Supplementary Information

For

*nBu₄NI-Catalyzed Intermolecular C-O Cross-coupling Reactions:

Synthesis of Alkyloxyamines

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I. General Considerations

All reagents were purchased from commercial sources and used without further treatment, unless otherwise indicated. All reactions were run under air with no precautions taken to exclude moisture. $^1$H NMR and $^{13}$C NMR spectra were recorded at 25 ºC on a Varian (400 MHz and 100 MHz). Melting points were obtained with a micro melting point XT4A Beijing Keyi electrooptic apparatus and are uncorrected. High resolution mass spectra were recorded on Bruck microtof. All reactions were monitored by TLC with Taizhou GF254 silica gel coated plates. Flash column chromatography was carried out using 200-300 mesh silica gel at increased pressure.

II. Synthesis Procedure

Substrates 2e was prepared by the reaction of p-cresol (1 equiv) and pivaloyl chloride (1.1 equiv), triethylamine (1.1 equiv) in CH$_2$Cl$_2$ at room temperature. Substrates 2f was prepared by the reaction of p-toluidine and pivaloyl chloride in CH$_2$Cl$_2$ at room temperature.$^1$ Substrates 2g was prepared according to literature procedure.$^2$

III. Optimization of the Reaction Conditions

Table 1 Optimization of the Reaction Conditions$^a$

<table>
<thead>
<tr>
<th>Entry</th>
<th>Oxidant</th>
<th>Catalyst</th>
<th>Solvent</th>
<th>T (ºC)</th>
<th>Yield(%)</th>
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<tr>
<td>1</td>
<td>TBHP</td>
<td>nBu$_4$NI</td>
<td>CH$_2$CN</td>
<td>130</td>
<td>85</td>
</tr>
<tr>
<td>2</td>
<td>TBHP</td>
<td>nBu$_4$NI</td>
<td>EtOAc</td>
<td>130</td>
<td>73</td>
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<tr>
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<td>nBu$_4$NI</td>
<td>DCM</td>
<td>130</td>
<td>55</td>
</tr>
<tr>
<td>4</td>
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<td>I$_2$</td>
<td>CH$_2$CN</td>
<td>130</td>
<td>25</td>
</tr>
<tr>
<td>5</td>
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<td>CH$_2$CN</td>
<td>130</td>
<td>28</td>
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<tr>
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<td>CH$_2$CN</td>
<td>130</td>
<td>0</td>
</tr>
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<td>130</td>
<td>trace</td>
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<tr>
<td>8</td>
<td>Na$_2$S$_2$O$_4$</td>
<td>nBu$_4$NI</td>
<td>CH$_2$CN</td>
<td>130</td>
<td>0</td>
</tr>
<tr>
<td>9</td>
<td>TBP</td>
<td>nBu$_4$NI</td>
<td>CH$_2$CN</td>
<td>130</td>
<td>trace</td>
</tr>
<tr>
<td>Entry</td>
<td>Reagent 1</td>
<td>Reagent 2</td>
<td>Solvent</td>
<td>Temperature</td>
<td>Yield</td>
</tr>
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<td>$n$Bu$_4$Ni</td>
<td>CH$_3$CN</td>
<td>130 °C</td>
<td>0 %</td>
</tr>
<tr>
<td>11</td>
<td>-</td>
<td>$n$Bu$_4$Ni</td>
<td>CH$_3$CN</td>
<td>130 °C</td>
<td>trace</td>
</tr>
<tr>
<td>12</td>
<td>TBHP</td>
<td>-</td>
<td>CH$_3$CN</td>
<td>130 °C</td>
<td>trace</td>
</tr>
<tr>
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<td>TBHP</td>
<td>$n$Bu$_4$Ni</td>
<td>CH$_3$CN</td>
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<td>92 %</td>
</tr>
<tr>
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<td>81 %</td>
</tr>
<tr>
<td>15</td>
<td>TBHP</td>
<td>$n$Bu$_4$Ni</td>
<td>CH$_3$CN</td>
<td>70 °C</td>
<td>52 %</td>
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</tbody>
</table>

* Reaction conditions: 1a (1.5 mmol), 2 (0.3 mmol), oxidant (0.6 mmol), catalysts (0.06 mmol), solvent (3.0 mL), 2 h. * TBHP (70% in water). * Yield of the isolated product. *H$_2$O$_2$ 30% in water.

**Entry 3 as an example**

To a solution of the $N$-Hydroxyphthalimide (NHPI) 2 (48.9 mg, 0.3 mmol) in dichloromethane (3.0 mL) was added o-xylene 1a (181 μL, 1.5 mmol), TBHP (82 μL, 0.6 mmol, 70% in water), and $n$Bu$_4$Ni (22.2 mg, 0.06 mmol) in a sealed tube. The sealed tube was then tightly sealed with a screw cap and the reaction was stirred for the 2.0 h at 130 °C. After the reaction finished, the reaction mixture was cooled to room temperature and quenched with saturated solution of Na$_2$S$_2$O$_3$ (3.0 mL). The mixture was extracted with ethyl acetate (3 × 5.0 mL). The combined organic phases were dried over anhydrous Na$_2$SO$_4$ and the solvent was evaporated under vacuum. The residue was purified by column chromatography to give the corresponding products 3a (44.1 mg, 55%).

**IV. General procedure for the preparation of 3 and 5**

**1a as an example**

![Chemical structure](attachment:image.png)

To a solution of the $N$-Hydroxyphthalimide (NHPI) 2 (48.9 mg, 0.3 mmol) in acetonitrile (3.0 ml) was added the o-xylene 1a (181 μL, 1.5 mmol), TBHP (82 μL, 0.6 mmol, 70% in water), and $n$Bu$_4$Ni (22.2 mg, 0.06 mmol) in a sealed tube. The sealed tube was then tightly sealed with a screw cap and the reaction was stirred for the 2.0 h at 100 °C. After the reaction finished, the reaction mixture was cooled to room temperature and quenched by the addition of a saturated solution of Na$_2$S$_2$O$_3$ (3.0 mL). The mixture was extracted with ethyl acetate (3 × 5.0 mL). The combined organic phases were dried over anhydrous Na$_2$SO$_4$ and the solvent was evaporated under vacuum. The residue was purified by column chromatography to give the corresponding products 3a (73.7 mg, 92%).

**V. General procedure for the preparation of 6 and 7**
3n as an example

\[
\begin{array}{c}
\text{3n} \\
\text{PINO} \\
\begin{array}{c}
\text{Mo(CO)}_6, \text{Et}_3\text{N} \\
\text{CH}_3\text{CN} / \text{H}_2\text{O}
\end{array} \\
80 \, ^\circ\text{C}, 12 \, \text{h}
\end{array} \\
\begin{array}{c}
\text{3n} \\
\text{PINO} \\
\begin{array}{c}
\text{H}_2\text{NNH}_2\cdot\text{H}_2\text{O} \\
\text{MeOH} / \text{CHCl}_3
\end{array} \\
25 \, ^\circ\text{C}
\end{array} \\
\begin{array}{c}
\text{6b} \\
\begin{array}{c}
\text{O}\text{-NH}_2
\end{array}
\end{array}
\end{array}
\]

A mixture of 3n (53 mg, 0.2 mmol), Mo(CO)$_6$ (53 mg, 0.2 mmol) and Et$_3$N (0.42 mL, 3.0 mmol) in MeCN-H$_2$O (15:1, 2.0 mL) was stirred at 80 °C for 12 h. The mixture was concentrated, and the residue was purified by column chromatography to give the corresponding products 6b (19.0 mg, 78%).

To a solution of 3n (53 mg, 0.2 mmol) in 10% MeOH in CHCl$_3$ (2.0 mL) was added hydrazine monohydrate (0.03 mL, 0.6 mmol), and the reaction mixture was stirred at room temperature. The progress of the reaction was monitored by TLC using ethyl acetate and petroleum ether as eluent. After completion, the colorless precipitate was filtered and washed with a 1:5 mixture of Et$_2$O : petroleum ether (4.0 mL). The solution was acidified to pH = 3 with H$_2$SO$_4$ in ether / petroleum ether (1:3). A white precipitate formed and was separated by filtration washing with ether / petroleum ether (1:3, 10.0 mL). The collected white precipitate was partitioned between ether (5.0 mL) and aqueous saturated K$_2$CO$_3$ solution (5.0 mL), and the aqueous phase was extracted with additional portions of ether (3 × 5.0 mL). The combined organic layers were dried and concentrated to give O-(1-phenylethyl)hydroxylamine 7b as a colorless oil (19.2 mg, 70%).

VI. Control experiment on the reaction mechanism

\[
\begin{array}{c}
\text{and} \\
\begin{array}{c}
\text{D} \\
\text{D} \\
\text{D} \\
\text{D} \\
\text{D} \\
\text{D} \\
\text{D} \\
\text{D}
\end{array}
\end{array} \\
\begin{array}{c}
\begin{array}{c}
\text{N-O} \\
\text{O}
\end{array} \\
\begin{array}{c}
\text{O} \\
\text{O}
\end{array}
\end{array} \\
\begin{array}{c}
\text{and} \\
\begin{array}{c}
\text{D} \\
\text{D} \\
\text{D} \\
\text{D} \\
\text{D} \\
\text{D} \\
\text{D} \\
\text{D}
\end{array}
\end{array} \\
\begin{array}{c}
\begin{array}{c}
\text{N-O} \\
\text{O}
\end{array} \\
\begin{array}{c}
\text{O} \\
\text{O}
\end{array}
\end{array} \\
\begin{array}{c}
\text{100 \, ^\circ\text{C}, CH}_3\text{CN, 1.0 h}
\end{array}
\end{array}
\]

To a solution of the NHPI 2 (48.9 mg, 0.3 mmol) in acetonitrile (3.0 ml) was added the toluene 1b (79 μL, 0.75 mmol), toluene-d$_8$ 1b-d$_8$ (80 μL, 0.75 mmol), TBHP (82 μL, 0.6 mmol, 70% in
water), and nBu₄NI (22.2 mg, 0.06 mmol) in a sealed tube. The sealed tube was then tightly sealed with a screw cap and the reaction mixture was stirred at 100 ºC for 1.0 h. After the reaction was quenched by saturated solution of Na₂S₂O₃ (3.0 mL), the mixture was extracted with ethyl acetate (3 × 5.0 mL), the combined organic phases were dried over anhydrous Na₂SO₄ and the solvent was evaporated under vacuum. The residue was purified by flash column chromatography. The KIE value was determined by average of multiplet runs and a representative ¹H NMR spectrum was provided as follows.

VII. Analytical data of Compounds 3, 5, 6, 7 and 8

2-((2-methylbenzyl)oxy)isoindoline-1,3-dione 3a
White solid. mp: 129-131 ºC ¹H NMR (400 MHz; CDCl₃): δ = 2.60 (s, 3H), 5.23 (s, 2H), 7.15 (t, ⁴J = 7.2 Hz, 1H), 7.22-7.30 (m, 2H), 7.37 (d, ⁴J = 7.6 Hz, 1H), 7.70-7.72 (m, 2H), 7.77-7.80 (m, 2H); ¹³C NMR (100 MHz; CDCl₃): δ = 18.9, 77.9, 123.2, 125.7, 128.7, 129.6, 130.4, 131.2, 131.5, 134.2, 138.9, 163.3. HRMS (ESI-TOF) Calcd for C₁₆H₁₄NO₃, [M+H]+ m/z 268.0974; Found 268.0980.
**2-(benzyloxy)isoindoline-1,3-dione 3b**

White solid. mp: 143-146 °C

$^1$H NMR (400 MHz; CDCl$_3$): $\delta = 5.21$ (s, 2H), 7.36-7.38 (m, 3H), 7.53 (t, $J = 3.6$ Hz, 2H), 7.71-7.73 (m, 2H), 7.79-7.81 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 79.8$, 123.4, 128.5, 128.8, 129.3, 129.8, 133.8, 134.4, 163.4. HRMS (ESI-TOF) Calcd for C$_{15}$H$_{12}$NO$_3$, [M+H]$^+$ m/z 254.0817; Found 254.0813.

**2-((3-methylbenzyl)oxy)isoindoline-1,3-dione 3c**

White solid. mp: 117-119 °C

$^1$H NMR (400 MHz; CDCl$_3$): $\delta = 2.34$ (s, 3H), 5.16 (s, 2H), 7.16 (t, $J = 7.6$ Hz, 1H), 7.25 (t, $J = 7.6$ Hz, 1H), 7.31 (d, $J = 7.6$ Hz, 1H), 7.35 (s, 1H), 7.70-7.72 (m, 2H), 7.78-7.80 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 21.2$, 79.8, 123.3, 126.8, 128.3, 128.7, 129.9, 130.4, 133.4, 134.3, 138.1, 163.3. HRMS (ESI-TOF) Calcd for C$_{16}$H$_{14}$NO$_3$, [M+H]$^+$ m/z 268.0974; Found 268.0978.

**2-((4-methylbenzyl)oxy)isoindoline-1,3-dione 3d**

White solid. mp: 134-136 °C

$^1$H NMR (400 MHz; CDCl$_3$): $\delta = 2.33$ (s, 3H), 5.16 (s, 2H), 7.16 (d, $J = 7.6$ Hz, 2H), 7.41 (d, $J = 8.0$ Hz, 2H), 7.69-7.72 (m, 2H), 7.77-7.79 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 21.2$, 79.6, 123.3, 128.8, 129.1, 129.9, 130.6, 134.3, 139.2, 163.4. HRMS (ESI-TOF) Calcd for C$_{16}$H$_{14}$NO$_3$, [M+H]$^+$ m/z 268.0974; Found 268.0975.

**4-(((1,3-dioxoisoindolin-2-yl)oxy)methyl)phenyl pivalate 3e**

White solid. mp: 142-144 °C

$^1$H NMR (400 MHz; CDCl$_3$): $\delta = 1.35$ (s, 9H), 5.20 (s, 2H), 7.07 (d, $J = 8.4$ Hz, 2H), 7.55 (d, $J = 8.4$ Hz, 2H), 7.72-7.75 (m, 2H), 7.79-7.80 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 27.1$, 39.1, 79.1, 121.7, 123.5, 128.9, 131.0, 131.1, 134.4, 151.9, 163.5, 176.8. HRMS (ESI-TOF) Calcd for C$_{20}$H$_{20}$NO$_5$, [M+H]$^+$ m/z 354.1341; Found 354.1341.
N-(4-(((1,3-dioxoisooindolin-2-yl)oxy)methyl)phenyl)pivalamide 3f
White solid. mp:163-164 °C 1H NMR (400 MHz; CDCl3): δ = 1.31 (s, 9H), 5.16 (s, 2H), 7.36 (s, 1H), 7.47 (d, J = 8.4 Hz, 2H), 7.55 (d, J = 8.4 Hz, 2H), 7.71-7.73 (m, 2H), 7.78-7.80 (m, 2H); 13C NMR (100 MHz, CDCl3): δ = 27.6, 39.7, 79.2, 119.6, 123.5, 128.9, 129.4, 130.9, 134.4, 139.0, 163.5, 176.6. HRMS (ESI-TOF) Calcd for C20H21N2O4, [M+H]+ m/z 353.1501; Found 353.1509.

tert-butyl (4-(((1,3-dioxoisooindolin-2-yl)oxy)methyl)phenyl)carbamate 3g
White solid. mp:169-171 °C 1H NMR (400 MHz; CDCl3): δ = 1.50 (s, 9H), 5.14 (s, 2H), 6.58 (s, 1H), 7.36 (d, J = 8.4 Hz, 2H), 7.43 (d, J = 8.4 Hz, 2H), 7.71-7.73 (m, 2H), 7.78-7.80 (m, 2H); 13C NMR (100 MHz, CDCl3): δ = 28.3, 79.3, 80.7, 118.1, 123.5, 128.0, 128.8, 131.0, 134.4, 139.4, 152.5, 163.5. HRMS (ESI-TOF) Calcd for C20H21N2O5, [M+H]+ m/z 369.1450; Found 369.1455.

2-((4-bromobenzyl)oxy)isoindoline-1,3-dione 3h
White solid. mp:135-137 °C 1H NMR (400 MHz; CDCl3): δ = 5.14 (s, 2H), 7.39 (d, J = 8.4 Hz, 2H), 7.48 (d, J = 8.4 Hz, 2H), 7.71-7.73 (m, 2H), 7.78-7.80 (m, 2H); 13C NMR (100 MHz, CDCl3): δ = 78.9, 123.5, 128.7, 131.3, 131.7, 132.7, 134.4, 163.3. HRMS (ESI-TOF) Calcd for C15H11BrNO3, [M+H]+ m/z 331.9922; Found 331.9928.

2-((2-nitrobenzyl)oxy)isoindoline-1,3-dione 3i
White solid. mp:145-147 °C 1H NMR (400 MHz; CDCl3): δ = 5.63 (s, 2H), 7.52 (t, J = 7.6 Hz, 1H), 7.68-7.72 (m, 1H), 7.74-7.76 (m, 2H), 7.81-7.83 (m, 2H), 7.99 (d, J = 7.6 Hz, 1H), 8.11 (dd, J1 = 0.8 Hz, J2 = 8.0 Hz, 1H); 13C NMR (100 MHz, CDCl3): δ = 75.8, 123.6, 124.9, 128.8, 129.2, 129.9, 130.7, 133.8, 134.6, 147.4, 163.2. HRMS (ESI-TOF) Calcd for C15H11N2O5, [M+H]+ m/z 299.0668; Found 299.0674.

2-((2-chlorobenzyl)oxy)isoindoline-1,3-dione 3j
White solid. mp:142-143 °C 1H NMR (400 MHz; CDCl3): δ = 5.36 (s, 2H), 7.29-7.32 (m, 2H), 7.38-7.40 (m, 1H), 7.62-7.64 (m, 1H), 7.73-7.75 (m, 2H), 7.80-7.82 (m, 2H); 13C NMR (100 MHz, CDCl3): δ = 76.4, 123.5, 127.0, 128.8, 129.6, 130.5, 131.8, 131.9, 134.4, 134.7, 163.3. HRMS (ESI-TOF) Calcd for C15H11ClNO3, [M+H]+ m/z 288.0427; Found 288.0432.
2-((2-bromobenzyl)oxy)isoindoline-1,3-dione 3k
White solid. mp:150-151 °C ¹H NMR (400 MHz; CDCl₃): δ = 5.36 (s, 2H), 7.21-7.25 (m, 1H), 7.35 (dt, J₁ = 1.2 Hz, J₂ = 7.6 Hz, 1H), 7.58 (dd, J₁ = 1.2 Hz, J₂ = 8.0 Hz, 1H), 7.65 (dd, J₁ = 1.6 Hz, J₂ = 7.6 Hz, 1H), 7.73-7.75 (m, 2H), 7.81-7.83 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 78.7, 123.5, 124.5, 127.6, 128.9, 130.7, 131.7, 132.9, 133.7, 134.5, 163.3. HRMS (ESI-TOF) Calcd for C₁₅H₁₁BrNO₃, [M+H]⁺ m/z 331.9922; Found 331.9929.

2-((3-bromobenzyl)oxy)isoindoline-1,3-dione 3l
White solid. mp:130-132 °C ¹H NMR (400 MHz; CDCl₃): δ = 5.15 (s, 2H), 7.25 (t, J = 7.6 Hz, 1H), 7.47-7.49 (m, 2H), 7.68 (s, 1H), 7.72-7.74 (m, 2H), 7.79-7.82 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 78.8, 122.4, 123.5, 128.1, 128.7, 130.1, 132.3, 132.5, 134.5, 135.9, 163.3. HRMS (ESI-TOF) Calcd for C₁₅H₁₁BrNO₃, [M+H]⁺ m/z 331.9922; Found 331.9928.

2-((3-iodobenzyl)oxy)isoindoline-1,3-dione 3m
White solid. mp:148-150 °C ¹H NMR (400 MHz; CDCl₃): δ = 5.12 (s, 2H), 7.12 (t, J = 7.6 Hz, 1H), 7.52 (d, J = 7.6 Hz, 1H), 7.69 (d, J = 8.0 Hz, 1H), 7.72-7.74 (m, 2H), 7.80-7.82 (m, 2H), 7.87 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 78.8, 94.2, 123.6, 128.8, 128.9, 130.3, 134.6, 136.0, 138.3, 138.4, 163.4. HRMS (ESI-TOF) Calcd for C₁₅H₁₁INO₃, [M+H]⁺ m/z 379.9784; Found 379.9788.

2-(1-phenylethoxy)isoindoline-1,3-dione 3n
White solid. mp:93-95 °C ¹H NMR (400 MHz; CDCl₃): δ = 1.72 (d, J = 6.4 Hz, 3H), 5.50 (q, J = 6.4 Hz, 1H), 7.30-7.36 (m, 3H), 7.50-7.53 (m, 2H), 7.66-7.68 (m, 2H), 7.72-7.75 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 20.3, 85.0, 123.2, 127.5, 128.2, 128.7, 128.8, 134.2, 138.9, 163.7. HRMS (ESI-TOF) Calcd for C₁₆H₁₄NO₃, [M+H]⁺ m/z 268.0974; Found 268.0983.
2-((2,3,3a,7a-tetrahydro-1H-inden-1-yl)oxy)isoindoline-1,3-dione 3o
White solid. mp: 98-99 ºC  ^1H NMR (400 MHz; CDCl$_3$): $\delta$ = 2.30-2.39 (m, 1H), 2.52-2.59 (m, 1H), 2.87-2.94 (m, 1H), 3.32-3.40 (m, 1H), 5.79 (dd, $J_1$ = 1.6 Hz, $J_2$ = 6.0 Hz, 1H), 7.18-7.22 (m, 1H), 7.33 (d, $J$ = 4.0 Hz, 2H), 7.53 (d, $J$ = 7.6 Hz, 1H), 7.73-7.75 (m, 2H), 7.81-7.84 (m, 2H);  $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 30.1, 31.2, 91.7, 123.4, 125.0, 126.3, 126.5, 128.9, 129.9, 134.4, 138.8, 145.9, 164.1. HRMS (ESI-TOF) Calcd for C$_{17}$H$_{16}$NO$_3$, [M+H]$^+$ m/z 282.1130; Found 282.1128.

2-((1,2,3,4,4a,8a-hexahydronaphthalen-1-yl)oxy)isoindoline-1,3-dione 3p
White solid. mp: 101-103 ºC  ^1H NMR (400 MHz; CDCl$_3$): $\delta$ = 1.80-1.88 (m, 2H), 2.30-2.36 (m, 2H), 2.71-2.79 (m, 1H), 2.91-2.97 (m, 1H), 5.31 (t, $J$ = 3.6 Hz, 1H), 7.16 (d, $J$ = 7.6 Hz, 1H), 7.20 (t, $J$ = 7.2 Hz, 1H), 7.25-7.29 (m, 1H), 7.70 (d, $J$ = 7.6 Hz, 1H), 7.72-7.74 (m, 2H), 7.81-7.83 (m, 2H);  $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 17.6, 27.2, 28.9, 83.0, 123.3, 125.8, 128.9, 128.9, 131.3, 131.7, 134.3, 138.7, 164.2. HRMS (ESI-TOF) Calcd for C$_{18}$H$_{18}$NO$_3$, [M+H]$^+$ m/z 296.1287; Found 296.1289.

2-(furan-2-ylmethoxy)isoindoline-1,3-dione 3q
White solid. mp: 132-134 ºC  ^1H NMR (400 MHz; CDCl$_3$): $\delta$ = 5.16 (s, 2H), 6.34-6.35 (m, 1H), 6.49 (d, $J$ = 3.2 Hz, 1H), 7.47 (d, $J$ = 0.8 Hz, 1H), 7.72-7.75 (m, 2H), 7.80-7.82 (m, 2H);  $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 70.3, 110.8, 113.2, 123.5, 128.8, 134.4, 144.5, 148.0, 163.3. HRMS (ESI-TOF) Calcd for C$_{13}$H$_{10}$NO$_4$, [M+H]$^+$ m/z 244.0610; Found 244.0600.

2-(naphthalen-1-ylmethoxy)isoindoline-1,3-dione 3r
White solid. mp: 128-130 ºC  ^1H NMR (400 MHz; CDCl$_3$): $\delta$ = 5.65 (s, 2H), 7.42-7.46 (m, 1H), 7.53-7.57 (m, 1H), 7.62 (d, $J$ = 6.8 Hz, 1H), 7.67 (dd, $J_1$ = 1.2 Hz, $J_2$ = 8.4 Hz, 1H), 7.70-7.72 (m,
2-(1-(p-tolyl)ethoxy)isoindoline-1,3-dione 3s and 2-((4-ethylbenzyl)oxy)isoindoline-1,3-dione 3s’
White solid. mp: 78-80 ºC ¹H NMR (400 MHz; CDCl₃): δ = 1.22 (t, J = 6.4 Hz, 3H), 1.71 (d, J = 6.4 Hz, 3H), 2.31 (s, 3H), 2.65 (q, J = 7.6 Hz, 2H), 5.19 (s, 2H), 5.48 (q, J = 6.4 Hz, 1H), 7.14 (d, J = 7.6 Hz, 2H), 7.21 (d, J = 8.0 Hz, 2H), 7.40 (d, J = 8.0 Hz, 2H), 7.46 (d, J = 8.0 Hz, 2H), 7.67-7.70 (m, 2H), 7.71-7.75 (m, 4H), 7.79-7.81 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 15.3, 20.3, 21.1, 28.6, 79.7, 84.8, 123.2, 123.3, 127.5, 127.9, 128.7, 128.8, 129.9, 130.8, 134.1, 134.3, 135.9, 138.7, 145.4, 163.4, 163.7. HRMS (ESI-TOF) Calcd for C₁₇H₁₄NO₃, [M+H]⁺ m/z 282.1130; Found 282.1126.

2-(cinnamyloxy)isoindoline-1,3-dione 5a
White solid. mp: 118-120 ºC ¹H NMR (400 MHz; CDCl₃): δ = 4.86 (d, J = 6.8 Hz, 2H), 6.42-6.50 (m, 1H), 6.67 (d, J = 16 Hz, 1H), 7.23-7.32 (m, 3H), 7.37 (d, J = 6.8 Hz, 2H), 7.70-7.72 (m, 2H), 7.79-7.81 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 78.6, 122.0, 123.5, 126.8, 128.4, 128.6, 128.7, 134.4, 135.7, 137.4, 163.4. HRMS (ESI-TOF) Calcd for C₁₇H₁₄NO₃, [M+H]⁺ m/z 280.0974; Found 280.0968.

(E)-2-((3-(2-methoxyphenyl)allyl)oxy)isoindoline-1,3-dione 5b
White solid. mp: 119-120 ºC ¹H NMR (400 MHz; CDCl₃): δ = 3.74 (s, 3H), 4.86 (dd, J₁ = 0.9 Hz, J₂ = 7.2 Hz, 2H), 6.41-6.49 (m, 1H), 6.80 (d, J = 8.4 Hz, 1H), 6.88-6.98 (m, 2H), 7.19-7.24 (m, 1H), 7.42 (dd, J₁ = 1.6 Hz, J₂ = 8.0 Hz, 1H), 7.68-7.70 (m, 2H), 7.77-7.80 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 55.2, 79.0, 110.6, 120.1, 122.4, 123.3, 124.7, 127.3, 128.8, 129.4, 132.6, 134.3, 156.8, 163.7. HRMS (ESI-TOF) Calcd for C₁₈H₁₆NO₄, [M+H]⁺ m/z 310.1079; Found 310.1085.
(E)-2-((3-(4-methoxyphenyl)allyl)oxy)isoindoline-1,3-dione 5c
White solid. mp: 133-135 °C
^1H NMR (400 MHz; CDCl$_3$): δ = 3.79 (s, 3H), 4.84 (d, J = 7.2 Hz, 2H), 6.28-6.36 (m, 1H), 6.61 (d, J = 15.6 Hz, 1H), 6.83 (d, J = 8.0 Hz, 2H), 7.31 (d, J = 8.4 Hz, 2H), 7.70-7.73 (m, 2H), 7.80-7.82 (m, 2H); ^13C NMR (100 MHz, CDCl$_3$): δ = 55.3, 78.9, 114.0, 119.6, 123.5, 128.2, 128.5, 128.9, 134.4, 137.3, 159.9, 163.8. HRMS (ESI-TOF) Calcd for C$_{18}$H$_{16}$NO$_4$, [M+H]$^+$ m/z 310.1079; Found 310.1072.

(E)-2-((3-(3,4-dimethoxyphenyl)allyl)oxy)isoindoline-1,3-dione 5d
White solid. mp: 136-137 °C
^1H NMR (400 MHz; CDCl$_3$): δ = 3.87 (s, 3H), 3.89 (s, 3H), 4.85 (d, J = 7.2 Hz, 2H), 6.30-6.37 (m, 1H), 6.61 (d, J = 16.0 Hz, 1H), 6.79 (d, J = 8.0 Hz, 1H), 6.90-6.93 (m, 2H), 7.71-7.73 (m, 2H), 7.80-7.82 (m, 2H); ^13C NMR (100 MHz, CDCl$_3$): δ = 55.8, 55.9, 78.8, 109.1, 111.0, 119.9, 120.4, 123.5, 128.9, 134.4, 137.5, 149.0, 149.5, 163.8. HRMS (ESI-TOF) Calcd for C$_{19}$H$_{18}$NO$_5$, [M+H]$^+$ m/z 340.1185; Found 340.1191.

2-(cyclohex-2-en-1-yloxy)isoindoline-1,3-dione 5e
White solid. mp: 65-66 °C
^1H NMR (400 MHz; CDCl$_3$): δ = 1.56-1.62 (m, 1H), 1.79-1.84 (m, 1H), 1.92-2.01 (m, 3H), 2.08-2.16 (m, 1H), 4.71-4.72 (m, 1H), 5.88-5.92 (m, 1H), 6.03-6.07 (m, 1H), 7.71-7.73 (m, 2H), 7.79-7.81 (m, 2H); ^13C NMR (100 MHz, CDCl$_3$): δ = 18.1, 25.1, 27.1, 80.8, 123.3, 123.7, 128.9, 134.3, 134.9, 164.2. HRMS (ESI-TOF) Calcd for C$_{14}$H$_{14}$NO$_3$, [M+H]$^+$ m/z 244.0974; Found 244.0977.

2-(cyclopent-2-en-1-yloxy)isoindoline-1,3-dione 5f
White solid. mp: 93-94 °C
^1H NMR (400 MHz; CDCl$_3$): δ = 2.17-2.37 (m, 3H), 2.57-2.65 (m, 1H), 5.41-5.43 (m, 1H), 5.96-5.98 (m, 1H), 6.24-6.26 (m, 1H), 7.74-7.76 (m, 2H), 7.83-7.85 (m, 2H); ^13C NMR (100 MHz, CDCl$_3$): δ = 28.9, 31.3, 94.1, 123.4, 128.1, 129.0, 134.4, 140.4, 164.4. HRMS (ESI-TOF) Calcd for C$_{13}$H$_{12}$NO$_3$, [M+H]$^+$ m/z 230.0817; Found 230.0809.
Phenylmethanol 6a
Colorless liquid. $^1$H NMR (400 MHz; CDCl$_3$): $\delta = 2.27$ (s, 1H), 4.65 (d, $J = 1.2$ Hz, 2H), 7.29-7.39 (m, 5H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 65.2$, 126.9, 127.5, 128.5, 140.8. HRMS (ESI-TOF) Calcd for C$_7$H$_9$O, [M+H]$^+$ m/z 109.0653; Found 109.0655.

1-phenylethanol 6b
Colorless liquid. $^1$H NMR (400 MHz; CDCl$_3$): $\delta = 1.50$ (d, $J = 6.4$ Hz, 3H), 1.90 (s, 1H), 4.90 (q, $J = 6.4$ Hz, 1H), 7.28-7.30 (m, 1H), 7.33-7.40 (m, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 25.1$, 70.4, 125.4, 127.5, 128.5, 145.8. HRMS (ESI-TOF) Calcd for C$_8$H$_{11}$O, [M+H]$^+$ m/z 123.0810; Found 123.0815.

O-benzylhydroxylamine 7a
Colorless liquid. $^1$H NMR (400 MHz; CDCl$_3$): $\delta = 4.70$ (s, 2H), 5.40 (s, 2H), 7.30-7.45 (m, 5H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 77.9$, 127.9, 128.3, 128.4, 137.4. HRMS (ESI-TOF) Calcd for C$_7$H$_{10}$NO, [M+H]$^+$ m/z 124.0762; Found 124.0764.

O-(1-phenylethyl)hydroxylamine 7b
Colorless liquid. $^1$H NMR (400 MHz; CDCl$_3$): $\delta = 1.44$ (d, $J = 6.8$ Hz, 3H), 4.66 (q, $J = 6.4$ Hz, 1H), 5.22 (s, 2H), 7.28-7.40 (m, 5H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 21.8$, 82.8, 126.3, 127.7, 128.5, 143.0. HRMS (ESI-TOF) Calcd for C$_8$H$_{12}$NO, [M+H]$^+$ m/z 138.0919; Found 138.0923.

2,2,6,6-tetramethyl-1-(1-phenylethoxy)piperidine 8
Colorless liquid. $^1$H NMR (400 MHz; CDCl$_3$): $\delta = 0.67$ (s, 3H), 1.04 (s, 3H), 1.18 (s, 3H), 1.23-1.38 (m, 6H), 1.49 (s, 4H), 1.50 (s, 2H), 4.79 (q, $J = 6.8$ Hz, 1H), 7.21-7.26 (m, 1H), 7.29-7.33 (m, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 17.2$, 20.3, 23.5, 34.1, 34.4, 40.4, 59.7, 83.1, 126.6, 126.7, 127.9, 145.8. HRMS (ESI-TOF) Calcd for C$_{17}$H$_{18}$NO, [M+H]$^+$ m/z 262.2171; Found 262.2170.

References:
VII. $^1$H and $^{13}$C Spectra of Compounds 3, 5, 6, 7 and 8

Product 3a
Product 3b
Product 3c
Product 3d
Product 3e
Product 3h
Product 3i
Product 3j
Product 3m
Product 3n
Product 3o
Product 3p
Product 3q
Product 3r
Product 3s and Product 3s’
Product 5a
Product 5b
Product 5e
Product 5f
Product 6b
Product 7a
Product 8