Supporting Information

Organic Ligand Free Carbonylation Reactions with Unsupported Bulk Pd as Catalyst

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Experimental Procedures

1. General information and experimental section

1.1 General Information:

XRD measurements were conducted by using a STADIP automated transmission diffractometer (STOE) equipped with an incident beam curved germanium monochromator with CuKα1 radiation and current of 40 kV and 150 mA, respectively. The XRD patterns were scanned in the 2 Theta range of 10-90°. XPS were obtained using a VG ES-CALAB 210 instrument equipped with a dual Mg/Al anode X-ray source, a hemispherical capacitor analyzer, and a 5 keV Ar+ iron gun. The electron binding energy was referenced to the C1s peak at 284.8 eV. The background pressure in the chamber was less than 10-7 Pa. The peaks were fitted by Gaussian–Lorentzian curves after a Shirley background subtraction. For quantitative analysis, the peak area was divided by the element-specific Scofield factor and the transmission function of the analyzer. TEM was carried out by using a Tecnai G2 F30 S-Twin transmission electron microscope operating at 300 kV. Single-particle EDX analysis was performed by using a Tecnai G2 F30 S-Twin Field Emission TEM in STEM mode. For TEM investigations, the catalysts were dispersed in ethanol by ultrasonication and deposited on carbon-coated copper grids. The Pd dispersion determined with H₂ titration technique using a Quantachrome autosorb IQ₂ at different temperature (25, 40, 80 °C). Hydrogen titration program: 1. Change Gas to Helium; 2. Ramp Temp by Rate to 140 °C at 20 deg/min; 3. Action: Flow Until Setpoint; 4. Action: Flow for 30 minutes; 5. Change Gas to port: 3, Hydrogen; 6. Ramp Temp by Rate to 400 °C at 20 deg/min; 7. Action: Flow Until Setpoint; 8. Action: Flow for 60 min; 9. Change Gas to Helium; 10. Action: Flow for 45 minutes; 11. Force Cool Furnace to 25/40/80 °C, stabilize; 12. Change Gas to #1, nitrogen; 13. TCD Device: On (Ib current:100, zeroing time out:5); 14. Action: Flow for 15 min; 15. Perform Titration Acquisition (Manual Analysis: No); 16. TCD Device: Off; 17. end of macro; NMR spectra were measured by using a Bruker ARX 400 or ARX 100 spectrometer at 400 MHz (¹H) and 100 MHz (¹³C). Chemical shifts were reported in parts per million (ppm) down field from TMS with the solvent resonance as the internal standard. Coupling constants (J) were reported in Hz.
and referred to apparent peak multiplications. Mass spectra were in general recorded on an Agilent 5977A MSD GC-MS.

1.2 Experimental Procedures

All solvents and chemicals were obtained commercially and used as received.

Typical procedure for carbonylation of olefins with amines and CO.

A mixture of olefins (2.0 mmol), amines (1 mmol), Pd-610 (1 mg), KI (5.0 mol%), H$_3$PO$_4$ (5.0 mol%) and dioxane (2 mL) were added a glass tube which was placed in an 100 mL autoclave. Then the autoclave was purged and charged with CO (40 bar). The reaction mixture was stirred at 130 °C for 24 h. After the reaction finished, the autoclave was cooled to room temperature and the pressure was carefully released. Subsequently, the reaction mixture was diluted with 5 mL of ethyl acetate for analysis by GC-MS. The regioselectivity were measured by $^1$H NMR. The crude reaction mixture was concentrated by rot-vap and purified by column chromatography on a silica gel column to give the desired products 3a and 4a.

Typical procedure for carbonylation of aryl iodides with amines and CO.

A mixture of aryl iodides (1.0 mmol), amines (1.5 mmol), Pd-610 (1 mg ), KI (5.0 mol%), K$_2$HPO$_4$ (2.0 mmol) and dioxane (2 mL) were added a glass tube which was placed in an 80 mL autoclave. Then the autoclave was purged and charged with CO (5 bar). The reaction mixture was stirred at 130 °C for 12 h. After the reaction finished, the autoclave was cooled to room temperature and the pressure was carefully released. Subsequently, the reaction mixture was diluted with 5 mL of methanol for analysis by GC-MS. The crude reaction mixture was concentrated by rot-vap and purified by column chromatography on a silica gel column to give the desired products.

Typical procedure for oxidative carbonylation of amines.

A mixture of amines (2 mmol), Pd-610 (1 mg ), KI (5.0 mol%) and dioxane (2 mL) were added a glass tube which was placed in an 80 mL autoclave. Then the autoclave was purged and charged with CO and O$_2$ (CO : O$_2$ = 35 : 5 bar). The reaction mixture was stirred at 130 °C for 24 h. After the reaction finished, the autoclave was cooled to room temperature and the pressure was carefully released. Subsequently, the reaction mixture was diluted with 5 mL of
methanol. The reaction mixture was concentrated by rot-vap and then heated for 0.5 h to remove aniline to give the desired products.

2. Optimization of the reaction conditions

Table S1. Screening of the catalyst for the carbonylation of styrene with aniline [a].

<table>
<thead>
<tr>
<th>Entry</th>
<th>Cat.</th>
<th>Con. (%)</th>
<th>Sel. (%)</th>
<th>b/l</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Pd-148</td>
<td>91</td>
<td>98</td>
<td>46:54</td>
</tr>
<tr>
<td>2</td>
<td>Pd-610</td>
<td>95</td>
<td>98</td>
<td>35:65</td>
</tr>
<tr>
<td>3</td>
<td>Pd-1540</td>
<td>16</td>
<td>&gt;99</td>
<td>60:40</td>
</tr>
<tr>
<td>4</td>
<td>PdCl₂</td>
<td>60</td>
<td>&gt;99</td>
<td>44:56</td>
</tr>
<tr>
<td>5</td>
<td>PdCl₂/(2-OMePh)₃P</td>
<td>39</td>
<td>81</td>
<td>36:64</td>
</tr>
<tr>
<td>6[cc]</td>
<td>PdI₂</td>
<td>58</td>
<td>94</td>
<td>65:35</td>
</tr>
<tr>
<td>7[dd]</td>
<td>PdI₂</td>
<td>40</td>
<td>&gt;99</td>
<td>59:61</td>
</tr>
</tbody>
</table>

[a] Reaction conditions: 1a (4.0 mmol), 2a (1.0 mmol), [Pd-10, Pd-148, Pd-610, Pd-1540] (1.0 mg), [PdCl₂] (1.77 mg), H₃PO₄ (5.0 mol%), KI (5.0 mol%), CO (40 bar), dioxane (2.0 mL), 130 °C, 12 h; [b] Determined by GC-MS; [c] 1a (2.0 mmol); [d] KI free.

Table S2. Screening of the [I] for the carbonylation of styrene with aniline [a].

<table>
<thead>
<tr>
<th>Entry</th>
<th>[I]</th>
<th>Con. (%)</th>
<th>Sel. (%)</th>
<th>b/l</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>KI</td>
<td>95</td>
<td>98</td>
<td>35:65</td>
</tr>
<tr>
<td>2[cc]</td>
<td>KI</td>
<td>93</td>
<td>99</td>
<td>35:65</td>
</tr>
<tr>
<td>3[cc]</td>
<td>KI</td>
<td>22</td>
<td>&gt;99</td>
<td>41:59</td>
</tr>
<tr>
<td>4[cc]</td>
<td>NaI</td>
<td>69</td>
<td>91</td>
<td>45:55</td>
</tr>
<tr>
<td>5[cc]</td>
<td>I₂</td>
<td>99</td>
<td>92</td>
<td>40:60</td>
</tr>
<tr>
<td>6</td>
<td>--</td>
<td>trace</td>
<td>--</td>
<td>--</td>
</tr>
</tbody>
</table>

[a] Reaction conditions: 1a (4.0 mmol), 2a (1.0 mmol), Pd-610 (1.0 mg), H₃PO₄ (5.0 mol%), [I] (5.0 mol%), CO (40 bar), dioxane (2.0 mL), 130 °C, 12 h; [b] Determined by GC-MS; [c] 1a (2.0 mmol); [d] 1a (1.2 mmol).
Table S3. Screening of the temperature for the carbonylation of styrene with aniline [a].

\[
\begin{align*}
\text{Ph} & \underset{\text{Pd-610, } \text{H}_2\text{PO}_4}{{+} \text{ Ph-NH}_2} \quad \text{Kl, T, CO} \quad \text{Ph} & \overset{\text{H}_2\text{N-P}}{\underset{\text{O-Ph}}{+}} \quad \text{Ph} & \overset{\text{H}}{\underset{\text{Ph}}{\text{O}}} \quad \text{Ph} \\
1a & \quad 2a & \quad 3a & \quad 4a
\end{align*}
\]

<table>
<thead>
<tr>
<th>Entry</th>
<th>(T (\degree \text{C}))</th>
<th>Con.(^a) (%)</th>
<th>Sel.(^b) (%)</th>
<th>b/l</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>130</td>
<td>95</td>
<td>98</td>
<td>35:65</td>
</tr>
<tr>
<td>2</td>
<td>150</td>
<td>97</td>
<td>91</td>
<td>36:64</td>
</tr>
<tr>
<td>3</td>
<td>110</td>
<td>12</td>
<td>&gt;99</td>
<td>68:32</td>
</tr>
</tbody>
</table>

[a] Reaction conditions: 1a (4.0 mmol), 2a (1.0 mmol), Pd-610 (1.0 mg), \(\text{H}_3\text{PO}_4\) (5.0 mol%), Kl (5.0 mol%), CO (40 bar), dioxane (2.0 mL), 12 h; [b] Determined by GC-MS.

Table S4. Screening of the solvent for the carbonylation of styrene with aniline [a].

\[
\begin{align*}
\text{Ph} & \underset{\text{Pd-610, } \text{H}_2\text{PO}_4}{{+} \text{ Ph-NH}_2} \quad \text{Kl, 130\degree \text{C, CO, Solvent}} \quad \text{Ph} & \overset{\text{H}_2\text{N-P}}{\underset{\text{O-Ph}}{+}} \quad \text{Ph} & \overset{\text{H}}{\underset{\text{Ph}}{\text{O}}} \quad \text{Ph} \\
1a & \quad 2a & \quad 3a & \quad 4a
\end{align*}
\]

<table>
<thead>
<tr>
<th>Entry</th>
<th>Solvent</th>
<th>Con.(^a) (%)</th>
<th>Sel.(^b) (%)</th>
<th>b/l</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>dioxane</td>
<td>95</td>
<td>98</td>
<td>35:65</td>
</tr>
<tr>
<td>2</td>
<td>NMP</td>
<td>14</td>
<td>72</td>
<td>46:54</td>
</tr>
<tr>
<td>3</td>
<td>DMF</td>
<td>n.r.</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>4</td>
<td>THF</td>
<td>85</td>
<td>97</td>
<td>49:51</td>
</tr>
</tbody>
</table>

[a] Reaction conditions: 1a (4.0 mmol), 2a (1.0 mmol), [Pd-610] (1.0 mg), \(\text{H}_3\text{PO}_4\) (5.0 mol%), Kl (5.0 mol%), CO (40 bar), solvent (2.0 mL), 130\degree \text{C}, 12 h; [b] Determined by GC-MS.

Table S5. Screening of the pressure CO for the carbonylation of styrene with aniline [a].

\[
\begin{align*}
\text{Ph} & \underset{\text{Pd-610, } \text{H}_2\text{PO}_4}{{+} \text{ Ph-NH}_2} \quad \text{Kl, 130\degree \text{C, CO, dioxane}} \quad \text{Ph} & \overset{\text{H}_2\text{N-P}}{\underset{\text{O-Ph}}{+}} \quad \text{Ph} & \overset{\text{H}}{\underset{\text{Ph}}{\text{O}}} \quad \text{Ph} \\
1a & \quad 2a & \quad 3a & \quad 4a
\end{align*}
\]

<table>
<thead>
<tr>
<th>Entry</th>
<th>[CO] (bar)</th>
<th>Con.(^a) (%)</th>
<th>Sel.(^b) (%)</th>
<th>b/l</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>40</td>
<td>95</td>
<td>98</td>
<td>35:65</td>
</tr>
<tr>
<td>2</td>
<td>30</td>
<td>62</td>
<td>93</td>
<td>47:53</td>
</tr>
<tr>
<td>3</td>
<td>20</td>
<td>60</td>
<td>60</td>
<td>32:68</td>
</tr>
</tbody>
</table>

[a] Reaction conditions: 1a (4.0 mmol), 2a (1.0 mmol), [Pd-610] (1.0 mg), \(\text{H}_3\text{PO}_4\) (5.0 mol%), Kl (5.0 mol%), dioxane (2.0 mL), 130\degree \text{C}, 12 h; [b] Determined by GC-MS.
Table S6. Screening of the amount of acids for the carbonylation of styrene with aniline \[a\].

\[
\begin{array}{cccc}
& \text{Pd-610, } [\text{H}_3\text{PO}_4] & \text{Kl, 130°C, CO, dioxane} & \text{Ph-NH}_2 \rightarrow \text{Ph-} \\
1a & 2a & 3a \text{ branched} & 4a \text{ linear} \\
\end{array}
\]

<table>
<thead>
<tr>
<th>Entry</th>
<th>$[\text{H}_3\text{PO}_4]$</th>
<th>Con.$^b$ (%)</th>
<th>Sel.$^b$ (%)</th>
<th>b/l</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0%</td>
<td>trace</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>2</td>
<td>1%</td>
<td>trace</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>3</td>
<td>2%</td>
<td>trace</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>4</td>
<td>5%</td>
<td>95</td>
<td>98</td>
<td>35:65</td>
</tr>
<tr>
<td>5</td>
<td>10%</td>
<td>88</td>
<td>96</td>
<td>51:49</td>
</tr>
</tbody>
</table>

[a] Reaction conditions: 1a (4.0 mmol), 2a (1.0 mmol), [Pd-610] (1.0 mg), [KI] (5.0 mol%), CO (40 bar), dioxane (2.0 mL), 130 °C, 12 h;

[b] Determined by GC-MS.

Table S7. Screening of the amount of acids for the carbonylation of styrene with aniline \[a\].

\[
\begin{array}{cccc}
& \text{Pd-610, } [\text{H}_3\text{PO}_4] & \text{Kl, 130°C, CO, dioxane} & \text{Ph-NH}_2 \rightarrow \text{Ph-} \\
1a & 2a & 3a \text{ branched} & 4a \text{ linear} \\
\end{array}
\]

<table>
<thead>
<tr>
<th>Entry</th>
<th>$[\text{KI}]$</th>
<th>Con.$^b$ (%)</th>
<th>Sel.$^b$ (%)</th>
<th>b/l</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1%</td>
<td>48</td>
<td>99</td>
<td>62:38</td>
</tr>
<tr>
<td>2</td>
<td>2%</td>
<td>82</td>
<td>97</td>
<td>55:45</td>
</tr>
<tr>
<td>3</td>
<td>5%</td>
<td>95</td>
<td>98</td>
<td>35:65</td>
</tr>
<tr>
<td>4</td>
<td>10%</td>
<td>trace</td>
<td>--</td>
<td>--</td>
</tr>
</tbody>
</table>

[a] Reaction conditions: 1a (4.0 mmol), 2a (1.0 mmol), [Pd-610] (1.0 mg), [H$_3$PO$_4$] (5.0 mol%), CO (40 bar), dioxane (2.0 mL), 130 °C, 12 h;

[b] Determined by GC-MS.
Table S8. Screening of the base for the carbonylation of iodobenzene with morpholine \[a\].

![Chemical structure](image)

<table>
<thead>
<tr>
<th>Entry</th>
<th>base</th>
<th>Con.(^b) (%)</th>
<th>Sel.(^b) (%)</th>
<th>7a</th>
<th>8a</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>KOH</td>
<td>trace</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>2</td>
<td>K(_3)PO(_4)</td>
<td>99</td>
<td>57</td>
<td>43</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>KH(_2)PO(_4)</td>
<td>69</td>
<td>99</td>
<td>--</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>K(_2)HPO(_4)</td>
<td>99</td>
<td>99</td>
<td>--</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>K(_2)CO(_3)</td>
<td>99</td>
<td>42</td>
<td>58</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Na(_3)PO(_4)</td>
<td>55</td>
<td>66</td>
<td>34</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>NaH(_2)PO(_4)</td>
<td>28</td>
<td>99</td>
<td>--</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>Na(_2)HPO(_4)</td>
<td>77</td>
<td>99</td>
<td>--</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>NaHCO(_3)</td>
<td>trace</td>
<td>--</td>
<td>--</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>CH(_3)COOK</td>
<td>56</td>
<td>70</td>
<td>--</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>KOtBu</td>
<td>48</td>
<td>6</td>
<td>--</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>Et(_3)N</td>
<td>trace</td>
<td>--</td>
<td>--</td>
<td></td>
</tr>
</tbody>
</table>

\[a\] Reaction conditions: 5a (1.5 mmol), 6a (1.0 mmol), [Pd-610] (1.0 mg), CO (5 bar), dioxane (2.0 mL), 130 °C, 6 h; \[b\] Determined by GC-MS.

Table S9. Screening of the amount of K\(_2\)HPO\(_4\) for the carbonylation of iodobenzene with morpholine \[a\].

![Chemical structure](image)

<table>
<thead>
<tr>
<th>Entry</th>
<th>K(_2)HPO(_4) (equiv.)</th>
<th>Con.(^b) (%)</th>
<th>Sel.(^b) (%)</th>
<th>7a</th>
<th>8a</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2</td>
<td>&gt;99</td>
<td>&gt;99</td>
<td>--</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>23</td>
<td>&gt;99</td>
<td>--</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>0.5</td>
<td>21</td>
<td>&gt;99</td>
<td>--</td>
<td></td>
</tr>
</tbody>
</table>

\[a\] Reaction conditions: 5a (1.5 mmol), 6a (1.0 mmol), [Pd-610] (1.0 mg), CO (5 bar), dioxane (2.0 mL), 130 °C, 6 h; \[b\] Determined by GC-MS.
Table S10. Screening of the pressure CO for the carbonylation of iodobenzene with morpholine [a].

![Chemical structure of iodobenzene and morpholine reaction](image)

<table>
<thead>
<tr>
<th>Entry</th>
<th><a href="bar">CO</a></th>
<th>Con.(^{b}) (%)</th>
<th>Sel.(^{b}) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5</td>
<td>&gt;99</td>
<td>&gt;99</td>
</tr>
<tr>
<td>2</td>
<td>3</td>
<td>&gt;99</td>
<td>&gt;99</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>80</td>
<td>&gt;99</td>
</tr>
</tbody>
</table>

[a] Reaction conditions: 5a (1.5 mmol), 6a (1.0 mmol), [Pd-610] (1.0 mg), CO (5 bar), dioxane (2.0 mL), 130 °C, 6 h; [b] Determined by GC-MS.

Table S11. The characterization results of the catalysts.

<table>
<thead>
<tr>
<th>Catalyst</th>
<th>Size (^a)</th>
<th>Pd 3d (eV) (^b)</th>
<th>XRD diffraction peaks (°) (^c)</th>
<th>Dispersion(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pd-148</td>
<td>148 nm</td>
<td>--</td>
<td>--</td>
<td>6.628</td>
</tr>
<tr>
<td>Pd-610</td>
<td>610 nm</td>
<td>335.4, 340.7</td>
<td>40.1, 46.7, 68.1, 82.1, 86.7</td>
<td>3.897</td>
</tr>
<tr>
<td>Pd-1580</td>
<td>1580 nm</td>
<td>--</td>
<td>18.1, 40.1, 46.7, 68.1, 82.1, 86.7</td>
<td>4.372</td>
</tr>
<tr>
<td>Used Pd-610</td>
<td>610 nm</td>
<td>n. d. (^d)</td>
<td>18.1, 40.1, 46.7, 68.1, 82.1, 86.7</td>
<td>--</td>
</tr>
</tbody>
</table>

[a] Determined by SEM; [b] Determined by XPS; [c] Determined by XRD; [d] Not detectable.

3. Characterization results of the catalysts

Fig. S1 TEM of catalyst. Pd-148 (a), Pd-610 (b), Pd-1540 (c), Pd-610 used three times (d).

Fig. S2 XPS spectra of catalysts. (a) fresh Pd-610 and (b) Pd-610 used three times.
Fig. S3 XRD patterns of catalysts. (a) fresh Pd-610 and (b) Pd-610 used three times.
4. Characterization data for products

3a N-Phenyl-2-phenylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1) to give the desired product which was a light grey solid; mp: 127-129°C (lit. 134-135°C); 1H NMR (400 MHz, CDCl₃) δ 7.35-7.43 (m, 6H), 7.25-7.33 (m, 3H), 7.04-7.08 (m, 2H), 3.72 (q, 7.2 MHz, 1H), 1.60 (d, 7.2 MHz, 3H); 13C NMR (100 MHz, CDCl₃) δ 172.2, 18.5, 140.9, 138.0, 129.1, 128.9, 127.7, 127.6, 124.2, 119.6, 48.1.

4a N,3-diphenylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1) to give the desired product which was a white solid; mp: 92-93°C (lit. 116-117°C); 1H NMR (400 MHz, CDCl₃) δ 7.43 (d, 7.60 MHz, 2H), 7.21-7.31 (m, 8H), 7.08 (t, 7.4 MHz, 1H), 3.04 (t, 7.6 MHz, 2H), 2.64 (t, 7.6 MHz, 2H); 13C NMR (100 MHz, CDCl₃) δ 170.5, 140.6, 137.7, 128.9, 128.6, 126.3, 124.3, 120.0, 39.3, 31.5.

3b 2-phenyl-N-(p-tolyl)propanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1) to give the desired product which was a light yellow solid; mp: 115-117°C; 1H NMR (400 MHz, CDCl₃) δ 7.35-7.38 (m, 4H), 7.29-7.31 (m, 3H), 7.05-7.08 (m, 3H), 3.70 (q, 7.2 MHz, 1H), 2.27 (s, 3H), 1.58 (d, 6.8 MHz, 3H); 13C NMR (100 MHz, CDCl₃) δ 172.2, 141.1, 135.3, 133.8, 129.3, 129.1, 127.7, 127.5, 119.7, 48.0, 20.8, 18.6.

4b 3-phenyl-N-p-tolylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1) to give the desired product which was a white solid; mp: 127-130°C; 1H NMR (400 MHz, CDCl₃) δ 7.26-7.31 (m, 5H), 7.19-7.21 (m, 3H), 7.06-7.08 (m, 2H), 3.02 (t, 7.6 MHz, 2H), 2.61
(t, 7.6 MHz, 2H), 2.29(s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 170.4, 140.7, 135.1, 133.9, 129.4, 128.6, 128.3, 126.3, 120.1, 39.3, 31.6, 20.8.

3c 2-phenyl-N-o-tolylpropanamide$: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1 to 10:1) to give the desired product which was a white solid; mp: 79-80°C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.88(d, 8.0 MHz, 1H), 7.31-7.43 (m, 5H), 6.98-7.19 (m, 3H), 6.86(brs, 1H), 3.79 (q, 7.2 MHz, 1H), 1.87(s, 3H), 1.65 (d, 7.2 MHz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 172.3, 140.9, 135.7, 130.3, 129.2, 128.0, 127.8, 127.7, 126.7, 124.7, 122.0, 48.1, 18.0, 17.1.

4c 3-phenyl-N-o-tolylpropanamide$^{[6]}$: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1 to 6:1) to give the desired product which was a white solid; mp: 115-116°C; $^1$H NMR (400 MHz, CDCl$_3$) δ 2.05(s, 3H), 2.69 (t, 7.4 MHz, 2H), 3.06 (t, 7.4 MHz, 2H), 6.90(brs, 1H), 7.03-7.06 (m, 1H), 7.12-7.32 (m, 8H), 7.70(d, 8.0MHz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 170.4, 140.5, 135.4, 130.4, 128.6, 128.4, 126.6, 126.4, 125.2, 123.3, 39.2, 31.7, 17.5.

3d N-(4-butylphenyl)-2-phenylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (50:1 to 30:1) to give the desired product which was a brown solid; mp: 74-75°C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.35-7.39 (m, 4H), 7.07 (d, 8.4MHz, 3H), 3.70 (q, 7.1 MHz, 1H), 2.53(t, 7.6MHz, 2H), 1.59 (d, 6.8 MHz, 3H), 1.49-1.57(m, 2H), 1.25-1.35(m, 2H), 0.89(t, 7.4 MHz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 172.2, 141.0, 138.9, 135.4, 129.1, 128.7, 127.7, 127.5, 119.7, 48.0, 35.0, 33.6, 22.2, 18.5, 13.9; HRMS(m/z): [M+H] calcd. for C$_{19}$H$_{24}$NO, 282.1852; found, 282.1862.

4d N-(4-butylphenyl)-3-phenylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography,
using petroleum ether/diethyl ether (30 : 1 to 10 : 1) to give the desired product which was a yellow solid; mp: 69-70°C; \(^1\text{H NMR}\) (400 MHz, CDCl\(_3\)) \(\delta\) 7.27-7.33 (m, 4H), 7.19-7.23 (m, 3H), 7.13 (brs, 1H), 7.09 (d, 8.4MHz, 2H), 3.04 (t, 7.6 MHz, 2H), 2.63 (t, 7.6 MHz, 2H), 2.55 (t, 7.8MHz, 2H), 1.52-1.59 (m, 2H), 1.25-1.37 (m, 2H), 0.91 (t, 7.4MHz, 3H); \(^{13}\text{C NMR}\) (100 MHz, CDCl\(_3\)) \(\delta\) 170.3, 140.7, 139.0, 135.3, 128.8, 128.6, 128.4, 126.3, 120.0, 39.4, 35.0, 33.6, 31.6, 22.2, 13.9; HRMS(m/z): [M+H] calcd. for C\(_{19}\)H\(_{24}\)NO, 282.1852; found, 282.1858.

\[\text{3e N-(4-tert-butylphenyl)-2-phenylpropanamide:}\] The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (15:1) to give the desired product which was a white solid; mp: 97-98°C; \(^1\text{H NMR}\) (400 MHz, CDCl\(_3\)) \(\delta\) 7.31-7.39 (m, 6H), 7.25-7.29 (m, 3H), 7.09 (brs, 1H), 3.71 (q, 7.2 MHz, 1H), 1.59 (d, 7.2MHz, 3H), 1.27 (s, 9H); \(^{13}\text{C NMR}\) (100 MHz, CDCl\(_3\)) \(\delta\) 172.1, 147.2, 141.0, 135.2, 129.1, 127.7, 127.5, 125.6, 119.4, 48.0, 34.3, 31.3, 18.5; HRMS(m/z): [M+H] calcd. for C\(_{19}\)H\(_{24}\)NO, 282.1852; found, 282.1849.

\[\text{4e N-(4-tert-butylphenyl)-3-phenylpropanamide:}\] The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (15:1) to give the desired product which was a white solid; mp: 99-101°C; \(^1\text{H NMR}\) (400 MHz, CDCl\(_3\)) \(\delta\) 7.34-7.36 (m, 2H), 7.25-7.30 (m, 5H), 7.19-7.22 (m, 3H), 3.03 (t, 7.6 MHz, 2H), 2.63 (t, 7.6 MHz, 2H), 1.28 (s, 9H); \(^{13}\text{C NMR}\) (100 MHz, CDCl\(_3\)) \(\delta\) 170.3, 147.2, 140.6, 135.1, 128.6, 128.4, 126.3, 125.7, 119.8, 39.3, 34.3, 31.6, 31.3; HRMS(m/z): [M+H] calcd. for C\(_{19}\)H\(_{24}\)NO, 282.1852; found, 282.1855.

\[\text{3f N-(3,5-dimethylphenyl)-2-phenylpropanamide:}\] The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (50 : 1 to 20 : 1) to give the desired product which was a white solid; mp: 12/89
86-87°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.48 (brs, 1H), 7.21-7.33 (m, 5H), 7.08 (s, 2H), 6.68 (s, 1H), 3.69 (q, 7.1 MHz, 1H), 2.20 (s, 6H), 1.55 (d, 7.2 MHz, 3H); \(^1\)^3C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 172.5, 141.0, 138.3, 137.7, 128.8, 127.5, 127.2, 117.6, 29.6, 21.1, 18.5; HRMS (m/z): [M+H] calcd. for C\(_{17}\)H\(_{26}\)NO, 254.1539; found, 254.1544.

4f N-(3,5-dimethylphenyl)-3-phenylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20 : 1 to 10 : 1) to give the desired product which was a white solid; mp: 82-84°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.28 (s, 1H), 7.08-7.20 (m, 7H), 6.66 (s, 1H), 2.94 (t, 7.6 MHz, 2H), 2.57 (t, 7.8 MHz, 2H), 2.15 (s, 6H); \(^1\)^3C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 171.1, 140.5, 138.1, 137.7, 128.2, 128.0, 125.9, 125.7, 118.0, 38.6, 31.4, 21.0; HRMS (m/z): [M+H] calcd. for C\(_{17}\)H\(_{26}\)NO, 254.1539; found, 254.1530.

3g Benzeneacetamide\(^{[5]}\): The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1) to give the desired product which was a brown solid; mp: 114-115°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.36-7.40 (m, 4H), 7.26-7.32 (m, 3H), 7.06-7.08 (m, 2H), 6.98 (brs, 1H), 3.70 (q, 7.07 MHz, 1H), 1.54 (d, 7.2 MHz, 3H); \(^1\)^3C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 172.4, 140.7, 136.4, 129.2, 128.8, 127.6, 120.9, 48.0, 18.5.

4g N-(4-chlorophenyl)-3-phenylpropanamide\(^{[7]}\): The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1) to give the desired product which was a white solid. Mp=136-137°C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.36 (d, 8.4 MHz, 2H), 7.20-7.31 (m, 7H), 7.17 (brs, 1H), 3.03 (t, 7.6 MHz, 2H), 2.64 (t, 7.6 MHz, 2H); \(^1\)^3C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 170.4, 140.4, 136.2, 129.2, 128.9, 128.7, 128.3, 126.5, 121.1, 39.4, 31.5.
3h  N-(4-methoxyphenyl)-2-phenylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (6:1) to give the desired product which was a light brown solid; mp: 139-140°C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.35-7.40 (m, 4H), 7.28-7.33 (m, 3H), 7.04 (brs, 1H), 6.78-6.82 (m, 2H), 3.75 (s, 3H), 3.70 (q, 7.1 MHz, 1H), 1.59(d, 6.8MHz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 172.1, 156.3, 141.0, 130.9, 129.1, 127.7, 127.5, 121.6, 114.0, 55.4, 47.9, 18.6.

4h  N-(4-methoxyphenyl)-3-phenylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (6:1) to give the desired product which was a light yellow solid; mp: 127-129°C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 2.62 (t, 7.6 MHz, 2H), 3.03 (t, 7.6 MHz, 2H), 3.76(s, 3H), 6.79-6.82 (m, 2H), 7.19-7.22 (m, 4H), 7.27-7.35 (m, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.3, 156.4, 140.7, 130.8, 128.6, 128.4, 126.3, 121.9, 114.0, 55.4, 39.2, 31.6.

3i  N-(2-fluorophenyl)-2-phenylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (30:1 to 10:1) to give the desired product which was a white solid; mp: 85-86°C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.29-8.33 (m, 1H), 7.36-7.41 (m, 4H), 7.29-7.34 (m, 2H), 7.05-7.11 (m, 1H), 6.98-7.02 (m, 2H), 3.76 (q, 7.2 MHz, 1H), 1.62 (d, 7.2 MHz, 3H); $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -131.67; $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 172.3, 152.3 ($J = 241.6$ Hz), 140.5, 129.1, 127.6, 126.3 ($J = 3.7$ Hz), 124.4 ($J = 7.6$ Hz), 121.5, 114.6 ($J = 19.0$ Hz), 48.2, 18.4; HRMS(m/z): [M+H] calcd. for C$_{15}$H$_{15}$FNO, 244.1132; found, 244.1129.

4i  N-(2-fluorophenyl)-3-phenylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1) to give the desired product which was a light yellow solid; mp: 91-94°C; $^1$H
NMR (400 MHz, CDCl$_3$) δ 8.29-8.33 (m, 1H), 7.20-7.34 (m, 6H), 7.00-7.14 (m, 3H), 3.07 (t, 7.8 MHz, 2H),
2.72 (t, 7.6 MHz, 2H); $^{19}$F NMR (376 MHz, CDCl$_3$) δ -116.79; $^{13}$C NMR (100 MHz, CDCl$_3$) δ 170.3, 152.2
($J = 241.3$ Hz), 140.4, 128.6, 128.3, 126.4, 126.2 ($J = 9.9$ Hz), 124.5 ($J = 3.6$ Hz), 124.2 ($J = 7.6$ Hz),
121.8, 114.7 ($J = 19.1$ Hz), 39.4, 31.3. HRMS (m/z): [M+H] calcd. for C$_{15}$H$_{15}$FNO, 244.1132; found,
244.1122.

3j ethyl 4-(2-phenylpropanamido)benzoate$^{[5]}$:

4j ethyl 4-(3-phenylpropanamido)benzoate$^{[4]}$:

The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (30 : 1 to 5 : 1) to give the desired product which were white solid; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.93-7.99 (m, 4H), 7.49-7.54 (m, 4H), 7.20-7.41 (m, 12H),
4.28-4.37 (m, 4H), 3.74 (q, 7.1 MHz, 1H), 3.05 (t, 7.6 MHz, 2H), 2.69 (t, 7.6 MHz, 2H), 1.60 (d, 7.2 MHz,
3H), 1.35-1.40 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 172.6, 170.7, 166.2, 166.1, 141.9, 140.5, 140.4,
130.7, 130.6, 129.2, 128.6, 128.3, 127.7, 127.6, 126.4, 125.8, 118.8, 118.7, 60.8, 48.2, 39.4, 31.3, 18.5,
14.3.

3k 2-phenyl-N-(quinolin-8-yl)propanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (30 : 1 to 20 : 1) to give the desired product which was a white solid; mp: 209-210°C;
$^1$H NMR (400 MHz, CDCl$_3$) δ 9.91 (brs, 1H), 8.77 (dd, 1.6 MHz, 7.6 MHz, 1H), 8.69 (dd, 1.6 MHz, 4.0
MHz, 1H), 8.10 (dd, 1.6 MHz, 8.0 MHz, 1H), 7.44-7.52 (m, 4H), 7.36-7.40 (m, 3H), 7.27-7.31 (m, 1H),
3.80 (q, 7.1 MHz, 1H), 1.69 (d, 7.2 MHz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 172.7, 148.0, 141.1, 138.4,
136.2, 134.5, 128.9, 127.8, 127.7, 127.3, 127.3, 121.5, 121.4, 116.3, 48.6, 18.6; HRMS (m/z): [M+H]
 calcd. for C$_{18}$H$_{17}$N$_3$O, 277.1335; found, 277.1342.
4k 3-phenyl-N-(quinolin-8-yl)propanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (50:1 to 30:1) to give the desired product which was a grey solid; mp: 58-60°C; $^1$H NMR (400 MHz, CDCl$_3$) δ 9.81 (brs, 1H), 8.79 (dd, 1.2 MHz, 7.2 MHz, 1H), 8.77 (dd, 1.6 MHz, 4.0 MHz, 1H), 8.15 (dd, 1.6 MHz, 8.4 MHz, 1H), 7.48-7.56 (m, 2H), 7.43-7.46 (m, 1H), 7.18-7.31 (m, 5H), 3.15 (t, 8.0 MHz, 2H), 2.89 (t, 8.0 MHz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 170.7, 148.0, 140.7, 138.2, 136.4, 134.4, 128.5, 128.4, 127.9, 127.4, 126.2, 121.5, 121.4, 116.5, 39.7, 31.4.

3l N-phenyl-2-p-tolylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1) to give the desired product which was a light grey brown solid; mp: 119-120°C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.40-7.42 (m, 2H), 7.24-7.28 (m, 4H), 7.17-7.19 (m, 2H), 7.04-7.08 (m, 2H), 3.68 (q, 7.1 MHz, 1H), 2.35(s, 3H), 1.58 (d, 6.8 MHz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 172.5, 137.8, 137.3, 129.8, 128.9, 127.6, 124.1, 119.6, 47.7, 21.0, 18.5.

4l N-phenyl-3-(p-tolyl)propanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1) to give the desired product which was a light brown solid; mp: 114-115°C (lit. 9 120-121°C); $^1$H NMR (400 MHz, CDCl$_3$) δ 7.42-7.44 (m, 2H), 7.22-7.30 (m, 3H), 7.06-7.13 (m, 5H), 3.00 (t, 7.6 MHz, 2H), 2.63 (t, 7.6 MHz, 2H), 2.31(s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 170.5, 137.7, 137.5, 135.9, 129.3, 128.9, 128.2, 124.2, 119.9, 39.5, 31.1, 21.0.

3m 2-(4-tert-butylphenyl)-N-phenylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1) to give the desired product which was a white solid; mp: 120-
171°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.38-7.44 (m, 4H), 7.25-7.30 (m, 4H), 7.04-7.10 (m, 2H), 3.70 (q, 7.1 MHz, 1H), 1.59(d, 7.2 MHz, 3H), 1.32 (s, 9H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 172.5, 150.5, 137.9, 137.7, 128.9, 127.3, 126.0, 124.1, 119.7, 47.6, 34.5, 31.3, 18.5; HRMS(m/z): [M+H] calcd. for C\(_{19}\)H\(_{24}\)NO, 282.1852; found, 282.1855.

**4m 3-(4-tert-butylphenyl)-N-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10 : 1) to give the desired product which was a white solid; mp: 110-113°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.04-7.42 (m, 2H), 7.25-7.33 (m, 4H), 7.16-7.18 (m, 3H), 7.06-7.10 (m, 1H), 3.02 (t, 7.6 MHz, 2H), 2.65 (t, 7.6 MHz, 2H), 1.31(s, 9H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 170.5, 149.2, 137.7, 137.5, 129.0, 125.5, 124.2, 119.9, 39.4, 34.4, 31.3, 31.0; HRMS(m/z): [M+H] calcd. for C\(_{19}\)H\(_{24}\)NO, 282.1852; found, 282.1848.

**3n 2-(4-fluorophenyl)-N-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1) to give the desired product which was a light grey solid; mp: 112-113°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.30-7.36 (m, 2H), 7.17-7.27 (m, 4H), 7.12 (brs, 1H), 6.95-7.02 (m, 3H), 3.61 (q, 7.1 MHz, 1H), 1.49 (d, 7.2 MHz, 3H); \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) 114.82; \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 172.1, 162.1 (J = 244.7 Hz), 137.7, 136.6 (J = 3.2 Hz), 129.2 (J = 8.0 Hz), 128.9, 124.4, 119.7, 115.9 (J = 21.2 Hz), 47.3, 18.8; HRMS(m/z): [M+H] calcd. for C\(_{15}\)H\(_{15}\)FNO, 244.1132; found, 244.1143.

**4n 3-(4-fluorophenyl)-N-phenylpropanamide:** The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1) to give the desired product which was a light yellow solid; mp: 128-129°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.43-7.45 (m, 2H), 7.26-7.31 (m, 3H), 7.15-7.18 (m, 2H), 7.08-7.11 (m, 1H), 6.94-6.98 (m, 2H), 3.01 (t, 7.6 MHz, 2H), 2.62 (t, 7.4 MHz, 2H); \(^{19}\)F NMR (376 MHz, CDCl\(_3\)) \(\delta\) -116.79; \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 170.2, 161.5 (J = 242.6 Hz), 137.6, 136.3 (J = 5.4 Hz), 129.8 (J = 178/89
3o 2-(4-bromophenyl)-N-phenylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1) to give the desired product which was a white solid; mp: 162-165°C; ^1H NMR (400 MHz, CDCl₃) δ 7.42-7.50 (m, 4H), 7.23-7.30 (m, 4H), 7.06-7.13 (m, 2H), 3.66 (q, 6.9 MHz, 1H), 1.56 (d, 6.8 MHz, 3H); ^13C NMR (100 MHz, CDCl₃) δ 171.7, 139.9, 137.7, 132.2, 129.4, 129.0, 124.5, 121.5, 119.8, 47.6, 18.7.

4o 3-(4-bromophenyl)-N-phenylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1) to give the desired product which was a white solid; mp: 158-160°C; ^1H NMR (400 MHz, CDCl₃) δ 7.38-7.45 (m, 4H), 7.25-7.31 (m, 3H), 7.07-7.12 (m, 3H), 2.99 (t, 7.6 MHz, 2H), 2.61 (t, 7.6 MHz, 2H); ^13C NMR (100 MHz, CDCl₃) δ 170.0, 139.6, 137.6, 131.6, 130.1, 129.0, 124.4, 120.1, 119.9, 39.0, 30.8; HRMS(m/z): [M+H] calcd. for C₁₆H₁₅BrNO, 304.0332; found, 304.0332.

3p 3-cyano-2-methyl-N-phenylpropanamide: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1) to give the desired product which was a white solid; mp: 125-126°C; ^1H NMR (400 MHz, CDCl₃) δ 7.34-7.36 (m, 2H), 7.19-7.30 (m, 7H), 7.05-7.09 (m, 1H), 6.88 (brs, 1H), 3.01-3.06 (m, 1H), 2.74-2.80 (m, 1H), 2.54-2.63 (m, 1H), 1.28 (d, 6.8 MHz, 3H); ^13C NMR (100 MHz, CDCl₃) δ 173.9, 139.6, 137.6, 128.9, 128.8, 128.5, 126.4, 124.2, 120.0, 44.7, 40.5, 17.7.
**4p N,4-diphenylbutanamide**: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1) to give the desired product which was a white solid; mp: 91-92°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.48-7.50 (m, 2H), 7.25-7.32 (m, 5H), 7.17-7.21 (m, 3H), 7.07-7.11 (m, 1H), 2.69 (t, 7.4 MHz, 2H), 2.33 (t, 7.4 MHz, 2H), 2.02-2.09 (m, 2H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 171.0, 141.3, 137.8, 128.9, 128.5, 128.4, 126.0, 124.2, 119.8, 36.7, 35.0, 26.8.

**3q 2-methyl-N-phenyloctanamide**: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (50:1) to give the desired product which was a white solid; mp: 61-64°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.53-7.55 (m, 2H), 7.26-7.33 (m, 3H), 7.08-7.12 (m, 1H), 2.30-2.37 (m, 1H), 1.60-1.78 (m, 2H), 1.22-1.30 (m, 11H), 0.85-0.88 (m, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 175.0, 138.0, 128.9, 124.1, 119.8, 42.7, 34.5, 31.7, 29.3, 27.5, 22.6, 17.9, 14.0.

**4q N-phenylnonanamide**: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (50:1) to give the desired product which was a white solid; mp: 51-52°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.51-7.53 (m, 2H), 7.26-7.35 (m, 3H), 7.08-7.11 (m, 1H), 2.32-2.37 (m, 2H), 1.59-1.76 (m, 2H), 1.26-1.34 (m, 10H), 0.86-0.89 (m, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 171.5, 137.9, 128.9, 124.1, 119.8, 37.8, 31.8, 29.3, 29.3, 29.1, 25.6, 22.6, 14.1.

**3r 2-methyl-N-phenyldodecanamide**: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (60:1 to 30:1) to give the desired product which was a light yellow solid; mp: 47-48°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.53-7.55 (m, 2H), 7.29-7.33 (m, 3H), 7.24 (brs, 1H), 7.08-7.11 (m, 1H), 2.28-2.37 (m, 2H), 1.41-1.78 (m, 4H), 1.22-1.26 (m, 16H), 0.86-0.90 (m, 4H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 175.0, 138.0, 128.9, 124.1, 119.8, 34.5, 33.9, 31.9, 29.6, 29.5, 29.5, 29.3,
27.5, 24.7, 22.7, 17.9, 14.1; HRMS(m/z): [M+H] calcd. for C_{19}H_{32}NO, 290.2478; found, 290.2491.

The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (30:1) to give the desired product which was a light yellow solid; mp: 70-71°C; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) δ 7.51-7.53 (m, 2H), 7.26-7.33 (m, 3H), 7.07-7.11 (m, 1H), 2.32-2.37 (m, 2H), 1.59-1.75 (m, 2H), 1.25-1.31 (m, 18H), 0.86-0.90 (m, 3H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}) δ 171.5, 138.0, 128.9, 124.1, 119.8, 37.8, 31.9, 29.6, 29.6, 29.5, 29.4, 29.3, 29.3, 28.7, 25.6, 22.7, 14.1.

\[ \text{4r N-phenyltridecanamide} \]

The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (30:1) to give the desired product which was a light yellow solid; mp: 70-71°C; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) δ 7.51-7.53 (m, 2H), 7.26-7.33 (m, 3H), 7.07-7.11 (m, 1H), 2.32-2.37 (m, 2H), 1.59-1.75 (m, 2H), 1.25-1.31 (m, 18H), 0.86-0.90 (m, 3H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}) δ 171.5, 138.0, 128.9, 124.1, 119.8, 37.8, 31.9, 29.6, 29.6, 29.5, 29.4, 29.3, 29.3, 28.7, 25.6, 22.7, 14.1.

\[ \text{3s 2-methyl-N-phenylhexadecanamide} \]

The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (100:1) to give the desired product which was a white solid; mp: 84-85°C; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) δ 7.69 (s, 1H), 7.54-7.56 (m, 2H), 7.26-7.30 (m, 2H), 7.06-7.09 (m, 1H), 2.32-2.42 (m, 1H), 1.59-1.75 (m, 2H), 1.16-1.26 (m, 27H), 0.86-0.89 (m, 3H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}) δ 175.1, 138.0, 128.9, 124.1, 119.8, 42.6, 34.4, 31.9, 29.7, 29.6, 29.6, 29.6, 29.5, 29.3, 27.5, 22.7, 17.9, 14.1; HRMS(m/z): [M+H] calcd. for C_{23}H_{40}NO, 346.3104; found, 346.3105.

\[ \text{4s N-phenylheptadecanamide} \]

The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (100:1) to give the desired product which was a white solid; mp: 86-87°C; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) δ 7.51-7.53 (m, 2H), 7.26-7.33 (m, 3H), 7.08-7.11 (m, 1H), 2.33-2.37 (m, 2H), 1.59-1.76 (m, 2H), 1.25-1.32 (m, 26H), 0.86-0.90 (m, 3H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}) δ 171.5, 137.9, 128.9, 124.1, 119.8, 37.8, 33.8, 31.9, 29.7, 29.6, 29.6, 29.5, 29.4, 29.3, 29.3, 29.1, 25.6, 24.8, 22.7, 14.1; HRMS(m/z): [M+H] calcd. for C_{23}H_{40}NO, 346.3104; found, 346.3114.
7a **N-hexylbenzamide**[^1]: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1) to give the desired product which was a light yellow oil; **$^1$H NMR** (400 MHz, CDCl$_3$) $\delta$ 7.77-7.79 (m, 2H), 7.44-7.48 (m, 2H), 7.36-7.40 (m, 2H), 6.65 (brs, 1H), 3.38-3.43 (m, 2H), 1.55-1.62 (m, 2H), 1.26-1.36 (m, 6H), 0.86-0.90 (m, 3H); **$^{13}$C NMR** (100 MHz, CDCl$_3$) $\delta$ 167.5, 134.7, 131.0, 128.3, 126.8, 40.0, 31.4, 29.5, 26.6, 22.4, 13.9.

7b **N-Phenyl-benzamide**[^1]: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1) to give the desired product which was a light yellow solid; mp: 163°C (lit. 162-163°C); **$^1$H NMR** (400 MHz, CDCl$_3$) $\delta$ 7.86-7.88 (m, 3H), 7.64-7.66 (m, 2H), 7.53-7.58 (m, 1H), 7.46-7.50 (m, 2H), 7.14-7.18 (m, 1H); **$^{13}$C NMR** (100 MHz, CDCl$_3$) $\delta$ 165.8, 137.9, 135.0, 131.8, 129.1, 128.8, 127.0, 124.6, 120.2.

7c **Morpholino(phenyl)methanone**[^13]: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (20:1 to 2:1) to give the desired product which was a white solid; mp: 68-70°C; **$^1$H NMR** (400 MHz, CDCl$_3$) $\delta$ 7.40-7.44 (m, 5H), 3.38-3.80 (m, 8H); **$^{13}$C NMR** (100 MHz, CDCl$_3$) $\delta$ 170.3, 135.2, 129.8, 128.4, 127.0, 66.8, 48.1, 42.4.

7d **N,N-Dibutylbenzamide**[^14]: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (50:1 to 10:1) to give the desired product which was a light yellow oil; **$^1$H NMR** (400 MHz, CDCl$_3$) $\delta$ 7.31-7.36
(m, 5H), 3.18-3.49 (m, 4H), 1.65 (m, 2H), 1.39-1.47 (m, 4H), 1.11-1.13 (m, 2H), 0.92-0.97 (m, 3H), 0.77-
0.86 (m, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 171.4, 137.2, 128.8, 128.1, 126.2, 48.5, 44.2, 30.6, 29.4,

7e  **N-methyl-N-phenylbenzamide**$^{[13]}$: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (50 : 1 to 5 : 1) to give the desired product which was a light brown oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.27-7.30 (m, 2H), 7.19-7.23 (m, 3H), 7.10-7.16 (m, 3H), 7.02-7.04 (m, 2H), 3.49 (s, 3H);
$^{13}$C NMR (100 MHz, CDCl$_3$) δ 170.6, 144.7, 135.8, 129.5, 129.0, 128.6, 127.6, 126.8, 126.4, 38.3.

7f  **N-(4-toluoyl)morpholine**$^{[15]}$: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (5:1) to give the desired product which was a white oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.27-7.30 (m, 2H), 7.19-
7.21 (m, 2H), 3.50-3.69 (m, 8H), 2.36 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 170.5, 140.0, 132.2, 129.0,
127.1, 66.8, 48.2, 42.6, 21.3.

7g  **N-(4-methoxybenzoyl)morpholine**$^{[15]}$: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (5:1) to give the desired product which was a brown oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.38-7.40 (m, 2H), 6.90-6.93 (m, 2H), 3.82 (s, 3H), 3.64-3.69 (m, 8H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 170.2, 160.7, 129.0, 127.1, 113.6, 66.7, 55.1, 42.8.

7h  **4-(2-Fluorobenzoyl)-morpholine**$^{[16]}$: The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (5:1) to give the desired product which was a light yellow solid; mp: 73-74 °C (lit.$^{[17]}$ 75-77 °C).
77°C; \( ^1H \) NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.39-7.44 (m, 2H), 7.20-7.27 (m, 1H), 7.08-7.13 (m, 1H), 3.77-3.83 (m, 4H), 3.64-3.65 (m, 2H), 3.35 (s, 2H); \( ^{19}F \) NMR (376 MHz, CDCl\(_3\)) \( \delta \) -114.94; \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)) \( \delta \) 165.2, 158.0 (J = 246.8Hz), 131.5 (J = 8.0Hz), 129.2 (J = 3.6Hz), 124.8 (J = 3.4Hz), 123.6 (J = 17.5Hz), 115.8 (J = 21.4Hz), 66.8, 66.7, 47.4, 42.4.

7i \( N\)-(4-Chlorobenzoyl)piperidine\(^[14]\): The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1 to 2:1) to give the desired product which was a light brown solid; mp: 66-68°C; \( ^1H \) NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.35-7.42 (m, 4H), 3.38-3.80 (m, 8H); \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)) \( \delta \) 169.3, 136.0, 128.8, 128.6, 66.8, 48.1.

7j 4-(morpholine-4-carbonyl)benzonitrile\(^[15]\): The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (10:1 to 1:1) to give the desired product which was a light yellow solid; mp: 141-142°C; \( ^1H \) NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.73-7.75 (m, 2H), 7.51-7.54 (m, 2H), 3.39-3.80 (m, 8H); \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)) \( \delta \) 168.2, 139.6, 132.5, 127.8, 117.9, 113.7, 66.7, 48.0, 42.5.

7k morpholino(naphthalen-1-yl)methanone\(^[17]\): The title compound was prepared according to the general procedure and purified by column chromatography using petroleum ether/diethyl ether (5:1 to 2:1) to give the desired product which was a brown oil; \( ^1H \) NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.83-7.85 (m, 3H), 7.39-7.54 (m, 4H), 3.78-4.01 (m, 4H), 3.44-3.47 (m, 2H), 3.13-3.17 (m, 2H); \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)) \( \delta \) 169.4, 133.6, 133.4, 129.4, 129.3, 128.4, 127.0, 126.4, 125.1, 124.5, 123.8, 67.0, 66.9, 47.5, 42.1.
9a 1,3-dihexylurea\(^{[18]}\): The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a brown oil; \(^1\text{H NMR}\) (400 MHz, DMSO-d6) \(\delta\) 5.73 (brs, 1H), 3.50 (s, 1H), 2.92-3.11 (m, 3H), 1.24-1.44 (m, 16H), 0.84-0.86 (m, 6H); \(^{13}\text{C NMR}\) (100 MHz, DMSO-d6) \(\delta\) 158.1, 39.2, 31.1, 30.0, 26.1, 22.1, 13.9.

9b 1,3-diheptylurea\(^{[18]}\): The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a yellow solid; mp: 65°C; \(^1\text{H NMR}\) (400 MHz, DMSO-d6) \(\delta\) 2.95-3.11 (m, 4H), 1.24-2.02 (m, 20H), 0.86 (s, 6H); \(^{13}\text{C NMR}\) (100 MHz, CDCl\(_3\)) \(\delta\) 158.7, 40.5, 31.7, 30.3, 29.0, 26.9, 22.5, 14.0.

9c 1,3-Dibenzyurea\(^{[19]}\): The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was light brown solid; mp: 209-210°C; \(^1\text{H NMR}\) (400 MHz, DMSO-d6) \(\delta\) 7.24-7.31 (m, 10H), 6.44 (brs , 2H), 4.23 (s, 4H); \(^{13}\text{C NMR}\) (100 MHz, DMSO-d6) \(\delta\) 158.1, 140.9, 128.2, 127.0, 126.5, 40.0.

9d 1,3-dicyclohexylurea\(^{[20]}\): The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a light grey solid; mp: 209-210°C; \(^1\text{HNMR}\) (400 MHz, CDCl\(_3\)) \(\delta\) 3.46-3.41 (m, 2H) 1.10-1.95 (m, 20H); \(^{13}\text{C NMR}\) (100 MHz, CDCl\(_3\)) \(\delta\) 156.7, 49.1, 33.9, 25.6, 24.9.

9e 1,3-diphenylurea\(^{[3]}\): The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a white solid; mp: 227-232°C; \(^1\text{H NMR}\) (400 MHz, DMSO-d6) \(\delta\) 8.68 (brs, 2H), 7.45-7.47 (m, 4H), 7.26-7.30 (m, 4H), 6.95-6.99 (m, 2H); \(^{13}\text{C NMR}\) (100 MHz, DMSO-d6) \(\delta\) 152.5, 139.7, 128.8, 121.8, 118.1.
9f 1,3-Bis(4-methylphenyl)urea\textsuperscript{(19)}: The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was white solid; mp: 266-267°C (lit.\textsuperscript{20} 281-282°C); \textsuperscript{1}H NMR (400 MHz, DMSO-d\textsubscript{6}) δ 8.51 (brs, 2H), 7.33 (d, 7.6 MHz, 4H), 7.07 (d, 7.6 MHz, 4H), 2.24 (s, 6H); \textsuperscript{13}C NMR (100 MHz, DMSO-d\textsubscript{6}) δ 152.6, 137.2, 130.5, 129.1, 118.2, 20.3.

9g 1,3-bis(3,5-dimethylphenyl)urea\textsuperscript{(21)}: The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a dark brown solid; mp: 267°C; \textsuperscript{1}H NMR (400 MHz, DMSO-d\textsubscript{6}) δ 8.49 (brs, 2H), 7.07 (s, 4H), 6.60 (s, 2H), 2.22 (s, 12H); \textsuperscript{13}C NMR (100 MHz, DMSO-d\textsubscript{6}) δ 152.4, 21.1, 139.6, 137.7, 123.3, 115.8.

9h 1,3-bis(4-butylphenyl)urea\textsuperscript{(22)}: The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a light brown solid; mp: 180-181°C (lit.\textsuperscript{20} 192-195°C); \textsuperscript{1}H NMR (400 MHz, DMSO-d\textsubscript{6}) δ 8.51 (brs, 2H), 7.34 (d, 8.4 MHz, 4H), 7.08 (d, 8.4 MHz, 4H), 2.49-2.52 (m, 4H), 1.45-1.55 (m, 4H), 1.25-1.34 (m, 4H), 0.89 (t, 7.2 MHz, 6H); \textsuperscript{13}C NMR (100 MHz, DMSO-d\textsubscript{6}) δ 152.6, 137.4, 135.5, 128.5, 118.2, 34.1, 33.3, 21.7, 13.8.

9i 1,3-Bis(4-chlorophenyl)urea\textsuperscript{(19)}: The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a brown solid; mp: 278-280°C (lit.\textsuperscript{20} 322-325°C); \textsuperscript{1}H NMR (400 MHz, DMSO-d\textsubscript{6}) δ 8.89 (brs, 2H), 7.48 (d, 8.0 MHz, 4H), 7.33 (d, 8.4 MHz, 4H); \textsuperscript{13}C NMR (100 MHz, DMSO-d\textsubscript{6}) δ 152.3, 138.5, 128.6, 125.5, 119.8.
The title compound was prepared according to the general procedure and purified by heating to remove the reactant to give the desired product which was a brown sticky oil. \( ^1\text{H NMR} \) (400 MHz, DMSO-d6) \( \delta \) 3.13-3.57 (m, 16H); \( ^{13}\text{C NMR} \) (100 MHz, DMSO-d6) \( \delta \) 162.6, 66.2, 65.9, 65.7, 46.8, 45.8, 40.6.
5. NMR spectra of the products
Catalyst used one time
Catalyst used two times

Catalyst used three times
6. References


7. Author contributions

S. L. developed the reactions and expanded the scope. H. W. conceived and prepared the manuscript. X. D. tested the Pd dispersion of bulk Pd catalyst and discussed the results. F.S. conceived and supervised the project as well as revised the manuscript.