

# Supporting Information

## Palladium-Catalyzed Suzuki–Miyaura Cross-Coupling Reactions Between Sulfamates and Potassium Boc- Protected Aminomethyltrifluoroborates

Gary A. Molander\* and Inji Shin

*Roy and Diana A. Vagelos Laboratories, Department of Chemistry, University of  
Pennsylvania, Philadelphia, Pennsylvania 19104-6323*

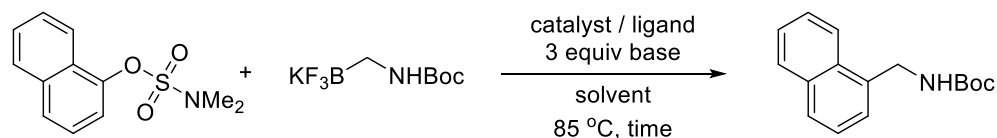
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## **General Considerations.**

Buchwald's second generation preformed catalyast (XPhos-Pd-G2) was synthesized prior to use.<sup>a</sup> *t*-Butanaol and H<sub>2</sub>O were degassed prior to use. Melting points (°C) are uncorrected. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded at 500 and 125.8 MHz, respectively. Analytical thin layer chromatography (TLC) was performed on TLC silica gel plates (250 μm) precoated with a fluorescent indicator. Standard flash column chromatography procedures<sup>b</sup> were followed using 32–63 μm silica gel. Visualization was effected with ultraviolet light and ninhydrin solution.

## Optimization of Cross-Coupling Reactions



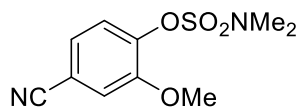
entry	cat./ligand (mol%)	base	solvent	M	time(h)	% yield <sup>a</sup> ( <sup>b</sup> )
1 <sup>c</sup>	PdCl <sub>2</sub> (cod) / RuPhos (5)	K <sub>3</sub> PO <sub>4</sub>	<i>t</i> -BuOH/H <sub>2</sub> O (1/1)	0.1	22	67(67)
2 <sup>c</sup>	biphenyl-Pd-XPhos (5)	K <sub>3</sub> PO <sub>4</sub>	<i>t</i> -BuOH/H <sub>2</sub> O (1/1)	0.1	22	88
3	biphenyl-Pd-XPhos (5)	K <sub>3</sub> PO <sub>4</sub>	<i>t</i> -BuOH/H <sub>2</sub> O (1/1)	0.25	22	94
4	biphenyl-Pd-XPhos (5)	K <sub>3</sub> PO <sub>4</sub>	<i>t</i> -BuOH/H <sub>2</sub> O (4/1)	0.25	22	89
5	biphenyl-Pd-XPhos (5)	K <sub>3</sub> PO <sub>4</sub>	<i>t</i> -BuOH/H <sub>2</sub> O (1/1)	0.5	22	92
6	biphenyl-Pd-XPhos (5)	Cs <sub>2</sub> CO <sub>3</sub>	<i>t</i> -BuOH/H <sub>2</sub> O (1/1)	0.5	22	96
7	biphenyl-Pd-XPhos (5)	Cs <sub>2</sub> CO <sub>3</sub>	toluene/H <sub>2</sub> O (4/1)	0.5	22	33
8	biphenyl-Pd-XPhos (5)	K <sub>2</sub> CO <sub>3</sub>	<i>t</i> -BuOH/H <sub>2</sub> O (1/1)	0.5	22	98
9	biphenyl-Pd-XPhos (5)	K <sub>2</sub> CO <sub>3</sub>	<i>t</i> -BuOH/H <sub>2</sub> O (1/1)	0.5	6	94
10	biphenyl-Pd-XPhos (5)	K <sub>2</sub> CO <sub>3</sub>	<i>t</i> -BuOH/H <sub>2</sub> O (1/1)	0.5	3	97
11	biphenyl-Pd-XPhos (4)	K <sub>2</sub> CO <sub>3</sub>	<i>t</i> -BuOH/H <sub>2</sub> O (1/1)	0.5	22	94
12	biphenyl-Pd-XPhos (4)	K <sub>2</sub> CO <sub>3</sub>	<i>t</i> -BuOH/H <sub>2</sub> O (1/1)	0.5	3	94(93/92)
13	biphenyl-Pd-XPhos (4)	K <sub>2</sub> CO <sub>3</sub>	<i>t</i> -BuOH/H <sub>2</sub> O (1/1)	0.5	2	91
14	biphenyl-Pd-XPhos (3)	K <sub>2</sub> CO <sub>3</sub>	<i>t</i> -BuOH/H <sub>2</sub> O (1/1)	0.5	22	85

<sup>a</sup> calculated by crude <sup>1</sup>H NMR

<sup>b</sup> isolated yield

<sup>c</sup> 7 equiv base

## General Procedure A for Synthesis of Sulfamates.



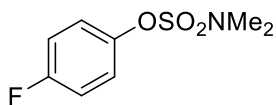
### 4-Cyano-2-methoxyphenyl Dimethylsulfamate.

A round bottomed flask was charged with 4-hydroxy-3-methoxybenzonitrile (300 mg, 1.0 equiv) and DMAP (12 mg, 0.10 mmol, 0.05 equiv) in CH<sub>2</sub>Cl<sub>2</sub> (7 mL). Et<sub>3</sub>N (244 mg, 2.41 mmol, 1.2 equiv) was slowly added to reaction flask at rt and the reaction mixture was stirred for 10 min. Dimethylsulfamoyl chloride (342 mg, 2.41 mmol, 1.2 equiv) was slowly added to reaction flask at rt, then the reaction was stirred at rt for 16 h. The reaction mixture was extracted with CH<sub>2</sub>Cl<sub>2</sub> (10 mL) and H<sub>2</sub>O (8 mL). The organic layer was washed with 1 M KOH (5 mL), then washed with H<sub>2</sub>O (10 mL). The combined

aqueous layers were extracted with CH<sub>2</sub>Cl<sub>2</sub> (10 mL). All organic layers were combined, washed with brine (5 mL), and dried (MgSO<sub>4</sub>). The crude mixture was concentrated under vacuo and purified by column chromatography (hexanes/EtOAc = 3:1) to afford the product (494 mg, 2.01 mmol) as a white solid in quantitative yield.

mp: 64–66 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.46 (d, *J* = 8.5 Hz, 1H), 7.29 (dd, *J* = 8.5, 2.0 Hz, 1H), 7.23 (d, *J* = 2.0 Hz, 1H), 3.93 (s, 3H), 3.01 (s, 6H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 152.1, 142.9, 125.5, 124.4, 118.1, 116.1, 111.1, 56.5, 38.8; IR (neat) 2231, 1380, 1169, 838, 759 cm<sup>-1</sup>; HRMS (ES<sup>+</sup>) calcd. for C<sub>10</sub>H<sub>13</sub>N<sub>2</sub>O<sub>4</sub>S [M+H]<sup>+</sup> 257.0596, found 257.0593.

#### General Procedure B for Synthesis of Sulfamates.

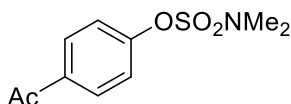


#### 4-Fluorophenyl Dimethylsulfamate.

A round bottomed flask was charged with NaH (60% in mineral oil, 107 mg, 1.2 equiv) and the flask was cooled to 0 °C. 4-fluorophenol (250 mg, 2.23 mmol, 1 equiv) in DME (7 mL) was slowly added to reaction flask at 0 °C. The reaction mixture was warmed to rt for 10 min, then cooled to 0 °C again. Dimethylsulfamoyl chloride (320 mg, 2.23 mmol, 1 equiv) was slowly added to reaction flask at 0 °C and stirred at rt for 16 h. The reaction was quenched by addition of H<sub>2</sub>O (5 mL). The crude mixture was extracted with Et<sub>2</sub>O (15 mL). The organic layer was washed with 1 M KOH (5 mL), then washed with H<sub>2</sub>O (10 mL). The combined aqueous layers were extracted with Et<sub>2</sub>O (10 mL). All organic layers were combined, washed with brine (5 mL), and dried (MgSO<sub>4</sub>). The crude mixture was

concentrated under vacuo and purified by column chromatography (hexanes/EtOAc = 4:1) to afford the product (485 mg, 2.21 mmol) as a white solid in 99% yield.

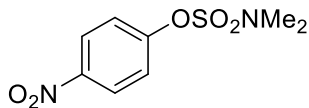
mp: 52–55 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.26 (dd,  $J = 8.5, 4.0$  Hz, 2H), 7.07 (dd,  $J = 8.5, 8.5$  Hz, 2H), 2.98 (s, 6H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  160.9 (d,  $J = 246.1$  Hz), 146.1 (d,  $J = 2.6$  Hz), 125.1 (d,  $J = 8.4$  Hz), 116.6 (d,  $J = 23.6$  Hz), 38.9; IR (neat) 2923, 1498, 1360, 1185, 844, 797  $\text{cm}^{-1}$ ; HRMS (CI+) calcd. for  $\text{C}_8\text{H}_{11}\text{NO}_3\text{SF}$   $[\text{M}+\text{H}]^+$  220.0444, found 220.0439.



#### 4-Acetylphenyl Dimethylsulfamate.

According to General Procedure B for Synthesis of Sulfamates, the desired product was obtained as a white solid in 70% isolated yield after column chromatography (hexanes/EtOAc = 3:1).

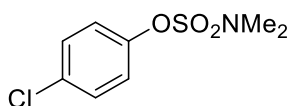
mp: 65–67 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.01 (d,  $J = 9.0$  Hz, 2H), 7.37 (d,  $J = 8.5$  Hz, 2H), 3.01 (s, 6H), 2.61 (s, 3H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  196.8, 154.0, 135.4, 130.4, 121.7, 38.9, 26.8; IR (neat) 1690, 1361, 1171, 848, 763  $\text{cm}^{-1}$ ; HRMS (ES+) calcd. for  $\text{C}_{10}\text{H}_{14}\text{NO}_4\text{S}$   $[\text{M}+\text{H}]^+$  244.0644, found 244.0644.



#### 4-Nitrophenyl Dimethylsulfamate.

According to General Procedure A for Synthesis of Sulfamates, the desired product was obtained as a white solid in 90% isolated yield after column chromatography (hexanes/EtOAc = 4:1).

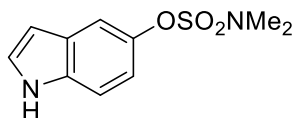
mp: 120–123 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.29 (d,  $J = 9.5$  Hz, 2H), 7.45 (d,  $J = 9.5$  Hz, 2H), 3.05 (s, 6H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  155.0, 145.8, 125.7, 122.2, 38.9; IR (neat) 2980, 1521, 1361, 1172, 1146, 862, 748  $\text{cm}^{-1}$ ; HRMS (CI+) calcd. for  $\text{C}_8\text{H}_{11}\text{N}_2\text{O}_5\text{S}$   $[\text{M}+\text{H}]^+$  247.0389, found 247.0388.



#### 4-Chlorophenyl Dimethylsulfamate **3**.

According to General Procedure B for Synthesis of Sulfamates, the desired product **3** was obtained as a light yellow oil in quantitative yield after column chromatography (hexanes/EtOAc = 7:1).

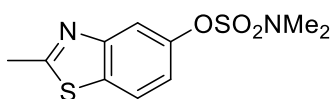
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.36 (d,  $J = 9.0$  Hz, 2H), 7.23 (d,  $J = 9.0$  Hz, 2H), 2.98 (s, 6H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  148.8, 132.4, 130.0, 123.3, 38.9; IR (neat) 1484, 1371, 1171, 856, 754  $\text{cm}^{-1}$ ; HRMS (ES+) calcd. for  $\text{C}_8\text{H}_{11}\text{NO}_3\text{SCl}$   $[\text{M}+\text{H}]^+$  236.0148, found 236.0149.



#### 1H-Indol-5-yl Dimethylsulfamate.

According to General Procedure B for Synthesis of Sulfamates, the desired product was obtained as a white solid in 80% isolated yield after column chromatography (hexanes/EtOAc = 3:1).

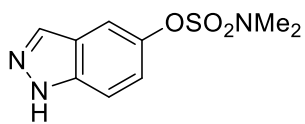
mp: 77–80 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.28 (br, 1H), 7.54 (s, 1H), 7.33 (d,  $J$  = 8.5 Hz, 1H), 7.26–7.23 (m, 1H), 7.13–7.06 (m, 1H), 6.54 (s, 1H), 2.96 (s, 6H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  144.1, 134.3, 128.3, 126.1, 116.4, 113.5, 111.8, 103.3, 38.9; IR (neat) 3384, 1350, 1169, 843  $\text{cm}^{-1}$ ; HRMS (ES $^-$ ) calcd. for  $\text{C}_{10}\text{H}_{11}\text{N}_2\text{O}_3\text{S}$   $[\text{M}-\text{H}]^-$  239.0490, found 239.0503.



### **2-Methylbenzo[d]thiazol-5-yl Dimethylsulfamate.**

According to General Procedure B for Synthesis of Sulfamates, stirred for 18 h at 70 °C instead of rt, the desired product was obtained as a white solid in 89% isolated yield after column chromatography (hexanes/EtOAc = 3:1 to 2:1).

mp: 95–97 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.84 (d,  $J$  = 2.5 Hz, 1H), 7.81 (d,  $J$  = 8.5 Hz, 1H), 7.34 (dd,  $J$  = 9.0, 2.5 Hz, 1H), 3.00 (s, 6H), 2.85 (s, 3H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  169.6, 154.2, 148.9, 134.1, 122.2, 119.3, 115.7, 38.9, 20.4; IR (neat) 1365, 1176, 815, 750  $\text{cm}^{-1}$ ; HRMS (CI $^+$ ) calcd. for  $\text{C}_{10}\text{H}_{13}\text{N}_2\text{O}_3\text{S}_2$   $[\text{M}+\text{H}]^+$  273.0368, found 273.0361.



### **1H-Indazol-5-yl Dimethylsulfamate.**

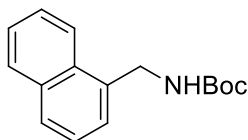
According to General Procedure B for Synthesis of Sulfamates, stirred for 18 h at 70 °C instead of rt, the desired product was obtained as a white solid in 32% isolated yield after column chromatography (hexanes/EtOAc = 1:1).

mp: 143–145 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 10.11 (br, 1H), 8.10 (s, 1H), 7.65 (d, *J* = 2.0 Hz, 1H), 7.51 (d, *J* = 9.0 Hz, 1H), 7.35 (dd, *J* = 9.0 2.5 Hz, 1H), 3.01 (s, 6H); <sup>13</sup>C NMR (125.8 MHz, CD<sub>2</sub>Cl<sub>2</sub>) δ 144.7, 138.8, 135.2, 123.5, 122.1, 113.6, 111.2, 39.0; IR (neat) 3152, 1502, 1360, 1170, 838 cm<sup>-1</sup>; HRMS (ES<sup>+</sup>) calcd. for C<sub>9</sub>H<sub>12</sub>N<sub>3</sub>O<sub>3</sub>S [M+H]<sup>+</sup> 242.0599, found 242.0591.

#### **General Procedure for the Suzuki–Miyaura Cross-coupling Reaction.**

A microwave vial was charged with potassium *tert*-butoxycarbonyl aminomethyltrifluoroborates (primary: 62 mg, 0.263 mmol, 1.05 equiv, or secondary: 77 mg, 0.263 mmol, 1.05 equiv), aryl or heteroaryl sulfamates (0.250 mmol, 1.0 equiv), XPhos-Pd-G2 (8 mg, 0.010 mmol, 0.04 equiv), and K<sub>2</sub>CO<sub>3</sub> (3.0, 5.0, or 7.0 equiv). The vial was capped, and then the mixture was degassed under vacuum and purged with argon. This procedure was repeated three times. *t*-BuOH/H<sub>2</sub>O (0.5 M, 1:1, 0.25 mL/0.25 mL) was then added to the reaction vial. The reaction mixture was stirred for 3 h or 18 h at 85 °C and then cooled to rt. H<sub>2</sub>O (2 mL) was added, and the resulting mixture was extracted with EtOAc (2 × 3 mL). The organic layer was combined, dried (MgSO<sub>4</sub>) and filtered. The solvent was removed in vacuo and the product was purified by column chromatography.



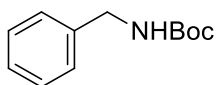


***tert*-Butyl (Naphthalen-1-ylmethyl)carbamate 2a.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **2a** was obtained as a white solid in 93% isolated yield after column chromatography (hexanes/EtOAc = 7:1).

mp: 95–98 °C (lit.: 99–100 °C); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.04 (d, *J* = 8.0 Hz, 1H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.80–7.75 (m, 1H), 7.56–7.46 (m, 2H), 7.44–7.37 (m, 2H), 4.85 (br, 1H), 4.76 (d, *J* = 5.0 Hz, 2H), 1.47 (s, 9H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 155.8, 134.3, 134.0, 131.5, 128.9, 128.9, 126.6, 126.2, 126.0, 125.5, 123.6, 79.6, 42.9, 28.5.

Data is consistent with that reported in the literature.<sup>c</sup>

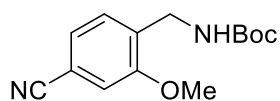


***tert*-Butyl Benzylcarbamate 2b.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **2b** was obtained as a white solid in 85% isolated yield after column chromatography (CH<sub>2</sub>Cl<sub>2</sub>/hexanes = 9:1).

mp: 54–56 °C (lit.: 57 °C); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.36–7.23 (m, 5H), 4.86 (br, 1H), 4.31 (d, *J* = 4.0 Hz, 2H), 1.46 (s, 9H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 156.0, 139.1, 128.7, 127.6, 127.4, 79.6, 44.8, 28.5.

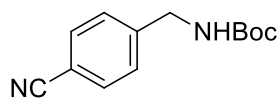
Data is consistent with that reported in the literature.<sup>d</sup>



***tert*-Butyl (4-Cyano-2-methoxybenzyl)carbamate 2f.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **2f** was obtained as a white solid in 90% isolated yield after column chromatography (hexanes/EtOAc = 4:1).

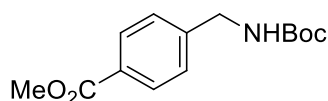
mp: 100–102 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.36 (d, *J* = 7.0 Hz, 1H), 7.24 (d, *J* = 7.5 Hz, 1H), 7.07 (s, 1H), 5.08 (br, 1H), 4.32 (d, *J* = 5.5 Hz, 2H), 3.88 (s, 3H), 1.44 (s, 9H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 157.4, 155.9, 133.2, 129.3, 125.0, 118.9, 113.1, 111.9, 79.8, 55.8, 40.0, 28.5; IR (neat) 3363, 3323, 2982, 2227, 1696, 1504, 1282, 1152 cm<sup>-1</sup>; HRMS (ES<sup>+</sup>) calcd. for C<sub>14</sub>H<sub>19</sub>N<sub>2</sub>O<sub>3</sub> [M+H]<sup>+</sup> 263.1396, found 263.1392.



***tert*-Butyl 4-Cyanobenzylcarbamate 2g.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **2g** was obtained as a white solid in 88% isolated yield after column chromatography (hexanes/EtOAc = 4:1).

mp: 106–109 °C (lit.: 111–113 °C); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.62 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 5.04 (br, 1H), 4.37 (d, *J* = 5.5 Hz, 2H), 1.46 (s, 9H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 156.0, 144.8, 132.5, 127.9, 118.9, 111.2, 80.1, 44.3, 28.5. Data is consistent with that reported in the literature.<sup>e</sup>

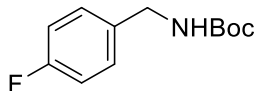


**Methyl 4-[(*tert*-Butoxycarbonyl)amino]methyl]benzoate **2h**.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **2h** was obtained as a white solid in 93% isolated yield after column chromatography (hexanes/EtOAc = 4:1).

mp: 86–89 °C (lit.: 88–90 °C); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.99 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 4.97 (br, 1H), 4.37 (d, *J* = 5.0 Hz, 2H), 3.91 (s, 3H), 1.47 (s, 9H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 167.0, 156.0, 144.4, 130.0, 129.3, 127.3, 79.9, 52.2, 44.5, 28.5.

Data is consistent with that reported in the literature.<sup>f</sup>

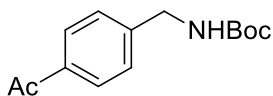


***tert*-Butyl (4-Fluorobenzyl)carbamate **2i**.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, *n*-PrOH instead of *t*-BuOH, the desired product **2i** was obtained as a white solid in 89% isolated yield after column chromatography (hexanes/EtOAc = 10:1).

mp: 64–66 °C (lit.: 68–70 °C); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.27–7.21 (m, 2H), 7.03–6.97 (m, 2H), 4.84 (br, 1H), 4.27 (d, *J* = 5.0 Hz, 2H), 1.46 (s, 9H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 162.3 (d, *J* = 245.0 Hz), 156.0, 134.9, 129.2 (d, *J* = 7.5 Hz), 115.5 (d, *J* = 21.4 Hz), 79.8, 44.2, 28.5.

Data is consistent with that reported in the literature.<sup>c</sup>

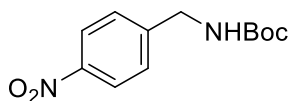


***tert*-Butyl 4-Acetylbenzylcarbamate **2j**.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **2j** was obtained as a white solid in 87% isolated yield after column chromatography (hexanes/EtOAc = 3:1).

mp: 69–71 °C (lit.: 67–69 °C); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.92 (d, *J* = 7.5 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 5.04 (br, 1H), 4.37 (d, *J* = 5.0 Hz, 2H), 2.59 (s, 3H), 1.46 (s, 9H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 197.9, 156.0, 144.7, 136.3, 128.8, 127.4, 79.9, 44.4, 28.5, 26.7.

Data is consistent with that reported in the literature.<sup>g</sup>

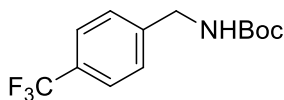


***tert*-Butyl (4-Nitrobenzyl)carbamate **2k**.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **2k** was obtained as a yellow solid in 60% isolated yield after column chromatography (hexanes/EtOAc = 10:1).

mp: 107–109 °C (lit.: 109–110 °C); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.19 (d, *J* = 8.5 Hz, 2H), 7.45 (d, *J* = 8.0 Hz, 2H), 5.08 (br, 1H), 4.42 (d, *J* = 5.5 Hz, 2H), 1.47 (s, 9H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 156.0, 147.3, 146.8, 127.9, 123.9, 80.3, 44.1, 28.5.

Data is consistent with that reported in the literature.<sup>h</sup>

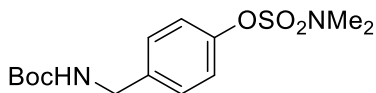


***tert*-Butyl 4-(Trifluoromethyl)benzylcarbamate **21**.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **21** was obtained as a white solid in 76% isolated yield after column chromatography (hexanes/EtOAc = 6:1).

mp: 70–72 °C (lit.: 70–71 °C); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.58 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 5.03 (br, 1H), 4.36 (d, *J* = 6.0 Hz, 2H), 1.46 (s, 9H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 156.0, 143.3, 129.7 (q, *J* = 32.5 Hz), 127.6, 125.6 (q, *J* = 3.5 Hz), 124.3 (q, *J* = 271.9 Hz), 80.0, 44.3, 28.5.

Data is consistent with that reported in the literature.<sup>f</sup>



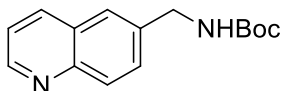
**4-[(*tert*-Butoxycarbonyl)amino]methyl phenyl Dimethylsulfamate **4**.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **4** was obtained as a white solid in 77% isolated yield after column chromatography (hexanes/EtOAc = 3:1).

When the solvents were toluene/H<sub>2</sub>O (0.5 M, 4:1, 0.4 mL/0.1 mL), the desired product **4** was obtained as a white solid in 81% isolated yield after column chromatography (hexanes/EtOAc = 3:1).

mp: 94–96 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.30 (d, *J* = 8.5 Hz, 2H), 7.23 (d, *J* = 8.5 Hz, 2H), 4.93 (br, 1H), 4.30 (d, *J* = 5.0 Hz, 2H), 2.96 (s, 6H), 1.45 (s, 9H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 156.0, 149.5, 137.9, 128.9, 122.0, 79.8, 44.1, 38.8, 28.5; IR (neat)

3328, 2976, 1676, 1356, 1170, 1149  $\text{cm}^{-1}$ ; HRMS (ES+) calcd. for  $\text{C}_{14}\text{H}_{22}\text{N}_2\text{O}_5\text{SNa}$   $[\text{M}+\text{Na}]^+$  353.1147, found 353.1131.

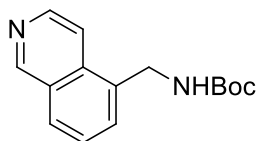


***tert*-Butyl (Quinolin-6-ylmethyl)carbamate 5a.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **5a** was obtained as a white solid in 88% isolated yield after column chromatography (hexanes/EtOAc = 1:1).

mp: 78–81 °C (lit.: 75–77 °C);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.90–8.85 (m, 1H), 8.09 (d,  $J = 8.0$  Hz, 1H), 8.05 (d,  $J = 8.5$  Hz, 1H), 7.67 (s, 1H), 7.63 (d,  $J = 8.5$  Hz, 1H), 7.37 (dd,  $J = 8.0, 4.0$  Hz, 1H), 5.26 (br, 1H), 4.49 (d,  $J = 5.5$  Hz, 2H), 1.48 (s, 9H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  156.1, 150.3, 147.8, 137.5, 135.9, 129.9, 129.3, 128.2, 125.7, 121.4, 79.8, 44.6, 28.5.

Data is consistent with that reported in the literature.<sup>g</sup>



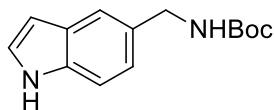
***tert*-Butyl (Isoquinolin-5-ylmethyl)carbamate 5b.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **5b** was obtained as a white solid in 91% isolated yield after column chromatography (hexanes/EtOAc = 1:1).

mp: 155–158 °C (lit.: 155–160 °C);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  9.22 (s, 1H), 8.54 (d,  $J = 5.5$  Hz, 1H), 7.89 (d,  $J = 8.0$  Hz, 1H), 7.83 (d,  $J = 5.5$  Hz, 1H), 7.65 (d,  $J = 7.0$  Hz,

1H), 7.54 (dd,  $J = 7.5, 7.5$  Hz, 1H), 5.17 (br, 1H), 4.75 (d,  $J = 5.5$  Hz, 2H), 1.47 (s, 9H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  155.8, 153.1, 143.5, 134.2, 133.9, 129.9, 128.9, 127.7, 126.9, 116.5, 79.8, 42.0, 28.5.

Data is consistent with that reported in the literature.<sup>f</sup>

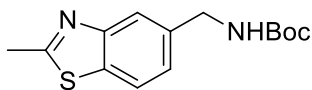


***tert*-Butyl {(1H-Indol-5-yl)methyl}carbamate 5c.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **5c** was obtained as a brown solid in 68% isolated yield after column chromatography ( $\text{CH}_2\text{Cl}_2/\text{hexanes} = 40:1$ ).

mp: 86–89 °C (lit.: 86–89 °C);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.31 (br, 1H), 7.54 (s, 1H), 7.34 (d,  $J = 8.0$  Hz, 1H), 7.20 (dd,  $J = 2.5, 2.5$  Hz, 1H), 7.12 (d,  $J = 8.0$  Hz, 1H), 6.53–6.49 (m, 1H), 4.84 (br, 1H), 4.40 (d,  $J = 5.5$  Hz, 2H), 1.47 (s, 9H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  156.0, 135.3, 130.2, 128.1, 124.9, 122.3, 119.9, 111.4, 102.6, 79.4, 45.4, 28.6.

Data is consistent with that reported in the literature.<sup>g</sup>

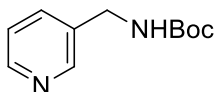


***tert*-Butyl {(2-Methylbenzo[d]thiazol-5-yl)methyl}carbamate 5d.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **5d** was obtained as a white solid in 85% isolated yield after column chromatography ( $\text{CH}_2\text{Cl}_2/\text{EtOAc} = 6:1$  to  $4:1$ ).

mp: 93–95 °C (lit.: 93–95 °C); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.82 (s, 1H), 7.74 (d, *J* = 8.0 Hz, 1H), 7.28 (d, *J* = 7.5 Hz, 1H), 5.10 (br, 1H), 4.43 (d, *J* = 5.5 Hz, 2H), 2.82 (s, 3H), 1.46 (s, 9H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 167.6, 156.0, 153.7, 137.5, 134.5, 124.4, 121.5, 121.0, 79.6, 44.5, 28.5, 20.2.

Data is consistent with that reported in the literature.<sup>f</sup>

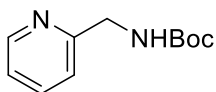


***tert*-Butyl (Pyridin-3-ylmethyl)carbamate 5e.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **5e** was obtained as a colorless oil in 83% isolated yield after column chromatography (CH<sub>2</sub>Cl<sub>2</sub>/EtOAc = 2:1).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.52 (s, 1H), 8.50 (d, *J* = 4.5 Hz, 1H), 7.63 (d, *J* = 7.5 Hz, 1H), 3.84 (dd, *J* = 7.5, 5.0 Hz, 1H), 5.17 (br, 1H), 4.32 (d, *J* = 4.0 Hz, 2H), 1.45 (s, 9H); <sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) δ 156.0, 149.1, 148.8, 135.3, 134.7, 123.6, 79.9, 42.3, 28.5.

Data is consistent with that reported in the literature.<sup>i</sup>



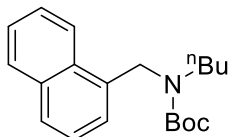
***tert*-Butyl (Pyridin-2-ylmethyl)carbamate 5f.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **5f** was obtained as a colorless oil in 58% isolated yield after column chromatography (hexanes/EtOAc = 1:1).



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.53 (d,  $J = 4.5$  Hz, 1H), 7.68–7.62 (m, 1H), 7.28 (d,  $J = 7.5$  Hz, 1H), 7.20–7.17 (m, 1H), 5.65 (br, 1H), 4.45 (d,  $J = 5.0$  Hz, 2H), 1.47 (s, 9H);  $^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  157.6, 156.1, 149.2, 136.8, 122.3, 121.8, 79.6, 45.9, 28.5.

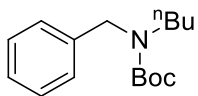
Data is consistent with that reported in the literature.<sup>g</sup>



***tert*-Butyl Butyl(naphthalen-1-ylmethyl)carbamate **7a**.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **7a** was obtained as a colorless oil in 92% isolated yield after column chromatography (hexanes/EtOAc = 20:1).

$^1\text{H}$  NMR (asterisk denotes minor rotamer peaks, 500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.18–7.98 (m, 1H), 7.89–7.81 (m, 1H), 7.77 (d,  $J = 8.0$  Hz, 1H), 7.54–7.46 (m, 2H), 7.46–7.39 (m, 1H), 7.34–7.28 (m, 1H), 5.00–4.89 (m, 2H), 3.22\* (s, 2H), 3.07 (s, 2H), 1.57–1.36 (m, 11H), 1.29–1.18 (m, 2H), 0.88–0.82 (m, 3H);  $^{13}\text{C}$  NMR (asterisk denotes minor rotamer peaks, 125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  155.9, 155.7\*, 133.9, 133.7, 131.9, 131.4\*, 128.7, 128.2, 127.8\*, 126.3\*, 126.2, 125.8, 125.3, 124.5\*, 124.0, 123.1, 79.6, 48.2\*, 47.7, 46.3\*, 45.4, 30.1, 28.6, 20.1, 13.9; IR (neat) 2962, 1689, 1415, 1171, 1142  $\text{cm}^{-1}$ ; HRMS (ES+) calcd. for  $\text{C}_{20}\text{H}_{28}\text{NO}_2$   $[\text{M}+\text{H}]^+$  314.2120, found 314.2122.

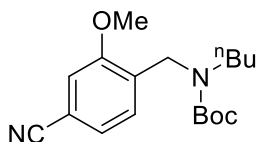


***tert*-Butyl Benzyl(butyl)carbamate **7b**.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **7b** was obtained as a colorless oil in 33% isolated yield after column chromatography (hexanes/EtOAc = 20:1).

$^1\text{H}$  NMR (asterisk denotes minor rotamer peaks, 500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.38–7.19 (m, 5H), 4.45 (s, 2H), 4.41\* (s, 2H), 3.23–3.10 (m, 2H), 1.59–1.397 (m, 11H), 1.35–1.22 (m, 2H), 0.89 (t,  $J = 7.0$  Hz, 3H);  $^{13}\text{C}$  NMR (asterisk denotes minor rotamer peaks, 125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  156.3, 155.8\*, 139.0\*, 138.7, 128.5, 127.8, 127.1, 79.6, 50.5\*, 49.8, 46.6\*, 46.2, 30.4\*, 30.1, 28.6, 20.1, 14.0.

Data is consistent with that reported in the literature.<sup>j</sup>

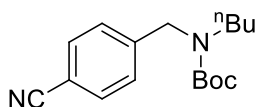


***tert*-Butyl Butyl(4-cyano-2-methoxybenzyl)carbamate **7d**.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **7d** was obtained as a white solid in 63% isolated yield after column chromatography (hexanes/EtOAc = 10:1).

mp: 63–66 °C;  $^1\text{H}$  NMR (asterisk denotes minor rotamer peaks, 500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.27–7.17 (m, 2H), 7.09–7.04 (m, 1H), 4.46 (s, 2H), 4.41\* (s, 2H), 3.86 (s, 3H), 3.28–

3.14 (m, 2H), 1.52–1.36 (m, 11), 1.36–1.24 (m, 2H), 0.91 (t,  $J = 7.5$  Hz, 3H);  $^{13}\text{C}$  NMR (asterisk denotes minor rotamer peaks, 125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  157.3, 157.0\*, 156.1, 155.7\*, 133.3\*, 133.0, 128.3, 127.8\*, 124.9, 124.8\*, 119.1, 123.0, 111.4, 79.8, 55.7, 47.4, 45.6\*, 45.0, 30.6, 30.3\*, 28.5, 28.4\*, 20.2\*, 20.0, 13.9; IR (neat) 2969, 2229, 1677, 1406, 1142  $\text{cm}^{-1}$ ; HRMS (ES+) calcd. for  $\text{C}_{18}\text{H}_{26}\text{N}_2\text{O}_3\text{Na}$   $[\text{M}+\text{Na}]^+$  341.1841, found 341.1847.

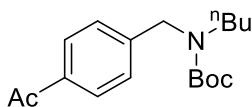


***tert*-Butyl Butyl(4-cyanobenzyl)carbamate 7e.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **7e** was obtained as a colorless oil in 53% isolated yield after column chromatography (hexanes/EtOAc = 15:1).

$^1\text{H}$  NMR (asterisk denotes minor rotamer peaks, 500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60 (d,  $J = 8.0$  Hz, 2H), 7.35–7.27 (m, 2H), 4.50–4.39 (m, 2H), 3.28–3.08 (m, 2H), 1.56–1.34 (m, 11H), 1.34–1.22 (m, 2H), 0.88 (t,  $J = 7.0$  Hz, 3H);  $^{13}\text{C}$  NMR (asterisk denotes minor rotamer peaks, 125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  156.2, 155.4\*, 144.9\*, 144.5, 132.4, 128.1, 127.5\*, 118.9, 111.0, 80.1, 50.6\*, 49.9, 47.3\*, 47.0, 30.4, 28.5, 20.0, 13.9.

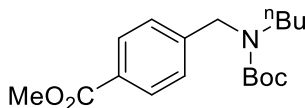
Data is consistent with that reported in the literature.<sup>k</sup>



***tert*-Butyl (4-Acetylbenzyl)(butyl)carbamate 7f.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **7f** was obtained as a colorless oil in 42% isolated yield after column chromatography (hexanes/EtOAc = 10:1).

<sup>1</sup>H NMR (asterisk denotes minor rotamer peaks, 500 MHz, CDCl<sub>3</sub>) δ 7.92 (d, *J* = 8.0 Hz, 2H), 7.31 (s, 2H), 4.49 (s, 2H), 4.45\* (s, 2H), 3.28–3.11 (m, 2H), 2.60 (s, 3H), 1.55–1.37 (m, 11H), 1.33–1.25 (m, 2H), 0.90 (t, *J* = 7.5 Hz, 3H); <sup>13</sup>C NMR (asterisk denotes minor rotamer peaks, 125.8 MHz, CDCl<sub>3</sub>) δ 197.9, 156.2, 144.7\*, 144.4, 136.2, 128.7, 127.7, 127.1\*, 79.9, 50.6\*, 49.9, 47.1\*, 46.8, 30.3, 28.5, 26.7, 20.1, 13.9; IR (neat) 2960, 2929, 1685, 1408, 1266, 1170, 1144cm<sup>-1</sup>; HRMS (ES<sup>+</sup>) calcd. for C<sub>18</sub>H<sub>27</sub>NO<sub>3</sub>Na [M+Na]<sup>+</sup> 328.1889, found 328.1886.

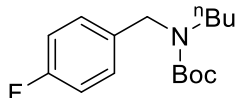


**Methyl 4-[(*tert*-Butoxycarbonyl)(butyl)amino]methyl benzoate **7g**.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **7g** was obtained as a colorless oil in 76% isolated yield after column chromatography (hexanes/EtOAc = 20:1).

<sup>1</sup>H NMR (asterisk denotes minor rotamer peaks, 500 MHz, CDCl<sub>3</sub>) δ 7.99 (d, *J* = 8.0 Hz, 2H), 7.32–7.26 (m, 2H), 4.49 (s, 2H), 4.44\* (s, 2H), 3.91 (s, 3H), 3.29–3.10 (m, 2H), 1.55–1.34 (m, 11), 1.34–1.23 (m, 2H), 0.90 (t, *J* = 7.5 Hz, 3H); <sup>13</sup>C NMR (asterisk denotes minor rotamer peaks, 125.8 MHz, CDCl<sub>3</sub>) δ 167.0, 156.2, 155.6\*, 144.5, 144.2\*, 129.9, 129.1, 127.5, 126.9\*, 79.9, 52.2, 50.6\*, 49.9, 47.1\*, 46.7, 30.4, 28.5, 20.1, 13.9;

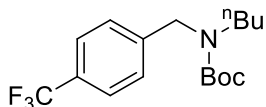
IR (neat) 2961, 1724, 1693, 1277, 1174  $\text{cm}^{-1}$ ; HRMS (ES+) calcd. for  $\text{C}_{18}\text{H}_{27}\text{NO}_4\text{Na}$   $[\text{M}+\text{Na}]^+$  344.1838, found 344.1839.



***tert*-Butyl Butyl(4-fluorobenzyl)carbamate 7h.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **7h** was obtained as a colorless oil in 47% isolated yield after column chromatography (hexanes/EtOAc = 30:1).

$^1\text{H}$  NMR (asterisk denotes minor rotamer peaks, 500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.26–7.15 (m, 2H), 7.00 (dd,  $J = 8.5, 8.5$  Hz, 2H), 4.48–4.32 (m, 2H), 3.22–3.07 (m, 2H), 1.55–1.42 (m, 11H), 1.33–1.25 (m, 2H), 0.89 (t,  $J = 7.5$  Hz, 3H);  $^{13}\text{C}$  NMR (asterisk denotes minor rotamer peaks, 125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  162.1 (d,  $J = 244.7$ ), 156.2, 155.6\*, 134.5, 129.4, 128.8\*, 115.5, 115.3\*, 79.7, 49.9\*, 49.3, 46.6\*, 46.3, 30.4, 28.6, 20.1, 14.0; IR (neat) 2961, 2929, 1692, 1509, 1410, 1222, 1171, 1143  $\text{cm}^{-1}$ ; HRMS (ES+) calcd. for  $\text{C}_{16}\text{H}_{24}\text{NO}_2\text{FNa}$   $[\text{M}+\text{Na}]^+$  304.1689, found 304.1689.

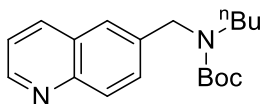


***tert*-Butyl Butyl{4-(trifluoromethyl)benzyl}carbamate 7i.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **7i** was obtained as a colorless oil in 42% isolated yield after column chromatography (hexanes/EtOAc = 20:1).

<sup>1</sup>H NMR (asterisk denotes minor rotamer peaks, 500 MHz, CDCl<sub>3</sub>) δ 7.58 (d, *J* = 8.0 Hz, 2H), 7.34 (s, 2H), 4.49 (s, 2H), 4.46\* (s, 2H), 3.24\* (s, 2H), 3.14 (s, 2H), 1.55–1.38 (m, 11H), 1.35–1.26 (m, 2H), 0.90 (t, *J* = 7.5 Hz, 3H); <sup>13</sup>C NMR (asterisk denotes minor rotamer peaks, 125.8 MHz, CDCl<sub>3</sub>) δ 156.2, 155.6\*, 143.3\*, 143.0, 129.5 (q, *J* = 32.3 Hz), 127.8, 127.2\*, 125.5 (q, *J* = 3.5 Hz), 124.3 (q, *J* = 271.6 Hz), 80.0, 50.3\*, 49.7, 47.0\*, 46.8, 30.4, 30.3\*, 28.5, 20.1, 13.9.

Data is consistent with that reported in the literature.<sup>k</sup>



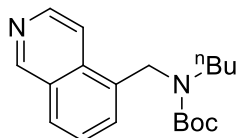
***tert*-Butyl Butyl(quinolin-6-ylmethyl)carbamate **8a**.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **8a** was obtained as a colorless oil in 84% isolated yield after column chromatography (CH<sub>2</sub>Cl<sub>2</sub>/EtOAc = 7:1).

<sup>1</sup>H NMR (asterisk denotes minor rotamer peaks, 500 MHz, CDCl<sub>3</sub>) δ 8.90 (s, 1H), 8.12 (d, *J* = 8.5 Hz, 1H), 8.08 (d, *J* = 9.0 Hz, 1H), 7.67–7.58 (m, 2H), 7.43–7.37 (m, 1H), 4.63 (s, 2H), 4.60\* (s, 2H), 3.29\* (s, 2H), 3.18 (s, 2H), 1.59–1.37 (m, 11H), 1.36–1.25 (m, 2H), 0.89 (t, *J* = 7.0 Hz, 3H); <sup>13</sup>C NMR (asterisk denotes minor rotamer peaks, 125.8 MHz,

CDCl<sub>3</sub>)  $\delta$  156.3, 155.7\*, 150.2, 147.8, 137.3, 135.9, 129.8, 129.6, 129.0\*, 128.2, 125.9, 125.1\*, 121.4, 79.8, 50.5\*, 49.8, 46.8\*, 46.4, 30.3, 28.5, 20.1, 13.9.

Data is consistent with that reported in the literature.<sup>k</sup>

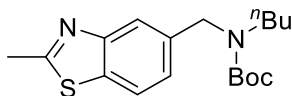


***tert*-Butyl Butyl(5-(2-(tert-butylbutylamino)methyl)isoquinolin-5-yl)methylcarbamate **8b**.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **8b** was obtained as a colorless oil in 83% isolated yield after column chromatography (hexanes/EtOAc = 5:1).

<sup>1</sup>H NMR (asterisk denotes minor rotamer peaks, 500 MHz, CDCl<sub>3</sub>)  $\delta$  9.27 (s, 1H), 8.57 (d,  $J$  = 6.0 Hz, 1H), 8.00–7.78 (m, 2H), 7.61–7.53 (m, 2H), 4.90 (s, 2H), 3.26–3.04 (m, 2H), 1.59–1.38 (m, 11H), 1.36–1.21 (m, 2H), 0.91–0.85 (m, 3H); <sup>13</sup>C NMR (asterisk denotes minor rotamer peaks, 125.8 MHz, CDCl<sub>3</sub>)  $\delta$  155.8, 153.2, 143.5, 134.6, 133.4, 130.1, 129.0, 128.6, 127.3\*, 126.7, 116.9, 116.1\*, 79.9, 47.6\*, 47.1, 46.5\*, 45.7, 30.1, 28.5, 20.1, 13.9.

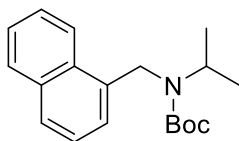
Data is consistent with that reported in the literature.<sup>k</sup>



***tert*-Butyl Butyl{(2-methylbenzo[d]thiazol-5-yl)methyl}carbamate **8c**.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **8c** was obtained as a colorless oil in 85% isolated yield after column chromatography (hexanes/EtOAc = 7:1).

<sup>1</sup>H NMR (asterisk denotes minor rotamer peaks, 500 MHz, CDCl<sub>3</sub>) δ 7.80 (s, 1H), 7.77 (d, *J* = 8.0 Hz, 1H), 7.30–7.21 (m, 1H), 4.58 (s, 2H), 4.53\* (s, 2H), 3.30–3.13 (m, 2H), 2.83 (s, 3H), 1.58–1.42 (m, 11H), 1.33–1.23 (m, 2H), 0.89 (t, *J* = 7.5 Hz, 3H); <sup>13</sup>C NMR (asterisk denotes minor rotamer peaks, 125.8 MHz, CDCl<sub>3</sub>) δ 167.4, 156.2, 155.5\*, 153.7, 137.2\*, 137.0, 134.3, 124.7, 124.1\*, 121.4, 121.1, 121.0\*, 79.6, 50.4\*, 49.7, 46.5\*, 46.2, 30.3, 30.1\*, 28.5, 20.2, 20.1\*, 20.0, 13.9; IR (neat) 2966, 2929, 1691, 1412, 1171 cm<sup>-1</sup>; HRMS (ES<sup>+</sup>) calcd. for C<sub>18</sub>H<sub>27</sub>N<sub>2</sub>O<sub>2</sub>S [M+H]<sup>+</sup> 335.1793, found 335.1782.



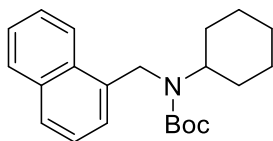
***tert*-Butyl Isopropyl(naphthalen-1-ylmethyl)carbamate 10a.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **10a** was obtained as a colorless oil in 71% isolated yield after column chromatography (hexanes/EtOAc = 25:1).

<sup>1</sup>H NMR (asterisk denotes minor rotamer peaks, 500 MHz, CDCl<sub>3</sub>) δ 8.03 (s, 1H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.54–7.46 (m, 2H), 7.43 (dd, *J* = 8.0, 8.0 Hz, 1H), 7.37–7.33 (m, 1H), 4.82 (s, 2H), 4.53 (s, 1H), 3.98\* (s, 1H), 1.62–1.23 (m, 9H), 1.17–1.08 (m, 6H); <sup>13</sup>C NMR (asterisk denotes minor rotamer peaks, 125.8 MHz, CDCl<sub>3</sub>) δ 155.9, 135.0, 133.7, 131.0, 128.9, 127.2, 126.0, 125.6, 125.4, 123.4, 122.6, 79.7, 48.5\*,



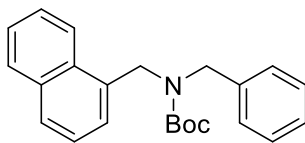
47.3, 45.7\*, 43.5, 28.5, 20.7; IR (neat) 2973, 1690, 1365, 1164  $\text{cm}^{-1}$ ; HRMS (ES+) calcd. for  $\text{C}_{19}\text{H}_{25}\text{NO}_2\text{Na}$   $[\text{M}+\text{Na}]^+$  322.1783, found 322.1786.



***tert*-Butyl Cyclohexyl(naphthalen-1-ylmethyl)carbamate 10b.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **10b** was obtained as a colorless oil in 68% isolated yield after column chromatography (hexanes/EtOAc = 25:1).

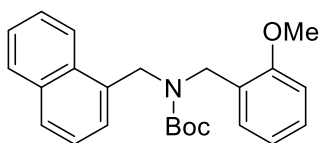
$^1\text{H}$  NMR (asterisk denotes minor rotamer peaks, 500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.00 (s, 1H), 7.86 (d,  $J = 7.5$  Hz, 1H), 7.72 (d,  $J = 8.0$  Hz, 1H), 7.54–7.46 (m, 2H), 7.42 (dd,  $J = 8.0, 8.0$  Hz, 1H), 7.36 (d,  $J = 7.0$  Hz, 1H), 4.84 (s, 2H), 4.16 (s, 1H), 3.67\* (s, 1H), 1.88–0.93 (m, 19H);  $^{13}\text{C}$  NMR (asterisk denotes minor rotamer peaks, 125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  156.0, 135.1, 133.6, 130.6, 128.9, 127.0, 125.9, 125.5, 125.3, 123.2, 122.4, 79.6, 57.2\*, 55.4, 45.4\*, 44.0, 31.0, 28.4, 26.0, 25.6; IR (neat) 2929, 1687, 1364, 1246, 1167  $\text{cm}^{-1}$ ; HRMS (ES+) calcd. for  $\text{C}_{22}\text{H}_{29}\text{NO}_2\text{Na}$   $[\text{M}+\text{Na}]^+$  362.2096, found 362.2094.



***tert*-Butyl Benzyl(naphthalen-1-ylmethyl)carbamate 10d.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **10d** was obtained as a colorless oil in 74% isolated yield after column chromatography (hexanes/EtOAc = 30:1).

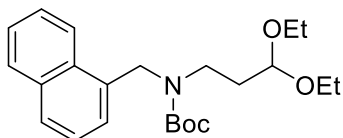
$^1\text{H}$  NMR (asterisk denotes minor rotamer peaks, 500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.17–7.94 (m, 1H), 7.88–7.83 (m, 1H), 7.78 (d,  $J = 8.5$  Hz, 1H), 7.53–7.37 (m, 3H), 7.35–7.15 (m, 6H), 4.93 (s, 2H), 4.84\* (s, 2H), 4.44\* (s, 2H), 4.28 (s, 2H), 1.52 (s, 9H);  $^{13}\text{C}$  NMR (asterisk denotes minor rotamer peaks, 125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  155.9, 138.2, 134.0, 133.1, 132.0, 131.6\*, 128.9\*, 128.7, 128.6, 128.4\*, 128.0, 127.5\*, 127.3, 126.8\*, 126.4, 126.2\*, 125.9, 125.3, 124.9, 124.1, 123.2, 80.3, 48.8, 47.1, 28.6; IR (neat) 2970, 1691, 1245, 1163  $\text{cm}^{-1}$ ; HRMS (ES+) calcd. for  $\text{C}_{23}\text{H}_{25}\text{NO}_2\text{Na}$   $[\text{M}+\text{Na}]^+$  370.1783, found 370.1784.



***tert*-Butyl (2-Methoxybenzyl)(naphthalen-1-ylmethyl)carbamate 10e.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **10e** was obtained as a light yellow oil in 82% isolated yield after column chromatography (hexanes/EtOAc =20:1).

$^1\text{H}$  NMR (asterisk denotes minor rotamer peaks, 500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.19–7.82 (m, 2H), 7.75 (d,  $J = 8.0$  Hz, 1H), 7.53–7.17 (m, 6H), 6.95–6.77 (m, 2H), 4.99 (s, 2H), 4.91\* (s, 2H), 4.53\* (s, 2H), 4.35 (s, 2H), 3.68 (s, 3H), 3.62\* (s, 3H), 1.48 (s, 9H);  $^{13}\text{C}$  NMR (asterisk denotes minor rotamer peaks, 125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  157.6\*, 157.4, 156.4, 134.0, 133.6, 132.0, 131.5\*, 128.9\*, 128.8, 128.4\*, 128.2, 128.1, 127.7\*, 126.3, 126.1, 125.9, 125.8\*, 125.6\*, 125.4, 124.3\*, 124.0, 123.4\*, 123.2, 120.7, 120.5, 110.8\*, 110.3, 80.1, 55.2, 48.1\*, 47.7, 44.5\*, 44.4, 28.6; IR (neat) 1689, 1242, 1159  $\text{cm}^{-1}$ ; HRMS (ES+) calcd. for  $\text{C}_{24}\text{H}_{27}\text{NO}_3\text{Na}$   $[\text{M}+\text{Na}]^+$  400.1889, found 400.1895.



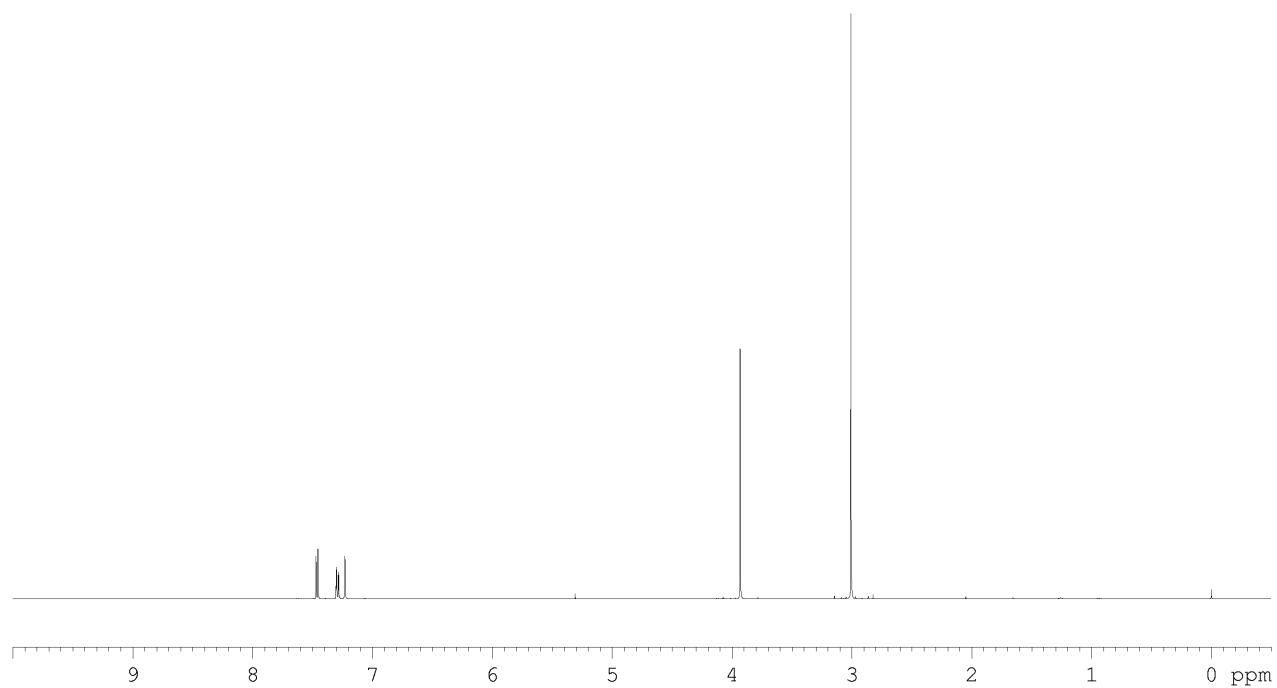
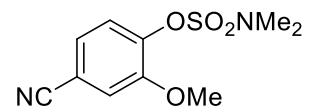
***tert*-Butyl (3,3-Diethoxypropyl)(naphthalen-1-ylmethyl)carbamate **10f**.**

According to General Procedure for Suzuki–Miyaura Cross-coupling Reaction, the desired product **10f** was obtained as a colorless oil in 86% isolated yield after column chromatography (hexanes/EtOAc = 20:1 to 10:1).

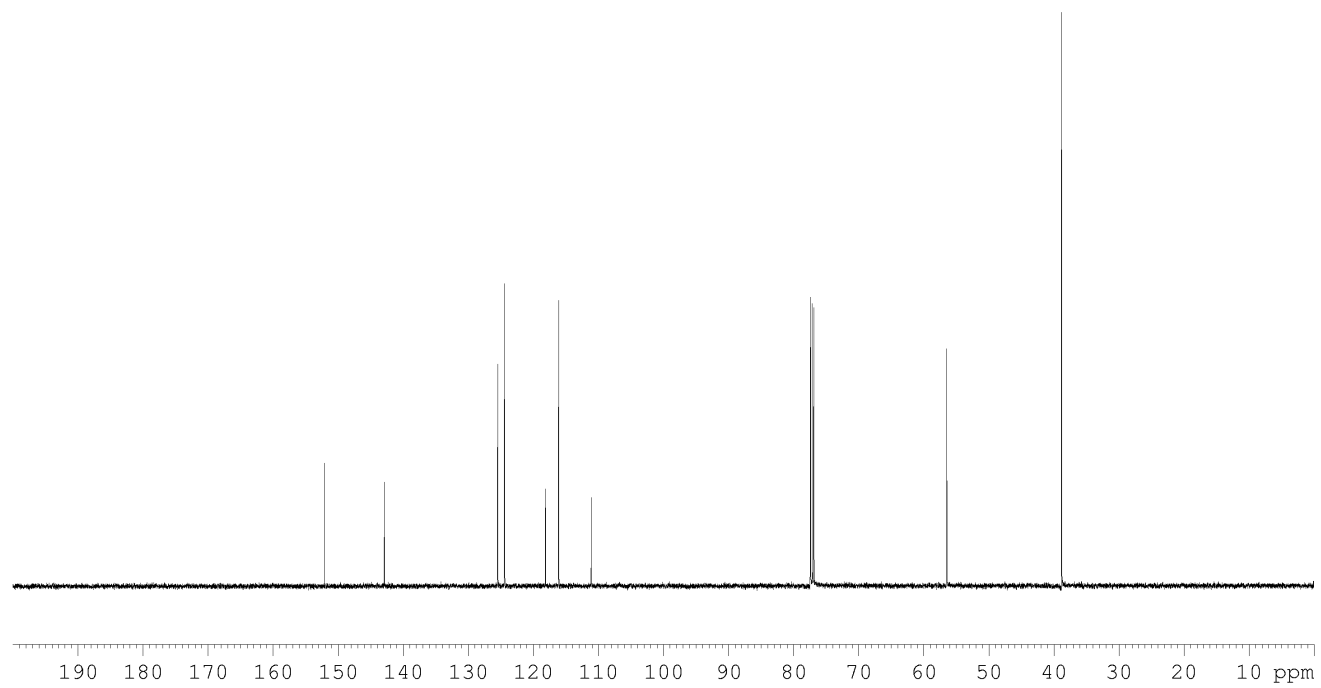
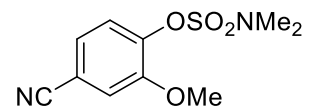
$^1\text{H}$  NMR (asterisk denotes minor rotamer peaks, 500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.14 (s, 1H), 8.04\* (s, 1H), 7.85 (d,  $J = 7.5$  Hz, 1H), 7.77 (d,  $J = 8.5$  Hz, 1H), 7.53–7.45 (m, 2H), 7.418 (dd,  $J = 7.0, 7.0$  Hz, 1H), 7.37–7.28 (m, 1H), 4.93 (s, 2H), 4.46\* (s, 1H), 4.37 (s, 1H), 3.59–3.11 (m, 6H), 1.88–1.74 (m, 2H), 1.56–1.44 (m, 9H), 1.14 (t,  $J = 7.0$  Hz, 6H);  $^{13}\text{C}$  NMR (asterisk denotes minor rotamer peaks, 125.8 MHz,  $\text{CDCl}_3$ )  $\delta$  155.7, 133.9, 133.5, 131.9, 131.5\*, 128.7, 128.3, 128.0\*, 126.5\*, 126.3, 125.8, 125.3, 124.9\*, 124.0, 123.1, 101.0, 79.9, 61.0, 48.7\*, 48.2, 42.6\*, 41.9, 32.1, 28.5, 15.3; IR (neat) 2975, 1690, 1167, 1136, 1062  $\text{cm}^{-1}$ ; HRMS (ES+) calcd. for  $\text{C}_{23}\text{H}_{33}\text{NO}_4\text{Na}$   $[\text{M}+\text{Na}]^+$  410.2307, found 410.2307.

## References:

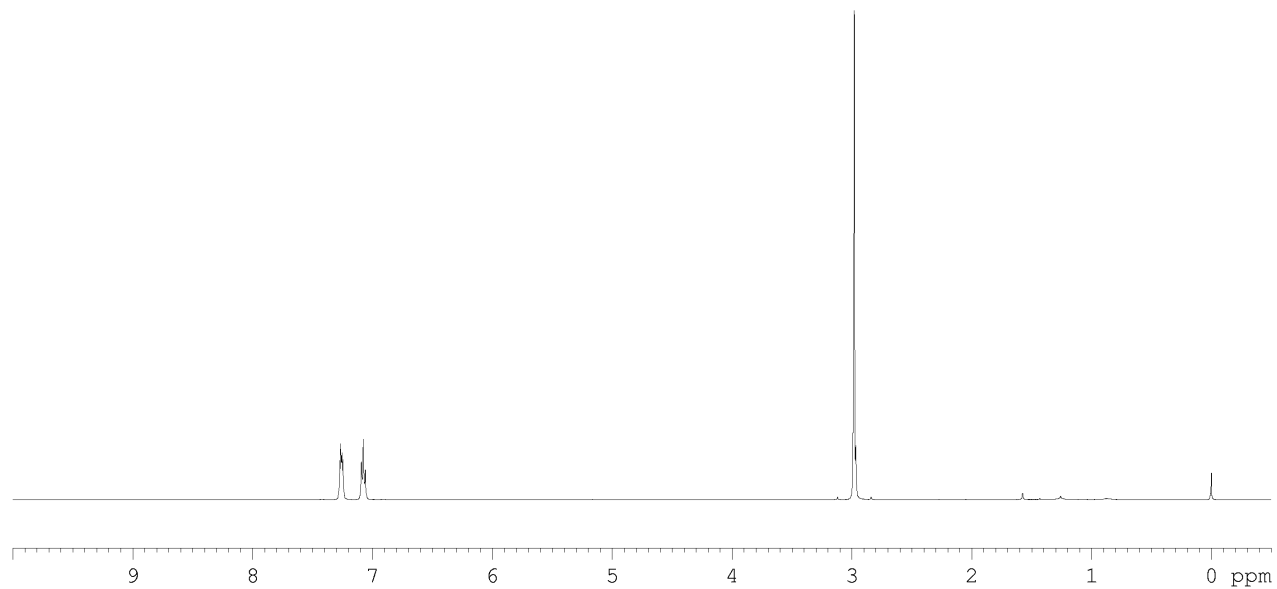
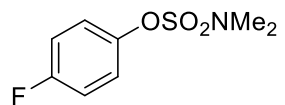
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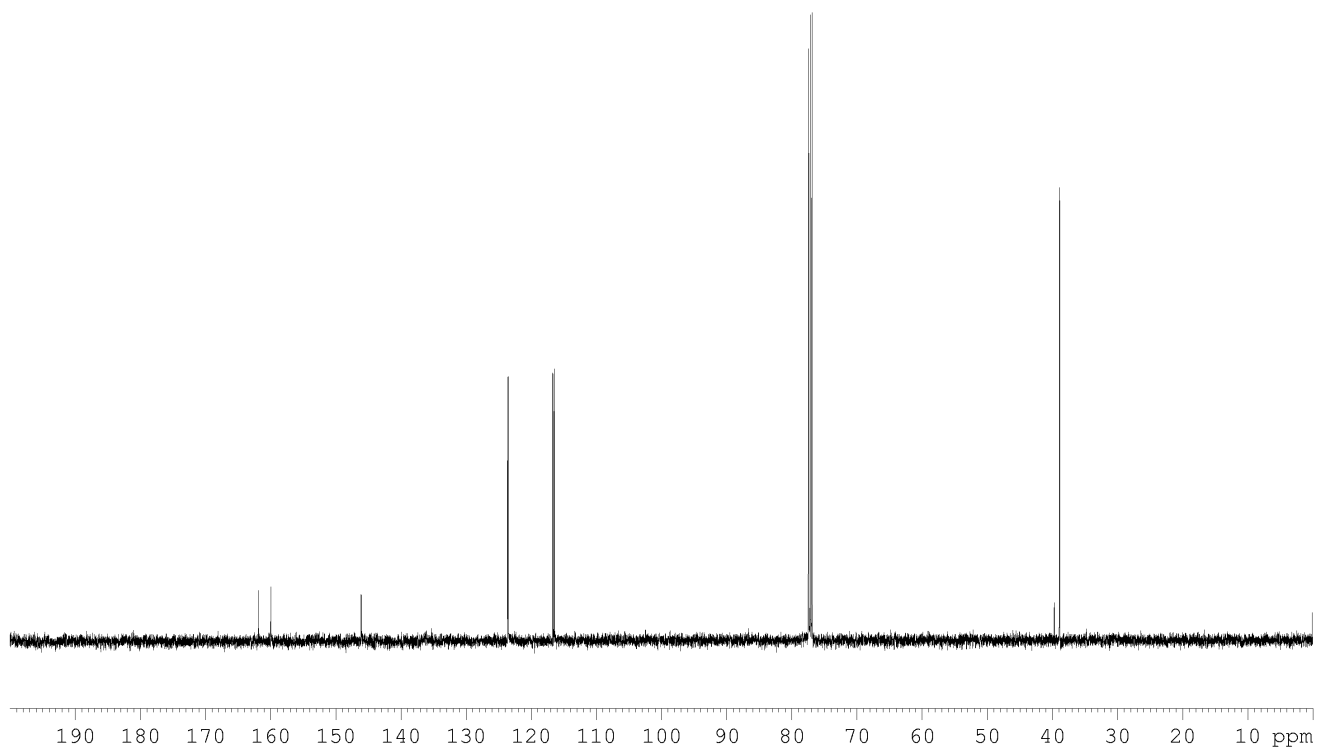
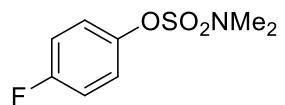
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum



<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum

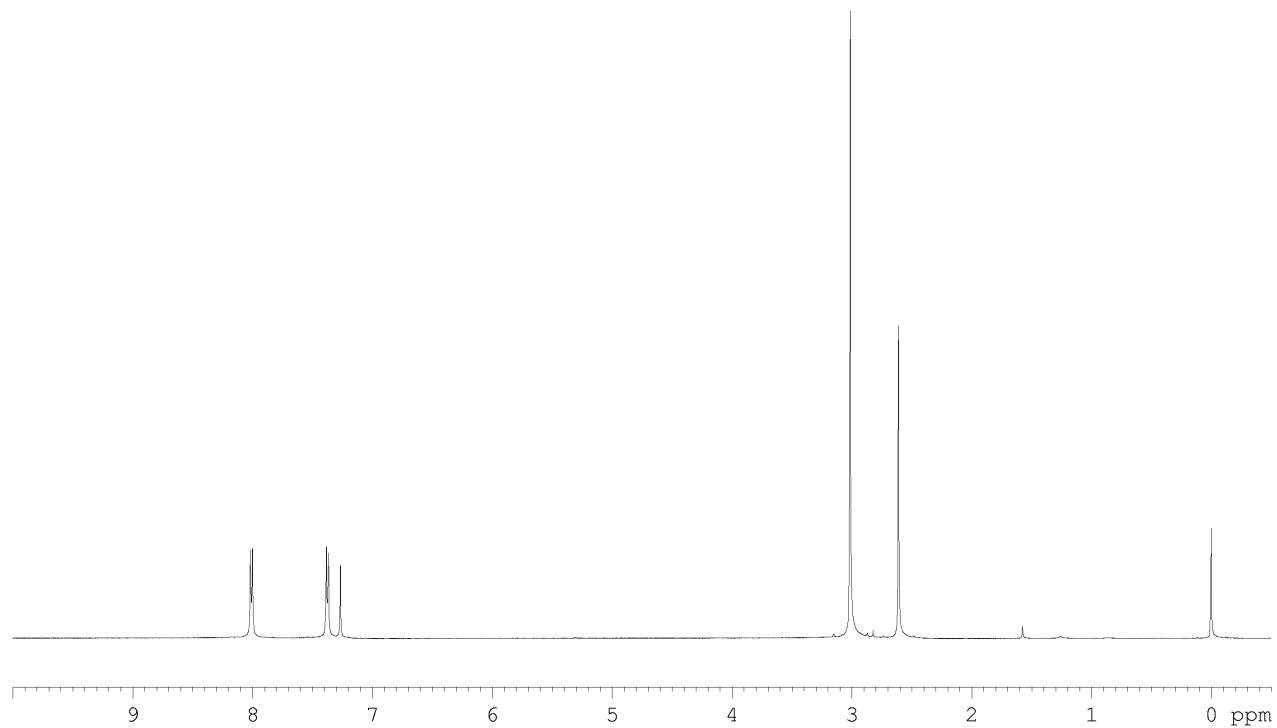
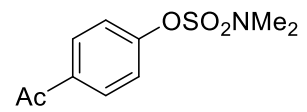


$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) Spectrum

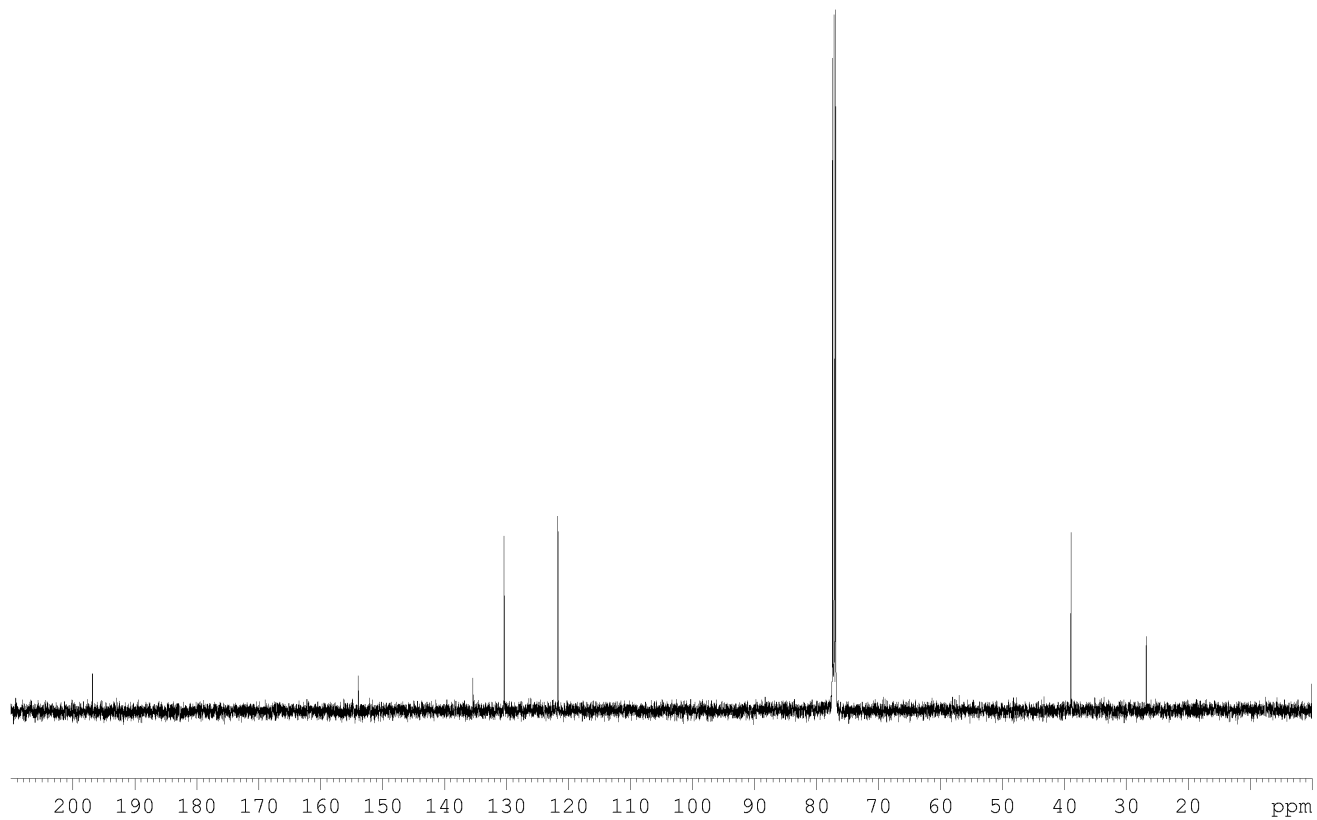
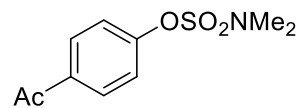


<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum

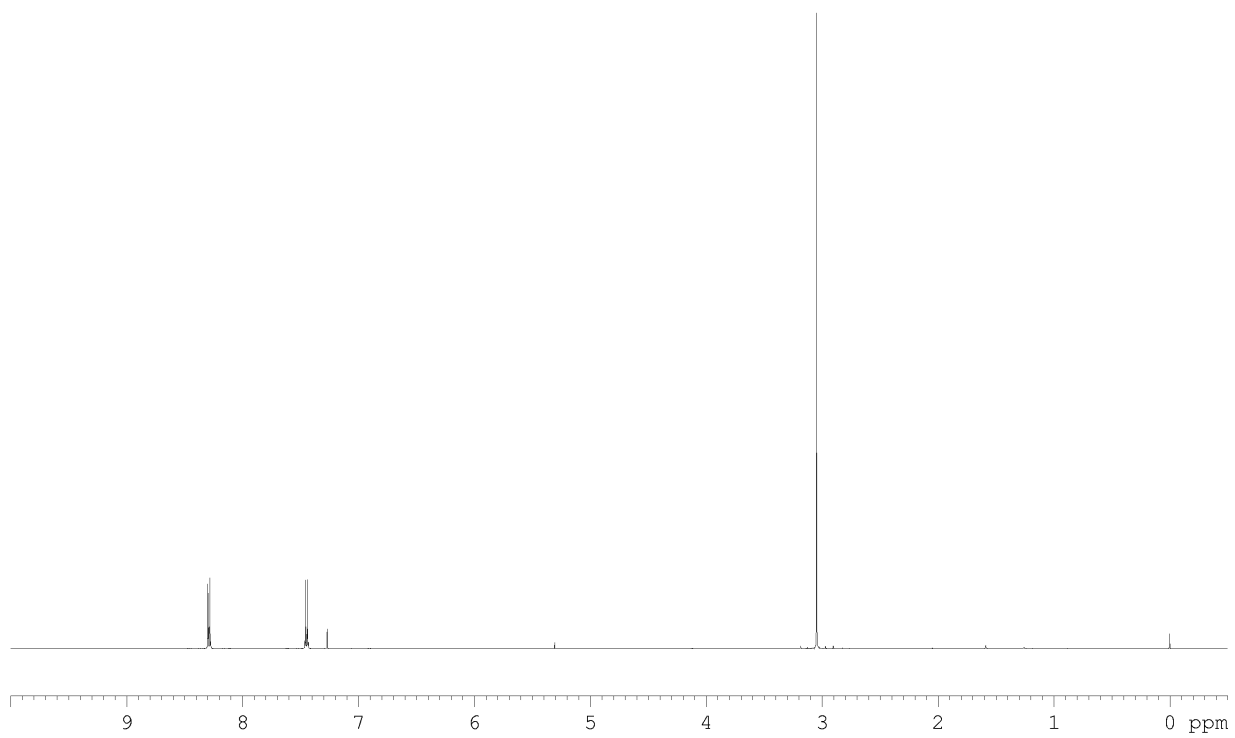
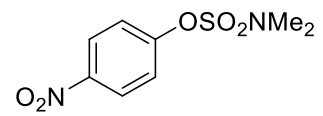




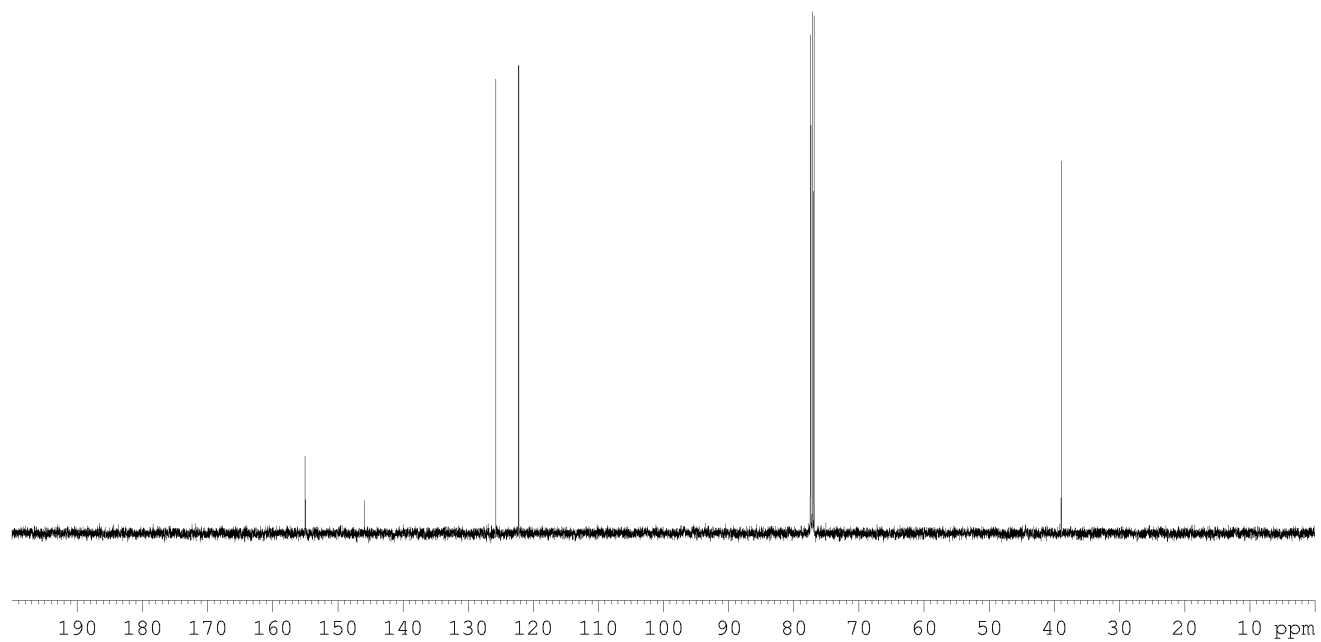
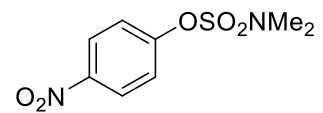
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum



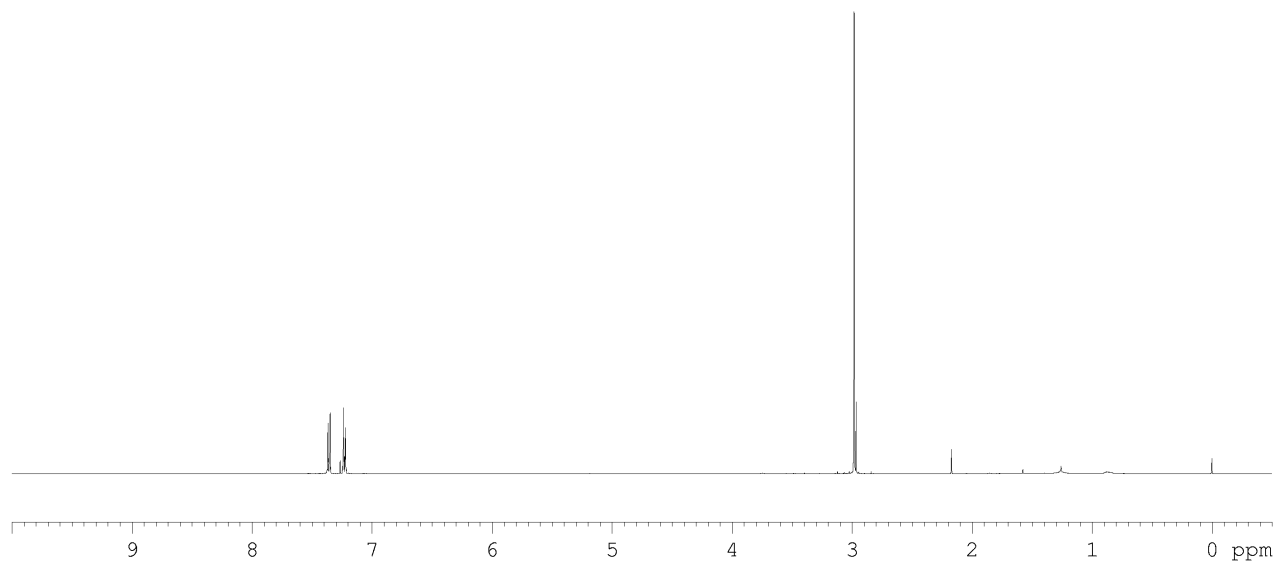
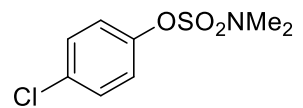
<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum



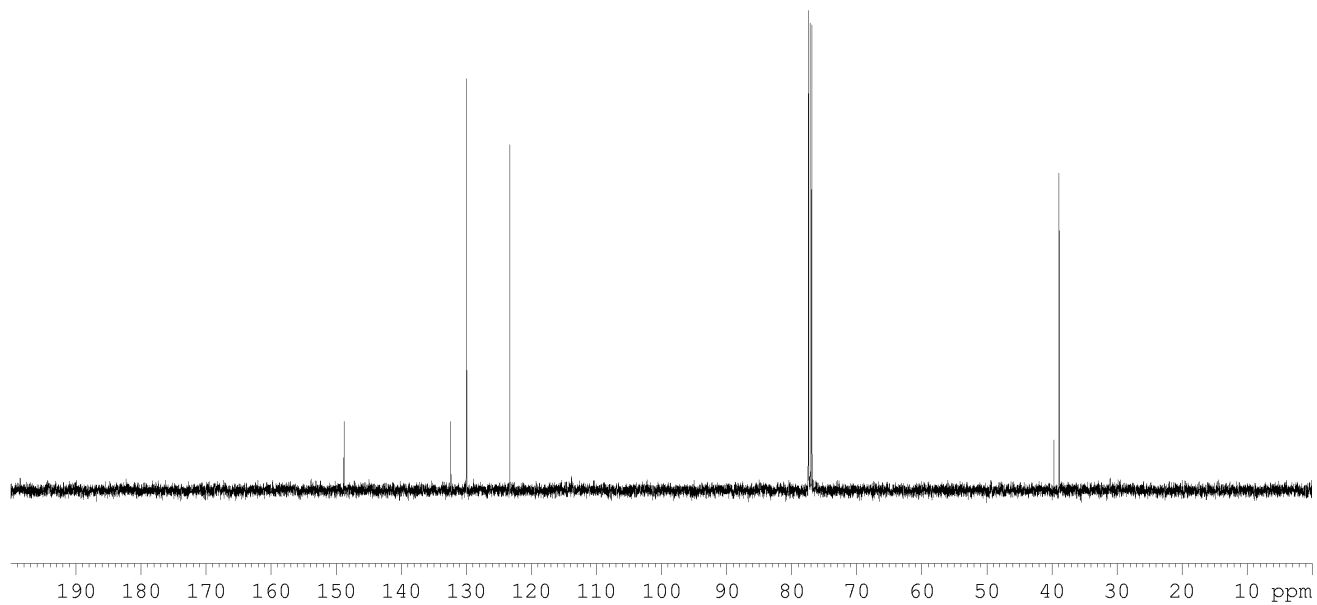
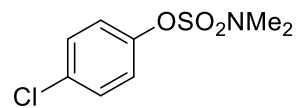
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) Spectrum



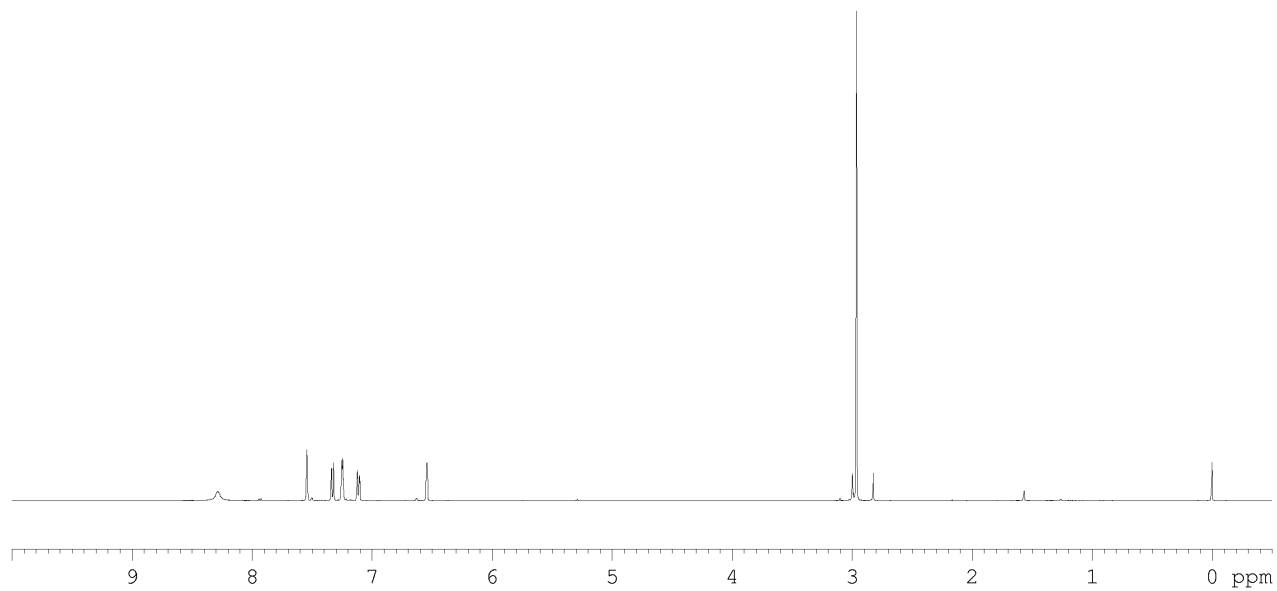
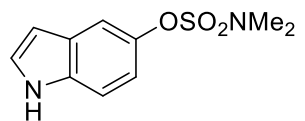
$^{13}\text{C}$  NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum



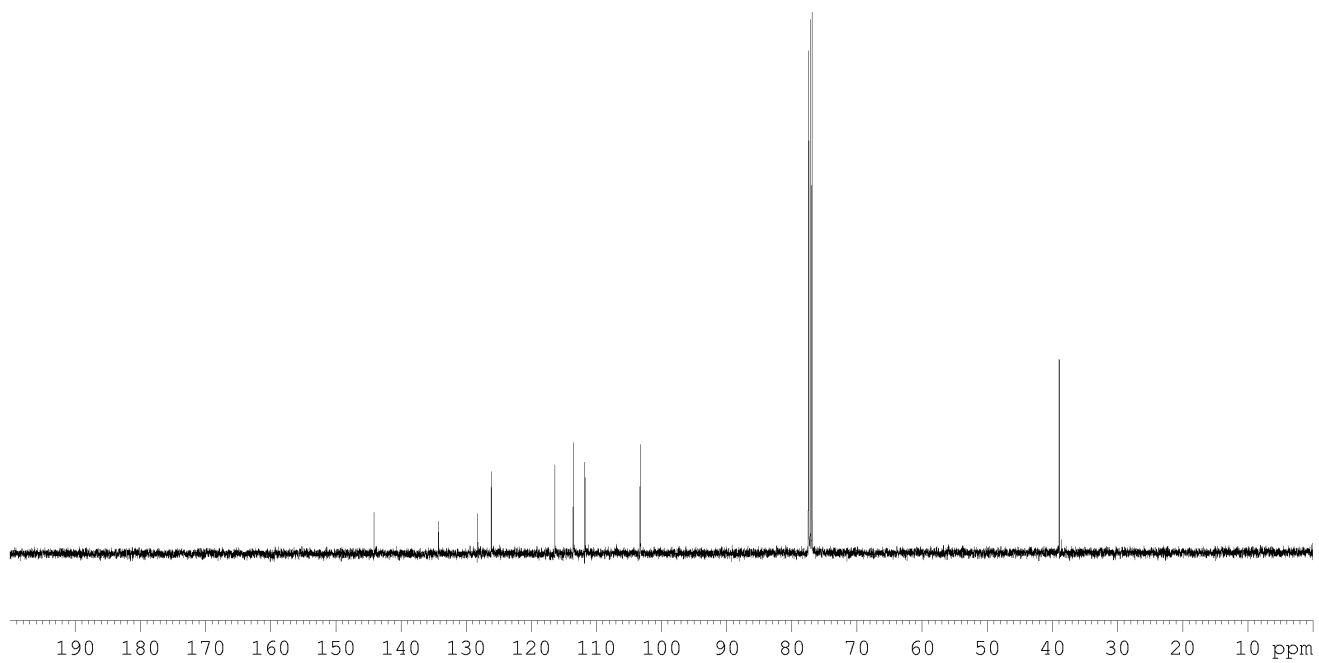
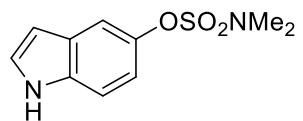
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) Spectrum of **3**



<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum of **3**

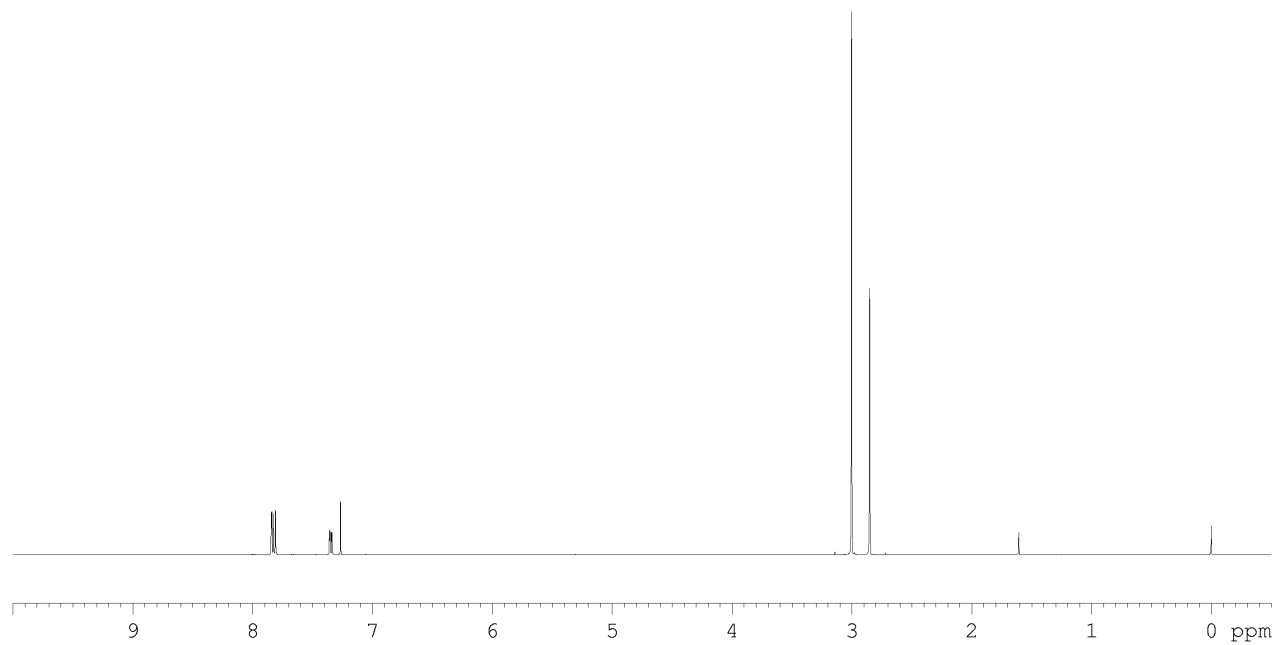
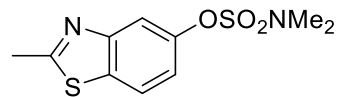


<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum



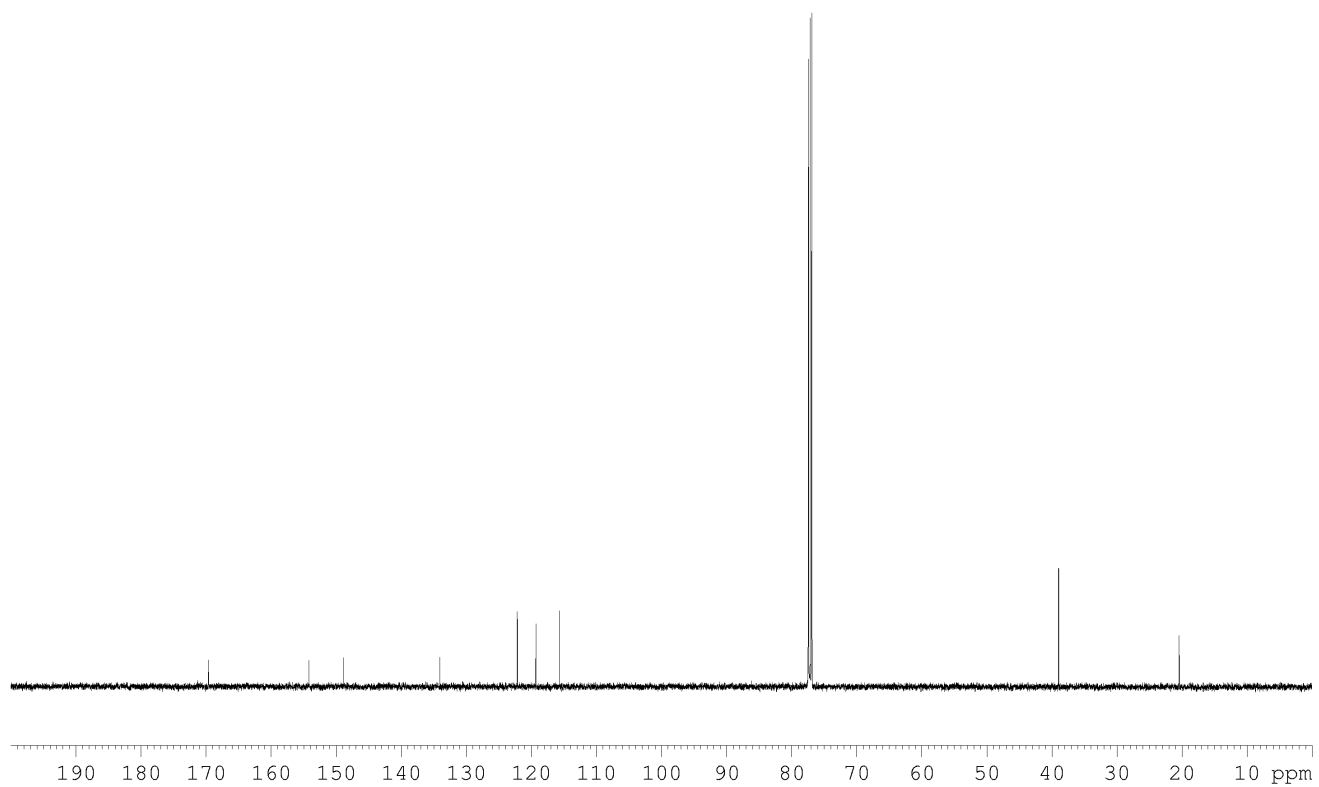
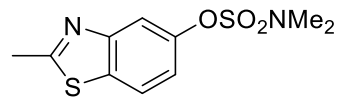
$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum



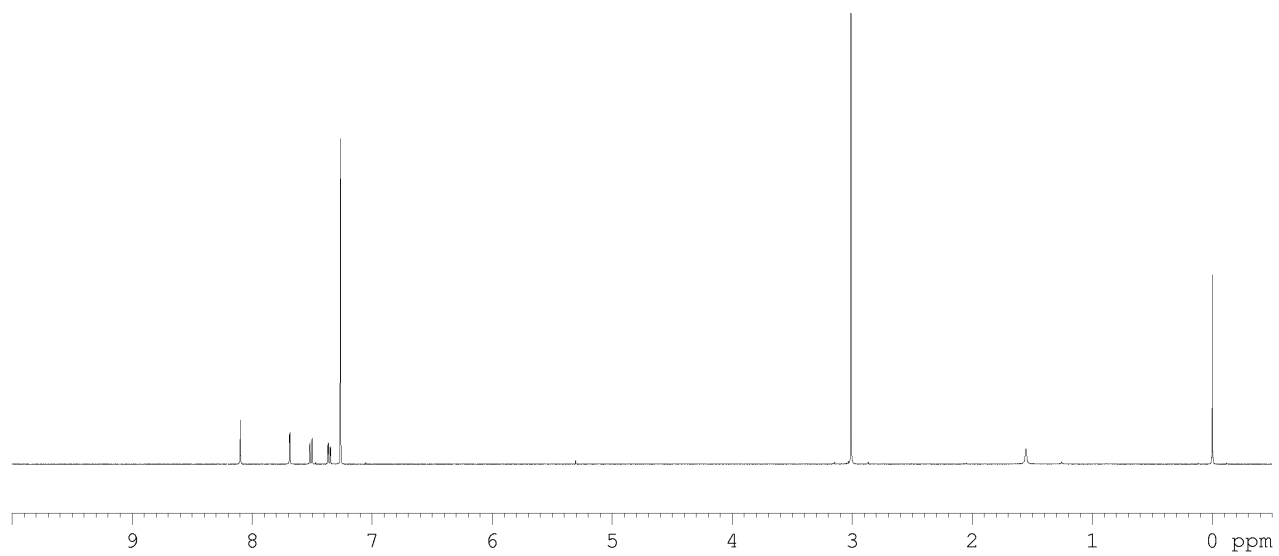
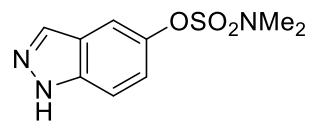


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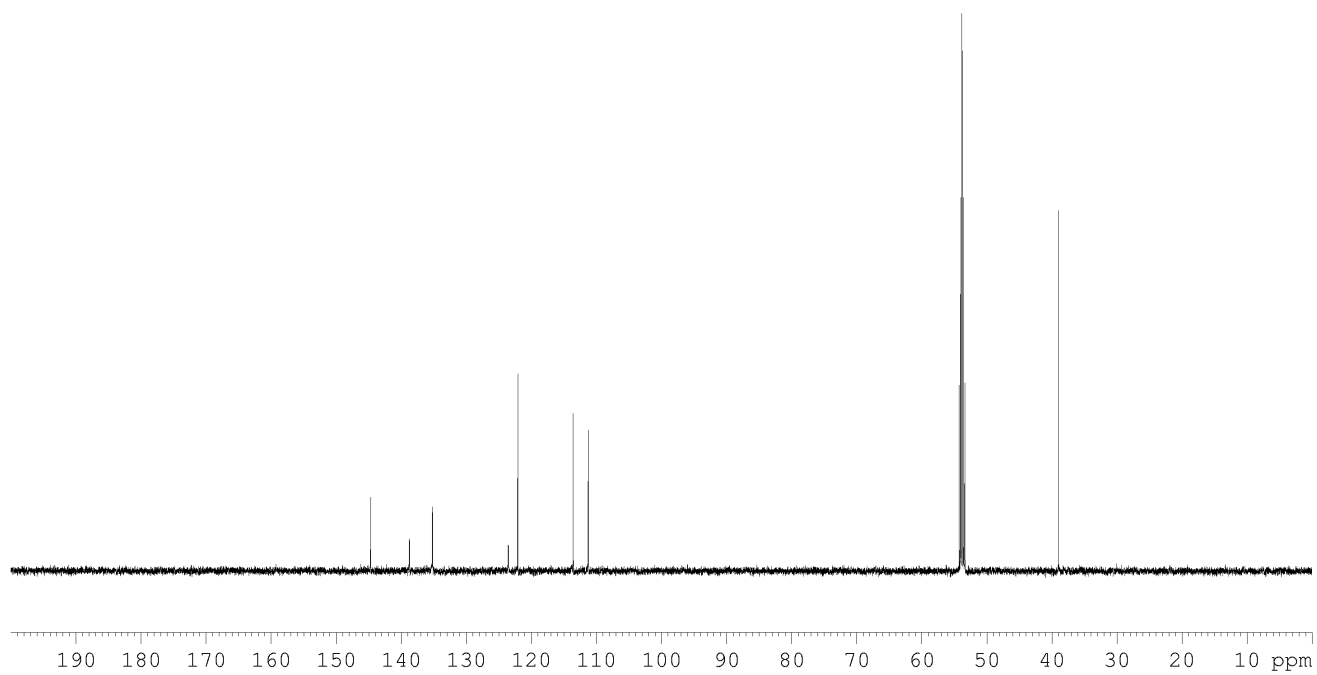
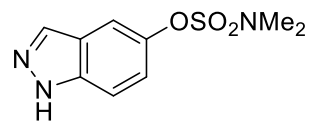
S41



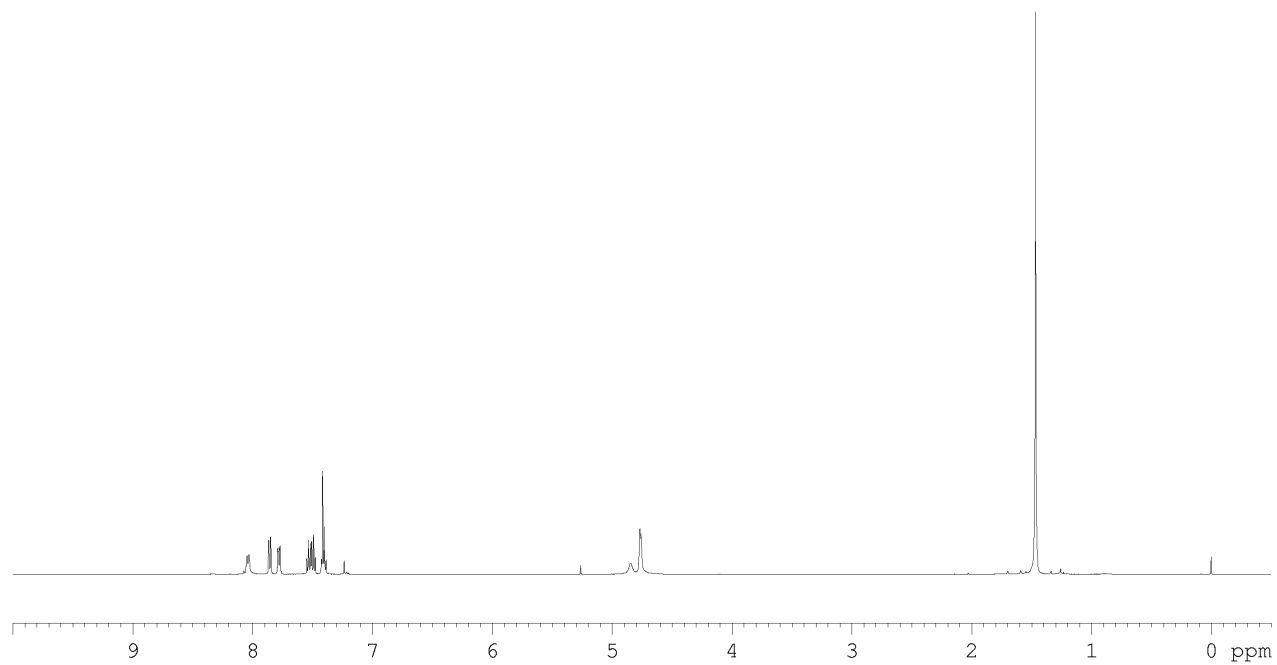
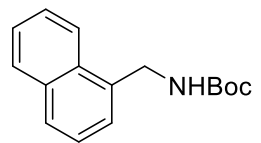
<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum



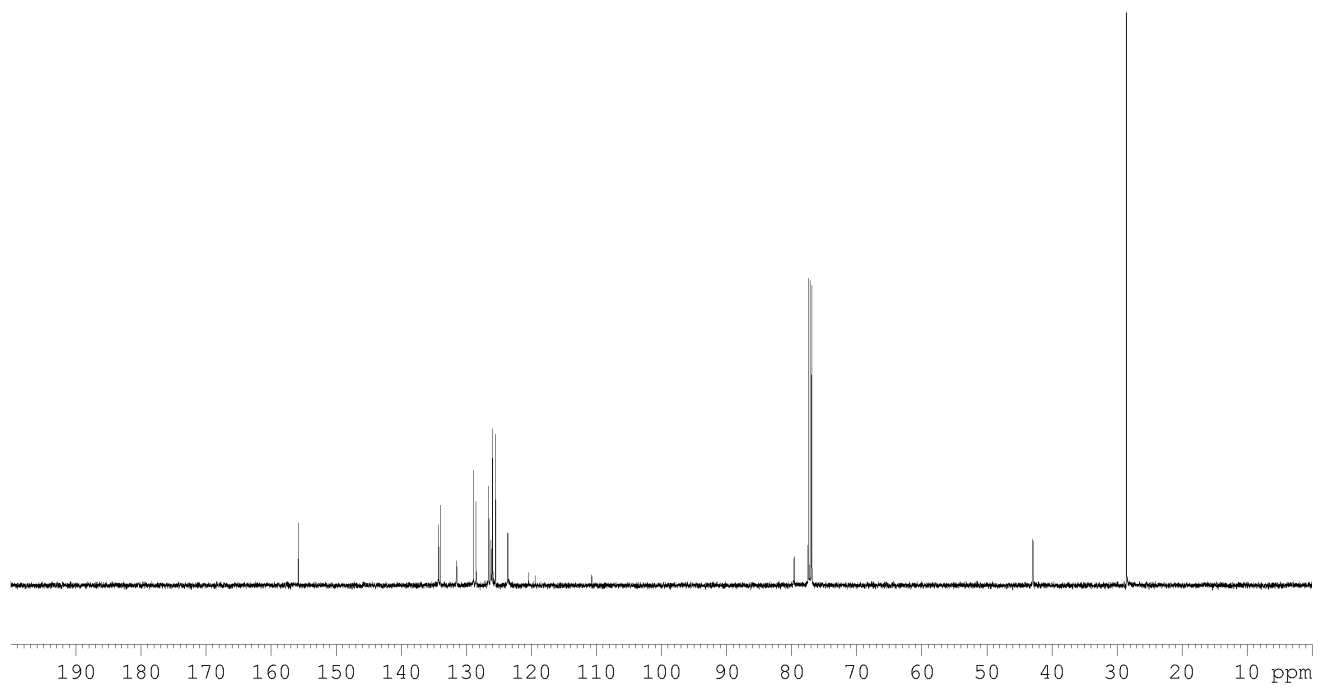
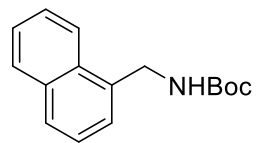
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum



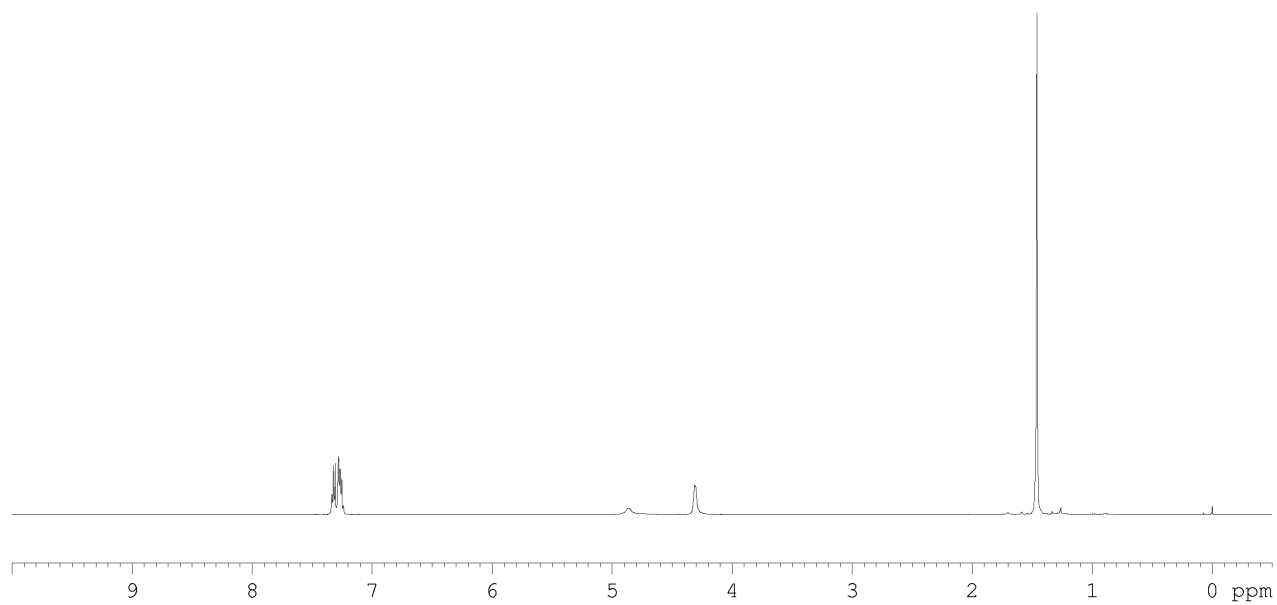
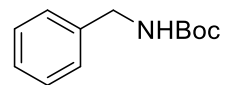
$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CD}_2\text{Cl}_2$ ) Spectrum



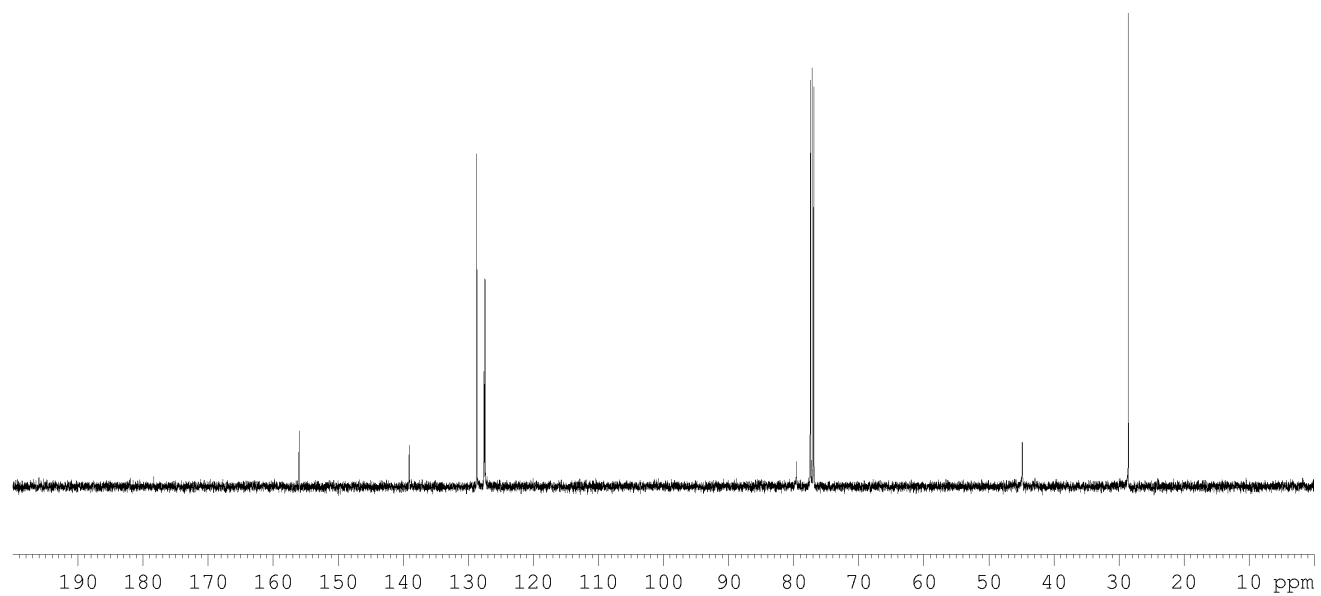
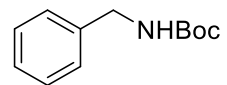
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **2a**



$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **2a**

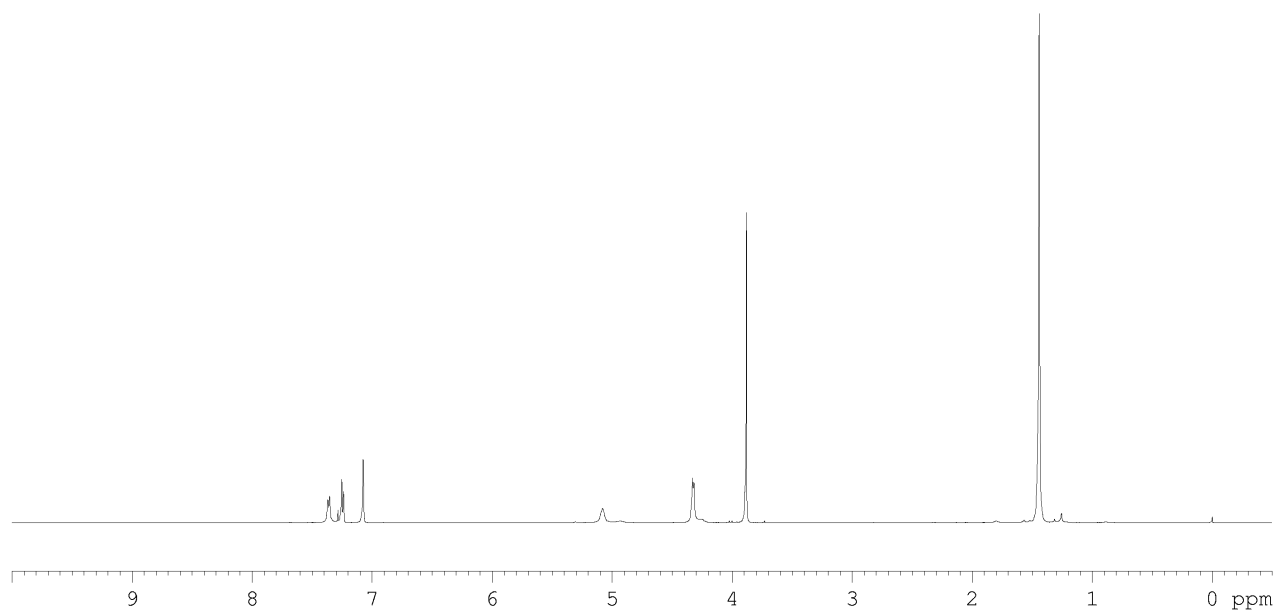
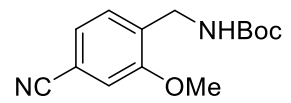


$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) Spectrum of **2b**

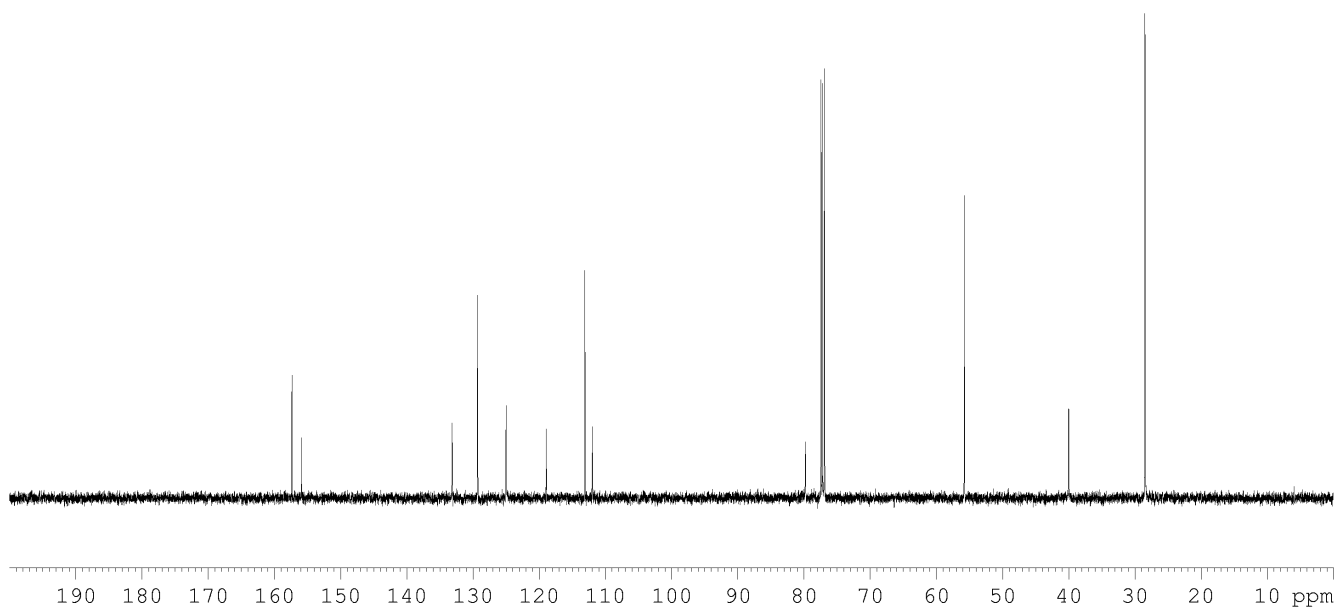
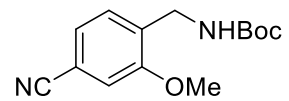


$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **2b**

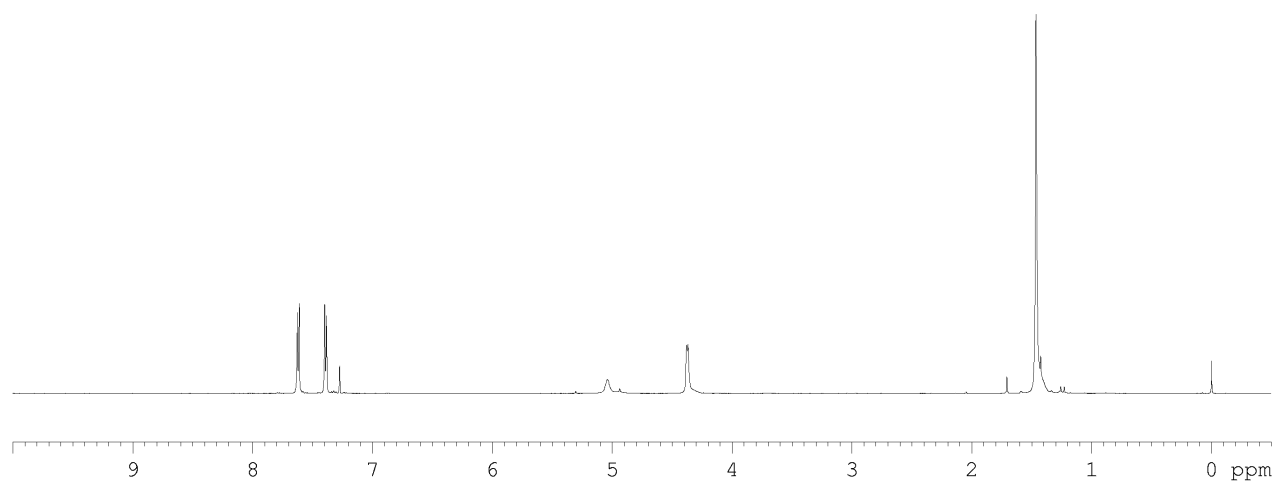
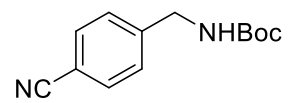




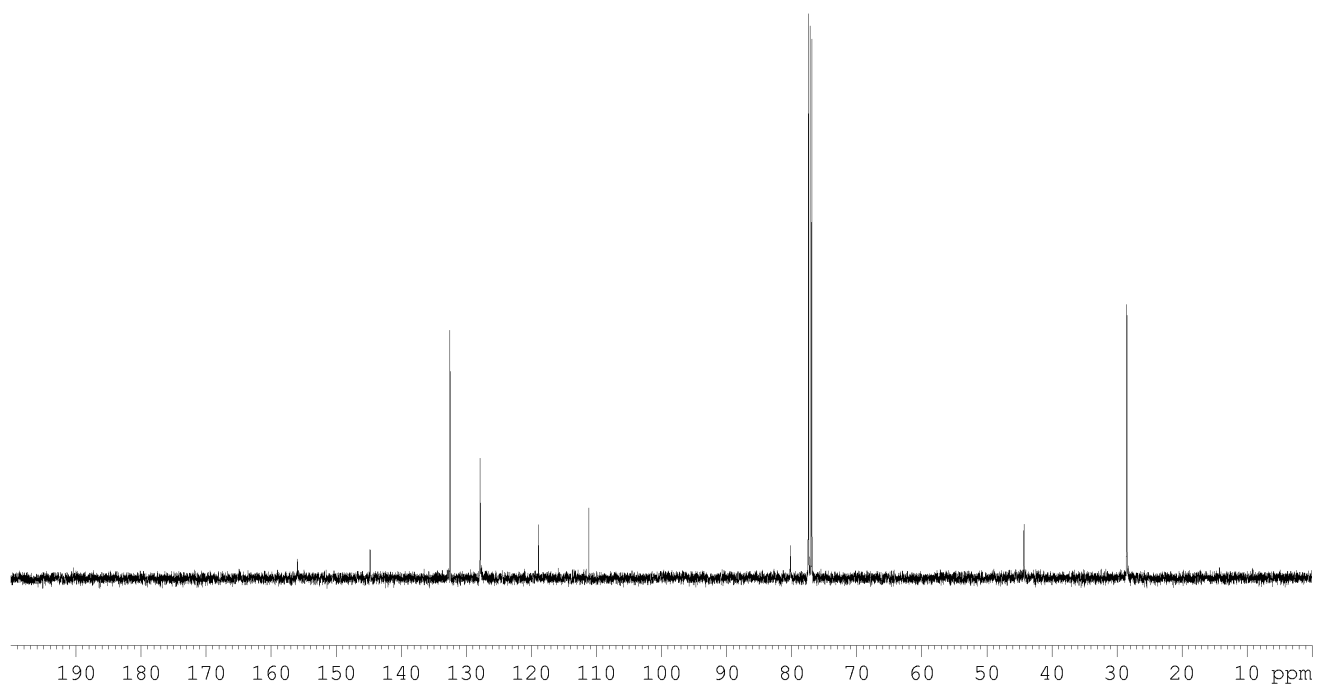
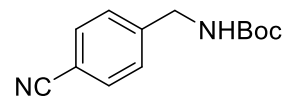
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) Spectrum of **2f**



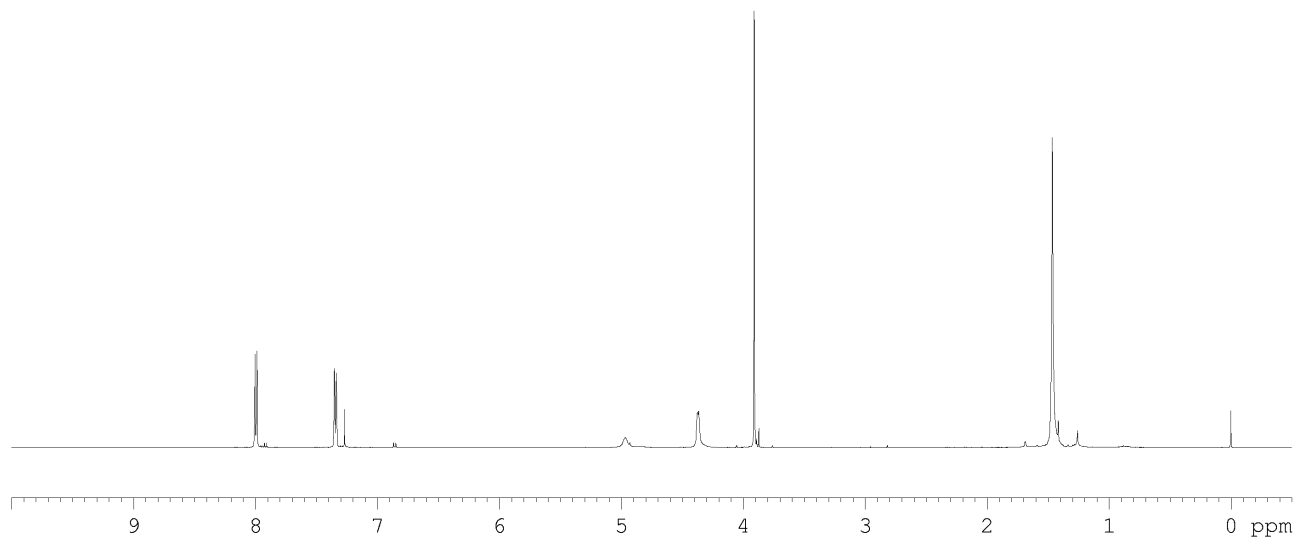
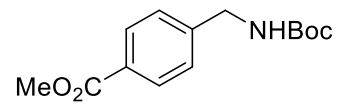
$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **2f**



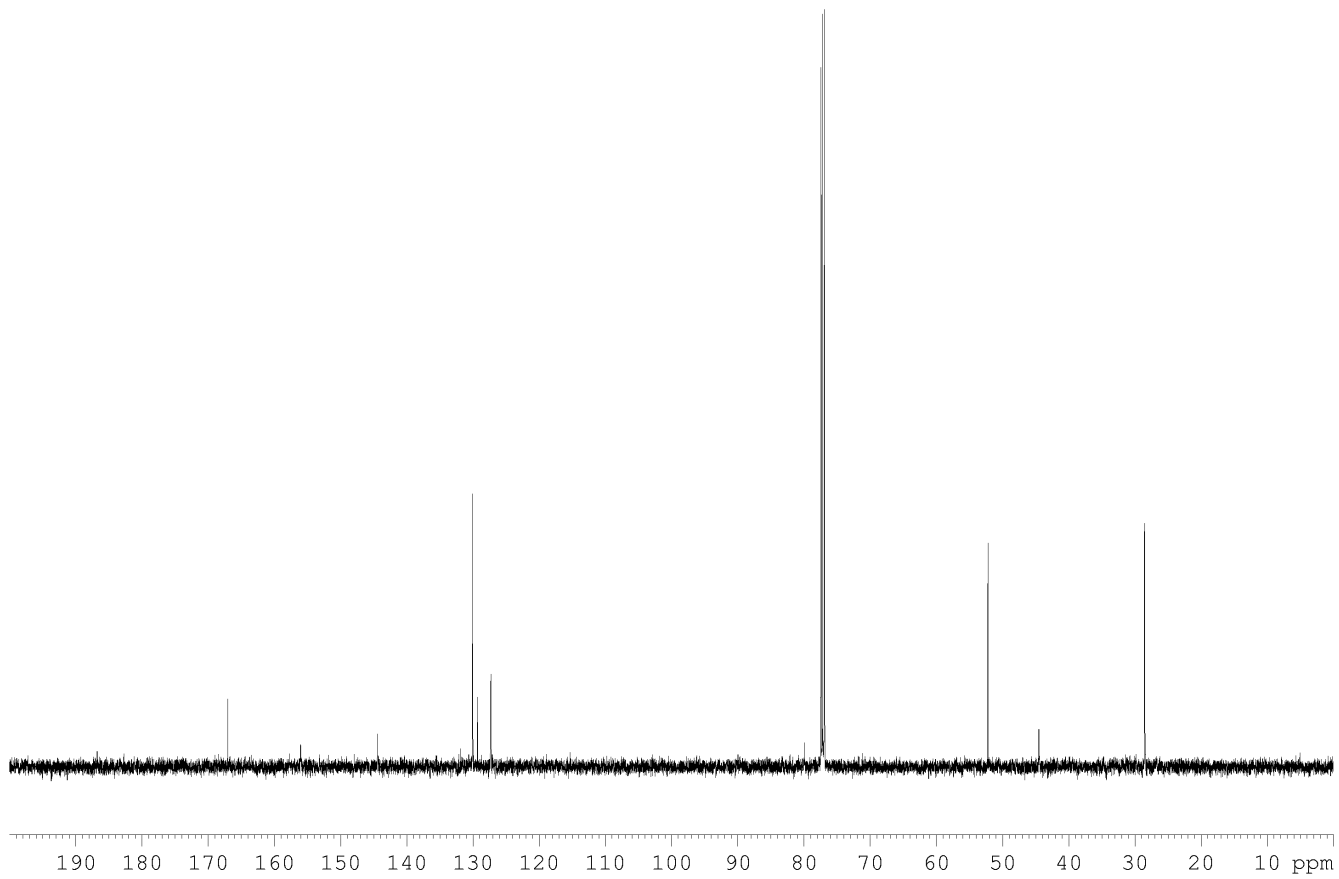
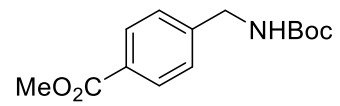
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **2g**



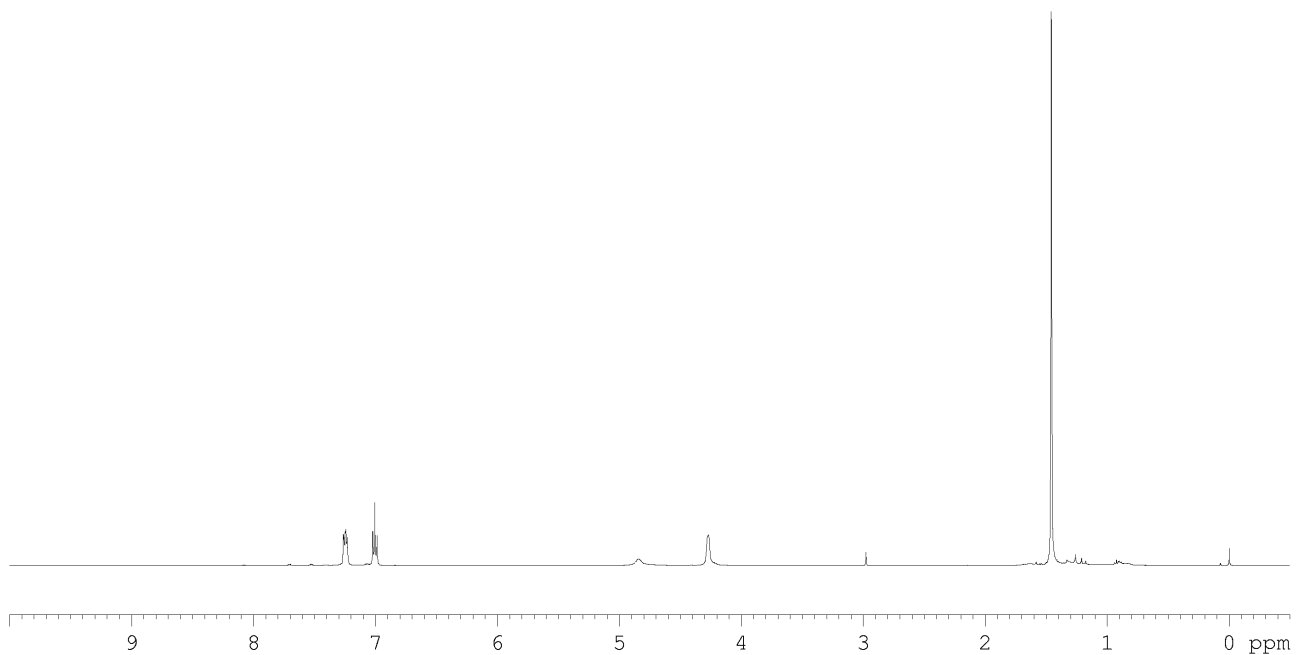
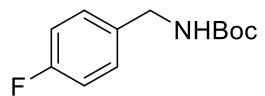
$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **2g**



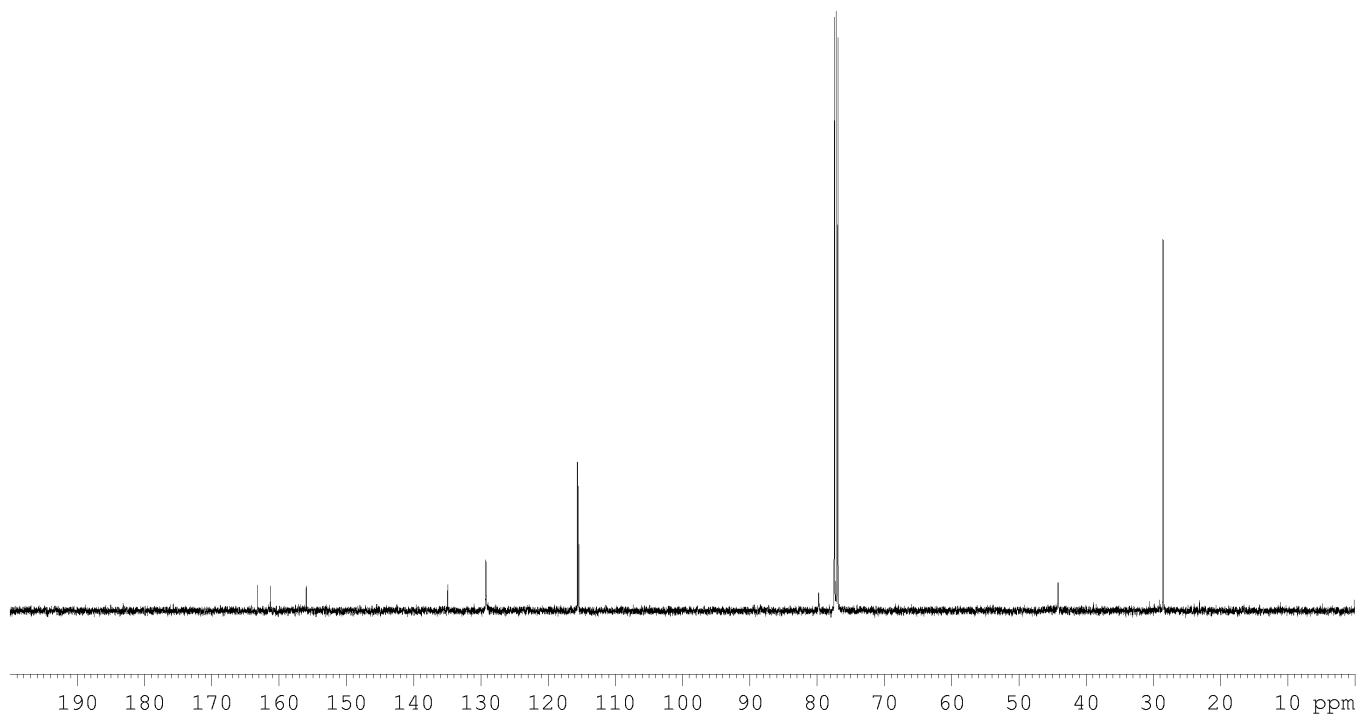
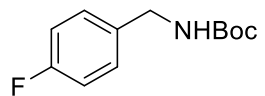
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **2h**



<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum of **2h**

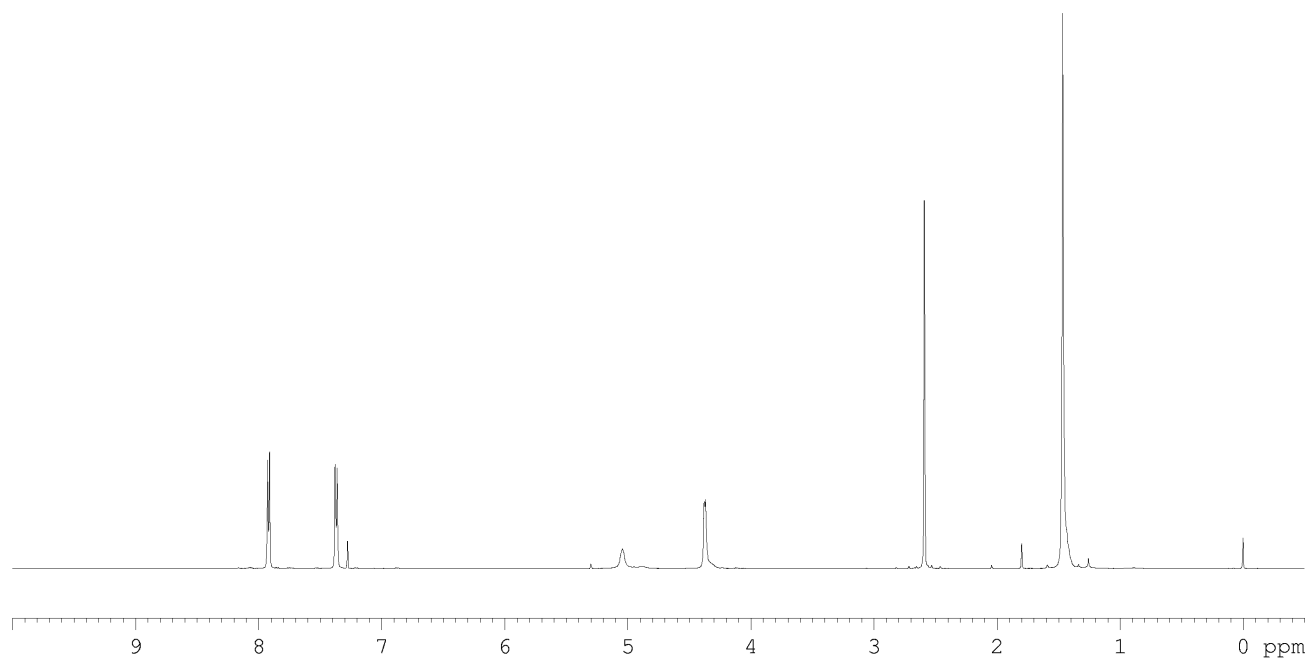
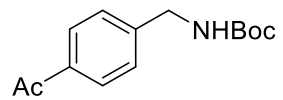


<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **2i**

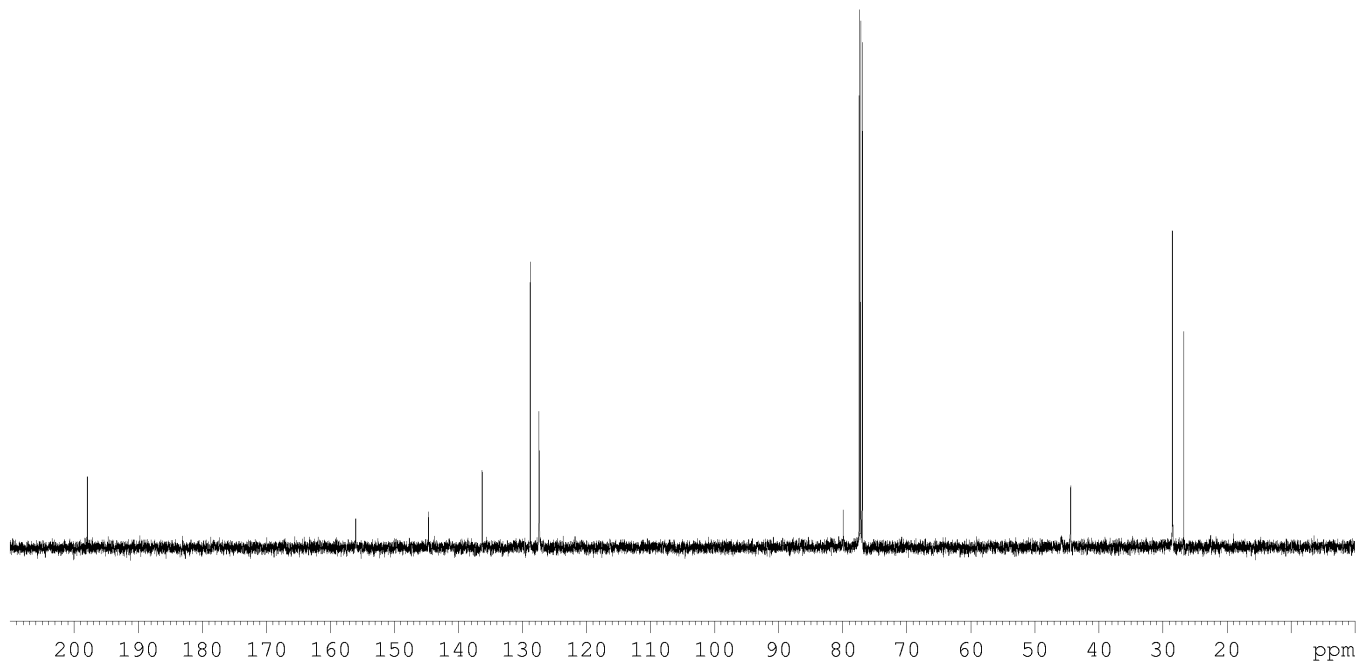
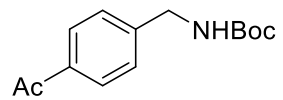


<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum of **2i**

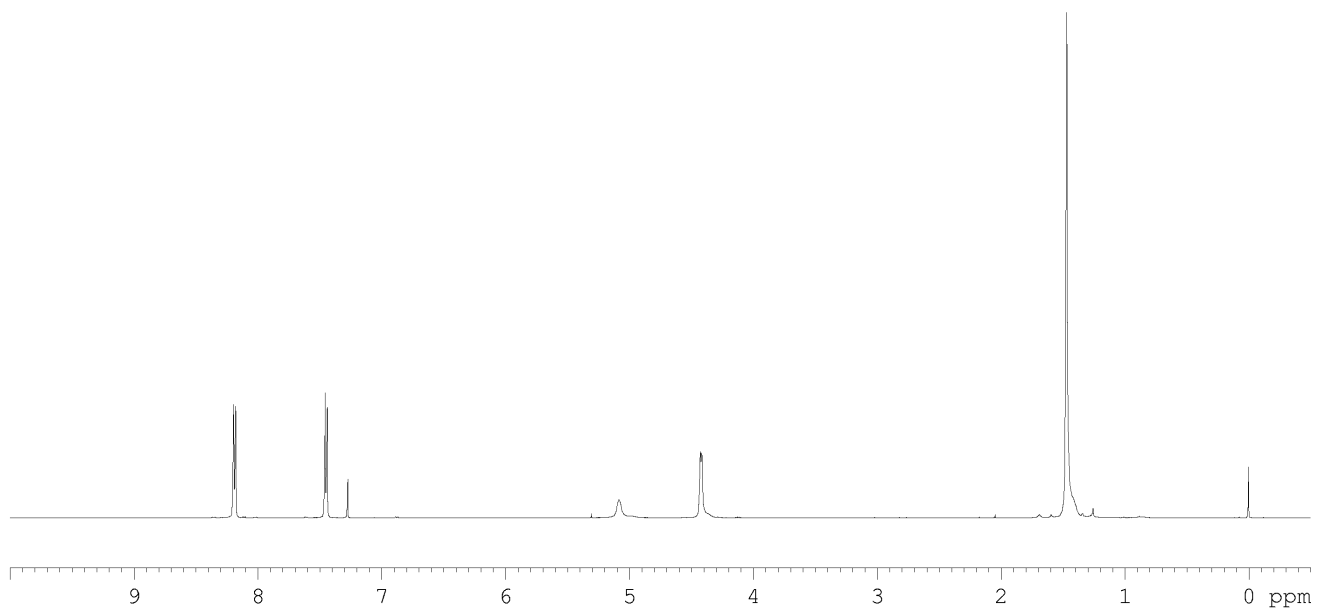
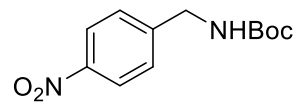




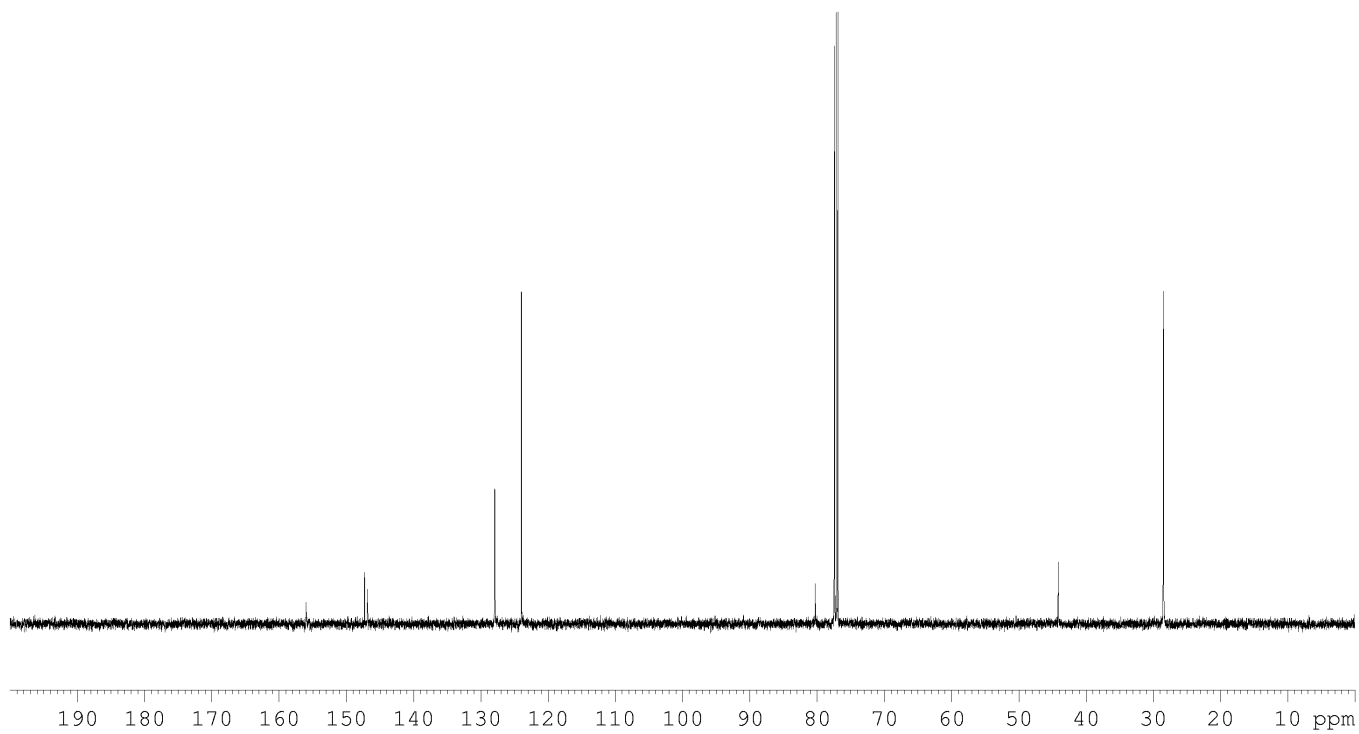
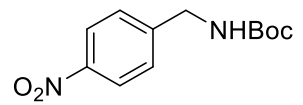
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **2j**



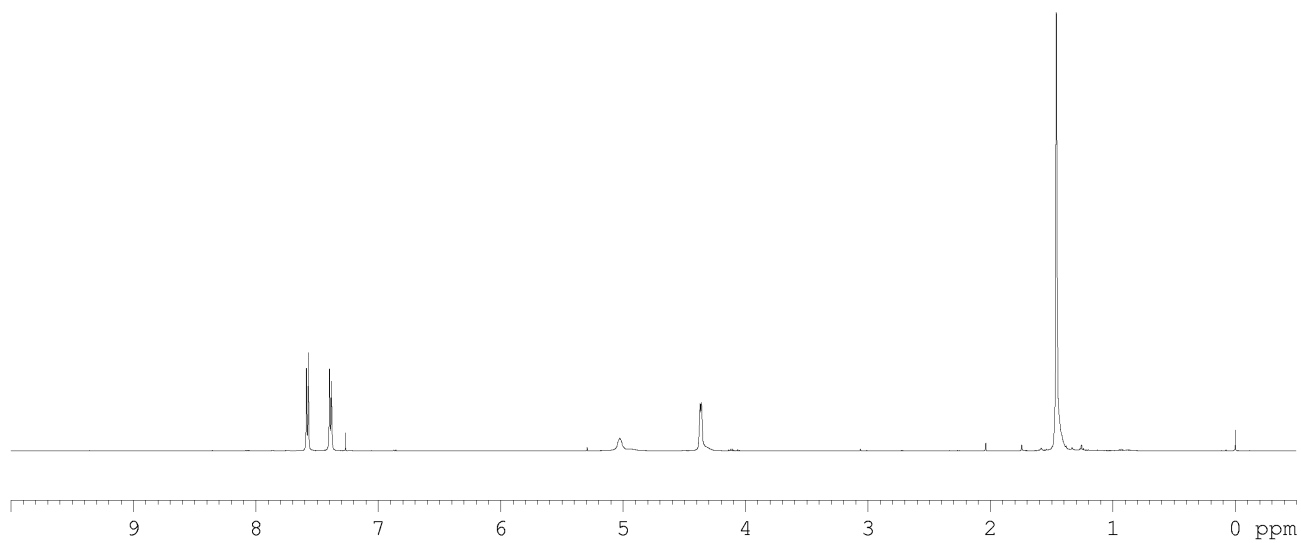
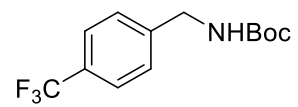
$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **2j**



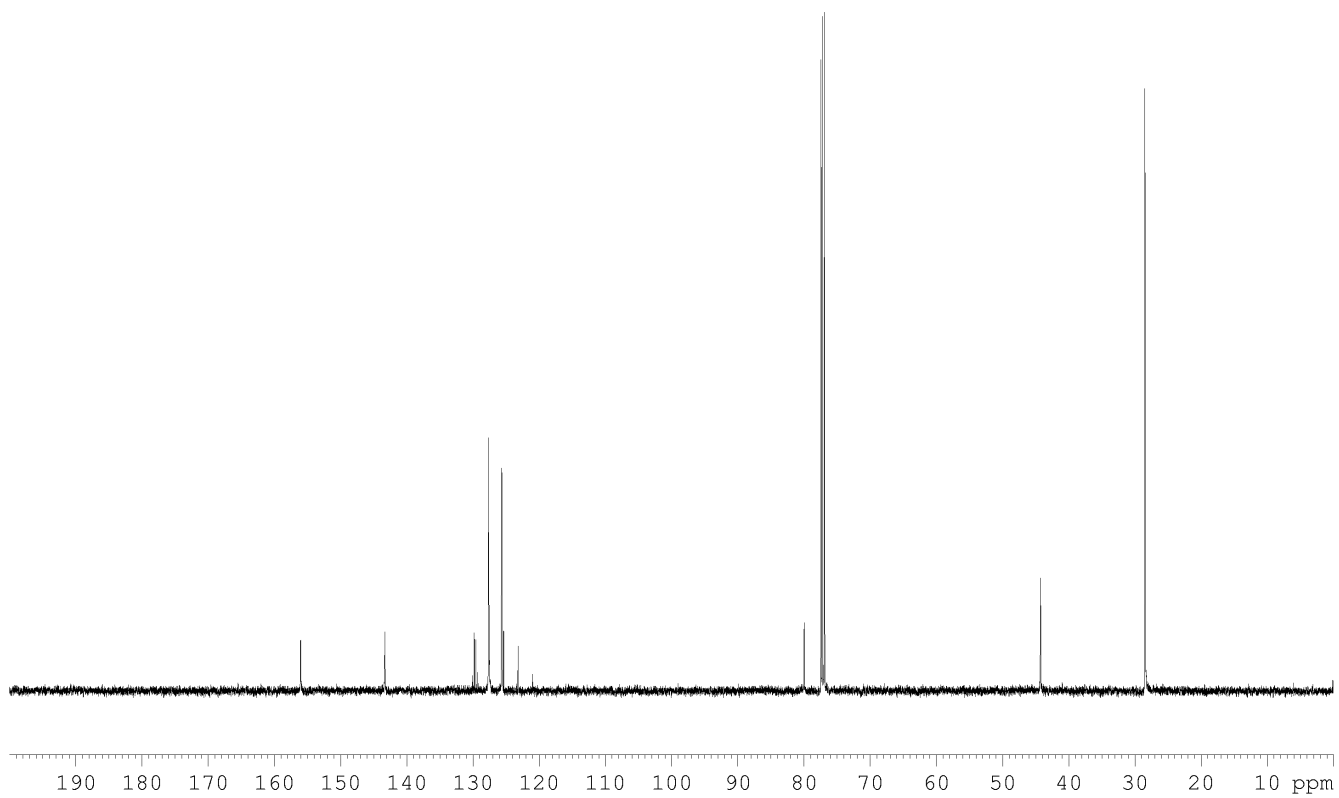
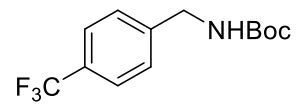
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **2k**



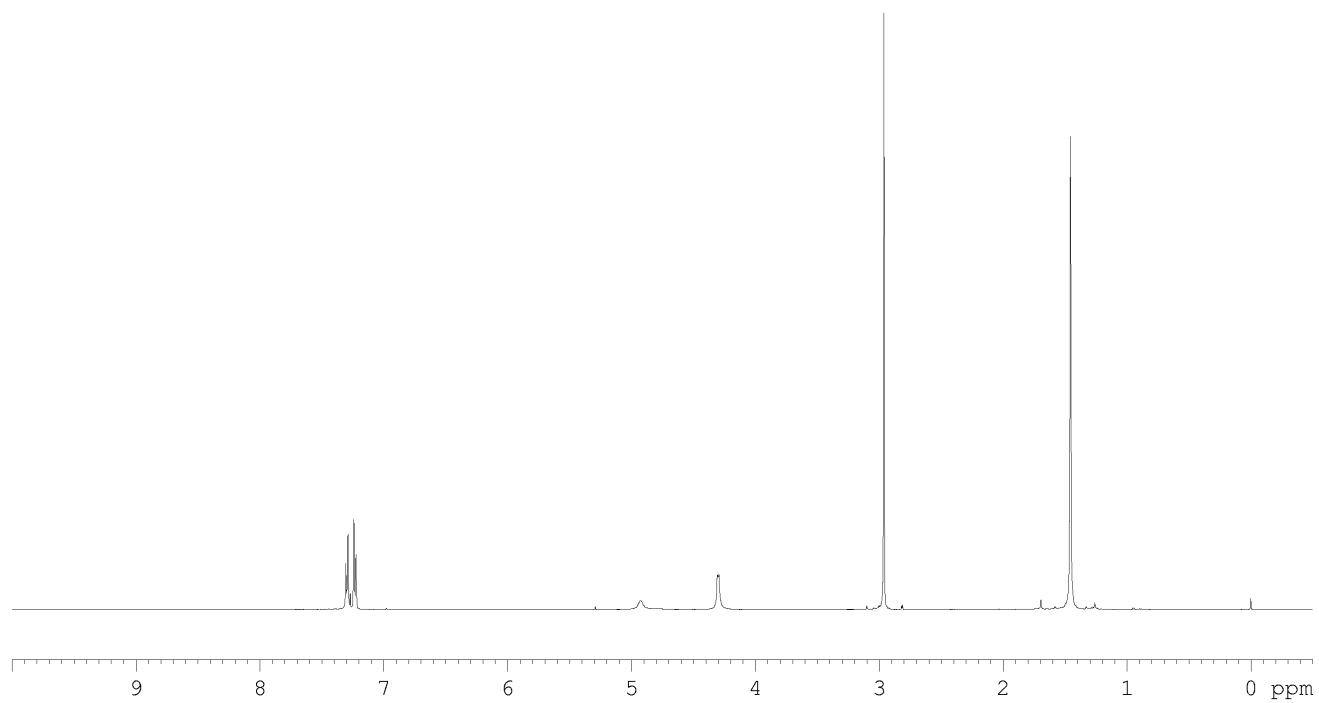
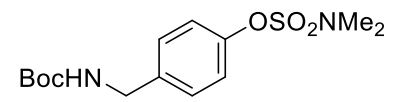
<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum of **2k**



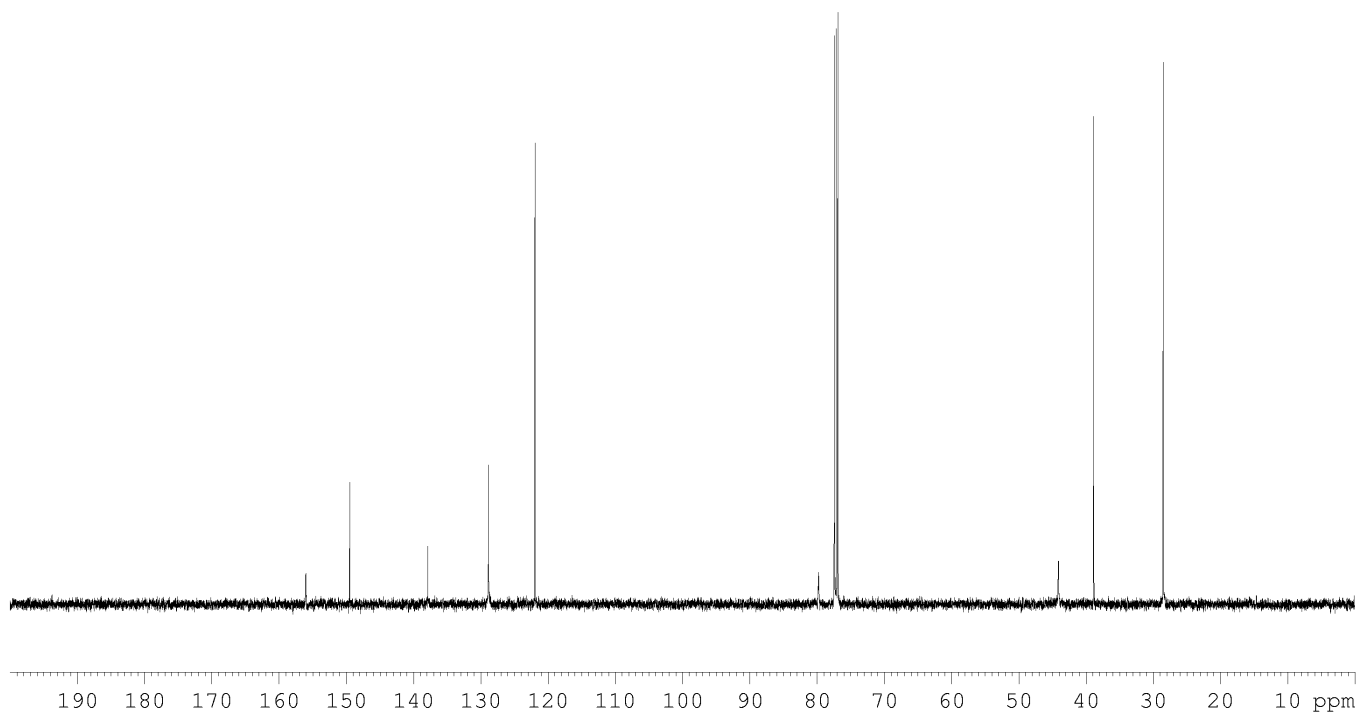
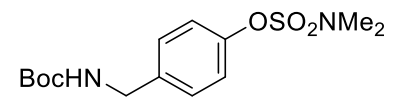
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **21**



$^{13}C$  NMR (125.8 MHz,  $CDCl_3$ ) Spectrum of **21**

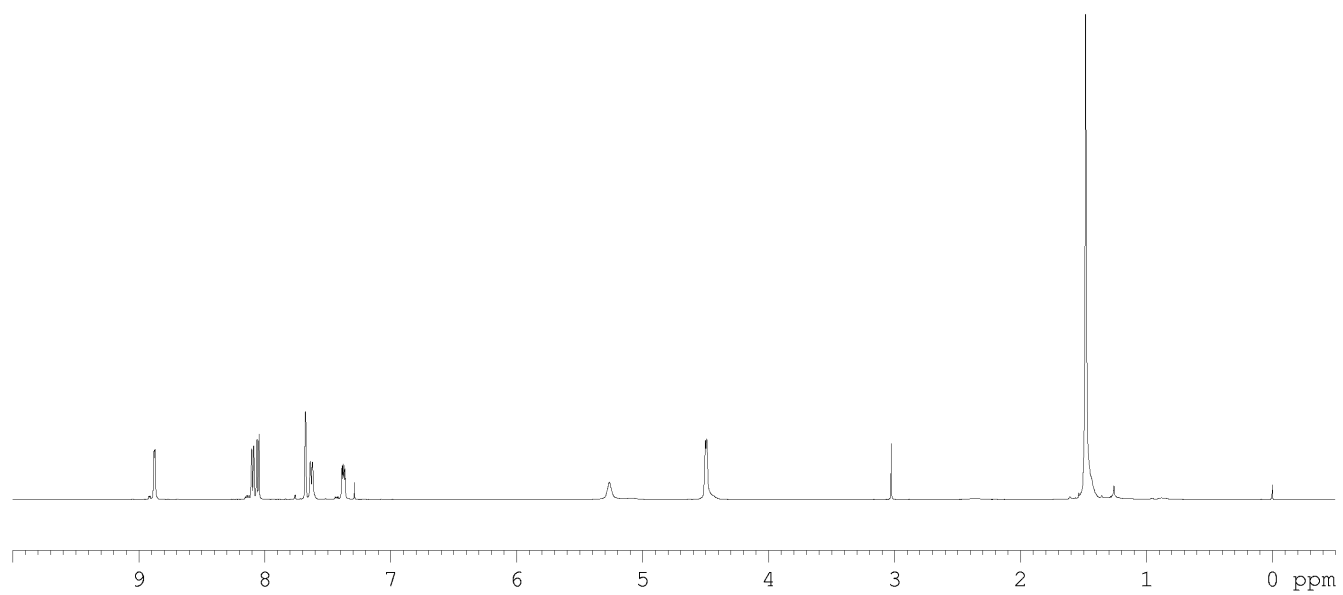
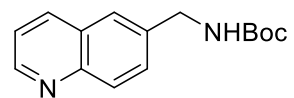


<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **4**

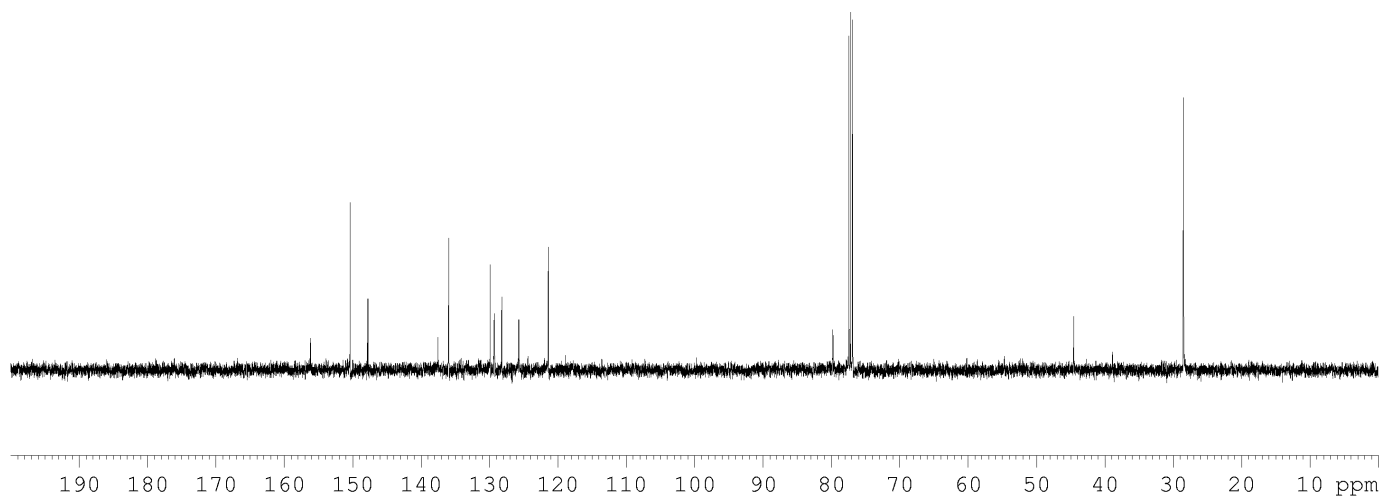
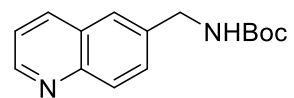


$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of 4

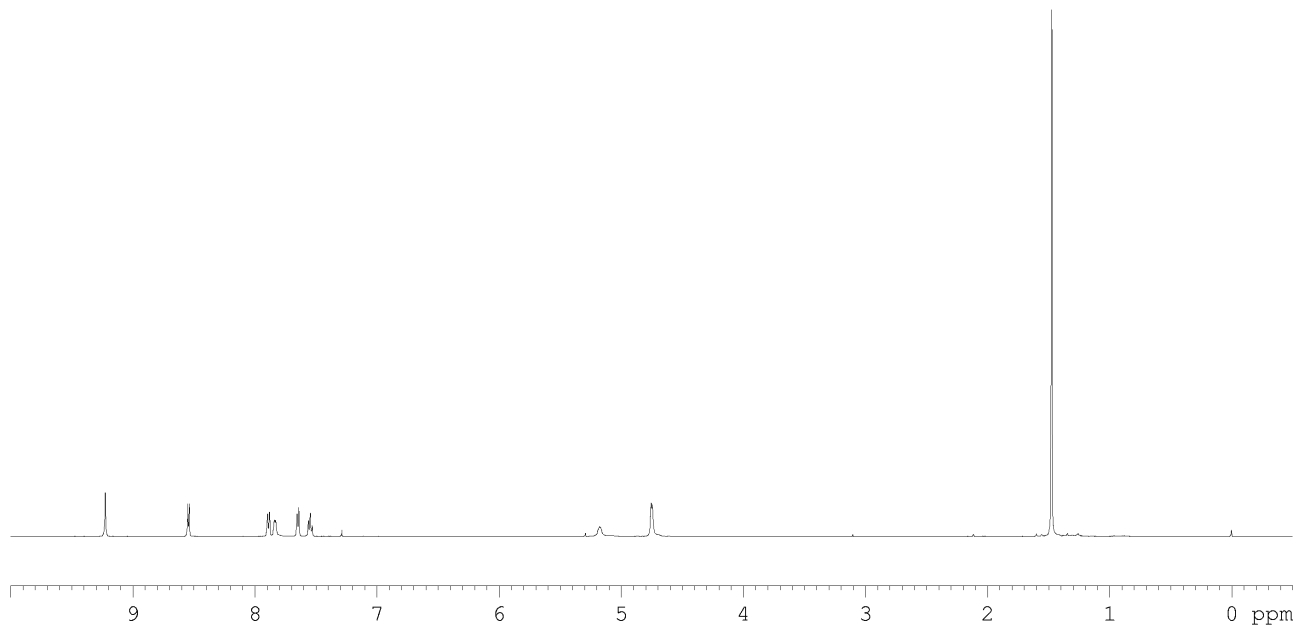
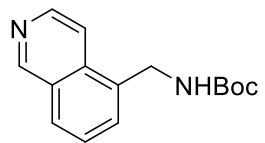




<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **5a**

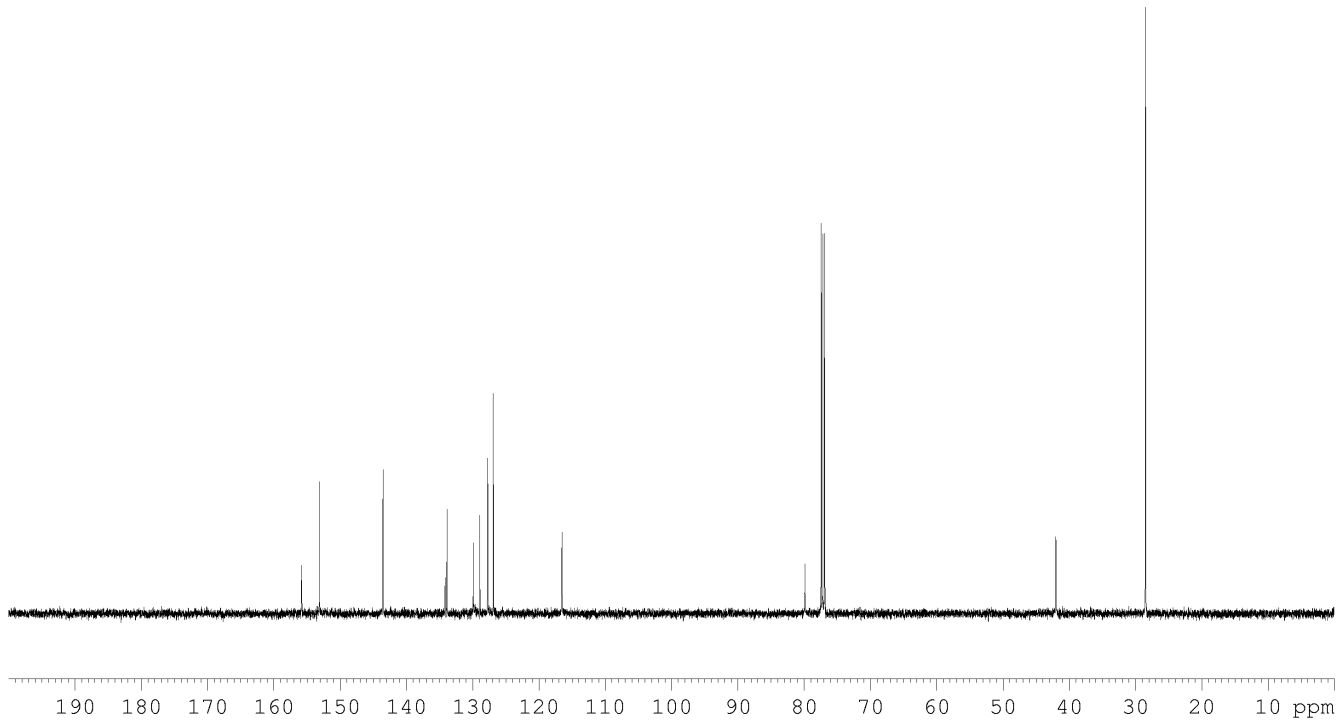
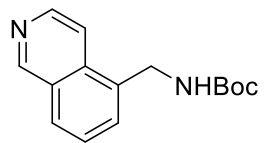


$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **5a**

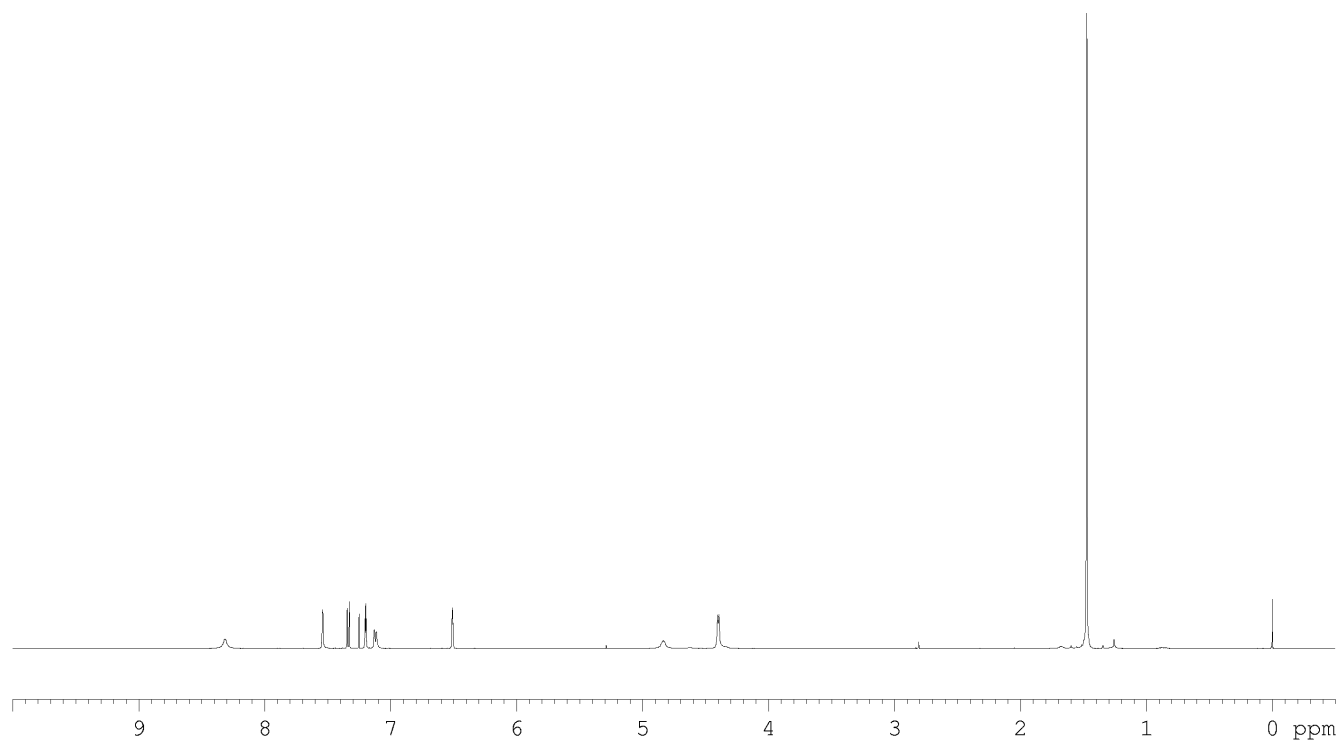
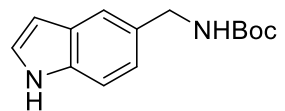


<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **5b**

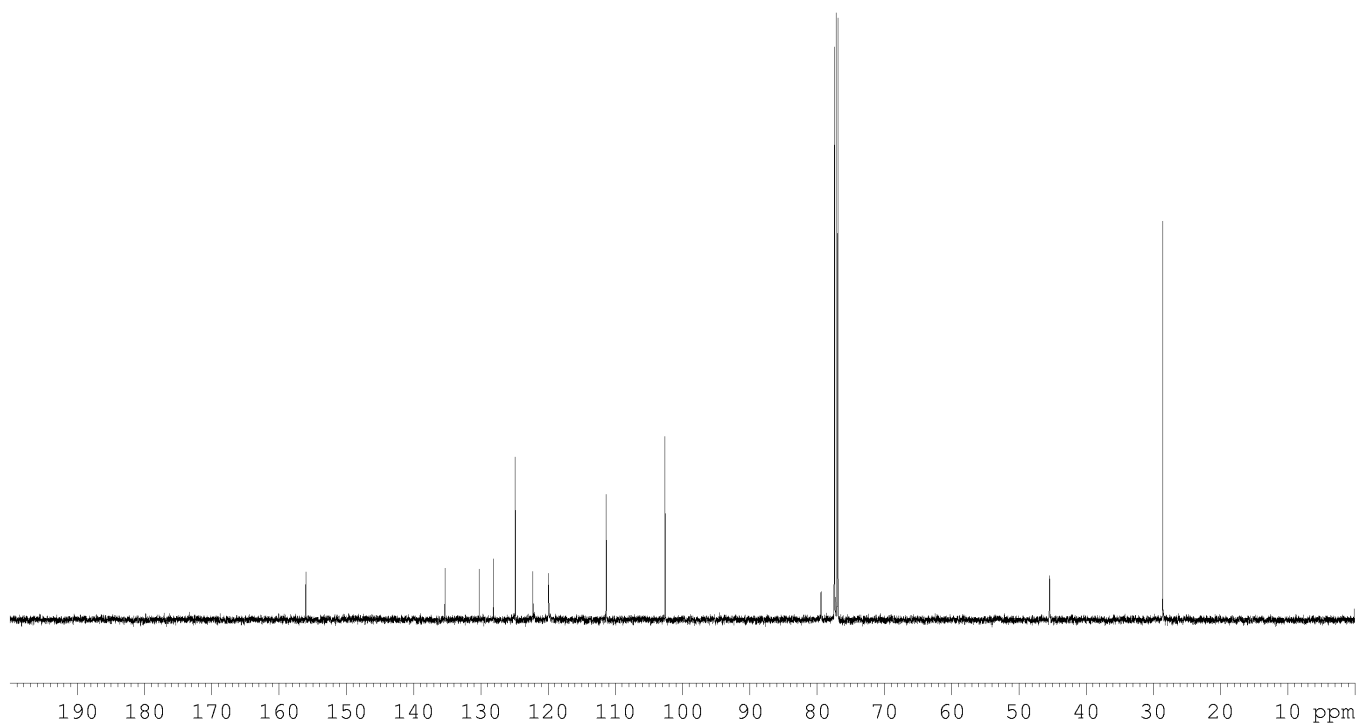
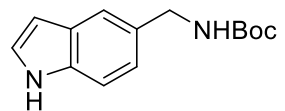
S67



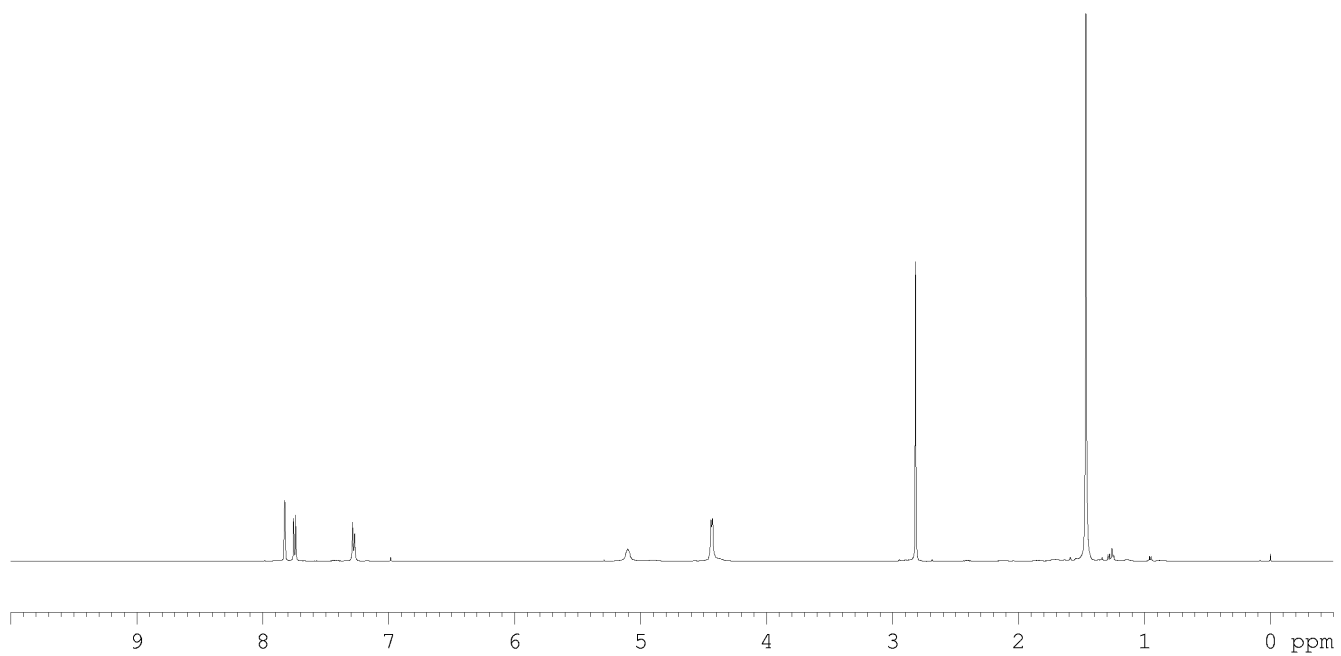
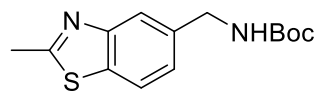
$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **5b**



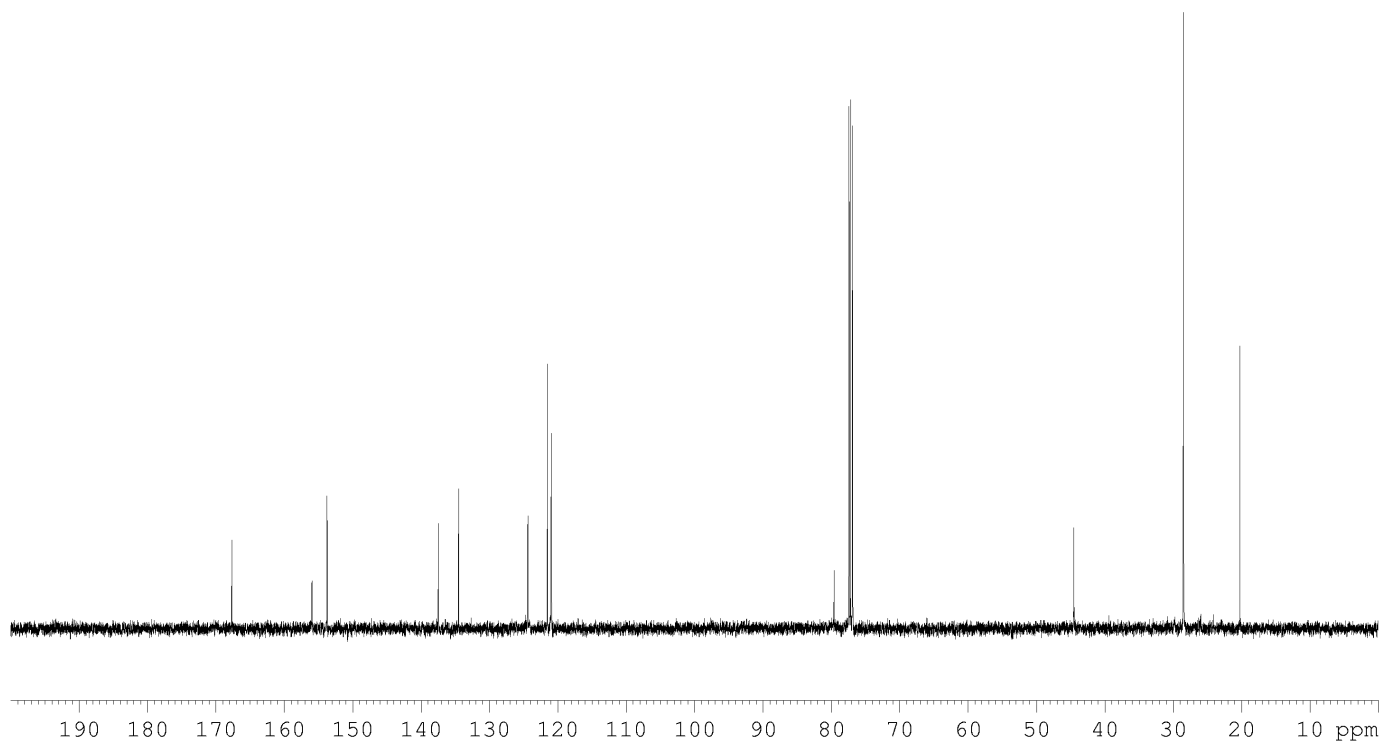
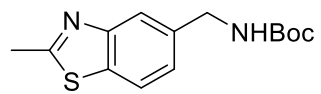
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **5c**



$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **5c**

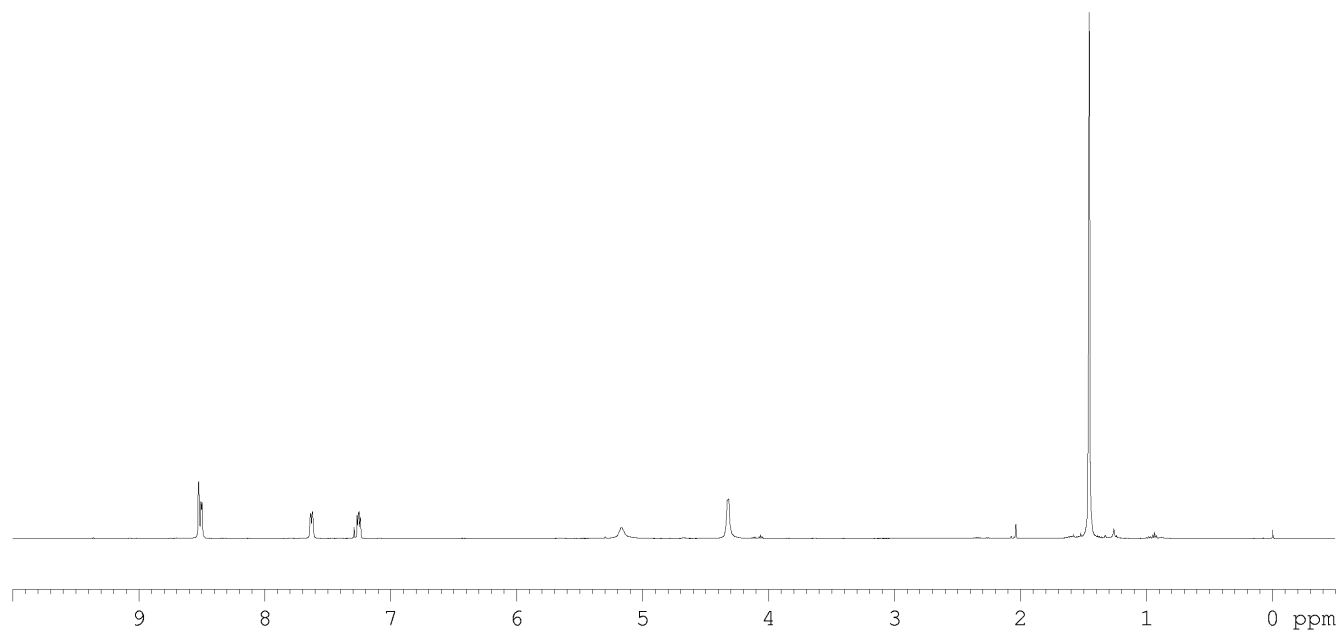
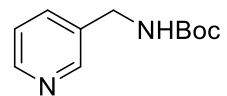


<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **5d**

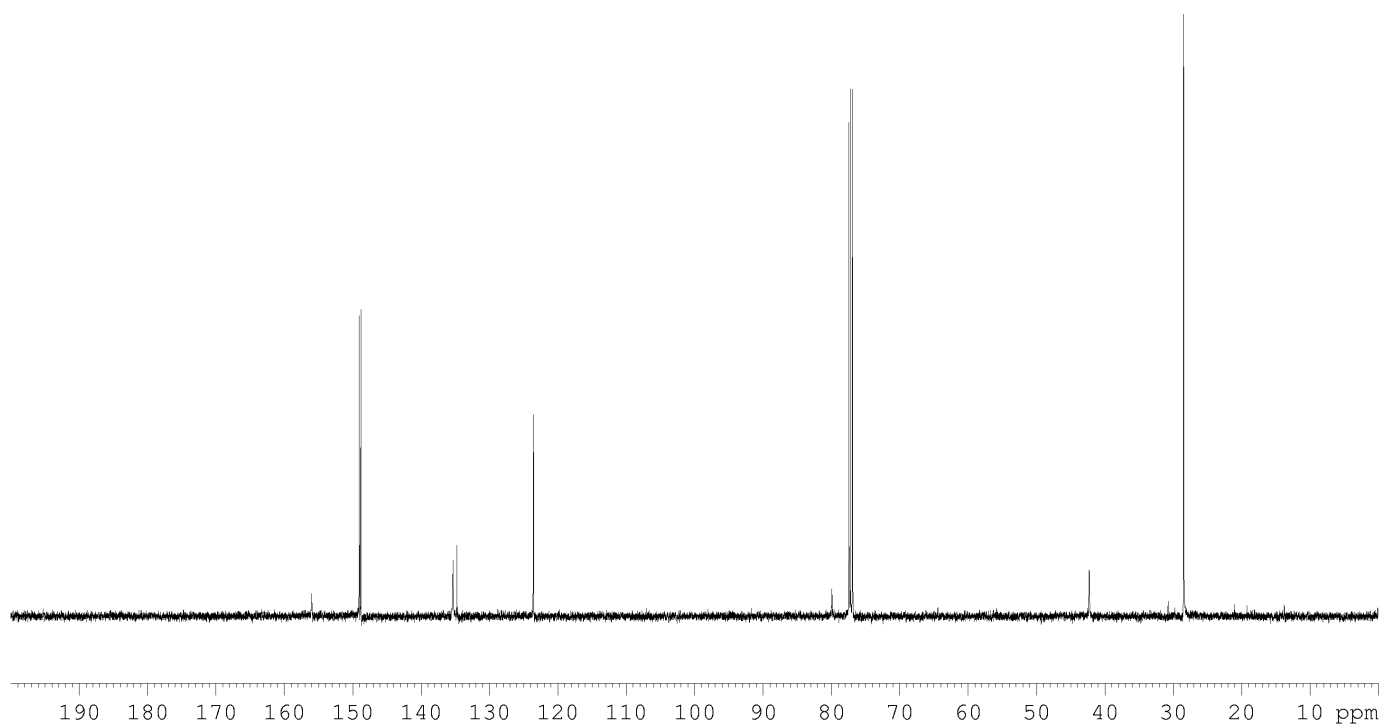
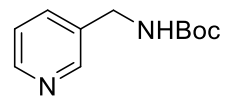


$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **5d**

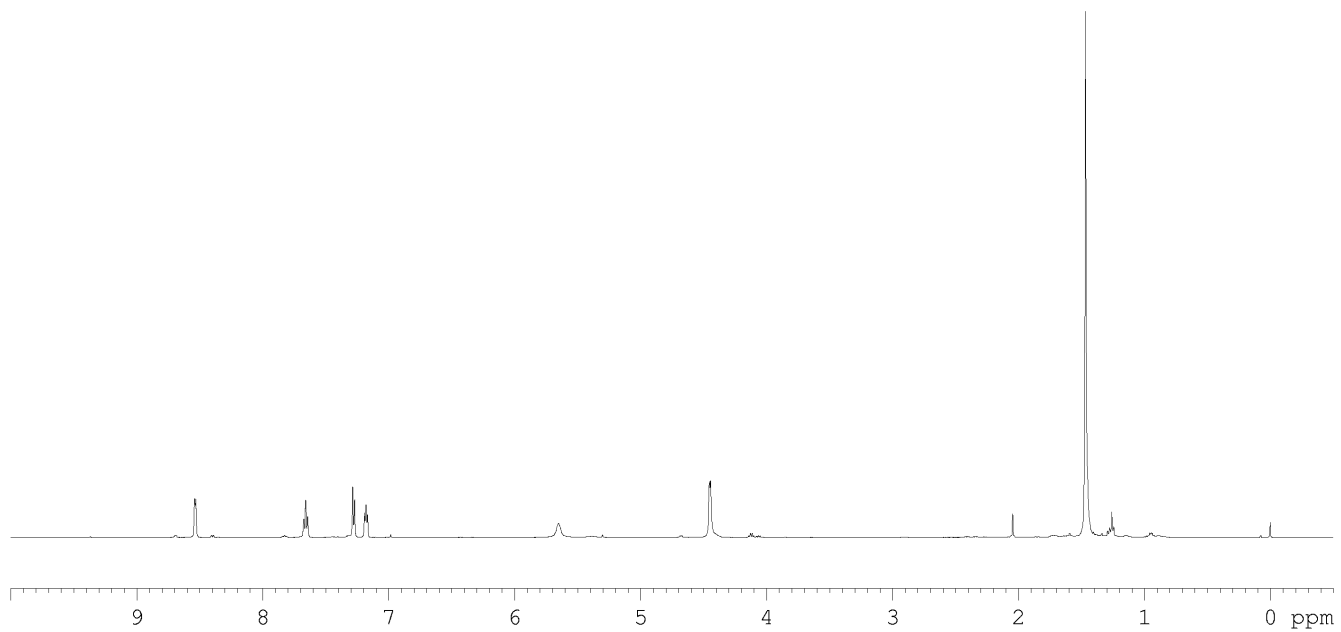
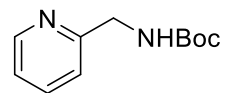




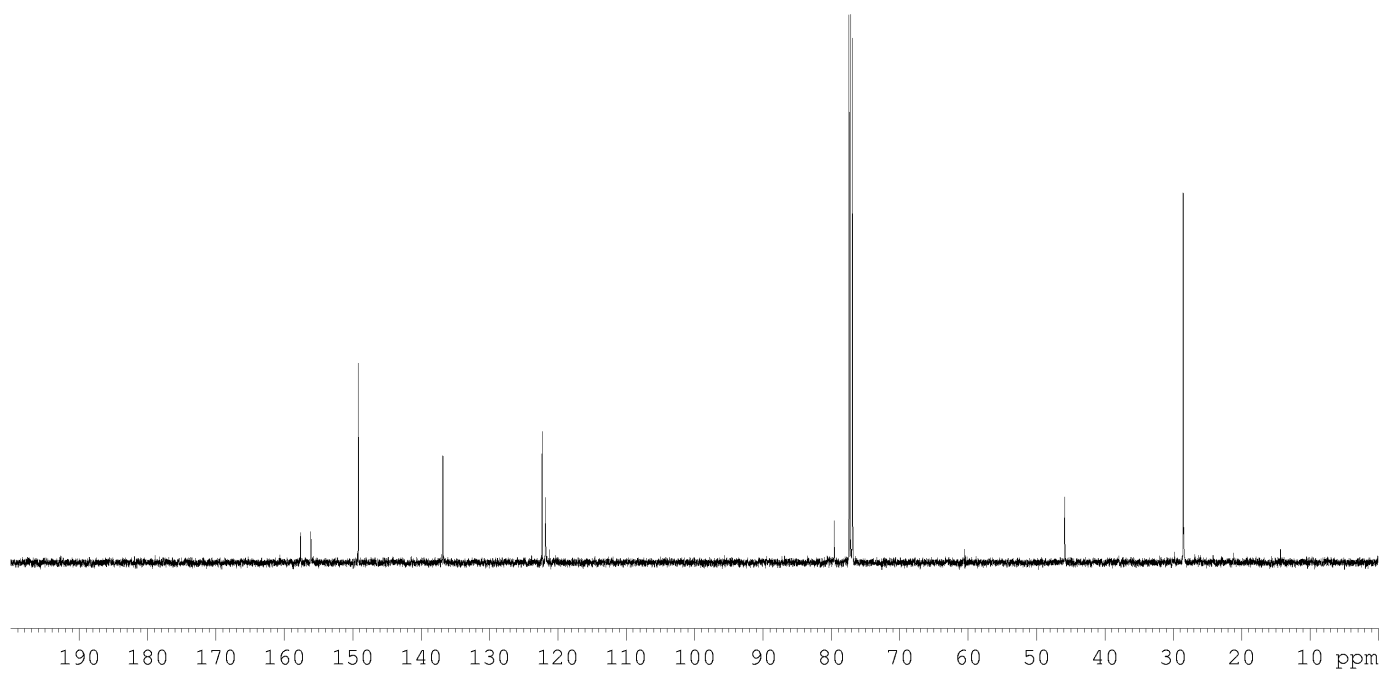
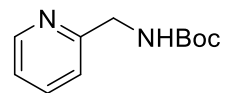
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **5e**



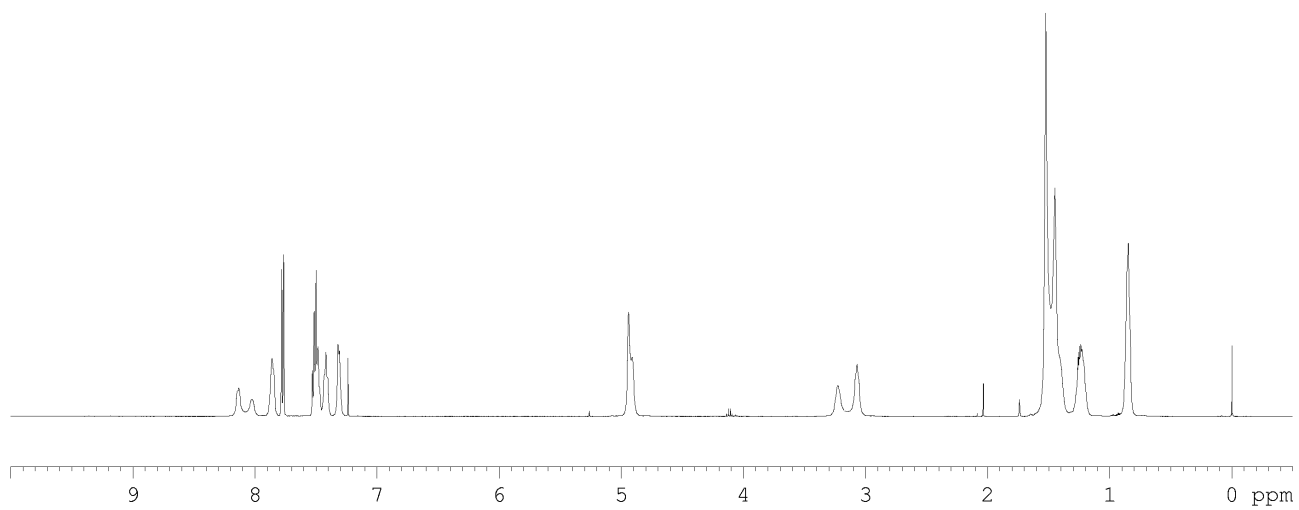
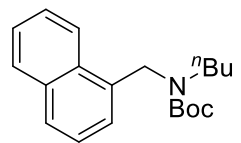
$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **5e**



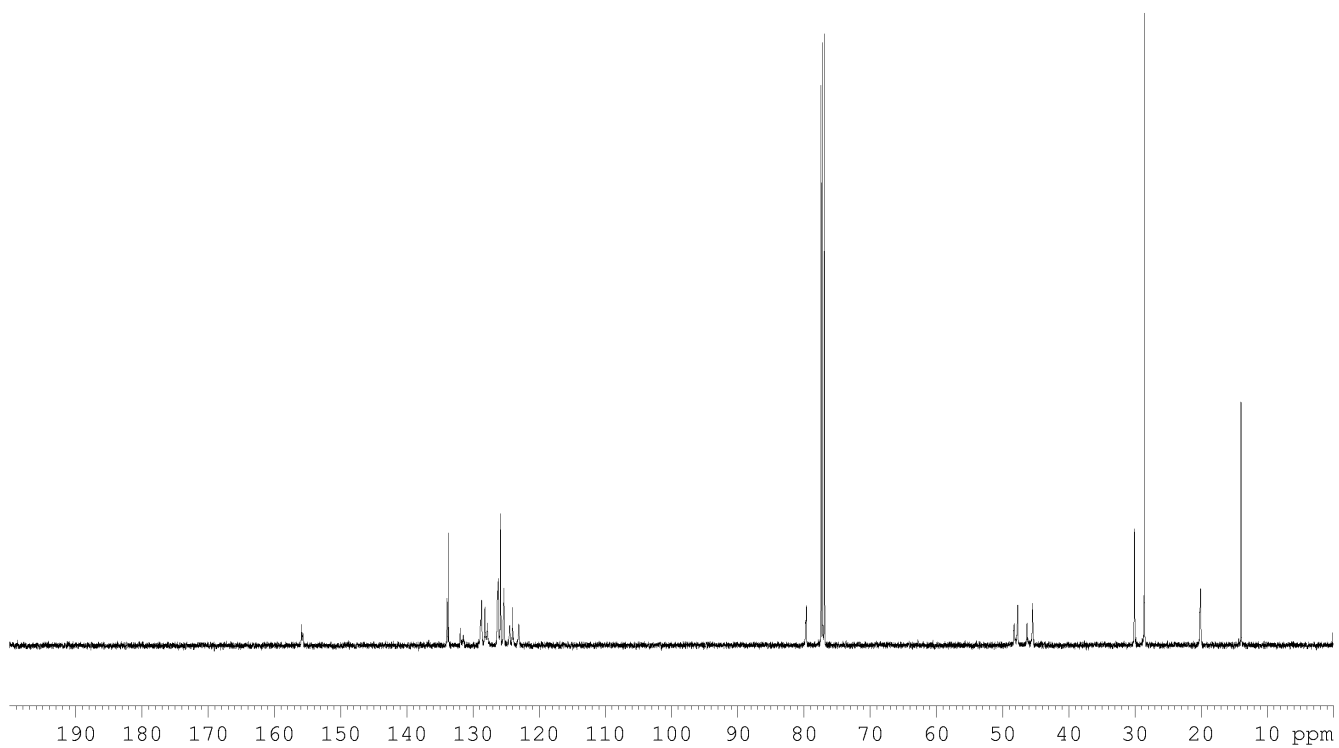
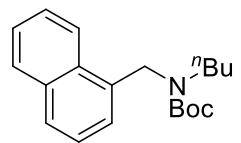
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **5f**



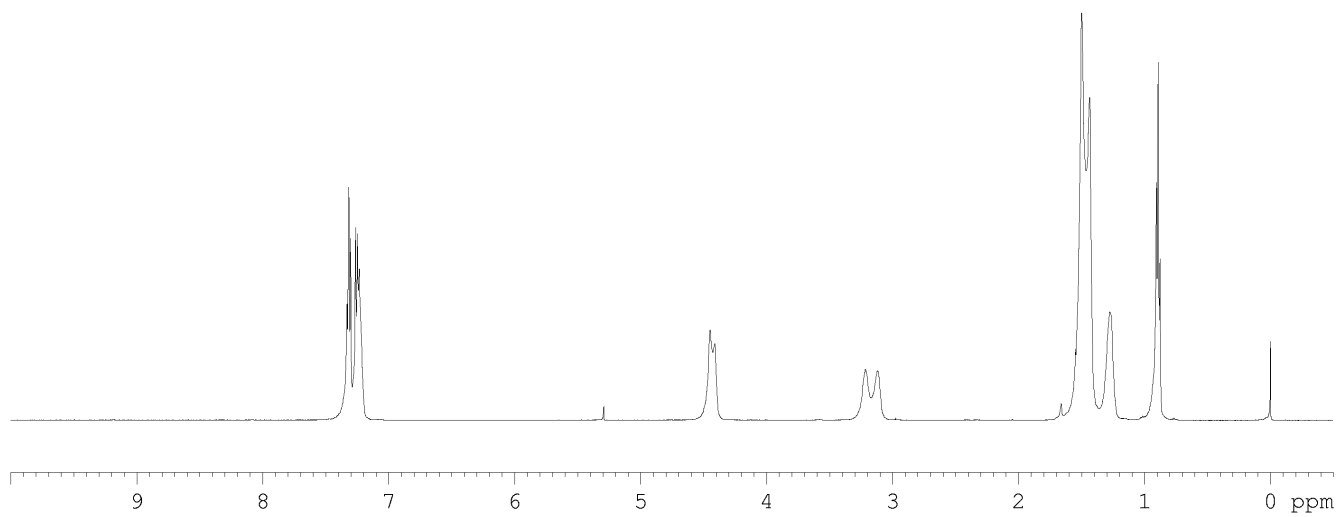
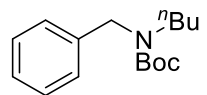
$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **5f**



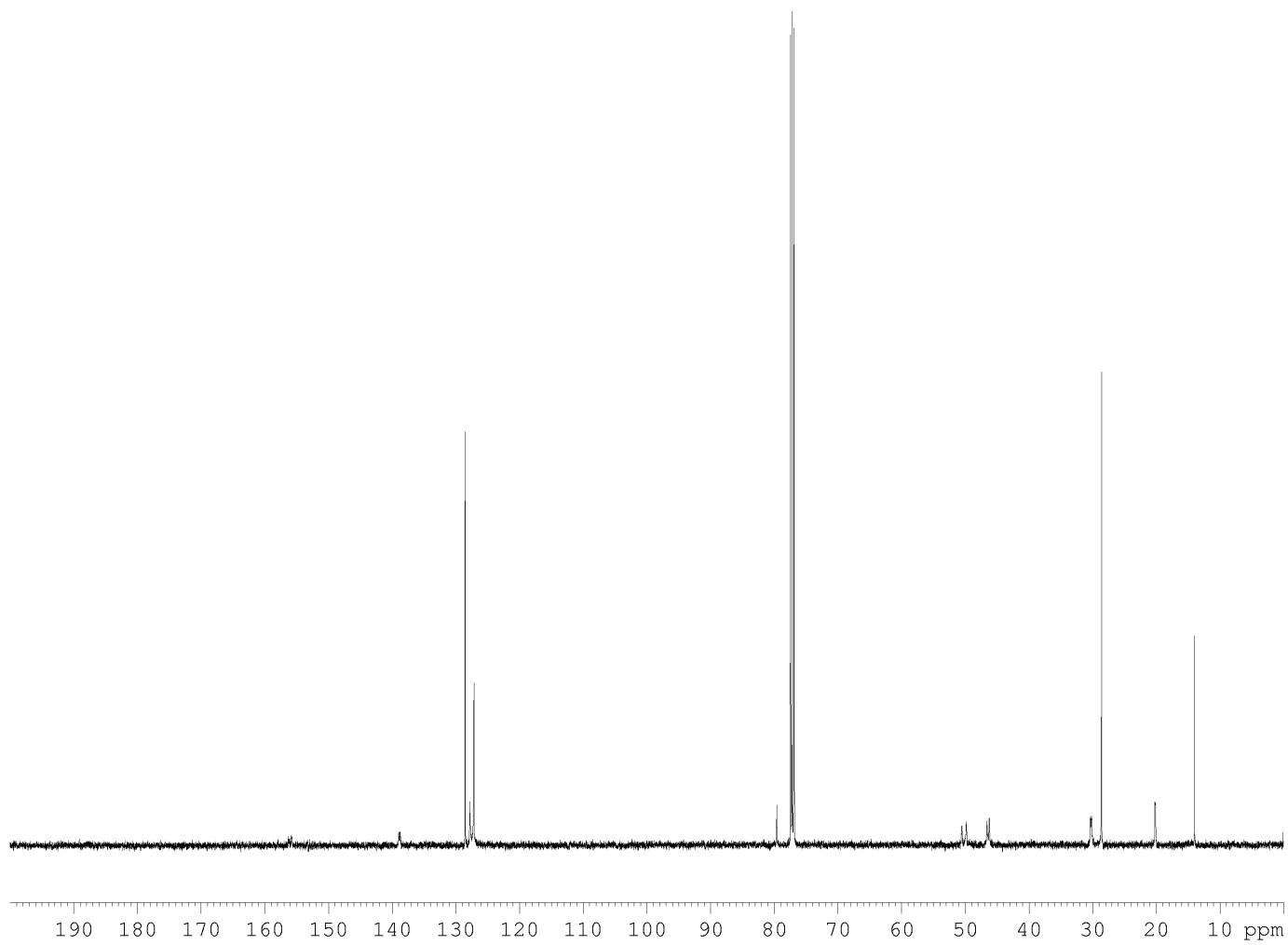
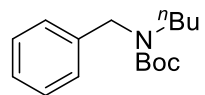
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) Spectrum of **7a**



$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **7a**



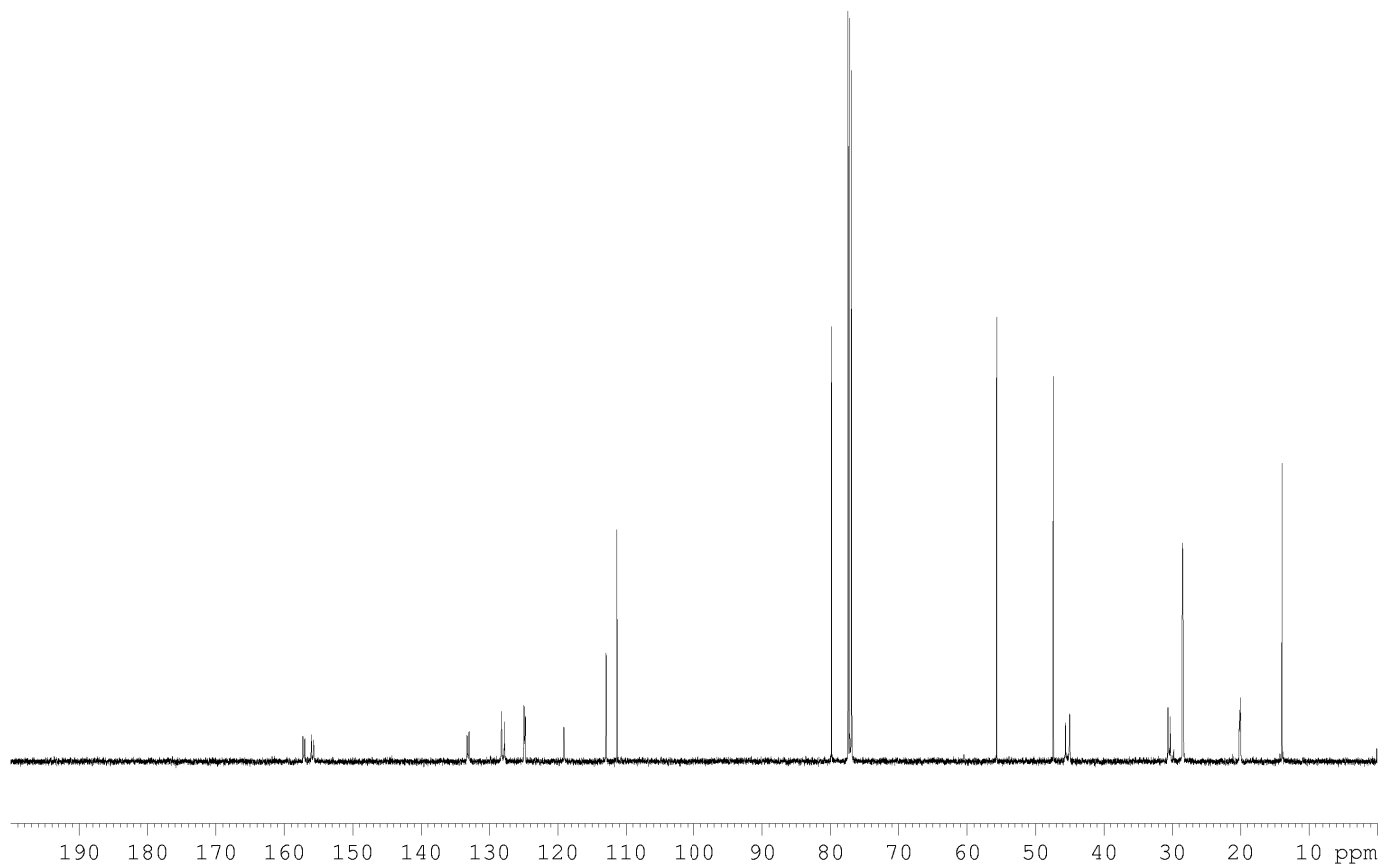
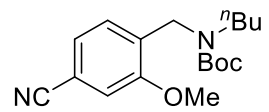
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) Spectrum of **7b**



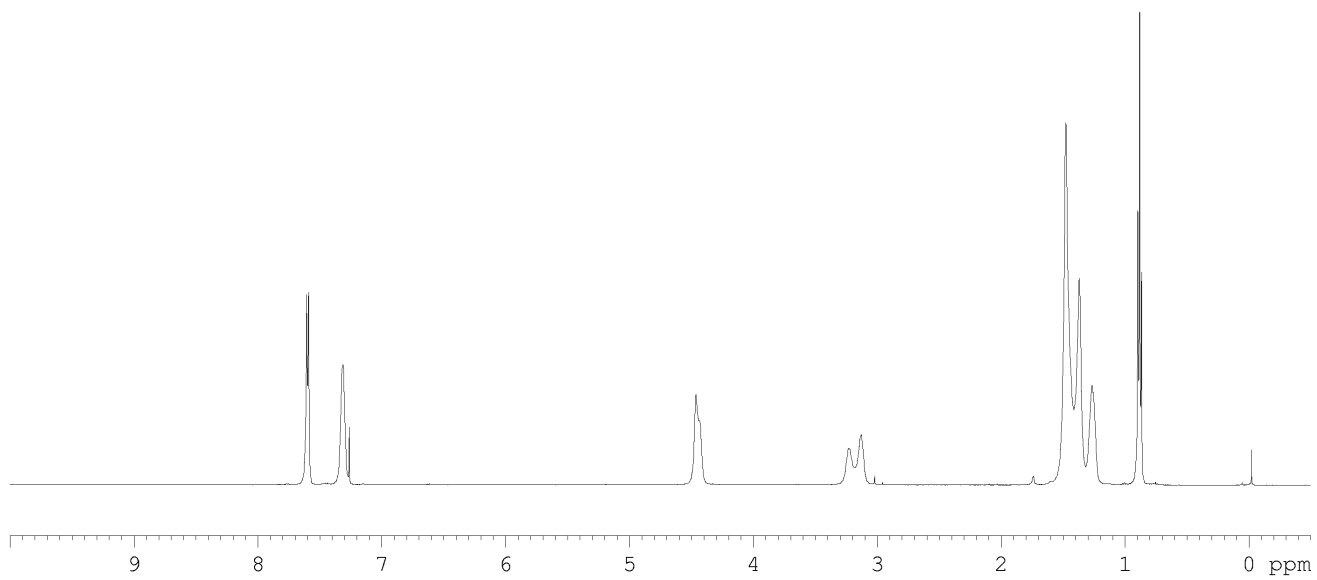
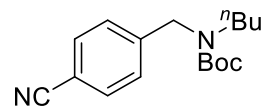
<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum of **7b**



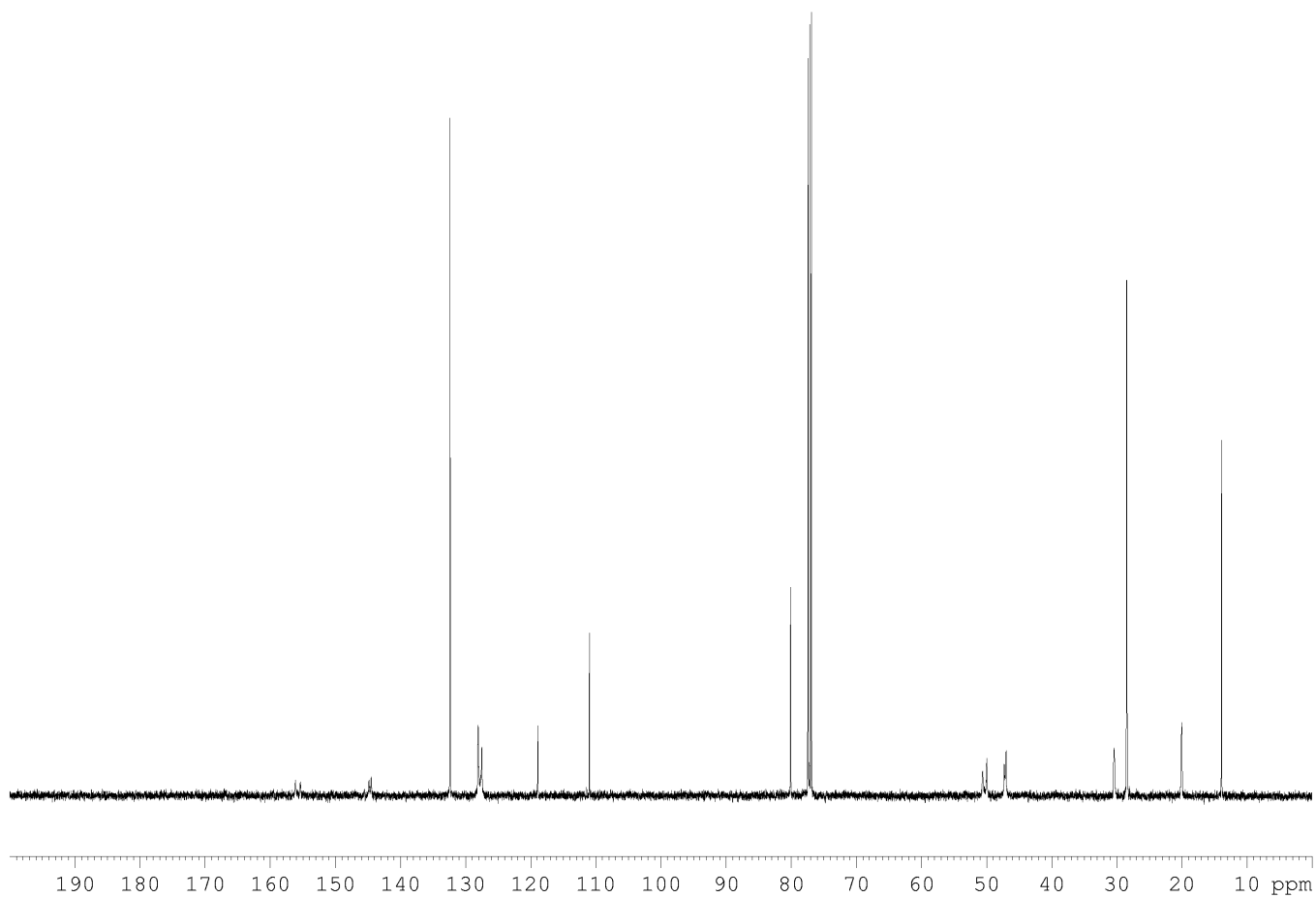
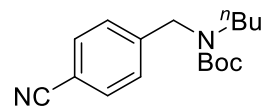




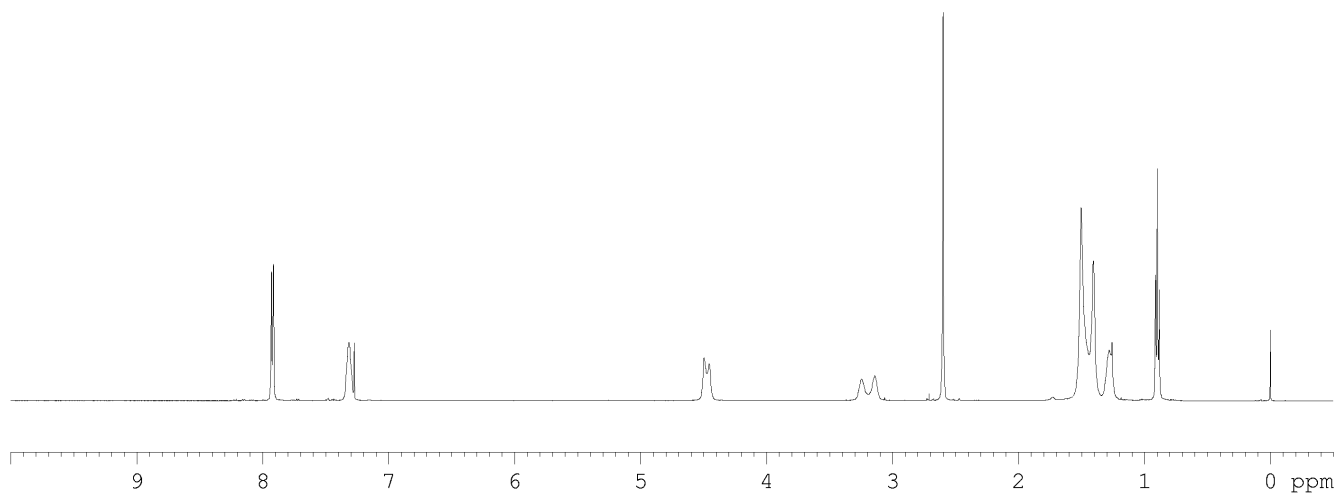
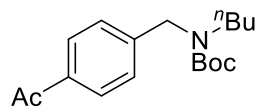
<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum of **7d**



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) Spectrum of **7e**

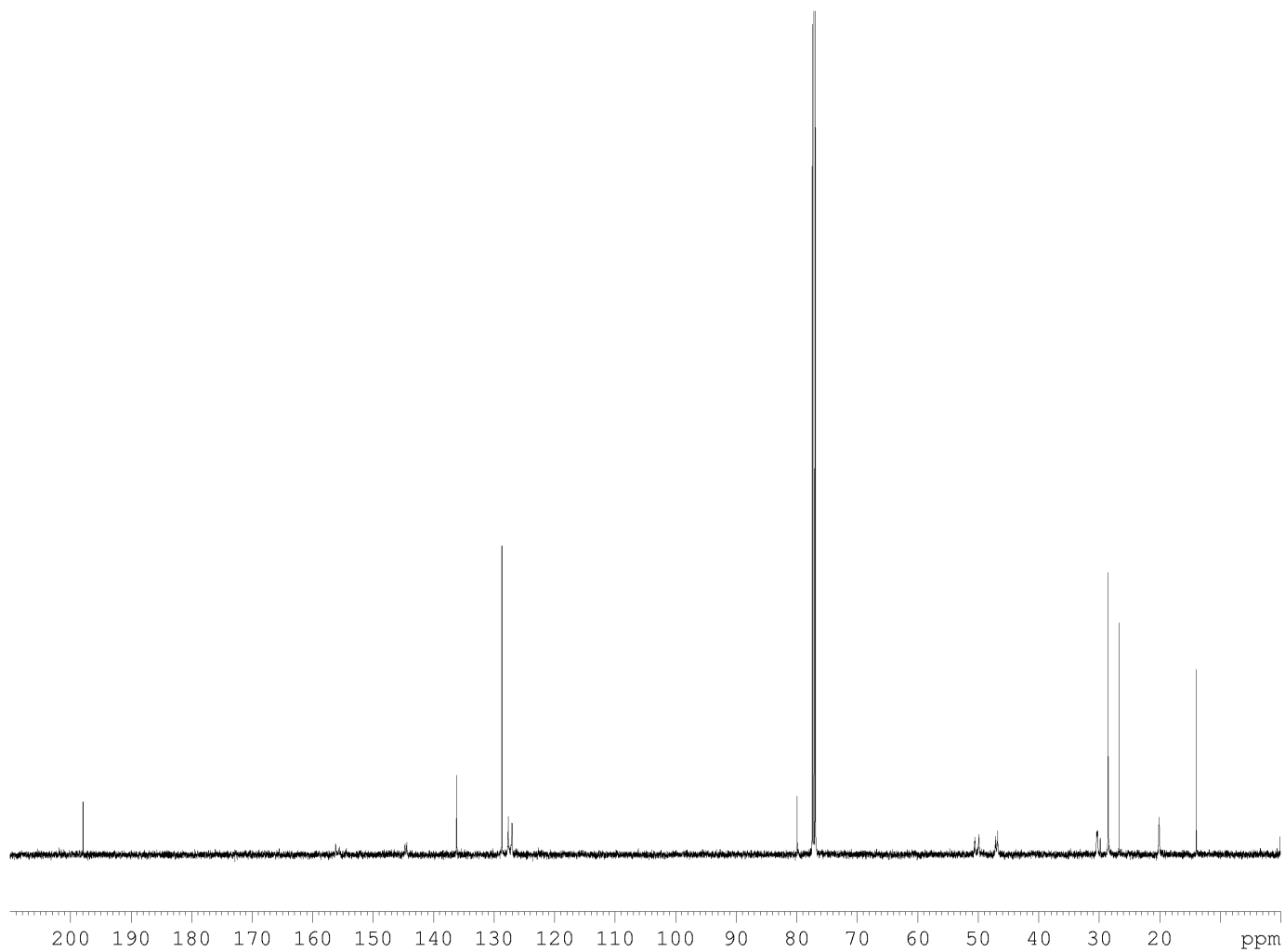
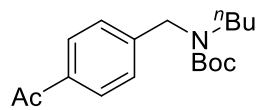


<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum of 7e

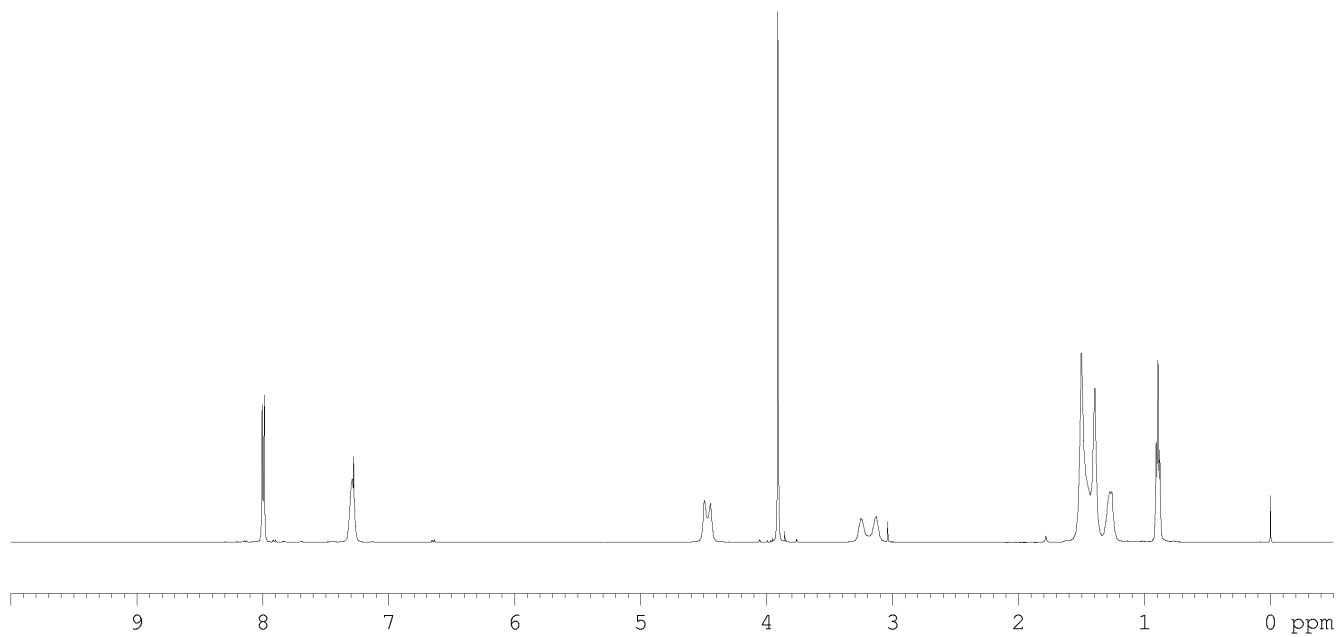
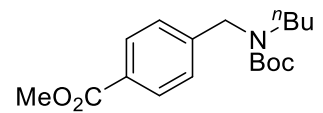


<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **7f**

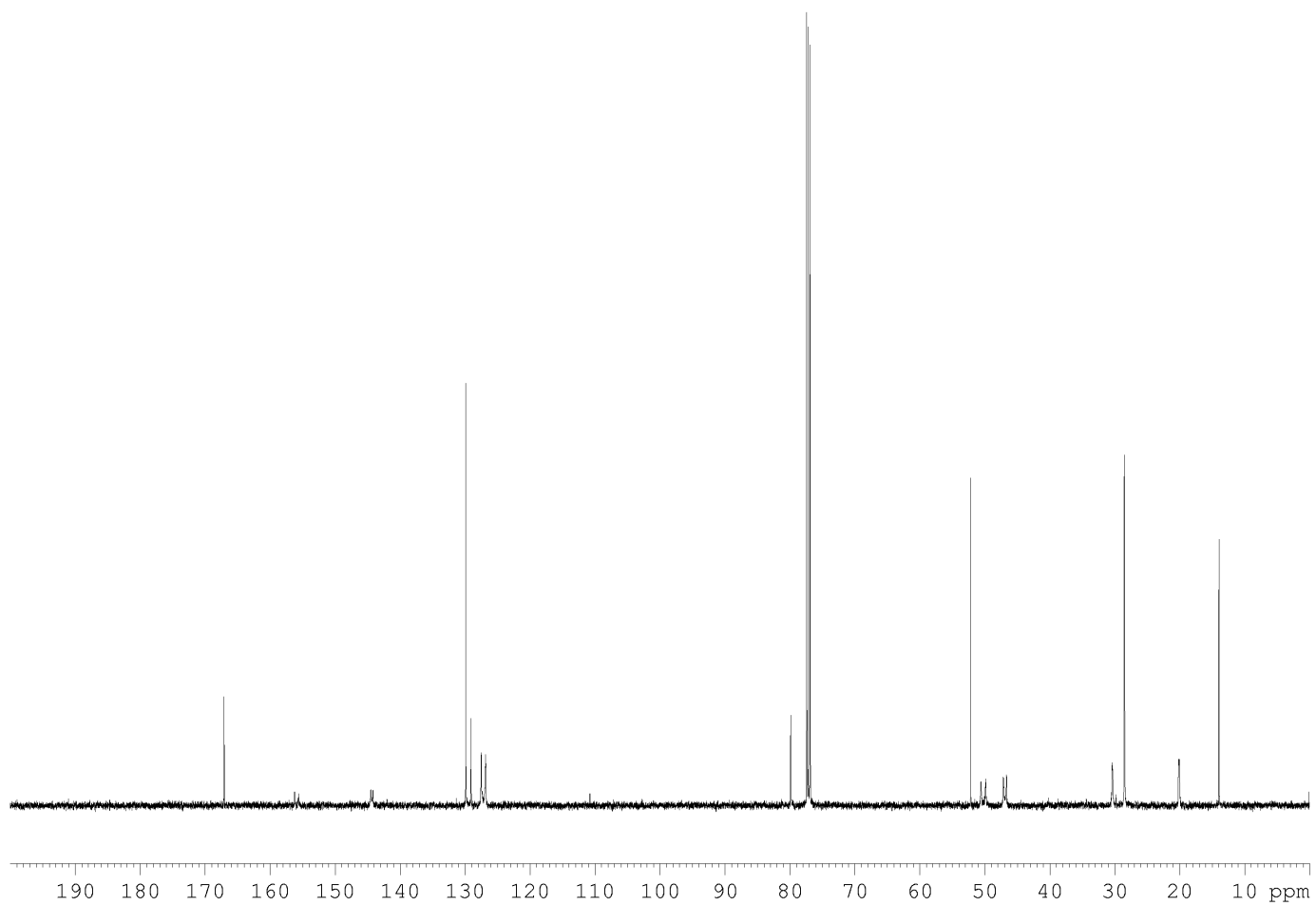
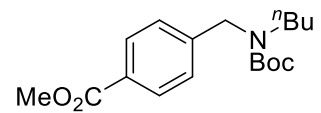
S85



<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum of **7f**

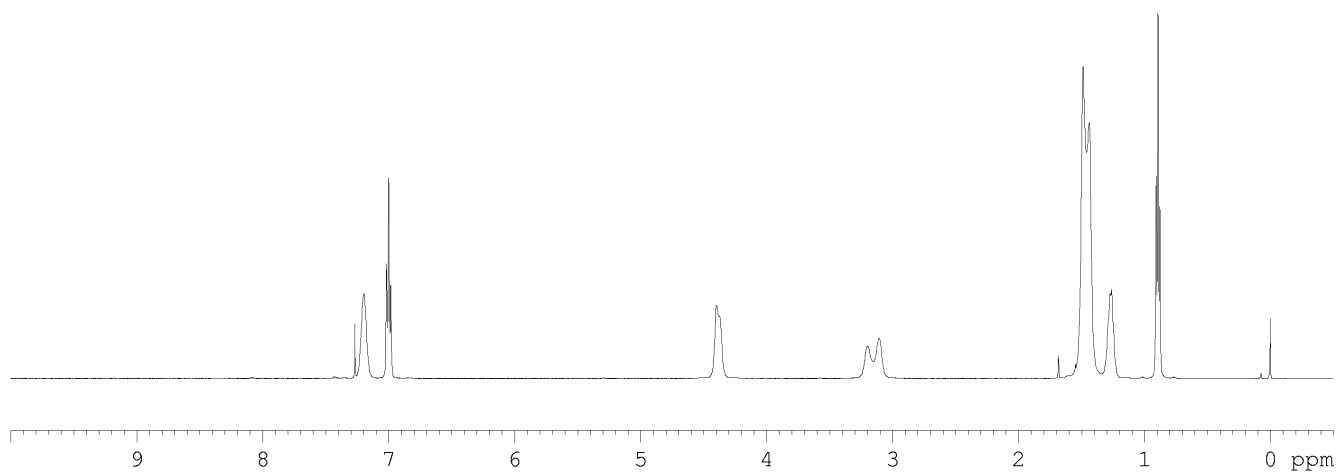
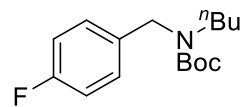


<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **7g**  
S87

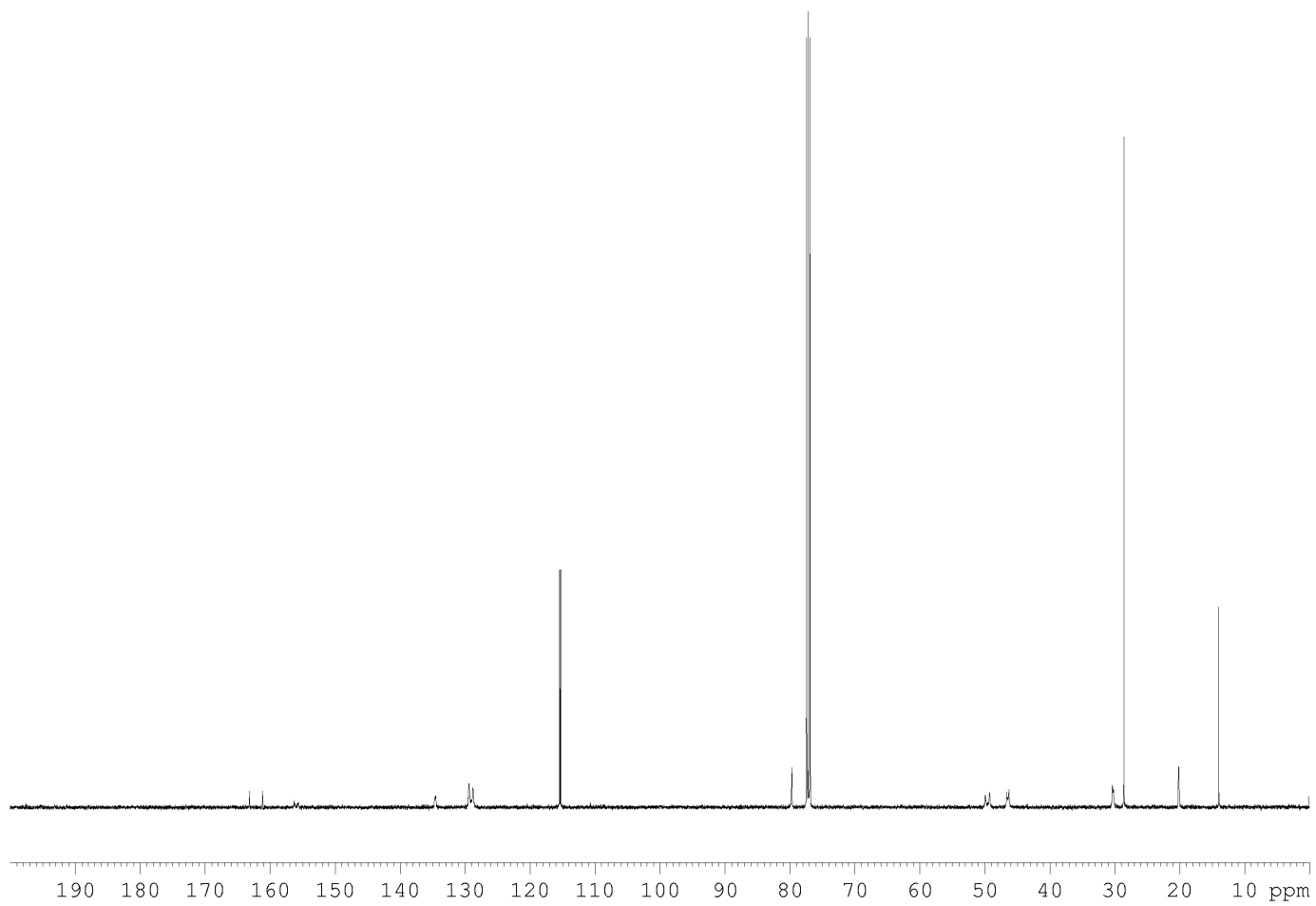
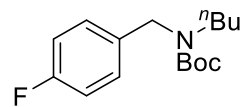


<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum of **7g**

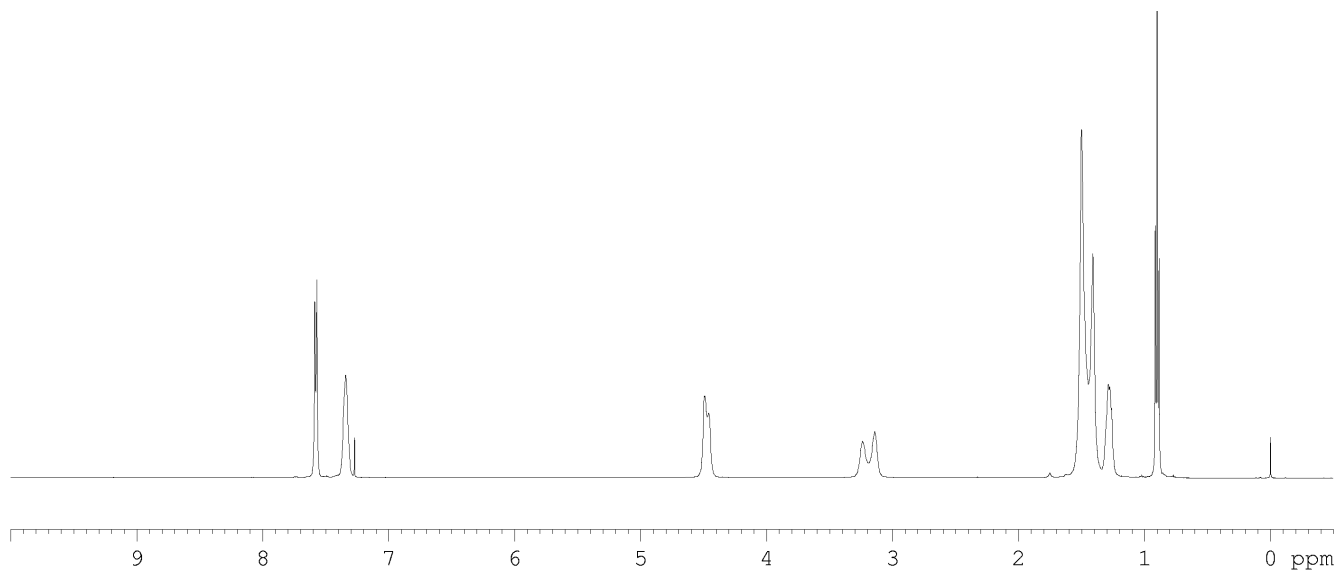
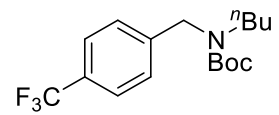




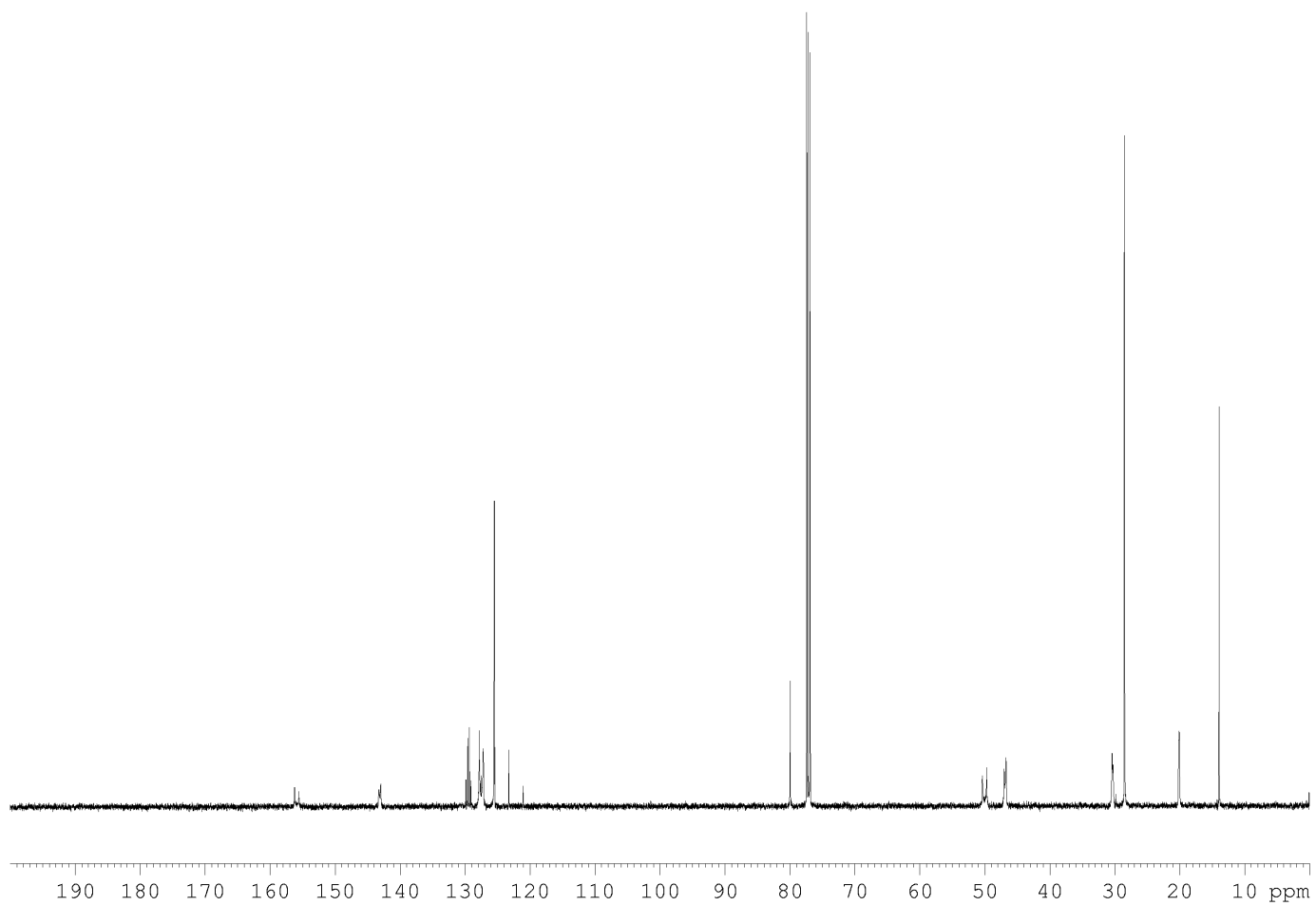
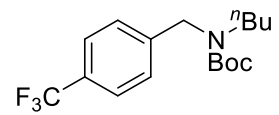
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **7h**  
S89



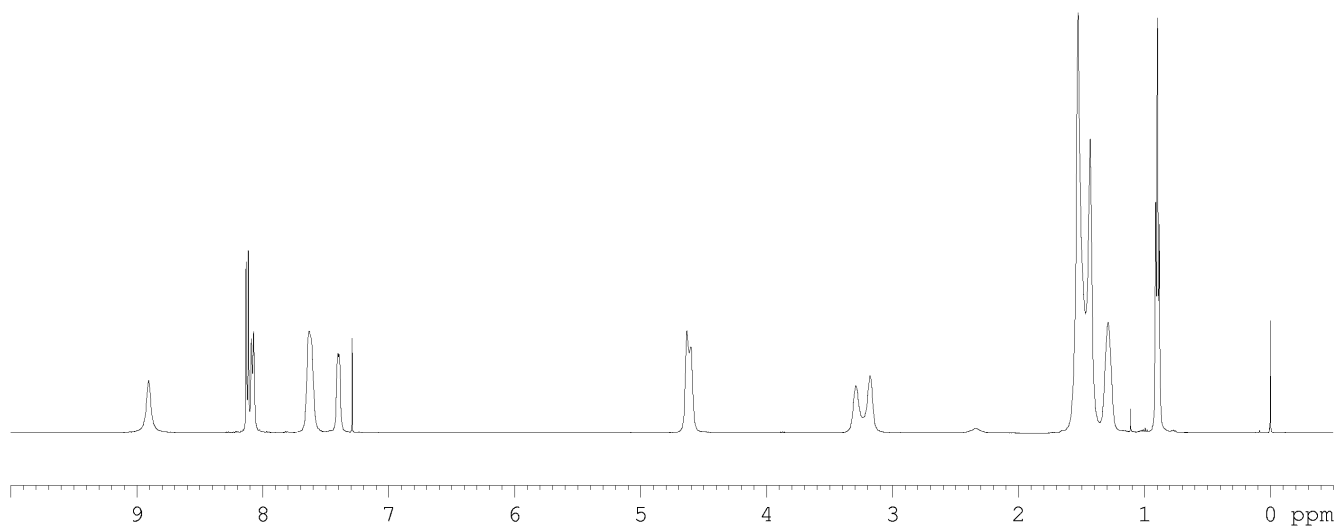
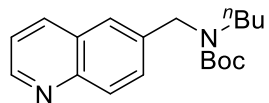
<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum of **7h**



$^1H$  NMR (500 MHz,  $CDCl_3$ ) Spectrum of **7i**  
S91

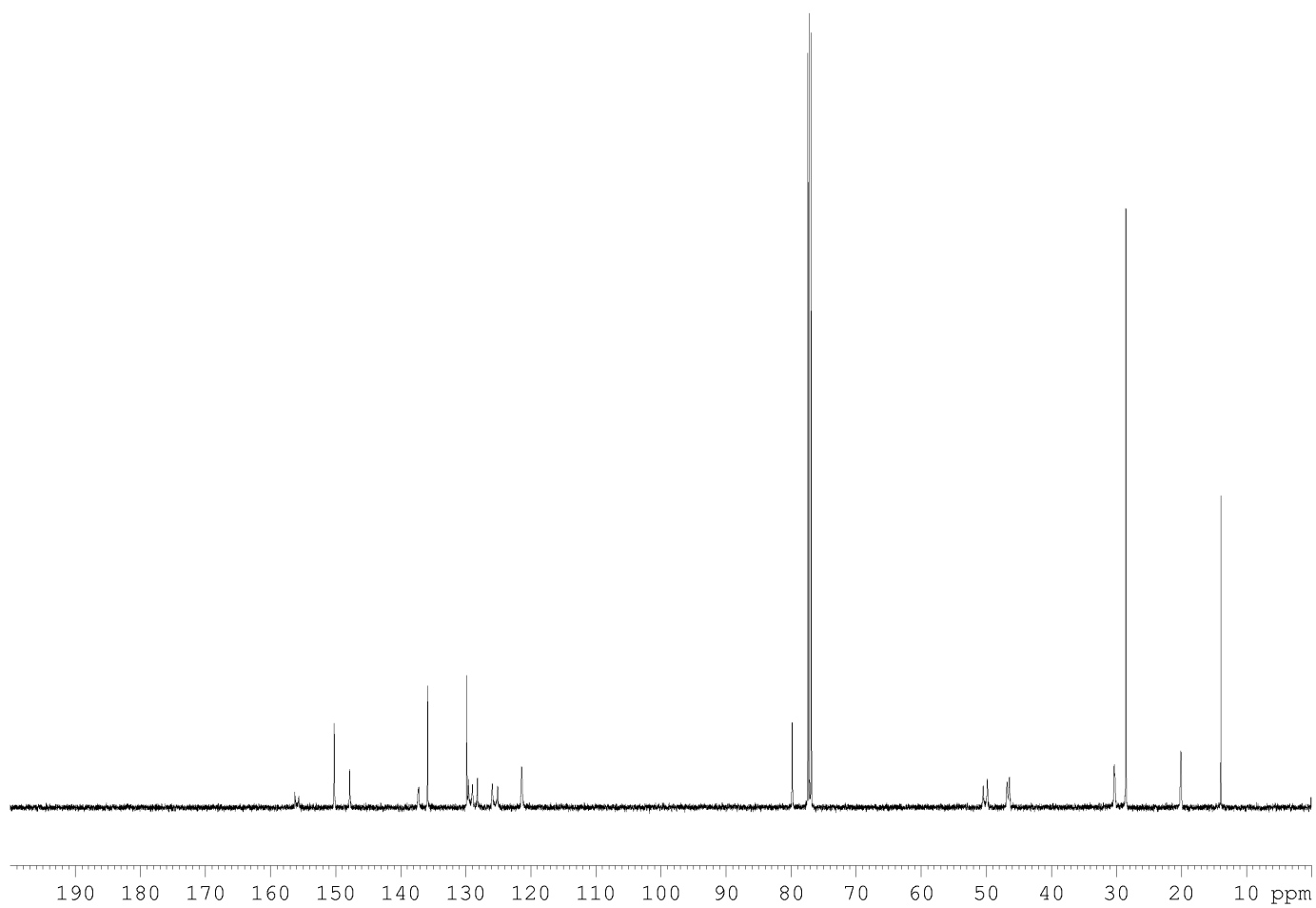
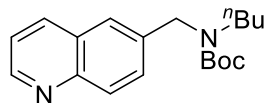


<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum of **7i**

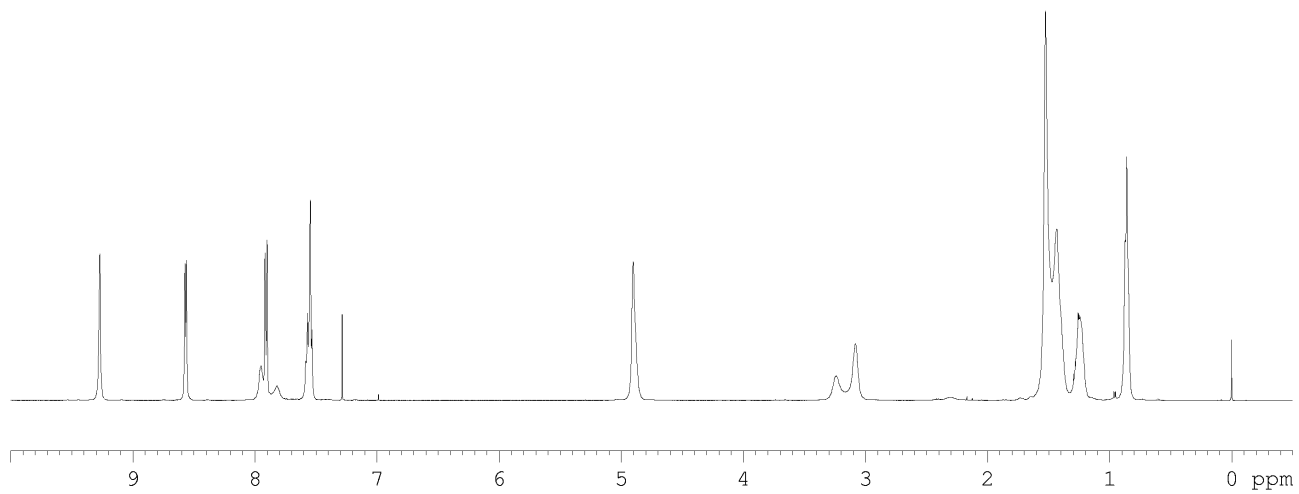
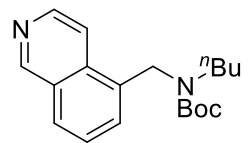


<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **8a**

S93

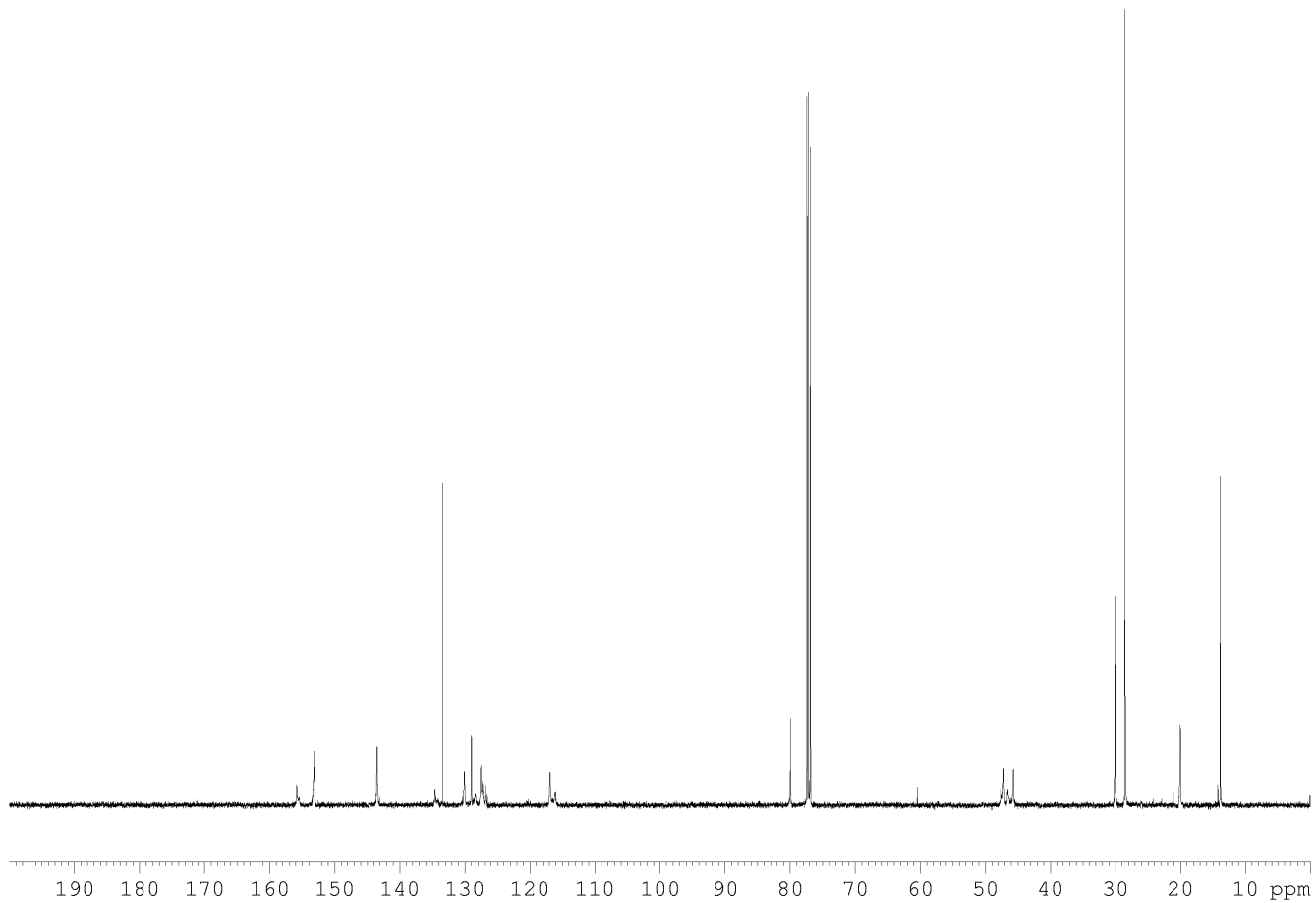
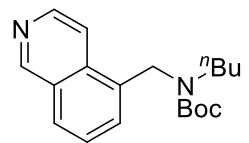


$^{13}\text{C}$  NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum of **8a**



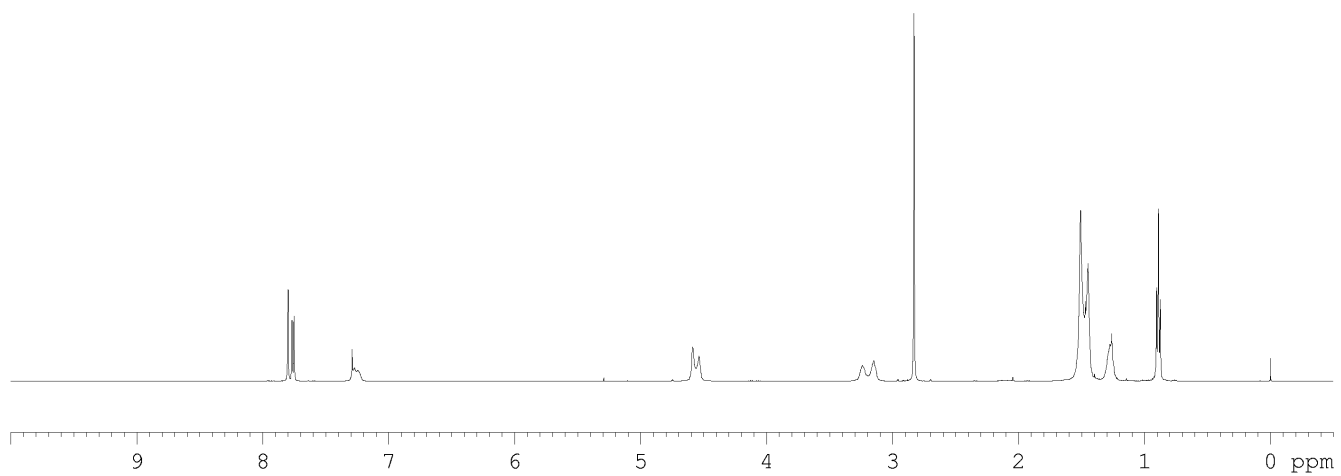
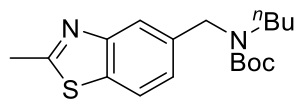
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **8b**

S95

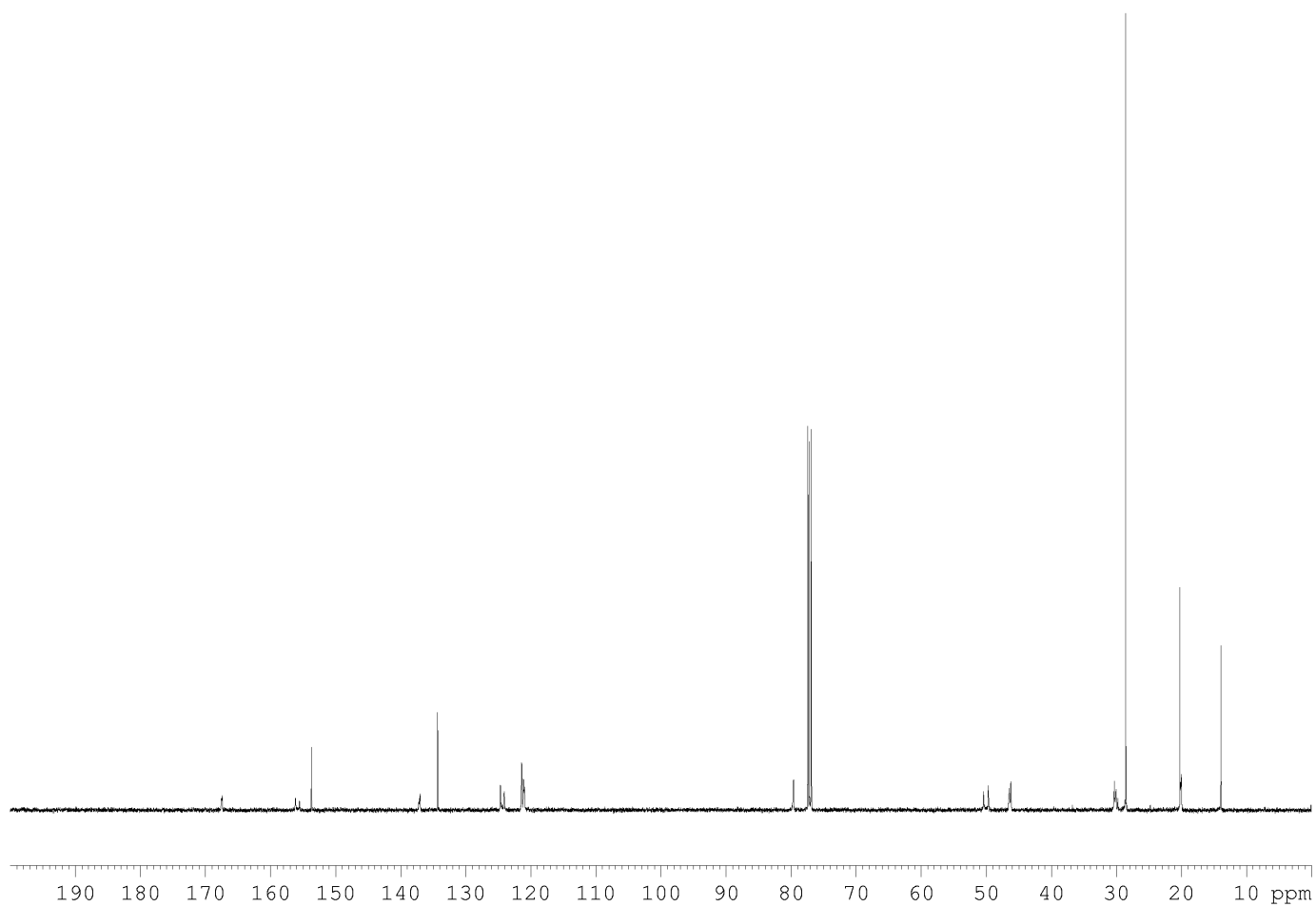
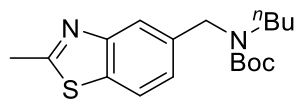


$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **8b**

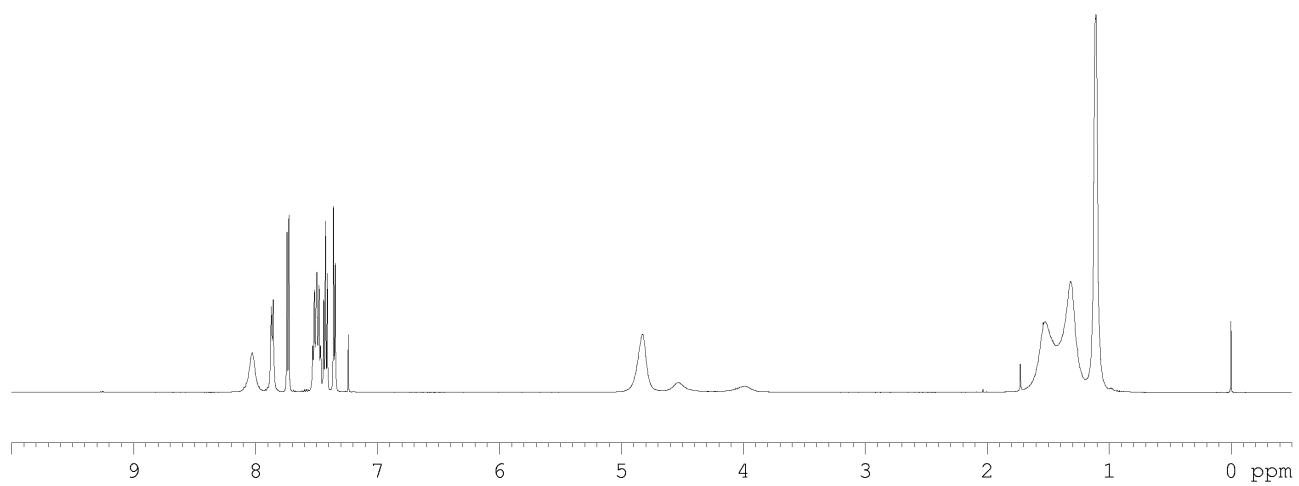
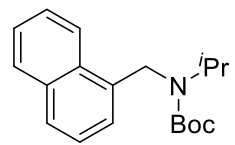




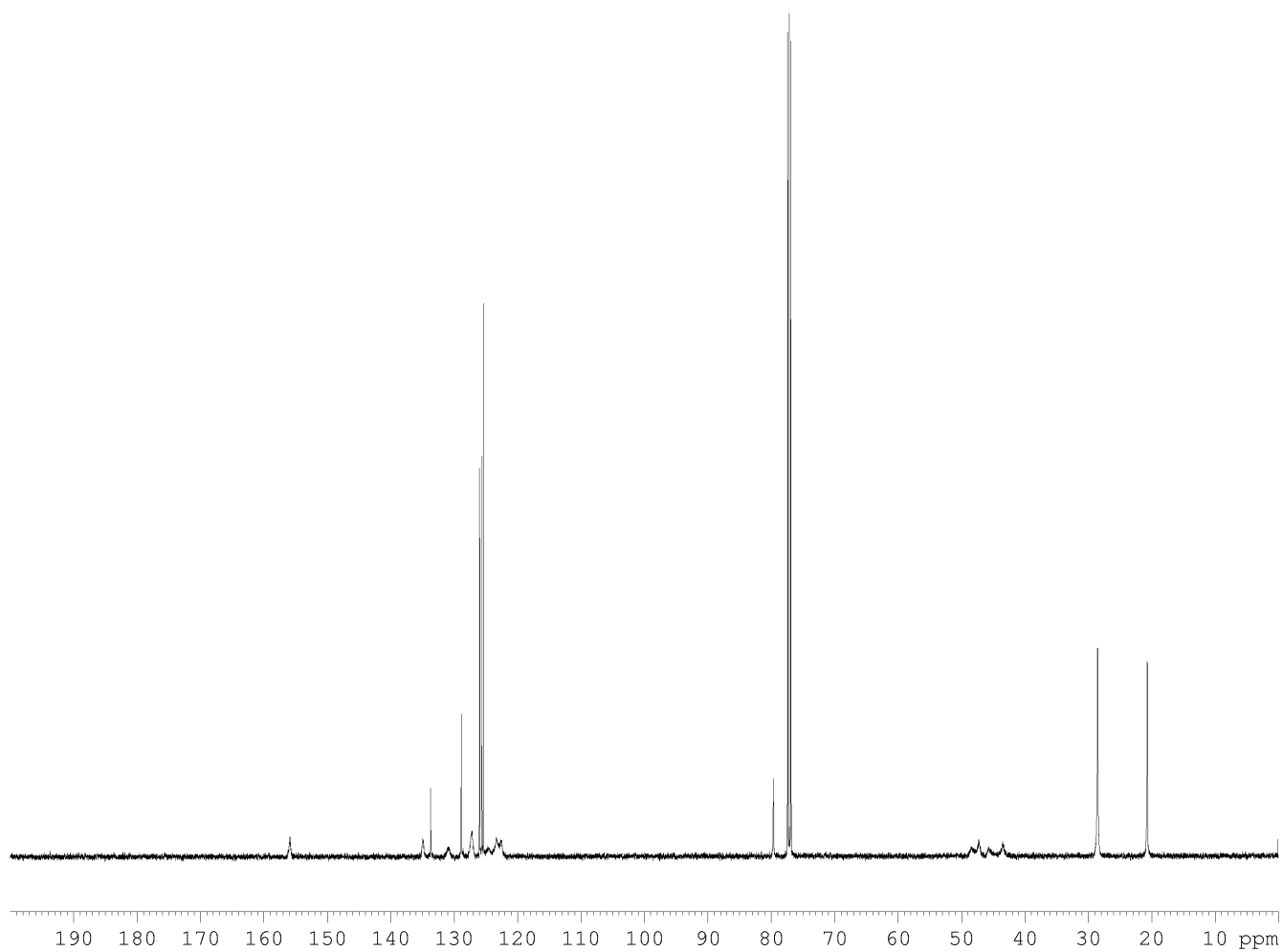
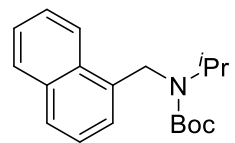
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **8c**



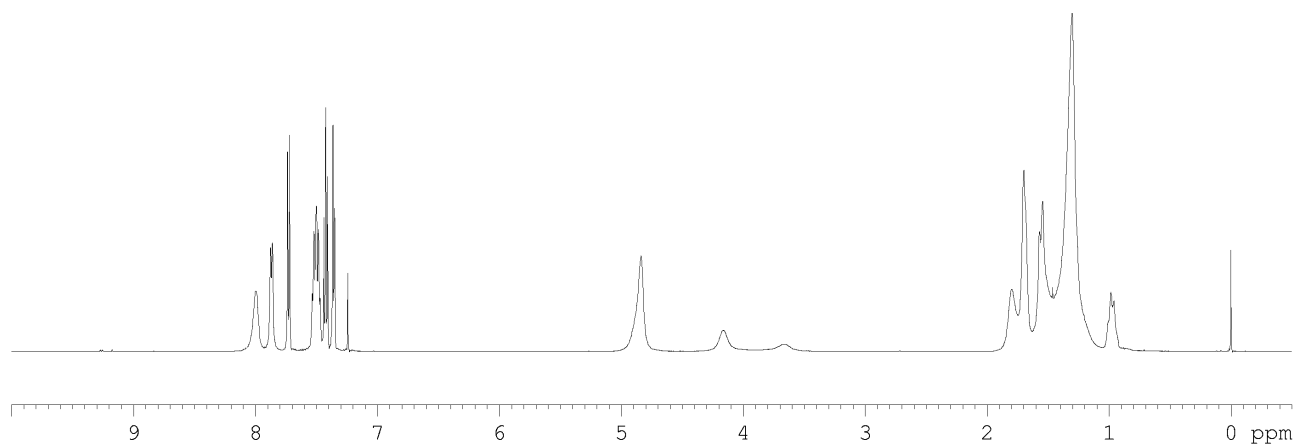
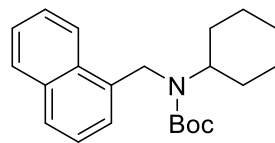
<sup>13</sup>C NMR (125.8 MHz, CDCl<sub>3</sub>) Spectrum of **8c**



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **10a**

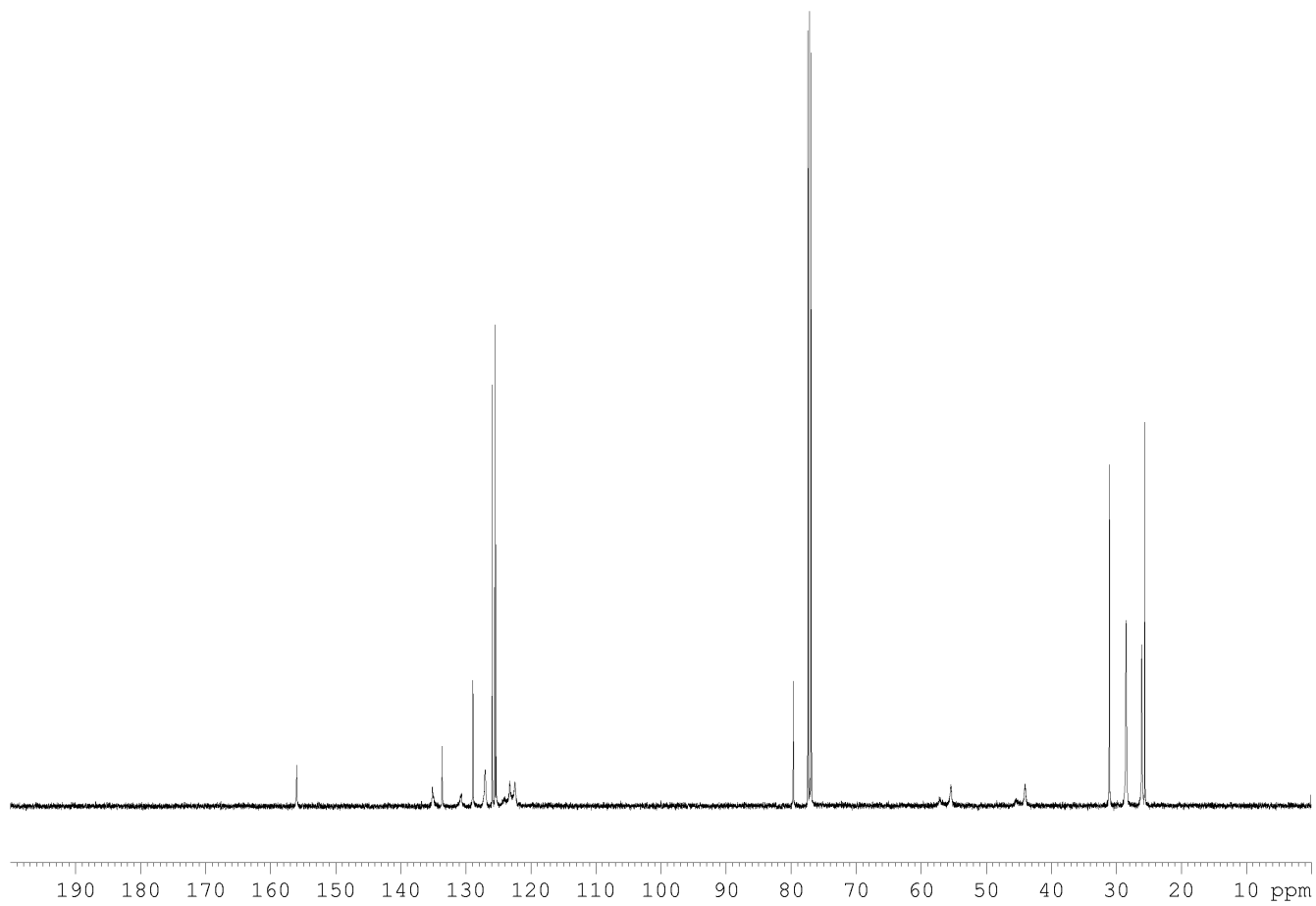
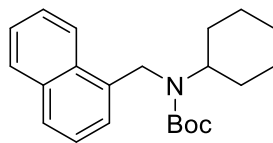


$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **10a**

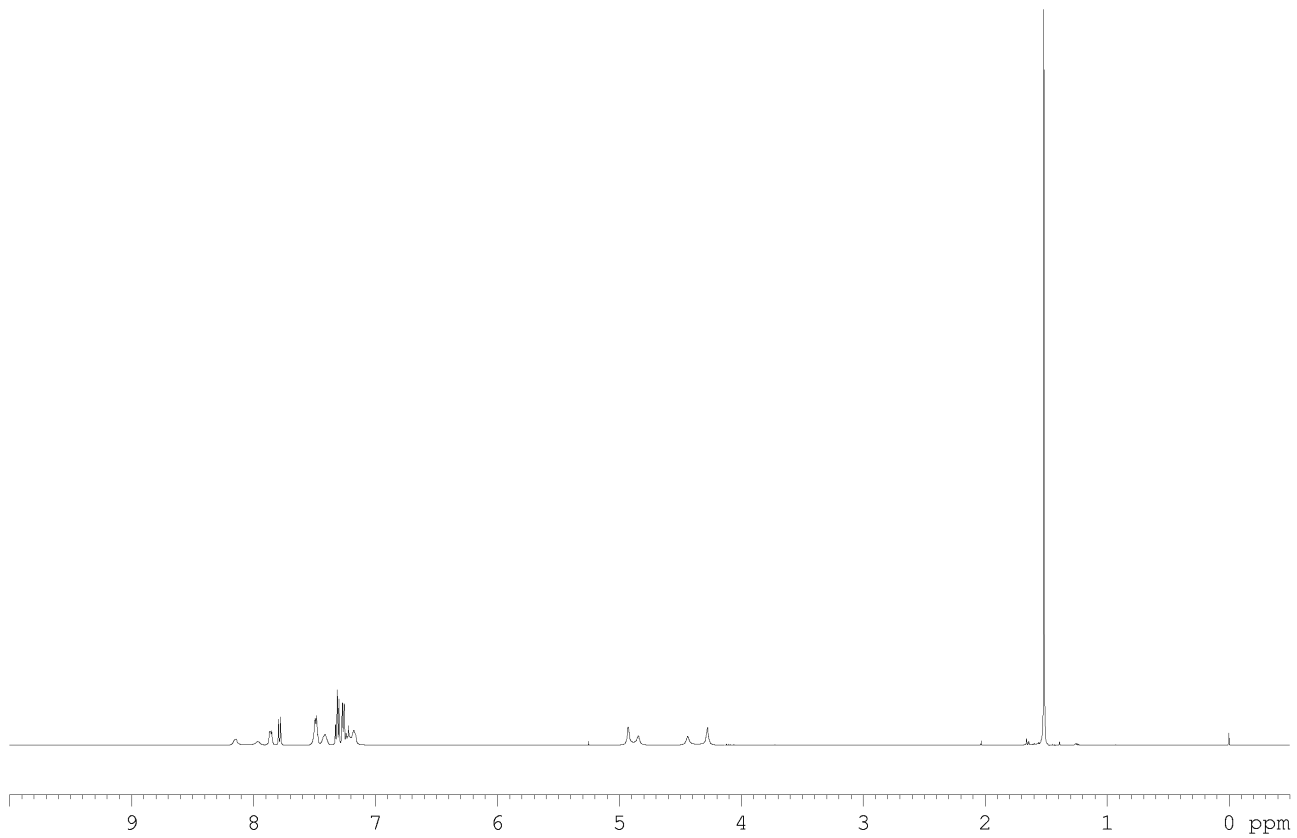
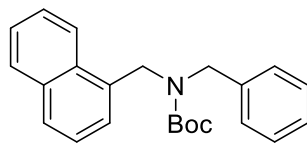


<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **10b**

S101

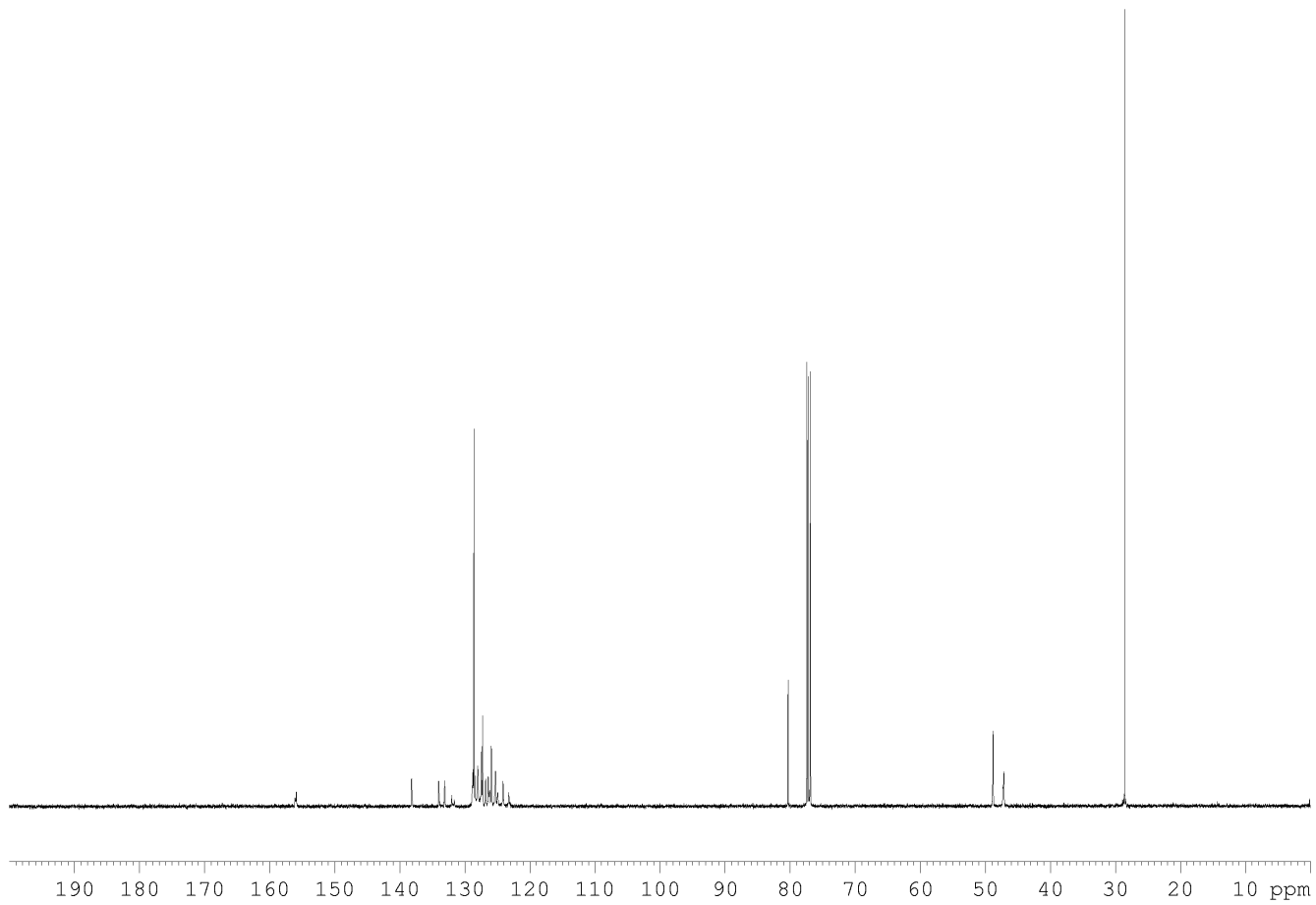
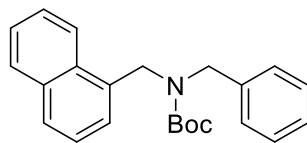


$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **10b**



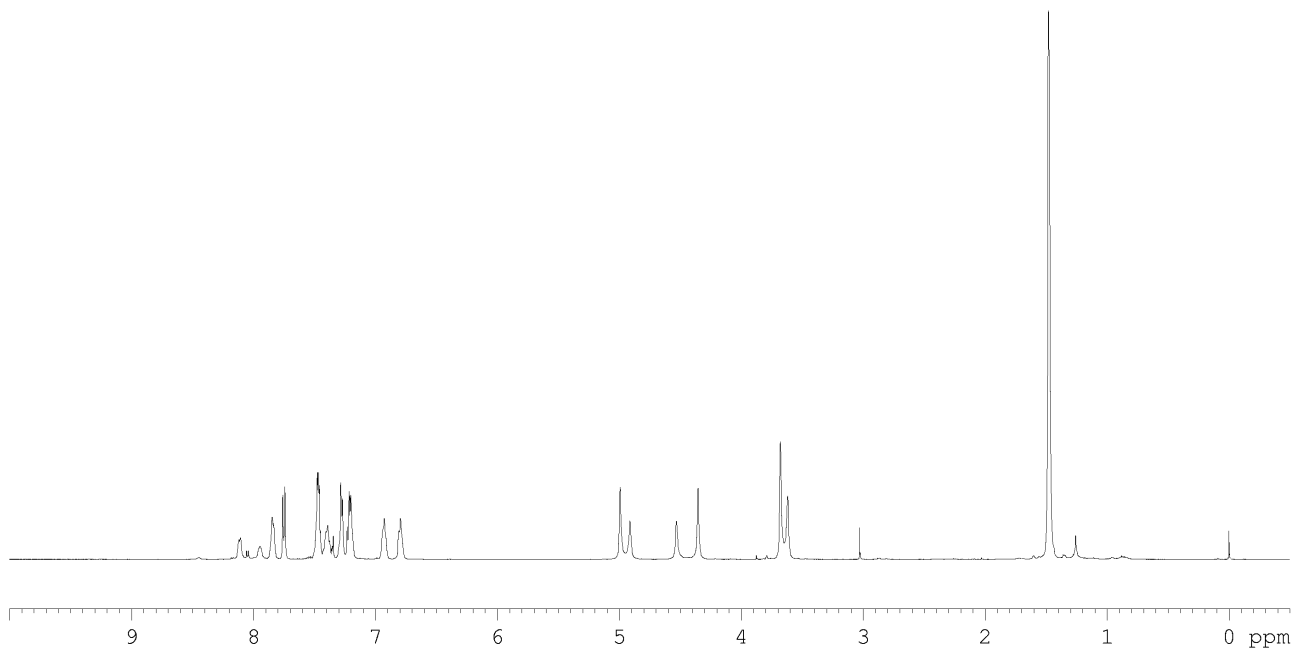
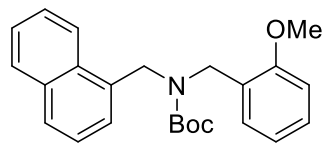
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) Spectrum of **10d**

S103



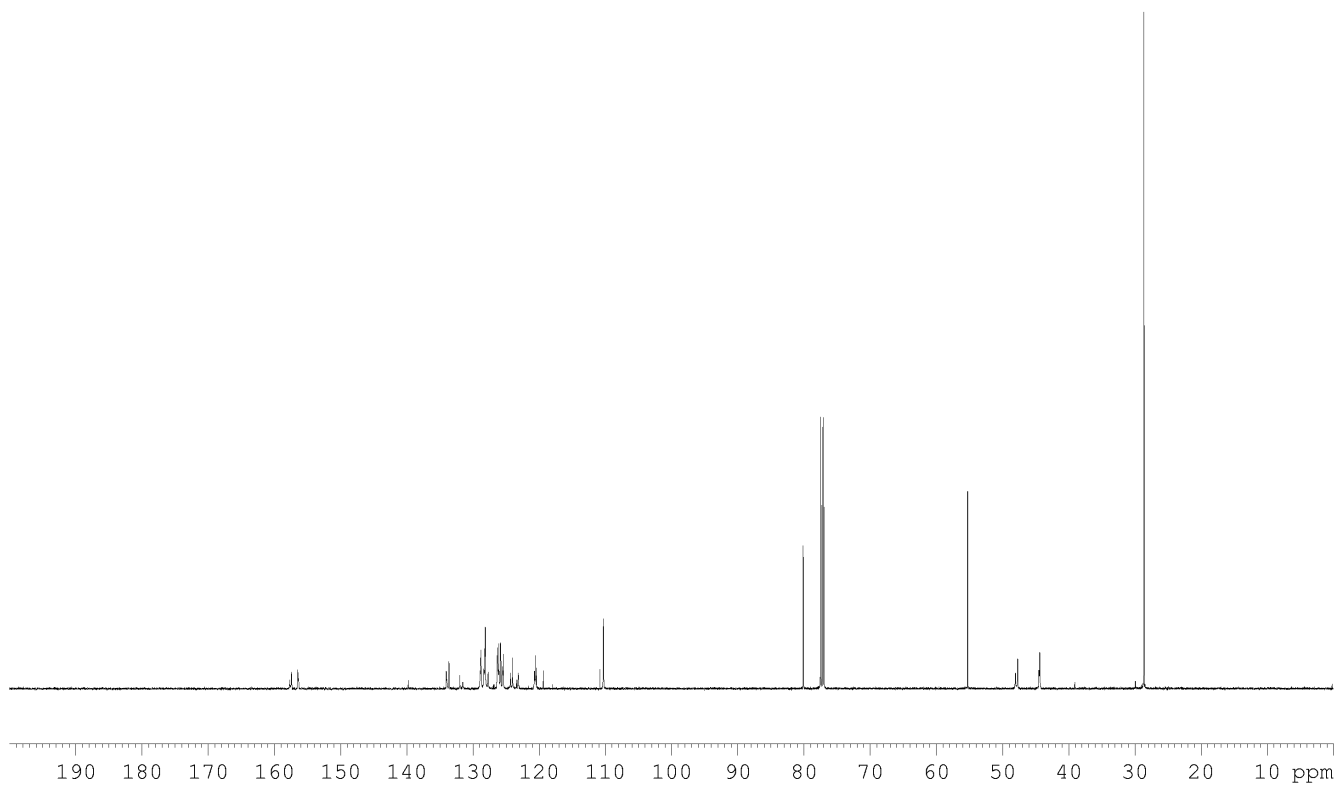
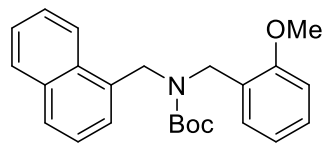
$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **10d**



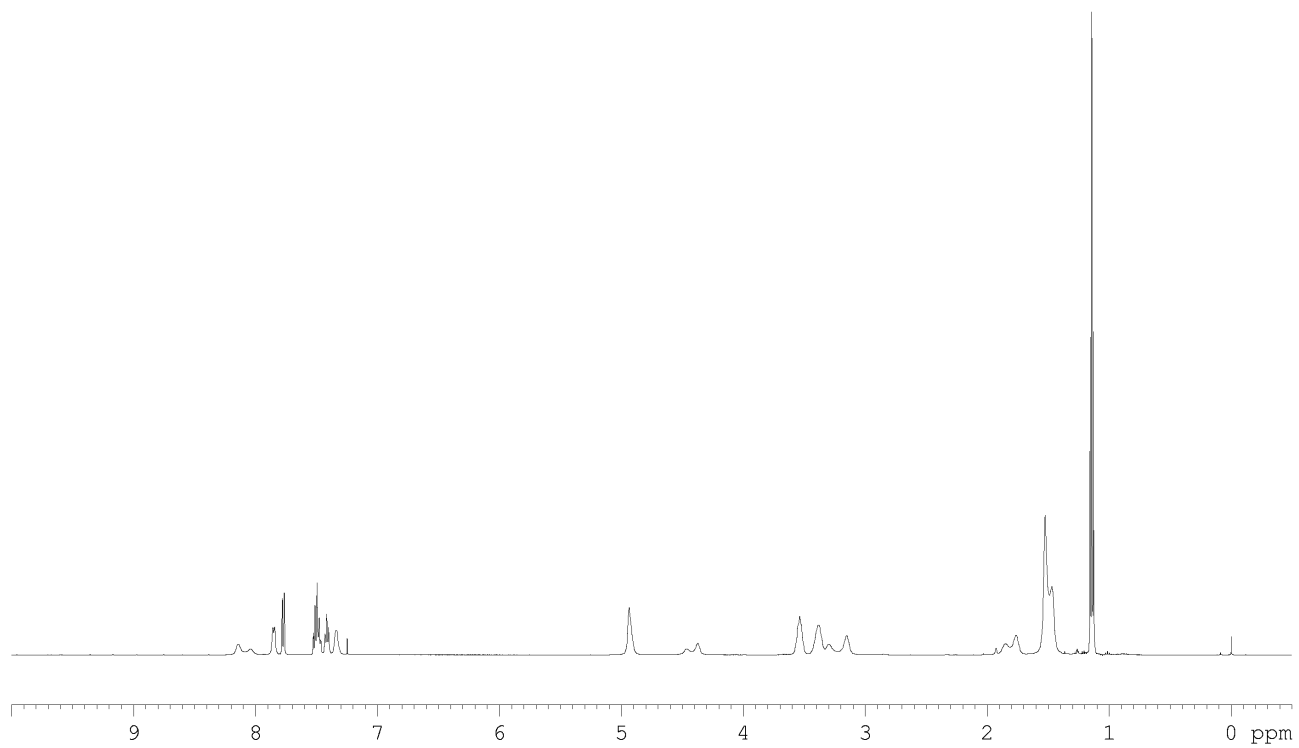
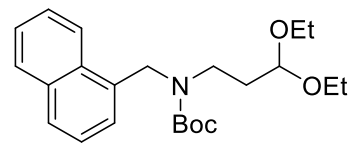


$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) Spectrum of **10e**

S105

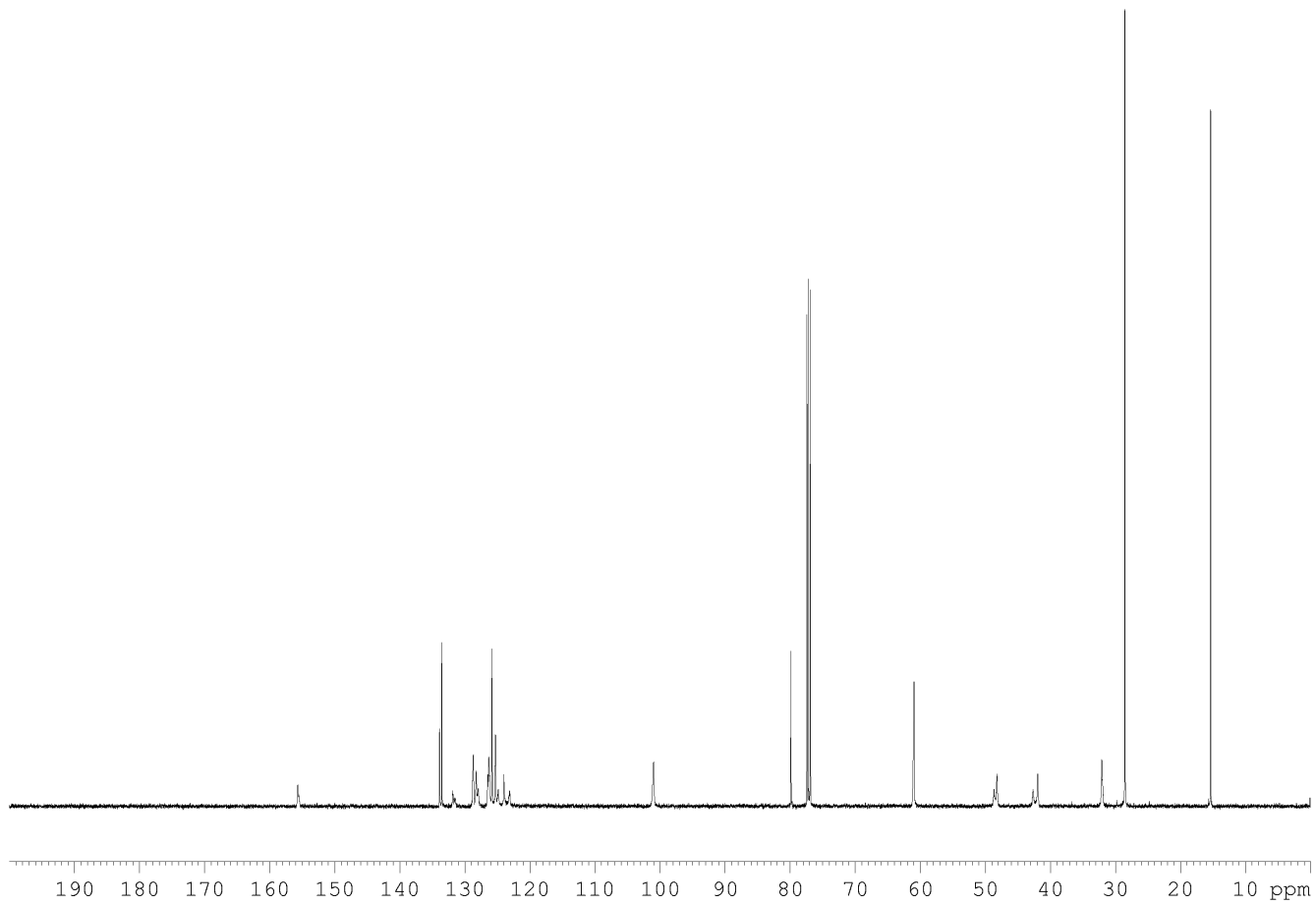
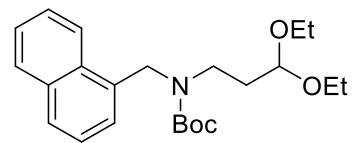


$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **10e**



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) Spectrum of **10f**

S107



$^{13}\text{C}$  NMR (125.8 MHz,  $\text{CDCl}_3$ ) Spectrum of **10f**