

Supporting Information

Highly Stereoselective Synthesis of *trans*-Alkenes via Electrochemical Ni-Catalyzed Hydroarylation of Alkynes with Aryl Iodides

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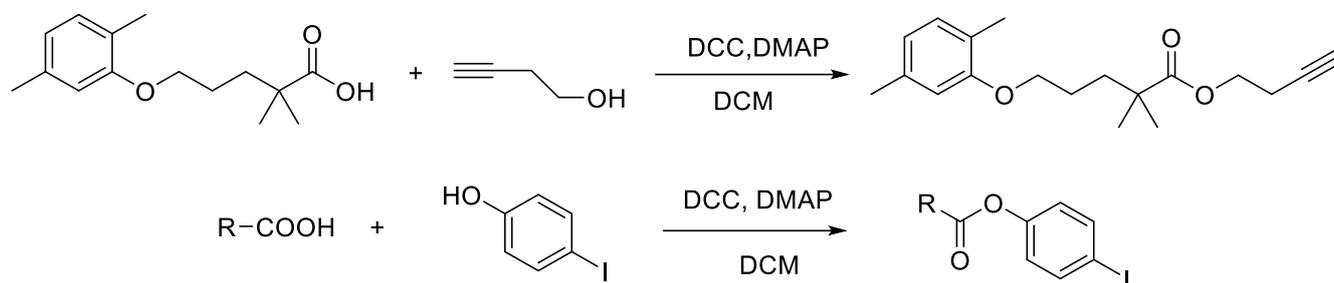
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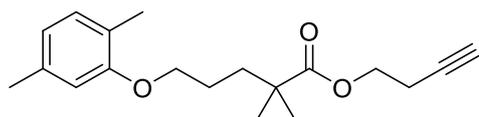
1. General Information

All commercially available reagents were directly used as received without further purification. All organic solvents applied in the reactions were not pre-dried by distillation. The electrochemical reactions were performed on a DJS-292B potentiostat (made in China) in constant current mode. All yields of products refer to the isolated yields after chromatography. All reactions were performed under three-neck flask, and two polytetrafluoroethylene electrode holders. Proton nuclear magnetic resonance (^1H NMR) spectra and carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on ZhongKe-NiuJin400 MHz (^{13}C NMR at 100 MHz) spectrometer and Bruker Ascend 400 MHz (^{13}C NMR at 100 MHz) spectrometer. Chemical shifts were recorded in parts per million (ppm, δ) relative to tetramethylsilane (TMS, δ 0.0 ppm) or chloroform (δ = 7.260, singlet). ^1H NMR splitting patterns are designated as singlet (s), doublet (d), triplet (t), quartet (q), dd (doublet of doublets); m (multiplets), and etc. High resolution mass spectral analysis (HRMS) was performed on Finnigan MAT 95 XP mass spectrometer (Thermo Electron Corporation). GC-MS was obtained using electron ionization (TRACE 1310 Mainframe MS). The reaction was monitored by TLC (GF-254) under UV light or treated with KMnO_4 followed by heating.

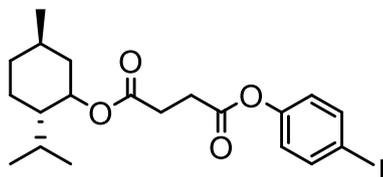
2. Preparation of Substrates



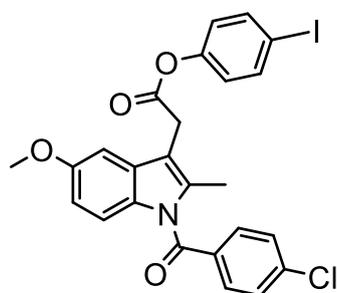
To a 50-mL oven-dried flask containing a magnetic stirring bar, acid (11 mmol, 1.1 equiv), alcohol (10 mmol, 1.0 equiv) in DCM (20 mL), was added DMAP (61 mg, 0.5 mmol, 5.0 mol %) and DCC (2.5 g, 12 mmol, 1.2 equiv) in sequence at 0°C. Then the reaction mixture was stirred for 4-5 hours at room temperature. Upon completion (monitored by TLC), the solvent was evaporated under vacuum after filtering through Celite. The residue was purified by column chromatography on silica gel (Hexanes/EtOAc) to give the pure products.



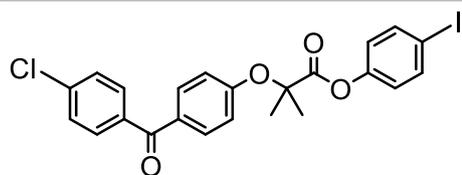
¹H NMR (400 MHz, CDCl₃): δ = 7.02 (d, J=7.88Hz, 1H), 6.70 (d, J=7.33Hz, 1H), 6.64 (s, 1H), 4.22 (t, J=6.4Hz, 2H), 3.96(m,2H), 2.56(td,J=6.8Hz, 2.5Hz, 2H), 2.35(s,3H), 2.21(s,3H), 2.00(t, J=2.6Hz,1H), 1.77(m, 4H), 1.26 (s, 6H);



¹H NMR (400 MHz, CDCl₃): δ = 7.70 (d, J = 8.7Hz, 2H), 6.90(d, J = 8.7Hz, 2H), 4.75 (td, J = 10.8Hz, 4.2Hz, 1H), 2.89 (t, J = 6.4Hz, 2H), 2.74 (t, J = 6.8Hz, 2H), 1.80-1.65(m, 4H), 1.42-1.25(m,4H), 1.15-1.05(m,1H), 0.91(t, J = 6.8Hz, 6H), 0.77(d, J = 7.0Hz, 3H).

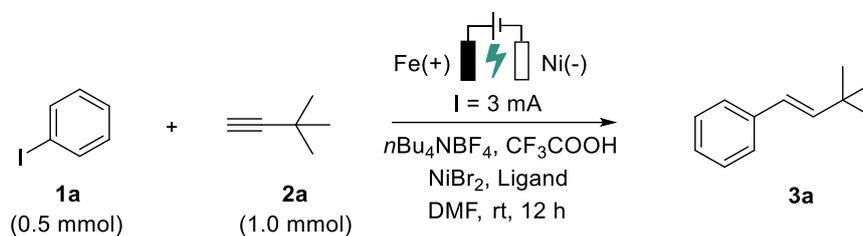


¹H NMR (400 MHz, CDCl₃): δ = 7.73-7.69(m, 4H), 7.52(d, J = 8.5Hz, 2H), 7.02(d, J = 2.2Hz, 1H), 6.89-6.82 (m, 4H), 6.70 (dd, J = 9.0Hz, 2.5Hz, 2H), 3.89(s, 2H), 3.83(s, 3H), 2.45(s, 3H).



$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.78(d, J = 8.8Hz, 2H), 7.71(d, J = 8.5Hz, 2H), 7.67(d, J = 8.8Hz, 2H), 6.97(d, J = 8.8Hz, 2H), 6.75(d, J = 8.8Hz, 2H), 1.82(s, 6H).

3. General Procedure



Equip a 25 mL three-necked flask with nickel plate cathode (10 × 10 × 0.15 mm) and iron plate anode (10 × 10 × 0.15 mm), the distance between which was approximately 2 cm. Then transfer the flask (equipped with electrode) into glovebox. Under N_2 protection, add the substrate iodobenzene **1a** (0.5 mmol) and 3,3-dimethylbut-1-yne **2a** (1.0 mmol, 2.0 equiv.), electrolyte $n\text{Bu}_4\text{NBF}_4$ (32.9 mg, 0.10 mmol, 0.02 M), NiBr_2 (0.1 mmol, 20% mmol) and 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (0.15 mmol, 30% mmol) followed by 5 mL solvent (DMF) and CF_3COOH (57.0 mg, 0.5 mmol, 1.0 equiv.). Solvent require anhydrous treatment. To minimize the evaporation, the system was closed with a rubber plug (**Fig. S1**). During the reaction, the constant current is 3 mA. Upon completion, the reaction mixture was poured into water and extracted with EtOAc for three times. The combined organic layer was dried over anhydrous Na_2SO_4 , and the solvent was then removed under reduced pressure. The resulting mixture was purified by column chromatography on silica gel (eluted with PE) to afford the desired product **3a** (a colorless transparent oily substance).

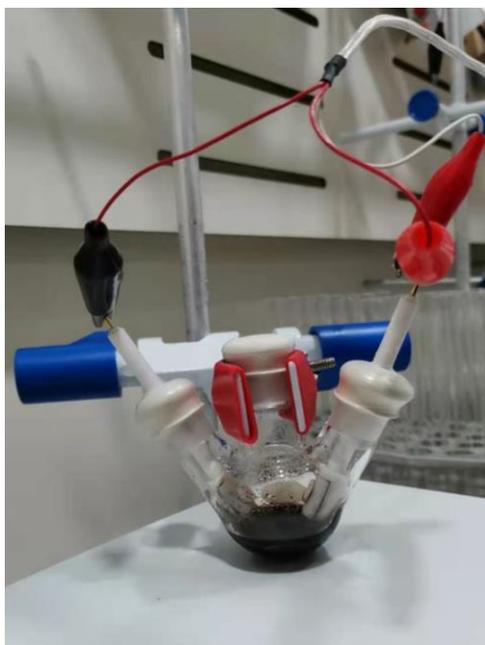
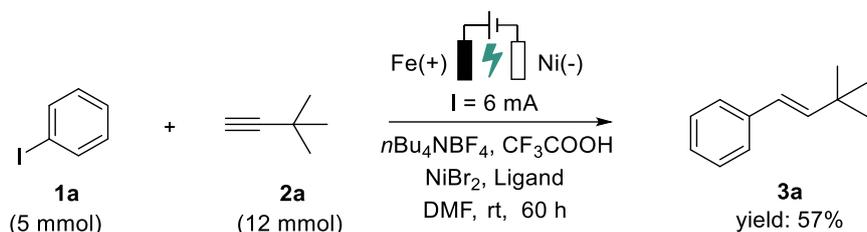


Fig. S1 Reaction setup

4. Gram-Scale Experiment (5.0 mmol scale)



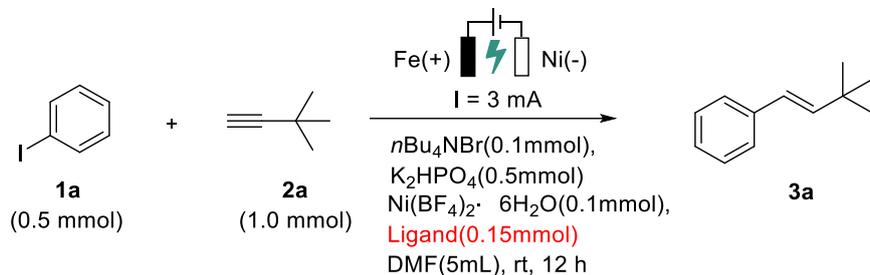
Equip a 100 mL solvent seal bottle with nickel plate cathode (5 cm × 5 cm × 0.2 mm) and iron plate anode (5 cm × 5 cm × 0.2 mm), the distance between which was approximately 2 cm. Then transfer the bottle (equipped with electrode) into glovebox. Under N_2 protection ,add the substrate iodobenzene **1a** (5 mmol) and 3,3-dimethylbut-1-yne **2a** (12 mmol, 2.1 equiv), electrolyte $n\text{Bu}_4\text{NBF}_4$ (0.329 g, 1 mmol, 0.02 M), NiBr_2 (1mmol, 20%mmol) and 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (1.5 mmol, 30%mmol) followed by 50 mL solvent (DMF) and CF_3COOH (5 mmol, 1.0 equiv.). Solvent require anhydro`us treatment. During the reaction, the constant current is 6 mA. Upon completion, the reaction mixture was poured into water and extracted with EtOAc for three times. The combined organic layer was dried over anhydrous Na_2SO_4 , and the solvent was then removed under reduced pressure. The resulting mixture was purified by column chromatography on silica gel (eluted with PE) to afford the desired product **3a** (yield: 57%,a colorless transparent oily substance).



Fig. S2 & S3 Gram-scale reaction setup

5. Optimization of the Reaction Conditions

Table S1. Optimization of the Ligand



Entry	Ligand	Yield(%)
1	L1	24
2	L2	12
3	L3	N.D.
4	L4	N.D.
5	L5	N.D.
6	L6	N.D.
7	L7	trace
8	L8	16
9	L9	N.D.
10	L10	N.D.
11	L11	N.D.
12	L12	N.D.
13	L13	N.D.
14	L14	N.D.
15	L15	N.D.
16	L16	46
17	L17	N.D.
18	L18	N.D.
19	L19	N.D.
20	L20	trace

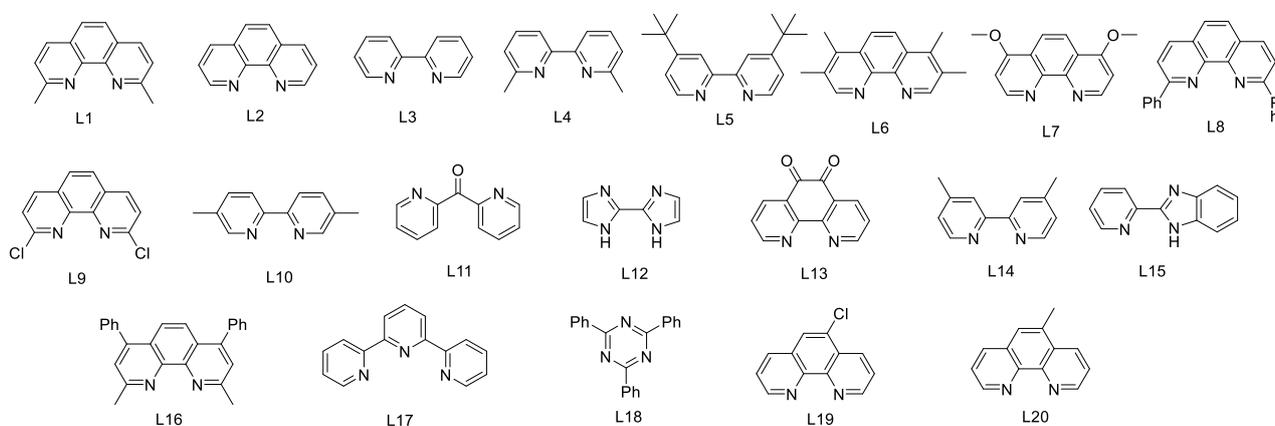
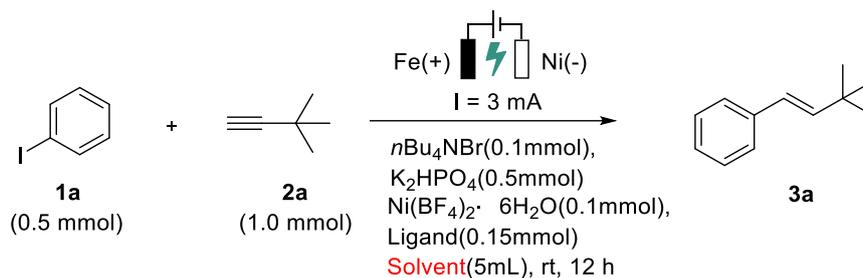
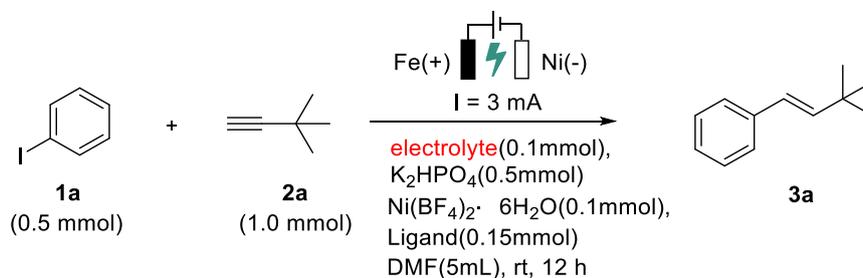
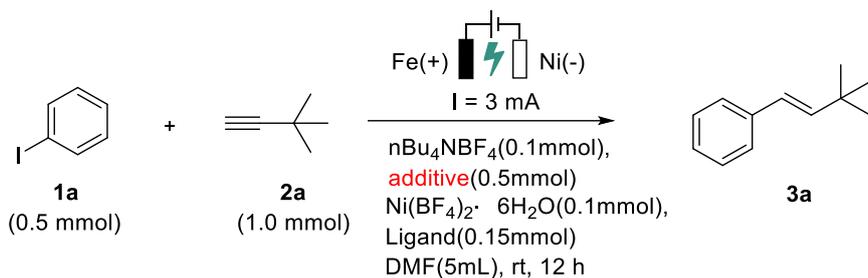


Table S2. Optimization of the solvent

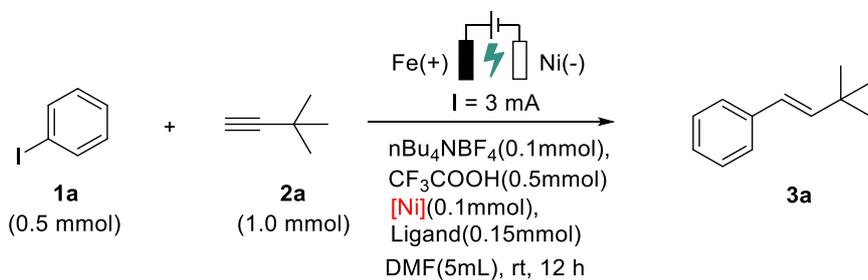
Entry	solvent	Yield(%)
1	DMF	46
2	DMA	11
3	MeOH	N.D.
4	MeCN	N.D.
5	NMP	N.D.
6	THF	N.D.
7	1,4-dioxane	N.D.
8	EtOH	N.D.

Table S3. Optimization of the electrolyte type

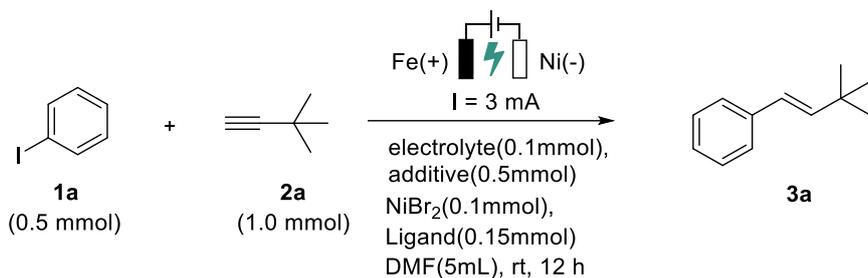
Entry	electrolyte	Yield(%)
1	$n\text{Bu}_4\text{NBr}$	46
2	$n\text{Bu}_4\text{NI}$	14
3	$n\text{Bu}_4\text{NPF}_6$	trace
4	$n\text{Bu}_4\text{NCl}$	16
5	$n\text{Bu}_4\text{NClO}_4$	15
6	$n\text{Bu}_4\text{NPO}_4$	trace
7	$n\text{Bu}_4\text{NBF}_4$	57
8	$n\text{Bu}_4\text{NSO}_4$	17

Table S4. Optimization of the additive

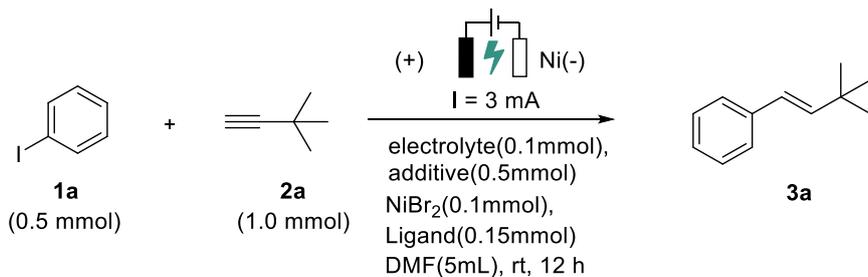
Entry	additive	Yield(%)
1	K ₂ HPO ₄	57
2	NH ₄ Cl	22
3	KH ₂ PO ₄	12
4	PhCOOH	trace
5	CF₃COOH	62
6	CH ₃ OH	trace

Table S5. Optimization of the quantity of electrolyte

Entry	[Ni]	Yield(%)
1	Ni(BF ₄) ₂ ·6H ₂ O	62
2	NiBr₂	71
3	NiBr ₂ ·glyme	38
4	NiI ₂	12
5	Ni(acac) ₂	N.D.
6	NiCl ₂	25
7	NiCl ₂ ·glyme	8
8	Ni(PPh ₃) ₂ Cl ₂	N.D.
9	Ni(PPh ₃) ₂ Br ₂	N.D.
10	Ni(Py) ₄ Cl ₂	N.D.

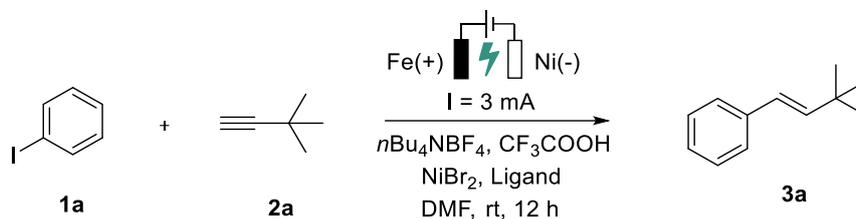
Table S6. Optimization of the current intensity

Entry	Current intensity	Yield(%)
1	1	trace
2	2	43
3	3	71
4	4	42
5	5	33

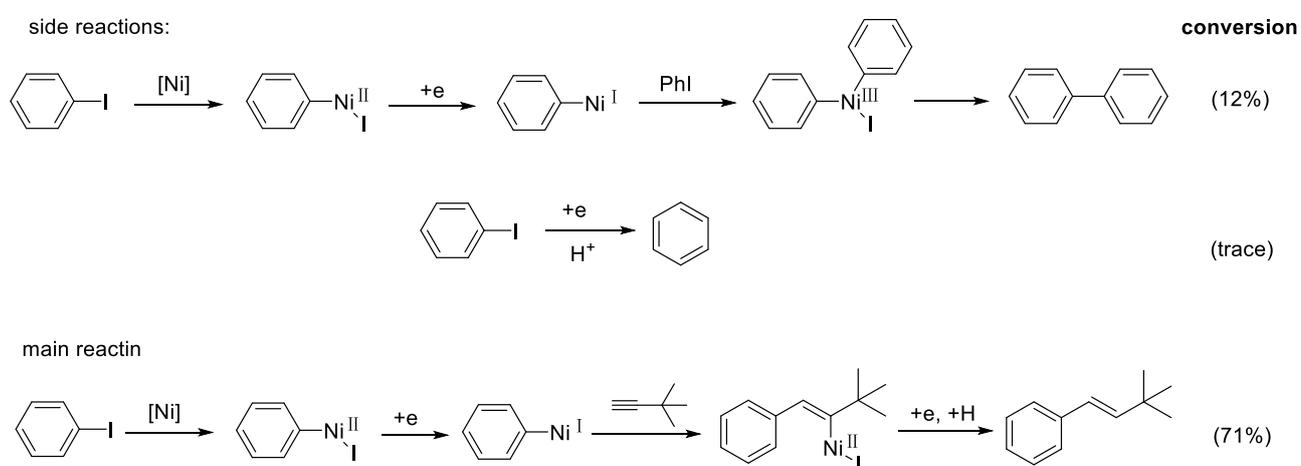
Table S7. Optimization of the anode

Entry	anode	Yield(%)
1	Cu	trace
2	Zn	21
3	Fe	71
4	Mg	trace

6. Target reaction and side reaction



In my reaction system, there is one main reaction and two side reactions.



After the separation of the reaction system, it was found that the main product was trans-olefin, and 12% of iodobenzene was self-coupled, while a small amount of iodobenzene was directly reduced by deiodination in electrochemical environment.

7. Mechanistic Studies

Cyclic voltammetry experiments

The cyclic voltammogram was collected with a CHI 760E Potentiostat. Cyclic voltammetry was performed in a three electrode cell connected to a schlenk line under nitrogen at room temperature. The working electrode was a glassy carbon electrode, the counter electrode a platinum sheet. The reference was saturated calomel electrode. DMF (10 mL) containing 1.0 mmol $n\text{Bu}_4\text{NBF}_4$ was poured into the electrochemical cell in all experiments and the sample was prepared with 0.2 mmol of target molecule. The scan rate is 0.1 V/s. The peak potentials vs. SCE for used. Maximum potential (E_p) of each compound was obtained using Origin.

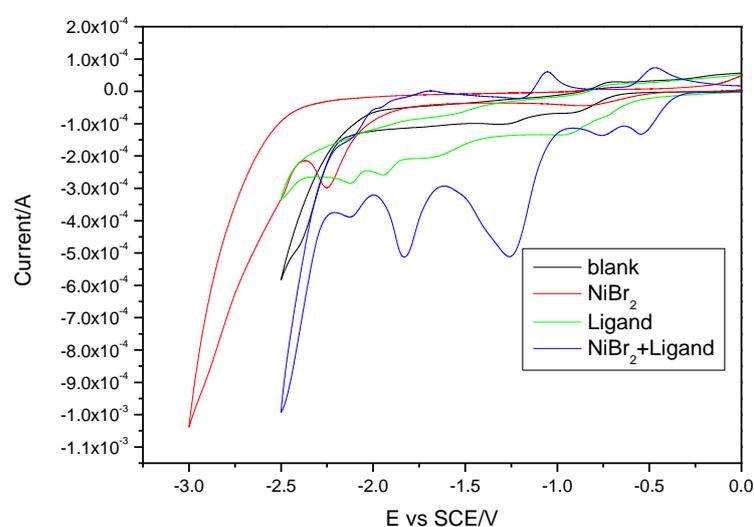


Fig. S4 The CV plot of $n\text{Bu}_4\text{NBF}_4$, NiBr_2 , Ligand, system of NiBr_2 and Ligand

The E_p values of NiBr_2 was determined as -2.25V, The E_p values of Ligand was determined as -1.95V, -2.12V, The E_p values of system of NiBr_2 and Ligand was determined as -1.24V, -1.82V(**Fig. S4**), and electrolyte $n\text{Bu}_4\text{NBF}_4$ was determined as no obvious peak.

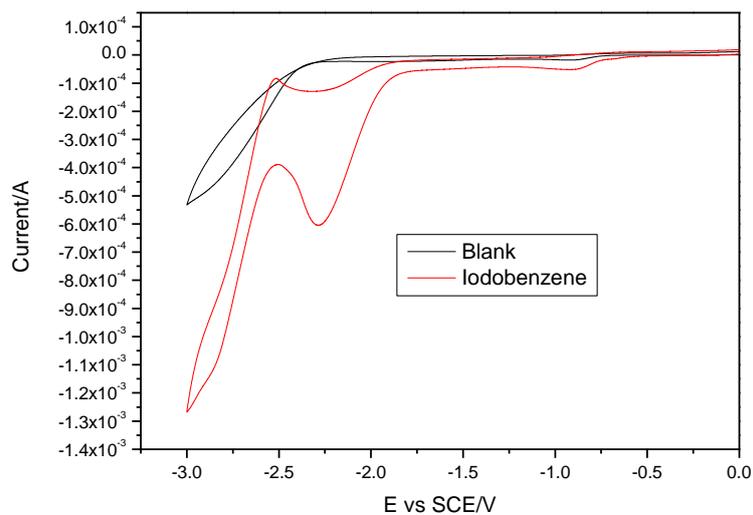


Fig. S5 The CV plot of iodobenzene

The E_p values of system of Iodobenzene was determined as -2.27V (**Fig. S5**), and electrolyte $n\text{Bu}_4\text{NBF}_4$ was determined as no obvious peak.

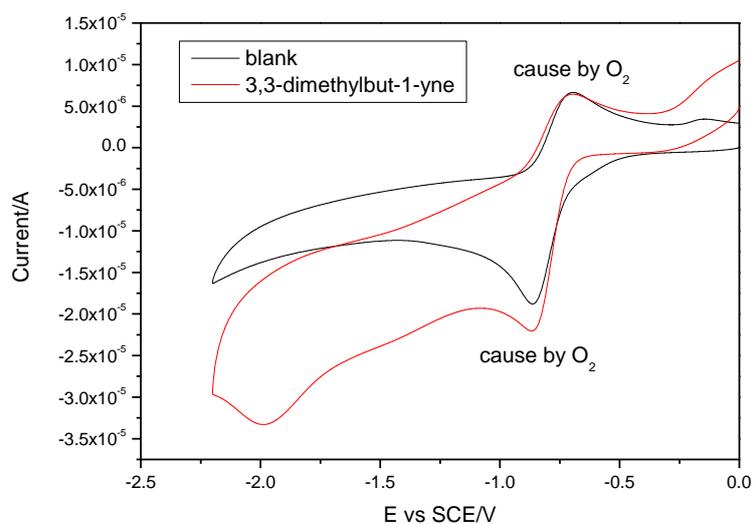


Fig. S6 The CV plot of iodobenzene, 3,3-dimethylbut-1-yne

The E_p values of system of 3,3-dimethylbut-1-yne was determined as -1.98V (**Fig. S6**).

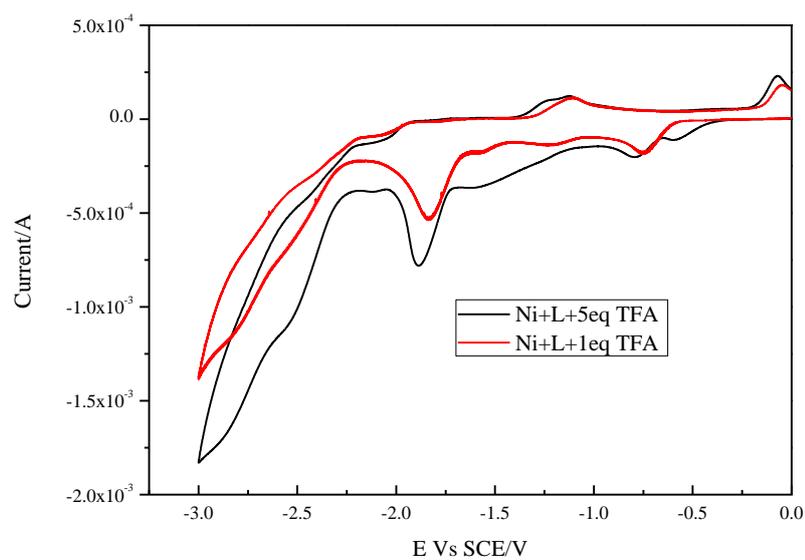


Fig. S7 The CV plot of Nickel complex with 1eq TFA or 5eq TFA

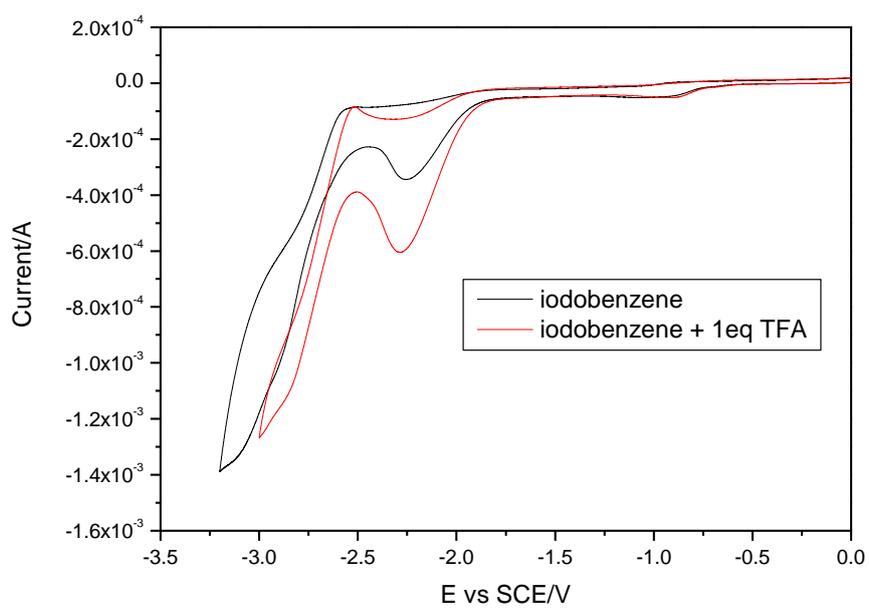
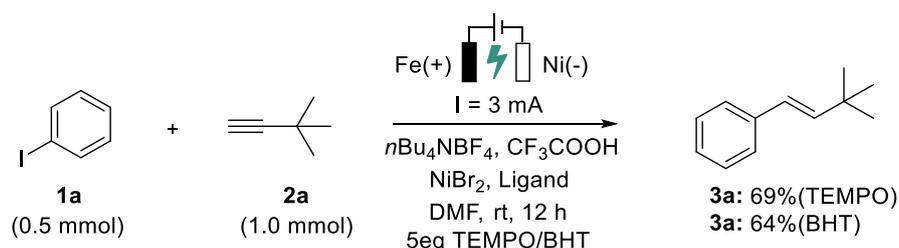


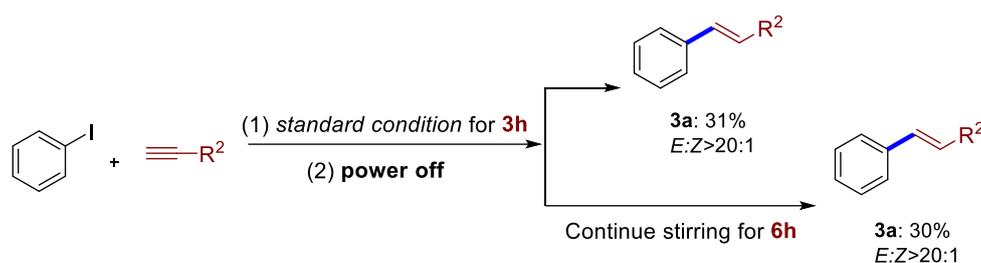
Fig. S8 The CV plot of iodobenzene with 1eq TFA.

Free radical capture experiment



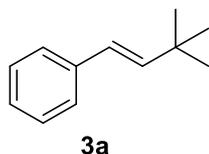
Equip a 25 mL three-necked flask with nickel plate cathode (10 × 10 × 0.15 mm) and iron plate anode (10 × 10 × 0.15 mm), the distance between which was approximately 2 cm. Then transfer the flask (equipped with electrode) into glovebox. Under N₂ protection, add the substrate iodobenzene **1a** (0.5 mmol) and 3,3-dimethylbut-1-yne **2a** (1.0 mmol, 2.0 equiv.), electrolyte *n*Bu₄NBF₄ (32.9 mg, 0.10 mmol, 0.02 M), NiBr₂ (0.1 mmol, 20% mmol) and 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (0.15 mmol, 30% mmol), TEMPO (0.39 g, 2.5 mmol, 5.0 eq) or BHT (0.55 g, 2.5 mmol, 5 eq) followed by 5 mL solvent (DMF) and CF₃COOH (57.0 mg, 0.5 mmol, 1.0 equiv.). Solvent requires anhydrous treatment. To minimize the evaporation, the system was closed with a rubber plug (**Fig. S1**). During the reaction, the constant current is 3 mA. Upon completion, the reaction mixture was poured into water and extracted with EtOAc for three times. The combined organic layer was dried over anhydrous Na₂SO₄, and the solvent was then removed under reduced pressure. The resulting mixture was purified by column chromatography on silica gel (eluted with PE) to afford the desired product **3a** (a colorless transparent oily substance).

On-off electrical experiment



Equip two 25 mL three-necked flasks with nickel plate cathode (10 × 10 × 0.15 mm) and iron plate anode (10 × 10 × 0.15 mm), the distance between which was approximately 2 cm. Then transfer the flask (equipped with electrode) into glovebox. Under N₂ protection, add the substrate iodobenzene **1a** (0.5 mmol) and 3,3-dimethylbut-1-yne **2a** (1.0 mmol, 2.0 equiv.), electrolyte *n*Bu₄NBF₄ (32.9 mg, 0.10 mmol, 0.02 M), NiBr₂ (0.1 mmol, 20% mmol) and 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (0.15 mmol, 30% mmol), followed by 5 mL solvent (DMF) and CF₃COOH (57.0 mg, 0.5 mmol, 1.0 equiv.). Solvent requires anhydrous treatment. To minimize the evaporation, the system was closed with a rubber plug (**Fig. S1**). During the reaction, the constant current is 3 mA. After three hours, the reaction was stopped, one of the reactions was post-treated and separated, and the other reaction was stirred for another six hours, and then post-treated and separated.

8. Spectroscopic Data of Products



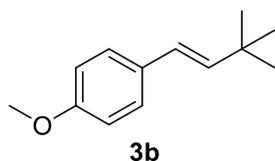
(E)-1-(3,3-dimethylbut-1-en-1-yl)benzene (3a)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether) afforded 56.8 mg (71% yield) of the title compound **3a**.

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.42-7.37 (m, 2H), 7.36-7.28 (m, 2H), 7.25-7.19 (m, 1H), 6.32 (d, $J = 16.3$ Hz, 1H), 6.27 (dd, $J = 16.1$ Hz, 0.7Hz, 1H), 1.16 (s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 141.9, 138.0, 128.5, 126.7, 126.0, 124.5, 33.4, 29.5.

In accordance with literature¹.



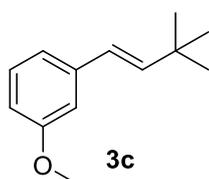
(E)-1-(3,3-dimethylbut-1-en-1-yl)-4-methoxybenzene (3b)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (20:1)) afforded 59.8 mg (63% yield) of the title compound **3b**.

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.32 (d, $J = 9.4$ Hz, 2H), 6.86 (d, $J = 9.6$ Hz, 2H), 6.26 (d, $J = 15.5$ Hz, 1H), 6.13 (d, $J = 15.1$ Hz, 1H), 3.84 (s, 3H), 1.15 (s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 158.6, 139.8, 130.8, 127.1, 123.8, 113.9, 55.3, 33.3, 29.7.

In accordance with literature².



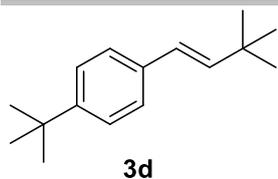
(E)-1-(3,3-dimethylbut-1-en-1-yl)-3-methoxybenzene (3c)

Lime-yellow oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (20:1)) afforded 72.2 mg (76% yield) of the title compound **3c**.

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.25 (t, 1H, $J = 7.7$ Hz), 7.01 (d, 1H, $J = 7.7$ Hz), 6.95 (t, 1H, $J = 2.0$ Hz), 6.79 (dd, 1H, $J = 8.2$ Hz, 2.2 Hz), 6.33 (d, 1H, $J = 15.8$ Hz), 6.28 (d, 1H, $J = 15.8$ Hz), 3.88 (s, 3H), 1.21 (s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 159.8, 142.2, 139.6, 129.5, 124.5, 118.7, 112.5, 111.3, 55.3, 33.4, 29.6.

In accordance with literature³.



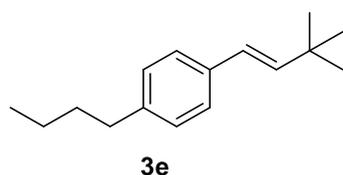
(E)-1-(tert-butyl)-4-(3,3-dimethylbut-1-en-1-yl)benzene (3d)

Yellow oil, purification by flash chromatography on silica gel (petroleum ether) afforded 61.5 mg (57% yield) of the title compound **3d**.

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.38 - 7.32 (m, 4H), 6.35 (d, $J = 16.2$ Hz, 1H), 6.27 (d, $J = 16.2$ Hz, 1H), 1.35 (s, 9H), 1.16 (s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 149.8, 141.2, 135.3, 125.7, 125.4, 124.2, 34.5, 33.3, 31.3, 29.6.

In accordance with literature⁴.



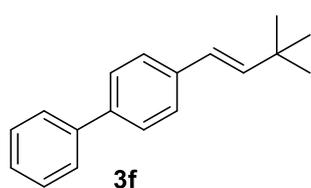
(E)-1-butyl-4-(3,3-dimethylbut-1-en-1-yl)benzene (3e)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether) afforded 62.6 mg (58% yield) of the title compound **3e**.

$^1\text{H NMR}$ (400 MHz, CDCl_3) $\delta =$ 7.29 (d, $J = 7.7$ Hz, 2H), 7.11 (d, $J = 7.6$ Hz, 2H) , 6.28 (d, $J = 16.3$ Hz, 1H), 6.21 (t, $J = 16.3$ Hz, 1H), 2.58 (t, $J = 7.4$ Hz, 2H), 1.59 (m, 2H), 1.32 (m, 2H), 1.11(s, 9H), 0.91(t, $J = 7.4$ Hz,3H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta =$ 140.9, 135.5, 128.6, 125.9, 124.4, 119.5, 35.4, 33.7, 31.7, 29.7, 22.4,14.0;

HRMS: calcd for $\text{C}_{16}\text{H}_{24}$ $[\text{M}+\text{H}]^+$: 217.1951, found: 217.1947.



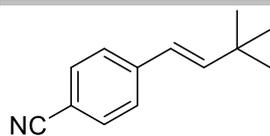
(E)-4-(3,3-dimethylbut-1-en-1-yl)-1,1'-biphenyl (3f)

White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 49.6 mg (42% yield) of the title compound **3f**.

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.60 (d, $J = 7.6$ Hz, 2H), 7.54 (d, $J = 8.3$ Hz, 2H), 7.43 (m,, 4H), 7.33 (t, $J = 7.5$ Hz, 1H), 6.35(d, $J = 16.5$ Hz, 1H), 6.30 (d, $J = 16.5$ Hz, 1H), 1.14(s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 142.1, 141.0, 139.5, 137.2, 128.8, 127.2, 127.1, 127.0, 126.5, 124.1, 33.5, 30.0.

In accordance with literature⁴.



3g

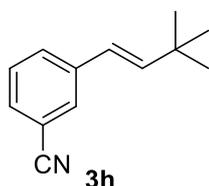
(E)-4-(3,3-dimethylbut-1-en-1-yl)benzonitrile (3g)

Yellow oil, purification by preparative thin layer chromatography (petroleum ether/ EtOAc (10:1)) afforded 59.2 mg (64% yield) of the title compound **3g**.

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.58 (d, $J = 8.4$ Hz, 2H), 7.44 (d, $J = 8.4$ Hz, 2H), 6.42 (d, $J = 15.4$ Hz, 1H), 6.32 (d, $J = 15.4$ Hz, 1H), 1.13 (s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 145.9, 142.7, 132.3, 126.5, 123.5, 119.2, 109.9, 33.8, 29.3.

In accordance with literature⁴.



3h

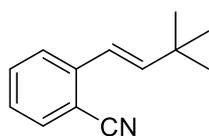
(E)-3-(3,3-dimethylbut-1-en-1-yl)benzonitrile (3h)

Yellow oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (10:1)) afforded 46.2 mg (50% yield) of the title compound **3h**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.64 (s, 1H), 7.56 (d, $J = 7.7$ Hz, 1H), 7.47 (d, $J = 7.8$ Hz, 1H), 7.39 (t, $J = 7.7$ Hz, 1H), 6.33 (d, $J = 16.2$ Hz, 1H), 6.27 (dd, $J = 16.2$ Hz, 1H), 1.13(s,9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta = 144.7, 139.4, 130.4, 130.1, 129.6, 129.3, 122.8, 119.0, 112.6, 33.7, 29.4$.

HRMS: calcd for $\text{C}_{13}\text{H}_{15}\text{N}$ $[\text{M}+\text{H}]^+$: 186.1277, found: 186.1277.



3i

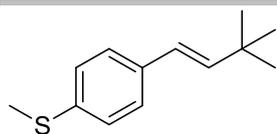
(E)-2-(3,3-dimethylbut-1-en-1-yl)benzonitrile (3i)

Yellow oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (10:1)) afforded 34.2 mg (37% yield) of the title compound **3i**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): $\delta = 7.65$ (t, $J = 8.4$ Hz, 2H), 7.55 (dd, $J = 7.6$ Hz, 1H), 7.30 (m, 1H), 6.70 (d, $J = 16.1$ Hz, 1H), 6.48 (d, $J = 16.1$ Hz, 1H), 1.20(s,9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta = 147.3, 141.5, 133.0, 132.6, 128.1, 126.9, 125.4, 121.0, 110.8, 34.0, 29.4$;

HRMS: calcd for $\text{C}_{13}\text{H}_{15}\text{N}$ $[\text{M}+\text{H}]^+$: 186.1277, found: 186.1271.



3j

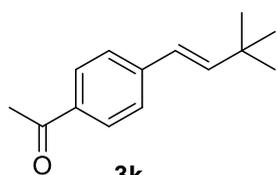
(E)-4-(3,3-dimethylbut-1-en-1-yl)phenyl(methyl)sulfane (3j)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (20:1)) afforded 52.5 mg (51% yield) of the title compound **3j**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.33 (d, $J = 8.2$ Hz, 2H), 7.24 (d, $J = 8.1$ Hz, 2H), 6.31 (d, $J = 16.5$ Hz, 1H), 6.26 (d, $J = 16.1$ Hz, 1H), 2.52 (s, 3H), 1.15 (s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 141.5, 136.4, 135.2, 126.9, 126.4, 123.9, 33.4, 29.6, 16.2.

In accordance with literature⁴.



3k

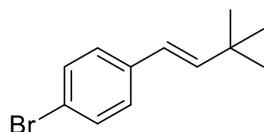
(E)-1-(4-(3,3-dimethylbut-1-en-1-yl)phenyl)ethan-1-one (3k)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (20:1)) afforded 38.4 mg (38% yield) of the title compound **3k**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.93 (d, $J = 8.6$ Hz, 2H), 7.47 (d, $J = 8.4$ Hz, 2H), 6.46 (d, $J = 16.4$ Hz, 1H), 6.39 (d, $J = 16.2$ Hz, 1H), 2.63 (s, 3H), 1.18 (s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta = 197.7, 145.0, 142.9, 135.4, 128.8, 126.1, 123.9, 33.7, 29.4, 26.6$.

In accordance with literature⁵.



3l

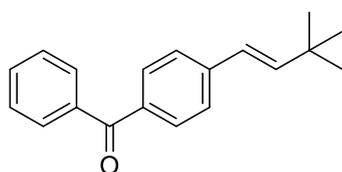
(E)-1-bromo-4-(3,3-dimethylbut-1-en-1-yl)benzene (3l)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether) afforded 57.1 mg (48% yield) of the title compound **3l**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): $\delta = 7.43$ (d, $J = 8.4$ Hz, 2H), 7.25 (d, $J = 8.4$ Hz, 2H), 6.26 (s, 2H), 1.15 (s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta = 142.7, 137.0, 131.5, 127.6, 123.5, 120.3, 33.5, 29.5$.

In accordance with literature⁷.



3m

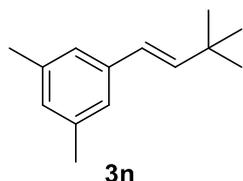
(E)-4-(3,3-dimethylbut-1-en-1-yl)phenyl(phenyl)methanone (3m)

White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 58.1mg (44% yield) of the title compound **3m**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.81 (m, 4H), 7.62 (m, 1H), 7.51 (m, 4H), 6.46 (d, J = 16.2 Hz, 1H), 6.40 (d, J = 16.2 Hz, 1H), 1.18 (s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 196.4, 144.9, 142.4, 138.0, 135.7, 132.3, 130.7, 130.0, 128.3, 125.9, 124.0, 33.8, 29.5.

HRMS: calcd for $\text{C}_{19}\text{H}_{20}\text{O}$ $[\text{M}+\text{H}]^+$:265.1587, found:265.1581 .

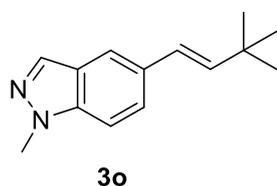
**(E)-1-(3,3-dimethylbut-1-en-1-yl)-3,5-dimethylbenzene (3n)**

Colourless oil, purification by flash chromatography on silica gel (petroleum ether) afforded 67.7 mg (72% yield) of the title compound **3n**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.03 (s, 2H), 6.88 (s, 1H), 6.28 (s, 2H), 2.34 (s, 6H), 1.15 (s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 141.5, 138.0, 137.9, 128.5, 124.6, 123.9, 33.3, 29.6, 21.3.

In accordance with literature.⁶

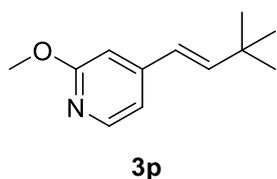
**(E)-5-(3,3-dimethylbut-1-en-1-yl)-1-methyl-1H-indazole (3o)**

Yellow solid, purification by flash chromatography on silica gel (petroleum ether/EtOAc (10:1)) afforded 50.3 mg (47% yield) of the title compound **3o**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.96 (s, 1H), 7.66 (s, 1H), 7.54 (dd, J = 8.7, 1.3 Hz, 1H), 7.35 (d, J = 8.8 Hz, 1H), 6.45 (d, J = 16.1 Hz, 1H), 6.27 (d, J = 16.1 Hz, 1H), 4.09 (s, 3H), 1.18 (s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 140.6, 139.4, 132.9, 131.0, 124.8, 124.6, 124.5, 118.4, 109.0, 35.7, 33.4, 29.8

HRMS: calcd for $\text{C}_{14}\text{H}_{18}\text{N}_2$ $[\text{M}+\text{H}]^+$: 215.1543, found: 215.1544.

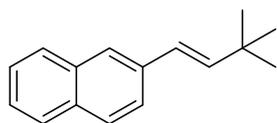
**(E)-4-(3,3-dimethylbut-1-en-1-yl)-2-methoxypyridine (3p)**

Yellow oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (5:1)) afforded 63.9 mg (67% yield) of the title compound **3p**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 8.08 (d, J = 5.4Hz, 1H), 6.90(dd, J = 1.3Hz, 5.4Hz, 1H), 6.67 (s, 1H), 6.46 (d, J = 16.2 Hz, 1H), 6.22 (d, J = 16.2 Hz, 1H), 3.95(s, 3H), 1.15 (s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 164.9, 148.4, 146.7, 146.4, 122.7, 114.2, 107.8, 55.5, 33.7, 29.3.

HRMS: calcd for $\text{C}_{12}\text{H}_{17}\text{NO}$ $[\text{M}+\text{H}]^+$: 192.1383, found: 192.1385.



3q

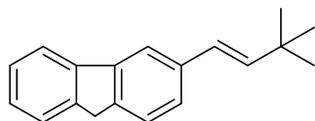
(E)-2-(3,3-dimethylbut-1-en-1-yl)naphthalene (**3q**)

White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 69.3 mg (61% yield) of the title compound **3q**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.82 (t, J = 7.4 Hz, 3H), 7.74 (s, 1H), 7.65 (dd, J = 8.5 Hz, 1.8Hz, 1H), 7.49 - 7.36 (m, 2H), 6.48 (d, J = 16.2Hz, 1H), 6.39 (d, J = 16.2Hz, 1H), 1.16 (s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 142.2, 135.4, 133.6, 132.5, 127.9, 127.7, 127.5, 126.0, 125.5, 125.3, 124.6, 123.5, 33.4, 29.6.

In accordance with literature².



3r

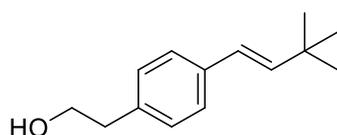
(E)-3-(3,3-dimethylbut-1-en-1-yl)-9H-fluorene (**3r**)

White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 81.8 mg (66% yield) of the title compound **3r**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.79 (d, J = 7.4 Hz, 1H), 7.74 (d, J = 7.4 Hz, 1H), 7.61 (m, 1H), 7.56 (d, J = 7.8Hz, 1H), 7.40 (m, 2H), 7.33 (m, 1H), 6.43 (d, J = 16.1 Hz, 1H), 6.36 (d, J = 16.1 Hz, 1H), 3.92 (s, 2H), 1.18(s,9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 143.7, 143.4, 141.7, 141.5, 140.6, 136.8, 126.8, 126.4, 125.2, 125.1, 124.9, 122.4,120.0, 119.8, 36.9, 33.5, 29.7.

HRMS: calcd for $\text{C}_{19}\text{H}_{20}$ $[\text{M}+\text{H}]^+$:249.1638, found:249.1638.



3s

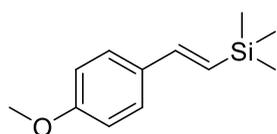
(E)-2-(4-(3,3-dimethylbut-1-en-1-yl)phenyl)ethan-1-ol (**3s**)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (5:1)) afforded 32.6 mg (32% yield) of the title compound **3s**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.35 (d, J = 7.9 Hz, 2H), 7.20 (d, J = 1.8 Hz, 2H), 6.32(d, J = 16.3Hz, 1H), 6.26(d, J = 16.3Hz, 1H), 3.88 (t, J = 6.2Hz, 2H), 2.88 (t, J = 6.8Hz, 2H), 1.15 (s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 141.7, 136.9, 136.5, 129.2, 126.3, 124.1, 36.8, 38.9, 33.4, 29.7.

HRMS: calcd for $\text{C}_{14}\text{H}_{20}\text{O}$ $[\text{M}+\text{H}]^+$: 205.1587, found: 205.1589.



3t

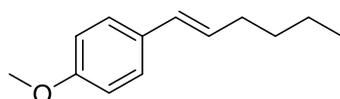
(E)-(4-methoxystyryl)trimethylsilane (**3t**)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (20:1)) afforded 64.9mg (63% yield) of the title compound **3t**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.37 (d, J = 8.7 Hz, 2H), 6.86 (d, J = 8.7Hz, 2H), 6.82(d, J = 19.8Hz, 1H), 6.32 (d, J = 20.0 Hz, 1H), 3.81 (s, 3H), 0.14(s, 9H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ =159.6, 143.0, 131.4, 127.6, 126.7, 114.0, 55.4, -1.0;

In accordance with literature⁸.



3u

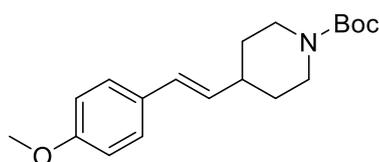
(E)-1-(hex-1-en-1-yl)-4-methoxybenzene (**3u**)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (20:1)) afforded 64.6 mg (68% yield) of the title compound **3u**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ =7.28 (d, J = 9.1 Hz, 2H), 6.81 (d, J = 8.7 Hz, 2H), 6.30 (d, J = 15.8 Hz, 1H), 6.08 (dt, J = 15.8, 6.9 Hz, 1H), 3.80 (s, 3H), 2.17 (m, 2H), 1.44–1.31 (m, 4H), 0.90 (td, J = 7.2 Hz, 2.7Hz, 3H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 158.6, 130.9, 129.9, 129.1, 127.0, 113.9, 55.3, 32.7, 31.7, 22.3, 14.0;

In accordance with literature⁹.



3v

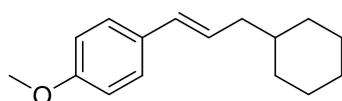
tert-butyl (E)-4-(4-methoxystyryl)piperidine-1-carboxylate (**3v**)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (10:1)) afforded 96.6 mg (61% yield) of the title compound **3v**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.31 (d, J = 8.2 Hz, 2H), 6.87 (d, J = 8.3 Hz, 2H), 6.36 (d, J = 16.0 Hz, 1H), 6.02 (dd, J = 16.0 Hz, 7.0 Hz, 1H), 4.15 (d, J = 12.0 Hz, 2H), 3.83 (s, 3H), 2.80 (t, J = 12.6 Hz, 2H), 2.34-2.22 (m, 1H), 1.77 (d, J = 12.6 Hz, 2H), 1.50 (s, 9H), 1.45-1.35 (m, 2H)

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 158.9, 155.0, 132.3, 130.3, 127.8, 127.2, 113.9, 79.4, 55.4, 39.4, 32.0, 28.5;

HRMS: calcd for $\text{C}_{19}\text{H}_{27}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 318.2064, found: 318.2069.



3w

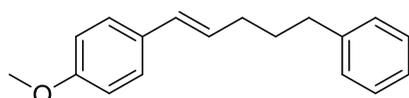
(E)-1-(3-cyclohexylprop-1-en-1-yl)-4-methoxybenzene (**3w**)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (20:1)) afforded 66.7 mg (58 % yield) of the title compound **3w**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.32 (d, J = 8.9 Hz, 2H), 6.88 (d, J = 8.6 Hz, 2H), 6.34 (d, J = 16.0 Hz, 1H), 6.05-6.17 (m, 1H), 3.84 (s, 3H), 2.12 (t, J = 6.8 Hz, 2H), 1.84-1.75 (m, 4H), 1.44-1.36 (m, 1H), 1.34-1.20 (m, 4H), 1.05-0.95 (m, 2H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 158.6, 130.9, 130.0, 127.7, 127.0, 113.9, 55.4, 41.1, 38.3, 33.3, 26.6, 26.4;

In accordance with literature¹⁰.



3x

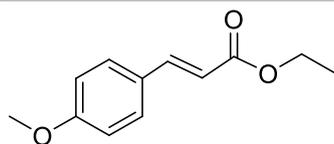
(E)-1-methoxy-4-(5-phenylpent-1-en-1-yl)benzene (**3x**)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (20:1)) afforded 93.2 mg (74 % yield) of the title compound **3x**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.34 - 7.24 (m, 4H), 7.22-7.16 (m, 3H), 6.84 (d, J = 8.4 Hz, 2H), 6.35 (d, J = 16.1 Hz, 1H), 6.09 (dt, J = 16.1, 6.7 Hz, 1H), 3.80 (s, 3H), 2.67 (t, J = 7.5 Hz, 2H), 2.23 (q, J = 7.0 Hz, 2H), 1.80 (p, J = 7.5 Hz, 2H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 158.7, 142.5, 130.7, 129.6, 128.6, 128.5, 128.4, 127.1, 125.8, 114.0, 55.4, 35.5, 32.6, 31.3;

In accordance with literature¹¹.



3y

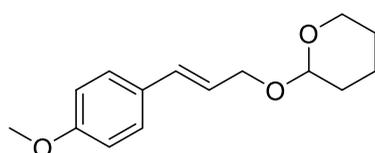
ethyl (E)-3-(4-methoxyphenyl)acrylate (3y)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (20:1)) afforded 73.1 mg (71% yield) of the title compound **3y**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.65 (d, J = 15.9 Hz, 1H), 7.51 – 7.45 (m, 2H), 6.92 – 6.87 (m, 2H), 6.30 (d, J = 16.0 Hz, 1H), 4.25 (q, J = 7.1 Hz, 2H), 3.83 (s, 3H), 1.33 (t, J = 7.2 Hz, 3H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 167.5, 161.4, 144.4, 129.8, 127.2, 115.8, 114.4, 60.5, 55.5, 14.3;

In accordance with literature¹².



3z

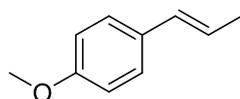
(E)-2-((3-(4-methoxyphenyl)allyl)oxy)tetrahydro-2H-pyran (3z)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (10:1)) afforded 52.8 mg (42% yield) of the title compound **3z**.

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ = 7.34 (d, J = 7.9 Hz, 2H), 6.85 (d, J = 7.9 Hz, 2H), 6.57 (d, J = 15.8 Hz, 1H), 6.57 (dt, J = 15.8 Hz, 6.5 Hz, 1H), 4.71 (t, J = 4.0 Hz, 1H), 4.39 (dd, J = 12.6 Hz, 5.7 Hz, 1H), 4.14 (dd, J = 12.4 Hz, 6.8 Hz, 1H), 3.95–3.86 (m, 1H), 3.81 (s, 3H), 3.54 (m, 1H), 1.85–1.55 (m, 6H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 159.3, 132.2, 129.6, 127.8, 123.7, 114.0, 97.8, 67.9, 62.3, 55.3, 30.7, 25.5, 19.6;

In accordance with literature¹³.



3aa

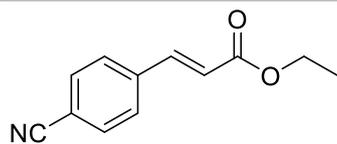
(E)-1-methoxy-4-(prop-1-en-1-yl)benzene (3aa)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (20:1)) afforded 52.5 mg (71% yield) of the title compound **3aa**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.25 (d, J = 8.6 Hz, 2H), 6.83 (d, J = 8.6 Hz, 2H), 6.34 (d, J = 15.8 Hz, 1H), 6.09 (dq, J = 15.7, 6.6 Hz, 1H), 3.77 (s, 3H), 1.85 (dd, J = 6.6 Hz, 1.4 Hz, 3H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 158.6, 130.9, 130.4, 127.0, 123.6, 114.0, 55.4, 18.5;

In accordance with literature¹⁴.



3bb

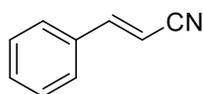
ethyl (E)-3-(4-cyanophenyl)acrylate (3bb)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (10:1)) afforded 72.4 mg (72% yield) of the title compound **3bb**.

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ = 7.74-7.60 (m, 5H), 6.54 (d, J = 16.1Hz, 1H), 4.31 (q, J = 7.0Hz, 2H), 1.38 (t, J = 7.0Hz, 3H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 166.2, 144.2, 138.8, 132.7, 128.4, 121.9, 118.4, 113.4, 61.0, 14.3;

In accordance with literature¹⁵.



3cc

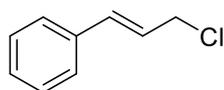
cinnamitrile (3cc)

Yellow oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (20:1)) afforded 49.0 mg (76% yield) of the title compound **3cc**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.46-7.37 (m, 6H), 5.87 (d, J = 16.6Hz, 1H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 150.7, 133.6, 131.3, 129.2, 127.5, 118.3, 96.4;

In accordance with literature¹⁶.



3dd

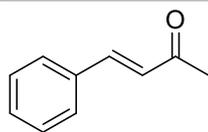
(E)-3-(3-chloroprop-1-en-1-yl)benzene (3dd)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether) afforded 56.2 mg (74% yield) of the title compound **3dd**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.43-7.39 (m, 2H), 7.37-7.33 (m, 2H), 7.35-7.30 (m, 1H), 6.71 (d, J = 15.6 Hz, 1H), 6.37 (dt, J = 15.5 Hz, J = 7.1 Hz, 1H), 4.29 (d, J = 7.3 Hz, 2H).;

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 136.0, 134.2, 128.7, 128.4, 126.8, 125.0, 45.6;

In accordance with literature¹⁷.



3ee

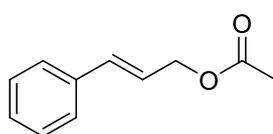
(E)-4-phenylbut-3-en-2-one (3ee)

Yellow oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (30:1)) afforded 56.9 mg (78% yield) of the title compound **3ee**.

¹H NMR (400 MHz, CDCl₃): δ = 7.58-7.49 (m, 3H), 7.42-7.38 (m, 3H), 6.72 (d, *J* = 16.2 Hz, 1H), 2.39 (s, 3H);

¹³C NMR (100 MHz, CDCl₃) δ = 198.5, 143.5, 134.5, 130.6, 129.0, 128.3, 127.1, 27.6;

In accordance with literature¹⁸.



3ff

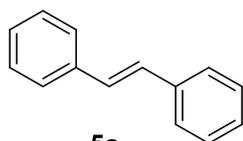
cinnamyl acetate (3ff)

Colourless oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (20:1)) afforded 59.8 mg (68% yield) of the title compound **3ff**.

¹H NMR (400 MHz, CDCl₃): δ = 7.46-7.41 (m, 2H), 7.39-7.34 (m, 2H), 7.31-7.27(m,1H), 6.70 (d, *J* = 16.0 Hz, 1H), 6.32 (dt, *J* = 16.0 Hz, 6.4Hz, 1H), 4.77(dd, *J* = 6.4Hz, 1.3Hz, 2H), 2.14 (s, 3H);

¹³C NMR (100 MHz, CDCl₃) δ =170.9, 136.3, 134.3, 128.7, 128.2, 126.7, 123.2, 65.2, 21.1;

In accordance with literature¹⁸.



5a

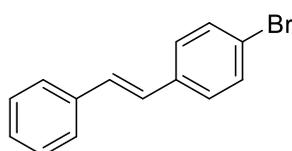
(E)-1,2-diphenylethene(5a)

White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 64.8 mg (72% yield) of the title compound **5a**.

¹H NMR (400 MHz, CDCl₃): δ = 7.61 (d, *J* = 7.6Hz, 4H), 7.35(t, *J* = 7.6Hz, 4H), 7.25(t, *J* = 7.2Hz, 2H), 7.11(s, 2H);

¹³C NMR (100 MHz, CDCl₃) : δ = 137.4, 128.8, 127.7, 126.6.

In accordance with literature¹⁹.



5b

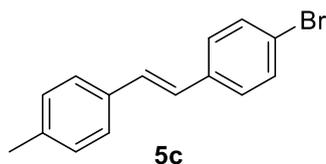
(E)-1-bromo-4-styrylbenzene(5b)

White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 78.7 mg (61% yield) of the title compound **5b**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.62-7.46(m, 4H), 7.39-7.33(m, 4H), 7.29-7.25(m, 1H), 7.10(d, J = 16.2 Hz, 1H), 7.03(d, J = 16.2 Hz, 1H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 137.0, 136.3, 131.8, 129.5, 128.8, 128.0, 127.9, 127.5, 126.6, 121.4.

In accordance with literature²⁰.



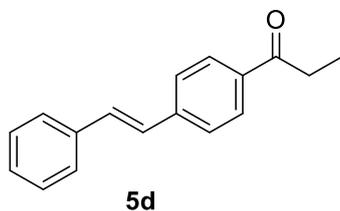
(E)-1-bromo-4-(4-methylstyryl)benzene(**5c**)

White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 63.9 mg (47% yield) of the title compound **5c**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.46(d, J = 8.3 Hz, 2H), 7.42-7.33(m, 4H), 7.17(d, J = 7.9 Hz, 2H), 7.07(d, J = 16.2 Hz, 1H), 6.98(d, J = 16.2 Hz, 1H), 2.36(s, 3H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 137.9, 136.5, 134.2, 131.8, 129.5, 129.4, 127.9, 126.5, 126.4, 121.1, 21.4.

In accordance with literature²¹.



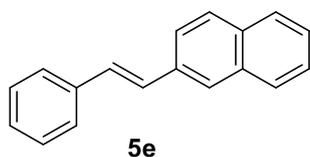
(E)-1-(4-styrylphenyl)propan-1-one(**5d**)

White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 75.5 mg (64% yield) of the title compound **5d**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 8.03(d, J = 8.1 Hz, 2H), 7.56-7.52(m, 4H), 7.40-7.35(m, 2H), 7.32-7.27(m, 1H), 7.22(d, J = 16.3 Hz, 1H), 7.12(d, J = 16.3 Hz, 1H), 4.38(q, J = 7.12 Hz, 2H), 1.40(t, J = 7.1 Hz, 3H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 166.5, 141.8, 136.8, 131.2, 130.1, 129.3, 128.9, 128.3, 127.7, 126.9, 126.4, 61.0, 14.4.

In accordance with literature²².



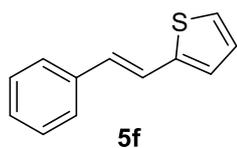
(E)-2-styrylnaphthalene(5e)

White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 67.7 mg (58% yield) of the title compound **5e**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.91-7.79(m, 4H), 7.77-7.73(m, 1H), 7.56(d, J = 7.6 Hz, 2H), 7.53-7.42(m, 2H), 7.38(t, J = 7.4 Hz, 2H), 7.31-7.23(m, 3H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) : δ = 137.4, 134.9, 133.8, 133.1, 129.1, 128.8, 128.8, 128.4, 128.1, 127.8, 127.7, 126.7, 126.6, 126.4, 126.0, 123.6.

In accordance with literature²³.



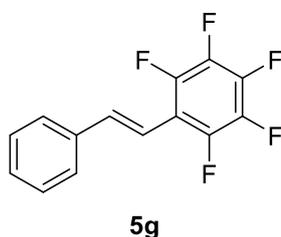
(E)-2-styrylthiophene(5f)

White solid, purification by flash chromatography on silica gel (petroleum ether/EtOAc (10:1)) afforded 51.2 mg (55% yield) of the title compound **5f**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.45(d, J = 8.3 Hz, 2H), 7.36-7.32(m, 2H), 7.28-7.17(m, 3H), 7.07-7.05(m, 1H), 7.01-6.97(m, 1H), 6.92(d, J = 16.3 Hz, 1H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) : δ = 143.0, 137.0, 128.8, 128.4, 127.7, 126.4, 126.2, 124.4, 121.9.

In accordance with literature²⁵.



(E)-1,2,3,4,5-pentafluoro-6-styrylbenzene(5g)

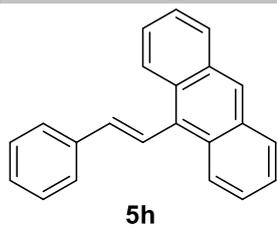
White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 90.5 mg (67% yield) of the title compound **5g**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.60-7.55(m, 2H), 7.51-7.36(m, 4H), 7.03(d, J = 16.2 Hz, 1H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) : δ = 146.2-145.9 (m), 143.8-143.4 (m), 141.2-140.8(m), 139.3-138.9(m), 138.9-138.3(m), 137.2 (t, J = 8.2 Hz,), 136.6, 129.0, 128.9, 126.9, 112.7, 112.4 (td, J = 13.4, 4.0 Hz).

$^{19}\text{F NMR}$ (100 MHz, CDCl_3) : δ = -142.74 (d, J = 7.9 Hz), -142.78 (d, J = 7.6 Hz), -156.55 (t, J = 20.8 Hz), -162.91 (t, J = 8.0 Hz), -162.98 (t, J = 13.3 Hz)

In accordance with literature²⁶.



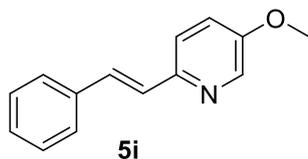
(E)-9-styrylanthracene(5h)

White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 98.0 mg (69% yield) of the title compound **5h**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 8.44-8.32(m, 3H), 8.00-7.97(m, 2H), 7.88(d, J = 16.8 Hz, 1H), 7.65(d, J = 7.6 Hz, 2H), 7.46-7.39 (m, 6H), 7.37-7.33(m, 1H), 6.92(d, J = 16.8 Hz, 1H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) : δ = 137.4, 132.9, 131.6, 129.8, 129.0, 128.8, 128.1, 126.7, 126.6, 126.1, 125.6, 125.3, 125.0.

In accordance with literature²⁷.



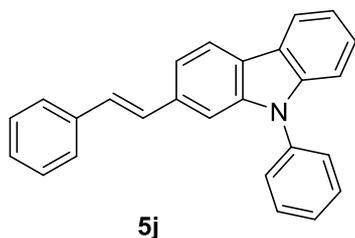
(E)-5-methoxy-2-styrylpyridine(5i)

Yellow oil, purification by flash chromatography on silica gel (petroleum ether/EtOAc (10:1)) afforded 70.7 mg (67% yield) of the title compound **5i**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 8.12(d, J = 5.4 Hz, 1H), 7.52(d, J = 7.3 Hz, 2H), 7.38(d, J = 6.8 Hz, 2H), 7.34-7.21(m, 2H), 7.04-6.94 (m, 2H), 6.79(s, 1H), 3.96(s, 3H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) : δ = 165.0, 147.5, 147.0, 136.3, 133.0, 128.9, 128.7, 127.1, 126.1, 114.2, 108.2, 53.6.

In accordance with literature²⁴.



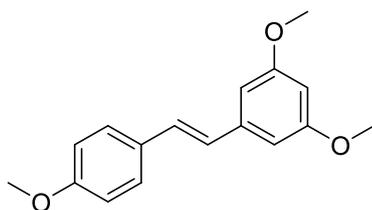
(E)-9-phenyl-2-styryl-9H-carbazole(5j)

White solid, purification by flash chromatography on silica gel (petroleum ether/EtOAc (10:1)) afforded 127.6 mg (74% yield) of the title compound **5j**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 8.20(s, 1H), 8.11(d, J = 7.6 Hz, 1H), 7.53-7.43(m, 7H), 7.38-7.23(m, 8H), 7.19(t, J = 7.6 Hz, 1H), 7.10(d, J = 16.4Hz, 1H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) : δ = 141.5, 140.8, 138.1, 137.7, 130.1, 129.9, 129.7, 128.9, 127.7, 127.3, 127.2, 126.6, 126.5, 126.4, 125.0, 124.0, 123.6, 120.6, 120.4, 118.8, 110.2.

HRMS: calcd for C₂₆H₁₉N [M+H]⁺ : 346.1591, found:346.1596.



5k

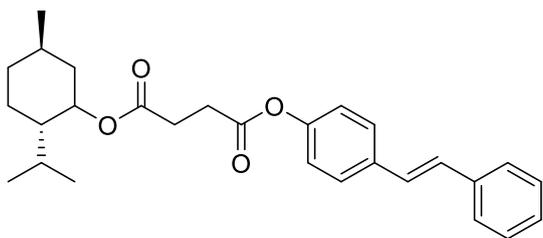
(E)-1,3-dimethoxy-5-(4-methoxystyryl)benzene(5k)

White solid, purification by flash chromatography on silica gel (petroleum ether/EtOAc (10:1)) afforded 83.7mg (74% yield) of the title compound **5k**.

¹H NMR (400 MHz, CDCl₃): δ = 7.44(d, J = 8.6 Hz, 2H), 7.04(d, J = 16.2 Hz, 1H), 6.92-6.88(m, 3H), 6.65(d, J = 1.8 Hz, 2H), 6.37(t, J = 2.5 Hz, 1H), 3.82(s, 9H);

¹³C NMR (100 MHz, CCl₃) : δ = 161.0, 159.4, 139.8, 130.0, 128.8, 127.9, 126.6, 114.2, 104.4, 99.7, 55.4.

In accordance with literature²⁸.



5l

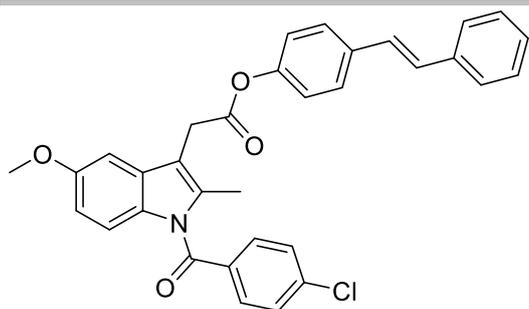
(2S,5R)-2-isopropyl-5-methylcyclohexyl 4-((E)-styryl)phenyl succinate(5l)

White solid, purification by flash chromatography on silica gel (petroleum ether/EtOAc (10:1)) afforded 143.2 mg (66% yield) of the title compound **5l**.

¹H NMR (400 MHz, CDCl₃): δ = 7.48(d, J = 8.1Hz, 4H), 7.33(t, J = 7.4Hz, 2H), 7.26-7.21(m, 1H), 7.09-7.04(m, 4H), 4.74(td, J = 10.8Hz, 4.2Hz, 1H), 2.86(t, J = 6.5Hz, 2H), 2.73-2.69(m, 2H), 2.02-1.98(m, 1H), 1.91-1.85(m, 1H), 1.68-1.65(m, 2H), 1.48-1.34(m, 2H), 1.09-0.94(m, 2H), 0.90-0.84(m, 7H), 0.75(d, J = 7.0Hz, 3H)

¹³C NMR (100 MHz, CDCl₃) : δ = 171.7, 171.0, 150.1, 137.2, 135.2, 129.0, 128.8, 127.8, 127.7, 127.5, 126.6, 121.8, 74.8, 47.1, 41.0, 34.3, 31.5, 29.5, 26.4, 23.5, 22.1, 20.9, 16.4.

HRMS: calcd for C₂₈H₃₄O₄ [M+H]⁺ : 435.2530, found:435.2533.



5m

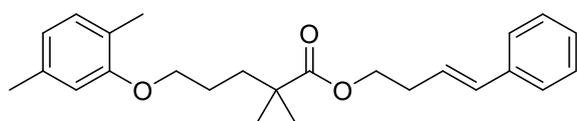
(E)-4-styrylbenzyl 1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indole-3-carboxylate(5m)

White solid, purification by flash chromatography on silica gel (petroleum ether/EtOAc (5:1)) afforded 82.9 mg (31% yield) of the title compound **5m**.

¹H NMR (400 MHz, CDCl₃): δ = 7.69(d, *J* = 8.1Hz, 2H), 7.51-7.47(m, 4H), 7.41(d, *J* = 8.1Hz, 2H) 7.37-7.33(m, 2H), 7.08-7.03(m, 4H), 6.98(s, 1H), 6.91(d, *J* = 8.7Hz, 1H), 6.84-6.81(m, 2H), 3.92(s, 2H), 3.85(s, 3H), 2.46(s, 3H).

¹³C NMR (100 MHz, CDCl₃): δ = 169.4, 167.4, 156.2, 139.4, 131.3, 129.2, 129.1, 128.8, 128.7, 128.2, 128.0, 127.8, 127.6, 127.5, 127.3, 126.7, 126.6, 126.3, 121.7, 115.7, 115.1, 111.9, 101.3, 55.8, 30.2, 13.5.

HRMS: calcd for C₃₃H₂₆ClNO₄ [M+H]⁺ : 536.1623, found:536.1627.



5n

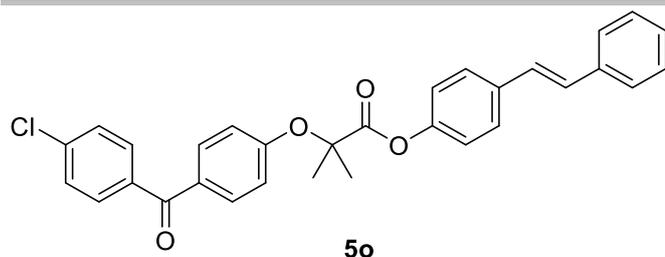
(E)-9-(2,5-dimethylphenoxy)-6,6-dimethyl-1-phenylnon-1-en-5-one (5n)

White solid, purification by flash chromatography on silica gel (petroleum ether/EtOAc (20:1)) afforded 98.2 mg (54% yield) of the title compound **5n**.

¹H NMR (400 MHz, CDCl₃): δ = 7.34-7.30(m, 2H), 7.29-7.26(m, 2H), 7.22-7.17(m, 1H), 6.90(d, *J* = 7.5Hz, 1H), 6.65(d, *J* = 7.5 Hz, 1H), 6.54(s,1H), 6.46(d, *J* = 15.9Hz, 1H), 6.15(dt, *J* = 15.9Hz, 7.0Hz, 1H), 4.20 (t, *J* = 6.7 Hz, 2H), 3.85-3.80 (m, 2H), 2.54(q, *J* = 6.7Hz, 2H), 2.30(s, 3H), 2.15(s, 3H), 1.73-1.69(m, 4H), 1.21(s, 6H);

¹³C NMR (100 MHz, CDCl₃) δ =177.8, 157.0, 137.3, 136.5, 132.4, 130.3, 128.9, 127.3, 126.1, 125.8, 123.9, 126.7, 111.9, 67.9, 63.5, 42.2, 37.2, 32.6, 25.3, 21.5, 15.8;

HRMS: calcd for C₂₅H₃₂O₃ [M+H]⁺ : 381.2424, found: 381.2423.



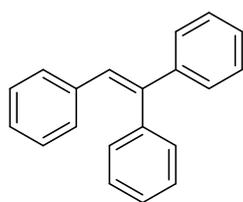
(E)-4-styrylphenyl 2-(4-benzoylphenoxy)-2-methylpropanoate(5o)

White solid, purification by flash chromatography on silica gel (petroleum ether/EtOAc (10:1)) afforded 140.9 mg (61% yield) of the title compound **5o**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.86-7.83(m, 2H), 7.79-7.76(m, 2H), 7.66-7.49(m, 6H), 7.42-7.39(m, 2H), 7.33-7.30(m, 1H), 7.12-7.10(m, 2H), 7.06-7.02(m, 4H), 1.88(s, 6H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 194.3, 172.5, 159.6, 149.8, 138.6, 137.1, 136.4, 135.7, 132.2, 131.3, 130.8, 129.4, 128.8, 128.7, 127.9, 127.6, 127.5, 126.6, 121.5, 117.4, 79.5, 25.5.

HRMS: calcd for $\text{C}_{31}\text{H}_{25}\text{ClO}_4$ $[\text{M}+\text{H}]^+$: 497.1514, found:497.1519.



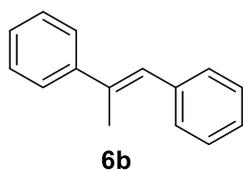
ethene-1,1,2-triyltribenzene(6a)

White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 85.8 mg (67% yield) of the title compound **6a**.

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ = 7.33-7.26(m, 8H), 7.22-7.19(m, 2H), 7.15-7.09(m, 3H), 7.04-7.02(m, 2H), 6.96(s, 1H);

$^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ = 143.5, 142.7, 140.4, 137.5, 130.5, 129.6, 128.7, 128.3, 128.2, 128.0, 127.7, 127.6, 127.5, 126.8.

In accordance with literature²⁹.

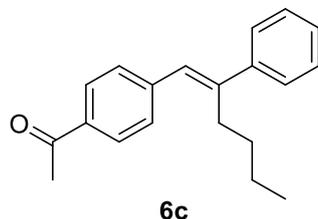


(E)-prop-1-ene-1,2-diyl dibenzene(6b)

White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 59.1 mg (61% yield) of the title compound **6b**.

¹H NMR (400 MHz, CDCl₃): δ = 7.59-7.57(m, 2H), 7.45-7.40(m, 6H), 7.35-7.28(m, 2H), 6.89(s, 1H), 2.34(s, 3H);
¹³C NMR (100 MHz, CDCl₃): δ = 144.02, 138.41, 137.48, 129.21, 128.39, 128.24, 127.76, 127.25, 126.53, 126.07, 17.55.

In accordance with literature³⁰.



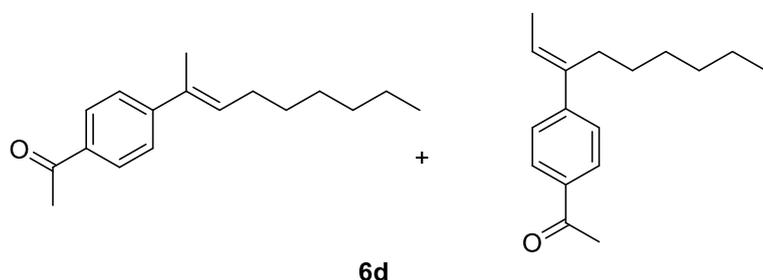
(E)-1-(4-(2-phenylhex-1-en-1-yl)phenyl)ethan-1-one(6c)

White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 61.2 mg (44% yield) of the title compound **6c**.

¹H NMR (400 MHz, CDCl₃): δ = 7.88(d, *J* = 8.4Hz, 2H), 7.26-7.24(m, 2H), 7.10-7.05(m, 3H), 6.91-6.89(m, 2H), 6.51(s, 1H), 2.60(s, 3H), 2.51(t, *J* = 6.3Hz, 2H), 1.38-1.34(m, 4H), 0.89(t, *J* = 7.0Hz, 3H);

¹³C NMR (100 MHz, CDCl₃): δ = 198.0, 146.9, 142.4, 137.1, 135.6, 129.1, 129.0, 128.7, 128.0, 127.3, 126.5, 40.0, 30.2, 26.7, 22.3, 14.0.

HRMS: calcd for C₂₀H₂₂O [M+H]⁺ : 279.1744, found:279.1736.



(E)-1-(4-(non-2-en-2-yl)phenyl)ethan-1-one and (E)-1-(4-(non-2-en-3-yl)phenyl)ethan-1-one (6d)

White solid, purification by flash chromatography on silica gel (petroleum ether) afforded 51.2 mg (42% yield) of the title compound **6d**.

¹H NMR (400 MHz, CDCl₃): δ = 7.90(dd, *J* = 8.6Hz, 1.9Hz 2H), 7.46(d, *J* = 8.5 Hz, 1.16H), 7.41(d, *J* = 8.5 Hz, 0.85H), 5.92(td, *J* = 7.2Hz, 1.1Hz, 0.57H), 5.36(q, *J* = 6.8Hz, 0.42H), 2.59(s, 3H), 2.51(t, *J* = 6.8Hz, 0.85H), 2.22(q, *J* = 6.8Hz, 1.23H), 2.05(s, 1.75H), 1.82(d, *J* = 7.0Hz, 1.28H), 1.50-1.28(m, 8H), 0.91-0.84(m, 3H).

¹³C NMR (100 MHz, CDCl₃): δ = 197.9, 140.5, 131.4, 128.5, 128.5, 126.3, 125.6, 125.1, 56.5, 31.8, 31.7, 29.5, 29.2, 29.0, 28.4, 26.6, 22.7, 15.6, 14.4, 14.1.

HRMS: calcd for C₁₇H₂₄O [M+H]⁺ : 245.1900, found:245.1905.

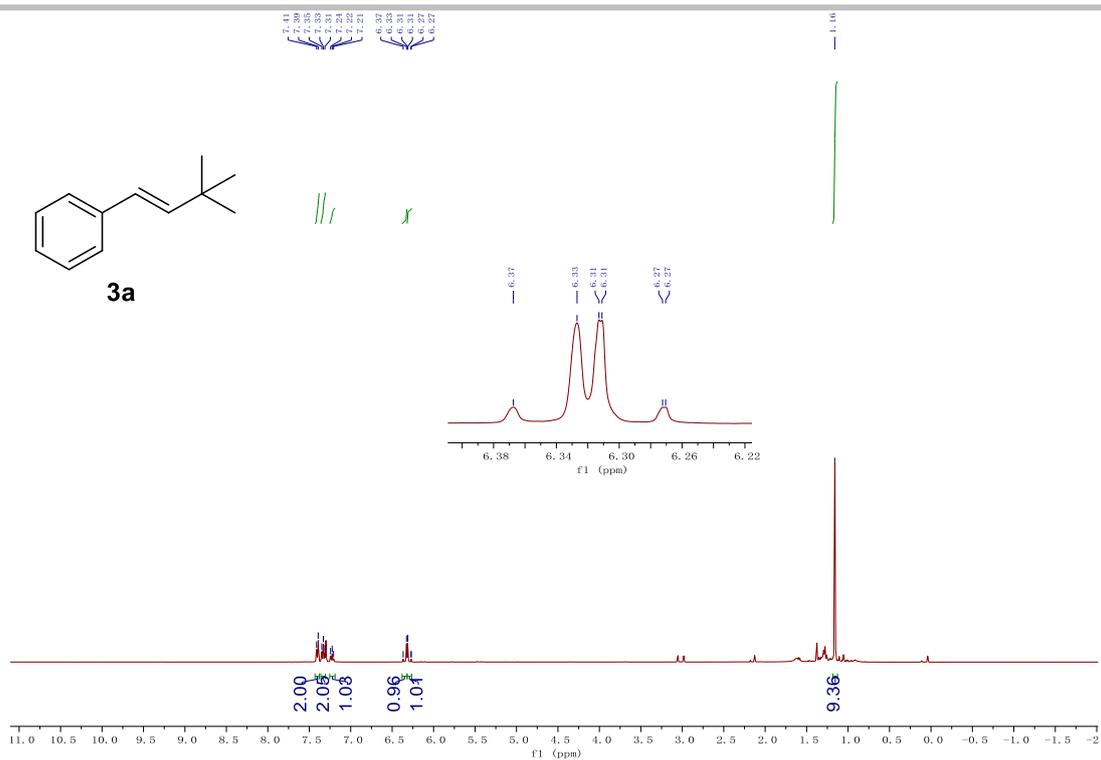
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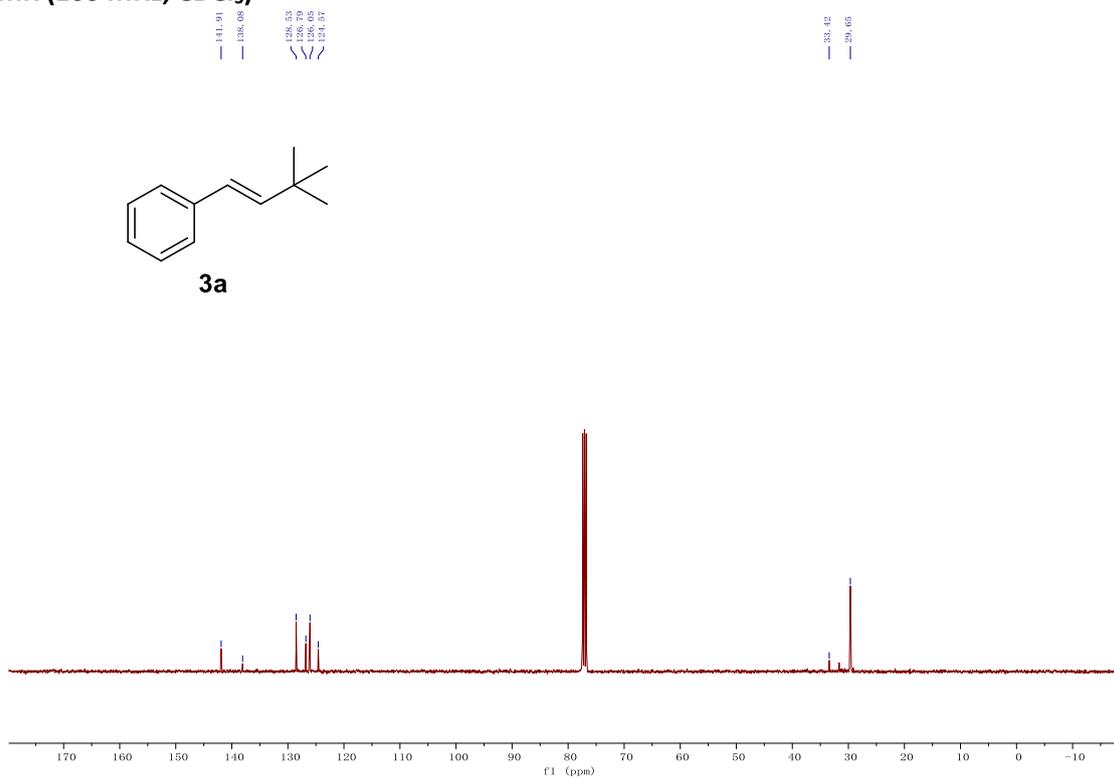
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10. Copies of NMR Spectra

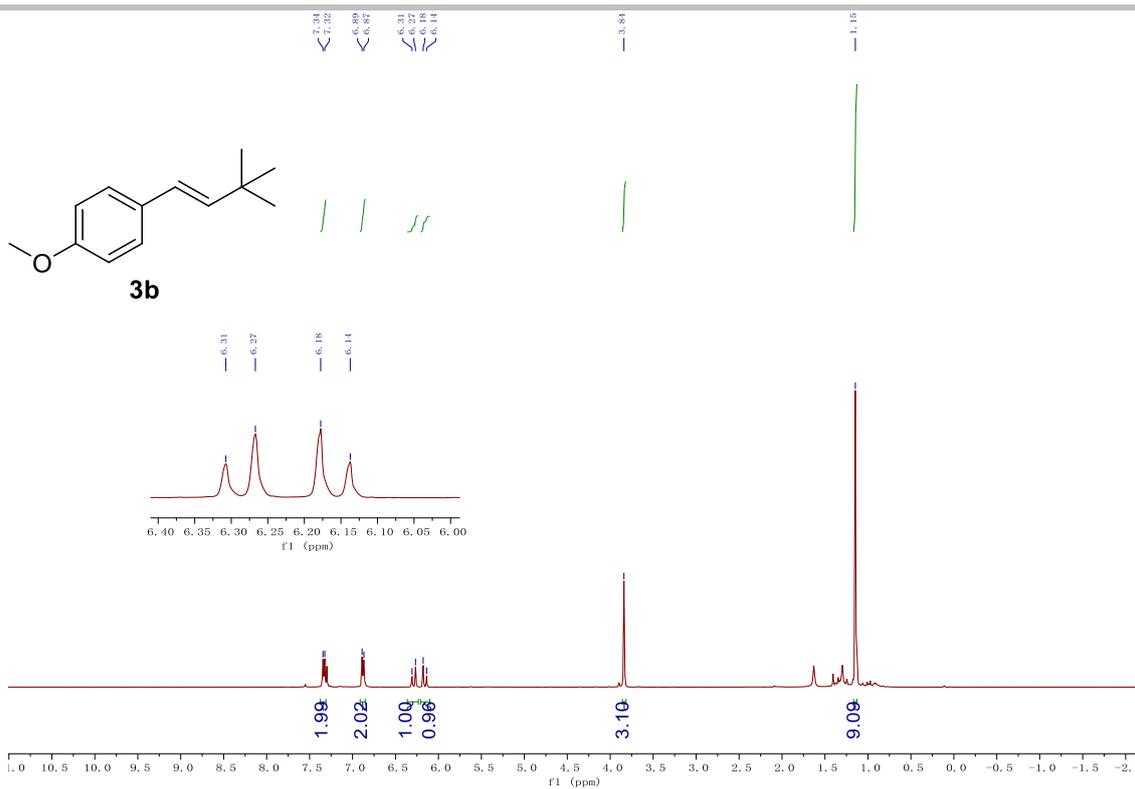
3a: ¹H NMR (400 MHz, CDCl₃)



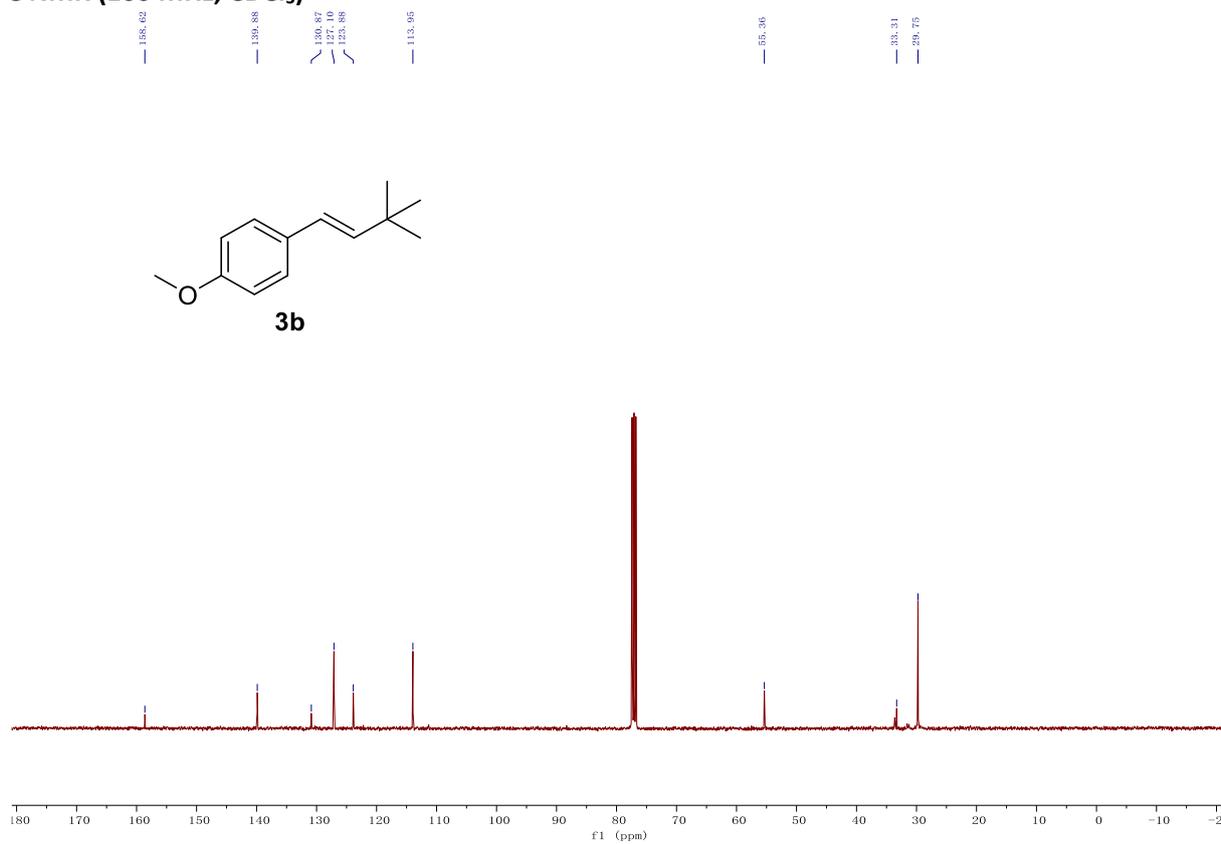
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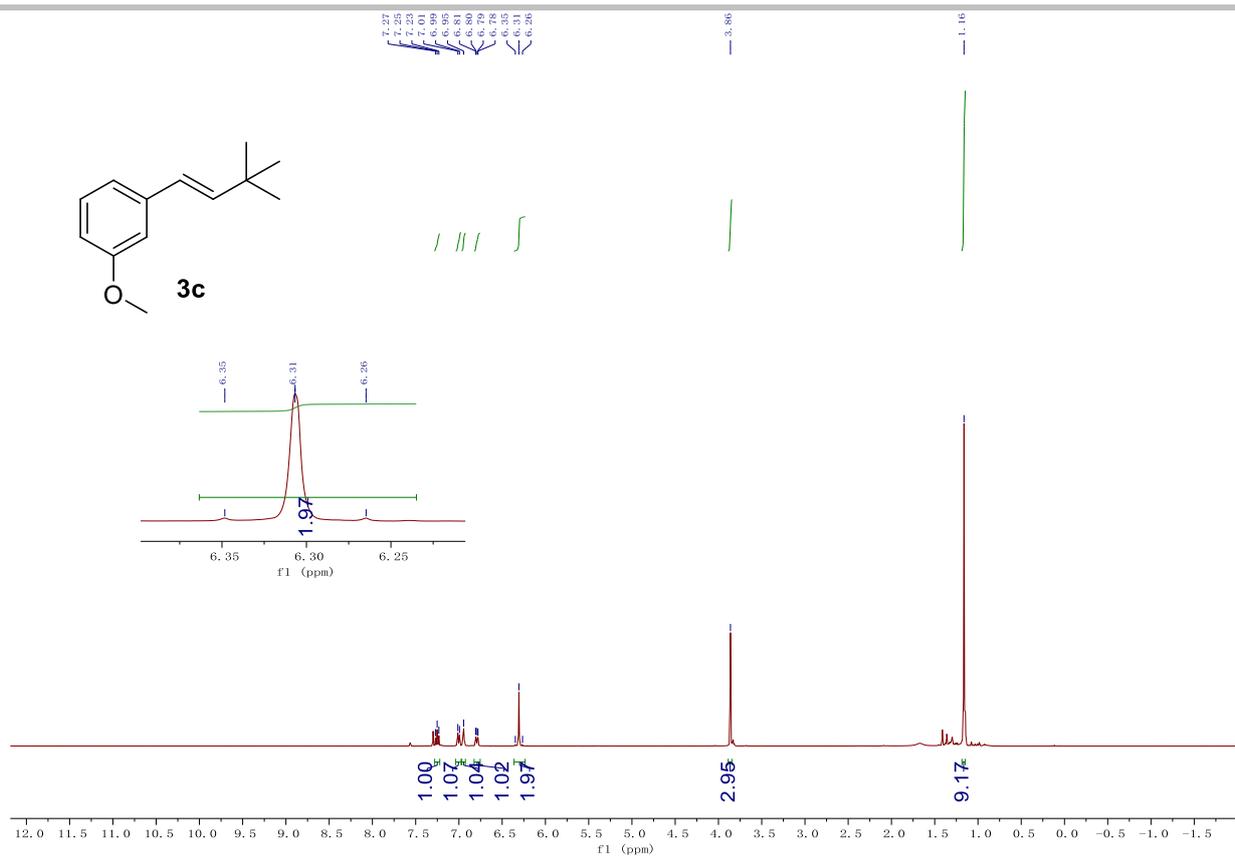
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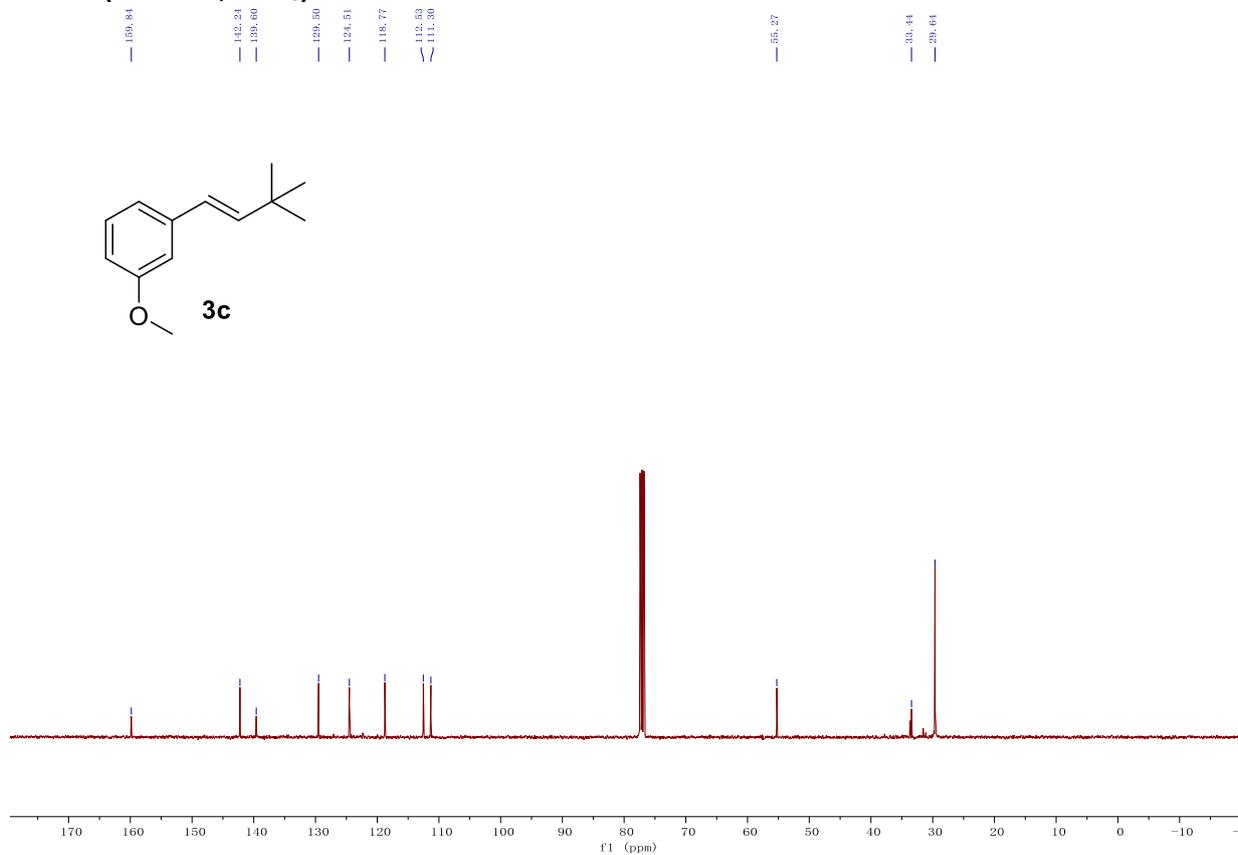
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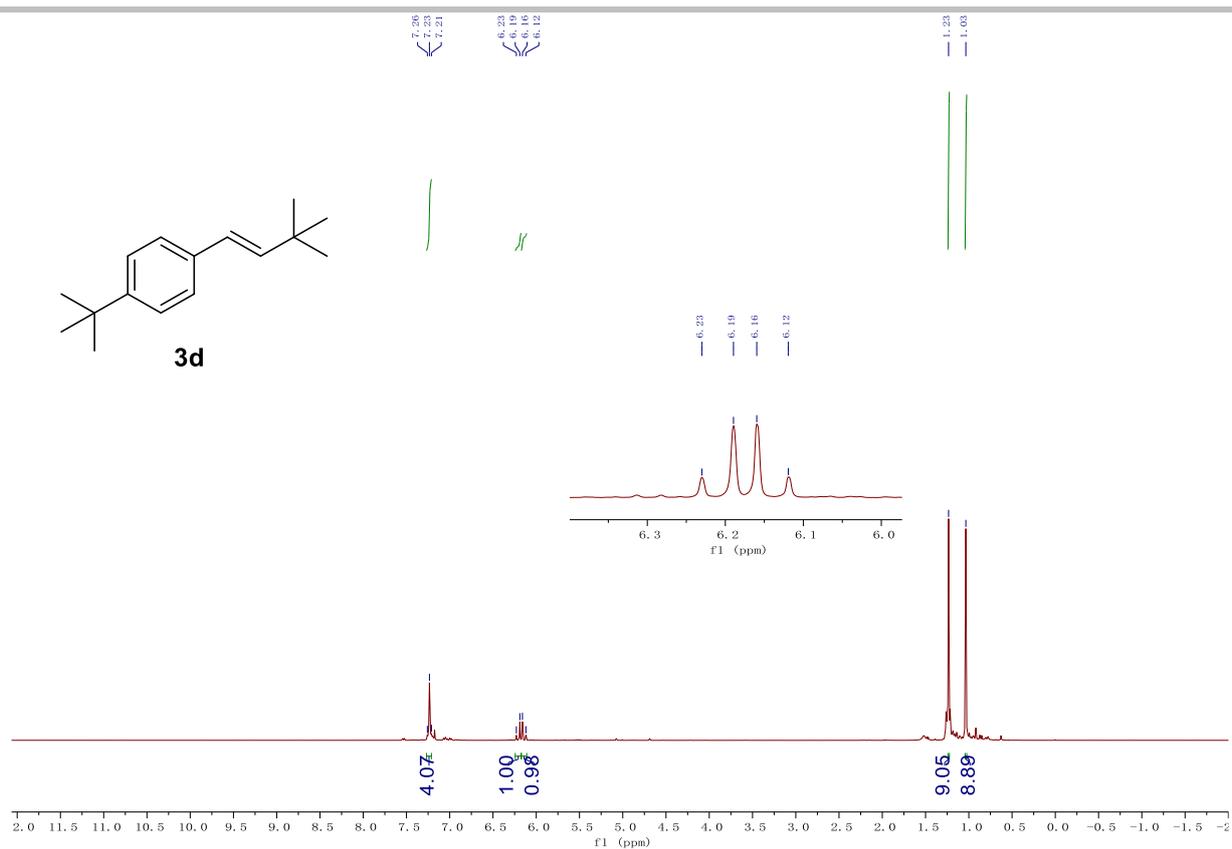
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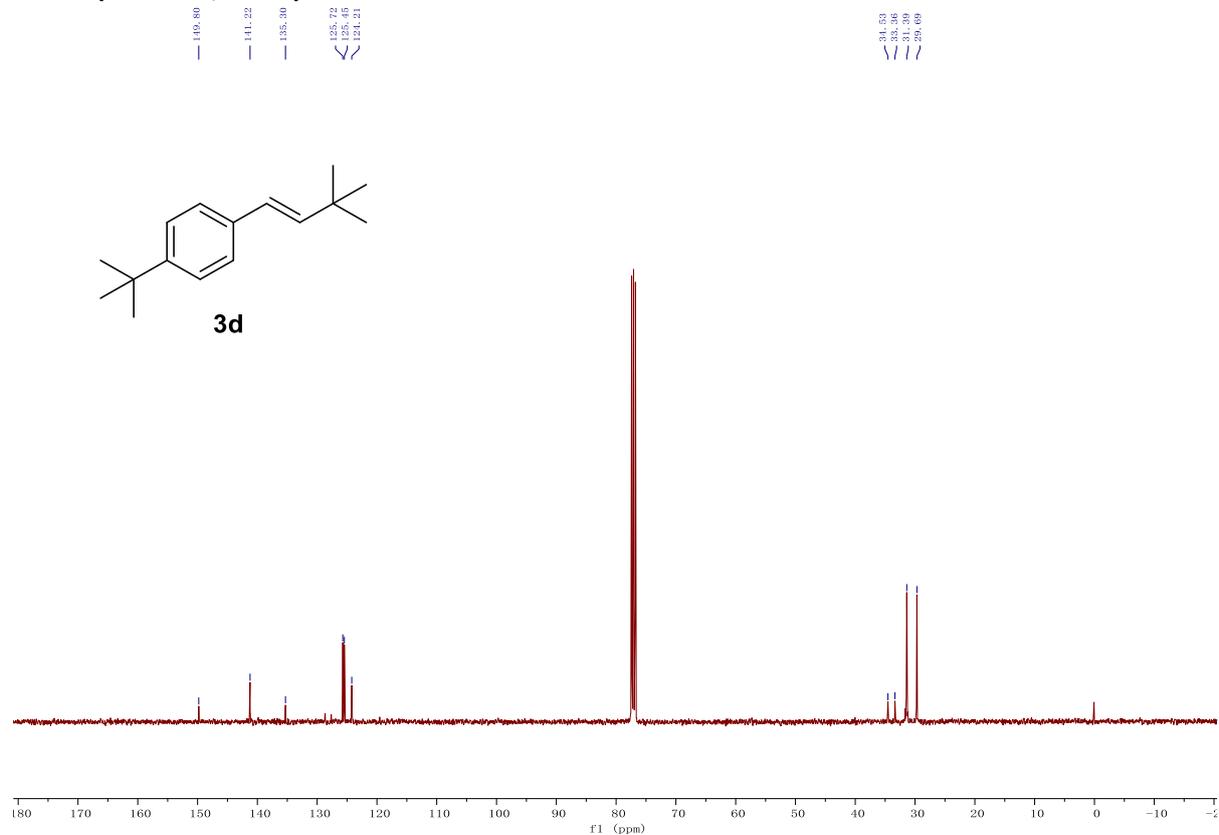
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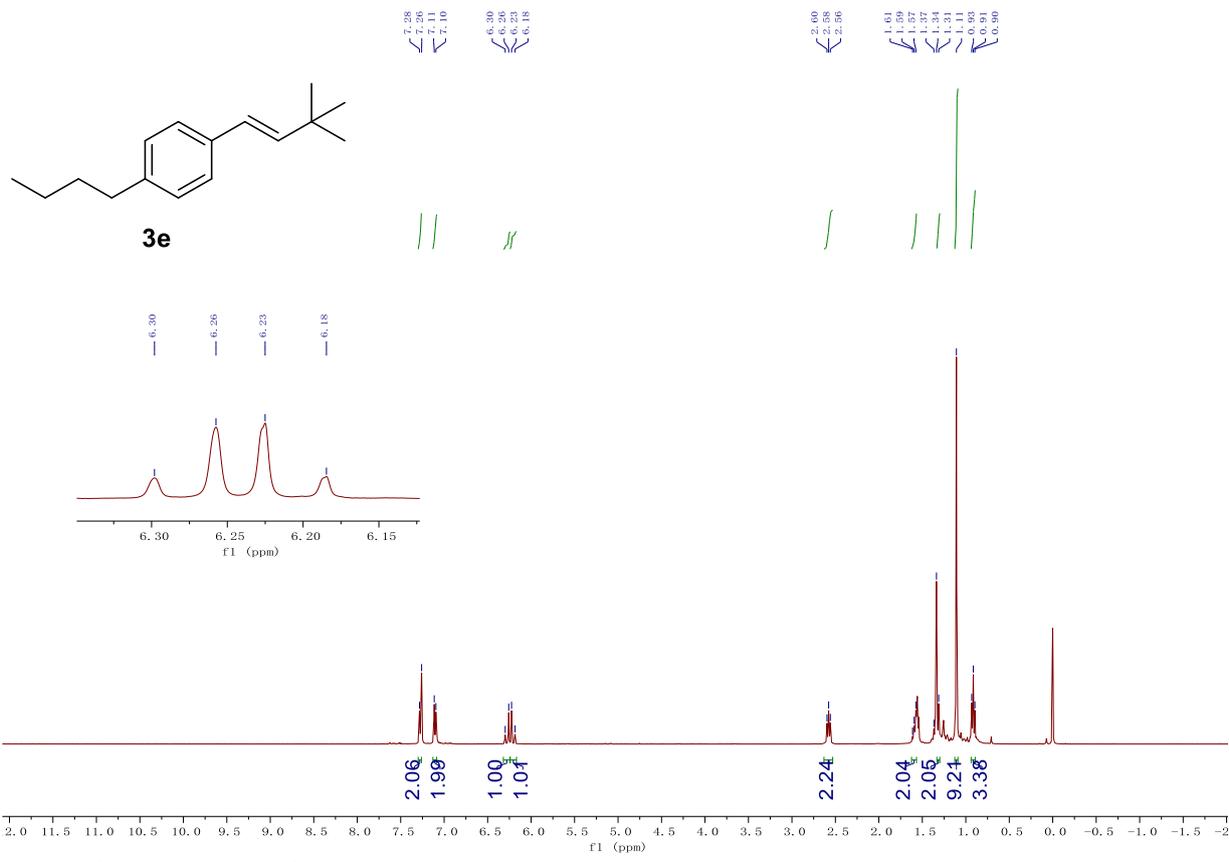
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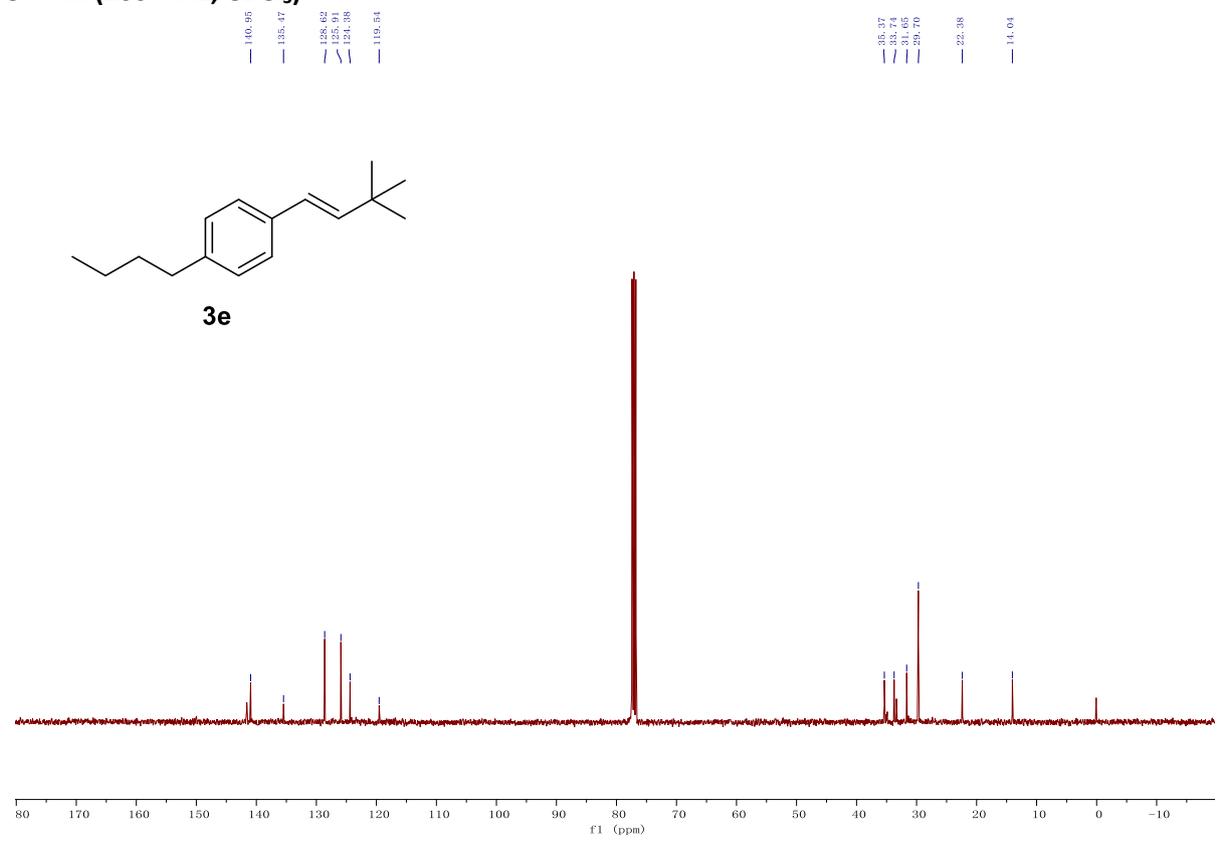
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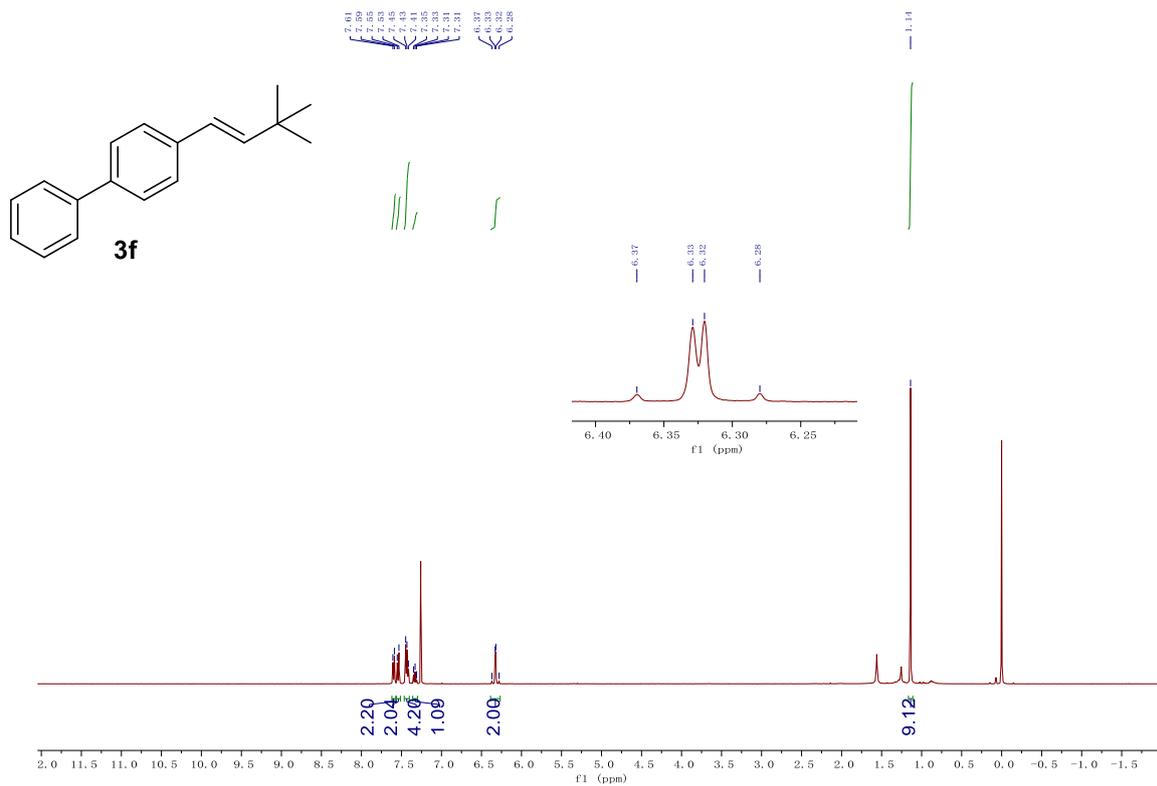
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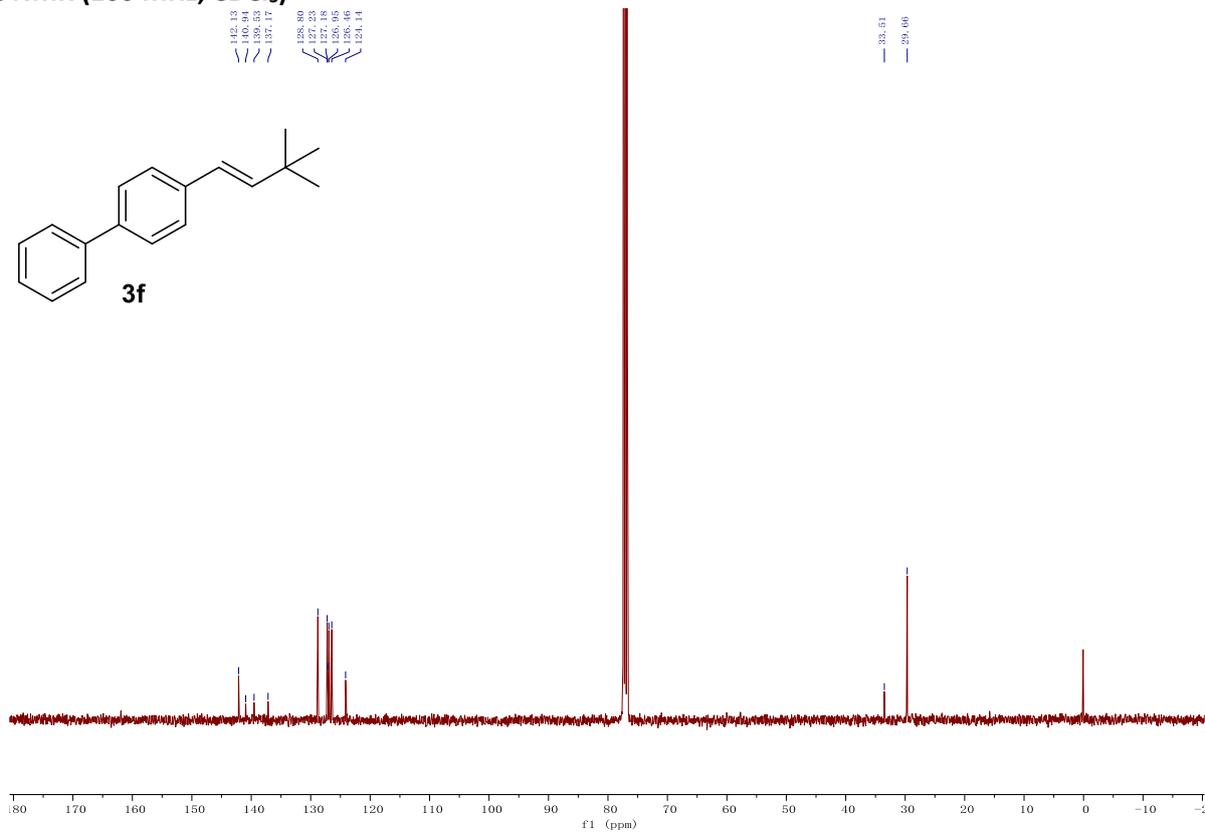
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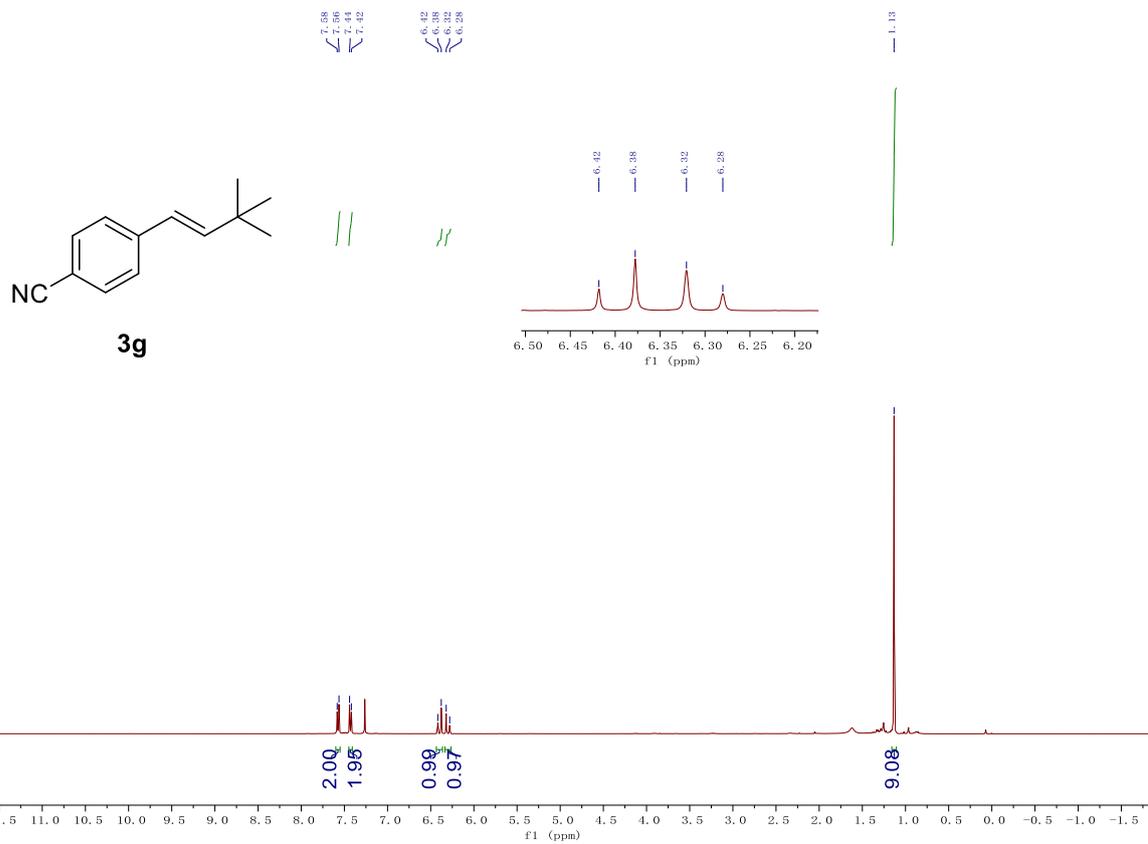
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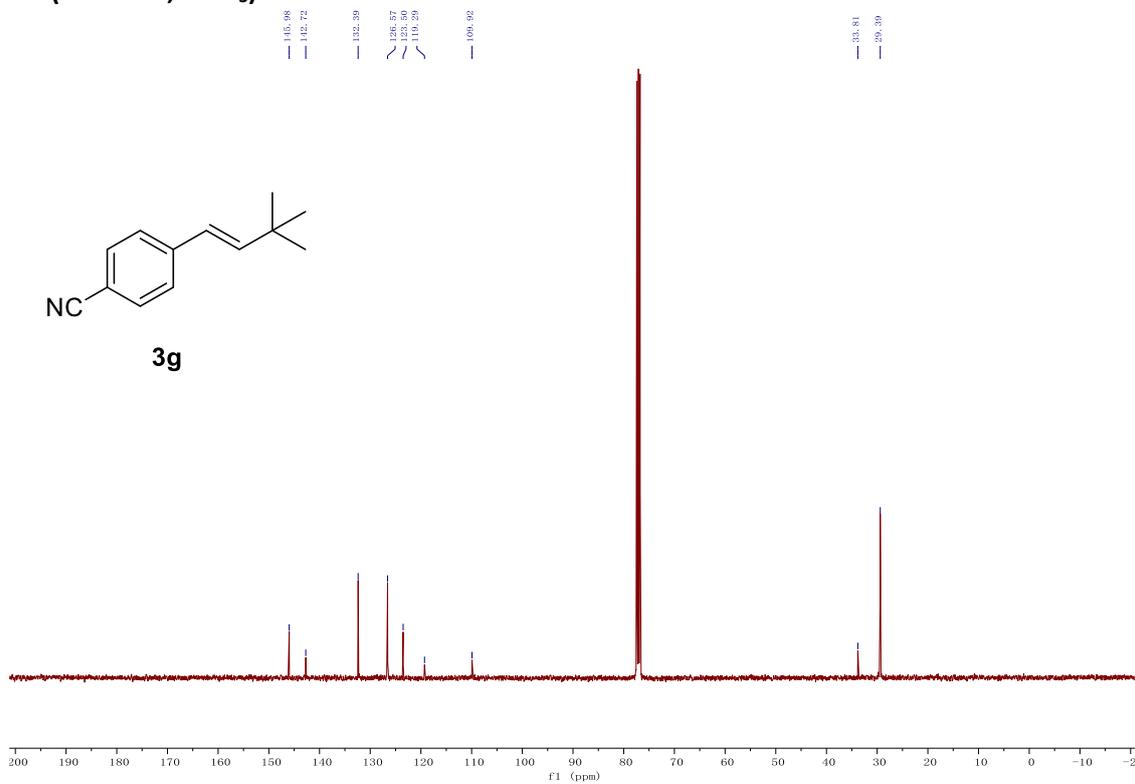
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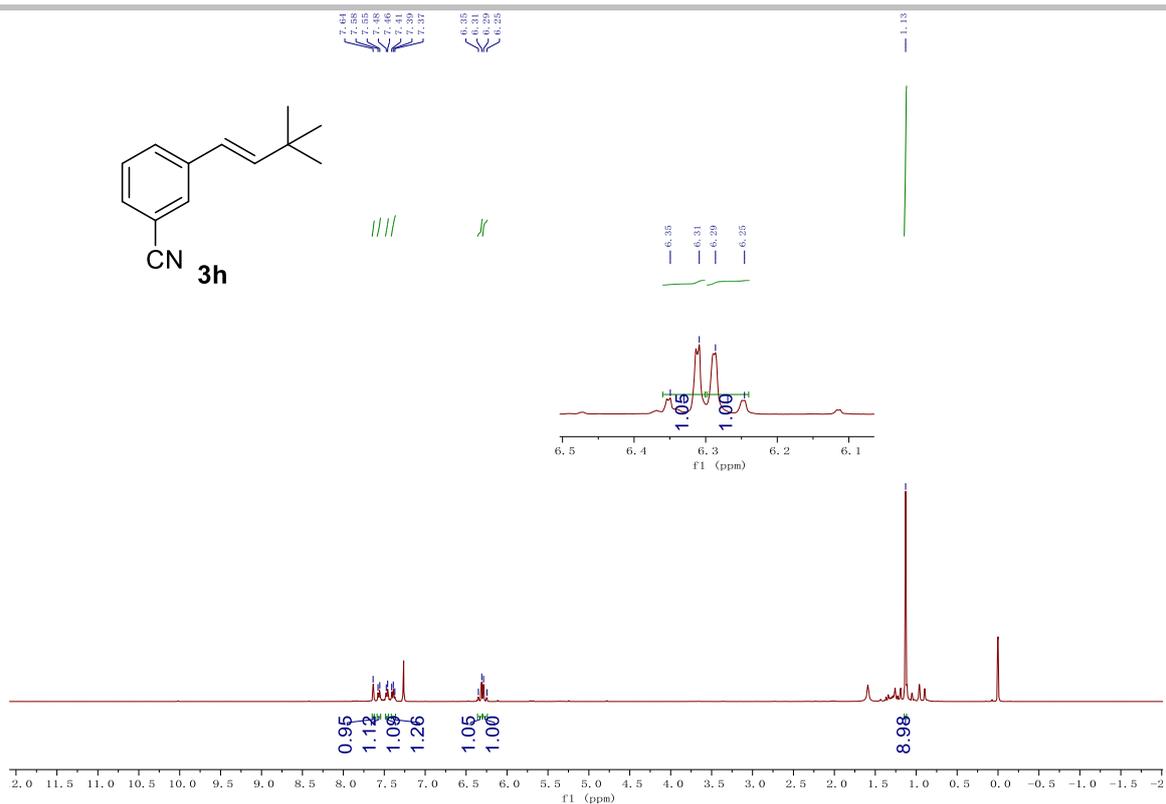
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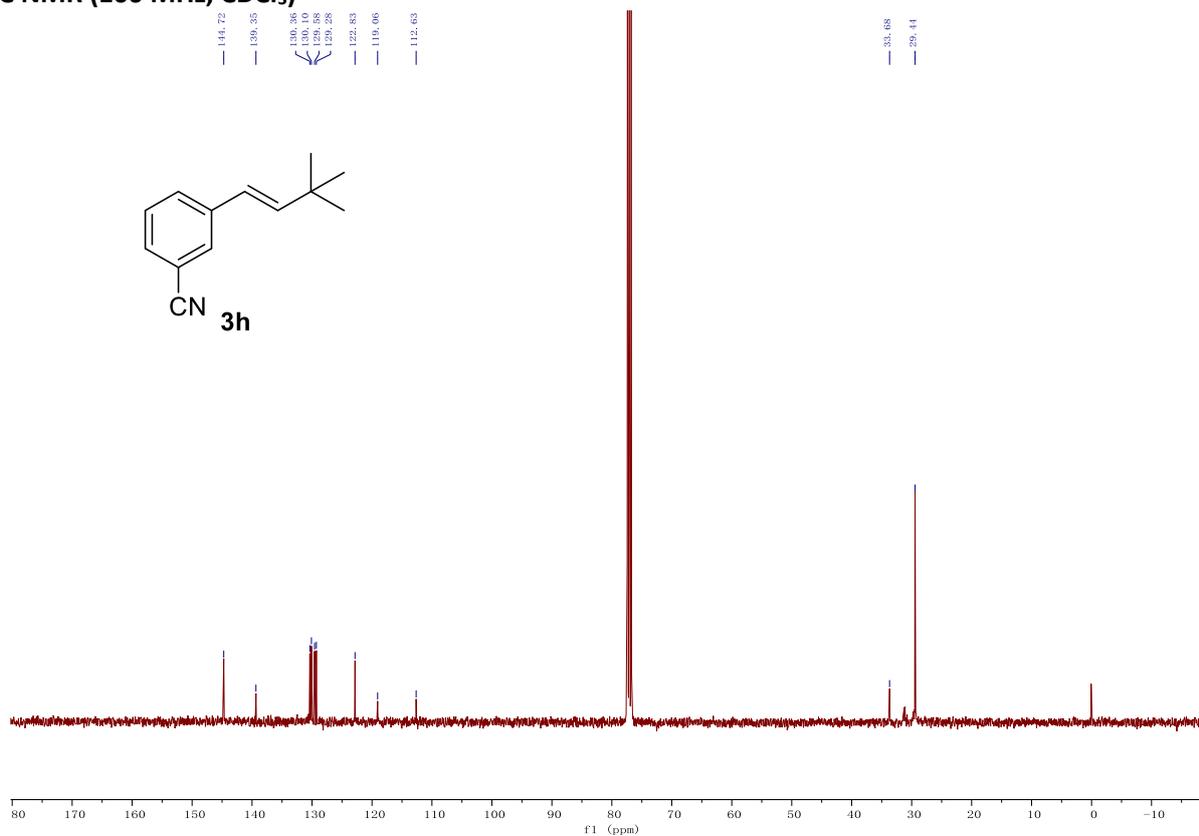
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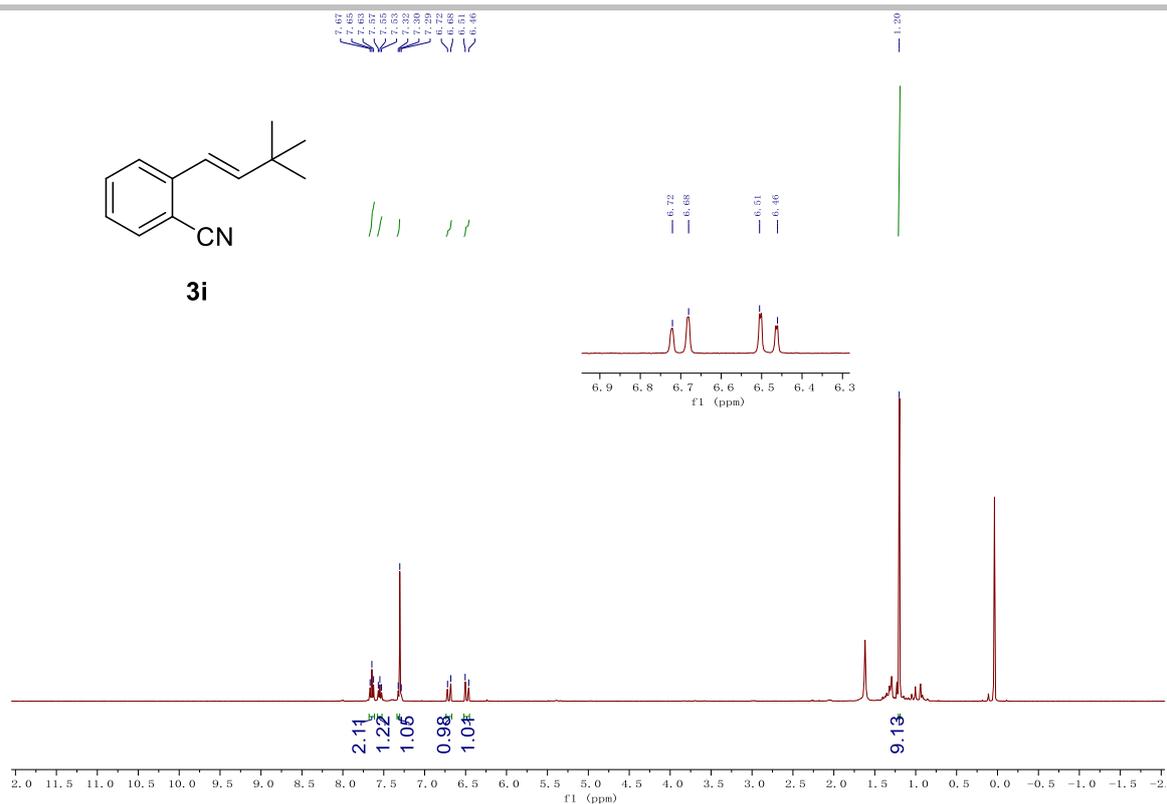
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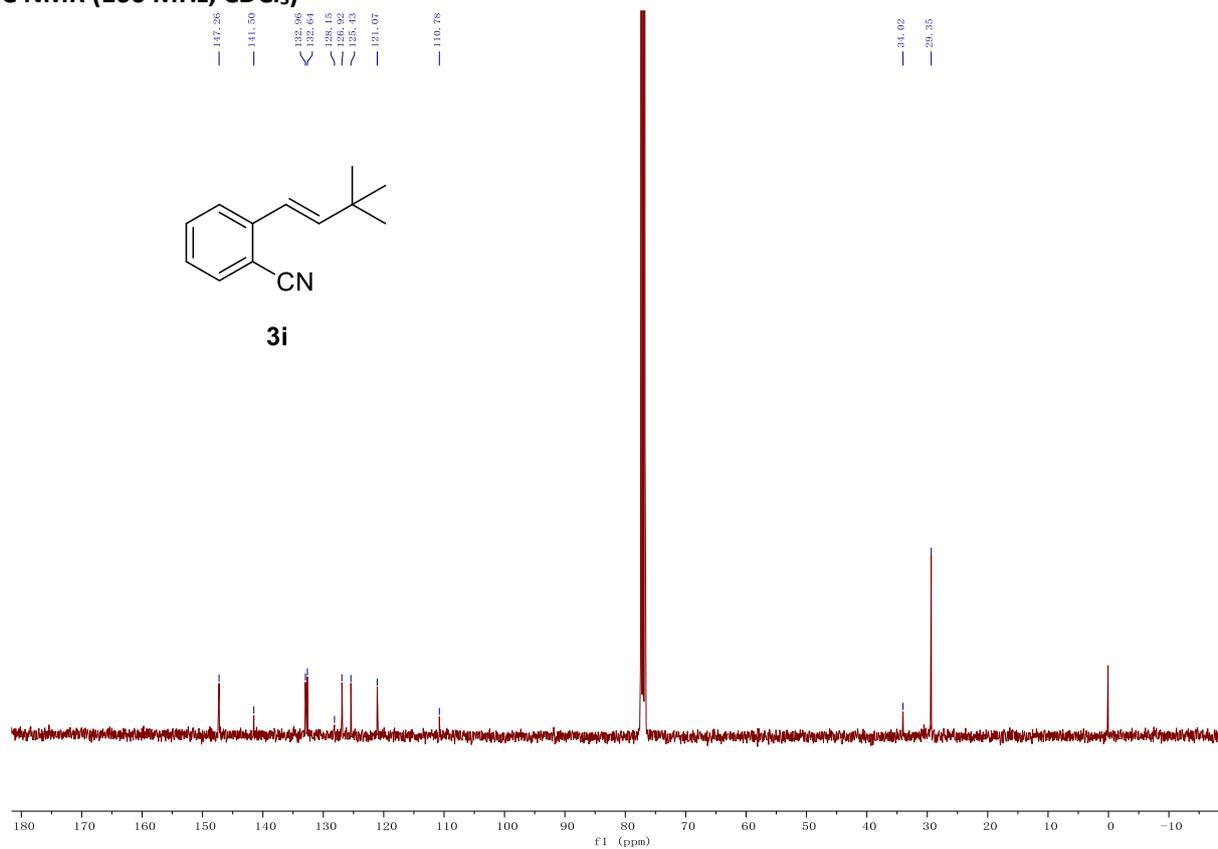
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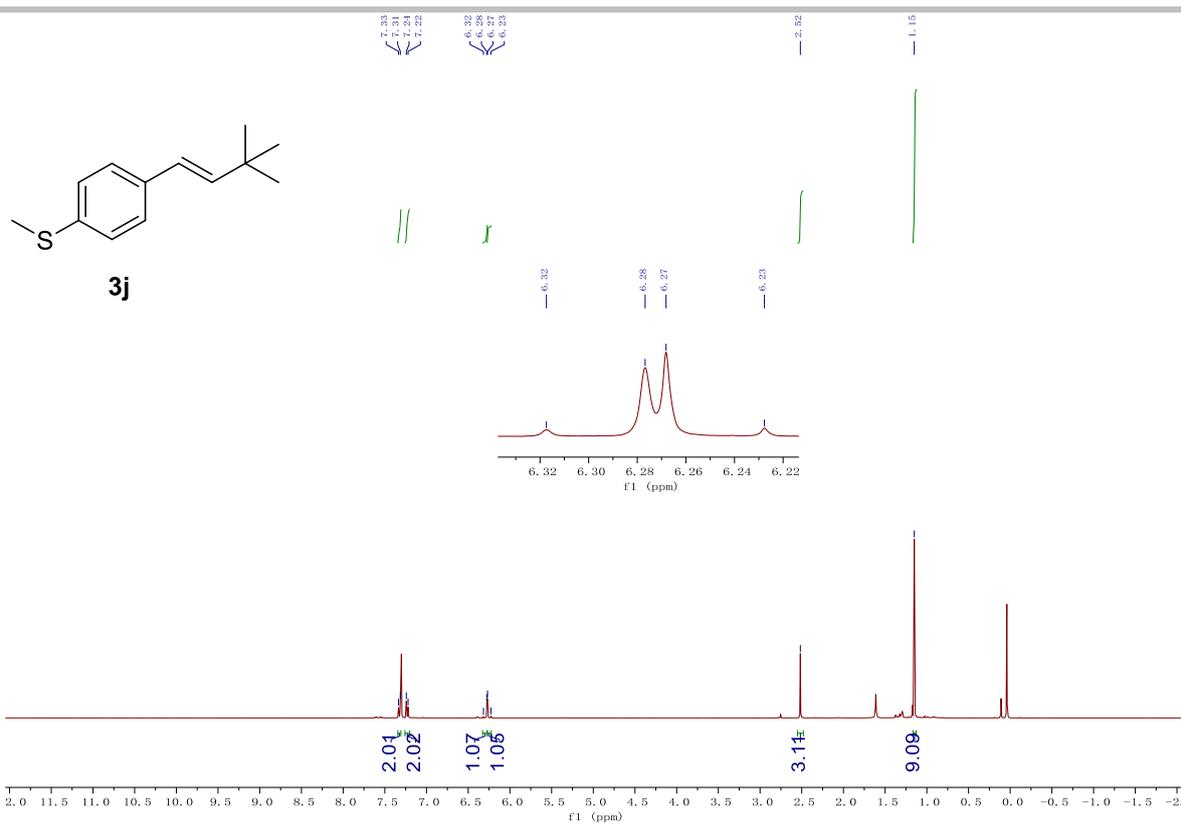
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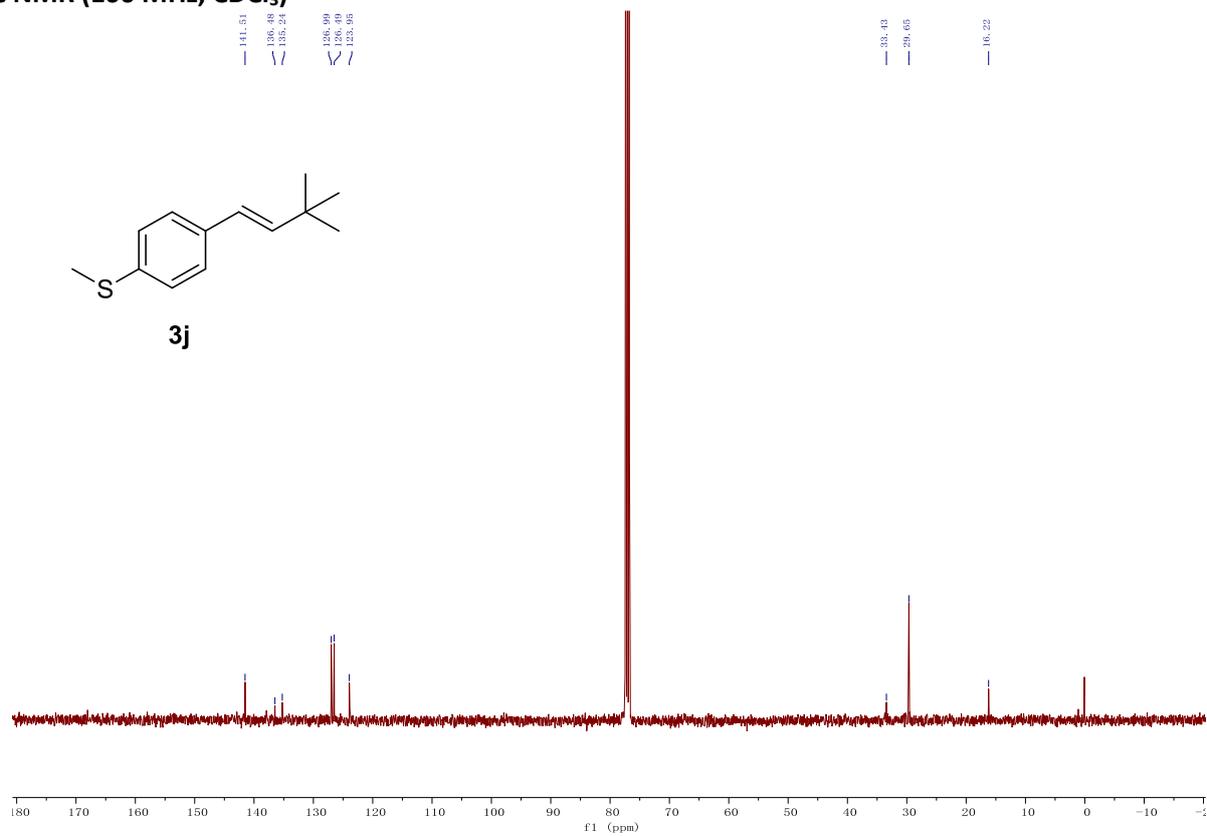
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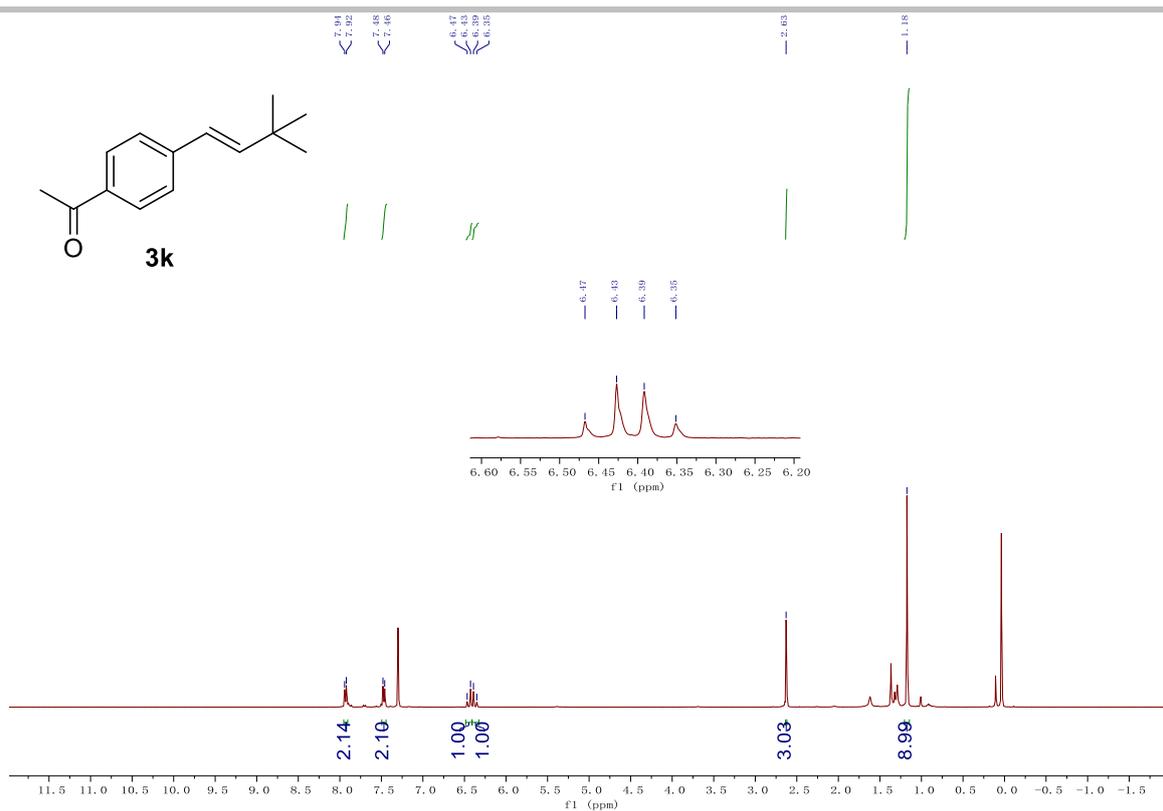
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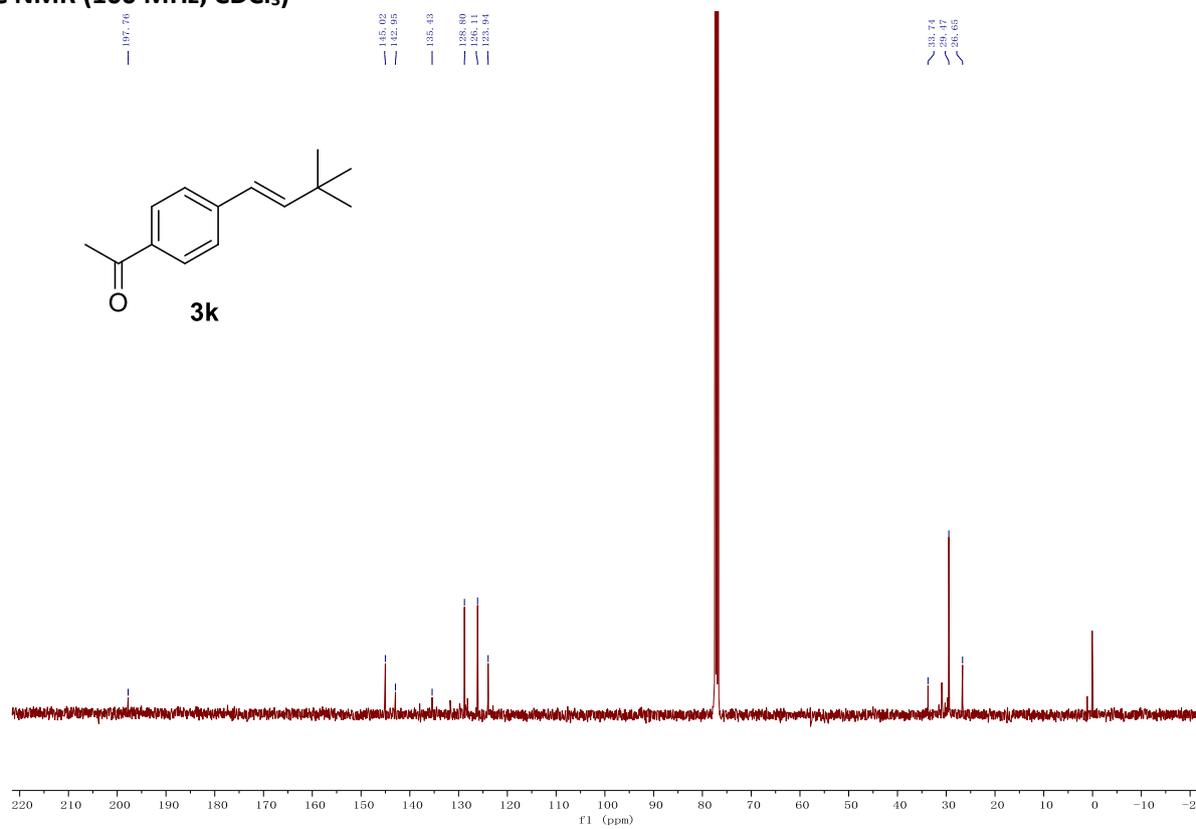
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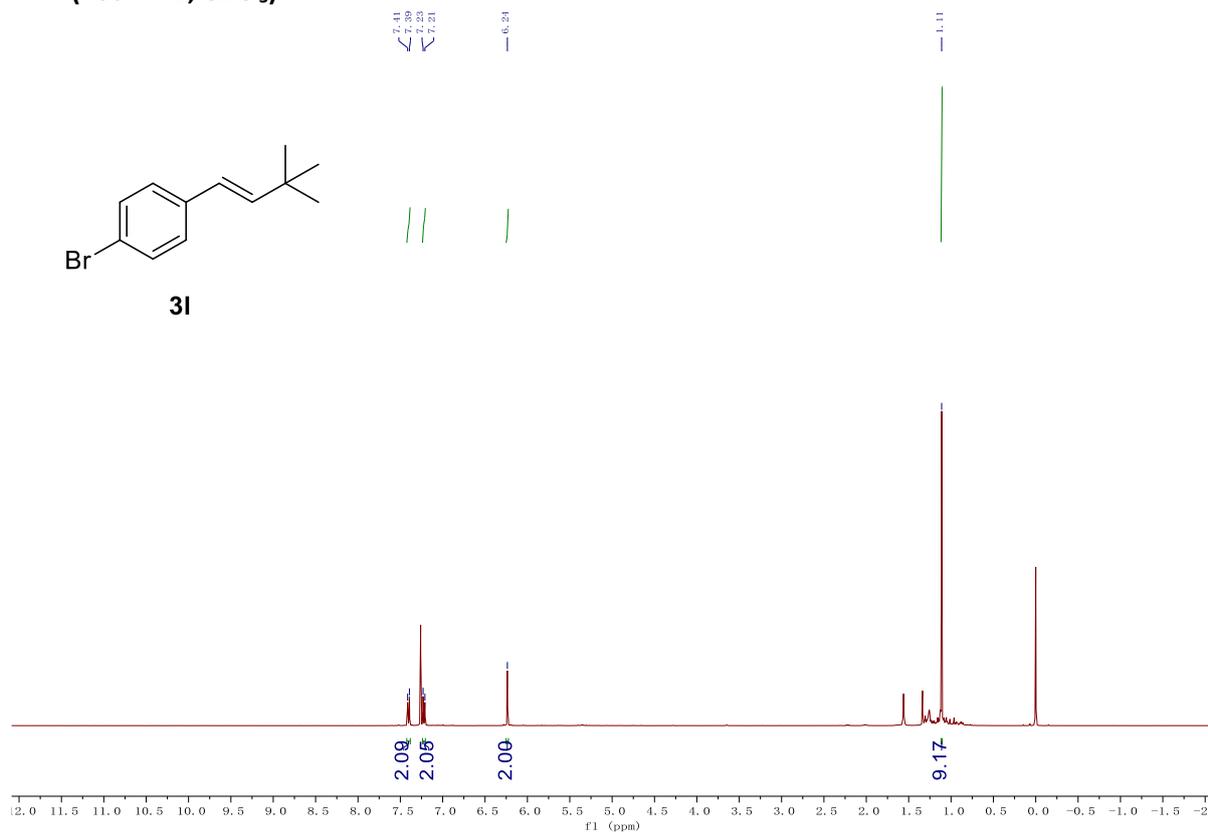
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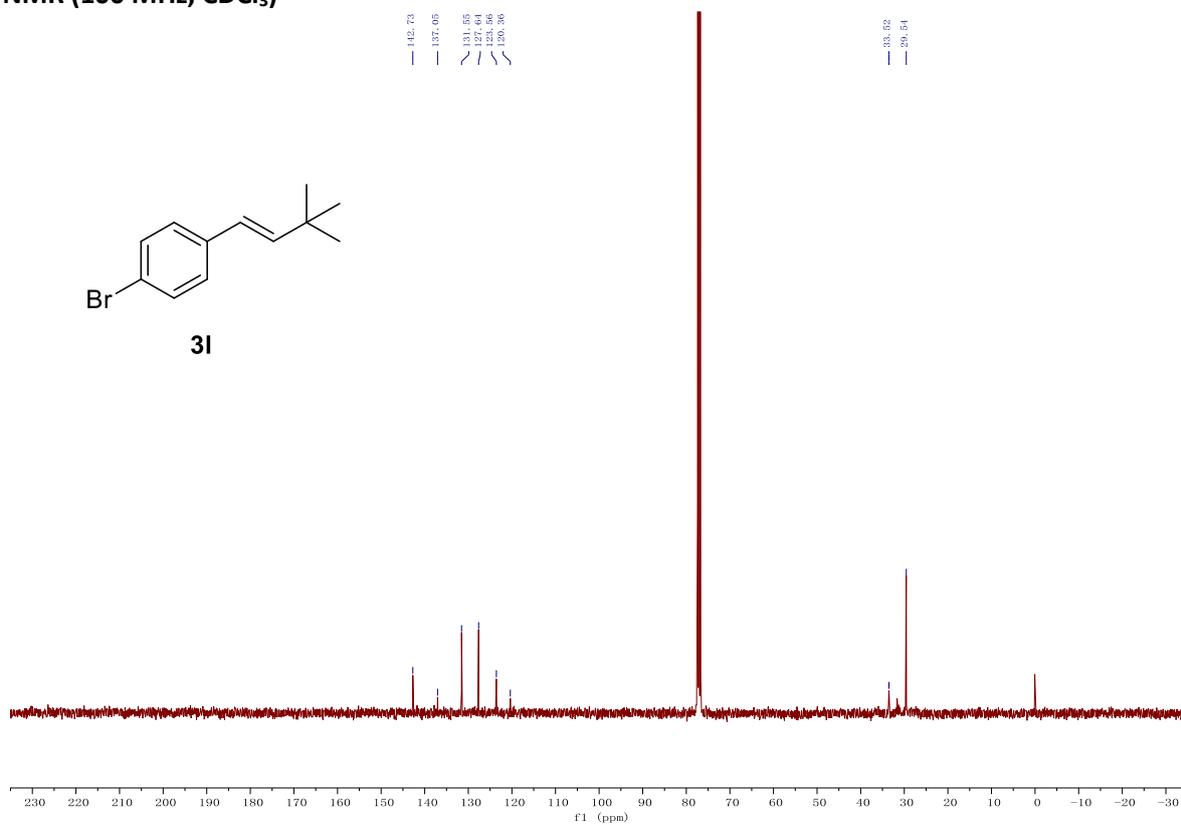
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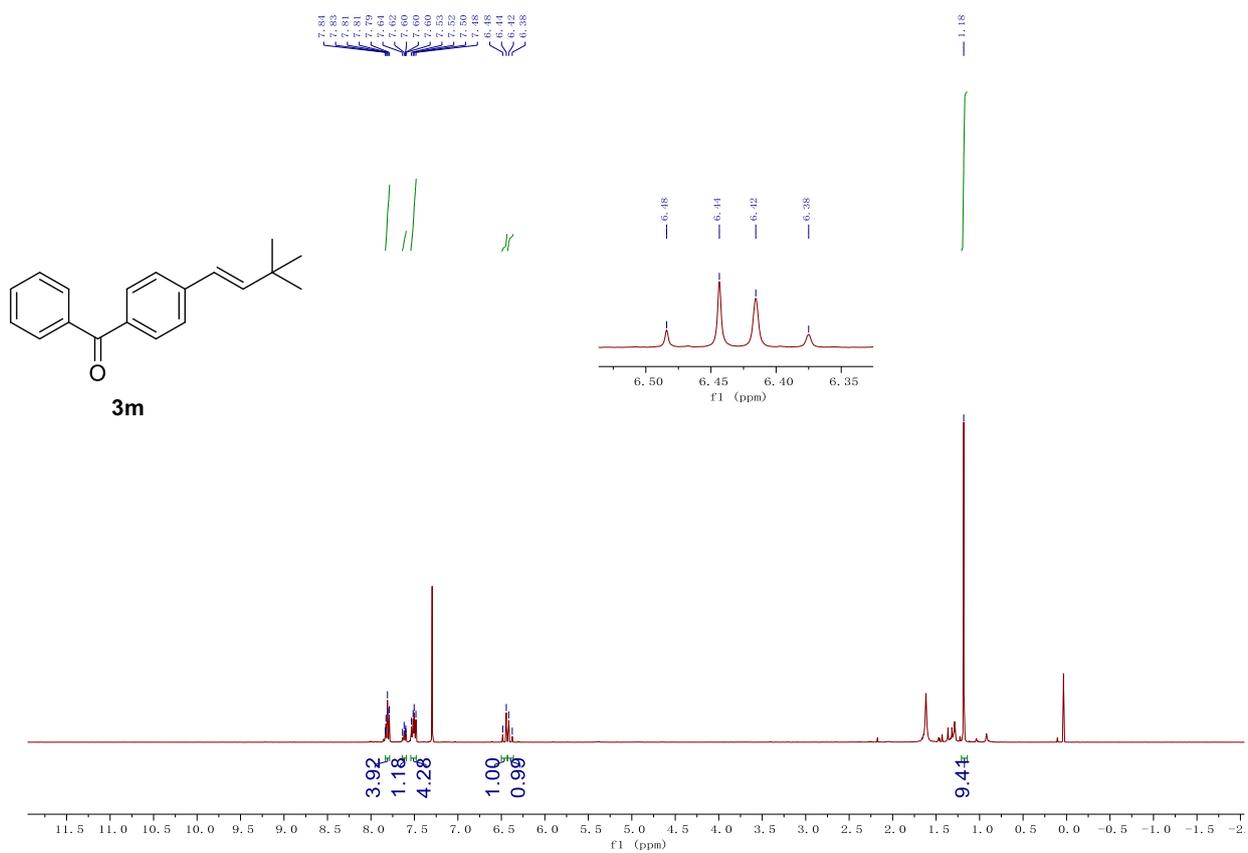
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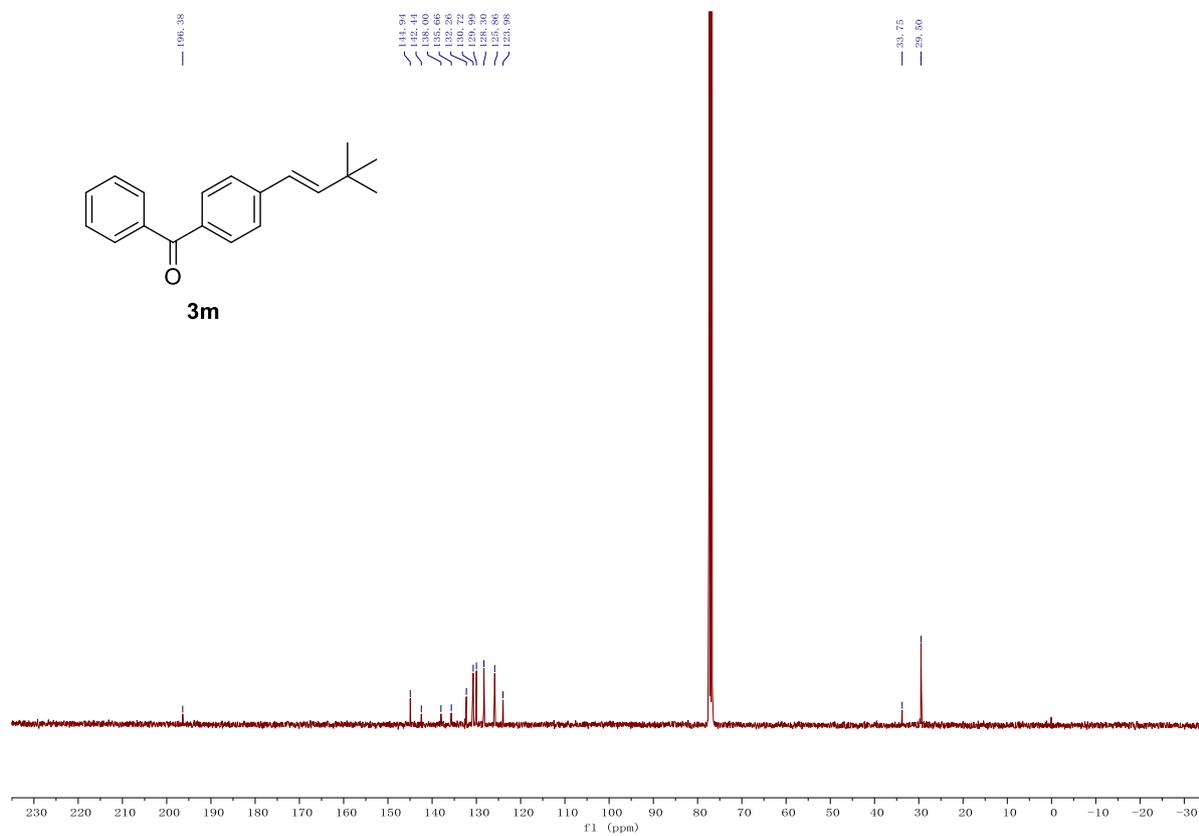
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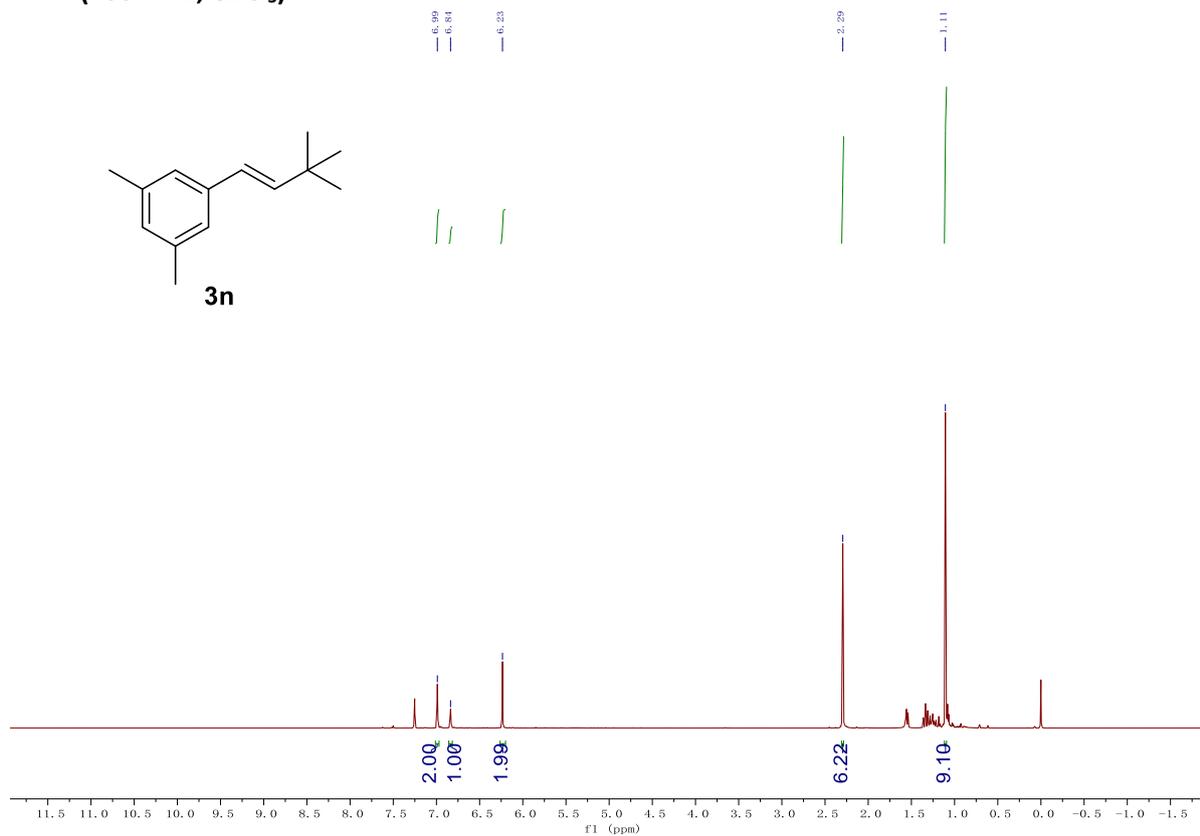
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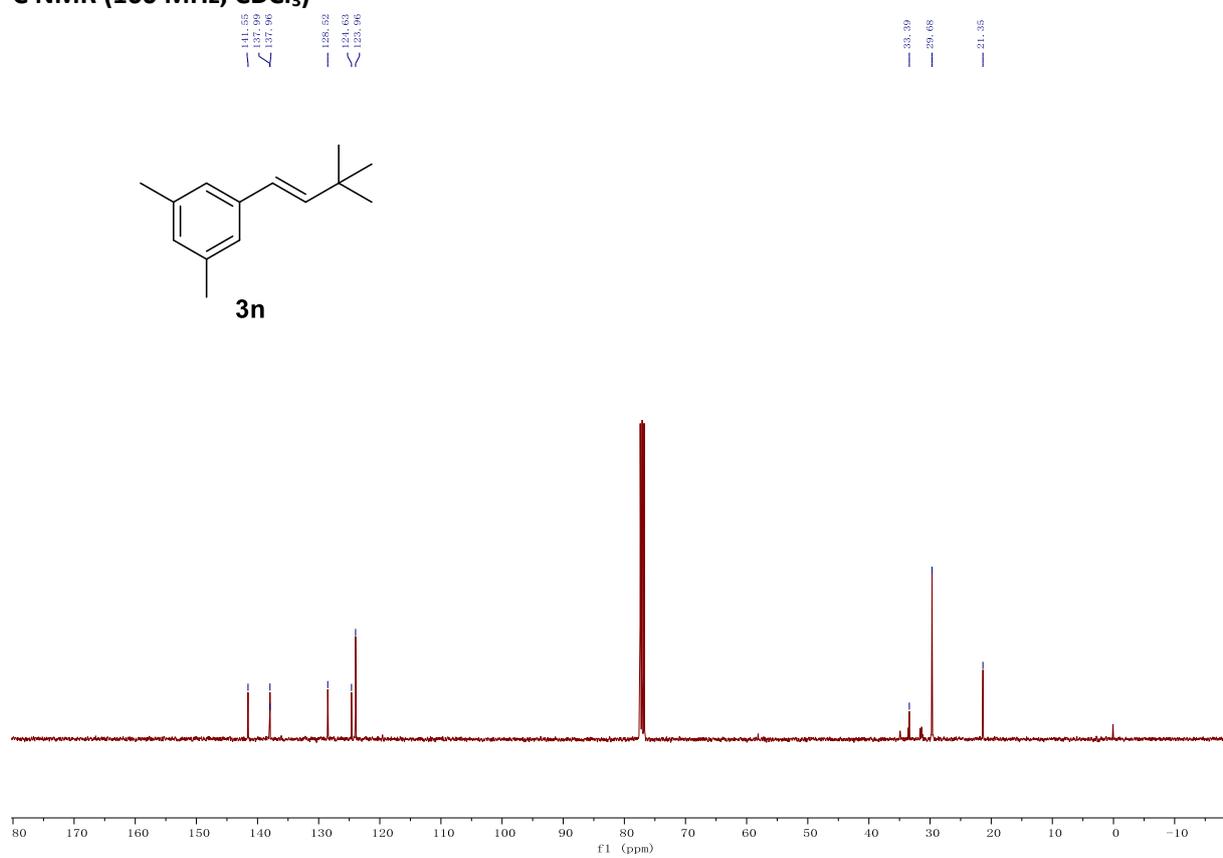
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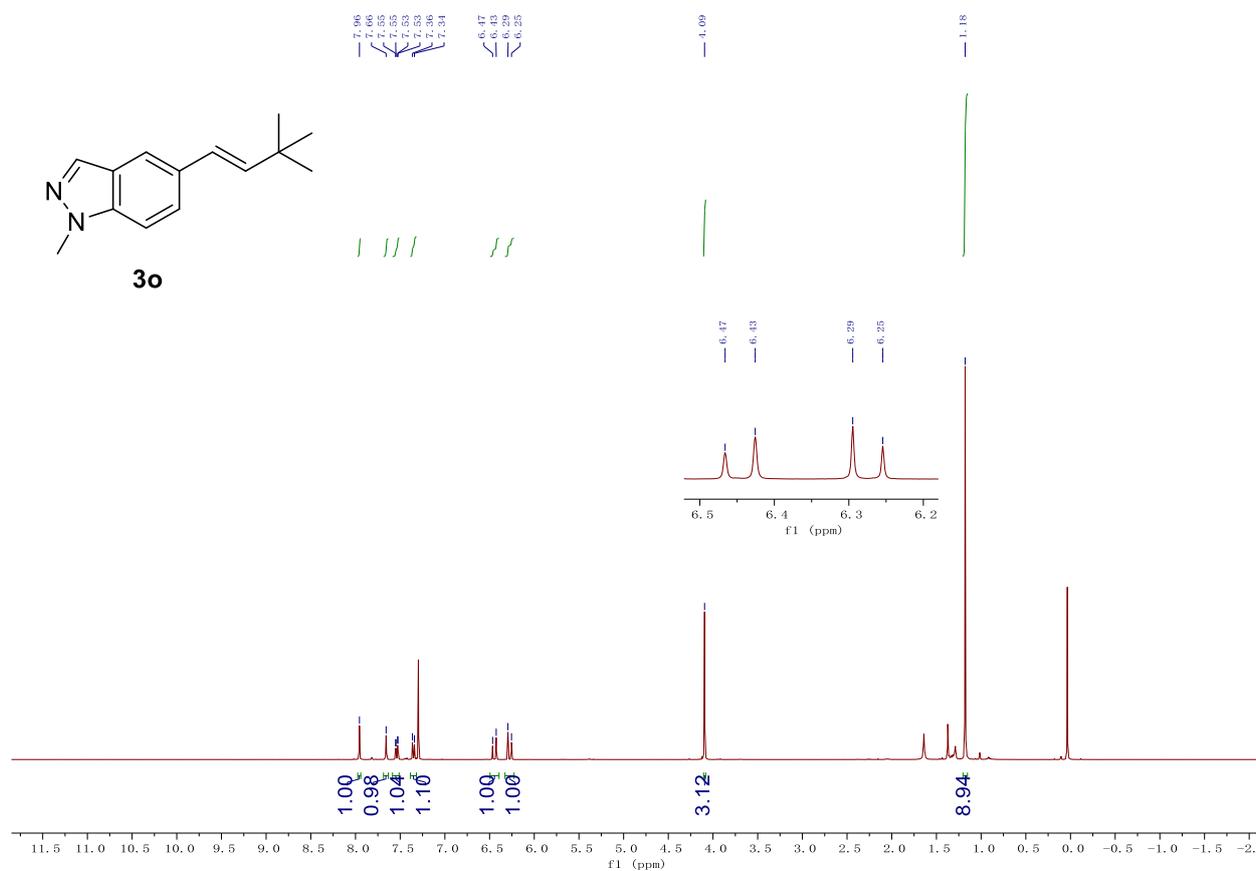
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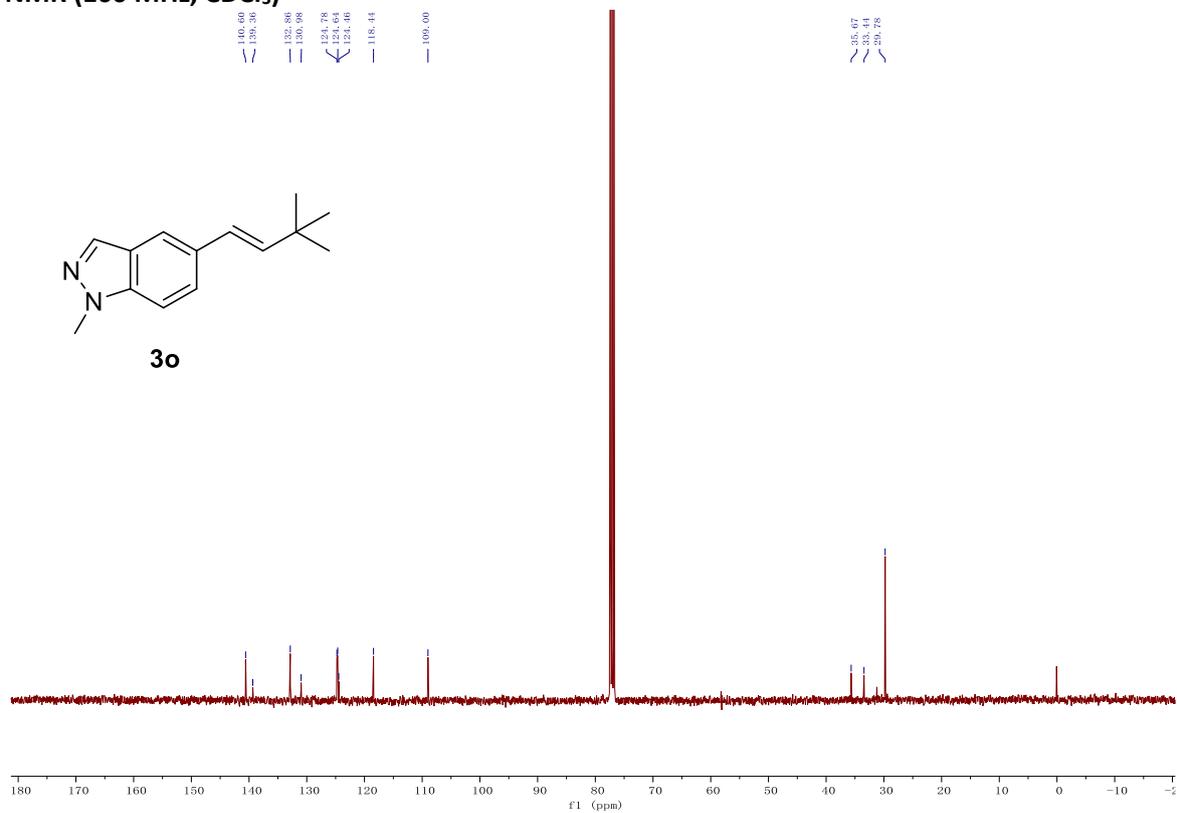
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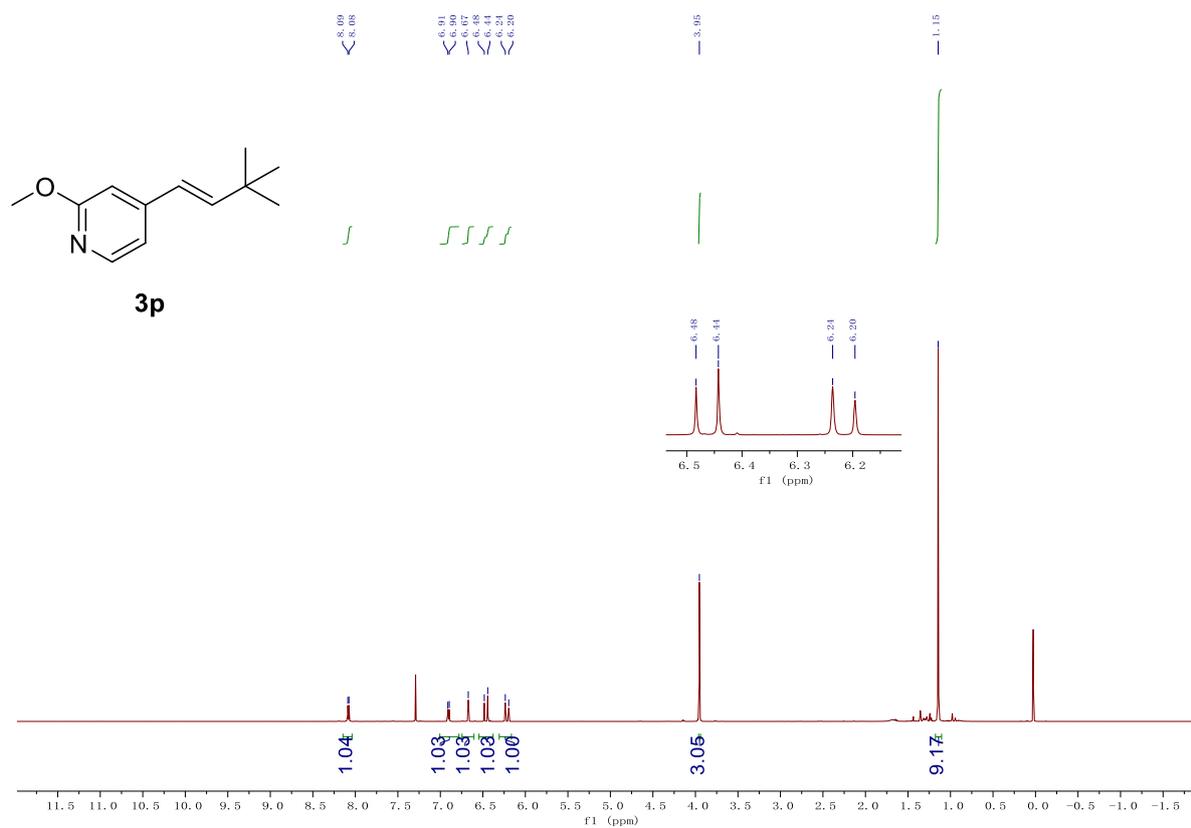
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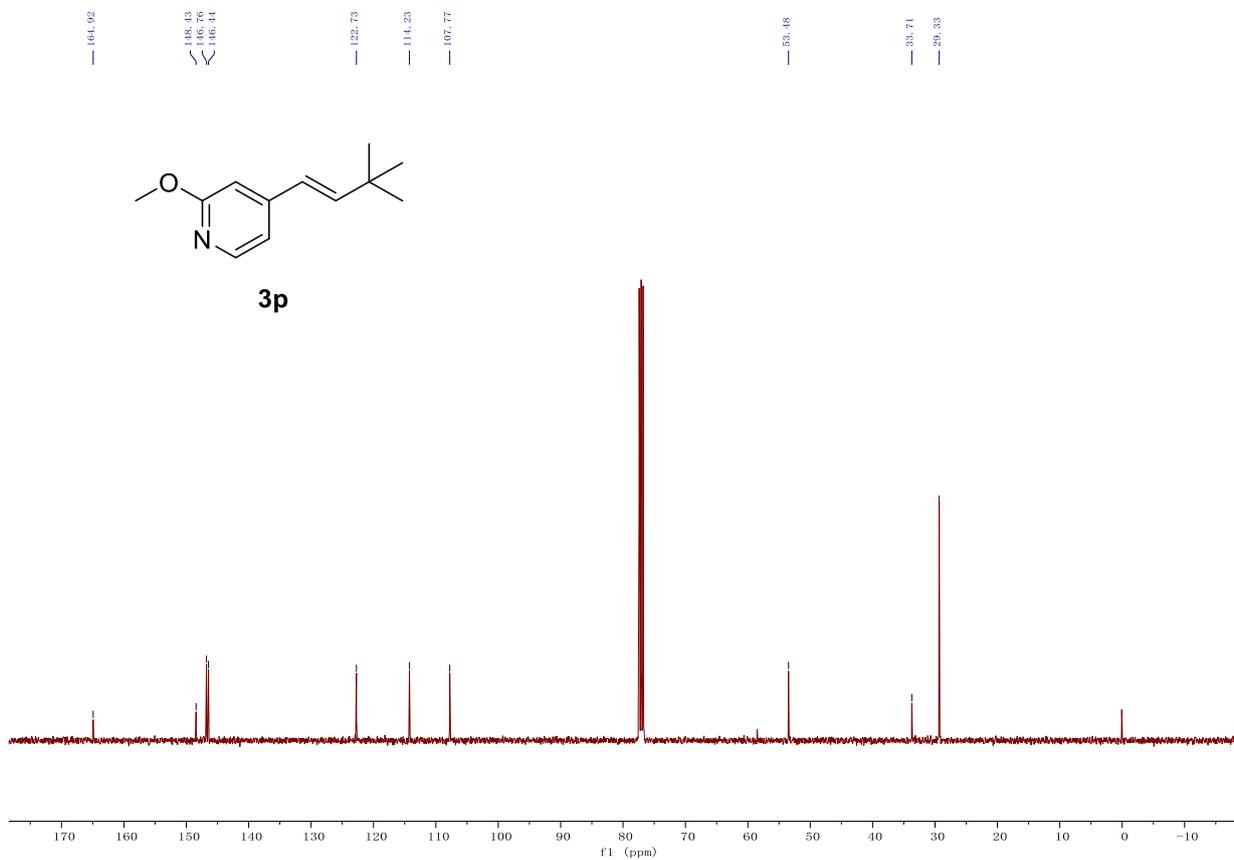
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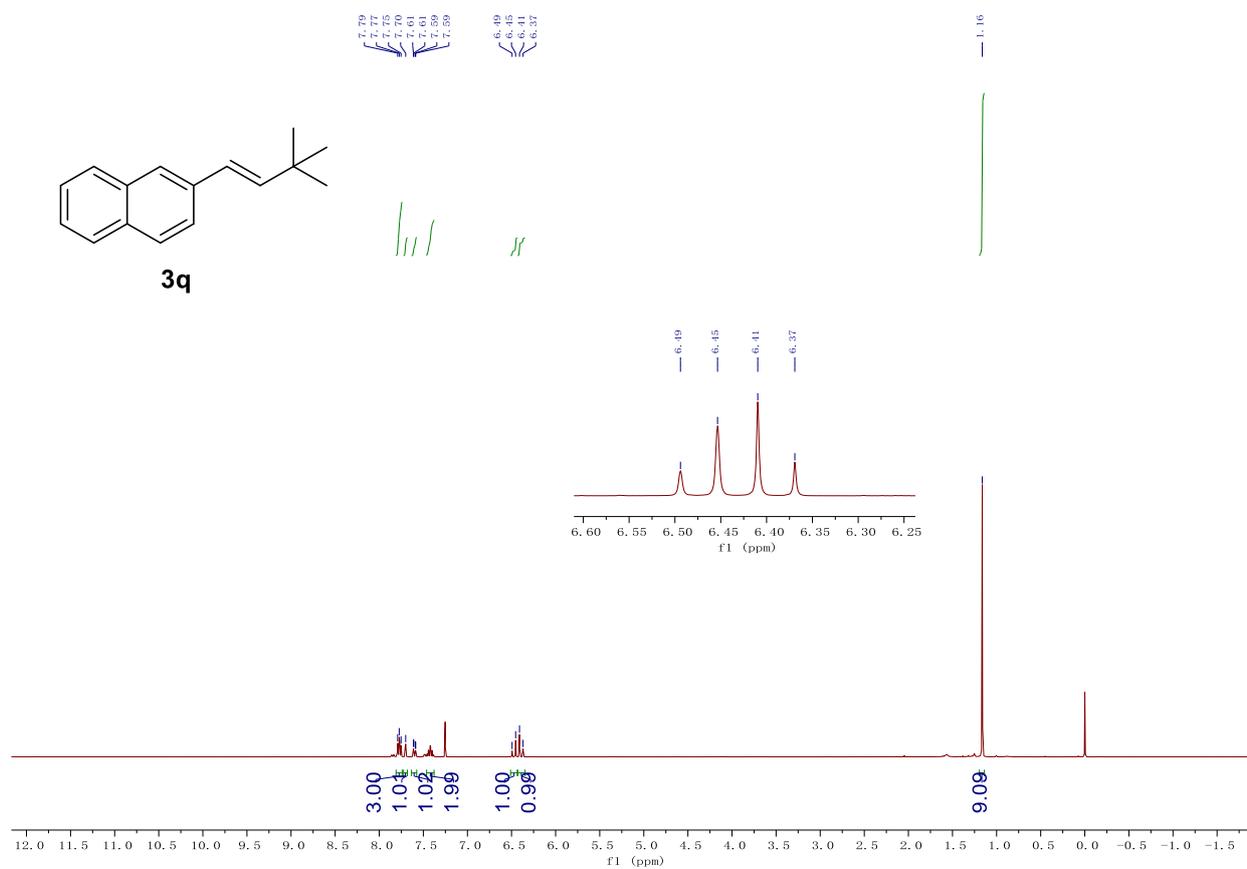
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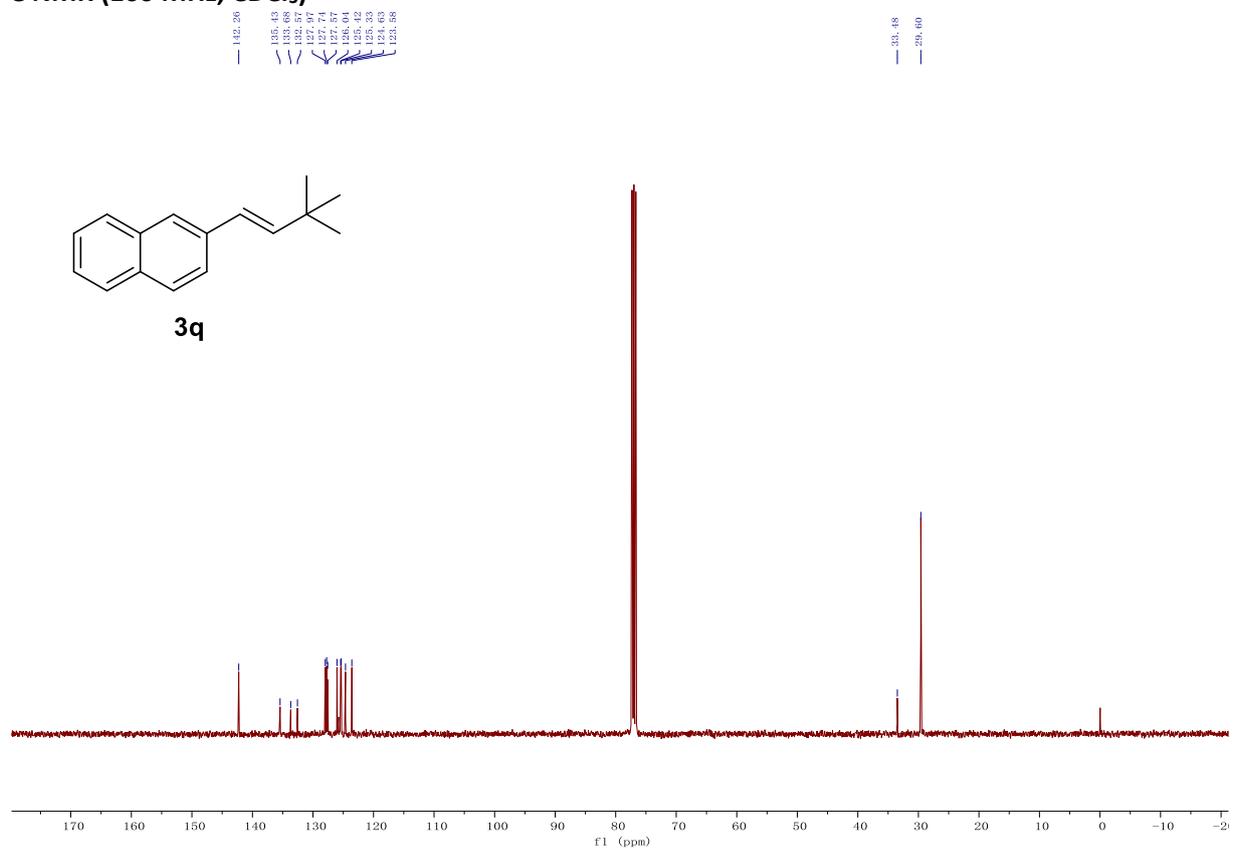
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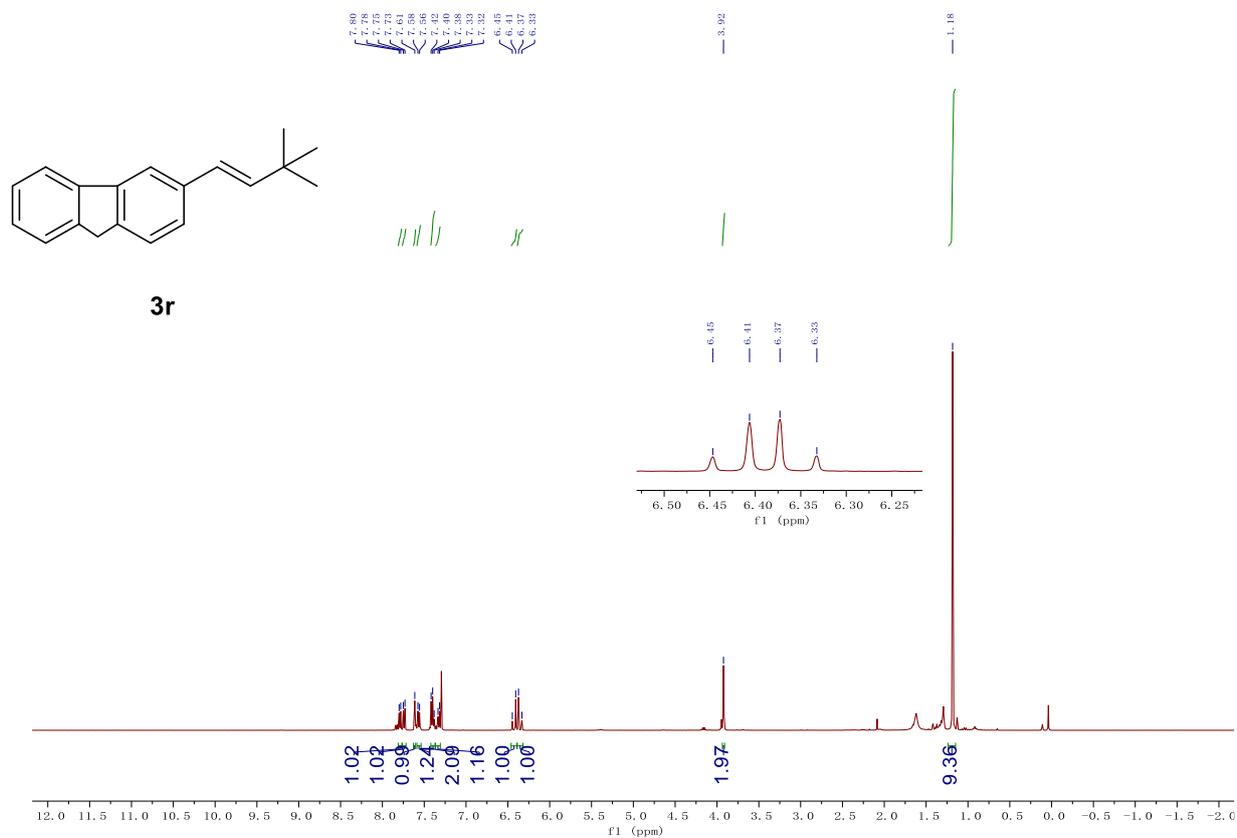
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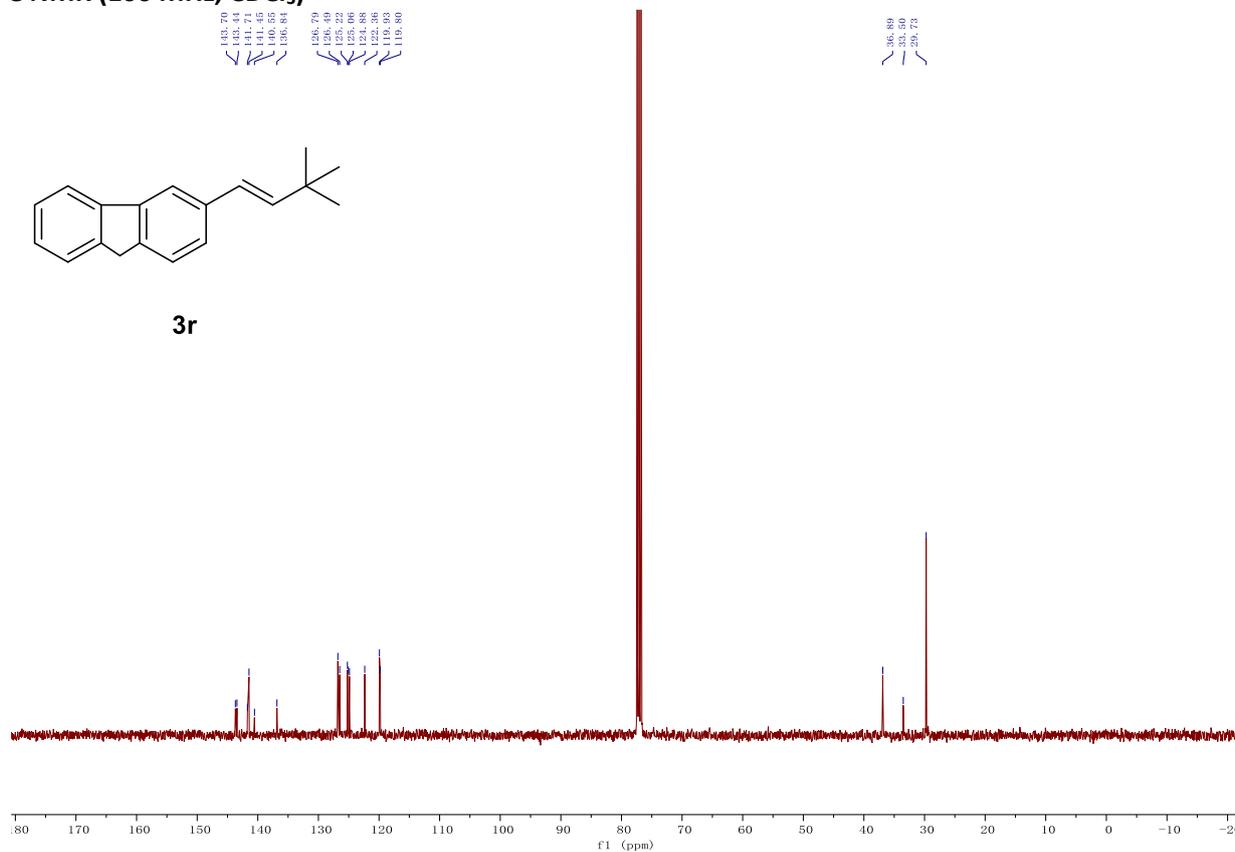
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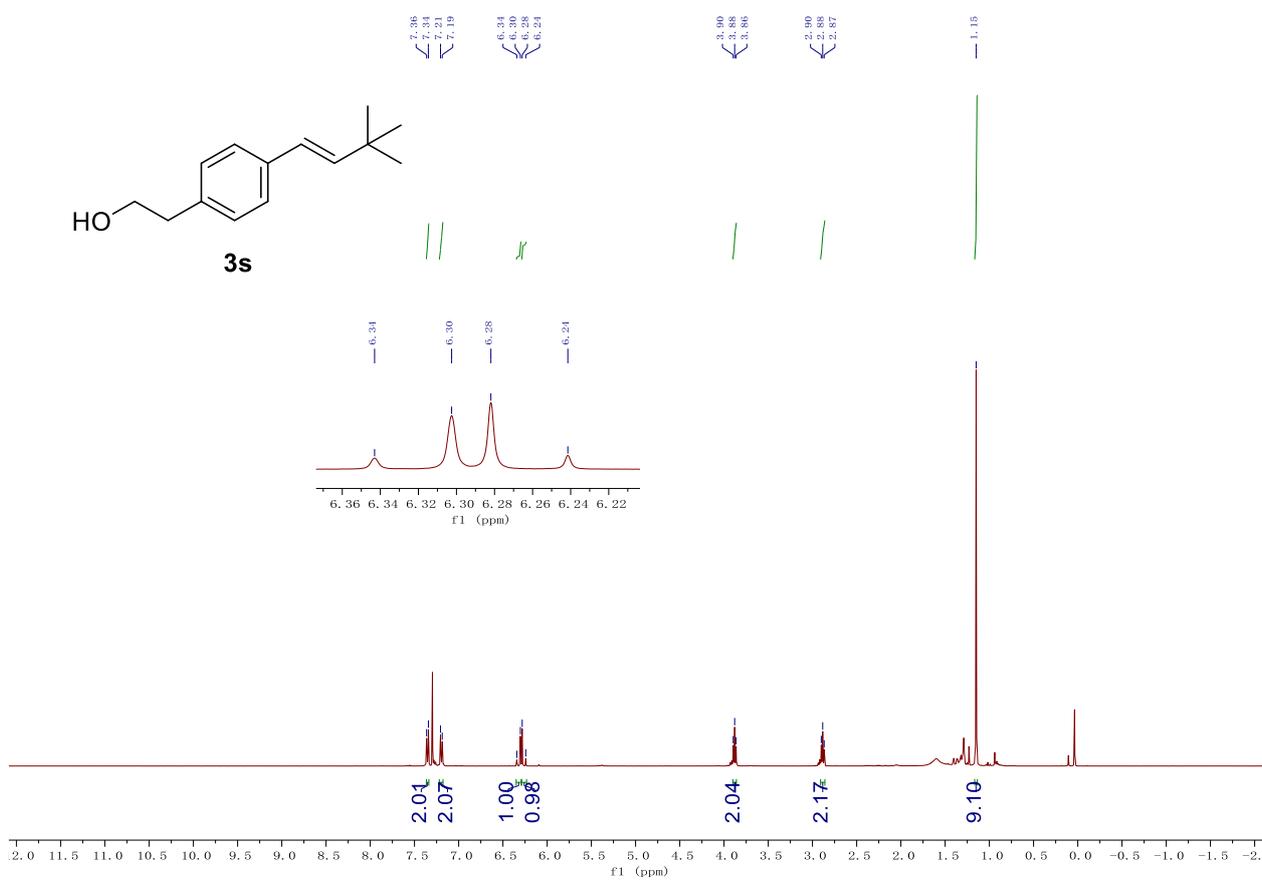
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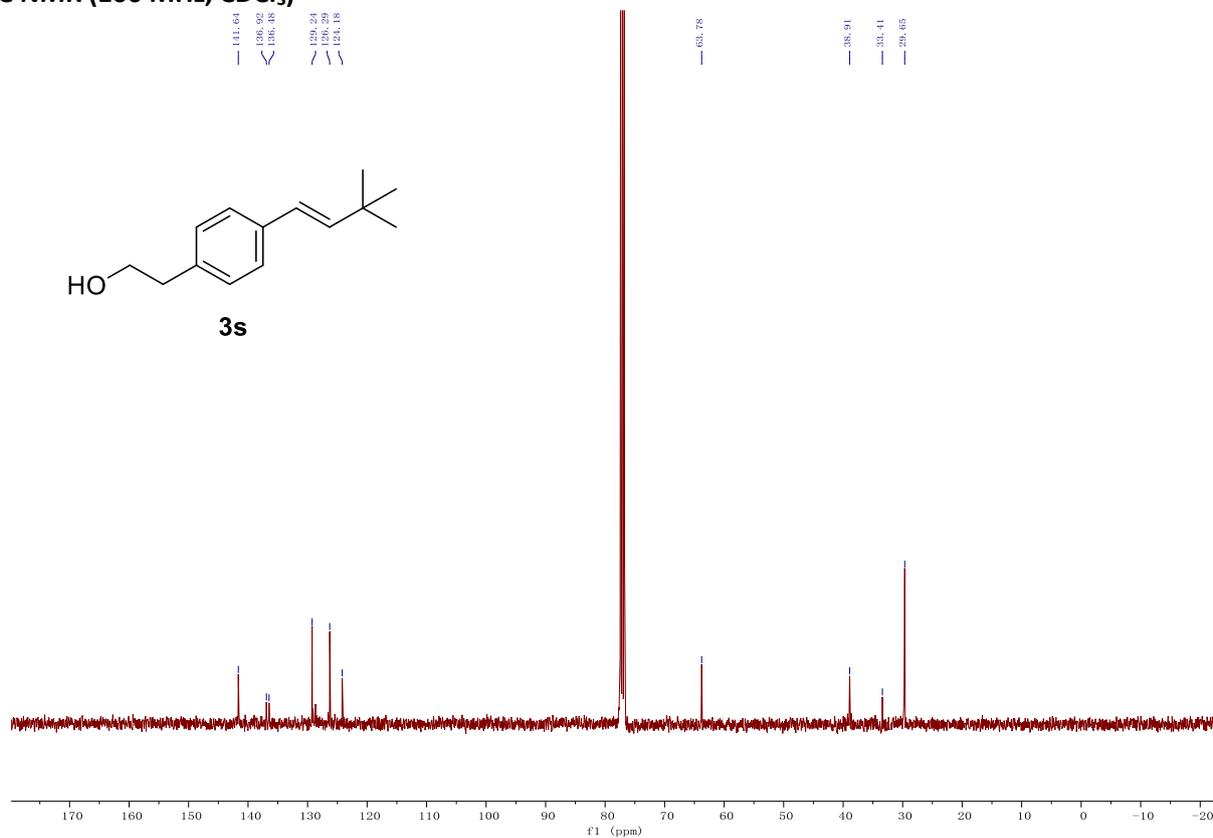
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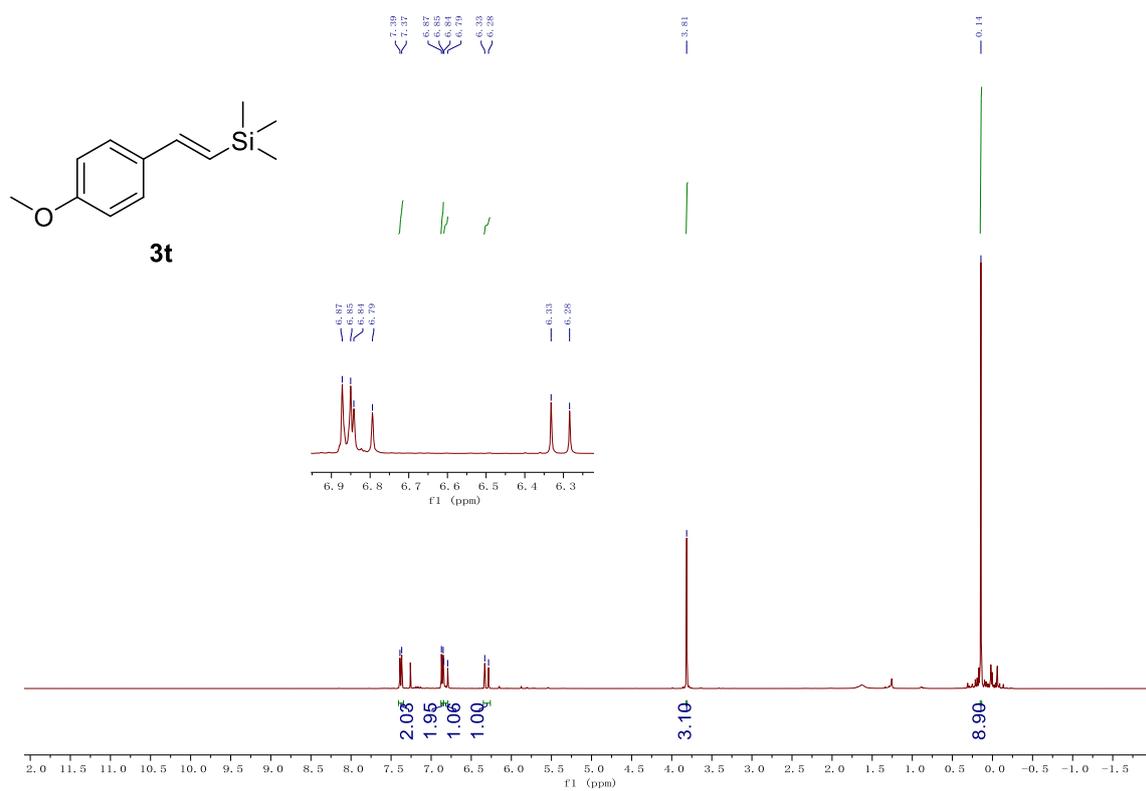
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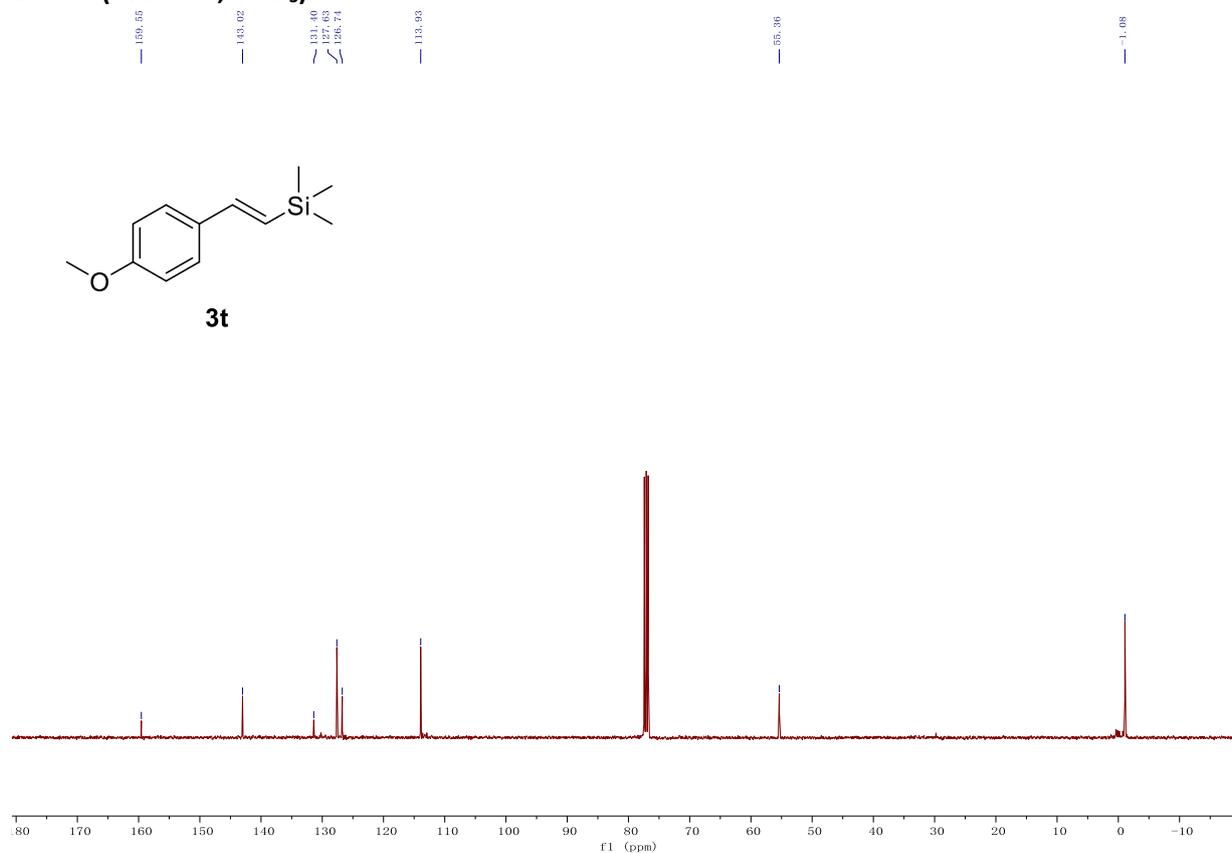
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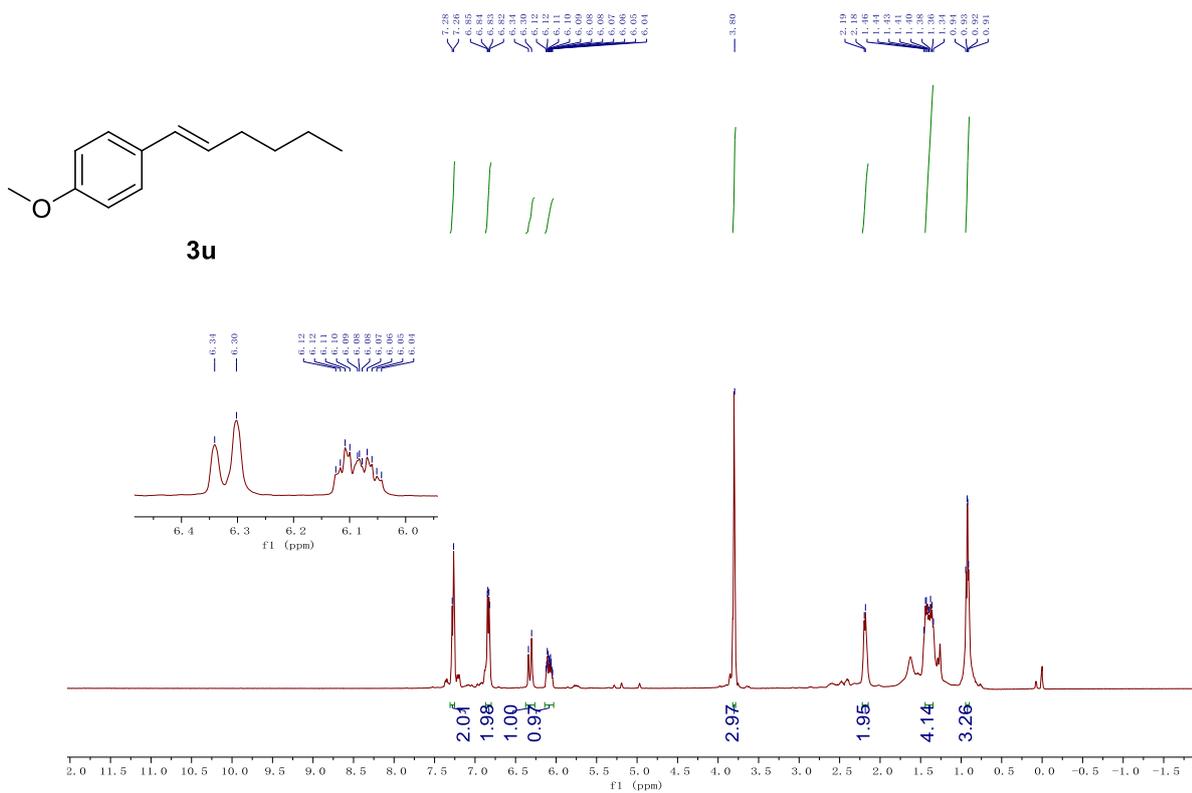
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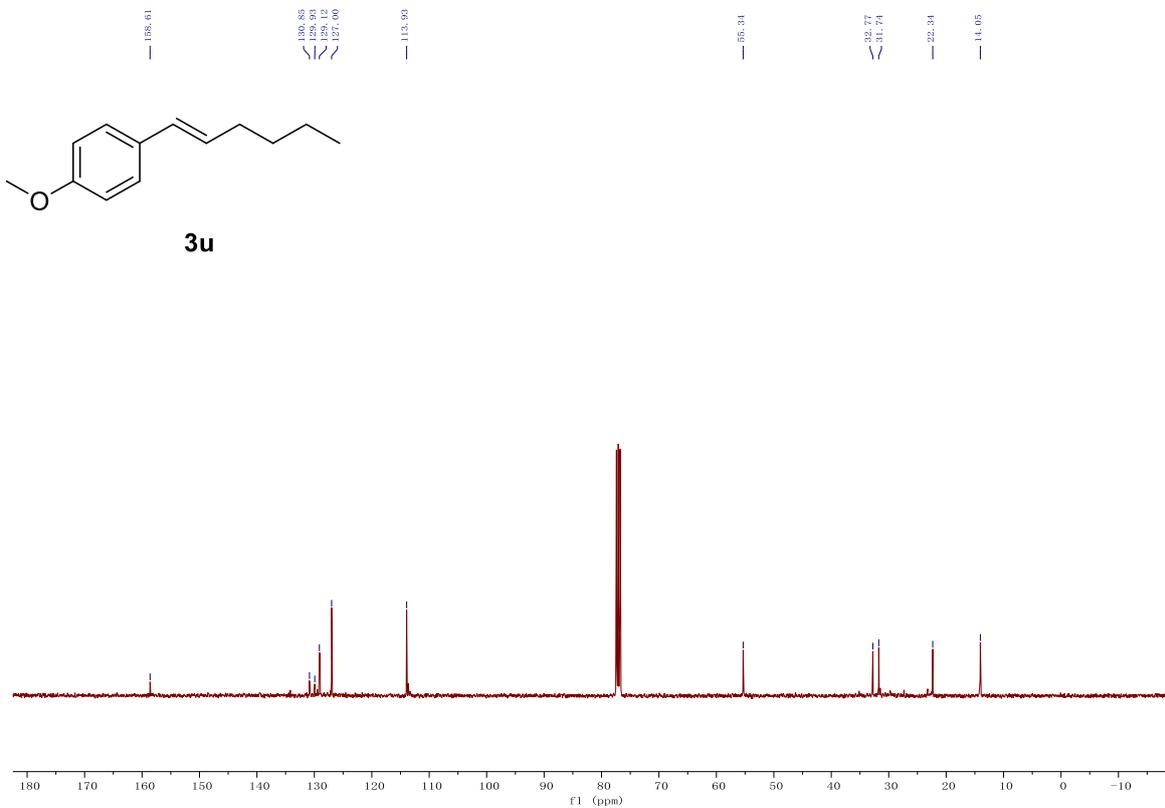
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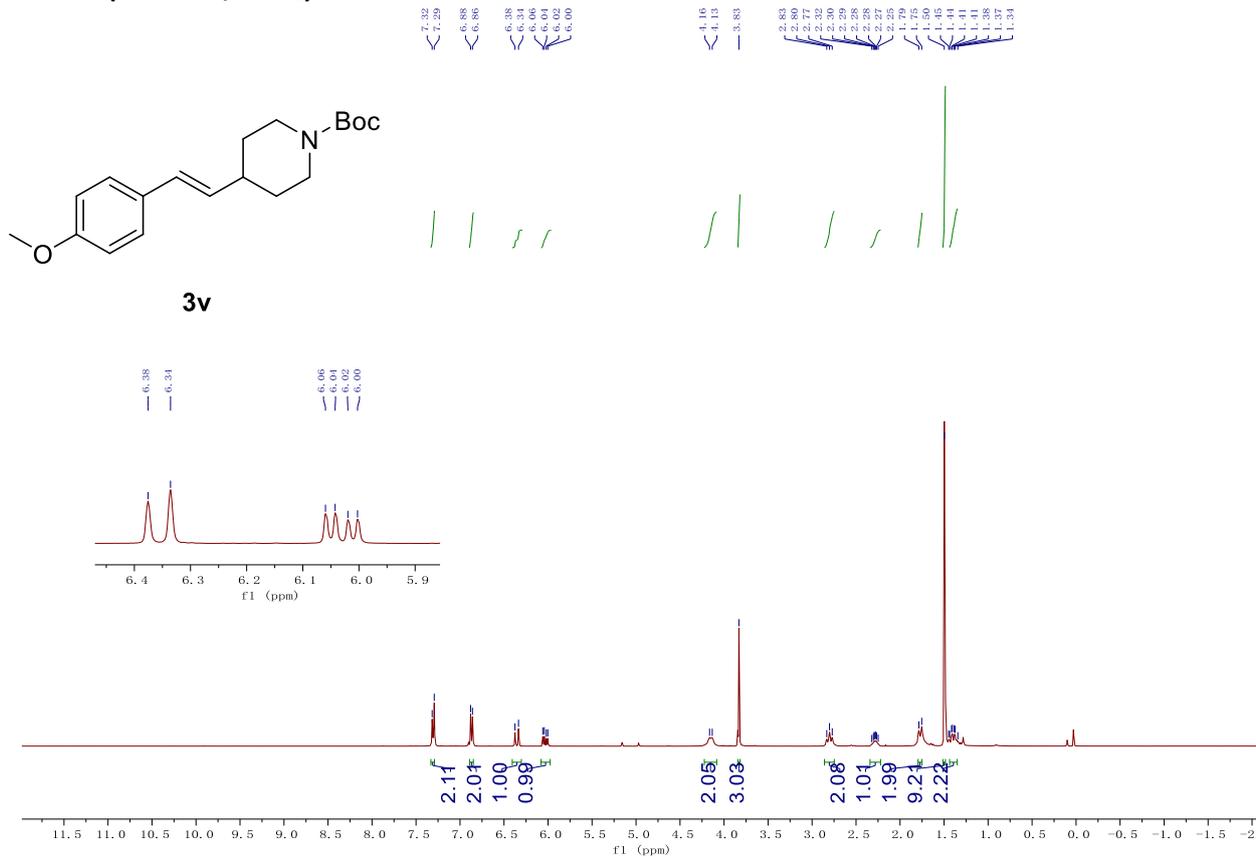
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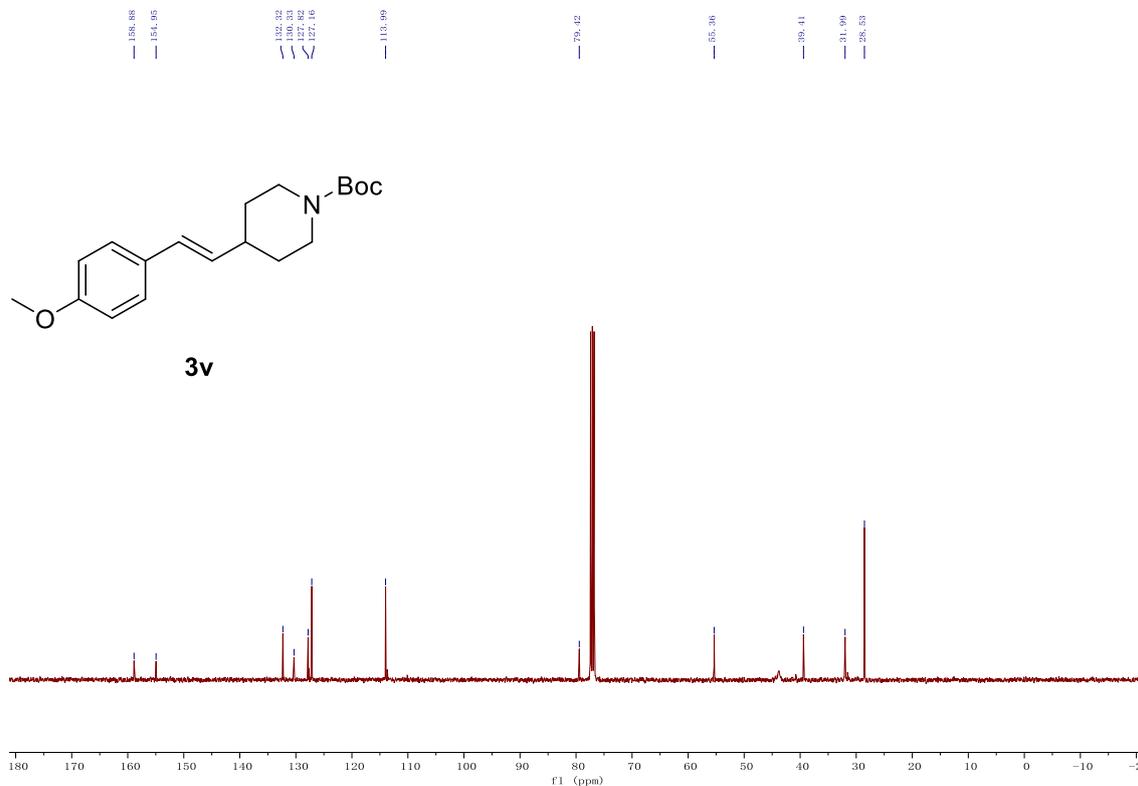
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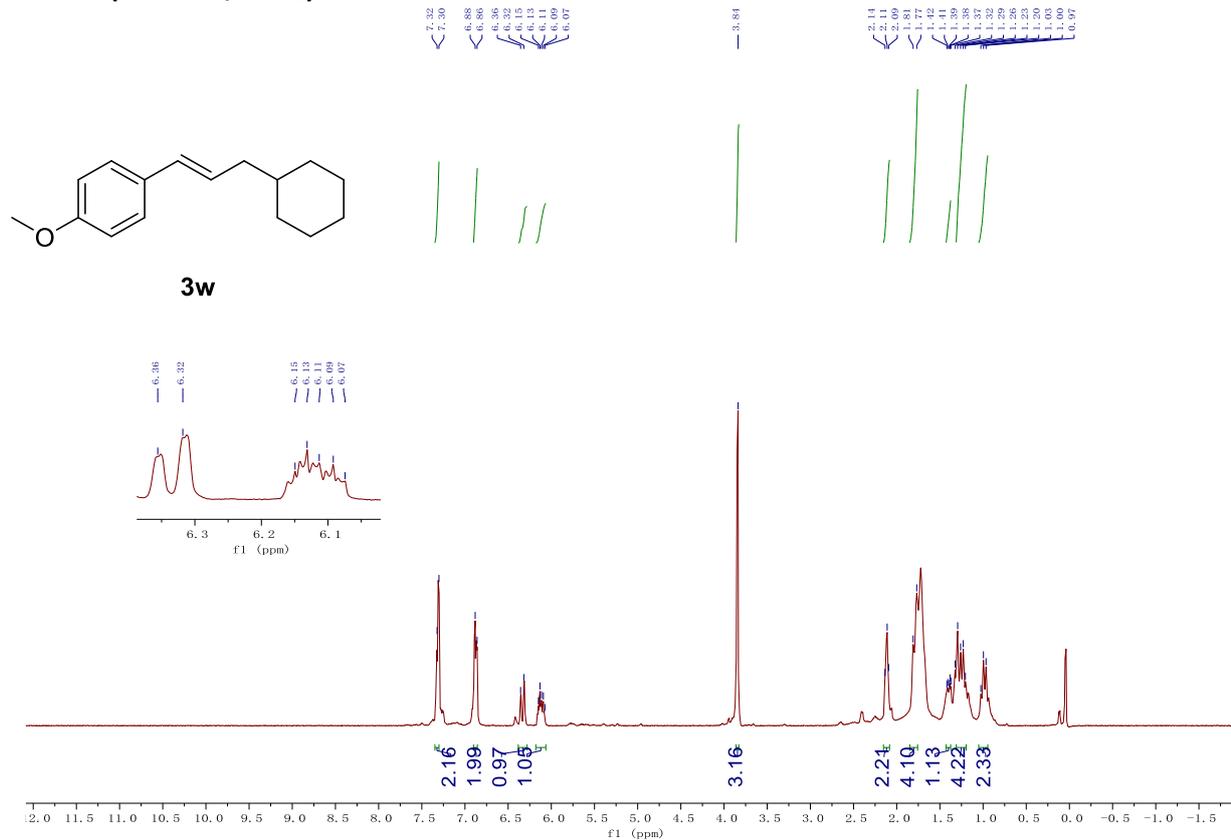
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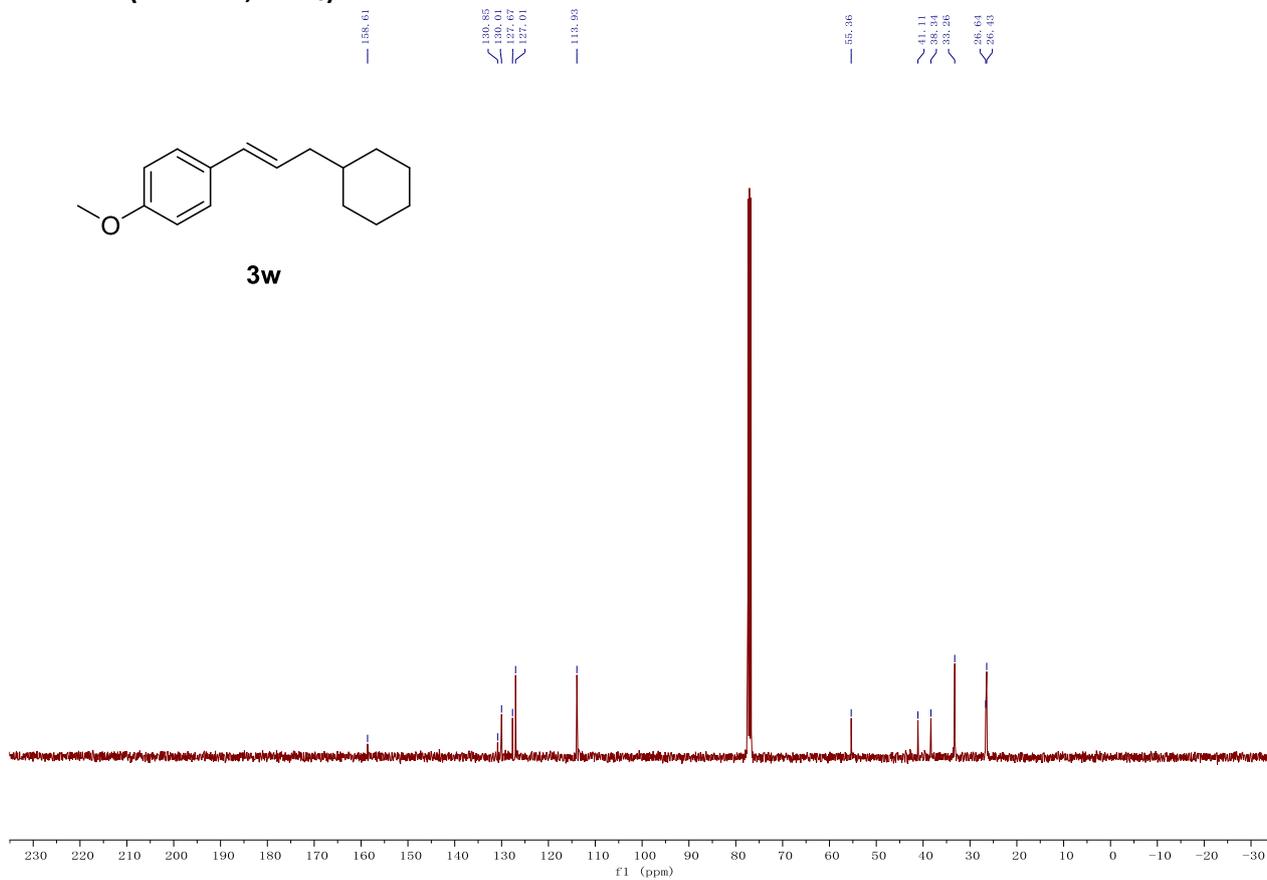
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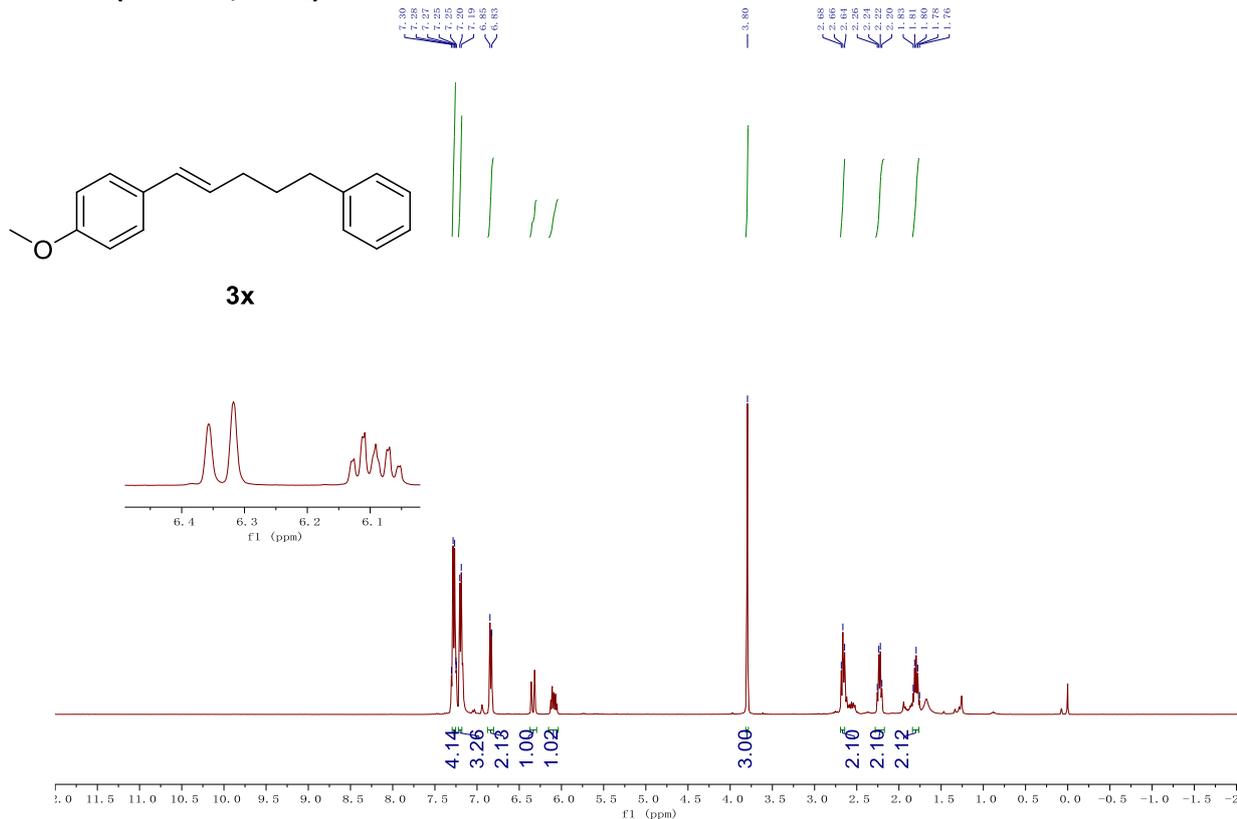
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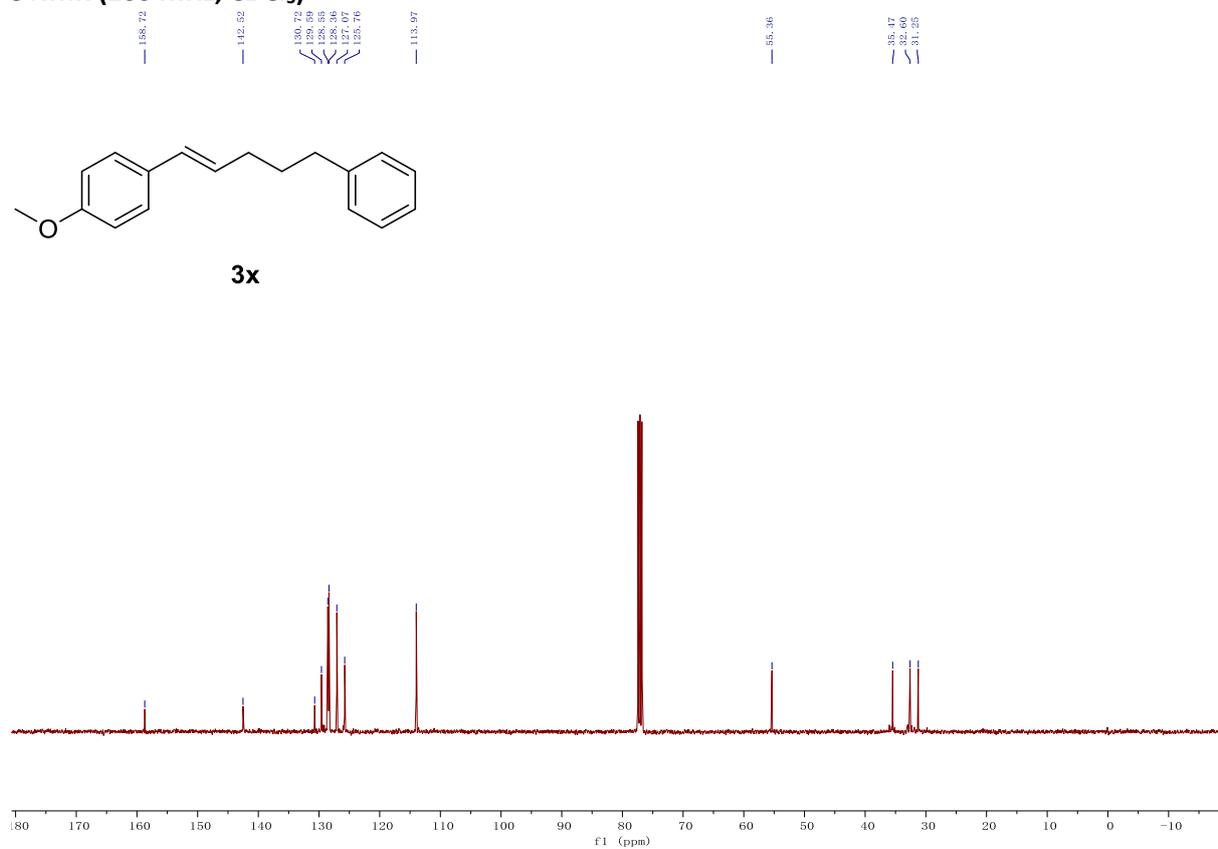
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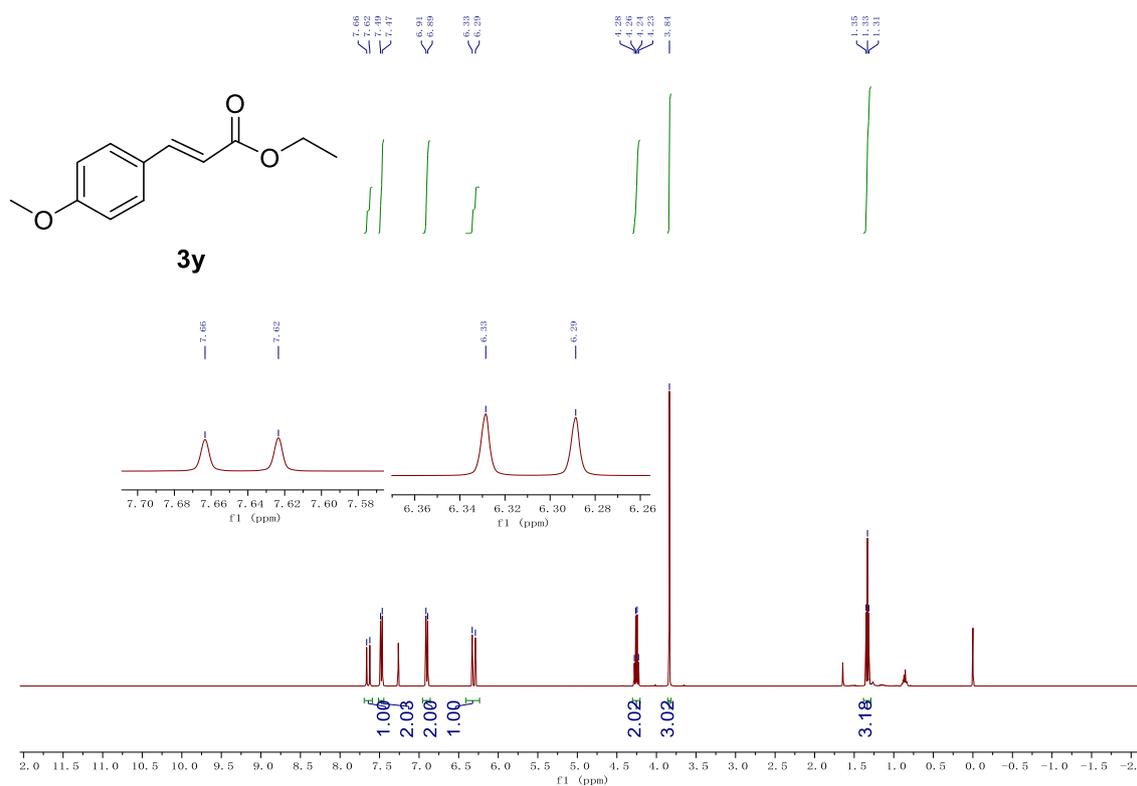
3x: ¹H NMR (400 MHz, CDCl₃)



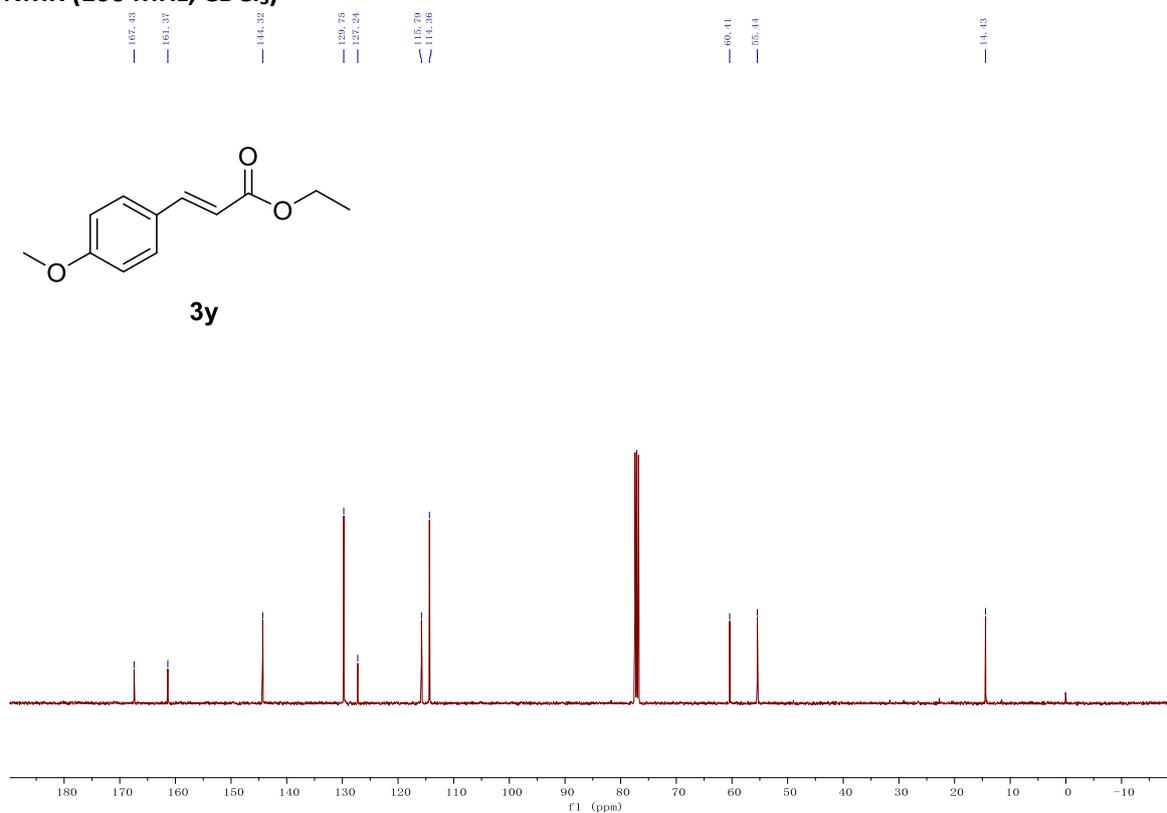
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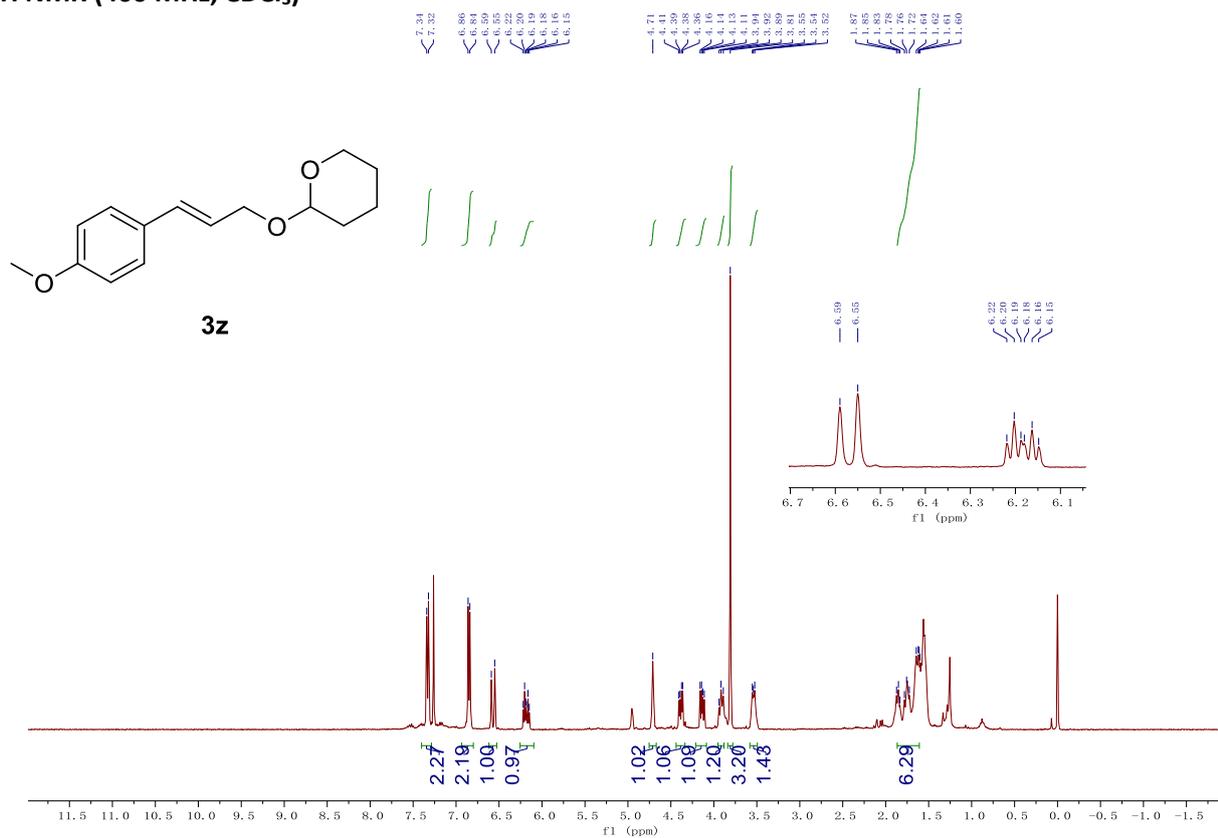
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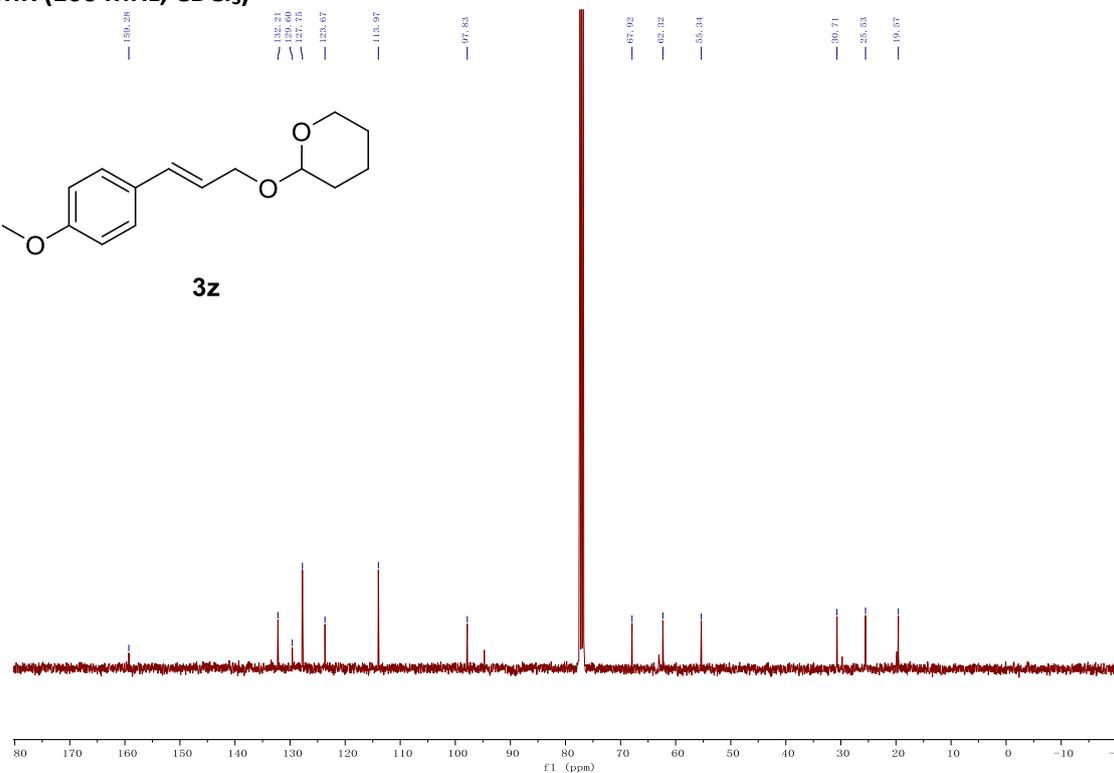
3y: ¹³C NMR (100 MHz, CDCl₃)



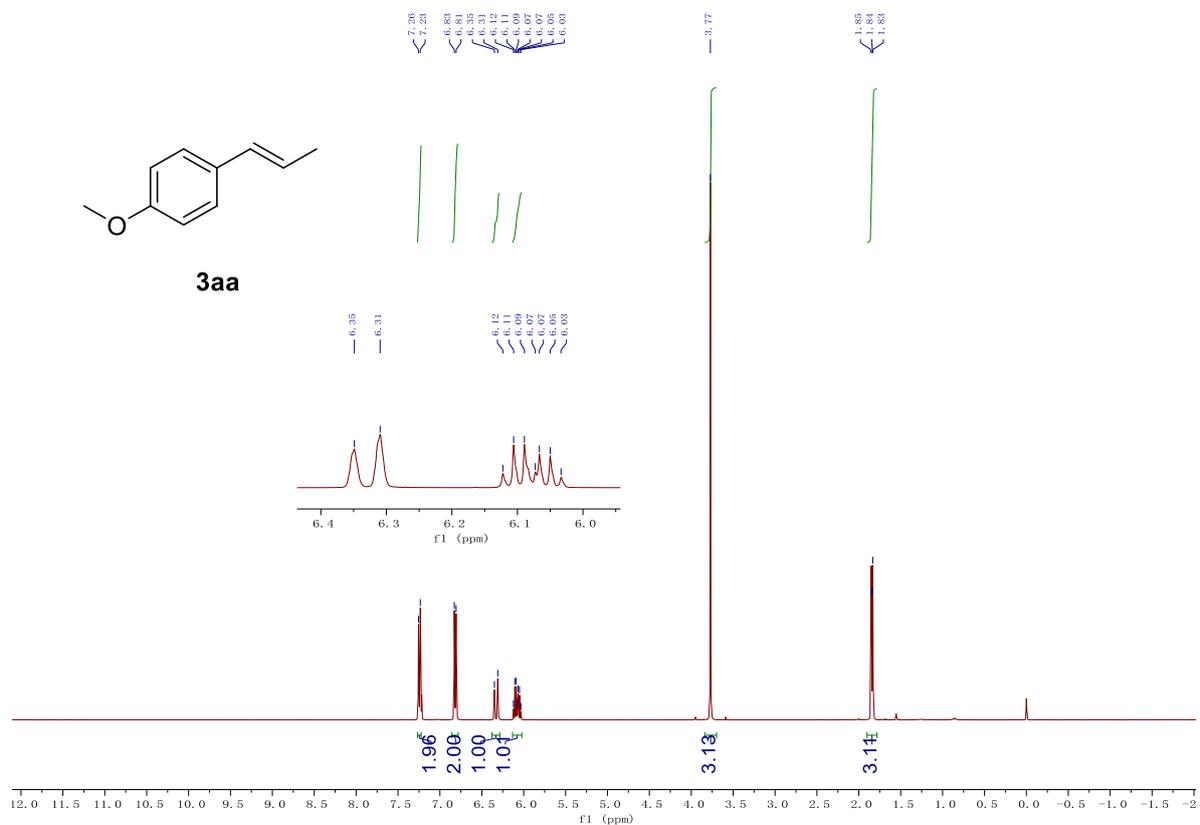
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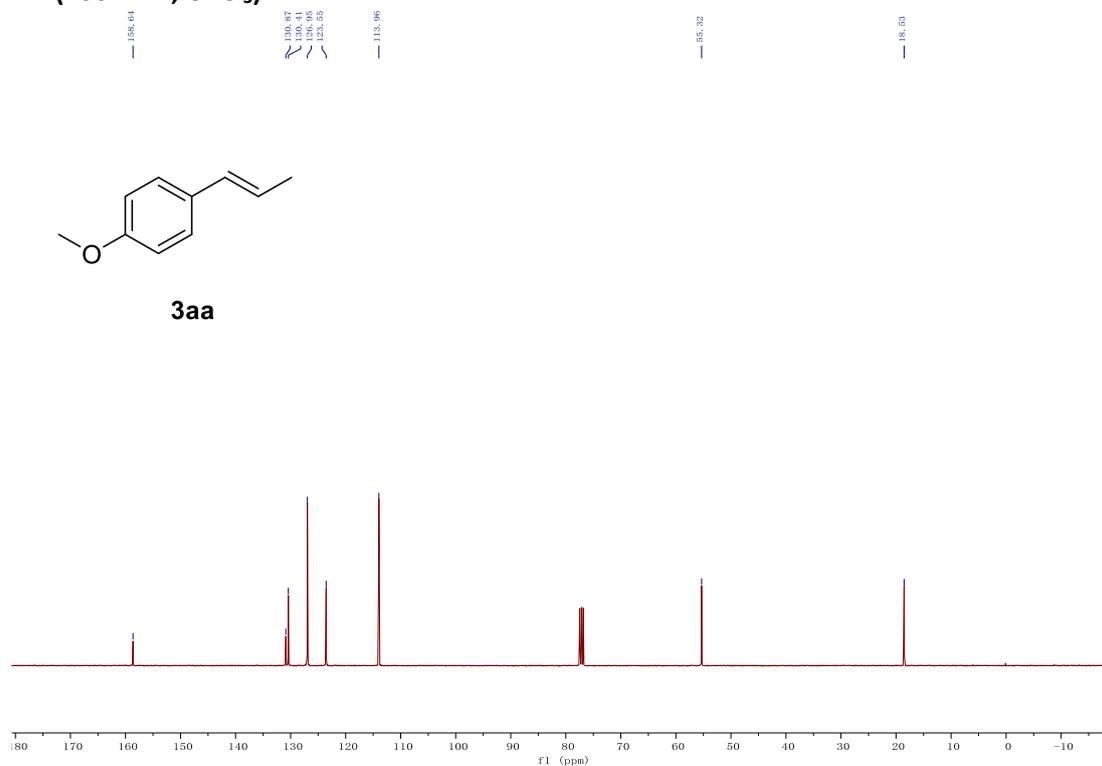
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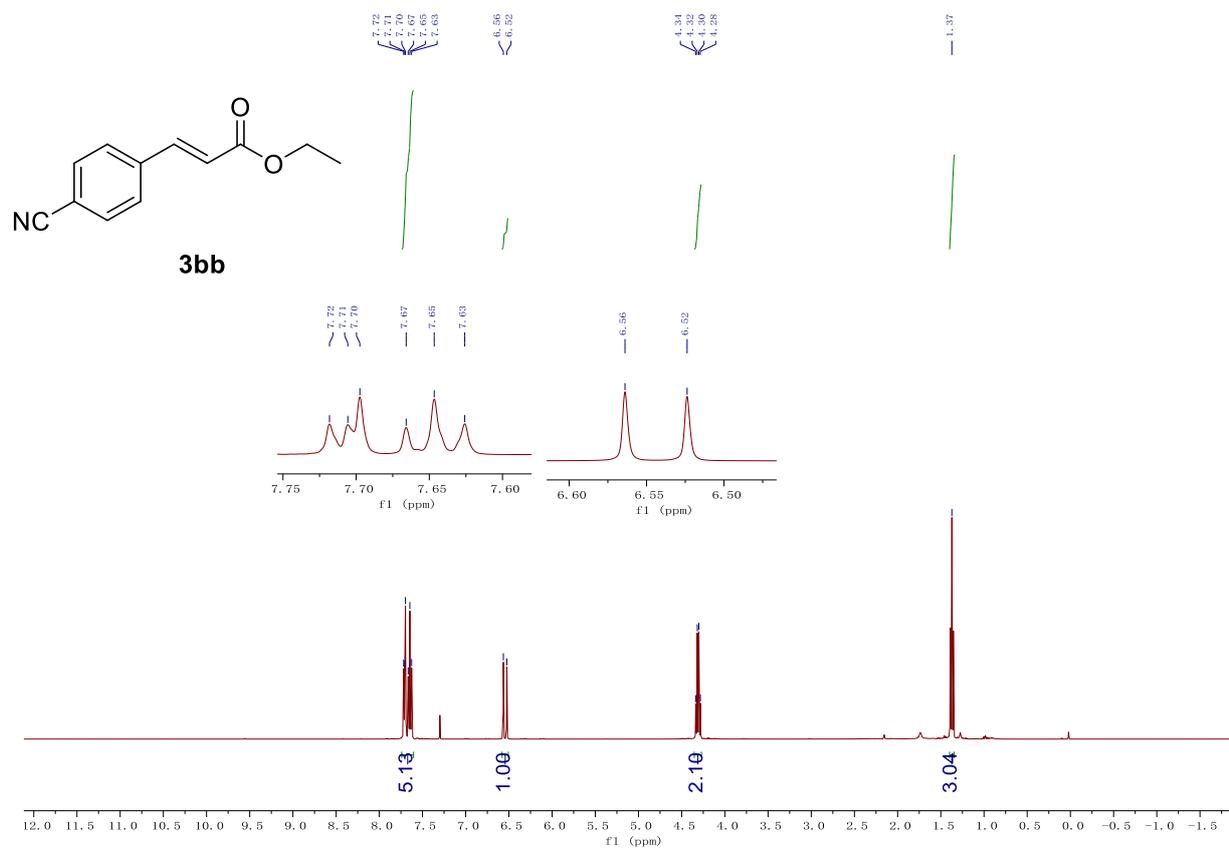
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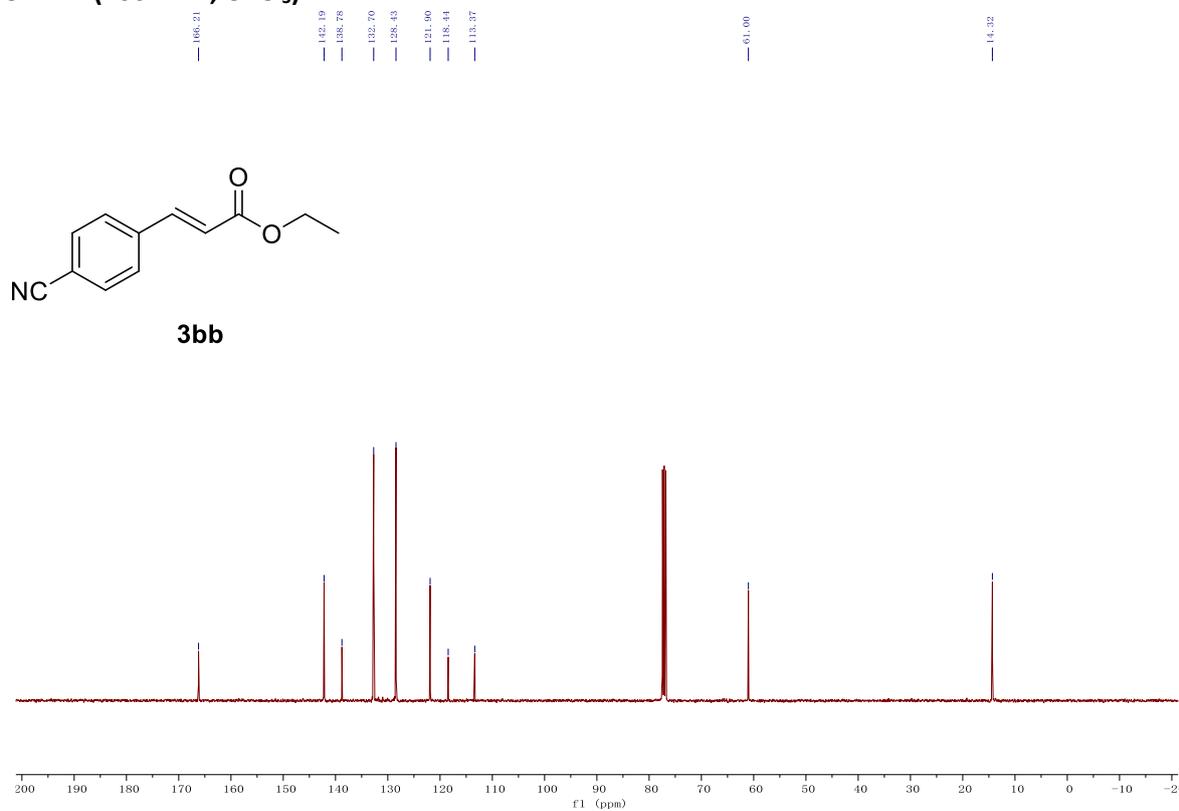
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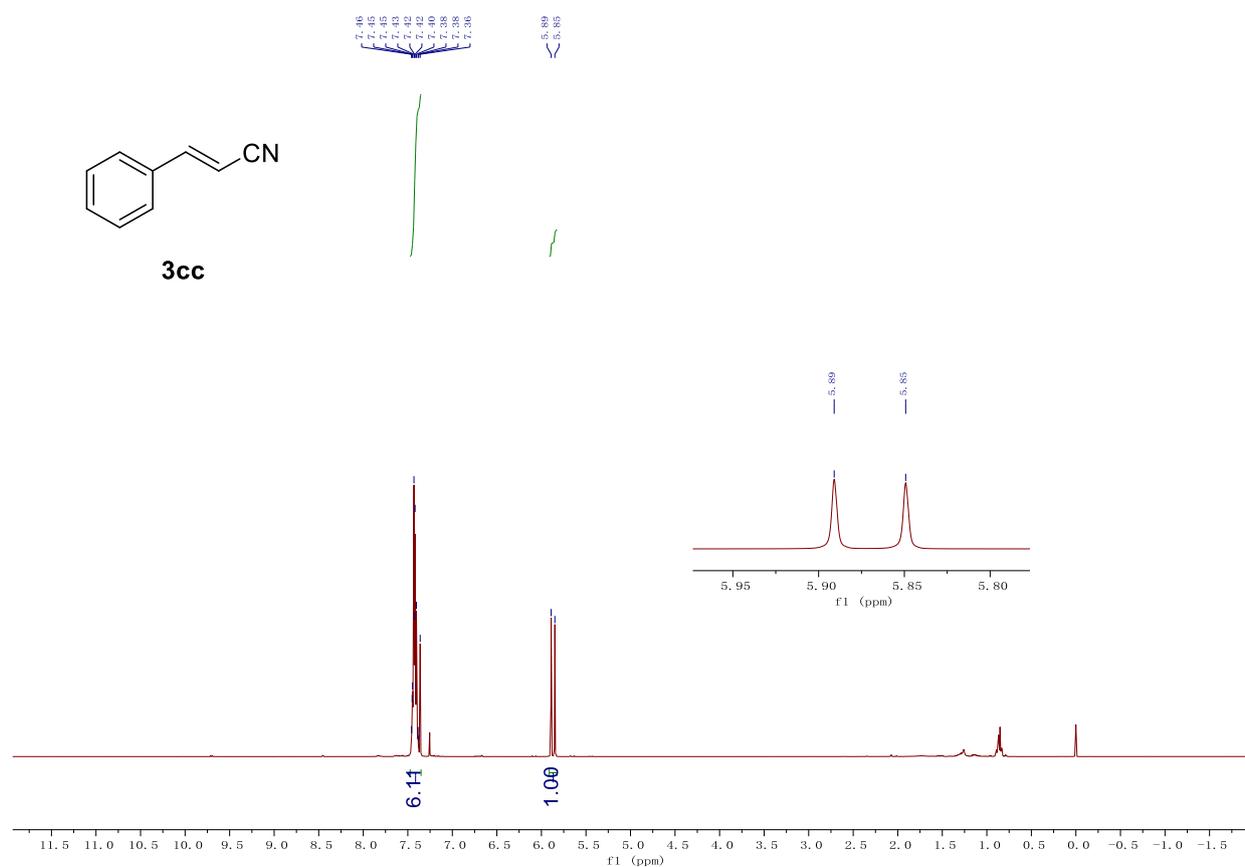
3bb: ¹H NMR (400 MHz, CDCl₃)



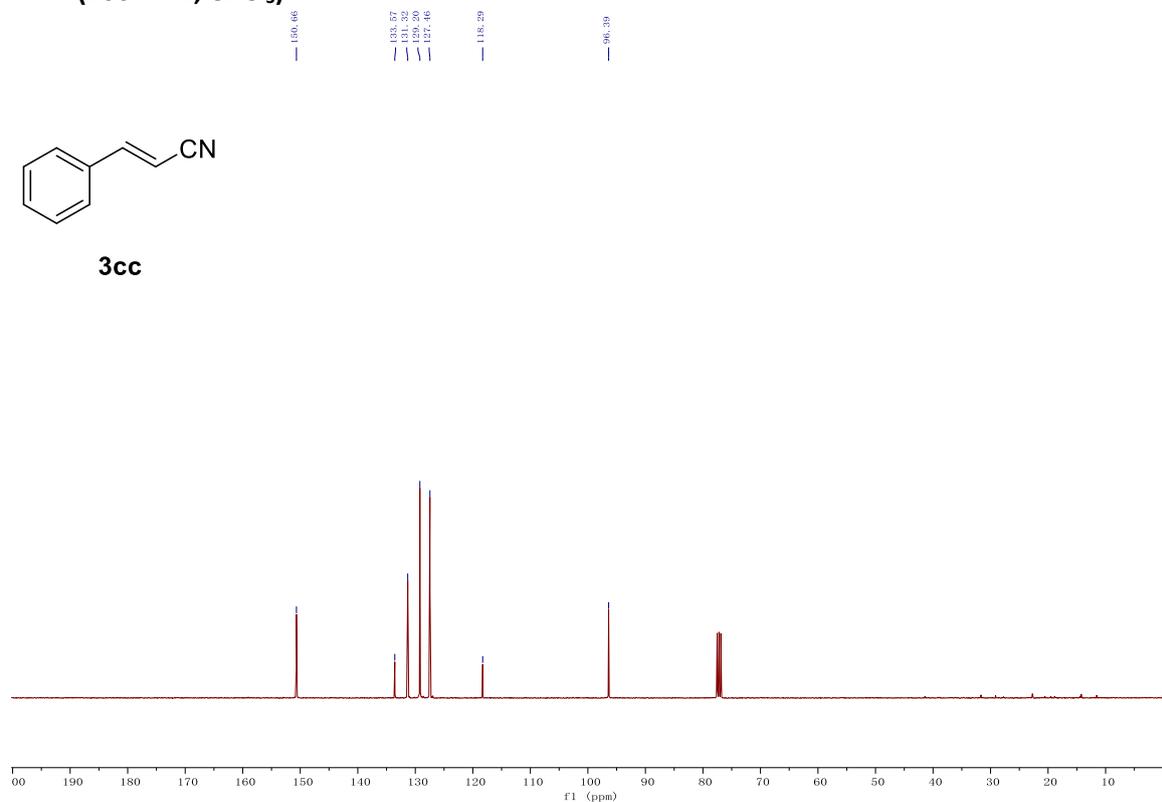
3bb: ¹³C NMR (100 MHz, CDCl₃)



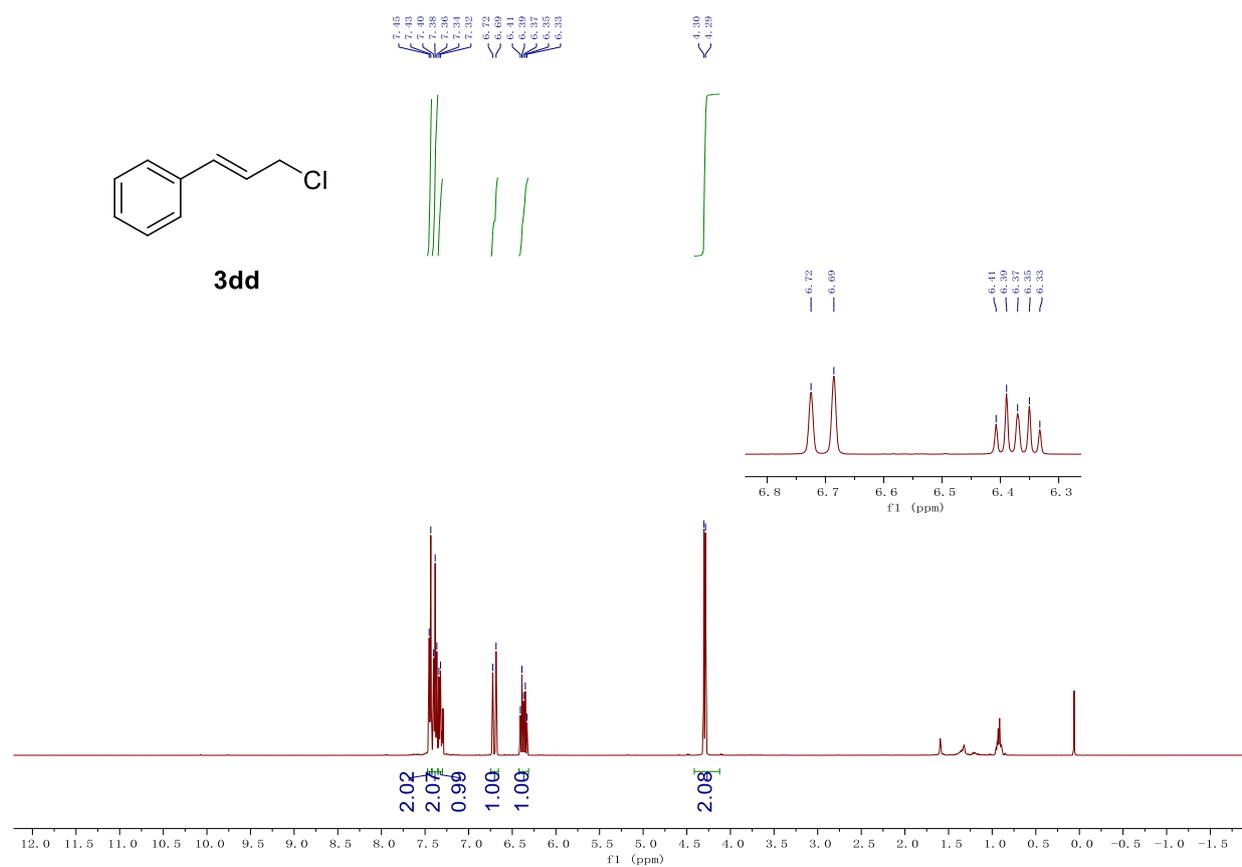
3cc: ¹H NMR (400 MHz, CDCl₃)



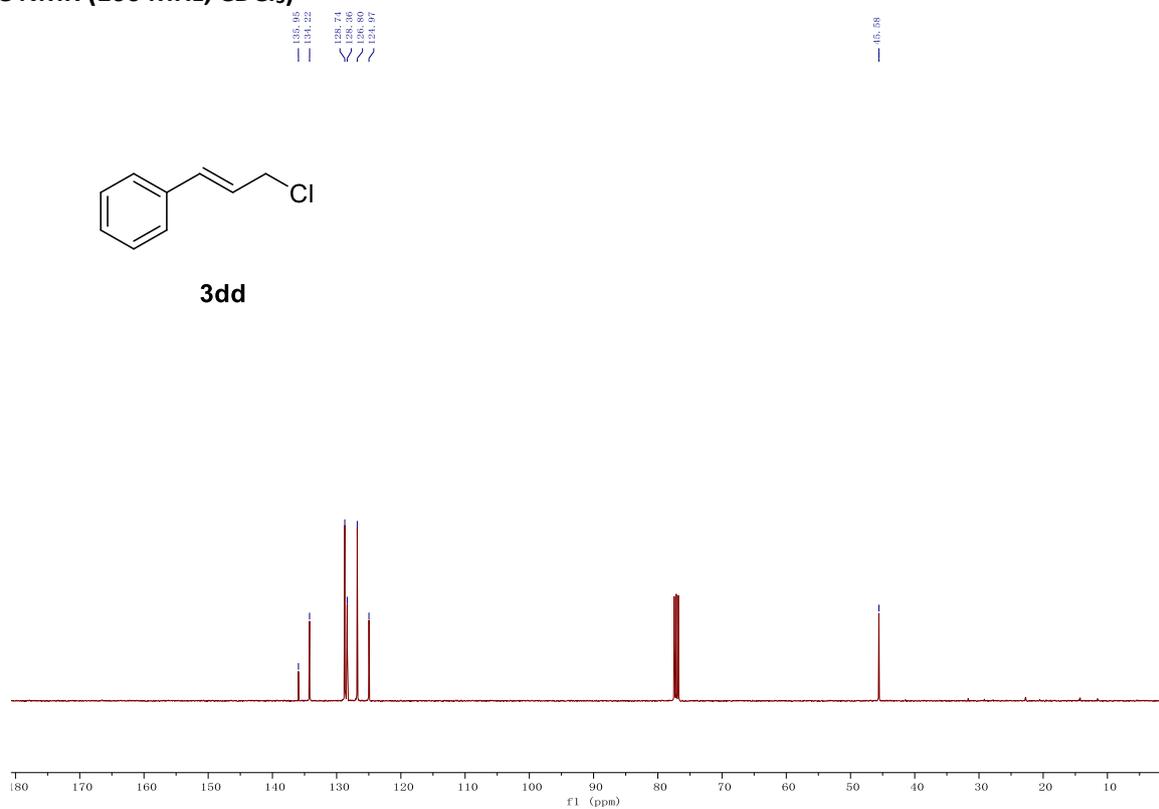
3cc: ¹³C NMR (100 MHz, CDCl₃)



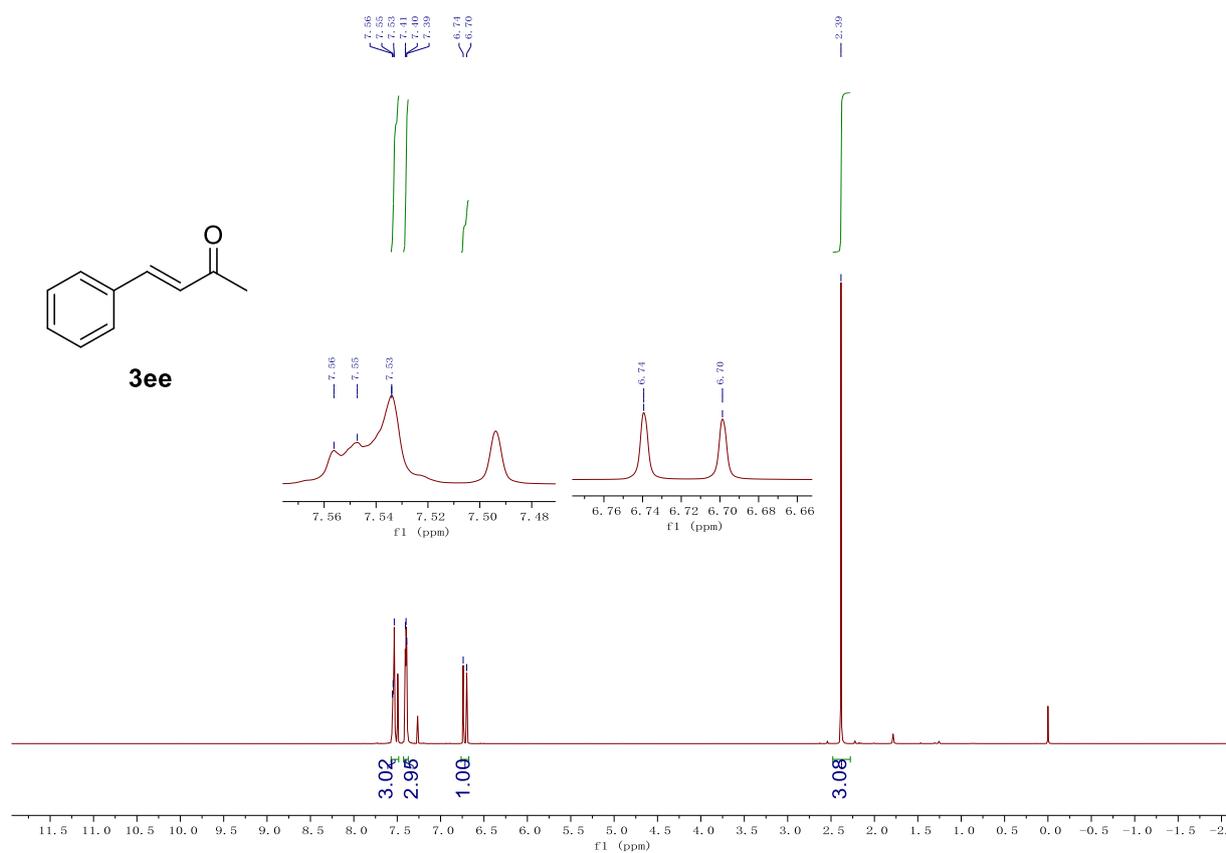
3dd: ¹H NMR (400 MHz, CDCl₃)



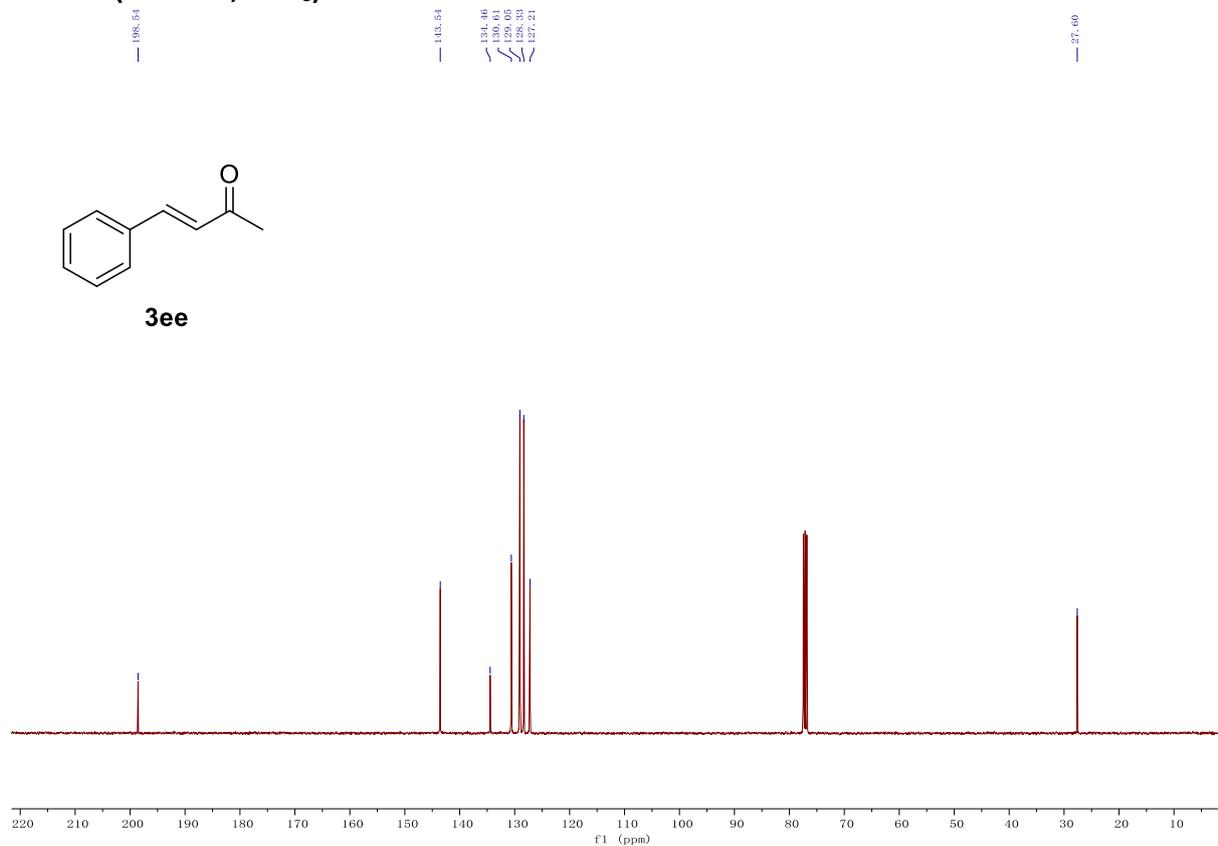
3dd: ¹³C NMR (100 MHz, CDCl₃)



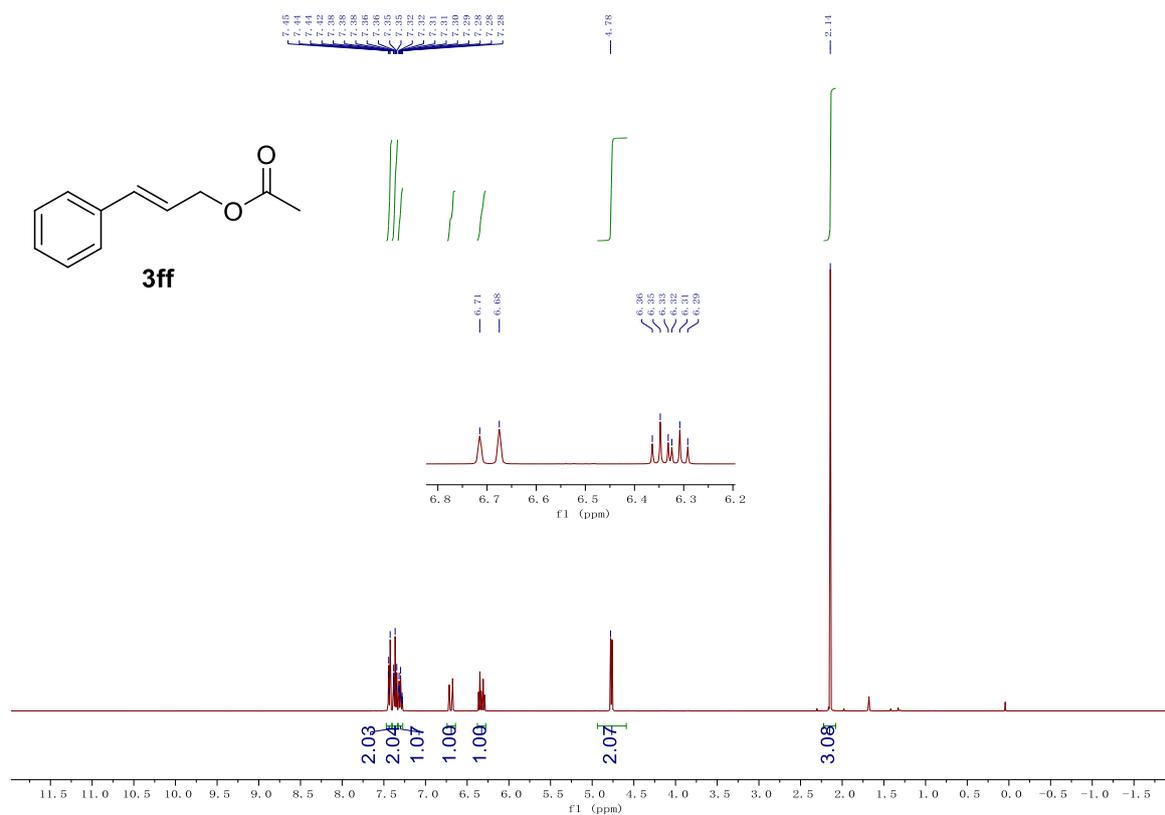
3ee: ¹H NMR (400 MHz, CDCl₃)



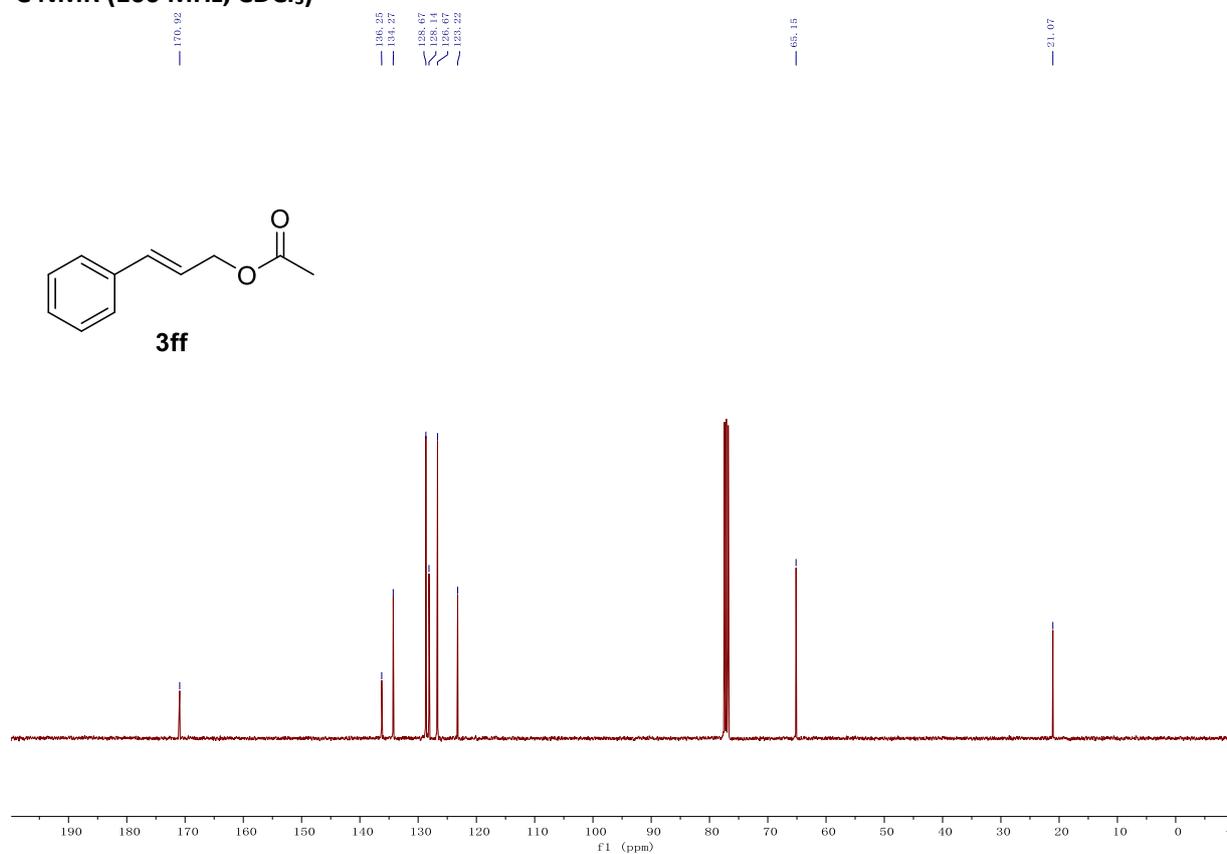
3ee: ¹³C NMR (100 MHz, CDCl₃)



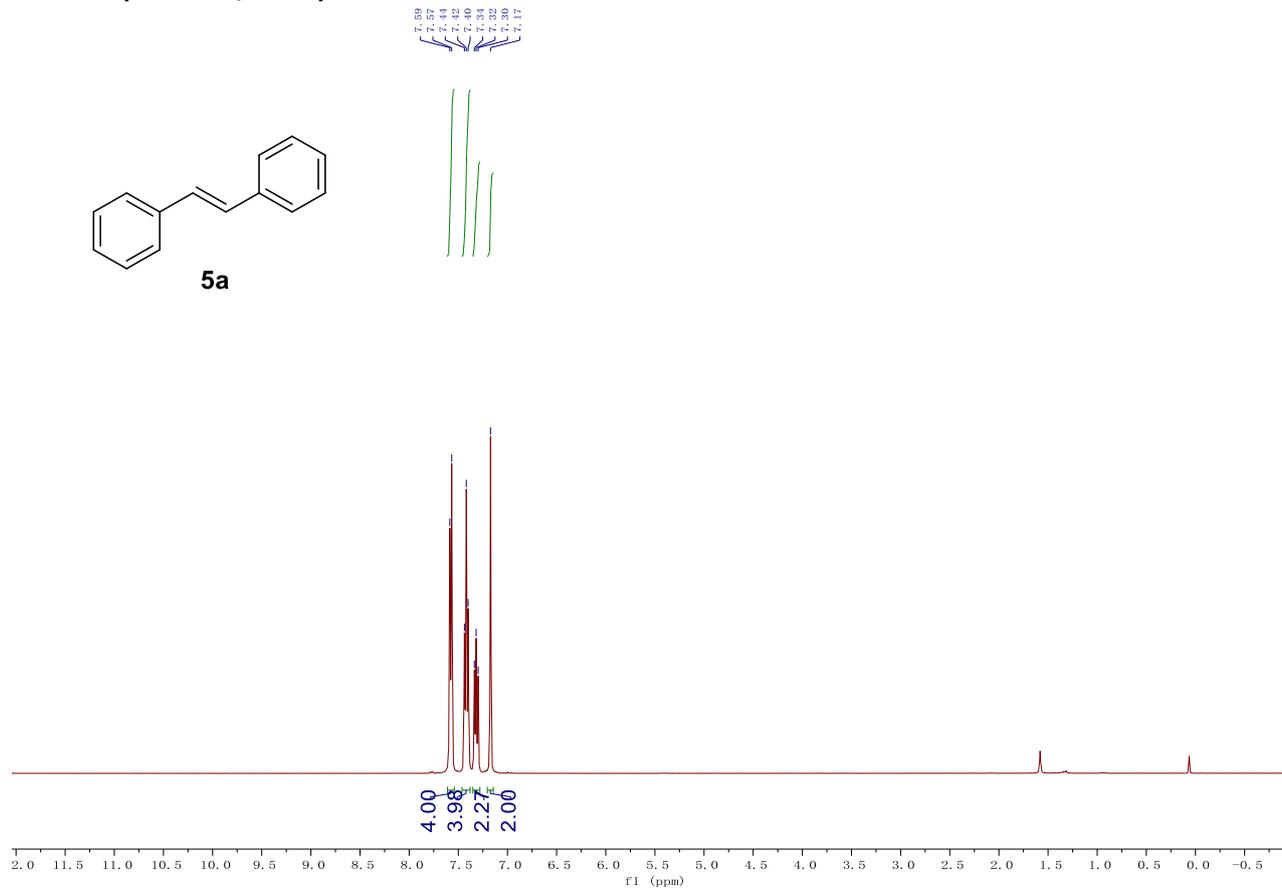
3ff: ¹H NMR (400 MHz, CDCl₃)



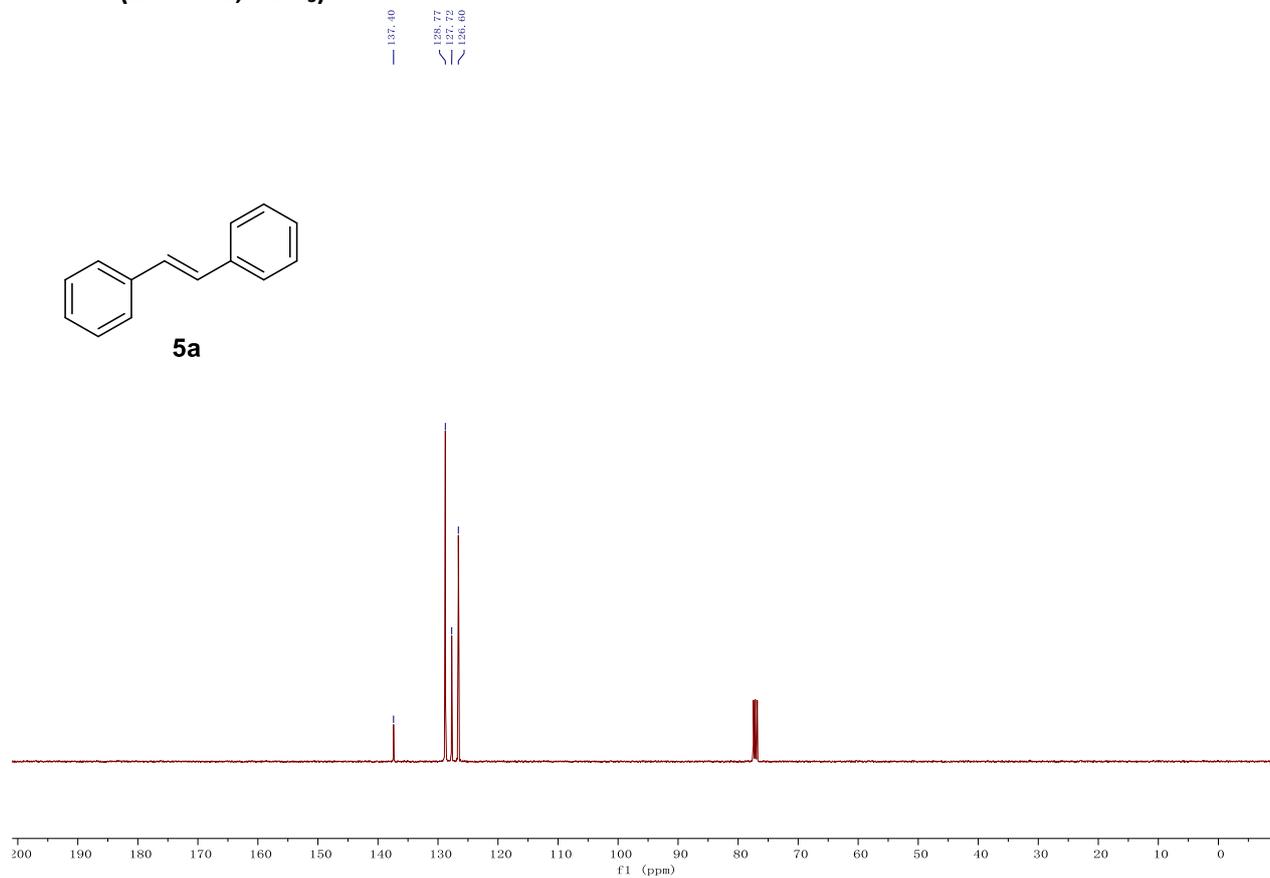
3ff: ¹³C NMR (100 MHz, CDCl₃)



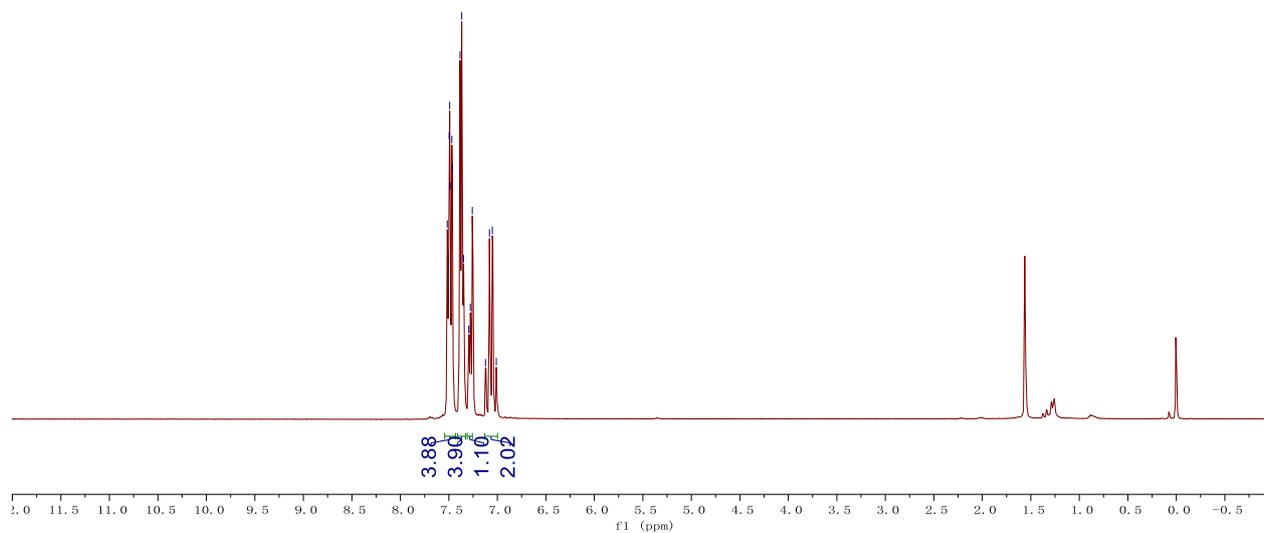
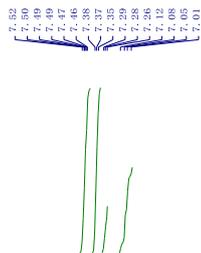
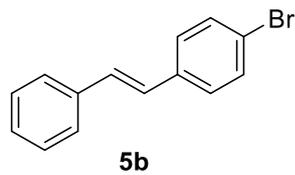
5a: ¹H NMR (400 MHz, CDCl₃)



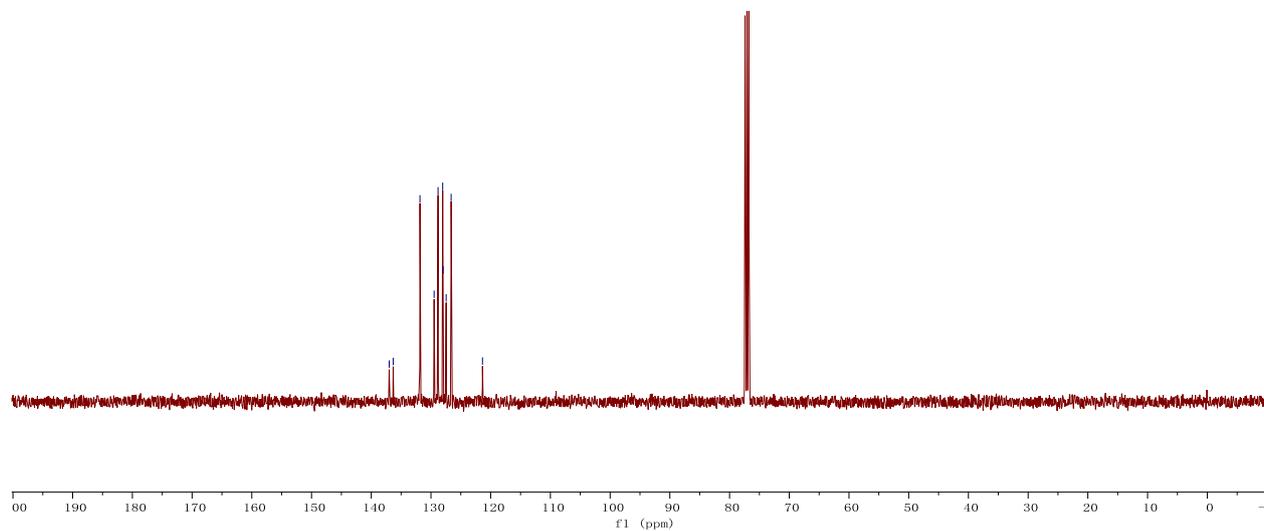
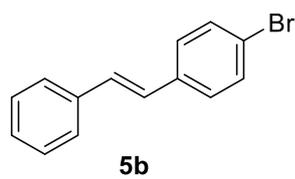
5a: ¹³C NMR (100 MHz, CDCl₃)



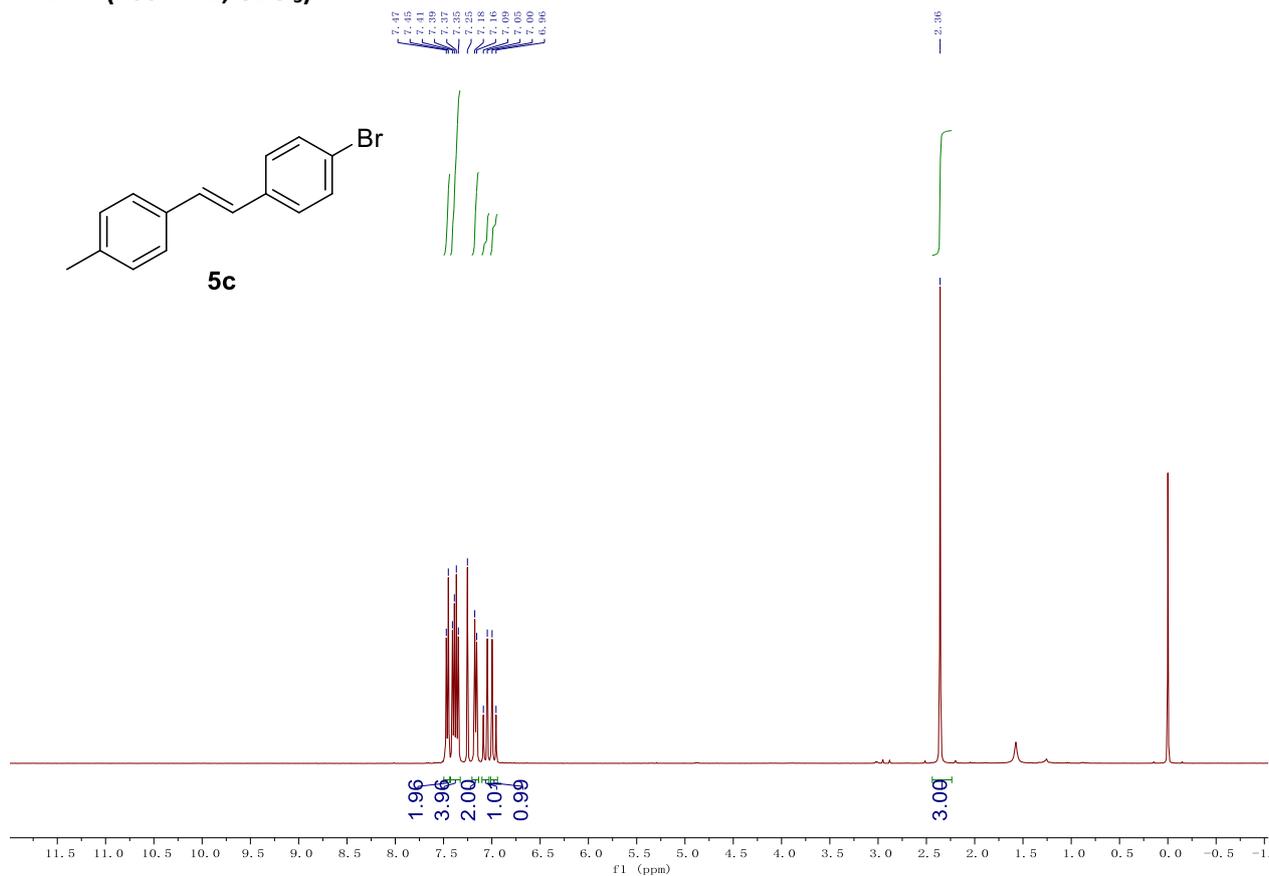
5b: ^1H NMR (400 MHz, CDCl_3)



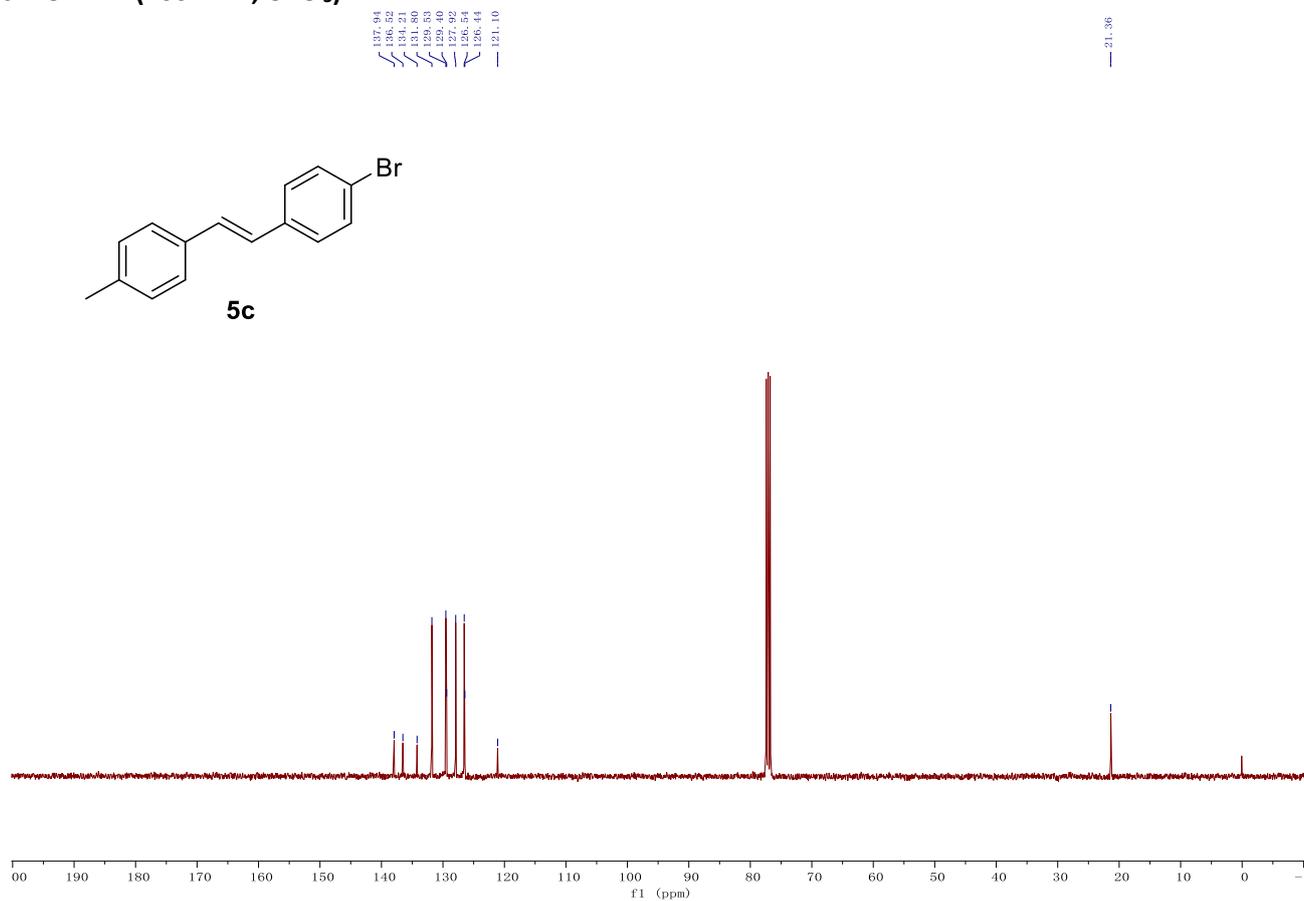
5b: ^{13}C NMR (100 MHz, CDCl_3)



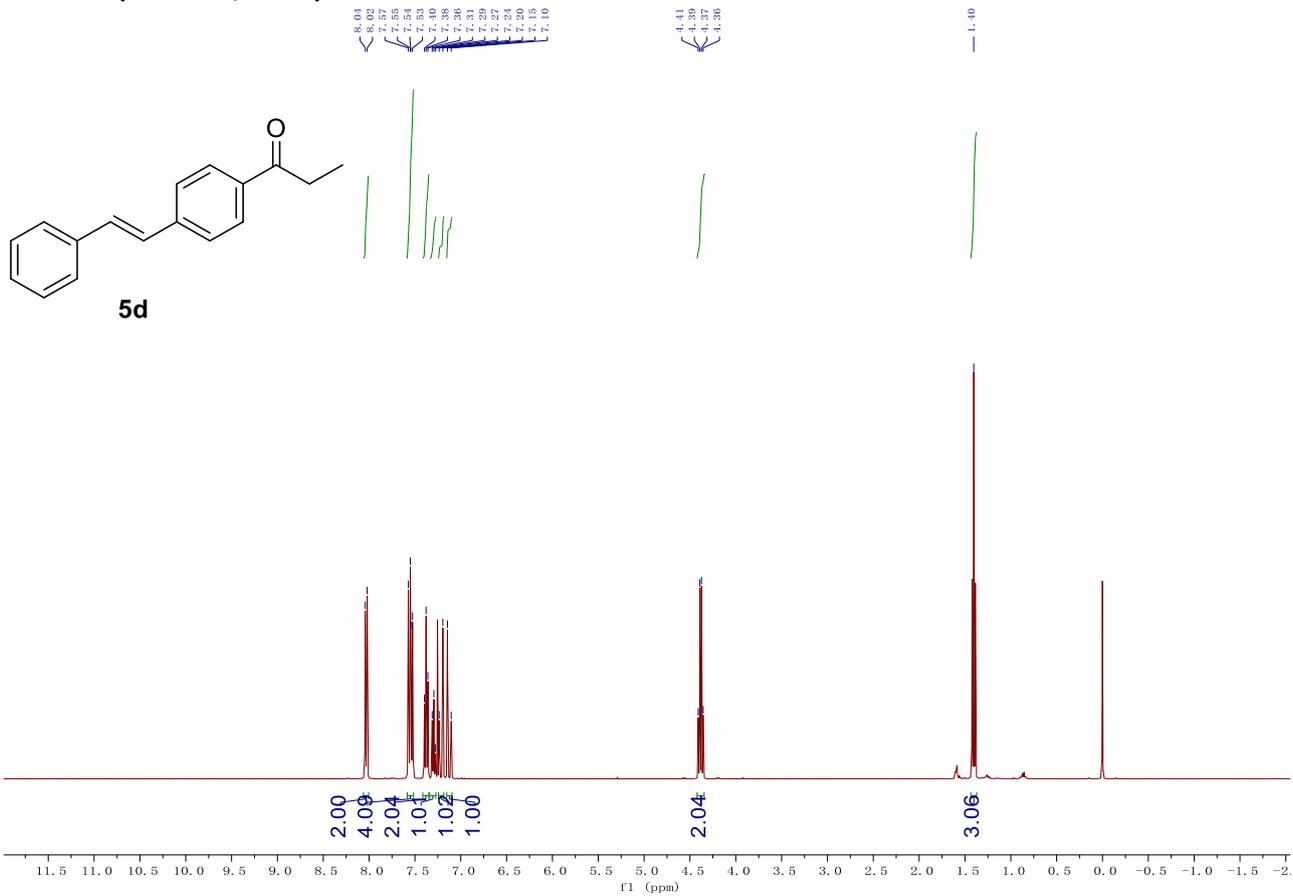
5c: ¹H NMR (400 MHz, CDCl₃)



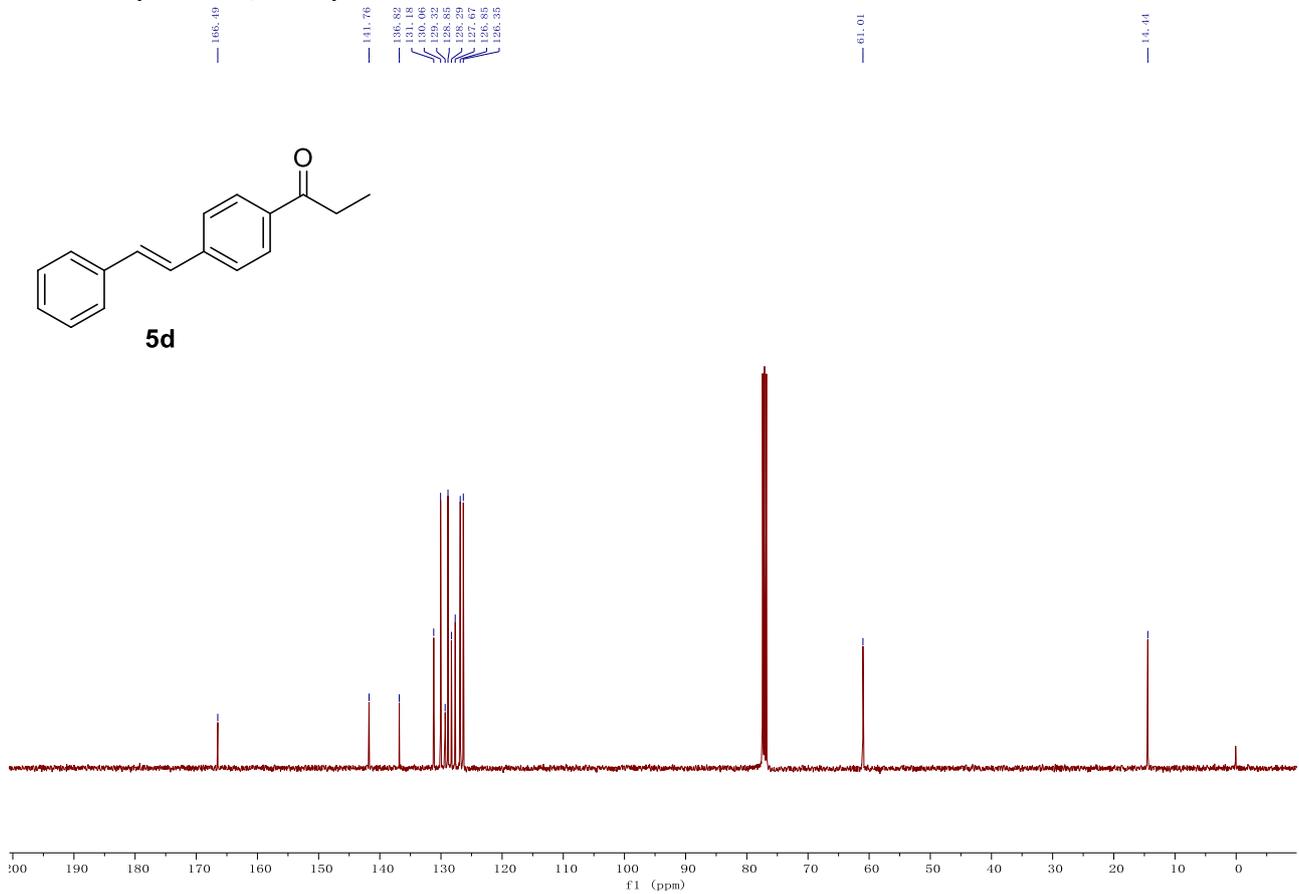
5c: ¹³C NMR (100 MHz, CDCl₃)



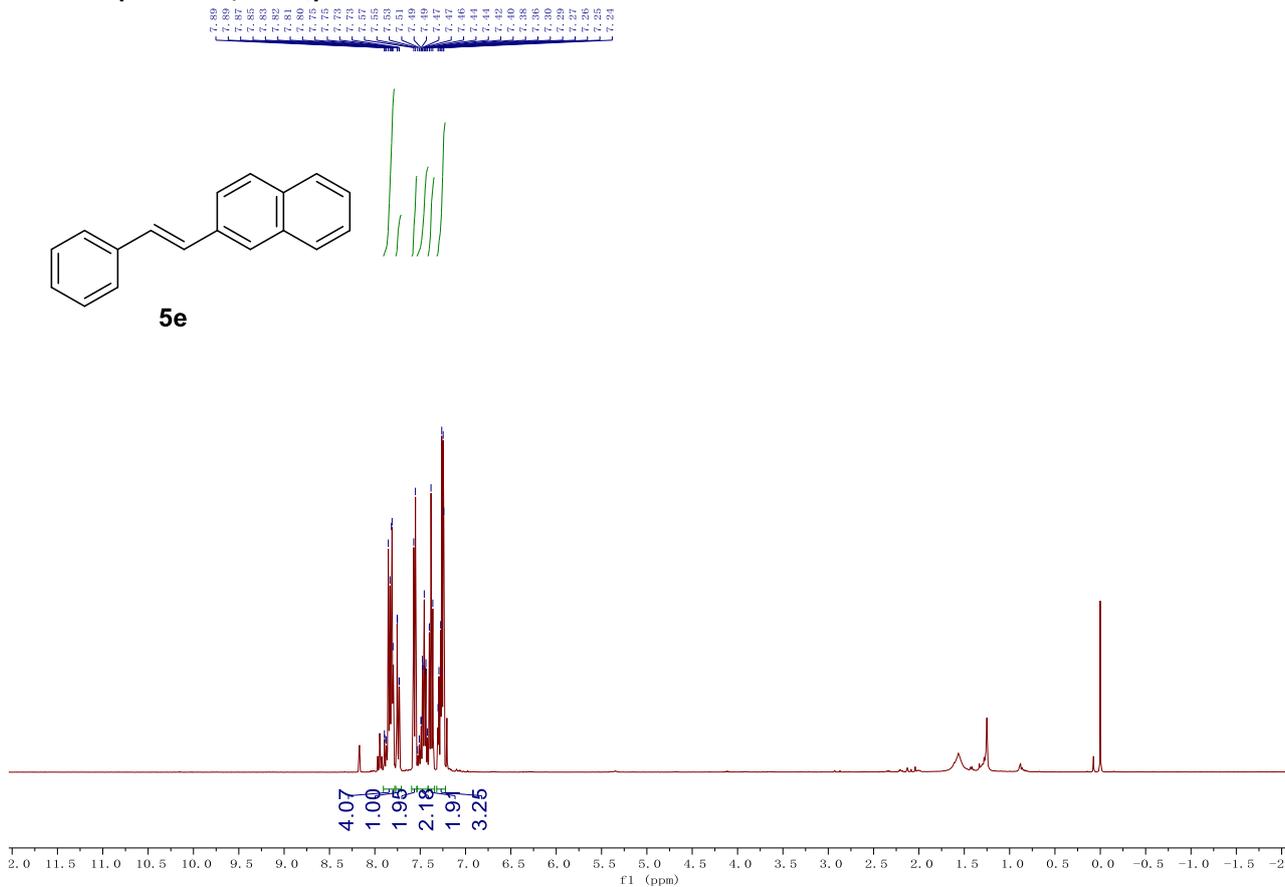
5d: ¹H NMR (400 MHz, CDCl₃)



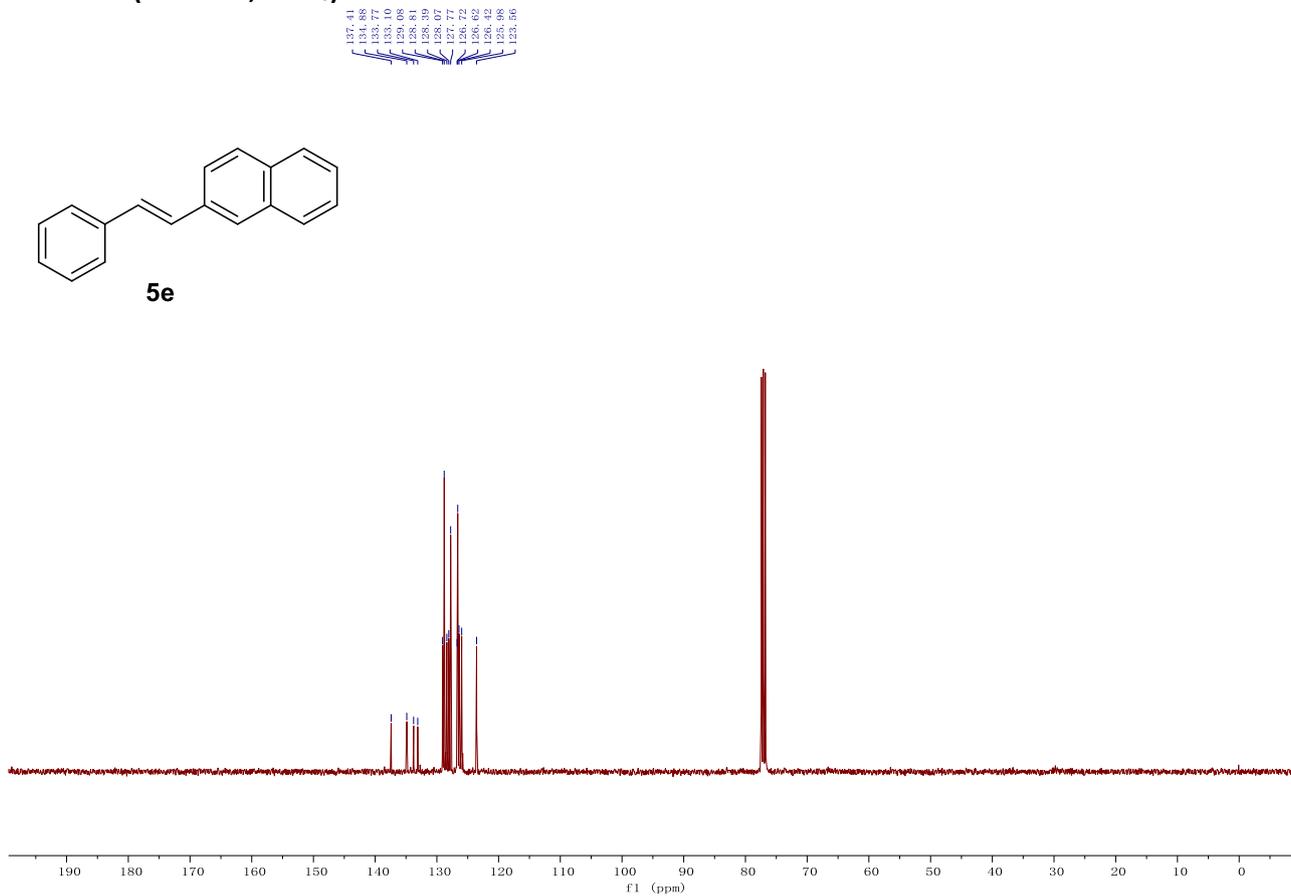
5d: ¹³C NMR (100 MHz, CDCl₃)



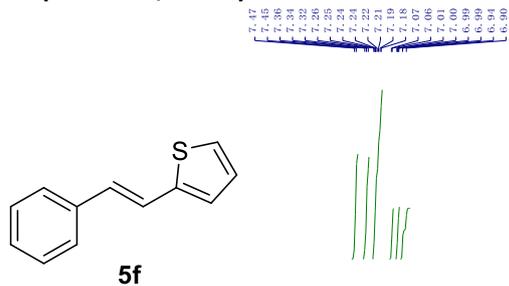
5e: ¹H NMR (400 MHz, CDCl₃)



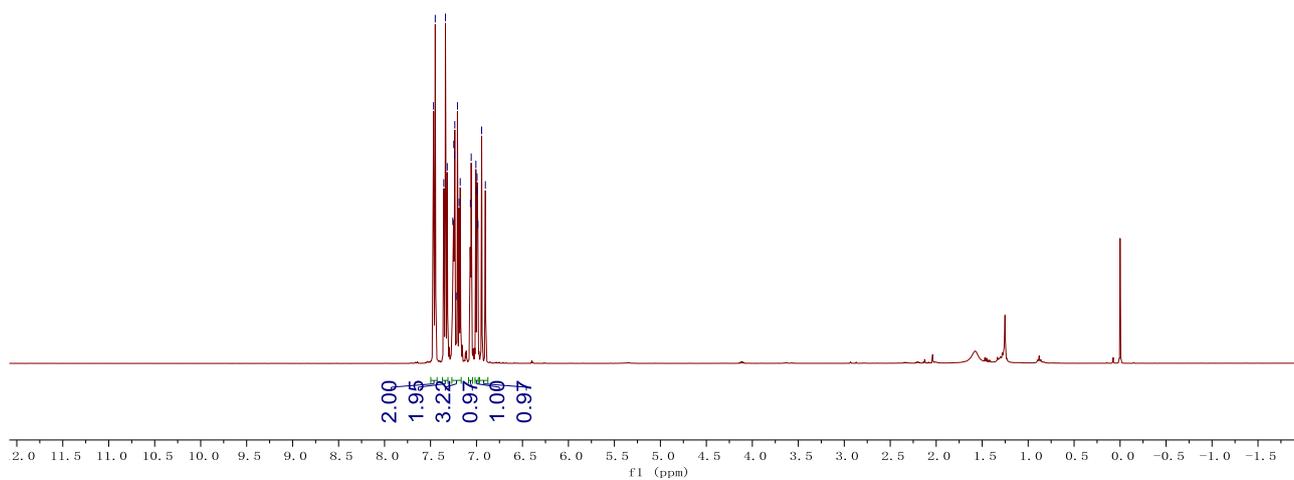
5e: ¹³C NMR (100 MHz, CDCl₃)



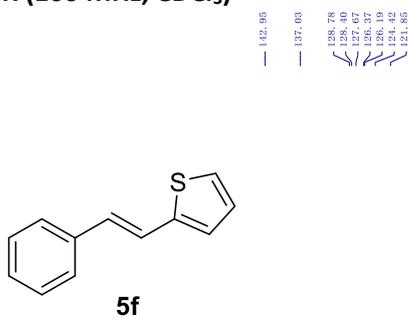
5f: ¹H NMR (400 MHz, CDCl₃)



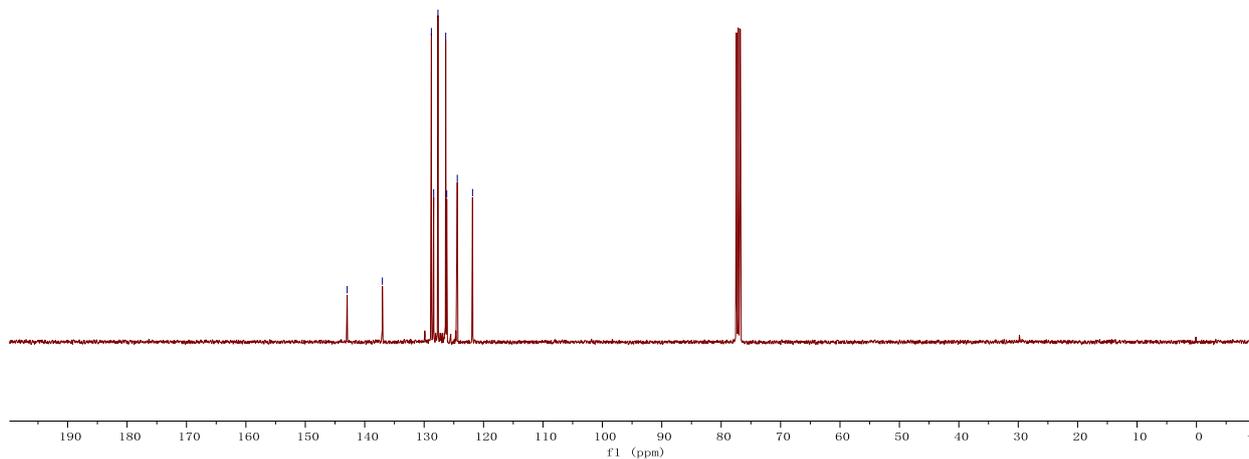
7.47
7.45
7.38
7.32
7.28
7.25
7.24
7.22
7.21
7.19
7.07
7.06
7.01
7.00
6.99
6.94
6.90



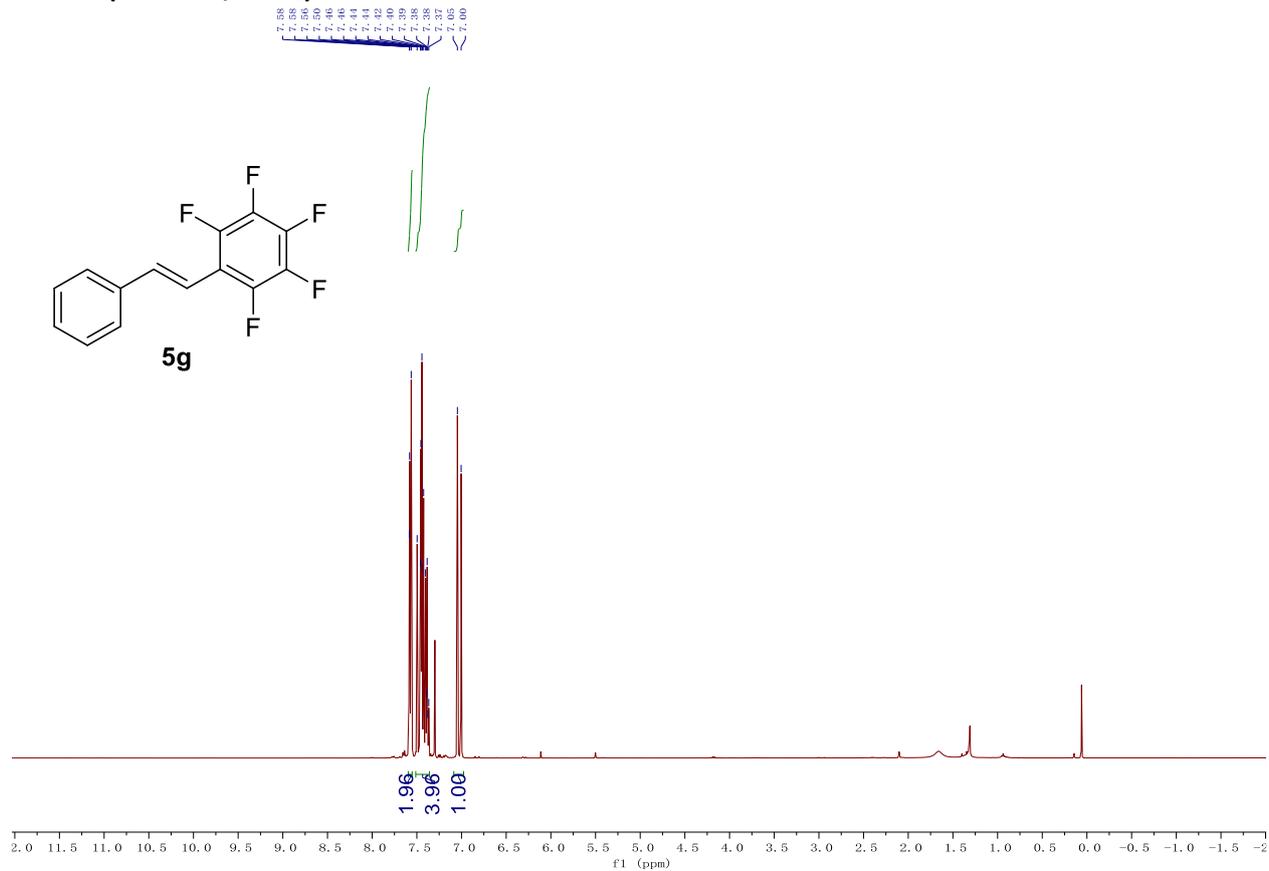
5f: ¹³C NMR (100 MHz, CDCl₃)



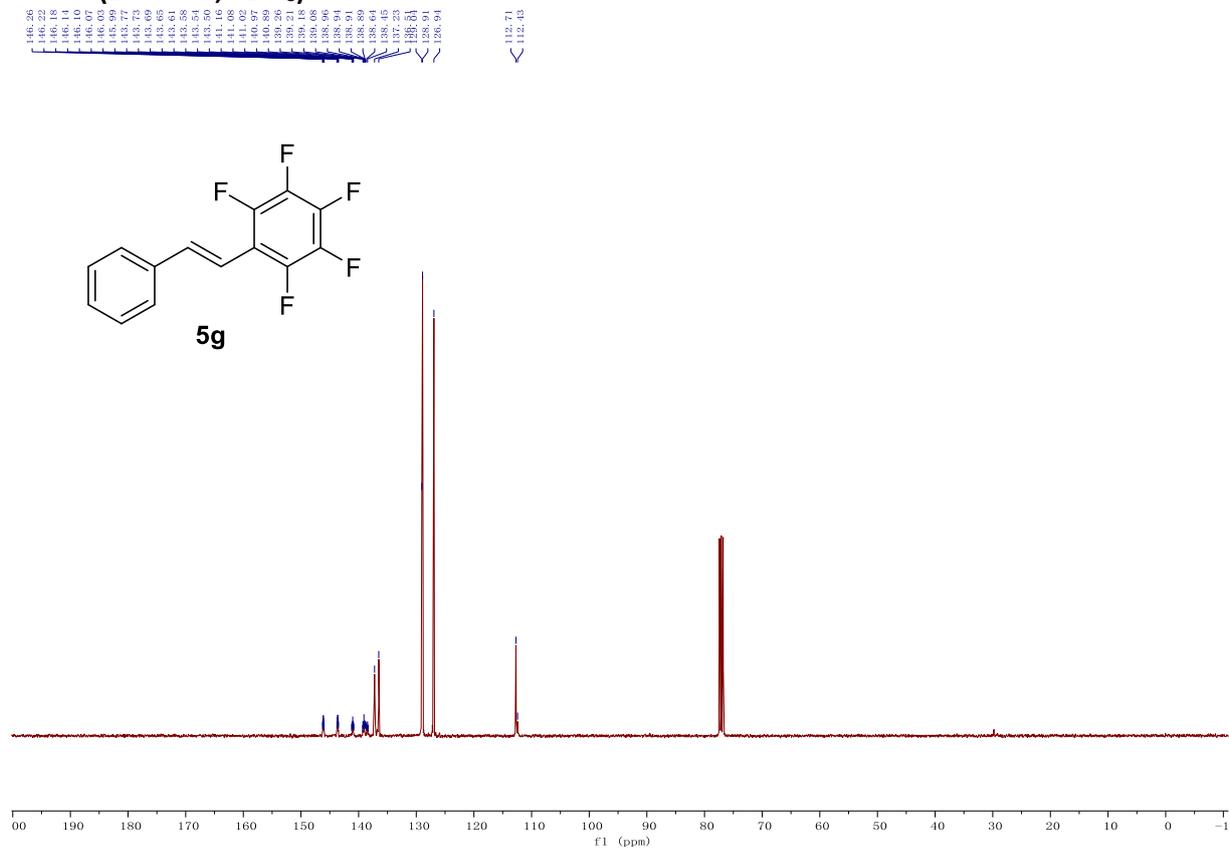
142.95
137.03
128.78
127.67
126.37
126.19
121.85



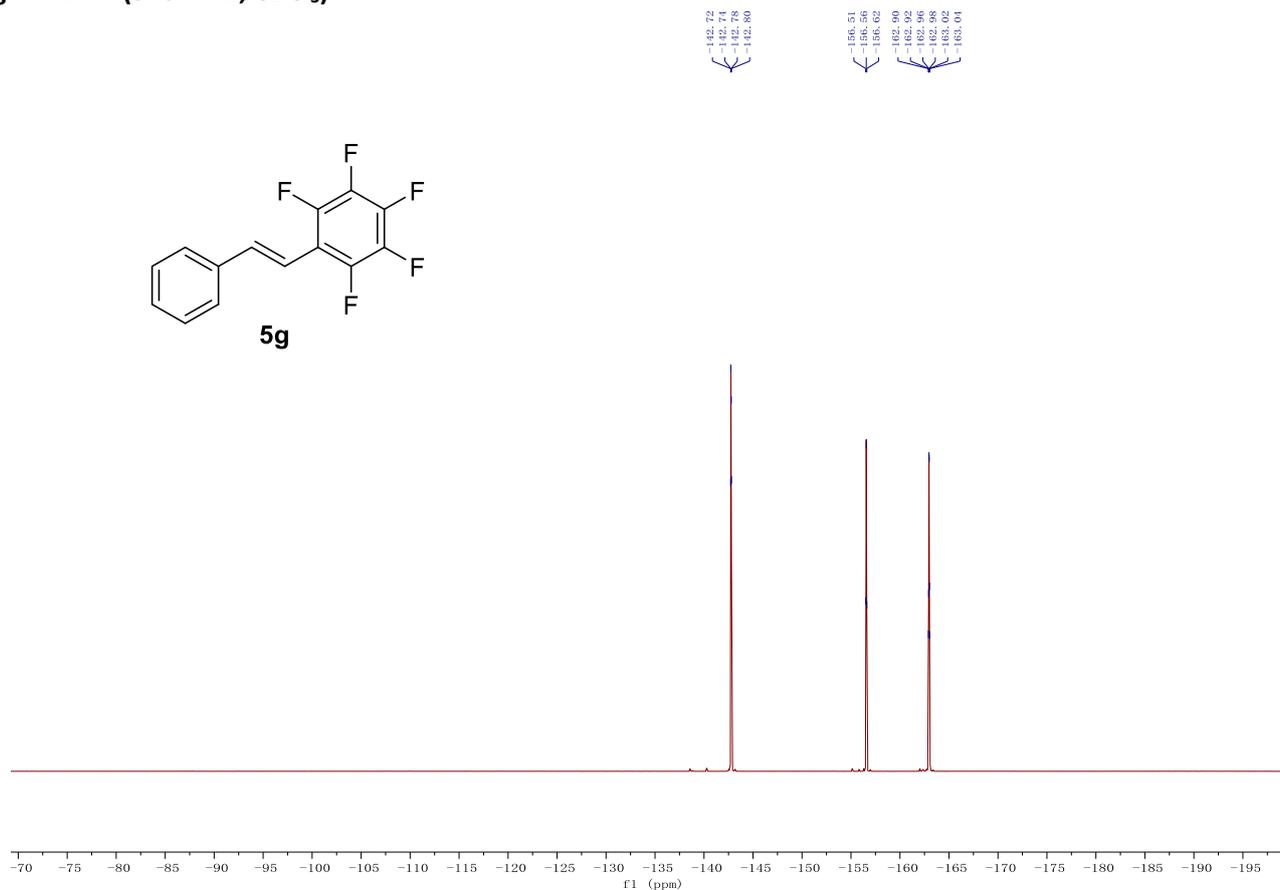
5g: ¹H NMR (400 MHz, CDCl₃)



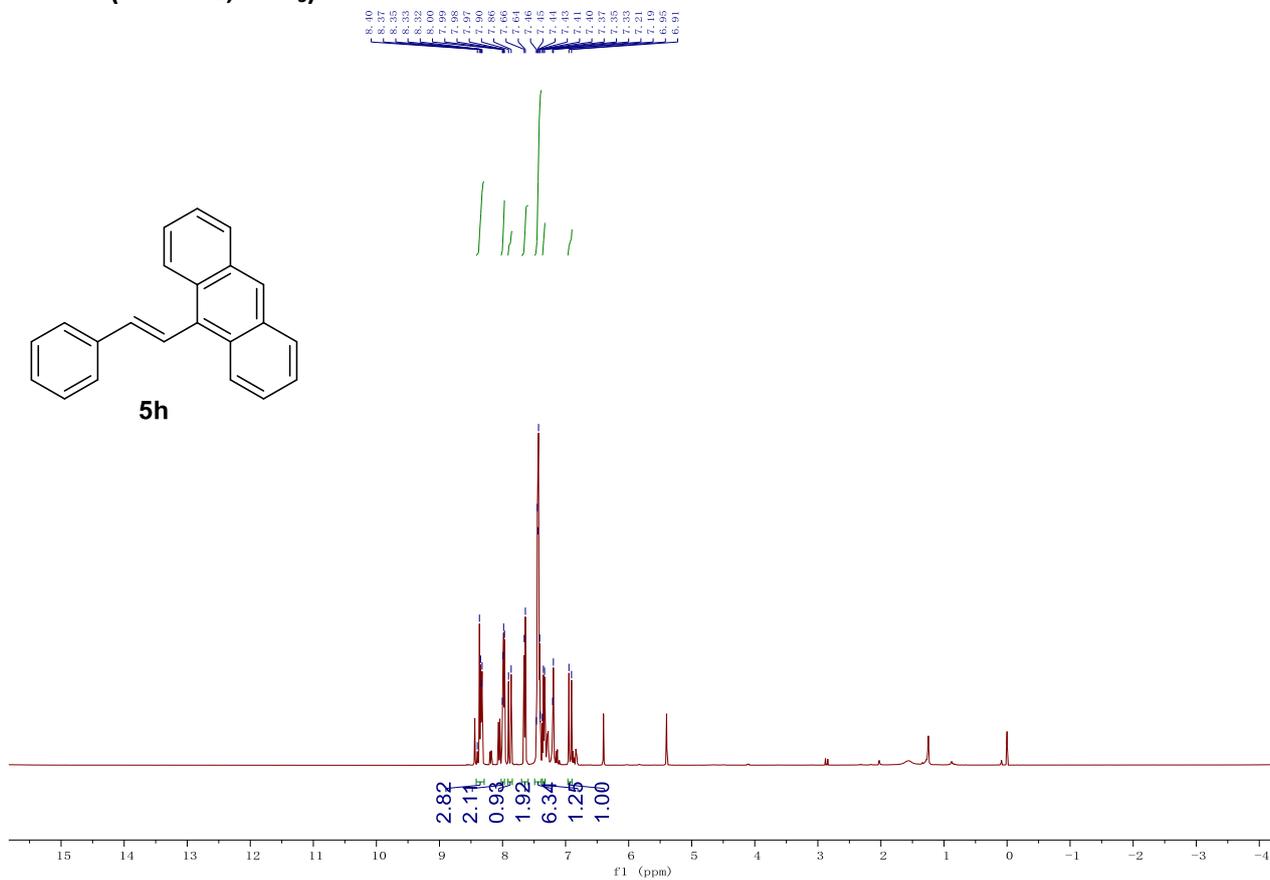
5g: ¹³C NMR (100 MHz, CDCl₃)



5g: ¹⁹F NMR (376 MHz, CDCl₃)

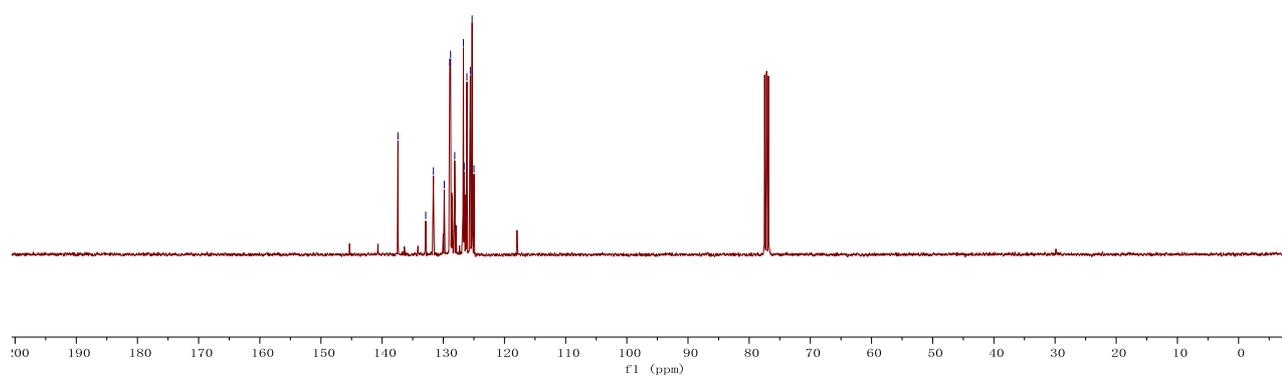
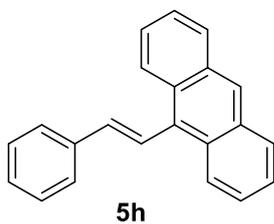


5h: ¹H NMR (400 MHz, CDCl₃)



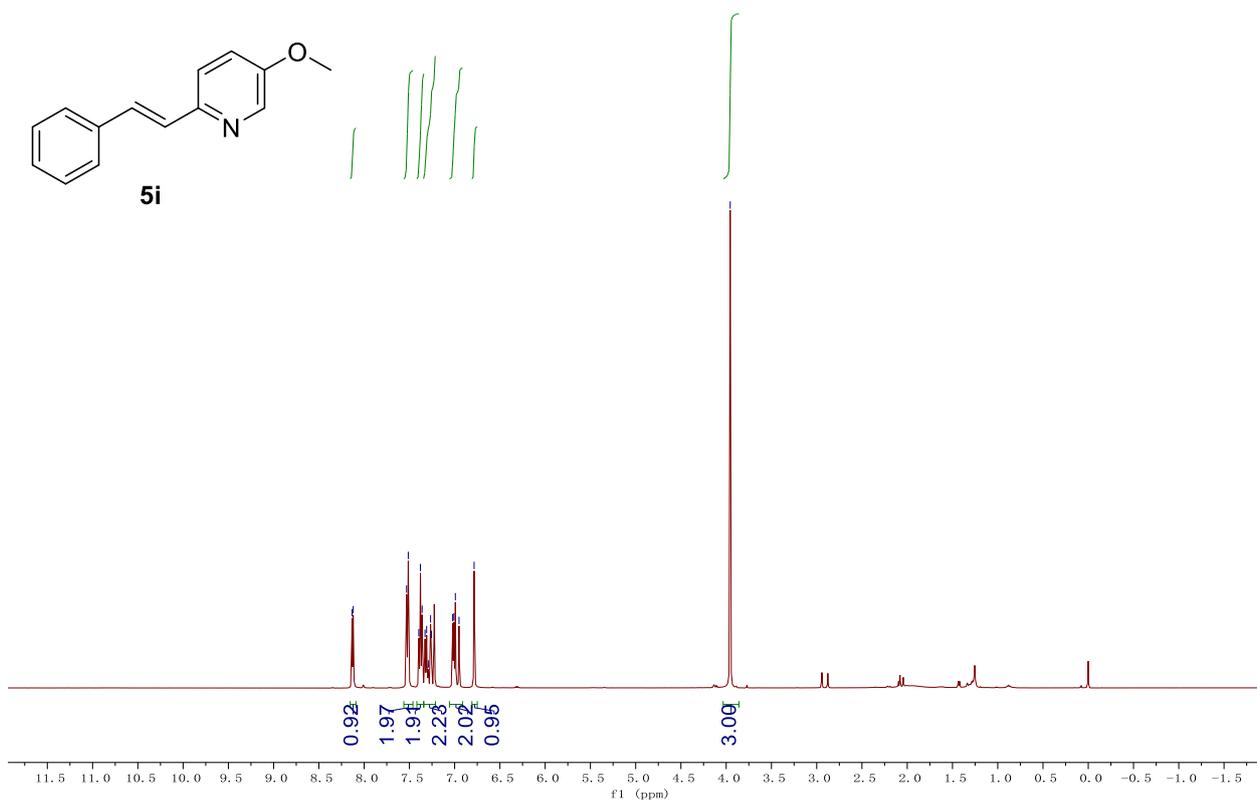
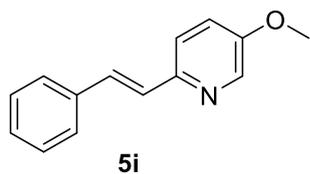
5h: ¹³C NMR (100 MHz, CDCl₃)

132.41
132.885
131.61
129.84
128.81
128.13
126.71
126.08
125.58
125.30
124.99

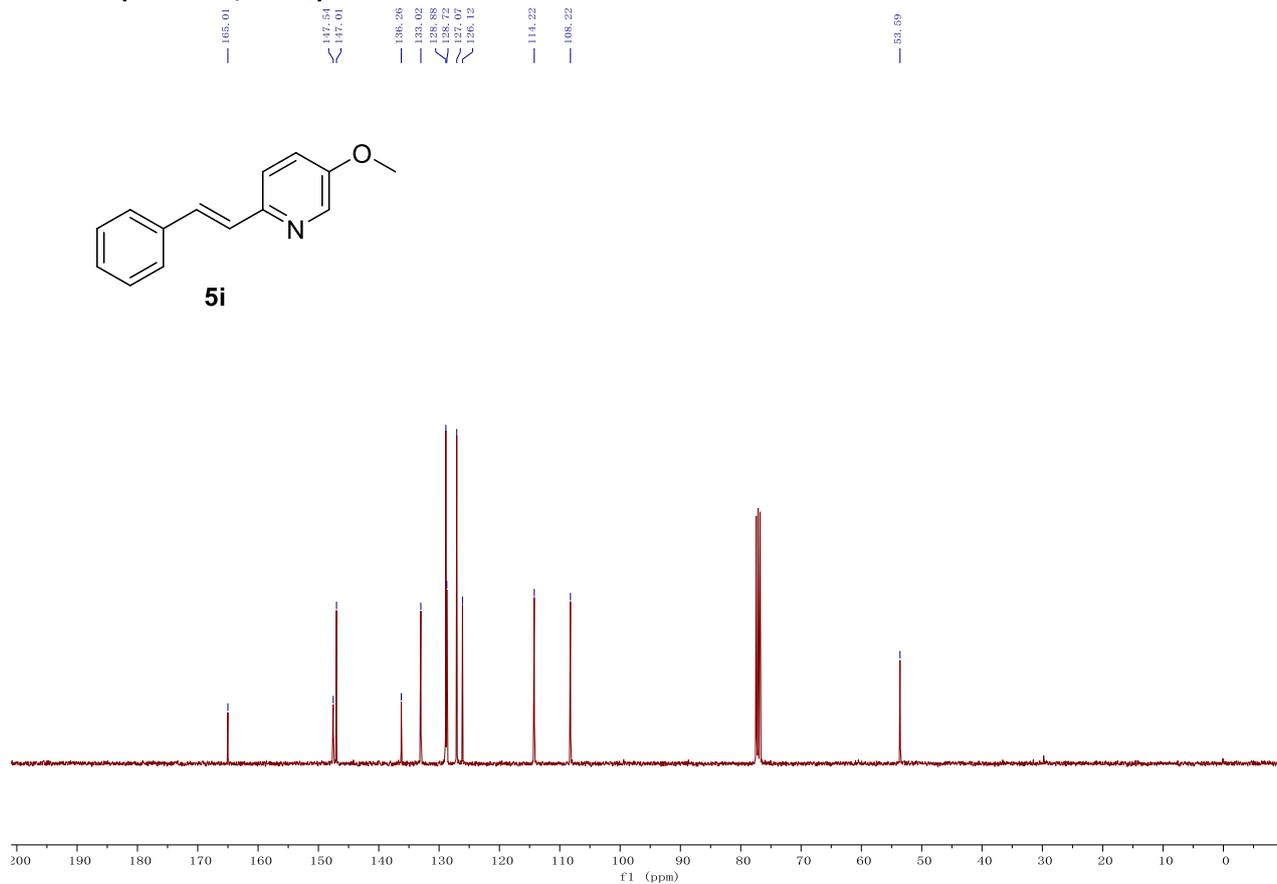


5i: ¹H NMR (400 MHz, CDCl₃)

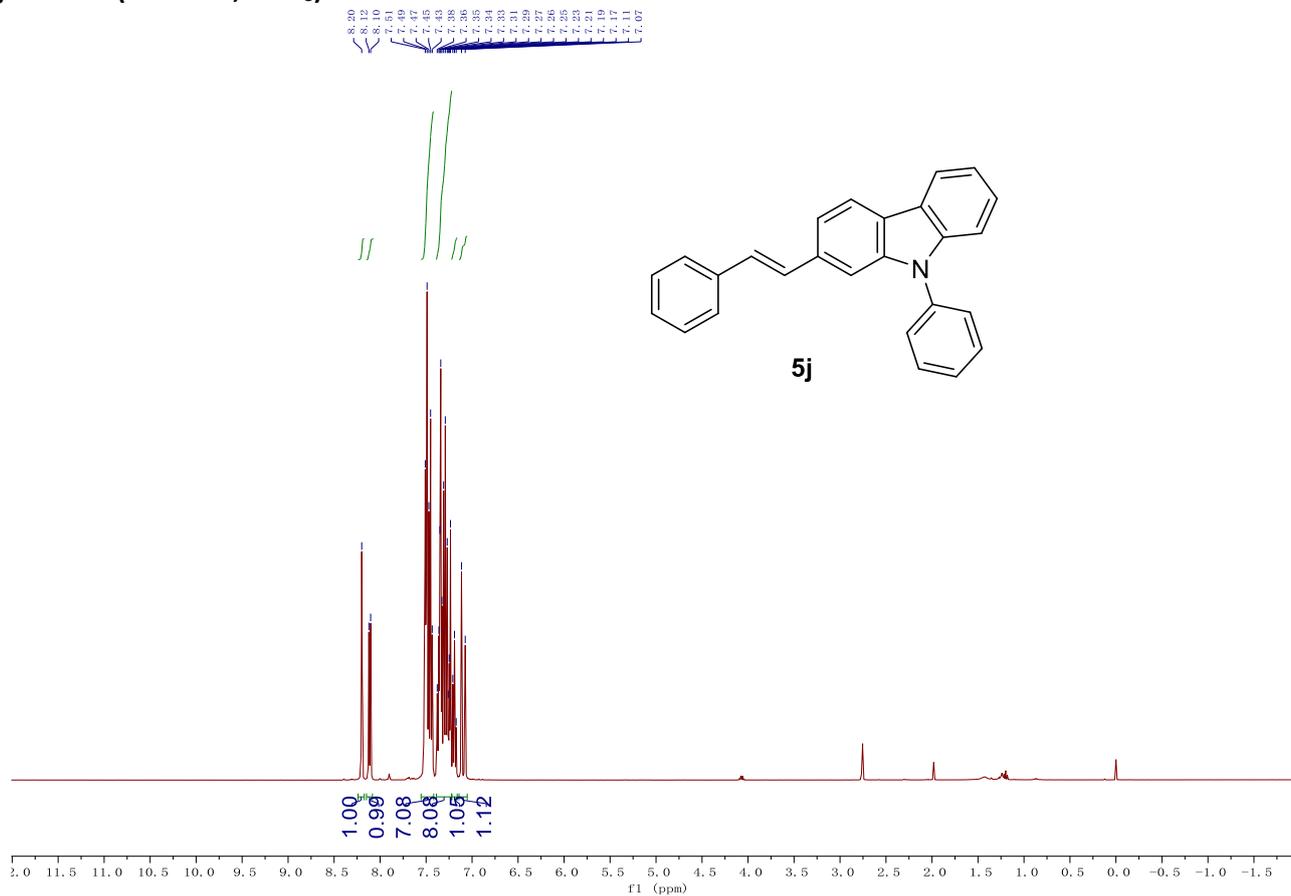
8.13
8.12
7.93
7.70
7.38
7.36
7.31
7.29
7.27
7.02
7.01
6.99
6.79
3.96



5i: ¹³C NMR (100 MHz, CDCl₃)

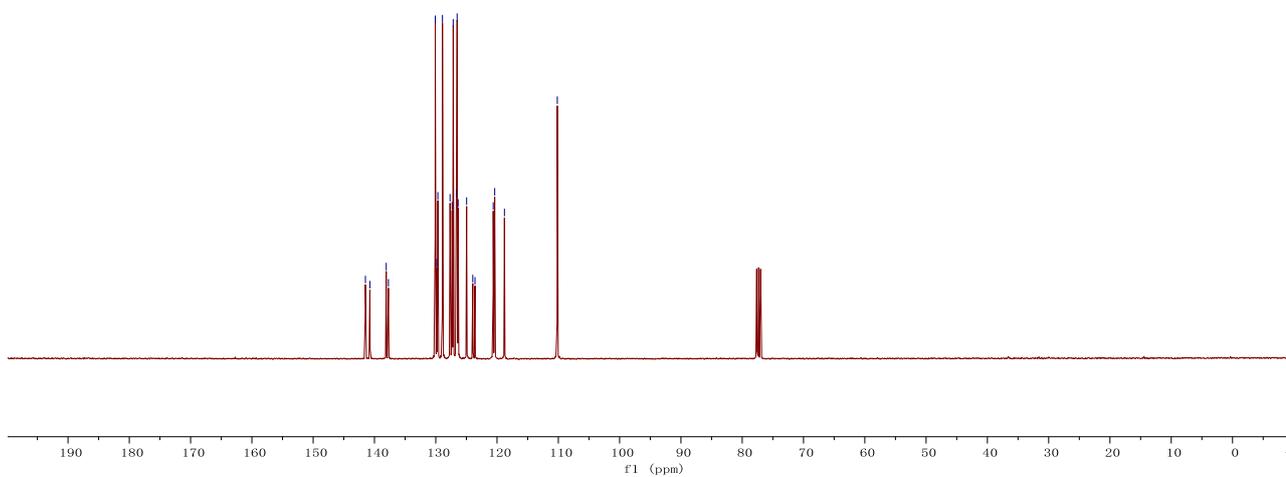
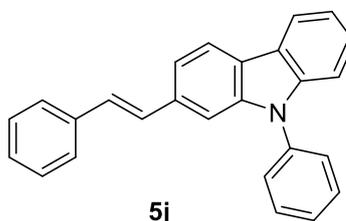


5j: ¹H NMR (400 MHz, CDCl₃)



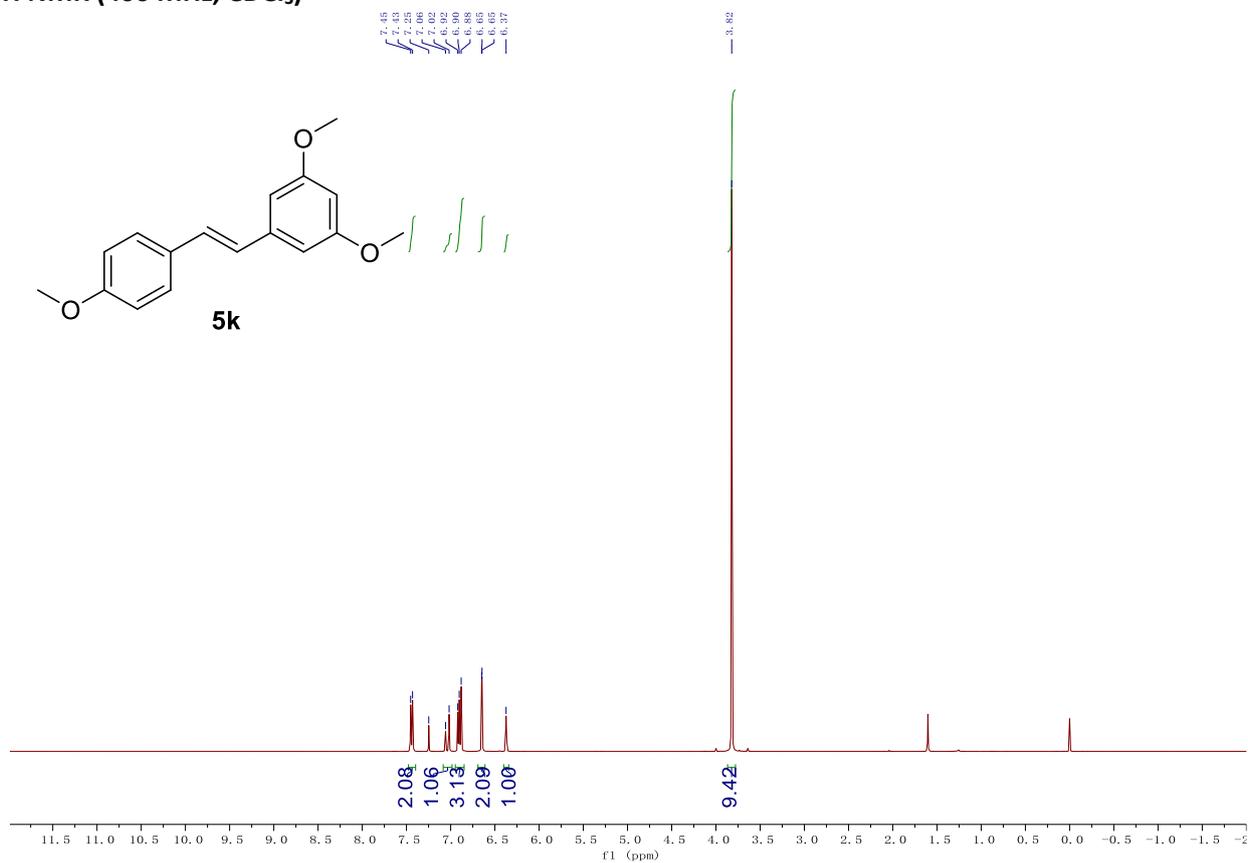
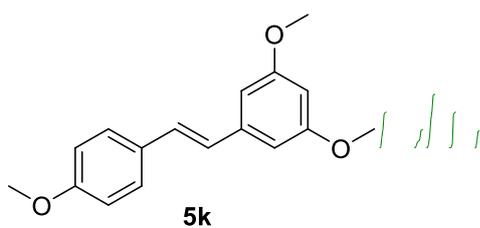
5j: ¹³C NMR (100 MHz, CDCl₃)

141.48
140.75
139.76
137.72
136.08
128.85
128.66
127.67
127.33
127.15
126.81
126.51
124.35
124.96
123.96
120.62
120.38
118.80
116.17

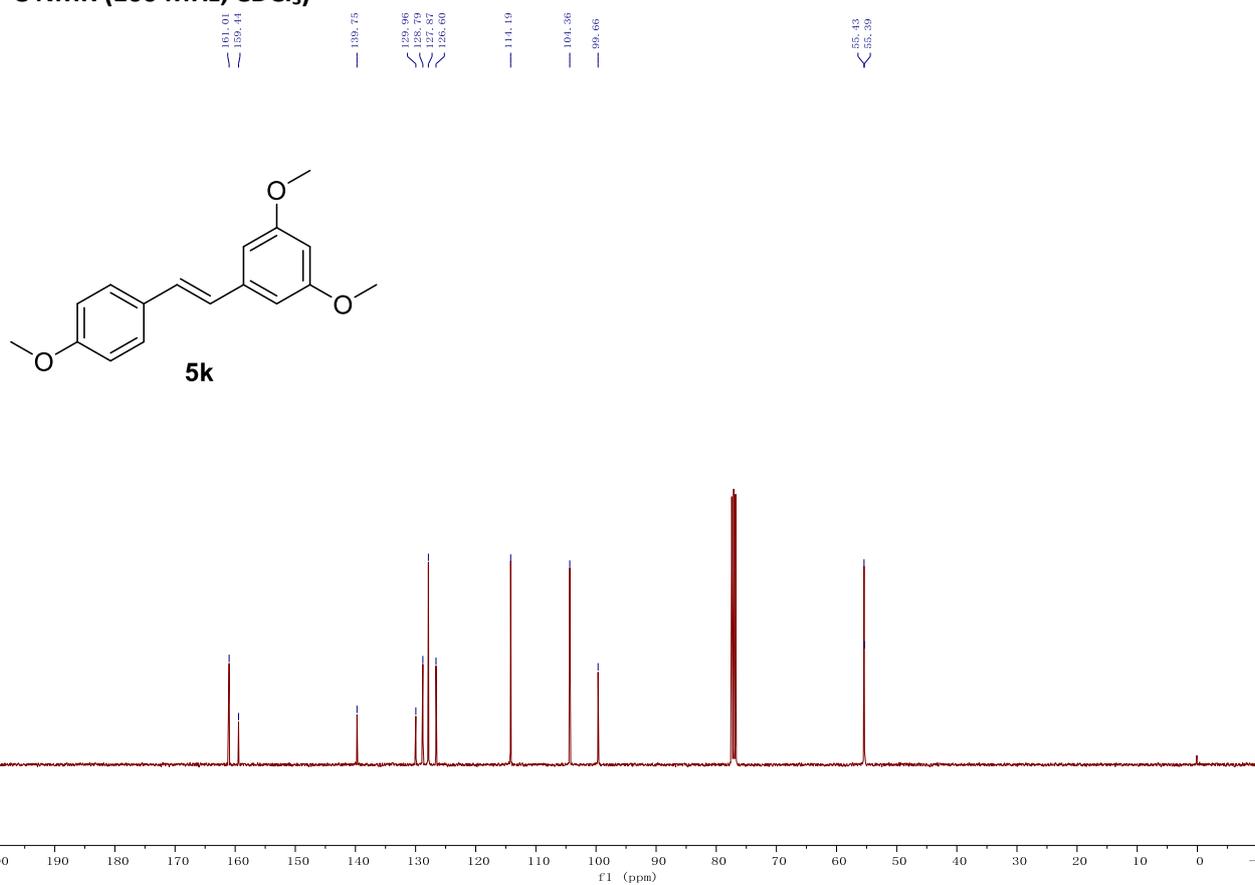


5k: ¹H NMR (400 MHz, CDCl₃)

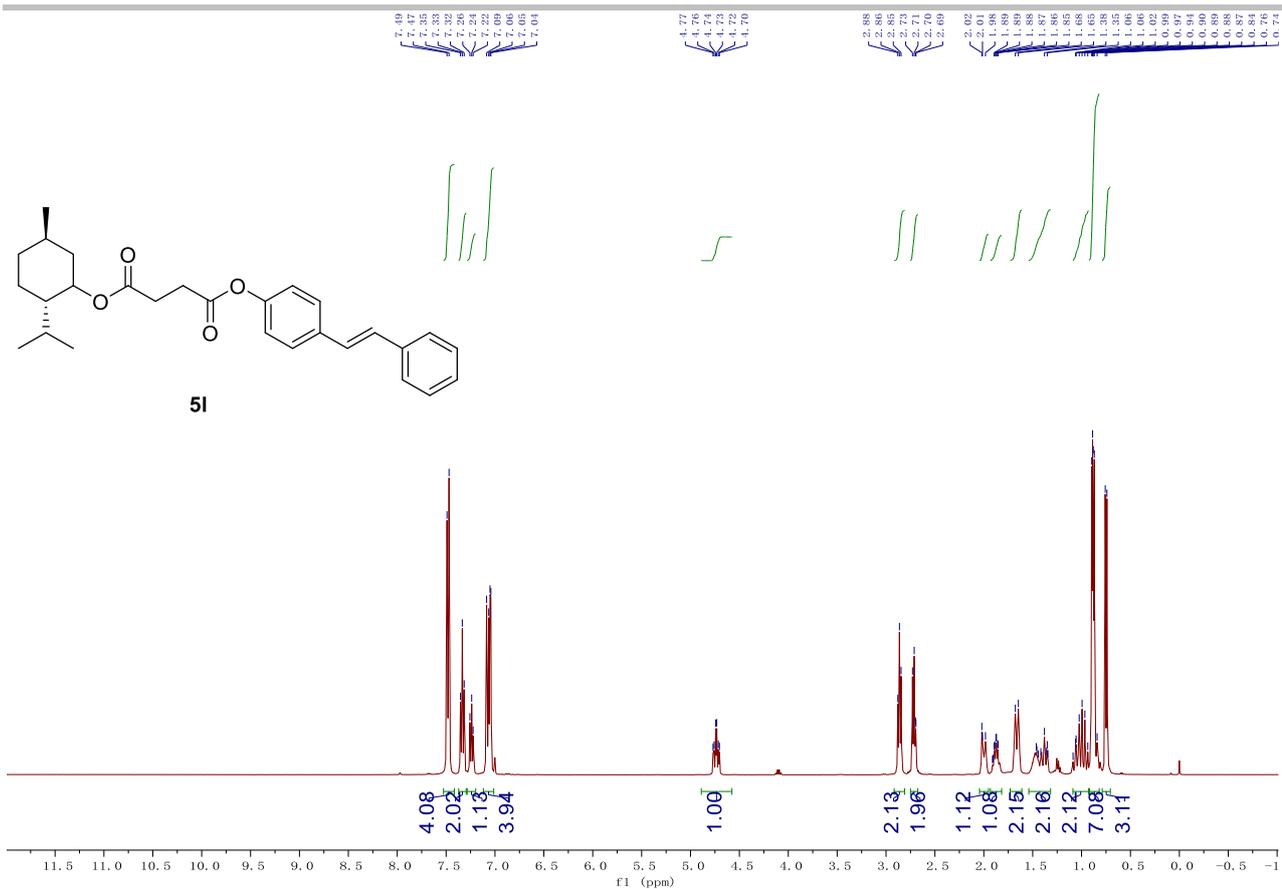
7.45
7.43
7.35
7.32
7.02
6.92
6.90
6.85
6.65
6.37



5k: ^{13}C NMR (100 MHz, CDCl_3)



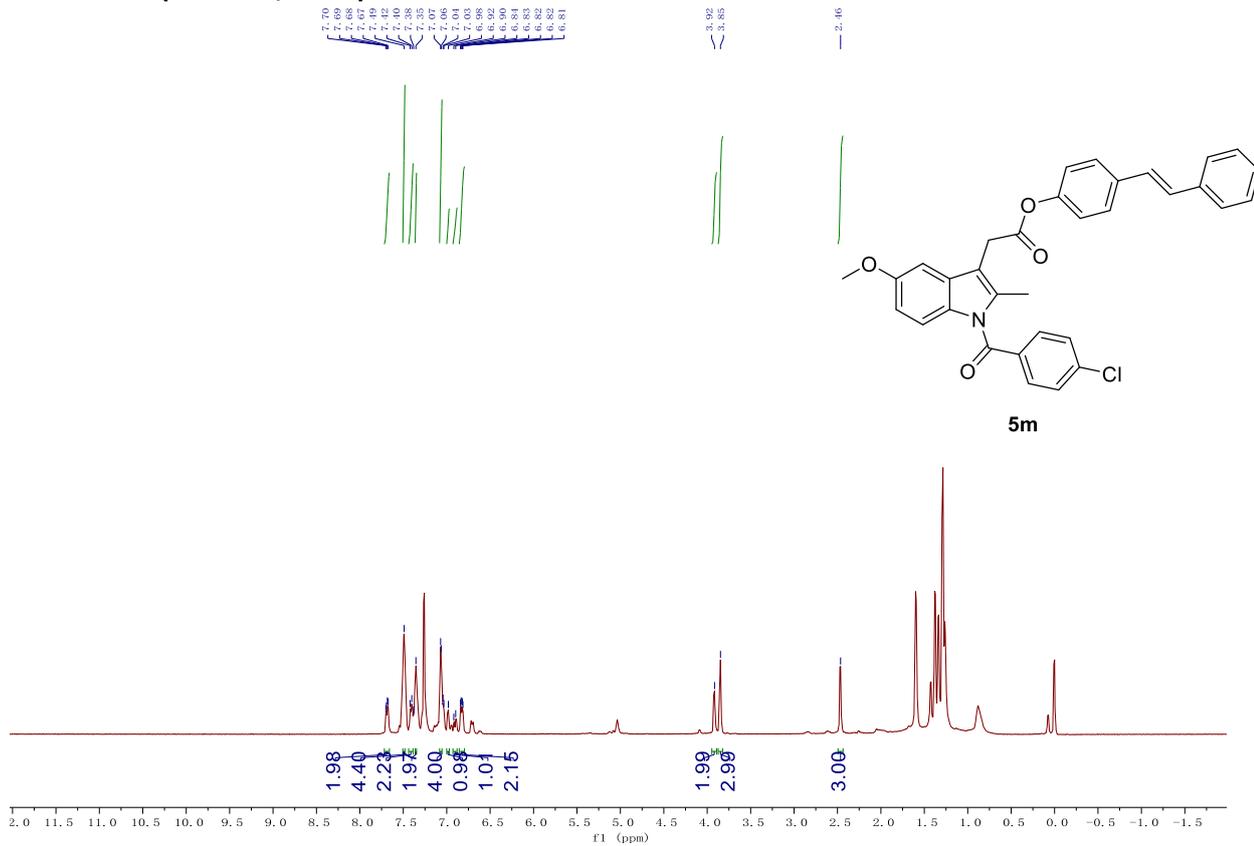
5l: ^1H NMR (400 MHz, CDCl_3)



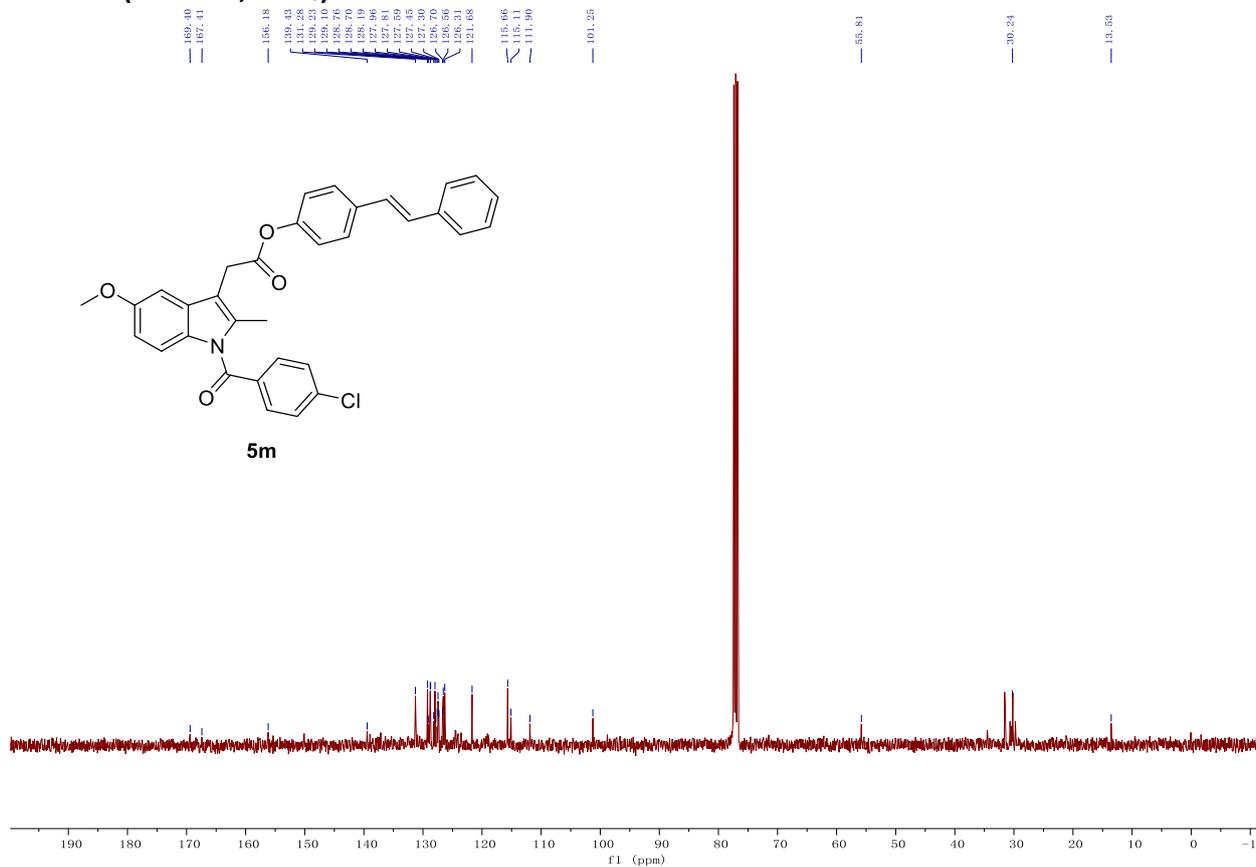
51: ¹³C NMR (100 MHz, CDCl₃)



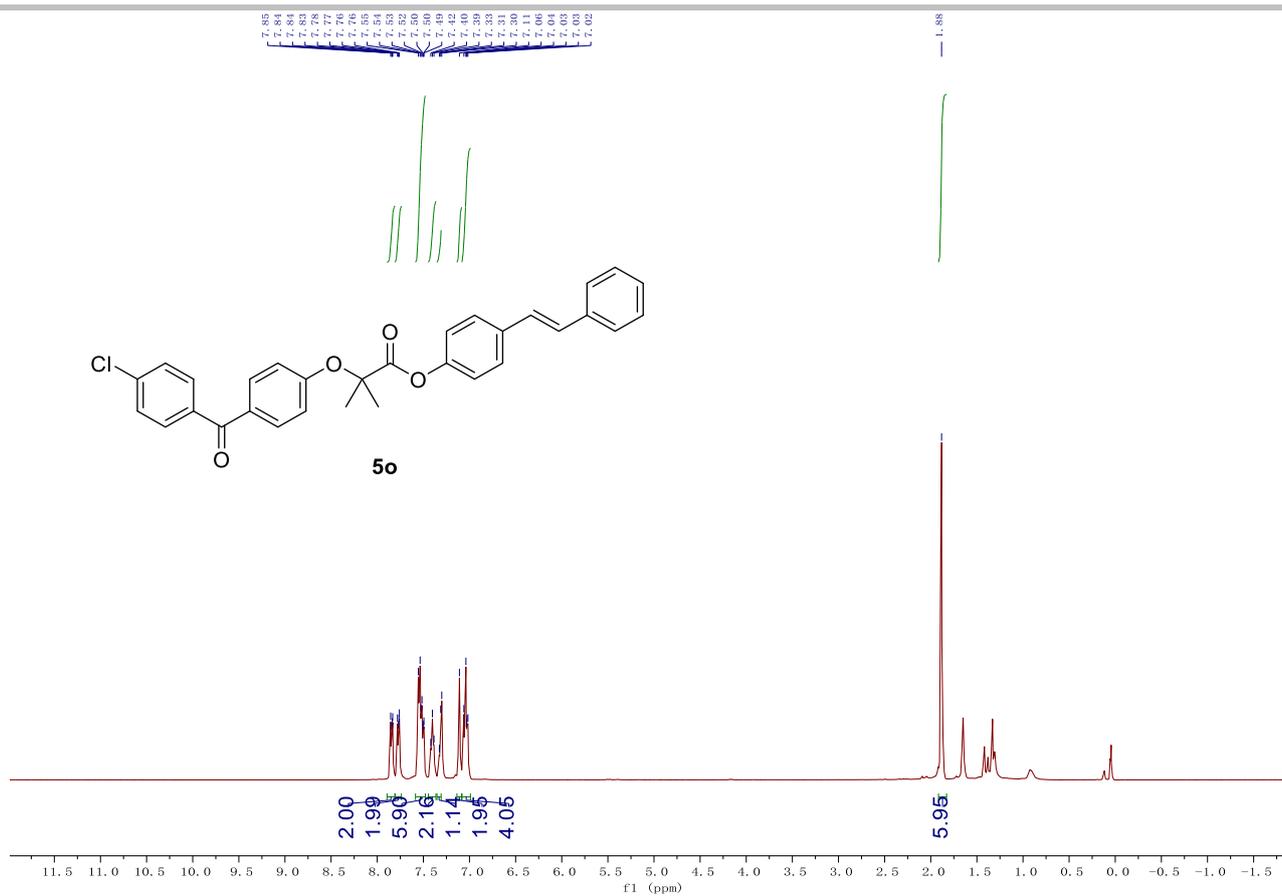
5m: ¹H NMR (400 MHz, CDCl₃)



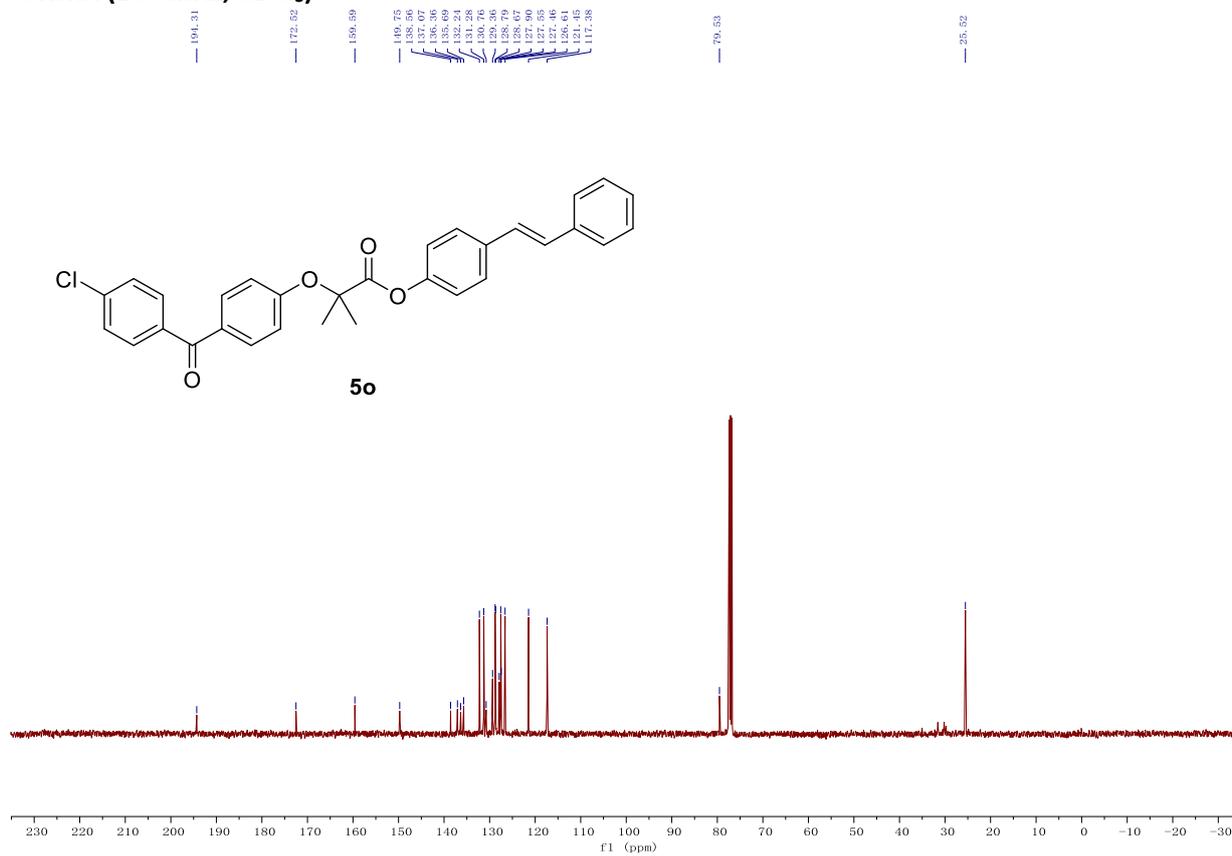
5m: ¹³C NMR (100 MHz, CDCl₃)



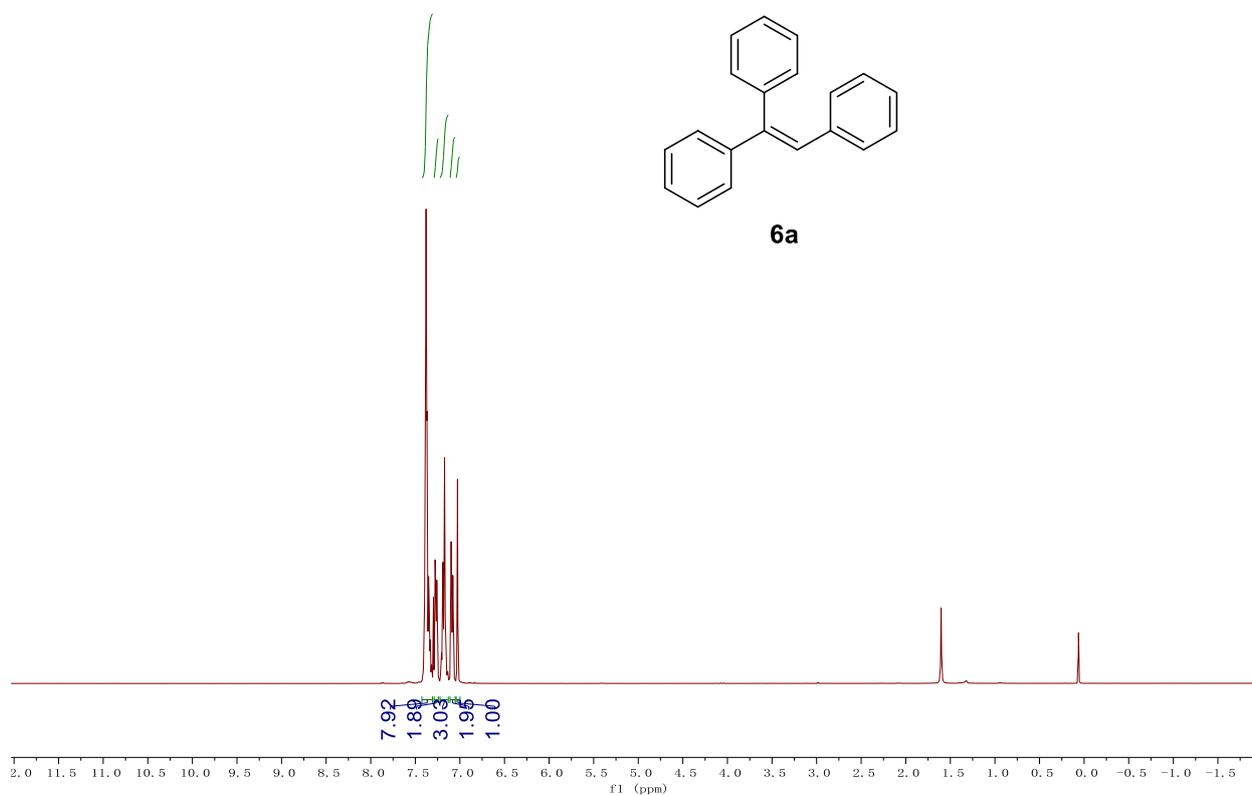
5n: ¹H NMR (400 MHz, CDCl₃)



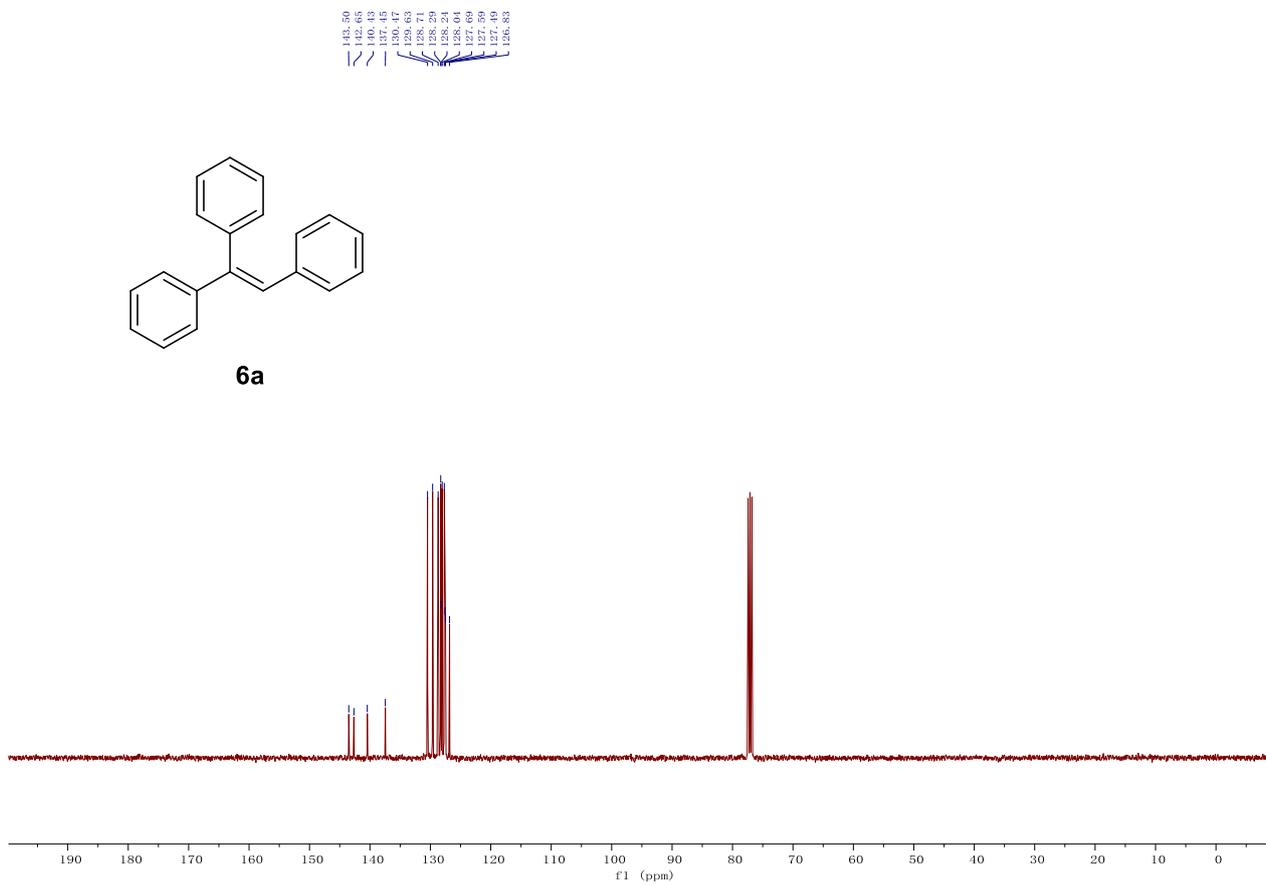
5o: ¹³C NMR (100 MHz, CDCl₃)



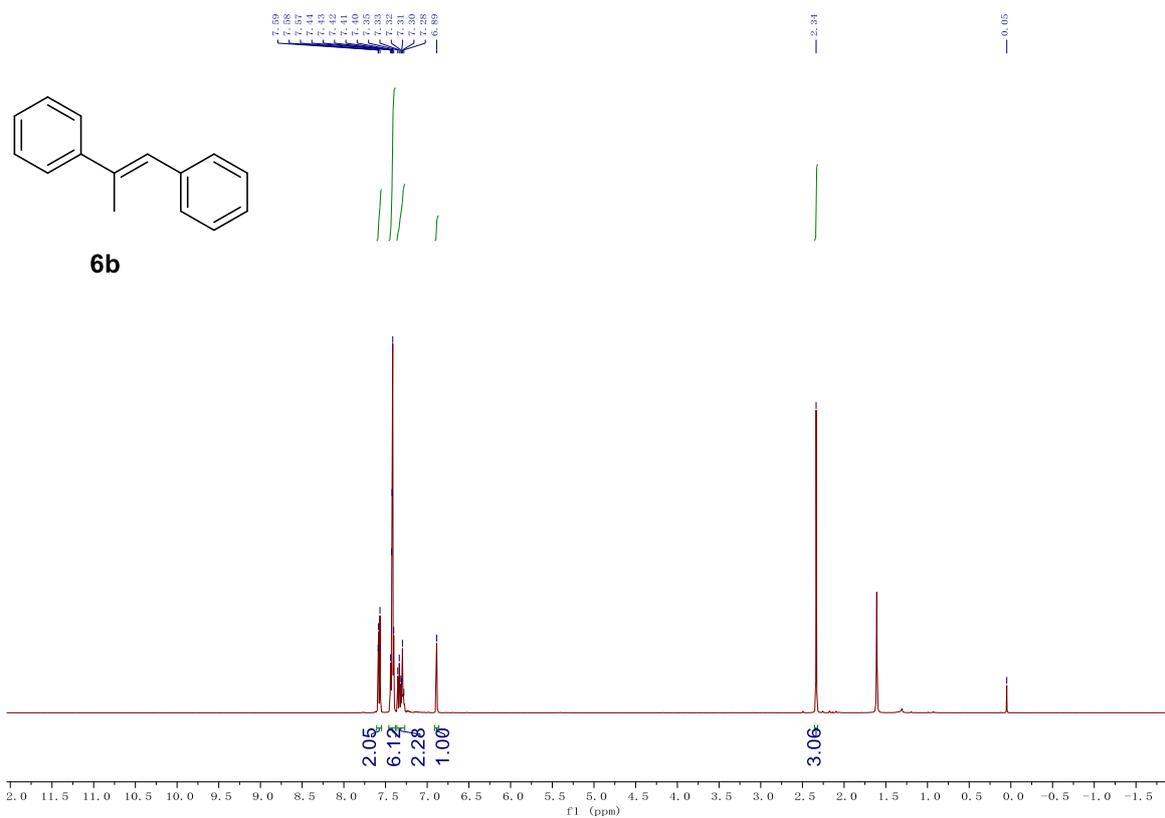
6a: ¹H NMR (400 MHz, CDCl₃)



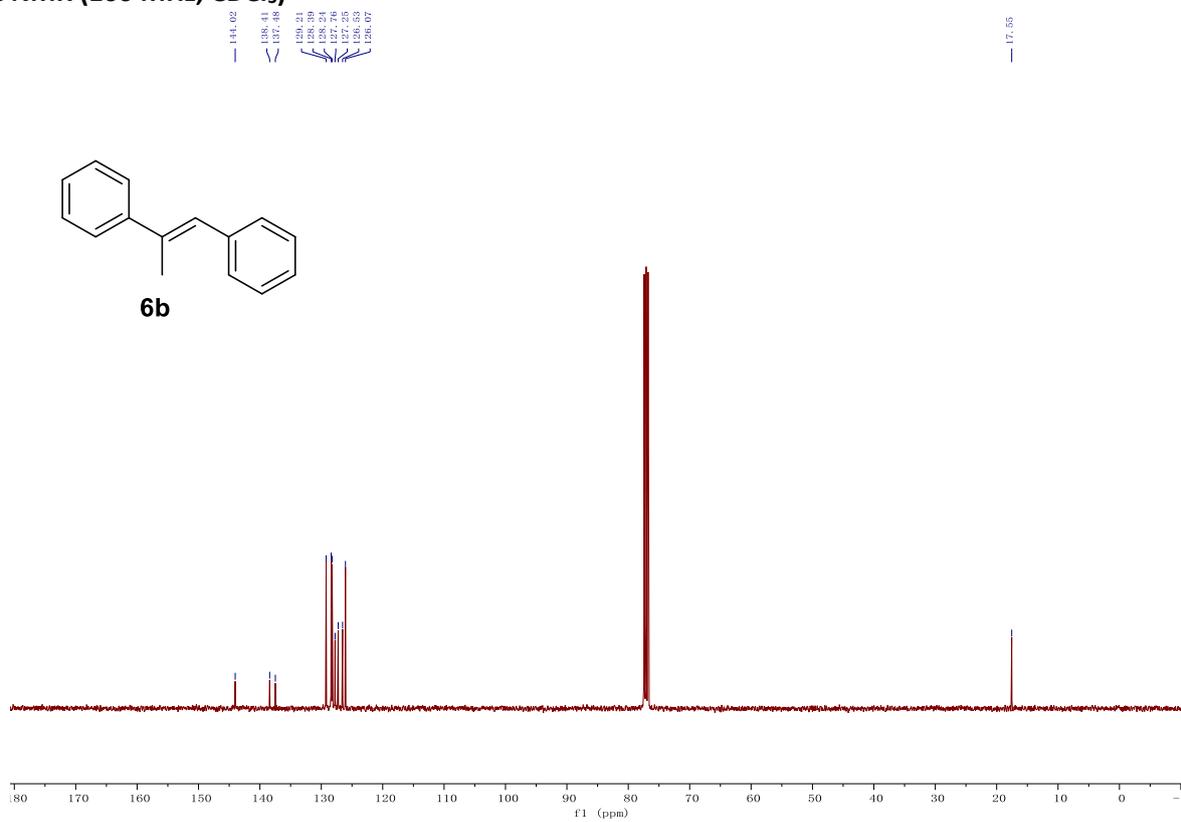
6a: ¹³C NMR (100 MHz, CDCl₃)



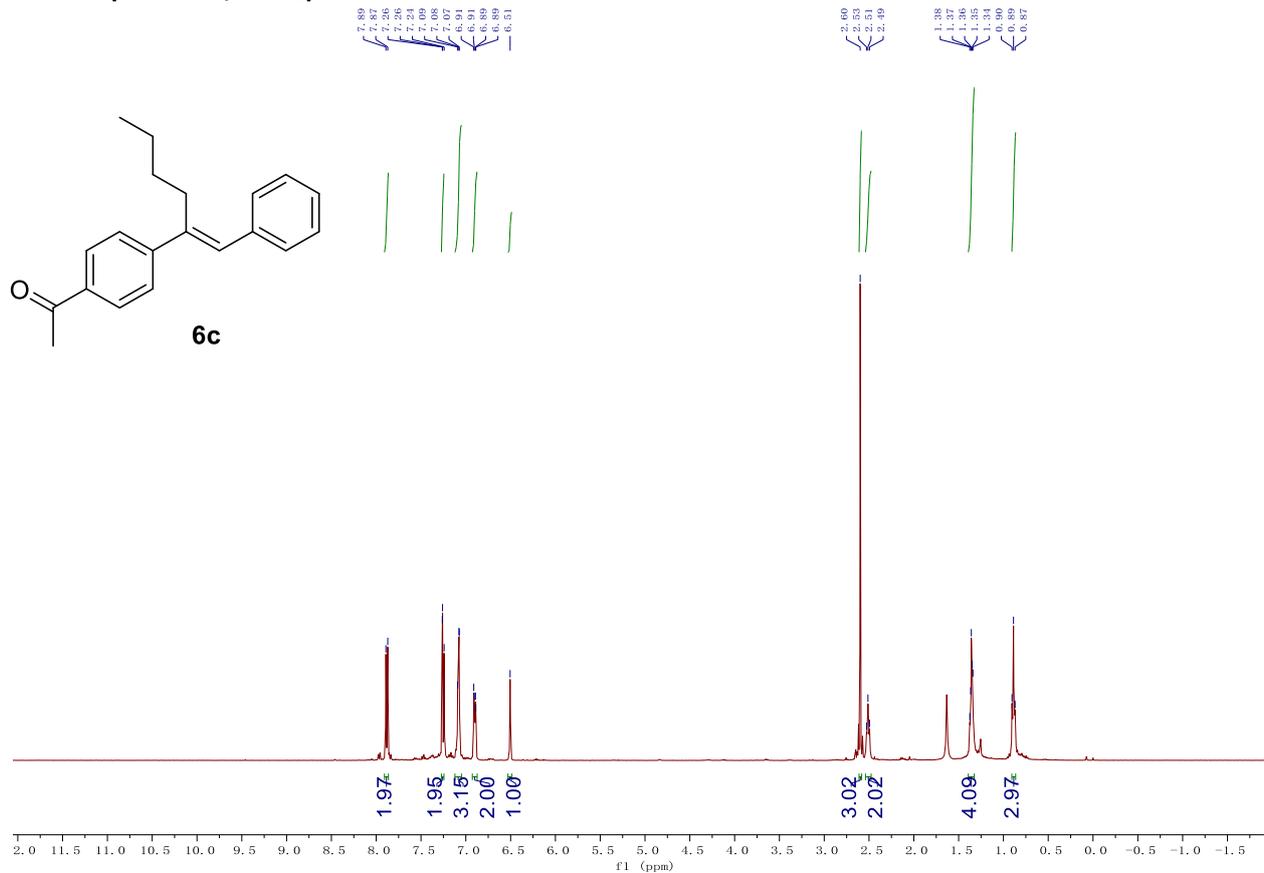
6b: ¹H NMR (400 MHz, CDCl₃)



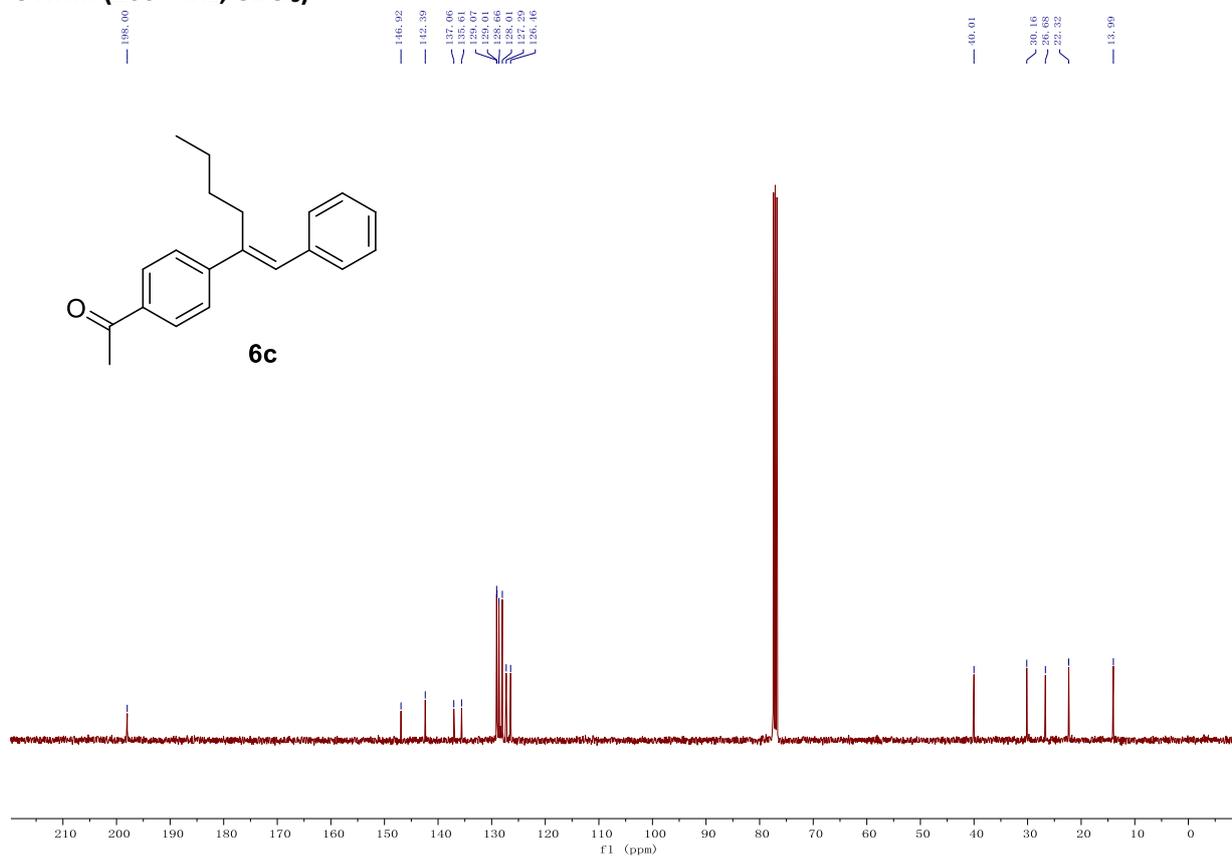
6b: ¹³C NMR (100 MHz, CDCl₃)



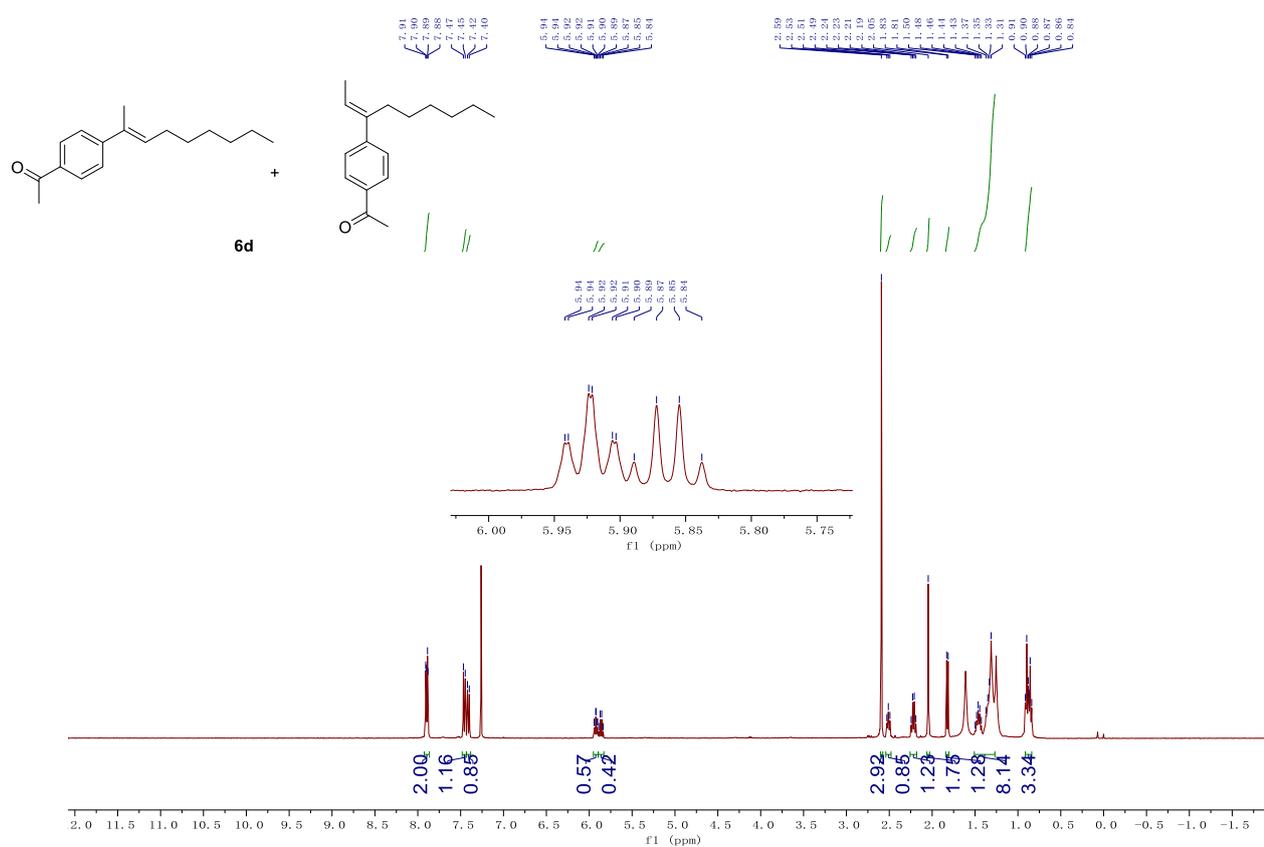
6c: ¹H NMR (400 MHz, CDCl₃)



6c: ¹³C NMR (100 MHz, CDCl₃)



6d: ¹H NMR (400 MHz, CDCl₃)



6d: ¹³C NMR (100 MHz, CDCl₃)

