Supporting Information

Controllable Carbon-Increasing Homologation of Electron-Deficient Alkenes via a Sew-and-Cut Strategy

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Contents

1.General experiment details and materials

All reagents were purchased from commercial suppliers and used without further purification. Flash chromatography was carried out with silica gel (200-300 mesh). Analytical TLC was performed with silica gel GF254 plates, and the products were visualized by UV detection. ^{1}H NMR, ^{13}C NMR, ^{19}F NMR and ^{31}P NMR spectra were detected in $CDCl_3$ or D_2O on a Bruker AM 400 MHz spectrometer. Chemical shifts in ^{1}H NMR spectra were reported in parts per million (ppm) on the δ scale from an internal standard of residual TMS (0 ppm) or D_2O (4.79 ppm). Data for ^{1}H NMR were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, brs = broad single), coupling constant in Herts (Hz) and integration. Data for ^{13}C NMR spectra were reported in terms of chemical shift in ppm from the central peak of $CDCl_3$ (77.0 ppm). Data for ^{19}F NMR were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant in Herts (Hz) and integration. The high-resolution mass spectra (HRMS) were measured on a Bruker Daltonics APEX II 47e spectrometer by ESI. UV/Vis absorption spectra were recorded on a Lambda 950+Refle UV/Vis spectrometer. Photochemical reactions were carried with 40 W blue LED and 12 W ultraviolet light LEDs obtained from SuZhou Uking Photoelectric Technology Co., Ltd.

2 General setup for the reaction

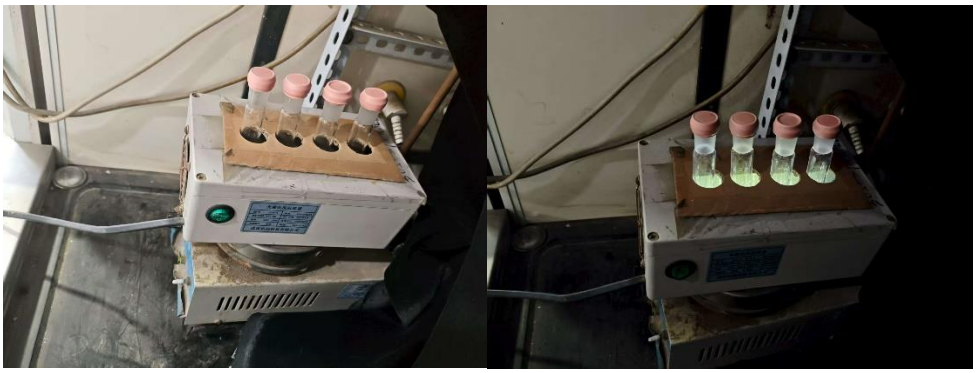
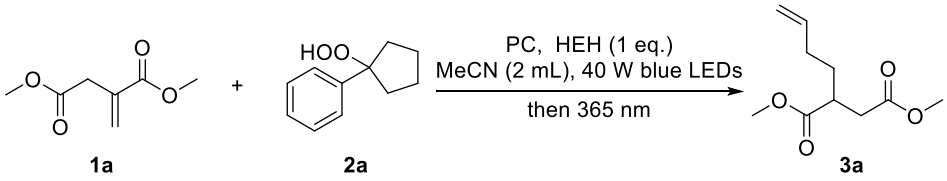


Figure S1. General setup for the reaction. A variable temperature photoreactor equipped with 12 W blue LEDs (λmax = 365 nm) was used for the reaction.

3 Optimization of reaction conditions

Table S1. Screening of photocatalyst[a]

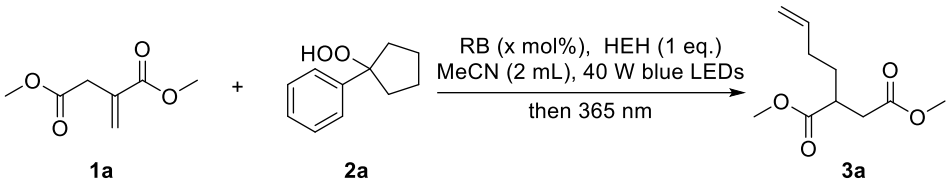


1a **2a** **3a**

Entry	Photocatalyst (5 mol%)	Yield (%)[b]
1	**Rhodamine B**	**53**
2	EY	49
3	4CzIPN	47
4	*fac*-Ir	39

[a]Reaction conditions: a mixture of **1a** (0.2 mmol, 1 eq.), **2a** (0.3 mmol, 1.5 eq.), HEH (0.2 mmol, 1 eq.), MeCN (2 mL) and PC (5 mol%) was irradiated with 40 W blue LEDs (456 nm) for 12 h at room temperature, then 12 W LEDs (365 nm) for 6 h at room temperature. [b]Yields were determined by analysis of the ^{1}HNMR spectra of the reaction mixture using 1,3,5-trimethoxybenzene as an internal standard.

Table S2. Screening of the amount of photocatalyst[a]

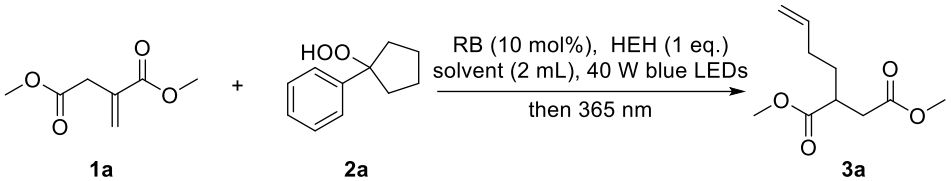


1a **2a** **3a**

Entry	Rhodamine B (x mol%)	Yield (%)[b]
1	2.5	50
2	5	53
3	**10**	**60**
4	20	61

[a]Reaction conditions: a mixture of **1a** (0.2 mmol, 1 eq.), **2a** (0.3 mmol, 1.5 eq.), HEH (0.2 mmol, 1 eq.) and MeCN (2 mL) and RB (x mol%) was irradiated with 40 W blue LEDs (456 nm) for 12 h at room temperature, then 12 W LEDs (365 nm) for 6 h at room temperature. [b]Yields were determined by analysis of the ^{1}HNMR spectra of the reaction mixture using 1,3,5-trimethoxybenzene as an internal standard.

Table S3. Screening of the solvent[a]

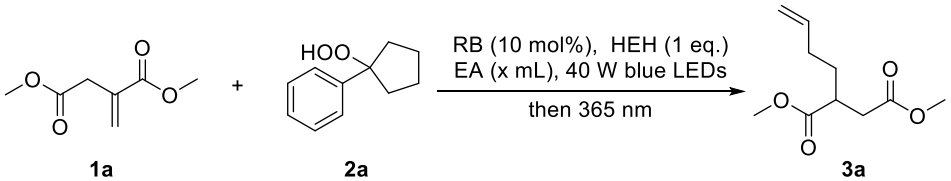


1a **2a** **3a**

Entry	solvent	Yield (%)[b]
1	**EA**	**64**
2	DMA	36
3	THF	39
4	PhMe	35
5	MeCN	60
6	acetone	58
7	MeOH	36

[a]Reaction conditions: a mixture of **1a** (0.2 mmol, 1 eq.), **2a** (0.3 mmol, 1.5 eq.), HEH (0.2 mmol, 1 eq.) and solvent (2 mL) and RB (10 mol%) was irradiated with 40 W blue LEDs (456 nm) for 12 h at room temperature, then 12 W LEDs (365 nm) for 6 h at room temperature. [b]Yields were determined by analysis of the ^{1}HNMR spectra of the reaction mixture using 1,3,5-trimethoxybenzene as an internal standard.

Table S4. Screening of the amount of solvent[a]



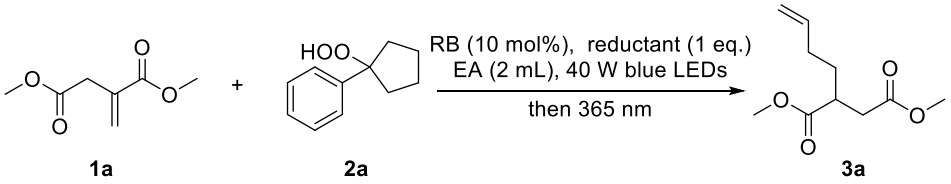
1a **2a** **3a**

Entry	EA (x mL)	Yield (%)[b]
1	0.5	55
2	1	60
3	**2**	**64**
4	4	63

[a]Reaction conditions: a mixture of **1a** (0.2 mmol, 1 eq.), **2a** (0.3 mmol, 1.5 eq.), HEH (0.2 mmol, 1 eq.)

and EA (x mL) and RB (10 mol%) was irradiated with 40 W blue LEDs (456 nm) for 12 h at room temperature, then 12 W LEDs (365 nm) for 6 h at room temperature. [b]Yields were determined by analysis of the ^{1}HNMR spectra of the reaction mixture using 1,3,5-trimethoxybenzene as an internal standard.

Table S5. Screening of the reductant[a]

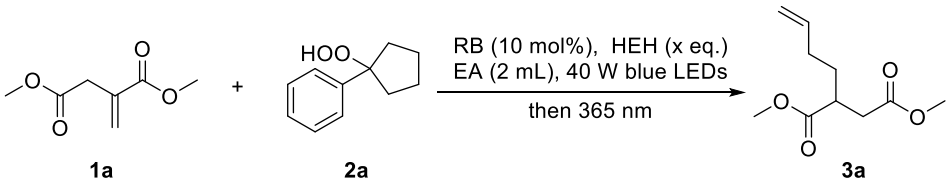


1a **2a** **3a**

Entry	reductant	Yield (%)[b]
1	**HEH**	**64**
2	DIPEA	55
3	TEA	54
4	γ-terpinene	42

[a]Reaction conditions: a mixture of **1a** (0.2 mmol, 1 eq.), **2a** (0.3 mmol, 1.5 eq.), reductant (0.2 mmol, 1 eq.) and EA (2 mL) and RB (10 mol%) was irradiated with 40 W blue LEDs (456 nm) for 12 h at room temperature, then 12 W LEDs (365 nm) for 6 h at room temperature. [b]Yields were determined by analysis of the ^{1}HNMR spectra of the reaction mixture using 1,3,5-trimethoxybenzene as an internal standard.

Table S6. Screening of the amount of reductant[a]

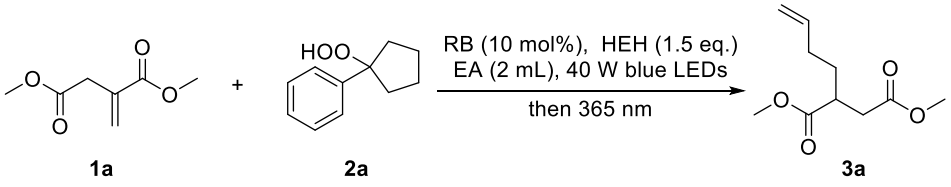


1a **2a** **3a**

Entry	HEH (x eq.)	Yield (%)[b]
1	1	64
2	**1.5**	**68**
3	2	65
4	2.5	66

[a]Reaction conditions: a mixture of **1a** (0.2 mmol, 1 eq.), **2a** (0.3 mmol, 1.5 eq.), HEH (x eq.) and EA (2 mL) and RB (10 mol%) was irradiated with 40 W blue LEDs (456 nm) for 12 h at room temperature, then 12 W LEDs (365 nm) for 6 h at room temperature. [b]Yields were determined by analysis of the ^{1}HNMR spectra of the reaction mixture using 1,3,5-trimethoxybenzene as an internal standard.

Table S7. Screening of the amount of 2a[a]



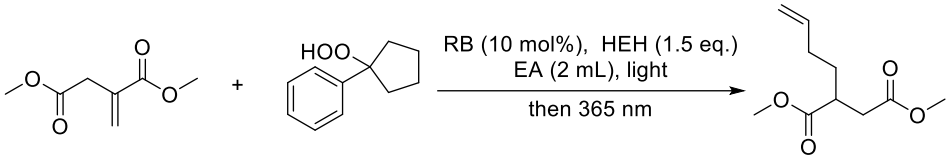
1a **2a** **3a**

Entry	**2a** (x eq.)	Yield (%)[b]
1	1	48
2	1.5	68
3	**1.8**	**74**

4	2.1	72

[a]Reaction conditions: a mixture of **1a** (0.2 mmol, 1 eq.), **2a** (x eq.), HEH (1.5 eq.) and EA (2 mL) and RB (10 mol%) was irradiated with 40 W blue LEDs (456 nm) for 12 h at room temperature, then 12 W LEDs (365 nm) for 6 h at room temperature. [b]Yields were determined by analysis of the ^{1}HNMR spectra of the reaction mixture using 1,3,5-trimethoxybenzene as an internal standard.

Table S8. Screening of the amount of light source[a]

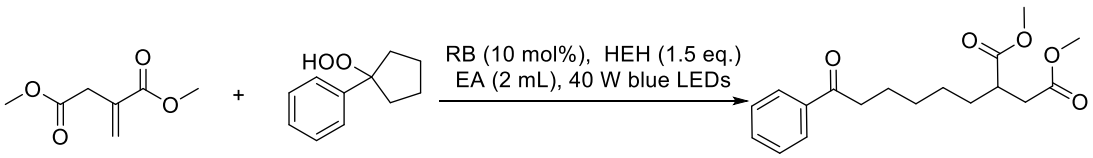


1a **2a** **3a**

Entry	Light source (x nm)	Yield (%)[b]
1	**456**	**74**
2	427	70
3	405	68
7	397	67

[a]Reaction conditions: a mixture of **1a** (0.2 mmol, 1 eq.), **2a** (x eq.), HEH (1.5 eq.) and EA (2 mL) and RB (10 mol%) was irradiated with 40 W blue LEDs (x nm) for 12 h at room temperature, then 12 W LEDs (365 nm) for 6 h at room temperature. [b]Yields were determined by analysis of the ^{1}HNMR spectra of the reaction mixture using 1,3,5-trimethoxybenzene as an internal standard.

Table S9. Control experiments for intermediate[a]

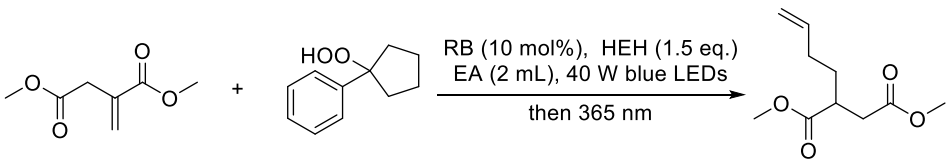


1a **2a** **6a**

Entry	Deviation	**6a** Yield (%)[b]
1	**none**	**84**
2	without HEH	n.d.
3	without Rhodamine B	36
4	without light	18
6	70 °C instead of 40 W LEDs	63
8	in air	53

[a]Reaction conditions: a mixture of **1a** (0.2 mmol, 1 eq.), **2a** (1.8 eq.), HEH (1.5 eq.) and EA (2 mL) and RB (10 mol%) was irradiated with 40 W blue LEDs (456 nm) for 12 h at room temperature. [b]Isolated yields.

Table S10. Control experiments for 3a[a]



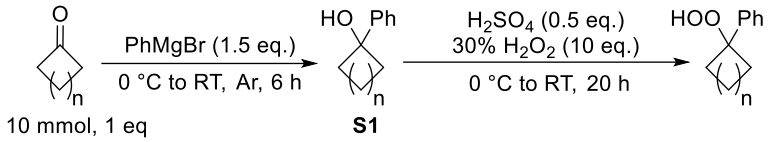
1a **2a** **3a**

Entry	Deviation	Yield (%)[b]

1	**none**	**74 (70)[c]**
2	without HEH	n.d.
3	without 365 nm	n.d.
4	365 nm lamp direct irradiation	68[d]
5	in air	48

[a]Reaction conditions: a mixture of **1a** (0.2 mmol, 1 eq.), **2a** (1.8 eq.), HEH (1.5 eq.) and EA (2 mL) and RB (10 mol%) was irradiated with 40 W blue LEDs (456 nm) for 12 h at room temperature, then 12 W LEDs (365 nm) for 6 h at room temperature. [b]Yields were determined by analysis of the ^{1}HNMR spectra of the reaction mixture using 1,3,5-trimethoxybenzene as an internal standard. [d]The system reacted directly under 365nm radiation without the need to switch light sources for 18 hours at room temperature.

4 Synthesis of starting materials



10 mmol, 1 eq **S1**

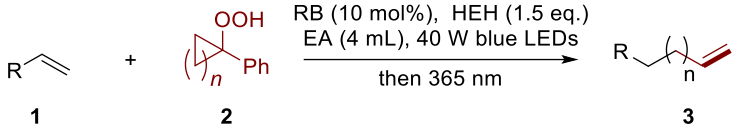
According to relevant literature reports,[1]-[3] the general synthesis process of peroxides is as follows:

Cyclopentanone (10 mmol, 1.0 eq.) was added drop-wise to a 1.0 M solution of the phenylmagnesium bromide at 0 °C under argon atmosphere. After being stirred at room temperature for 6 h, the reaction mixture was quenched with sat. NH_4Cl aq. at 0 °C and extracted with a solution of hexane / EA (4 / 1) three times. The combined organic layer was dried over Na_2SO_4 and concentrated. The crude product **S1** was used for the next step without further purification.

To **S1** in a flask was added a solution of H_2SO_4 / 30% H_2O_2 aq. (0.3 mL / 10 mL) slowly at 0 °C. After being stirred vigorously at room temperature for 20 h, the reaction mixture was diluted with H_2O (10 mL) and then extracted with DCM three times. The combined organic layer was dried over Na_2SO_4 and concentrated. The combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated to afford residue, which was purified by column chromatography on silica gel (petroleum ether/ethyl acetate) to give the target products.

5 General procedure for synthesis of products

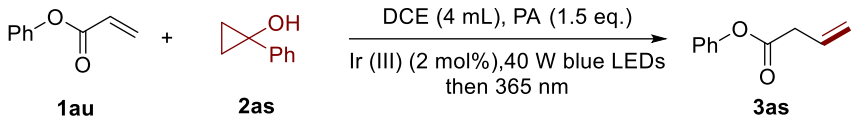
Conditions A:



1 **2** **3**

In an oven dried 10 mL screw-caped tube equipped with a magnetic stirring bar, **1** (0.4 mmol, 1 eq.) (if solid), **2** (0.72 mmol, 1.8 eq.) (if solid), HEH (0.6 mmol, 1.5 eq.) and RB (0.04mmol, 10 mol%) were added. The tube was evacuated and back-filled with argon for 3 times. Subsequently, EA (4 mL), **1** (0.4 mmol, 1 eq.) (if liquid) and **2** (0.72 mmol, 1.8 eq.) (if liquid) were added under argon. The reaction mixture was irradiated with 40 W blue LEDs (456 nm) and monitored by TLC. After complete consumption of olefin **1**, the system was irradiated with 12 W ultraviolet light LEDs (365 nm) and monitored by TLC. Finally, the solution was concentrated under in vacuo and the crude product was purified by flash column chromatography (gradient eluent of ethyl acetate and petroleum ether) to give the corresponding product **3**. The identity and purity of the product was confirmed by ^{1}H and ^{13}C NMR spectroscopic analysis.

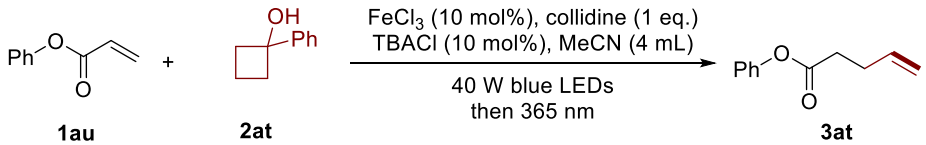
Conditions B:



1au **2as** **3as**

In an oven dried 10 mL screw-caped tube equipped with a magnetic stirring bar, PA (1,1'-Binaphthyl-2,2'-diylhydrogenphosphate) (0.1 mmol, 0.25 eq.) and Ir (III) ($[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$) (2 mol%) were added. The tube was evacuated and back-filled with argon for 3 times. Subsequently, DCE (4 mL) and **1au** (0.4 mmol, 1 eq.) and **2as** (1.2 mmol, 3 eq.) were added under argon. The reaction mixture was stirred at room temperature and monitored by TLC under irradiation from 40 W blue LEDs (456 nm). After complete consumption of olefin **1au**, the system was irradiated with 12 W ultraviolet light LEDs (365 nm) and monitored by TLC. Finally, the solution was concentrated under in vacuo and the crude product was purified by flash column chromatography (gradient eluent of ethyl acetate and petroleum ether) to give the corresponding product **3as**. The identity and purity of the product was confirmed by ^{1}H and ^{13}C NMR spectroscopic analysis.

Conditions C:

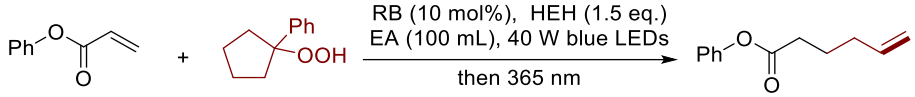


1au **2at** **3at**

In an oven dried 10 mL screw-caped tube equipped with a magnetic stirring bar, $FeCl_3$ (0.04 mmol, 10 mol%), TBACl (tetrabutyl ammonium chloride) (0.04 mmol, 10 mol%) were added. The tube was evacuated and back-filled with argon for 3 times. Subsequently, MeCN (4 mL), collidine (0.4 mmol, 1 eq.), **1au** (0.4 mmol, 1 eq.) and **2at** (1.2 mmol, 3 eq.) were added under argon. The reaction mixture was stirred at room temperature and monitored by TLC under irradiation from 40 W blue LEDs (456 nm). After complete consumption of olefin **1au**, the system was irradiated with 12 W ultraviolet light LEDs (365 nm) and monitored by TLC. Finally, the solution was concentrated under in vacuo and the crude product was purified by flash column chromatography (gradient eluent of ethyl acetate and petroleum ether) to give the corresponding product **3at**. The identity and purity of the product was confirmed by ^1H and ^{13}C NMR spectroscopic analysis.

5 Scale-up experiment

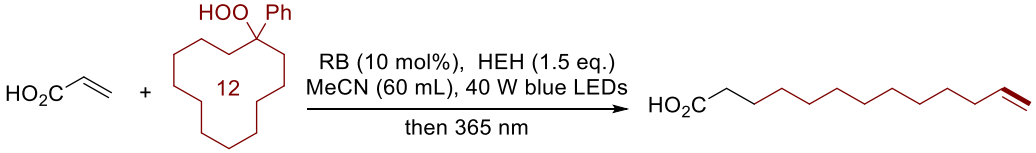
Gram-scale synthesis of 3au:



1au **2a** **3au**, 1.2 g, 63%

In an oven dried 200 mL round bottom flask equipped with a magnetic stirring bar, HEH (4.60 g, 15 mmol, 1.5 eq.) and RB (479 mg, 1 mmol, 10 mol%) were added. The tube was evacuated and back-filled with argon for 3 times. Subsequently, EA (100 mL), **1au** (1.18 g, 10 mmol, 1 eq.) and **2a** (3.21 g, 18 mmol, 1.8 eq.) were added under argon. The reaction mixture was irradiated with 40 W blue LEDs (456 nm) and monitored by TLC. After complete consumption of olefin **1au**, the system was irradiated with 40 W ultraviolet light LEDs (365 nm) and monitored by TLC. Finally, the solution was concentrated under in vacuo and the crude product was purified by flash column chromatography (gradient eluent of ethyl acetate and petroleum ether) to give the corresponding product **3au** (1.2 g, 63%).

Scale-up synthesis of 3bn:

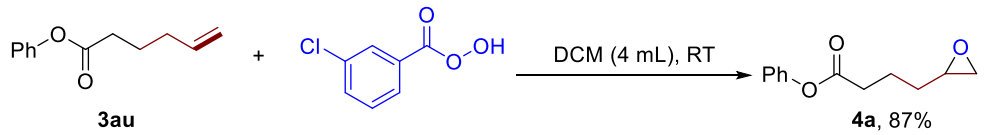


1bn **2bn** **3bn**, 701 mg, 55%

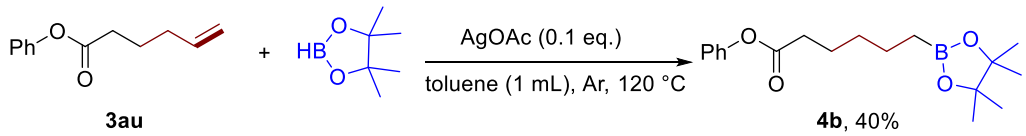
In an oven dried 200 mL round bottom flask equipped with a magnetic stirring bar, HEH (2.28 g, 9 mmol, 1.5 eq.), RB (287 mg, 0.6 mmol, 10 mol%) and **2bn** (2.99 g, 10.8 mmol, 1.8 eq.) were added. The tube was evacuated and back-filled with argon for 3 times.

Subsequently, MeCN (60 mL) and **1bn** (432 mg, 6 mmol, 1 eq.) were added under argon. The reaction mixture was irradiated with 40 W blue LEDs (456 nm) and monitored by TLC. After complete consumption of olefin **1bn**, the system was irradiated with 40 W ultraviolet light LEDs (365 nm) and monitored by TLC. Finally, the solution was concentrated under in vacuo and the crude product was purified by flash column chromatography (gradient eluent of ethyl acetate and petroleum ether) to give the corresponding product **3bn** (701 mg, 55%).

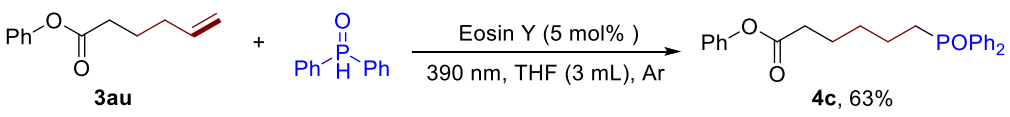
6 Follow-up transformations



To a solution of the **3au** (76 mg, 0.4 mmol, 1.0 eq.) in DCM (4 mL) was added *m*CPBA (138 mg, 0.8 mmol, 2 eq.) at room temperature. Then, the mixture was stirred for 20 h at room temperature. After that, the resulting mixture was concentrated under in vacuo. The residue was purified by flash chromatography on silica gel (gradient eluent of petroleum ether / ethyl acetate) directly to give the desired product **4a** with 72 mg in 87% yield.

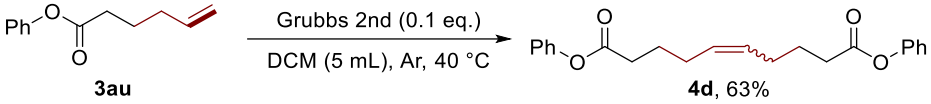


In an oven dried 10 mL screw-caped tube equipped with a magnetic stirring bar, pinacolborane (77 mg, 0.6 mmol, 1.5 eq.) and AgOAc (7 mg, 0.04 mmol, 0.1 eq.) were added. The tube was evacuated and back-filled with argon for 3 times. Subsequently, toluene (1 mL) and **3au** (76 mg, 0.4 mmol, 1.0 eq.) were added under argon. The reaction mixture was allowed to stir at 120 °C for 12 h. Finally, the solution was concentrated under in vacuo and the crude product was purified by flash column chromatography (gradient eluent of ethyl acetate and petroleum ether) to give the corresponding product **4b** with 51 mg in 40% yield.



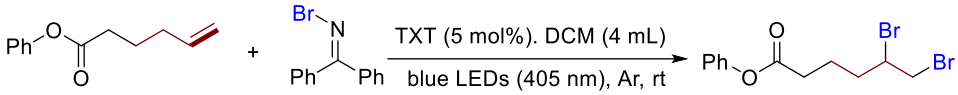
In an oven dried 10 mL screw-caped tube equipped with a magnetic stirring bar, diphenylphosphine oxide (324 mg, 1.6 mmol, 4 eq.) and Eosin Y (13 mg, 0.02 mmol, 5 mol%) was added. The tube was evacuated and back-filled with argon for 3 times.

Subsequently, THF (3 mL) and **3au** (76 mg, 0.4 mmol, 1.0 eq.) were added under argon. The reaction mixture was allowed to stir at room temperature for 20 h monitoring the conversion *via* TLC. Finally, the solution was concentrated under in vacuo and the crude product was purified by flash column chromatography (gradient eluent of ethyl acetate and petroleum ether) to give the corresponding product **4c** with 99 mg in 63% yield.



3au **4d**, 63%

In an oven dried 10 mL screw-caped tube equipped with a magnetic stirring bar, Grubbs 2nd (34 mg, 0.04 mmol, 0.1 eq.) was added. The tube was evacuated and back-filled with argon for 3 times. Subsequently, DCM (5 mL) and **3au** (76 mg, 0.4 mmol, 1.0 eq.) were added under argon. The reaction mixture was allowed to stir at 40 °C for 24 h monitoring the conversion *via* TLC. Finally, the solution was concentrated under in vacuo and the crude product was purified by flash column chromatography (gradient eluent of ethyl acetate and petroleum ether) to give the corresponding product **4d** with 44 mg in 63% yield.

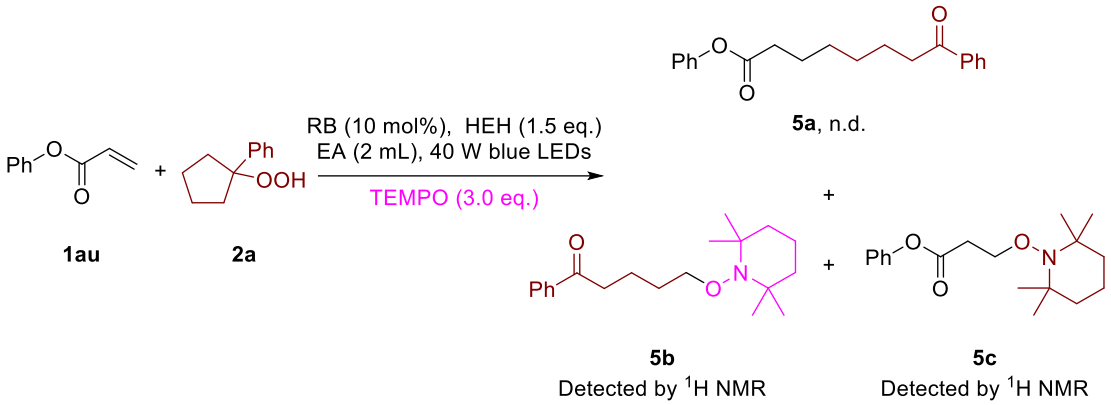


3au NBB **4e,** 78 %

In an oven dried 10 mL screw-caped tube equipped with a magnetic stirring bar, thioxanthone (TXT, 4.2 mg, 0.02 mmol, 5 mol%) and N-bromo-benzophenonimines (NBB, 260 mg, 1 mmol, 2.5 eq.) was added. The tube was evacuated and back-filled with argon for 3 times. Subsequently, DCM (4 mL) and **3au** (76 mg, 0.4 mmol, 1.0 eq.) were added under argon. The reaction mixture was irradiated with 6 W blue LEDs (405 nm) and monitored by TLC. Finally, the solution was concentrated under in vacuo. The residue was purified by flash chromatography on silica gel (gradient eluent of petroleum ether / ethyl acetate) directly to give the desired product **4e** with 109 mg in 78% yield.

7 Mechanistic investigations

7.1 radical capture experiment



In an oven dried 10 mL screw-caped tube equipped with a magnetic stirring bar, HEH (76 mg, 0.3 mmol, 1.5 eq.), RB (10 mg, 0.02 mmol, 10 mol%) and TEMPO (94 mg, 0.6 mmol, 3 eq.) were added. The tube was evacuated and back-filled with argon for 3 times. Subsequently, EA (2 mL), **1au** (30 mg, 0.2 mmol, 1 eq.) and **2a** (64 mg, 0.36 mmol, 1.8 eq.) were added under argon. The reaction mixture was irradiated with 40 W blue LEDs (456 nm) and monitored by TLC. Finally, the solution was concentrated under in vacuo and the crude product was purified by flash column chromatography (gradient eluent of ethyl acetate and petroleum ether) to give a mixture of **5b** and **5c** (Due to their similar polarity, they cannot be completely separated.). The identity and purity of the product was confirmed by ^{1}H NMR spectroscopic analysis. The spectral data is consistent with literature reports.[4]

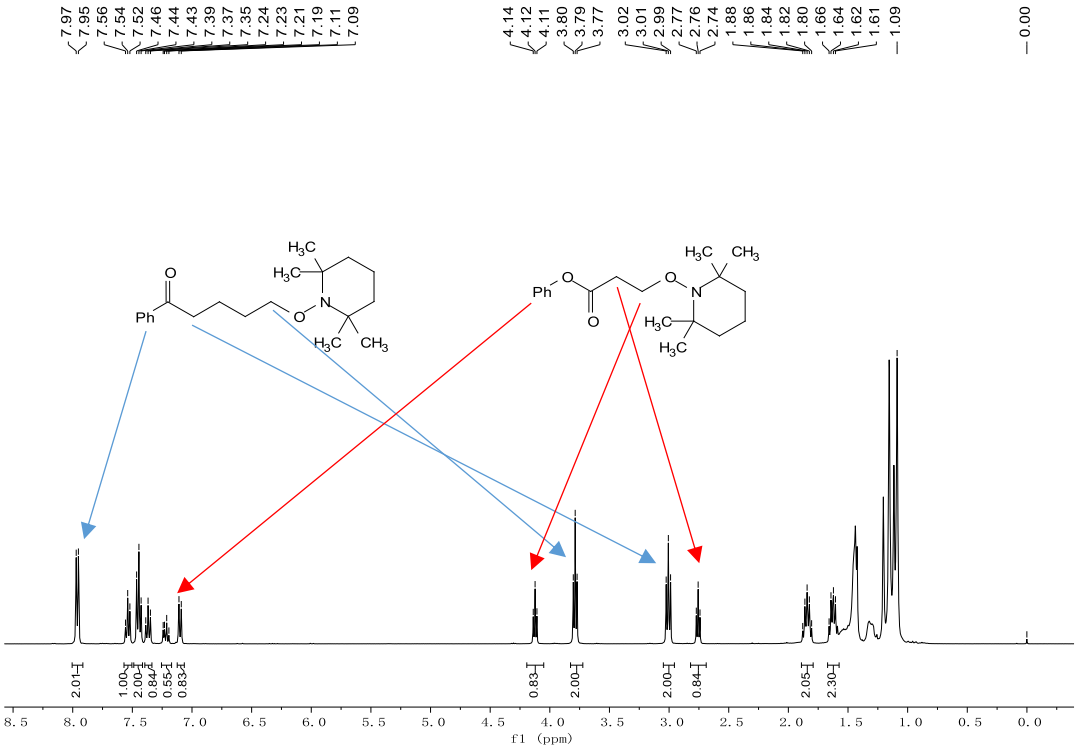
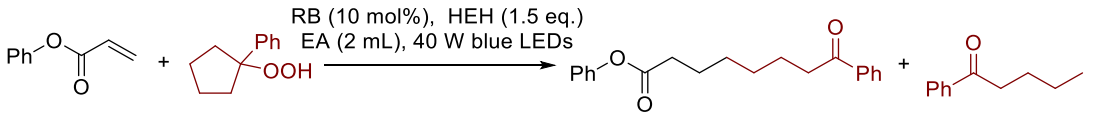
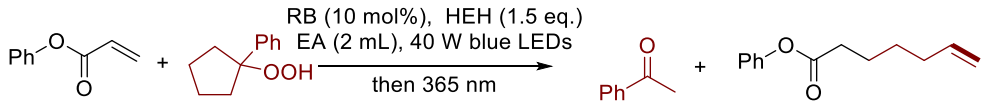


Figure S2: ^{1}H NMR of **5b** and **5c**.

7.2 Separation of intermediates and by-products

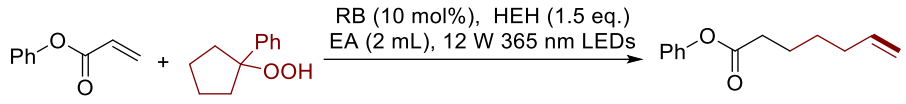


In an oven dried 10 mL screw-caped tube equipped with a magnetic stirring bar, HEH (76 mg, 0.3 mmol, 1.5 eq.) and RB (10 mg, 0.02 mmol, 10 mol%) were added. The tube was evacuated and back-filled with argon for 3 times. Subsequently, EA (2 mL), **1au** (30 mg, 0.2 mmol, 1 eq.) and **2a** (64 mg, 0.36 mmol, 1.8 eq.) were added under argon. The reaction mixture was irradiated with 40 W blue LEDs (456 nm) and monitored by TLC. Finally, the solution was concentrated under in vacuo and the crude product was purified by flash column chromatography (gradient eluent of ethyl acetate and petroleum ether) to give **5a** (48 mg, 78%) and **5d** (29 mg).



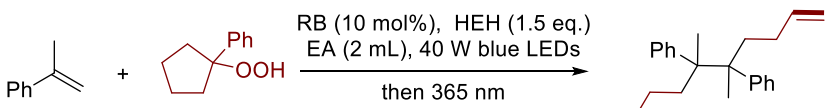
1au **2a** **5e**, 0.32 mmol, 39 mg **3au**, 71%

In an oven dried 10 mL screw-caped tube equipped with a magnetic stirring bar, HEH (76 mg, 0.3 mmol, 1.5 eq.) and RB (10 mg, 0.02 mmol, 10 mol%) were added. The tube was evacuated and back-filled with argon for 3 times. Subsequently, EA (2 mL), **1au** (30 mg, 0.2 mmol, 1 eq.) and **2a** (64 mg, 0.36 mmol, 1.8 eq.) were added under argon. The reaction mixture was irradiated with 40 W blue LEDs (456 nm) and monitored by TLC. After complete consumption of olefin **1au**, the system was irradiated with 12 W ultraviolet light LEDs (365 nm) and monitored by TLC. Finally, the solution was concentrated under in vacuo and the crude product was purified by flash column chromatography (gradient eluent of ethyl acetate and petroleum ether) to give **5e** (39 mg) and **3au** (29 mg, 71%).



1au **2a** **3au**, 60%

In an oven dried 10 mL screw-caped tube equipped with a magnetic stirring bar, HEH (76 mg, 0.3 mmol, 1.5 eq.) and RB (10 mg, 0.02 mmol, 10 mol%) were added. The tube was evacuated and back-filled with argon for 3 times. Subsequently, EA (2 mL), **1au** (30 mg, 0.2 mmol, 1 eq.) and **2a** (64 mg, 0.36 mmol, 1.8 eq.) were added under argon. The reaction mixture was irradiated with 12 W purple LEDs (365 nm) and monitored by TLC. Finally, the solution was concentrated under in vacuo and the crude product was purified by flash column chromatography (gradient eluent of ethyl acetate and petroleum ether) to give **3au** (24 mg, 60%).



2a **5f**, 61%

In an oven dried 10 mL screw-caped tube equipped with a magnetic stirring bar, HEH (76 mg, 0.3 mmol, 1.5 eq.) and RB (10 mg, 0.02 mmol, 10 mol%) were added. The tube was evacuated and back-filled with argon for 3 times. Subsequently, EA (2 mL), 2-phenyl-1-

propene (24 mg, 0.2 mmol, 1 eq.) and **2a** (64 mg, 0.36 mmol, 1.8 eq.) were added under argon. The reaction mixture was irradiated with 40 W blue LEDs (456 nm) and monitored by TLC. After complete consumption of olefin, the system was irradiated with 12 W ultraviolet light LEDs (365 nm) and monitored by TLC. Finally, the solution was concentrated under in vacuo and the crude product was purified by flash column chromatography (gradient eluent of ethyl acetate and petroleum ether) to give **5f** (20 mg, 61%). The identity and purity of the product was confirmed by ^{1}H and ^{13}C NMR spectroscopic analysis.

7.3 UV/Vis absorption spectroscopy

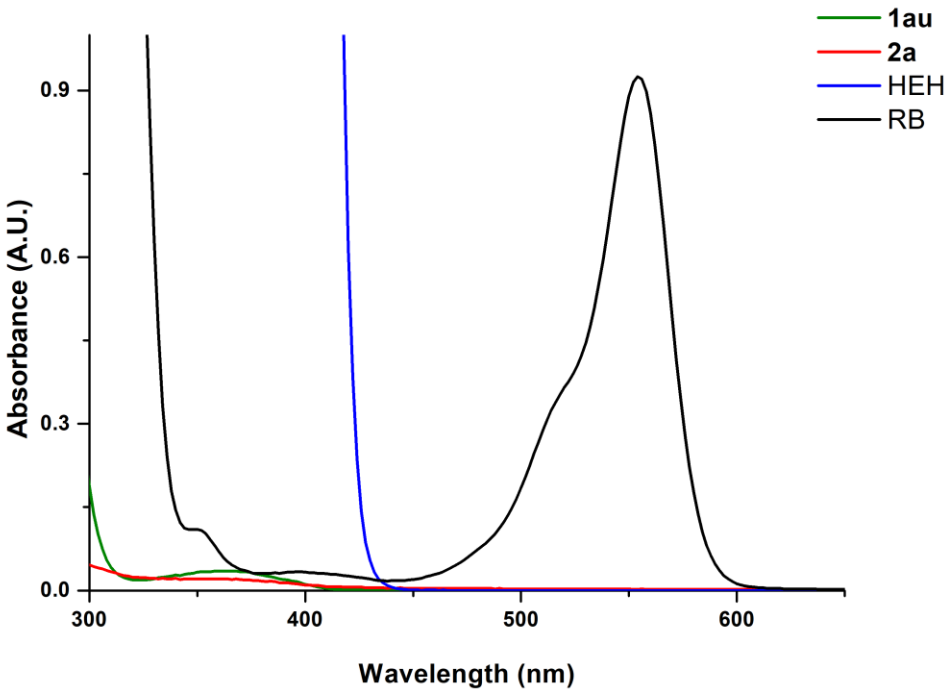


Figure S3. UV-Vis absorption spectra of photocatalyst **1au** (25 mM), **2a** (25 mM), HEH (25 mM) and Rhodamine B (0.05 mM) in deoxygenated EtOAc.

7.4 Fluorescence quenching experiment

Stern-Volmer luminescence quenching analysis was conducted using a Jasco FP-8300

spectrofluorometer. The following parameters were employed: Excitation bandwidth = 5nm, data interval = 5 nm, scan speed = 2000 nm/min. The solution of Rhodamine B (0.05 mM in EtOAc) were excited at λex = 456 nm and the emission was collected at 582 nm. The substrates 2a, HEH and phenyl acrylate **1au** were dissolved in EtOAc (500 mM), respectively. For each quenching experiment, 30 μL of the stock solution were titrated to a solution (3 mL) of thioxanthone. The addition of 30 μL stock solution refers to an increase of the quencher concentration of 5 mM. I_0 is the luminescence intensity without the quencher, I is the intensity in the presence of the quencher.

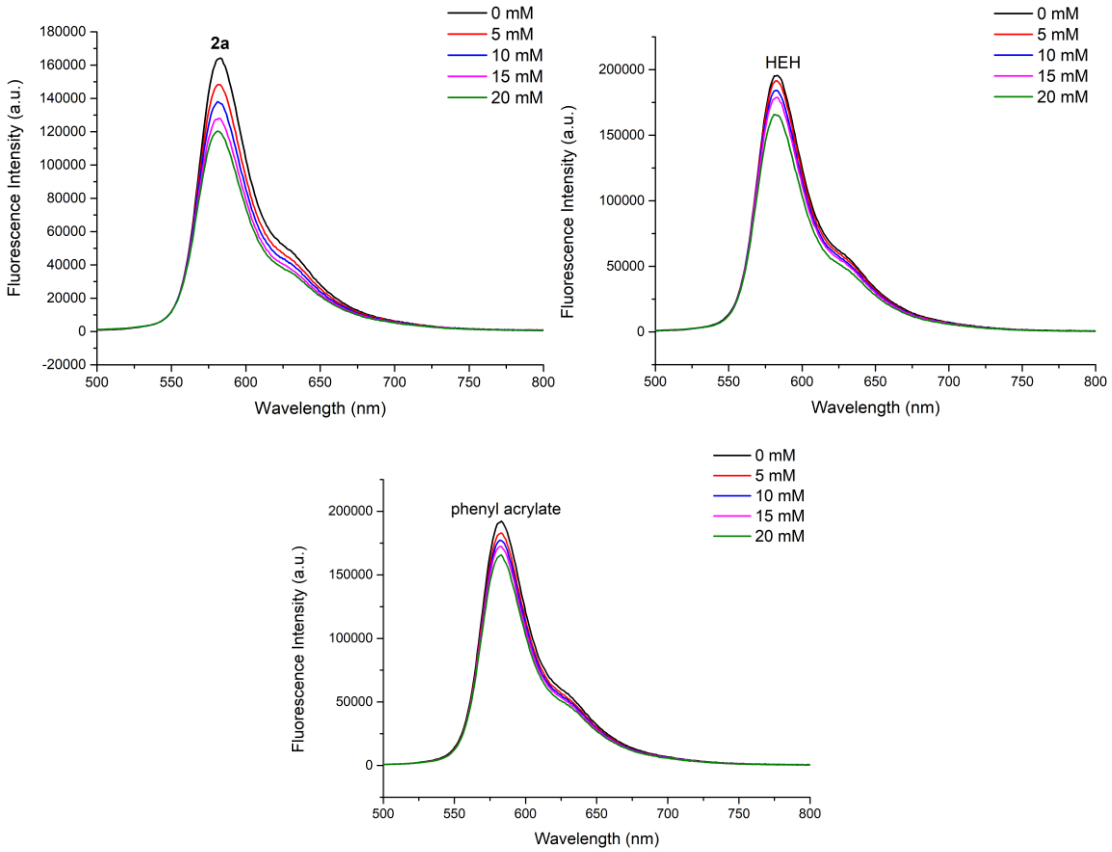


Figure S4. Fluorescence emission spectra of Rhodamine B (0.05 mM in MeCN) with different concentration of **2a**, HEH and phenyl acrylate **1au**.

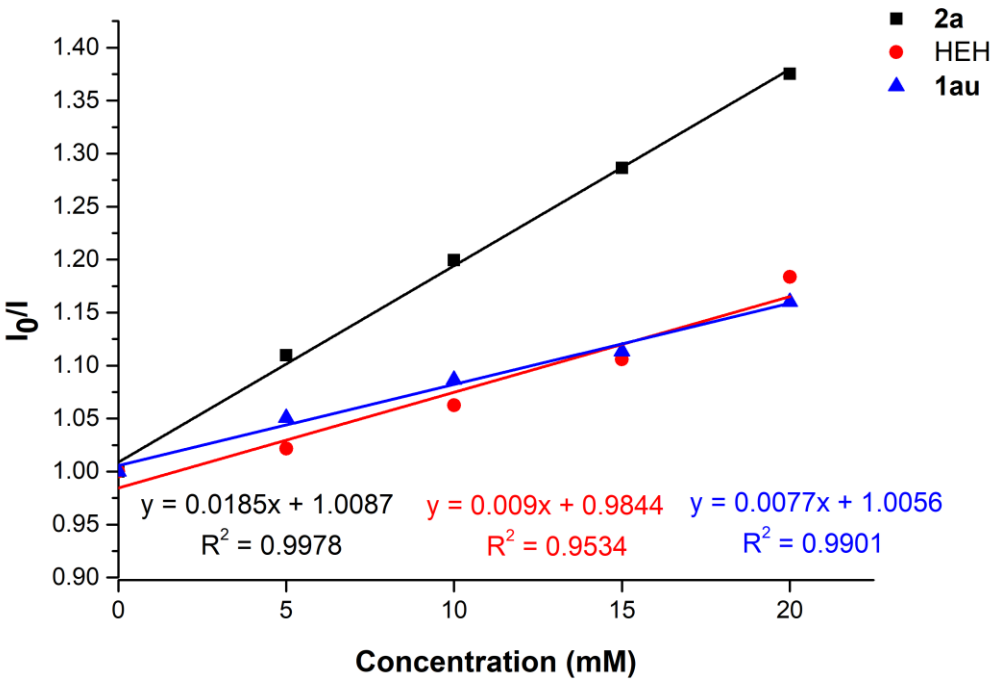
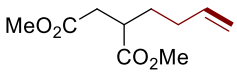


Figure S5. Stern-Volmer plot for the fluorescence quenching of 4CzIPN (0.05 mM in EtOAc) by **2a**, HE, and phenyl acrylate.

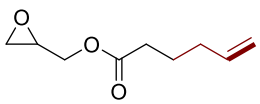
8 Characterization data of the products

Dimethyl 2-(but-3-en-1-yl)succinate (3a)



White solid; mp 45–47 °C; (59 mg, 74%); R_f = 0.27 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.83 – 5.70 (m, 1H), 5.08 – 4.96 (m, 2H), 3.71 (s, 3H), 3.68 (s, 3H), 2.93 – 2.83 (m, 1H), 2.78 – 2.69 (m, 1H), 2.49 – 2.42 (m, 1H), 2.11 – 2.03 (m, 2H), 1.82 – 1.71 (m, 1H), 1.65 – 1.56 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 175.2, 172.3, 137.2, 115.5, 51.8, 51.8, 40.5, 35.7, 31.0, 31.0; In line with previously reported spectra.[5]

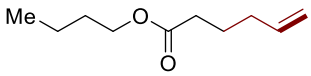
oxiran-2-ylmethyl hex-5-enoate (3b)



Colorless liquid; (52 mg, 64%); R_f = 0.20 (20 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.84 – 5.72 (m, 1H), 5.08 – 4.96 (m, 2H), 4.45 – 4.39 (m, 1H), 3.96 – 3.87 (m, 1H), 3.23 – 3.18 (m, 1H), 2.89 – 2.82 (m, 1H), 2.69 – 2.61 (m, 1H), 2.37 (t,

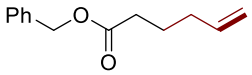
J = 7.5 Hz, 2H), 2.16 – 2.05 (m, 2H), 1.81 – 1.69 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.2, 137.5, 115.4, 64.8, 49.3, 44.6, 33.2, 33.0, 23.9. ESI-HRMS: Calcd for $C_9H_{15}O_3^+$ $[M+H]^+$ 171.1016, found 171.1016.

butyl hex-5-enoate (3c)



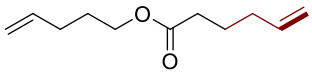
Colorless liquid; (41 mg, 60%); R_f = 0.28 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.85 – 5.71 (m, 1H), 5.08 – 4.94 (m, 2H), 4.07 (t, *J* = 6.7 Hz, 2H), 2.31 (t, *J* = 7.5 Hz, 2H), 2.09 (q, *J* = 7.1 Hz, 2H), 1.77 – 1.69 (m, 2H), 1.65 – 1.57 (m, 2H), 1.43 – 1.33 (m, 2H), 0.93 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 173.7, 137.7, 115.3, 64.1, 33.6, 33.1, 30.7, 24.1, 19.1, 13.7; ESI-HRMS: Calcd for $C_{10}H_{18}O_2^+$ $[M+H]^+$ 171.1380, found 171.1375.

benzyl hex-5-enoate (3d)



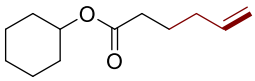
Colorless liquid; (52 mg, 64%); R_f = 0.26 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.33 – 7.22 (m, 5H), 5.76 – 5.62 (m, 1H), 5.04 (s, 2H), 4.98 – 4.87 (m, 2H), 2.30 (t, *J* = 7.5 Hz, 2H), 2.02 (q, *J* = 7.1 Hz, 2H), 1.73 – 1.63 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 173.4, 137.6, 136.1, 128.5, 128.2, 115.4, 66.1, 33.6, 33.0, 24.0; In line with previously reported spectra.[6]

pent-4-en-1-yl hex-5-enoate (3e)



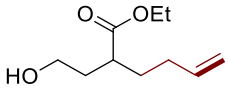
Colorless liquid; (42 mg, 57%); R_f = 0.29 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.86 – 5.72 (m, 2H), 5.07 – 4.96 (m, 4H), 4.08 (t, *J* = 6.6 Hz, 2H), 2.32 (t, *J* = 7.5 Hz, 2H), 2.17 – 2.06 (m, 4H), 1.78 – 1.67 (m, 4H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 173.6, 137.7, 137.5, 115.3, 115.2, 63.7, 33.6, 33.1, 30.0, 27.8, 24.1; ESI-HRMS: Calcd for $C_{11}H_{18}O_2^+$ $[M+H]^+$ 183.1380, found 183.1375.

cyclohexyl hex-5-enoate (3f)



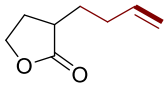
Colorless liquid; (51 mg, 65%); R_f = 0.27 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.86 – 5.72 (m, 1H), 5.11 – 4.93 (m, 2H), 4.83 – 4.68 (m, 1H), 2.29 (t, *J* = 7.5 Hz, 2H), 2.09 (q, *J* = 7.0 Hz, 2H), 1.88 – 1.80 (m, 2H), 1.76 – 1.68 (m, 4H), 1.58 – 1.50 (m, 1H), 1.45 – 1.33 (m, 4H), 1.30 – 1.22 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 173.0, 137.8, 115.2, 72.3, 34.0, 33.1, 31.6, 25.4, 24.2, 23.7; ESI-HRMS: Calcd for $C_{12}H_{20}O_2^+$ $[M+H]^+$ 197.1536, found 197.1529.

ethyl 2-(2-hydroxyethyl)hex-5-enoate (3g)



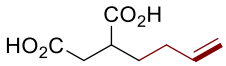
Colorless liquid; (49 mg, 66%); R_f = 0.26 (20 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.86 – 5.71 (m, 1H), 5.05 – 4.95 (m, 2H), 4.15 (q, *J* = 7.1 Hz, 2H), 3.65 (t, *J* = 6.3 Hz, 2H), 2.59 – 2.51 (m, 1H), 2.09 – 2.03 (m, 2H), 1.93 – 1.82 (m, 1H), 1.81 – 1.70 (m, 2H), 1.61 – 1.52 (m, 1H), 1.27 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 176.2, 137.6, 115.2, 60.6, 60.4, 41.8, 34.9, 31.5, 31.4, 14.2; ESI-HRMS: Calcd for $C_{10}H_{18}O_3^+$ $[M+H]^+$ 187.1329, found 187.1322.

3-(but-3-en-1-yl)dihydrofuran-2(3H)-one (3h)



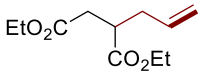
Colorless liquid; (38 mg, 67%); R_f = 0.27 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.84 – 5.71 (m, 1H), 5.11 – 4.99 (m, 2H), 4.39 – 4.31 (m, 1H), 4.24 – 4.15 (m, 1H), 2.60 – 2.50 (m, 1H), 2.46 – 2.35 (m, 1H), 2.28 – 2.09 (m, 2H), 2.06 – 1.88 (m, 2H), 1.61 – 1.49 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 179.4, 137.2, 115.7, 66.4, 38.5, 31.3, 29.5, 28.7; In line with previously reported spectra.[7]

(R)-2-allylsuccinic acid (3i)



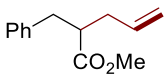
White solid; mp 185–187 °C; (47 mg, 75%); R_f = 0.21 (3 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.24 (br, 2H), 5.79 – 5.66 (m, 1H), 5.12 – 4.97 (m, 2H), 2.74 – 2.66 (m, 1H), 2.52 – 2.43 (m, 1H), 2.36 – 2.27 (m, 2H), 2.26 – 2.17 (m, 1H); ^{13}C NMR (101 MHz, DMSO-d_6): δ 175.7, 173.5, 135.7, 117.7, 40.7, 35.9, 35.3; In line with previously reported spectra.[8]

diethyl (R)-2-allylsuccinate (3j)



Colorless liquid; (69 mg, 80%); R_f = 0.28 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.79 – 5.66 (m, 1H), 5.14 – 5.03 (m, 2H), 4.22 – 4.07 (m, 4H), 2.97 – 2.87 (m, 1H), 2.74 – 2.64 (m, 1H), 2.49 – 2.39 (m, 2H), 2.34 – 2.24 (m, 1H), 1.30 – 1.20 (m, 6H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 174.1, 171.8, 134.4, 117.7, 60.6, 60.5, 40.8, 35.9, 35.2, 14.1, 14.1; In line with previously reported spectra.[9]

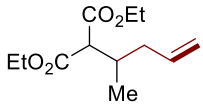
methyl (S)-2-benzylpent-4-enoate (3k)



White solid; mp 40–42 °C; (34 mg, 41%); R_f = 0.26 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.29 – 7.25 (m, 2H), 7.23 – 7.19 (m, 1H), 7.18 – 7.11 (m, 2H), 5.82 – 5.68 (m, 1H), 5.10 – 5.01 (m, 2H), 3.60 (s, 3H), 3.00 – 2.89 (m, 1H), 2.81

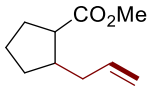
– 2.70 (m, 2H), 2.42 – 2.34 (m, 1H), 2.32 – 2.23 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 175.3, 139.1, 135.1, 128.9, 128.4, 126.4, 117.1, 51.4, 47.3, 37.8, 36.0; In line with previously reported spectra.[10]

diethyl (R)-2-(pent-4-en-2-yl)malonate (3l)



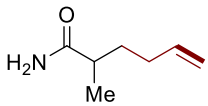
Colorless liquid; (52 mg, 57%); R_f = 0.25 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.81 – 5.69 (m, 1H), 5.10 – 5.00 (m, 2H), 4.20 (q, *J* = 7.1 Hz, 4H), 3.26 (d, *J* = 8.0 Hz, 1H), 2.40 – 2.30 (m, 1H), 2.26 – 2.17 (m, 1H), 2.06 – 1.95 (m, 1H), 1.29 – 1.25 (m, 6H), 1.00 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 168.8, 168.6, 135.8, 117.1, 61.1, 61.1, 56.8, 38.7, 33.1, 16.8, 14.1, 14.0; In line with previously reported spectra.[11]

methyl (2R)-2-allylcyclopentane-1-carboxylate (3m)



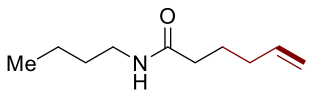
Colorless liquid; (39 mg, 58%); R_f = 0.31 (50 : 1 petroleum ether / ethyl acetate); dr = 1:1; ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.83 – 5.70 (m, 1H), 5.05 – 4.94 (m, 2H), 3.67 (s, 1.5H), 3.65 (s, 1.5H), 2.90 – 2.81 (m, 0.5H), 2.41 – 2.34 (m, 0.5H), 2.26 – 2.18 (m, 1H), 2.16 – 2.04 (m, 1H), 1.97 – 1.77 (m, 4H), 1.70 – 1.64 (m, 1H), 1.59 – 1.45 (m, 1H), 1.32 – 1.20 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 177.0, 175.7, 137.6, 137.0, 115.8, 115.4, 51.6, 51.1, 49.6, 47.3, 43.6, 43.0, 39.2, 35.5, 32.0, 30.9, 30.3, 28.3, 24.6, 23.7; ESI-HRMS: Calcd for $C_{10}H_{16}O_2^+$ $[M+H]^+$ 169.1223, found 169.1218.

2-methylhex-5-enamide (3n)



Colorless liquid; (25 mg, 50%); R_f = 0.26 (1 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.85 – 5.73 (m, 1H), 5.51 (br, 2H), 5.08 – 4.95 (m, 2H), 2.37 – 2.23 (m, 1H), 2.11 (p, *J* = 6.7 Hz, 2H), 1.83 – 1.73 (m, 1H), 1.50 (m, 1H), 1.17 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 178.8, 138.0, 115.1, 39.9, 33.1, 31.4, 17.8; ESI-HRMS: Calcd for $C_7H_{13}NO^+$ $[M+H]^+$ 128.1070, found 128.1065.

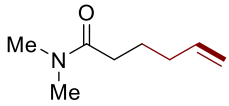
N-butylhex-5-enamide (3o)



Colorless liquid; (41 mg, 60%); R_f = 0.29 (3 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.84 – 5.72 (m, 1H), 5.56 (br, 1H), 5.06 – 4.95 (m, 2H), 3.25 (q, *J* = 7.0 Hz, 2H), 2.17 (t, *J* = 7.6 Hz, 2H), 2.13 – 2.05 (m, 2H), 1.79 – 1.70 (m, 2H), 1.52 – 1.44 (m, 2H), 1.38 – 1.31 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz,

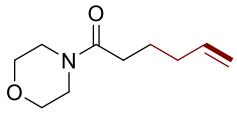
Chloroform-*d*): δ 172.7, 137.9, 115.2, 39.2, 36.0, 33.1, 31.7, 24.8, 20.0, 13.7; In line with previously reported spectra.[12]

N,N-dimethylhex-5-enamide (3p)



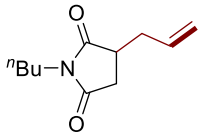
Colorless liquid; (38 mg, 67%); R_f = 0.31 (5 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.86 – 5.74 (m, 1H), 5.08 – 4.94 (m, 2H), 3.00 (s, 3H), 2.95 (s, 3H), 2.32 (t, 2H), 2.12 (q, *J* = 7.1 Hz, 2H), 1.81 – 1.69 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 173.0, 138.1, 115.0, 37.2, 35.3, 33.3, 32.4, 24.2; In line with previously reported spectra.[13]

1-morpholinohex-5-en-1-one (3q)



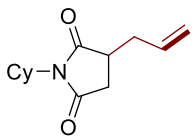
Colorless liquid; (46 mg, 63%); R_f = 0.33 (5 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.91 – 5.73 (m, 1H), 5.07 – 4.97 (m, 2H), 3.69 – 3.65 (m, 4H), 3.64 – 3.59 (m, 2H), 3.48 – 3.43 (m, 2H), 2.35 – 2.29 (m, 2H), 2.12 (q, *J* = 7.1 Hz, 2H), 1.80 – 1.71 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 171.5, 137.9, 115.2, 66.9, 66.6, 45.9, 41.8, 33.2, 32.1, 24.2; In line with previously reported spectra.[14]

(S)-3-allyl-1-butylpyrrolidine-2,5-dione (3r)



White solid; mp 83–85 °C; (62 mg, 79%); R_f = 0.32 (20 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.76 – 5.63 (m, 1H), 5.19 – 5.09 (m, 2H), 3.49 (t, *J* = 7.4 Hz, 2H), 2.95 – 2.86 (m, 1H), 2.82 – 2.72 (m, 1H), 2.65 – 2.56 (m, 1H), 2.47 – 2.40 (m, 1H), 2.39 – 2.31 (m, 1H), 1.59 – 1.48 (m, 2H), 1.35 – 1.25 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 179.3, 176.5, 133.1, 118.8, 39.1, 38.6, 35.1, 33.3, 29.7, 20.0, 13.6; ESI-HRMS: Calcd for $C_{11}H_{17}NO_2^+$ $[M+H]^+$ 196.1332, found 196.1326.

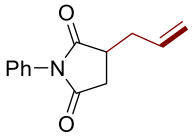
(S)-3-allyl-1-cyclohexylpyrrolidine-2,5-dione (3s)



White solid; mp 79–82 °C; (68 mg, 77%); R_f = 0.33 (20 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.75 – 5.62 (m, 1H), 5.17 – 5.09 (m, 2H), 4.01 – 3.91 (m, 1H), 2.87 – 2.79 (m, 1H), 2.76 – 2.68 (m, 1H), 2.61 – 2.53 (m, 1H), 2.43 – 2.30 (m, 2H), 2.20 – 2.07 (m, 2H), 1.86 – 1.78 (m, 2H), 1.69 – 1.62 (m, 1H), 1.59 – 1.52 (m, 2H), 1.36 – 1.19 (m, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 179.4, 176.6, 133.0, 118.8, 51.7, 38.7,

35.2, 33.3, 28.8, 28.8, 25.8, 25.0; ESI-HRMS: Calcd for $C_{13}H_{19}NO_2^+$ $[M+H]^+$ 222.1489, found 222.1480.

(S)-3-allyl-1-phenylpyrrolidine-2,5-dione (3t)

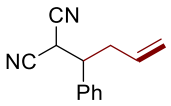


White solid; mp 84–86 °C; (71 mg, 83%); R_f = 0.27 (20 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.49 – 7.43 (m, 2H), 7.40 – 7.34 (m, 1H), 7.30 – 7.23 (m, 2H), 5.83 – 5.70 (m, 1H), 5.23 – 5.13 (m, 2H), 3.10 – 3.01 (m, 1H), 2.97 – 2.87 (m, 1H), 2.70 – 2.63 (m, 1H), 2.62 – 2.55 (m, 1H), 2.52 – 2.42 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 178.2, 175.3, 132.8, 131.8, 129.0, 128.4, 126.3, 119.0, 39.2, 35.1, 33.4; ESI-HRMS: Calcd for $C_{13}H_{13}NO_2^+$ $[M+H]^+$ 216.1019, found 216.1011.

hex-5-enenitrile (3u)

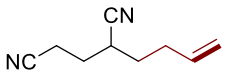
Colorless liquid; (24 mg, 63%); R_f = 0.31 (100 : 1 petroleum ether / ethyl acetate); Because hex-5-enenitrile is volatile, the yield was measured by crude ^{1}H NMR spectroscopy using 1,3,5-trimethoxybenzene as an internal standard. ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.83 – 5.63 (m, 1H), 5.16 – 4.99 (m, 2H), 2.33 (t, *J* = 7.2 Hz, 2H), 2.25 – 2.15 (m, 2H), 1.79 – 1.67 (m, 2H); In line with previously reported spectra.[15]

(R)-2-(1-phenylbut-3-en-1-yl)malononitrile (3v)



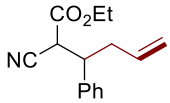
Colorless liquid; (33 mg, 42%); R_f = 0.32 (20 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.49 – 7.30 (m, 5H), 5.76 – 5.58 (m, 1H), 5.32 – 5.13 (m, 2H), 4.02 (d, *J* = 5.6 Hz, 1H), 3.34 – 3.22 (m, 1H), 2.80 – 2.68 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 136.3, 133.0, 129.2, 129.0, 127.9, 119.9, 111.9, 111.5, 45.9, 36.4, 28.9; In line with previously reported spectra.[16]

2-(but-3-en-1-yl)pentanedinitrile (3w)



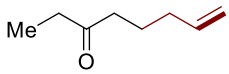
Colorless liquid; (38 mg, 64%); R_f = 0.31 (30 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.82 – 5.70 (m, 1H), 5.17 – 5.07 (m, 2H), 2.83 – 2.72 (m, 1H), 2.67 – 2.51 (m, 2H), 2.39 – 2.29 (m, 1H), 2.28 – 2.18 (m, 1H), 2.02 – 1.94 (m, 2H), 1.85 – 1.76 (m, 1H), 1.76 – 1.66 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 135.6, 120.0, 117.9, 117.0, 30.9, 30.9, 30.1, 28.1, 15.3; ESI-HRMS: Calcd for $C_9H_{12}N_2^+$ $[M+H]^+$ 149.1073, found 149.1068.

ethyl (3R)-2-cyano-3-phenylhex-5-enoate (3x)



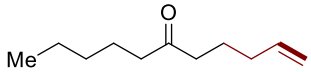
White solid; mp 56–58 °C; (73 mg, 75%); R_f = 0.27 (20 : 1 petroleum ether / ethyl acetate); dr = 1.3:1; ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.37 – 7.26 (m, 5H), 5.77 – 5.53 (m, 1H), 5.31 – 4.96 (m, 2H), 4.18 – 4.03 (m, 2H), 3.88 (d, *J* = 5.5 Hz, 1H), 3.70 (d, *J* = 6.4 Hz, 0H), 3.51 – 3.37 (m, 1H), 2.76 – 2.62 (m, 2H), 1.17 (t, *J* = 7.1 Hz, 1H), 1.09 (t, *J* = 7.1 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 165.4, 165.1, 138.8, 138.2, 134.3, 134.1, 128.8, 128.6, 128.0, 128.0, 127.9, 127.8, 118.9, 118.2, 115.6, 115.2, 62.7, 62.6, 45.5, 45.0, 44.2, 43.3, 37.6, 36.5, 13.8; ESI-HRMS: Calcd for $C_{15}H_{17}NO_2^+$ $[M+H]^+$ 244.1332, found 244.1325.

oct-7-en-3-one (3y)



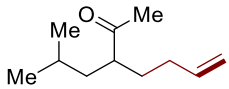
Colorless liquid; (31 mg, 62%); R_f = 0.32 (60 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.76 – 5.63 (m, 1H), 5.00 – 4.87 (m, 2H), 2.40 – 2.28 (m, 4H), 1.99 (q, *J* = 7.0 Hz, 2H), 1.67 – 1.56 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 211.6, 138.0, 115.2, 41.5, 36.0, 33.1, 22.9, 7.8; In line with previously reported spectra.[17]

undec-1-en-6-one (3z)



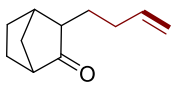
Colorless liquid; (45 mg, 67%); R_f = 0.26 (60 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.77 – 5.63 (m, 1H), 4.99 – 4.85 (m, 2H), 2.32 (q, *J* = 7.8 Hz, 4H), 1.98 (q, *J* = 7.1 Hz, 2H), 1.64 – 1.56 (m, 2H), 1.54 – 1.46 (m, 2H), 1.26 – 1.16 (m, 4H), 0.82 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 211.2, 138.0, 115.1, 42.8, 41.8, 33.1, 31.4, 23.5, 22.8, 22.4, 13.9; ESI-HRMS: Calcd for $C_{11}H_{20}O^+$ $[M+H]^+$ 169.1587, found 169.1581.

3-isobutylhept-6-en-2-one (3aa)



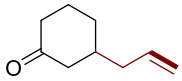
Colorless liquid; (46 mg, 68%); R_f = 0.27 (60 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.82 – 5.69 (m, 1H), 5.05 – 4.94 (m, 2H), 2.62 – 2.49 (m, 1H), 2.13 (s, 3H), 2.01 (q, *J* = 7.0 Hz, 2H), 1.72 – 1.63 (m, 1H), 1.56 – 1.43 (m, 3H), 1.26 – 1.18 (m, 1H), 0.91 – 0.87 (m, 6H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 212.8, 137.9, 115.1, 50.4, 40.9, 31.5, 31.0, 28.7, 26.1, 22.9, 22.4; ESI-HRMS: Calcd for $C_{11}H_{20}O^+$ $[M+H]^+$ 169.1587, found 169.1582.

3-(but-3-en-1-yl)bicyclo[2.2.1]heptan-2-one (3ab)



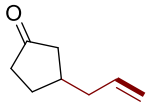
Colorless liquid; (43 mg, 66%); R_f = 0.27 (60 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.85 – 5.73 (m, 1H), 5.10 – 4.92 (m, 2H), 2.65 – 2.57 (m, 2H), 2.18 – 2.00 (m, 3H), 1.86 – 1.76 (m, 2H), 1.71 – 1.65 (m, 1H), 1.62 – 1.56 (m, 3H), 1.46 – 1.36 (m, 1H), 1.33 – 1.26 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 220.0, 137.9, 115.1, 52.9, 50.5, 38.1, 37.0, 32.0, 25.5, 25.3, 21.1; In line with previously reported spectra.[18]

(R)-3-allylcyclohexan-1-one (3ac)



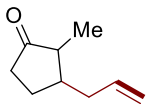
Colorless liquid; (22 mg, 40%); R_f = 0.26 (60 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.81 – 5.68 (m, 1H), 5.10 – 4.99 (m, 2H), 2.45 – 2.33 (m, 2H), 2.30 – 2.21 (m, 1H), 2.13 – 2.00 (m, 4H), 1.93 – 1.81 (m, 2H), 1.70 – 1.61 (m, 1H), 1.40 – 1.32 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 211.8, 135.7, 116.8, 47.8, 41.4, 40.8, 38.8, 30.9, 25.2; In line with previously reported spectra.[19]

(R)-3-allylcyclopentan-1-one (3ad)



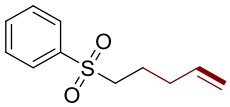
Colorless liquid; (24 mg, 48%); R_f = 0.27 (60 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.86 – 5.73 (m, 1H), 5.12 – 4.99 (m, 2H), 2.42 – 2.24 (m, 3H), 2.22 – 2.11 (m, 4H), 1.91 – 1.81 (m, 1H), 1.64 – 1.52 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 219.6, 136.2, 116.4, 44.7, 39.5, 38.3, 36.6, 28.9; In line with previously reported spectra.[20]

(3R)-3-allyl-2-methylcyclopentan-1-one (3ae)



Colorless liquid; (23 mg, 41%); R_f = 0.29 (60 : 1 petroleum ether / ethyl acetate); dr = 3.8 : 1; ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.90 – 5.74 (m, 1H), 5.14 – 5.00 (m, 2H), 2.49 – 2.31 (m, 2H), 2.25 – 2.05 (m, 3H), 1.80 – 1.69 (m, 2H), 1.50 – 1.39 (m, 1H), 1.09 (d, *J* = 6.5 Hz, 2.37 H), 1.01 (d, *J* = 6.9 Hz, 0.63 H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 221.0, 136.7, 135.8, 116.6, 116.2, 49.7, 47.0, 44.4, 39.5, 38.3, 37.3, 35.8, 33.7, 26.8, 25.0, 12.6, 9.7; In line with previously reported spectra.[20]

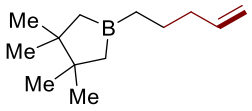
(pent-4-en-1-ylsulfonyl)benzene (3af)



White solid; mp 70–72 °C; (64 mg, 77%); R_f = 0.27 (20 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.95 – 7.88 (m, 2H), 7.69 – 7.64 (m, 1H), 7.61 – 7.54 (m, 2H), 5.75 – 5.61 (m, 1H), 5.05 – 4.94 (m, 2H), 3.14 – 3.04 (m, 2H), 2.13 (q, *J* = 7.1 Hz,

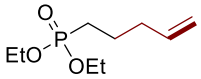
2H), 1.87 – 1.76 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 139.0, 136.2, 133.6, 129.2, 127.9, 116.4, 55.4, 31.9, 21.7; In line with previously reported spectra.[21]

4,4,5,5-tetramethyl-2-(pent-4-en-1-yl)-1,3,2-dioxaborolane (3ag)



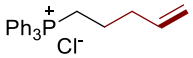
Colorless liquid; (38 mg, 49%); R_f = 0.32 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.86 – 5.74 (m, 1H), 5.05 – 4.87 (m, 2H), 2.05 (q, *J* = 7.0 Hz, 2H), 1.55 – 1.46 (m, 2H), 1.24 (s, 12H), 0.79 (t, *J* = 7.8 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 138.9, 114.4, 82.9, 36.4, 24.8, 23.4; In line with previously reported spectra.[22]

diethyl pent-4-en-1-ylphosphonate (3ah)



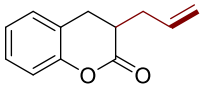
Colorless liquid; (54 mg, 66%); R_f = 0.30 (3 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.85 – 5.69 (m, 1H), 5.08 – 4.96 (m, 2H), 4.15 – 4.04 (m, 4H), 2.14 (q, *J* = 6.5 Hz, 2H), 1.78 – 1.67 (m, 4H), 1.32 (t, *J* = 7.1 Hz, 6H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 137.3, 115.6, 61.3 (d, *J* = 6.5 Hz), 34.3 (d, *J* = 17.3 Hz), 24.9 (d, *J* = 141.1 Hz), 21.6 (d, *J* = 4.9 Hz), 16.4 (d, *J* = 5.9 Hz); In line with previously reported spectra.[23]

pent-4-en-1-yltriphenylphosphonium chloride (3ai)



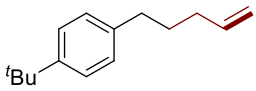
White solid; mp 153–155 °C; (84 mg, 57%); R_f = 0.25 (10 : 1 dichloromethane / methanol); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.86 – 7.79 (m, 9H), 7.76 – 7.71 (m, 6H), 5.79 – 5.65 (m, 1H), 5.10 – 4.97 (m, 2H), 3.81 – 3.69 (m, 2H), 2.46 – 2.36 (m, 2H), 1.82 – 1.72 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 135.95 , 134.76 (d, *J* = 2.9 Hz), 133.21 (d, *J* = 10.0 Hz), 130.21 (d, *J* = 12.5 Hz), 117.81 (d, *J* = 85.9 Hz), 116.63 , 33.44 (d, *J* = 16.5 Hz), 21.56 (d, *J* = 9.0 Hz), 21.29 (d, *J* = 38.0 Hz); In line with previously reported spectra.[24]

(R)-3-allylchroman-2-one (3aj)



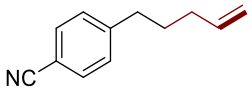
White solid; mp 70–72 °C; (28 mg, 37%); R_f = 0.29 (30 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.28 – 7.23 (m, 1H), 7.19 – 7.15 (m, 1H), 7.12 – 7.07 (m, 1H), 7.06 – 7.02 (m, 1H), 5.90 – 5.78 (m, 1H), 5.18 – 5.10 (m, 2H), 3.04 – 2.96 (m, 1H), 2.87 – 2.67 (m, 3H), 2.40 – 2.30 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 170.4, 151.6, 134.3, 128.2, 128.1, 124.3, 122.5, 118.2, 116.6, 38.7, 33.9, 28.6; In line with previously reported spectra.[25]

1-(tert-butyl)-4-(pent-4-en-1-yl)benzene (3ak)



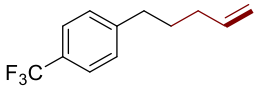
Colorless liquid; (31mg, 38%); R_f = 0.52 (petroleum ether); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.30 (d, *J* = 8.3 Hz, 2H), 7.12 (d, *J* = 8.3 Hz, 2H), 5.90 – 5.78 (m, 1H), 5.06 – 4.94 (m, 2H), 2.59 (t, *J* = 7.6 Hz, 2H), 2.10 (q, *J* = 7.0 Hz, 2H), 1.77 – 1.66 (m, 2H), 1.31 (s, 9H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 148.4, 139.4, 138.7, 128.1, 125.1, 114.6, 34.8, 34.3, 33.4, 31.4, 30.6; In line with previously reported spectra.[25]

4-(pent-4-en-1-yl)benzonitrile (3al)



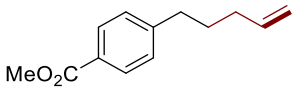
Colorless liquid; (29 mg, 43%); R_f = 0.35 (petroleum ether); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.57 (d, *J* = 8.2 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 5.88 – 5.73 (m, 1H), 5.07 – 4.96 (m, 2H), 2.68 (t, *J* = 7.7 Hz, 2H), 2.09 (q, *J* = 7.1 Hz, 2H), 1.73 (p, *J* = 7.5 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 148.1, 137.9, 132.1, 129.2, 119.1, 115.2, 109.6, 35.4, 33.1, 30.0; In line with previously reported spectra.[26]

1-(pent-4-en-1-yl)-4-(trifluoromethyl)benzene (3am)



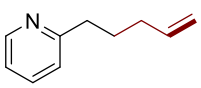
Colorless liquid; (34 mg, 40%); R_f = 0.43 (petroleum ether); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.53 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 5.88 – 5.76 (m, 1H), 5.07 – 4.96 (m, 2H), 2.68 (t, *J* = 7.6 Hz, 2H), 2.10 (q, *J* = 7.0 Hz, 2H), 1.78 – 1.68 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 146.5 (q, *J* = 1.1 Hz), 138.2, 128.7, 128.6 (q, *J* = 32.3 Hz), 125.2 (q, *J* = 3.7 Hz), 124.4 (q, *J* = 272.8 Hz), 115.1, 35.1, 33.1, 30.3; ^{19}F NMR (376 MHz, Chloroform-*d*): δ -62.3. In line with previously reported spectra.[27]

methyl 4-(pent-4-en-1-yl)benzoate (3an)



Colorless liquid; (34 mg, 42%); R_f = 0.21 (petroleum ether); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.88 (d, *J* = 7.9 Hz, 2H), 7.17 (d, *J* = 8.1 Hz, 2H), 5.82 – 5.65 (m, 1H), 4.99 – 4.87 (m, 2H), 3.83 (s, 3H), 2.60 (t, 2H), 2.01 (q, *J* = 7.0 Hz, 2H), 1.72 – 1.62 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 167.1, 148.0, 138.2, 129.6, 128.5, 115.0, 51.9, 35.3, 33.2, 30.2; In line with previously reported spectra.[28]

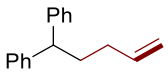
2-(pent-4-en-1-yl)pyridine (3ao)



Colorless liquid; (32 mg, 54%); R_f = 0.31 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 8.59 – 8.48 (m, 1H), 7.64 – 7.52 (m, 1H), 7.19 – 7.03 (m, 2H), 5.94 – 5.75 (m, 1H), 5.12 – 4.90 (m, 2H), 2.80 (t, *J* = 7.8 Hz, 2H), 2.12 (q, *J* = 7.2 Hz, 2H),

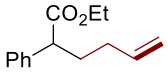
1.94 – 1.75 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 162.0, 149.1, 138.3, 136.1, 122.6, 120.8, 114.7, 37.6, 33.3, 28.9; In line with previously reported spectra.[29]

pent-4-ene-1,1-diyldibenzene (3ap)



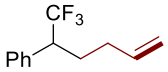
Colorless liquid; (47 mg, 53%); R_f = 0.24 (petroleum ether); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.30 – 7.22 (m, 8H), 7.19 – 7.15 (m, 2H), 5.89 – 5.75 (m, 1H), 5.02 – 4.93 (m, 2H), 3.92 (t, *J* = 7.7 Hz, 1H), 2.18 – 2.10 (m, 2H), 2.01 (q, *J* = 7.1 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 144.9, 138.3, 128.4, 127.9, 126.1, 114.9, 50.5, 34.7, 32.0; In line with previously reported spectra.[30]

ethyl 2-phenylhex-5-enoate (3aq)



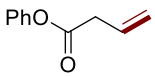
Colorless liquid; (42 mg, 48%); R_f = 0.28 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.34 – 7.29 (m, 4H), 7.27 – 7.23 (m, 1H), 5.84 – 5.72 (m, 1H), 5.04 – 4.95 (m, 2H), 4.19 – 4.03 (m, 2H), 3.56 (t, *J* = 7.6 Hz, 1H), 2.23 – 2.12 (m, 1H), 2.02 (q, *J* = 7.0 Hz, 2H), 1.91 – 1.81 (m, 1H), 1.20 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 173.9, 139.0, 137.6, 128.6, 127.9, 127.2, 115.4, 60.7, 50.9, 32.6, 31.5, 14.1; In line with previously reported spectra.[31]

(1,1,1-trifluorohex-5-en-2-yl)benzene (3ar)



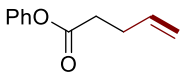
Colorless liquid; (39 mg, 45%); R_f = 0.32 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.39 – 7.31 (m, 3H), 7.29 – 7.24 (m, 2H), 5.79 – 5.67 (m, 1H), 5.04 – 4.91 (m, 2H), 3.32 – 3.21 (m, 1H), 2.13 – 1.93 (m, 3H), 1.91 – 1.79 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 137.0, 134.5 (q, *J* = 2.0 Hz), 129.1, 128.7, 128.2, 127.0 (q, *J* = 280.7 Hz), 115.9, 49.1 (q, *J* = 26.5 Hz), 30.5, 27.7 (q, *J* = 26.5 Hz); ^{19}F NMR (376 MHz, Chloroform-*d*): δ -69.7; ESI-HRMS: Calcd for $C_{12}H_{13}F_3^+$ $[M+H]^+$ 215.1042, found 215.1033.

phenyl but-3-enoate (3as)



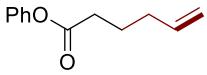
Colorless liquid; (21 mg, 33%); R_f = 0.28 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.37 – 7.31 (m, 2H), 7.22 – 7.16 (m, 1H), 7.10 – 7.04 (m, 2H), 6.07 – 5.95 (m, 1H), 5.29 – 5.20 (m, 2H), 3.32 – 3.28 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 169.7, 150.5, 129.5, 129.3, 125.7, 121.3, 119.0, 38.9; In line with previously reported spectra.[32]

phenyl pent-4-enoate (3at)



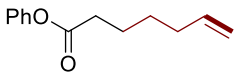
Colorless liquid; (16 mg, 23%); R_f = 0.30 (40 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.40 – 7.33 (m, 2H), 7.24 – 7.17 (m, 1H), 7.13 – 7.03 (m, 2H), 5.97 – 5.82 (m, 1H), 5.18 – 5.02 (m, 2H), 2.66 (t, *J* = 7.5 Hz, 2H), 2.50 (q, *J* = 7.2 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 171.4, 150.7, 136.3, 129.3, 125.7, 121.5, 115.9, 33.6, 28.8. In line with previously reported spectra.[33]

Phenyl hex-5-enoate (3au)



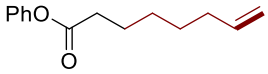
Colorless liquid; (54 mg, 71%); R_f = 0.26 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.41 – 7.34 (m, 2H), 7.25 – 7.19 (m, 1H), 7.10 – 7.04 (m, 2H), 5.88 – 5.77 (m, 1H), 5.11 – 5.01 (m, 2H), 2.57 (t, *J* = 7.5 Hz, 2H), 2.19 (q, *J* = 7.1 Hz, 2H), 1.91 – 1.82 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 172.1, 150.7, 137.5, 129.4, 125.7, 121.5, 115.6, 33.6, 33.0, 24.0; In line with previously reported spectra.[34]

phenyl hept-6-enoate (3av)



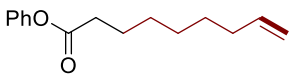
Colorless liquid; (52 mg, 64%); R_f = 0.28 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.33 – 7.26 (m, 2H), 7.17 – 7.11 (m, 1H), 7.02 – 6.97 (m, 2H), 5.82 – 5.66 (m, 1H), 5.01 – 4.85 (m, 2H), 2.48 (t, *J* = 7.5 Hz, 2H), 2.08 – 2.01 (m, 2H), 1.75 – 1.65 (m, 2H), 1.49 – 1.40 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 172.1, 150.7, 138.2, 129.3, 125.7, 121.5, 114.8, 34.2, 33.3, 28.3, 24.3. ESI-HRMS: Calcd for $C_{13}H_{17}O_2^+$ $[M+H]^+$ 205.1223, found 205.1224.

phenyl oct-7-enoate (3aw)



Colorless liquid; (58 mg, 66%); R_f = 0.29 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.40 – 7.34 (m, 2H), 7.24 – 7.18 (m, 1H), 7.10 – 7.04 (m, 2H), 5.87 – 5.75 (m, 1H), 5.05 – 4.93 (m, 2H), 2.55 (t, *J* = 7.5 Hz, 2H), 2.08 (q, *J* = 6.8 Hz, 2H), 1.83 – 1.69 (m, 2H), 1.49 – 1.40 (m, 4H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 172.1, 150.7, 138.7, 129.3, 125.6, 121.5, 114.5, 34.3, 33.5, 28.5, 28.5, 24.7; In line with previously reported spectra.[35]

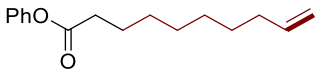
phenyl non-8-enoate (3ax)



Colorless liquid; (56 mg, 60%); R_f = 0.30 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.41 – 7.34 (m, 2H), 7.25 – 7.19 (m, 1H), 7.10 – 7.04 (m, 2H), 5.87 – 5.75 (m, 1H), 5.04 – 4.91 (m, 2H), 2.55 (t, *J* = 7.5 Hz, 2H), 2.06 (q, *J* = 6.8 Hz, 2H),

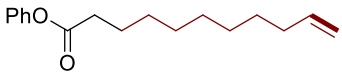
1.80 – 1.71 (m, 2H), 1.47 – 1.36 (m, 6H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 172.3, 150.7, 138.9, 129.4, 125.7, 121.6, 114.3, 34.4, 33.7, 28.9, 28.7, 28.7, 24.9; ESI-HRMS: Calcd for $C_{15}H_{20}O_2^+$ $[M+H]^+$ 233.1536, found 233.1531.

phenyl dec-9-enoate (3ay)



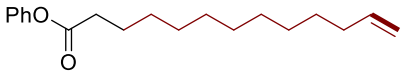
Colorless liquid; (57 mg, 58%); R_f = 0.27 (40 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.34 – 7.26 (m, 2H), 7.18 – 7.11 (m, 1H), 7.02 – 6.96 (m, 2H), 5.81 – 5.66 (m, 1H), 4.98 – 4.81 (m, 2H), 2.47 (t, *J* = 7.5 Hz, 2H), 1.98 (q, *J* = 6.9 Hz, 2H), 1.72 – 1.62 (m, 2H), 1.37 – 1.25 (m, 8H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 172.2, 150.7, 139.0, 129.3, 125.7, 121.5, 114.2, 34.4, 33.7, 29.1, 29.0, 28.9, 28.8, 24.9. ESI-HRMS: Calcd for $C_{16}H_{23}O_2^+$ $[M+H]^+$ 247.1693, found 247.1693.

phenyl undec-10-enoate (3az)



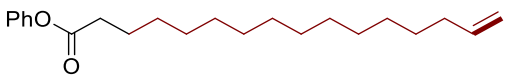
Colorless liquid; (61 mg, 59%); R_f = 0.29 (40 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.40 – 7.34 (m, 2H), 7.24 – 7.18 (m, 1H), 7.11 – 7.02 (m, 2H), 5.89 – 5.71 (m, 1H), 5.04 – 4.86 (m, 2H), 2.55 (t, *J* = 7.5 Hz, 2H), 2.04 (q, *J* = 6.8 Hz, 2H), 1.79 – 1.71 (m, 2H), 1.44 – 1.29 (m, 10H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 172.3, 150.7, 139.1, 129.3, 125.7, 121.5, 114.1, 34.4, 33.8, 29.3, 29.2, 29.1, 29.0, 28.9, 24.9; In line with previously reported spectra.[36]

phenyl tridec-12-enoate (3ba)



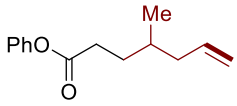
Colorless liquid; (70 mg, 61%); R_f = 0.32 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.34 – 7.27 (m, 2H), 7.17 – 7.12 (m, 1H), 7.02 – 6.98 (m, 2H), 5.82 – 5.67 (m, 1H), 4.97 – 4.82 (m, 2H), 2.48 (t, *J* = 7.5 Hz, 2H), 1.97 (q, *J* = 6.8 Hz, 2H), 1.74 – 1.63 (m, 2H), 1.35 – 1.20 (m, 14H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 172.3, 150.8, 139.2, 129.4, 125.7, 121.6, 114.1, 34.4, 33.8, 29.5, 29.5, 29.4, 29.2, 29.1, 29.1, 28.9, 25.0; ESI-HRMS: Calcd for $C_{19}H_{28}O_2^+$ $[M+H]^+$ 289.2162, found 289.2152.

phenyl hexadec-15-enoate (3bb)



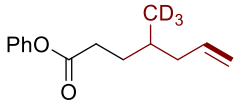
Colorless liquid; (73 mg, 55%); R_f = 0.28 (40 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.40 – 7.33 (m, 2H), 7.24 – 7.18 (m, 1H), 7.10 – 7.05 (m, 2H), 5.87 – 5.75 (m, 1H), 5.03 – 4.89 (m, 2H), 2.55 (t, *J* = 7.5 Hz, 2H), 2.08 – 1.99 (m, 2H), 1.80 – 1.69 (m, 2H), 1.44 – 1.26 (m, 20H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.3, 150.8, 139.2, 129.3, 125.6, 121.6, 114.1, 34.4, 33.8, 29.6, 29.6, 29.6, 29.6, 29.5, 29.4, 29.2, 29.1, 29.1, 28.9, 24.9. ESI-HRMS: Calcd for $C_{22}H_{35}O_2^+$ $[M+H]^+$ 331.2632, found 331.2632.

phenyl (R)-4-methylhept-6-enoate (3bc)



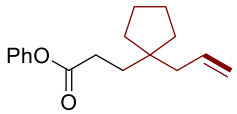
Colorless liquid; (62 mg, 71%); R_f = 0.32 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.40 – 7.32 (m, 2H), 7.24 – 7.18 (m, 1H), 7.09 – 7.04 (m, 2H), 5.86 – 5.73 (m, 1H), 5.08 – 4.99 (m, 2H), 2.64 – 2.48 (m, 2H), 2.17 – 2.07 (m, 1H), 2.02 – 1.92 (m, 1H), 1.87 – 1.77 (m, 1H), 1.68 – 1.52 (m, 2H), 0.95 (d, *J* = 6.5 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 172.3, 150.7, 136.8, 129.3, 125.6, 121.5, 116.1, 41.0, 32.3, 32.1, 31.3, 19.1; ESI-HRMS: Calcd for $C_{14}H_{18}O_2$ $[M+H]^+$ 219.1380, found 219.1372.

phenyl (R)-4-(methyl-d3)hept-6-enoate (3bd)



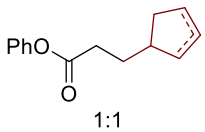
Colorless liquid; (56 mg, 63%); R_f = 0.31 (40 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.40 – 7.34 (m, 2H), 7.24 – 7.18 (m, 1H), 7.11 – 7.04 (m, 2H), 5.86 – 5.73 (m, 1H), 5.09 – 4.98 (m, 2H), 2.63 – 2.50 (m, 2H), 2.17 – 2.07 (m, 1H), 2.02 – 1.92 (m, 1H), 1.88 – 1.77 (m, 1H), 1.65 – 1.53 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.4, 150.7, 136.8, 129.4, 125.7, 121.5, 116.1, 40.9, 32.2, 32.1, 31.2, 18.2 (heptet, *J* = 19.0 Hz). ESI-HRMS: Calcd for $C_{14}H_{16}D_3O_2^+$ $[M+H]^+$ 222.1568, found 222.1571.

phenyl 3-(1-allylcyclopentyl)propanoate (3be)



Colorless liquid; (74 mg, 72%); R_f = 0.31 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.41 – 7.34 (m, 2H), 7.25 – 7.19 (m, 1H), 7.10 – 7.05 (m, 2H), 5.89 – 5.76 (m, 1H), 5.11 – 5.04 (m, 2H), 2.58 – 2.48 (m, 2H), 2.14 – 2.05 (m, 2H), 1.81 – 1.74 (m, 2H), 1.67 – 1.60 (m, 4H), 1.54 – 1.46 (m, 2H), 1.45 – 1.37 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 172.7, 150.8, 135.5, 129.4, 125.7, 121.5, 117.1, 44.6, 42.7, 37.0, 33.7, 30.3, 24.7; ESI-HRMS: Calcd for $C_{17}H_{22}O_2^+$ $[M+H]^+$ 259.1693, found 259.1683.

phenyl (R)-3-(cyclopent-2-en-1-yl)propanoate and phenyl 3-(cyclopent-3-en-1-yl)propanoate (3bf)

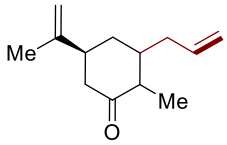


1:1

Colorless liquid; (55 mg, 64%); R_f = 0.30 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.32 – 7.27 (m, 2H), 7.16 – 7.11 (m, 1H), 7.02 – 6.97 (m, 2H), 5.73 – 5.68 (m, 0.5H), 5.64 – 5.60 (m, 1.5H), 2.73 – 2.66 (m, 0.5H), 2.52 – 2.48 (m, 2.5H), 2.46 – 2.43 (m, 0.5H), 2.32 – 2.21 (m, 1.5H), 2.07 – 1.97 (m, 1H), 1.96 – 1.93 (m, 0.5H), 1.82 – 1.76 (m, 1.5H), 1.73 – 1.66 (m, 0.5H), 1.43 – 1.35 (m, 0.5H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 172.3, 172.2, 150.7, 133.9, 131.2, 129.7, 129.3, 125.7, 121.5, 44.8, 38.6,

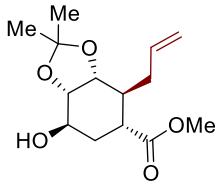
37.1, 33.3, 32.6, 32.0, 31.3, 30.8, 29.3; ESI-HRMS: Calcd for $C_{14}H_{16}O_2^+$ $[M+Na]^+$ 239.1043, found 239.1041.

(3R,5S)-3-allyl-2-methyl-5-(prop-1-en-2-yl)cyclohexan-1-one (3bg)



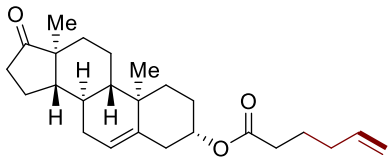
Colorless liquid; (32 mg, 41%); R_f = 0.34 (50 : 1 petroleum ether / ethyl acetate); dr = 6:3:1; ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.83 – 5.60 (m, 1H), 5.09 – 4.94 (m, 2H), 4.81 – 4.64 (m, 2H), 2.72 – 2.23 (m, 4H), 2.22 – 2.05 (m, 2H), 2.02 – 1.92 (m, 1H), 1.72 – 1.60 (m, 5H), 1.10 (d, *J* = 6.8 Hz, 0.91H), 1.04 (d, *J* = 6.5 Hz, 0.29H), 1.01 (d, *J* = 6.8 Hz, 1.8H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 213.7, 212.8, 147.5, 146.9, 136.4, 135.7, 117.1, 116.4, 111.4, 109.8, 49.1, 48.3, 46.2, 43.8, 40.6, 40.5, 40.4, 39.6, 38.0, 33.0, 31.8, 31.2, 21.6, 20.7, 13.7, 11.7; In line with previously reported spectra.[20]

methyl (3aS,4S,7S,7aR)-4-allyl-7-hydroxy-2,2-dimethylhexahydrobenzo[d][1,3]dioxole-5-carboxylate (3bh)



Colorless liquid; (32 mg, 30%); R_f = 0.29 (3 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.86 – 5.72 (m, 1H), 5.11 – 5.07 (m, 1H), 5.05 (s, 1H), 4.28 – 4.19 (m, 1H), 4.05 – 3.97 (m, 2H), 3.68 (s, 3H), 2.57 (dt, *J* = 8.62, 4.72 Hz, 1H), 2.31 – 2.15 (m, 3H), 2.10 – 2.02 (m, 2H), 1.73 – 1.67 (m, 1H), 1.48 (s, 3H), 1.35 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 175.0, 134.9, 117.6, 108.8, 78.1, 76.4, 67.8, 51.7, 39.5, 39.1, 35.2, 30.6, 28.1, 26.2; ESI-HRMS: Calcd for $C_{14}H_{23}O_5^+$ $[M+H]^+$ 271.1540, found 271.1542.

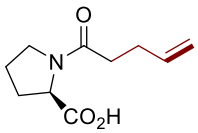
(3S,8R,9S,10R,13S,14S)-10,13-dimethyl-17-oxo-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl hex-5-enoate (3bi)



White solid; mp 145–147 °C; (92 mg, 60%): R_f = 0.28 (30 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.86 – 5.71 (m, 1H), 5.41 (d, *J* = 5.1 Hz, 1H), 5.08 – 4.96 (m, 2H), 4.67 – 4.57 (m, 1H), 2.51 – 2.42 (m, 1H), 2.36 – 2.27 (m, 4H), 2.14 – 2.05 (m, 4H), 2.00 – 1.92 (m, 1H), 1.91 – 1.82 (m, 3H), 1.77 – 1.70 (m, 2H), 1.69 – 1.64 (m, 3H), 1.62 – 1.44 (m, 3H), 1.34 – 1.25 (m, 2H), 1.21 – 1.11 (m, 1H), 1.05 (s, 3H), 1.04 – 0.99 (m, 1H), 0.89 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 221.0, 173.0, 137.7, 121.8, 115.3, 73.5, 51.7, 50.1, 47.5, 38.1, 36.9, 36.7, 35.8, 33.9, 33.0, 31.4, 31.4, 30.7, 27.7, 24.1, 21.8,

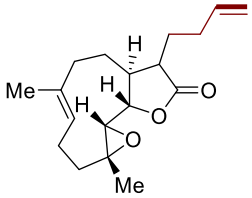
20.3, 19.3, 13.5; ESI-HRMS: Calcd for $C_{25}H_{36}O_3^+$ [M+H]$^+$ 385.2737, found 385.2723.

hex-5-enoyl-D-proline (3bj)



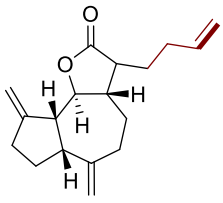
Colorless liquid; (39 mg, 46%); R_f = 0.28 (100 : 3 : 1 dichloromethane / methanol / acetic acid); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 10.71 (s, 1H), 5.85 – 5.72 (m, 1H), 5.07 – 4.96 (m, 2H), 4.61 – 4.52 (m, 1H), 3.64 – 3.57 (m, 1H), 3.53 – 3.42 (m, 1H), 2.39 – 2.23 (m, 3H), 2.16 – 1.99 (m, 5H), 1.84 – 1.70 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 174.3, 173.5, 137.8, 115.3, 59.5, 47.6, 33.4, 33.0, 27.9, 24.6, 23.5. In line with previously reported spectra.[37]

(1aS,7aR,10aR,10bR,Z)-8-(but-3-en-1-yl)-1a,5-dimethyl-2,3,6,7,7a,8,10a,10b-octahydrooxireno[2',3':9,10]cyclodeca[1,2-b]furan-9(1aH)-one (3bk)



Colorless liquid; (70 mg, 60%); R_f = 0.27 (5 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.91 – 5.73 (m, 1H), 5.21 – 5.15 (m, 1H), 5.13 – 5.06 (m, 1H), 5.05 – 5.00 (m, 1H), 3.80 (t, *J* = 9.0 Hz, 1H), 2.70 (d, *J* = 9.0 Hz, 1H), 2.43 – 2.11 (m, 7H), 2.09 – 1.95 (m, 2H), 1.93 – 1.74 (m, 3H), 1.70 (s, 3H), 1.68 – 1.59 (m, 1H), 1.29 (s, 3H), 1.26 – 1.18 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 176.7, 137.5, 134.4, 125.0, 115.7, 82.0, 66.4, 61.4, 49.1, 46.1, 41.1, 36.6, 30.4, 29.9, 27.4, 24.0, 17.1, 16.9. ESI-HRMS: Calcd for $C_{18}H_{27}O_3^+$ [M+H]$^+$ 291.1955, found 291.1959.

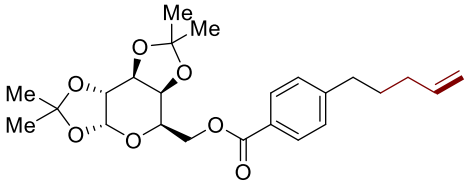
(3aS,6aR,9aR,9bS)-3-(but-3-en-1-yl)-6,9-dimethylenedecahydroazuleno[4,5-b]furan-2(3H)-one (3bl)



Colorless liquid; (46 mg, 42%); R_f = 0.24 (20 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 5.86 – 5.74 (m, 1H), 5.21 – 5.17 (m, 1H), 5.10 – 5.03 (m, 2H), 5.03 – 4.98 (m, 1H), 4.87 (s, 1H), 4.78 (s, 1H), 3.90 (t, *J* = 9.3 Hz, 1H), 2.91 – 2.85 (m, 1H), 2.83 – 2.73 (m, 1H), 2.56 – 2.43 (m, 3H), 2.37 – 2.18 (m, 3H), 2.17 – 2.00 (m, 3H), 1.98 – 1.79 (m, 3H), 1.73 – 1.63 (m, 1H), 1.38 – 1.28 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 178.0, 151.6, 149.8, 137.6, 115.5, 111.9, 109.2, 84.9, 52.0, 47.4, 47.1, 45.7, 37.4, 32.7, 32.4, 30.6, 30.1, 27.8. ESI-HRMS: Calcd for $C_{18}H_{25}O_2^+$ [M+H]$^+$ 273.1849, found

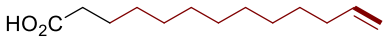
273.1849.

((3aR,5R,5aS,8aS,8bR)-2,2,7,7-tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5-yl)methyl 4-(pent-4-en-1-yl)benzoate (3bm)



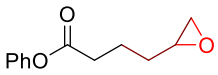
White solid; mp 100–103 °C; (74 mg, 43%); R_f = 0.30 (30 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.96 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.1 Hz, 2H), 5.90 – 5.74 (m, 1H), 5.57 (d, *J* = 4.9 Hz, 1H), 5.07 – 4.94 (m, 2H), 4.68 – 4.63 (m, 1H), 4.56 – 4.48 (m, 1H), 4.45 – 4.38 (m, 1H), 4.37 – 4.31 (m, 2H), 4.18 (t, *J* = 6.1 Hz, 1H), 2.72 – 2.61 (m, 2H), 2.09 (q, *J* = 7.0 Hz, 2H), 1.77 – 1.67 (m, 2H), 1.52 (s, 3H), 1.48 (s, 3H), 1.36 (s, 3H), 1.34 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 166.5, 148.1, 138.2, 129.8, 128.5, 127.6, 115.0, 109.7, 108.8, 96.3, 71.1, 70.7, 70.5, 66.1, 63.7, 35.3, 33.2, 30.2, 26.0, 26.0, 25.0, 24.5; ESI-HRMS: Calcd for $C_{24}H_{32}O_7^+$ $[M+H]^+$ 433.2221, found 433.2206.

tridec-12-enoic acid (3bn)



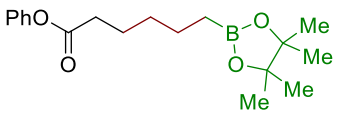
Colorless liquid; (701 mg, 55%); R_f = 0.25 (10 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 5.89 – 5.72 (m, 1H), 5.03 – 4.89 (m, 2H), 2.35 (t, *J* = 7.5 Hz, 2H), 2.04 (q, *J* = 7.0 Hz, 2H), 1.68 – 1.58 (m, 2H), 1.41 – 1.24 (m, 14H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 180.4, 139.2, 114.1, 34.1, 33.8, 29.5, 29.4, 29.4, 29.2, 29.1, 29.0, 28.9, 24.6. In line with previously reported spectra.[38]

phenyl 4-(oxiran-2-yl)butanoate (4a)



Colorless liquid; (72 mg, 87%); R_f = 0.29 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.41 – 7.33 (m, 2H), 7.25 – 7.19 (m, 1H), 7.09 – 7.04 (m, 2H), 3.01 – 2.92 (m, 1H), 2.77 (t, *J* = 4.5 Hz, 1H), 2.67 – 2.60 (m, 2H), 2.52 – 2.46 (m, 1H), 1.98 – 1.86 (m, 2H), 1.79 – 1.68 (m, 1H), 1.65 – 1.54 (m, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 171.7, 150.6, 129.3, 125.7, 121.4, 51.7, 46.8, 33.8, 31.7, 21.4; ESI-HRMS: Calcd for $C_{12}H_{14}O_3^+$ $[M+H]^+$ 207.1016, found 207.1008.

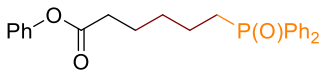
phenyl 6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexanoate (4b)



Colorless liquid; (51 mg, 40%); R_f = 0.24 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.40 – 7.34 (m, 3H), 7.24 – 7.18 (m, 1H), 7.09 – 7.05 (m, 2H), 2.55 (t, *J* = 7.6 Hz, 2H), 1.80 – 1.72 (m, 2H), 1.50 – 1.39 (m, 4H), 1.25 (s, 12H), 0.81 (t, *J* = 7.5 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 172.3, 150.8, 129.3, 125.6, 121.6,

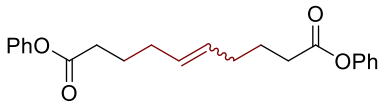
82.9, 34.3, 31.8, 24.8, 24.7, 23.6; ESI-HRMS: Calcd for $C_{23}H_{22}NO_2^+$ $[M+H]^+$ 319.2075, found 319.2061.

phenyl 6-(diphenylphosphoryl)hexanoate (4c)



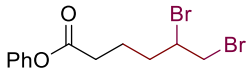
Colorless liquid; (99 mg, 63%); R_f = 0.23 (1 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.78 – 7.68 (m, 4H), 7.55 – 7.43 (m, 6H), 7.39 – 7.32 (m, 2H), 7.24 – 7.16 (m, 1H), 7.09 – 6.98 (m, 2H), 2.51 (t, *J* = 7.4 Hz, 2H), 2.34 – 2.24 (m, 2H), 1.79 – 1.63 (m, 4H), 1.59 – 1.48 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 171.8, 150.6, 132.9 (d, *J* = 98.0 Hz), 131.6 (d, *J* = 2.6 Hz), 130.7 (d, *J* = 9.2 Hz), 129.3, 128.6 (d, *J* = 11.6 Hz), 125.6, 121.4, 33.9, 30.2 (d, *J* = 14.6 Hz), 29.5 (d, *J* = 72.1 Hz), 24.2, 21.1 (d, *J* = 3.8 Hz); ESI-HRMS: Calcd for $C_{24}H_{25}O_3P$ $[M+H]^+$ 393.1614, found 393.1599.

diphenyl dec-5-enedioate (4d)



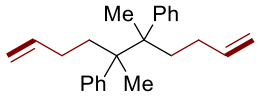
Colorless liquid; (89 mg, 63%); E:Z = 4:1; R_f = 0.30 (50 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.40 – 7.33 (m, 4H), 7.24 – 7.18 (m, 2H), 7.09 – 7.04 (m, 4H), 5.52 – 5.43 (m, 2H), 2.59 – 2.52 (m, 4H), 2.23 – 2.11 (m, 4H), 1.88 – 1.77 (m, 4H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 172.1, 150.7, 130.3, 129.7, 129.4, 125.7, 121.6, 33.7, 33.7, 31.9, 26.6, 24.8, 24.7; ESI-HRMS: Calcd for $C_{22}H_{24}O_4^+$ $[M+H]^+$ 353.1747, found 353.1734.

phenyl 5,6-dibromohexanoate (4e)



Colorless liquid; (109 mg, 78%); R_f = 0.27 (30 : 1 petroleum ether / ethyl acetate); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.35 – 7.27 (m, 2H), 7.19 – 7.12 (m, 1H), 7.07 – 6.95 (m, 2H), 4.18 – 4.08 (m, 1H), 3.83 – 3.77 (m, 1H), 3.57 (t, *J* = 10.1 Hz, 1H), 2.60 – 2.50 (m, 2H), 2.29 – 2.19 (m, 1H), 2.05 – 1.94 (m, 1H), 1.90 – 1.76 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 171.4, 150.6, 129.4, 125.8, 121.5, 51.9, 35.9, 35.2, 33.4, 22.3; ESI-HRMS: Calcd for $C_{12}H_{14}Br_2O_2^+$ $[M+H]^+$ 348.9433, found 348.9420.

(5,6-dimethyldeca-1,9-diene-5,6-diyl)dibenzene (5e)



Colorless liquid; (20 mg, 61%); R_f = 0.32 (petroleum ether); ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.23 – 7.11 (m, 6H), 7.03 – 6.86 (m, 4H), 5.82 – 5.66 (m, 2H), 4.97 – 4.81 (m, 4H), 2.28 – 2.04 (m, 2H), 1.83 – 1.74 (m, 2H), 1.68 – 1.54 (m, 4H), 1.30 (s, 3H), 1.26 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*): δ 143.4, 139.6, 129.6, 129.5, 126.7, 126.6, 125.5, 113.7, 47.9, 47.7, 34.8, 34.4, 29.2, 29.1, 21.9, 21.4.

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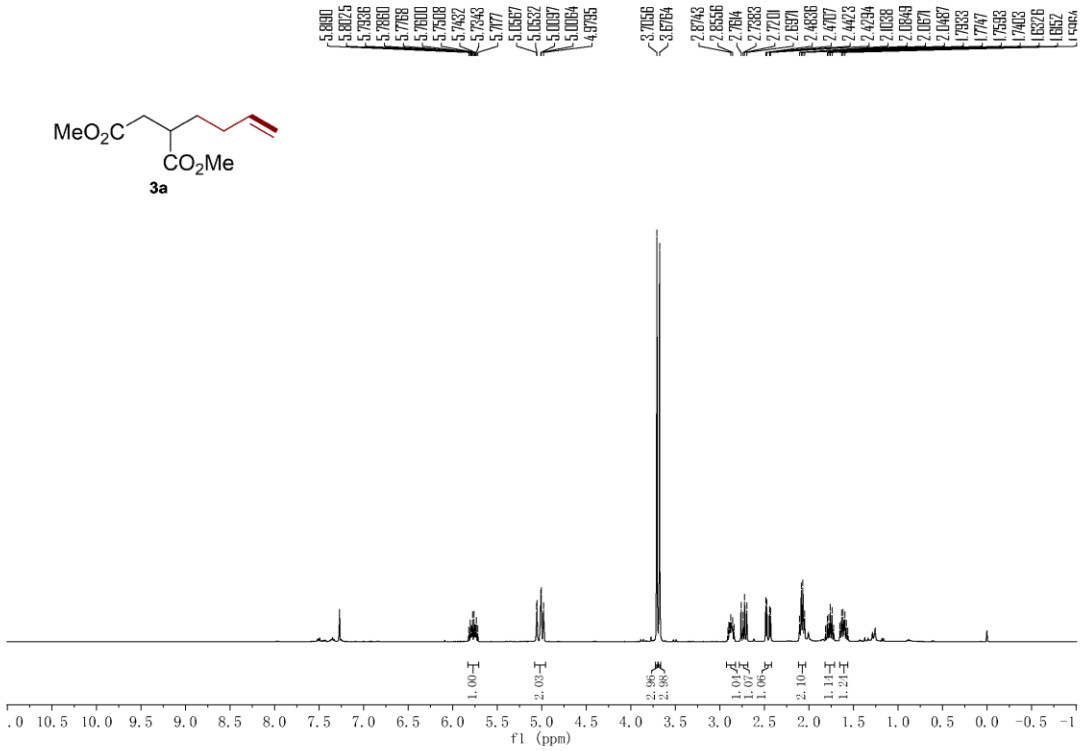
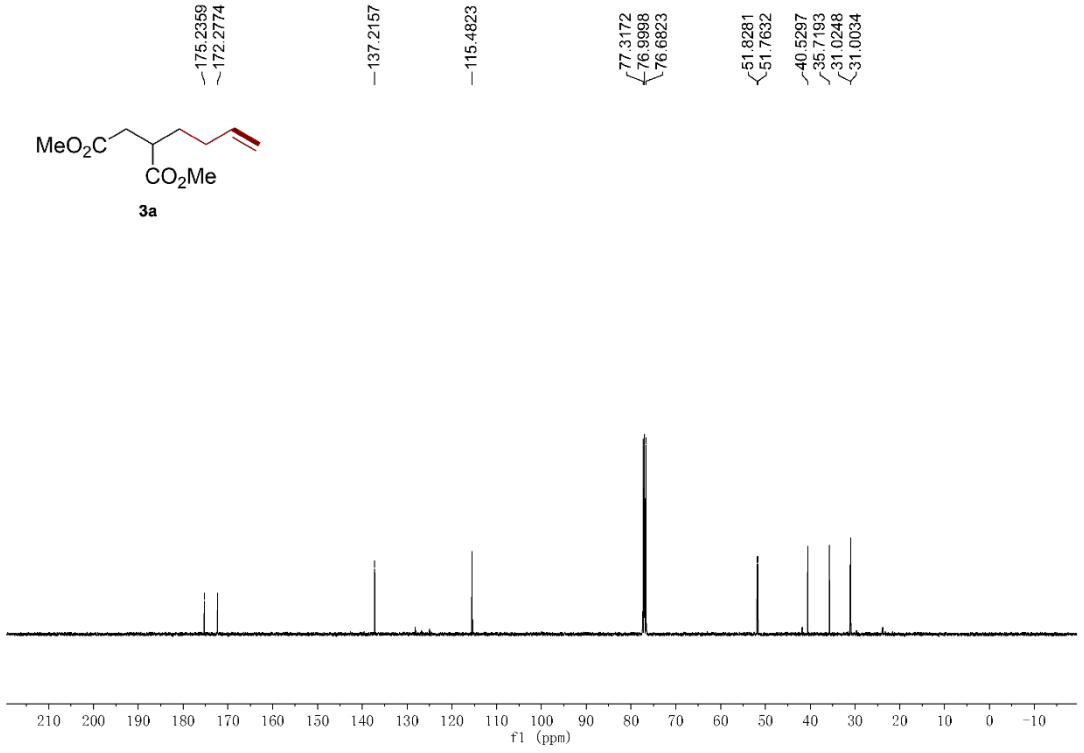
10 Spectral Data

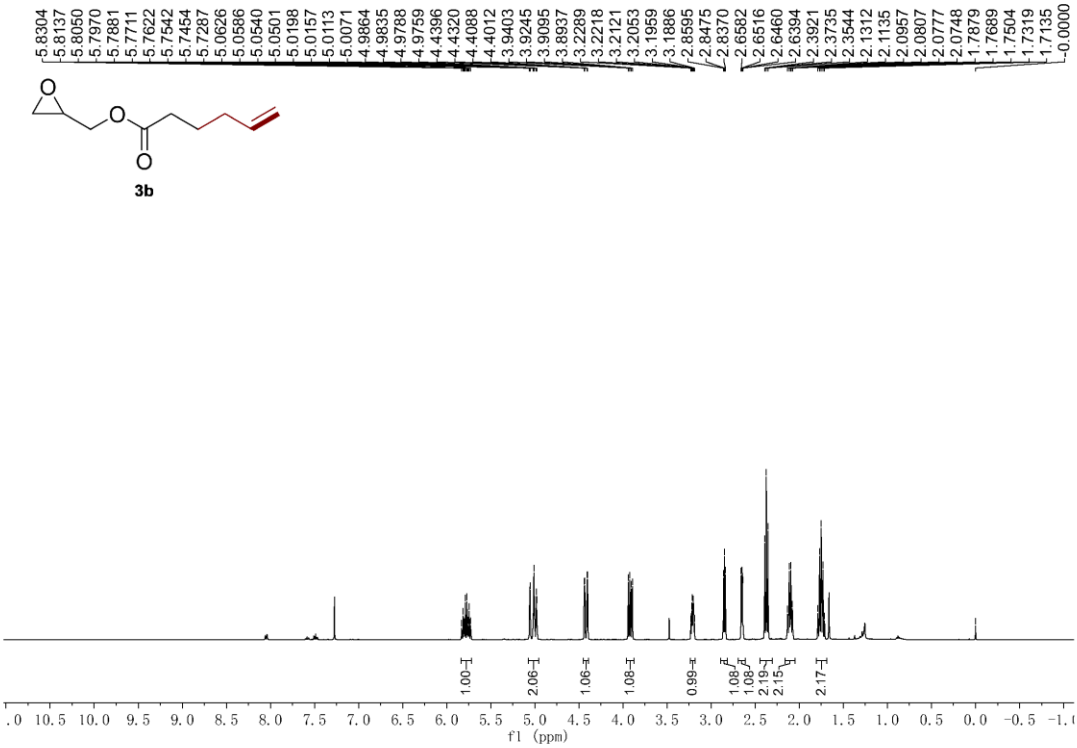
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3a



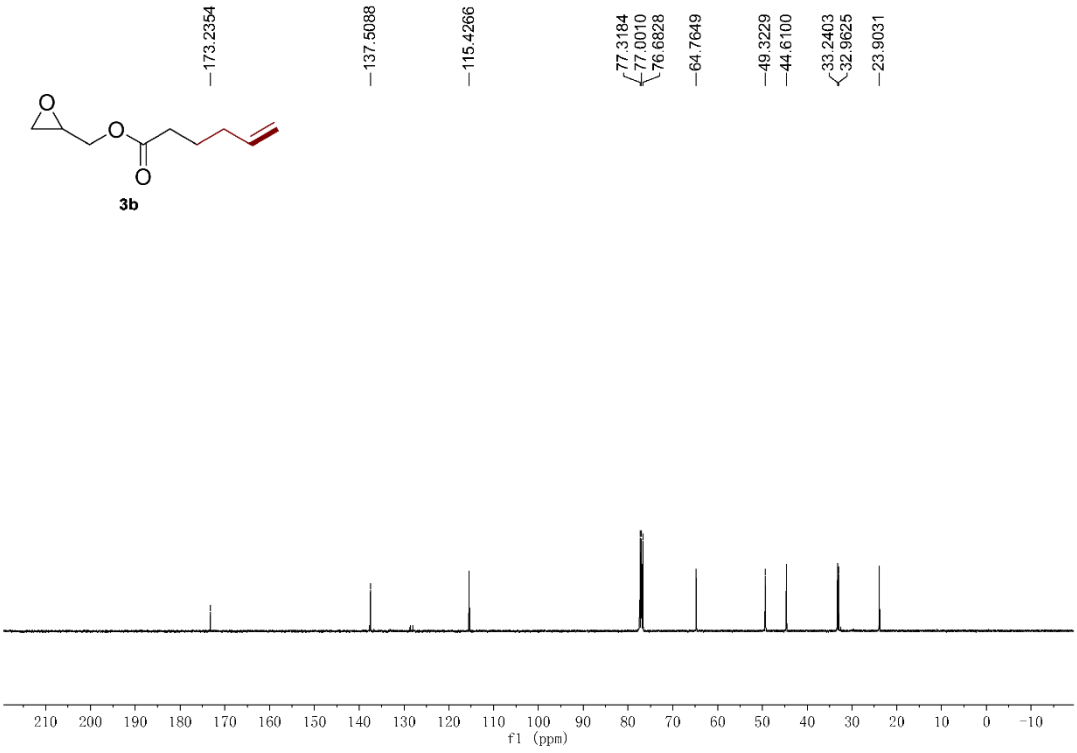
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3a



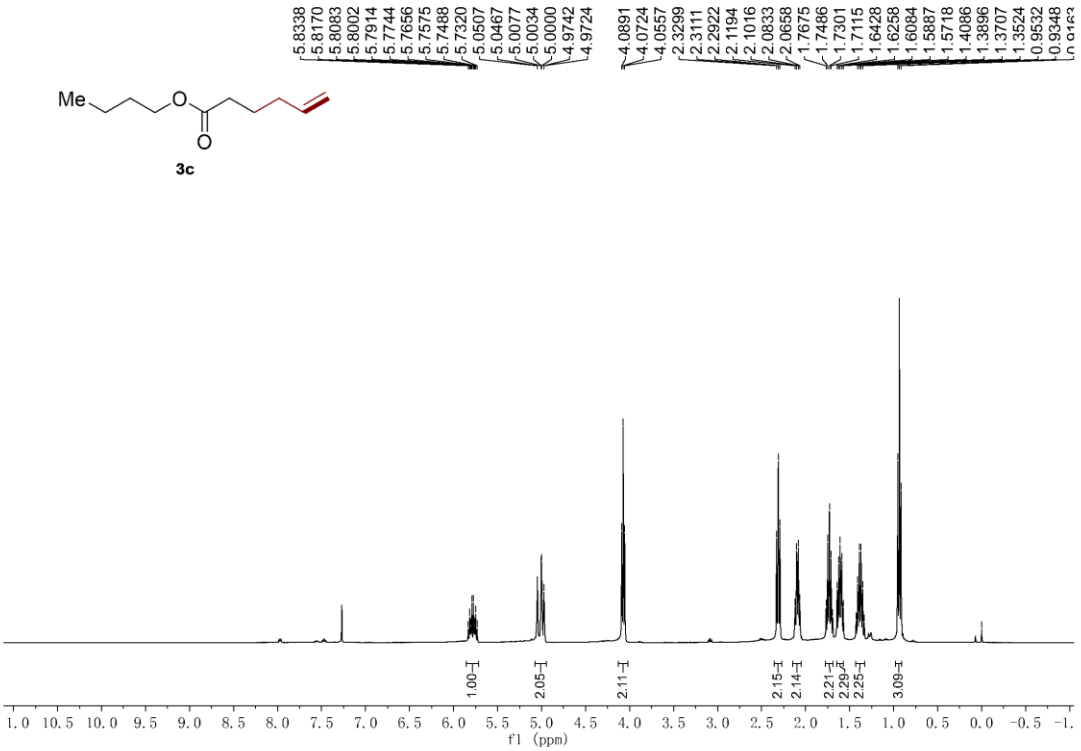
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3b



^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3b



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3c



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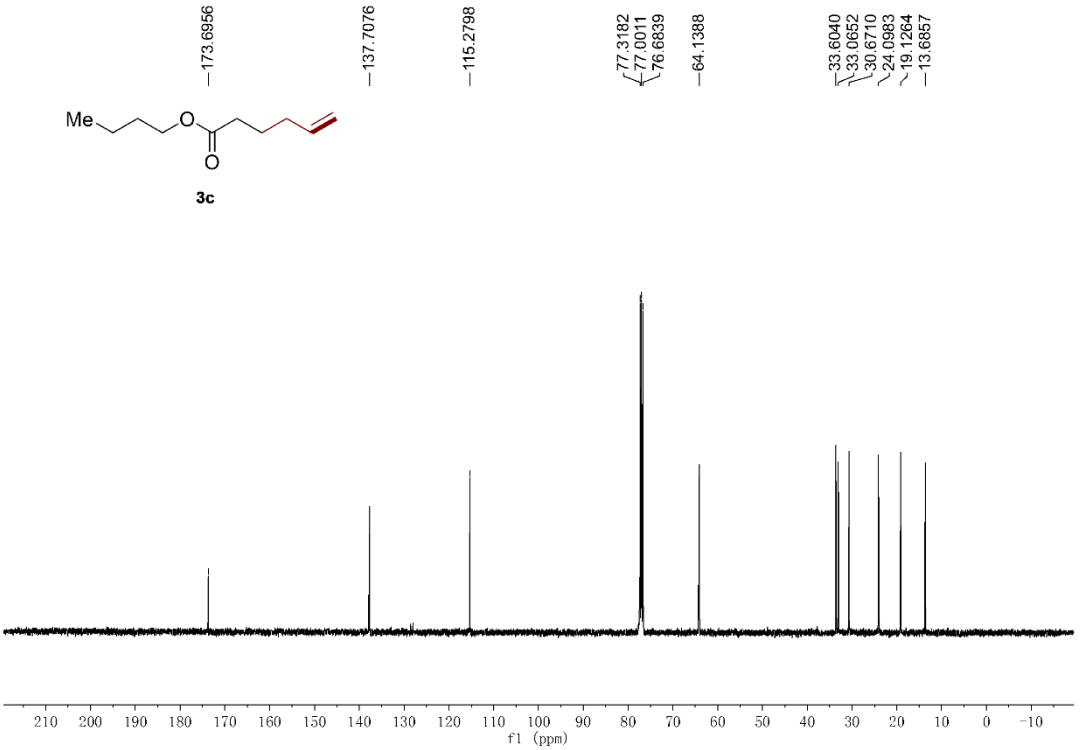
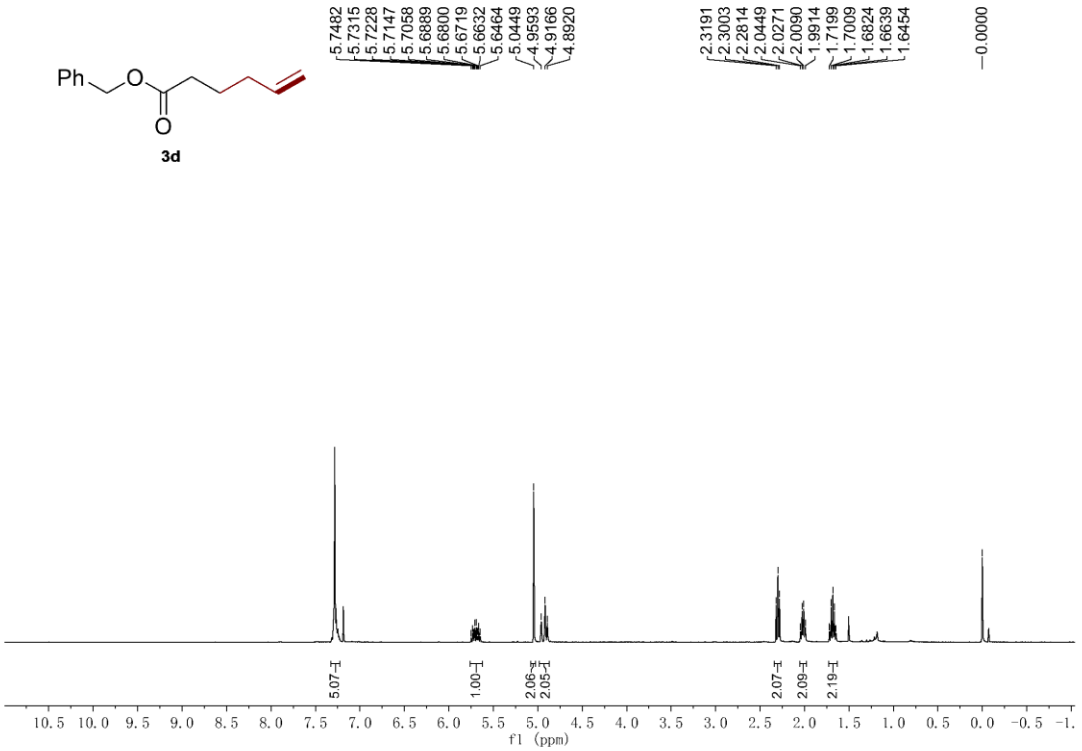


^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3d



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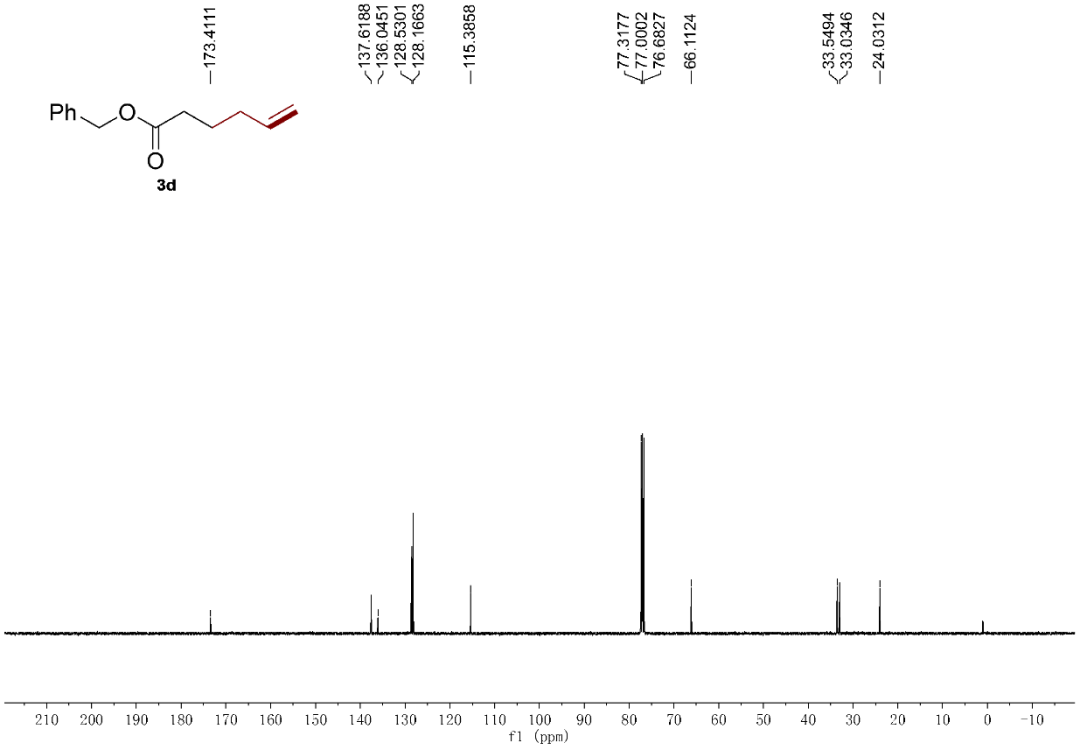
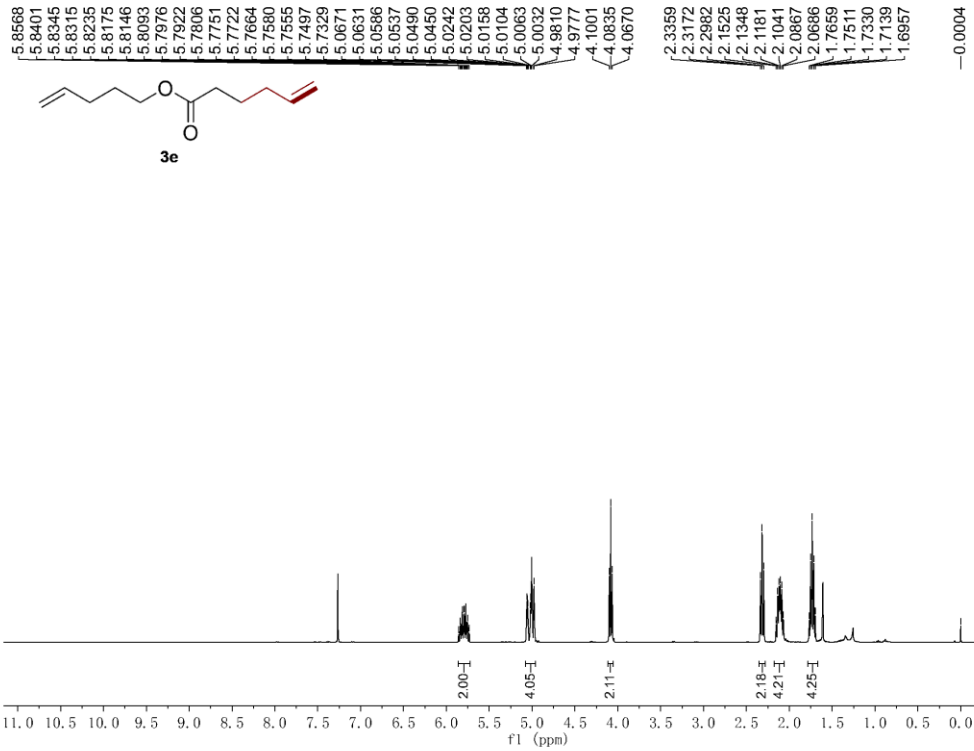
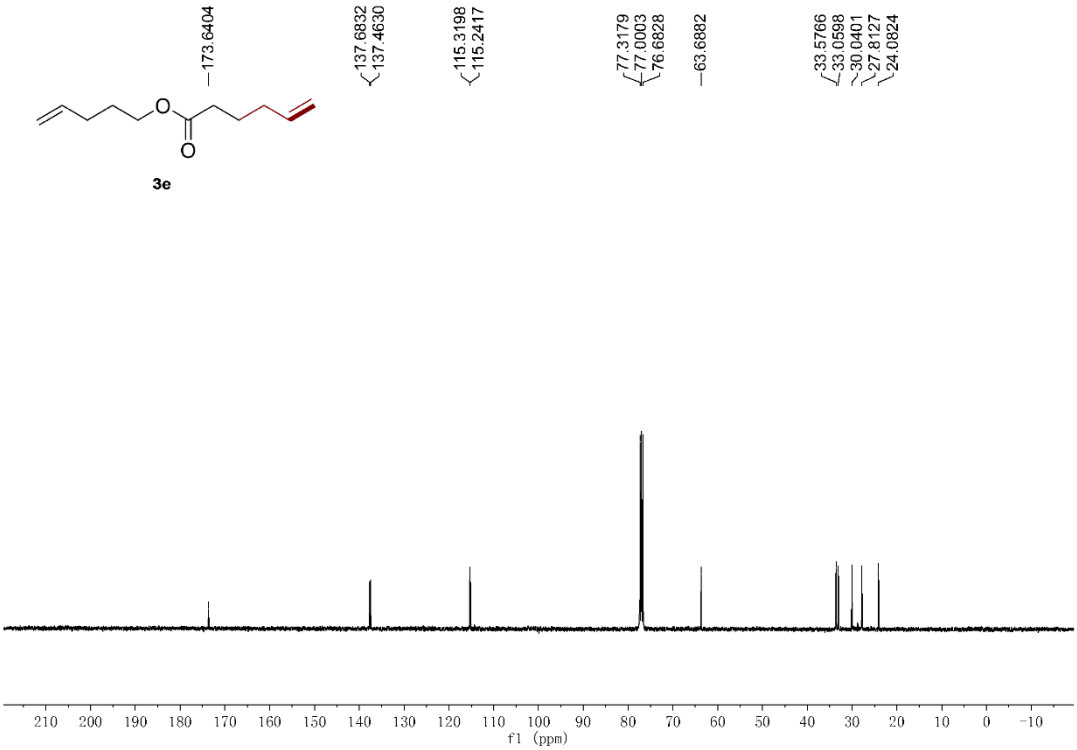


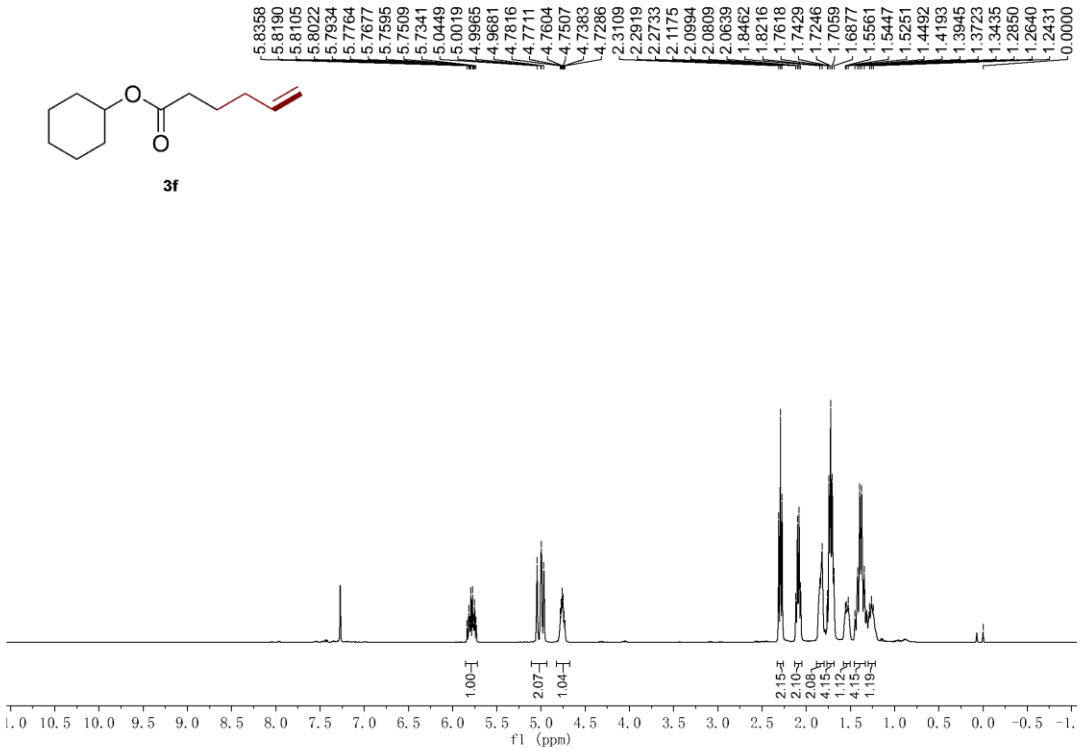
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3e



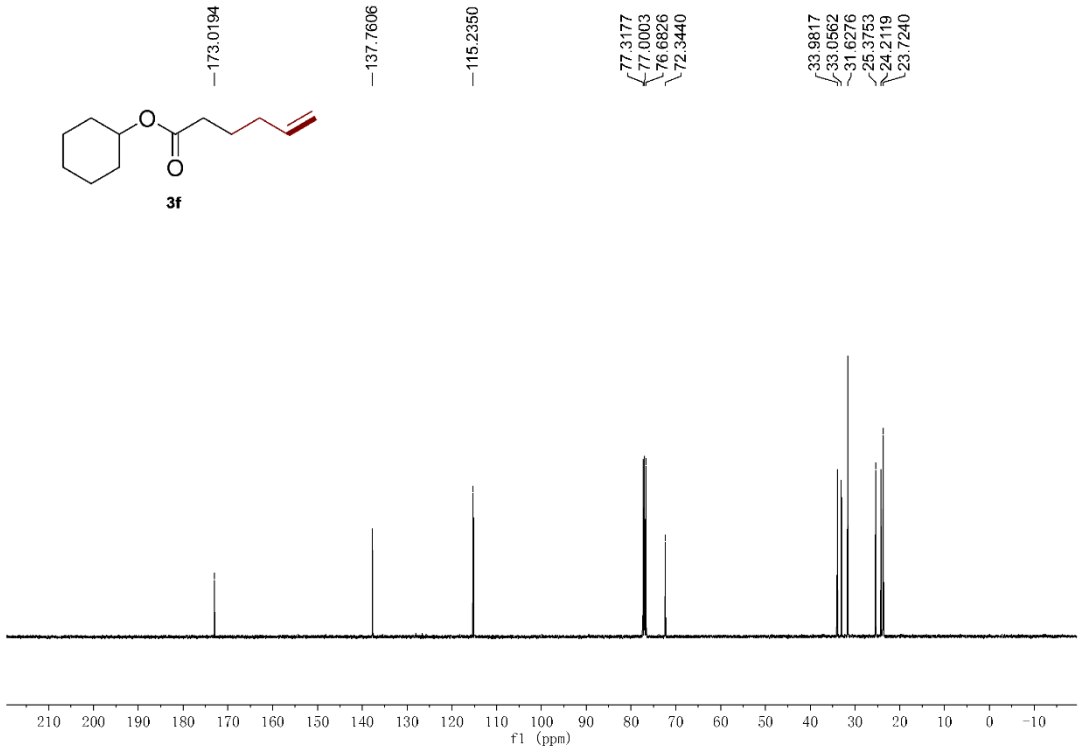
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3e



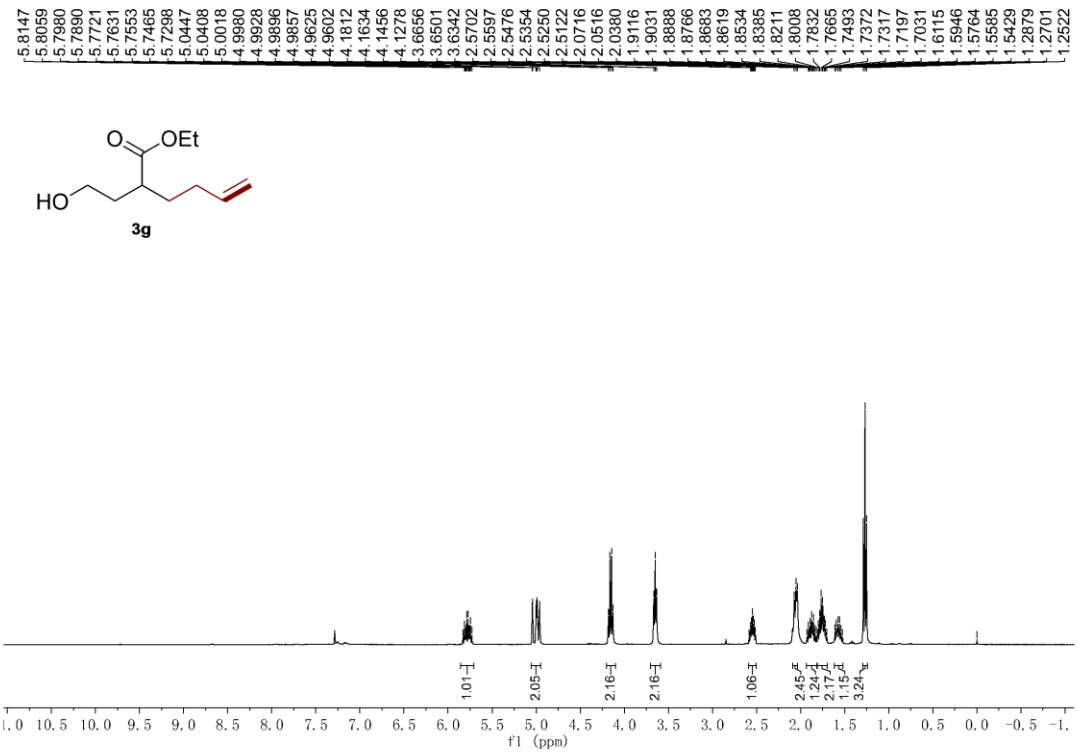
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3f



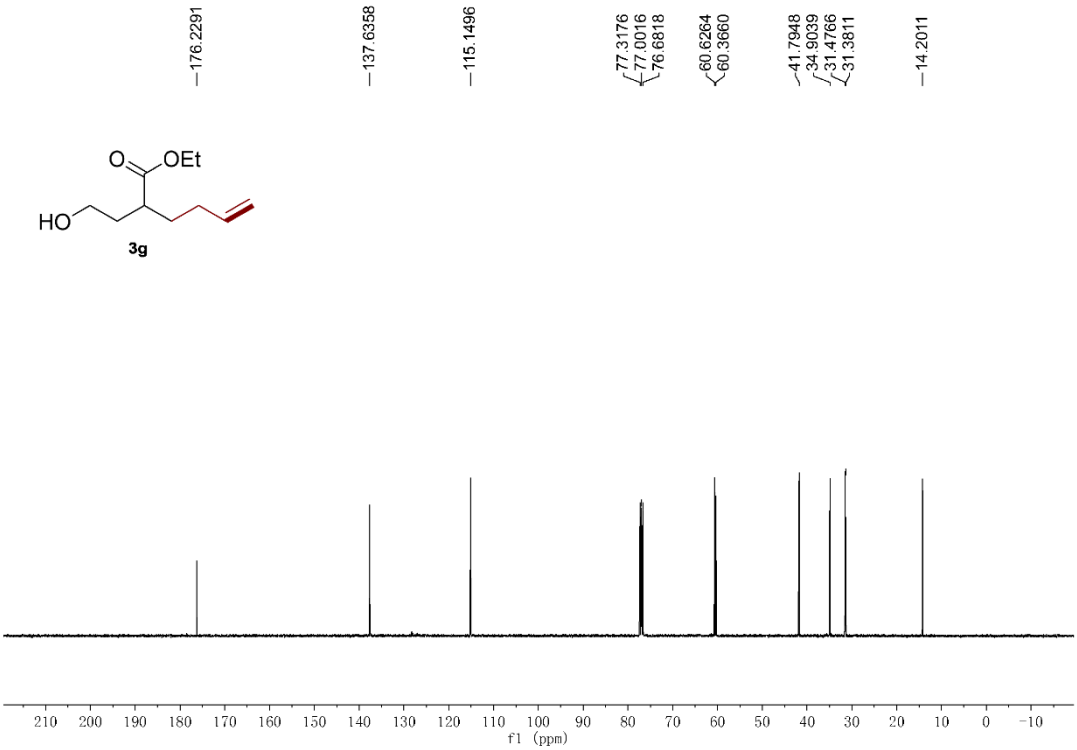
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3f



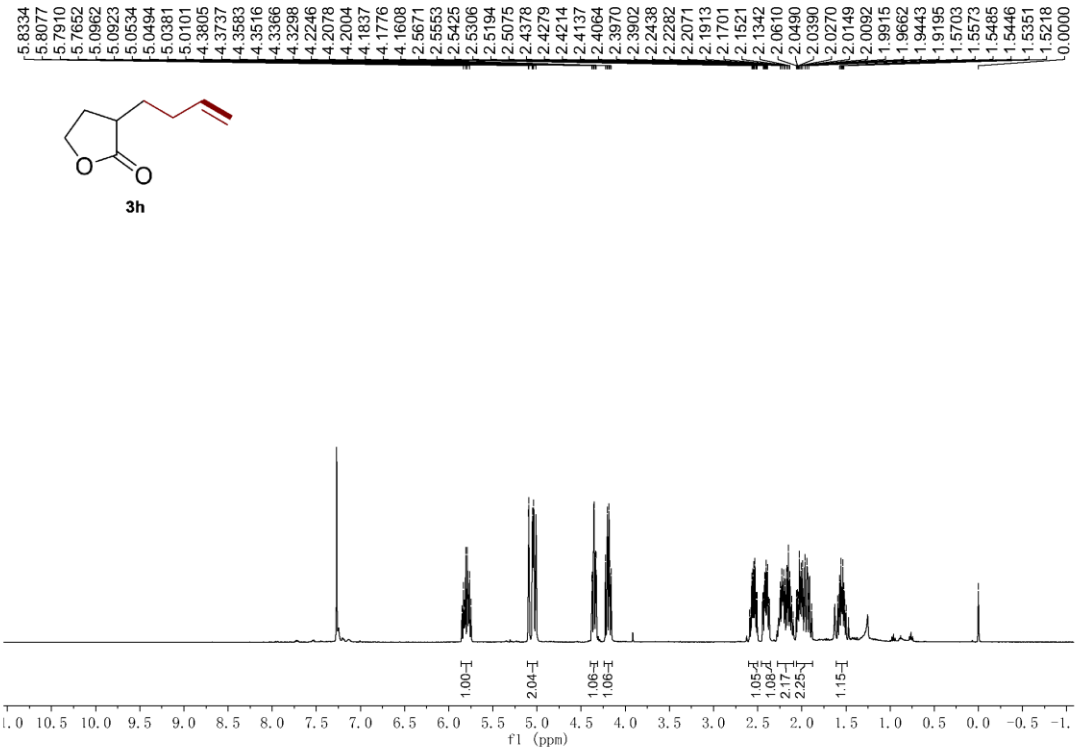
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3g



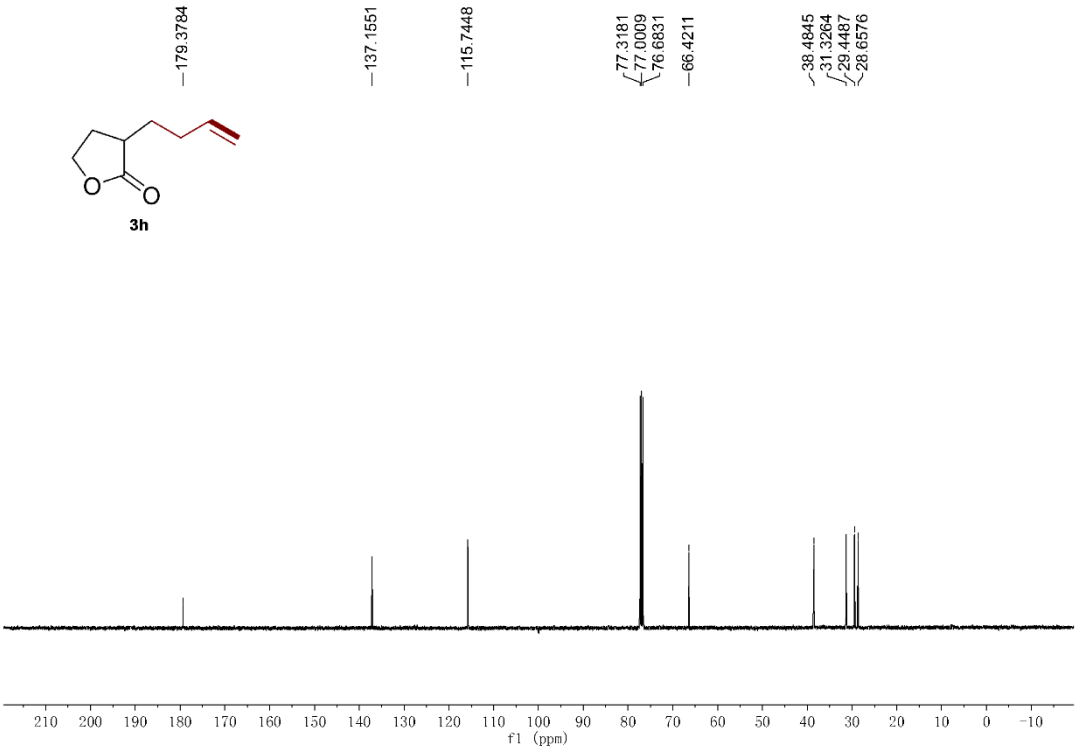
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3g



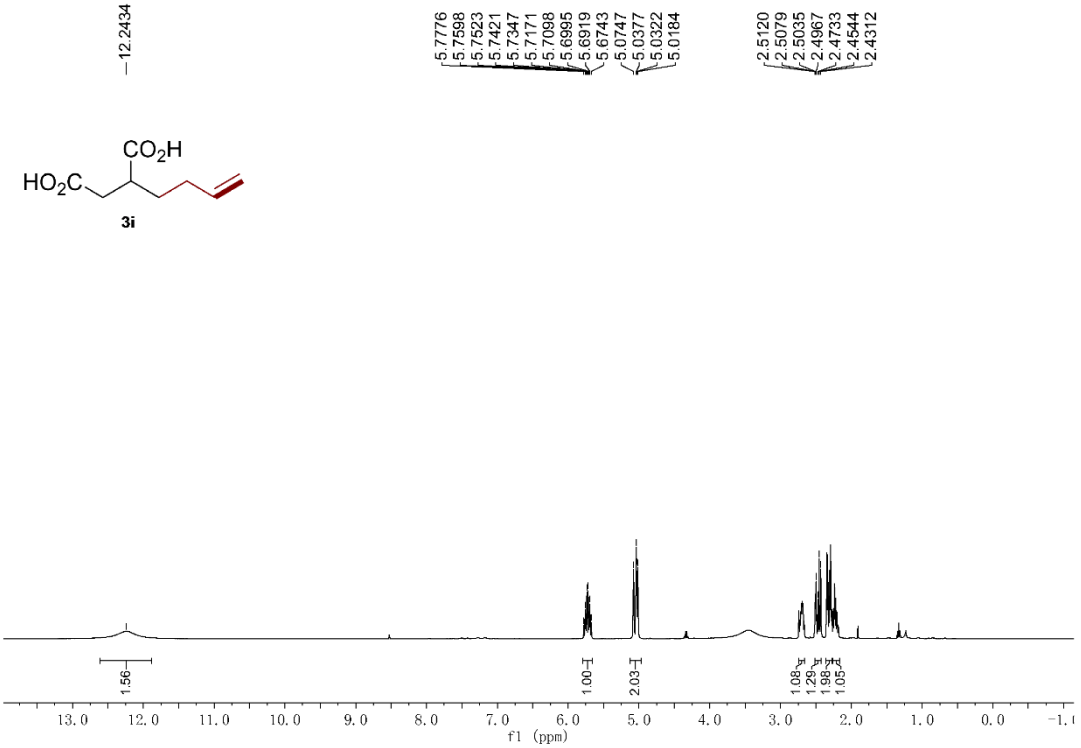
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3h



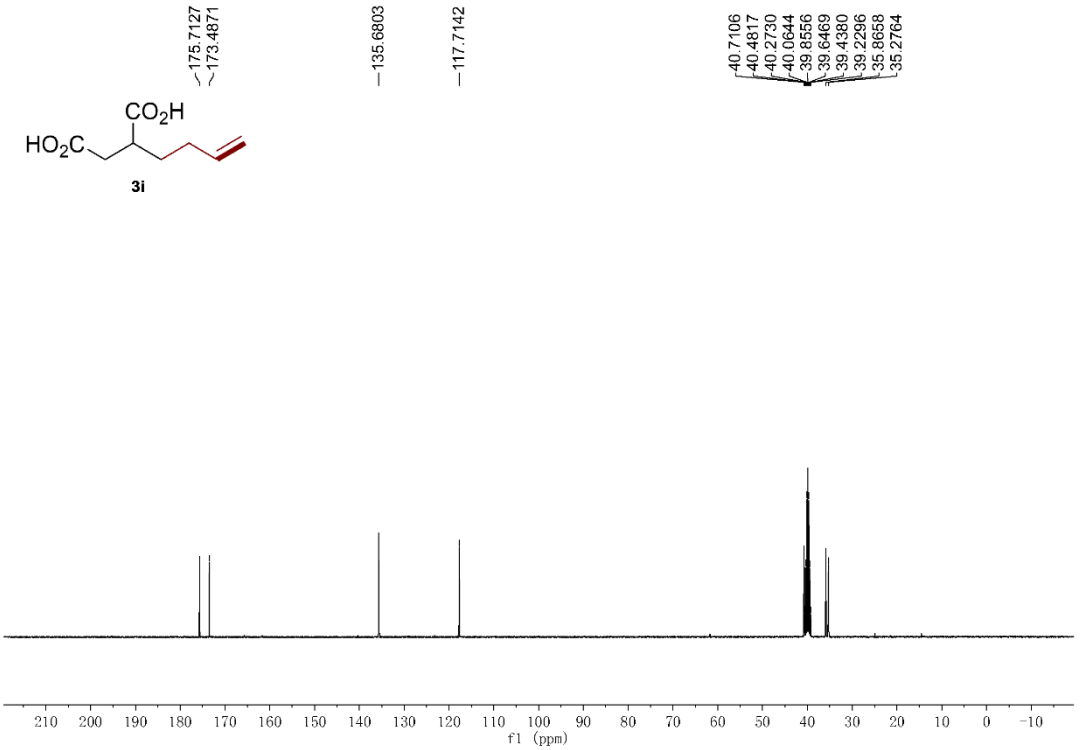
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3h



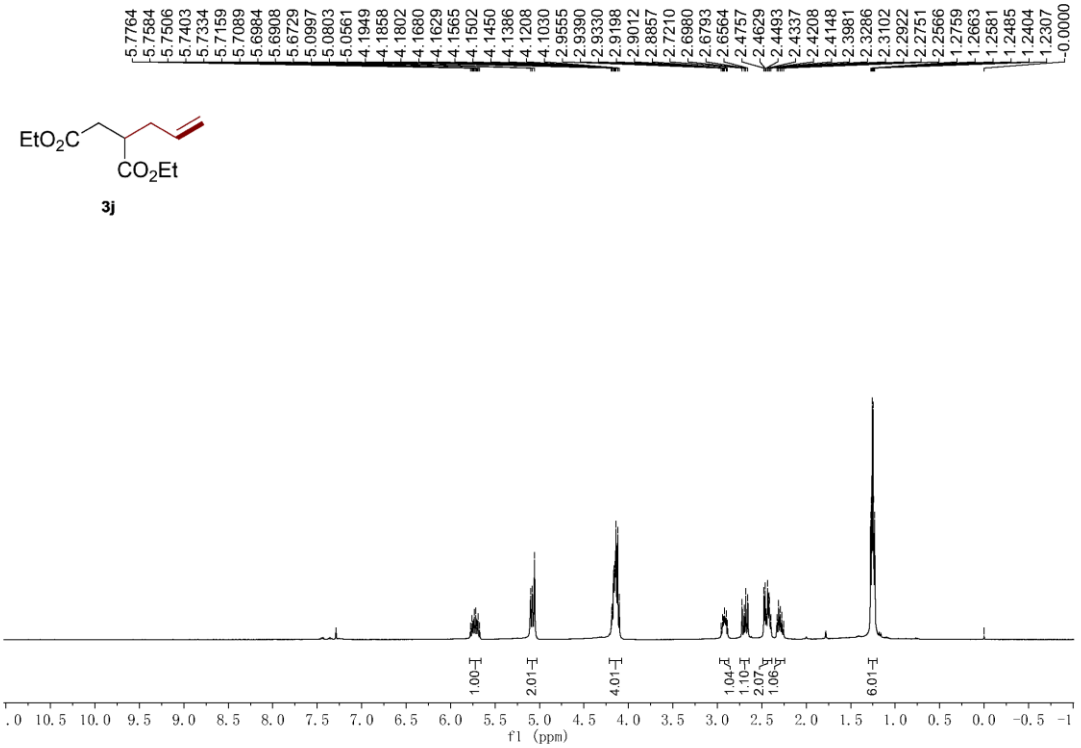
^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 3i



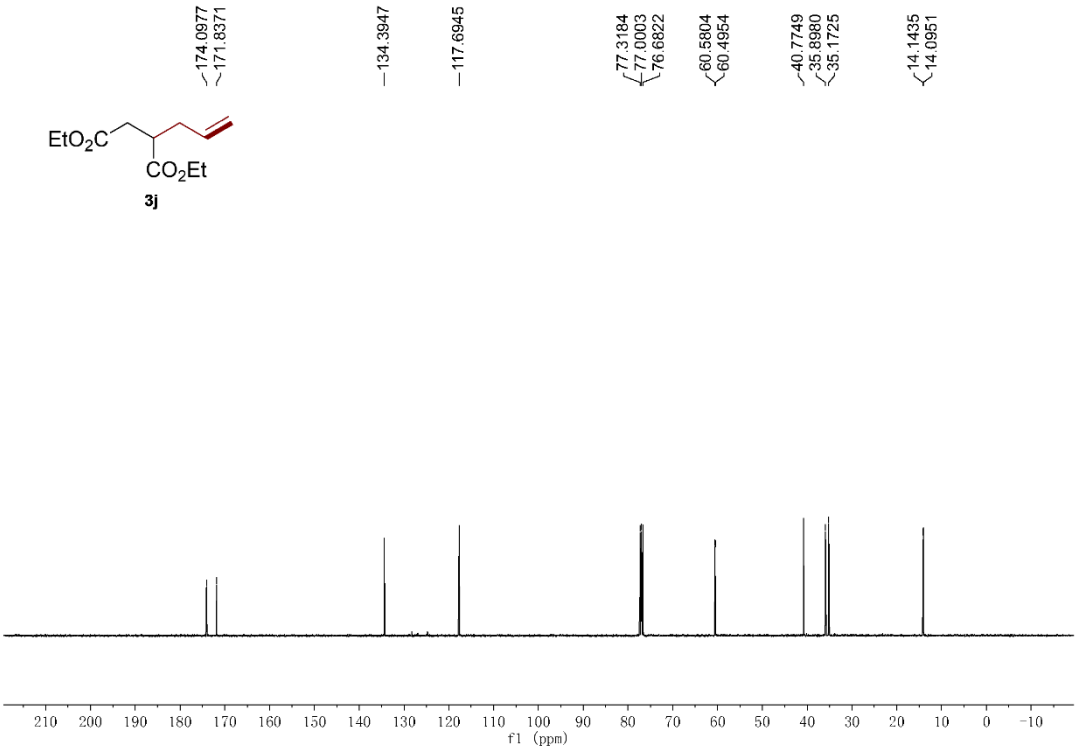
^{13}C NMR (101 MHz, DMSO-d_6) spectrum of 3i



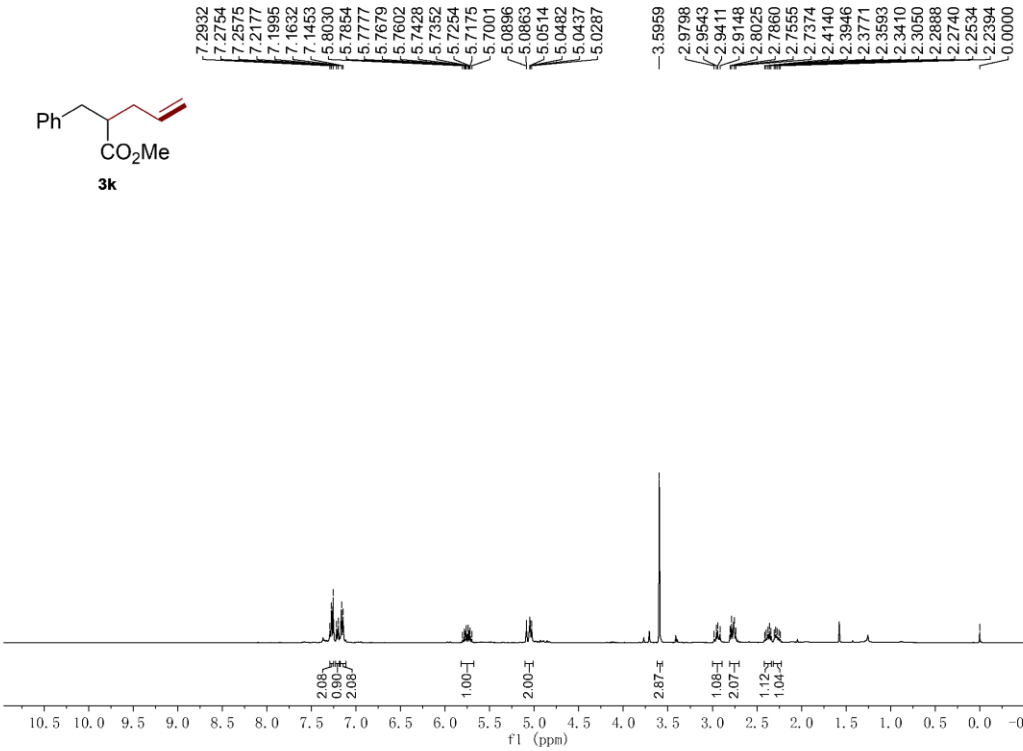
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3j



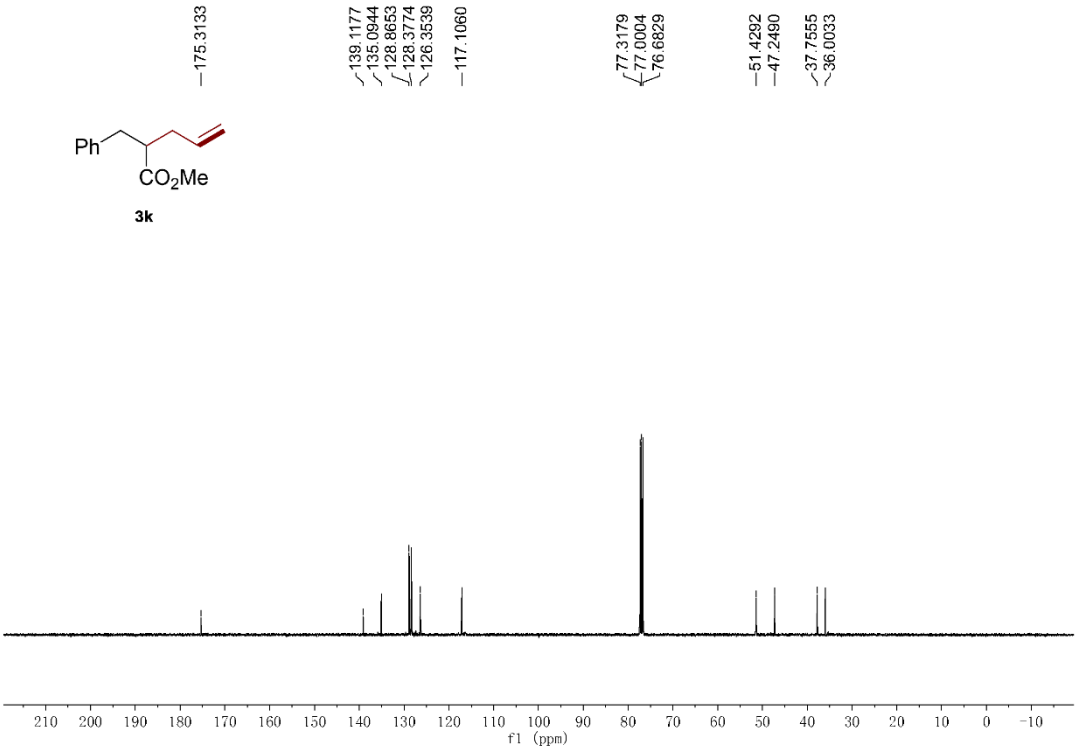
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3j



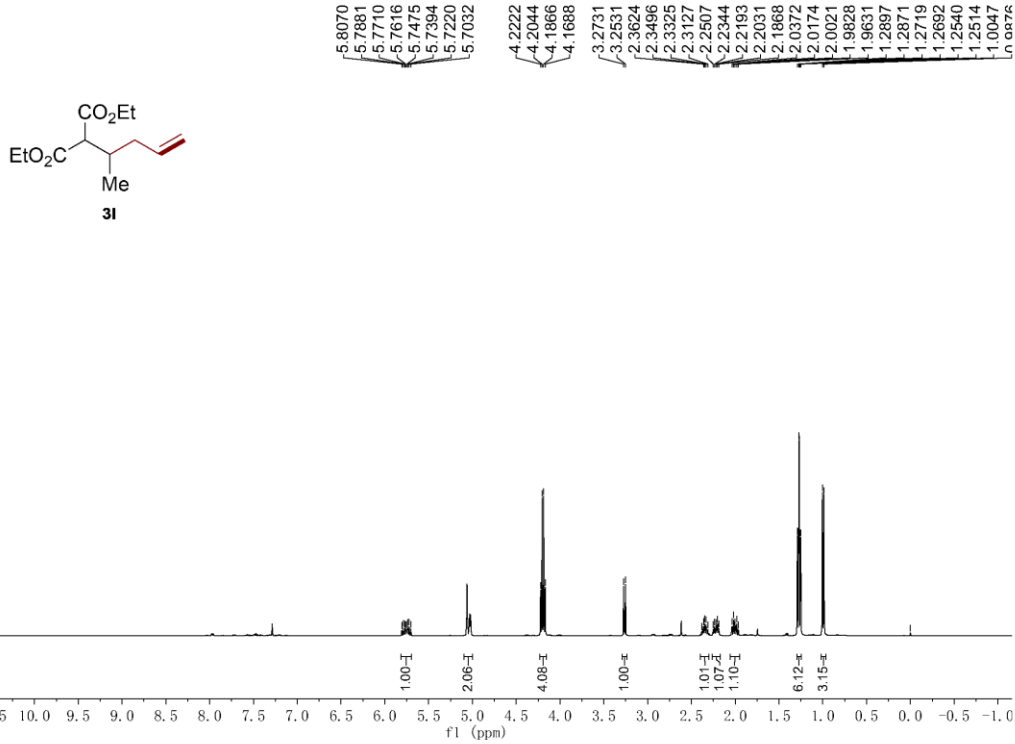
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3k



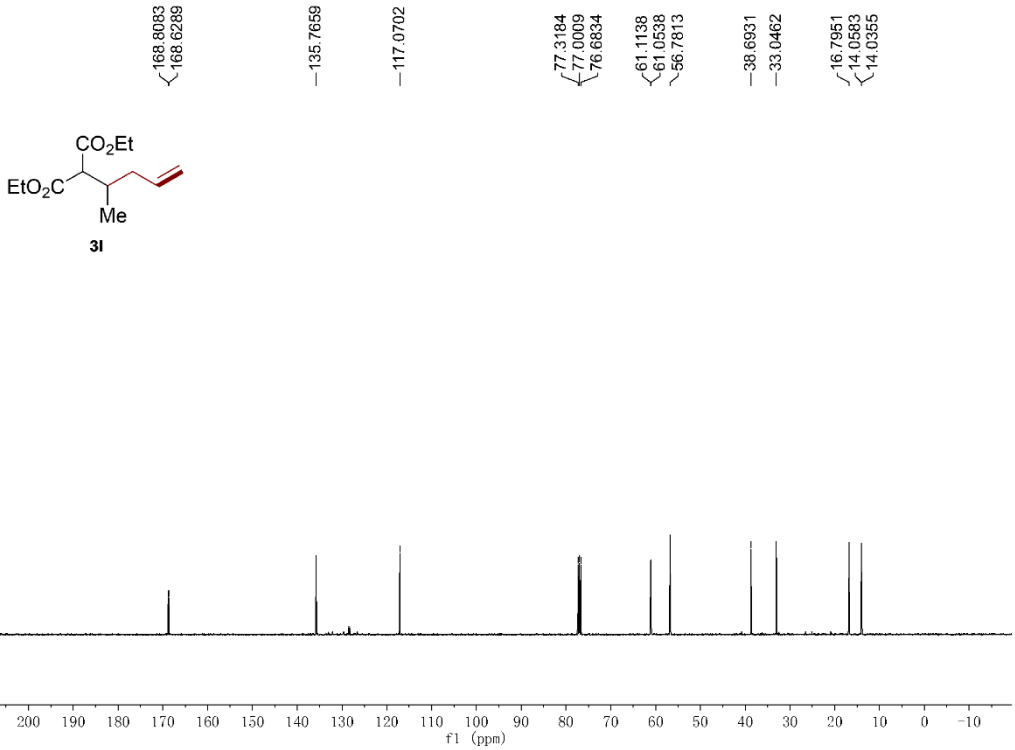
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3k



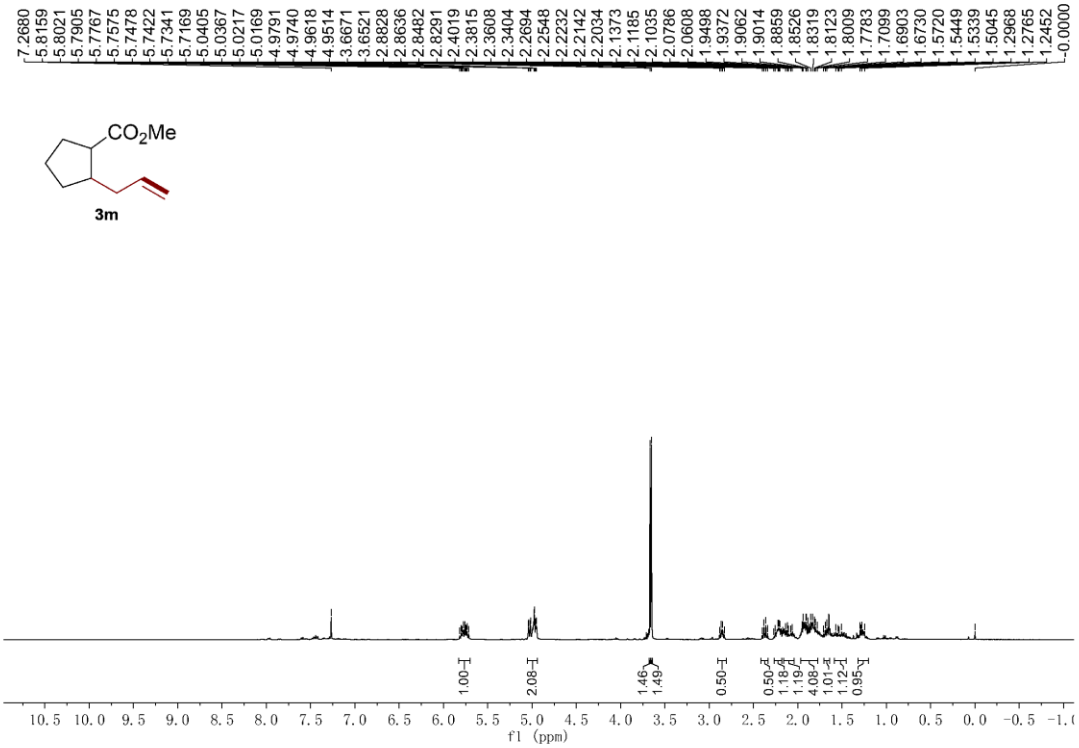
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3l



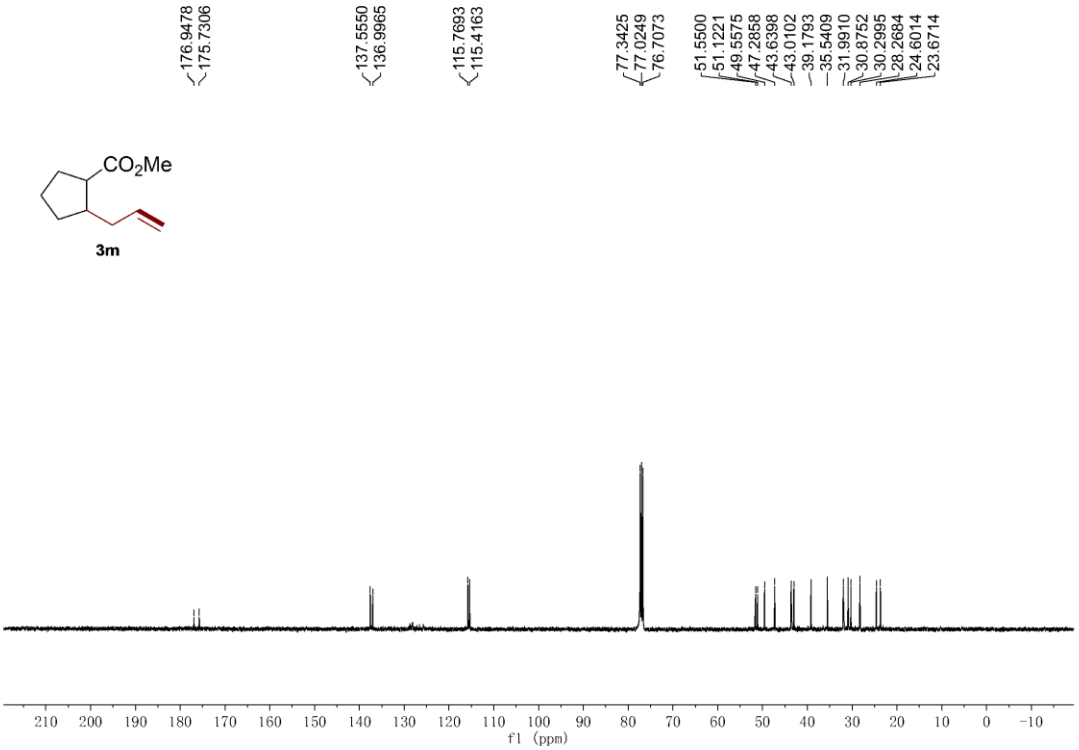
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3l



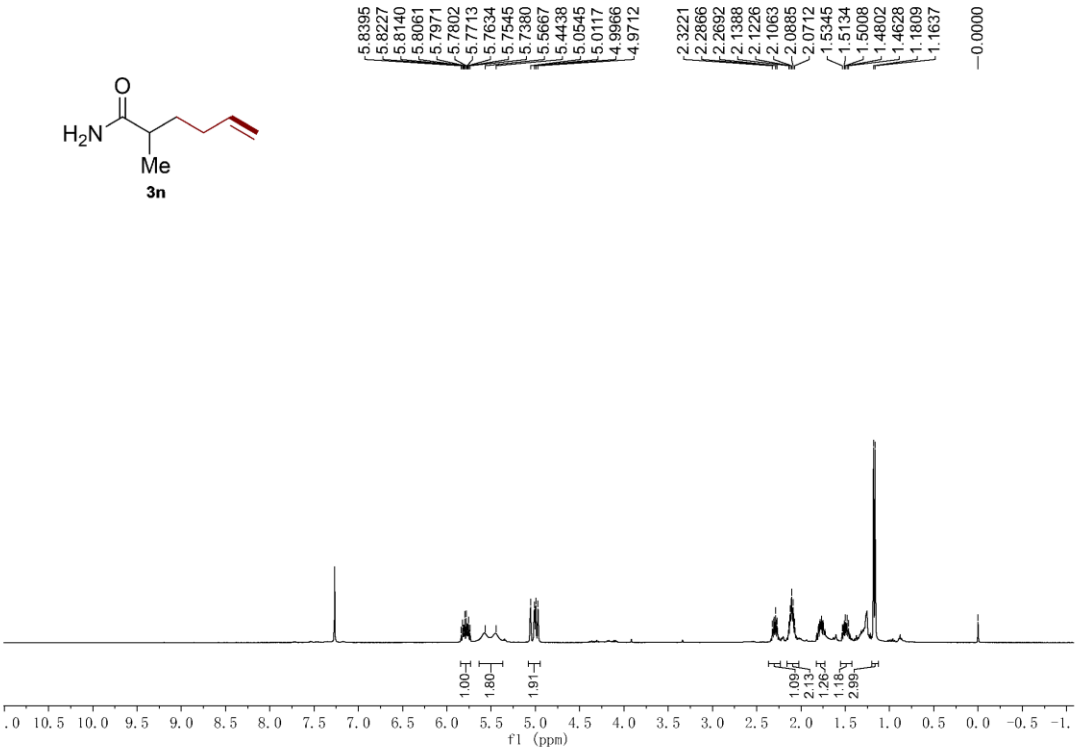
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3m



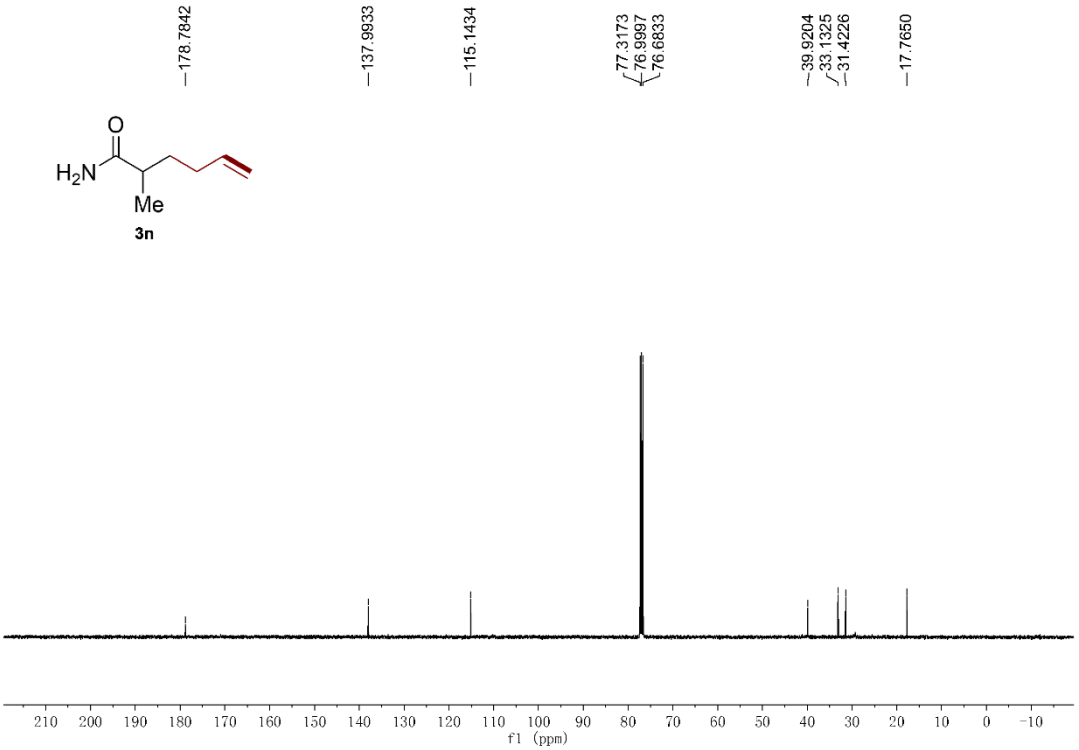
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3m



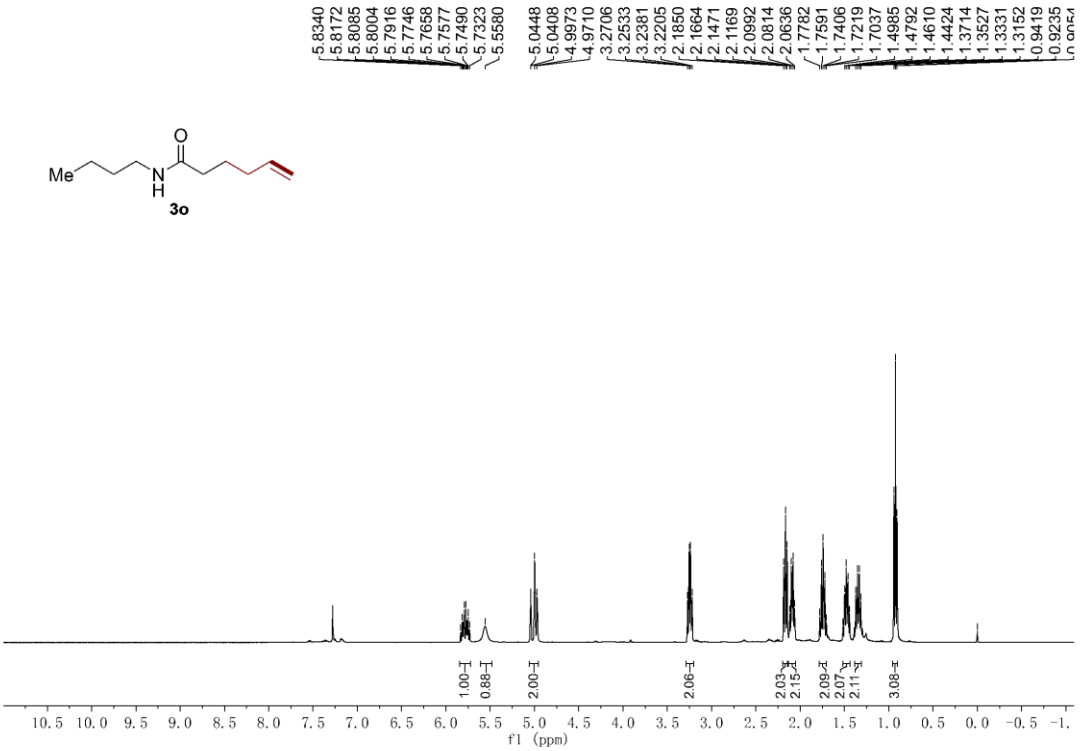
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3n



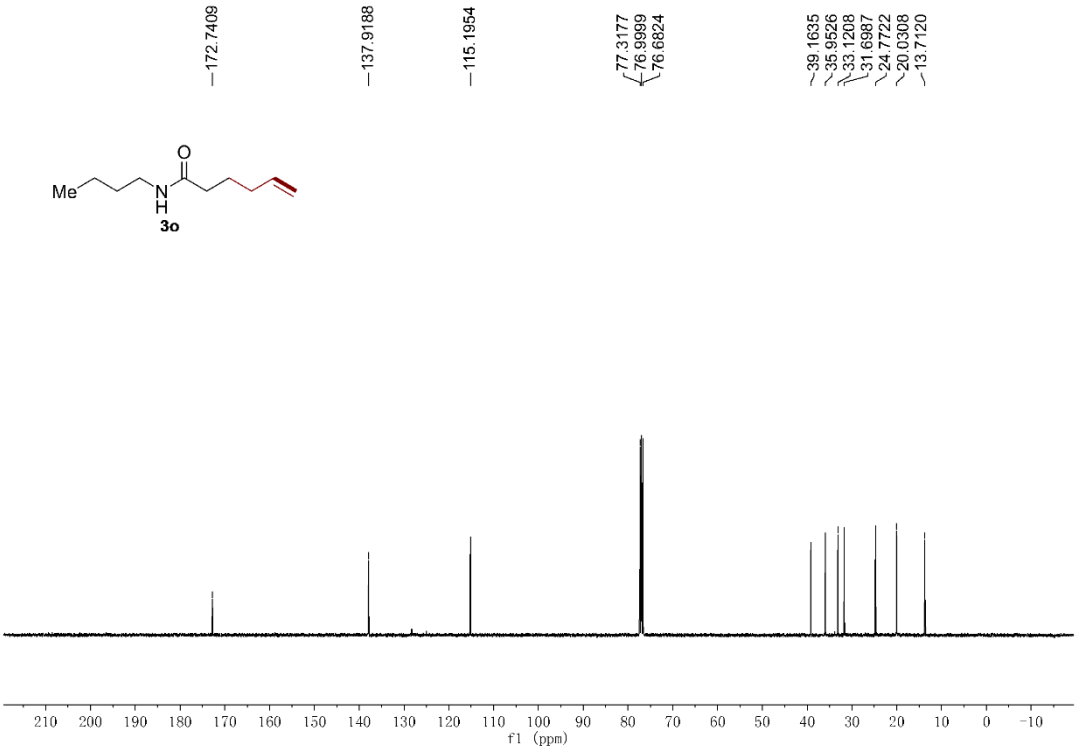
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3n



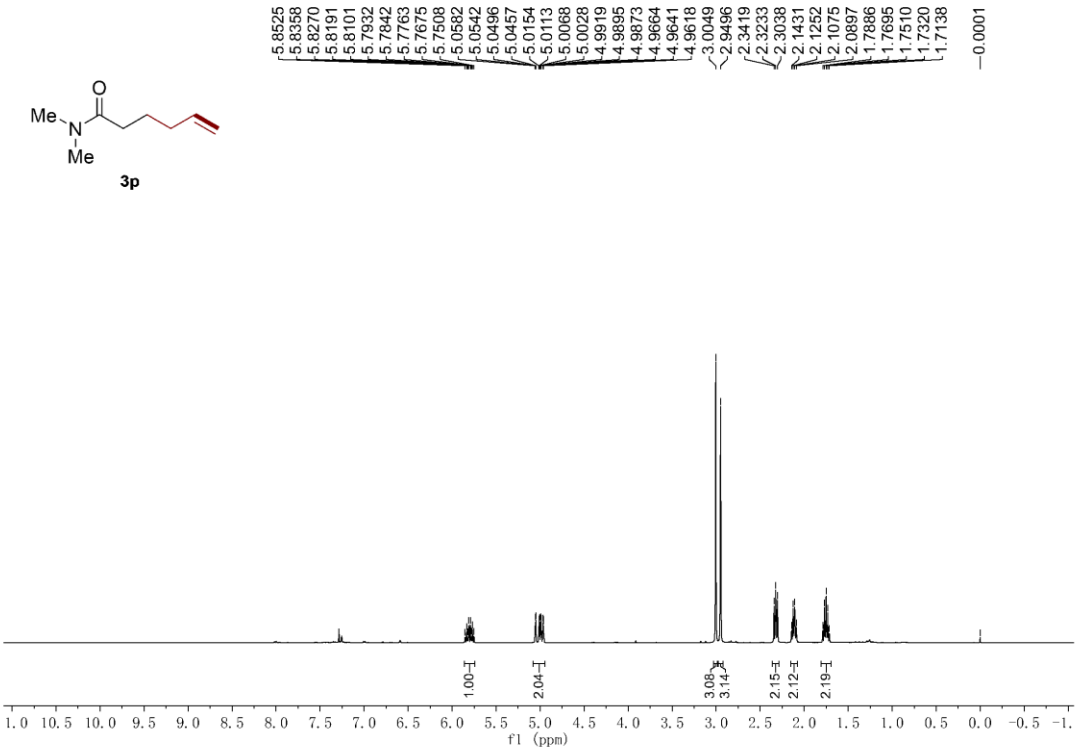
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3o



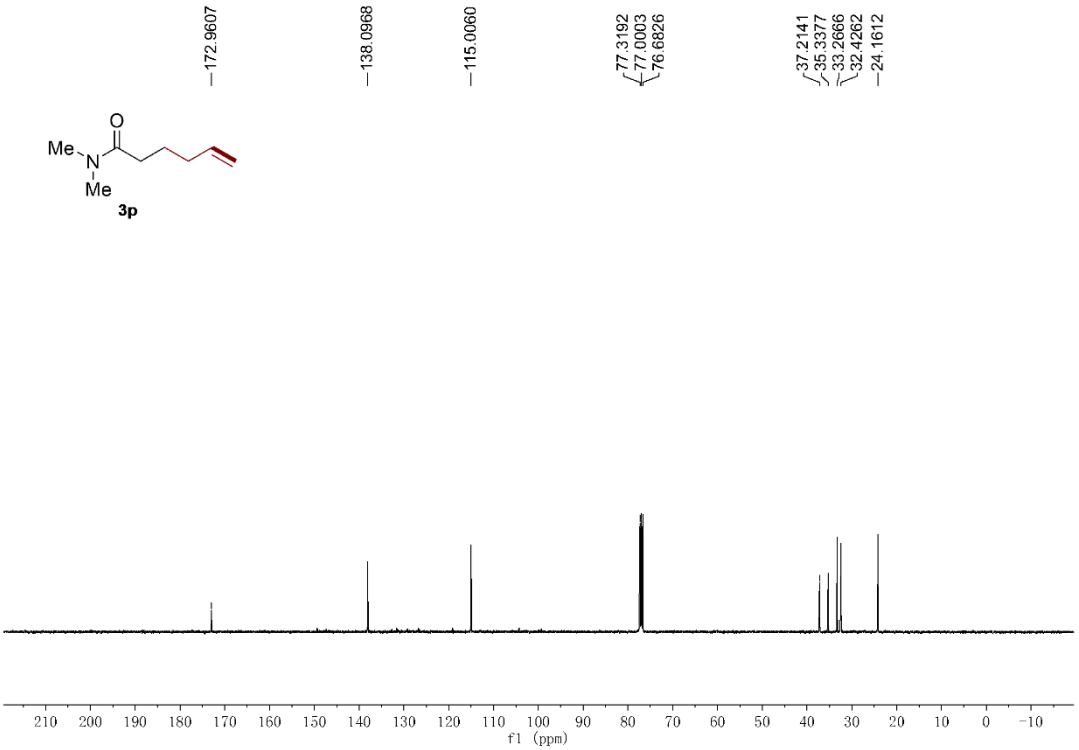
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3o



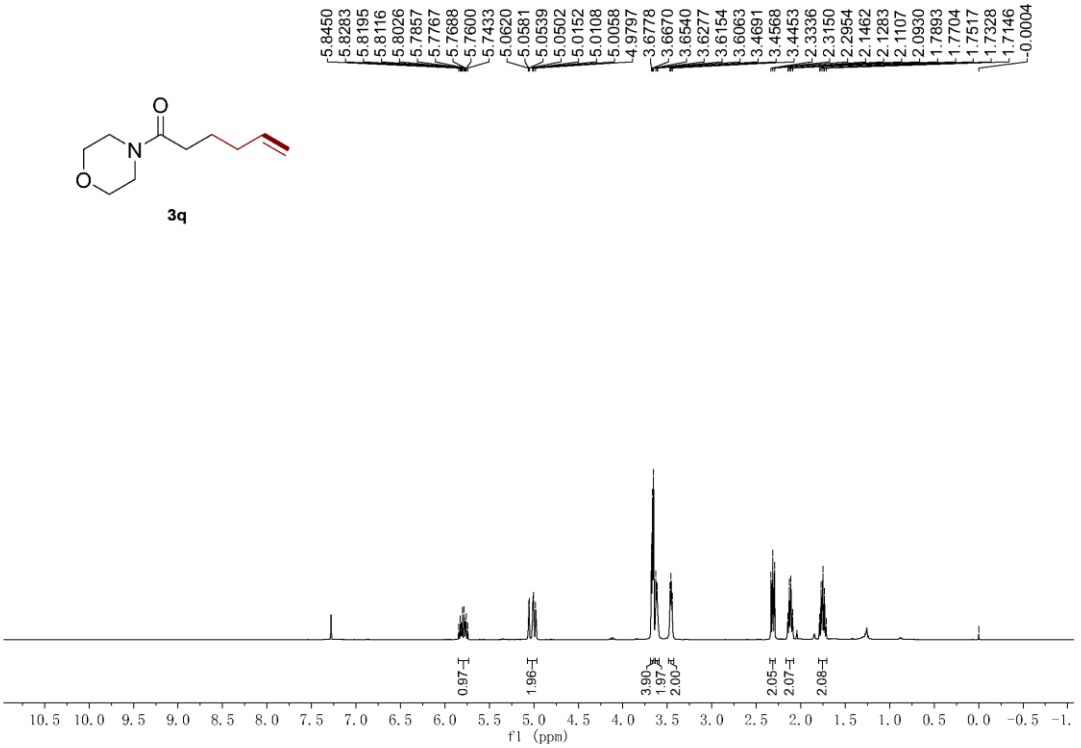
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3p



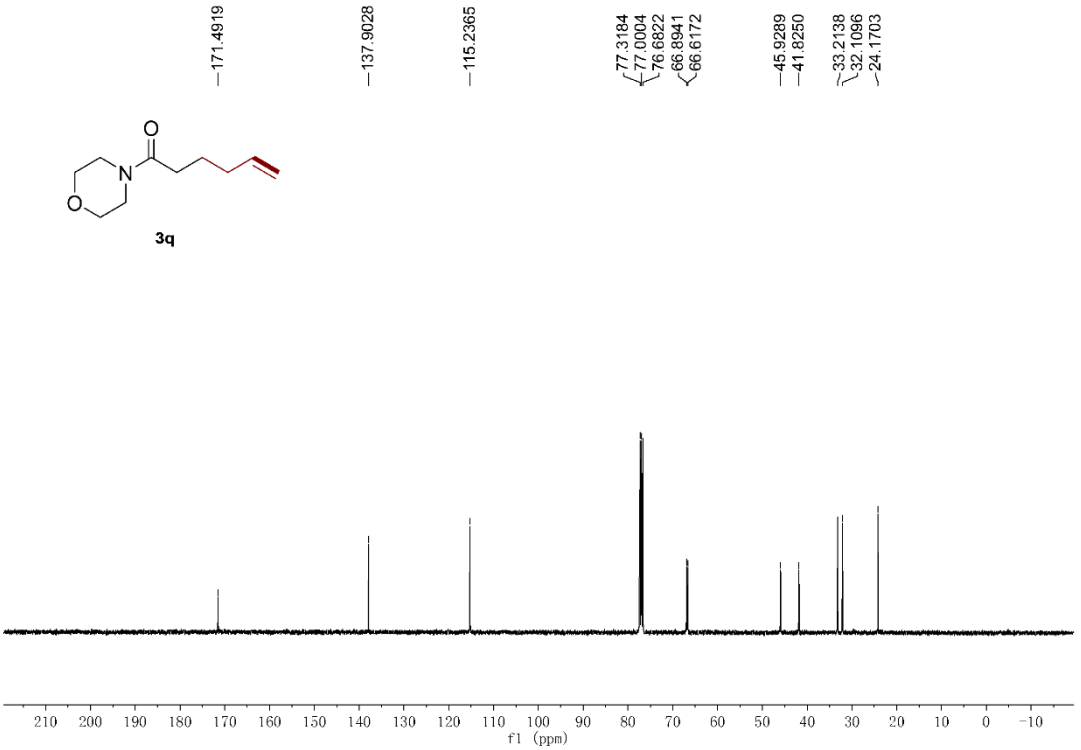
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3p



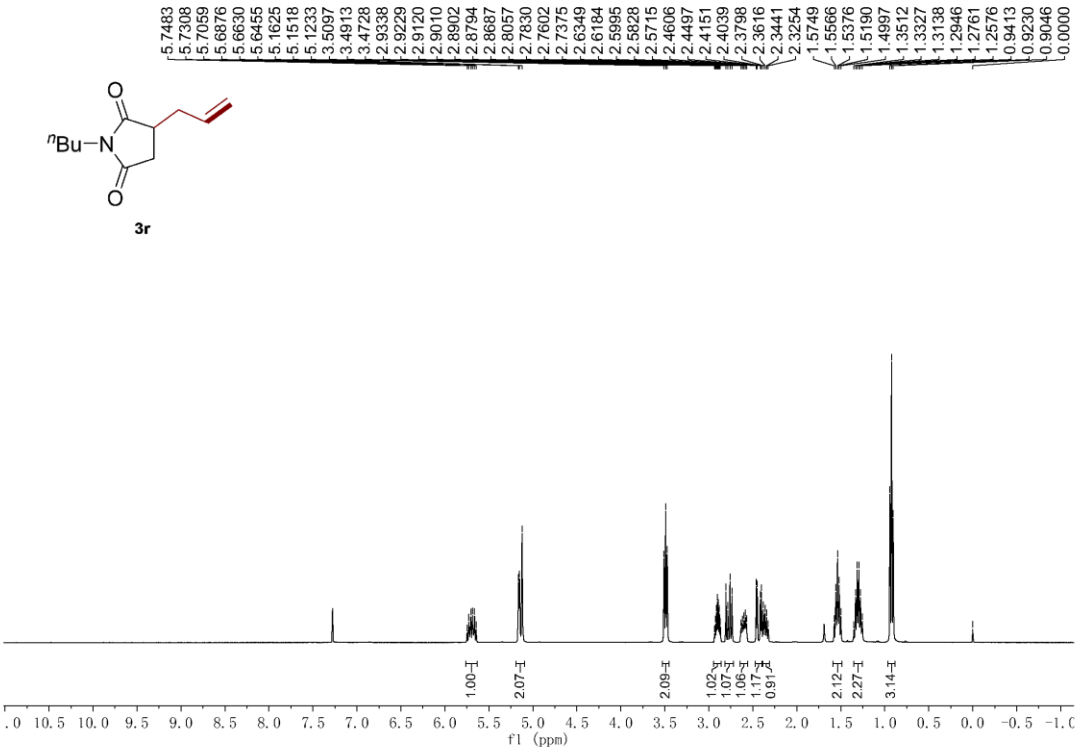
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3q



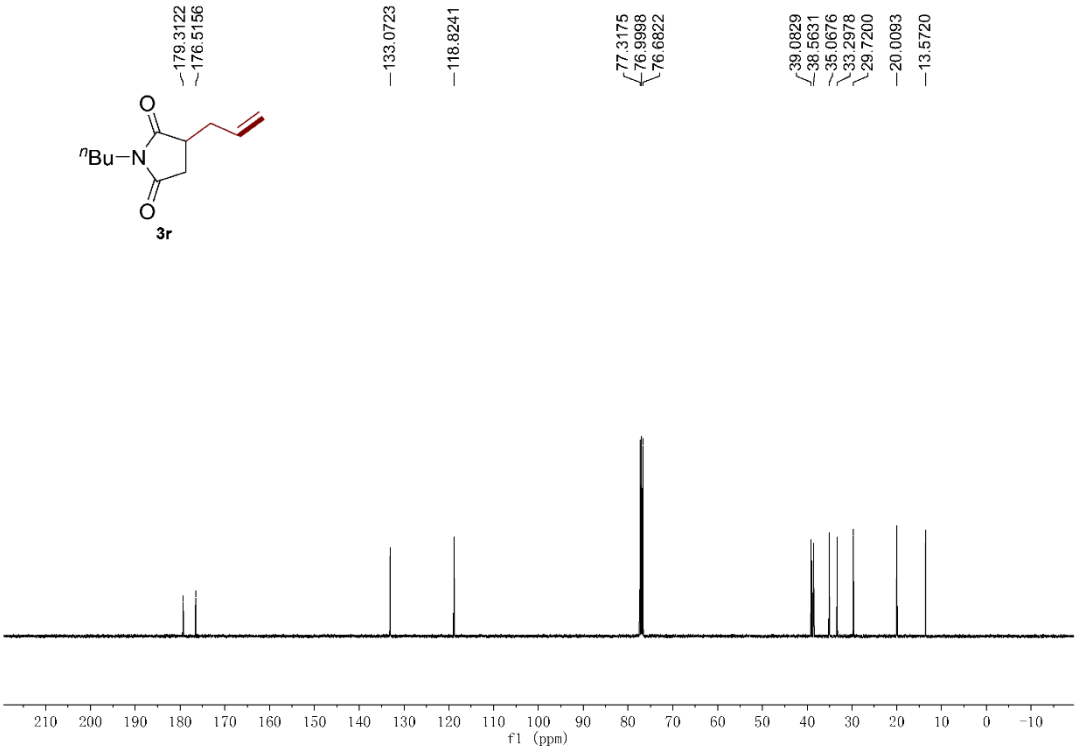
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3q



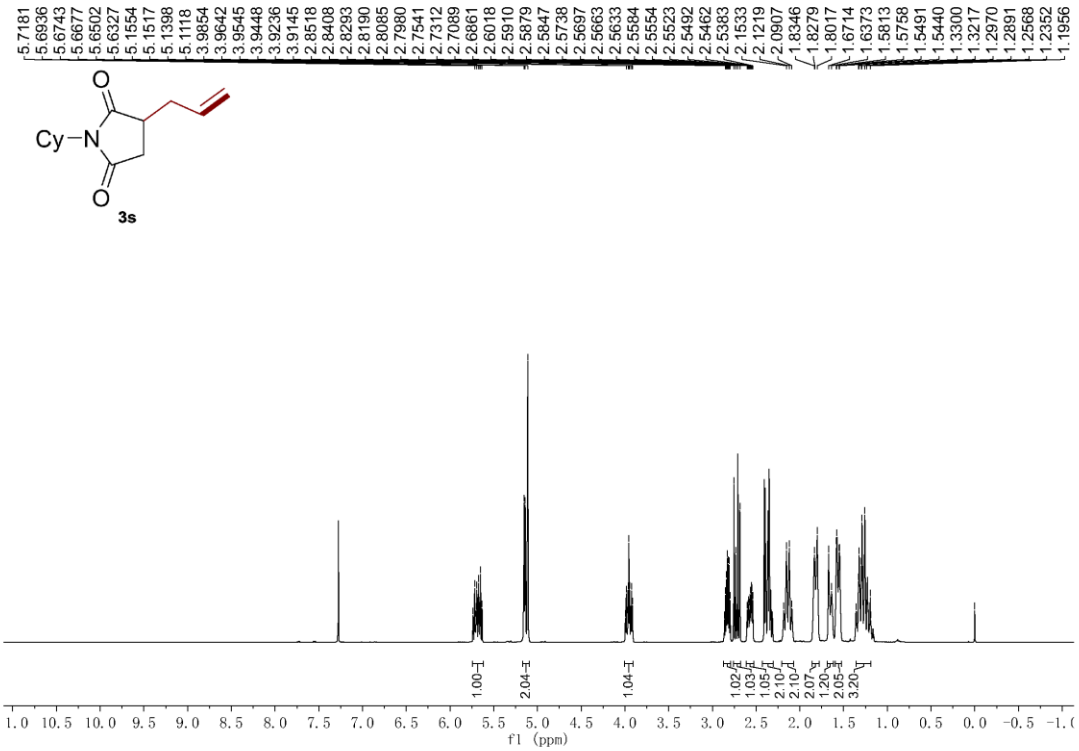
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3r



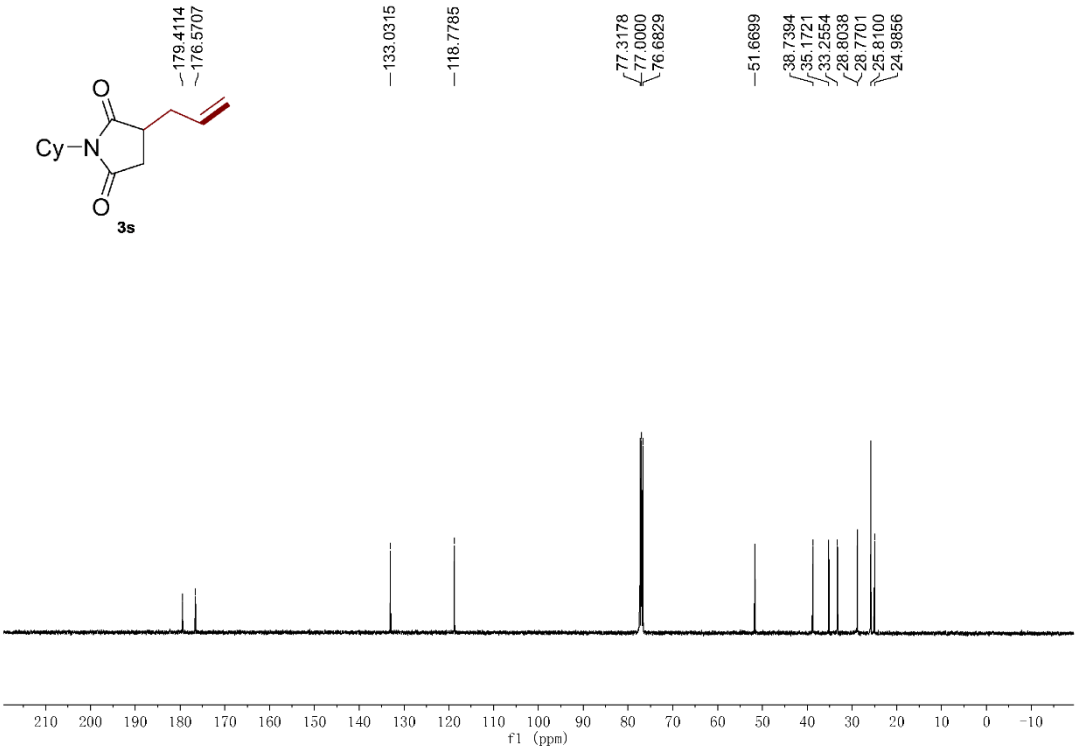
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3r



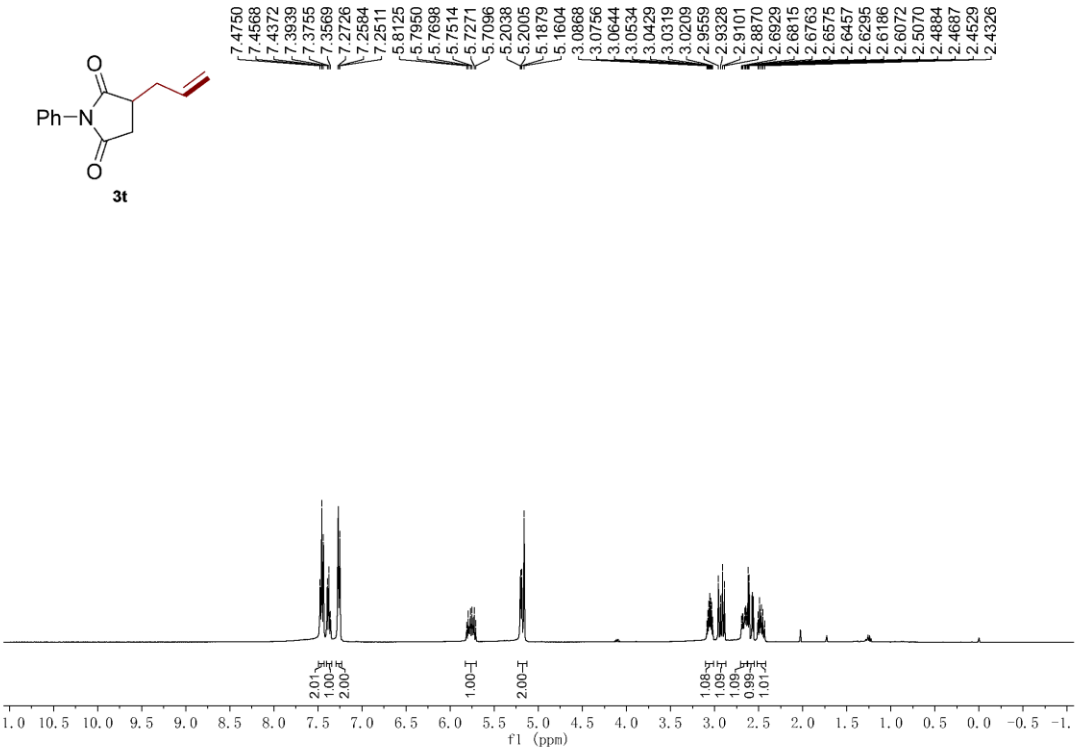
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3s



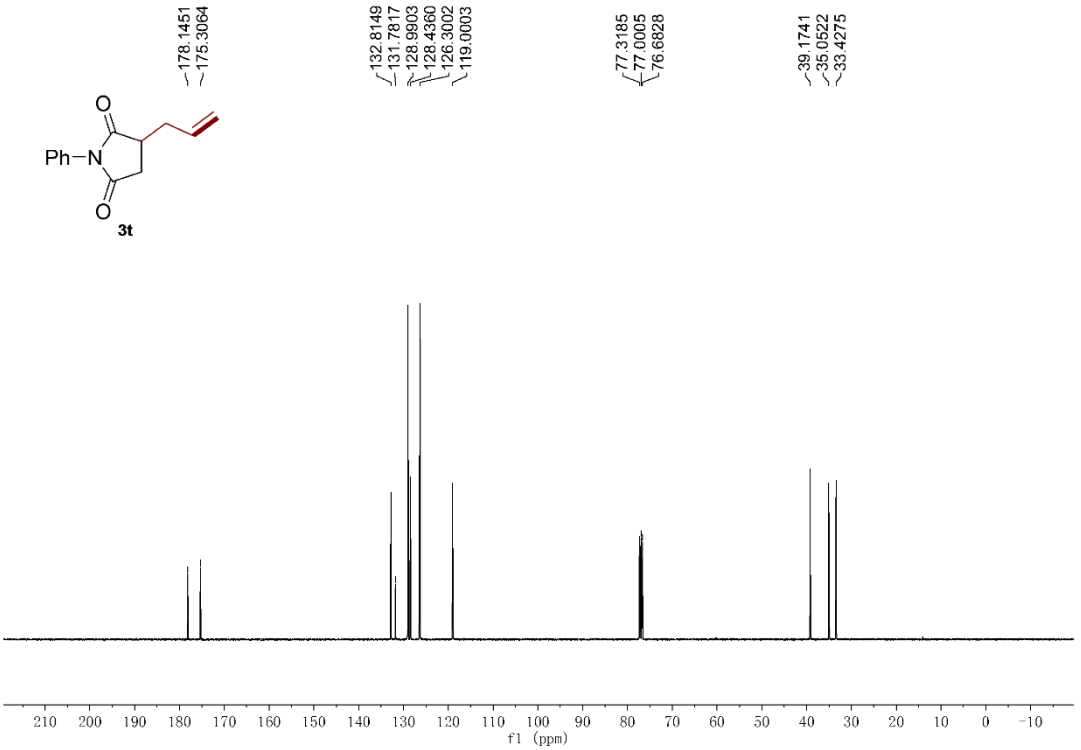
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3s



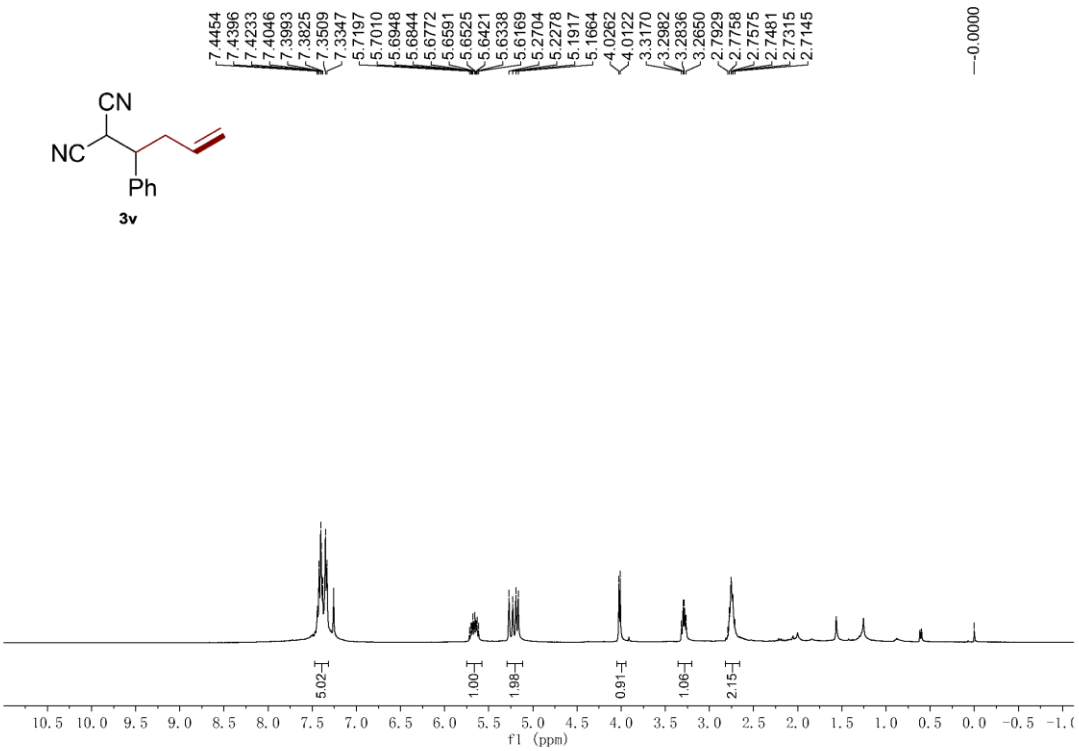
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3t



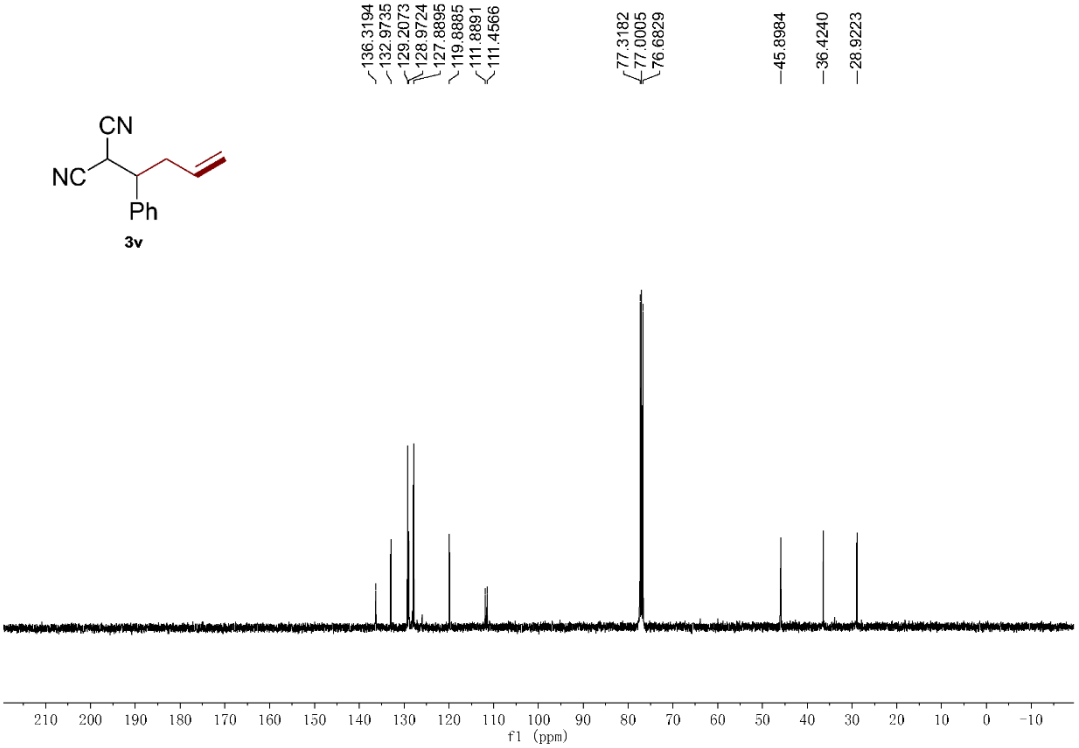
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3t



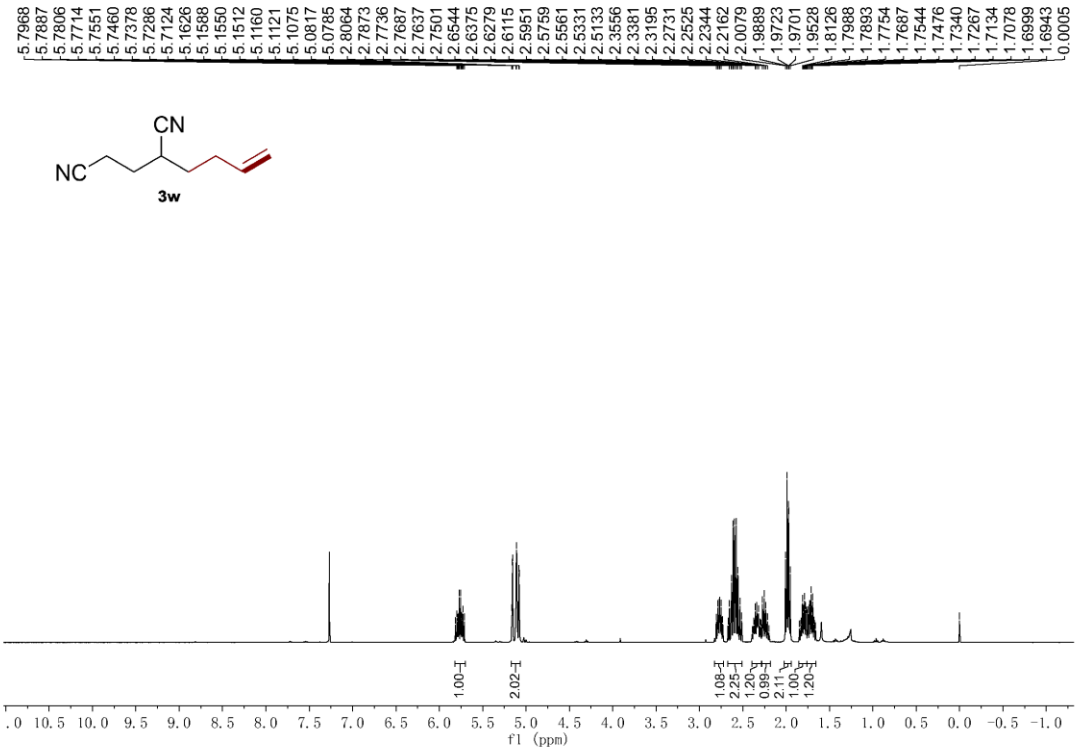
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3v



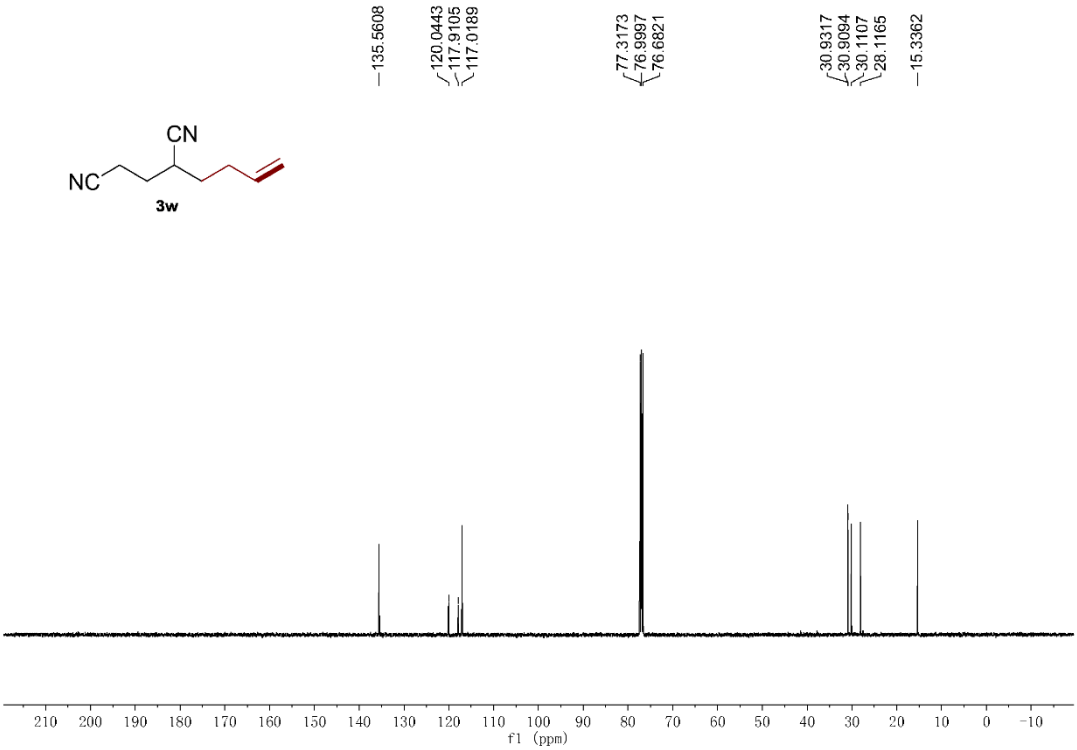
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3v



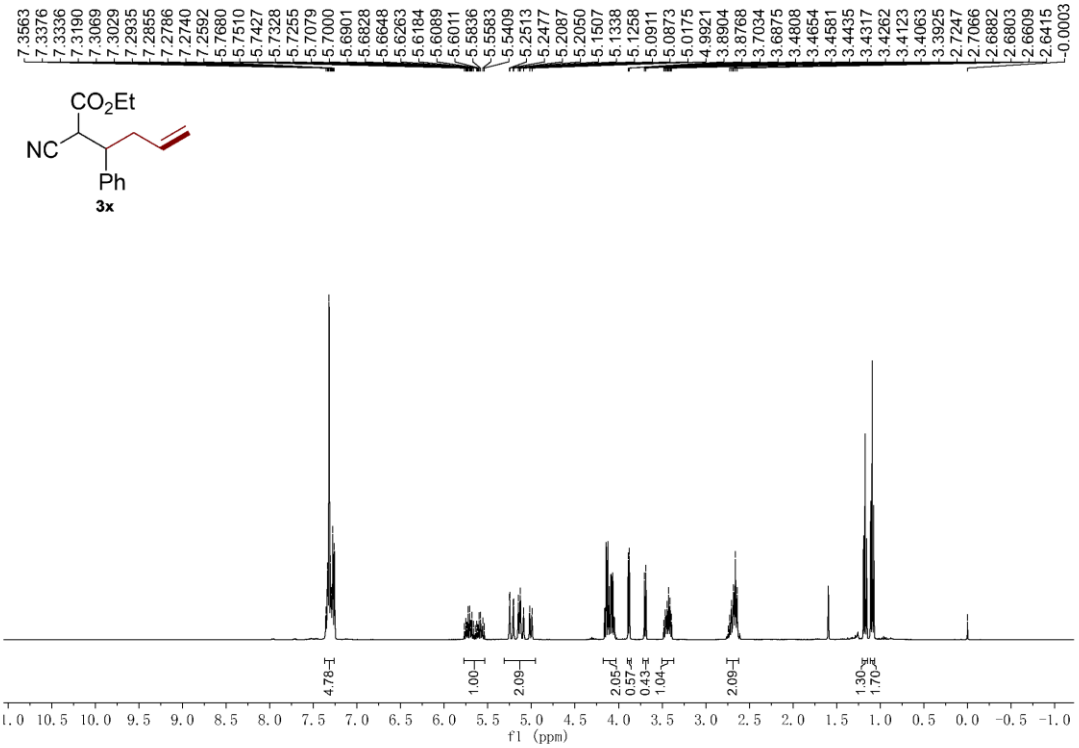
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3w



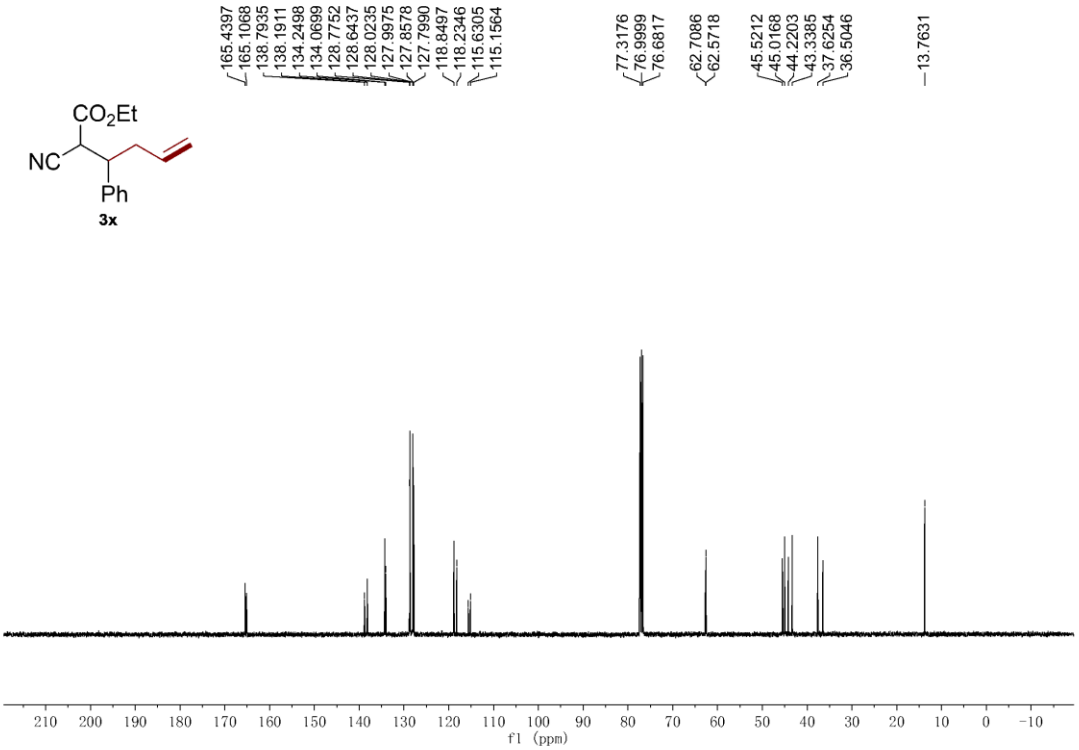
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3w



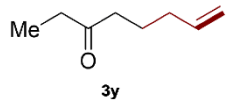
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3x



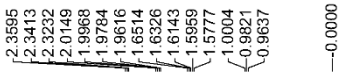
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3x

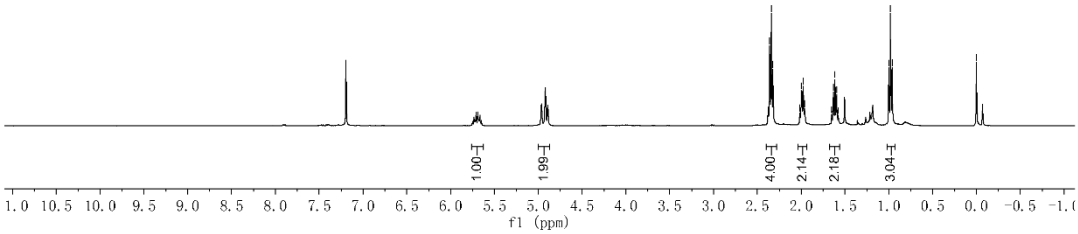


^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3y



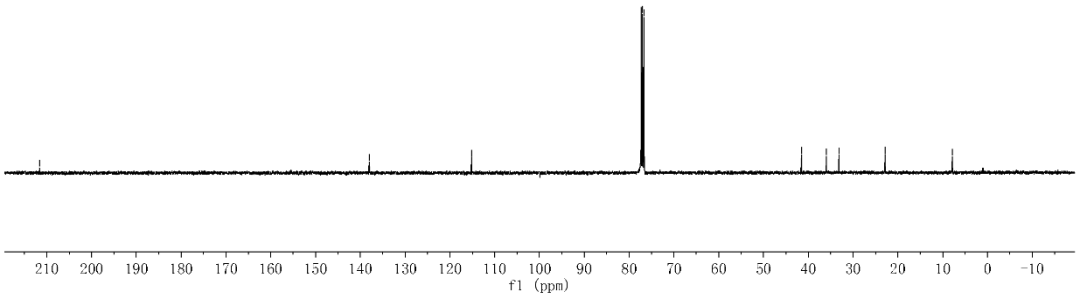
3y



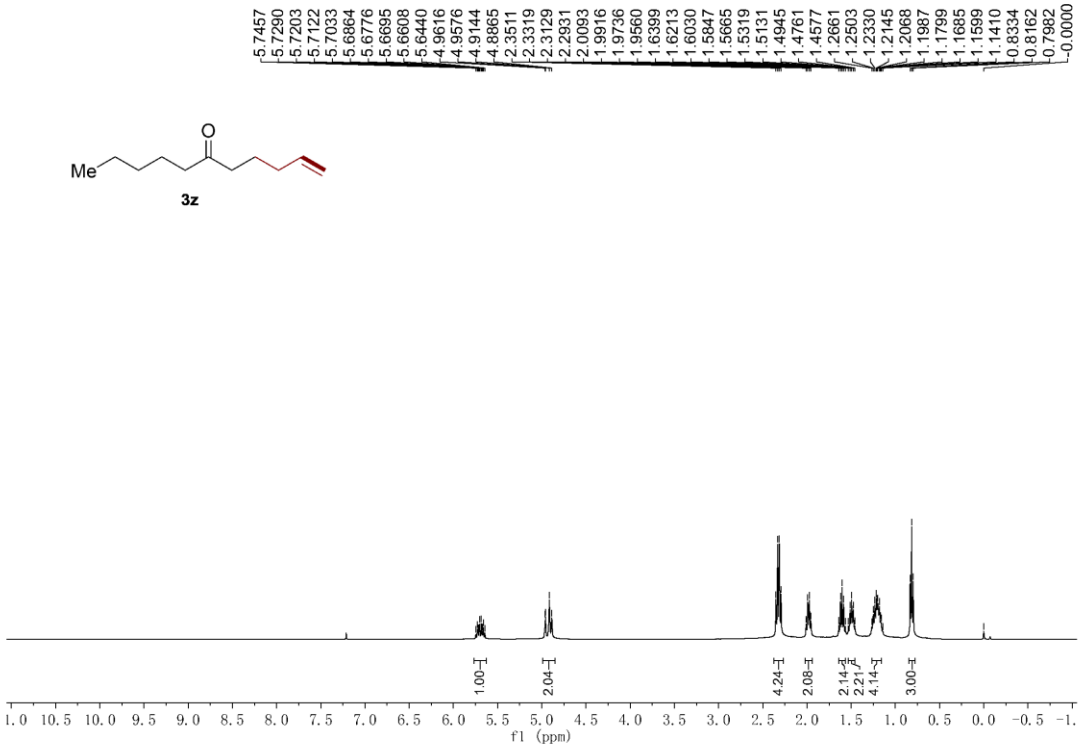


^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3y

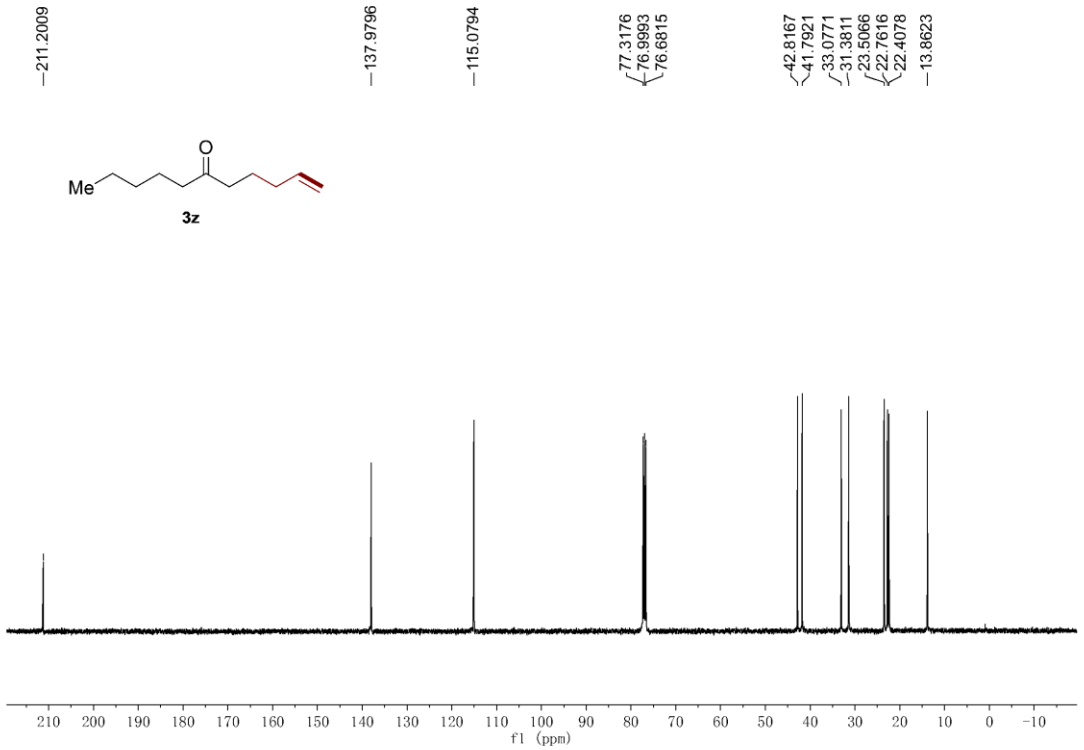
3y



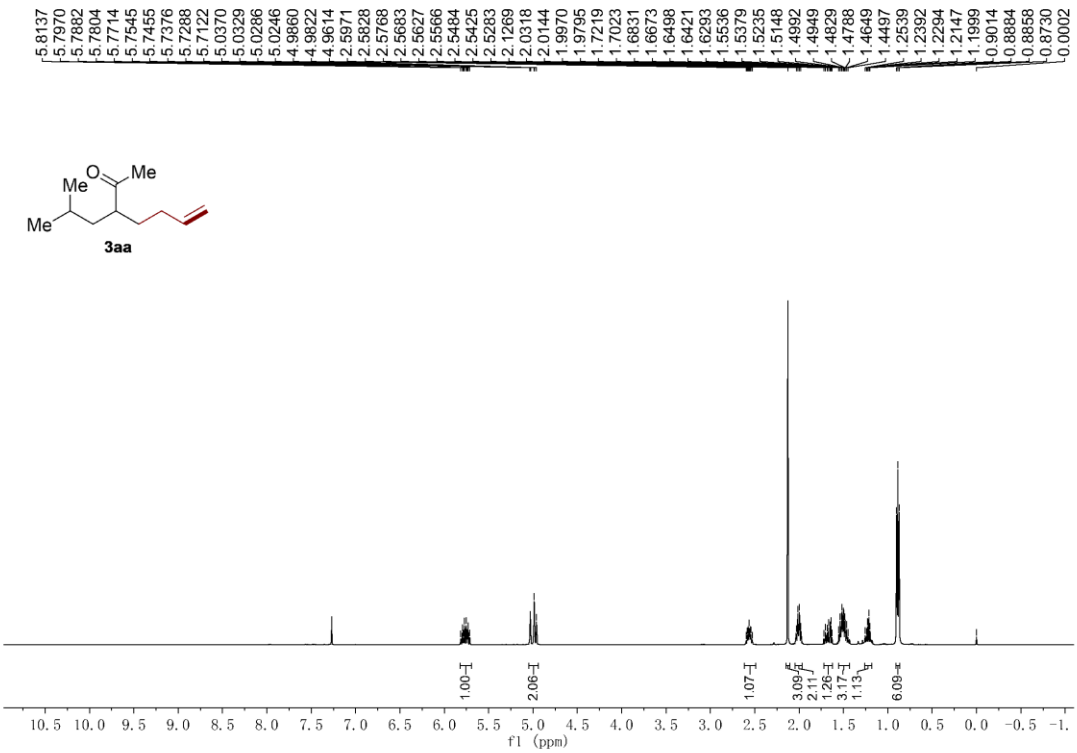
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3z



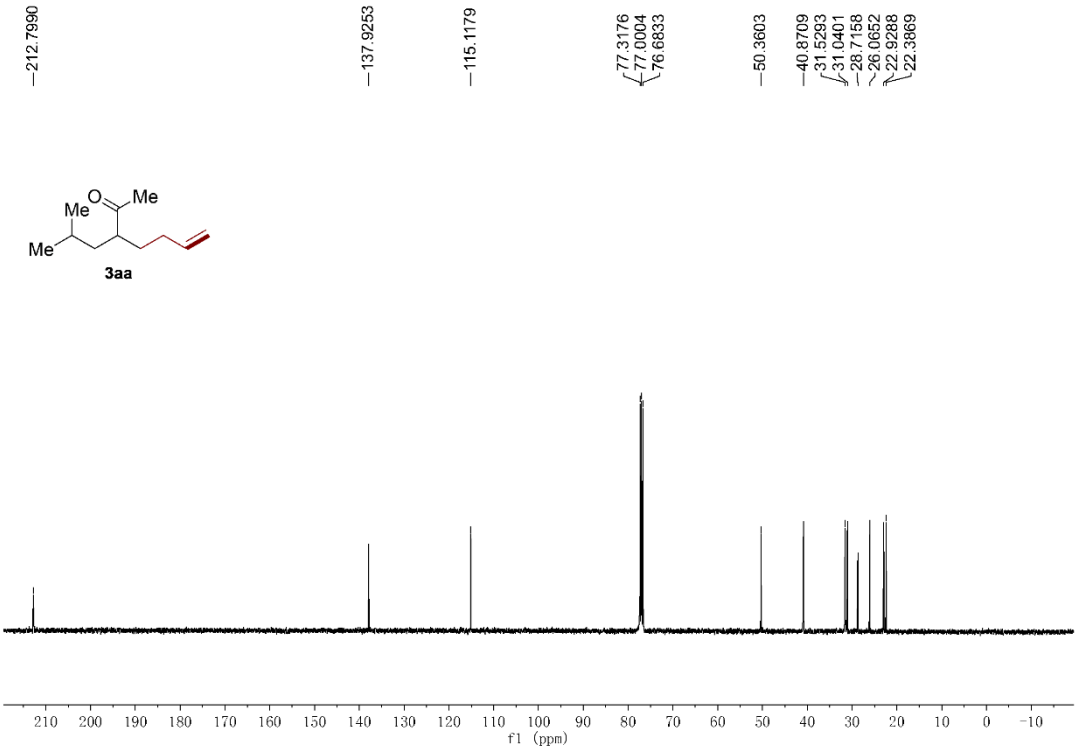
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3z



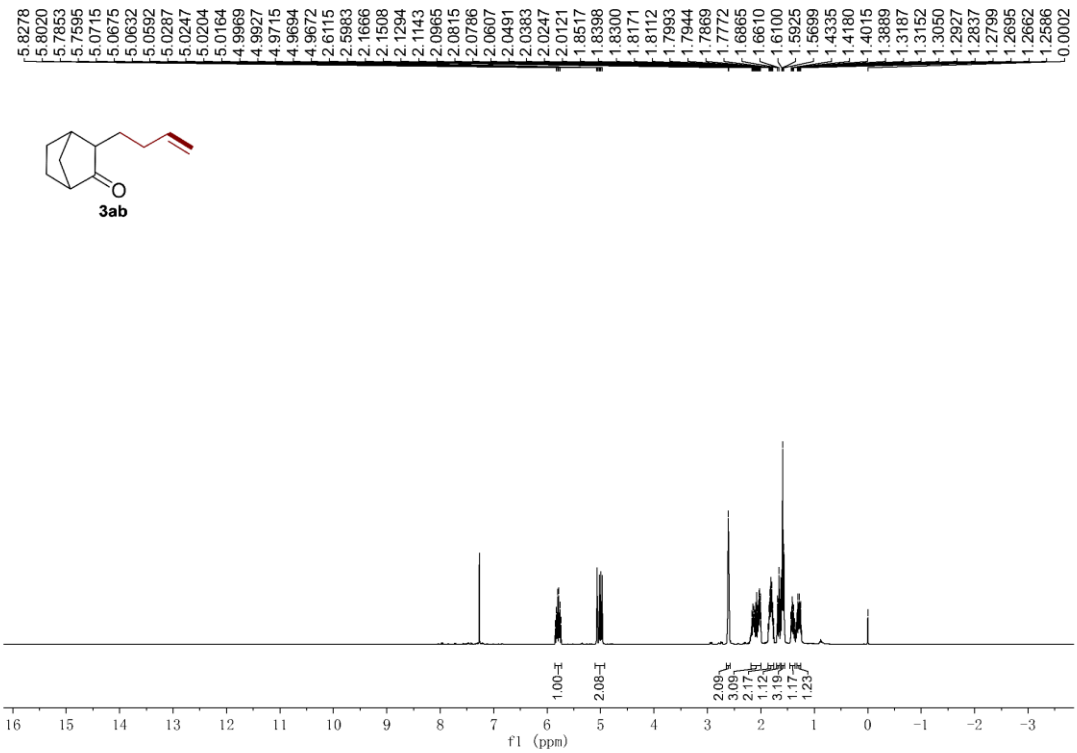
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3aa



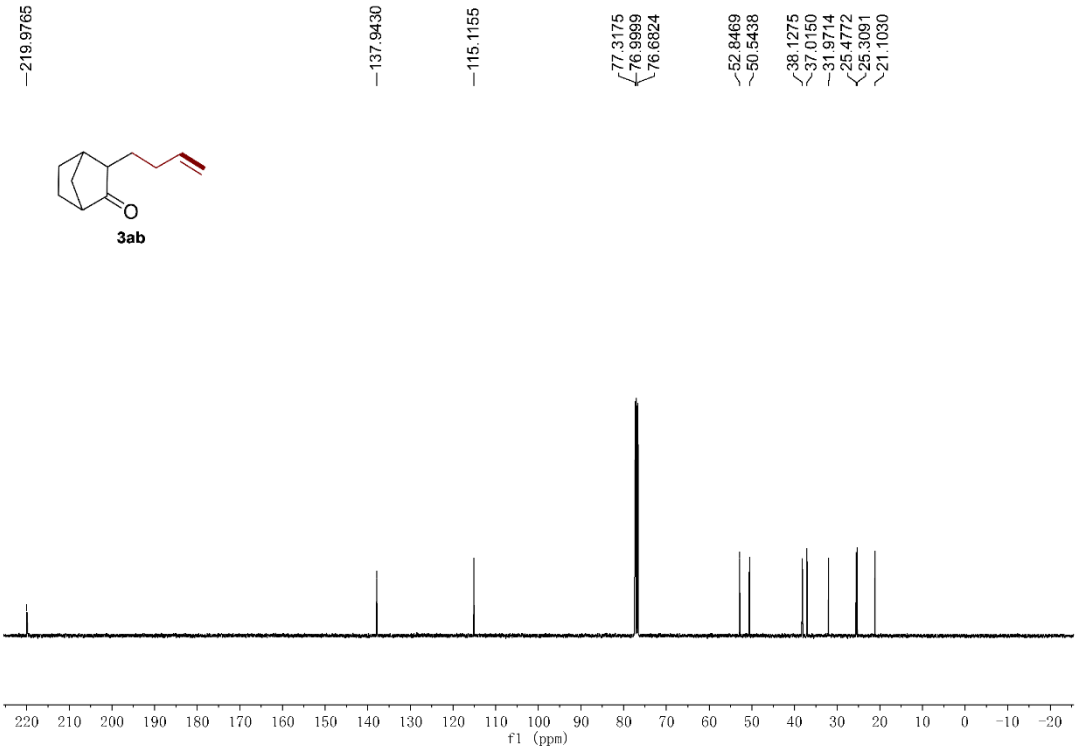
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3aa



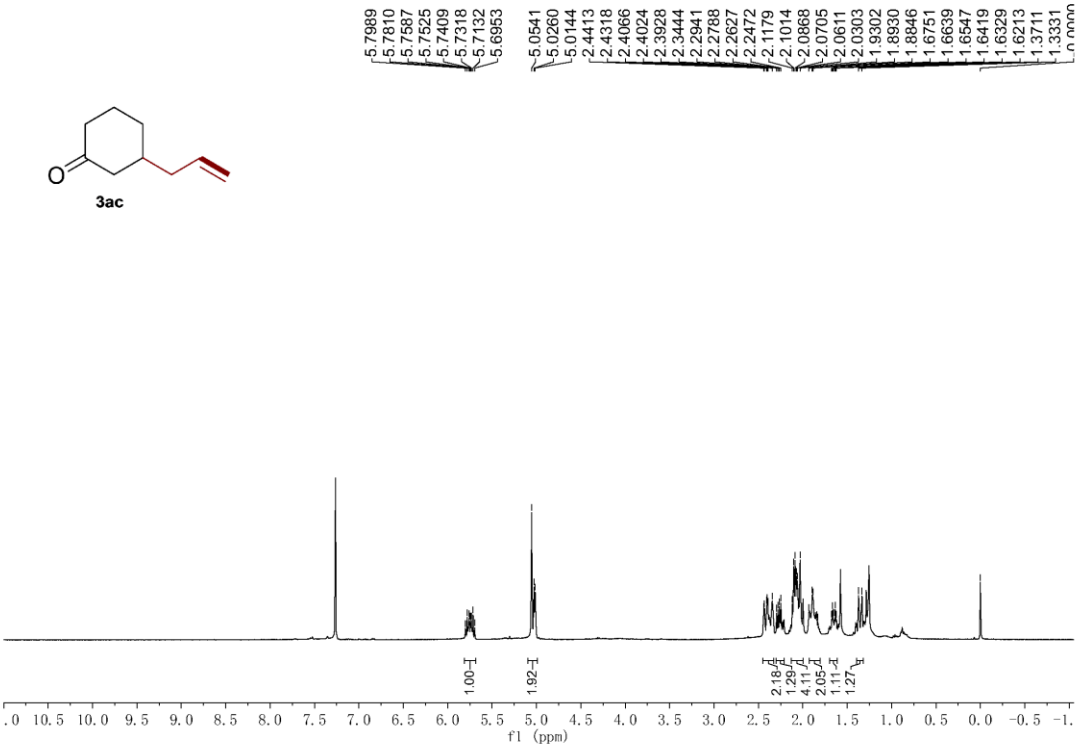
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ab



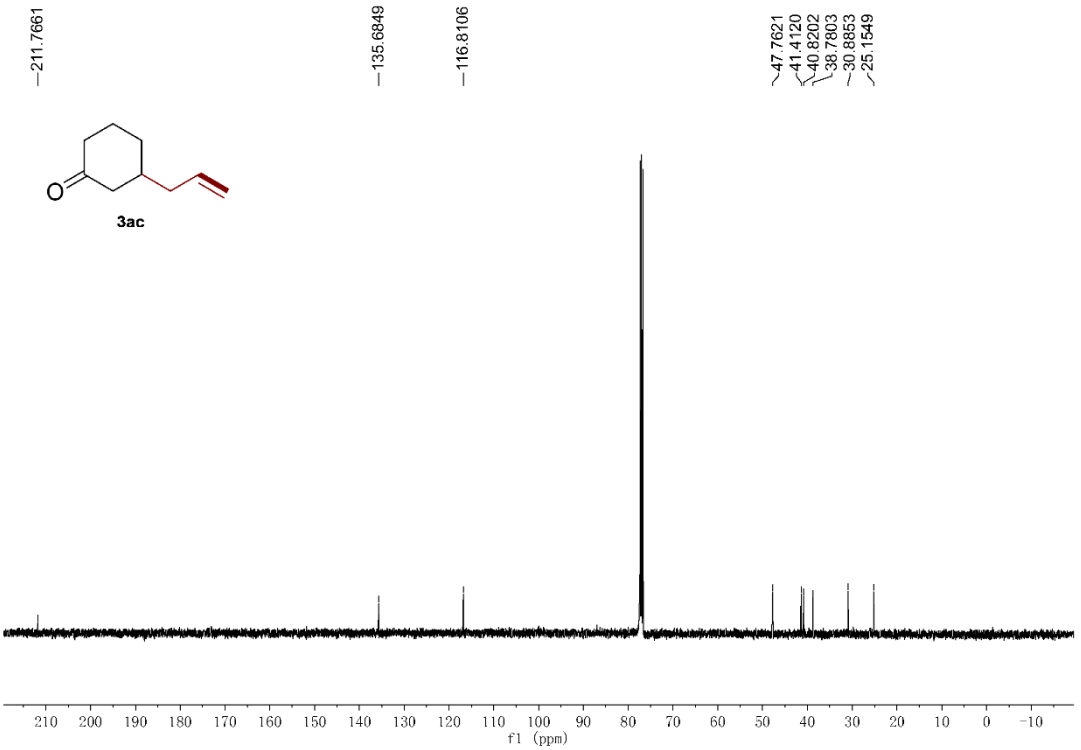
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3ab



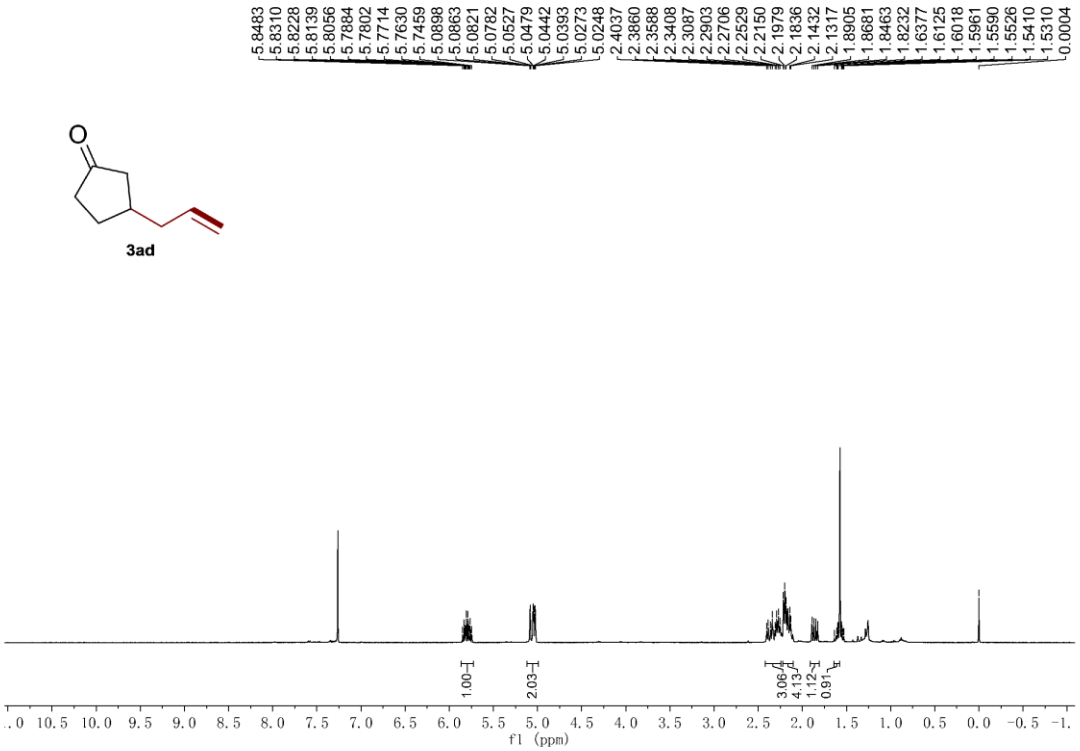
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ac



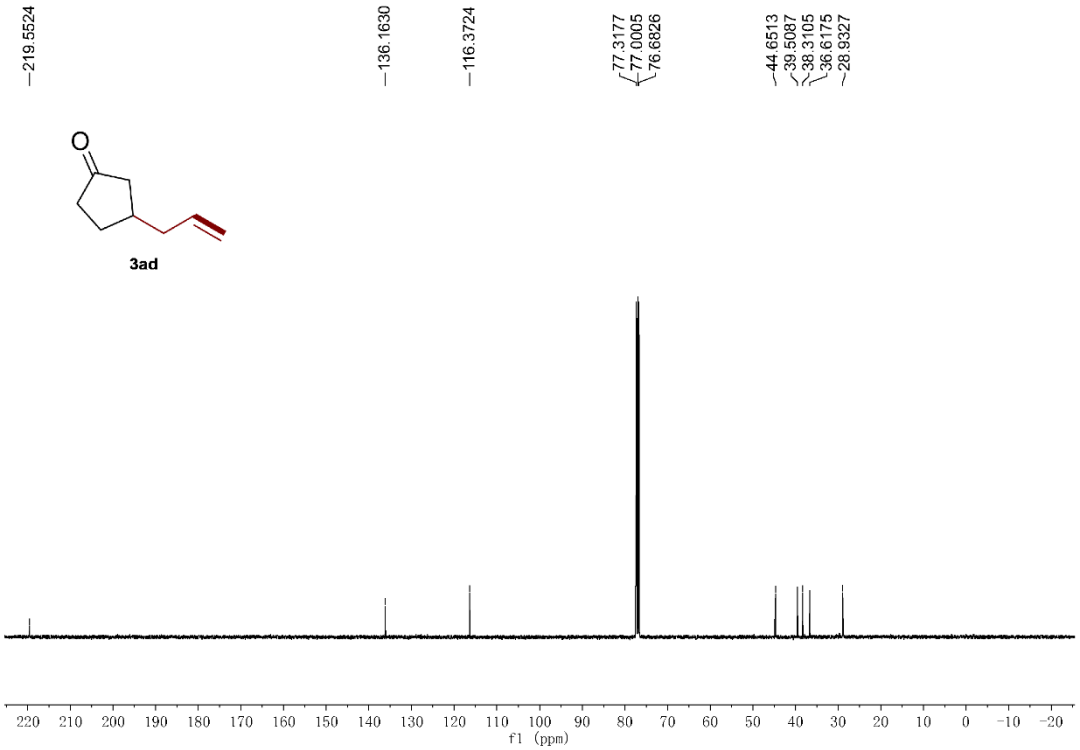
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3ac



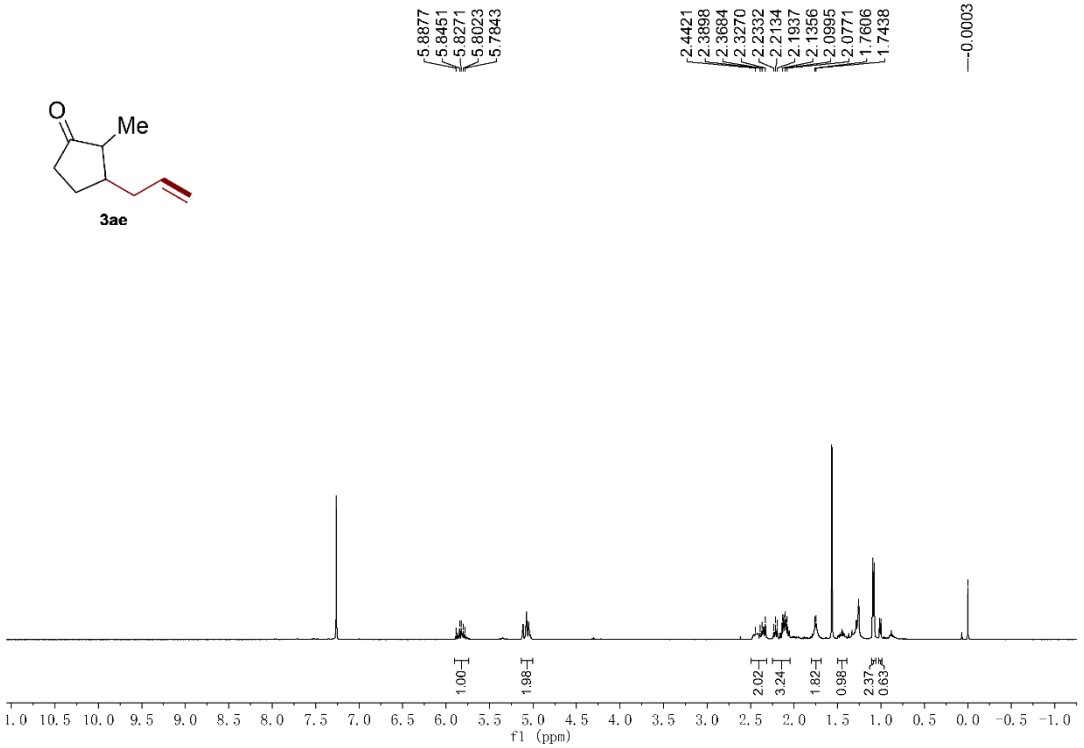
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ad



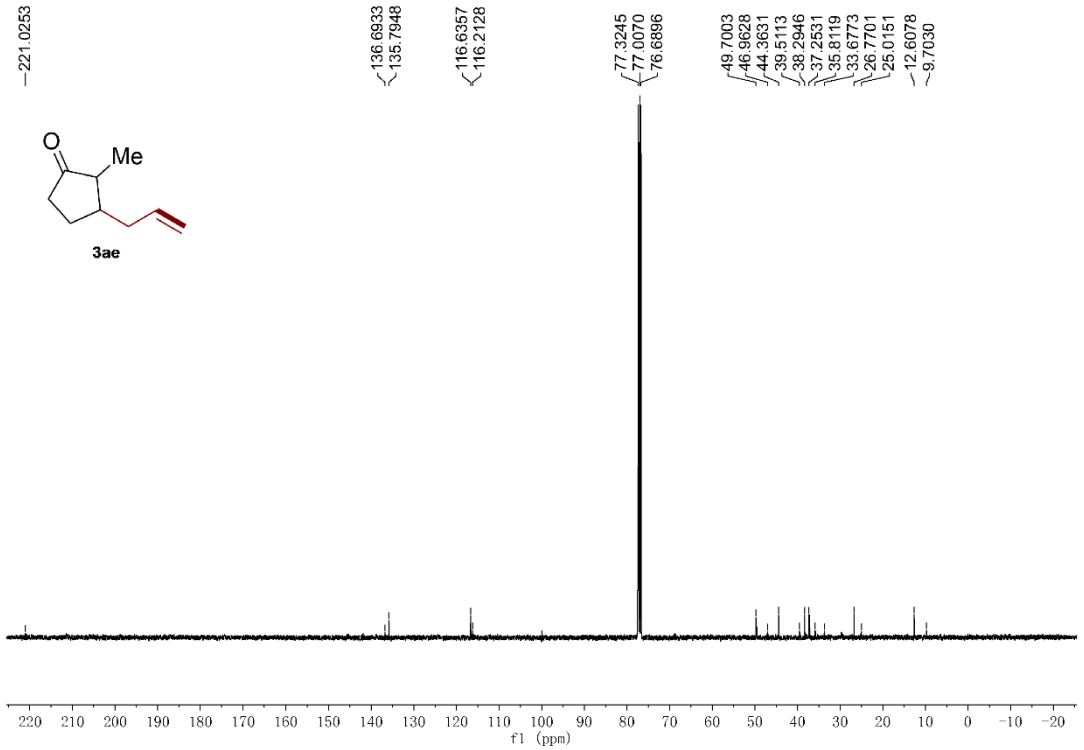
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3ad



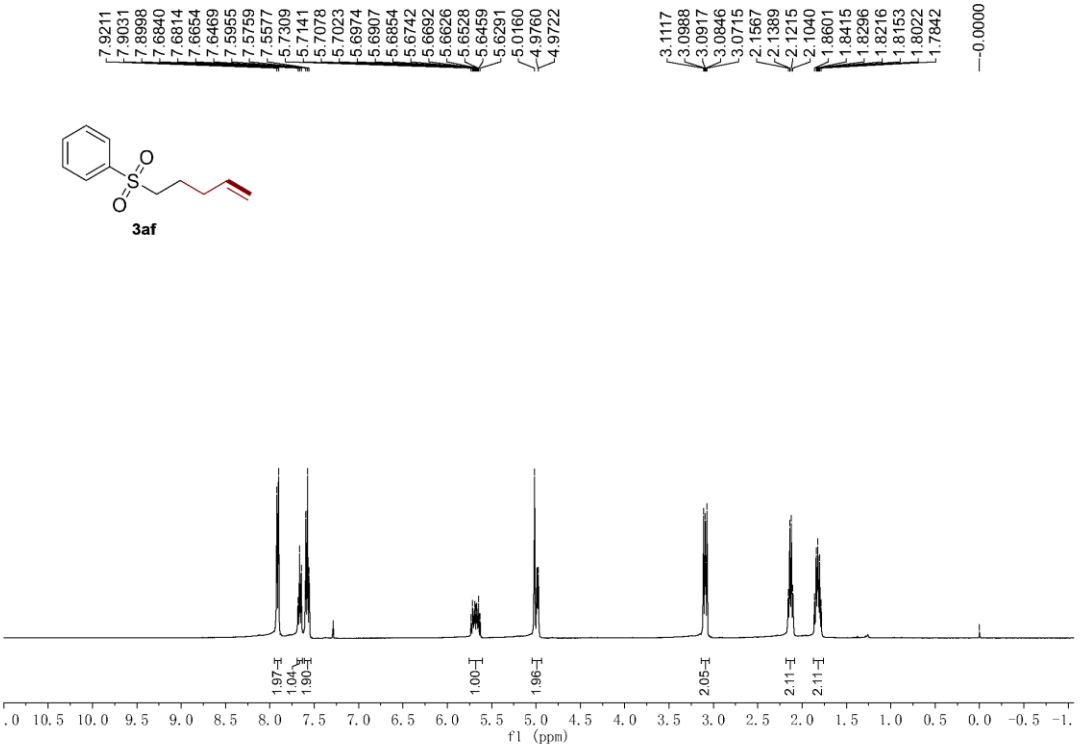
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ae



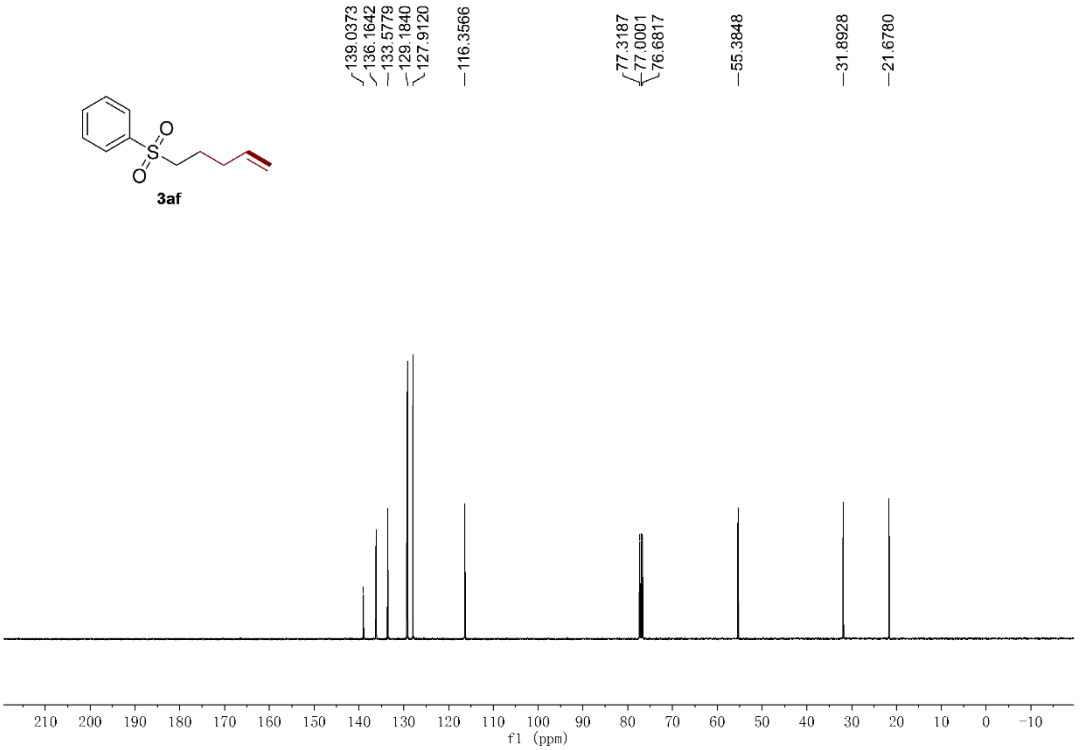
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3ae



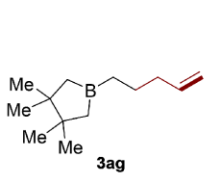
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3af



^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3af

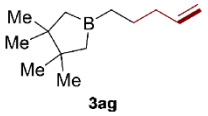


^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ag



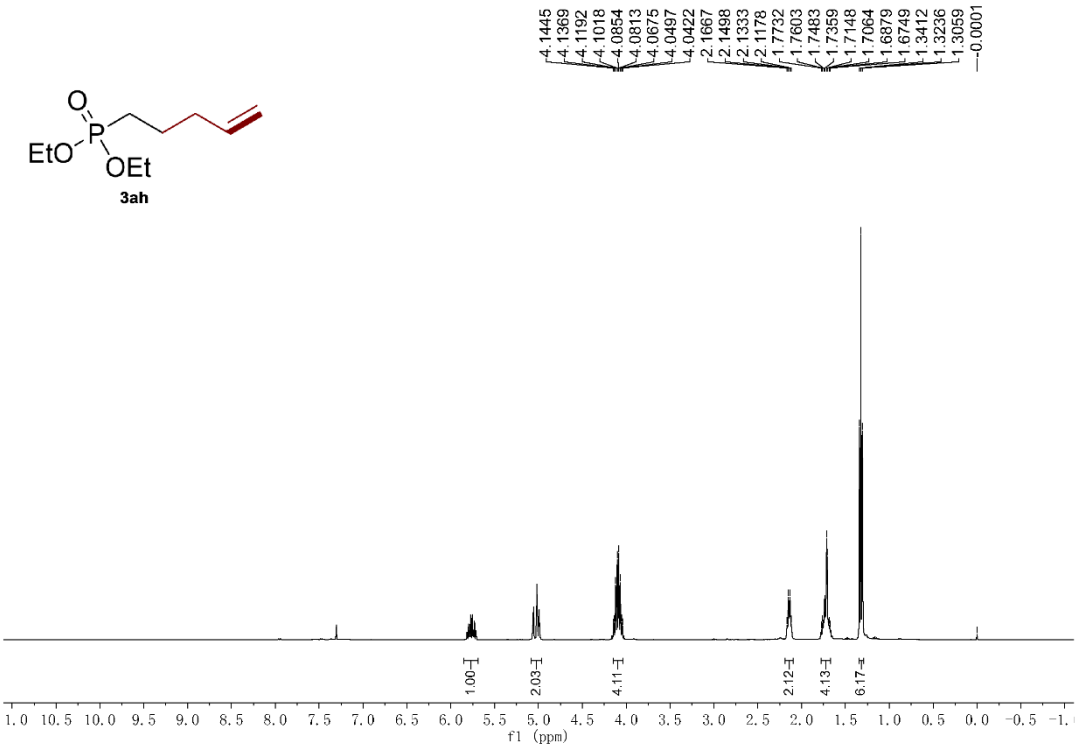
3ag

^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3ag

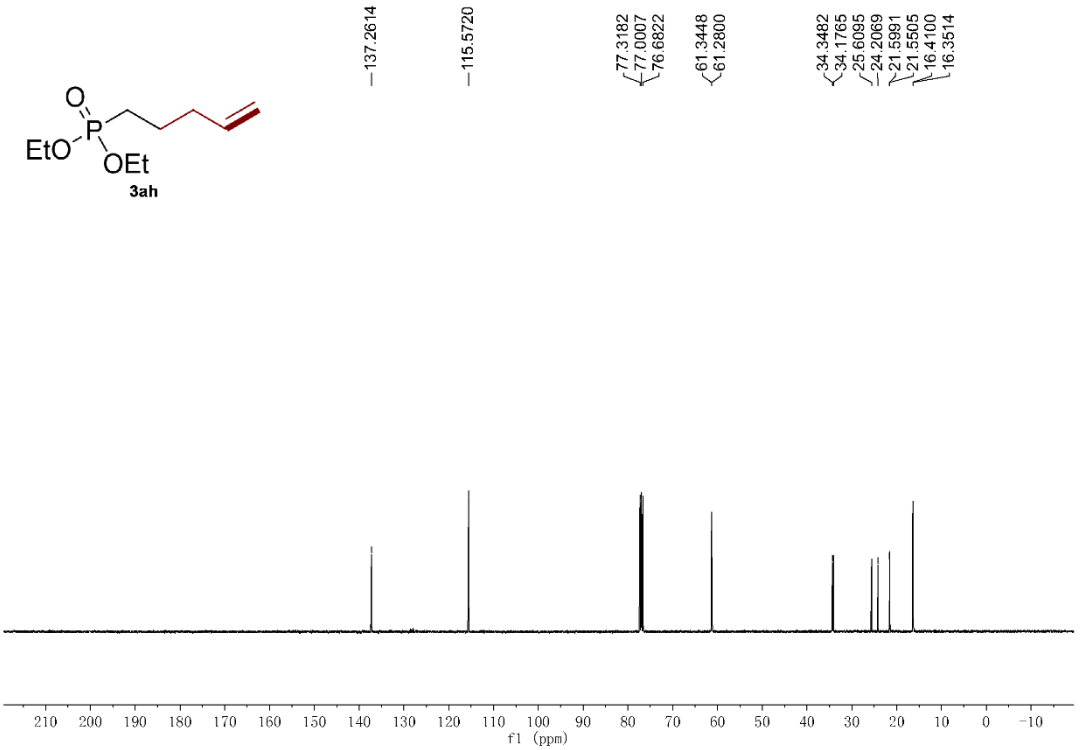


3ag

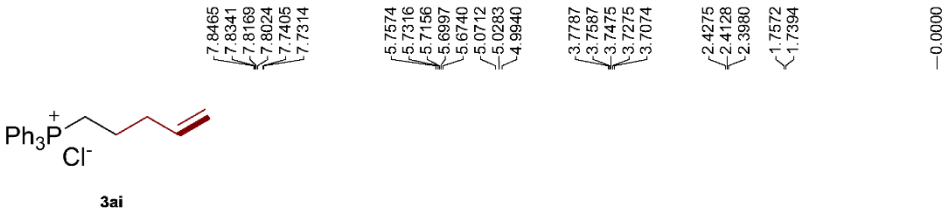
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ah



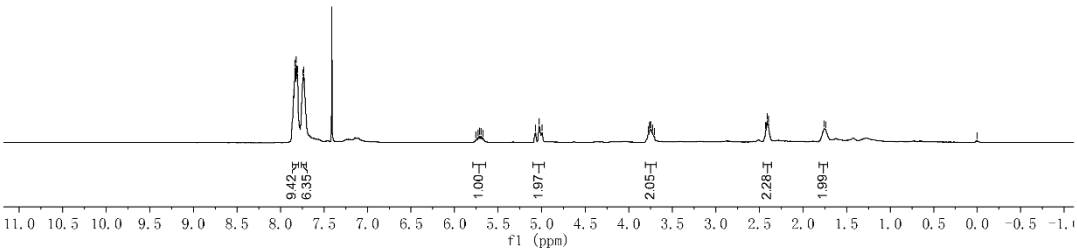
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3ah



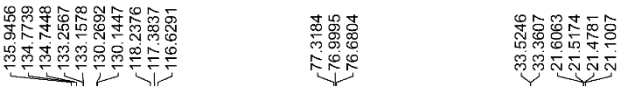
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ai

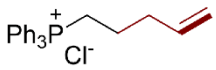


3ai

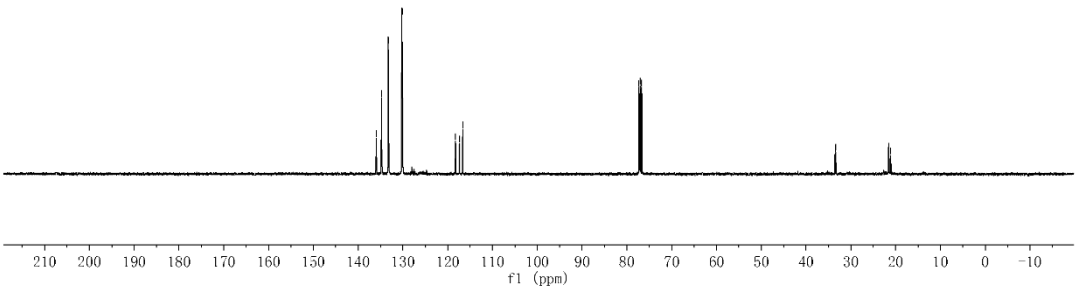


^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3ai

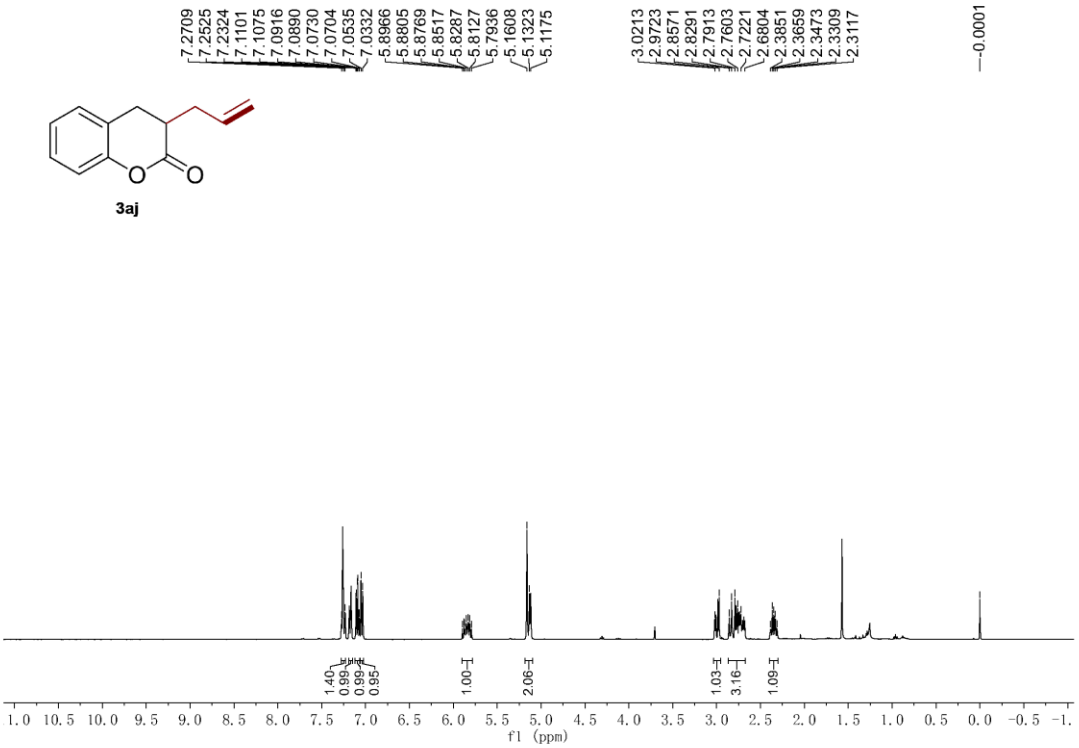




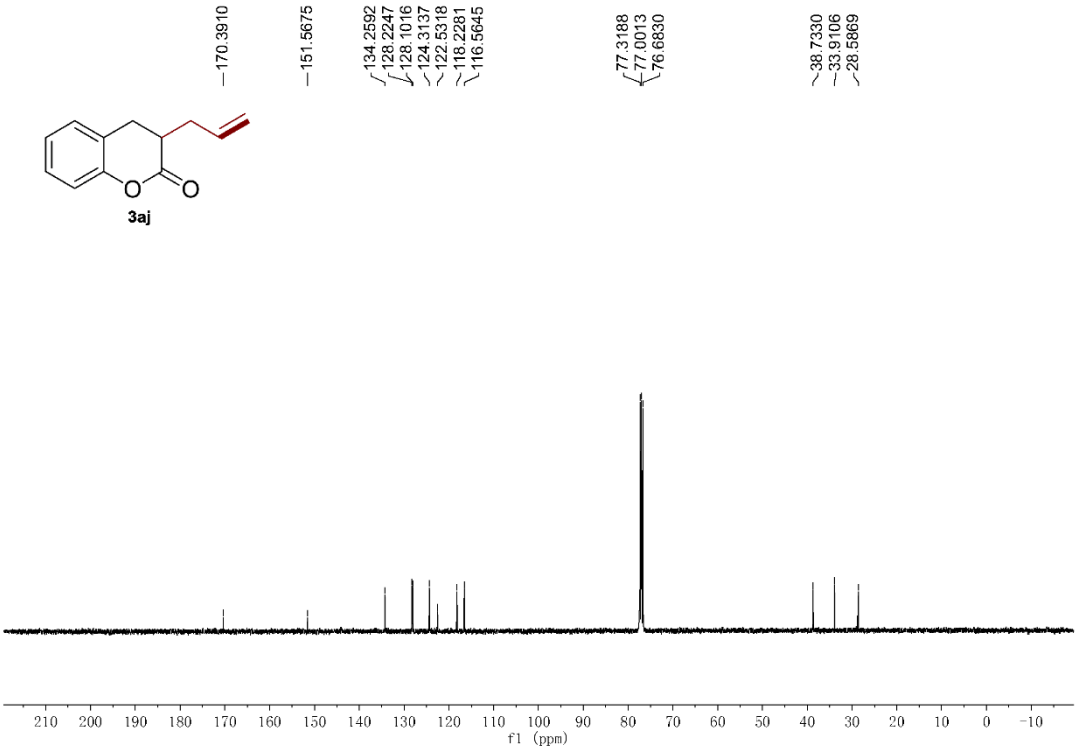
3ai



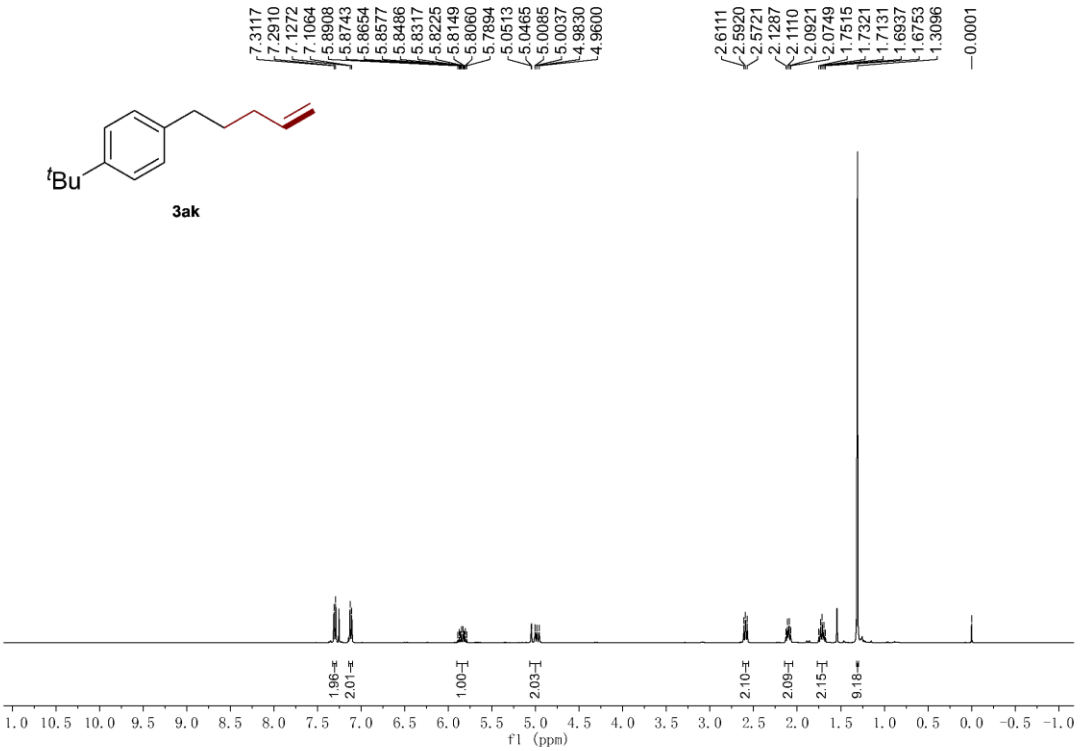
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3aj



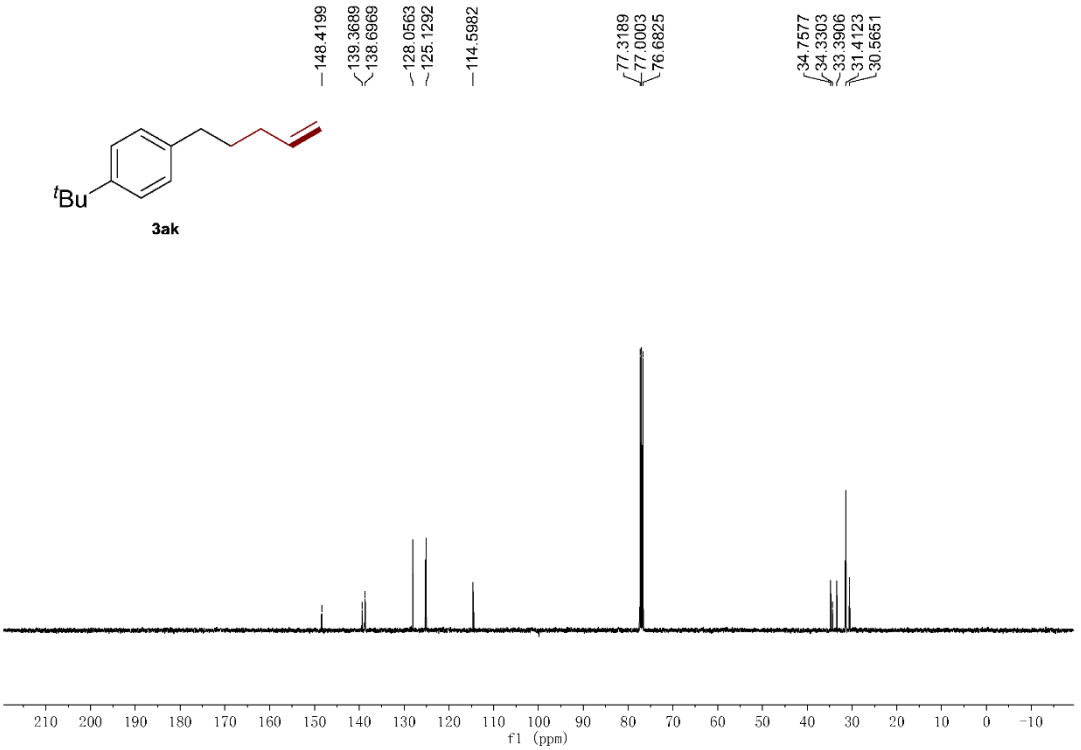
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3aj



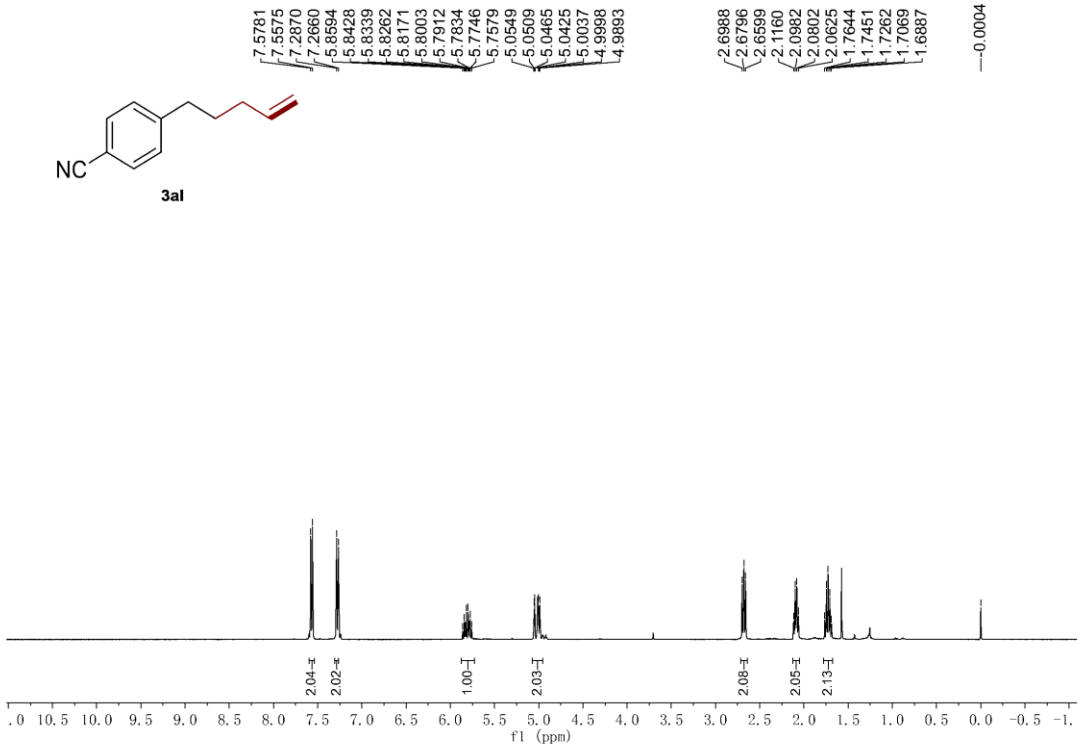
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ak



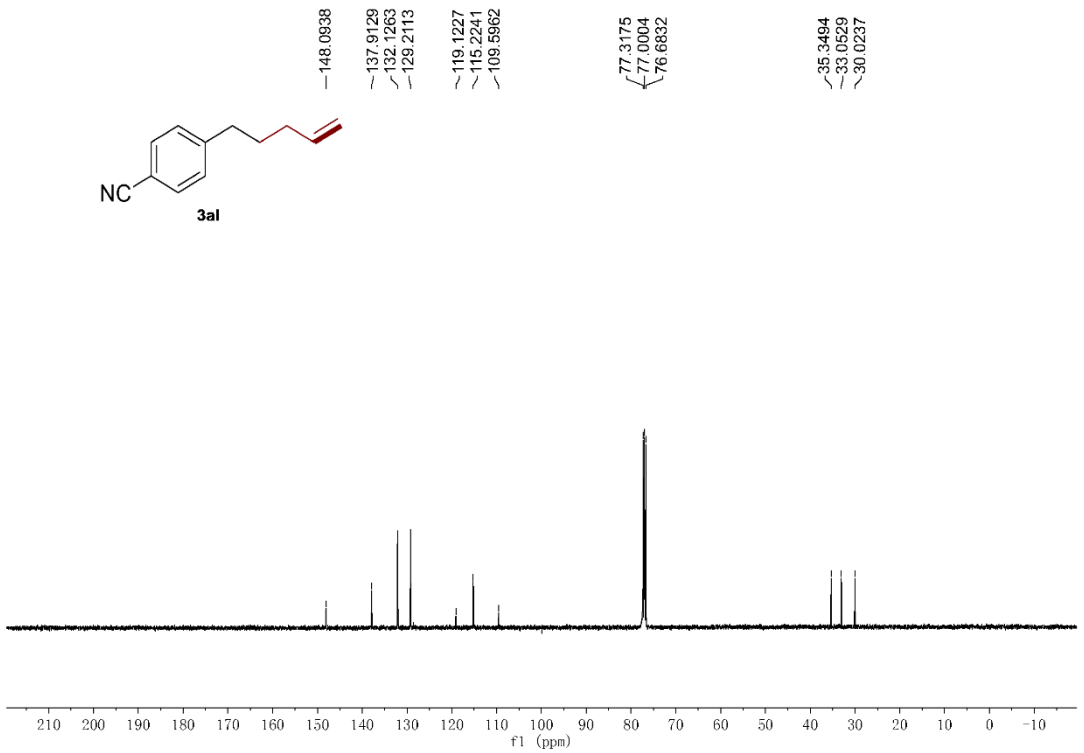
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3ak



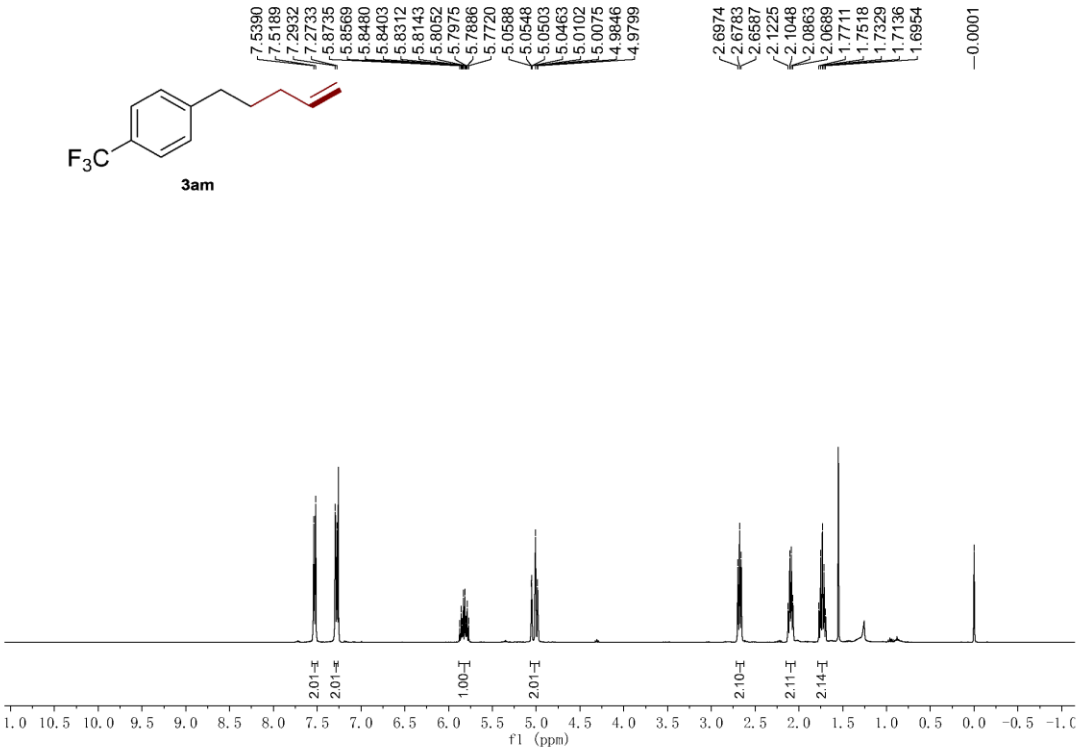
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3al



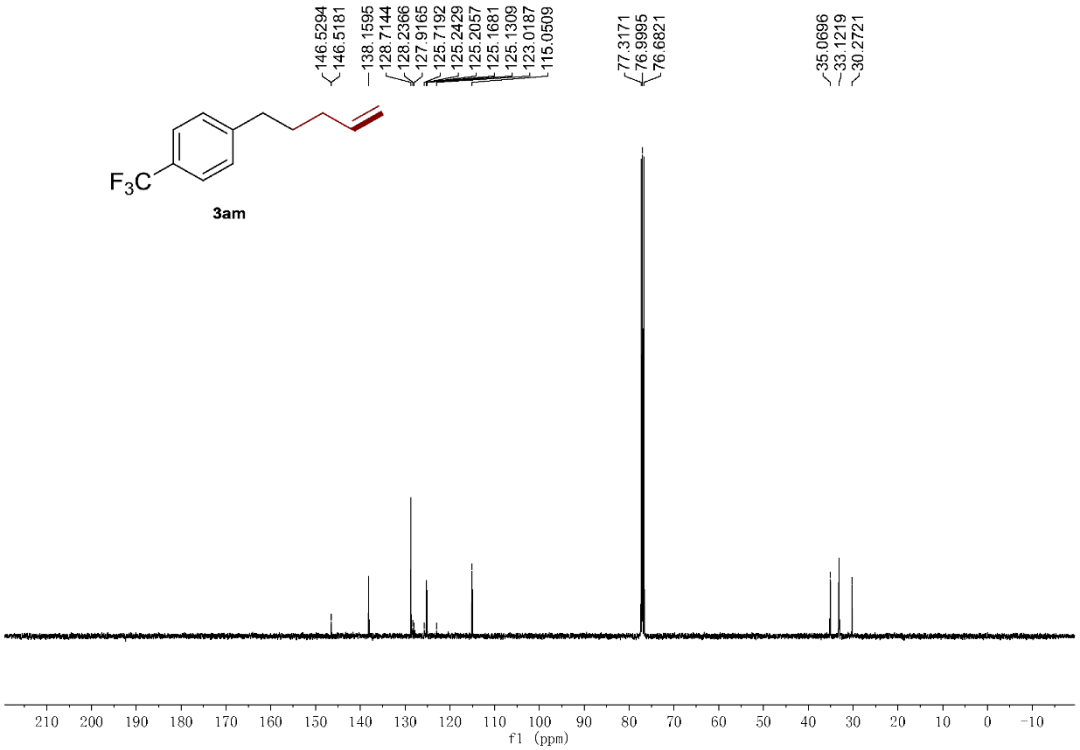
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3al



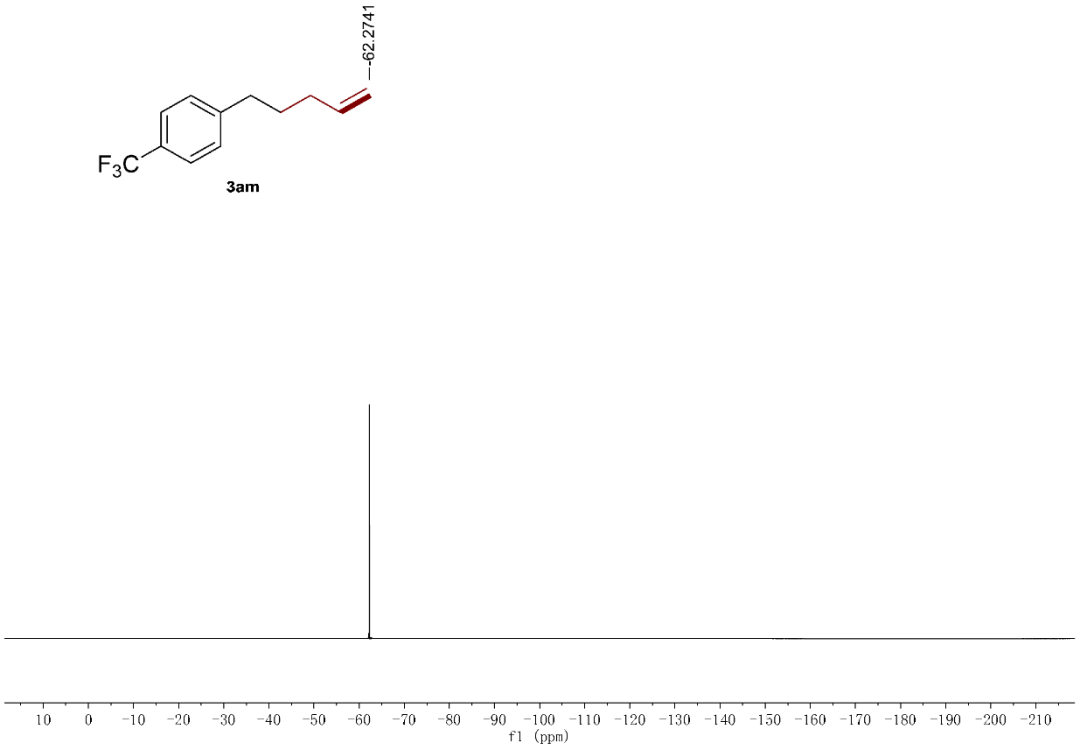
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3am



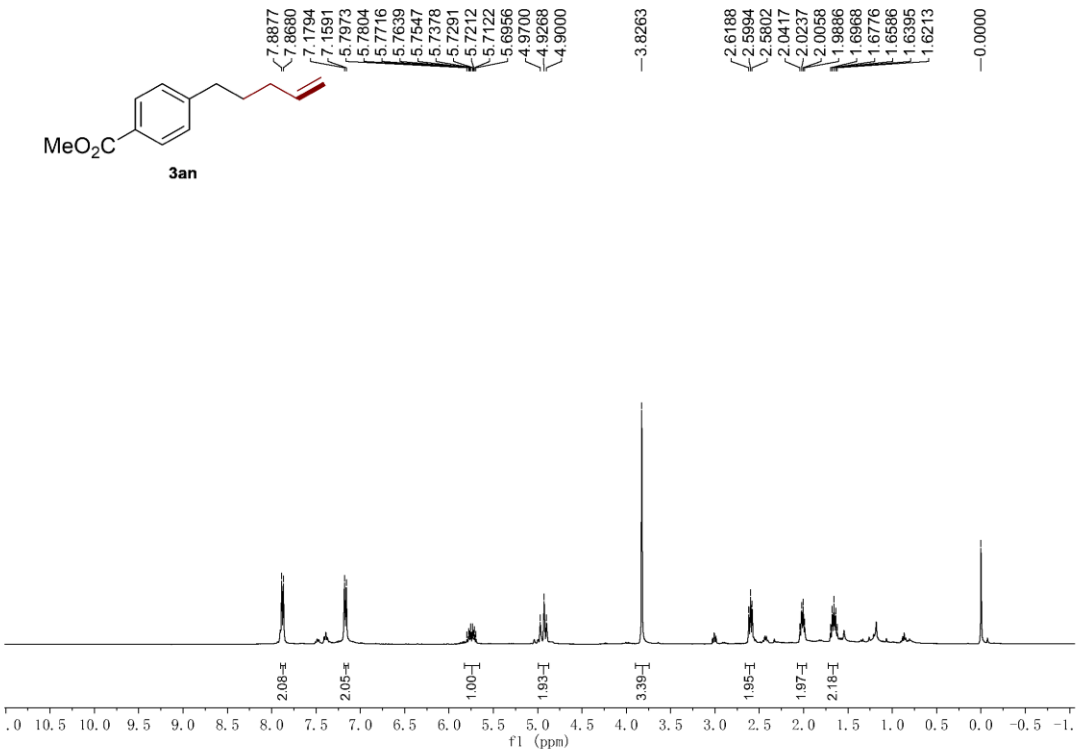
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3am



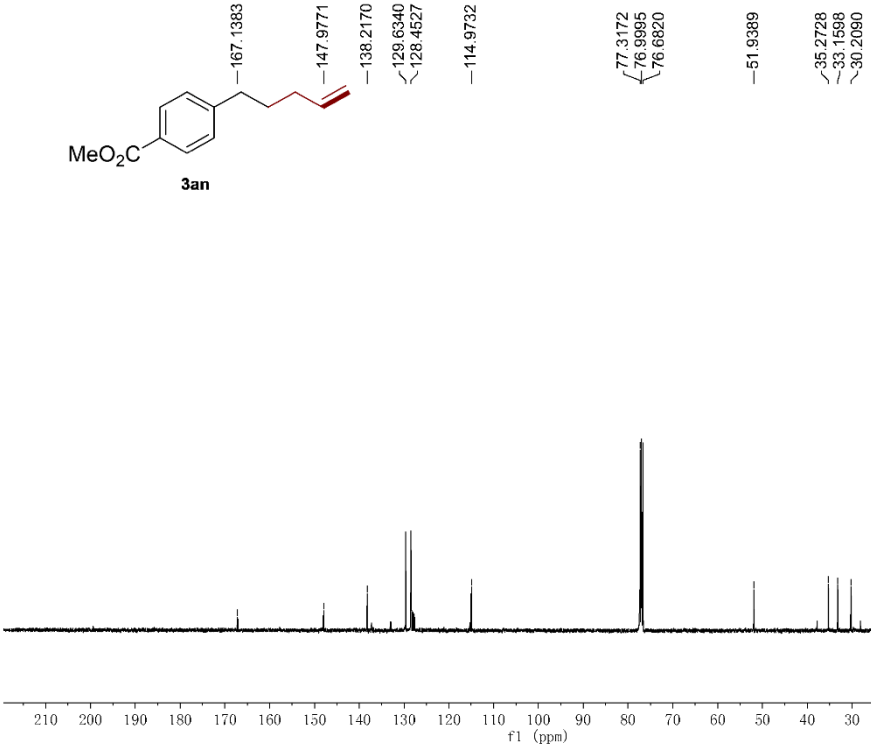
^{19}F NMR (376 MHz, $CDCl_3$) spectrum of 3am



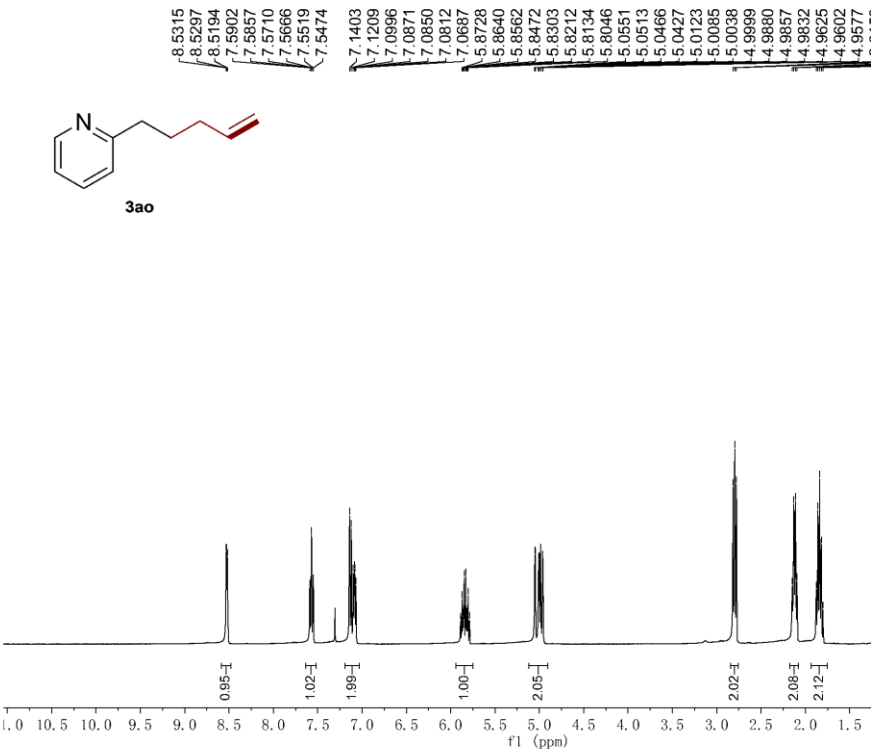
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3an



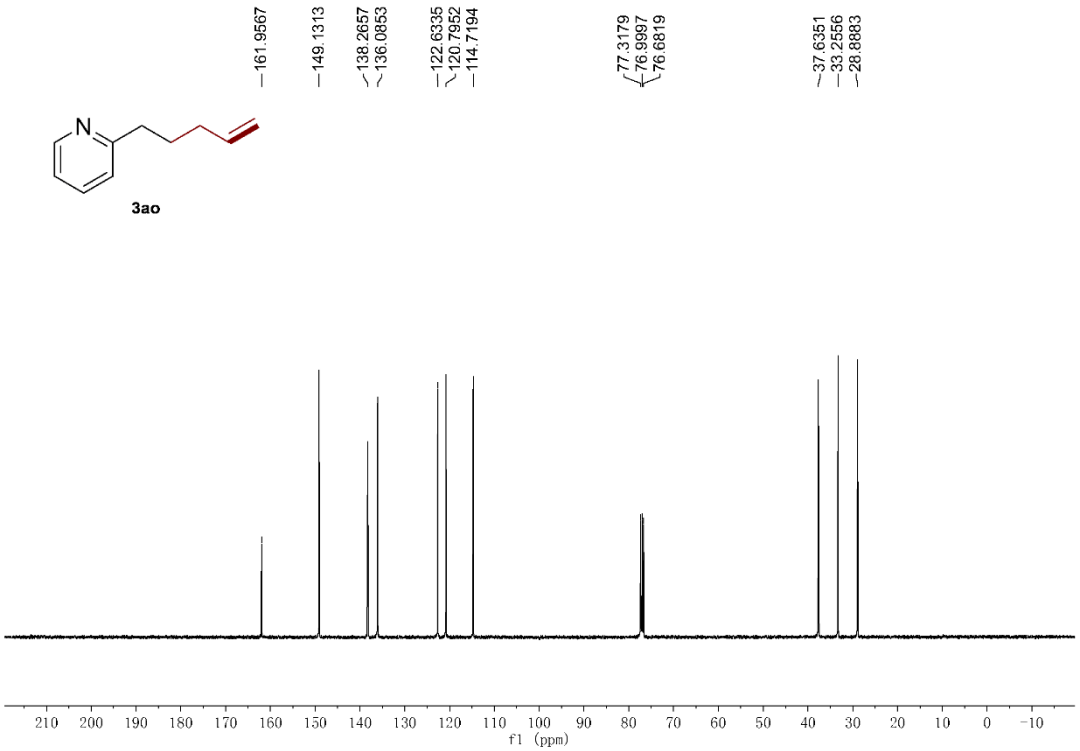
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3an



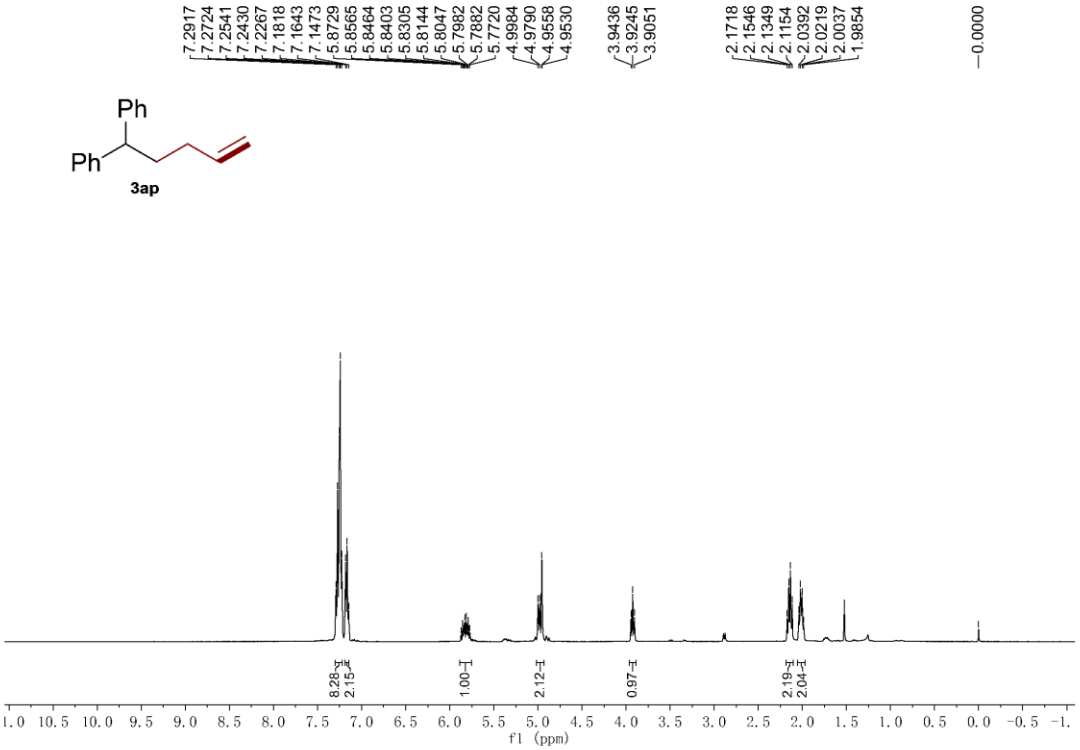
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ao



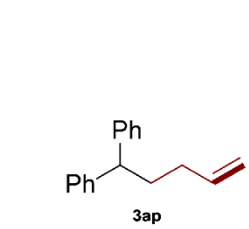
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3ao



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ap

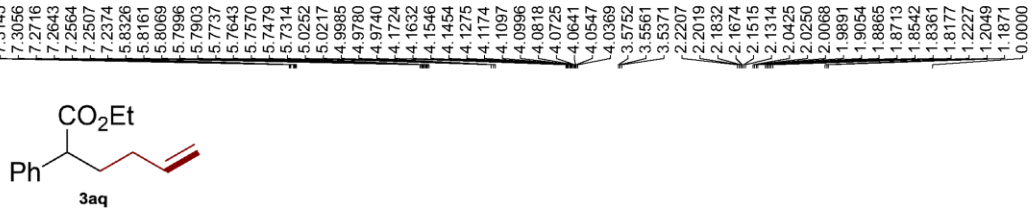


^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3ap

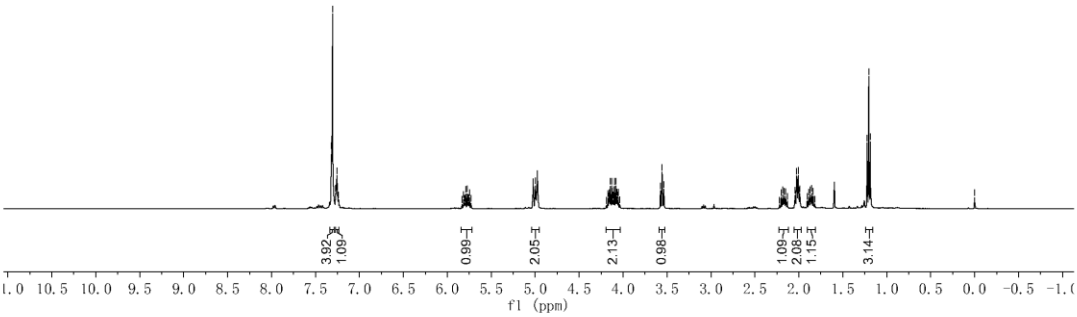


3ap

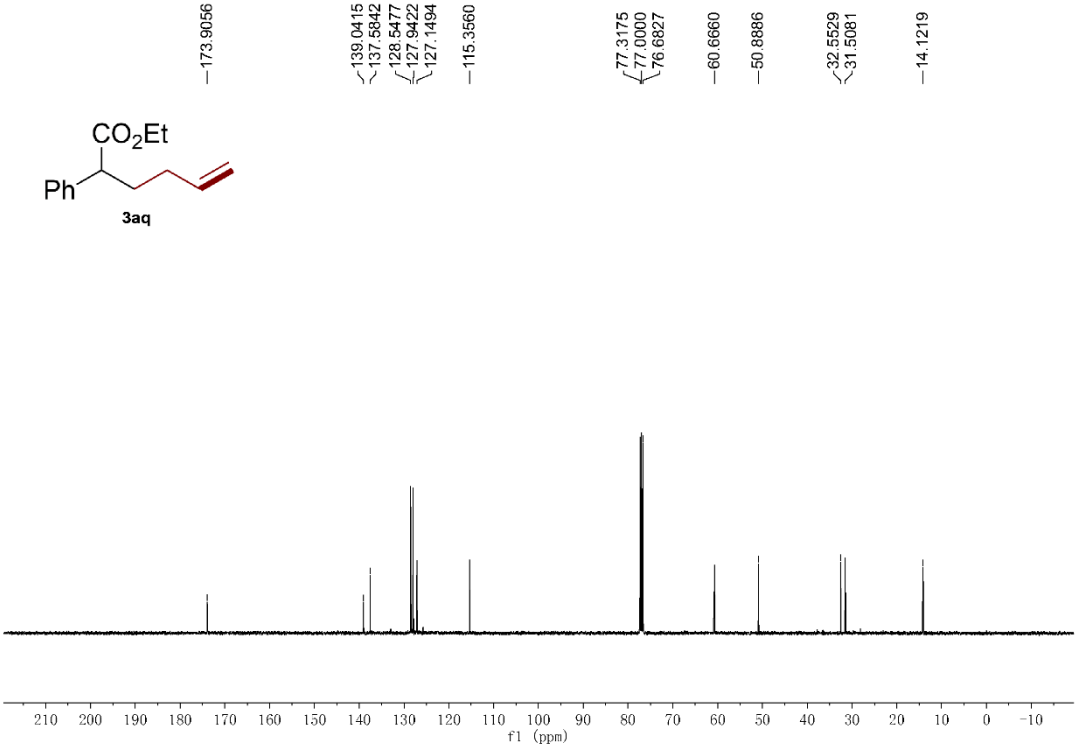
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3aq



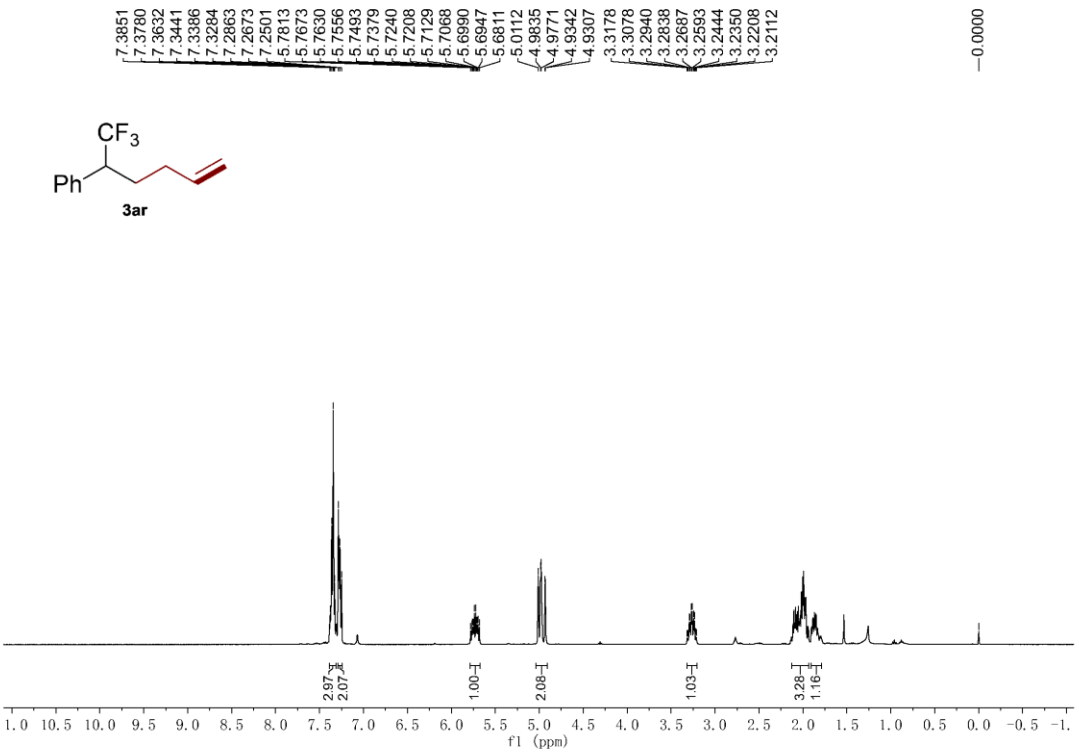
3aq



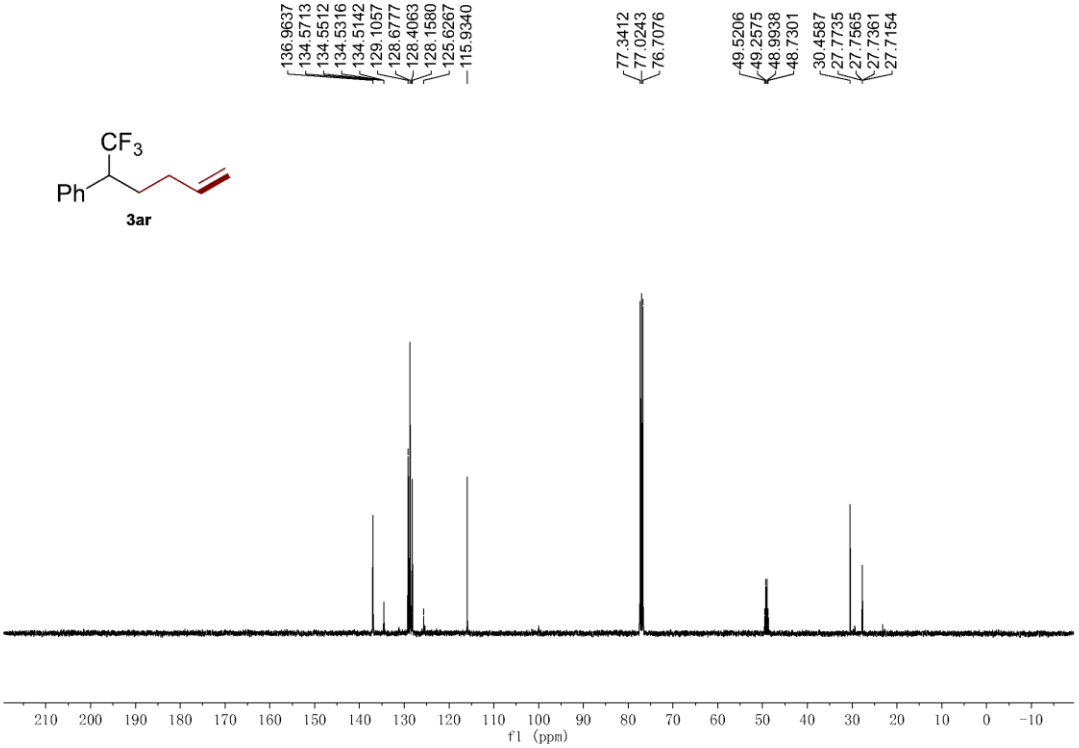
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3aq



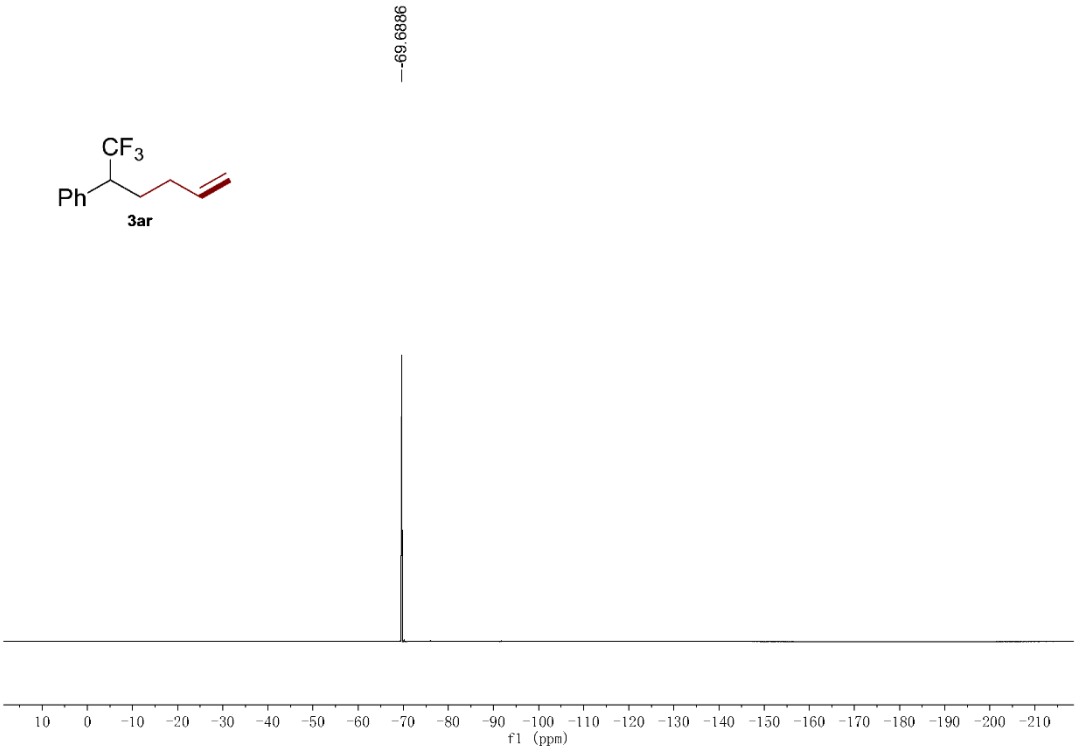
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ar



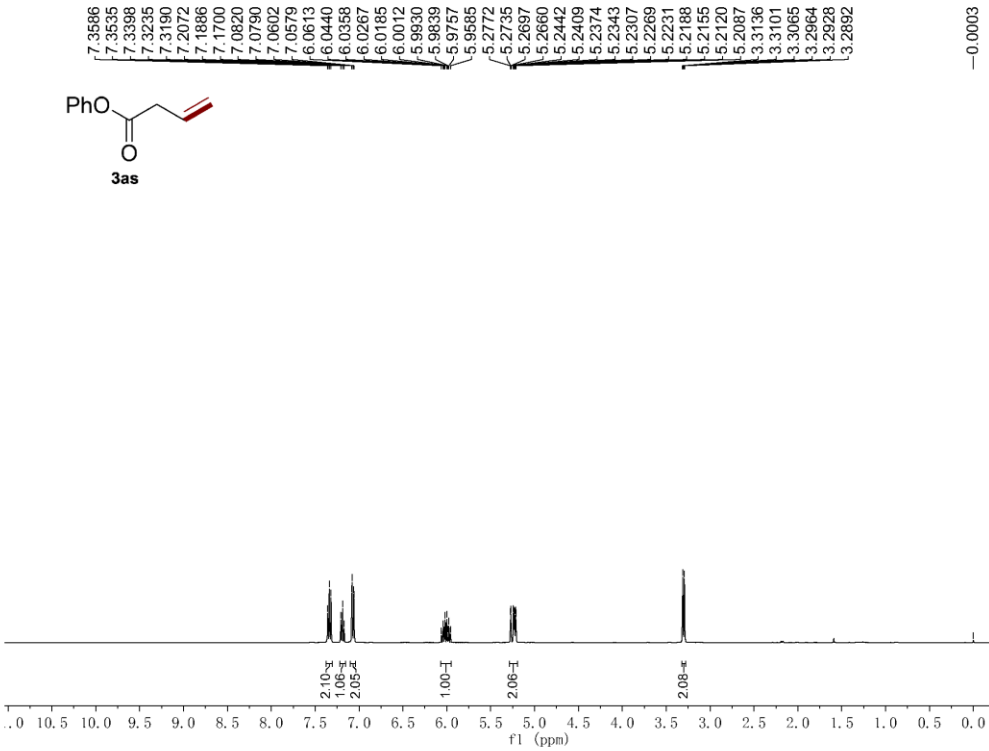
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3ar



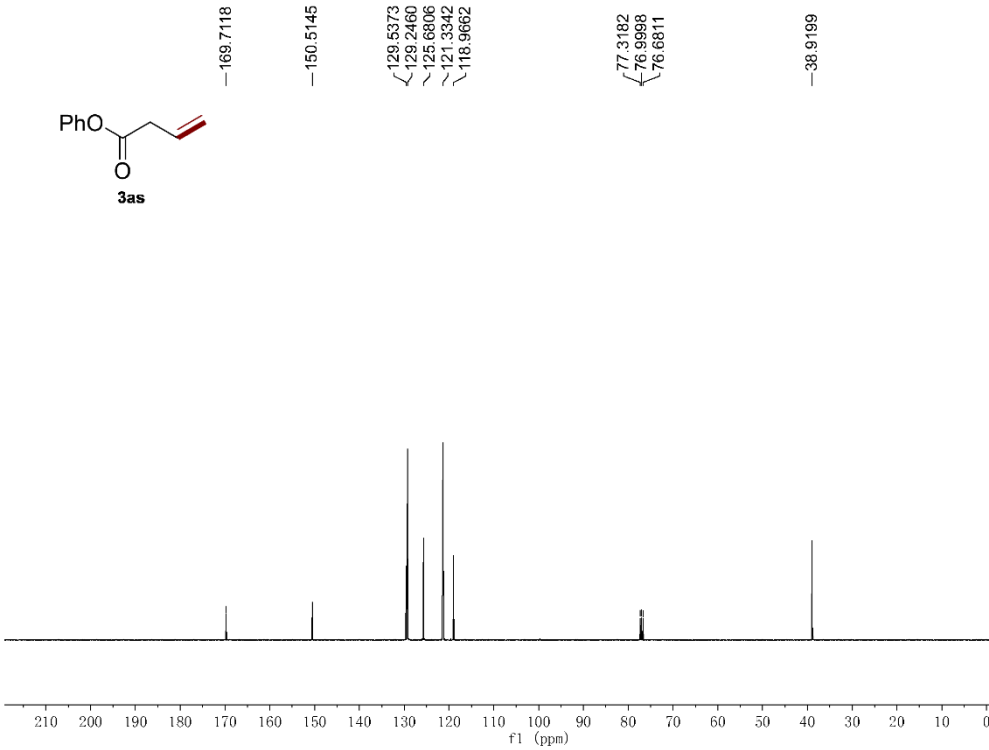
^{19}F NMR (376 MHz, $CDCl_3$) spectrum of 3ar



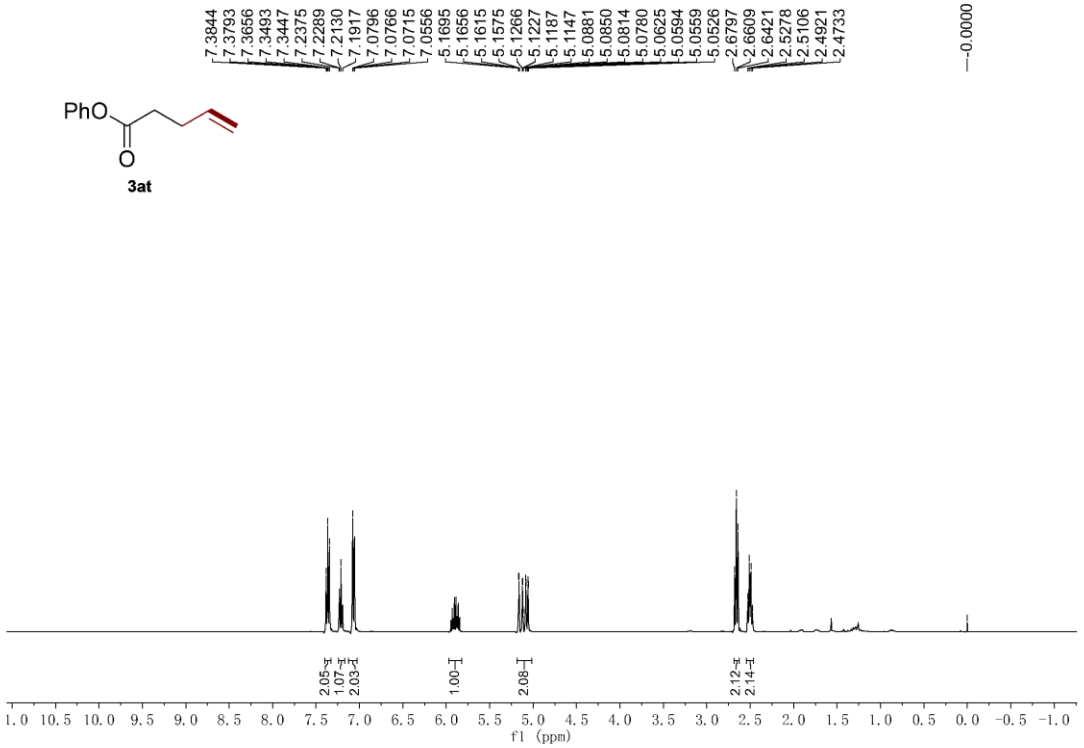
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3as



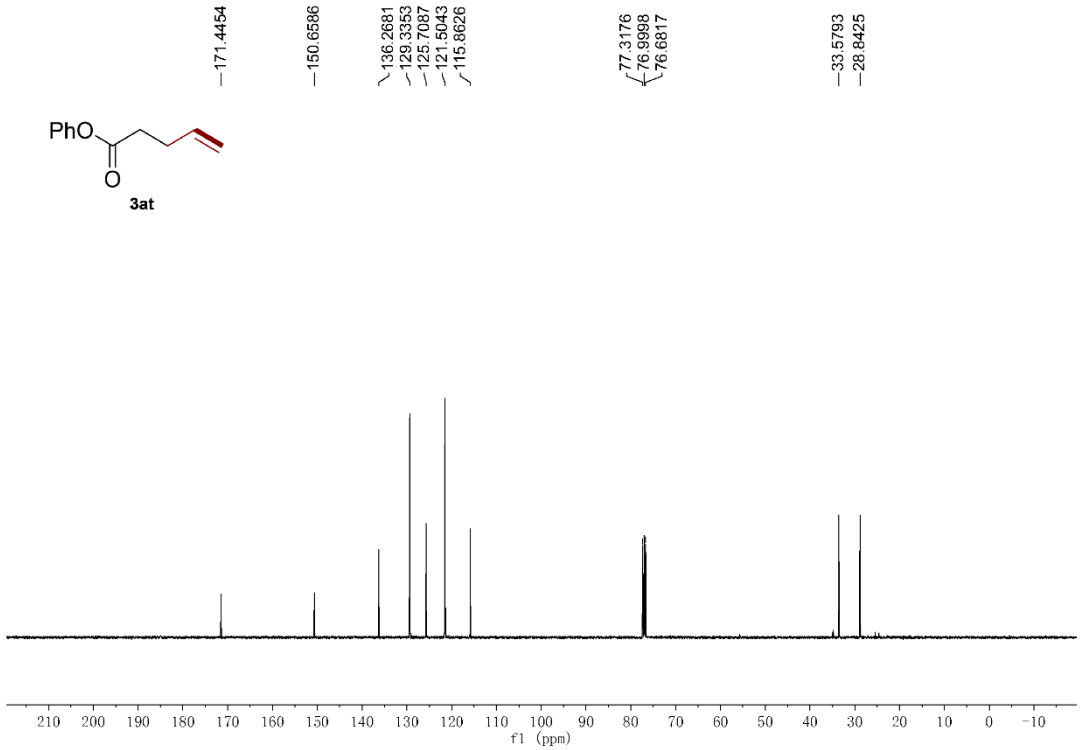
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3as



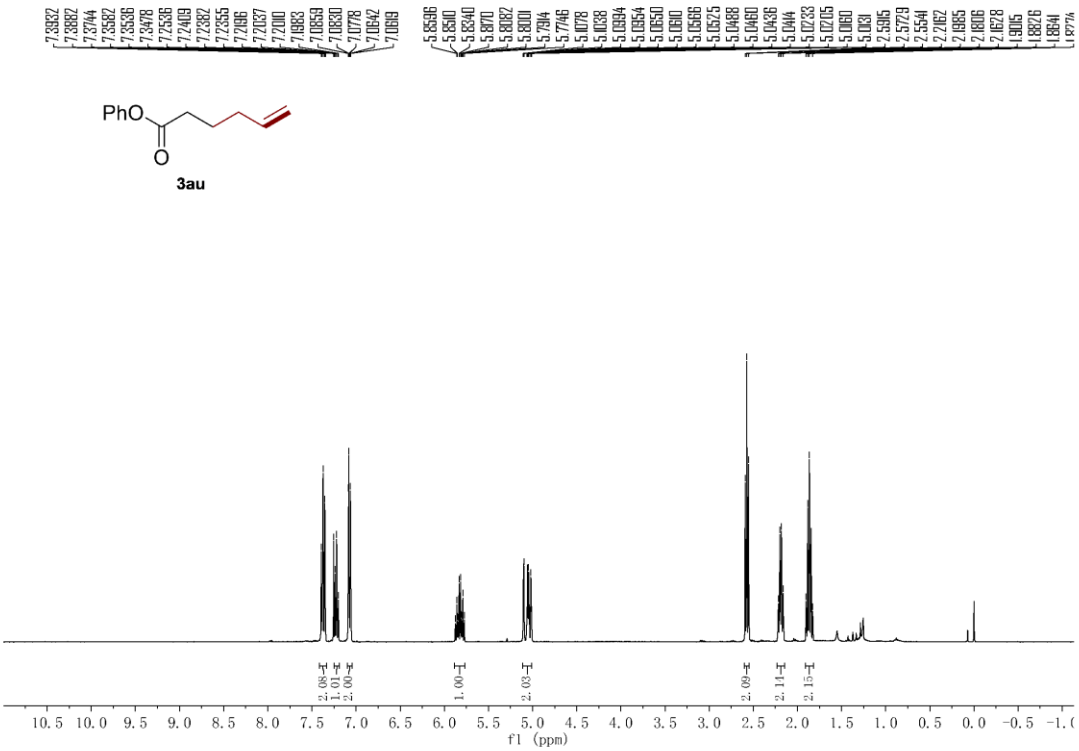
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3at



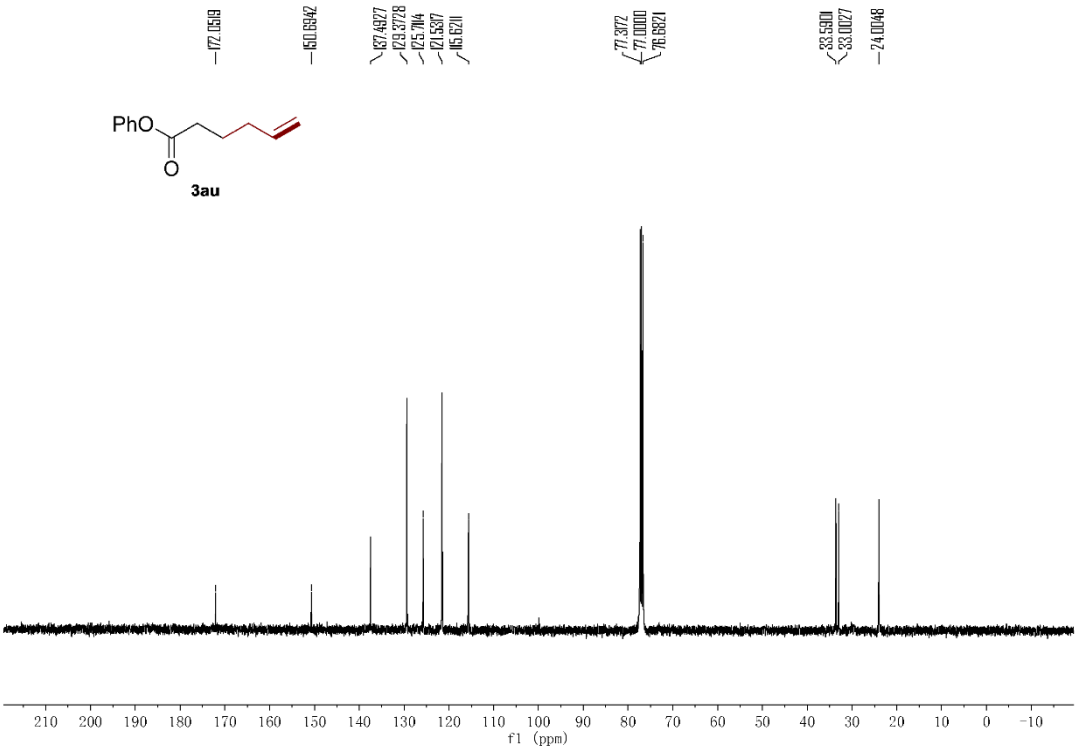
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3at



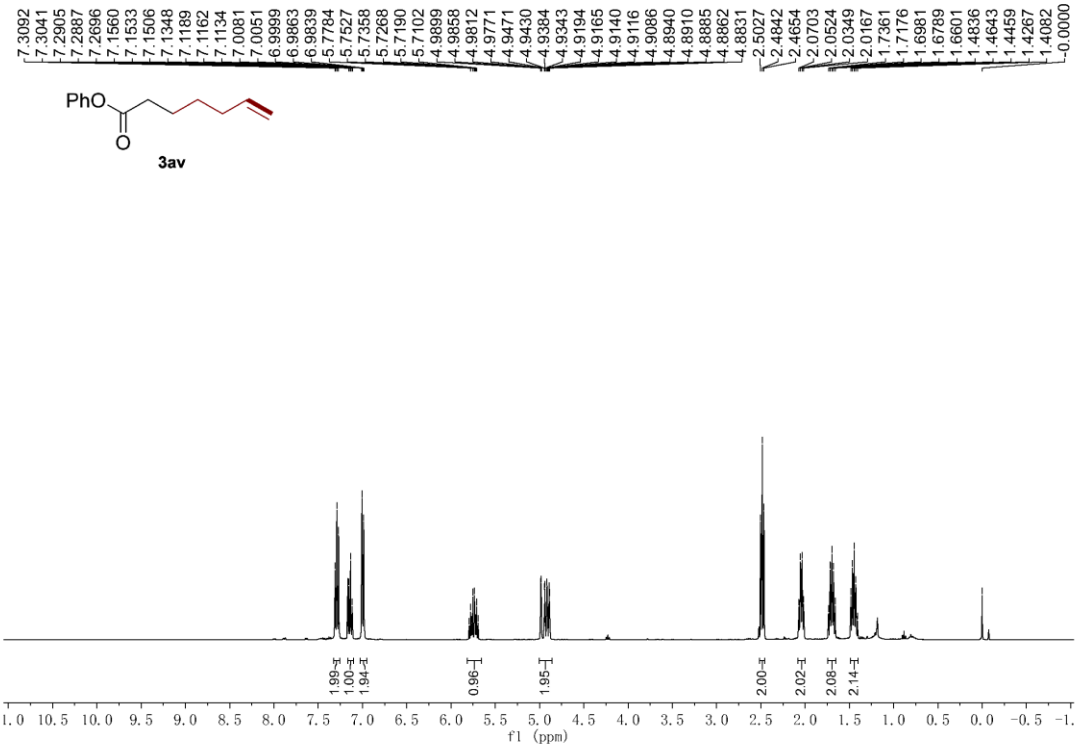
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3au



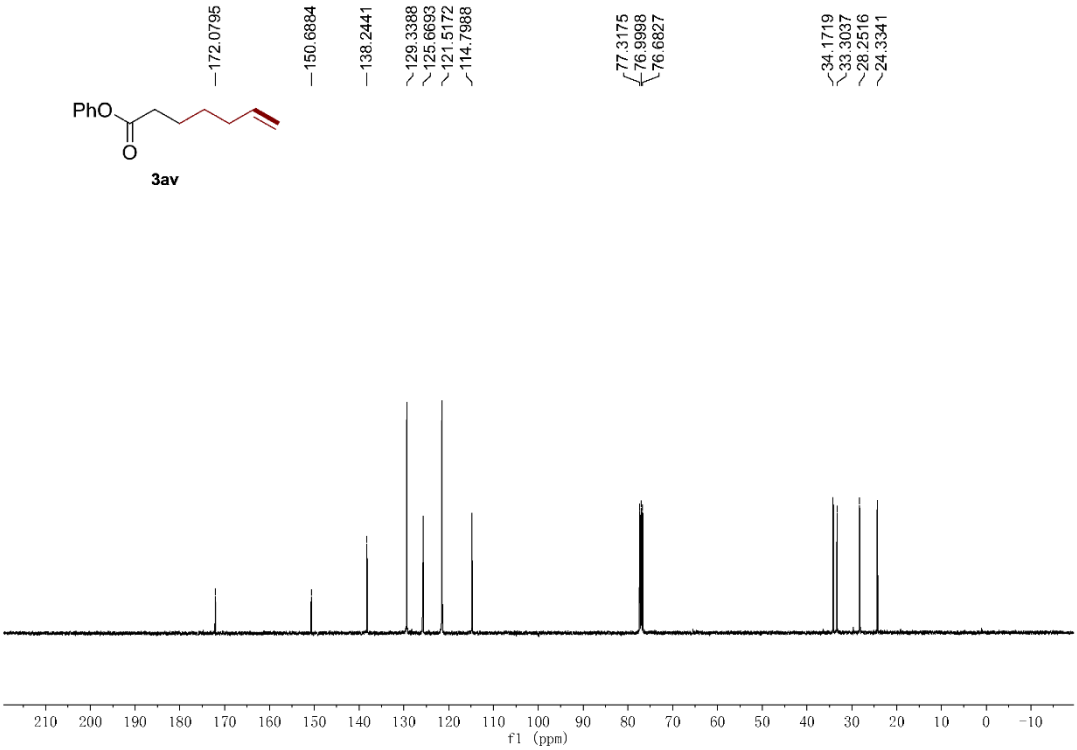
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3au



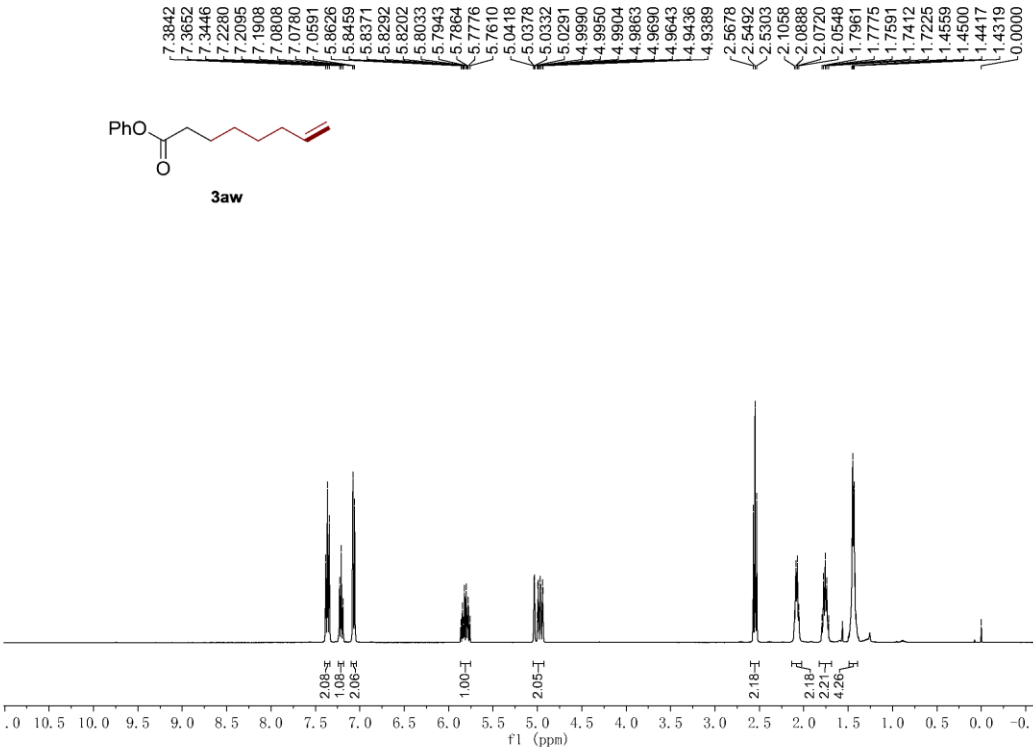
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3av



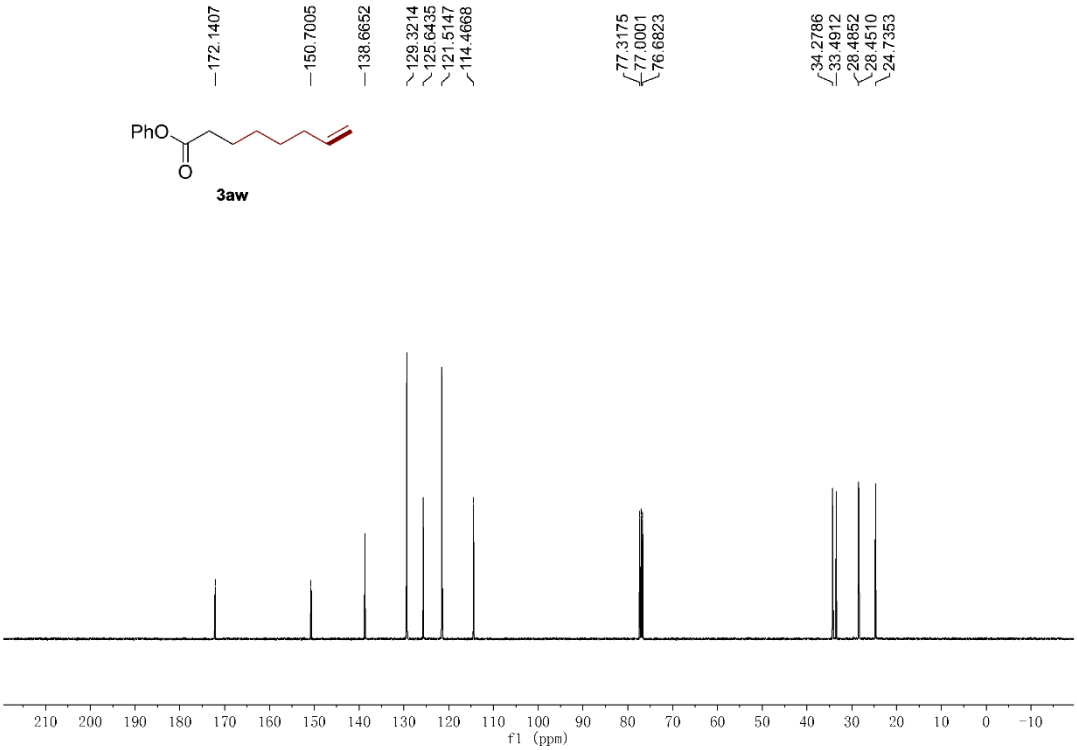
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3av



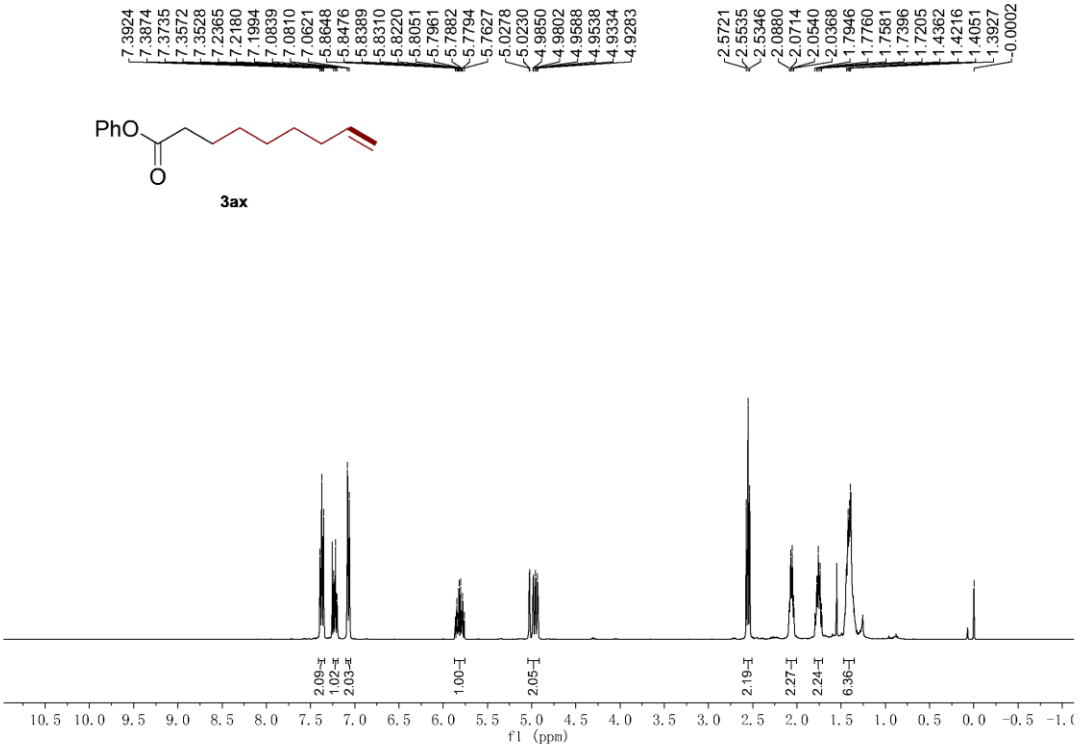
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3aw



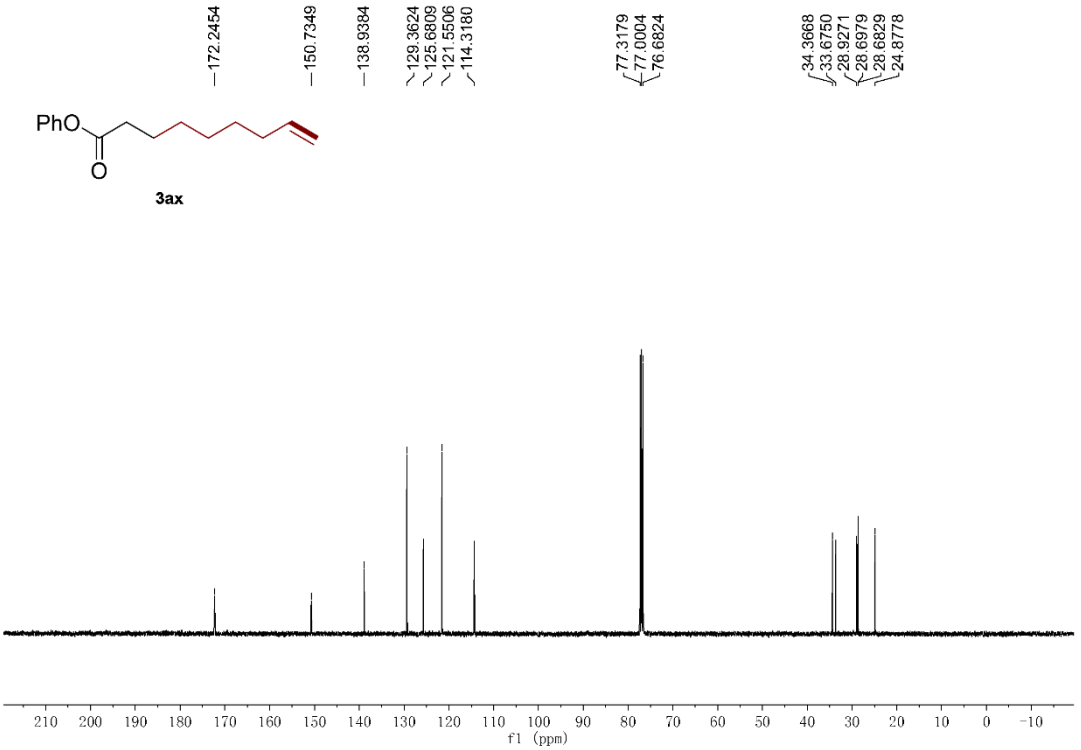
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3aw



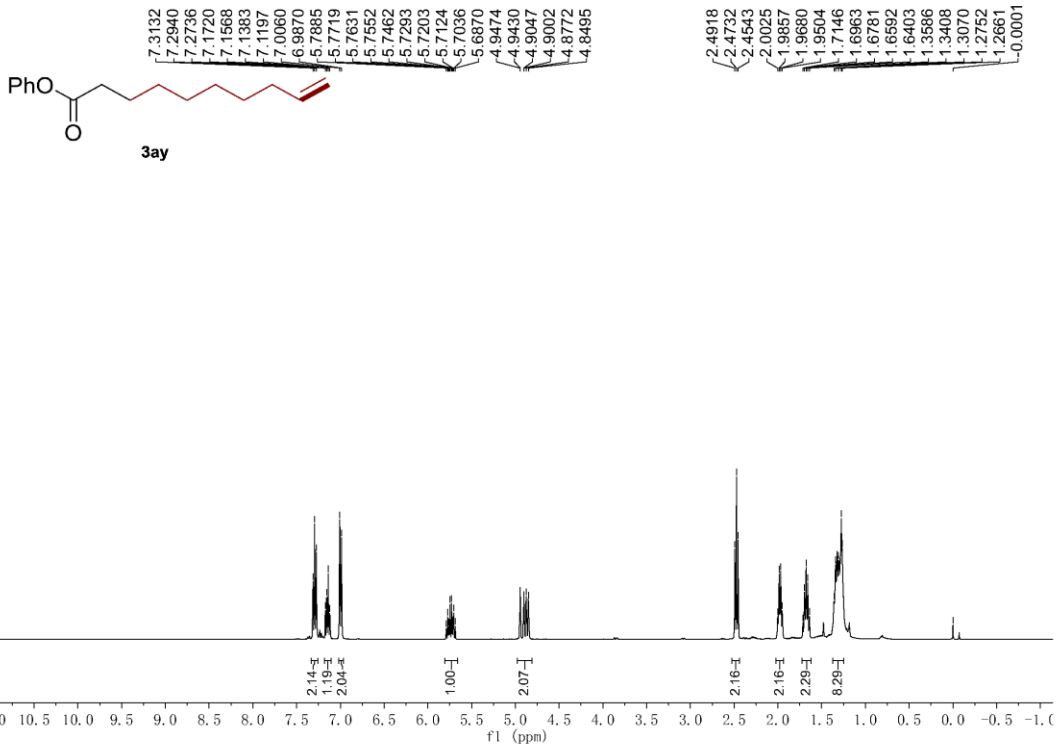
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ax



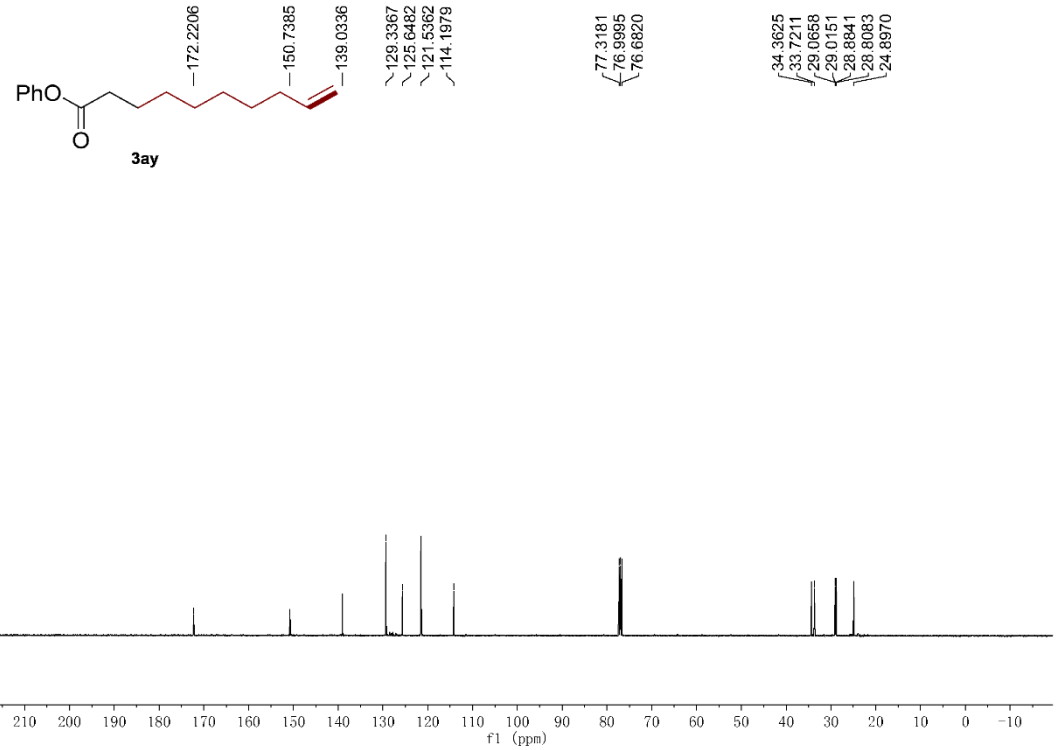
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3ax



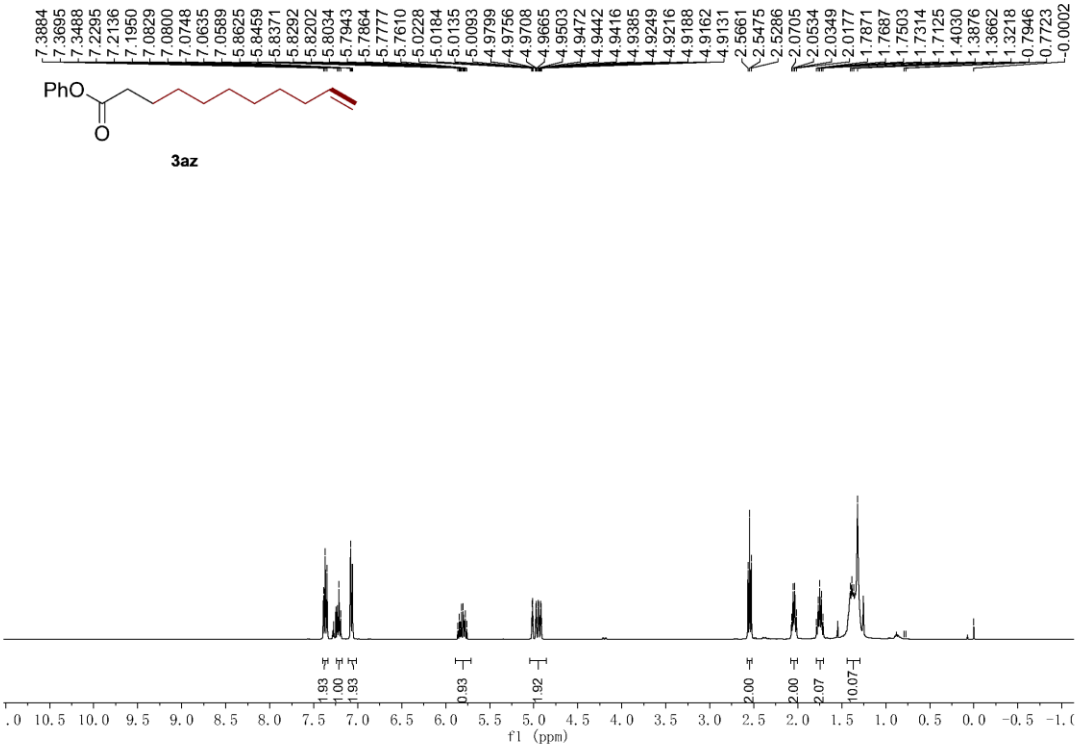
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ay



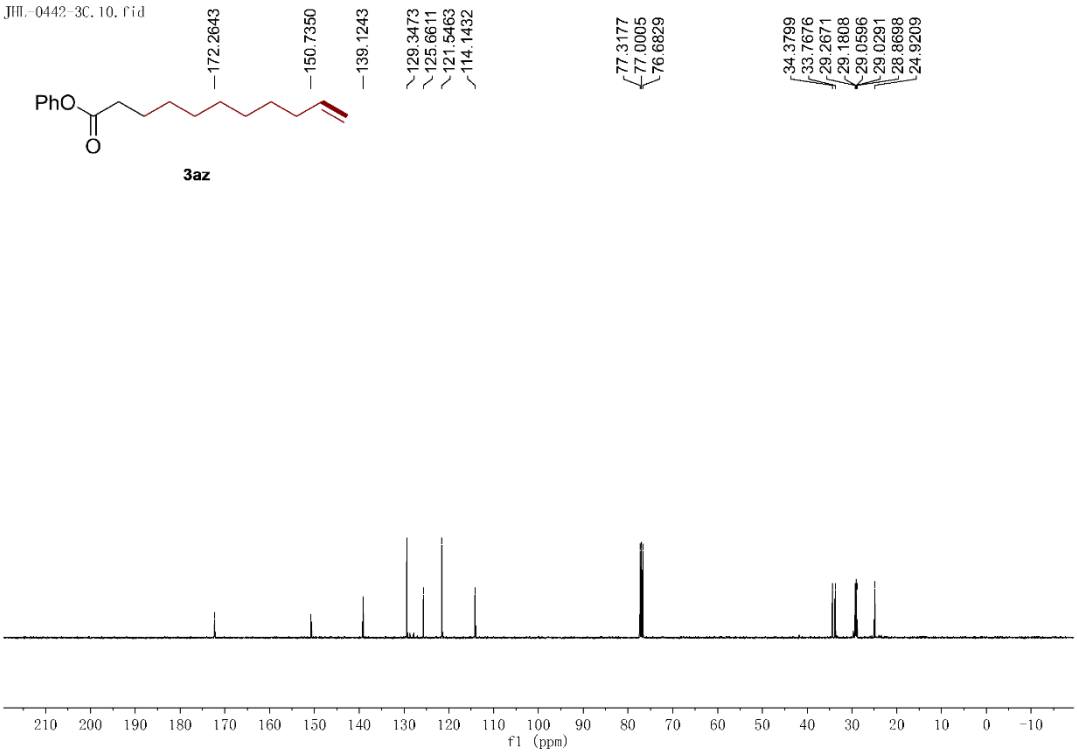
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3ay



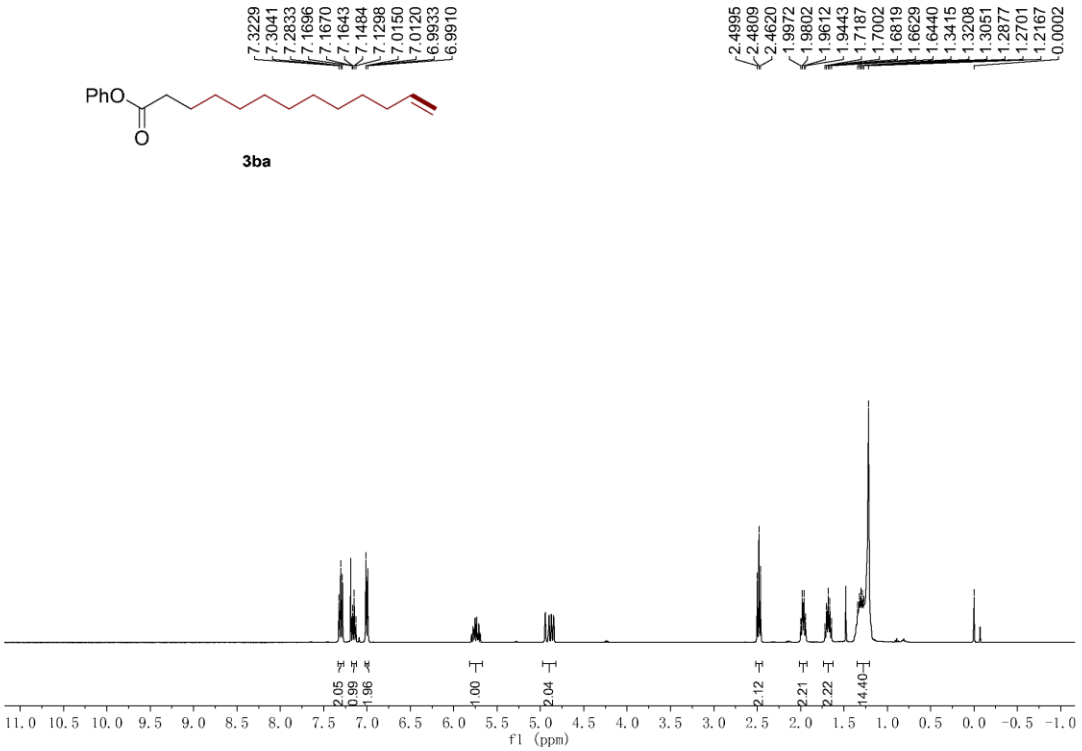
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3az



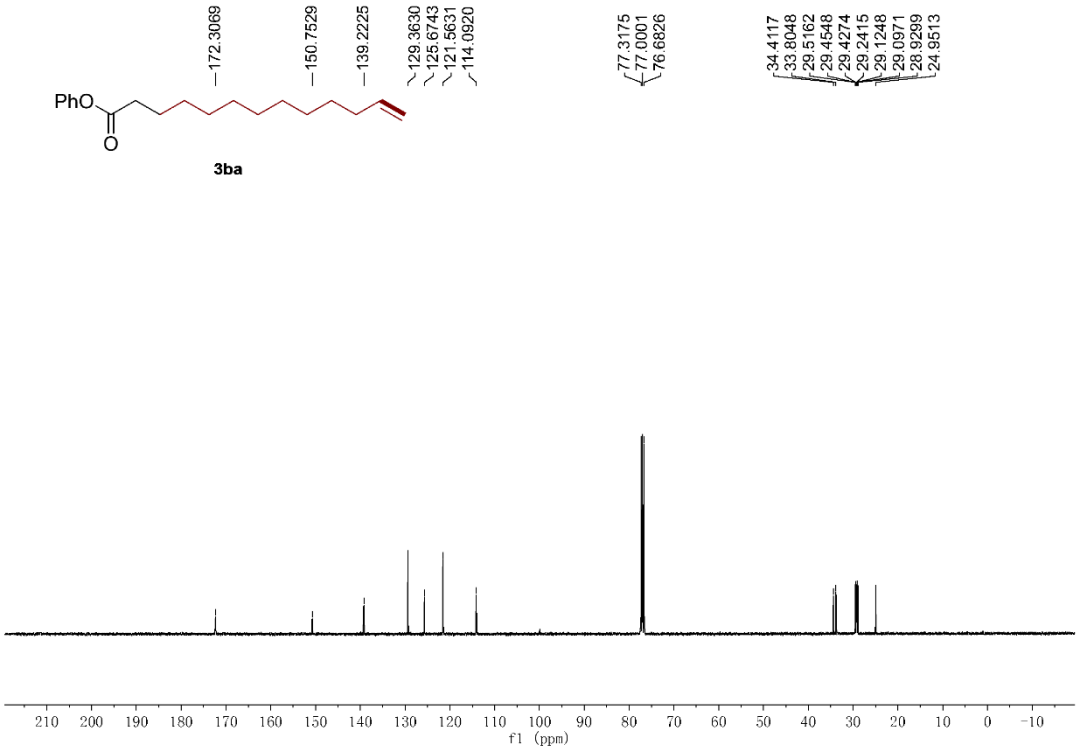
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3az



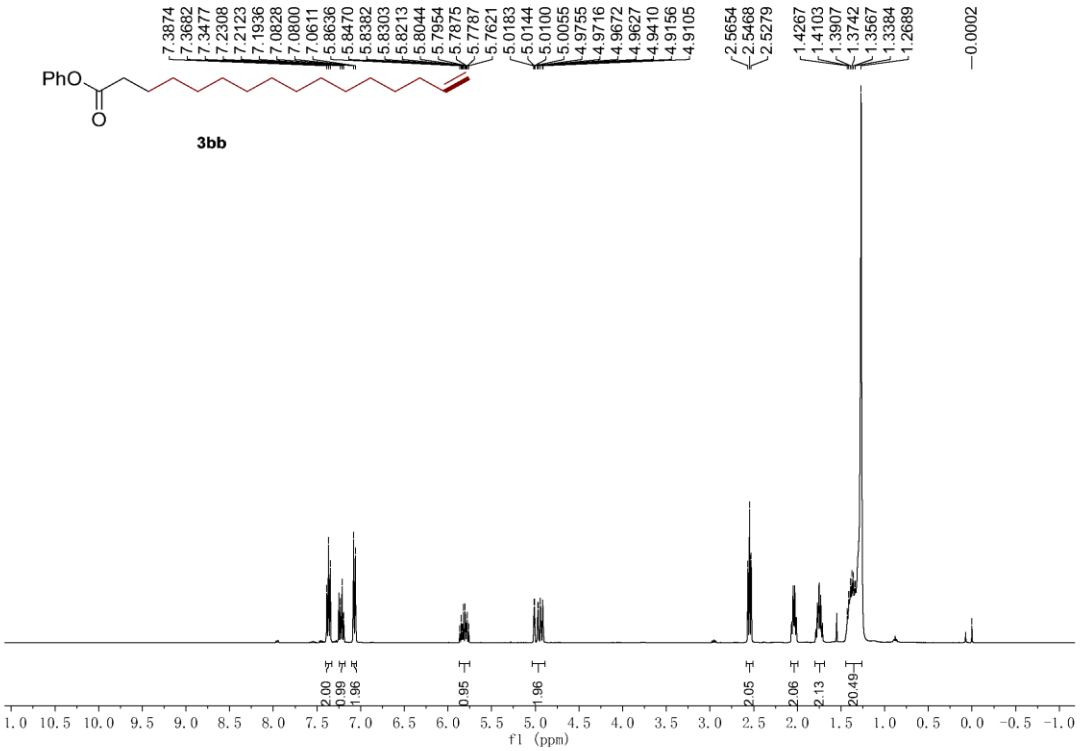
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ba



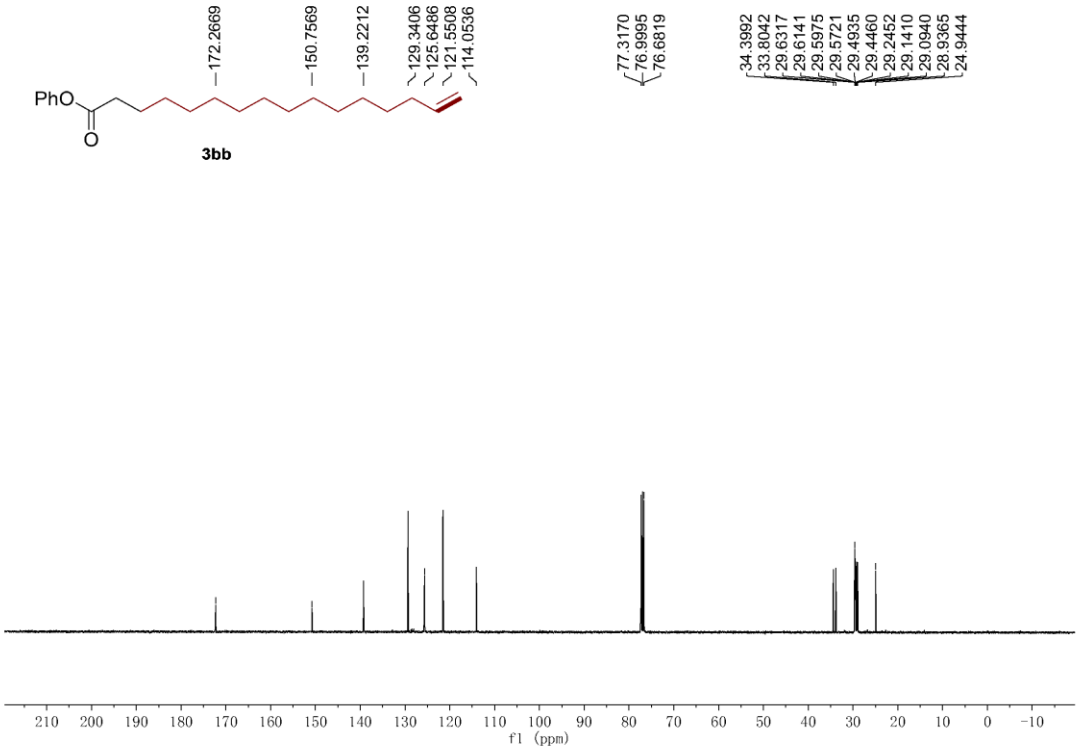
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3ba



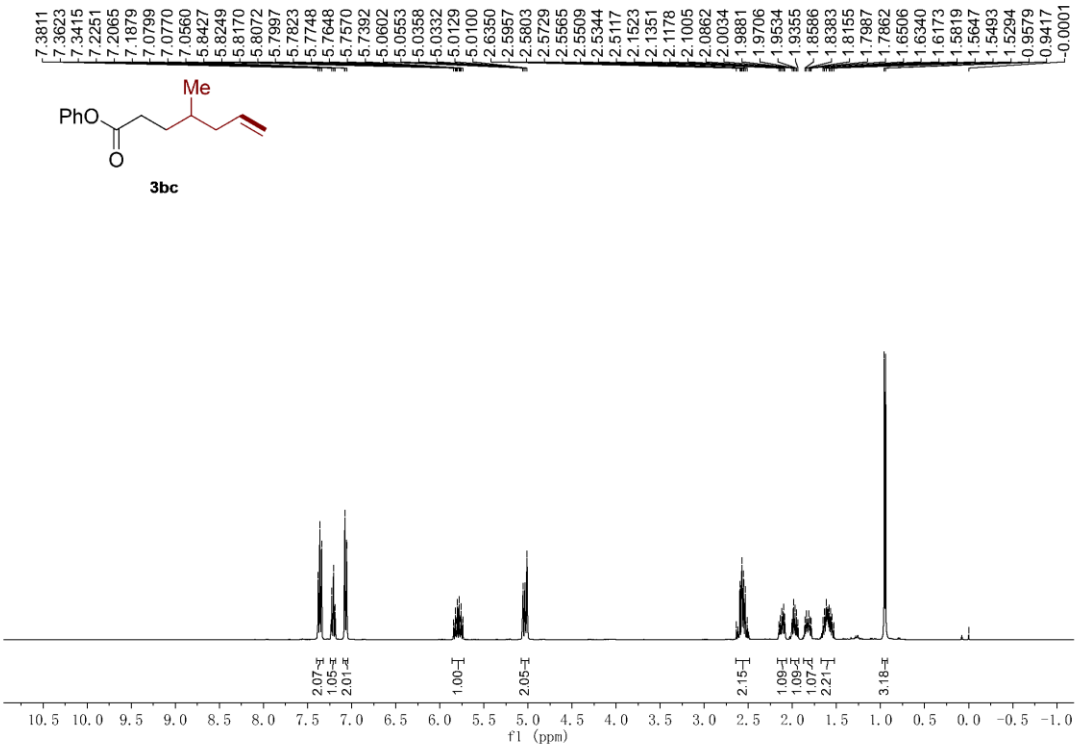
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3bb



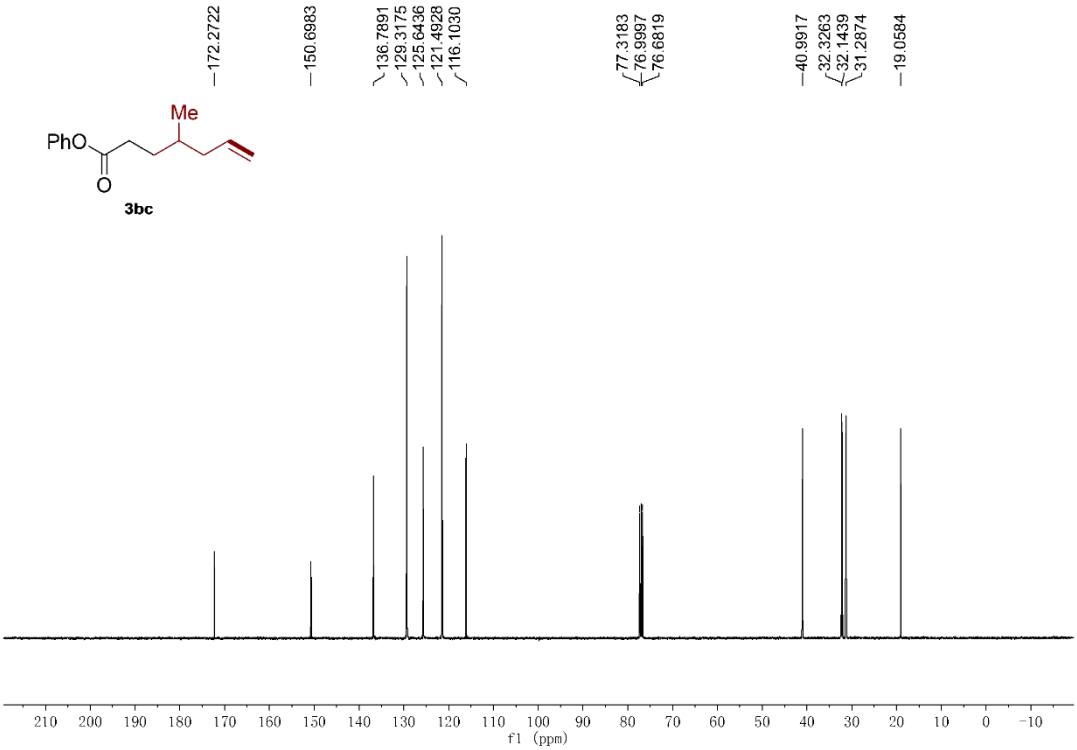
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3bb



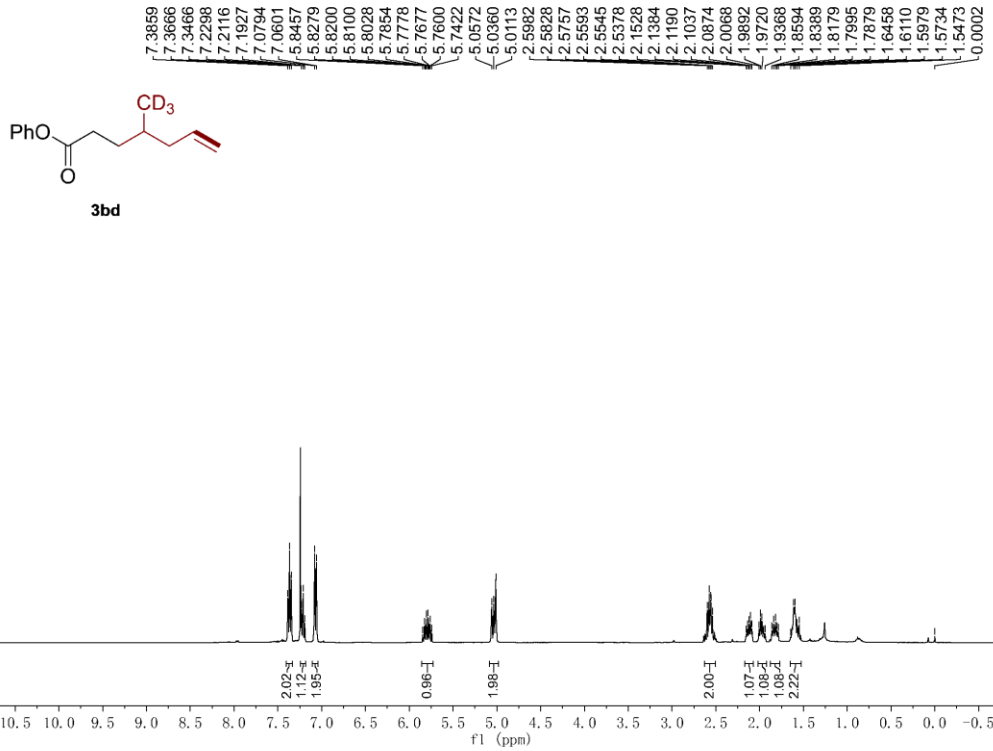
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3bc



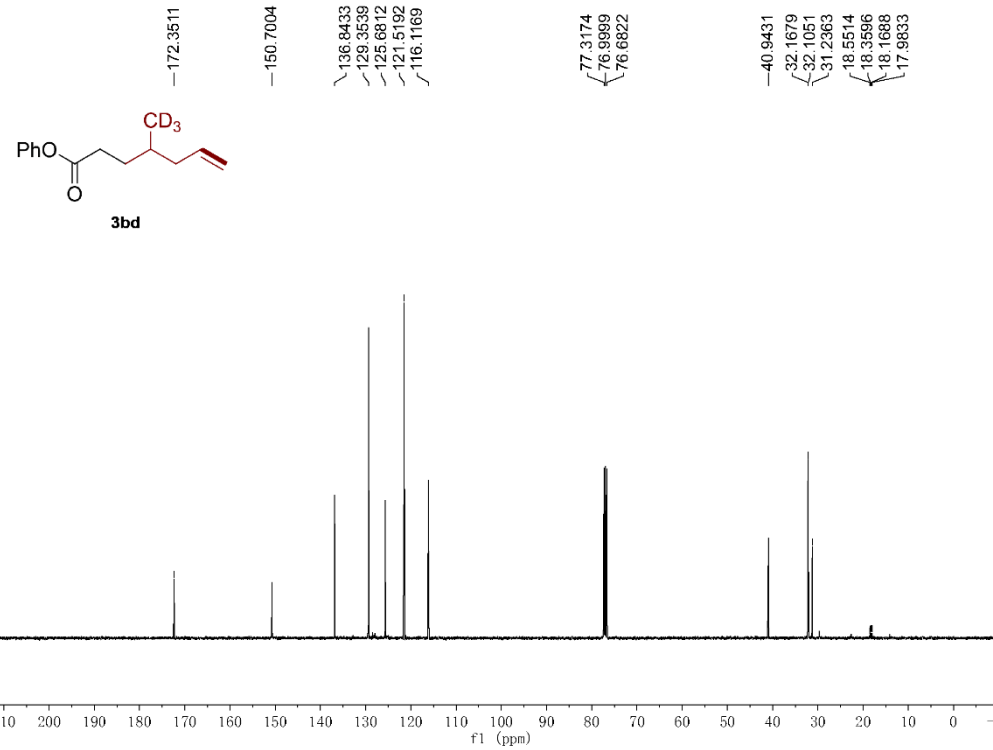
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3bc



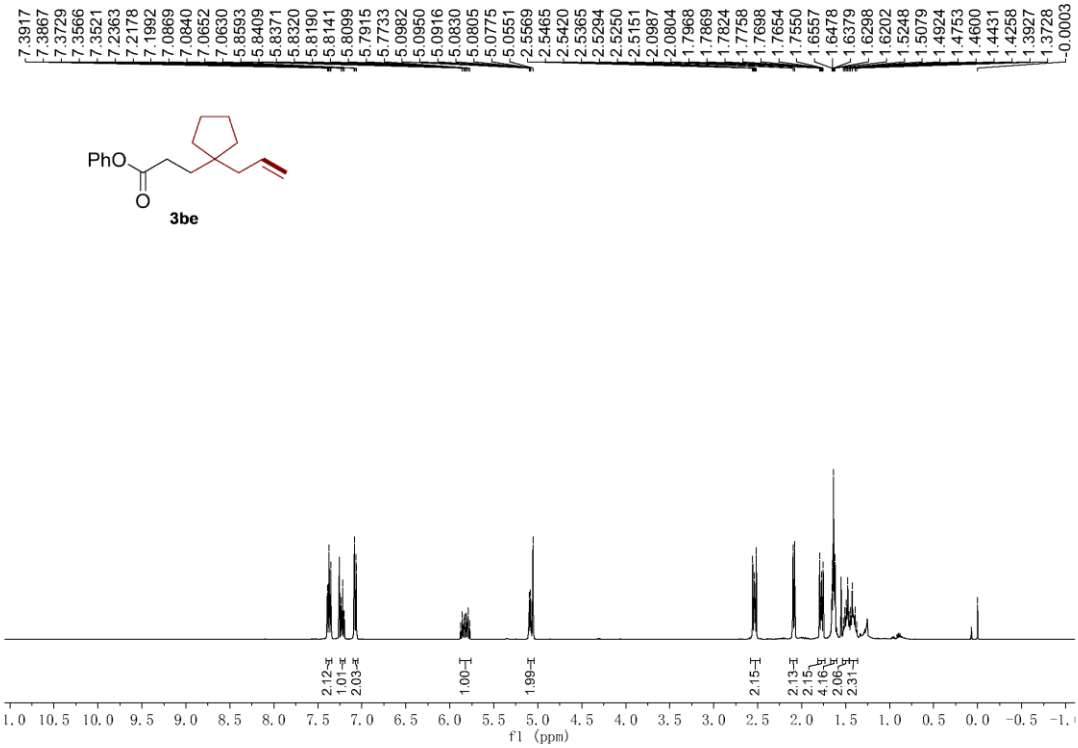
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3bd



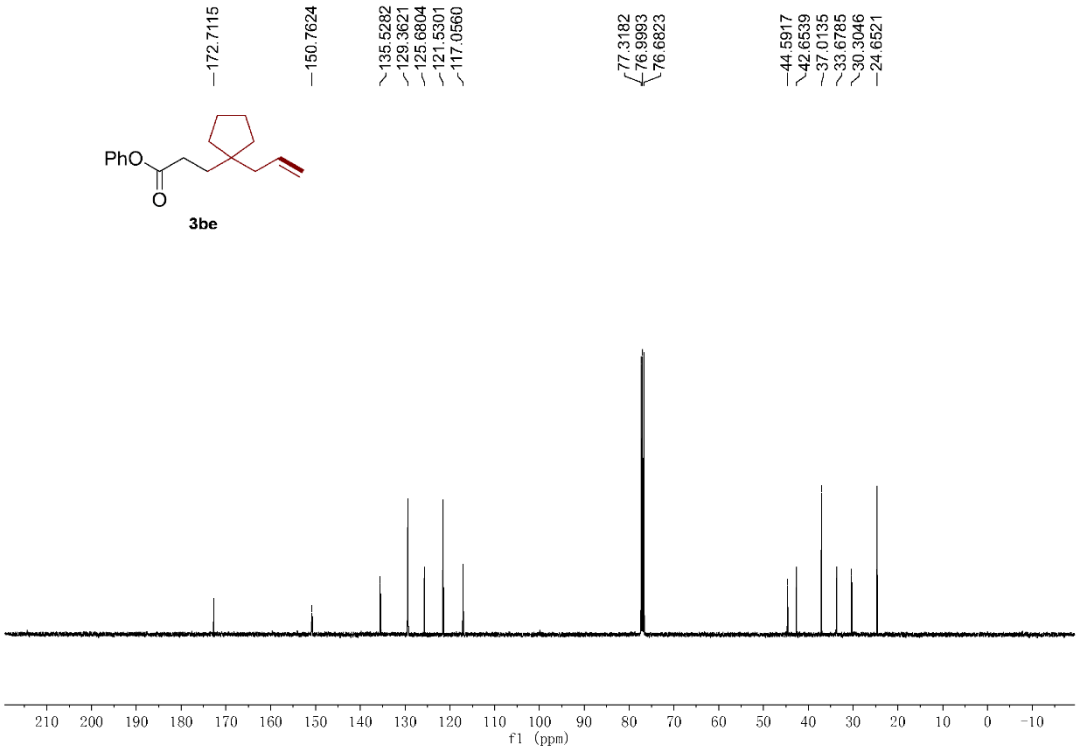
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3bd



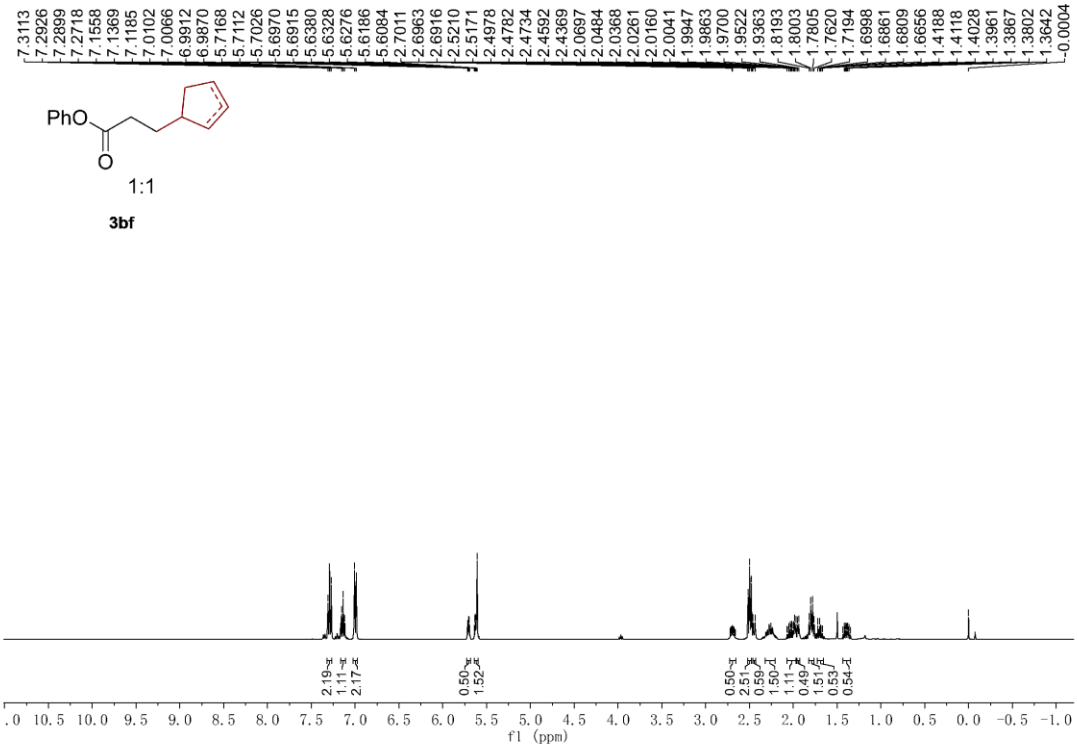
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3be



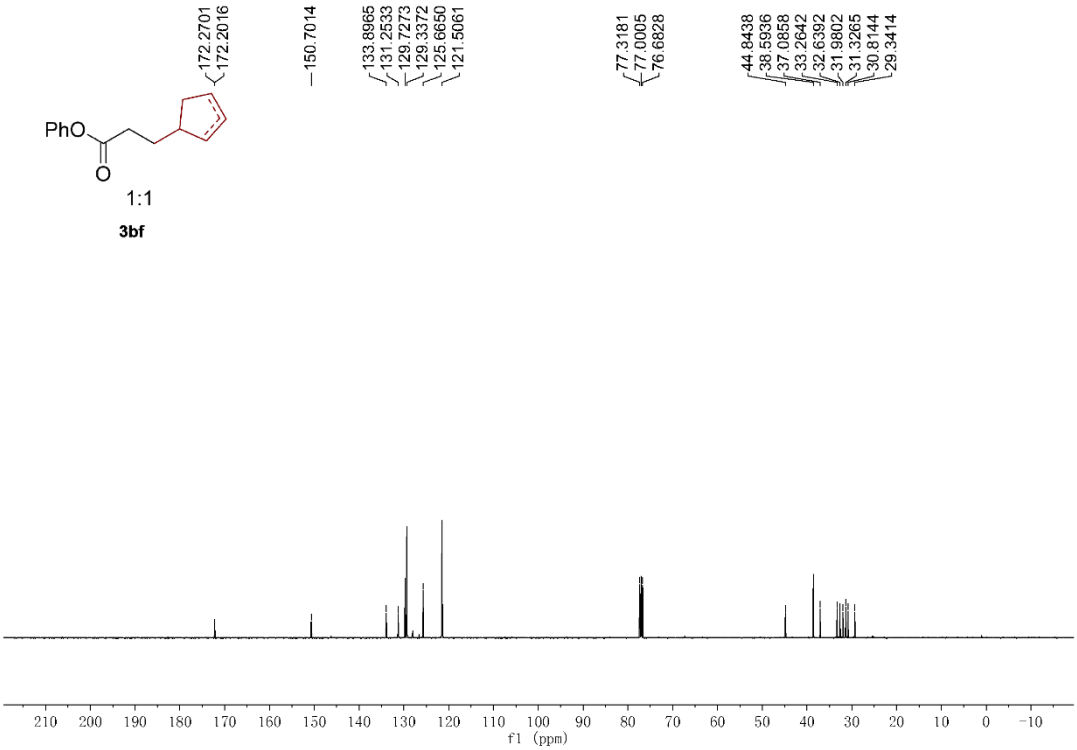
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3be



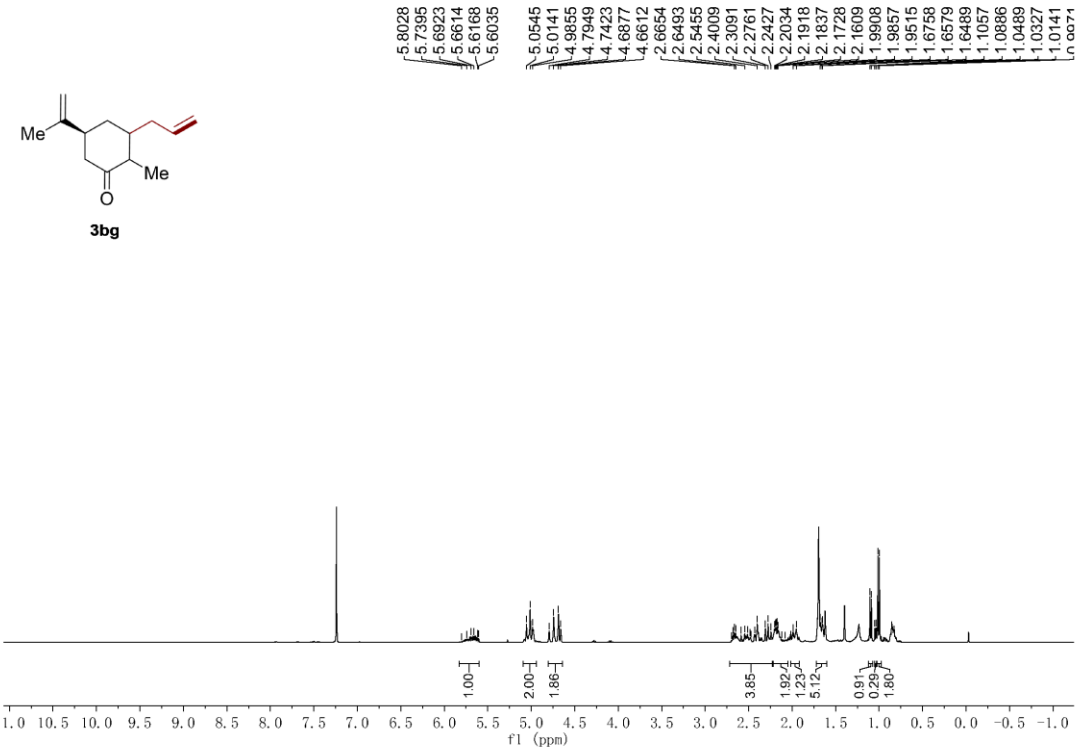
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3bf



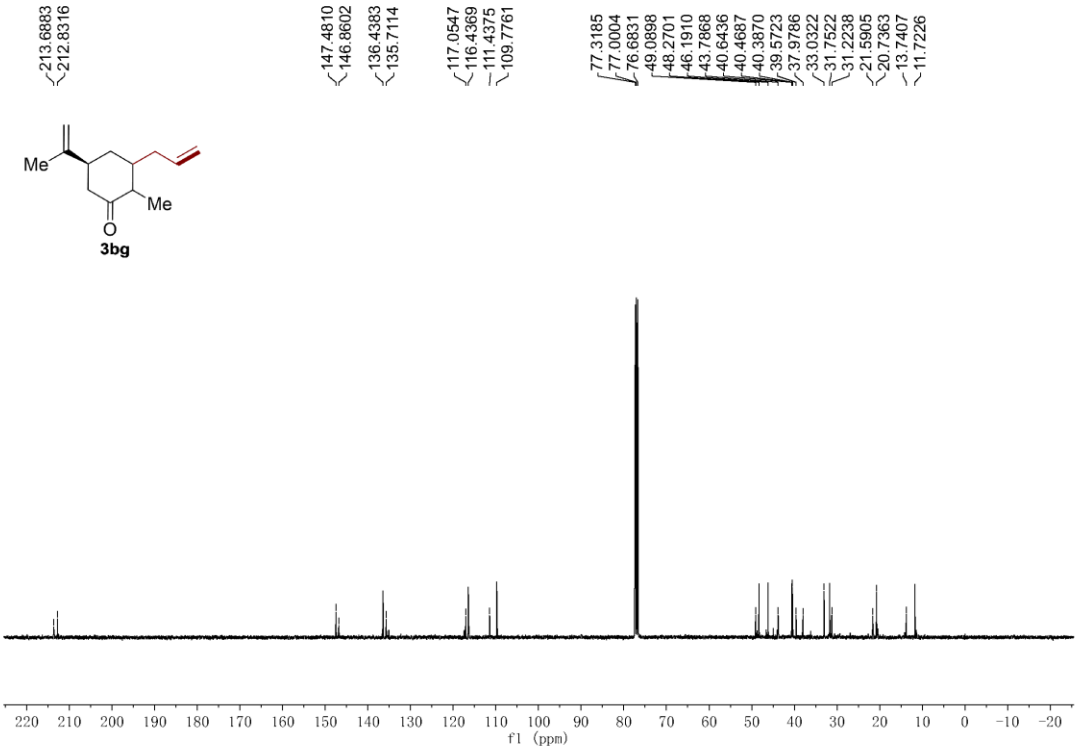
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3bf



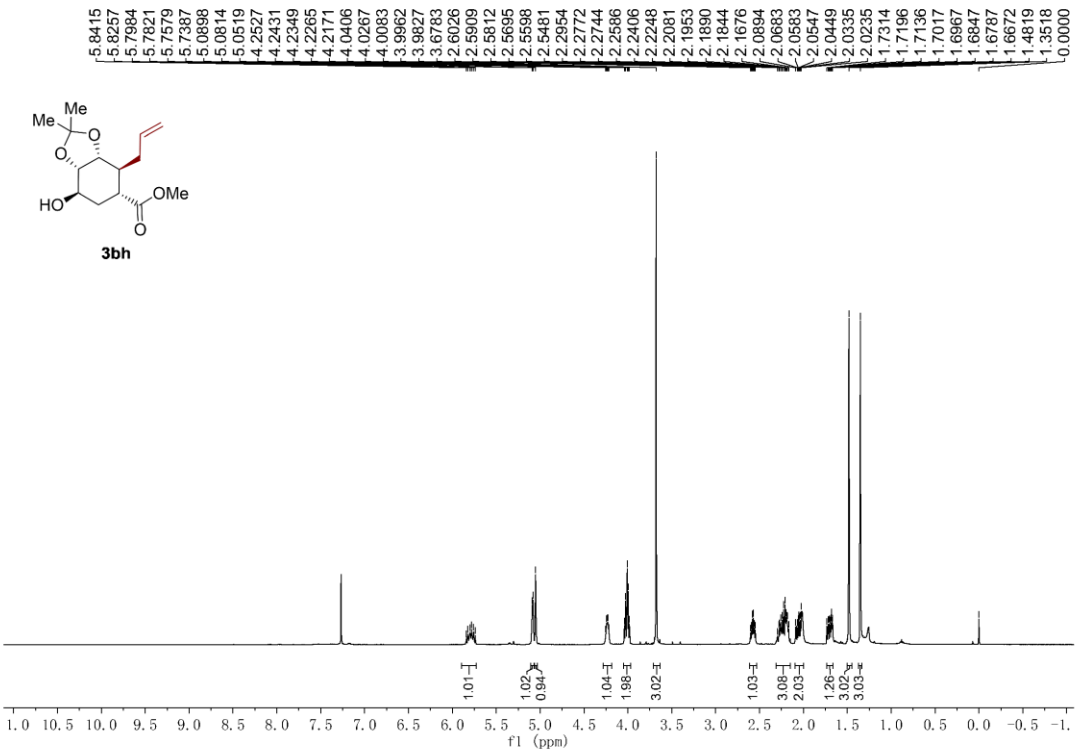
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3bg



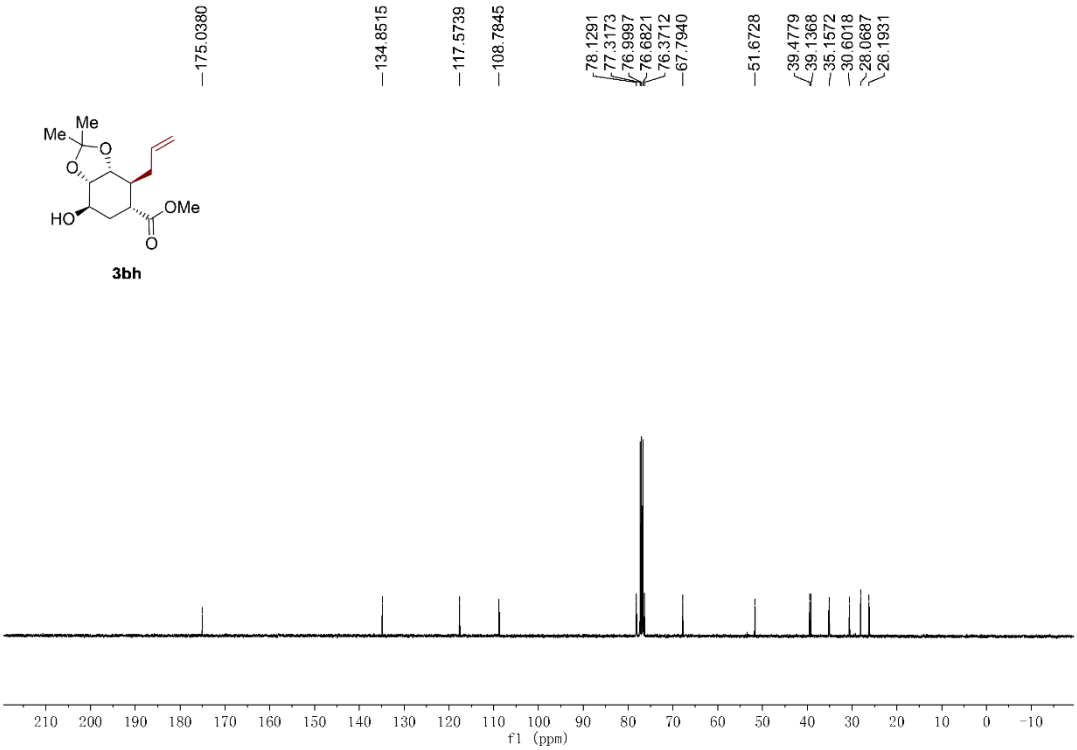
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3bg



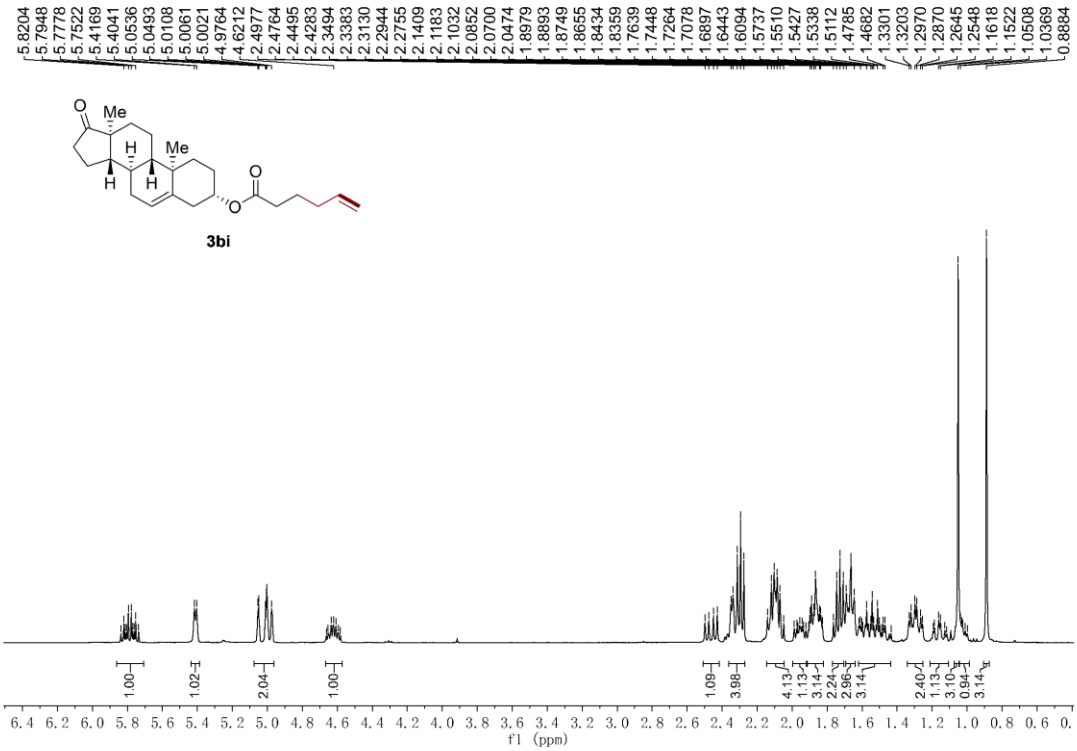
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3bh



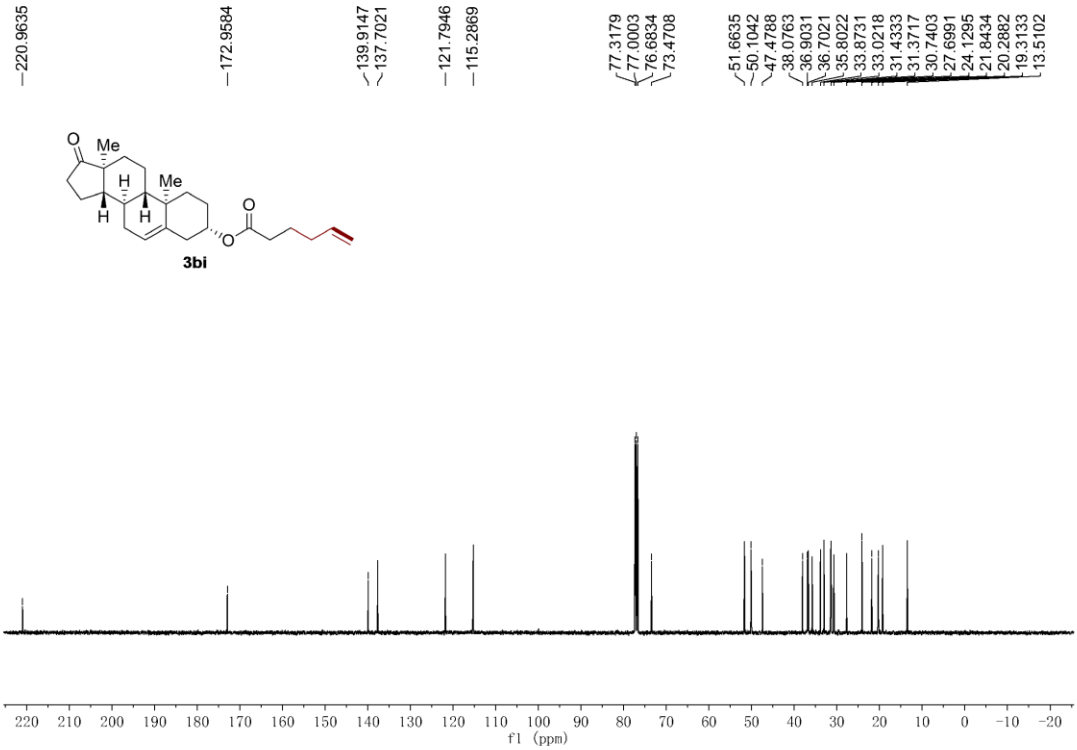
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3bh



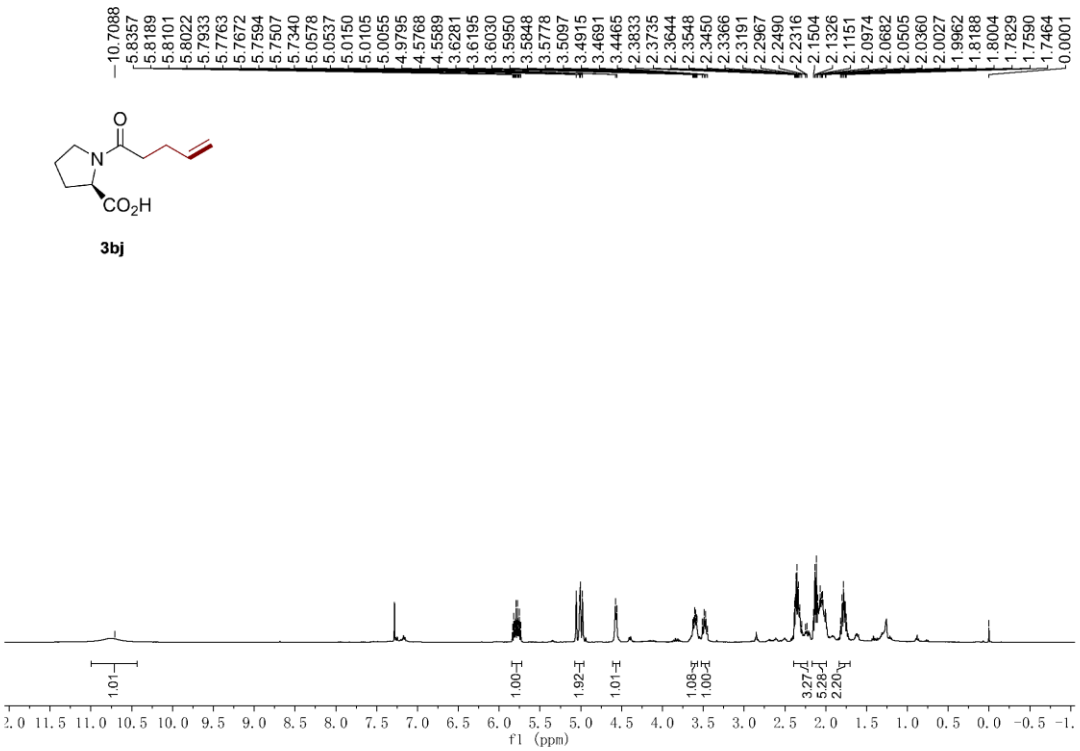
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3bi



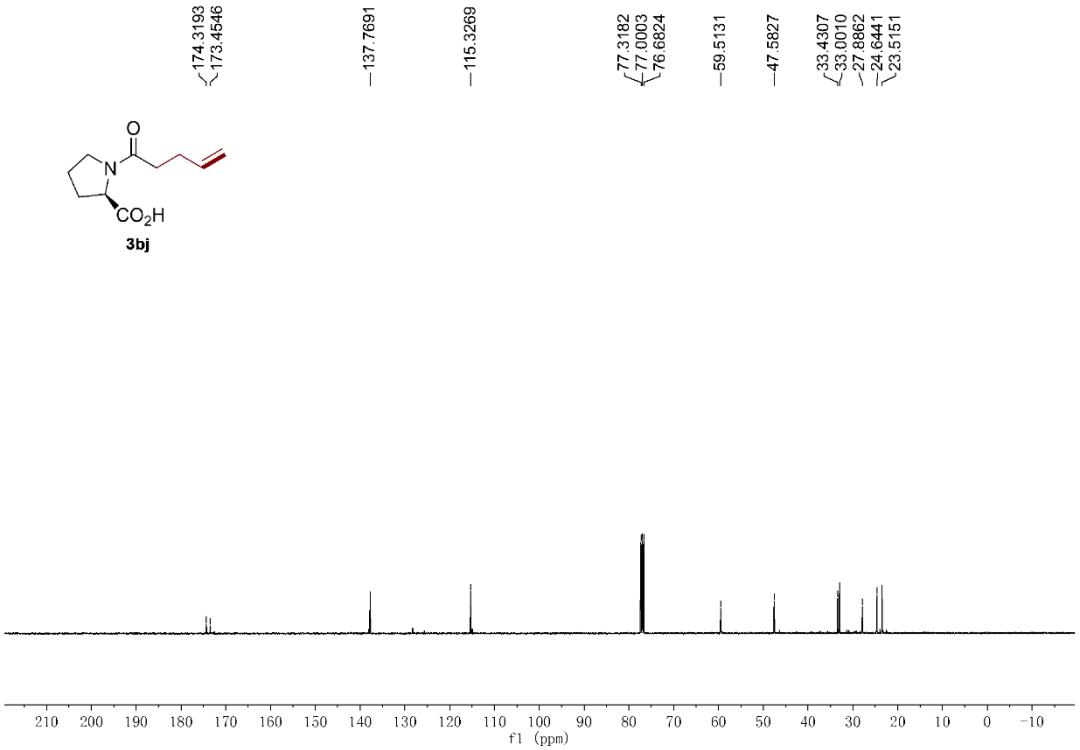
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3bi



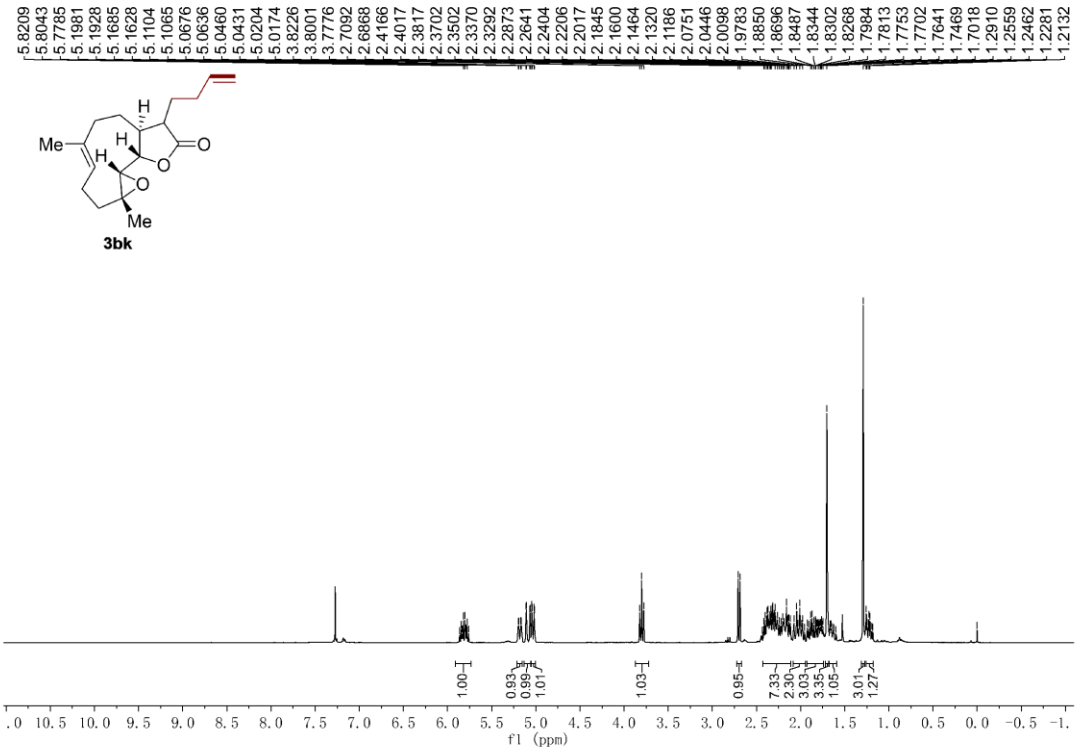
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3bj



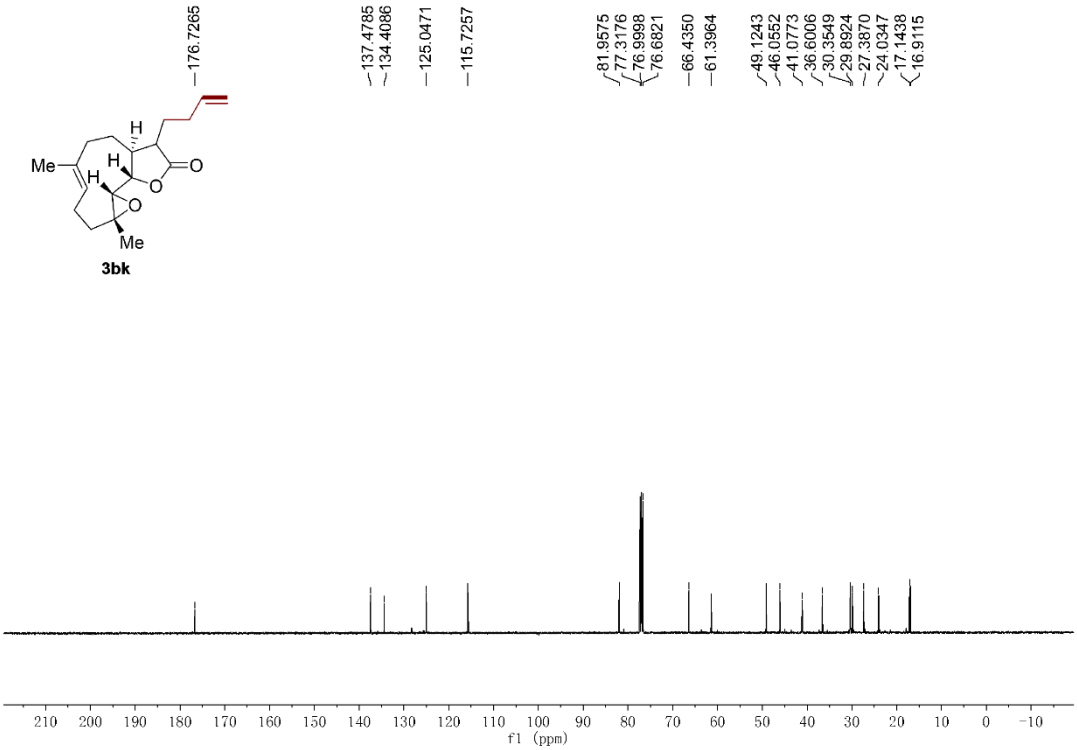
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3bj



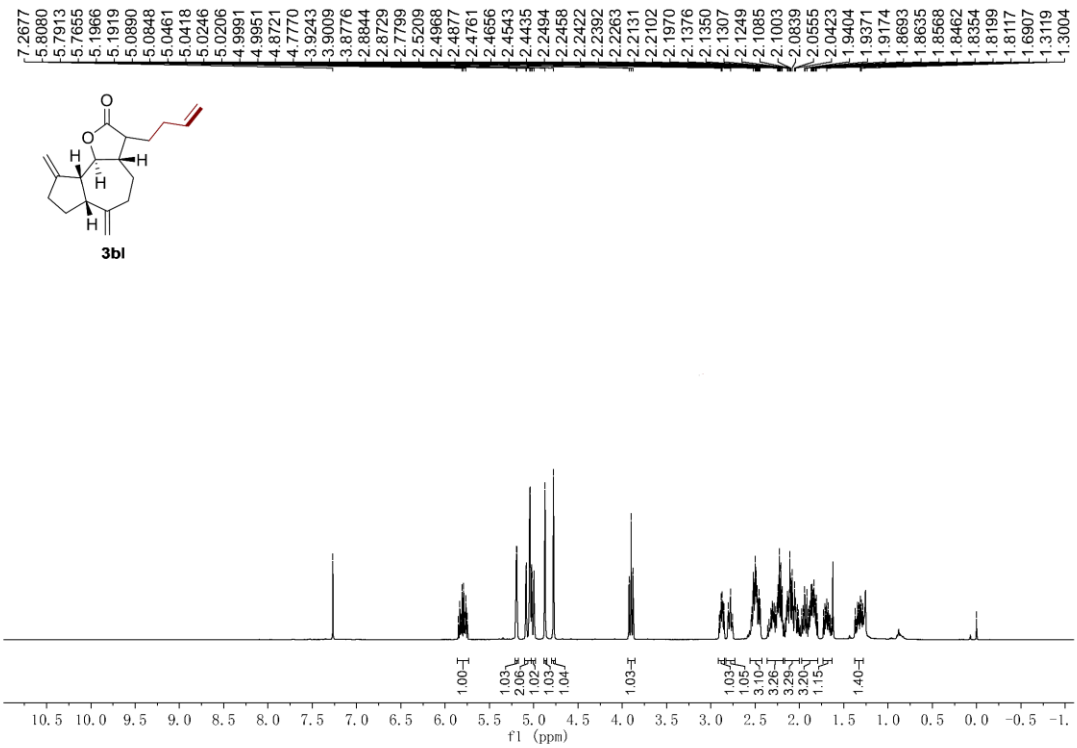
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3bk



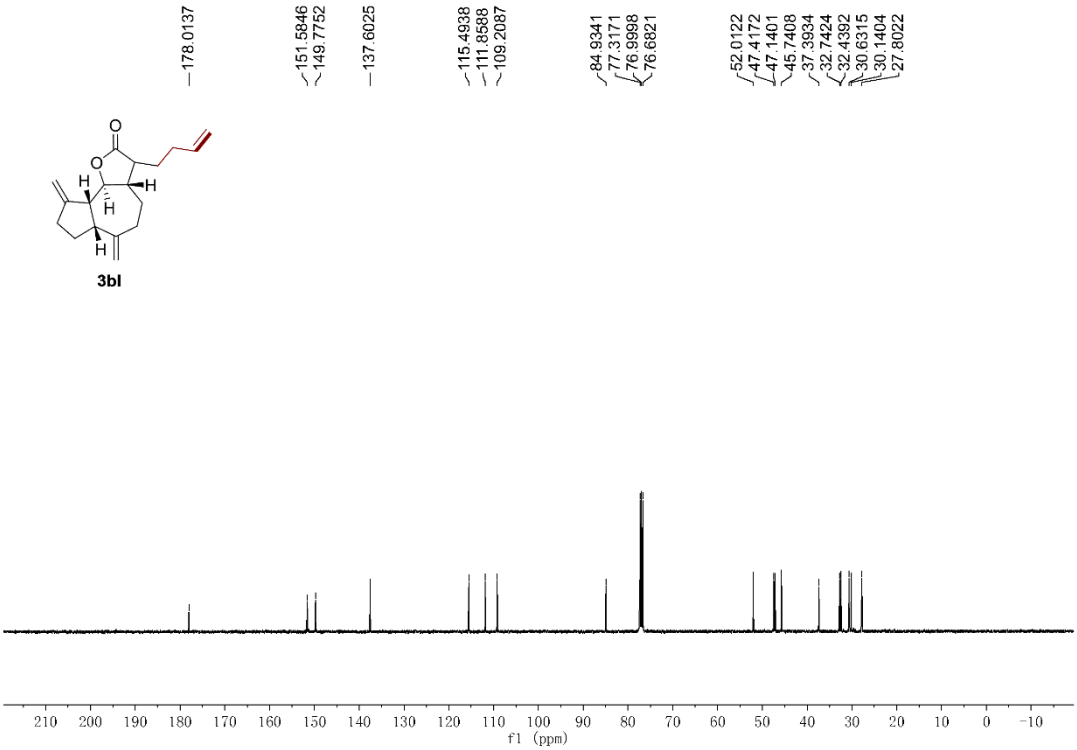
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3bk



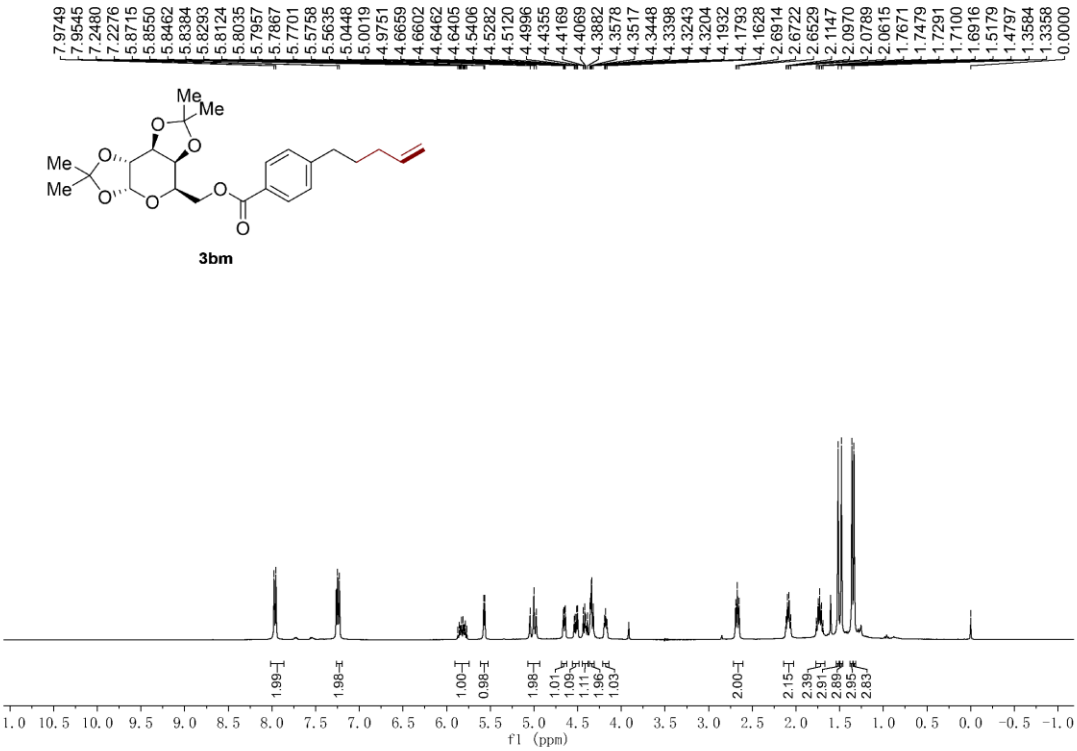
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3bl



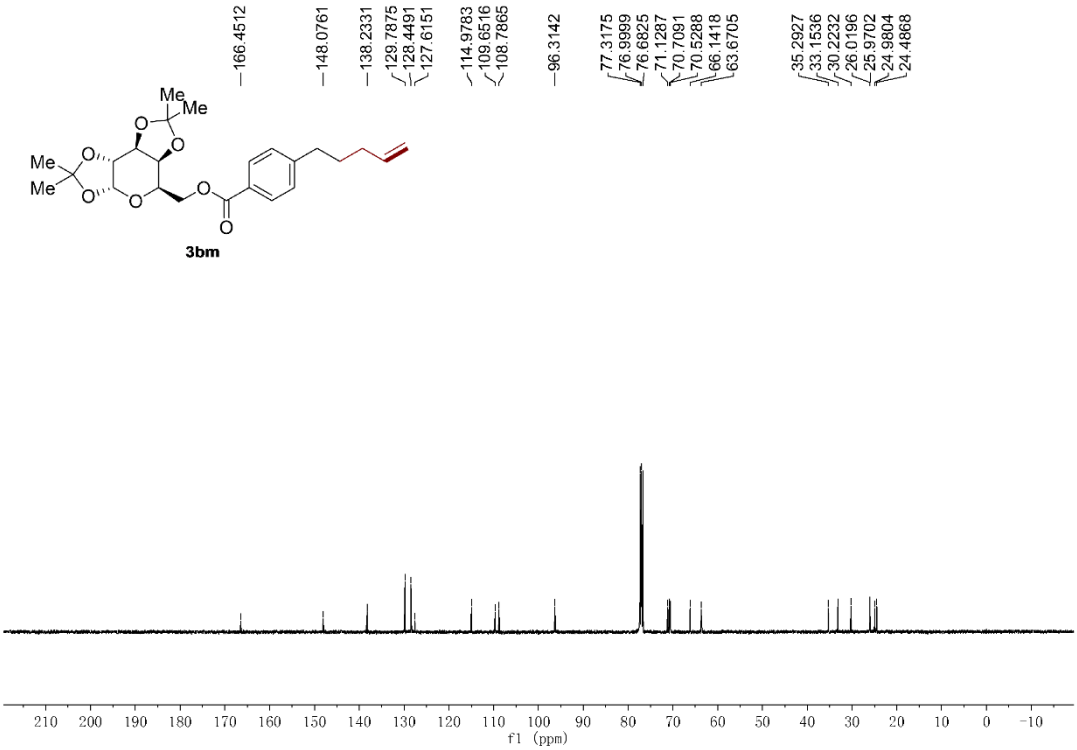
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3bl



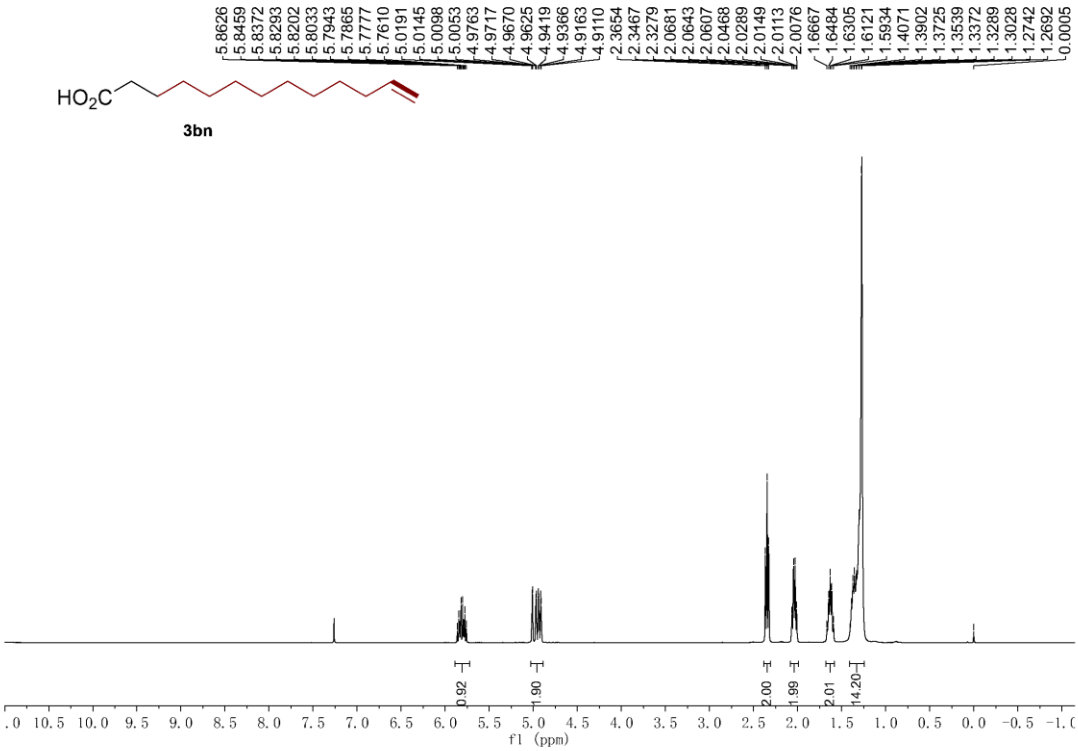
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3bm



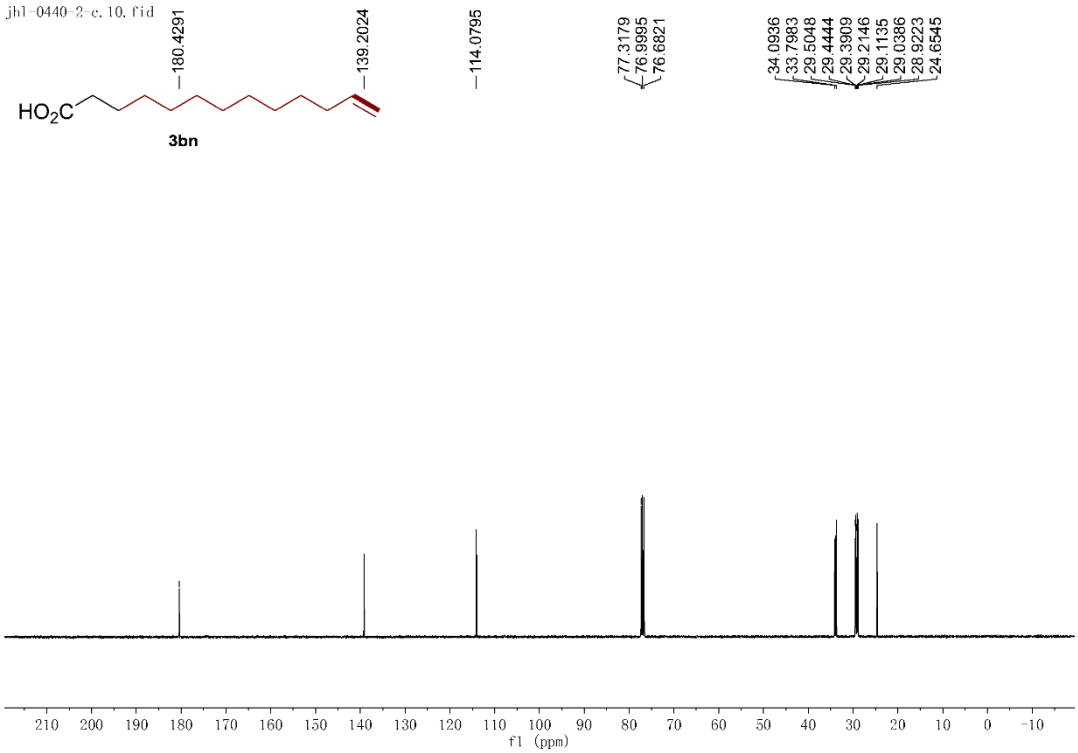
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3bm



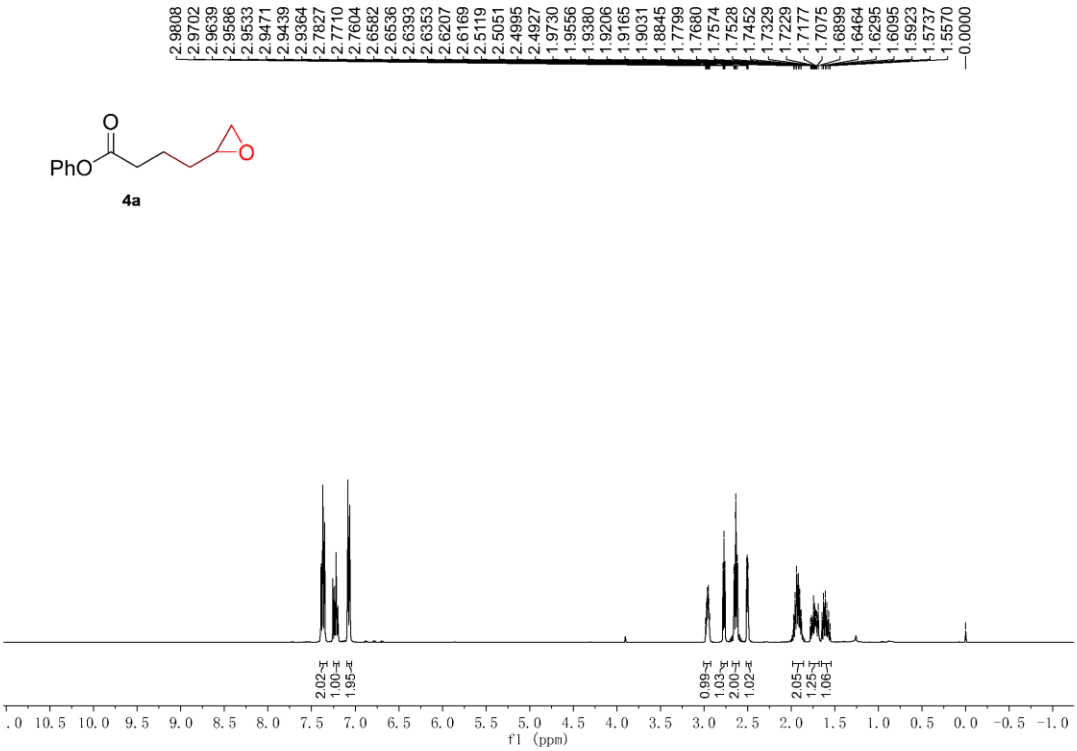
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3bn



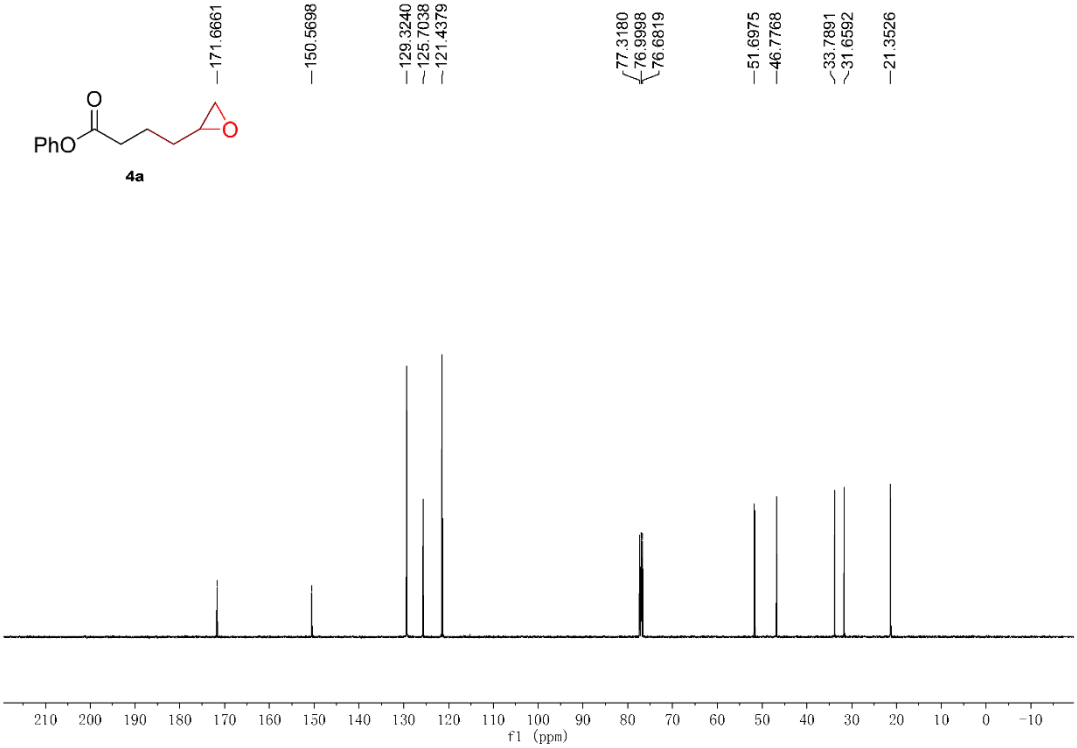
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 3bn



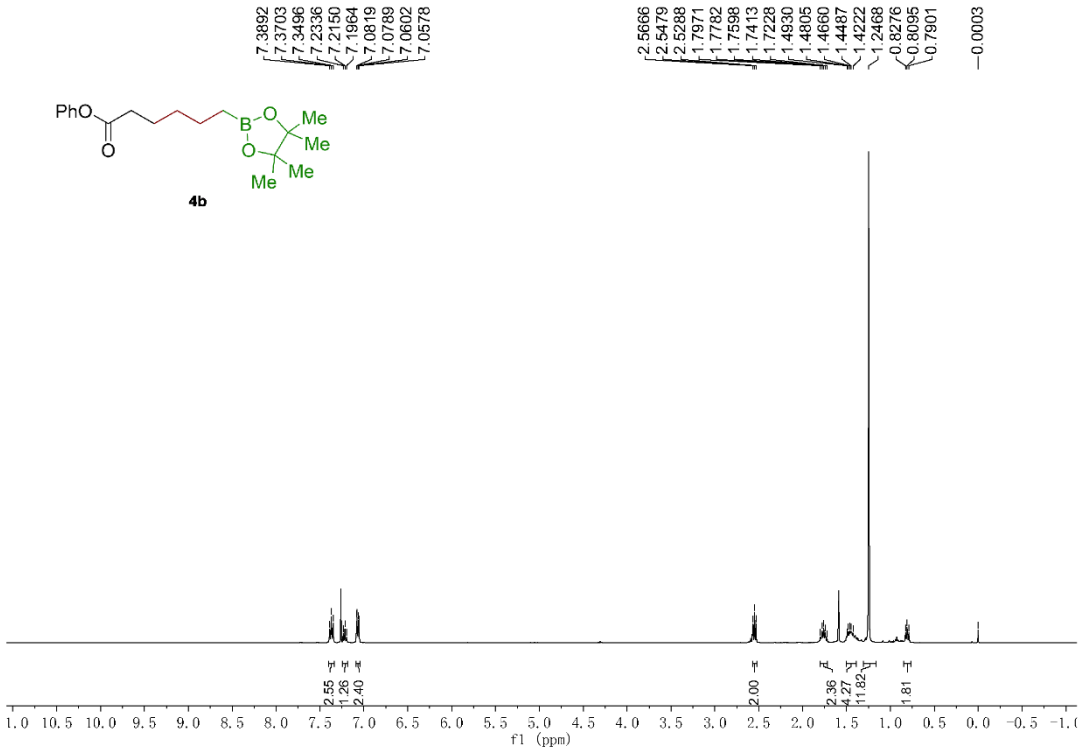
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 4a



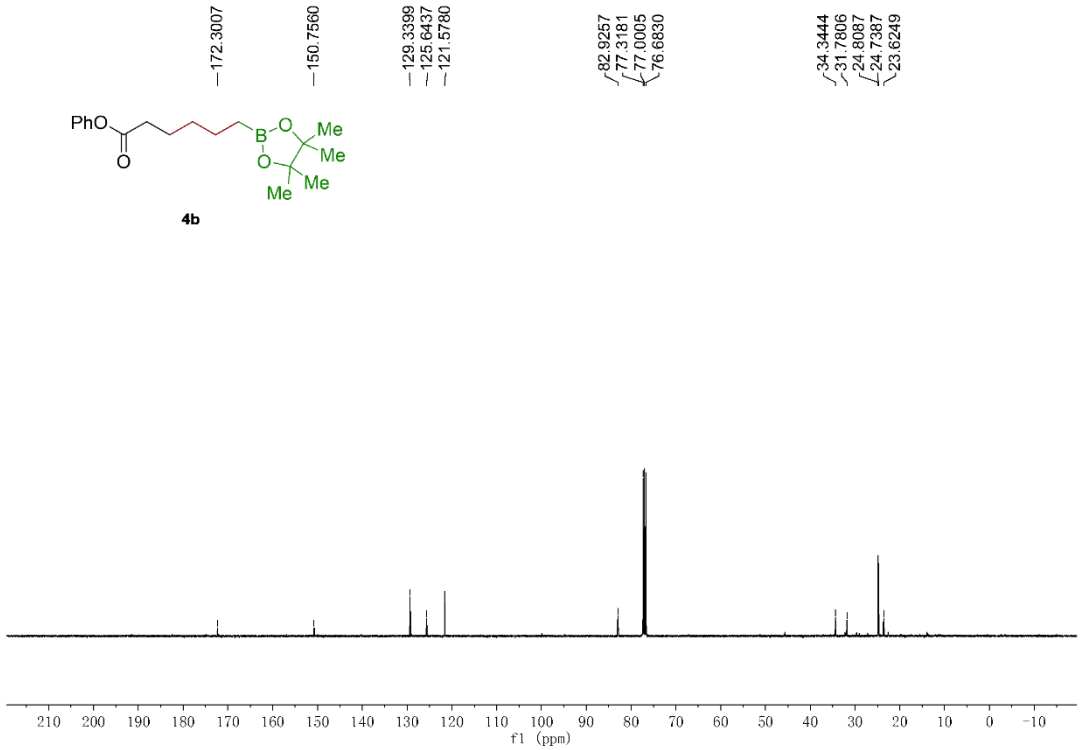
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 4a



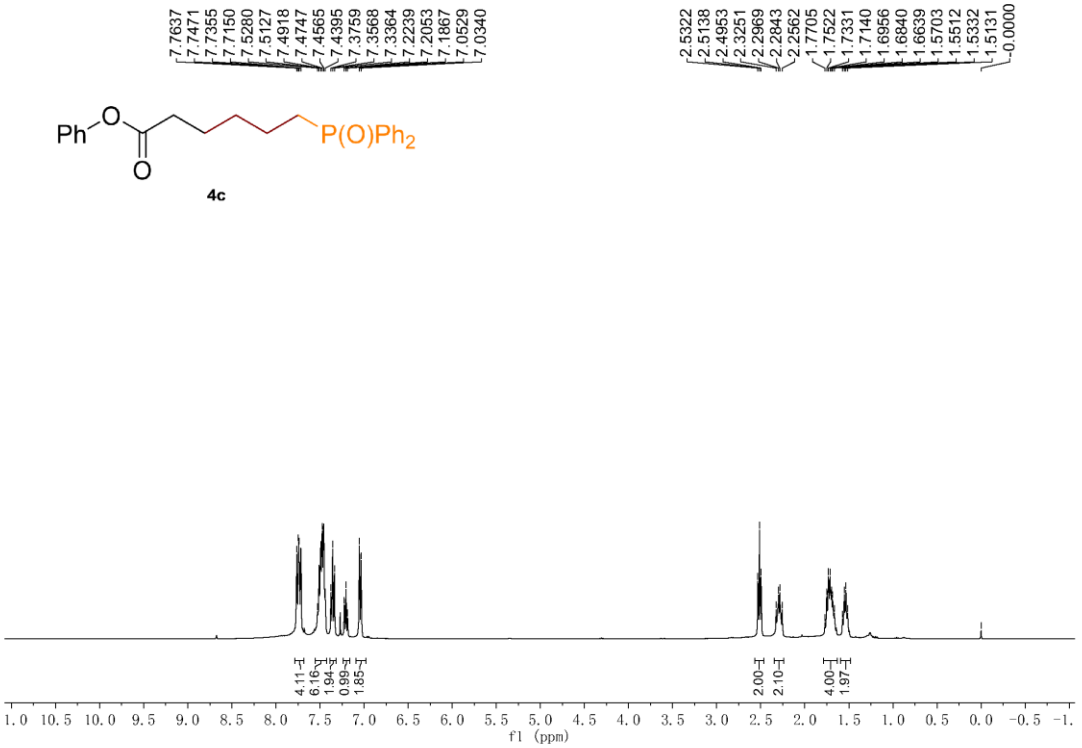
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 4b



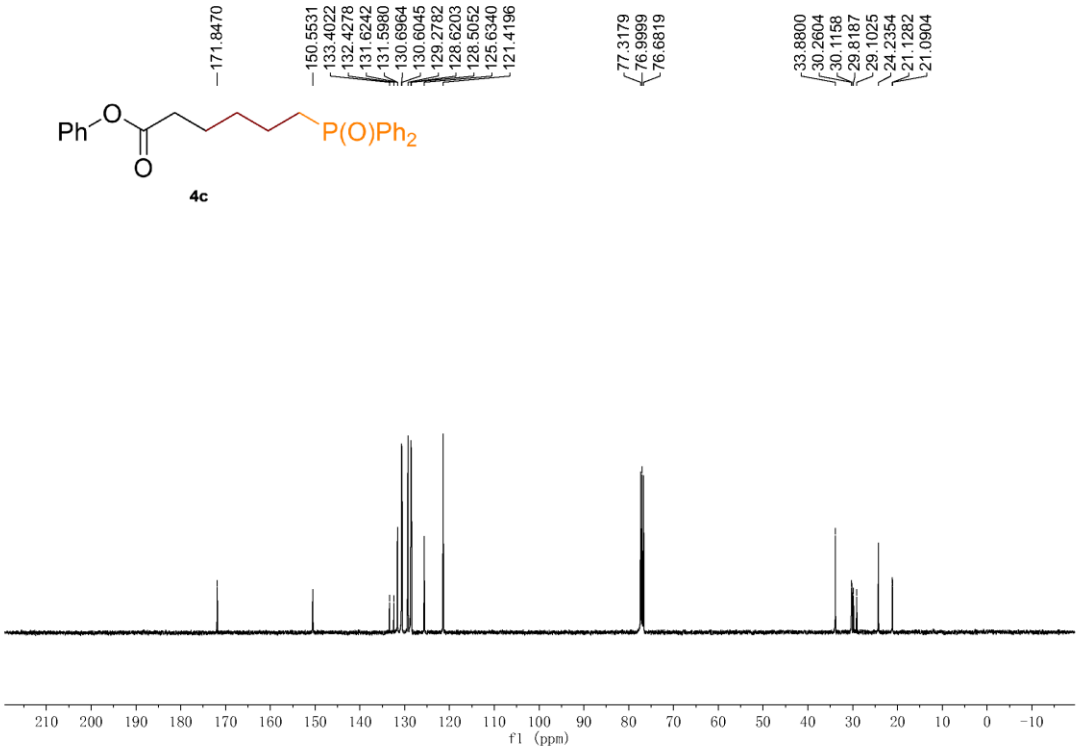
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 4b



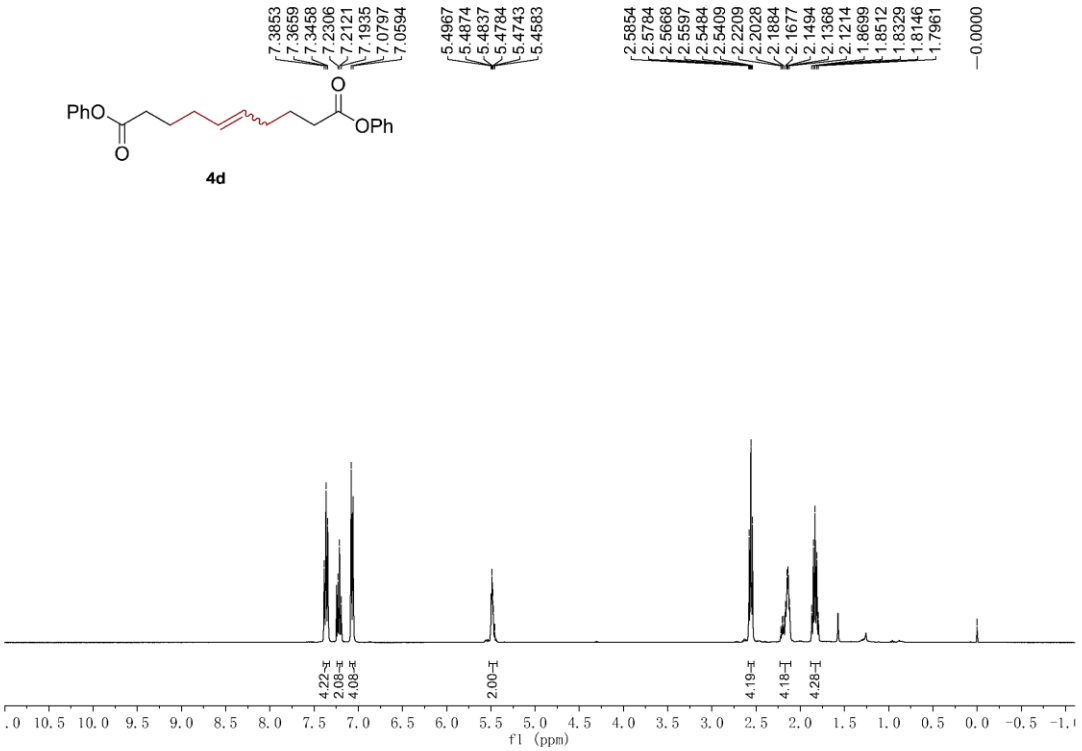
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 4c



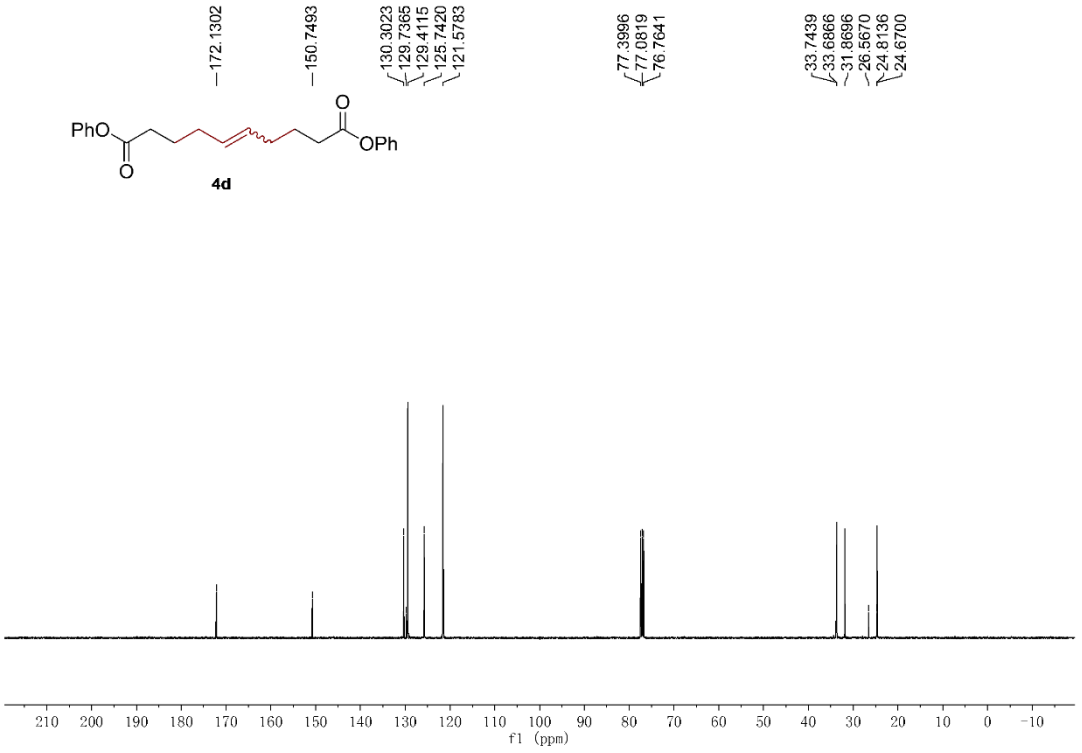
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 4c



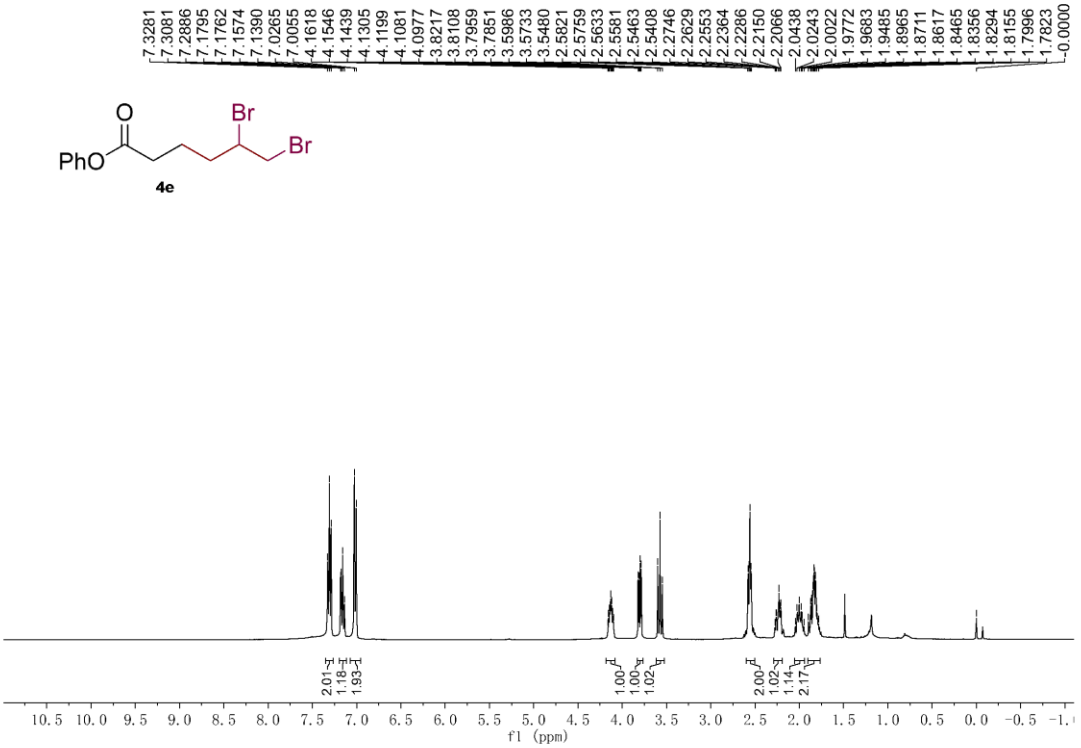
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 4d



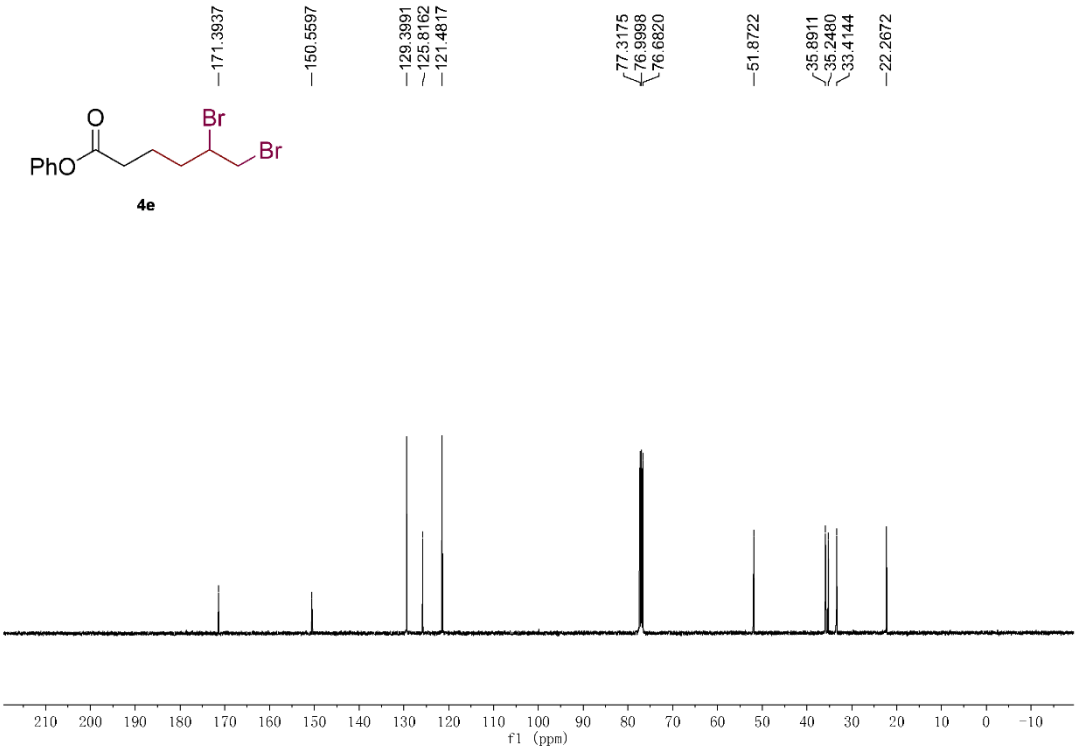
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 4d



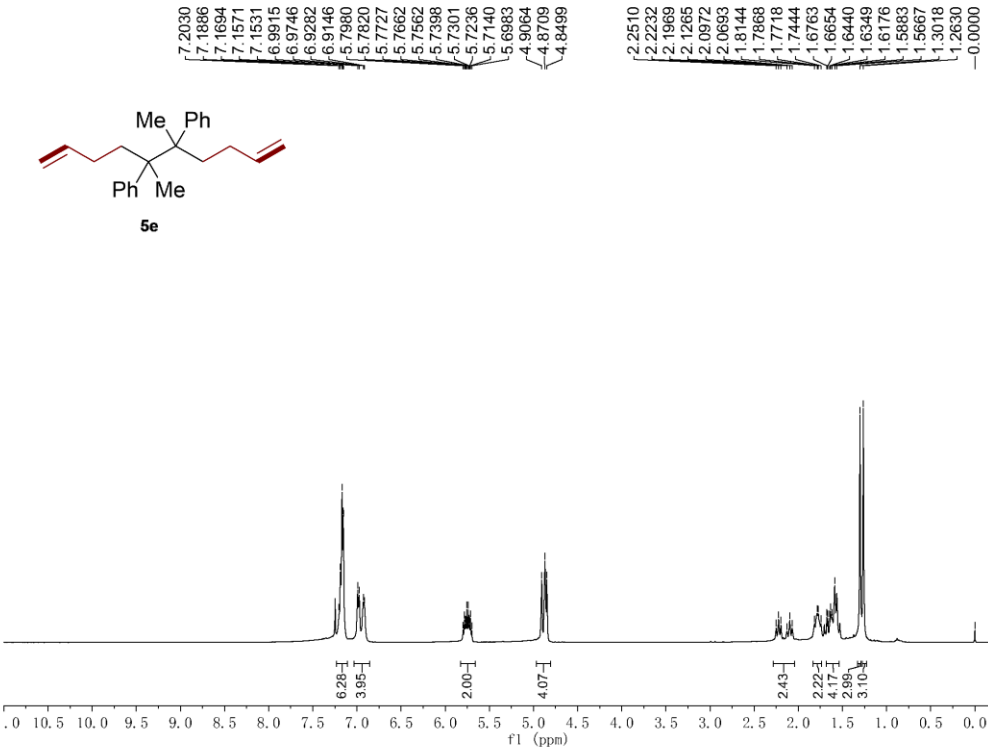
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 4e



^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 4e



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 5e



^{13}C NMR (101 MHz, $CDCl_3$) spectrum of 5e