Iodide-catalyzed Amide Synthesis from Alcohols and Amines

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I. General Methods

All reagents were purchased from commercial suppliers and used without further purification. $^1$H-NMR, $^{13}$C-NMR spectra were measured on a Bruker AM 400 NMR spectrometer (400 MHz or 100MHz, respectively) with CDCl$_3$ as solvent and recorded in ppm relative to internal tetramethylsilane standard. The following abbreviations were used to describe splitting patterns: s = singlet, d = doublet, t = triplet, q = quartet. Coupling constants $J$ are given in Hz. Mass spectra were recorded on a Waters Q-TOF Premier instrument by using ESI method or Agilent 6890-5973N GCMS-EI instrument. Thin layer chromatography was performed on fluorescence indicator marked precoated silica gel 60 plates and visualization was achieved by UV light (254 nm). Flash chromatography was performed on silica gel (0.040-0.063mm). Unless otherwise stated, all commercially available substances and reagents were used as received from their suppliers.

**General procedure for NHS active ester. (Table 2)**

Alcohol 1 (0.5 mmol) and N-hydroxysuccinimide 2a (0.75 mmol) were added to a tube (10 mL) with a magnetic stirring bar and solvent (CH$_3$CN, 2 mL). To this mixture 10 mol% of $n$Bu$_4$NI (0.05 mmol) and anhydrous TBHP (4.0 equiv) were added at room temperature. After stirring at 80 °C for 18h, 10 mL water was added and the mixture was extracted with EtOAc (3×15 mL). The combined organic phase was wished by saturated solution of NaCl (1×10 mL) and dried with Na$_2$SO$_4$ and evaporated in vacuum. The residue was purified by flash silica gel column chromatography using a mixture of petroleum ether and ethyl acetate (8:1 to 4:1) as eluent to afford the desired products.

**General procedure for NHPI active ester. (Table 3)**

Alcohol 1 (0.5 mmol) and N-hydroxyphthalimide 2b (0.75 mmol) were added to a tube (10 mL) with a magnetic stirring bar and solvent (CH$_3$CN, 2 mL). To this mixture 10 mol% of NaI (0.05 mmol), 0.2mmol KOH and aqueous TBHP (4.0 equiv) were added at room temperature. After stirring at 80 °C for 8h, the solvent was removed under vacuum and the residue was purified by flash silica gel column chromatography using a mixture of petroleum ether and ethyl acetate (7:1) as eluent to afford the desired products.

**General procedure for amide. (Table 5)**

Alcohol 1 (0.5 mmol) and N-hydroxysuccinimide 2a (0.75 mmol) were added to a tube (10 mL) with a magnetic stirring bar and solvent (CH$_3$CN, 2 mL). To this mixture 10 mol% of $n$Bu$_4$NI (0.05 mmol) and anhydrous TBHP (4.0 equiv) were added at room temperature. The mixture stir at 80 °C for 18h. After the mixture was cooled to room temperature, amine (0.75 mmol) was added to the mixture in one portion and the mixture was stirred at room temperature for 12 h. 10 mL water was added and the mixture was extracted with EtOAc (3×15 mL). The combined organic phase was wished by saturated solution of NaCl (1×10 mL) and dried with Na$_2$SO$_4$ and evaporated in vacuum. The residue was purified by flash silica gel column chromatography using a mixture of petroleum ether and ethyl acetate (10:1) as eluent to afford the desired products.
II. Added Experimental Data

Table S1 Optimization of the conditions for the esterification of NHPI.\textsuperscript{[a]}

![Chemical structure](image)

<table>
<thead>
<tr>
<th>Entry</th>
<th>n(1a): n(2a)</th>
<th>Catalyst</th>
<th>additive</th>
<th>Yield(%)</th>
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<tbody>
<tr>
<td>1</td>
<td>1.0 : 0.5</td>
<td>NaI</td>
<td>NaOH</td>
<td>65%</td>
</tr>
<tr>
<td>2</td>
<td>1.0 : 0.5</td>
<td>KI</td>
<td>NaOH</td>
<td>58%</td>
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<tr>
<td>3</td>
<td>1.0 : 0.5</td>
<td>I₂</td>
<td>NaOH</td>
<td>65%</td>
</tr>
<tr>
<td>4</td>
<td>1.0 : 0.5</td>
<td>(n\text{Bu₄NI})</td>
<td>NaOH</td>
<td>61%</td>
</tr>
<tr>
<td>5</td>
<td>1.0 : 0.5</td>
<td>Et₄NI</td>
<td>NaOH</td>
<td>57%</td>
</tr>
<tr>
<td>6</td>
<td>1.0 : 0.5</td>
<td>(\text{PhI(OAc)}_2)</td>
<td>NaOH</td>
<td>0%</td>
</tr>
<tr>
<td>7</td>
<td>1.0 : 0.5</td>
<td>-</td>
<td>NaOH</td>
<td>0%</td>
</tr>
<tr>
<td>8</td>
<td>0.5 : 1.0</td>
<td>NaI</td>
<td>Cs₂CO₃</td>
<td>71%</td>
</tr>
<tr>
<td>9</td>
<td>0.5 : 1.0</td>
<td>NaI</td>
<td>K₃PO₄</td>
<td>78%</td>
</tr>
<tr>
<td>10</td>
<td>0.5 : 1.0</td>
<td>NaI</td>
<td>KOH</td>
<td>95%</td>
</tr>
<tr>
<td>11</td>
<td>0.5 : 1.0</td>
<td>NaI</td>
<td>K₂CO₃</td>
<td>87%</td>
</tr>
<tr>
<td>12\textsuperscript{[b]}</td>
<td>0.5 : 0.75</td>
<td>NaI</td>
<td>KOH</td>
<td>34%</td>
</tr>
<tr>
<td>13\textsuperscript{[c]}</td>
<td>0.5 : 0.75</td>
<td>NaI</td>
<td>KOH</td>
<td>93%</td>
</tr>
</tbody>
</table>

\textsuperscript{[a]} Unless otherwise specified, all reactions were carried out using benzyl alcohol and NHPI in ethyl acetate (2.0 mL) with 10 mol% of catalyst and aqueous TBHP (4.0 equiv) at 80°C for 18 hours. \textsuperscript{[b]} 4 hours. \textsuperscript{[c]} 8 hours.
III. Experimental Data of Products (Table 2, Table 3 and Table 5)

Table 2:

2,5-dioxopyrrolidin-1-yl benzoate

(3aa) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.15 (d, $J = 7.2$ Hz, 2H), 7.69 (t, $J = 7.5$ Hz, 1H), 7.52 (t, $J = 7.9$ Hz, 2H), 2.91 (s, 4H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.3, 161.9, 135.0, 130.6, 128.9, 125.1, 25.7.

MS (ESI) m/z 242.01 [(M+Na)$^+$]

2,5-dioxopyrrolidin-1-yl 4-methylbenzoate

(3ba) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.02 (d, $J = 8.0$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 2H), 2.91 (s, 4H), 2.45 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.4, 161.9, 146.1, 130.6, 129.6, 122.3, 25.7, 21.9.

MS (ESI) m/z 256.06 [(M+Na)$^+$]

2,5-dioxopyrrolidin-1-yl 4-methoxybenzoate

(3ca) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.09 (d, $J = 9.0$ Hz, 2H), 6.98 (d, $J = 9.0$ Hz, 2H), 3.89 (s, 3H), 2.90 (s, 4H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.6, 164.9, 161.5, 132.9, 117.0, 114.2, 55.6, 25.7.

MS (ESI) m/z 272.06 [(M+Na)$^+$]

2,5-dioxopyrrolidin-1-yl 4-fluorobenzoate

(3da) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.17 (m, 2H), 7.20 (t, $J = 8.4$ Hz, 2H), 2.92 (s, 4H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.3, 168.1, 165.6, 160.9, 133.4, 121.4, 116.3, 25.7.

MS (ESI) m/z 260.04 [(M+Na)$^+$]
2,5-dioxopyrrolidin-1-yl 4-chlorobenzoate

(3ea) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.08 (d, $J = 8.6$ Hz, 2H), 7.50 (d, $J = 8.4$ Hz, 2H), 2.92 (s, 4H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.2, 161.1, 141.7, 131.9, 129.4, 123.5, 25.7.

MS (ESI) m/z 276.04 [(M+Na)$^+$]

2,5-dioxopyrrolidin-1-yl 4-bromobenzoate

(3fa) White solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.00 (d, $J = 8.6$ Hz, 2H), 7.67 (d, $J = 8.6$ Hz, 2H), 2.92 (s, 4H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.2, 161.3, 132.3, 131.9, 130.5, 124.0, 25.7.

MS (ESI) m/z 319.77 [(M+Na)$^+$]

2,5-dioxopyrrolidin-1-yl 3-chlorobenzoate

(3ga) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.12 (s, 1H), 8.03 (d, $J = 8.0$ Hz, 1H), 7.65 (m, 1H), 7.47 (t, $J = 8.0$ Hz, 1H), 2.92 (s, 4H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.1, 160.9, 135.1, 135.0, 130.5, 130.3, 128.7, 126.8, 25.7.

MS (ESI) m/z 276.02 [(M+Na)$^+$]

2,5-dioxopyrrolidin-1-yl 2-methylbenzoate

(3ha) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.12 (d, $J = 7.3$ Hz, 1H), 7.52 (t, $J = 6.7$ Hz, 1H), 7.32 (t, $J = 7.6$ Hz, 2H), 2.91 (s, 4H), 2.62 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.5, 162.1, 142.1, 134.0, 132.0, 131.3, 126.1, 124.2, 25.7, 21.6.

MS (ESI) m/z 256.10 [(M+Na)$^+$]

2,5-dioxopyrrolidin-1-yl 2-chlorobenzoate
(3ia) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.12 (d, $J = 8.2$ Hz, 1H), 7.57 (m, 2H), 7.40 (m, 1H), 2.92 (s, 4H).
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.1, 160.2, 135.6, 134.7, 132.4, 131.7, 126.9, 124.5, 25.7.
MS (ESI) m/z 276.02 [(M+Na)$^+$]

**2,5-dioxopyrrolidin-1-yl 1-naphthoate**

(3ja) white solid

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$8.81 (d, $J = 8.6$ Hz, 1H), 8.46 (d, $J = 7.3$ Hz, 1H), 8.15 (d, $J = 8.0$ Hz, 1H), 7.92 (d, $J = 8.0$ Hz, 1H), 7.66 (t, $J = 7.2$ Hz, 1H), 7.58 (t, $J = 8.0$ Hz, 2H), 2.95 (s, 4H).
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.6, 162.2, 135.6, 133.7, 131.9, 131.4, 128.8, 128.7, 126.9, 125.2, 124.5, 121.5, 25.8.
MS (ESI) m/z 292.08 [(M+Na)$^+$]

**2,5-dioxopyrrolidin-1-yl 4-cyanobenzoate**

(3ka) white solid

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.25 (d, $J = 8.4$ Hz, 2H), 7.83 (d, $J = 8.4$ Hz, 2H), 2.94 (s, 4H).
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 168.7, 160.5, 132.5, 130.9, 129.0, 126.9, 118.2, 117.3, 105.4, 25.7
MS (ESI) m/z 267.06 [(M+Na)$^+$]

**2,5-dioxopyrrolidin-1-yl thiophene-2-carboxylate**

(3la) yellow solid

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.04 (d, $J = 3.7$ Hz, 1H), 7.78 (d, $J = 4.8$ Hz, 1H), 7.21 (t, $J = 4.3$ Hz, 1H), 2.91 (s, 4H).
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.2, 157.4, 136.7, 135.8, 128.4, 126.9, 25.7.
MS (ESI) m/z 248.04 [(M+Na)$^+$]

**2,5-dioxopyrrolidin-1-yl furan-2-carboxylate**
2,5-dioxopyrrolidin-1-yl cinnamate

Table 3:

1,3-dioxoisooindolin-2-yl benzoate

1,3-dioxoisooindolin-2-yl 4-methylbenzoate

1,3-dioxoisooindolin-2-yl 4-fluorobenzoate
(3db) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.23 (m, 2H), 7.93 (d, $J$=2.9Hz, 2H), 7.83 (d, $J$=1.7Hz, 2H), 7.23 (t, $J$=8.6Hz, H=2).
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 168.2, 165.6, 162.0, 161.9, 134.9, 133.5, 133.4, 128.9, 124.1, 116.4, 116.2.
MS (ESI) m/z 308.06 [(M+Na$^+$)].

**1,3-dioxoisoindolin-2-yl 4-chlorobenzoate**

(3eb) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.14 (d, $J$ = 8.0 Hz, 2H), 7.93 (dd, $J$ = 4.9, 3.1 Hz, 2H), 7.82 (dd, $J$ = 4.9, 3.1 Hz, 2H), 7.53 (d, $J$ = 8.4 Hz, 2H).
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 162.1, 162.0, 141.7, 134.9, 132.0, 129.4, 128.9, 124.1, 123.7.
MS (ESI) m/z 324.01 [(M+Na$^+$)].

**1,3-dioxoisoindolin-2-yl 4-bromobenzoate**

(3fb) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.06 (d, $J$ = 8.3 Hz, 2H), 7.93 (dd, $J$ = 4.8, 3.1 Hz, 2H), 7.82 (dd, $J$ = 4.9, 3.1 Hz, 2H), 7.69 (d, $J$ = 8.3 Hz, 2H).
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 162.3, 162.0, 135.0, 132.4, 132.0, 130.5, 128.9, 124.2, 124.1.
MS (ESI) m/z 367.94 [(M+Na$^+$)].

**1,3-dioxoisoindolin-2-yl 2-methylbenzoate**

(3hb) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.21 (d, $J$ = 7.4 Hz, 1H), 7.93 (dd, $J$ = 5.2, 3.0 Hz, 2H), 7.82 (dd, $J$ = 5.2, 3.0 Hz, 2H), 7.55 (t, $J$=7.1, 1H), 7.35 (t, $J$=6.4, 2H), 2.65 (s, 3H).
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 171.2, 163.1, 162.3, 142.2, 134.8, 134.0, 132.0, 131.4, 129.0, 126.1, 124.0, 21.7.
MS (ESI) m/z 304.07 [(M+Na$^+$)].
1,3-dioxoisindolin-2-yl 2-chlorobenzoate

(3ib) white solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.19 (d, \(J = 7.8\) Hz, 1H), 7.94 (dd, \(J = 5\), 3.2 Hz, 2H), 7.83 (dd, \(J = 5\), 3.2 Hz, 2H), 7.57 (s, 2H), 7.44 (m, 1H).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 161.9, 161.2, 135.6, 134.9, 134.7, 132.5, 131.7, 129.0, 126.9, 124.7, 124.1.

MS (ESI) m/z 324.01 [(M+Na\(^+\)]

1,3-dioxoisindolin-2-yl 1-naphthoate

(3jb) white solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.84 (d, \(J = 8.5\) Hz, 1H), 8.54 (d, \(J = 7.2\) Hz, 1H), 8.16 (d, \(J = 8.1\) Hz, 1H), 7.93 (m, 3H), 7.81 (dd, \(J = 5.3\), 3.1 Hz, 2H), 7.66 (t, \(J = 7.0\) Hz, 1H), 7.59 (t, \(J = 7.2\) Hz).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 163.1, 162.4, 135.6, 134.8, 133.7, 132.0, 131.5, 129.1, 128.9, 128.8, 126.8, 125.3, 124.5, 124.1, 121.6.

MS (ESI) m/z 340.0 [(M+Na\(^+\)]

1,3-dioxoisindolin-2-yl thiophene-2-carboxylate

(3lb) white solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.09 (t, \(J = 1.0\) Hz, 1H), 7.93 (dd, \(J = 5.4\), 3.1 Hz, 2H), 7.82 (m, 3H), 7.23 (t, \(J = 3.8\) Hz, 1H).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 162.0, 158.3, 136.7, 135.7, 134.9, 128.9, 128.5, 127.0, 124.1.

MS (ESI) m/z 295.98 [(M+Na\(^+\)]

1,3-dioxoisindolin-2-yl furan-2-carboxylate

(3mb) yellow solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.93 (dd, \(J = 5.3\), 3.1 Hz, 2H), 7.82 (dd, \(J = 5.3\), 3.1 Hz, 2H), 7.77 (s, 1H), 7.55 (d, \(J = 3.5\) Hz, 1H), 6.66 (dd, \(J = 3.4\), 1.4 Hz, 1H).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 161.9, 154.5, 148.8, 139.8, 134.9, 128.8, 124.1, 122.3, 112.7.

MS (ESI) m/z 280.02 [(M+Na\(^+\)]

S9
Table 5:  
N-benzylbenzamide

![N-benzylbenzamide structure](image)

(4a) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.79 (d, J = 7.2 Hz, 2H), 7.49 (m, 1H), 7.41 (t, J = 7.2 Hz, 2H), 7.32 (m, 5H), 6.5 (br, 1H), 4.63 (d, J = 6.4 Hz, 2H).
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 167.5, 138.2, 134.4, 131.5, 128.8, 128.6, 127.9, 127.6, 127.0, 44.1. 
MS (EI) m/z (%) 211 (M$^+$, 100), 105 (86), 91 (9), 77 (42).

N-benzyl-4-methylbenzamide

![N-benzyl-4-methylbenzamide structure](image)

(4b) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.68 (d, J = 6.8 Hz, 2H), 7.33 (m, 5H), 7.22 (d, J = 6.8 Hz, 2H), 6.54 (br, 1H), 4.61 (d, J = 7.9 Hz, 2H), 2.38 (s, 3H).
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 167.4, 142.0, 138.3, 131.5, 129.2, 128.8, 127.9, 127.6, 127.0, 44.1, 21.4. 
MS (EI) m/z (%) 225 (M$^+$, 48), 119 (100), 106 (12), 91 (67), 77 (15).

N-benzyl-4-chlorobenzamide

![N-benzyl-4-chlorobenzamide structure](image)

(4c) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.70 (s, 2H), 6.78 (m, 7H), 6.61 (br, 1H), 4.60 (s, 2H).
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 166.4, 138.0, 137.8, 132.7, 128.8, 128.5, 127.9, 127.7, 44.2. 
MS (EI) m/z (%) 247 (M$^+$, 20), 245 (M$^+$, 62), 139 (100), 111(48), 91 (28), 77 (30).

N-benzyl-1-naphthamide

![N-benzyl-1-naphthamide structure](image)

(4d) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.31 (s, 1H), 7.87 (m, 2H), 7.36 (m, 9H), 6.39 (br, 1H), 4.89 (s, 2H).
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.5, 138.1, 134.2, 133.7, 130.7, 130.2, 128.8, 128.3, 127.9, 127.7, 127.2, 126.5, 125.4, 125.0, 124.7, 44.1. 
MS (EI) m/z (%) 261 (M$^+$, 58), 155 (72), 127 (100), 91 (25), 77 (28).
N-benzyl-4-methoxybenzamide

\[
\begin{aligned}
\text{MeO} & \quad \begin{array}{c}
\text{O} \\
\text{N}
\end{array} \\
\text{H} & \quad \begin{array}{c}
\text{MeO}
\end{array}
\end{aligned}
\]

(4e) white solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta \) 8.06 (d, \(J = 10.0\) Hz, 1H), 7.76 (d, \(J = 10.0\) Hz, 2H), 7.32 (m, 3H), 6.91 (t, \(J = 8.0\) Hz, 3H), 6.46 (br, 1H), 6.62 (d, \(J = 4.6\) Hz, 2H), 3.83 (s, 3H).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta \) 167.0, 162.3, 138.4, 132.3, 128.8, 127.9, 127.6, 126.6, 113.8, 55.4, 44.1.

MS (EI) m/z (%) 241 (M\(^+\), 48), 135 (100), 127 (6), 107 (9), 92 (20), 77 (32).

N-phenethylbenzamide

\[
\begin{aligned}
\text{O} & \quad \begin{array}{c}
\text{N}
\end{array} \\
\text{H} & \quad \begin{array}{c}
\text{MeO}
\end{array}
\end{aligned}
\]

(4f) white solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta \) 7.69 (d, \(J = 7.2\) Hz, 2H), 7.47 (t, \(J = 6.8\) Hz, 1H), 7.39 (t, \(J = 6.8\) Hz, 2H), 7.32 (t, \(J = 6.8\) Hz, 2H), 7.24 (s, 3H), 6.21 (br, 1H), 3.72 (d, \(J = 6.0\) Hz, 2H), 2.93 (t, \(J = 6.0\) Hz, 2H).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta \) 167.5, 134.7, 131.4, 128.8, 128.7, 128.6, 126.8, 126.6, 41.2, 35.7.

MS (EI) m/z (%) 225 (M\(^+\), 26), 134 (12), 105 (100), 91 (18), 77 (59).

N-(1-phenylethyl)benzamide

\[
\begin{aligned}
\text{O} & \quad \begin{array}{c}
\text{N}
\end{array} \\
\text{H} & \quad \begin{array}{c}
\text{MeO}
\end{array}
\end{aligned}
\]

(4g) white solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta \) 7.76 (d, \(J = 7.2\) Hz, 2H), 7.26-7.48 (m, 8H), 6.43 (br, 1H), 5.33 (t, \(J = 6.8\) Hz, 1H), 1.60 (d, \(J = 6.8\) Hz, 3H).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta \) 166.7, 143.1, 134.6, 131.5, 128.8, 128.7, 126.9, 126.3, 49.3, 21.7.

MS (EI) m/z (%) 225 (M\(^+\), 39), 210 (8), 120 (7), 105 (100), 91 (2), 77 (64).

N-cyclohexylbenzamide

\[
\begin{aligned}
\text{O} & \quad \begin{array}{c}
\text{N}
\end{array} \\
\text{H} & \quad \begin{array}{c}
\text{MeO}
\end{array}
\end{aligned}
\]

(4h) white solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta \) 7.75 (d, \(J = 7.2\) Hz, 2H), 7.43 (m, 3H), 6.03 (br, 1H), 3.98 (t, \(J = 4.0\) Hz, 1H), 2.03(d, \(J = 7.0\) Hz, 2H), 1.75(d, \(J = 12.8\) Hz, 2H), 1.65(d, \(J = 12.4\) Hz, 1H), 1.43(q, \(J = 24.3,12.8\) Hz, 2H), 1.24(q, \(J = 23.2,10.7\) Hz, 2H).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta \) 166.7, 135.1, 131.2, 128.5, 126.8, 48.7, 33.2, 25.6, 24.9.

MS (EI) m/z (%) 203 (M\(^+\), 34), 160 (5), 122 (66), 105 (100), 92 (20), 77 (62).
N, N-diethylbenzamide

![Structure of N, N-diethylbenzamide](image)

(4i) colourless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.38 (m, 5H), 3.55 (s, 2H), 3.25 (s, 2H), 1.24 (s, 3H), 1.11 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 171.3, 137.3, 129.1, 128.4, 126.2, 43.3, 39.2, 14.2, 12.9.

MS (EI) m/z (%) 177 (M$^+$, 22), 176 (60), 148 (4), 134 (3), 105 (100), 77 (34).

phenyl (pyrrolidin-1-yl) methanone

![Structure of phenyl (pyrrolidin-1-yl) methanone](image)

(4j) colourless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.51 (m, 2H), 7.39 (m, 3H), 3.65 (t, $J$ = 7.2 Hz, 2H), 3.42 (t, $J$ = 7.2 Hz, 2H), 1.96 (m, 2H), 1.87 (m, 2H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.7, 137.2, 129.8, 128.2, 127.0, 49.6, 46.1, 26.4, 24.5.

MS (EI) m/z (%) 175 (M$^+$, 63), 146 (22), 105 (100), 77 (46).

(S)-methyl 2-benzamido-3-phenylpropanoate

![Structure of (S)-methyl 2-benzamido-3-phenylpropanoate](image)

(4k) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.65 (d, $J$ = 7.2 Hz, 2H), 7.44 (t, $J$ = 7.2 Hz, 1H), 7.35 (t, $J$ = 7.6 Hz, 2H), 7.20 (m, 3H), 7.06 (d, $J$ = 6.8 Hz, 2H), 6.51 (br, 1H), 5.02 (q, $J$ = 13.2, 7.2 Hz, 1H), 3.70 (s, 3H), 3.19 (m, 2H), 1.63 (s, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 172.1, 166.8, 135.9, 133.9, 131.8, 129.3, 128.6, 128.6, 127.2, 127.0, 53.5, 52.4, 37.9.

MS (EI) m/z (%) 283 (M$^+$, 2), 224 (7), 207 (7), 162 (72), 131 (14), 105 (100), 91 (10), 77 (35).

N-phenylbenzamide

![Structure of N-phenylbenzamide](image)

(4l) white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.98 (br, 1H), 7.85 (d, $J$ = 7.2 Hz, 2H), 7.63 (d, $J$ = 7.6 Hz, 2H), 7.47 (m, 3H), 7.35 (t, $J$ = 7.2 Hz, 2H), 7.14 (t, $J$ = 7.2 Hz, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 165.9, 138.0, 135.0, 131.8, 129.0, 128.8, 127.1, 124.6, 120.3.

MS (EI) m/z (%) 197 (M$^+$, 40), 105 (100), 92 (4), 77 (69).
IV. Copies of $^1$H and $^{13}$C-NMR Spectra

2,5-dioxopyrrolidin-1-yl benzoate (3aa)
2,5-dioxopyrrolidin-1-yl 4-methylbenzoate (3ba)
2,5-dioxopyrrolidin-1-yl 4-methoxybenzoate (3ca)

![Diagram of 2,5-dioxopyrrolidin-1-yl 4-methoxybenzoate (3ca)](image)

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2,5-dioxopyrrolidin-1-yl 4-fluorobenzoate (3da)
2,5-dioxopyrrolidin-1-yl 4-chlorobenzoate (3ea)
2,5-dioxopyrrolidin-1-yl 4-bromobenzoate (3fa)
2,5-dioxopyrrolidin-1-yl 3-chlorobenzoate (3ga)
2,5-dioxopyrrolidin-1-yl 2-methylbenzoate (3ha)
2,5-dioxopyrrolidin-1-yl 2-chlorobenzoate (3ia)
2,5-dioxopyrrolidin-1-yl 1-naphthoate (3ja)
2,5-dioxopyrrolidin-1-yl thiophene-2-carboxylate (3la)
2,5-dioxopyrrolidin-1-yl furan-2-carboxylate (3ma)
2,5-dioxopyrrolidin-1-yl cinnamate (3na)

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Table 3:

1,3-dioxoisindolin-2-yl benzoate (3ab)
1,3-dioxoisindolin-2-yl 4-methylbenzoate (3bb)
1,3-dioxoisooindolin-2-yl 4-fluorobenzoate (3db)
1,3-dioxoisindolin-2-yl 4-chlorobenzoate (3eb)
1,3-dioxoisooindolin-2-yl 4-bromobenzoate (3fb)
1,3-dioxoisindolin-2-yl 2-methylbenzoate (3hb)
1,3-dioxoisooindolin-2-yl 2-chlorobenzoate (3ib)
1,3-dioxoisooindolin-2-yl 1-naphthoate (3jb)
1,3-dioxoisindolin-2-yl thiophene-2-carboxylate (3lb)

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1,3-dioxoisindolin-2-yl furan-2-carboxylate (3mb)

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Table 5: N-benzylbenzamide (4a)
N-benzyl-4-methylbenzamide (4b)
N-benzyl-4-chlorobenzamide (4c)
N-benzyl-1-naphthamide(4d)
N-cyclohexylbenzamide (4h)
N, N-diethylbenzamide (4i)
phenyl (pyrrolidin-1-yl) methanone (4j)
(S)-methyl 2-benzamido-3-phenylpropanoate (4k)
N-phenylbenzamide (4l)