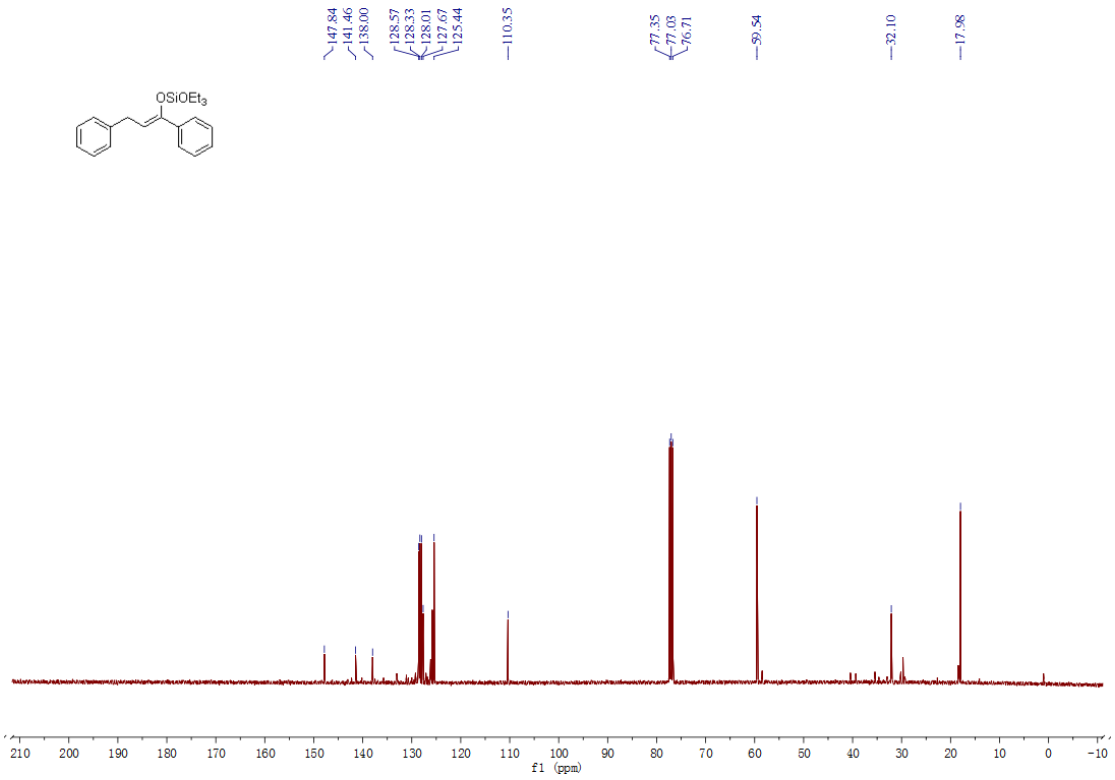

201.49
151.30
143.72
140.90
130.78
130.73
130.43
130.37
130.02
127.26
122.27
79.69
79.37
79.06
60.88
34.61
32.47
20.57
0.00
OSiMe2OEt
f1 (ppm)

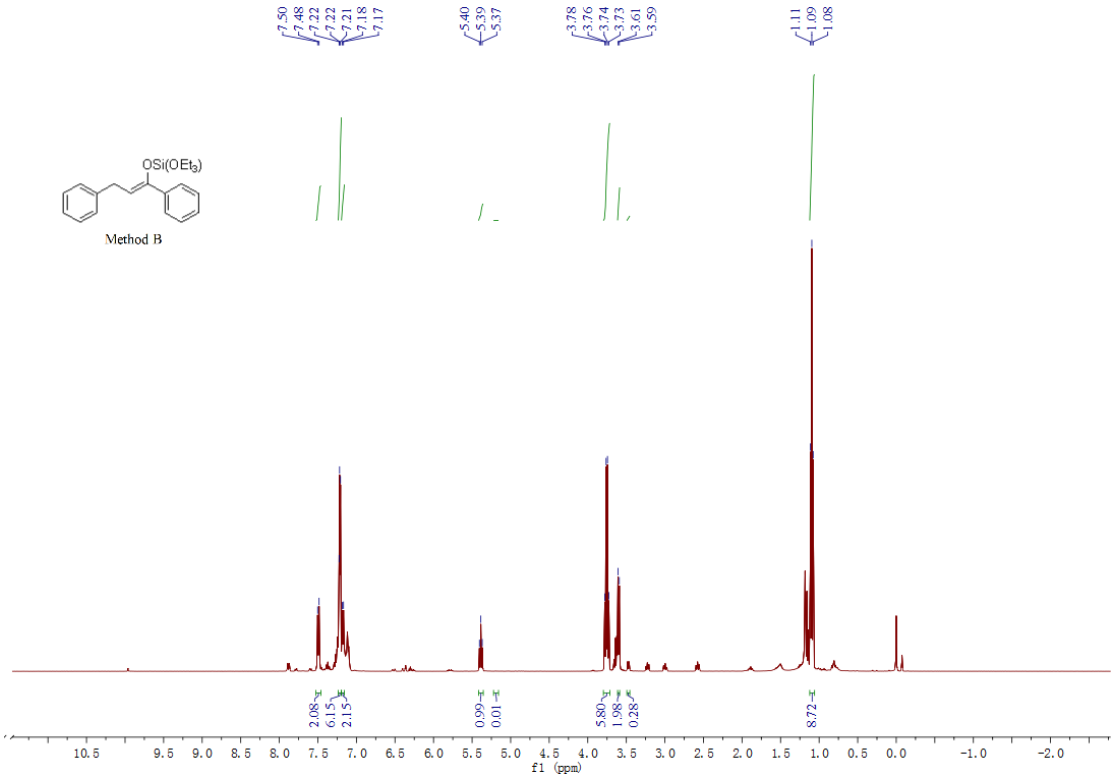

7.49
7.47
7.27
7.26
7.25
7.23
7.21
7.20
7.18
7.17
7.16
7.15
5.33
5.31
5.29
3.60
3.58
1.02
0.00
OSiMe2tBu
Metnod A
1.91
4.86
2.02
0.95
0.96
0.05
1.90
0.10
7.40
5.70
f1 (ppm)

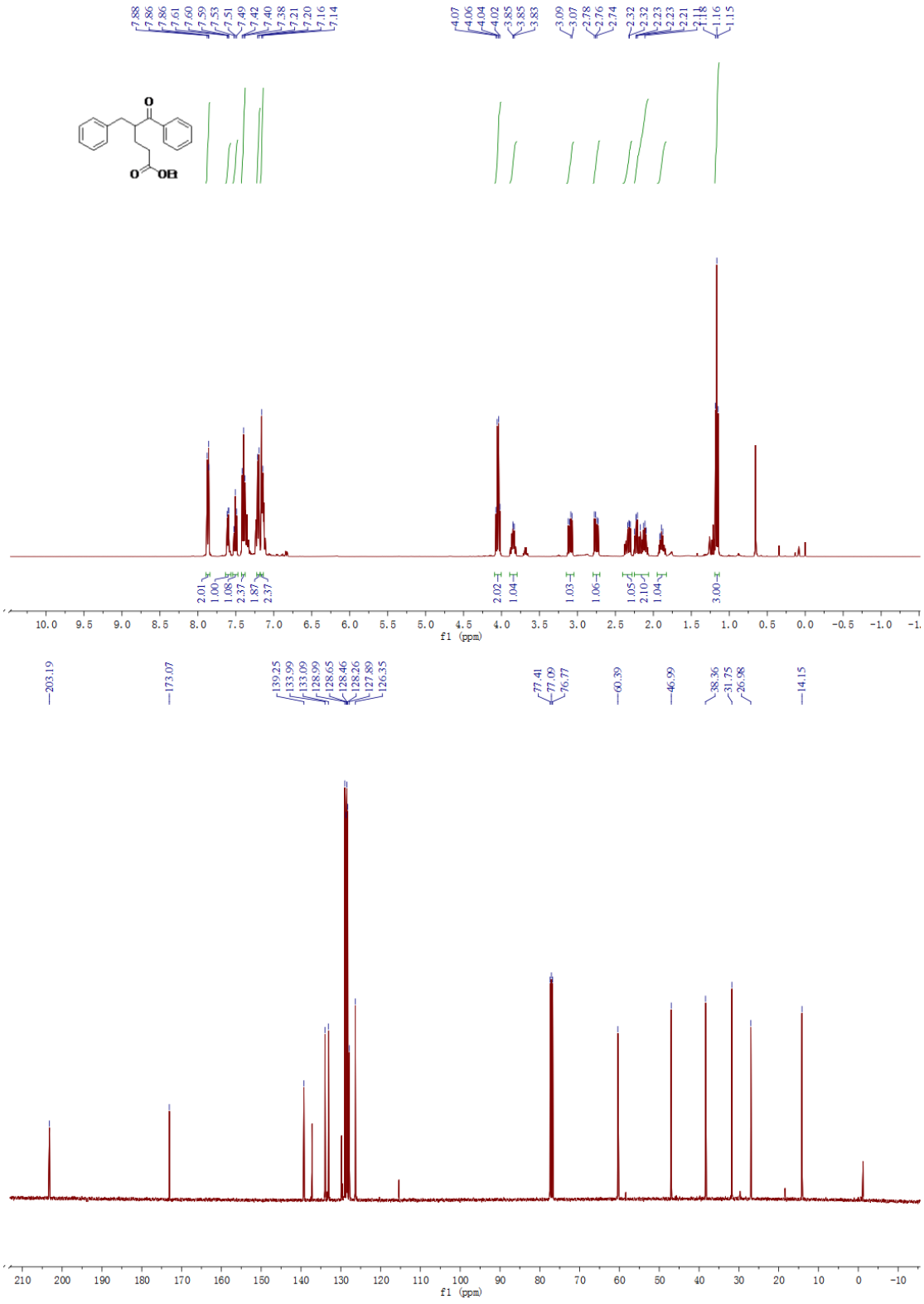

150.22
141.70
139.72
128.59
128.52
128.09
127.79
126.22
125.98
110.46
77.51
77.19
76.87
32.53
26.10
18.52
3.72
OSiMe2tBu
f1 (ppm)

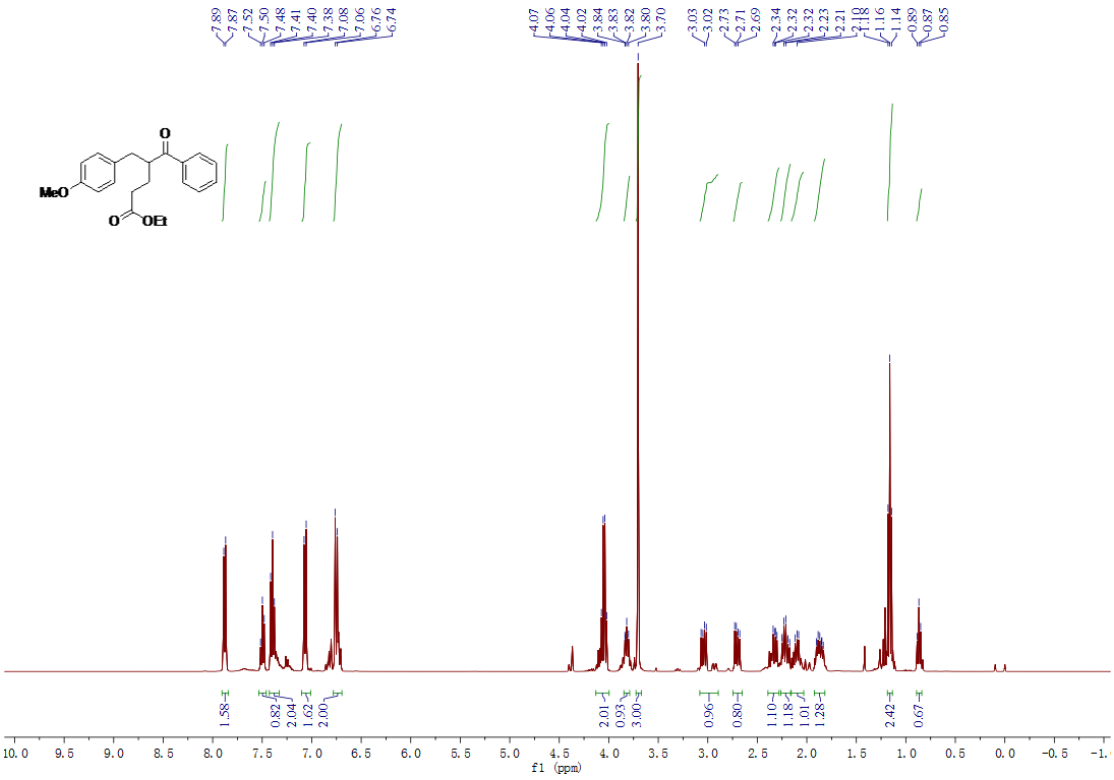

OSiMe2tBu
Method B
7.74
7.73
7.71
7.51
7.50
7.49
7.47
7.45
7.41
7.40
5.57
5.55
5.53
3.85
3.83
1.27
0.25
2.01
5.09
0.86
1.03
0.95
0.99
0.01
2.00
9.11
6.00
f1 (ppm)

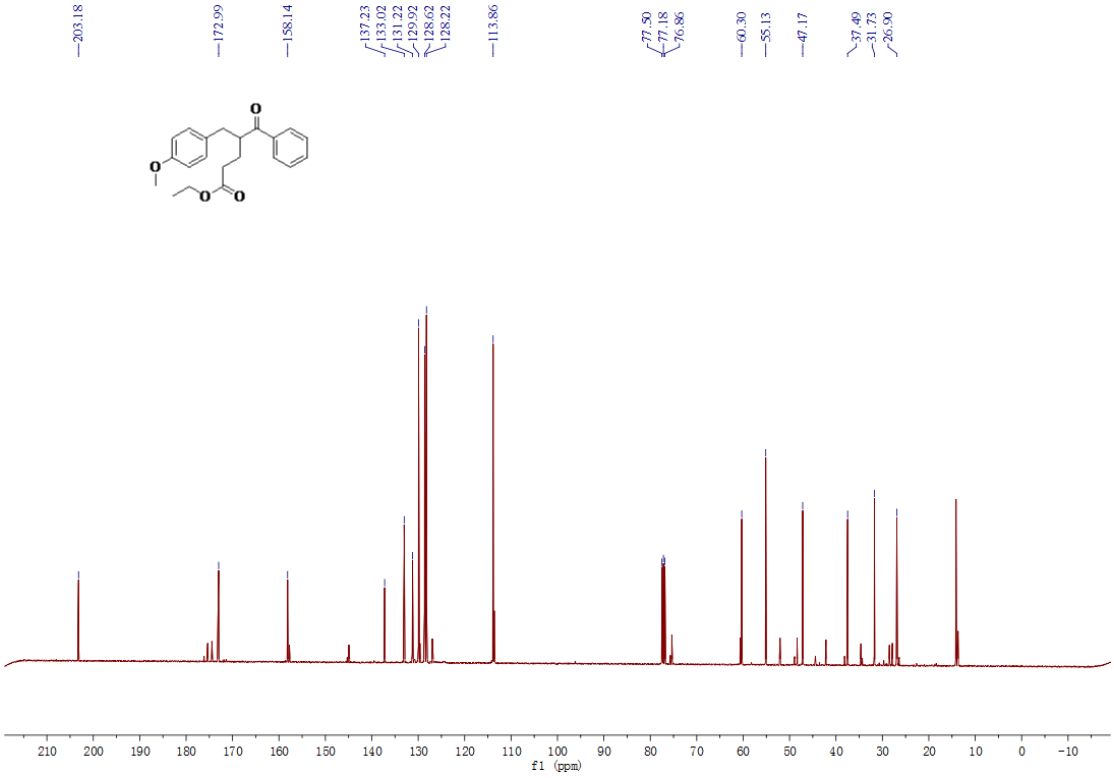

OSiOEt3
Metnod A
7.50
7.48
7.22
7.21
7.18
7.17
5.40
5.39
5.37
3.78
3.76
3.74
3.73
3.61
3.59
1.11
1.09
1.08
2.08
6.15
2.15
0.99
0.01
5.80
1.98
0.28
8.72
f1 (ppm)

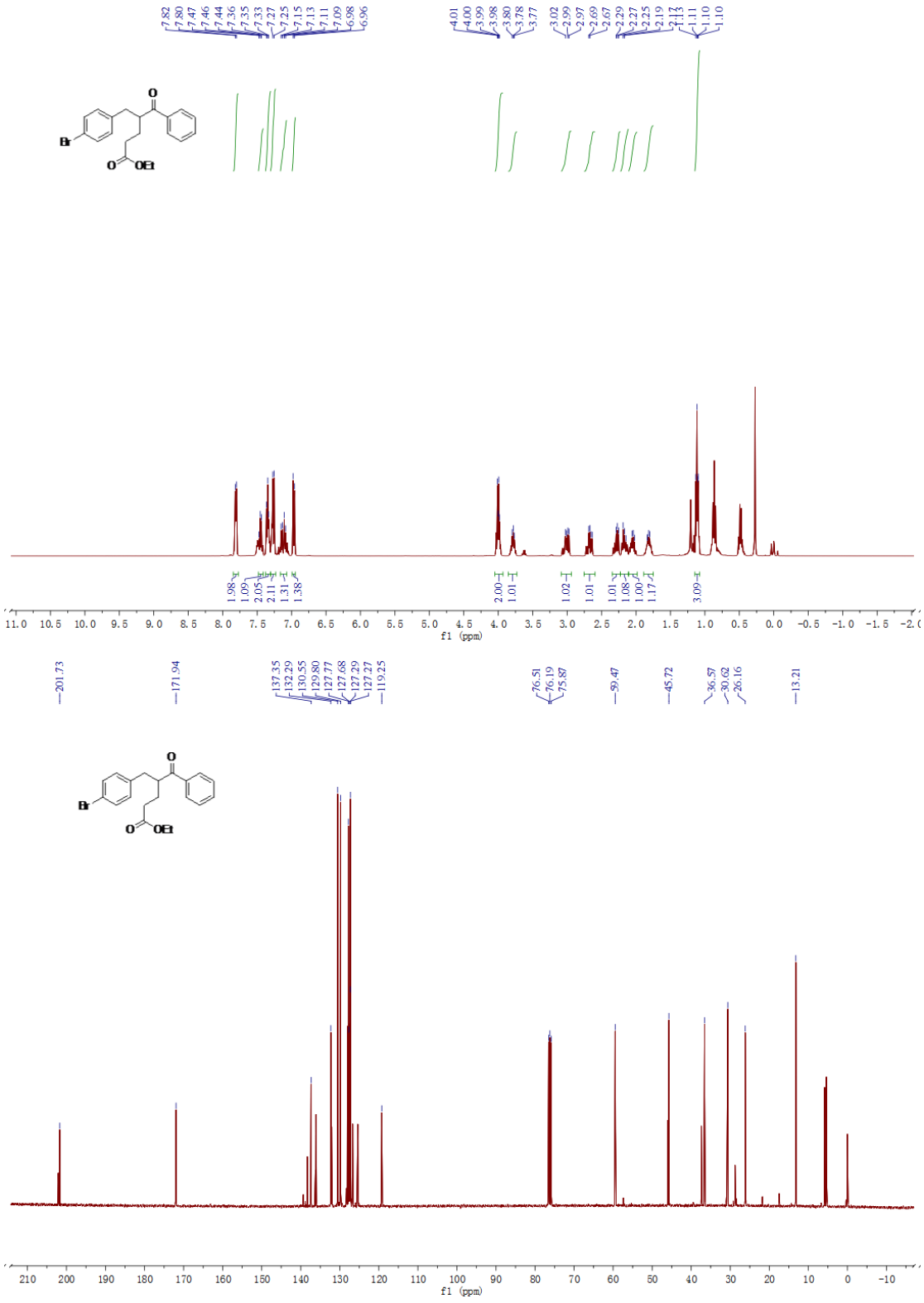

147.84
141.46
138.00
128.57
128.33
128.01
127.67
125.44
110.35
77.35
77.03
76.71
59.54
32.10
17.98
OSiOEt3
f1 (ppm)

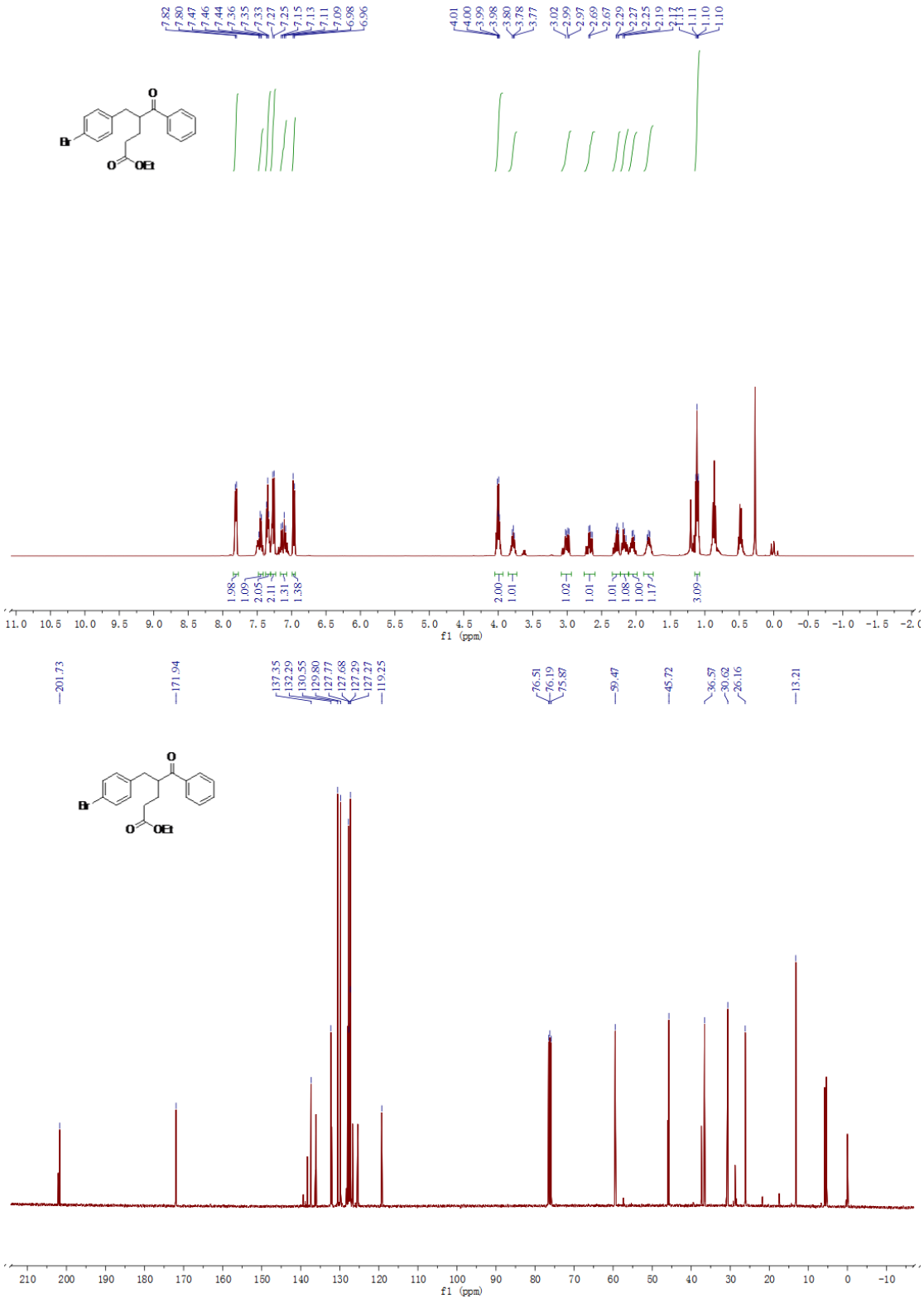

7.50
7.48
7.22
7.22
7.21
7.18
7.17
5.40
5.39
5.37
3.78
3.76
3.74
3.73
3.61
3.59
1.11
1.09
1.08
OSi(OEt3)
Method B
2.08
6.15
2.15
0.99
0.01
5.80
1.98
0.28
8.72
f1 (ppm)


7.88
7.86
7.86
7.61
7.60
7.59
7.53
7.51
7.49
7.42
7.40
7.38
7.21
7.20
7.16
7.14
4.07
4.06
4.04
4.02
3.85
3.85
3.83
3.09
3.07
2.78
2.76
2.74
2.32
2.32
2.23
2.23
2.21
1.18
1.16
1.15
O
O
OEt
2.01
1.00
1.08
2.37
1.87
2.37
2.02
1.04
1.03
1.06
1.05
2.10
1.04
3.00
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)
203.19
173.07
139.25
133.99
133.09
128.99
128.65
128.46
128.26
127.89
126.35
77.41
77.09
76.77
60.39
46.99
38.36
31.75
26.98
14.15
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)


7.89
7.87
7.52
7.50
7.48
7.41
7.40
7.38
7.08
7.06
6.76
6.74
4.07
4.06
4.04
4.02
3.84
3.83
3.82
3.80
3.70
3.03
3.02
2.73
2.71
2.69
2.34
2.32
2.32
2.23
2.21
1.18
1.16
1.14
0.89
0.87
0.85
MeO
O
OEt
1.58
0.82
2.04
1.62
2.00
2.01
0.93
3.00
0.96
0.80
1.10
1.18
1.01
1.28
2.42
0.67
f1 (ppm)


203.18
172.99
158.14
137.23
133.02
131.22
129.92
128.62
128.22
113.86
77.50
77.18
76.86
60.30
55.13
47.17
37.49
31.73
26.90
O
O
O
O
f1 (ppm)


7.82
7.80
7.47
7.46
7.44
7.36
7.35
7.33
7.27
7.25
7.15
7.13
7.11
7.09
6.98
6.96
4.01
4.00
3.99
3.98
3.80
3.78
3.77
3.02
2.99
2.97
2.69
2.67
2.29
2.27
2.25
2.19
2.13
1.11
1.10
1.10
Br
O
O
OEt
1.98
1.09
2.05
2.11
1.31
1.38
2.00
1.01
1.02
1.01
1.01
1.08
1.00
1.17
3.09
11.0 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 -1.5 -2.0
f1 (ppm)
201.73
171.94
137.35
132.29
130.55
129.80
127.77
127.68
127.29
127.27
119.25
76.51
76.19
75.87
59.47
45.72
36.57
30.62
26.16
13.21
Br
O
O
OEt
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)


7.82
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7.11
7.09
6.98
6.96
4.01
4.00
3.99
3.98
3.80
3.78
3.77
3.02
2.99
2.97
2.69
2.67
2.29
2.27
2.25
2.19
2.13
1.11
1.10
1.10
Br
O
O
OEt
1.98
1.09
2.05
2.11
1.31
1.38
2.00
1.01
1.02
1.01
1.01
1.08
1.00
1.17
3.09
f1 (ppm)
201.73
171.94
137.35
132.29
130.55
129.80
127.77
127.68
127.29
127.27
119.25
76.51
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45.72
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30.62
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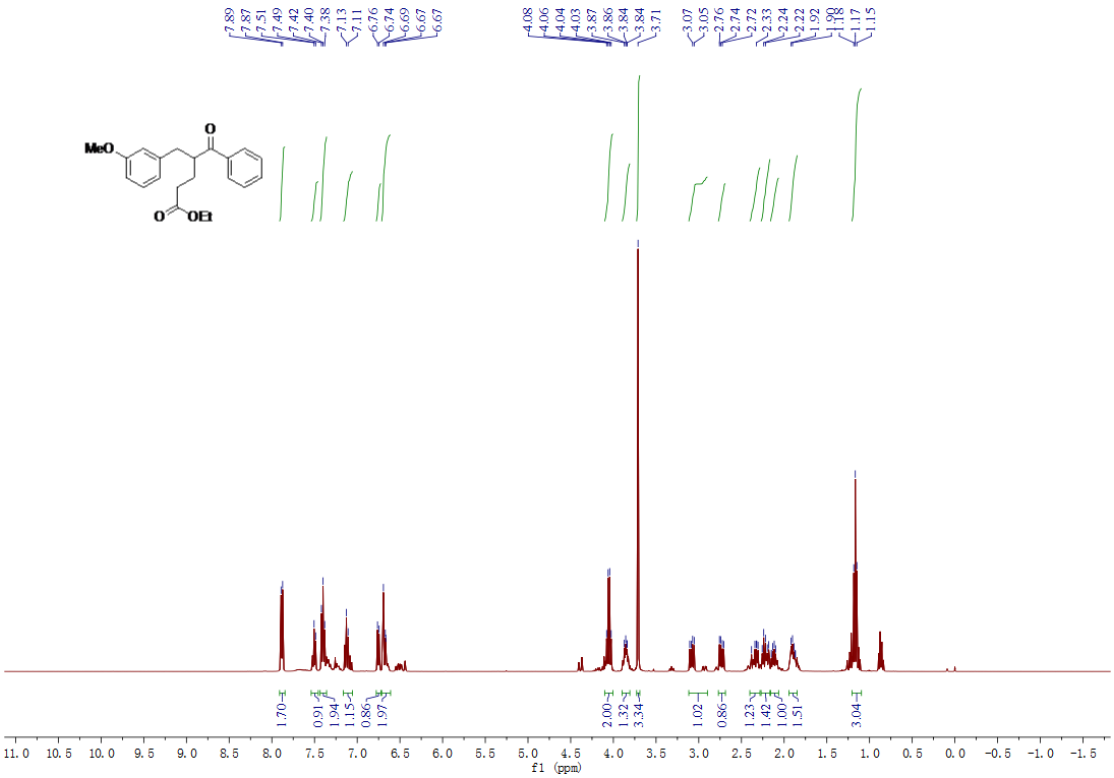
13.21
Br
O
O
OEt
f1 (ppm)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6.67
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4.06
4.04
4.03
3.87
3.86
3.84
3.84
3.71
3.07
3.05
2.76
2.74
2.72
2.33
2.24
2.22
1.92
1.98
1.96
1.17
1.15
MeO
O
O
OEt
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0.91
1.94
1.15
0.86
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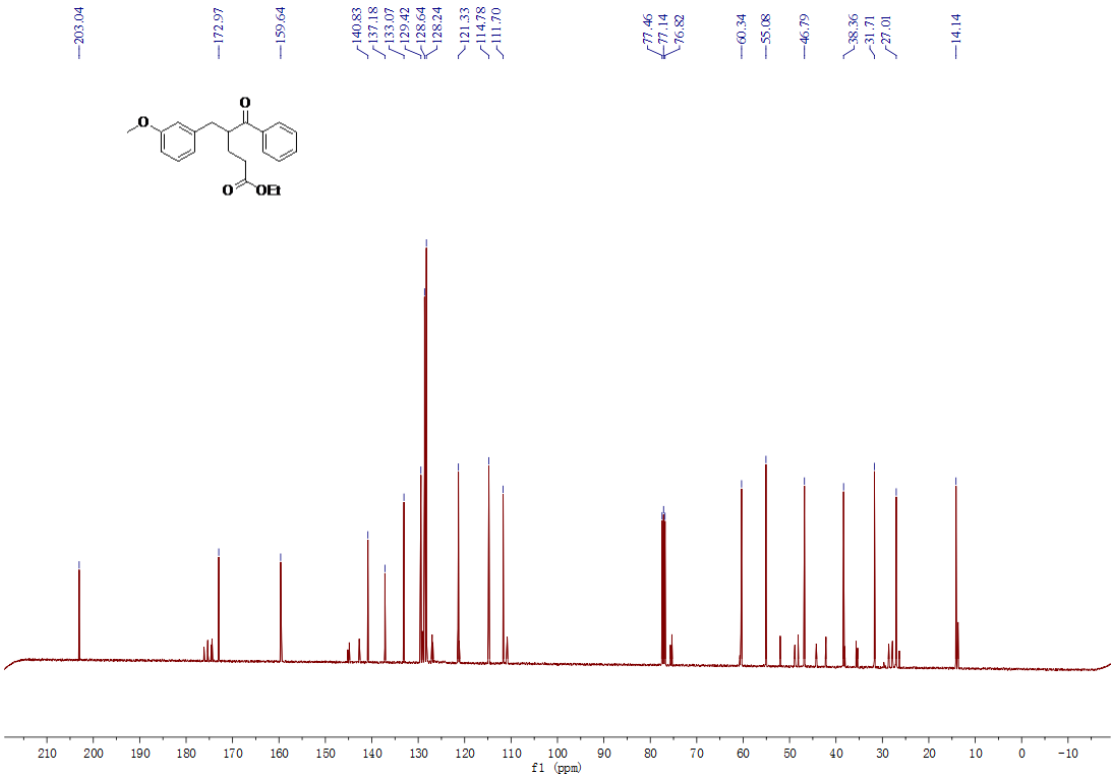
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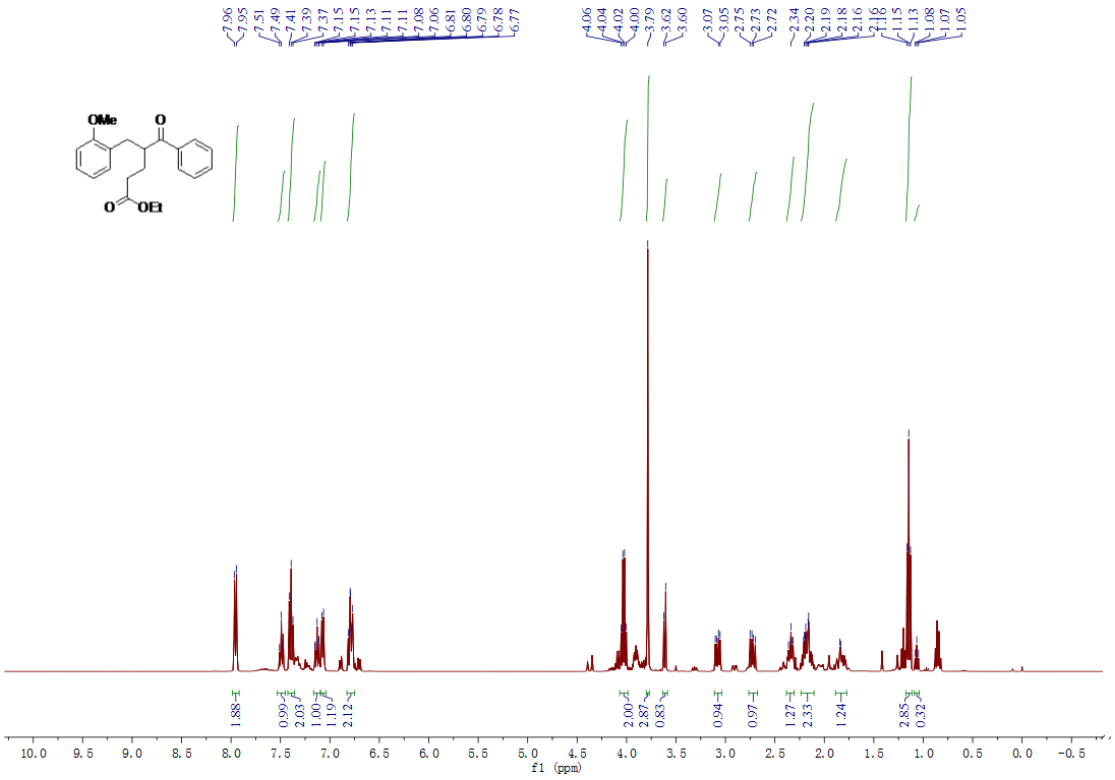
27.01
14.14
O
O
OEt
f1 (ppm)


OMe
O
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7.15
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7.13
7.11
7.11
7.08
7.06
6.81
6.80
6.79
6.78
6.77
4.06
4.04
4.02
4.00
3.79
3.62
3.60
3.07
3.05
2.75
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2.18
2.16
1.16
1.15
1.13
1.08
1.07
1.05
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1.00
1.19
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0.97
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1.24
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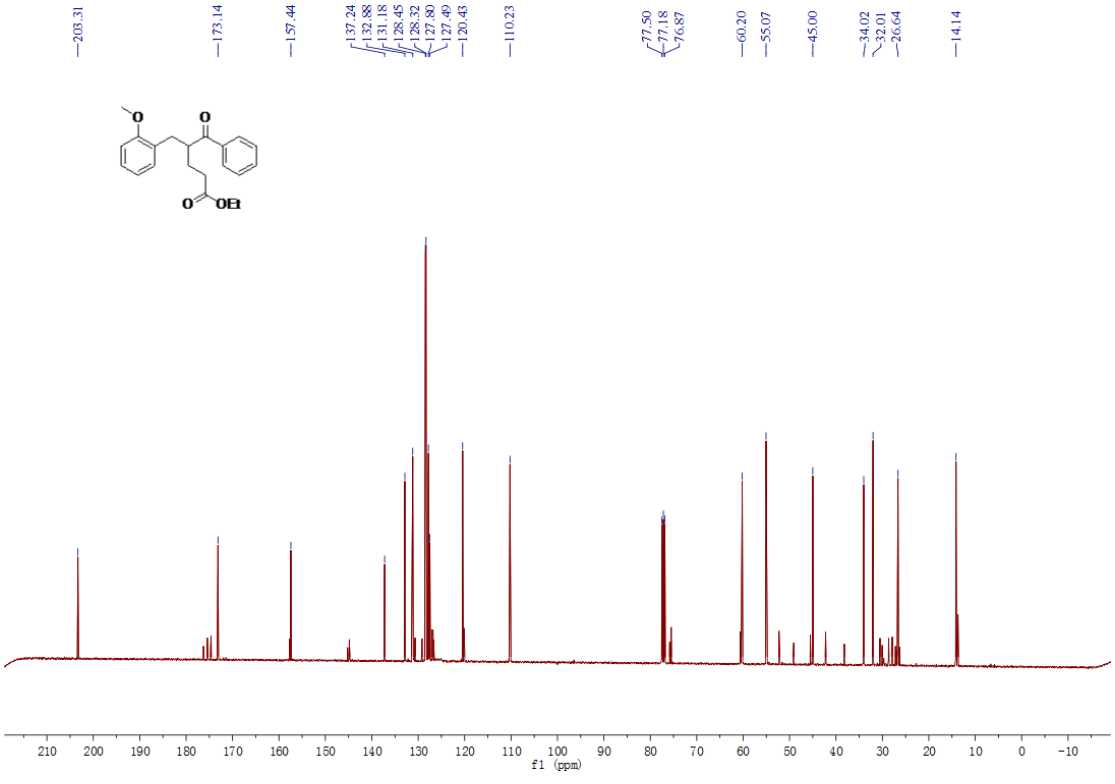
f1 (ppm)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O
O
OEt
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131.18
128.45
128.32
127.80
127.49
120.43
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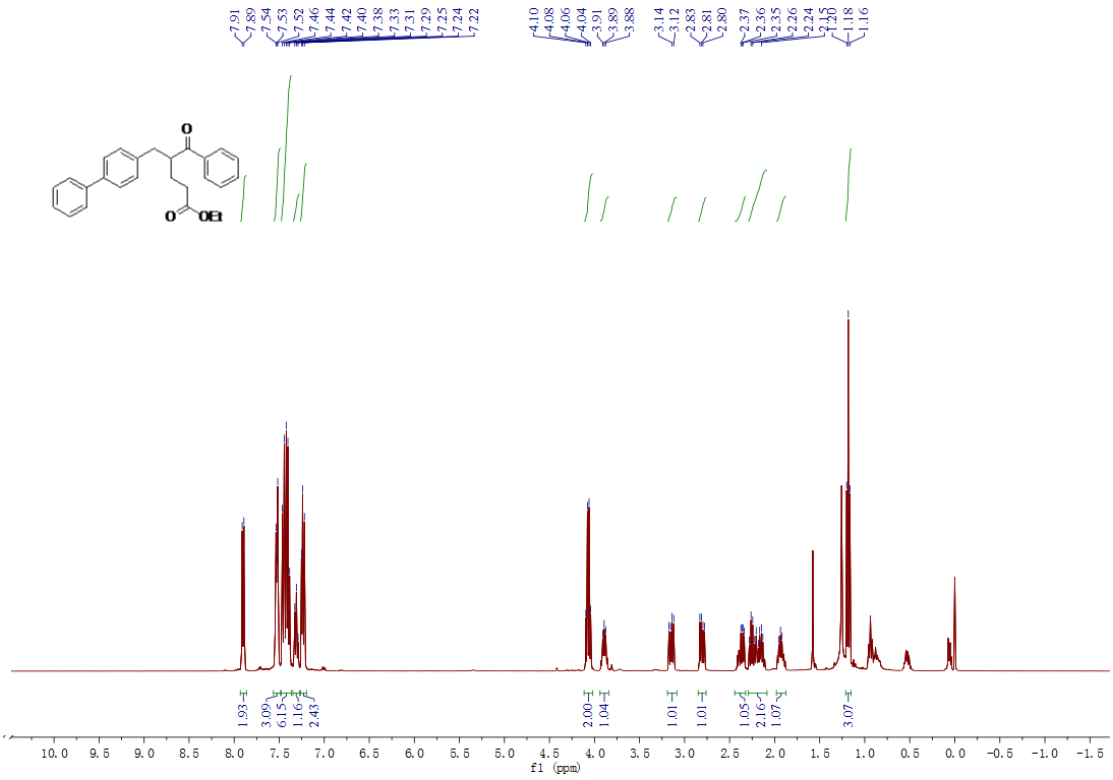
76.87
60.20
55.07
45.00
34.02
32.01
26.64
14.14
f1 (ppm)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7.22
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4.08
4.06
4.04
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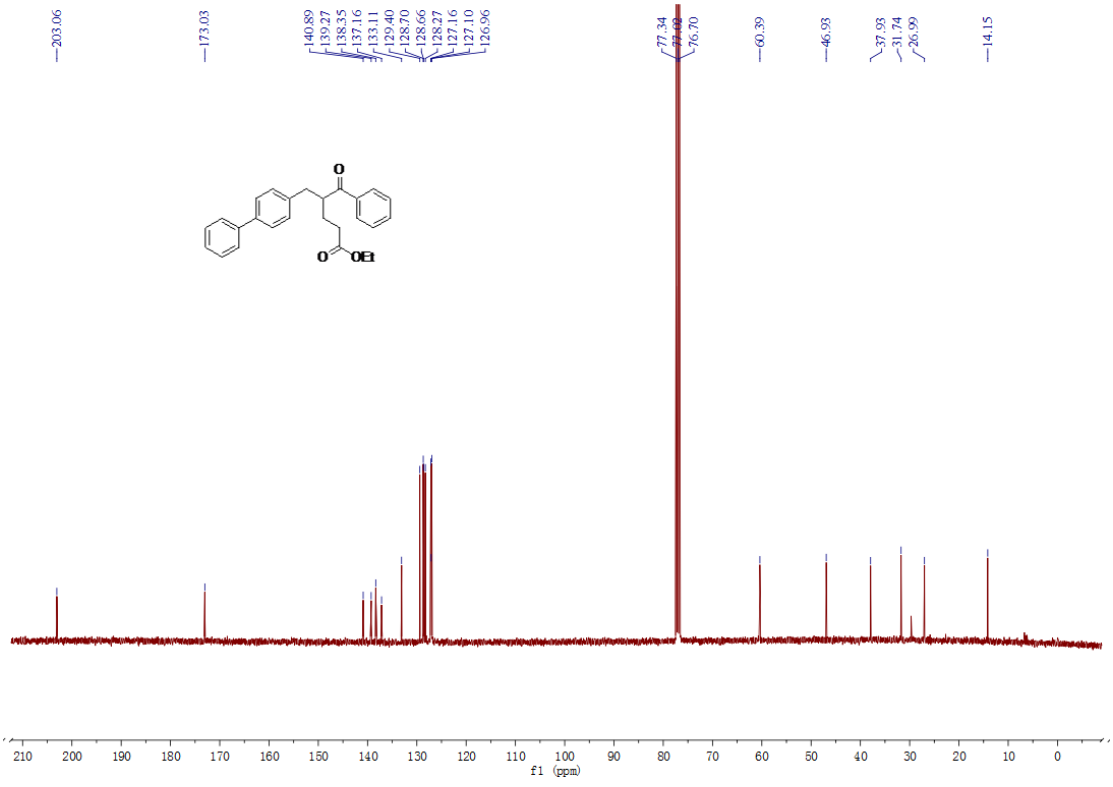
3.89
3.88
3.14
3.12
2.83
2.81
2.80
2.37
2.36
2.35
2.26
2.24
2.15
1.20
1.18
1.16
O
O
OEt
1.93
3.09
6.15
1.16
2.43
2.00
1.04
1.01
1.01
1.05
2.16
1.07
3.07
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)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137.16
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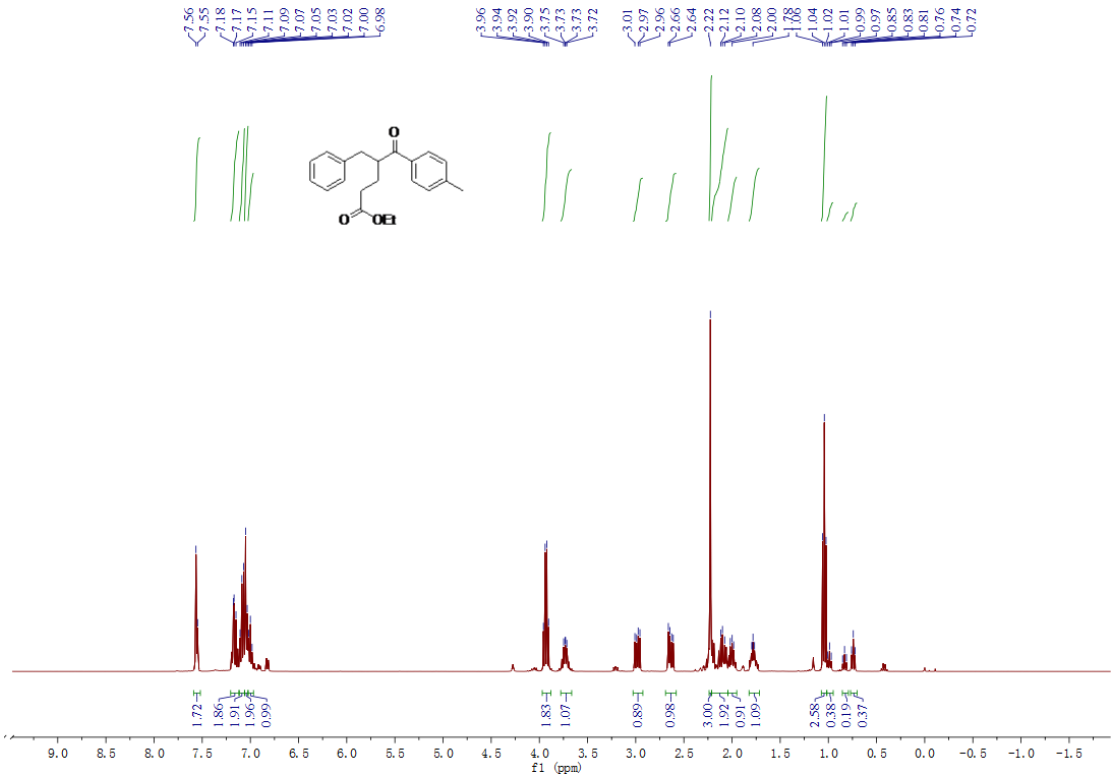
128.70
128.66
128.27
127.16
127.10
126.96
77.34
77.08
76.70
60.39
46.93
37.93
31.74
26.99
14.15
O
O
OEt
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.03
7.02
7.00
6.98
3.96
3.94
3.92
3.90
3.75
3.73
3.73
3.72
3.01
2.97
2.96
2.66
2.64
2.22
2.12
2.10
2.08
2.00
1.88
1.04
1.02
1.01
0.99
0.97
0.85
0.83
0.81
0.76
0.74
0.72
O
O
OEt
1.72
1.86
1.91
1.96
0.99
1.83
1.07
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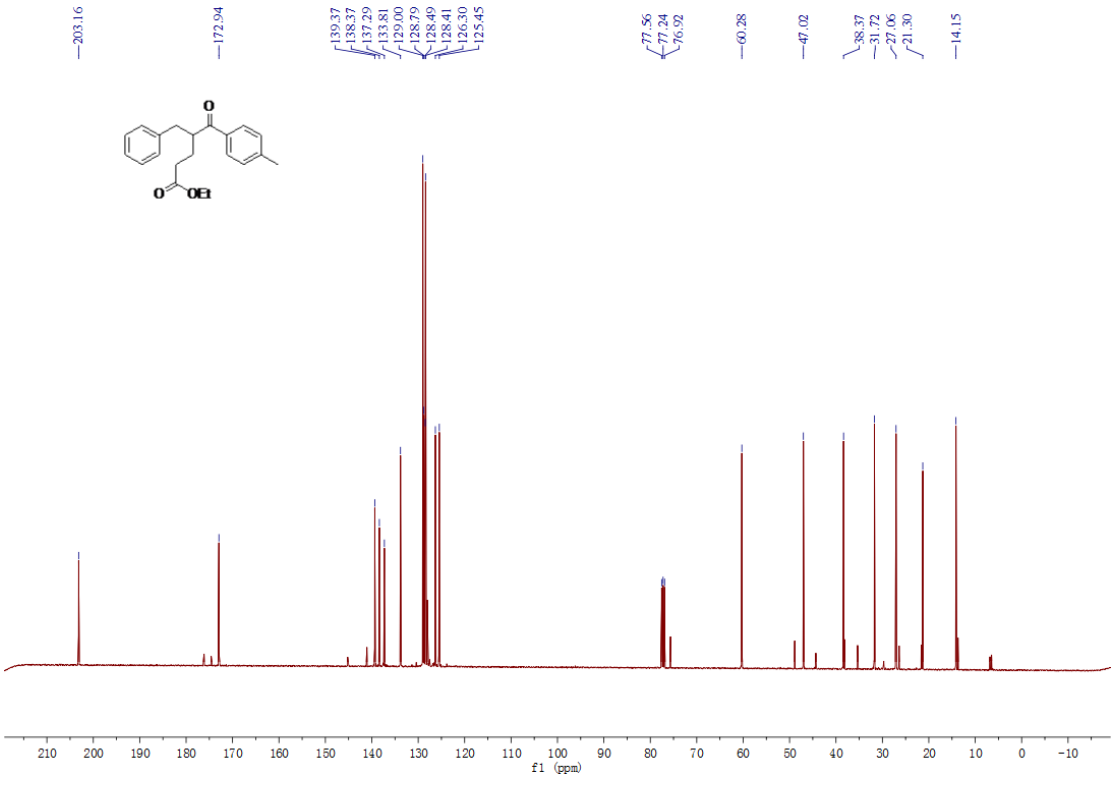
1.09
2.58
0.38
0.19
0.37
f1 (ppm)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77.56
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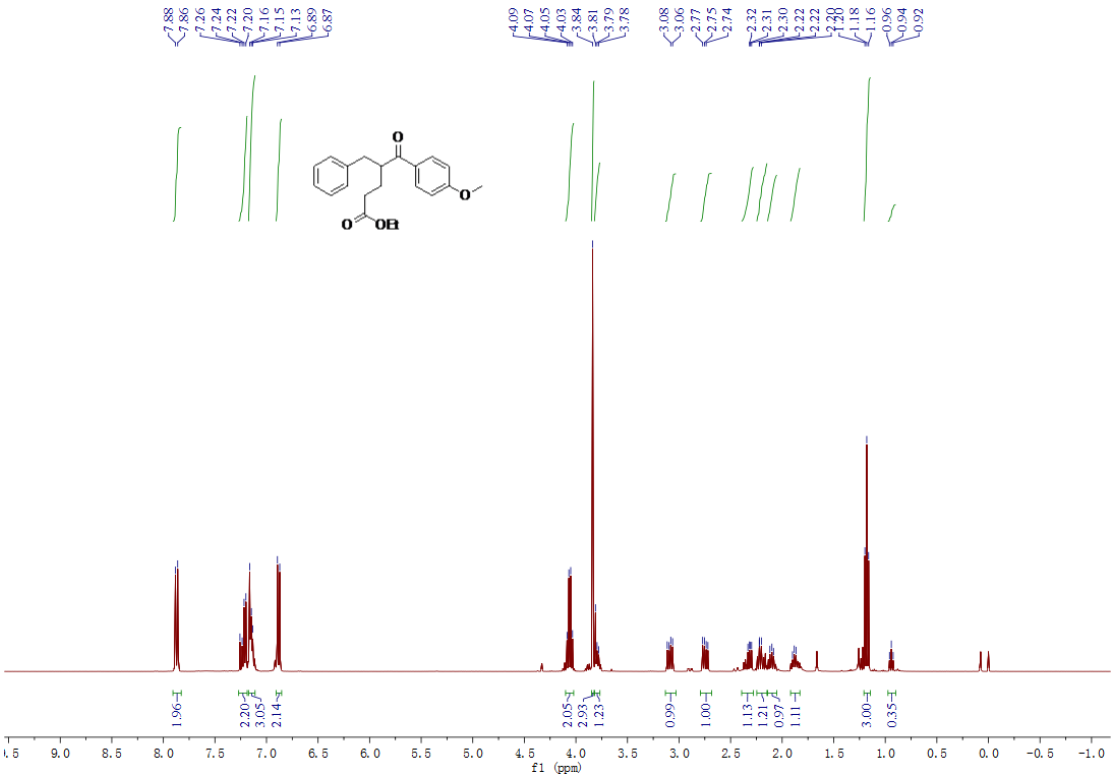
47.02
38.37
31.72
27.06
21.30
14.15
O
O
OEt
f1 (ppm)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6.87
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4.07
4.05
4.03
3.84
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3.79
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3.08
3.06
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2.75
2.74
2.32
2.31
2.30
2.22
2.22
1.18
1.16
0.96
0.94
0.92
1.96
2.20
3.05
2.14
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2.93
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0.99
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1.13
1.21
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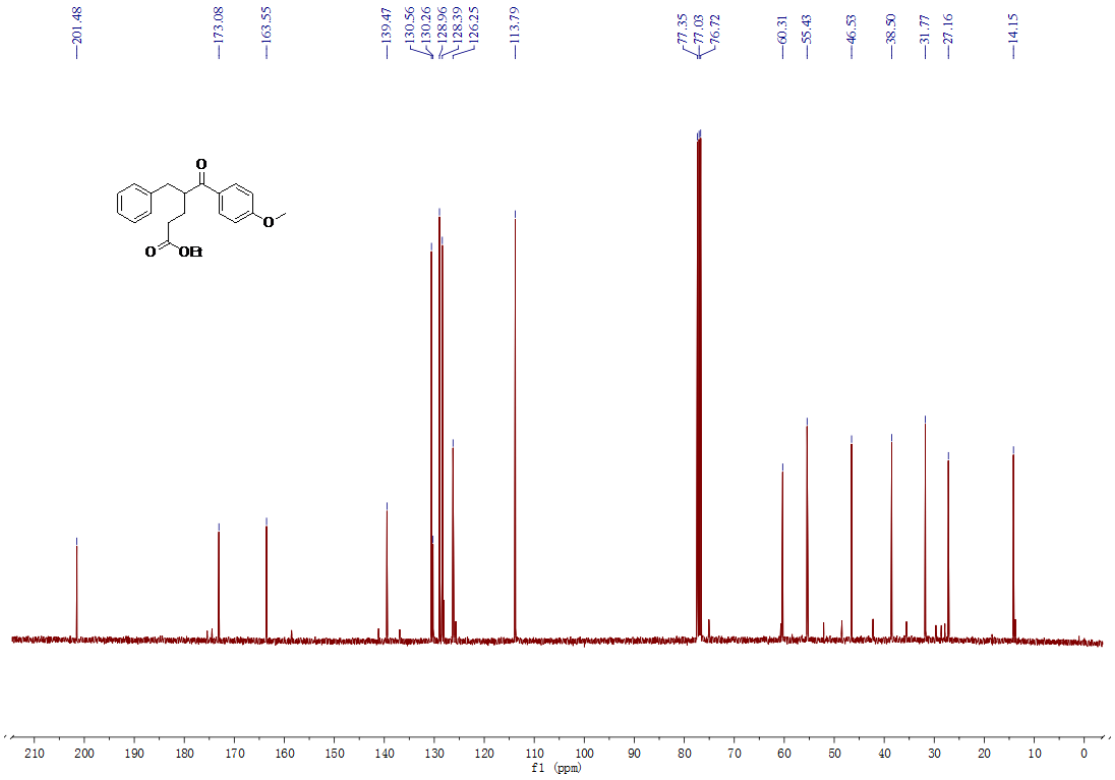
1.11
3.00
0.35
f1 (ppm)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77.35
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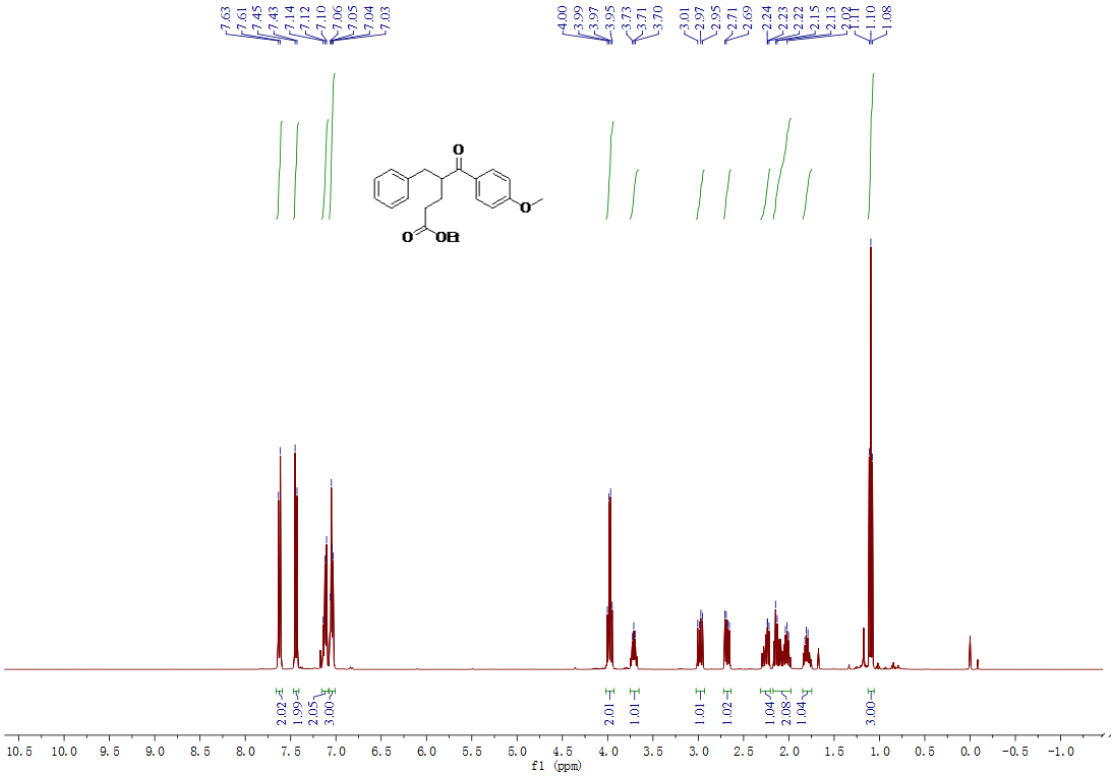
76.72
60.31
55.43
46.53
38.50
31.77
27.16
14.15
f1 (ppm)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7.04
7.03
4.00
3.99
3.97
3.95
3.73
3.71
3.70
3.01
2.97
2.95
2.71
2.69
2.24
2.23
2.22
2.15
2.13
1.11
1.10
1.08
2.02
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2.05
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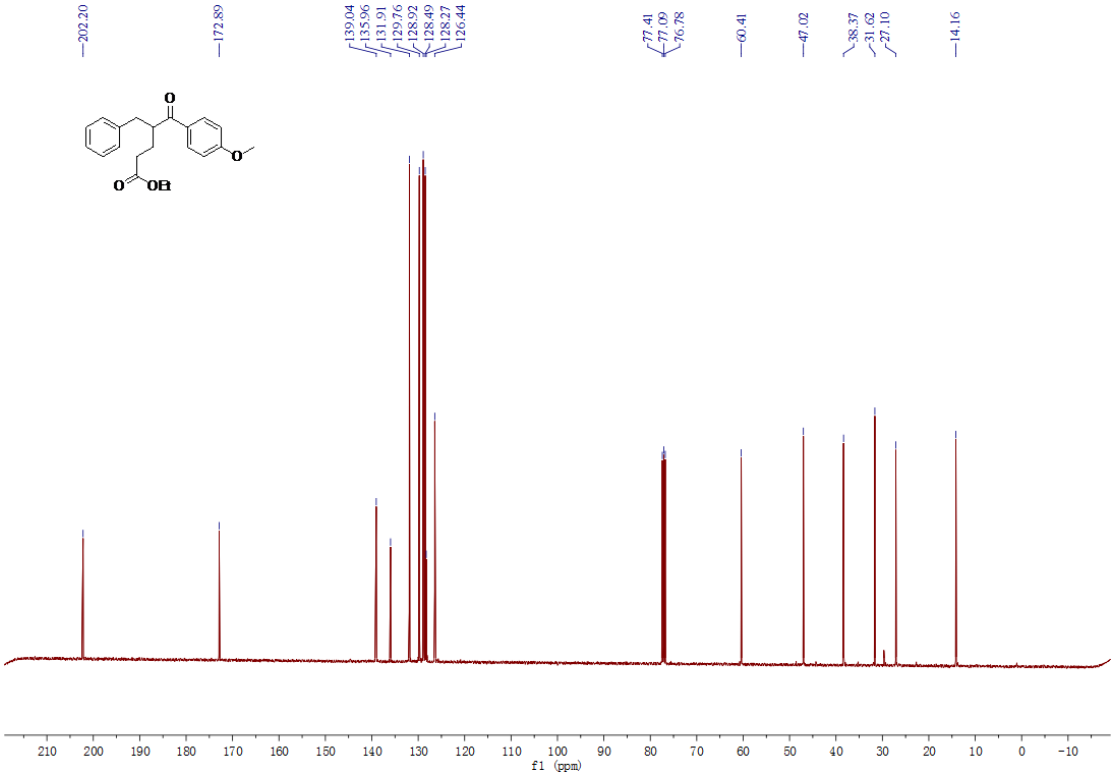
2.01
1.01
1.01
1.02
1.04
2.08
1.04
3.00
f1 (ppm)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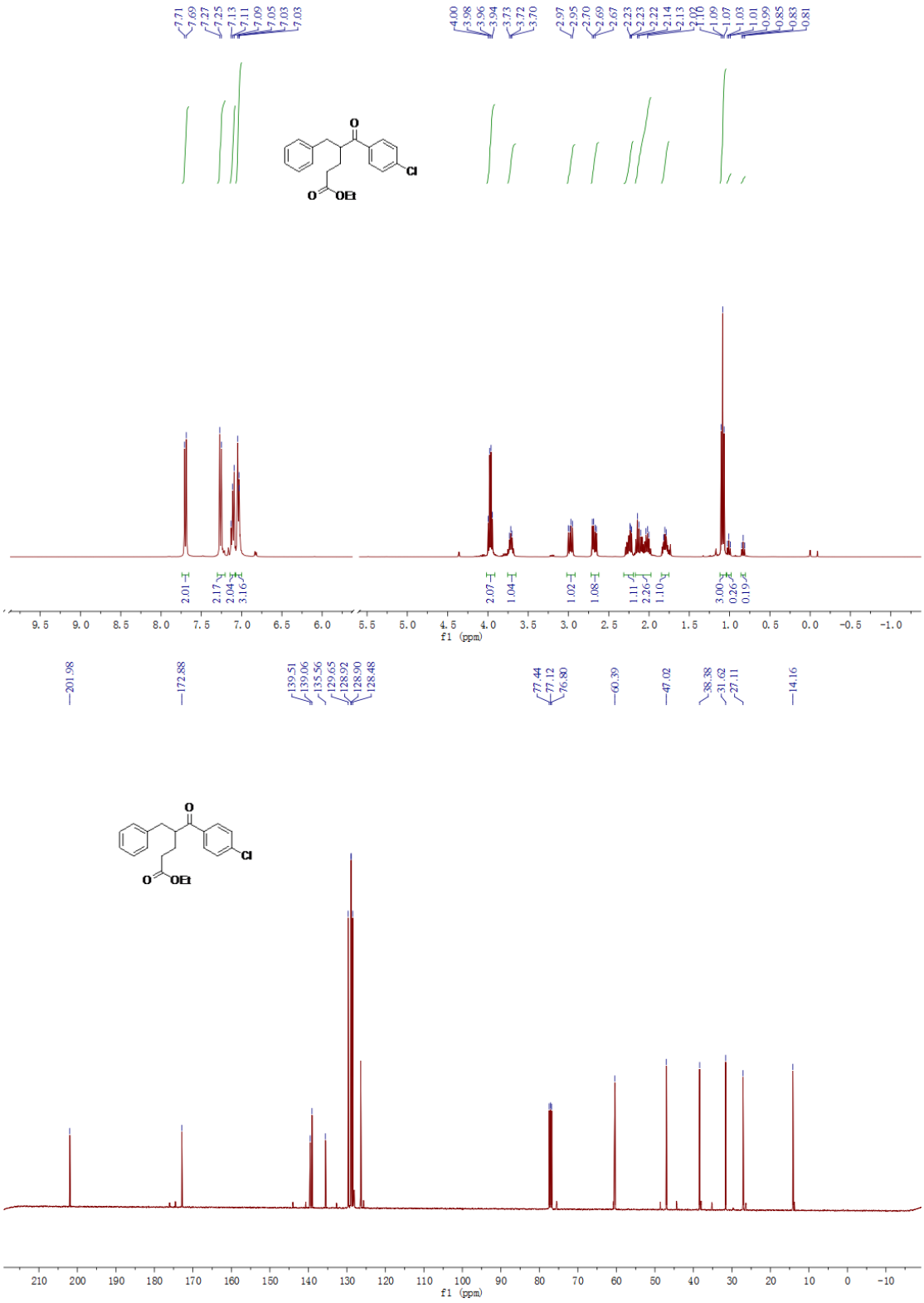
60.41
47.02
38.37
31.62
27.10
14.16
f1 (ppm)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7.03
4.00
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3.96
3.94
3.73
3.72
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2.95
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2.67
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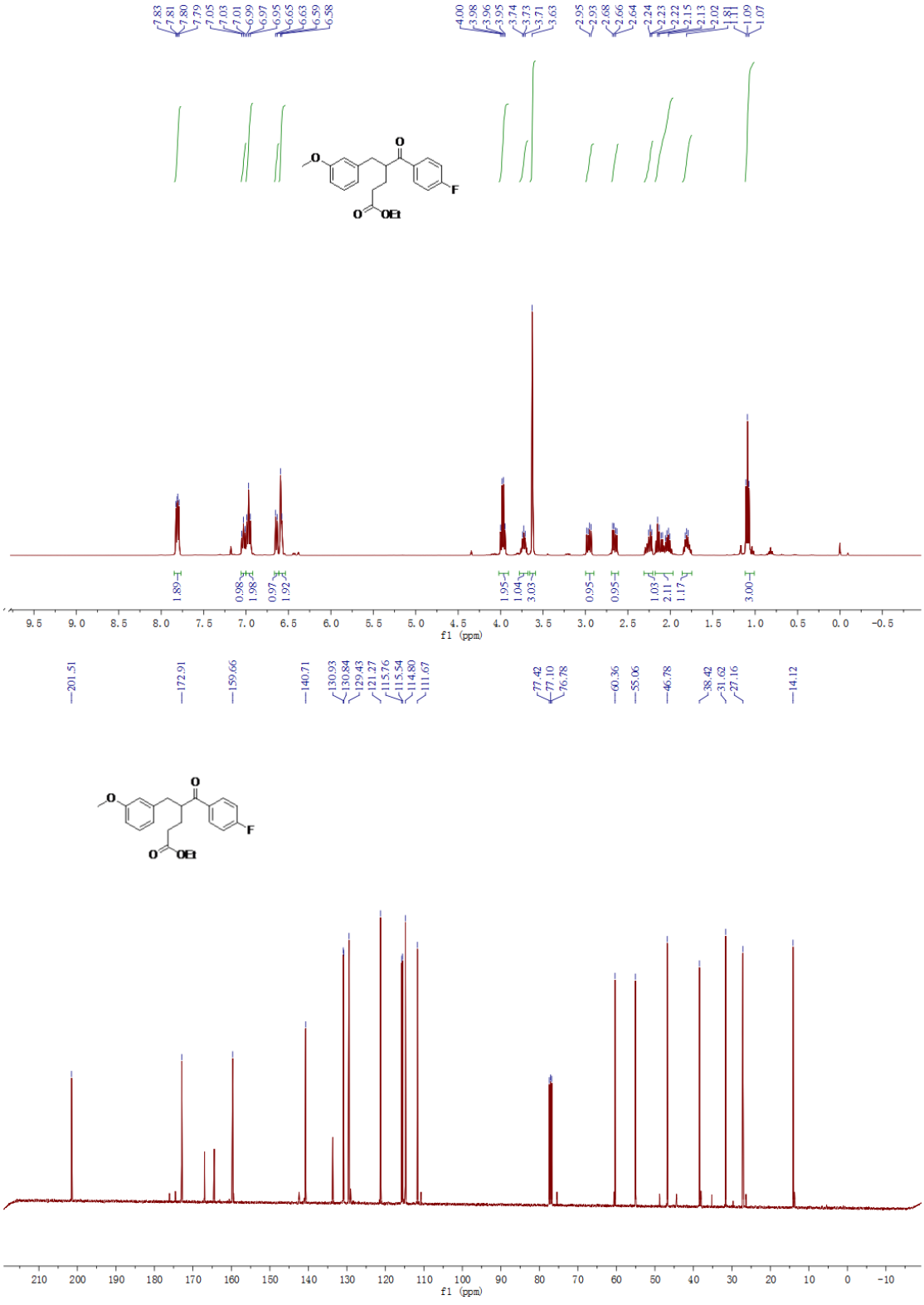
2.23
2.22
2.14
2.13
2.02
1.10
1.09
1.07
1.03
1.01
0.99
0.85
0.83
0.81
O
Cl
O
OEt
2.01
2.17
2.04
3.16
2.07
1.04
1.02
1.08
1.11
2.26
1.10
3.00
0.26
0.19
f1 (ppm)
201.98
172.88
139.51
139.06
135.56
129.65
128.92
128.90
128.48
77.44
77.12
76.80
60.39
47.02
38.38
31.62
27.11
14.16
O
Cl
O
OEt
f1 (ppm)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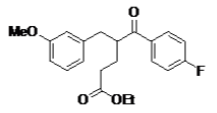
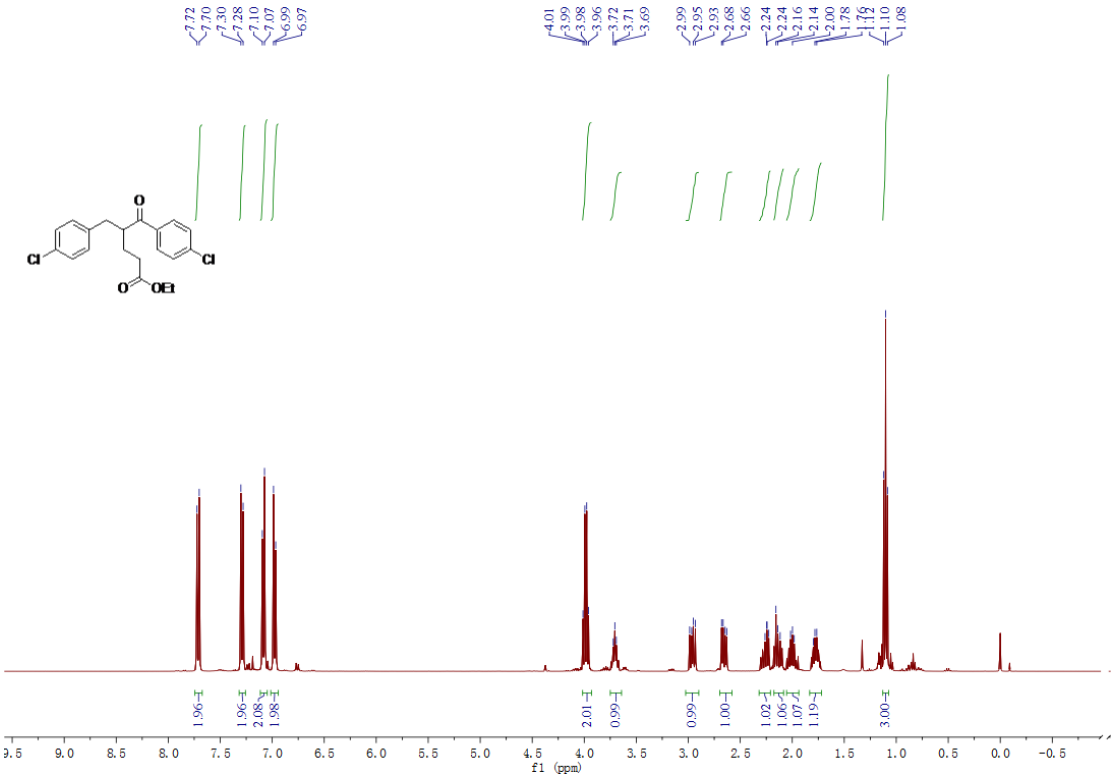
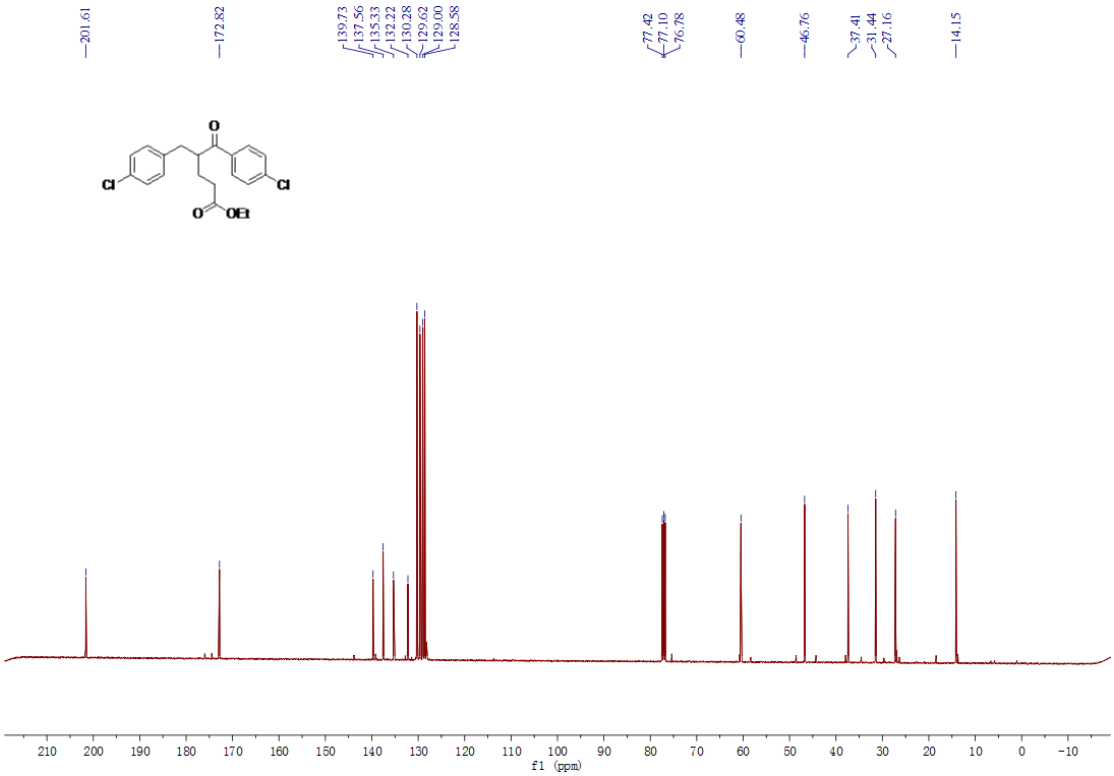
6.58
4.00
3.98
3.96
3.95
3.74
3.73
3.71
3.63
2.95
2.93
2.68
2.66
2.64
2.24
2.23
2.22
2.15
2.13
2.02
1.81
1.11
1.09
1.07
O
O
F
O
OEt
1.89
0.98
1.98
0.97
1.92
1.95
1.04
3.03
0.95
0.95
1.03
2.11
1.17
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
201.51
172.91
159.66
140.71
130.93
130.84
129.43
121.27
115.76
115.54
114.80
111.67
77.42
77.10
76.78
60.36
55.06
46.78
38.42
31.62
27.16
14.12
O
O
F
O
OEt
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)



^{19}F NMR (471 MHz, $CDCl_3$) δ -105.17 (s).

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129.62
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128.58
77.42
77.10
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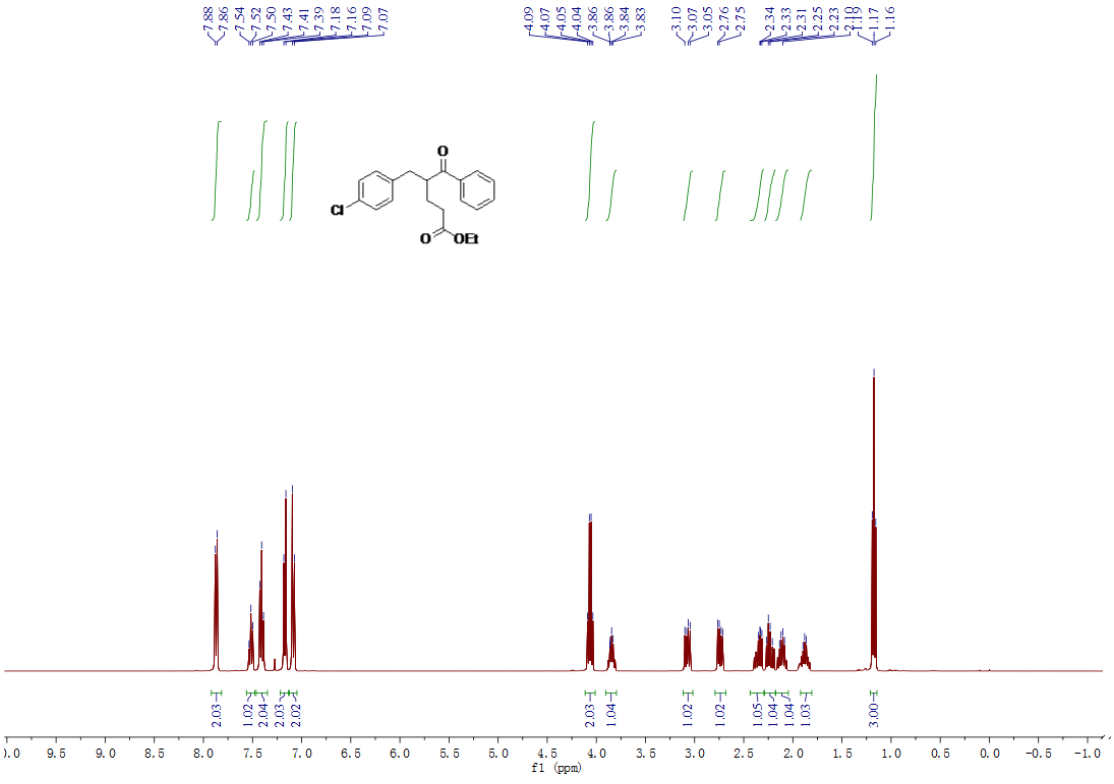
60.48
46.76
37.41
31.44
27.16
14.15
Cl
Cl
O
O
OEt
f1 (ppm)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7.09
7.07
4.09
4.07
4.05
4.04
3.86
3.84
3.83
3.10
3.07
3.05
2.76
2.75
2.34
2.33
2.31
2.25
2.23
2.19
1.17
1.16
Cl
O
O
OEt
2.03
1.02
2.04
2.03
2.02
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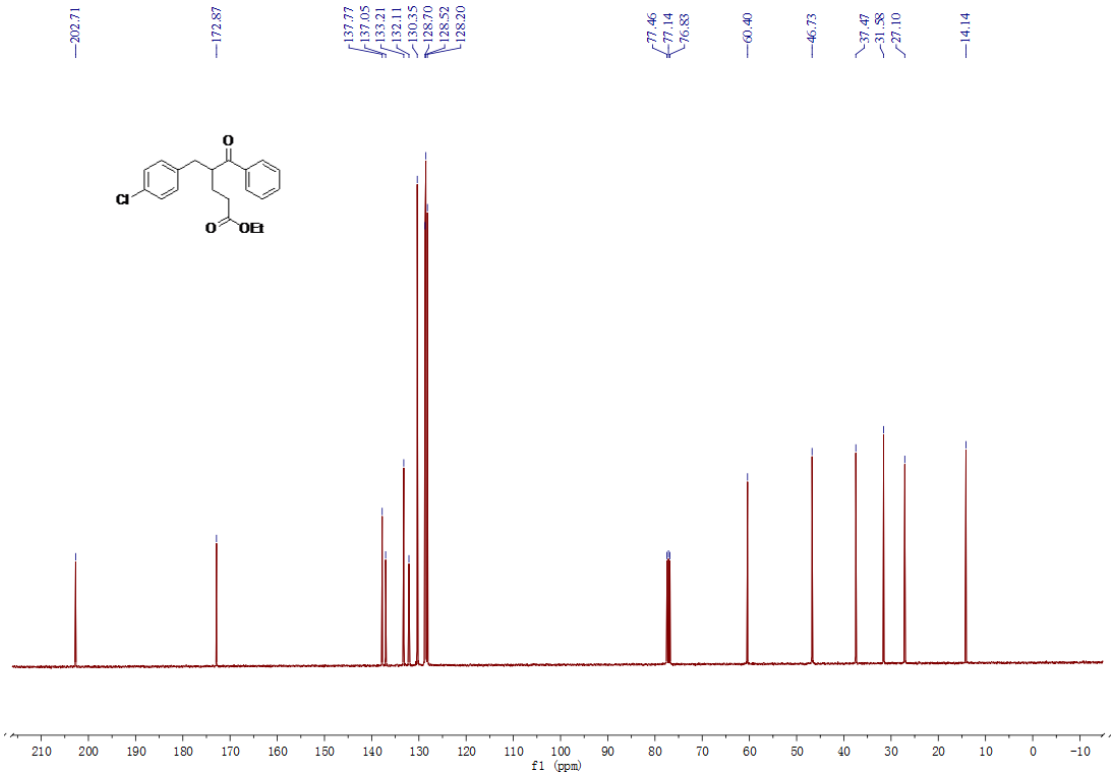
1.04
1.02
1.02
1.05
1.04
1.04
1.03
3.00
f1 (ppm)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128.20
77.46
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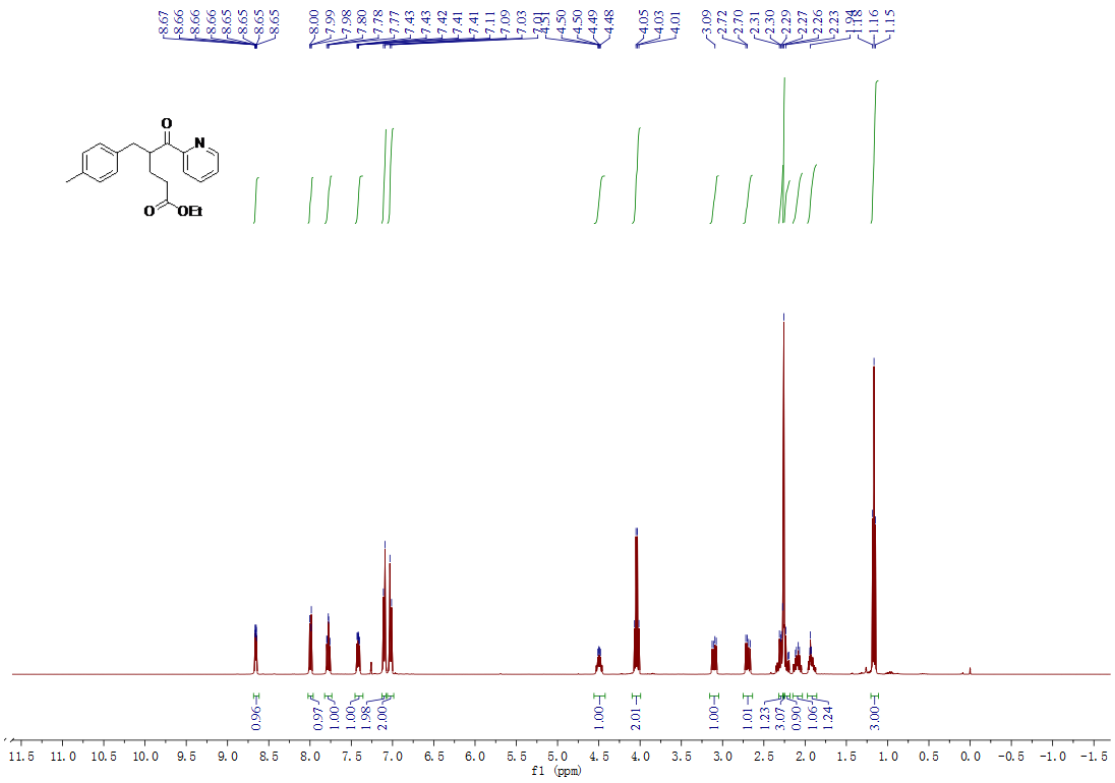
46.73
37.47
31.38
27.10
14.14
Cl
O
O
OEt
f1 (ppm)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8.65
8.00
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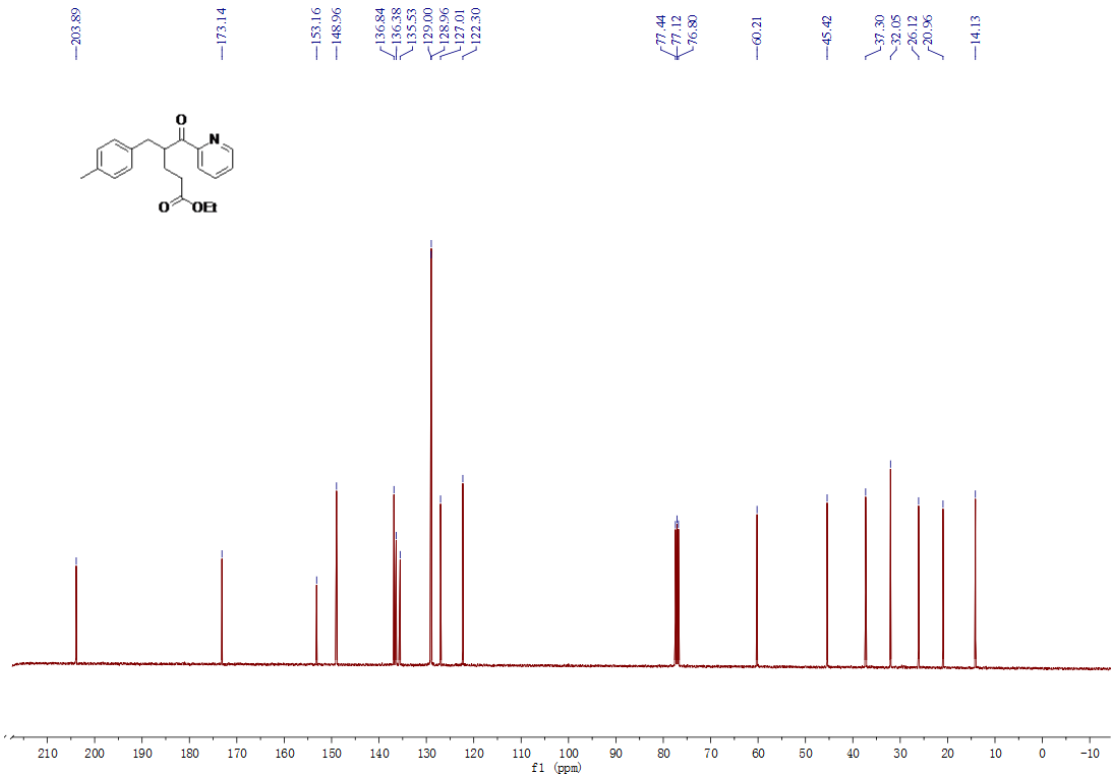
7.98
7.80
7.78
7.77
7.43
7.42
7.41
7.41
7.11
7.09
7.03
4.50
4.50
4.49
4.48
4.05
4.03
4.01
3.09
2.72
2.70
2.31
2.30
2.29
2.27
2.26
2.23
1.18
1.16
1.15
O
N
O
OEt
0.96
0.97
1.00
1.00
1.98
2.00
1.00
2.01
1.00
1.01
1.23
3.07
0.90
1.06
1.24
3.00
f1 (ppm)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127.01
122.30
77.44
77.12
76.80
60.21
45.42
37.30
32.05
26.12
20.96
14.13
O
N
O
OEt
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)