

# Supporting Information

## DBU-mediated synthesis of amides from carbodiimides and unstrained 1,3-diketones

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## 1. General Information

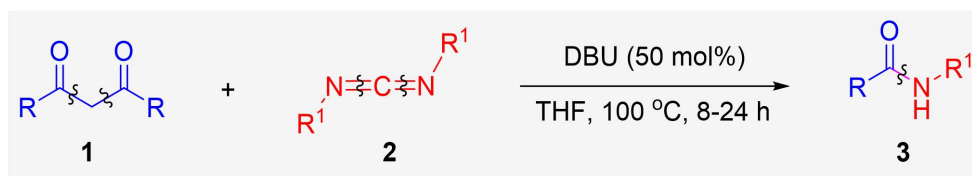
Reactions were monitored by thin layer chromatography using UV light to visualize the course of reaction. Purification of reaction products was carried out by flash chromatography on silica gel. Chemical yields refer to pure isolated substances.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were obtained using a Bruker DPX-400 spectrometer. Chemical shifts are reported in ppm from  $\text{CDCl}_3$  with the solvent resonance as the internal standard. The following abbreviations were used to designate chemical shift multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, h = heptet, m = multiplet, br = broad.

Anhydrous solvents such as  $\text{CH}_2\text{Cl}_2$ ,  $\text{CH}_3\text{CN}$ , THF, toluene, and EtOAc, and the catalysts such as  $\text{Et}_3\text{N}$ , DABCO, DBU,  $\text{Cs}_2\text{O}_3$ ,  $t\text{BuONa}$ , and  $\text{CH}_3\text{ONa}$  were purchased from Energy Chemical. Unless otherwise stated, all purchased reagents were used without further purification. The; All reactions involving air- or moisture-sensitive compounds were carried out under nitrogen atmosphere in dried Schlenk tube. The 1,3-diketones **1**<sup>[1]</sup> and carbodiimides **2**<sup>[2]</sup> was prepared using the literature procedures.

## References

- [1] Y. Ning, Q. Song, P. Sivaguru, L. Wu, E. A. Anderson and X. Bi, *Org. Lett.*, 2022, **24**, 631.  
[2] (a) Z. Zhang, Z. Li, B. Fu and Z. Zhang, *Chem. Commun.*, 2015, **51**, 16312; (b) R. S. Pathare, A. J. Ansari, S. Verma, A. Maurya, A. K. Maurya, V. K. Agnihotri, A. Sharon, R. T. Pardasani and D. M. Sawant, *J. Org. Chem.*, 2018, **83**, 9530; (c) H. E. Houck, K. A. McConnell, C. J. Klingler, A. L. Koenig, G. K. Himka, M. B. Larsen, *ACS Macro Lett.*, 2023, **12**, 1112.

## 2. General procedure and spectral data of products 3.



**General Procedure:** To a 5.0 mL schlenk tube were successively added DBU (0.05 mmol), 1, 3-diketones **1** (0.10 mmol), anhydrous THF (1.0 mL) and di-*p*-tolylcarbodiimide **2** (0.12 mmol). The reaction mixture was stirred vigorously at 100 °C for 8-24 h under N<sub>2</sub> atmosphere till full consumption of **1**. The reaction mixture was then concentrated by rotary vaporation, and the residue was subjected to column chromatography using petroleum ether/ethyl acetate (10:1-4:1) as eluent to afford the desired products **3**.

The reaction was run at 100 °C for 8 h, affording product **3a** as a white solid (75% yield, 15.8 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 2.34 (s, 3H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.45-7.49 (m, 2H), 7.45-7.49 (m, 3H), 7.85-7.87 (m, 3H); <sup>13</sup>{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) δ 165.6, 135.3, 135.0, 134.2, 131.7, 129.5, 128.7, 127.0, 120.3, 20.9; HRMS (ESI): Exact mass calcd for C<sub>14</sub>H<sub>13</sub>NONa [M+Na]<sup>+</sup>: 234.0895, Found: 234.0901.

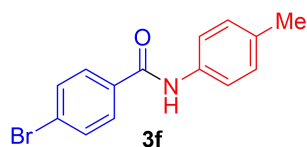
The reaction was run at 100 °C for 8 h, affording product **3b** as a white solid (80% yield, 17.8 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 2.34 (s, 3H), 2.42 (s, 3H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.51 (d, *J* = 8.4 Hz, 2H), 7.76 (d, *J* = 8.4 Hz, 3H); <sup>13</sup>{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>): δ 165.5, 142.2, 135.4, 134.1, 132.2, 129.5, 129.4, 127.0, 120.2, 21.5, 20.9; HRMS (ESI): Exact mass calcd for C<sub>15</sub>H<sub>15</sub>NONa [M+Na]<sup>+</sup>: 248.1052, Found: 248.1050.

The reaction was run at 100 °C for 10 h, affording product **3c** as a white solid (67% yield, 16.1 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 2.33 (s, 3H), 3.85 (s, 3H), 6.91-6.95 (m, 2H), 7.14 (d, *J* = 8.4 Hz, 2H), 7.50 (d, *J* = 8.4 Hz, 2H), 7.80-7.84 (m, 2H), 7.86 (s, 1H); <sup>13</sup>{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) δ 165.2, 162.3, 135.5, 133.9, 129.4, 128.9, 127.1, 125.4, 120.3, 113.8, 55.4, 20.8; HRMS (ESI): Exact mass calcd for C<sub>15</sub>H<sub>15</sub>NO<sub>2</sub>Na [M+Na]<sup>+</sup>: 264.1001, Found: 264.0998.

The reaction was run at 100 °C for 8 h, affording product **3d** as a white solid (85% yield, 19.5 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 2.34 (s, 3H), 7.12-7.18 (m, 4H), 7.49 (d, *J* = 8.4 Hz, 2H), 7.75 (s, 1H), 7.85-7.90 (m, 2H); <sup>13</sup>{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) δ 164.8 (d, *J* = 201 Hz), 164.6, 135.1, 134.4, 131.2 (d, *J* = 2.5 Hz), 129.6, 129.3 (d, *J* = 7.1 Hz), 120.3, 115.8 (d, *J* = 18 Hz), 20.9; <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) -107.64. HRMS (ESI): Exact mass calcd for C<sub>14</sub>H<sub>12</sub>FNONa [M+Na]<sup>+</sup>: 252.0801, Found: 252.0796.

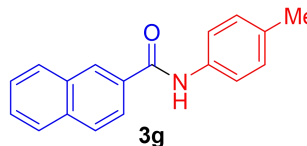
The reaction was run at 100 °C for 8 h, affording product **3e** as a white solid (82% yield, 20.1 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 2.34 (s, 3H), 7.17 (d, *J* = 8.4 Hz, 2H), 7.44-7.49 (m, 2H), 7.50 (d, *J* = 8.4 Hz, 2H), 7.76 (s, 1H), 7.79-7.82 (m, 2H); <sup>13</sup>{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) δ 164.5, 138.0, 135.0, 134.5, 133.4, 129.6, 129.0, 128.4,

120.3, 20.9; HRMS (ESI): Exact mass calcd for  $C_{14}H_{12}ClNONa$   $[M+Na]^+$ : 268.0505, Found: 268.0499.



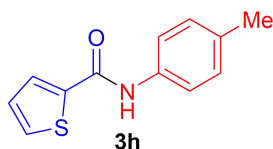
The reaction was run at 100 °C for 8 h, affording product **3f** as a white solid (71% yield, 20.5 mg).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  2.34 (s, 3H), 7.18, (d,  $J$  = 8.0 Hz, 2H), 7.50 (d,  $J$  = 8.0 Hz, 2H), 7.60-7.64 (m, 2H), 7.72-7.75 (m, 3H);  $^{13}\{^1H\}$  NMR (100 MHz,  $DMSO-d_6$ )  $\delta$  164.3, 136.4, 134.1, 132.8, 131.4, 129.8, 129.0, 125.2, 120.4,

20.5; HRMS (ESI): Exact mass calcd for  $C_{14}H_{12}BrNONa$   $[M+Na]^+$ : 312.0000, Found: 312.0015.



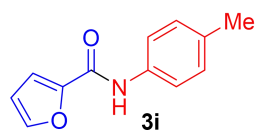
The reaction was run at 100 °C for 8 h, affording product **3g** as a white solid (62% yield, 16.2 mg).  $^1H$  NMR (400 MHz,  $CDCl_3$ ):  $\delta$  2.36 (s, 3H), 7.20 (d,  $J$  = 8.0 Hz, 2H), 7.54-7.62 (m, 4H), 7.89-7.96 (m, 5H), 8.37 (s, 1H);  $^{13}\{^1H\}$  NMR (100 MHz,  $CDCl_3$ ):  $\delta$  165.7, 135.4, 134.8, 134.3, 132.6, 132.2, 129.6, 128.9, 128.7, 127.8,

127.8, 127.4, 126.9, 123.5, 120.3, 20.9. HRMS (ESI): Exact mass calcd for  $C_{18}H_{15}NONa$   $[M+Na]^+$ : 284.1052, Found: 284.1047.



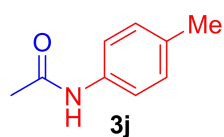
The reaction was run at 100 °C for 8 h, affording product **3h** as a yellow solid (69% yield, 15.0 mg).  $^1H$  NMR (500 MHz,  $CDCl_3$ ):  $\delta$  2.33 (s, 3H), 7.10-7.16 (m, 3H), 7.48-7.52 (m, 3H), 7.62 (s, 1H), 7.76 (s, 1H);  $^{13}\{^1H\}$  NMR (100 MHz,  $CDCl_3$ ):  $\delta$  159.9, 139.4, 135.0, 134.3, 130.5, 129.5, 128.3, 127.7, 120.3, 20.9. HRMS (ESI): Exact mass

calcd for  $C_{12}H_{11}NOSNa$   $[M+Na]^+$ : 240.0459, Found: 240.0457.



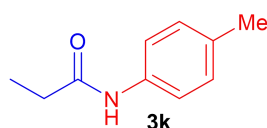
The reaction was run at 100 °C for 8 h, affording product **3i** as a yellow solid (66% yield, 13.3 mg).  $^1H$  NMR (400 MHz,  $CDCl_3$ ):  $\delta$  2.33 (s, 3H), 6.55 (dd,  $J$  = 3.6, 1.6 Hz, 1H), 7.16 (d,  $J$  = 8.0 Hz, 2H), 7.22 (dd,  $J$  = 3.6, 0.8 Hz, 1H), 7.50 (dd,  $J$  = 1.6, 0.8 Hz, 1H), 7.51-7.55 (m, 2H), 8.04 (s, 1H);  $^{13}\{^1H\}$  NMR (100 MHz,  $CDCl_3$ ):  $\delta$  156.0, 147.9, 144.0,

134.7, 134.1, 129.6, 119.9, 115.0, 112.5, 20.9. HRMS (ESI): Exact mass calcd for  $C_{12}H_{11}NO_2Na$   $[M+Na]^+$ : 224.0688, Found: 224.0681.



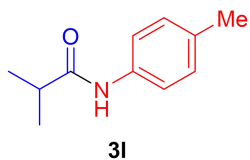
The reaction was run at 100 °C for 8 h, affording product **3j** as a white solid (61% yield, 10.1 mg).  $^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  2.12 (s, 3H), 2.30 (s, 3H), 7.09 (d,  $J$  = 8.0 Hz, 2H), 7.37 (d,  $J$  = 8.5 Hz, 2H), 7.80 (s, 1H);  $^{13}\{^1H\}$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  168.6, 135.4,

133.8, 129.3, 120.1, 24.3, 20.8; HRMS (ESI): Exact mass calcd for  $C_9H_{11}NONa$   $[M+Na]^+$ : 172.0733, Found: 172.0736.

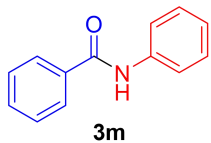


The reaction was run at 100 °C for 24 h, affording product **3k** as a white solid (57% yield, 9.3 mg).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  1.23 (t,  $J$  = 7.6 Hz, 3H), 2.30 (s, 3H), 2.36 (q,  $J$  = 7.6 Hz, 2H), 7.10 (d,  $J$  = 8.0 Hz, 2H), 7.33 (s, 1H), 7.39 (d,  $J$  = 8.0 Hz, 2H);

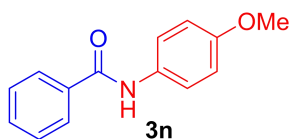
$^{13}\{^1H\}$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  172.0, 135.4, 133.7, 129.4, 119.9, 30.6, 20.8, 9.7; HRMS (ESI): Exact mass calcd for  $C_{10}H_{13}NONa$   $[M+Na]^+$ : 186.0895, Found: 186.0890.



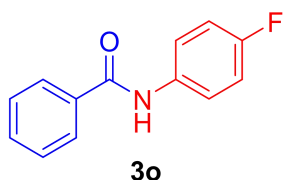
The reaction was run at 100 °C for 24 h, affording product **3l** as a white solid (52% yield, 9.2 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 1.24 (d, *J* = 6.8 Hz, 6H), 2.30 (s, 3H), 2.49 (sep, *J* = 6.8 Hz, 1H), 7.11 (d, *J* = 8.0 Hz, 2H), 7.19 (s, 1H), 7.40 (d, *J* = 8.4 Hz, 2H); <sup>13</sup>{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>): δ 175.1, 135.4, 133.7, 129.4, 119.8, 36.6, 20.8, 19.6; HRMS (ESI) Exact mass calcd for C<sub>11</sub>H<sub>15</sub>NONa [M+Na]<sup>+</sup>: 200.1052, Found: 200.1058.



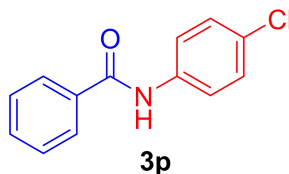
The reaction was run at 100 °C for 8 h, affording product **3m** as a yellow solid (61% yield, 12.0 mg). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 6.88 (tt, *J* = 7.0, 1.5 Hz, 1H), 6.98-7.07 (m, 6H), 7.10-7.14 (m, 1H), 7.43-7.45 (m, 2H), 12.49 (s, 1H); <sup>13</sup>{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>) δ 197.9, 157.5, 143.2, 137.3, 130.5, 129.2, 128.5, 127.6, 124.9, 122.7; HRMS (ESI): Exact mass calcd for C<sub>13</sub>H<sub>11</sub>NONa [M+Na]<sup>+</sup>: 220.0739, Found: 220.0724.



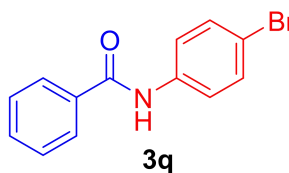
The reaction was run at 100 °C for 8 h, affording product **3n** as a yellow solid (64% yield, 14.5 mg). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 3.81 (s, 3H), 6.89 (d, *J* = 9.0 Hz, 2H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.59 – 7.49 (m, 3H), 7.85 (d, *J* = 7.0 Hz, 3H). <sup>13</sup>{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>) δ 165.7, 156.6, 135.0, 131.6, 131.0, 128.7, 127.0, 122.1, 114.2, 55.5. HRMS (ESI): Exact mass calcd for C<sub>14</sub>H<sub>13</sub>NO<sub>2</sub>Na [M+Na]<sup>+</sup>: 250.0838, Found: 250.0819.



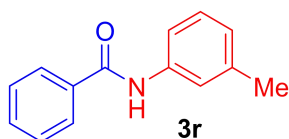
The reaction was run at 100 °C for 8 h, affording product **3o** as a white solid (68% yield, 14.6 mg). <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>) δ 7.17-7.22 (m, 2H), 7.53 (t, *J* = 7.5 Hz, 2H), 7.59 (tt, *J* = 7.5, 1.0 Hz, 1H), 7.78-7.81 (m, 2H), 7.94-7.96 (m, 2H), 10.30 (s, 1H); <sup>13</sup>{<sup>1</sup>H} NMR (125 MHz, DMSO-*d*<sub>6</sub>) δ 165.5, 159.2, 158.3 (d, *J* = 239 Hz), 134.8, 131.6, 128.4, 127.6, 122.2 (d, *J* = 7.8 Hz), 115.2 (d, *J* = 22 Hz); <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) -117.60. HRMS (ESI): Exact mass calcd for C<sub>13</sub>H<sub>10</sub>FNONa [M+Na]<sup>+</sup>: 238.0644, Found: 238.0638.



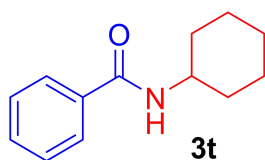
The reaction was run at 100 °C for 8 h, affording product **3p** as a white solid (72% yield, 16.6 mg). <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>): δ 7.51-7.55 (m, 4H), 7.58-7.62 (m, 1H), 7.75-7.79 (m, 2H), 7.93-7.96 (m, 2H), 10.37 (s, 1H); <sup>13</sup>{<sup>1</sup>H} NMR (100 MHz, DMSO-*d*<sub>6</sub>): δ 165.6, 138.6, 134.7, 131.7, 131.4, 128.4, 127.7, 122.2, 115.3. HRMS (ESI): Exact mass calcd for C<sub>13</sub>H<sub>10</sub>ClNONa [M+Na]<sup>+</sup>: 254.0349, Found: 254.0444.



The reaction was run at 100 °C for 8 h, affording product **3q** as a white solid (51% yield, 12.8 mg). <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 7.51-7.55 (m, 4H), 7.58-7.62 (m, 1H), 7.75-7.79 (m, 2H), 7.93-7.96 (m, 2H), 10.37 (s, 1H); <sup>13</sup>{<sup>1</sup>H} NMR (125 MHz, DMSO-*d*<sub>6</sub>) δ 165.7, 138.6, 134.7, 131.7, 131.4, 128.4, 127.7, 122.2, 115.3; HRMS (ESI): Exact mass calcd for C<sub>13</sub>H<sub>10</sub>BrNONa [M+Na]<sup>+</sup>: 297.9838 Found: 297.9844.

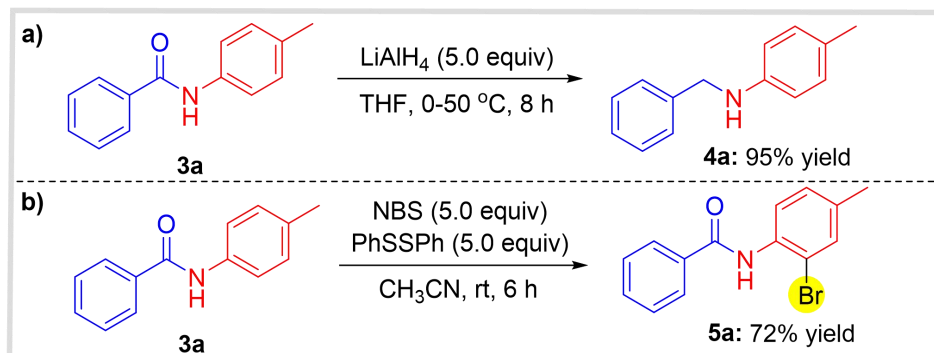


The reaction was run at 100 °C for 24 h, affording product **3r** as a white solid (76% yield, 16.0 mg). <sup>1</sup>H NMR (500 MHz, Chloroform-*d*) δ 8.12 – 8.05 (m, 1H), 7.85 (d, *J* = 7.6 Hz, 2H), 7.51 (d, *J* = 3.9 Hz, 2H), 7.46 – 7.40 (m, 3H), 7.22 (t, *J* = 7.8 Hz, 1H), 6.96 (d, *J* = 7.4 Hz, 1H), 2.33 (s, 3H). <sup>13</sup>{<sup>1</sup>H} NMR (126 MHz, Chloroform-*d*) δ 165.9, 138.9, 137.8, 134.9, 131.7, 130.0, 128.8, 128.6, 128.3, 127.0, 125.3, 121.0, 117.4, 21.4. HRMS (ESI): Exact mass calcd for C<sub>14</sub>H<sub>13</sub>NONa [M+Na]<sup>+</sup>: 234.0889, Found: 234.0880.

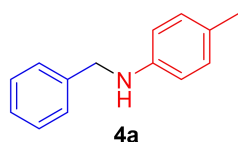


The reaction was run at 130 °C for 24 h, affording product **3t** as a white solid (37% yield, 7.5 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  1.17-1.28 (m, 3H), 1.36-1.47 (m, 2H), 1.62-1.67 (m, 1H), 1.72-1.77 (m, 2H), 2.00-2.04 (m, 2H), 3.92-4.02 (m, 1H), 6.04 (s, 1H), 7.38-7.42 (m, 2H), 7.45-7.49 (m, 1H), 7.73-7.75 (m, 2H);  $^{13}\{^1\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  166.6, 135.0, 131.2, 128.5, 126.8, 48.6, 33.2, 25.5, 24.9; HRMS (ESI): Exact mass calcd for  $\text{C}_{13}\text{H}_{17}\text{NONa}$   $[\text{M}+\text{Na}]^+$ : 226.1202, Found: 226.1208.

### 3. Further transformations of product 3a.

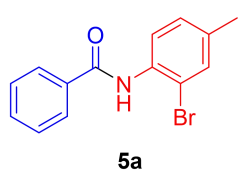


**The preparation of 4a:** To a solution of **3a** (400 mg, 1.9 mmol) in THF (2 mL) was added LiAlH<sub>4</sub> (3.0 mmol). The reaction mixture was stirred at 0 °C for 4 h and then at 50 °C for 2 h. The mixture was cooled to rt and carefully quenched with H<sub>2</sub>O (2 mL), 6 N NaOH (1 mL) and H<sub>2</sub>O (2 mL). The resulting slurry was extracted with EtOAc (20 mL × 3) and the combined organic layers were washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated to afford **4a** (355 mg, 95%) as a yellowish powder.



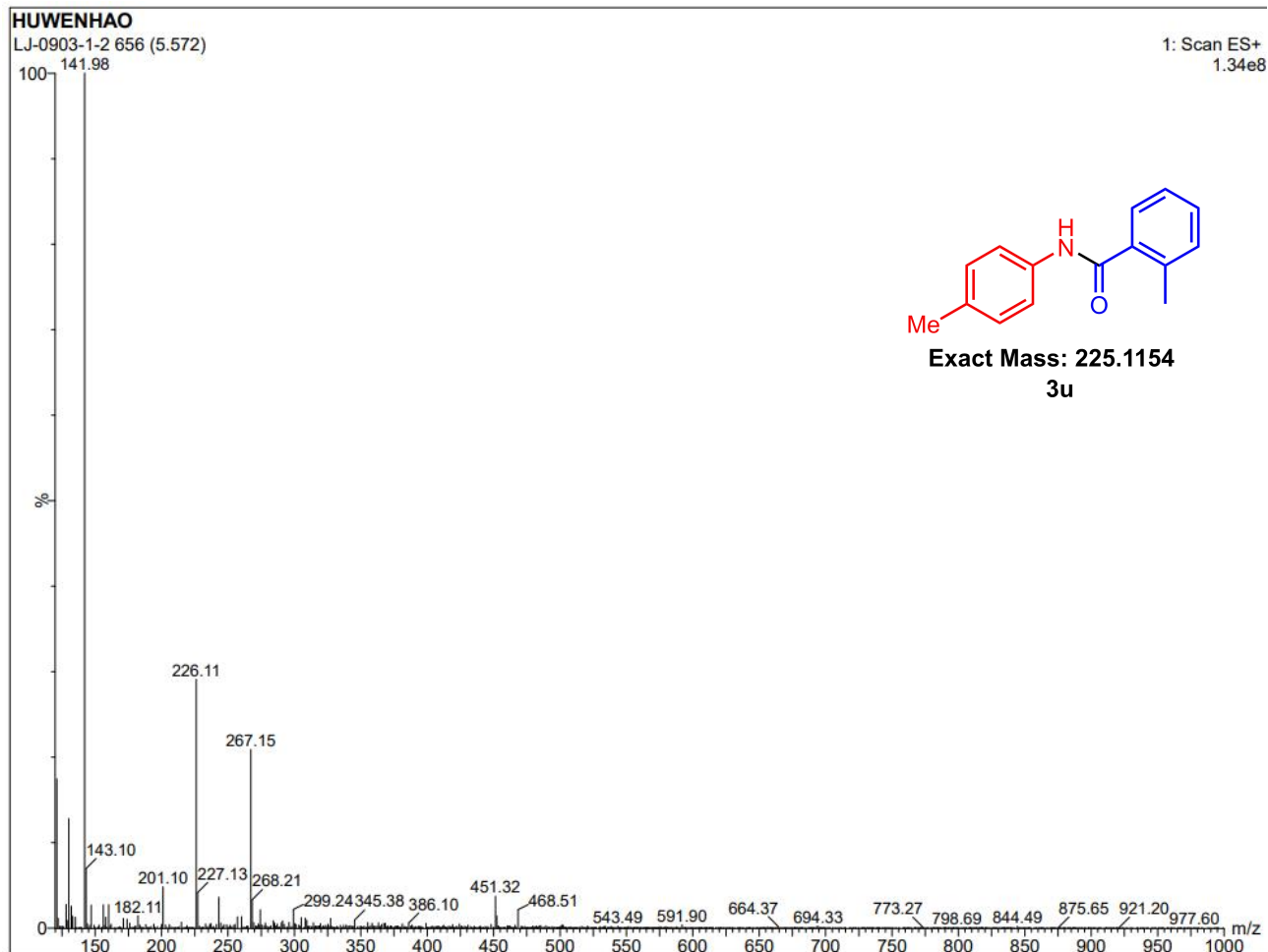
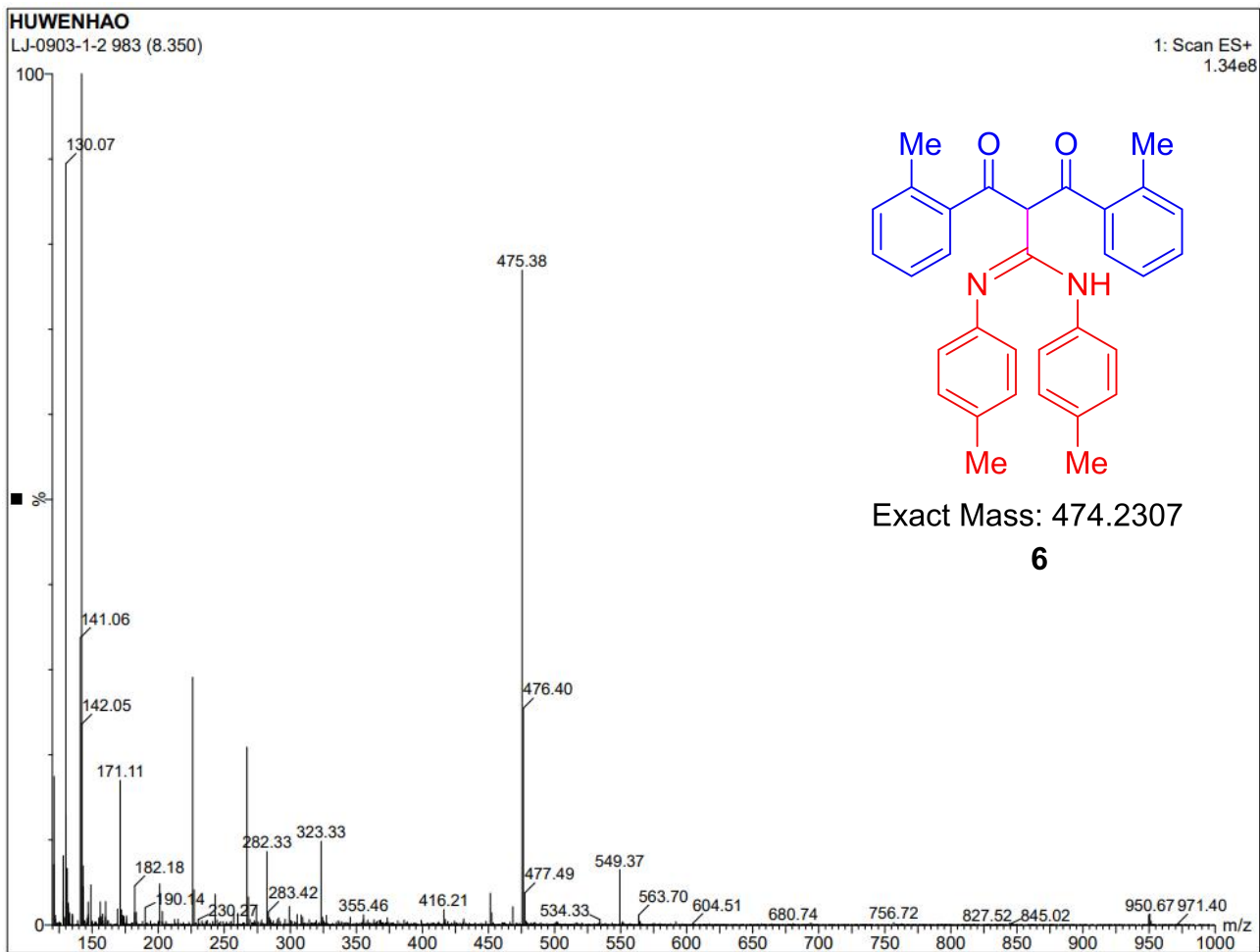
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 2.15 (s, 3H), 3.62 (s, 1H), 4.21 (s, 2H), 6.48 (d, *J* = 8.4 Hz, 2H), 6.89 (d, *J* = 8.0 Hz, 2H), 7.13-7.28 (m, 5H); <sup>13</sup>{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>) δ 145.7, 139.5, 129.7, 128.5, 127.5, 127.1, 126.8, 113.1, 48.7, 20.4. HRMS (ESI): Exact mass calcd for C<sub>14</sub>H<sub>15</sub>NNa [M+Na]<sup>+</sup>: 220.1097, Found: 220.1095.

**The preparation of 5a:** To a solution of **3a** (42.2 mg, 0.2 mmol) in CH<sub>3</sub>CN (1.00 mL) was added PhSSPh (13.2 mg, 0.06 mmol) and NBS (106.7 mg, 0.6 mmol), and the resulting solution was stirred at room temperature for 6 h. After completion of the reaction, a saturated NaHCO<sub>3</sub> and Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>(1:1) aqueous solution was added to the resultant solution, and the mixture was extracted with EtOAc. The organic layer was washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, and evaporated in vacuo. The residue was purified by column chromatography on a silica gel (PE/EtOAc = 10/1 to 4/1) to give **5a** (41.6 mg, 72%).



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 2.32 (s, 3H), 7.16 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.39 (d, *J* = 1.2 Hz, 1H), 7.48-7.53 (m, 2H), 7.55-7.59 (m, 1H), 7.92-7.94 (m, 2H), 8.38-8.40 (m, 2H); <sup>13</sup>{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>) δ 165.1, 135.3, 134.6, 133.2, 132.4, 132.0, 129.0, 128.8, 127.0, 121.6, 113.6, 20.5. HRMS (ESI): Exact mass calcd for C<sub>14</sub>H<sub>12</sub>BrNONa [M+Na]<sup>+</sup>: 311.9994 Found: 311.9982.

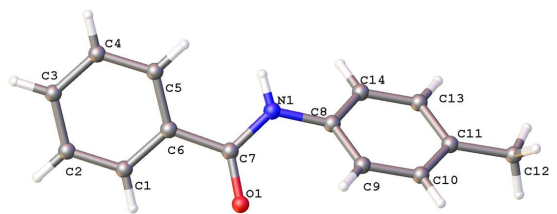
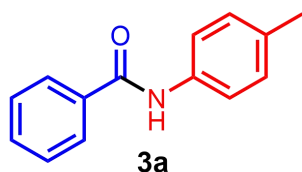
#### 4. Mechanistic studies.





## 5. X-Ray crystallographic data for compounds 3a.

Data intensity of compound **3a** was collected using a Bruker 'Bruker APEX-II CCD' diffractometer at 179.99 (10) K. Data collection and reduction were done by using Olex2 and the structure was solved with the ShelXS structure solution program using direct methods and refined by full-matrix least-squares on  $F^2$  with anisotropic displacement parameters for non-H atoms using SHELX-97. Hydrogen atoms were added at their geometrically ideal positions and refined isotropically. CCDC 2390753.



X-ray structure of product **3a**  
CCDC 2390753

Empirical formula	C <sub>14</sub> H <sub>13</sub> NO
Formula weight	211.25
Temperature/K	149.99(10)
Crystal system	orthorhombic
Space group	Pbca
a/Å	9.1158(10)
b/Å	9.8374(13)
c/Å	25.365(3)
α/°	90
β/°	90
γ/°	90
Volume/Å <sup>3</sup>	2274.6(5)
Z	8
ρ <sub>calc</sub> /g/cm <sup>3</sup>	1.234
μ/mm <sup>-1</sup>	0.613
F(000)	896.0
Crystal size/mm <sup>3</sup>	0.15 × 0.12 × 0.11
Radiation	Cu Kα (λ = 1.54184)
2θ range for data collection/°	6.97 to 142.38
Index ranges	-7 ≤ h ≤ 11, -9 ≤ k ≤ 11, -30 ≤ l ≤ 29
Reflections collected	7979
Independent reflections	2090 [R <sub>int</sub> = 0.0704, R <sub>sigma</sub> = 0.0615]
Data/restraints/parameters	2090/0/146
Goodness-of-fit on F <sup>2</sup>	1.111
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0771, wR <sub>2</sub> = 0.2090
Final R indexes [all data]	R <sub>1</sub> = 0.1071, wR <sub>2</sub> = 0.2623
Largest diff. peak/hole / e Å <sup>-3</sup>	0.33/-0.39

6) Copies of  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra

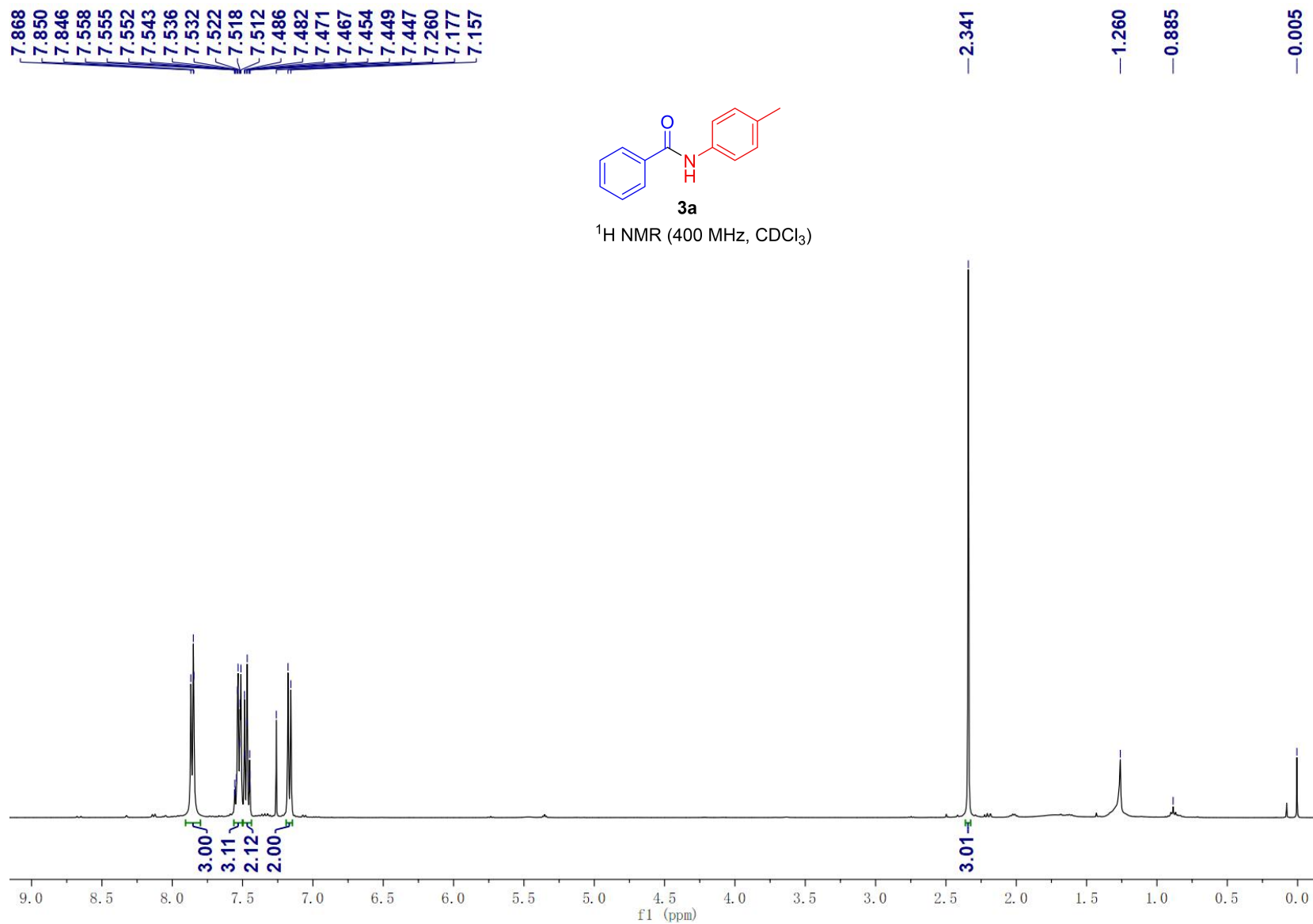


Figure 1:  $^1\text{H}$  NMR spectrum of product **3a**

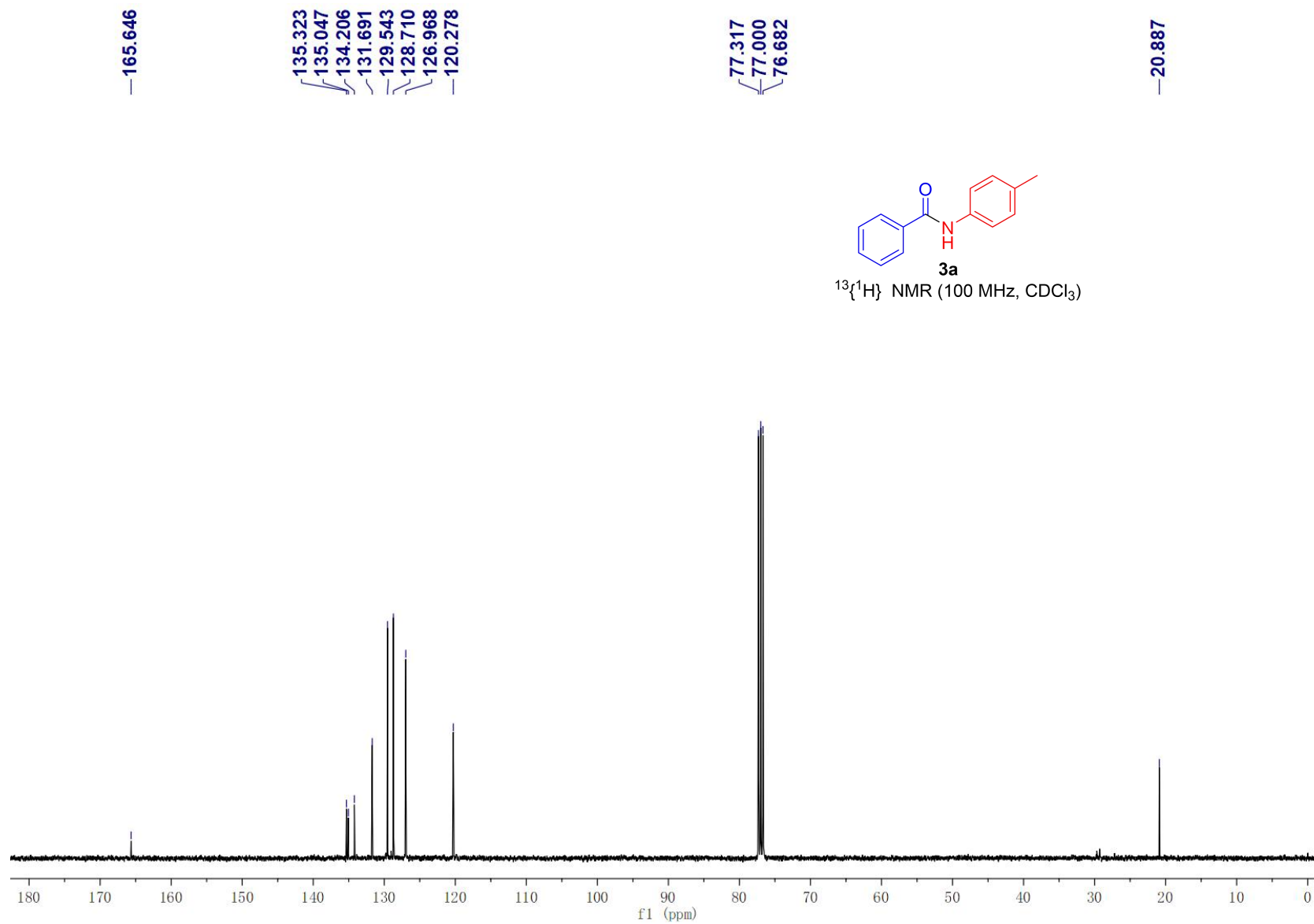


Figure 2:  $^{13}\text{C}$  NMR spectrum of product **3a**

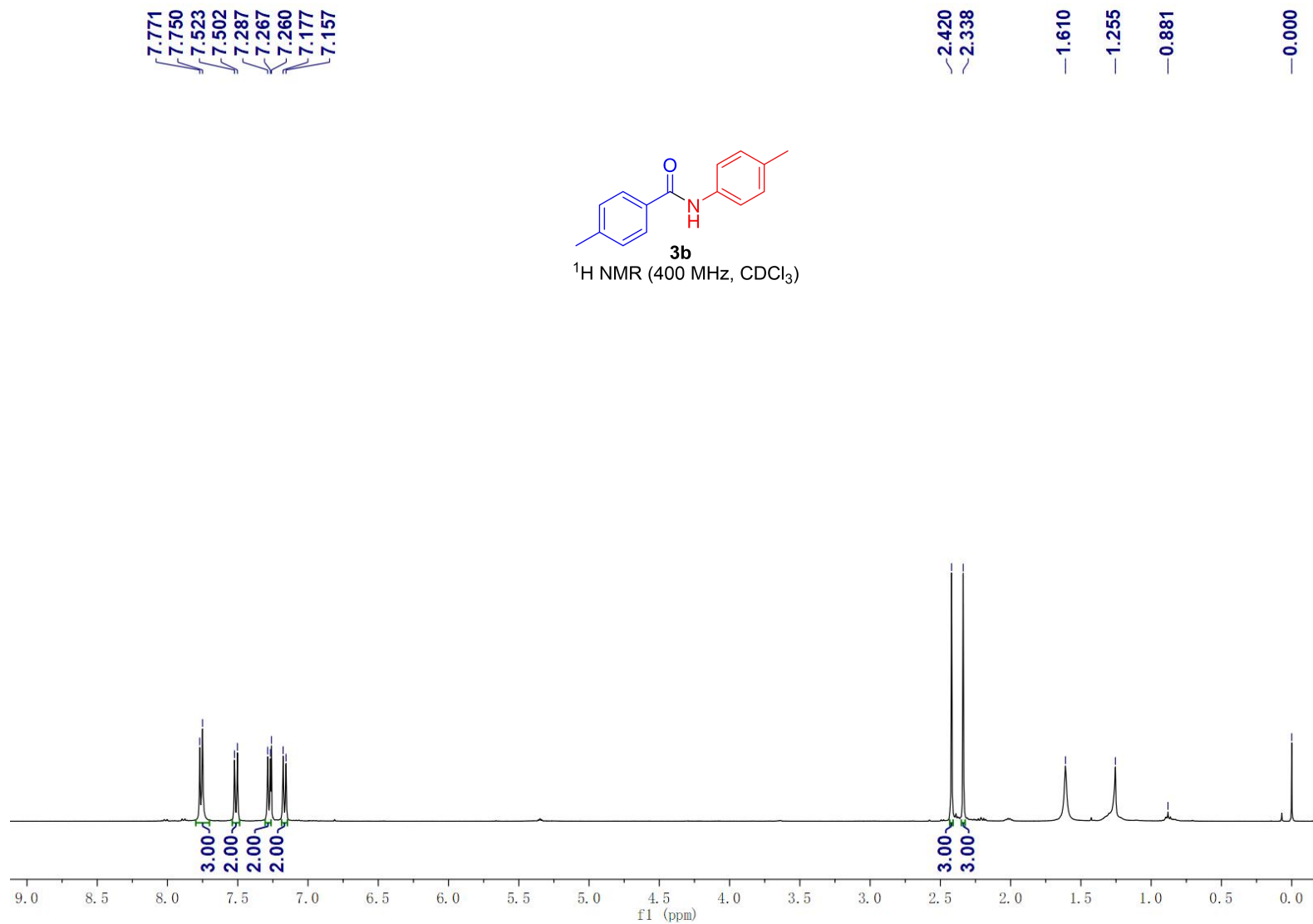
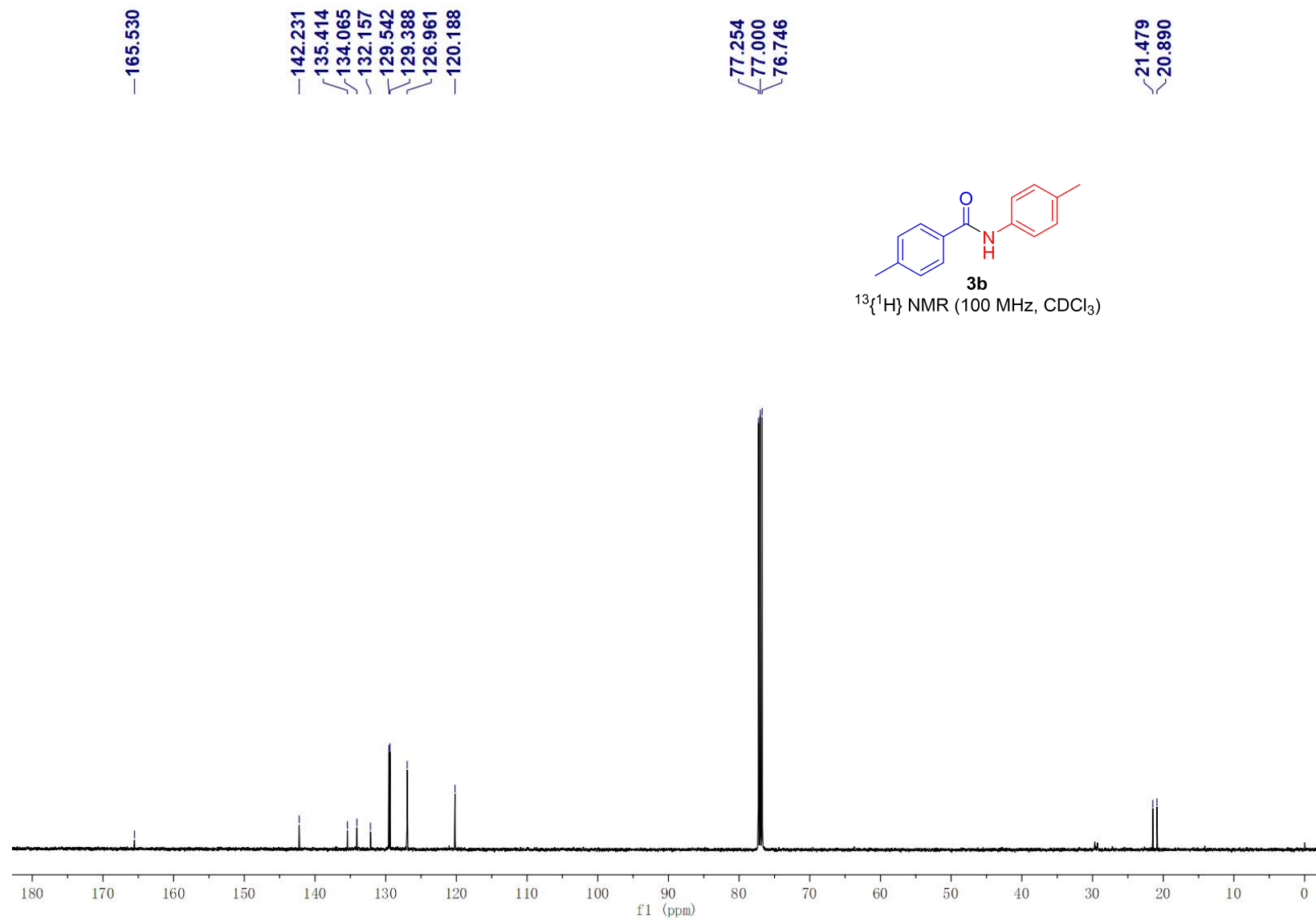


Figure 3:  $^1\text{H}$  NMR spectrum of product **3b**



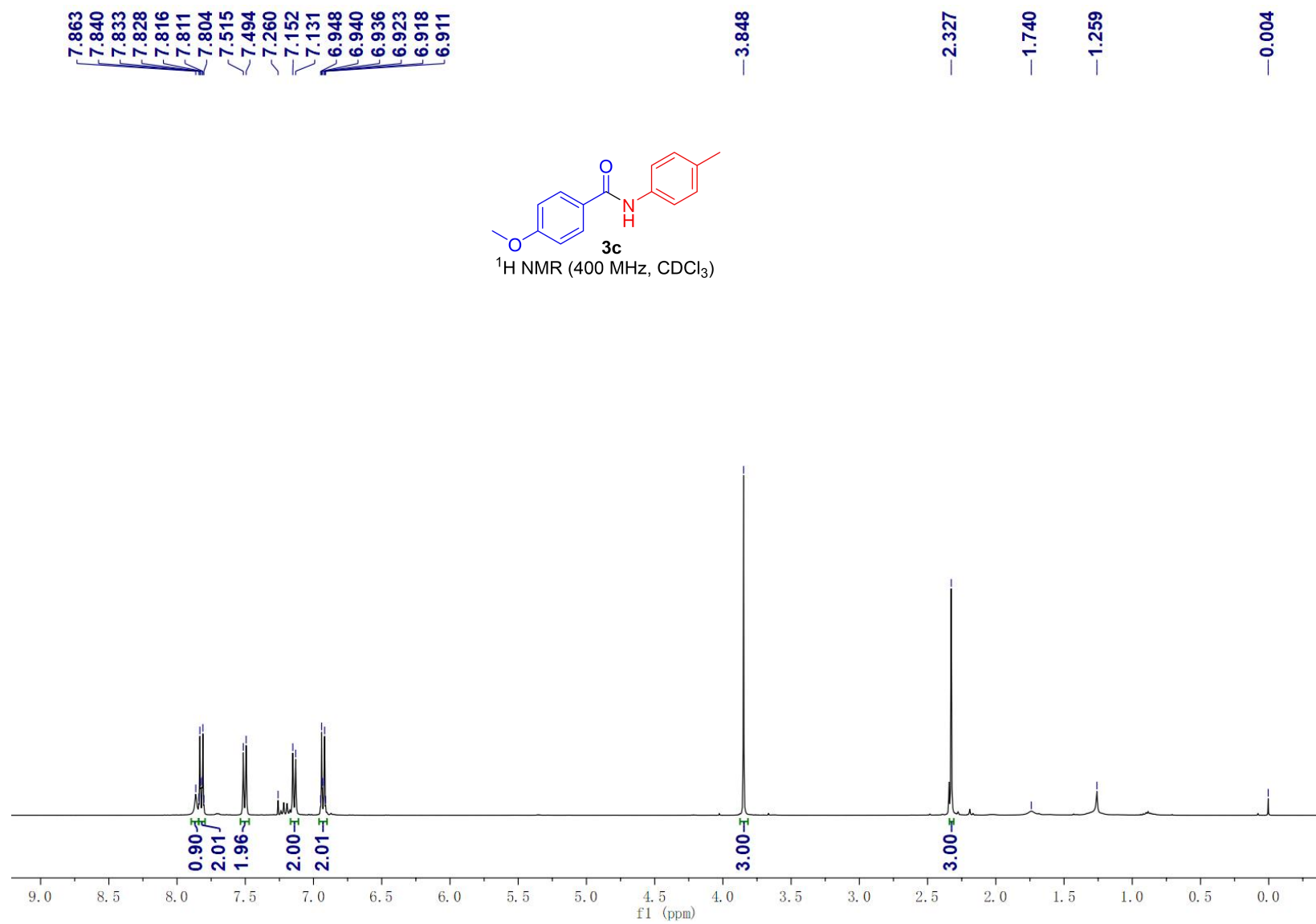


Figure 5:  $^1\text{H}$  NMR spectrum of product **3c**

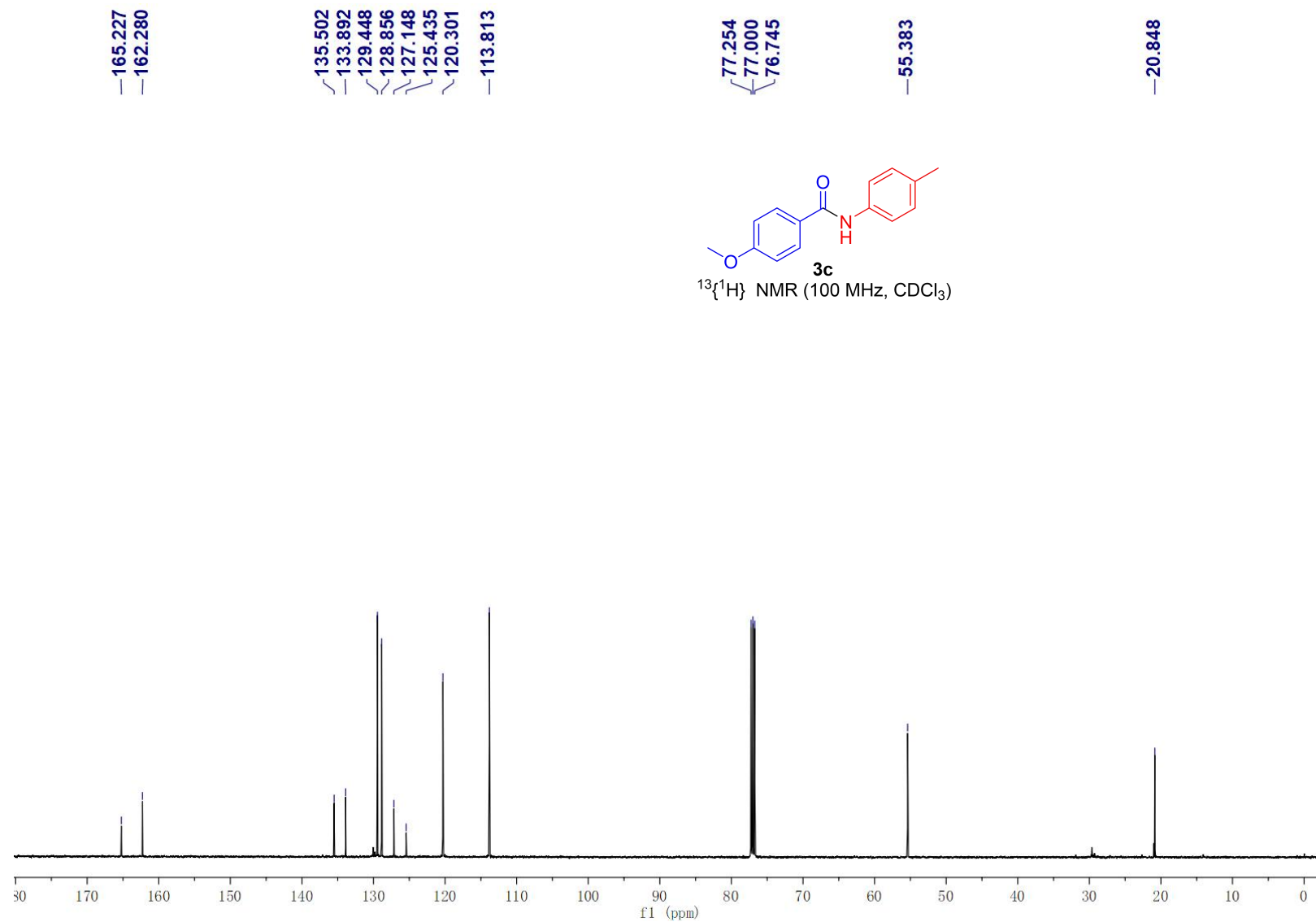


Figure 6: <sup>13</sup>C NMR spectrum of product **3c**

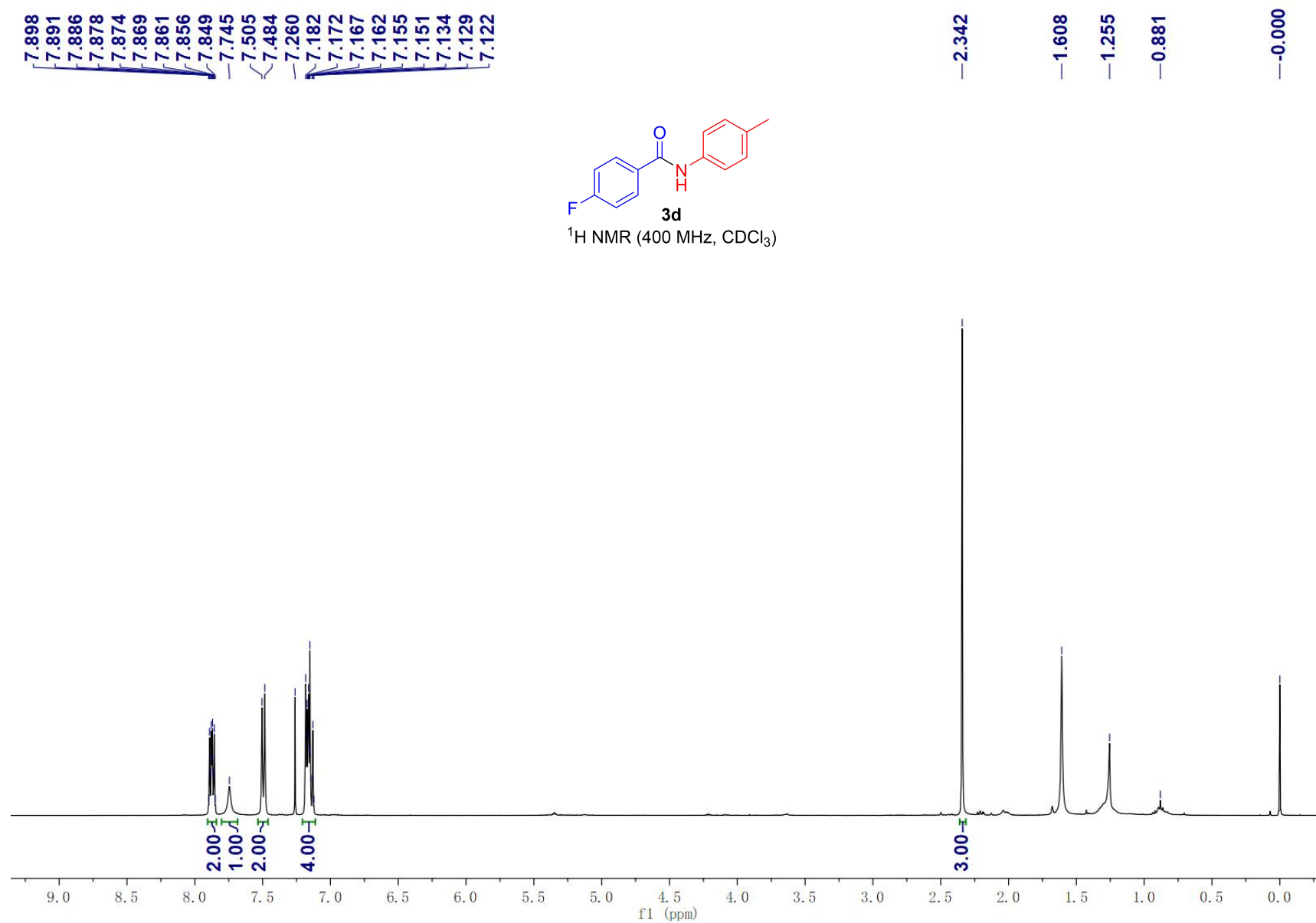


Figure 7: <sup>1</sup>H NMR spectrum of product **3d**



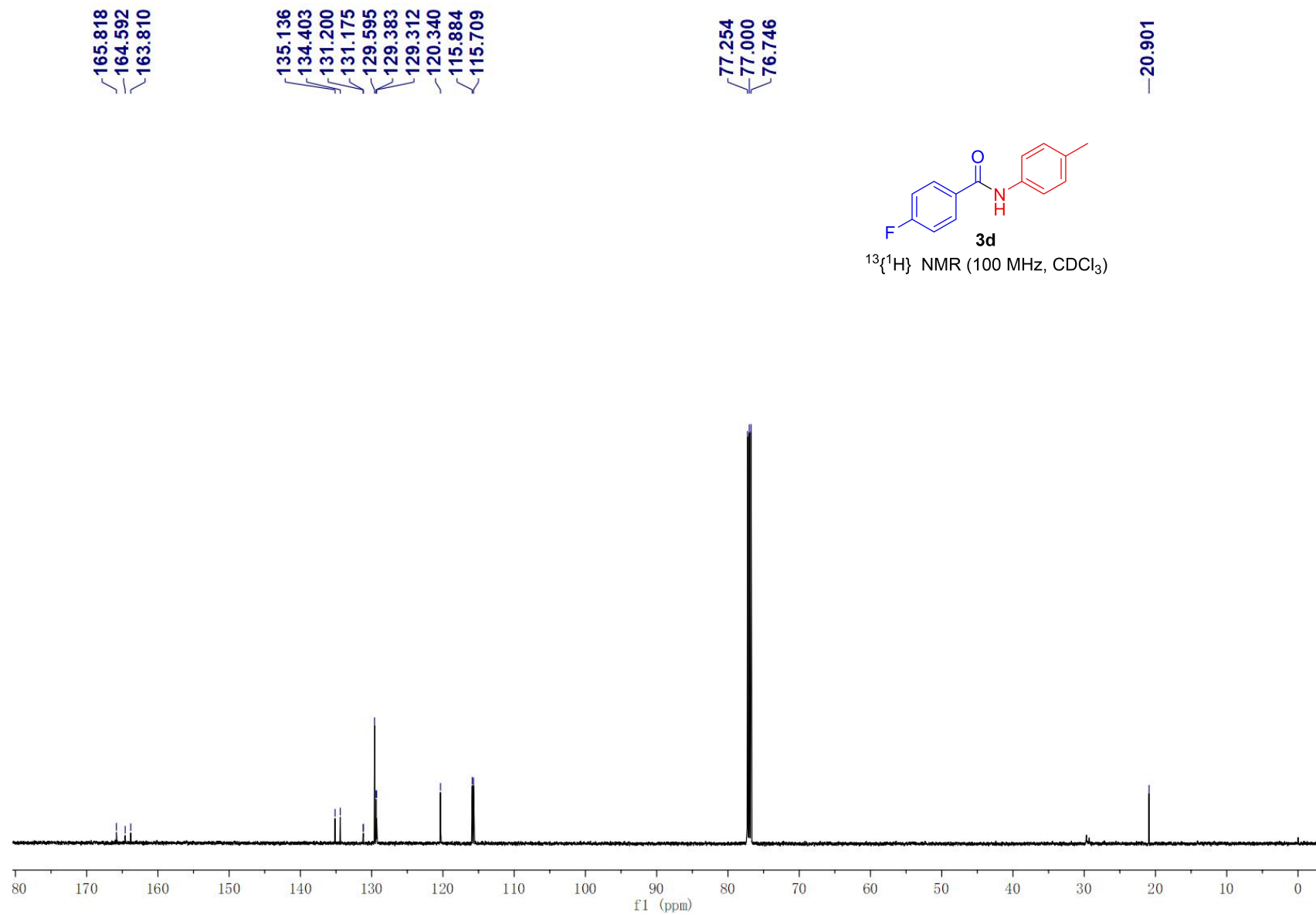


Figure 8: <sup>13</sup>C NMR spectrum of product **3d**

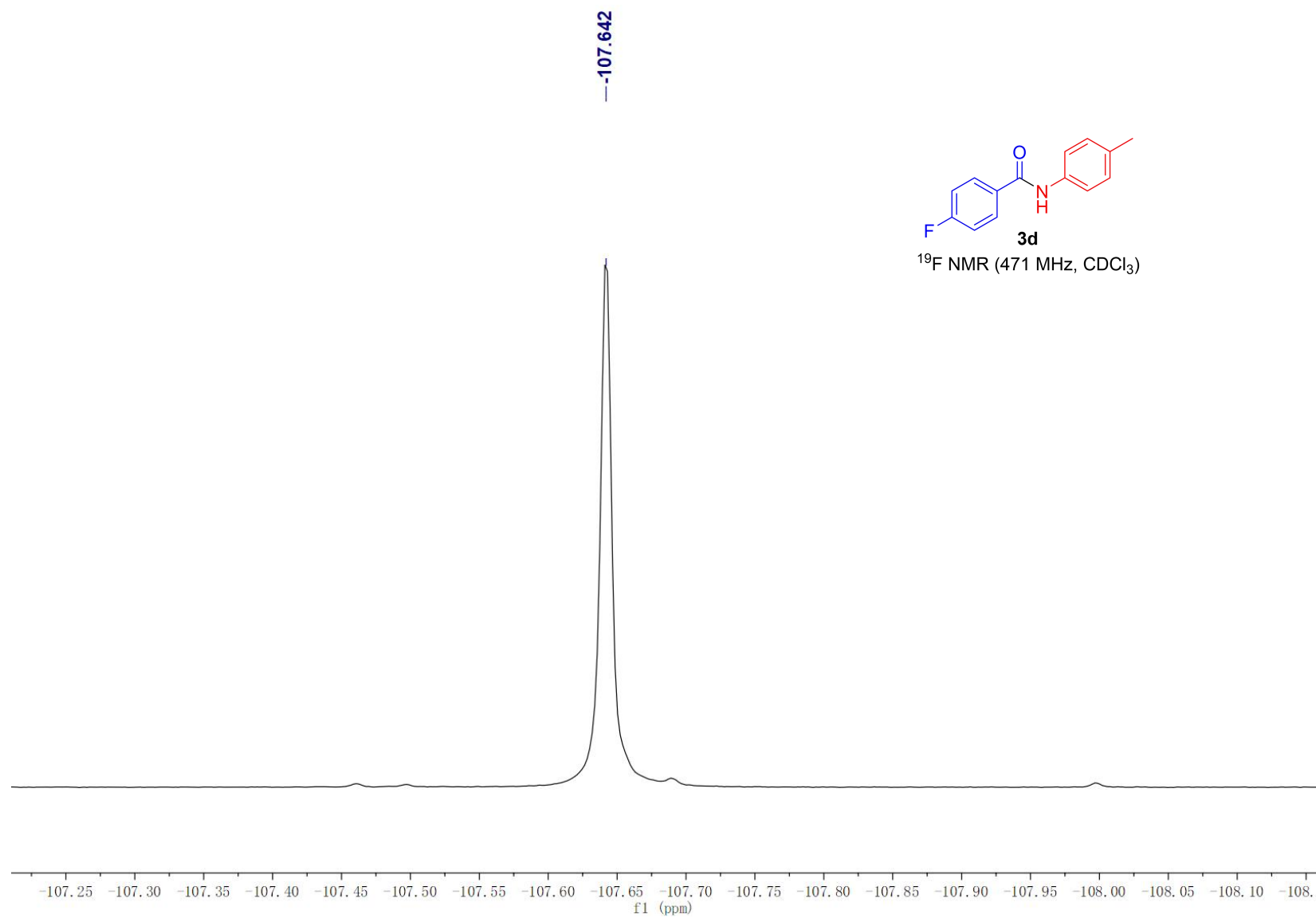


Figure 9:  $^{19}\text{F}$  NMR spectrum of product **3d**

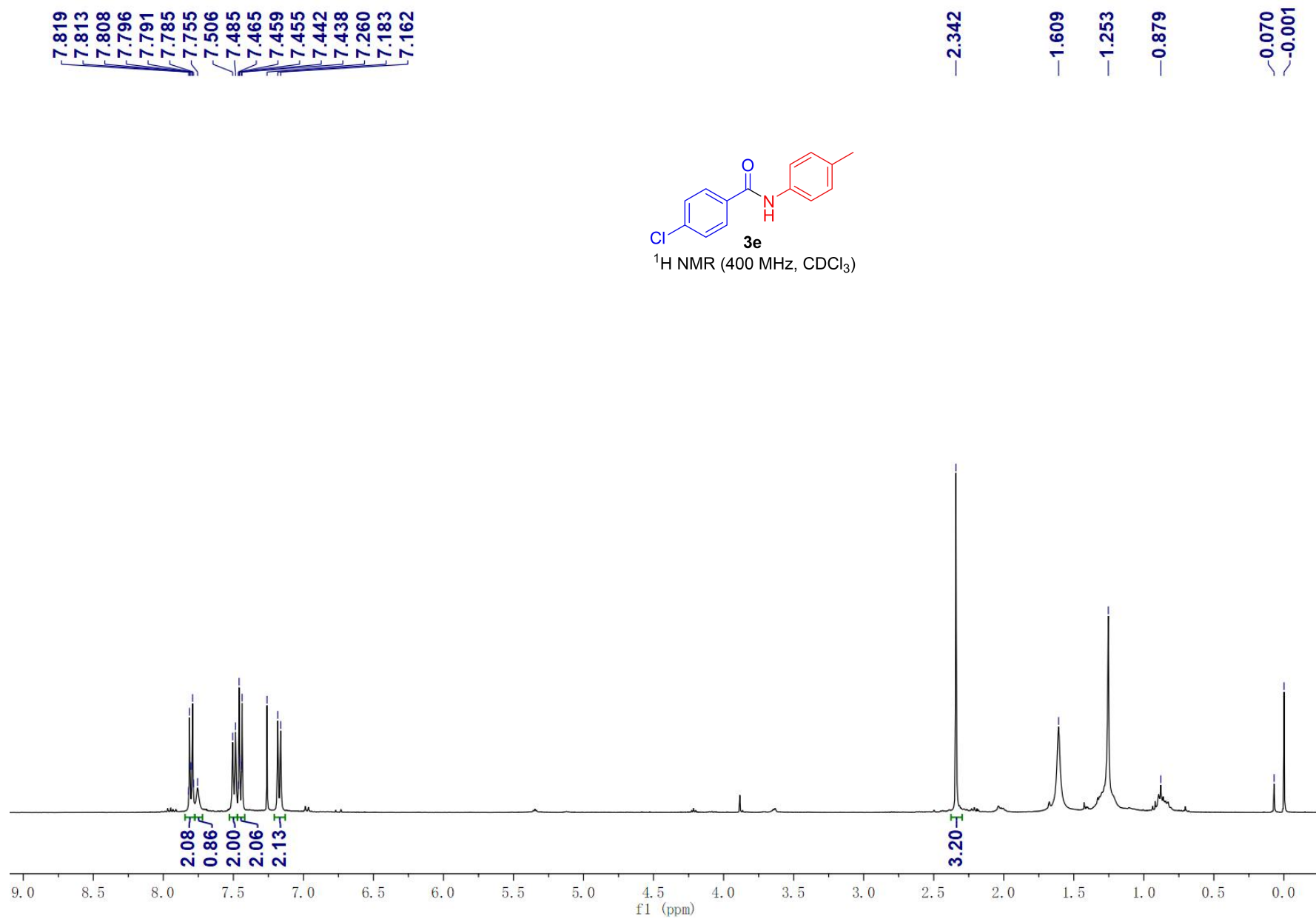


Figure 10: <sup>1</sup>H NMR spectrum of product **3e**

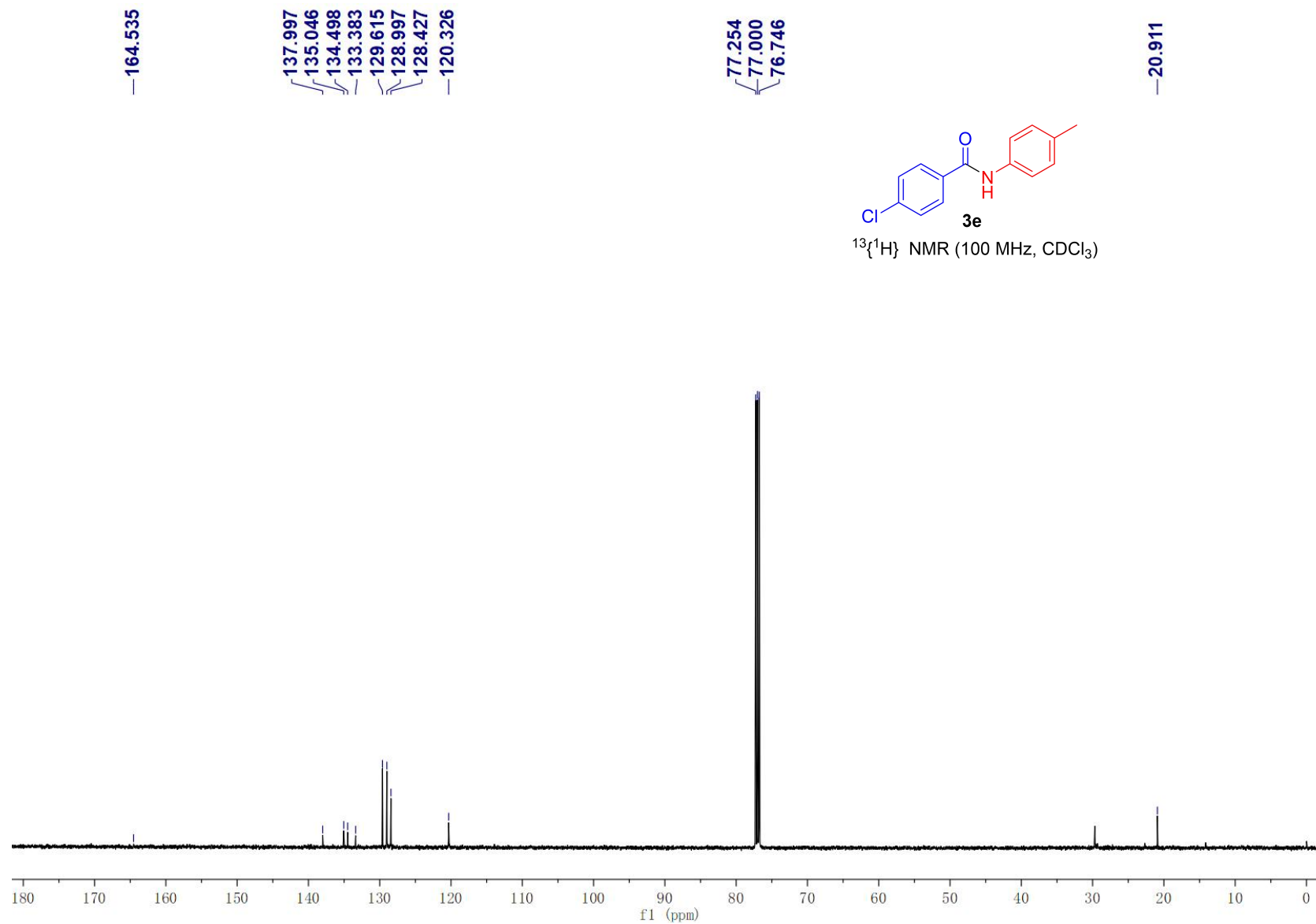


Figure 11: <sup>13</sup>C NMR spectrum of product 3e

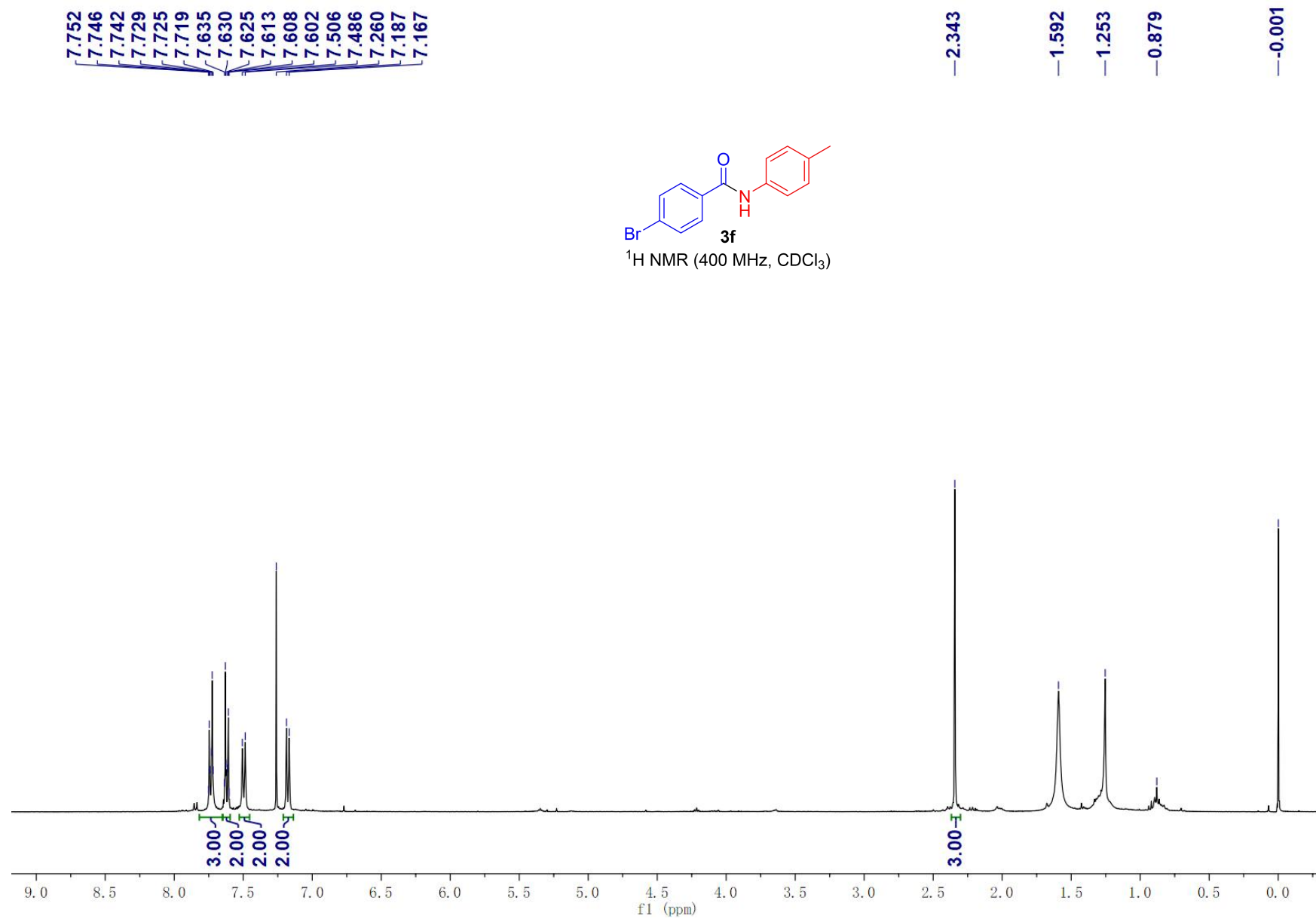


Figure 12: <sup>1</sup>H NMR spectrum of product **3f**

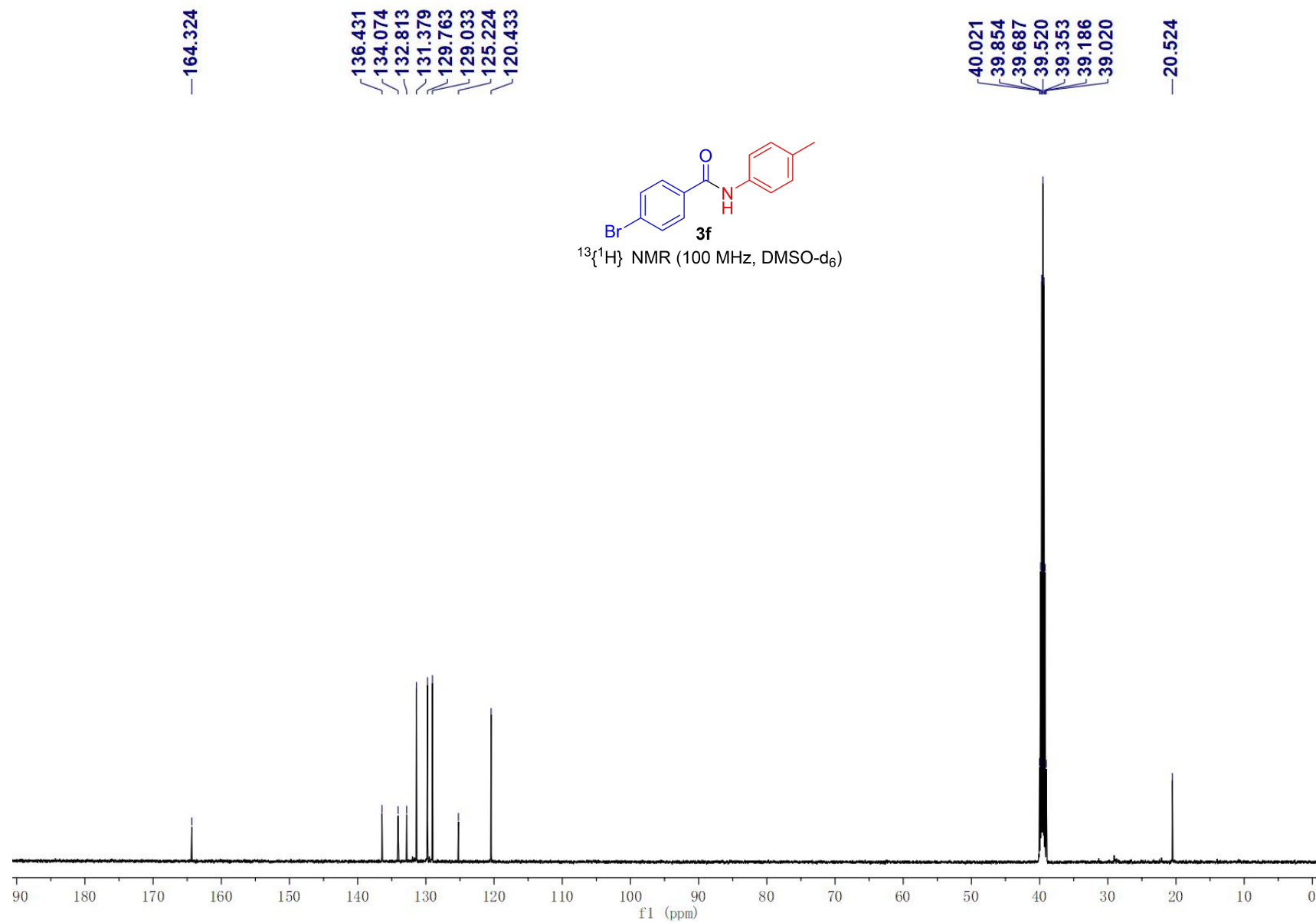


Figure 13:  $^{13}\text{C}$  NMR spectrum of product **3f**

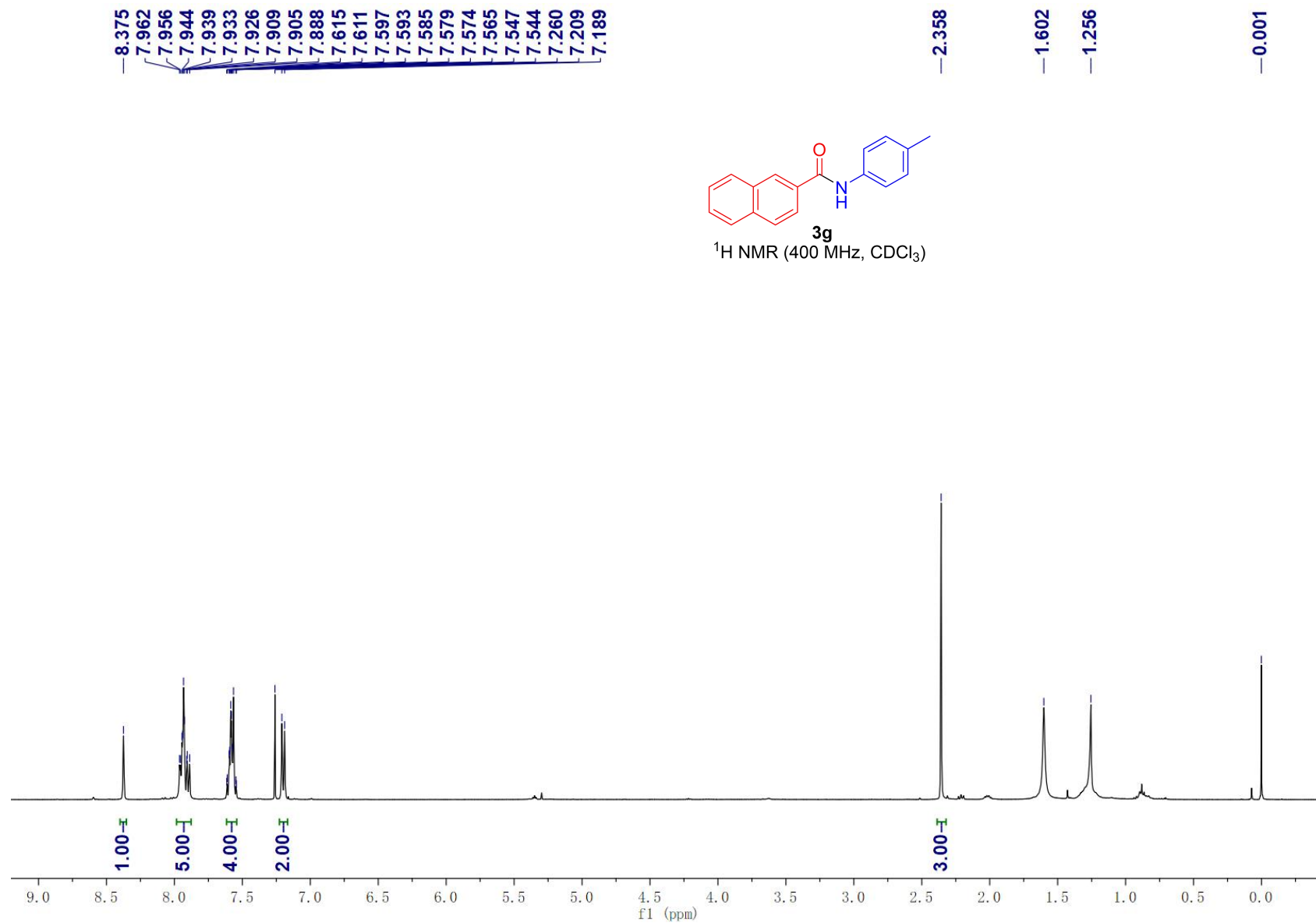


Figure 14: <sup>1</sup>H NMR spectrum of product **3g**

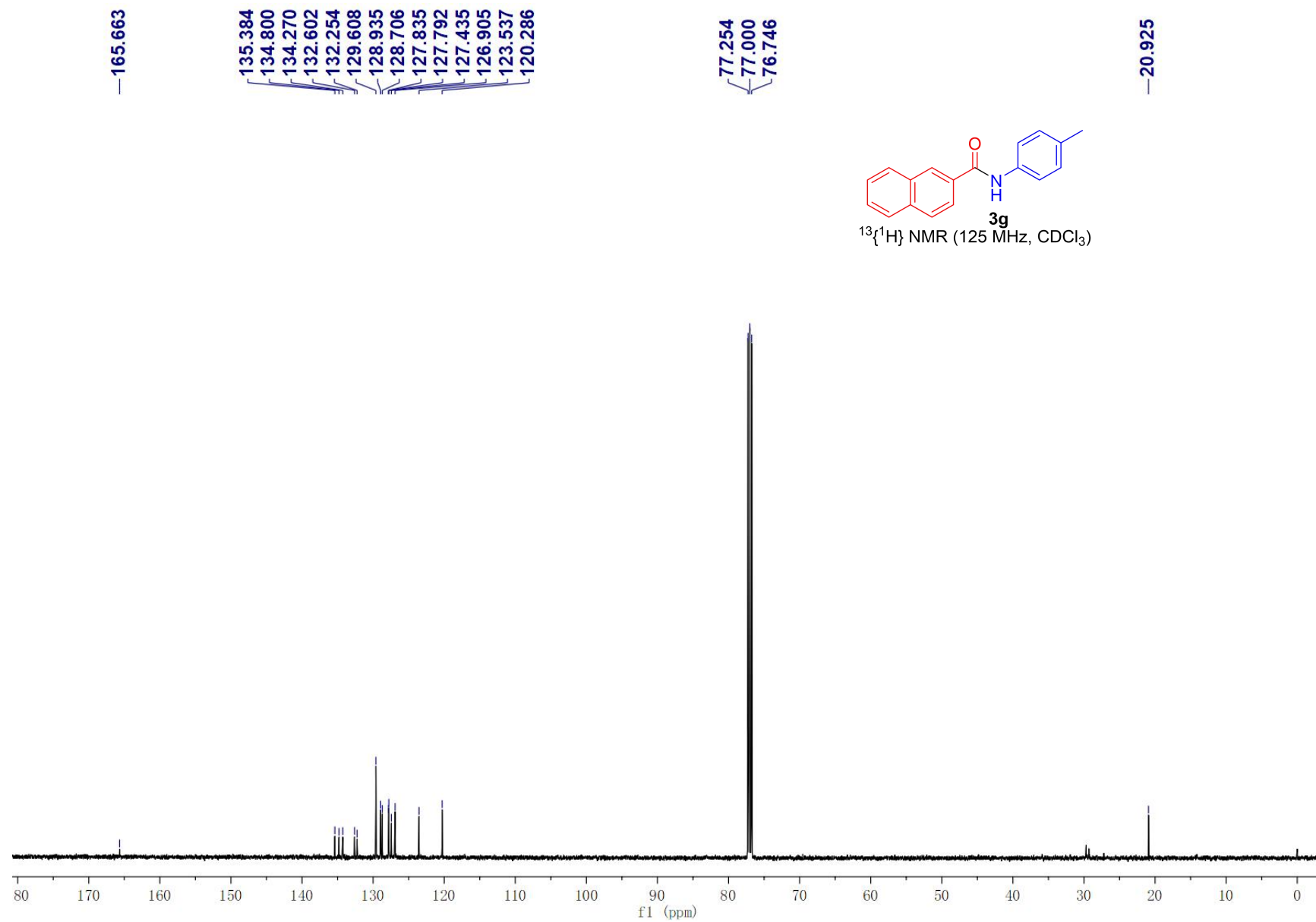


Figure 15:  $^{13}\text{C}$  NMR spectrum of product **3g**



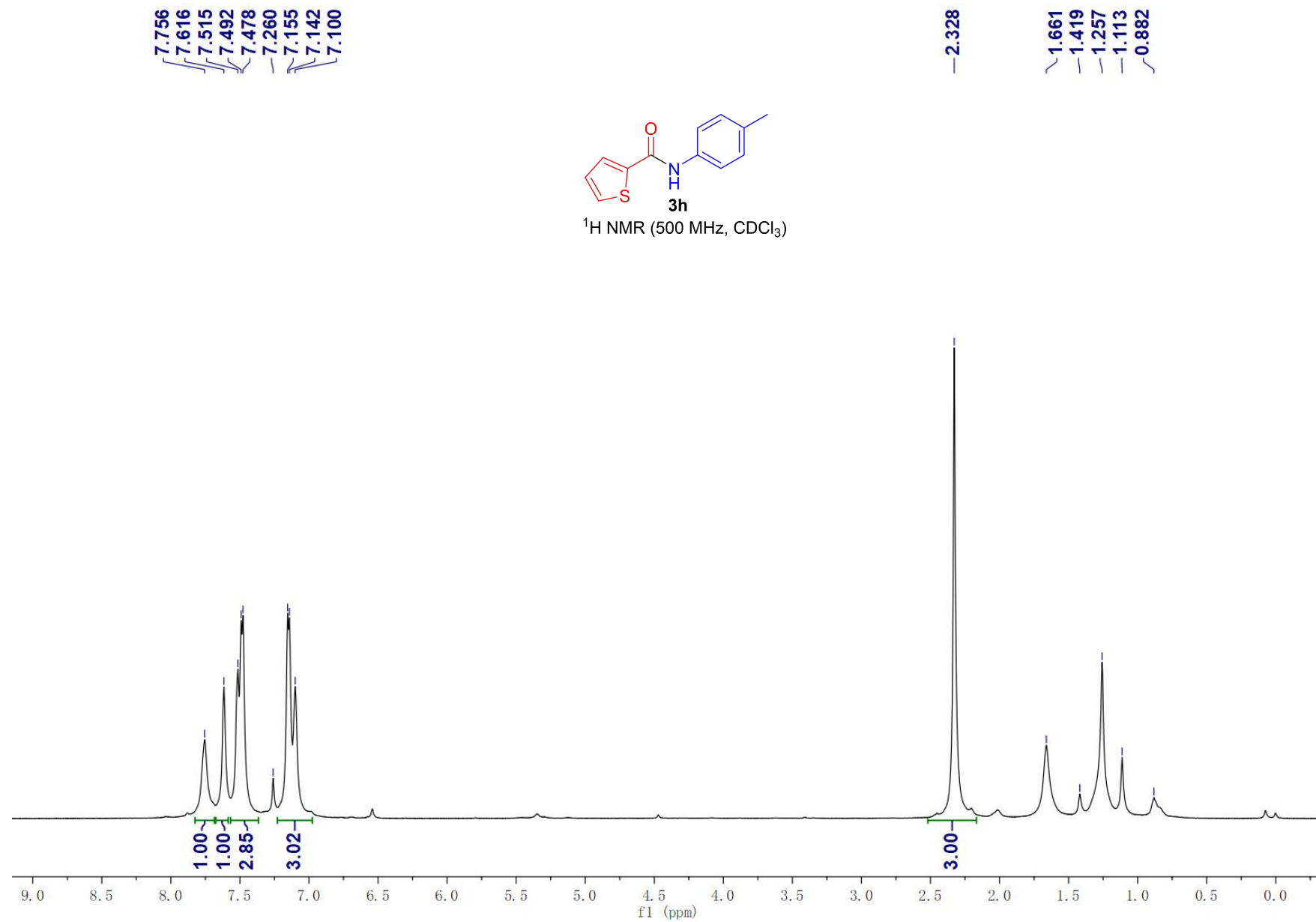


Figure 16: <sup>1</sup>H NMR spectrum of product **3h**

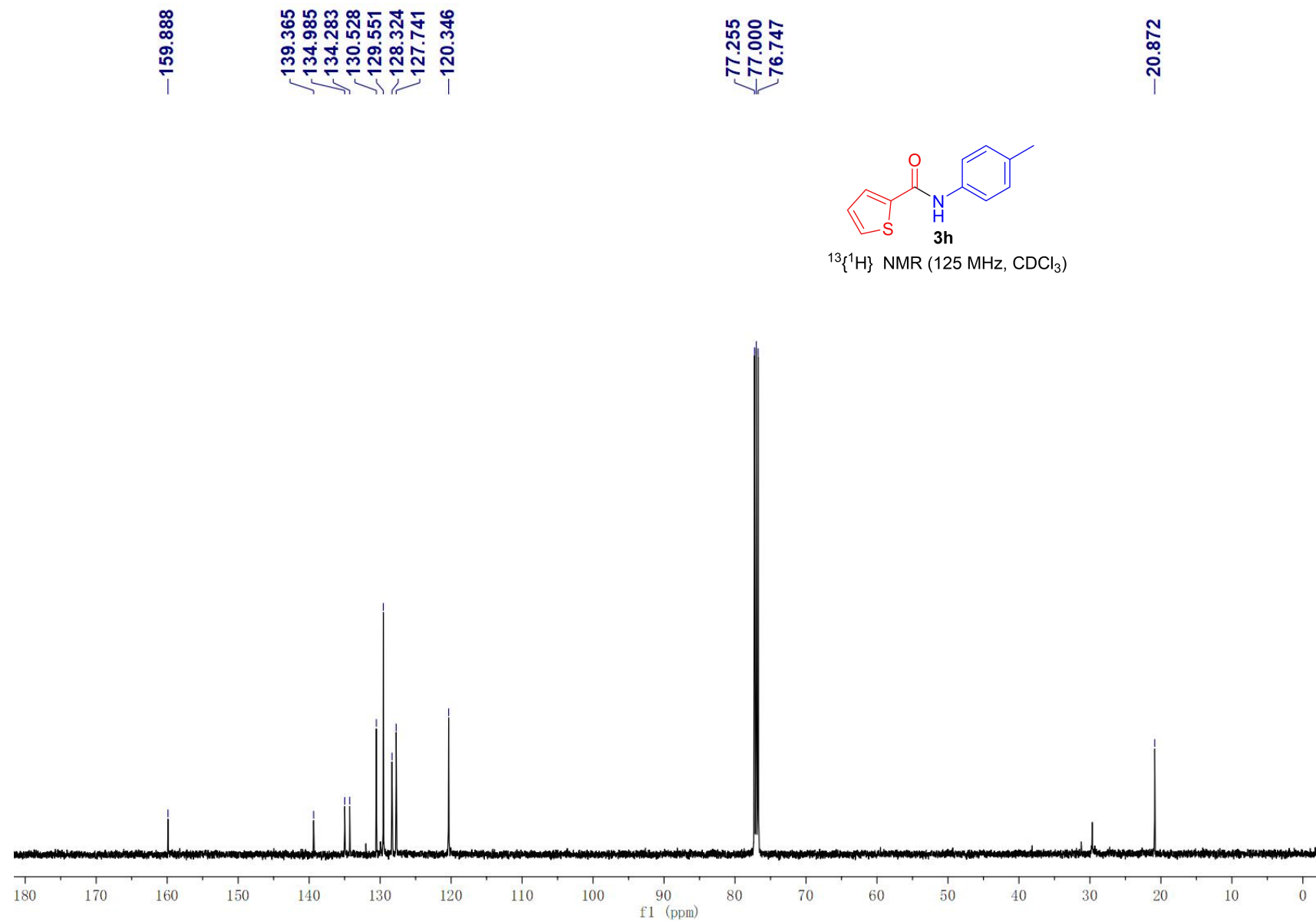


Figure 17: <sup>13</sup>C NMR spectrum of product **3h**

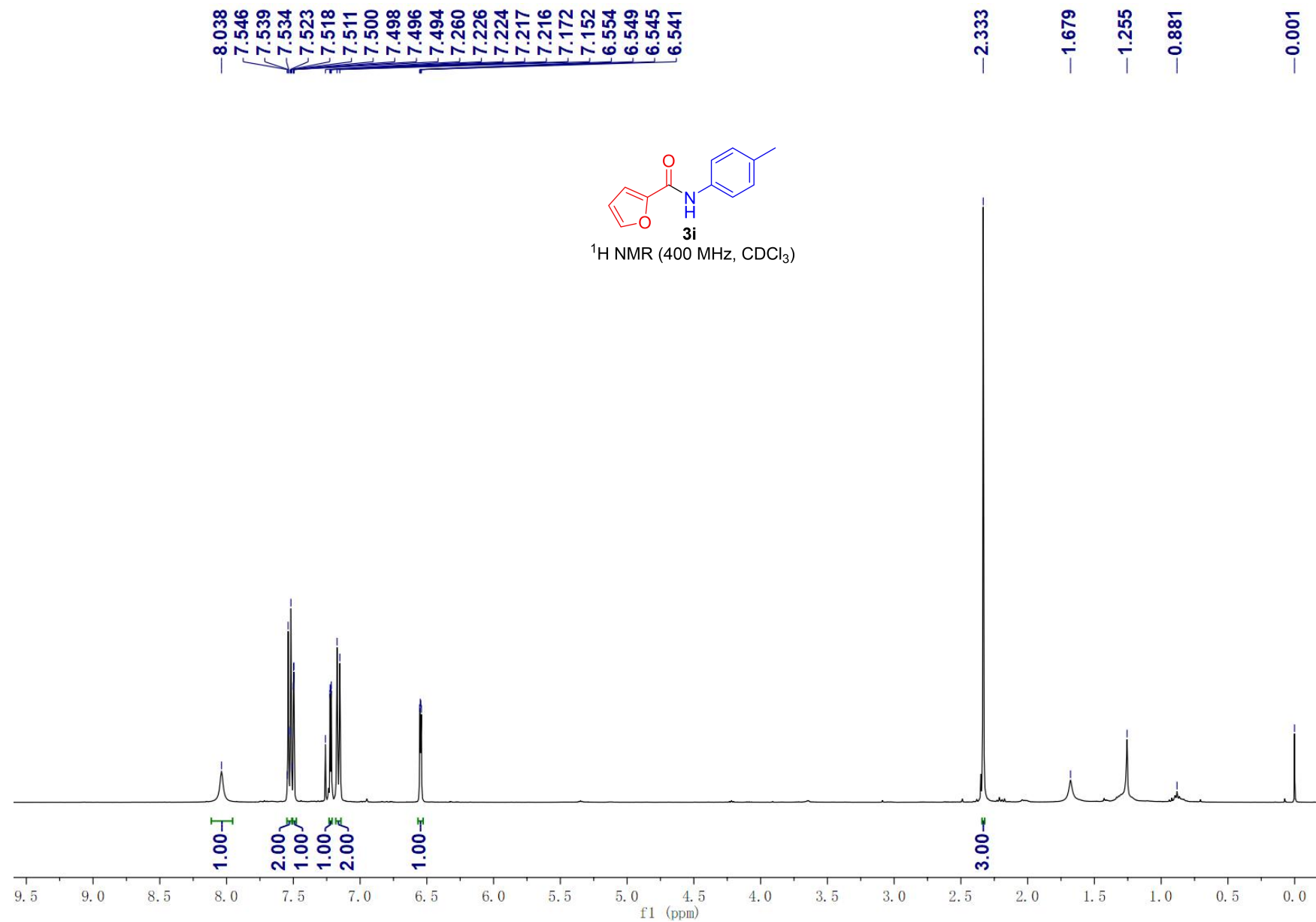


Figure 18: <sup>1</sup>H NMR spectrum of product **3i**

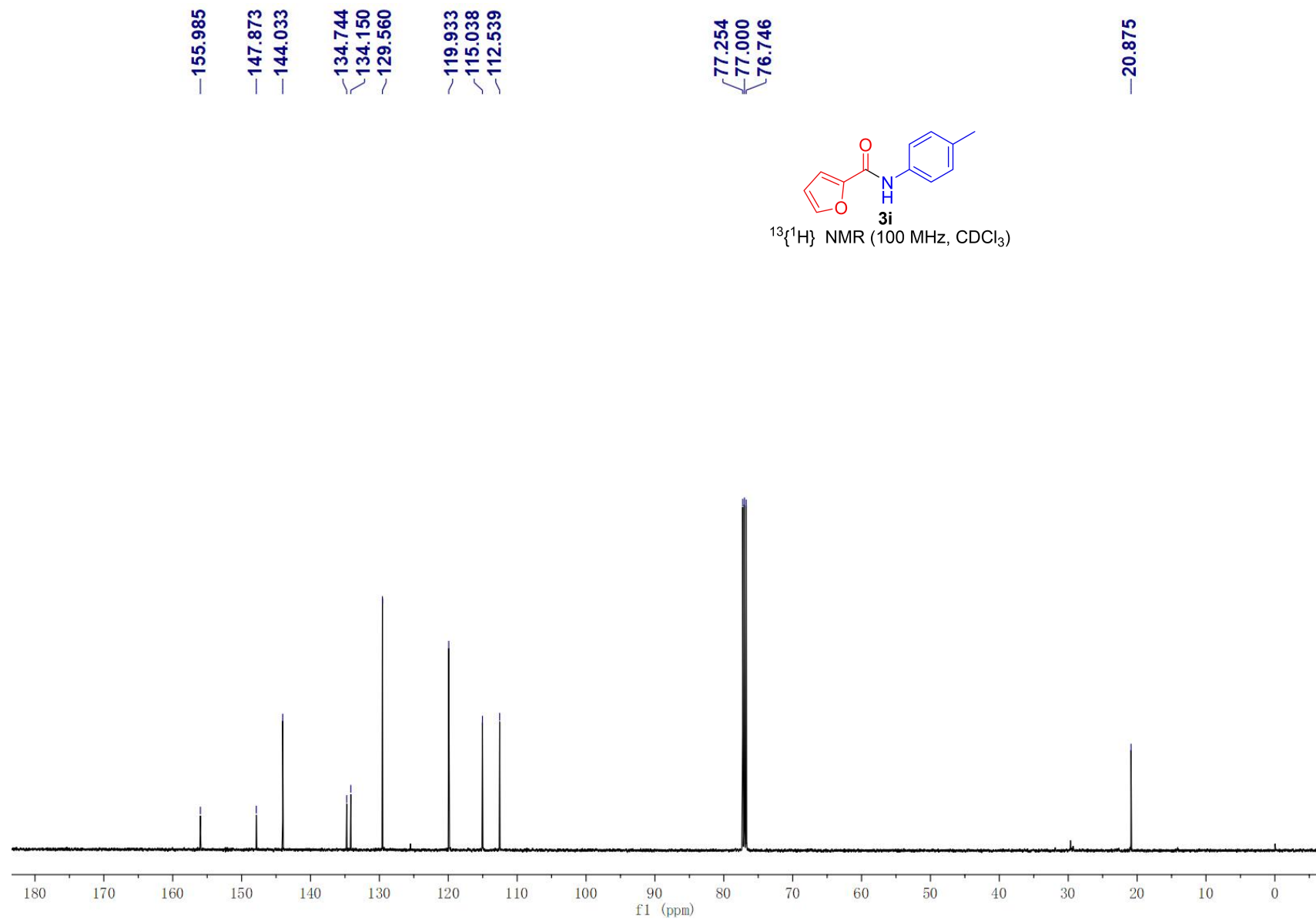


Figure 19:  $^{13}\text{C}$  NMR spectrum of product **3i**

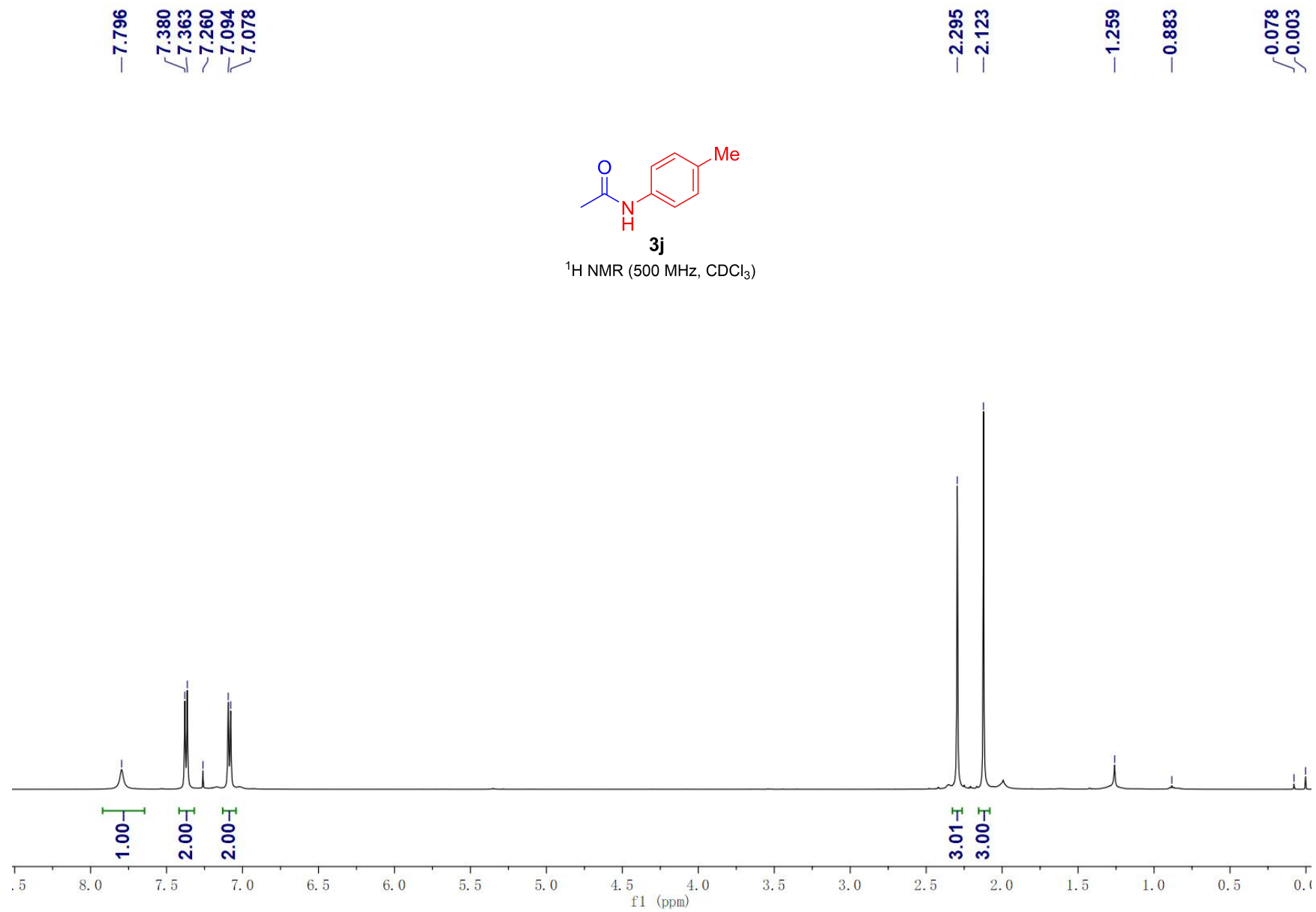


Figure 20: <sup>1</sup>H NMR spectrum of product **3j**

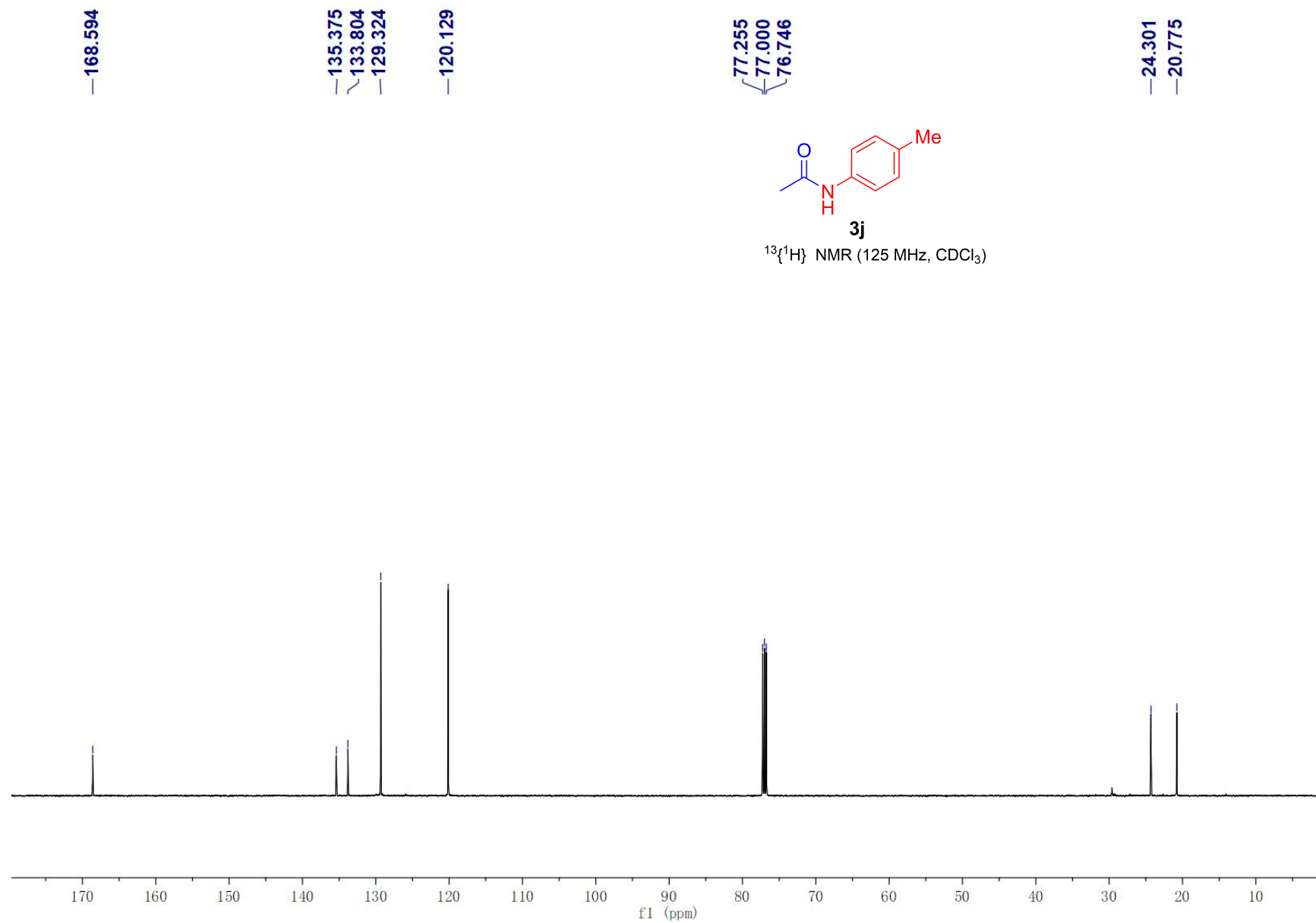


Figure 21:  $^{13}\text{C}$  NMR spectrum of product **3j**

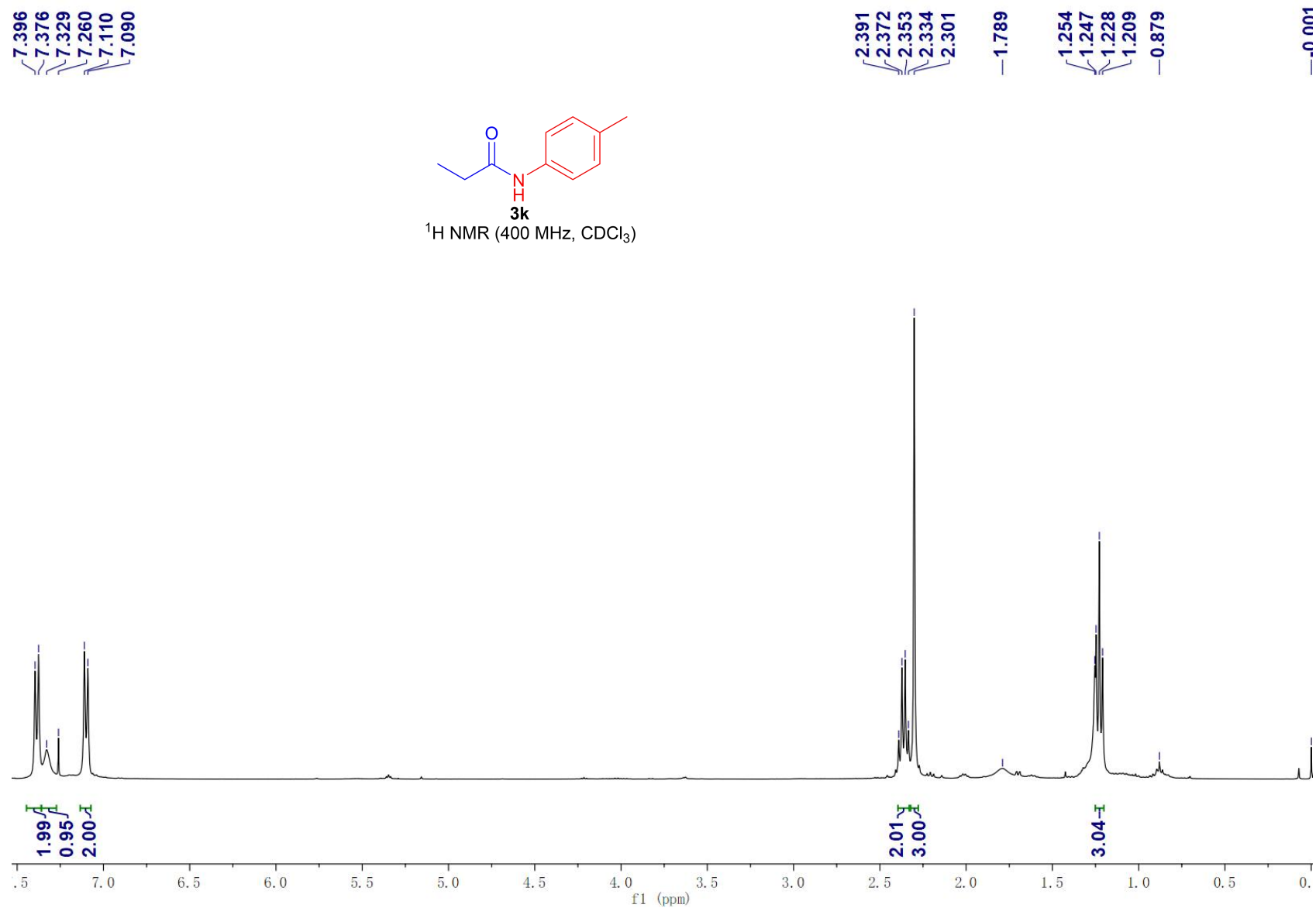


Figure 22: <sup>1</sup>H NMR spectrum of product **3k**

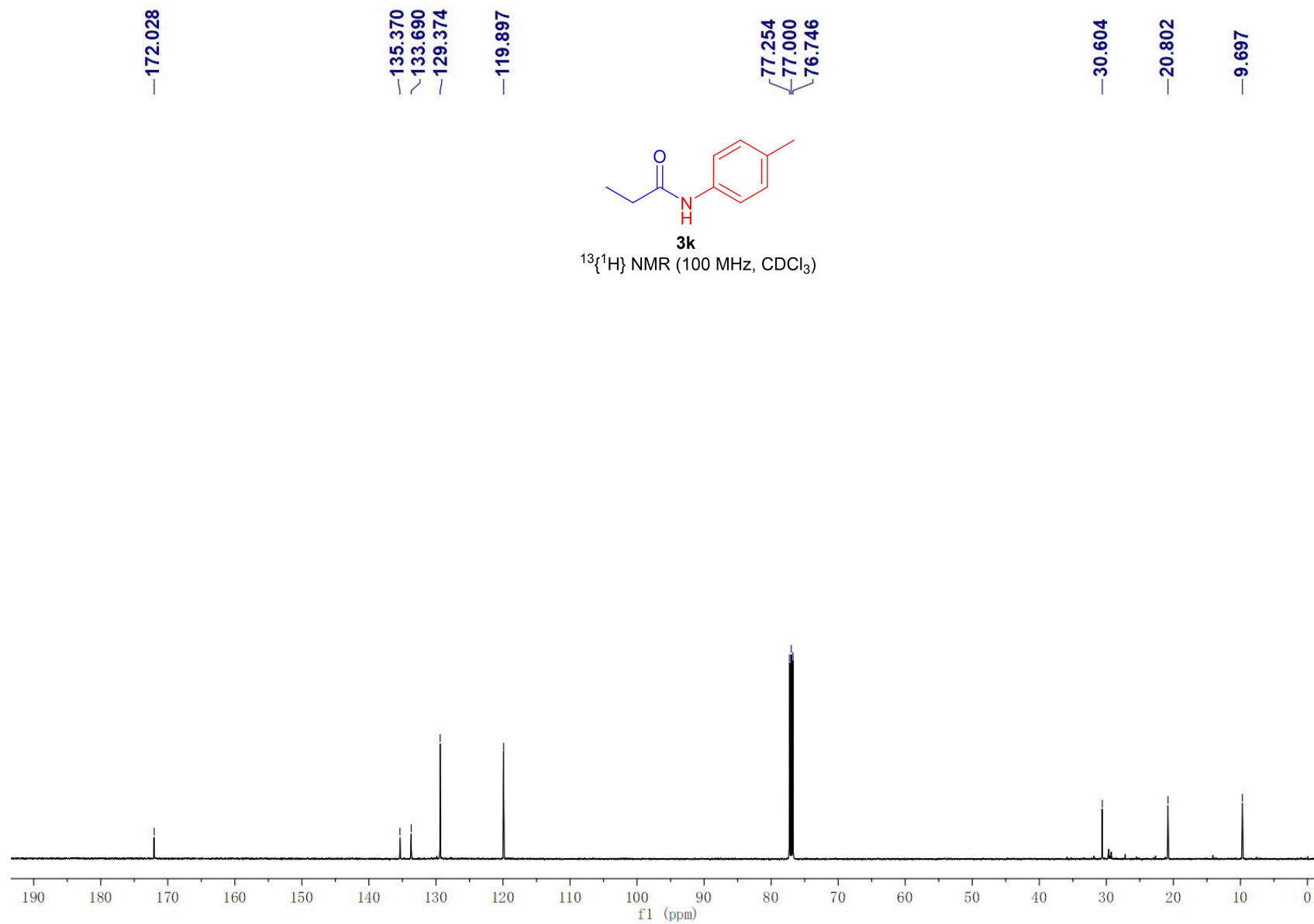


Figure 23:  $^{13}\text{C}$  NMR spectrum of product **3k**



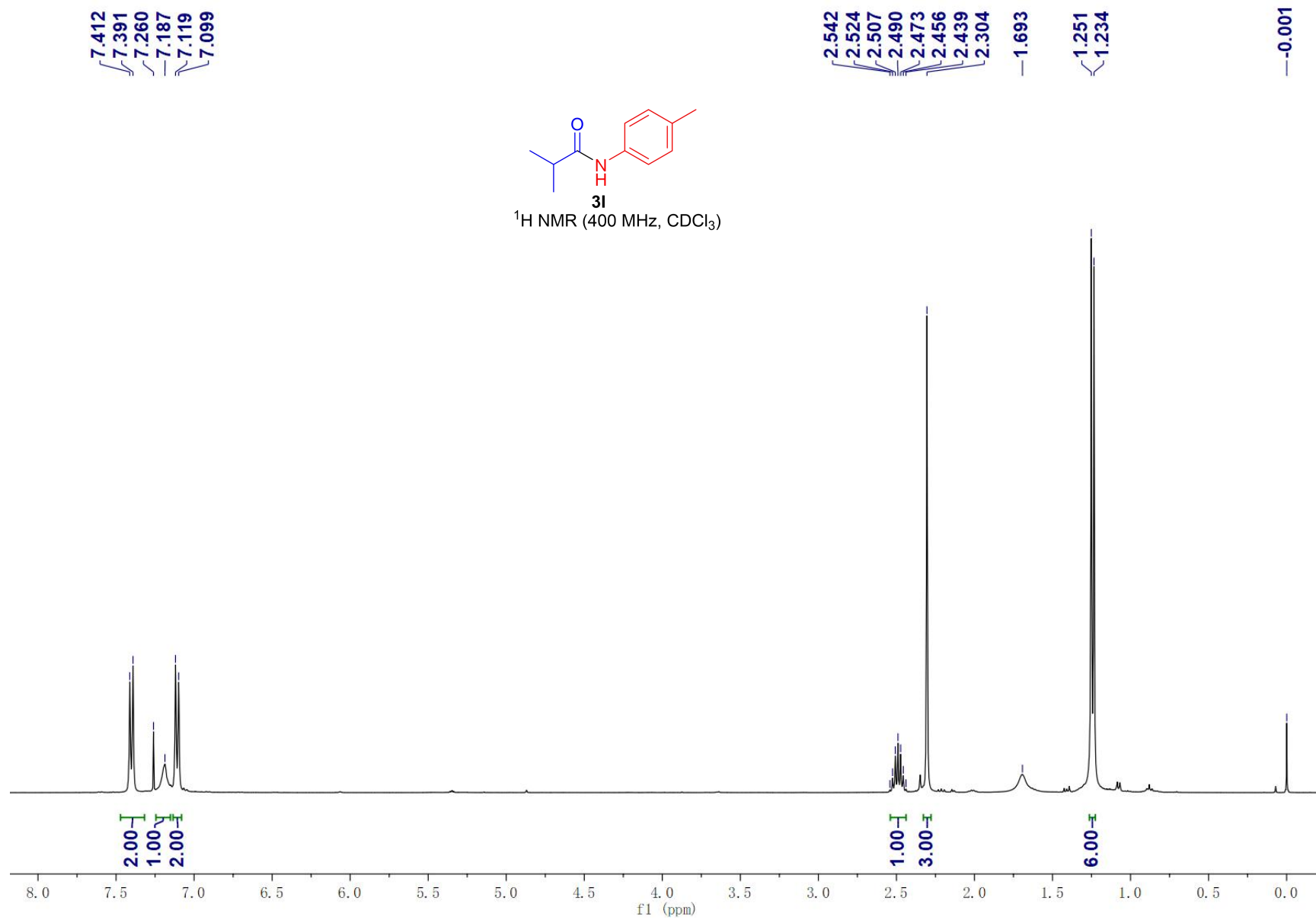


Figure 24: <sup>1</sup>H NMR spectrum of product **31**

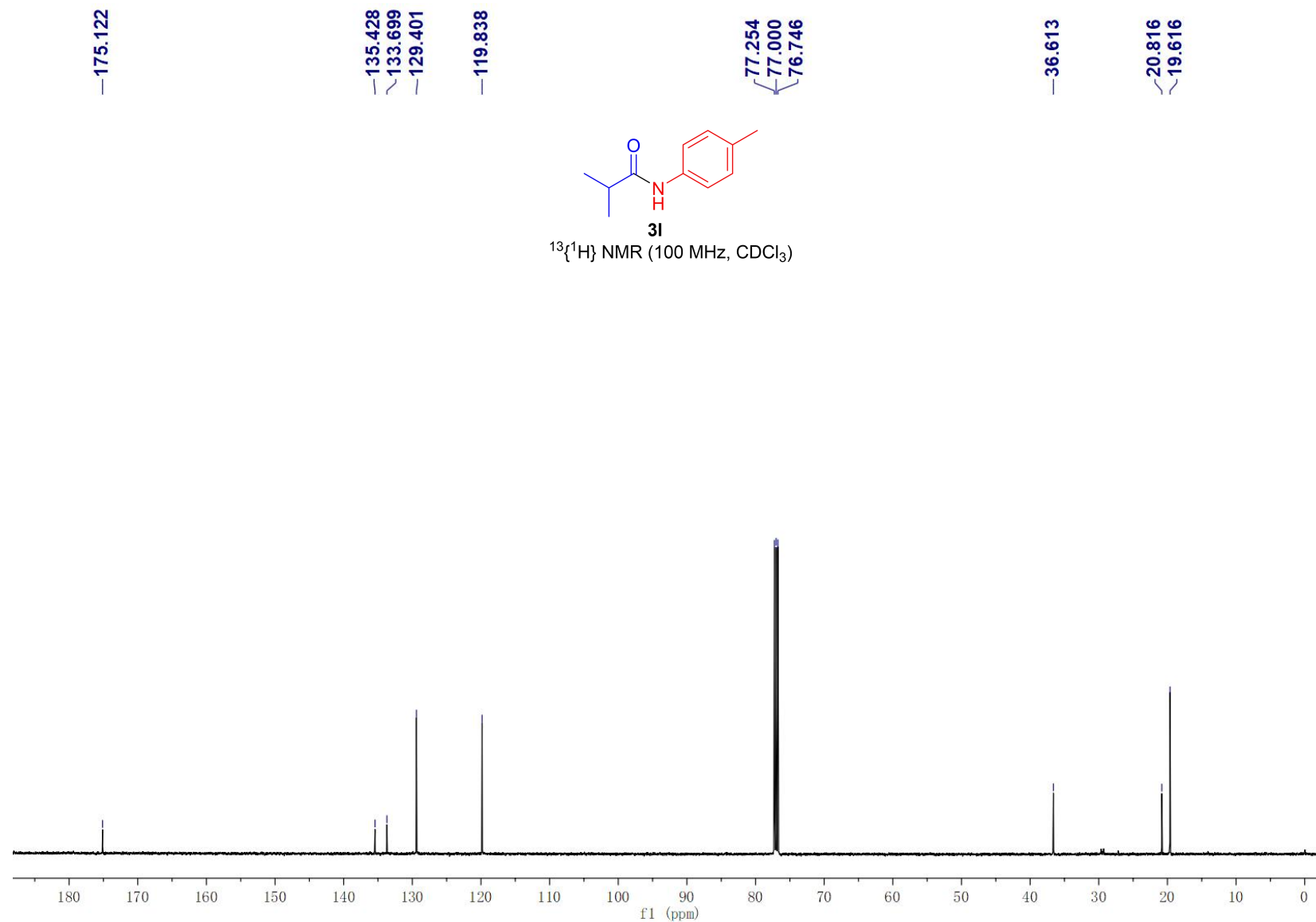


Figure 25:  $^{13}\text{C}$  NMR spectrum of product **31**

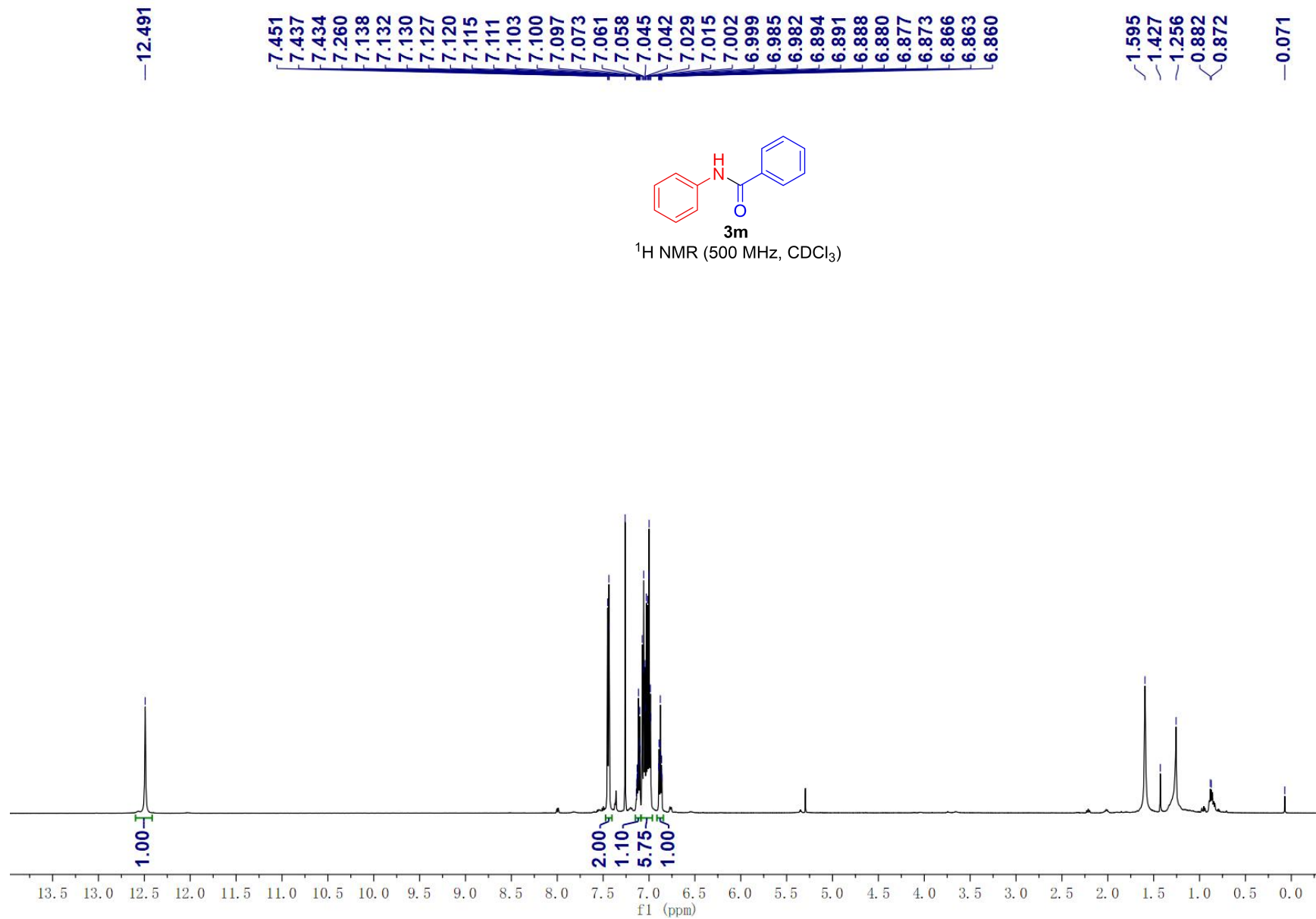


Figure 26: <sup>1</sup>H NMR spectrum of product **3m**

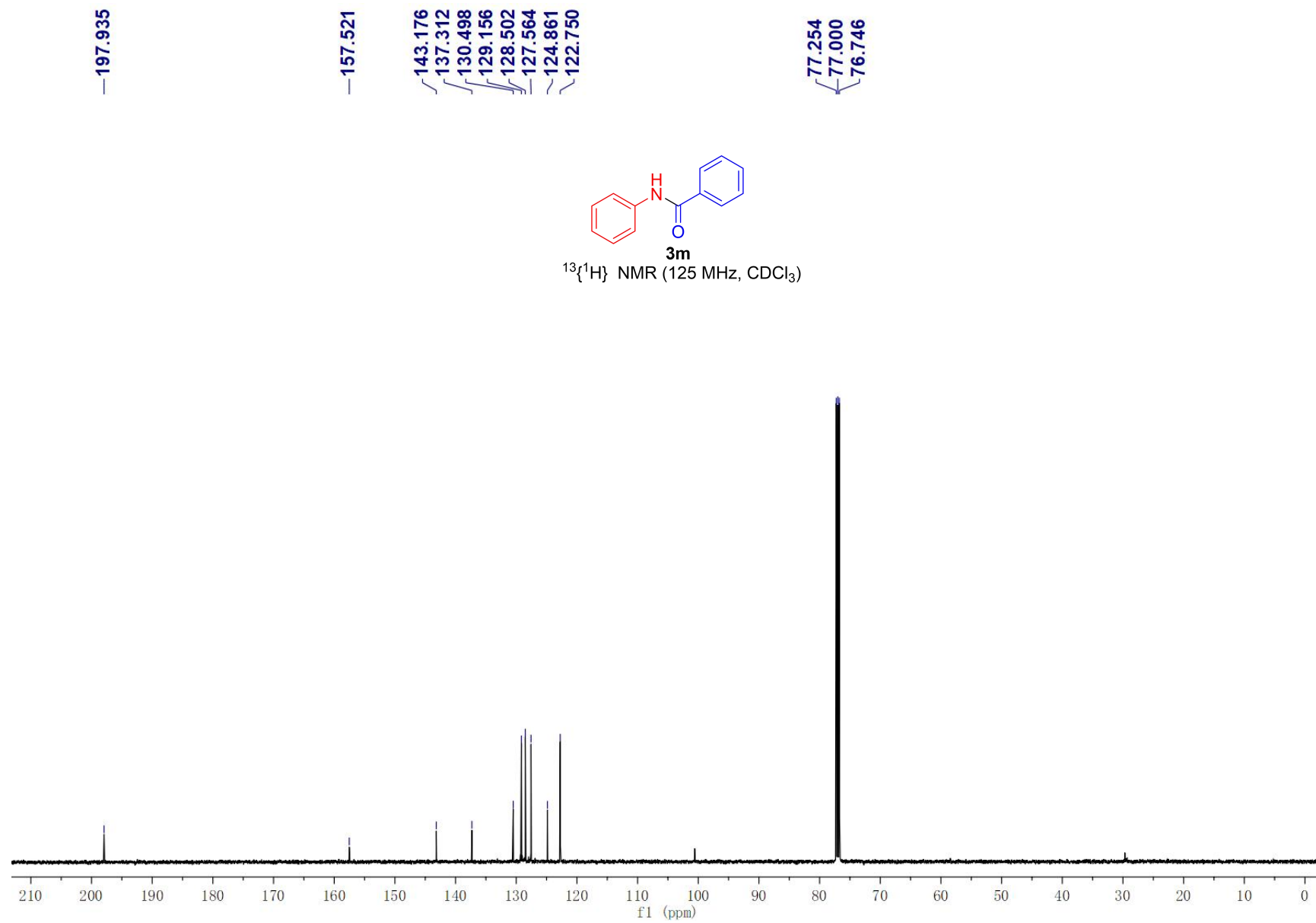


Figure 27:  $^{13}\text{C}$  NMR spectrum of product **3m**

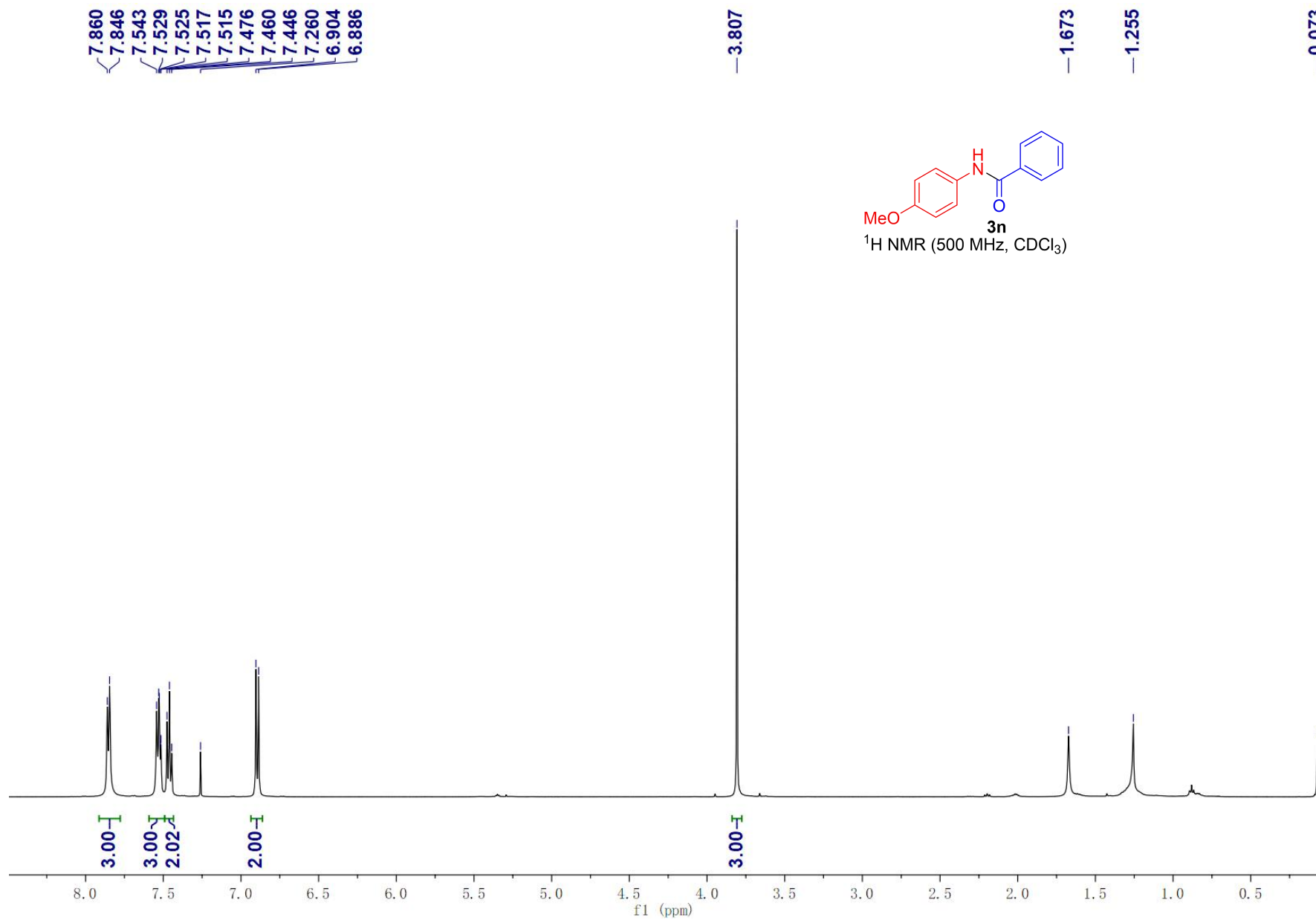


Figure 28: <sup>1</sup>H NMR spectrum of product **3n**

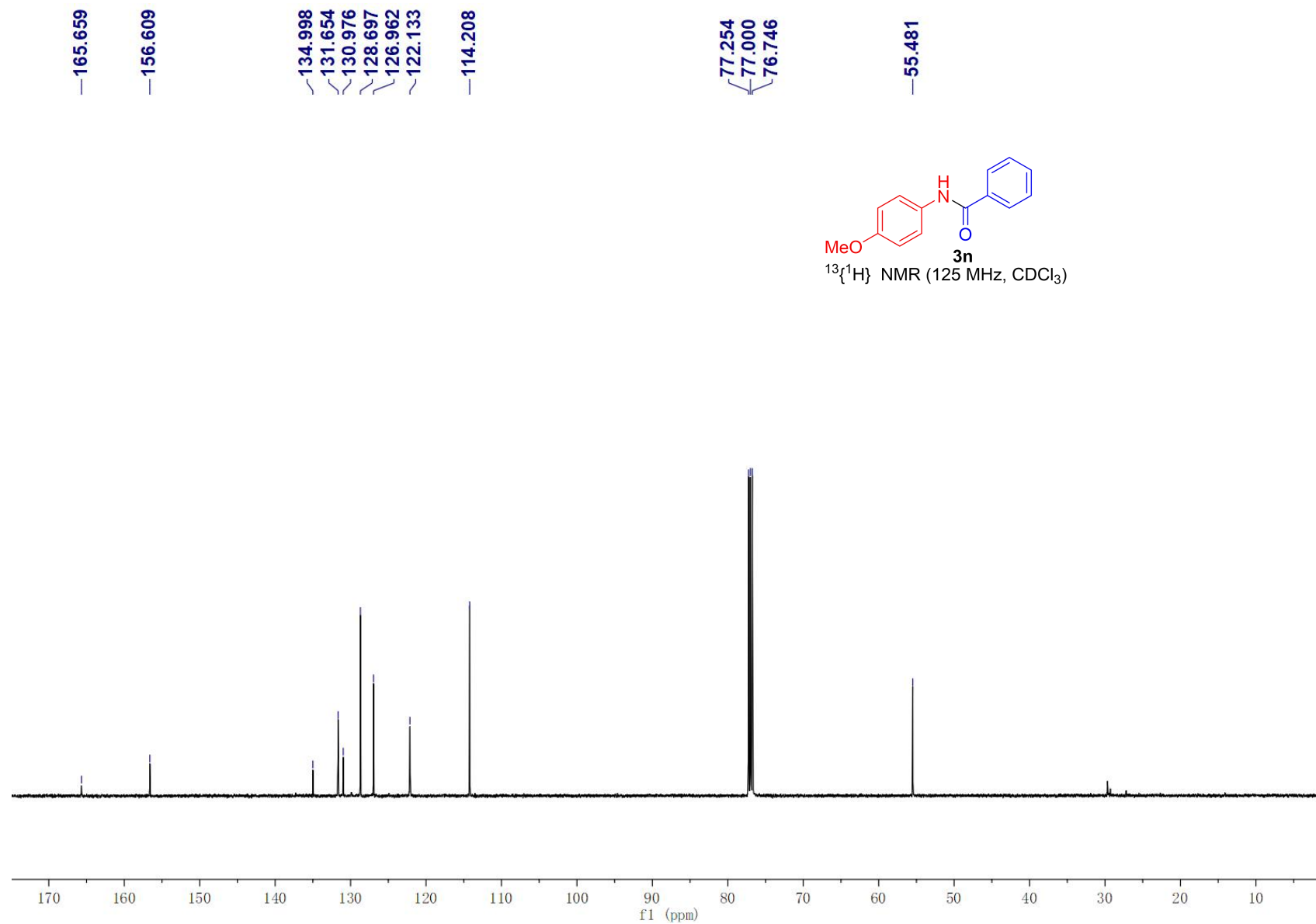


Figure 29: <sup>13</sup>C NMR spectrum of product **3n**

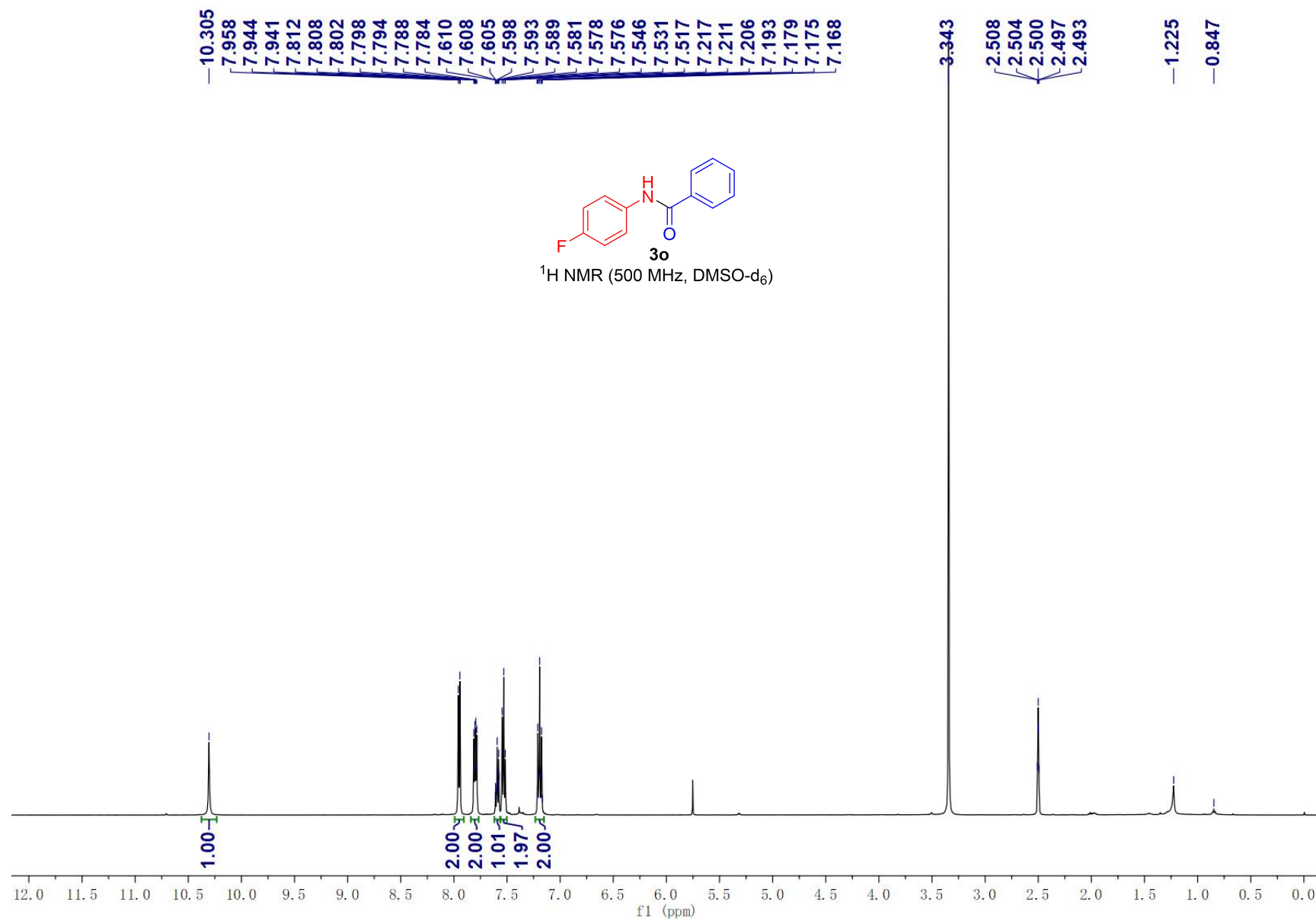


Figure 30: <sup>1</sup>H NMR spectrum of product **3o**

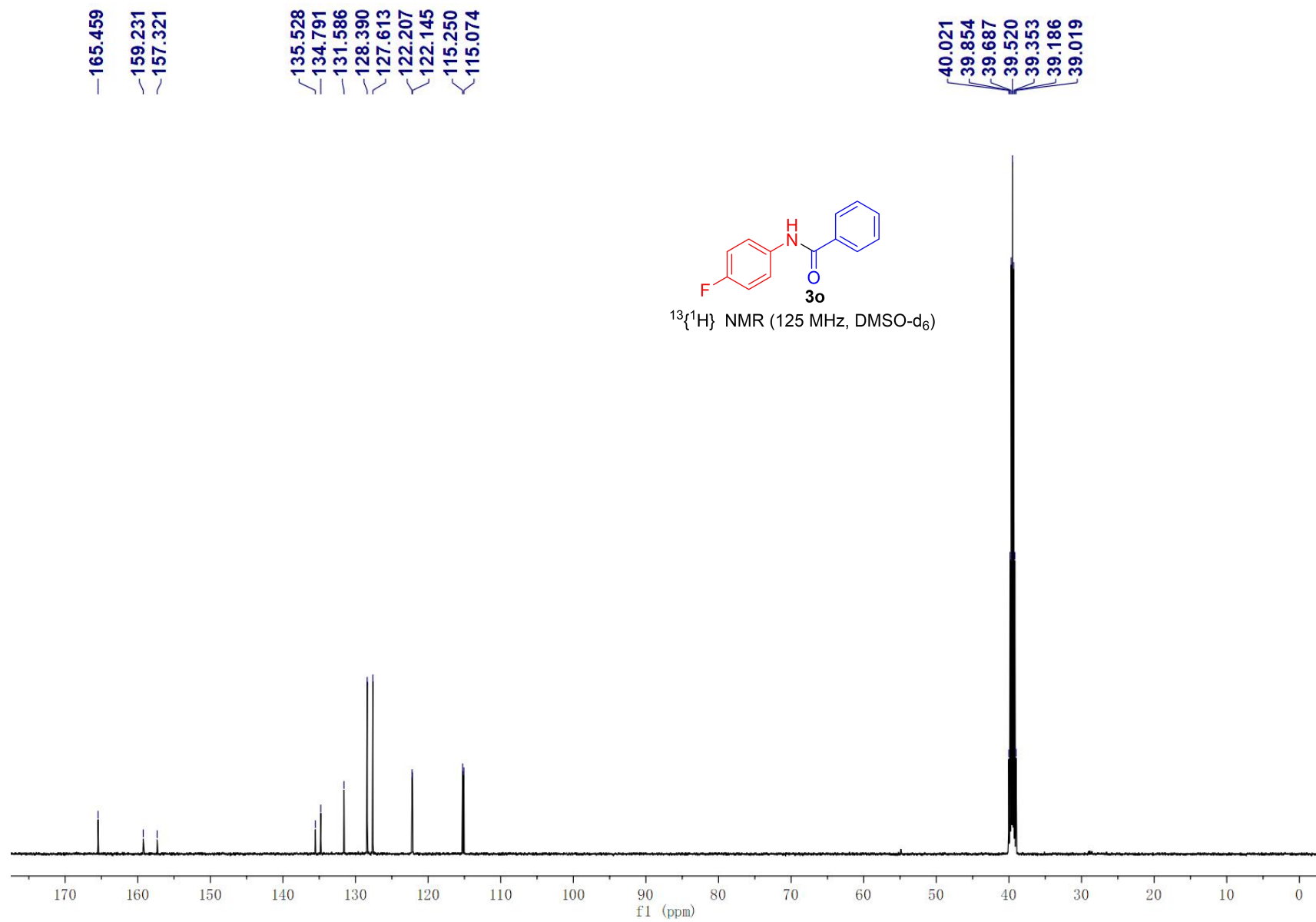


Figure 31:  $^{13}\text{C}$  NMR spectrum of product **3o**



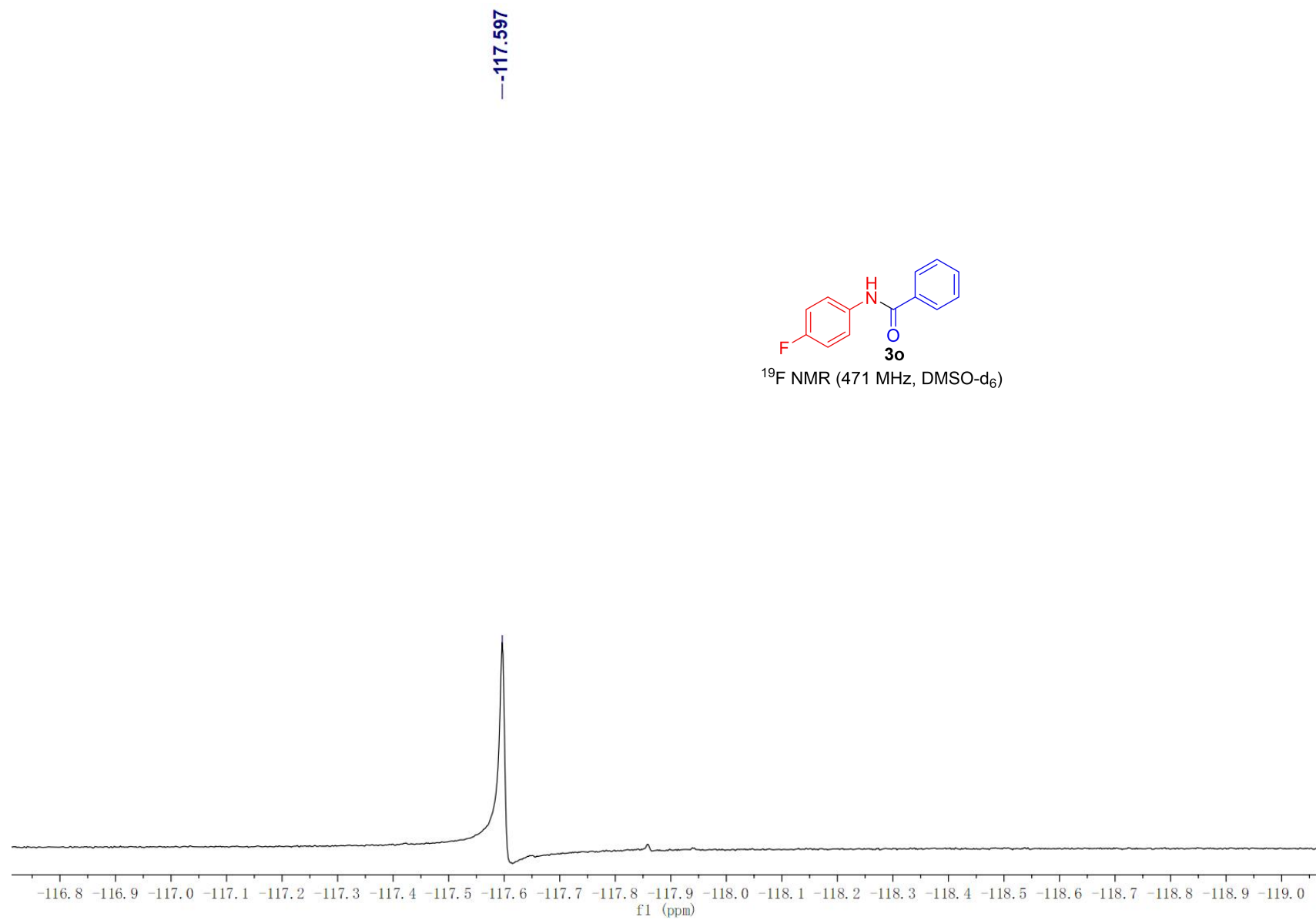


Figure 32: <sup>13</sup>C NMR spectrum of product **3o**

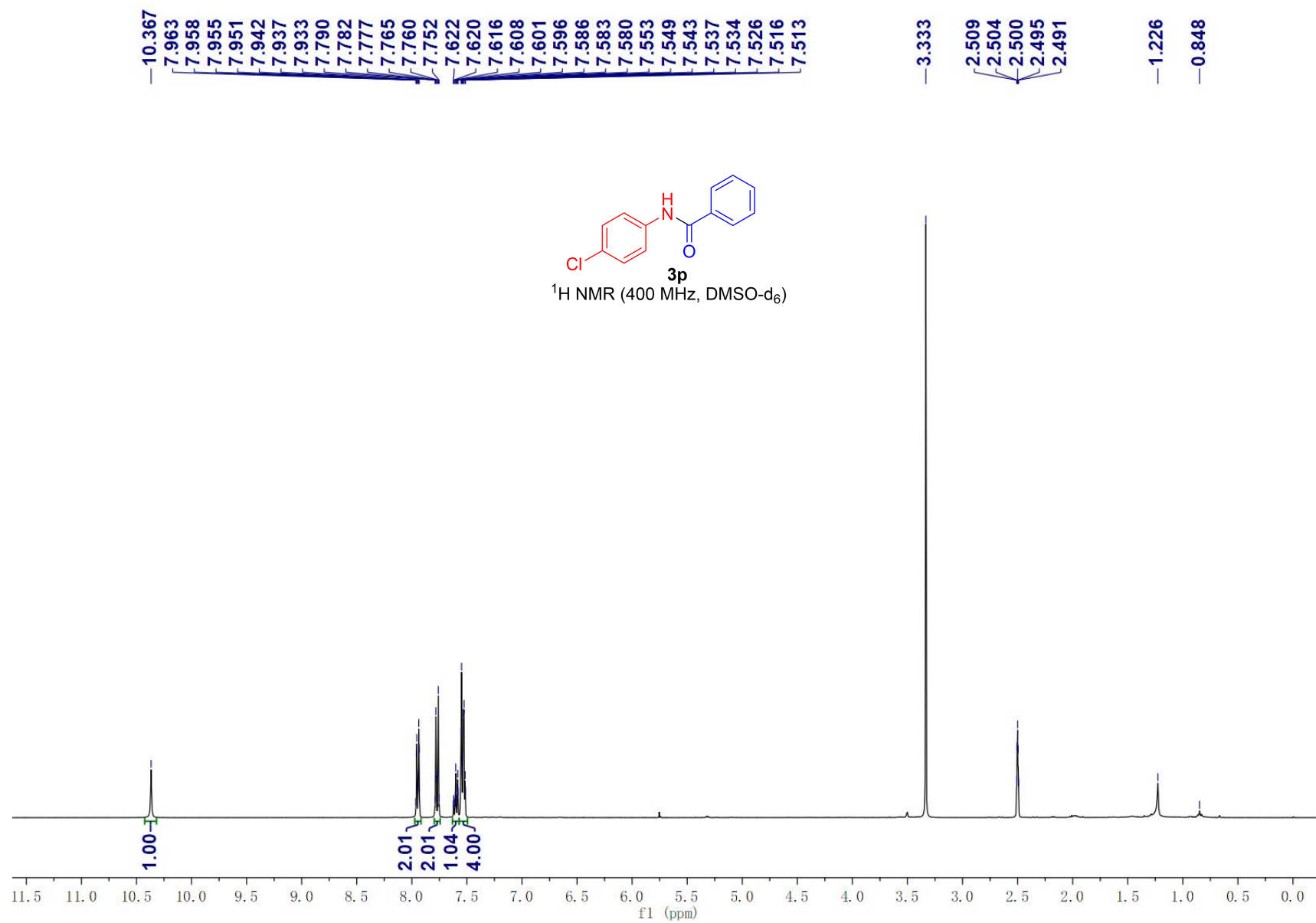


Figure 33: <sup>1</sup>H NMR spectrum of product **3p**

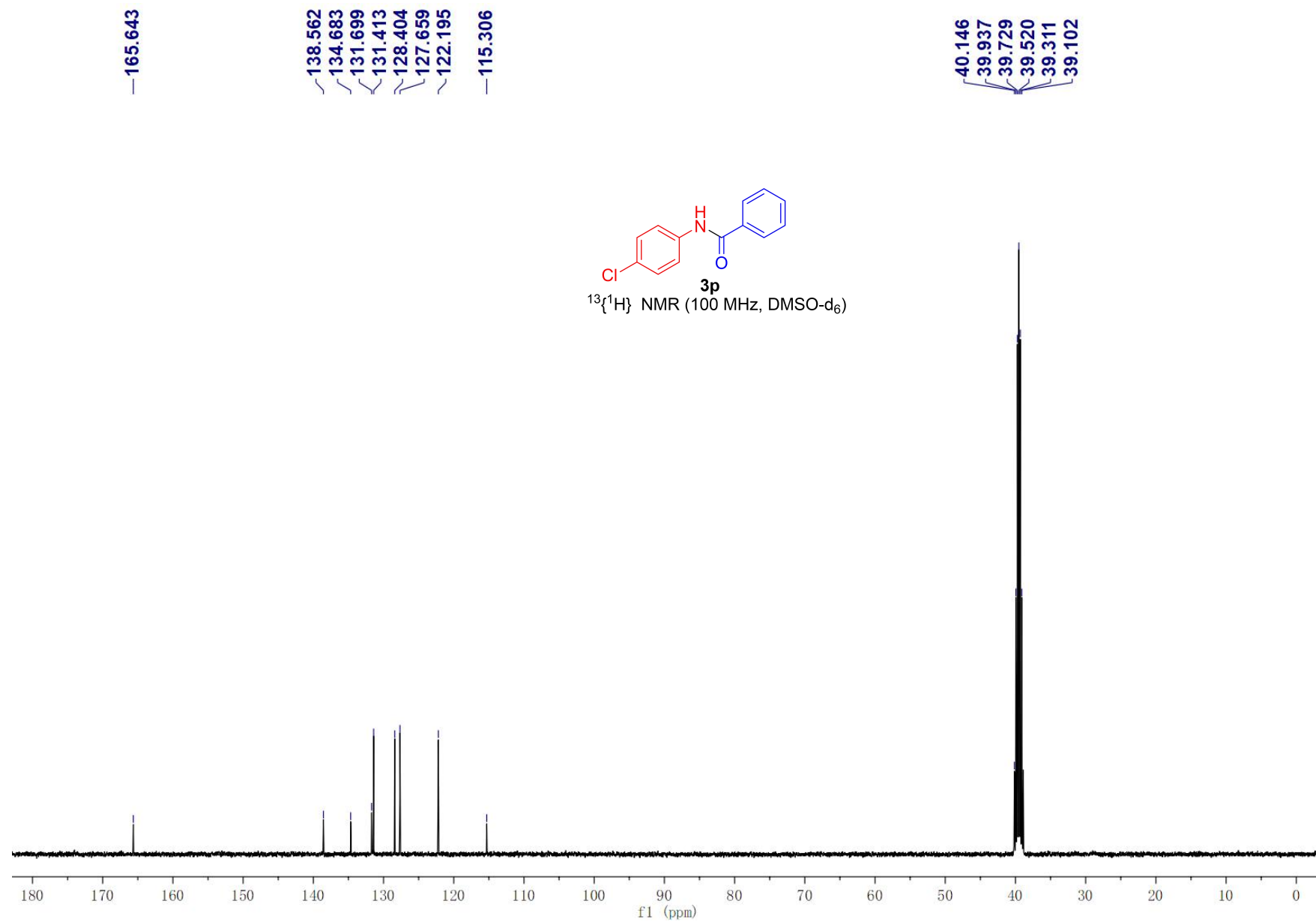


Figure 34:  $^{13}\text{C}$  NMR spectrum of product **3p**

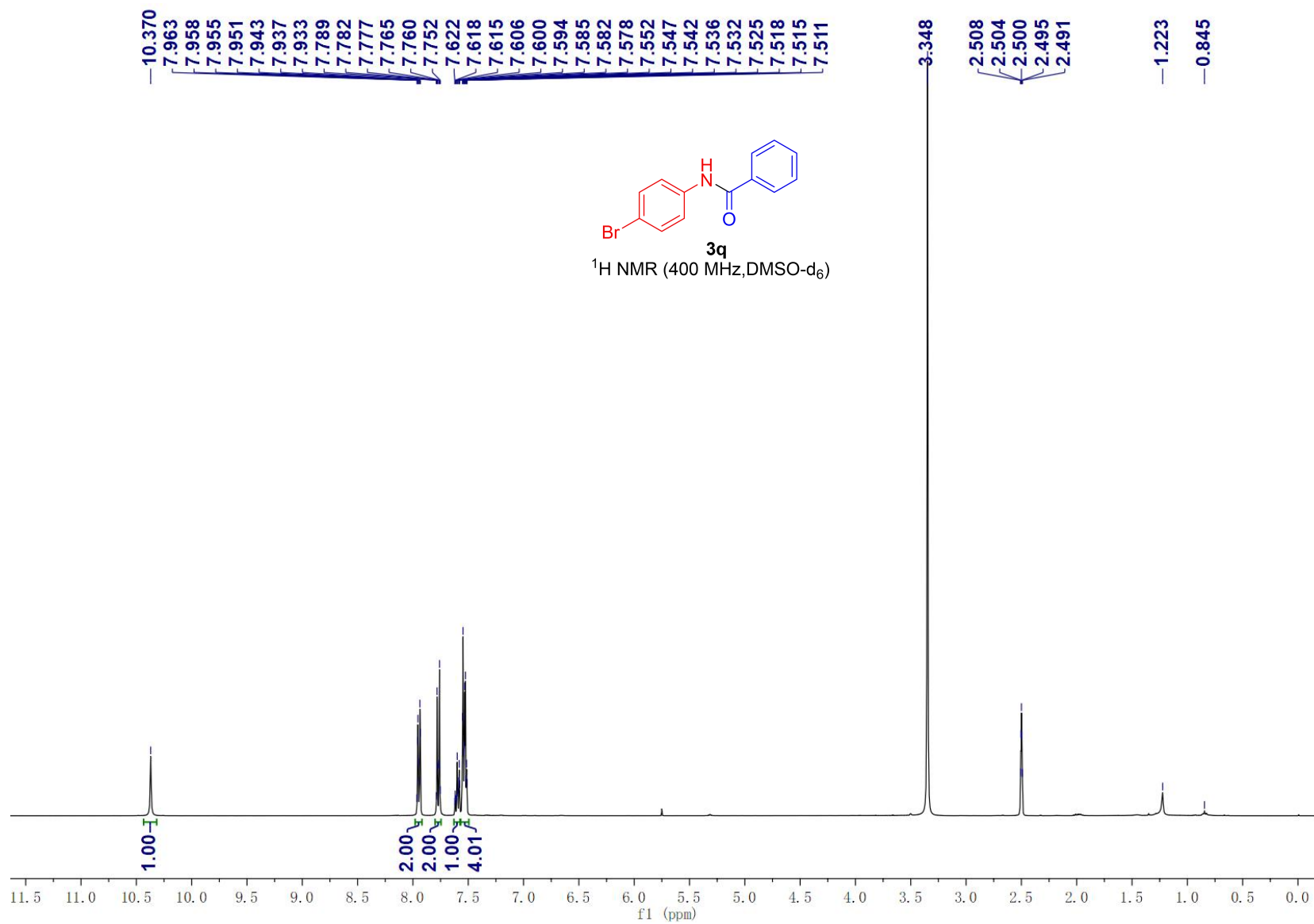


Figure 35: <sup>1</sup>H NMR spectrum of product **3q**

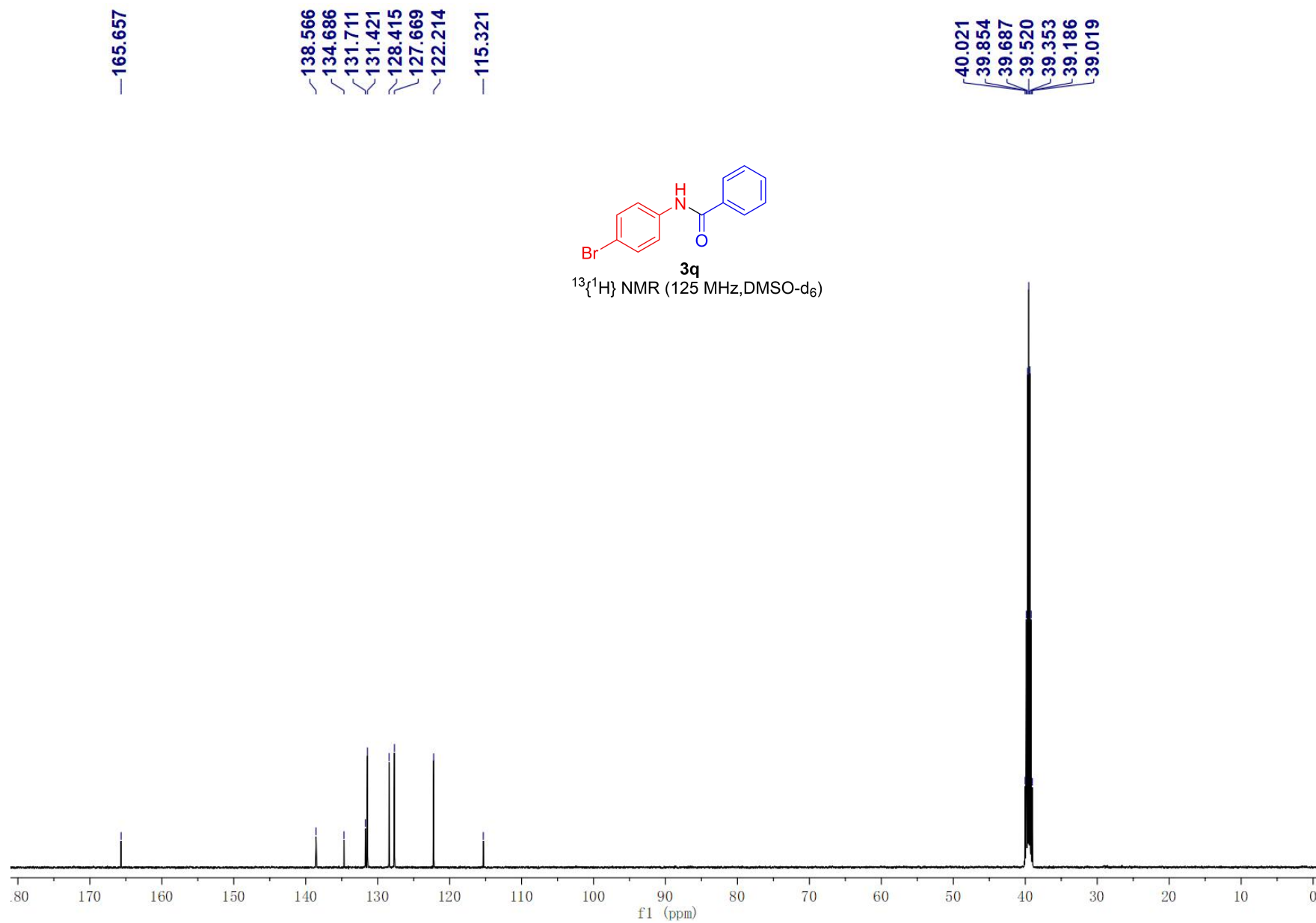


Figure 36:  $^{13}\text{C}$  NMR spectrum of product **3q**

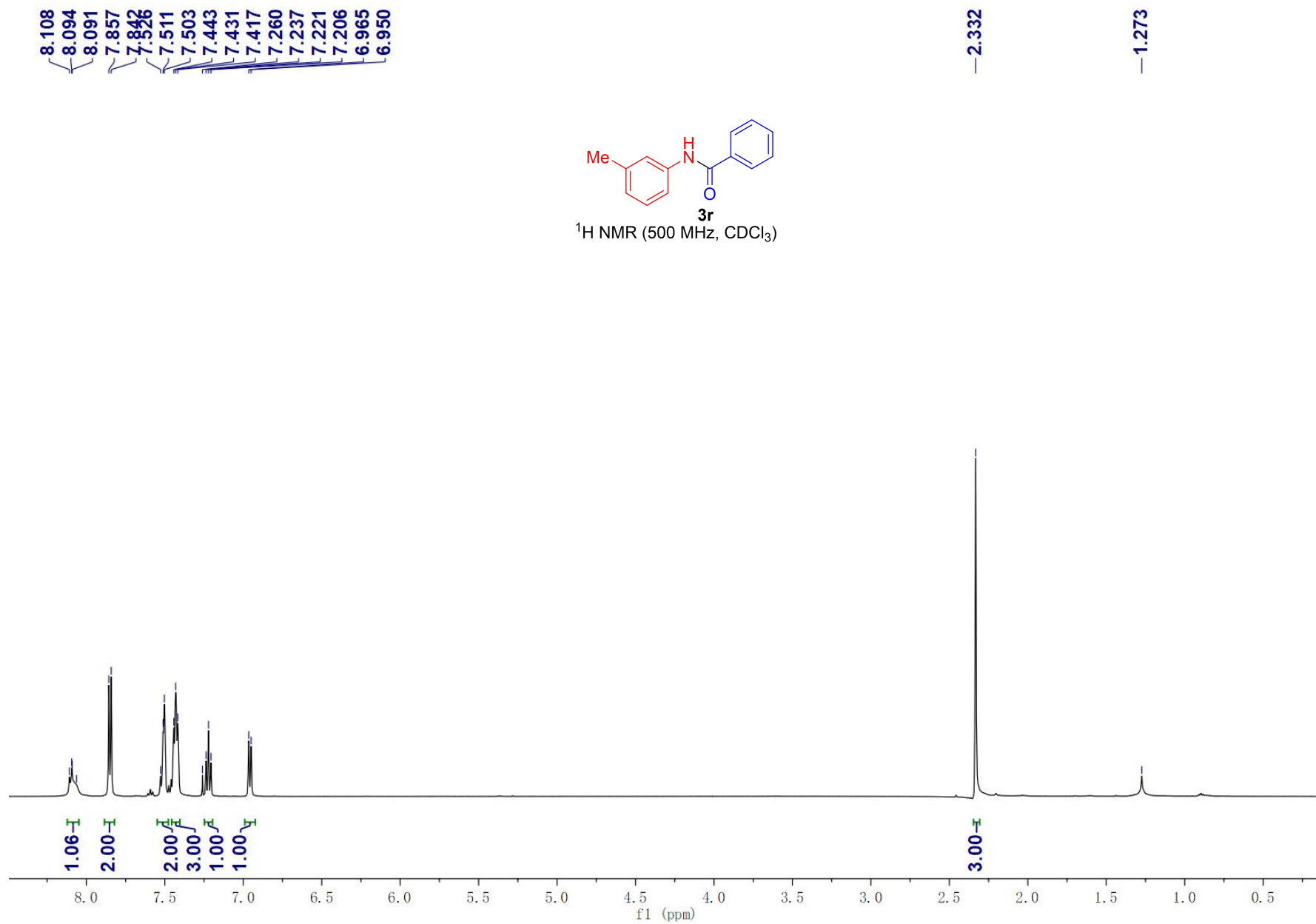


Figure 37: <sup>1</sup>H NMR spectrum of product **3r**

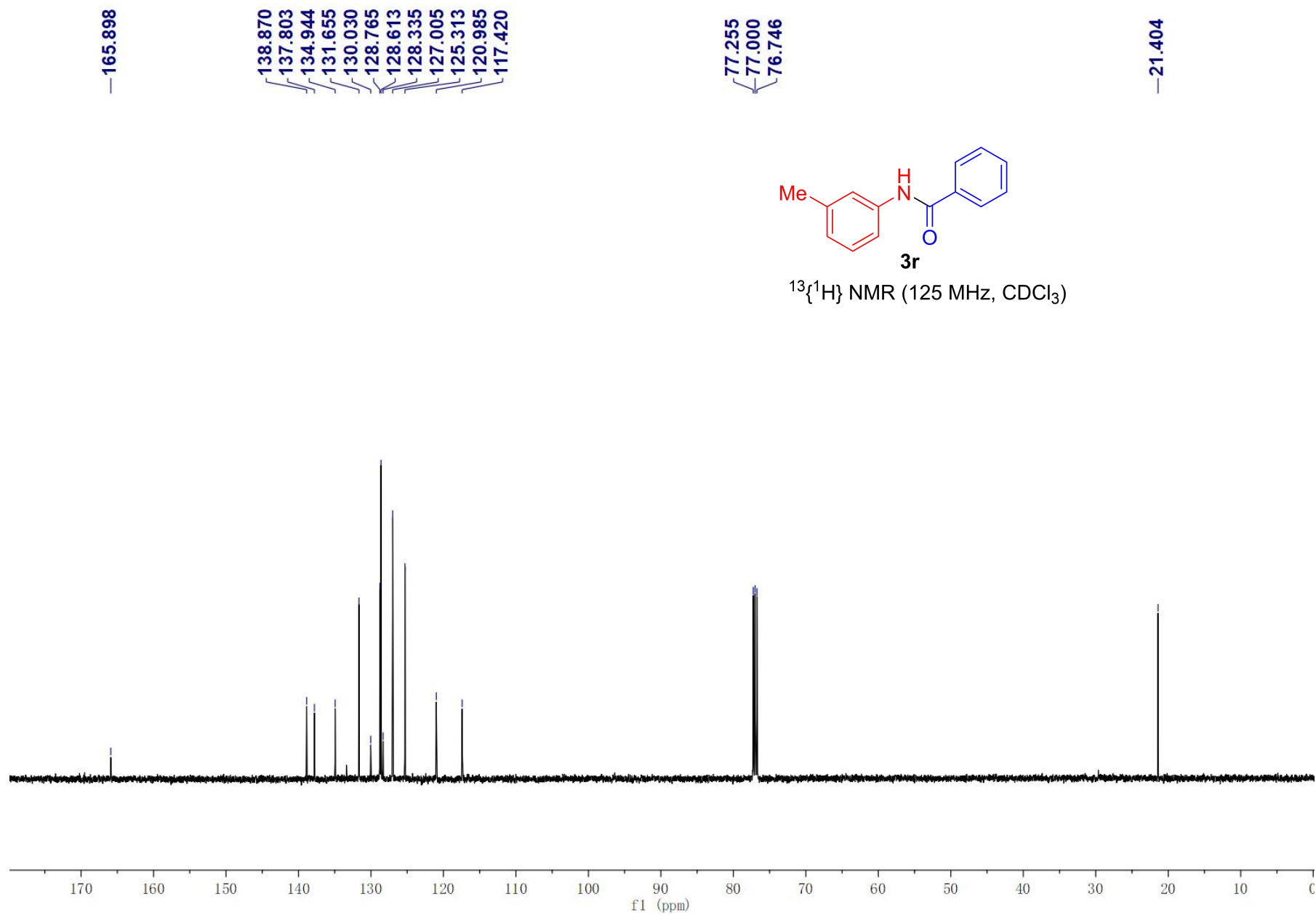


Figure 38: <sup>13</sup>C NMR spectrum of product **3r**

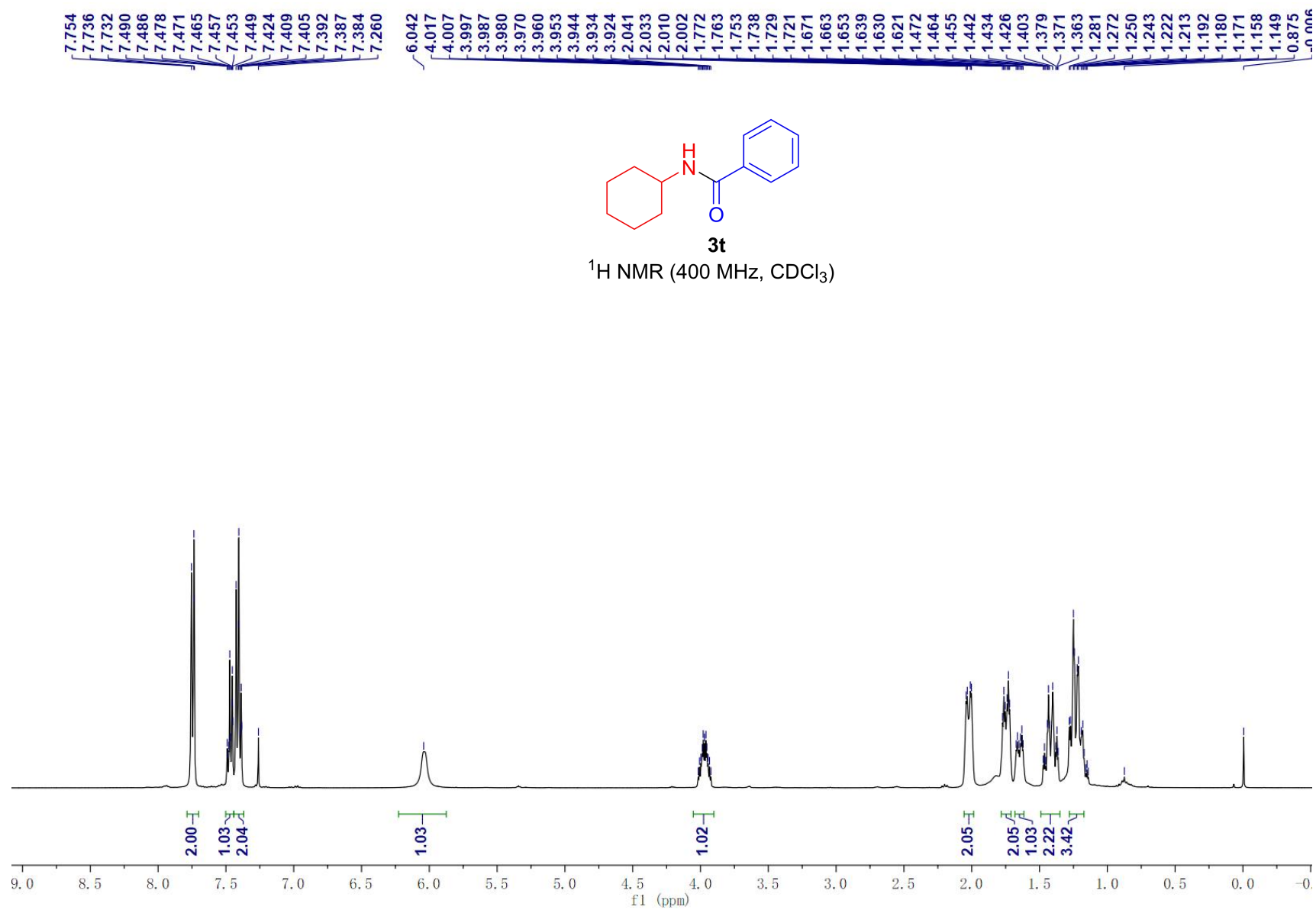


Figure 39: <sup>1</sup>H NMR spectrum of product **3t**



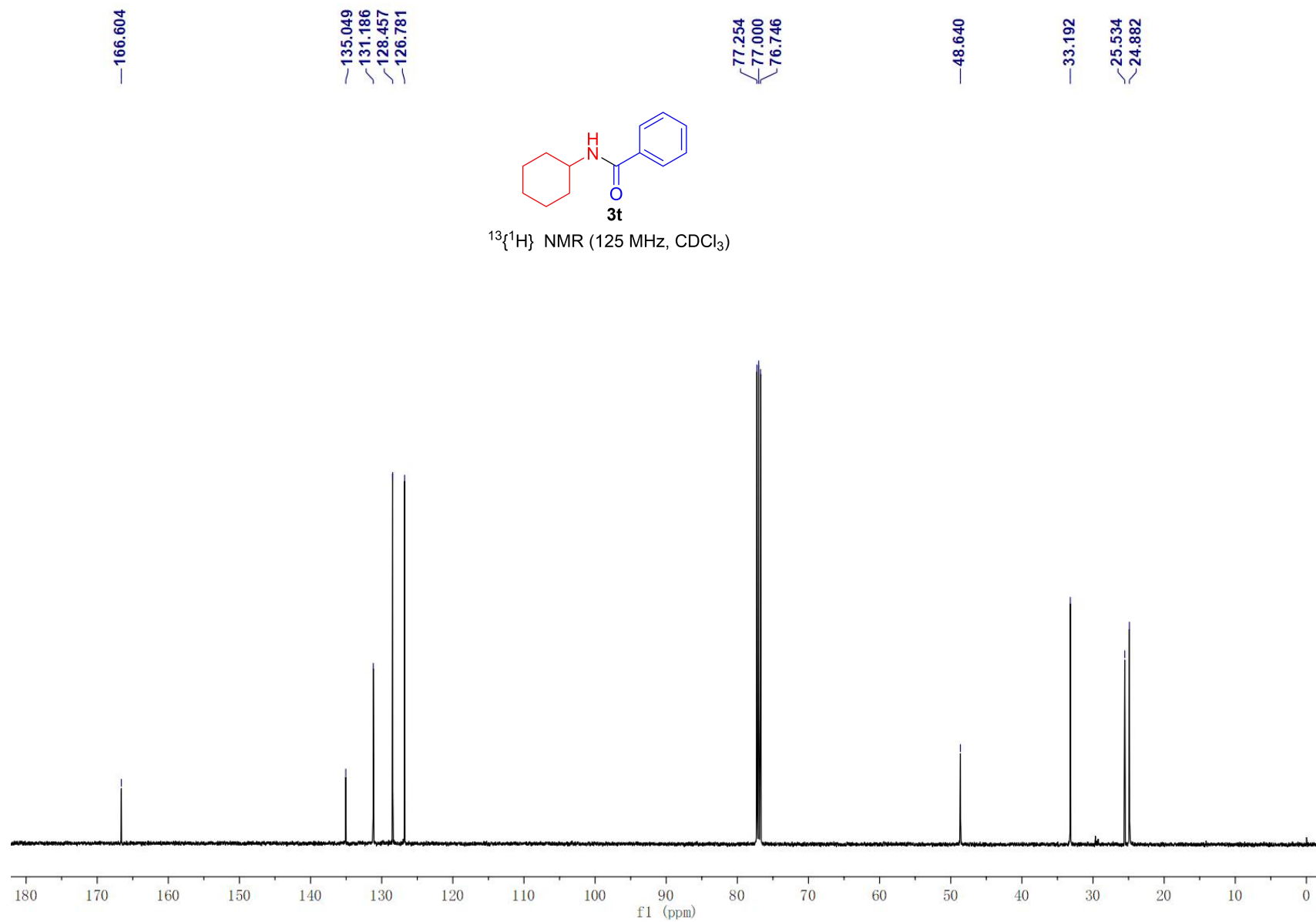


Figure 40:  $^{13}\text{C}$  NMR spectrum of product **3t**

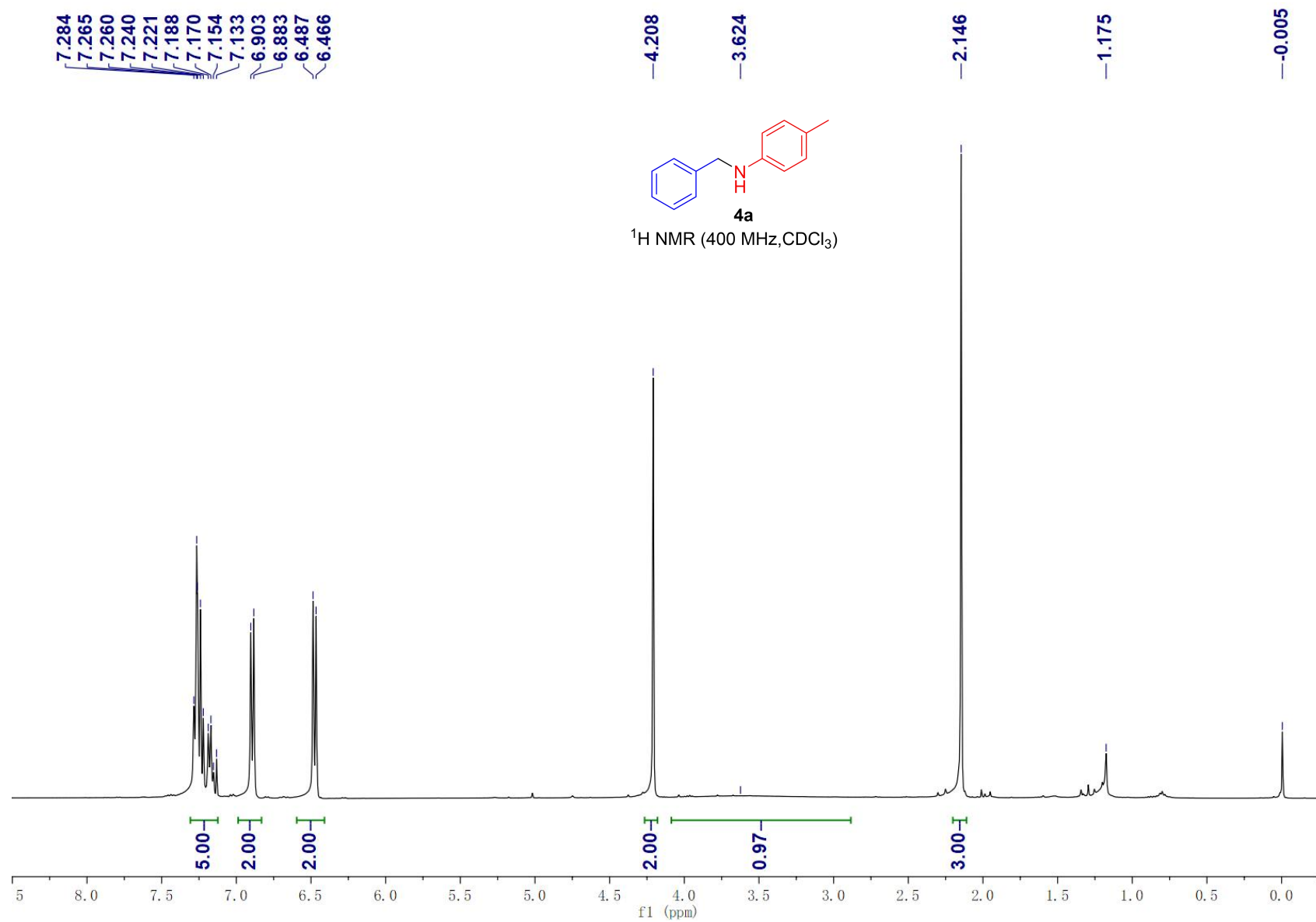


Figure 41:  $^1\text{H}$  NMR spectrum of product **4a**

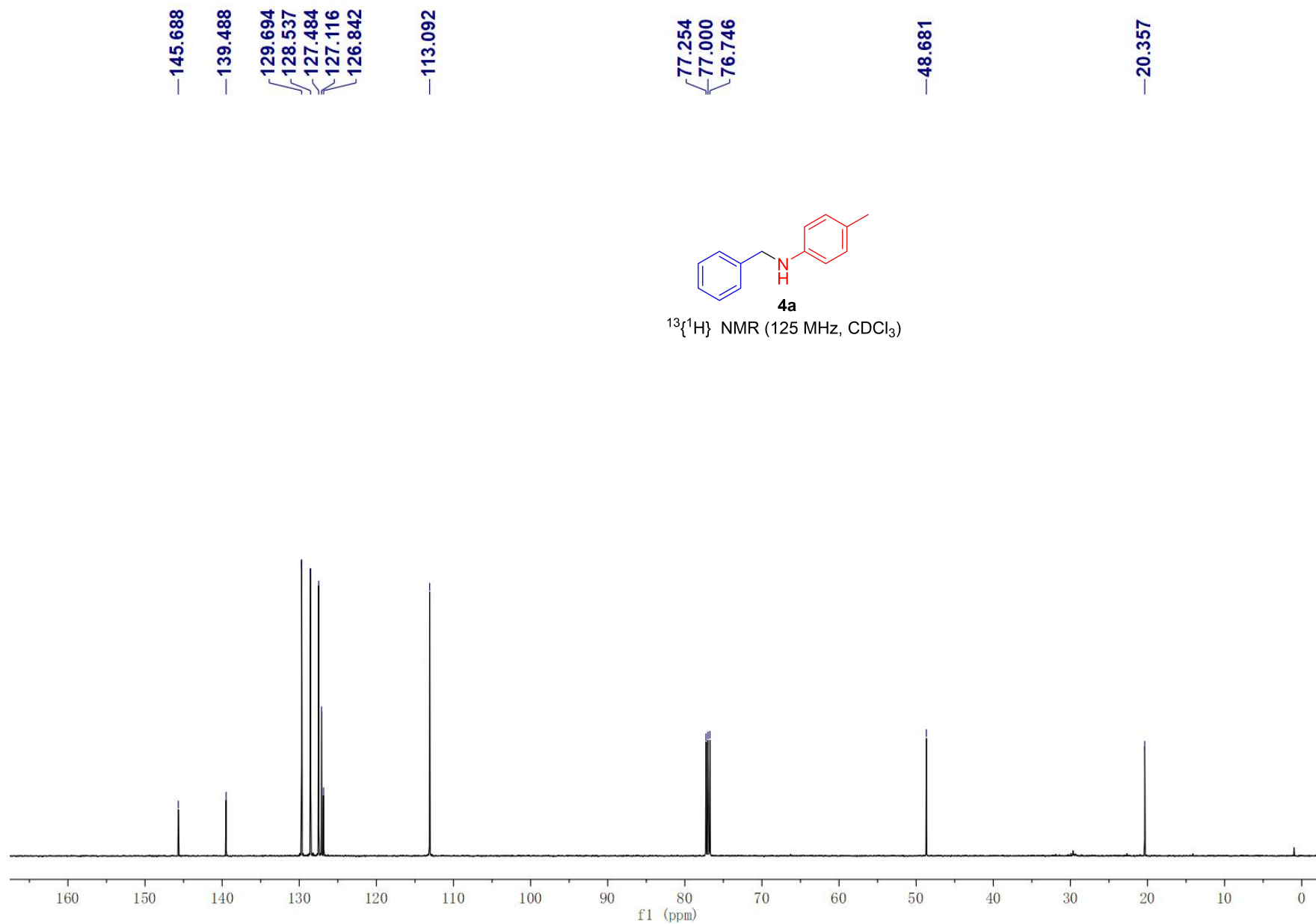


Figure 42: <sup>13</sup>C NMR spectrum of product **4a**

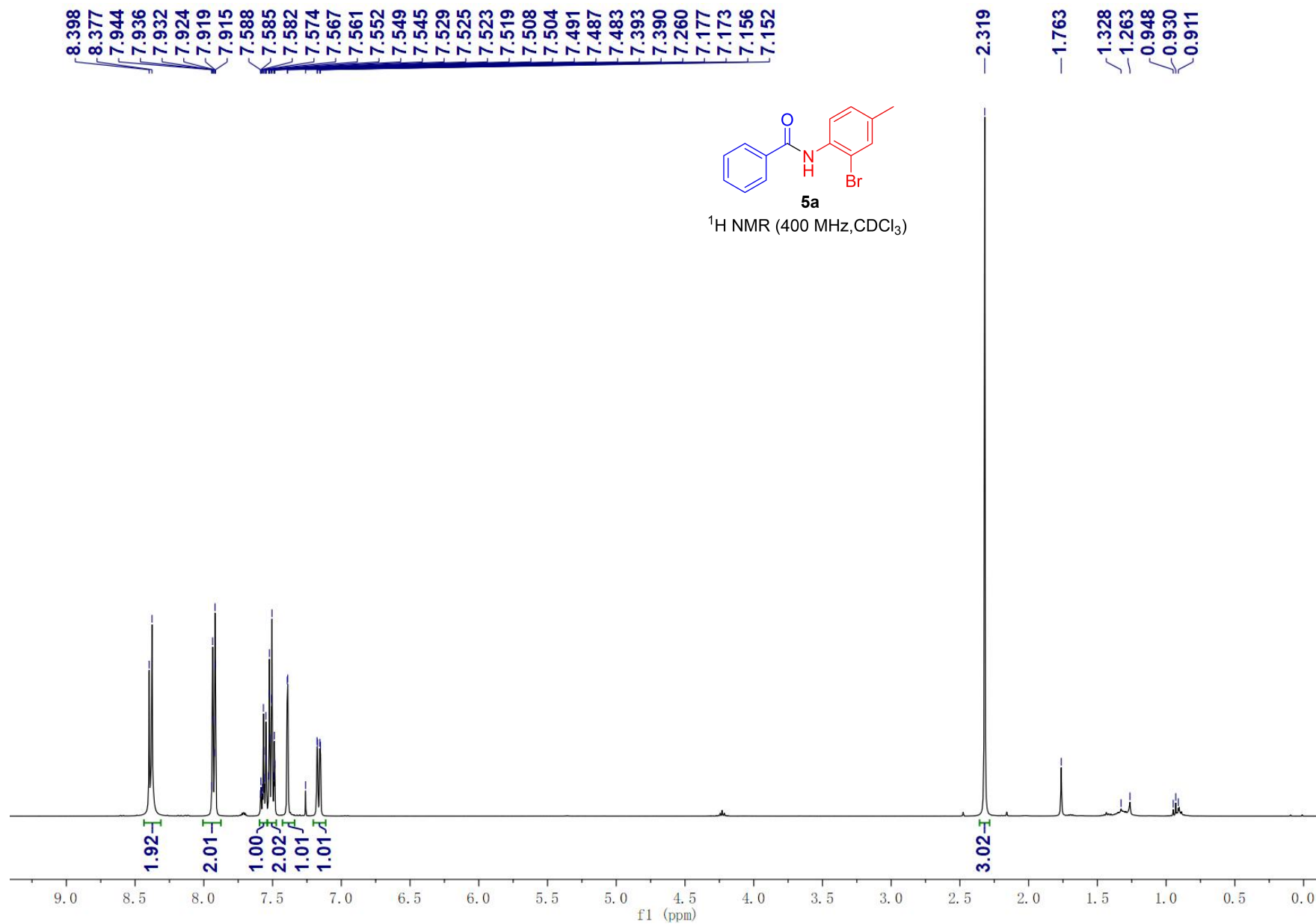


Figure 43: <sup>1</sup>H NMR spectrum of product **5a**

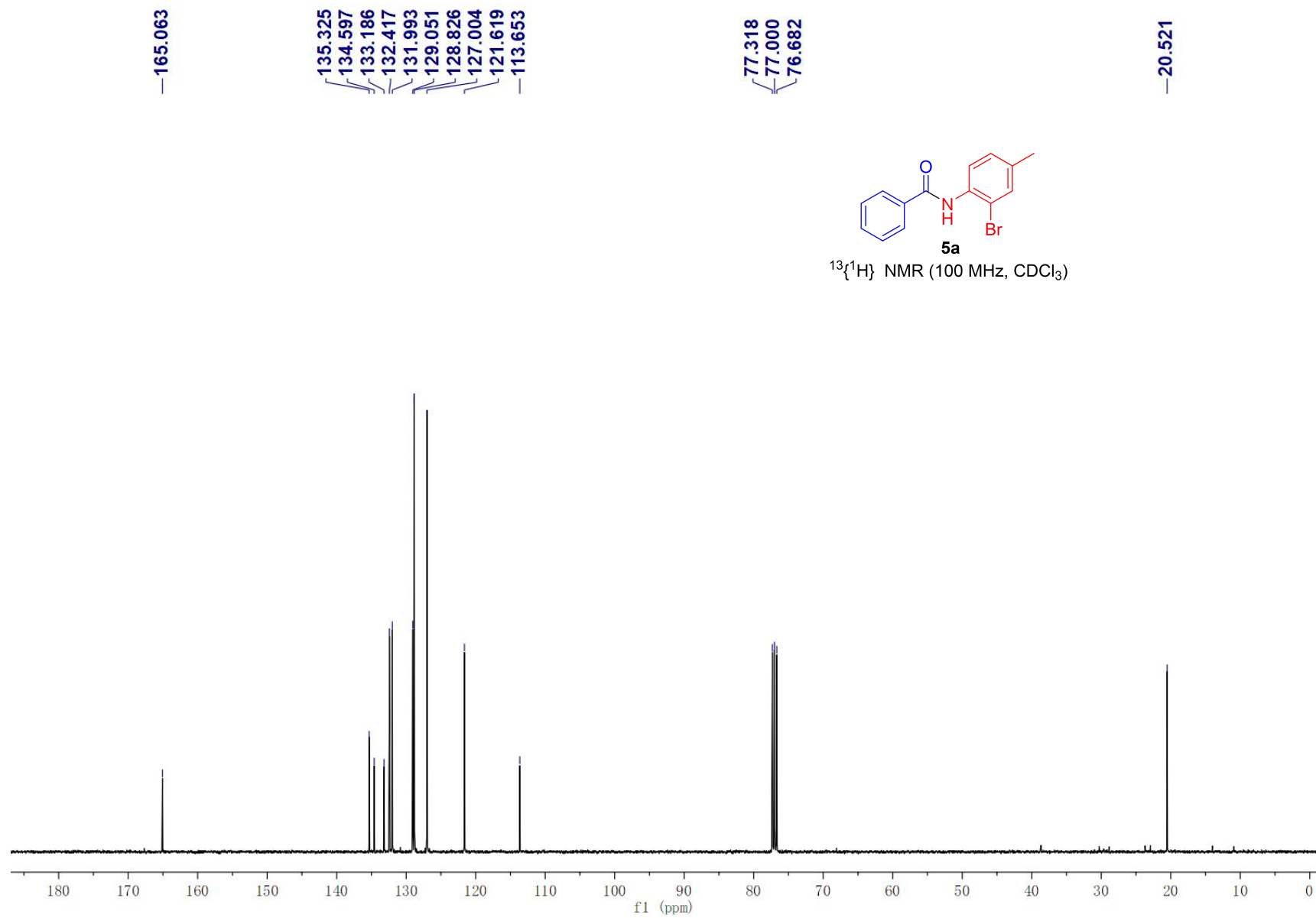


Figure 44: <sup>13</sup>C NMR spectrum of product **5a**