Supplementary Information

For

Metal-free Intermolecular C-O Cross-Coupling Reactions: Synthesis of N-hydroxyimide esters

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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. General procedure for the preparation of 3

3a as an example

$$\text{Cl} \quad + \quad \text{OH} \quad \overset{\text{selectfluor}}{\underset{\text{CH}_3\text{CN, 90 ºC}}{\rightarrow}} \quad \text{Cl} \quad \text{N-O-C} \quad \text{H} \quad \text{N} \quad 3a$$

To a solution of the N-Hydroxyphthalimide (NHPI) 2 (58.7 mg, 0.36 mmol) in acetonitrile (3.0 ml) was added the 4-chlorobenzaldehyde 1a (35 μL, 0.3 mmol) and selectfluor (127.5 mg, 0.36 mmol) in a sealed tube. The sealed tube was then tightly sealed with a screw cap and the reaction was stirred for the 3.0 h at 90 ºC. After the reaction finished, the reaction mixture was cooled to room temperature. 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 (82.3 mg, 91%).

III. General procedure for the preparation of 5

3a as an example

$$\text{F} \quad + \quad \text{OH} \quad \overset{\text{selectfluor}}{\underset{\text{CH}_3\text{CN, 90 ºC}}{\rightarrow}} \quad \text{F} \quad \text{N-O-C} \quad \text{H} \quad \text{N} \quad 5a$$

To a solution of the N-Hydroxysuccinimide 4 (NHSI, 41.4 mg, 0.36 mmol) in acetonitrile (3.0 ml) was added the 2-fluorobenzaldehyde 1c (32 μL, 0.3 mmol) and selectfluor (127.5 mg, 0.36 mmol)
in a sealed tube. The sealed tube was then tightly sealed with a screw cap and the reaction was stirred for the 1.0 h at 90 ºC. After the reaction finished, the reaction mixture was cooled to room temperature. 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 column chromatography to give the corresponding products 5a (50.5 mg, 71%).

**IV. General procedure for the preparation of 7**

**5a as an example**

To a solution of 3a (60.3 mg, 0.2 mmol) in EtOAc (2.0 mL) was added propan-1-amine 6a (49.2 μL, 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 crude mixture was concentrated and purified by column chromatography to afford the desired products 7a (32.8 mg, 83%).

**V. Analytical data of Compounds 3, 5 and 7**

**1,3-dioxoisindolin-2-yl methyl terephthalate 3a**

White solid. mp: 182–183 ºC. ¹H NMR (400 MHz; CDCl₃): δ = 7.52 (d, J = 8.8 Hz, 2H), 7.81 (dd, J₁ = 3.2 Hz, J₂ = 5.6 Hz, 2H), 7.92 (dd, J₁ = 3.2 Hz, J₂ = 5.6 Hz, 2H), 8.13 (d, J = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 123.7, 124.0, 128.9, 129.3, 131.9, 134.8, 141.6, 161.9, 162.0. HRMS (ESI-TOF) Calcd for C₁₅H₉ClNO₄, [M+H]⁺ m/z 302.0220; Found 302.0208.

**1,3-dioxoisindolin-2-yl benzoate 3b**

White solid. mp: 98–101 ºC. ¹H NMR (400 MHz; CDCl₃): δ = 7.54 (t, J = 7.6 Hz, 2H), 7.70 (t, J = 7.6 Hz, 2H), 7.81 (dd, J₁ = 3.2 Hz, J₂ = 5.2 Hz, 2H), 7.92 (dd, J₁ = 3.2 Hz, J₂ = 5.2 Hz, 2H), 8.19 (d, J = 7.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 124.0, 125.3, 128.8, 129.0, 130.6, 134.8, 134.8, 162.0, 162.8. HRMS (ESI-TOF) Calcd for C₁₅H₁₀NO₅, [M+H]⁺ m/z 268.0610; Found 268.0615.
1,3-dioxoisooindolin-2-yl 2-fluorobenzoate 3c[2]
White solid. mp: 173-175 °C \( ^1H \) NMR (400 MHz; CDCl\(_3\)): \( \delta = 7.22-7.34 \) (m, 2H), 7.67-7.69 (m, 1H), 7.81 (dd, \( J_1 = 3.2 \) Hz, \( J_2 = 5.6 \) Hz, 2H), 7.92 (dd, \( J_1 = 3.2 \) Hz, \( J_2 = 5.6 \) Hz, 2H), 8.12-8.15 (m, 1H); \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)): \( \delta = 113.9, 114.0, 117.3, 117.5, 124.0, 124.4, 124.5, 129.0, 132.7, 134.8, 136.7, 136.8, 160.2, 160.3, 161.2, 161.8, 163.8 \). HRMS (ESI-TOF) Calcd for C\(_{15}\)H\(_9\)FNO\(_4\), [M+H]\(^+\) m/z 286.0516; Found 286.0531.

1,3-dioxoisooindolin-2-yl 2-chlorobenzoate 3d[1]
White solid. mp: 134-135 °C \( ^1H \) NMR (400 MHz; CDCl\(_3\)): \( \delta = 7.41-7.45 \) (m, 1H), 7.57 (d, \( J = 5.6 \)Hz, 2H), 7.81-7.83 (m, 2H), 7.93 (dd, \( J_1 = 3.2 \) Hz, \( J_2 = 5.6 \) Hz, 2H), 8.18 (d, \( J = 8.0 \)Hz, 1H); \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)): \( \delta = 124.0, 124.8, 126.9, 129.0, 131.6, 132.4, 134.6, 134.8, 135.6, 161.2, 161.9 \). HRMS (ESI-TOF) Calcd for C\(_{15}\)H\(_9\)ClNO\(_4\), [M+H]\(^+\) m/z 302.0220; Found 302.0224.

1,3-dioxoisooindolin-2-yl 2-methylbenzoate 3e
White solid. mp: 139-140 °C \( ^1H \) NMR (400 MHz; CDCl\(_3\)): \( \delta = 2.64 \) (s, 3H), 7.33-7.37 (m, 2H), 7.52-7.56 (m, 1H), 7.80-7.82 (m, 2H), 7.93 (dd, \( J_1 = 3.2 \) Hz, \( J_2 = 5.6 \) Hz, 2H), 8.19-8.21 (m, 1H); \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)): \( \delta = 21.6, 124.0, 124.4, 126.1, 129.1, 131.4, 132.0, 133.9, 142.1, 162.2, 163.1 \). HRMS (ESI-TOF) Calcd for C\(_{16}\)H\(_{12}\)NO\(_4\), [M+H]\(^+\) m/z 282.0766; Found 282.0761.

1,3-dioxoisooindolin-2-yl 2-methoxybenzoate 3f
White solid. mp: 159-160 °C \( ^1H \) NMR (400 MHz; CDCl\(_3\)): \( \delta = 3.93 \) (s, 3H), 7.03-7.07 (m, 2H), 7.59-7.63 (m, 1H), 7.79 (dd, \( J_1 = 3.2 \) Hz, \( J_2 = 5.6 \) Hz, 2H), 7.91 (dd, \( J_1 = 3.2 \) Hz, \( J_2 = 5.6 \) Hz, 2H), 8.11 (dd, \( J_1 = 1.6 \) Hz, \( J_2 = 8.0 \) Hz, 1H); \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)): \( \delta = 56.1, 112.2, 114.2, 120.3, 123.9, 129.1, 132.7, 134.6, 135.9, 160.6, 161.3, 162.3 \). HRMS (ESI-TOF) Calcd for C\(_{16}\)H\(_{13}\)NO\(_5\), [M+H]\(^+\) m/z 298.0715; Found 298.0726.
1,3-dioxoisindolin-2-yl 3-fluorobenzoate 3g
White solid. mp: 162−163 °C 1H NMR (400 MHz; CDCl3): δ = 7.37-7.43 (m, 1H), 7.50-7.56 (m, 1H), 7.81 (dd, J1 = 3.2 Hz, J2 = 5.6 Hz, 2H), 7.86 (d, J = 8.0 Hz, 1H), 7.92 (dd, J1 = 3.2 Hz, J2 = 5.6 Hz, 2H), 7.99 (d, J = 7.6 Hz, 1H); 13C NMR (100 MHz, CDCl3): δ = 117.3, 117.6, 122.0, 122.2, 124.0, 126.4, 127.2, 127.3, 128.9, 130.6, 130.7, 134.8, 161.2, 161.8, 163.7. HRMS (ESI-TOF) Calcd for C15H9FNO4, [M+H]+ m/z 286.0516; Found 286.0531.

1,3-dioxoisindolin-2-yl 3-chlorobenzoate 3h
White solid. mp: 176−178 °C 1H NMR (400 MHz; CDCl3): δ = 7.49 (t, J = 8.0 Hz 1H), 7.67 (d, J = 8.0 Hz, 1H), 7.81 (dd, J1 = 3.2 Hz, J2 = 5.2 Hz, 2H), 7.92 (dd, J1 = 3.2 Hz, J2 = 5.2 Hz, 2H), 8.07 (d, J = 8.0 Hz, 1H) 8.15 (s, 1H); 13C NMR (100 MHz, CDCl3): δ = 124.0, 126.9, 128.7, 128.8, 130.2, 130.5, 134.9, 134.9, 135.1, 161.7, 161.8. HRMS (ESI-TOF) Calcd for C15H9ClNO4, [M+H]+ m/z 302.0220.; Found 302.0204.

1,3-dioxoisindolin-2-yl 3-bromobenzoate 3i
White solid. mp: 209−210 °C 1H NMR (400 MHz; CDCl3): δ = 7.42 (t, J = 8.0 Hz 1H), 7.80-7.82 (m, 3H), 7.90-7.92 (m, 2H), 8.11 (d, J = 7.6 Hz, 1H), 8.30 (s, 1H); 13C NMR (100 MHz, CDCl3): δ = 122.8, 124.0, 127.1, 128.8, 129.1, 130.4, 133.3, 134.8, 137.8, 161.6, 161.8. HRMS (ESI-TOF) Calcd for C15H9BrNO4, [M+H]+ m/z 345.9715.; Found 345.9731.

1,3-dioxoisindolin-2-yl 3-nitrobenzoate 3j
White solid. mp: 214−215 °C 1H NMR (400 MHz; CDCl3): δ = 7.79 (t, J = 8.0 Hz 1H), 7.85 (dd, J1 = 3.2 Hz, J2 = 5.6 Hz, 2H), 7.96 (dd, J1 = 3.2 Hz, J2 = 5.2 Hz, 2H), 8.51-8.53 (m, 1H), 8.56-8.58 (m, 1H), 9.04-9.05 (m, 1H); 13C NMR (100 MHz, CDCl3): δ = 124.2, 125.6, 127.2, 128.9, 129.2, 130.3, 135.0, 136.0, 148.5, 161.1, 161.6. HRMS (ESI-TOF) Calcd for C15H9N2O6, [M+H]+ m/z 313.0461.; Found 313.0444.
1,3-dioxoisindolin-2-yl 3-methylbenzoate 3k
White solid. mp: 141–142 °C. $^1$H NMR (400 MHz; CDCl$_3$): $\delta$ = 2.44 (s, 3H), 7.42 (t, $J$ = 7.6 Hz, 1H), 7.50 (d, $J$ = 7.6 Hz, 1H), 7.81 (dd, $J_1$ = 3.2 Hz, $J_2$ = 5.6 Hz, 2H), 7.92 (dd, $J_1$ = 3.2 Hz, $J_2$ = 5.6 Hz, 2H), 8.00 (d, $J$ = 8.8 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 21.2, 124.0, 125.2, 127.8, 128.7, 129.1, 131.0, 134.7, 135.6, 138.8, 162.1, 162.9. HRMS (ESI-TOF) Calcd for C$_{16}$H$_{12}$NO$_4$, [M+H]$^+$ m/z 282.0766; Found 282.0771.

1,3-dioxoisindolin-2-yl 3-methoxybenzoate 3l
White solid. mp: 132–134 °C. $^1$H NMR (400 MHz; CDCl$_3$): $\delta$ = 3.87 (s, 3H), 7.21–7.24 (m, 1H), 7.43 (t, $J$ = 8.0 Hz 1H), 7.65–7.66 (m, 1H), 7.78–7.82 (m, 3H), 7.90–7.92 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 55.5, 114.7, 121.6, 123.0, 124.0, 126.3, 129.0, 129.9, 134.8, 159.7, 162.0, 162.7. HRMS (ESI-TOF) Calcd for C$_{16}$H$_{12}$NO$_5$, [M+H]$^+$ m/z 298.0715; Found 298.0721.

1,3-dioxoisindolin-2-yl 4-fluorobenzoate 3m
White solid. mp: 194–195 °C. $^1$H NMR (400 MHz; CDCl$_3$): $\delta$ = 7.19–7.25 (m, 2H), 7.79–7.82 (m, 2H), 7.90–7.92 (m, 2H), 8.20–8.24 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 116.2, 116.4, 121.5, 124.0, 129.0, 133.4, 133.5, 134.8, 161.8, 162.0, 165.6, 168.1. HRMS (ESI-TOF) Calcd for C$_{15}$H$_9$FNO$_4$, [M+H]$^+$ m/z 286.0516; Found 286.0513.

1,3-dioxoisindolin-2-yl 4-bromobenzoate 3n
White solid. mp: 190–192 °C. $^1$H NMR (400 MHz; CDCl$_3$): $\delta$ = 7.68 (dd, $J_1$ = 1.6 Hz, $J_2$ = 6.8 Hz, 2H), 7.80–7.82 (m, 2H), 7.92 (dd, $J_1$ = 3.2 Hz, $J_2$ = 5.6 Hz, 2H), 8.04 (dd, $J_1$ = 2.0 Hz, $J_2$ = 6.8 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 124.0, 124.1, 128.9, 130.4, 131.9, 132.3, 134.8, 161.9, 162.0. HRMS (ESI-TOF) Calcd for C$_{15}$H$_9$BrNO$_4$, [M+H]$^+$ m/z 345.9715; Found 345.9725.

1,3-dioxoisindolin-2-yl 4-cyanobenzoate 3o
White solid. mp: 219–221 °C. $^1$H NMR (400 MHz; CDCl$_3$): $\delta$ = 7.82–7.86 (m, 4H), 7.94 (d, $J_1$ = 3.2 Hz, $J_2$ = 5.6 Hz, 2H), 8.30 (d, $J$ = 8.4 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ = 117.4, 118.2, 124.2, 128.8, 129.2, 131.0, 132.6, 135.0, 161.5, 161.6. HRMS (ESI-TOF) Calcd for C$_{16}$H$_9$N$_2$O$_4$, [M+H]$^+$ m/z 293.0562; Found 293.0552.
1,3-dioxoisoindolin-2-yl methyl terephthalate 3p
White solid. mp: 177–178 °C. 1H NMR (400 MHz; CDCl₃): δ = 3.95 (s, 3H), 7.80 (d, J = 2.8 Hz, 2H), 7.89 (d, J = 2.8 Hz, 2H), 8.16 (d, J = 8.4 Hz, 2H), 8.23 (d, J = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 52.5, 124.0, 128.8, 128.9, 129.8, 130.5, 134.8, 135.5, 161.7, 162.1, 165.7. HRMS (ESI-TOF) Calcd for C₁₇H₁₂NO₆, [M+H]^+ m/z 326.0665; Found 326.0657.

1,3-dioxoisoindolin-2-yl 4-methylbenzoate 3q
White solid. mp: 167–168 °C. 1H NMR (400 MHz; CDCl₃): δ = 2.47 (s, 3H), 7.33 (d, J = 8.0 Hz, 2H), 7.81 (dd, J₁ = 3.2 Hz, J₂ = 5.2 Hz, 2H), 7.92–7.94 (m, 2H), 8.08 (d, J = 8.0 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 21.9, 122.5, 124.0, 129.1, 129.6, 130.7, 134.7, 146.0, 162.1, 162.8. HRMS (ESI-TOF) Calcd for C₁₆H₁₂NO₄, [M+H]^+ m/z 282.0766; Found 282.0772.

1,3-dioxoisoindolin-2-yl 4-methoxybenzoate 3r
White solid. mp: 165–166 °C. ¹H NMR (400 MHz; CDCl₃): δ = 3.89 (s, 3H), 6.99 (d, J = 8.8 Hz, 2H), 7.79–7.81 (m, 2H), 7.90–7.91 (m, 2H), 8.14 (d, J = 8.8 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 55.6, 114.2, 117.2, 123.9, 129.0, 132.9, 134.7, 162.2, 162.4, 164.9. HRMS (ESI-TOF) Calcd for C₁₆H₁₂NO₅, [M+H]^+ m/z 298.0715; Found 298.0721.

1,3-dioxoisoindolin-2-yl 2,4-dichlorobenzoate 3s
White solid. mp: 180–182 °C. ¹H NMR (400 MHz; CDCl₃): δ = 7.42 (d, J = 8.4 Hz, 1H), 7.58 (s, 1H), 7.82 (d, J = 3.2 Hz, 2H), 7.92 (dd, J₁ = 3.2 Hz, J₂ = 4.8 Hz, 2H), 8.13 (d, J = 8.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 123.1, 124.1, 127.4, 128.9, 131.7, 133.4, 134.9, 136.8, 140.8, 160.4, 161.8. HRMS (ESI-TOF) Calcd for C₁₅H₈Cl₂NO₄, [M+H]^+ m/z 335.9830; Found 335.9835.

1,3-dioxoisoindolin-2-yl 1-naphthoate 3t
White solid. mp: 180–182 °C. ¹H NMR (400 MHz; CDCl₃): δ = 7.57–7.62 (m, 2H), 7.64–7.69 (m,
1H), 7.81-7.83 (m, 2H), 7.92-7.96 (m, 3H), 8.16 (d, J = 8.0 Hz, 1H), 8.54 (dd, J1 = 1.2 Hz, J2 = 7.2 Hz, 1H), 8.85 (d, J = 8.4 Hz, 1H); 13C NMR (100 MHz, CDCl3): δ = 121.7, 124.0, 124.5, 125.3, 126.8, 128.7, 128.9, 129.1, 131.5, 131.9, 133.7, 134.8, 135.5 162.3 163.1. HRMS (ESI-TOF) Calcd for C19H12NO4, [M+H]+ m/z 318.0766; Found 318.0760.

1,3-dioxoisooindolin-2-yl cinnamate 3u[2]
White solid. mp: 56−57 ºC 1H NMR (400 MHz; CDCl3): δ = 6.67 (d, J = 16.0 Hz, 1H), 7.43-7.47 (m, 3H), 7.59-7.61 (m, 2H), 7.80-7.82 (m, 2H), 7.92 (dd, J1 = 3.2 Hz, J2 = 5.2 Hz, 2H), 7.97(d, J = 16.0Hz, 1H); 13C NMR (100 MHz, CDCl3): δ = 111.8, 124.0, 128.7, 129.1, 129.1, 131.5, 133.6, 134.7, 150.0, 162.1, 163.0. HRMS (ESI-TOF) Calcd for C17H12NO4, [M+H]+ m/z 294.0766; Found 294.0754.

1,3-dioxoisooindolin-2-yl 3-phenylpropanoate 3v
White solid. mp: 66−67 ºC 1H NMR (400 MHz; CDCl3): δ = 3.00-3.02 (m, 2H), 3.10-3.14 (m, 2H), 7.27-7.37 (m, 5H), 7.79-7.81 (m, 2H), 7.90 (dd, J1 = 3.2 Hz, J2 = 5.2 Hz, 2H); 13C NMR (100 MHz, CDCl3): δ = 30.5, 32.7, 123.9, 126.7, 128.3, 128.9, 134.7, 139.1, 161.8, 168.8. HRMS (ESI-TOF) Calcd for C17H14NO4, [M+H]+ m/z 296.0923; Found 296.0917.

1,3-dioxoisooindolin-2-yl propionate 3w
White solid. mp: 86−88 ºC 1H NMR (400 MHz; CDCl3): δ = 1.31 (t, J = 7.6 Hz, 3H), 2.71 (q, J = 7.6Hz, 2H), 7.79 (dd, J1 = 3.2 Hz, J2 = 5.6 Hz, 2H), 7.89 (dd, J1 = 3.2 Hz, J2 = 5.6 Hz, 2H); 13C NMR (100 MHz, CDCl3): δ = 8.7, 24.5, 123.9, 129.0, 134.7, 162.0, 170.3. HRMS (ESI-TOF) Calcd for C11H10NO4, [M+H]+ m/z 220.0610; Found 220.0608.

1,3-dioxoisooindolin-2-yl butyrate 3x
Colorless oil 1H NMR (400 MHz; CDCl3): δ = 1.08 (t, J = 7.2Hz, 3H), 1.78-1.88 (m, 2H), 2.65 (t, J = 7.2Hz, 2H), 7.79 (dd, J1 = 3.2 Hz, J2 = 5.6 Hz, 2H), 7.89 (dd, J1 = 3.2 Hz, J2 = 5.6 Hz, 2H); 13C NMR (100 MHz, CDCl3): δ = 13.4, 18.3, 32.8, 123.9, 129.0, 134.7, 162.0, 169.5. HRMS (ESI-TOF) Calcd for C12H12NO4, [M+H]+ m/z 234.0766; Found 234.0761.
1,3-dioxoisindolin-2-yl pentanoate 3y
White solid. mp: 55–56 °C ¹H NMR (400 MHz; CDCl₃): δ = 0.97 (t, J = 7.2 Hz, 3H), 1.45-1.50 (m, 2H), 1.73-1.81 (m, 2H), 2.67 (t, J = 7.2Hz, 2H), 7.77-7.92 (m, 2H), 7.87-7.89 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 13.6, 22.0, 26.7, 30.7, 123.9, 129.0, 134.7, 162.0, 169.6. HRMS (ESI-TOF) Calcd for C₁₃H₁₄NO₄, [M+H]⁺ m/z 248.0923; Found 248.0919.

2,5-dioxopyrrolidin-1-yl 2-fluorobenzoate 5a
White solid. mp: 113–114 °C ¹H NMR (400 MHz; CDCl₃): δ = 2.90 (s, 4H), 7.19-7.30 (m, 2H), 7.65-7.67 (m, 1H), 8.04-8.08 (m, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 25.6, 113.6, 113.7, 117.2, 117.4, 124.4, 124.4, 132.5, 136.7, 136.8, 159.2, 159.3, 161.1, 163.7, 169.1. HRMS (ESI-TOF) Calcd for C₁₁H₉FNO₄, [M+H]⁺ m/z 238.0516; Found 238.0508.

2,5-dioxopyrrolidin-1-yl 2-methoxybenzoate 5b
White solid. mp: 177–178 °C ¹H NMR (400 MHz; CDCl₃): δ = 2.88 (s, 4H), 3.92 (s, 3H), 7.01-7.04 (m, 2H), 7.57-7.61 (m, 1H), 8.03-8.05 (m, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 25.7, 56.1, 112.2, 114.1, 120.3, 132.7, 135.9, 160.3, 160.6, 169.4. HRMS (ESI-TOF) Calcd for C₁₂H₁₂NO₅, [M+H]⁺ m/z 250.0715; Found 250.0721.

2,5-dioxopyrrolidin-1-yl 3-fluorobenzoate 5c
White solid. mp: 147–148 °C ¹H NMR (400 MHz; CDCl₃): δ = 2.91 (s, 4H), 7.37-7.41 (m, 1H), 7.48-7.54 (m, 1H), 7.81-7.83 (m, 1H), 7.94 (d, J = 7.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 25.7, 117.3, 117.6, 122.0, 122.2, 126.4, 126.4, 127.1, 127.2, 130.6, 130.7, 160.9, 160.9, 161.3, 163.7, 169.0. HRMS (ESI-TOF) Calcd for C₁₁H₉FNO₄, [M+H]⁺ m/z 238.0516; Found 238.0510.

2,5-dioxopyrrolidin-1-yl 3-bromobenzoate 5d
White solid. mp: 152–153 °C ¹H NMR (400 MHz; CDCl₃): δ = 2.91 (s, 4H), 7.38-7.42 (m, 1H), 7.80-7.82 (m, 1H), 8.07 (d, J = 8.0 Hz, 1H), 8.27 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 25.7, 122.9, 127.1, 129.1, 130.4, 133.4, 137.9, 160.7, 168.9. HRMS (ESI-TOF) Calcd for C₁₁H₈BrNO₄,
2,5-dioxopyrrolidin-1-yl 3-nitrobenzoate 5e
White solid. mp: 137–138 °C ¹H NMR (400 MHz; CDCl₃): δ = 2.96 (s, 4H), 7.76-7.80 (m, 1H), 8.47 (d, J = 7.6 Hz, 1H), 8.56 (d, J = 8.0 Hz, 1H), 8.99 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 25.7, 125.5, 127.1, 129.2, 130.3, 135.9, 148.4, 160.1, 168.7. HRMS (ESI-TOF) Calcd for C₁₁H₉N₂O₆, [M+H]+ m/z 265.0461; Found 265.0455.

2,5-dioxopyrrolidin-1-yl 3-methoxybenzoate 5f
White solid. mp: 106–107 °C ¹H NMR (400 MHz; CDCl₃): δ = 2.89 (s, 4H), 3.85 (s, 3H), 7.19-7.22 (m, 1H), 7.41 (t, J = 8.0 Hz, 1H), 7.60 (s, 1H), 7.73 (d, J = 7.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 25.6, 55.5, 114.6, 121.6, 123.0, 126.2, 129.9, 159.7, 161.8, 169.2. HRMS (ESI-TOF) Calcd for C₁₂H₁₂NO₅, [M+H]+ m/z 250.0715; Found 250.0711.

2,5-dioxopyrrolidin-1-yl 4-chlorobenzoate 5g
White solid. mp: 207–208 °C ¹H NMR (400 MHz; CDCl₃): δ = 2.90 (s, 4H), 7.49 (d, J = 8.4 Hz, 2H), 8.06 (d, J = 8.8 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 25.6, 123.6, 129.3, 131.9, 141.7, 161.1, 169.1. HRMS (ESI-TOF) Calcd for C₁₁H₉ClNO₄, [M+H]+ m/z 254.0220; Found 254.0225.

2,5-dioxopyrrolidin-1-yl 4-cyanobenzoate 5h
White solid. mp: 223–224 °C ¹H NMR (400 MHz; CDCl₃): δ = 2.92 (s, 4H), 7.82 (d, J = 8.0 Hz, 2H), 8.23 (d, J = 8.0 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 25.6, 117.3, 118.3, 129.1, 131.0, 132.6, 160.5, 168.7. HRMS (ESI-TOF) Calcd for C₁₂H₉N₂O₄, [M+H]+ m/z 245.0562; Found 245.0565.
2,5-dioxopyrrolidin-1-yl methyl terephthalate 5i
White solid. mp: 170–171 °C. 1H NMR (400 MHz; CDCl₃): δ = 2.92 (s, 4H), 3.97 (s, 3H), 8.16-8.22 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ = 25.7, 52.6, 128.9, 129.9, 130.5, 135.7, 161.2, 165.8, 168.9. HRMS (ESI-TOF) Calcd for C₁₃H₁₂NO₆, [M+H]⁺ m/z 278.0665; Found 278.0661.

2,5-dioxopyrrolidin-1-yl 4-methylbenzoate 5j
White solid. mp: 180–181 °C. 1H NMR (400 MHz; CDCl₃): δ = 2.42 (s, 3H), 2.87 (s, 4H), 7.29 (d, J = 8.0 Hz, 2H), 8.00 (d, J = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 21.8, 25.6, 122.2, 129.5, 130.5, 146.0, 161.8, 169.3. HRMS (ESI-TOF) Calcd for C₁₂H₁₂NO₄, [M+H]⁺ m/z 234.0766; Found 234.0770.

4-chloro-N-propylbenzamide 7a
White solid. mp: 98–101 °C. 1H NMR (400 MHz; CDCl₃): δ = 0.96 (t, J = 7.2 Hz, 3H), 1.57-1.66 (m, 2H), 3.38 (q, J = 6.8 Hz, 2H), 6.38 (s, 1H), 7.36 (d, J = 8.4 Hz, 2H), 7.69 (d, J = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 11.4, 22.8, 41.8, 128.3, 128.7, 133.2, 137.4, 166.5. HRMS (ESI-TOF) Calcd for C₁₀H₁₃ClNO, [M+H]⁺ m/z 198.0686; Found 198.0682.

N-benzyl-4-chlorobenzamide 7b
White solid. mp: 161–164 °C. 1H NMR (400 MHz; CDCl₃): δ = 4.60 (d, J = 5.6 Hz, 2H), 6.58 (s, 1H), 7.29-7.38 (m, 7H), 7.71 (d, J = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 44.2, 127.7, 127.9, 128.4, 128.8, 132.7, 137.7, 137.9, 166.3. HRMS (ESI-TOF) Calcd for C₁₄H₁₃ClNO, [M+H]⁺ m/z 246.0684; Found 246.0684.

4-chloro-N-phenethylbenzamide 7c
White solid. mp: 129–130 °C. 1H NMR (400 MHz; CDCl₃): δ = 2.92 (t, J = 6.8 Hz, 2H), 3.67-3.71 (m, 2H), 6.31 (s, 1H), 7.20-7.24 (m, 3H), 7.30-7.37 (m, 4H), 7.62 (d, J = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 35.6, 41.2, 126.6, 128.2, 128.7, 128.7, 133.0, 137.6, 138.7, 166.4. HRMS (ESI-TOF) Calcd for C₁₅H₁₅ClNO, [M+H]⁺ m/z 260.0842; Found 260.0839.

References
VI. $^1$H and $^{13}$C Spectra of Compounds 3, 5 and 7

Product 3a
Product 3d
Product 3e

[Chemical structure image]

[Graphical representation of chemical analysis]

[Additional chemical structure image]
Product 3g
Product 3h
Product 3i
Product 3j
Product 3k
Product 3l
Product 3m
Product 3n
Product 3o
Product 3p
Product 3q
Product 3r
Product 3t
Product 3u

[Chemical structure diagram]

[Chemical spectrum diagram]
Product 3v
Product 3x
Product 3y

[Chemical structure and spectroscopic data]

S37
Product 5a
Product 5c
Product 5d
Product 5e
Product 5f
Product 5h
Product 5i
Product 7a
Product 7b
Product 7c