

*Supporting Information for*

**Merging deoxygenation with acyl migration process:  
photoredox-catalyzed synthesis of  $\alpha$ -amido- $\alpha$ -  
(hetero)aryl-1,4-diketones**

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# 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 enimides were prepared according to our previous works.<sup>1-3</sup> Carboxylic acids, photocatalysts, and dried solvents were all purchased from local suppliers.

## 1.2 Instruments

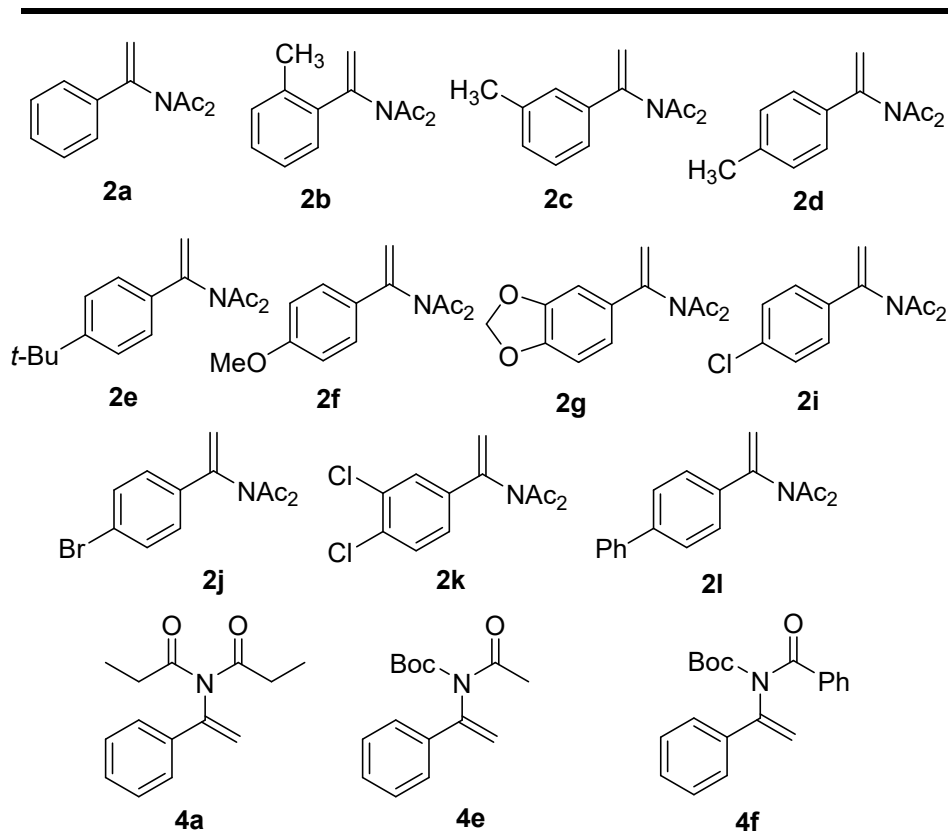
NMR spectra were recorded on the Bruker Avance 500 spectrometer (500 MHz). Chemical shifts were reported in ppm downfield from tetramethylsilane, and calibrated using residue undeuterated solvent (CHCl<sub>3</sub> at 7.26 ppm <sup>1</sup>H NMR, 77.0 ppm <sup>13</sup>C NMR). Spectra were reported as follows: 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 UV254 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

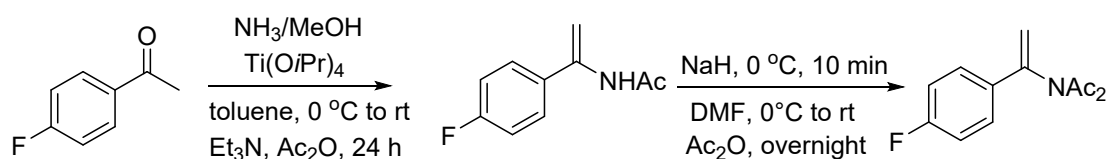


## 2. Preparation of *N*-vinylimides

### 2.1 Known enimides reported in our previous work<sup>1-2,4-6</sup>



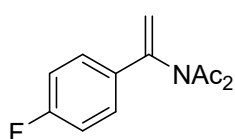
### 2.2 Synthesis of *N*-vinylimides<sup>7</sup>



(1) To a 100 mL Schlenk tube was added 4-fluorophenophenone (1.38 g, 10.0 mmol, 1.0 equivalent) and anhydrous toluene (6 mL) under nitrogen. The mixed solution was stirred and cooled to  $0^\circ\text{C}$ . To the obtained solution was added 7 N  $\text{NH}_3$  in MeOH (2.2 mL, 15.0 mmol, 1.5 equiv). Then  $\text{Ti}(\text{O}i\text{-Pr})_4$  (5.95 mL, 20.0 mmol, 2.0 equiv) was added dropwise and the final solution was continued to stir for 24 h at room temperature. The reaction mixture was then cooled in  $-5^\circ\text{C}$  and added  $\text{Et}_3\text{N}$  (5.58 mL, 40 mmol, 4.0 equiv) followed by  $\text{Ac}_2\text{O}$  (1.89 mL, 20 mmol, 2.0 equiv). The mixed solution was stirred at room temperature for 3 h. Then the mixed solutions was added

*N,N,N',N'*-tetrakis (2-hydroxyethyl) ethylenediamine (75% W.t., 6.0 mL, 21.0 mmol, 2.1 equiv) at room temperature and the solution was warmed at 55°C for 20 min. The completion of the reaction was cooled to room temperature and added with NH<sub>4</sub>OH (15 mL), water (15 mL), and EtOAc (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.

(2) The obtained enamide (5 mmol) was dissolved in dry DMF (10 mL) in a dry Schlenk tube under nitrogen. The mixed solution was cooled to 0 °C and sodium hydride (60% dispersion in mineral oil) (300 mg, 7.5 mmol) was added in portions. The resulting suspension was stirred at the same temperature for 15 min. Then Ac<sub>2</sub>O (0.95 mL, 10.0 mmol, 2.0 equiv) was added dropwise and the final solution was stirred for overnight at room temperature. After the reaction was complete, the reaction solution was quenched by adding 5 mL water at 0 °C. 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.



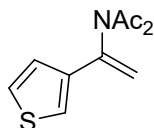
***N*-Acetyl-*N*-(1-(4-fluorophenyl)vinyl)acetamide (2h).** White solid.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.41 – 7.36 (m, 2H), 7.09 – 7.03 (m, 2H), 5.95 (d, *J* = 1.4 Hz, 1H), 5.29 (d, *J* = 1.7 Hz, 1H), 2.40 (s, 6H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 172.7, 163.2 (d, *J* = 250.0 Hz), 143.7, 131.5, 126.9 (d, *J* = 8.2 Hz), 116.0 (d, *J* = 21.9 Hz), 115.5, 26.2.

**<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)** δ -112.0.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>12</sub>H<sub>12</sub>NO<sub>2</sub>NaF<sup>+</sup>: 244.0744, found 244.0753.

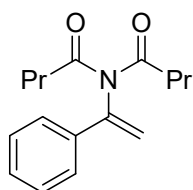


***N*-Acetyl-*N*-(1-(thiophen-3-yl)vinyl)acetamide (2m).** Yellow oil.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.33 (dd, *J* = 5.1, 3.0 Hz, 1H), 7.21 (dd, *J* = 5.1, 1.4 Hz, 1H), 7.15 (dd, *J* = 3.0, 1.4 Hz, 1H), 5.88 (d, *J* = 1.2 Hz, 1H), 5.25 (d, *J* = 1.0 Hz, 1H), 2.41 (s, 6H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 172.6, 140.4, 138.2, 127.3, 124.9, 122.2, 115.0, 26.2.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>10</sub>H<sub>11</sub>NO<sub>2</sub>Na<sup>+</sup>: 232.0403, found 232.0411.

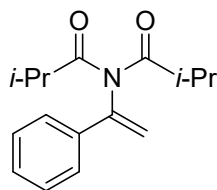


***N*-Pentanoyl-*N*-(1-phenylvinyl)pentanamide (4b).** White solid.

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.41 – 7.30 (m, 5H), 6.00 (s, 1H), 5.26 (s, 1H), 2.70 (t, *J* = 7.3 Hz, 4H), 1.70 – 1.62 (m, 4H), 0.92 (t, *J* = 7.4 Hz, 6H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 175.8, 144.2, 135.6, 129.1, 129.0, 125.0, 115.5, 39.9, 18.3, 13.6.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>16</sub>H<sub>21</sub>NO<sub>2</sub>Na<sup>+</sup>: 282.1464, found 282.1474.



***N*-Isobutyryl-*N*-(1-phenylvinyl)isobutyramide (4c).** Yellow oil.

**<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 (m, 2H), 1.16 (d, *J* = 6.7 Hz, 12H).

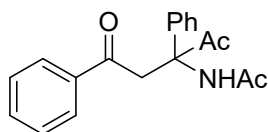
**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 181.1, 144.5, 136.0, 129.1, 128.9, 125.1, 114.5, 35.4, 19.8.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>16</sub>H<sub>21</sub>NO<sub>2</sub>Na<sup>+</sup>: 282.1464, found 282.1472.

### 3. General procedure of photoredox-catalyzed acyl migration or Giese-type reactions



To a 10 mL dried Schlenk tube equipped with a magnetic stirrer was added  $\text{Ir}[\text{dF}(\text{CF}_3)\text{ppy}]_2(\text{dtbbpy})\text{PF}_6$  (4.5 mg, 0.004 mmol, 0.02 equiv),  $\text{K}_2\text{HPO}_4$  (69.6 mg, 0.4 mmol, 2.0 equiv),  $\text{PPh}_3$  (104.9 mg, 0.4 mmol, 2.0 equiv), *N*-vinylimide (0.2 mmol, 1.0 equiv), and carboxylic acid (0.4 mmol, 2.0 equiv). The reaction tube was allowed to be vacuumed and purged with nitrogen for three times. Then, degassed  $\text{CH}_3\text{CN}$  (3 mL) were added under nitrogen atmosphere. The reaction mixture was irradiated with 9 W blue LEDs at room temperature for 48 h. After the reaction was complete, the reaction solution was quenched by adding water (5 mL). The aqueous layer was extracted with ethyl acetate ( $3 \times 10 \text{ mL}$ ), 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.

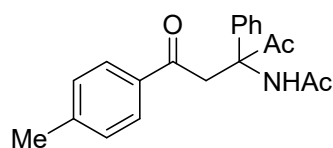


***N*-(1,4-Dioxo-1,3-diphenylpentan-3-yl)acetamide (3a).** Yellow solid (39.6 mg, 64% yield).

$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.00 (d,  $J = 7.4 \text{ Hz}$ , 2H), 7.60 – 7.56 (m, 2H), 7.48 – 7.42 (m, 4H), 7.38 – 7.35 (m, 2H), 7.31 – 7.28 (m, 1H), 5.17 (d,  $J = 17.5 \text{ Hz}$ , 1H), 3.88 (d,  $J = 17.5 \text{ Hz}$ , 1H), 2.02 (s, 3H), 1.91 (s, 3H).

$^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  203.0, 198.0, 169.5, 137.7, 136.5, 133.8, 129.0, 128.7, 128.3, 128.1, 125.7, 67.5, 41.0, 23.6, 23.1.

The analytical data were in good agreement with our previous report.<sup>6</sup>

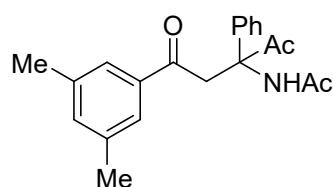


***N*-(1,4-Dioxo-3-phenyl-1-(*p*-tolyl)pentan-3-yl)acetamide (3b).** Yellow solid (38.5 mg, 60% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)**  $\delta$  7.90 (d,  $J$  = 8.2 Hz, 2H), 7.52 (brs, 1H), 7.45 – 7.40 (m, 2H), 7.37 (t,  $J$  = 7.7 Hz, 2H), 7.32 – 7.26 (m, 3H), 5.14 (d,  $J$  = 17.3 Hz, 1H), 3.84 (d,  $J$  = 17.3 Hz, 1H), 2.41 (s, 3H), 2.02 (s, 3H), 1.90 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)**  $\delta$  203.1, 197.6, 169.4, 144.8, 137.8, 134.1, 129.4, 129.0, 128.5, 128.1, 125.8, 67.5, 40.9, 23.6, 23.1, 21.7.

The analytical data were in good agreement with our previous report.<sup>6</sup>

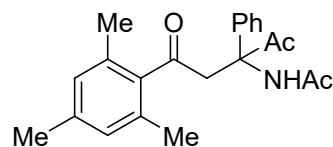


***N*-(1-(3,5-Dimethylphenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3c).** White solid (39.1 mg, 58% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)**  $\delta$  7.61 (s, 2H), 7.51 (brs, 1H), 7.45 – 7.42 (m, 2H), 7.40 – 7.34 (m, 2H), 7.32 – 7.27 (m, 1H), 7.22 (s, 1H), 5.11 (d,  $J$  = 17.7 Hz, 1H), 3.88 (d,  $J$  = 17.7 Hz, 1H), 2.36 (s, 6H), 2.00 (s, 3H), 1.92 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)**  $\delta$  203.0, 198.2, 169.4, 138.4, 137.8, 136.5, 135.4, 129.0, 128.1, 126.1, 125.7, 67.4, 41.2, 23.6, 23.1, 21.2.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>21</sub>H<sub>23</sub>NO<sub>3</sub>Na<sup>+</sup>: 360.1570, found 360.1569.



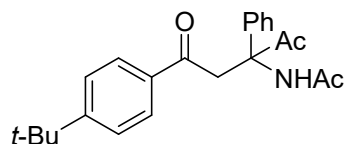
***N*-(1-Mesityl-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3d).** White solid (38.6 mg, 55% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)**  $\delta$  7.61 (brs, 1H), 7.30 – 7.42 (m, 4H), 7.32 – 7.27 (m, 1H), 6.82 (s, 2H), 4.64 (d,  $J$  = 19.1 Hz, 1H), 3.81 (d,  $J$  = 19.1 Hz, 1H), 2.27 (s, 3H),

2.15 (s, 6H), 2.00(2) (s, 3H), 1.99(5) (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  208.0, 202.6, 169.2, 138.9, 138.1, 137.3, 132.6, 129.0, 128.7, 128.2, 125.7, 67.0, 47.3, 23.6, 23.1, 21.0, 18.8.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{22}\text{H}_{25}\text{NO}_3\text{Na}^+$ : 374.1727, found 374.1733.

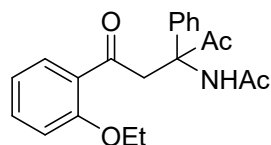


***N*-(1-(4-(*tert*-Butyl)phenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3e).** White solid (44.6 mg, 61% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.94 (d,  $J = 8.5$  Hz, 2H), 7.55 (brs, 1H), 7.48 (d,  $J = 8.5$  Hz, 2H), 7.45 – 7.41 (m, 2H), 7.39 – 7.34 (m, 2H), 7.32 – 7.27 (m, 1H), 5.15 (d,  $J = 17.4$  Hz, 1H), 3.86 (d,  $J = 17.5$  Hz, 1H), 2.02 (s, 3H), 1.90 (s, 3H), 1.33 (s, 9H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  203.0, 197.6, 169.4, 157.7, 137.8, 134.0, 129.0, 128.3, 128.1, 125.7(4), 125.6(8), 67.5, 41.0, 35.1, 31.0, 23.6, 23.1.

HRMS (ESI)  $[\text{M}+\text{H}]^+$ : calculated for  $\text{C}_{23}\text{H}_{28}\text{NO}_3^+$ : 366.2064, found 366.2073.

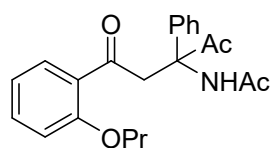


***N*-(1-(2-Ethoxyphenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3f).** Yellow solid (42.4 mg, 60% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.68 (dd,  $J = 7.9, 1.9$  Hz, 1H), 7.52 (brs, 1H), 7.48 – 7.40 (m, 3H), 7.35 (t,  $J = 7.7$  Hz, 2H), 7.30 – 7.27 (m, 1H), 6.99 – 6.93 (m, 2H), 5.10 (d,  $J = 18.6$  Hz, 1H), 4.21 (q,  $J = 6.9$  Hz, 2H), 4.12 (d,  $J = 18.6$  Hz, 1H), 1.99 (s, 3H), 1.90 (s, 3H), 1.64 (t,  $J = 7.0$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  203.2, 199.0, 168.9, 158.6, 138.2, 134.3, 130.2, 128.9, 128.0, 127.3, 125.9, 120.5, 112.4, 67.3, 64.2, 46.8, 23.7, 23.1, 14.9.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{21}\text{H}_{23}\text{NO}_4\text{Na}^+$ : 376.1519, found 376.1527.



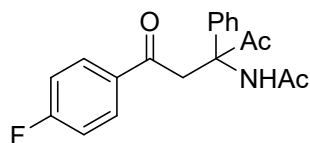
***N*-(1,4-Dioxo-3-phenyl-1-(2-propoxyphenyl)pentan-3-yl)acetamide (3g).** Yellow

solid (42.6 mg, 58% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.68 (dd, *J* = 8.0, 1.8 Hz, 1H), 7.51 (s, 1H), 7.48 – 7.40 (m, 3H), 7.35 (t, *J* = 7.7 Hz, 2H), 7.30 – 7.26 (m, 1H), 6.98 – 6.95 (m, 2H), 5.11 (d, *J* = 18.7 Hz, 1H), 4.15– 4.05 (m, 3H), 2.05 (p, *J* = 7.1 Hz, 2H), 1.99 (s, 3H), 1.91 (s, 3H), 1.19 (t, *J* = 7.4 Hz, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 203.2, 199.0, 168.9, 158.8, 138.2, 134.4, 130.3, 128.8, 128.0, 127.2, 125.9, 120.5, 112.4, 70.3, 67.4, 46.9, 23.7, 23.1, 22.7, 10.9.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>22</sub>H<sub>25</sub>NO<sub>4</sub>Na<sup>+</sup>: 390.1676, found 390.1682.



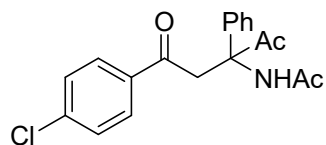
***N*-(1-(4-Fluorophenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3h)**. Yellow oil (36.1 mg, 55% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 8.04 – 8.01 (m, 2H), 7.54 (brs, 1H), 7.43 – 7.40 (m, 2H), 7.38 – 7.35 (m, 2H), 7.31 – 7.28 (m, 1H), 7.14 – 7.10 (m, 2H), 5.13 (d, *J* = 17.3 Hz, 1H), 3.83 (d, *J* = 17.3 Hz, 1H), 2.02 (s, 3H), 1.90 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 202.9, 196.5, 169.5, 166.1 (d, *J* = 256.6 Hz), 137.6, 133.0 (d, *J* = 2.8 Hz), 131.1 (d, *J* = 9.3 Hz), 129.0, 128.2, 125.7, 115.9 (d, *J* = 22.0 Hz), 67.5, 40.9, 23.6, 23.1.

**<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)** δ -103.7.

The analytical data were in good agreement with our previous report.<sup>6</sup>



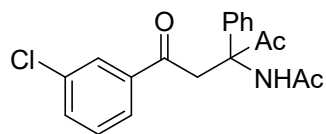
***N*-(1-(4-Chlorophenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3i)**. Yellow oil (43.9 mg, 64% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.95 – 7.92 (m, 2H), 7.51 (brs, 1H), 7.45 – 7.36 (m, 6H), 7.32 – 7.29 (m, 1H), 5.13 (d, *J* = 17.3 Hz, 1H), 3.82 (d, *J* = 17.3 Hz, 1H), 2.02 (s, 3H), 1.91 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 202.9, 197.0, 169.6, 140.4, 137.6, 134.9, 129.8,

129.1, 128.3, 125.7, 67.5, 40.9, 23.6, 23.1.

The analytical data were in good agreement with our previous report.<sup>6</sup>

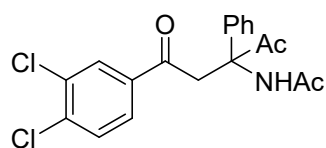


***N*-(1-(3-Chlorophenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3j).** Yellow oil (26.1 mg, 38% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.95 (t, *J* = 1.9 Hz, 1H), 7.89 (dt, *J* = 7.9, 1.4 Hz, 1H), 7.58 – 7.55 (m, 1H), 7.46 (brs, 1H), 7.45 – 7.36 (m, 5H), 7.34 – 7.28 (m, 1H), 5.11 (d, *J* = 17.6 Hz, 1H), 3.86 (d, *J* = 17.6 Hz, 1H), 2.02 (s, 3H), 1.93 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 202.8, 196.8, 169.5, 138.0, 137.5, 135.2, 133.7, 130.1, 129.1, 128.4, 128.3, 126.5, 125.6, 67.5, 41.2, 23.7, 23.0.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>19</sub>H<sub>18</sub>NO<sub>3</sub>NaCl<sup>+</sup>: 366.0867, found 366.0876.

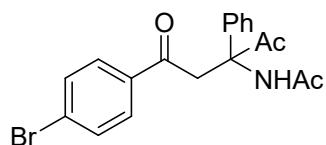


***N*-(1-(3,4-Dichlorophenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3k).** Yellow solid (33.9 mg, 45% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 8.07 (d, *J* = 2.1 Hz, 1H), 7.83 (dd, *J* = 8.4, 2.1 Hz, 1H), 7.55 (d, *J* = 8.4 Hz, 1H), 7.48 (brs, 1H), 7.43 – 7.35 (m, 4H), 7.37 – 7.28 (m, 1H), 5.09 (d, *J* = 17.5 Hz, 1H), 3.83 (d, *J* = 17.5 Hz, 1H), 2.02 (s, 3H), 1.94 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 202.7, 196.0, 169.7, 138.5, 137.4, 136.0, 133.6, 130.9, 130.3, 129.1, 128.4, 127.4, 125.6, 67.5, 41.0, 23.6, 23.0.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>19</sub>H<sub>17</sub>NO<sub>3</sub>NaCl<sub>2</sub><sup>+</sup>: 400.0478, found 400.0486.



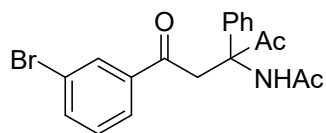
***N*-(1-(4-Bromophenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3l).** Yellow solid (40.3 mg, 52 % yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.88 – 7.84 (m, 2H), 7.64 – 7.59 (m, 2H), 7.47 (s, 1H), 7.43 – 7.28 (m, 4H), 7.34 – 7.28 (m, 1H), 5.12 (d, *J* = 17.3 Hz, 1H), 3.81 (d, *J* = 17.3

Hz, 1H), 2.02 (s, 3H), 1.91 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  202.9, 197.2, 169.5, 137.6, 135.3, 132.1, 129.9, 129.2, 129.1, 128.3, 125.7, 67.5, 40.9, 23.7, 23.1.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{19}\text{H}_{18}\text{NO}_3\text{NaBr}^+$ : 410.0362, found 410.0369.

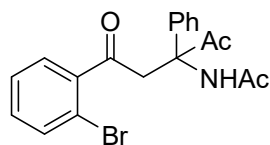


***N*-(1-(3-Bromophenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3m)**. Yellow solid (34.1mg, 44% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.11 (t,  $J = 1.8$  Hz, 1H), 7.93 (d,  $J = 7.7$  Hz, 1H), 7.73-7.68 (m, 1H), 7.50 (brs, 1H), 7.44 – 7.28 (m, 6H), 5.10 (d,  $J = 17.6$  Hz, 1H), 3.87 (d,  $J = 17.6$  Hz, 1H), 2.01 (s, 3H), 1.93 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  202.8, 196.7, 169.6, 138.2, 137.4, 136.6, 131.3, 130.3, 129.1, 128.3, 126.9, 125.6, 123.1, 67.4, 41.1, 23.6, 23.0.

HRMS (ESI)  $[\text{M}+\text{H}]^+$ : calculated for  $\text{C}_{19}\text{H}_{19}\text{NO}_3\text{Br}^+$ : 388.0543, found 388.0551.

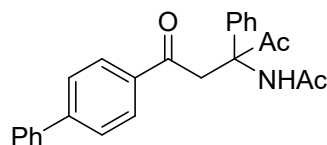


***N*-(1-(2-Bromophenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3n)**. Yellow solid (37.2 mg, 48% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.61 (dd,  $J = 7.9, 1.3$  Hz, 1H), 7.54 (brs, 1H), 7.44 (dd,  $J = 7.6, 1.8$  Hz, 1H), 7.40 – 7.34 (m, 5H), 7.32 – 7.38 (m, 2H), 4.98 (d,  $J = 17.8$  Hz, 1H), 3.97 (d,  $J = 17.8$  Hz, 1H), 2.06 (s, 3H), 1.96 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  202.6, 201.2, 169.4, 140.4, 137.4, 133.9, 132.2, 129.0, 128.9, 128.3, 127.5, 125.7, 118.8, 67.4, 45.0, 23.6, 23.1.

HRMS (ESI)  $[\text{M}+\text{H}]^+$ : calculated for  $\text{C}_{19}\text{H}_{19}\text{NO}_3\text{Br}^+$ : 388.0543, found 388.0552.

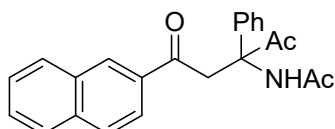


***N*-(1-([1,1'-Biphenyl]-4-yl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3o)**. Yellow oil (52.4 mg, 68% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 8.08 (d, *J* = 8.4 Hz, 2H), 7.70 (d, *J* = 8.4 Hz, 2H), 7.65 – 7.61 (m, 2H), 7.53 (brs, 1H), 7.50 – 7.36 (m, 7H), 7.34 – 7.29 (m, 1H), 5.21 (d, *J* = 17.3 Hz, 1H), 3.89 (d, *J* = 17.3 Hz, 1H), 2.05 (s, 3H), 1.93 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 203.0, 197.6, 169.5, 146.5, 139.6, 137.8, 135.3, 129.1, 129.0(1), 128.9(8), 128.4, 128.2, 127.4, 127.3, 125.8, 67.6, 41.0, 23.7, 23.1.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>25</sub>H<sub>23</sub>NO<sub>3</sub>Na<sup>+</sup>: 408.1570, found 408.1577.

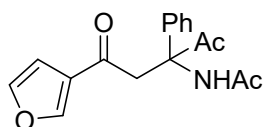


***N*-(1-(Naphthalen-2-yl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3p)**. White solid (38.8 mg, 54% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 8.60 (s, 1H), 8.03 – 7.97 (m, 2H), 7.88 (t, *J* = 8.1 Hz, 2H), 7.65 – 7.53 (m, 3H), 7.51 – 7.45 (m, 2H), 7.42 – 7.36 (m, 2H), 7.34 – 7.29 (m, 1H), 5.33 (d, *J* = 17.3 Hz, 1H), 3.99 (d, *J* = 17.3 Hz, 1H), 2.07 (s, 3H), 1.90 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 203.1, 197.9, 169.6, 137.8, 135.8, 133.9, 132.4, 130.7, 129.8, 129.0, 128.9, 128.6, 128.2, 127.7, 126.9, 125.8, 123.5, 67.6, 41.1, 23.6, 23.1.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>23</sub>H<sub>21</sub>NO<sub>3</sub>Na<sup>+</sup>: 382.1414, found 382.1422.

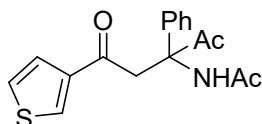


***N*-(1-(Furan-3-yl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3q)**. Yellow solid (32.3 mg, 54% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 8.17 (s, 1H), 7.50 (brs, 1H), 7.43 (t, *J* = 1.6 Hz, 1H), 7.41 – 7.34 (m, 4H), 7.34 – 7.28 (m, 1H), 6.75 – 6.72 (m, 1H), 4.79 (d, *J* = 16.3 Hz, 1H), 3.70 (d, *J* = 16.2 Hz, 1H), 2.05 (s, 3H), 1.90 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 202.9, 192.7, 169.5, 148.9, 144.5, 137.7, 129.1, 128.2, 127.9, 125.7, 108.3, 67.6, 42.5, 23.7, 23.2.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>17</sub>H<sub>17</sub>NO<sub>4</sub>Na<sup>+</sup>: 322.1050, found 322.1056.

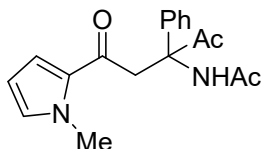


***N*-(1,4-Dioxo-3-phenyl-1-(thiophen-3-yl)pentan-3-yl)acetamide (3r).** Yellow solid (30.2 mg, 48% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 8.21 (dd, *J* = 2.8, 1.3 Hz, 1H), 7.53 (dd, *J* = 5.1, 1.2 Hz, 1H), 7.51 (brs, 1H), 7.42 – 7.34 (m, 4H), 7.33 – 7.28 (m, 2H), 5.01 (d, *J* = 16.7 Hz, 1H), 3.79 (d, *J* = 16.7 Hz, 1H), 2.04 (s, 3H), 1.89 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 203.0, 192.3, 169.5, 142.0, 137.7, 133.9, 129.0, 128.2, 126.8, 126.7, 125.7, 67.6, 42.0, 23.7, 23.2.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>17</sub>H<sub>17</sub>NO<sub>3</sub>NaS<sup>+</sup>: 338.0821, found 338.0823.

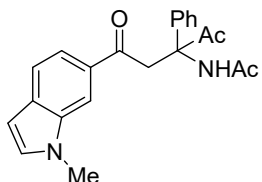


***N*-(1-(1-Methyl-1*H*-pyrrol-2-yl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3s).** Yellow solid (31.2 mg, 50% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.51 (brs, 1H), 7.41 – 7.34 (m, 4H), 7.32 – 7.27 (m, 1H), 7.15 (dd, *J* = 4.2, 1.7 Hz, 1H), 6.83 (t, *J* = 2.1 Hz, 1H), 6.13 (dd, *J* = 4.2, 2.4 Hz, 1H), 4.90 (d, *J* = 16.0 Hz, 1H), 3.88 (s, 3H), 3.68 (d, *J* = 16.0 Hz, 1H), 2.05 (s, 3H), 1.88 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 203.6, 187.6, 169.0, 138.2, 132.2, 130.7, 128.9, 128.0, 125.9, 121.3, 108.6, 67.6, 41.4, 37.7, 23.8, 23.5.

**HRMS (ESI) [M+H]<sup>+</sup>:** calculated for C<sub>18</sub>H<sub>21</sub>N<sub>2</sub>O<sub>3</sub><sup>+</sup>: 313.1547 found 313.1555.

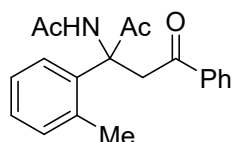


***N*-(1-(1-Methyl-1*H*-indol-6-yl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3t).** White solid (45.7 mg, 63% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 8.08 (s, 1H), 7.82 – 7.75 (m, 1H), 7.65 (d, *J* = 8.4 Hz, 1H), 7.61 (s, 1H), 7.51 – 7.44 (m, 2H), 7.38 (t, *J* = 7.8 Hz, 2H), 7.33 – 7.27 (m, 1H),

7.25 (d,  $J = 3.0$  Hz, 1H), 6.52 (dd,  $J = 3.0, 0.9$  Hz, 1H), 5.27 (d,  $J = 17.1$  Hz, 1H), 3.95 (d,  $J = 17.1$  Hz, 1H), 3.85 (s, 3H), 2.07 (s, 3H), 1.88 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  203.2, 197.9, 169.4, 138.0, 136.0, 133.1, 132.8, 130.2, 128.9, 128.0, 125.8, 120.7, 119.4, 110.8, 101.5, 67.8, 41.0, 33.0, 23.6, 23.2.

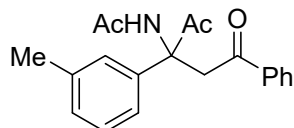


***N*-(1,4-Dioxo-1-phenyl-3-(*o*-tolyl)pentan-3-yl)acetamide (3u).** Yellow solid (35.6 mg, 55% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.03 – 7.97 (m, 2H), 7.65 (d,  $J = 7.9$  Hz, 1H), 7.59 (t,  $J = 7.4$  Hz, 1H), 7.47 (t,  $J = 7.8$  Hz, 2H), 7.35 – 7.29 (m, 2H), 7.24 (t,  $J = 7.1$  Hz, 1H), 7.11 (d,  $J = 7.4$  Hz, 1H), 5.29 (d,  $J = 14.9$  Hz, 1H), 3.65 (d,  $J = 14.9$  Hz, 1H), 2.17 (s, 3H), 2.06 (s, 3H), 1.76 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  202.5, 198.6, 168.5, 137.1, 136.2, 135.3, 133.8, 133.3, 128.8, 128.5, 128.4, 127.2, 126.7, 68.3, 41.8, 23.7, 23.2, 20.8.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{20}\text{H}_{21}\text{NO}_3\text{Na}^+$ : 346.1414, found 346.1418.

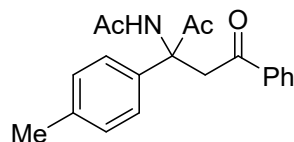


***N*-(1,4-Dioxo-1-phenyl-3-(*m*-tolyl)pentan-3-yl)acetamide (3v).** Yellow oil (38.8 mg, 60% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.05 – 8.00 (m, 2H), 7.64 – 7.58 (m, 1H), 7.55 (brs, 1H), 7.52 – 7.44 (m, 2H), 7.30 – 7.20 (m, 3H), 7.13 (d,  $J = 7.2$  Hz, 1H), 5.17 (d,  $J = 17.5$  Hz, 1H), 3.88 (d,  $J = 17.5$  Hz, 1H), 2.36 (s, 3H), 2.04 (s, 3H), 1.93 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  203.1, 198.1, 169.5, 138.7, 137.6, 136.6, 133.7, 129.0, 128.8, 128.7, 128.3, 126.3, 122.8, 67.4, 41.0, 23.6, 23.1, 21.6.

HRMS (ESI)  $[\text{M}+\text{Na}]^+$ : calculated for  $\text{C}_{20}\text{H}_{21}\text{NO}_3\text{Na}^+$ : 346.1414, found 346.1423.

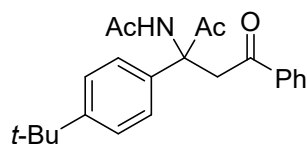


***N*-(1,4-Dioxo-1-phenyl-3-(*p*-tolyl)pentan-3-yl)acetamide (3w).** Yellow oil (37.8 mg, 58% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 8.00 (d, *J* = 7.7 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.50 (brs, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 7.17 (d, *J* = 8.1 Hz, 2H), 5.14 (d, *J* = 17.5 Hz, 1H), 3.85 (d, *J* = 17.5 Hz, 1H), 2.32 (s, 3H), 2.02 (s, 3H), 1.91 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 203.1, 198.1, 169.5, 138.0, 136.6, 134.7, 133.8, 129.8, 128.7, 128.4, 125.6, 67.3, 41.0, 23.7, 23.0, 21.0.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>20</sub>H<sub>21</sub>NO<sub>3</sub>Na<sup>+</sup>: 346.1414, found 346.1420.

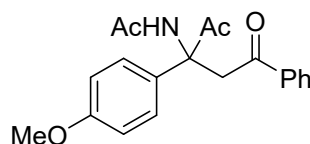


***N*-(3-(4-(*tert*-Butyl)phenyl)-1,4-dioxo-1-phenylpentan-3-yl)acetamide (3x).** Yellow solid (46.7 mg, 64% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 8.03 – 7.98 (m, 2H), 7.62 – 7.56 (m, 1H), 7.54 (brs, 1H), 7.46 (t, *J* = 7.8 Hz, 2H), 7.38 – 7.30 (m, 4H), 5.17 (d, *J* = 17.5 Hz, 1H), 3.87 (d, *J* = 17.5 Hz, 1H), 2.03 (s, 3H), 1.91 (s, 3H), 1.28 (s, 9H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 203.1, 198.2, 169.5, 151.0, 136.6, 134.4, 133.7, 128.7, 128.3, 126.0, 125.4, 67.3, 41.0, 34.4, 31.2, 23.7, 23.1.

**HRMS (ESI) [M+H]<sup>+</sup>:** calculated for C<sub>23</sub>H<sub>28</sub>NO<sub>3</sub><sup>+</sup>: 366.2064, found 366.2072.

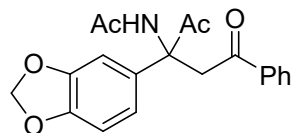


***N*-(3-(4-Methoxyphenyl)-1,4-dioxo-1-phenylpentan-3-yl)acetamide (3y).** Yellow oil (35.3 mg, 52% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 8.01 – 7.99 (m, 2H), 7.61 – 7.57 (m, 1H), 7.50 – 7.45 (m, 3H), 7.35 – 7.32 (m, 2H), 6.91 – 6.88 (m, 2H), 5.16 (d, *J* = 17.5 Hz, 1H), 3.82 (d, *J* = 17.4 Hz, 1H), 3.79 (s, 3H), 2.02 (s, 3H), 1.90 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ** 203.1, 198.1, 169.5, 159.3, 136.6, 133.8, 129.6, 128.7, 128.4, 127.0, 114.4, 66.9, 55.2, 41.0, 23.7, 23.0.

The analytical data were in good agreement with our previous report.<sup>6</sup>



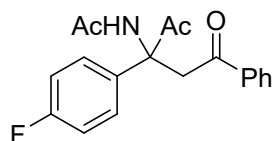
***N*-(3-(Benzo[d][1,3]dioxol-5-yl)-1,4-dioxo-1-phenylpentan-3-yl)acetamide (3z).**

Yellow oil (32.5 mg, 46% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ** 8.02 – 7.97 (m, 2H), 7.62 – 7.56 (m, 1H), 7.51–7.43 (m, 3H), 6.95–6.90 (m, 1H), 6.86 (s, 1H), 6.82–6.77 (m, 1H), 5.96 (s, 2H), 5.13 (d, *J* = 17.3 Hz, 1H), 3.78 (d, *J* = 17.4 Hz, 1H), 2.03 (s, 3H), 1.90 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ** 202.8, 197.9, 169.5, 148.4, 147.5, 136.5, 133.8, 131.6, 128.8, 128.4, 119.4, 108.5, 106.4, 101.4, 67.1, 41.1, 23.7, 22.9.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>20</sub>H<sub>19</sub>NO<sub>5</sub>Na<sup>+</sup>: 376.1155, found 376.1164.



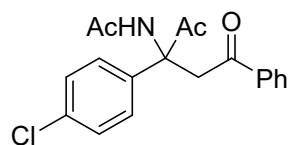
***N*-(3-(4-Fluorophenyl)-1,4-dioxo-1-phenylpentan-3-yl)acetamide (3aa).** Yellow oil (39.3 mg, 60% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ** 8.03 – 7.95 (m, 2H), 7.64 – 7.57 (m, 1H), 7.51 – 7.46 (m, 3H), 7.44 – 7.37 (m, 2H), 7.07 (t, *J* = 8.4 Hz, 2H), 5.17 (d, *J* = 17.2 Hz, 1H), 3.83 (d, *J* = 17.2 Hz, 1H), 2.02 (s, 3H), 1.90 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ** 202.8, 197.8, 169.4, 162.4 (d, *J* = 248.9 Hz), 136.5, 133.9, 133.6 (d, *J* = 2.9 Hz), 128.8, 128.4, 127.7 (d, *J* = 8.2 Hz), 116.0 (d, *J* = 21.8 Hz), 67.0, 41.0, 23.7, 23.1.

**<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) δ** -113.7.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>19</sub>H<sub>18</sub>NO<sub>3</sub>FNa<sup>+</sup>: 350.1163, found 350.1172.



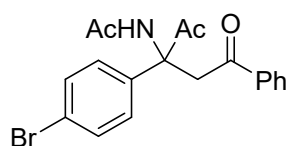
***N*-(3-(4-Chlorophenyl)-1,4-dioxo-1-phenylpentan-3-yl)acetamide (3ab).** Yellow

oil (42.5 mg, 62% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.99 – 7.98 (m, 2H), 7.61 – 7.58 (m, 1H), 7.51 (brs, 1H), 7.49 – 7.46 (m, 2H), 7.38 – 7.33 (m, 4H), 5.14 (d, *J* = 17.3 Hz, 1H), 3.83 (d, *J* = 17.4 Hz, 1H), 2.02 (s, 3H), 1.90 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 202.5, 197.8, 169.5, 136.4(0), 136.3(7), 134.3, 133.9, 129.2, 128.8, 128.4, 127.2, 67.1, 41.0, 23.6, 23.1.

The analytical data were in good agreement with our previous report.<sup>6</sup>

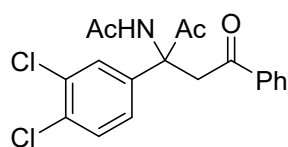


***N*-(1-(4-Bromophenyl)-1,4-dioxo-3-phenylpentan-3-yl)acetamide (3ac).** Yellow oil (43.3 mg, 56% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.99 (dd, *J* = 8.3, 1.1 Hz, 2H), 7.63 – 7.57 (m, 1H), 7.53 – 7.44 (m, 5H), 7.33 – 7.28 (m, 2H), 5.13 (d, *J* = 17.4 Hz, 1H), 3.83 (d, *J* = 17.4 Hz, 1H), 2.02 (s, 3H), 1.90 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 202.5, 197.7, 169.5, 137.0, 136.4, 134.0, 132.2, 128.8, 128.4, 127.6, 122.5, 67.2, 40.9, 23.6, 23.1.

**HRMS (ESI) [M+H]<sup>+</sup>:** calculated for C<sub>19</sub>H<sub>19</sub>NO<sub>3</sub>Br: 388.0543, found 388.0543.



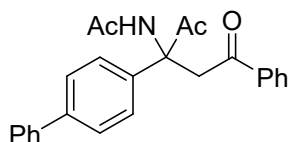
***N*-(3-(3,4-Dichlorophenyl)-1,4-dioxo-1-phenylpentan-3-yl)acetamide (3ad).**

Yellow solid (31.7 mg, 42% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.97 – 7.93 (m, 2H), 7.68 (d, *J* = 8.4 Hz, 1H), 7.63 – 7.56 (m, 1H), 7.49 – 7.44 (m, 2H), 7.40 – 7.35 (m, 2H), 7.34 (brs, 1H), 5.18 (d, *J* = 13.9 Hz, 1H), 3.46 (d, *J* = 13.9 Hz, 1H), 2.12 (s, 3H), 1.74 (s, 3H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 201.0, 197.9, 169.2, 137.2, 135.0, 134.1, 133.9, 133.1, 131.7, 130.3, 128.8, 128.5, 127.5, 67.5, 41.3, 24.3, 23.2.

**HRMS (ESI) [M+H]<sup>+</sup>:** calculated for C<sub>19</sub>H<sub>18</sub>NO<sub>3</sub>Cl<sub>2</sub><sup>+</sup>: 378.0658, found 378.0665.



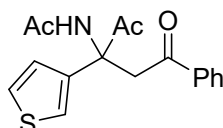
***N*-(3-((1,1'-Biphenyl)-4-yl)-1,4-dioxo-1-phenylpentan-3-yl)acetamide (3ae).**

Yellow oil (51.6 mg, 67% yield).

$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.03 – 8.01 (m, 2H), 7.62 – 7.56 (m, 6H), 7.51 – 7.47 (m, 4H), 7.45 – 7.42 (m, 2H), 7.36 – 7.33 (m, 1H), 5.22 (d,  $J = 17.4$  Hz, 1H), 3.92 (d,  $J = 17.5$  Hz, 1H), 2.07 (s, 3H), 1.94 (s, 3H).

$^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  202.9, 198.1, 169.6, 141.0, 140.1, 136.6, 136.5, 133.8, 130.1, 128.8, 128.4, 127.7, 127.6, 127.1, 126.2, 67.4, 41.1, 23.7, 23.2.

The analytical data were in good agreement with our previous report.<sup>6</sup>

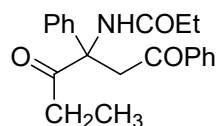


***N*-(1,4-Dioxo-1-phenyl-3-(thiophen-3-yl)pentan-3-yl)acetamide (3af).** Yellow oil (35.1 mg, 56% yield).

$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.02 – 7.96 (m, 2H), 7.63 – 7.56 (m, 1H), 7.49 – 7.46 (m, 3H), 7.38 – 7.30 (m, 2H), 6.98 (dd,  $J = 4.9, 1.5$  Hz, 1H), 5.17 (d,  $J = 17.8$  Hz, 1H), 3.84 (d,  $J = 17.8$  Hz, 1H), 2.07 (s, 3H), 1.93 (s, 3H).

$^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  202.5, 197.7, 169.6, 139.1, 136.4, 133.8, 128.8, 128.3, 126.9, 125.5, 122.3, 65.7, 41.9, 23.7, 22.9.

**HRMS (ESI)  $[\text{M}+\text{Na}]^+$ :** calculated for  $\text{C}_{17}\text{H}_{17}\text{NO}_3\text{NaS}^+$ : 338.0821, found 338.0831.

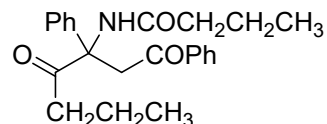


***N*-(1,4-Dioxo-1,3-diphenylhexan-3-yl)propionamide (5a).** Yellow oil (43.8 mg, 65% yield).

$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.02 – 7.95 (m, 2H), 7.60 – 7.56 (m, 2H), 7.46 (t,  $J = 7.7$  Hz, 2H), 7.42 – 7.32 (m, 4H), 7.30 – 7.26 (m, 1H), 5.19 (d,  $J = 17.2$  Hz, 1H), 3.87 (d,  $J = 17.2$  Hz, 1H), 2.44 – 2.36 (m, 1H), 2.30 – 2.22 (m, 1H), 2.17 – 2.10 (m, 2H), 0.98 (t,  $J = 7.6$  Hz, 3H), 0.94 (t,  $J = 7.2$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  206.4, 198.2, 172.9, 138.3, 136.7, 133.7, 129.0, 128.7, 128.4, 128.0, 125.7, 67.1, 41.0, 29.9, 28.4, 9.5, 8.4.

HRMS (ESI)  $[\text{M}+\text{H}]^+$ : calculated for  $\text{C}_{21}\text{H}_{24}\text{NO}_3$ : 338.1751, found 338.1758.

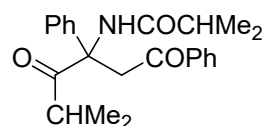


***N*-(1,4-Dioxo-1,3-diphenyloctan-3-yl)pentanamide (5b)**. Yellow oil (39.2 mg, 54% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.02 – 7.96 (m, 2H), 7.61 – 7.56 (m, 1H), 7.54 (brs, 1H), 7.46 (t,  $J = 7.7$  Hz, 2H), 7.43 – 7.38 (m, 2H), 7.38 – 7.33 (m, 2H), 7.32 – 7.27 (m, 1H), 5.19 (d,  $J = 17.3$  Hz, 1H), 3.88 (d,  $J = 17.3$  Hz, 1H), 2.39 – 2.33 (m, 1H), 2.26 – 2.20 (m, 1H), 2.12 – 2.06 (m, 2H), 1.54 – 1.45 (m, 4H), 0.77 (t,  $J = 7.4$  Hz, 3H), 0.65 (t,  $J = 7.4$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  205.3, 198.2, 172.2, 138.1, 136.7, 133.7, 128.9, 128.8, 128.4, 128.1, 125.8, 67.2, 41.0, 38.8, 36.9, 18.9, 17.5, 13.6, 13.3.

HRMS (ESI)  $[\text{M}+\text{H}]^+$ : calculated for  $\text{C}_{23}\text{H}_{28}\text{NO}_3^+$ : 366.2064, found 366.2071.

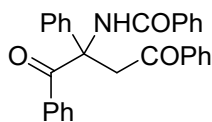


***N*-(5-Methyl-1,4-dioxo-1,3-diphenylhexan-3-yl)isobutyramide (5c)**. Yellow oil (43.7 mg, 60% yield).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.99 (d,  $J = 7.4$  Hz, 2H), 7.62 (brs, 1H), 7.57 (t,  $J = 7.4$  Hz, 1H), 7.49 – 7.39 (m, 4H), 7.36 (t,  $J = 7.6$  Hz, 2H), 7.30 – 7.26 (m, 1H), 5.17 (d,  $J = 17.0$  Hz, 1H), 3.89 (d,  $J = 17.1$  Hz, 1H), 2.85 (hept,  $J = 6.7$  Hz, 1H), 2.32 (hept,  $J = 6.9$  Hz, 1H), 0.99 (d,  $J = 6.9$  Hz, 3H), 0.95 (d,  $J = 6.7$  Hz, 3H), 0.93 (d,  $J = 6.9$  Hz, 3H), 0.58 (d,  $J = 6.6$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  210.7, 198.1, 175.5, 137.8, 136.8, 133.6, 128.9, 128.7, 128.4, 128.1, 125.9, 67.0, 40.7, 35.6, 34.2, 21.6, 20.8, 19.2, 19.1.

HRMS (ESI)  $[\text{M}+\text{H}]^+$ : calculated for  $\text{C}_{23}\text{H}_{28}\text{NO}_3^+$ : 366.2064, found 366.2072.

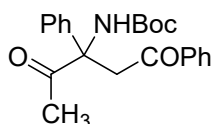


***N*-(1,4-Dioxo-1,2,4-triphenylbutan-2-yl)benzamide (5d).** White solid (62.4 mg, 72% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 8.70 (brs, 1H), 7.93-7.91 (m, 2H), 7.77 – 7.75 (m, 2H), 7.71 – 7.69 (m, 2H), 7.54– 7.49 (m, 3H), 7.47 – 7.43 (m, 3H), 7.40 – 7.33 (m, 6H), 7.25 – 7.22 (m, 2H), 5.52 (d, *J* = 17.7 Hz, 1H), 4.16 (d, *J* = 17.7 Hz, 1H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 198.0, 197.1, 165.8, 137.8, 136.5, 135.6, 134.5, 133.5, 131.8, 131.6, 129.1, 129.0, 128.6, 128.5, 128.4, 128.2, 128.1, 127.0, 126.7, 66.3, 42.8.

**HRMS (ESI) [M+H]<sup>+</sup>:** calculated for C<sub>29</sub>H<sub>24</sub>NO<sub>3</sub><sup>+</sup>: 434.1751, found 434.1758.

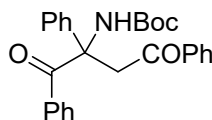


***tert*-Butyl (1,4-dioxo-1,3-diphenylpentan-3-yl)carbamate (5e).** Yellow oil (26.4 mg, 36% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 8.01 (d, *J* = 7.3 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.47–7.42 (m, 4H), 7.37 (t, *J* = 7.7 Hz, 2H), 7.32–7.29 (m, 1H), 6.57 (brs, 1H), 4.98 (d, *J* = 16.9 Hz, 1H), 3.77 (d, *J* = 16.8 Hz, 1H), 2.07 (s, 3H), 1.27 (s, 9H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 203.2, 198.4, 154.5, 138.4, 137.0, 133.6, 129.0, 128.7, 128.4, 128.0, 125.7, 79.8, 67.7, 41.2, 28.1, 23.3.

**HRMS (ESI) [M+Na]<sup>+</sup>:** calculated for C<sub>22</sub>H<sub>25</sub>NO<sub>4</sub>Na<sup>+</sup>: 390.1676, found 390.1685.



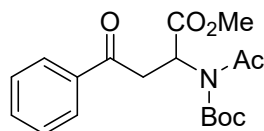
***tert*-Butyl (1,4-dioxo-1,2,4-triphenylbutan-2-yl)carbamate (5f).** White solid (53.2 mg, 62% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.94 (d, *J* = 7.7 Hz, 2H), 7.62 – 7.50 (m, 5H), 7.46 – 7.37 (m, 4H), 7.37-7.28 (m, 2H), 7.23 (t, *J* = 7.7 Hz, 2H), 6.83 (brs, 1H), 5.05 (d, *J* = 17.1 Hz, 1H), 3.94 (d, *J* = 17.1 Hz, 1H), 1.20 (s, 9H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 197.8, 197.7, 154.1, 138.2, 137.0, 135.9, 133.3,

131.5, 129.1, 128.9, 128.5, 128.2, 128.1, 127.9, 126.4, 79.7, 66.4, 43.1, 28.0.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>27</sub>H<sub>27</sub>NO<sub>4</sub>Na<sup>+</sup>: 452.1832, found 452.1838.



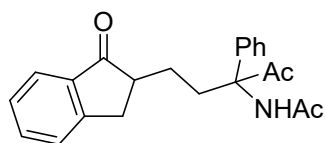
**Methyl 2-(N-(tert-butoxycarbonyl)acetamido)-4-oxo-4-phenylbutanoate (7)**

Yellow oil (51.7 mg, 74% yield).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** δ 7.95 (d, *J* = 7.7 Hz, 2H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 2H), 5.94 (t, *J* = 6.3 Hz, 1H), 4.05 (dd, *J* = 17.4, 6.7 Hz, 1H), 3.70 (s, 3H), 3.19 (dd, *J* = 17.4, 5.8 Hz, 1H), 2.48 (s, 3H), 1.53 (s, 9H).

**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** δ 196.6, 172.7, 170.5, 152.1, 136.5, 133.3, 128.6, 128.1, 84.4, 52.7, 52.5, 39.3, 27.9, 26.5.

**HRMS (ESI) [M+Na]<sup>+</sup>**: calculated for C<sub>18</sub>H<sub>23</sub>NO<sub>6</sub>Na<sup>+</sup>: 372.1418, found 372.1427.



**N-(4-Oxo-1-(3-oxo-2,3-dihydro-1H-inden-1-yl)-3-phenylpentan-3-yl)acetamide**

**(11)**. White solid (46.8 mg, 67% yield, dr = 1.1:1).

**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)** (mixture of diastereoisomers) δ 7.76 (d, *J* = 7.7 Hz, 0.48H), 7.73 (d, *J* = 7.7 Hz, 0.52H), 7.64 – 7.56 (m, 1H), 7.49 (d, *J* = 7.7 Hz, 0.48H), 7.45 (d, *J* = 7.7 Hz, 0.52H), 7.42 – 7.34 (m, 3H), 7.34 – 7.27 (m, 4H), 3.47 – 3.31 (m, 1H), 3.29 – 3.18 (m, 0.5H), 3.10 – 3.00 (m, 0.5H), 2.95 – 2.86 (m, 0.5H), 2.84 – 2.72 (m, 1.5H), 2.71 – 2.63 (m, 0.5H), 2.32 – 2.17 (m, 0.5H), 2.02 (s, 1.44H), 2.00 (s, 1.56H), 1.98 (s, 1.56H), 1.97 (s, 1.44H), 1.91 – 1.82 (m, 0.5H), 1.60 – 1.51 (m, 0.5H), 1.45 – 1.36 (m, 1H).

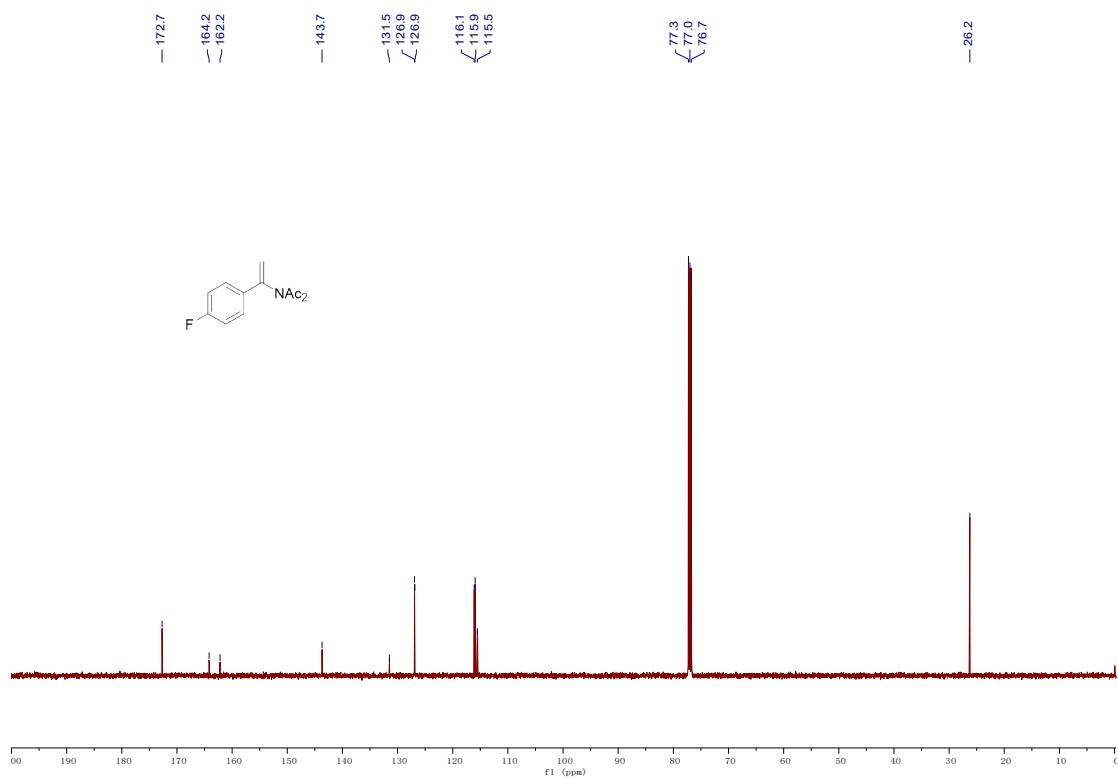
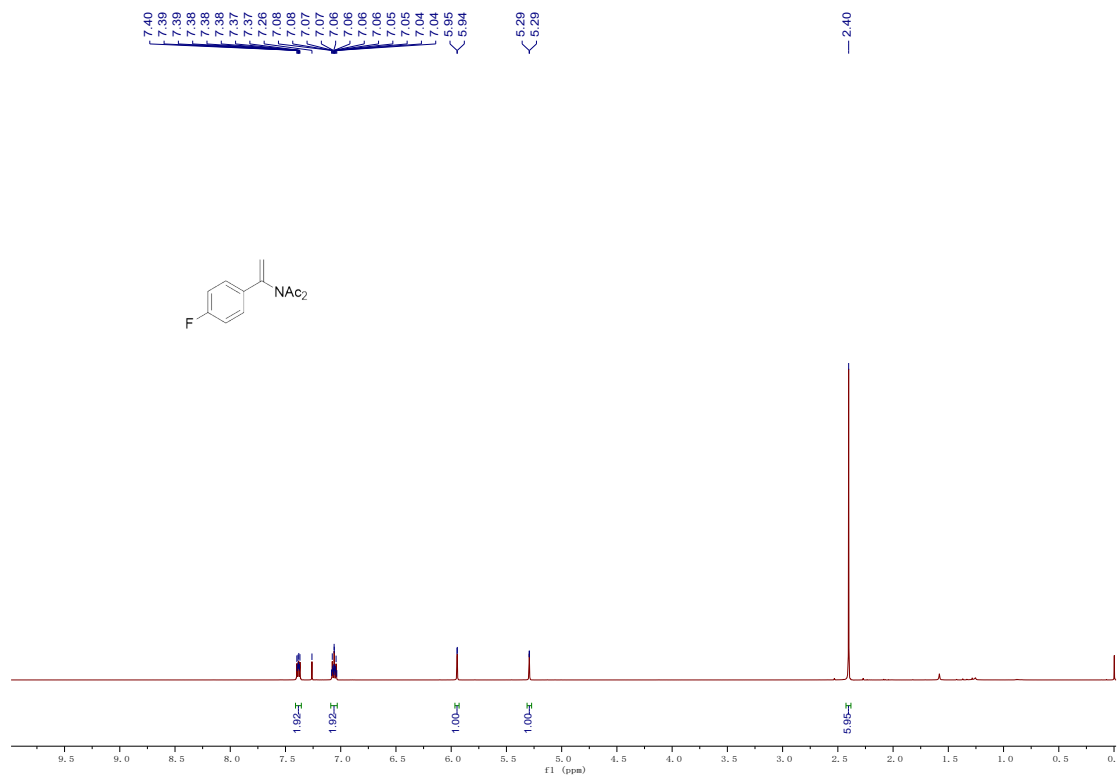
**<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)** (mixture of diastereoisomers) δ 208.5, 208.3, 205.3(2), 205.2(7), 168.5(2), 168.4(9), 153.7, 153.5, 139.2, 139.1, 136.9, 136.5, 134.9(9), 134.9(6), 129.0, 128.9, 128.1, 128.0, 127.6, 127.5, 126.8, 126.7, 126.1(3), 126.0(7), 124.0, 123.9, 69.8, 69.7, 47.0, 46.5, 33.5, 31.8, 29.4, 27.0, 26.2, 24.3, 24.0, 23.9, 23.5, 23.4.

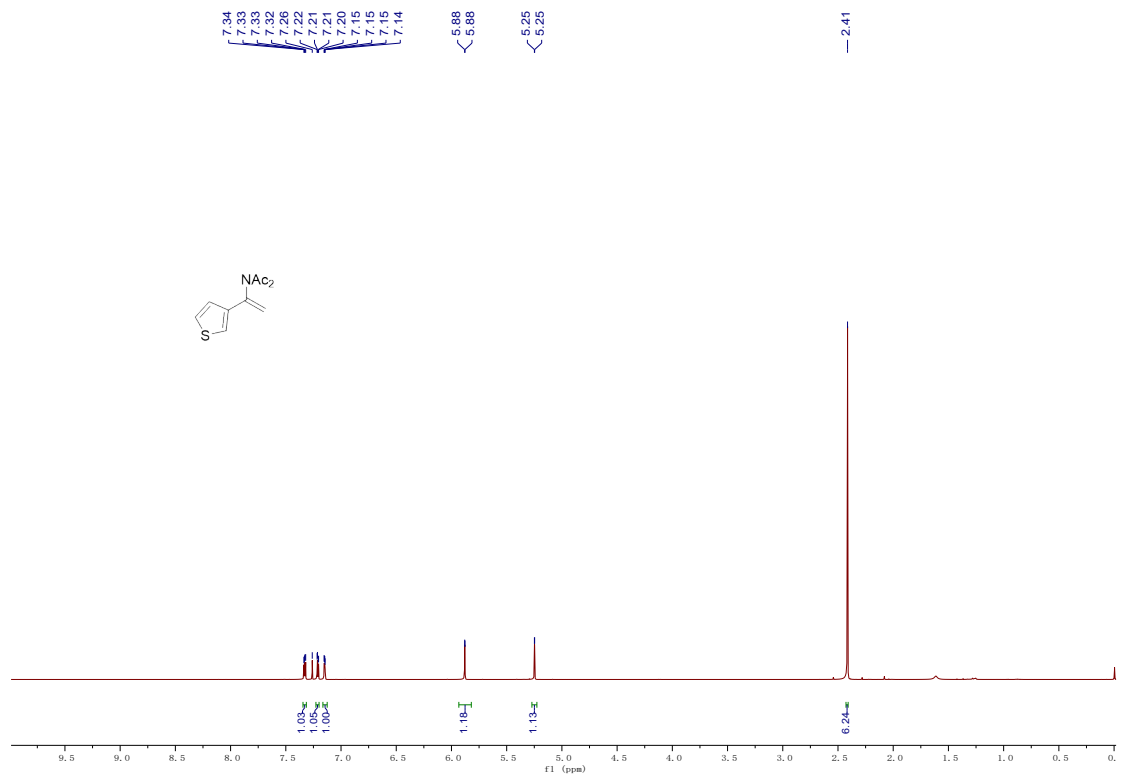
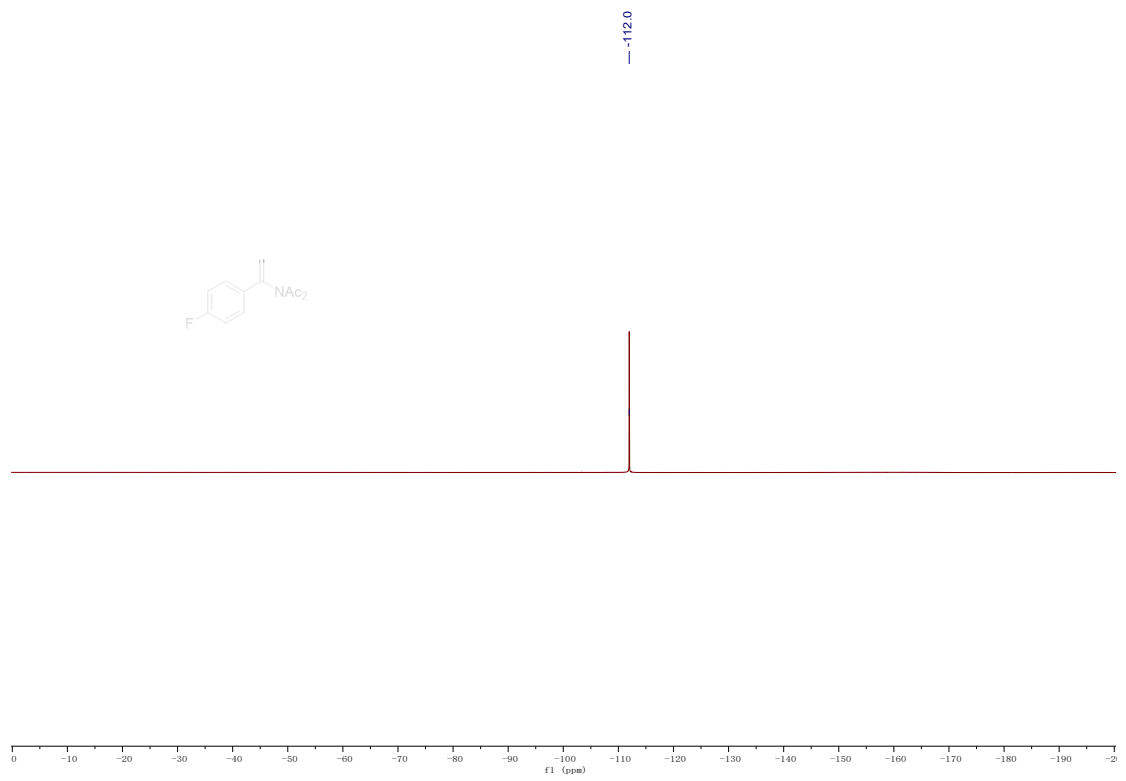
HRMS (ESI)  $[M+H]^+$ : calculated for  $C_{22}H_{24}NO_3^+$ : 350.1751, found 350.1758.

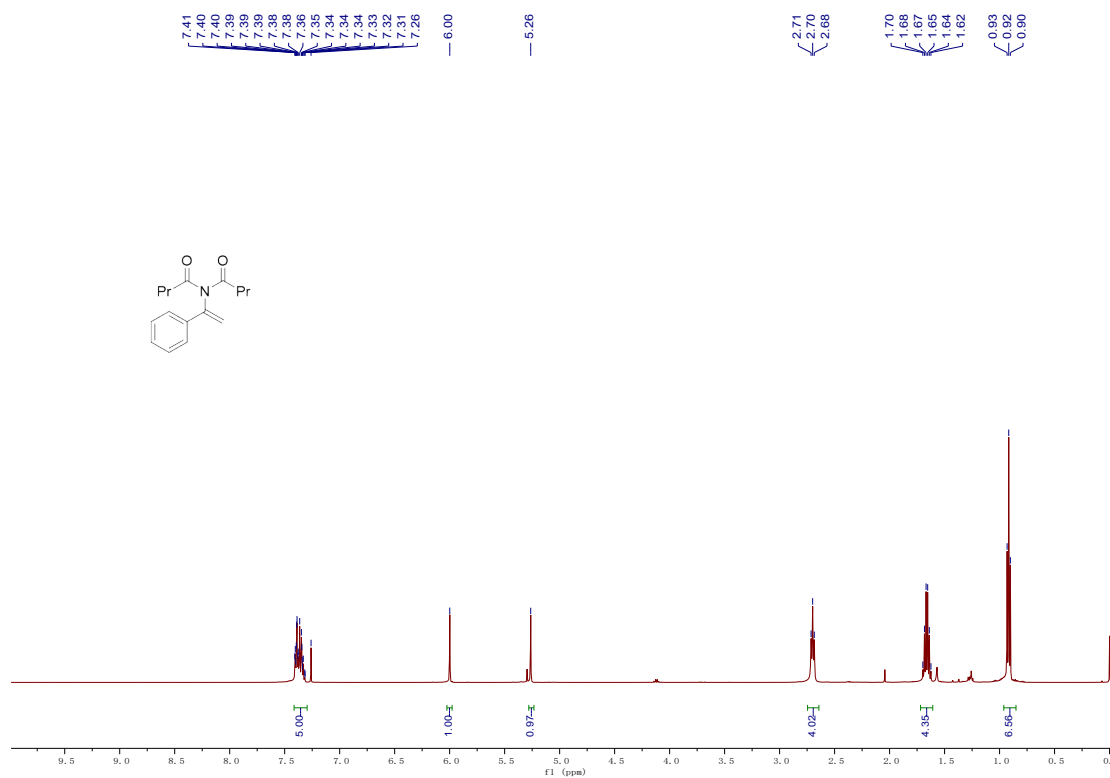
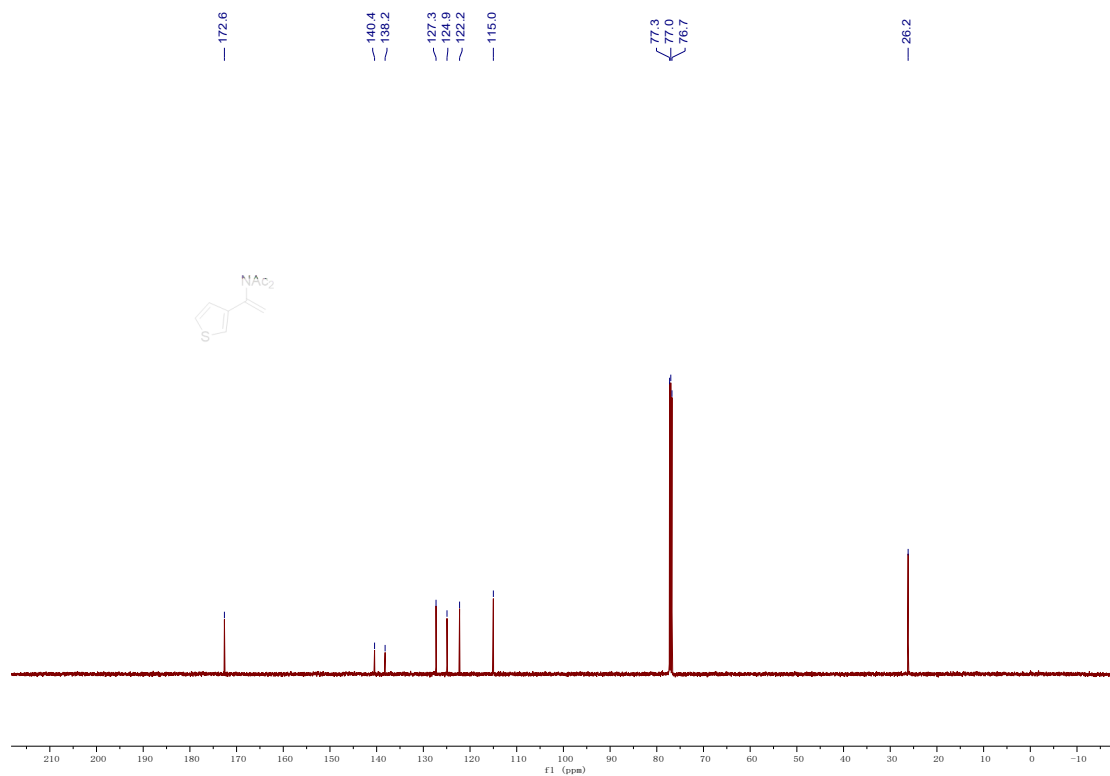
## 4. References

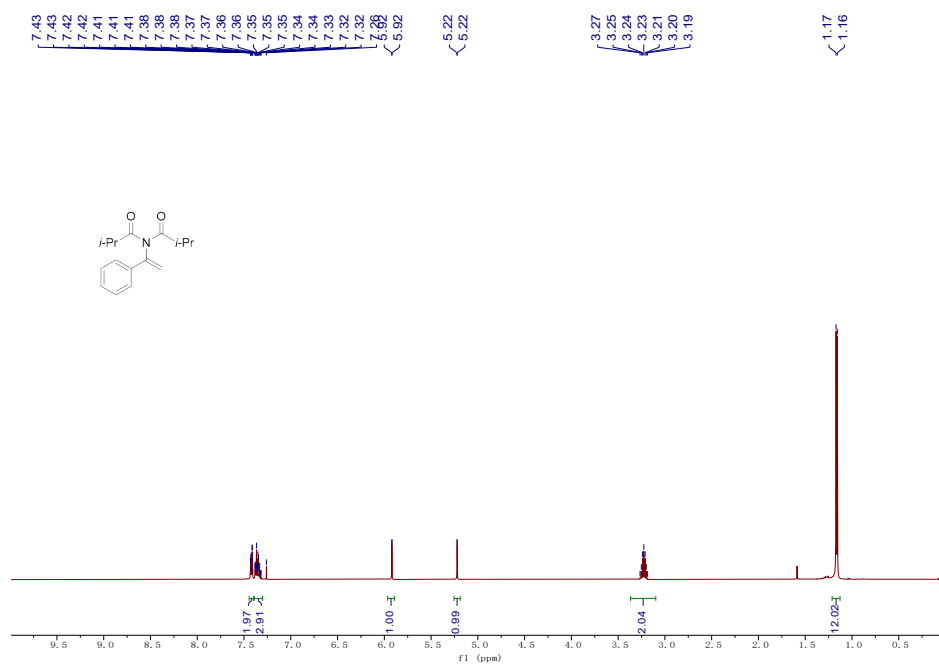
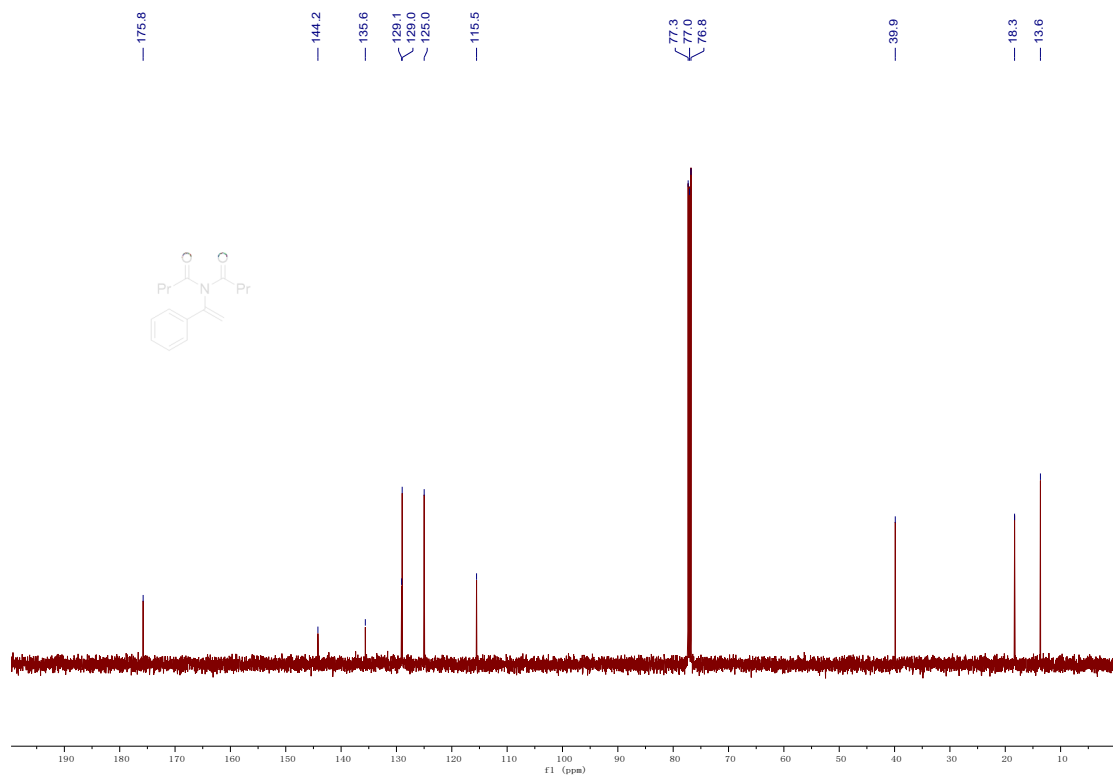
- 1 L. Huai, L. Zhang, Z. Wang and Y. Fang, *Org. Chem. Front.*, 2023, **10**, 1245.
- 2 L. Huai, L. Zhang, Z. Wang and Y. Fang, *Org. Chem. Front.*, 2024, **11**, 2344.
- 3 I. Furusawa, T. Suzuki, Y. Yu and T. Arai, *Adv. Synth. Catal.*, 2023, **365**, 3247.
- 4 Y. Zhao, Y. Jin, Y. Li, L. Qiu and Y. Fang, *Org. Chem. Front.*, 2025, **12**, 3834.
- 5 H. Huang, Y. Zhao, L. Zhang, Y. Li and Y. Fang, *Org. Biomol. Chem.*, 2025, **23**, 4079.
- 6 Y. Jing, L. Zhang, L. Qiu and Y. Fang, *RSC Adv.*, 2025, **15**, 11582.
- 7 J. T. Reeves, Z. Tan, Z. S. Han, G. Li, Y. Zhang, Y. Xu, D. C. Reeves, N. C. Gonnella, S. Ma, H. Lee, B. Z. Lu and C. H. Senanayake, *Angew. Chem. Int. Ed.*, 2012, **51**, 1400.

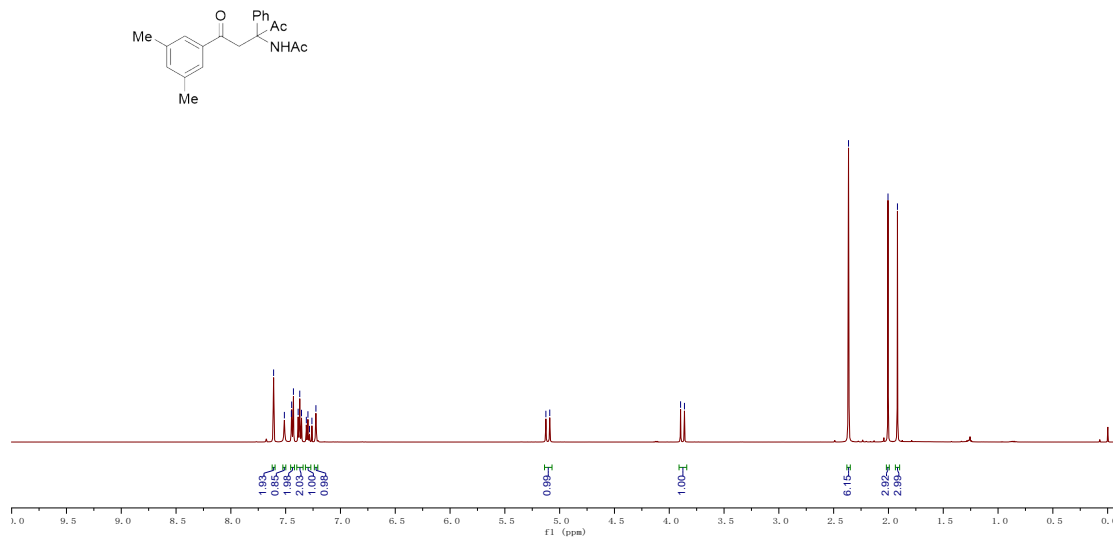
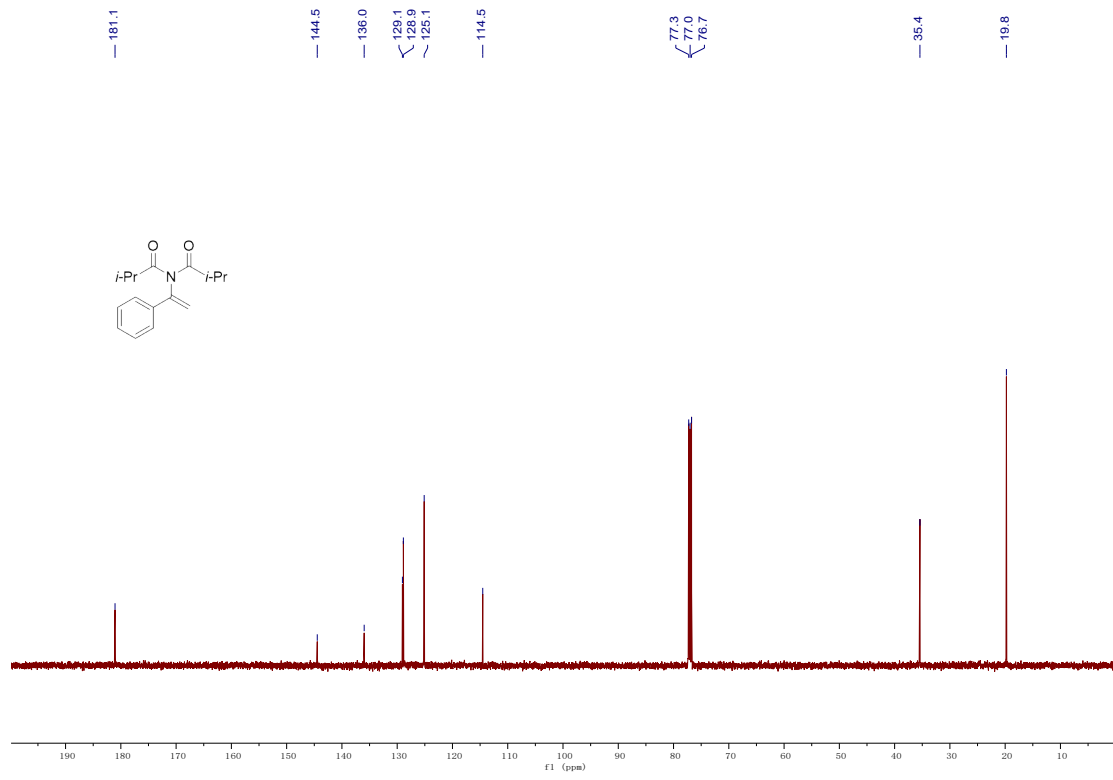
## 5. NMR Spectra of new compounds

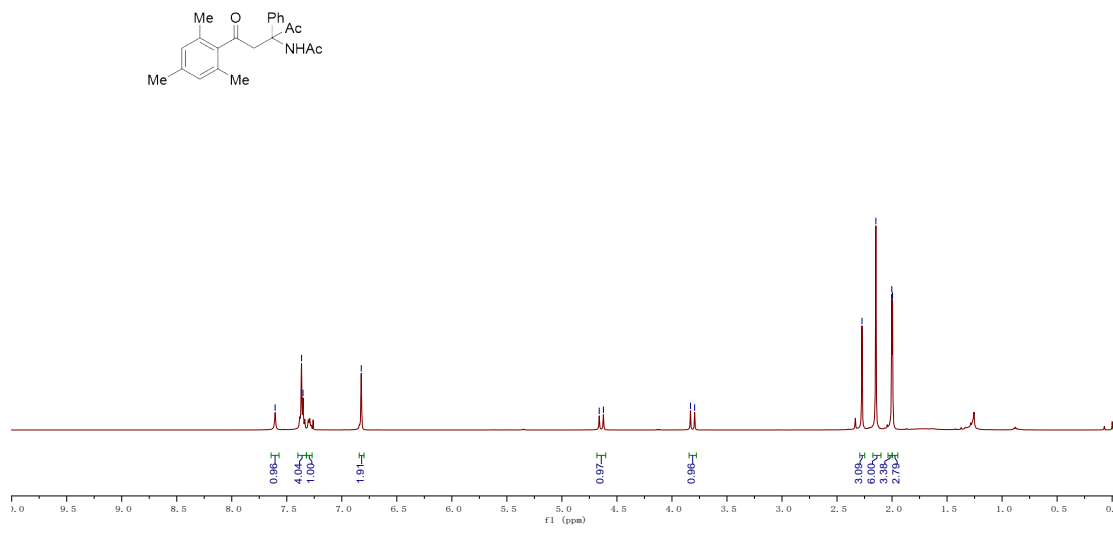
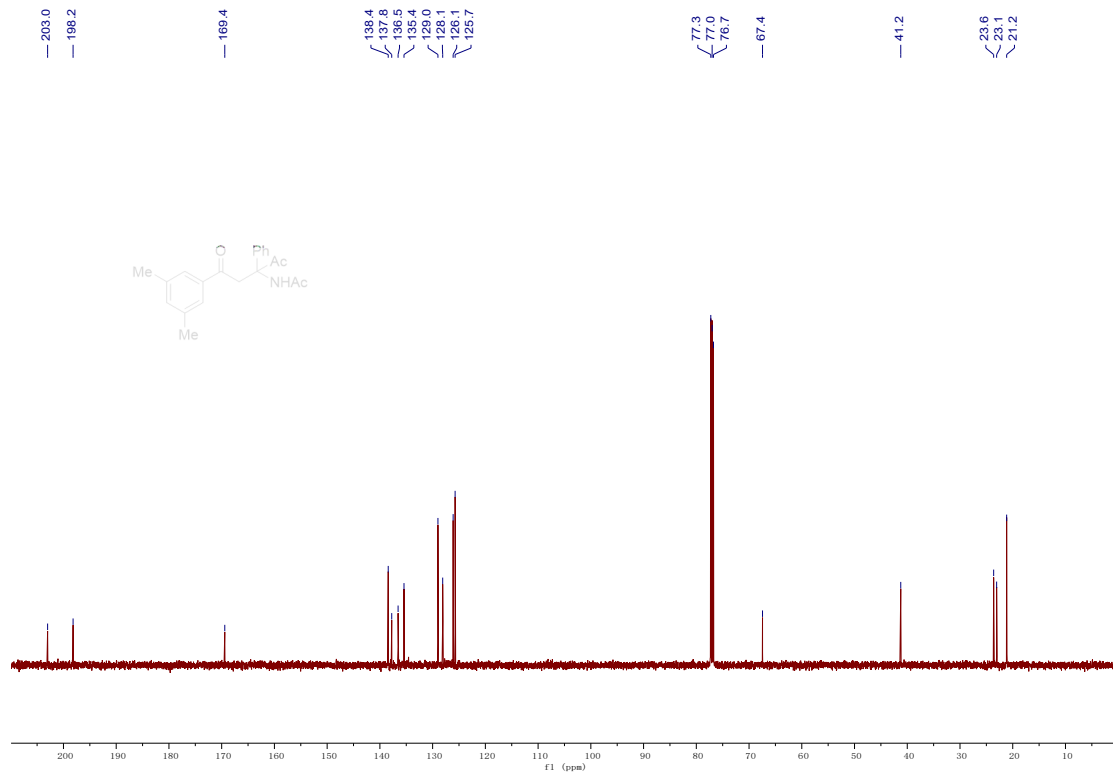


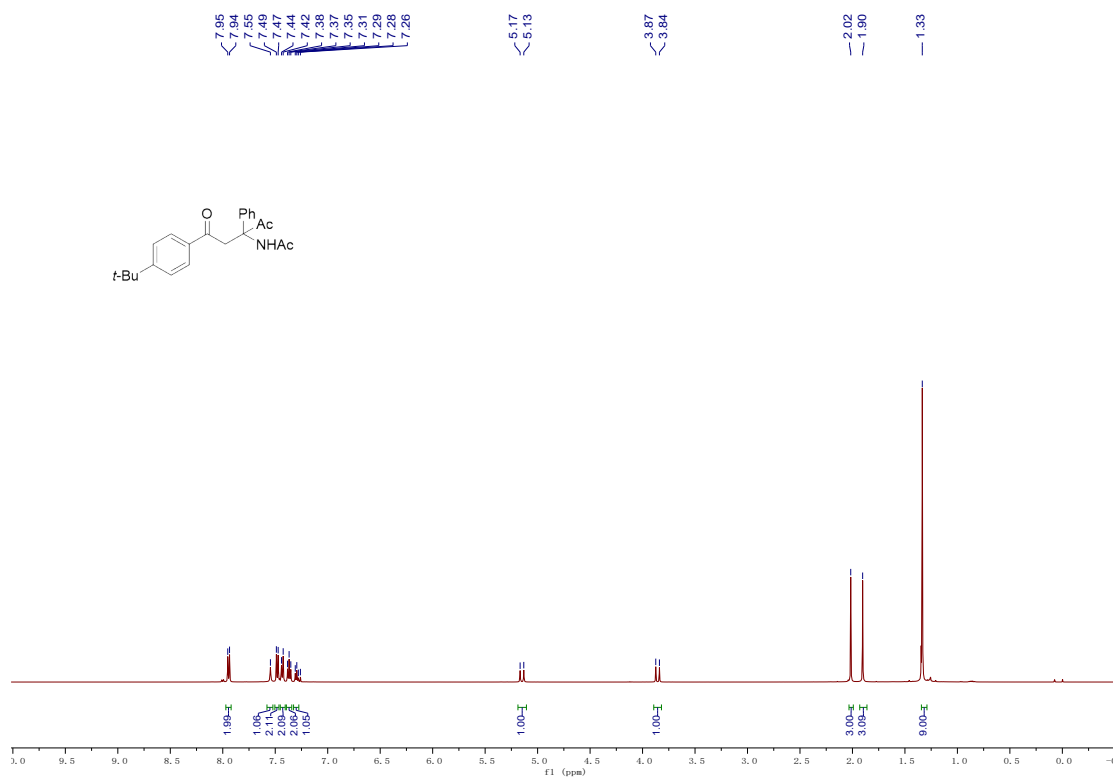
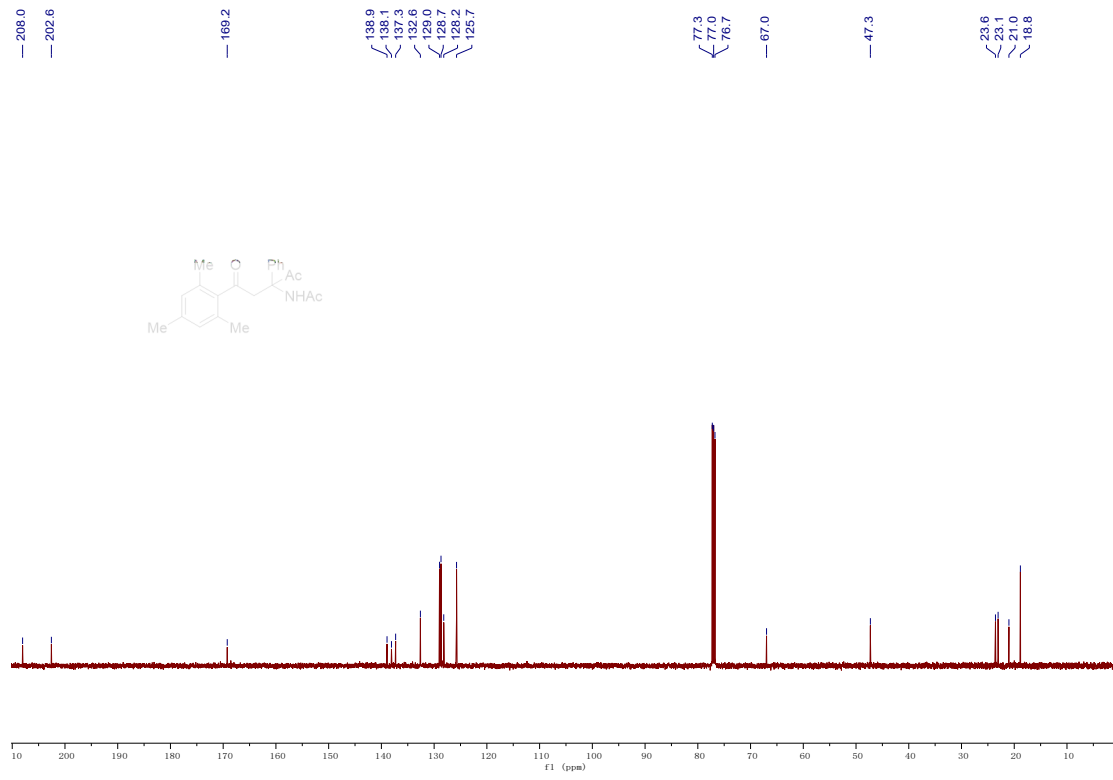


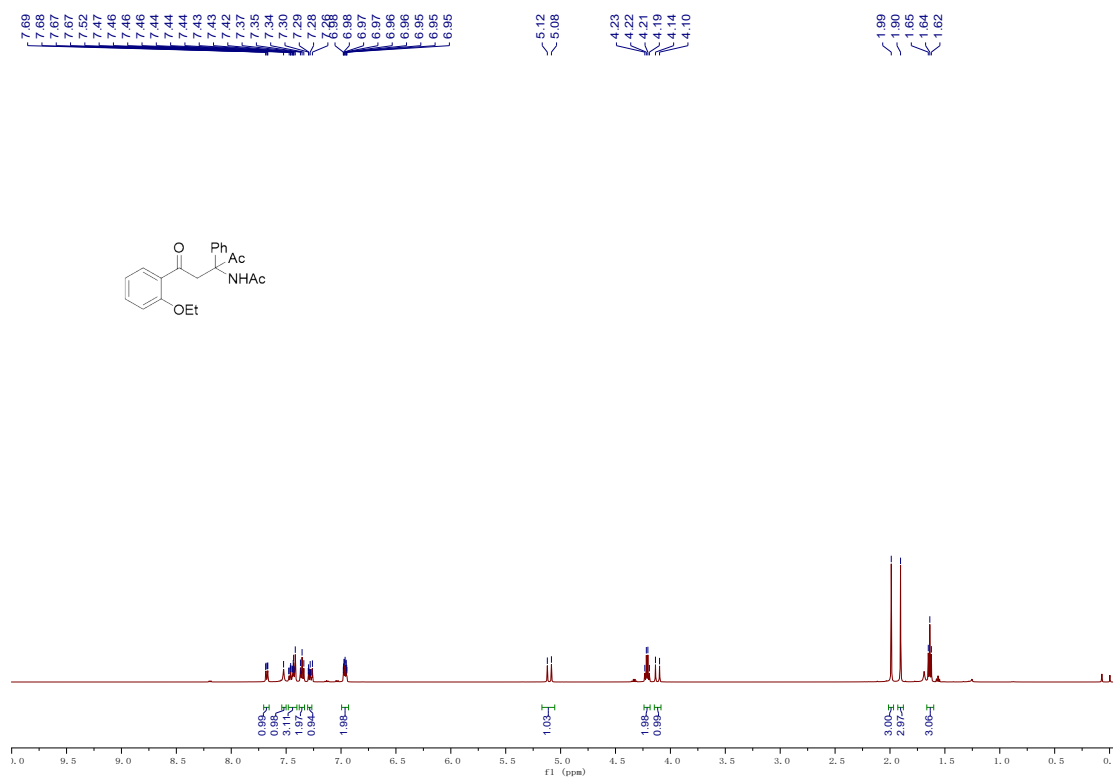
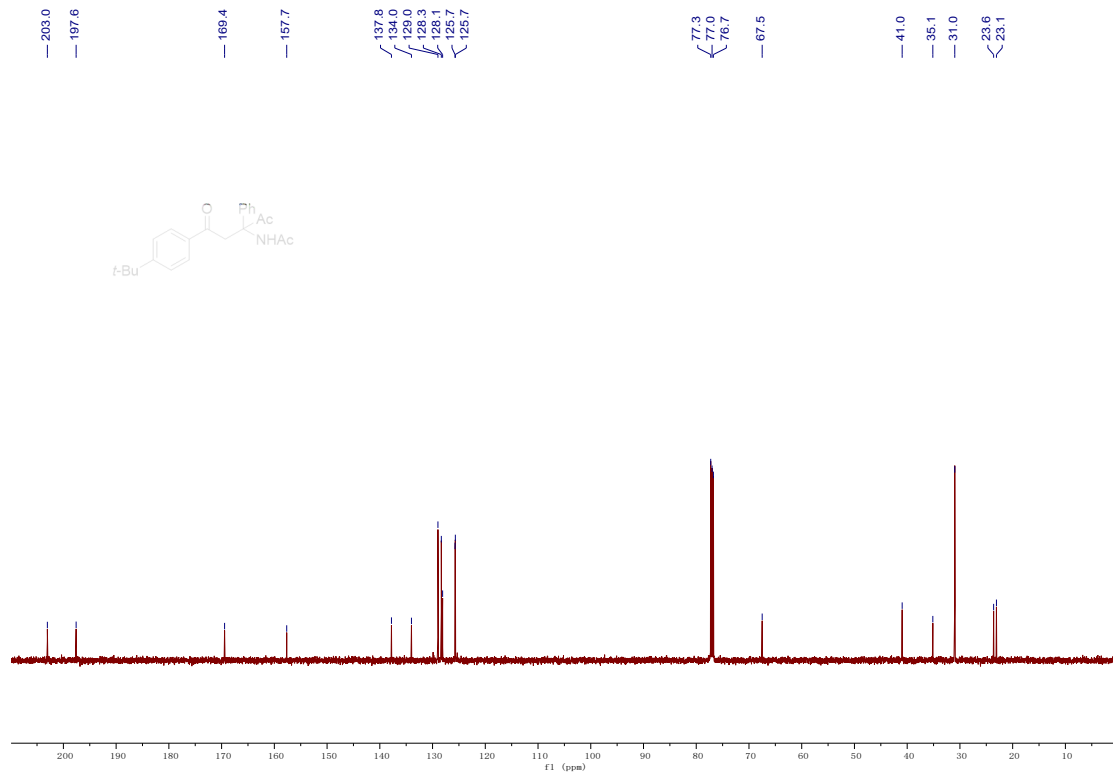


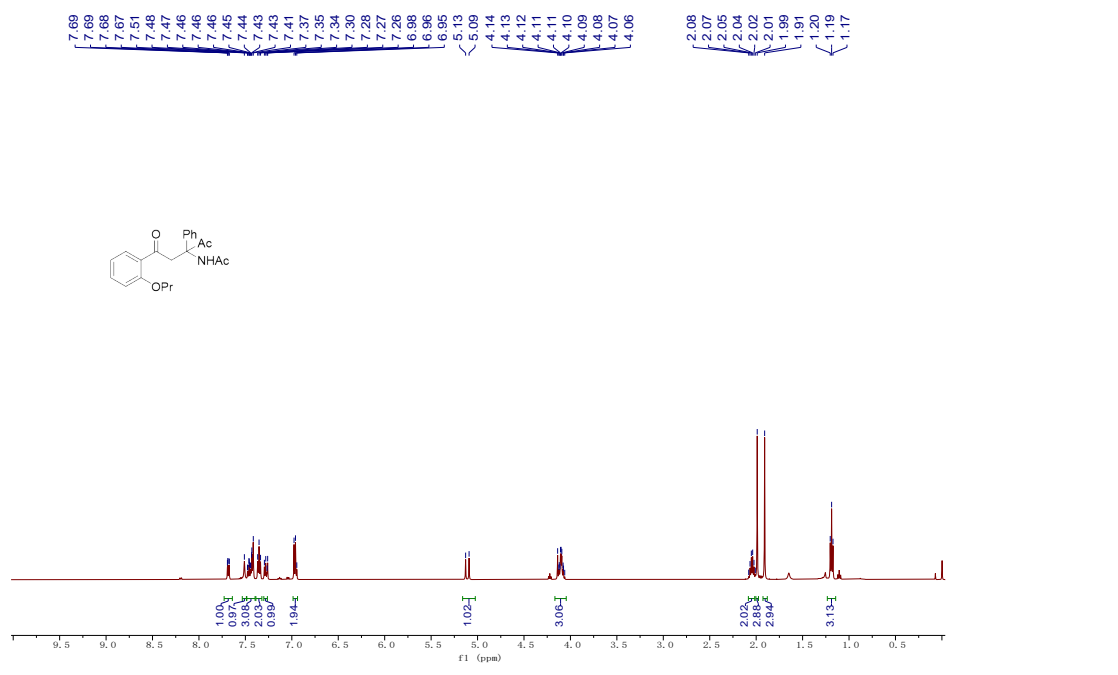
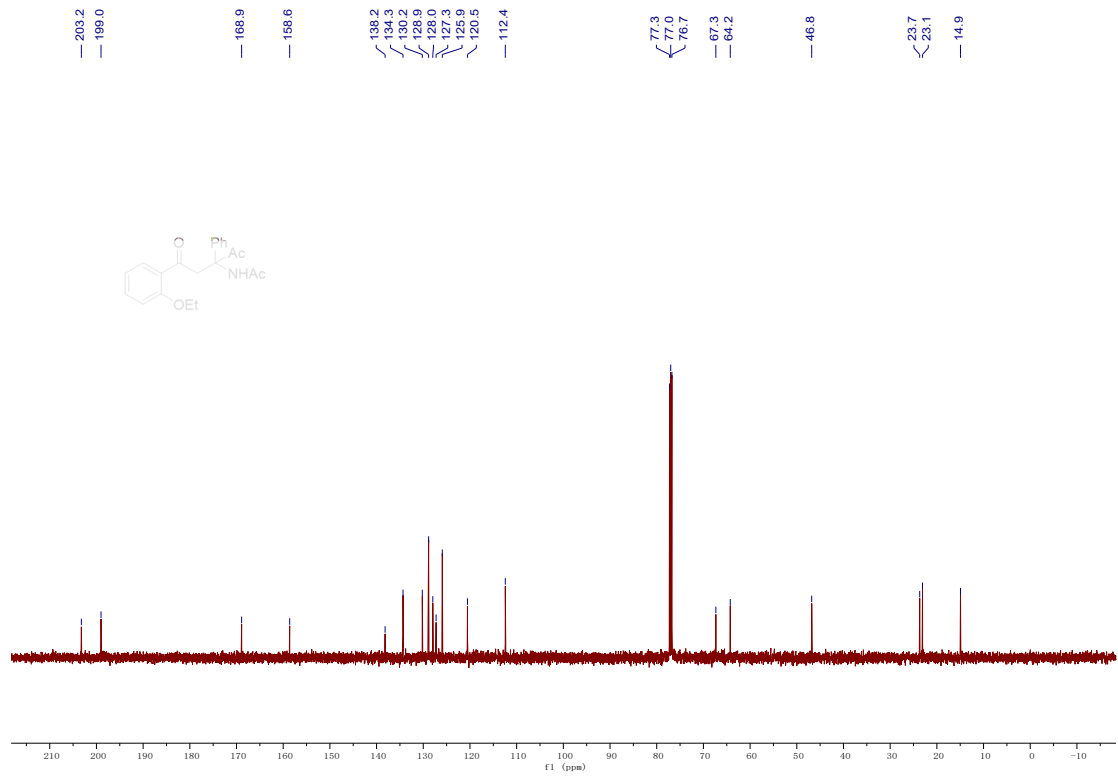




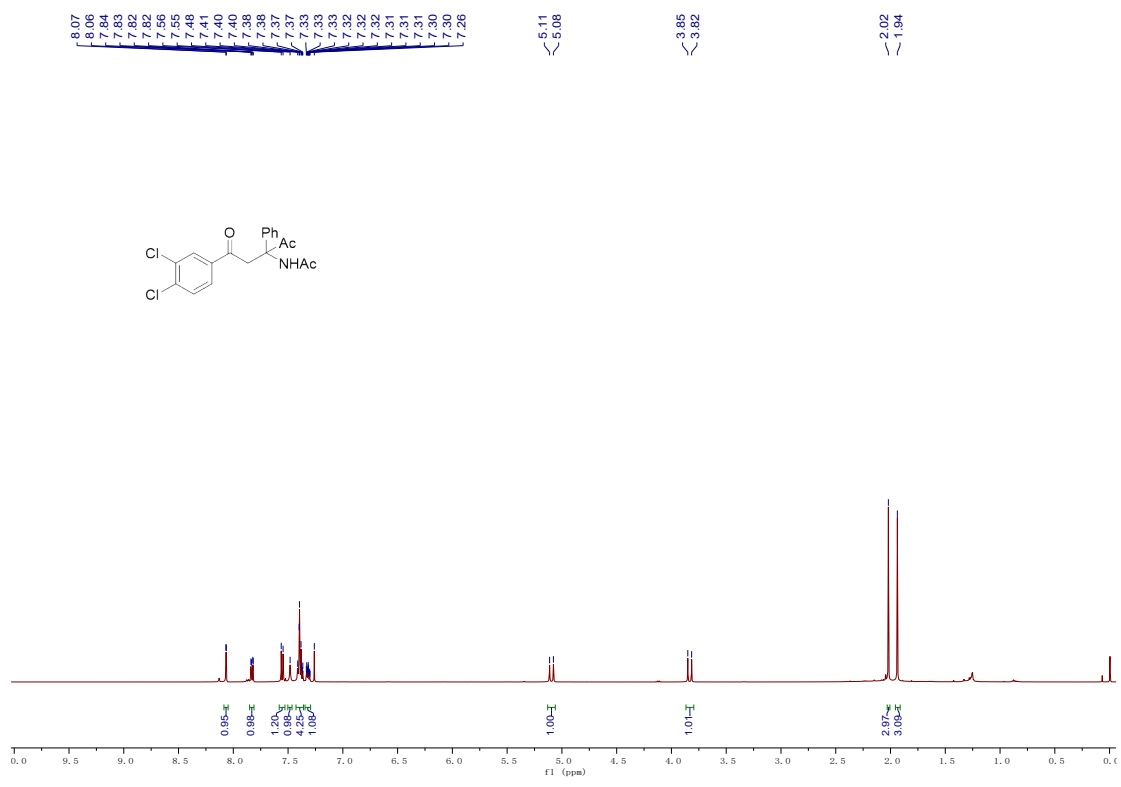
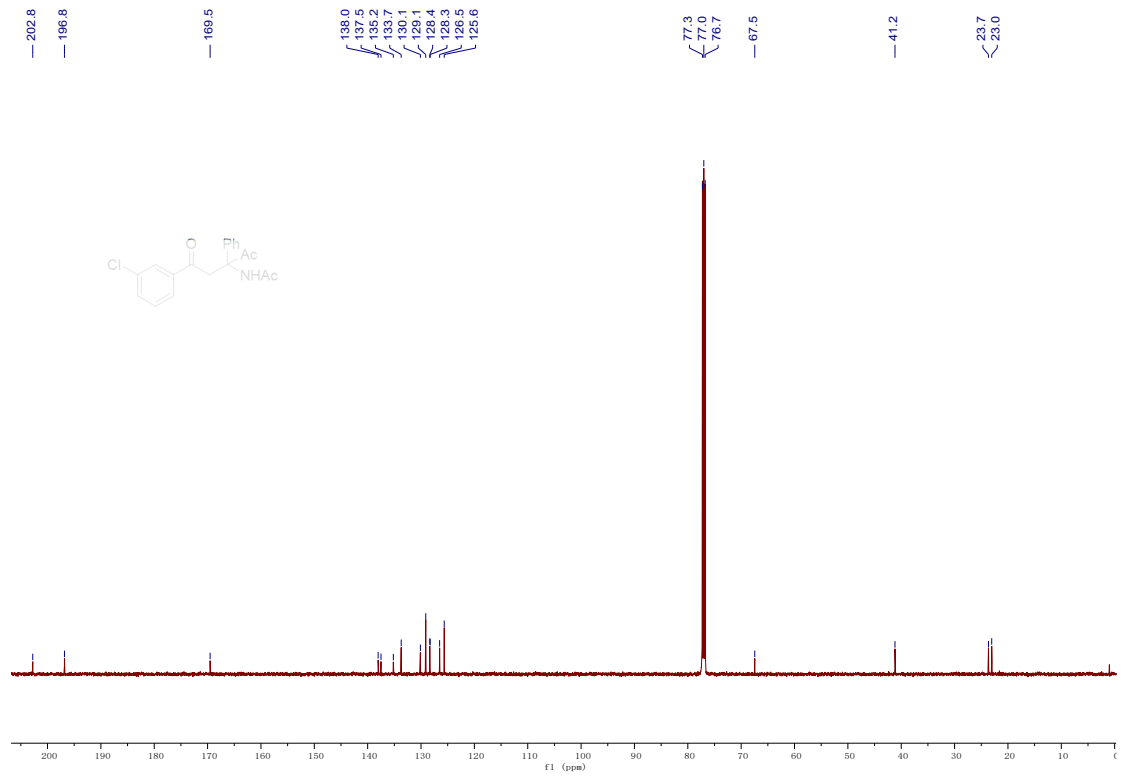


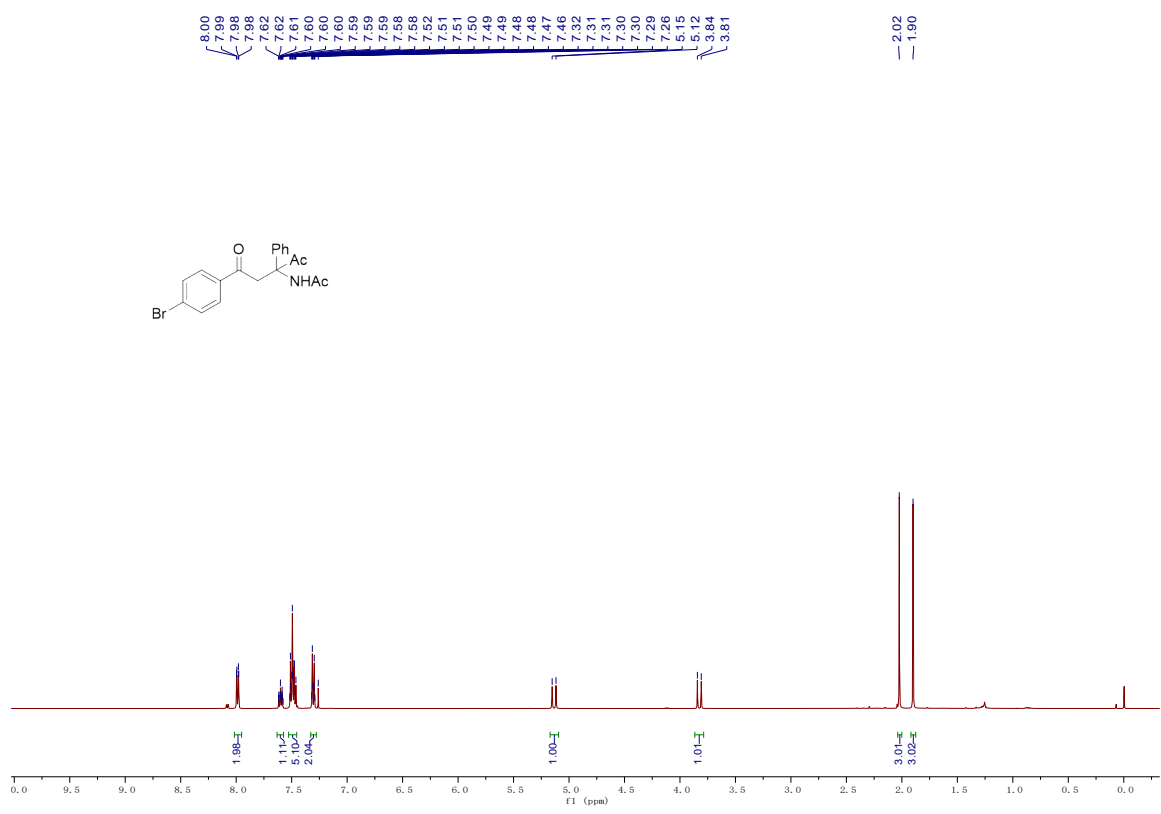
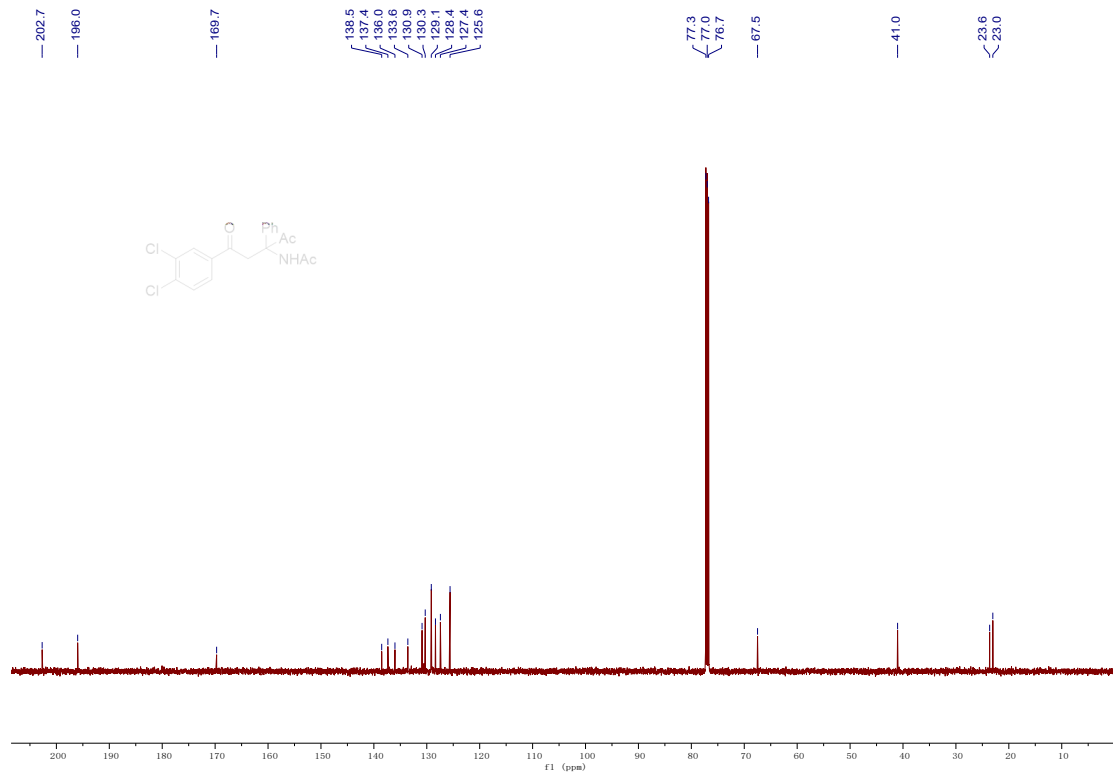


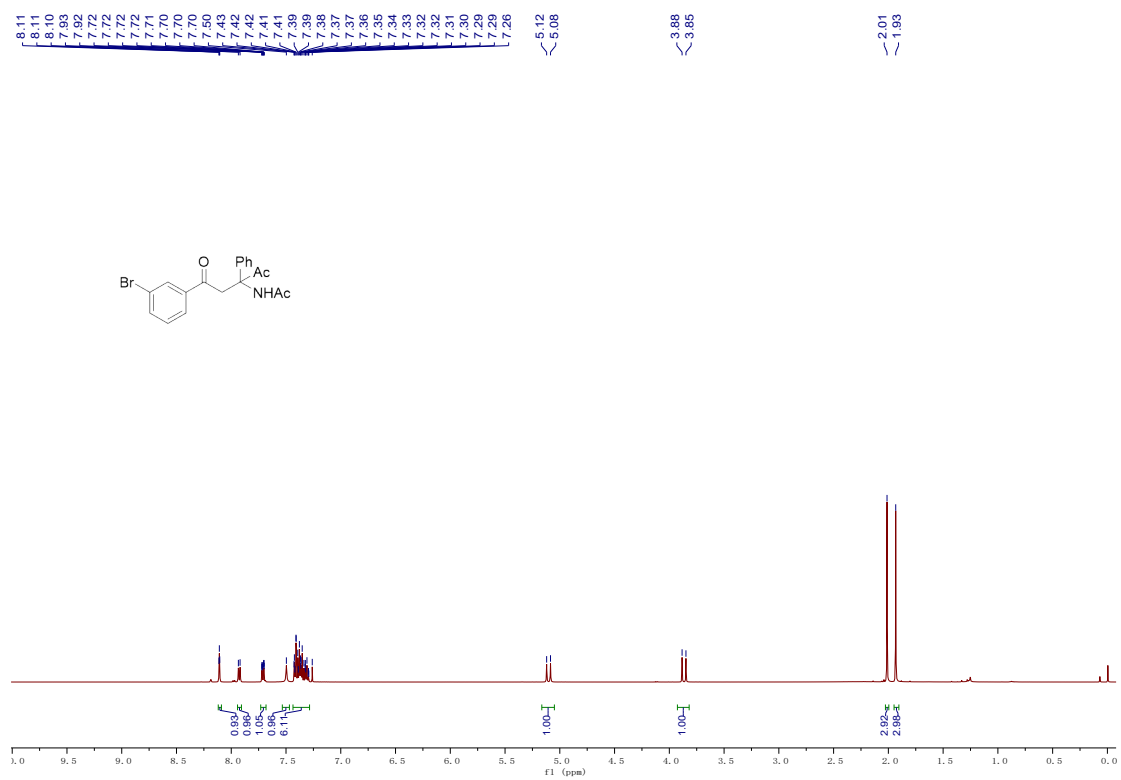
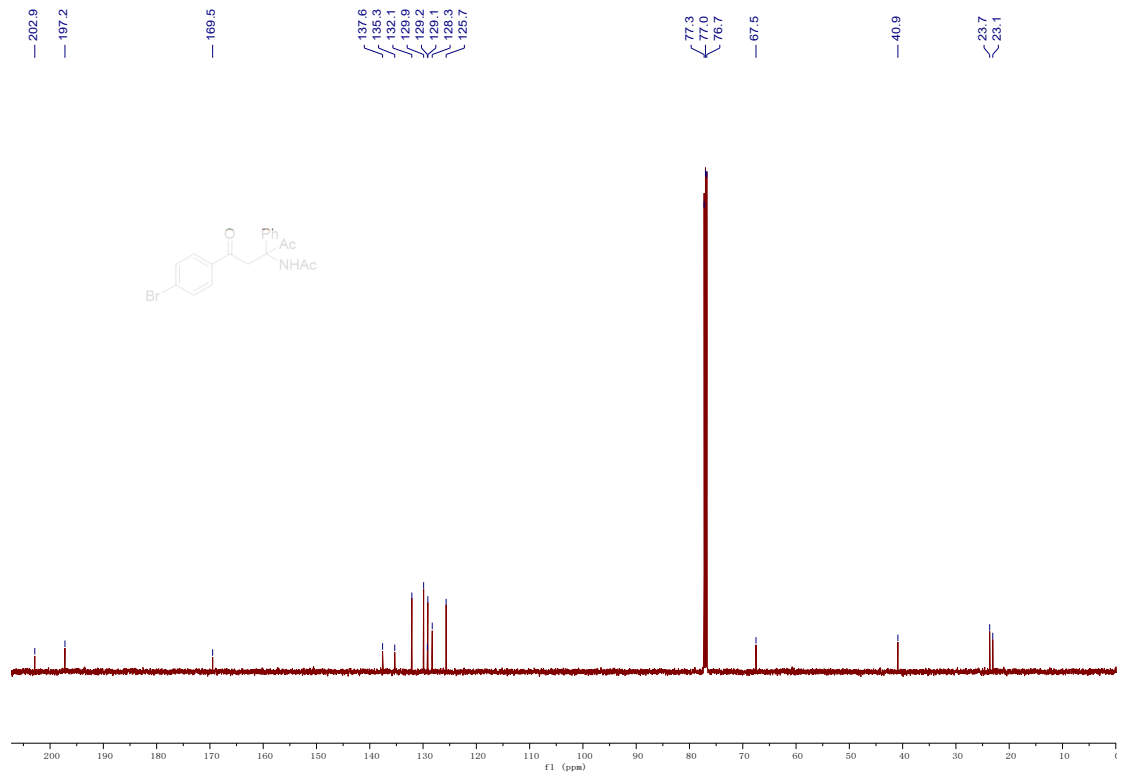


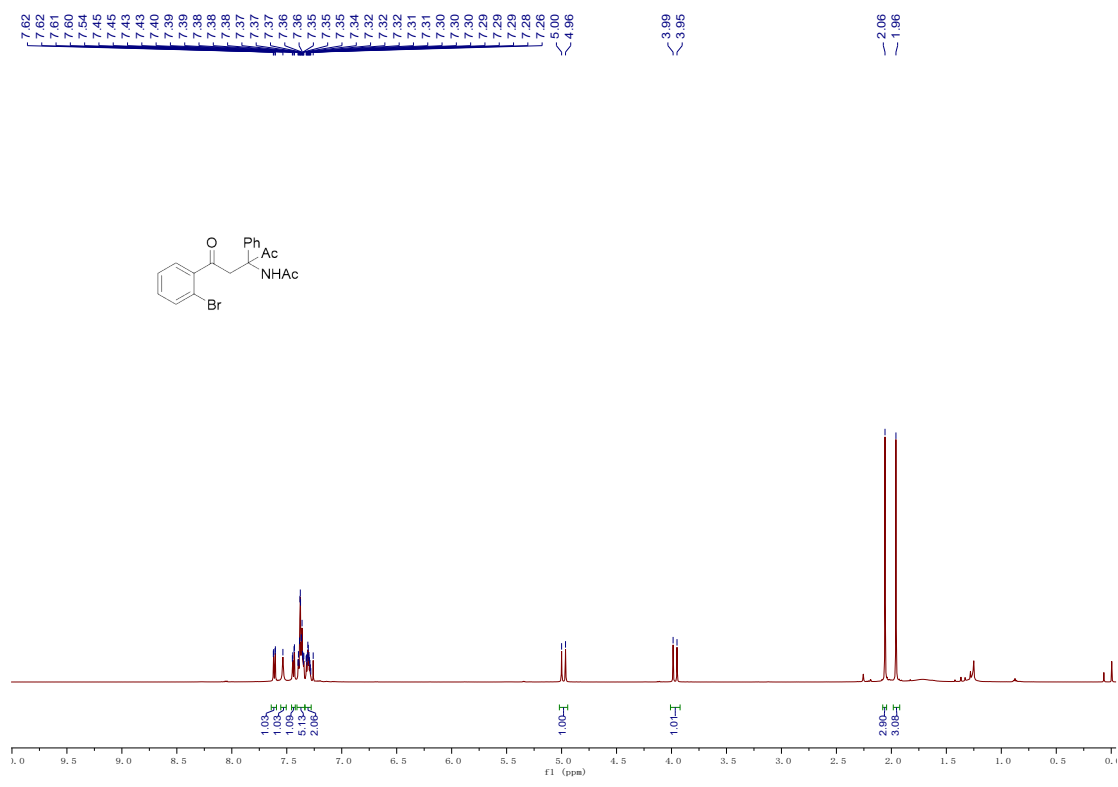
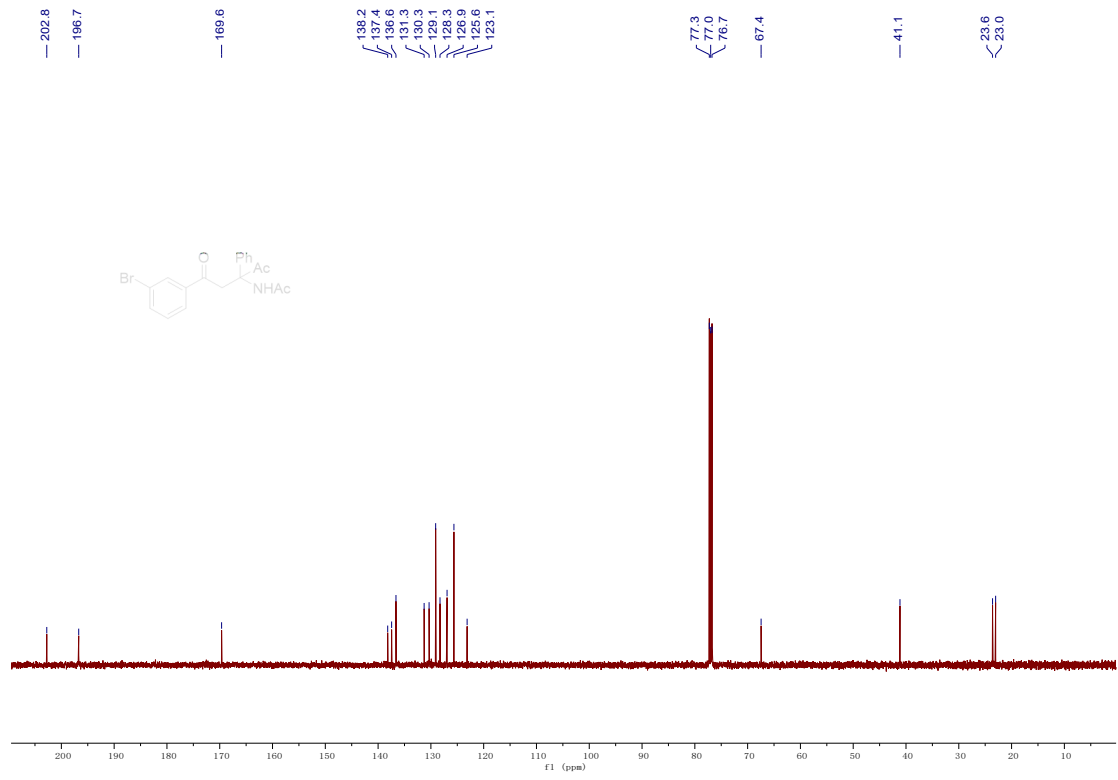


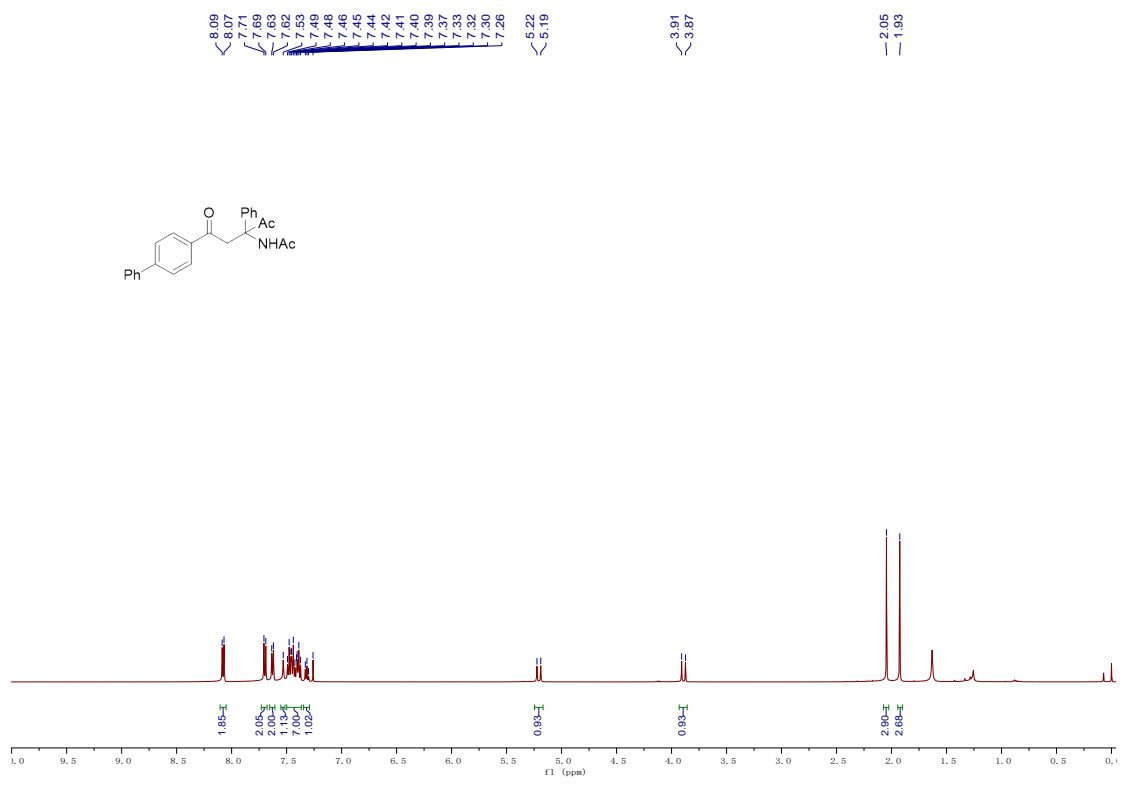
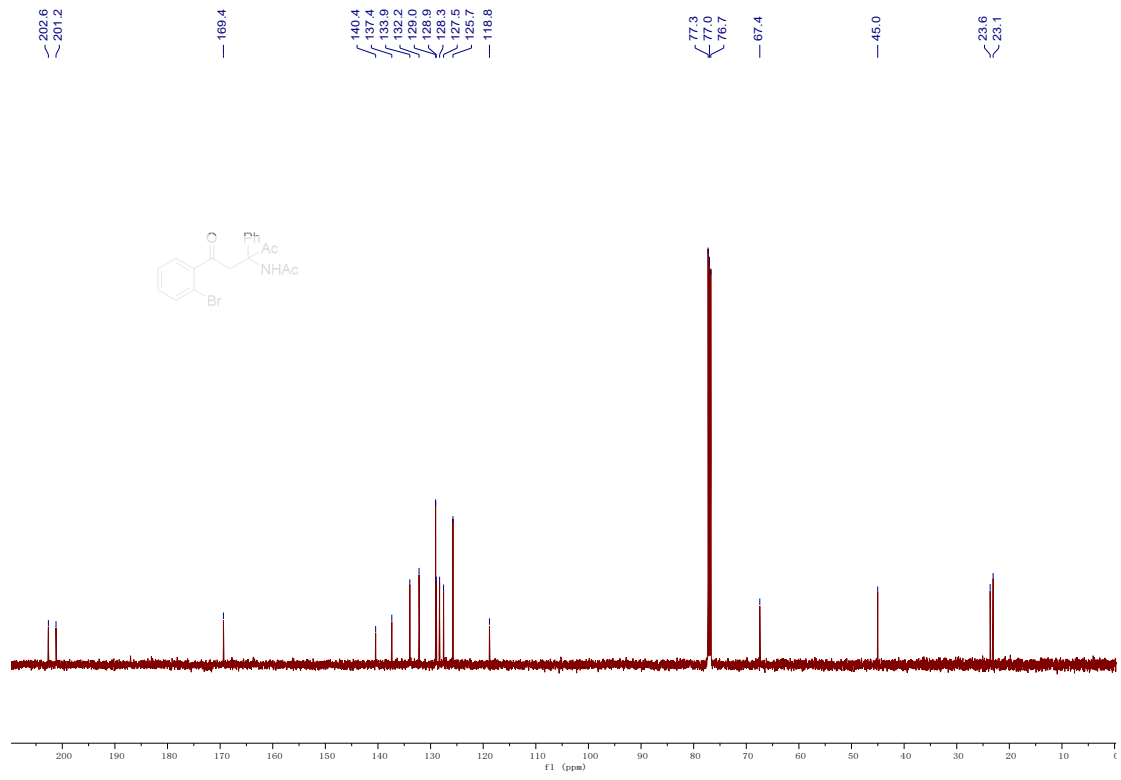


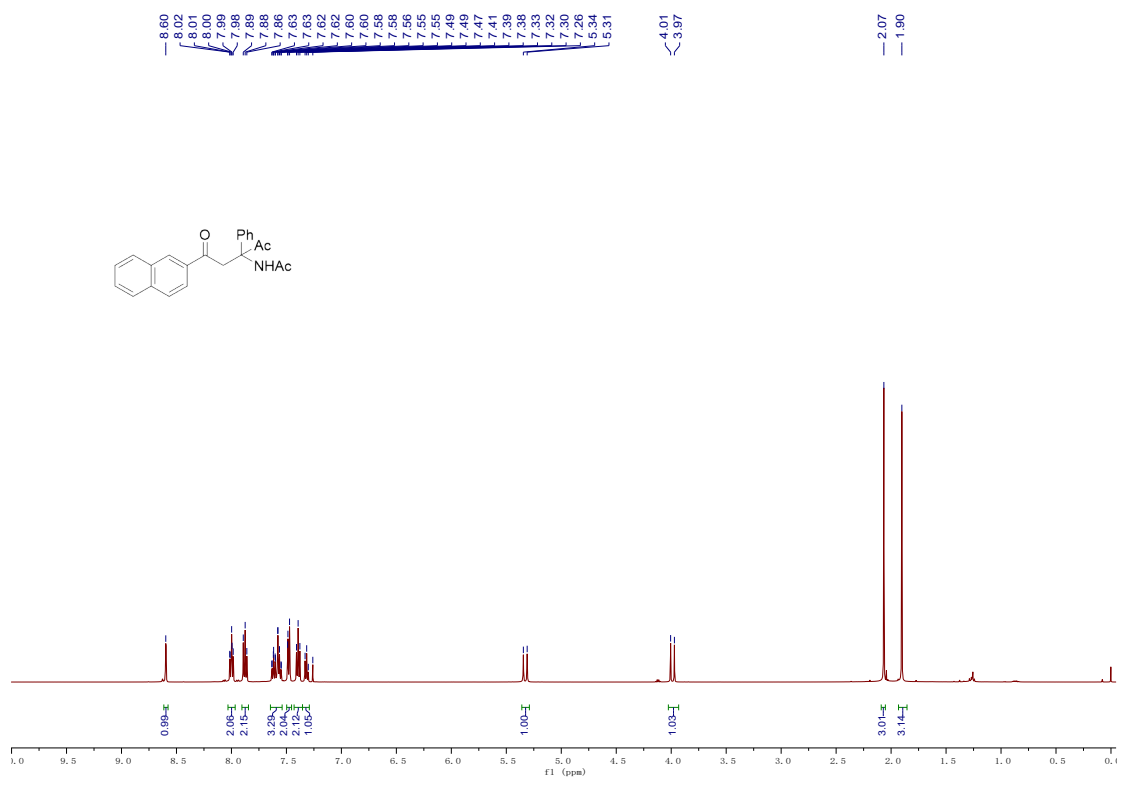
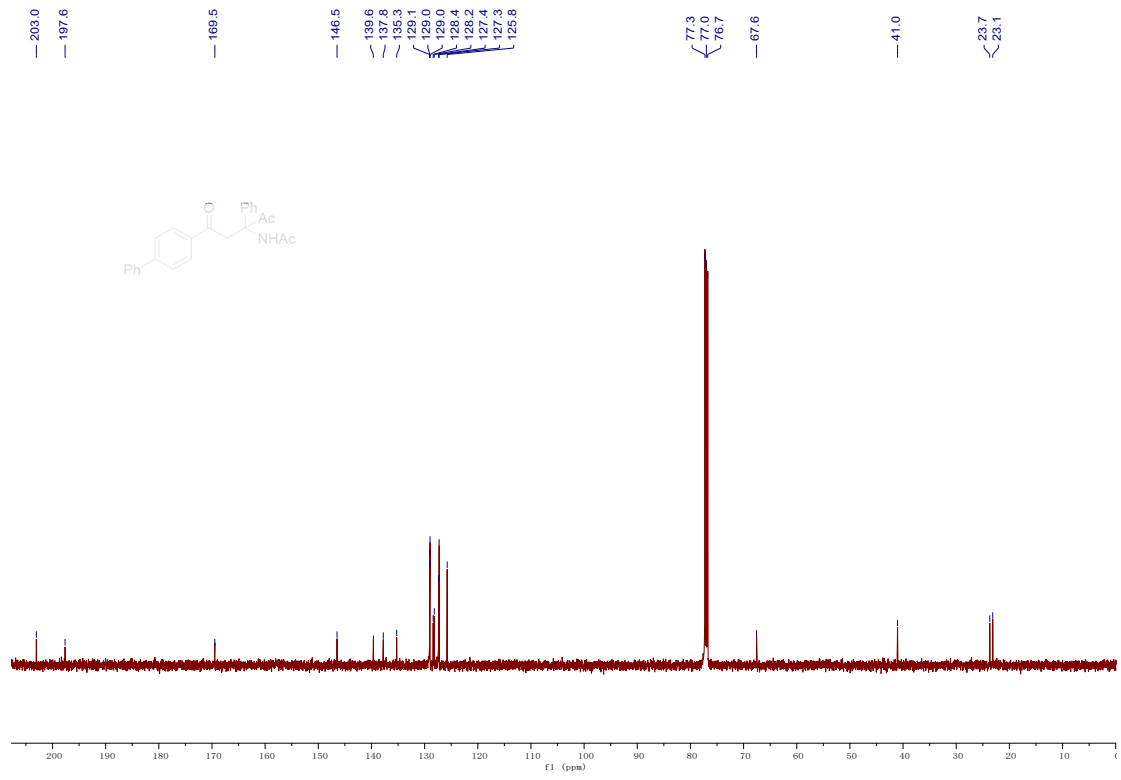


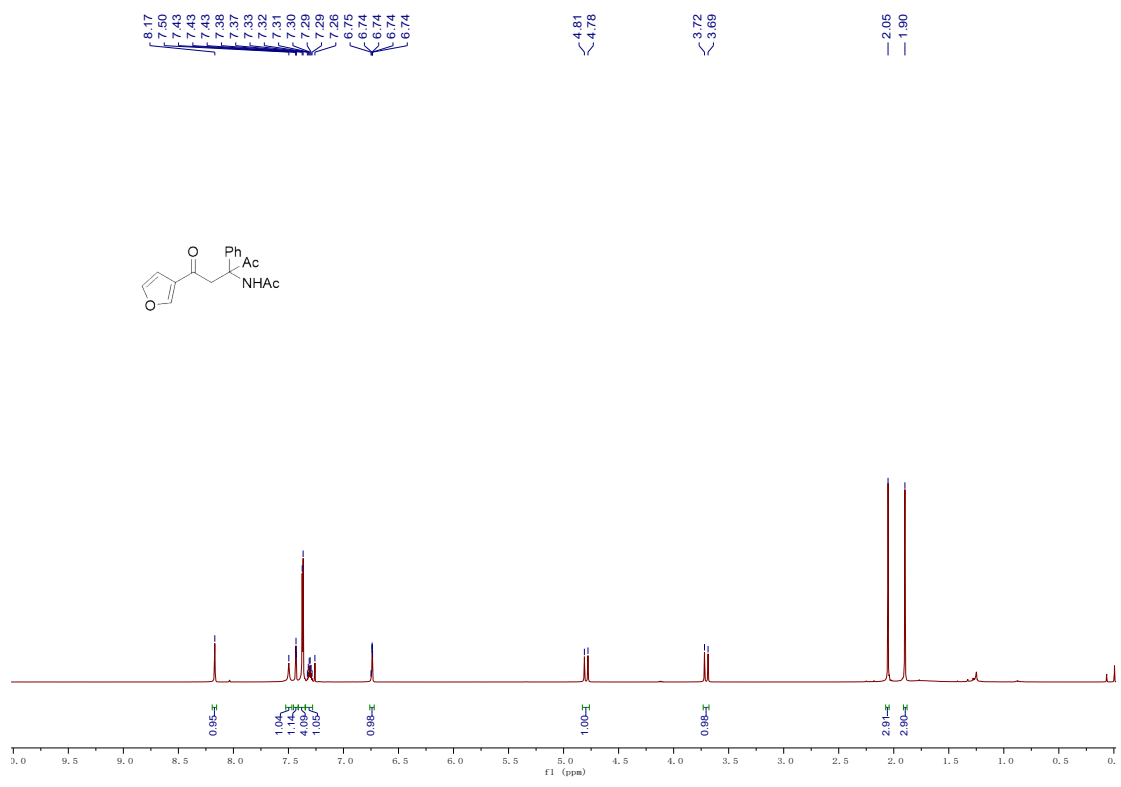
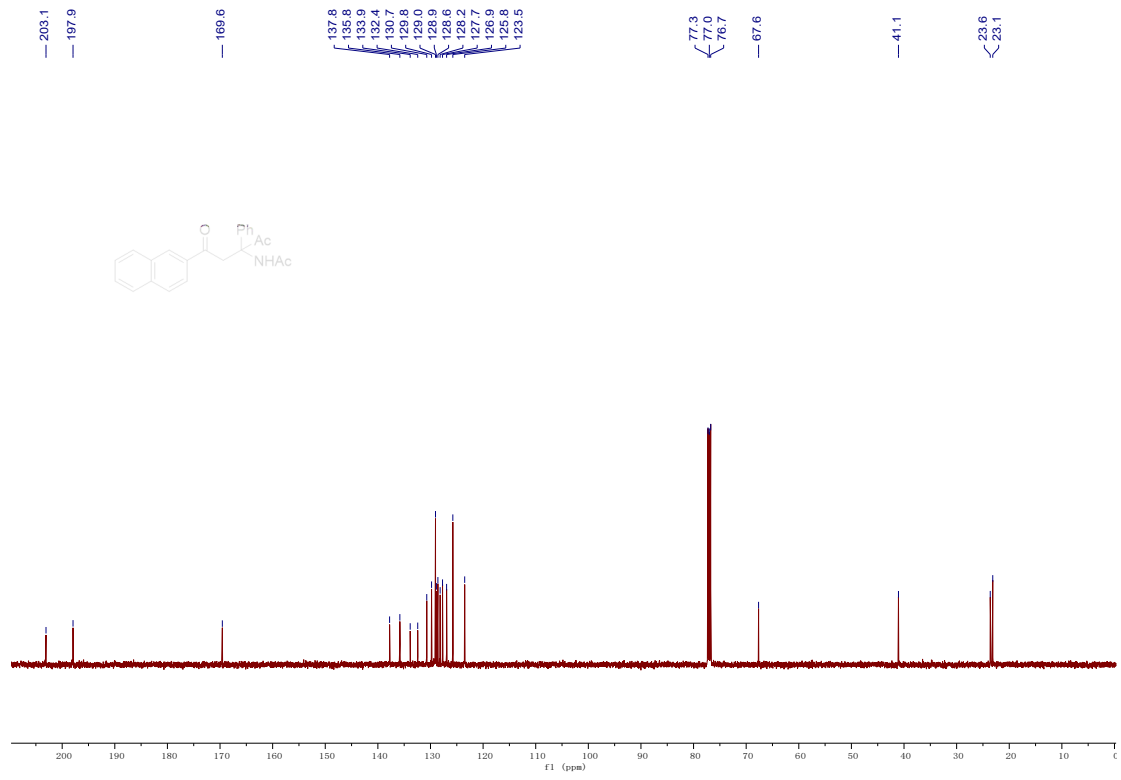




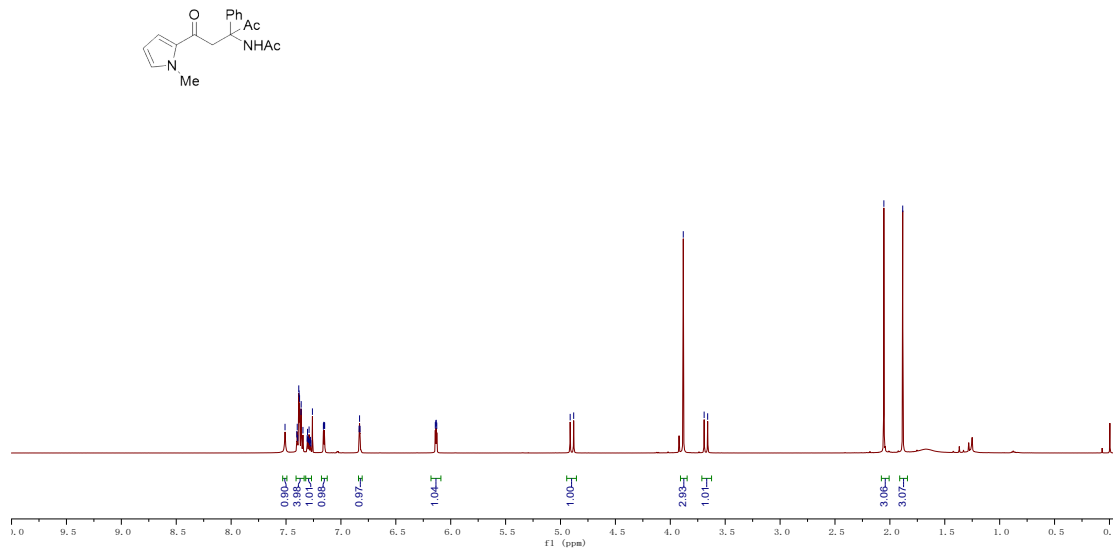
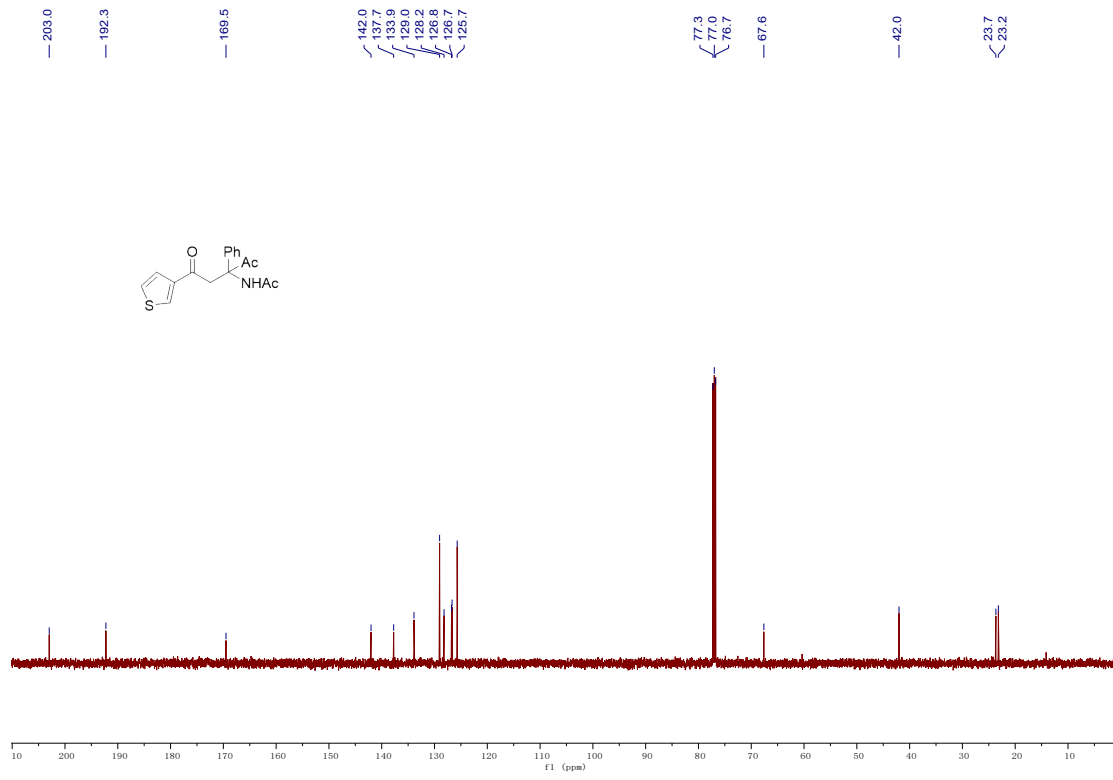


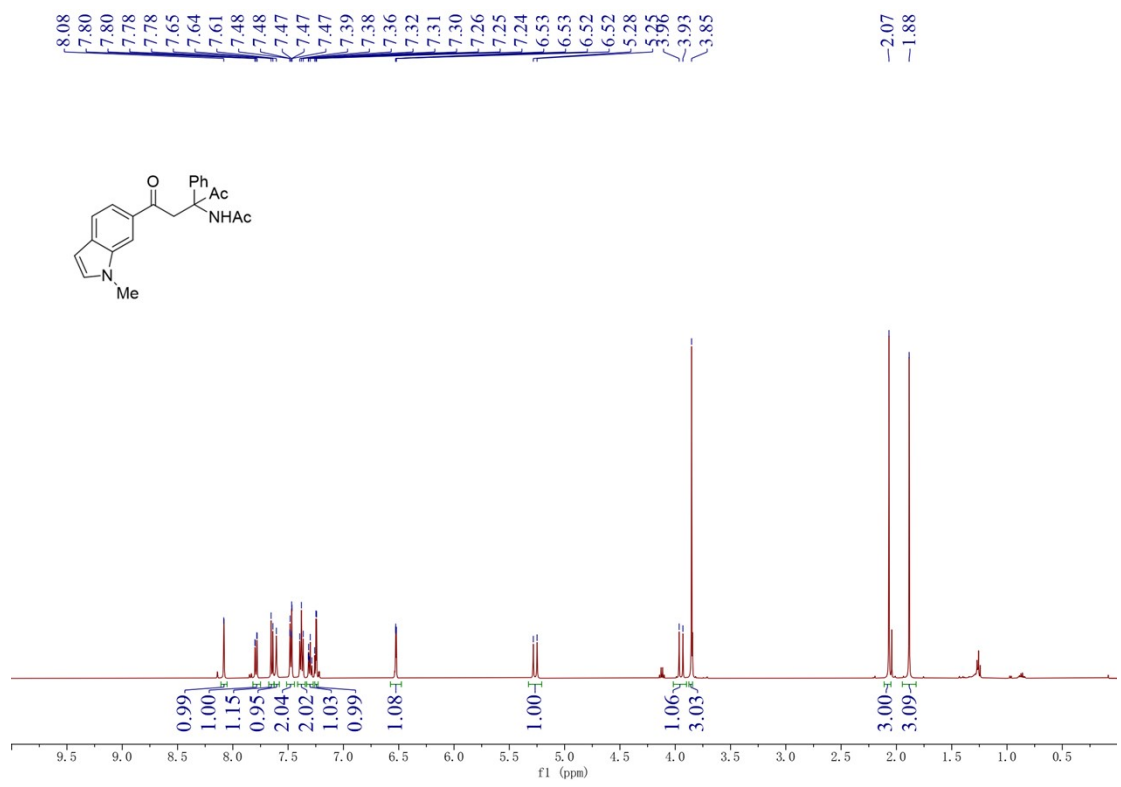
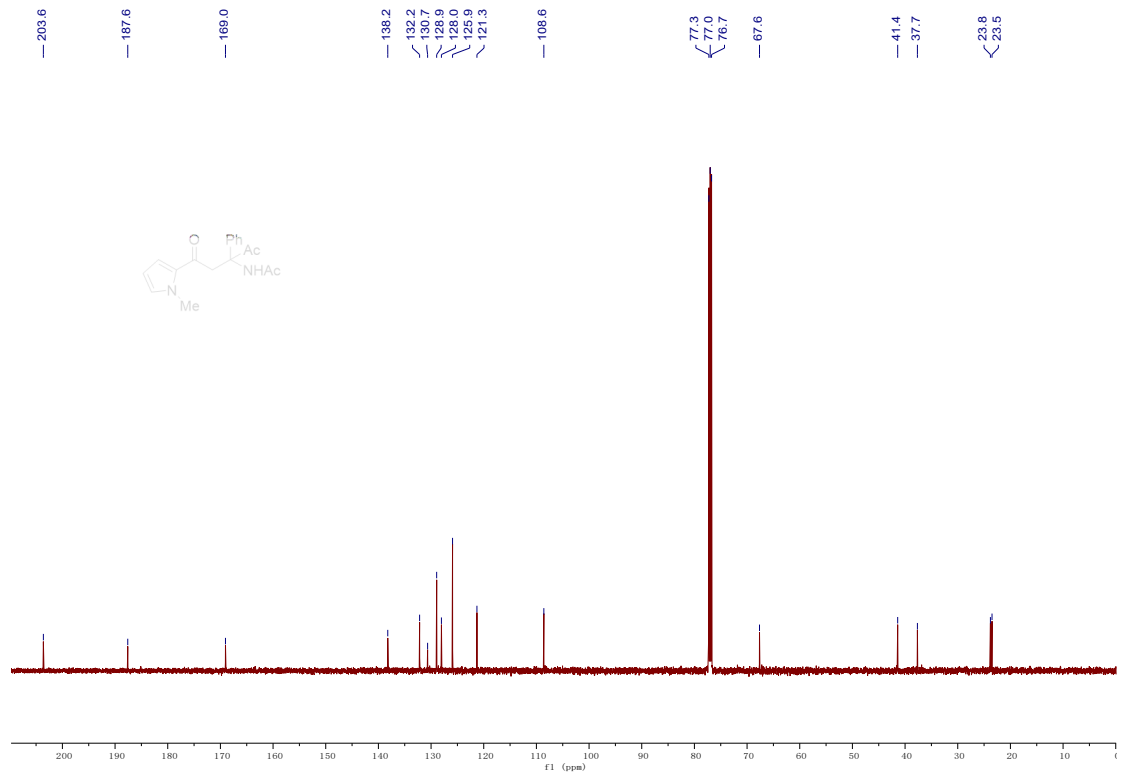


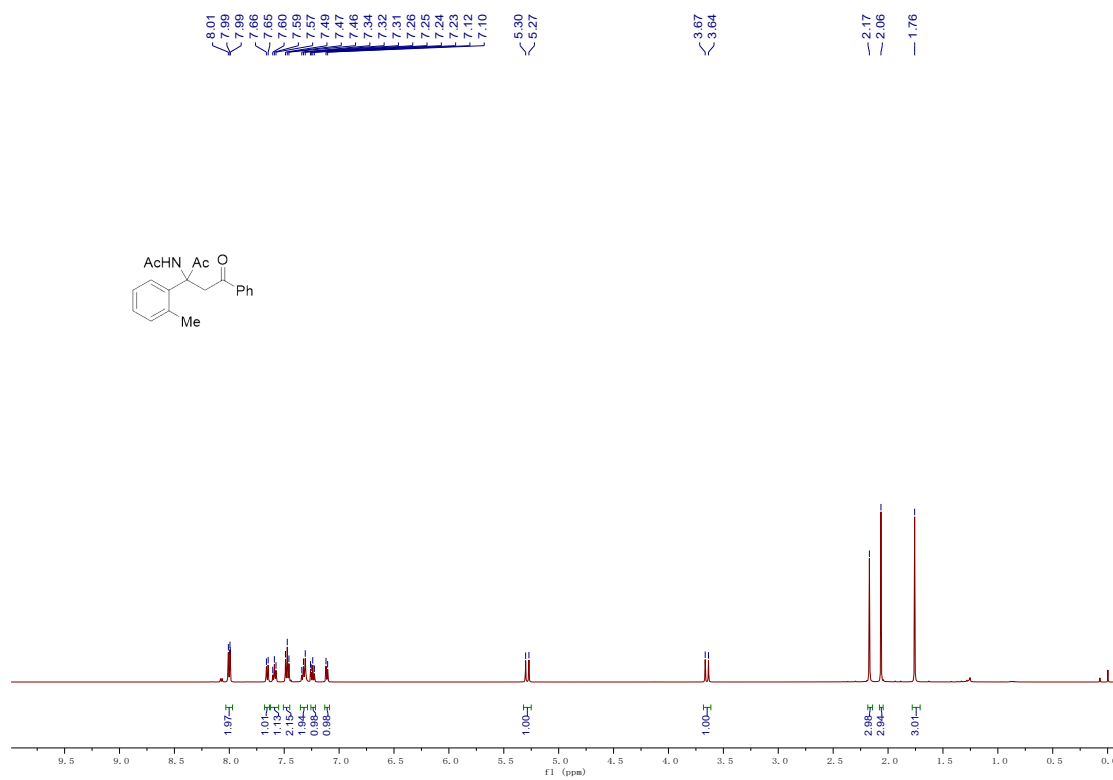
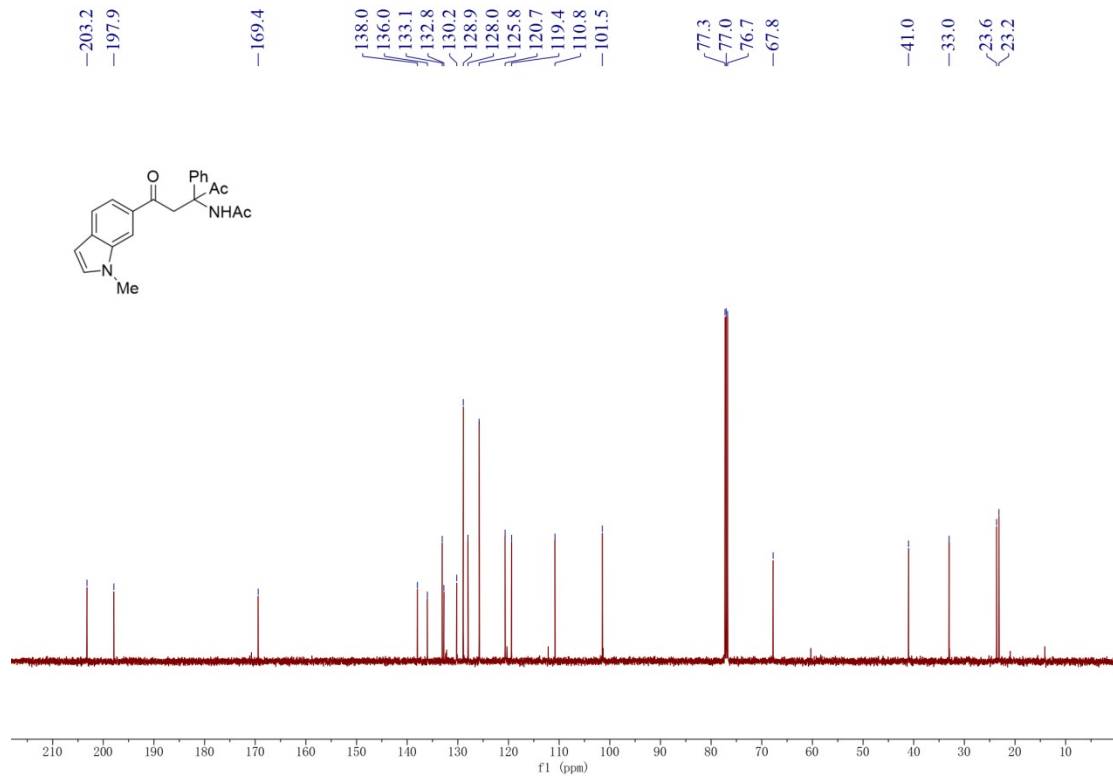


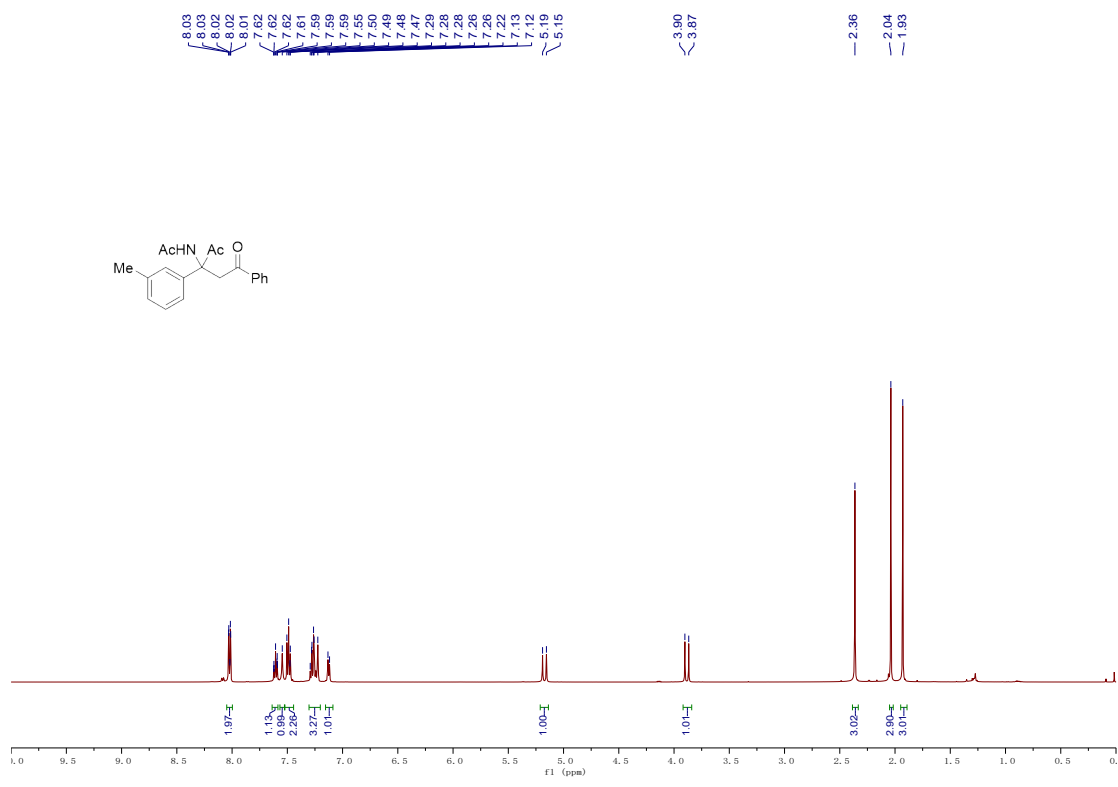
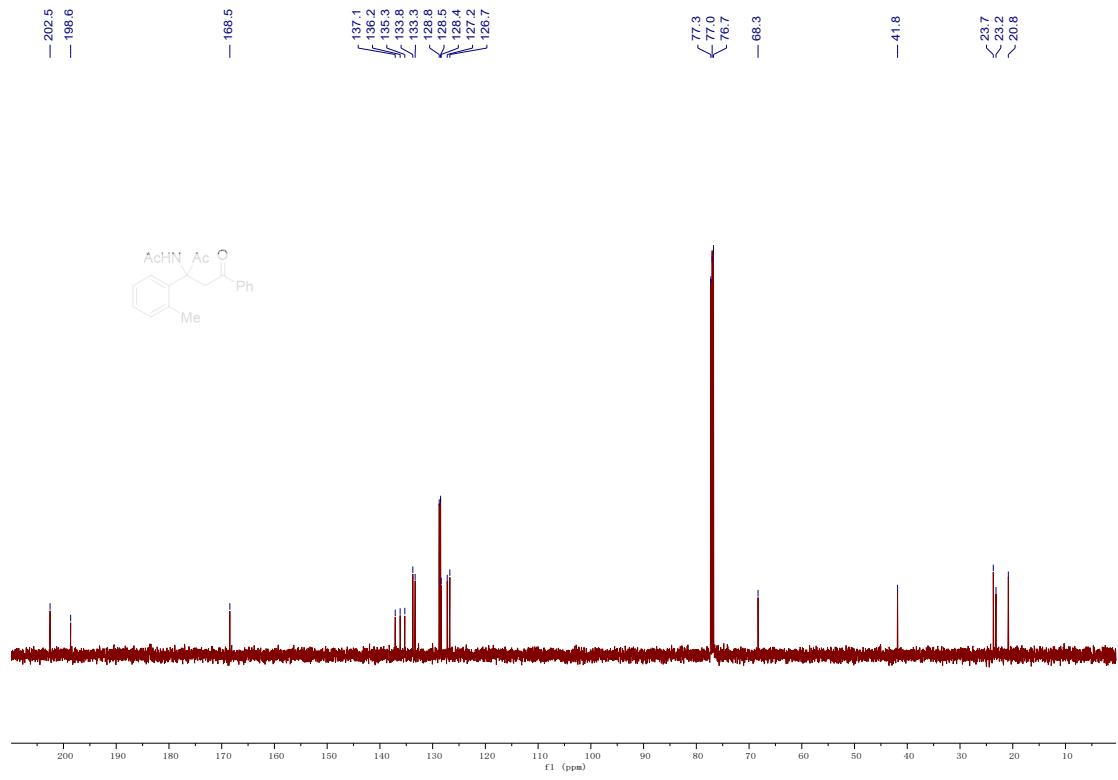


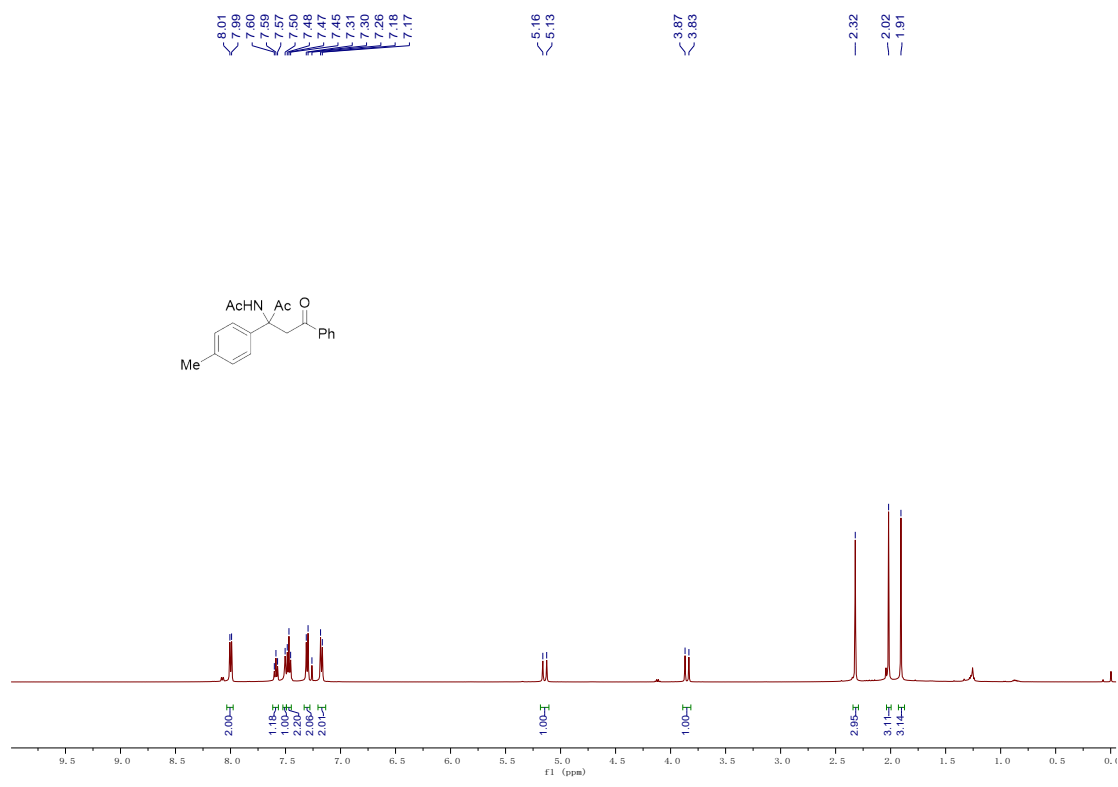
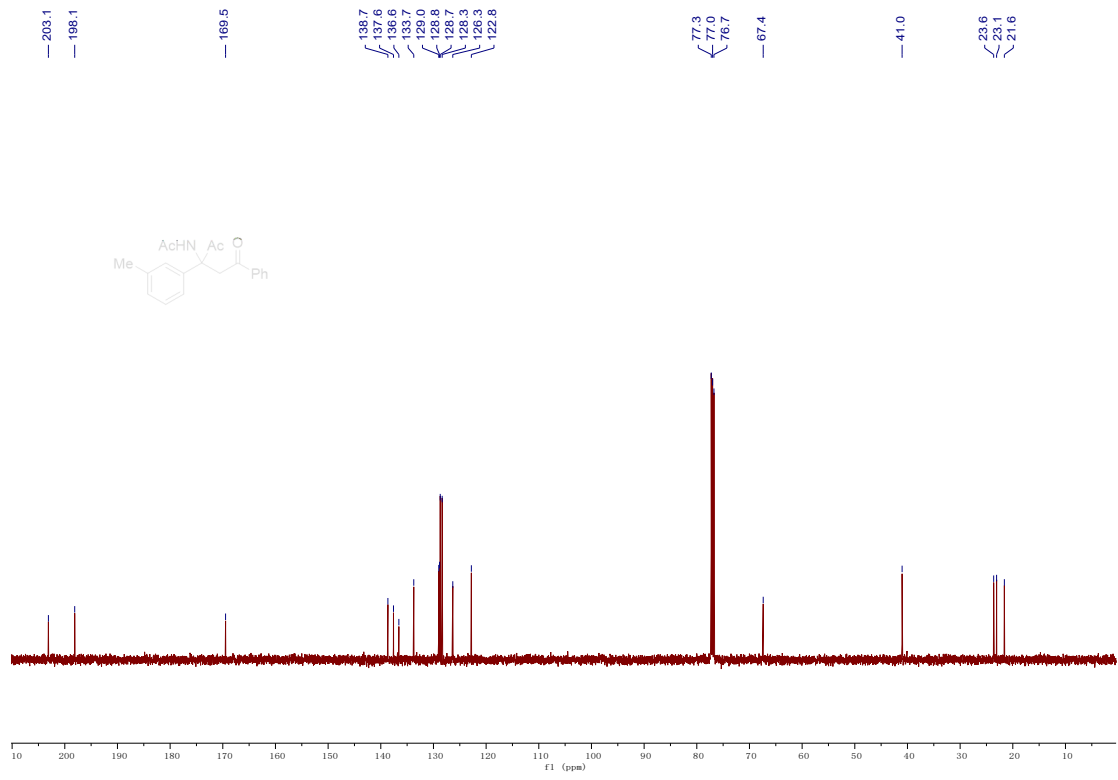


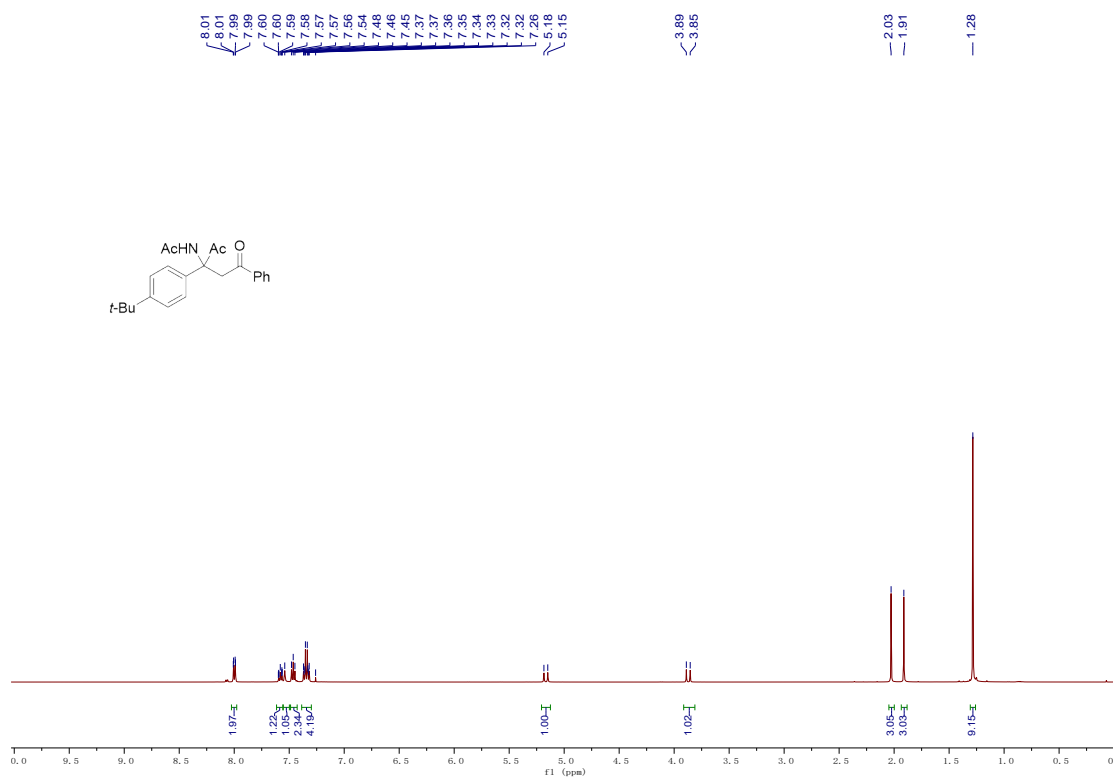
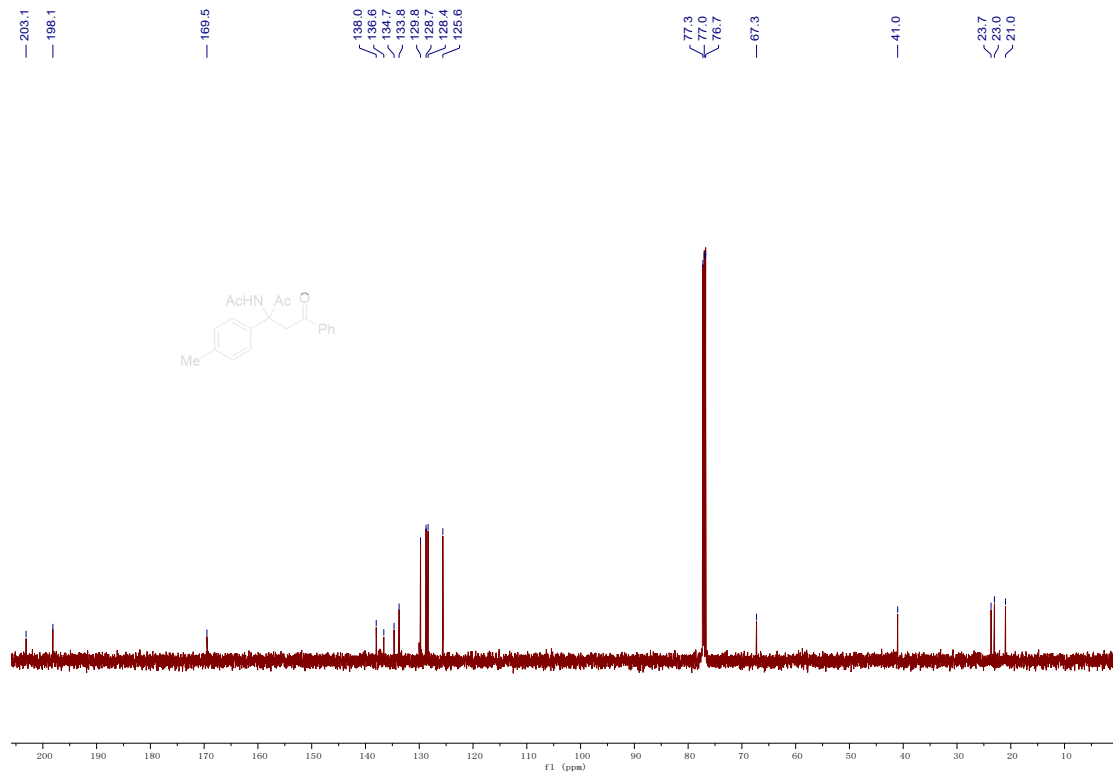


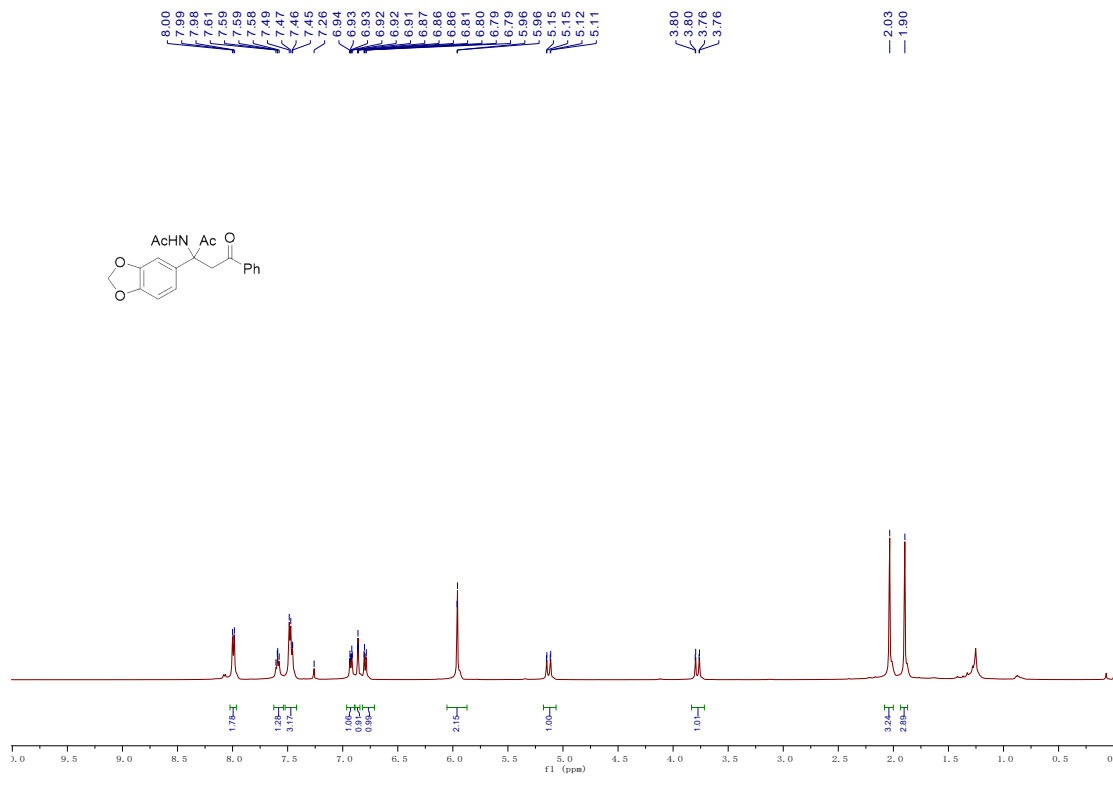
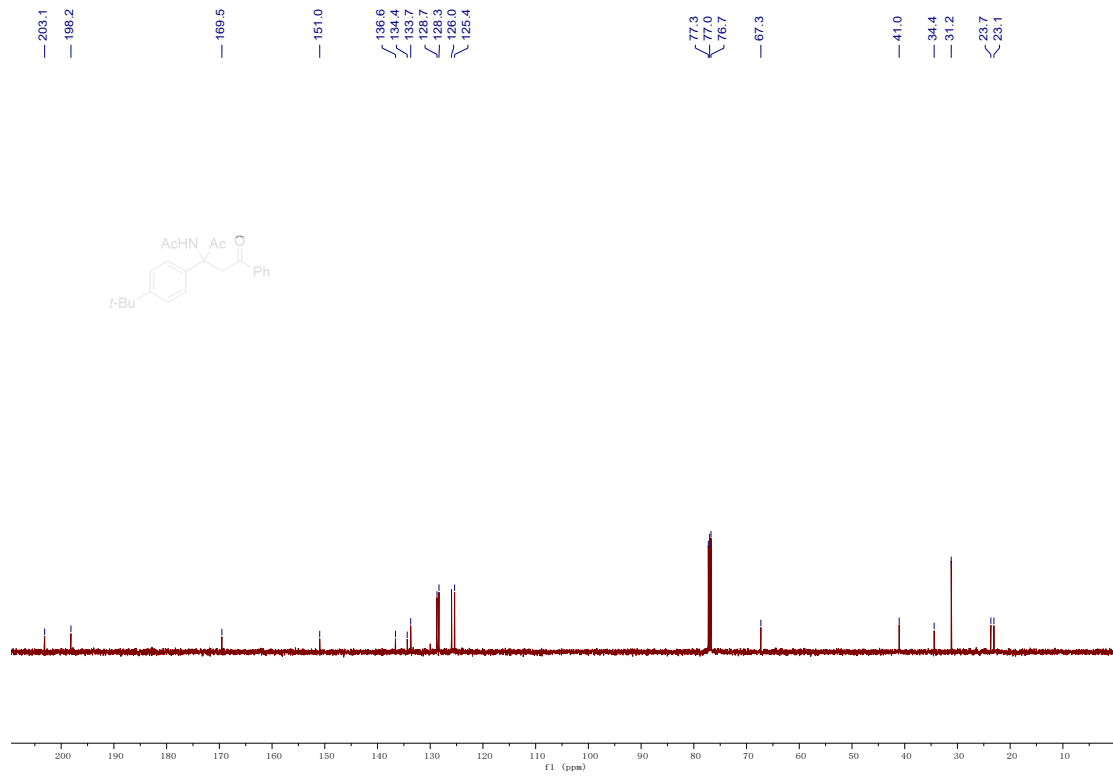


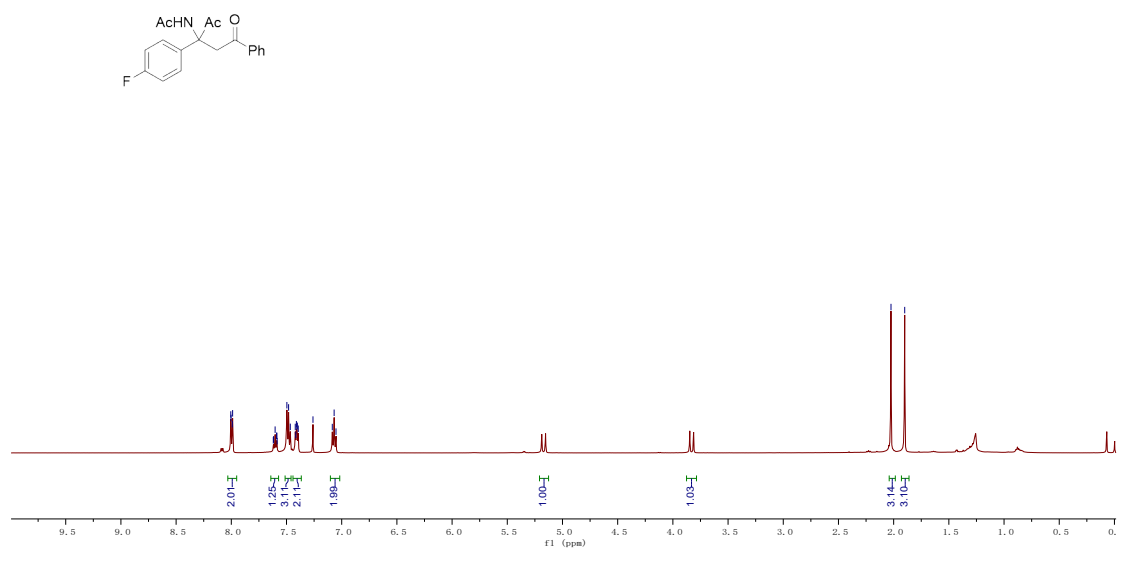
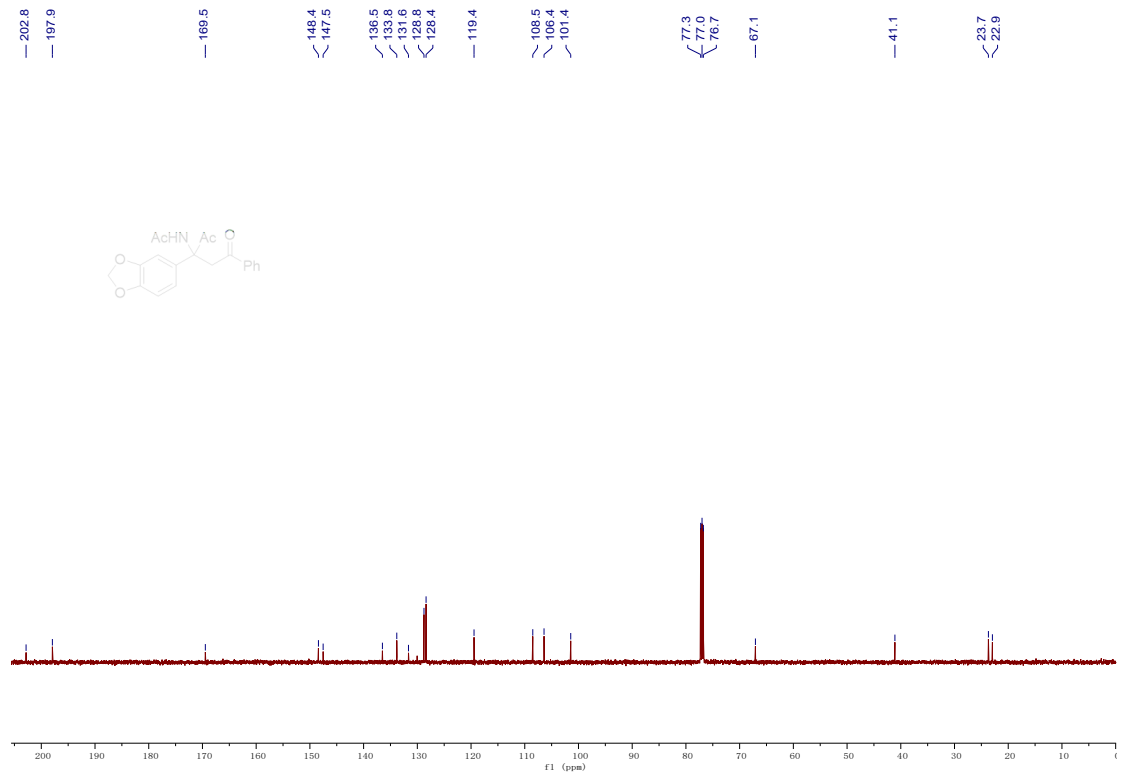


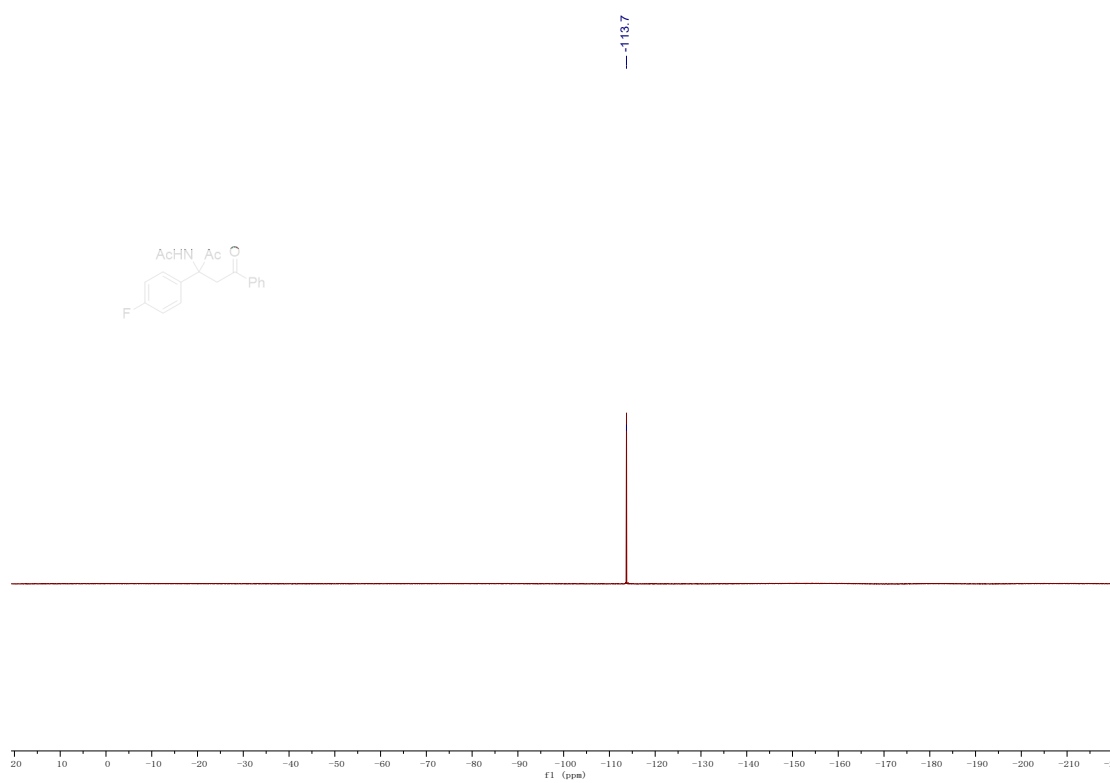
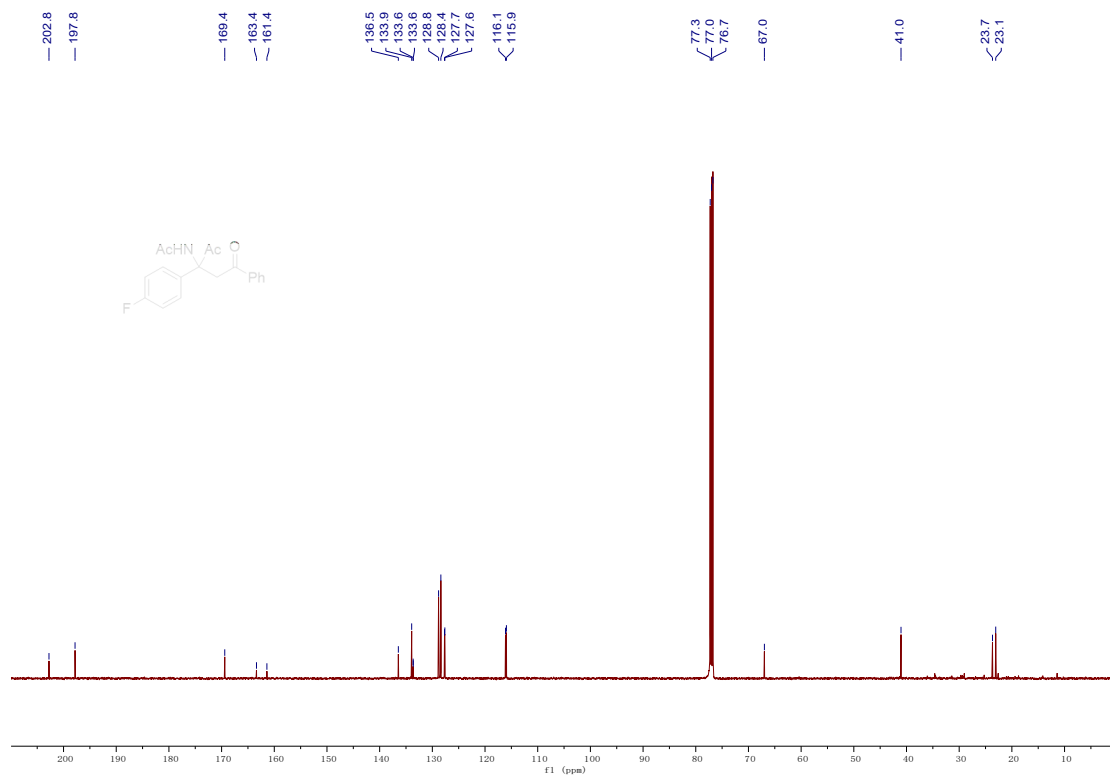


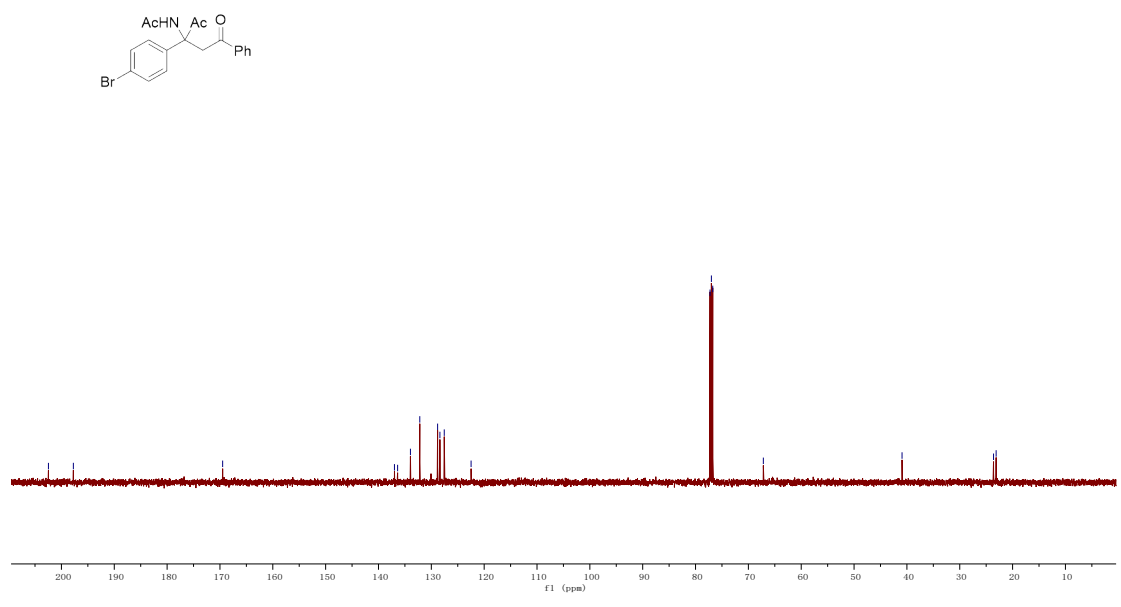
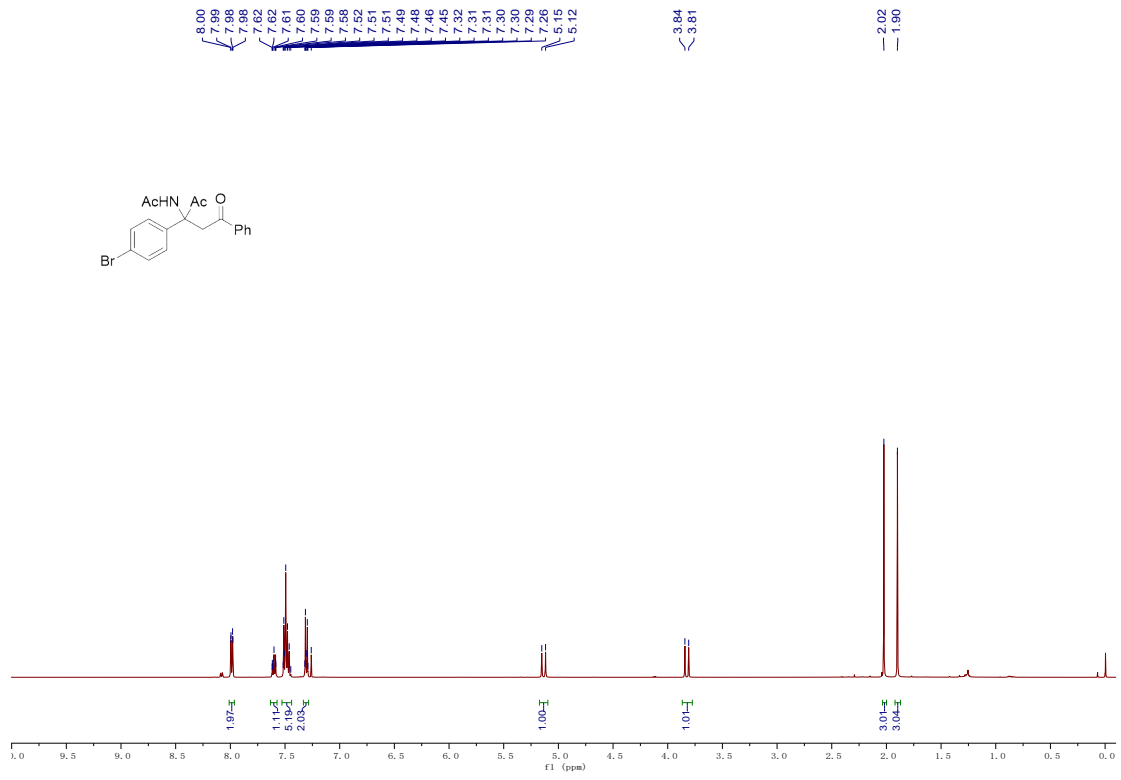


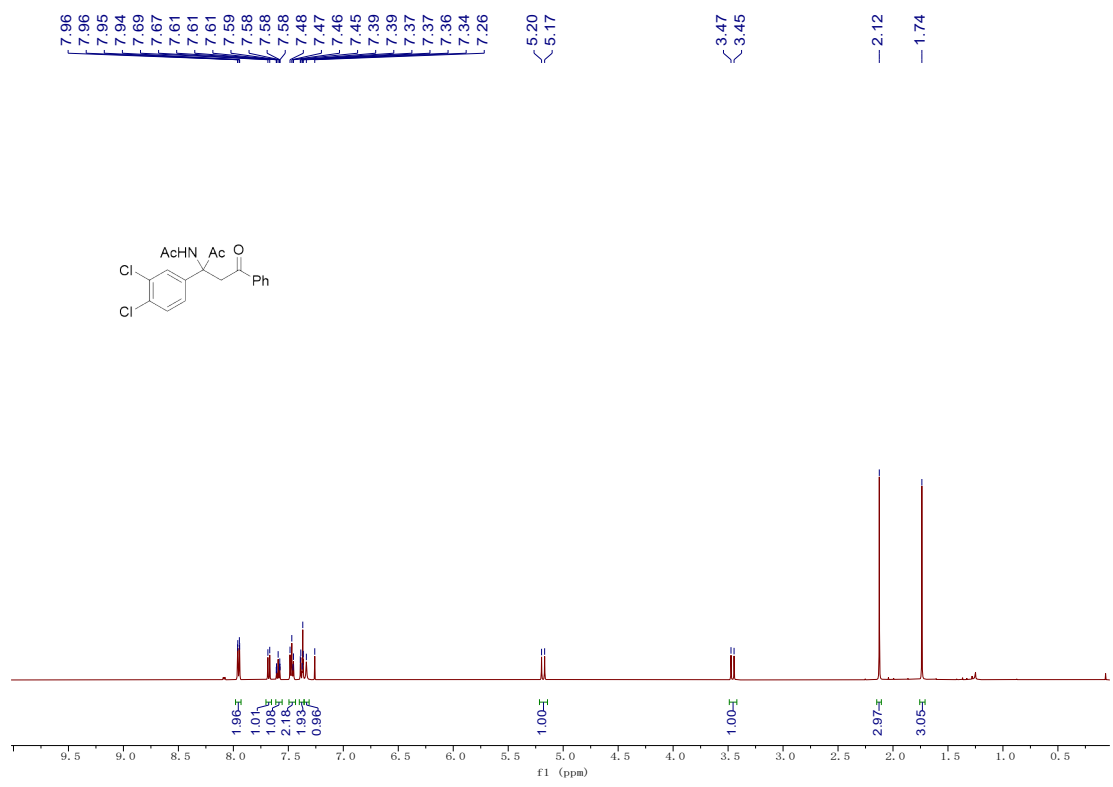


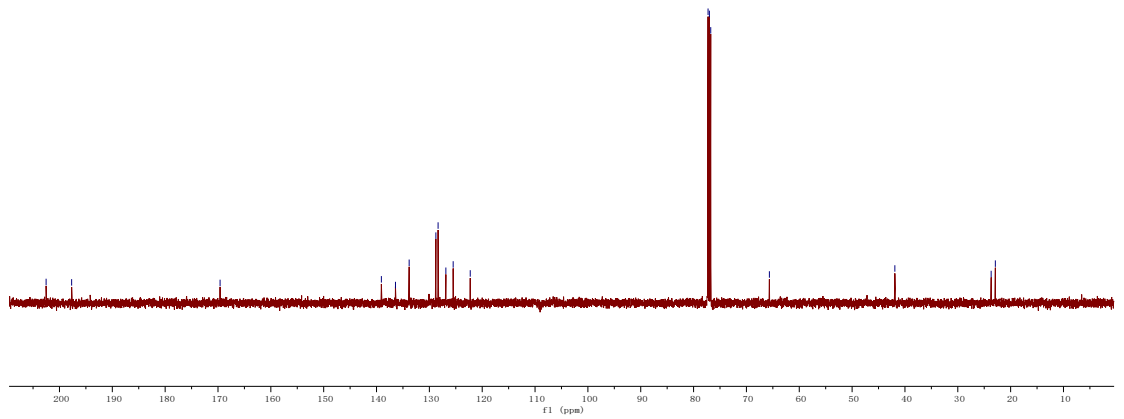
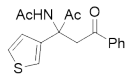
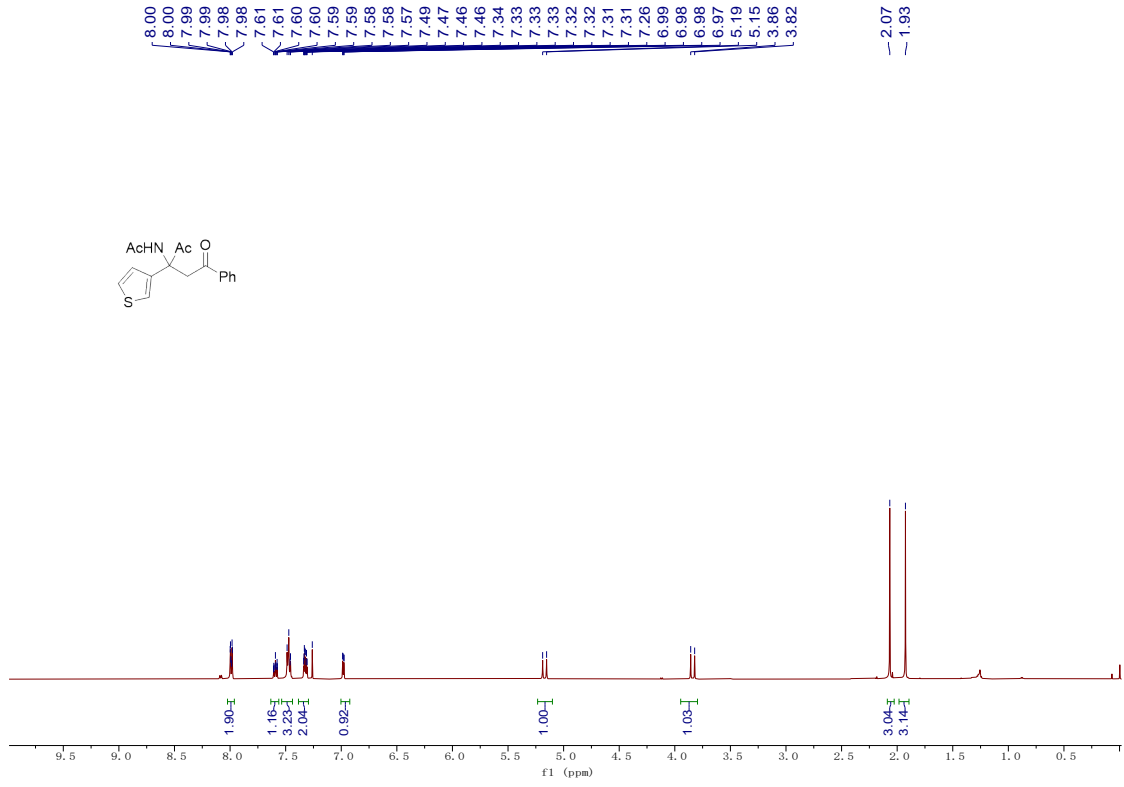
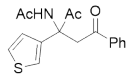




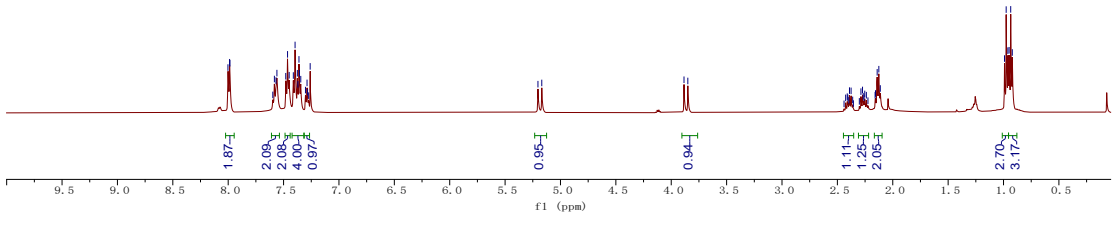
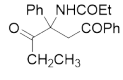








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