

**Supporting Information for:**

**Stereoselective Synthesis of 2,6-*trans*-4-Oxopiperidines using an  
Acid-Mediated 6-*endo-trig* Cyclisation**

*Jonathan D. Bell,<sup>a</sup> Alexander H. Harkiss,<sup>a</sup> Christopher R. Wellaway<sup>b</sup> and Andrew Sutherland<sup>\*a</sup>*

*<sup>a</sup>WestCHEM, School of Chemistry, The Joseph Black Building, University of Glasgow, Glasgow G12 8QQ, UK. <sup>b</sup>Allergic Inflammation Discovery Performance Unit and Respiratory Therapy Area Unit, GlaxoSmithKline Medicines Research Centre, Gunnels Wood Road, Stevenage SG1 2NY, United Kingdom. Email: Andrew.Sutherland@glasgow.ac.uk; Tel: 0141 330 5936.*

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## 1. Characterisation Data for Minor *cis*-Isomers 12a–i

***tert*-butyl (2*R*,6*S*)-2-methyl-4-oxo-6-propylpiperidine-1-carboxylate (12a):**  $\nu_{\max}/\text{cm}^{-1}$  (neat) 2969 (CH), 1719 (C=O), 1690 (C=O), 1403, 1354, 1174, 1116;  $[\alpha]_{\text{D}}^{22} -14.2$  ( $c$  0.5,  $\text{CHCl}_3$ );  $\delta_{\text{H}}$  (500 MHz,  $\text{CDCl}_3$ ) 0.91 (3H, t,  $J$  7.3 Hz, 3''-H<sub>3</sub>), 1.16–1.38 (5H, m, 1'-H<sub>3</sub> and 2''-H<sub>2</sub>), 1.40–1.52 (10H, m, 1''-HH and 3 × CH<sub>3</sub>), 1.54–1.62 (1H, m, 1''-HH), 2.23–2.35 (2H, m, 3-HH and 5-HH), 2.65 (1H, dd,  $J$  15.0, 7.5 Hz, 5-HH), 2.70 (1H, dd,  $J$  15.0, 8.0 Hz, 3-HH), 4.52–4.62 (1H, m, 6-H), 4.64–4.76 (1H, m, 2-H);  $\delta_{\text{C}}$  (126 MHz,  $\text{CDCl}_3$ ) 14.0 (CH<sub>3</sub>), 20.2 (CH<sub>2</sub>), 22.8 (CH<sub>3</sub>), 28.6 (3 × CH<sub>3</sub>), 39.3 (CH<sub>2</sub>), 43.9 (CH<sub>2</sub>), 45.6 (CH<sub>2</sub>), 48.5 (CH), 52.6 (CH), 80.3 (C), 154.9 (C), 208.9 (C);  $m/z$  (ESI) 278.1715 (MNa<sup>+</sup>. C<sub>14</sub>H<sub>25</sub>NNaO<sub>3</sub> requires 278.1727).

***tert*-Butyl (2*R*,6*S*)-6-butyl-2-methyl-4-oxopiperidine-1-carboxylate (12b):**  $\nu_{\max}/\text{cm}^{-1}$  2929 (C-H), 1692 (C=O), 1367, 1175;  $[\alpha]_{\text{D}}^{20} -9.2$  ( $c$  0.1,  $\text{CHCl}_3$ );  $\delta_{\text{H}}$  (500 MHz,  $\text{CDCl}_3$ ) 0.89 (3H, t,  $J$  7.1 Hz, 4''-H<sub>3</sub>), 1.26 (3H, d,  $J$  7.0 Hz, 1'-H<sub>3</sub>), 1.29–1.38 (4H, m, 2''-H<sub>2</sub> and 3''-H<sub>2</sub>), 1.44–1.53 (10H, m, 3 × CH<sub>3</sub> and 1''-HH), 1.55–1.66 (1H, m, 1''-HH), 2.28 (1H, ddd,  $J$  15.0, 4.0, 1.8 Hz, 3-HH), 2.33 (1H, dt,  $J$  15.0, 1.8 Hz, 5-HH), 2.67 (1H, ddd,  $J$  15.0, 7.5, 1.0 Hz, 5-HH), 2.72 (1H, ddd,  $J$  15.0, 8.0, 1.0 Hz, 3-HH), 4.48–4.64 (1H, m, 6-H), 4.64–4.80 (1H, m, 2-H);  $\delta_{\text{C}}$  (126 MHz,  $\text{CDCl}_3$ ) 14.1 (CH<sub>3</sub>), 22.6 (CH<sub>2</sub>), 22.8 (CH<sub>3</sub>), 28.6 (3 × CH<sub>3</sub>), 29.3 (CH<sub>2</sub>), 36.8 (CH<sub>2</sub>), 44.0 (CH<sub>2</sub>), 45.7 (CH<sub>2</sub>), 48.5 (CH), 52.9 (CH), 80.3 (C), 155.0 (C), 208.9 (C);  $m/z$  (ESI) 292.1887 (MNa<sup>+</sup>. C<sub>15</sub>H<sub>27</sub>NNaO<sub>3</sub> requires 292.1889).

***tert*-Butyl (2*R*,6*S*)-2-methyl-4-oxo-6-pentylpiperidine-1-carboxylate (12c):**  $\nu_{\max}/\text{cm}^{-1}$  2931 (C-H), 1690 (C=O), 1350, 1173, 1072;  $[\alpha]_{\text{D}}^{20} -20.8$  ( $c$  0.2,  $\text{CHCl}_3$ );  $\delta_{\text{H}}$  (500 MHz,  $\text{CDCl}_3$ ) 0.81 (3H, t,  $J$  7.1 Hz, 5''-H<sub>3</sub>), 1.15–1.30 (9H, m, 1'-H<sub>3</sub>, 2''-H<sub>2</sub>, 3''-H<sub>2</sub> and 4''-H<sub>2</sub>), 1.38–1.47 (10H, m, 3 × CH<sub>3</sub> and 1''-HH), 1.49–1.57 (1H, m, 1''-HH), 2.21 (1H, ddd,  $J$  15.0, 3.5, 1.5 Hz, 3-HH), 2.26 (1H, dt,  $J$  15.0, 1.5 Hz, 5-HH), 2.59 (1H, ddd,  $J$  15.0, 8.0, 1.0 Hz, 5-HH), 2.64 (1H, ddd,  $J$  15.0, 8.0, 1.0 Hz, 3-HH), 4.43–4.57 (1H, m, 6-H), 4.58–4.74 (1H, m, 2-H);  $\delta_{\text{C}}$  (126 MHz,  $\text{CDCl}_3$ ) 14.1 (CH<sub>3</sub>), 22.7 (CH<sub>2</sub>), 22.8 (CH<sub>3</sub>), 26.8 (CH<sub>2</sub>), 28.6 (3 × CH<sub>3</sub>), 31.7 (CH<sub>2</sub>), 37.1 (CH<sub>2</sub>), 44.0 (CH<sub>2</sub>), 45.7 (CH<sub>2</sub>), 48.5 (CH), 52.9 (CH), 80.3 (C), 155.0 (C), 209.0 (C);  $m/z$  (ESI) 306.2039 (MNa<sup>+</sup>. C<sub>16</sub>H<sub>29</sub>NNaO<sub>3</sub> requires 306.2040).

***tert*-Butyl (2*R*,6*S*)-2-methyl-6-octyl-4-oxopiperidine-1-carboxylate (12d):**  $\nu_{\max}/\text{cm}^{-1}$  2924 (C-H), 1721 (C=O), 1690 (C=O), 1350, 1173;  $[\alpha]_{\text{D}}^{30} -15.4$  ( $c$  0.2,  $\text{CHCl}_3$ );  $\delta_{\text{H}}$  (500 MHz,  $\text{CDCl}_3$ ) 0.87 (3H, t,  $J$  7.0 Hz, 8''-H<sub>3</sub>), 1.15–1.39 (15H, m, 1'-H<sub>3</sub>, 2''-H<sub>2</sub>, 3''-H<sub>2</sub>, 4''-H<sub>2</sub>, 5''-H<sub>2</sub>, 6''-H<sub>2</sub> and 7''-H<sub>2</sub>), 1.48–1.64 (11H, m, 3 × CH<sub>3</sub> and 1''-H<sub>2</sub>), 2.27 (1H, ddd,  $J$  15.0, 3.5, 1.5 Hz, 3-HH), 2.32 (1H, dt,  $J$  15.0, 1.5 Hz, 5-HH), 2.66 (1H, dd,  $J$  15.0, 7.5 Hz, 5-HH), 2.72 (1H, dd,  $J$  15.0, 8.0 Hz, 3-HH), 4.49–

4.62 (1H, m, 6-H), 4.64–4.76 (1H, m, 2-H);  $\delta_{\text{C}}$  (126 MHz,  $\text{CDCl}_3$ ) 14.2 ( $\text{CH}_3$ ), 22.8 ( $\text{CH}_2$  and  $\text{CH}_3$ ), 27.1 ( $\text{CH}_2$ ), 28.6 ( $3 \times \text{CH}_3$ ), 29.3 ( $\text{CH}_2$ ), 29.5 ( $\text{CH}_2$ ), 29.7 ( $\text{CH}_2$ ), 32.0 ( $\text{CH}_2$ ), 37.2 ( $\text{CH}_2$ ), 44.0 ( $\text{CH}_2$ ), 45.7 ( $\text{CH}_2$ ), 48.5 (CH), 52.9 (CH), 80.3 (C), 154.9 (C), 209.0 (C);  $m/z$  (ESI) 348.2494 ( $\text{MNa}^+$ ,  $\text{C}_{19}\text{H}_{35}\text{NNaO}_3$  requires 348.2509).

***tert*-Butyl (2*R*,6*S*)-2-methyl-6-nonyl-4-oxopiperidine-1-carboxylate (12e).**<sup>1</sup> Spectroscopic data were consistent with the literature.<sup>1</sup>  $[\alpha]_{\text{D}}^{23} -13.4$  ( $c$  0.9,  $\text{CHCl}_3$ );  $\delta_{\text{H}}$  (500 MHz,  $\text{CDCl}_3$ ) 0.87 (3H, t,  $J$  7.0 Hz, 9''-H<sub>3</sub>), 1.17–1.40 (17H, m, 1'-H<sub>3</sub>, 2''-H<sub>2</sub>, 3''-H<sub>2</sub>, 4''-H<sub>2</sub>, 5''-H<sub>2</sub>, 6''-H<sub>2</sub>, 7''-H<sub>2</sub> and 8''-H<sub>2</sub>), 1.44–1.65 (11H, 3  $\times$   $\text{CH}_3$  and 1''-H<sub>2</sub>), 2.27 (1H, ddd,  $J$  15.0, 4.0, 2.0 Hz, 3-*HH*), 2.32 (1H, dt,  $J$  15.0, 2.0 Hz, 5-*HH*), 2.66 (1H, dd,  $J$  15.0, 7.5 Hz, 5-*HH*), 2.71 (1H, dd,  $J$  15.0, 8.0 Hz, 3-*HH*), 4.53–4.63 (1H, m, 6-H), 4.66–4.76 (1H, m, 2-H);  $\delta_{\text{C}}$  (126 MHz,  $\text{CHCl}_3$ ) 14.2 ( $\text{CH}_3$ ), 22.8 ( $\text{CH}_2$  and  $\text{CH}_3$ ), 27.1 ( $\text{CH}_2$ ), 28.6 ( $3 \times \text{CH}_3$ ), 29.4 ( $\text{CH}_2$ ), 29.5 ( $\text{CH}_2$ ), 29.6 ( $\text{CH}_2$ ), 29.7 ( $\text{CH}_2$ ), 32.0 ( $\text{CH}_2$ ), 37.2 ( $\text{CH}_2$ ), 44.0 ( $\text{CH}_2$ ), 45.7 ( $\text{CH}_2$ ), 48.5 (CH), 52.9 (CH), 80.3 (C), 154.9 (C), 209.0 (C);  $m/z$  (ESI) 362 ( $\text{MNa}^+$ , 100%).

***tert*-Butyl (2*R*,6*S*)-2-methyl-4-oxo-6-undecylpiperidine-1-carboxylate (12f).**<sup>1</sup> Spectroscopic data were consistent with the literature.<sup>1</sup>  $[\alpha]_{\text{D}}^{24} -11.0$  ( $c$  0.9,  $\text{CHCl}_3$ );  $\delta_{\text{H}}$  (400 MHz,  $\text{CDCl}_3$ ) 0.88 (3H, t,  $J$  6.9 Hz, 11''-H<sub>3</sub>), 1.18–1.40 (21H, m, 1'-H<sub>3</sub>, 2''-H<sub>2</sub>, 3''-H<sub>2</sub>, 4''-H<sub>2</sub>, 5''-H<sub>2</sub>, 6''-H<sub>2</sub>, 7''-H<sub>2</sub>, 8''-H<sub>2</sub>, 9''-H<sub>2</sub> and 10''-H<sub>2</sub>), 1.45–1.65 (11H, 3  $\times$   $\text{CH}_3$  and 1''-H<sub>2</sub>), 2.28 (1H, ddd,  $J$  15.0, 3.6, 1.6 Hz, 3-*HH*), 2.32 (1H, dt,  $J$  15.0, 1.6 Hz, 5-*HH*), 2.66 (1H, dd,  $J$  15.0, 7.6 Hz, 5-*HH*), 2.71 (1H, dd,  $J$  15.0, 8.0 Hz, 3-*HH*), 4.51–4.64 (1H, m, 6-H), 4.65–4.80 (1H, m, 2-H);  $\delta_{\text{C}}$  (101 MHz,  $\text{CHCl}_3$ ) 14.2 ( $\text{CH}_3$ ), 22.8 ( $\text{CH}_2$  and  $\text{CH}_3$ ), 27.1 ( $\text{CH}_2$ ), 28.6 ( $3 \times \text{CH}_3$ ), 29.5 ( $2 \times \text{CH}_2$ ), 29.7 ( $4 \times \text{CH}_2$ ), 32.0 ( $\text{CH}_2$ ), 37.2 ( $\text{CH}_2$ ), 44.0 ( $\text{CH}_2$ ), 45.7 ( $\text{CH}_2$ ), 48.5 (CH), 52.9 (CH), 80.3 (C), 154.9 (C), 208.9 (C);  $m/z$  (ESI) 390 ( $\text{MNa}^+$ , 100%).

***tert*-Butyl (2*R*,6*S*)-6-(4''-chlorobutyl)-2-methyl-4-oxopiperidine-1-carboxylate (12g).**  $\nu_{\text{max}}/\text{cm}^{-1}$  (neat) 2970 (CH), 1720 (C=O), 1682 (C=O), 1350, 1165;  $[\alpha]_{\text{D}}^{24} -10.2$  ( $c$  1.2,  $\text{CHCl}_3$ );  $\delta_{\text{H}}$  (400 MHz,  $\text{CDCl}_3$ ) 1.26 (3H, d,  $J$  7.0 Hz, 1'-H<sub>3</sub>), 1.41–1.57 (12H, m, 3  $\times$   $\text{CH}_3$ , 1''-*HH* and 2''-H<sub>2</sub>), 1.60–1.69 (1H, m, 1''-*HH*), 1.72–1.85 (2H, m, 3''-H<sub>2</sub>), 2.27 (1H, ddd,  $J$  14.8, 3.6, 1.8 Hz, 3-*HH*), 2.32 (1H, dt,  $J$  14.8, 1.8 Hz, 5-*HH*), 2.68 (1H, dd,  $J$  14.8, 7.6 Hz, 5-*HH*), 2.73 (1H, dd,  $J$  14.8, 8.0 Hz, 3-*HH*), 3.52 (2H, t,  $J$  6.5 Hz, 4''-H<sub>2</sub>), 4.53–4.66 (1H, m, 6-H), 4.67–4.80 (1H, m, 2-H);  $\delta_{\text{C}}$  (101 MHz,  $\text{CDCl}_3$ ) 22.7 ( $\text{CH}_3$ ), 24.2 ( $\text{CH}_2$ ), 28.4 ( $3 \times \text{CH}_3$ ), 32.1 ( $\text{CH}_2$ ), 36.2 ( $\text{CH}_2$ ), 43.8 ( $\text{CH}_2$ ), 44.8 ( $\text{CH}_2$ ), 45.5 ( $\text{CH}_2$ ), 48.5 (CH), 52.5 (CH), 80.4 (C), 154.8 (C), 208.5 (C);  $m/z$  (ESI) 326.1480 ( $\text{MNa}^+$ ,  $\text{C}_{15}\text{H}_{26}^{35}\text{ClNNaO}_3$  requires 326.1493).

***tert*-Butyl (2*R*,6*S*)-6-(2''-cyclohexylethyl)-2-methyl-4-oxopiperidine-1-carboxylate (12h).**

$\nu_{\max}/\text{cm}^{-1}$  (neat) 2924 (CH), 1721 (C=O), 1690 (C=O), 1358, 1173;  $[\alpha]_{\text{D}}^{25} -12.3$  ( $c$  0.3,  $\text{CHCl}_3$ );  $\delta_{\text{H}}$  (400 MHz,  $\text{CDCl}_3$ ) 0.78–0.95 (2H, m,  $\text{CH}_2$ ), 1.04–1.35 (10H, m, 1'- $\text{H}_3$ , 2''- $\text{H}_2$ , 3''-H and  $2 \times \text{CH}_2$ ), 1.40–1.48 (10H, m,  $3 \times \text{CH}_3$  and 1''- $\text{HH}$ ), 1.60–1.74 (5H, m, 1''- $\text{HH}$  and  $2 \times \text{CH}_2$ ), 2.27 (1H, ddd,  $J$  15.0, 4.0, 1.6 Hz, 3- $\text{HH}$ ), 2.31 (1H, dt,  $J$  15.0, 1.6 Hz, 5- $\text{HH}$ ), 2.66 (1H, dd,  $J$  15.0, 7.6 Hz, 5- $\text{HH}$ ), 2.71 (1H, dd,  $J$  15.0, 8.0 Hz, 3- $\text{HH}$ ), 4.46–4.62 (1H, m, 6-H), 4.63–4.78 (1H, m, 2-H);  $\delta_{\text{C}}$  (101 MHz,  $\text{CDCl}_3$ ) 22.7 ( $\text{CH}_3$ ), 26.3 ( $2 \times \text{CH}_2$ ), 26.6 ( $\text{CH}_2$ ), 28.4 ( $3 \times \text{CH}_3$ ), 33.3 ( $\text{CH}_2$ ), 33.4 ( $\text{CH}_2$ ), 34.3 ( $\text{CH}_2$ ), 34.7 ( $\text{CH}_2$ ), 37.5 (CH), 43.9 ( $\text{CH}_2$ ), 45.6 ( $\text{CH}_2$ ), 48.4 (CH), 53.1 (CH), 80.2 (C), 154.8 (C), 208.8 (C);  $m/z$  (ESI) 346.2341 ( $\text{MNa}^+$ .  $\text{C}_{19}\text{H}_{33}\text{NNaO}_3$  requires 346.2353).

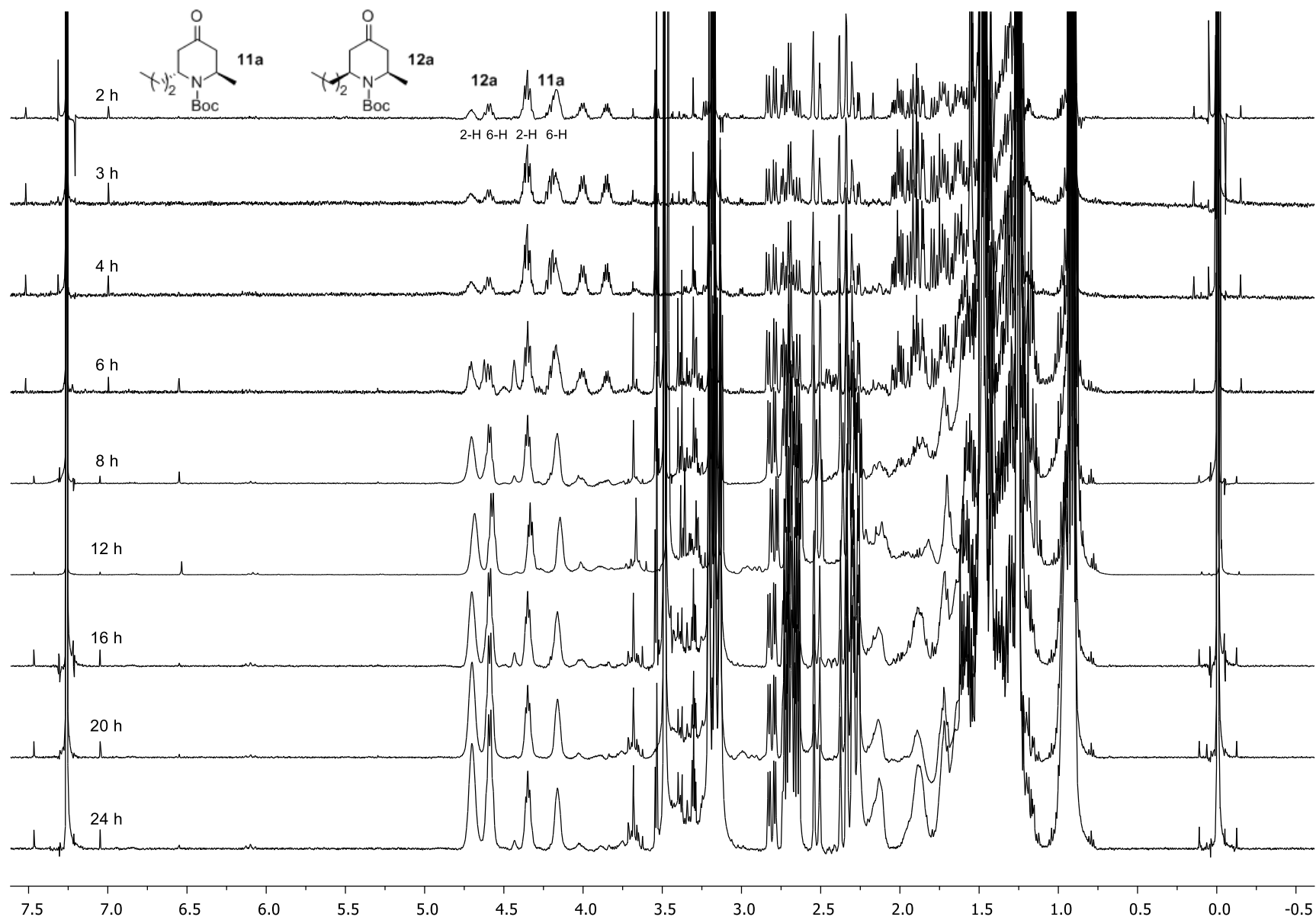
***tert*-Butyl (2*R*,6*S*)-2-methyl-4-oxo-6-(2''-phenylethyl)piperidine-1-carboxylate (12i).**  $\nu_{\max}/\text{cm}^{-1}$

(neat) 2974 (CH), 1719 (C=O), 1687 (C=O), 1353, 1169;  $[\alpha]_{\text{D}}^{19} +6.1$  ( $c$  1.0,  $\text{CHCl}_3$ );  $\delta_{\text{H}}$  (400 MHz,  $\text{CDCl}_3$ ) 1.31 (3H, d,  $J$  6.9 Hz, 1'- $\text{H}_3$ ), 1.48 (9H, s,  $3 \times \text{CH}_3$ ), 1.75–2.01 (2H, m, 1''- $\text{H}_2$ ), 2.30 (1H, dd,  $J$  15.2, 3.6 Hz, 3- $\text{HH}$ ), 2.38 (1H, br d,  $J$  15.2 Hz, 5- $\text{HH}$ ), 2.58 (1H, dd,  $J$  15.2, 5.6 Hz, 5- $\text{HH}$ ), 2.64 (1H, dd,  $J$  15.2, 6.0 Hz, 3- $\text{HH}$ ), 2.66–2.77 (2H, m, 2''- $\text{H}_2$ ), 4.60–4.82 (2H, m, 2-H and 6-H), 7.13–7.33 (5H, m, Ph);  $\delta_{\text{C}}$  (101 MHz,  $\text{CDCl}_3$ ) 22.8 ( $\text{CH}_3$ ), 28.4 ( $3 \times \text{CH}_3$ ), 33.4 ( $\text{CH}_2$ ), 39.0 ( $\text{CH}_2$ ), 43.7 ( $\text{CH}_2$ ), 45.5 ( $\text{CH}_2$ ), 48.4 (CH), 52.6 (CH), 80.4 (C), 126.1 (CH), 128.3 ( $2 \times \text{CH}$ ), 128.5 ( $2 \times \text{CH}$ ), 141.2 (C), 154.8 (C), 208.3 (C);  $m/z$  (ESI) 340.1878 ( $\text{MNa}^+$ .  $\text{C}_{19}\text{H}_{27}\text{NNaO}_3$  requires 340.1883).

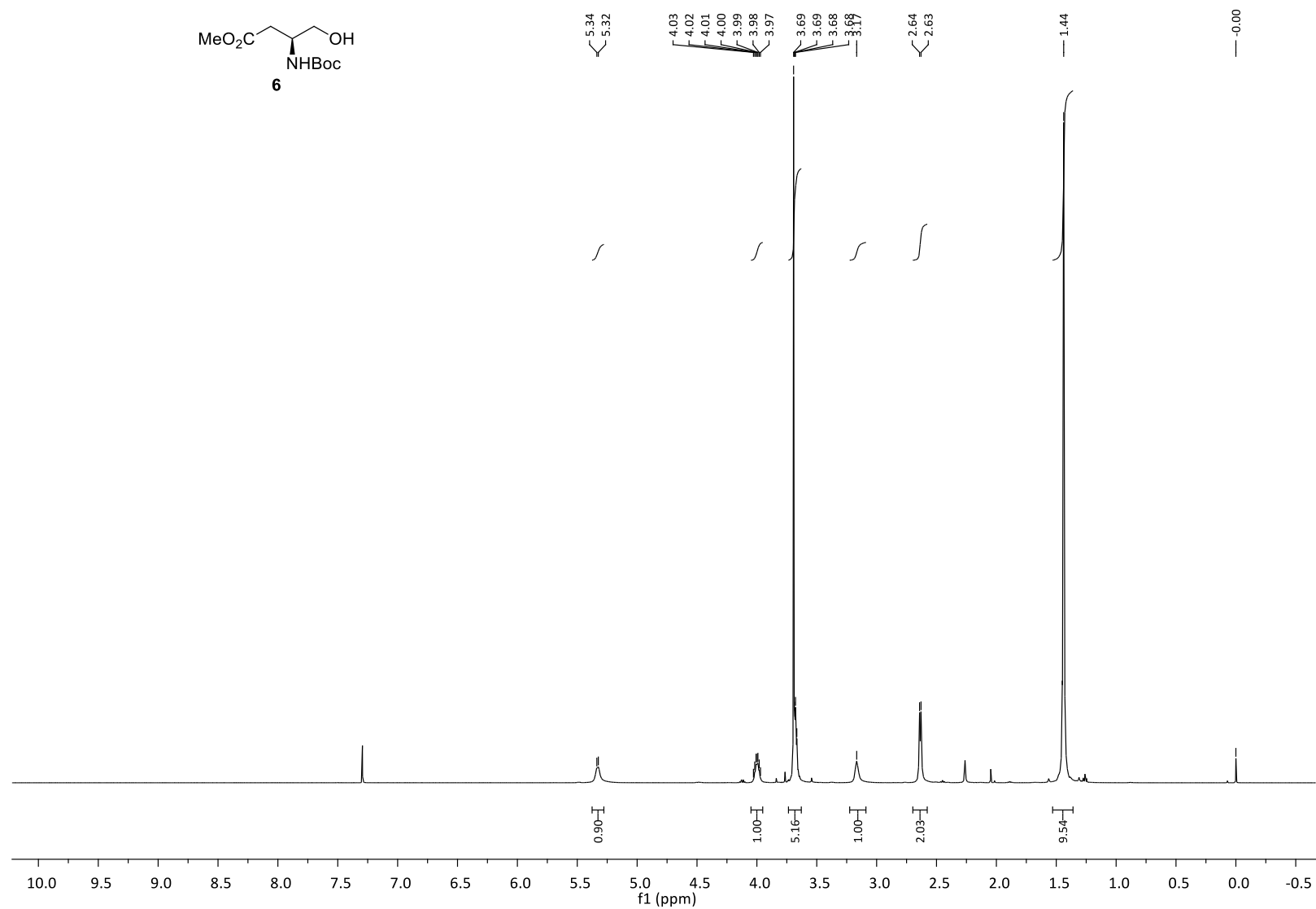
## 2. Reference

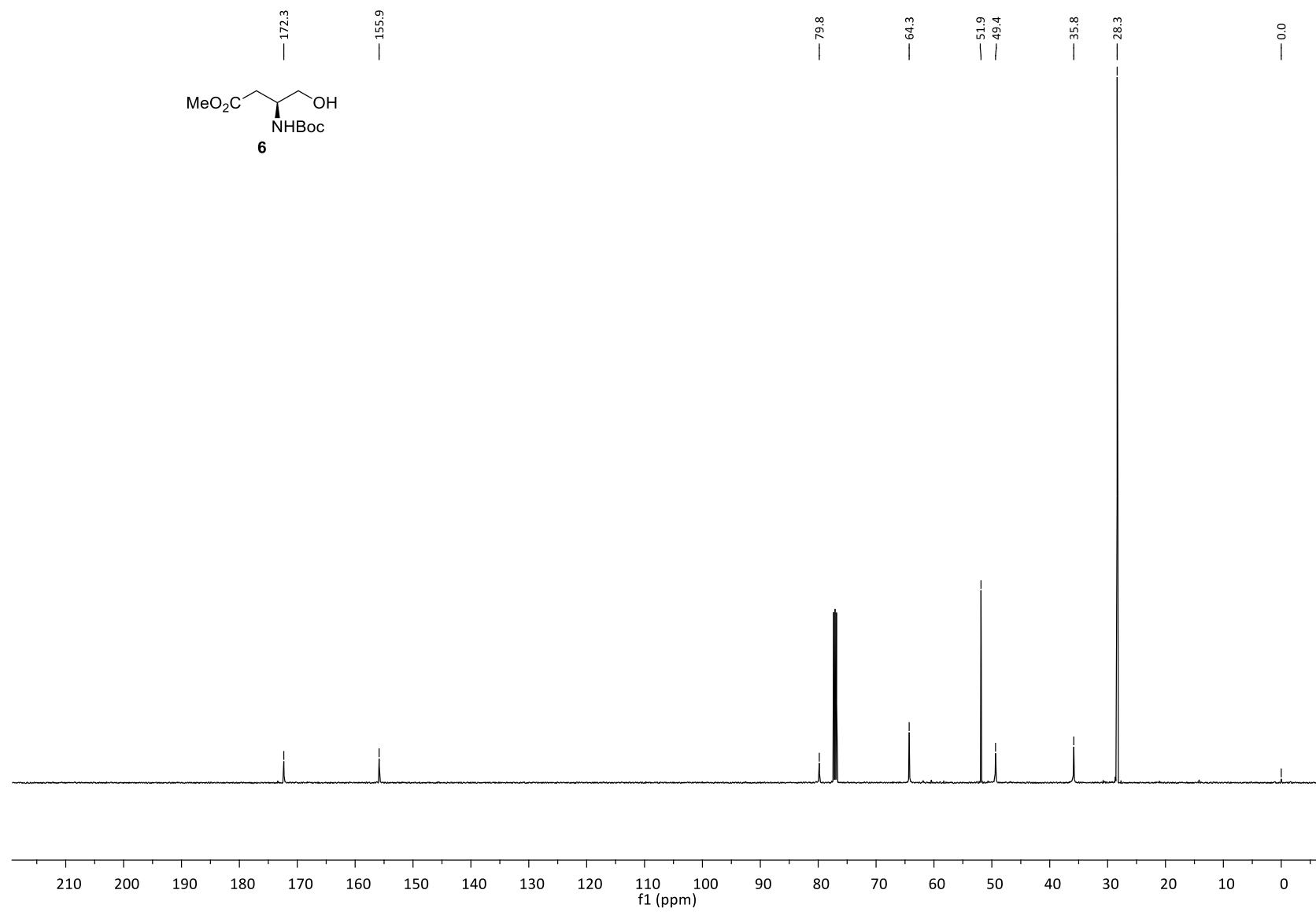
1. N. Gouault, M. Le Roch, G. de Campos Pinto and M. David, *Org. Biomol. Chem.*, 2012, **10**, 5541.

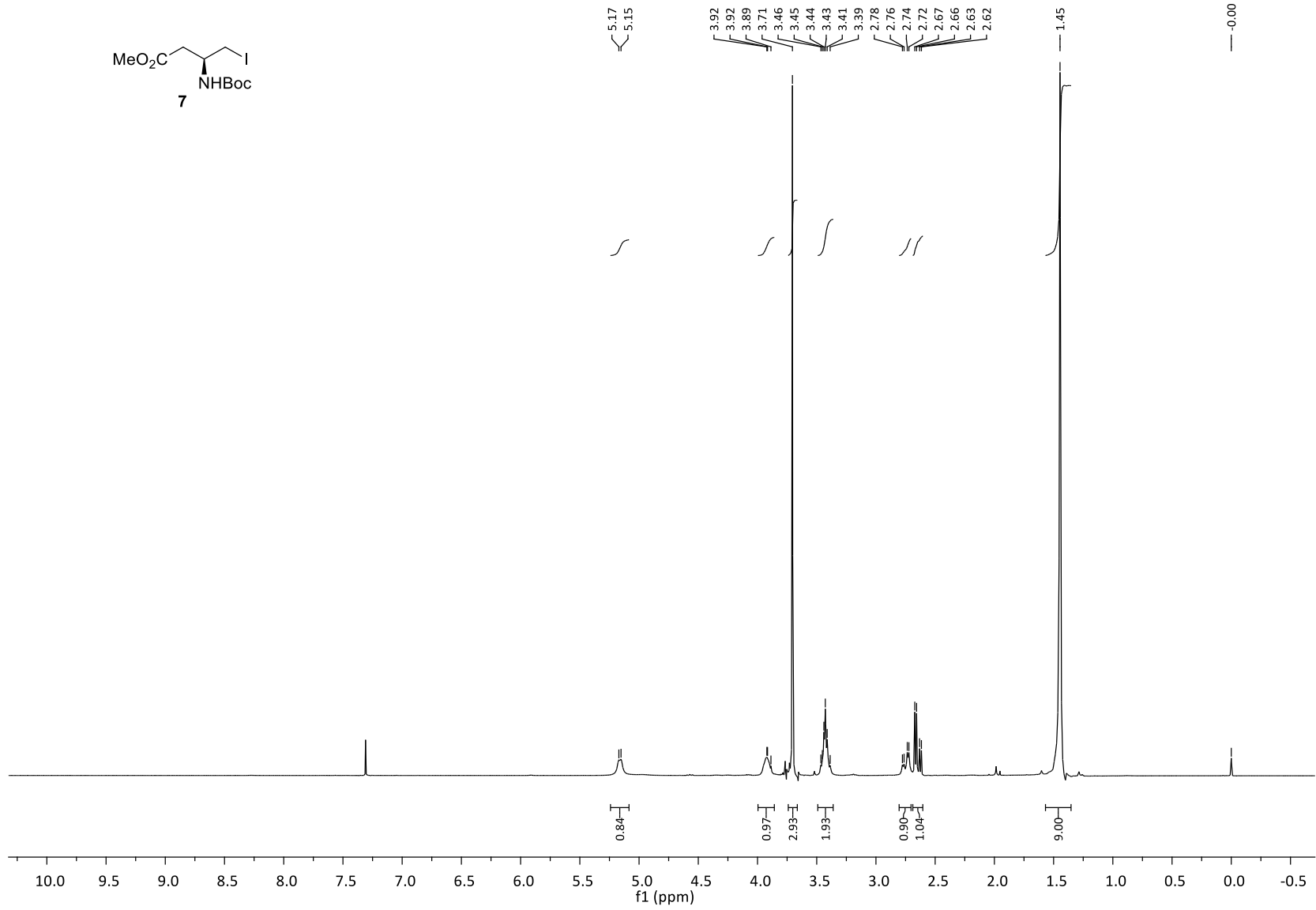
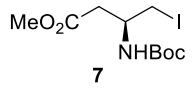
### 3. <sup>1</sup>H NMR Spectra for Cyclisation of 10a from 2–24 h



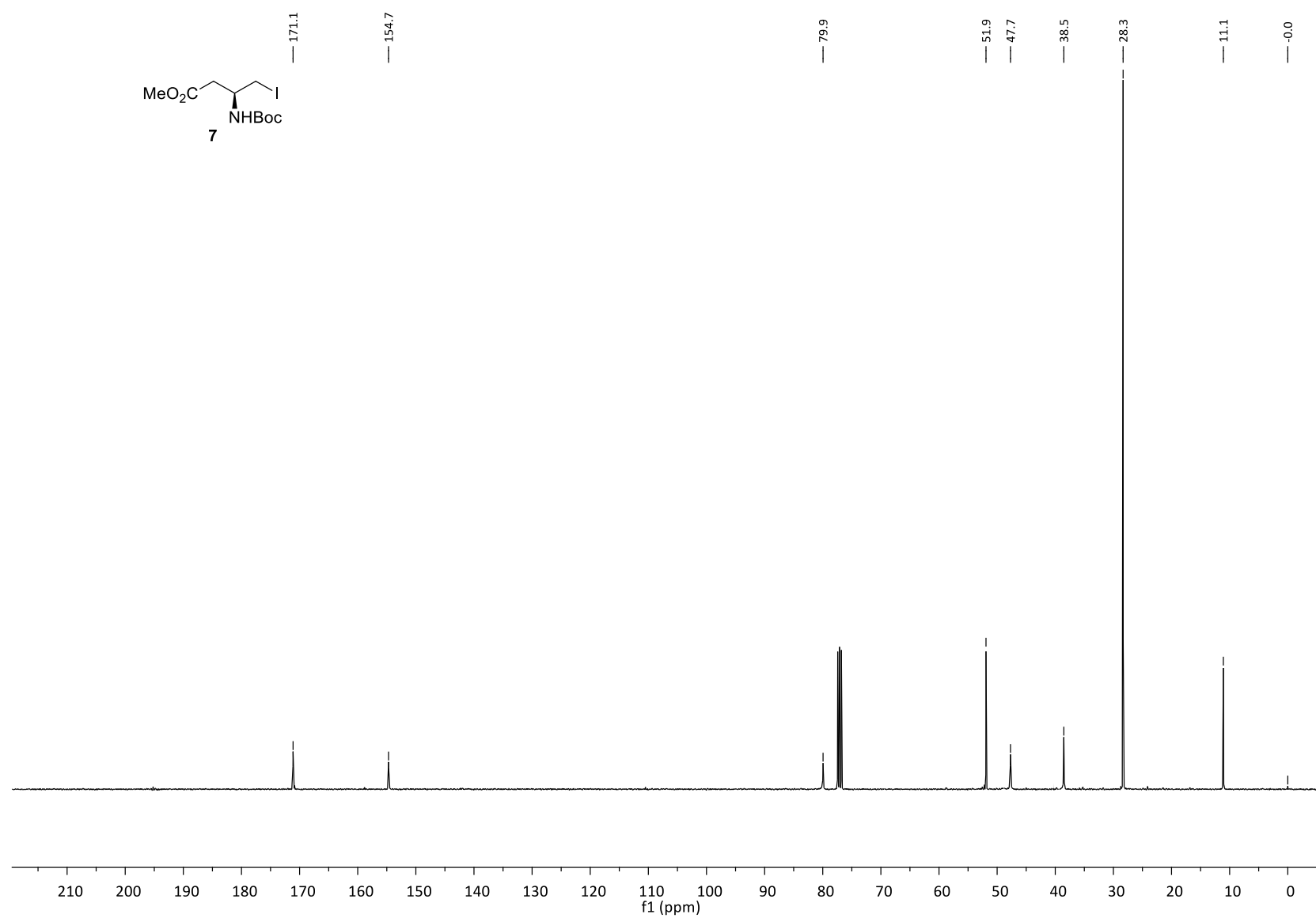
#### 4. <sup>1</sup>H NMR and <sup>13</sup>C NMR Spectra of All Compounds

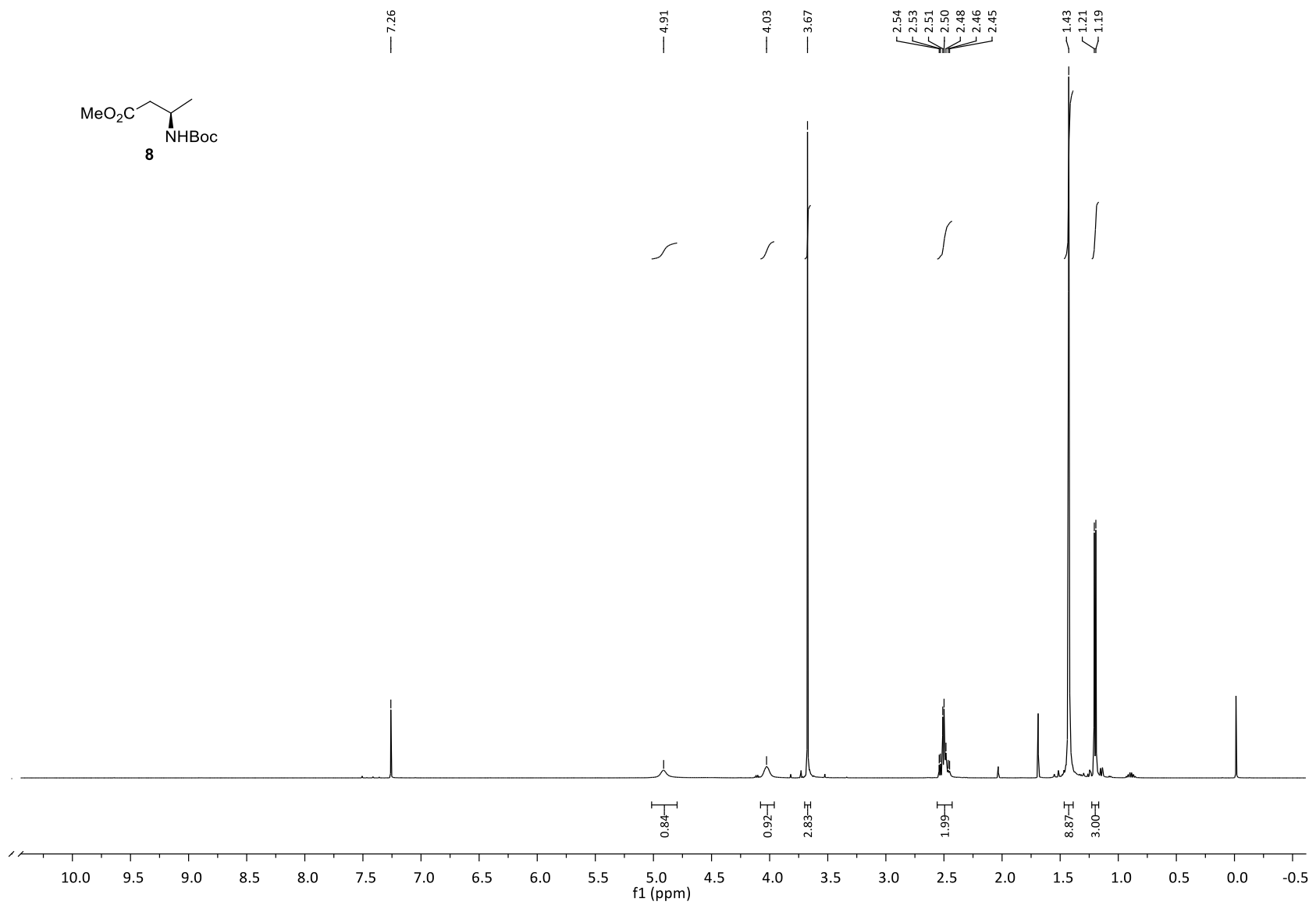
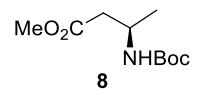


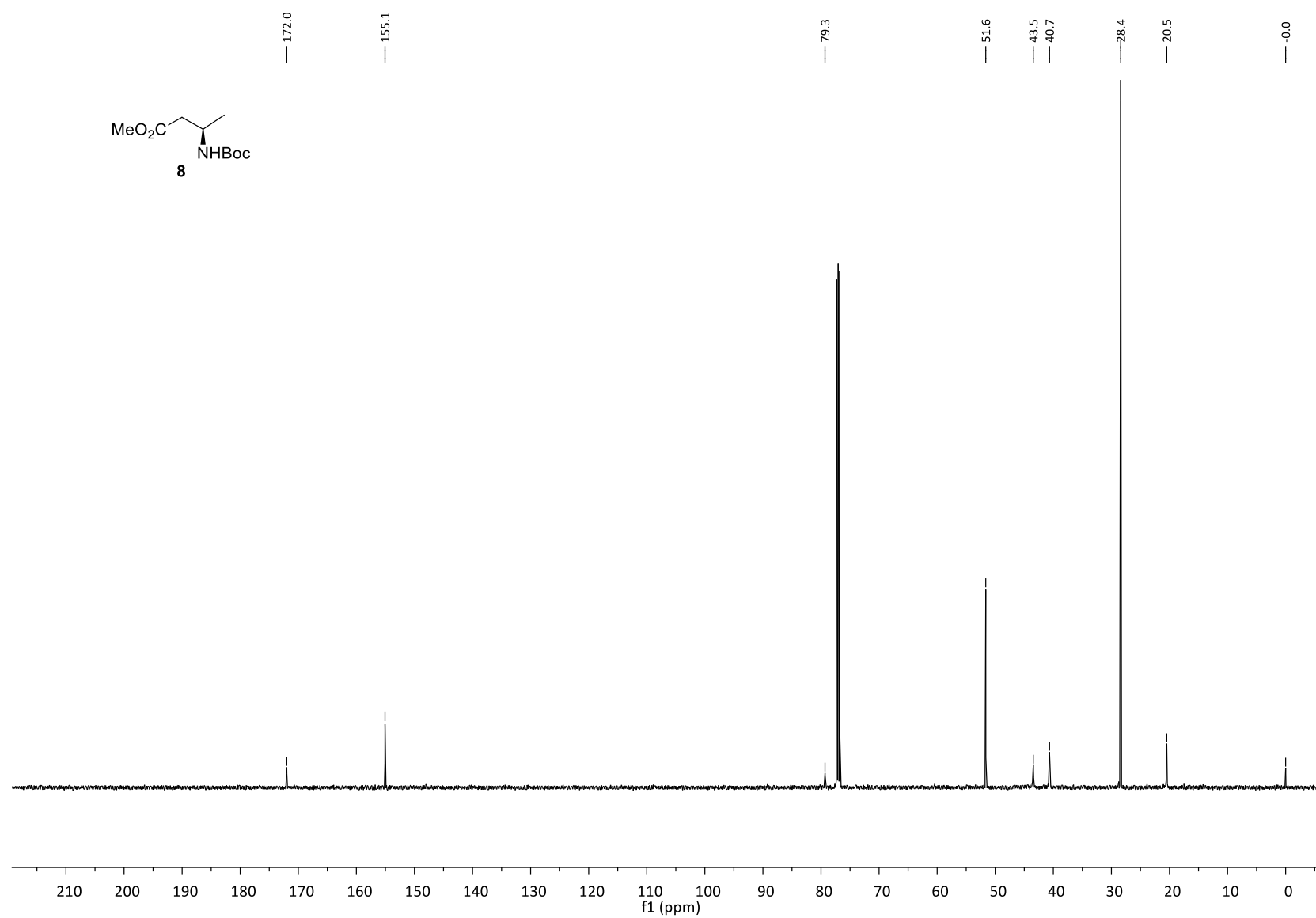


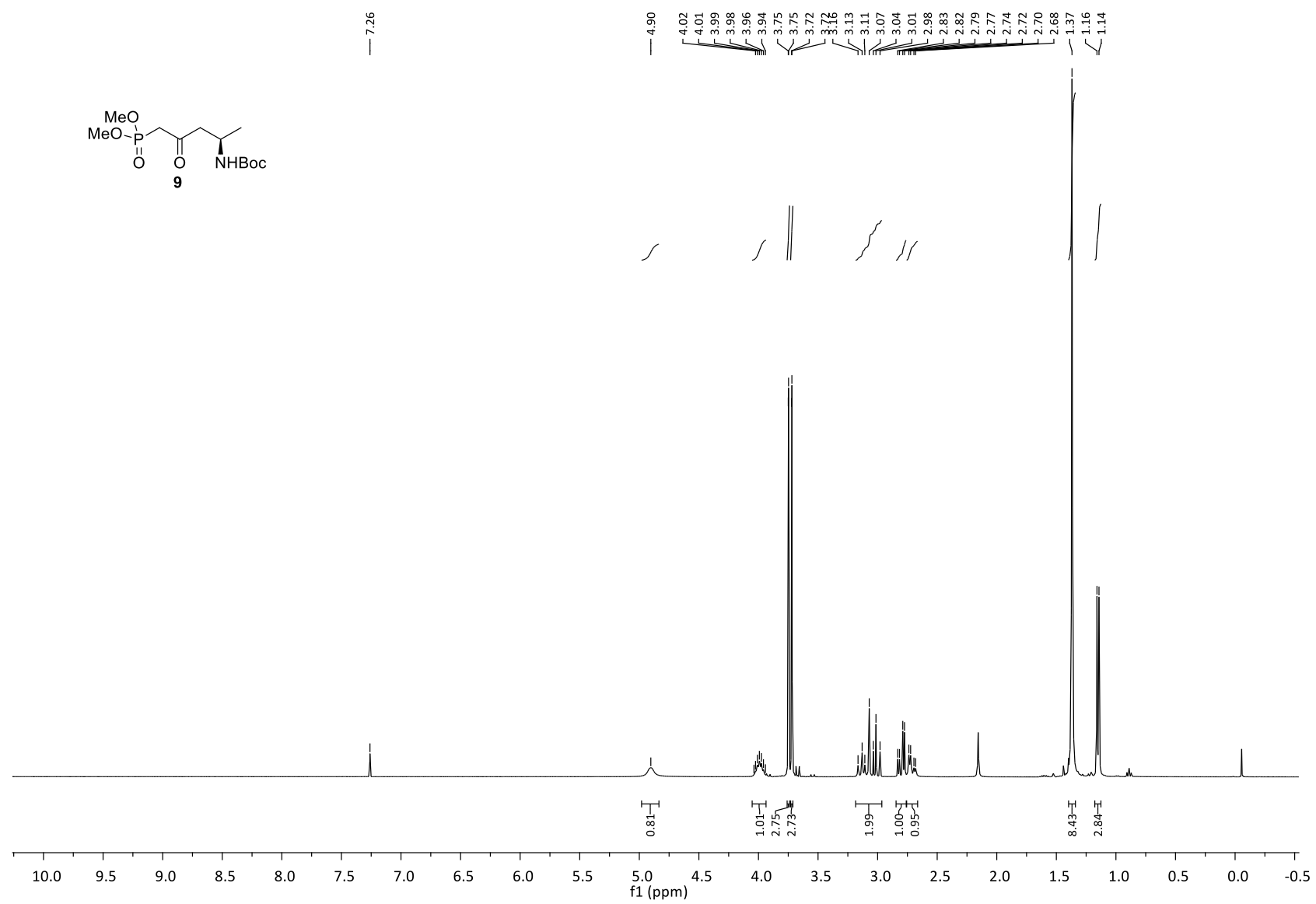


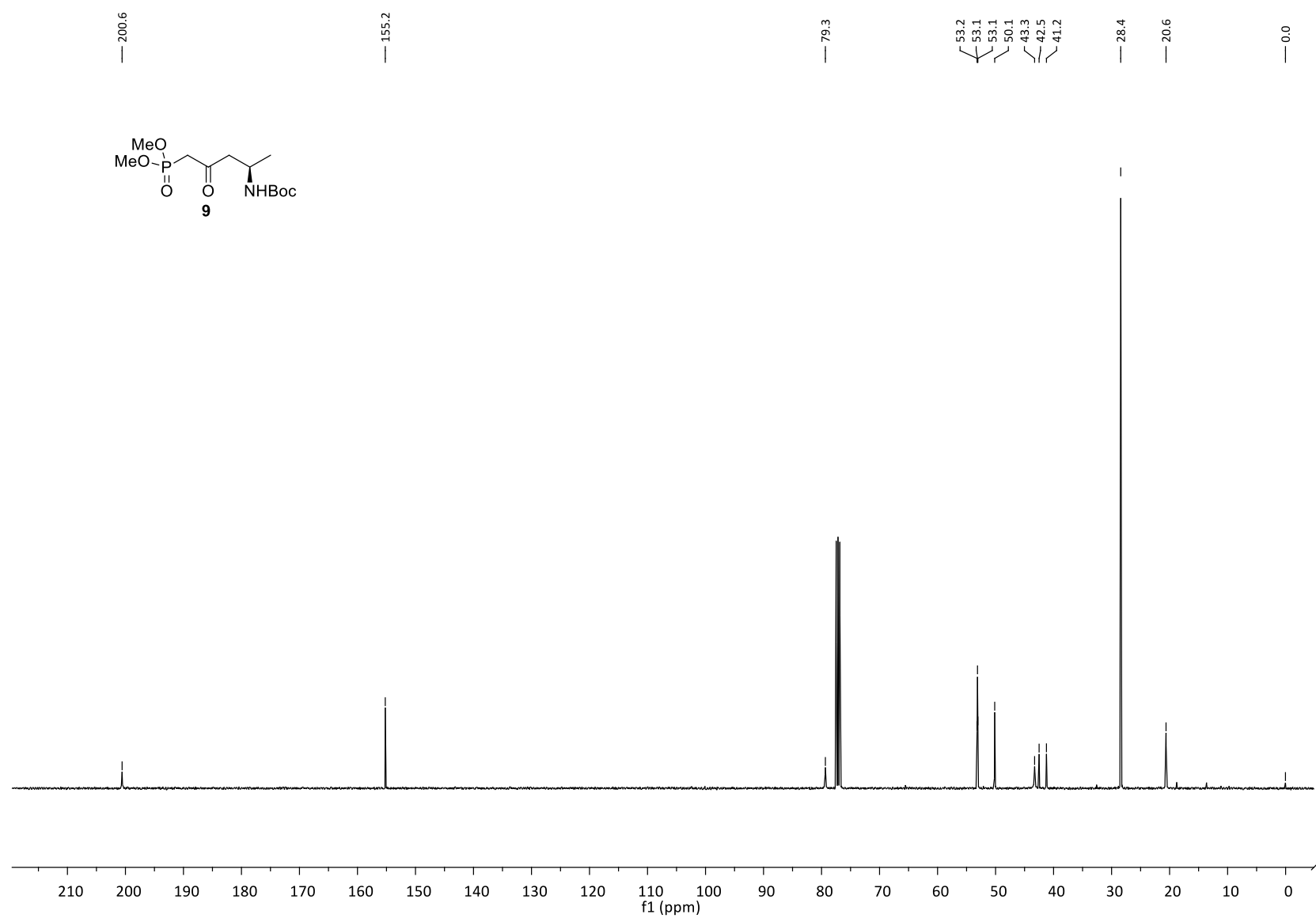


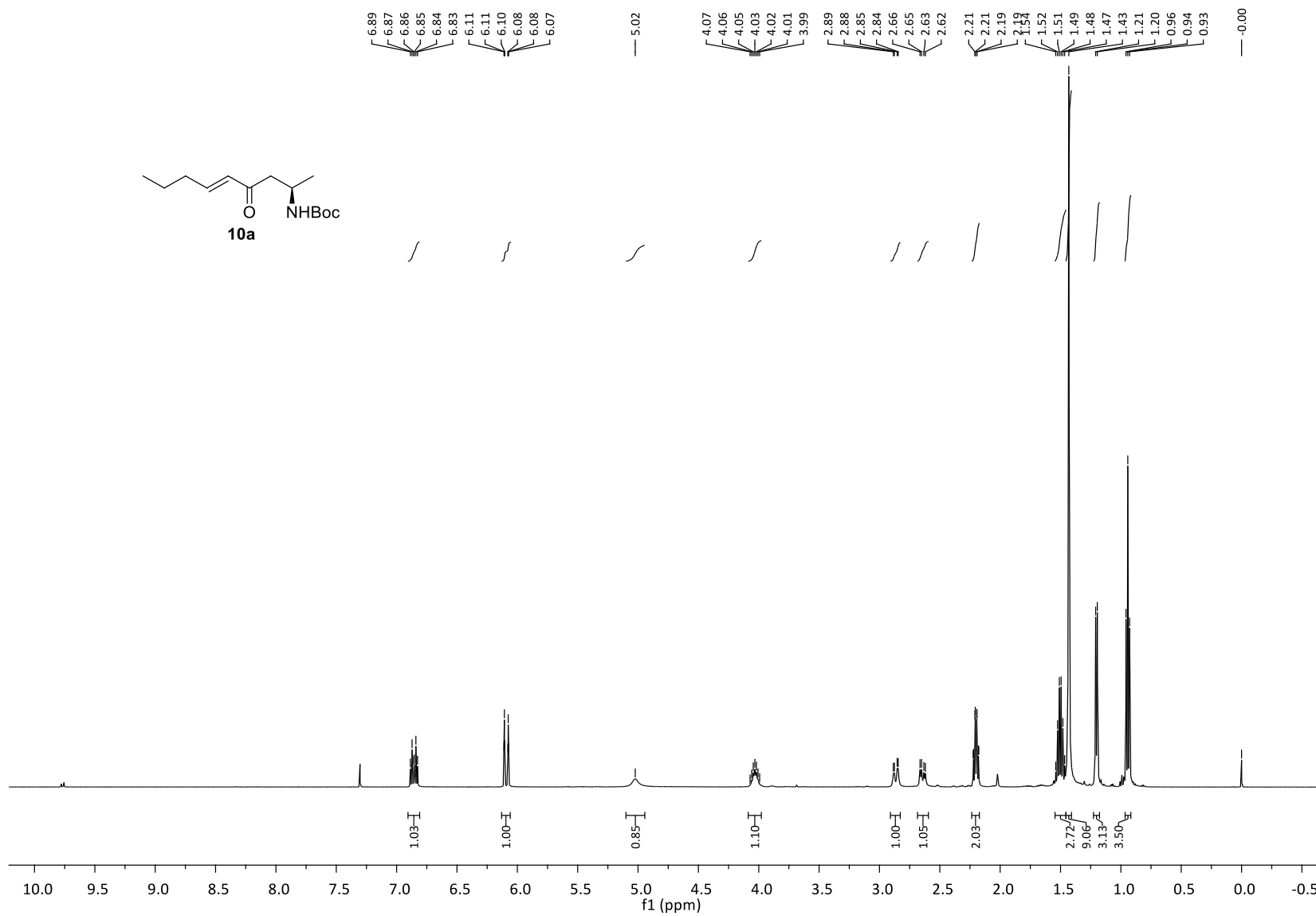


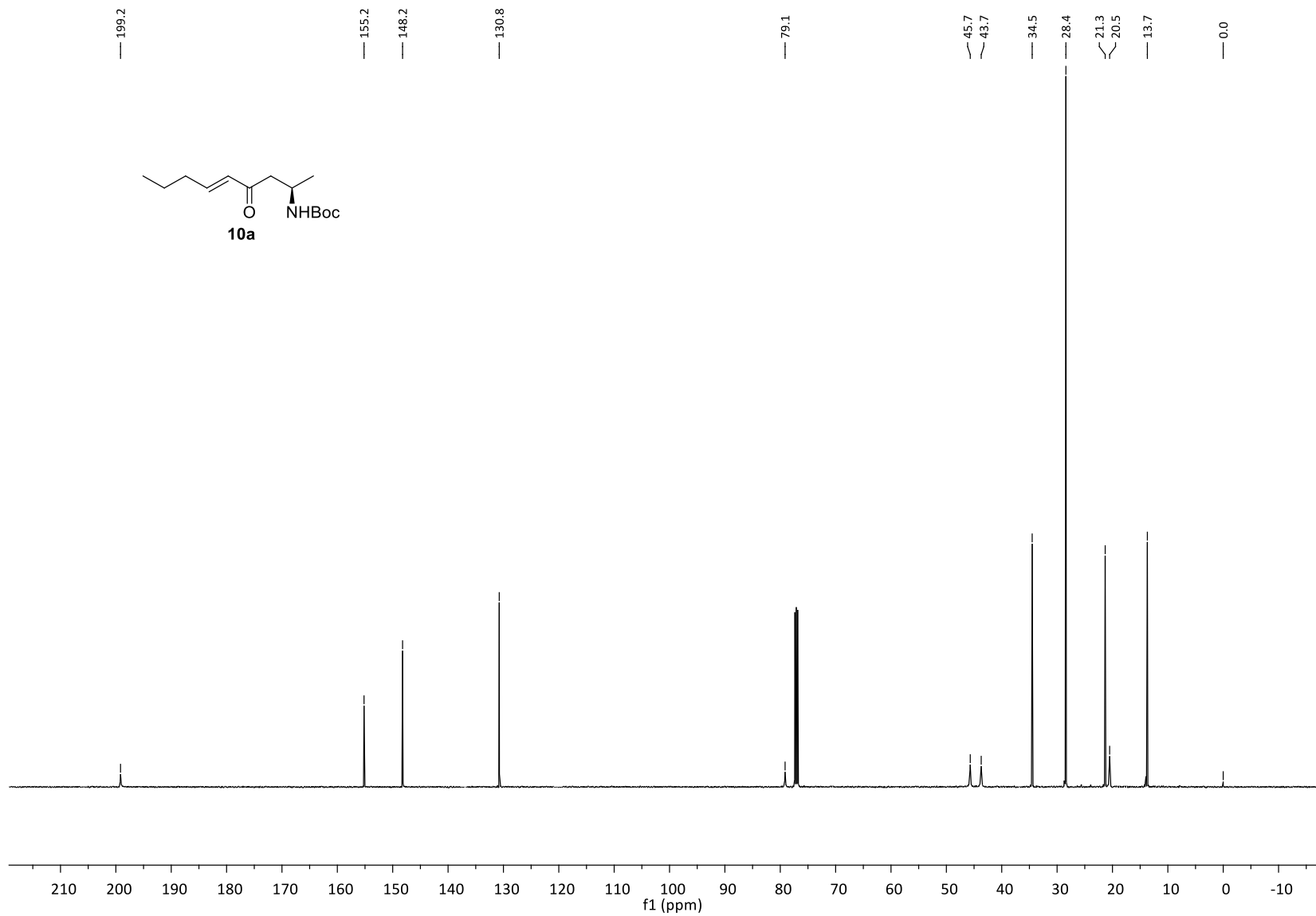


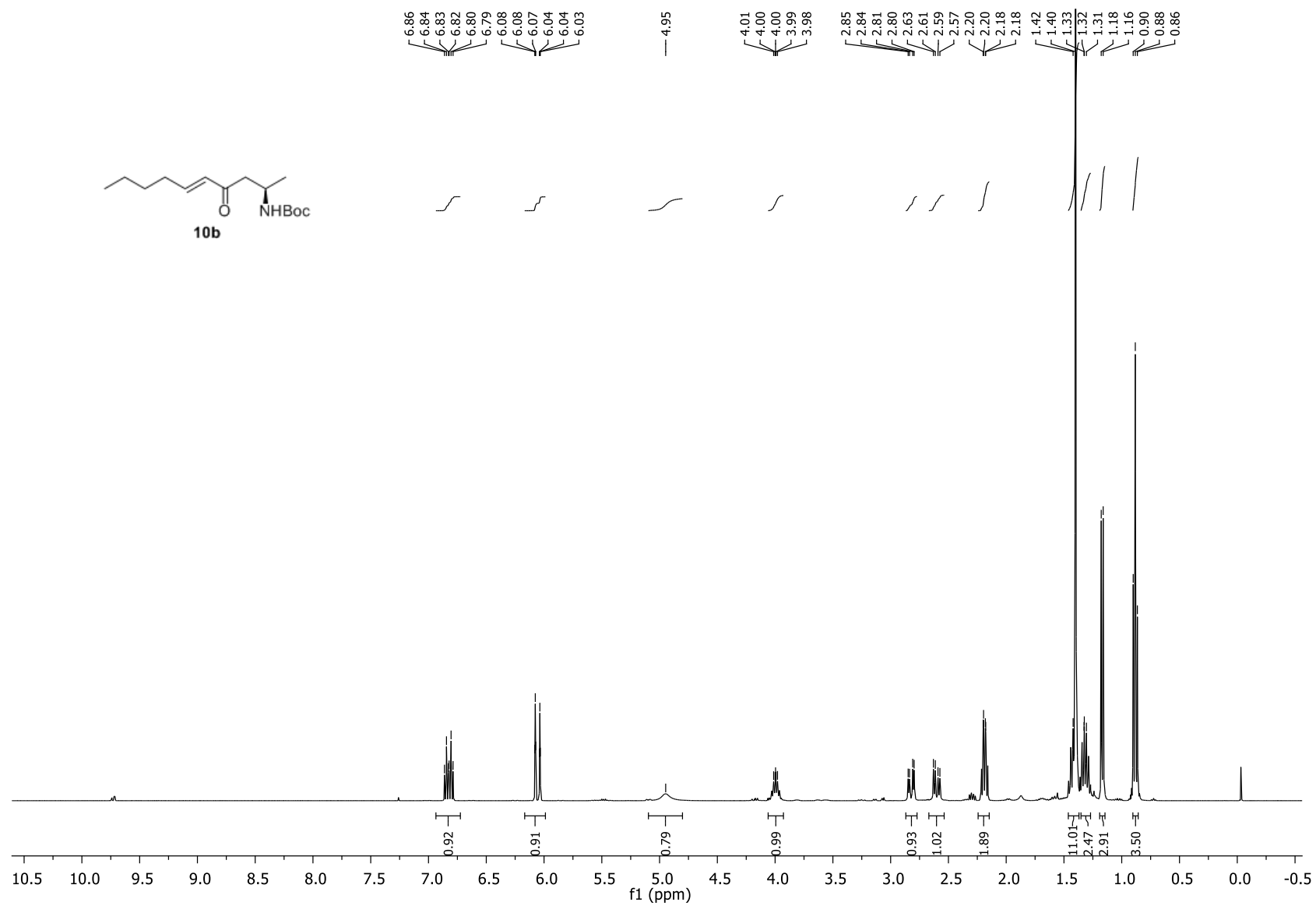




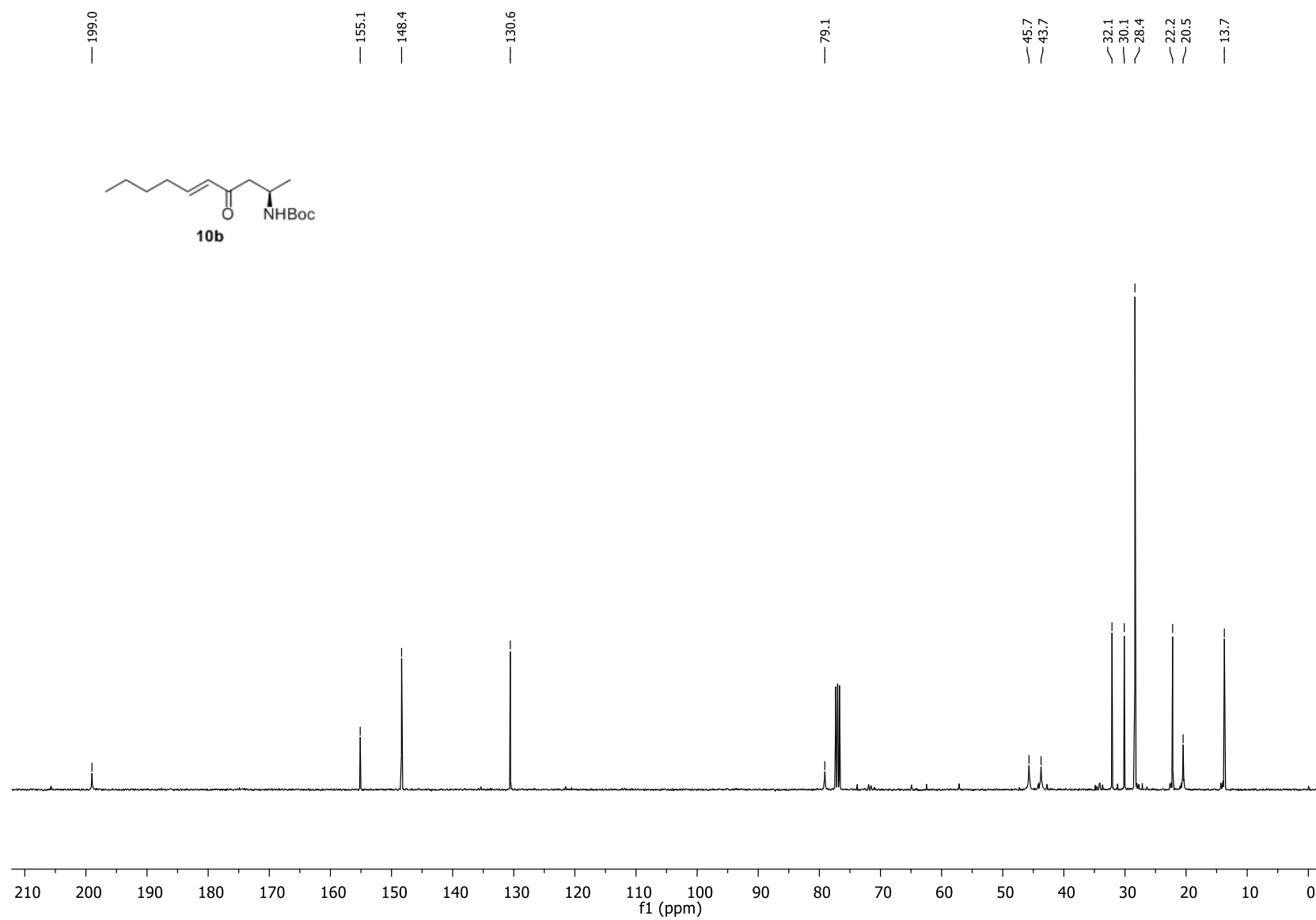


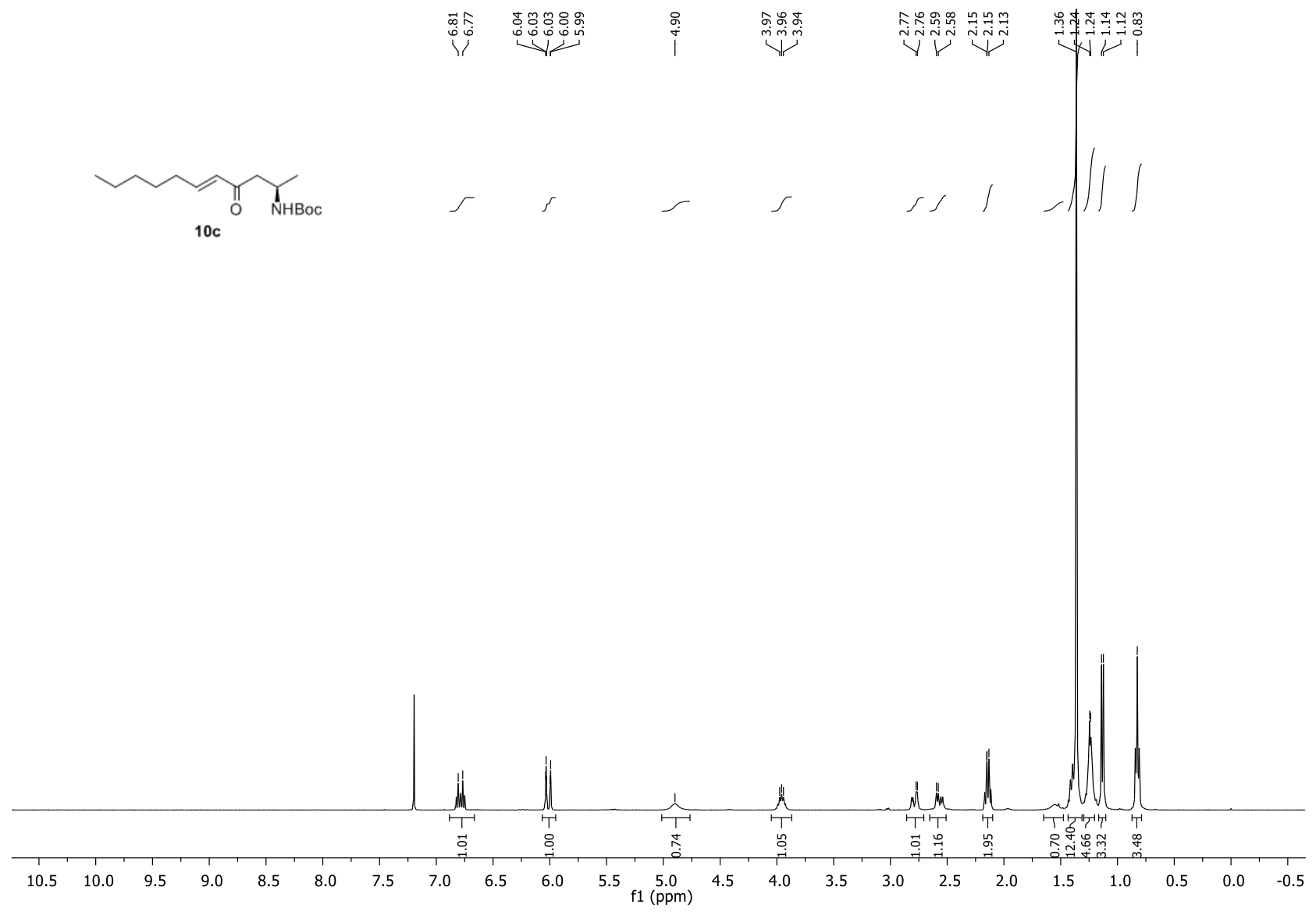


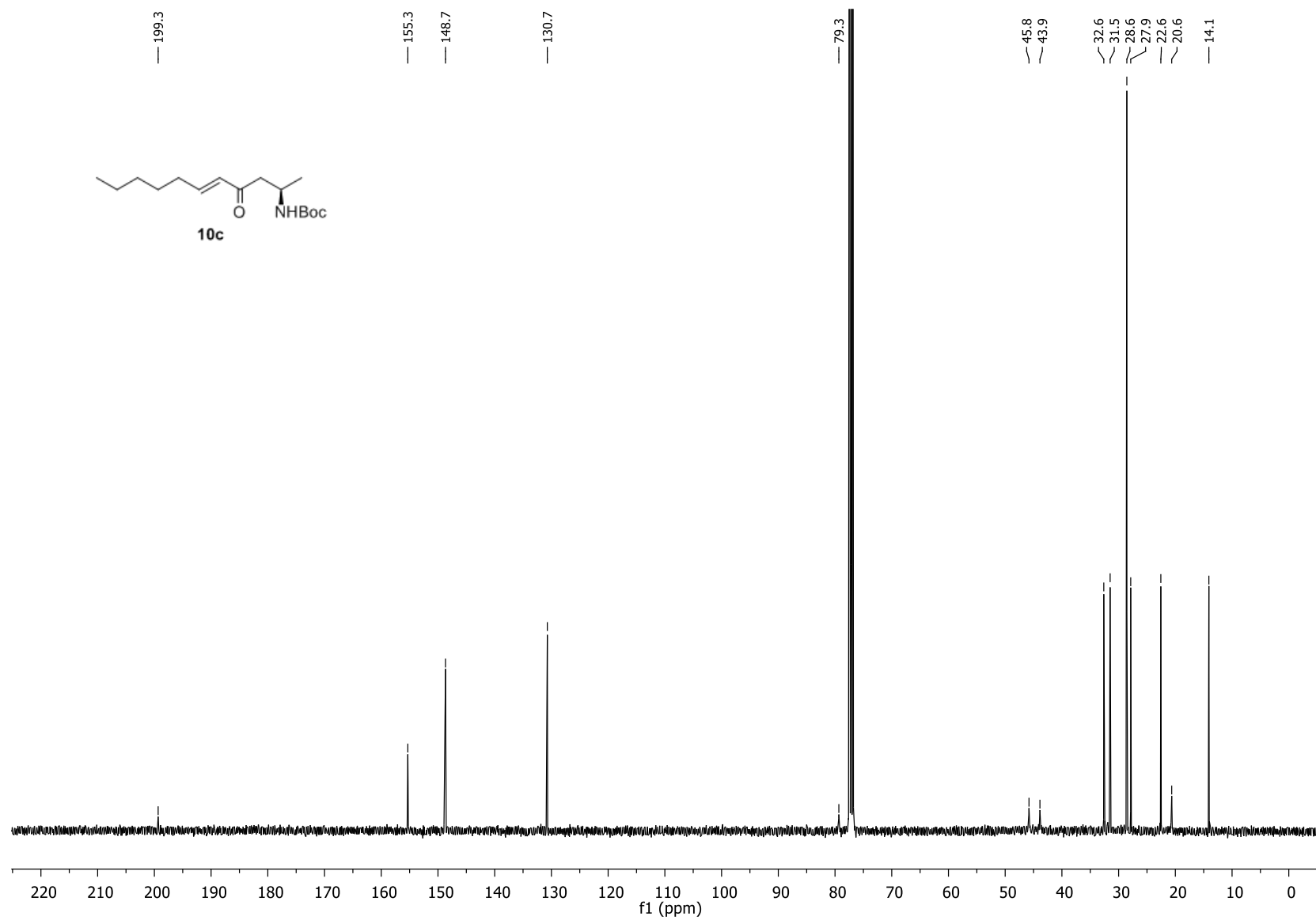


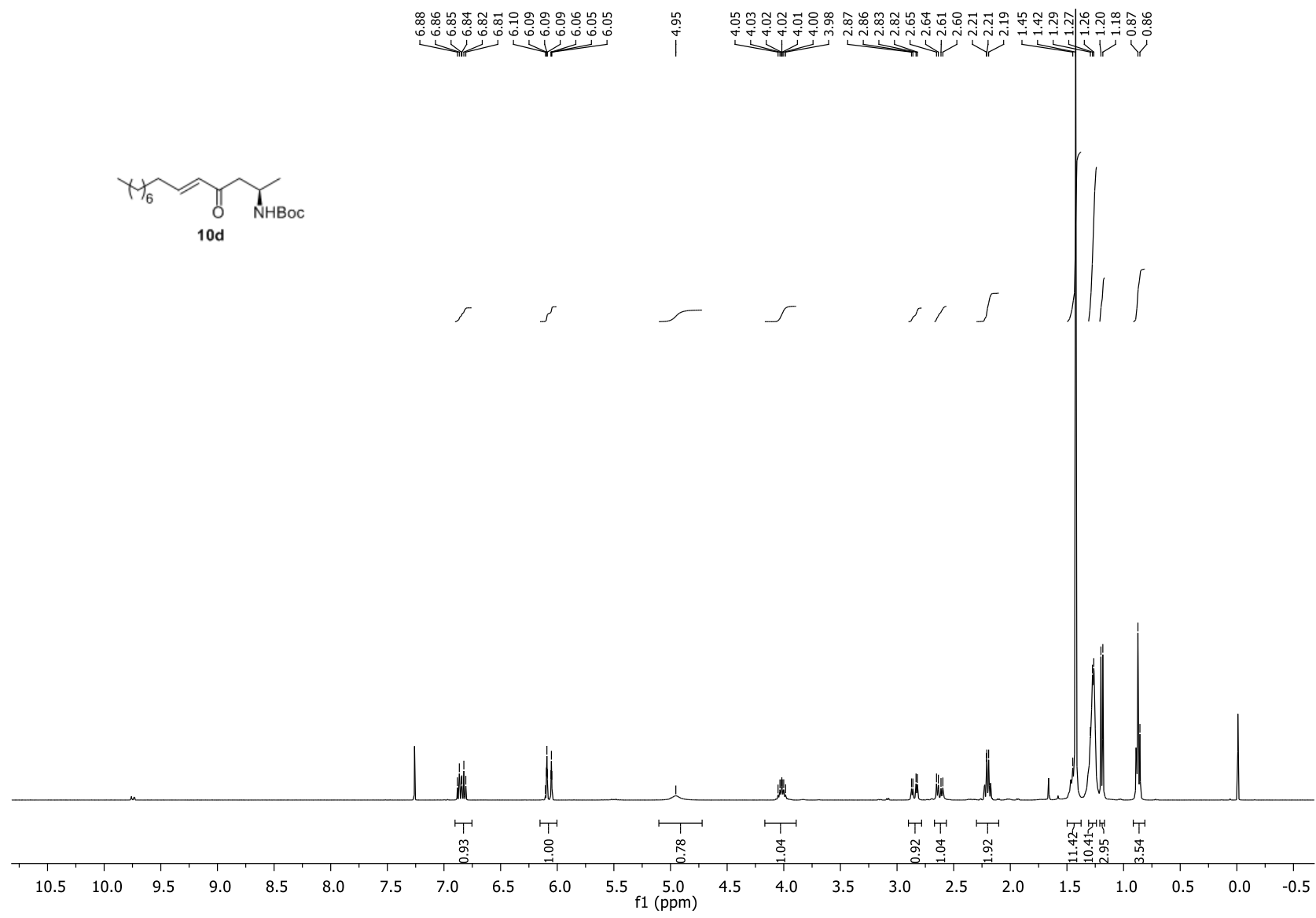


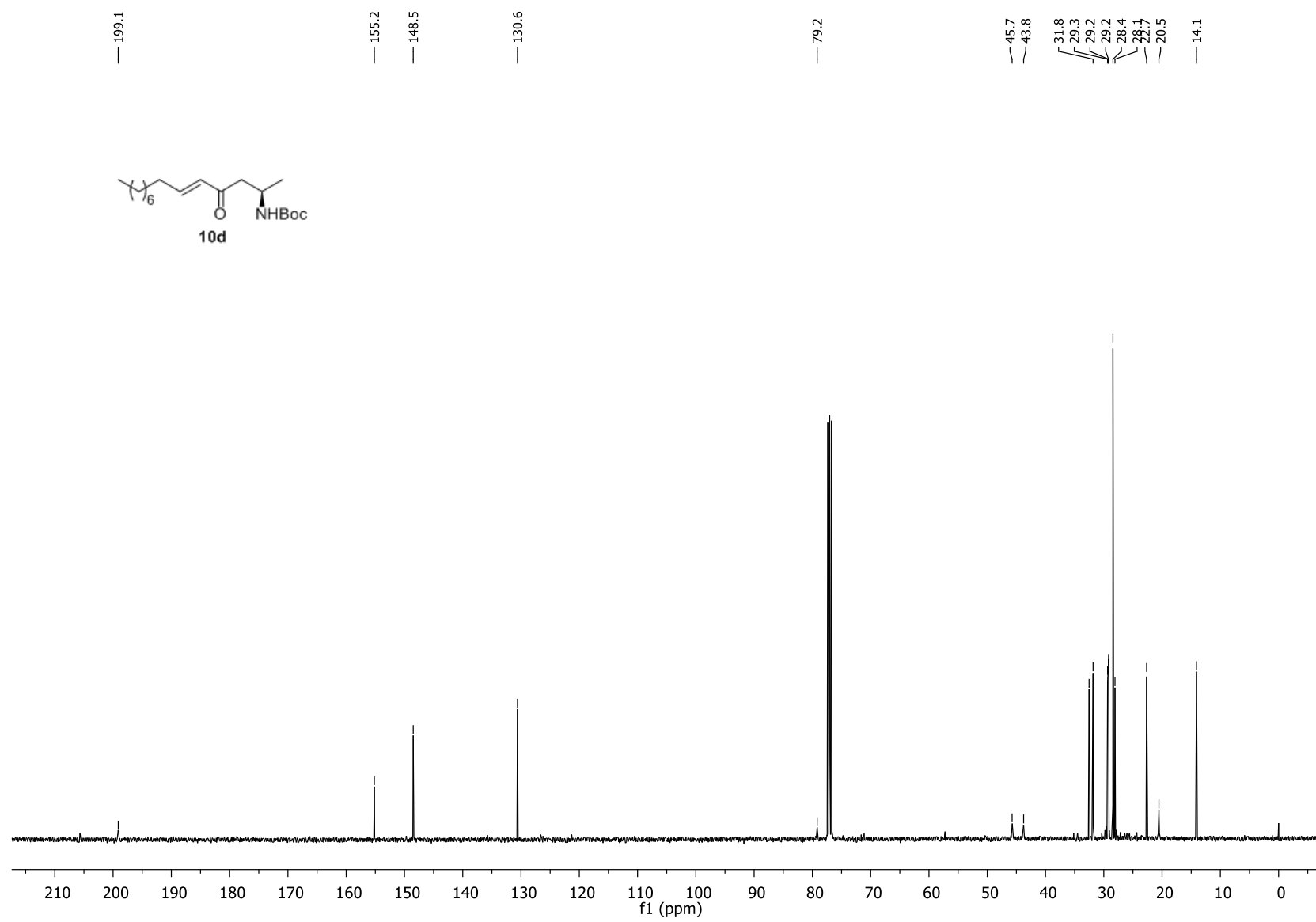


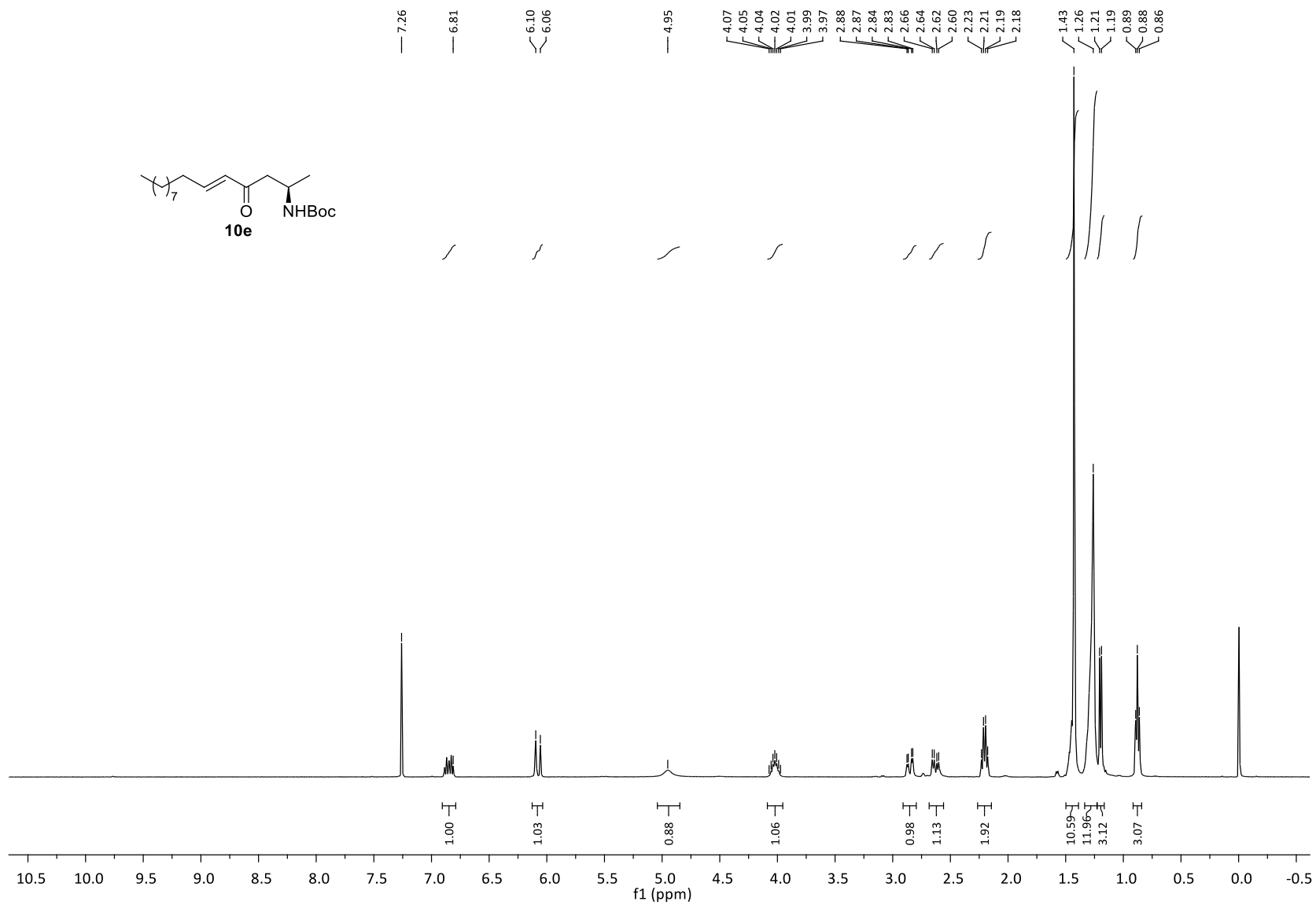


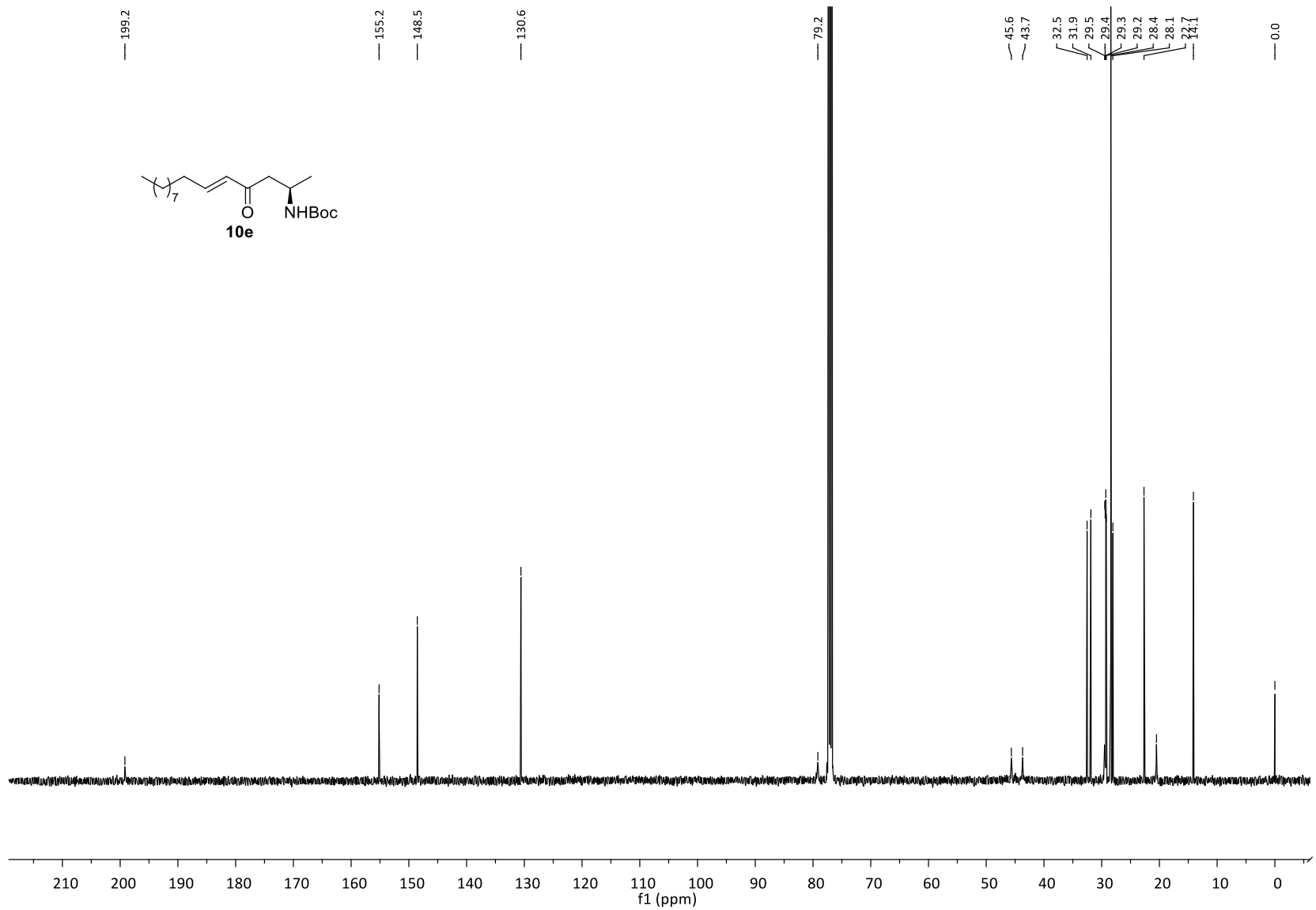


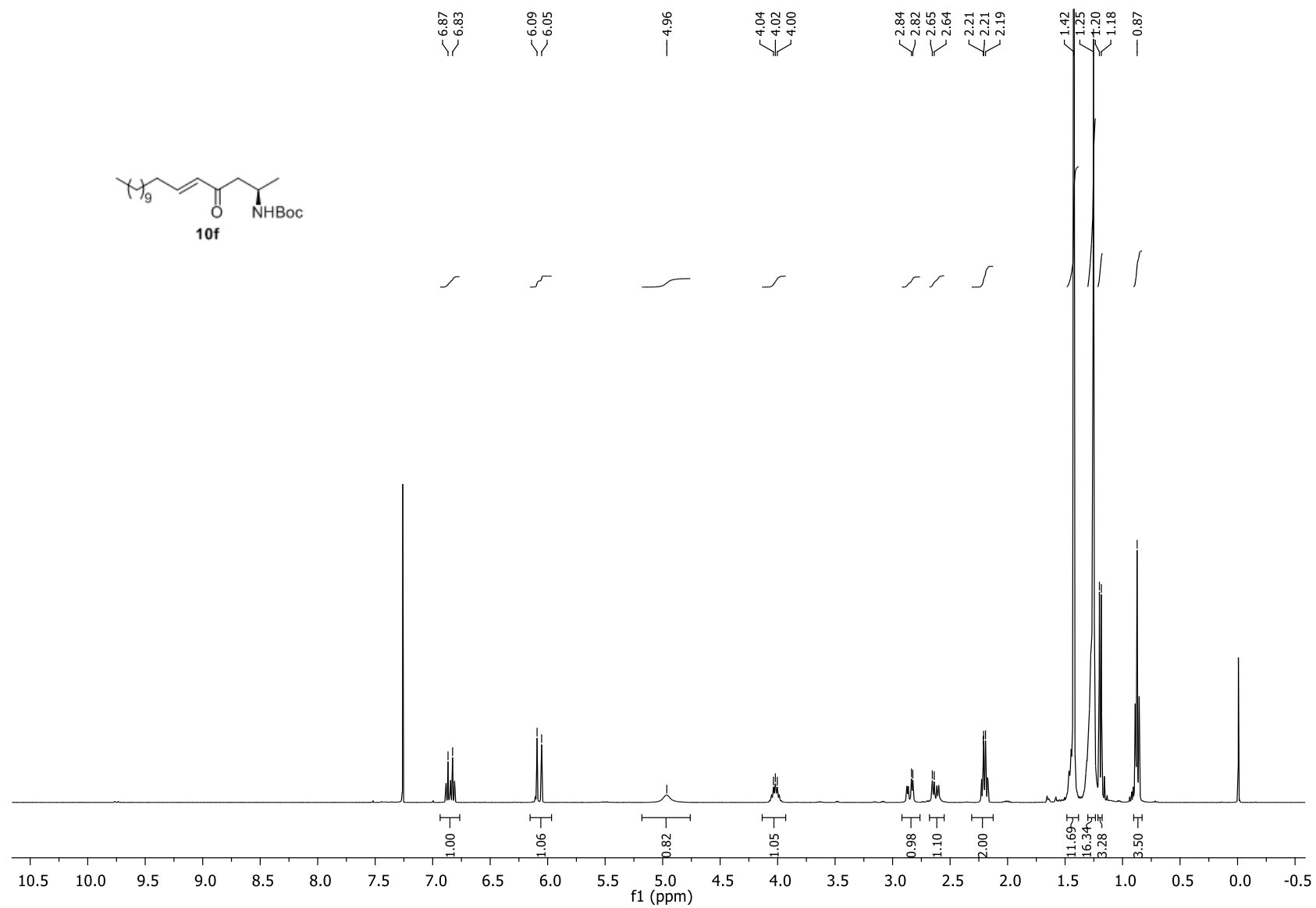




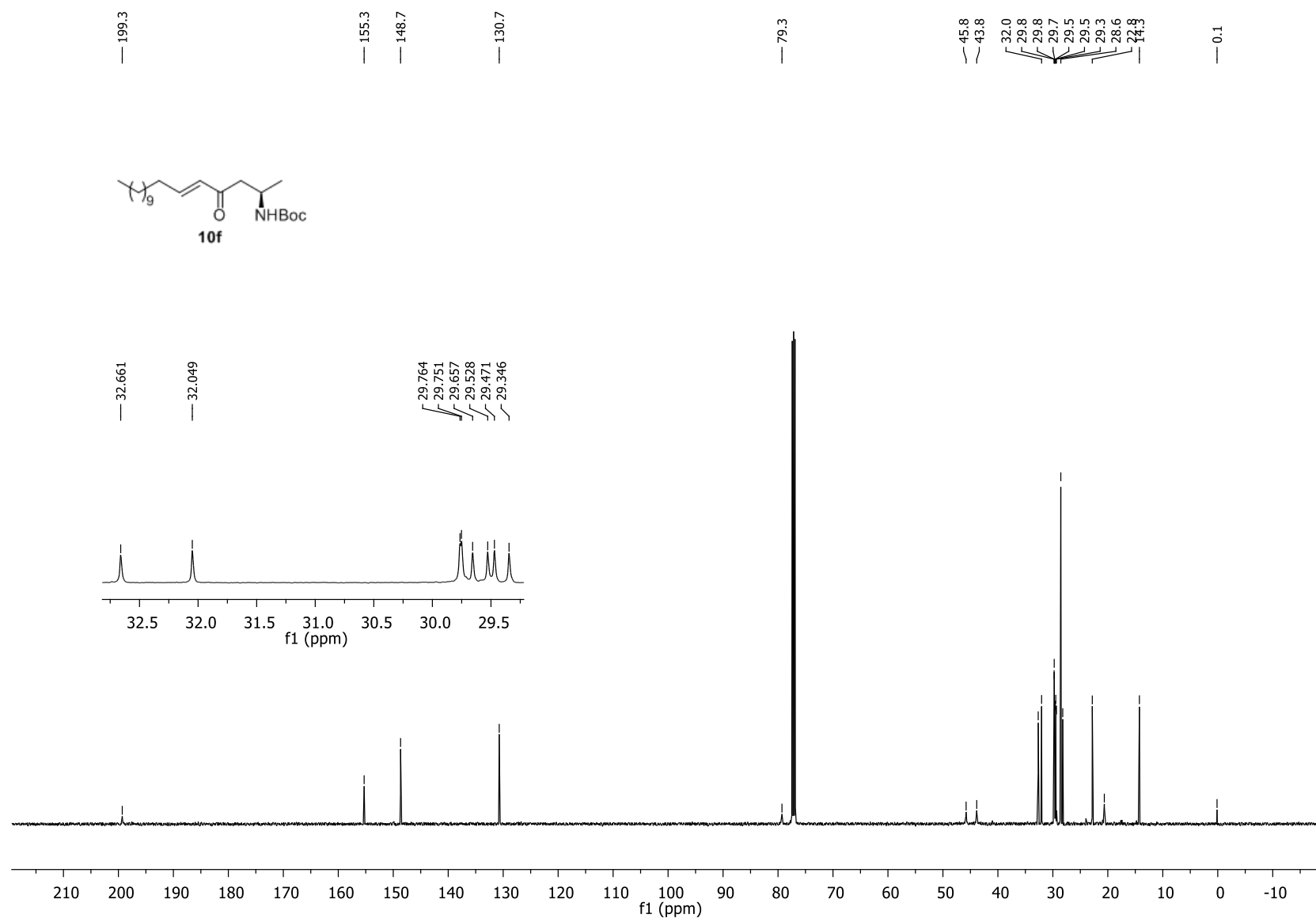


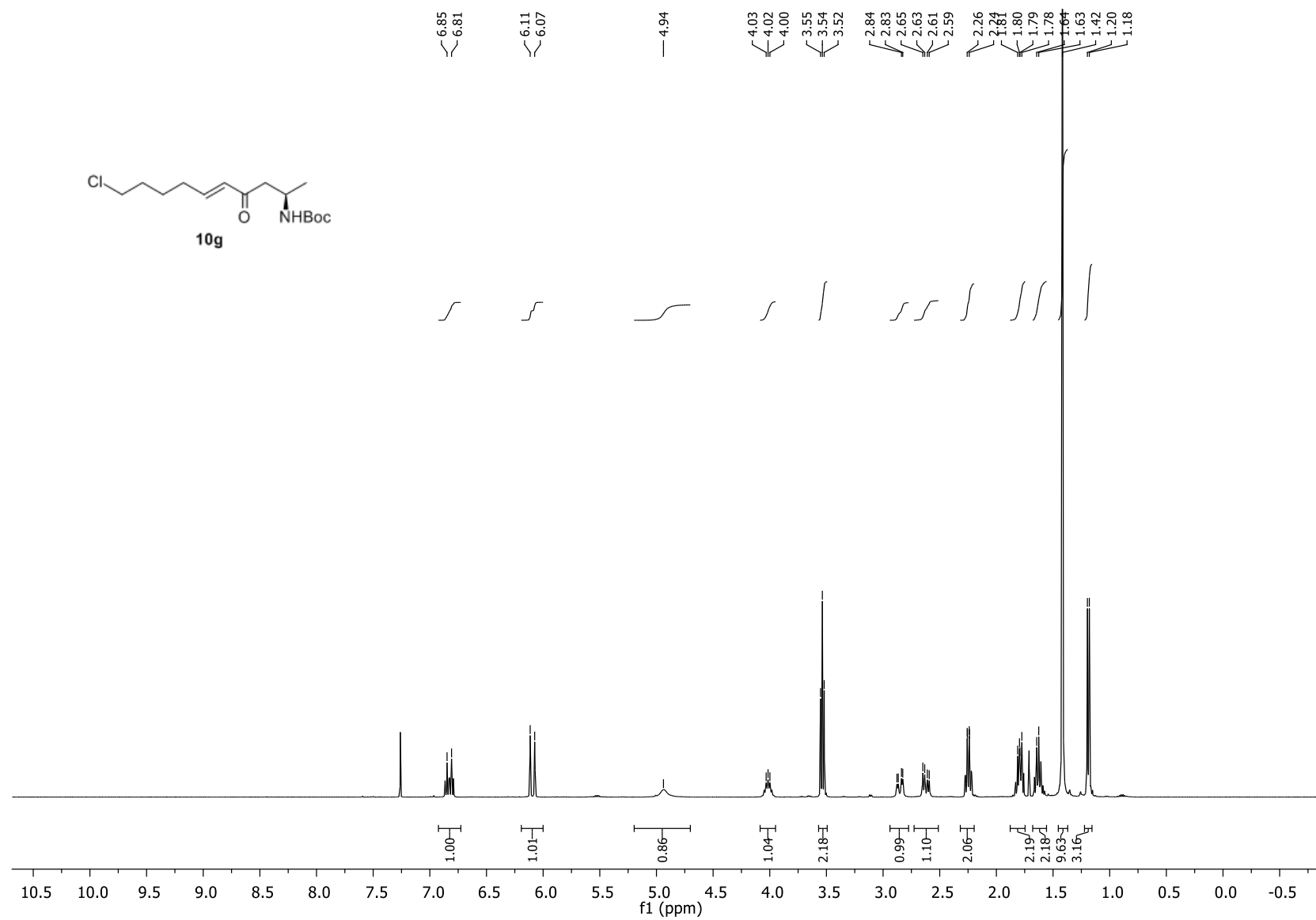


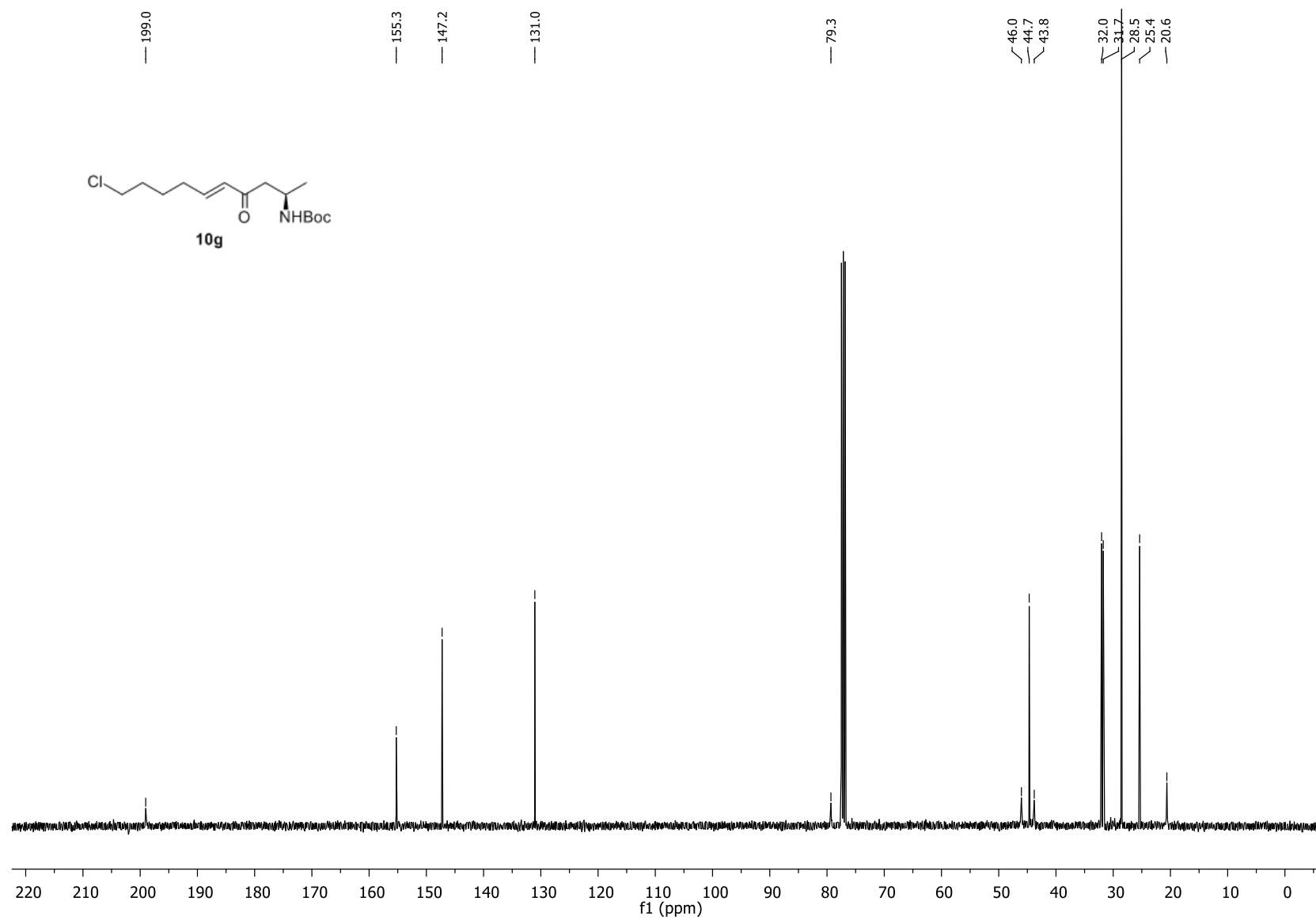


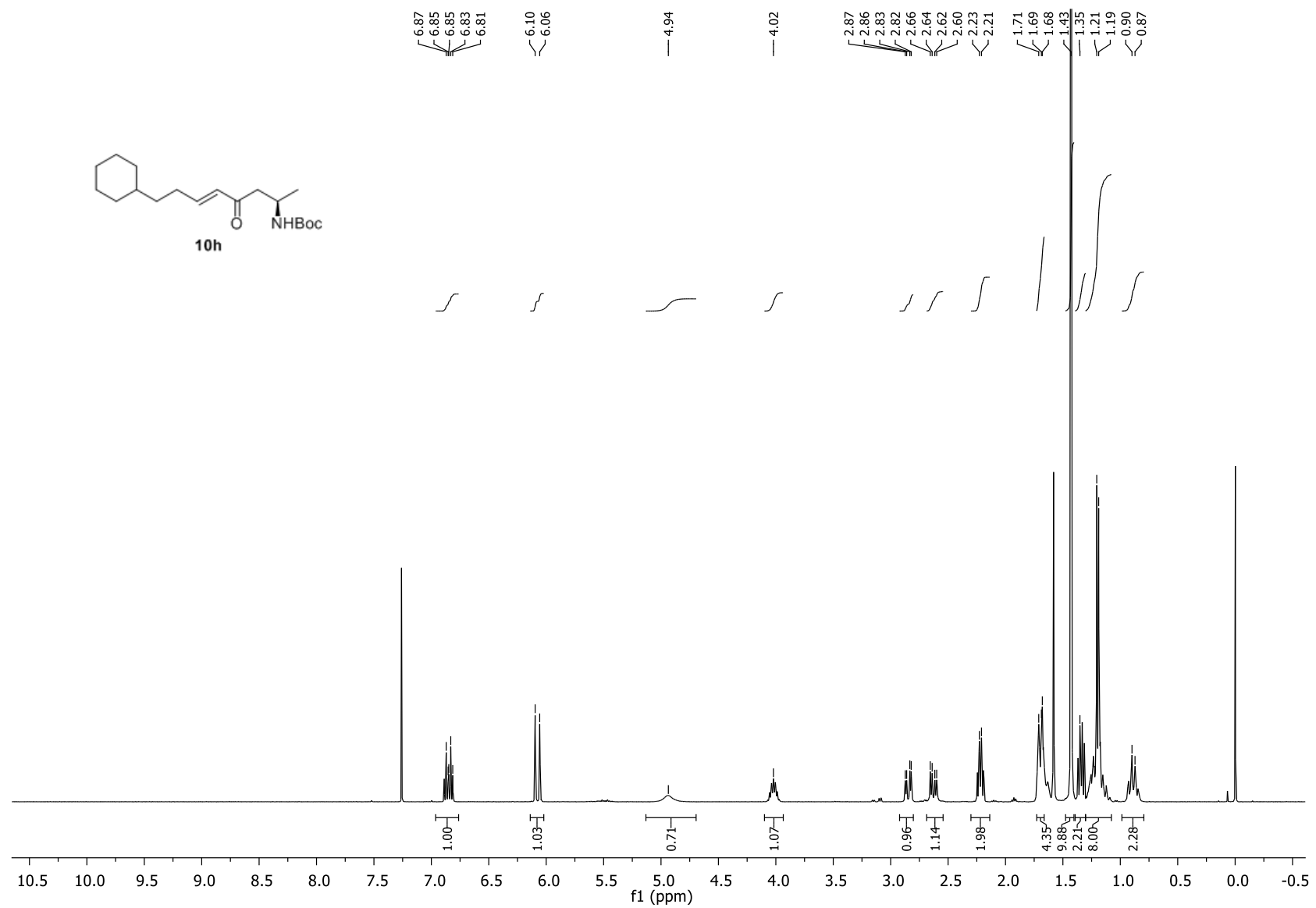


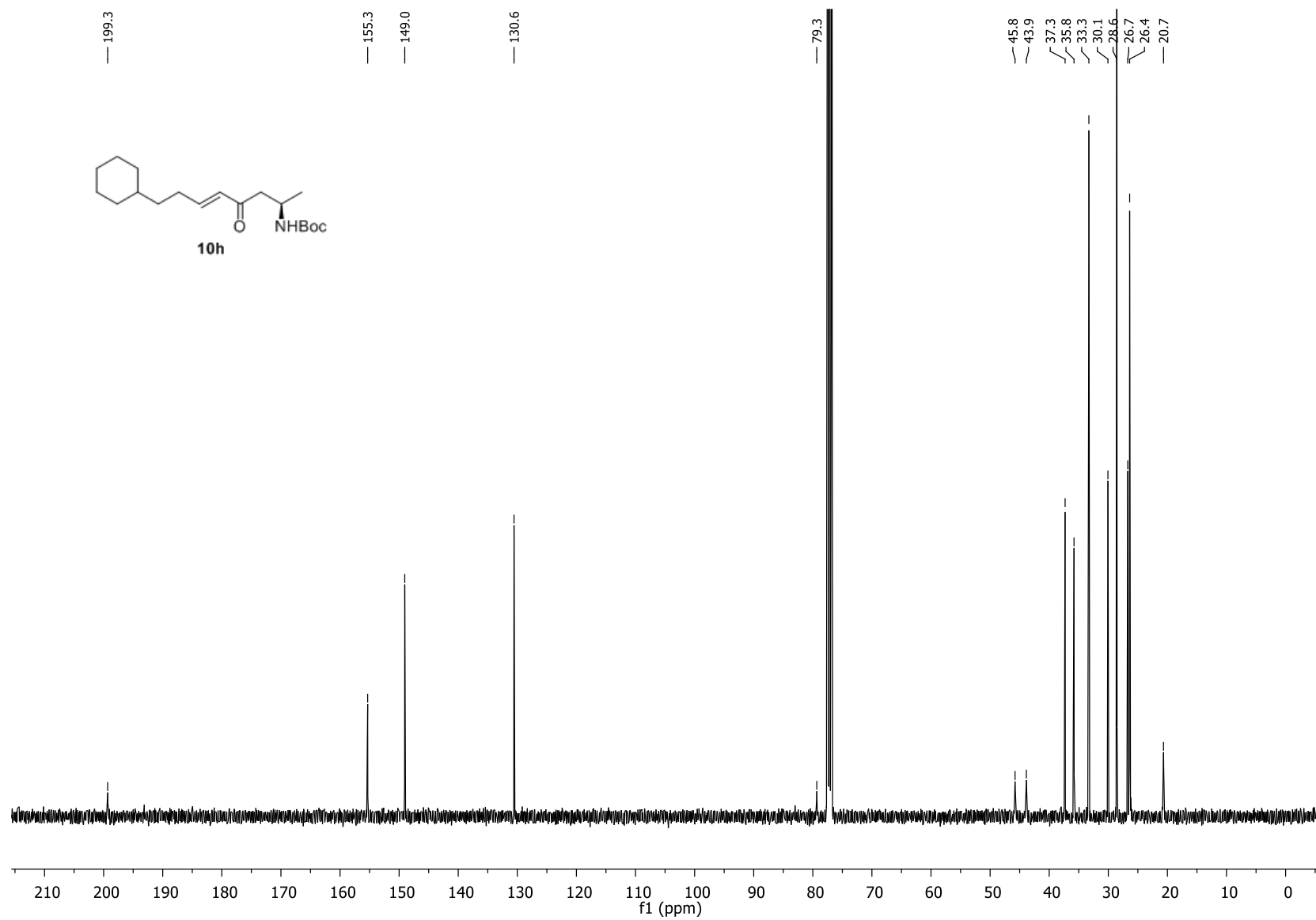


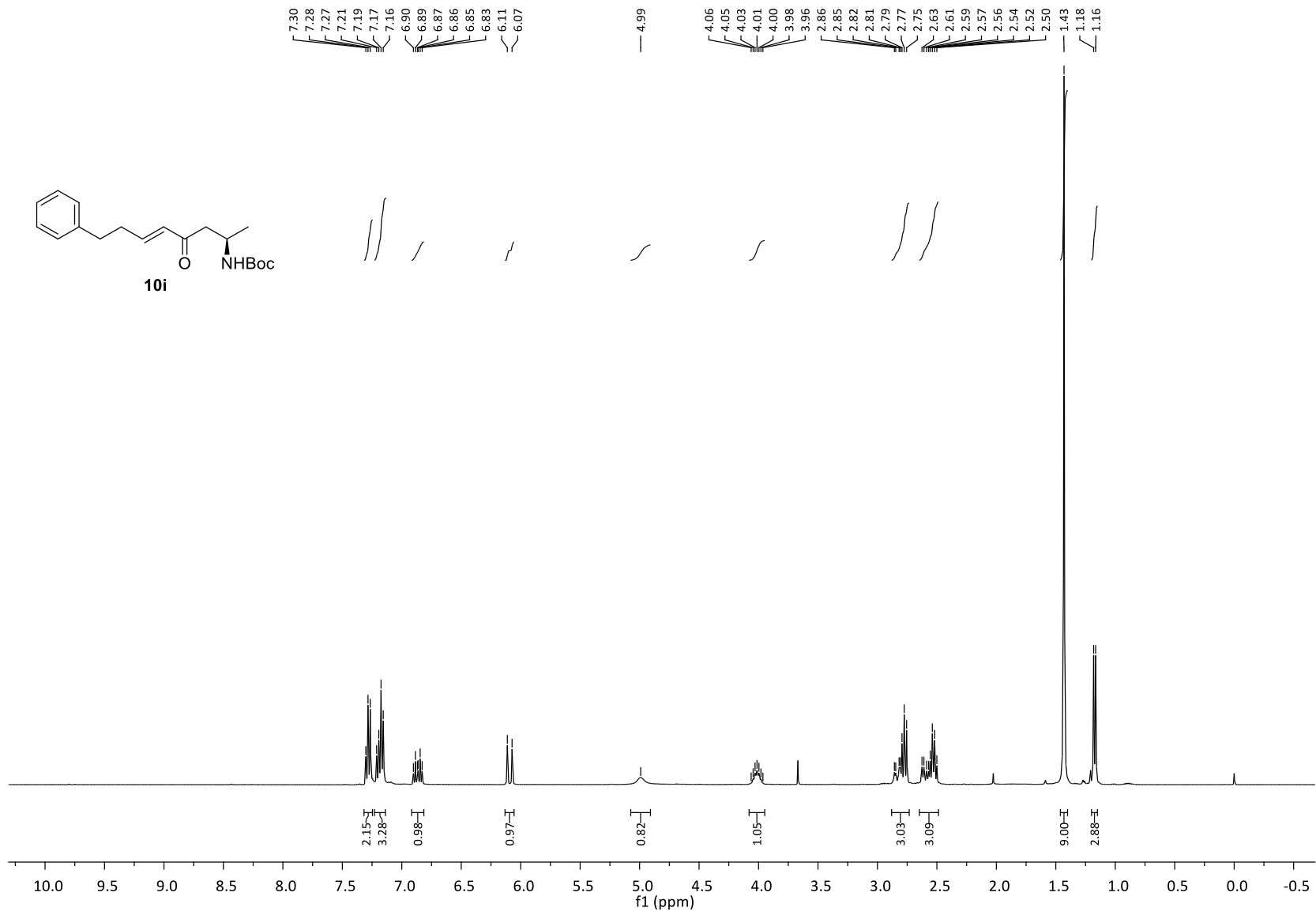


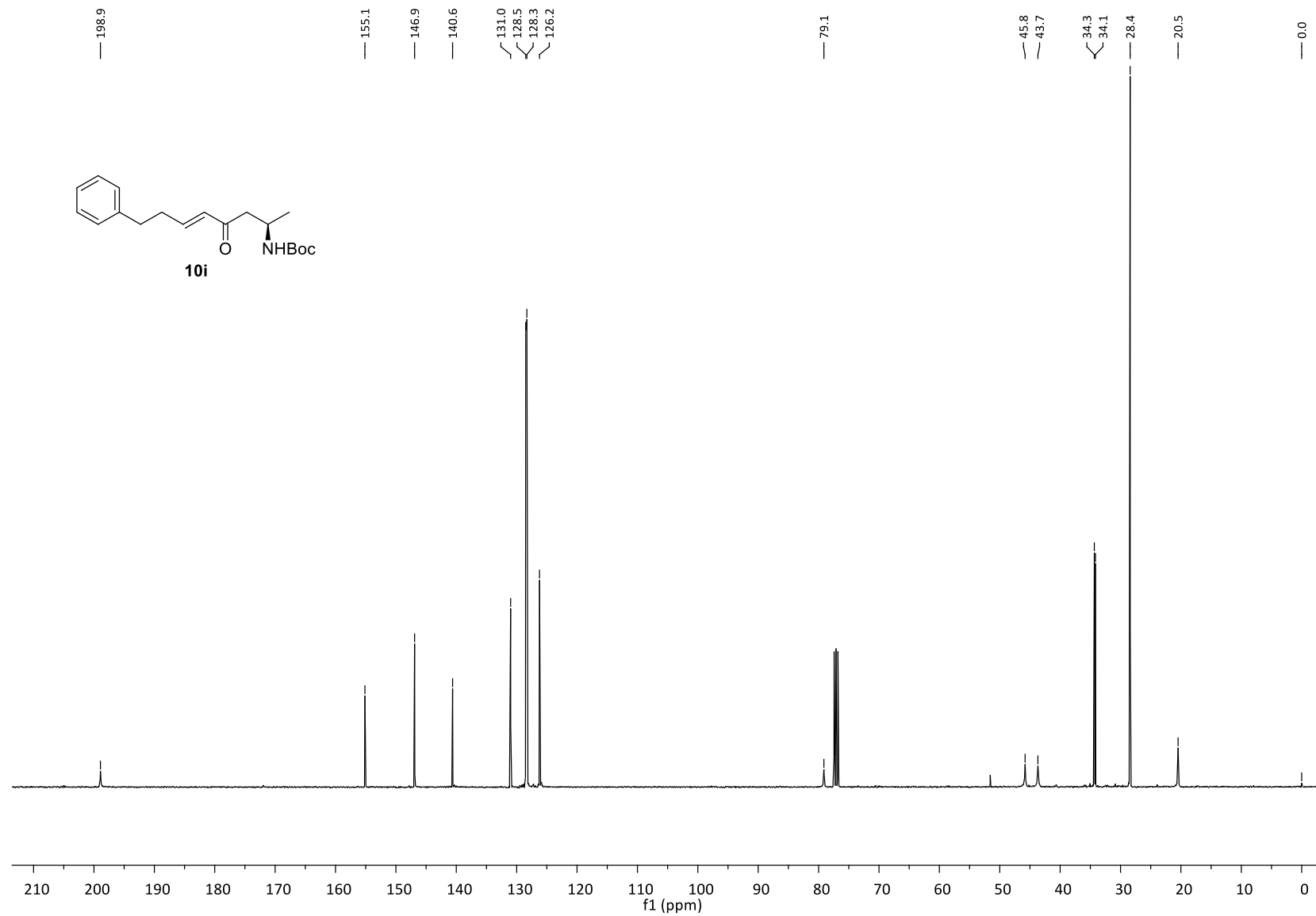


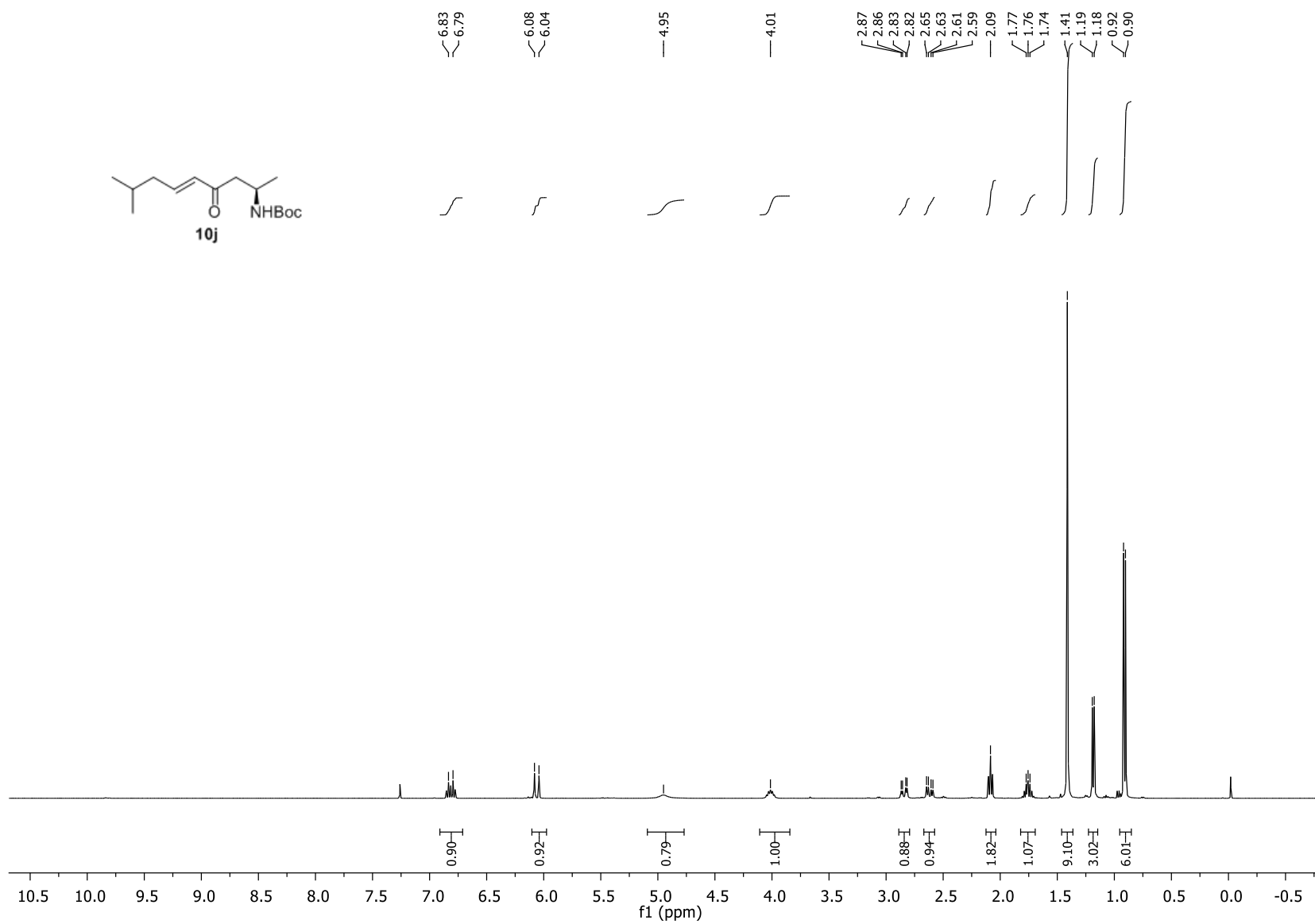
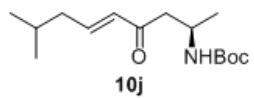




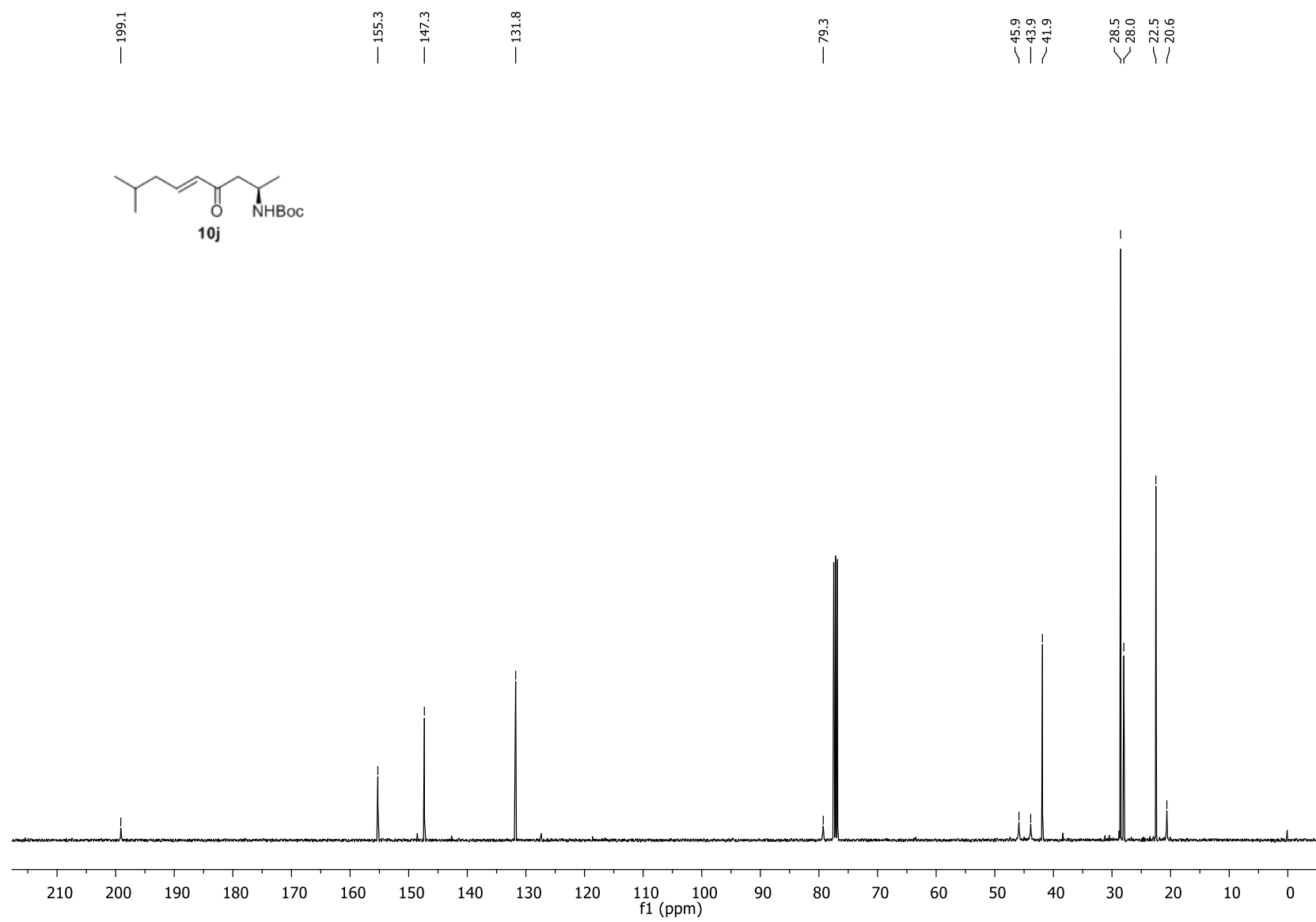


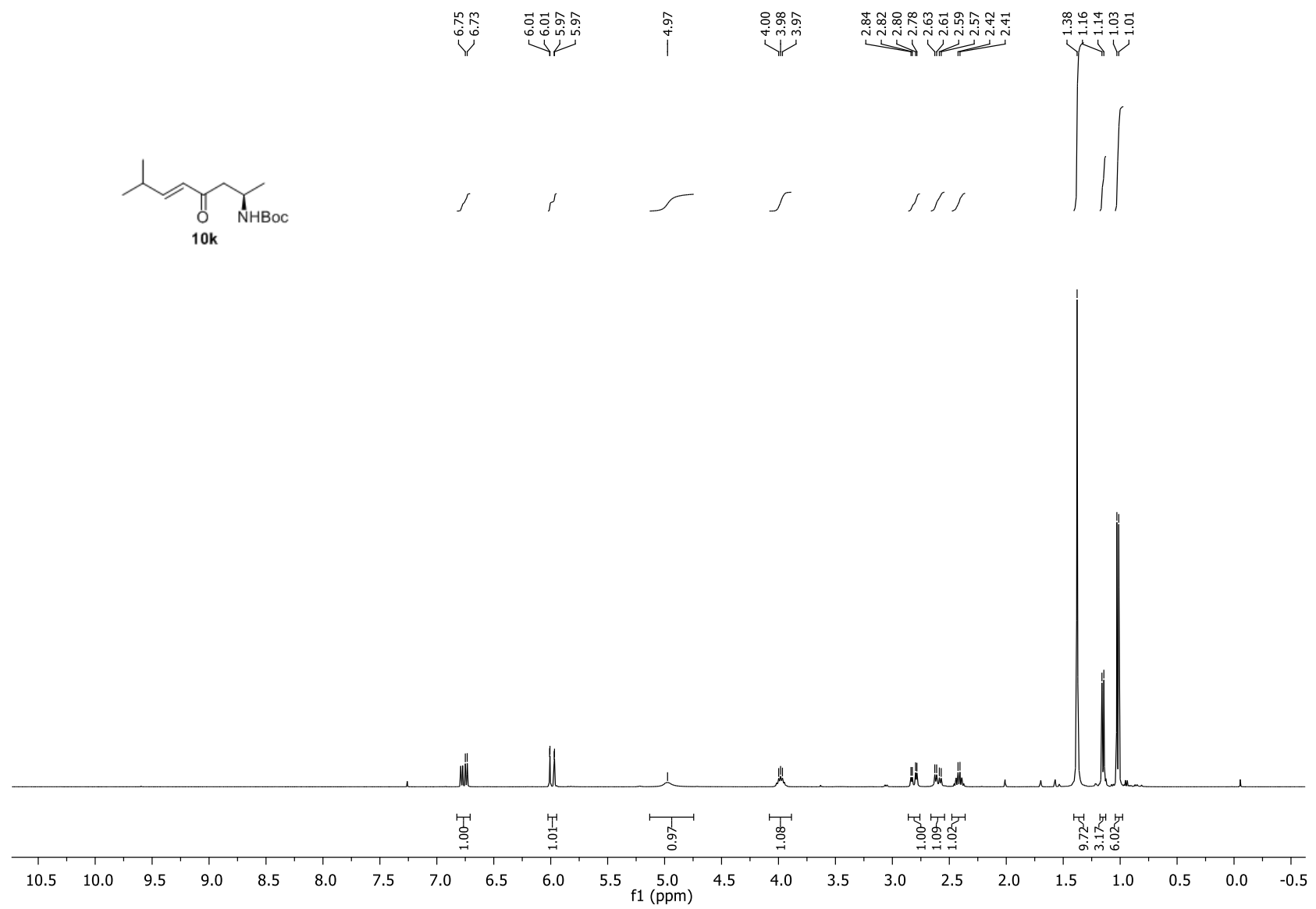


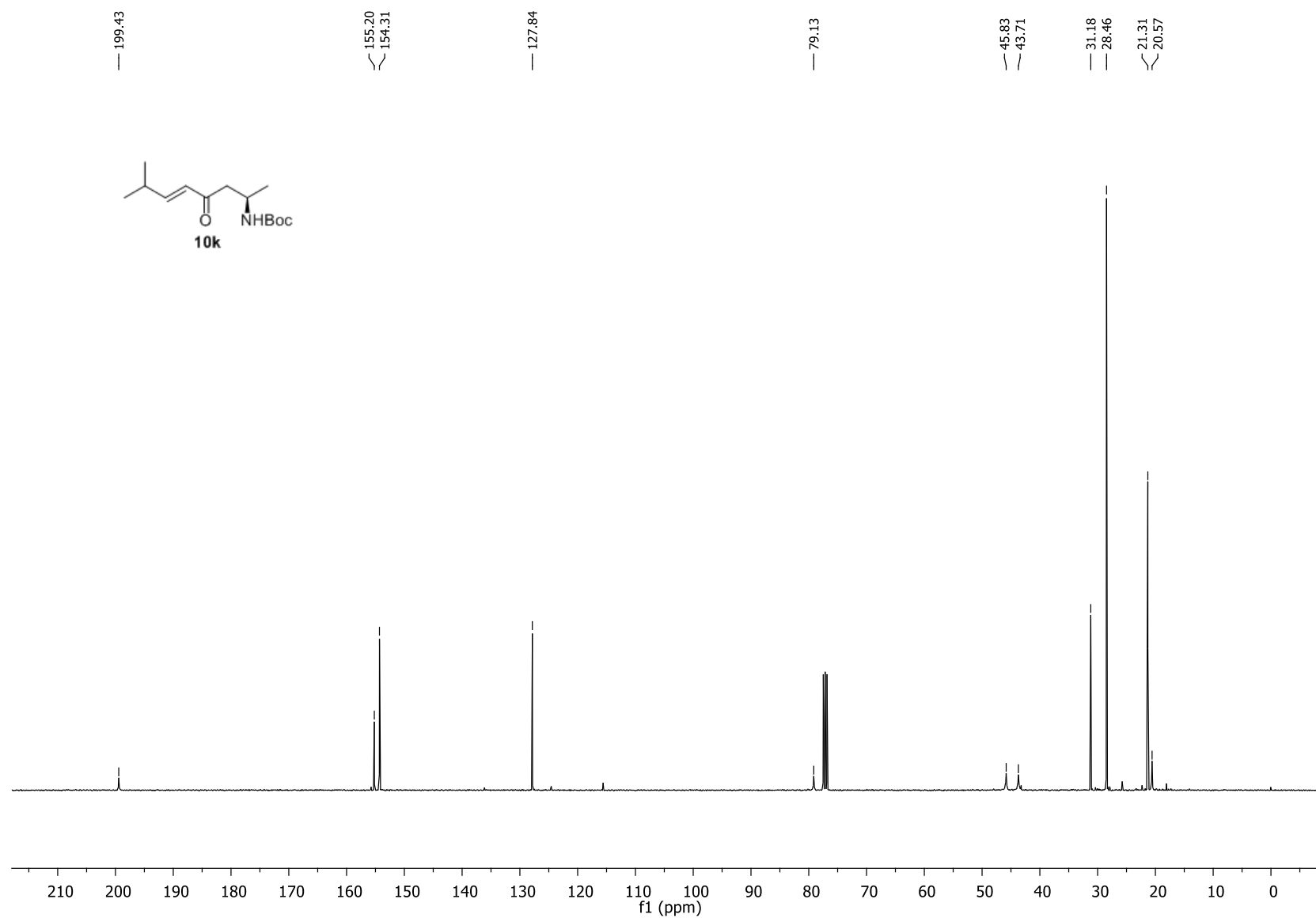


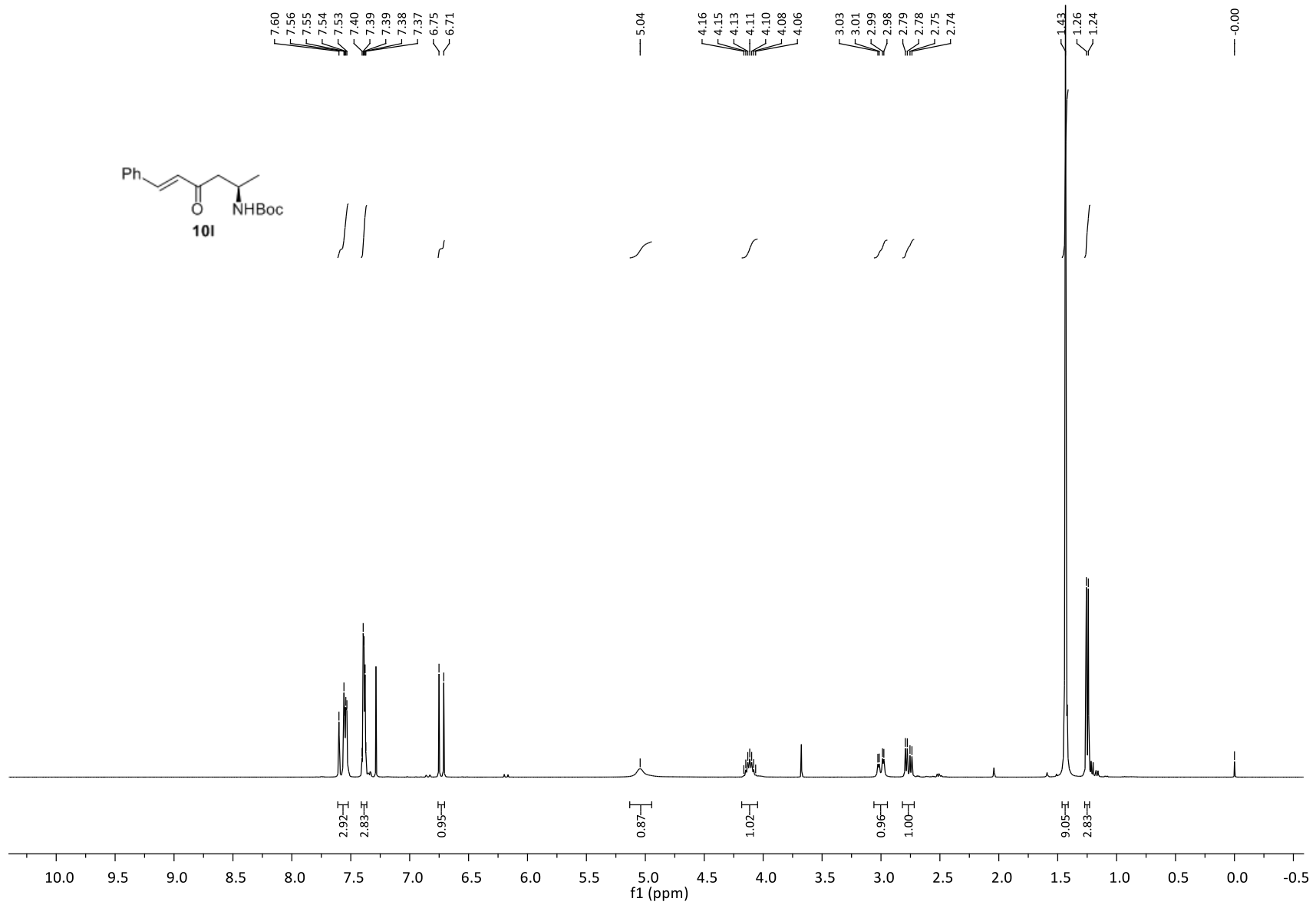


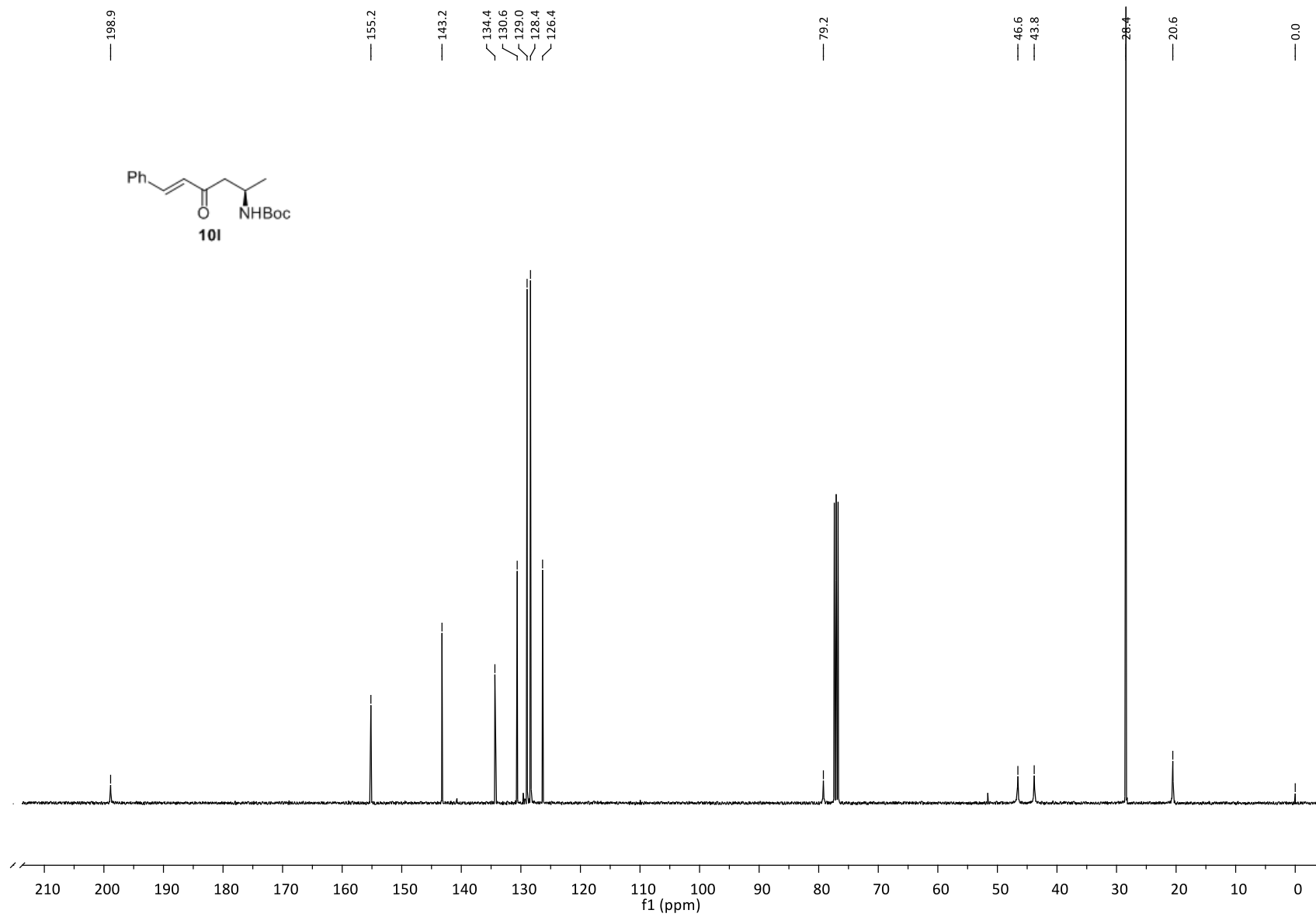


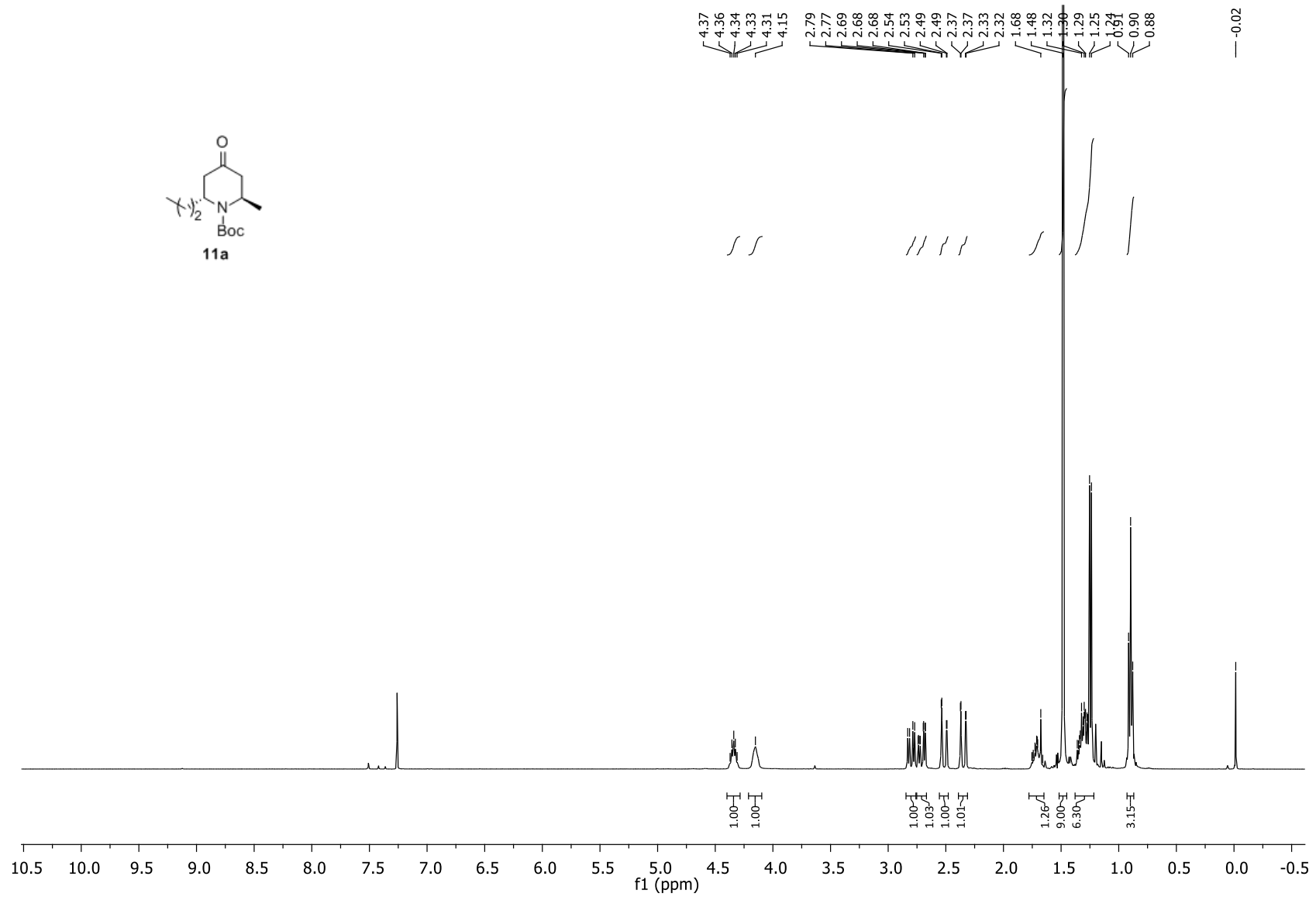
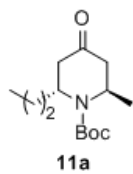


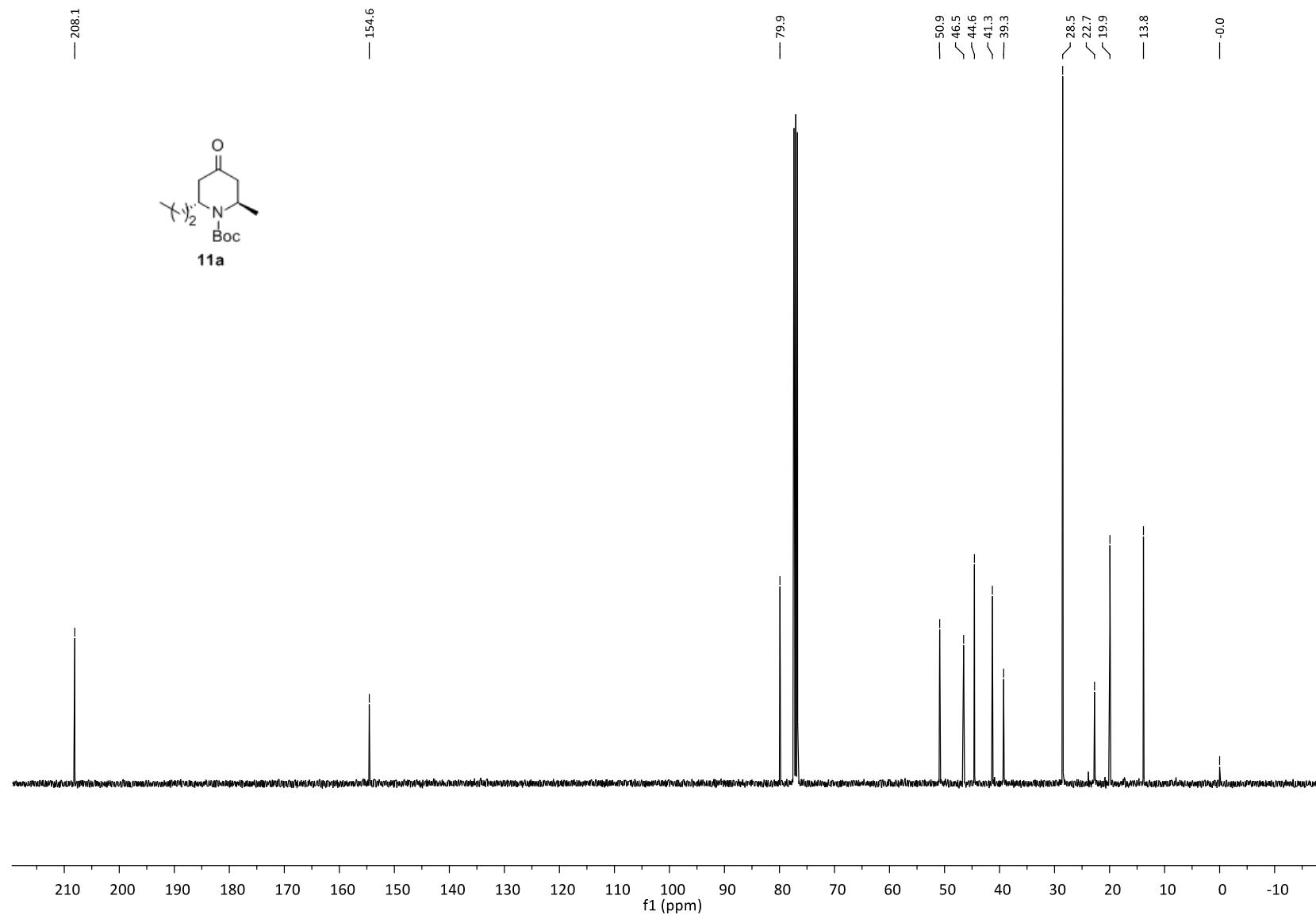








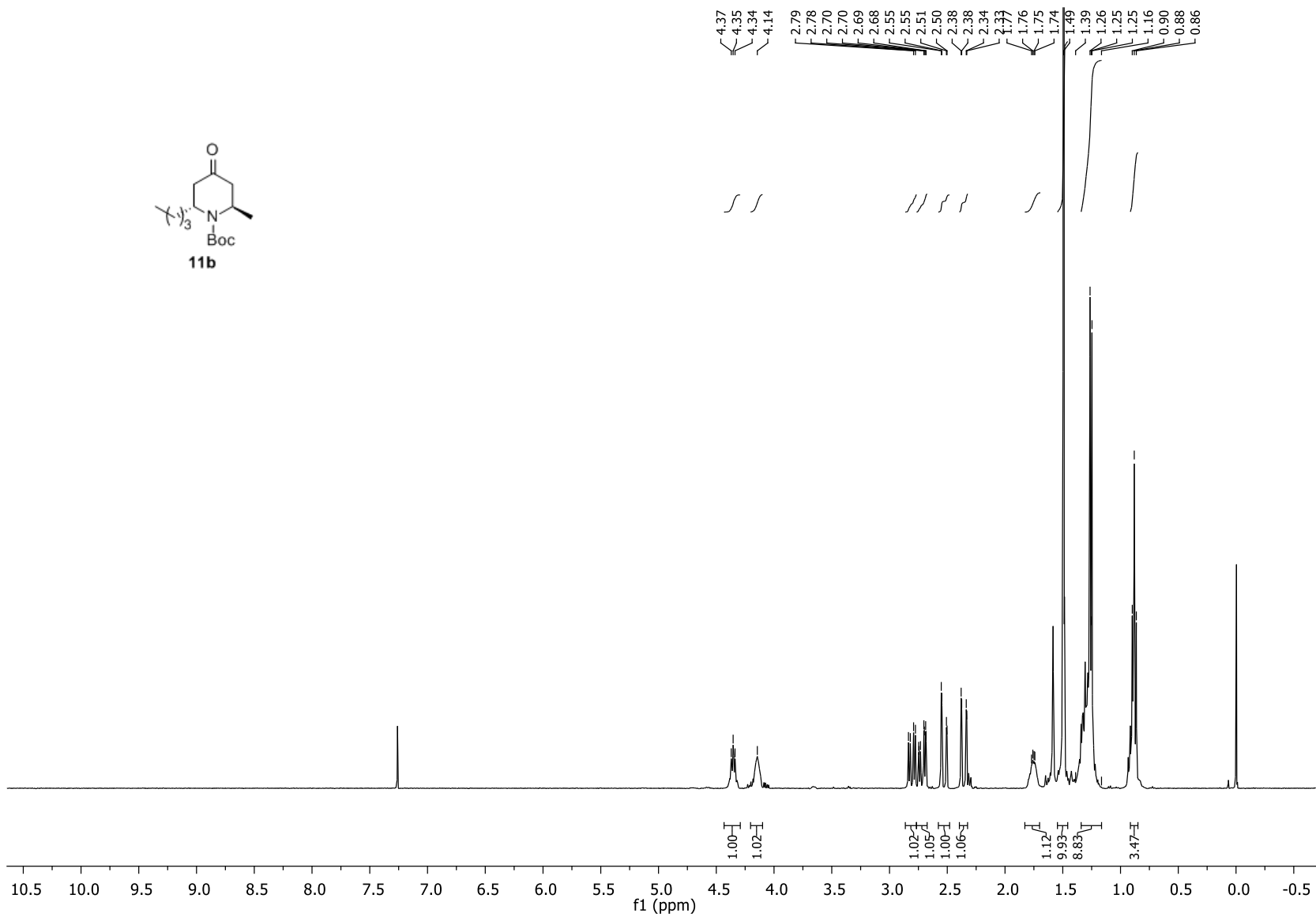
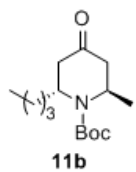




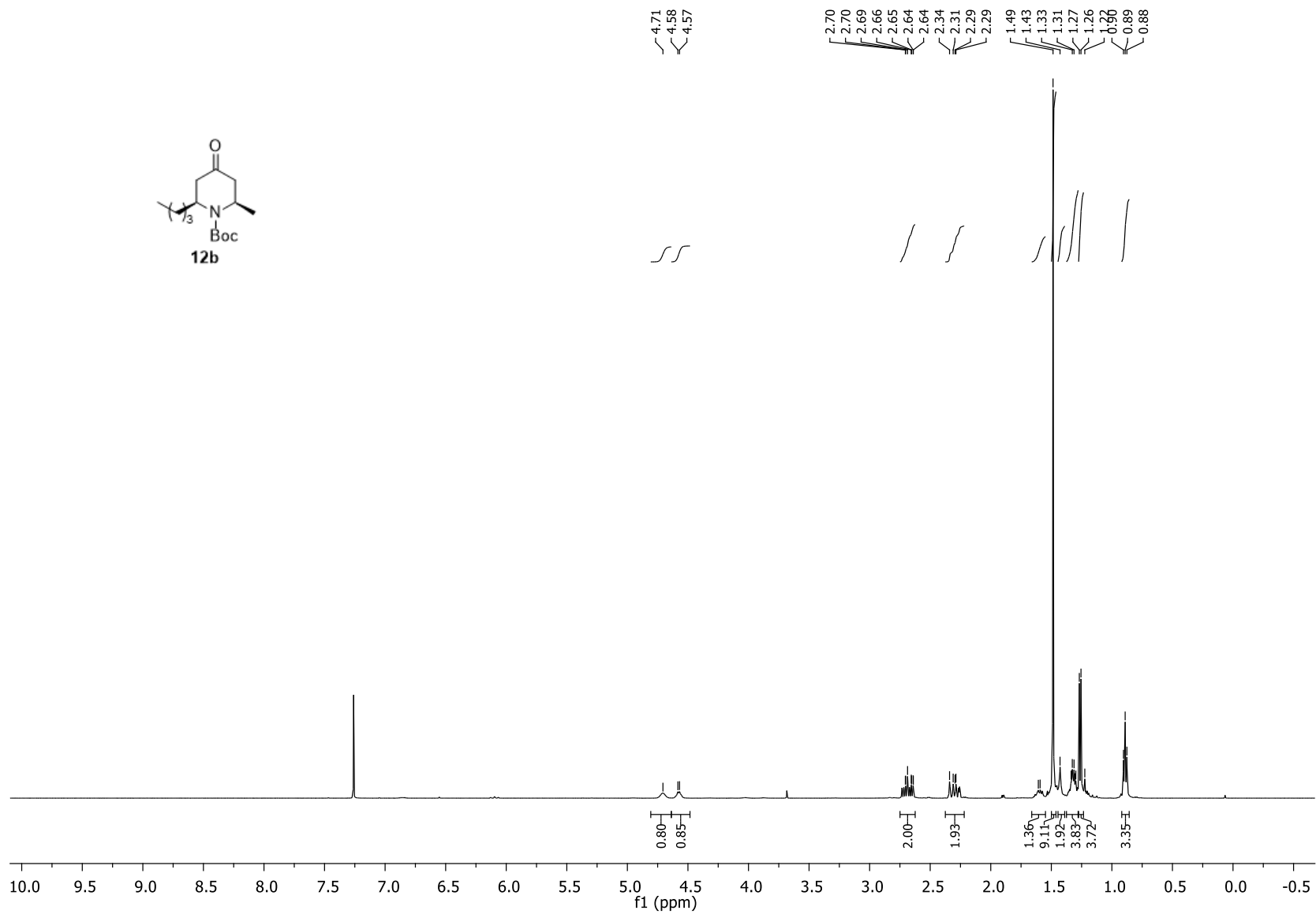
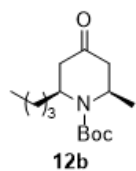




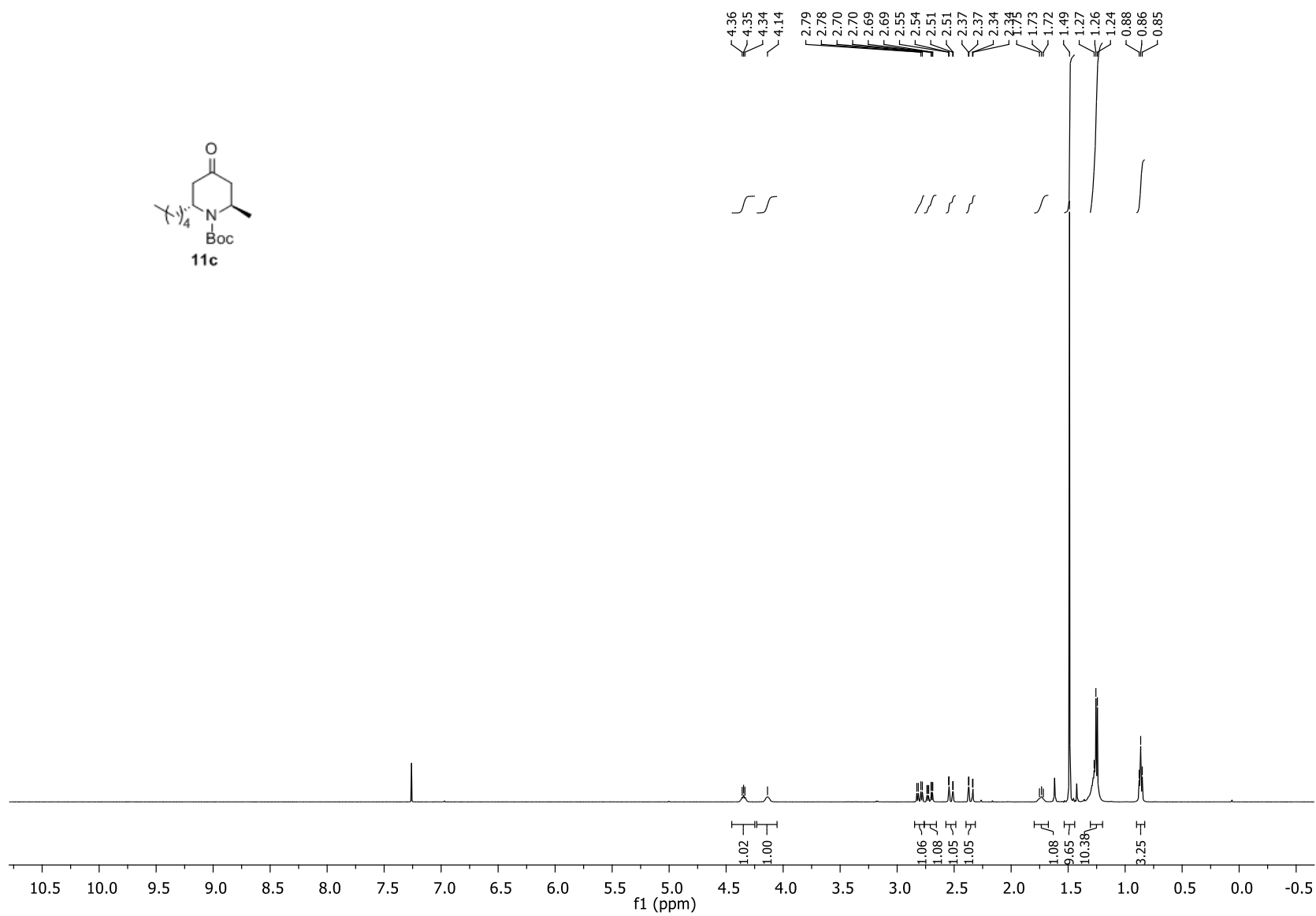
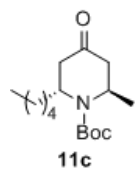




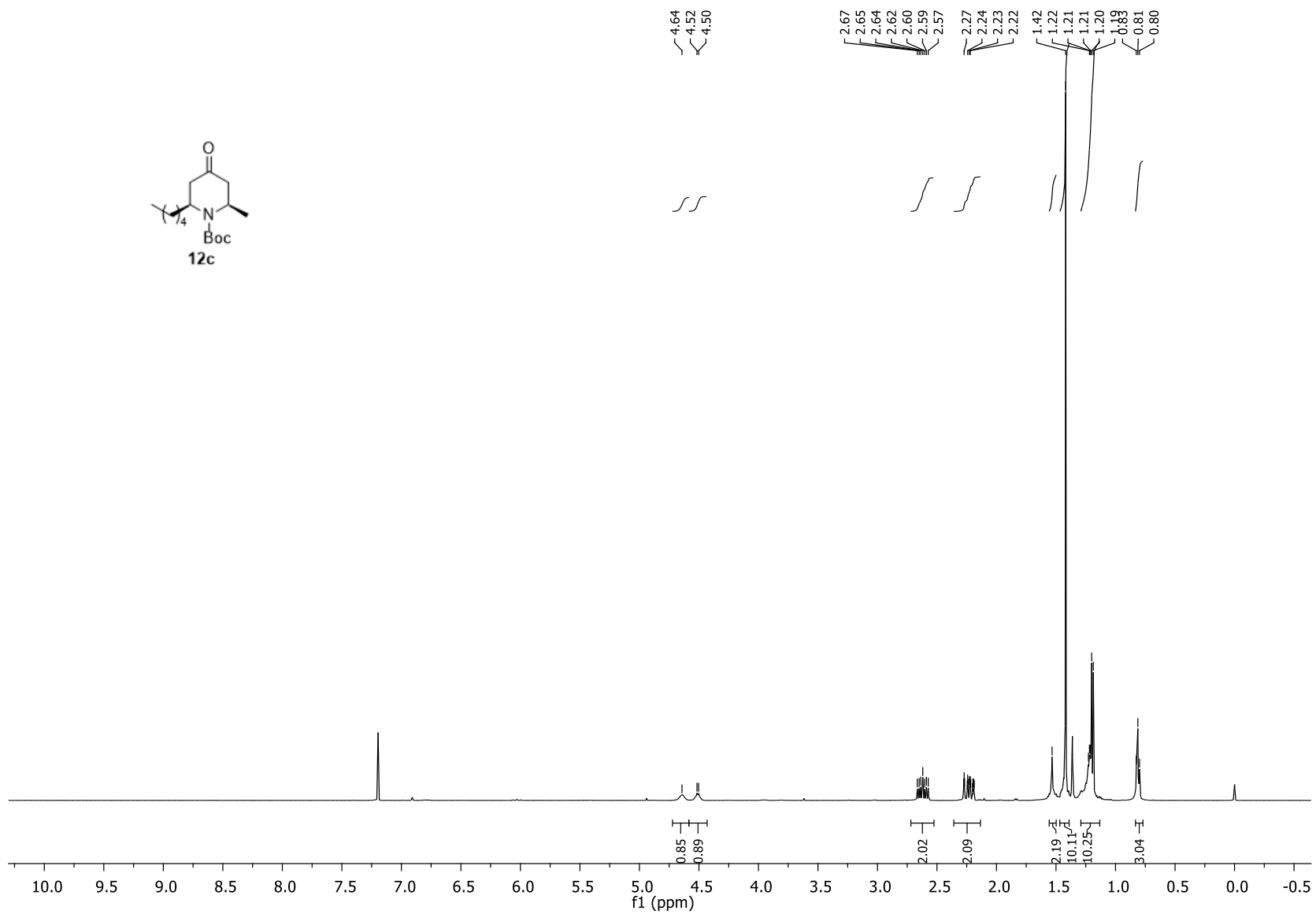
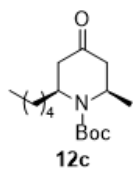




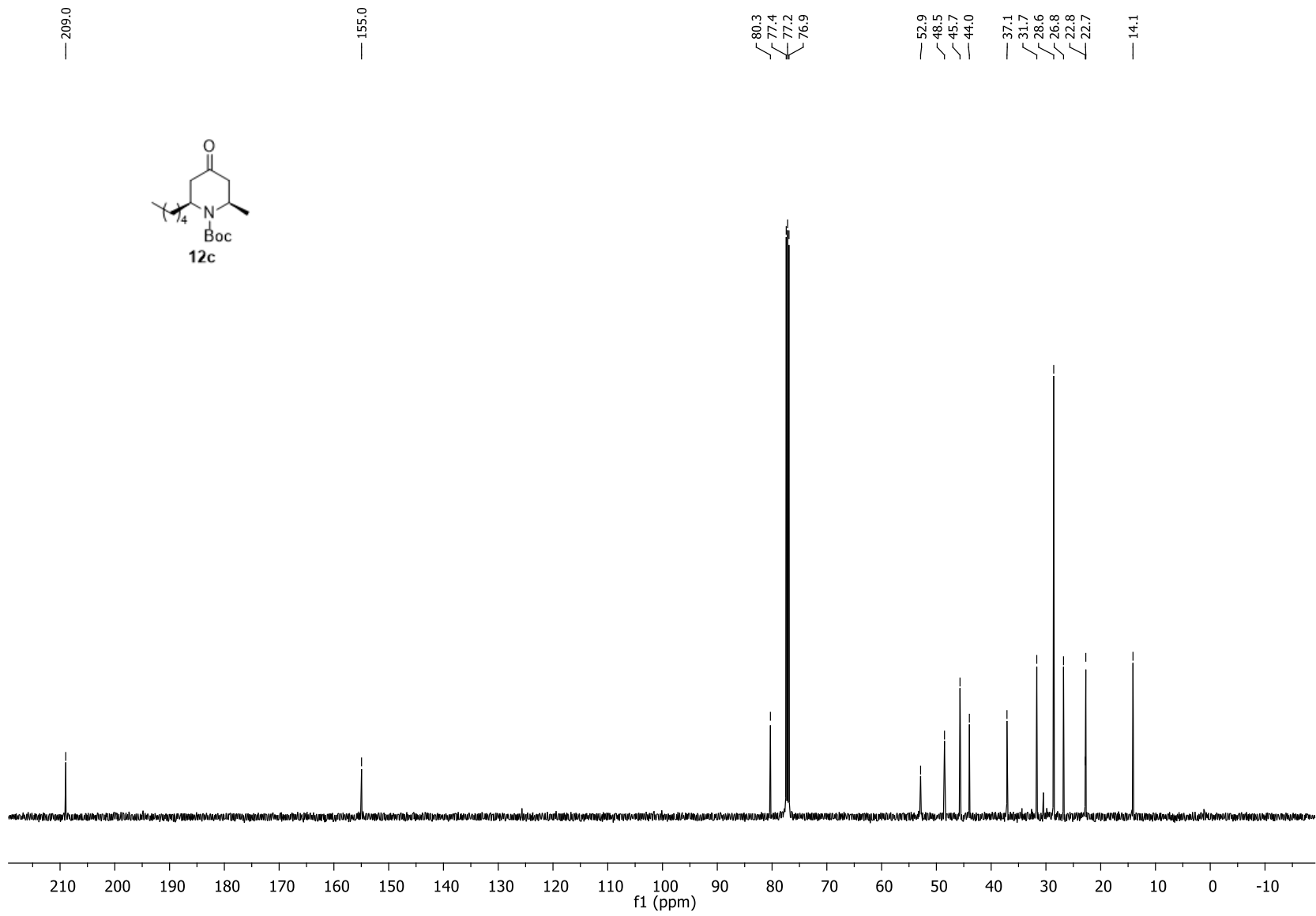


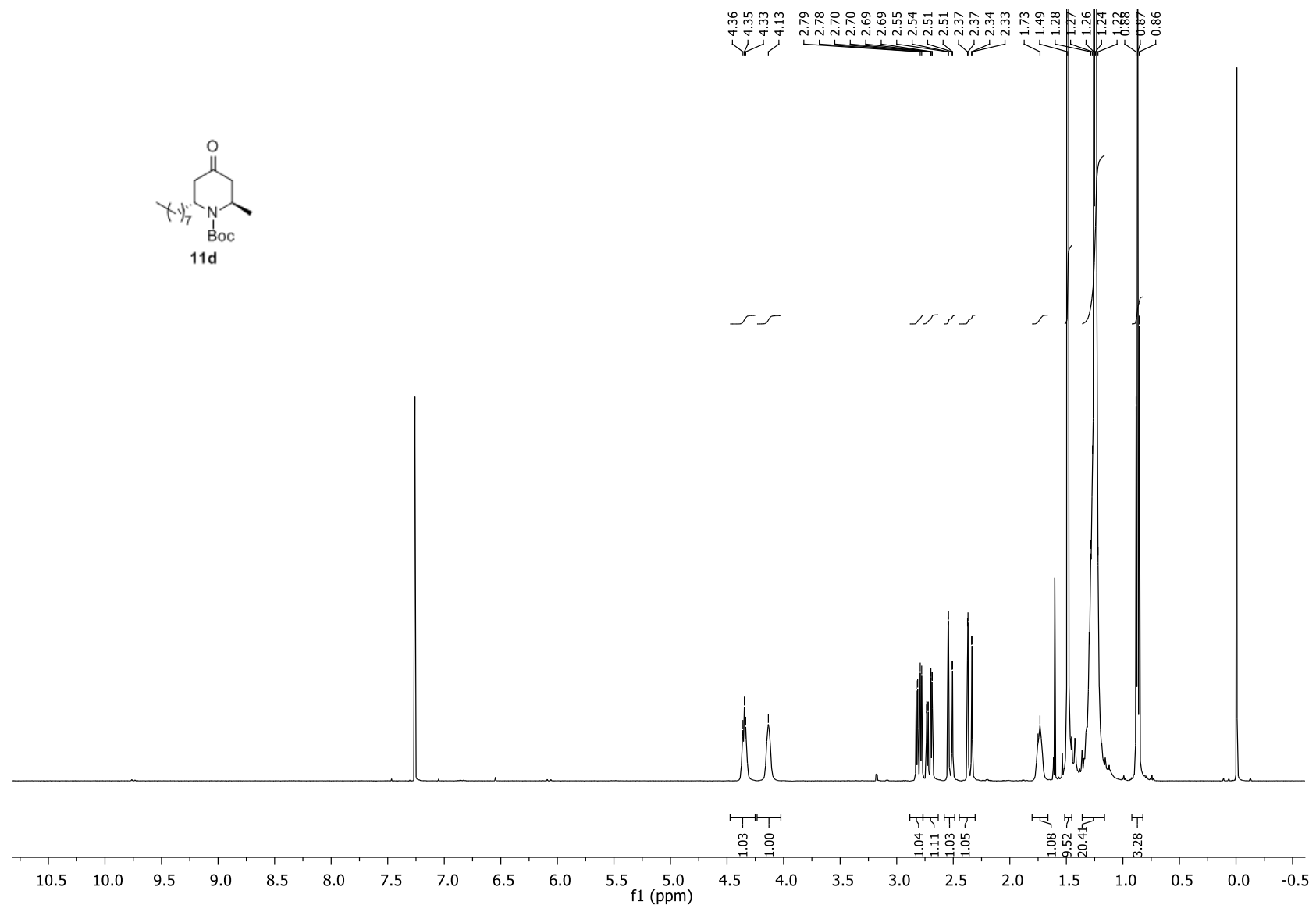
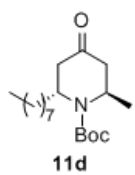


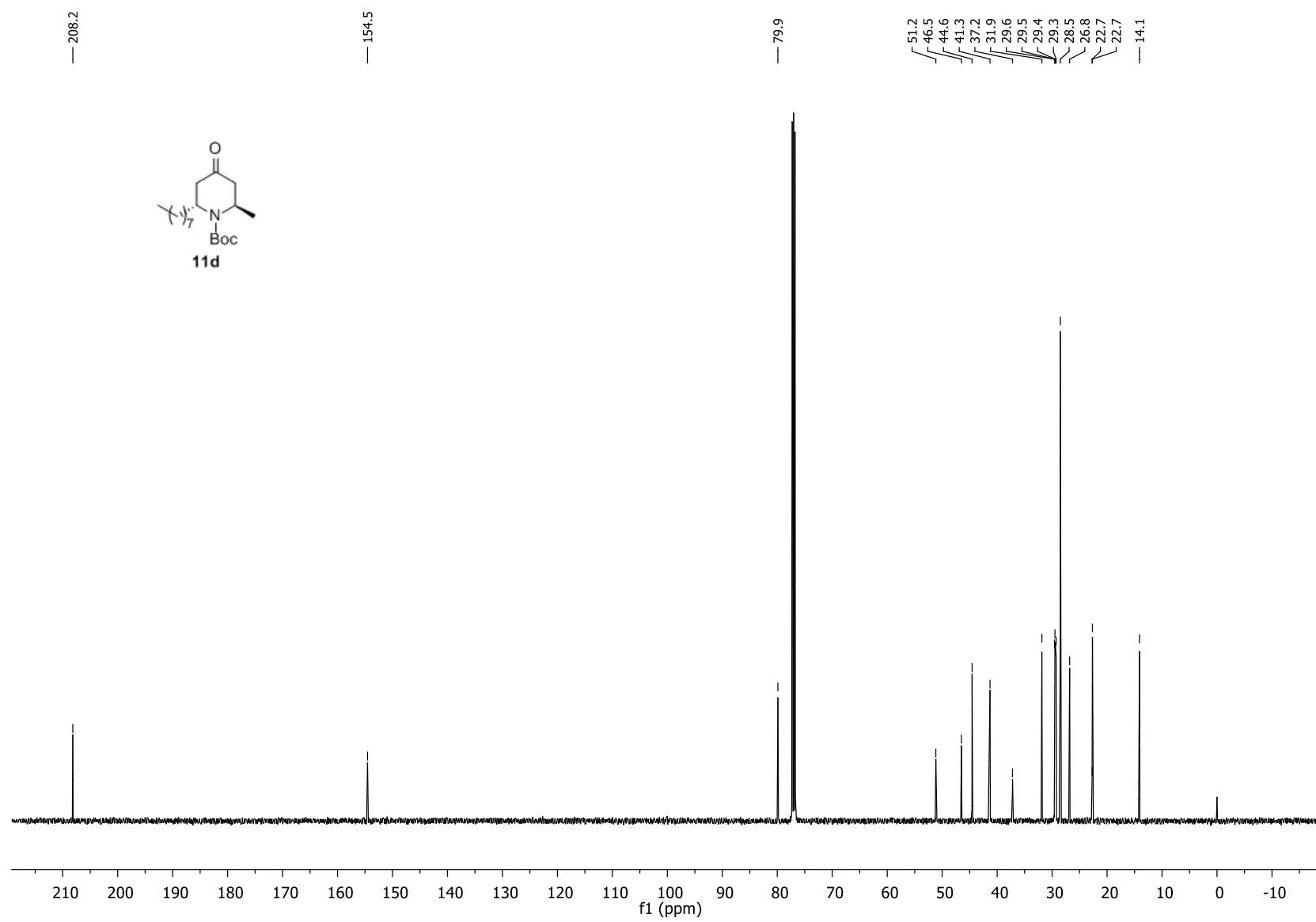


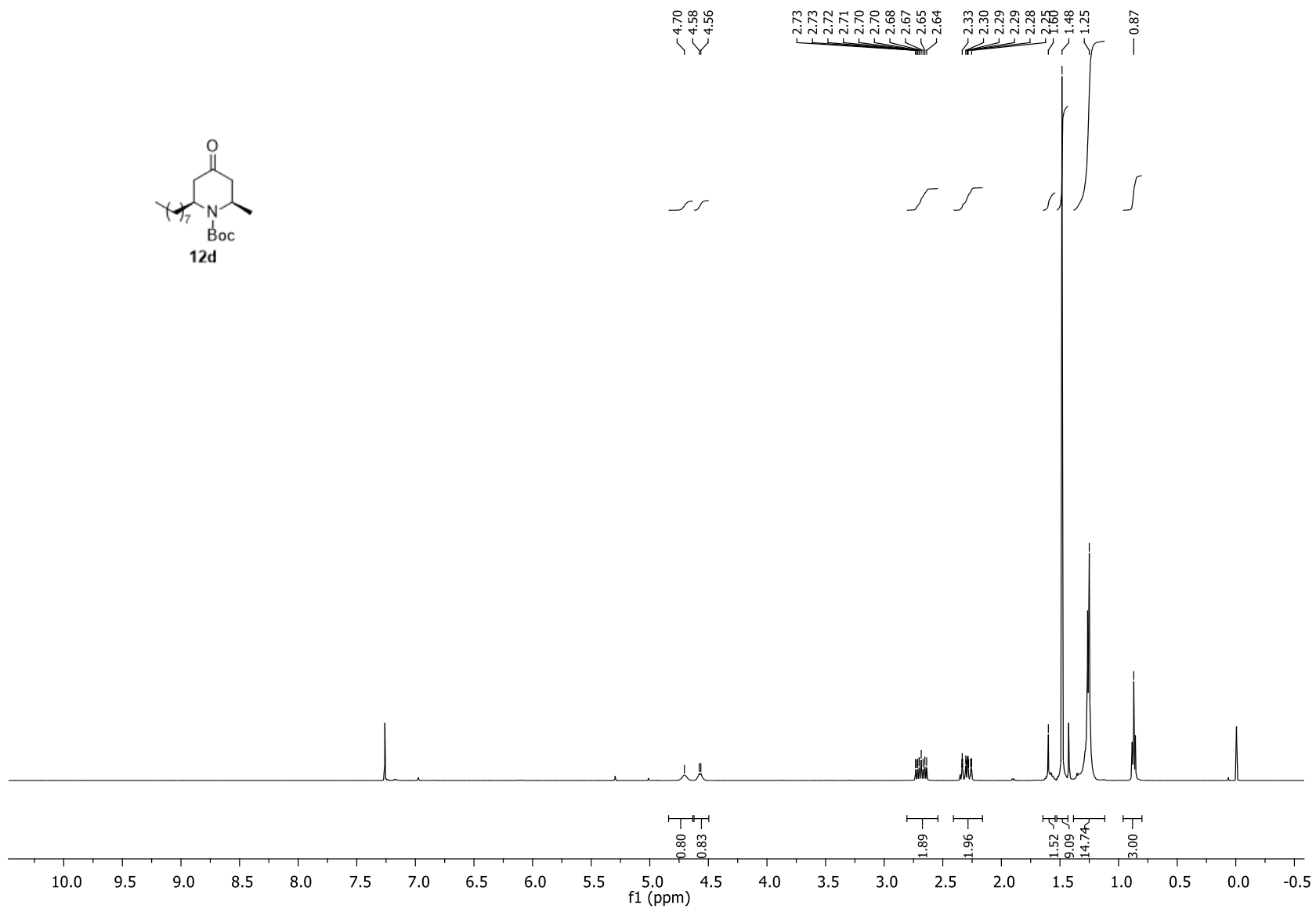
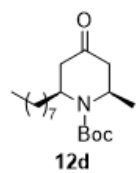


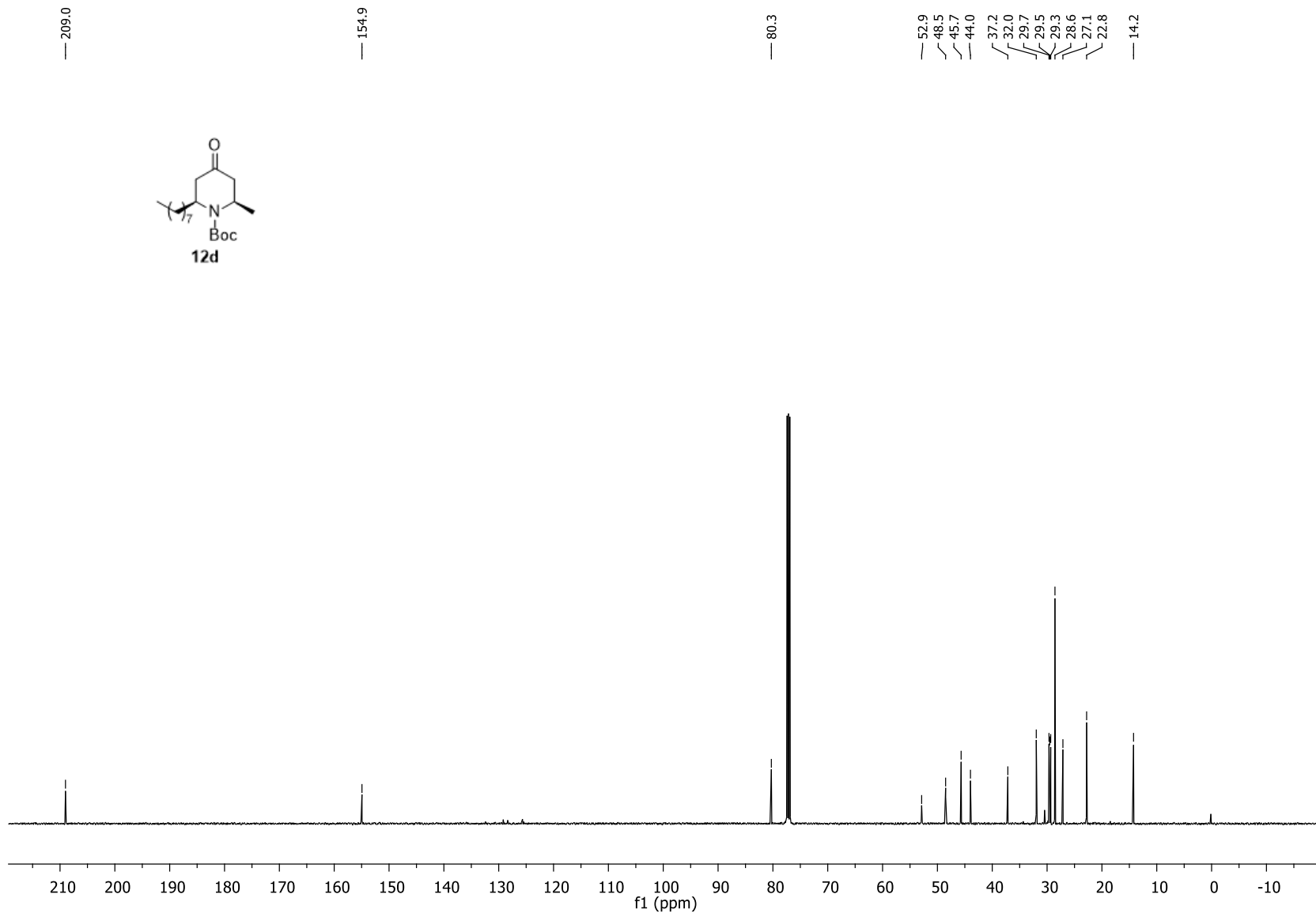


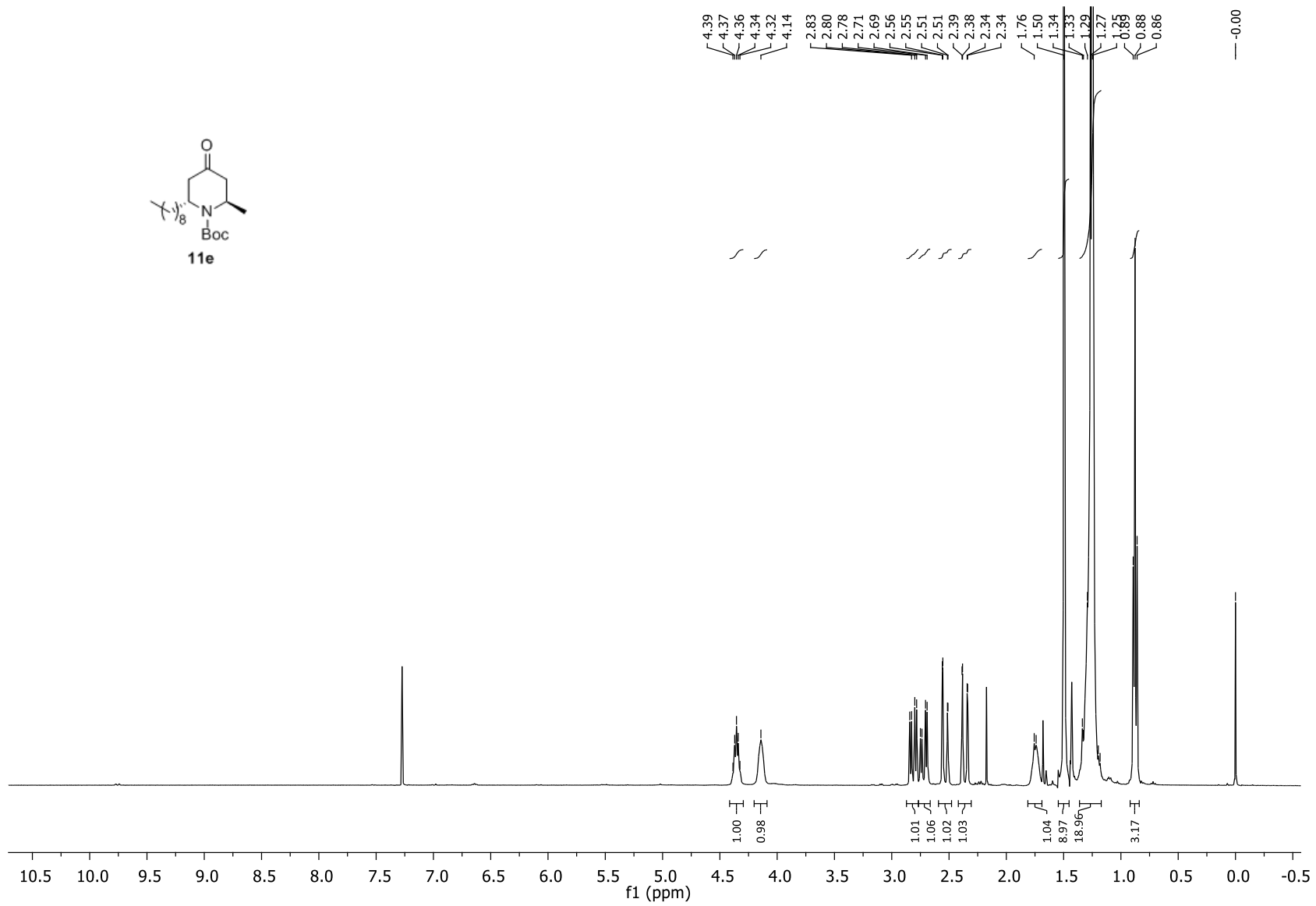
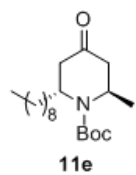




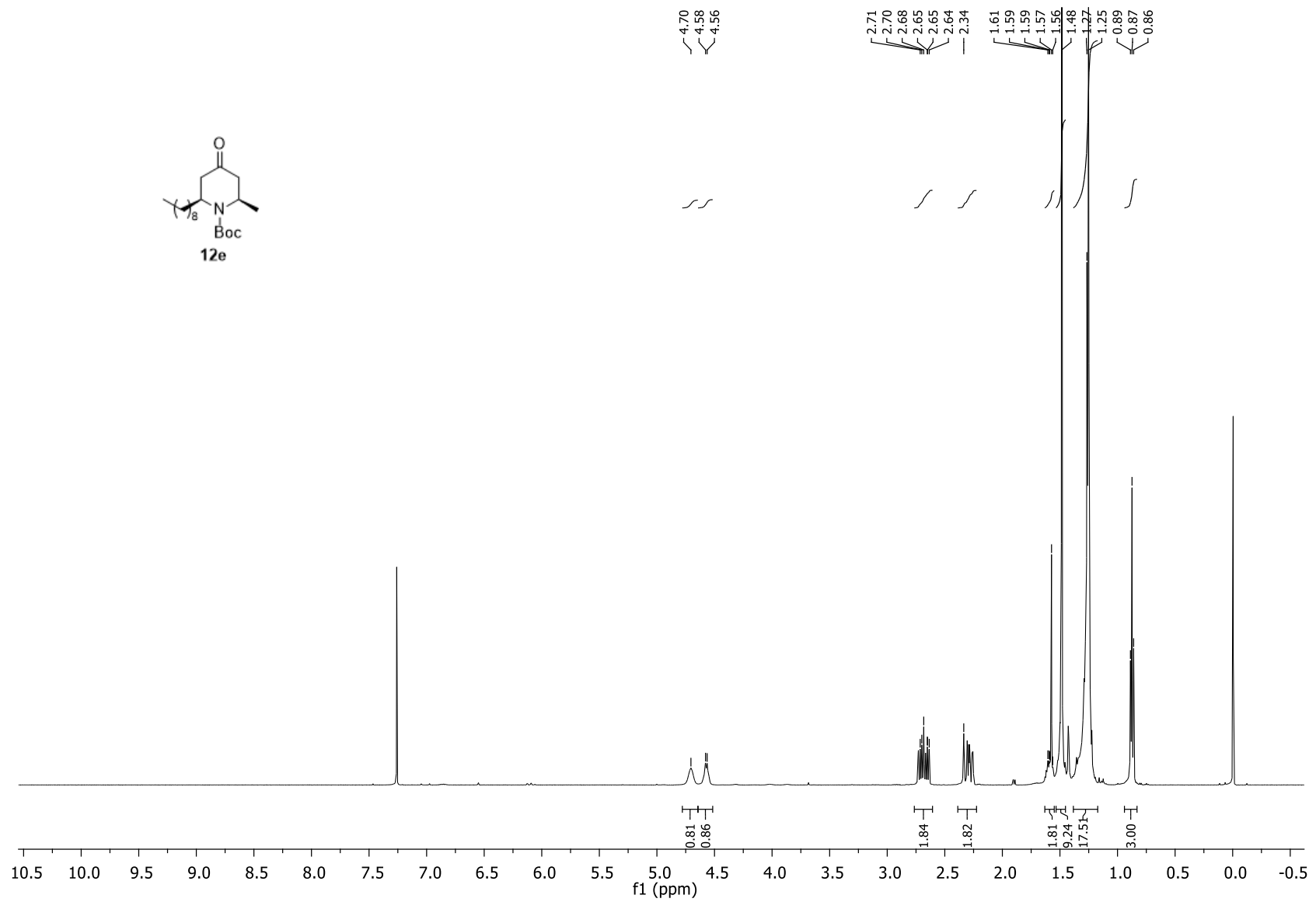
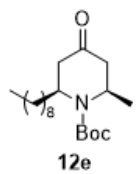




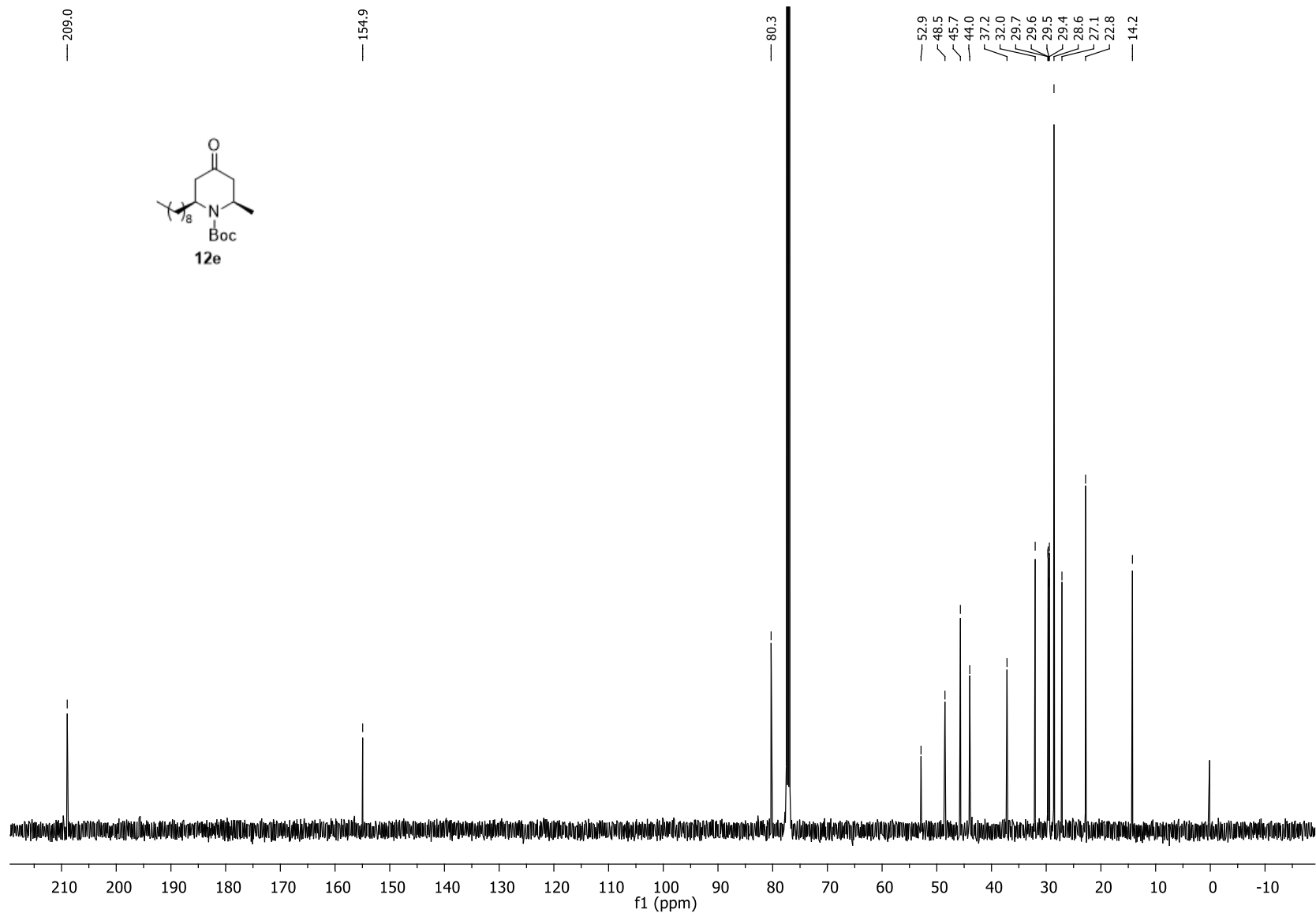


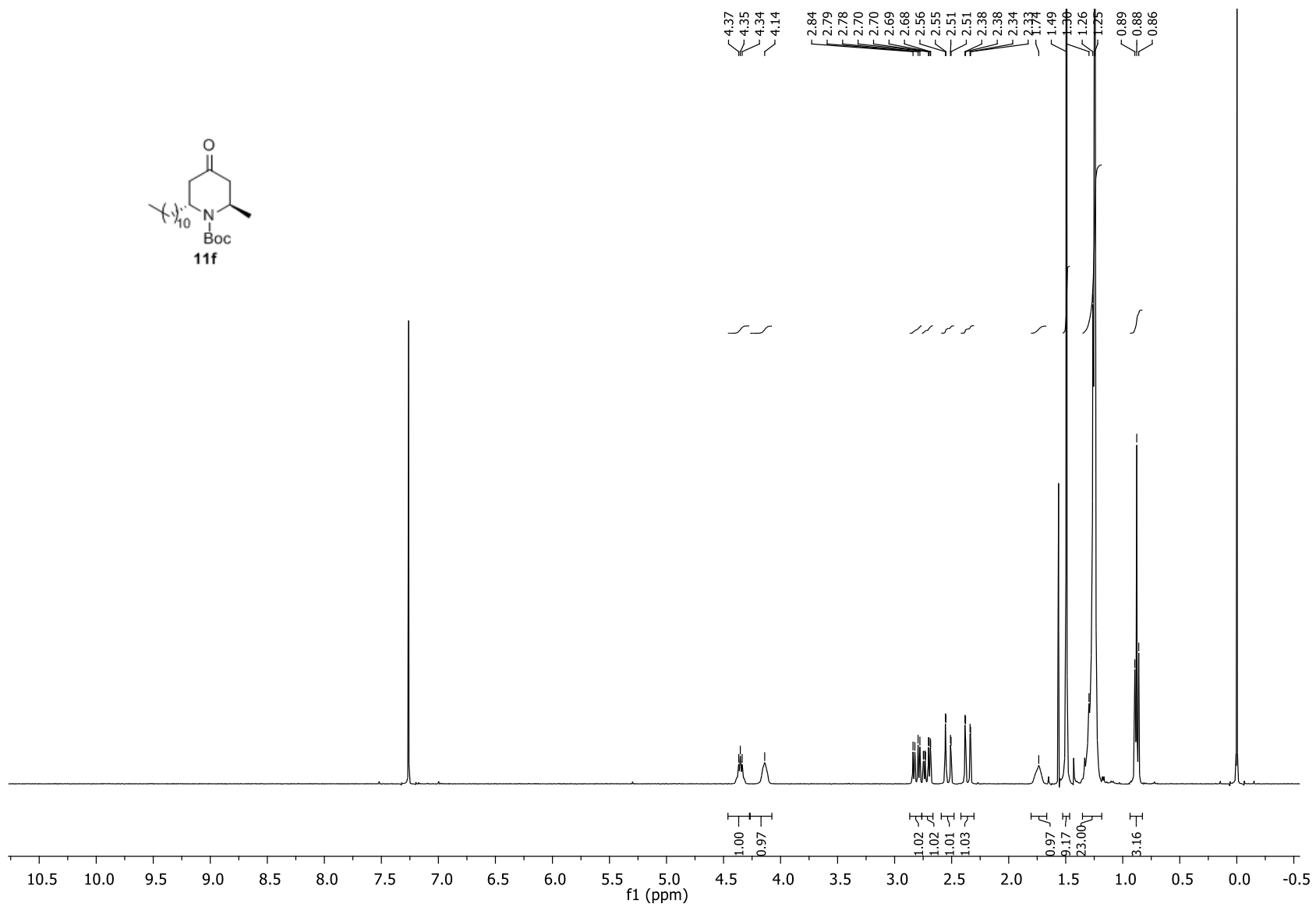
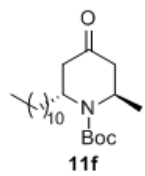


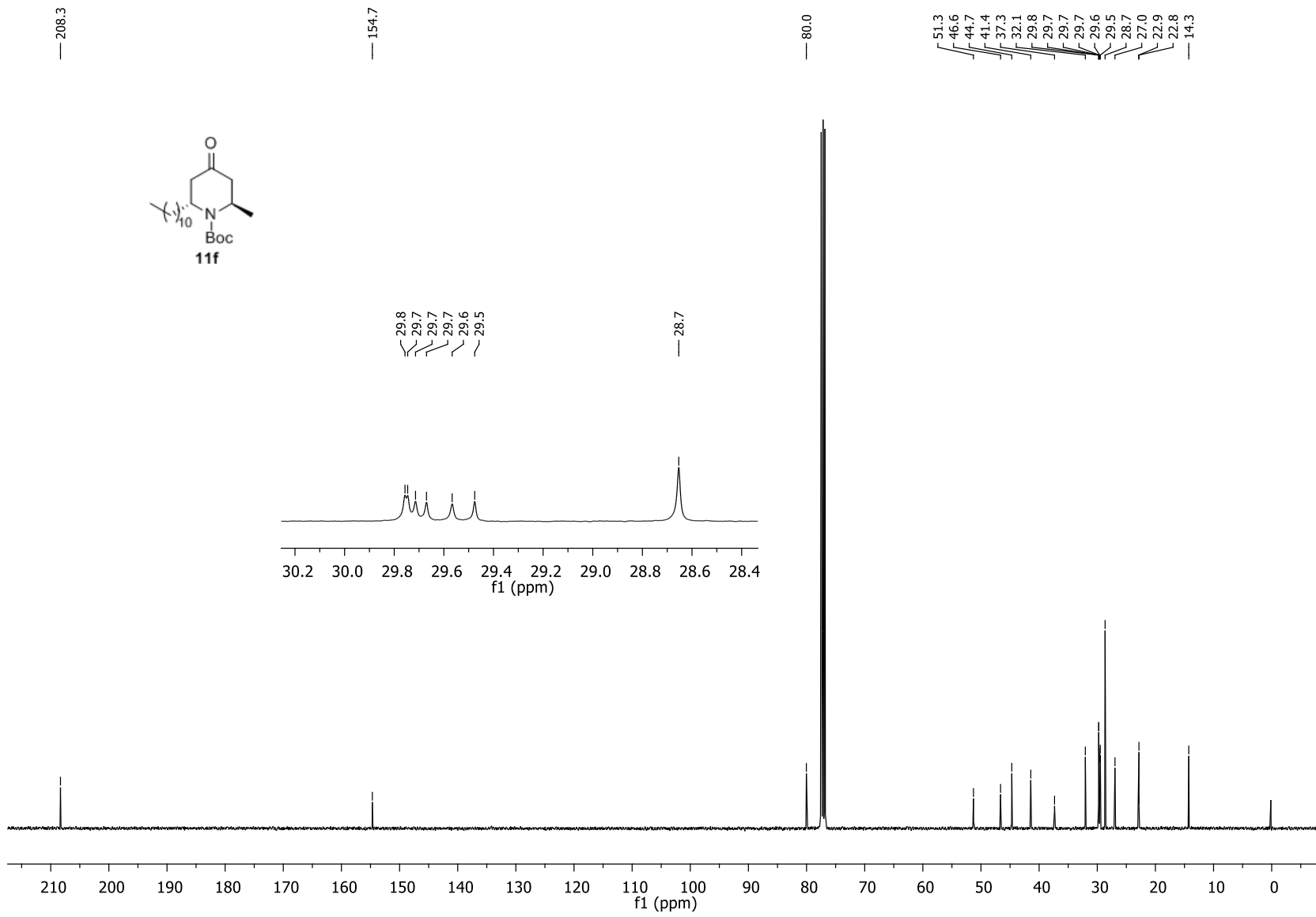




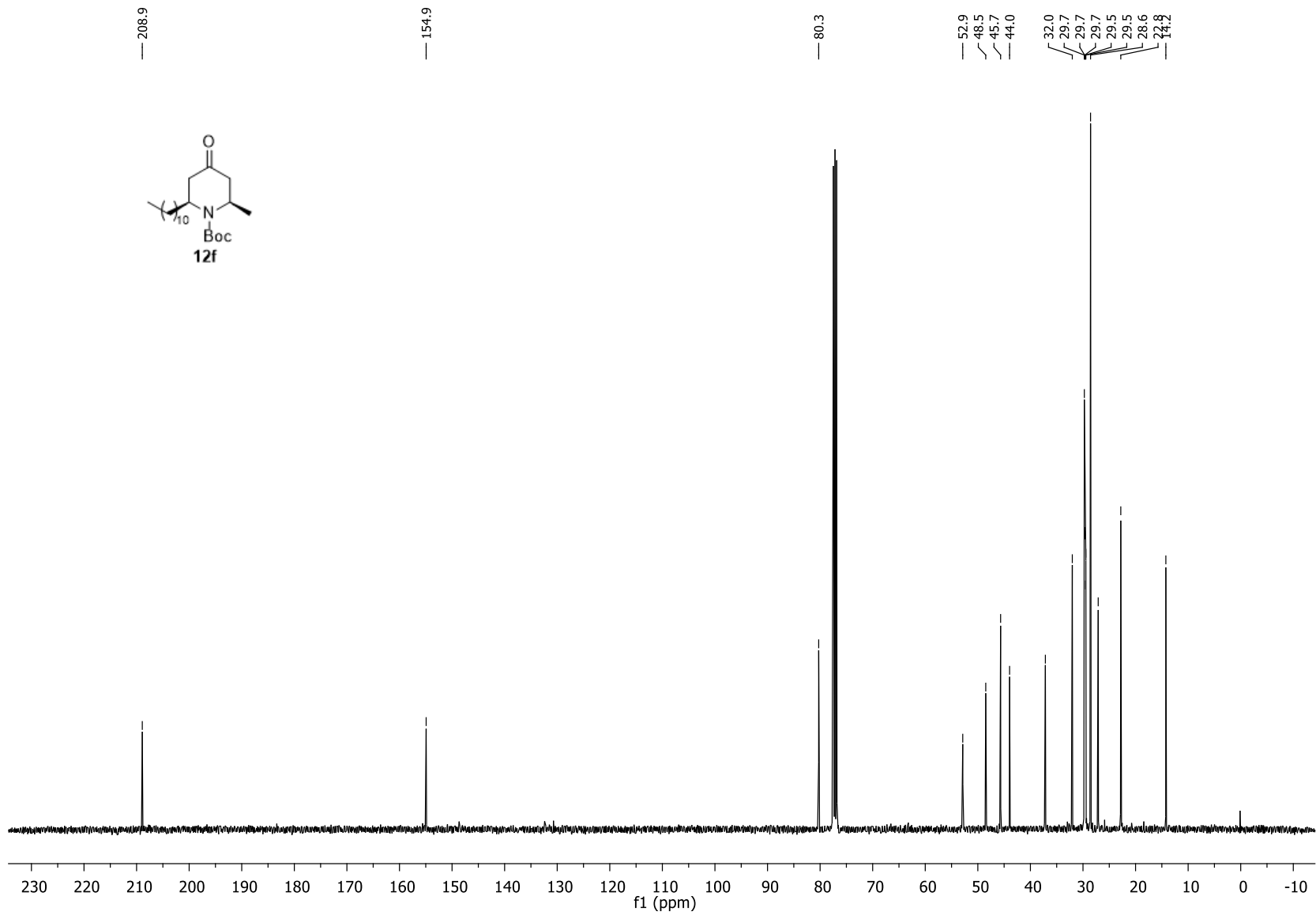


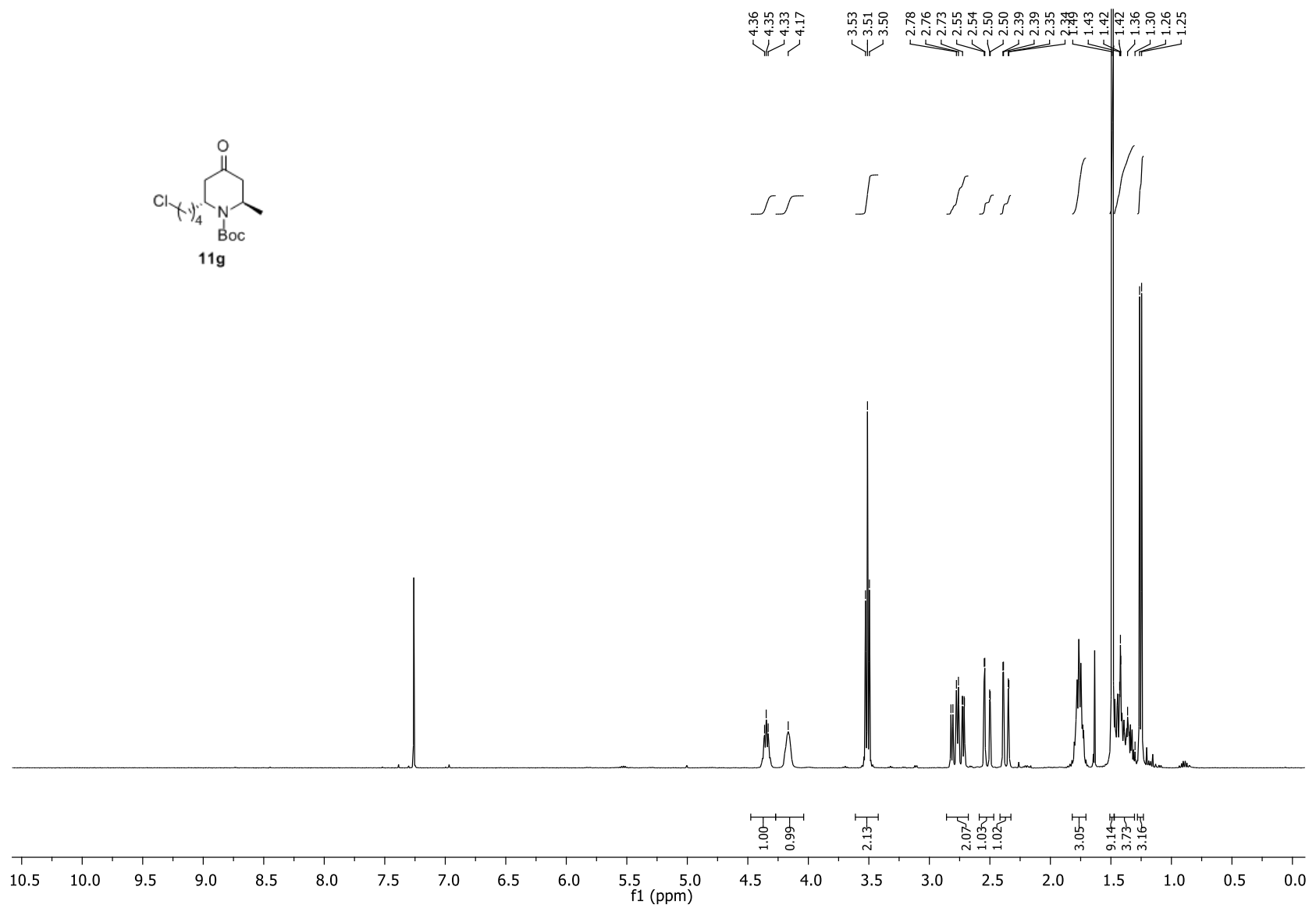
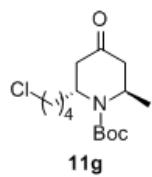


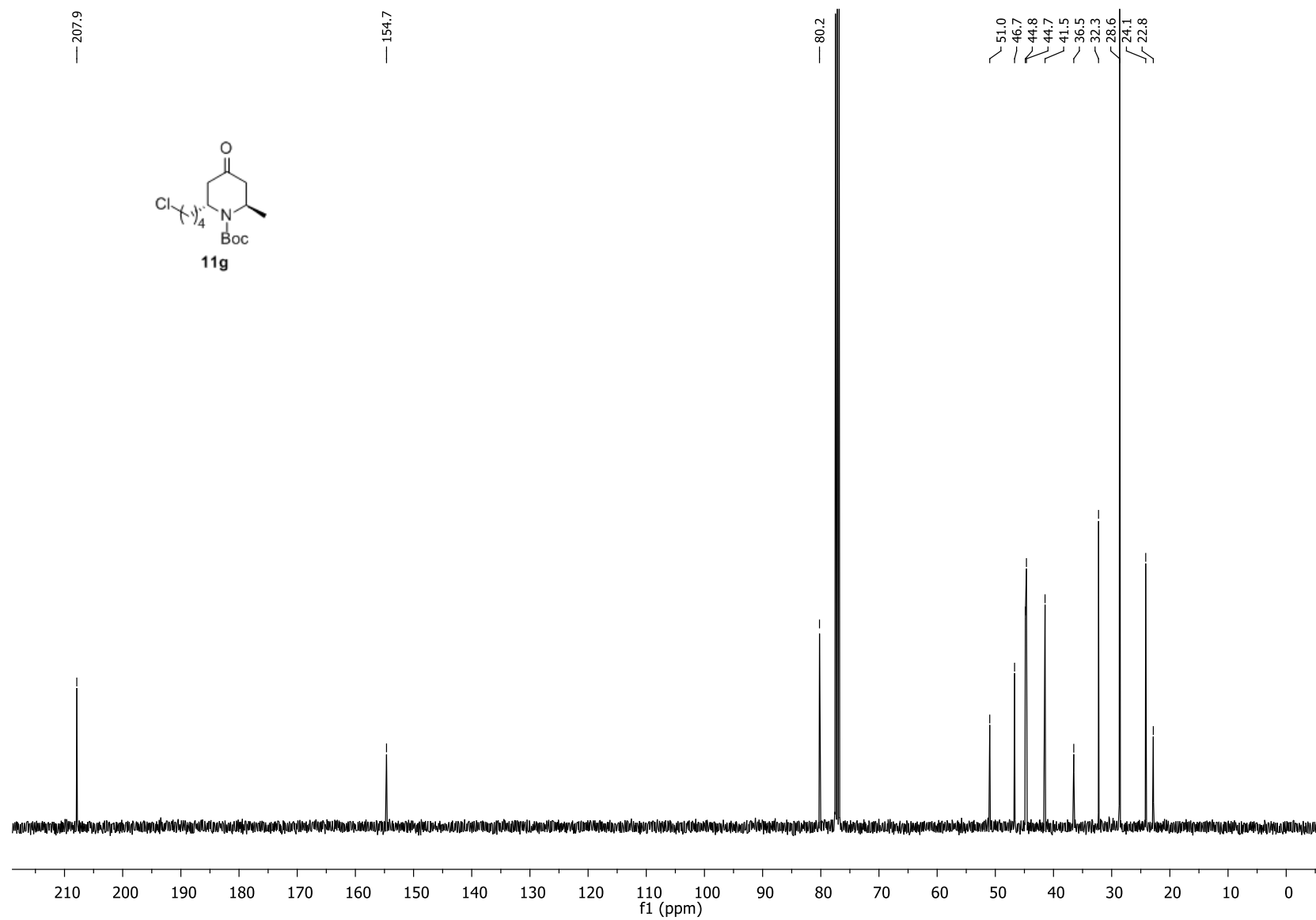


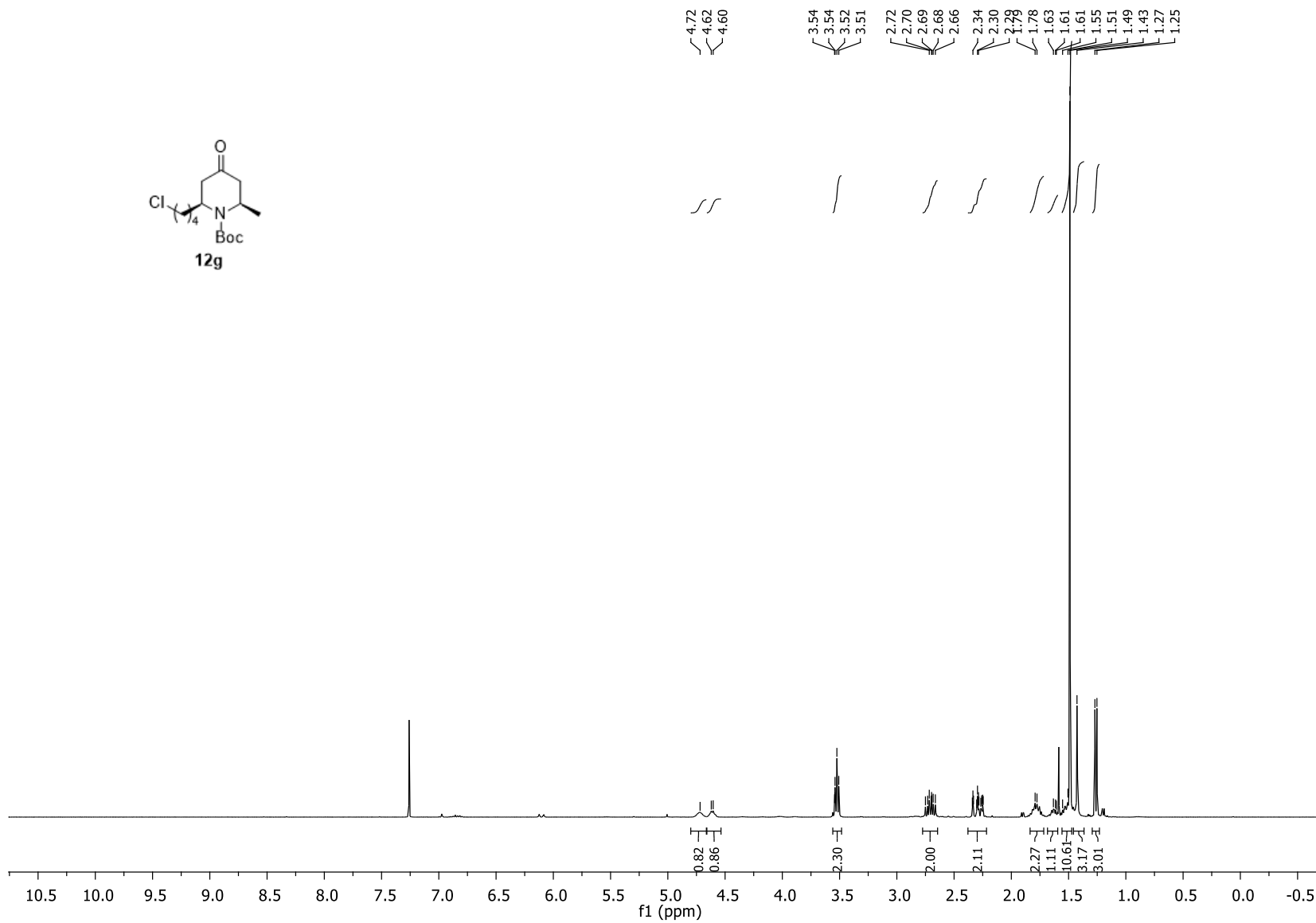
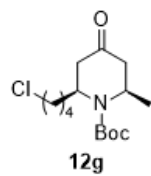




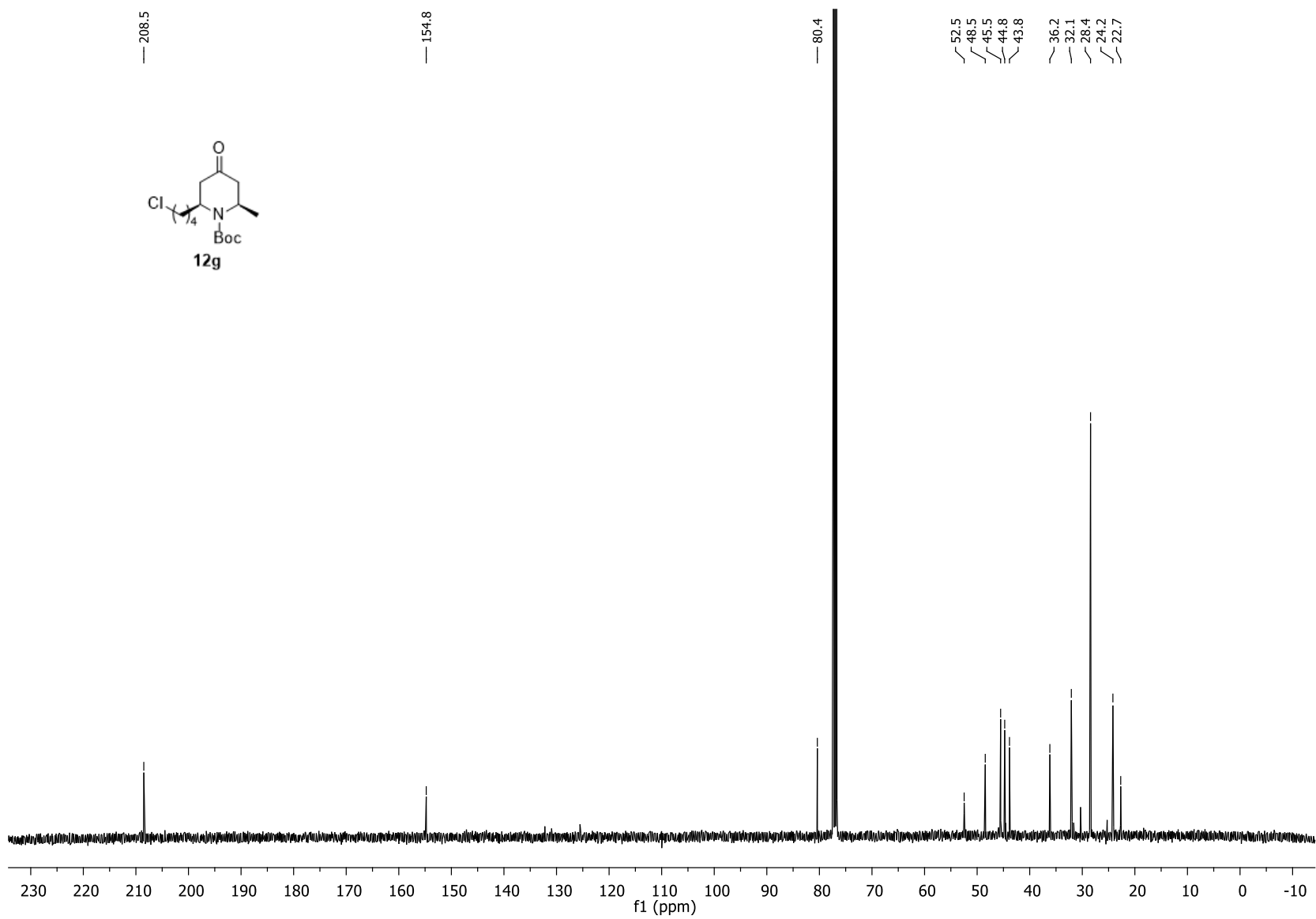


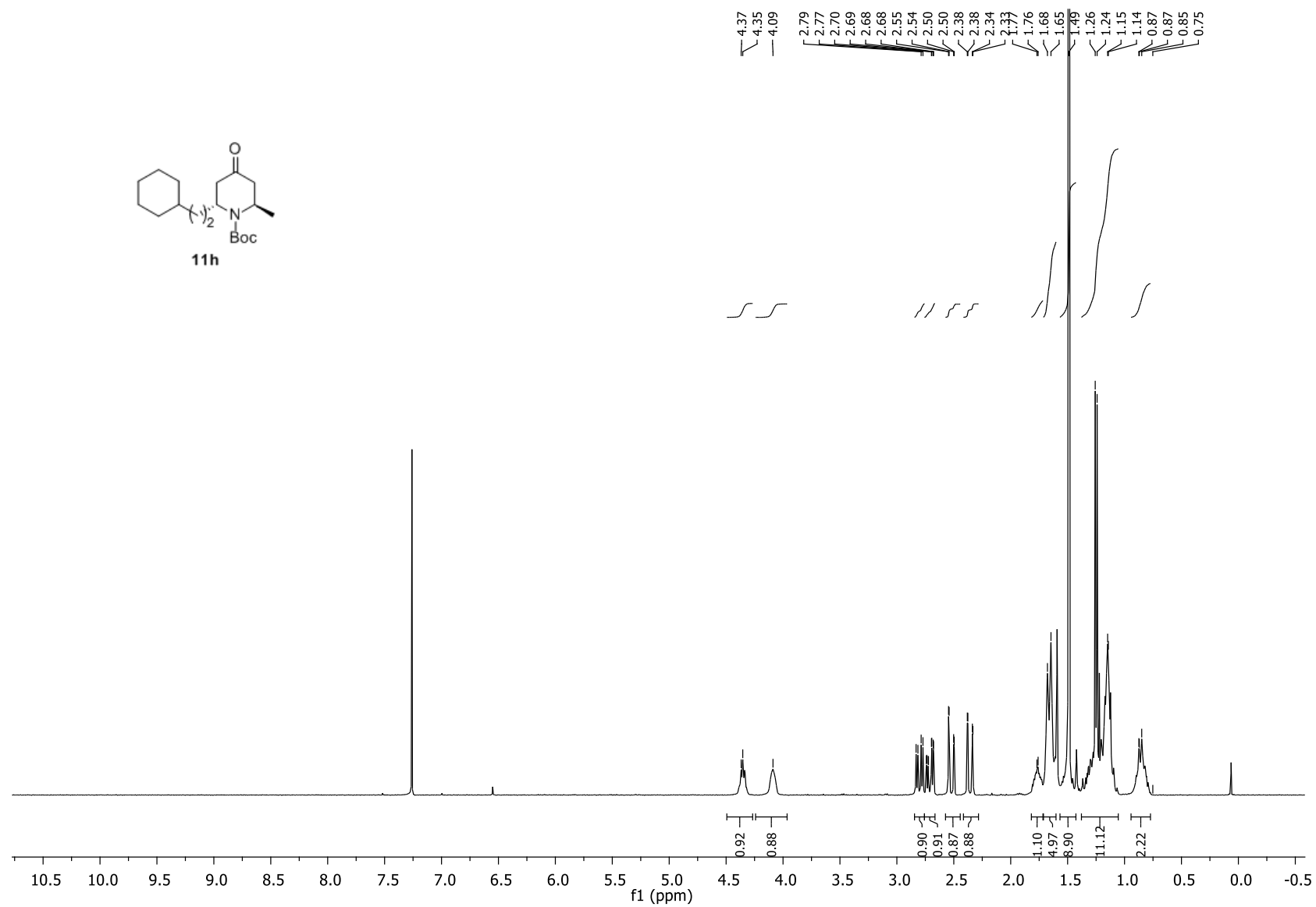


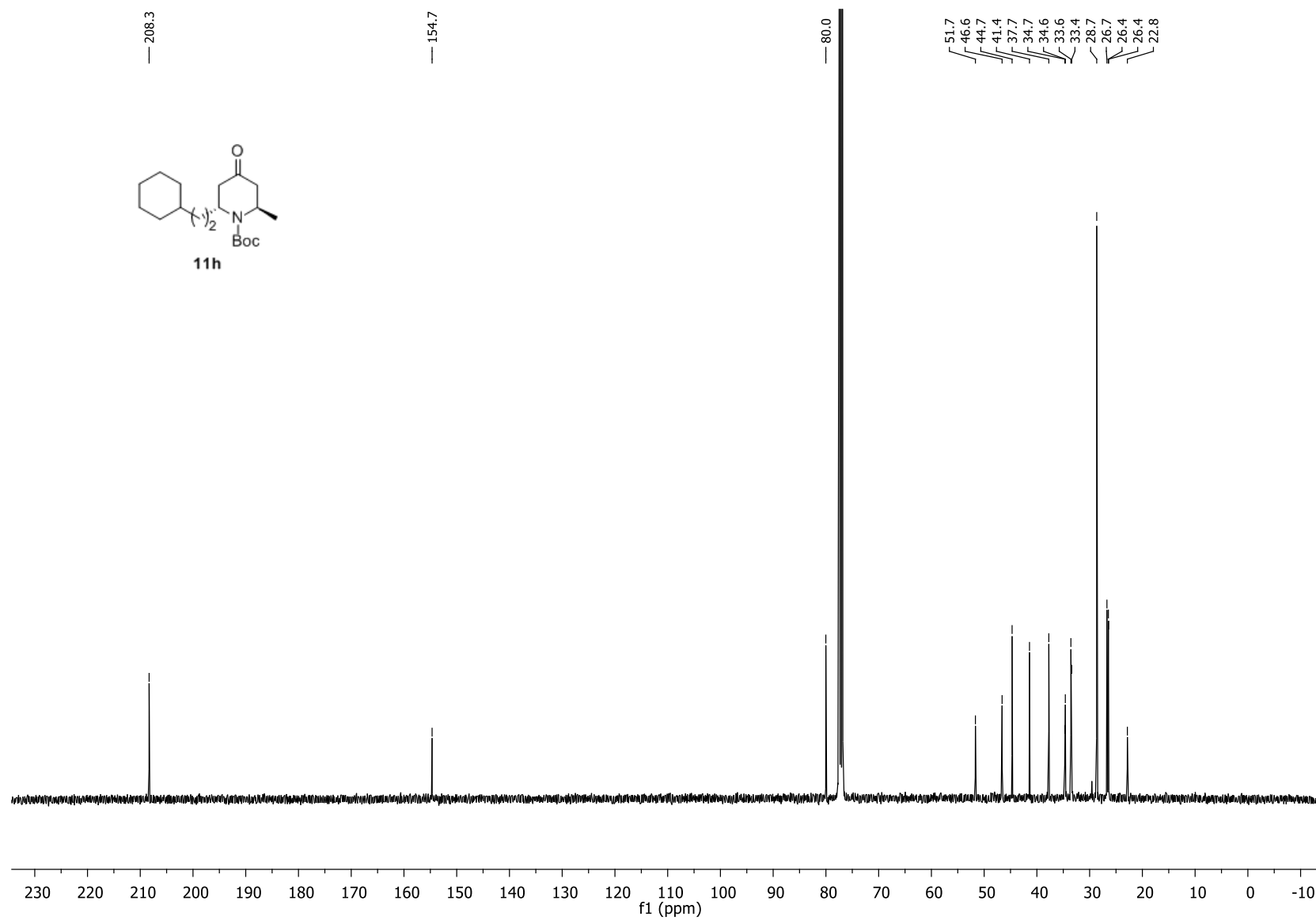


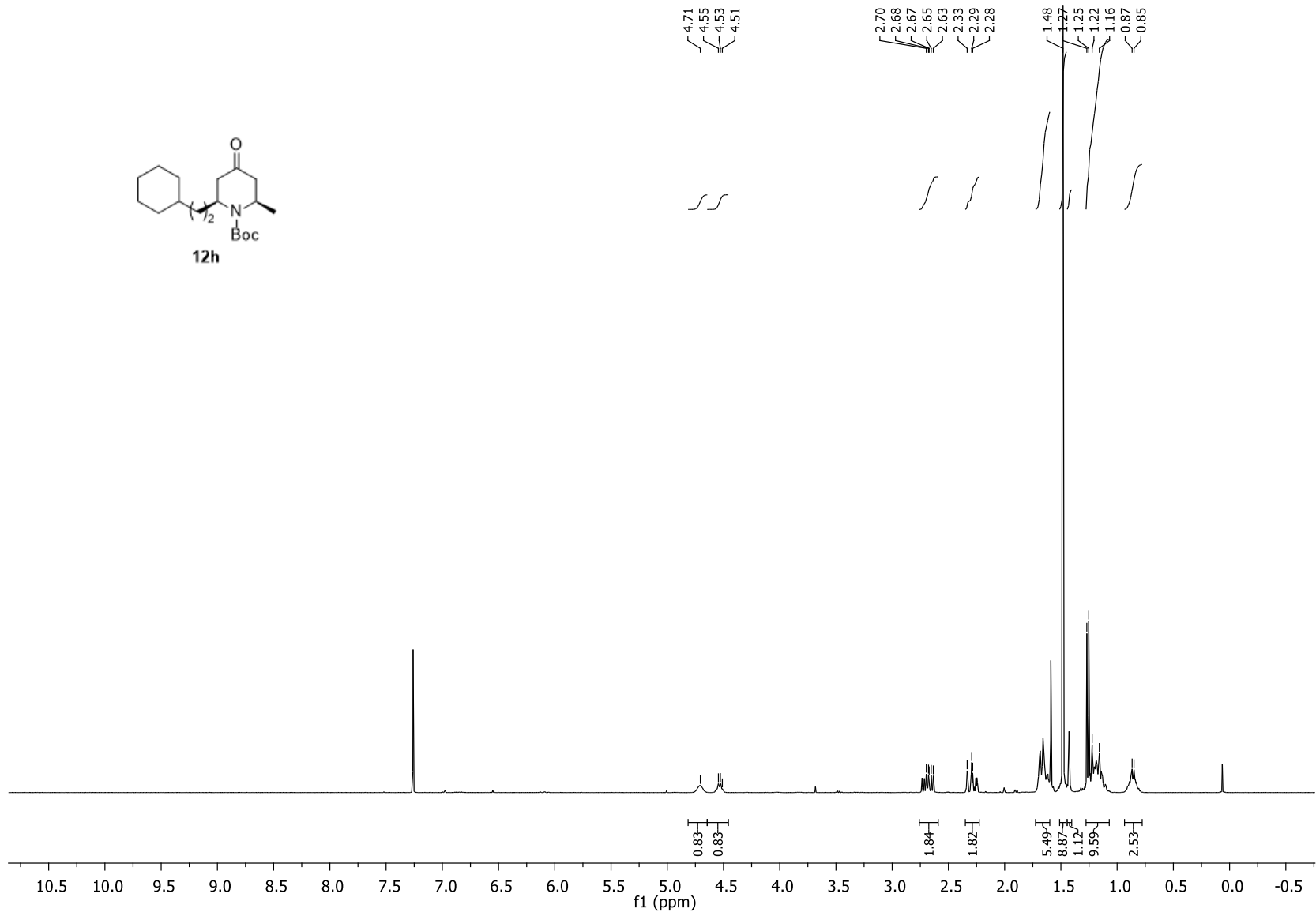


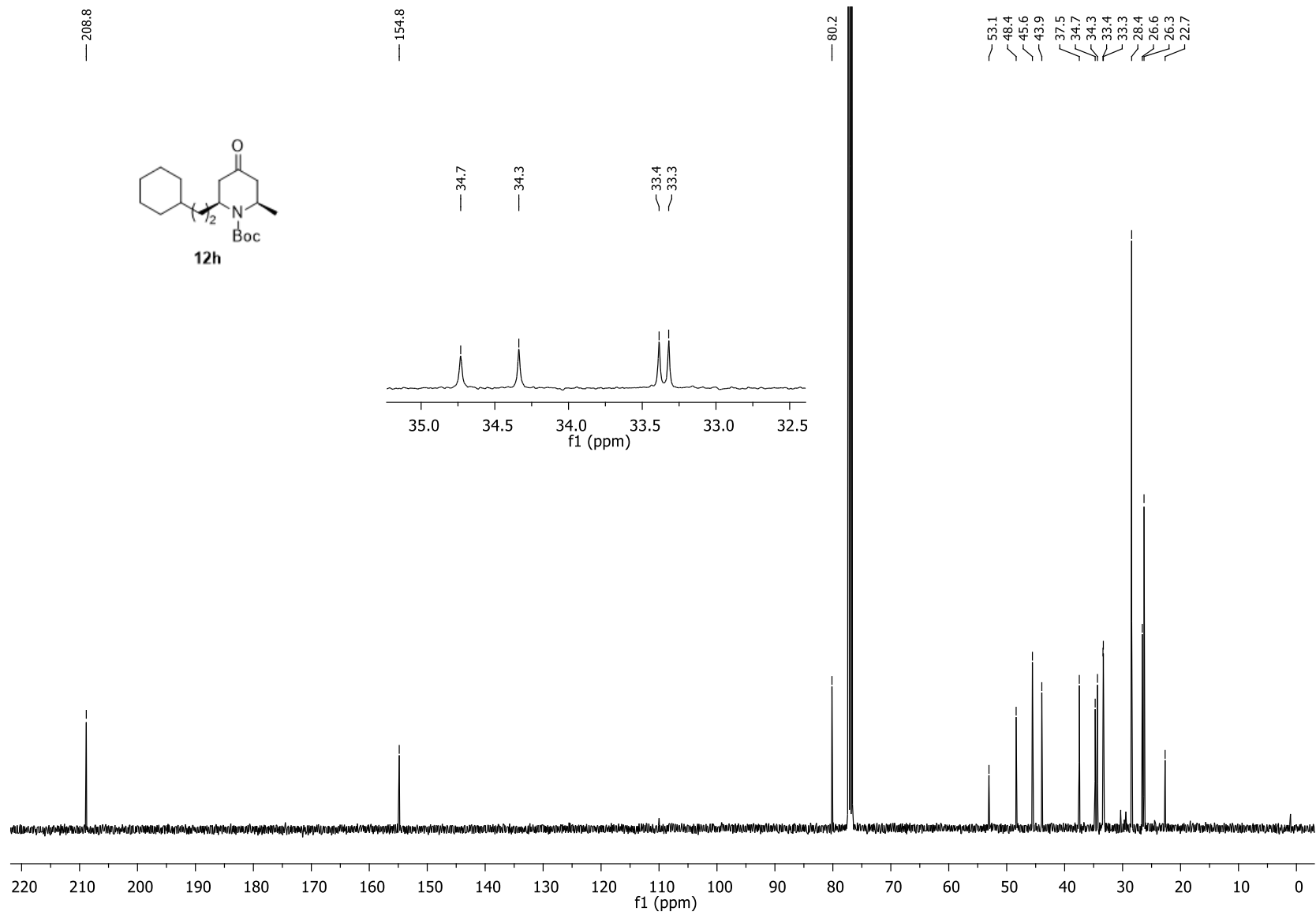


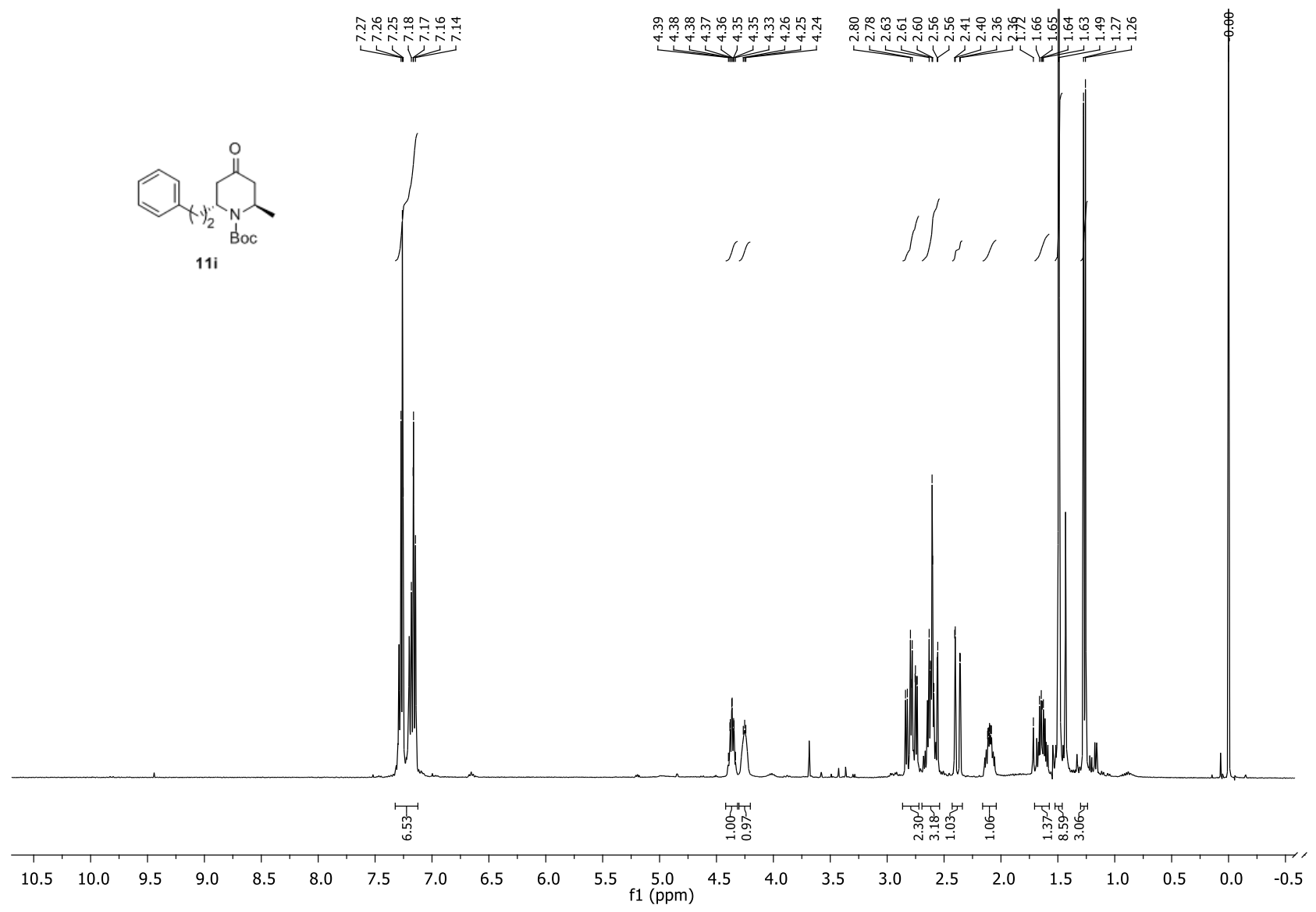


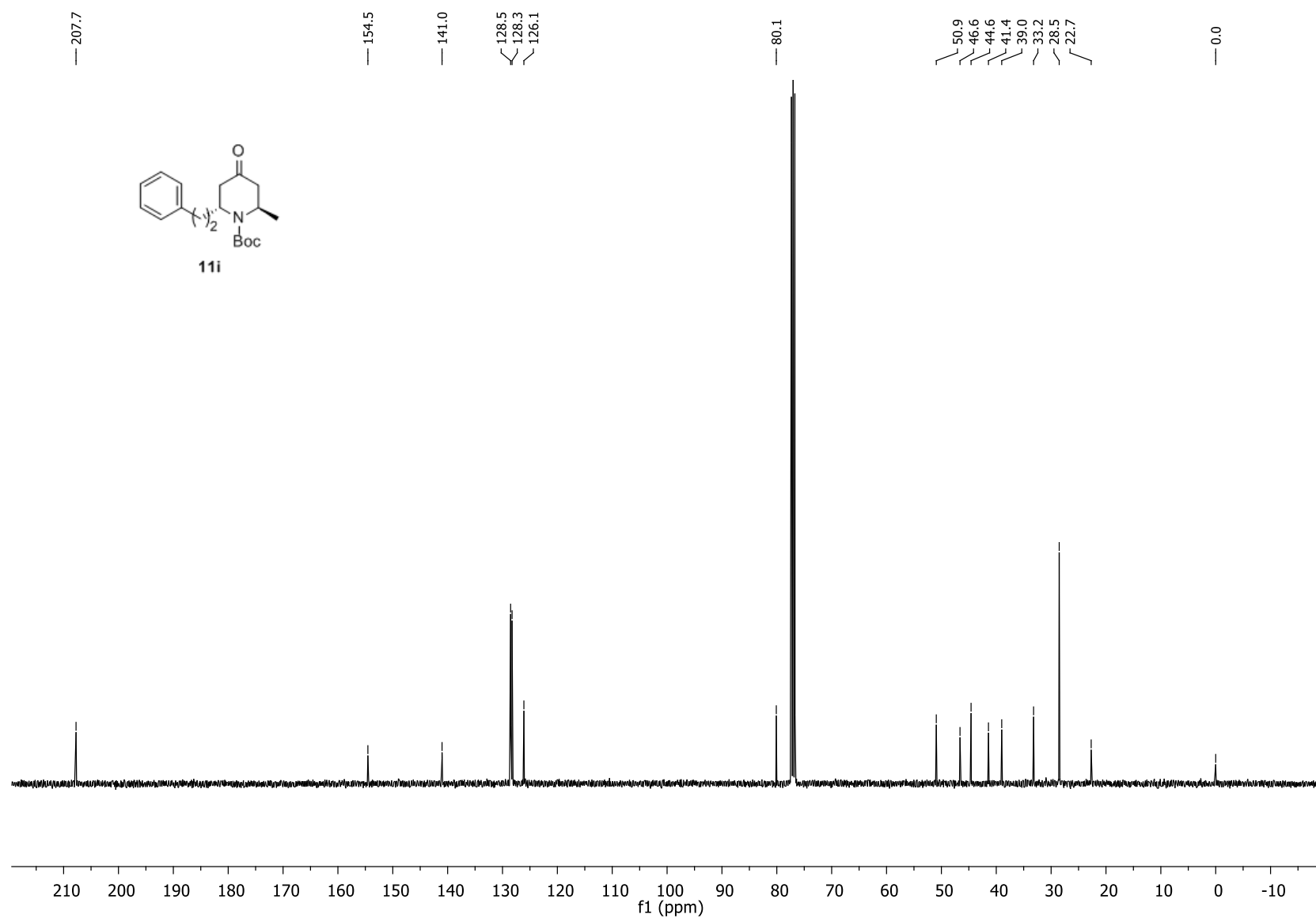


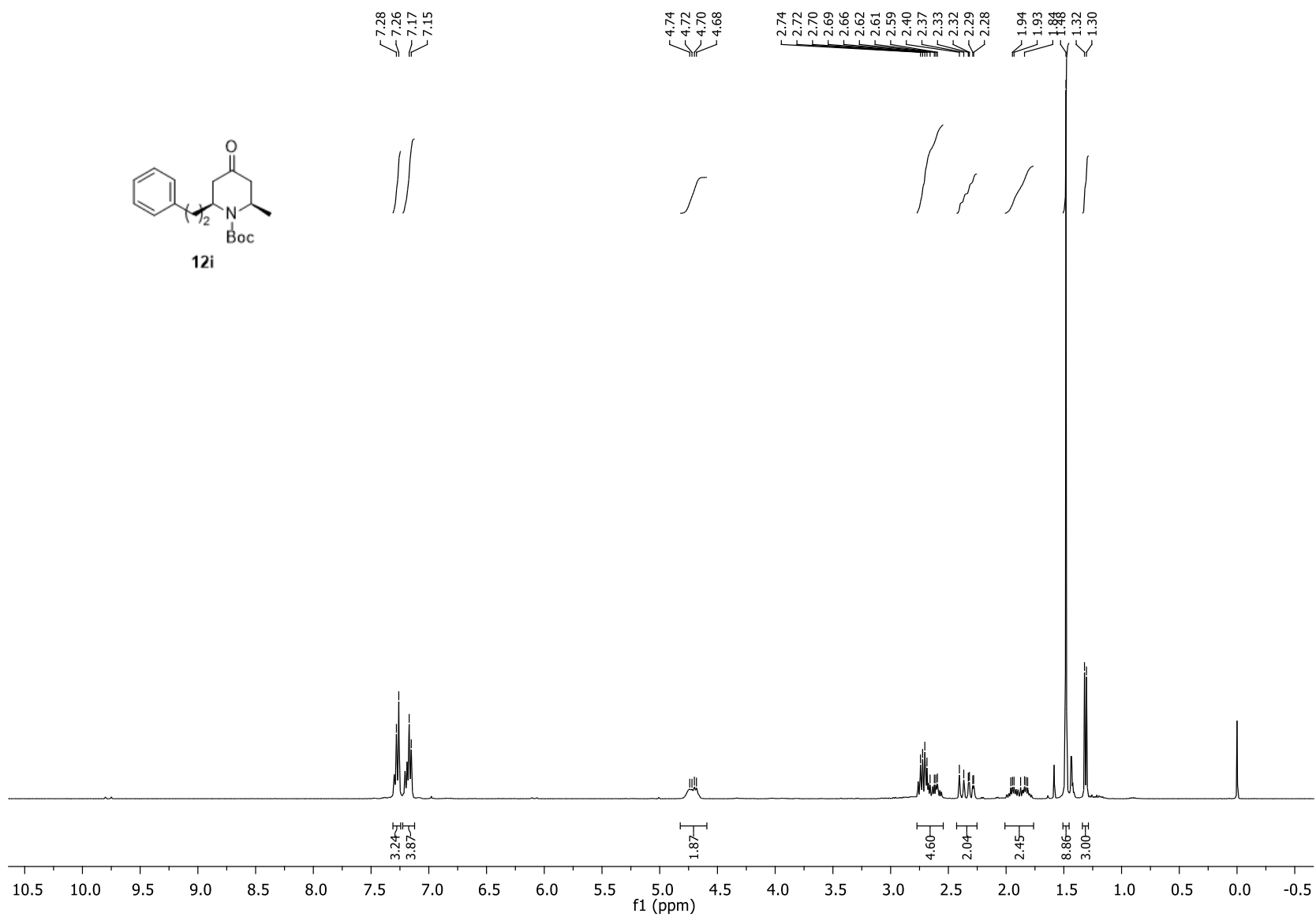




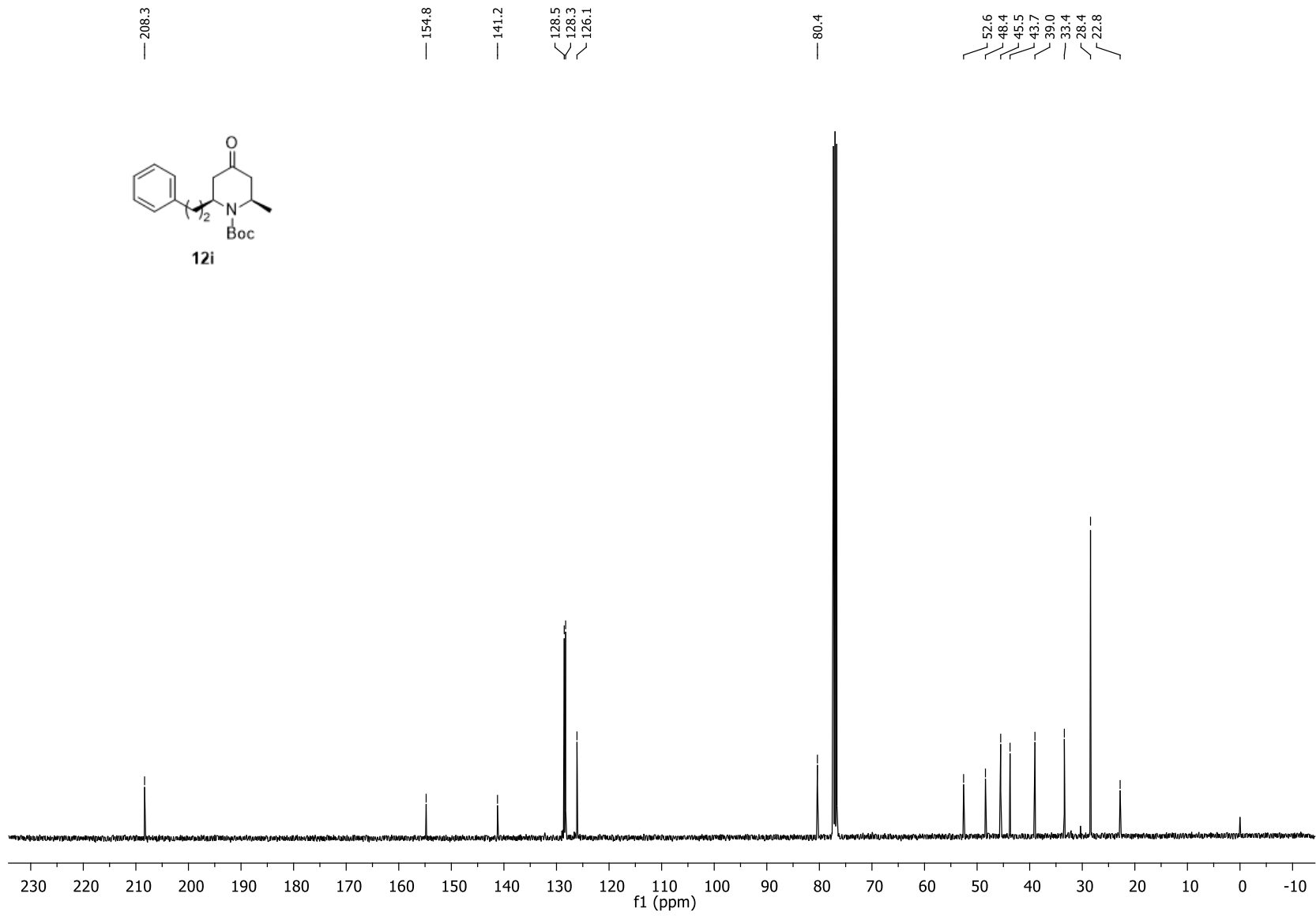


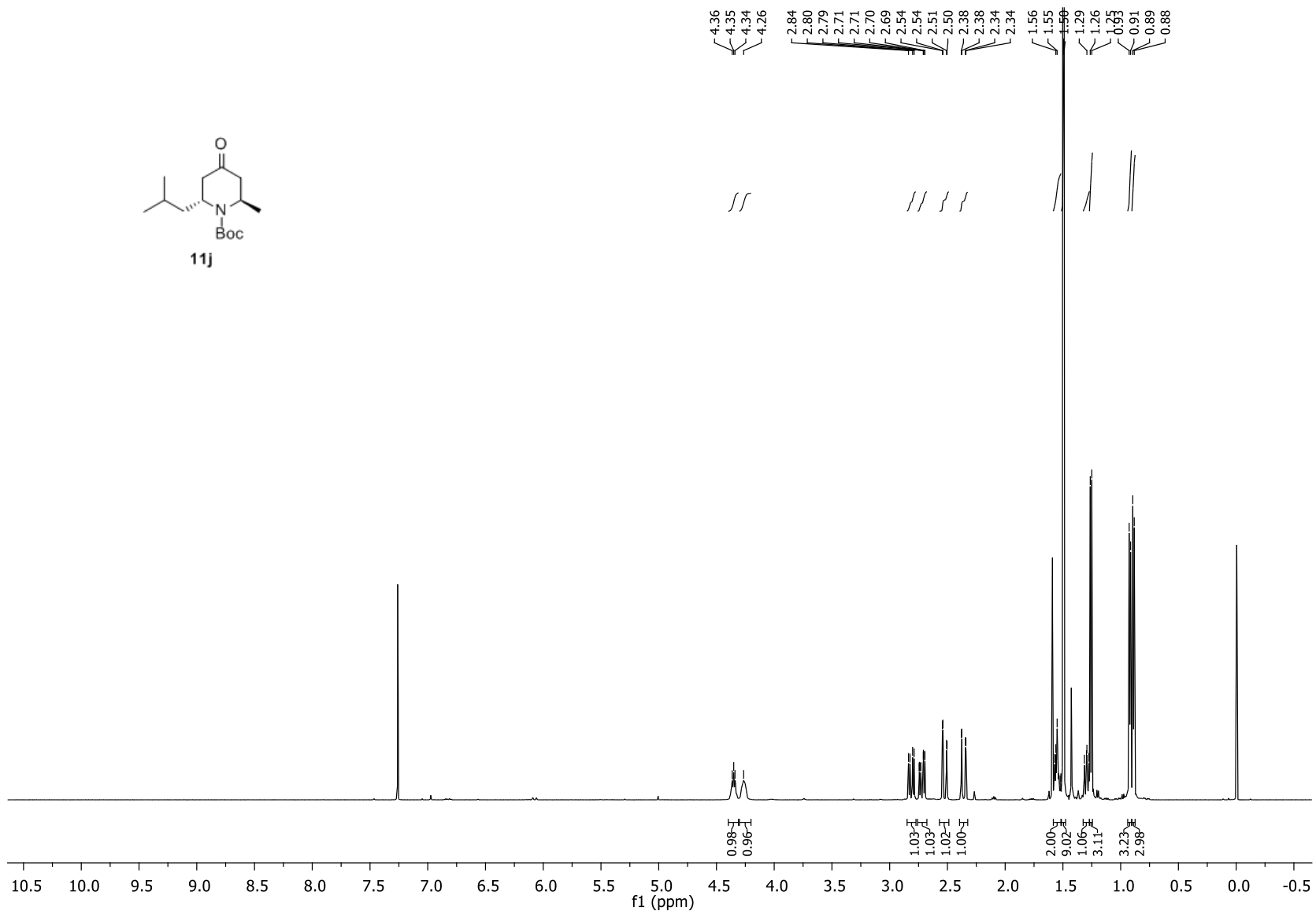
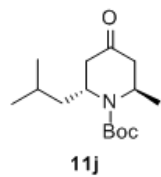


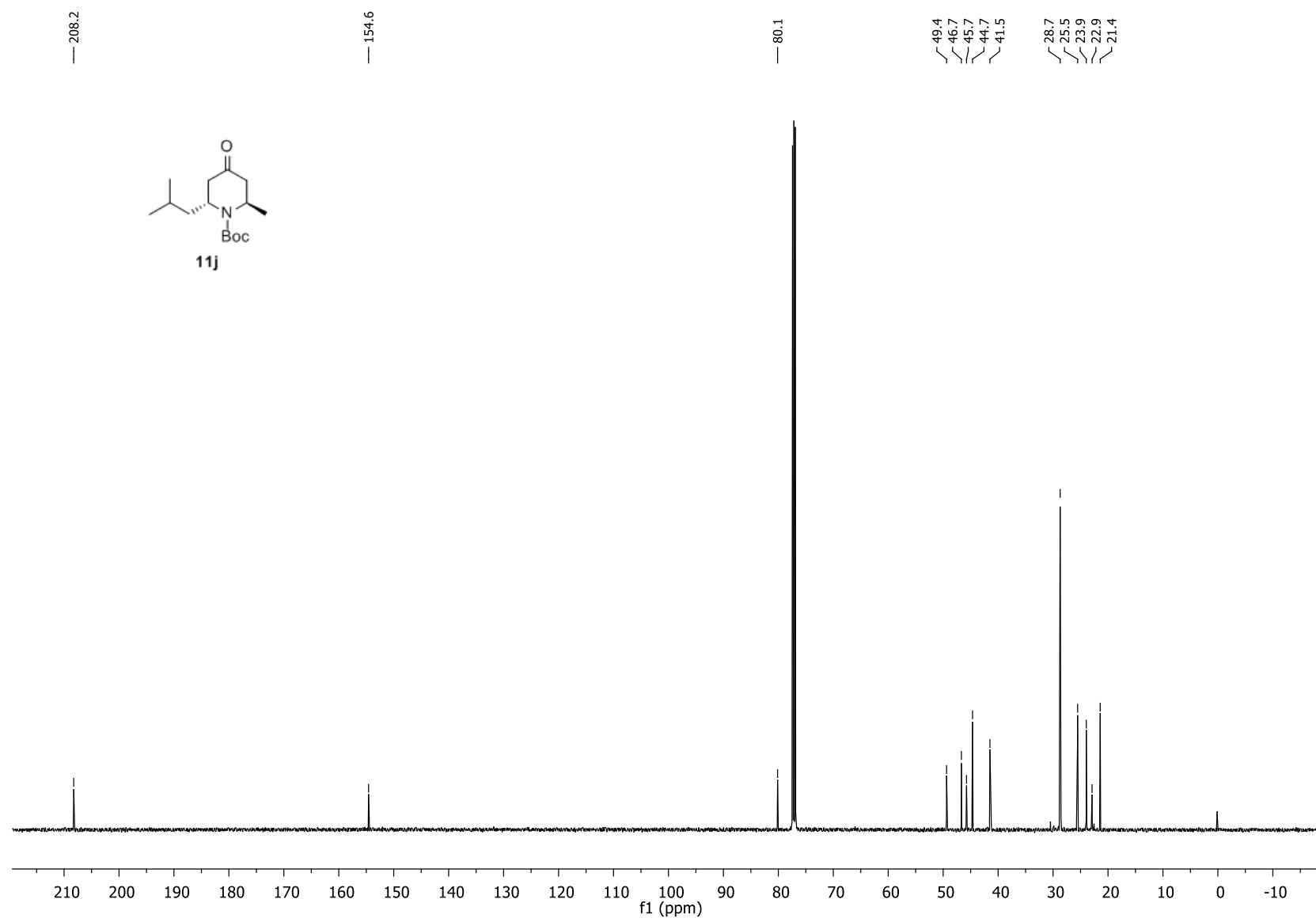


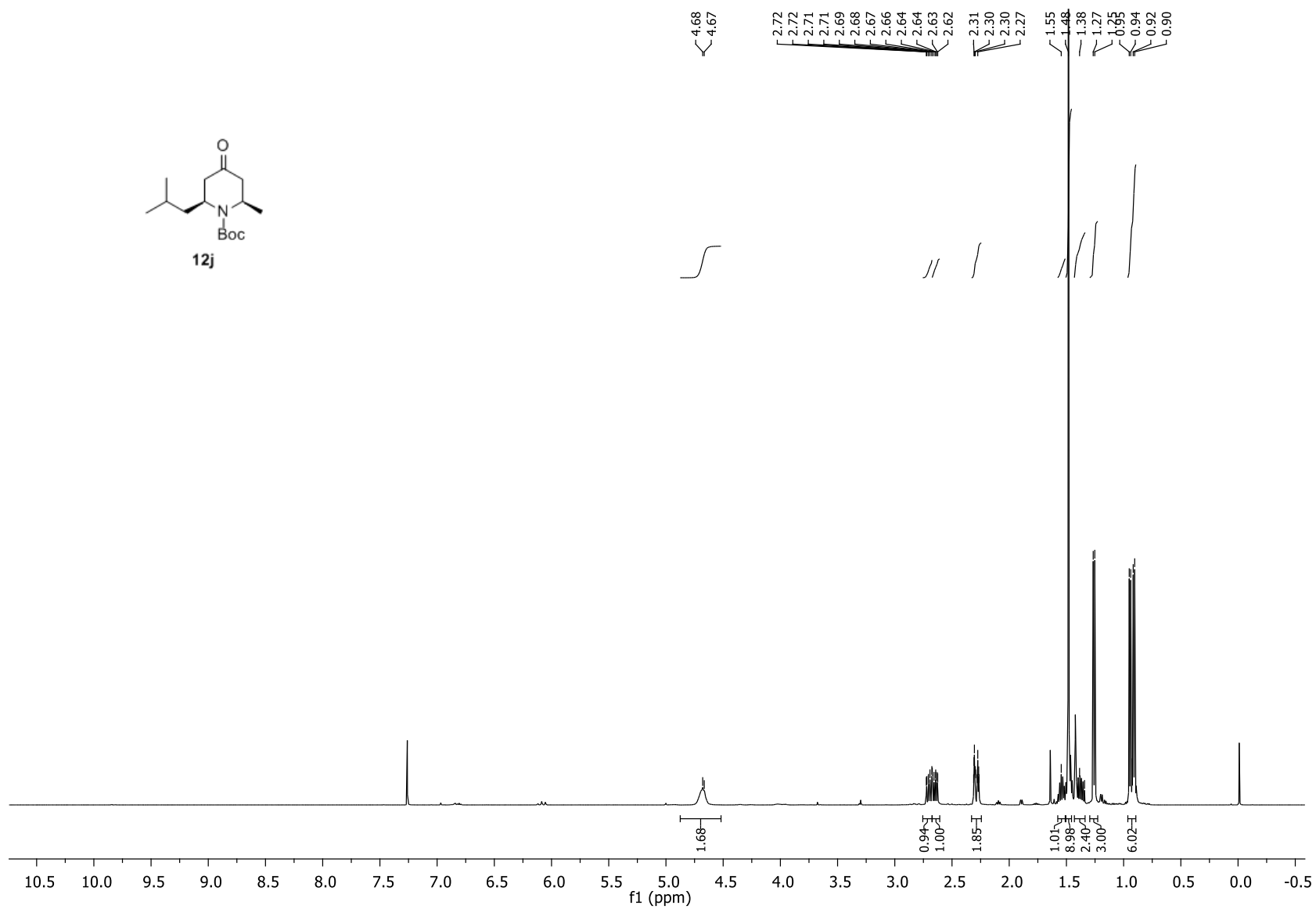
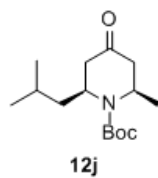


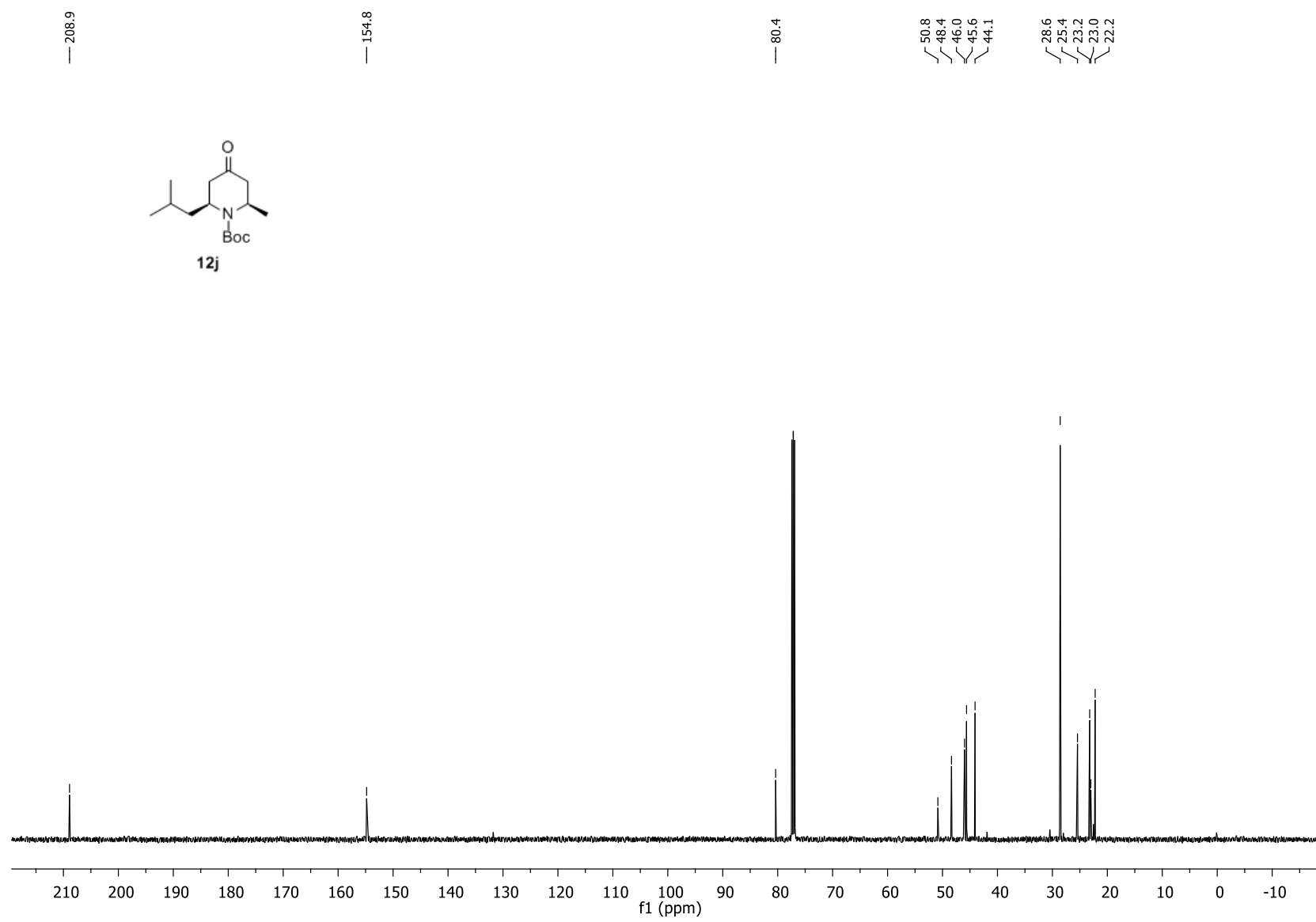


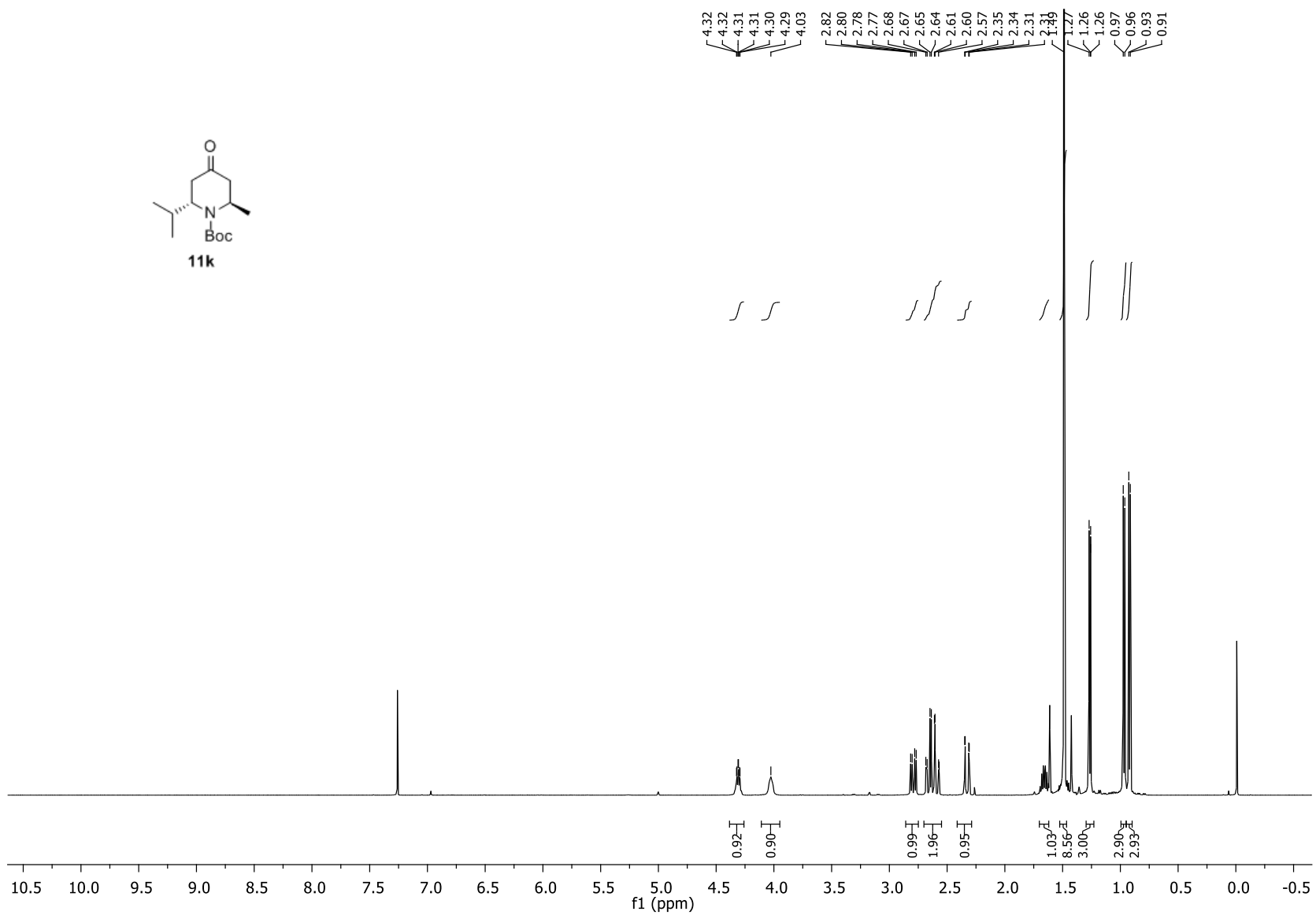
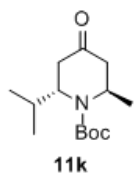


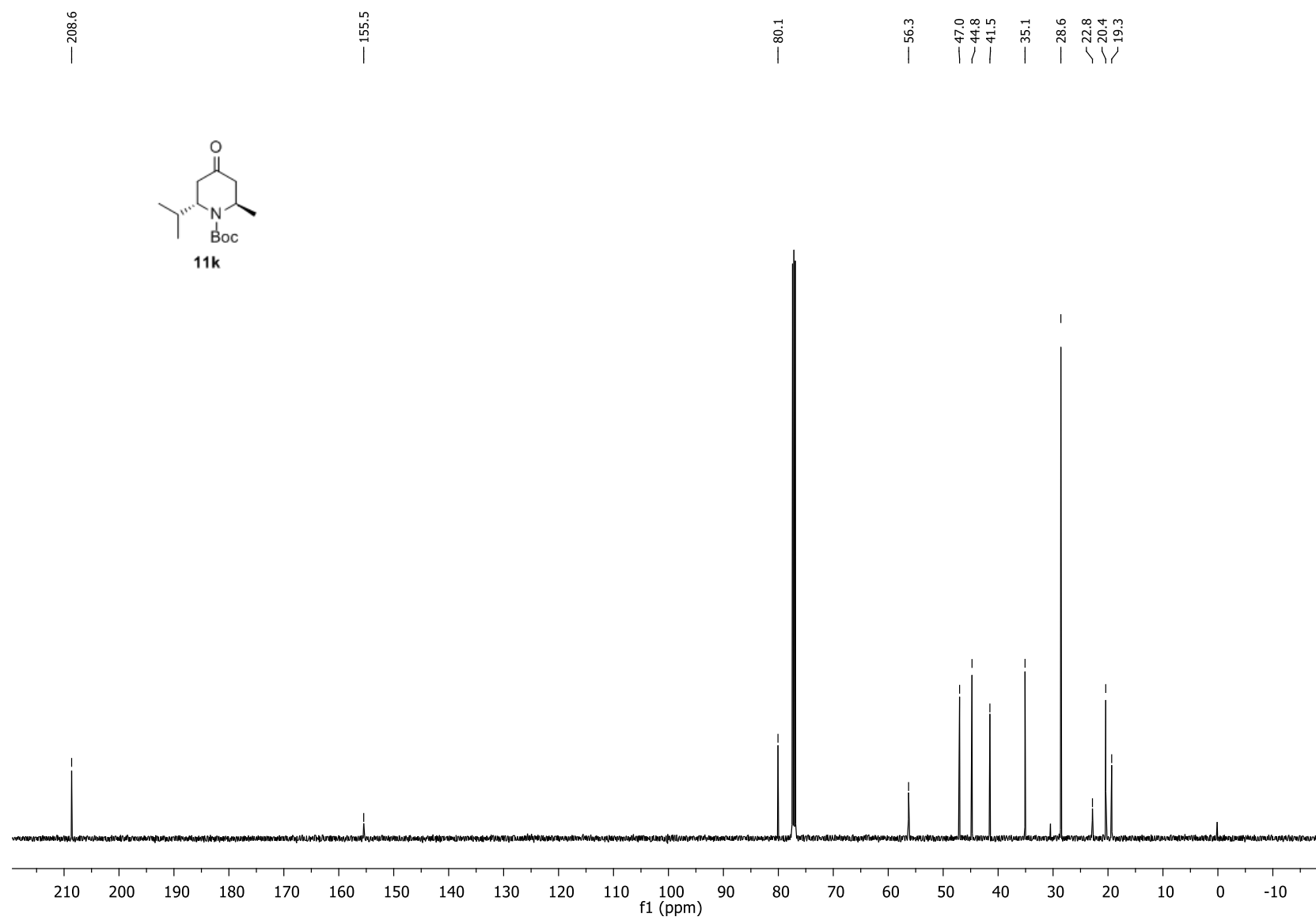


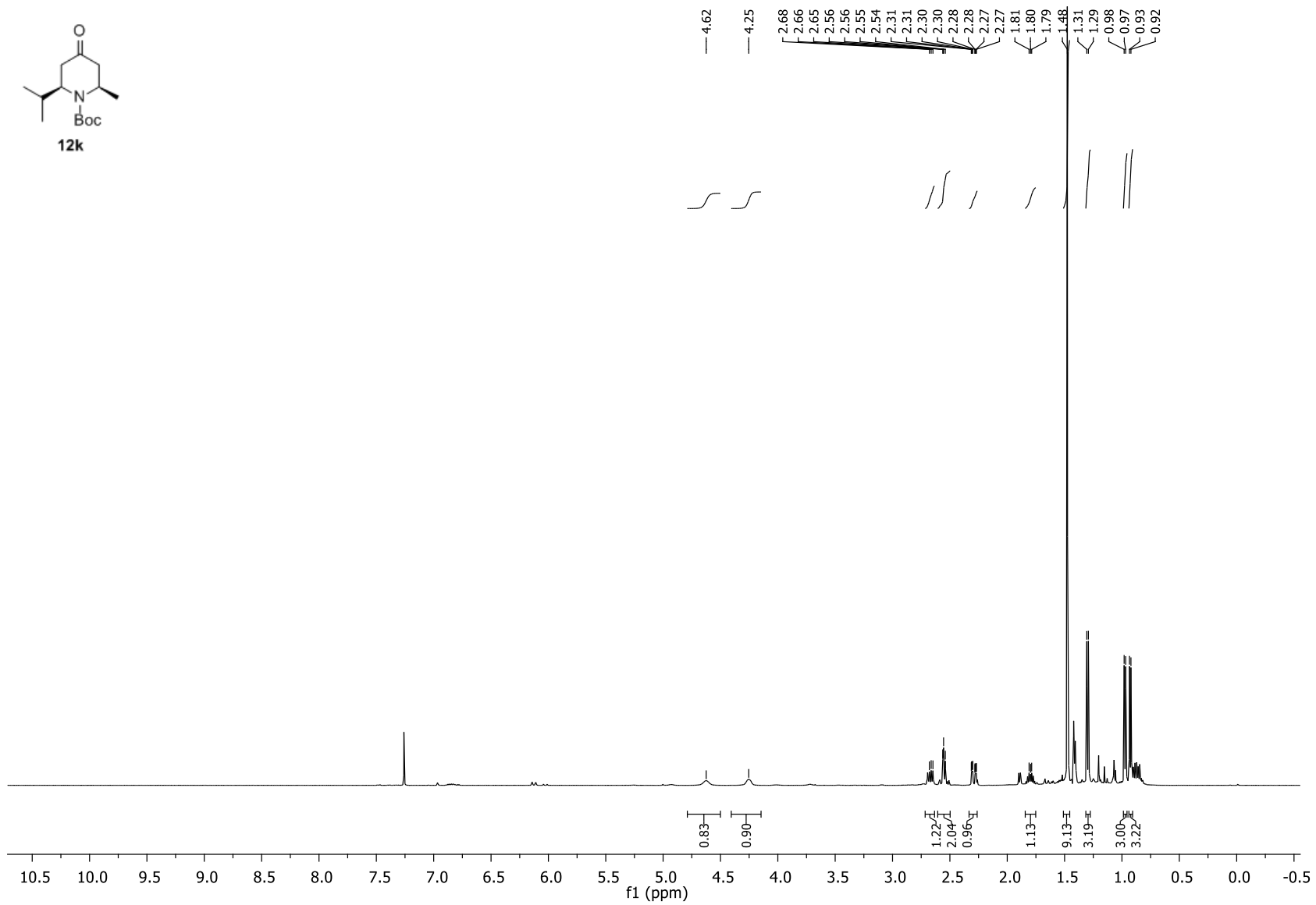
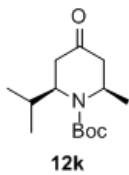




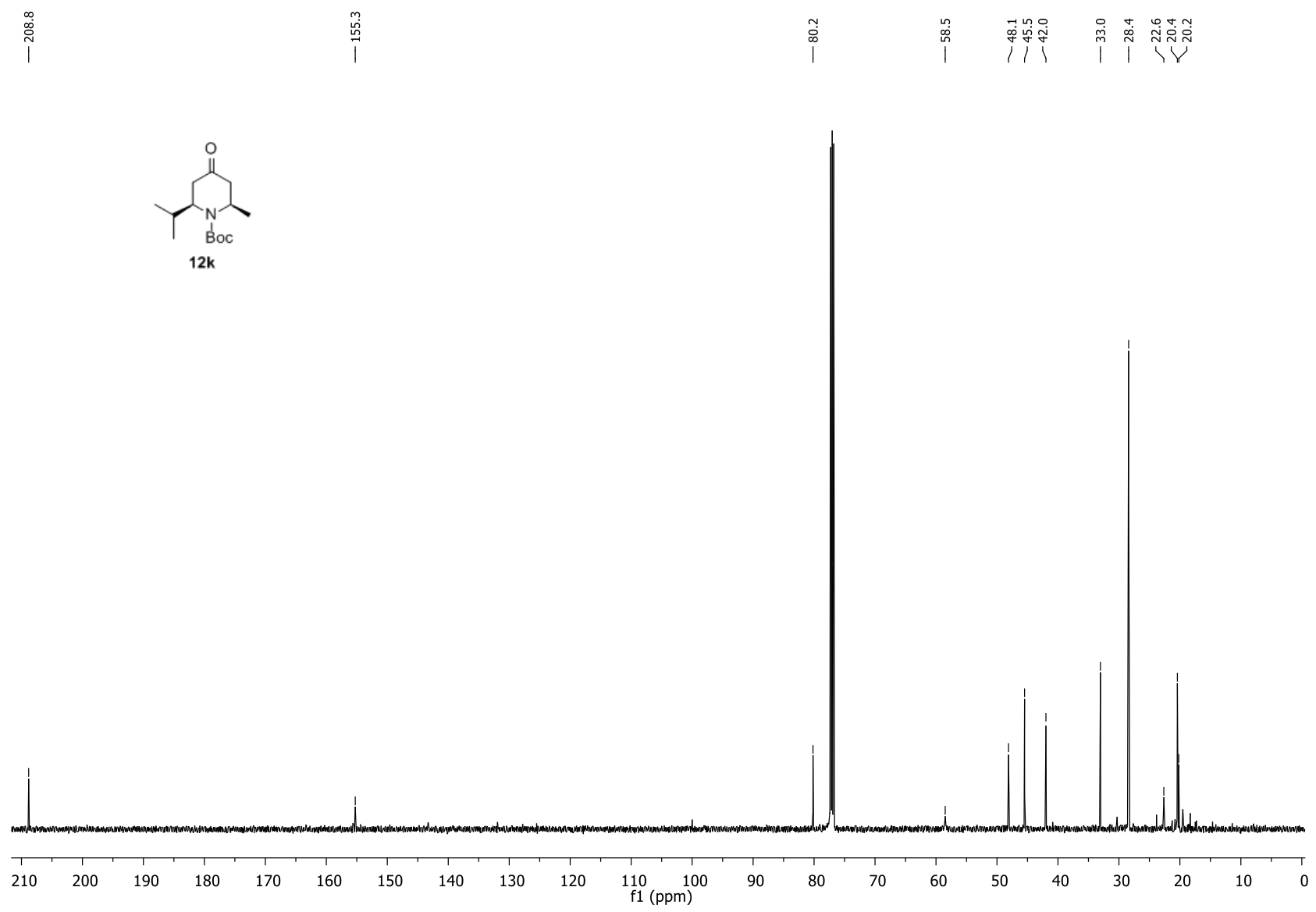


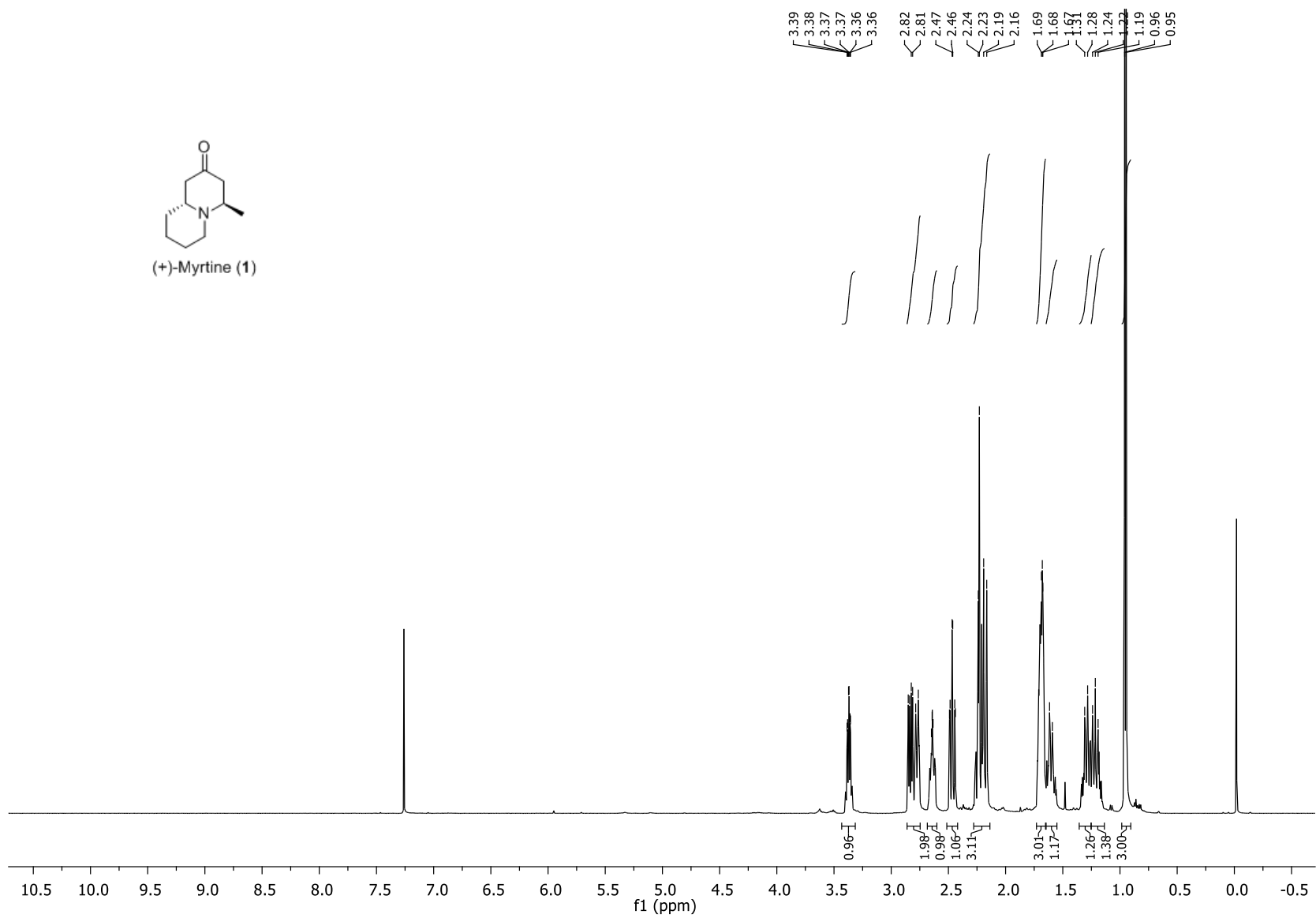
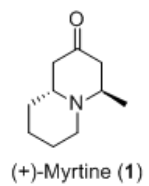




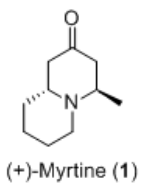








— 209.7



57.2  
53.6  
51.5  
48.8  
48.2

— 34.4

26.0  
23.5

— 11.2

