

## Supplementary Information

For

### Metal-free Intermolecular C–O Cross-Coupling Reactions: Synthesis of *N*-hydroxyimide esters

Yunhe Lv,\* Kai Sun, Weiya Pu, Shukuan Mao, Gang Li, Jiejie Niu, Qian Chen, and  
Tingting Wang

<sup>a</sup> College of Chemistry and Chemical Engineering, Anyang Normal University,  
Anyang, 455000, China. E-mail: [lvyunhe0217@163.com](mailto:lvyunhe0217@163.com)

<sup>b</sup> Jilin Province Key Laboratory of Organic Functional Molecular Design & Synthesis,  
Changchun, 130024, China. E-mail: [luyh086@nenu.edu.cn](mailto:luyh086@nenu.edu.cn)

## Table of Contents

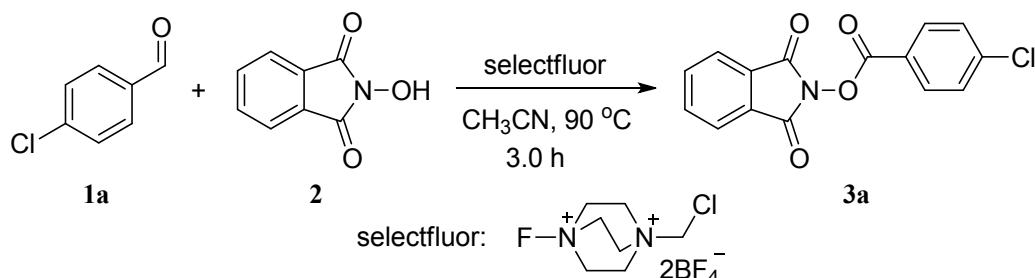
I . General Considerations	S2
II . General procedure for the preparation of 3	S2
III. General procedure for the preparation of 5	S2
IV. General procedure for the preparation of 7	S3
V . Analytical data of Compounds 3, 5 and 7	S3
VI. <sup>1</sup> H and <sup>13</sup> C Spectra of Compounds 3, 5 and 7	S12

## I . General Considerations

All reagents were purchased from commercial sources and used without further treatment, unless otherwise indicated. All reactions were run under air with no precautions taken to exclude moisture.  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were recorded at 25 °C on a Varian (400 MHz and 100 MHz). Melting points were obtained with a micro melting point XT4A Beijing Keyi electrooptic apparatus and are uncorrected. High resolution mass spectra were recorded on Bruker microtof. All reactions were monitored by TLC with Taizhou GF254 silica gel coated plates. Flash column chromatography was carried out using 200-300 mesh silica gel at increased pressure.

## II . General procedure for the preparation of 3

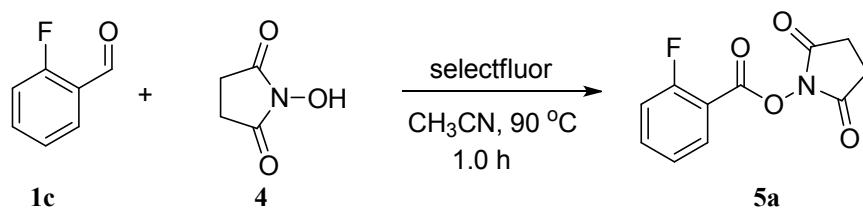
### 3a as an example



To a solution of the *N*-Hydroxyphthalimide (NHPI) **2** (58.7 mg, 0.36 mmol) in acetonitrile (3.0 ml) was added the 4-chlorobenzaldehyde **1a** (35  $\mu\text{L}$ , 0.3 mmol) and selectfluor (127.5 mg, 0.36 mmol) in a sealed tube. The sealed tube was then tightly sealed with a screw cap and the reaction was stirred for the 3.0 h at 90 °C. After the reaction finished, the reaction mixture was cooled to room temperature. The mixture was extracted with ethyl acetate ( $3 \times 5.0$  mL), the combined organic phases were dried over anhydrous  $\text{Na}_2\text{SO}_4$  and the solvent was evaporated under vacuum. The residue was purified by column chromatography to give the corresponding products **3a** (82.3 mg, 91%).

## III. General procedure for the preparation of 5

### 3a as an example

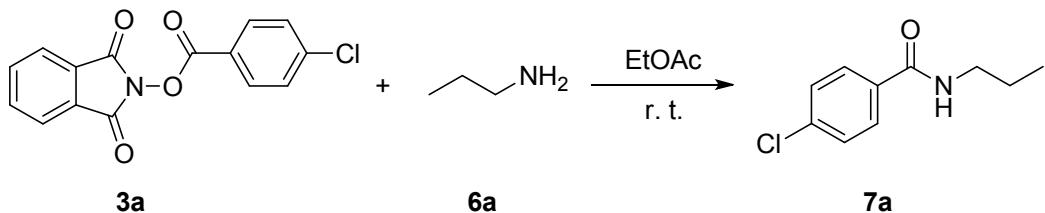


To a solution of the *N*-Hydroxysuccinimide **4** (NHSI, 41.4 mg, 0.36 mmol) in acetonitrile (3.0 ml) was added the 2-fluorobenzaldehyde **1c** (32  $\mu\text{L}$ , 0.3 mmol) and selectfluor (127.5 mg, 0.36 mmol)

in a sealed tube. The sealed tube was then tightly sealed with a screw cap and the reaction was stirred for the 1.0 h at 90 °C. After the reaction finished, the reaction mixture was cooled to room temperature. The mixture was extracted with ethyl acetate ( $3 \times 5.0$  mL), the combined organic phases were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and the solvent was evaporated under vacuum. The residue was purified by column chromatography to give the corresponding products **5a** (50.5 mg, 71%).

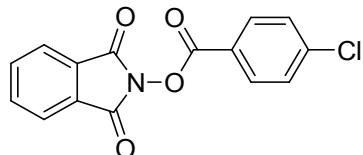
#### IV. General procedure for the preparation of 7

##### **5a** as an example



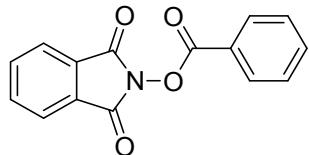
To a solution of **3a** (60.3 mg, 0.2 mmol) in EtOAc (2.0 mL) was added propan-1-amine **6a** (49.2  $\mu$ L, 0.6 mmol), and the reaction mixture was stirred at room temperature. The progress of the reaction was monitored by TLC using ethyl acetate and petroleum ether as eluent. After completion, the crude mixture was concentrated and purified by column chromatography to afford the desired products **7a** (32.8 mg, 83%).

#### V. Analytical data of Compounds 3, 5 and 7



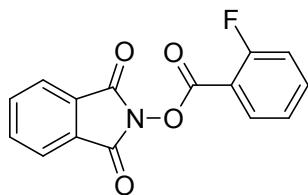
##### **1,3-dioxoisindolin-2-yl methyl terephthalate 3a<sup>[1]</sup>**

White solid. mp: 182–183 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.52 (d,  $J$  = 8.8 Hz, 2H), 7.81 (dd,  $J_1$  = 3.2 Hz,  $J_2$  = 5.6 Hz, 2H), 7.92 (dd,  $J_1$  = 3.2 Hz,  $J_2$  = 5.6 Hz, 2H), 8.13 (d,  $J$  = 8.4 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 123.7, 124.0, 128.9, 129.3, 131.9, 134.8, 141.6, 161.9, 162.0. HRMS (ESI-TOF) Calcd for C<sub>15</sub>H<sub>9</sub>ClNO<sub>4</sub>, [M+H]<sup>+</sup> *m/z* 302.0220; Found 302.0208.



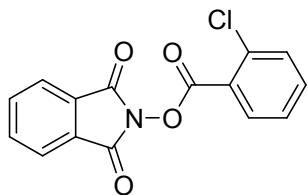
##### **1,3-dioxoisindolin-2-yl benzoate 3b<sup>[1]</sup>**

White solid. mp: 98–101 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.54 (t,  $J$  = 7.6 Hz, 2H), 7.70 (t,  $J$  = 7.6 Hz, 2H), 7.81 (dd,  $J_1$  = 3.2 Hz,  $J_2$  = 5.2 Hz, 2H), 7.92 (dd,  $J_1$  = 3.2 Hz,  $J_2$  = 5.2 Hz, 2H), 8.19 (d,  $J$  = 7.6 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  = 124.0, 125.3, 128.8, 129.0, 130.6, 134.8, 134.8, 162.0, 162.8. HRMS (ESI-TOF) Calcd for C<sub>15</sub>H<sub>10</sub>NO<sub>5</sub>, [M+H]<sup>+</sup> *m/z* 268.0610; Found 268.0615.



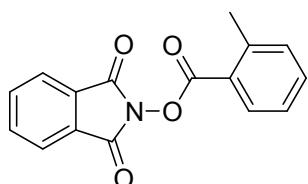
**1,3-dioxoisindolin-2-yl 2-fluorobenzoate 3c<sup>[2]</sup>**

White solid. mp: 173–175 °C <sup>1</sup>H NMR (400 MHz; CDCl<sub>3</sub>): δ = 7.22–7.34 (m, 2H), 7.67–7.69 (m, 1H), 7.81 (dd, J<sub>1</sub> = 3.2 Hz, J<sub>2</sub> = 5.6 Hz, 2H), 7.92 (dd, J<sub>1</sub> = 3.2 Hz, J<sub>2</sub> = 5.6 Hz, 2H), 8.12–8.15 (m, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 113.9, 114.0, 117.3, 117.5, 124.0, 124.4, 124.5, 129.0, 132.7, 134.8, 136.7, 136.8, 160.2, 160.3, 161.2, 161.8, 163.8. HRMS (ESI-TOF) Calcd for C<sub>15</sub>H<sub>9</sub>FNO<sub>4</sub>, [M+H]<sup>+</sup> m/z 286.0516; Found 286.0531.



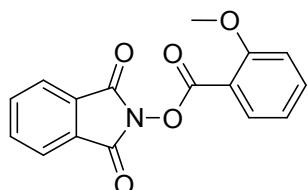
**1,3-dioxoisindolin-2-yl 2-chlorobenzoate 3d<sup>[1]</sup>**

White solid. mp: 134–135 °C <sup>1</sup>H NMR (400 MHz; CDCl<sub>3</sub>): δ = 7.41–7.45 (m, 1H), 7.57 (d, J = 5.6 Hz, 2H), 7.81–7.83 (m, 2H), 7.93 (dd, J<sub>1</sub> = 3.2 Hz, J<sub>2</sub> = 5.6 Hz, 2H), 8.18 (d, J = 8.0 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 124.0, 124.8, 126.9, 129.0, 131.6, 132.4, 134.6, 134.8, 135.6, 161.2, 161.9. HRMS (ESI-TOF) Calcd for C<sub>15</sub>H<sub>9</sub>ClNO<sub>4</sub>, [M+H]<sup>+</sup> m/z 302.0220; Found 302.0224.



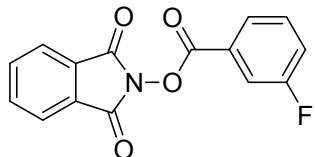
**1,3-dioxoisindolin-2-yl 2-methylbenzoate 3e**

White solid. mp: 139–140 °C <sup>1</sup>H NMR (400 MHz; CDCl<sub>3</sub>): δ = 2.64 (s, 3H), 7.33–7.37 (m, 2H), 7.52–7.56 (m, 1H), 7.80–7.82 (m, 2H), 7.93 (dd, J<sub>1</sub> = 3.2 Hz, J<sub>2</sub> = 5.6 Hz, 2H), 8.19–8.21 (m, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 21.6, 124.0, 124.4, 126.1, 129.1, 131.4, 132.0, 133.9, 134.7, 142.1, 162.2, 163.1. HRMS (ESI-TOF) Calcd for C<sub>16</sub>H<sub>12</sub>NO<sub>4</sub>, [M+H]<sup>+</sup> m/z 282.0766; Found 282.0761.



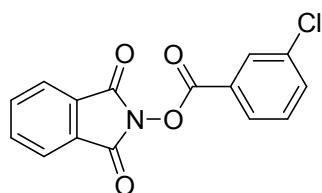
**1,3-dioxoisindolin-2-yl 2-methoxybenzoate 3f**

White solid. mp: 159–160 °C <sup>1</sup>H NMR (400 MHz; CDCl<sub>3</sub>): δ = 3.93 (s, 3H), 7.03–7.07 (m, 2H), 7.59–7.63 (m, 1H), 7.79 (dd, J<sub>1</sub> = 3.2 Hz, J<sub>2</sub> = 5.6 Hz, 2H), 7.91 (dd, J<sub>1</sub> = 3.2 Hz, J<sub>2</sub> = 5.6 Hz, 2H), 8.11 (dd, J<sub>1</sub> = 1.6 Hz, J<sub>2</sub> = 8.0 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 56.1, 112.2, 114.2, 120.3, 123.9, 129.1, 132.7, 134.6, 135.9, 160.6, 161.3, 162.3. HRMS (ESI-TOF) Calcd for C<sub>16</sub>H<sub>12</sub>NO<sub>5</sub>, [M+H]<sup>+</sup> m/z 298.0715; Found 298.0726.



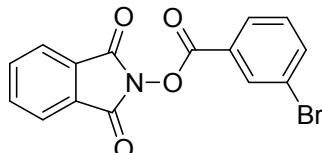
**1,3-dioxoisindolin-2-yl 3-fluorobenzoate 3g<sup>[2]</sup>**

White solid. mp: 162–163 °C <sup>1</sup>H NMR (400 MHz; CDCl<sub>3</sub>): δ = 7.37–7.43 (m, 1H), 7.50–7.56 (m, 1H), 7.81 (dd, J<sub>1</sub> = 3.2 Hz, J<sub>2</sub> = 5.6 Hz, 2H), 7.86 (d, J = 8.0 Hz, 1H), 7.92 (dd, J<sub>1</sub> = 3.2 Hz, J<sub>2</sub> = 5.6 Hz, 2H), 7.99 (d, J = 7.6 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 117.3, 117.6, 122.0, 122.2, 124.0, 126.4, 126.4, 127.2, 127.3, 128.9, 130.6, 130.7, 134.8, 161.2, 161.8, 163.7. HRMS (ESI-TOF) Calcd for C<sub>15</sub>H<sub>9</sub>FNO<sub>4</sub>, [M+H]<sup>+</sup> m/z 286.0516; Found 286.0531.



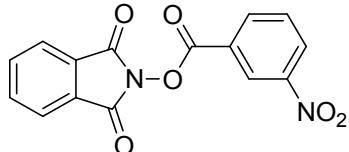
**1,3-dioxoisindolin-2-yl 3-chlorobenzoate 3h<sup>[1]</sup>**

White solid. mp: 176–178 °C <sup>1</sup>H NMR (400 MHz; CDCl<sub>3</sub>): δ = 7.49 (t, J = 8.0 Hz 1H), 7.67 (d, J = 8.0 Hz, 1H), 7.81 (dd, J<sub>1</sub> = 3.2 Hz, J<sub>2</sub> = 5.2 Hz, 2H), 7.92 (dd, J<sub>1</sub> = 3.2 Hz, J<sub>2</sub> = 5.2 Hz, 2H), 8.07 (d, J = 8.0 Hz, 1H) 8.15 (s, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 124.0, 126.9, 128.7, 128.8, 130.2, 130.5, 134.9, 134.9, 135.1, 161.7, 161.8. HRMS (ESI-TOF) Calcd for C<sub>15</sub>H<sub>9</sub>ClNO<sub>4</sub>, [M+H]<sup>+</sup> m/z 302.0220.; Found 302.0204.



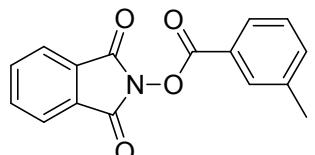
**1,3-dioxoisindolin-2-yl 3-bromobenzoate 3i<sup>[2]</sup>**

White solid. mp: 209–210 °C <sup>1</sup>H NMR (400 MHz; CDCl<sub>3</sub>): δ = 7.42 (t, J = 8.0 Hz 1H), 7.80–7.82 (m, 3H), 7.90–7.92 (m, 2H), 8.11 (d, J = 7.6 Hz, 1H), 8.30 (s, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 122.8, 124.0, 127.1, 128.8, 129.1, 130.4, 133.3, 134.8, 137.8, 161.6, 161.8. HRMS (ESI-TOF) Calcd for C<sub>15</sub>H<sub>9</sub>BrNO<sub>4</sub>, [M+H]<sup>+</sup> m/z 345.9715.; Found 345.9731.



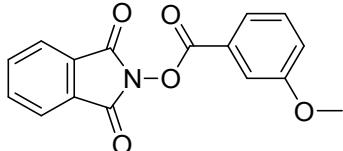
**1,3-dioxoisindolin-2-yl 3-nitrobenzoate 3j**

White solid. mp: 214–215 °C <sup>1</sup>H NMR (400 MHz; CDCl<sub>3</sub>): δ = 7.79 (t, J = 8.0 Hz 1H), 7.85 (dd, J<sub>1</sub> = 3.2 Hz, J<sub>2</sub> = 5.6 Hz, 2H), 7.96 (dd, J<sub>1</sub> = 3.2 Hz, J<sub>2</sub> = 5.2 Hz, 2H), 8.51–8.53 (m, 1H), 8.56–8.58 (m, 1H), 9.04–9.05 (m, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 124.2, 125.6, 127.2, 128.9, 129.2, 130.3, 135.0, 136.0, 148.5, 161.1, 161.6. HRMS (ESI-TOF) Calcd for C<sub>15</sub>H<sub>9</sub>N<sub>2</sub>O<sub>6</sub>, [M+H]<sup>+</sup> m/z 313.0461.; Found 313.0444.



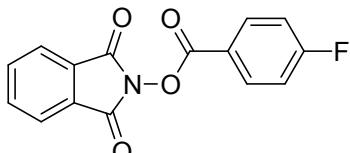
**1,3-dioxoisooindolin-2-yl 3-methylbenzoate 3k**

White solid. mp: 141–142 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 2.44 (s, 3H), 7.42 (t,  $J$  = 7.6 Hz, 1H), 7.50 (d,  $J$  = 7.6 Hz, 1H), 7.81 (dd,  $J_1$  = 3.2 Hz,  $J_2$  = 5.6 Hz, 2H), 7.92 (dd,  $J_1$  = 3.2 Hz,  $J_2$  = 5.6 Hz, 2H), 8.00 (d,  $J$  = 8.8 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 21.2, 124.0, 125.2, 127.8, 128.7, 129.1, 131.0, 134.7, 135.6, 138.8, 162.1, 162.9. HRMS (ESI-TOF) Calcd for  $\text{C}_{16}\text{H}_{12}\text{NO}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  282.0766; Found 282.0771.



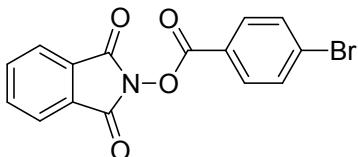
**1,3-dioxoisooindolin-2-yl 3-methoxybenzoate 3l**

White solid. mp: 132–134 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 3.87 (s, 3H), 7.21–7.24 (m, 1H), 7.43 (t,  $J$  = 8.0 Hz 1H), 7.65–7.66 (m, 1H), 7.78–7.82 (m, 3H), 7.90–7.92 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 55.5, 114.7, 121.6, 123.0, 124.0, 126.3, 129.0, 129.9, 134.8, 159.7, 162.0, 162.7. HRMS (ESI-TOF) Calcd for  $\text{C}_{16}\text{H}_{12}\text{NO}_5$ ,  $[\text{M}+\text{H}]^+$   $m/z$  298.0715; Found 298.0721.



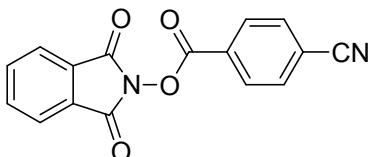
**1,3-dioxoisooindolin-2-yl 4-fluorobenzoate 3m<sup>[2]</sup>**

White solid. mp: 194–195 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 7.19–7.25 (m, 2H), 7.79–7.82 (m, 2H), 7.90–7.93 (m, 2H), 8.20–8.24 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 116.2, 116.4, 121.5, 124.0, 129.0, 133.4, 133.5, 134.8, 161.8, 162.0, 165.6, 168.1. HRMS (ESI-TOF) Calcd for  $\text{C}_{15}\text{H}_9\text{FNO}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  286.0516; Found 286.0513.



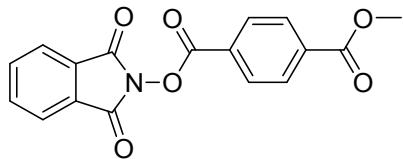
**1,3-dioxoisooindolin-2-yl 4-bromobenzoate 3n<sup>[2]</sup>**

White solid. mp: 190–192 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 7.68 (dd,  $J_1$  = 1.6 Hz,  $J_2$  = 6.8 Hz, 2H), 7.80–7.82 (m, 2H), 7.92 (dd,  $J_1$  = 3.2 Hz,  $J_2$  = 5.6 Hz, 2H), 8.04 (dd,  $J_1$  = 2.0 Hz,  $J_2$  = 6.8 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 124.0, 124.1, 128.9, 130.4, 131.9, 132.3, 134.8, 161.9, 162.0. HRMS (ESI-TOF) Calcd for  $\text{C}_{15}\text{H}_9\text{BrNO}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  345.9715; Found 345.9725.



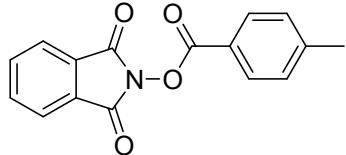
**1,3-dioxoisooindolin-2-yl 4-cyanobenzoate 3o**

White solid. mp: 219–221 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 7.82–7.86 (m, 4H), 7.94 (dd,  $J_1$  = 3.2 Hz,  $J_2$  = 5.6 Hz, 2H), 8.30 (d,  $J$  = 8.4 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 117.4, 118.2, 124.2, 128.8, 129.2, 131.0, 132.6, 135.0, 161.5, 161.6. HRMS (ESI-TOF) Calcd for  $\text{C}_{16}\text{H}_9\text{N}_2\text{O}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  293.0562; Found 293.0552.



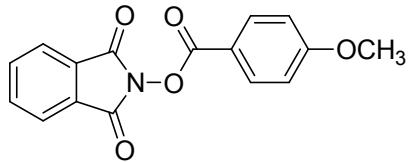
**1,3-dioxoisindolin-2-yl methyl terephthalate 3p**

White solid. mp: 177–178 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 3.95 (s, 3H), 7.80 (d,  $J$  = 2.8 Hz, 2H), 7.89 (d,  $J$  = 2.8 Hz, 2H), 8.16 (d,  $J$  = 8.4 Hz, 2H), 8.23 (d,  $J$  = 8.4 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 52.5, 124.0, 128.8, 128.9, 129.8, 130.5, 134.8, 135.5, 161.7, 162.1, 165.7. HRMS (ESI-TOF) Calcd for  $\text{C}_{17}\text{H}_{12}\text{NO}_6$ ,  $[\text{M}+\text{H}]^+$   $m/z$  326.0665; Found 326.0657.



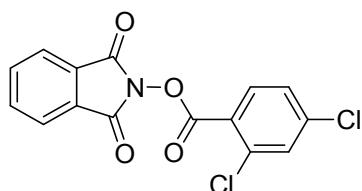
**1,3-dioxoisindolin-2-yl 4-methylbenzoate 3q<sup>[1]</sup>**

White solid. mp: 167–168 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 2.47 (s, 3H), 7.33 (d,  $J$  = 8.0 Hz, 2H), 7.81 (dd,  $J_1$  = 3.2 Hz,  $J_2$  = 5.2 Hz, 2H), 7.92–7.94 (m, 2H), 8.08 (d,  $J$  = 8.0 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 21.9, 122.5, 124.0, 129.1, 129.6, 130.7, 134.7, 146.0, 162.1, 162.8. HRMS (ESI-TOF) Calcd for  $\text{C}_{16}\text{H}_{12}\text{NO}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  282.0766; Found 282.0772.



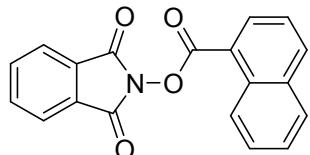
**1,3-dioxoisindolin-2-yl 4-methoxybenzoate 3r<sup>[1]</sup>**

White solid. mp: 165–166 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 3.89 (s, 3H), 6.99 (d,  $J$  = 8.8 Hz, 2H), 7.79–7.81 (m, 2H), 7.90–7.91 (m, 2H), 8.14 (d,  $J$  = 8.8 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 55.6, 114.2, 117.2, 123.9, 129.0, 132.9, 134.7, 162.2, 162.4, 164.9. HRMS (ESI-TOF) Calcd for  $\text{C}_{16}\text{H}_{12}\text{NO}_5$ ,  $[\text{M}+\text{H}]^+$   $m/z$  298.0715; Found 298.0721.



**1,3-dioxoisindolin-2-yl 2,4-dichlorobenzoate 3s**

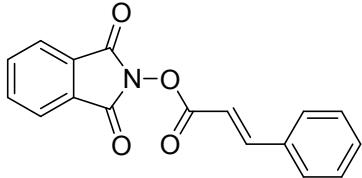
White solid. mp: 180–182 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 7.42 (d,  $J$  = 8.4 Hz, 1H), 7.58 (s, 1H), 7.82 (d,  $J$  = 3.2 Hz, 2H), 7.92 (dd,  $J_1$  = 3.2 Hz,  $J_2$  = 4.8 Hz, 2H), 8.13 (d,  $J$  = 8.8 Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 123.1, 124.1, 127.4, 128.9, 131.7, 133.4, 134.9, 136.8, 140.8, 160.4, 161.8. HRMS (ESI-TOF) Calcd for  $\text{C}_{15}\text{H}_8\text{Cl}_2\text{NO}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  335.9830; Found 335.9835.



**1,3-dioxoisindolin-2-yl 1-naphthoate 3t<sup>[1]</sup>**

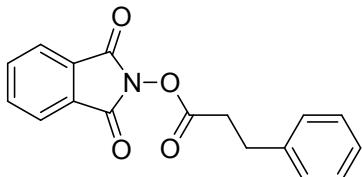
White solid. mp: 180–182 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 7.57–7.62 (m, 2H), 7.64–7.69 (m,

1H), 7.81-7.83 (m, 2H), 7.92-7.96 (m, 3H), 8.16 (d,  $J = 8.0$  Hz, 1H), 8.54 (dd,  $J_1 = 1.2$  Hz,  $J_2 = 7.2$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 121.7, 124.0, 124.5, 125.3, 126.8, 128.7, 128.9, 129.1, 131.5, 131.9, 133.7, 134.8, 135.5$ ; HRMS (ESI-TOF) Calcd for  $\text{C}_{19}\text{H}_{12}\text{NO}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  318.0766; Found 318.0760.



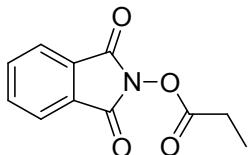
### 1,3-dioxoisindolin-2-yl cinnamate 3u<sup>[2]</sup>

White solid. mp: 56–57 °C.  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta = 6.67$  (d,  $J = 16.0$  Hz, 1H), 7.43-7.47 (m, 3H), 7.59-7.61 (m, 2H), 7.80-7.82 (m, 2H), 7.92 (dd,  $J_1 = 3.2$  Hz,  $J_2 = 5.2$  Hz, 2H), 7.97 (d,  $J = 16.0$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 111.8, 124.0, 128.7, 129.1, 129.1, 131.5, 133.6, 134.7, 150.0, 162.1, 163.0$ . HRMS (ESI-TOF) Calcd for  $\text{C}_{17}\text{H}_{12}\text{NO}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  294.0766; Found 294.0754.



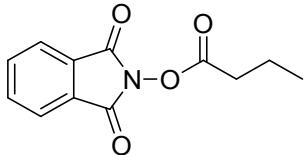
### 1,3-dioxoisindolin-2-yl 3-phenylpropanoate 3v

White solid. mp: 66–67 °C.  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta = 3.00-3.02$  (m, 2H), 3.10-3.14 (m, 2H), 7.27-7.37 (m, 5H), 7.79-7.81 (m, 2H), 7.90 (dd,  $J_1 = 3.2$  Hz,  $J_2 = 5.2$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 30.5, 32.7, 123.9, 126.7, 128.3, 128.7, 128.9, 134.7, 139.1, 161.8, 168.8$ . HRMS (ESI-TOF) Calcd for  $\text{C}_{17}\text{H}_{14}\text{NO}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  296.0923; Found 296.0917.



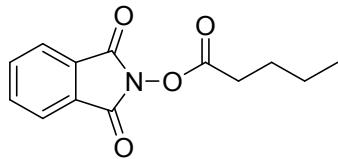
### 1,3-dioxoisindolin-2-yl propionate 3w

White solid. mp: 86–88 °C.  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta = 1.31$  (t,  $J = 7.6$  Hz, 3H), 2.71 (q,  $J = 7.6$  Hz, 2H), 7.79 (dd,  $J_1 = 3.2$  Hz,  $J_2 = 5.6$  Hz, 2H), 7.89 (dd,  $J_1 = 3.2$  Hz,  $J_2 = 5.6$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 8.7, 24.5, 123.9, 129.0, 134.7, 162.0, 170.3$ . HRMS (ESI-TOF) Calcd for  $\text{C}_{11}\text{H}_{10}\text{NO}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  220.0610; Found 220.0608.



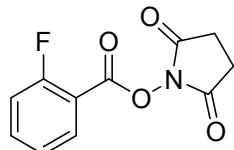
### 1,3-dioxoisindolin-2-yl butyrate 3x

Colorless oil.  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta = 1.08$  (t,  $J = 7.2$  Hz, 3H), 1.78-1.88 (m, 2H), 2.65 (t,  $J = 7.2$  Hz, 2H), 7.79 (dd,  $J_1 = 3.2$  Hz,  $J_2 = 5.6$  Hz, 2H), 7.89 (dd,  $J_1 = 3.2$  Hz,  $J_2 = 5.2$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta = 13.4, 18.3, 32.8, 123.9, 129.0, 134.7, 162.0, 169.5$ . HRMS (ESI-TOF) Calcd for  $\text{C}_{12}\text{H}_{12}\text{NO}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  234.0766; Found 234.0761.



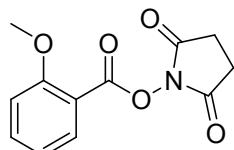
**1,3-dioxoisooindolin-2-yl pentanoate 3y**

White solid. mp: 55–56 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 0.97 (t,  $J$  = 7.2 Hz, 3H), 1.45–1.50 (m, 2H), 1.73–1.81 (m, 2H), 2.67 (t,  $J$  = 7.2 Hz, 2H), 7.77–7.92 (m, 2H), 7.87–7.89 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 13.6, 22.0, 26.7, 30.7, 123.9, 129.0, 134.7, 162.0, 169.6. HRMS (ESI-TOF) Calcd for  $\text{C}_{13}\text{H}_{14}\text{NO}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  248.0923; Found 248.0919.



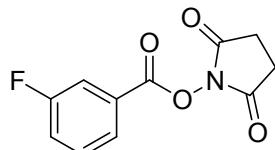
**2,5-dioxopyrrolidin-1-yl 2-fluorobenzoate 5a**

White solid. mp: 113–114 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 2.90 (s, 4H), 7.19–7.30 (m, 2H), 7.65–7.67 (m, 1H), 8.04–8.08 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 25.6, 113.6, 113.7, 117.2, 117.4, 124.4, 124.4, 132.5, 136.7, 136.8, 159.2, 159.3, 161.1, 163.7, 169.1. HRMS (ESI-TOF) Calcd for  $\text{C}_{11}\text{H}_9\text{FNO}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  238.0516; Found 238.0508.



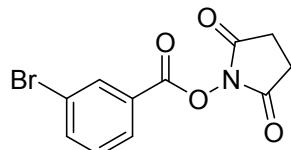
**2,5-dioxopyrrolidin-1-yl 2-methoxybenzoate 5b**

White solid. mp: 177–178 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 2.88 (s, 4H), 3.92 (s, 3H), 7.01–7.04 (m, 2H), 7.57–7.61 (m, 1H), 8.03–8.05 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 25.7, 56.1, 112.2, 114.1, 120.3, 132.7, 135.9, 160.3, 160.6, 169.4. HRMS (ESI-TOF) Calcd for  $\text{C}_{12}\text{H}_{12}\text{NO}_5$ ,  $[\text{M}+\text{H}]^+$   $m/z$  250.0715; Found 250.0721.



**2,5-dioxopyrrolidin-1-yl 3-fluorobenzoate 5c**

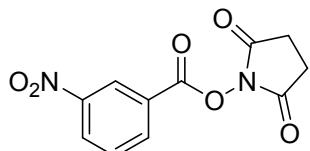
White solid. mp: 147–148 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 2.91 (s, 4H), 7.37–7.41 (m, 1H), 7.48–7.54 (m, 1H), 7.81–7.83 (m, 1H), 7.94 (d,  $J$  = 7.6 Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 25.7, 117.3, 117.6, 122.0, 122.2, 126.4, 126.4, 127.1, 127.2, 130.6, 130.7, 160.9, 160.9, 161.3, 163.7, 169.0. HRMS (ESI-TOF) Calcd for  $\text{C}_{11}\text{H}_9\text{FNO}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  238.0516; Found 238.0510.



**2,5-dioxopyrrolidin-1-yl 3-bromobenzoate 5d<sup>[2]</sup>**

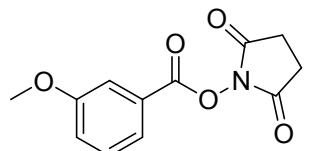
White solid. mp: 152–153 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 2.91 (s, 4H), 7.38–7.42 (m, 1H), 7.80–7.82 (m, 1H), 8.07 (d,  $J$  = 8.0 Hz, 1H), 8.27 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 25.7, 122.9, 127.1, 129.1, 130.4, 133.4, 137.9, 160.7, 168.9. HRMS (ESI-TOF) Calcd for  $\text{C}_{11}\text{H}_9\text{BrNO}_4$ ,

$[M+H]^+$   $m/z$  297.9715; Found 297.9711.



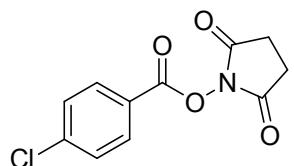
**2,5-dioxopyrrolidin-1-yl 3-nitrobenzoate 5e**

White solid. mp: 137–138 °C  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 2.96 (s, 4H), 7.76–7.80 (m, 1H), 8.47 (d,  $J$  = 7.6 Hz, 1H), 8.56 (d,  $J$  = 8.0 Hz, 1H), 8.99 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 25.7, 125.5, 127.1, 129.2, 130.3, 135.9, 148.4, 160.1, 168.7. HRMS (ESI-TOF) Calcd for  $\text{C}_{11}\text{H}_9\text{N}_2\text{O}_6$ ,  $[M+H]^+$   $m/z$  265.0461; Found 265.0455.



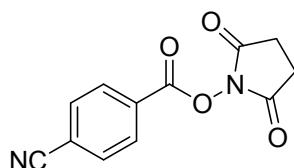
**2,5-dioxopyrrolidin-1-yl 3-methoxybenzoate 5f**

White solid. mp: 106–107 °C  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 2.89 (s, 4H), 3.85 (s, 3H), 7.19–7.22 (m, 1H), 7.41 (t,  $J$  = 8.0 Hz, 1H), 7.60 (s, 1H), 7.73 (d,  $J$  = 7.6 Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 25.6, 55.5, 114.6, 121.6, 123.0, 126.2, 129.9, 159.7, 161.8, 169.2. HRMS (ESI-TOF) Calcd for  $\text{C}_{12}\text{H}_{12}\text{NO}_5$ ,  $[M+H]^+$   $m/z$  250.0715; Found 250.0711.



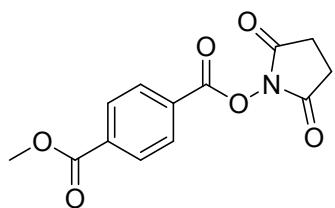
**2,5-dioxopyrrolidin-1-yl 4-chlorobenzoate 5g**

White solid. mp: 207–208 °C  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 2.90 (s, 4H), 7.49 (d,  $J$  = 8.4 Hz, 2H), 8.06 (d,  $J$  = 8.8 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 25.6, 123.6, 129.3, 131.9, 141.7, 161.1, 169.1. HRMS (ESI-TOF) Calcd for  $\text{C}_{11}\text{H}_9\text{ClNO}_4$ ,  $[M+H]^+$   $m/z$  254.0220; Found 254.0225.



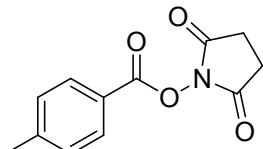
**2,5-dioxopyrrolidin-1-yl 4-cyanobenzoate 5h**

White solid. mp: 223–224 °C  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 2.92 (s, 4H), 7.82 (d,  $J$  = 8.0 Hz, 2H), 8.23 (d,  $J$  = 8.0 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 25.6, 117.3, 118.3, 129.1, 131.0, 132.6, 160.5, 168.7. HRMS (ESI-TOF) Calcd for  $\text{C}_{12}\text{H}_9\text{N}_2\text{O}_4$ ,  $[M+H]^+$   $m/z$  245.0562; Found 245.0565.



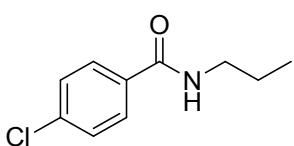
**2,5-dioxopyrrolidin-1-yl methyl terephthalate 5i**

White solid. mp: 170–171 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 2.92 (s, 4H), 3.97 (s, 3H), 8.16–8.22 (m, 4H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 25.7, 52.6, 128.9, 129.9, 130.5, 135.7, 161.2, 165.8, 168.9. HRMS (ESI-TOF) Calcd for  $\text{C}_{13}\text{H}_{12}\text{NO}_6$ ,  $[\text{M}+\text{H}]^+$   $m/z$  278.0665; Found 278.0661.



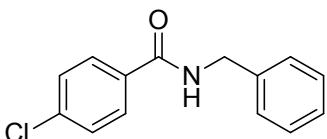
**2,5-dioxopyrrolidin-1-yl 4-methylbenzoate 5j<sup>[2]</sup>**

White solid. mp: 180–181 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 2.42 (s, 3H), 2.87 (s, 4H), 7.29 (d,  $J$  = 8.0 Hz, 2H), 8.00 (d,  $J$  = 8.4 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 21.8, 25.6, 122.2, 129.5, 130.5, 146.0, 161.8, 169.3. HRMS (ESI-TOF) Calcd for  $\text{C}_{12}\text{H}_{12}\text{NO}_4$ ,  $[\text{M}+\text{H}]^+$   $m/z$  234.0766; Found 234.0770.



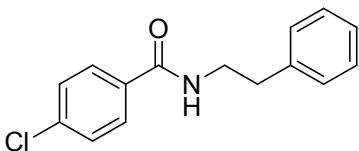
**4-chloro-N-propylbenzamide 7a**

White solid. mp: 98–101 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 0.96 (t,  $J$  = 7.2 Hz, 3H), 1.57–1.66 (m, 2H), 3.38 (q,  $J$  = 6.8 Hz, 2H), 6.38 (s, 1H), 7.36 (d,  $J$  = 8.4 Hz, 2H), 7.69 (d,  $J$  = 8.4 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 11.4, 22.8, 41.8, 128.3, 128.7, 133.2, 137.4, 166.5. HRMS (ESI-TOF) Calcd for  $\text{C}_{10}\text{H}_{13}\text{ClNO}$ ,  $[\text{M}+\text{H}]^+$   $m/z$  198.0686; Found 198.0682.



**N-benzyl-4-chlorobenzamide 7b<sup>[2]</sup>**

White solid. mp: 161–164 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 4.60 (d,  $J$  = 5.6 Hz, 2H), 6.58 (s, 1H), 7.29–7.38 (m, 7H), 7.71 (d,  $J$  = 8.4 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 44.2, 127.7, 127.9, 128.4, 128.8, 132.7, 137.7, 137.9, 166.3. HRMS (ESI-TOF) Calcd for  $\text{C}_{14}\text{H}_{13}\text{ClNO}$ ,  $[\text{M}+\text{H}]^+$   $m/z$  246.0686; Found 246.0684.



**4-chloro-N-phenethylbenzamide 7c<sup>[2]</sup>**

White solid. mp: 129–130 °C  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ):  $\delta$  = 2.92 (t,  $J$  = 6.8 Hz, 2H), 3.67–3.71 (m, 2H), 6.31 (s, 1H), 7.20–7.24 (m, 3H), 7.30–7.37 (m, 4H), 7.62 (d,  $J$  = 8.4 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 35.6, 41.2, 126.6, 128.2, 128.7, 128.7, 133.0, 137.6, 138.7, 166.4. HRMS (ESI-TOF) Calcd for  $\text{C}_{15}\text{H}_{15}\text{ClNO}$ ,  $[\text{M}+\text{H}]^+$   $m/z$  260.0842; Found 260.0839.

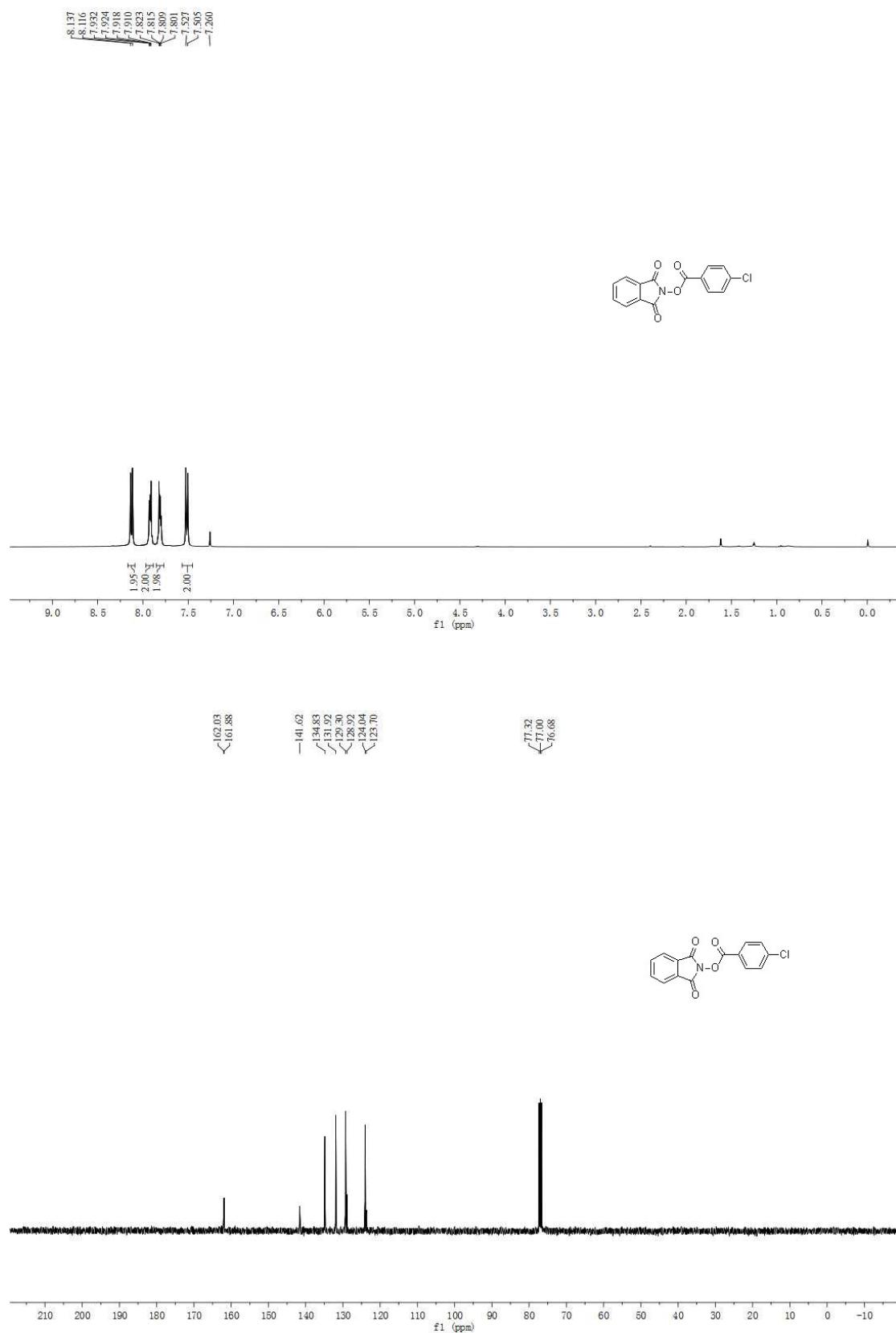
## References

- [1] B. Tan, N. Toda and C. F. Barbas III, *Angew. Chem., Int. Ed.*, 2012, **51**, 12538.

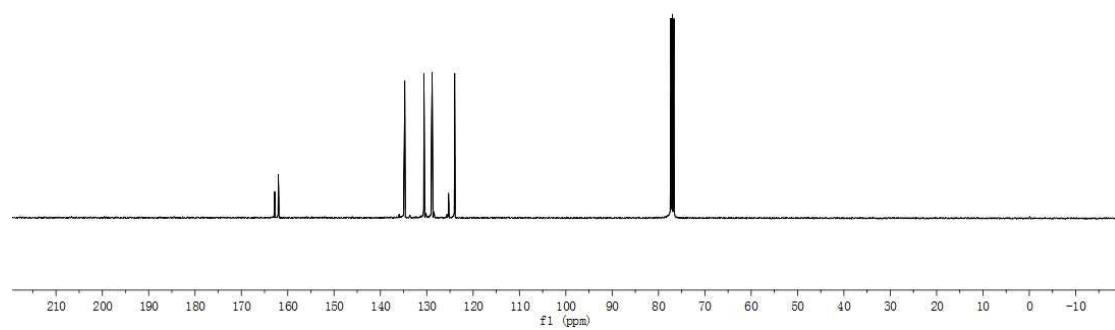
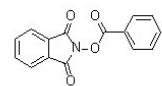
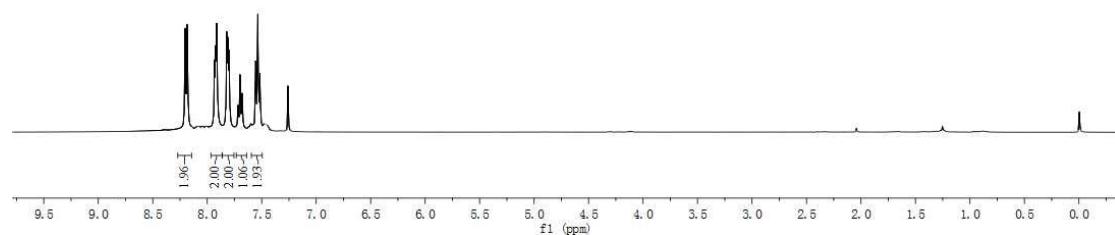
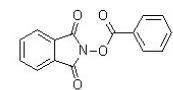
[2] M. Dinda, C. Bose, T. Ghosh and S. Maity, *RSC Adv.*, 2015, **5**, 44928.

## VI. $^1\text{H}$ and $^{13}\text{C}$ Spectra of Compounds 3, 5 and 7

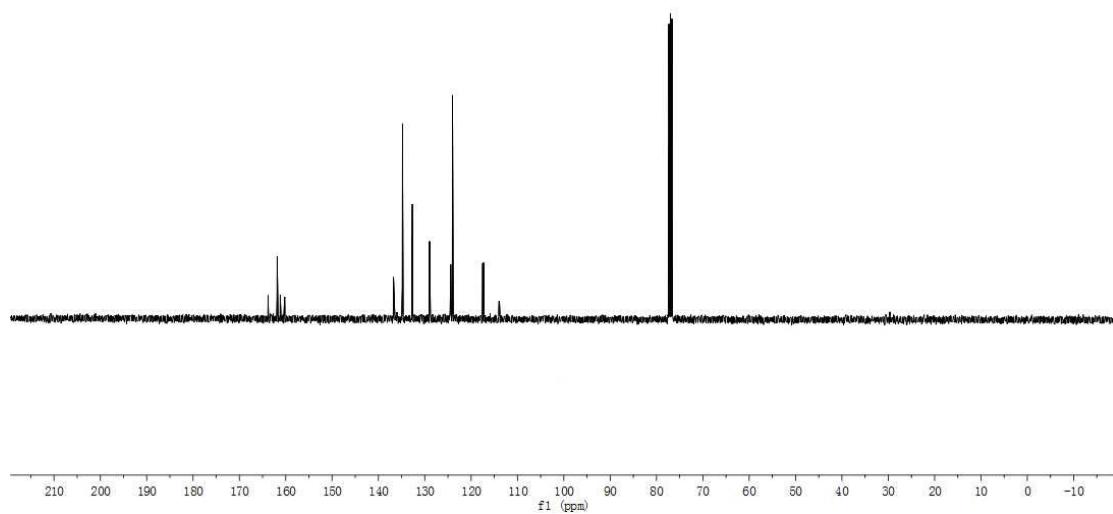
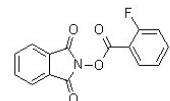
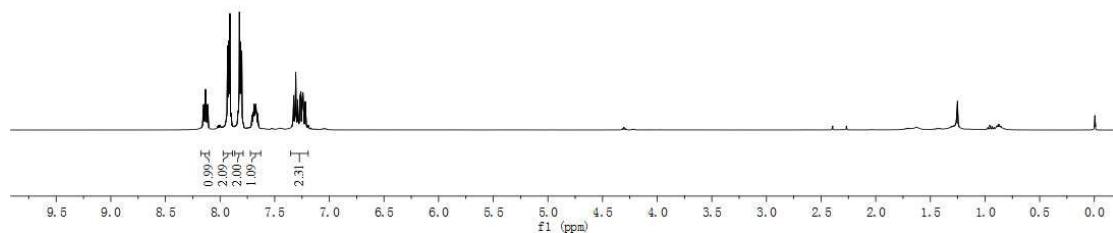
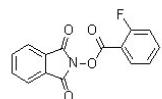
### Product 3a



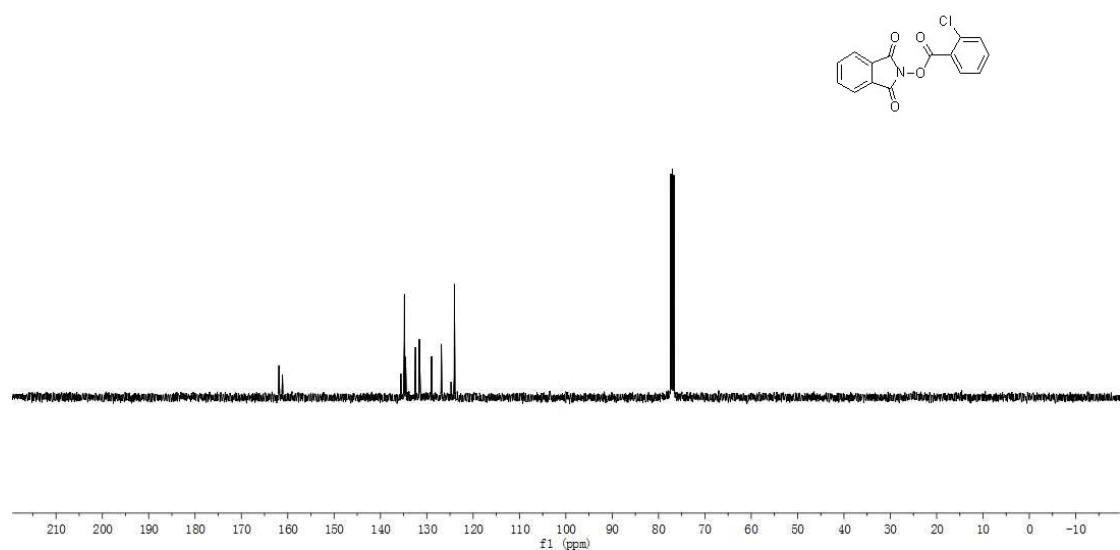
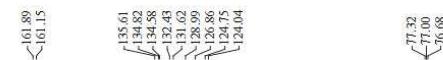
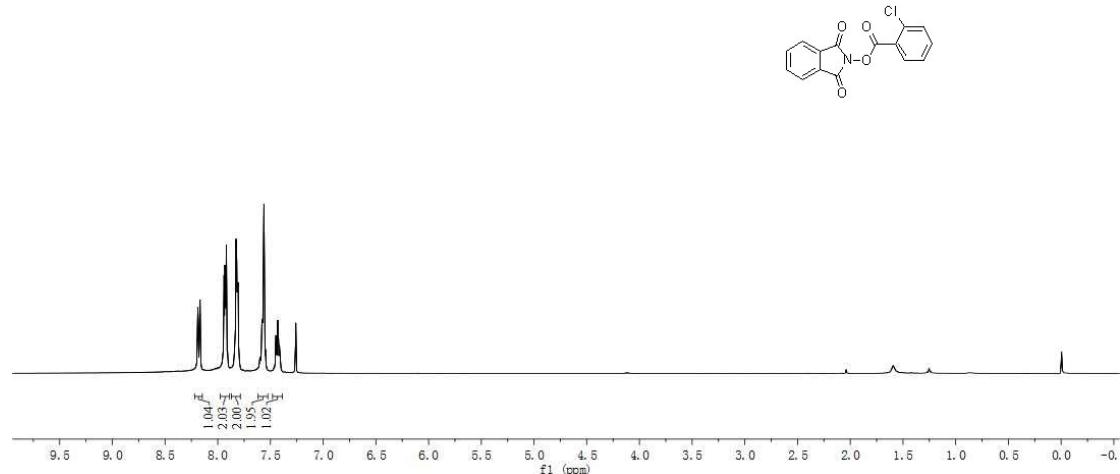
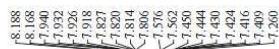
**Product 3b**



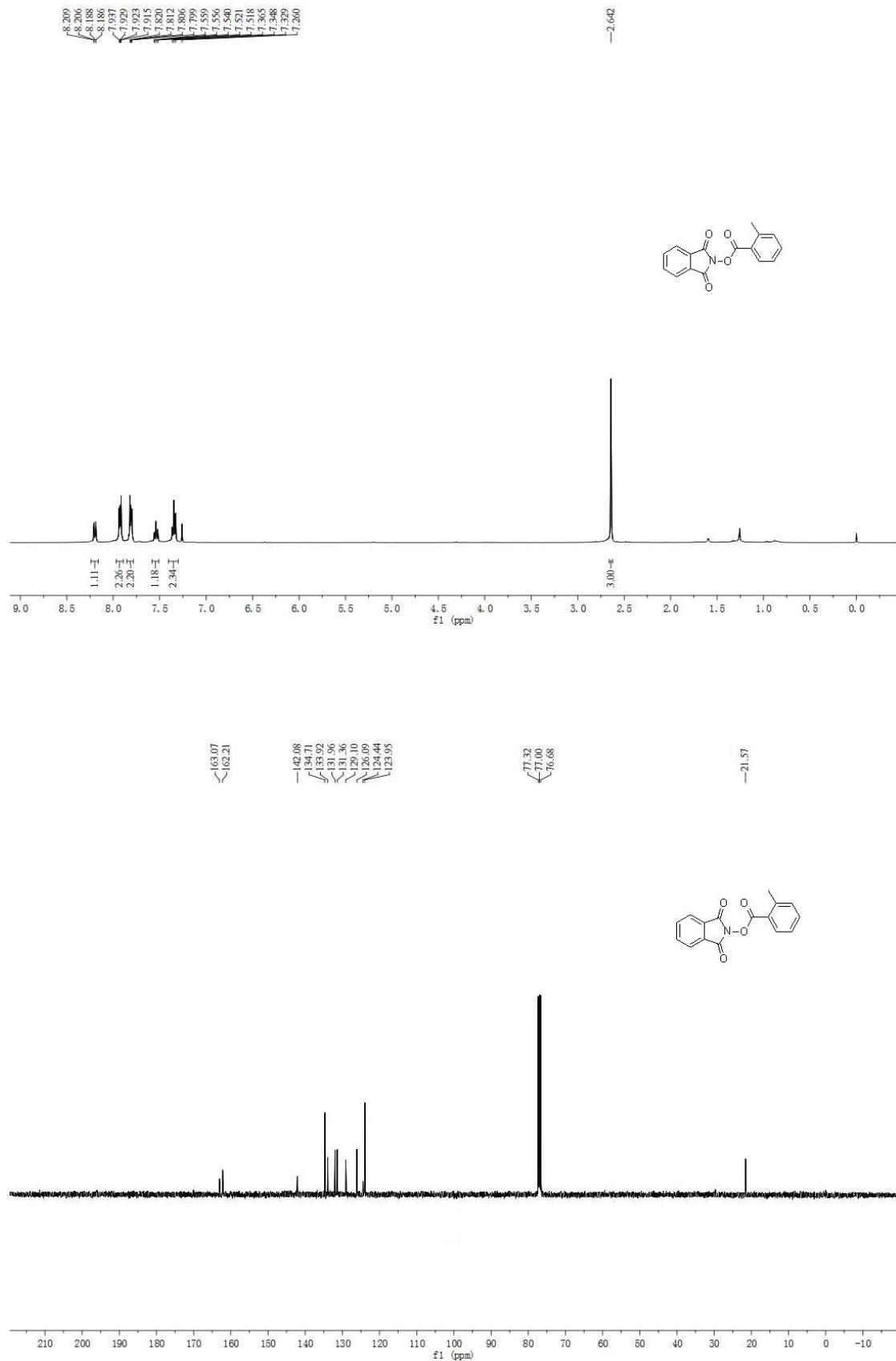
### **Product 3c**



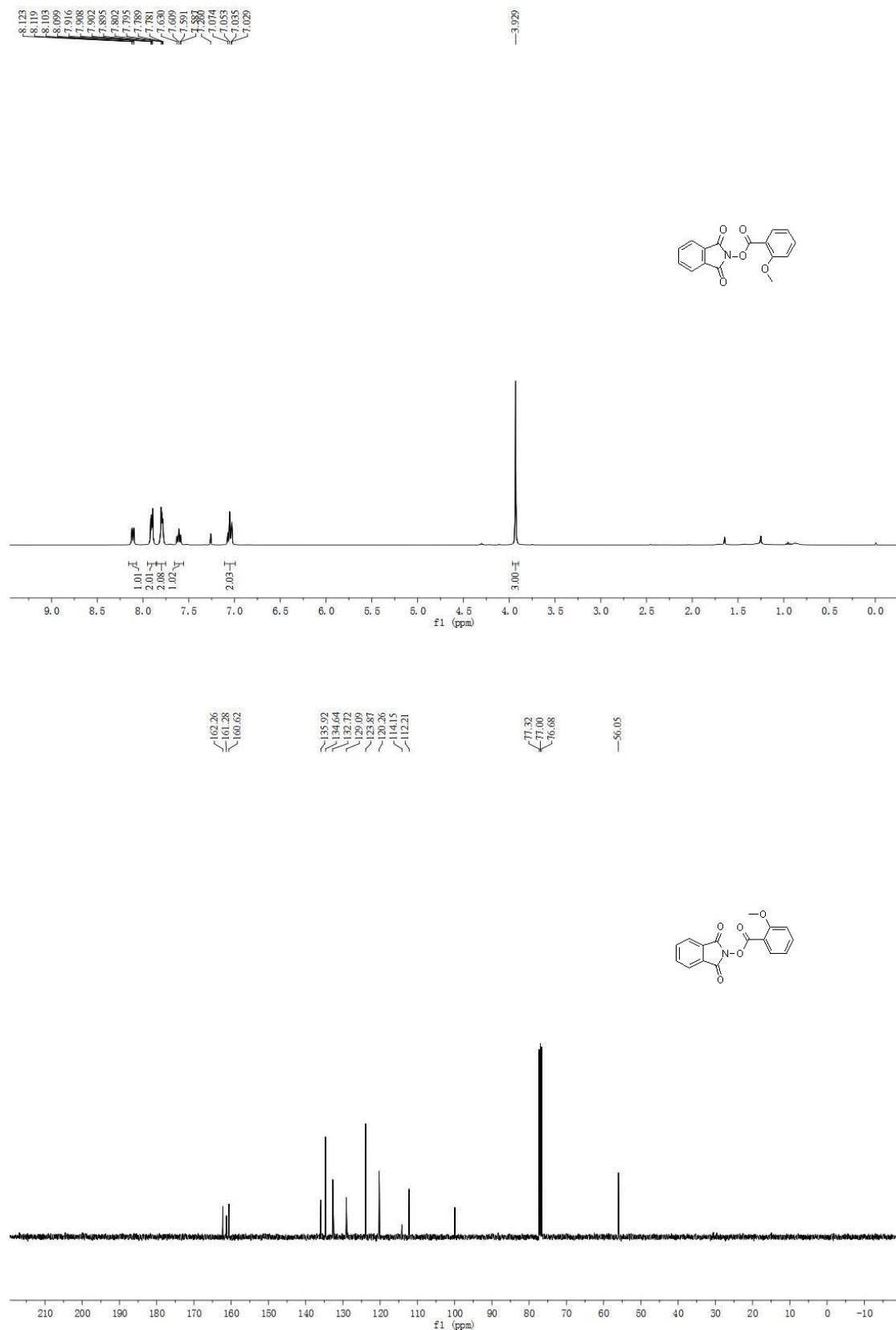
**Product 3d**



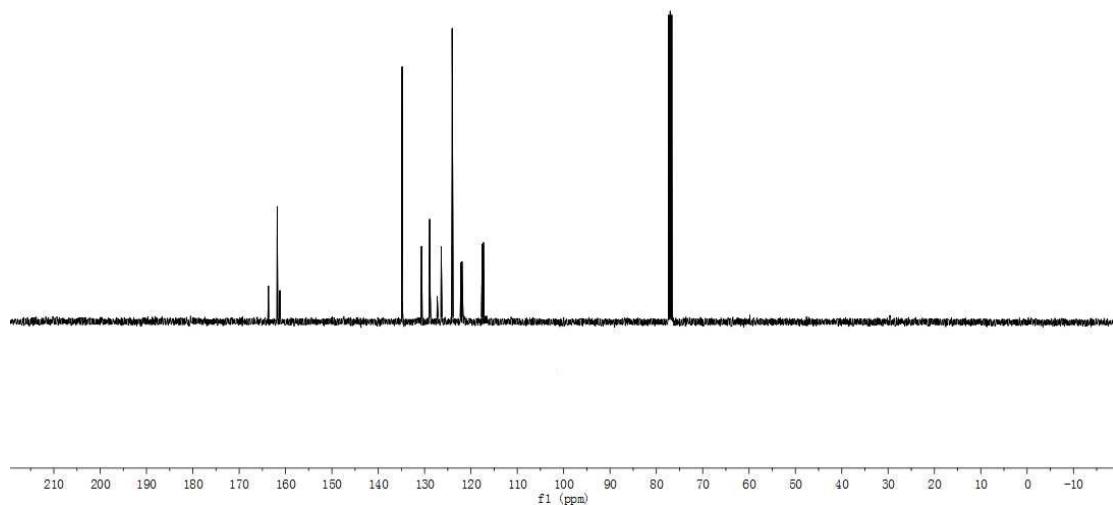
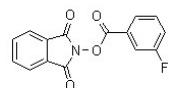
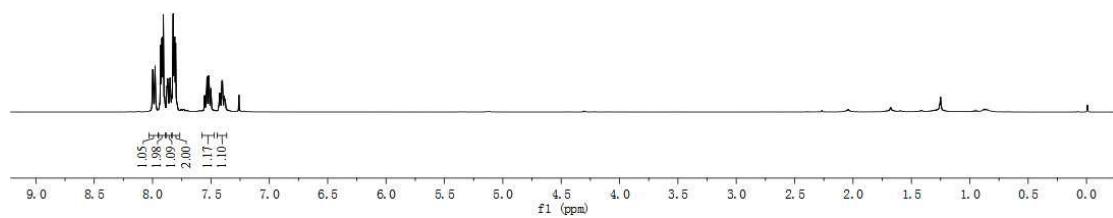
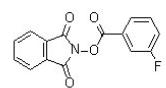
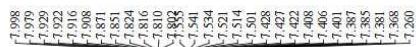
**Product 3e**



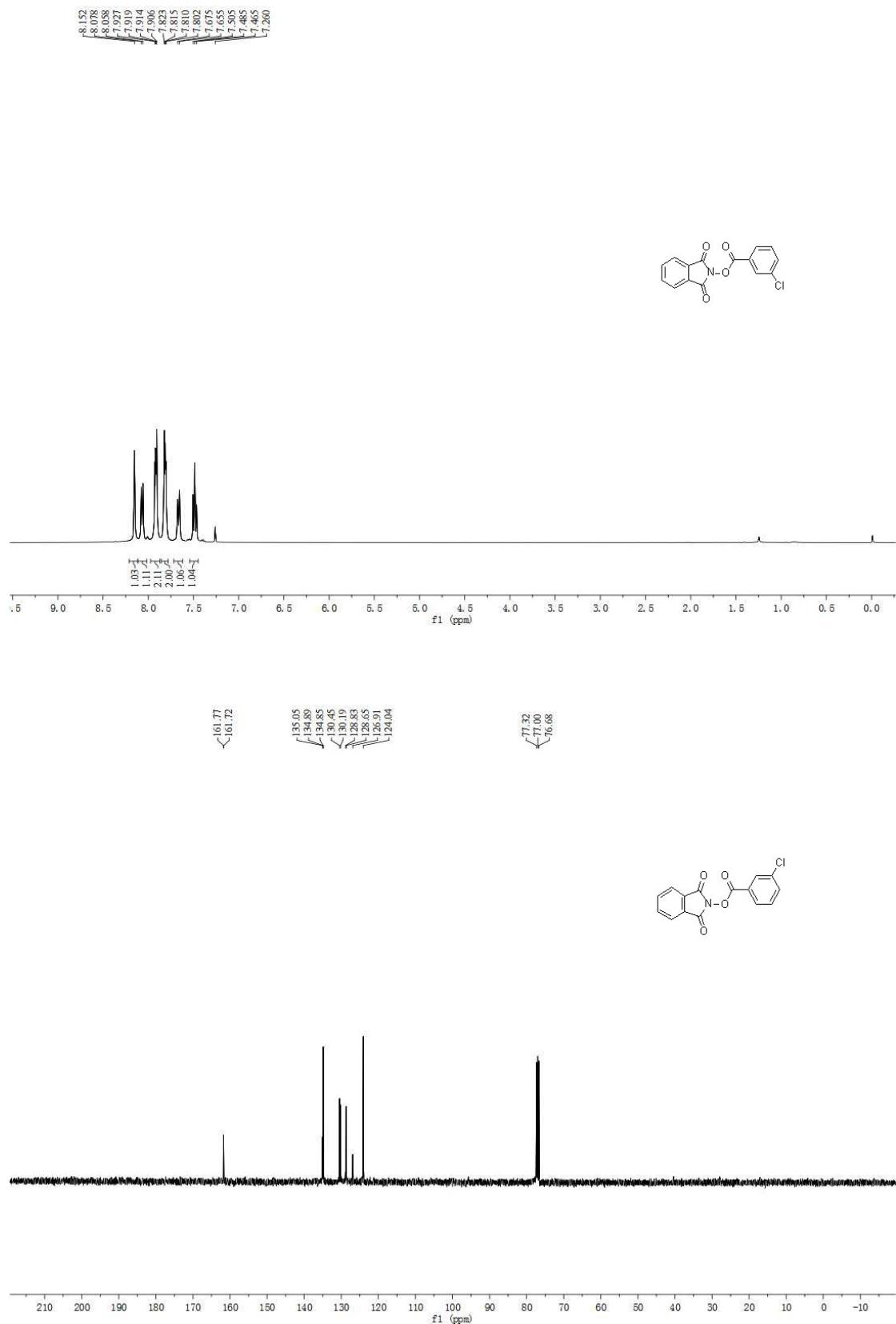
**Product 3f**



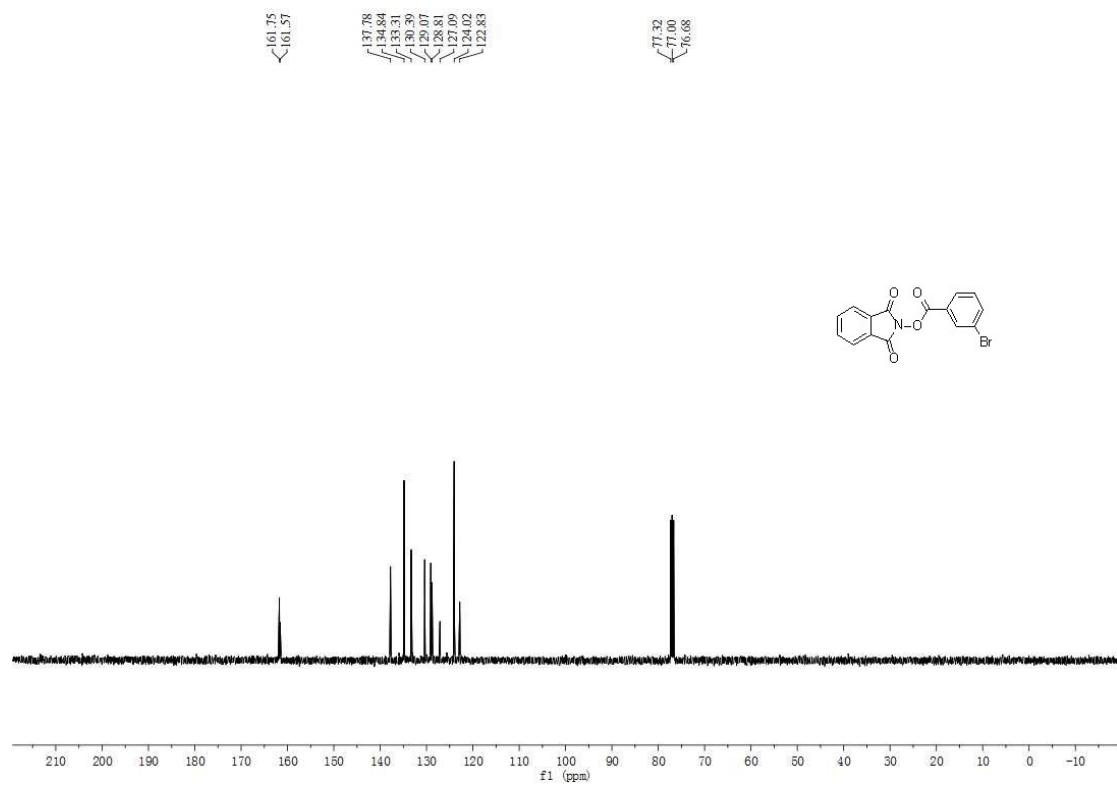
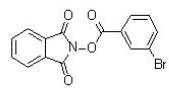
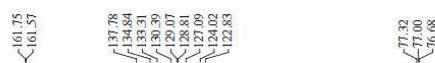
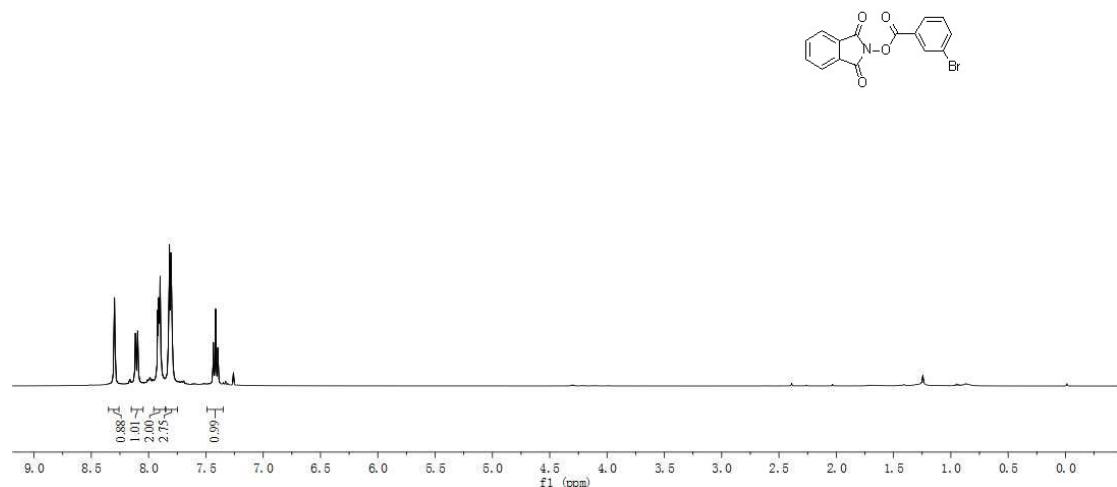
## Product 3g



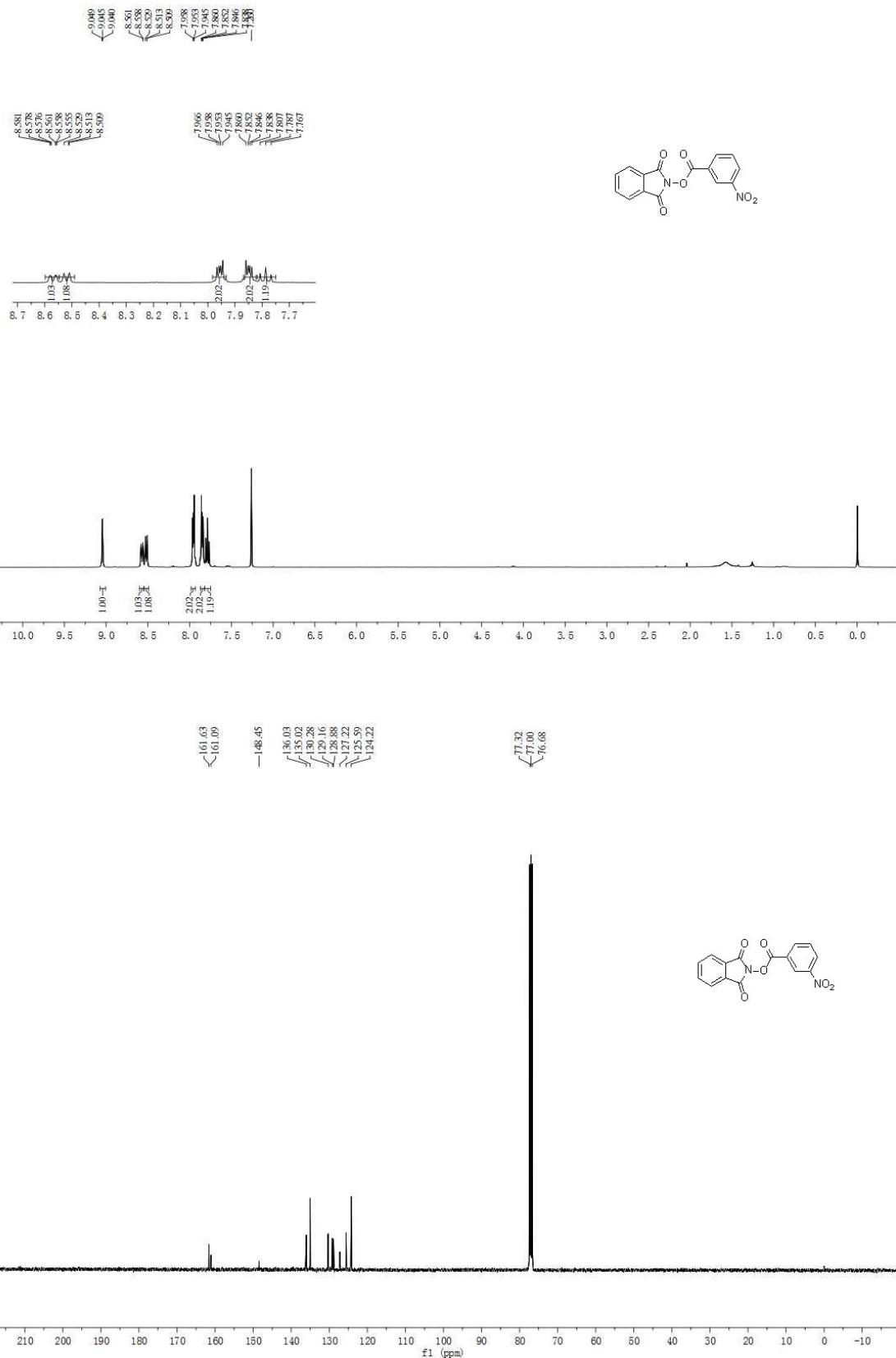
**Product 3h**



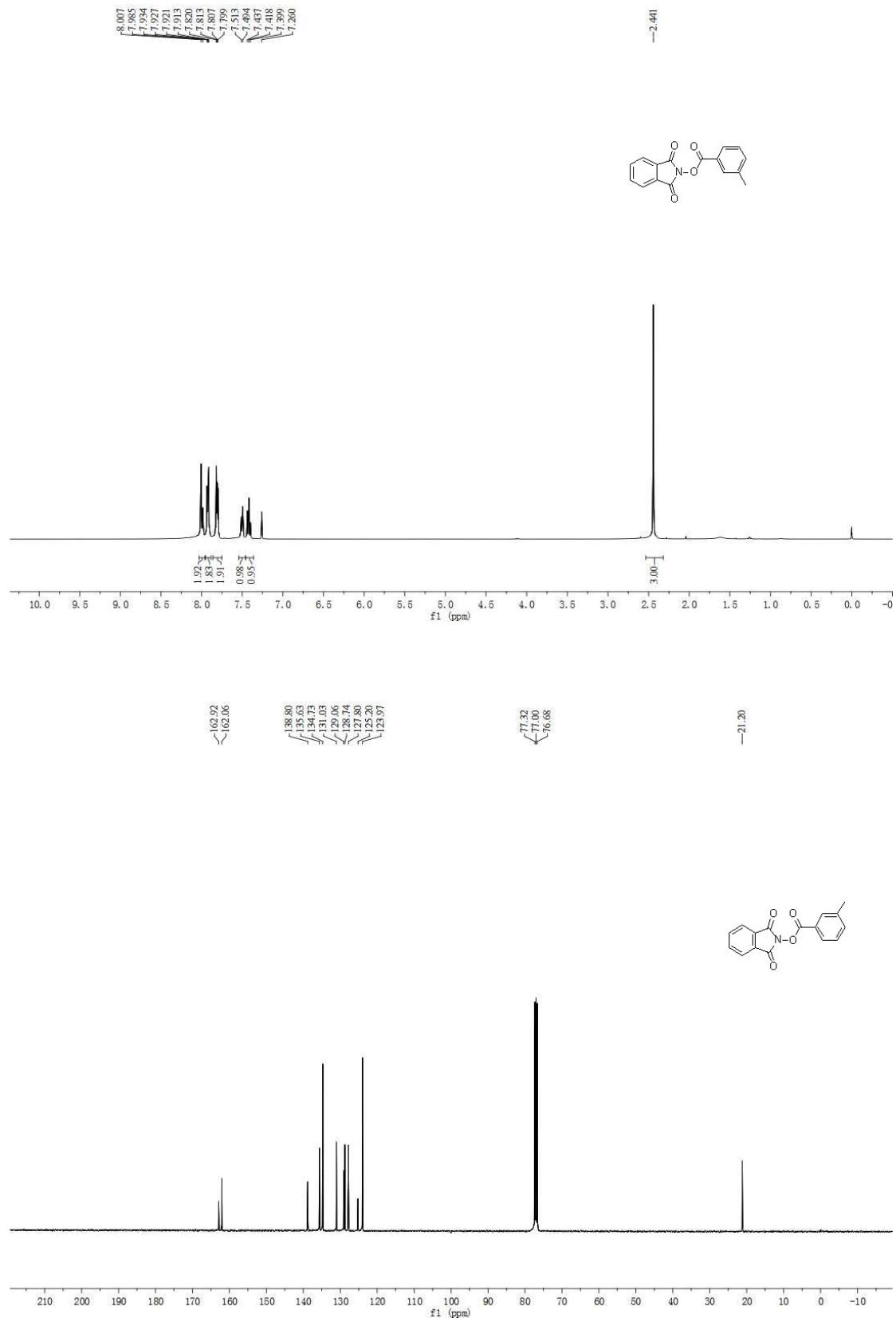
**Product 3i**



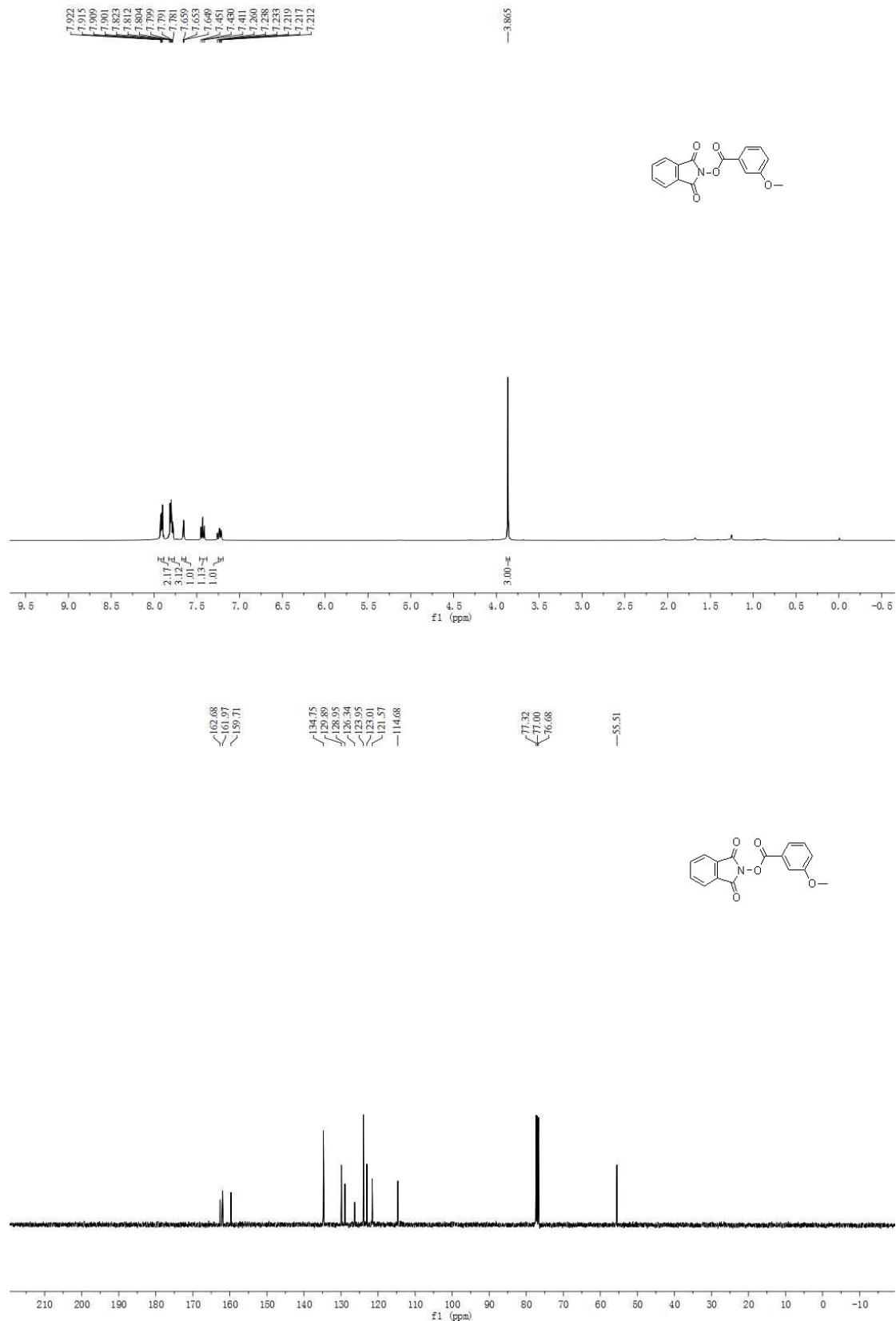
## Product 3j



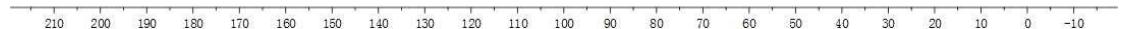
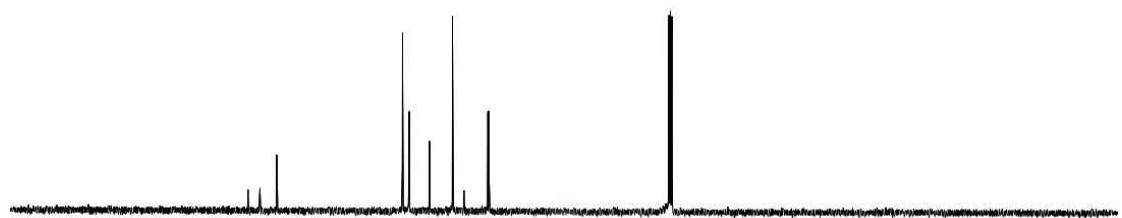
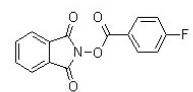
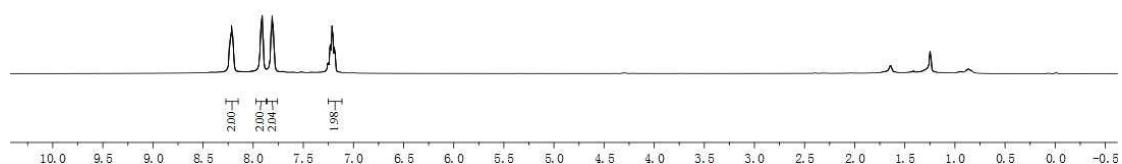
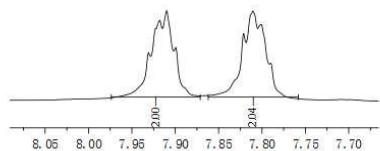
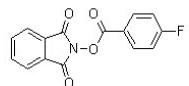
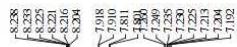
**Product 3k**



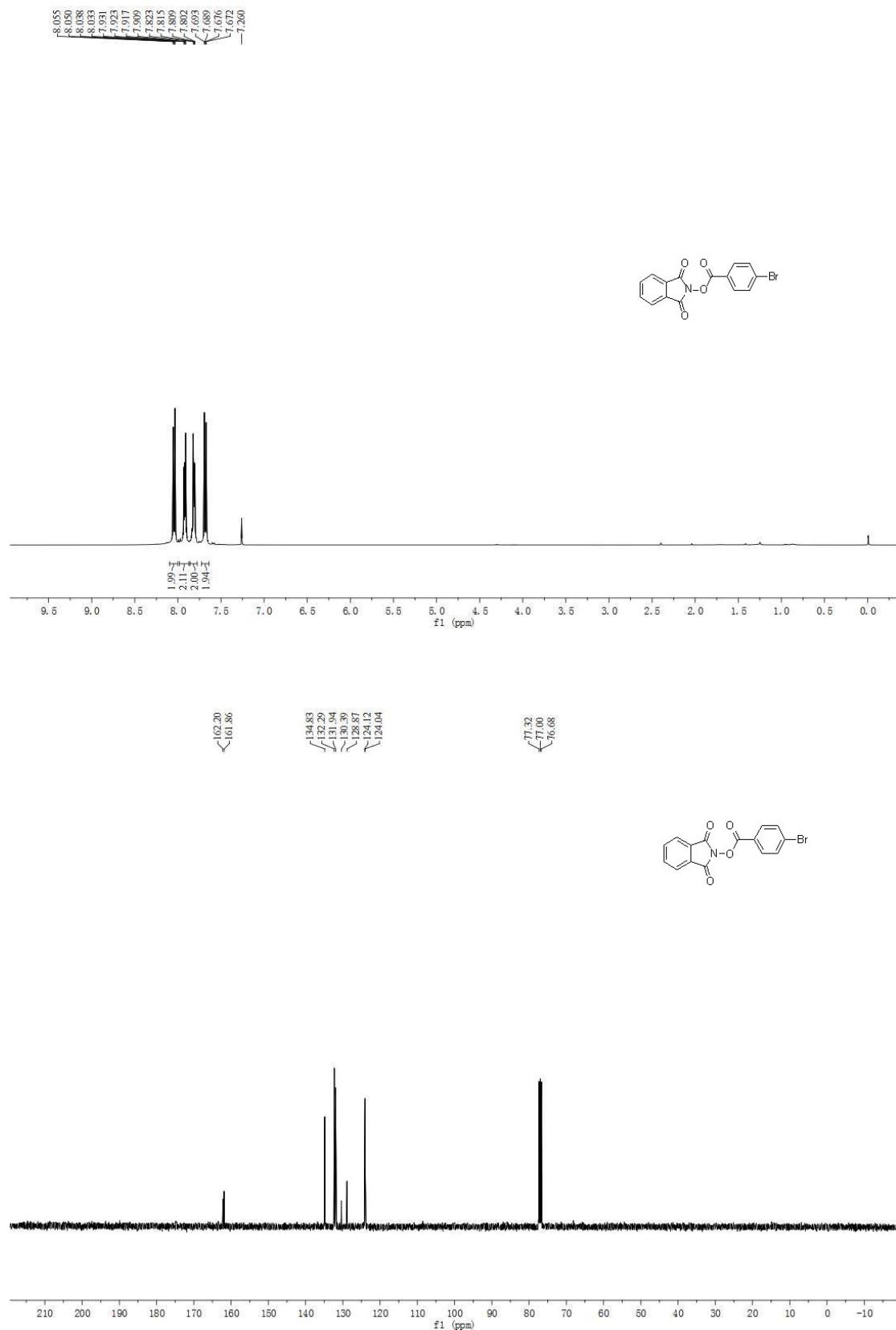
**Product 3l**



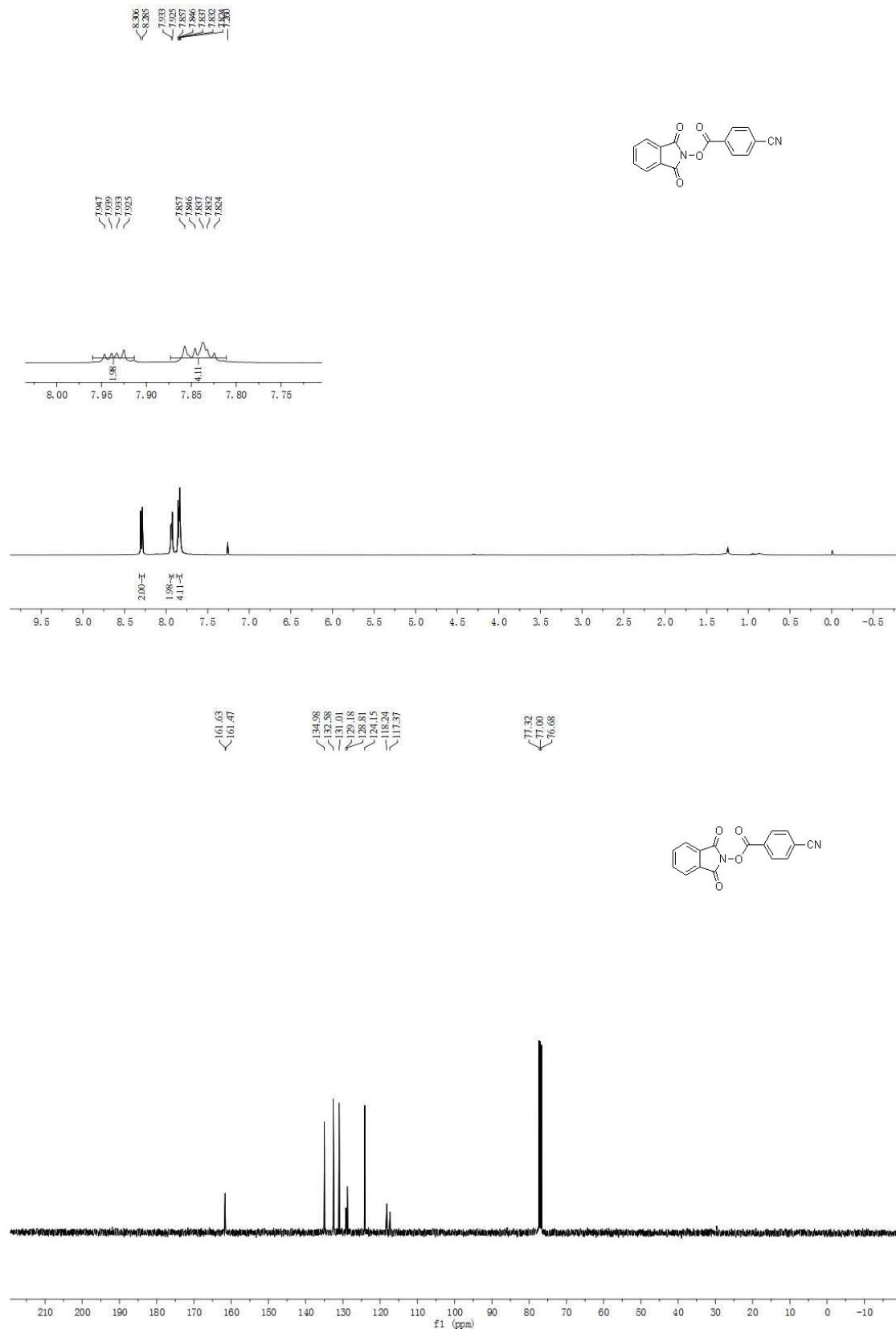
## Product 3m



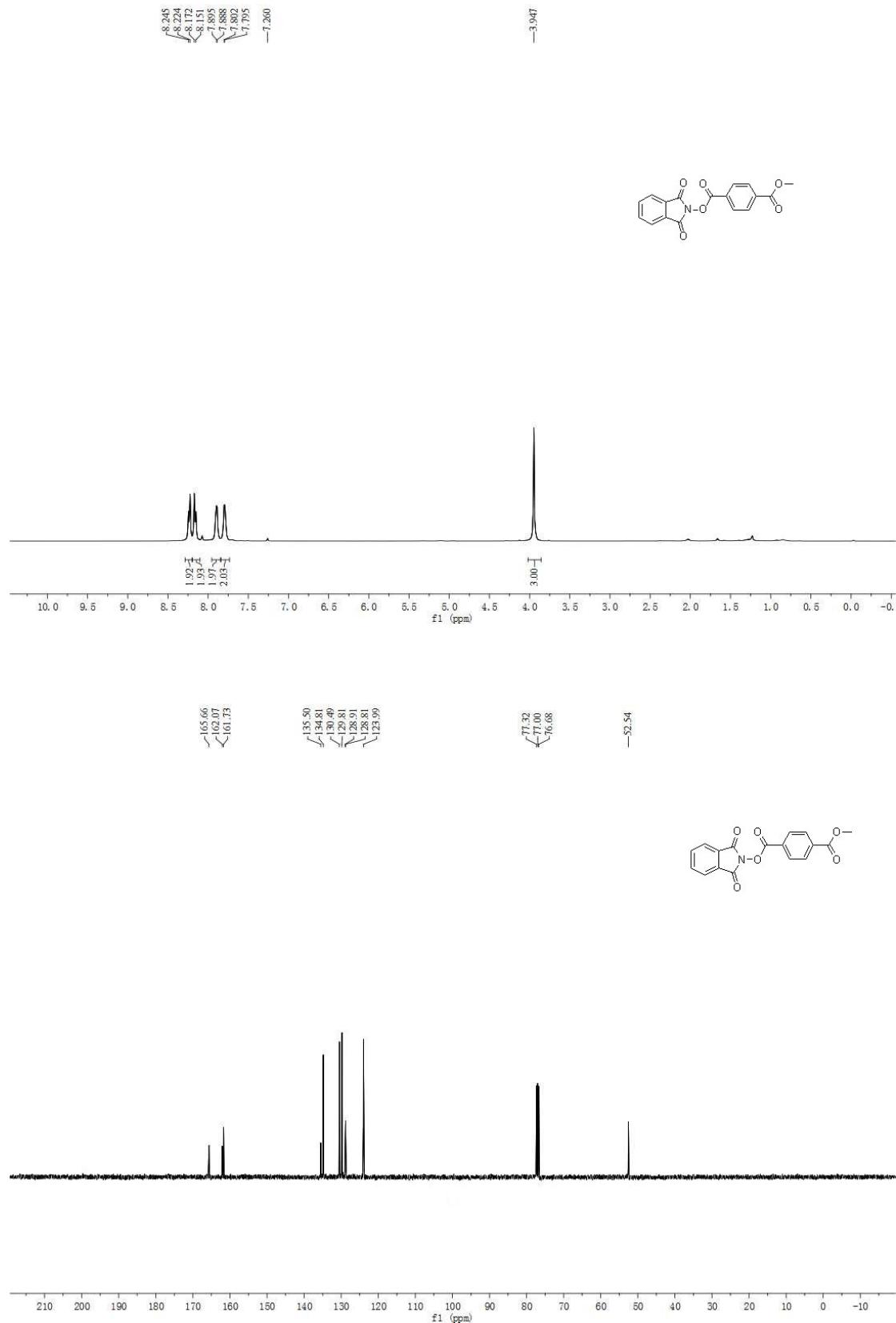
**Product 3n**



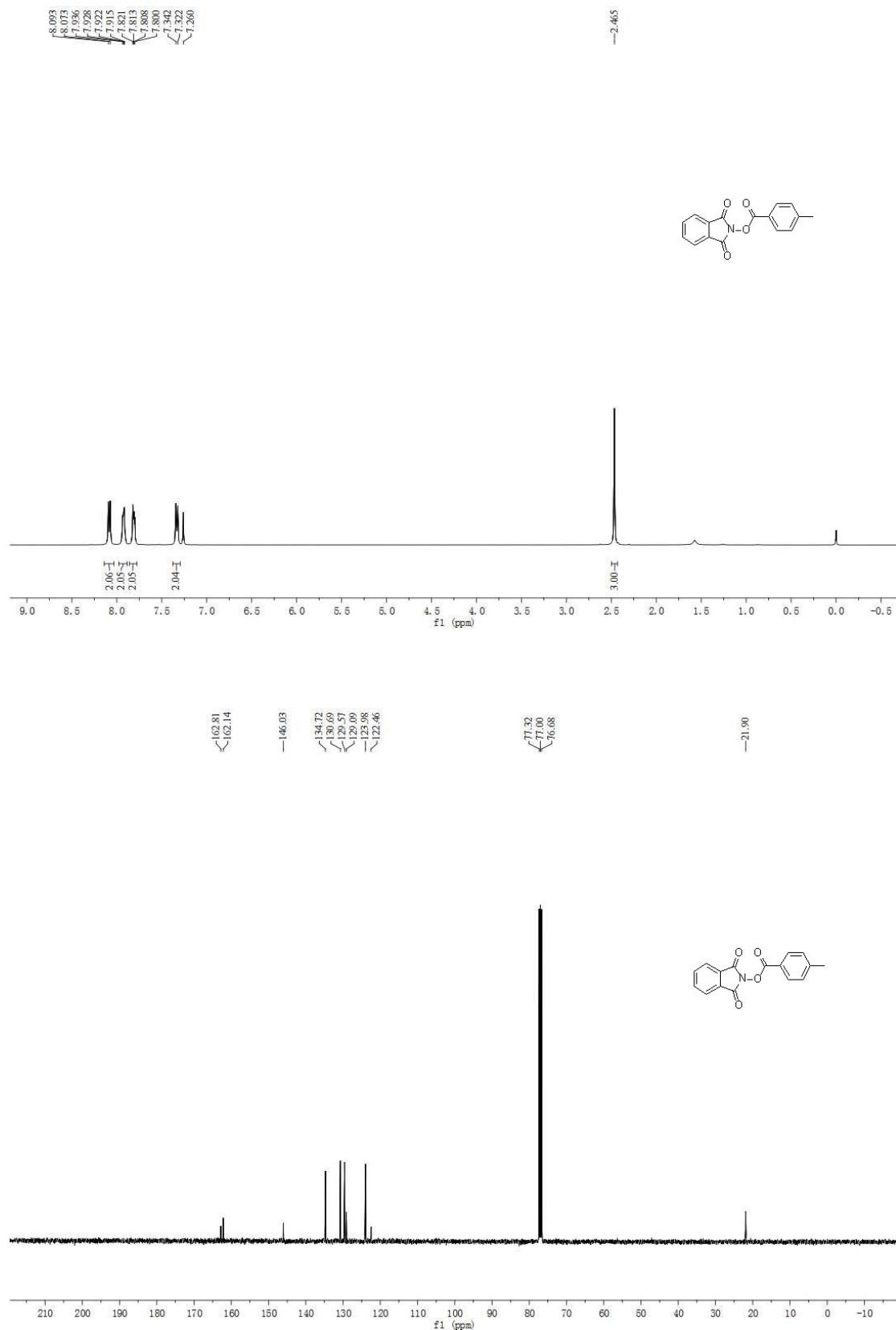
**Product 3o**



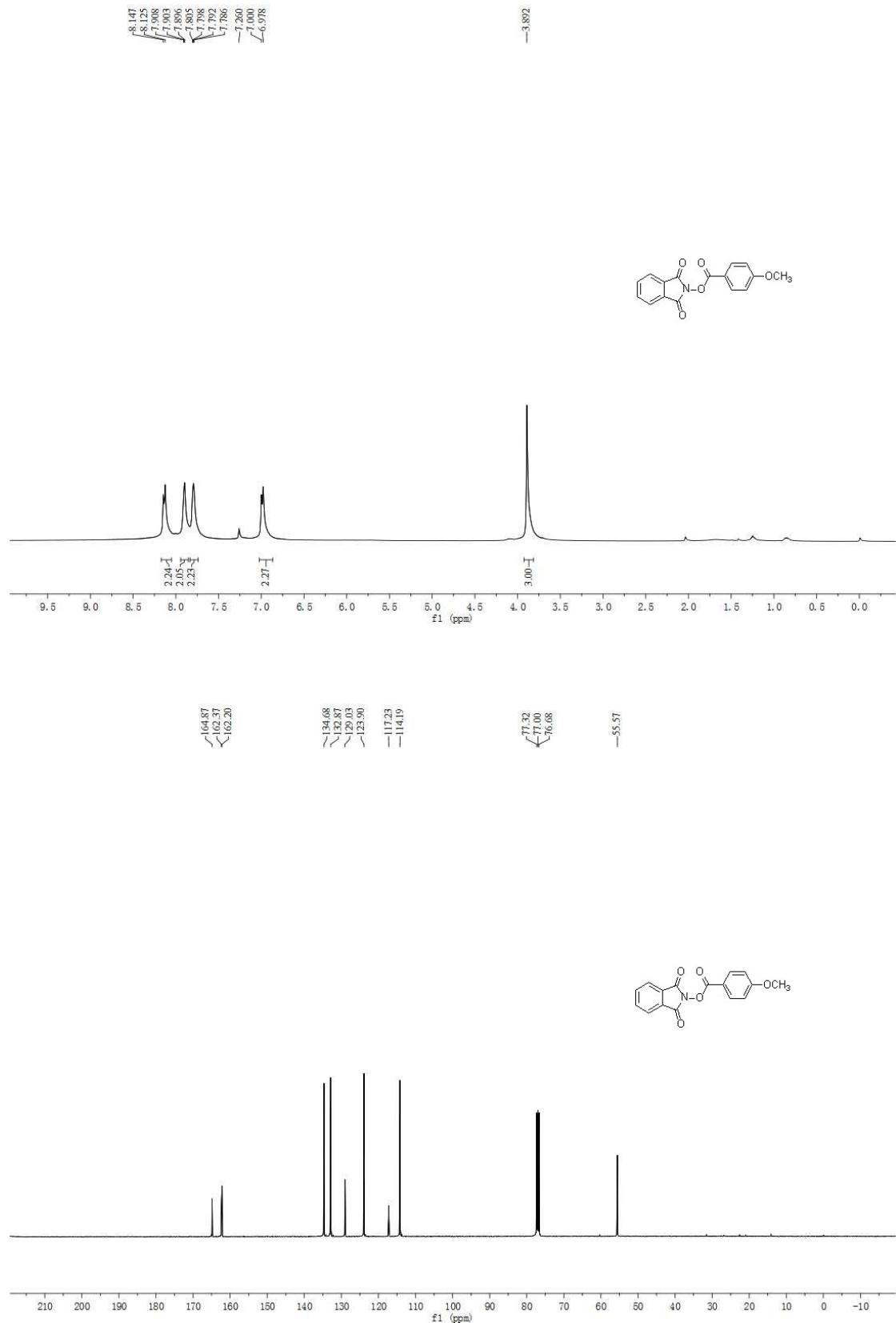
**Product 3p**



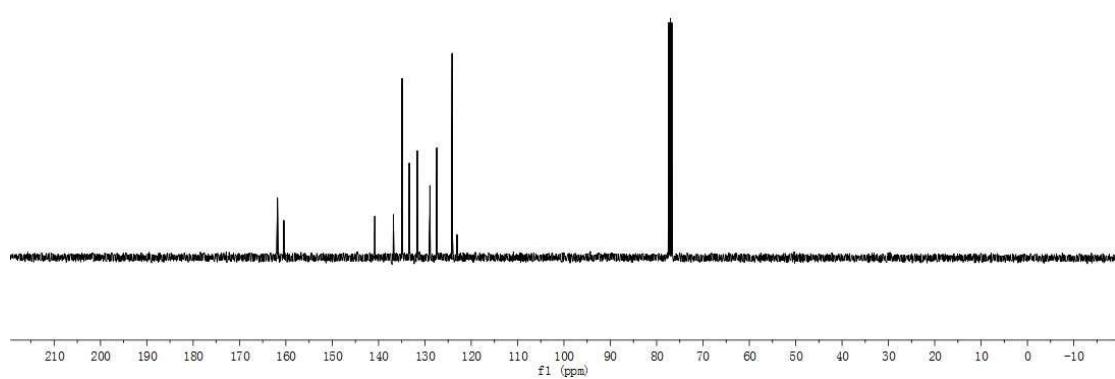
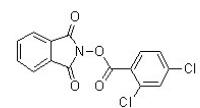
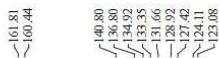
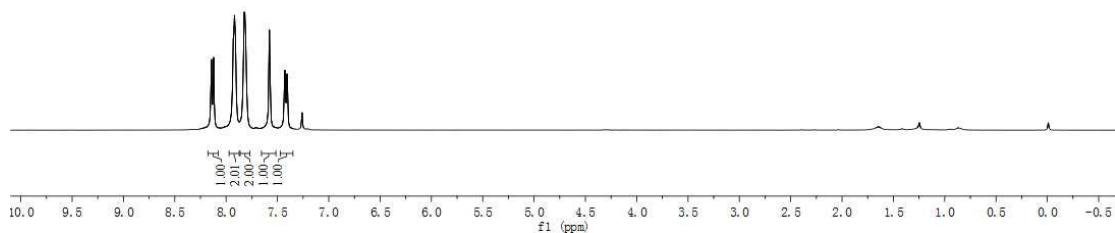
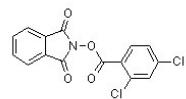
**Product 3q**



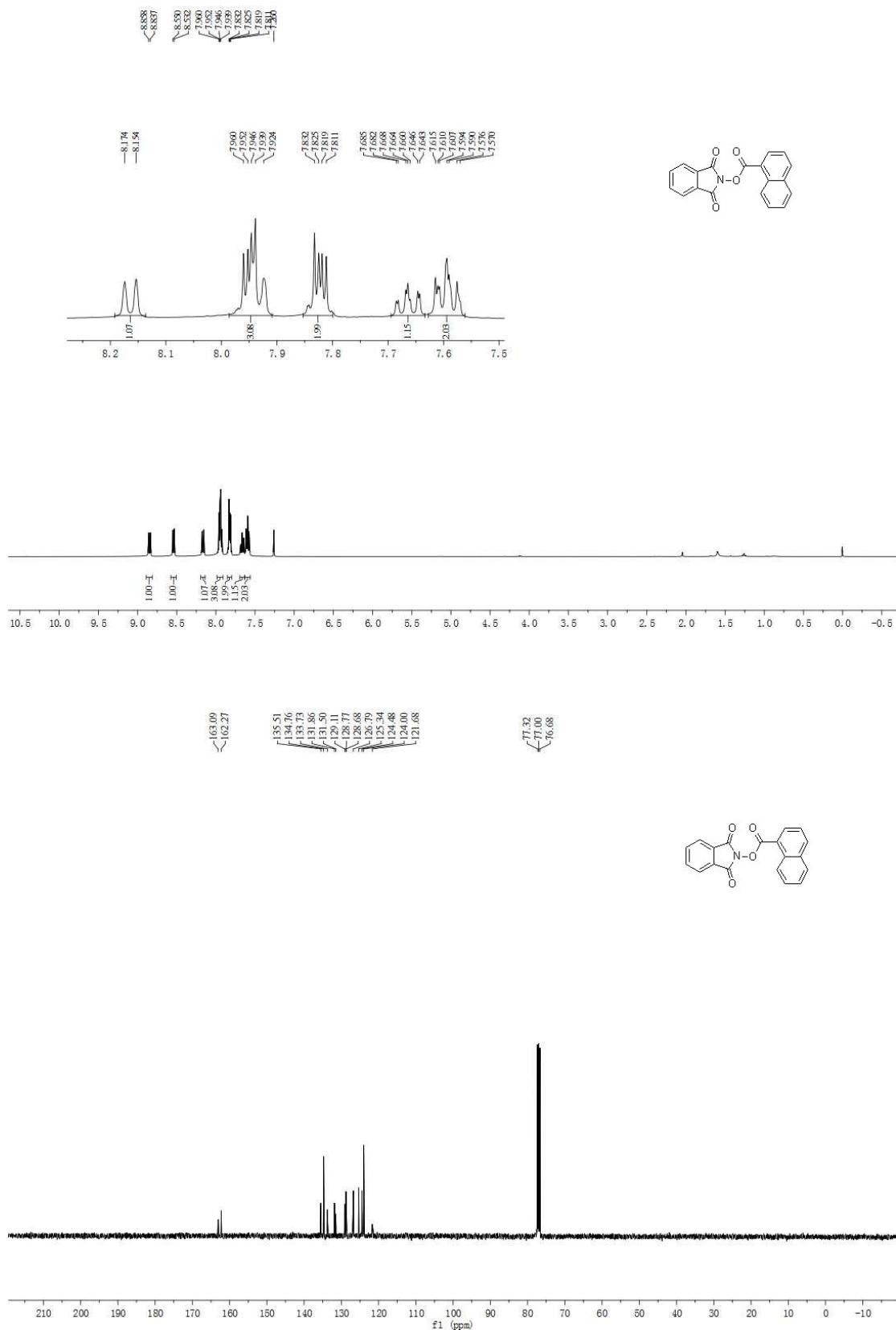
**Product 3r**



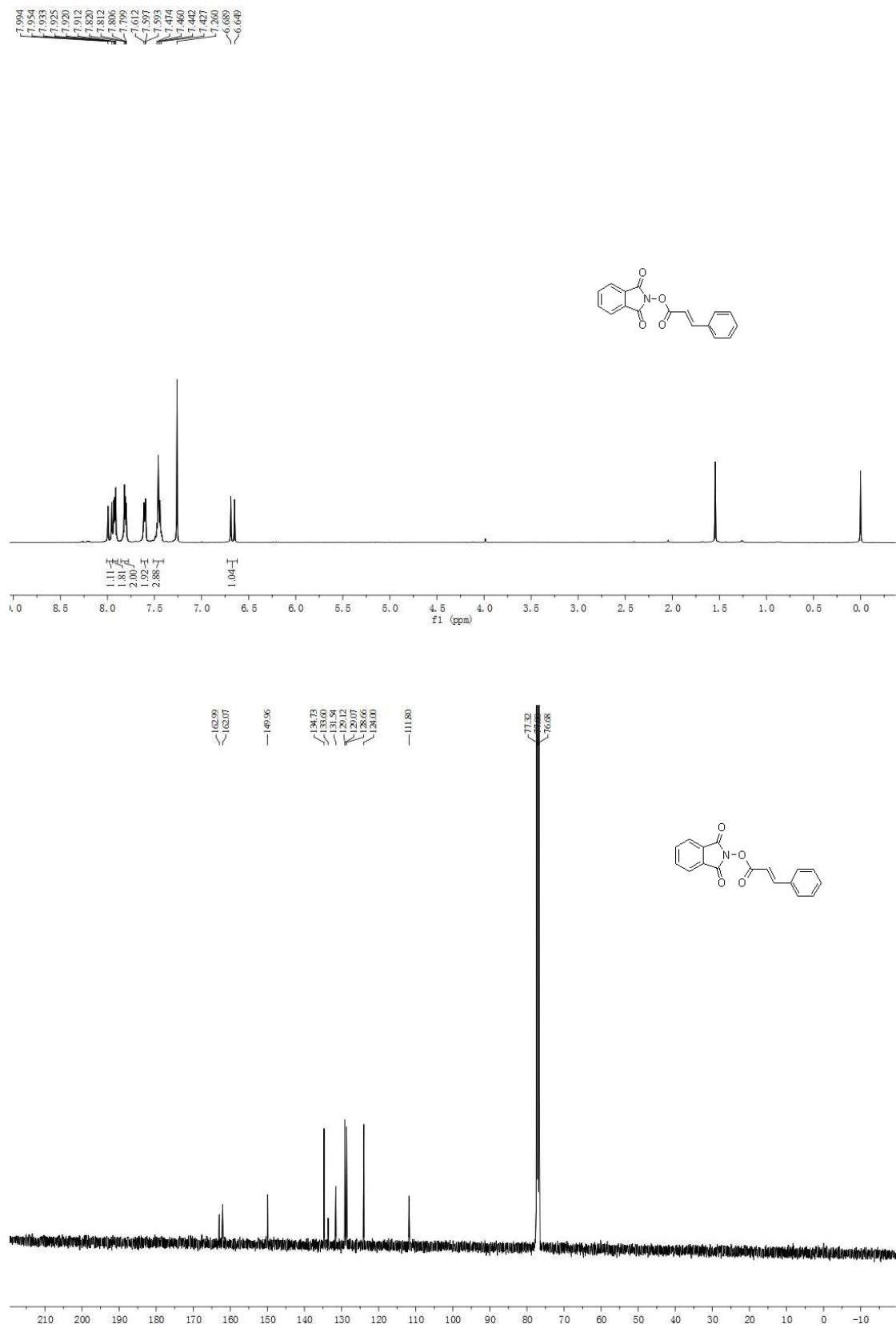
## Product 3s



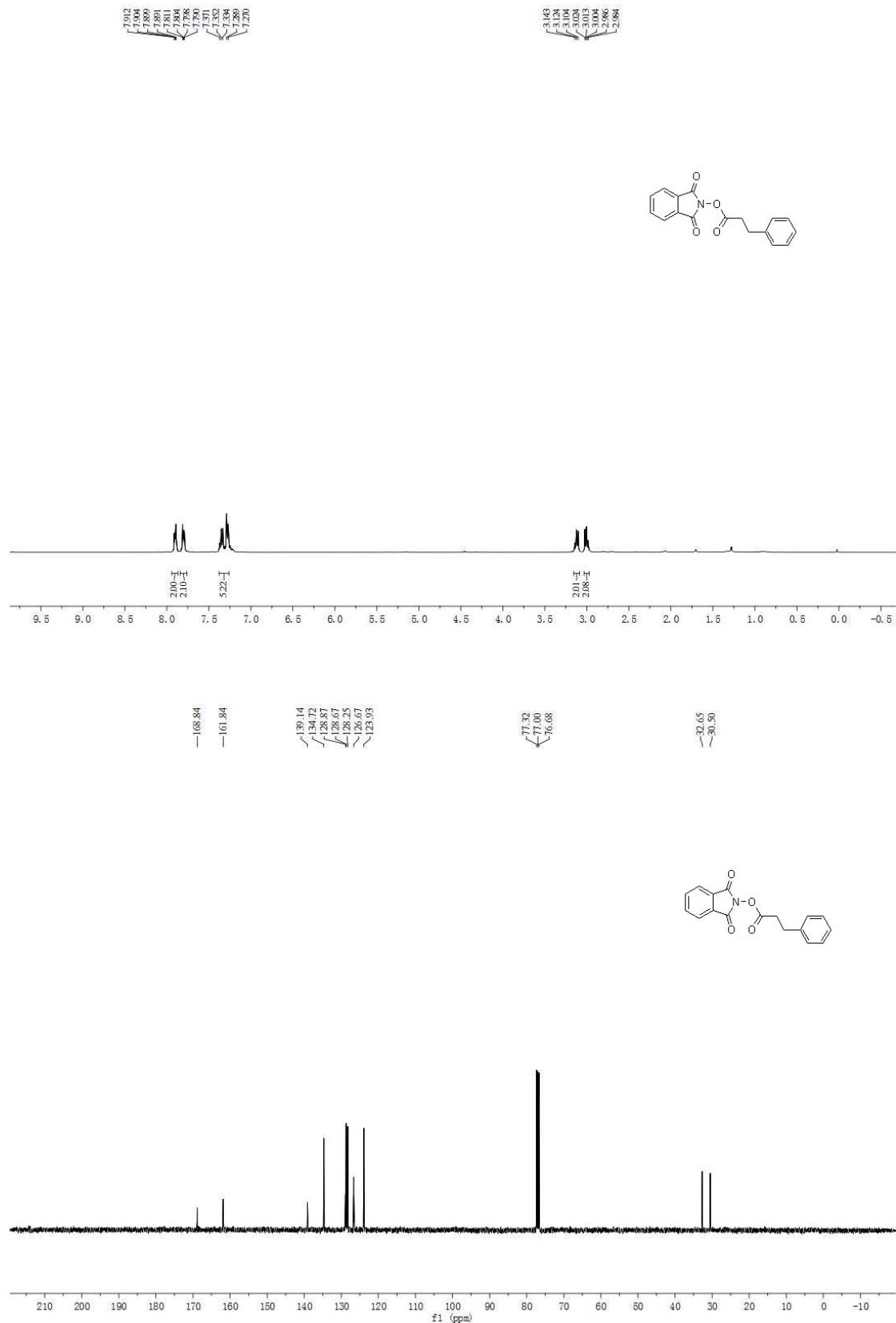
## Product 3t



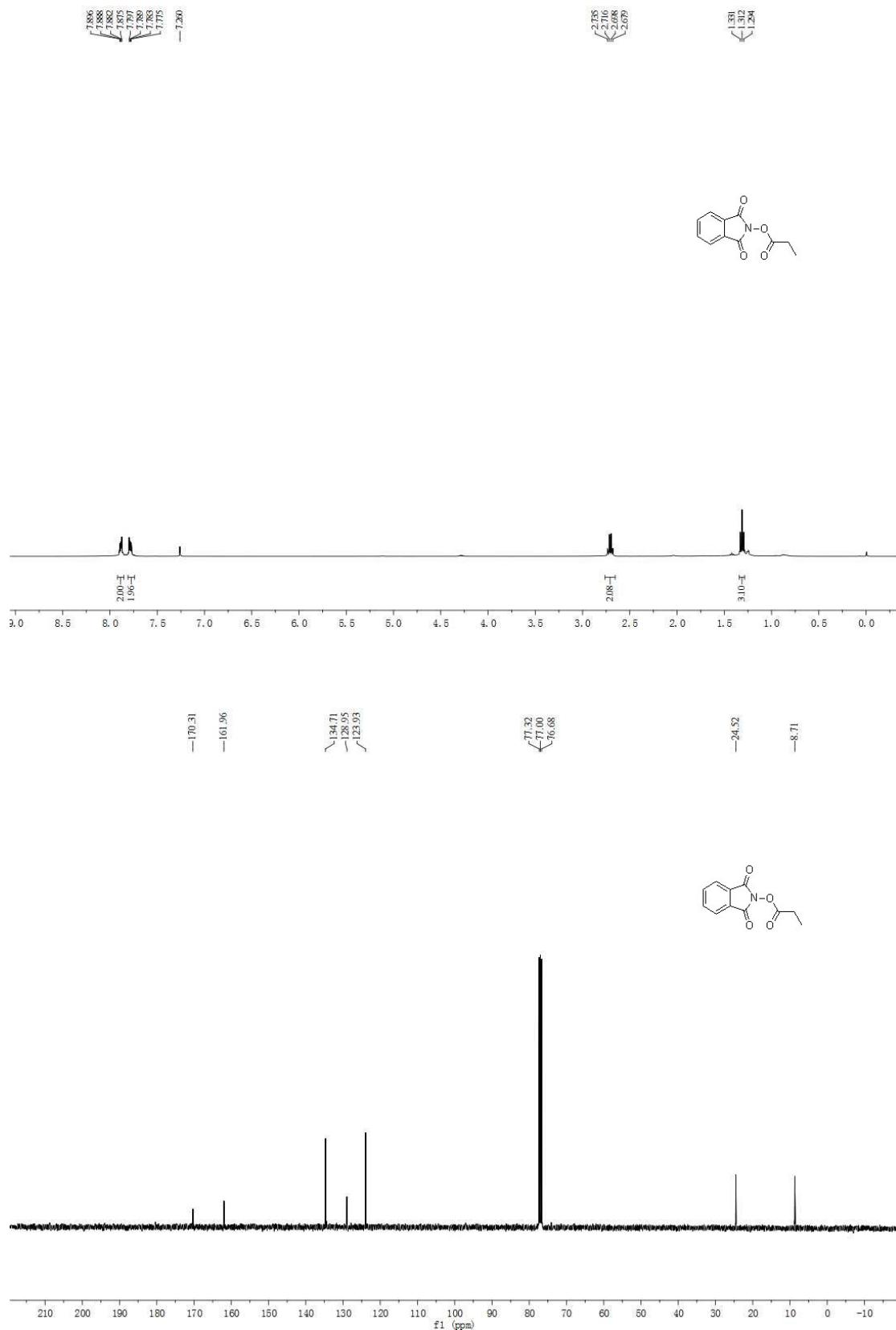
**Product 3u**



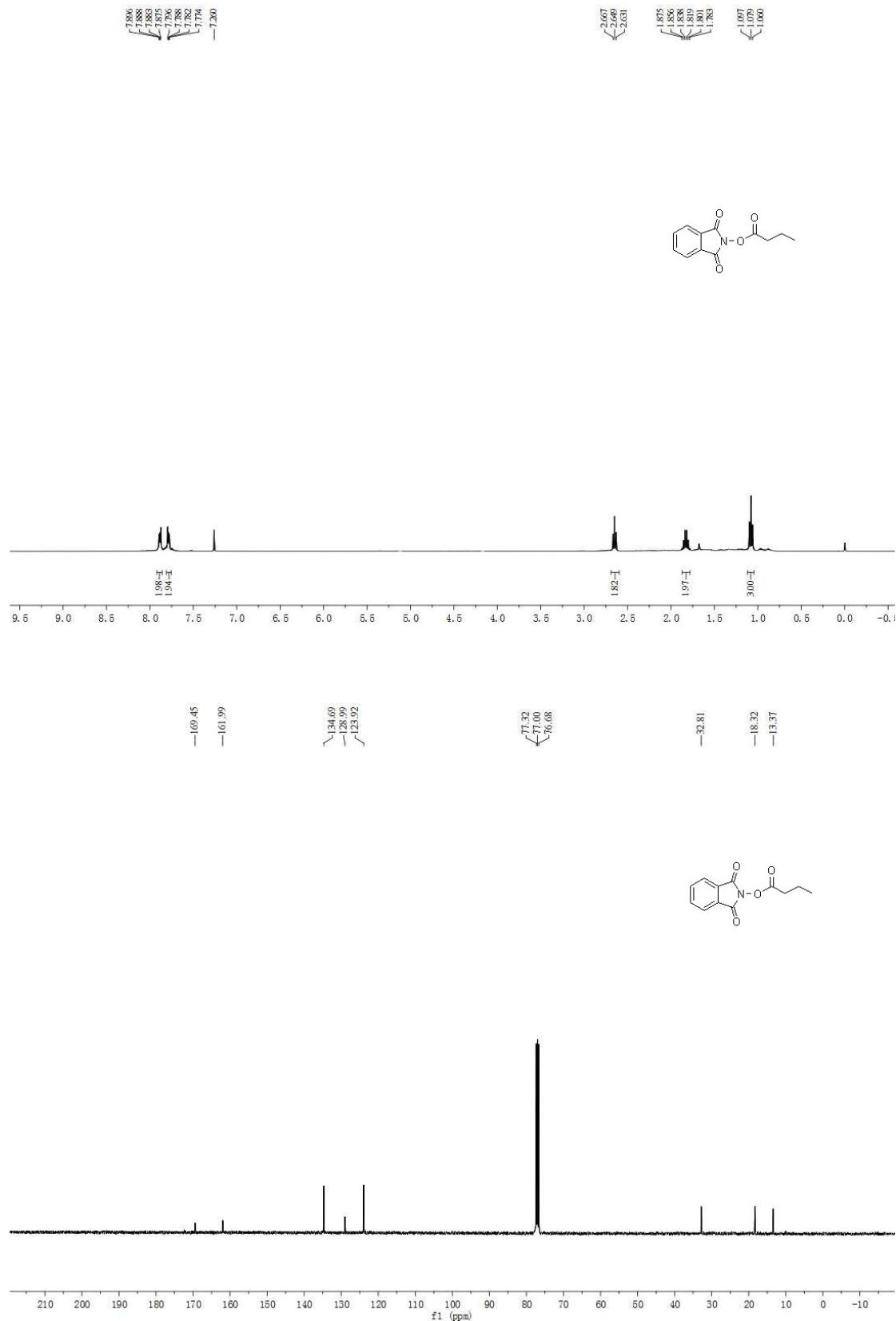
**Product 3v**



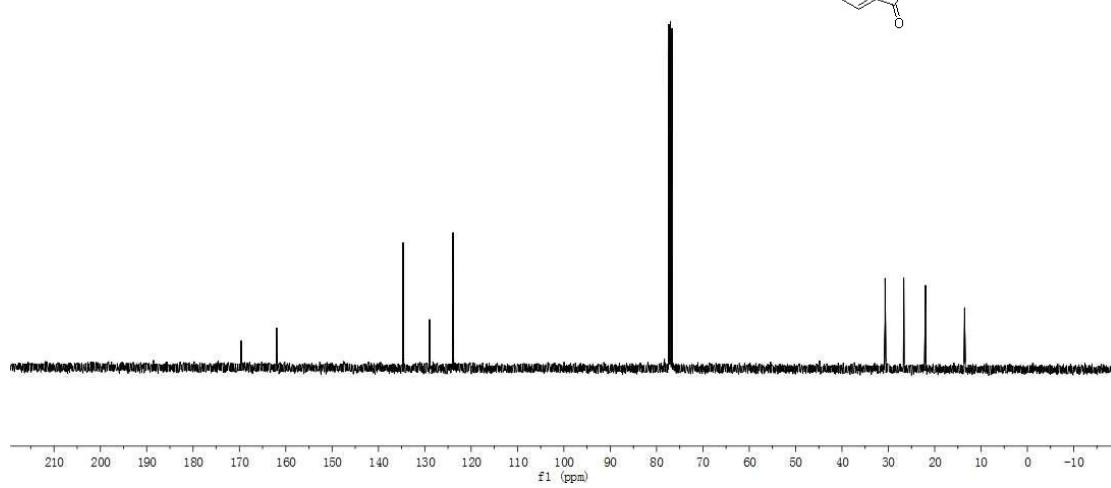
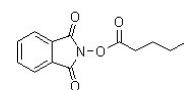
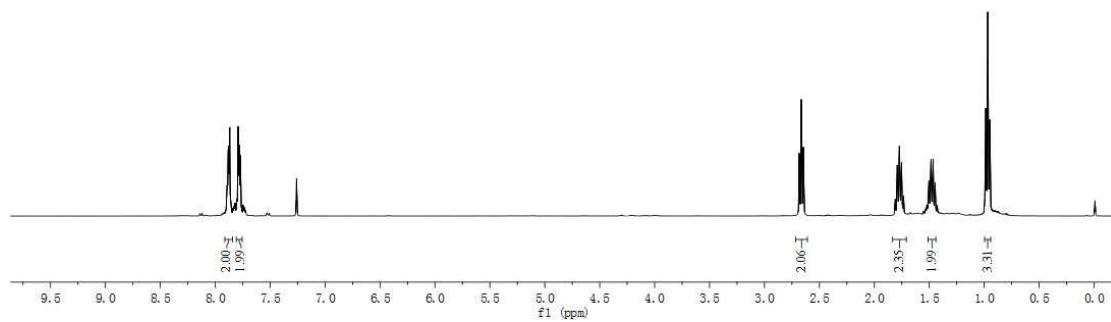
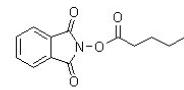
**Product 3w**



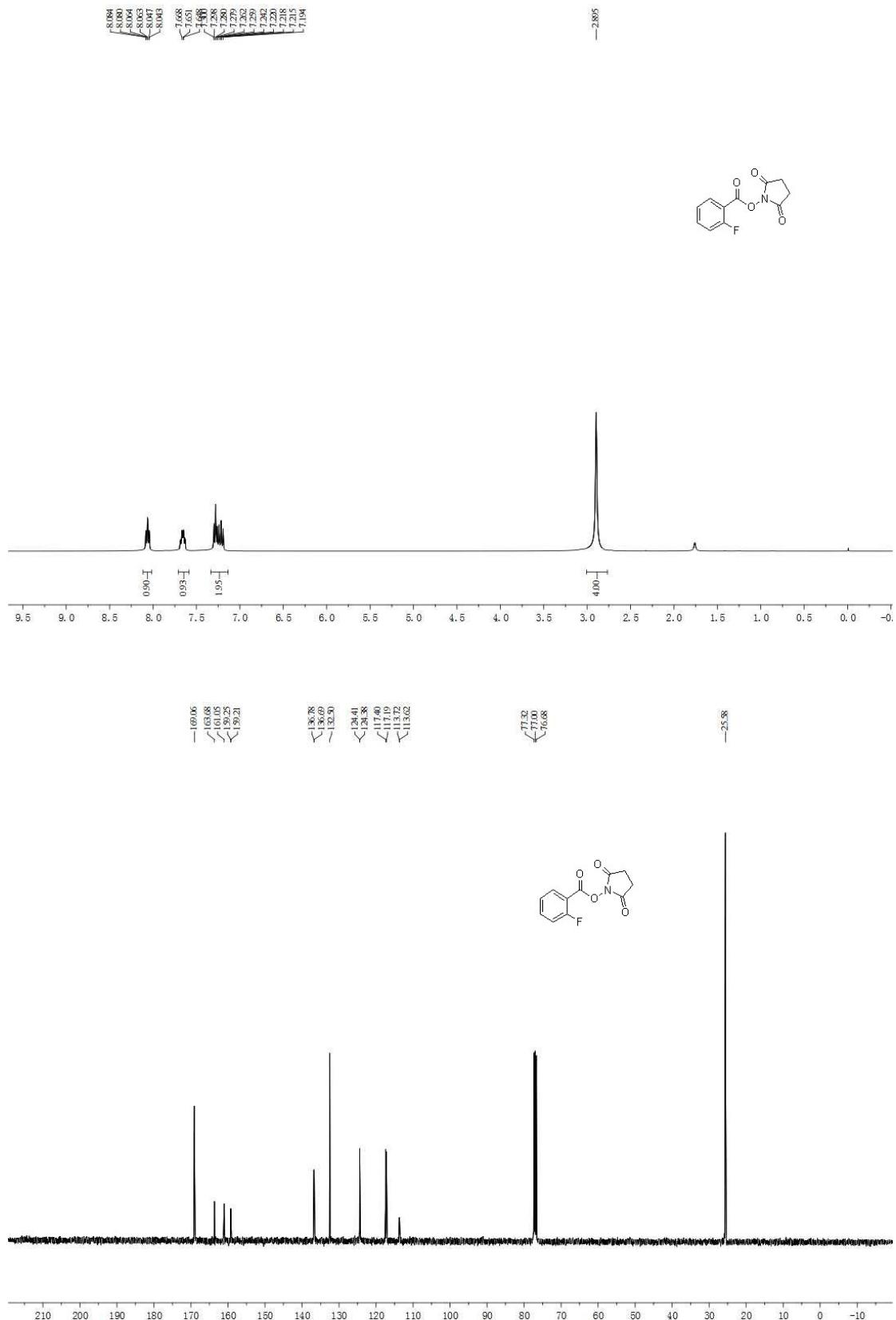
**Product 3x**



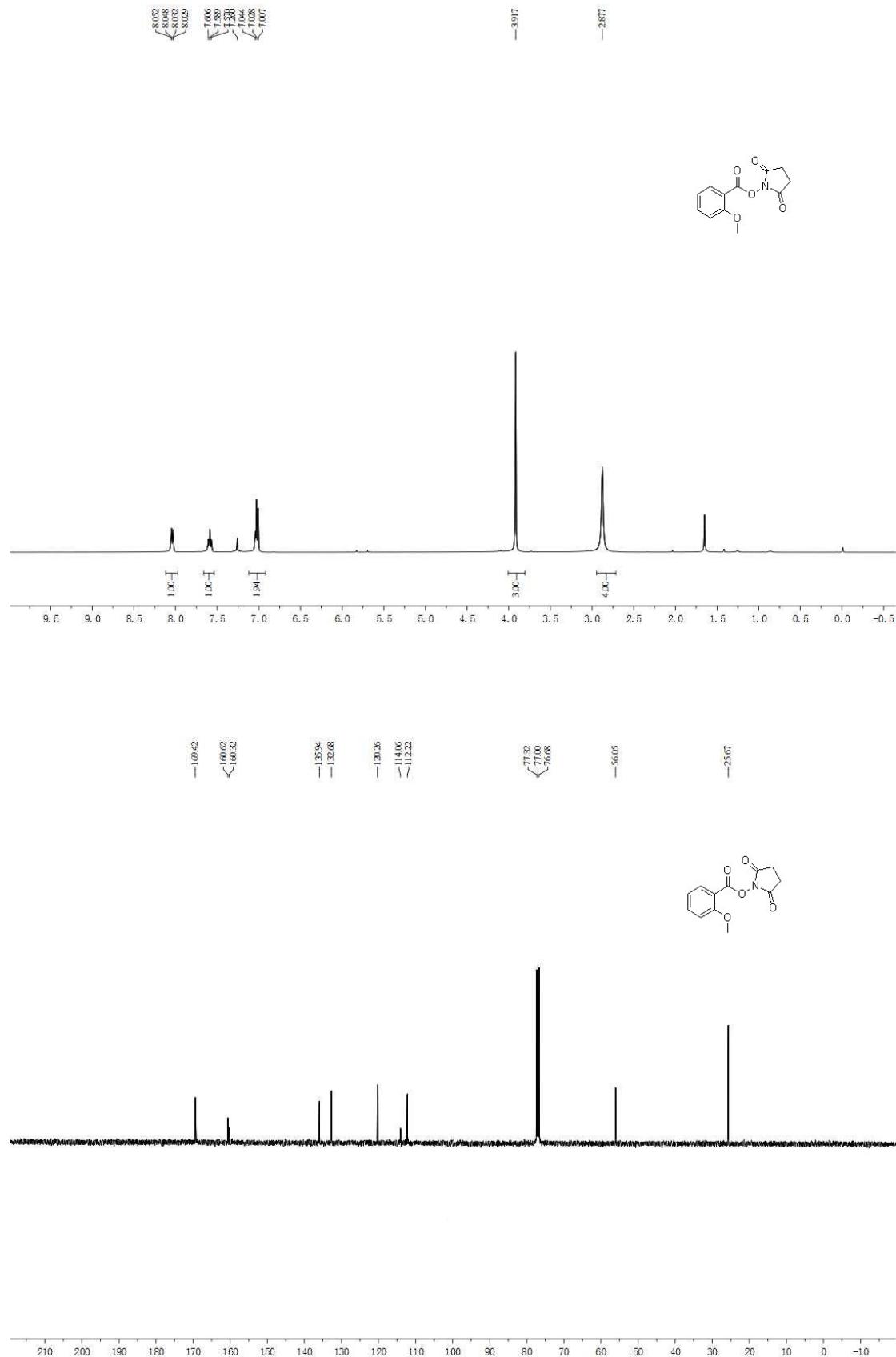
## Product 3y



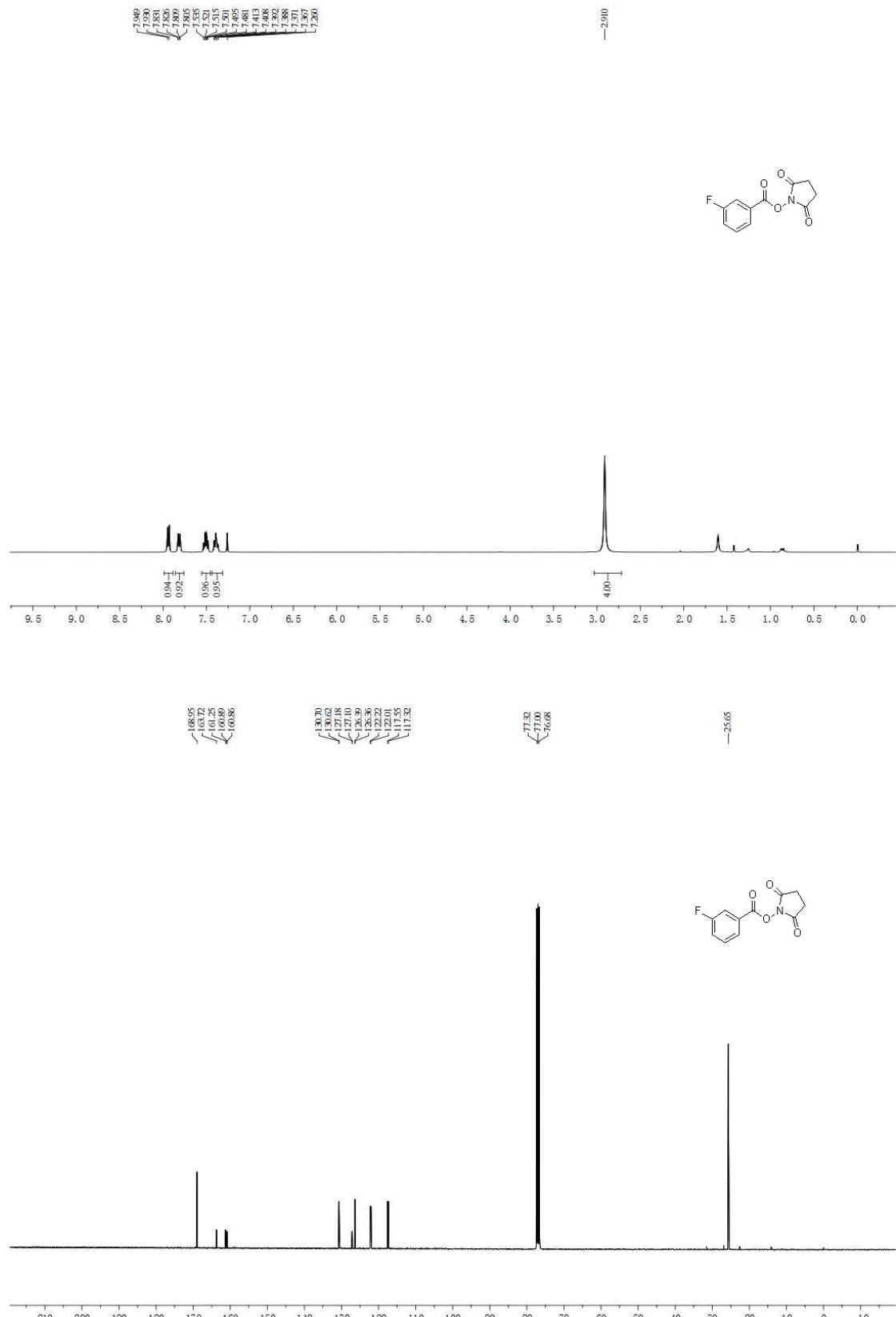
## Product 5a



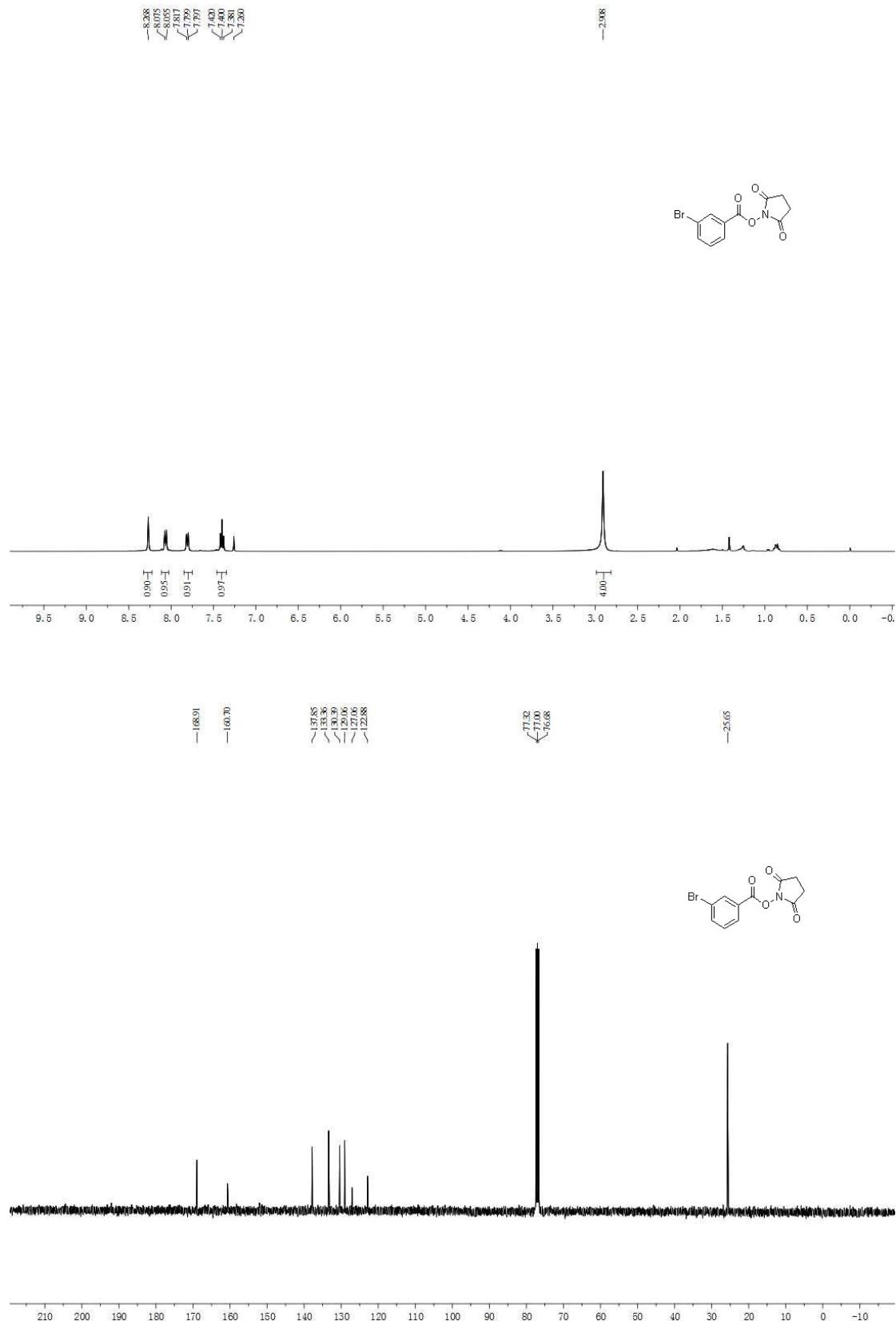
**Product 5b**



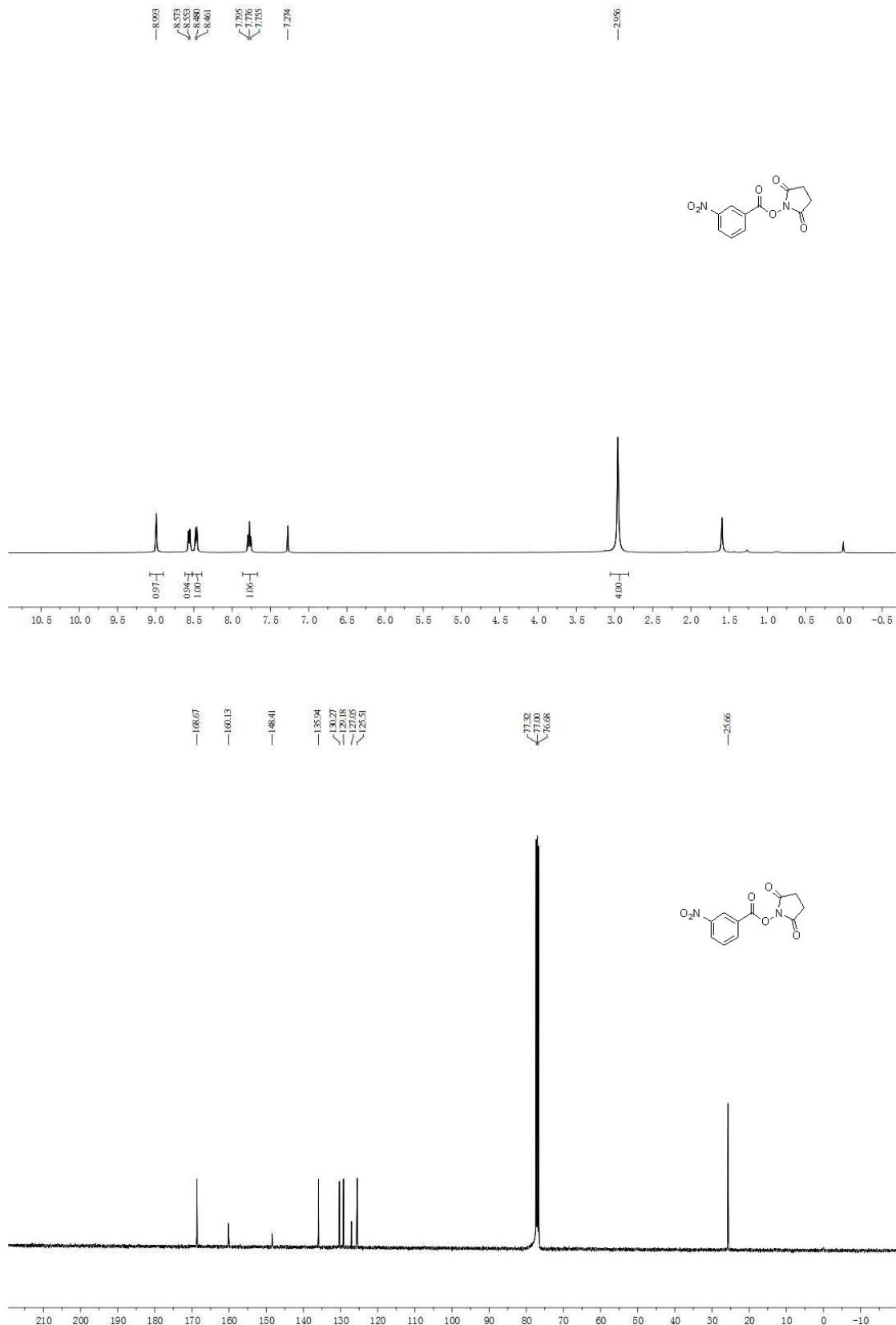
**Product 5c**



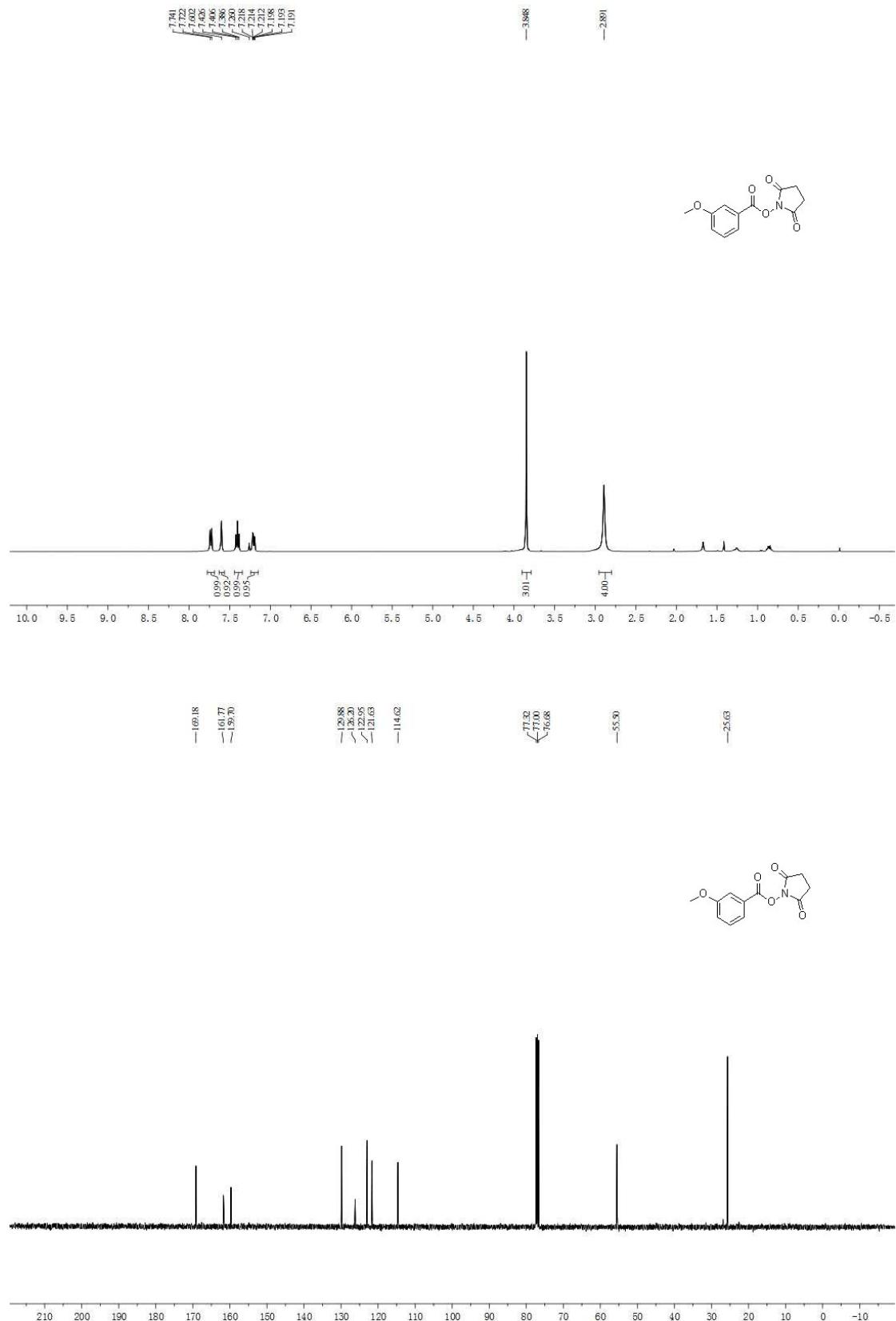
**Product 5d**



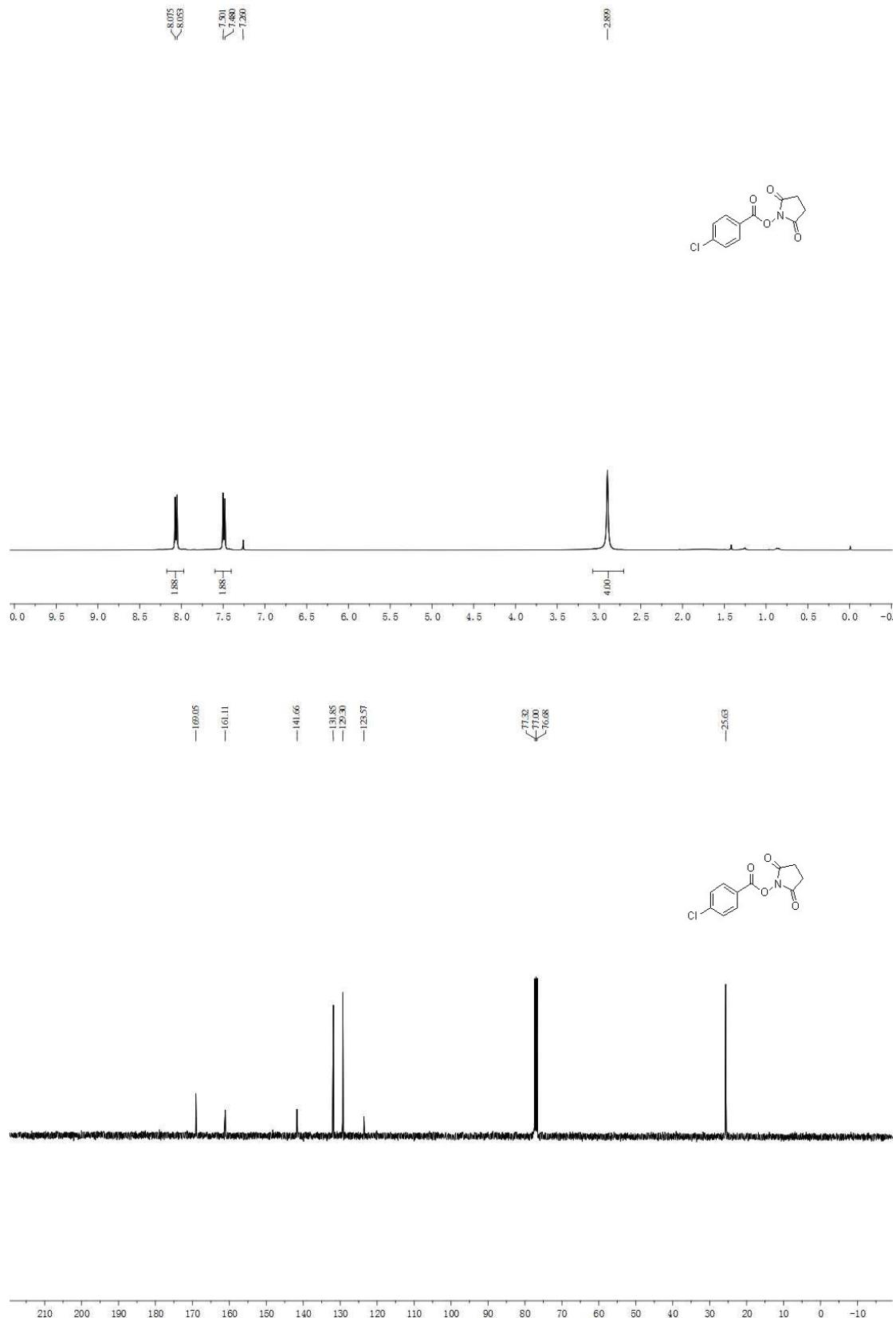
## Product 5e



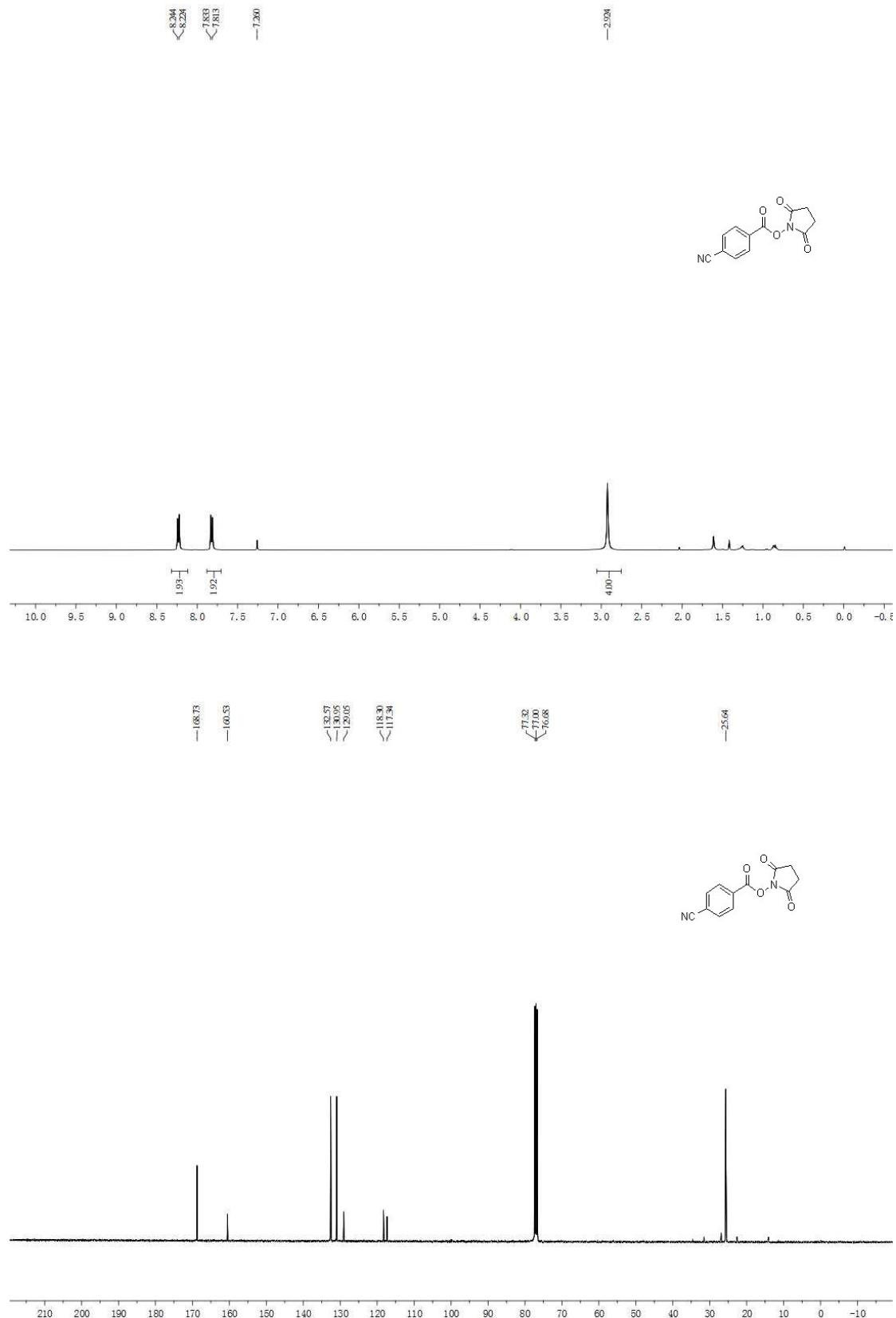
**Product 5f**



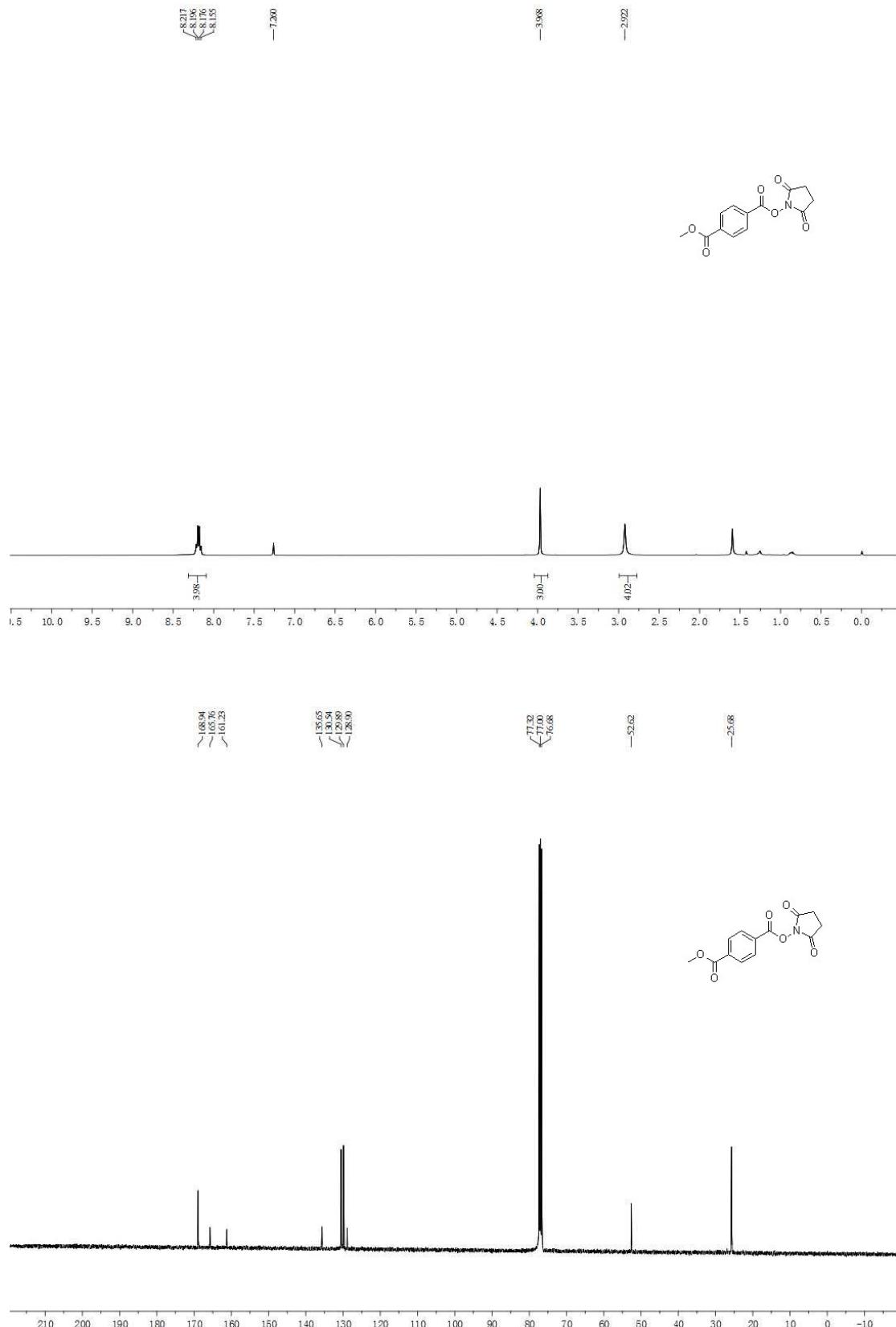
**Product 5g**



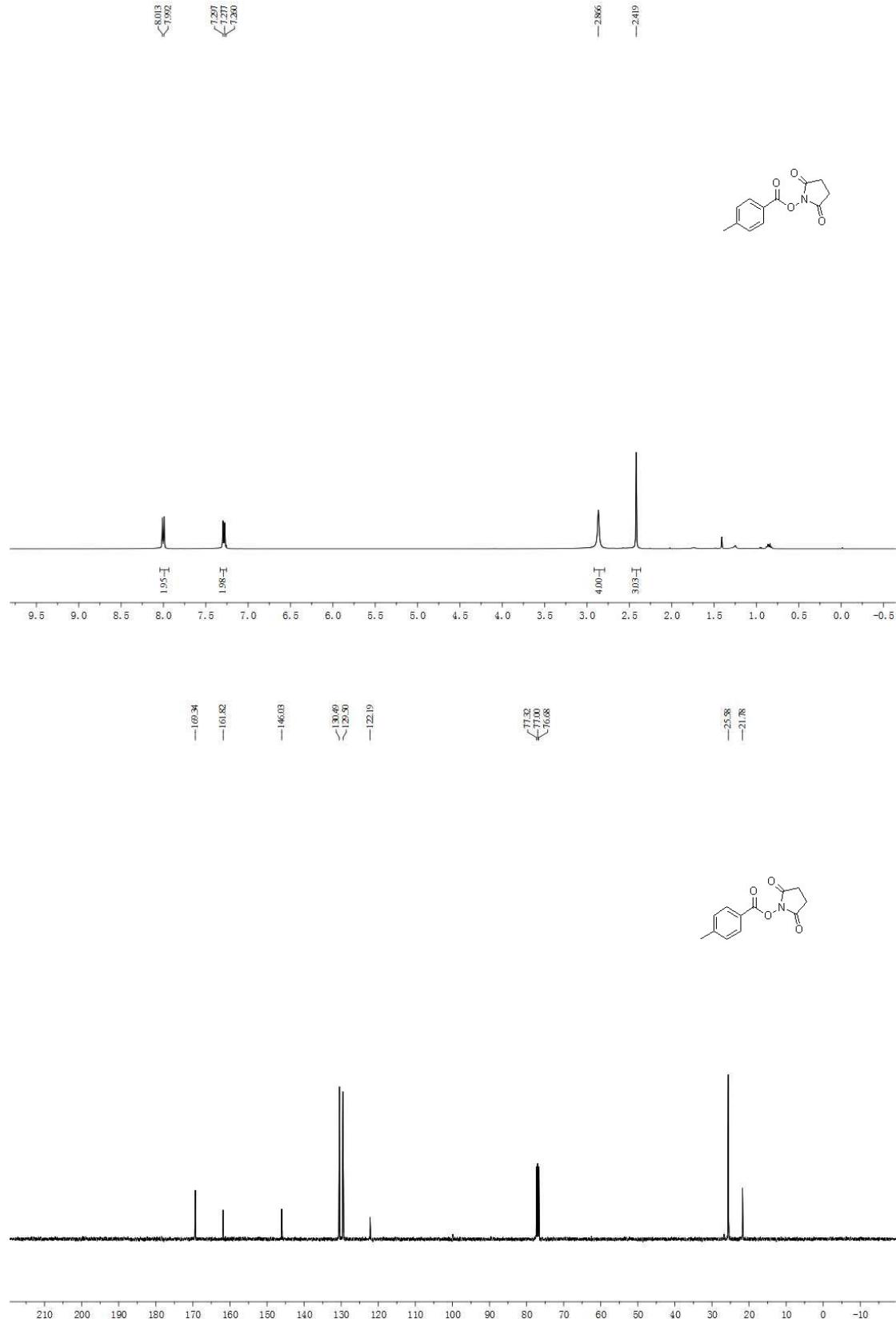
**Product 5h**



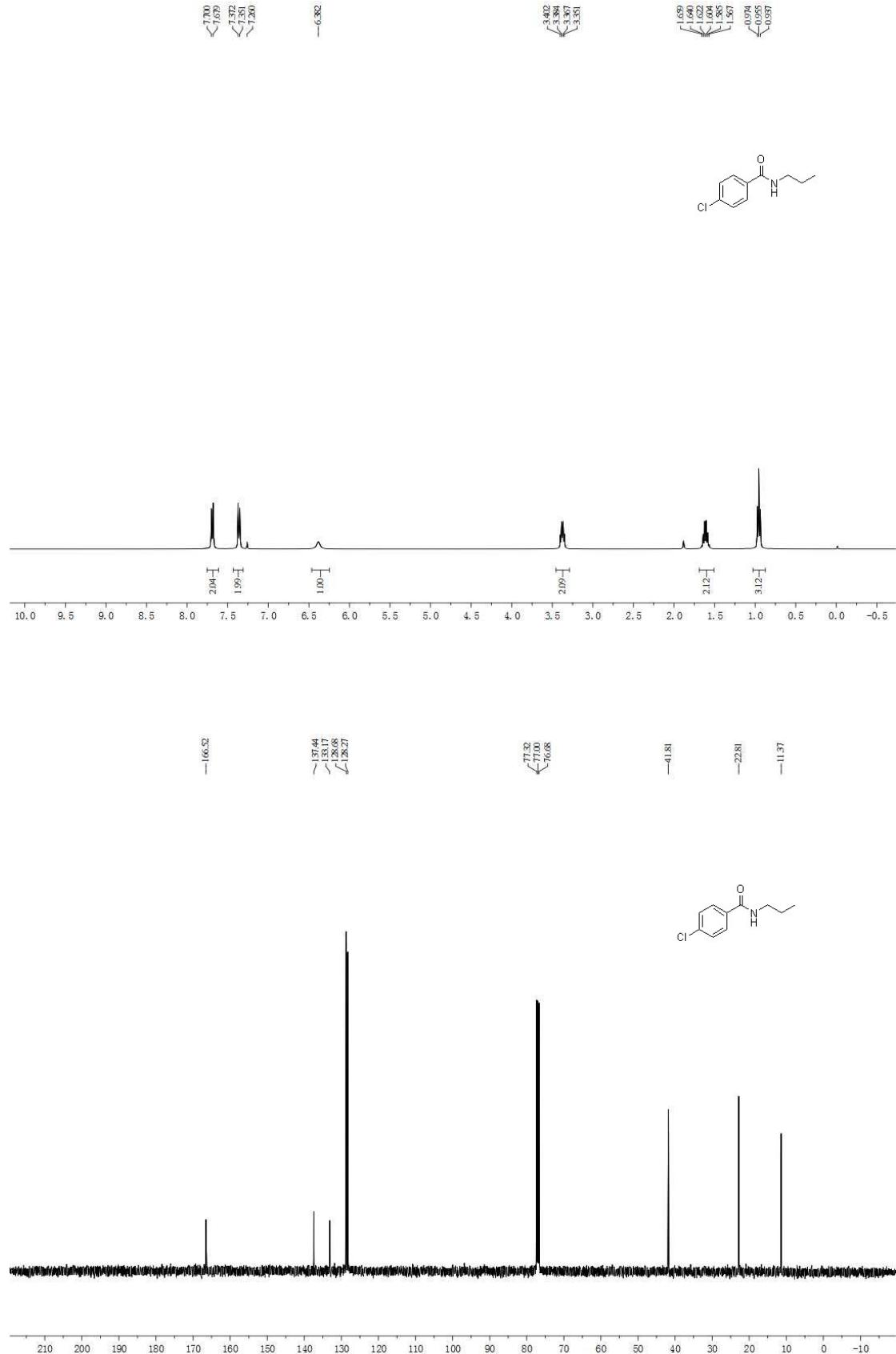
**Product 5i**



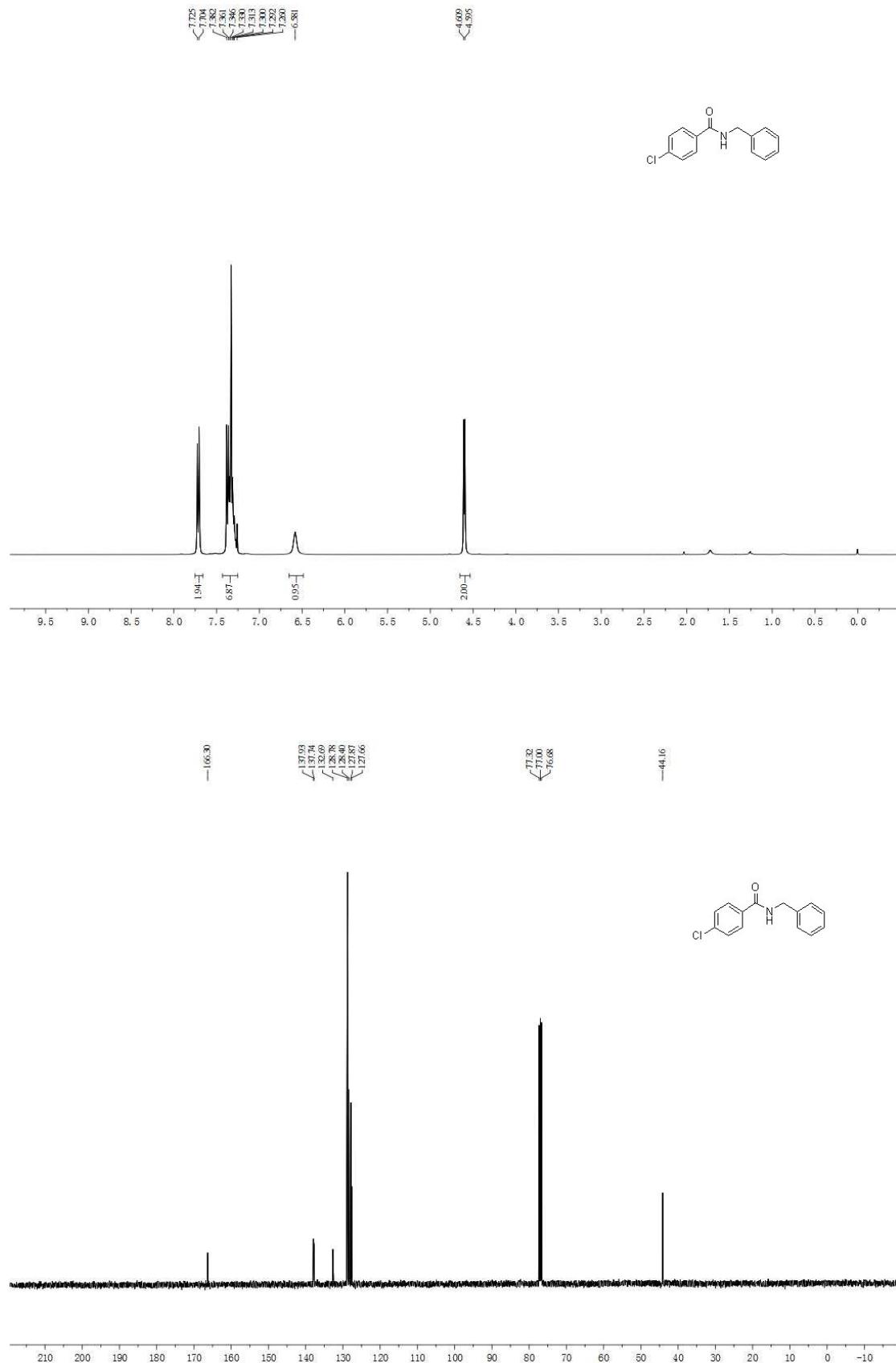
**Product 5j**



**Product 7a**



**Product 7b**



**Product 7c**

