# Electrochemical N-acylation Synthesis of Amides under Aqueous Conditions

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#### **General information**

All reagents were purchased from commercial suppliers and used without further purification. Shanghai chenhua CHI600E electrochemical workstation was used in the standard configuration as delivered, including proprietary software. Catalytic reactions were carried out in undivided electrochemical cells using glassware, if not noted otherwise. Column chromatography was carried out with silica gel (200-300 mesh). Thin layer chromatography was carried out using Merck silica gel GF254 plates. All products were characterized by NMR. <sup>1</sup>H NMR spectra were recorded at 500 MHz and <sup>13</sup>C NMR spectra were recorded at 126 MHz (Bruker DPX) with CDCl<sub>3</sub> and DMSO-d<sub>6</sub> as solvent. Chemical shifts are reported in ppm using TMS as internal standard. Gas chromatography - mass spectra (GC/MS) were recorded on an Agilent Technologies 6890 N instrument with an Agilent 5973N mass detector (EI) and a HP5-MS 30 m x 0.25 mm capillary apolar column (Stationary phase: 5% diphenyldimethylpolysiloxane film, 0.25 µm). GC/MS method: Initial temperature: 150 °C; Initial time: 1 min; Ramp: about 15°C/min until 250 °C then 20 min.

#### General procedure for the catalytic reactions



In a 10 mL glassware benzoic acid (1.0 mmol), amine (1.0 mmol), TBAB (1.0 mmol),  $Cs_2CO_3$  (1.0 mmol) and 2.0 ml of water were placed. Then put in the electrode. The electrochemical workstation was used for 40 mA current catalysis. The reaction mixture was maintained under this condition for 30 minutes. The reaction mixture was continuously stirred during the reaction. After the reaction is completed, open the reaction vessel, add ethanol, adjust the pH to 4 with hydrochloric acid, and then remove the solvent in vacuum. The residue was purified by silica gel column chromatography to obtain the corresponding product. All products were confirmed by nuclear magnetic resonance and mass spectrometry.

#### General procedure for the gram scale experiment

In 50 ml glassware equipped with aniline (5.50 mmol), benzoic acid (5.0 mmol), TBAB (10.0 mmol), KOH (10.0 mmol), and a stir bar. The bottle was equipped with Pt wire electrode as the anode and cathode. Then, 10 mL water was dded. The reaction mixture was stirred and electrolyzed at a constant current of 40 mA under room temperature for 2 h. When the reaction was completed, After the reaction is completed, open the reaction vessel, add ethanol, adjust the pH to 4 with hydrochloric acid, and the combined solution were concentrated with a rotary evaporator. The product was purified by flash column chromatography on silica gel.

#### General procedure for the synthesis of melatonin



In a 10 mL glassware Acetic acid (1.0 mmol), 5-methoxytryptamine (1.0 mmol), TBAB (1.0 mmol),  $Cs_2CO_3$  (2.0 mmol) and 2.0 ml of water were placed. Then put in the electrode. The electrochemical workstation was used for 40 mA current catalysis. The reaction mixture was maintained under this condition for 40 minutes. The reaction mixture was continuously stirred during the reaction. After the reaction is completed, open the reaction vessel, add ethanol, adjust the pH to 4 with hydrochloric acid, and then remove the solvent in vacuum. The residue was purified by silica gel column chromatography to obtain the corresponding product. All products were confirmed by nuclear magnetic resonance and mass spectrometry.

#### **Details of radical trapping experiment**

In a 10 mL glassware, the 2, 2, 6, 6-tetramethylpiperidin-1-oxyl (2.0 mmol), aniline (1.10 mmol), benzoic acid (1.0 mmol), TBAB (1.0 mmol), KOH (1.0 mmol) and 2.0 ml of water were placed. The mixture was stirred at room temperature for 10 min in the air. Then, the reaction mixture was stirred under 40 mA current catalysis. After the reaction was completed the solution of the crude product was concentrated in vacuo, and the residue was purified by column chromatography on a silica gel (petroleum ether/ethyl acetate=5/1) to afford the target product as a yellow solid.<sup>1</sup>H NMR (500 MHz, Chloroform-d)  $\delta$  7.42 – 7.29 (m, 2H), 7.08 (d, J = 8.5 Hz, 2H), 6.94 (t, J = 10.0 Hz, 1H), 1.47 – 1.15 (m, 12H), 0.87 (d, J = 7.6 Hz, 2H), 0.07 (s, 4H).<sup>13</sup>C NMR (126 MHz, Chloroform-d)  $\delta$  144.45, 128.19, 123.35, 122.46, 119.99, 30.49, 29.13, 28.68, 13.10.

#### Cyclic voltammetry experiment

Cyclic voltammograms were measured using Shanghai chenhua CHI600E electrochemical workstation with electrochemical analysis software, using a conventional three-electrode cell. The working electrode was a glassy carbon working electrode, The counter and reference electrodes consisted of a Pt wire and a SCE, espectively. The glassy carbon working electrode was polished with a polishing cloth before each measurement, the concentration of all tested compounds was 1 mmol L-1. The scan rate was 0.1 V/s.

#### **Scheme S1 Electrode selection experiment**

Entries	Electrode	Yield (%)
1	Pt-Ag	93
2	Pt-C	94
3	C-C	90
4	Ag-C	94
5	Ag-Ag	91

#### **Reaction devices**



## Experimental procedures and characterization data

N-phenylbenzamide<sup>1</sup>

<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>) δ 10.26 (s, 1H), 7.97 (d, J = 7.0 Hz, 2H), 7.80 (d, J = 7.8 Hz, 2H), 7.57 (dt, J = 29.7, 7.2 Hz, 3H), 7.36 (t, J = 7.8 Hz, 2H), 7.11 (t, J = 7.4 Hz, 1H). <sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) δ 166.00, 139.64, 135.46, 131.98, 129.04, 128.82, 128.10, 124.10, 120.82. MS (EI, m/z): 197 [M+].

N-(2-hydroxyethyl)benzamide<sup>2</sup>

<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>) δ 7.98 - 7.87 (m, 2H), 7.47 - 7.41 (m, 1H), 7.40 - 7.33 (m, 2H), 3.46 - 3.42 (m, 2H), 3.19 - 3.14 (m, 2H).
 <sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) δ 169.20, 136.99, 130.76, 129.52, 128.11, 55.97, 39.66.
 MS (EI, m/z): 165 [M+].

N-(2-aminoethyl)benzamide<sup>3</sup>

NH<sub>2</sub>

<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>) δ 7.95 – 7.89 (m, 2H), 7.47 – 7.41 (m, 1H), 7.38 (t, J = 7.4 Hz, 2H), 5.88 (s, 2H), 4.17 – 4.11 (m, 2H), 3.75 (m, 2H). <sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) δ 169.20, 136.99, 130.76, 129.52, 128.11, 40.49, 39.49. MS (EI, m/z): 164 [M+].

Benzamide<sup>4</sup>

<sup>1</sup>**H** NMR (500 MHz, DMSO-d<sub>6</sub>) δ 7.92 (d, *J* = 6.8 Hz, 2H), 7.44 (t, *J* = 7.2 Hz, 1H), 7.39 (d, *J* = 7.6 Hz, 2H), 5.84 (s, 2H).

<sup>13</sup>C NMR (126 MHz, DMSO) δ 169.06, 136.73, 130.91, 129.51, 128.18. MS (EI, m/z): 121 [M+].

morpholino(phenyl)methanone<sup>4</sup>

<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>) 7.94 – 7.88 (m, 2H), 7.37 – 7.26 (m, 3H), 3.49 – 3.35 (m, 8H). <sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) δ 171.21, 137.41, 131.93, 130.82, 124.16, 56.48, 19.01. MS (EI, m/z): 191 [M+].

N-(pyridin-2-yl)benzamide<sup>5</sup>

<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>) δ 7.96 (dd, J = 8.2, 1.6 Hz, 2H), 7.89 (dd, J = 5.0, 1.8 Hz, 1H), 7.65 – 7.58 (m, 1H), 7.50 (t, J = 7.8 Hz, 2H), 7.36 (ddd, J = 8.8, 7.0, 2.0 Hz, 1H), 6.49 – 6.43 (m, 2H). <sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) δ 167.96, 160.06, 147.75, 137.55, 133.18, 131.48, 129.70, 128.96, 112.20, 108.56. MS (EI, m/z): 198 [M+].

3-amino-N-phenylbenzamide<sup>6</sup>

<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>)  $\delta$  10.26 (s, 1H), 8.04 – 7.90 (m, 2H), 7.84 – 7.76 (m, 2H), 7.60 (t, *J* = 7.3 Hz, 1H), 7.54 (dd, *J* = 8.2, 6.6 Hz, 2H), 7.45 – 7.28 (m, 2H), 4.03 (d, J = 7.0 Hz, 2H). <sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>)  $\delta$ 167.93, 145.86, 143.06, 133.97, 131.39, 122.87, 117.16, 108.40, 108.16, 106.94, 106.53. MS (EL, m/z): 212 [M+].

2-nitro-N-phenylbenzamide7

<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>) δ7.84 (d, J = 8.2 Hz, 1H), 7.73 (d, J = 7.6 Hz, 2H), 7.61 (d, J = 7.8 Hz, 2H), 7.57 – 7.51 (m, 2H), 7.45 (t, J = 7.6 Hz, 2H).
<sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) δ 166.32, 148.85, 133.53, 132.82, 130.33, 129.74, 128.07, 127.79, 125.35, 124.15, 124.13.
MS (EI, m/z): 242 [M+].

2-iodo-N-phenylbenzamide<sup>8</sup>

<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>) δ 10.44 (s, 1H), 7.94 (d, J = 7.8 Hz, 1H), 7.73 (d, J = 8.0 Hz, 2H), 7.50 (t, J = 5.4 Hz, 2H), 7.36 (t, J = 7.8 Hz, 2H), 7.24 (t, J = 7.6 Hz, 1H), 7.12 (t, J = 7.6 Hz, 1H).
<sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) δ 168.04, 143.61, 139.51, 139.48, 131.49, 129.23, 128.62, 128.52, 124.25, 120.10, 94.15.
MS (EI, m/z): 322 [M+].

N-(thiazol-2-yl)benzamide9

<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>) 6.93 (d, J = 3.3 Hz, 2H), 6.87 (s, 4H), 6.54 (d, J = 3.6 Hz, 2H). <sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>)  $\delta$  159.77, 151.56, 138.39, 136.81, 129.30, 127.79, 119.62, 116.46. MS (EI, m/z): 204 [M+].

3-hydroxy-N-phenylbenzamide1

<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>)  $\delta$  10.17 (s, 1H), 9.76 (s, 1H), 7.80 – 7.75 (m, 2H), 7.41 – 7.29 (m, 5H), 7.09 (tt, J = 7.4, 1.2 Hz, 1H), 6.98 (ddd, J = 8.0, 2.6, 1.0 Hz, 1H). <sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>)  $\delta$  166.04, 157.81, 139.67, 136.92, 129.84, 129.00, 124.00, 120.78, 118.91, 118.62, 115.00.

MS (EI, m/z): 213 [M+].

4-nitro-N-phenylbenzamide1

<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>)  $\delta$  10.59 (s, 1H), 8.37 (d, 2H), 8.27 (d, 2H), 7.85 (t, *J* = 7.8 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.38 (d, *J* = 8.4 Hz, 2H).

<sup>13</sup>C NMR (126 MHz, DMSO-*d*<sub>6</sub>) δ 163.33, 153.73, 139.49, 132.80, 130.15, 128.96, 123.71, 120.75, 115.35. MS (EI, m/z): 242 [M+].

4-hydroxy-N-phenylbenzamide<sup>10</sup>

<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>)δ 10.09 (s, 1H), 9.98 (s, 1H), 7.86 (d, *J* = 8.4 Hz, 2H), 7.76 (d, *J* = 8.0 Hz, 2H), 7.33 (t, *J* = 7.8 Hz, 2H), 7.07 (t, *J* = 7.4 Hz, 1H), 6.87 (d, *J* = 8.4 Hz, 2H).
 <sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) δ 165.55, 160.97, 139.93, 130.13, 128.94, 125.92, 123.70, 120.74, 115.35. MS (EI, m/z): 213 [M+].

3-nitro-N-phenylbenzamide1

NO-

<sup>1</sup>**H NMR (500 MHz, DMSO-d<sub>6</sub>)**  $\delta$  7.46 (dd, J = 8.4, 4.1 Hz, 2H), 7.35 – 7.17 (m, 3H), 6.95 (d, J = 8.3 Hz, 2H), 6.87 (t, J = 7.4 Hz, 2H).

<sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) δ 165.93, 148.35, 135.79, 135.78, 133.02, 130.93, 130.10, 127.70, 125.73, 124.11, 123.93.

MS (EI, m/z): 242 [M+].

4-bromo-N-phenylbenzamide1

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  10.55 (q, J = 7.1 Hz, 1H). 8.14 (d, J = 8.7 Hz, 2H), 7.99 (d, J = 8.7 Hz, 2H), 7.85 (s, 1H), 7.74 (t, J = 6.9 Hz, 2H), 7.47 – 7.41 (m, 2H). <sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) 167.04, 132.37, 132.12, 131.98, 131.72, 130.53, 129.69, 128.97, 127.27. MS (EI, m/z): 275 [M+].

4-methoxy-N-phenylbenzamide11

H<sub>2</sub>CO

<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>) δ 10.08 (s, 1H), 8.01 – 7.93 (m, 2H), 7.78 (dd, *J* = 8.6, 1.2 Hz, 2H), 7.34 (dd, *J* = 8.6, 7.4 Hz, 2H), 7.11 – 7.03 (m, 3H), 3.85 (s, 3H). <sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>)δ 165.34, 162.35, 139.81, 130.02, 128.97, 127.48, 123.84, 120.81, 114.04, 55.89. MS (EI, m/z): 227 [M+].

2-hydroxy-N-phenylbenzamide<sup>12</sup>

<sup>1</sup>**H NMR (500 MHz, DMSO-d<sub>6</sub>)** 10.17 (s, 1H), 9.76 (s, 1H), 7.67 – 7.62 (m, 2H), 7.50 – 7.44 (m, 2H), 7.29 – 7.25 (m, 1H), 7.22 (d, *J* = 7.8 Hz, 2H), 7.06 – 7.00 (m, 2H).

<sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) δ 172.33, 161.60, 136.01, 131.95, 130.71, 127.79, 124.13, 119.57, 117.51, 115.56, 113.5.

MS (EI, m/z): 213 [M+].

4-cyano-N-phenylbenzamide<sup>13</sup>



<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>) δ 10.17 (s, 1H), 7.78 (d, J = 7.3 Hz, 2H), 7.38 (d, J = 7.8 Hz, 2H), 7.34 (dd, J = 8.2, 2.8 Hz, 4H), 7.12 – 7.07 (m, 1H), 6.98 (d, J = 6.6 Hz, 1H). <sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) δ 166.48, 135.37, 133.10, 132.01, 130.33, 130.36, 119.43, 118.61, 115.51, 112.40. MS (EI, m/z): 222 [M+].

N-(4-methoxyphenyl)benzamide<sup>14</sup>

<sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>) δ 10.19 (s, 1H), 8.00 (d, *J* = 7.4 Hz, 2H), 7.73 (d, *J* = 8.6 Hz, 2H), 7.62 (d, *J* = 7.2 Hz, 1H), 7.57 (t, *J* = 7.6 Hz, 2H), 6.98 (d, *J* = 8.6 Hz, 2H), 3.80 (s, 3H).
<sup>13</sup>C NMR (126 MHz, DMSO-*d*<sub>6</sub>) δ 165.58, 156.00, 135.50, 132.68, 131.86, 128.83, 128.01, 122.43, 114.19, 55.63. MS (EI, m/z): 227 [M+].

N-(4-chlorophenyl)benzamide11

<sup>1</sup>H NMR (500 MHz, DMSO- $d_6$ ) $\delta$  10.44 (s, 1H), 8.00 (d, J = 7.6 Hz, 2H), 7.88 (d, J = 8.6 Hz, 2H), 7.65 (d, J = 7.2 Hz, 1H), 7.59 (t, J = 7.6 Hz, 2H), 7.47 (d, J = 8.6 Hz, 2H). <sup>13</sup>C NMR (126 MHz, DMSO- $d_6$ )  $\delta$  166.14, 138.62, 135.16, 132.20, 129.01, 128.91, 128.15, 127.72, 122.30. MS (EI, m/z): 231 [M+].

N-phenylacetamide<sup>15</sup>

<sup>1</sup>**H NMR (500 MHz, DMSO-***d*<sub>6</sub>) δ 9.94 (s, 1H), 7.62 – 7.54 (m, 2H), 7.33 – 7.24 (m, 2H), 7.05 – 6.99 (m, 1H), 2.05 (s, 3H).

<sup>13</sup>C NMR (126 MHz, DMSO-*d*<sub>6</sub>) δ 168.76, 139.79, 129.12, 123.43, 119.43, 24.46. MS (EI, m/z): 135 [M+].

N-p-tolylbenzamide16

<sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>) δ 10.22 (s, 1H), 7.98 (d, J = 7.4 Hz, 2H), 7.70 (d, J = 8.0 Hz, 2H), 7.61 (t, J = 7.2 Hz, 1H), 7.55 (t, J = 7.4 Hz, 2H), 7.19 (d, J = 8.0 Hz, 2H), 2.31 (s, 3H). <sup>13</sup>C NMR (126 MHz, DMSO-*d*<sub>6</sub>) δ 165.83, 137.11, 135.51, 133.08, 131.92, 129.46, 128.82, 128.06, 120.86, 20.96. MS (EI, m/z): 211 [M+].

N-(4-nitrophenyl)benzamide17

NO<sub>2</sub>

<sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>) δ 10.87 (s, 1H), 8.31 (d, J = 8.8 Hz, 2H), 8.12 (d, J = 8.8 Hz, 2H), 8.03 (d, J = 7.8 Hz, 2H), 7.67 (d, J = 7.2 Hz, 1H), 7.61 (t, J = 7.6 Hz, 2H). <sup>13</sup>C NMR (126 MHz, DMSO-*d*<sub>6</sub>) δ 166.78, 145.98, 142.91, 134.69, 132.65, 128.99, 128.39, 125.26, 120.29. MS (EI, m/z): 242 [M+]. 2-acetamidoacetic acid<sup>19</sup>

<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>) δ 12.49 (s, 1H), 8.16 (t, J = 6.0 Hz, 1H), 3.73 (d, J = 6.0 Hz, 2H), 1.86 (s, 3H). <sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) δ 171.86, 170.1, 41.07, 22.69. MS (EI, m/z): 118 [M+].

Benzohydrazide<sup>20</sup>



<sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>) δ 9.93 (s, 1H), 7.58 (d, J = 8.1 Hz, 2H), 7.28 (d, J = 7.8 Hz, 2H), 7.03 (d, J = 7.4 Hz, 1H), 2.04 (s, 2H).
<sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) δ 164.90, 135.93, 125.27, 119.58, 115.56.
MS (EI, m/z): 137 [M+].

Melatonin<sup>18</sup>

<sup>1</sup>**H NMR (500 MHz, DMSO-d<sub>6</sub>)**  $\delta$  10.66 – 10.62 (m, 1H), 7.94 (t, *J* = 5.8 Hz, 1H), 7.24 (d, *J* = 8.8 Hz, 1H), 7.11 (d, *J* = 2.6 Hz, 1H), 7.04 (d, *J* = 2.6 Hz, 1H), 6.73 (dd, *J* = 8.8, 2.4 Hz, 1H), 3.77 (s, 3H), 3.33 (td, *J* = 7.4, 5.8 Hz, 2H), 2.79 (t, *J* = 7.4 Hz, 2H), 1.83 (s, 3H).

<sup>13</sup>C NMR (126 MHz, DMSO-d<sub>6</sub>) δ 169.47, 153.46, 131.89, 128.05, 123.70, 112.42, 112.18, 111.48, 100.70, 55.84, 39.92, 25.74, 23.16.

MS (EI, m/z): 232 [M+].

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## $^{1}\mathrm{H}$ NMR and $^{13}\mathrm{C}$ NMR spectra for the products

N-phenylbenzamide<sup>1</sup>







N-(2-aminoethyl)benzamide<sup>2</sup>



benzamide<sup>2</sup>





N-(pyridin-2-yl)benzamide<sup>4</sup>



3-amino-N-phenylbenzamide<sup>5</sup>



2-nitro-N-phenylbenzamide<sup>6</sup>



### 2-iodo-N-phenylbenzamide



N-(thiazol-2-yl)benzamide<sup>7</sup>



3-hydroxy-N-phenylbenzamide<sup>8</sup>



4-nitro-N-phenylbenzamide7













4-bromo-N-phenylbenzamide



4-methoxy-N-phenylbenzamide







4-cyano-N-phenylbenzamide



N-(4-methoxyphenyl)benzamide



N-(4-chlorophenyl)benzamide



N-phenylacetamide



N-p-tolylbenzamide



N-(4-nitrophenyl)benzamide



melatonin



2-acetamidoaceticacid



Benzohydrazide



