

# **Ir<sup>III</sup> Catalyzed Facile Syntheses of Amides and Esters Using Nitriles as “Acid Equivalents”: A Photochemical Pathway**

Ranadeep Talukdar<sup>\*a</sup>

<sup>a</sup>Molecular Synthesis and Drug Discovery Laboratory, Centre of Biomedical Research,  
Sanjay Gandhi Postgraduate Institute of Medical Sciences, Lucknow-226014, India

**\*Email:** [ranadeep@chem.iitkgp.ernet.in](mailto:ranadeep@chem.iitkgp.ernet.in)

## Experimental Section

**General Methods.** Analytical thin layer chromatography (TLC) was carried out using silica gel 60 F254 pre-coated plates. Visualization was accomplished with UV lamp or charring the TLC plate in phosphomolybdic acid (PMA). Silica gel 230-400 mesh size was used for column chromatography using the combination of ethyl acetate and petroleum ether as an eluent. Wherever appropriate, solvents and all reagents were purified prior to use following the guidelines of Perrin and Armarego<sup>1</sup> and Vogel<sup>2</sup>. All the reagents and photocatalysts were purchased from Sigma-Aldrich (Merck) and were used as received without further purification. All other commercial reagents were used as received. Borosilicate glass round bottomed flasks were used for every photochemical reaction. Blue LED lights from VictorLED private Ltd. (model LED M2E16) was used. Compound names were determined using ChemBioDraw Ultra (v.12) software. NMR spectra were recorded on a Bruker 400 Ultra Shield in CDCl<sub>3</sub> as deuterated solvent. Proton nuclear magnetic resonance (<sup>1</sup>H NMR) spectra were recorded at 400 MHz. Chemical shifts were recorded in parts per million (ppm,  $\delta$ ) relative to tetramethyl silane ( $\delta$  0.00). <sup>1</sup>H NMR splitting patterns are designated as singlet (s), broad singlet (bs), doublet (d), doublets of doublet (dd), doublets of doublets of doublet (ddd), triplet (t), triplets of triplet (tt), quartet (q), quintet (qnt), sextet (sxt), heptate (hpt) or multiplet (m). Carbon nuclear magnetic resonance (<sup>13</sup>C NMR) spectra were recorded at 100 MHz. High Resolution Mass Spectra (HRMS) were obtained on an Agilent Technologies 6530 Accurate-Mass Q-TOF LC/MS spectrometer using electron spray ionization (ESI) technique. Melting points were determined on a Büchi Melting Point M-560 hot stage apparatus and are reported as uncorrected.

**General synthetic route to amides (3a-u,9) or esters (5a-ab,7,10).** In a 10 mL round bottom flask equipped with a magnetic stirring bar, appropriate amines (**1**, 2.0 mmol, 1.0 equivalent) or alcohols (**3**, 2.0 mmol, 1.0 equivalent), photocatalyst Ir<sup>III</sup>[df(CF<sub>3</sub>)ppy]<sub>2</sub>(dtbbpy)PF<sub>6</sub> (23 mg, 0.02 mmol, 0.01 equivalent), CCl<sub>3</sub>Br (**2**, 793 mg, 4.0 mmol, 2.0 equivalent) and nitrile (1.0 mL or 1.0 g, solvent/solid, excess) were taken under argon atmosphere at room temperature (for solid nitriles, the mixture of the nitrile, CCl<sub>3</sub>Br and the amine/alcohol acted as solvent). The flask was degassed three times following freeze-pump-thaw method. Then it was stirred at room temperature under blue light irradiation (LED,  $\lambda_{\max}$  = 445  $\pm$  10 nm, 300 mA, 5.0 W, no extra filter) at a distance of approximately 2.0 cm from the LED light. After completion of the reaction

(progress of the reaction monitored by TLC) the solvent was evaporated under reduced pressure. The residue was distributed in 10 mL of water and extracted by 20x3 mL ethyl acetate. The combined organic layers were kept over anhydrous MgSO<sub>4</sub> and were concentrated under reduced pressure. The crude product was purified by column chromatography using 230-400 mesh silica gel using 10-30% ethyl acetate in hexanes solvent mixture to afford pure amides (**3a-u**, **9**) or 1-2% ethyl acetate in hexanes solvent mixture to afford pure esters (**5a-ab**, **7**, **10**) as crystalline solids or colourless oils.

## SPECTRAL DATA

**N-Benzylacetamide (3a).**<sup>3</sup> Reaction time 9 h. Off-white solid, Yield 78% (233 mg), M.P. 55-57 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.29-7.20 (m, 5H), 6.68 (bs, 1H), 4.32 (d, *J* = 4.0 Hz, 2H), 1.92 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 170.4, 138.3, 128.5, 127.7, 127.3, 43.5, 23.0; HRMS (ESI) calcd. for C<sub>9</sub>H<sub>12</sub>NO [M+H]<sup>+</sup> 150.0919, found 150.0915.

**N-(4-methoxyphenyl)acetamide (3b).**<sup>4</sup> Reaction time 25 h. Grey solid, Yield 40% (132 mg), M.P. 128-130 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.89 (bs, 1H), 7.35 (t, *J* = 8.0 Hz, 2H), 6.78 (t, *J* = 8.0 Hz, 2H), 3.75 (s, 3H), 2.09 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 169.1, 156.4, 131.3, 122.2, 114.0, 55.5, 24.1; HRMS (ESI) calcd. for C<sub>9</sub>H<sub>12</sub>NO<sub>2</sub> [M+H]<sup>+</sup> 166.0868, found 166.0865.

**N-butylacetamide (3c).**<sup>5</sup> Reaction time 7.5 h. Colourless oil, Yield 87% (401 mg); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 5.85 (bs, 1H), 3.19 (q, *J* = 8.0 Hz, 2H), 1.93 (s, 3H), 1.45 (qnt, *J* = 8.0 Hz, 2H), 1.32 (sxt, *J* = 8.0 Hz, 2H), 0.90 (t, *J* = 8.0 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 170.4, 39.1, 31.3, 22.7, 19.9, 13.5; HRMS (ESI) calcd. for C<sub>6</sub>H<sub>13</sub>NNaO [M+Na]<sup>+</sup> 138.0895, found 138.0885.

**N-((3s,5s,7s)-adamantan-1-yl)acetamide (3d).**<sup>6</sup> Reaction time 8 h. Bright white crystalline solid, Yield 82% (317 mg), M.P. 146-148 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 5.32 (bs, 1H), 2.02 (s, 3H), 1.96 (s, 6H), 1.87 (s, 3H), 1.63 (s, 6H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 169.4, 51.9, 41.7, 36.4, 29.5, 24.7; HRMS (ESI) calcd. for C<sub>12</sub>H<sub>20</sub>NO [M+H]<sup>+</sup> 194.1545, found 194.1544.

**1-(Pyrrolidin-1-yl)ethanone (3e).**<sup>7</sup> Reaction time 11 h. Colourless oil, Yield 85% (192 mg); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 3.44-3.38 (m, 4H), 2.02 (s, 3H), 1.94 (qnt, *J* = 8.0 Hz, 2H), 1.85

(qnt,  $J = 8.0$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  168.9, 46.9, 45.0, 25.5, 24.0, 21.8; HRMS (ESI) calcd. for  $\text{C}_6\text{H}_{11}\text{NNaO}$   $[\text{M}+\text{Na}]^+$  136.0738, found 136.0728.

**1-Morpholinoethanone (3f).**<sup>8</sup> Reaction time 12 h. Colourless oil, Yield 88% (227 mg);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.69-3.65 (m, 4H), 3.62-3.59 (m, 2H), 3.47-3.44 (m, 2H), 2.09 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.2, 66.7, 66.5, 46.6, 41.7, 21.1; HRMS (ESI) calcd. for  $\text{C}_6\text{H}_{11}\text{NNaO}_2$   $[\text{M}+\text{Na}]^+$  152.0687, found 152.0683.

**Morpholino(phenyl)methanone (3g).**<sup>9</sup> Reaction time 10 h. Colourless oil, Yield 77% (294 mg);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.39-7.35 (m, 5H), 3.74-3.40 (m, 8H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  170.4, 135.2, 129.9, 128.5, 127.0, 66.8, 48.1, 42.5; HRMS (ESI) calcd. for  $\text{C}_{11}\text{H}_{14}\text{NO}_2$   $[\text{M}+\text{H}]^+$  192.1019, found 192.1012.

**Phenyl(pyrrolidin-1-yl)methanone (3h).**<sup>10</sup> Reaction time 9.5 h. Colourless oil, Yield 79% (277 mg);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.52-7.49 (m, 2H), 7.41-7.35 (m, 3H), 3.63 (t,  $J = 8.0$  Hz, 2H), 3.40 (t,  $J = 8.0$  Hz, 2H), 1.93 (qnt,  $J = 8.0$  Hz, 2H), 1.84 (qnt,  $J = 8.0$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.4, 136.8, 129.5, 127.9, 126.8, 49.3, 45.9, 26.1, 24.1; HRMS (ESI) calcd. for  $\text{C}_{11}\text{H}_{14}\text{NO}$   $[\text{M}+\text{H}]^+$  176.1070, found 176.1072.

**N,N-Diisopropylbenzamide (3i).**<sup>11</sup> Reaction time 12 h. White solid, Yield 80% (328 mg), M.P. 65-67 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.37-7.26 (m, 5H), 3.77 (bs, 1H), 3.53 (bs, 1H), 1.47 (bs, 6H), 1.14 (bs, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  171.0, 138.9, 128.6, 128.4, 125.5, 50.8, 45.9, 20.7; HRMS (ESI) calcd. for  $\text{C}_{13}\text{H}_{20}\text{NO}$   $[\text{M}+\text{H}]^+$  206.1539, found 206.1530.

**N,N-Dibenzylbenzamide (3j).**<sup>12</sup> Reaction time 16 h. White solid, Yield 62% (373 mg), M.P. 109-111 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.56-7.53 (m, 2H), 7.41-7.30 (m, 11H), 7.18-7.17 (m, 2H), 4.75 (s, 2H), 4.43 (s, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  172.3, 136.9, 136.4, 136.1, 129.7, 128.9, 128.7, 128.6, 128.4, 127.7, 127.5, 127.0, 126.7, 51.5, 46.8; HRMS (ESI) calcd. for  $\text{C}_{19}\text{H}_{16}\text{NO}$   $[\text{M}+\text{H}]^+$  274.1226, found 274.1221.

**N-benzyl-N-(2-hydroxyethyl)benzamide (3k).**<sup>13</sup> Reaction time 15 h. Colourless oil, Yield 66% (337 mg); Mixture of rotamers;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.46-7.45 (m, 2H), 7.37-7.28 (m, 7H), 7.17-7.15 (m, 1H), 4.82 (s, 1H), 4.59 (s, 1H), 3.90-3.77 (m, 2H), 3.63-3.29 (m, 2H), 2.32 (bs, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  174.0, 172.9, 137.4, 136.5, 135.7, 130.3, 129.4, 128.9

128.6, 128.0, 127.8, 127.5, 126.9, 126.8, 61.2, 59.6, 54.1, 50.0, 48.5, 47.9; **HRMS** (ESI) calcd. for  $C_{16}H_{18}NO_2$   $[M+H]^+$  256.1332, found 256.1341.

**N-isopropylbenzamide (3l).**<sup>14</sup> Reaction time 13.5 h. Bright white solid, Yield 68% (222 mg), M.P. 92-94 °C; **<sup>1</sup>H NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  7.74 (d,  $J = 4.0$  Hz, 2H), 7.49-7.39 (m, 3H), 5.91 (bs, 1H), 4.35-4.23 (m, 1H), 1.27 (d,  $J = 8.0$  Hz, 6H); **<sup>13</sup>C NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  166.9, 134.9, 131.1, 128.3, 126.9, 41.8, 22.7; **HRMS** (ESI) calcd. for  $C_{10}H_{13}NNaO$   $[M+Na]^+$  186.0895, found 186.0893.

**N-benzyl-N-phenylbenzamide (3m).**<sup>15</sup> Reaction time 20.5 h. Low melting solid, Yield 55% (316 mg), **<sup>1</sup>H NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  7.43-7.37 (m, 4H), 7.32-7.13 (m, 9H), 6.96 (d,  $J = 8.0$  Hz, 2H), 5.20 (s, 2H); **<sup>13</sup>C NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  170.3, 143.3, 137.4, 135.8, 129.5, 128.8, 128.6, 128.3, 128.2, 127.6, 127.5, 127.2, 126.5, 53.6; **HRMS** (ESI) calcd. for  $C_{20}H_{18}NO$   $[M+H]^+$  288.1383, found 288.1380.

**1-(Piperidin-1-yl)ethanone (3n).**<sup>16</sup> Reaction time 14.5 h. Colourless oil, Yield 80% (203 mg); **<sup>1</sup>H NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  3.45 (t,  $J = 4.0$  Hz, 2H), 3.32 (t,  $J = 6.0$  Hz, 2H), 1.99 (s, 3H), 1.57-1.43 (m, 6H); **<sup>13</sup>C NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  168.4, 47.1, 42.1, 26.0, 25.1, 24.1, 21.1; **HRMS** (ESI) calcd. for  $C_7H_{13}NNaO$   $[M+Na]^+$  150.0895, found 150.0876.

**1-(Piperidin-1-yl)pentan-1-one (3o).**<sup>17</sup> Reaction time 10 h. Colourless oil, Yield 77% (261 mg); **<sup>1</sup>H NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  3.54 (t,  $J = 4.0$  Hz, 2H), 3.37 (t,  $J = 4.0$  Hz, 2H), 2.31-2.27 (m, 2H), 1.64-1.47 (m, 8H), 1.34 (sxt,  $J = 4.0$  Hz, 2H), 0.90 (t,  $J = 8.0$  Hz, 3H); **<sup>13</sup>C NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  171.5, 46.7, 42.5, 33.2, 27.6, 26.5, 25.6, 24.6, 22.6, 13.9; **HRMS** (ESI) calcd. for  $C_{10}H_{20}NO$   $[M+H]^+$  170.1539, found 170.1540.

**Cyclohexyl(piperidin-1-yl)methanone (3p).**<sup>18</sup> Reaction time 12 h. Colourless oil, Yield 73% (285 mg); **<sup>1</sup>H NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  3.51 (t,  $J = 4.0$  Hz, 2H), 3.39 (t,  $J = 4.0$  Hz, 2H), 2.47-2.40 (m, 1H), 1.76-1.44 (m, 12H), 1.29-1.15 (m, 4H); **<sup>13</sup>C NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  174.5, 46.5, 42.7, 40.5, 29.5, 26.9, 26.0(2), 25.7, 24.8; **HRMS** (ESI) calcd. for  $C_{12}H_{22}NO$   $[M+H]^+$  196.1696, found 196.1694.

**3-Phenyl-1-(piperidin-1-yl)propan-1-one (3q).**<sup>19</sup> Reaction time 11 h. Colourless oil, Yield 64% (278 mg); **<sup>1</sup>H NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  7.39-7.35 (m, 2H), 7.31-7.26 (m, 3H), 3.64 (t,  $J = 8.0$  Hz, 2H), 3.41 (t,  $J = 8.0$  Hz, 2H), 3.05 (t,  $J = 8.0$  Hz, 2H), 2.71 (t,  $J = 8.0$  Hz, 2H), 1.72-1.66 (m,

2H), 1.63-1.51 (m, 4H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  170.7, 141.5, 128.5(2), 126.2, 46.7, 42.8, 35.2, 31.7, 26.4, 25.6, 24.6; HRMS (ESI) calcd. for  $\text{C}_{14}\text{H}_{19}\text{NNaO}$   $[\text{M}+\text{Na}]^+$  240.1364, found 240.1354.

**Phenyl(piperidin-1-yl)methanone (3r).**<sup>20</sup> Reaction time 13 h. Colourless oil, Yield 72% (272 mg);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.37 (bs, 5H), 3.70 (bs, 2H), 3.32 (bs, 2H), 1.65 (bs, 4H), 1.49 (bs, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  170.0, 136.2, 129.1, 128.1, 126.5, 48.5, 42.8, 26.2, 25.3, 24.3; HRMS (ESI) calcd. for  $\text{C}_{12}\text{H}_{16}\text{NO}$   $[\text{M}+\text{H}]^+$  190.1226, found 190.1224.

**(4-Methoxyphenyl)(piperidin-1-yl)methanone (3s).**<sup>21</sup> Reaction time 7.5 h. Colourless liquid, Yield 68% (298 mg),  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.37 (d,  $J = 8.0$  Hz, 2H), 6.90 (d,  $J = 8.0$  Hz, 2H), 3.81 (s, 3H), 3.66 (bs, 2H), 3.41 (bs, 2H), 1.66-1.58 (m, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  170.1, 160.3, 128.7, 128.3, 113.4, 55.1, 48.8, 43.2, 26.3, 25.6, 24.5; HRMS calcd. For  $\text{C}_{13}\text{H}_{18}\text{NO}_2$   $[\text{M}+\text{H}]^+$  220.1338, found 220.1325.

**(4-Nitrophenyl)(piperidin-1-yl)methanone (3t).**<sup>22</sup> Reaction time 5 h. Light yellow solid, Yield 41% (192 mg), M.P. 117-119 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.23-8.21 (m, 2H), 7.53-7.50 (m, 2H), 3.68 (bs, 2H), 3.24 (bs, 2H), 1.65 (bs, 4H), 1.48 (bs, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.9, 148.2, 142.7, 127.8, 123.8, 48.6, 43.2, 26.5, 25.5, 24.4; HRMS calcd. For  $\text{C}_{12}\text{H}_{15}\text{N}_2\text{O}_3$   $[\text{M}+\text{H}]^+$  235.1083, found 235.1077.

**Piperidin-1-yl(pyridin-4-yl)methanone (3u).**<sup>23</sup> Reaction time 12 h. Yellow oil, Yield 69% (263 mg),  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.63 (dd,  $J = 7.0, 3.0$  Hz, 2H), 7.23 (dd,  $J = 4.0, 2.0$  Hz, 2H), 3.67-3.65 (m, 2H), 3.23 (d,  $J = 4.0$  Hz, 2H), 1.64 (bs, 4H), 1.47 (bs, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.6, 150.2, 144.1, 121.1, 48.5, 43.0, 26.5, 25.5, 24.4; HRMS (ESI) calcd. For  $\text{C}_{11}\text{H}_{15}\text{N}_2\text{O}$   $[\text{M}+\text{H}]^+$  191.1184, found 191.1176.

**Phenethyl acetate (5a).**<sup>24</sup> Reaction time 8 h. Colourless oil, Yield 90% (296 mg);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.30 (t,  $J = 8.0$  Hz, 2H), 7.23 (t,  $J = 8.0$  Hz, 3H), 4.29 (t,  $J = 8.0$ , 2H), 2.94 (t,  $J = 8.0$  Hz, 2H), 2.04 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  170.7, 137.7, 128.7, 128.4, 126.4, 64.7, 34.9, 20.7; HRMS (ESI) calcd. for  $\text{C}_{10}\text{H}_{12}\text{NaO}_2$   $[\text{M}+\text{Na}]^+$  187.0735, found 187.0730.

**5-Phenylpentyl acetate (5b).**<sup>25</sup> Reaction time 11.5 h. Colourless oil, Yield 88% (363 mg);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.20-7.16 (m, 2H), 7.10-7.06 (m, 3H), 3.96 (t,  $J = 8.0$  Hz, 2H), 2.52 (t,  $J = 8.0$  Hz, 2H), 1.94 (s, 3H), 1.59-1.52 (m, 4H), 1.34-1.26 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,

CDCl<sub>3</sub>):  $\delta$  171.3, 142.5, 128.5, 128.4, 125.8, 64.6, 35.9, 31.2, 28.6, 25.7, 21.1; **HRMS** (ESI) calcd. for C<sub>13</sub>H<sub>18</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> 229.1204, found 229.1180.

**1-Butoxypropan-2-yl acetate (5c).**<sup>26</sup> Reaction time 15 h. Colourless oil, Yield 67% (233 mg); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  4.97-4.91 (m, 1H), 3.39-3.27 (m, 4H), 1.94 (s, 3H), 1.47-1.41 (m, 2H), 1.31-1.22 (m, 2H), 1.12 (d, *J* = 8.0 Hz, 3H), 0.82 (t, *J* = 8.0 Hz, 3H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>):  $\delta$  170.5, 73.0, 71.1, 69.3, 31.6, 21.2, 19.2, 16.7, 13.8; **HRMS** (ESI) calcd. for C<sub>9</sub>H<sub>19</sub>O<sub>3</sub> [M+H]<sup>+</sup> 175.1334, found 175.1326.

**Decyl acetate (5d).**<sup>27</sup> Reaction time 9 h. Colourless oil, Yield 80% (321 mg); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  3.99 (t, *J* = 8.0 Hz, 2H), 1.98 (s, 3H), 1.58-1.53 (m, 2H), 1.31-1.24 (m, 14H), 0.84 (t, *J* = 8.0 Hz, 3H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>):  $\delta$  170.9, 64.5, 31.9, 29.5, 29.3(2), 28.6, 25.9, 22.7, 20.8, 14.0; **HRMS** (ESI) calcd. for C<sub>12</sub>H<sub>24</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> 223.1674, found 223.1667.

**Cyclohexyl acetate (5e).**<sup>28</sup> Reaction time 10 h. Colourless oil, Yield 80% (228 mg); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  4.66-4.61 (m, 1H), 1.95 (s, 3H), 1.80-1.76 (m, 2H), 1.69-1.64 (m, 2H), 1.50-1.47 (m, 1H), 1.37-1.17 (m, 5H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>):  $\delta$  170.5, 72.6, 31.6, 25.4, 23.8, 21.3; **HRMS** (ESI) calcd. for C<sub>8</sub>H<sub>14</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> 165.0891, found 165.0893.

**2-Isopropyl-5-methylcyclohexyl acetate (5f, menthyl acetate).**<sup>29</sup> Reaction time 16 h. Colourless oil, Yield 63% (250 mg); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  4.62-4.56 (m, 1H), 1.95-1.90 (m, 4H), 1.83-1.76 (m, 1H), 1.62-1.59 (m, 2H), 1.45-1.40 (m, 1H), 1.28 (t, *J* = 8.0 Hz, 1H), 1.04-0.76 (m, 9H), 0.70 (d, *J* = 8.0 Hz, 3H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>):  $\delta$  170.6, 74.1, 47.0, 41.0, 34.3, 31.4, 26.3, 23.5, 22.1, 21.3, 20.8, 16.4; **HRMS** (ESI) calcd. for C<sub>12</sub>H<sub>23</sub>O<sub>2</sub> [M+H]<sup>+</sup> 199.1698, found 199.1697.

**But-3-yn-1-yl acetate (5g).**<sup>30</sup> Reaction time 18 h. Colourless oil, Yield 46% (103 mg); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  4.08 (t, *J* = 8.0 Hz, 2H), 2.44 (td, *J* = 8.0 Hz, 2H), 1.99 (s, 3H), 1.92 (t, *J* = 4.0 Hz, 1H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>):  $\delta$  170.6, 80.0, 69.9, 62.0, 20.7, 18.8; **HRMS** (ESI) calcd. for C<sub>6</sub>H<sub>9</sub>O<sub>2</sub> [M+H]<sup>+</sup> 113.0603, found 113.0591.

**3,7-Dimethyloct-6-en-1-yl acetate (5h, citronellyl acetate).**<sup>31</sup> Reaction time 12 h. Colourless oil, Yield 52% (206 mg); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>):  $\delta$  5.03 (t, *J* = 8.0 Hz, 1H), 4.10-4.00 (m, 2H), 2.00-1.90 (m, 5H), 1.67-1.49 (m, 8H), 1.44-1.29 (m, 2H), 1.20-1.12 (sxt, *J* = 8.0 Hz, 1H), 0.89 (d, *J* = 4.0 Hz, 3H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>):  $\delta$  170.1, 131.2, 124.6, 63.0, 37.0, 35.4,

29.4, 25.7, 25.4, 20.9, 19.4, 17.6; **HRMS** (ESI) calcd. for  $C_{12}H_{22}NaO_2$   $[M+Na]^+$  221.1517, found 221.1505.

**Butyl benzoate (5i).**<sup>32</sup> Reaction time 12 h. Pale yellow oil, Yield 77% (274 mg);  **$^1H$  NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  8.06-8.03 (m, 2H), 7.56-7.50 (m, 1H), 7.42 (t,  $J = 8.0$  Hz, 2H), 4.32 (t,  $J = 8.0$  Hz, 2H), 1.74 (qnt,  $J = 8.0$  Hz, 2H), 1.47 (sxt,  $J = 8.0$  Hz, 2H), 0.97 (t,  $J = 7.0$  Hz, 3H);  **$^{13}C$  NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  166.7, 132.8, 130.6, 129.6, 128.3, 64.8, 30.8, 19.3, 13.8; **HRMS** (ESI) calcd. for  $C_{11}H_{15}O_2$   $[M+H]^+$  179.1072, found 179.1064.

**Hexyl benzoate (5j).**<sup>33</sup> Reaction time 19 h. Colourless liquid, Yield 60% (247 mg);  **$^1H$  NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  8.06-8.03 (m, 2H), 7.55 (tt,  $J = 8.0, 2.0$  Hz, 1H), 7.46-7.42 (m, 2H), 4.32 (t,  $J = 8.0$  Hz, 2H), 1.77 (q,  $J = 8.0$  Hz, 2H), 1.48-1.41 (m, 2H), 1.35 (sxt,  $J = 4.0$  Hz, 4H), 0.92-0.88 (m, 3H);  **$^{13}C$  NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  166.9, 132.9, 130.7, 129.7, 128.5, 65.3, 31.6, 28.8, 25.9, 22.7, 14.2; **HRMS** (ESI) calcd for  $C_{13}H_{18}NaO_2$   $[M+Na]^+$  229.1204, found 229.1194.

**Decyl benzoate (5k).**<sup>34</sup> Reaction time 23 h. Colourless oil, Yield 54% (283 mg);  **$^1H$  NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  8.06-8.04 (m, 2H), 7.55-7.52 (m, 1H), 7.42 (t,  $J = 8.0$  Hz, 2H), 4.31 (t,  $J = 8.0$  Hz, 2H), 1.76 (qnt,  $J = 8.0$  Hz, 2H), 1.47-1.27 (m, 14H), 0.88 (t,  $J = 8.0$  Hz, 3H);  **$^{13}C$  NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  166.7, 132.8, 130.6, 129.6, 128.3, 65.2, 32.0, 29.6, 29.4(2), 28.8, 26.1, 22.8, 14.2; **HRMS** (ESI) calcd. for  $C_{17}H_{27}O_2$   $[M+H]^+$  263.2006, found 263.2003.

**Isopropyl benzoate (5l).**<sup>35</sup> Reaction time 10 h. Colourless oil, Yield 82% (269 mg);  **$^1H$  NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  8.02 (d,  $J = 8.0$  Hz, 2H), 7.50 (t,  $J = 8.0$  Hz, 1H), 7.40 (t,  $J = 8.0$  Hz, 2H), 5.24 (hpt,  $J = 6.0$  Hz, 1H), 1.36 (t,  $J = 8.0$  Hz, 6H);  **$^{13}C$  NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  166.1, 132.7, 130.9, 129.5, 128.3, 68.3, 21.9; **HRMS** (ESI) calcd. for  $C_{10}H_{12}NaO_2$   $[M+Na]^+$  187.0735, found 187.0731.

**Cyclohexyl benzoate (5m).**<sup>36</sup> Reaction time 17 h. Colourless oil, Yield 55% (225 mg);  **$^1H$  NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  8.07-8.04 (m, 2H), 7.54 (tt,  $J = 8.0, 4.0$  Hz, 1H), 7.46-7.40 (m, 2H), 5.04 (hpt,  $J = 4.0$  Hz, 1H), 1.98-1.93 (m, 2H), 1.83-1.77 (m, 2H), 1.64-1.30 (m, 6H);  **$^{13}C$  NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  166.1, 132.7, 131.1, 129.6, 128.3, 73.1, 31.7, 25.6, 23.8; **HRMS** (ESI) calcd. for  $C_{13}H_{16}NaO_2$   $[M+Na]^+$  227.1048, found 227.1039.

**2-Chloroethyl benzoate (5n).**<sup>37</sup> Reaction time 15 h. Yellow oil, Yield 69% (255 mg);  **$^1H$  NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  8.09-8.06 (m, 2H), 7.58 (tt,  $J = 8.0, 2.0$  Hz, 1H), 7.48-7.44 (m, 2H), 4.59-



4.56 (m, 2H), 3.83-3.80 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  166.3, 133.4, 129.9, 129.8, 128.6, 64.6, 41.8; HRMS (ESI) calcd. for  $\text{C}_9\text{H}_{10}\text{ClO}_2$   $[\text{M}+\text{H}]^+$  185.0364, found 185.0347.

**2-Bromoethyl benzoate (5o).**<sup>38</sup> Reaction time 13.5 h. Reddish oil, Yield 71% (325 mg);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.06 (t,  $J = 8.0$  Hz, 2H), 7.56 (d,  $J = 8.0$  Hz, 1H), 7.43 (d,  $J = 8.0$  Hz, 2H), 4.60 (t,  $J = 8.0$  Hz, 2H), 3.62 (d,  $J = 8.0$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  166.0, 133.3, 129.7, 129.6, 128.5, 64.2, 28.9; HRMS (ESI) calcd. for  $\text{C}_9\text{H}_{10}\text{BrO}_2$   $[\text{M}+\text{H}]^+$  228.9864, found 228.9858.

**(Tetrahydrofuran-2-yl)methyl benzoate (5p).**<sup>39</sup> Reaction time 17 h. Pale yellow oil, Yield 58% (239 mg);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.07-8.04 (m, 2H), 7.54 (t,  $J = 8.0$  Hz, 1H), 7.42 (t,  $J = 8.0$  Hz, 2H), 4.40-4.34 (m, 1H), 4.29-4.23 (m, 2H), 3.92 (q,  $J = 8.0$  Hz, 1H), 3.82 (q,  $J = 8.0$  Hz, 1H), 2.10-1.88 (m, 3H), 1.76-1.67 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  166.6, 133.1, 130.2, 129.8, 128.4, 76.7, 68.6, 67.0, 28.2, 25.9; HRMS (ESI) calcd. for  $\text{C}_{12}\text{H}_{15}\text{O}_3$   $[\text{M}+\text{H}]^+$  207.1016, found 207.1011.

**2-(2-Chloroethoxy)ethyl benzoate (5q).**<sup>40</sup> Reaction time 24 h. Pale yellow oil, Yield 51% (233 mg);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.07-8.04 (m, 2H), 7.55 (tt,  $J = 8.0, 4.0$  Hz, 1H), 7.45-7.41 (m, 2H), 4.49-4.47 (m, 2H), 3.86-3.84 (m, 2H), 3.80 (t,  $J = 6.0$  Hz, 2H), 3.63 (t,  $J = 4.0$  Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  166.6, 133.1, 130.1, 129.8, 128.5, 71.4, 69.3, 64.0, 42.8; HRMS (ESI) calcd. for  $\text{C}_{11}\text{H}_{13}\text{ClNaO}_3$   $[\text{M}+\text{Na}]^+$  251.0451, found 251.0445.

**2-(2-Ethoxyethoxy)ethyl benzoate (5r).**<sup>41</sup> Reaction time 20 h. Colourless oil, Yield 49% (234 mg);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.05-8.02 (m, 2H), 7.54-7.50 (m, 1H), 7.42-7.38 (m, 2H), 4.47-4.45 (m, 2H), 3.83-3.81 (m, 2H), 3.69-3.66 (m, 2H), 3.59-3.57 (m, 2H), 3.50 (qd,  $J = 8.0, 2.0$  Hz, 2H), 1.18 (qd,  $J = 8.0, 2.0$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  166.6, 133.0, 130.2, 129.7, 128.4, 70.8, 69.9, 69.3, 66.7, 64.2, 15.2; HRMS (ESI) calcd. for  $\text{C}_{13}\text{H}_{19}\text{O}_4$   $[\text{M}+\text{H}]^+$  239.1278, found 239.1271.

**Benzyl benzoate (5s).**<sup>42</sup> Reaction time 22 h. Colourless oil, Yield 47% (200 mg);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.14-8.12 (m, 2H), 7.60-7.54 (m, 1H), 7.50-7.36 (m, 7H), 5.40 (s, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  166.5, 136.1, 133.1, 130.1, 129.8, 128.7, 128.4, 128.3, 128.2, 66.7; HRMS (ESI) calcd. for  $\text{C}_{14}\text{H}_{13}\text{O}_2$   $[\text{M}+\text{H}]^+$  213.0916, found 213.0911.

**4-Methoxybenzyl benzoate (5t).**<sup>43</sup> Reaction time 24 h. Colourless sticky solid, Yield 57% (276 mg); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.08-8.06 (m, 2H), 7.55 (tt, *J* = 8.0, 2.0 Hz, 1H), 7.45-7.38 (m, 4H), 6.93 (dt, *J* = 8.0, 2.0 Hz, 2H), 5.31 (s, 2H), 3.82 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 166.6, 159.8, 133.1, 130.4, 130.2, 129.8, 128.5, 128.3, 114.1, 66.7, 55.4; HRMS (ESI) calcd. for C<sub>15</sub>H<sub>14</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> 265.0835, found 265.0826.

**2-(1*H*-indol-1-yl)ethyl benzoate (5u).**<sup>44</sup> Reaction time 16 h. Colourless solid, Yield 75% (398 mg); M.P. 101-103 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.84 (d, *J* = 8.0 Hz, 2H), 7.55 (d, *J* = 8.0 Hz, 1H), 7.30 (t, *J* = 8.0 Hz, 1H), 7.32-7.28 (m, 3H), 7.13 (t, *J* = 8.0 Hz, 1H), 7.05-7.02 (m, 2H), 6.44 (d, *J* = 4.0 Hz, 1H), 4.47 (t, *J* = 8.0 Hz, 2H), 4.33 (t, *J* = 8.0 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 166.3, 136.1, 133.3, 129.7, 129.6, 128.7, 128.5, 128.1, 121.8, 121.1, 119.7, 109.3, 101.9, 63.6, 45.1; HRMS (ESI) calcd. for C<sub>17</sub>H<sub>15</sub>NNaO<sub>2</sub> [M+Na]<sup>+</sup> 288.1000, found 288.0992.

**Butyl propionate (5v).**<sup>45</sup> Reaction time 11 h. Colourless oil, Yield 85% (221 mg); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 4.01 (t, *J* = 8.0 Hz, 2H), 2.25 (q, *J* = 8.0 Hz, 2H), 1.58-1.51 (m, 2H), 1.36-1.27 (m, 2H), 1.10-1.05 (m, 3H), 0.87 (t, *J* = 8.0 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 174.5, 64.1, 30.8, 27.6, 19.2, 13.7, 9.1; HRMS (ESI) calcd. for C<sub>7</sub>H<sub>15</sub>O<sub>2</sub> [M+H]<sup>+</sup> 131.1072, found 131.1065.

**Butyl butyrate (5w).**<sup>46</sup> Reaction time 12 h. Colourless oil, Yield 80% (231 mg); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 4.01 (t, *J* = 8.0 Hz, 2H), 2.21 (t, *J* = 8.0 Hz, 2H), 1.63-1.51 (m, 4H), 1.36-1.27 (m, 2H), 0.90-0.85 (m, 6H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 173.8, 64.1, 36.3, 30.8, 19.2, 18.5, 13.7(2); HRMS (ESI) calcd. for C<sub>8</sub>H<sub>17</sub>O<sub>2</sub> [M+H]<sup>+</sup> 145.1229, found 145.1222.

**Butyl cyclohexanecarboxylate (5x).**<sup>47</sup> Reaction time 16 h. Colourless oil, Yield 78% (287 mg); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 4.03 (t, *J* = 8.0 Hz, 2H), 2.25 (tt, *J* = 12.0, 4.0 Hz, 1H), 1.88-1.83 (m, 2H), 1.74-1.70 (m, 2H), 1.62-1.53 (m, 3H), 1.45-1.16 (m, 7H), 0.90 (t, *J* = 8 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 176.3, 64.0, 43.4, 30.8, 29.1, 25.9, 25.6, 19.2, 13.8; HRMS (ESI) calcd. for C<sub>11</sub>H<sub>20</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> 207.1361, found 207.1353.

**Butyl 2-phenylacetate (5y).**<sup>48</sup> Reaction time 20 h. Colourless oil, Yield 67% (258 mg); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.39-7.28 (m, 5H), 4.15 (t, *J* = 8.0 Hz, 2H), 3.66 (s, 2H), 1.69-1.62 (m, 2H), 1.40 (sxt, *J* = 8.0 Hz, 2H), 0.96 (t, *J* = 8.0 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ

171.6, 134.2, 129.2, 128.5, 127.0, 64.7, 41.4, 30.6, 19.1, 13.7; **HRMS** (ESI) calcd. for  $C_{12}H_{17}O_2$   $[M+H]^+$  193.1229, found 193.1234.

**Butyl 4-methoxybenzoate (5z).**<sup>49</sup> Reaction time 8 h. Colourless liquid, Yield 74% (308 mg); **<sup>1</sup>H NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  7.99 (dt,  $J = 8.0, 4.0$  Hz, 2H), 6.91 (dt,  $J = 8.0, 4.0$  Hz, 2H), 4.29 (t,  $J = 8.0$  Hz, 2H), 3.85 (s, 3H), 1.77-1.70 (m, 2H), 1.51-1.42 (m, 2H), 0.97 (t,  $J = 7.0$  Hz, 3H); **<sup>13</sup>C NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  166.6, 163.4, 131.7, 123.1, 113.7, 64.7, 55.5, 31.0, 19.4, 13.9; **HRMS** calcd for  $C_{12}H_{17}O_3$   $[M+H]^+$  209.1178, found 209.1170.

**Butyl 4-nitrobenzoate (5aa).**<sup>50</sup> Reaction time 4.5 h. White low melting solid, Yield 34% (152 mg); **<sup>1</sup>H NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  8.21-8.12 (m, 4H), 4.33-4.29 (m, 2H), 1.71 (qnt,  $J = 8.0$  Hz, 2H), 1.42 (t,  $J = 8.0$  Hz, 2H), 0.95-0.90 (m, 3H); **<sup>13</sup>C NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  164.6, 150.4, 135.8, 130.6, 123.4, 65.7, 30.6, 19.2, 13.7; **HRMS** calcd for  $C_{11}H_{13}NNaO_4$   $[M+Na]^+$  246.0742, found 246.0729.

**Butyl nicotinate (5ab).**<sup>51</sup> Reaction time 23 h. Colourless oil, Yield 52% (186 mg); **<sup>1</sup>H NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  9.21 (d,  $J = 2.0$  Hz, 1H), 8.76 (dd,  $J = 4.0, 2.0$  Hz, 1H), 8.29 (dt,  $J = 8.0, 4.0$  Hz, 1H), 7.38 (ddd,  $J = 8.0, 4.0, 1.0$  Hz, 1H), 4.35 (t,  $J = 8.0$  Hz, 2H), 1.75 (qnt,  $J = 8.0$  Hz, 2H), 1.47 (sxt,  $J = 8.0$  Hz, 2H), 0.97 (t,  $J = 8.0$  Hz, 3H); **<sup>13</sup>C NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  165.2, 153.2, 150.8, 136.9, 126.3, 123.2, 65.2, 30.6, 19.1, 13.6; **HRMS** (ESI) calcd. for  $C_{10}H_{14}NO_2$   $[M+H]^+$  180.1025, found 180.1037.

**Butane-1,4-diyl dibenzoate (7).**<sup>52</sup> Reaction time 24 h. White crystalline solid, Yield 35% (104 mg); M.P. 78-80°C; **<sup>1</sup>H NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  8.06-8.04 (m, 4H), 7.56 (tt,  $J = 8.0, 4.0$  Hz, 2H), 7.46-7.42 (m, 4H), 4.42-4.39 (m, 4H), 1.97-1.94 (m, 4H); **<sup>13</sup>C NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  166.7, 133.1, 130.4, 129.7, 128.5, 64.6, 25.7; **HRMS** (ESI) calcd. for  $C_{18}H_{19}O_4$   $[M+H]^+$  299.1278, found 299.1273.

**N<sup>1</sup>,N<sup>2</sup>-diisopropylphthalamide (9).**<sup>53</sup> Reaction time 17 h. Bright white solid, Yield 53% (132 mg), M.P. 175-177 °C; **<sup>1</sup>H NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  7.54-7.52 (m, 2H), 7.42-7.40 (m, 2H), 6.67 (bs, 2H), 4.18 (sxt,  $J = 8.0$  Hz, 2H), 1.22 (d,  $J = 8.0$  Hz, 12H); **<sup>13</sup>C NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  168.5, 134.8, 130.1, 128.4, 42.4, 22.7; **HRMS** (ESI) calcd. for  $C_{14}H_{20}N_2NaO_2$   $[M+Na]^+$  271.1422, found 271.1415.

**Diisopropyl phthalate (10).**<sup>54</sup> Reaction time 20 h. Colourless oil, Yield 45% (113 mg); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.68 (dd, *J* = 8.0, 4.0 Hz, 2H), 7.49 (dd, *J* = 8.0, 4.0 Hz, 2H), 5.23 (hpt, *J* = 4.0 Hz, 2H), 1.35 (d, *J* = 8.0 Hz, 12H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 167.2, 132.8, 130.8, 128.8, 69.3, 21.8; HRMS (ESI) calcd. for C<sub>14</sub>H<sub>19</sub>O<sub>4</sub> [M+H]<sup>+</sup> 251.1283, found 251.1272.

## References

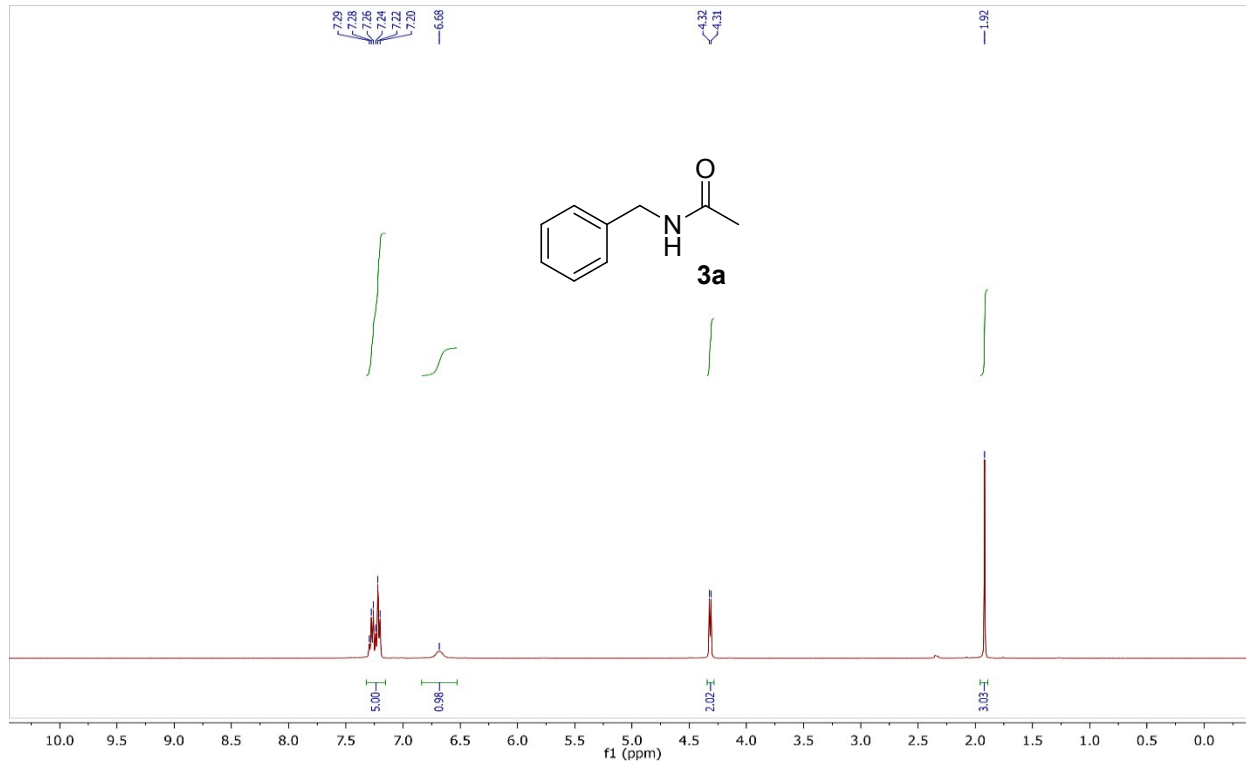
1. D. D. Perin and W. L. F. Armarego, *Purification of Laboratory Chemicals*. Third Edition, Pergamon Press, 1988, Oxford.
2. B. S. Furniss, A. J. Hannaford, P. W. G. Smith and A. R. Tatchell, *Vogel's Textbook of Practical Organic Chemistry*, Fifth Edition, Longman Group, 1989, U. K. Ltd.
3. A. Charvieux, L. Le Moigne, L. G. Borrego, N. Duguet and E. Metay, *Eur. J. Org. Chem.*, 2019, 6842-6846.
4. T. Ghosh, S. Jana and J. Dash, *Org. Lett.*, 2019, **21**, 6690-6694.
5. R. M. N. Kalla, S. S. Reddy and I. Kim, *Catal. Lett.*, 2019, **149**, 2696-2705.
6. J.-P. Berndt, F. R. Erb, L. Ochmann, J. Beppler and P. R. Schreiner, *Synlett*, 2019, **30**, 493-498.
7. X. Si, L. Zhang and A. S. K. Hashmi, *Org. Lett.*, 2019, **21**, 6329-6332.
8. T. N. Allah, S. Savourey, J.-C. Berthet, E. Nicolas and T. Cantat, *Angew. Chem., Int. Ed.*, 2019, **58**, 10884-10887.
9. H. Sepahvand, E. Ghasemi, M. Sharbati, M. S. Mohammadi, M. A. Pirlar and G. H. Shahverdizadeh, *New J. Chem.*, 2019, **43**, 16555-16565.
10. Z.-J. Niu, L.-H. Li, X.-Y. Liu and Y.-M. Liang, *Adv. Synth. Catal.*, 2019, **361**, 5217-5222.
11. G. Li, C.-L. Ji, X. Hong and M. Szostak, *J. Am. Chem. Soc.*, 2019, **141**, 11161-11172.
12. F. Dai, Y. Yang, J. Gu, Z. Fang, Z. Yang, C. Liu, W. He, N. Zhu, B. Lu and K. Guo, *ChemistrySelect*, 2019, **4**, 3500-3504.

13. Y. Nishii, T. Hirai, S. Fernandez, P. Knochel and K. Mashima, *Eur. J. Org. Chem.*, 2017, 5010-5014.
14. P. Sureshababu, S. Azeez, N. Muniyappan, S. Sabiah and J. Kandasamy, *J. Org. Chem.*, 2019, **84**, 11823-11838.
15. M. Wang and Z. Huang, *Org. Biomol. Chem.*, 2016, **14**, 10185-10188.
16. H. M. Savanur, S. S. Malunavar, P. Prabhala, S. M. Sutar, R. G. Kalkhambkar and K. K. Laali, *Tetrahedron Lett.*, 2019, **60**, 151-159.
17. C. E. Coomber, V. Laserna, L. T. Martin, P. D. Smith, H. C. Hailes, M. J. Porter and T. D. Sheppard, *Org. Biomol. Chem.*, 2019, **17**, 6465-6469.
18. M. Albert-Soriano and I. M. Pastor, *Eur. J. Org. Chem.*, 2016, 5180-5188.
19. L.-H. Li, Z.-J. Niu and Y.-M. Liang, *Org. Biomol. Chem.*, 2018, **16**, 7792-7796.
20. Y. Zhao, S. Zhang, Y. Yamamoto, M. Bao, T. Jin and M. Terada, *Adv. Synth. Catal.*, 2019, **361**, 4817-4824.
21. K. Khosravi and S. Naserifar, *ChemistrySelect*, 2019, **4**, 1576-1585.
22. R. Ramkumar and S. Chandrasekaran, *S. Synthesis*, 2019, **51**, 921-932.
23. B.-L. Jiang, B.-H. Xu, M.-L. Wang, Z.-X. Li, D.-S. Liu and S.-J. Zhang, *Asian J. Org. Chem.*, 2018, **7**, 977-983.
24. A. Marandi, M. Bahadori, S. Tangestaninejad, M. Moghadam, V. Mirkhani, I. Mohammadpoor-Baltork, R. Frohnhoven, S. Mathur, A. Sandleben and A. Klein, *New J. Chem.*, 2019, **43**, 15585-15595.
25. P. Lu, T. Hou, X. Gu and P. Li, *Org. Lett.*, 2015, **17**, 1954-1957.
26. S. M. Resnick, F. A. Donate, T. C. Frank, T. C. Thyne and P. Foley, Eur. Patent 1490501, October 9, 2003.
27. I. Jain, R. Sharma and P. Malik, *Synth. Commun.*, 2019, **49**, 2952-2960.
28. G. Zheng and X. Li, *Synth. Commun.*, 2019, **49**, 933-941.

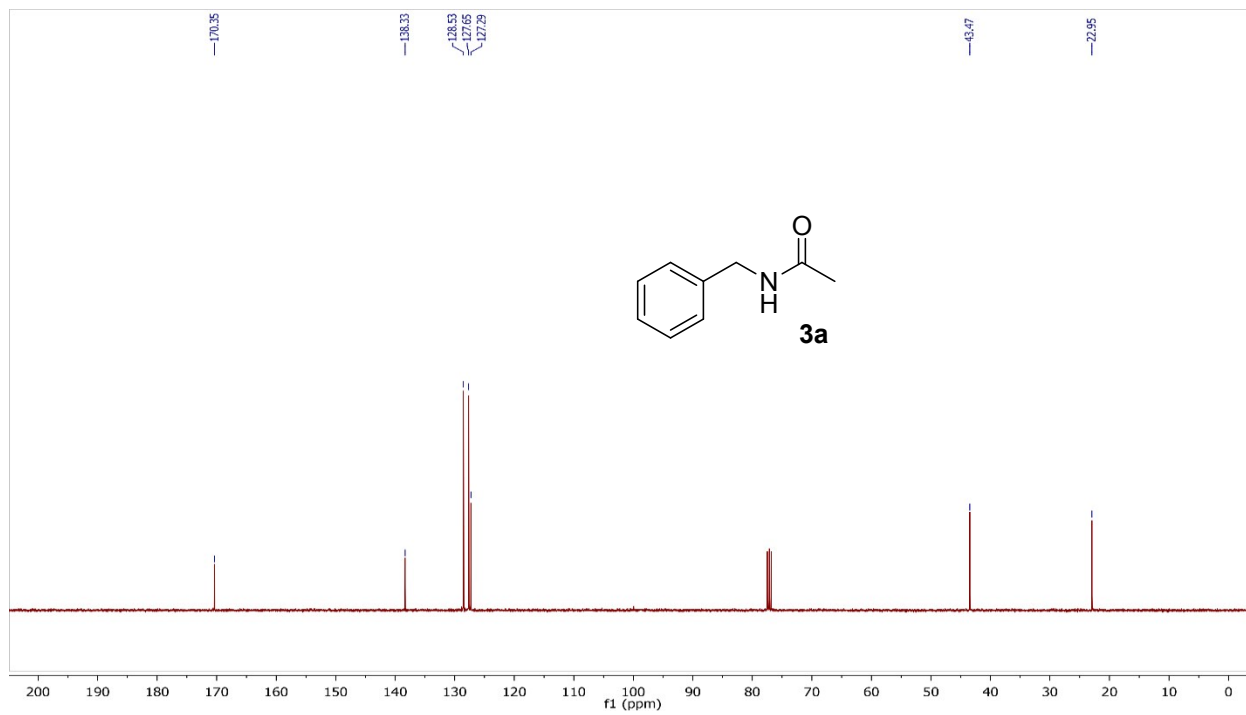
29. N. Lu, W.-H. Chang, R.-J. Wei, Y.-C. Fang, T.-W. Han, G.-Q. Wang, J.-Y. Chang, Y.-S. Wen and L.-K. Liu, *Tetrahedron*, 2016, **72**, 3468-3476.
30. V. A. Dyakonov, G. N. Kadikova, R. N. Nasretidinov, L. U. Dzhemileva and U. M. Dzhemilev, *J. Org. Chem.*, 2019, **84**, 9058-9066.
31. M. D. Vu, M. Das, A. Guo, Z.-E. Ang, M. Dokic, H. S. Soo and X.-W. Liu, *ACS Catal.*, 2019, **9**, 9009-9014.
32. B. Ghaffari, J. Mendes-Burak, K. W. Chan and C. Coperet, *Chem. Eur. J.*, 2019, **25**, 13869-13873.
33. R. Tiwari, A. Rahman, N. S. Bhat, S. B. Onkarappa, S. S. Mal and S. Dutta, *ChemistrySelect*, 2019, **4**, 9119-9123.
34. N. Wakita and S. Hara, *Tetrahedron*, 2010, **66**, 7939-7945.
35. Y. Yuan and X.-F. Wu, *Synlett*, 2019, **30**, 1820-1824.
36. S. Karthik, R. Sreedharan and T. Gandhi, *ChemistrySelect*, 2019, **4**, 175-180.
37. J. Jian, Z. Wang, L. Chen, Y. Gu, L. Miao, Y. Liu and Z. Zeng, *Synthesis*, 2019, **51**, 4078-4084.
38. H.-H. Chen, G.-Z. Wang, J. Han, M.-Y. Xu, Y.-Q. Zhao and H.-J. Xu, *Tetrahedron*, 2014, **70**, 212-217.
39. D. Ye, Z. Liu, H. Chen, J. L. Sessler and C. Lei, *Org. Lett.*, 2019, **21**, 6888-6892.
40. Q. Guo, T. Miyaji, R. Hara, B. Shen and T. Takahashi, *Tetrahedron*, 2002, **58**, 7327-7334.
41. M. Hatano, Y. Tabata, Y. Yoshida, K. Toh, K. Yamashita, Y. Ogura and K. Ishihara, *Green Chem.* 2018, **20**, 1193-1198.
42. H. Tsuji, K. Hashimoto and M. Kawatsura, *Org. Lett.*, 2019, **21**, 8837-8841.
43. Y. Chen, C. Li, Y. Cui, M. Sun, X. Jia and J. Li, *Synthesis*, 2019, **51**, 3667-3674.
44. J. Bloxham, C. J. Moody and A. M. Z. Slawin, *Tetrahedron*, 2002, **58**, 3709-3720.

45. S. Banerjee, V. Balasanthiran, R. T. Koodali and G. A. Sereda, *Org. Biomol. Chem.*, 2010, **8**, 4316-4321.
46. N. Scotti, F. Zaccheria, C. Evangelisti, R. Psaro and N. Ravasio, *Catal. Sci. Technol.*, 2017, **7**, 1386-1393.
47. S. Zhang, H. Neumann and M. Beller, *Org. Lett.*, 2019, **21**, 3528-3532.
48. W. Yu, S. Yang, F. Xiong, T. Fan, Y. Feng, Y. Huang, J. Fu and T. Wang, *Org. Biomol. Chem.*, 2018, **16**, 3099-3103.
49. J.-F. Li, Y.-F. Wang, Y.-Y. Wu, W.-J. Liu and J.-W. Wang, *Catal. Lett.*, 2019, DOI: 10.1007/s10562-019-02966-6.
50. L. Sekerova, E. Vyskocilova, L. Cerveny and J. Sedlacek, *Tetrahedron*, 2019, **75**, 2877-2882.
51. Y. Li, H.-H. Chen, C.-F. Wang, X.-L. Xu and Y.-S. Feng, *Tetrahedron Lett.*, 2012, **53**, 5796-5799.
52. S. Ding, L. Bai, Y. Cong and T. Chen, *Synth. Commun.*, 2018, **48**, 2559-2565.
53. L. O. Okunrobo and C. O. Usifoh, *Afr. J. Biotechnol.*, 2006, **5**, 643-647.
54. H. Valizadeh and E. Khalili, *J. Iran. Chem. Soc.*, 2012, **9**, 529-534.

## SELECTED NMR SPECTRA

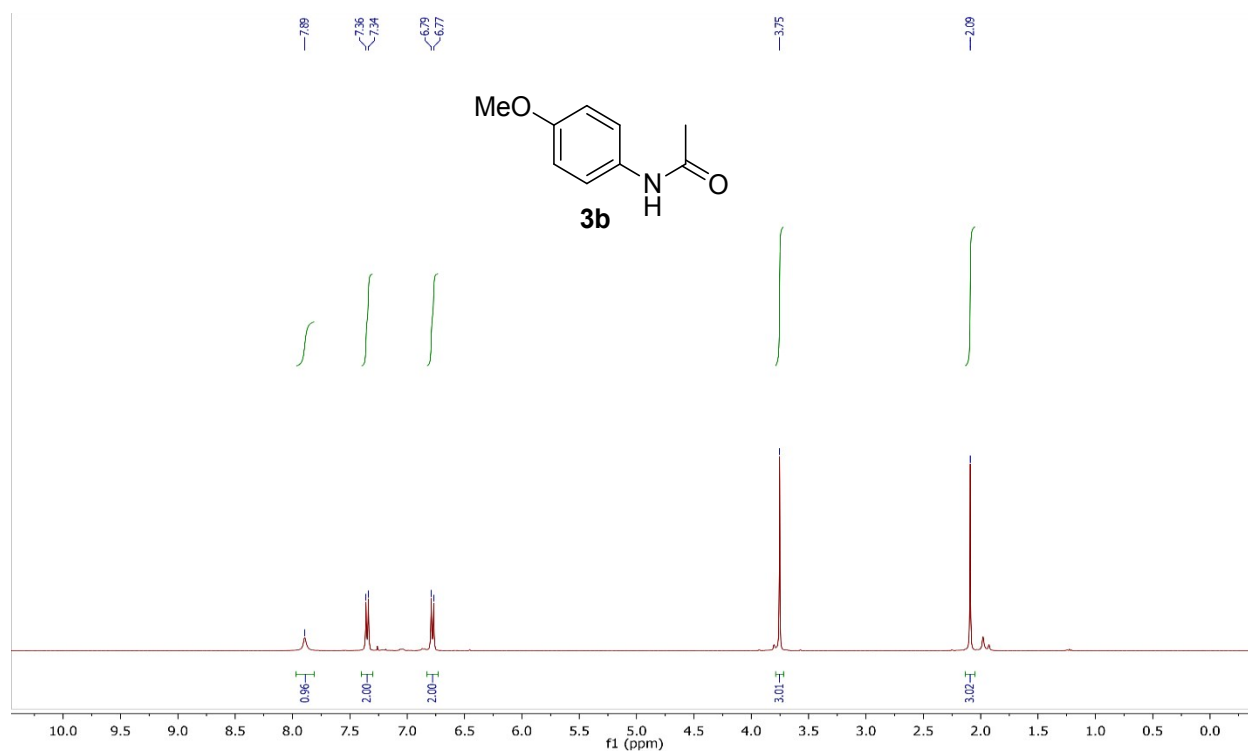


<sup>1</sup>H NMR of compound 3a

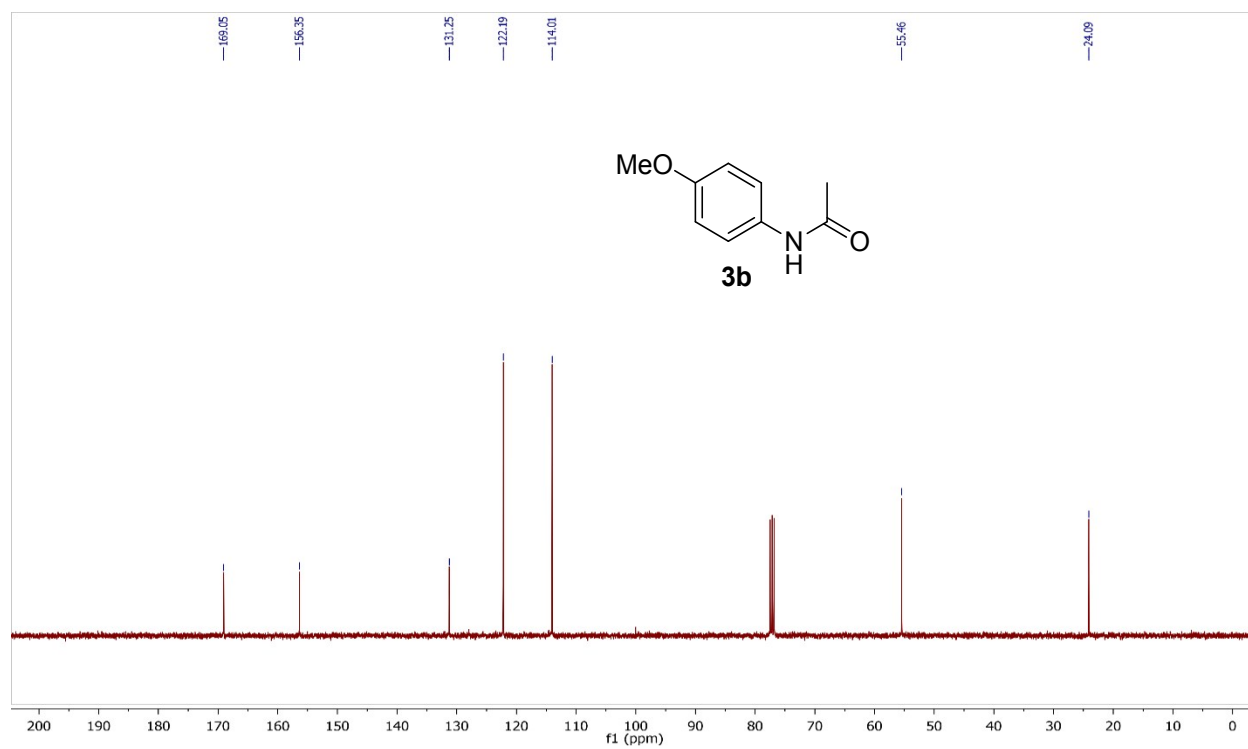


<sup>13</sup>C{<sup>1</sup>H} NMR of compound 3a

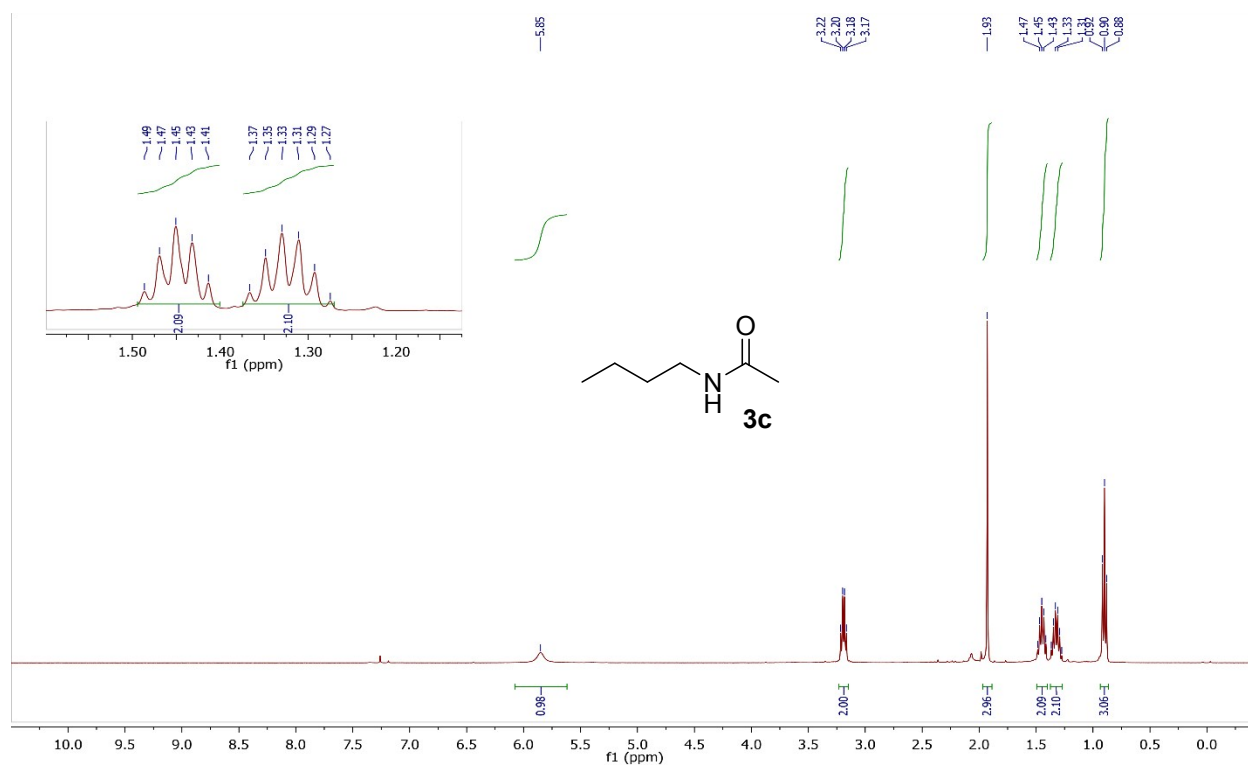




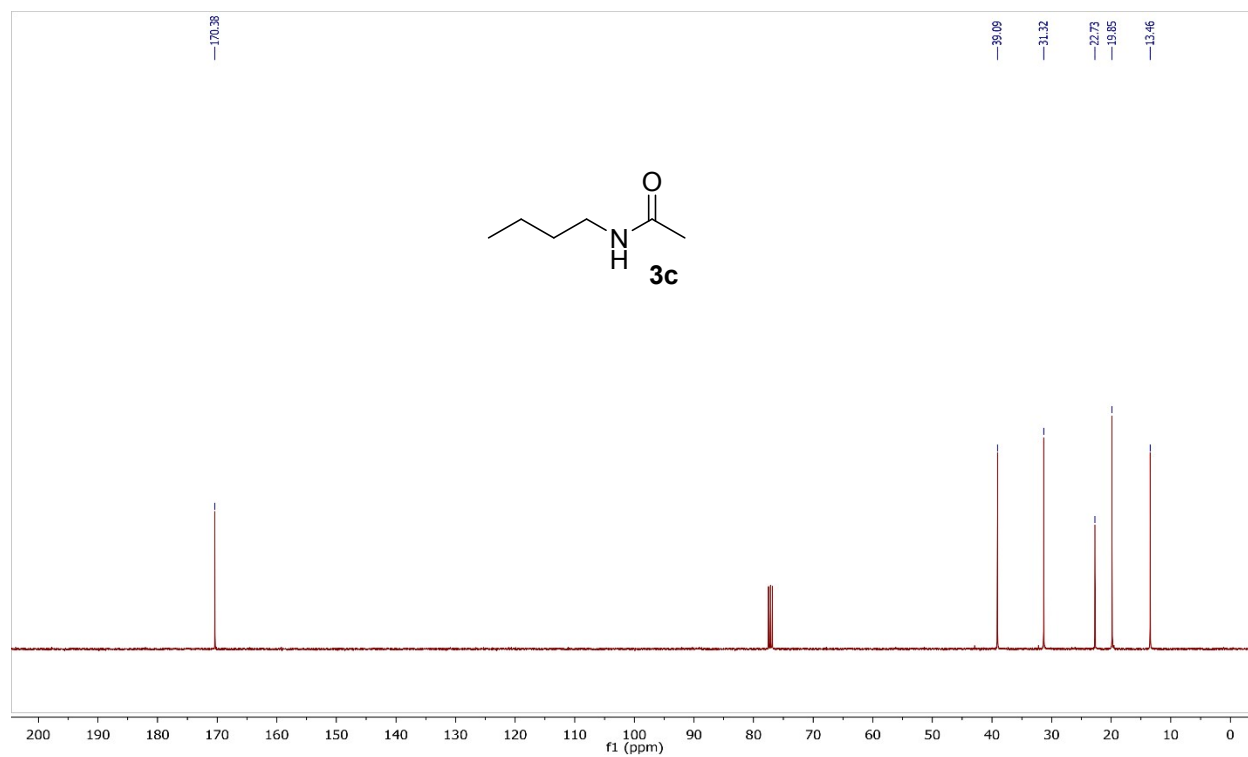
$^1\text{H}$  NMR of compound **3b**



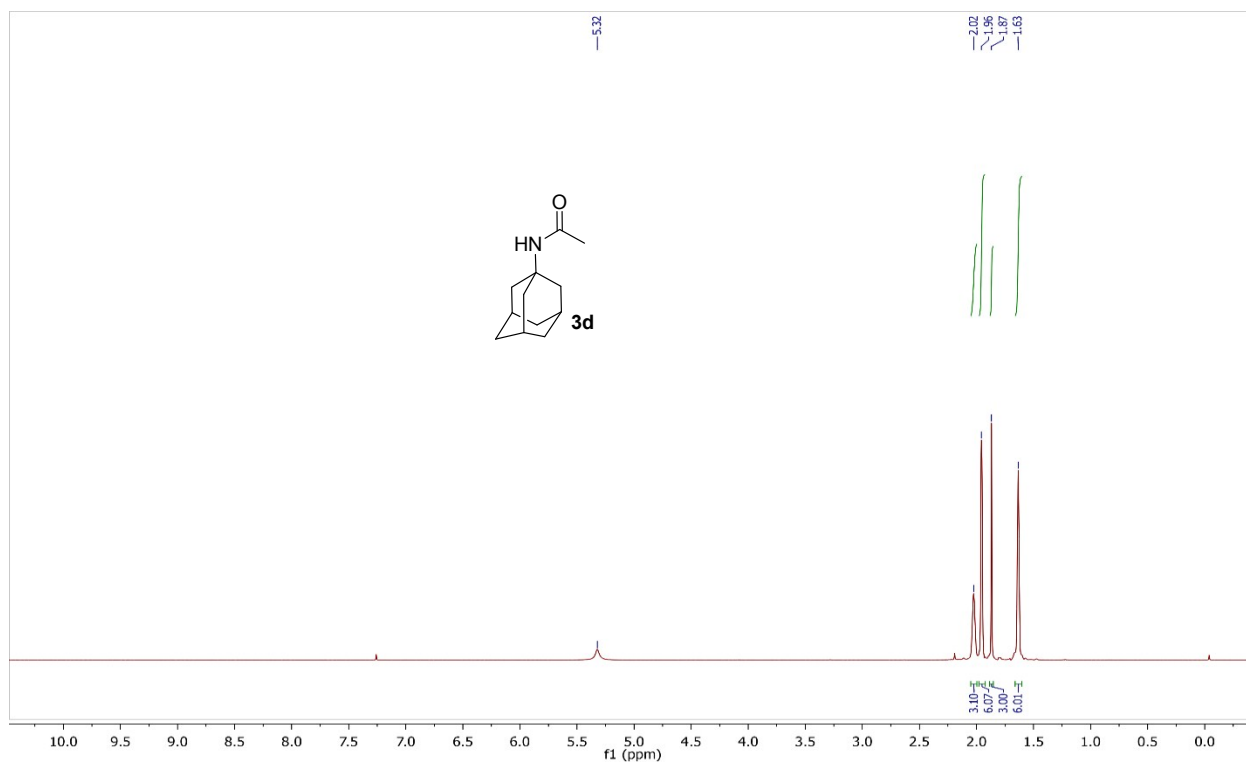
$^{13}\text{C}$   $\{^1\text{H}\}$  NMR of compound **3b**



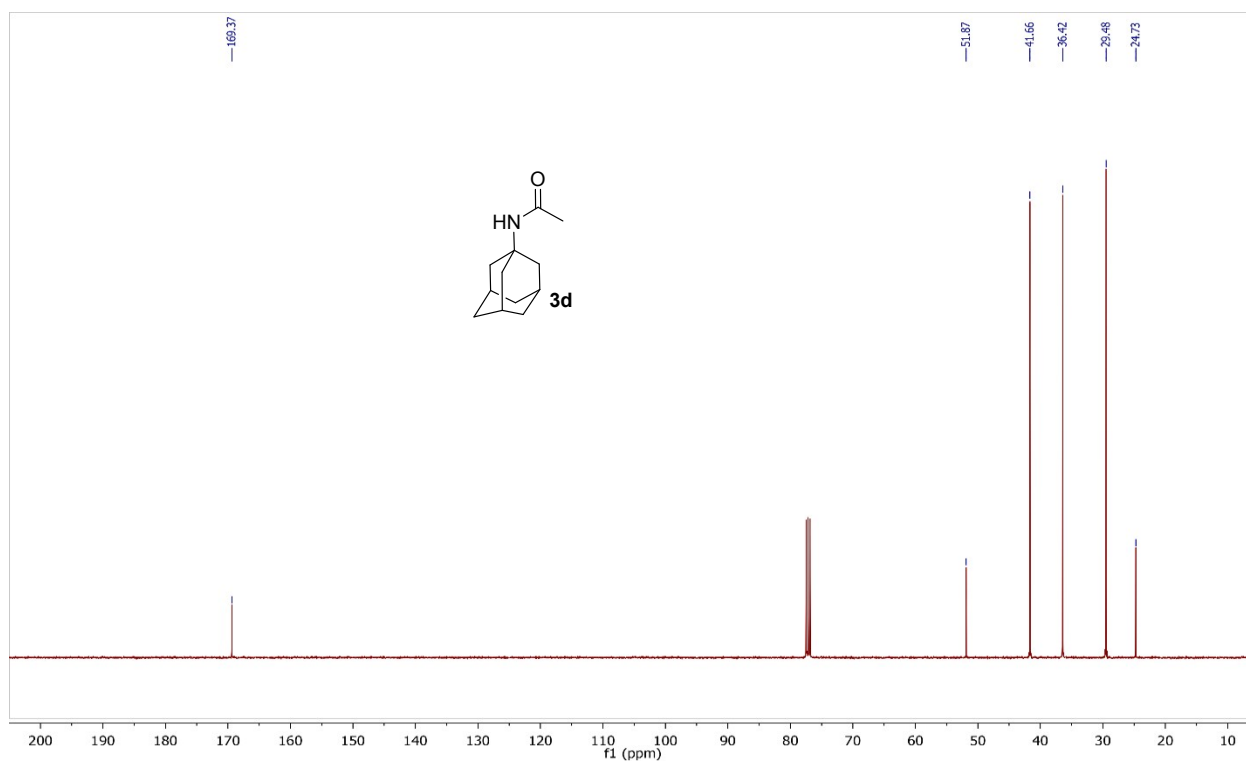
$^1\text{H}$  NMR of compound **3c**



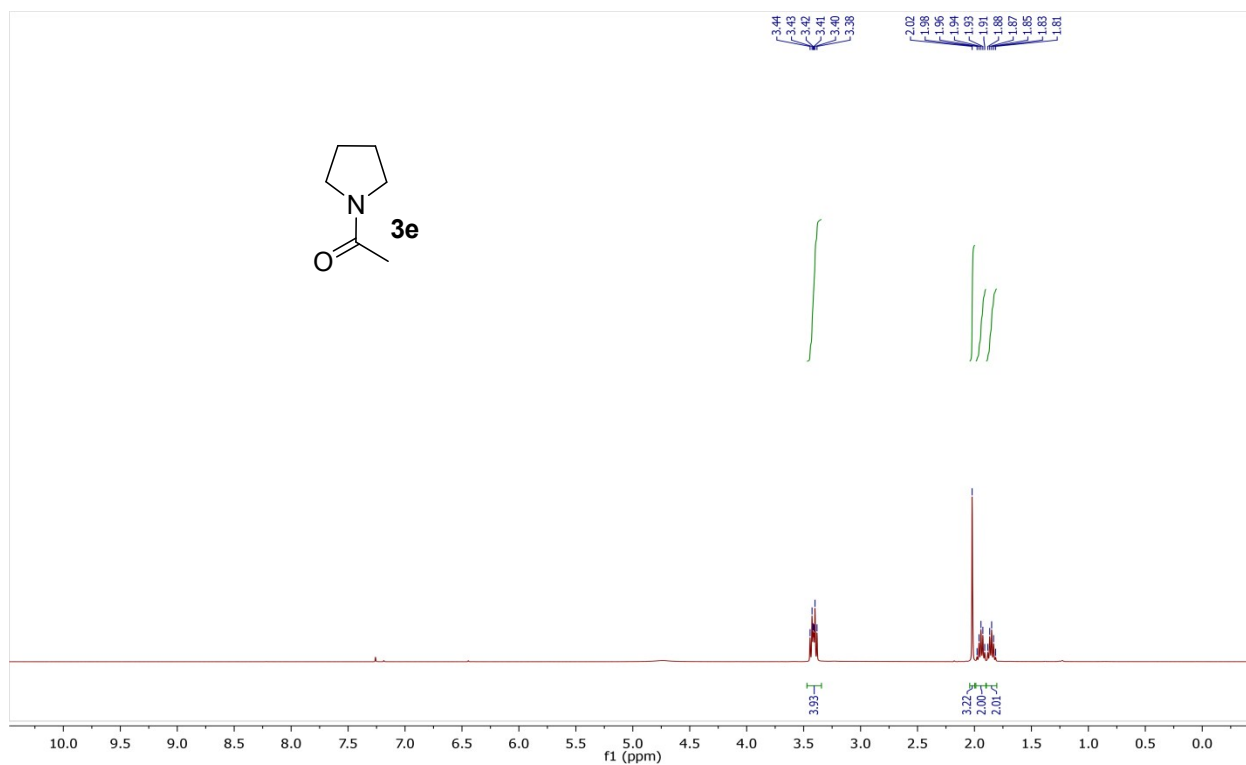
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **3c**



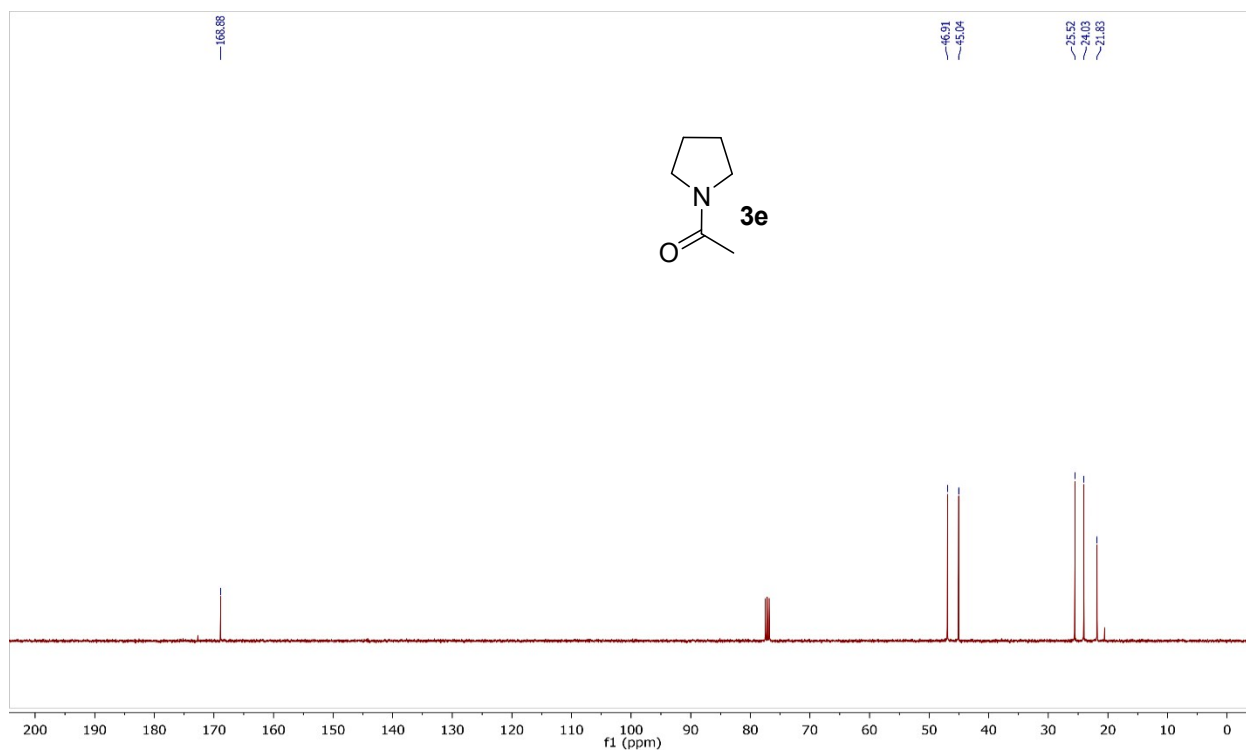
$^1\text{H}$  NMR of compound **3d**



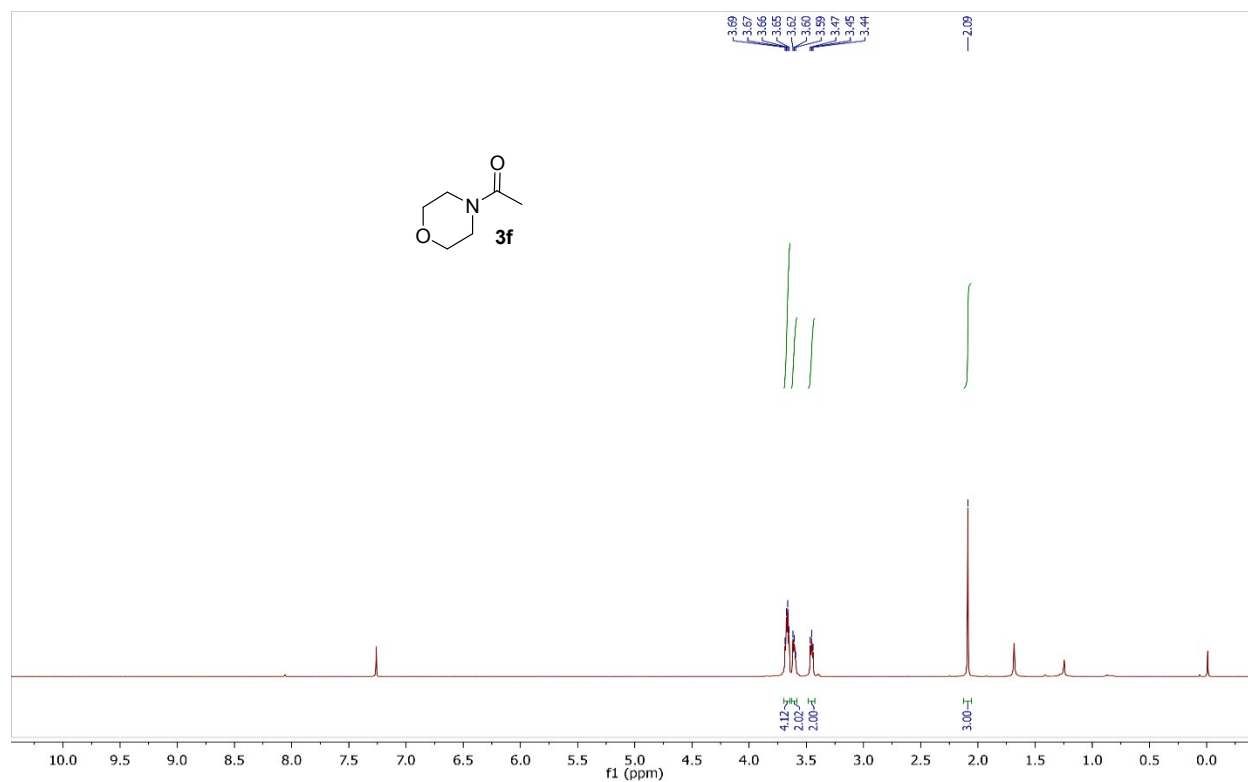
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **3d**



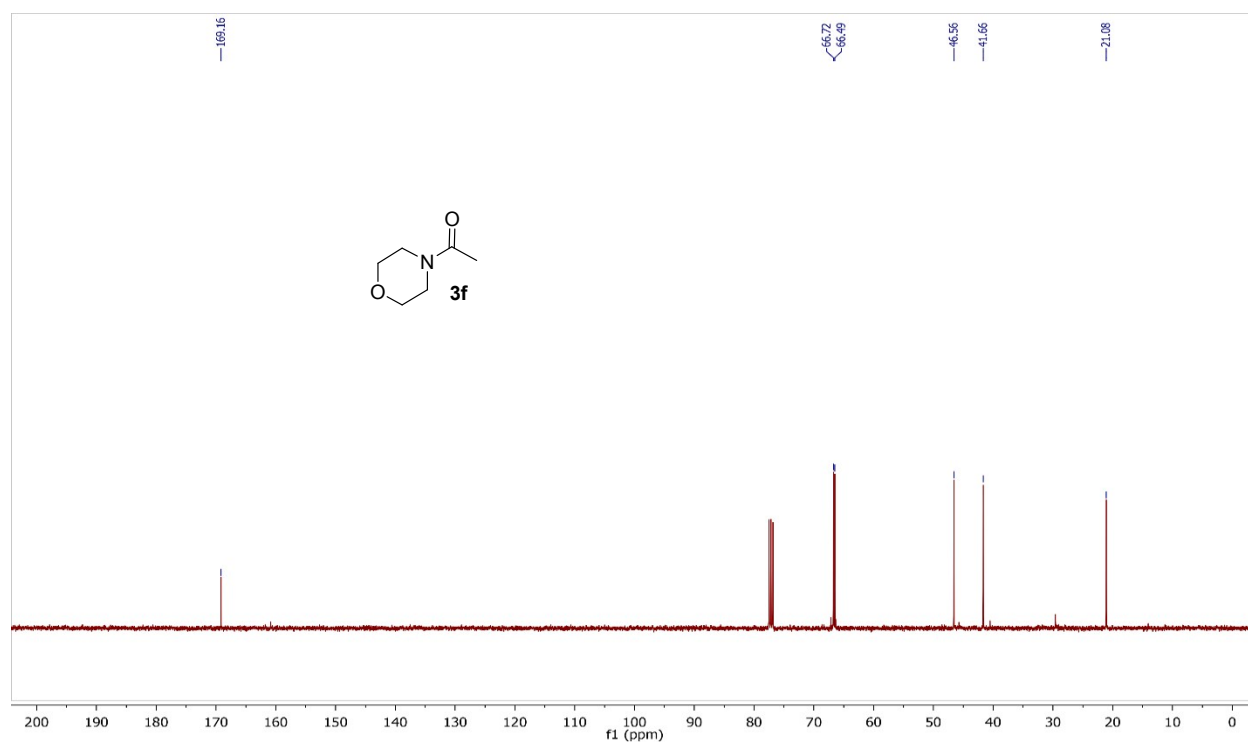
$^1\text{H}$  NMR of compound **3e**



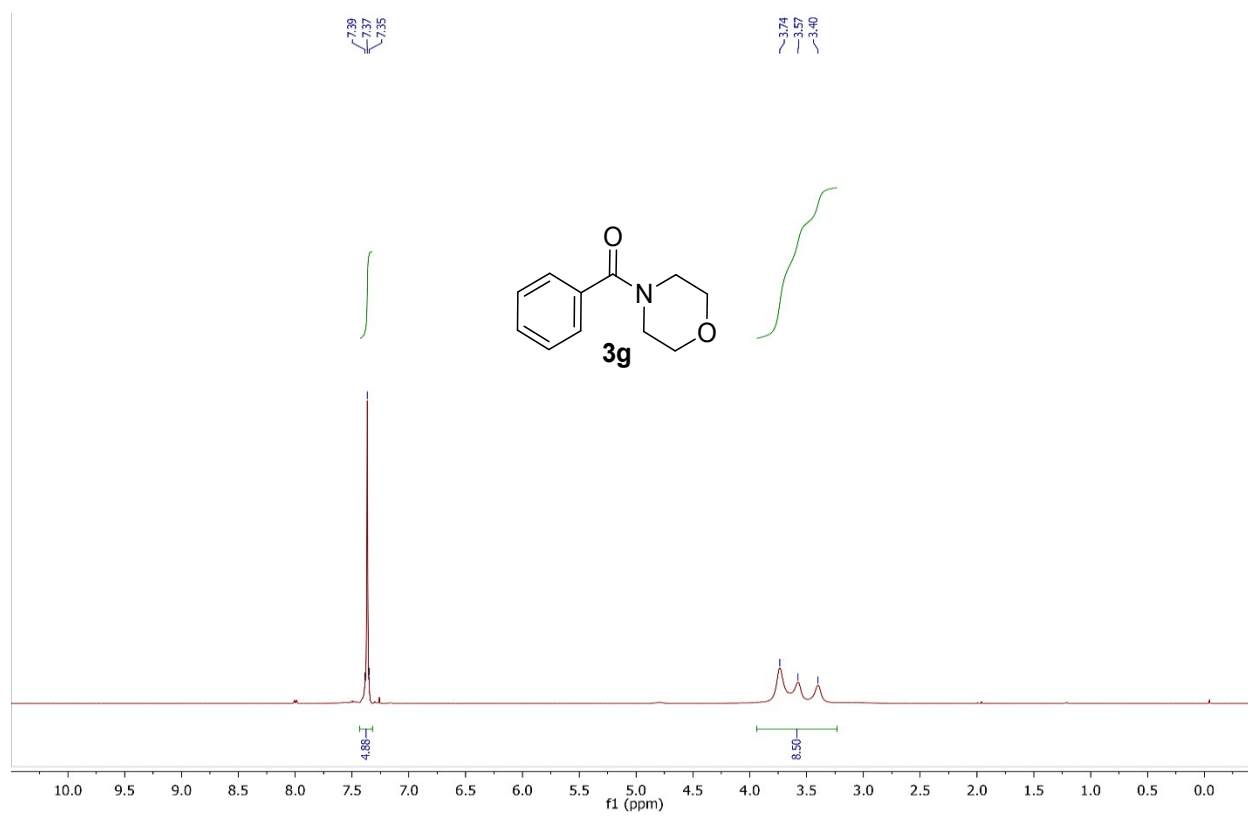
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **3e**



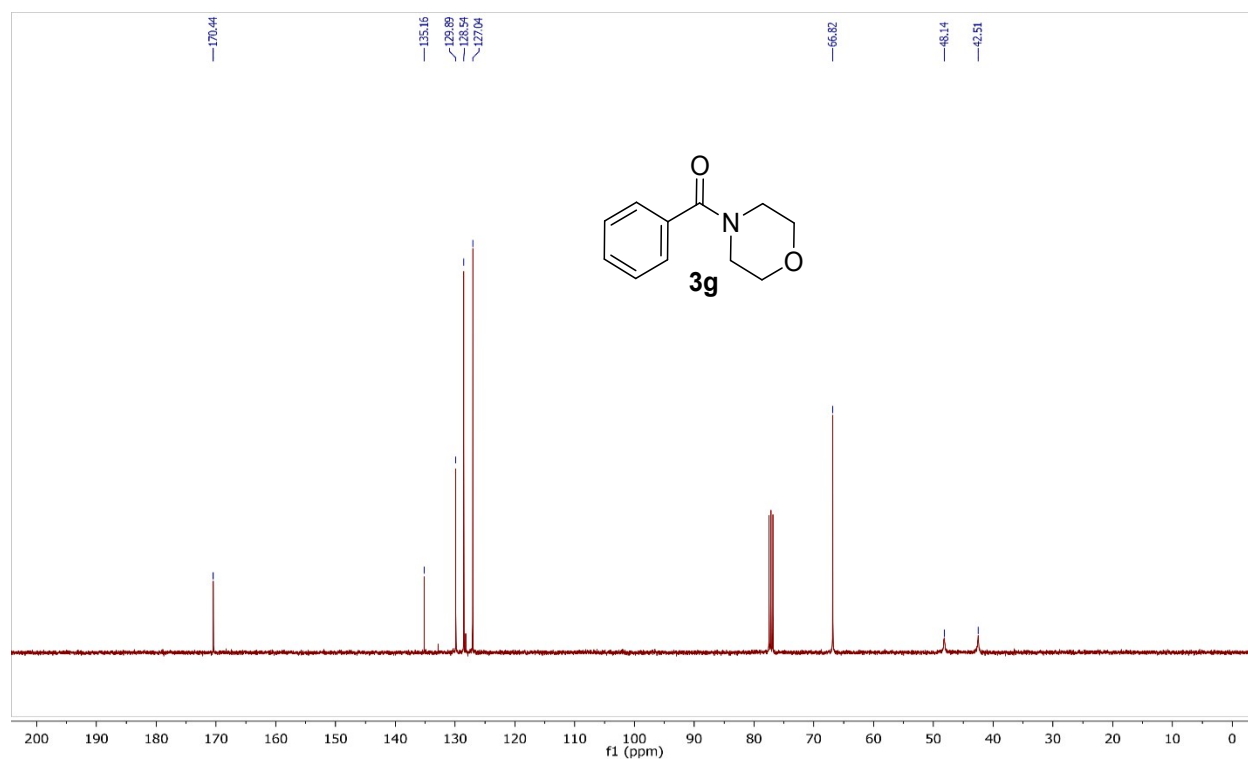
$^1\text{H}$  NMR of compound **3f**



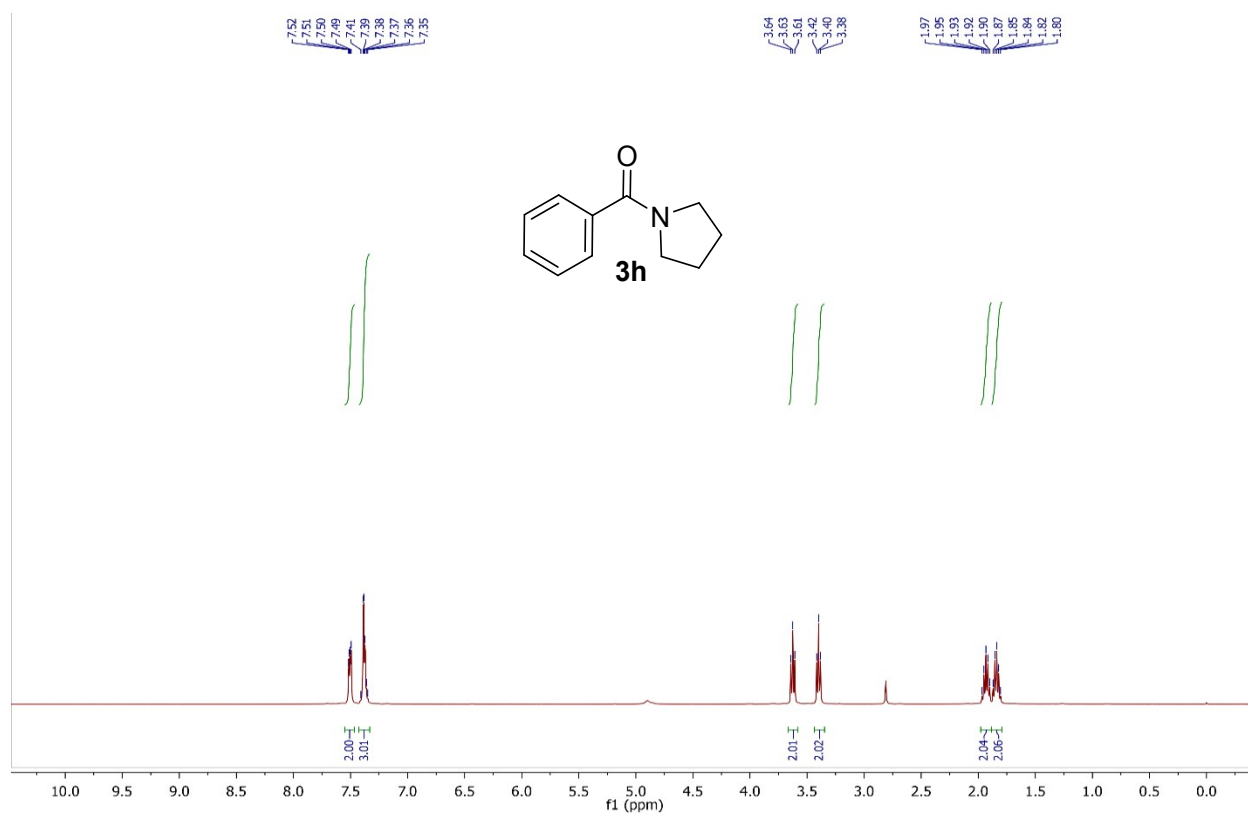
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **3f**



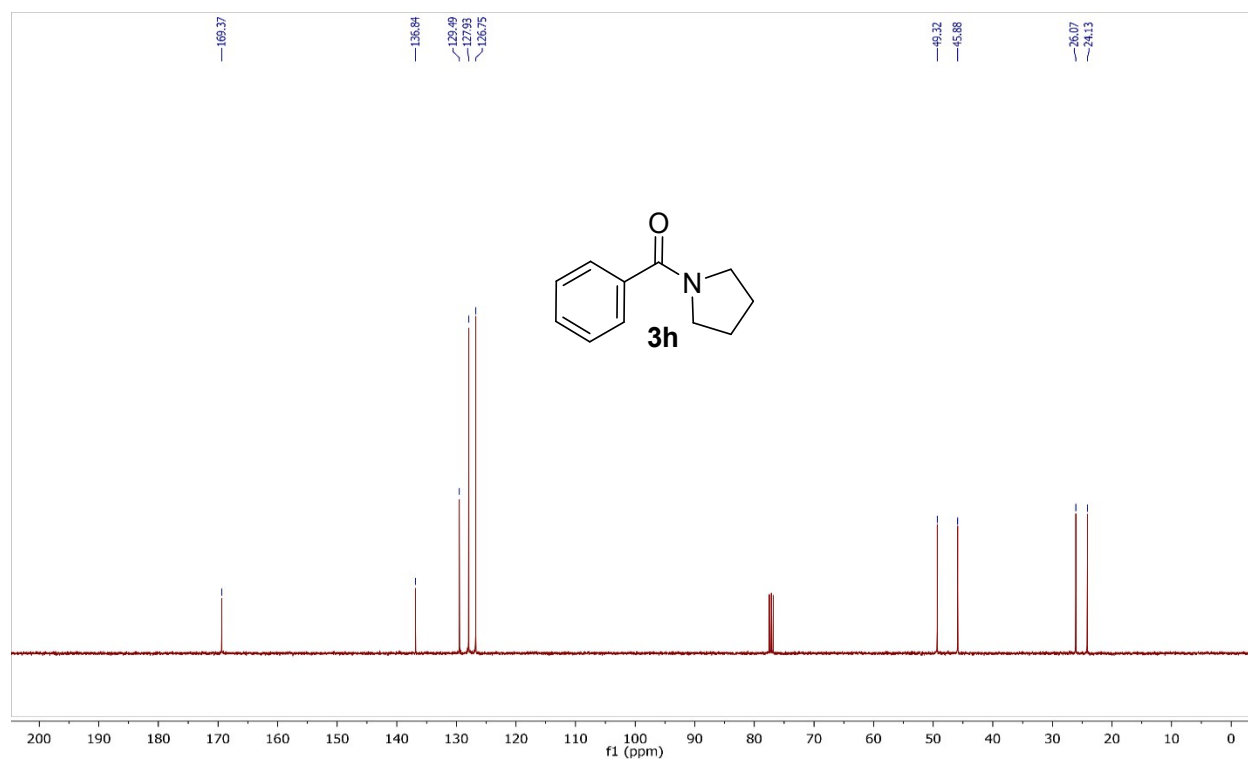
$^1\text{H}$  NMR of compound **3g**



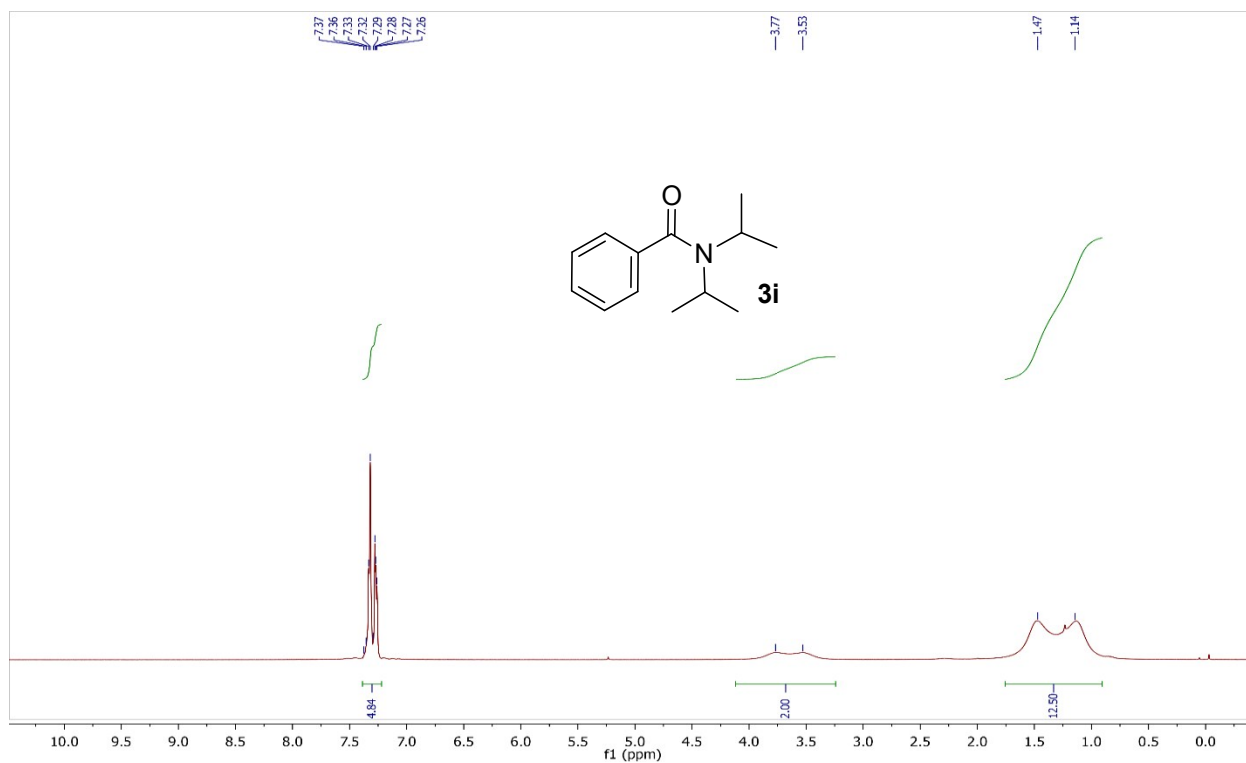
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **3g**



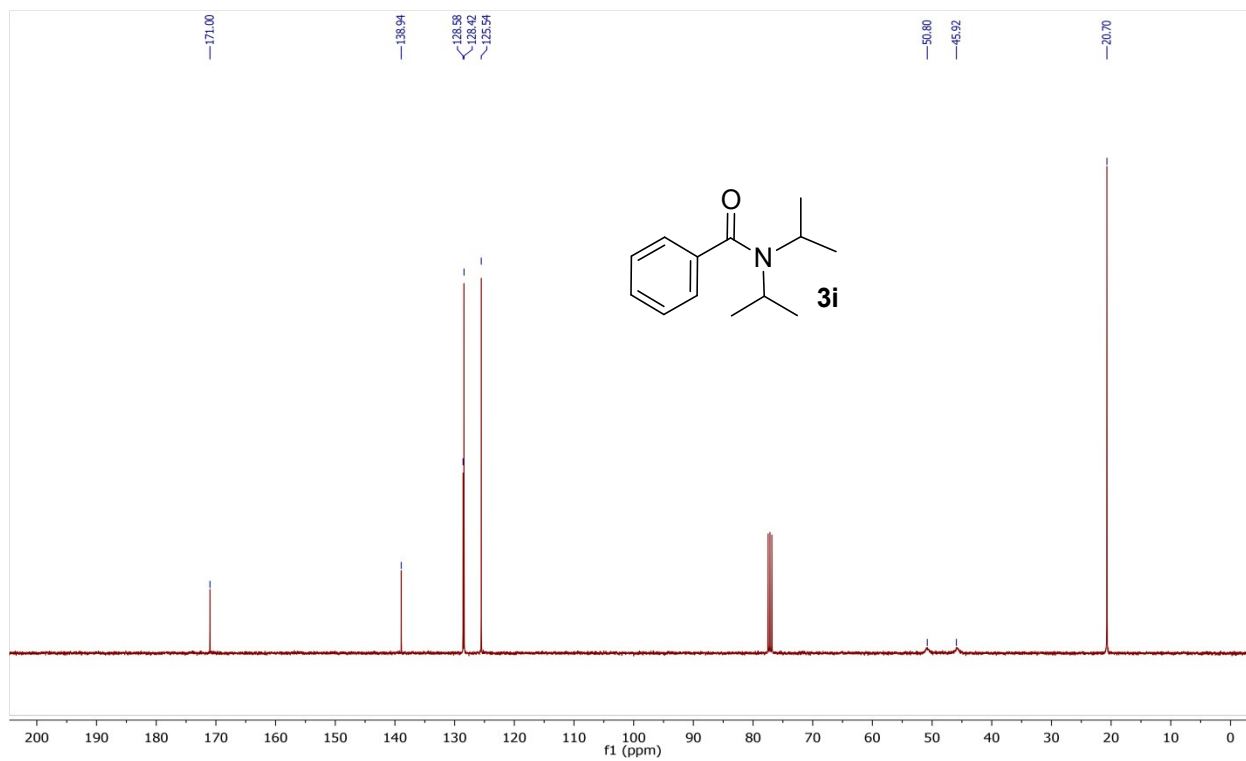
<sup>1</sup>H NMR of compound 3h



<sup>13</sup>C{<sup>1</sup>H} NMR of compound 3h

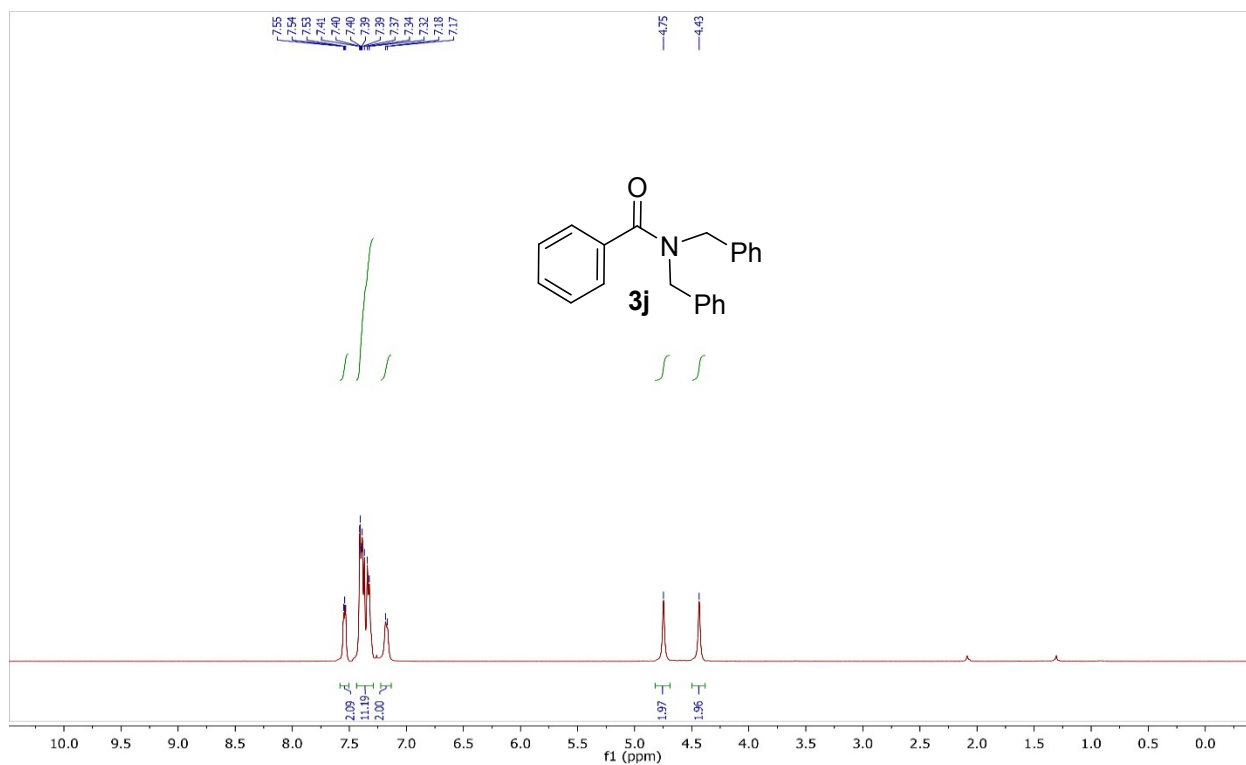


<sup>1</sup>H NMR of compound **3i**

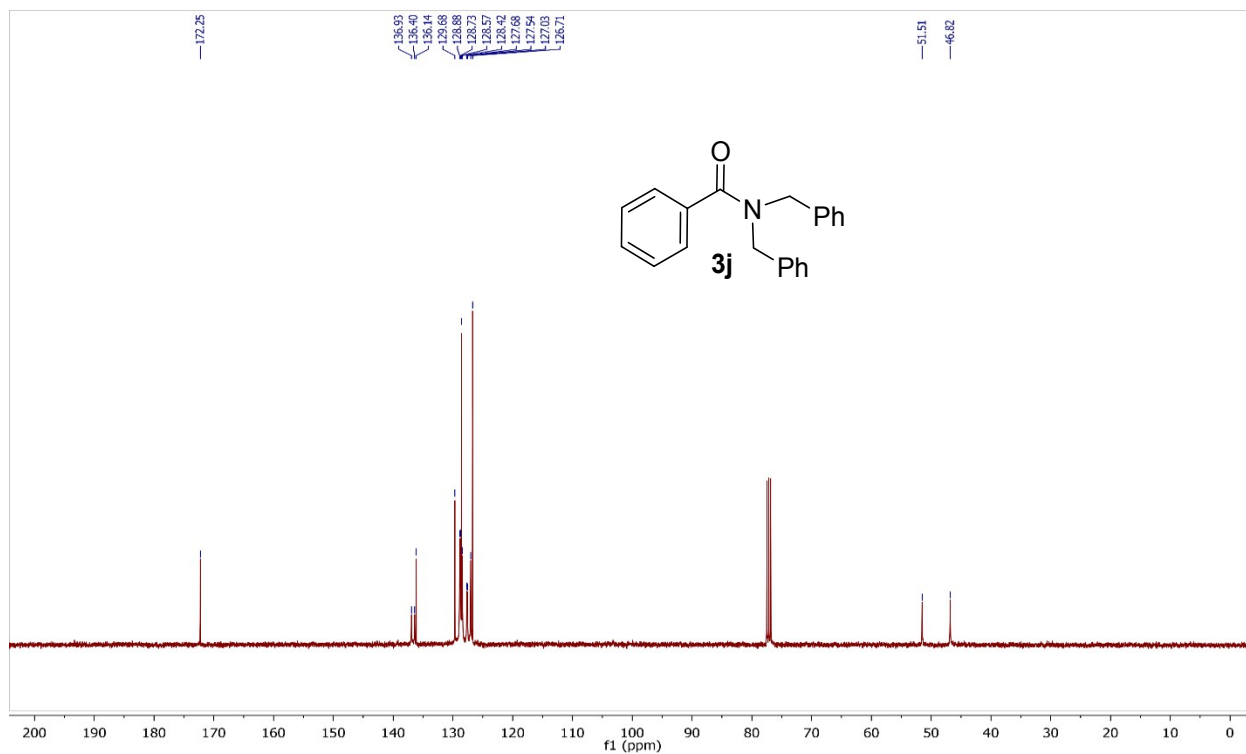


<sup>13</sup>C{<sup>1</sup>H} NMR of compound **3i**

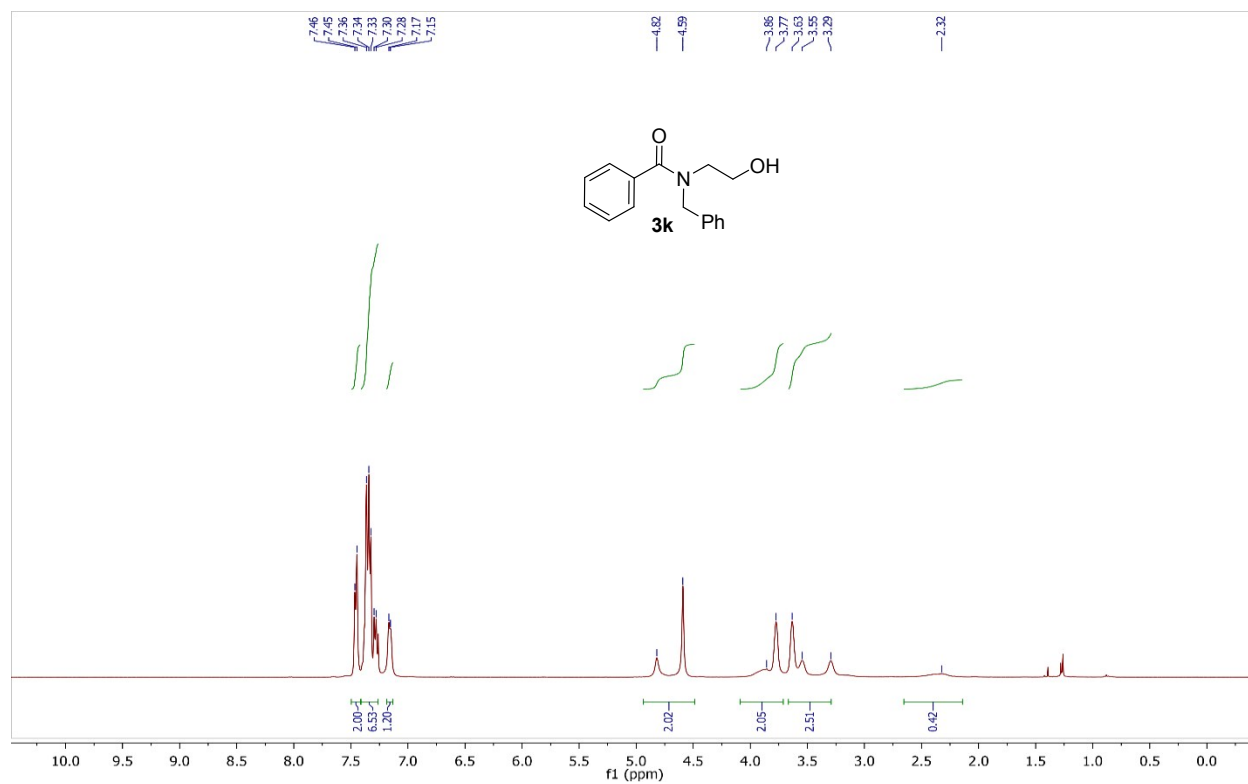




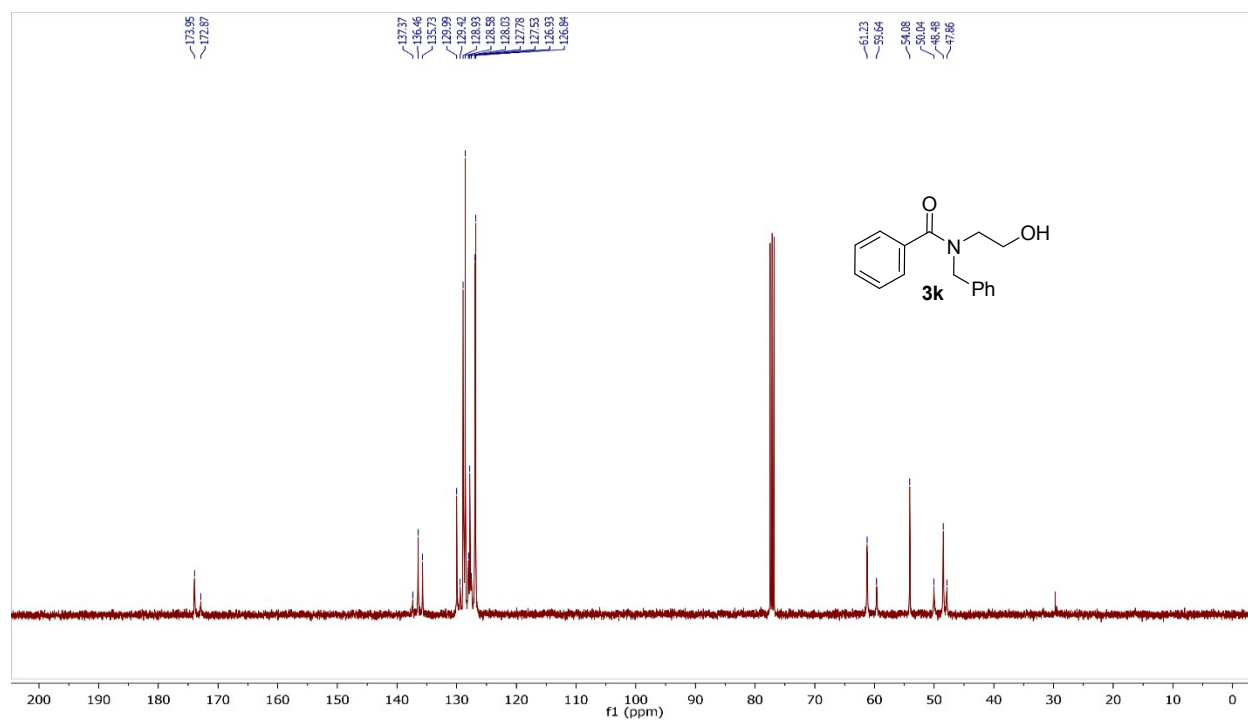
$^1\text{H NMR}$  of compound **3j**



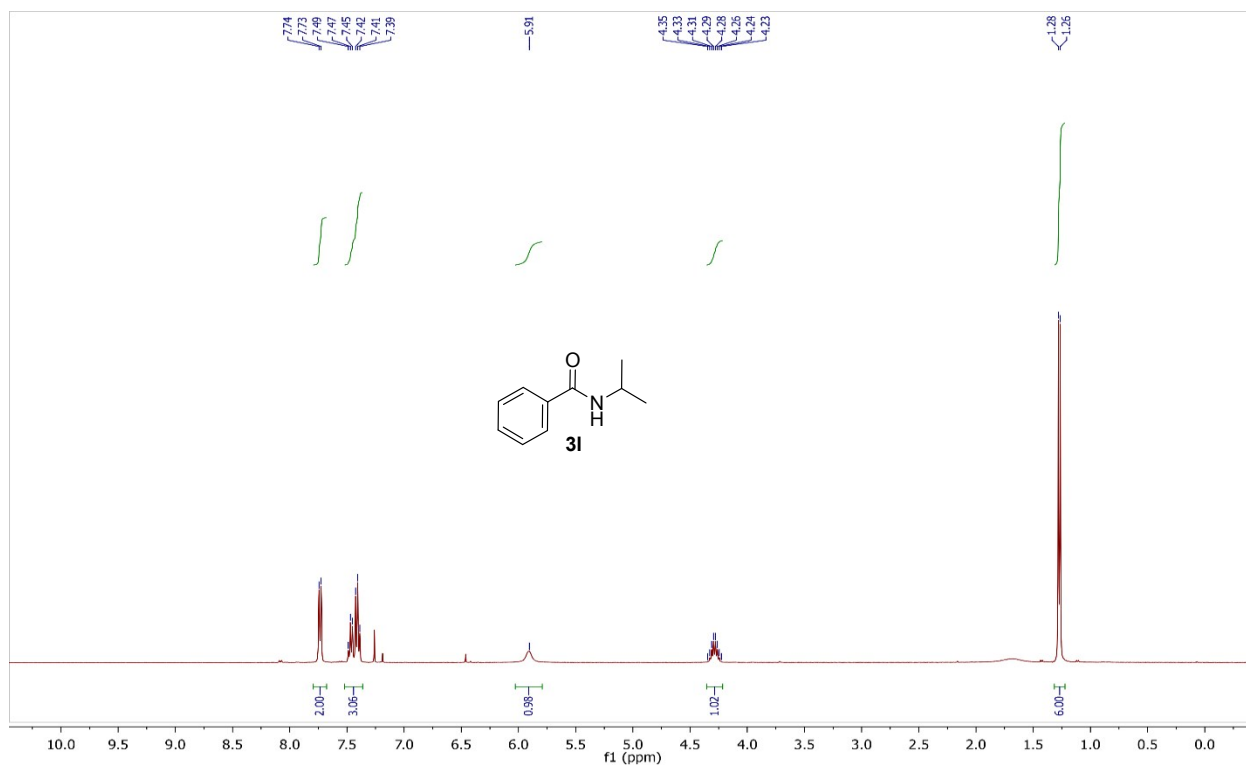
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **3j**



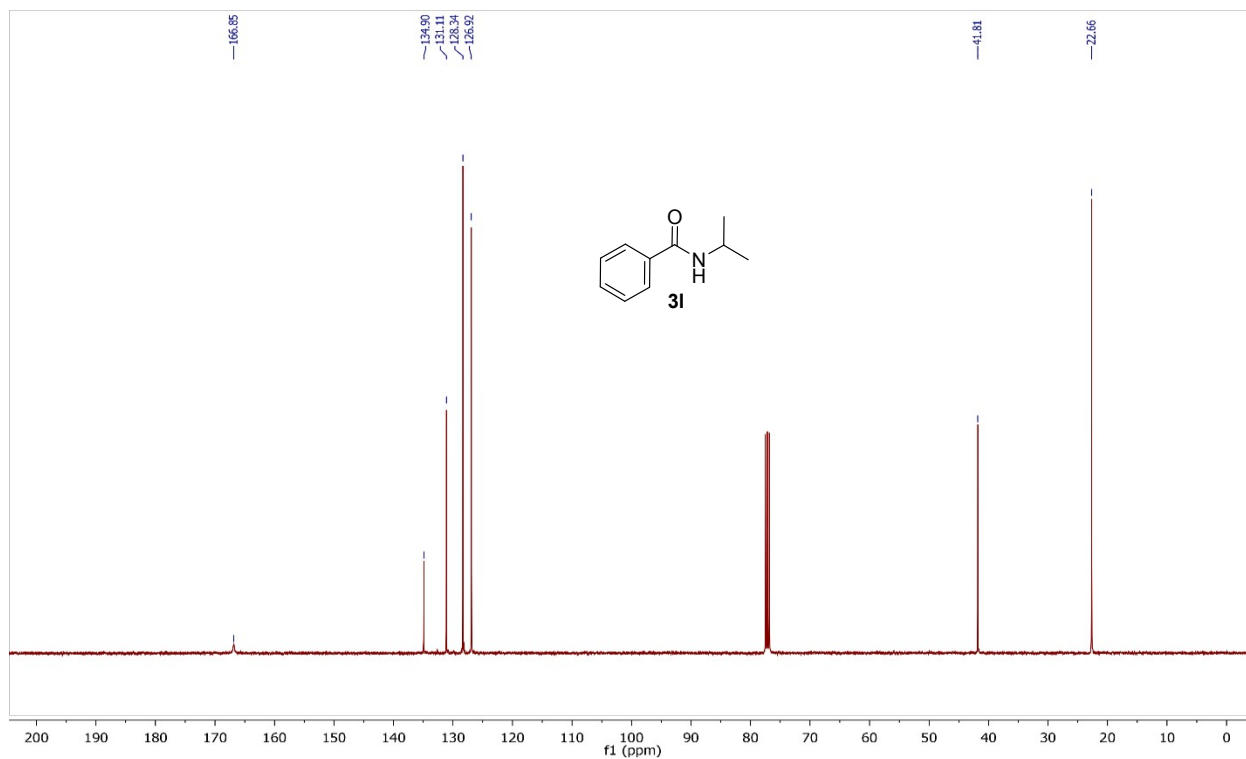
$^1\text{H}$  NMR of compound **3k**



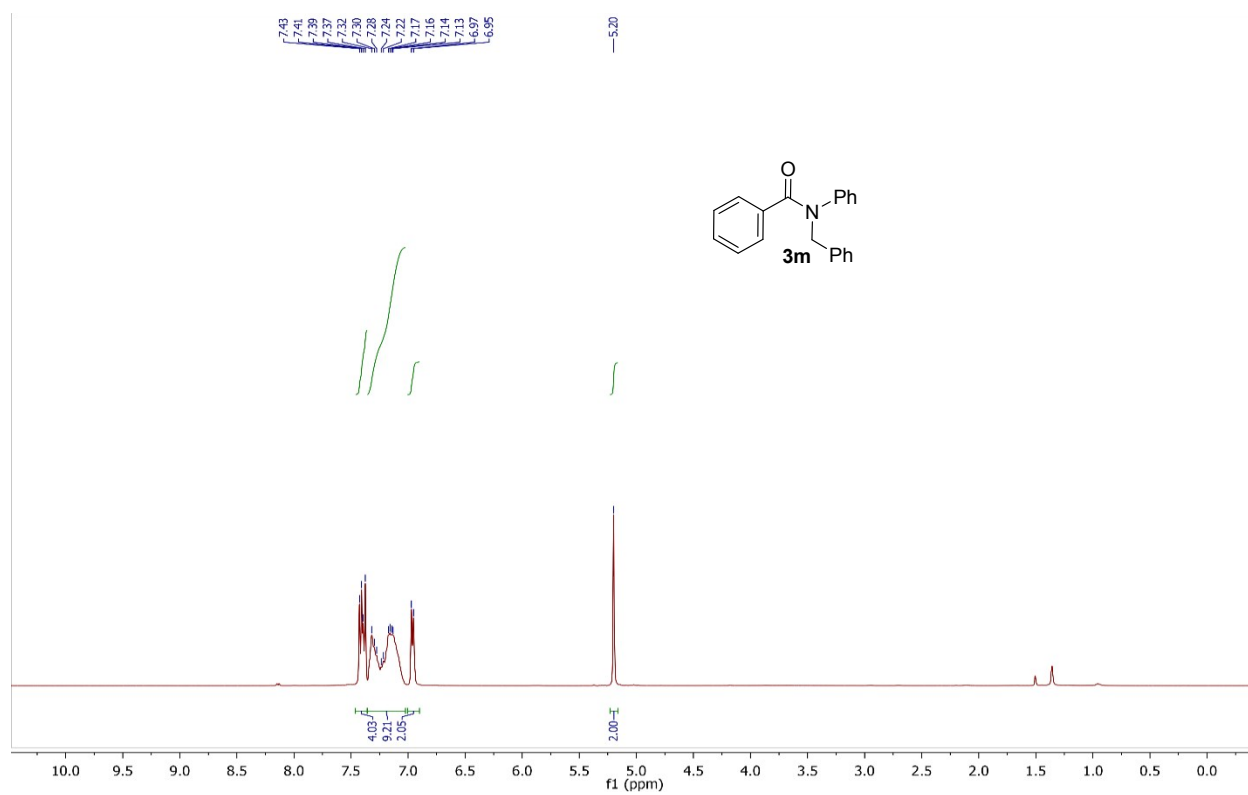
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **3k**



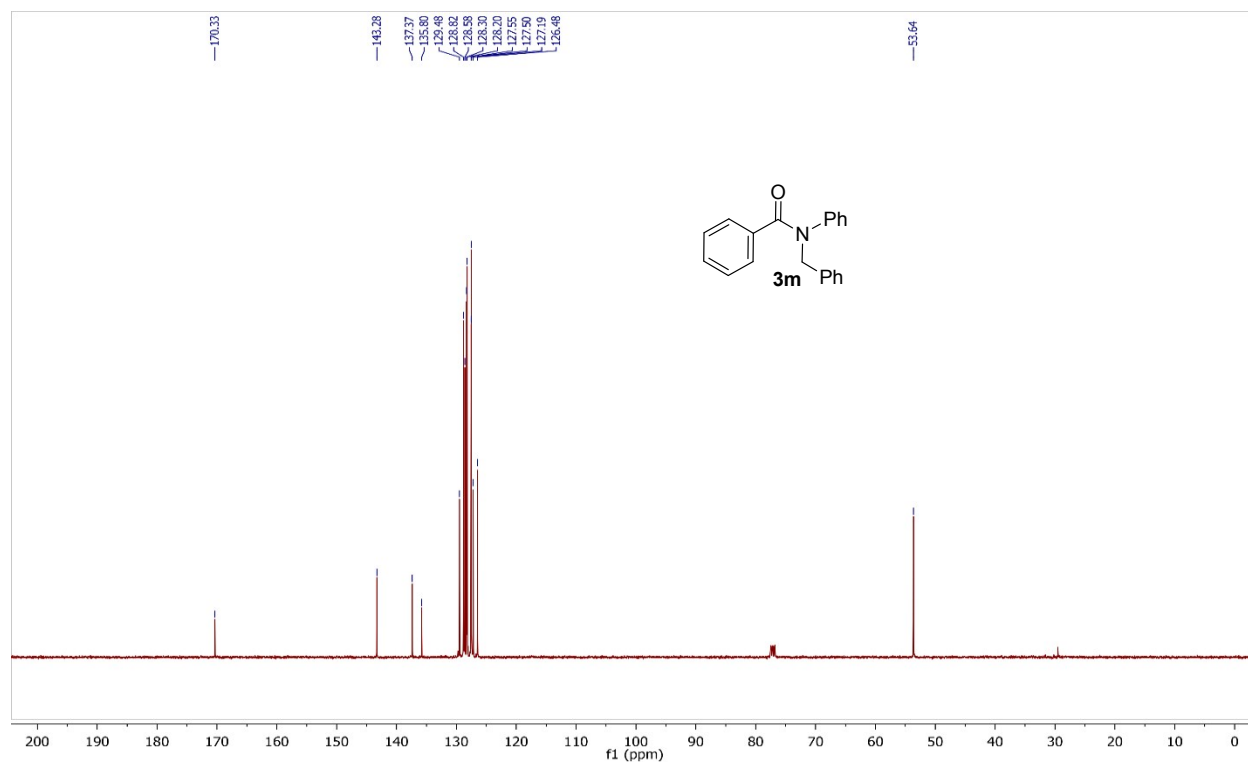
$^1\text{H}$  NMR of compound **31**



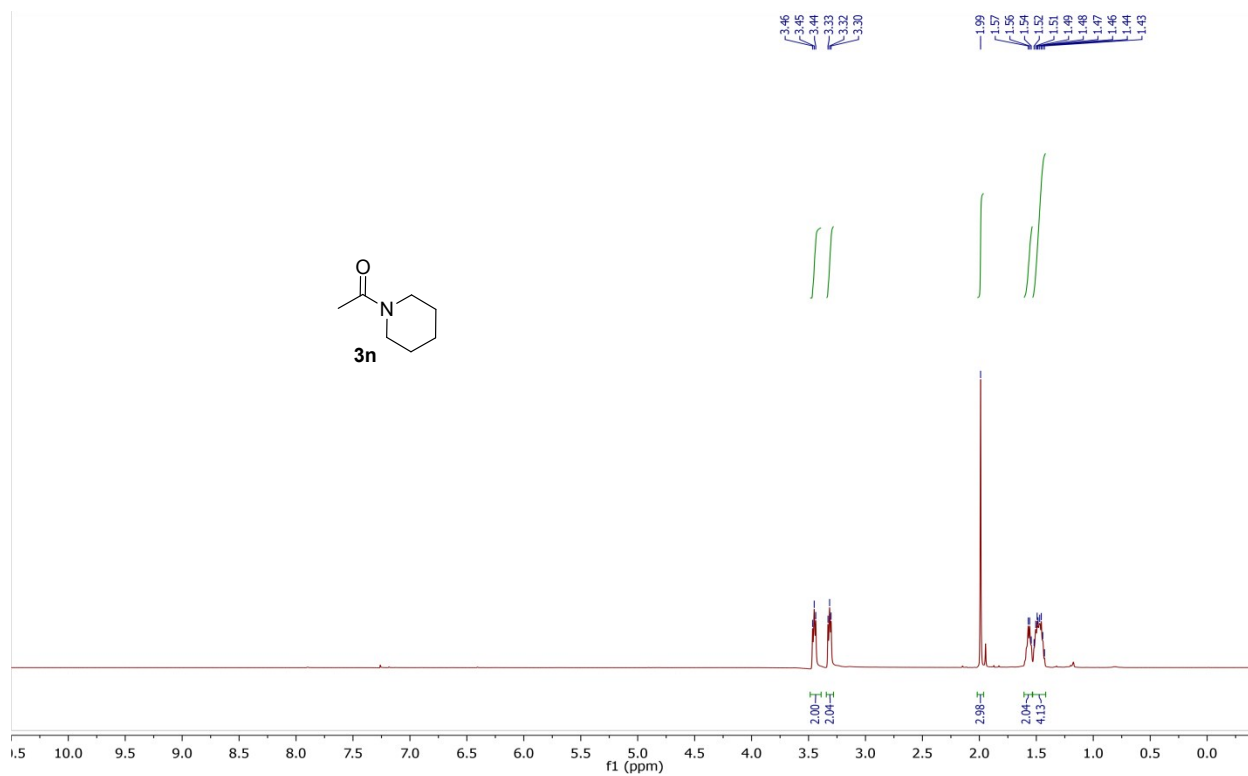
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **31**



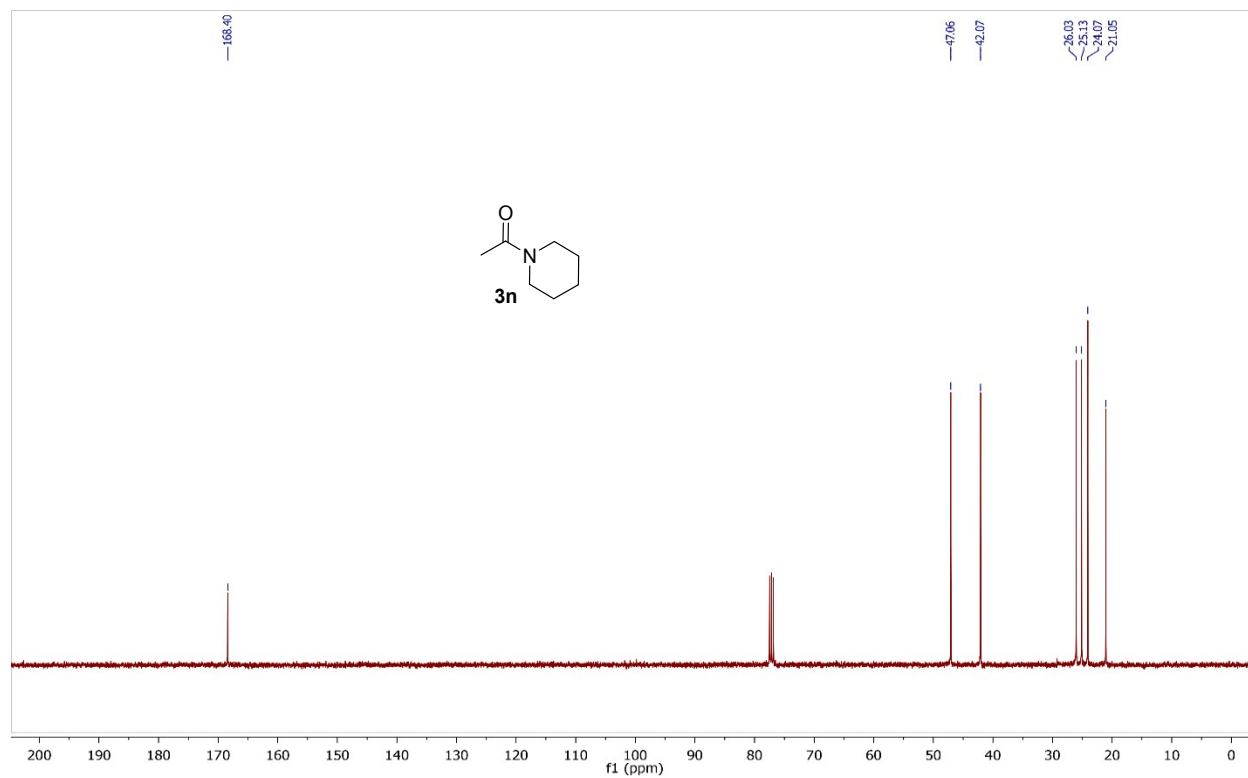
$^1\text{H}$  NMR of compound **3m**



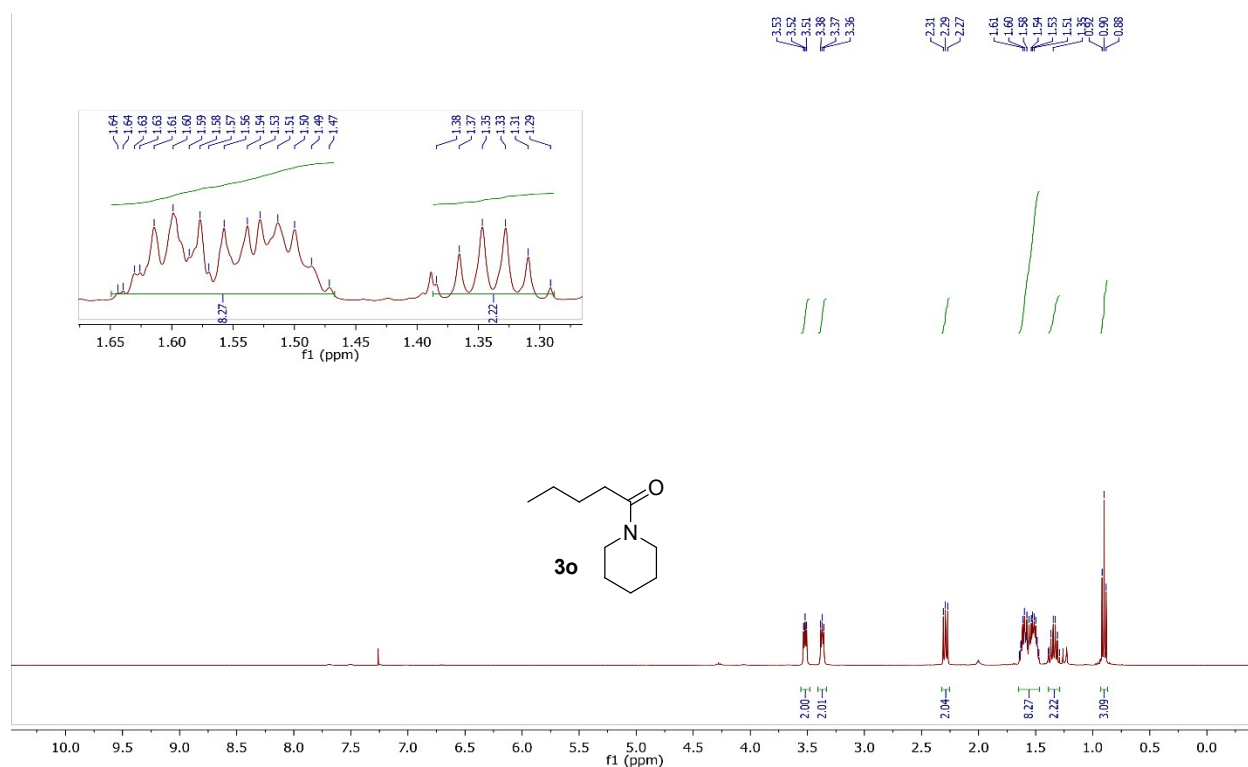
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **3m**



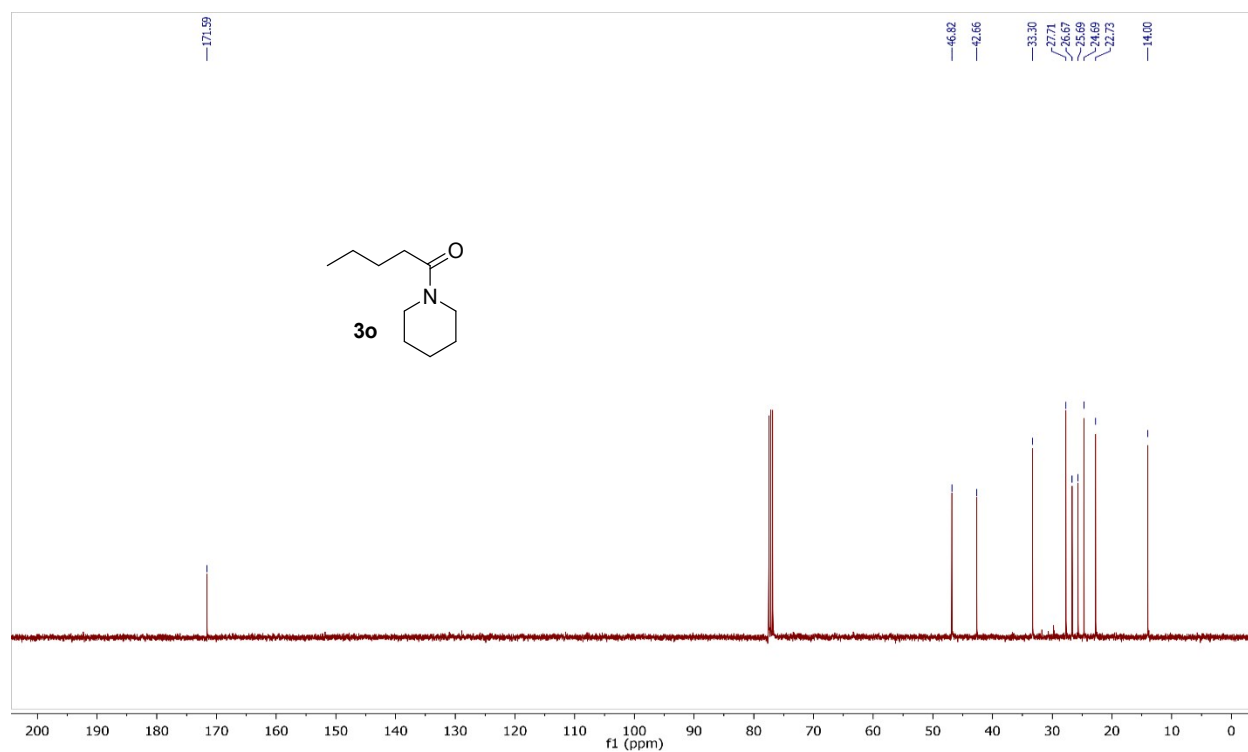
**<sup>1</sup>H NMR of compound 3n**



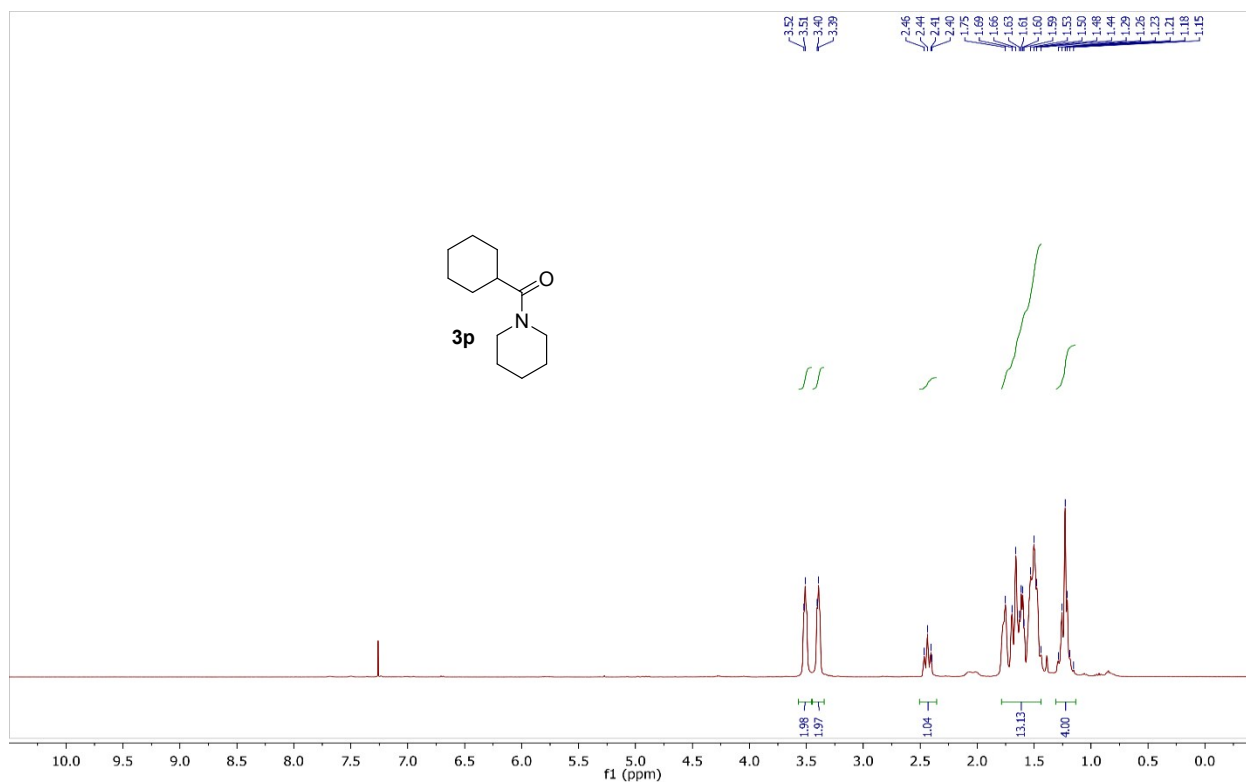
**<sup>13</sup>C{<sup>1</sup>H} NMR of compound 3n**



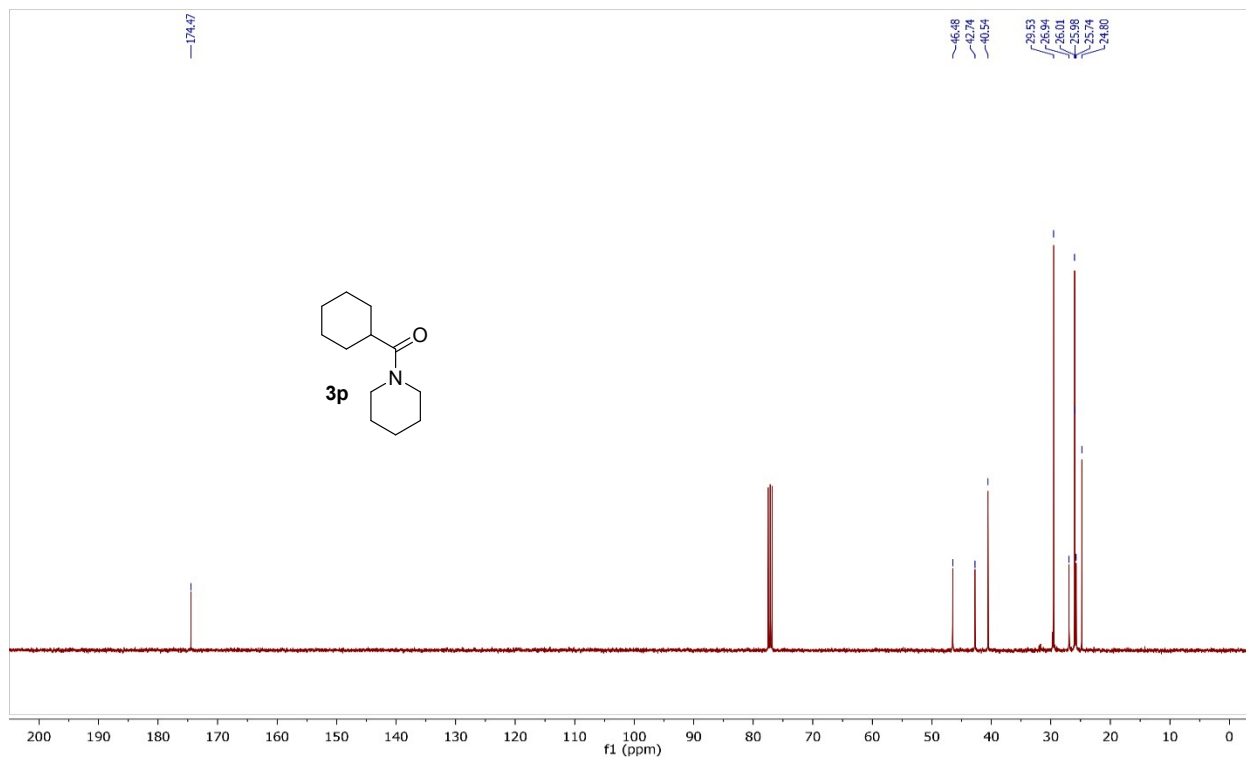
$^1\text{H}$  NMR of compound **3o**



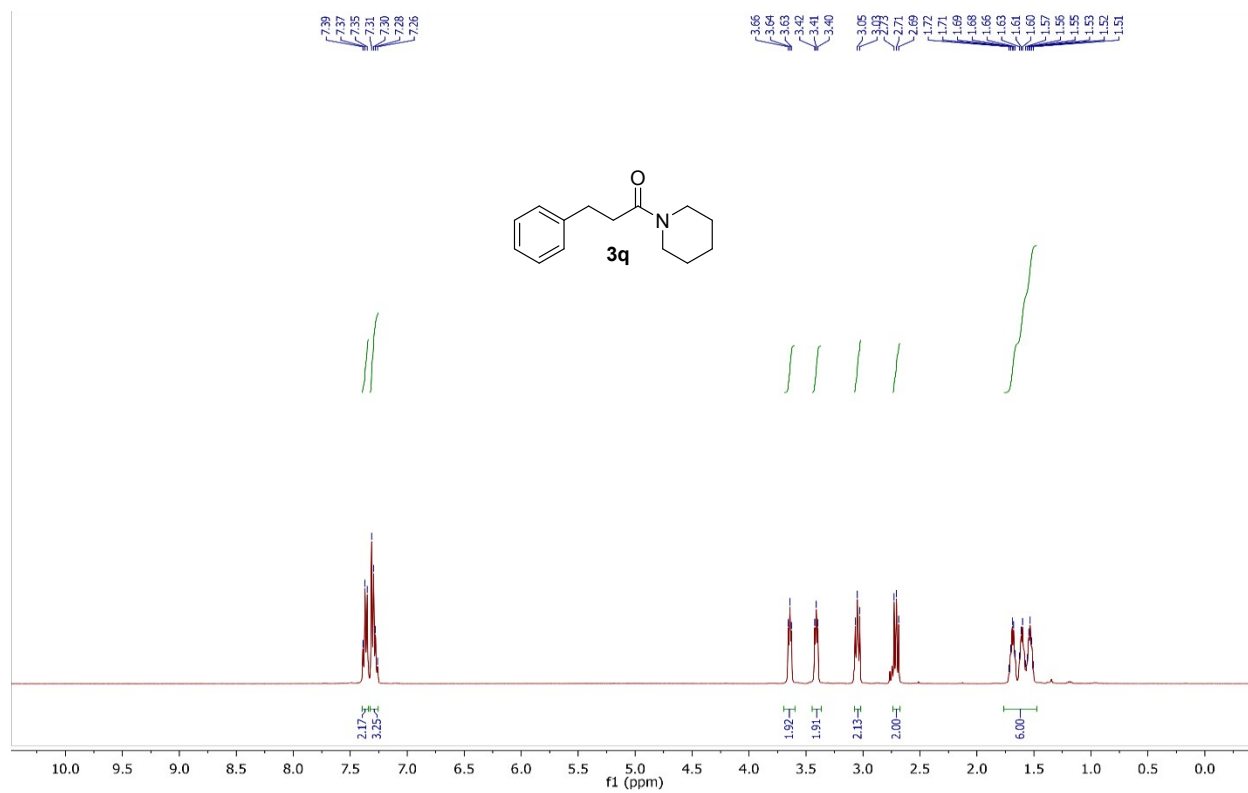
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **3o**



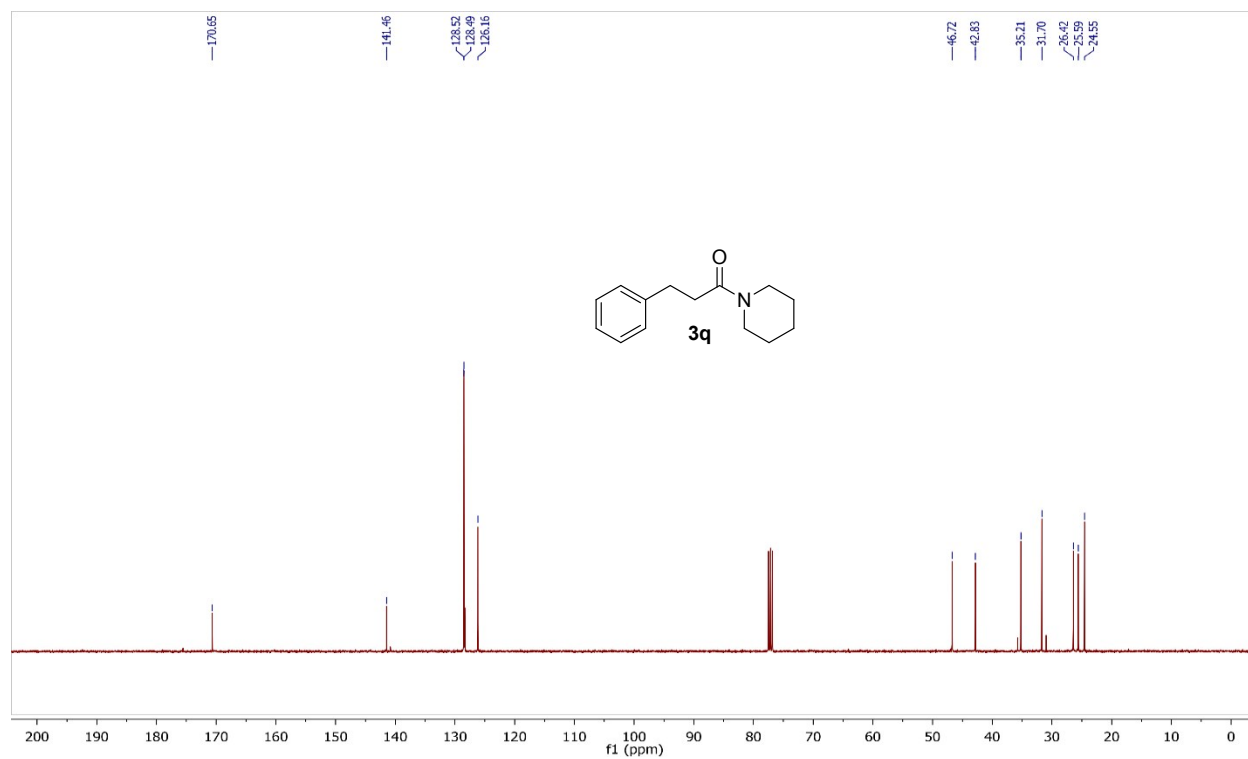
$^1\text{H}$  NMR of compound **3p**



$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **3p**

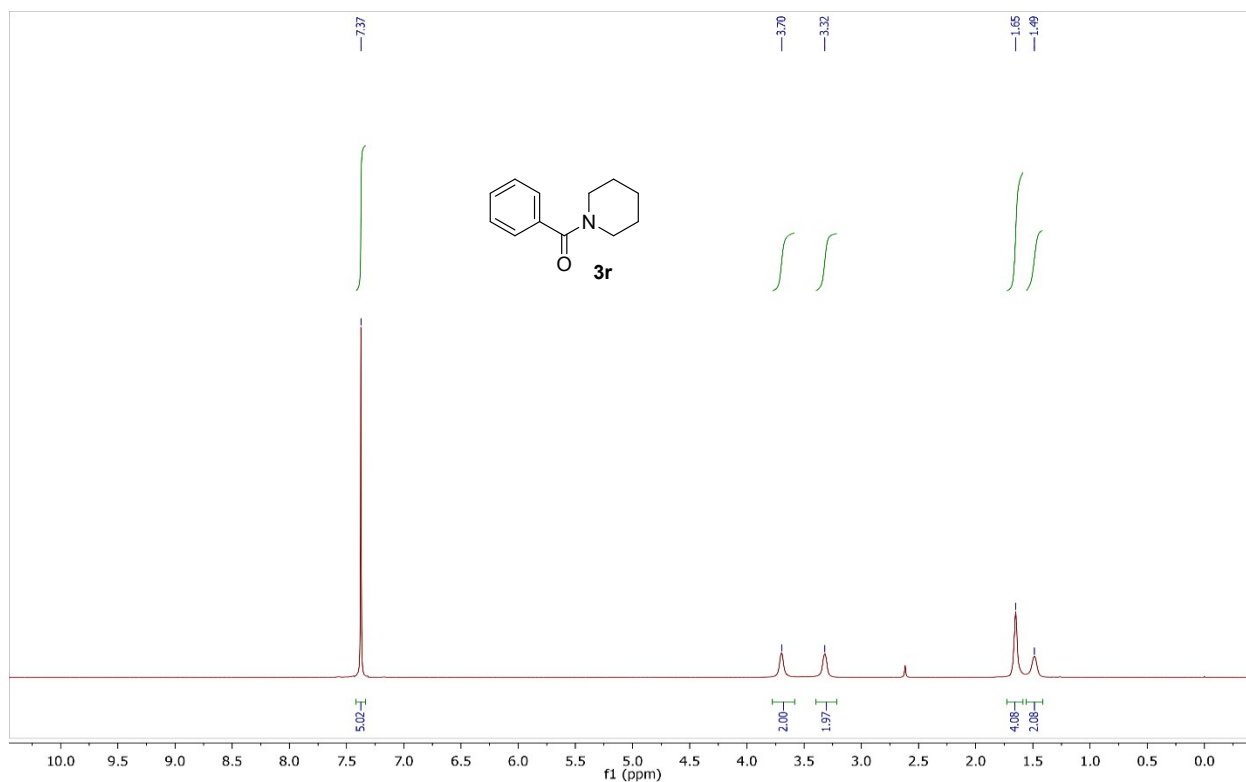


$^1\text{H}$  NMR of compound **3q**

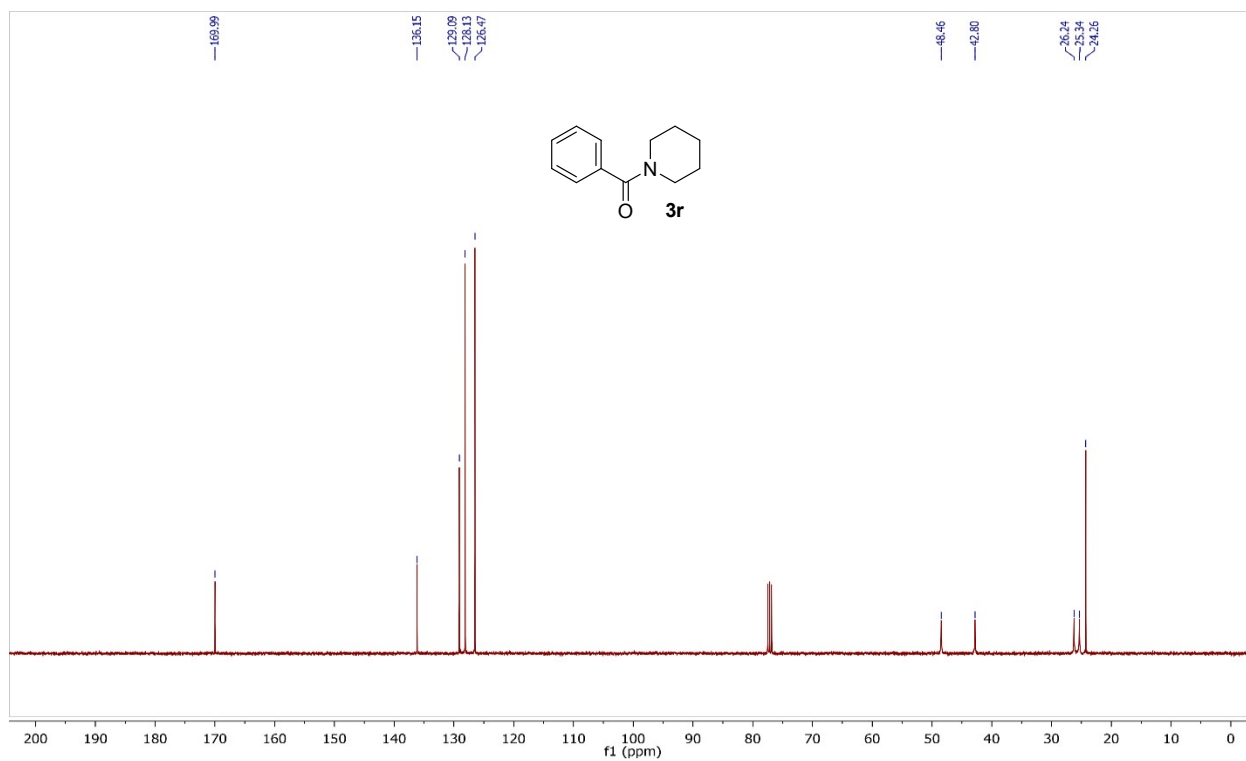


$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **3q**

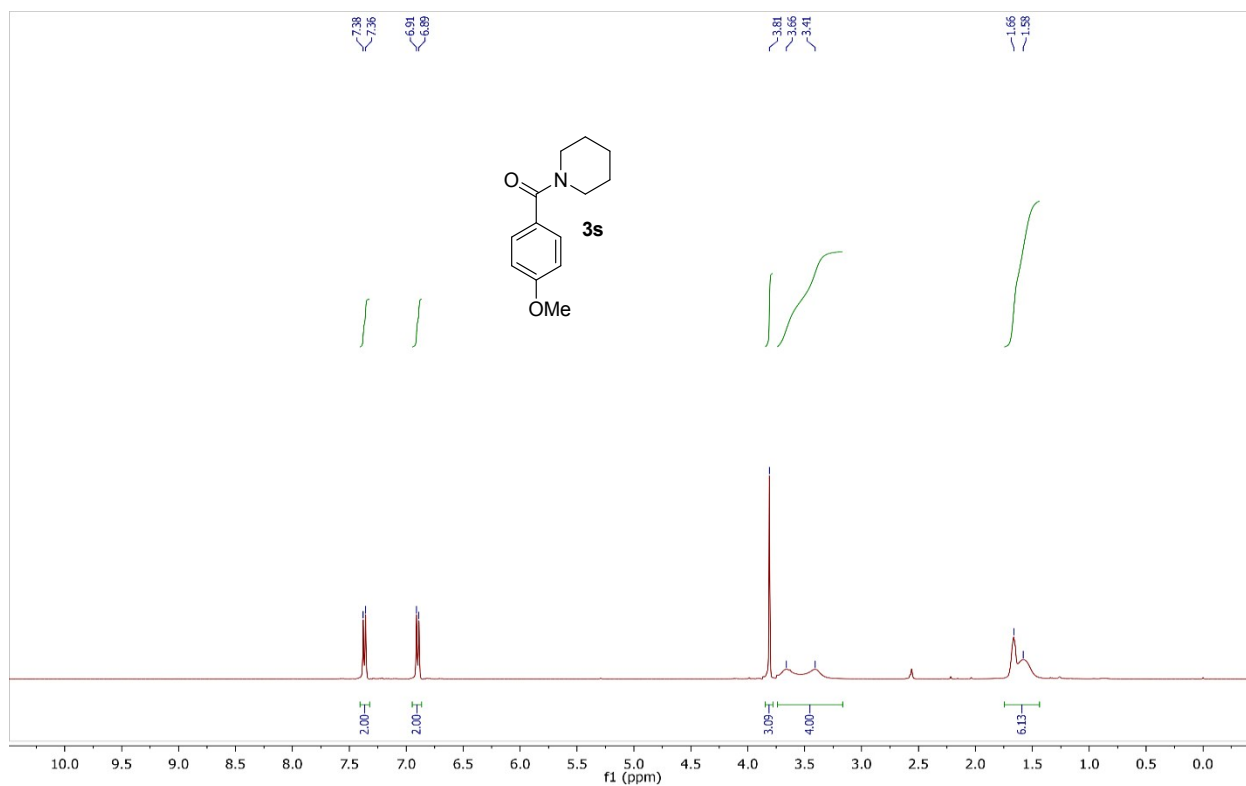




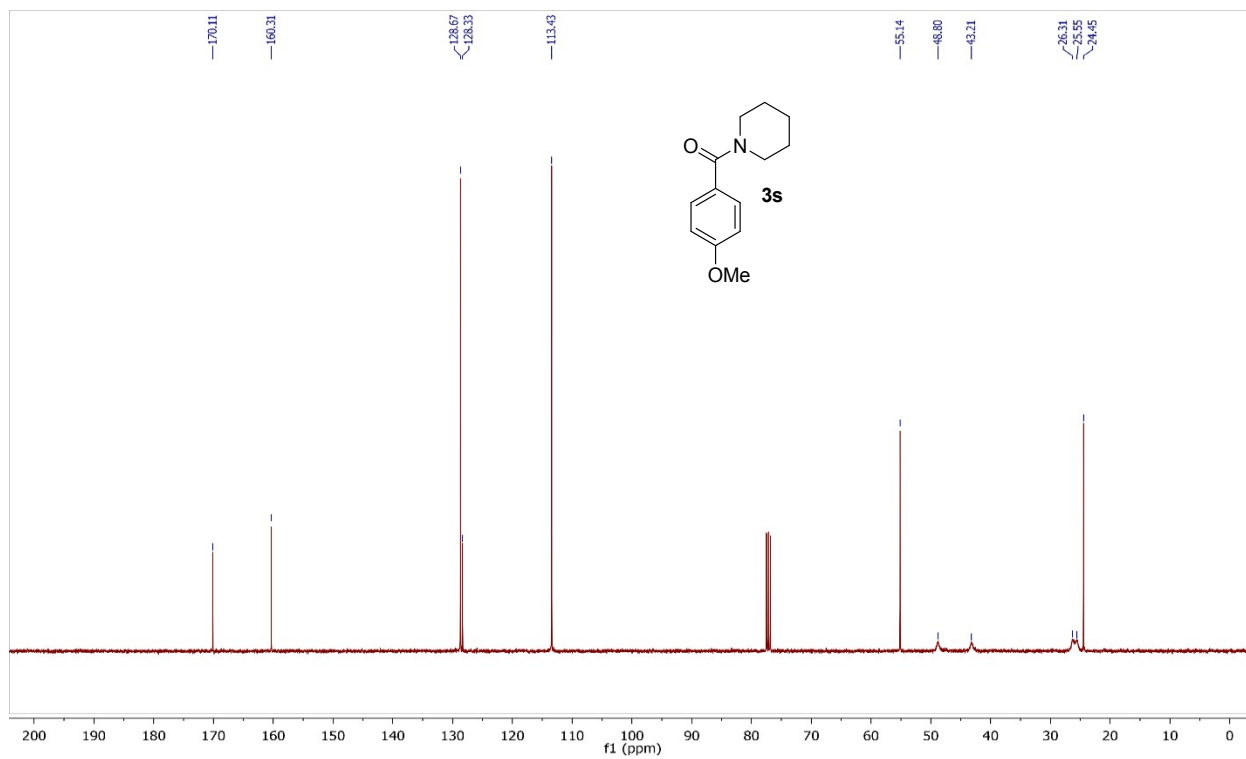
$^1\text{H}$  NMR of compound **3r**



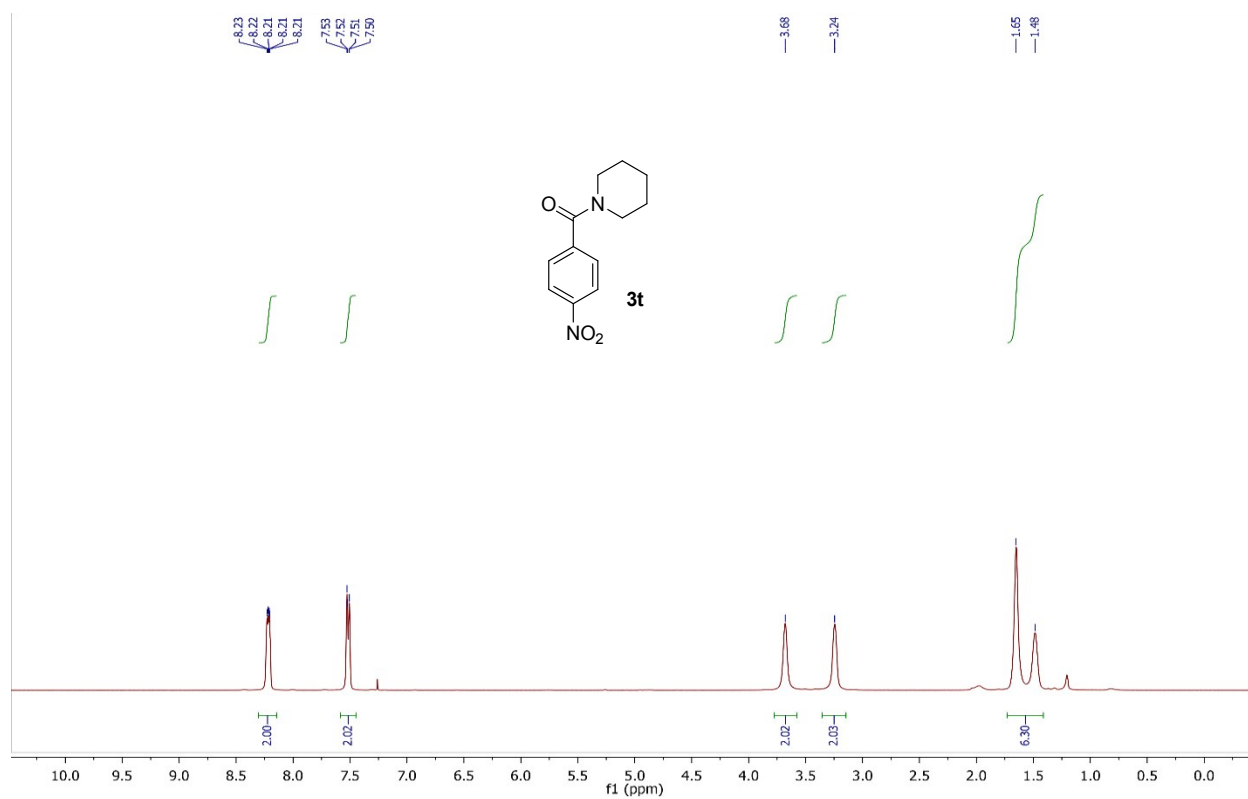
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **3r**



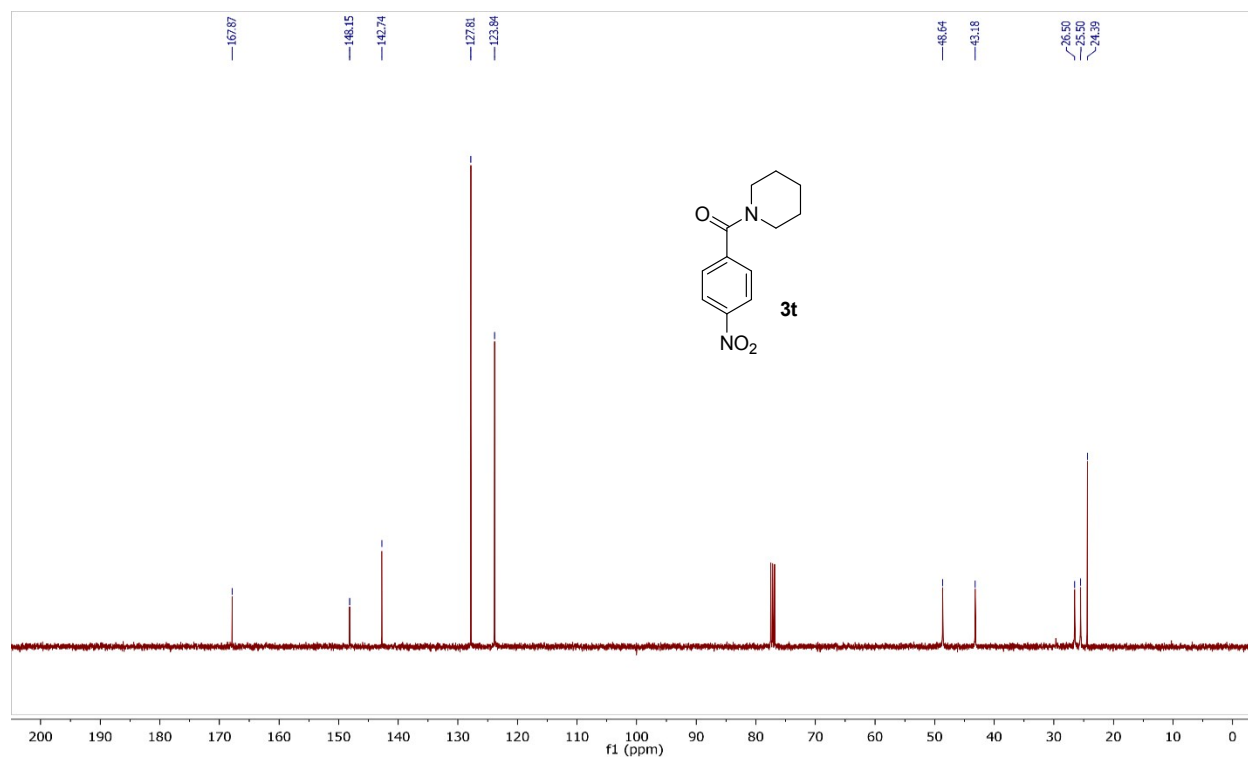
$^1\text{H NMR}$  of compound **3s**



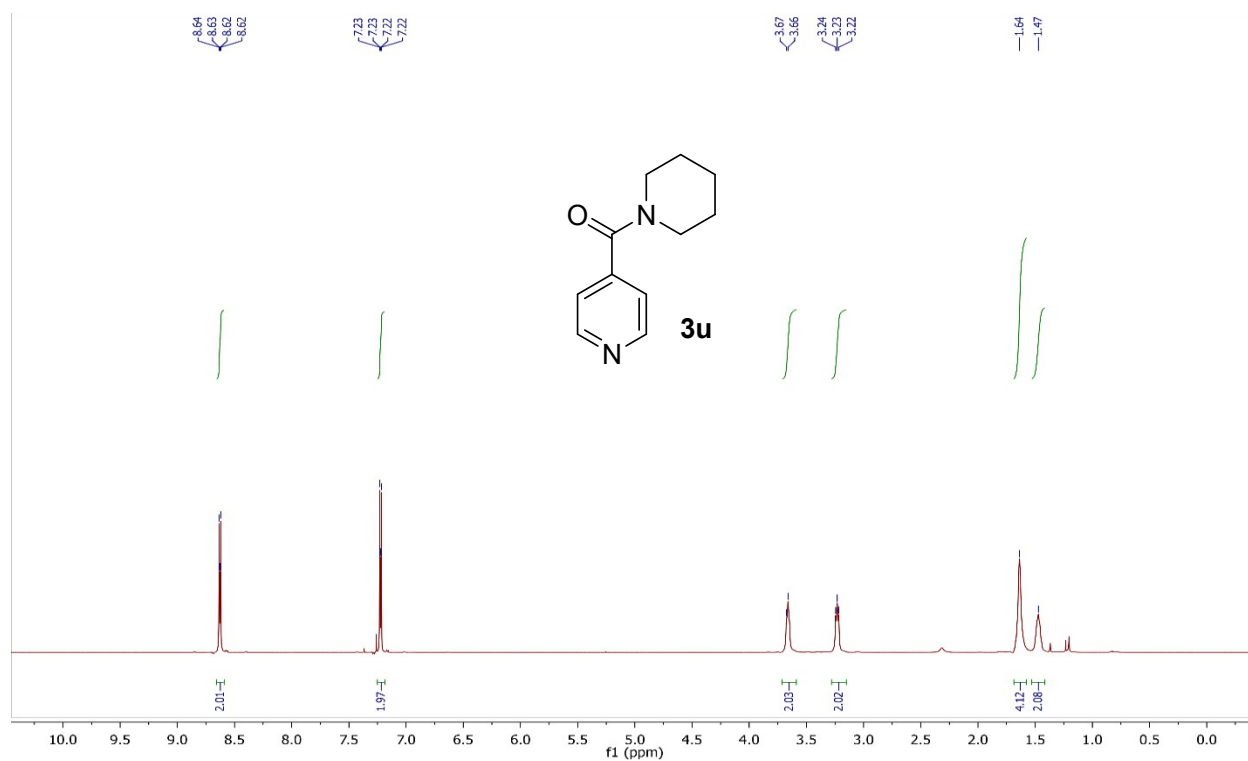
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **3s**



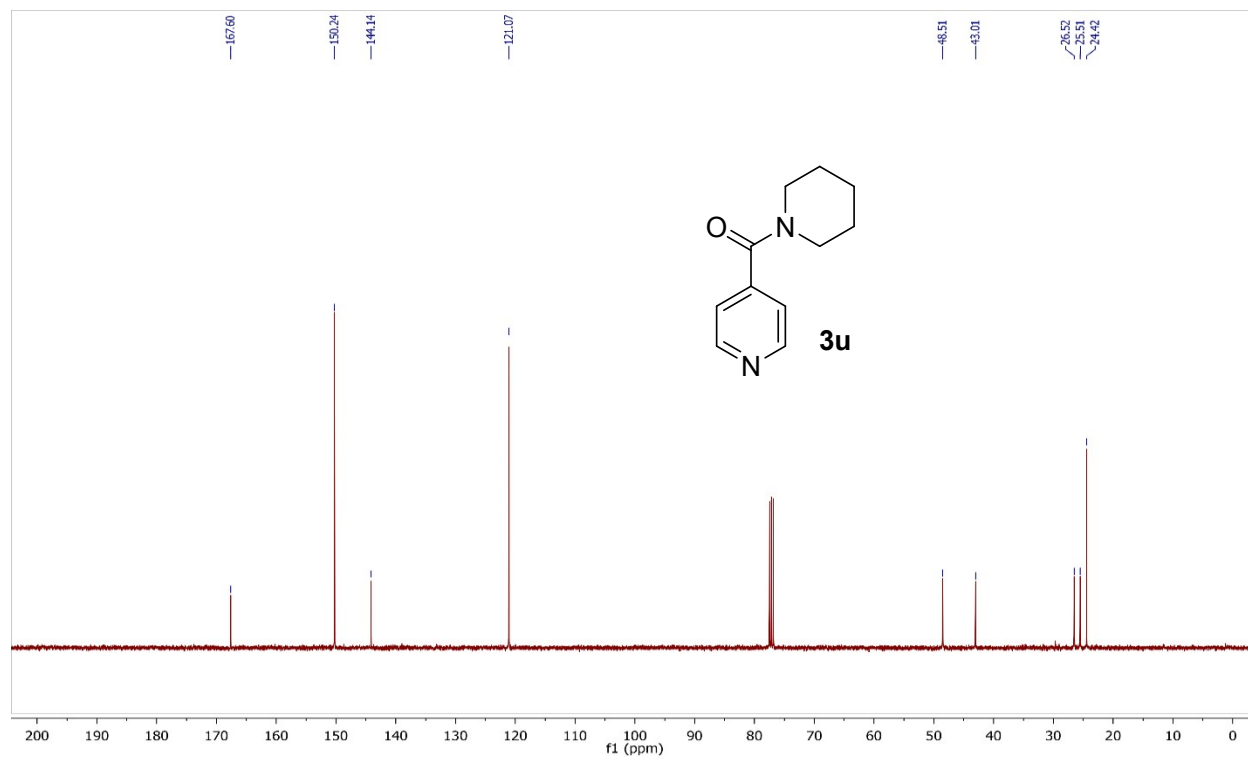
<sup>1</sup>H NMR of compound **3t**



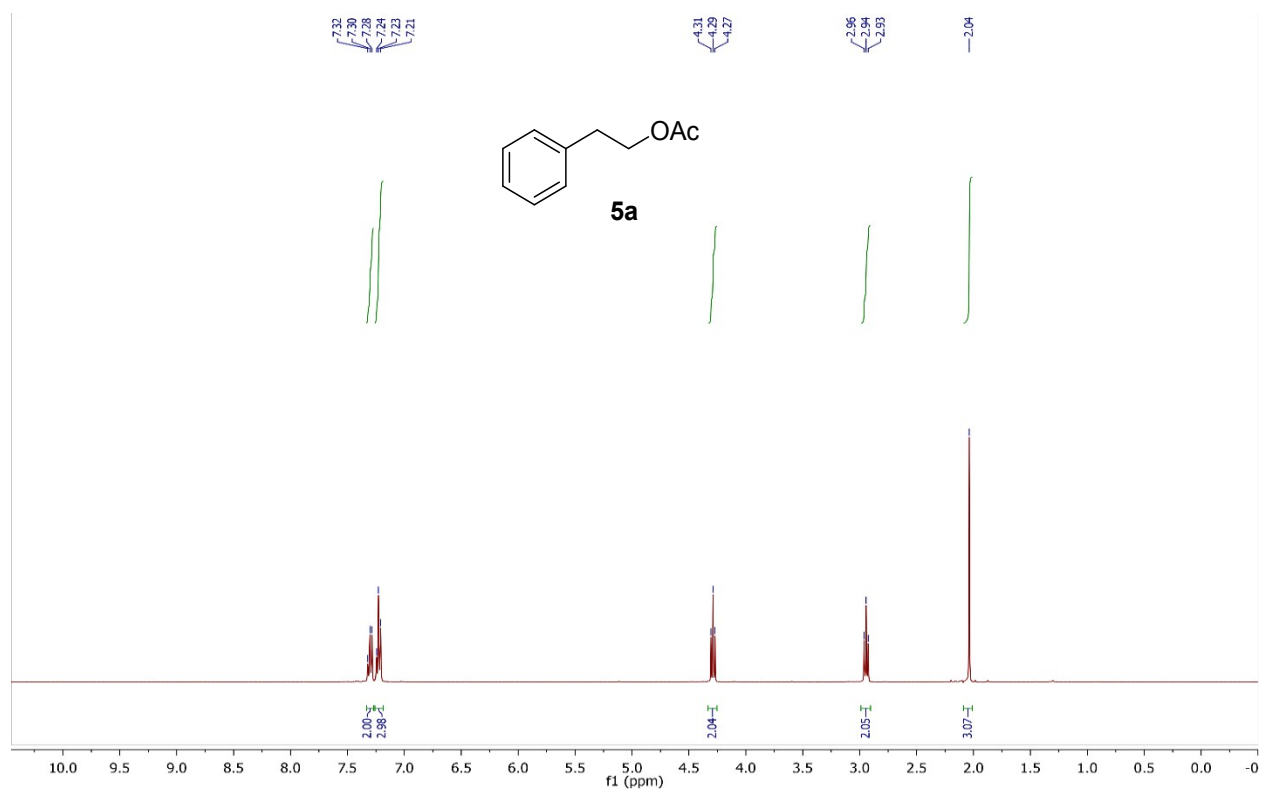
<sup>13</sup>C{<sup>1</sup>H} NMR of compound **3t**



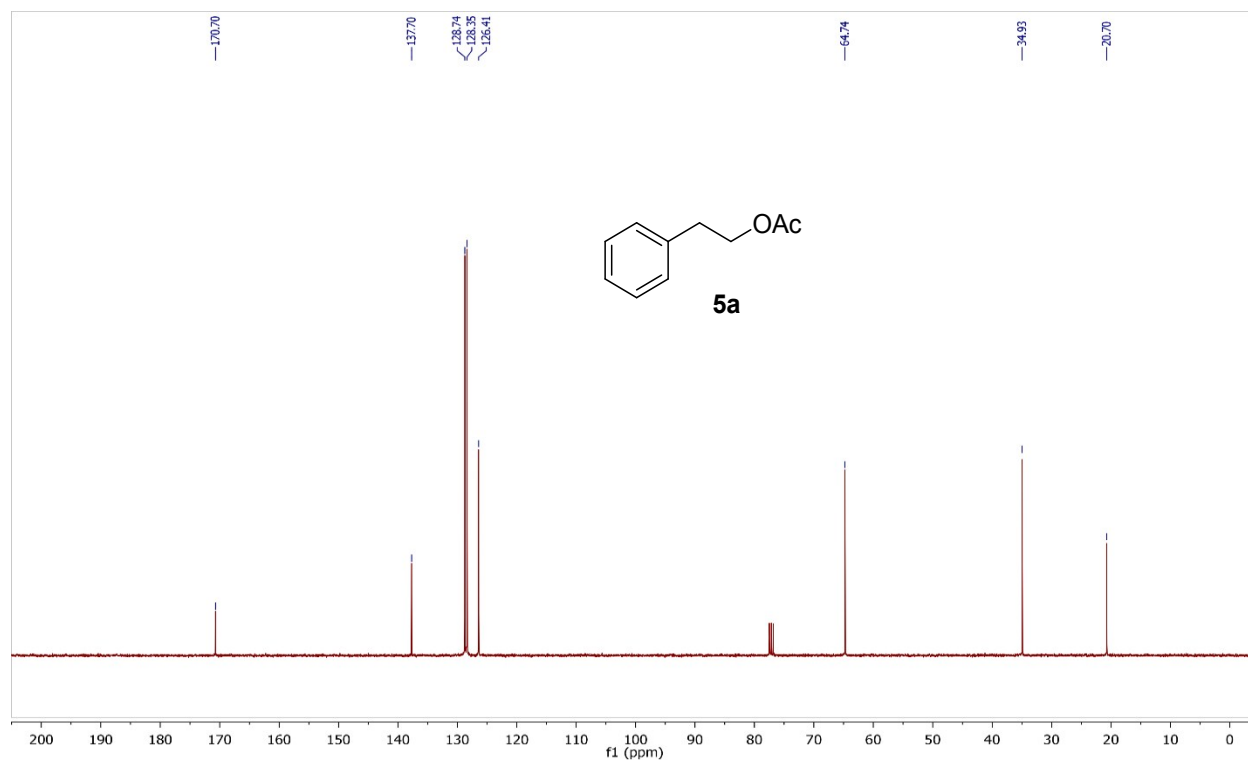
$^1\text{H}$  NMR of compound **3u**



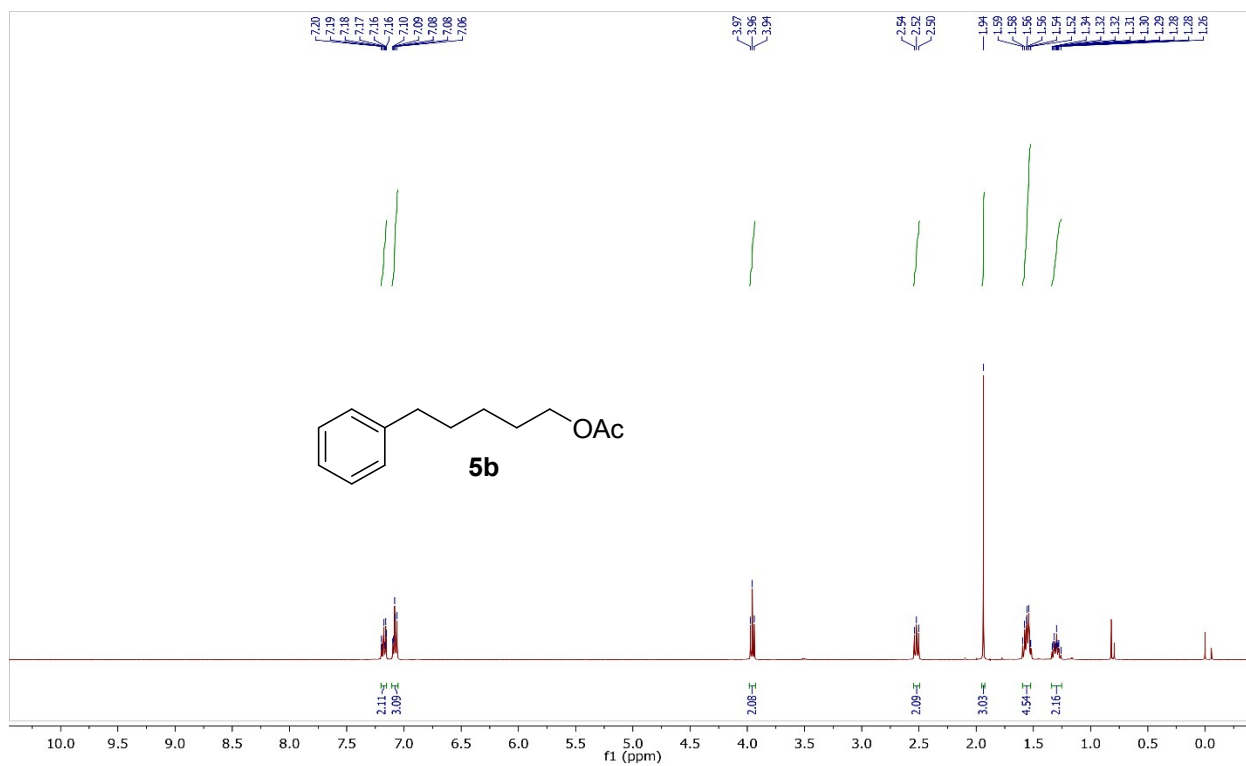
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **3u**



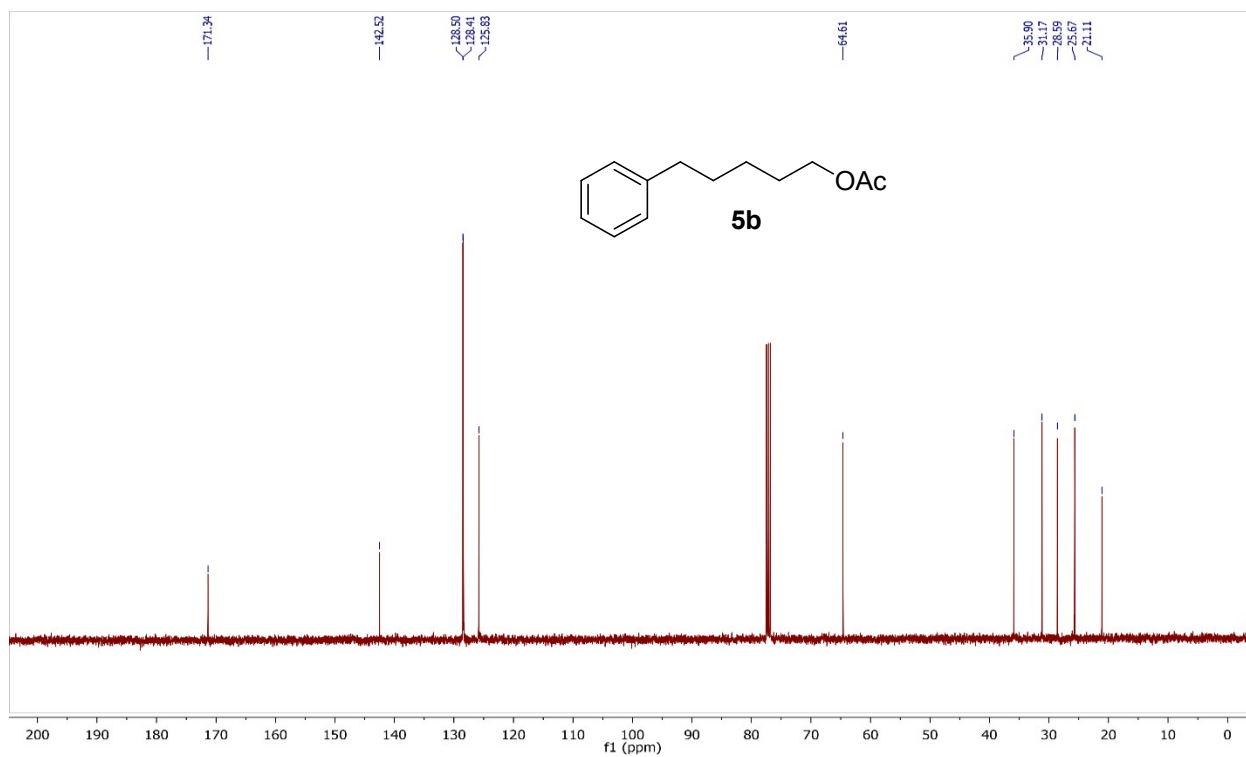
$^1\text{H}$  NMR of compound **5a**



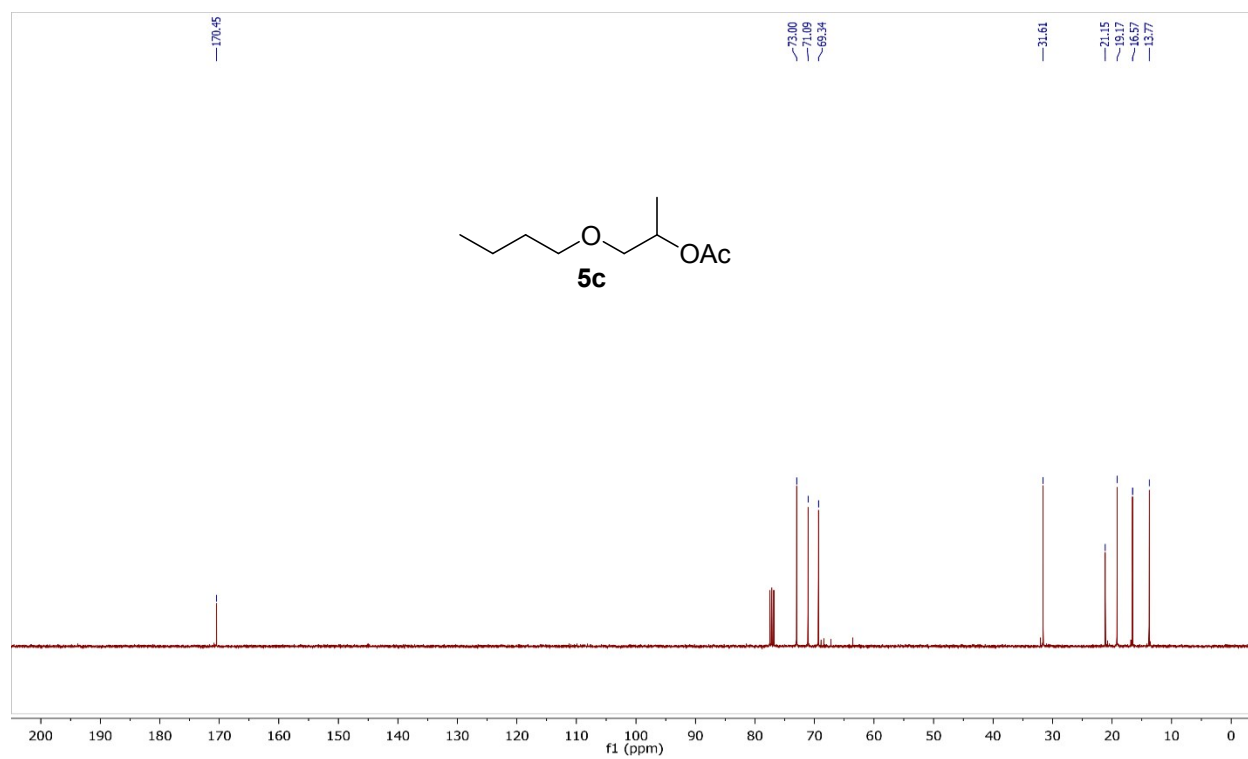
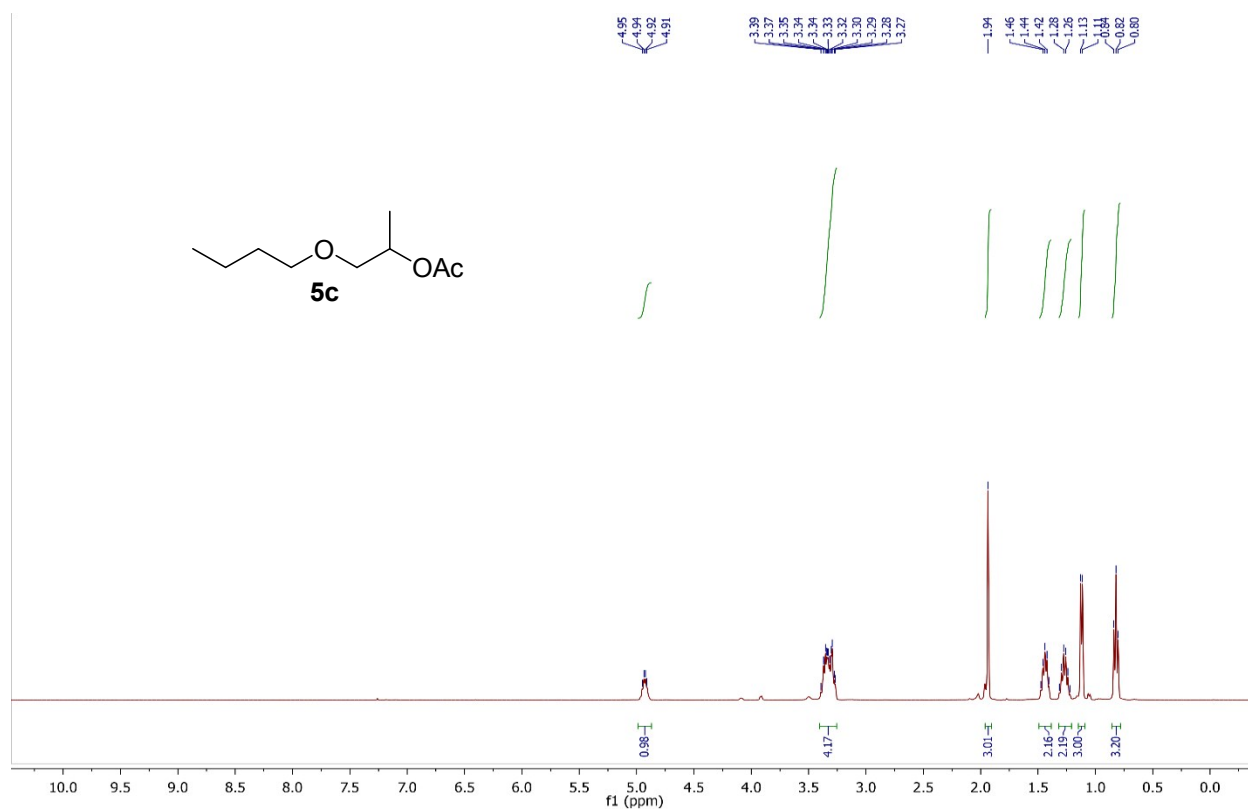
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5a**

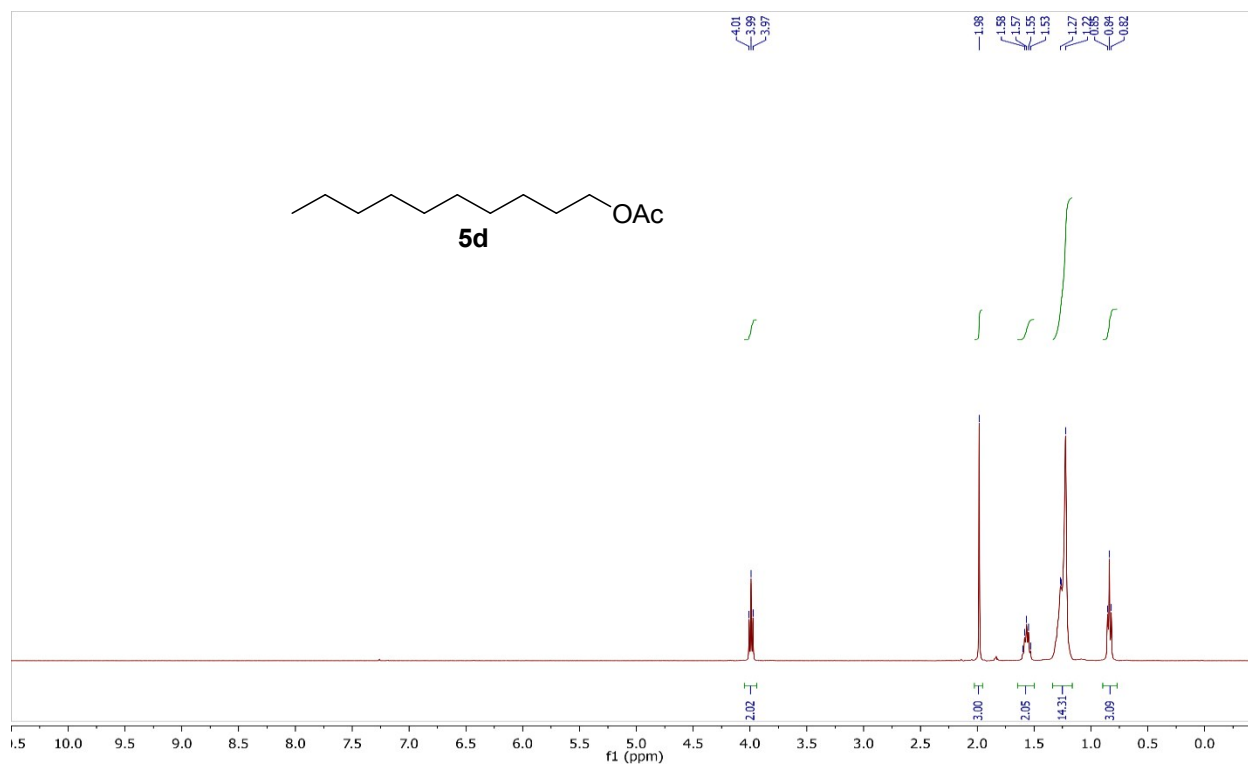


$^1\text{H}$  NMR of compound **5b**

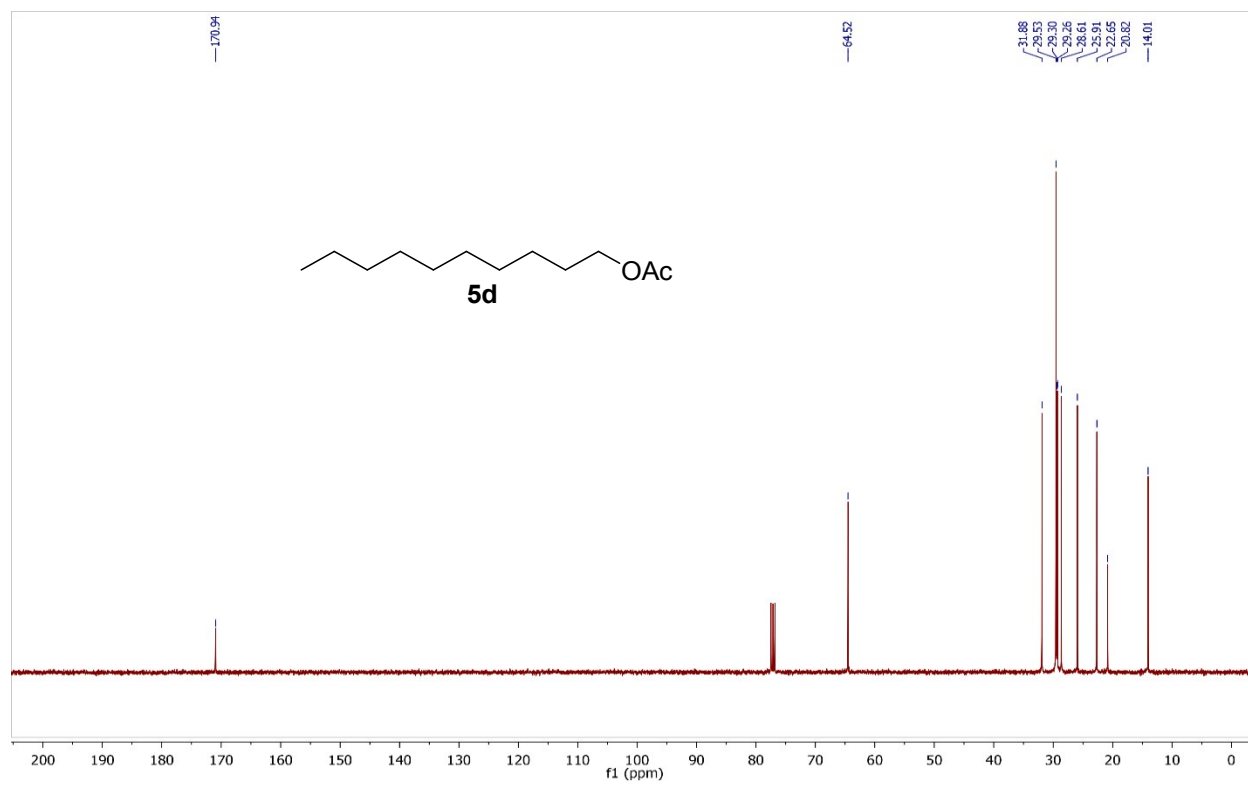


$^{13}\text{C}$   $\{^1\text{H}\}$  NMR of compound **5b**



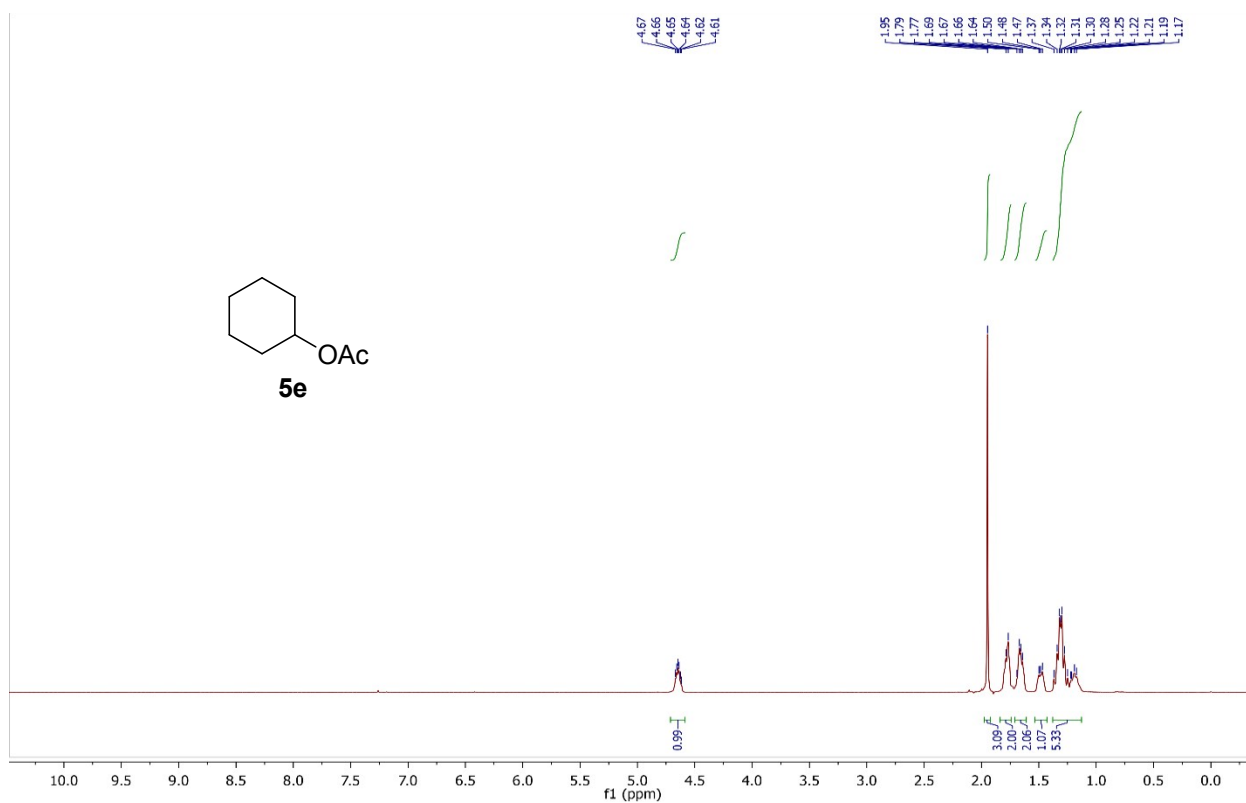


$^1\text{H}$  NMR of compound **5d**

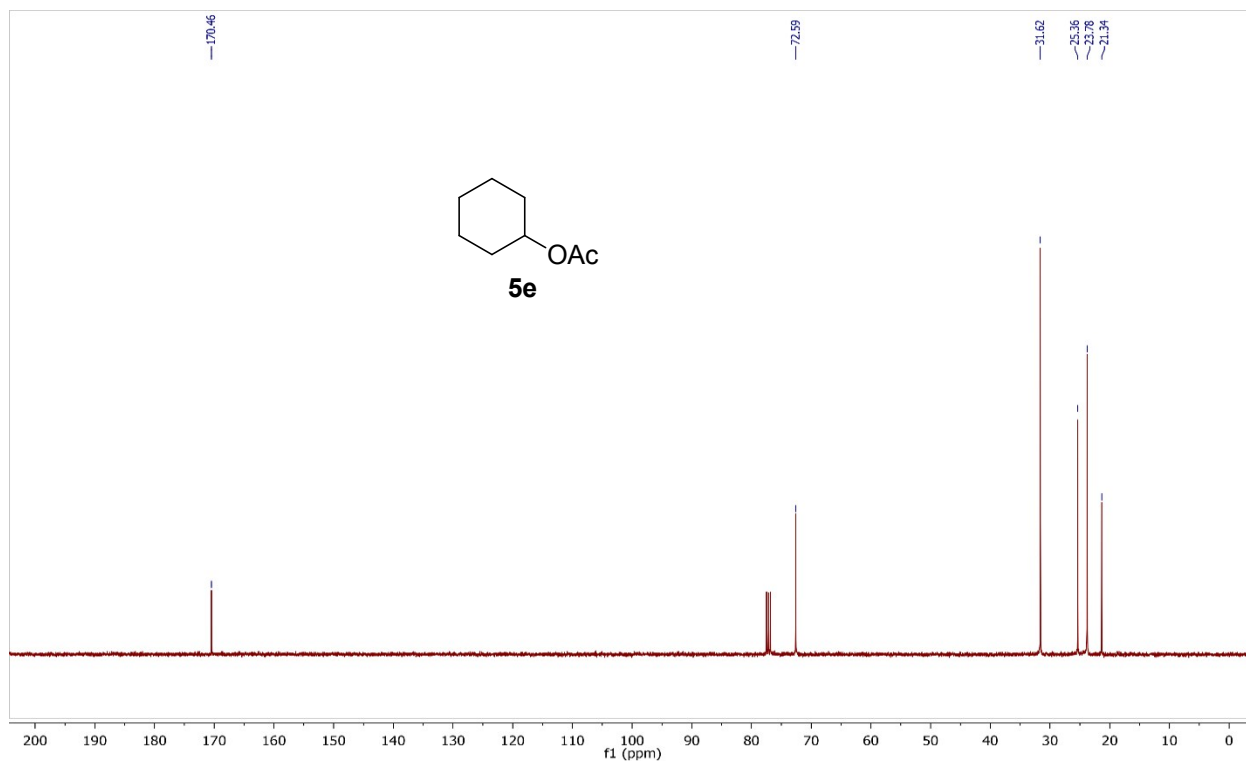


$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5d**

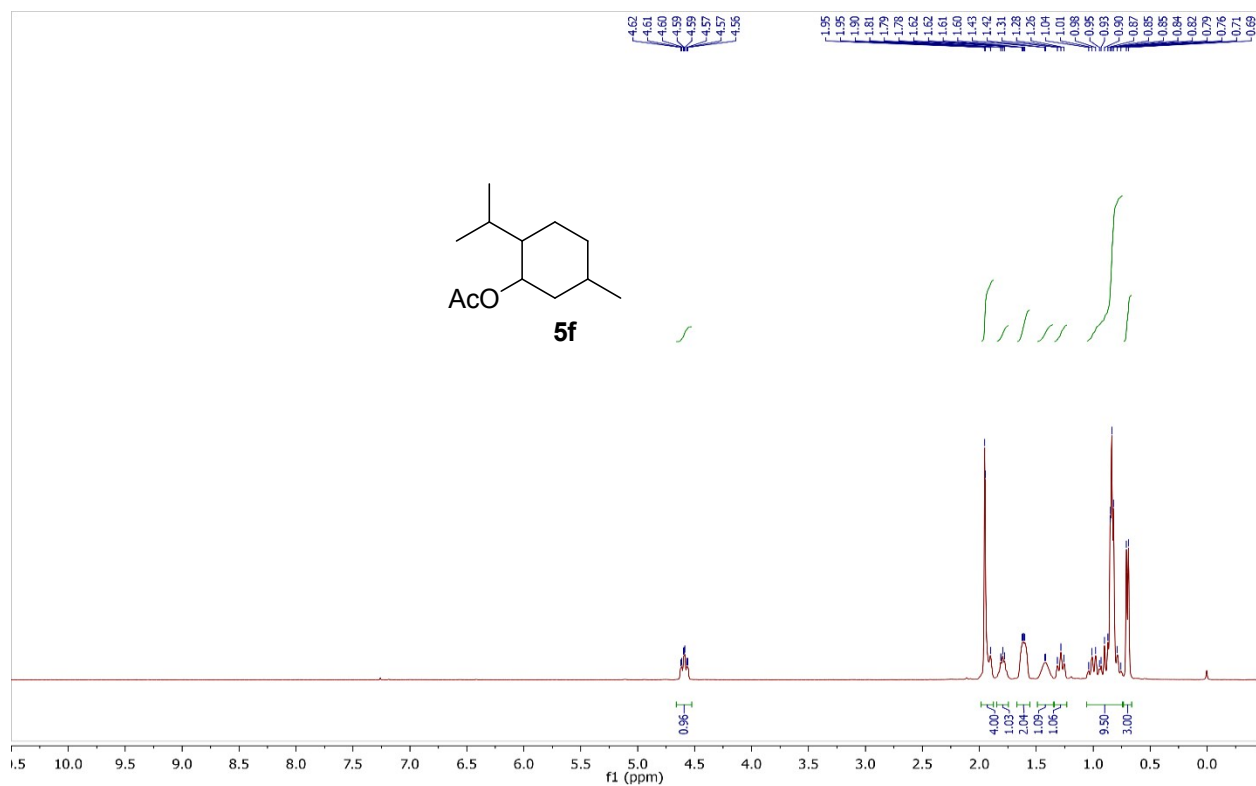




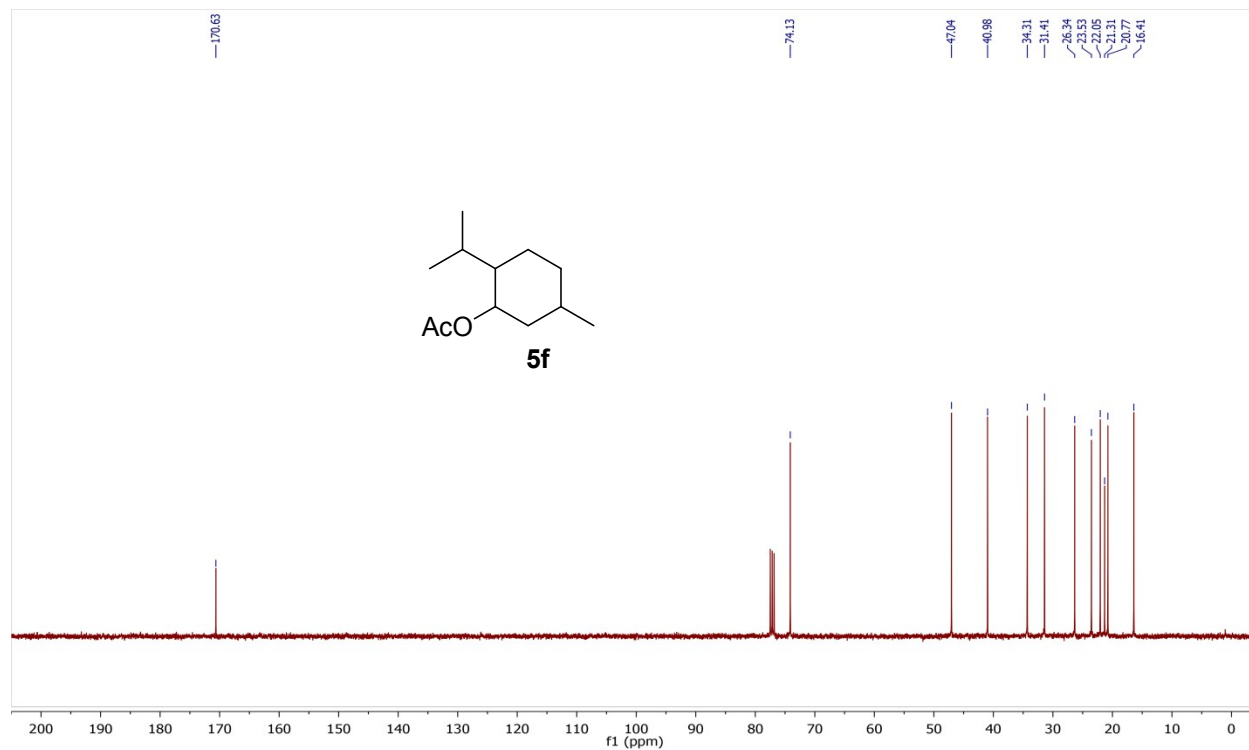
$^1\text{H}$  NMR of compound **5e**



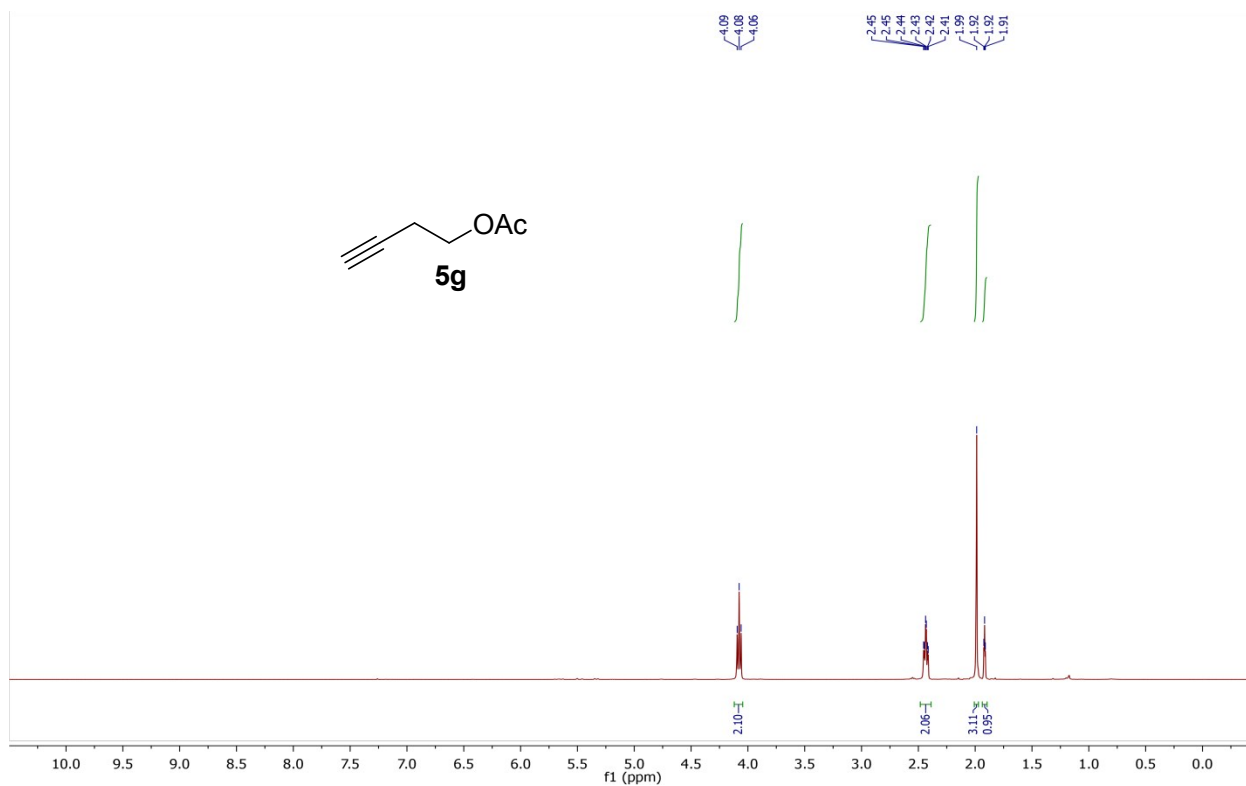
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5e**



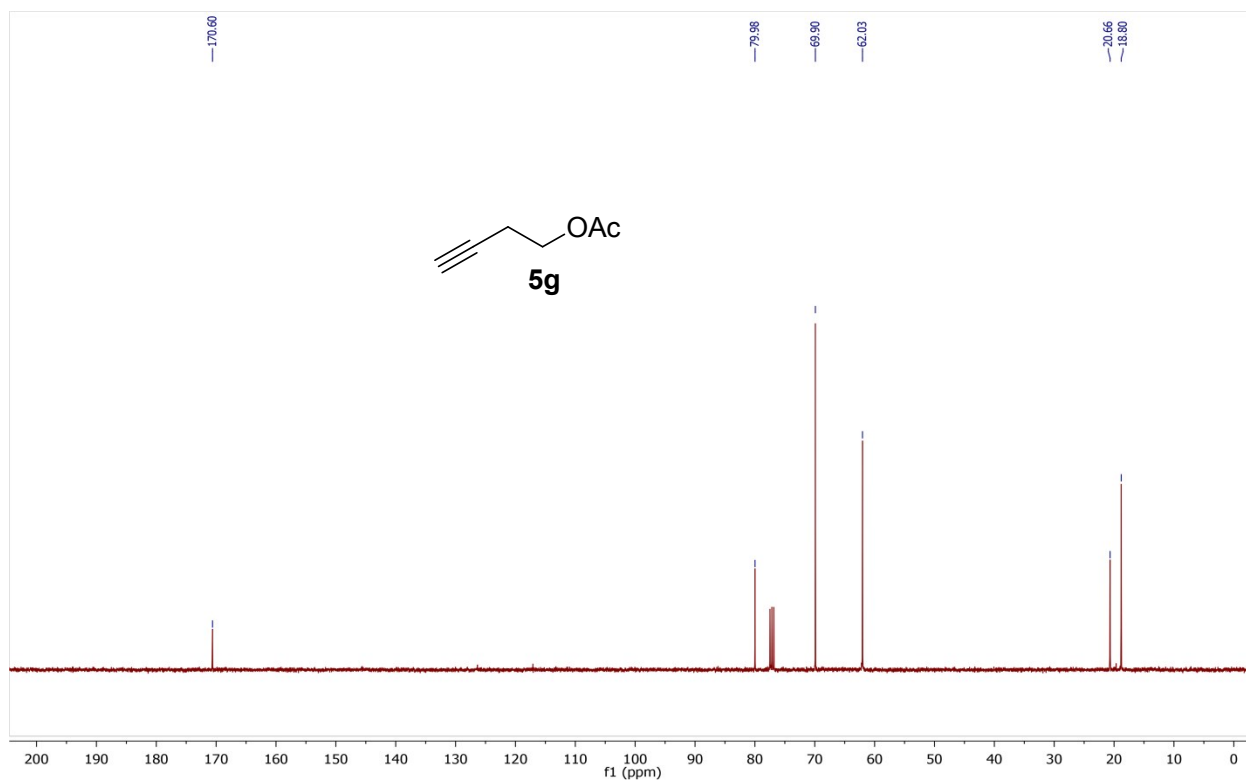
$^1\text{H NMR}$  of compound **5f**



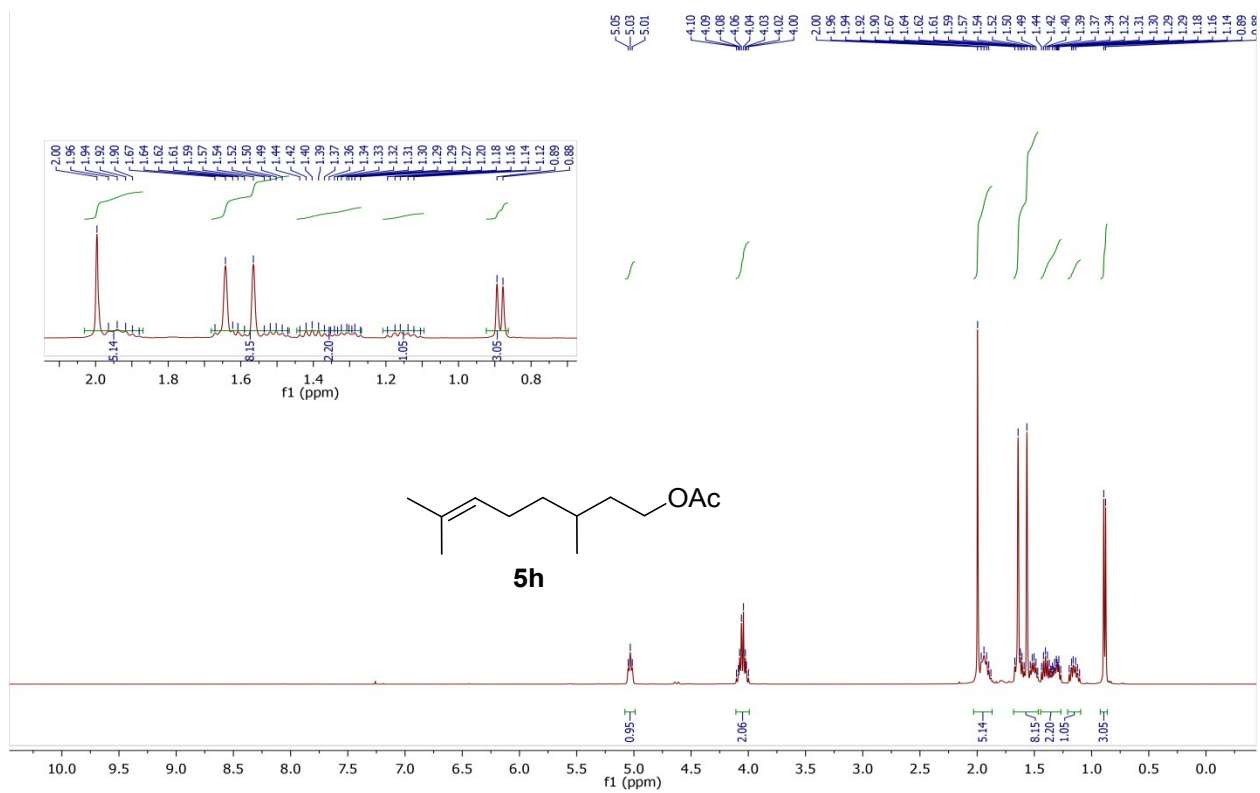
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5f**



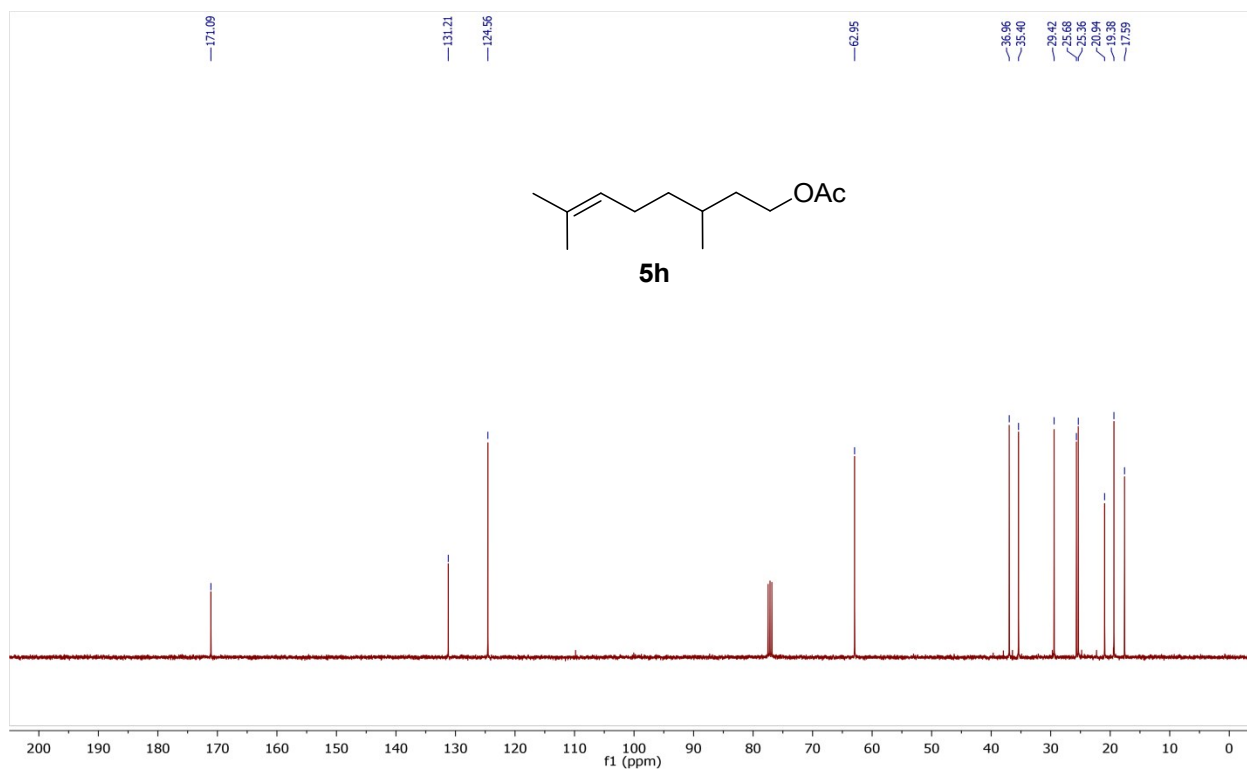
<sup>1</sup>H NMR of compound **5g**



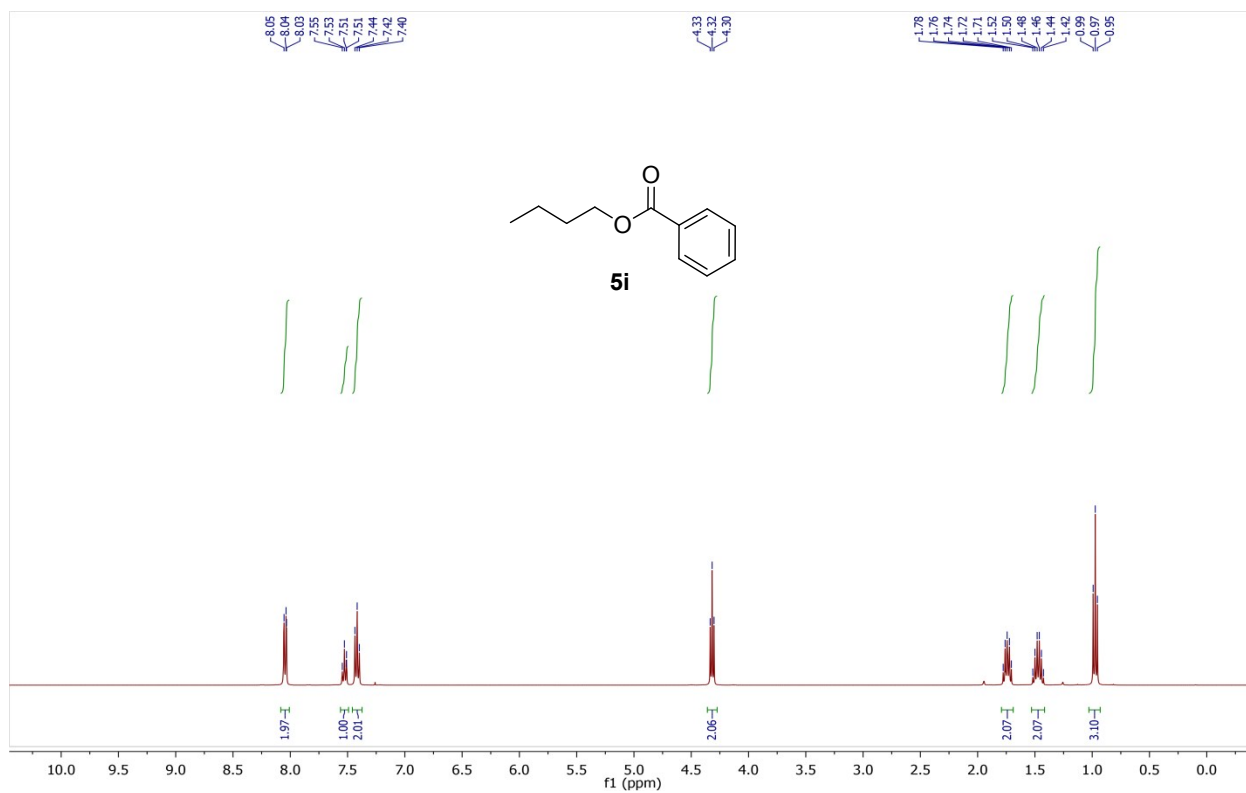
<sup>13</sup>C{<sup>1</sup>H} NMR of compound **5g**



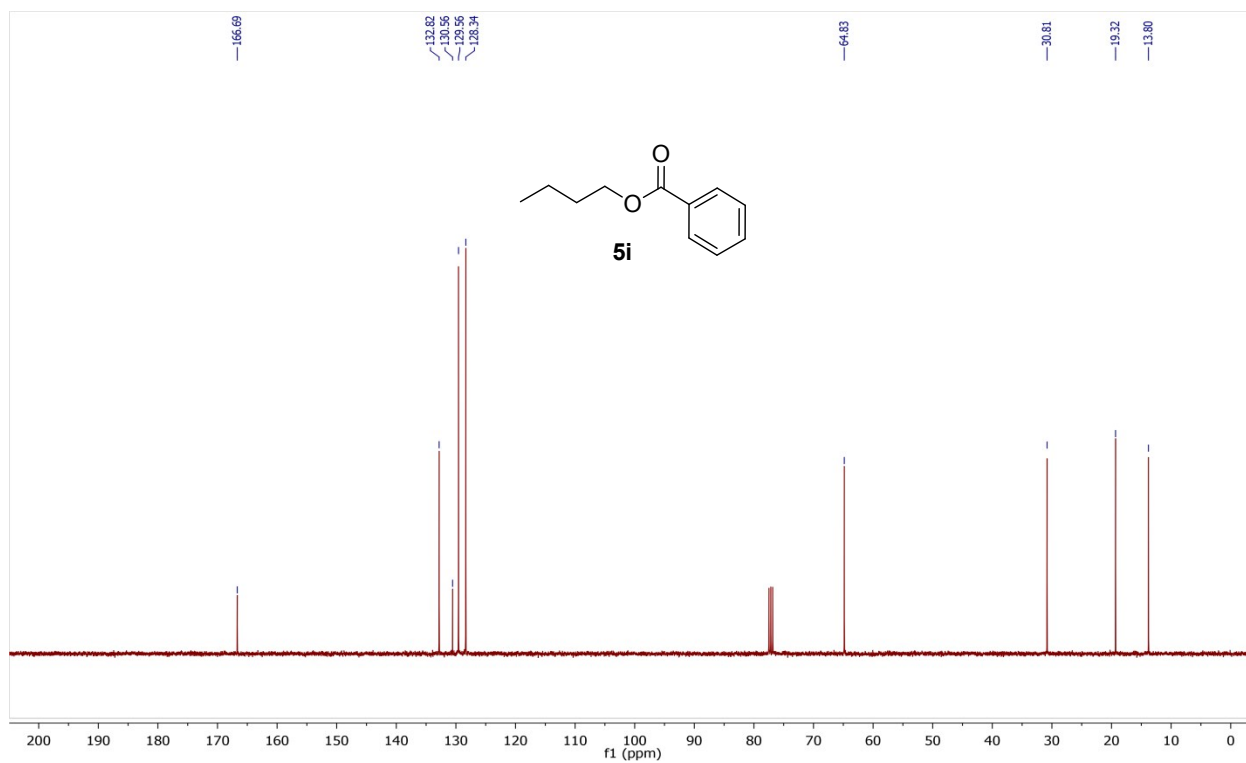
$^1\text{H}$  NMR of compound **5h**



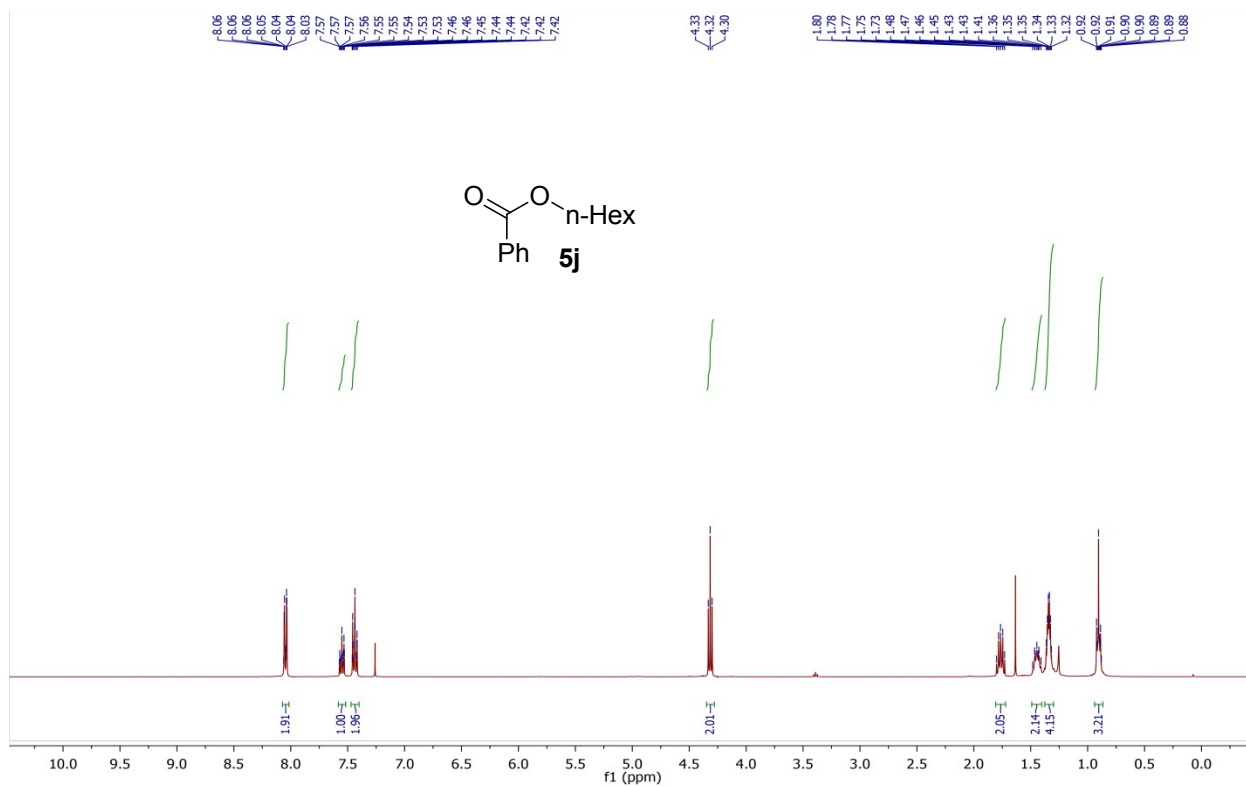
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5h**



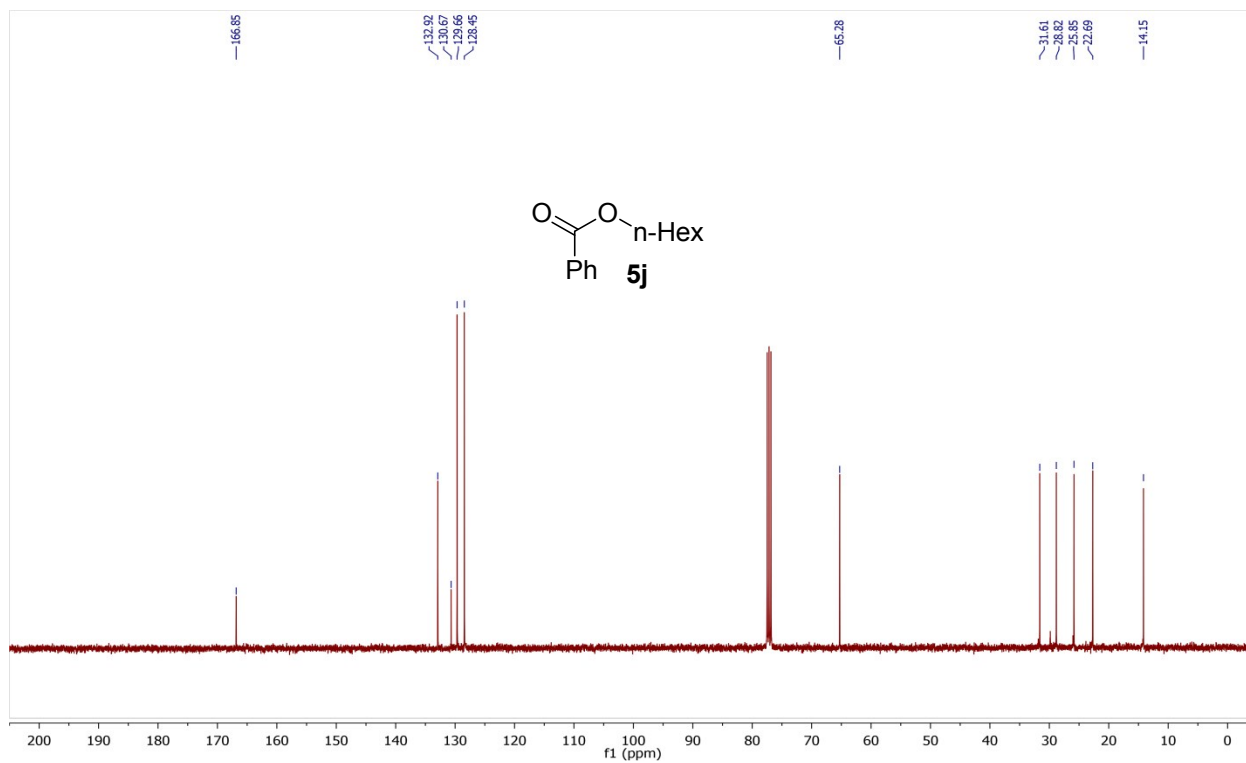
$^1\text{H}$  NMR of compound **5i**



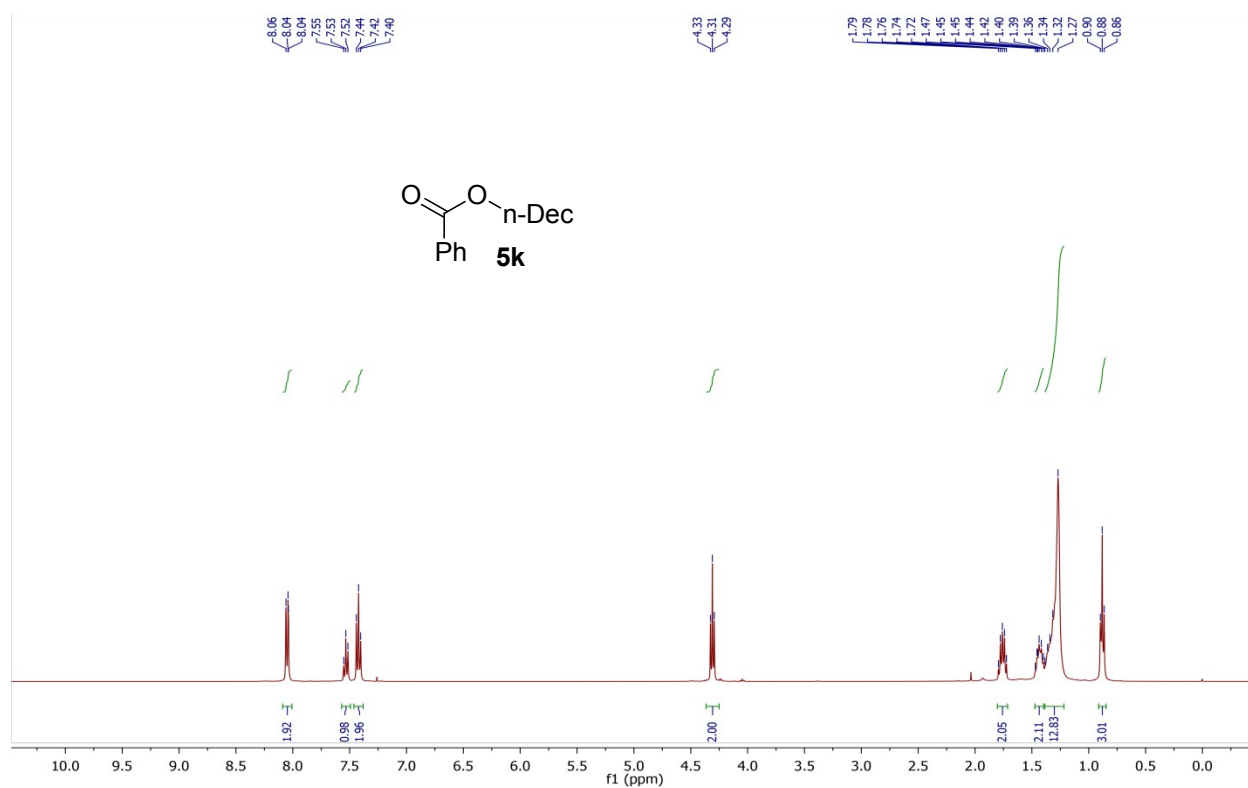
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5i**



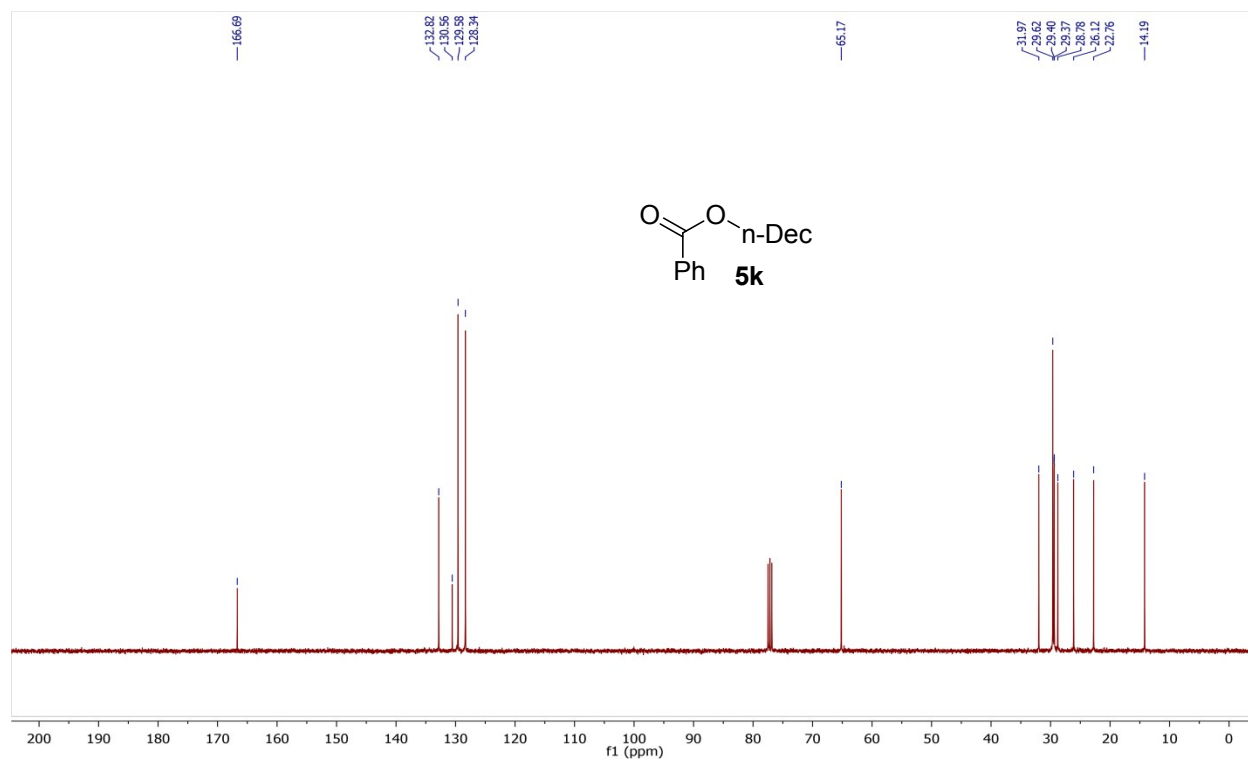
<sup>1</sup>H NMR of compound **5j**



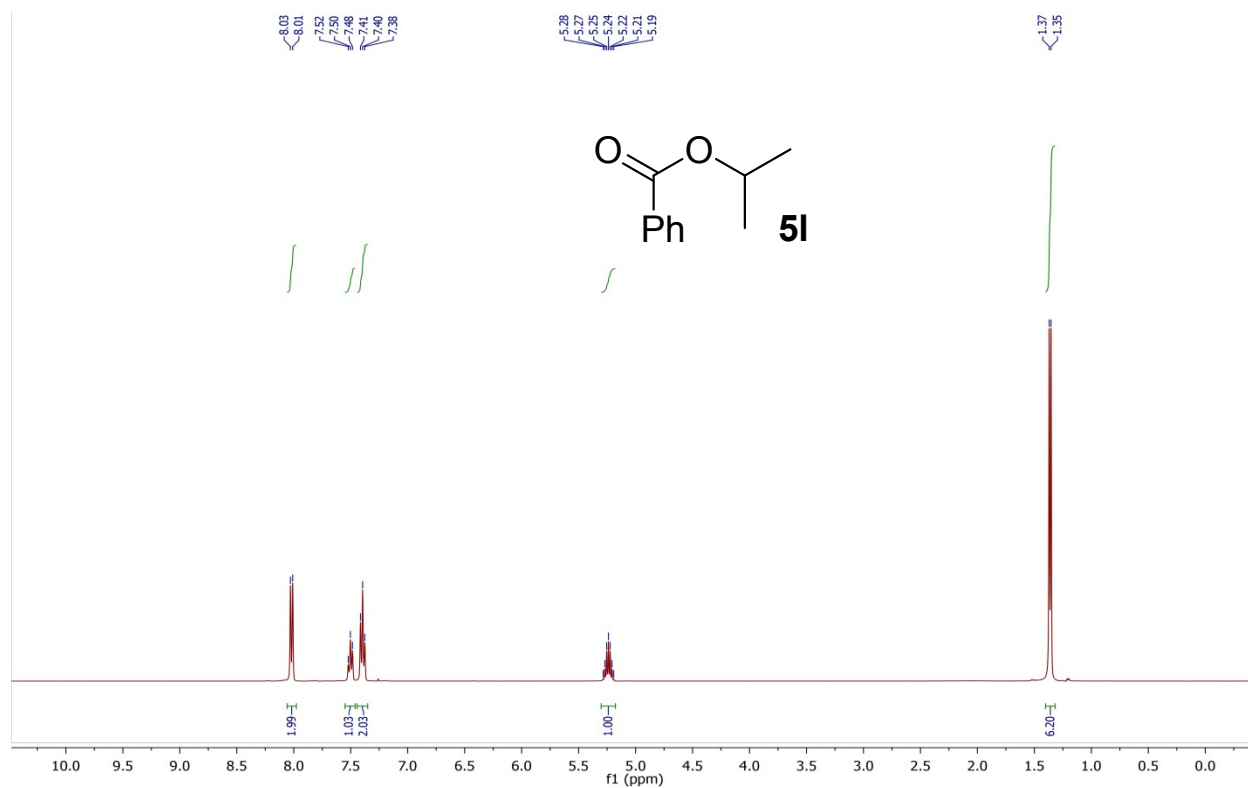
<sup>13</sup>C {<sup>1</sup>H} NMR of compound **5j**



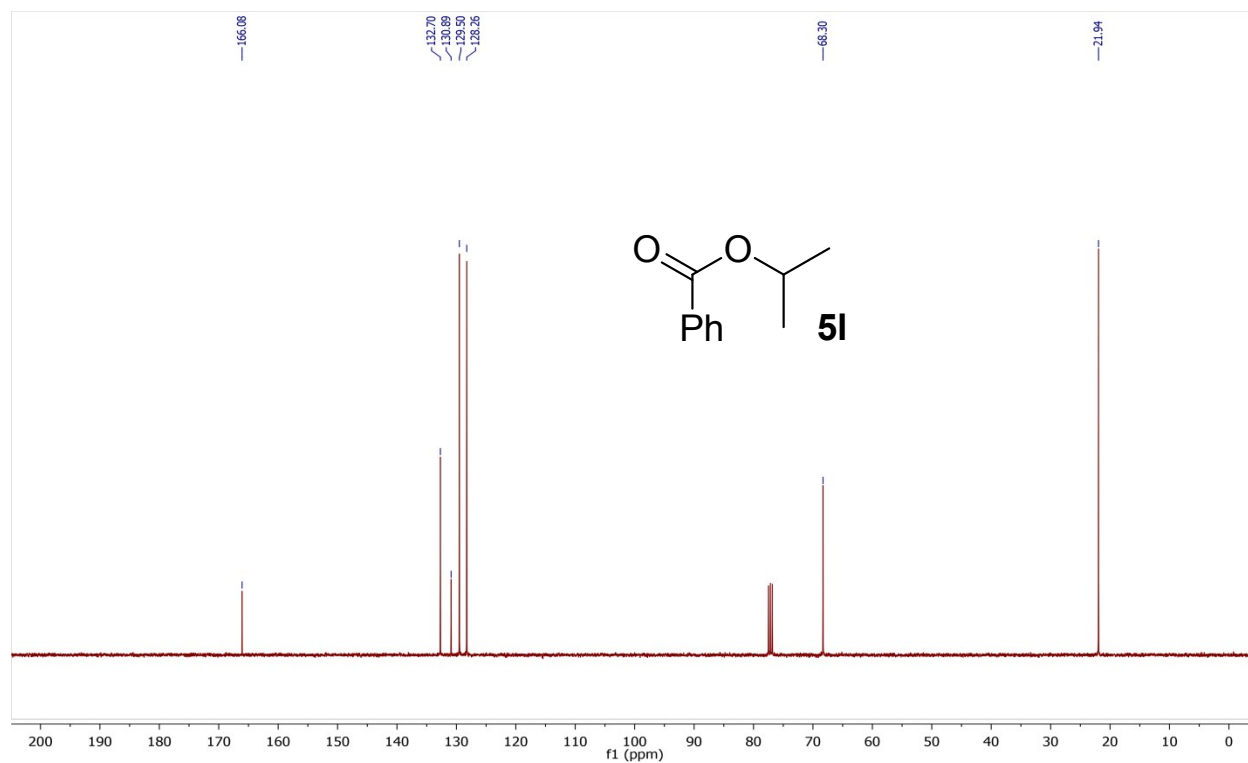
$^1\text{H}$  NMR of compound **5k**



$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5k**

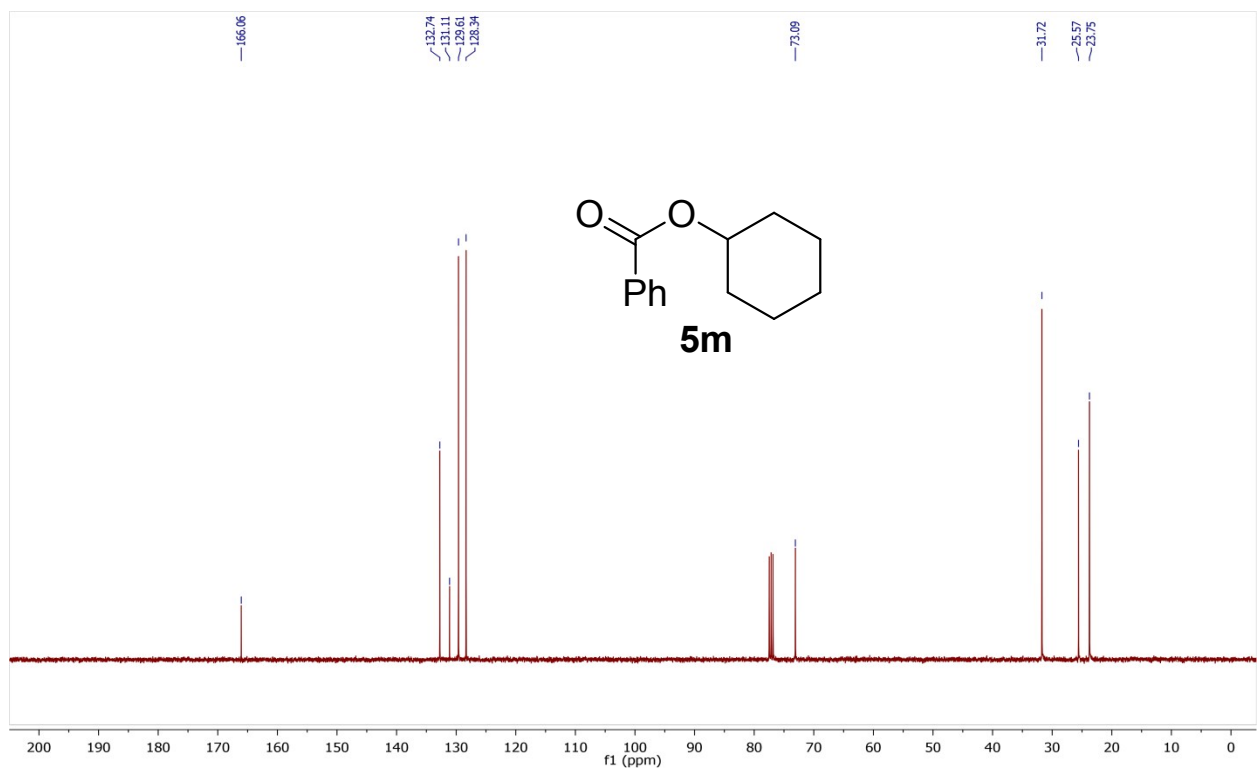
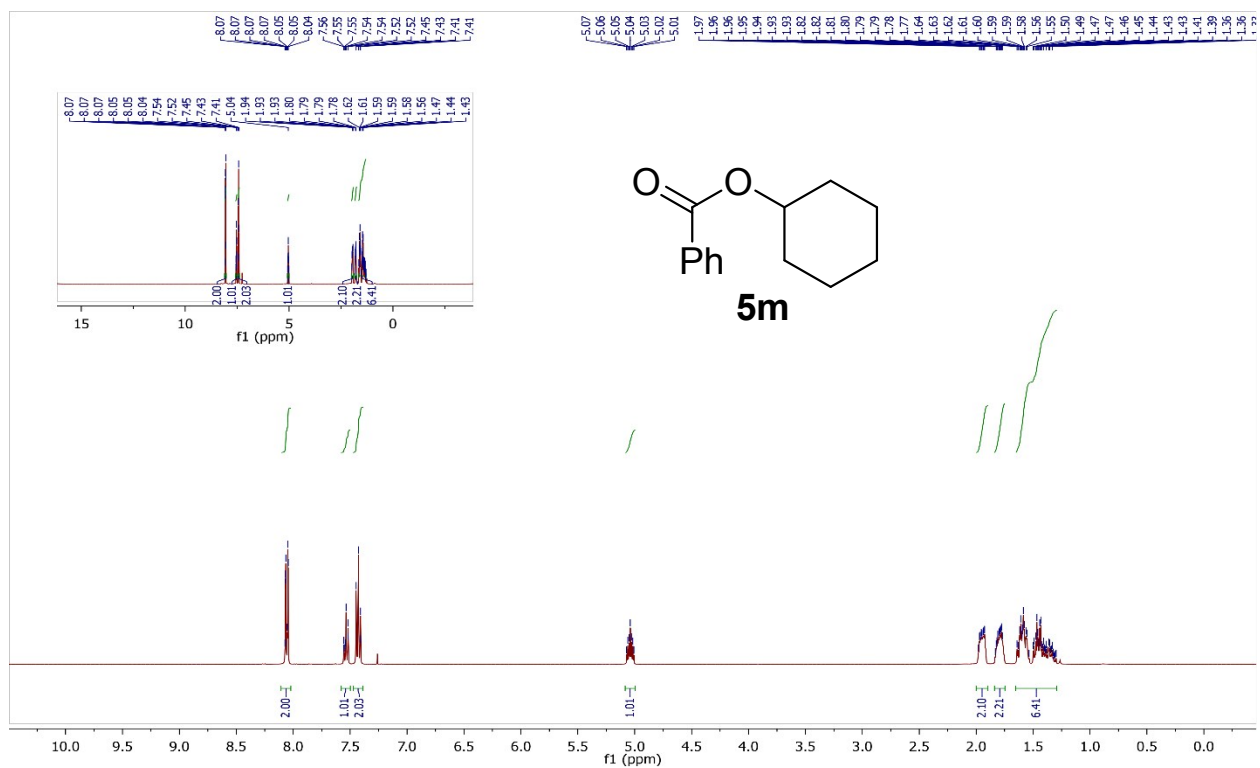


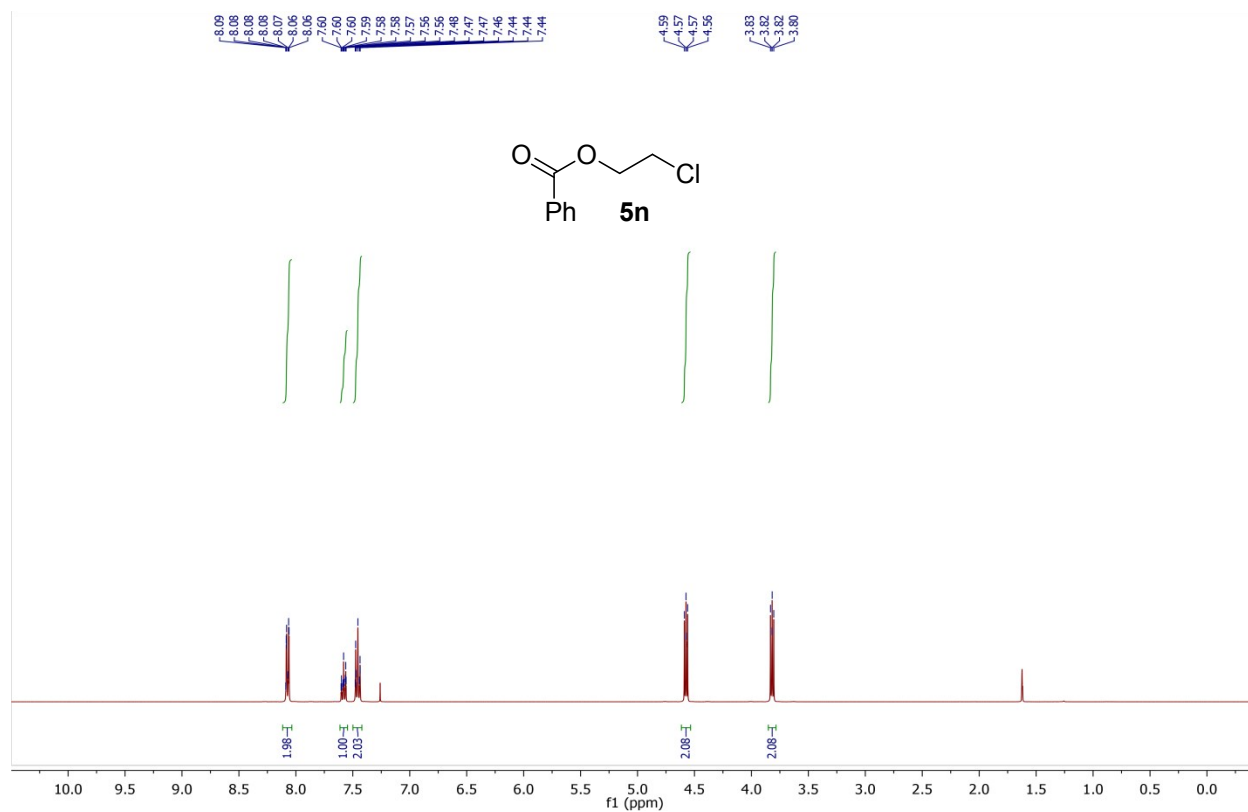
$^1\text{H NMR}$  of compound **5I**



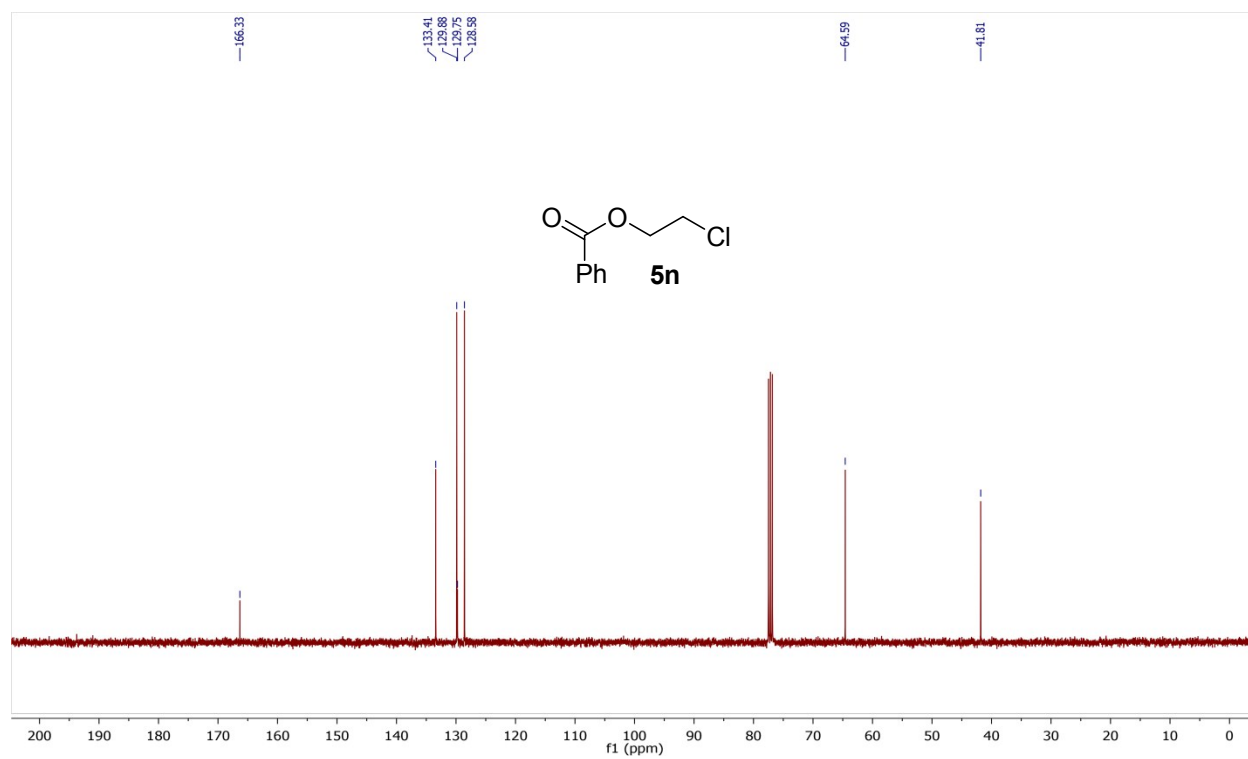
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5I**



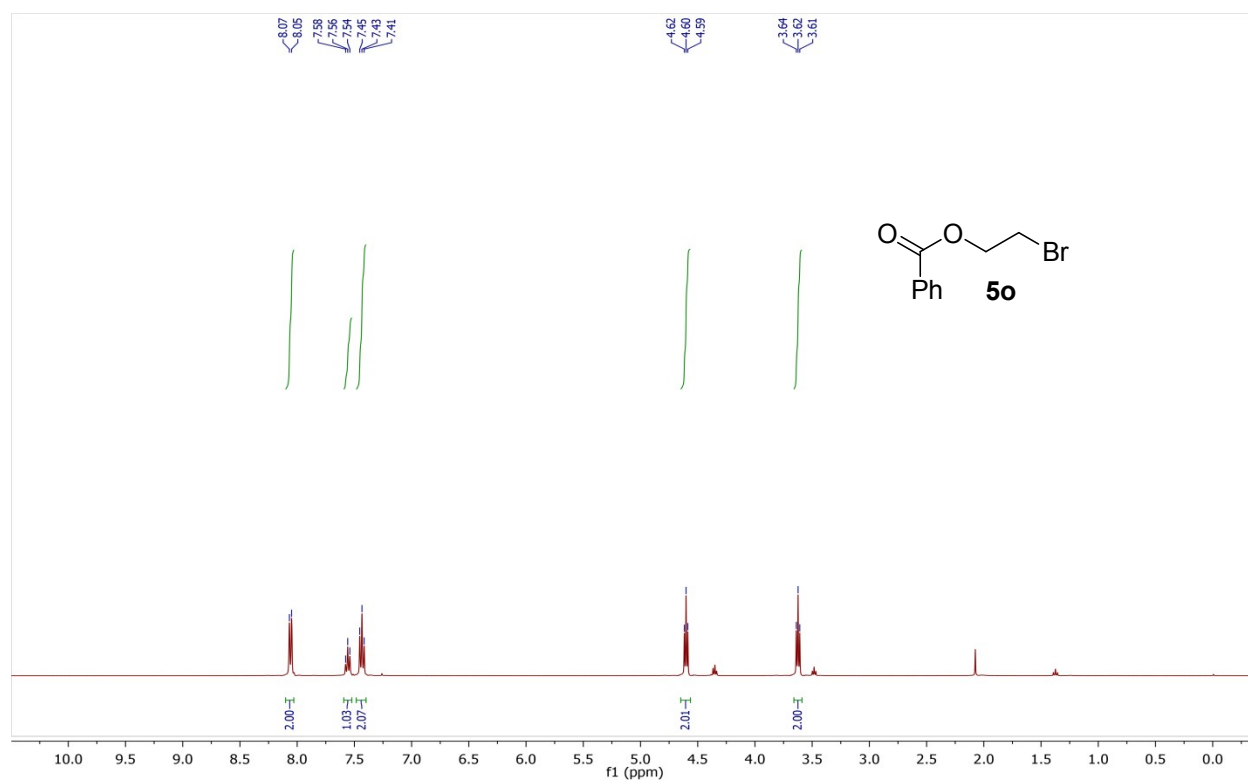




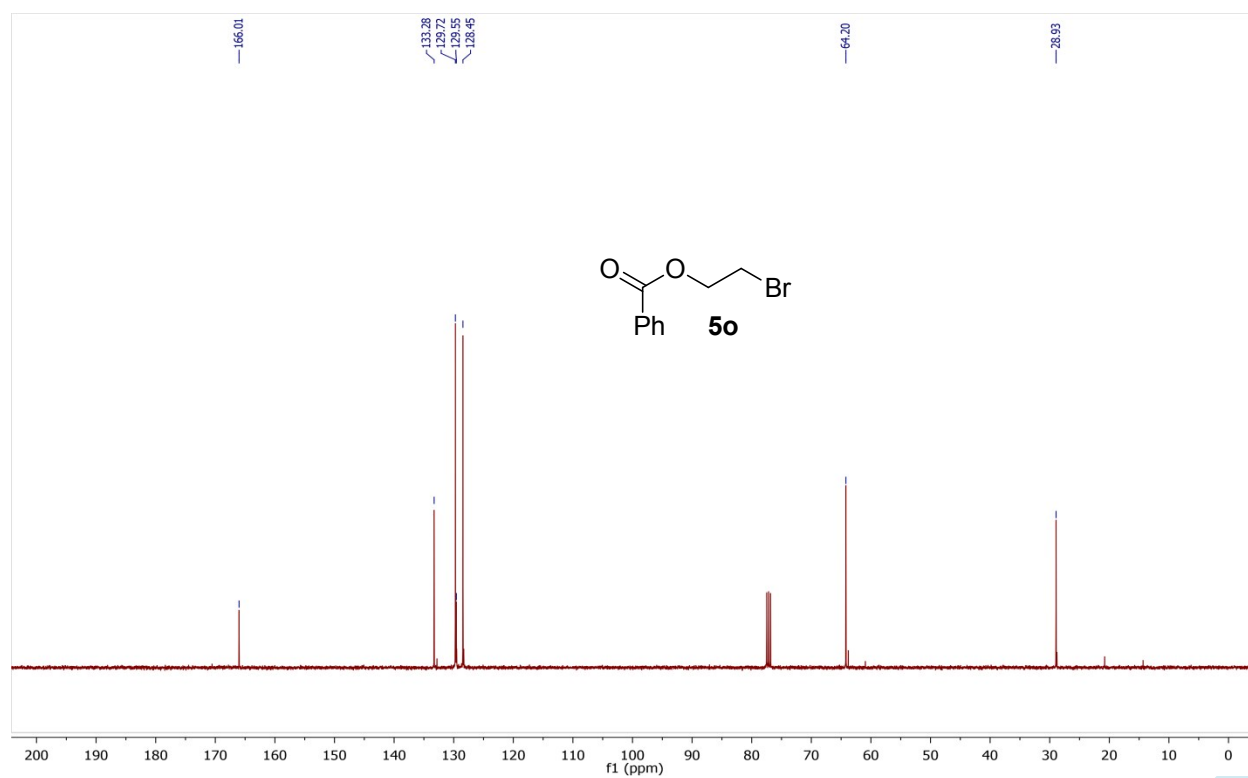
<sup>1</sup>H NMR of compound **5n**



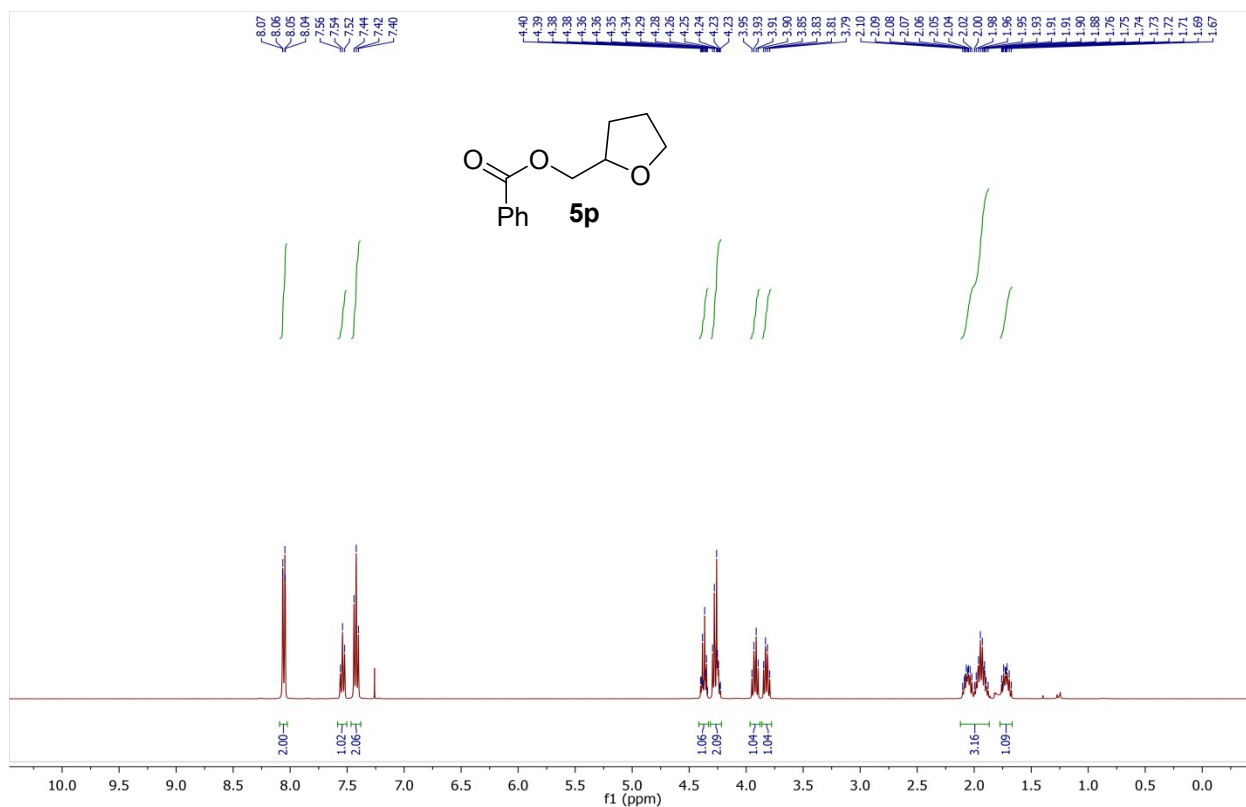
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5n**



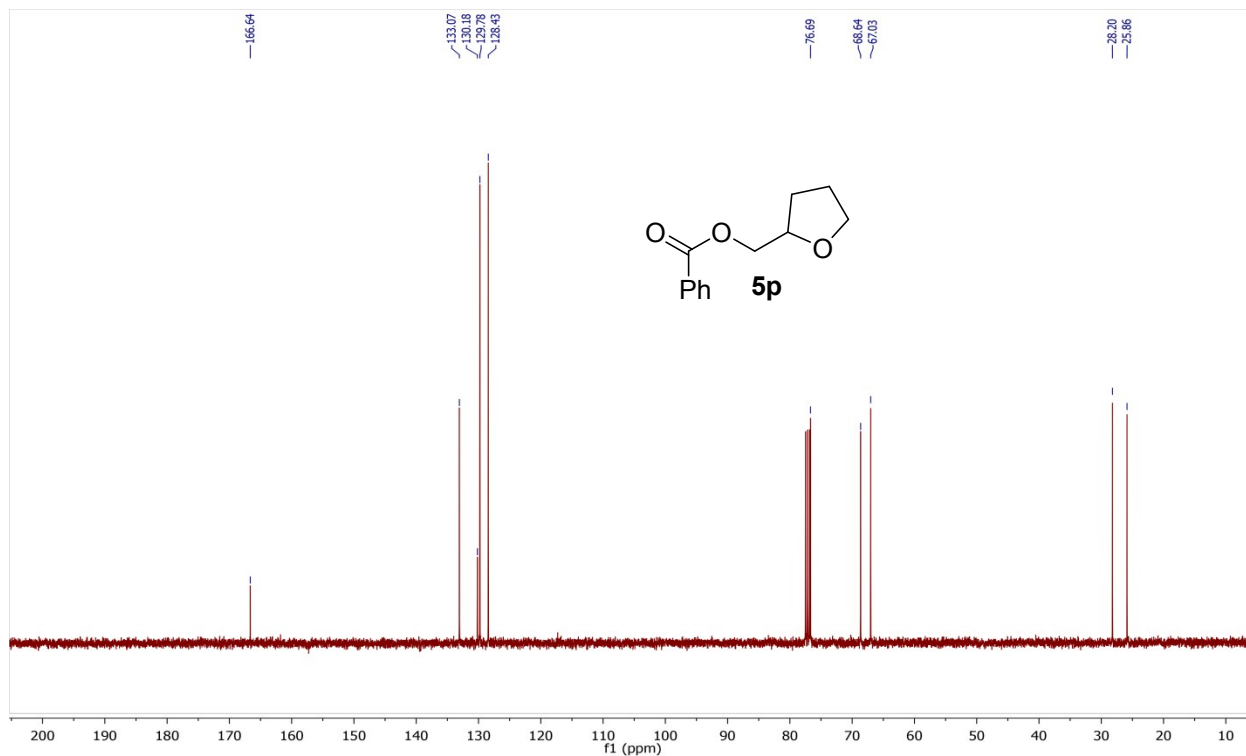
$^1\text{H}$  NMR of compound **5o**



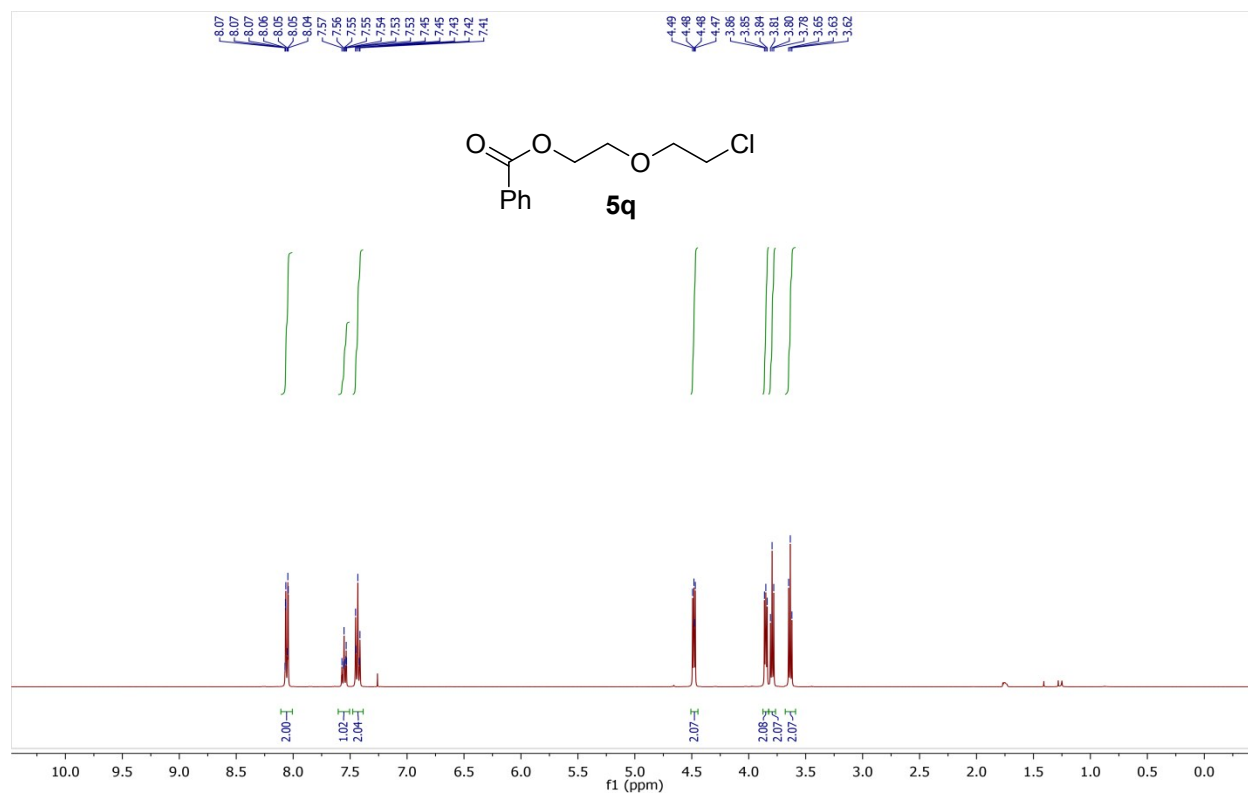
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5p**



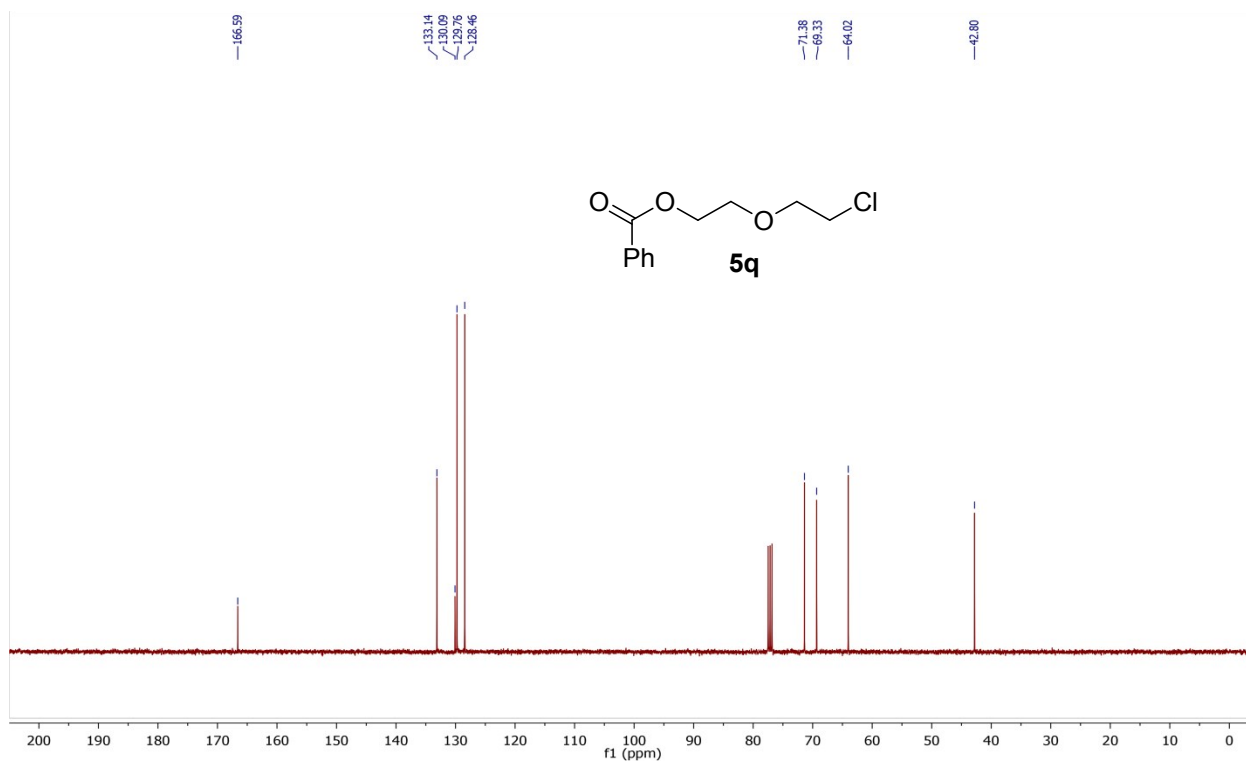
$^1\text{H}$  NMR of compound **5p**



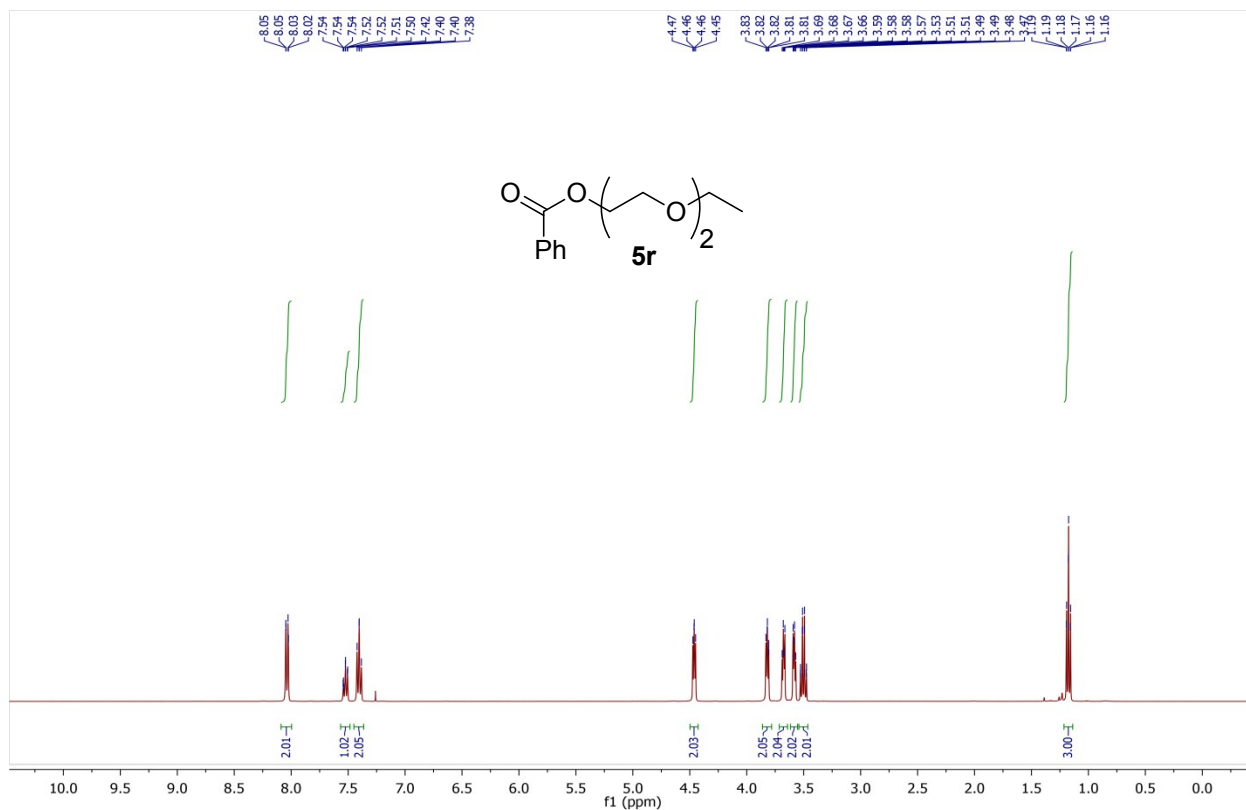
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5p**



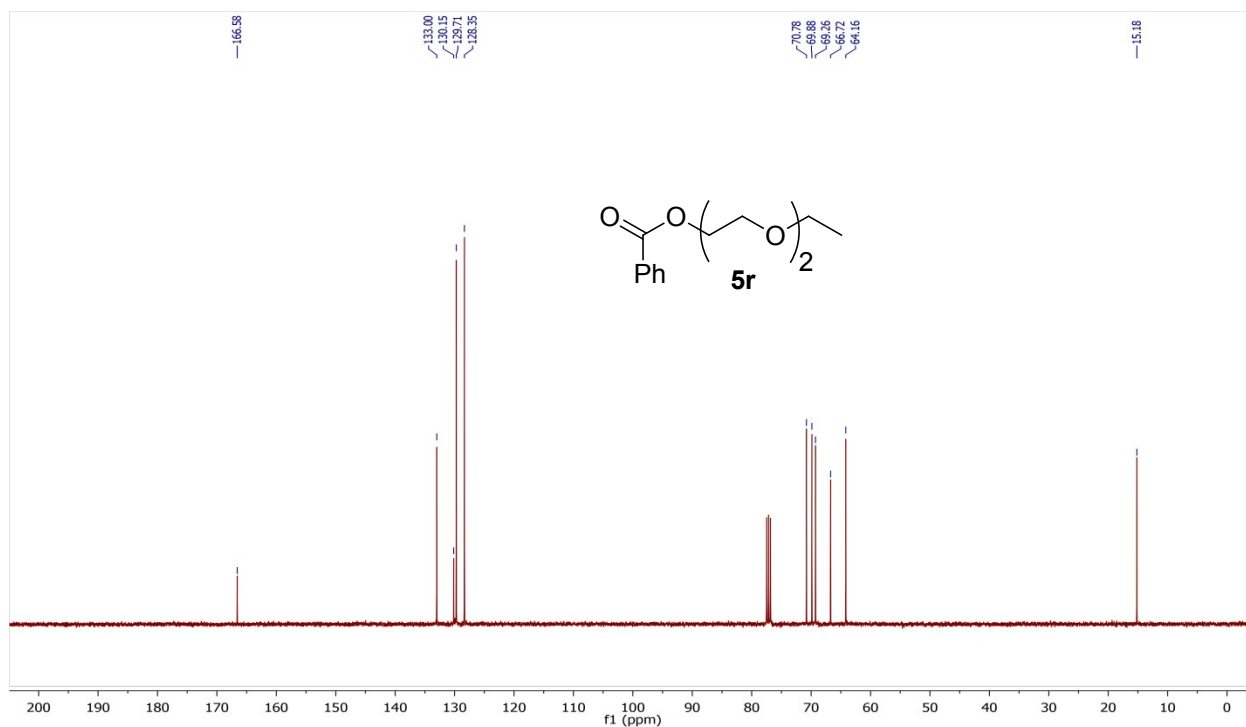
$^1\text{H}$  NMR of compound **5q**



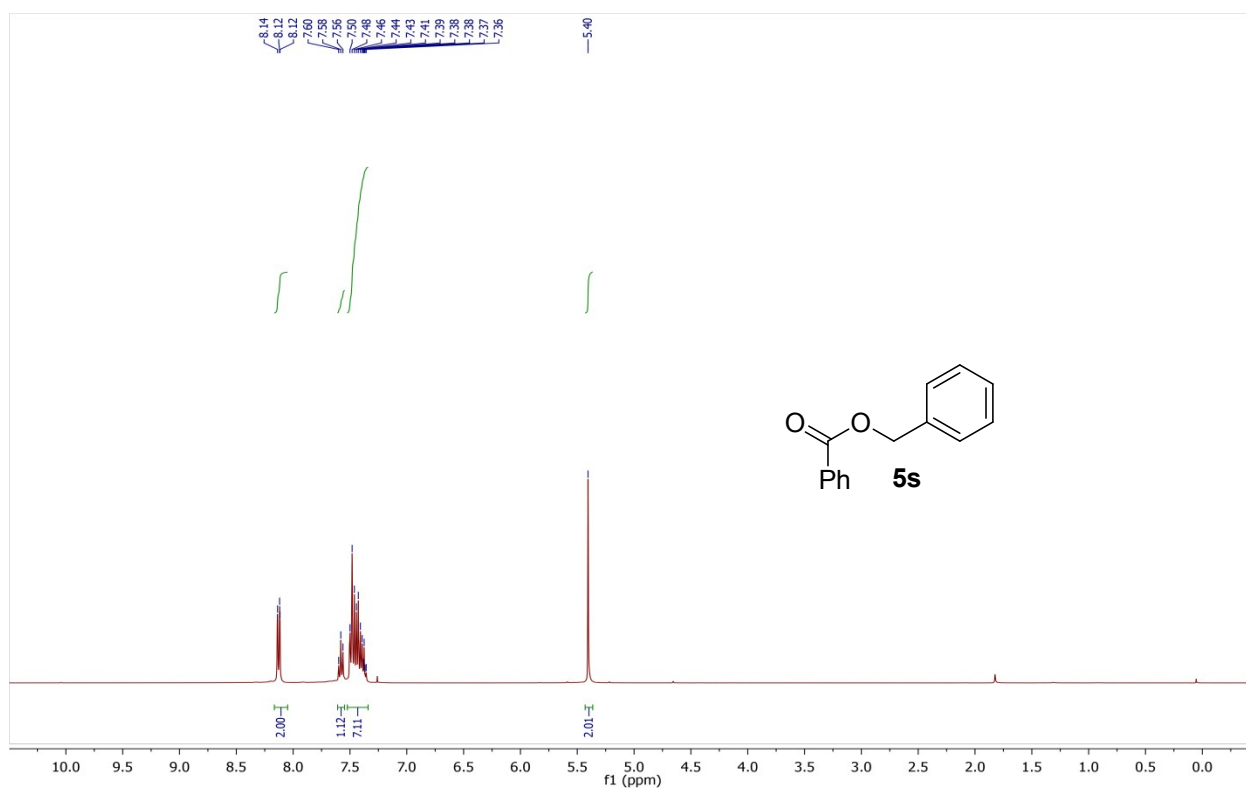
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5q**



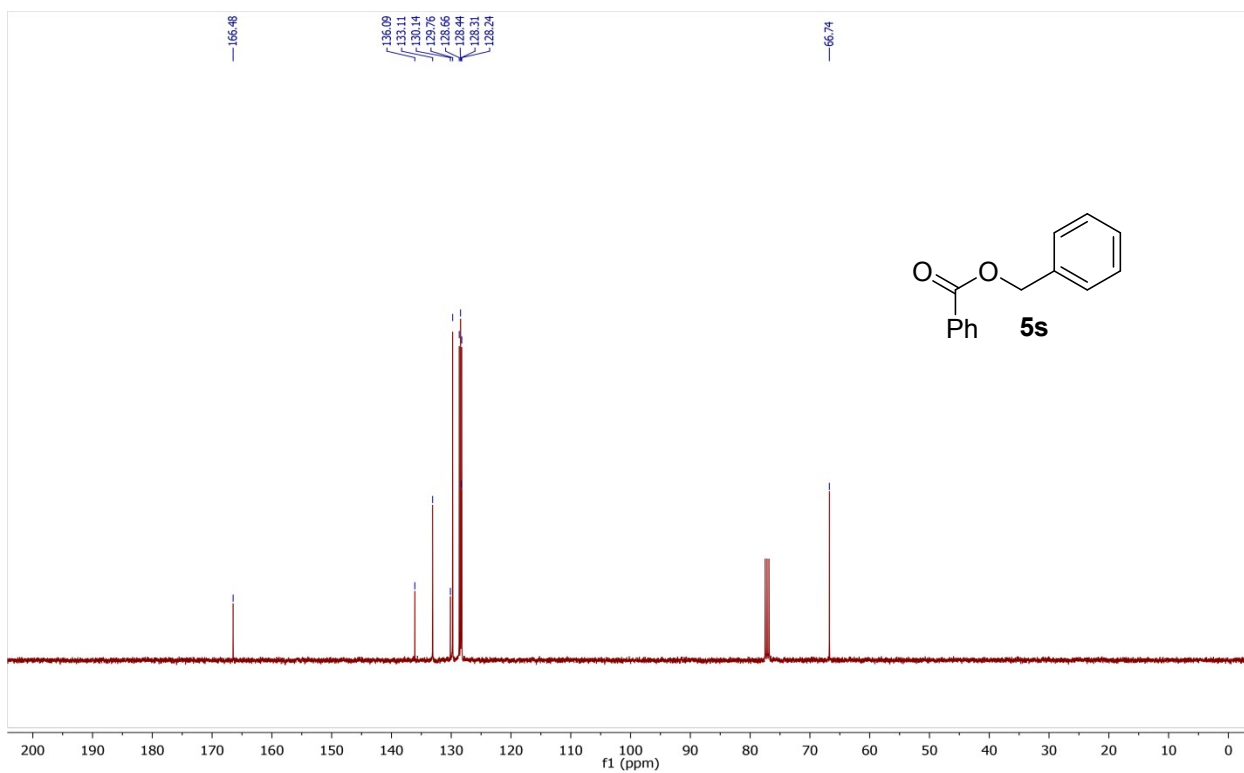
$^1\text{H}$  NMR of compound **5r**



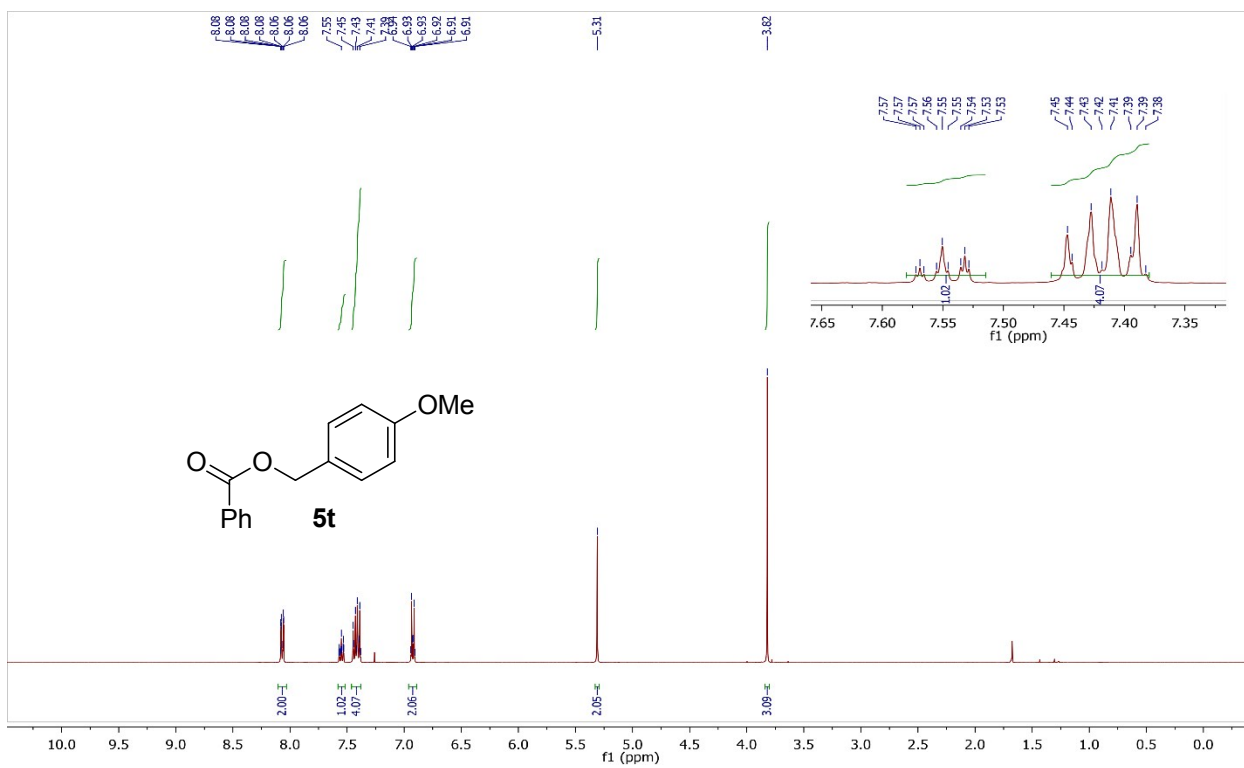
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5r**



$^1\text{H}$  NMR of compound **5s**

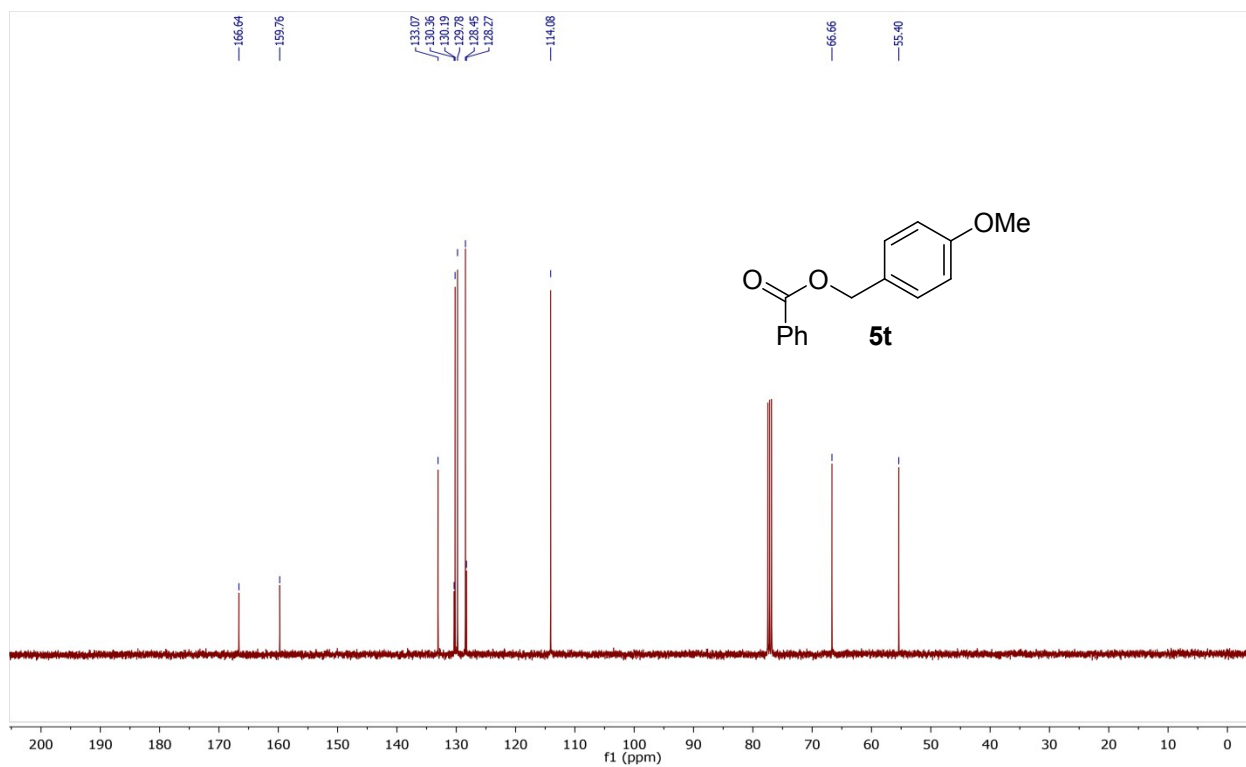


$^{13}\text{C}\{^1\text{H}\}$ NMR of compound **5s**

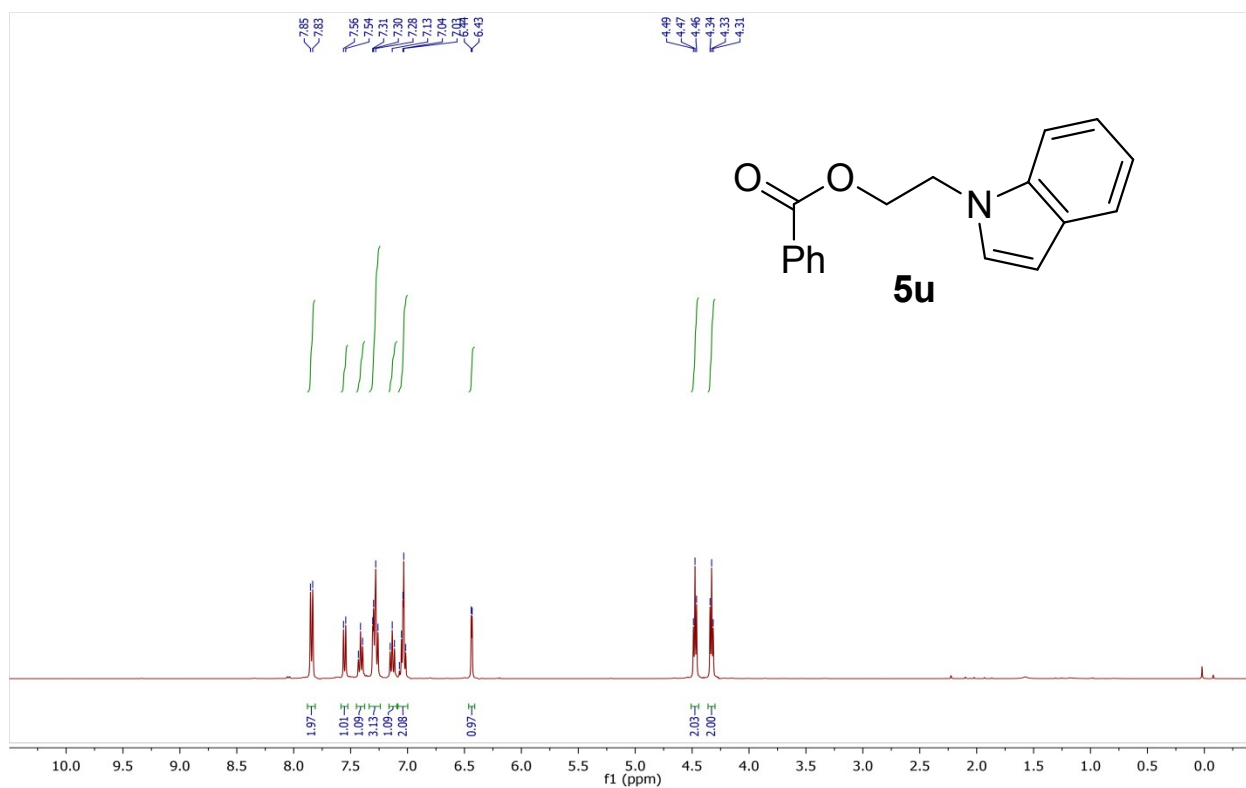


$^1\text{H}$  NMR of compound **5t**

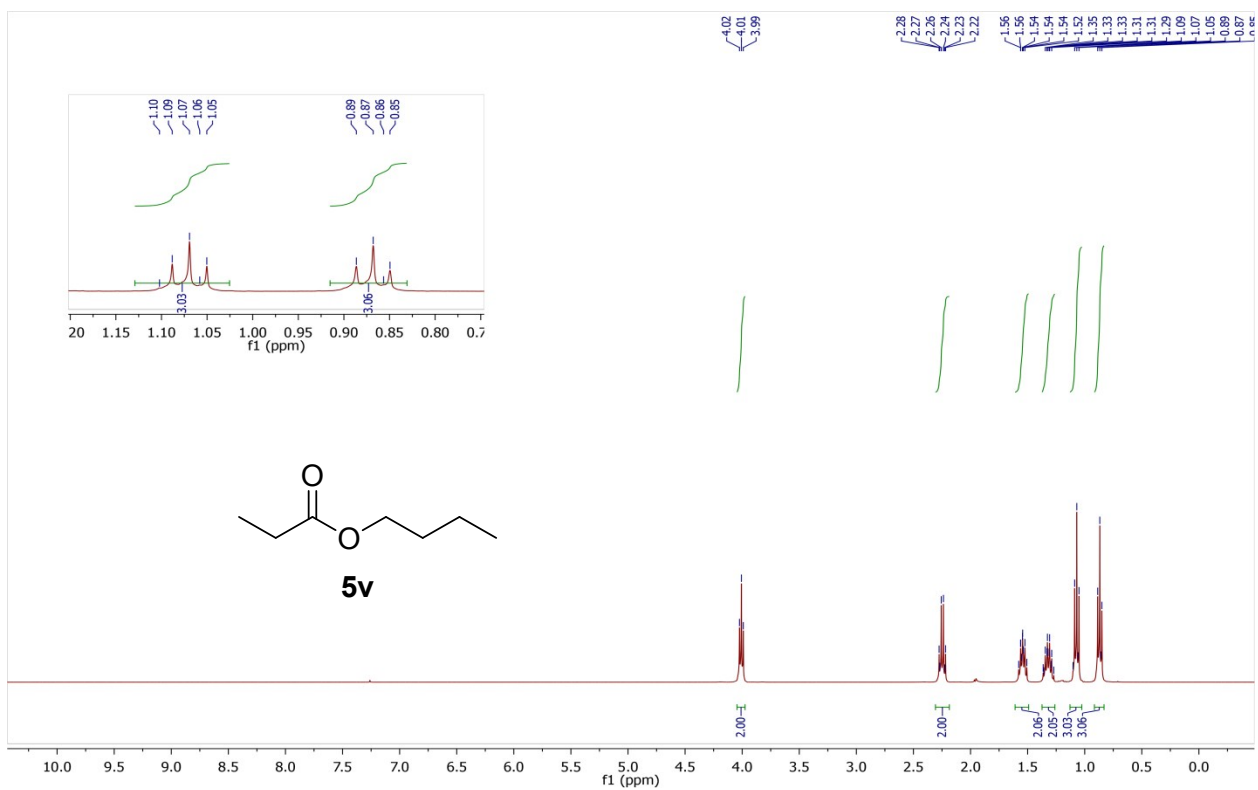
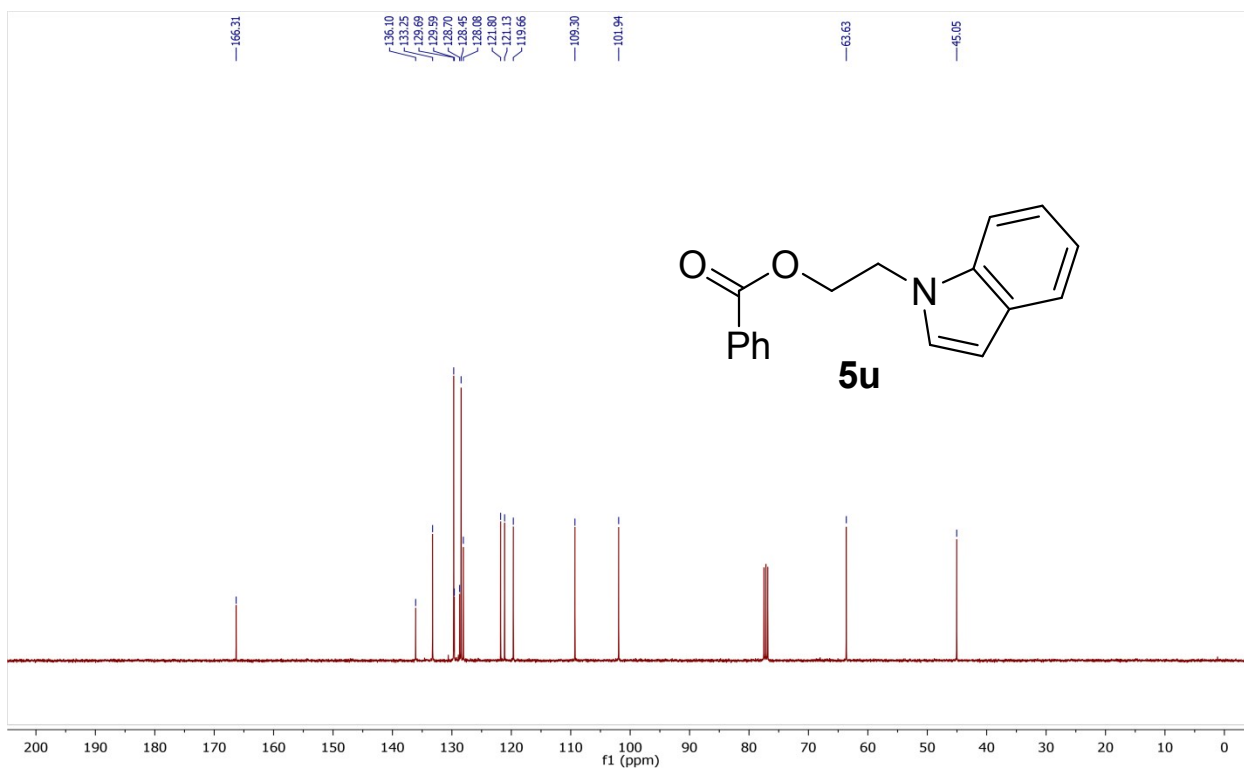


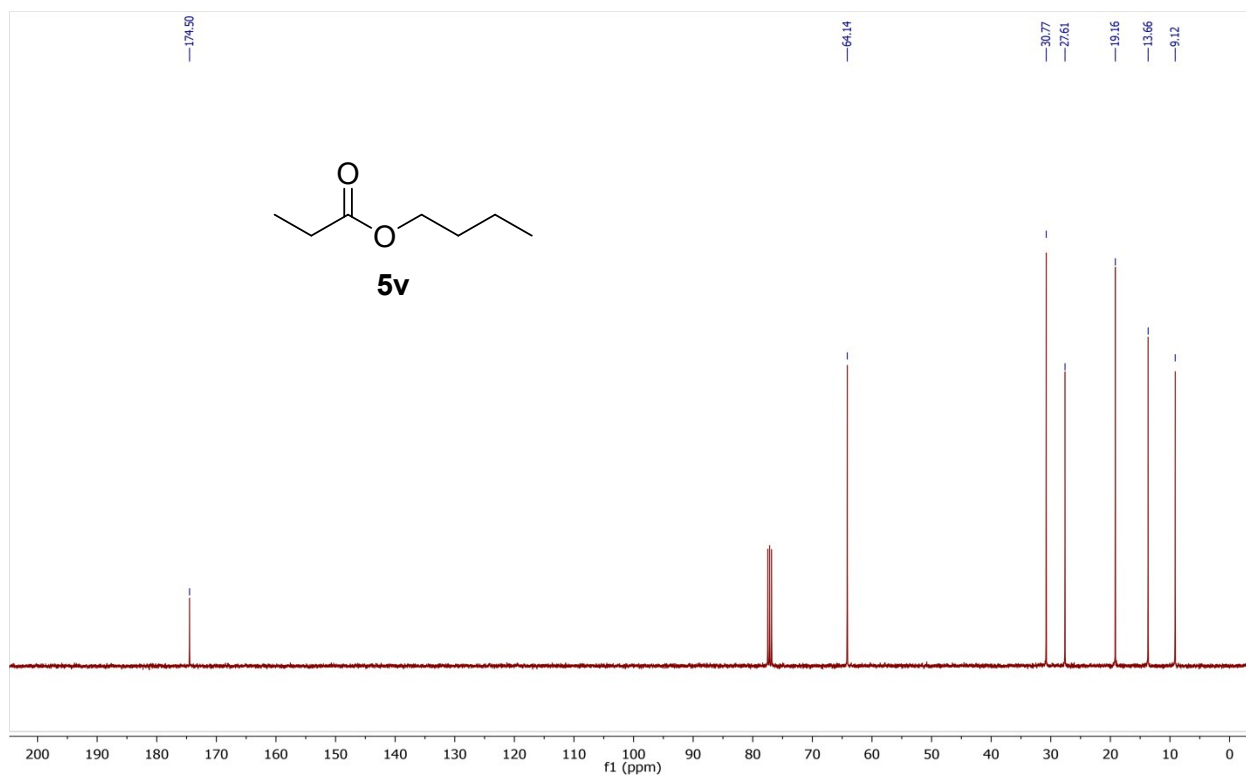


$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5t**

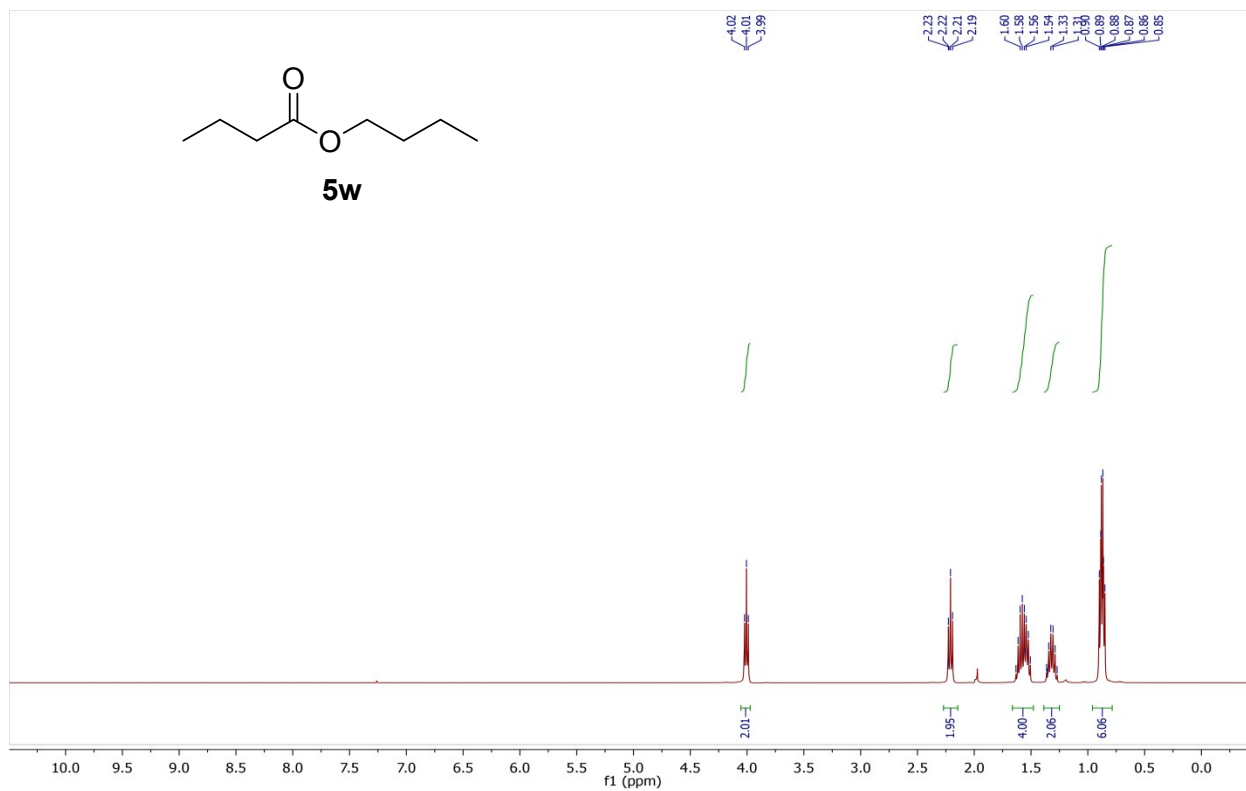


$^1\text{H}$  NMR of compound **5u**

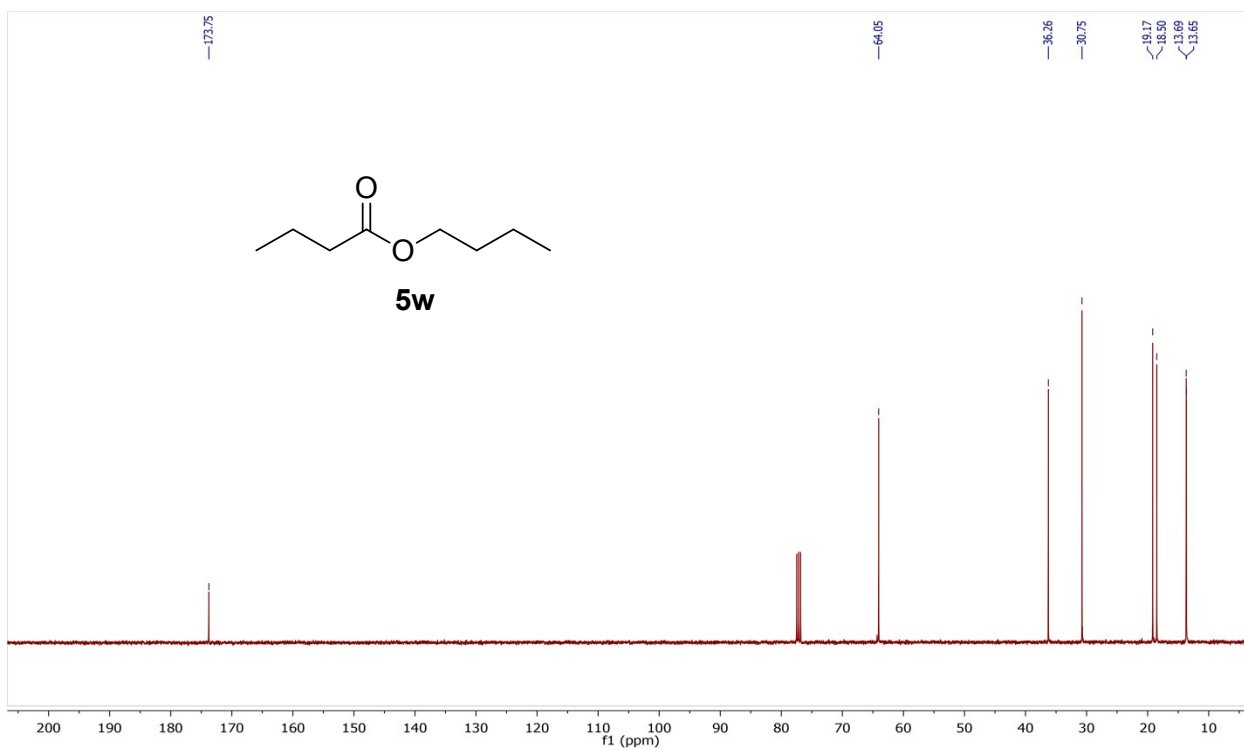




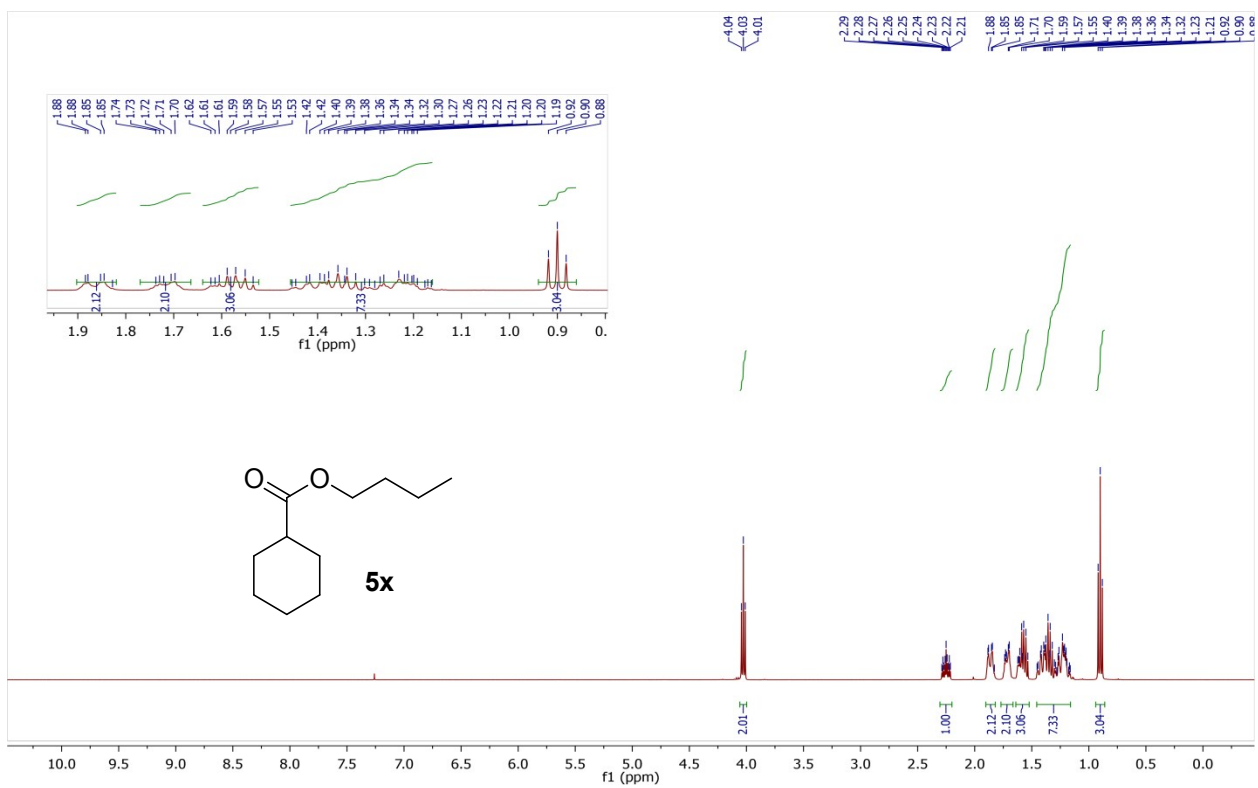
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5v**



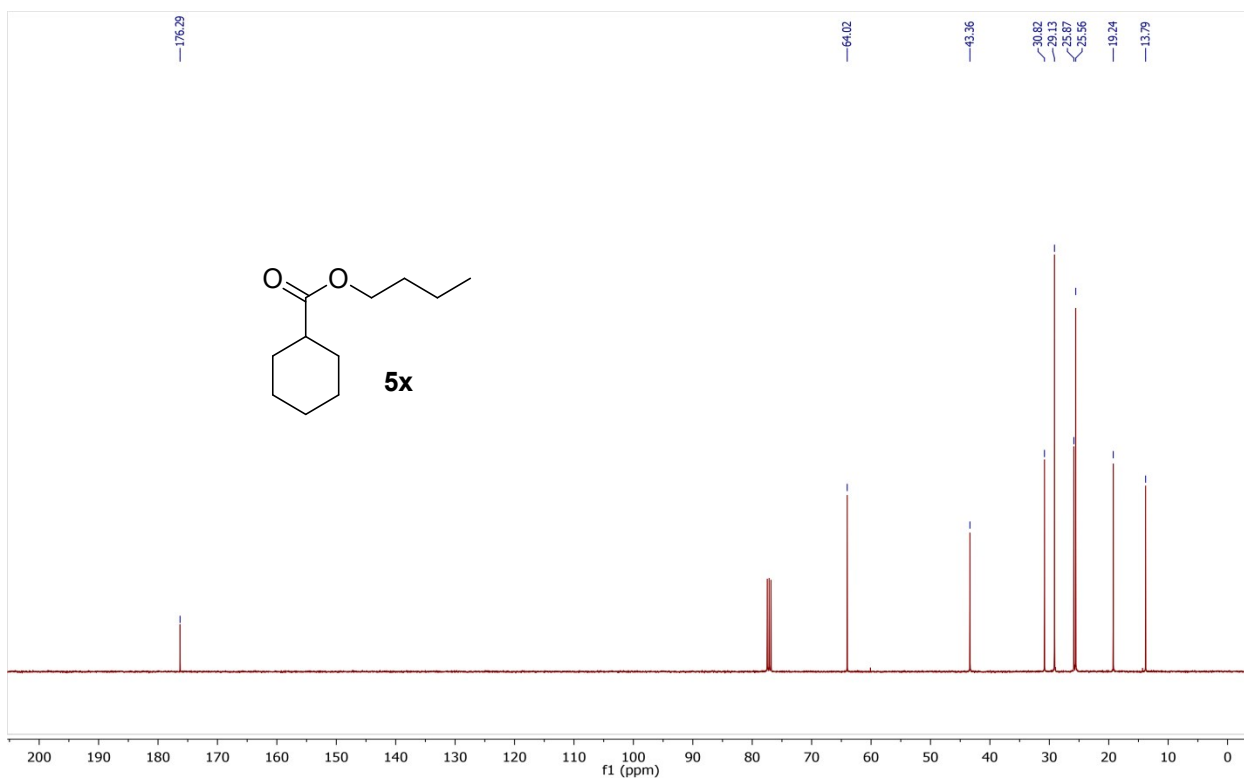
$^1\text{H}$  NMR of compound **5w**



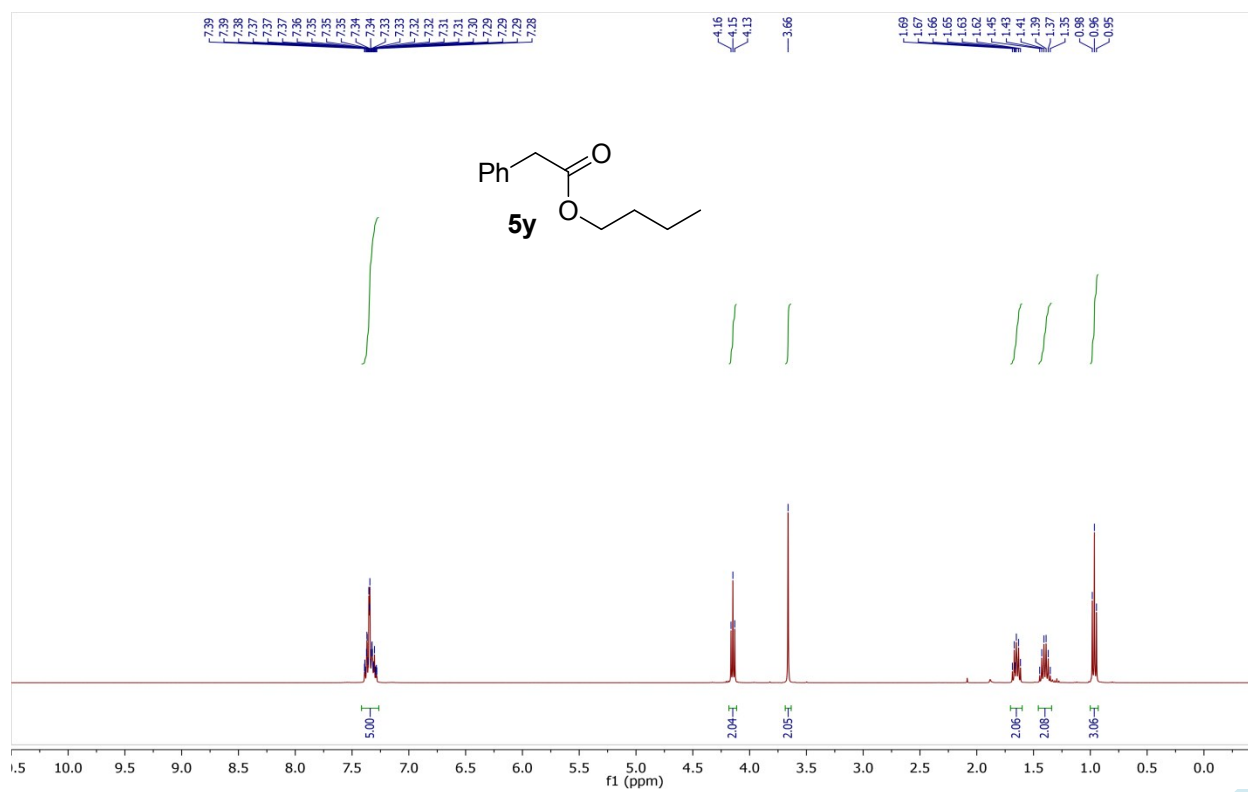
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5w**



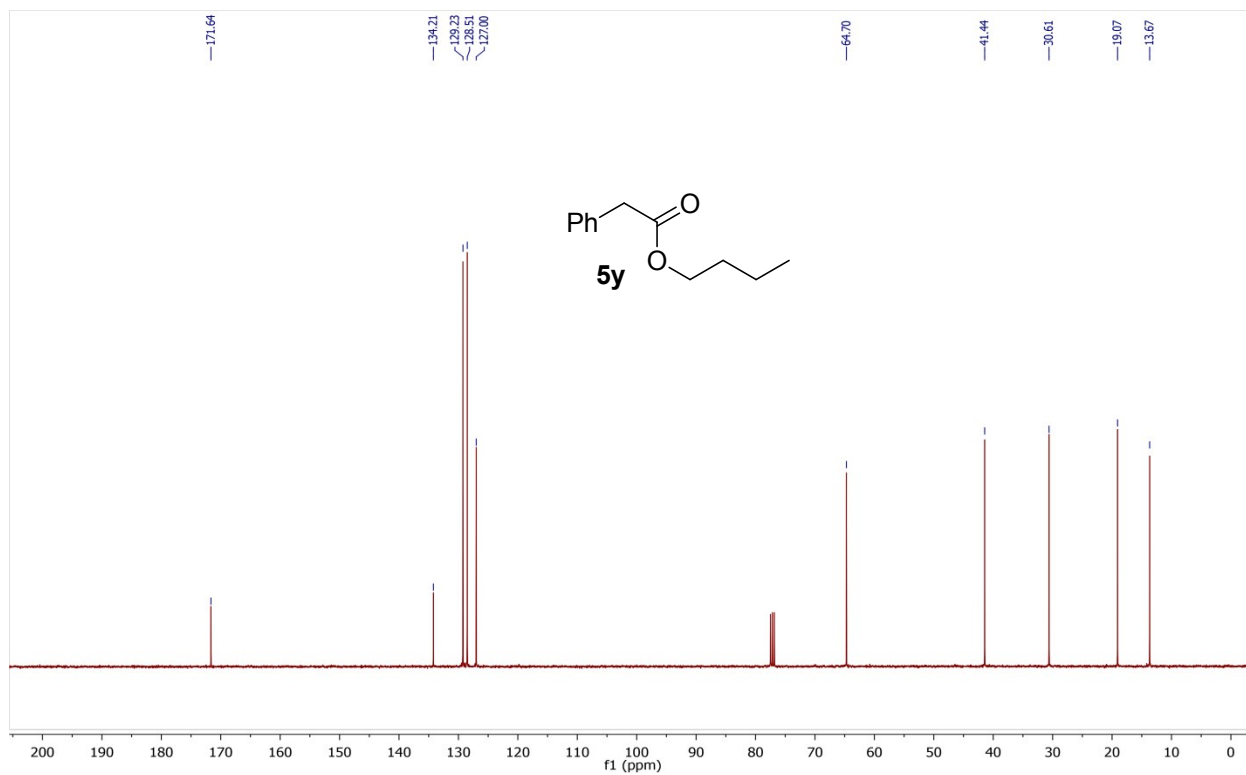
$^1\text{H}$  NMR of compound **5x**



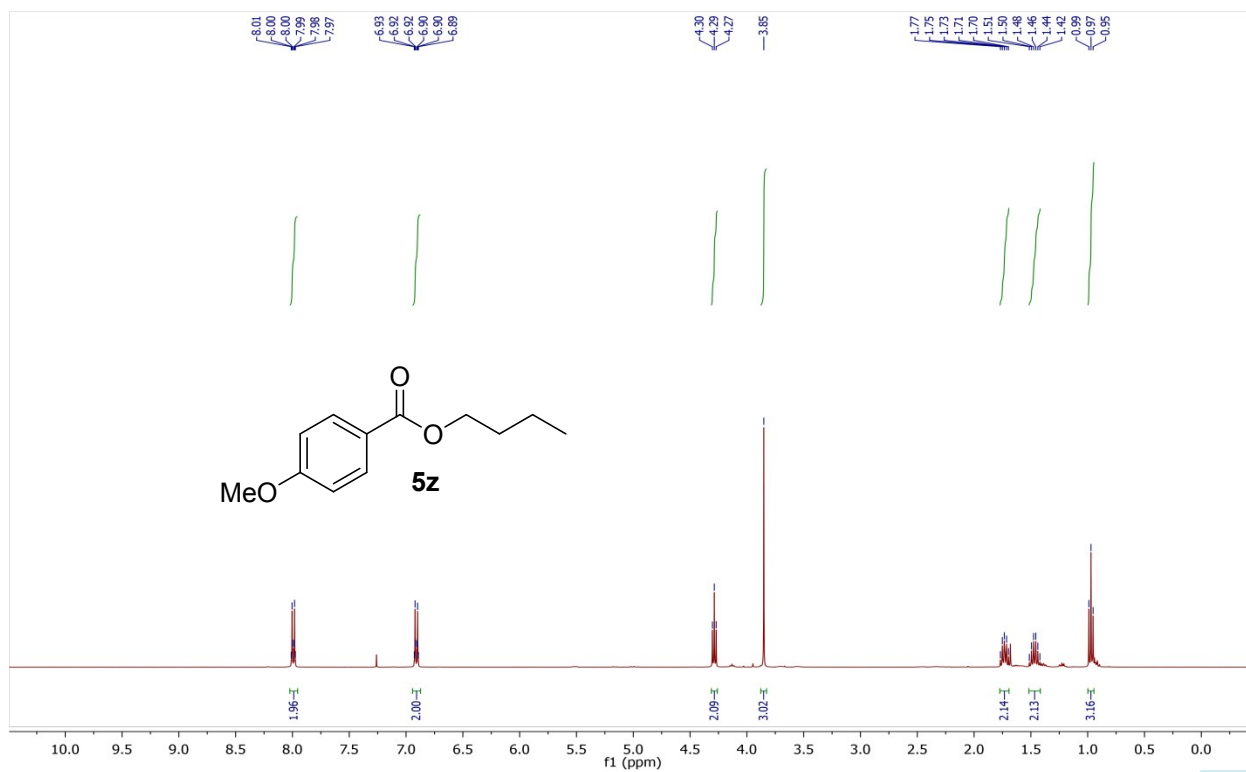
$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5x**



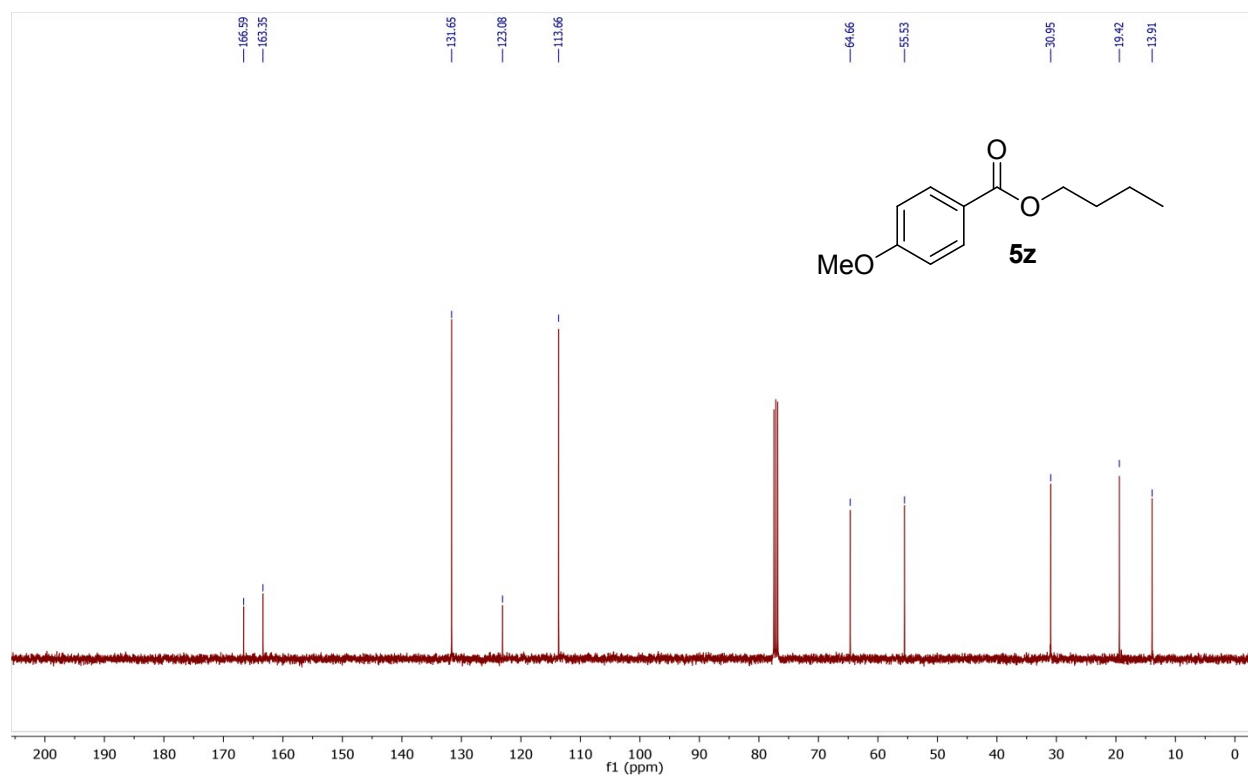
### $^1\text{H}$ NMR of compound **5y**



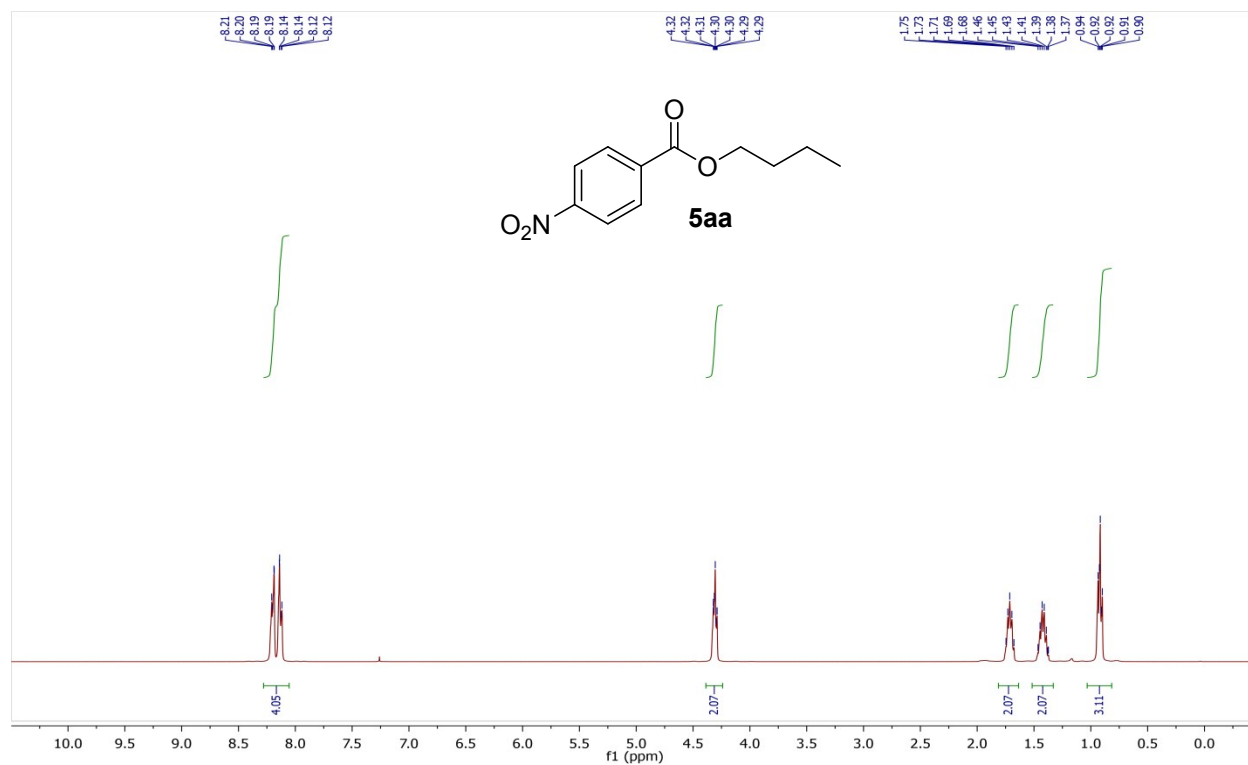
### $^{13}\text{C}\{^1\text{H}\}$ NMR of compound **5y**



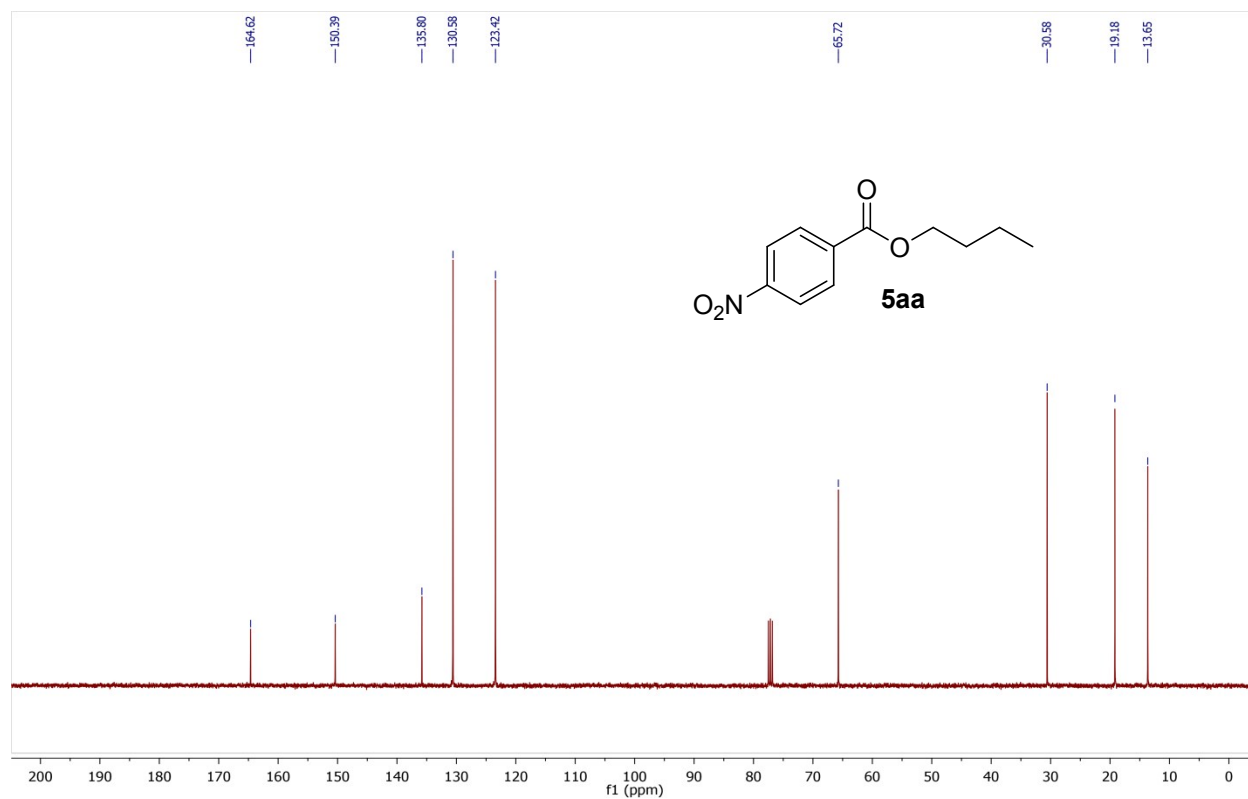
$^1\text{H}$  NMR of compound **5z**



$^{13}\text{C}\{^1\text{H}\}$  NMR of compound **5z**

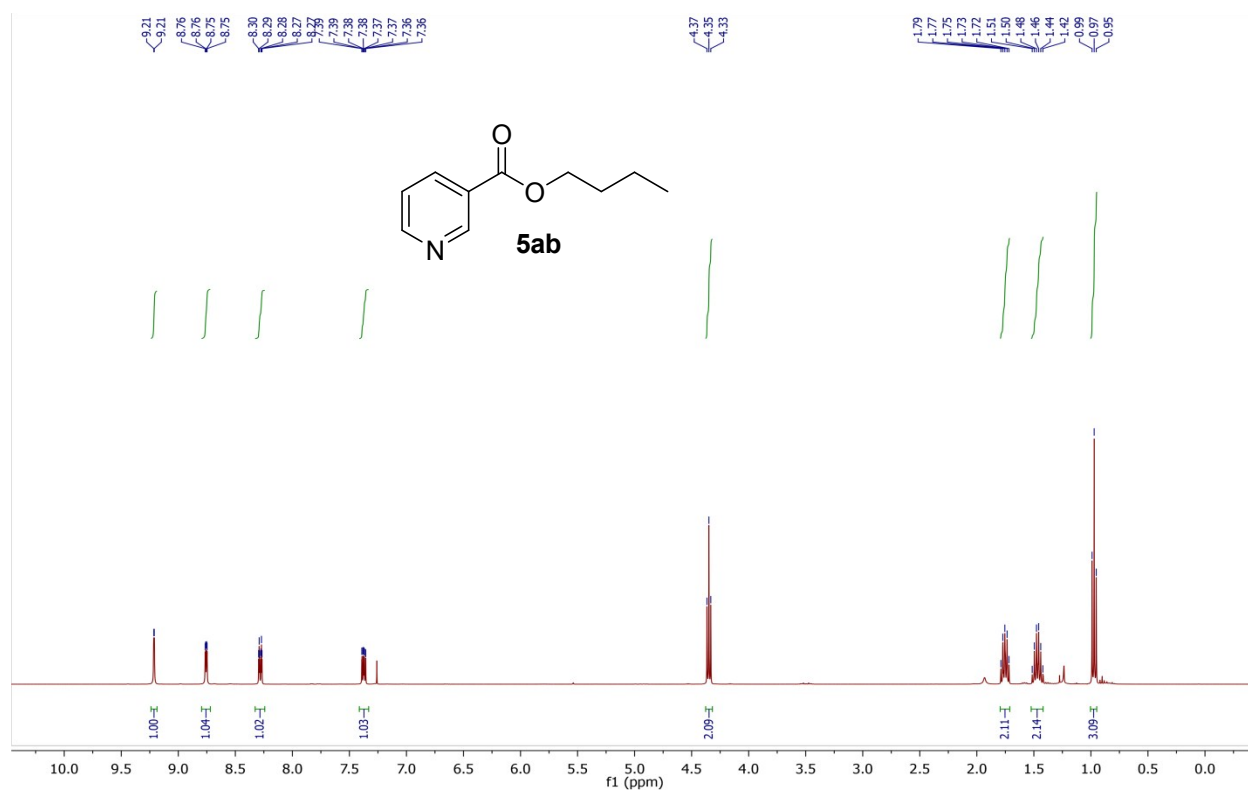


$^1\text{H}$  NMR of compound **5aa**

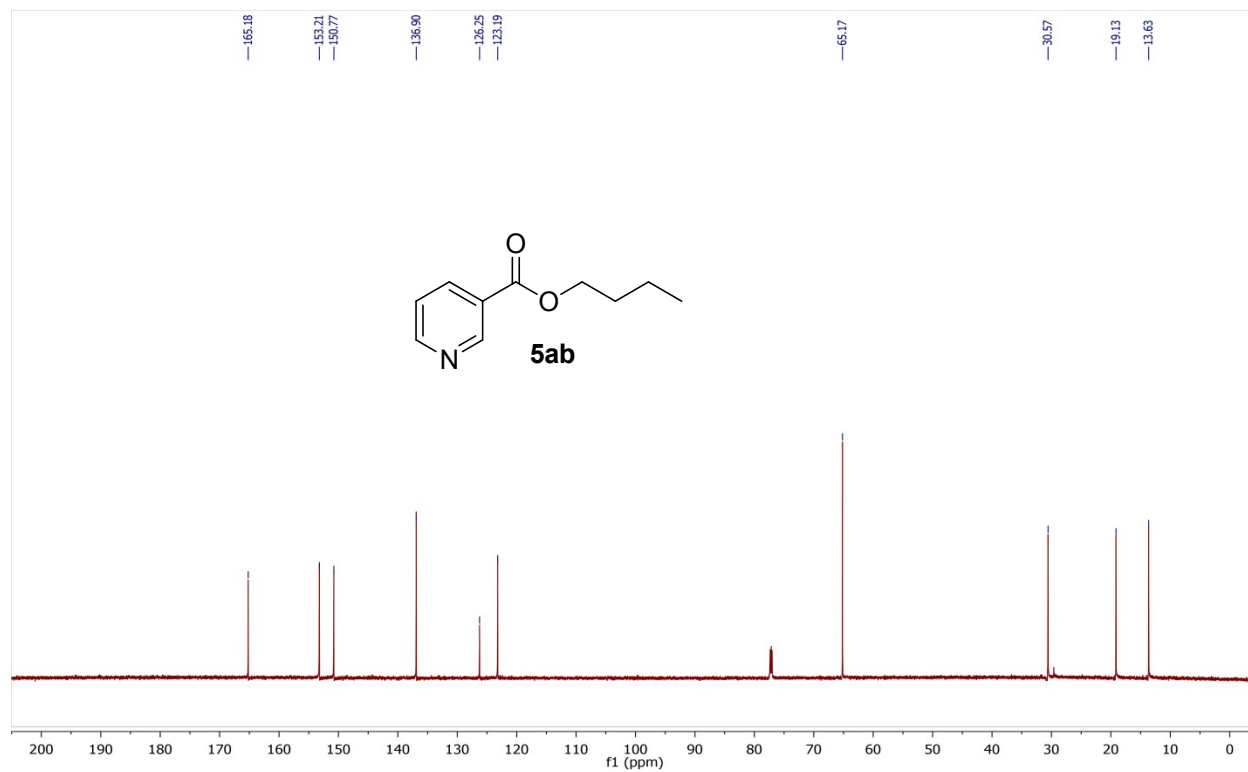


$^{13}\text{C}$   $\{^1\text{H}\}$  NMR of compound **5aa**

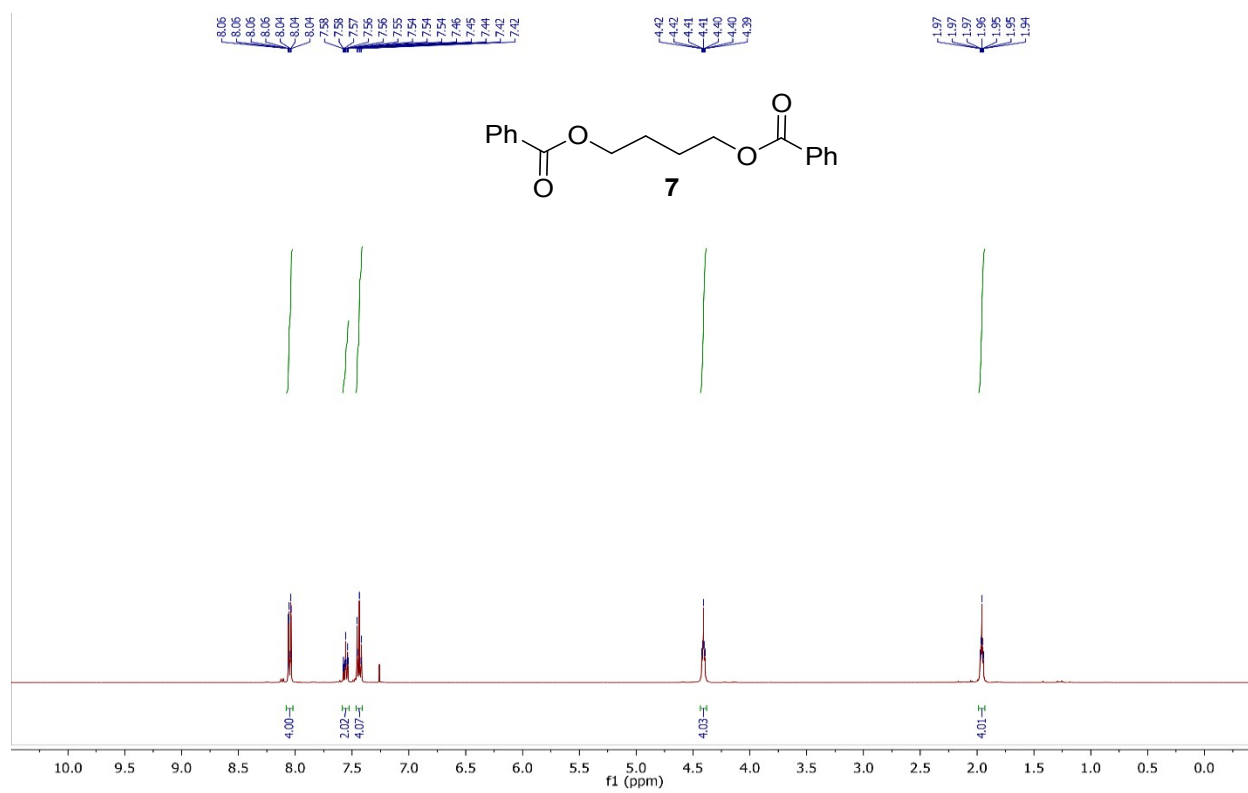




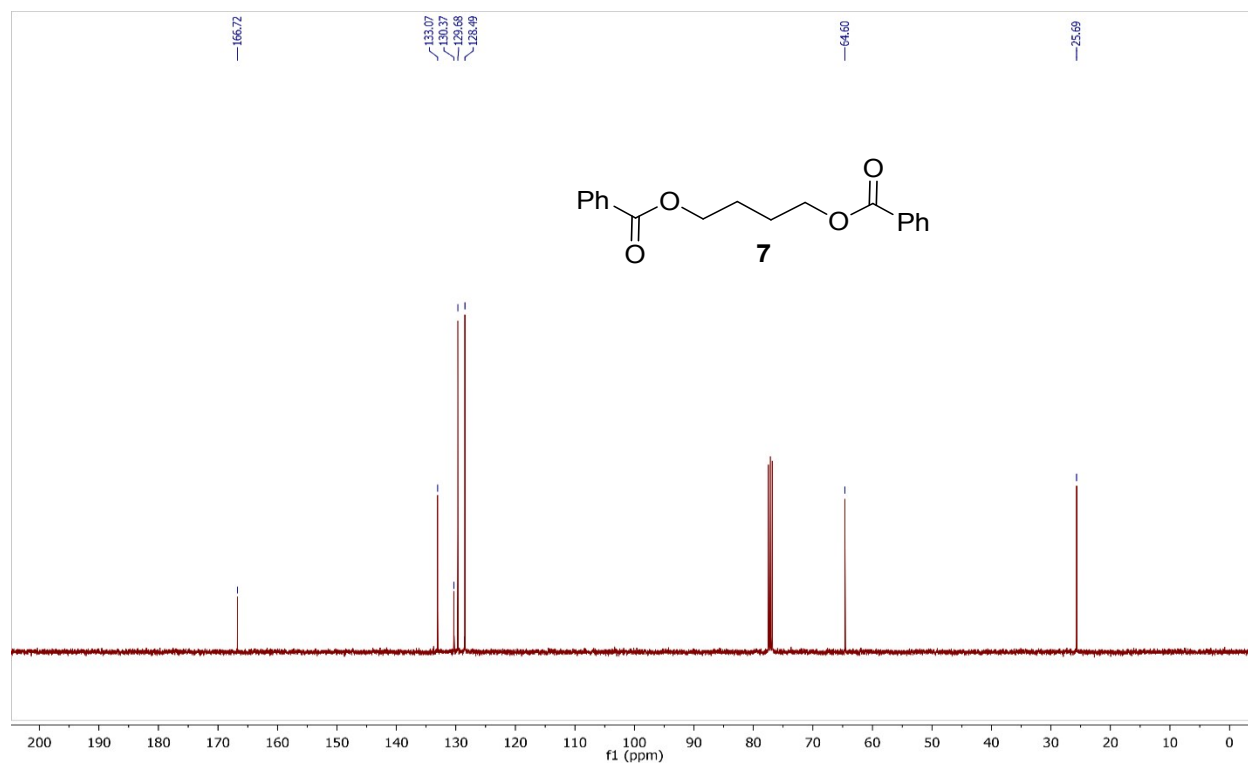
<sup>1</sup>H NMR of compound **5ab**



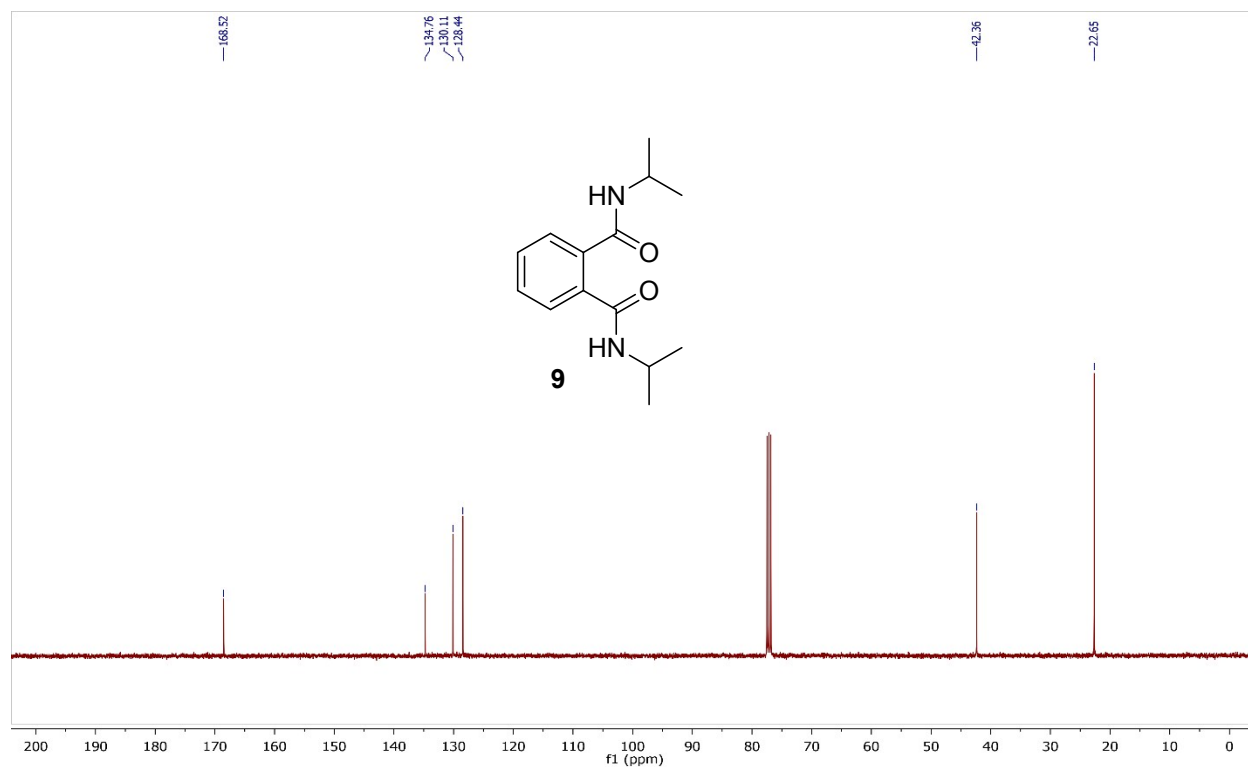
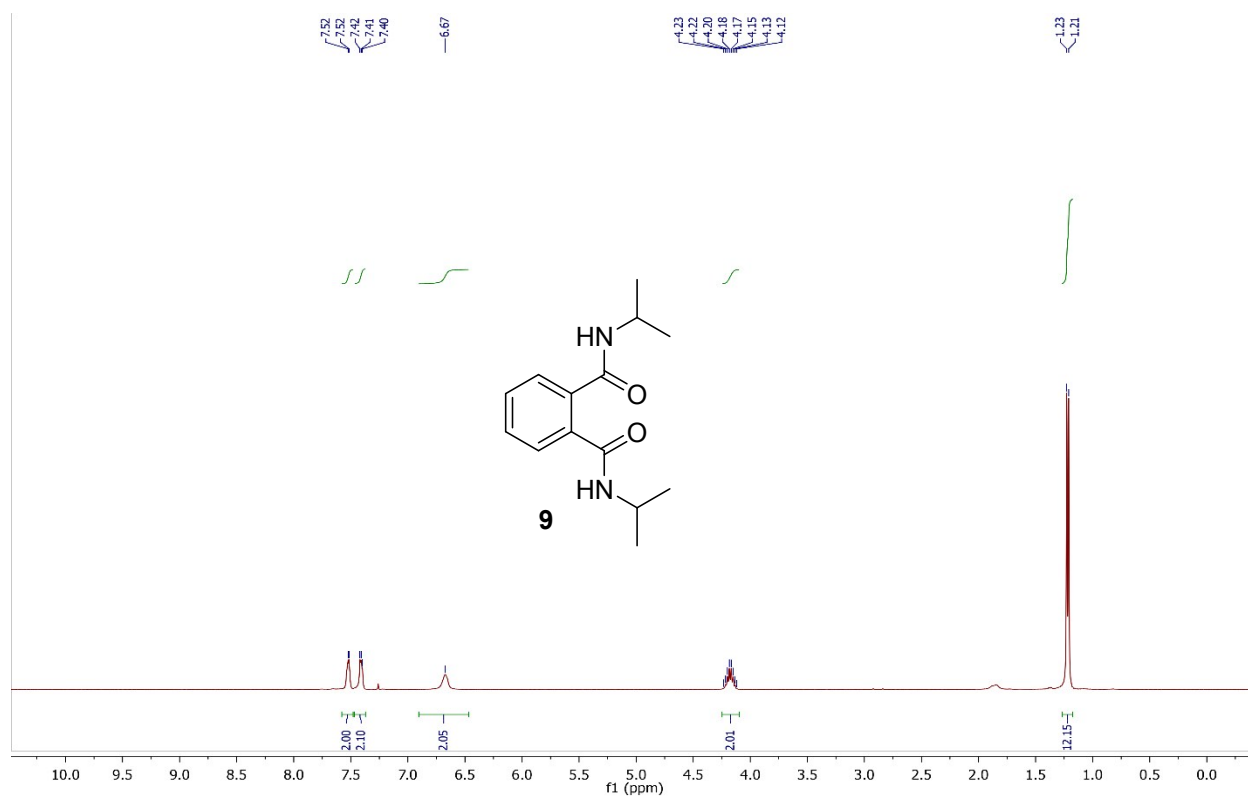
<sup>13</sup>C {<sup>1</sup>H} NMR of compound **5ab**

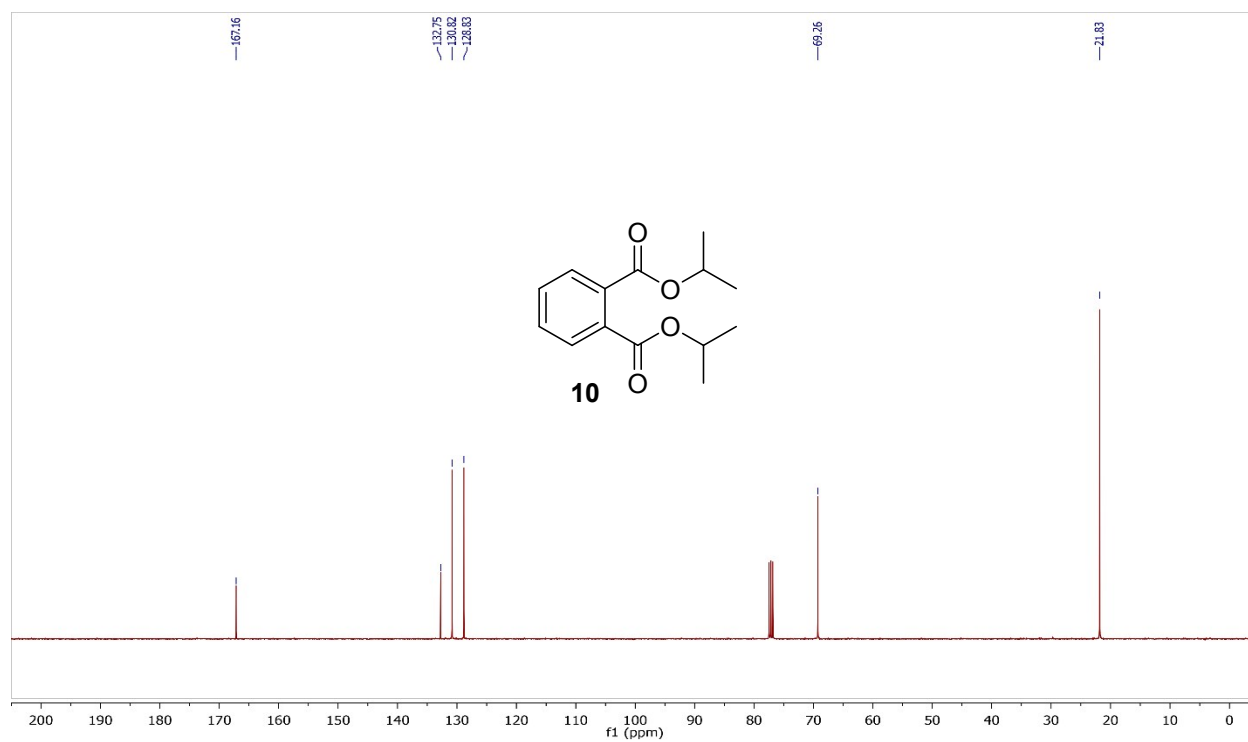


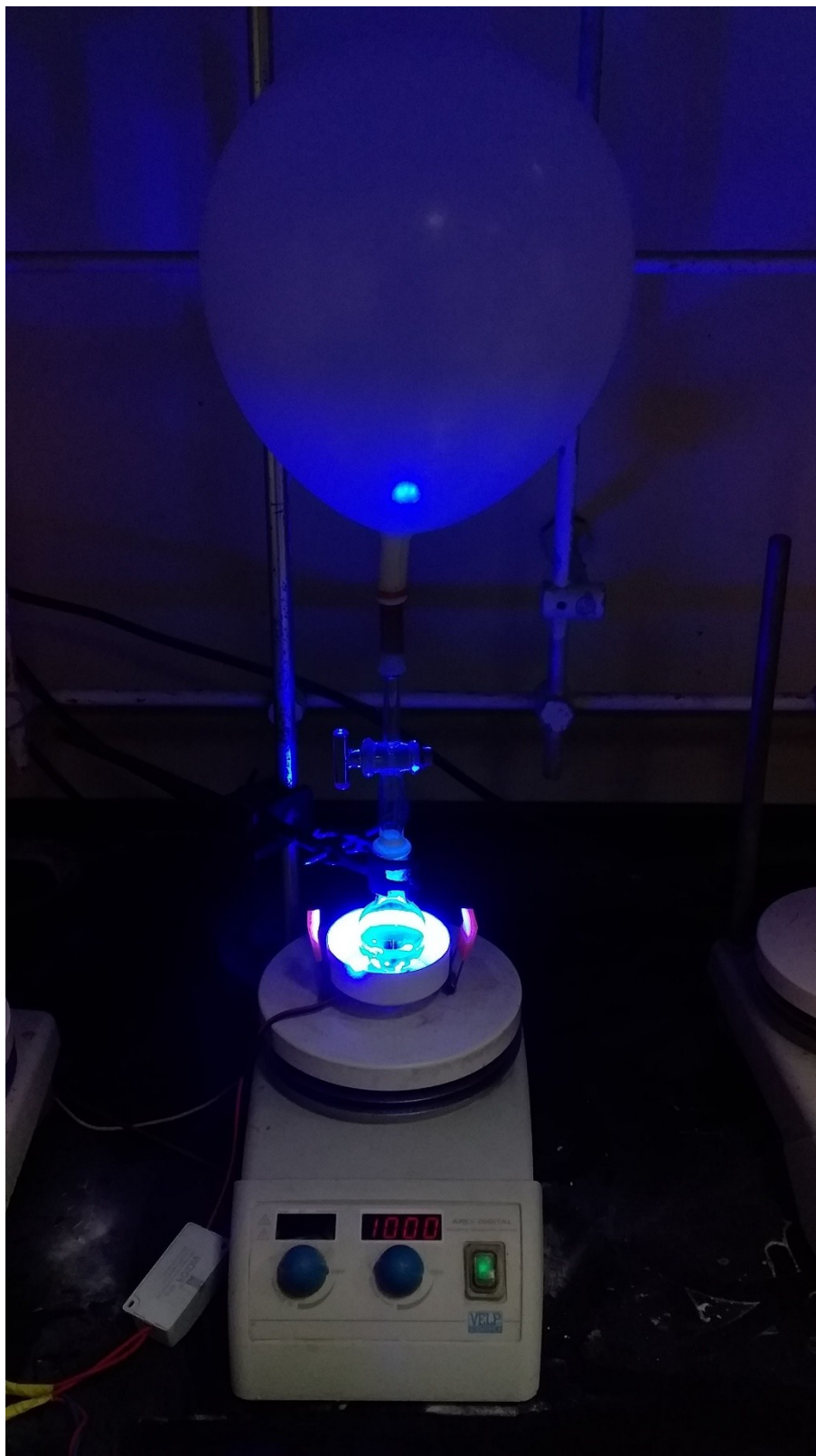
$^1\text{H}$  NMR of compound 7



$^{13}\text{C}$   $\{^1\text{H}\}$  NMR of compound 7







**Picture of Reaction Setup**