

Supporting Information

Oxygen-Directed Copper-Catalyzed, Diastereoselective Protosilylation of Cyclopropenes

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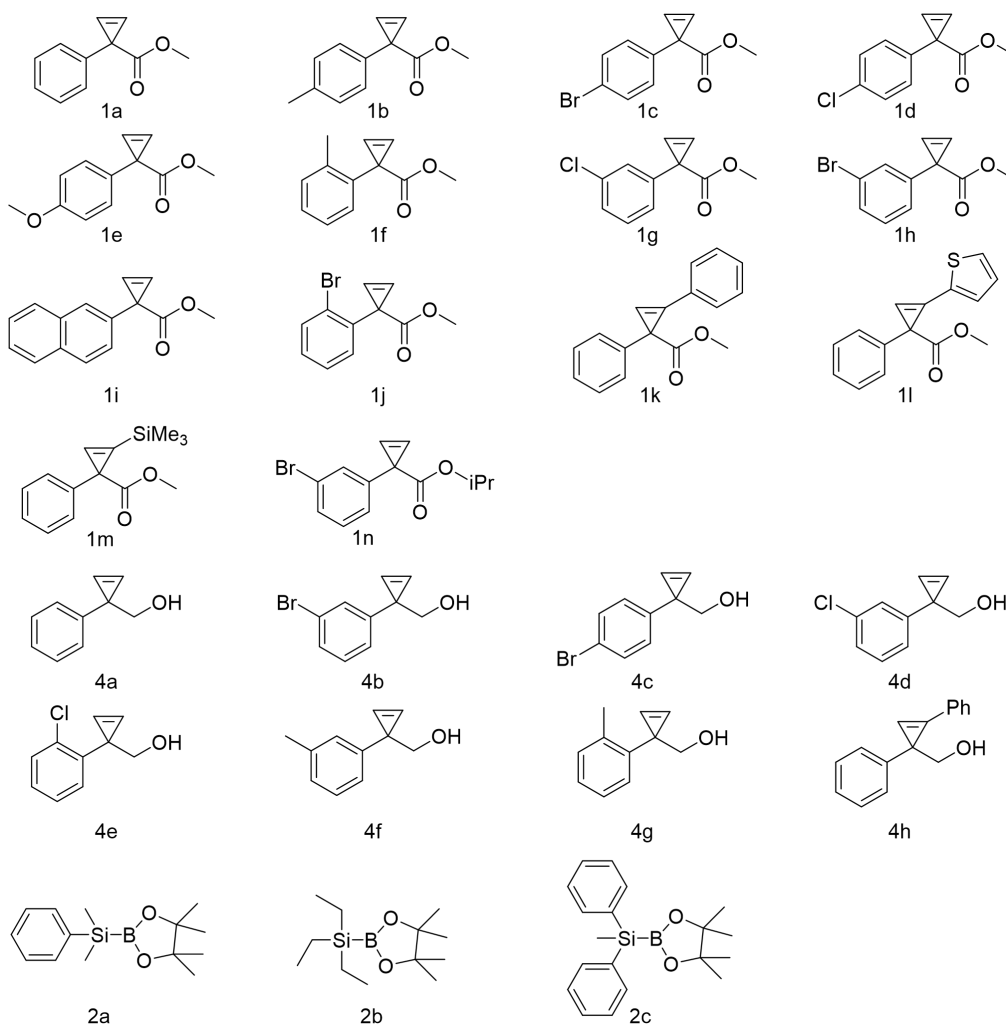
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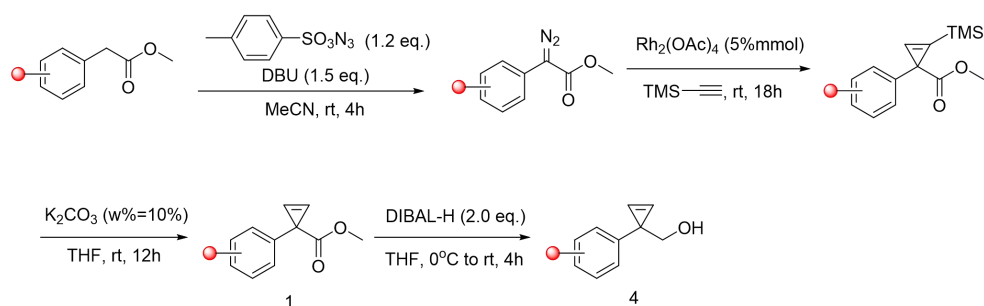
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1. General information

Unless otherwise noted, all the materials were commercially available and used without further purification. All solvents were dried before use according to the standard methods. All reactions were performed in an N₂-filled glovebox using standard Schlenk techniques unless otherwise noted. All reactions were monitored by thin-layer chromatography(TLC), visualized by UV. Chromatographic purification of products was accomplished by silica gel chromatography. ¹H NMR and ¹³C NMR spectra were recorded on a Bruker Avance II Noe400, Noe500. NMR data is reported relative to internal CDCl₃ (¹H, δ = 7.26 ppm; ¹³C, δ = 77.0 ppm). Data for ¹H NMR spectra are reported as follows: chemical shift (δ) in ppm; multiplicities are indicated s (singlet), brs (broad singlet), d (doublet), t (triplet), m (multiplet); coupling constants (J) are in Hertz (Hz). **2a** is Commercially available. The NMR data of **1a**^[1], **1b**^[2], **1c**^[2], **1d**^[3], **1h**^[2], **1k**^[4], **4a**^[5], **4h**^[6], **2b**^[7] and **2c**^[8] were confirmed according to the literature.



2. Experimental procedures



GPA: DBU (1.5 equiv.) and the indicated α -arylacetate (1.0 equiv.) were added to a solution of tosyl azide (1.2 equiv.) in CH₃CN (1 M) at room temperature and the resulting mixture was stirred for 4 hours. The reaction mixture was then diluted with distilled water and extracted with diethyl ether. The combined organic layer was washed with 10% NaHCO₃ solution and brine, then dried over MgSO₄, filtered, and concentrated under reduced pressure. The desired methyl diazophenylacetate was obtained after purification by flash column chromatography (SiO₂, hexane-EtOAc) as a red or Orange oil. Yield 95-97%.

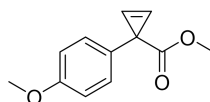
A solution of methyl diazophenylacetate (1.0 equiv.) in trimethylsilylacetylene (20 mL) was added via a syringe pump over 16 hours to a stirred suspension of Rh₂(OAc)₄ (5 mol%) in trimethylsilylacetylene (0.2 M) at 50 °C under N₂ atmosphere. After the addition was completed, the reaction mixture was refluxed for an additional 2 hours. The reaction mixture was concentrated under vacuum, diluted with DCM (20 mL). After the reaction was completed, wash the reaction mixture with water and extract with DCM. Remove the DCM by rotation evaporation and was purified by column chromatography (SiO₂, hexane-EtOAc) to give methyl 1-aromatic -2-(trimethylsilyl)cycloprop-2-ene-1-carboxylate as a oil. Yield 48-54%.

To a solution of methyl 1-aromatic -2-(trimethylsilyl)cycloprop-2-ene-1-carboxylate (1.0 equiv.) in THF (1 M), cooled to 0 °C, a solution of K₂CO₃ (w%=10%, 1.5 equiv) was added dropwise. The reaction mixture was stirred at r.t. for 2 hours, then extracted with EtOAc. The combined organic layers were washed with brine (20 mL), dried over Na₂SO₄, filtered and concentrated under vacuum to give the crude product, which was purified by column chromatography (SiO₂, hexane-EtOAc) to give methyl 1-aromatic cycloprop-2-ene-1-carboxylate as a yellow oil. Yield 72-78%.

GPB: To a stirred solution of give methyl 1-aromatic cycloprop-2-ene-1-carboxylate (1.0 equiv.) in dry THF (2 M), DIBAL-H (1.0 M solution in hexanes, 2.0 equiv.) was added dropwise at -78 °C. The mixture was stirred for 1 hours at -78 °C, then warmed to room temperature and stirred for additional 1 hours, and then quenched with saturated NH₄Cl. Formed gel was dissolved in 2 N aqueous HCl, and extracted with ether. Combined ethereal layer was washed with NaHCO₃ and brine, dried MgSO₄,

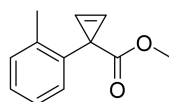
filtered, and evaporated. The residue was purified by column chromatography on Silica gel, eluent hexane-EtOAc to obtain alcohol **4**. Yield 72-76%.

methyl 1-(4-methoxyphenyl)cycloprop-2-ene-1-carboxylate (1e)



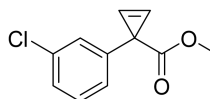
Prepared by GPA. $^1\text{H NMR}$ (500 MHz, Chloroform-d) δ 7.26 – 7.19 (m, 4H), 6.88 (d, J = 8.8 Hz, 2H), 3.81 (s, 3H), 3.72 (s, 3H). $^{13}\text{C NMR}$ (101 MHz, Chloroform-d) δ 175.8, 158.3, 133.8, 129.3, 113.6, 107.9, 55.2, 52.2, 29.9. HRMS(ESI): Calcd for $\text{C}_{12}\text{H}_{12}\text{O}_3$ $[\text{M}+\text{H}]^+$:205.0859, found 205.0859.

methyl 1-(o-tolyl)cycloprop-2-ene-1-carboxylate (1f)



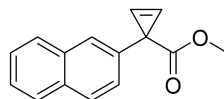
Prepared by GPA. $^1\text{H NMR}$ (500 MHz, Chloroform-d) δ 7.37 (s, 2H), 7.24 – 7.16 (m, 3H), 7.15 – 7.11 (m, 1H), 3.69 (s, 3H), 2.36 (s, 3H). $^{13}\text{C NMR}$ (126 MHz, Chloroform-d) δ 176.1, 137.3, 130.1, 128.4, 127.2, 126.2, 109.2, 52.5, 30.0, 19.2. HRMS(ESI): Calcd for $\text{C}_{12}\text{H}_{12}\text{O}_2$ $[\text{M}+\text{H}]^+$:189.0910, found 189.0910.

methyl 1-(3-chlorophenyl)cycloprop-2-ene-1-carboxylate (1g)



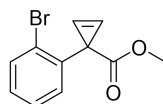
Prepared by GPA. $^1\text{H NMR}$ (400 MHz, Chloroform-d) δ 7.32 – 7.12 (m, 6H), 3.72 (s, 3H). $^{13}\text{C NMR}$ (101 MHz, Chloroform-d) δ 174.8, 143.6, 133.9, 129.4, 128.5, 126.8, 126.5, 107.3, 52.4, 30.2. HRMS(ESI): Calcd for $\text{C}_{11}\text{H}_9\text{ClO}_2$ $[\text{M}+\text{H}]^+$:209.0364, found 209.0364.

methyl 1-(naphthalen-2-yl)cycloprop-2-ene-1-carboxylate (1i)



Prepared by GPA. $^1\text{H NMR}$ (500 MHz, Chloroform-d) δ 7.92 – 7.80 (m, 3H), 7.72 (s, 1H), 7.56 – 7.43 (m, 3H), 7.34 (s, 2H), 3.77 (s, 3H). $^{13}\text{C NMR}$ (101 MHz, Chloroform-d) δ 175.6, 139.1, 133.4, 132.3, 127.8, 127.7, 127.6, 126.8, 126.7, 126.0, 125.7, 107.9, 52.4, 30.8. HRMS(ESI): Calcd for $\text{C}_{15}\text{H}_{12}\text{O}_2$ $[\text{M}+\text{H}]^+$:225.0910, found 225.0910.

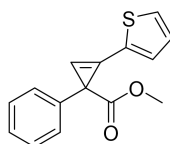
methyl 1-(2-bromophenyl)cycloprop-2-ene-1-carboxylate (1j)



Prepared by GPA. $^1\text{H NMR}$ (500 MHz, Chloroform-*d*) δ 7.56 (d, J = 8.0 Hz, 1H), 7.36 (s, 2H), 7.29 (d, J = 6.3 Hz, 1H), 7.22 (d, J = 1.5 Hz, 1H), 7.15 (s, 1H), 3.70 (s, 3H). $^{13}\text{C NMR}$ (126 MHz, Chloroform-*d*) δ 175.1, 141.7, 132.6, 130.3, 128.7, 127.7, 125.2, 108.8, 52.6, 32.4.

HRMS(ESI): Calcd for $\text{C}_{11}\text{H}_9\text{BrO}_2$ $[\text{M}+\text{H}]^+$:252.9859, found 252.9859.

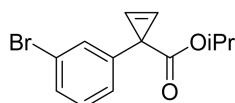
methyl 1-phenyl-2-(thiophen-2-yl)cycloprop-2-ene-1-carboxylate (**1l**)



Dissolve methyl diazophenylacetate in DCM (0.25 M). Add the resulting solution to a suspension of $\text{Rh}_2(\text{OAc})_4$ (0.01 equiv) in thienylacetylene (3.0 equiv) via syringe pump at room temperature 16 h. After the addition is completed, allow the reaction mixture to stir for another 2 h. The mixture was quenched with H_2O , and extracted with ethyl acetate. The combined organic layers were dried over anhydrous Na_2SO_4 , concentrated in vacuo. The crude product was purified by silica gel column chromatography to afford **1l**, yield 75%. $^1\text{H NMR}$ (500 MHz, Chloroform-*d*) δ 7.55 (dd, J = 5.1, 1.1 Hz, 1H), 7.49 (dd, J = 8.3, 1.2 Hz, 2H), 7.44 – 7.34 (m, 3H), 7.30 (d, J = 7.4 Hz, 1H), 7.16 (s, 1H), 7.13 (dd, J = 5.1, 3.7 Hz, 1H), 3.78 (s, 3H). $^{13}\text{C NMR}$ (126 MHz, Chloroform-*d*) δ 174.6, 140.4, 130.7, 130.3, 128.3, 128.1, 128.0, 127.8, 126.7, 111.4, 97.9, 52.3, 34.6.

HRMS(ESI): Calcd for $\text{C}_{15}\text{H}_{12}\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$:257.0631, found 257.0631.

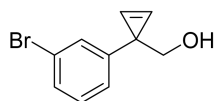
isopropyl 1-(3-bromophenyl)cycloprop-2-ene-1-carboxylate (**1n**)



Add LiBr (5.0 equiv) to a solution of **1h** (1.0 equiv) and tBuNH_2 (15.0 equiv) in iPrOH (0.1M) for 12 h at 85 °C in oil bath. The mixture was concentrated in vacuo to remove the most iPrOH . The crude product was purified by silica gel column chromatography to afford **1n**, yield 40%. $^1\text{H NMR}$ (500 MHz, Chloroform-*d*) δ 7.43 – 7.40 (m, 1H), 7.38 – 7.34 (m, 1H), 7.26 – 7.22 (m, 1H), 7.21 – 7.16 (m, 3H), 5.06 (hept, J = 6.3 Hz, 1H), 1.24 (d, J = 6.3 Hz, 6H). $^{13}\text{C NMR}$ (126 MHz, Chloroform-*d*) δ 173.8, 144.1, 131.3, 129.5, 129.5, 127.0, 122.0, 107.3, 68.6, 30.5, 21.8.

HRMS(ESI): Calcd for $\text{C}_{13}\text{H}_{14}\text{BrO}_2$ $[\text{M}+\text{H}]^+$:281.0172, found 281.0174.

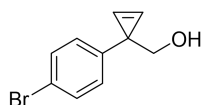
(1-(3-bromophenyl)cycloprop-2-en-1-yl)methanol (4b)



Prepared by GPB. ¹H NMR (500 MHz, Chloroform-d) δ 7.40 – 7.31 (m, 4H), 7.23 – 7.14 (m, 2H), 4.06 (s, 2H). ¹³C NMR (126 MHz, Chloroform-d) δ 148.4, 129.7, 129.6, 128.9, 125.0, 122.6, 112.8, 68.0, 28.9.

HRMS(ESI): Calcd for C₁₀H₉BrO [M+H]⁺:224.9910, found 224.9911.

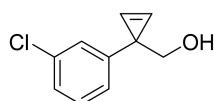
(1-(4-bromophenyl)cycloprop-2-en-1-yl)methanol (4c)



Prepared by GPB. ¹H NMR (500 MHz, Chloroform-d) δ 7.43 (d, *J* = 8.1 Hz, 2H), 7.35 (s, 2H), 7.13 (d, *J* = 8.1 Hz, 2H), 4.06 (s, 2H). ¹³C NMR (126 MHz, Chloroform-d) δ 144.9, 131.2, 128.2, 119.7, 112.9, 68.0, 28.8.

HRMS(ESI): Calcd for C₁₀H₉BrO [M+H]⁺:224.9910, found 224.9910.

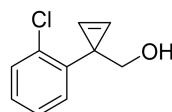
(1-(3-chlorophenyl)cycloprop-2-en-1-yl)methanol (4d)



Prepared by GPB. ¹H NMR (500 MHz, Chloroform-d) δ 7.35 (s, 2H), 7.27 – 7.21 (m, 2H), 7.21 – 7.13 (m, 2H), 4.07 (s, 2H). ¹³C NMR (126 MHz, Chloroform-d) δ 148.1, 134.3, 129.5, 126.7, 126.0, 124.5, 112.7, 68.0, 28.9.

HRMS(ESI): Calcd for C₁₀H₉ClO [M+H]⁺:181.0415, found 181.0415.

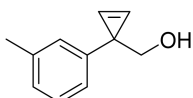
(1-(2-chlorophenyl)cycloprop-2-en-1-yl)methanol (4e)



Prepared by GPB. ¹H NMR (500 MHz, Chloroform-d) δ 7.67 (s, 2H), 7.34 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.30 (dd, *J* = 7.5, 1.9 Hz, 1H), 7.25 – 7.15 (m, 2H), 3.87 (s, 2H). ¹³C NMR (126 MHz, Chloroform-d) δ 142.6, 134.2, 130.7, 129.7, 128.1, 127.1, 116.9, 67.9, 30.2.

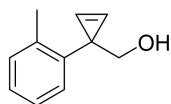
HRMS(ESI): Calcd for C₁₀H₉ClO [M+H]⁺:181.0415, found 181.0414.

(1-(*m*-tolyl)cycloprop-2-en-1-yl)methanol (4f)



Prepared by GPB. ¹H NMR (500 MHz, Chloroform-d) δ 7.37 (s, 2H), 7.26 – 7.18 (m, 1H), 7.11 – 7.00 (m, 3H), 4.10 (s, 2H), 2.36 (s, 3H). **¹³C NMR (126 MHz, Chloroform-d)** δ 145.6, 137.9, 128.2, 127.1, 126.7, 123.4, 113.3, 68.0, 29.1, 21.5. HRMS(ESI): Calcd for C₁₁H₁₂O [M+H]⁺:161.0961, found 161.0961.

(1-(o-tolyl)cycloprop-2-en-1-yl)methanol (4g)



Prepared by GPB. ¹H NMR (500 MHz, Chloroform-d) δ 7.69 (s, 2H), 7.23 (dd, *J* = 6.4, 3.1 Hz, 1H), 7.20 – 7.14 (m, 3H), 3.80 (s, 2H), 2.44 (s, 3H). **¹³C NMR (126 MHz, Chloroform-d)** δ 143.1, 136.6, 130.5, 129.1, 126.9, 126.2, 117.7, 67.7, 30.1, 19.2. HRMS(ESI): Calcd for C₁₁H₁₂O [M+H]⁺:161.0961, found 161.0961.

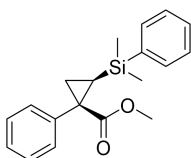
3. Experimental Procedures and Characterization of Products

3.1 General procedure

To an oven-dried 5 mL reaction tube were added NHC*i*Pr-Cu-Cl (4.8 mg, 0.01 mmol, 10 mol%), K₂CO₃ (13.8 mg, 0.1 mmol, 1.0 eq) and anhydrous MeOH (1 mL) in a nitrogen-filled glove box. The resulting mixture was stirred for 5 min, followed by adding 2-cyclopropene-1-carboxylic acid-1-phenyl-methyl ester (0.1 mmol, 1.0 equiv) and 2-(Dimethylphenylsilyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.11 mmol, 1.1 equiv) in sequence, and sealed with a screwed cap. The sealed tube was placed on r.t. for 12 h. The mixture was concentrated in vacuo, and extracted with ethyl acetate. The combined organic layers were dried over anhydrous Na₂SO₄, concentrated in vacuo. The crude product was purified by silica gel column chromatography to afford the silylated product.

3.2 Characterization of products

methyl (1*S*,2*R* / 1*R*,2*S*)-2-(dimethyl(phenyl)silyl)-1-phenylcyclopropane-1-carboxylate (3a)

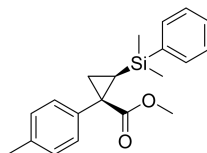


Prepared according to the general procedure from methyl 1-phenylcycloprop-2-ene-1-carboxylate (17.4 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **3a** (23.7 mg, 76% yield) as a transparent oil.

¹H NMR (500 MHz, Chloroform-*d*) δ 7.68 – 7.60 (m, 2H), 7.43 – 7.31 (m, 7H), 7.30 – 7.26 (m, 1H), 3.41 (s, 3H), 1.75 (dd, *J* = 8.9, 3.3 Hz, 1H), 1.54 (dd, *J* = 10.8, 3.3 Hz, 1H), 0.78 (dd, *J* = 10.7, 9.0 Hz, 1H), 0.47 (s, 3H), 0.42 (s, 3H). ¹³C NMR (126 MHz, Chloroform-*d*) δ 174.4, 141.3, 139.5, 133.6, 130.1, 128.8, 128.1, 127.7, 127.1, 52.0, 34.4, 19.8, 17.3, -1.6, -3.0.

HRMS(ESI): Calcd for C₁₉H₂₂O₂Si [M+H]⁺:311.1462, found 311.1463.

methyl (1*S*,2*R* / 1*R*,2*S*)-2-(dimethyl(phenyl)silyl)-1-(*p*-tolyl)cyclopropane-1-carboxylate (3b)

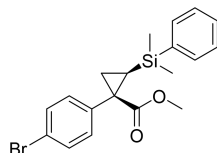


Prepared according to the general procedure from methyl 1-(*p*-tolyl)cycloprop-2-ene-1-carboxylate (18.8 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **3b** (22.7 mg, 75% yield) as a transparent oil.

¹H NMR (500 MHz, Chloroform-*d*) δ 7.71 – 7.57 (m, 2H), 7.45 – 7.35 (m, 3H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 7.9 Hz, 2H), 3.41 (s, 3H), 2.37 (s, 3H), 1.73 (dd,

$J = 8.9, 3.3$ Hz, 1H), 1.52 (dd, $J = 10.8, 3.4$ Hz, 1H), 0.77 (dd, $J = 10.7, 9.0$ Hz, 1H), 0.46 (s, 3H), 0.42 (s, 3H). ^{13}C NMR (126 MHz, Chloroform- d) δ 174.5, 139.6, 138.4, 136.8, 133.6, 129.9, 128.8, 128.8, 127.7, 52.0, 34.0, 21.2, 19.9, 17.4, -1.5, -2.9. HRMS(ESI): Calcd for $\text{C}_{20}\text{H}_{24}\text{O}_2\text{Si}$ $[\text{M}+\text{H}]^+$:325.1618, found 325, 1618.

methyl (1S,2R / 1R,2S)-1-(4-bromophenyl)-2-(dimethyl(phenyl)silyl) cyclopropane-1-carboxylate (3c)

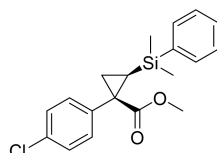


Prepared according to the general procedure from methyl 1-(4-bromophenyl) cycloprop-2-ene-1-carboxylate (25.2 mg, 0.1 mmol) and dimethyl (phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **3c** (25.6 mg, 66% yield) as a white solid.

^1H NMR (400 MHz, Chloroform- d) δ 7.64 (dd, $J = 6.4, 2.9$ Hz, 2H), 7.55 – 7.36 (m, 5H), 7.24 (d, $J = 8.3$ Hz, 2H), 3.41 (s, 3H), 1.77 (dd, $J = 9.0, 3.4$ Hz, 1H), 1.52 (dd, $J = 10.8, 3.4$ Hz, 1H), 0.74 (dd, $J = 10.6, 9.2$ Hz, 1H), 0.48 (s, 3H), 0.44 (s, 3H). ^{13}C NMR (101 MHz, Chloroform- d) δ 173.8, 140.4, 139.3, 133.6, 131.9, 131.2, 128.9, 127.8, 121.0, 52.1, 33.9, 19.9, 17.6, -1.5, -3.0.

HRMS(ESI): Calcd for $\text{C}_{19}\text{H}_{21}\text{BrO}_2\text{Si}$ $[\text{M}+\text{H}]^+$:389.0567, found 389.0572.

methyl (1S,2R / 1R,2S)-1-(4-chlorophenyl)-2-(dimethyl(phenyl)silyl) cyclopropane-1-carboxylate (3d)

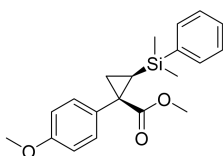


Prepared according to the general procedure from methyl 1-(4-chlorophenyl) cycloprop-2-ene-1-carboxylate (20.8 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **3d** (25.1 mg, 73% yield) as a transparent oil.

^1H NMR (400 MHz, Chloroform- d) δ 7.61 (dd, $J = 6.4, 2.9$ Hz, 2H), 7.46 – 7.35 (m, 3H), 7.32 – 7.24 (m, 4H), 3.40 (s, 3H), 1.74 (dd, $J = 9.0, 3.4$ Hz, 1H), 1.50 (dd, $J = 10.8, 3.4$ Hz, 1H), 0.72 (dd, $J = 10.6, 9.2$ Hz, 1H), 0.45 (s, 3H), 0.41 (s, 3H). ^{13}C NMR (126 MHz, Chloroform- d) δ 173.9, 139.8, 139.3, 133.6, 132.9, 131.5, 128.9, 128.2, 127.78, 52.1, 33.8, 19.9, 17.6, -1.6, -3.0.

HRMS(ESI): Calcd for $\text{C}_{19}\text{H}_{21}\text{ClO}_2\text{Si}$ $[\text{M}+\text{H}]^+$:345.1072, found 345.1072.

methyl (1S,2R / 1R,2S)-2-(dimethyl(phenyl)silyl)-1-(4-methoxyphenyl) cyclopropane-1-carboxylate (3e)

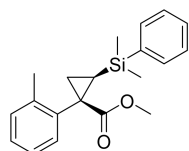


Prepared according to the general procedure from methyl 1-(4-methoxyphenyl)cycloprop-2-ene-1-carboxylate (20.4 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol). The crude residue was purified by column chromatography to yield **3e** (21.1 mg, 62% yield) as a transparent oil.

¹H NMR (400 MHz, Chloroform-d) δ 7.71 – 7.54 (m, 2H), 7.44 – 7.35 (m, 3H), 7.33 – 7.23 (m, 2H), 6.86 (d, J = 8.5 Hz, 2H), 3.82 (s, 3H), 3.39 (s, 3H), 1.71 (dd, J = 8.9, 3.2 Hz, 1H), 1.49 (dd, J = 10.7, 3.2 Hz, 1H), 0.80 – 0.66 (m, 1H), 0.45 (s, 3H), 0.41 (s, 3H). **¹³C NMR (101 MHz, CDCl₃)** δ 174.6, 158.5, 139.6, 133.6, 133.6, 131.1, 128.8, 127.7, 113.4, 55.3, 52.0, 33.6, 20.0, 17.5, -1.5, -3.0.

HRMS(ESI): Calcd for C₂₀H₂₄O₃Si [M+H]⁺:341.1567, found 341.1567.

methyl (1S,2R / 1R,2S)-2-(dimethyl(phenyl)silyl)-1-(o-tolyl)cyclopropane-1-carboxylate (3f)

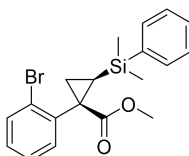


Prepared according to the general procedure from methyl 1-(o-tolyl)cycloprop-2-ene-1-carboxylate (18.8 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol). The crude residue was purified by column chromatography to yield **3f** (17.5 mg, 54% yield) as a transparent oil.

¹H NMR (500 MHz, Chloroform-d) δ 7.73 – 7.56 (m, 2H), 7.43 – 7.34 (m, 3H), 7.28 (d, J = 8.2 Hz, 1H), 7.23 – 7.07 (m, 3H), 3.45 (s, 3H), 2.32 (s, 3H), 1.81 (dd, J = 8.9, 3.2 Hz, 1H), 1.43 (dd, J = 10.8, 3.1 Hz, 1H), 0.85 – 0.77 (m, 1H), 0.46 (s, 3H), 0.46 (s, 3H). **¹³C NMR (126 MHz, Chloroform-d)** δ 174.5, 139.7, 139.7, 138.6, 133.7, 130.1, 129.9, 128.8, 127.7, 127.3, 125.6, 52.1, 33.1, 21.4, 19.6, 17.8, -1.8, -2.7.

HRMS(ESI): Calcd for C₂₀H₂₄O₂Si [M+H]⁺:325.1618, found 325.1627.

methyl (1S,2R / 1R,2S)-1-(2-bromophenyl)-2-(dimethyl(phenyl)silyl)cyclopropane-1-carboxylate (3g)

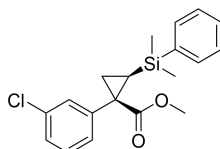


Prepared according to the general procedure from methyl 1-(2-bromophenyl)cycloprop-2-ene-1-carboxylate (25.2 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol). The crude residue was purified by column chromatography to yield **3g** (25.6 mg, 66% yield) as a transparent oil.

¹H NMR (500 MHz, Chloroform-d) δ 7.67 – 7.62 (m, 2H), 7.57 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.41 – 7.36 (m, 3H), 7.35 – 7.32 (m, 1H), 7.29 (td, *J* = 7.5, 1.2 Hz, 1H), 7.19 – 7.13 (m, 1H), 3.48 (s, 3H), 1.88 (dd, *J* = 9.1, 3.5 Hz, 1H), 1.47 (dd, *J* = 11.2, 3.7 Hz, 1H), 0.93 – 0.83 (m, 1H), 0.48 (s, 6H). **¹³C NMR (126 MHz, Chloroform-d)** δ 173.7, 140.9, 139.8, 133.7, 132.6, 131.8, 128.8, 128.7, 127.7, 127.2, 127.0, 52.3, 35.4, 22.4, 18.7, -1.8, -2.5.

HRMS(ESI): Calcd for C₁₉H₂₁BrO₂Si [M+H]⁺:389.0567, found 389.0568.

methyl (1S,2R / 1R,2S)-1-(3-chlorophenyl)-2-(dimethyl(phenyl)silyl)cyclopropane-1-carboxylate (3h)

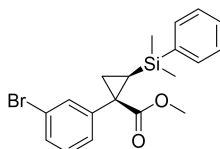


Prepared according to the general procedure from methyl 1-(3-chlorophenyl)cycloprop-2-ene-1-carboxylate (20.8 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **3g** (22.0 mg, 64% yield) as a transparent oil.

¹H NMR (400 MHz, Chloroform-d) δ 7.62 (dd, *J* = 5.7, 2.4 Hz, 2H), 7.50 – 7.36 (m, 3H), 7.32 (s, 1H), 7.26 – 7.21 (m, 3H), 3.41 (s, 3H), 1.74 (dd, *J* = 9.0, 3.5 Hz, 1H), 1.52 (dd, *J* = 10.9, 3.4 Hz, 1H), 0.88 – 0.67 (m, 1H), 0.45 (s, 3H), 0.42 (s, 3H). **¹³C NMR (126 MHz, Chloroform-d)** δ 173.7, 143.2, 139.2, 133.7, 133.6, 130.2, 129.3, 128.9, 128.4, 127.8, 127.3, 52.1, 34.1, 19.9, 17.5, -1.6, -3.0.

HRMS(ESI): Calcd for C₁₉H₂₁ClO₂Si [M+H]⁺:345.1072, found 345.1072.

methyl (1S,2R / 1R,2S)-1-(3-bromophenyl)-2-(dimethyl(phenyl)silyl)cyclopropane-1-carboxylate (3i)

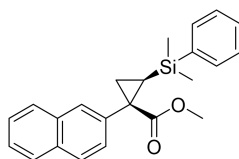


Prepared according to the general procedure from methyl 1-(3-bromophenyl)cycloprop-2-ene-1-carboxylate (25.2 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **3h** (22.5 mg, 58% yield) as a transparent oil.

¹H NMR (500 MHz, Chloroform-d) δ 7.69 – 7.56 (m, 2H), 7.48 (t, *J* = 1.8 Hz, 1H), 7.40 (dd, *J* = 5.1, 1.8 Hz, 4H), 7.32 – 7.23 (m, 1H), 7.19 (t, *J* = 7.8 Hz, 1H), 3.41 (s, 3H), 1.74 (dd, *J* = 9.0, 3.5 Hz, 1H), 1.52 (dd, *J* = 10.9, 3.5 Hz, 1H), 0.74 (dd, *J* = 10.8, 9.0 Hz, 1H), 0.45 (s, 3H), 0.42 (s, 3H). **¹³C NMR (126 MHz, Chloroform-d)** δ 173.7, 143.5, 139.2, 133.6, 133.1, 130.2, 129.6, 128.9, 128.9, 127.8, 121.9, 52.1, 34.1, 19.9, 17.5, -1.6, -3.0.

HRMS(ESI): Calcd for C₁₉H₂₁BrO₂Si [M+H]⁺:389.0567, found 389.0568.

methyl (1S,2R / 1R,2S)-2-(dimethyl(phenyl)silyl)-1-(naphthalen-2-yl)cyclopropane-1-carboxylate (3j)

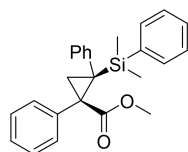


Prepared according to the general procedure from methyl 1-(naphthalen-2-yl)cycloprop-2-ene-1-carboxylate (22.4 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **3j** (25.9 mg, 72% yield) as a transparent oil.

¹H NMR (500 MHz, Chloroform-d) δ 7.88 – 7.79 (m, 3H), 7.78 (s, 1H), 7.71 – 7.66 (m, 2H), 7.54 – 7.46 (m, 3H), 7.45 – 7.37 (m, 3H), 3.42 (s, 3H), 1.83 (dd, $J = 9.0, 3.4$ Hz, 1H), 1.66 (dd, $J = 10.8, 3.4$ Hz, 1H), 0.88 (dd, $J = 10.9, 9.2$ Hz, 1H), 0.51 (s, 3H), 0.47 (s, 3H). **¹³C NMR (101 MHz, Chloroform-d)** δ 174.4, 139.5, 138.8, 133.7, 133.1, 132.5, 128.9, 128.8, 128.0, 127.8, 127.8, 127.6, 127.6, 126.1, 125.9, 52.0, 34.6, 20.1, 17.4, -1.5, -2.9.

HRMS(ESI): Calcd for C₂₃H₂₄O₂Si [M+H]⁺:361.1618, found 361.1618.

methyl (1S,2S / 1R,2S)-2-(dimethyl(phenyl)silyl)-1,2-diphenylcyclopropane-1-carboxylate (3k)

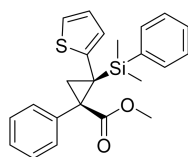


Prepared according to the general procedure from methyl 1,2-diphenylcycloprop-2-ene-1-carboxylate (25.0 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **3k** (16.2 mg, 42% yield) as a transparent oil.

¹H NMR (400 MHz, Chloroform-d) δ 7.52 – 7.44 (m, 2H), 7.38 – 7.29 (m, 3H), 7.20 (d, $J = 7.4$ Hz, 2H), 7.12 – 6.82 (m, 8H), 3.49 (s, 3H), 2.12 (d, $J = 5.0$ Hz, 1H), 2.00 (d, $J = 5.0$ Hz, 1H), 0.35 (s, 3H), 0.32 (s, 3H). **¹³C NMR (126 MHz, Chloroform-d)** δ 172.7, 140.0, 137.6, 135.8, 134.7, 129.6, 128.8, 127.5, 127.3, 127.2, 126.7, 125.1, 52.2, 40.5, 31.5, 20.2, -2.4, -3.1.

HRMS(ESI): Calcd for C₂₅H₂₆O₂Si [M+H]⁺:387.1775, found 387.1774.

methyl (1S,2R / 1R,2S)-2-(dimethyl(phenyl)silyl)-1-phenyl-2-(thiophen-2-yl)cyclopropane-1-carboxylate (3l)

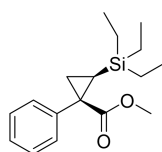


Prepared according to the general procedure from methyl 1-phenyl-2-(thiophen-2-yl)cycloprop-2-ene-1-carboxylate (25.6 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **3l** (30.5 mg, 78% yield) as a transparent oil.

¹H NMR (500 MHz, Chloroform-d) δ 7.59 (dd, J = 6.4, 3.1 Hz, 2H), 7.45 – 7.30 (m, 5H), 7.17 – 7.06 (m, 3H), 6.79 (dd, J = 5.1, 1.0 Hz, 1H), 6.61 (dd, J = 5.1, 3.6 Hz, 1H), 6.52 (dd, J = 3.5, 1.0 Hz, 1H), 3.44 (s, 3H), 2.16 (d, J = 5.0 Hz, 1H), 2.13 (d, J = 5.0 Hz, 1H), 0.42 (s, 3H), 0.41 (s, 3H). **¹³C NMR (126 MHz, Chloroform-d)** δ 172.2, 145.0, 137.4, 135.6, 134.6, 130.5, 129.0, 127.5, 127.4, 127.4, 127.1, 126.2, 122.9, 52.2, 41.9, 25.3, 22.5, -2.2, -2.7.

HRMS(ESI): Calcd for C₂₃H₂₄O₂SSi [M+H]⁺:393.1339, found 393.1338.

methyl (1S,2R/ 1R,2S)-1-phenyl-2-(triethylsilyl)cyclopropane-1-carboxylate (**3m**)

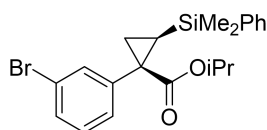


Prepared according to the general procedure from methyl 1-phenylcycloprop-2-ene-1-carboxylate (17.4 mg, 0.1 mmol) and triethyl(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (24.2 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **3m** (21.2 mg, 73% yield) as a transparent oil.

¹H NMR (500 MHz, Chloroform-d) δ 7.49 – 7.20 (m, 5H), 3.63 (s, 3H), 1.67 (dd, J = 9.2, 3.3 Hz, 1H), 1.44 (dd, J = 10.9, 3.3 Hz, 1H), 1.04 (t, J = 7.9 Hz, 9H), 0.81 – 0.60 (m, 6H), 0.51 (dd, J = 10.8, 9.2 Hz, 1H). **¹³C NMR (126 MHz, Chloroform-d)** δ 174.9, 141.8, 130.2, 128.1, 127.0, 52.2, 33.5, 19.5.

HRMS(ESI): Calcd for C₁₇H₂₆O₂Si [M+H]⁺:291.1775, found 291.1773.

isopropyl (1S,2R/ 1R,2S)-1-(3-bromophenyl)-2-(dimethyl(phenyl)silyl)cyclopropane-1-carboxylate (**3n**)

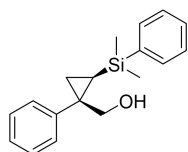


Prepared according to the general procedure from isopropyl 1-(3-bromophenyl)cycloprop-2-ene-1-carboxylate (28.1 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **3n** (20.0 mg, 48% yield) as a transparent oil.

¹H NMR (400 MHz, Chloroform-d) δ 7.66 – 7.61 (m, 2H), 7.48 – 7.44 (m, 1H), 7.42 – 7.35 (m, 4H), 7.28 – 7.24 (m, 1H), 7.16 (t, J = 7.8 Hz, 1H), 4.79 (hept, J = 6.3 Hz, 1H), 1.68 (dd, J = 9.0, 3.5 Hz, 1H), 1.49 (dd, J = 10.9, 3.5 Hz, 1H), 1.05 (d, J = 6.3 Hz, 3H), 1.00 (d, J = 6.3 Hz, 3H), 0.71 (dd, J = 10.8, 9.0 Hz, 1H), 0.47 (s, 3H), 0.41 (s, 3H). **¹³C NMR (101 MHz, Chloroform-d)** δ 172.9, 143.9, 139.7, 133.7, 132.8, 129.9, 129.4, 128.9, 128.7, 127.7, 121.7, 68.7, 34.4, 21.5, 21.5, 19.7, 17.3, -1.6, -2.9.

HRMS(ESI): Calcd for C₂₁H₂₅BrO₂SiNa [M+Na]⁺:439.0700, found 439.0702.

((1S,2R / 1R,2S)-2-(dimethyl(phenyl)silyl)-1-phenylcyclopropyl)methanol (5a)

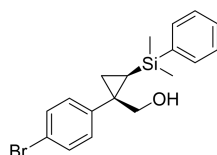


Prepared according to the general procedure from (1-phenylcycloprop-2-en-1-yl)methanol (14.6mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **5a** (21.4 mg, 80% yield) as a transparent oil.

¹H NMR (400 MHz, Chloroform-d) δ 7.65 (dd, *J* = 6.3, 3.0 Hz, 2H), 7.49 – 7.20 (m, 8H), 3.74 – 3.53 (m, 2H), 1.32 (dd, *J* = 10.2, 3.9 Hz, 1H), 0.92 (dd, *J* = 7.8, 3.9 Hz, 1H), 0.46 (s, 3H), 0.44 (s, 3H), 0.39 (dd, *J* = 10.1, 7.9 Hz, 1H). ¹³C NMR (101 MHz, Chloroform-d) δ 144.4, 139.3, 133.8, 129.2, 129.0, 128.4, 128.0, 126.6, 68.8, 33.8, 15.8, 12.1, -1.4, -1.7.

HRMS(ESI): Calcd for C₁₈H₂₂O₂Si [M+H]⁺:283.1513, found 283.1513.

((1S,2R / 1R,2S)-1-(4-bromophenyl)-2-(dimethyl(phenyl)silyl)cyclopropyl)methanol (5b)

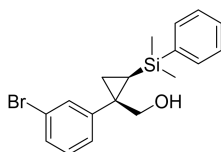


Prepared according to the general procedure from (1-(4-bromophenyl)cycloprop-2-en-1-yl)methanol (22.4 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **5b** (24.9 mg, 72% yield) as a transparent oil.

¹H NMR (500 MHz, Chloroform-d) δ 7.75 – 7.57 (m, 2H), 7.50 – 7.39 (m, 5H), 7.23 (d, *J* = 7.4 Hz, 2H), 3.66 – 3.53 (m, 2H), 1.28 (dd, *J* = 9.9, 3.9 Hz, 1H), 0.91 (dd, *J* = 7.4, 4.0 Hz, 1H), 0.45 (s, 3H), 0.44 (s, 3H), 0.38 – 0.32 (m, 1H). ¹³C NMR (126 MHz, Chloroform-d) δ 143.6, 139.0, 133.7, 131.4, 130.6, 129.3, 128.1, 120.3, 68.5, 33.3, 15.9, 12.4, -1.4, -1.9.

HRMS(ESI): Calcd for C₁₈H₂₁BrOSi [M+H]⁺:361.0618, found 361.0618.

((1S,2R / 1R,2S)-1-(3-bromophenyl)-2-(dimethyl(phenyl)silyl)cyclopropyl)methanol (5c)



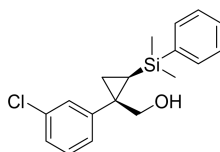
Prepared according to the general procedure from (1-(3-bromophenyl)cycloprop-2-en-1-yl)methanol (22.4 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-

dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **5b** (25.6 mg, 74% yield) as a transparent oil.

¹H NMR (500 MHz, Chloroform-d) δ 7.64 (dd, *J* = 6.3, 2.9 Hz, 2H), 7.49 (s, 1H), 7.45–7.40 (m, 3H), 7.37 (d, *J* = 7.9 Hz, 1H), 7.31 – 7.26 (m, 1H), 7.19 (t, *J* = 7.8 Hz, 1H), 3.72 – 3.52 (m, 2H), 1.31 (dd, *J* = 10.3, 4.0 Hz, 1H), 0.92 (dd, *J* = 7.8, 4.0 Hz, 1H), 0.46 (s, 3H), 0.44 (s, 3H), 0.37 (dd, *J* = 10.1, 8.0 Hz, 1H). **¹³C NMR (101 MHz, Chloroform-d)** δ 147.0, 138.9, 133.7, 131.9, 129.9, 129.7, 129.3, 128.1, 127.6, 122.4, 68.4, 33.5, 16.0, 12.5, -1.5, -1.9.

HRMS(ESI): Calcd for C₁₈H₂₁BrOSi [M+H]⁺:361.0618, found 361.0618.

((1S,2R / 1R,2S)-1-(3-chlorophenyl)-2-(dimethyl(phenyl)silyl)cyclopropyl) methanol (5d)

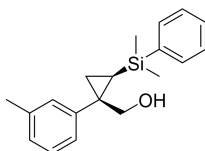


Prepared according to the general procedure from (1-(3-chlorophenyl)cycloprop-2-en-1-yl)methanol (18.0 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **5d** (19.3 mg, 64% yield) as a transparent oil.

¹H NMR (400 MHz, Chloroform-d) δ 7.64 (dd, *J* = 6.4, 3.0 Hz, 2H), 7.49 – 7.40 (m, 3H), 7.33 (s, 1H), 7.26 – 7.12 (m, 3H), 3.68 – 3.50 (m, 2H), 1.31 (dd, *J* = 10.4, 4.1 Hz, 1H), 0.92 (dd, *J* = 7.9, 4.0 Hz, 1H), 0.46 (s, 3H), 0.44 (s, 3H), 0.38 (dd, *J* = 10.1, 8.0 Hz, 1H). **¹³C NMR (126 MHz, Chloroform-d)** δ 146.6, 138.9, 134.1, 133.7, 129.6, 129.3, 129.0, 128.1, 127.0, 126.7, 68.4, 33.5, 16.0, 12.5, -1.5.

HRMS(ESI): Calcd for C₁₈H₂₁ClOSi [M+H]⁺:317.1123, found 317.1123.

((1S,2R / 1R,2S)-2-(dimethyl(phenyl)silyl)-1-(m-tolyl)cyclopropyl)methanol (5e)

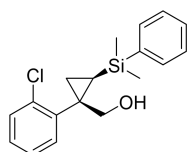


Prepared according to the general procedure from (1-(m-tolyl)cycloprop-2-en-1-yl)methanol (16.0 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **5f** (20.5 mg, 69% yield) as a transparent oil.

¹H NMR (400 MHz, Chloroform-d) δ 7.77 – 7.60 (m, 2H), 7.48 – 7.37 (m, 3H), 7.26 – 7.14 (m, 3H), 7.06 (d, *J* = 7.2 Hz, 1H), 3.75 – 3.49 (m, 2H), 2.37 (s, 3H), 1.32 (dd, *J* = 10.3, 3.8 Hz, 1H), 0.90 (dd, *J* = 7.8, 3.9 Hz, 1H), 0.47 (s, 3H), 0.44 (s, 3H), 0.41 – 0.34 (m, 1H). **¹³C NMR (126 MHz, Chloroform-d)** δ 144.2, 139.3, 138.0, 133.8, 129.6, 129.2, 128.3, 128.0, 127.4, 126.0, 68.8, 33.7, 21.5, 15.8, 12.0, -1.4, -1.7.

HRMS(ESI): Calcd for C₁₉H₂₄OSi [M+H]⁺:297.1669, found 297.1669.

((1S,2R / 1R,2S)-1-(2-chlorophenyl)-2-(dimethyl(phenyl)silyl)cyclopropyl) methanol (5f)



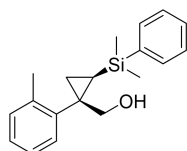
Prepared according to the general procedure from (1-(2-chlorophenyl)cycloprop-2-en-1-yl)methanol (18.0 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **5e** (20.8 mg, 69% yield) as a transparent oil.

¹H NMR (400 MHz, Chloroform-d) δ 7.66 (dd, *J* = 6.2, 2.8 Hz, 2H), 7.52 – 7.32 (m, 5H), 7.26 – 7.13 (m, 2H), 3.77 – 3.57 (m, 2H), 1.30 (dd, *J* = 10.4, 3.9 Hz, 1H), 0.98 (dd, *J* = 7.9, 4.1 Hz, 1H), 0.50 (s, 3H), 0.47 (s, 3H), 0.42 (dd, *J* = 11.8, 3.6 Hz, 1H).

¹³C NMR (126 MHz, Chloroform-d) δ 141.3, 139.1, 135.3, 133.8, 133.1, 129.7, 129.2, 128.2, 128.0, 126.6, 67.5, 33.4, 16.4, 11.7, -1.5, -1.7.

HRMS(ESI): Calcd for C₁₈H₂₁ClOSi [M+H]⁺:317.1123, found 317.1123.

((1S,2R / 1R,2S)-2-(dimethyl(phenyl)silyl)-1-(o-tolyl)cyclopropyl)methanol (5g)

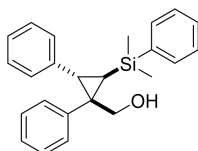


Prepared according to the general procedure from (1-(o-tolyl)cycloprop-2-en-1-yl)methanol (16.0 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **5g** (19.2 mg, 68% yield) as a transparent solid.

¹H NMR (500 MHz, Chloroform-d) δ 7.66 (dd, *J* = 6.4, 3.0 Hz, 2H), 7.49 – 7.37 (m, 3H), 7.33 – 7.27 (m, 1H), 7.21 – 7.11 (m, 3H), 3.75 – 3.50 (m, 2H), 2.42 (s, 3H), 1.25 (dd, *J* = 10.2, 3.8 Hz, 1H), 0.96 (dd, *J* = 7.8, 3.8 Hz, 1H), 0.50 (s, 3H), 0.48 (s, 3H), 0.36 (dd, *J* = 10.1, 7.8 Hz, 1H). **¹³C NMR (126 MHz, Chloroform-d)** δ 141.7, 139.2, 137.7, 133.8, 131.6, 130.5, 129.1, 127.9, 127.0, 125.7, -1.5, -1.7.

HRMS(ESI): Calcd for C₁₉H₂₄OSi [M+H]⁺:297.1669, found 297.1673.

((1S,2R,3S / 1R,2S,3R)-2-(dimethyl(phenyl)silyl)-1,3-diphenylcyclopropyl)methanol (5h)

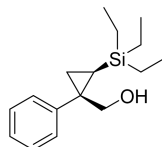


Prepared according to the general procedure from (1,2-diphenylcycloprop-2-en-1-yl)methanol (22.2 mg, 0.1 mmol) and dimethyl(phenyl)(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (28.8 mg, 0.11 mmol), The crude residue was purified by column chromatography to yield **5h** (15.0 mg, 42% yield) as a transparent oil.

¹H NMR (500 MHz, Chloroform-d) δ 7.78 – 7.63 (m, 2H), 7.49 – 7.38 (m, 3H), 7.23 – 7.02 (m, 8H), 6.93 – 6.77 (m, 2H), 3.85 (d, *J* = 11.4 Hz, 1H), 3.54 (d, *J* = 11.4 Hz, 1H), 2.42 (d, *J* = 8.1 Hz, 1H), 1.08 (d, *J* = 8.0 Hz, 1H), 0.56 (s, 3H), 0.54 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 139.9, 139.1, 139.0, 133.9, 131.1, 129.3, 128.1, 128.0, 127.7, 127.6, 126.7, 125.6, 70.0, 43.3, 32.6, 17.3, -1.4, -1.6.
HRMS(ESI): Calcd for $\text{C}_{24}\text{H}_{26}\text{OSi}$ $[\text{M}+\text{H}]^+$:359.1826, found 359.1826.

((1*S*,2*R* / 1*R*, 2*S*)-1-phenyl-2-(triethylsilyl)cyclopropyl)methanol (5i**)**

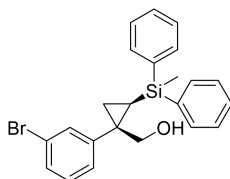


Prepared according to the general procedure from (1-phenylcycloprop-2-en-1-yl)methanol (14.6 mg, 0.1 mmol) and triethyl(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (24.2 mg, 0.11 mmol). The crude residue was purified by column chromatography to yield **5i** (27.2 mg, 93% yield) as a transparent oil.

^1H NMR (500 MHz, Chloroform-*d*) δ 7.44 – 7.37 (m, 2H), 7.34 (t, J = 7.6 Hz, 2H), 7.30 – 7.20 (m, 1H), 3.98 – 3.81 (m, 1H), 3.65 – 3.51 (m, 1H), 1.37 – 1.19 (m, 1H), 1.05 (t, J = 7.9 Hz, 9H), 0.86 (dd, J = 8.0, 3.8 Hz, 1H), 0.75 – 0.57 (m, 6H), 0.11 (dd, J = 10.3, 8.0 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 144.5, 129.0, 128.4, 126.6, 69.4, 33.0, 15.4, 9.5, 7.6, 4.5.

HRMS(ESI): Calcd for $\text{C}_{16}\text{H}_{26}\text{OSi}$ $[\text{M}+\text{Na}]^+$:285.1645, found 285.1643.

((1*S*,2*R* / 1*R*, 2*S*)-1-(3-bromophenyl)-2-(methyl-diphenylsilyl)cyclopropyl)methanol (5j**)**



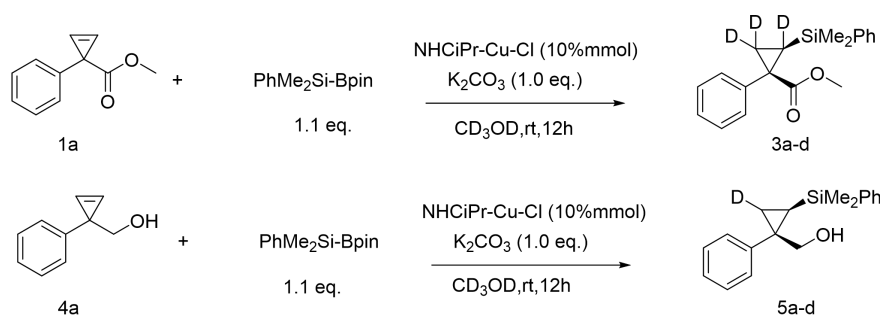
Prepared according to the general procedure from (1-(3-bromophenyl)cycloprop-2-en-1-yl)methanol (22.4 mg, 0.1 mmol) and methyl-diphenyl(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)silane (32.4 mg, 0.11 mmol). The crude residue was purified by column chromatography to yield **5j** (16.8 mg, 40% yield) as a transparent oil.

^1H NMR (500 MHz, Chloroform-*d*) δ 7.75 – 7.60 (m, 5H), 7.52 (d, J = 1.9 Hz, 1H), 7.48 – 7.31 (m, 10H), 7.20 (d, J = 7.8 Hz, 1H), 3.57 (d, J = 11.7 Hz, 1H), 3.48 (d, J = 11.7 Hz, 1H), 1.44 (dd, J = 10.3, 4.0 Hz, 1H), 1.04 (dd, J = 7.9, 4.0 Hz, 1H), 0.73 (s, 3H), 0.70 – 0.61 (m, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 146.8, 137.1, 137.1, 136.9, 134.7, 134.6, 134.0, 131.8, 129.9, 129.7, 129.6, 129.5, 128.2, 128.0, 127.9, 127.5, 122.4, 68.1, 33.5, 16.2, 11.4, -1.2, -3.0.

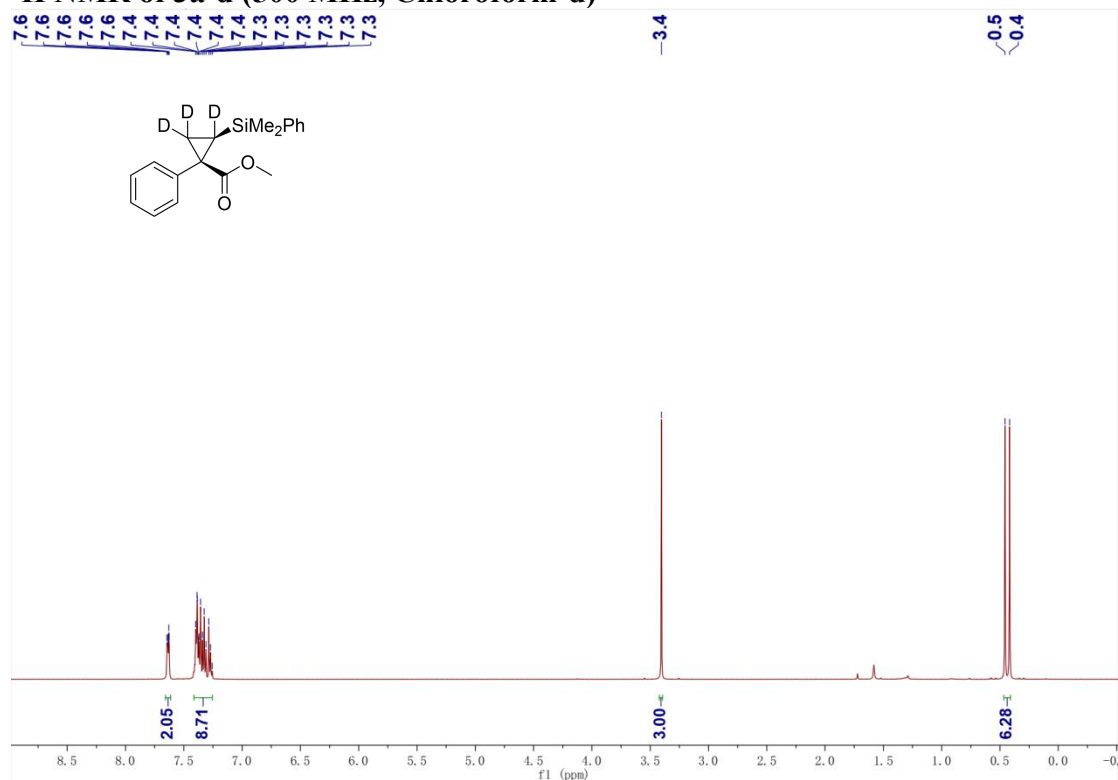
HRMS(ESI): Calcd for $\text{C}_{23}\text{H}_{23}\text{BrOSi}$ $[\text{M}+\text{Na}]^+$:445.0594, found 445.0590.

4. Mechanistic experiment

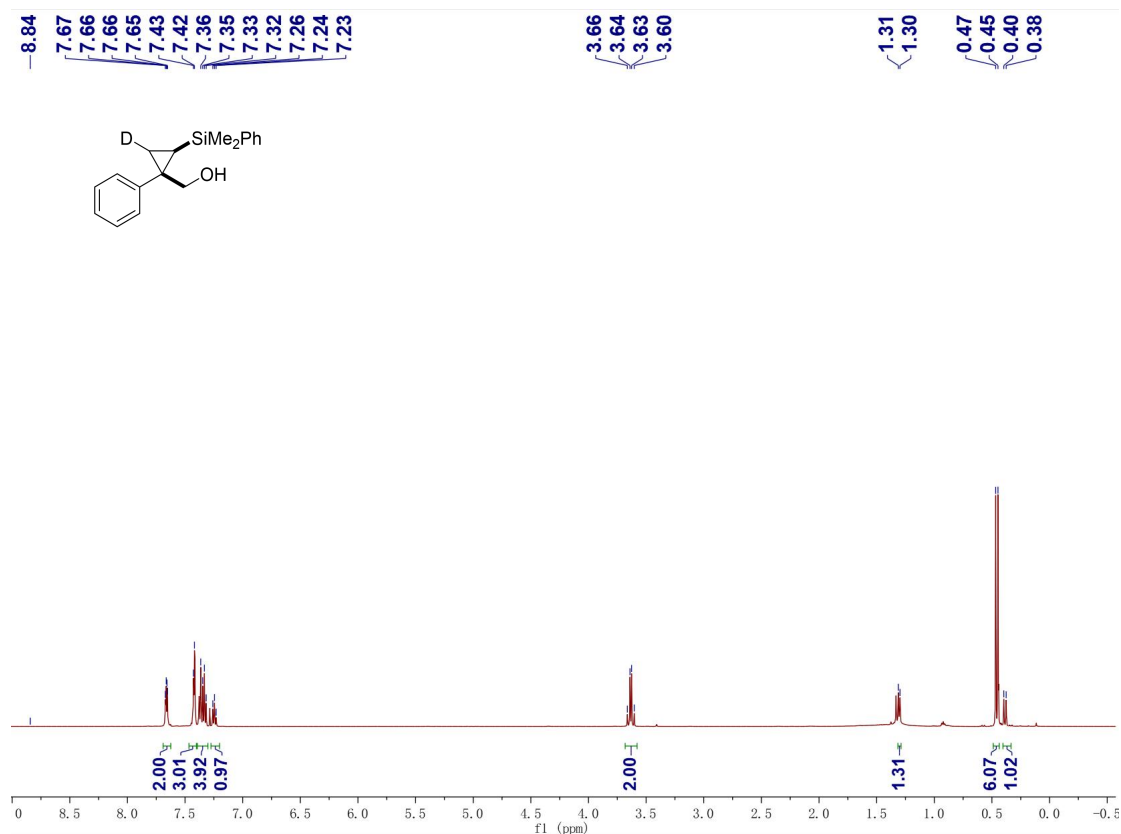
H/D exchange experiments(a/b): In an nitrogen-filled glovebox, a screw-capped test tube was charged with K_2CO_3 (0.10 mmol, 13.8 mg, 1.0 equiv), $NHCiPr-Cu-Cl$ (0.01 mmol, 4.8 mg, 10 mol%), **1a** or **4a** (0.1 mmol, 1.0 equiv) and $PhMe_2Si-Bpin$ (0.11 mmol, 28.8 mg, 1.1 equiv) are added sequentially. The test tube was capped and brought out of the glovebox. The reaction mixture was stirred for 12 h at room temperature. The mixture was concentrated in vacuo and directly purified by silica gel column chromatography (hexane-EtOAc) to afford **3a-d** or **5a-d**.



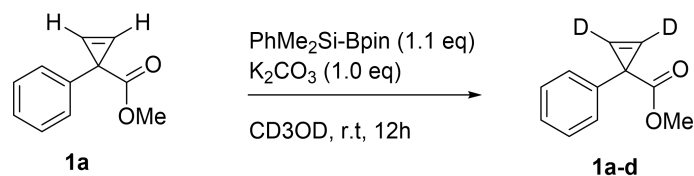
1H NMR of **3a-d** (500 MHz, Chloroform-d)



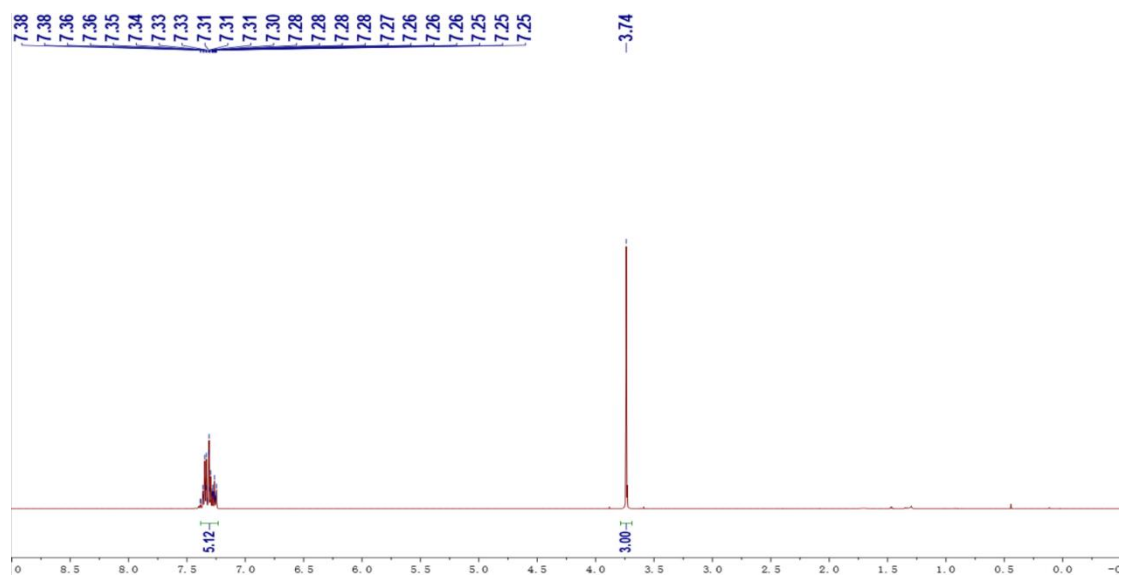
1H NMR of **5a-d** (500 MHz, Chloroform-d)



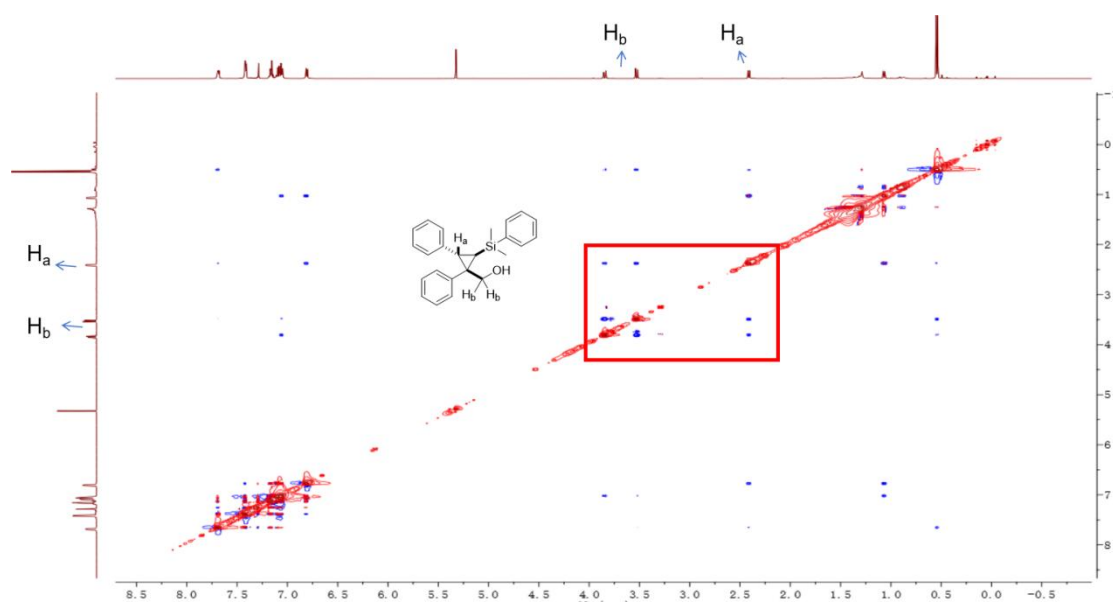
H/D exchange experiments(e): In a nitrogen-filled glovebox, a screw-capped test tube was charged with K_2CO_3 (0.10 mmol, 13.8 mg, 1.0 equiv). After the addition of MeOH-d_4 (1 mL), **1a** (0.1 mmol, 1.0 equiv) and $\text{PhMe}_2\text{Si-Bpin}$ (0.11 mmol, 28.8 mg, 1.1 equiv) are added sequentially. The test tube was capped and brought out of the glovebox. The reaction mixture was stirred for 12 h at room temperature. The mixture was concentrated in vacuo and directly purified by silica gel column chromatography (hexane-EtOAc) to afford **1a-d**.

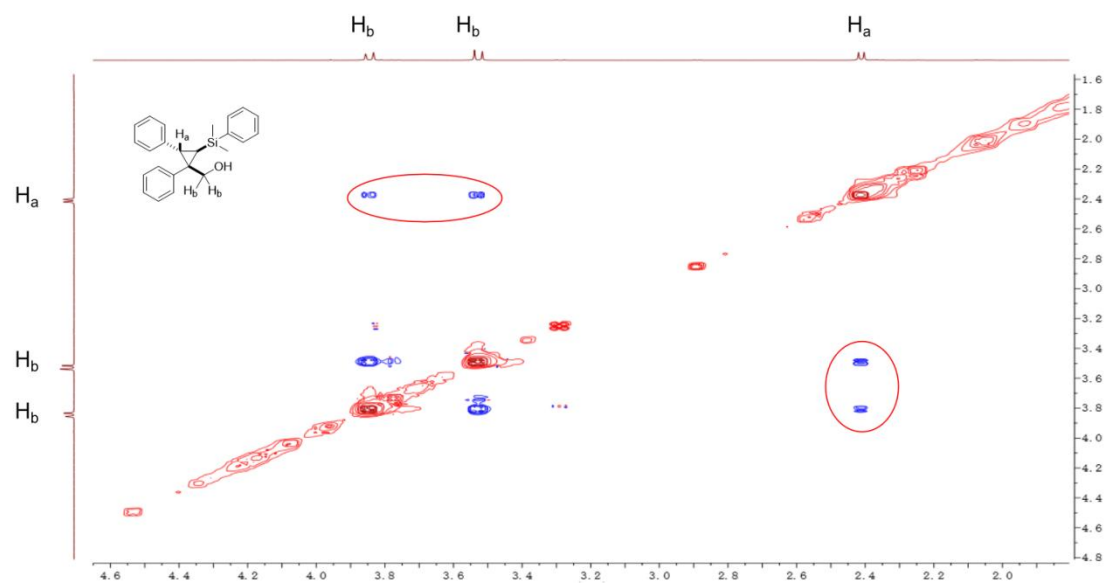


^1H NMR of 1a-d (500 MHz, Chloroform-d)

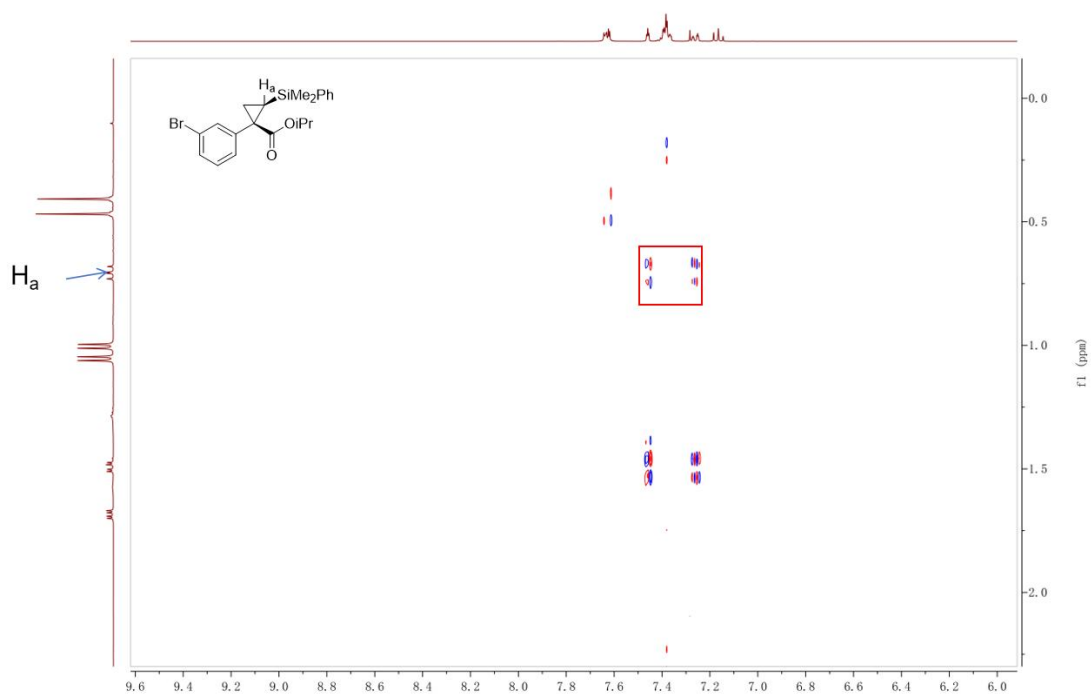
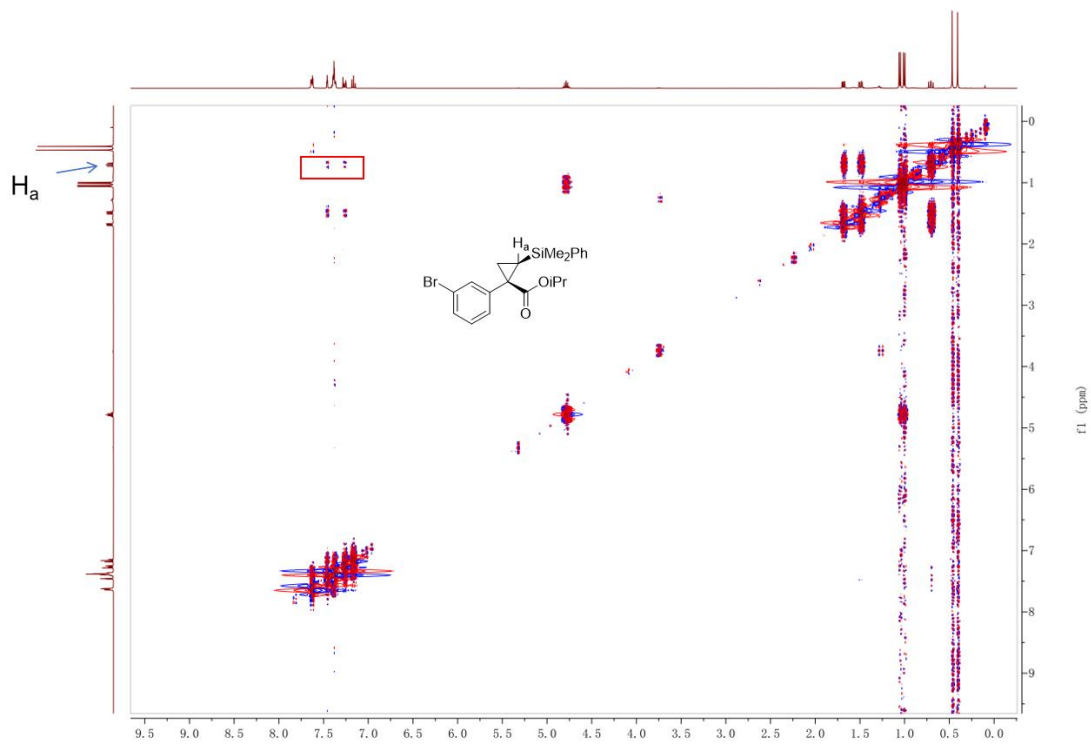


NOE experiments: According to the NOSEY 2D-1H NMR of **5h**, there is a signal between **H_a** and **H_b**.

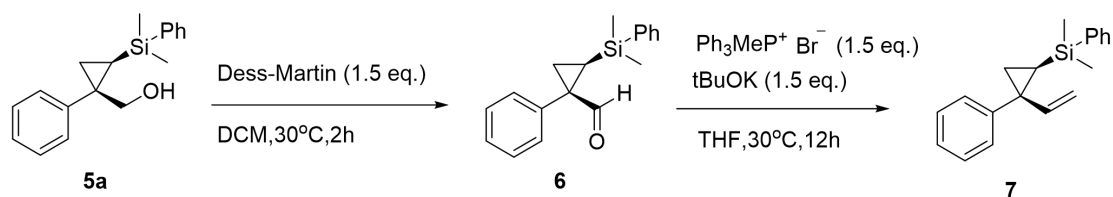




NOE experiments: According to the NOSEY 2D- 1H NMR of **3n**, there is a signal between H_a and meta-Br- C_6H_4 .

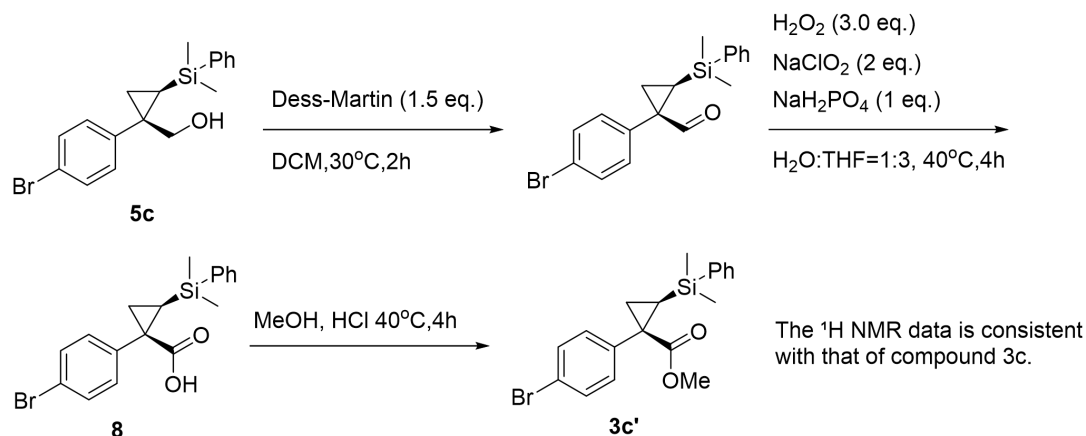


5. Derivatization Experiment



Add Dess-Martin (127.2 mg, 0.3 mmol) to a solution of **5a** (0.2 mmol) in anhydrous CH_2Cl_2 (2 mL) for 4 h at 30 °C in oil bath. The mixture was quenched with H_2O , and extracted with dichloromethane. The combined organic layers were dried over anhydrous Na_2SO_4 , concentrated in vacuo. The crude product was purified by silica gel column chromatography to afford **6**, yield 98%.

Charge an oven-dried round-bottom flask with $\text{CH}_3\text{PPh}_3^+\text{Br}^-$ (1.5 equiv) and THF (0.2 mL), and add *t*BuOK (1.5 equiv) to the suspension at 0 °C. Then allow the resulting mixture to warm up to room temperature, stir the resulting mixture for 1 h. Cool the yellow suspension to 0 °C, add **6** (1 equiv) portionwise to the mixture. Stir the mixture at room temperature for 12 h. The combined organic layers were dried over anhydrous Na_2SO_4 , concentrated in vacuo. The crude product was purified by silica gel column chromatography to afford **7**, yield 82%.

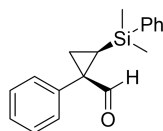


Add Dess-Martin (127.2 mg, 0.3 mmol) to a solution of **5c** (0.2 mmol) in anhydrous CH_2Cl_2 (2 mL) for 4 h at 30 °C in oil bath. The mixture was quenched with H_2O , and extracted with dichloromethane. The combined organic layers were dried over anhydrous Na_2SO_4 , concentrated in vacuo. The crude product was purified by silica gel column chromatography to afford aldehyde, yield 98%.

Add NaClO_2 (2.0 equiv.), NaH_2PO_4 (1.0 equiv.) and H_2O_2 (3.0 equiv.) to a solution of aldehyde (1.0 equiv.) in $\text{H}_2\text{O}:\text{THF}=1:3$ (2 mL) at 40 °C in oil bath, and the resulting mixture was stirred for 4 hours. The reaction mixture was then diluted with distilled

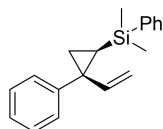
water and extracted with diethyl ether, then dried over MgSO₄, concentrated in vacuo. The crude product was purified by silica gel column chromatography to afford **8**, yield 76%.

(1S,2R / 1R,2S)-2-(dimethyl(phenyl)silyl)-1-phenylcyclopropane-1-carbaldehyde (6)



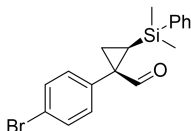
¹H NMR (400 MHz, Chloroform-*d*) δ 9.14 (s, 1H), 7.81 – 7.55 (m, 2H), 7.51 – 7.20 (m, 8H), 1.84 (dd, *J* = 10.2, 4.0 Hz, 1H), 1.68 (dd, *J* = 9.1, 4.0 Hz, 1H), 0.98 (t, *J* = 9.7 Hz, 1H), 0.50 (s, 3H), 0.49 (s, 3H). **¹³C NMR (126 MHz, Chloroform-*d*)** δ 200.7, 138.8, 138.2, 133.7, 129.9, 129.4, 128.5, 128.1, 127.5, 43.3, 19.8, 18.9, -1.7, -1.8. HRMS(ESI): Calcd for C₁₈H₂₀OSi [M+H]⁺:281.1356, found 281.1353.

dimethyl(phenyl)((1R,2R)-2-phenyl-2-vinylcyclopropyl)silane (7)



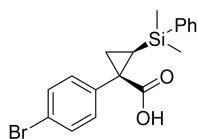
¹H NMR (500 MHz, Chloroform-*d*) δ 7.63 (dd, *J* = 6.3, 2.9 Hz, 2H), 7.44 – 7.37 (m, 3H), 7.34 – 7.29 (m, 4H), 7.26 – 7.20 (m, 1H), 5.77 (dd, *J* = 17.0, 10.4 Hz, 1H), 4.95 (d, *J* = 10.4 Hz, 1H), 4.62 (d, *J* = 18.0 Hz, 1H), 1.45 (dd, *J* = 10.4, 3.8 Hz, 1H), 1.16 (dd, *J* = 8.0, 3.8 Hz, 1H), 0.61 (dd, *J* = 10.3, 8.1 Hz, 1H), 0.42 (s, 3H), 0.41 (s, 3H). **¹³C NMR (126 MHz, Chloroform-*d*)** δ 144.6, 143.4, 139.5, 133.8, 129.7, 128.9, 128.1, 127.8, 126.3, 114.3, 34.2, 18.4, 14.0, -1.6, -1.8. HRMS(ESI): Calcd for C₁₉H₂₂Si [M+H]⁺:279.1564, found 279.1564.

(1S,2R / 1R,2S)-1-(4-bromophenyl)-2-(dimethyl(phenyl)silyl)cyclopropane-1-carbaldehyde (7)



¹H NMR (400 MHz, Chloroform-*d*) δ 9.01 (s, 1H), 7.60 (dd, *J* = 6.3, 3.0 Hz, 2H), 7.53 – 7.37 (m, 5H), 7.13 (d, *J* = 8.4 Hz, 2H), 1.82 (dd, *J* = 10.2, 4.2 Hz, 1H), 1.68 (dd, *J* = 9.1, 4.2 Hz, 1H), 0.93 (t, *J* = 9.7 Hz, 1H), 0.51 (s, 3H), 0.49 (s, 3H). **¹³C NMR (126 MHz, Chloroform-*d*)** δ 199.87, 137.77, 137.7, 133.7, 131.6, 129.6, 128.2, 121.6, 42.8, 19.6, 18.7, -1.7, -1.8. HRMS(ESI): Calcd for C₁₈H₁₉BrOSi [M+H]⁺:359.0461, found 359.0456.

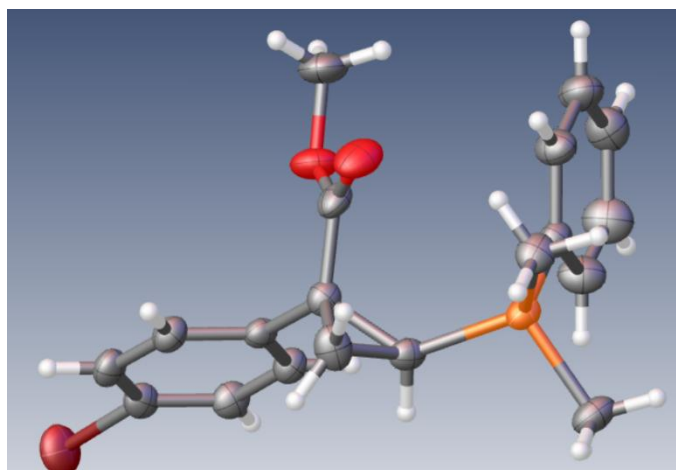
(1S,2R / 1R,2S)-1-(4-bromophenyl)-2-(dimethyl(phenyl)silyl)cyclopropane-1-carboxylic acid (8)



¹H NMR (400 MHz, Chloroform-d) δ 7.58 (dd, $J = 6.3, 2.9$ Hz, 2H), 7.45 (d, $J = 8.4$ Hz, 2H), 7.41 – 7.33 (m, 3H), 7.22 (d, $J = 8.4$ Hz, 2H), 1.72 (dd, $J = 9.2, 3.4$ Hz, 1H), 1.59 (dd, $J = 10.8, 3.4$ Hz, 1H), 0.84 – 0.65 (m, 1H), 0.43 (s, 3H), 0.40 (s, 3H). **¹³C NMR (101 MHz, Chloroform-d)** δ 180.2, 139.6, 139.2, 133.5, 131.8, 131.3, 128.9, 127.8, 121.3, 33.5, 20.9, 19.1, -1.9, -2.8.
HRMS(ESI): Calcd for C₁₈H₁₉BrO₂Si [M+H]⁺:375.0410, found 375.0410.

6. Crystal data and structure of products 3c

Sample preparation and structure refinement of **3c**. The compound **3c** was dissolved in the n-hexane and kept at room temperature for slow evaporation to obtain crystals. Plate colorless crystals were formed, which were subjected to X-ray diffraction. Single crystals of $C_{19}H_{21}BrO_2Si$ (**3c**) were grown by slow evaporation from a mixed solvent system of n-hexane. A suitable crystal was selected and mounted on a MiTeGen loop on a Bruker APEX-II CCD diffractometer. The crystal was kept at 180.00(10) K during data collection. Using Olex2, the structure was solved with the ShelXS structure solution program using Direct Method and refined with the ShelXL refinement package using least-squares minimisation. CCDC Deposition Number: 2517408



Empirical formula	$C_{19}H_{21}BrO_2Si$
Formula weight	389.36
Temperature/K	180.00
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	12.5608(8)
b/Å	6.7690(4)
c/Å	21.8646(14)
$\alpha/^\circ$	90
$\beta/^\circ$	97.770(2)
$\gamma/^\circ$	90
Volume/Å ³	1841.9(2)

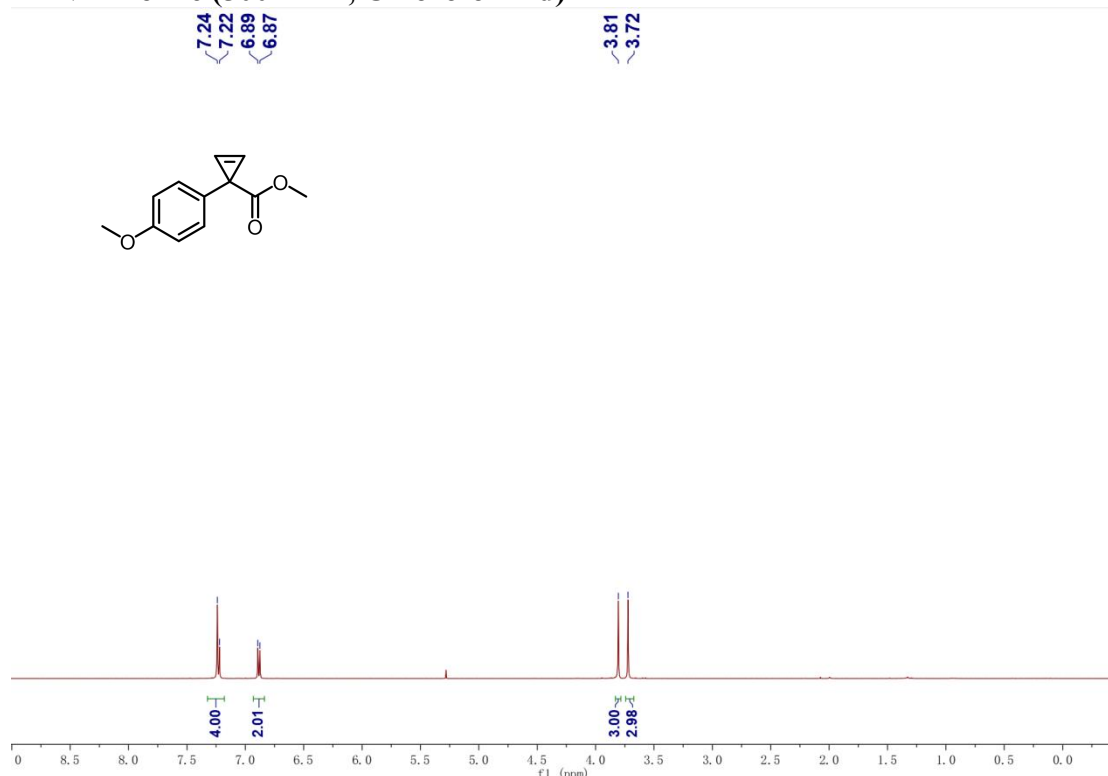
Z	4
$\rho_{\text{calc}}/\text{cm}^3$	1.404
μ/mm^{-1}	2.443
F(000)	800.0
Crystal size/ mm^3	0.56 × 0.2 × 0.19
Radiation	GaK α ($\lambda = 1.34138$)
2 θ range for data collection/ $^\circ$	6.696 to 144.684
Index ranges	-17 ≤ h ≤ 17, -8 ≤ k ≤ 9, -30 ≤ l ≤ 29
Reflections collected	24979
Independent reflections	5052 [$R_{\text{int}} = 0.0730$, $R_{\text{sigma}} = 0.0613$]
Data/restraints/parameters	5052/0/212
Goodness-of-fit on F^2	1.108
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0630$, $wR_2 = 0.1694$
Final R indexes [all data]	$R_1 = 0.0744$, $wR_2 = 0.1791$
Largest diff. peak/hole / $e \text{ \AA}^{-3}$	0.73/-0.94

7. Reference

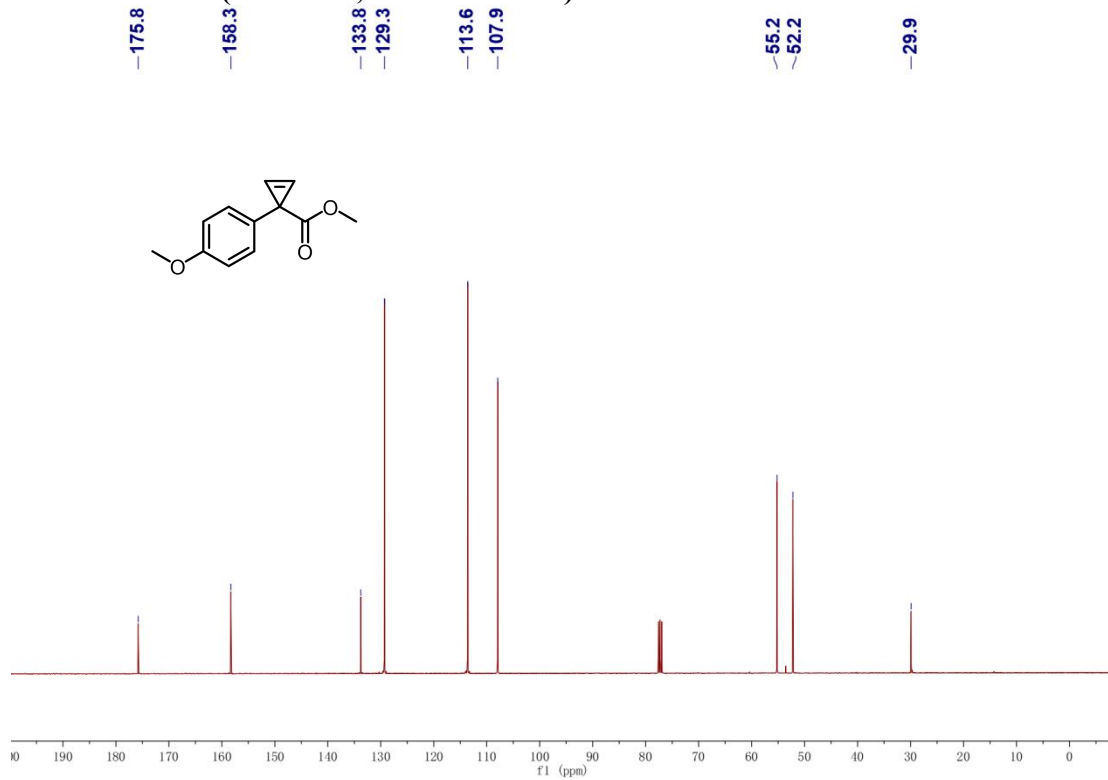
- [1] Lefebvre, G.; Charron, O.; Cossy, J.; Meyer, C. *Organic Letters*. **2021**, *23*, 5491-5495.
- [2] Edwards, A.; Rubin, M. *Tetrahedron*. **2015**, *71*, 3237-3246.
- [3] Gao, X., Ren, X. X., Deng, W., Zhang, X. G. *Chinese Journal of Chemistry*. **2023**, *41*, 3521-3527.
- [4] Hommelsheim, R.; Guo, Y.; Yang, Z.; Empel, C.; Rene, M. K. *Angewandte Chemie International Edition*. **2019**, *58*, 1203-1207.
- [5] Liu, X. Z.; Fox, J. M. *Journal of the American Chemical Society*. **2006**, *128*, 5600-5601.
- [6] Li, C. K.; Zeng, Y.; Wang, J. B. *Tetrahedron Letters*. **2009**, *50*, 2956-2959.
- [7] Guo, L.; Chatupheeraphat, A.; Rueping, M. *Angewandte Chemie International Edition*. **2016**, *128*, 11989-11992.
- [8] Suginome, M.; Matsuda, T.; Ito, Y. *Organometallics*. **2000**, *19*, 4647-4649.

8.NMR Spectra

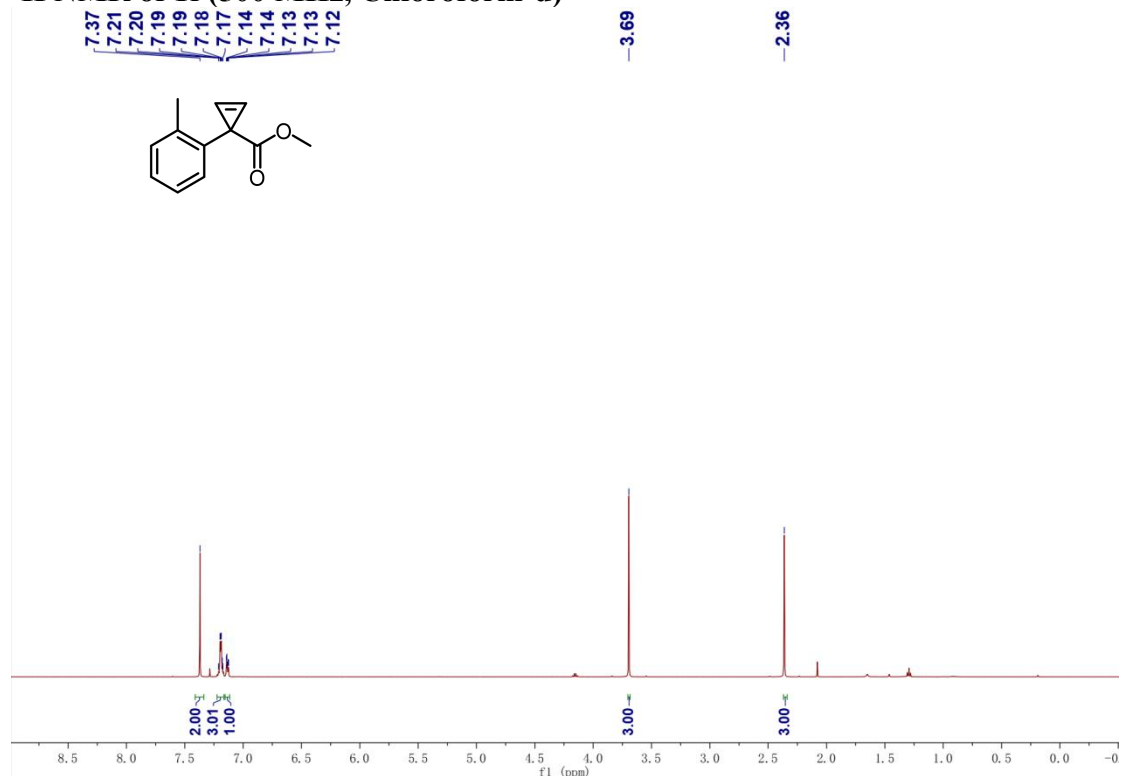
¹H NMR of 1e (500 MHz, Chloroform-d)



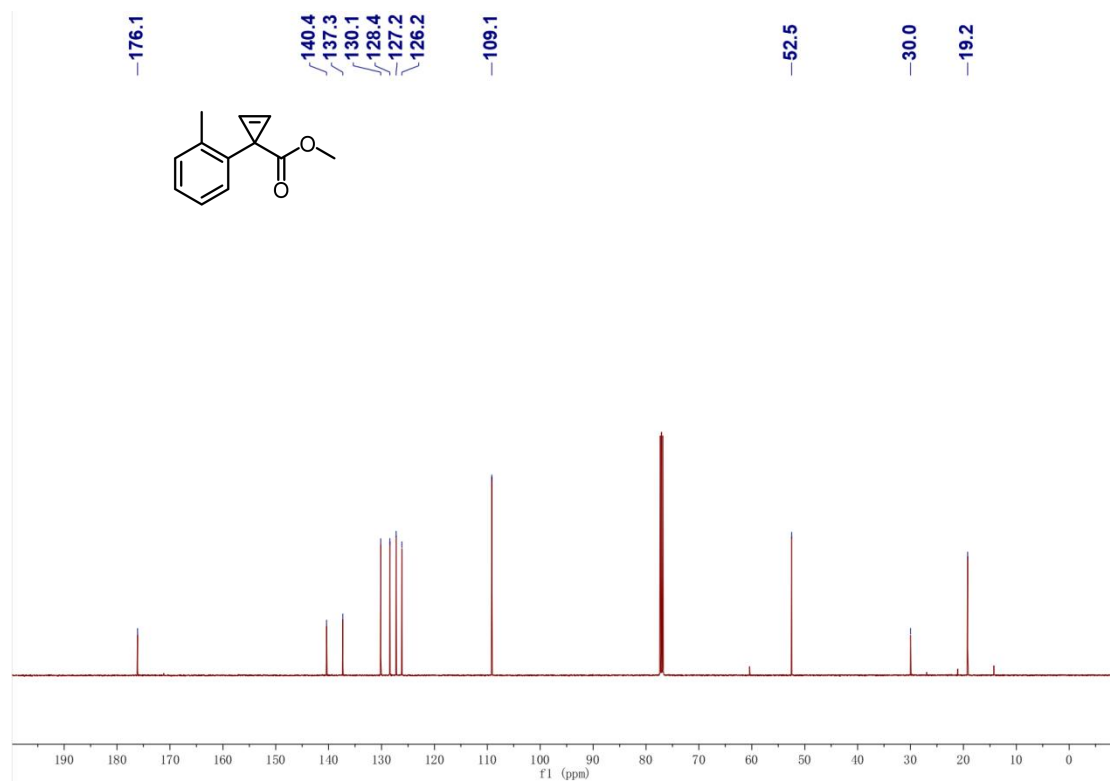
¹³C NMR of 1e (101 MHz, Chloroform-d)



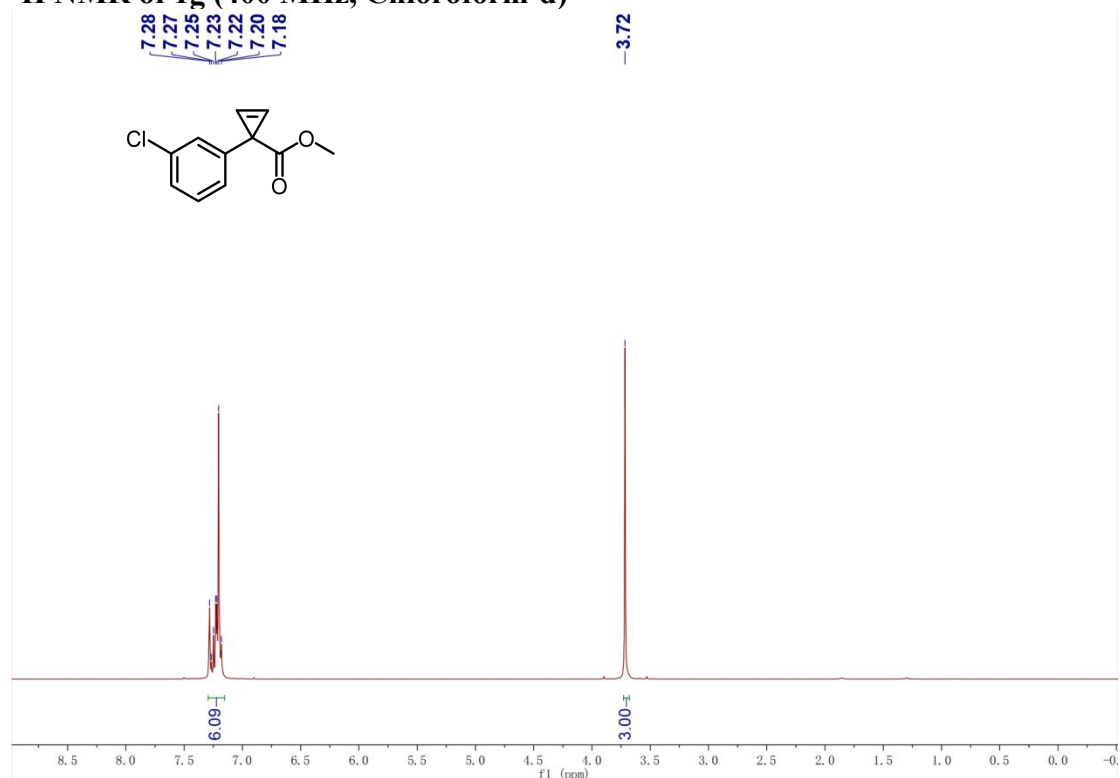
¹H NMR of 1f (500 MHz, Chloroform-d)



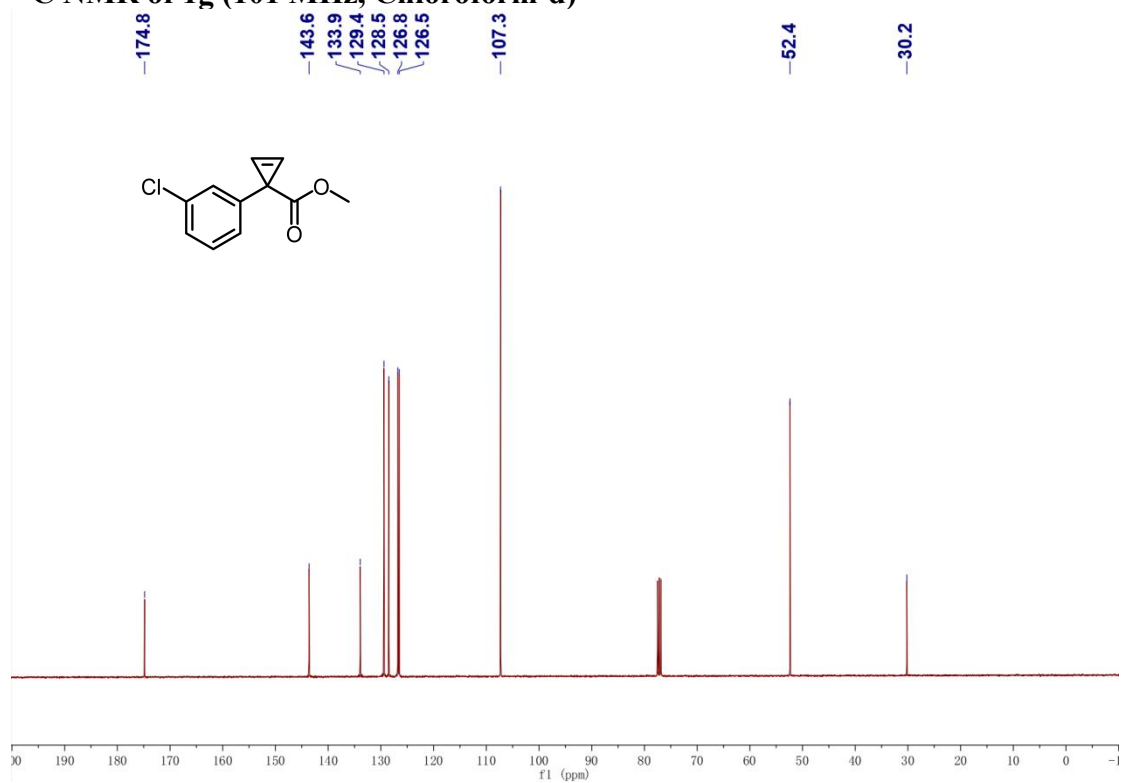
¹³C NMR of 1f (126 MHz, Chloroform-d)



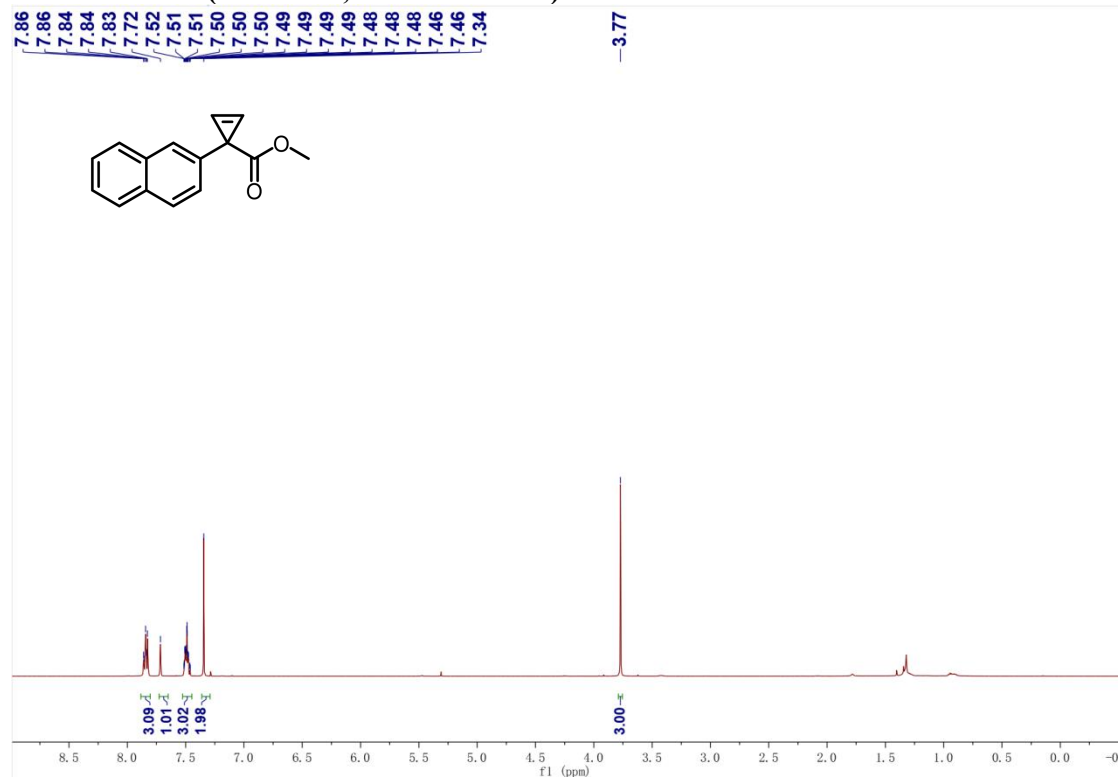
¹H NMR of 1g (400 MHz, Chloroform-d)



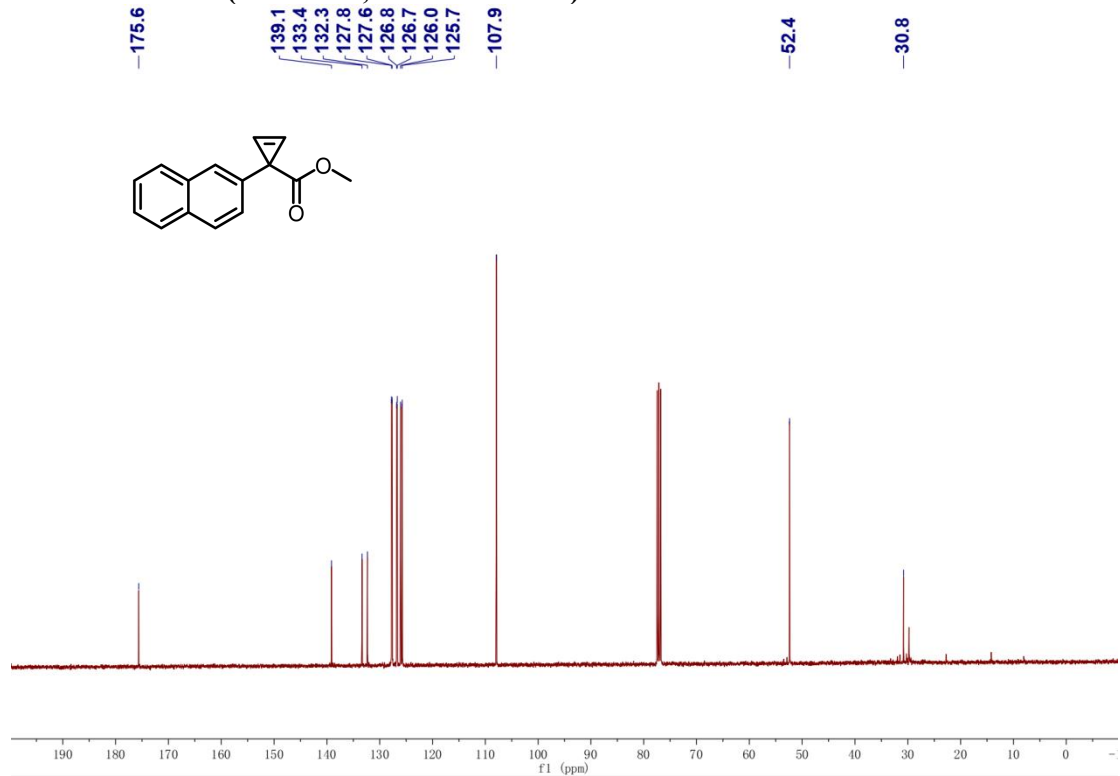
¹³C NMR of 1g (101 MHz, Chloroform-d)



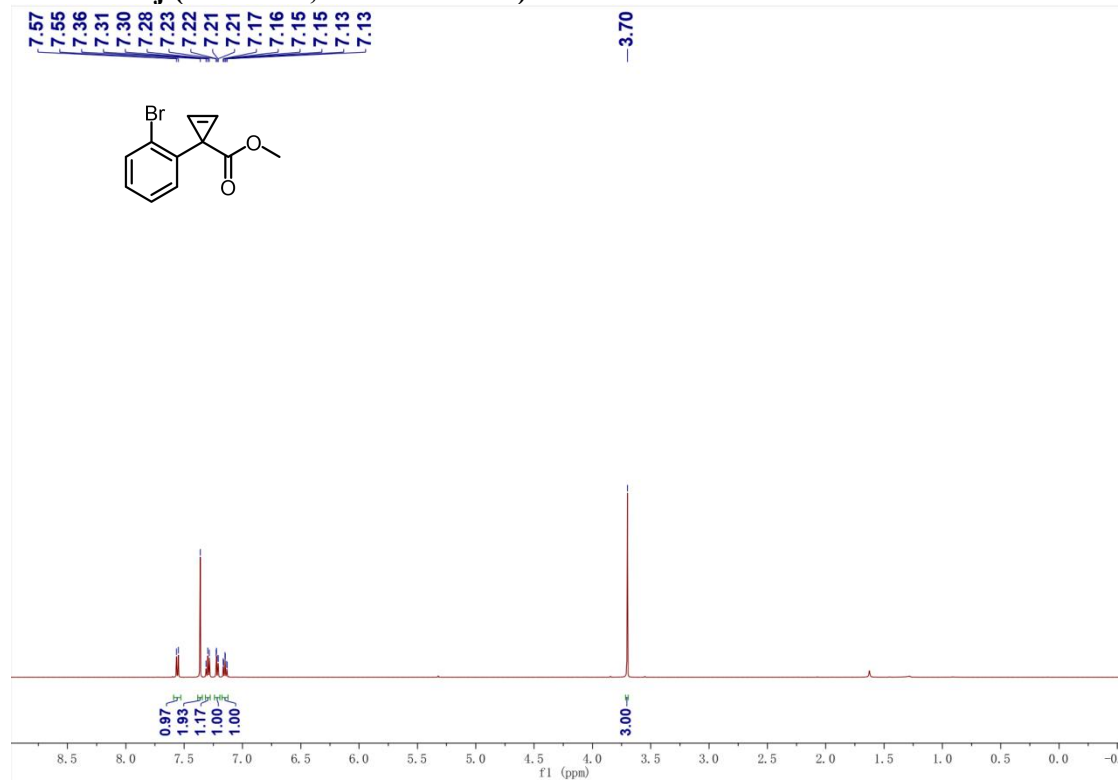
¹H NMR of 1i (500 MHz, Chloroform-d)



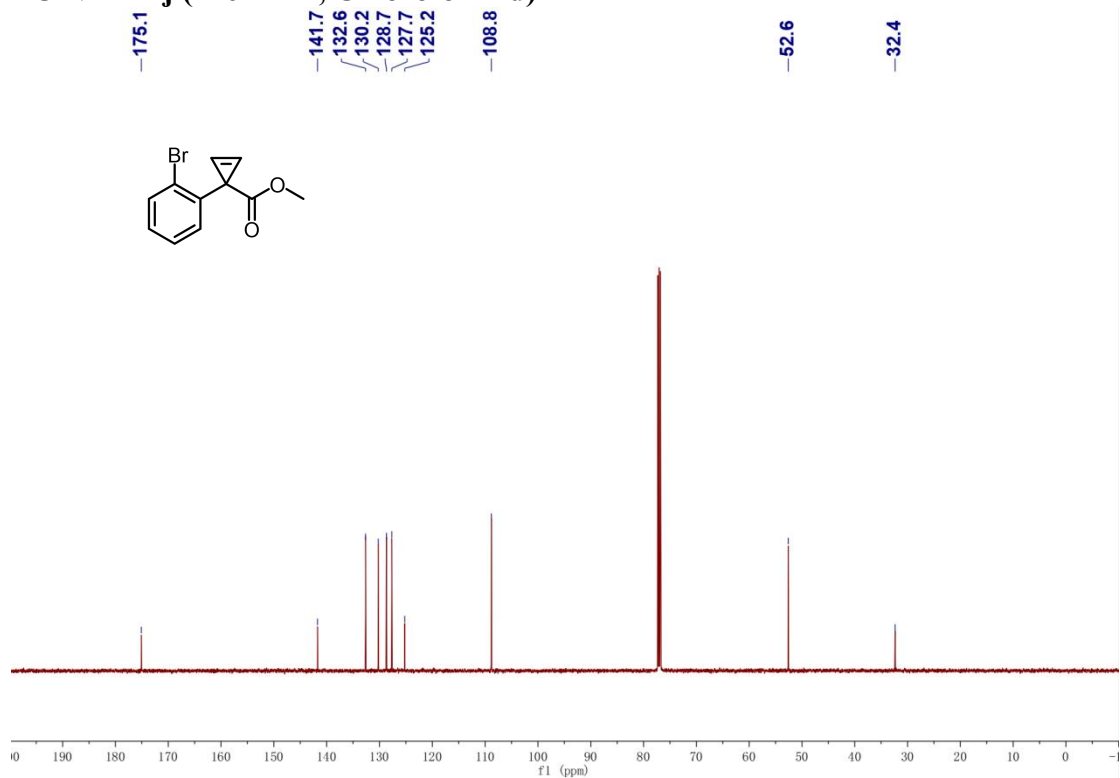
¹³C NMR of 1i (101 MHz, Chloroform-d)



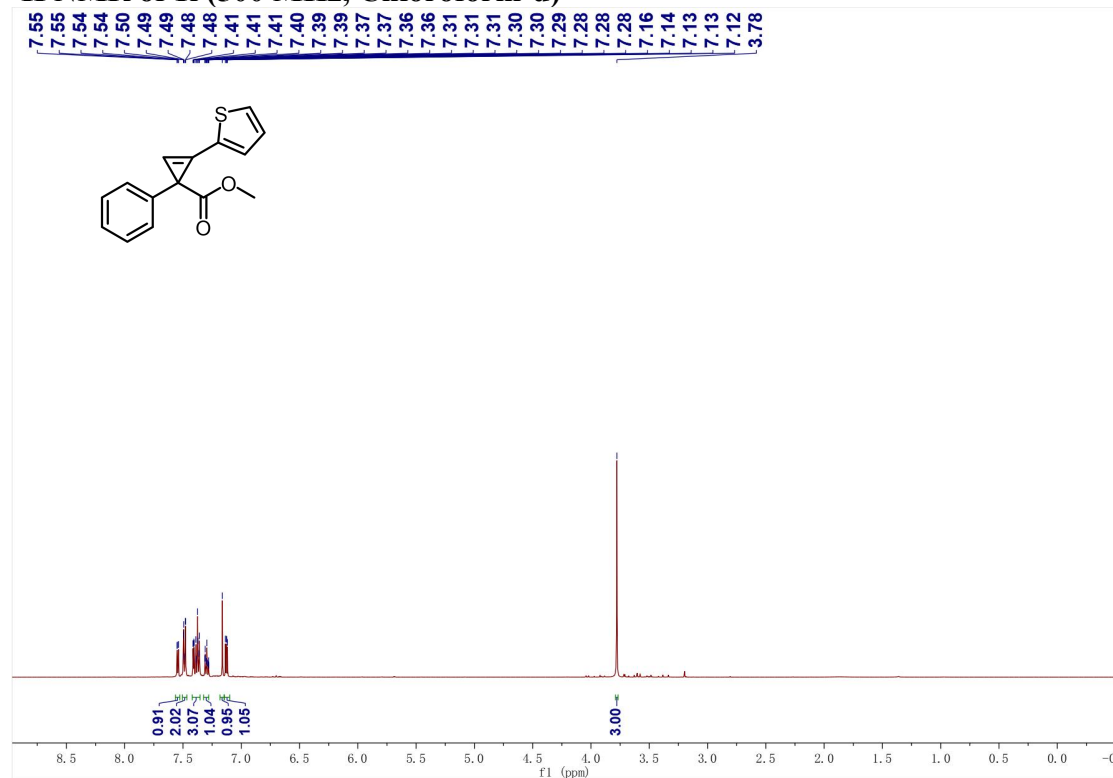
¹H NMR 1j (500 MHz, Chloroform-d)



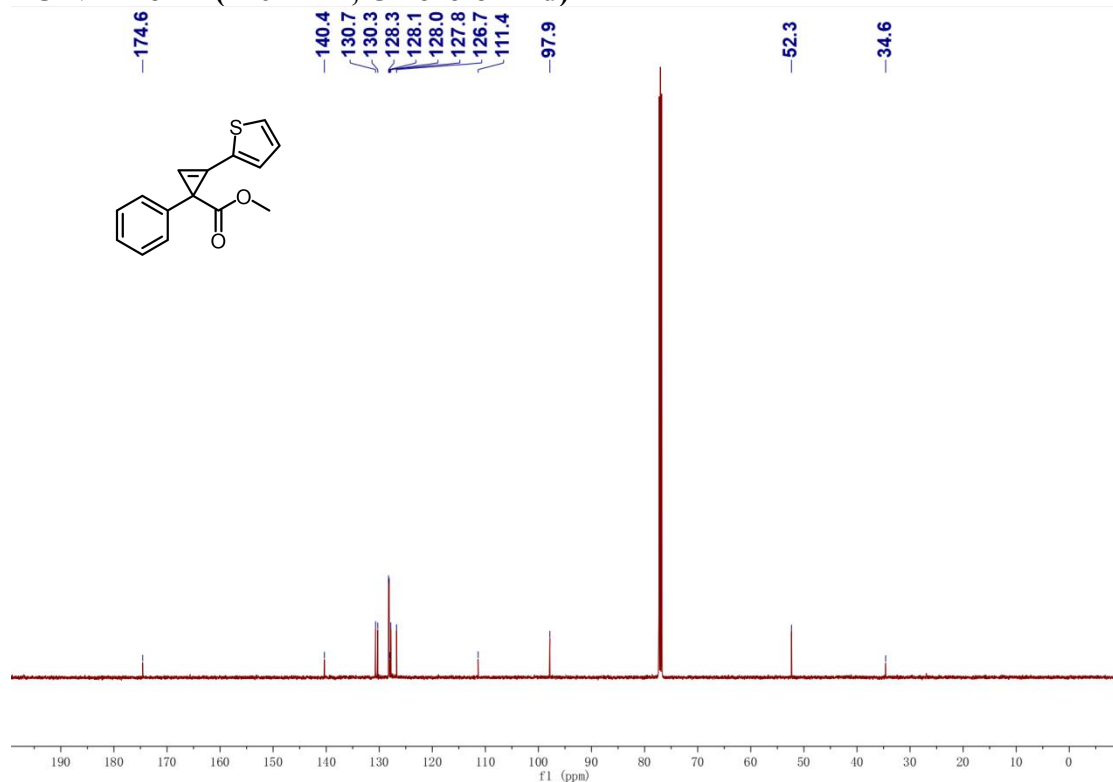
¹³C NMR 1j (126 MHz, Chloroform-d)



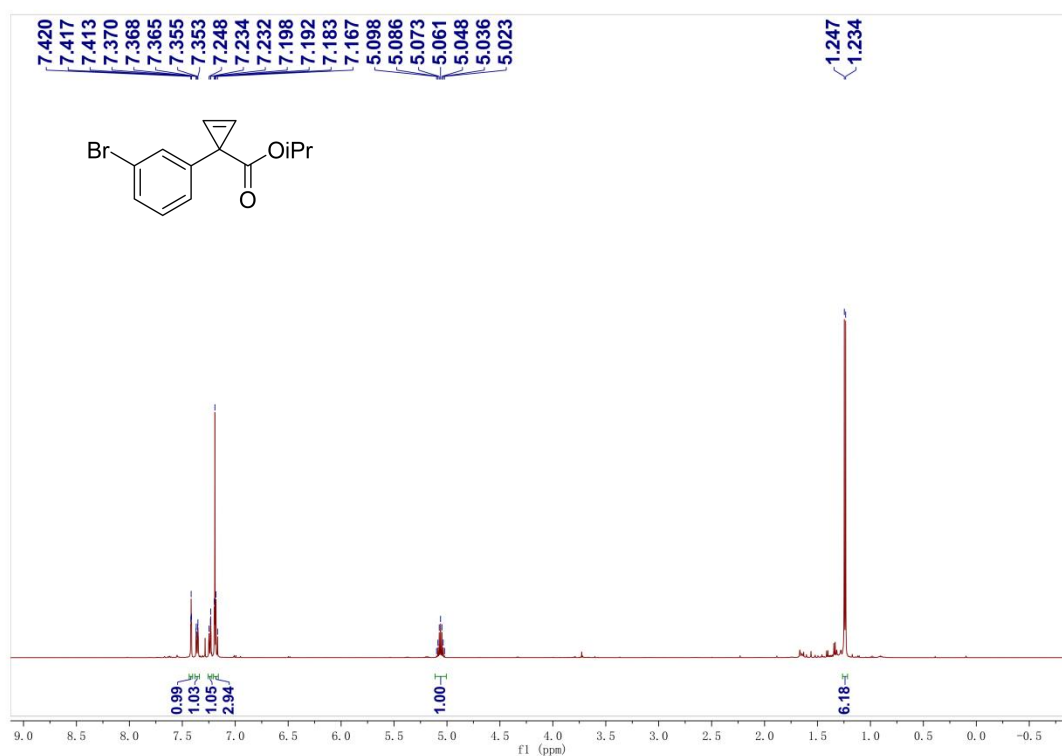
¹H NMR of 11 (500 MHz, Chloroform-d)



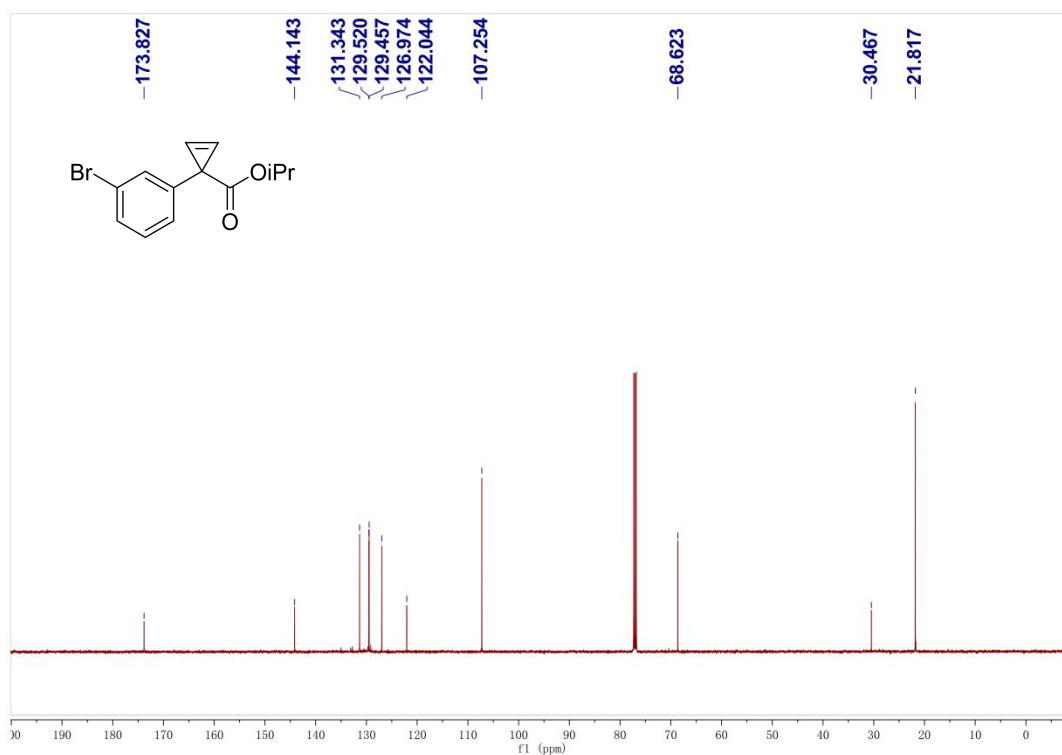
¹³C NMR of 11 (126 MHz, Chloroform-d)



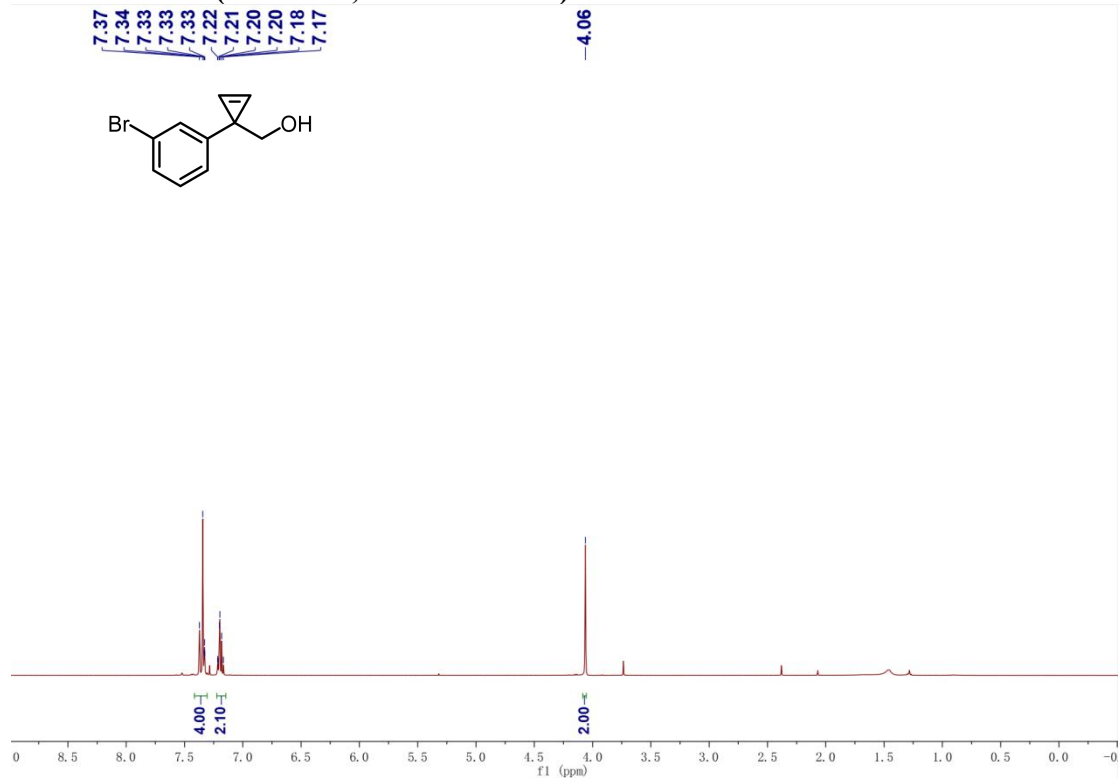
¹H NMR of 1n (500 MHz, Chloroform-d)



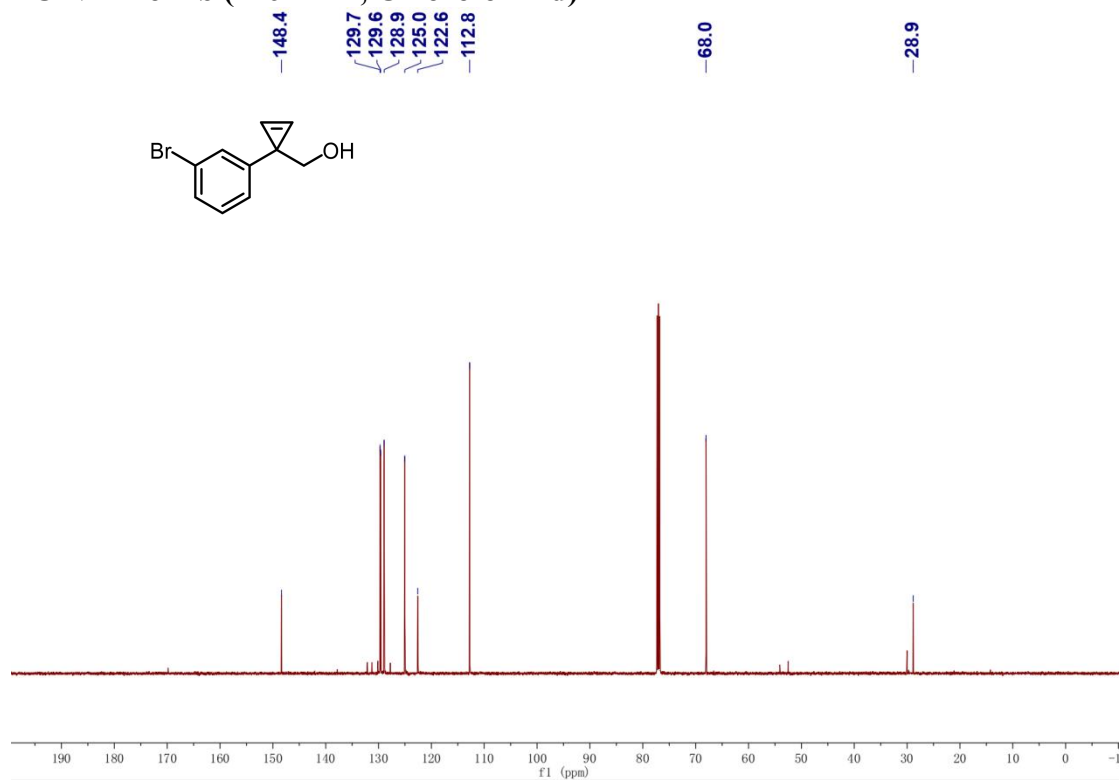
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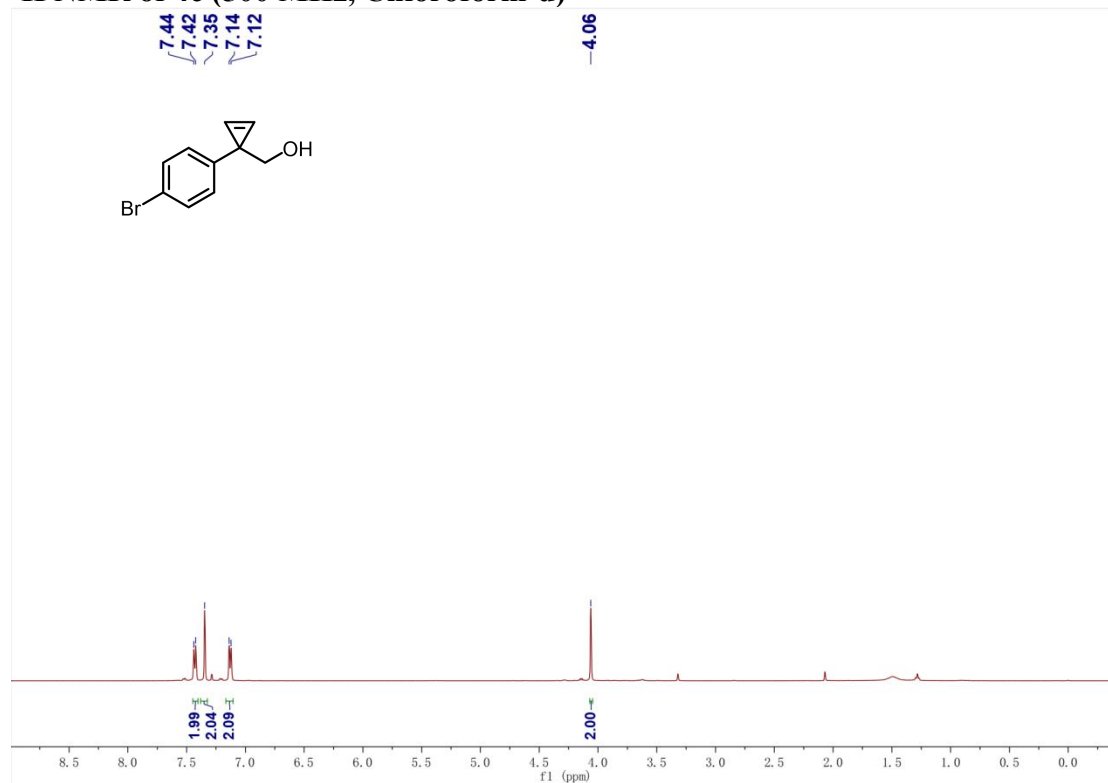
¹H NMR of 4b (500 MHz, Chloroform-d)



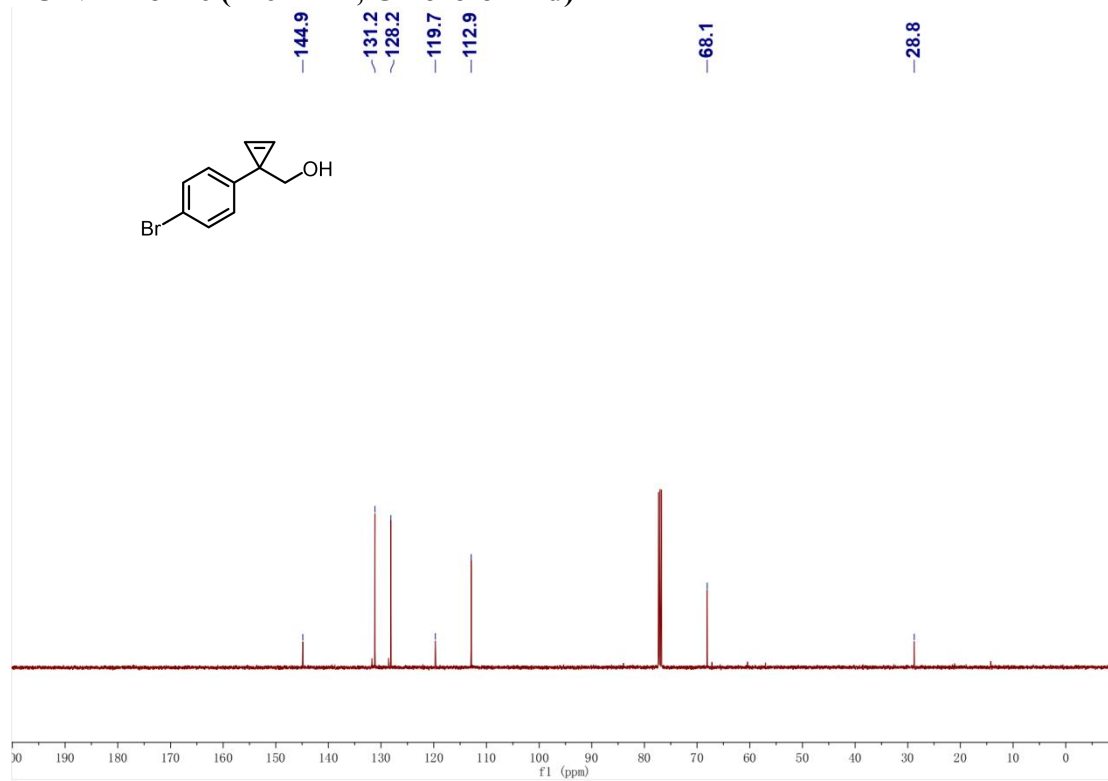
¹³C NMR of 4b (126 MHz, Chloroform-d)



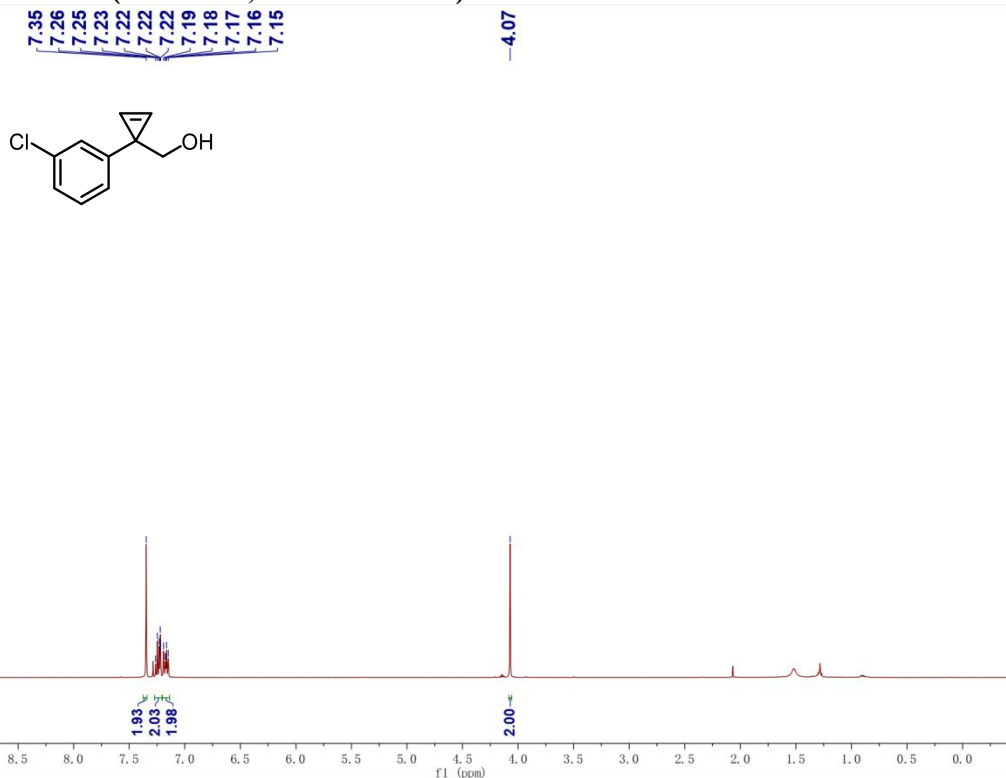
¹H NMR of 4c (500 MHz, Chloroform-d)



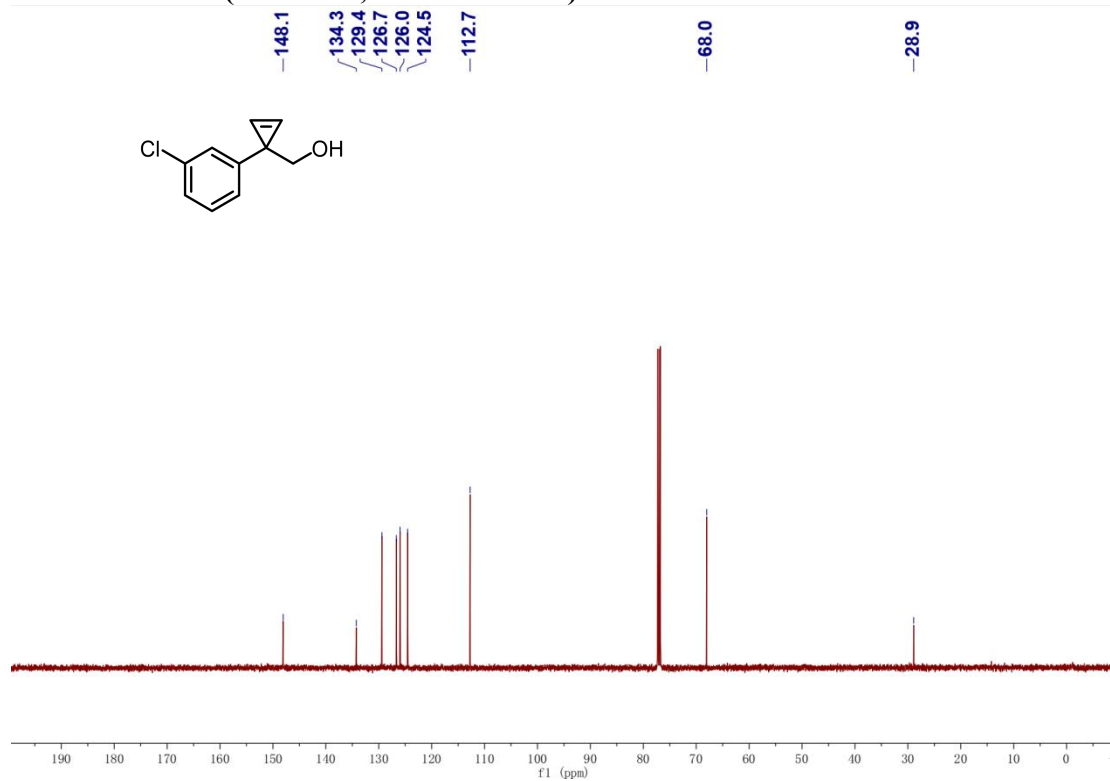
¹³C NMR of 4c (126 MHz, Chloroform-d)



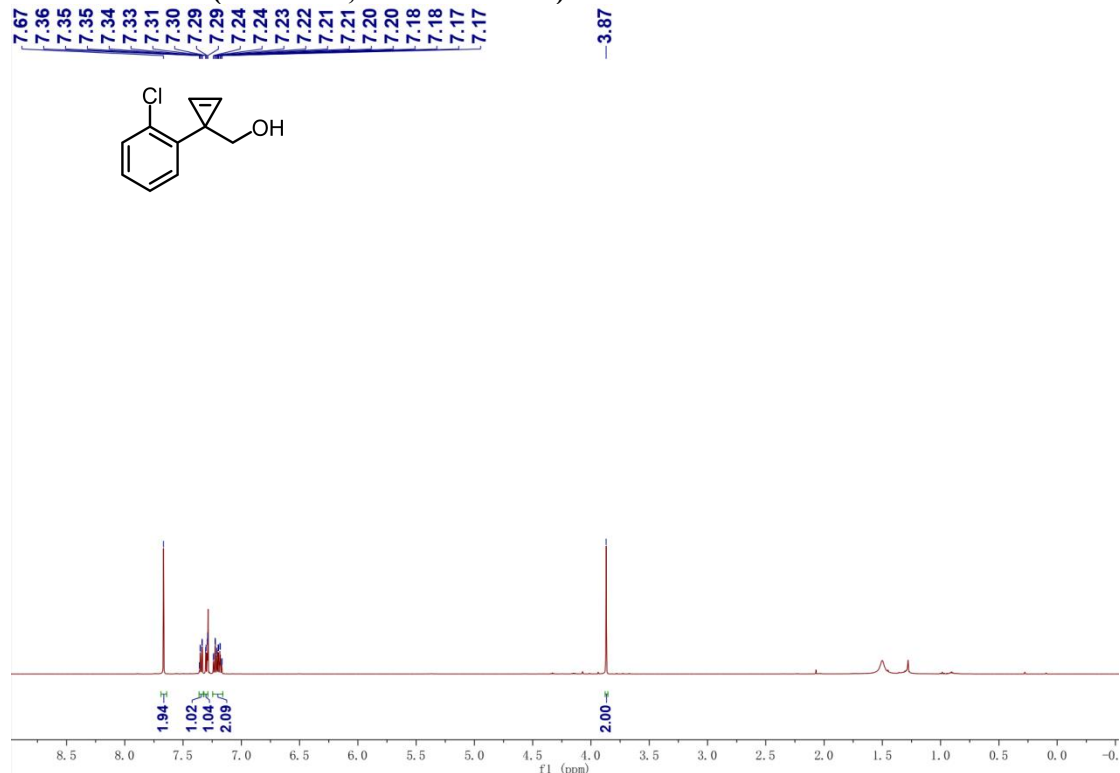
¹H NMR of 4d (500 MHz, Chloroform-d)



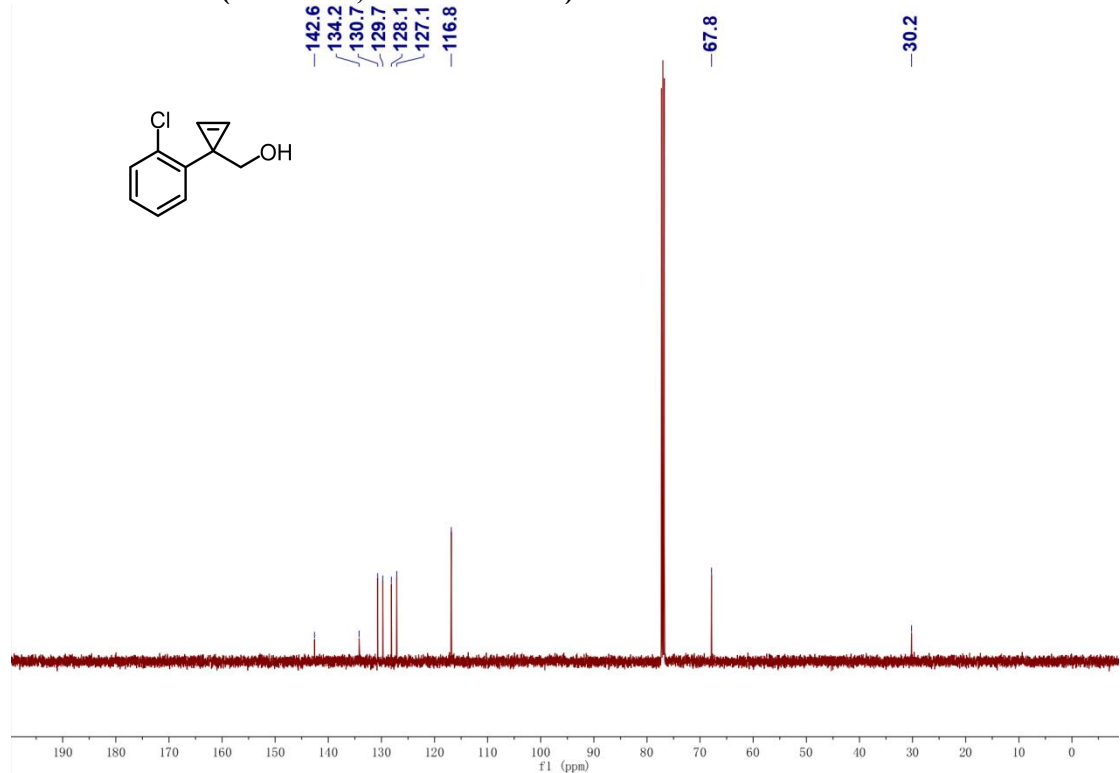
¹³C NMR of 4d (126 MHz, Chloroform-d)



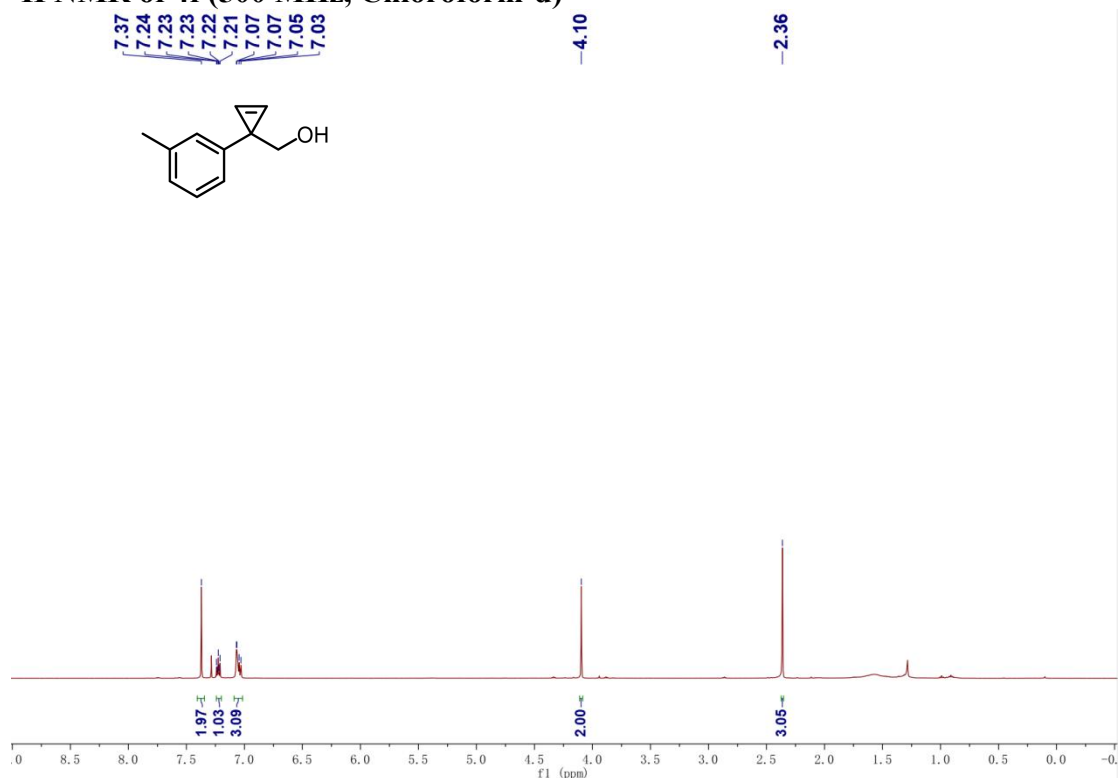
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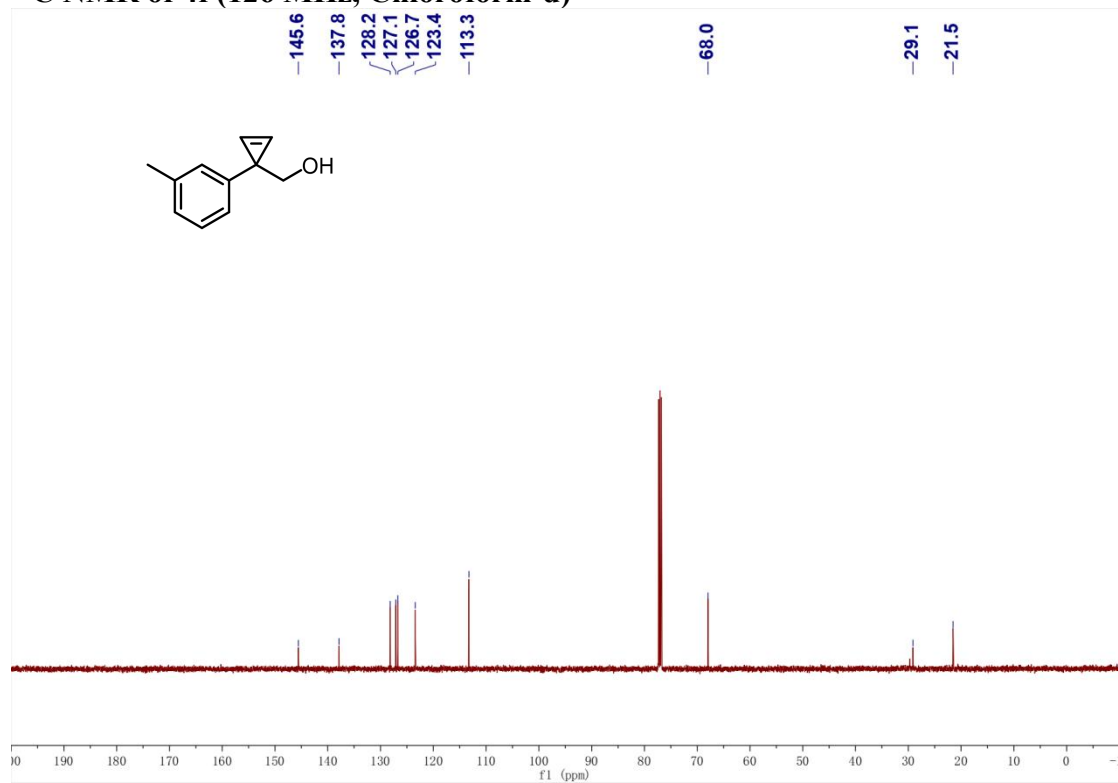
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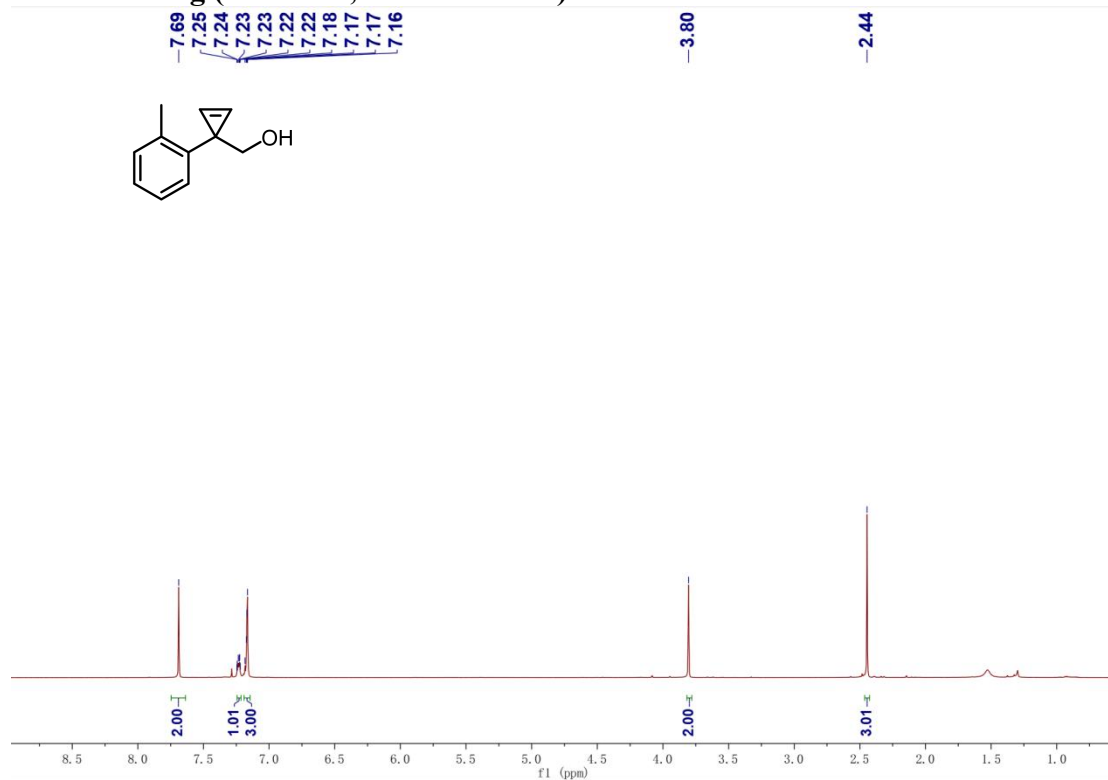
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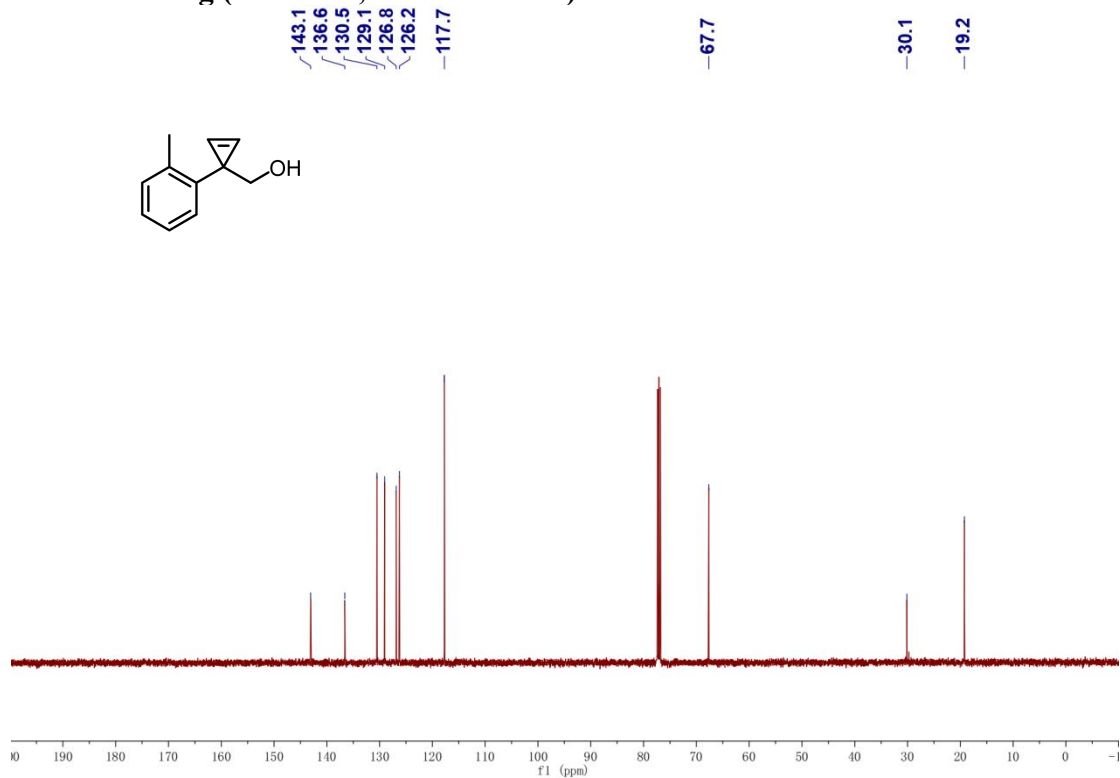
¹³C NMR of 4f (126 MHz, Chloroform-d)



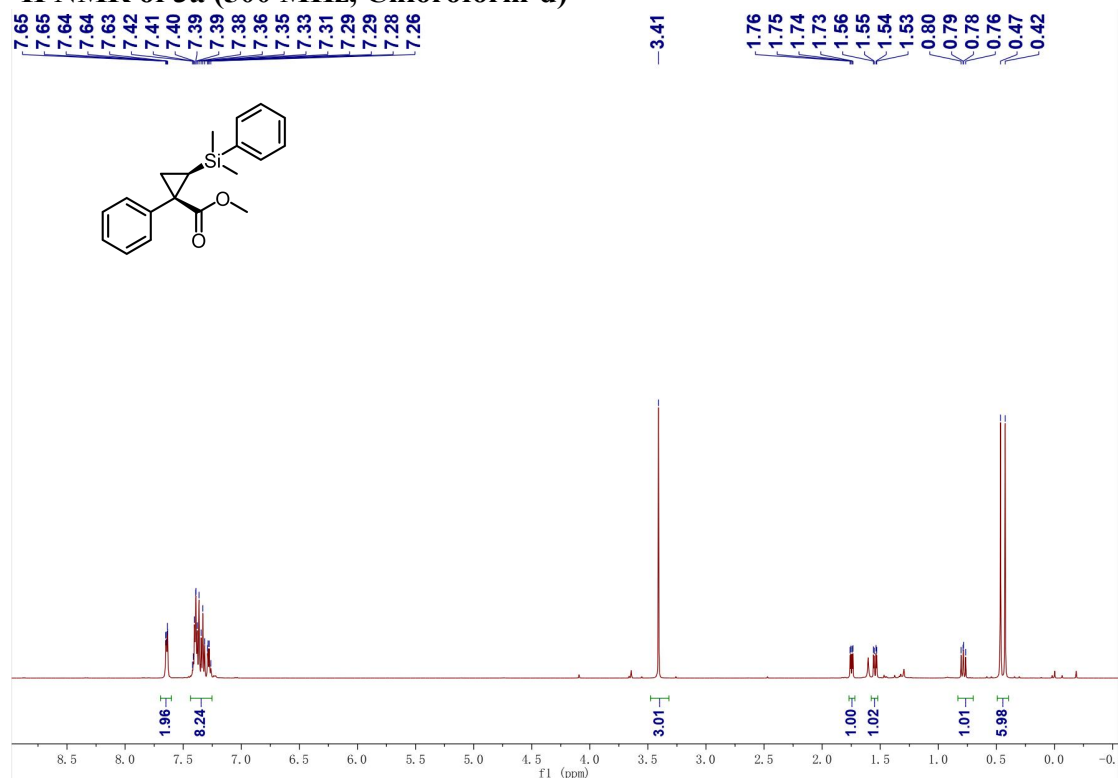
¹H NMR of 4g (500 MHz, Chloroform-d)



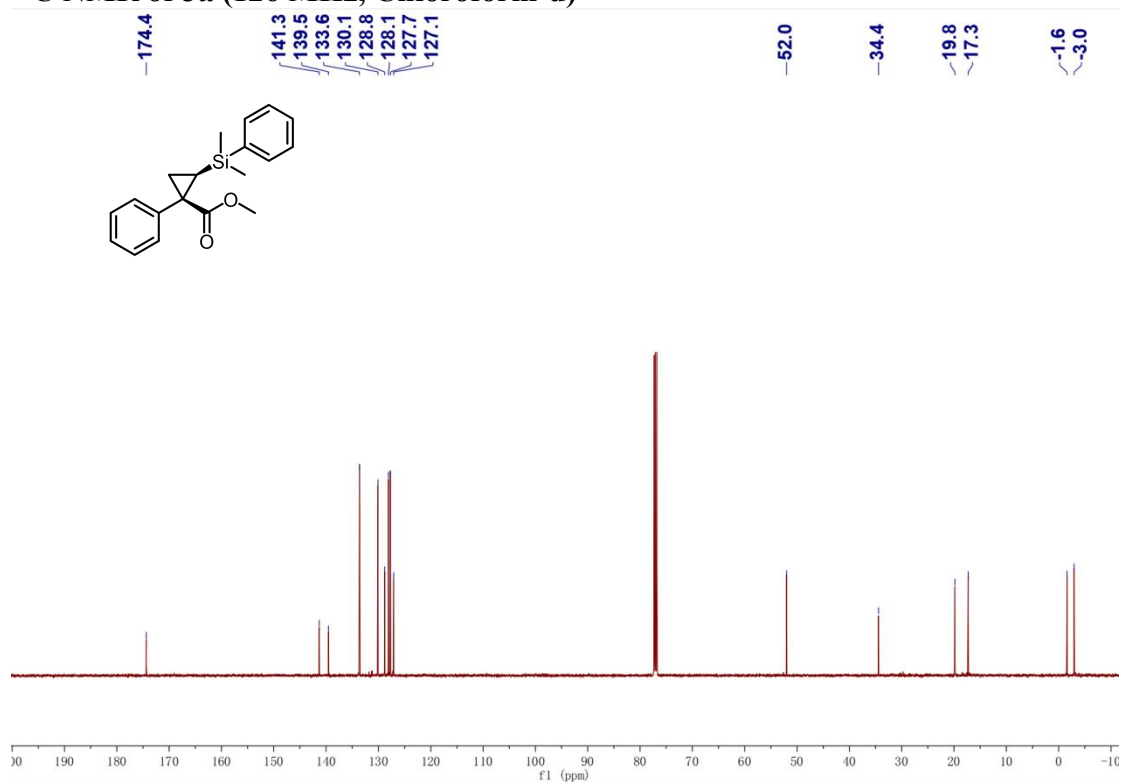
¹³C NMR of 4g (126 MHz, Chloroform-d)



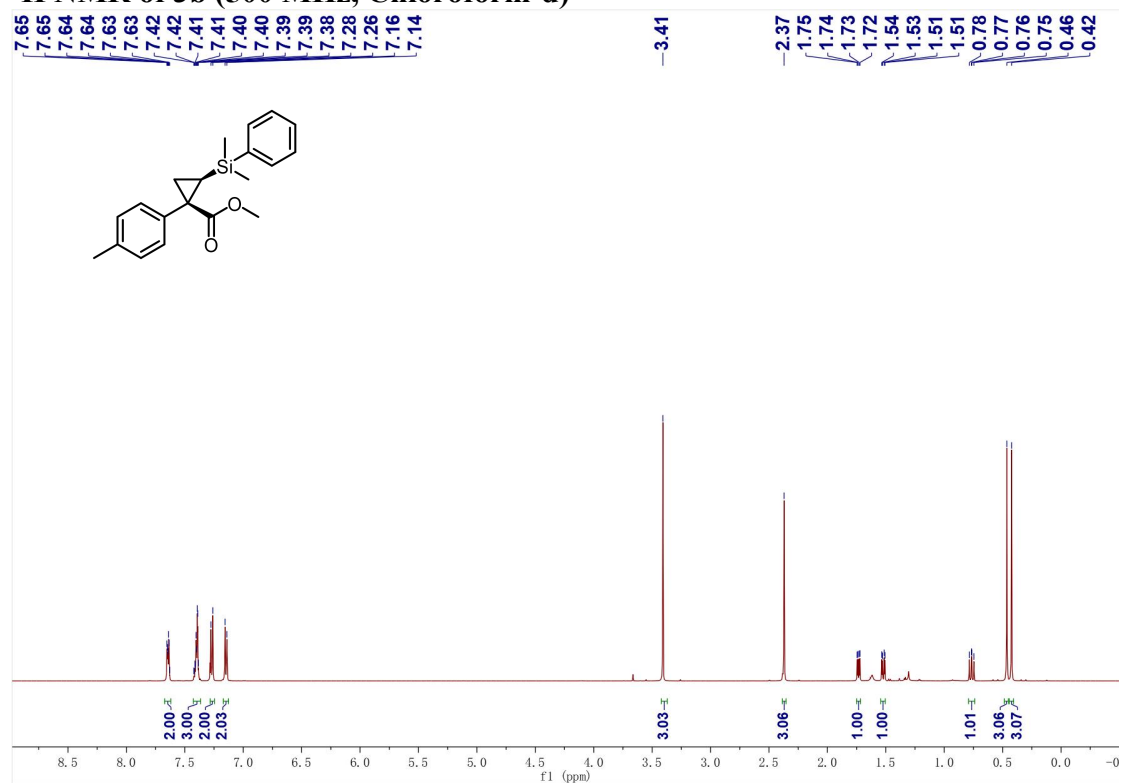
¹H NMR of 3a (500 MHz, Chloroform-d)



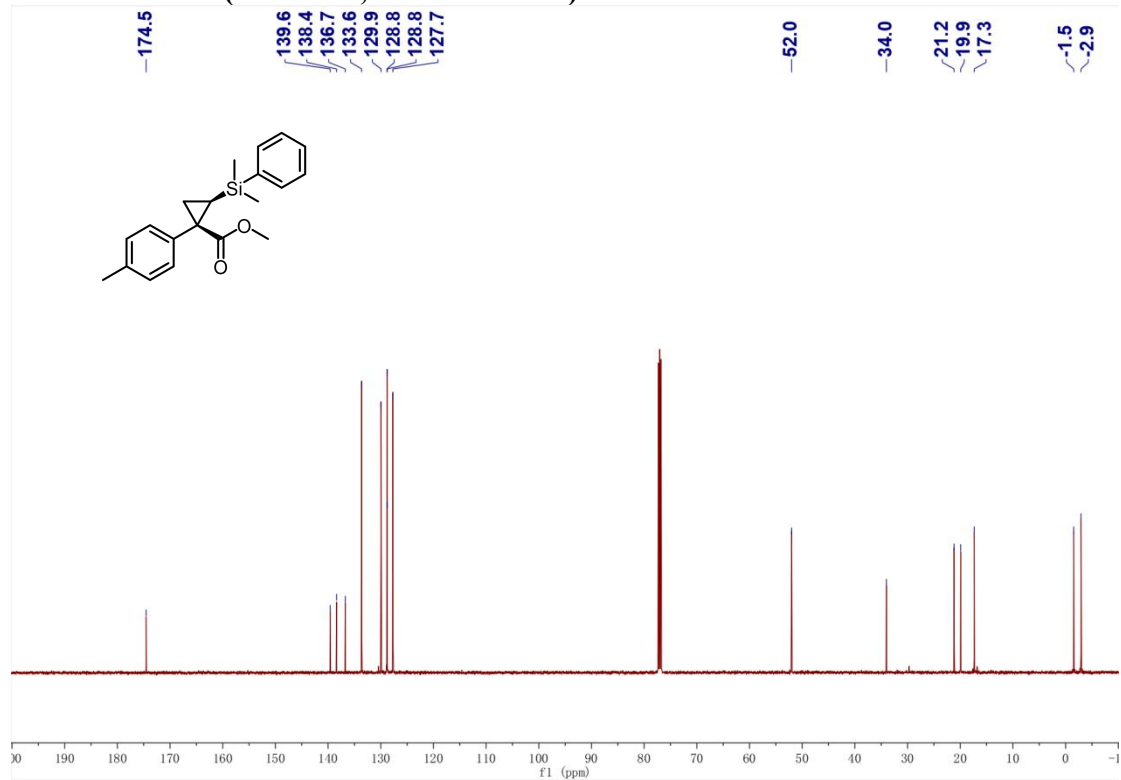
¹³C NMR of 3a (126 MHz, Chloroform-d)



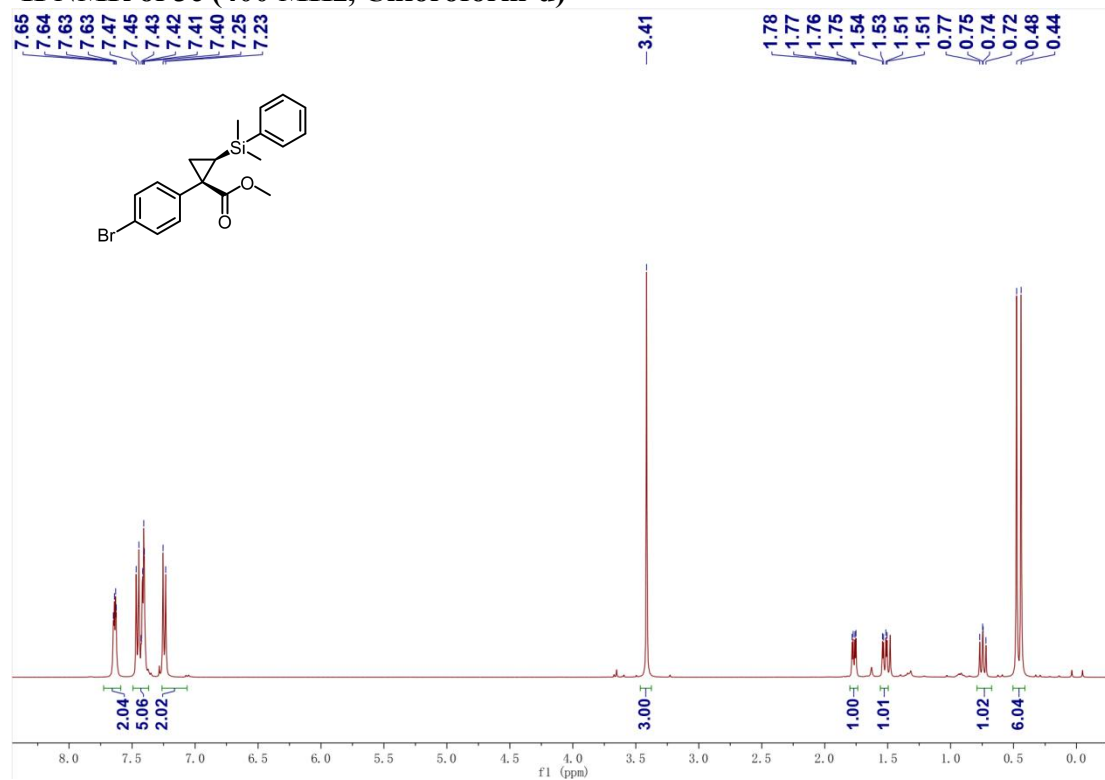
¹H NMR of 3b (500 MHz, Chloroform-d)



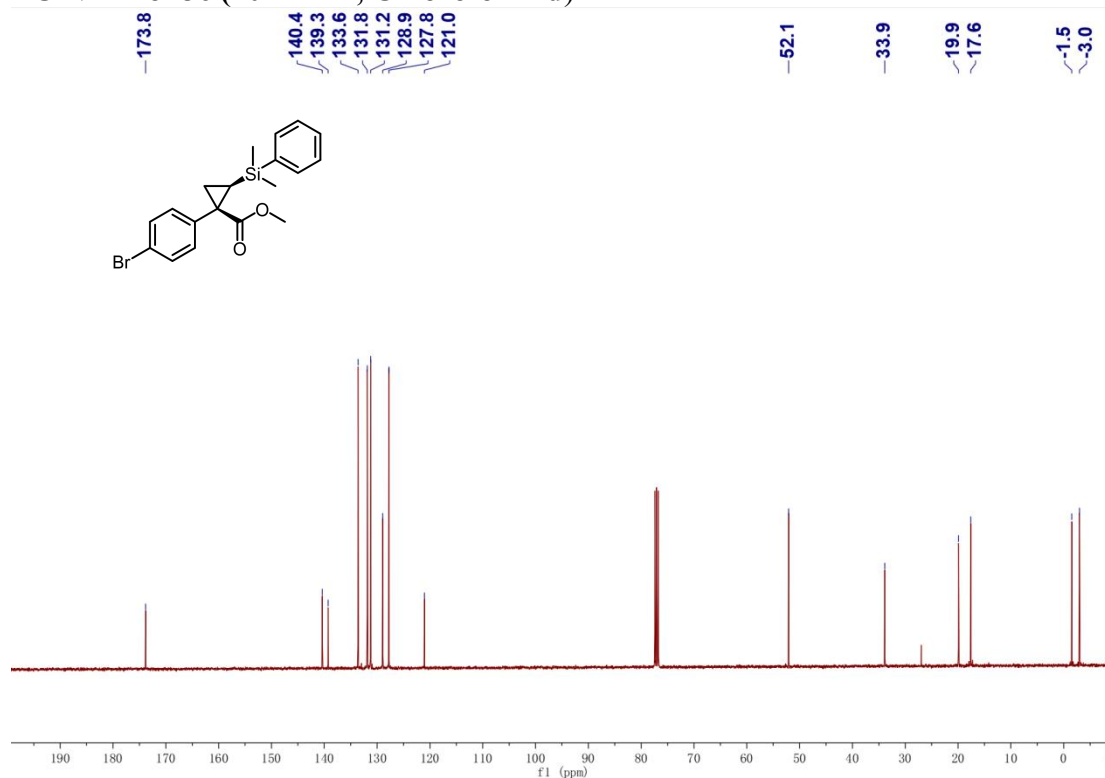
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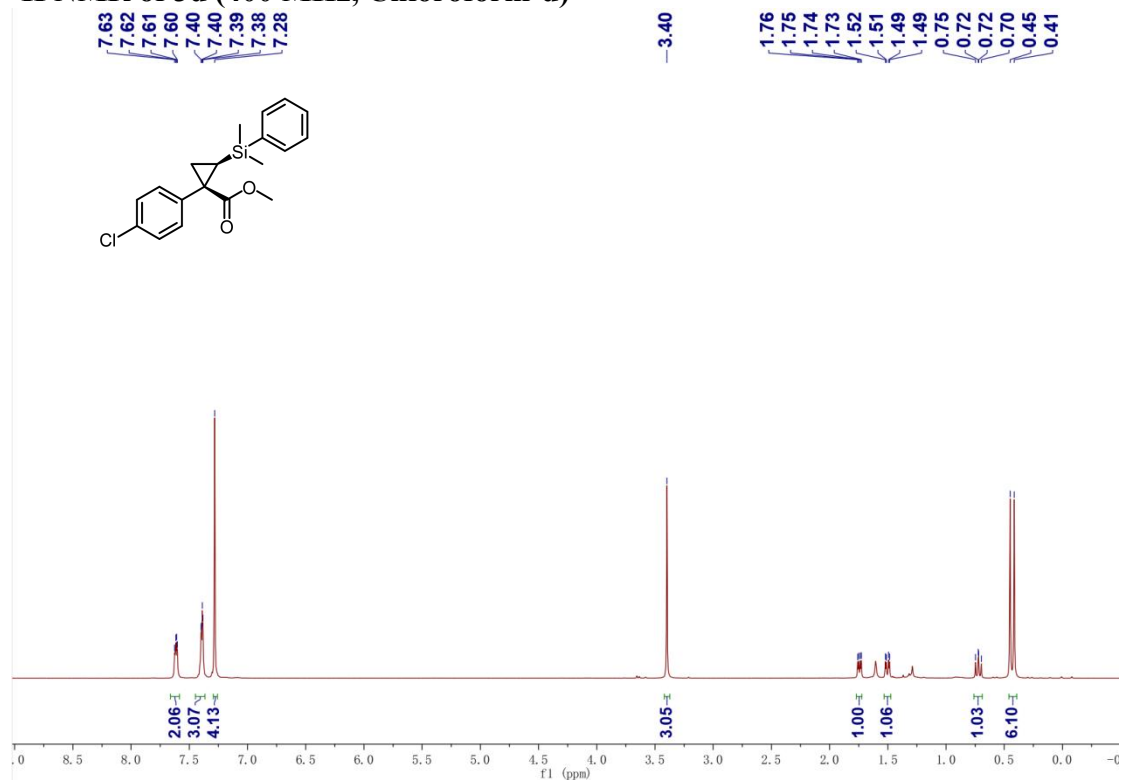
¹H NMR of 3c (400 MHz, Chloroform-d)



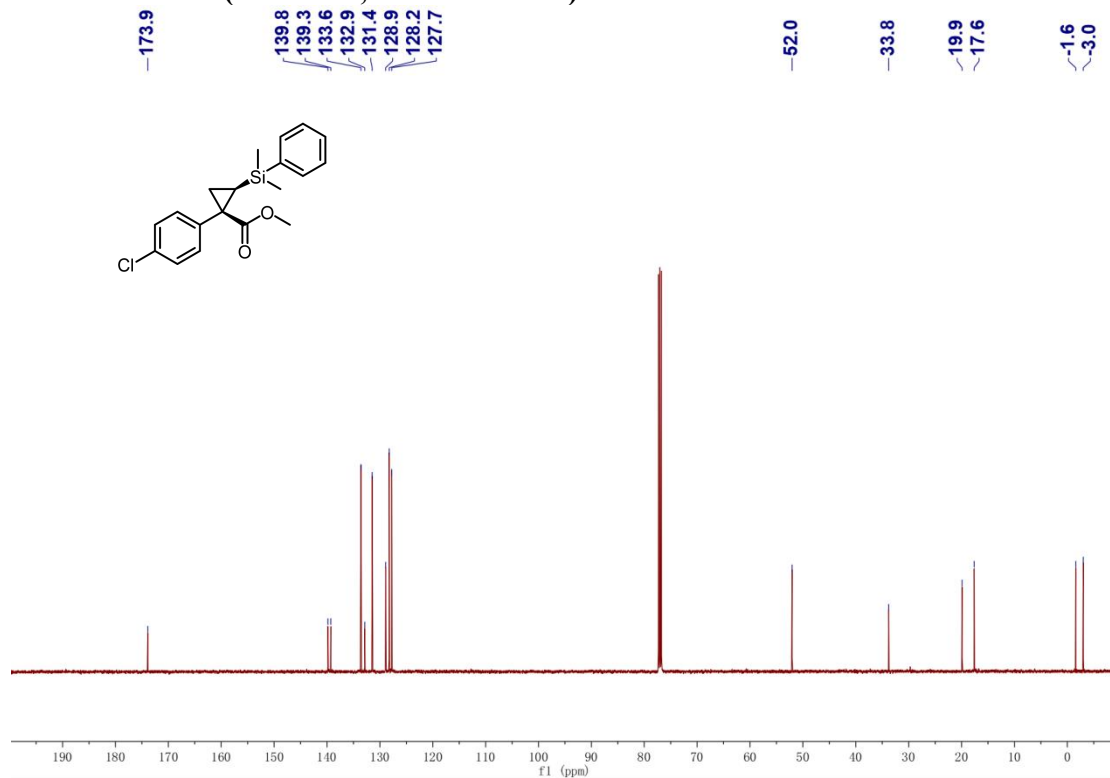
¹³C NMR of 3c (101 MHz, Chloroform-d)



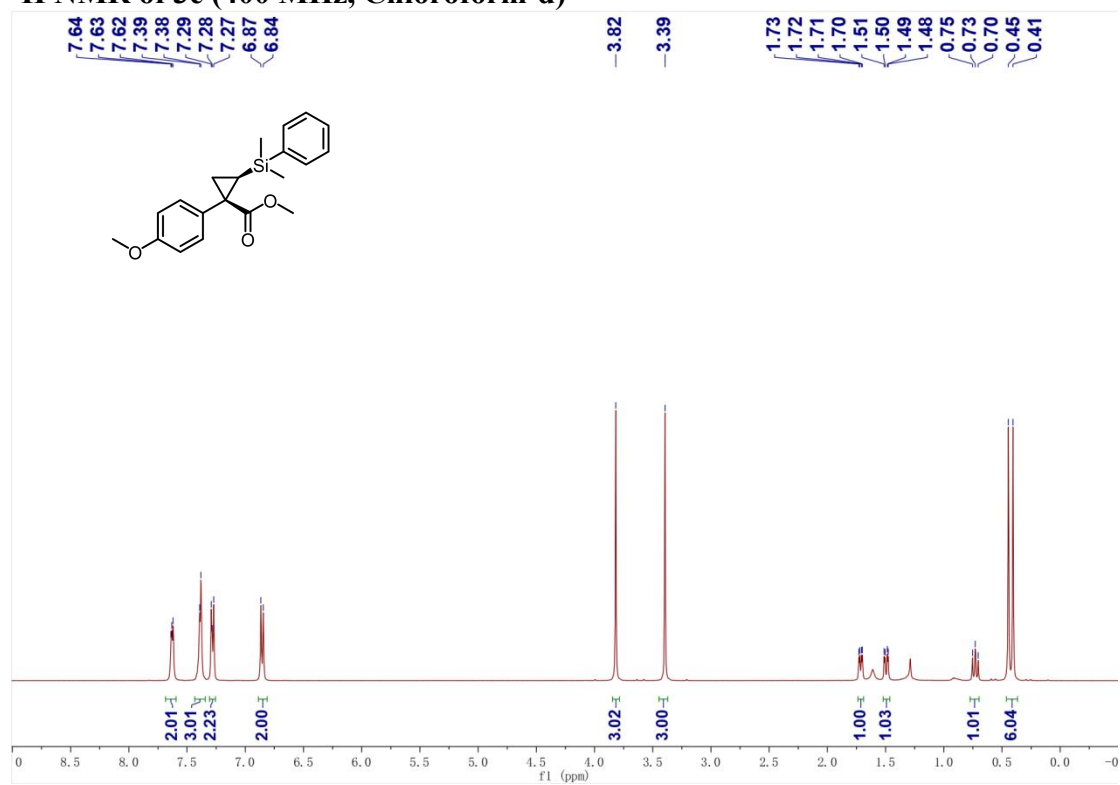
¹H NMR of 3d (400 MHz, Chloroform-d)



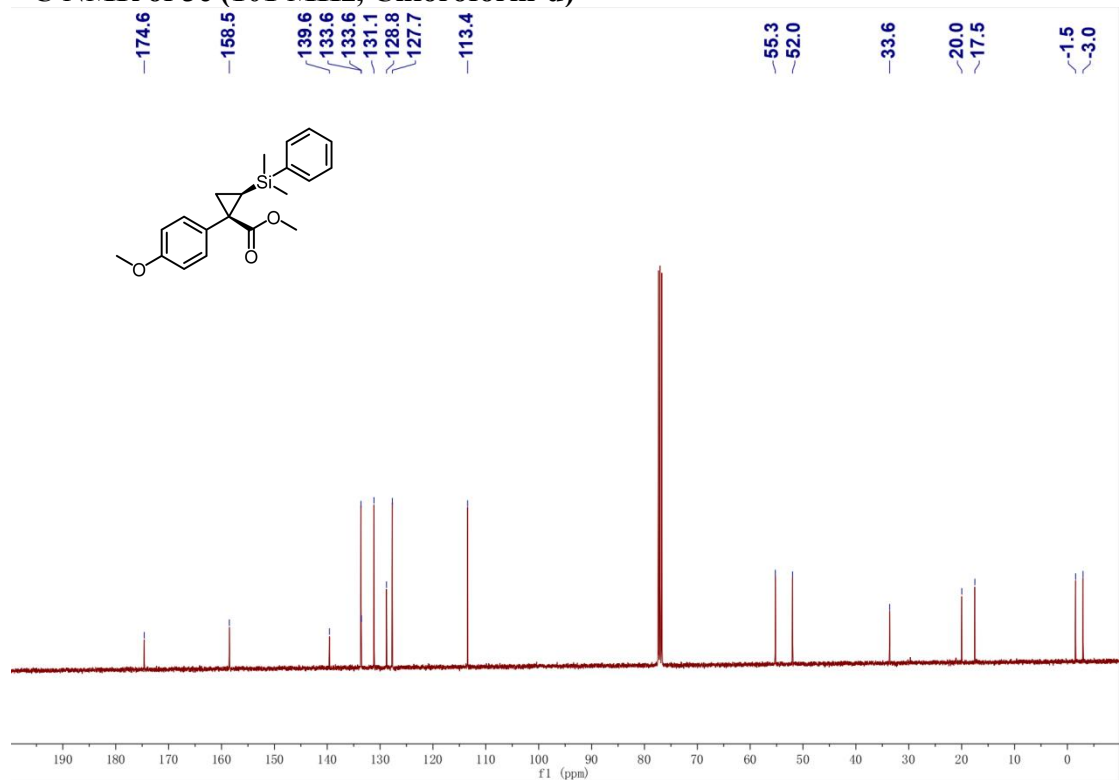
¹³C NMR of 3d (126 MHz, Chloroform-d)



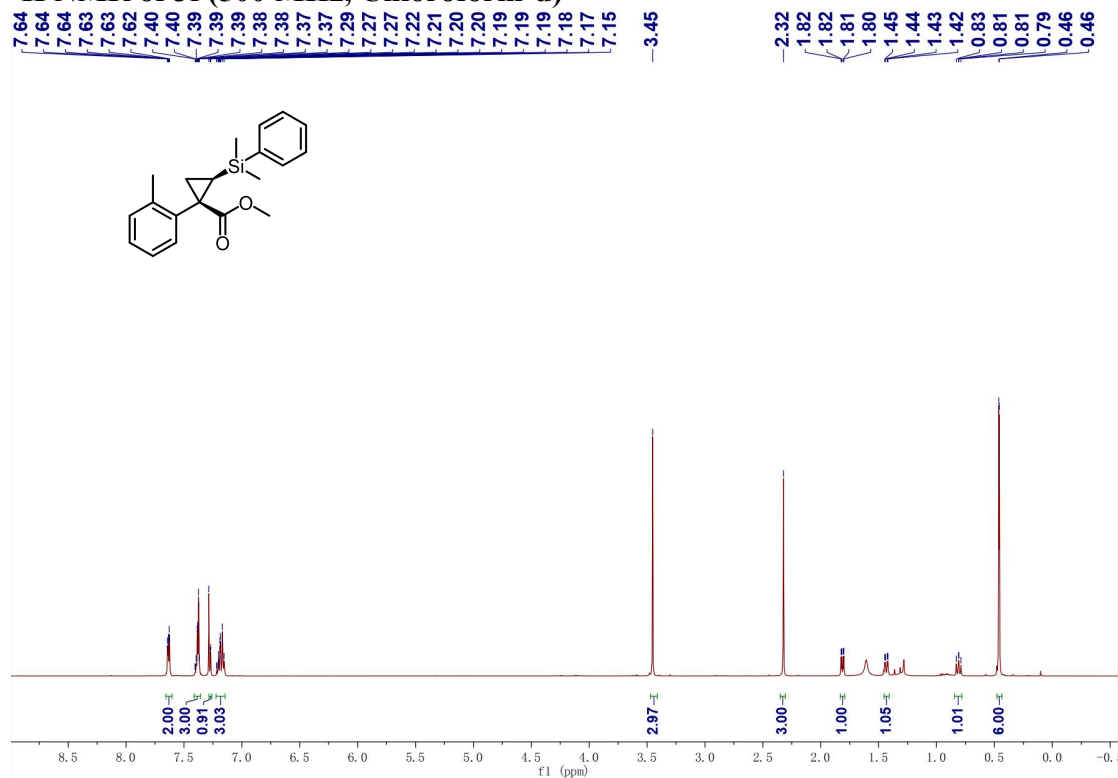
¹H NMR of 3e (400 MHz, Chloroform-d)



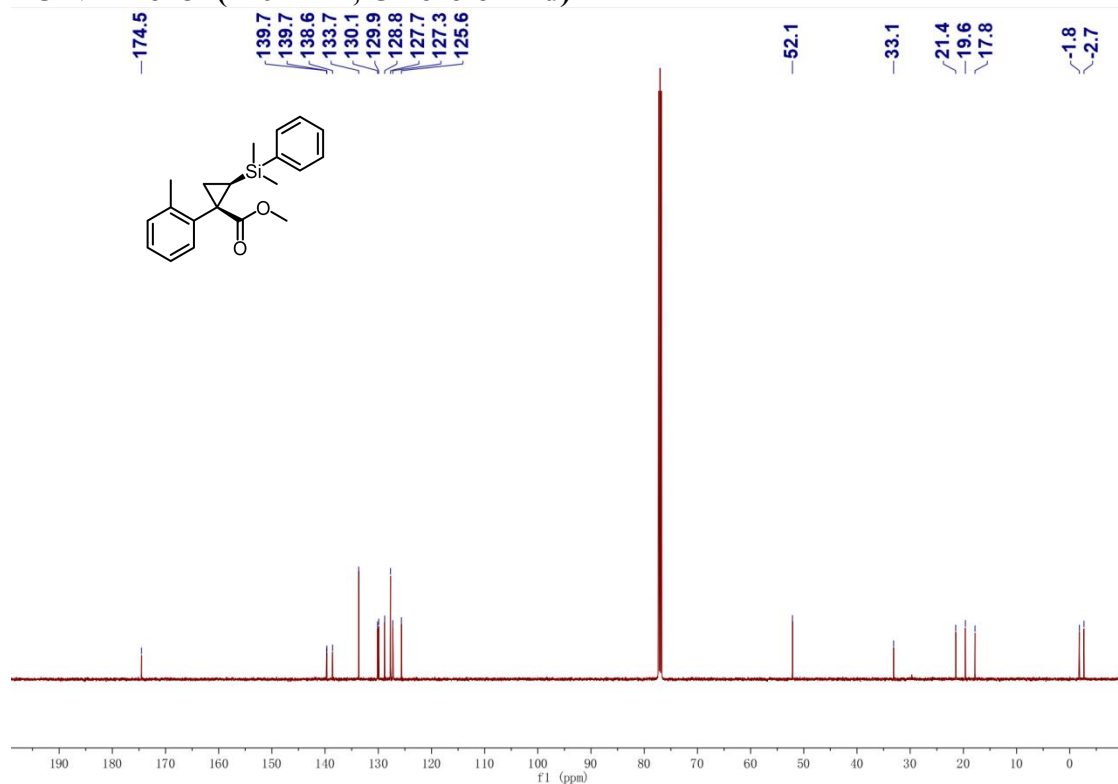
¹³C NMR of 3e (101 MHz, Chloroform-d)



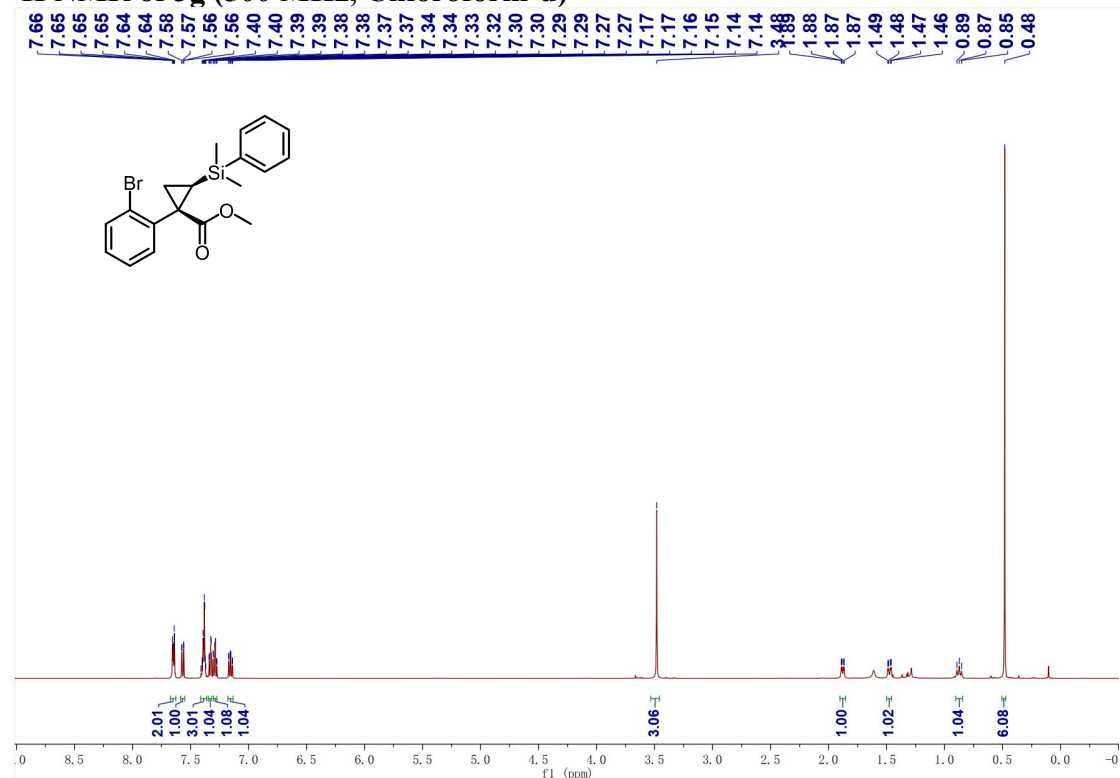
¹H NMR of 3f (500 MHz, Chloroform-d)



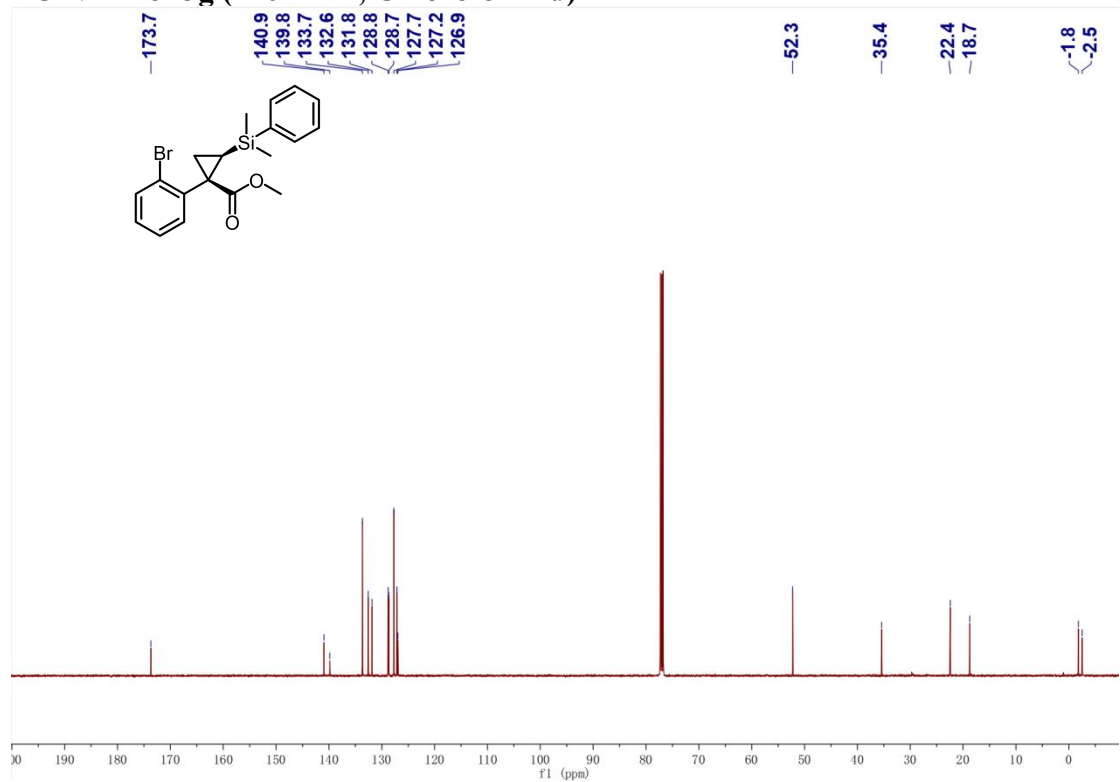
¹³C NMR of 3f (126 MHz, Chloroform-d)



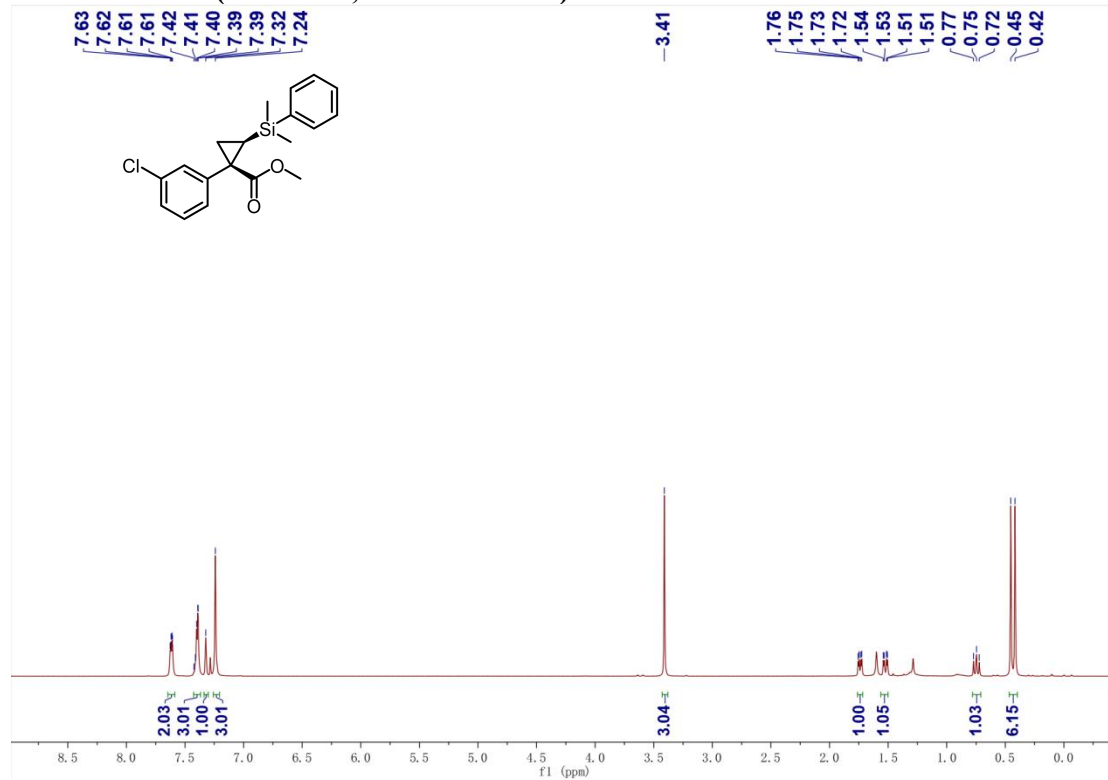
¹H NMR of 3g (500 MHz, Chloroform-d)



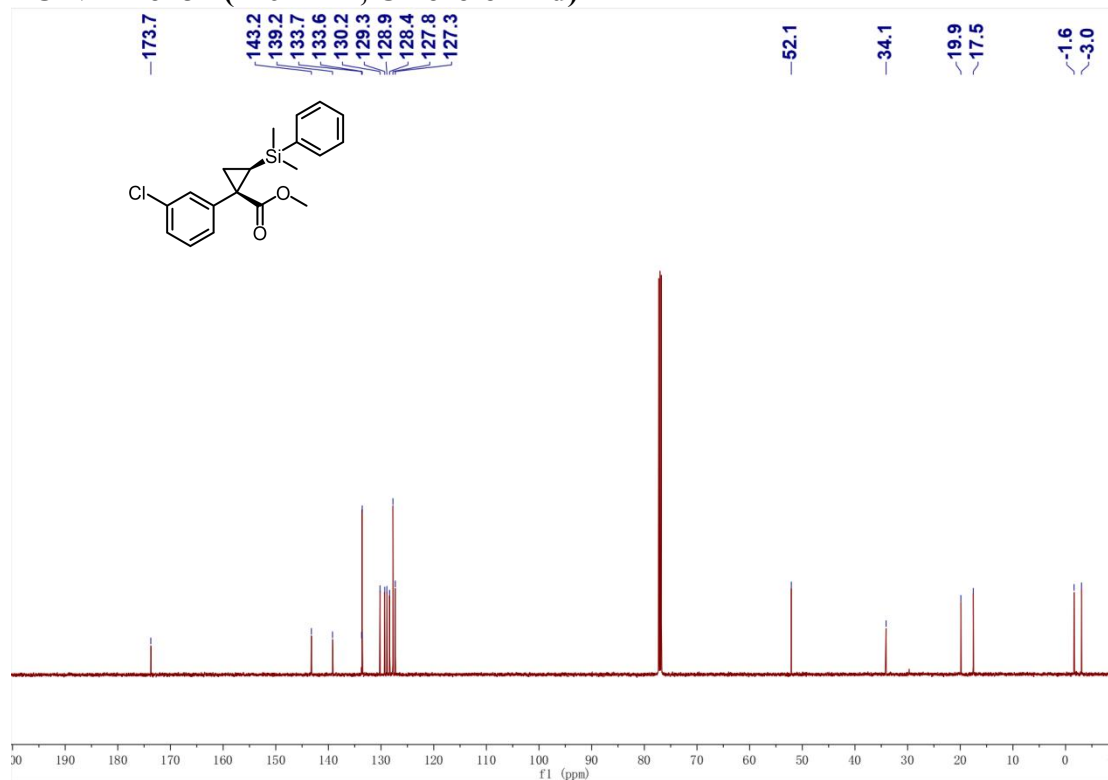
¹³C NMR of 3g (126 MHz, Chloroform-d)



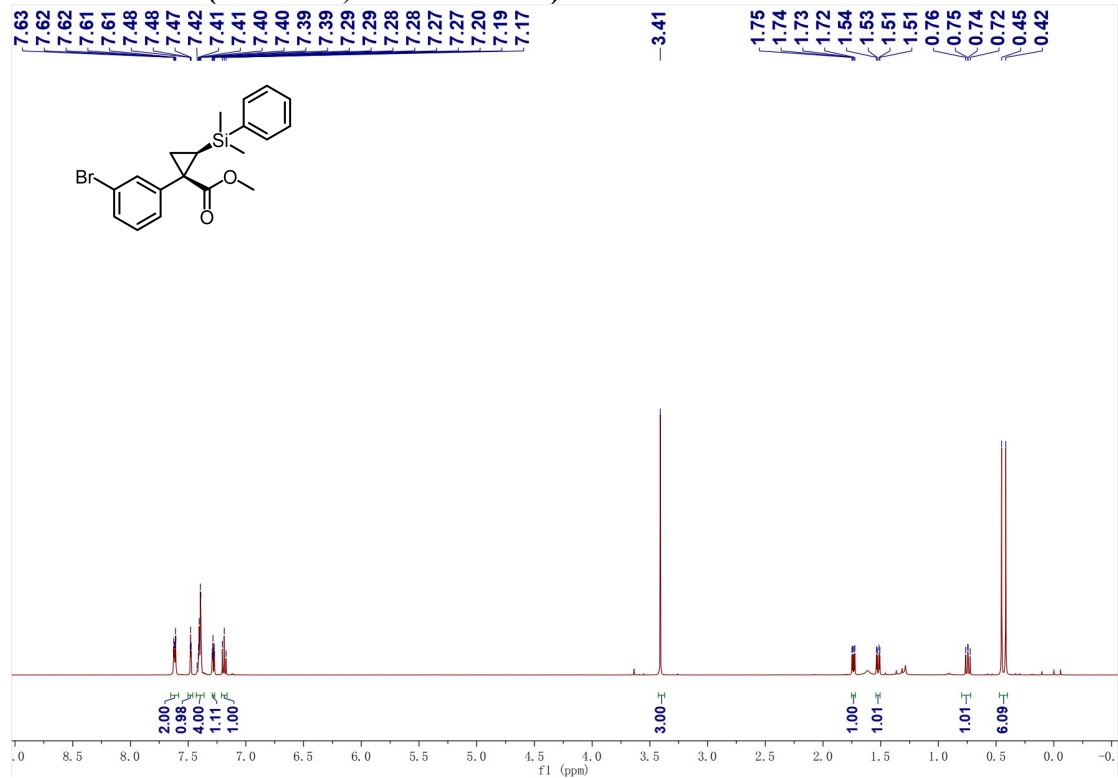
¹H NMR of 3h (400 MHz, Chloroform-d)



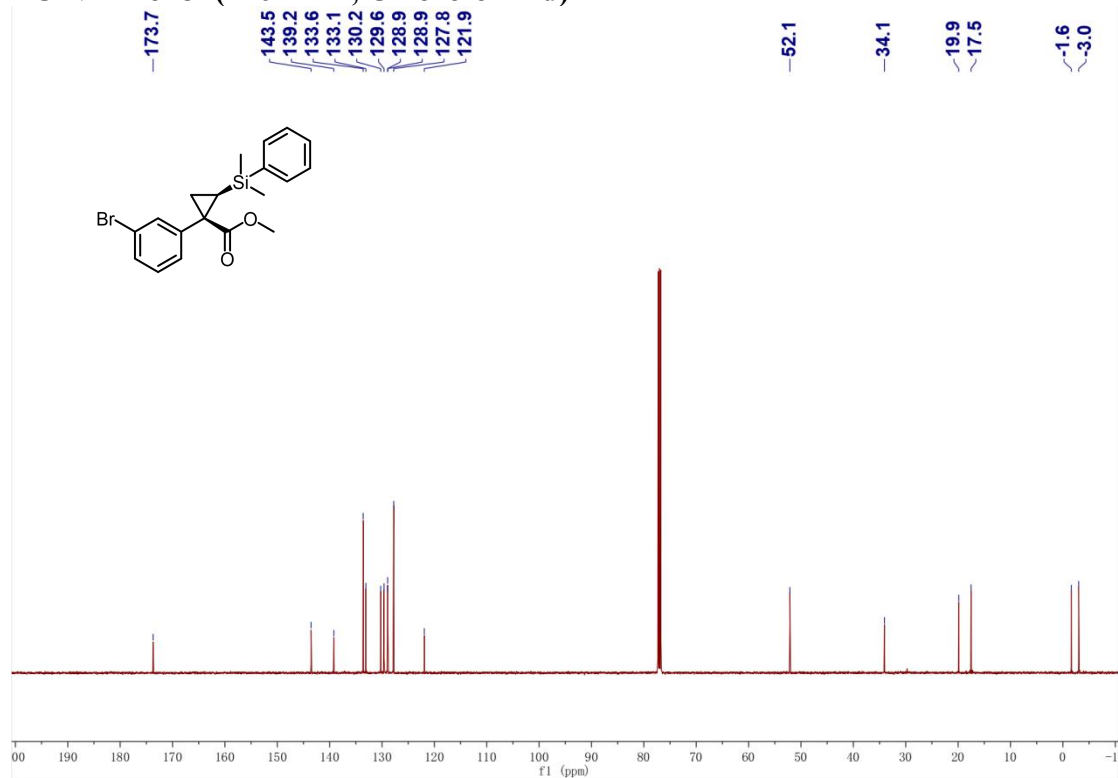
¹³C NMR of 3h (126 MHz, Chloroform-d)



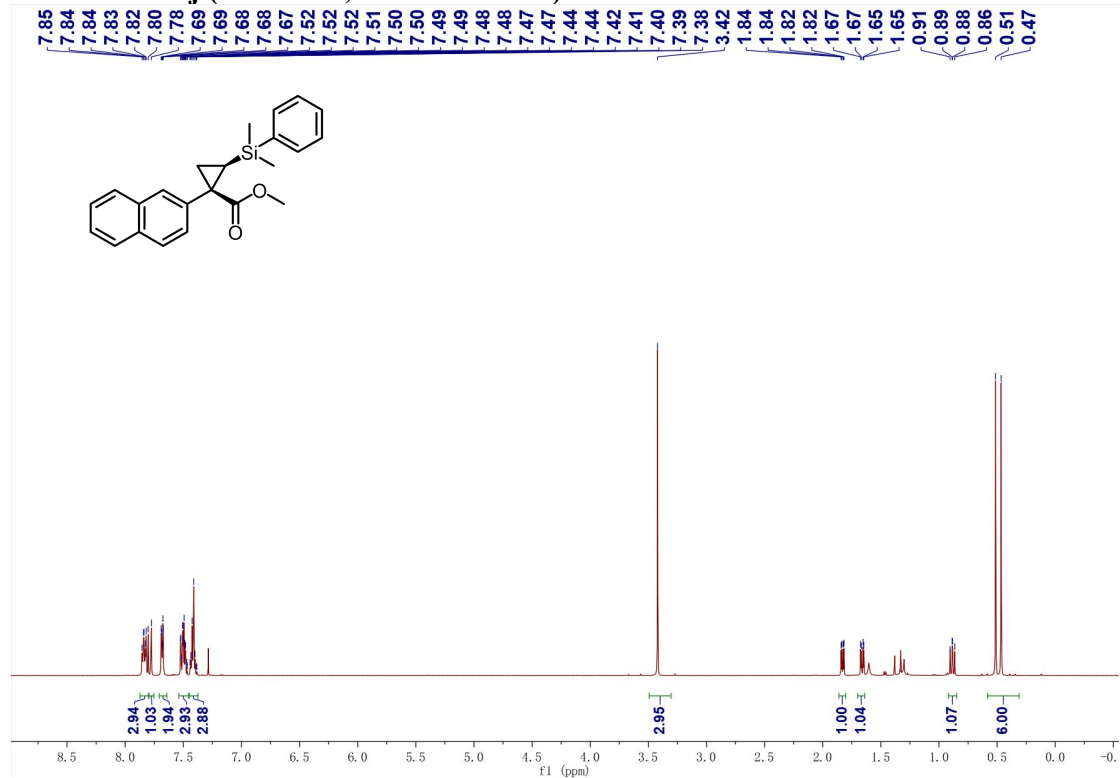
¹H NMR of 3i (500 MHz, Chloroform-d)



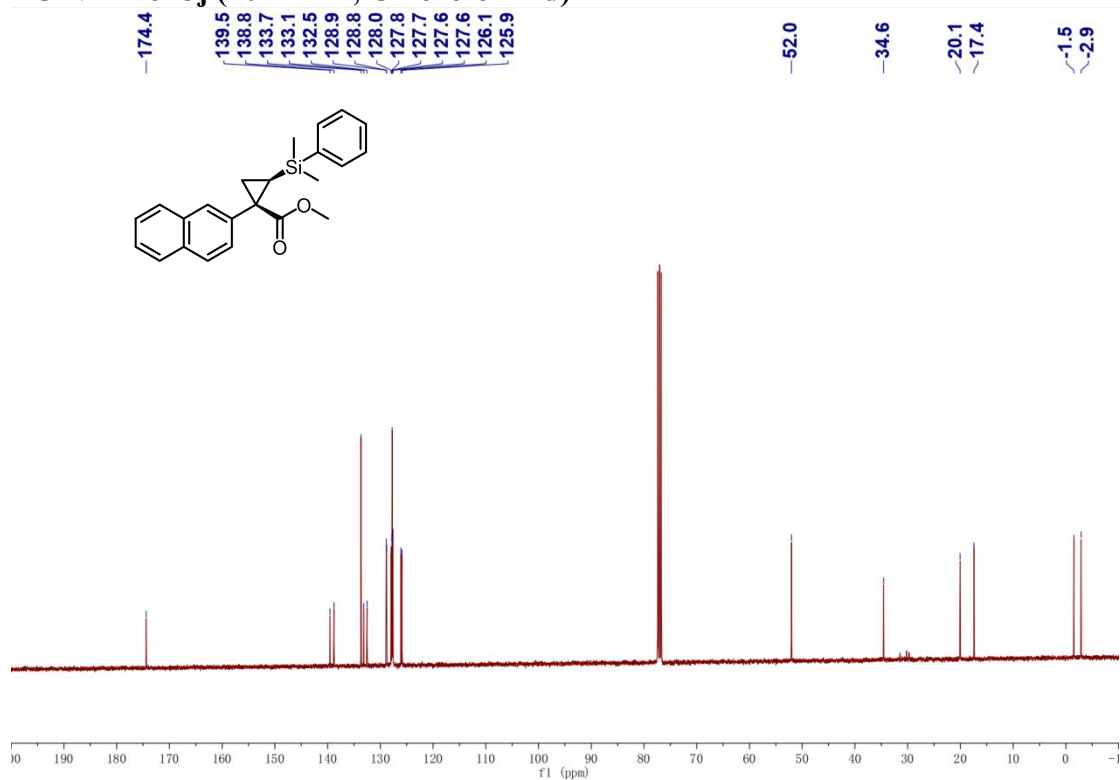
¹³C NMR of 3i (126 MHz, Chloroform-d)



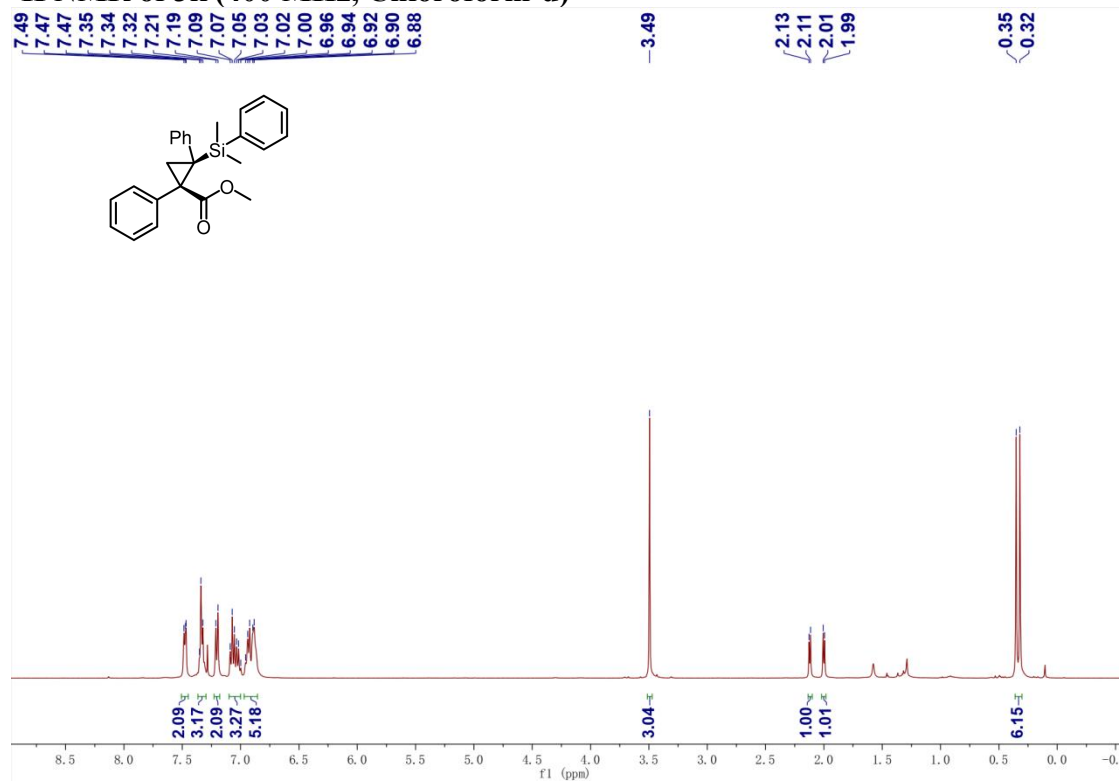
¹H NMR of 3j (500 MHz, Chloroform-d)



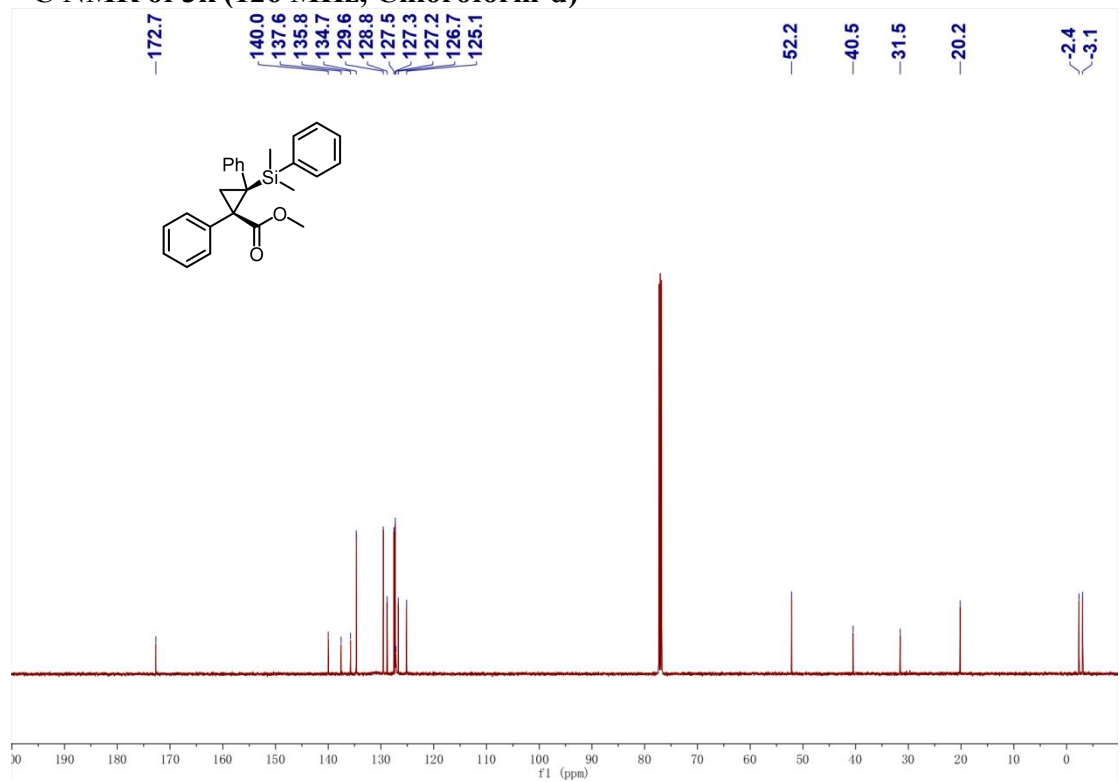
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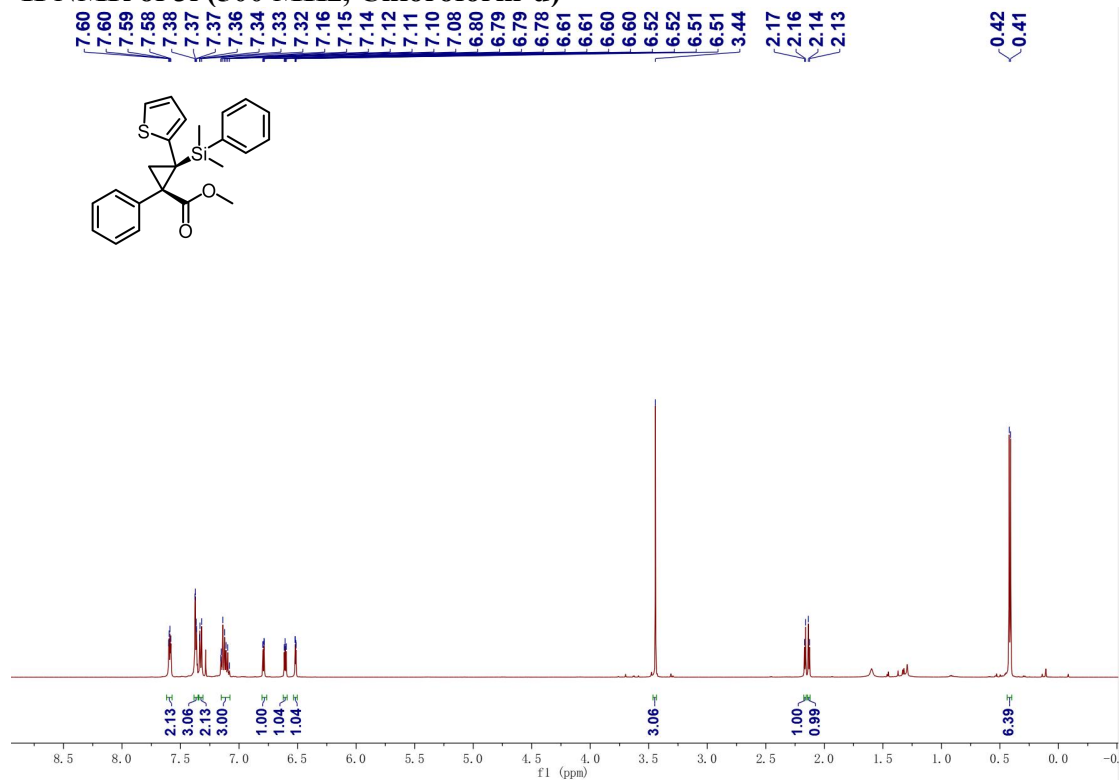
¹H NMR of 3k (400 MHz, Chloroform-d)



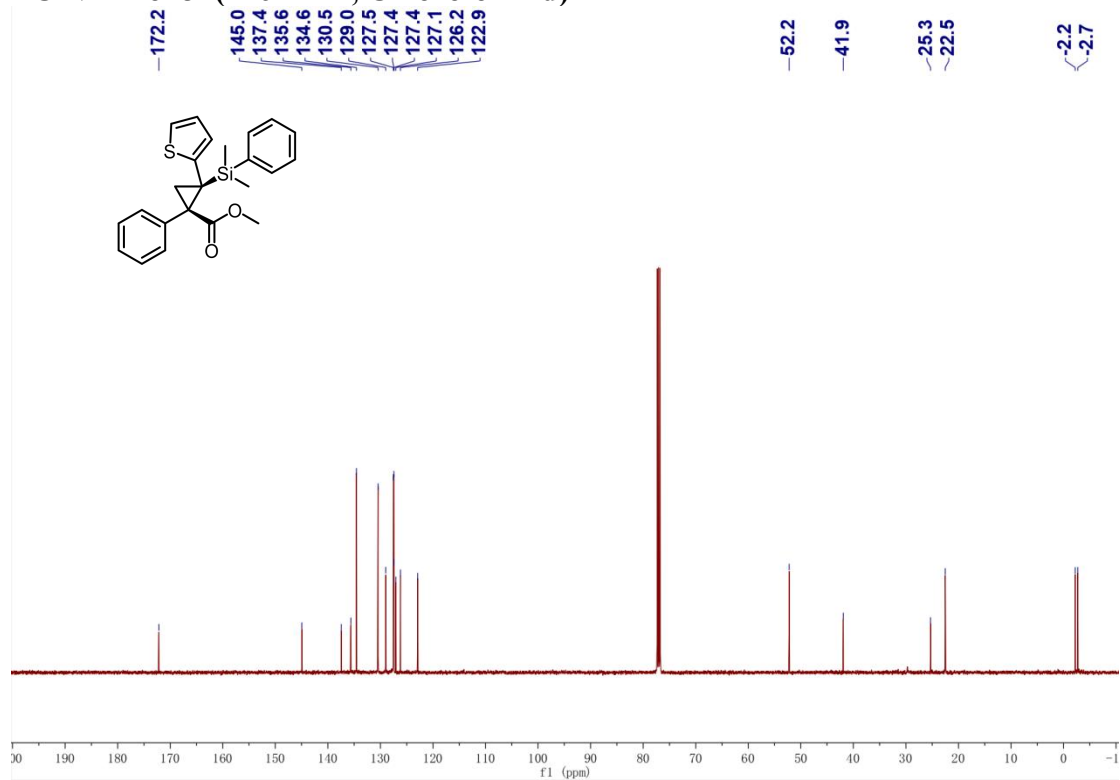
¹³C NMR of 3k (126 MHz, Chloroform-d)



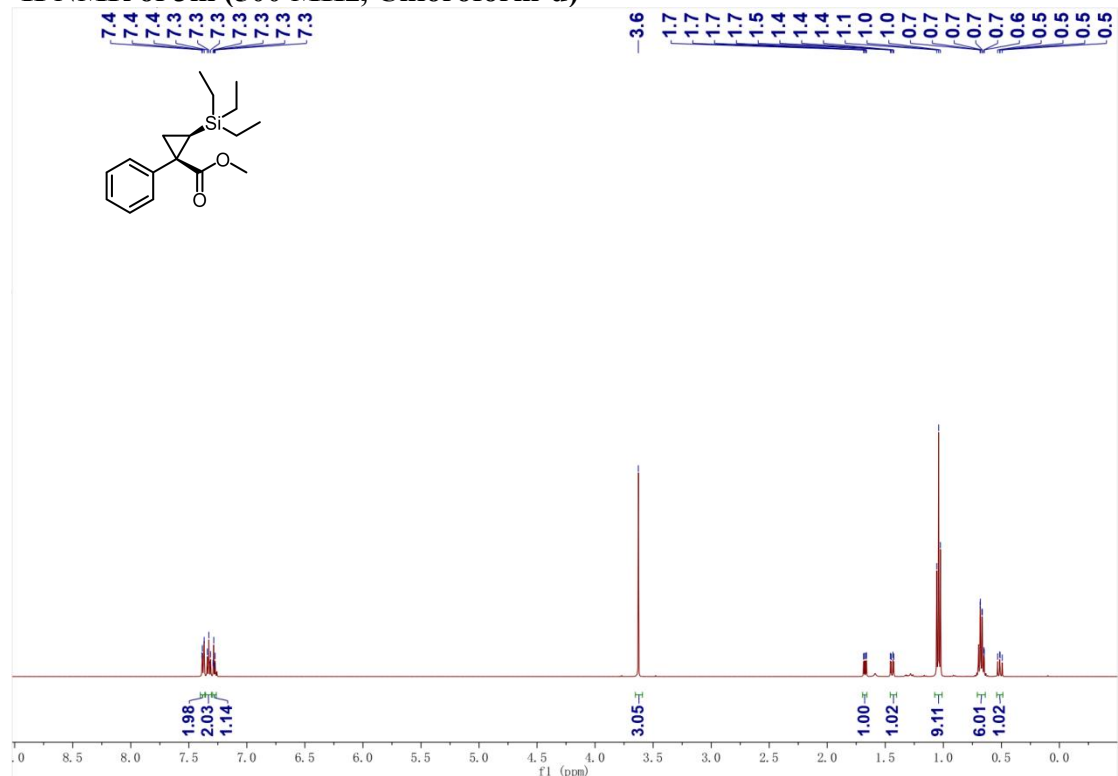
¹H NMR of 3l (500 MHz, Chloroform-d)



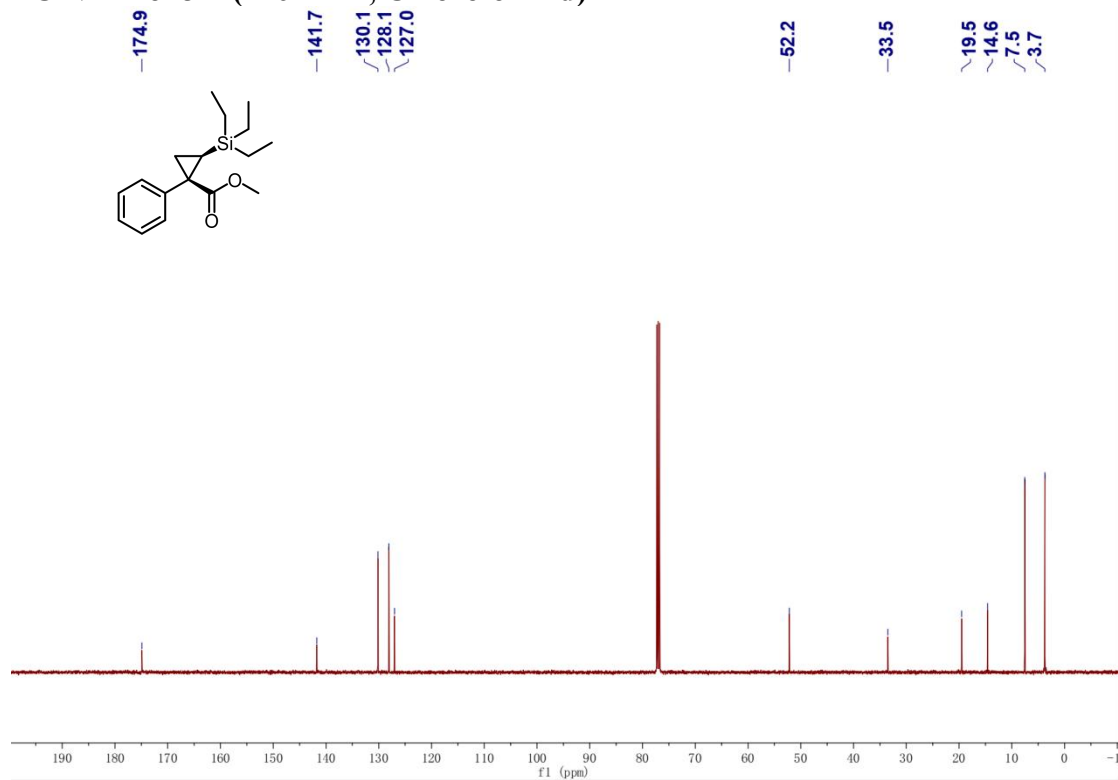
¹³C NMR of 3l (126 MHz, Chloroform-d)



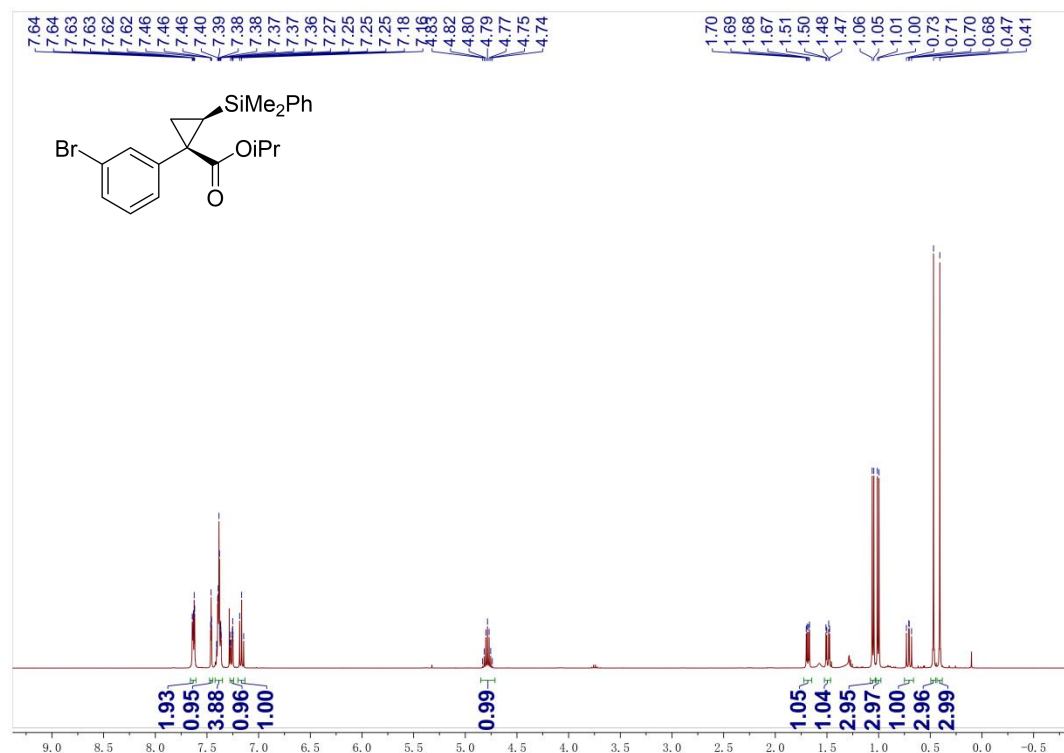
¹H NMR of 3m (500 MHz, Chloroform-d)



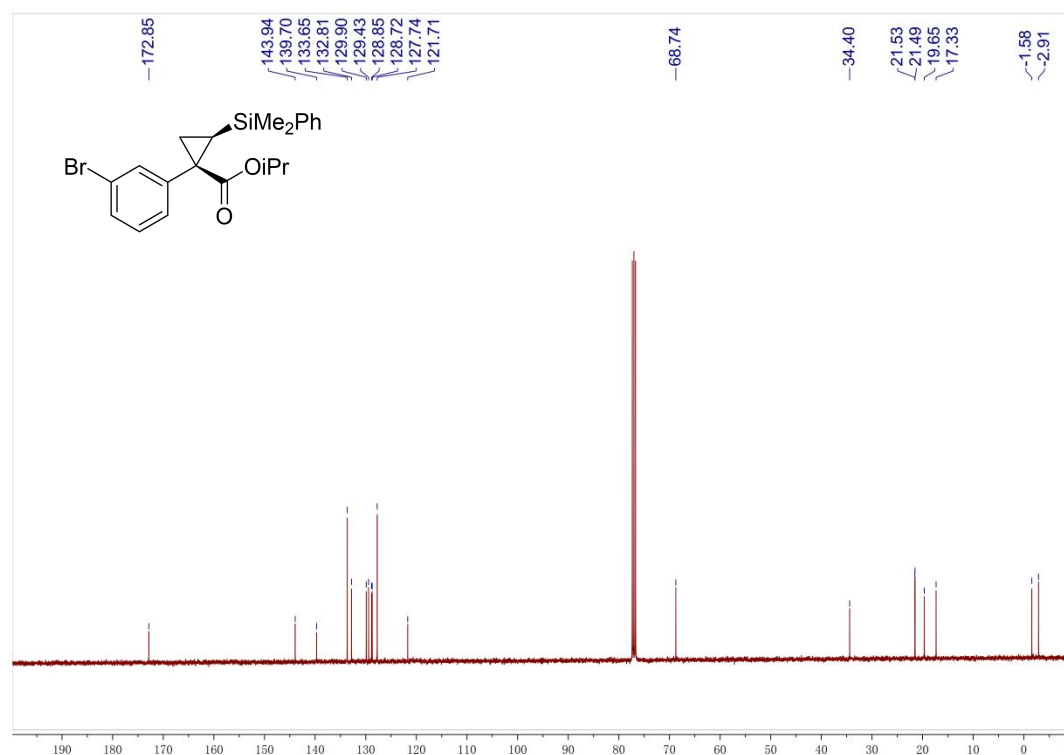
¹³C NMR of 3m (126 MHz, Chloroform-d)



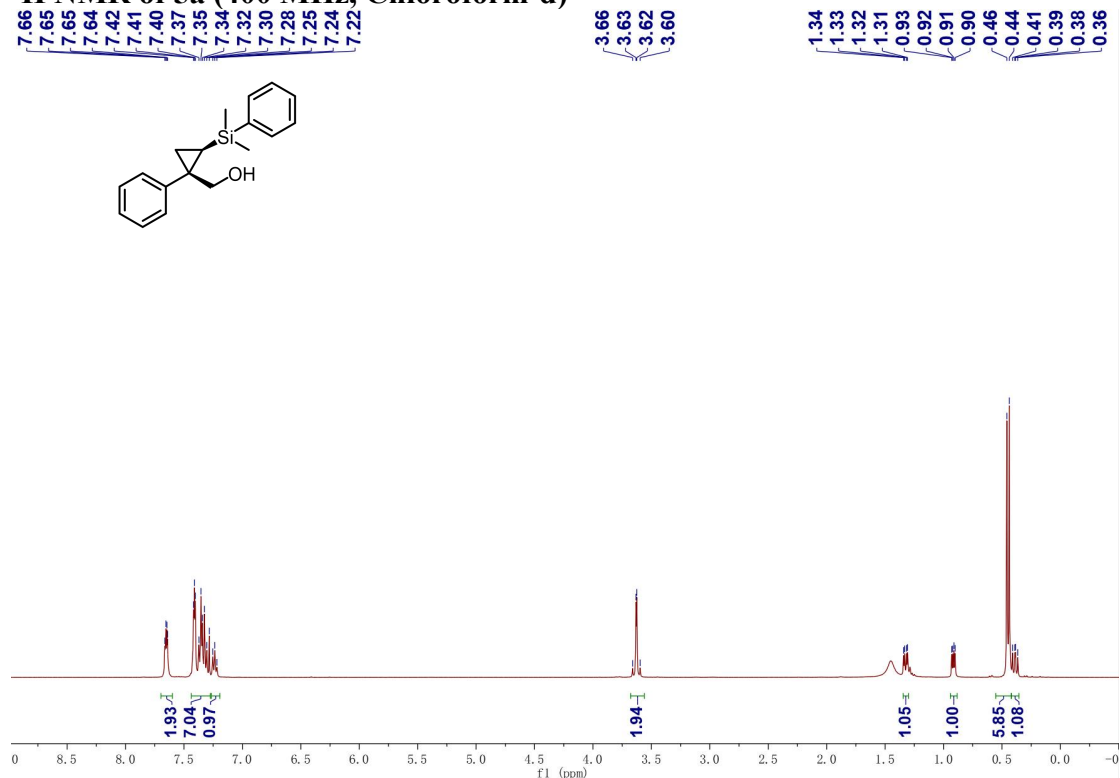
^1H NMR of 3n (400 MHz, Chloroform-d)



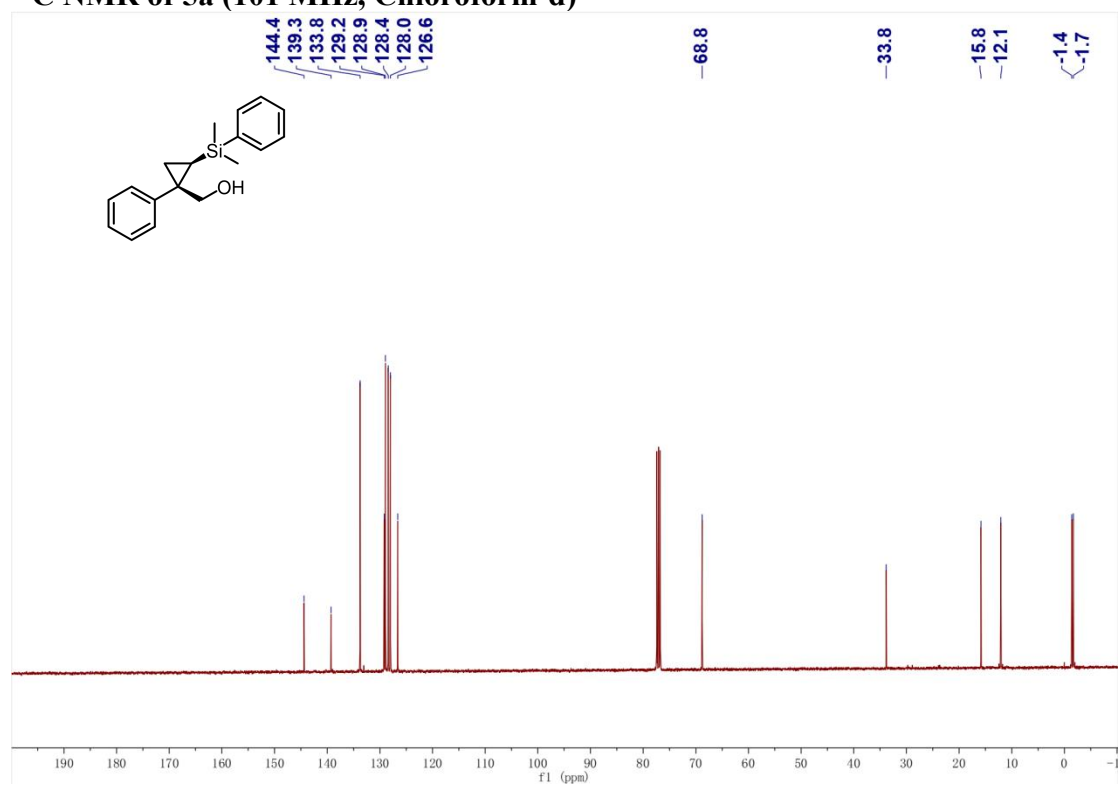
^{13}C NMR of 3n (101 MHz, Chloroform-d)



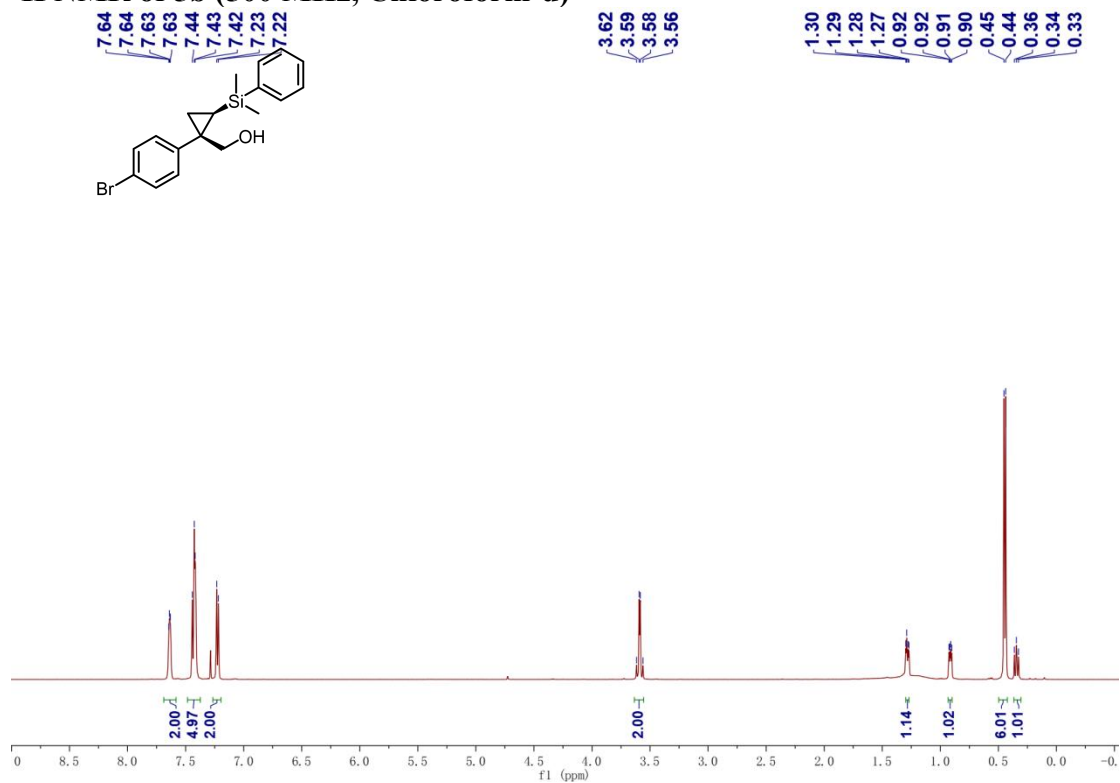
¹H NMR of 5a (400 MHz, Chloroform-d)



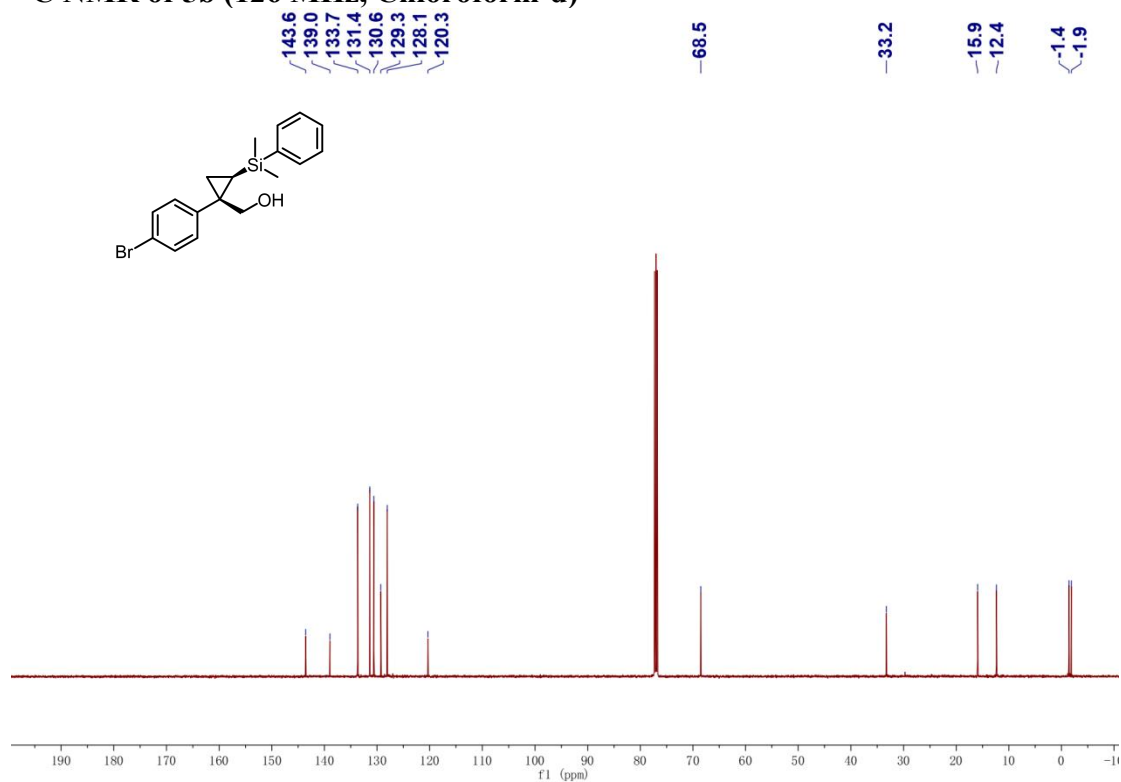
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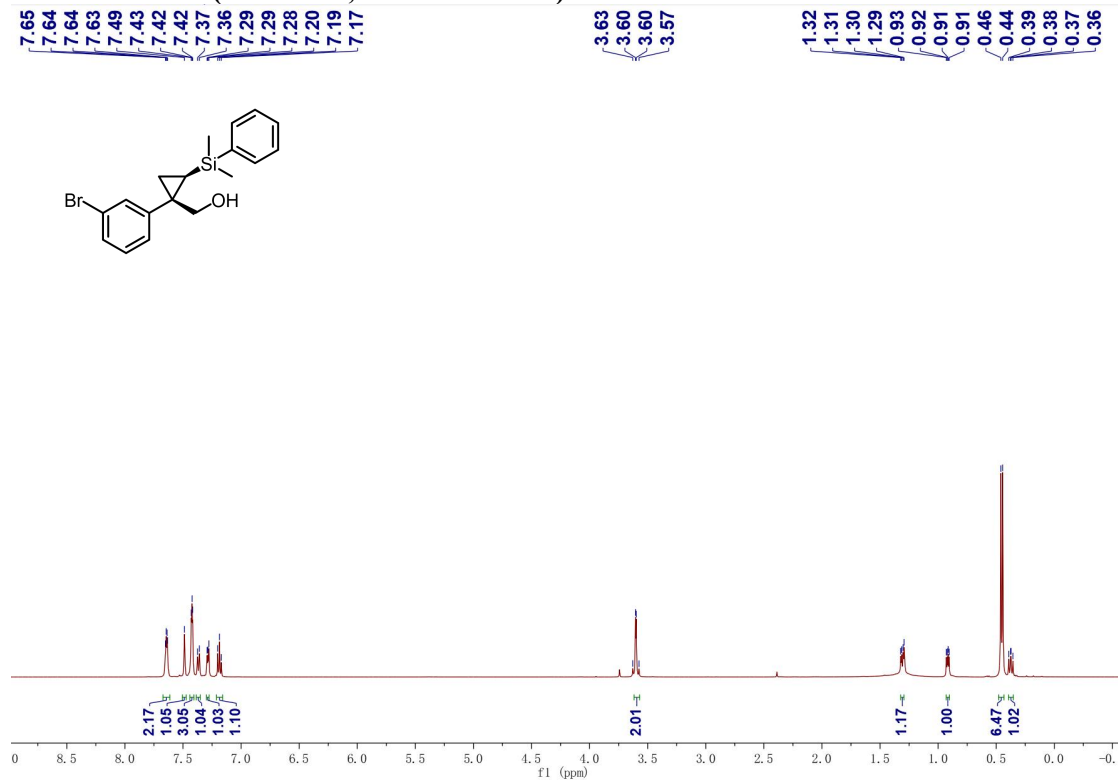
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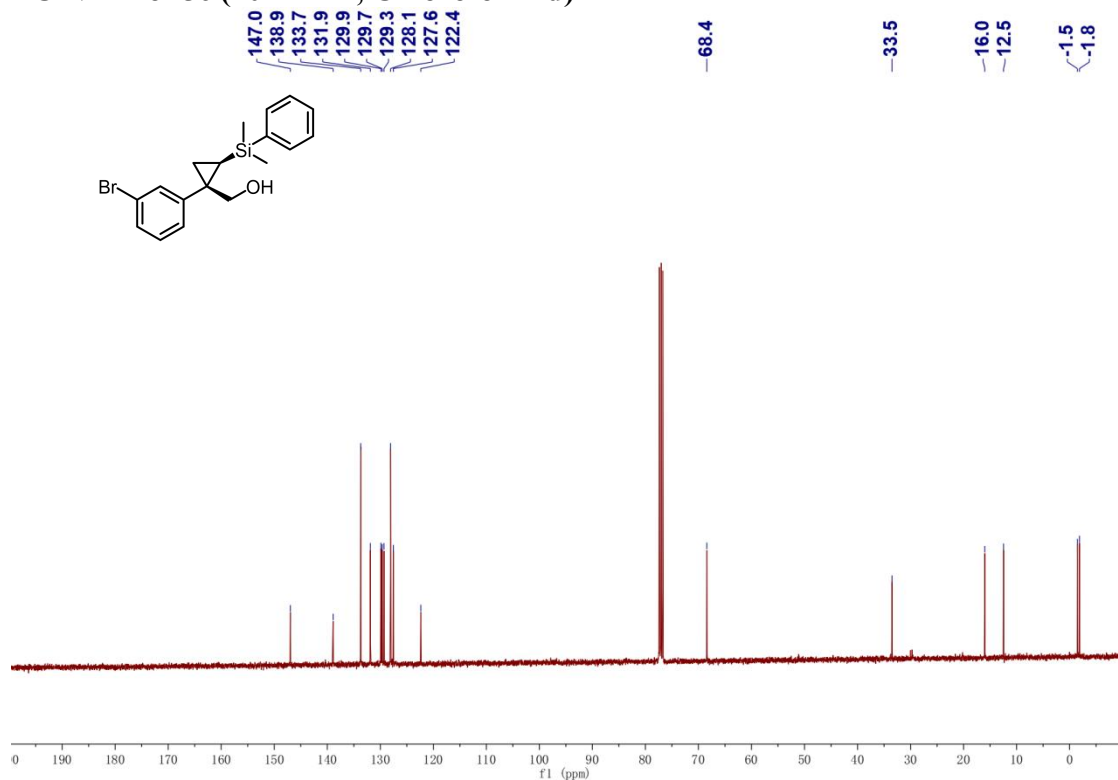
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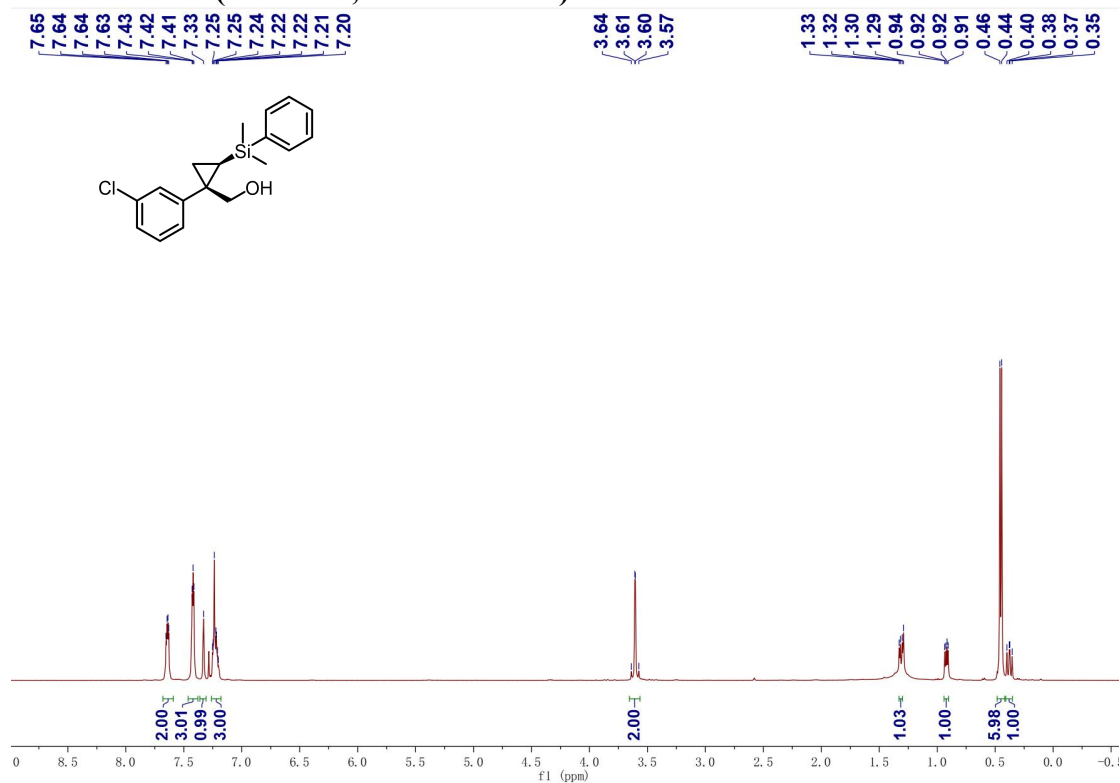
¹H NMR of 5c (500 MHz, Chloroform-d)



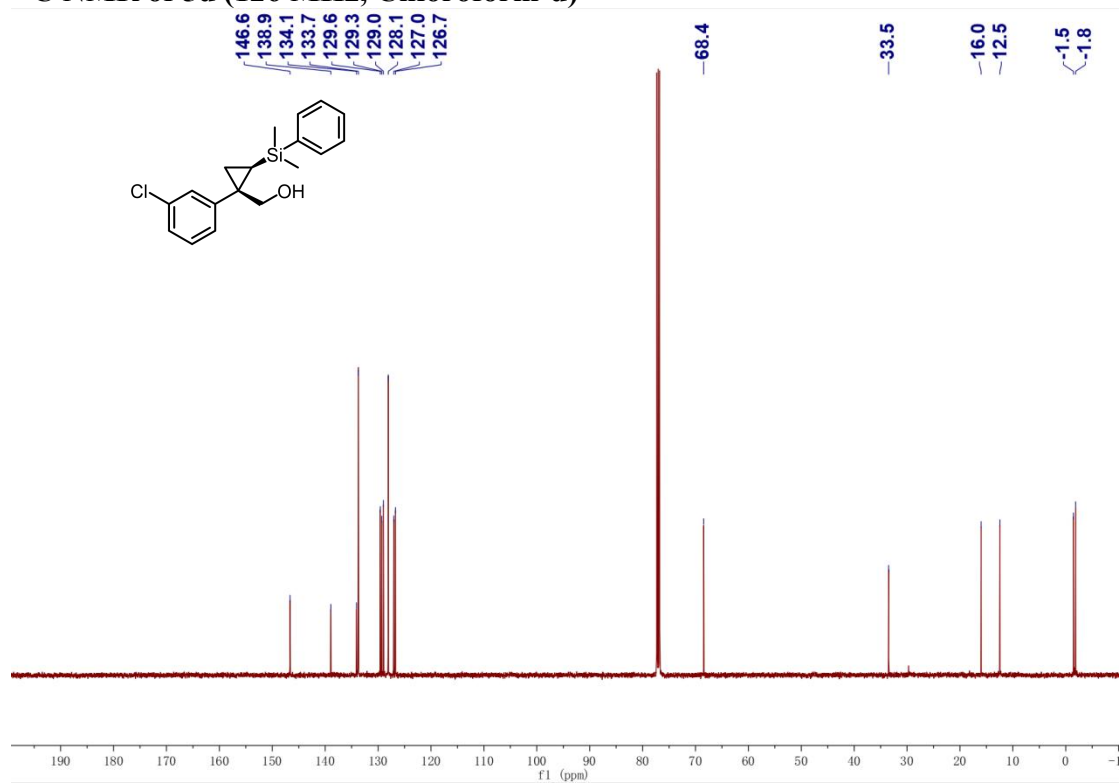
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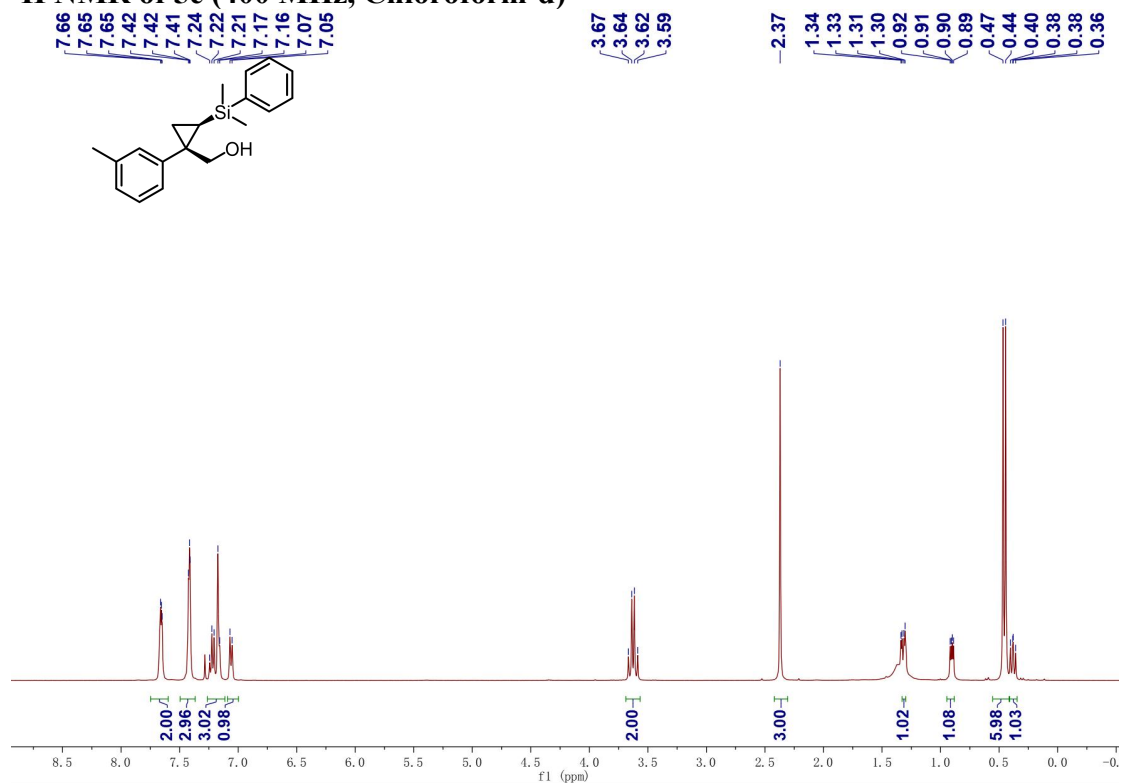
¹H NMR of 5d (400 MHz, Chloroform-d)



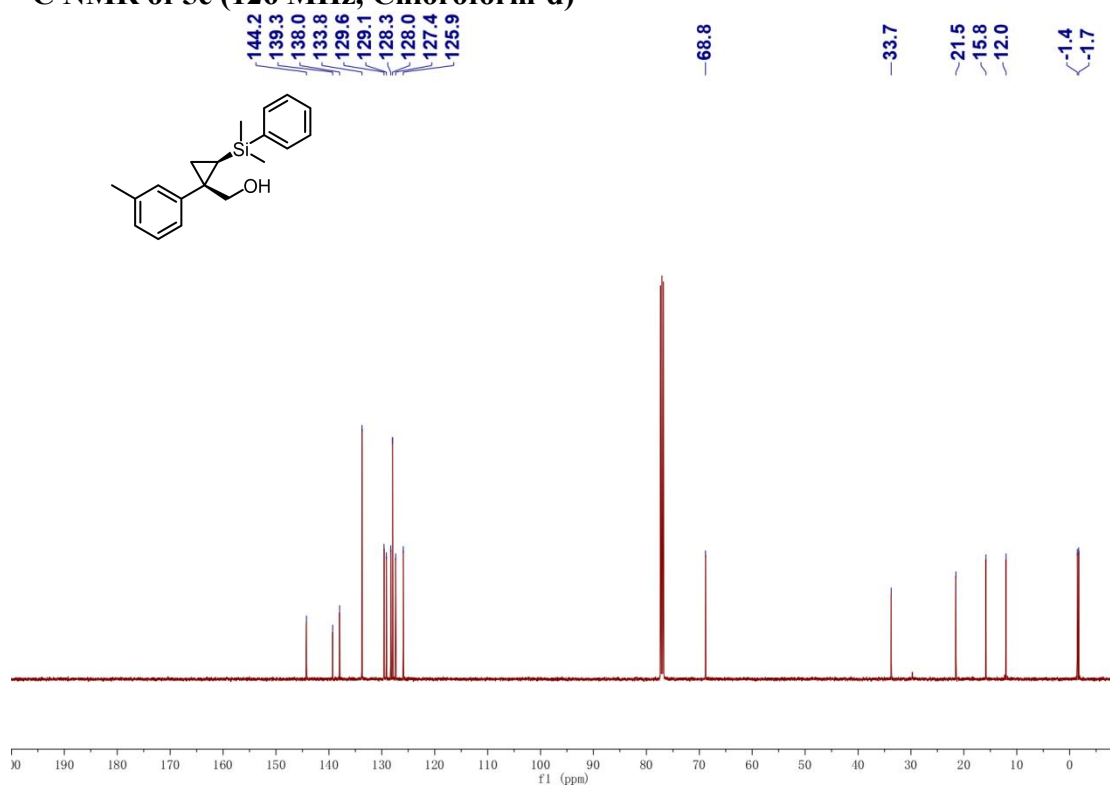
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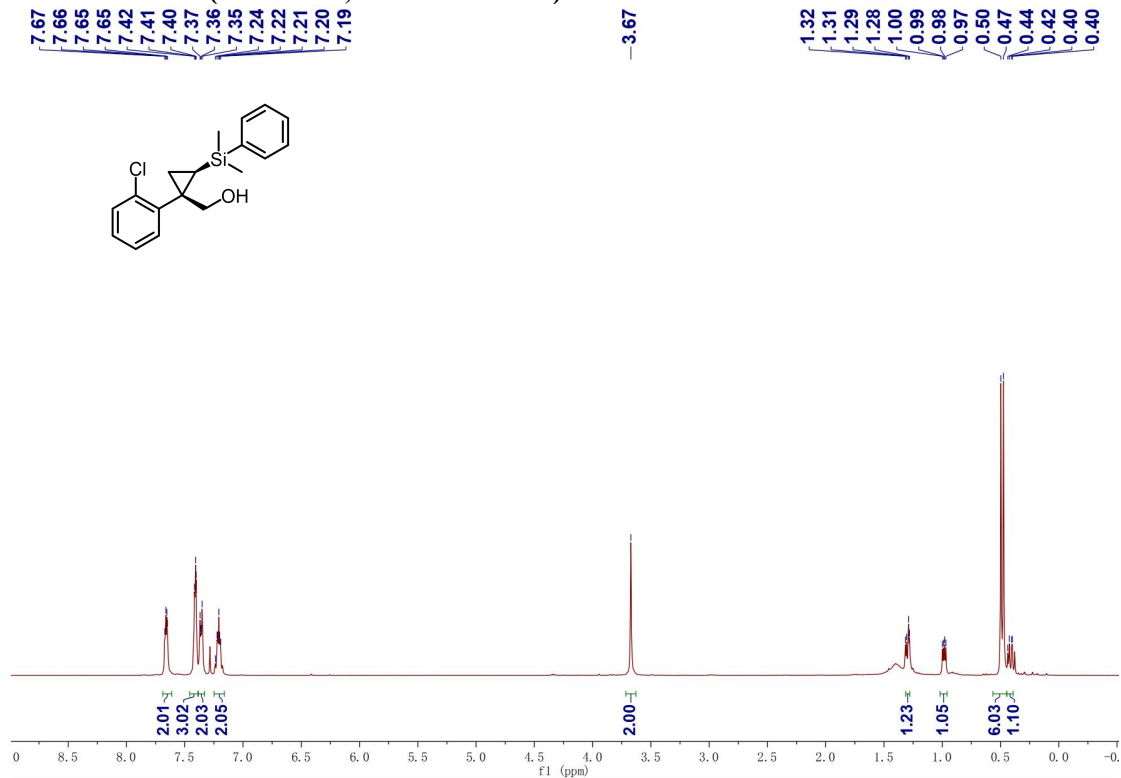
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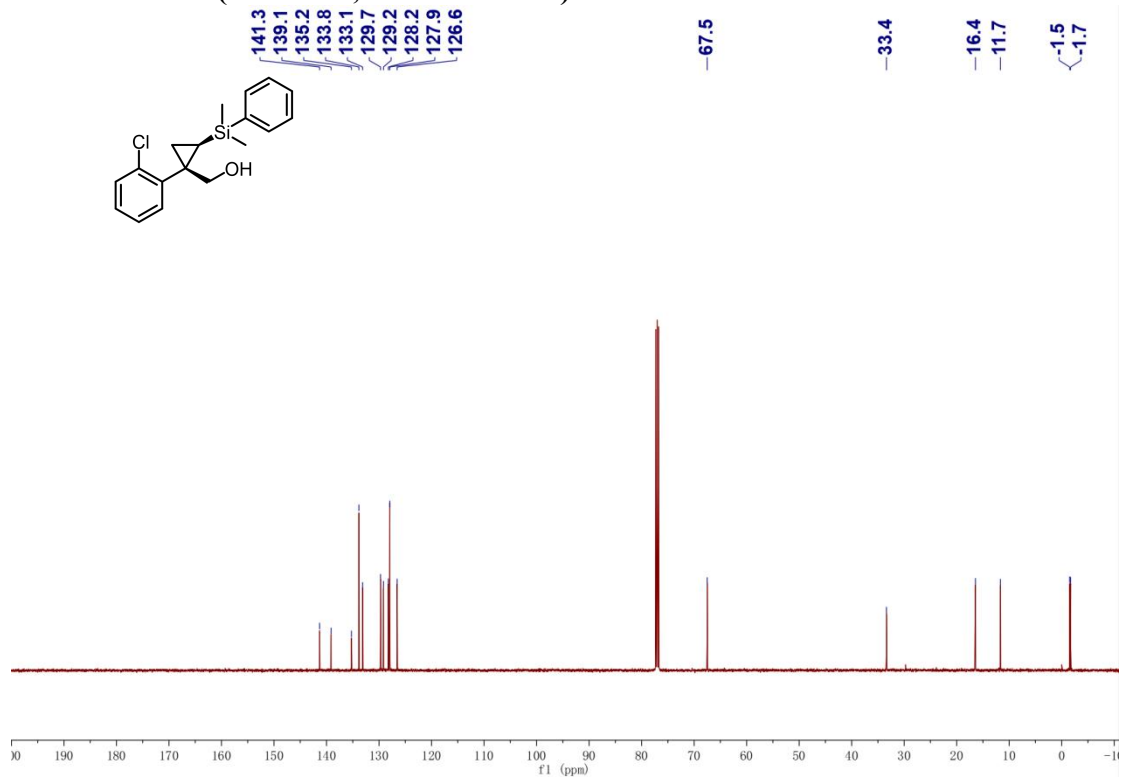
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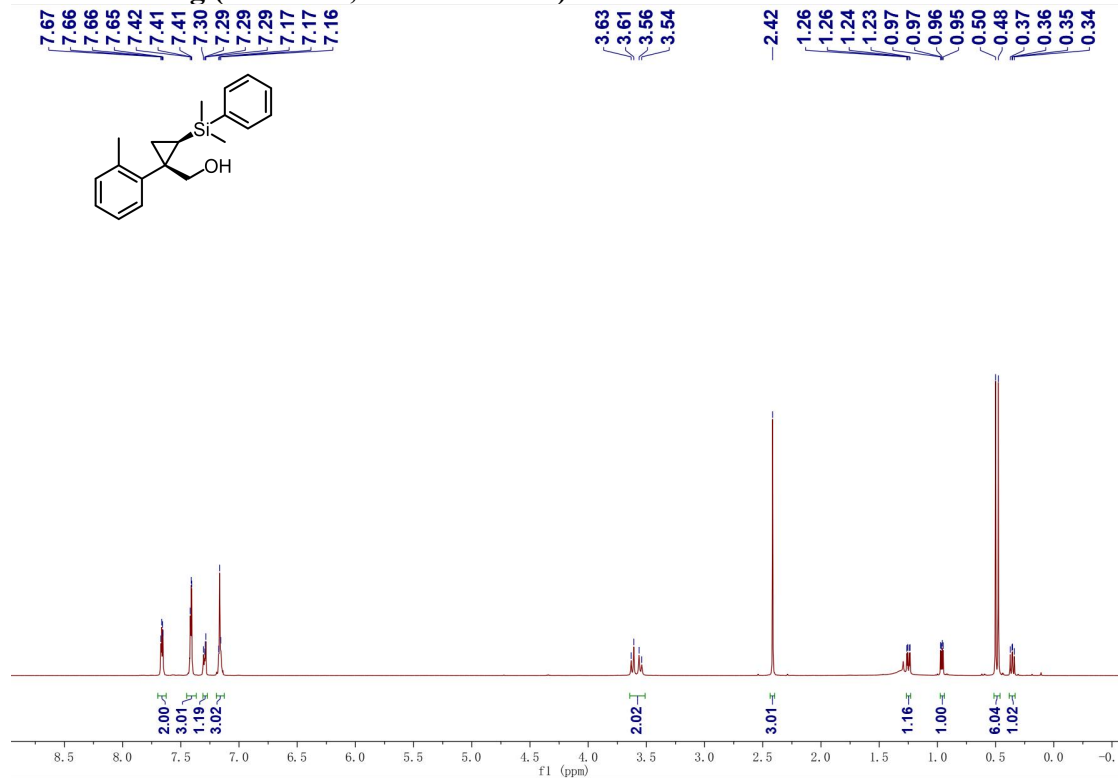
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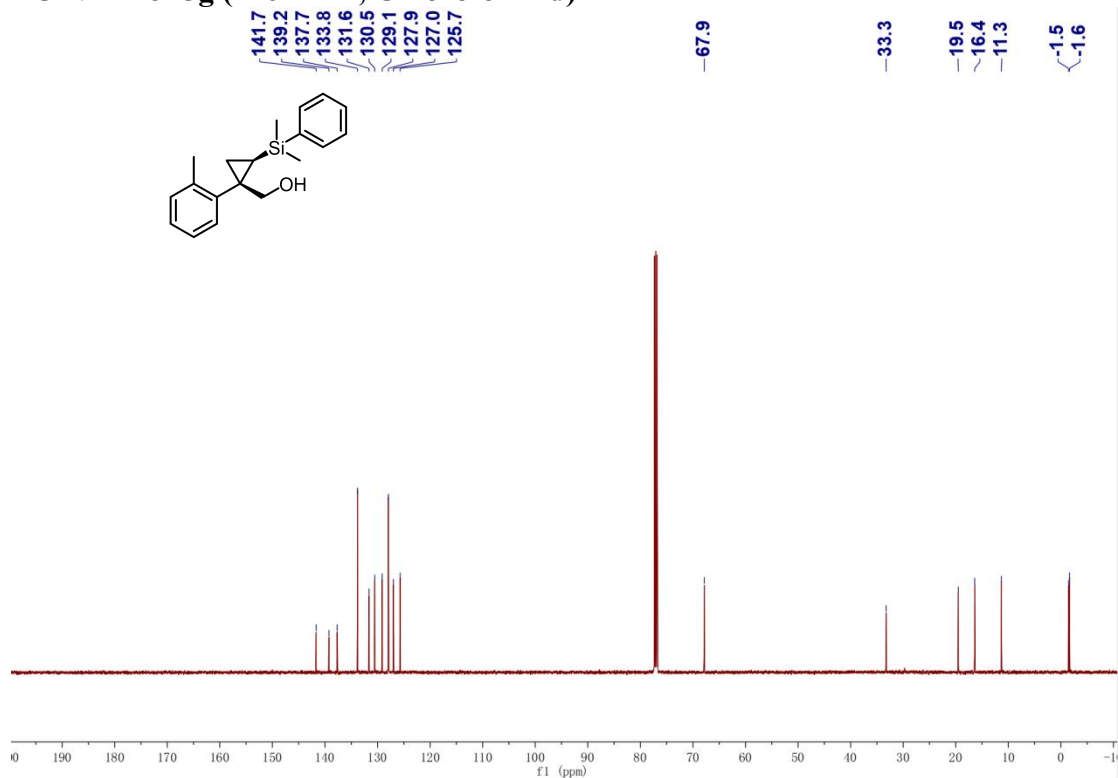
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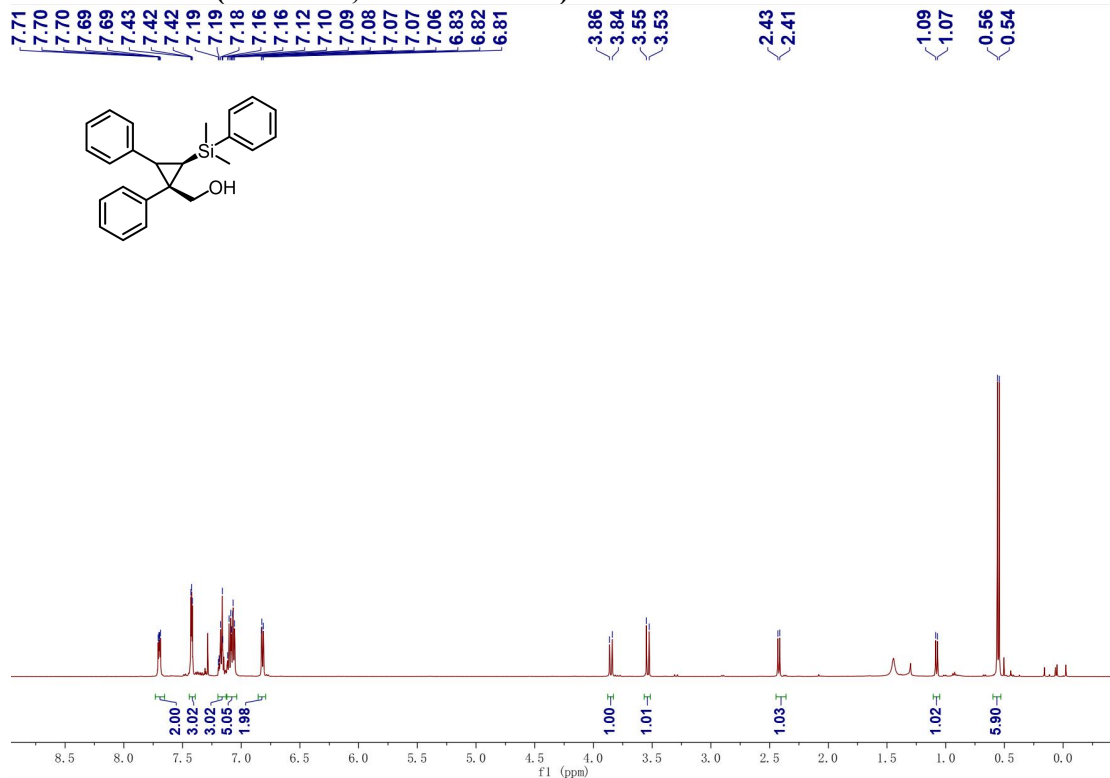
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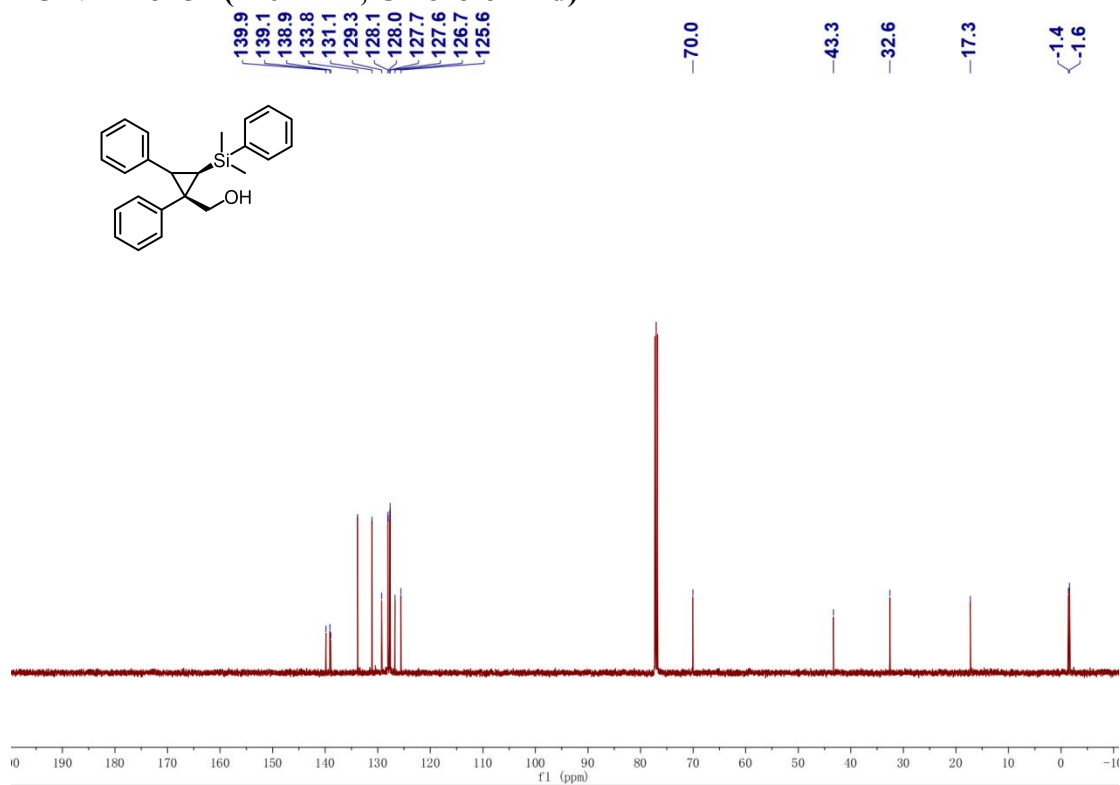
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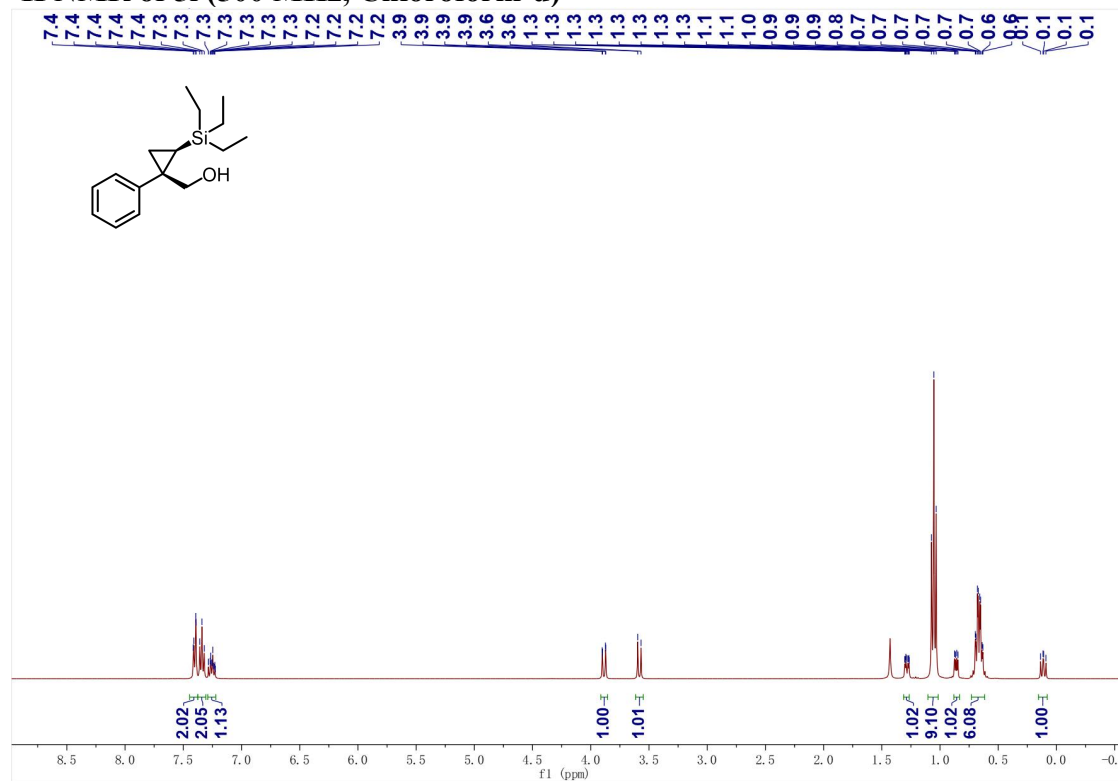
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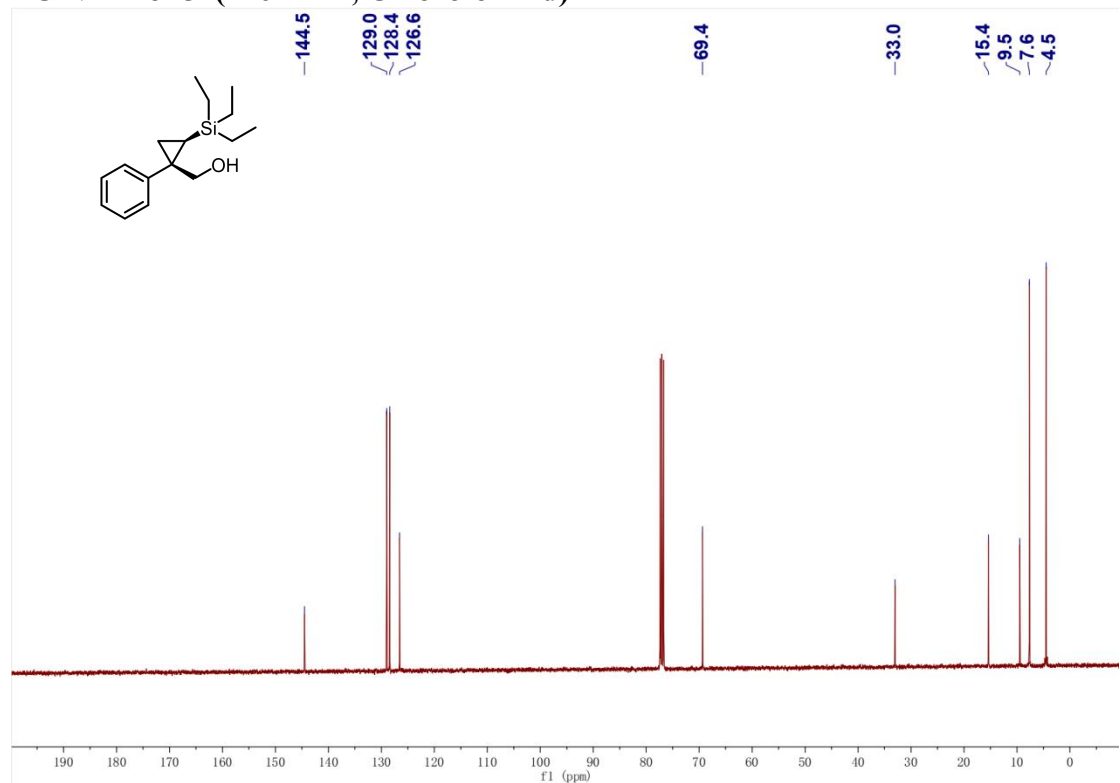
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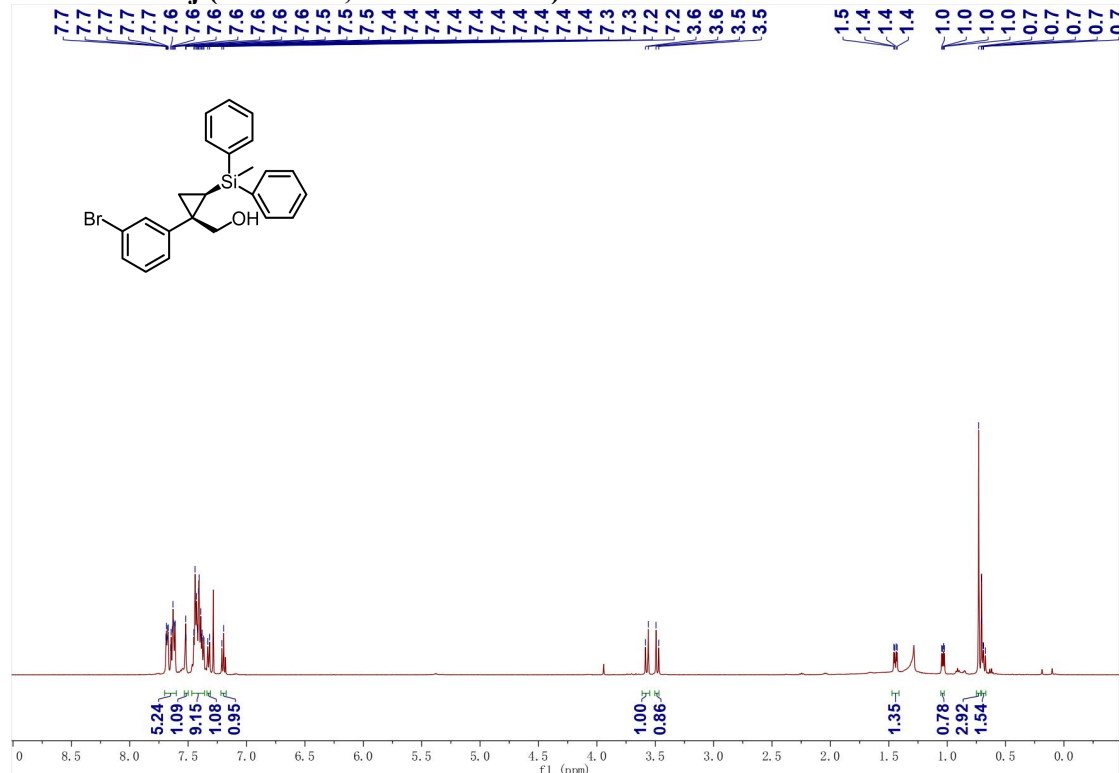
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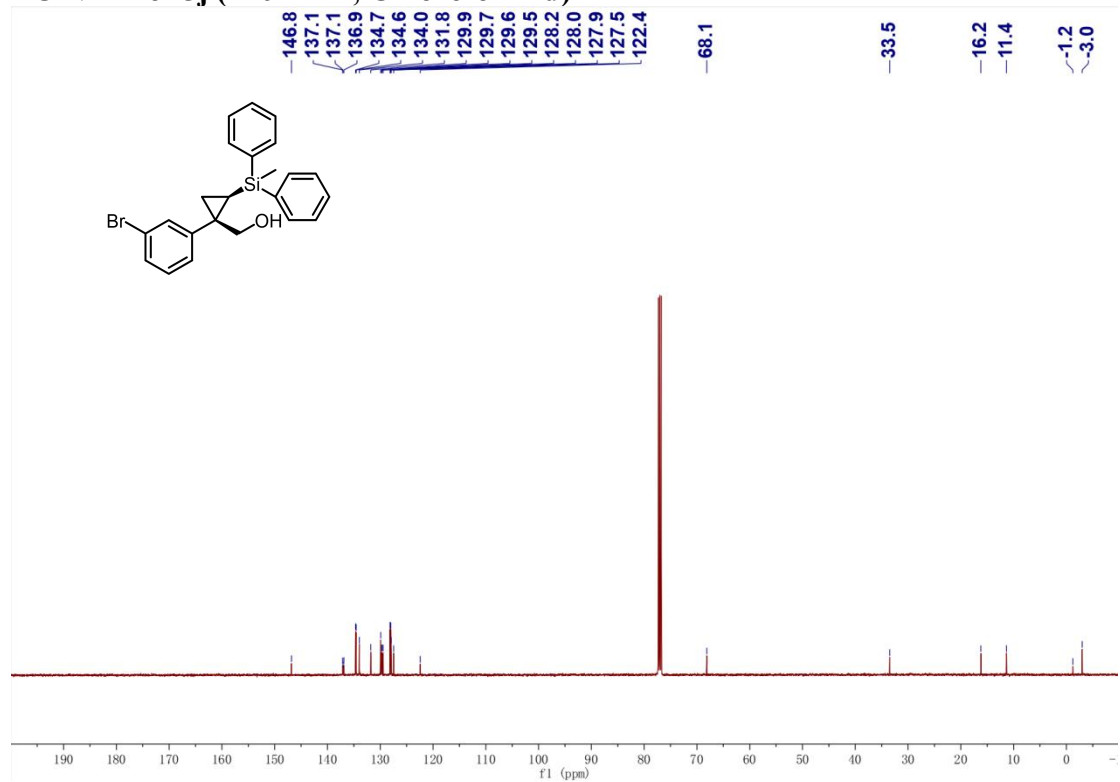
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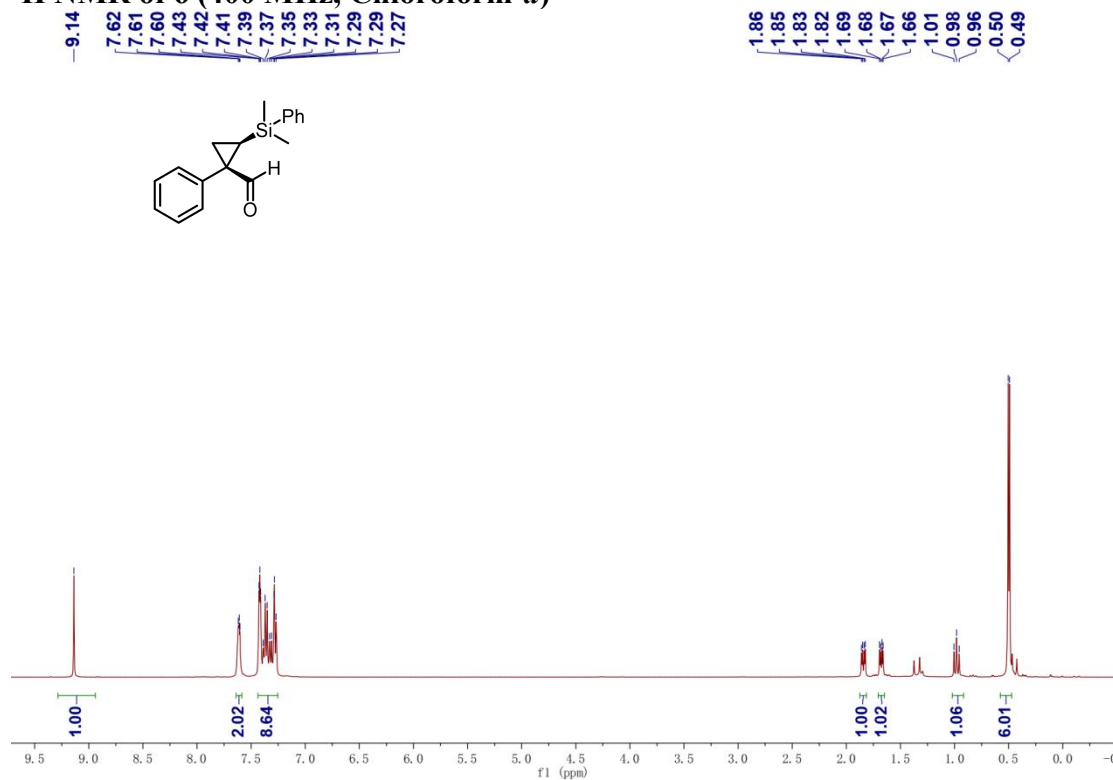
¹H NMR of 5j (400 MHz, Chloroform-d)



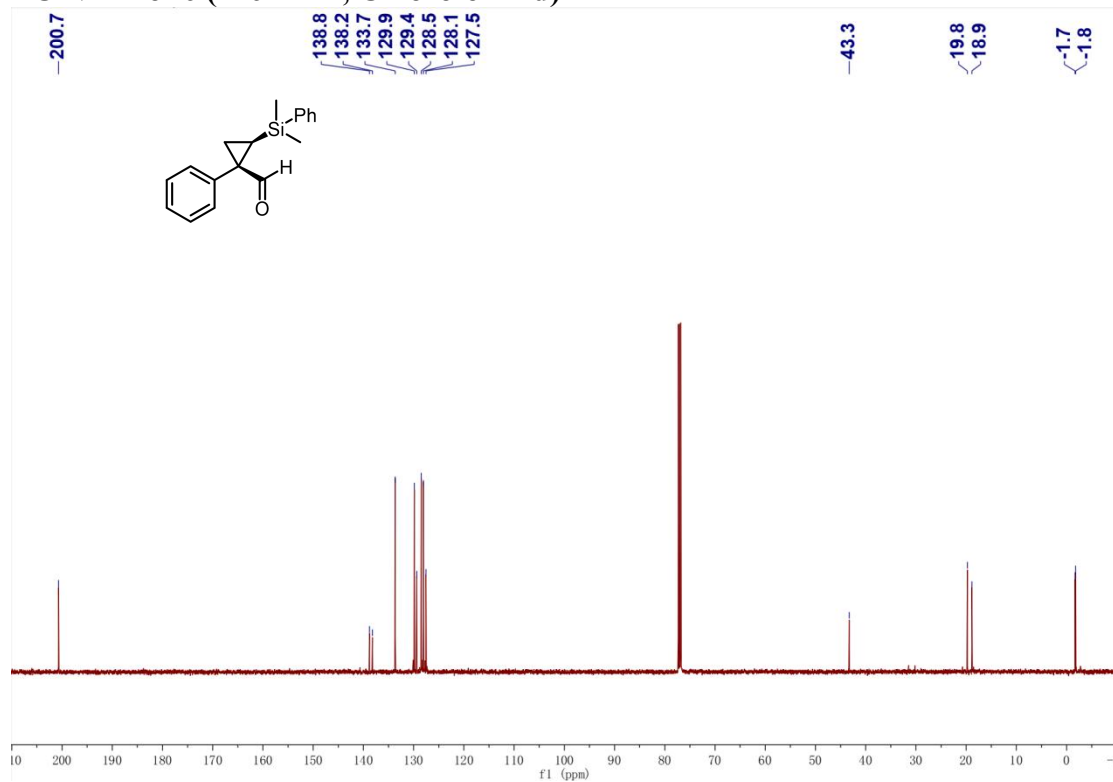
¹³C NMR of 5j (126 MHz, Chloroform-d)



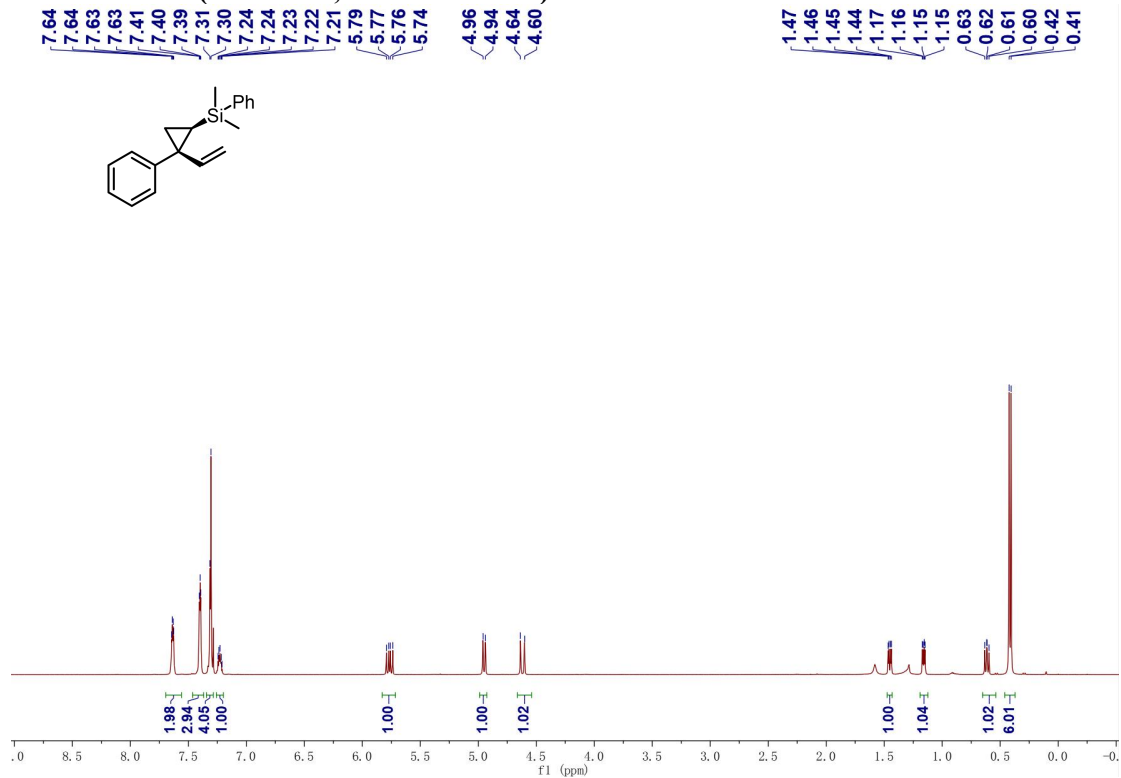
¹H NMR of 6 (400 MHz, Chloroform-d)



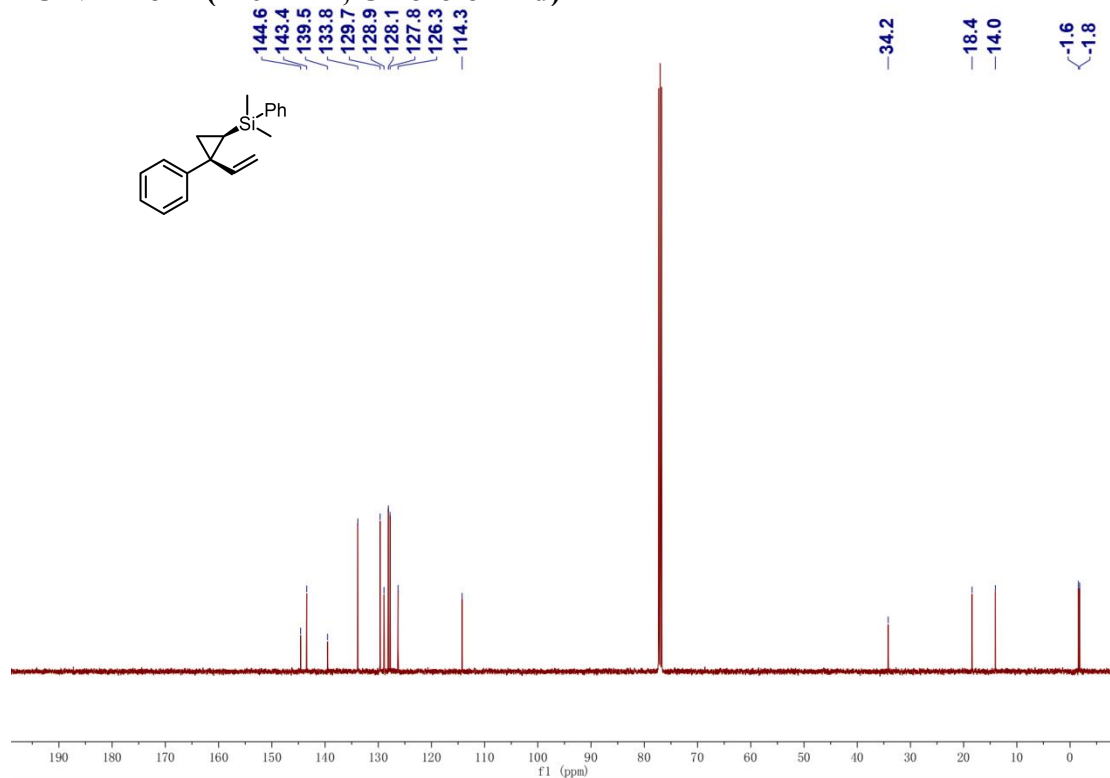
¹³C NMR of 6 (126 MHz, Chloroform-d)



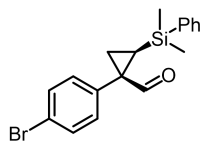
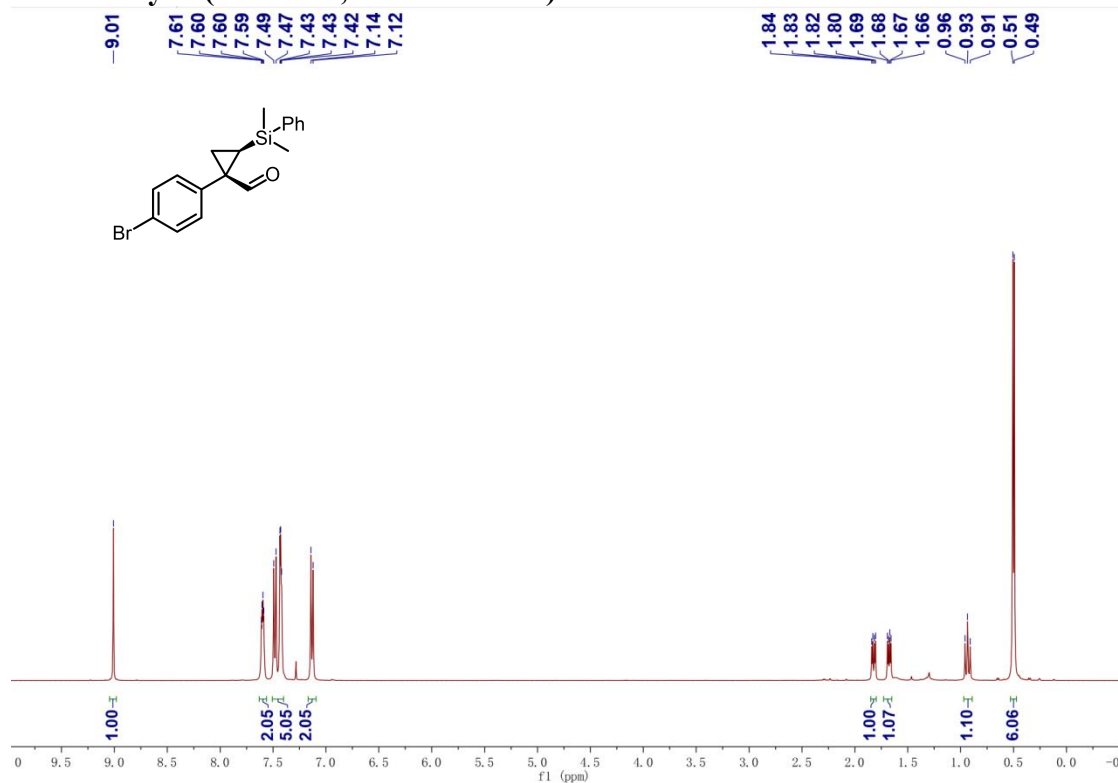
¹H NMR of 7 (400 MHz, Chloroform-d)



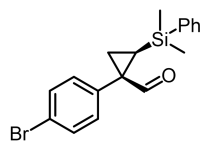
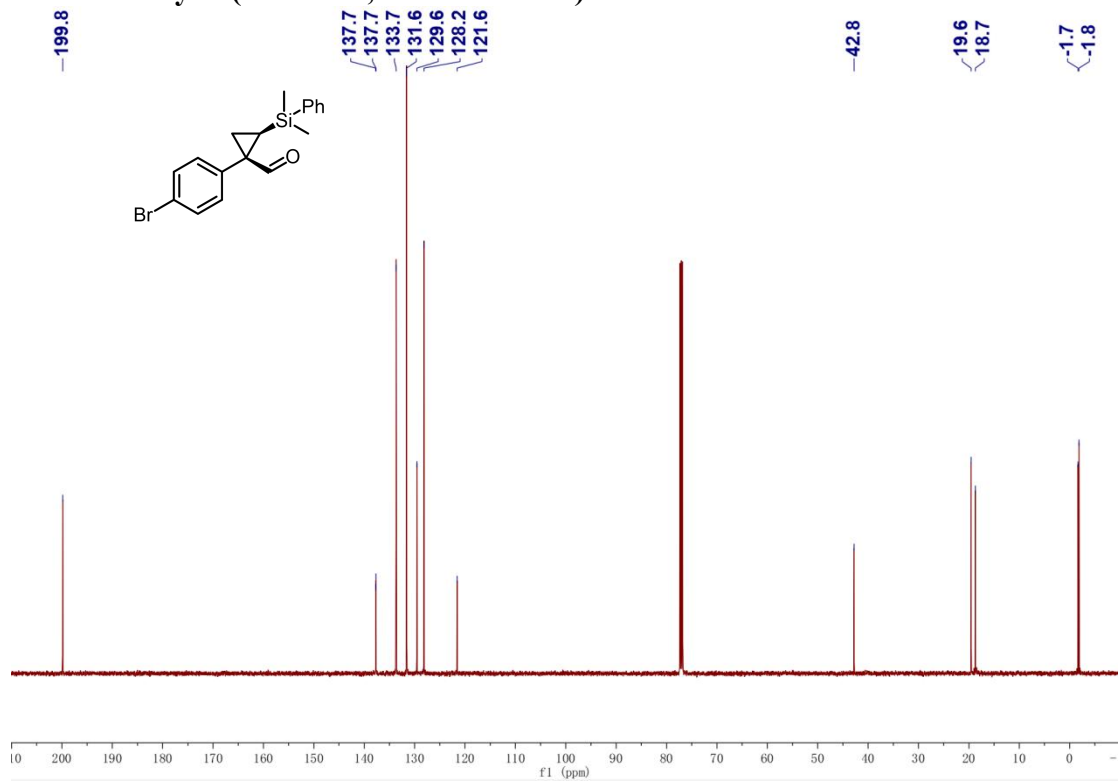
¹³C NMR of 7 (126 MHz, Chloroform-d)



¹H NMR of (1S,2R)-1-(4-bromophenyl)-2-(dimethyl(phenyl)silyl)cyclopropane-1-carbaldehyde (400 MHz, Chloroform-*d*)



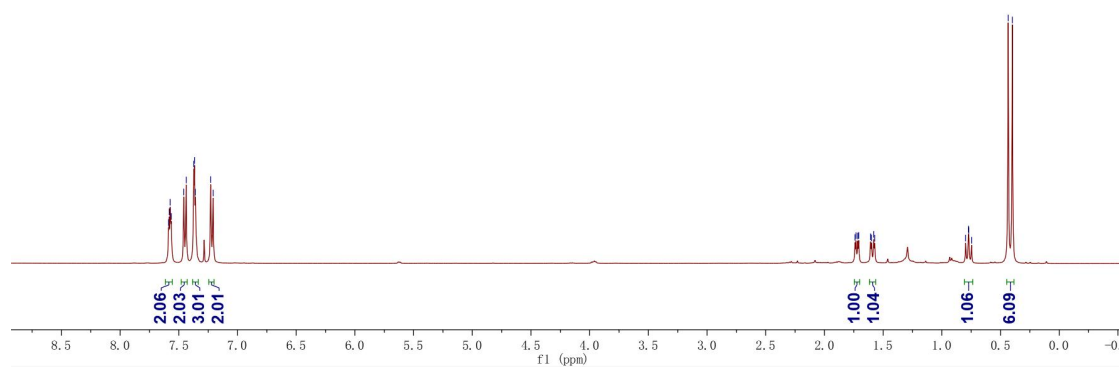
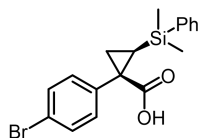
¹³C NMR of (1S,2R)-1-(4-bromophenyl)-2-(dimethyl(phenyl)silyl)cyclopropane-1-carbaldehyde (126 MHz, Chloroform-*d*)



¹H NMR of 8 (400 MHz, Chloroform-*d*)

7.587
7.582
7.573
7.564
7.458
7.437
7.372
7.367
7.360
7.229
7.208

1.739
1.731
1.716
1.708
1.607
1.599
1.580
1.572
0.797
0.773
0.771
0.747
0.435
0.399



¹³C NMR of 8 (126 MHz, Chloroform-*d*)

180.2

139.6
139.2
133.5
131.8
131.3
129.0
127.8
121.3

33.5

20.9
19.1

1.9
2.8

