

*Supporting Information for*

**Practical and robust access to enimides via DMAPO-catalyzed  
coupling of carboxylic acids with secondary enamides**

Xianghe Wu, Feng Yu, Li Zhang and Yewen Fang

## Table of contents

1. General Information.....	S3
1.1 Solvents, Reagents, and Starting Materials.....	S3
1.2 Instruments.....	S3
1.3 Picture of a typical reaction setup for photocatalytic reaction.....	S3
2. General procedure of DMAPO-catalyzed coupling of carboxylic acids with secondary enamides.....	S4
3. Procedure of gram-scale synthesis.....	S18
4. General procedure of photocatalyzed reaction .....	S19
5. References.....	S20
6. NMR Spectra of new compounds .....	S21

# 1. General Information

## 1.1 Solvents, Reagents, and Starting Materials

All reactions were carried out in Schlenk tubes within a N<sub>2</sub> atmosphere. The enamides (**1** and **7**)<sup>1,2</sup> and radical precursors (**11-13**)<sup>3</sup> used in this work have been reported in our previous works. The carboxylic acids, DMAPO, photocatalysts, and other chemicals were purchased from local suppliers.

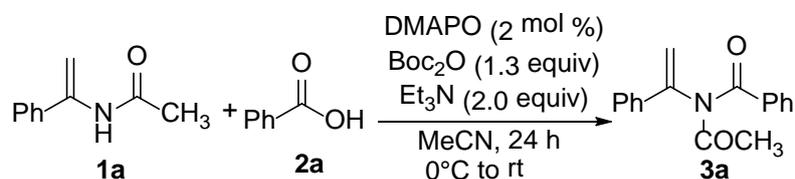
## 1.2 Instruments

NMR spectra were acquired on the Bruker Avance 500 spectrometer (500 MHz). Chemical shifts were reported in ppm downfield from tetramethylsilane, and calibrated using residue undeuterated solvent (CHCl<sub>3</sub> at 7.26 ppm <sup>1</sup>H NMR, 77.0 ppm <sup>13</sup>C NMR). Spectra were reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz). We conducted high-resolution mass spectrometry analysis by using the Agilent 6210 ESI/TOF ESI-Q-TOF spectrometer. TLC analysis was performed on pre-coated GF254 silica gel plates and was visible under UV 254 nm light or I<sub>2</sub> staining. Use 300-400 mesh silica gel for column chromatography and hexane/ethyl acetate for separation, unless otherwise specified.

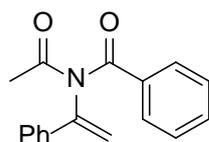
## 1.3 Picture of a typical reaction setup for photocatalytic reaction



## 2. General procedure of DMAPO-catalyzed coupling of carboxylic acids with secondary enamides



To a 10 mL dried Schlenk tube equipped with a magnetic stirrer was added DMAPO (5.5 mg, 0.04 mmol, 0.02 equiv), *N*-(1-phenylvinyl)acetamide **1a** (322.2 mg, 2.0 mmol, 1.0 equiv), and benzoic acid **2a** (2.0 mmol, 1.0 equiv). The reaction tube was allowed to be vacuumed and purged with nitrogen for three times. Then,  $\text{CH}_3\text{CN}$  (6.0 mL) was added by syringe under nitrogen atmosphere. After the reaction mixture was cooled to 0 °C,  $\text{Et}_3\text{N}$  (0.56 mL, 4.0 mmol, 2.0 equiv) and  $\text{Boc}_2\text{O}$  (0.60 mL, 2.6 mmol, 1.3 equiv) were successively added dropwise to the reaction mixture and the final solution was stirred for 24 h at room temperature. After the reaction was complete, the reaction solution was quenched by adding water (5 mL). The aqueous layer was extracted with ethyl acetate ( $3 \times 10$  mL), and the combined organic layers were washed with brine, dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and concentrated under reduced pressure. The crude product was purified by column chromatography over silica gel with hexane/ethyl acetate as the eluent.

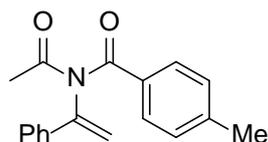


***N*-Benzoyl-*N*-(1-phenylethenyl)acetamide (3a).** White solid (456.0 mg, 86% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.71 – 7.64 (m, 2H), 7.52 – 7.43 (m, 3H), 7.40 – 7.30 (m, 5H), 5.80 (d,  $J = 1.2$  Hz, 1H), 5.22 (d,  $J = 1.3$  Hz, 1H), 2.42 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  173.7, 173.0, 145.0, 136.0, 134.9, 132.1, 129.0, 128.9, 128.4, 128.3, 125.5, 115.6, 25.4.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{17}\text{H}_{15}\text{NO}_2\text{Na}$ : 288.1000, found 288.1003.

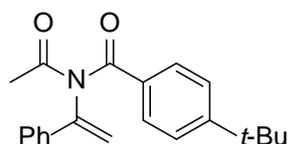


***N*-(4-Methylbenzoyl)-*N*-(1-phenylethenyl)acetamide (3b).** White solid (267.9 mg, 48% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.60 (d, *J* = 8.2 Hz, 2H), 7.51 – 7.46 (m, 2H), 7.39 – 7.30 (m, 3H), 7.16 (d, *J* = 7.9 Hz, 2H), 5.78 (d, *J* = 1.1 Hz, 1H), 5.19 (d, *J* = 1.2 Hz, 1H), 2.39 (s, 3H), 2.37 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 173.8, 172.9, 145.1, 143.1, 136.0, 131.9, 129.0, 128.9, 128.8, 128.7, 125.5, 115.3, 25.3, 21.6.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>18</sub>H<sub>17</sub>NO<sub>2</sub>Na: 302.1157, found 302.1159.

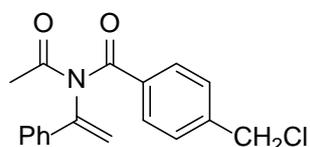


***N*-Acetyl-4-(*tert*-butyl)-*N*-(1-phenylvinyl)benzamide (3c).** White solid (398.3 mg, 62% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.68 – 7.63 (m, 2H), 7.52 – 7.46 (m, 2H), 7.40 – 7.31 (m, 5H), 5.78 (d, *J* = 1.2 Hz, 1H), 5.19 (d, *J* = 1.2 Hz, 1H), 2.39 (s, 3H), 1.31 (s, 9H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 173.8, 172.8, 156.1, 145.1, 136.1, 131.7, 128.9, 128.8, 128.7, 125.6, 125.3, 115.3, 35.0, 31.0, 25.3.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>21</sub>H<sub>23</sub>NO<sub>2</sub>Na: 344.1626, found 344.1630.

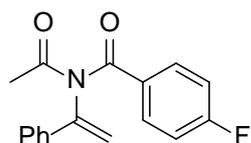


***N*-(4-(Chloromethyl)benzoyl)-*N*-(1-phenylethenyl)acetamide (3d).** White solid (419.4 mg, 67% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.66 (d, *J* = 8.4 Hz, 2H), 7.51 – 7.43 (m, 2H), 7.41 – 7.31 (m, 5H), 5.82 (d, *J* = 1.2 Hz, 1H), 5.22 (d, *J* = 1.2 Hz, 1H), 4.57 (s, 2H), 2.42 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 173.7, 172.3, 144.8, 141.4, 135.9, 134.8, 129.1, 128.9, 128.8, 128.4, 125.5, 115.7, 45.2, 25.3.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>18</sub>H<sub>16</sub>NO<sub>2</sub>NaCl: 336.0767, found 336.0768.



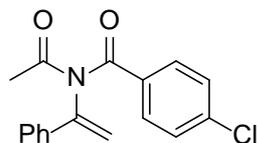
**N-(4-Fluorobenzoyl)-N-(1-phenylethenyl)acetamide (3e)**. Yellow solid (486.9 mg, 86% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.73 – 7.65 (m, 2H), 7.48 – 7.43 (m, 2H), 7.39 – 7.31 (m, 3H), 7.03 (t, *J* = 8.6 Hz, 2H), 5.82 (d, *J* = 1.2 Hz, 1H), 5.22 (d, *J* = 1.2 Hz, 1H), 2.41 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 173.6, 171.7, 164.9 (d, *J* = 254.3 Hz), 144.9, 135.8, 131.0 (d, *J* = 9.2 Hz), 130.9 (d, *J* = 2.7 Hz), 129.0, 128.9, 125.4, 115.5 (d, *J* = 3.6 Hz), 115.3, 25.2.

**<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)** δ -106.0.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>17</sub>H<sub>14</sub>NO<sub>2</sub>NaF: 306.0906, found 306.0911.

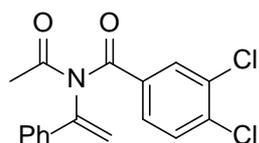


**N-(4-Chlorobenzoyl)-N-(1-phenylethenyl)acetamide (3f)**. White solid (514.4 mg, 86% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.77 – 7.55 (m, 2H), 7.55 – 7.42 (m, 2H), 7.41 – 7.29 (m, 5H), 5.82 (d, *J* = 1.2 Hz, 1H), 5.22 (d, *J* = 1.2 Hz, 1H), 2.41 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 173.6, 171.9, 144.8, 138.4, 135.8, 133.3, 129.8, 129.1, 128.9, 128.6, 125.4, 115.7, 25.3.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>17</sub>H<sub>14</sub>NO<sub>2</sub>NaCl: 322.0611, found 322.0613.

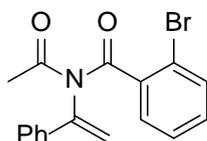


**N-Acetyl-3,4-dichloro-N-(1-phenylvinyl)benzamide (3g)**. White solid (539.5 mg, 81% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.73 (d, *J* = 1.9 Hz, 1H), 7.46 – 7.40 (m, 4H), 7.40 – 7.32 (m, 3H), 5.86 (d, *J* = 1.3 Hz, 1H), 5.25 (d, *J* = 1.2 Hz, 1H), 2.40 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  173.4, 170.6, 144.6, 136.4, 135.6, 134.7, 132.8, 130.2(9), 130.2(7), 129.3, 129.0, 127.2, 125.4, 116.0, 25.1.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{17}\text{H}_{13}\text{NO}_2\text{NaCl}_2$ : 356.0221, found 356.0222.

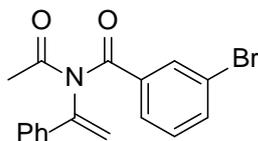


***N*-(2-Bromobenzoyl)-*N*-(1-phenylethenyl)acetamide (3h)**. White solid (356.7 mg, 52% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.53 – 7.50 (m, 1H), 7.38 – 7.31 (m, 5H), 7.24 – 7.16 (m, 2H), 7.08 – 7.02 (m, 1H), 5.86 (d,  $J = 1.1$  Hz, 1H), 5.58 (d,  $J = 1.1$  Hz, 1H), 2.48 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  172.8, 170.4, 143.8, 138.4, 136.0, 132.6, 130.7, 129.1, 128.8, 127.8, 127.0, 125.4, 118.8, 116.1, 25.8.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{17}\text{H}_{14}\text{NO}_2\text{NaBr}$ : 366.0106, found 366.0106.

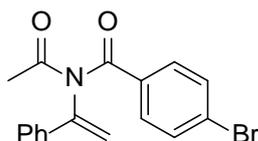


***N*-(3-Bromobenzoyl)-*N*-(1-phenylethenyl)acetamide (3i)**. White solid (507.6 mg, 74% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.77 (s, 1H), 7.61 – 7.56 (m, 1H), 7.54 (d,  $J = 7.9$  Hz, 1H), 7.49 – 7.42 (m, 2H), 7.41 – 7.32 (m, 3H), 7.22 (t,  $J = 7.9$  Hz, 1H), 5.84 (d,  $J = 1.2$  Hz, 1H), 5.24 (d,  $J = 1.2$  Hz, 1H), 2.42 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  173.5, 171.4, 144.7, 136.9, 135.8, 134.8, 131.3, 129.7, 129.2, 129.0, 126.6, 125.4, 122.3, 115.9, 25.3.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{17}\text{H}_{14}\text{NO}_2\text{NaBr}$ : 366.0106, found 366.0109.



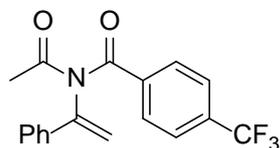
***N*-(4-Bromobenzoyl)-*N*-(1-phenylethenyl)acetamide (3j)**. White solid (535.1 mg, 78% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.53 – 7.48 (m, 4H), 7.46 – 7.43 (m, 2H), 7.39 – 7.33

(m, 3H), 5.83 (d,  $J = 1.2$  Hz, 1H), 5.21 (d,  $J = 1.2$  Hz, 1H), 2.41 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  173.6, 172.0, 144.8, 135.8, 133.7, 131.6, 129.9, 129.2, 129.0, 126.9, 125.4, 115.8, 25.3.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{17}\text{H}_{14}\text{NO}_2\text{NaBr}$ : 366.0106, found 366.0109.



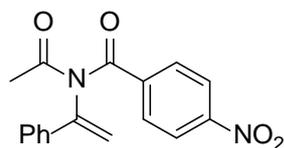
*N*-(1-Phenylethenyl)-*N*-(4-(trifluoromethyl)benzoyl)acetamide (**3k**). Yellow solid (599.6 mg, 90% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.72 (d,  $J = 8.0$  Hz, 2H), 7.62 (d,  $J = 8.1$  Hz, 2H), 7.48 – 7.43 (m, 2H), 7.41 – 7.32 (m, 3H), 5.87 (d,  $J = 1.3$  Hz, 1H), 5.26 (d,  $J = 1.3$  Hz, 1H), 2.43 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  173.5, 171.6, 144.5, 138.5, 135.6, 133.3 (q,  $J = 32.9$  Hz), 129.3, 129.0, 128.4, 125.4, 125.3 (q,  $J = 3.7$  Hz), 123.5 (q,  $J = 273.6$  Hz), 116.1, 25.3.

$^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ )  $\delta$  -63.1.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{18}\text{H}_{14}\text{NO}_2\text{F}_3\text{Na}$ : 356.0874, found 356.0872.

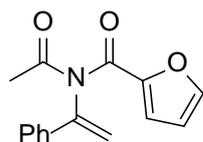


*N*-(4-Nitrobenzoyl)-*N*-(1-phenylethenyl)acetamide (**3l**). Yellow solid (421.6 mg, 68% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.23 – 8.19 (m, 2H), 7.73 (d,  $J = 8.6$  Hz, 2H), 7.46 – 7.42 (m, 2H), 7.40 – 7.37 (m, 2H), 5.91 (d,  $J = 1.1$  Hz, 1H), 5.30 (d,  $J = 1.3$  Hz, 1H), 2.43 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  173.3, 170.9, 149.3, 144.3, 141.0, 135.4, 129.5, 129.1, 128.8, 125.3, 123.5, 116.5, 25.2.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{17}\text{H}_{14}\text{N}_2\text{O}_4\text{Na}$ : 333.0851, found 338.0852.

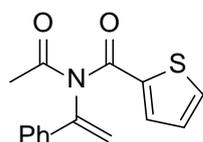


***N*-Acetyl-*N*-(1-phenylvinyl)furan-2-carboxamide (3m).** White solid (345.0 mg, 68% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.92–7.91 (m, 1H), 7.52 – 7.43 (m, 2H), 7.40 – 7.31 (m, 3H), 7.30 (t, *J* = 1.8 Hz, 1H), 6.60 (dd, *J* = 2.0, 0.9 Hz, 1H), 5.88 (d, *J* = 1.2 Hz, 1H), 5.27 (d, *J* = 1.2 Hz, 1H), 2.51 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 173.4, 165.6, 147.9, 144.4, 143.0, 135.6, 129.1, 129.0, 125.2, 122.1, 115.8, 110.4, 25.9.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>15</sub>H<sub>13</sub>NO<sub>3</sub>Na: 278.0793, found 278.0797.

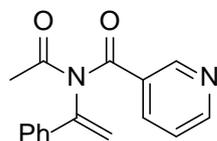


***N*-(1-Phenylethenyl)-*N*-(thiophen-2-ylcarbonyl)acetamide (3n).** Yellow solid (336.1 mg, 62% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.66 (dd, *J* = 3.8, 1.2 Hz, 1H), 7.55 (dd, *J* = 5.0, 1.2 Hz, 1H), 7.53 – 7.49 (m, 2H), 7.40 – 7.31 (m, 3H), 6.99 (dd, *J* = 5.0, 3.9 Hz, 1H), 5.86 (d, *J* = 1.2 Hz, 1H), 5.31 (d, *J* = 1.3 Hz, 1H), 2.49 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 173.5, 165.5, 144.9, 137.6, 135.8, 133.6, 133.5, 129.0, 128.9, 127.6, 125.5, 115.8, 25.6.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>15</sub>H<sub>13</sub>NO<sub>2</sub>NaS: 294.0565, found 294.0568.

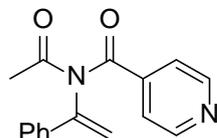


***N*-(1-Phenylethenyl)-*N*-(pyridin-3-ylcarbonyl)acetamide (3o).** Yellow solid (324.6 mg, 61% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 8.86 (d, *J* = 2.3 Hz, 1H), 8.67 (dd, *J* = 4.9, 1.7 Hz, 1H), 7.90 (dt, *J* = 7.9, 2.0 Hz, 1H), 7.46 – 7.41 (m, 2H), 7.39 – 7.32 (m, 3H), 7.30 (dd, *J* = 7.9, 4.8 Hz, 1H), 5.88 (d, *J* = 1.4 Hz, 1H), 5.28 (d, *J* = 1.2 Hz, 1H), 2.44 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  173.5, 170.9, 152.3, 149.0, 144.5, 135.7(0), 135.6(5), 131.1, 129.3, 129.0, 125.4, 123.1, 116.3, 25.3.

HRMS (ESI)  $[\text{M}+\text{H}]^+$ : calculated for  $\text{C}_{16}\text{H}_{15}\text{N}_2\text{O}_2$ : 267.1134, found 267.1136.

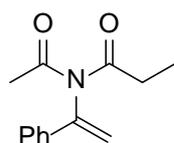


***N*-(1-Phenylethenyl)-*N*-(pyridin-4-ylcarbonyl)acetamide (3p)**. Yellow solid (330.1 mg, 62% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.70 – 8.58 (m, 2H), 7.44 – 7.41 (m, 2H), 7.41 – 7.33 (m, 5H), 5.88 (d,  $J = 1.3$  Hz, 1H), 5.26 (d,  $J = 1.3$  Hz, 1H), 2.44 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  173.2, 170.9, 150.1, 144.1, 142.6, 135.4, 129.4, 129.1, 125.3, 121.2, 116.5, 25.3.

HRMS (ESI)  $[\text{M}+\text{H}]^+$ : calculated for  $\text{C}_{16}\text{H}_{15}\text{N}_2\text{O}_2$ : 267.1134, found 267.1137.

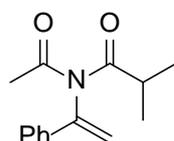


***N*-(1-Phenylethenyl)-*N*-propanoylacetamide (5a)**. Yellow solid (325.6 mg, 75% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.42 – 7.36 (m, 5H), 6.02 (d,  $J = 1.1$  Hz, 1H), 5.30 (d,  $J = 1.1$  Hz, 1H), 2.70 (q,  $J = 7.2$  Hz, 2H), 2.45 (s, 3H), 1.11 (t,  $J = 7.3$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  176.4, 172.8, 144.3, 135.3, 129.1, 129.0, 124.9, 115.6, 31.1, 26.5, 9.0.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{13}\text{H}_{15}\text{NO}_2\text{Na}$ : 240.1000, found 240.1000.

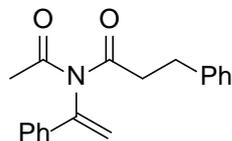


***N*-(2-Methylpropanoyl)-*N*-(1-phenylethenyl)acetamide (5b)**. White solid (270.1 mg, 58% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.42 – 7.39 (m, 2H), 7.38 – 7.33 (m, 3H), 5.98 (d,  $J = 1.0$  Hz, 1H), 5.28 (d,  $J = 1.0$  Hz, 1H), 3.22 – 3.16 (m, 1H), 2.43 (s, 3H), 1.14 (d,  $J = 6.7$  Hz, 6H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  180.5, 173.1, 144.3, 135.5, 129.1, 128.9, 125.0, 115.1, 35.2, 26.3, 19.7.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{14}\text{H}_{17}\text{NO}_2\text{Na}$ : 254.1157, found 254.1160.

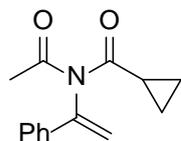


***N*-(1-Phenylethenyl)-*N*-(3-phenylpropanoyl)acetamide (5c)**. Yellow oil (392.8 mg, 67% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35 – 7.29 (m, 5H), 7.29 – 7.24 (m, 2H), 7.22 – 7.14 (m, 3H), 5.98 (d,  $J = 0.92$  Hz, 1H), 5.21 (d,  $J = 0.89$  Hz, 1H), 3.07 – 3.00 (m, 2H), 3.00 – 2.94 (m, 2H), 2.41 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  174.8, 172.8, 144.1, 140.5, 135.1, 129.1, 128.9, 128.5, 128.4, 126.2, 124.9, 115.7, 39.4, 30.9, 26.3.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{19}\text{H}_{19}\text{NO}_2\text{Na}$ : 316.1313, found 316.1316.

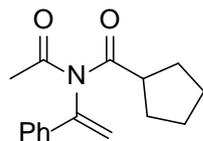


***N*-(Cyclopropylcarbonyl)-*N*-(1-phenylethenyl)acetamide (5d)**. White solid (339.1 mg, 74% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.49 – 7.44 (m, 2H), 7.40 – 7.31 (m, 3H), 6.01 (d,  $J = 1.0$  Hz, 1H), 5.35 (d,  $J = 1.0$  Hz, 1H), 2.49 (s, 3H), 2.17 – 2.09 (m, 1H), 1.12 – 1.06 (m, 2H), 0.88 – 0.81 (m, 2H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  176.6, 172.8, 144.2, 135.7, 129.0, 128.9, 125.0, 115.6, 26.5, 15.5, 11.0.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{14}\text{H}_{15}\text{NO}_2\text{Na}$ : 252.1000, found 252.1005.

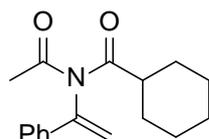


***N*-(Cyclopentylcarbonyl)-*N*-(1-phenylethenyl)acetamide (5e)**. Yellow oil (365.1 mg, 71% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.43 – 7.39 (m, 2H), 7.39 – 7.32 (m, 3H), 5.99 (d, *J* = 0.9 Hz, 1H), 5.27 (d, *J* = 0.9 Hz, 1H), 3.22 (p, 1H), 2.46 (s, 3H), 1.86– 1.77 (m, 4H), 1.76 – 1.69 (m, 2H), 1.56 – 1.48 (m, 2H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 179.9, 173.1, 144.3, 135.6, 129.1, 128.9, 125.0, 115.3, 45.6, 31.5, 26.5, 26.3.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>16</sub>H<sub>19</sub>NO<sub>2</sub>Na: 280.1313, found 280.1317.

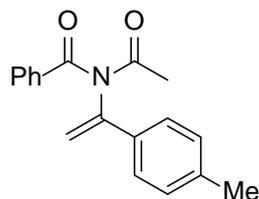


***N*-Acetyl-*N*-(1-phenylvinyl)cyclohexanecarboxamide (5f)**. Yellow oil (352.5 mg, 65% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.43 – 7.31 (m, 5H), 5.96 (d, *J* = 1.0 Hz, 1H), 5.26 (d, *J* = 0.9 Hz, 1H), 2.98 – 2.92 (m, 1H), 2.40 (s, 3H), 1.83 – 1.79 (m, 2H), 1.76 – 1.71 (m, 2H), 1.66 – 1.63 (m, 1H), 1.53 – 1.44 (m, 2H), 1.21 – 1.14 (m, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 179.4, 173.1, 144.4, 135.7, 129.1, 128.9, 125.1, 115.1, 45.2, 29.7, 26.3, 25.6, 25.4.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>17</sub>H<sub>21</sub>NO<sub>2</sub>Na: 294.1470, found 294.1475.

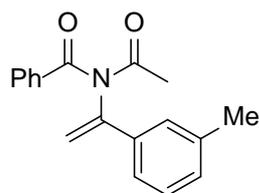


***N*-Benzoyl-*N*-(1-(4-methylphenyl)ethenyl)acetamide (6a)**. White solid (323.8 mg, 58% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.70 – 7.65 (m, 2H), 7.50 – 7.44 (m, 1H), 7.40 – 7.33 (m, 4H), 7.17 (d, *J* = 7.9 Hz, 2H), 5.75 (d, *J* = 0.9 Hz, 1H), 5.15 (d, *J* = 0.9 Hz, 1H), 2.41 (s, 3H), 2.35 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 173.8, 172.9, 144.9, 139.0, 134.9, 133.1, 132.1, 129.6, 128.4, 128.2, 125.4, 114.6, 25.3, 21.1.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>18</sub>H<sub>17</sub>NO<sub>2</sub>Na: 302.1157, found 302.1160.

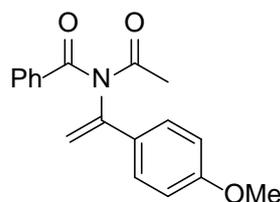


***N*-Benzoyl-*N*-(1-(3-methylphenyl)ethenyl)acetamide (6b).** White solid (362.9 mg, 65% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.68 (d, *J* = 8.0 Hz, 2H), 7.48 (t, *J* = 6.9 Hz, 1H), 7.36 (t, *J* = 7.7 Hz, 2H), 7.26 – 7.22 (m, 3H), 7.15 (d, *J* = 6.7 Hz, 1H), 5.78 (s, 1H), 5.19 (s, 1H), 2.41 (s, 3H), 2.36 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 173.7, 172.9, 145.1, 138.5, 136.0, 134.9, 132.1, 129.8, 128.7, 128.4, 128.2, 126.2, 122.6, 115.3, 25.3, 21.5.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>18</sub>H<sub>17</sub>NO<sub>2</sub>Na: 302.1157, found 302.1158.

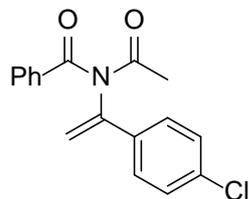


***N*-Acetyl-*N*-(1-(4-methoxyphenyl)vinyl)benzamide (6c).** Yellow oil (318.7 mg, 54% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.66 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.49 – 7.45 (m, 1H), 7.42 – 7.33 (m, 4H), 6.90 – 6.84 (m, 2H), 5.68 (d, *J* = 1.2 Hz, 1H), 5.11 (d, *J* = 1.1 Hz, 1H), 3.82 (s, 3H), 2.41 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 173.8, 173.0, 160.2, 144.6, 134.9, 132.1, 128.6, 128.4, 128.3, 126.9, 114.2, 113.6, 55.3, 25.3.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>18</sub>H<sub>17</sub>NO<sub>3</sub>Na: 318.1106, found 318.1110.

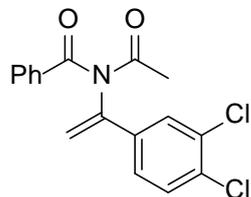


***N*-Benzoyl-*N*-(1-(4-chlorophenyl)ethenyl)acetamide (6d).** White solid (490.5 mg, 82% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.66 – 7.61 (m, 2H), 7.52 – 7.45 (m, 1H), 7.40 – 7.30 (m, 6H), 5.77 (d, *J* = 1.4 Hz, 1H), 5.22 (d, *J* = 1.4 Hz, 1H), 2.42 (s, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 173.6, 172.9, 144.0, 134.9, 134.6(2), 134.6(1), 132.3, 129.1, 128.4, 128.3, 126.8, 116.0, 25.4.

HRMS (ESI) [M+Na]<sup>+</sup>: calculated for C<sub>17</sub>H<sub>14</sub>NO<sub>2</sub>NaCl: 322.0611, found 322.0614.

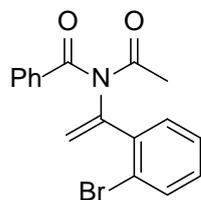


*N*-Benzoyl-*N*-(1-(3,4-dichlorophenyl)ethenyl)acetamide (**6e**). White solid (452.9 mg, 68% yield).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.67 – 7.58 (m, 2H), 7.52 – 7.46 (m, 1H), 7.42 – 7.32 (m, 3H), 7.22 (d, *J* = 8.4 Hz, 1H), 7.19 – 7.14 (m, 1H), 5.72 (d, *J* = 1.1 Hz, 1H), 5.56 (d, *J* = 1.2 Hz, 1H), 2.43 (s, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 173.3, 172.8, 140.7, 134.9(2), 134.8(9), 134.1, 132.7, 132.5, 131.9, 130.2, 128.8, 128.4, 127.3, 120.6, 25.4.

HRMS (ESI) [M+Na]<sup>+</sup>: calculated for C<sub>17</sub>H<sub>13</sub>NO<sub>2</sub>NaCl<sub>2</sub>: 356.0221, found 356.0223.

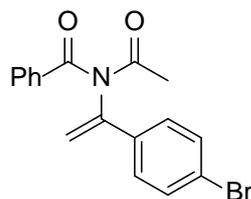


*N*-Acetyl-*N*-(1-(2-bromophenyl)vinyl)benzamide (**6f**). White solid (308.7 mg, 45% yield).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.65 – 7.61 (m, 2H), 7.54 – 7.45 (m, 2H), 7.39 – 7.34 (m, 2H), 7.29 – 7.26 (m, 1H), 7.24 – 7.19 (m, 1H), 7.13 – 7.08 (m, 1H), 5.75 (d, *J* = 1.1 Hz, 1H), 5.57 (d, *J* = 1.0 Hz, 1H), 2.44 (s, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 173.5, 173.1, 142.4, 137.3, 135.1, 133.8, 132.4, 131.3, 129.8, 128.8, 128.4, 127.5, 121.6, 120.1, 25.4.

HRMS (ESI) [M+Na]<sup>+</sup>: calculated for C<sub>17</sub>H<sub>14</sub>NO<sub>2</sub>NaBr: 366.0106, found 366.0109.

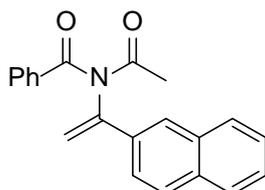


***N*-Benzoyl-*N*-(1-(4-bromophenyl)ethenyl)acetamide (6g).** White solid (493.9 mg, 72% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.67 – 7.62 (m, 2H), 7.51 – 7.45 (m, 3H), 7.39 – 7.29 (m, 4H), 5.77 (d, *J* = 1.0 Hz, 1H), 5.22 (d, *J* = 1.0 Hz, 1H), 2.42 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 173.6, 172.8, 144.1, 135.1, 134.6, 132.3, 132.0, 128.4(3), 128.3(5), 127.1, 123.1, 116.1, 25.4.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>17</sub>H<sub>14</sub>NO<sub>2</sub>NaBr: 366.0106, found 366.0109.

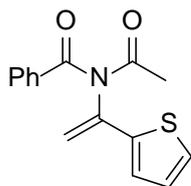


***N*-Benzoyl-*N*-(1-(naphthalen-2-yl)ethenyl)acetamide (6h).** White solid (447.5 mg, 71% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.90(d, *J* = 1.9 Hz, 1H), 7.86 – 7.81 (m, 3H), 7.75 – 7.69 (m, 2H), 7.59 (dd, *J* = 8.7, 2.0 Hz, 1H), 7.54 – 7.45 (m, 3H), 7.35 (t, *J* = 7.8 Hz, 2H), 5.95 (d, *J* = 1.4 Hz, 1H), 5.32 (d, *J* = 1.3 Hz, 1H), 2.47 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 173.8, 173.0, 145.0, 134.8, 133.4(2), 133.3(7), 133.2, 132.2, 128.8, 128.5(2), 128.4(6), 128.3, 127.6, 126.8, 126.6, 124.9, 123.1, 116.1, 25.4.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>21</sub>H<sub>17</sub>NO<sub>2</sub>Na: 338.1157, found 338.1154.

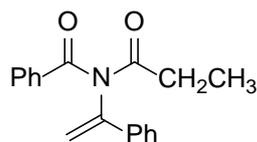


***N*-Acetyl-*N*-(1-(thiophen-2-yl)vinyl)benzamide (6i).** Yellow solid (385.0 mg, 71% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.72 – 7.67 (m, 2H), 7.49 (t, *J* = 7.5 Hz, 1H), 7.39 – 7.36 (m, 2H), 7.25 (d, *J* = 5.1 Hz, 1H), 7.14– 7.13 (m, 1H), 7.00– 6.99 (m, 1H), 5.73 (d, *J* = 1.4 Hz, 1H), 5.11 (d, *J* = 1.4 Hz, 1H), 2.45 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 173.4, 172.5, 140.8, 139.3, 134.7, 132.2, 128.4, 128.3, 127.8, 126.2, 125.5, 114.8, 25.1.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>15</sub>H<sub>13</sub>NO<sub>2</sub>NaS: 294.0565, found 294.0567.

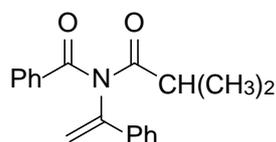


***N*-(1-Phenylvinyl)-*N*-propionylbenzamide (7a).** Yellow solid (267.0 mg, 48% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.67 (d, *J* = 7.5 Hz, 2H), 7.49–7.46 (m, 3H), 7.38 – 7.32 (m, 5H), 5.81 (d, *J* = 1.1 Hz, 1H), 5.21 (d, *J* = 1.1 Hz, 1H), 2.71 (q, *J* = 7.3 Hz, 2H), 1.16 (t, *J* = 7.4 Hz, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 177.7, 173.1, 144.9, 136.2, 135.1, 132.0, 129.0, 128.9, 128.3(5), 128.3(0), 125.5, 115.4, 30.8, 9.5.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>18</sub>H<sub>17</sub>NO<sub>2</sub>Na: 302.1157, found 302.1160.

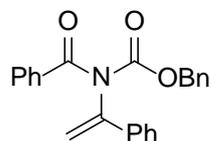


***N*-Isobutyryl-*N*-(1-phenylvinyl)benzamide (7b).** Yellow solid (211.1 mg, 36% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.73 – 7.71 (m, 2H), 7.55 – 7.46 (m, 3H), 7.41 – 7.31 (m, 5H), 5.79 (d, *J* = 1.2 Hz, 1H), 5.21 (d, *J* = 1.2 Hz, 1H), 3.11 (Sept, *J* = 6.8 Hz, 1H), 1.2 (d, *J* = 6.8 Hz, 6H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 181.5, 173.4, 145.1, 136.3, 135.1, 132.1, 128.9, 128.8, 128.3, 128.2, 125.5, 114.9, 35.3, 19.7.

**HRMS (ESI) [M+H]<sup>+</sup>:** calculated for C<sub>19</sub>H<sub>20</sub>NO<sub>2</sub>: 294.1494, found 294.1490.

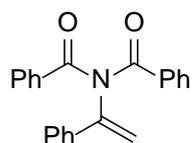


**Benzyl benzoyl(1-phenylethenyl)carbamate (7c).** Yellow oil (542.8 mg, 76% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.77 – 7.68 (m, 2H), 7.56 – 7.47 (m, 3H), 7.41 (t, *J* = 7.8 Hz, 2H), 7.38 – 7.31 (m, 3H), 7.29 – 7.24 (m, 1H), 7.21 (t, *J* = 7.3 Hz, 2H), 6.93 (d, *J* = 6.8 Hz, 2H), 5.77 (d, *J* = 1.2 Hz, 1H), 5.28 (d, *J* = 1.2 Hz, 1H), 5.03 (s, 2H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 172.1, 154.3, 144.5, 136.5, 135.4, 134.4, 132.1, 128.8, 128.7, 128.3(4), 128.3(3), 128.2(8), 128.2(6), 128.1, 125.6, 114.3, 68.8.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>23</sub>H<sub>19</sub>NO<sub>3</sub>Na: 380.1263, found 380.1266.

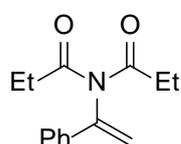


**N-Benzoyl-N-(1-phenylvinyl)benzamide (9a)**. White solid (425.3 mg, 65% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.85–7.82 (m, 4H), 7.62–7.60 (m, 2H), 7.50–7.47 (m, 2H), 7.39–7.31 (m, 7H), 5.70 (d, *J* = 1.4 Hz, 1H), 5.11 (d, *J* = 1.5 Hz, 1H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 173.3, 145.8, 136.0, 134.4, 132.6, 129.0, 128.9(3), 128.8(9), 128.6, 125.6, 114.5.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>22</sub>H<sub>17</sub>NO<sub>2</sub>Na: 350.1157, found 350.1160.

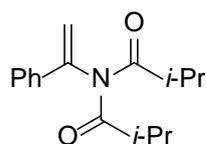


**N-(1-Phenylvinyl)-N-propionylpropionamide (9b)**. Yellow oil (184.9 mg, 40% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.47–7.21 (m, 5H), 6.01 (s, 1H), 5.28 (s, 1H), 2.73 (q, *J* = 6.9 Hz, 4H), 1.10 (t, *J* = 7.3 Hz, 6H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 176.5, 144.1, 135.5, 129.0, 128.9, 124.9, 115.6, 31.4, 9.1.

The characterization data were consistent with the literature.<sup>[2b]</sup>



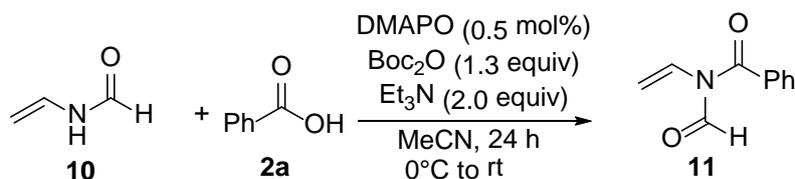
**N-Isobutyryl-N-(1-phenylvinyl)isobutyramide (9c)**. Yellow oil (181.3 mg, 35% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.44–7.40 (m, 2H), 7.39–7.30 (m, 3H), 5.92 (d, *J* = 1.0 Hz, 1H), 5.22 (d, *J* = 0.9 Hz, 1H), 3.23 (Sept, *J* = 6.8 Hz, 2H), 1.16 (d, *J* = 6.7 Hz, 12H).

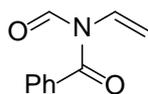
**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 181.1, 144.5, 136.0, 129.1, 128.9, 125.1, 114.5, 35.4, 19.8.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>16</sub>H<sub>21</sub>NO<sub>2</sub>Na: 282.1470, found 282.1472.

### 3. Procedure of gram-scale synthesis



To a 250 mL dried Schlenk tube equipped with a magnetic stirrer was added DMAPO (6.9 mg, 0.05 mmol, 0.005 equiv), benzoic acid **2a** (1.2 g, 10.0 mmol, 1.0 equiv), and *N*-vinylformamide **10** (0.71 g, 10.0 mmol, 1.0 equiv). The reaction tube was allowed to be vacuumed and purged with nitrogen for three times. Then, CH<sub>3</sub>CN (30.0 mL) was added by syringe under nitrogen atmosphere. After the reaction mixture was cooled to 0 °C, Et<sub>3</sub>N (2.78 mL, 20.0 mmol, 2.0 equiv) and Boc<sub>2</sub>O (2.98 mL, 13 mmol, 1.3equiv) were successively added dropwise to the reaction mixture and the final solution was stirred for 24 h at room temperature. After the reaction was complete, the reaction solution was quenched by adding water (15 mL). The aqueous layer was extracted with ethyl acetate (4 × 15 mL), and the combined organic layers were washed with brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated under reduced pressure. The crude product was purified by column chromatography over silica gel with hexane/ethyl acetate as the eluent.



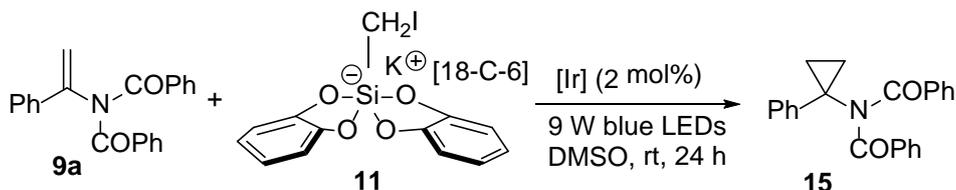
***N*-Formyl-*N*-vinylbenzamide (11).** Yellow oil (927.8 mg, 53% yield).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 9.01 (s, 1H), 7.65 – 7.61 (m, 2H), 7.58 – 7.53 (m, 1H), 7.47 – 7.44 (m, 2H), 6.76 (dd, *J* = 16.2, 9.4 Hz, 1H), 5.49 (d, *J* = 16.2 Hz, 1H), 5.09 (d, *J* = 9.3 Hz, 1H).

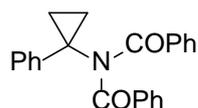
<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 171.4, 162.4, 132.8, 132.7, 129.4, 128.7, 127.6, 109.6.

HRMS (ESI) [M+Na]<sup>+</sup>: calculated for C<sub>10</sub>H<sub>9</sub>NO<sub>2</sub>Na: 198.0531, found 198.0520.

## 4. General procedure of photocatalyzed reaction



To a 10 mL dried Schlenk tube equipped with a magnetic stirrer was added Ir[dF(CF<sub>3</sub>)ppy]<sub>2</sub>(dtbbpy)PF<sub>6</sub> (4.5 mg, 0.004 mmol, 0.02 equiv), *N*-benzoyl-*N*-(1-phenylvinyl)benzamide **9a** (65.4 mg, 0.2 mmol, 1.0 equiv), and potassium [18-crown-6] bis(catecholato)iodomethylsilicate **11** (275.5 mg, 0.4 mmol, 2.0 equiv). The reaction tube was allowed to be vacuumed and purged with nitrogen for three times. Then, DMSO (6.0 mL, 0.033 M) was added by syringe under nitrogen atmosphere. The reaction mixture was irradiated with 9 W blue LEDs at room temperature for 24 h. After the reaction was complete, the reaction solution was quenched by adding water (5 mL). The aqueous layer was extracted with ethyl acetate (3 × 10 mL), and the combined organic layers were washed with brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated under reduced pressure. The crude product was purified by column chromatography over silica gel with hexane/ethyl acetate as the eluent.

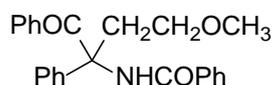


***N*-Benzoyl-*N*-(1-phenylcyclopropyl)benzamide (15)**. Yellow solid (40.9 mg, 60% yield).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.71 (d, *J* = 7.4 Hz, 2H), 7.36 – 7.31 (m, 2H), 7.31 – 7.23 (m, 5H), 7.23 – 7.17 (m, 2H), 7.13 – 7.07 (m, 4H), 1.57 (d, *J* = 2.8 Hz, 2H), 1.55 (d, *J* = 2.8 Hz, 2H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 174.2, 141.2, 137.7, 131.7, 128.3(9), 128.3(7), 128.2, 127.3, 42.2, 17.1.

HRMS (ESI) [M+Na]<sup>+</sup>: calculated for C<sub>23</sub>H<sub>19</sub>NO<sub>2</sub>Na: 364.1313, found 364.1317.



***N*-(4-Methoxy-1-oxo-1,2-diphenylbutan-2-yl)benzamide (16)**. Yellow oil (50.6 mg, 68% yield using biscatecholato silicate **12** as the radical precursor).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.70 (s, 1H), 7.96 – 7.81 (m, 2H), 7.60 – 7.56 (m, 2H), 7.55 – 7.51 (m, 3H), 7.49 – 7.45 (m, 2H), 7.44 – 7.39 (m, 3H), 7.36 – 7.31 (m, 1H), 7.30 – 7.24 (m, 2H), 3.66 – 3.59 (m, 1H), 3.55 – 3.49 (m, 1H), 3.43 – 3.36 (m, 1H), 3.09 (s, 3H), 2.98 – 2.90 (m, 1H).

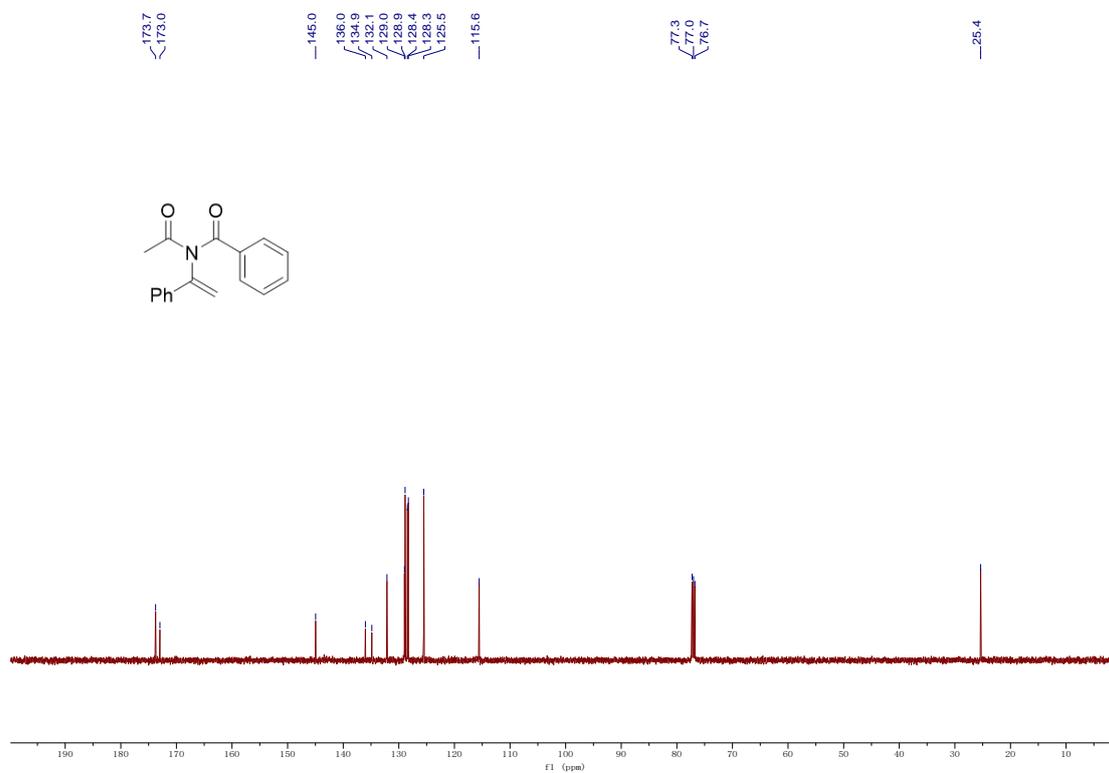
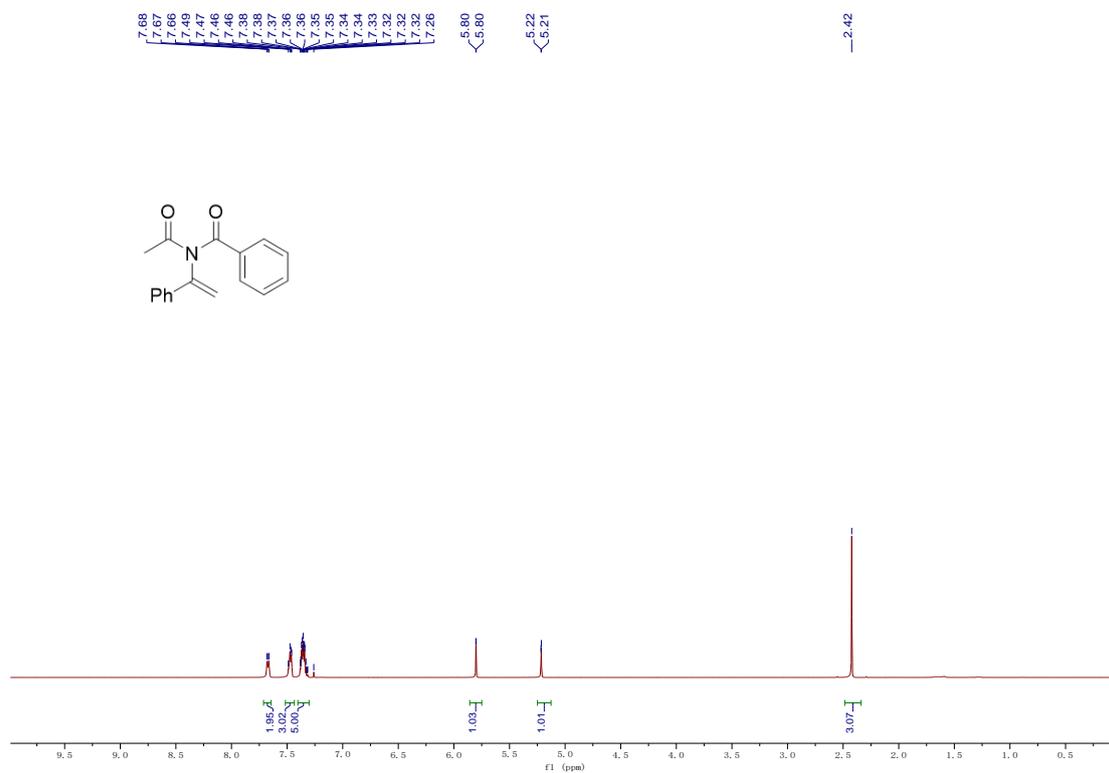
<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 198.4, 165.0, 139.4, 135.3, 134.7, 132.0, 131.5, 129.6, 128.8, 128.5, 128.1, 128.0, 127.0, 126.9, 68.3, 67.5, 58.4, 32.7.

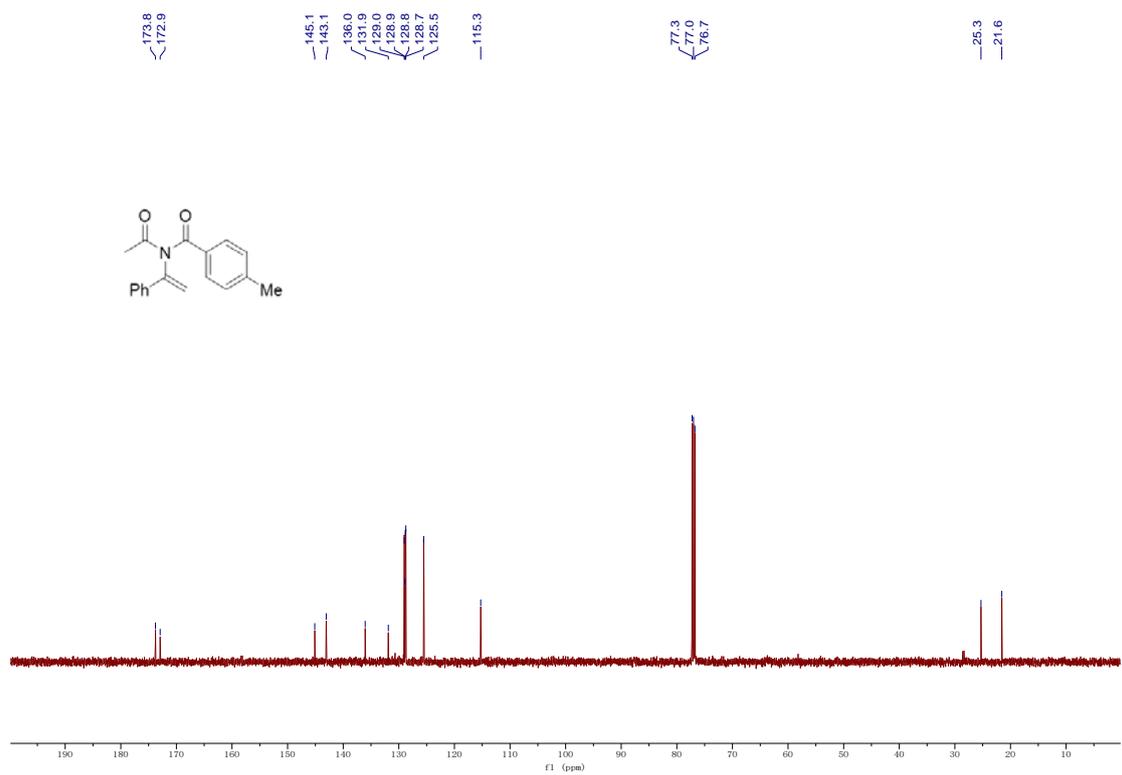
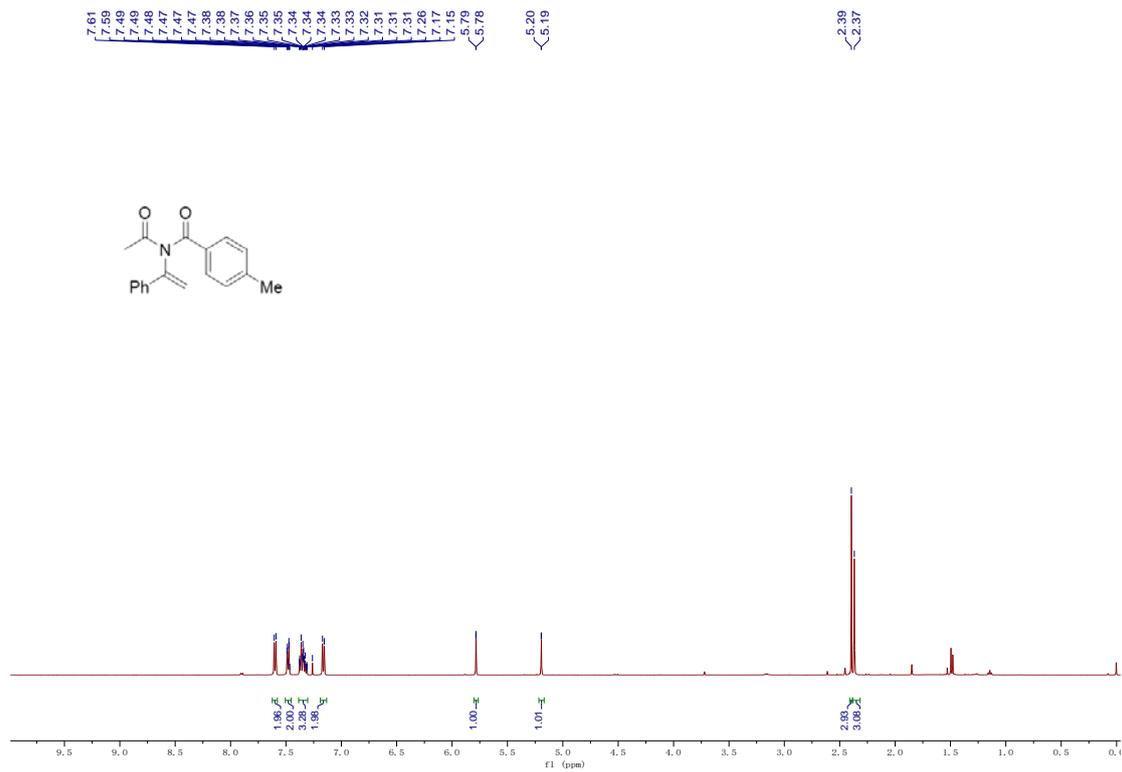
HRMS (ESI) [M+H]<sup>+</sup>: calculated for C<sub>24</sub>H<sub>24</sub>NO<sub>3</sub>: 374.1756, found 374.1760.

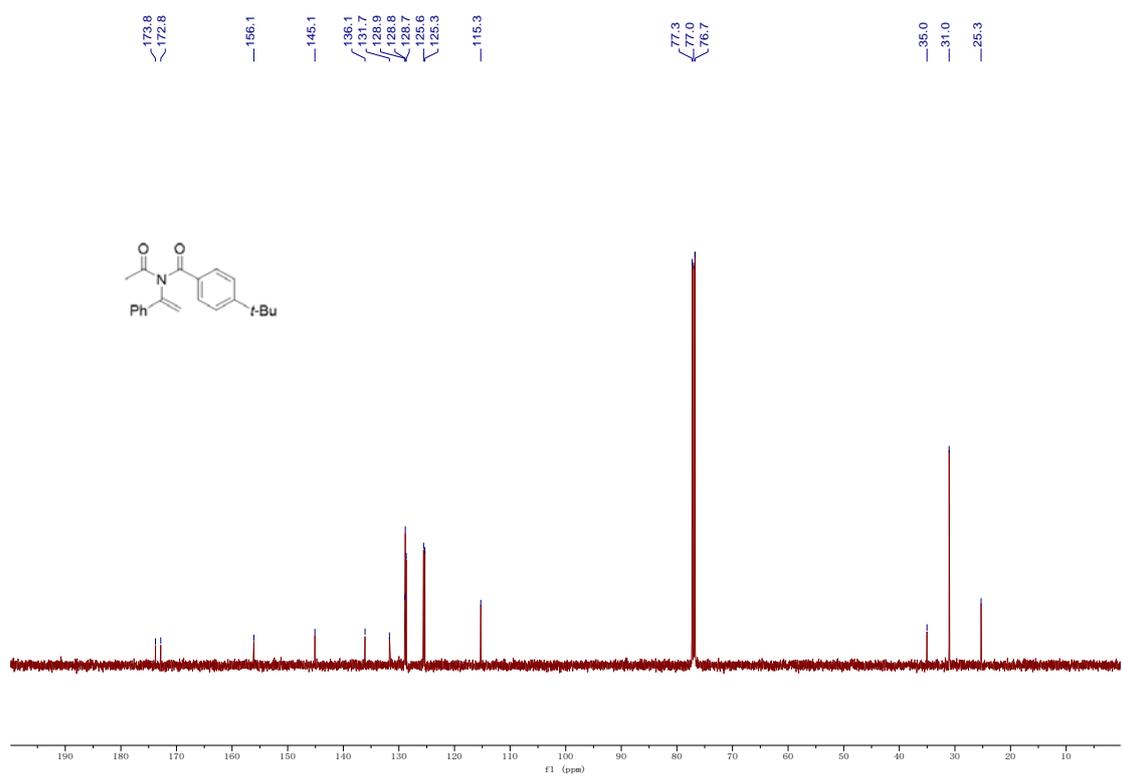
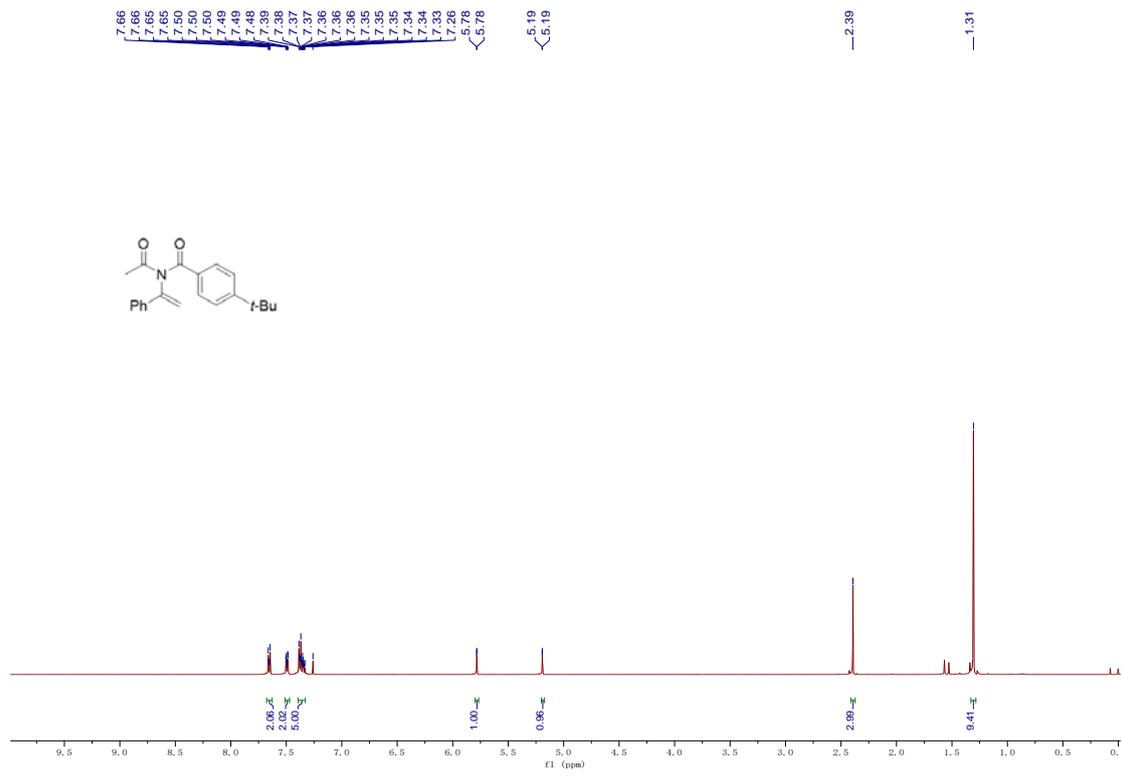
## 5. References

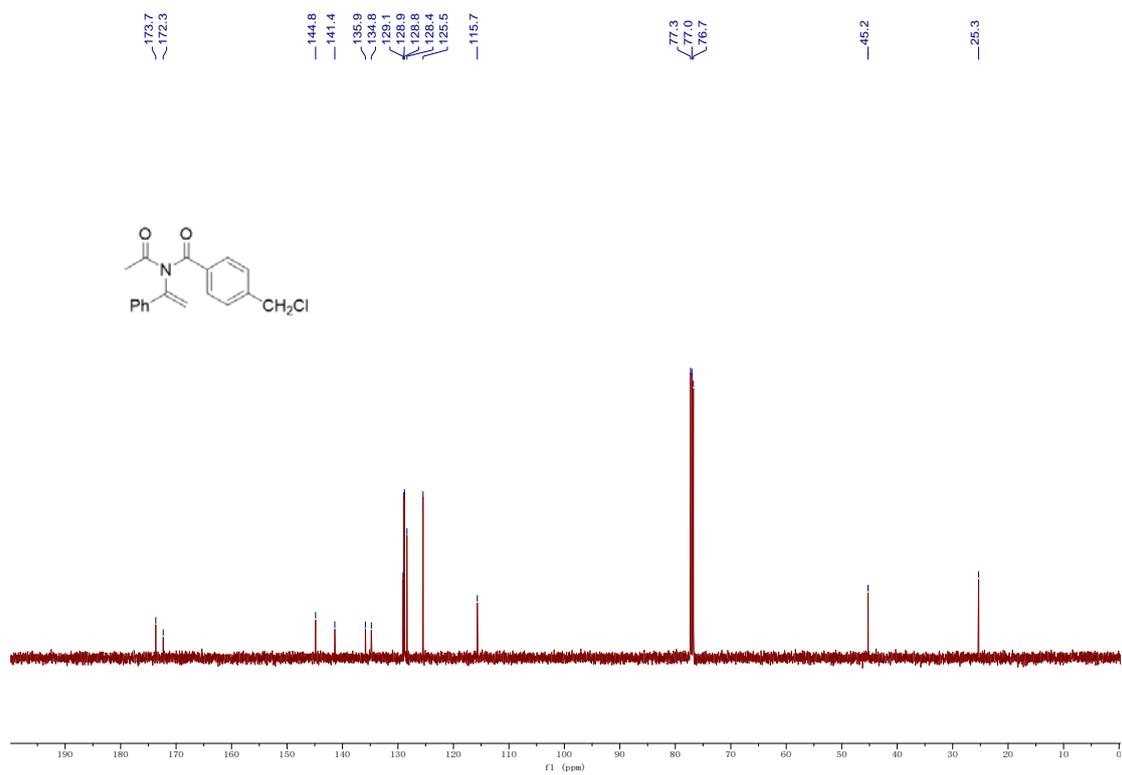
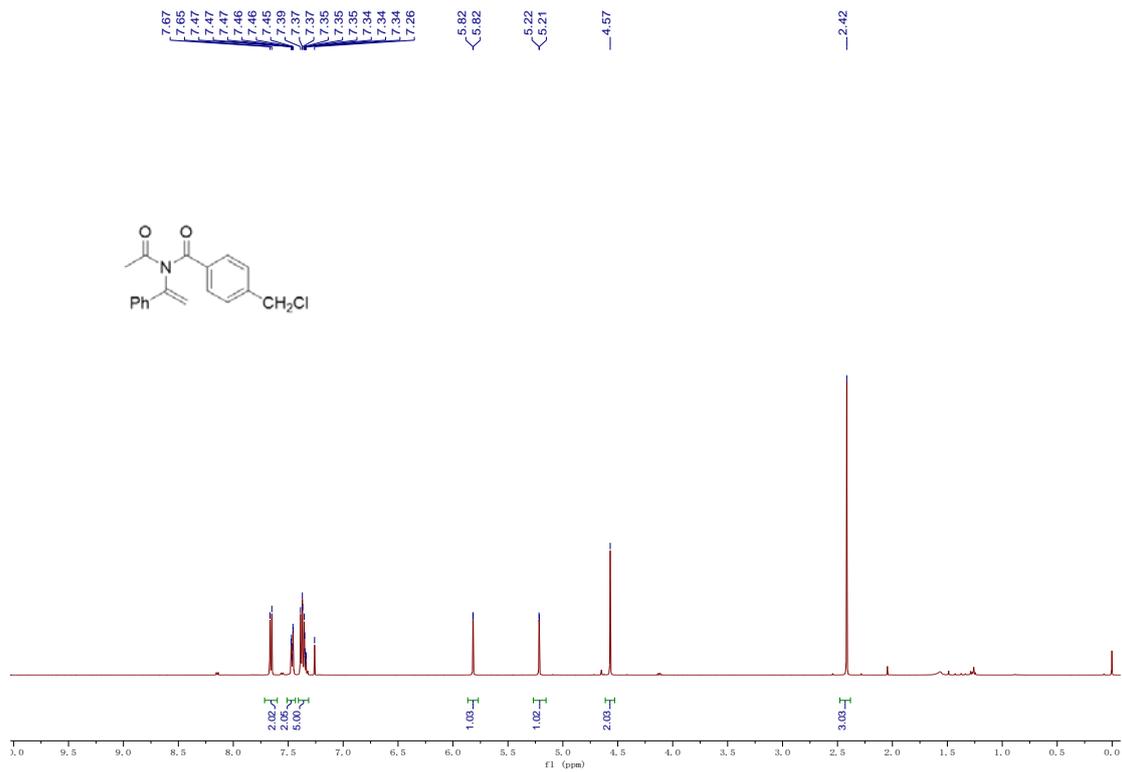
1. (a) W. Solomin, M. Liard, P. Jubault and T. Castanheiro, *Org. Chem. Front.*, 2025, **12**, 1467; (b) R.-H. Liu, Q.-C. Shan, Y. Gao, T.-P. Loh and X.-H. Hu, *Chin. Chem. Lett.*, 2021, **32**, 1411; (c) H.-Q. Cao, H.-N. Liu, Z.-Y. Liu, B.-K. Qiao, F.-G. Zhang and J.-A. Ma, *Chem. Eur. J.*, 2020, **26**, 5515; (d) W. Zhu, L. Zhao and M.-X. Wang, *J. Org. Chem.*, 2015, **80**, 12047.
2. (a) L. Huai, L. Zhang, Z. Wang and Y. Fang, *Org. Chem. Front.*, 2024, **11**, 2344; (b) Y. Zhao, Y. Jin, Y. Li, L. Qiu and Y. Fang, *Org. Chem. Front.*, 2025, **12**, 3834; (c) H. Huang, Y. Zhao, L. Zhang, Y. Li and Y. Fang, *Org. Biomol. Chem.*, 2025, **23**, 4079.
3. (a) Y. Jing, L. Zhang, L. Qiu and Y. Fang, *RSC Adv.*, 2025, **15**, 11582; (b) L. Huai, L. Zhang, Z. Wang, H. Wu and Y. Fang, *Org. Chem. Front.*, 2023, **10**, 1245.

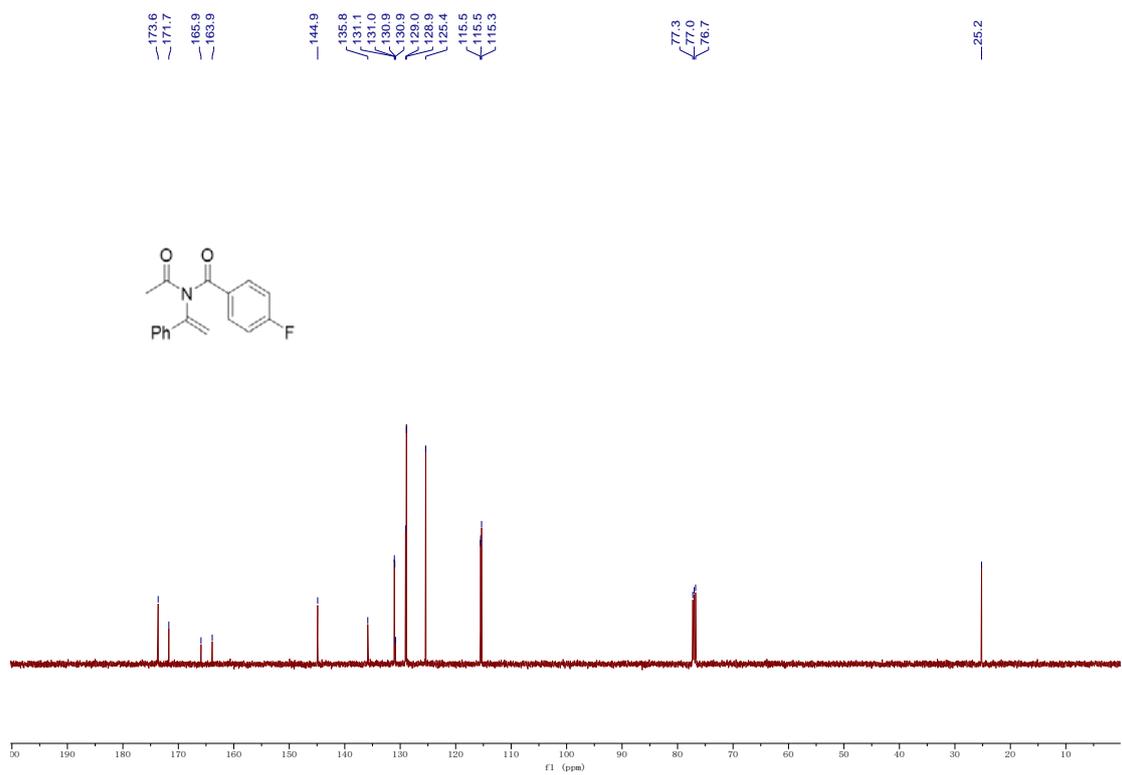
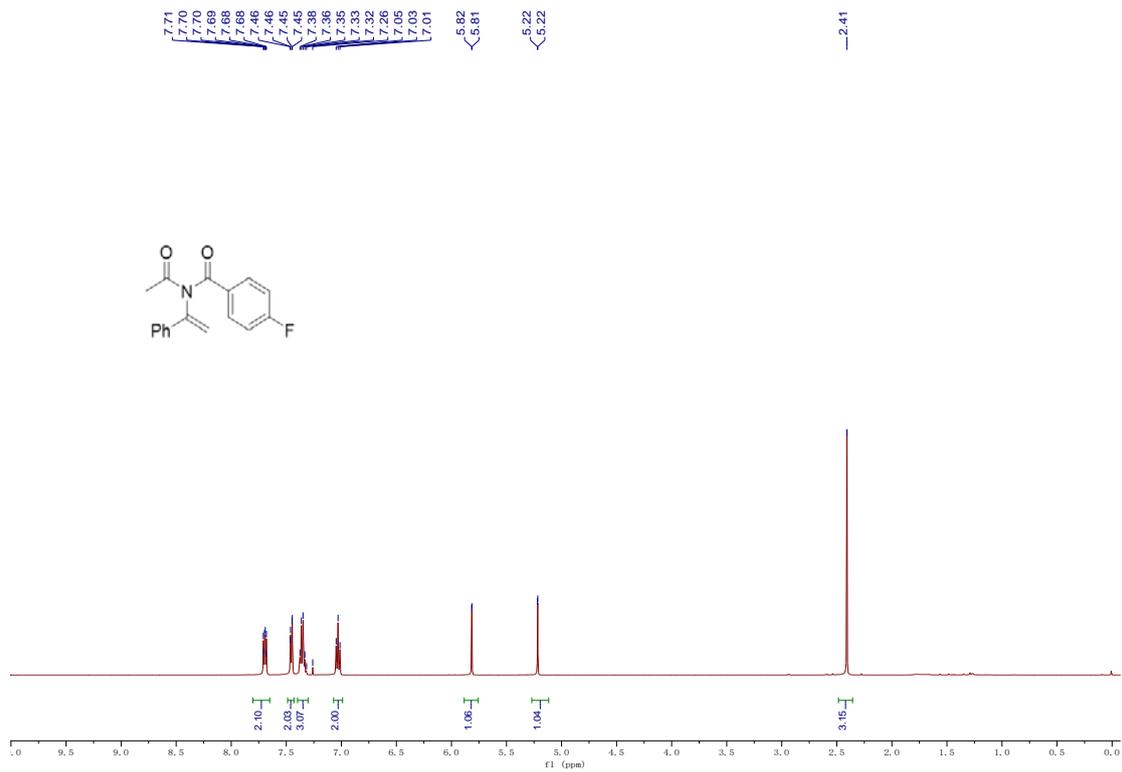
## 6. NMR Spectra of new compounds

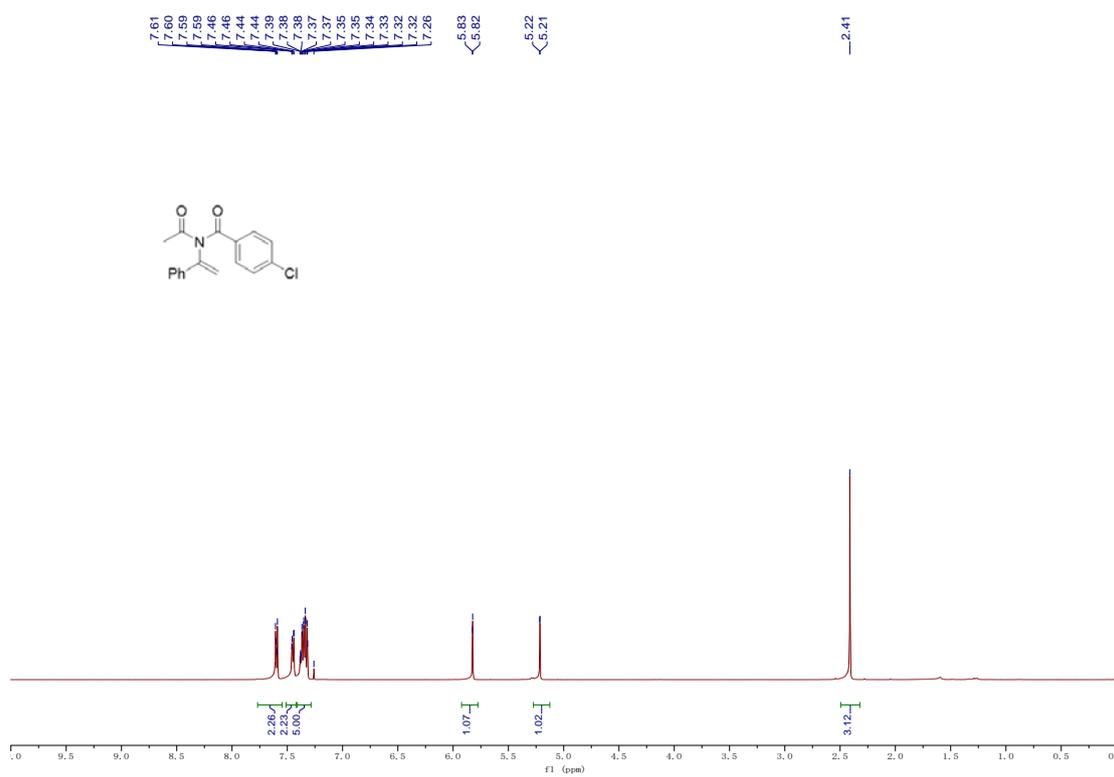
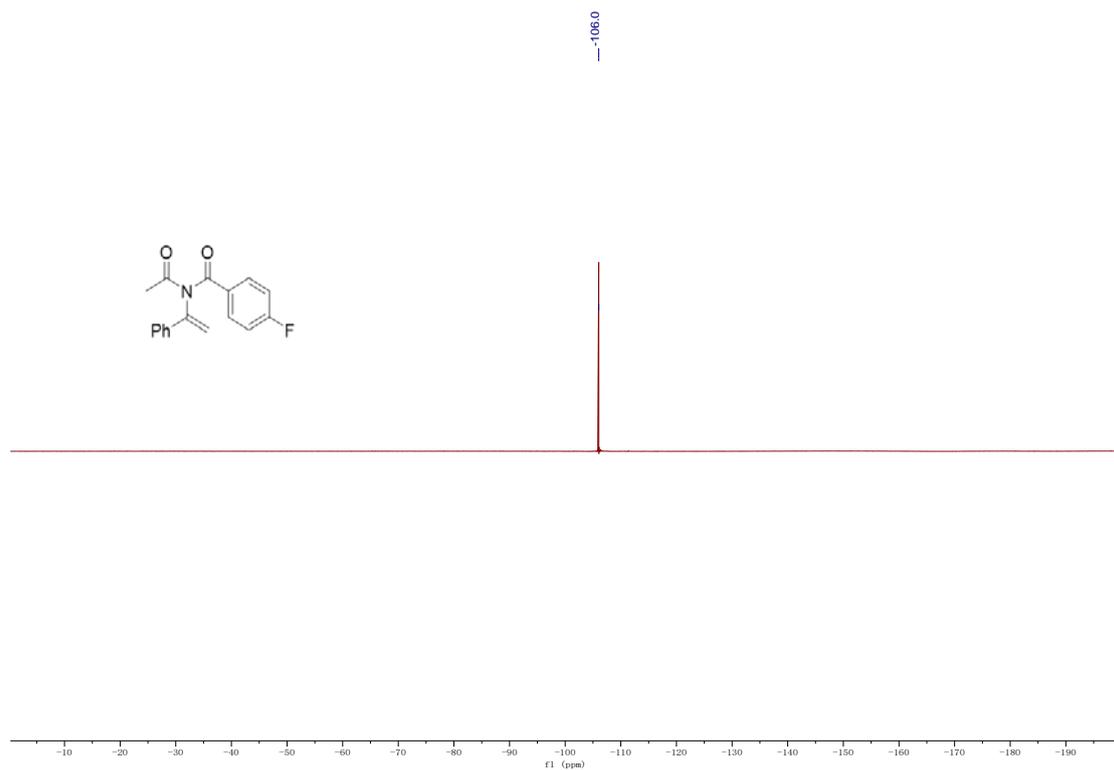


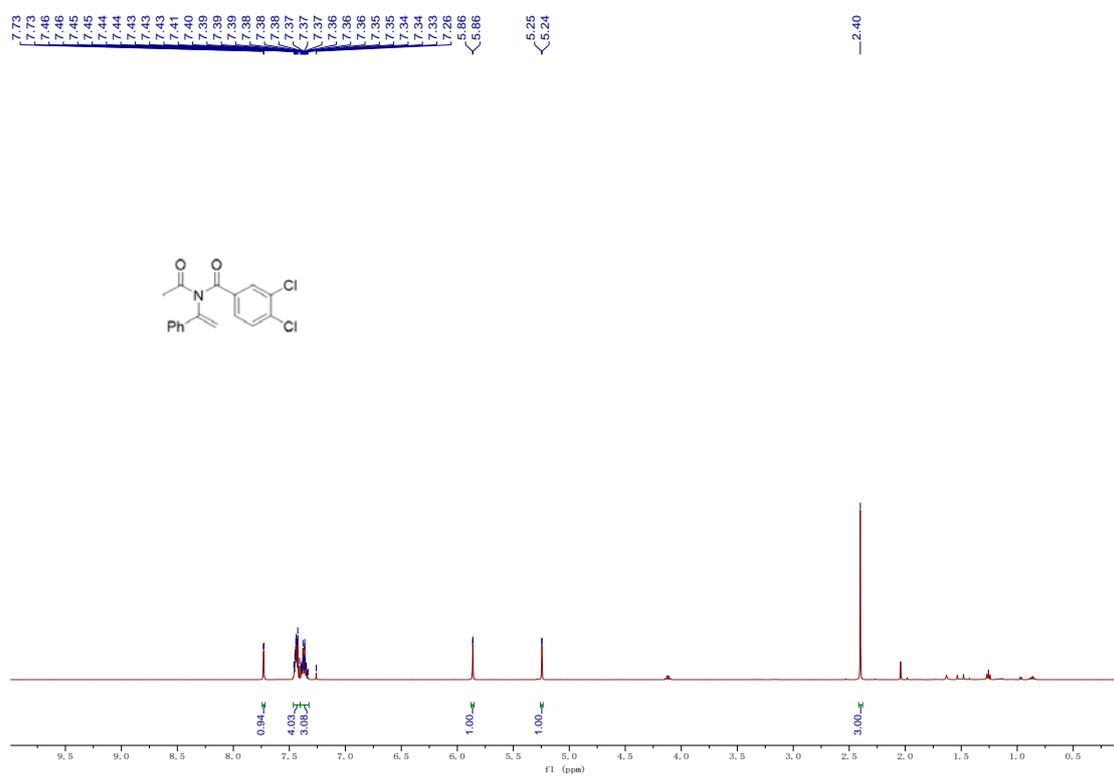
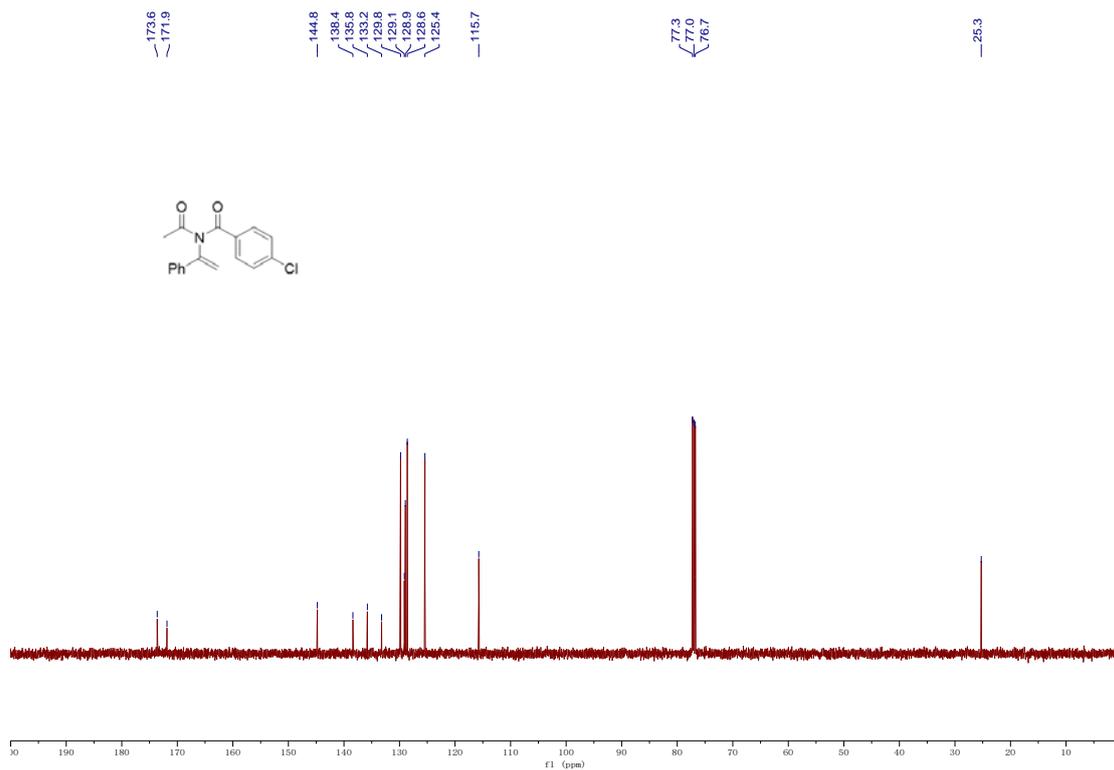


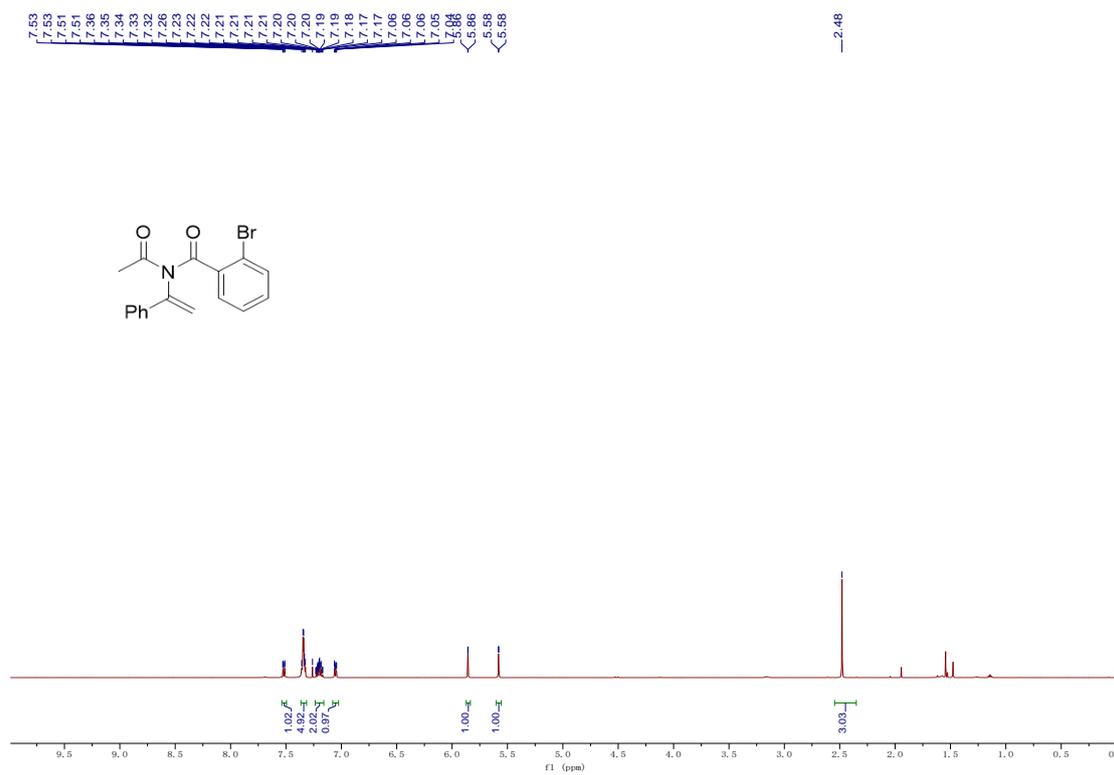
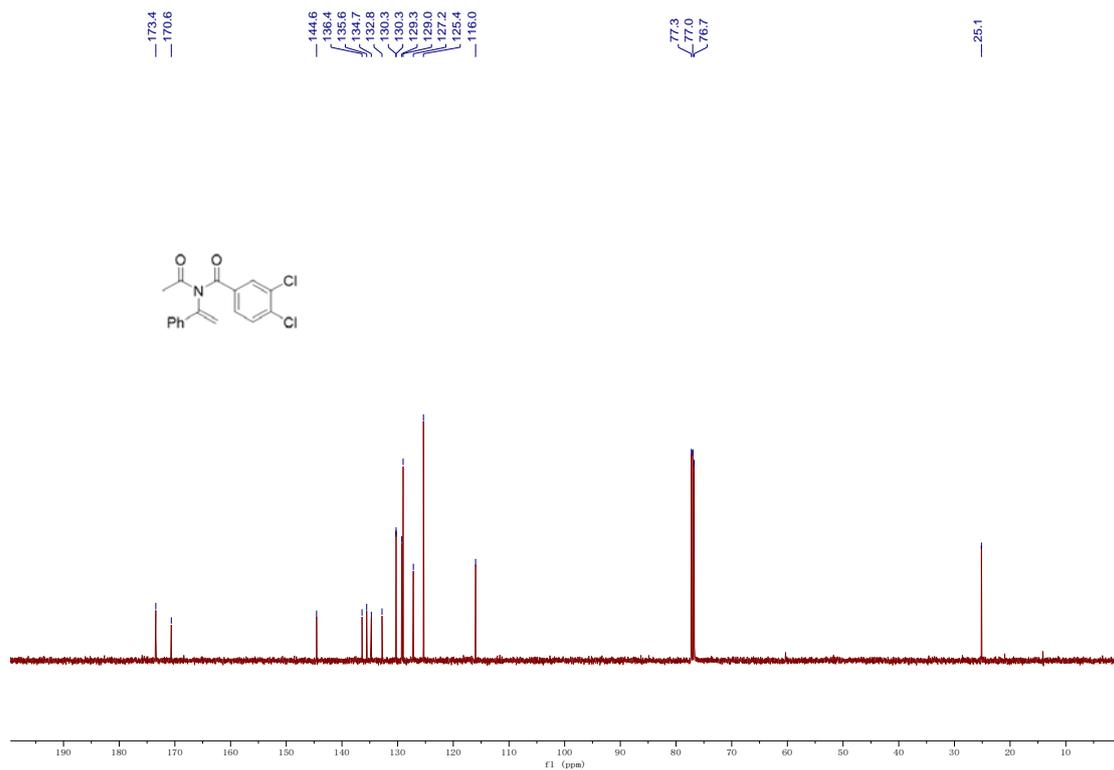


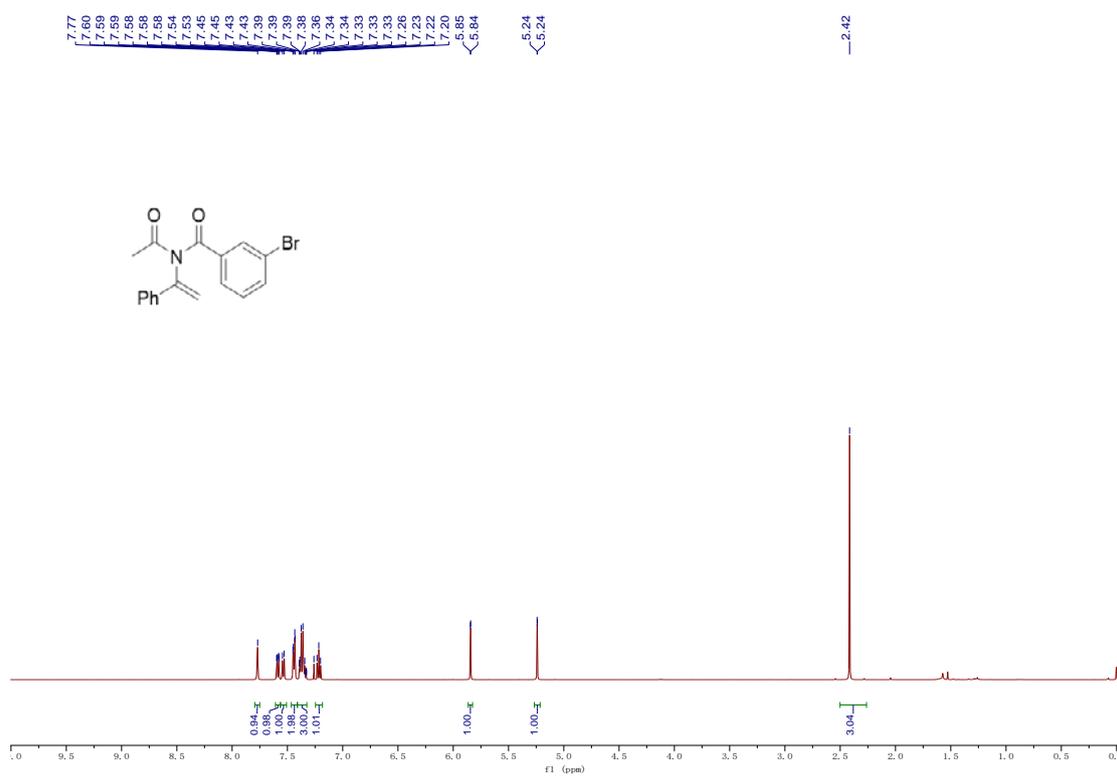
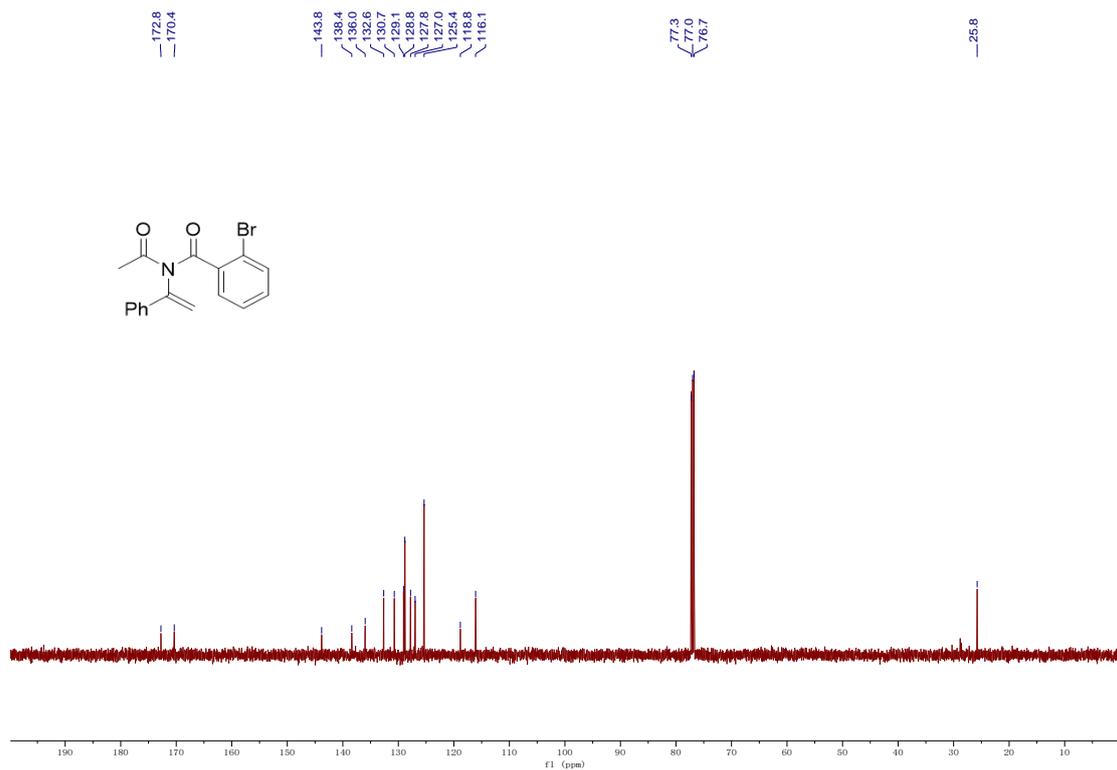


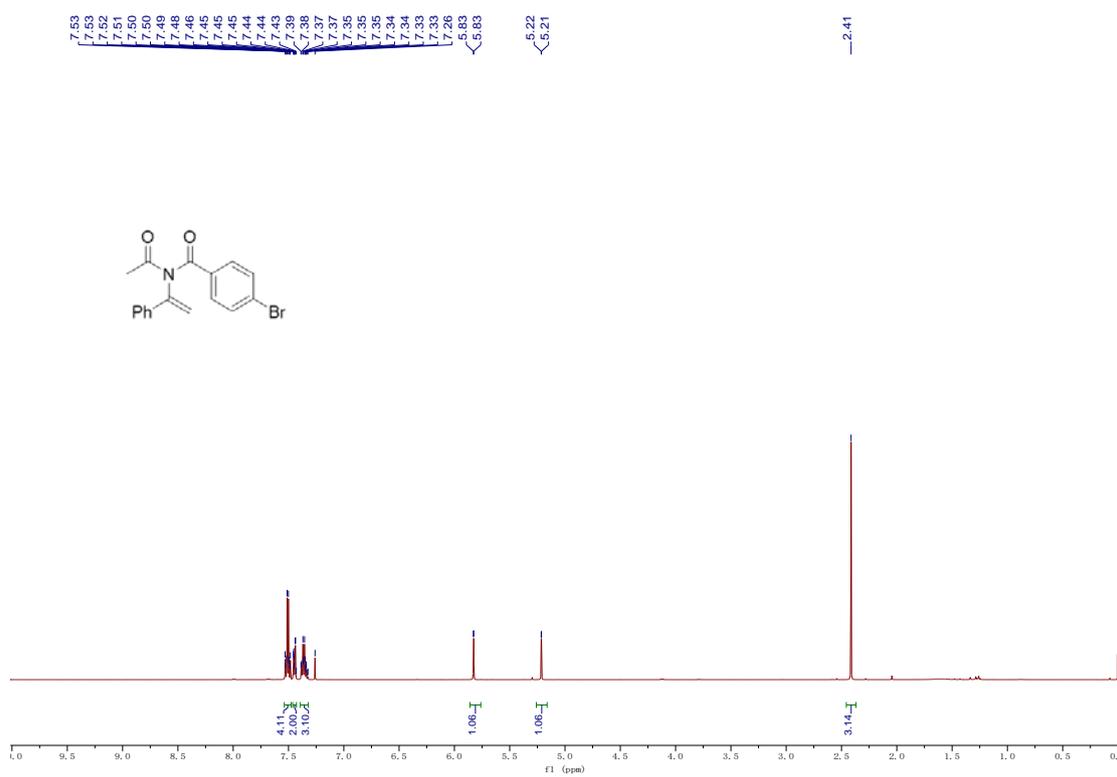
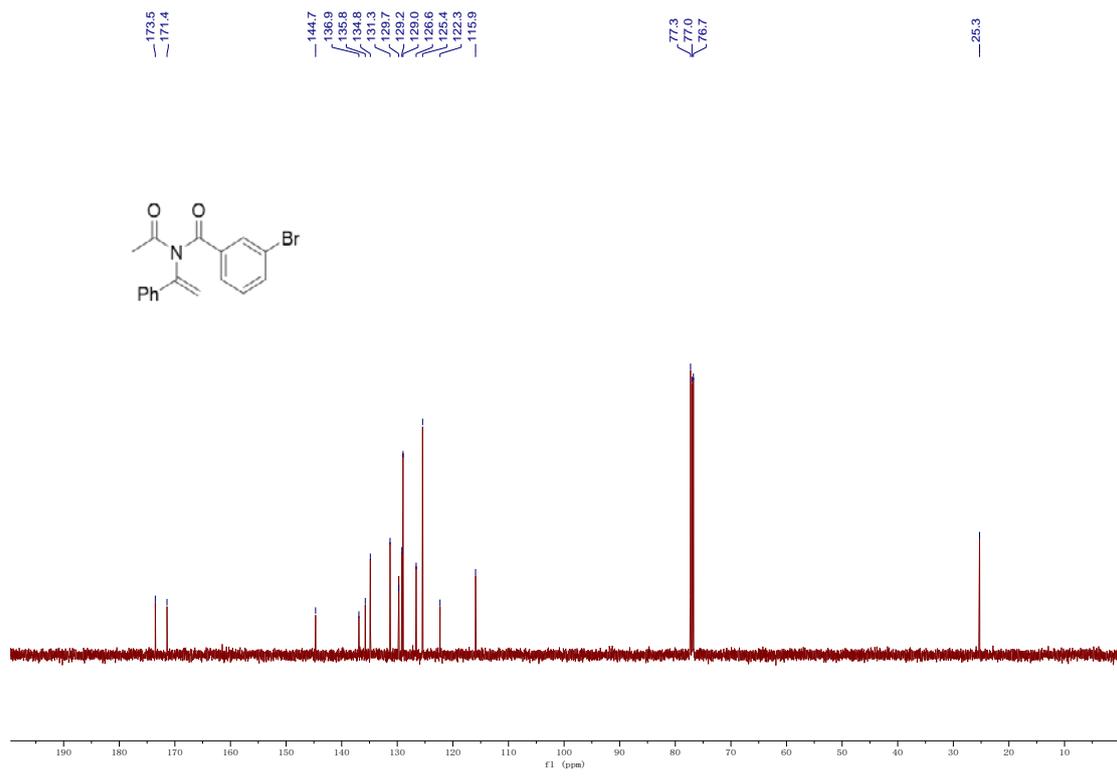




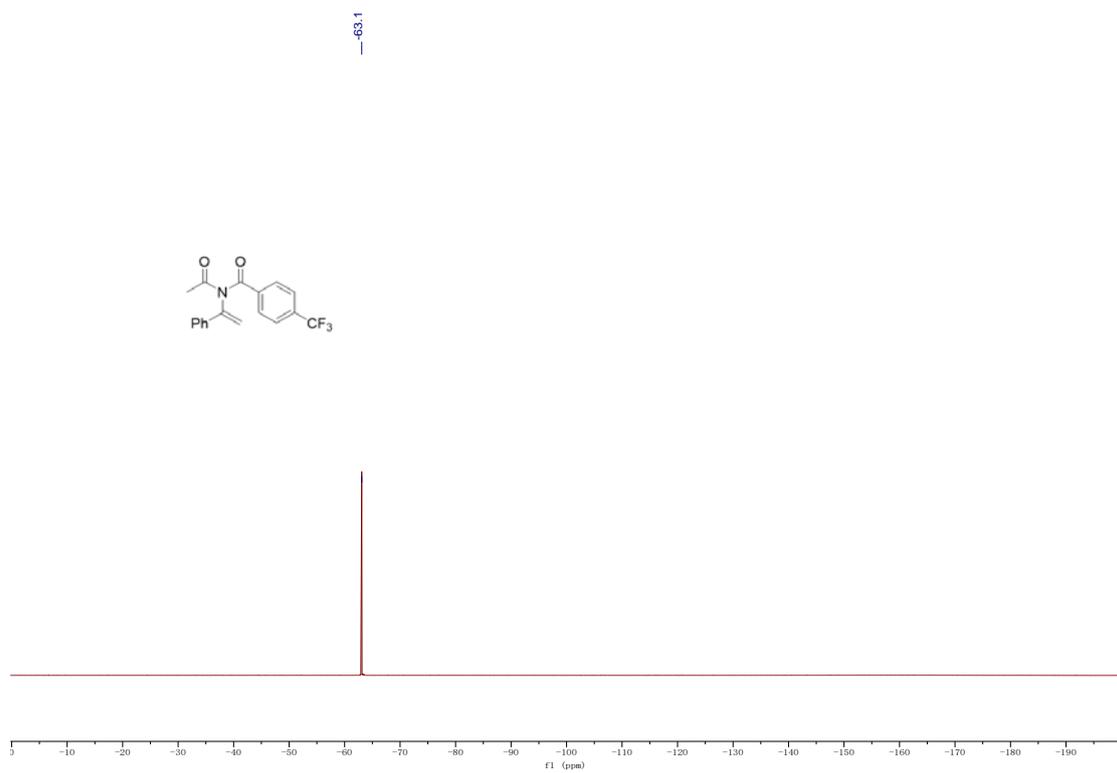
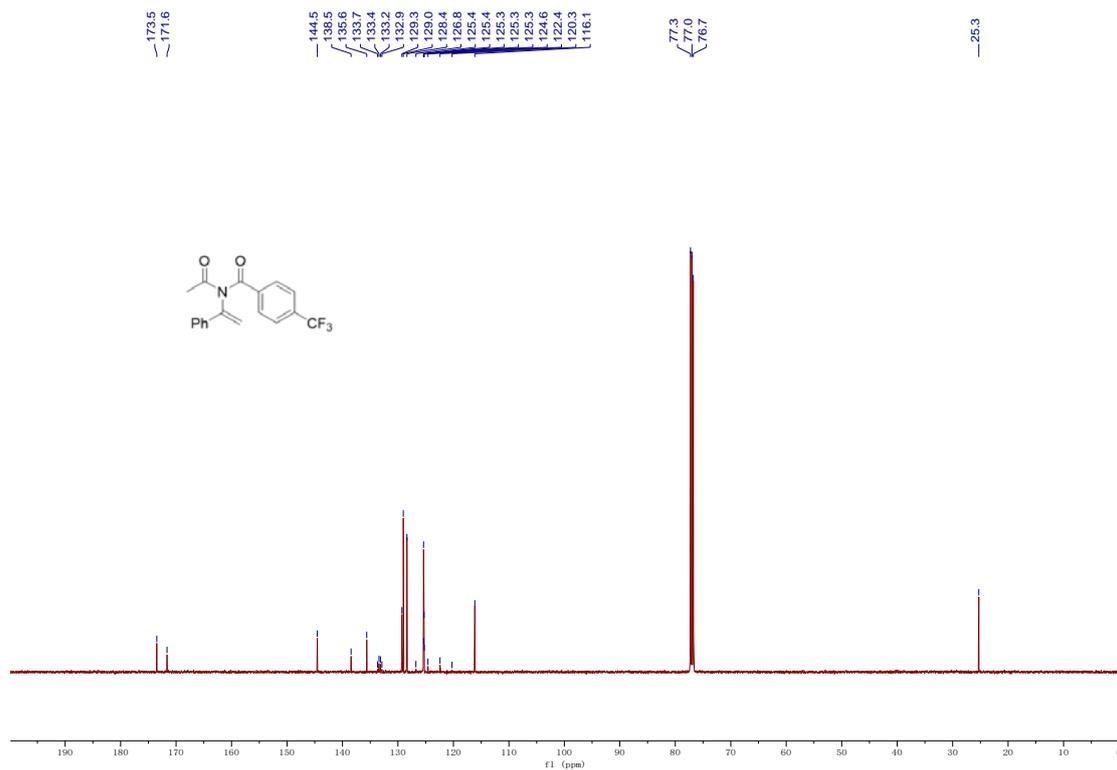


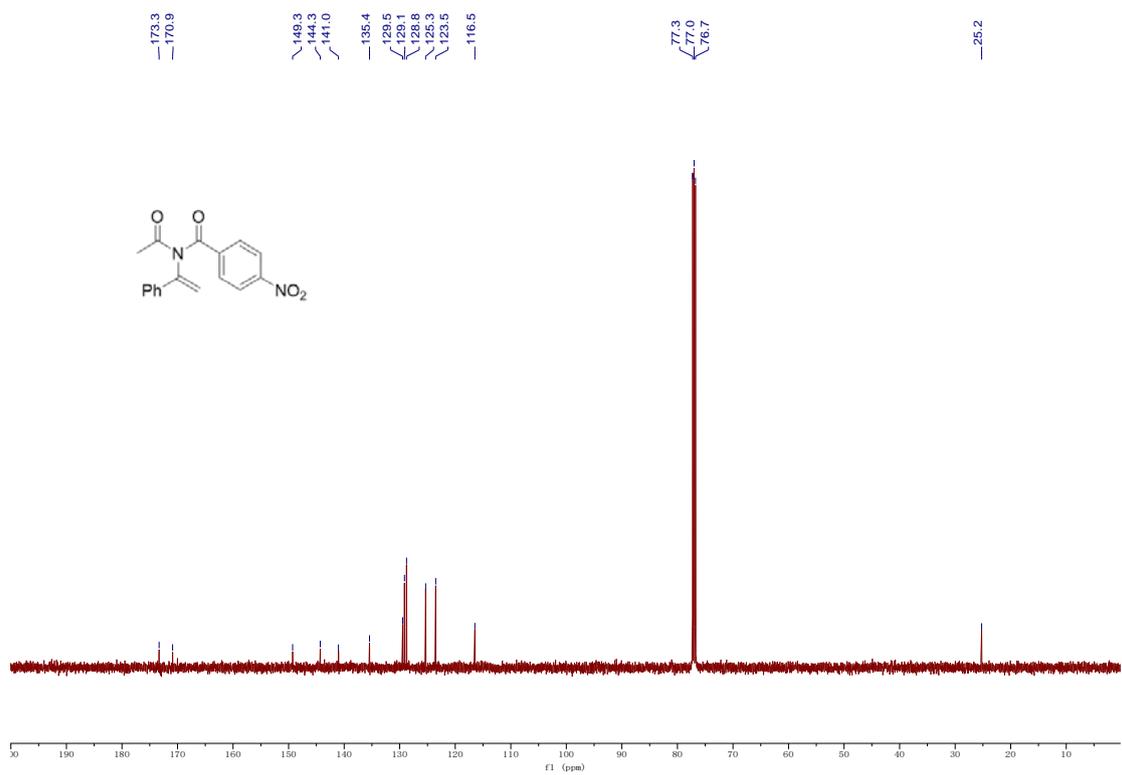
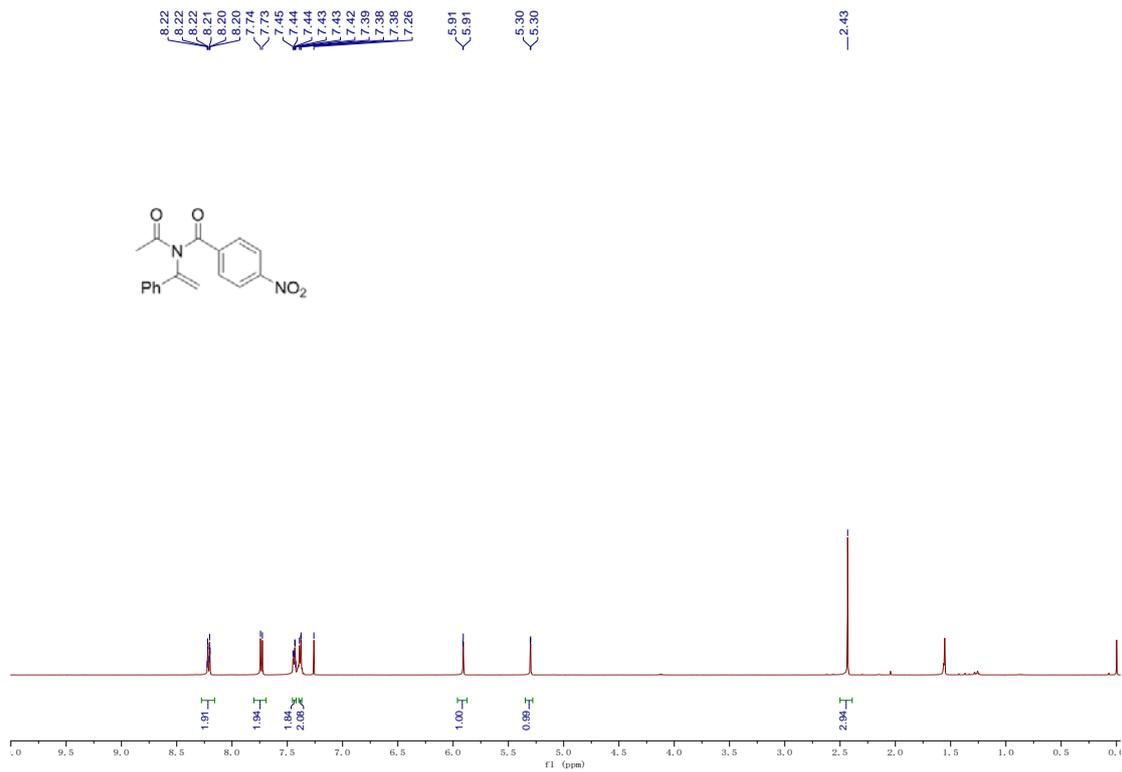




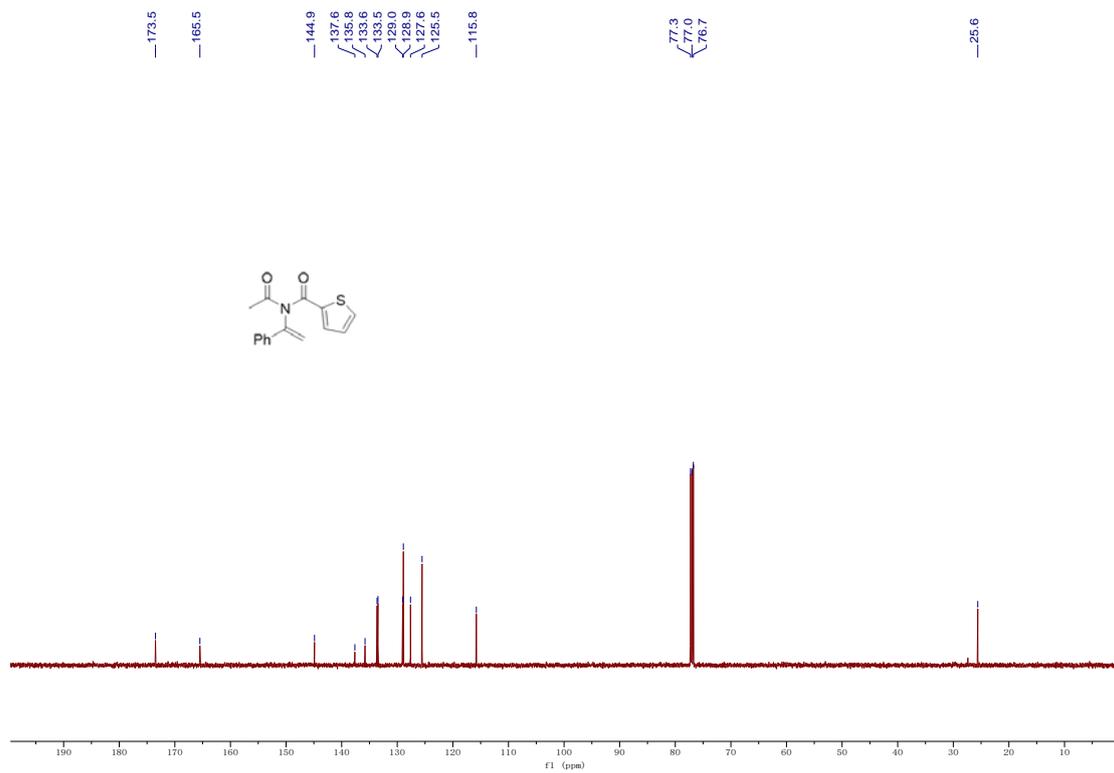
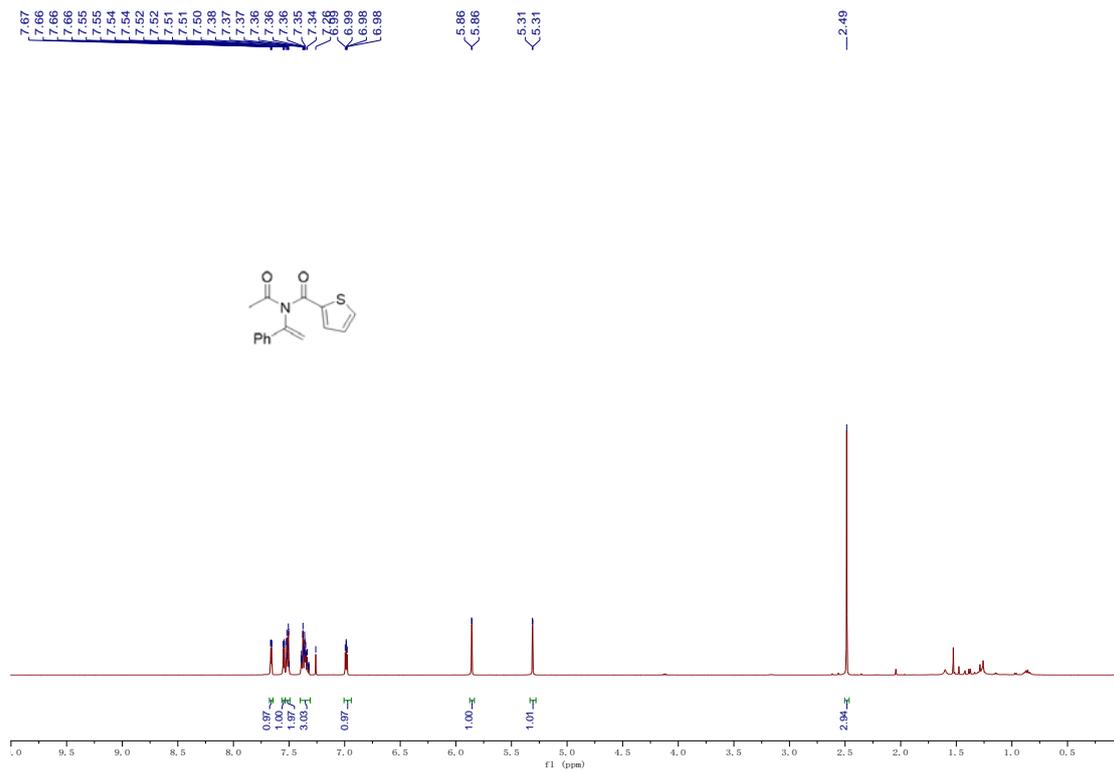


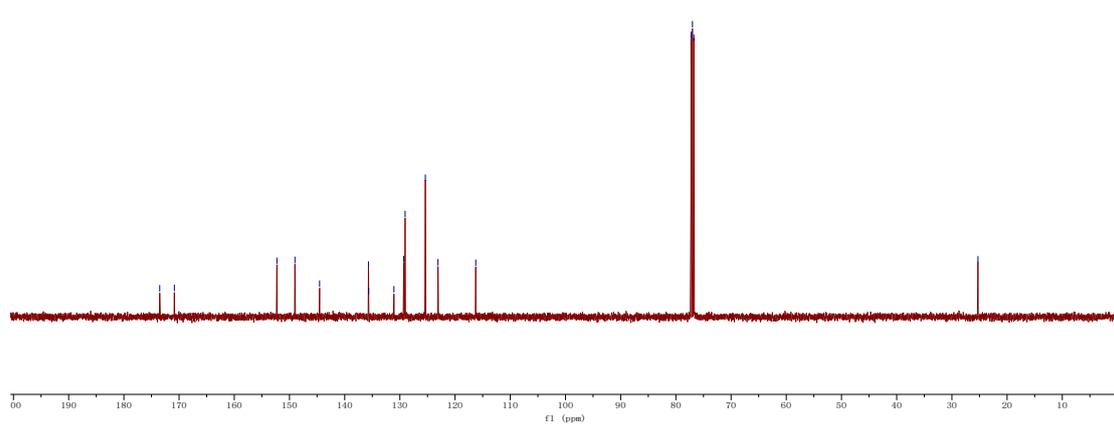
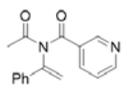
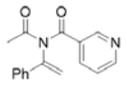
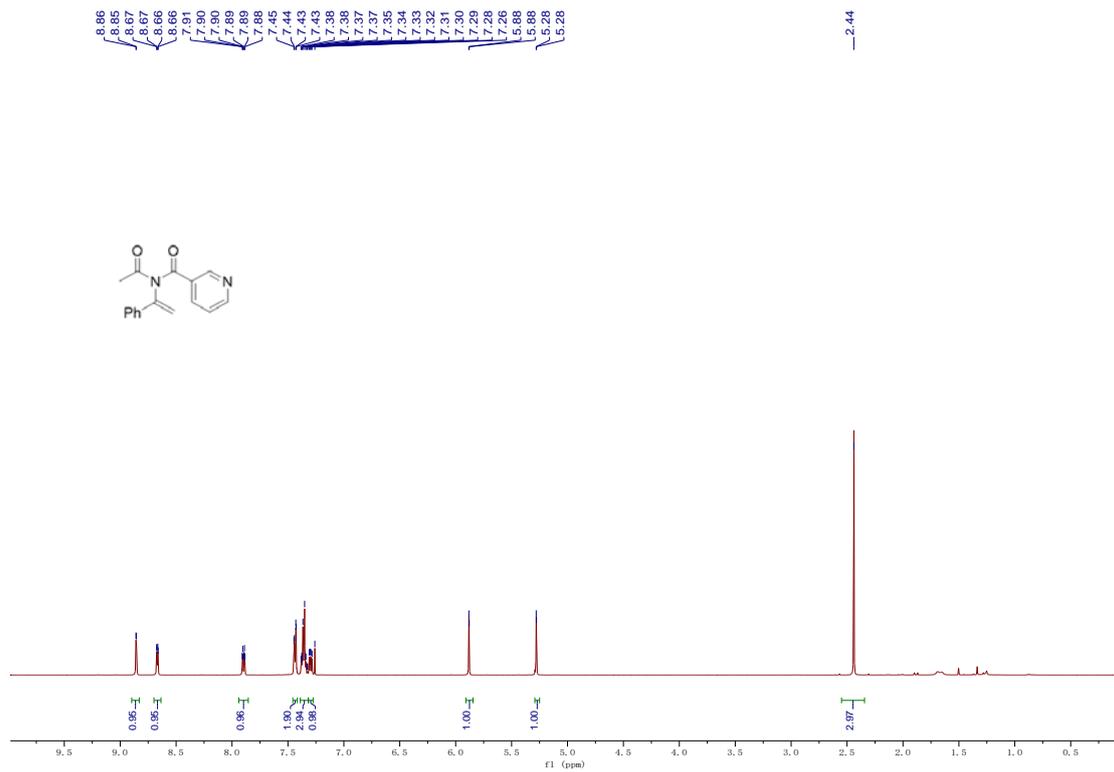


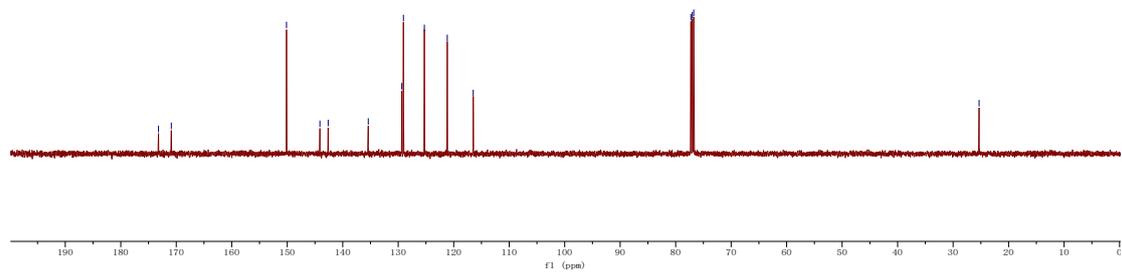
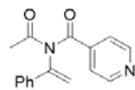
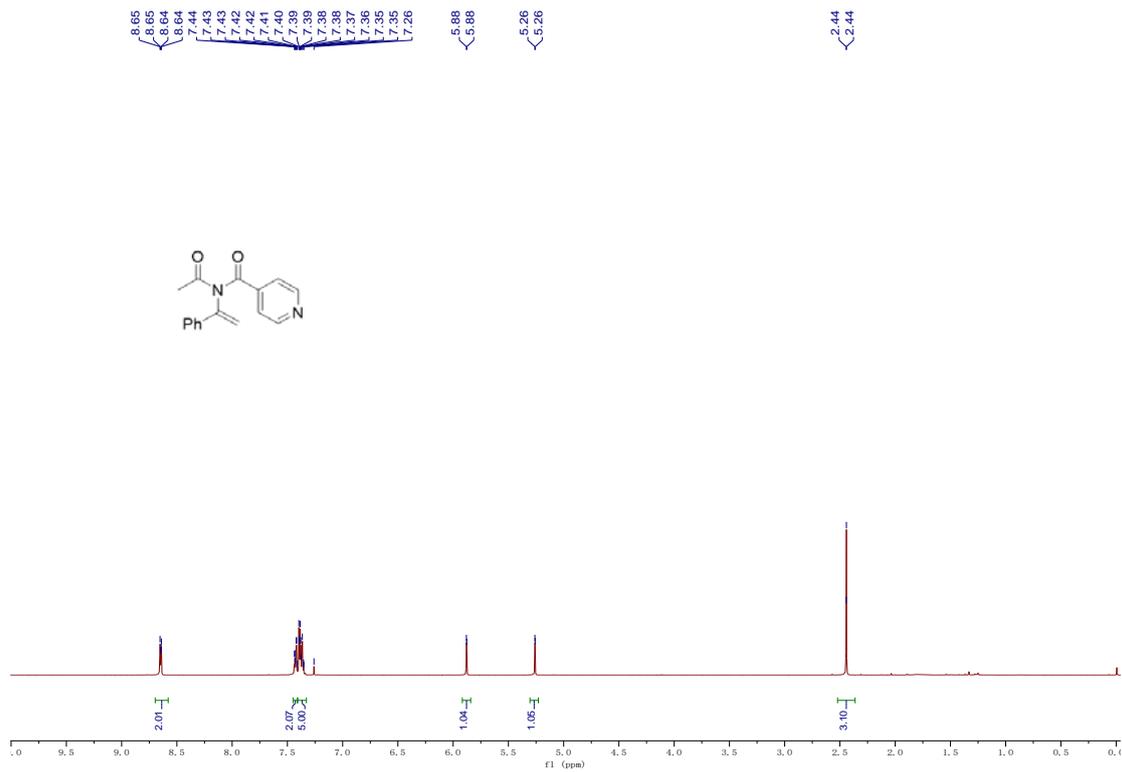


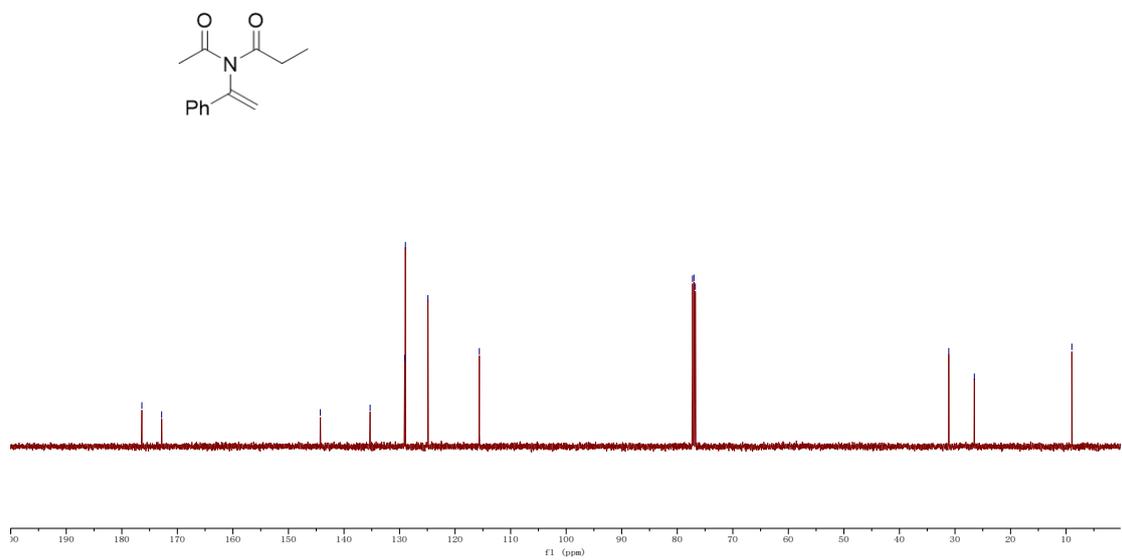
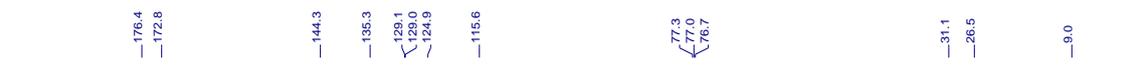
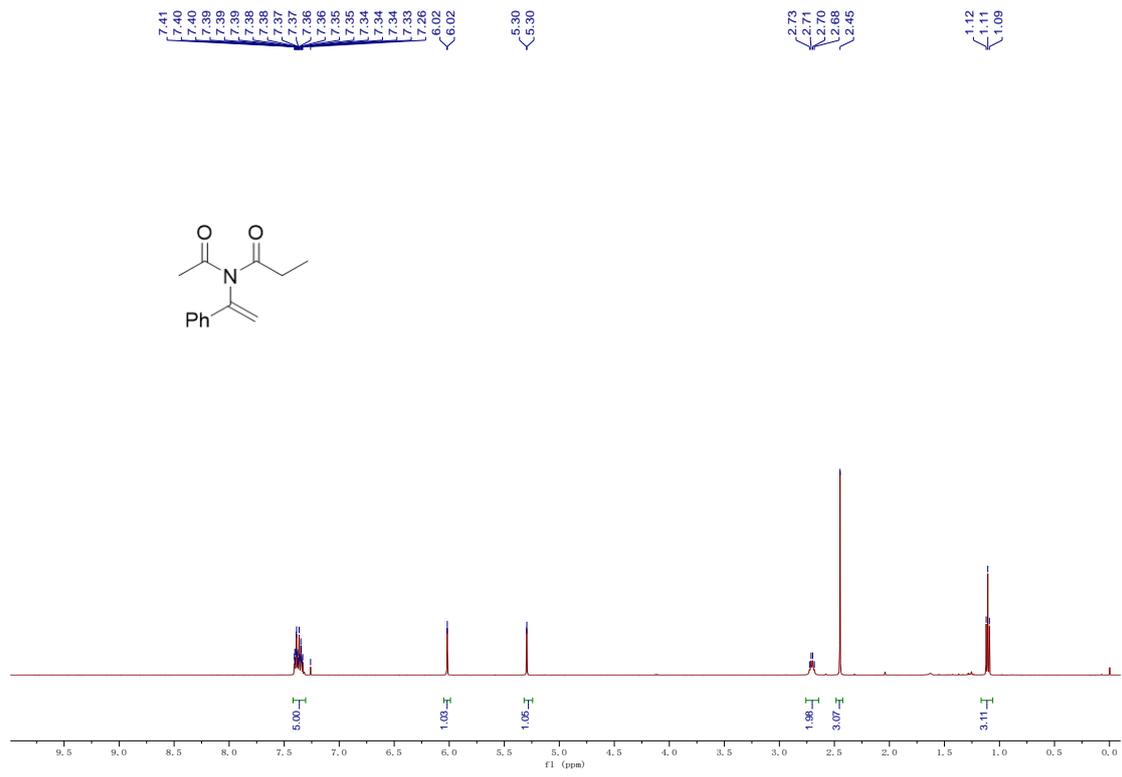


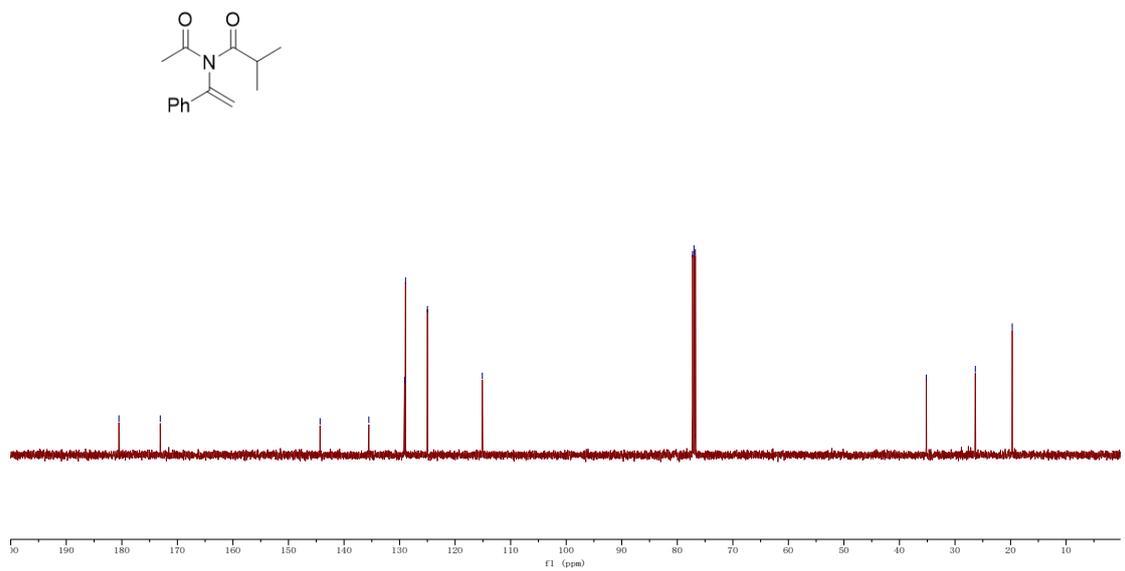
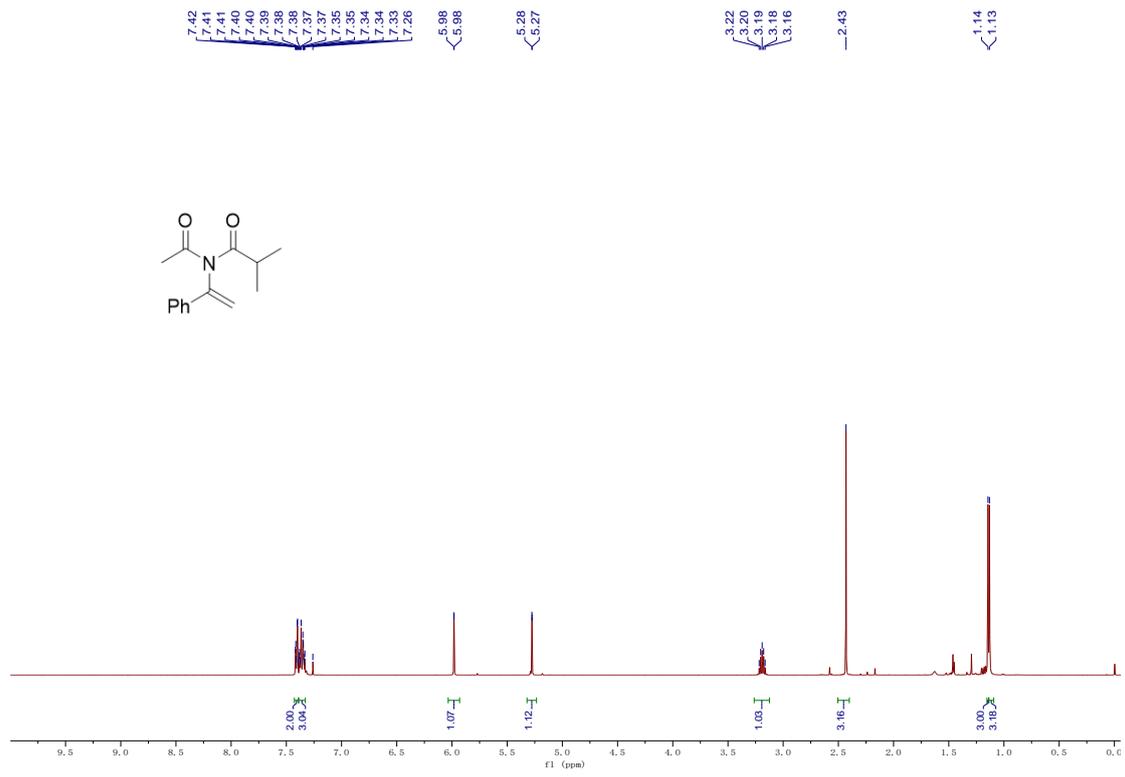


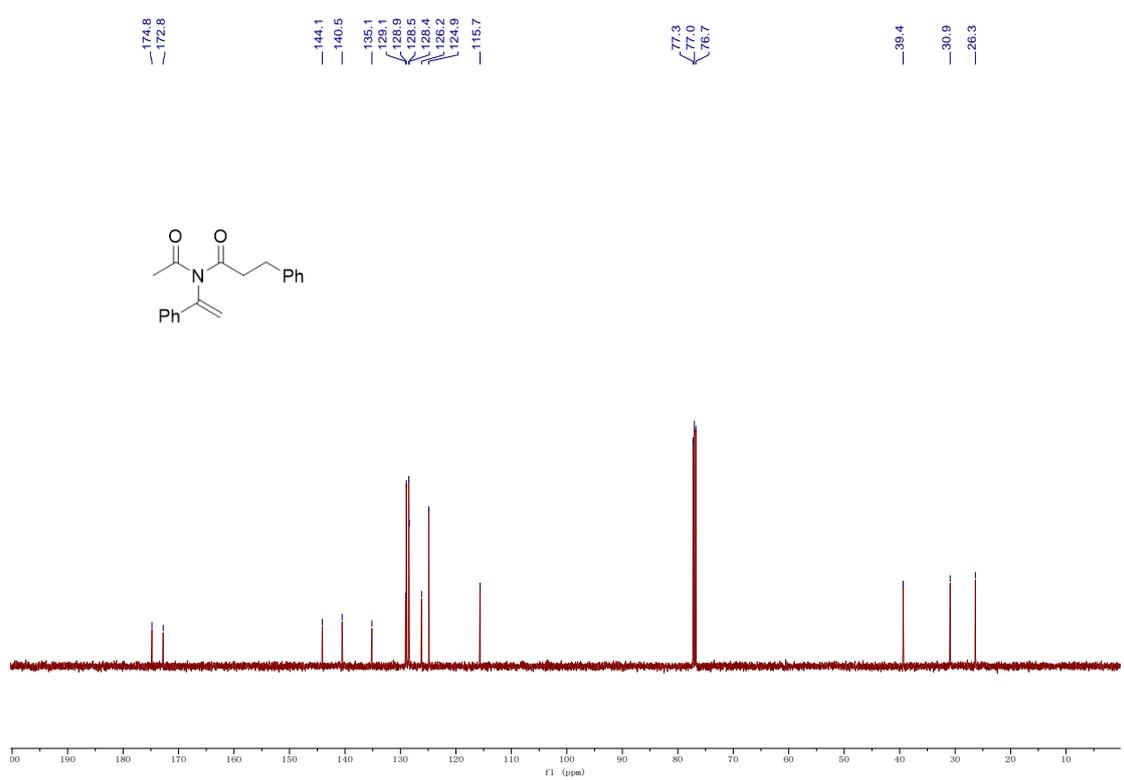
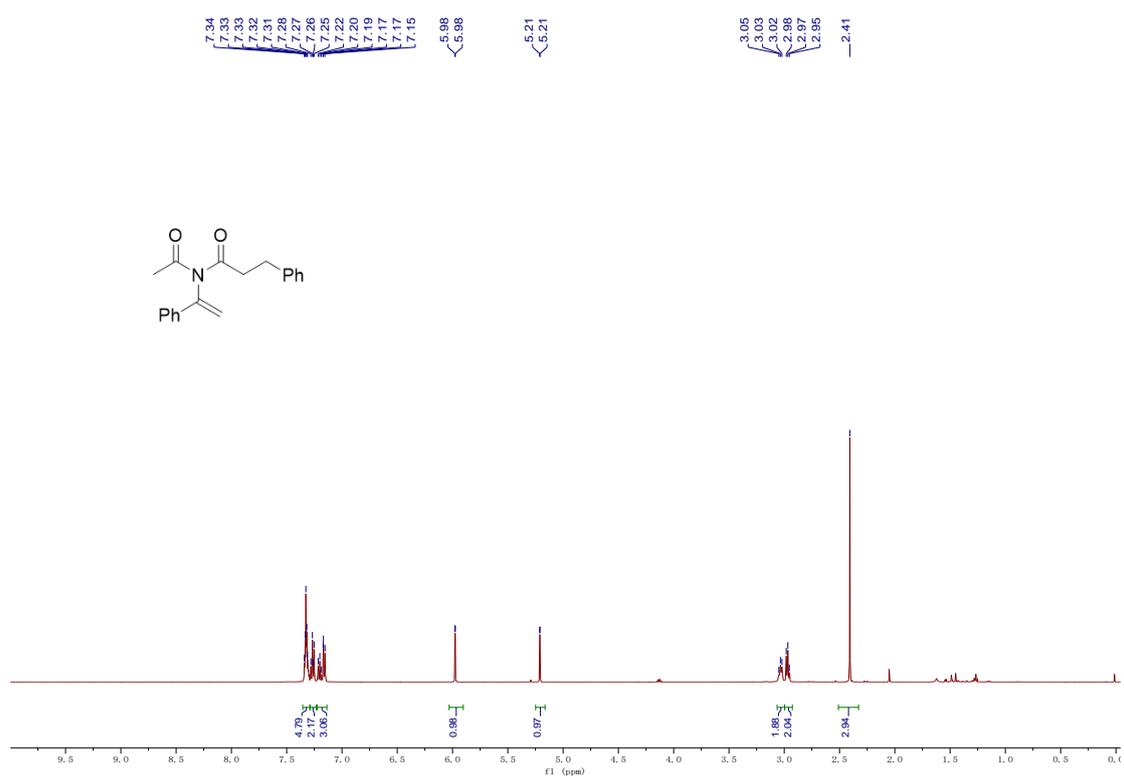


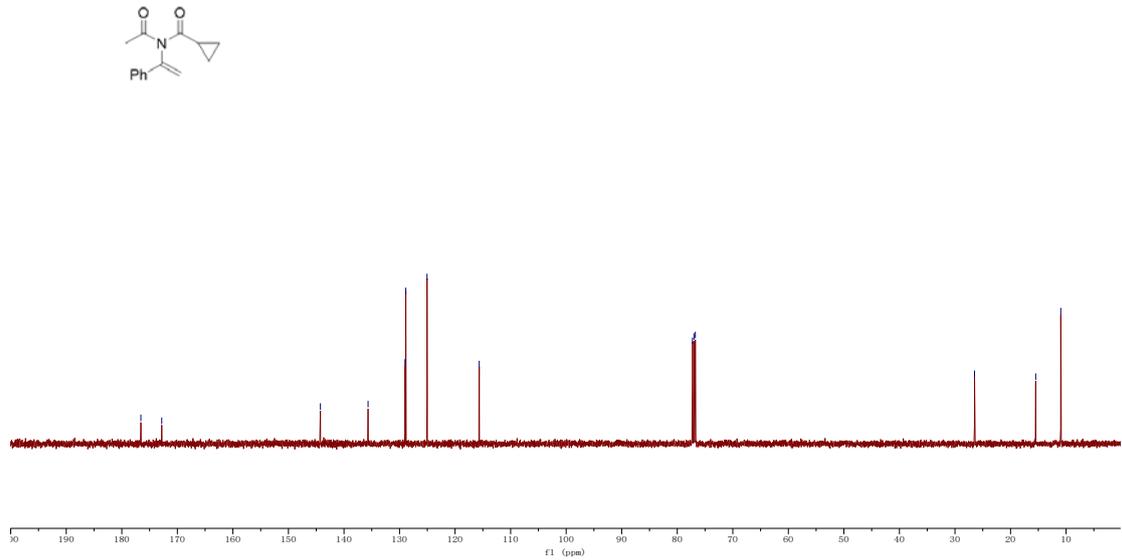
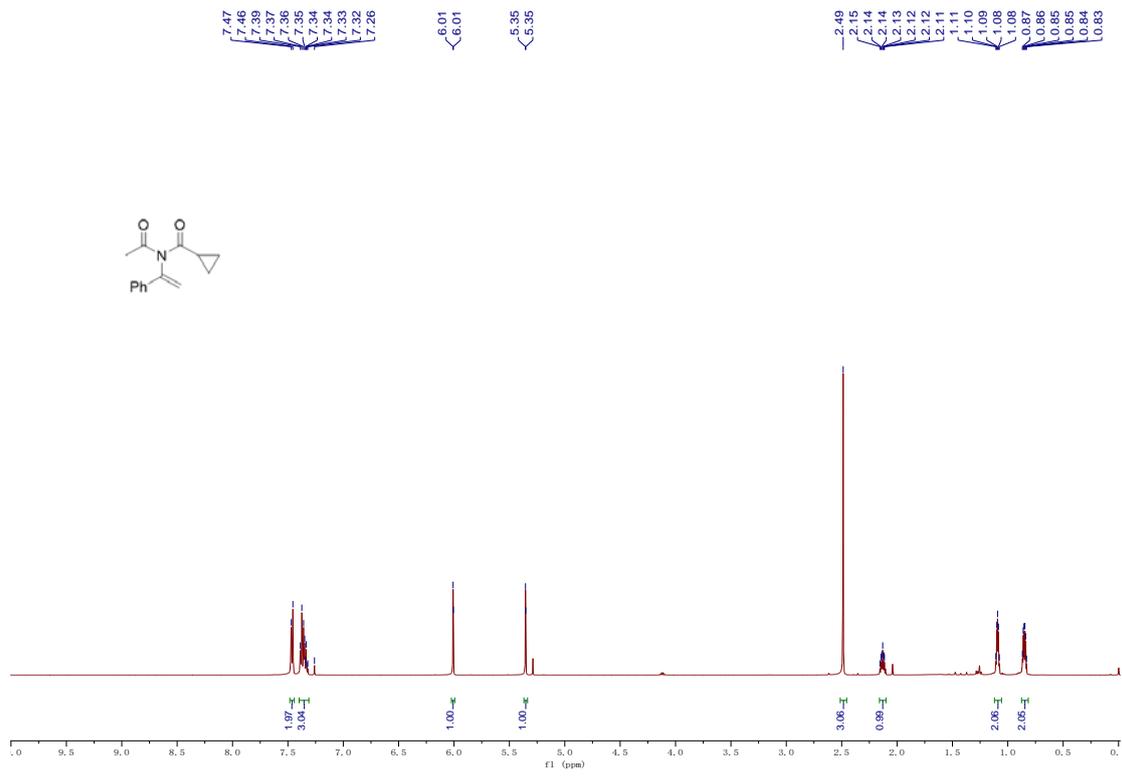


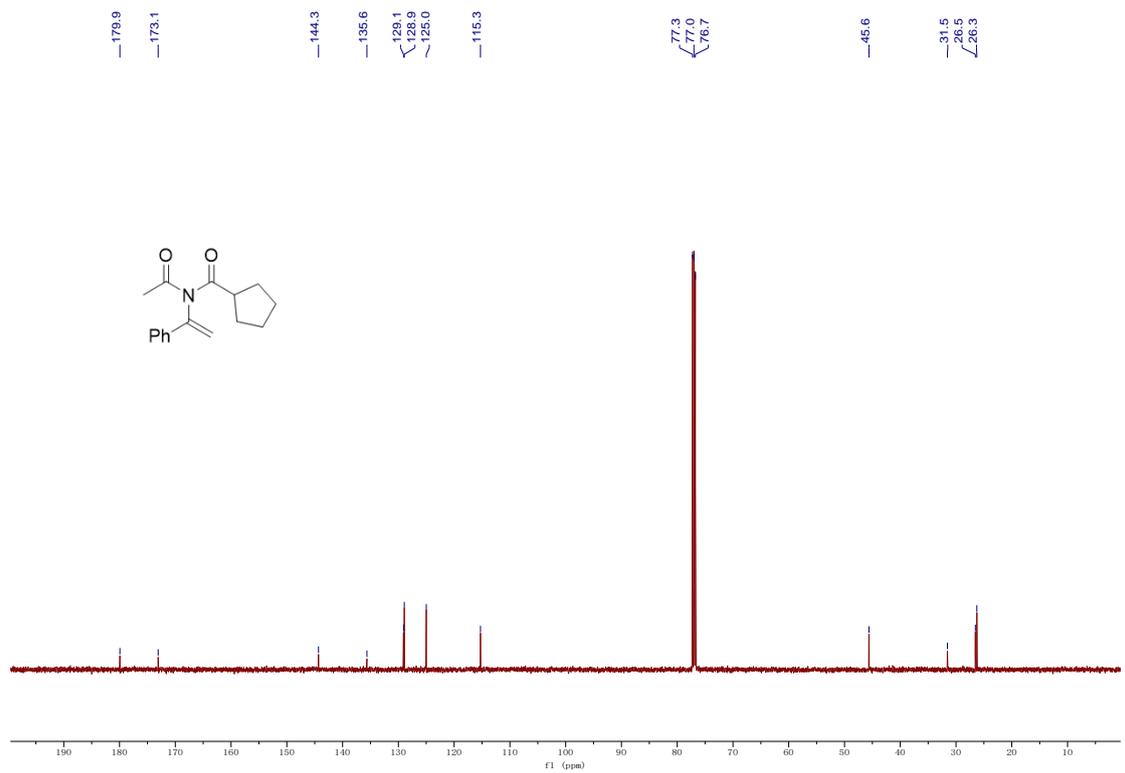
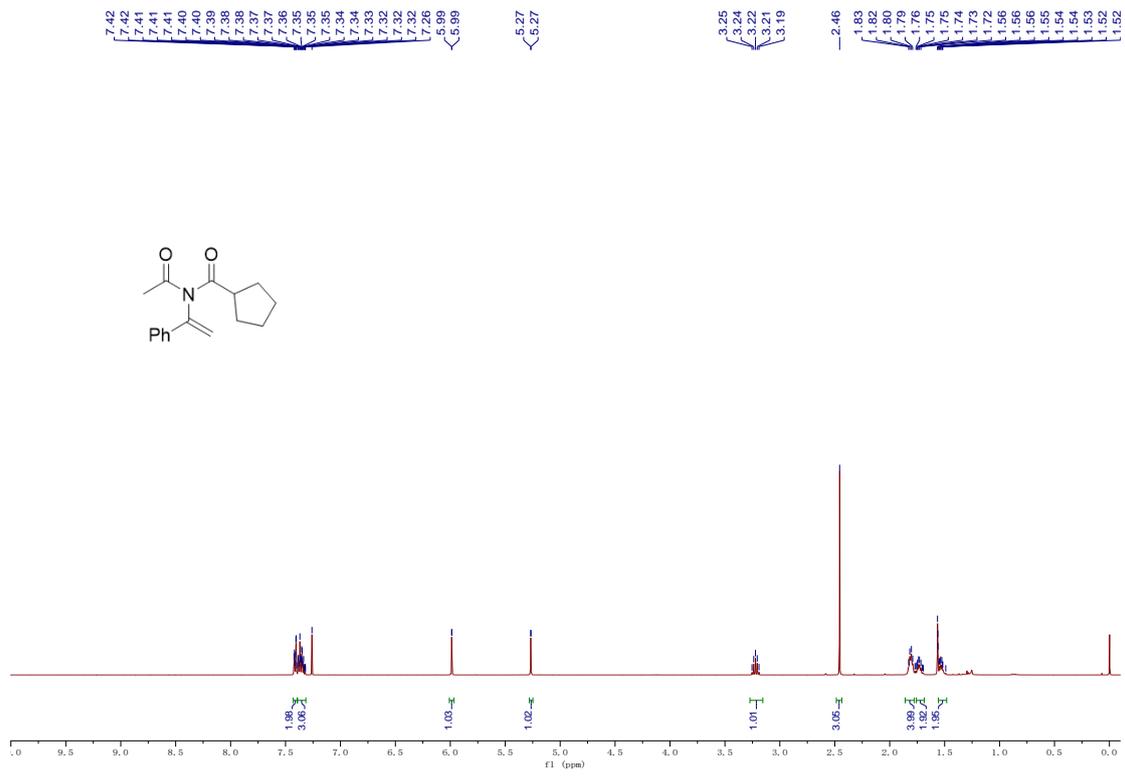


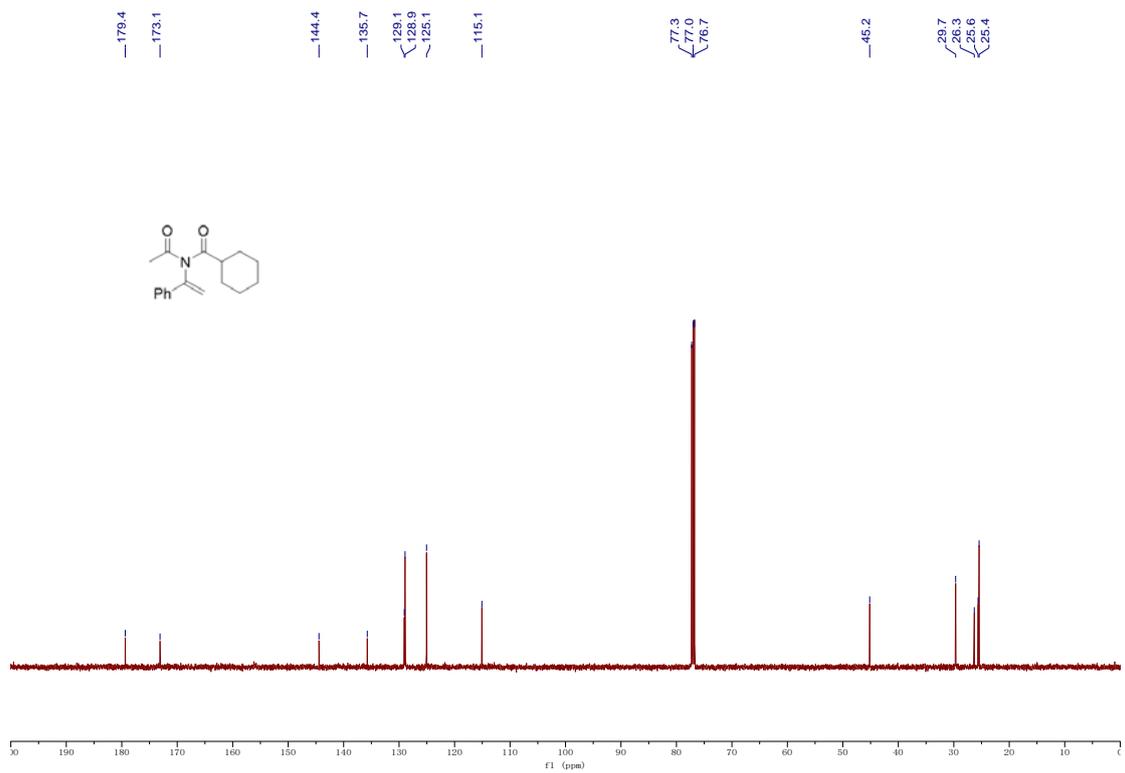
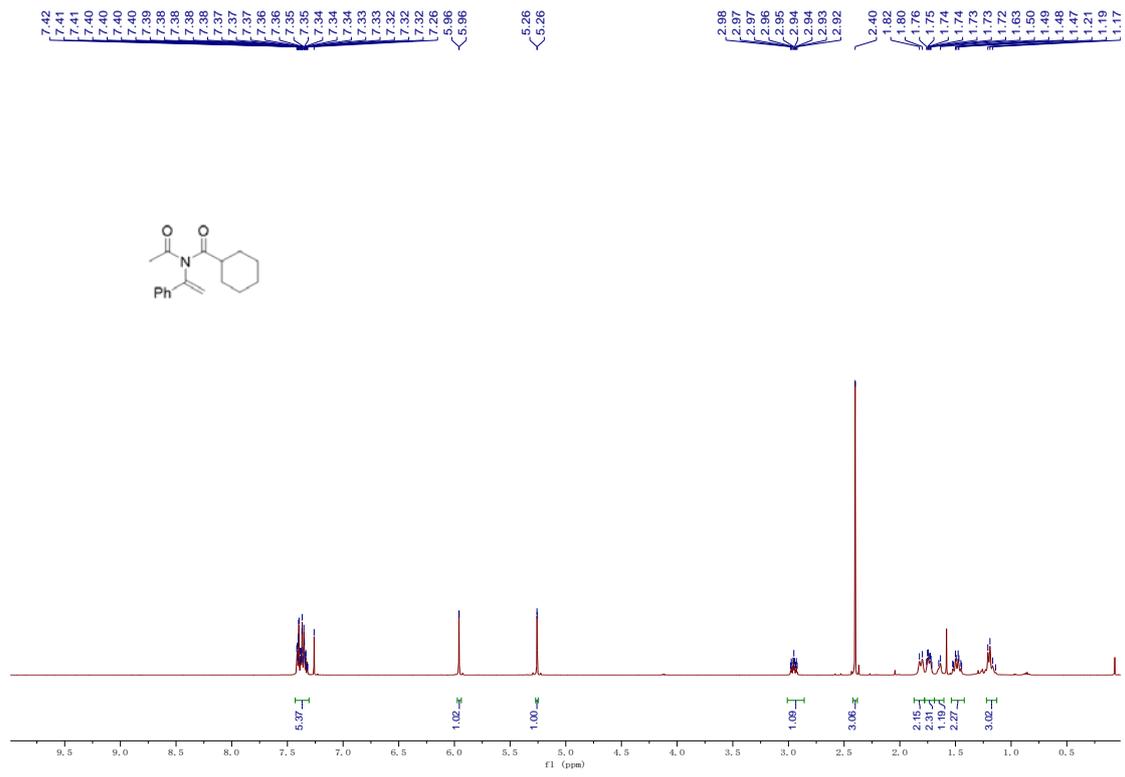


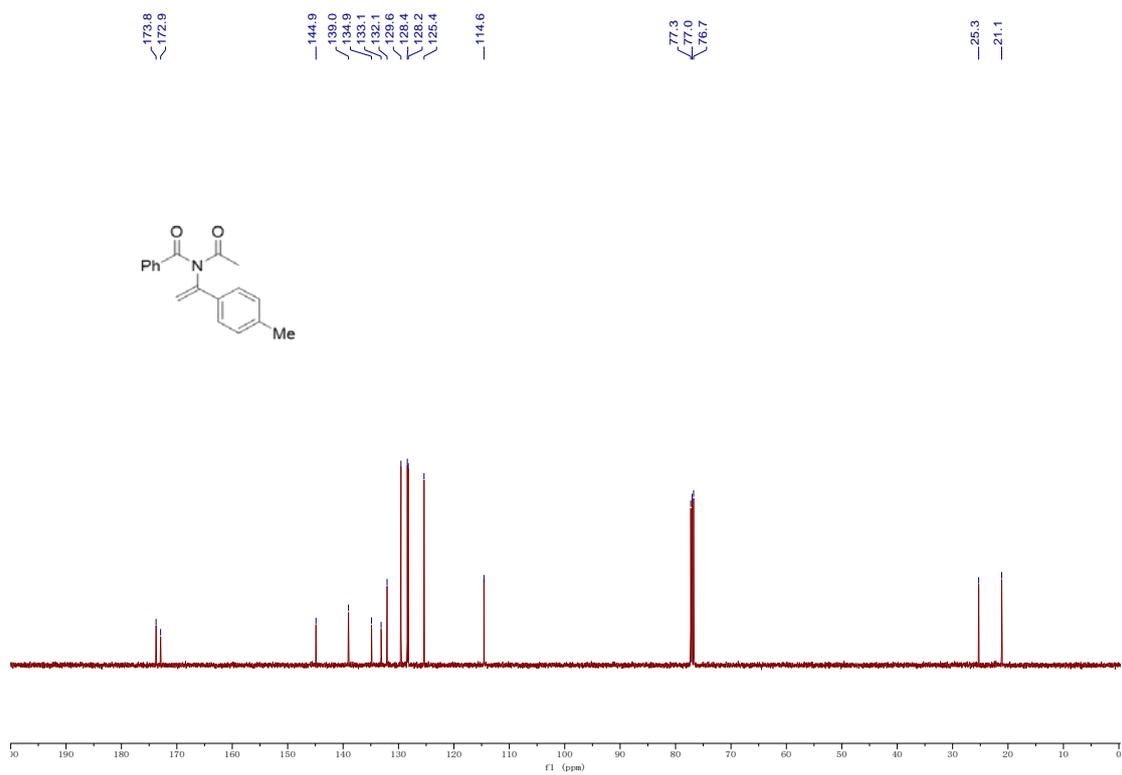
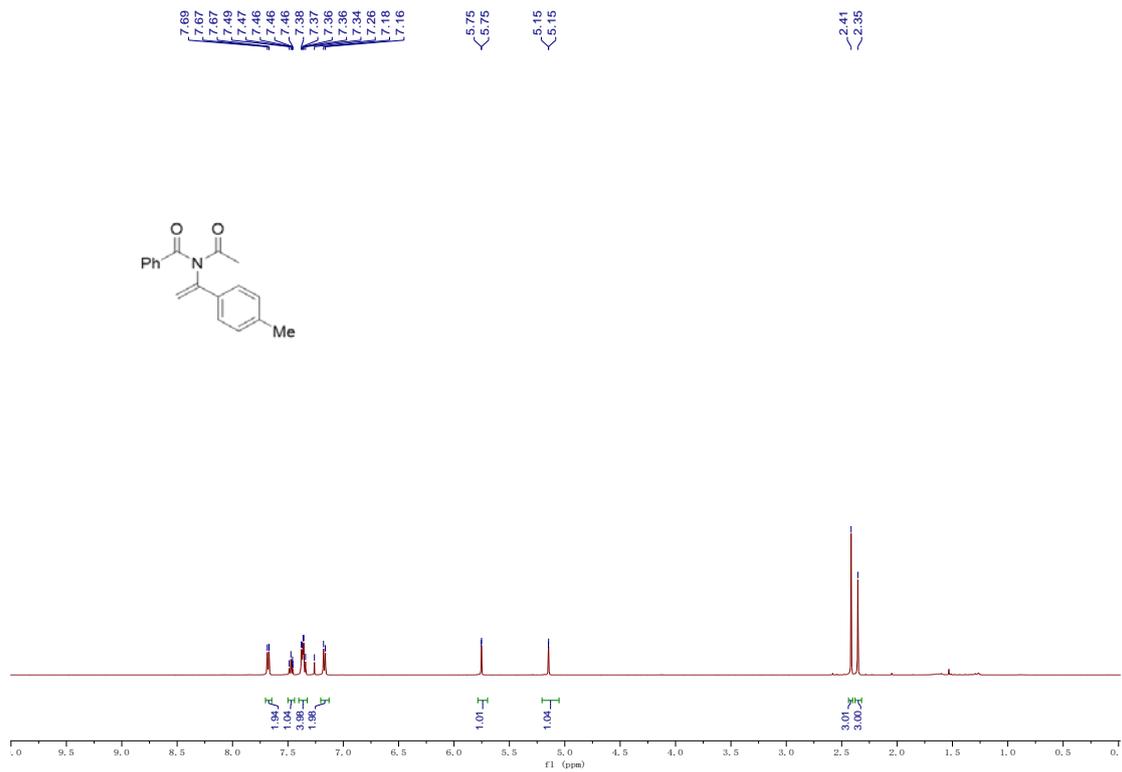


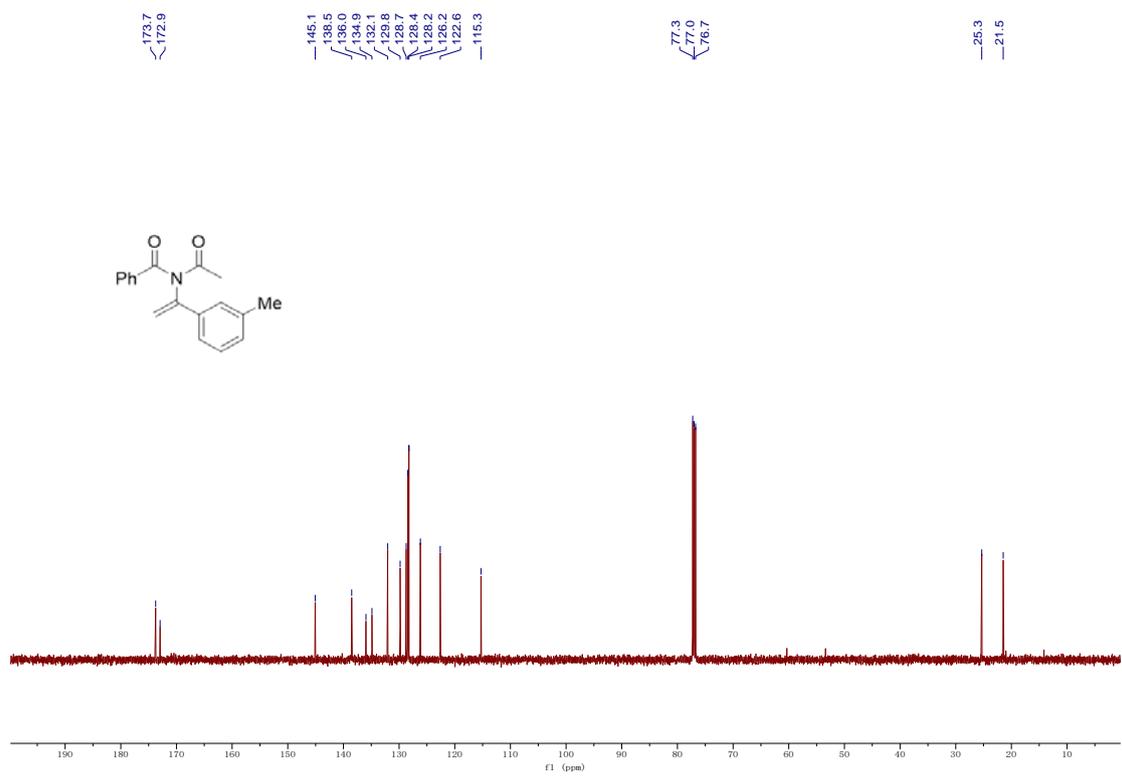
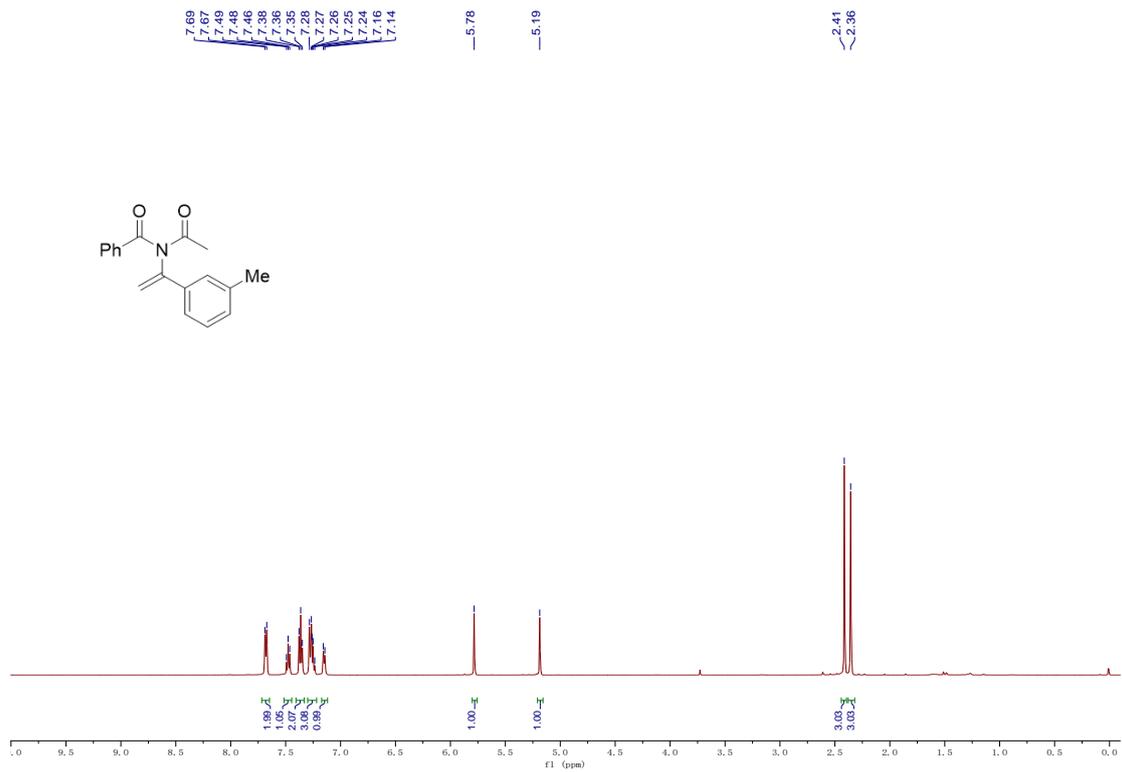


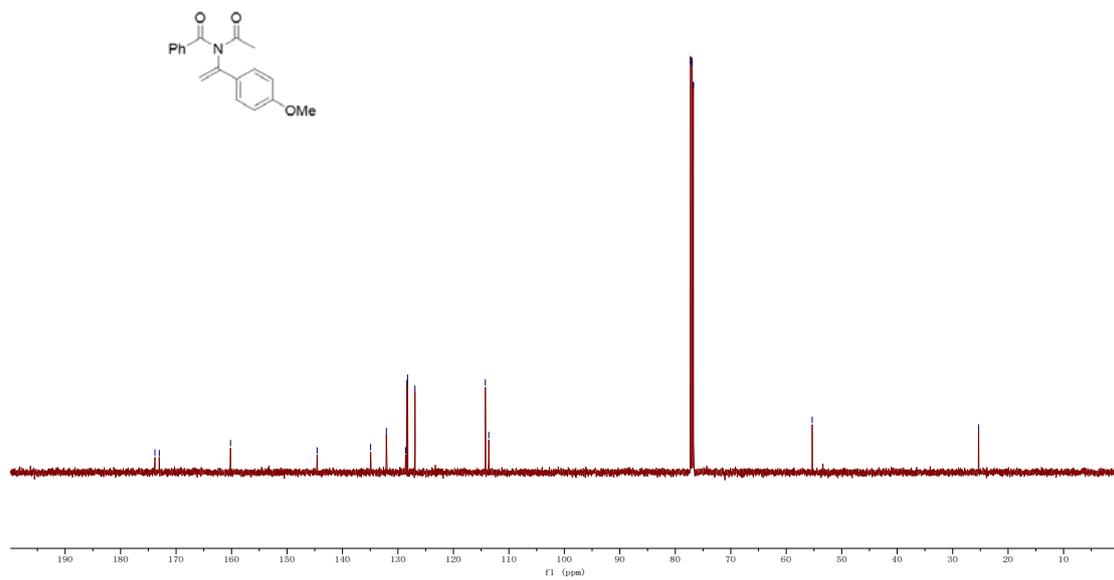
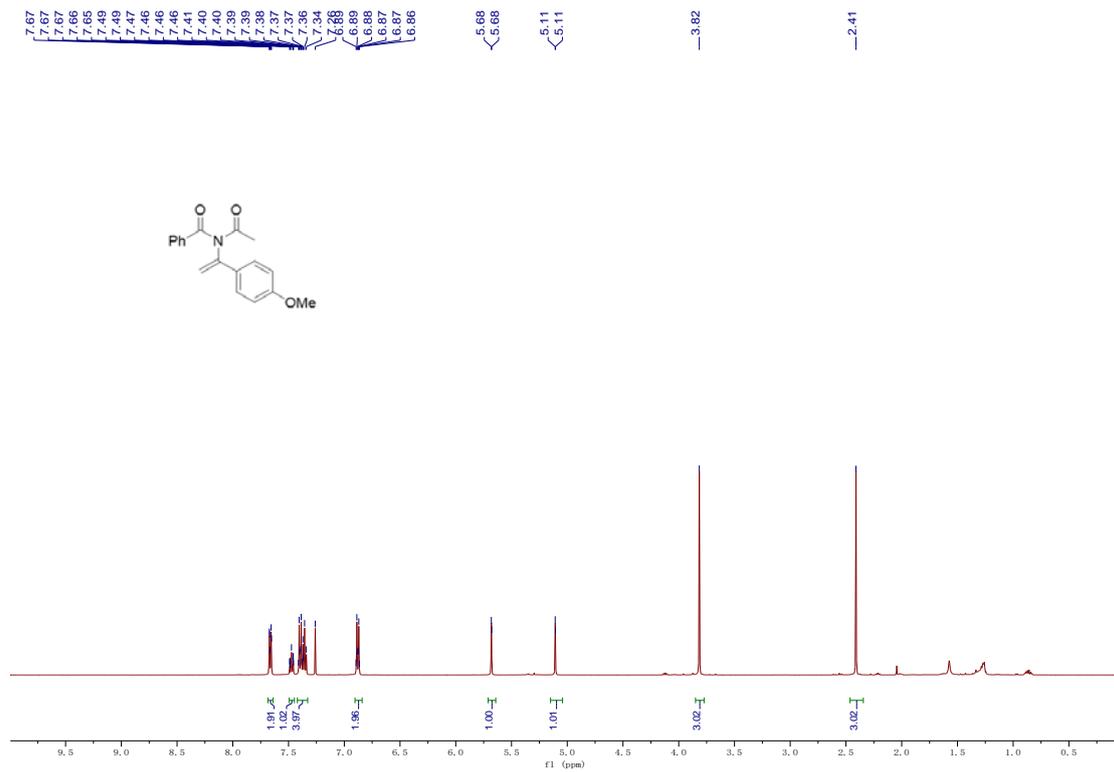


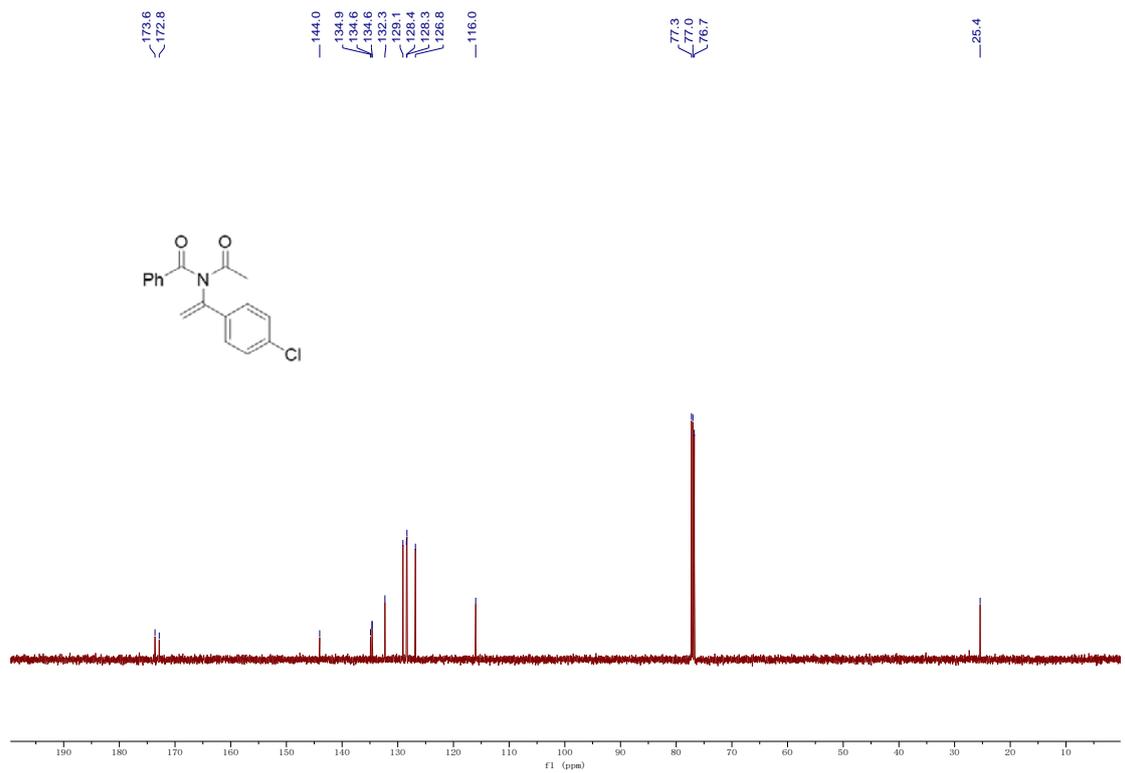
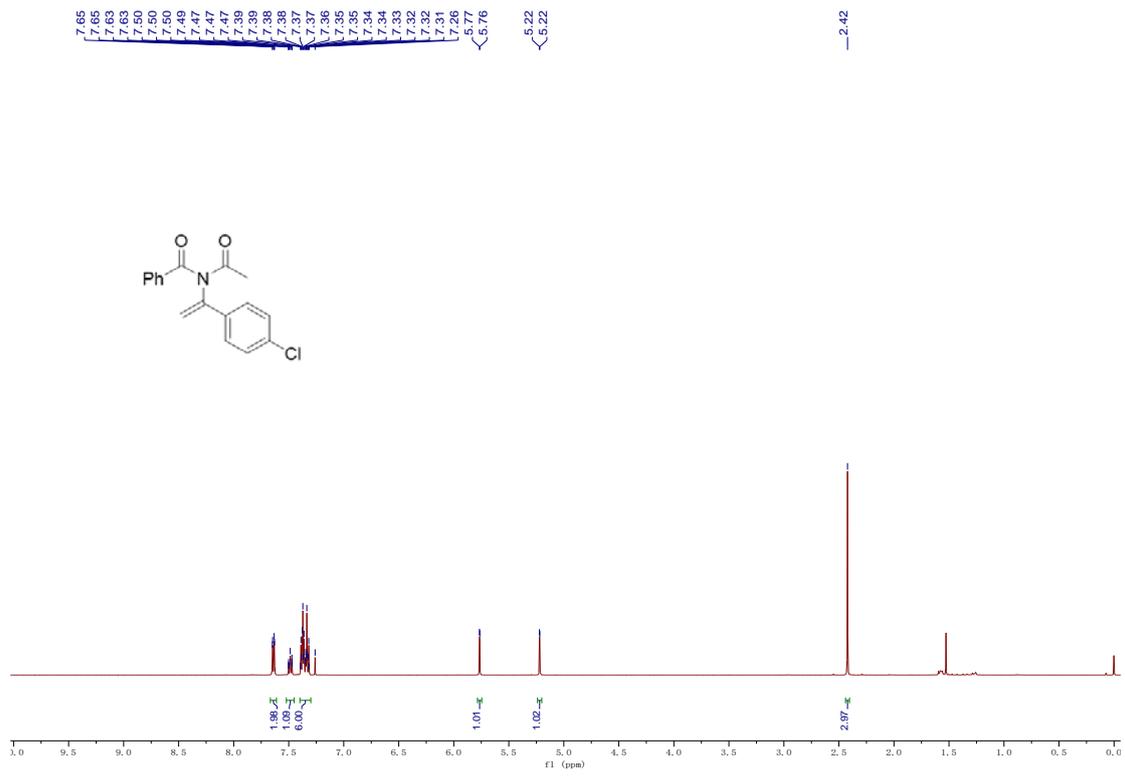






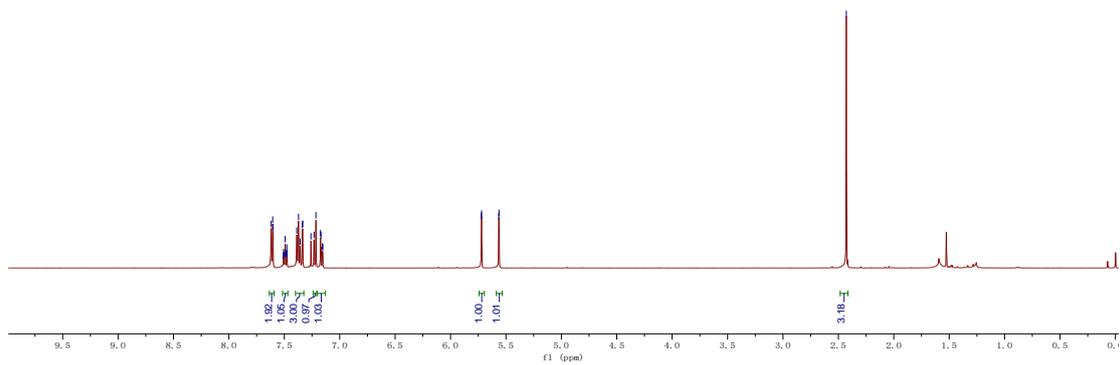
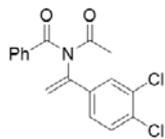






7.62  
7.60  
7.60  
7.51  
7.50  
7.49  
7.48  
7.46  
7.39  
7.37  
7.36  
7.34  
7.33  
7.26  
7.23  
7.21  
7.17  
7.16  
7.15  
5.72  
5.57  
5.56

2.43



173.3  
172.8

140.7  
134.9  
134.9  
134.1  
132.7  
131.5  
130.2  
128.8  
128.4  
127.3  
120.6

77.3  
77.0  
76.7

25.4

