## **Supporting Information**

# Highly Efficient Dehydrogenative Cross-Coupling of Aldehydes with Amines and Alcohols

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#### **General Experimental:**

All chemicals were obtained from Sigma-Aldrich Company and used as received. <sup>1</sup>H and <sup>13</sup>C NMR spectras were recorded on Brucker-Avance DPX FT-NMR 500 and 400 MHz instruments. Chemical data for protons are reported in parts per million (ppm) downfield from tetramethylsilane and are referenced to the residual proton in the NMR solvent (CDCl<sub>3</sub>, 7.26 ppm). Carbon nuclear magnetic resonance spectra (<sup>13</sup>C NMR) were recorded at 125 MHz or 100 MHz: chemical data for carbons are reported in parts per million (ppm,  $\delta$  scale) downfield from tetramethylsilane and are referenced to the carbon resonance of the solvent. ESI-MS and HRMS spectra were recorded on Agilent 1100 LC-Q-TOF and HRMS-6540-UHD machines.

### **Experimental procedures:**

General procedure for N-benzoylation of secondary amines; Aldehyde (1 mmol) was added to a solution of secondary amine (1 equiv), base (1 equiv) and TBHP (1.5 equiv) in CH<sub>3</sub>CN (2 ml). The reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC. After completion of reaction, solvent was evaporated under vacuum and residue was purified by column chromatography using ethyl acetate and hexane to afford the desired products.

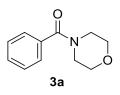
General procedure for N-acetylation of anilines; Acetaldehyde (15 mmol) was added to a solution of amine (1 mmol),  $Bu_4NI$  (0.2 equiv) and TBHP (3 equiv) in CH<sub>3</sub>CN (2 ml). The reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC. After completion of reaction, solvent was evaporated under vacuum and residue was purified by column chromatography using ethyl acetate and hexane to afford the desired products.

General procedure for N-benzoylation of anilines; aldehyde or ethyl 2-oxoacetate (1 mmol) was added to a solution of amine (1 equiv),  $Bu_4NI$  (0.2 equiv) and TBHP (3 equiv) in CH<sub>3</sub>CN (2 ml). The reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC. After completion of reaction, solvent was evaporated under vacuum and residue was purified by column chromatography using ethyl acetate and hexane to afford the desired products.

**General procedure for synthesis of esters;** Aryl aldehyde (1 mmol) was added to a solution of Bu<sub>4</sub>NI (0.2 equiv) and TBHP (3 equiv) in CH<sub>3</sub>OH (2 ml). The reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC. After completion of reaction, solvent was evaporated under vacuum and residue was purified by column chromatography using ethyl acetate and hexane to afford corresponding products.

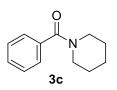
General procedure for synthesis of  $\alpha$ -ketoesters; 2-Oxoaldehydes (1 mmol) was added to a solution of Bu<sub>4</sub>NI (0.2 equiv) and TBHP (3 equiv) in CH<sub>3</sub>OH (2 ml). The reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC. After completion of reaction, solvent was evaporated under vacuum and residue was purified by column chromatography using ethyl acetate and hexane to afford corresponding  $\alpha$ -ketoesters.

#### **Spectroscopic Data:**



**Morpholino(phenyl)methanone (3a);** The title compound was prepared according to the general procedure described above using benzaldehyde (100  $\mu$ l, 0.94 mmol), morpholine (82  $\mu$ l, 0.94 mmol), pyridine (74  $\mu$ l, 0.94 mmol) and TBHP (127  $\mu$ l, 1.41 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as gummy gel product (144 mg, 80% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>1</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.48 – 7.32 (m, 5H), 3.88 – 3.39 (m, 8H).

**Phenyl(thiomorpholino)methanone (3b);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), thiomorpholine (97 µl, 0.94 mmol), pyridine (74 µl, 0.94 mmol) and TBHP (127 µl, 1.41 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as gummy gel product (140 mg, 72% from aldehyde).<sup>1</sup> The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>2</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.48 – 7.29 (m, 5H), 4.02 (s, 2H), 3.67 (s, 2H), 2.79 – 2.49 (m, 4H).



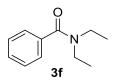
3b

**Phenyl(piperidin-1-yl)methanone (3c);** The title compound was prepared according to the general procedure described above using benzaldehyde (100  $\mu$ l, 0.94 mmol), piperidine (80  $\mu$ l, 0.94 mmol), pyridine (74  $\mu$ l, 0.94 mmol) and TBHP (127  $\mu$ l, 1.41 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as gummy gel (135 mg, 76% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>3</sup> <sup>1</sup>H NMR (400

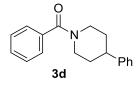
MHz, CDCl<sub>3</sub>) δ 7.58 – 7.27 (m, 5H), 3.71 (s, 2H), 3.34 (s, 2H), 1.76 – 1.43 (m, 6H).

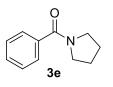
**Phenyl(4-phenylpiperidin-1-yl)methanone (3d);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), 4-phenylpiperidine (151 mg, 0.94 mmol), pyridine (74 µl, 0.94 mmol) and TBHP (127 µl, 1.41 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as light brown solid (195 mg, 78% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>4</sup> <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.50 – 7.45 (m, 5H), 7.39 – 7.35 (m, 2H), 7.27 (dd, *J* = 7.2, 5.5 Hz, 3H), 4.94 (s, 1H), 3.93 (s, 1H), 3.28 – 2.78 (m, 3H), 2.11 – 1.71 (m, 4H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  170.4, 145.1, 136.2, 129.6, 128.6, 128.5, 126.9, 126.7, 126.6, 48.4, 42.8, 33.9, 32.9.

**Phenyl(pyrrolidin-1-yl)methanone (3e);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), pyrrolidine (66 µl, 0.94 mmol), pyridine (74 µl, 0.94 mmol) and TBHP (127 µl, 1.41 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as gummy gel (135 mg, 82% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>3</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.53 (d,*J* = 7.2 Hz, 2H), 7.43 (dd, *J* = 5.2, 1.2 Hz, 3H), 3.65 (t, *J* = 6.9 Hz, 2H), 3.42 (t, *J* = 6.6 Hz, 2H), 1.97 – 1.85 (m, 4H).



**N,N-Diethylbenzamide (3f);** The title compound was prepared according to the general procedure described above using benzaldehyde (100  $\mu$ l, 0.94 mmol), diethylamine (63  $\mu$ l, 0.94 mmol), pyridine (74  $\mu$ l, 0.94 mmol) and TBHP (127  $\mu$ l, 1.41 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was



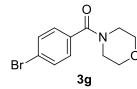


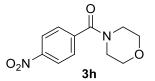
monitored by TLC, purified by column chromatography as liquid product (113 mg, 68% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>1</sup> <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.46 – 7.30 (m, 5H), 3.54 (d, *J* = 5.2 Hz, 2H), 3.24 (d, *J* = 5.1 Hz, 2H), 1.27 – 1.07 (m, 6H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  171.3, 137.2, 129.1, 128.4, 126.2, 43.3, 39.2, 14.2, 12.9.

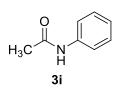
(4-Bromophenyl)(morpholino)methanone (3g); The title compound was prepared according to the general procedure described above using 4-bromo benzaldehyde (100 µl, 0.54 mmol), morpholine (47 µl, 0.54 mmol), pyridine (43 µl, 0.54 mmol) and TBHP (73 µl, 0.81 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as solid (107 mg, 73% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>1</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.55 (d, *J* = 8.5 Hz, 2H), 7.29 (d, *J* = 8.3 Hz, 2H), 3.58 (m, 8H).

**Morpholino(4-nitrophenyl)methanone (3h);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.66 mmol), morpholine (57 µl, 0.66 mmol), pyridine (52 µl, 0.66 mmol) and TBHP (89 µl, 0.99 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as solid (121 mg, 78% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>3</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.32 – 8.23 (m, 2H), 7.60 (dd, *J* = 7.2, 1.5 Hz, 1H), 3.85 – 3.35 (m, 8H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  168.0, 148.4, 141.4, 128.2, 123.9, 66.7, 48.0, 42.5.

**N-Phenylacetamide (3i);** The title compound was prepared according to the general procedure described above using acetaldehyde (709  $\mu$ l, 16.12 mmol), aniline (100  $\mu$ l, 1.07 mmol), Bu<sub>4</sub>NI (79 mg, 0.21 mmol) and TBHP





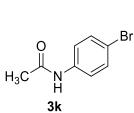


(289 µl, 3.21 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (84 mg, 58% from amime). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>5</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.50 (d, *J* = 7.8 Hz, 2H), 7.42 (s, 1H), 7.31 (t, *J* = 7.8 Hz, 2H), 7.10 (t, *J* = 7.4 Hz, 1H), 2.17 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  169.1, 138.1, 128.9, 124.3, 120.2, 24.4.

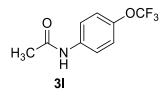
**N-(p-tolyl)acetamide (3j);** The title compound was prepared according to the general procedure described above using acetaldehyde (616 µl, 14.0 mmol), *p*-toluidine (100 mg, 0.93 mmol), Bu<sub>4</sub>NI (68 mg, 0.18 mmol) and TBHP (251 µl, 2.79 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (86 mg, 63% from amine). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>5 1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.41 (d, *J* = 8.3 Hz, 2H), 7.14 (d, *J* = 8.1 Hz, 2H), 2.34 (s, 3H), 2.18 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  168.6, 135.4, 133.9, 129.4, 120.1, 24.4, 20.8.

N-(4-bromophenyl)acetamide (3k); The title compound was prepared according to the general procedure described above using acetaldehyde (377 μl, 8.57 mmol), *p*-bromoaniline (100 mg, 0.57 mmol), Bu<sub>4</sub>NI (42 mg, 0.11 mmol) and TBHP (154 μl, 1.71 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (73 mg, 59% from amine). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>5</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.47 – 7.35 (m, 4H), 2.17 (s, 3H).

N-(4-(trifluoromethoxy)phenyl)acetamide (3l); The title compound was prepared according to the general procedure described above using



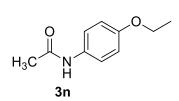
3j



acetaldehyde (369 µl, 8.4 mmol), *p*-(trifluoromethoxy)aniline (100 µl, 0.56 mmol), Bu<sub>4</sub>NI (41 mg, 0.11 mmol) and TBHP (151 µl, 1.68 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (69 mg, 56% from amine). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.99 (s, 1H), 7.52 (d, *J* = 8.9 Hz, 2H), 7.13 (d, *J* = 8.7 Hz, 2H), 2.16 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  168.7, 145.3, 136.5, 121.7, 121.5, 121.1, 119.4, 24.4; HRMS (TOF) m/z [M + H]<sup>+</sup> Calcd for C<sub>9</sub>H<sub>9</sub>F<sub>3</sub>NO<sub>2</sub> 220.0580 found 220.0590.

H<sub>3</sub>C N H 3m **N-(4-hydroxyphenyl)acetamide (3m);** The title compound was prepared according to the general procedure described above using acetaldehyde (605 µl, 13.76 mmol), 4-aminophenol (100 mg, 0.91 mmol), Bu<sub>4</sub>NI (67 mg, 0.18 mmol) and TBHP (245 µl, 2.73 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (83 mg, 60% from amine). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>5 1</sup>H NMR (400 MHz, MeOD)  $\delta$  7.32 (d, *J* = 8.8 Hz, 2H), 6.74 (d, *J* = 8.8 Hz, 2H), 4.91 (s, 1H), 2.10 (s, 3H); <sup>13</sup>C NMR (125 MHz, MeOD)  $\delta$  171.4, 155.4, 131.7, 123.3, 116.18, 23.5.

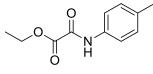
**N-(4-ethoxyphenyl)acetamide (3n);** The title compound was prepared according to the general procedure described above using acetaldehyde (481 µl, 10.94 mmol), 4-ethoxyaniline (100 µl, 0.72 mmol), Bu<sub>4</sub>NI (53 mg, 0.14 mmol) and TBHP (194 µl, 2.16 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (84 mg, 65% from amine). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>5</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.48 (s, 1H), 7.37 (d, *J* = 8.9 Hz, 2H), 6.83 (d,



*J* = 8.7 Hz, 2H), 4.00 (q, *J* = 7.0 Hz, 2H), 2.13 (s, 3H), 1.39 (t, *J* = 7.0 Hz, 3H).

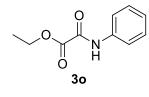
Ethyl 2-oxo-2-(phenylamino)acetate (30); The title compound was prepared according to the general procedure described above using ethyl 2-oxoacetate (100 µl, 0.98 mmol), aniline (91 µl, 0.98 mmol), Bu4NI (72 mg, 0.19 mmol) and TBHP (264 µl, 2.94 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (151 mg, 80% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>6</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.88 (s, 1H), 7.64 (d, *J* = 7.8 Hz, 2H), 7.38 (t, *J* = 7.9 Hz, 2H), 7.19 (t, *J* = 7.4 Hz, 1H), 4.42 (q, *J* = 7.1 Hz, 2H), 1.43 (t, *J* = 7.1 Hz, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  161.0, 153.9, 136.3, 129.3, 125.5, 119.8, 63.8, 14.0.

**Ethyl 2-((4-bromophenyl)amino)-2-oxoacetate (3p);** The title compound was prepared according to the general procedure described above using ethyl 2-oxoacetate (100 µl, 0.98 mmol), *p*-bromoaniline (166 mg, 0.98 mmol), Bu<sub>4</sub>NI (72 mg, 0.19 mmol) and TBHP (264 µl, 2.94 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (203 mg, 77% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>6</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.90 (s, 1H), 7.55 (d, *J* = 8.9 Hz, 2H), 7.49 (d, *J* = 8.8 Hz, 2H), 4.42 (q, *J* = 7.1 Hz, 2H), 1.43 (t, *J* = 7.1 Hz, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  160.8, 153.9, 135.4, 132.2, 121.3, 118.3, 63.9, 14.0.



3p

**Ethyl 2-oxo-2-(p-tolylamino)acetate (3q);** The title compound was prepared according to the general procedure described above using ethyl 2-oxoacetate (100  $\mu$ l, 0.98 mmol), *p*-toluidine (104 mg, 0.98 mmol), Bu<sub>4</sub>NI (72 mg, 0.19 mmol) and TBHP (264  $\mu$ l, 2.94 mmol) in CH<sub>3</sub>CN (2 ml) and

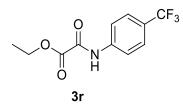


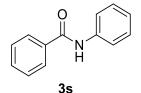
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reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (168 mg, 83% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>7</sup> <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.86 (s, 1H), 7.53 (d, *J* = 8.4 Hz, 2H), 7.18 (d, *J* = 8.4 Hz, 2H), 4.42 (q, *J* = 7.2 Hz, 2H), 2.35 (s, 3H), 1.44 (t, *J* = 7.1 Hz, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  161.1, 153.7, 135.3, 133.8, 129.7, 119.8, 63.7, 20.9, 14.0.

Ethyl 2-oxo-2-((4-(trifluoromethyl)phenyl)amino)acetate (3r); The title compound was prepared according to the general procedure described ethyl 2-oxoacetate above using (100)μl, 0.98 mmol). 4-(trifluoromethyl)aniline (157 µl, 0.98 mmol), Bu<sub>4</sub>NI (72 mg, 0.19 mmol) and TBHP (264 µl, 2.94 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (184 mg, 72% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>6</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  9.02 (s, 1H), 7.78 (d, J = 8.5 Hz, 2H), 7.64 (d, J = 8.6 Hz, 2H), 4.43 (q, J = 7.1 Hz, 2H), 1.43 (t, J = 7.1 Hz, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  162.0, 155.5, 140.7, 128.8, 128.6, 127.92 (d, J = 3.7 Hz), 121.0, 65.4, 15.3.

**N-Phenylbenzamide (3s);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), aniline (87 µl, 0.94 mmol), Bu<sub>4</sub>NI (69 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (137 mg, 74% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>8</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.87 (d, *J* = 7.8 Hz, 2H), 7.84 (s, 1H), 7.65 (d, *J* = 7.8 Hz, 2H), 7.56 (t, *J* = 7.3

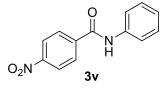




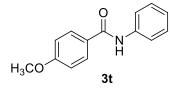
Hz, 1H), 7.49 (t, *J* = 7.3 Hz, 2H), 7.38 (t, *J* = 7.9 Hz, 2H), 7.16 (t, *J* = 7.4 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 165.8, 137.9, 134.9, 131.9, 129.1, 128.8, 127.1, 124.6, 120.2.

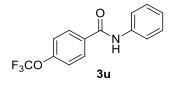
**4-Methoxy-N-phenylbenzamide (3t);** The title compound was prepared according to the general procedure described above using 4-methoxybenzaldehyde (100 µl, 0.73 mmol), aniline (68 µl, 0.73 mmol), Bu<sub>4</sub>NI (54 mg, 0.14 mmol) and TBHP (197 µl, 2.19 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (131 mg, 79% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>8</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.84-7.80(m, 3H), 7.63 (d, *J* = 7.7 Hz, 2H), 7.36 (t, *J* = 7.9 Hz, 2H), 7.14 (t, *J* = 7.4 Hz, 1H), 6.97 (d, *J* = 8.8 Hz, 2H), 3.87 (s, 3H).

**N-Phenyl-4-(trifluoromethoxy)benzamide (3u);** The title compound was prepared according to the general procedure described above using 4-(trifluoromethoxy)benzaldehyde (100 µl, 0.52 mmol), aniline (48 µl, 0.52 mmol), Bu<sub>4</sub>NI (38 mg, 0.10 mmol) and TBHP (140 µl, 1.56 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (93 mg, 63% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>9 1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.95 (t, *J* = 7.7 Hz, 2H), 7.91 (s, 1H), 7.67 (d, *J* = 7.9 Hz, 2H), 7.42 (t, *J* = 7.9 Hz, 2H), 7.34 (t, *J* = 11.0 Hz, 2H), 7.22 (t, *J* = 7.4 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  164.5, 151.7, 137.6, 133.4, 129.2, 129.0, 124.9, 120.8, 120.3.



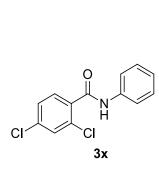
**4-Nitro-N-phenylbenzamide (3v);** The title compound was prepared according to the general procedure described above using 4-nitrobenzaldehyde (100 mg, 0.66 mmol), aniline (61  $\mu$ l, 0.66 mmol), Bu<sub>4</sub>NI (48 mg, 0.13 mmol) and TBHP (178  $\mu$ l, 1.98 mmol) in CH<sub>3</sub>CN (2 ml) and





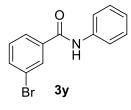
reaction mixture was heated at 80 °C for 10 h and the product formation was monitored by TLC, purified by column chromatography as white solid (72 mg, 45% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>8</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.36 (d, *J* = 8.4 Hz, 2H), 8.05 (d, *J* = 8.6 Hz, 2H), 7.81 (s, 1H), 7.64 (d, *J* = 7.7 Hz, 2H), 7.41 (t, *J* = 7.8 Hz, 2H), 7.21 (t, *J* = 7.4 Hz, 1H).

**4-Fluoro-N-phenylbenzamide (3w);** The title compound was prepared according to the general procedure described above using 4-fluorobenzaldehyde (100 µl, 0.80 mmol), aniline (75 µl, 0.80 mmol), Bu<sub>4</sub>NI (59 mg, 0.16 mmol) and TBHP (216 µl, 2.4 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (123 mg, 71% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>8</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.89 (m, 2H), 7.77 (s, 1H), 7.62 (d, *J* = 7.8 Hz, 2H), 7.38 (t, *J* = 7.9 Hz, 2H), 7.23 – 7.10 (m, 3H).



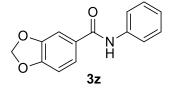
3w

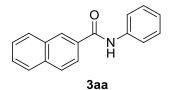
2,4-Dichloro-N-phenylbenzamide (3x); The title compound was prepared according to the general procedure described above using 2,4dichlorobenzaldehyde (100 mg, 0.57 mmol), aniline (53 µl, 0.57 mmol), Bu<sub>4</sub>NI (42 mg, 0.11 mmol) and TBHP (153 µl, 1.71 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (102 mg, 67% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>10 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.97 (s, 1H), 7.69 (d, J = 8.3 Hz, 1H), 7.62 (d, *J* = 7.9 Hz, 2H), 7.46 (d, *J* = 1.4 Hz, 1H), 7.37 (dd, *J* = 15.9, 7.9 Hz, 3H), 7.18 (t, J = 7.4 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  163.5, 137.28 (d, J = 17.2 Hz), 133.5, 131.43 (d, J = 15.1 Hz), 130.2, 129.2, 127.7, 125.1, 120.2.



**3-Bromo-N-phenylbenzamide (3y);** The title compound was prepared according to the general procedure described above using 3-bromobenzaldehyde (100 µl, 0.54 mmol), aniline (50 µl, 0.54 mmol), Bu<sub>4</sub>NI (40 mg, 0.10 mmol) and TBHP (146 µl, 1.62 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (109 mg, 73% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>10</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.01 (s, 1H), 7.79 (d, *J* = 7.4 Hz, 2H), 7.67 (t, *J* = 7.8 Hz, 1H), 7.63 (d, *J* = 7.9 Hz, 2H), 7.38 (dd, *J* = 14.1, 7.6 Hz, 3H), 7.18 (t, *J* = 7.4 Hz, 1H).

**N-Phenylbenzo[d][1,3]dioxole-5-carboxamide (3z);** The title compound was prepared according to the general procedure described above using benzo[d][1,3]dioxole-5-carbaldehyde (100 mg, 0.66 mmol), aniline (61 µl, 0.66 mmol), Bu<sub>4</sub>NI (48.7 mg, 0.13 mmol) and TBHP (178 µl, 1.98 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (120 mg, 75% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>11 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.78 (s, 1H), 7.61 (d, *J* = 7.7 Hz, 2H), 7.37 (dt, *J* = 15.8, 4.7 Hz, 4H), 7.14 (t, *J* = 7.4 Hz, 1H), 6.86 (d, *J* = 8.0 Hz, 1H), 6.05 (s, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  165.0, 150.7, 148.2, 137.9, 129.1, 124.5, 121.7, 120.2, 108.1, 107.7, 101.8.



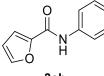


**N-Phenyl-2-naphthamide (3aa);** The title compound was prepared according to the general procedure described above using 2-naphthaldehyde (100 mg, 0.64 mmol), aniline (60  $\mu$ l, 0.64 mmol), Bu<sub>4</sub>NI (47.2 mg, 0.13 mmol) and TBHP (173  $\mu$ l, 1.92 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (109 mg, 69% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C

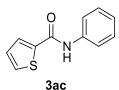
NMR) was consistent with that previously reported in the literature.<sup>8</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.39 (s, 1H), 8.04 – 7.85 (m, 5H), 7.70 (d, *J* = 7.8 Hz, 2H), 7.65 – 7.52 (m, 2H), 7.40 (t, *J* = 7.9 Hz, 2H), 7.18 (t, *J* = 7.4 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  163.6, 135.7, 132.6, 130.3, 129.9, 126.9, 126.7, 126.5, 125.62 (d, *J* = 12.6 Hz), 125.2, 124.7, 122.4, 121.3, 118.0.

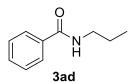
**N-Phenylfuran-2-carboxamide (3ab);** The title compound was prepared according to the general procedure described above using furan-2-carbaldehyde (100 µl, 1.04 mmol), aniline (96 µl, 1.04 mmol), Bu<sub>4</sub>NI (76 mg, 0.20 mmol) and TBHP (280 µl, 3.12 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (67 mg, 56% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>8 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.07 (s, 1H), 7.66 (d, *J* = 8.0 Hz, 2H), 7.54 – 7.49 (m, 1H), 7.37 (t, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 3.5 Hz, 1H), 7.15 (t, *J* = 7.4 Hz, 1H), 6.56 (dd, *J* = 3.5, 1.8 Hz, 1H).

**N-Phenylthiophene-2-carboxamide (3ac);** The title compound was prepared according to the general procedure described above using thiophene-2-carbaldehyde (100 µl, 0.89 mmol), aniline (83 µl, 0.89 mmol), Bu<sub>4</sub>NI (65.6 mg, 0.18 mmol) and TBHP (240 µl, 2.67 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (117 mg, 64% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>10 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.76 (s, 1H), 7.66 – 7.57 (m, 3H), 7.54 (d, *J* = 5.0 Hz, 1H), 7.36 (t, *J* = 7.9 Hz, 2H), 7.18 – 7.08 (m, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  160.1, 139.3, 137.6, 130.8, 129.1, 128.5, 127.8, 124.6, 120.3.



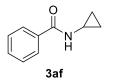
3ab



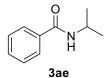


**N-Propylbenzamide (3ad);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), propan-1-amine (55 µl, 0.94 mmol.), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (90 mg, 59% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>12 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.77 (d, *J* = 7.5 Hz, 2H), 7.46 (t, *J* = 7.3 Hz, 1H), 7.38 (t, *J* = 7.4 Hz, 2H), 6.65 (s, 1H), 3.39 (dd, *J* = 13.5, 6.7 Hz, 2H), 1.60 (dt, *J* = 7.2, 4.9 Hz, 2H), 0.95 (t, *J* = 7.4 Hz, 3H).

**N-Isopropylbenzamide** (**3ae**); The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), propan-2-amine (55 µl, 0.94 mmol), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (98 mg, 64% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>12</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.76 (d, *J* = 7.5 Hz, 2H), 7.45 (t, *J* = 7.3 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 2H), 6.33 (s, 1H), 4.34 – 4.20 (m, 1H), 1.24 (d, *J* = 6.6 Hz, 6H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  166.7, 134.9, 131.3, 128.5, 126.8, 41.9, 22.9.



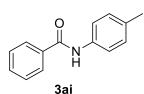
**N-Cyclopropylbenzamide** (**3af**); The title compound was prepared according to the general procedure described above using benzaldehyde (100  $\mu$ l, 0.94 mmol), cyclopropanamine (57  $\mu$ l, 0.94 mmol), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and TBHP (254  $\mu$ l, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (104 mg, 69% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>13</sup> <sup>1</sup>H



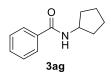
NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.74 (d, *J* = 7.3 Hz, 2H), 7.48 (dd, *J* = 10.4, 4.3 Hz, 1H), 7.40 (t, *J* = 7.4 Hz, 2H), 6.38 (s, 1H), 2.90 (ddt, *J* = 10.7, 7.0, 3.7 Hz, 1H), 0.91 – 0.76 (m, 2H), 0.68 – 0.53 (m, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  168.9, 134.4, 131.5, 128.5, 126.8, 23.1, 6.8.

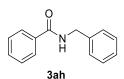
**N-Cyclopentylbenzamide** (**3ag**); The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), cyclopentanamine (80 µl, 0.94 mmol), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (115 mg, 65% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>12</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.75 (d, *J* = 7.5 Hz, 2H), 7.47 (t, *J* = 7.3 Hz, 1H), 7.40 (t, *J* = 7.3 Hz, 2H), 6.18 (s, 1H), 4.45 – 4.34 (m, 1H), 2.08 (dq, *J* = 12.0, 6.0 Hz, 2H), 1.67 (ddt, *J* = 11.1, 9.1, 5.9 Hz, 4H), 1.53 – 1.45 (m, 2H).

**N-Benzylbenzamide (3ah);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), phenylmethanamine (100 µl, 0.94 mmol), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (111 mg, 56% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>8</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.76 (d, *J* = 7.3 Hz, 2H), 7.44 (t, *J* = 7.4 Hz, 1H), 7.35 (t, *J* = 7.6 Hz, 2H), 7.31 – 7.22 (m, 5H), 6.95 (s, 1H), 4.55 (d, *J* = 5.8 Hz, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  165.8, 136.6, 132.6, 129.8, 127.0, 126.8, 126.1, 125.8, 125.4, 42.3.



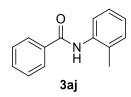
**N-(p-tolyl)benzamide (3ai);** The title compound was prepared according to the general procedure described above using benzaldehyde (100  $\mu$ l, 0.94 mmol), p-toluidine (100 mg, 0.94 mmol), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and

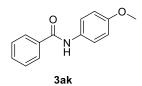




TBHP (254 µl, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (151 mg, 76% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>8</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.85 (dd, *J* = 7.9, 6.7 Hz, 3H), 7.51 (ddt, *J* = 21.8, 14.7, 7.2 Hz, 5H), 7.17 (d, *J* = 8.2 Hz, 2H), 2.34 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  165.1 134.8, 134.5, 133.7, 131.2, 129.0, 128.2, 126.4, 119.7, 20.4.

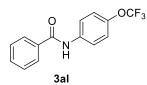
**N-(o-tolyl)benzamide (3aj);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), o-toluidine (100 µl, 0.94 mmol), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (49.3 mg, 71% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>8</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.93 (d, *J* = 7.8 Hz, 1H), 7.88 (d, *J* = 7.2 Hz, 2H), 7.71 (s, 1H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.49 (t, *J* = 7.4 Hz, 2H), 7.26 – 7.20 (m, 2H), 7.12 (t, *J* = 7.1 Hz, 1H), 2.33 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  165.7, 135.8, 135.0, 131.8, 130.6, 128.8, 127.1, 126.9, 125.4, 123.2, 17.8.





**N-(4-methoxyphenyl)benzamide (3ak);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), 4-methoxyaniline (116 mg, 0.94 mmol), Bu<sub>4</sub>NI (69 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (169 mg, 79% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>8</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.86 (d, *J* = 8.3 Hz, 2H), 7.81 (s, 1H), 7.53 (dd, *J* = 8.0, 4.0 Hz, 3H), 7.47 (t, *J* = 7.4 Hz, 2H), 6.90 (d, *J* = 8.9

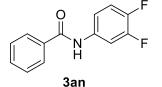
Hz, 2H), 3.81 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 165.6, 156.6, 135.0, 131.6, 131.0, 128.7, 126.9, 122.1, 114.2, 55.5.



 $CF_3$ 

**N-(4-(trifluoromethoxy)phenyl)benzamide (3al);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 μl, 0.94 mmol), 4-(trifluoromethyl)aniline (166 μl, 0.94 mmol), Bu<sub>4</sub>NI (69 mg, 0.18 mmol) and TBHP (254 μl, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (169 mg, 64% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>CNMR) was consistent with that previously reported in the literature.<sup>14</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.87 (d, *J* = 7.2 Hz, 2H), 7.68 (d, *J* = 9.0 Hz, 2H), 7.58 (t, *J* = 7.3 Hz, 1H), 7.51 (t, *J* = 7.4 Hz, 2H), 7.24 (d, *J* = 8.6 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 165.6, 156.6, 135.0, 131.6, 131.0, 128.7, 126.9, 122.1, 114.2, 55.5.

**N-(4-(trifluoromethyl)phenyl)benzamide (3am);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), 4-(trifluoromethyl)aniline (151µl, 0.94 mmol), Bu<sub>4</sub>NI (69 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (142 mg, 57% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>17 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.96 (s, 1H), 7.87 (d, *J* = 7.0 Hz, 2H), 7.79 (d, *J* = 8.5 Hz, 2H), 7.67 – 7.56 (m, 3H), 7.52 (t, *J* = 7.4 Hz, 2H).



N H

3am

**N-(3,4-difluorophenyl)benzamide (3an);** The title compound was prepared according to the general procedure described above using benzaldehyde (100  $\mu$ l, 0.94 mmol), 3,4-difluoroaniline (121  $\mu$ l, 0.94 mmol), Bu<sub>4</sub>NI (69 mg, 0.18 mmol) and TBHP (254  $\mu$ l, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product

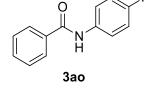
formation was monitored by TLC, purified by column chromatography as white solid (134 mg, 61% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>18</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.88 – 7.83 (m, 2H), 7.76 (ddd, J = 12.1, 7.1, 2.4 Hz, 1H), 7.58 (t, J = 7.3 Hz, 1H), 7.50 (t, J = 7.4 Hz, 2H), 7.20 (t, J = 7.2 Hz, 1H), 7.17 – 7.08 (m, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  165.7, 134.3, 132.2, 128.9, 127.0, 117.32 (d, J = 18.3 Hz), 115.8, 110.08 (d, J = 22.0 Hz).

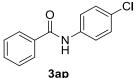
**N-(4-Fluorophenyl)benzamide** (**3ao**); The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), 4-fluoroaniline (104 µl, 0.94 mmol), Bu<sub>4</sub>NI (69 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 6 h and the product formation was monitored by TLC, purified by column chromatography as white solid (135 mg, 67% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>14</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.87 (d, *J* = 7.2 Hz, 2H), 7.81 (s, 1H), 7.63 – 7.54 (m, 3H), 7.50 (t, *J* = 7.4 Hz, 2H), 7.07 (t, *J* = 8.6 Hz, 2H).

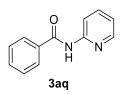
N-(4-Chlorophenyl)benzamide (3ap); The title compound was prepared

according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), 4-chloroaniline (119 mg, 0.94 mmol), Bu<sub>4</sub>NI (69 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 7 h and the product formation was monitored by TLC, purified by column chromatography as white solid (150 mg, 69% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>8 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.86 (d, *J* = 7.2 Hz, 2H), 7.82 (s, 1H), 7.65 – 7.52 (m, 3H), 7.50 (t, *J* = 7.4 Hz, 2H), 7.34 (d, *J* = 8.8 Hz, 2H).

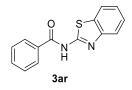
**N-(Pyridin-2-yl)benzamide** (**3aq**); The title compound was prepared according to the general procedure described above using benzaldehyde



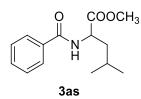




(100 µl, 0.94 mmol), pyridin-2-amine (88.3 mg, 0.94 mmol), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and TBHP (127 µl, 1.4 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (99 mg, 53% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>10</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  9.47 (s, 1H), 8.41 (d, *J* = 8.4 Hz, 1H), 8.06 (d, *J* = 2.3 Hz, 1H), 7.99 – 7.86 (m, 2H), 7.72 (td, *J* = 8.5, 1.7 Hz, 1H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 2H), 6.99 (dd, *J* = 6.7, 5.5 Hz, 1H).



**N-(Benzo[d]thiazol-2-yl)benzamide (3ar);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), benzo[d]thiazol-2-amine (141 mg, 0.94 mmol), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and TBHP (254 µl, 2.8 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (136 mg, 57% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>19</sup> <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.95 – 7.88 (m, 2H), 7.75 (d, *J* = 7.8 Hz, 1H), 7.48 – 7.41 (m, 1H), 7.05 (d, *J* = 8.0 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  166.2, 160.0, 147.6, 133.1, 132.1, 131.8, 129.0, 128.1, 126.1, 124.0, 121.4, 120.6.

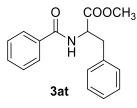


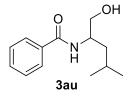
**Methyl 2-benzamido-4-methylpentanoate (3as);** The title compound was prepared according to the general procedure described above using benzaldehyde (100  $\mu$ l, 0.94 mmol), methyl 2-amino-4-methylpentanoate (136 mg, 0.94 mmol), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and TBHP (254  $\mu$ l, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (155 mg, 66% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously

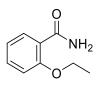
reported in the literature.<sup>15 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.78 (t, *J* = 13.6 Hz, 2H), 7.51 (t, *J* = 7.3 Hz, 1H), 7.43 (t, *J* = 7.4 Hz, 2H), 6.65 (s, 1H), 4.87 (td, *J* = 8.4, 5.1 Hz, 1H), 3.77 (s, 3H), 1.81 – 1.67 (m, 3H), 1.02 – 0.96 (m, 6H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  173.8, 167.2, 133.8, 131.7, 128.6, 127.1, 52.4, 51.1, 41.8, 24.9, 22.8, 22.0.

**Methyl 2-benzamido-3-phenylpropanoate (3at);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), methyl 2-amino-3-phenylpropanoate (168 mg, 0.94 mmol), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (165 mg, 62% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>16 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.76 – 7.66 (m, 2H), 7.48 (q, *J* = 7.7 Hz, 1H), 7.40 (t, *J* = 7.5 Hz, 2H), 7.31 – 7.22 (m, 3H), 7.17 – 7.10 (m, 2H), 6.69 (d, *J* = 5.6 Hz, 1H), 5.09 (dt, *J* = 7.4, 5.7 Hz, 1H), 3.74 (d, *J* = 10.1 Hz, 3H), 3.25 (qd, *J* = 13.8, 5.7 Hz, 2H).

**N-(1-Hydroxy-4-methylpentan-2-yl)benzamide** (3au); The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), 2-amino-4-methylpentan-1-ol (110 µl, 0.94 mmol), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (143 mg, 69% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>15</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.76 (d, *J* = 7.5 Hz, 2H), 7.48 (t, *J* = 7.3 Hz, 1H), 7.40 (t, *J* = 7.5 Hz, 2H), 6.42 (d, *J* = 6.9 Hz, 1H), 4.26 (dd, *J* = 8.4, 4.8 Hz, 1H), 3.70 (ddd, *J* = 16.3, 11.0, 4.3 Hz, 2H), 1.70 (tt, *J* = 13.0, 6.6 Hz, 1H), 1.57 – 1.37 (m, 2H), 0.96 (d, *J* = 6.5 Hz, 6H).

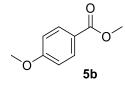






3av

0 0 5a



**2-Ethoxybenzamide (3av);** The title compound was prepared according to the general procedure described above using 2-ethoxybenzaldehyde (100  $\mu$ l, 0.66 mmol), ammonia (113  $\mu$ l, 6.6 mmol), Bu<sub>4</sub>NI (48 mg, 0.13 mmol) and TBHP (178  $\mu$ l, 1.98 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (68 mg, 62% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>20</sup> <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.08 – 7.93 (m, 1H), 7.45 (ddd, *J* = 9.2, 5.7, 1.8 Hz, 1H), 7.04 – 6.90 (m, 2H), 4.22 (qd, *J* = 7.0, 3.6 Hz, 2H), 1.45 (ddd, *J* = 9.3, 4.6, 2.2 Hz, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  165.8, 157.5, 135.1, 133.4, 121.9, 117.5, 112.7, 65.9, 14.5.

**Methyl benzoate** (**5a**); The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in CH<sub>3</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as liquid product (125 mg, 78% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>23</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.04 (dd, *J* = 8.2, 1.0 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 2H), 3.91 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  167.1, 132.9, 130.2, 129.6, 128.3, 52.1

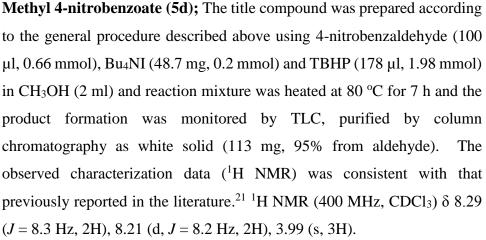
**Methyl 4-methoxybenzoate (5b);** The title compound was prepared according to the general procedure described above using 4-methoxybenzaldehyde (100  $\mu$ l, 0.73 mmol), Bu<sub>4</sub>NI (51.6 mg, 0.14 mmol) and TBHP (197  $\mu$ l, 2.19 mmol) in CH<sub>3</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (102 mg, 84% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>21</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

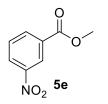
δ 7.98 (d, *J* = 8.9 Hz, 2H), 6.89 (d, *J* = 8.7 Hz, 2H), 3.86 (s, 3H), 3.82 (s, 3H).

**Methyl 3,4-dimethoxybenzoate (5c);** The title compound was prepared according to the general procedure described above using 3,4-dimethoxybenzaldehyde (100 µl, 0.60 mmol), Bu<sub>4</sub>NI (44.2 mg, 0.12 mmol) and TBHP (162 µl, 1.80 mmol) in CH<sub>3</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (95 mg, 81% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>21 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.67 (d, *J* = 8.4 Hz, 1H), 7.55 (t, *J* = 4.9 Hz, 1H), 6.88 (d, *J* = 8.4 Hz, 1H), 3.92 (s, 6H), 3.89 (s, 3H).

O in  $O_2N$  5d ch

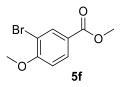
5c





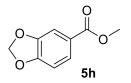
**Methyl 3-nitrobenzoate (5e);** The title compound was prepared according to the general procedure described above using 3-nitrobenzaldehyde (100  $\mu$ l, 0.66 mmol), Bu<sub>4</sub>NI (48.7 mg, 0.13 mmol) and TBHP (178  $\mu$ l, 1.98 mmol) in CH<sub>3</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 7 h and the product formation was monitored by TLC, purified by column chromatography as white solid (109 mg, 91% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>21</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.90 – 8.85 (m, 1H), 8.45 – 8.35 (m, 2H), 7.67 (t, *J* = 8.0 Hz, 1H), 4.00 (s, 3H);

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.9, 135.3, 131.8, 129.6, 127.4, 124.6, 52.8,



**Methyl 3-bromo-4-methoxybenzoate** (**5f**); The title compound was prepared according to the general procedure described above using 3-bromo-4-methoxybenzaldehyde (100 µl, 0.46 mmol), Bu<sub>4</sub>NI (37 mg, 0.1 mmol) and TBHP (124 µl, 1.38 mmol) in CH<sub>3</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (99 mg, 87% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>24</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.19 (t, *J* = 4.7 Hz, 1H), 7.94 (d, *J* = 8.6 Hz, 1H), 6.88 (dd, *J* = 10.0, 5.7 Hz, 1H), 3.92 (s, 3H), 3.87 (s, 3H).

**Methyl 4-chlorobenzoate** (**5g**); The title compound was prepared according to the general procedure described above using 4-chlorobenzaldehyde (100 µl, 0.71 mmol), Bu<sub>4</sub>NI (52.7 mg, 0.14 mmol) and TBHP (192 µl, 2.13 mmol) in CH<sub>3</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as liquid product (103 mg, 85% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>22</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.97 (d, *J* =8.8 Hz, 2H), 7.41 (d, *J* = 8.7 Hz, 2H), 3.91 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  165.2, 138.3, 129.9, 127.7, 127.5, 51.1



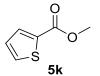
5g

**Methyl benzo[d][1,3]dioxole-5-carboxylate (5h);** The title compound was prepared according to the general procedure described above using benzo[d][1,3]dioxole-5-carbaldehyde (100  $\mu$ l, 0.66 mmol), Bu<sub>4</sub>NI (47.9 mg, 0.13 mmol) and TBHP (178  $\mu$ l, 1.98 mmol) in CH<sub>3</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (98 mg, 82% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>23</sup> <sup>1</sup>H NMR

(400 MHz, CDCl<sub>3</sub>) δ 7.65 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.46 (d, *J* = 1.5 Hz, 1H), 6.83 (d, *J* = 8.2 Hz, 1H), 6.03 (s, 2H), 3.88 (s, 3H).

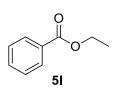
**Methyl 2-naphthoate (5i);** The title compound was prepared according to the general procedure described above using 2-naphthaldehyde (100 µl, 0.64 mmol), Bu<sub>4</sub>NI (47.3 mg, 0.12 mmol) and TBHP (173 µl, 1.92 mmol) in CH<sub>3</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (101 mg, 85% from aldehyde). The observed characterization data (<sup>1</sup>H & <sup>13</sup>C NMR) was consistent with that previously reported in the literature.<sup>21 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.61 (s, 1H), 8.07 (dd, *J* = 8.5, 1.3 Hz, 1H), 7.92 (d, *J* = 8.5 Hz, 1H), 7.86 (d, *J* = 8.7 Hz, 2H), 7.61 – 7.46 (m, 2H), 3.97 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  167.3, 135.5, 132.5, 131.1, 129.4, 128.22 (d, *J* = 10.4 Hz), 127.8, 127.4, 126.6, 125.2, 52.2.

**Methyl picolinate (5j);** The title compound was prepared according to the general procedure described above using picolinaldehyde (100 µl, 0.93 mmol), Bu<sub>4</sub>NI (68.6 mg, 0.18 mmol) and TBHP (251 µl, 2.79 mmol) in CH<sub>3</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as liquid product (94 mg, 74% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>22</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.76 (d, *J* = 3.8 Hz, 1H), 8.20 – 8.06 (m, 1H), 7.86 (tdd, *J* = 7.7, 4.0, 1.8 Hz, 1H), 7.56 – 7.43 (m, 1H), 4.01 (s, 3H).



**Methyl thiophene-2-carboxylate (5k)**; The title compound was prepared according to the general procedure described above using thiophene-2-carbaldehyde (100  $\mu$ l, 0.89 mmol), Bu<sub>4</sub>NI (62.73 mg, 0.17 mmol) and TBHP (240  $\mu$ l, 2.67 mmol) in CH<sub>3</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as liquid product (86 mg, 68% from

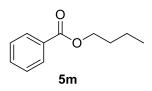
aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>26</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.79 (dd, *J* = 3.7, 1.2 Hz, 1H), 7.54 (dd, *J* = 5.0, 1.2 Hz, 1H), 7.08 (dt, *J* = 14.1, 7.0 Hz, 1H), 3.86 (s, 3H).



**Ethyl benzoate (51);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in C<sub>2</sub>H<sub>5</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as liquid product (87 mg, 62% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>25</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.09 – 7.99 (d, *J* = 8.2 Hz, 2H), 7.53 (t, *J* = 7.4 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 4.37 (q, *J* = 7.1 Hz, 2H), 1.37 (q, *J* = 7.5 Hz, 3H).

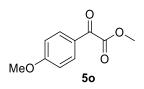
**Butyl benzoate (5m);** The title compound was prepared according to the general procedure described above using benzaldehyde (100 µl, 0.94 mmol), Bu<sub>4</sub>NI (66.4 mg, 0.18 mmol) and TBHP (254 µl, 2.82 mmol) in butanol (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as liquid product (90 mg, 54% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>25 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.04 (d, *J* = 8.2 Hz, 2H), 7.54 (dd, *J* = 10.5, 4.3 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 4.32 (t, *J* = 6.6 Hz, 2H), 1.80 – 1.67 (m, 2H), 1.55 – 1.39 (m, 2H), 0.98 (t, *J* = 7.4 Hz, 3H)

**Methyl 2-oxo-2-phenylacetate (5n);** The title compound was prepared according to the general procedure described above using 2-oxo-2-phenylacetaldehyde (100 mg, 0.74 mmol), Bu<sub>4</sub>NI (55.3 mg, 0.15 mmol) and TBHP (200  $\mu$ l, 2.22 mmol) in CH<sub>3</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC,





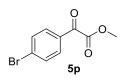
purified by column chromatography as white solid (68 mg, 56% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>27 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.06-7.99 (m, 2H), 7.67 (t, *J* = 7.4 Hz, 1H), 7.52 (t, *J* = 7.8 Hz, 2H), 3.99 (s, 3H).



Methyl 2-(4-methoxyphenyl)-2-oxoacetate (50); The title compound was prepared according to the general procedure described above using 2-(4-methoxyphenyl)-2-oxoacetaldehyde (100 µl, 0.61 mmol), Bu<sub>4</sub>NI (45 mg, 0.12 mmol) and TBHP (164 µl, 1.83 mmol) in CH<sub>3</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (74 mg, 63% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>27</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.01 (d, *J* = 8.0 Hz, 2H), 6.98 (d, *J* = 6.9 Hz, 2H), 3.96 (s, 3H), 3.90 (s, 3H).

Methyl 2-(4-bromophenyl)-2-oxoacetate (5p); The title compound was prepared according to the general procedure described above using 2-(4-bromophenyl)-2-oxoacetaldehyde (100 µl, 0.46 mmol), Bu<sub>4</sub>NI (34.6 mg, 0.1 mmol) and TBHP (124.2 µl, 1.38 mmol) in CH<sub>3</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as white solid (77 mg, 68% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>27</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.91 (d, *J* = 8.6 Hz, 2H), 7.66 (d, *J* = 8.7 Hz, 2H), 3.98 (s, 3H).

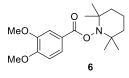
Methyl 2-(4-chlorophenyl)-2-oxoacetate (5q); The title compound was prepared according to the general procedure described above using 2-(4chlorophenyl)-2-oxoacetaldehyde (100  $\mu$ l, 0.59 mmol), Bu<sub>4</sub>NI (43.5 mg, 0.11 mmol) and TBHP (159  $\mu$ l, 1.77 mmol) in CH<sub>3</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was



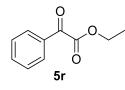
monitored by TLC, purified by column chromatography as white solid (77 mg, 65% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>27</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.87 (d, *J* = 8.6 Hz, 2H), 7.66 – 7.60 (d, *J* = 8.5 Hz, 2H), 3.94 (s, 3H).

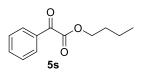
Ethyl 2-oxo-2-phenylacetate (5r); The title compound was prepared according to the general procedure described above using 2-oxo-2-phenylacetaldehyde (100 µl, 0.74 mmol), Bu<sub>4</sub>NI (55 mg, 0.15 mmol) and TBHP (200 µl, 2.22 mmol) in C<sub>2</sub>H<sub>5</sub>OH (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as solid product (66 mg, 50% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>27 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.04-7.98 (m, 2H), 7.66 (t, *J* = 7.4 Hz, 1H), 7.52 (t, *J* = 7.8 Hz, 2H), 4.46 (q, *J* = 7.1 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

**Butyl 2-oxo-2-phenylacetate** (5s); The title compound was prepared according to the general procedure described above using 2-oxo-2-phenylacetaldehyde (100 µl, 0.74 mmol), Bu<sub>4</sub>NI (55 mg, 0.15 mmol) and TBHP (200 µl, 2.22 mmol) in butanol (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as solid product (64 mg, 42% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>28 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.00 (d, *J* = 7.3 Hz, 2H), 7.65 (t, *J* = 7.4 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 2H), 4.39 (t, *J* = 6.7 Hz, 2H), 1.81 – 1.70 (m, 2H), 1.45 (dq, *J* = 14.7, 7.3 Hz, 2H), 0.96 (t, *J* = 7.4 Hz, 3H).



**2,2,6,6-Tetramethylpiperidin-1-yl 3,4-dimethoxybenzoate** (6): The title compound was prepared according to the general procedure described above using 3,4-dimethoxybenzaldehyde (100 mg, 0.60 mmol), aniline (56  $\mu$ l, 0.60 mmol), TEMPO (93.6 mg, 0.60 mmol) Bu<sub>4</sub>NI (44.2 mg, 0.12





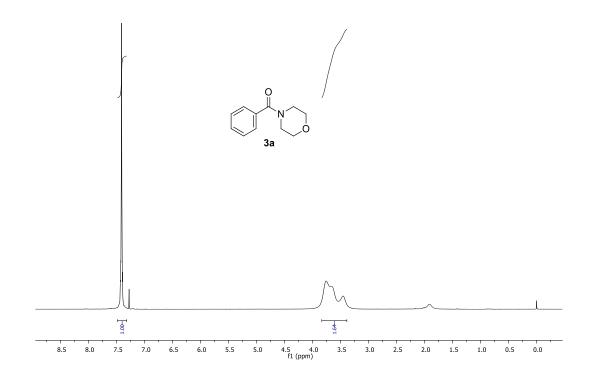
mmol) and TBHP (162 µl, 1.80 mmol) in CH<sub>3</sub>CN (2 ml) and reaction mixture was heated at 80 °C for 8 h and the product formation was monitored by TLC, purified by column chromatography as solid product (81 mg, 42% from aldehyde). The observed characterization data (<sup>1</sup>H NMR) was consistent with that previously reported in the literature.<sup>29</sup> <sup>1</sup>H NMR (400 MHz, CDCl3)  $\delta$  7.72 (dd, *J* = 8.4, 1.9 Hz, 2H), 7.59 (t, *J* = 4.4 Hz, 2H), 6.90 (*J* = 8.4 Hz, 1 H), 3.95 (s, 6H), 1.83 – 1.48 (m, 6H), 1.27 (s, 6H), 1.12 (s, 6H).

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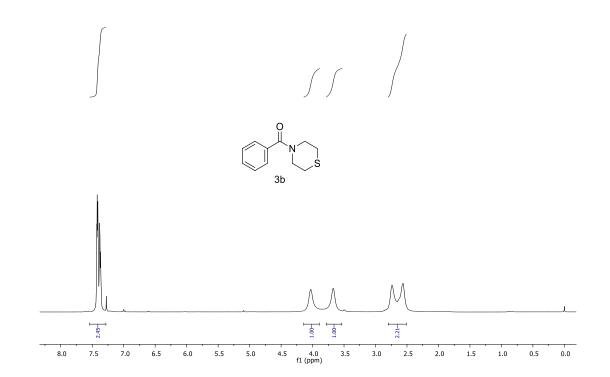
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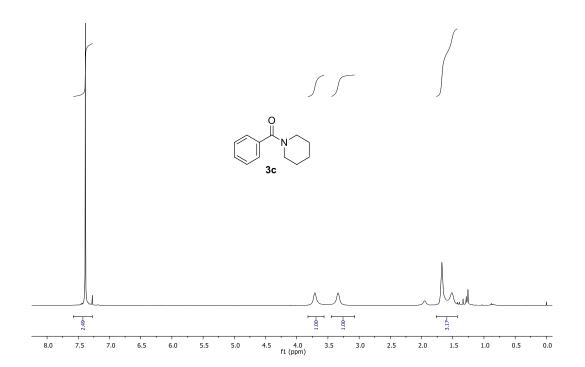
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**3a:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

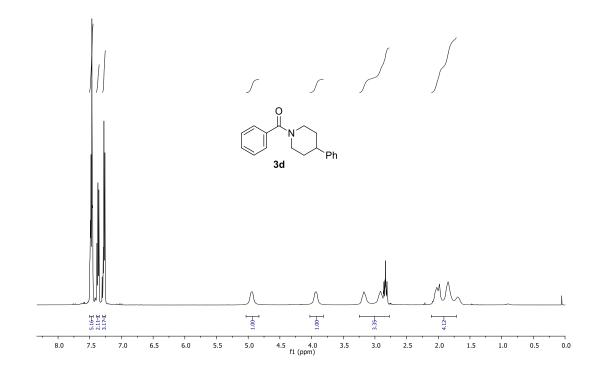


**3b:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

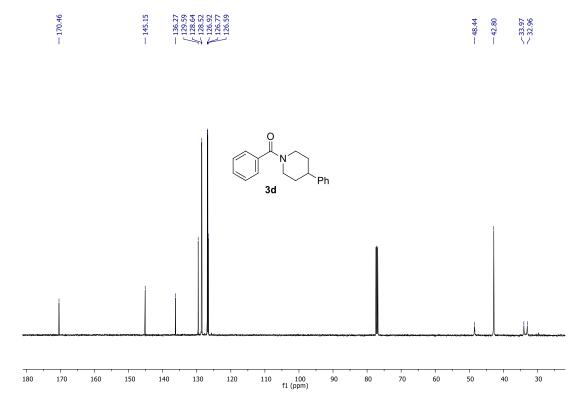




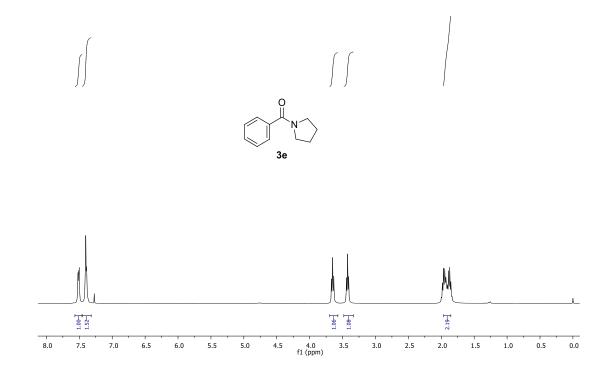
**3d:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)



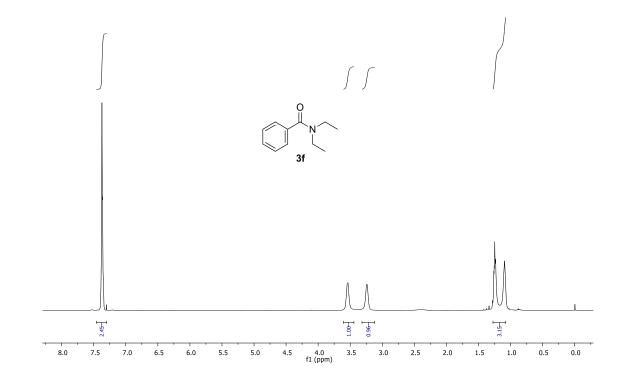
# **3d:** <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)



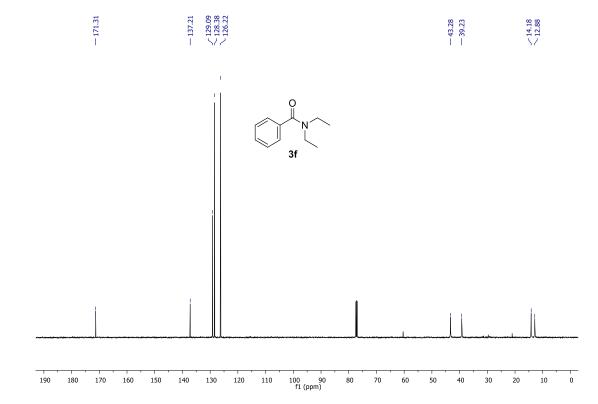
**3e:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

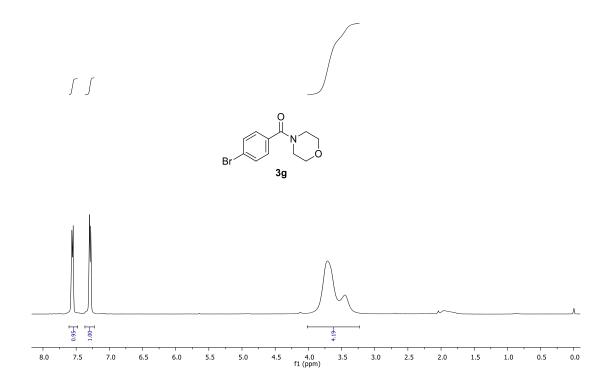


## **3f:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)

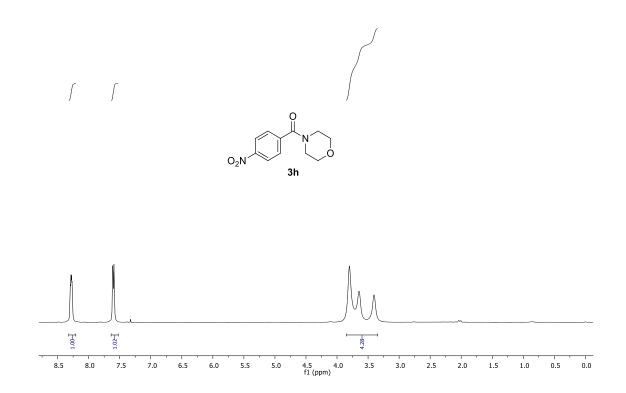


**3f:** <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

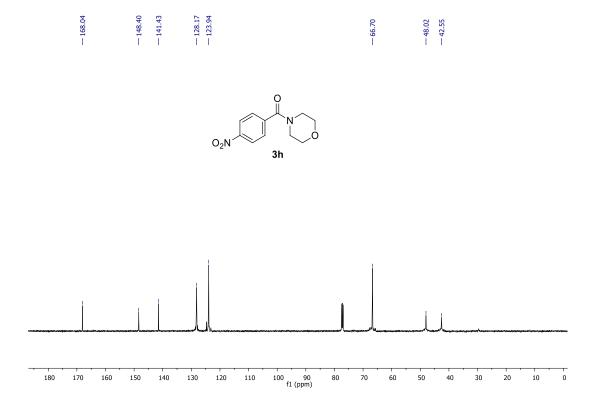




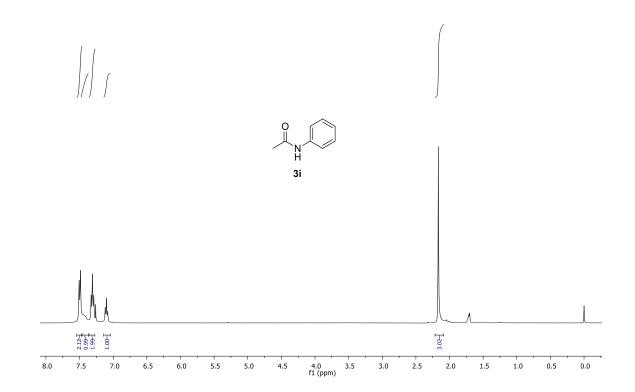
**3h:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



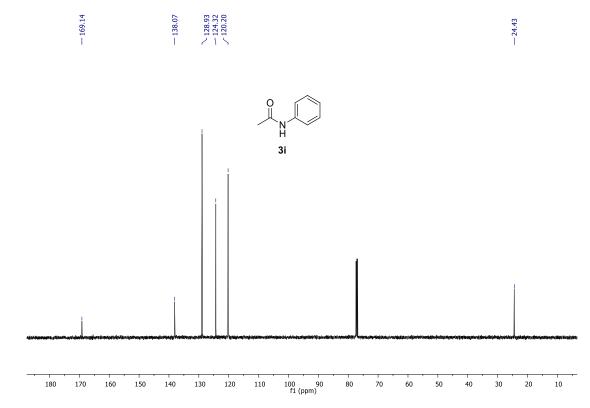
## 3h: <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)



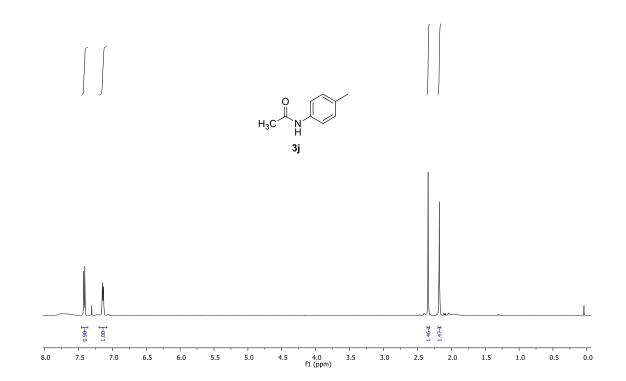
**3i:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



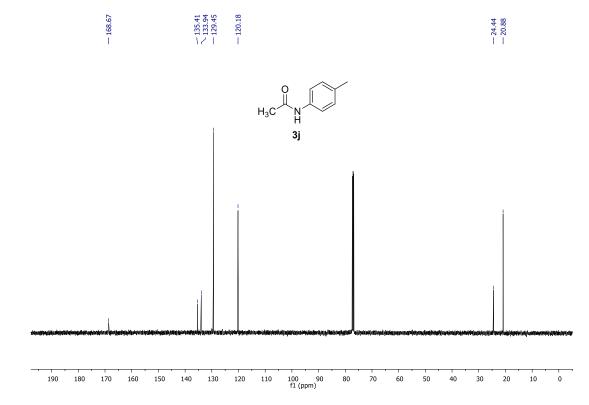
# **3i:** <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)



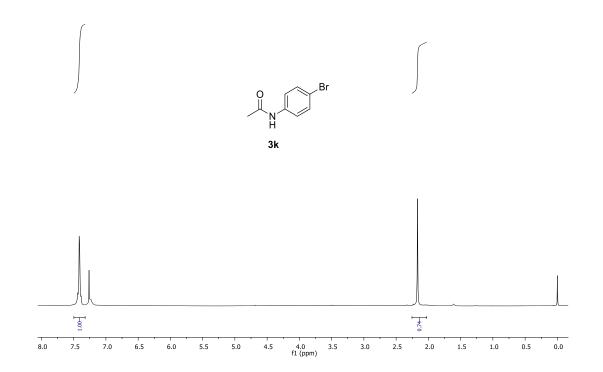
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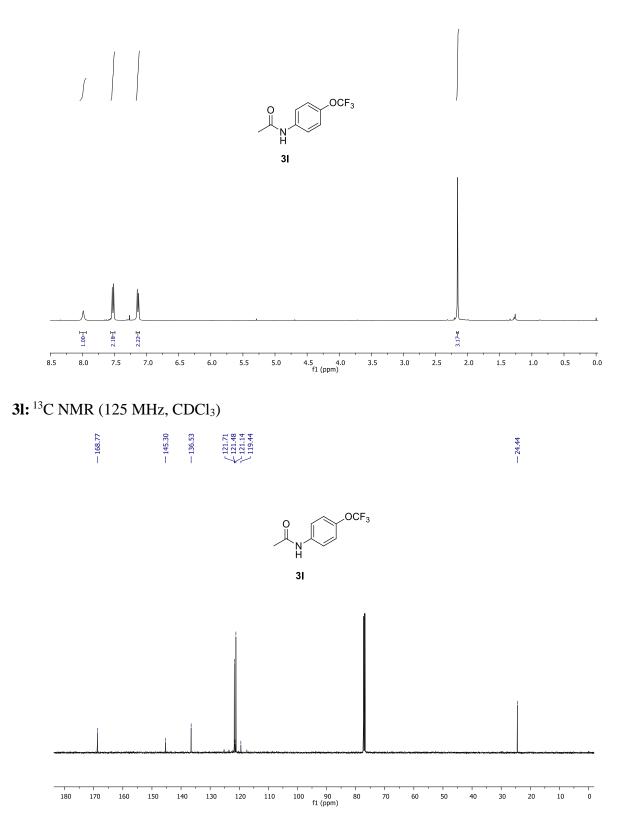


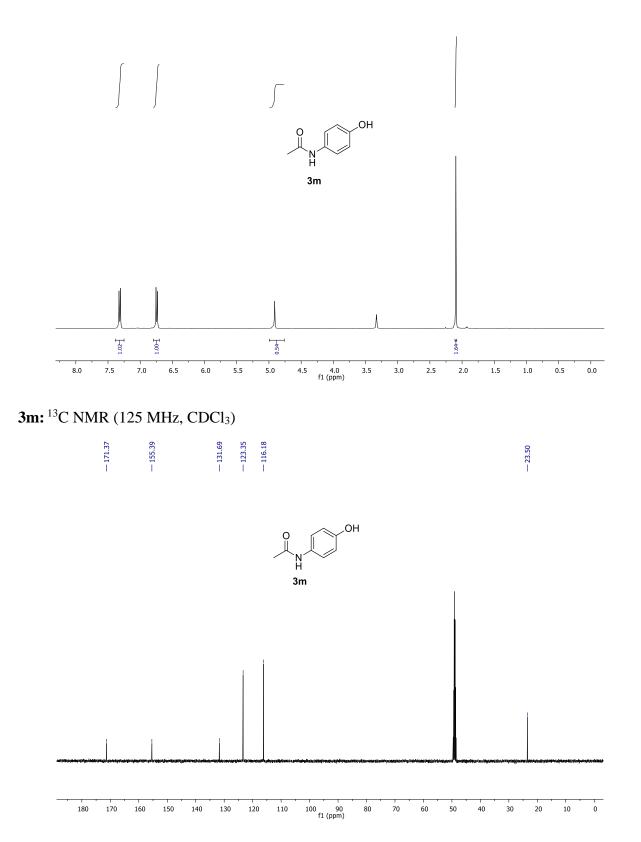
# **3j:** <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

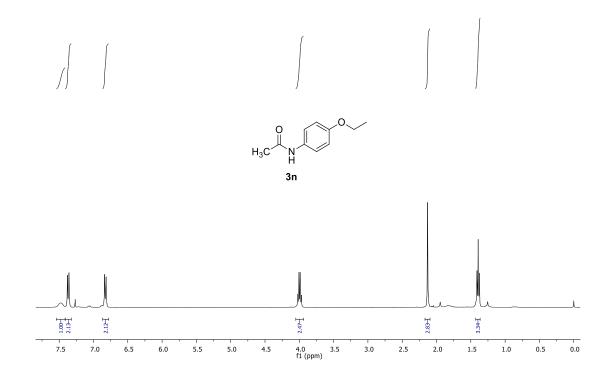


**3k:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

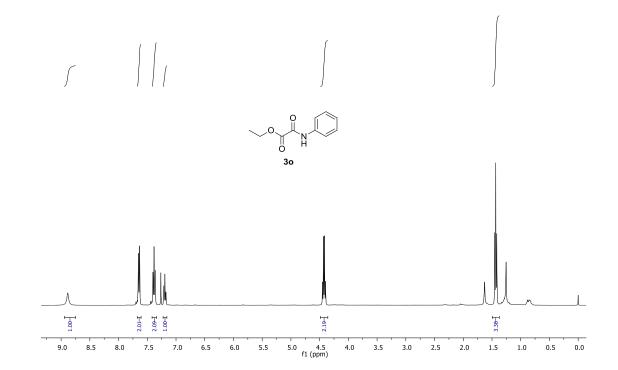




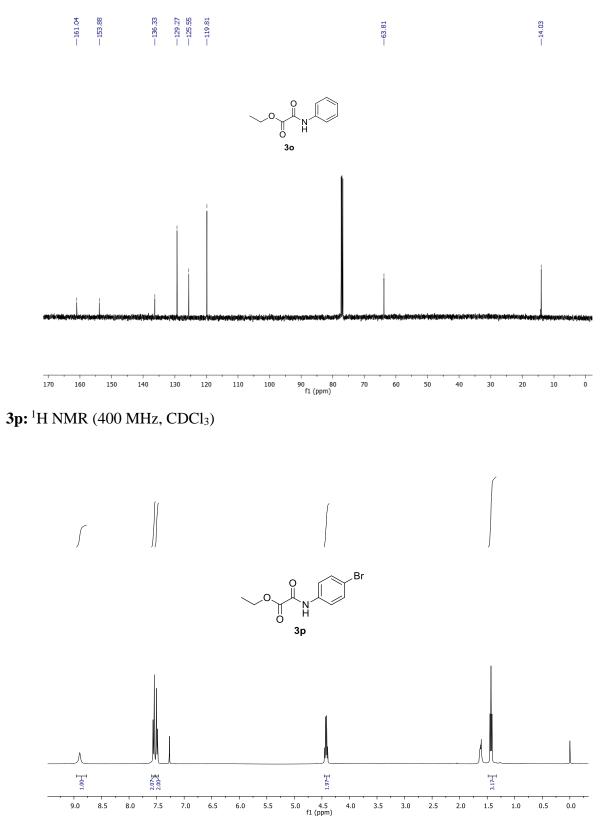




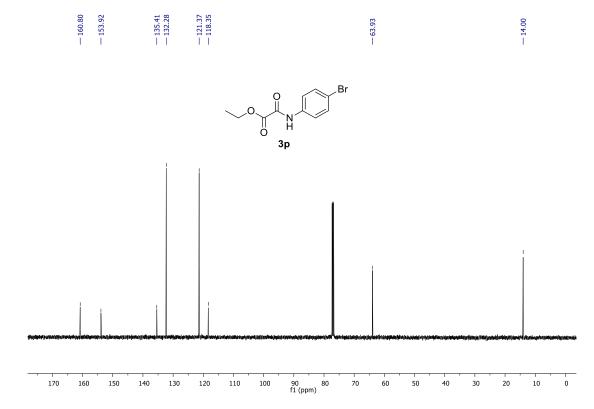
**30:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



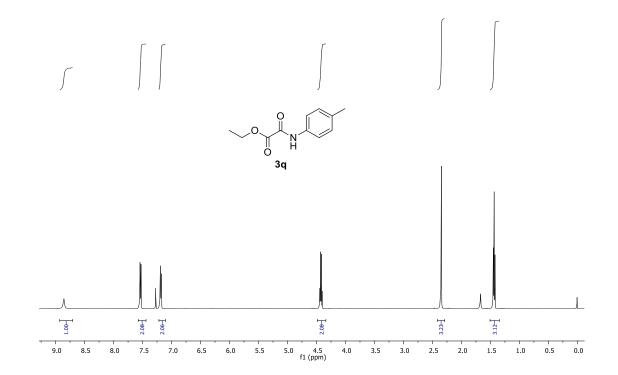
#### **30:** <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)



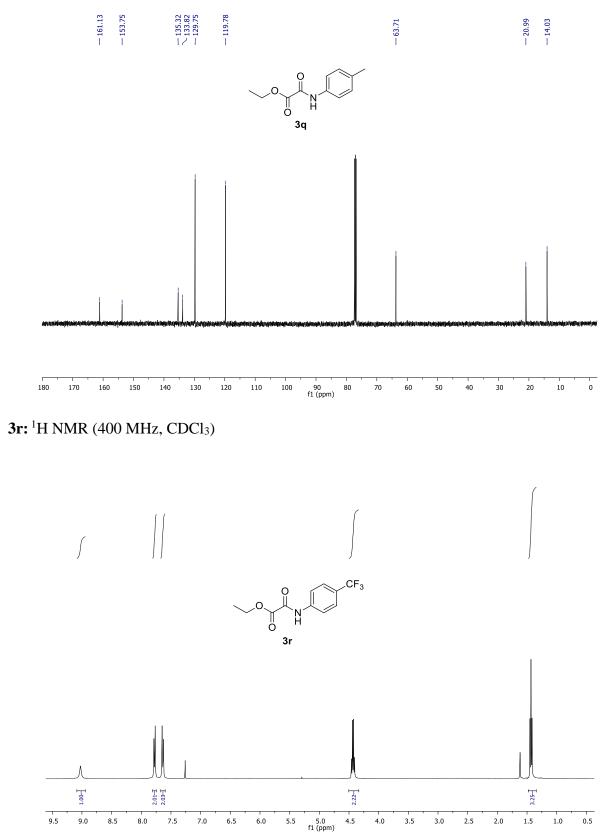
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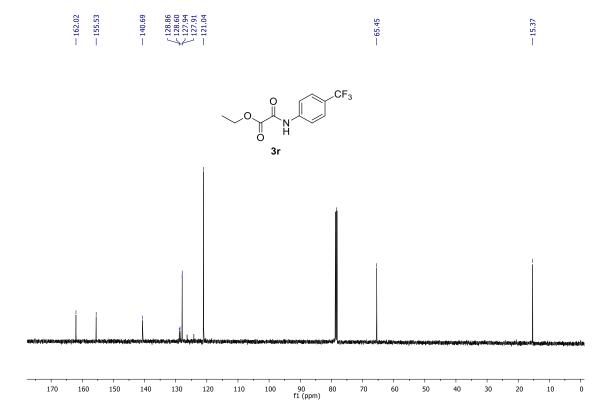
**3q:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)



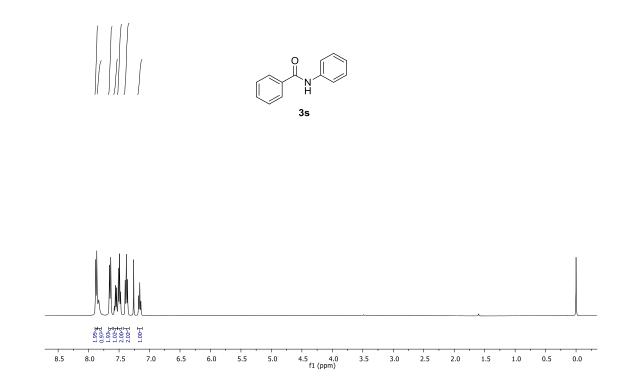
# **3q:** <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)



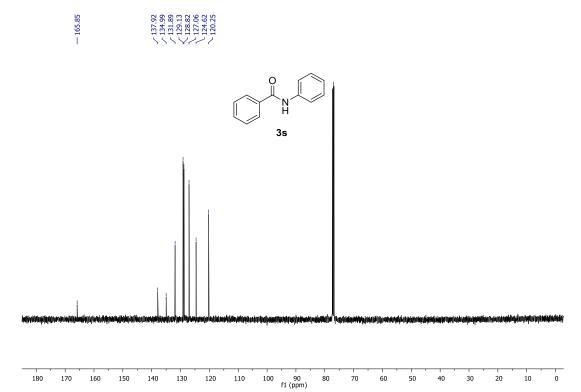
# **3r:** <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)



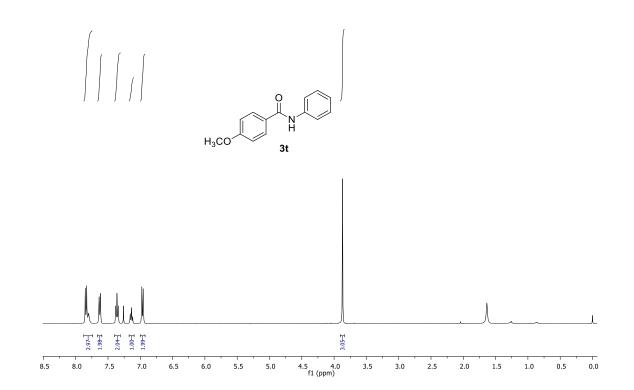
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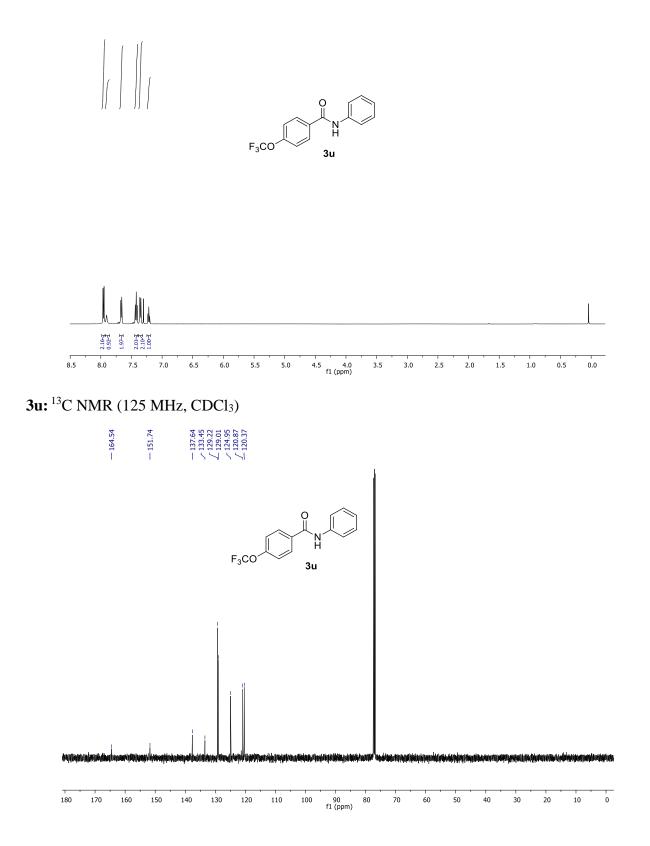
# **3s:** <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

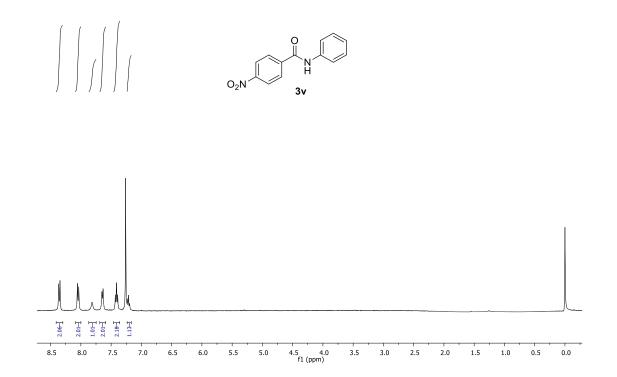


**3t:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

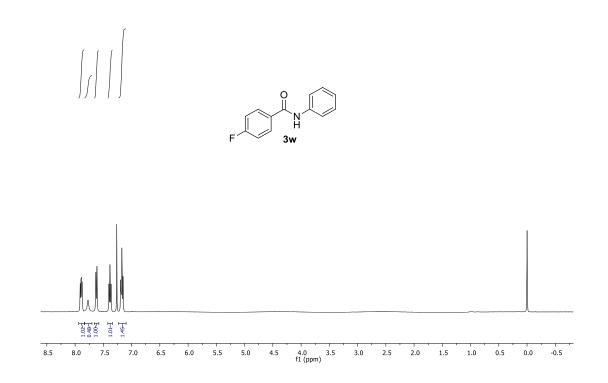


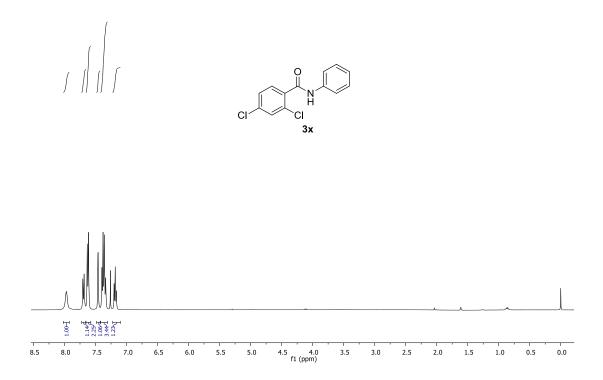
#### **3u:** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)





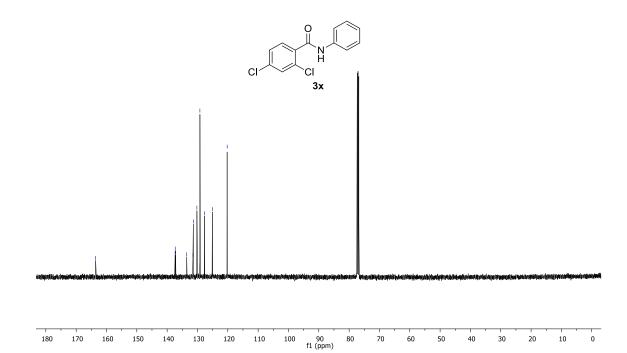


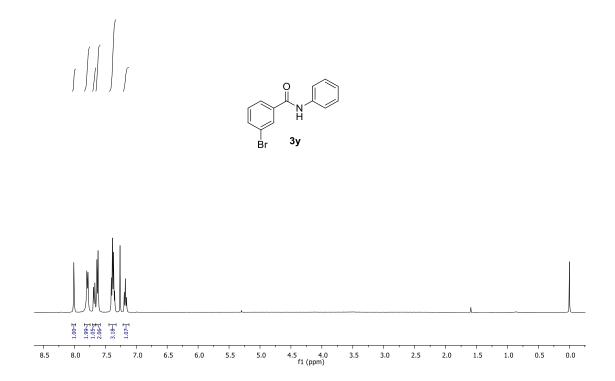




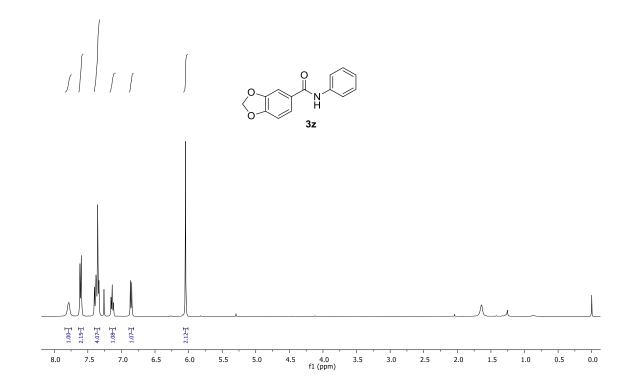
## **3x:** <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

- 163.57 - 163.57 - 137.24 - 137.24 - 131.49 - 131.49 - 131.49 - 131.49 - 131.49 - 131.49 - 131.49 - 120.19 - 120.19 - 120.20 - 1

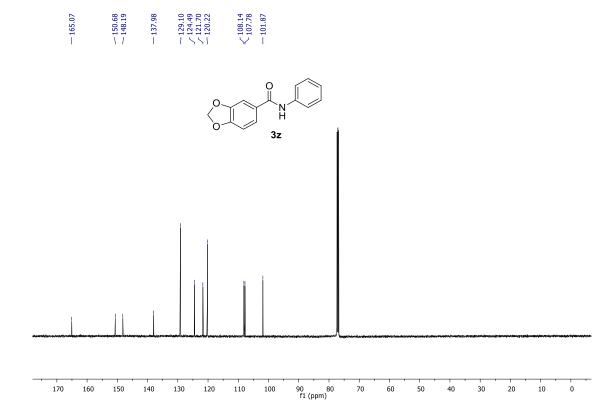




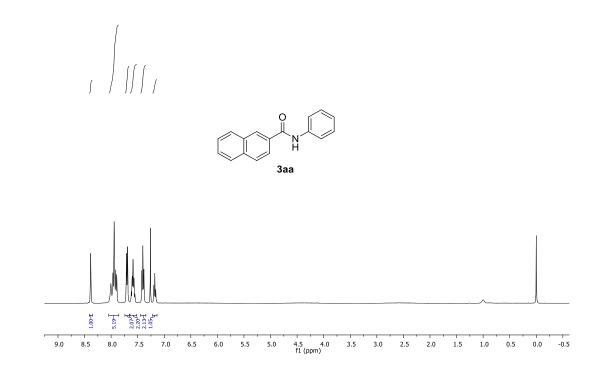
**3z:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



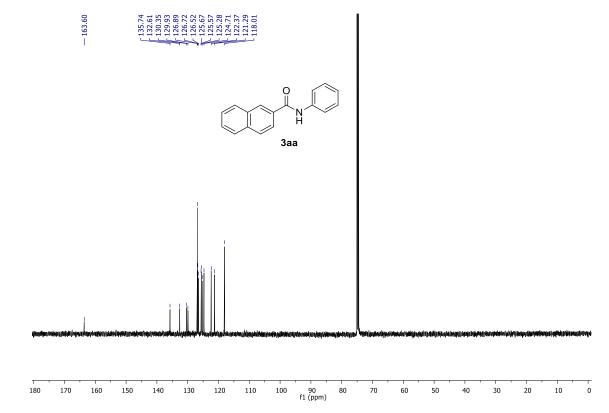
# **3z:** <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)



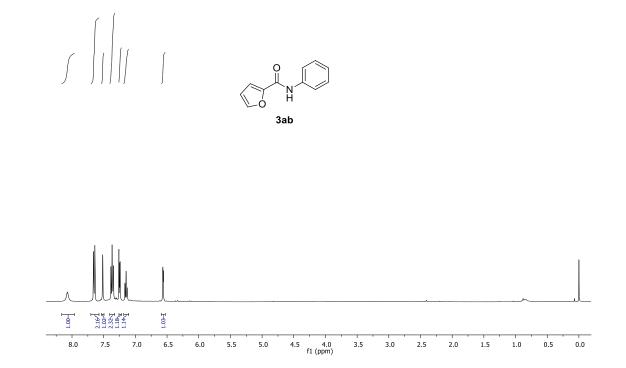
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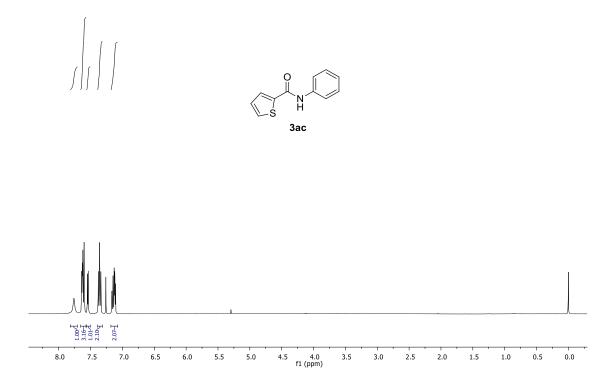


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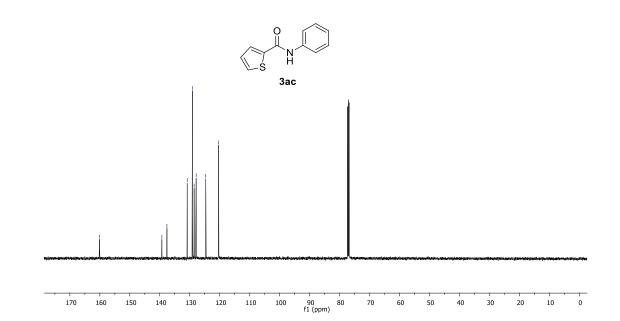


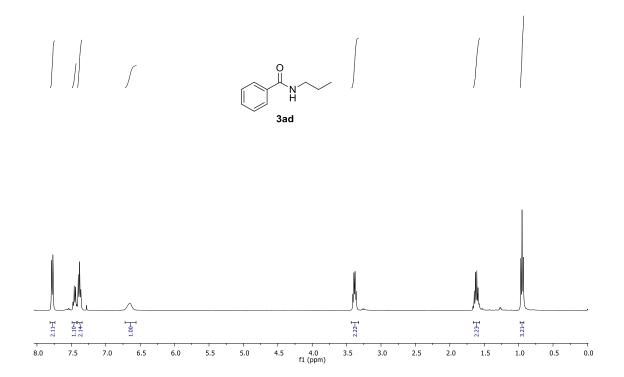
3ab: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



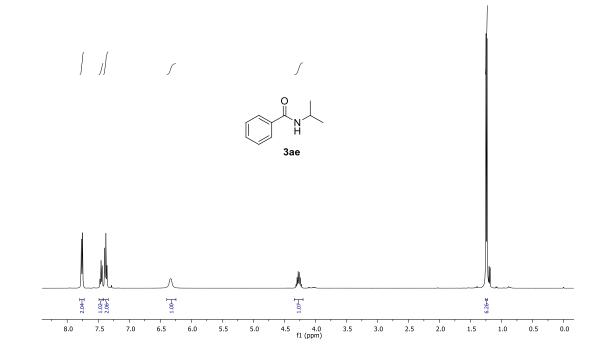


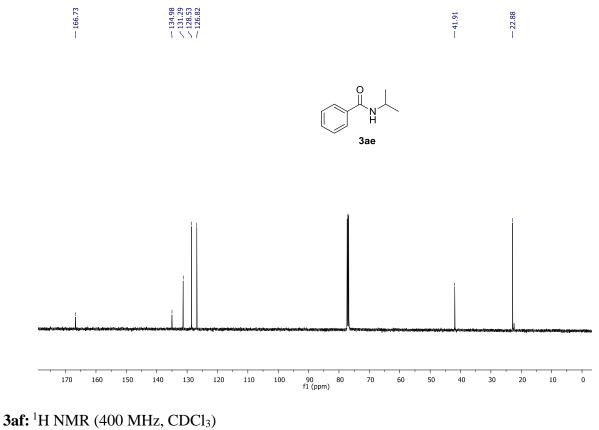
3ac: <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

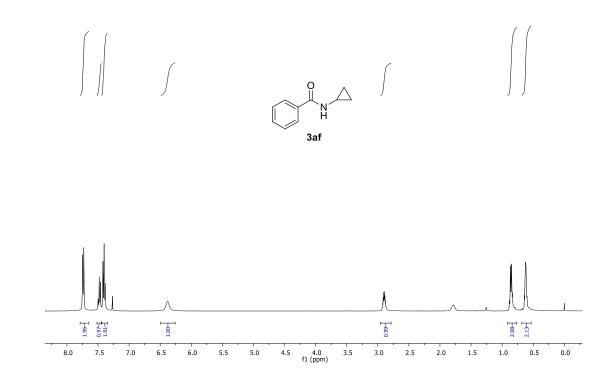


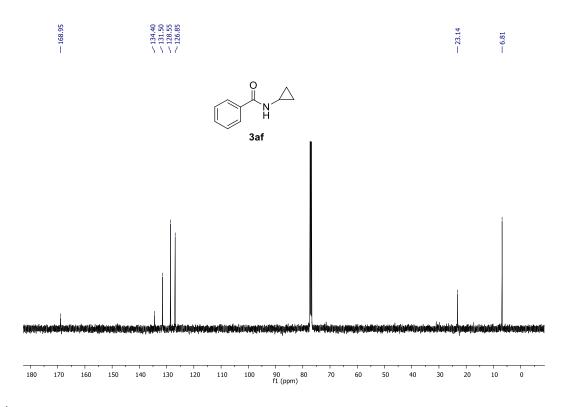


3ae: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

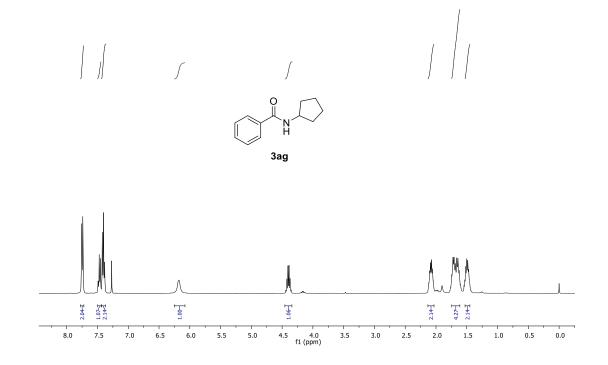


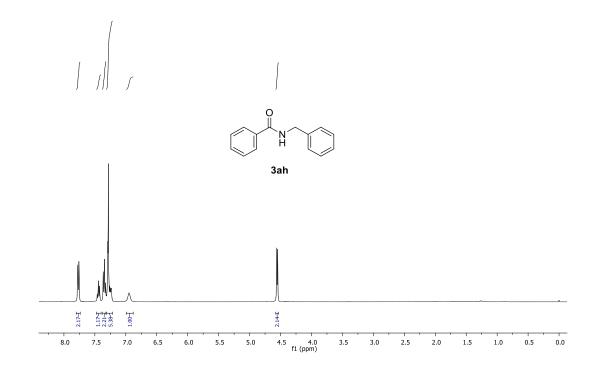


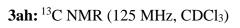


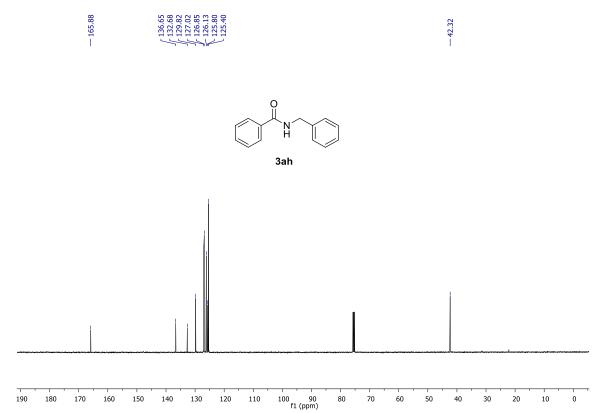


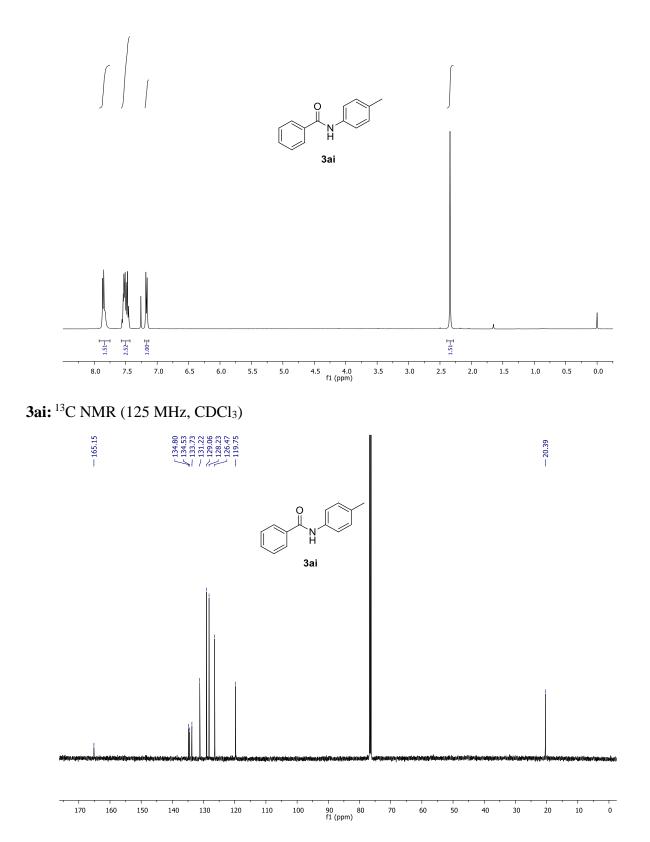
3ag: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

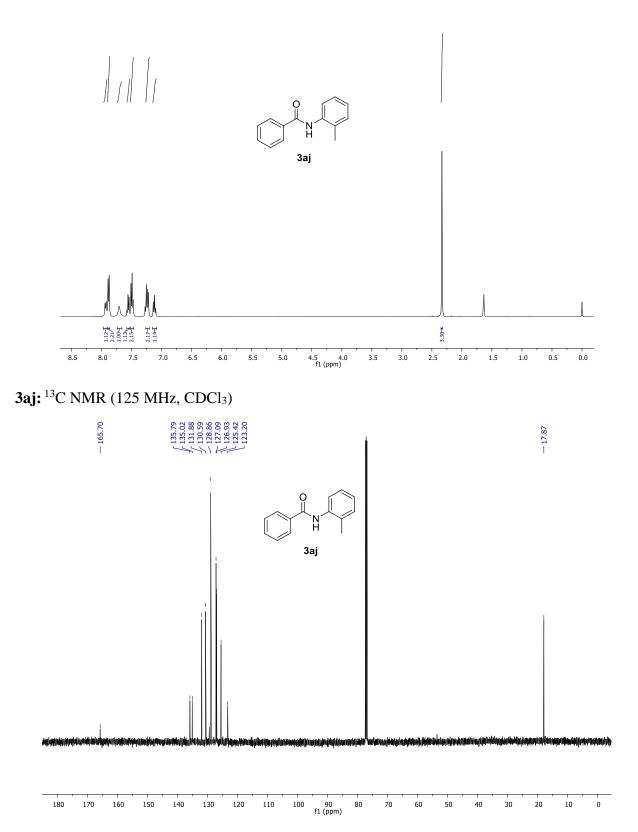


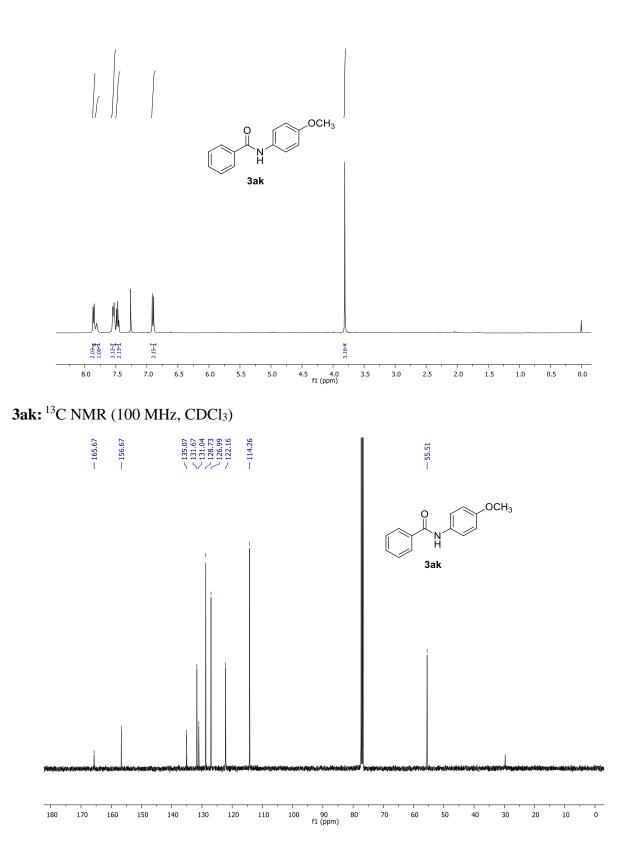


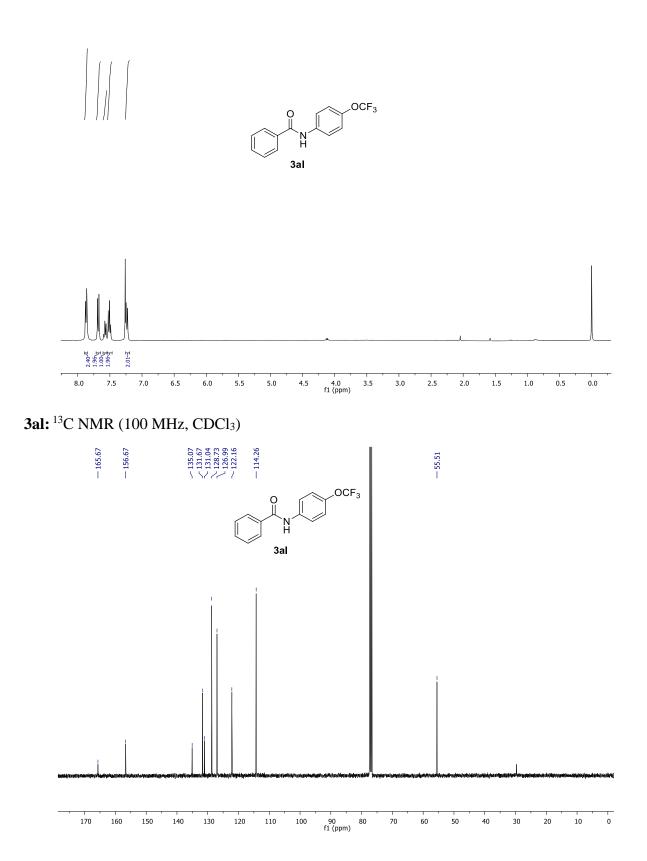


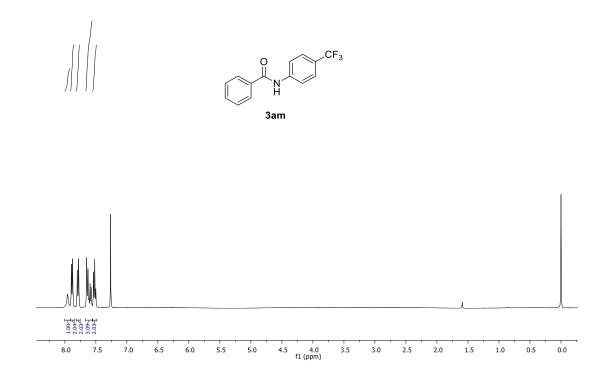




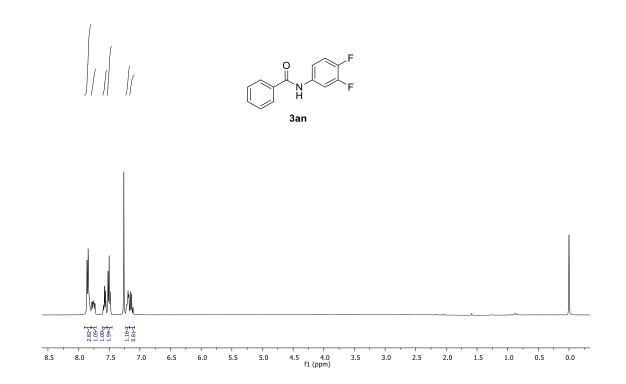




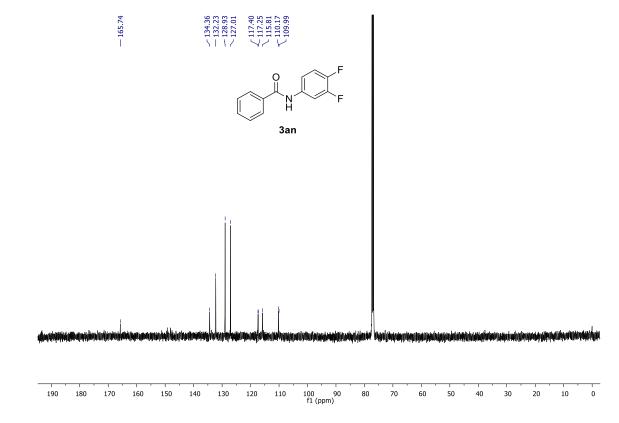




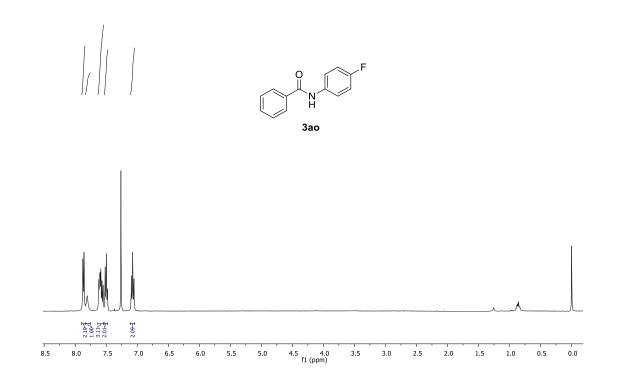
3an: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

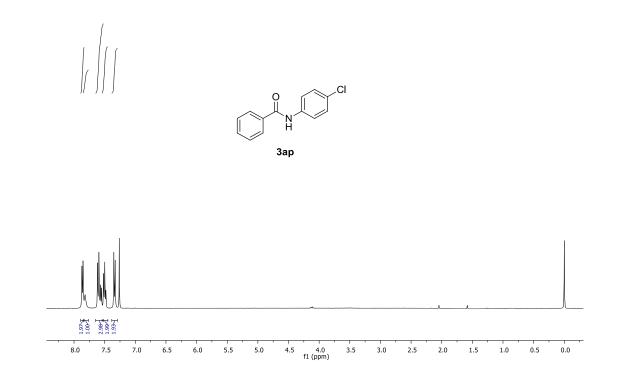


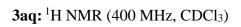
# 3an: <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

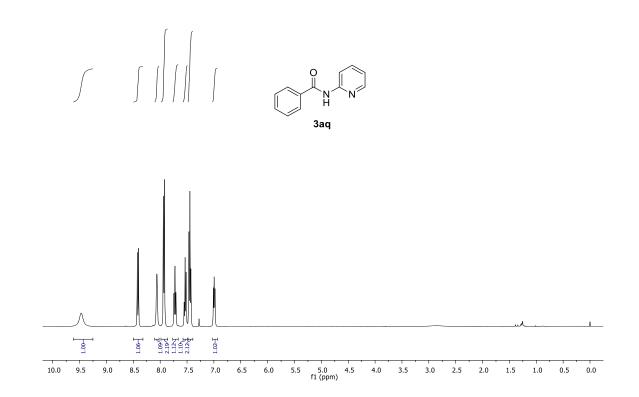


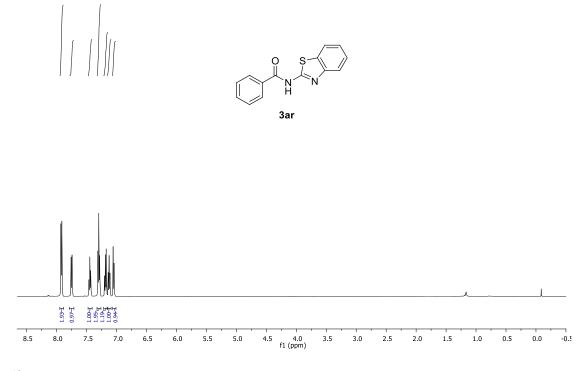
3ao: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)





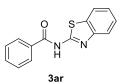


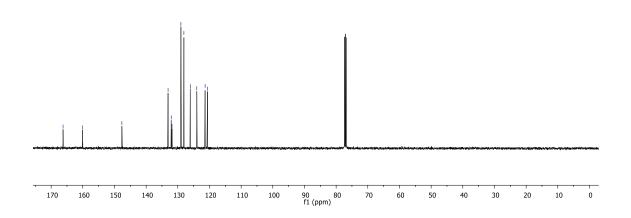


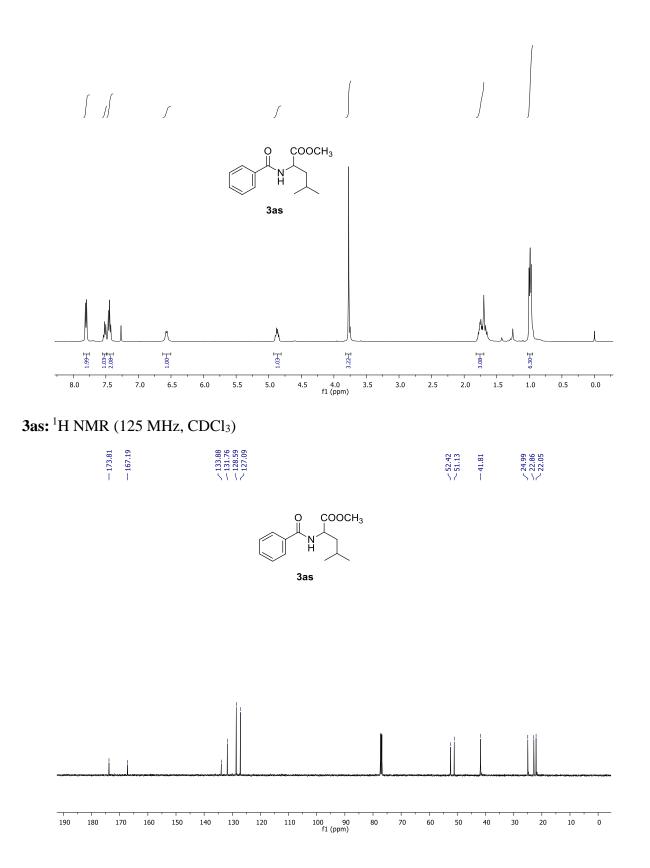


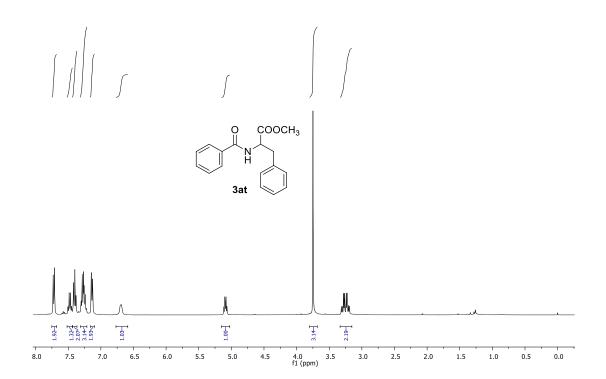
### 3ar: <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

| 166.23 | 160.06 | 147.65 | 133.07<br>132.08<br>131.82<br>129.01<br>128.07<br>126.07<br>124.00<br>121.38<br>121.38 |
|--------|--------|--------|--|
|        |        | 1      |  |

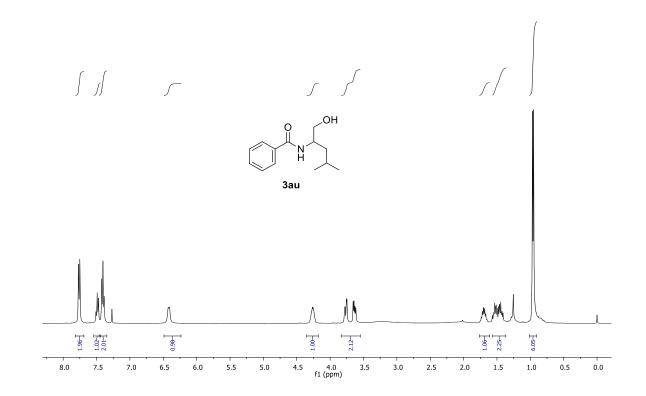


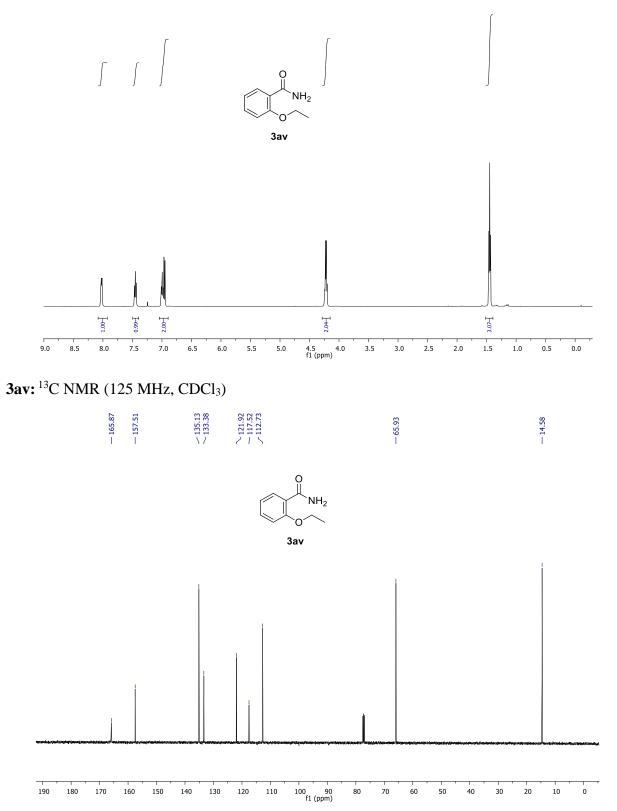


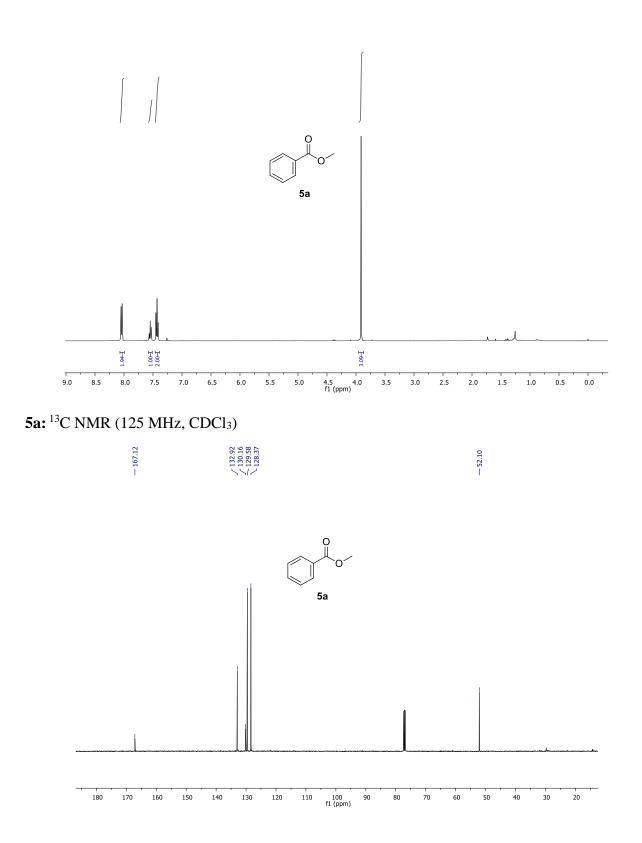




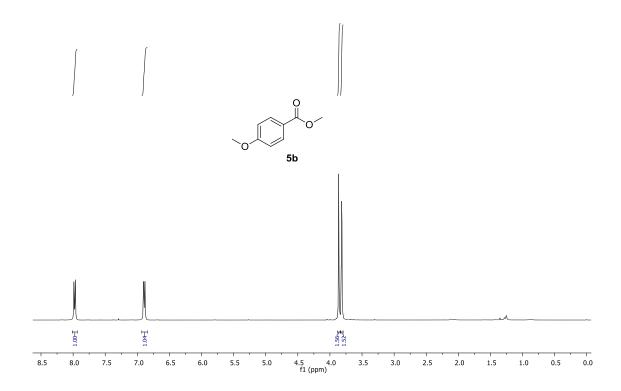
3au: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



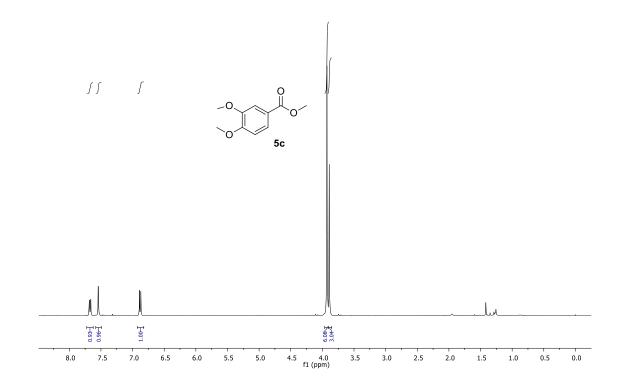




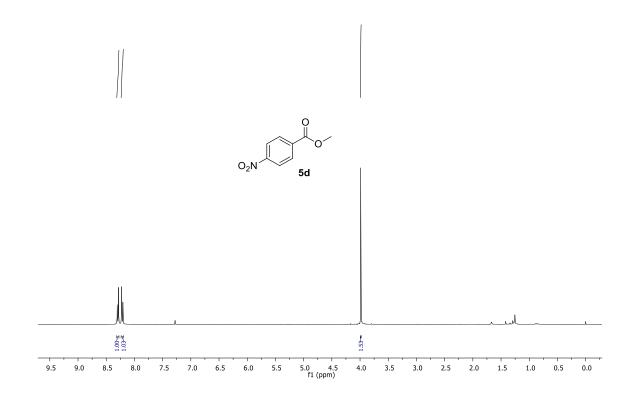
#### **5b:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



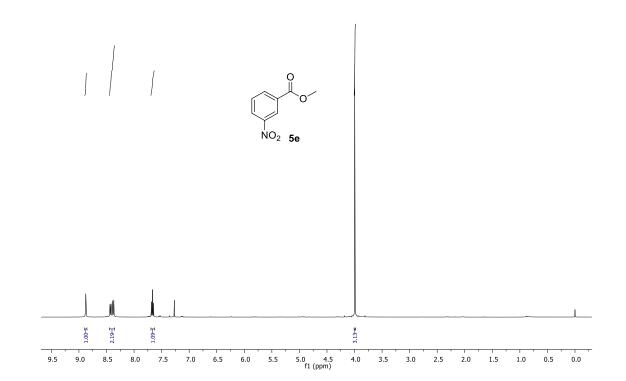
5c: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

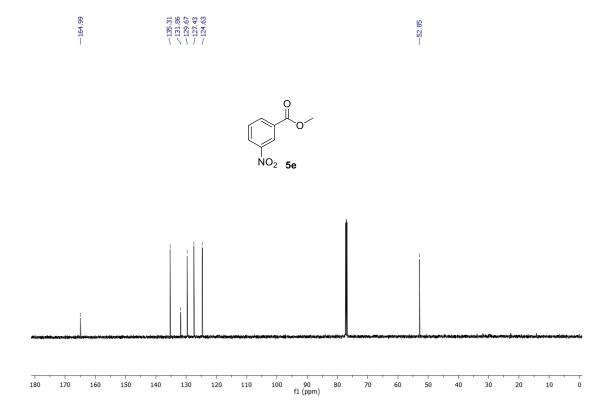


#### **5d:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

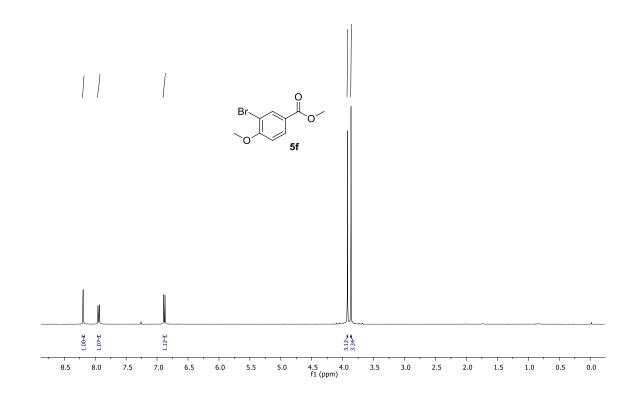


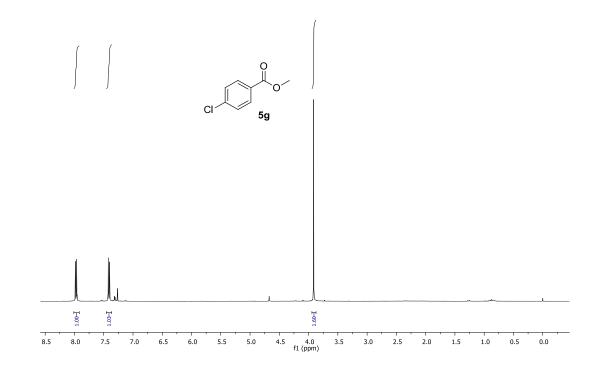
**5e:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

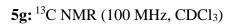




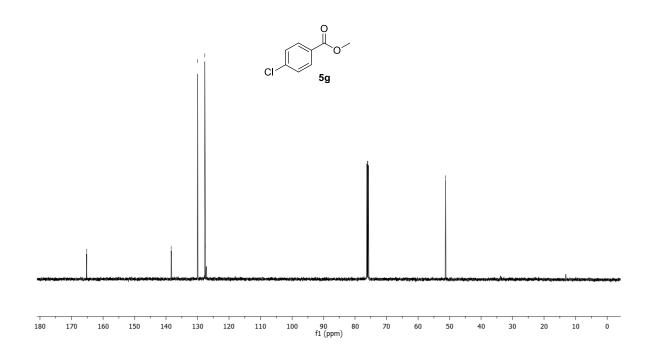
**5f:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

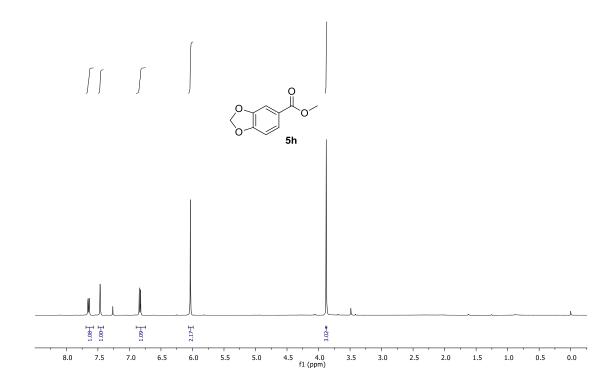




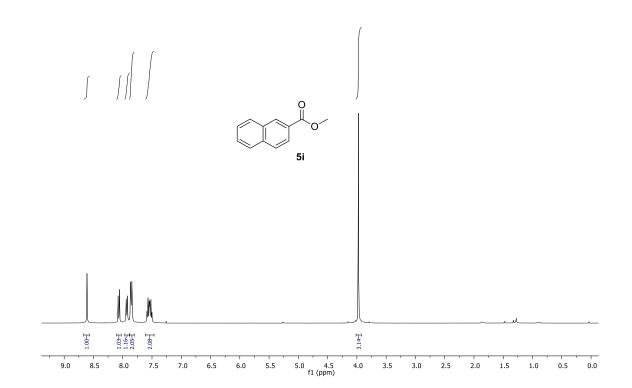




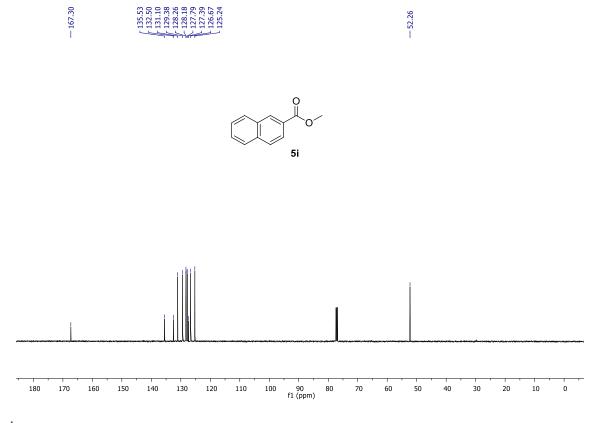




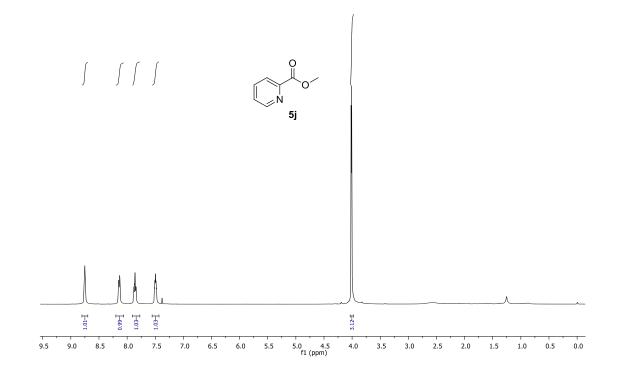
### **5i:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

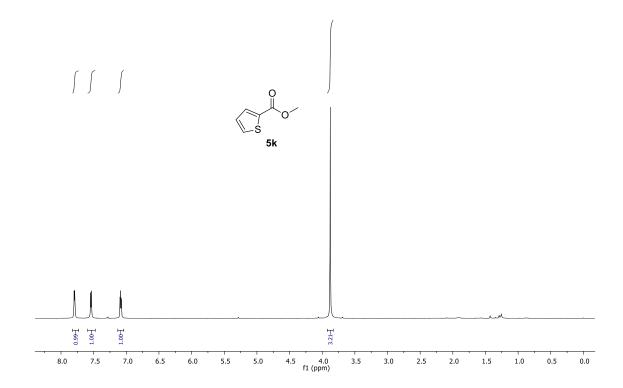


#### **5i:** <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

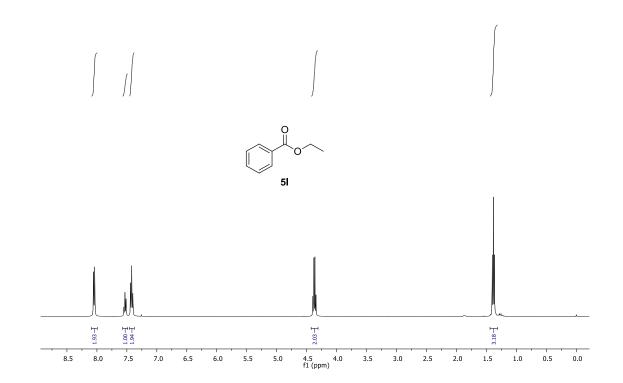


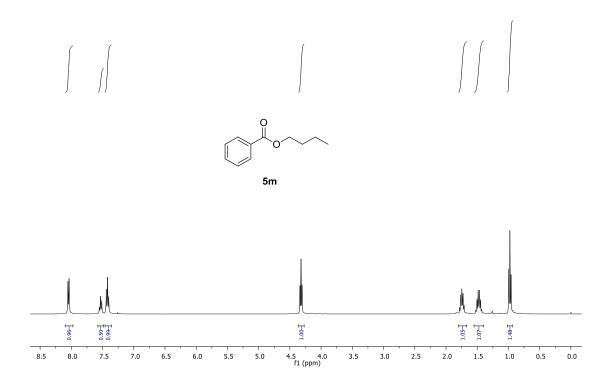
### 5j: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



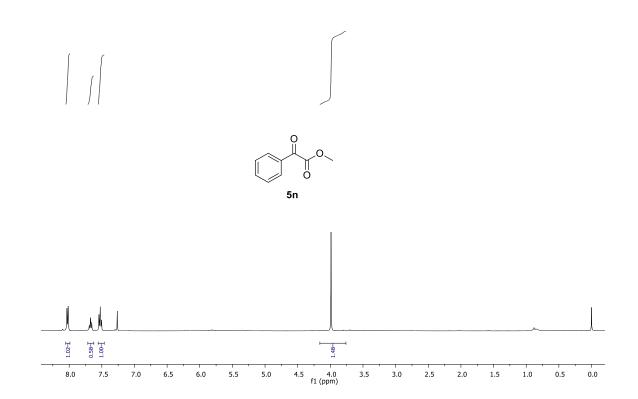


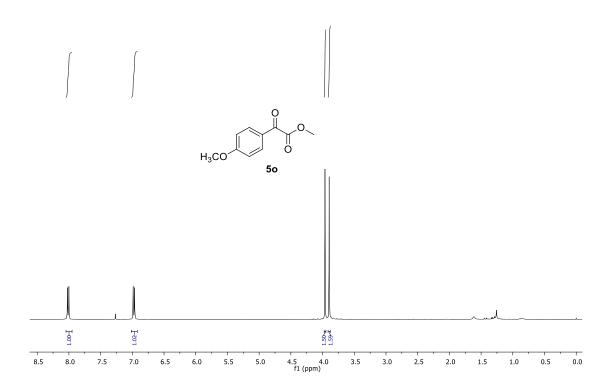
**51:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



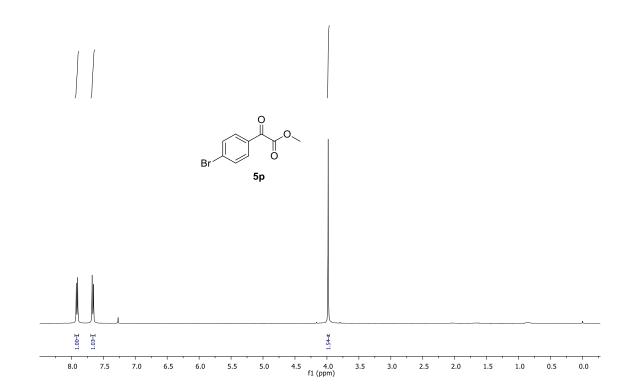


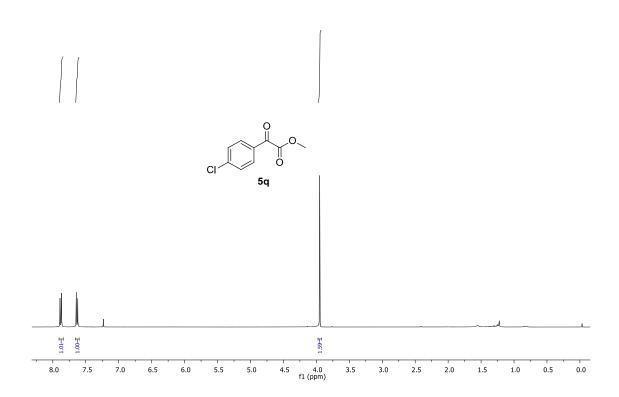
**5n:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



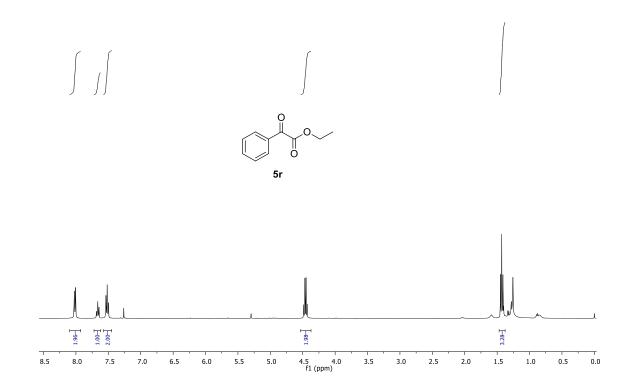


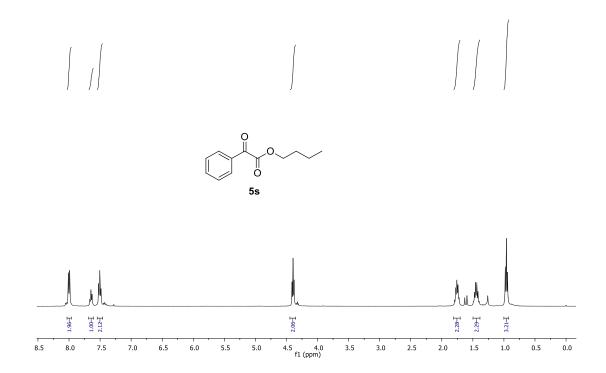
**5p:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)





**5r:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)





**6:** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

