Palladium-Catalyzed Carbonylation of Benzylic Ammonium Salts via C-N Bond Activation under atmosphere pressure

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Supporting Information

Part 1. General informations.................................................................S1
Part 2. Optimization details.............................................................S2
Part 3. Synthesis of Benzylic Ammonium Salts....................................S6
Part 4. Scale-up experiment.............................................................S11
Part 5. Characterization of carbonylation products of amines..............S13
Part 6. Characterization of carbonylation products of esters(reaction with alcohols).....S26
Part 7. Characterization of carbonylation products of esters(reaction with phenols)....S33
Part 8. References.............................................................................S40
Part 9. $^1$H NMR and $^{13}$C NMR Spectra.............................................S41
Part 1. General informations

1. Analytical methods. All the reactions were monitored by thin-layer chromatography (TLC) or LC-MS (EI); products purification was done using silica gel column chromatography. $^1$H/$^{13}$C NMR spectra were recorded on Bruker avance 400 MHz and Bruker AMX 400 MHz spectrometer at 400/100 MHz, respectively, in CDCl$_3$ unless otherwise stated, using either TMS or the undeuterated solvent residual signal as the reference. Chemical shifts are given in ppm and are measured relative to CDCl$_3$ or DMSO-d$_6$ as an internal standard. Mass spectra were obtained by the electrospray ionization time-of-flight (ESI-TOF) mass spectrometry. GC yields were obtained using biphenyl as an internal standard. Flash column chromatography purification of compounds was carried out by gradient elution using ethyl acetate (EA) in light petroleum ether (PE). Melting points were determined on an X-4 digital display microscope apparatus.
Part 2. Optimization details

**General Procedure A**: Carbonylation of 1a and 2a with CO (Table 1)

Ammonium salts 1a (119.6 mg, 0.40 mmol), PdCl₂(dppf) (4.4 mg, 0.006 mmol), PPh₃ (15.7 mg, 0.06 mmol) and Na₂CO₃ (42.4 mg, 0.40 mmol) were added into a 25 mL Schlenk tube equipped with a magnetic stirred bar. The reaction mixture was degassed 3 times by CO and stirred under CO balloon. Toluene (1.0 mL), amine 2a (18.6 mg, 0.20 mmol) and DMSO (0.2 mL) were then added into the tube by syringe. The reaction mixture was heated to 100 ºC and monitored by TLC. Upon completion, the reaction was diluted by AcOEt (5 mL), The reaction was extracted with EA (20 mL x 3). The combined organic layers were washed with brine (20 mL) and dried over Na₂SO₄, filtered, and concentrated in vacuo. Purification by flash column chromatography (PE/EA = 4:1) afford the amide 3aa. Alternatively, 1,1'-biphenyl (30.8 mg, 0.2 mmol) was added into the residue as internal standard (k = 1.235), and the yield of the product 3aa was determined by GC analysis.

**Table 1. Optimization for the reaction of N,N,N-trimethyl-1-phenylmethan-aminium trifluoromethanesulfonate (1a) and aniline (2a)**

<table>
<thead>
<tr>
<th>Entry</th>
<th>Cat. (X mol%)</th>
<th>Ligand (Y mol%)</th>
<th>Solvent</th>
<th>Base</th>
<th>Yield⁵ (%)</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>PdCl₂ (5)</td>
<td>PPh₃ (10)</td>
<td>PhMe/DMSO</td>
<td>EtN₃</td>
<td>22</td>
</tr>
<tr>
<td>2</td>
<td>Pd(PPh₃)ₓ₄ (5)</td>
<td>PPh₃ (10)</td>
<td>PhMe/DMSO</td>
<td>EtN₃</td>
<td>34</td>
</tr>
<tr>
<td>3</td>
<td>PdCl₂(PPh₃)₂ (5)</td>
<td>PPh₃ (10)</td>
<td>PhMe/DMSO</td>
<td>EtN₃</td>
<td>48</td>
</tr>
<tr>
<td>4</td>
<td>PdCl₂(dppf) (5)</td>
<td>PPh₃ (10)</td>
<td>PhMe/DMSO</td>
<td>EtN₃</td>
<td>66 (65)⁶</td>
</tr>
<tr>
<td>5</td>
<td>Pd(OAc)₂ (5)</td>
<td>PPh₃ (10)</td>
<td>PhMe/DMSO</td>
<td>EtN₃</td>
<td>trace</td>
</tr>
<tr>
<td>6</td>
<td>Pd(TFA)₂ (5)</td>
<td>PPh₃ (10)</td>
<td>PhMe/DMSO</td>
<td>EtN₃</td>
<td>trace</td>
</tr>
<tr>
<td>7</td>
<td>Pd(acac)₂ (5)</td>
<td>PPh₃ (10)</td>
<td>PhMe/DMSO</td>
<td>EtN₃</td>
<td>trace</td>
</tr>
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<td>8</td>
<td>PdCl₂(dppf) (5)</td>
<td>BINAP (10)</td>
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<td>EtN₃</td>
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<td>9</td>
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<td>EtN₃</td>
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<td>11</td>
<td>PdCl₂(dppf) (5)</td>
<td>1,10-Phen (10)</td>
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<td>EtN₃</td>
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<td>PPh₃ (10)</td>
<td>PhMe/DMSO</td>
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<td>trace</td>
</tr>
<tr>
<td></td>
<td>Reaction Conditions</td>
<td>Product</td>
<td>Isolated Yield (%)</td>
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<td></td>
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<tr>
<td>---</td>
<td>---------------------</td>
<td>---------</td>
<td>--------------------</td>
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<tr>
<td>15</td>
<td>PdCl₂(dppf) (5) PPh₃ (10) PhMe/DMSO KO'Bu</td>
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<tr>
<td>16</td>
<td>PdCl₂(dppf) (5) PPh₃ (10) PhMe/DMSO K₂CO₃</td>
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<td>19</td>
<td>PdCl₂(dppf) (5) PPh₃ (10) DMF Na₂CO₃</td>
<td>trace</td>
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<td></td>
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<td>20</td>
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<td>22</td>
<td>PdCl₂(dppf) (5) PPh₃ (10) (PhMe/DMSO) Na₂CO₃</td>
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<td>PdCl₂(dppf) (5) PPh₃ (5) (PhMe/DMSO) Na₂CO₃</td>
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<td>26</td>
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<tr>
<td>27</td>
<td>PdCl₂(dppf) (3) PPh₃ (30) (PhMe/DMSO) Na₂CO₃</td>
<td>97 (97)</td>
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<td></td>
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<td>28</td>
<td>PdCl₂(dppf) (3) PPh₃ (30) (PhMe/DMSO) Na₂CO₃</td>
<td>87</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>29</td>
<td>PdCl₂(dppf) (3) PPh₃ (30) (PhMe/DMSO) Na₂CO₃</td>
<td>trace</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>PdCl₂(dppf) (3) PPh₃ (30) (PhMe/DMSO) Na₂CO₃</td>
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General Procedure B: Carbonylation of 1a and 4a with CO (Table 2)

Ammonium salts 1a (59.8 mg, 0.2 mmol), PdCl₂(dppf) (14.6 mg, 0.02 mmol), and Na₂CO₃ (21.2 mg, 0.2 mmol) was added to a 25 mL schlenk tube equipped with a magnetic stirred bar, and a balloon filled with CO was connected to the Schlenk tube through the side arm after exhaust the air, benzyl alcohol 2a (108 mg, 1.0 mmol) and toluene (2.0 mL) were then injected into the tube by syringe. The reaction was then heated to 100 °C and stirred corresponding time. Upon completion, the reaction was quenched by AcOEt (5 mL). The reaction was extracted with EA (20 mL x 3). The combined organic layers were washed with brine (20 mL) and dried over Na₂SO₄, filtered, and concentrated in vacuo. Purification by flash column chromatography (PE/EA = 50:1) afford the amide 3aa. Alternatively, 1,1'-biphenyl (30.8 mg, 0.2 mmol) was added into the residue as internal standard (k=1.108). The yield of the product 5aa was determined by GC analysis.

Table 2. Optimization for the reaction of N,N,N-trimethyl-1-phenylmethan-1-aminium trifluoromethanesulfonate (1a) and benzyl alcohol (4a)
<table>
<thead>
<tr>
<th>Entry</th>
<th>Cat. (X mol%)</th>
<th>Ligand (Y mol%)</th>
<th>Solvent</th>
<th>Base</th>
<th>Yield(^b) (%)</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>PdCl(_2)(dpf) (5)</td>
<td>PPh(_3) (10)</td>
<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
<td>25</td>
</tr>
<tr>
<td>2</td>
<td>Pd(PPh(_3))(_4) (5)</td>
<td>PPh(_3) (10)</td>
<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
<td>17</td>
</tr>
<tr>
<td>3</td>
<td>PdCl(_2)(PPh(_3))(_2) (5)</td>
<td>PPh(_3) (10)</td>
<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
<td>trace</td>
</tr>
<tr>
<td>4</td>
<td>Pd(acac)(_2) (5)</td>
<td>PPh(_3) (10)</td>
<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
<td>trace</td>
</tr>
<tr>
<td>5</td>
<td>Pd(OAc)(_2) (5)</td>
<td>PPh(_3) (10)</td>
<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
<td>ND</td>
</tr>
<tr>
<td>6</td>
<td>PdCl(_2) (5)</td>
<td>PPh(_3) (10)</td>
<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
<td>ND</td>
</tr>
<tr>
<td>7</td>
<td>Pd(_2)(dba)(_3) (5)</td>
<td>PPh(_3) (10)</td>
<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
<td>ND</td>
</tr>
<tr>
<td>8</td>
<td>PdCl(_2)(dpf) (5)</td>
<td>BINAP (10)</td>
<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
<td>trace</td>
</tr>
<tr>
<td>9</td>
<td>PdCl(_2)(dpf) (5)</td>
<td>dppe (10)</td>
<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
<td>19</td>
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<td>10</td>
<td>PdCl(_2)(dpf) (5)</td>
<td>dppf (10)</td>
<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
<td>20</td>
</tr>
<tr>
<td>11</td>
<td>PdCl(_2)(dpf) (5)</td>
<td>Xantphos (10)</td>
<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
<td>ND</td>
</tr>
<tr>
<td>12</td>
<td>PdCl(_2)(dpf) (5)</td>
<td>--</td>
<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
<td>24</td>
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<td>13</td>
<td>PdCl(_2)(dpf) (5)</td>
<td>--</td>
<td>1,4-dioxane</td>
<td>Na(_2)CO(_3)</td>
<td>trace</td>
</tr>
<tr>
<td>14</td>
<td>PdCl(_2)(dpf) (5)</td>
<td>--</td>
<td>DMF</td>
<td>Na(_2)CO(_3)</td>
<td>ND</td>
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<tr>
<td>15</td>
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<td>--</td>
<td>PhCH(_2)OH</td>
<td>Na(_2)CO(_3)</td>
<td>72 (70)c</td>
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<tr>
<td>16</td>
<td>PdCl(_2)(dpf) (5)</td>
<td>--</td>
<td>PhMe/DMSO (1:1)</td>
<td>Na(_2)CO(_3)</td>
<td>17</td>
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<tr>
<td>17</td>
<td>PdCl(_2)(dpf) (5)</td>
<td>--</td>
<td>PhMe/DMF (1:1)</td>
<td>Na(_2)CO(_3)</td>
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<tr>
<td>18(^d)</td>
<td>PdCl(_2)(dpf) (5)</td>
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<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
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<tr>
<td>19(^e)</td>
<td>PdCl(_2)(dpf) (5)</td>
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<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
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</tr>
<tr>
<td>20(^f)</td>
<td>PdCl(_2)(dpf) (5)</td>
<td>--</td>
<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
<td>79</td>
</tr>
<tr>
<td>21(^g)</td>
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<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
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<td>22(^h)</td>
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<td>Na(_2)CO(_3)</td>
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<td>23(^i)</td>
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<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
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<tr>
<td>24</td>
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<td>PhMe</td>
<td>K(_2)CO(_3)</td>
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<td>PhMe</td>
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<td>PhMe</td>
<td>NaHCO(_3)</td>
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<td>Na(_2)CO(_3)</td>
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<td>29</td>
<td>PdCl(_2)(dpf) (10)</td>
<td>--</td>
<td>PhMe</td>
<td>Na(_2)CO(_3)</td>
<td>84 (82)</td>
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</table>
General Procedure C: Carbonylation of 1a and 6a with CO  (Table 3)

Ammonium salts 1a (59.8 mg, 0.2 mmol), PdCl₂(dppf) (14.6 mg, 0.02 mmol), dppp (16.5 mg, 0.04 mmol), and Na₂CO₃ (21.2 mg, 0.2 mmol) was added to a 25 mL schlenk tube equipped with a magnetic stirred bar, and a balloon filled with CO was connected to the Schlenk tube through the side arm after exhaust the air, phenol 6a (56.4 mg, 0.6 mmol) and toluene (2.0 mL) were then injected into the tube by syringe. The reaction was then heated to 100 ºC and stirred corresponding time. Upon completion, the reaction was quenched by AcOEt (5 mL). The reaction was extracted with EA (20 mL x 3). The combined organic layers were washed with brine (20 mL) and dried over Na₂SO₄, filtered, and concentrated in vacuo. Purification by flash column chromatography (PE/EA = 50:1) afford the amide 3aa. Alternatively, 1,1′-biphenyl (30.8 mg, 0.2 mmol) was added into the residue as internal standard (k=1.026). The yield of the product 7aa was determined by GC analysis.

Table 3. Optimization for the reaction of N,N,N-trimethyl-1-phenylmethan -aminium trifluoromethanesulfonate (1a) and phenol (4a)³
<table>
<thead>
<tr>
<th>Entry</th>
<th>Cat. (X mol%)</th>
<th>Ligand (Y mol%)</th>
<th>Solvent</th>
<th>Base</th>
<th>Yieldb (%)</th>
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<td>Na₂CO₃</td>
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<td>PdCl₂(dppf) (10)</td>
<td>TFP (20)</td>
<td>PhMe</td>
<td>Na₂CO₃</td>
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<td>BINAP (20)</td>
<td>PhMe</td>
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<td>Na₂CO₃</td>
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<td>Na₂CO₃</td>
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<td>8</td>
<td>Pd(PPh₃)₄ (10)</td>
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<td>PhMe</td>
<td>Na₂CO₃</td>
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<tr>
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<td>PdCl₂ (10)</td>
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<td>Na₂CO₃</td>
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<td>10</td>
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<td>PPh₃ (20)</td>
<td>PhMe</td>
<td>Na₂CO₃</td>
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<td>Pd(OAc)₂ (10)</td>
<td>PPh₃ (20)</td>
<td>PhMe</td>
<td>Na₂CO₃</td>
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<td>Na₂CO₃</td>
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*aReaction condition: 1a (0.2 mmol), 6a (0.6 mmol), catalyst (X mol%), ligand (Y mol%), base (0.2 mmol) in PhMe (2.0 mL) under 1 atm CO at 100 °C for 12 h. bYield were determined by GC analysis. cIsolated yield; dNa₂CO₃ (0.4 mmol 2.0 equiv); eNa₂CO₃ (0.1 mmol 0.5 equiv); f90 °C instead of 100°C; g110 °C instead of 100°C.
Part 3: Synthesis of Benzylic Ammonium Salts

General Procedure D (Preparation of Benzyl Ammonium Triflates): Preparation of N,N,N-Trimethyl-1-phenylmethanaminium trifluoromethanesulfonate

Dimethylbenzylamine (2.5 g, 18.5 mmol, 1.0 equiv) was dissolved in Et₂O (15 mL, 4.0 M). MeOTf (2.7 mL, 23.9 mmol, 1.3 equiv) was added dropwise at 0 °C. White precipitate formed immediately. After complete addition the reaction mixture was stirred for an additional 30 minutes at 0 °C. The precipitate was isolated by filtration and washed with Et₂O (2 x 20 mL). The resulting solid was dried under vacuum to give salt 1a (5.17 g, 94%) as a white solid (mp 97–99 °C).

Substituted dimethyl benzyl amines were prepared either from the benzyl amines using Escheweiler–Clarke conditions or via reductive amination of the benzaldehyde or acetophenone derivative.

N,N,N-trimethyl-1-phenylmethanaminium trifluoromethanesulfonate, was synthesized following the procedure D from dimethyl benzyl amine, white solid, 95%, Mp: 97-99 °C, Analytical data were in agreement with previous reports.

\[
\text{N,N,N-trimethyl-1-phenylmethanaminium trifluoromethanesulfonate, was synthesized following the procedure D from corresponding dimethyl benzyl amine, white solid, 95%, Mp: 110-113 °C, } \ \text{Analytical data were in agreement with previous reports.} \]

\[
\text{N,N,N-trimethyl-1-(p-tolyl)methanaminium trifluoromethanesulfonate, was synthesized following the procedure D from corresponding dimethyl benzyl amine, white solid, 90%, Mp: 116-118 °C. } \ \text{HRMS(ESI) calcld for C}_{11}\text{H}_{18}\text{N}^+[\text{M-OTf}]^- \text{ 164.1434, found 164.1442.}
\]

\[
\text{N,N,N-trimethyl-1-(o-tolyl)methanaminium trifluoromethanesulfonate, was synthesized following the procedure D from corresponding dimethyl benzyl amine, white solid, 90%, Mp: 116-118 °C. } \ \text{HRMS(ESI) calcld for C}_{12}\text{H}_{18}\text{N}^+[\text{M-OTf}]^- \text{ 164.1434, found 164.1438.}
\]
1-(4-methoxyphenyl)-N,N,N-trimethylmethanaminium trifluoromethanesulfonate, was synthesized following the procedure D from corresponding dimethyl benzyl amine, white soild, 95%, Mp: 94-95°C, Analytical data were in agreement with previous reports.\textsuperscript{3}

1-(3-methoxyphenyl)-N,N,N-trimethylmethanaminium, trifluoromethanesulfonate, was synthesized following the procedure D from corresponding dimethyl benzyl amine, light brown soild, 89%, Mp: 59-62°C, Analytical data were in agreement with previous reports.\textsuperscript{3}

1-(4-fluorophenyl)-N,N,N-trimethylmethanaminium trifluoromethanesulfonate, was synthesized following the procedure D from corresponding dimethyl benzyl amine, white soild, 89%, Mp: 130-132°C. \textsuperscript{1}H NMR (400 MHz, DMSO-d\textsubscript{6}) \(\delta\) 7.66 – 7.55 (m, 2H), 7.41 – 7.31 (m, 2H), 4.51 (d, \(J = 3.2\) Hz, 2H), 3.01 (d, \(J = 2.7\) Hz, 9H). \textsuperscript{13}C NMR (101 MHz, DMSO-d\textsubscript{6}) \(\delta\) 163.7 (d, \(J_{C,F} = 247.6\) Hz), 135.6 (d, \(J_{C,F} = 8.8\) Hz), 125.2, 121.2 (d, \(J_{C,F} = 322.9\) Hz), 116.4 (d, \(J_{C,F} = 22.2\) Hz), 67.5, 52.2. FTIR(KBr): 3036, 1488, 1261, 1156, 1035 HRMS(ESI) calcd for C\textsubscript{10}H\textsubscript{15}FN\textsuperscript+[M-OTf]\textsuperscript{+} 168.1183, found 168.1189.

1-(4-chlorophenyl)-N,N,N-trimethylmethanaminium trifluoromethanesulfonate, was synthesized following the procedure D from corresponding dimethyl benzyl amine, white soild, 90%, Mp: 120-122°C. \textsuperscript{1}H NMR (400 MHz, DMSO) \(\delta\) 7.62 – 7.54 (m, 4H), 4.54 (dd, \(J = 19.5, 2.9\) Hz, 2H), 3.02 (d, \(J = 2.3\) Hz, 9H). \textsuperscript{13}C NMR (101 MHz, DMSO) \(\delta\) 135.8, 135.1, 129.5, 127.8, 121.2 (d, \(J = 320.3\) Hz), 67.4, 52.3. FTIR(KBr): 3038, 1488, 1261, 1156, 1035. HRMS(ESI) calcd for C\textsubscript{10}H\textsubscript{15}ClN\textsuperscript+[M-OTf]\textsuperscript{+} 184.0888, found 184.0892.
1-(3,4-difluorophenyl)-N,N,N-trimethylmethanaminium trifluoromethanesulfonate, was synthesized following the procedure D from corresponding dimethyl benzyl amine, white solid, 88%, Mp: 117-119 °C. Analytical data were in agreement with previous reports.\(^3\)

\[
\begin{array}{c}
\text{F}_3\text{C} \quad \text{procedure D} \quad \text{F}_3\text{C} \\
\text{NMe}_2 \quad \text{OTf} \quad \text{NMe}_3\text{OTf}
\end{array}
\]

N,N,N-trimethyl-1-(4-(trifluoromethyl)phenyl)methanaminium trifluoromethanesulfonate, was synthesized following the procedure D from corresponding dimethyl benzyl amine, white solid, 90%, Mp: 117-119 °C. Analytical data were in agreement with previous reports.

1-(4-cyanophenyl)-N,N,N-trimethylmethanaminium trifluoromethanesulfonate, was synthesized following the procedure D from corresponding dimethyl benzyl amine, white solid, 90%, Mp: 133-135 °C. \(^1\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) 8.02 (d, \(J = 8.2\) Hz, 2H), 7.74 (d, \(J = 8.2\) Hz, 2H), 4.61 (s, 2H), 3.05 (s, 9H).

\[^{13}\text{C}\) NMR (101 MHz, DMSO-\(d_6\)) \(\delta\) 134.2, 133.9, 133.2, 121.2 (d, \(J_{C-F} = 320.6\) Hz), 118.7, 113.6, 67.4, 52.6. FTIR(KBr): 3038, 2230, 1485, 1493, 1285, 1260, 1156, 1028. HRMS(ESI) calcd for C\(_{11}\)H\(_{15}\)N\(_2\)[M-OTf]\(^+\) 175.1230, found 175.1238

1-(4-(methoxycarbonyl)phenyl)-N,N,N-trimethylmethanaminium trifluoromethane sulfonate, was synthesized following the procedure D from corresponding dimethyl benzyl amine, white solid, 92%, Mp: 168-171 °C. \(^1\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) 8.08 (d, \(J = 7.6\) Hz, 2H), 7.69 (d, \(J = 8.1\) Hz, 2H), 4.60 (d, \(J = 3.0\) Hz, 2H), 3.89 (d, \(J = 1.5\) Hz, 3H), 3.05 (d, \(J = 3.5\) Hz, 9H). \[^{13}\text{C}\) NMR (101 MHz, DMSO-\(d_6\)) \(\delta\) 166.2, 133.7, 131.7, 130.0, 121.2 (d, \(J_{C-F} = 320.6\) Hz), 67.6, 52.9, 52.5. FTIR(KBr): 3034, 1728, 1282, 1259, 1163, 1032, 1032. HRMS(ESI) calcd for C\(_{12}\)H\(_{18}\)NO\(_2\)[M-OTf]\(^+\) 208.1332, found 208.1340

1-(4-(tert-butoxycarbonyl)phenyl)-N,N,N-trimethylmethanaminium trifluoromethanesulfonate, was synthesized following the procedure D from corresponding dimethyl benzyl amine, white solid, 92%, Mp: 114-116 °C. \(^1\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) 8.02 (d, \(J = 8.2\) Hz, 2H), 7.66 (d, \(J = 9.5\) Hz, 2H), 4.59 (d, \(J = 3.0\) Hz, 2H), 3.89 (d, \(J = 1.5\) Hz, 3H), 3.05 (d, \(J = 3.5\) Hz, 9H). \[^{13}\text{C}\) NMR (101 MHz, DMSO-\(d_6\)) \(\delta\) 134.2, 133.9, 133.2, 121.2 (d, \(J_{C-F} = 320.6\) Hz), 67.6, 52.9, 52.5. FTIR(KBr): 3034, 1728, 1282, 1259, 1163, 1032, 1032. HRMS(ESI) calcd for C\(_{12}\)H\(_{18}\)NO\(_2\)[M-OTf]\(^+\) 208.1332, found 208.1340
2.2 Hz, 2H), 3.04 (d, \( J = 2.4 \) Hz, 9H), 1.56 (s, 9H). \(^{13}\)C NMR (101 MHz, DMSO-\(d_6\)) \( \delta \) 164.9, 133.6, 133.4, 133.2, 129.9, 121.2 (d, \( J_{C-F} = 314.3 \) Hz), 81.8, 67.7, 52.5, 28.2. FTIR(KBr): 3037, 2994, 1696, 1492, 1304, 1141, 1123, 1031. HRMS(ESI) calcd for C\(_{13}\)H\(_{24}\)NO\(_2\)\([\text{M-OTf}]^+\) 250.1802, found 250.1809

\[
\begin{align*}
\text{N,N,N-trimethyl-1-(naphthalen-2-yl)methanaminium trifluoromethanesulfonate,} & \\
\text{was synthesized following the procedure D from corresponding dimethyl benzyl amine, white solid, 90%, Mp: 108-110 \degree C,} \\
\text{Analytical data were in agreement with previous reports.}^3
\end{align*}
\]
Part 4: Scale-up experiment

According to Procedure A, ammonium salts 1a (2.99 g, 10 mmol), PdCl₂(dppf) (109.8 mg, 0.15 mmol), PPh₃ (393 mg, 1.5 mmol), and Na₂CO₃ (1.06 g, 10 mmol) was added to a 50 mL schlenk flask equipped with a magnetic stirred bar, a and a balloon filled with CO was connected to the Schlenk tube through the side arm after exhaust the air, DMSO (2 mL), amine 2a (18.6 mg, 0.2 mmol) and toluene (10 mL) were then injected into the tube by syringe. The reaction was then heated to 100 ºC and stirred 18 h (Fig 1). Upon completion, the reaction was quenched by AcOEt (5 mL). The reaction was extracted with EA (60 mL x 3). The combined organic layers were washed with brine (60 mL) and dried over Na₂SO₄, filtered, and concentrated in vacuo. Purification by flash column chromatography (silica gel, hexanes:EtOAc= 4:1), 0.72 g (68%) 3aa was obtained as light yellow solid.

According to Procedure B, ammonium salts 1a (1.50 g, 5 mmol), PdCl₂(dppf) (366 mg, 0.5 mmol), and Na₂CO₃ (530 mg, 5 mmol) was added to a 50 mL schlenk flask equipped with a magnetic stirred bar, and a balloon filled with CO was connected to the Schlenk tube through the side arm after exhaust the air,, benzyl alchol 4a (10 mL) were then injected into the tube by syringe. The reaction was then heated to 100 ºC and stirred 16 h. Upon completion. The reaction was extracted with EA
(60 mL x 3). The combined organic layers were washed with brine (60 mL) and dried over Na₂SO₄, filtered, and concentrated in vacuo. Purification by flash column chromatography (silica gel, hexanes:EtOAc= 100:1), 0.97 g (86%) 5aa was obtained as yellow liquid.

According to Procedure C, ammonium salts 1a (1.50 g, 5 mmol), PdCl₂(dppf) (370 mg, 0.5 mmol), dppp (415 mg, 1 mmol), Na₂CO₃ (530 mg, 5 mmol), and phenol 6a (1.4 g, 15 mmol) was added to a 50 mL schlenk flask equipped with a magnetic stirred bar, and a balloon filled with CO was connected to the Schlenk tube through the side arm after exhaust the air, toluene (10 mL) were then injected into the tube by syringe. The reaction was then heated to 100 ºC and stirred 12 h (Fig 1). Upon completion, the reaction was quenched by AcOEt (5 mL). The reaction was extracted with EA (60 mL x 3). The combined organic layers were washed with brine (60 mL) and dried over Na₂SO₄, filtered, and concentrated in vacuo. Purification by flash column chromatography (silica gel, hexanes:EtOAc= 100:1), 0.74 g (70%) 3aa was obtained as colorless liquid.
Part 5. Characterization of carbonylation products of amines (3aa—3bj)

N,2-diphenylacetamide (3aa)\(^4,5\), this compound was prepared according to General Procedure A for 12 h, Yield: 97% (40.9 mg), light yellow solid. Mp: 118 – 120°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.45 – 7.29 (d, 7H), 7.28 – 7.23 (m, 2H), 7.08 (t, \(J = 7.4\) Hz, 1H), 3.70 (s, 2H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 169.2, 137.7, 134.6, 129.5, 129.2, 128.9, 127.6, 124.5, 120.0, 44.8. HRMS(ESI\(^+\)) calculated for \(\text{C}_{14}\text{H}_{13}\text{NO} [\text{M+H}]^+: 212.1075\); found: 212.1077.

N-phenyl-2-(o-tolyl)acetamide (3ab)\(^4\), this compound was prepared according to General Procedure A for 12 h, Yield: 95% (42.6 mg), light yellow solid. Mp: 133 - 135°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.38 (d, \(J = 7.9\) Hz, 2H), 7.28 – 7.21 (m, 7H), 7.06 (t, \(J = 7.2\) Hz, 1H), 3.70 (s, 2H), 2.32 (s, 3H). \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 169.1, 137.6, 137.3, 133.0, 130.9, 130.5, 128.9, 128.1, 126.8, 124.4, 120.0, 42.8, 19.5. HRMS(ESI\(^+\)) calculated for \(\text{C}_{15}\text{H}_{15}\text{NO} [\text{M+H}]^+: 226.1232\); found: 226.1237.

N-phenyl-2-(p-tolyl)acetamide (3ac)\(^5\), this compound was prepared according to General Procedure A for 12 h, Yield: 95% (42.7 mg), yellow solid. Mp: 156 - 158°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.44 (d, \(J = 8.0\) Hz, 2H), 7.29 (dd, \(J = 10.0, 5.4\) Hz, 3H), 7.23 (s, 4H), 7.10 (t, \(J = 7.3\) Hz, 1H), 3.71 (s, 2H), 2.39 (s, 3H). \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 169.5, 137.7, 137.4, 131.4, 130.0, 129.5, 128.9, 128.1, 124.4, 119.9, 44.5, 21.1. HRMS(ESI\(^+\)) calculated for \(\text{C}_{15}\text{H}_{15}\text{NO} [\text{M+Na}]^+: 248.1051\); found: 248.1055.

2-(4-methoxyphenyl)-N-phenylacetamide (3ad)\(^5\), this compound was prepared according to General Procedure A for 10 h, Yield: 96% (46.2 mg), yellow solid. Mp: 115 - 117°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.44 (s, 1H), 7.34 (d, \(J = 7.9\) Hz, 2H), 7.15 (dd, \(J = 18.4, 8.2\) Hz, 4H), 6.98 (t, \(J = 7.3\) Hz, 1H), 6.80 (d, \(J = 8.2\) Hz, 2H), 3.71 (s, 3H), 3.54 (s, 2H). \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 169.8, 159.1, 137.8, 130.6, 128.9, 126.5, 124.4, 119.9, 114.5, 55.3, 43.8. HRMS(ESI\(^+\)) calculated for \(\text{C}_{15}\text{H}_{15}\text{NO}_2 [\text{M+Na}]^+: 264.1000\); found: 264.1004.
2-(4-fluorophenyl)-N-phenylacetamide (3ae), this compound was prepared according to General Procedure A for 16 h, Yield: 81% (37.2 mg), light yellow solid. Mp: 124 - 126°C; ¹H NMR (400 MHz, CDCl₃) δ 7.43 (d, J = 8.0 Hz, 2H), 7.31 – 7.22 (m, 4H), 7.11 – 7.01 (m, 3H), 3.66 (s, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 169.0, 162.24 (d, Jₖ₋₈ = 246.4 Hz), 137.6, 131.1 (d, Jₖ₋₈ = 8.0 Hz), 130.3 (d, Jₖ₋₈= 3.4 Hz), 129.0, 124.6, 120.0, 116.0 (d, Jₖ₋₈ = 22.5 Hz), 43.8; HRMS(ESI⁺) calculated for C₁₄H₁₂FNO [M+H]⁺: 230.0981; found: 230.0978.

2-(4-chlorophenyl)-N-phenylacetamide (3af), this compound was prepared according to General Procedure A for 12 h, Yield: 89% (43.7 mg), light yellow solid. Mp: 170 - 172°C; ¹H NMR (400 MHz, DMSO-d₆) δ 10.19 (s, 1H), 7.60 (d, J = 7.9 Hz, 2H), 7.37 (q, J = 8.6 Hz, 4H), 7.30 (t, J = 7.8 Hz, 2H), 7.04 (t, J = 7.3 Hz, 1H), 3.66 (s, 2H). ¹³C NMR (100 MHz, DMSO-d₆) δ 168.8, 139.2, 135.1, 131.4, 131.1, 128.8, 128.3, 123.4, 119.2, 42.5. FTIR(KBr): 1668 cm⁻¹ (CH₂CONR₁R₂), HRMS(ESI⁺) calculated for C₁₄H₁₂ClNO [M+H]⁺: 246.0686; found: 246.0685

2-(4-cyanophenyl)-N-phenylacetamide (3ag), this compound was prepared according to General Procedure A for 14 h, Yield: 85% (40.3 mg), yellow solid. Mp: 156 - 159°C; ¹H NMR (400 MHz, DMSO) δ 10.26 (s, 1H), 7.81 (d, J = 8.1 Hz, 2H), 7.62 (d, J = 7.8 Hz, 2H), 7.50 (d, J = 7.9 Hz, 2H), 7.31 (t, J = 7.8 Hz, 2H), 7.05 (t, J = 7.3 Hz, 1H), 3.79 (s, 2H). ¹³C NMR (100 MHz, DMSO-d₆) δ 168.2, 141.9, 139.1, 132.2, 130.4, 128.8, 123.5, 119.3, 119.0, 109.6, 43.2. FTIR(KBr): 1664 cm⁻¹ (CH₂CONR₁R₂), HRMS(ESI⁺) calculated for C₁₅H₁₂N₂O [M+Na]⁺: 237.1028; found: 237.1032.

Methyl 4-(2-oxo-2-(phenylamino)ethyl)benzoate (3ah), this compound was prepared according to General Procedure A for 12 h, Yield: 94% (50.8 mg), white solid. Mp: 164 - 166°C; ¹H NMR (400 MHz, DMSO-d₆) δ 10.25 (s, 1H), 7.95 (d, J = 8.0 Hz, 2H), 7.62 (d, J = 7.8 Hz, 2H), 7.50 (d, J = 7.9 Hz, 2H), 7.31 (t, J = 7.7 Hz, 2H), 7.05 (t, J = 7.3 Hz, 1H), 3.85 (s, 3H), 3.77 (s, 2H). ¹³C NMR (100 MHz, DMSO-d₆) δ 168.5, 166.2, 141.7, 139.2, 129.7, 129.3, 128.9, 128.1, 123.4, 119.2, 52.1, 43.3. FTIR(KBr): 1664 cm⁻¹ (CH₂CONR₁R₂), HRMS(ESI⁺) calculated for C₁₆H₁₅NO₃[M+Na]⁺: 292.0950; found: 292.0953.
N-phenyl-2-(4-(trifluoromethyl)phenyl)acetamide (3ai), this compound was prepared according to General Procedure A for 8 h, Yield: 98% (54.7 mg), light red solid, Mp: 135 - 138°C; 1H NMR (400 MHz, DMSO-d$_6$) δ 10.25 (s, 1H), 7.70 (d, J = 8.0 Hz, 2H), 7.63 (d, J = 8.1 Hz, 2H), 7.58 (d, J = 8.0 Hz, 2H), 7.31 (t, J = 7.8 Hz, 2H), 7.06 (t, J = 7.3 Hz, 1H), 3.80 (s, 2H). 13C NMR (100 MHz, DMSO-d$_6$) δ 168.8, 141.2 (d, J$_{C-F}$ = 1.0 Hz), 139.5, 130.5, 129.2, 127.8 (d, J$_{C-F}$ = 31.4 Hz), 125.5 (q, J$_{C-F}$ = 7.1 Hz), 124.9 (d, J$_{C-F}$ = 270.2 Hz), 123.8, 119.7, 43.4. FTIR(KBr): 1658 cm$^{-1}$ (CH$_2$CONR$_1$R$_2$), HRMS(ESI$^+$) calculated for C$_{15}$H$_{12}$F$_3$NO[M+H]$^+$: 280.0949; found: 280.0947.

2-(3,4-difluorophenyl)-N-phenylacetamide (3aj), this compound was prepared according to General Procedure A for 8 h, Yield: 95% (47.2 mg), light yellow solid, Mp: 108 - 110°C; 1H NMR (400 MHz, DMSO-d$_6$) δ 10.19 (s, 1H), 7.62 (d, J = 7.9 Hz, 2H), 7.40 (dd, J = 16.2, 8.6 Hz, 2H), 7.31 (t, J = 7.7 Hz, 2H), 7.19 (s, 1H), 7.05 (t, J = 7.3 Hz, 1H), 3.69 (s, 2H). 13C NMR (100 MHz, DMSO-d$_6$) δ 168.6, 150.1 (dd, J$_{C-F}$ = 70.3, 12.4 Hz), 147.6 (dd, J$_{C-F}$ = 69.3, 12.5 Hz), 139.2, 133.7 (dd, J$_{C-F}$ = 6.3, 3.8 Hz), 128.9, 126.1 (dd, J$_{C-F}$ = 6.3, 3.3 Hz), 123.4, 119.3, 118.3 (d, J$_{C-F}$ = 17.1 Hz), 117.2 (d, J$_{C-F}$ = 16.9 Hz), 42.2. FTIR(KBr): 1655 cm$^{-1}$ (CH$_2$CONR$_1$R$_2$), HRMS(ESI$^+$) calculated for C$_{14}$H$_{11}$F$_2$NO[M+H]$^+$: 248.0887; found: 248.0884.

N,2-diphenylacetamide (3ak), this compound was prepared according to General Procedure A For 14 h, Yield 63% (32.8 mg), white solid, Mp: 118 - 120°C; 1H NMR (400 MHz, DMSO-d$_6$) δ 10.24 (s, 1H), 7.88 (d, J = 8.0 Hz, 3H), 7.85 (s, 1H), 7.63 (d, J = 7.8 Hz, 2H), 7.54 –7.47 (m, 3H), 7.31 (t, J = 7.8 Hz, 2H), 7.05 (t, J = 7.4 Hz, 1H), 3.84 (s, 2H). 13C NMR (100 MHz, DMSO-d$_6$) δ 169.5, 139.7, 134.2, 133.5, 132.3, 129.2, 128.2, 128.1, 126.6, 126.1, 123.72 (s), 119.7, 43.9. FTIR(KBr): 1658 cm$^{-1}$ (CH$_2$CONR$_1$R$_2$); HRMS(ESI$^+$) calculated for C$_{18}$H$_{15}$NO[M+H]$^+$: 262.1232; found: 262.1238.

2-phenyl-N-(p-tolyl)acetamide (3al), this compound was prepared according to General Procedure A for 12 h, Yield: 87% (39.3 mg), white solid, Mp: 128 - 131°C; 1H NMR (400 MHz, CDCl$_3$) δ...
7.38 (s, 1H), 7.31 – 7.24 (m, 2H), 7.21 (d, J = 7.3 Hz, 5H), 6.97 (d, J = 8.0 Hz, 2H), 3.56 (s, 2H), 2.19 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 169.3, 135.2, 134.7, 134.1, 129.5, 129.4, 129.1, 127.5, 120.1, 44.7, 20.9. HRMS(ESI$^+$) calculated for C$_{15}$H$_{15}$NO [M+H]$^+$: 248.1051; found: 248.1053.

N-(4-isopropylphenyl)-2-phenylacetamide (3am), this compound was prepared according to General Procedure A for 8 h, Yield: 96% (48.5 mg), white solid, Mp: 134 - 136°C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.40 (s, 1H), 7.34 (m, 2H), 7.31 (t, J = 5.4 Hz, 5H), 7.11 (d, J = 8.4 Hz, 2H), 3.68 (s, 2H), 2.88 – 2.78 (m, 1H), 1.20 (s, 2H), 1.19 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 169.2, 145.2, 135.4, 134.7, 129.5, 129.1, 127.5, 126.8, 120.2, 44.7, 33.6, 24.0. FTIR(KBr): 1658 cm$^{-1}$ (CH$_2$CONR$_1$R$_2$), HRMS(ESI$^+$) calculated for C$_{17}$H$_{19}$NO$_2$[M+H]$^+$: 276.1364; found: 276.1366.

N-(4-methoxyphenyl)-2-phenylacetamide (3an), this compound was prepared according to General Procedure A for 12 h, Yield: 89% (42.7 mg), light yellow solid, Mp: 121 – 124°C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.38 – 7.25 (m, 8H), 6.77 (d, J = 8.6 Hz, 2H), 3.73 (s, 3H), 3.63 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 169.3, 156.5, 134.8, 130.9, 129.4, 129.0, 127.4, 126.8, 120.2, 44.7, 33.6, 24.0. HRMS(ESI$^+$) calculated for C$_{15}$H$_{15}$NO$_2$[M+H]$^+$: 264.1000; found: 264.1005.

2-phenyl-N-(o-tolyl)acetamide (3ao), this compound was prepared according to General Procedure A for 8 h, Yield 99% (44.6 mg), white solid, Mp: 158 - 161°C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.77 (d, J = 8.0 Hz, 1H), 7.35 – 7.29 (m, 2H), 7.26 (d, J = 7.3 Hz, 3H), 7.08 (t, J = 7.5 Hz, 1H), 6.99 (d, J = 7.2 Hz, 1H), 6.91 (dd, J = 14.6, 7.1 Hz, 2H), 3.67 (s, 2H), 1.80 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 169.1, 135.6, 134.7, 130.4, 129.7, 129.4, 128.3, 127.8, 126.8, 125.0, 122.3, 44.9, 17.1. HRMS(ESI$^+$) calculated for C$_{15}$H$_{15}$NO[M+H]$^+$: 248.1051; found: 248.1052.

N-(2-methoxyphenyl)-2-phenylacetamide (3ap), this compound was prepared according to General Procedure A for 8 h, Yield 99% (47.7 mg), white solid, Mp: 158 - 161°C; $^1$H NMR (400 MHz, CDCl$_3$) δ 8.26 (d, J = 7.8 Hz, 1H), 7.72 (s, 1H), 7.32 – 7.27 (m, 2H), 7.27 – 7.22 (m, 3H), 6.90
(t, J = 7.7 Hz, 1H), 6.83 (t, J = 7.7 Hz, 1H), 6.70 (d, J = 8.0 Hz, 1H), 3.65 (s, 2H), 3.61 (s, 3H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) 168.9, 147.9, 134.6, 129.0, 127.5, 127.5, 123.7, 121.0, 119.5, 110.0, 55.6, 45.1. HRMS(ESI\(^+\)) calculated for C\(_{15}\)H\(_{15}\)NO\(_2\) [M+H]\(^+\): 264.1000; found: 264.1004.

N-([1,1'-biphenyl]-2-yl)-2-phenylacetamide (3aq), this compound was prepared according to General Procedure A for 12 h, Yield 90% (47.7 mg), white solid, Mp: 158 - 161°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.35 (d, J = 8.2 Hz, 1H), 7.35 – 7.29 (m, 2H), 7.29 – 7.23 (m, 3H), 7.19 (s, 2H), 7.11 (d, J = 6.5 Hz, 2H), 7.03 (d, J = 6.5 Hz, 5H), 3.58 (s, 2H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 168.9, 137.6, 134.7, 133.8, 132.1, 129.9, 129.3, 129.1, 128.9, 128.8, 128.3, 127.6, 127.4, 124.1, 120.9, 45.1. FTIR(KBr): 1655 cm\(^{-1}\) (CH\(_2\)CONR\(_1\)R\(_2\)); HRMS(ESI\(^+\)) calculated for C\(_{20}\)H\(_{17}\)NO[M+H]\(^+\): 310.1208; found: 310.1208.

N-(4-fluorophenyl)-2-phenylacetamide (3ar), this compound was prepared according to General Procedure A for 10 h, Yield: 95% (43.7 mg), brown solid, Mp: 133 - 135°C; \(^1\)H NMR (400 MHz, DMSO-d\(_6\)) \(\delta\) 10.22 (s, 1H), 7.63 (dd, J = 8.9, 5.0 Hz, 2H), 7.35 – 7.30 (m, 4H), 7.27 – 7.23 (m, 1H), 7.13 (t, J = 8.8 Hz, 2H), 3.64 (s, 2H). \(^{13}\)C NMR (100 MHz, DMSO-d\(_6\)) \(\delta\) 169.5, 158.44 (d, J\(_{C,F}\) = 239.7 Hz), 136.4, 136.1 (d, J\(_{C,F}\) = 2.6 Hz), 129.6, 128.8, 127.0, 121.4 (d, J\(_{C,F}\) = 7.8 Hz), 115.7 (d, J\(_{C,F}\) = 22.2 Hz), 43.7. HRMS(ESI\(^+\)) calculated for C\(_{14}\)H\(_{12}\)FNO[M+H]\(^+\): 230.0981; found: 230.0981.

N-(4-chlorophenyl)-2-phenylacetamide (3as), this compound was prepared according to General Procedure A for 14 h, Yield: 87% (42.8 mg), light yellow solid, Mp: 170 - 172°C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.37 (dd, J = 7.7, 5.4 Hz, 5H), 7.30 (d, J = 7.4 Hz, 3H), 7.21 (d, J = 8.7 Hz, 2H), 3.70 (s, 2H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 169.3, 136.2, 134.2, 129.5, 129.3, 128.9, 127.8, 126.4, 121.2, 44.7. HRMS(ESI\(^+\)) calculated for C\(_{14}\)H\(_{12}\)ClNO[M+H]\(^+\): 246.0686; found: 246.0684.
N-(4-cyanophenyl)-2-phenylacetamide (3at), this compound was prepared according to General Procedure A for 12 h, Yield: 95% (44.7 mg), yellow solid; $^1$H NMR (400 MHz, DMSO-d$_6$) $\delta$ 10.62 (s, 1H), 7.78 (q, $J = 8.5$ Hz, 4H), 7.34 (d, $J = 3.8$ Hz, 4H), 7.26 (d, $J = 3.4$ Hz, 1H), 3.71 (s, 2H). $^{13}$C NMR (100 MHz, DMSO-d$_6$) $\delta$ 170.1, 143.5, 135.5, 133.4, 129.3, 128.4, 126.8, 119.2, 119.1, 105.1, 43.4. HRMS(ESI$^+$) calculated for C$_{15}$H$_{12}$N$_2$O: $\text{[M+H]$^+$}: 237.1028; found: 237.1030.

![2-phenyl-N-(3-(trifluoromethyl)phenyl)acetamide (3au)](image)

2-phenyl-N-(3-(trifluoromethyl)phenyl)acetamide (3au), this compound was prepared according to General Procedure A for 8 h, Yield: 98% (54.7 mg), light yellow solid, Mp: 84 - 86°C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.76 (s, 1H), 7.65 (s, 1H), 7.52 (d, $J = 7.3$ Hz, 1H), 7.27 – 7.17 (m, 7H), 3.60 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.6, 138.2, 134.1, 131.6 (q, $J = 32.6$ Hz), 129.4, 129.3, 127.8, 123.8 (q, $J_{C-F} = 270.7$ Hz), 123.0, 121.0 (d, $J_{C-F} = 3.7$ Hz), 119.7, 116.6 (d, $J_{C-F} = 3.6$ Hz), 44.7. FTIR(KBr): 1639 cm$^{-1}$ (CH$_2$CONR$_1$R$_2$), HRMS(ESI$^+$) calculated for C$_{15}$H$_{12}$F$_3$NO: $\text{[M+H]$^+$}: 280.0949; found: 280.0948.

N-(2-hydroxyphenyl)-2-phenylacetamide (3av), this compound was prepared according to General Procedure A for 12 h, Yield: 78% (35.3 mg), yellow solid, Mp: 61 - 64°C; $^1$H NMR (400 MHz, DMSO-d$_6$) $\delta$ 9.79 (s, 1H), 9.34 (s, 1H), 7.78 (d, $J = 7.9$ Hz, 1H), 7.38 – 7.30 (m, 4H), 7.27 – 7.22 (m, 1H), 6.93 (t, $J = 7.6$ Hz, 1H), 6.86 (d, $J = 7.9$ Hz, 1H), 6.75 (t, $J = 7.6$ Hz, 1H), 3.75 (s, 2H). $^{13}$C NMR (100 MHz, DMSO-d$_6$) $\delta$ 169.5, 147.6, 136.1, 129.2, 128.3, 126.5, 126.3, 124.5, 121.9, 118.9, 115.5, 42.9. FTIR(KBr): 1639 cm$^{-1}$ (CH$_2$CONR$_1$R$_2$), HRMS(ESI$^+$) calculated for C$_{14}$H$_{13}$NO: $\text{[M+H]$^+$}: 250.0844; found: 250.0846.

N-(naphthalen-2-yl)-2-phenylacetamide (3aw), this compound was prepared according to General Procedure A for 14 h, Yield: 76% (39.6 mg), brown solid, Mp: 157 - 160°C; $^1$H NMR (400 MHz, DMSO-d$_6$) $\delta$ 10.39 (s, 1H), 8.32 (s, 1H), 7.85 (d, $J = 4.7$ Hz, 1H), 7.82 (d, $J = 3.0$ Hz, 1H), 7.62 (dd, $J = 8.8$, 1.6 Hz, 1H), 7.45 (t, $J = 7.2$ Hz, 1H), 7.42 (d, $J = 7.8$ Hz, 3H), 7.37 (s, 1H), 7.33 (d, $J = 7.6$ Hz, 2H), 7.26 (d, $J = 7.2$ Hz, 1H), 3.72 (s, 2H). $^{13}$C NMR (100 MHz, DMSO-d$_6$) $\delta$ 169.5, 136.9, 136.1, 133.5, 129.8, 129.2, 128.4, 128.4, 127.5, 127.3, 126.6, 126.5, 124.6, 120.0, 115.3, 43.5. HRMS (ESI$^+$) calculated for C$_{18}$H$_{15}$NO: $\text{[M+H]$^+$}: 284.1051; found: 284.1050.
2-phenyl-N-(pyridin-2-yl)acetamide (3ax), this compound was prepared according to General Procedure A for 12 h, Yield: 89% (37.6 mg), brown solid. Mp: 123 - 124°C; ¹H NMR (400 MHz, CDCl₃) δ 8.47 (s, 1H), 8.18 – 8.06 (m, 2H), 7.59 (t, J = 7.8 Hz, 1H), 7.29 – 7.23 (m, 2H), 7.21 (d, J = 7.0 Hz, 3H), 6.95 – 6.87 (m, 1H), 3.65 (s, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 169.7, 151.4, 147.6, 138.4, 134.1, 129.4, 129.1, 127.6, 119.9, 114.2, 44.8. FTIR(KBr): 1639 cm⁻¹ (CH₂CONR₁R₂), 1640 cm⁻¹ (CH₂CONR₁R₂), HRMS(ESI⁺) calculated for C₁₃H₁₂N₂O [M+H]⁺: 213.1028; found: 213.1024.

N-butyl-2-phenylacetamide (3ay), this compound was prepared according to General Procedure A for 12 h, Yield: 79% (30.1 mg), brown solid. Mp: 53 - 55°C; ¹H NMR (400 MHz, CDCl₃) δ 7.35 (t, J = 7.2 Hz, 2H), 7.30 (d, J = 7.1 Hz, 1H), 7.26 (dd, J = 5.4, 4.4 Hz, 2H), 5.45 (s, 1H), 3.56 (s, 2H), 3.20 (dd, J = 13.1, 6.9 Hz, 2H), 1.40 (dt, J = 14.8, 7.2 Hz, 2H), 1.25 (dd, J = 15.0, 7.4 Hz, 2H), 0.87 (t, J = 7.3 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 170.9, 135.1, 129.5, 129.0, 127.3, 43.9, 39.4, 31.5, 20.0, 13.7. HRMS (ESI⁺) calculated for C₁₂H₁₇NO [M+H]⁺: 192.1388; found: 192.1391.

N-benzyl-2-phenylacetamide (3az), this compound was prepared according to General Procedure A for 12 h, Yield: 72% (32.4 mg), yellow solid. Mp: 123 - 125°C; ¹H NMR (400 MHz, CDCl₃) δ 7.36 – 7.30 (m, 2H), 7.26 (t, J = 7.0 Hz, 6H), 7.16 (d, J = 7.2 Hz, 2H), 5.87 (s, 1H), 4.39 (d, J = 5.7 Hz, 2H), 3.60 (s, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 170.9, 138.2, 134.8, 129.4, 129.0, 128.6, 127.5, 127.4, 43.8, 43.6. HRMS (ESI⁺) calculated for C₁₅H₁₅NO [M+H]⁺: 226.1232; found: 226.1229.

N-phenethyl-2-phenylacetamide (3ba), this compound was prepared according to General Procedure A for 10 h, Yield: 81% (38.6 mg), yellow solid. Mp: 92 - 95°C; ¹H NMR (400 MHz, CDCl₃) δ 7.32 (d, J = 7.3 Hz, 2H), 7.26 (dd, J = 14.0, 6.8 Hz, 3H), 7.16 (d, J = 6.9 Hz, 3H), 7.02 (d, J = 7.0 Hz, 2H), 5.43 (s, 1H), 3.52 (s, 2H), 3.45 (dd, J = 12.9, 6.5 Hz, 2H), 2.71 (t, J = 6.8 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 170.9, 138.7, 134.8, 129.5, 129.0, 128.7, 128.6, 127.3, 126.4, 43.9,
40.7, 35.5. FTIR(KBr): 1639 cm⁻¹ (CH₂CONR₁R₂), 1640 cm⁻¹ (CH₂CONR₁R₂), HRMS (ESI⁺) calculated for C₁₆H₁₇NO [M+Na]⁺: 262.1208; found: 262.1210.

2-phenyl-N-(1-phenylethyl)acetamide (3bb), this compound was prepared according to General Procedure A for 10 h. Yield: 91% (43.4 mg), white solid, Mp: 115 - 117°C; ¹H NMR (400 MHz, CDCl₃) δ 7.34 (t, J = 7.1 Hz, 2H), 7.31 – 7.22 (m, 6H), 7.18 (d, J = 7.0 Hz, 2H), 5.16 – 5.06 (m, 1H), 3.56 (s, 2H), 1.39 (d, J = 6.9 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 170.0, 143.1, 135.0, 129.4, 129.0, 128.6, 127.3, 127.3, 126.0, 48.8, 43.9, 21.8. FTIR(KBr): 1641 cm⁻¹ (CH₂CONR₁R₂), HRMS(ESI⁺) calculated for C₁₆H₁₇NO [M+Na]⁺: 262.1208; found: 262.1206.

N-cyclopentyl-2-phenylacetamide (3bc), this compound was prepared according to General Procedure A for 16 h. Yield: 63% (25.8 mg), yellow solid, Mp: 116 - 119°C; ¹H NMR (400 MHz, CDCl₃) δ 7.35 (t, J = 7.2 Hz, 2H), 7.29 (d, J = 7.1 Hz, 1H), 7.24 (d, J = 7.2 Hz, 2H), 5.40 (s, 1H), 4.18 (dd, J = 13.9, 7.0 Hz, 1H), 3.54 (s, 2H), 1.92 (dd, J = 12.1, 6.1 Hz, 2H), 1.55 (s, 4H), 1.28 – 1.22 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 170.5, 135.2, 129.4, 129.0, 127.2, 51.3, 43.9, 33.0, 23.6. FTIR(KBr): 1641 cm⁻¹ (CH₂CONR₁R₂), HRMS(ESI⁺) calculated for C₁₃H₁₇NO [M+Na]⁺: 226.1208; found: 226.1207.

N-cyclohexyl-2-phenylacetamide (3bd), this compound was prepared according to General Procedure A for 12 h. Yield: 85% (36.9 mg), light yellow solid, Mp: 131 - 133°C; ¹H NMR (400 MHz, CDCl₃) δ 7.34 (t, J = 7.3 Hz, 2H), 7.28 (d, J = 7.8 Hz, 1H), 7.25 (d, J = 7.1 Hz, 2H), 5.40 (s, 1H), 3.80 – 3.69 (m, 1H), 3.53 (s, 2H), 1.84 – 1.81 (m, 2H), 1.66 – 1.54 (m, 2H), 1.37 – 1.21 (m, 2H), 1.06 – 0.98 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 170.0, 135.2, 129.3, 128.9, 127.2, 51.3, 43.9, 33.0, 23.6. HRMS(ESI⁺) calculated for C₁₄H₁₉NO [M+Na]⁺: 240.1364; found: 240.1367.

N,N-diethyl-2-phenylacetamide (3be), this compound was prepared according to General Procedure A for 12 h. Yield: 55% (21.2 mg), white solid, Mp: 57-59°C; ¹H NMR (400 MHz, CDCl₃) δ 7.34 (t, J = 7.3 Hz, 2H), 7.29 (d, J = 7.8 Hz, 1H), 7.25 (d, J = 7.1 Hz, 2H), 5.40 (s, 1H), 3.80 – 3.69 (m, 1H), 3.53 (s, 2H), 1.84 – 1.81 (m, 2H), 1.66 – 1.54 (m, 2H), 1.37 – 1.21 (m, 2H), 1.06 – 0.98 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 170.0, 135.2, 129.3, 128.9, 127.2, 51.3, 43.9, 33.0, 23.6. HRMS(ESI⁺) calculated for C₁₄H₁₉NO [M+Na]⁺: 240.1364; found: 240.1367.
CDCl$_3$ δ 7.36 – 7.28 (m, 2H), 7.28 – 7.23 (m, 3H), 3.70 (s, 2H), 3.39 (q, $J = 7.1$ Hz, 2H), 3.30 (q, $J = 7.1$ Hz, 2H), 1.15 – 1.06 (m, 6H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 170.2, 135.6, 128.6, 128.6, 126.7, 42.4, 40.9, 40.2, 14.2, 13.0. HRMS(ESI$^+$) calculated for C$_{12}$H$_{17}$NO [M+Na$^+$]: 214.1208; found: 214.1206.

2-phenyl-1-thiomorpholinoethan-1-one (3bf), this compound was prepared according to General Procedure A for 12 h, Yield: 55% (24.4 mg), yellow solid. Mp: 83 - 85°C; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.33 (t, $J = 7.3$ Hz, 2H), 7.24 (d, $J = 7.6$ Hz, 3H), 3.92 – 3.87 (m, 2H), 3.73 (s, 2H), 3.71 – 3.68 (m, 2H), 2.60 – 2.55 (m, 2H), 2.33 – 2.28 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 169.5, 134.8, 128.9, 128.5, 127.0, 48.8, 44.4, 41.4, 27.5, 27.3. FTIR(KBr): 1649 cm$^{-1}$ (CH$_2$CONR$_1$R$_2$), HRMS(ESI$^+$) calculated for C$_{12}$H$_{15}$NOS [M+Na$^+$]: 244.0772; found: 244.0769.

1-((2S,6R)-2,6-dimethylmorpholino)-2-phenylethan-1-one (3bg), this compound was prepared according to General Procedure A for 12 h, Yield: 79% (37.0 mg), yellow liquid; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.32 (t, $J = 7.2$ Hz, 2H), 7.24 (t, $J = 6.9$ Hz, 3H), 4.48 (d, $J = 13.2$ Hz, 1H), 3.73 (s, 2H), 3.63 (d, $J = 13.1$ Hz, 1H), 3.52 – 3.42 (m, 1H), 3.27 – 3.17 (m, 1H), 2.76 – 2.66 (m, 1H), 2.37 – 2.26 (m, 1H), 1.17 (d, $J = 6.2$ Hz, 3H), 1.07 (d, $J = 6.1$ Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 169.32 (s), 134.9, 128.8, 128.5, 126.9, 71.9, 71.6, 51.7, 47.2, 41.1, 18.7, 18.6. FTIR(thin film): 1644 cm$^{-1}$ (CH$_2$CONR$_1$R$_2$), HRMS(ESI$^+$) calculated for C$_{14}$H$_{19}$NO$_2$ [M+Na$^+$]: 256.1313; found: 256.1317.

(S)-Methyl 2-(2-phenylacetamido)propanoate (3bh), this compound was prepared according to General Procedure A for 12 h ($\alpha$-amino-esters was in the hydrochloride form ), Yield: 83% (36.7 mg), yellow solid. Mp: 63 - 65°C; $^1$H NMR (400 MHz, CDCl$_3$) 7.36 (t, $J = 7.0$ Hz, 2H), 7.29 (t, $J = 8.1$ Hz, 3H), 6.07 (s, 1H), 4.58 (m, $J = 7.2$ Hz, 1H), 3.71 (s, 3H), 3.59 (s, 2H), 1.34 (d, $J = 7.1$ Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 173.4, 170.5, 134.6, 129.4, 129.0, 127.4, 52.4, 48.1, 43.6, 18.3.
FTIR(KBr): 1613 cm\(^{-1}\) (CH\(_2\)CONR\(_1\)R\(_2\)), HRMS(ESI\(^+\)) calculated for C\(_{12}\)H\(_{15}\)NO\(_3\) [M+Na\(^+\)]: 244.0950; found: 244.0952. HPLC (Chiral IC, n-hexane:isopropanol=75:25, flow rate = 1.0 mL/min), t\(R\)= 11.893, 13.737 min, ee = 90%.

(Rac-HPLC spectrum)
(S)-Ethyl 3-methyl-2-(2-phenylacetamido)butanoate (3bi), this compound was prepared according to General Procedure A for 12 h (α-amino-esters was in the hydrochloride form, equal dppf instead of PPh₃), Yield: 42% (22.3 mg), yellow solid. Mp: 58 - 60°C; ¹H NMR (400 MHz, CDCl₃) δ 7.36 (t, J = 9.7, 2H), 7.30 (t, J = 6.6 Hz, 3H), 5.89 (d, J = 7.9 Hz, 1H), 4.52 (dd, J = 8.8, 4.8 Hz, 1H), 4.21 – 4.09 (m, 2H), 3.62 (s, 2H), 2.16 – 2.05 (m, 1H), 1.24 (t, J = 7.1 Hz, 3H), 0.86 (d, J = 6.9 Hz, 3H), 0.77 (d, J = 6.9 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 171.8, 170.8, 134.8, 129.4, 129.1, 127.5, 61.2, 57.0, 43.8, 31.3, 18.9, 17.6, 14.2. FTIR(KBr): 1640 cm⁻¹ (CH₂CONR₁R₂), HRMS(ESI⁺) calculated for C₁₂H₁₅NO₃ [M+Na]⁺: 264.1600; found: 264.1601. HPLC (Chiral IC, n-hexane:isopropanol=75:25, flow rate = 1.0 mL/min), tR= 9.820, 10.957 min, ee = 100%.
(Rac-HPLC spectrum)

(3ak HPLC spectrum)
(S)-Ethyl 3-phenyl-2-(2-phenylacetamido)propanoate (3bj), this compound was prepared according to General Procedure A for 16 h (α-amino-esters was in the hydrochloride form, equal dppf instead of PPh₃), Yield: 39% (22.9 mg), brown oil;¹H NMR (400 MHz, CDCl₃) δ 7.36 – 7.28 (m, 3H), 7.21 – 7.16 (m, 5H), 6.91 (dd, J = 6.5, 3.0 Hz, 2H), 5.84 (d, J = 7.3 Hz, 1H), 4.87 – 4.79(m, 1H), 4.13 (q, J = 7.1 Hz, 2H), 3.55 (s, 2H), 3.10 – 2.97 (m, 2H), 1.21 (t, J = 7.2 Hz, 3H).¹³C NMR (100 MHz, CDCl₃) δ 171.3, 170.4, 135.7, 134.5, 129.4, 129.2, 129.0, 128.5, 127.4, 127.0, 61.5, 53.0, 43.7, 37.7, 14.1. FTIR(KBr): 1655 cm⁻¹ (CH₂CONR₁R₂), HRMS(ESI⁺) calculated for C₁₂H₁₅NO₃ [M+H⁺]: 312.1600; found: 312.1594. HPLC (Chiral IC, n-hexane:isopropanol=75:25, flow rate = 1.0 mL/min), tR= 12.820, 17.343 min, ee = 94%.

(Rac-HPLC spectrum)
Part 6. Characterization of carbonylation products of esters (reaction with alcohols) (5aa—5ay)

Benzyl 2-phenylacetate (5aa), this compound was prepared according to General Procedure B for 12 h, Yield: 82% (36.7 mg), light yellow liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.46 – 7.06 (m, 10H), 5.12 (s, 2H), 3.66 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 171.4, 135.9, 133.9, 129.3, 128.6, 128.2, 128.1, 127.2, 66.6, 41.4. HRMS(ESI$^+$) calculated for C$_{15}$H$_{14}$O$_2$ [M+Na$^+$] 249.0891, found 249.0890.

Benzyl 2-(p-tolyl)acetate (5ab), this compound was prepared according to General Procedure B for 10 h, Yield: 86% (41.2 mg), light green liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.36 – 7.26 (m, 5H), 7.14 (m, 4H), 5.11 (s, 2H), 3.62 (s, 2H), 2.32 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 171.7, 136.8,
Benzyl 2-(o-tolyl)acetate (5ac), Yield: 86% (41.1 mg), this compound was prepared according to General Procedure B for 10 h, light green liquid; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.37 – 7.26 (m, 5H), 7.24 – 7.12 (m, 4H), 5.14 (s, 2H), 3.67 (s, 2H), 2.28 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 171.3, 136.9, 136.0, 132.7, 130.4, 130.2, 128.5, 128.2, 128.1, 127.5, 126.2, 66.6, 39.2, 19.6. FTIR(thin film): 1739 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{16}$H$_{16}$O$_2$ [M+H$^+$] 241.1229, found 241.1223.

![Benzyl 2-(o-tolyl)acetate (5ac)](image)

Benzyl 2-(4-fluorophenyl)acetate (5ad), this compound was prepared according to General Procedure B for 12 h, Yield: 82% (39.9 mg), light green liquid; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.37 – 7.28 (m, 5H), 7.36 – 7.21 (m, 2H), 6.99 (t, $J = 8.7$ Hz, 2H), 5.13 (s, 2H), 3.63 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 171.3, 162.1 (d, $J_{C-F} = 245.4$ Hz), 135.8, 130.9 (d, $J_{C-F} = 8.0$ Hz), 129.7 (d, $J_{C-F} = 3.3$ Hz), 128.6, 128.3, 128.2, 115.4 (d, $J_{C-F} = 21.5$ Hz), 66.8, 40.5. FTIR(thin film): 1731 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{15}$H$_{13}$FO$_2$ [M +H$^+$] 245.0978, found 245.0972.

![Benzyl 2-(4-fluorophenyl)acetate (5ad)](image)

Benzyl 2-(3,4-difluorophenyl)acetate (5ae), this compound was prepared according to General Procedure B for 12 h, Yield: 81% (47.7 mg), light yellow liquid; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.37 – 7.29 (m, 5H), 7.14 – 7.04 (m, 2H), 7.00 – 6.95 (m, 1H), 5.13 (s, 2H), 3.61 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 170.7, 151.18 (dd, $J_{C-F} = 51.6$, 12.7 Hz), 148.71 (dd, $J_{C-F} = 50.9$, 12.7 Hz), 135.6, 130.7 (dd, $J_{C-F} = 50.9$, 6.1 Hz), 128.6, 128.4, 128.3, 125.4 (dd, $J_{C-F} = 6.2$, 3.7 Hz), 118.4 (d, $J_{C-F} = 17.7$ Hz), 117.3 (d, $J_{C-F} = 17.4$ Hz), 66.9, 40.3 (d, $J_{C-F} = 1.2$ Hz). FTIR(thin film): 1738 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{15}$H$_{12}$F$_2$O$_2$ [M+Na$^+$] 285.0703, found 285.0695.

![Benzyl 2-(3,4-difluorophenyl)acetate (5ae)](image)
Benzyl 2-(4-(trifluoromethyl)phenyl)acetate (5af), this compound was prepared according to General Procedure B for 10 h, Yield: 80% (47.1 mg), light yellow liquid; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.57 (d, $J$ = 8.1 Hz, 2H), 7.39 (d, $J$ = 8.1 Hz, 2H), 7.36 – 7.27 (m, 5H), 5.14 (s, 2H), 3.72 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 171.2, 139.9 (d, $J_{C,F}$ = 4.8 Hz), 133.7, 130.4 (q, $J_{C,F}$ = 32.5 Hz), 129.3, 128.7, 128.0, 127.3, 125.5 (q, $J_{C,F}$ = 3.8 Hz), 124.1 (q, $J_{C,F}$ = 272.1 Hz), 65.6, 41.4. FTIR(thin film): 1731 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{16}$H$_{13}$F$_3$O$_2$ [M+H$^+$] 295.0946, found 295.0940.

Benzyl 2-(4-methoxyphenyl)acetate (5ag), this compound was prepared according to General Procedure B for 14 h, Yield: 62% (31.8 mg), colorless liquid; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.37 – 7.28 (m, 5H), 7.20 (d, $J$ = 8.7 Hz, 2H), 6.85(d, $J$ = 8.8 Hz,2H), 5.12 (s, 2H), 3.79 (s, 3H), 3.60 (s,2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 171.8, 158.8, 136.0, 130.4, 128.6, 128.2, 126.0, 114.1, 66.6, 55.3, 40.5. FTIR(thin film): 1735 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{16}$H$_{16}$O$_3$ [M+H$^+$]257.1178, found 241.1172.

Methyl 4-(2-(benzyloxy)-2-oxoethyl)benzoate (5ah), this compound was prepared according to General Procedure B for 12 h, Yield: 66% (37.1 mg), colorless liquid; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.99(d, $J$ = 8.4 Hz,2H), 7.41 – 7.28 (m, 7H), 5.14 (s, 2H), 3.90 (s, 3H), 3.72 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 170.7, 166.9, 139.0, 135.7, 129.9, 129.4, 129.1, 128.6, 128.4, 128.2, 126.0, 66.9, 52.1, 41.3. FTIR(thin film): 1740 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{17}$H$_{16}$O$_4$ [M+H$^+$] 285.1127, found 285.1131.

Benzyl-4-(2-(benzyloxy)-2-oxoethyl)benzoate (5ai), this compound was prepared according to General Procedure B for 16 h, Yield: 40%/45%, (27.5 mg/ 30.8), from 4-chloro/bromo benzyl ammonium salts, yellow liquid; $^1$H NMR (400 MHz, CDCl$_3$) δ 8.03 (d, $J$ = 8.3 Hz, 2H), 7.49 – 7.18 (m, 12H), 5.35 (s, 2H), 5.13 (s, 2H), 3.71 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 170.6, 166.2, 139.2, 136.1, 135.7, 130.0, 129.4, 129.1, 128.6, 128.4, 128.3, 128.2, 126.0, 66.9, 66.7, 41.3.
FTIR (thin film): 1731 cm\(^{-1}\) (CH\(_2\)COOR); HRMS (ESI\(^+\)) calculated for C\(_{23}\)H\(_{20}\)O\(_4\) [M+Na\(^+\)] 383.1259, found 383.1261.

**Benzyl 2-(naphthalen-2-yl)acetate (5aj),** this compound was prepared according to General Procedure B for 16 h, Yield: 47% (25.9 mg), light yellow liquid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.83 – 7.76 (m, m, 3H), 7.72 (s, 1H), 7.47 – 7.39 (m, 3H), 7.36 – 7.29 (m, 5H), 5.15 (s, 2H), 3.83 (s, 2H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 171.4, 135.9, 133.5, 132.6, 131.4, 128.6, 128.3, 128.2, 128.1, 127.7, 127.4, 126.2, 125.9, 66.8, 41.6. FTIR (thin film): 1731 cm\(^{-1}\) (CH\(_2\)COOR); HRMS (ESI\(^+\)) calculated for C\(_{19}\)H\(_{16}\)O\(_2\) [M+H\(^+\)] 277.1229, found 277.1236.

**4-methoxybenzyl 2-phenylacetate (5ak),** this compound was prepared according to General Procedure B for 12 h, Yield: 57% (29.2 mg), yellow liquid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.35 – 7.22 (m, 7H), 6.87 (d, \(J = 8.5\) Hz, 2H), 5.06 (s, 2H), 3.79 (s, 3H), 3.62 (s, 2H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 171.5, 159.7, 134.0, 130.0, 129.3, 128.6, 128.0, 127.1, 114.0, 66.5, 55.3, 41.4. FTIR (thin film): 1740 cm\(^{-1}\) (CH\(_2\)COOR); HRMS (ESI\(^+\)) calculated for C\(_{16}\)H\(_{16}\)O\(_2\) [M+H\(^+\)] 257.1178, found 257.1180.

**4-(trifluoromethyl)benzyl 2-phenylacetate (5al),** this compound was prepared according to General Procedure B for 10 h, Yield: 79% (46.7 mg), yellow liquid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.58 (d, \(J = 8.1\) Hz, 2H), 7.38 (d, \(J = 8.0\) Hz, 2H), 7.36 – 7.24 (m, 5H), 5.17 (s, 2H), 3.69 (s, 2H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 171.2, 139.9 (d, \(J_{C-F} = 1.2\) Hz), 133.7, 130.4 (d, \(J_{C-F} = 32.5\) Hz), 129.3, 128.7, 128.0, 127.3, 125.5 (q, \(J_{C-F} = 3.8\) Hz), 124.1 (d, \(J_{C-F} = 272.1\) Hz), 65.6, 41.4. FTIR (thin film): 1729 cm\(^{-1}\) (CH\(_2\)COOR); HRMS (ESI\(^+\)) calculated for C\(_{16}\)H\(_{13}\)F\(_3\)O\(_2\) [M+H\(^+\)] 295.0946, found 295.0950.

**Ethyl 2-phenylacetate (5am),** this compound was prepared according to General Procedure B for 16 h, Yield: 48% (15.8 mg), 20 mol% xantphos extra added. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.53 – 6.97
Butyl 2-phenylacetate (5an), this compound was prepared according to General Procedure B for 12 h, Yield: 54% (21.1 mg), yellow liquid; \(^1^H\) NMR (400 MHz, CDCl\(_3\)) \(\delta \) 7.34 – 7.25 (m, 5H), 4.09 (t, \(J = 6.7\) Hz, 2H), 3.61 (s, 2H), 1.62 – 1.54 (m, 4H), 1.38 – 1.31 (m, 2H), 0.91 (t, \(J = 7.4\) Hz, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta \) 171.8, 134.3, 129.3, 128.6, 127.1, 64.6, 44.8, 41.5, 32.1, 27.9, 23.3. FTIR (thin film): 1736 cm\(^{-1}\) (CH\(_2\)COOR); HRMS (ESI\(^+\)) calculated for C\(_{12}\)H\(_{16}\)O\(_2\) [M+H\(^+\)] 215.1094, found 215.1090.

5-chloropentyl 2-phenylacetate (5ao), this compound was prepared according to General Procedure B for 10 h, Yield: 61% (26.3 mg), yellow liquid; \(^1^H\) NMR (400 MHz, CDCl\(_3\)) \(\delta \) 7.35 – 7.25 (m, 5H), 4.10 (t, \(J = 6.5\) Hz, 2H), 3.61 (s, 2H), 3.49 (t, \(J = 6.6\) Hz, 2H), 1.80 – 1.71 (m, 2H), 1.69 – 1.61 (m, 2H), 1.51 – 1.42 (m, 2H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta \) 171.6, 134.1, 129.3, 128.6, 127.1, 64.6, 44.8, 41.5, 32.1, 27.9, 23.3. FTIR (thin film): 1736 cm\(^{-1}\) (CH\(_2\)COOR); HRMS (ESI\(^+\)) calculated for C\(_{13}\)H\(_{17}\)ClO\(_2\) [M+H\(^+\)] 241.0995, found 241.0990.

2-methoxyethyl 2-phenylacetate (5ap), this compound was prepared according to General Procedure B for 10 h, Yield: 59% (22.9 mg), yellow liquid; \(^1^H\) NMR (400 MHz, CDCl\(_3\)) \(\delta \) 7.35 – 7.24 (m, 5H), 4.25 (t, \(J = 5.4\) Hz, 2H), 3.66 (s, 2H), 3.58 (t, \(J = 4.1\) Hz, 2H), 3.36 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta \) 171.6, 134.0, 129.3, 128.6, 127.1, 70.4, 63.9, 59.0, 41.2. FTIR (thin film): 1737 cm\(^{-1}\) (CH\(_2\)COOR); HRMS (ESI\(^+\)) calculated for C\(_{11}\)H\(_{14}\)O\(_3\) [M+Na\(^+\)] 263.0841, found 263.0835.

Pyridin-3-ylmethyl 2-phenylacetate (5aq), this compound was prepared according to General Procedure B for 12 h, Yield: 65% (29.5 mg), yellow liquid; \(^1^H\) NMR (400 MHz, CDCl\(_3\)) \(\delta \) 8.60 – 8.53 (m, 2H), 7.68 – 7.59 (m, 1H), 7.37 – 7.22 (m, 6H), 5.14 (s, 2H), 3.67 (s, 2H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta \) 171.6, 134.0, 129.3, 128.6, 127.1, 70.4, 63.9, 59.0, 41.2. FTIR (thin film): 1737 cm\(^{-1}\) (CH\(_2\)COOR); HRMS (ESI\(^+\)) calculated for C\(_{11}\)H\(_{15}\)O\(_3\) [M+Na\(^+\)] 263.0841, found 263.0835.
Furan-3-ylmethyl 2-phenylacetate (5ar), this compound was prepared according to General Procedure B for 12 h, Yield: 54% (24.1 mg), yellow liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.42 (d, $J$ = 0.6 Hz, 1H), 7.37 (d, $J$ = 1.5 Hz, 1H), 7.34 –7.25 (m, 5H), 6.39 (s, 1H), 5.00 (s, 2H), 3.63 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 171.4, 143.4, 141.6, 133.9, 129.3, 128.6, 127.2, 120.4, 110.5, 58.2, 41.3. FTIR(thin film): 1738 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{14}$H$_{13}$NO$_2$ [M+H$^+$] 228.1025, found 228.1019.

Thiophen-3-ylmethyl 2-phenylacetate (5as), this compound was prepared according to General Procedure B for 12 h, Yield: 63% (29.2 mg), yellow liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.35 – 7.25 (m, 6H), 7.23 (s, 2H), 7.03 (d, $J$ = 4.9 Hz, 1H), 5.12 (s, 2H), 3.64 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 171.4, 136.7, 133.9, 129.3, 128.6, 127.5, 127.2, 126.2, 124.2, 61.7, 41.3. FTIR(thin film): 1736 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{13}$H$_{12}$O$_2$S$[\text{M+Na}^+]$ 255.0456, found 255.0450.

Benzo[d][1,3]dioxol-5-ylmethyl 2-phenylacetate (5at), this compound was prepared according to General Procedure B for 10 h, Yield: 74% (37.1 mg), yellow solid; Mp: 112-114 °C, $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.34 – 7.24 (m, 5H), 6.81– 6.74 (m, 3H), 5.94 (s, 2H), 5.02 (s, 2H), 3.64 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 171.4, 147.9, 147.7, 134.0, 129.7, 129.3, 128.6, 127.2, 122.2, 109.0, 108.3, 101.2, 66.6, 41.4. FTIR(KBr): 1736 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{16}$H$_{14}$O$_3$ [M+Na$^+$] 277.0841, found 277.0838.

Pent-4-en-1-yl 2-phenylacetate (5au), this compound was prepared according to General Procedure B for 10 h, Yield: 80% (32.6 mg), yellow liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.36 – 7.25 (m, 5H),
5.83 – 5.70 (m, 1H), 5.03 – 4.93 (m, 2H), 4.10 (t, J = 9.0 Hz, 2H), 3.61 (s,2H), 2.07 (dd, J = 14.3, 7.3 Hz, 2H), 1.76 – 1.57 (m, 3H). ^13^C NMR (100 MHz, CDCl$_3$) δ 171.6, 137.5, 134.2, 129.3, 128.6, 127.1, 115.3, 64.3, 41.5, 30.0, 27.9. FTIR(thin film): 1761 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{13}$H$_{16}$O$_2$ [M+Na$^+$] 227.1048, found 227.1043.

**Cyclobutyl 2-phenylacetate (5av),** this compound was prepared according to General Procedure B for 10 h, Yield: 77% (29.1 mg), colorless liquid; ^1^H NMR (400 MHz, CDCl$_3$) δ 7.42 – 7.20 (m, 5H), 5.07 – 4.83 (m, 1H), 3.58 (s, 2H), 2.44 – 2.26 (m, 2H), 2.09 – 1.58 (m, 2H). ^13^C NMR (100 MHz, CDCl$_3$) δ 171.0, 134.2, 129.3, 128.6, 127.0, 69.3, 41.8, 32.7, 23.7. FTIR(thin film): 1736 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{12}$H$_{14}$O$_2$ [M+Na$^+$] 213.0891, found 213.0886.

**Cyclopentyl 2-phenylacetate (5aw),** this compound was prepared according to General Procedure B for 12 h, Yield: 61% (24.8 mg), colorless liquid; ^1^H NMR (400 MHz, CDCl$_3$) δ 7.37 – 7.18 (m, 5H), 5.24 – 5.13 (m, 1H), 3.57 (s, 2H), 1.89 – 1.78 (m, 2H), 1.72 – 1.62 (m, 4H), 1.60 – 1.54 (m, 2H). ^13^C NMR (100 MHz, CDCl$_3$) δ 171.4, 134.4, 129.2, 128.5, 127.0, 77.6, 41.8, 32.7, 23.7. FTIR(thin film): 1734 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{13}$H$_{16}$O$_2$ [M+Na$^+$] 227.1048, found 227.1043.

**Cyclohexyl 2-phenylacetate (5ax),** this compound was prepared according to General Procedure B for 12 h, Yield: 66% (28.6 mg), colorless liquid; ^1^H NMR (400 MHz, CDCl$_3$) δ 7.36 – 7.21 (m, 5H), 4.81 – 4.73 (m, 1H), 3.59 (s, 2H), 1.86 – 1.77 (m, 2H), 1.74 – 1.64 (m, 2H), 1.55 – 1.25 (m, 6H). ^13^C NMR (100 MHz, CDCl$_3$) δ 171.1, 134.5, 129.2, 128.5, 127.0, 73.1, 41.9, 31.6, 25.4, 23.7. FTIR(thin film): 1732 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{14}$H$_{18}$O$_2$ [M+H$^+$] 219.1385, found 219.1385.

**1-methylcyclohexyl 2-phenylacetate (5ay),** this compound was prepared according to General Procedure B for 16 h, Yield: 20% (9.4 mg), colorless liquid; ^1^H NMR (400 MHz, CDCl$_3$) δ 7.33 – 7.31 (m, 3H), 7.29 (d, J = 2.6 Hz, 2H), 3.58 (s, 2H), 1.47 (s, 3H), 1.46 – 1.27 (m, 10H). ^13^C NMR (100 MHz, CDCl$_3$) δ 170.8, 134.9, 129.4, 128.5, 126.9, 82.4, 43.0, 36.6, 25.6, 25.4, 22.0. FTIR(thin film): 1736 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{15}$H$_{20}$O$_2$ [M+Na$^+$] 241.1295, found 241.1295.
Part 7. Characterization of carbonylation products of esters (reaction with phenols) (7aa—7ay)

Phenyl 2-phenylacetate (7aa)[1722-01-9], this compound was prepared according to General Procedure C for 8 h, Yield: 81% (34.3 mg), colorless liquid; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.41 – 7.28 (m, 7H), 7.20 (t, $J = 7.4$ Hz, 1H), 7.05 (d, $J = 7.8$ Hz, 2H), 3.86 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 170.0, 150.8, 133.5, 129.4, 129.3, 128.8, 127.4, 125.9, 121.5, 41.5. HRMS(ESI$^+$) calculated for C$_{14}$H$_{12}$O$_2$ [M+H$^+$] 213.0916, found 213.0910.
Phenyl 2-(p-tolyl)acetate (7ab), this compound was prepared according to General Procedure C for 8 h, Yield: 88% (40.2 mg), colorless liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.34 (dd, $J$ = 10.7, 5.1 Hz, 2H), 7.26 (d, $J$ = 7.9 Hz, 2H), 7.23 – 7.14 (m, 3H), 7.08 – 7.01 (m, 2H), 3.80 (s, 2H), 2.34 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.2, 150.8, 137.0, 130.4, 129.4, 129.2, 125.8, 121.5, 41.1. FTIR (thin film): 1746 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{15}$H$_{14}$O$_2$ [M+Na$^+$] 249.0891, found 249.0886.

Phenyl 2-(m-tolyl)acetate (7ac), this compound was prepared according to General Procedure C for 10 h, Yield: 70% (31.6 mg), colorless liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.38 – 7.27 (m, 3H), 7.25 – 7.17 (m, 4H), 7.05 (m, d, $J$ = 8.8 Hz, 2H), 3.87 (s, 2H), 2.40 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.9, 150.8, 136.9, 132.3, 130.5, 130.3, 129.4, 127.7, 126.3, 125.9, 121.5, 39.4 19.7. FTIR (thin film): 1754 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{15}$H$_{14}$O$_2$ [M+Na$^+$] 249.0895, found 249.0895.

Phenyl 2-(4-methoxyphenyl)acetate (7ad), this compound was prepared according to General Procedure C for 8 h, Yield: 85% (41.2 mg), colorless liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.35 (t, $J$ = 7.6 Hz, 2H), 7.30 (d, $J$ = 8.7 Hz, 2H), 7.22 (t, $J$ = 7.6 Hz, 1H), 7.05 (d, $J$ = 8.8 Hz, 2H), 6.90 (d, $J$ = 6.8 Hz, 2H), 3.80 (s, 3H), 3.79 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.3, 158.9, 150.8, 130.4, 129.4, 125.8, 125.5, 121.5, 114.2, 55.3, 40.6. FTIR (thin film): 1754 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{15}$H$_{14}$O$_3$ [M+H$^+$] 243.1021, found 243.1016.

Phenyl 2-(4-fluorophenyl)acetate (7ae), this compound was prepared according to General Procedure C for 8 h, Yield: 85% (39.1 mg), colorless liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.39 – 7.32 (m, 4H), 7.22 (dd, $J_{CF} = 12.7$, 5.3 Hz, 1H), 7.08 – 7.02 (m, 4H), 3.82 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.9, 162.2 (d, $J_{C-F}$ = 245.8 Hz), 150.7, 131.0 (d, $J_{C-F} = 8.1$ Hz), 129.5, 129.2 (d, $J_{C-F}$ = 3.2 Hz), 126.0, 121.4, 115.6 (d, $J_{C-F} = 21.5$ Hz), 40.6. FTIR (thin film): 1755 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{14}$H$_{11}$FO$_2$ [M+Na$^+$] 253.0641, found 253.0635.
Phenyl 2-(4-chlorophenyl)acetate (7af), this compound was prepared according to General Procedure C for 10 h, Yield: 80% (39.2mg), colorless liquid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.40 – 7.29 (m, 6H), 7.22 (dd, \(J = 12.4, 5.0\) Hz, 1H), 7.05 (d, \(J = 7.6\) Hz, 2H), 3.82 (s, 2H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 169.6, 150.6, 133.4, 131.9, 130.7, 129.5, 128.9, 126.0, 121.4, 40.7. FTIR (thin film): 1762 cm\(^{-1}\) (CH\(_2\)COOR); HRMS(ESI\(^+\)) calculated for C\(_{14}\)H\(_{11}\)ClO\(_2\) [M +Na\(^+\)] 269.0345, found 269.0340.

Phenyl 2-(4-cyanophenyl)acetate (7ag), this compound was prepared according to General Procedure C for 12 h, Yield: 56% (26.5 mg), white solid; Mp: 64-66 °C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.66 (d, \(J = 8.3\) Hz, 2H), 7.50 (d, \(J = 8.1\) Hz, 2H), 7.38 (t, \(J = 8.0\) Hz, 2H), 7.06 (m, \(J = 8.0\) Hz, 2H), 3.93 (s, 2H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 168.8, 150.5, 138.7, 132.5, 130.3, 129.5, 126.2, 121.3, 118.6, 111.5, 41.3. FTIR (KBr): 1749 cm\(^{-1}\) (CH\(_2\)COOR); HRMS(ESI\(^+\)) calculated for C\(_{15}\)H\(_{11}\)FO\(_2\) [M+H\(^+\)] 260.0687, found 260.0682.

Phenyl 2-(4-(trifluoromethyl)phenyl)acetate (7ah), this compound was prepared according to General Procedure C for 14 h, Yield: 48% (27.0 mg), colorless liquid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.63 (d, \(J = 8.1\) Hz, 2H), 7.51 (d, \(J = 8.1\) Hz, 2H), 7.37(t, \(J = 7.8\) Hz, 2H), 7.22 (t, \(J = 7.6\) Hz, 1H), 7.07 (t, \(J = 8.4\) Hz, 2H), 3.92 (s, 2H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 169.2, 150.8, 137.4, 129.8, 129.8(d, \(J_{C,F} = 128.0\) Hz), 129.5, 126.1, 125.7 (q, \(J_{C,F} = 3.8\) Hz), 124.1 (d, \(J_{C,F} = 272.0\) Hz), 121.4, 41.1. FTIR (thin film): 1756 cm\(^{-1}\) (CH\(_2\)COOR); HRMS(ESI\(^+\)) calculated for C\(_{15}\)H\(_{11}\)F\(_3\)O\(_2\) [M+Na\(^+\)] 303.0609, found 303.0603.

Tert-butyl 4-(2-oxo-2-phenoxyethyl)benzoate (7ai), this compound was prepared according to General Procedure C for 12 h, Yield: 67% (42.0 mg), colorless liquid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.99 (d, \(J = 8.2\) Hz, 2H), 7.44 (d, \(J = 8.2\) Hz, 2H), 7.36 (t, \(J = 7.6\) Hz, 2H), 7.21 (t, \(J = 7.4\) Hz, 1H), 7.04(d,7.6 Hz, 2H), 3.90 (s, 2H), 1.60 (s, 9H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 169.4, 165.5, 150.7, 138.0, 131.2, 129.9, 129.5, 129.2, 126.0, 121.4, 81.1, 41.4, 28.2. FTIR (thin film): 1757 cm\(^{-1}\) (CH\(_2\)COOR); HRMS(ESI\(^+\)) calculated for C\(_{19}\)H\(_{20}\)O\(_4\) [M+Na\(^+\)] 313.1440, found 313.1445.
Phenyl 2-(3,4-difluorophenyl)acetate (7aj), this compound was prepared according to General Procedure C for 10 h, Yield: 83% (41.2 mg), colorless liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.37 (t, $J = 8.0$ Hz, 2H), 7.25 – 7.19 (m, 2H), 7.18 – 7.02 (m, 4H), 3.81 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.3, 151.3 (dd, $J = 47.0$, 12.7 Hz), 150.6, 148.8 (dd, $J = 46.5$, 12.5 Hz), 130.2 (q, $J_{C,F} = 40.0$ Hz), 129.5, 126.1, 125.5 (dd, $J_{C,F} = 39.6$, 9.6 Hz), 121.4, 118.5 (d, $J = 17.7$ Hz), 117.5 (d, $J = 17.3$ Hz), 40.4. FTIR (thin film): 1761 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{14}$H$_{10}$F$_2$O$_2$ [M+Na$^+$] 271.0547, found 271.0548.

Phenyl 2-(naphthalen-2-yl)acetate (7ak), this compound was prepared according to General Procedure C for 12 h, Yield: 32% (16.9 mg), white solid; Mp: 109-110 $^\circ$C, $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.84 (t, $J = 8.8$ Hz, 4H), 7.50 (m, $J = 9.2$, 8.0, 2.7 Hz, 3H), 7.34 (t, $J = 7.9$ Hz, 2H), 7.20 (t, $J = 7.4$ Hz, 1H), 7.06 (d, $J = 8.8$ Hz,2H), 4.02 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.0, 150.8, 133.5, 132.6, 131.0, 129.4, 128.5, 128.2, 127.8, 127.7, 127.3, 126.3, 126.0, 125.9, 121.5, 41.7. FTIR (KBr): 1755 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{18}$H$_{14}$O$_2$ [M+Na$^+$] 285.0891, found 285.0886.

P-tolyl 2-phenylacetate (7al), this compound was prepared according to General Procedure C for 8 h, Yield: 83% (37.5 mg), colorless liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.42 – 7.26 (m, 5H), 7.13 (d, $J = 8.3$ Hz, 2H), 6.93 (d, $J = 8.3$ Hz, 2H), 3.83 (s, 2H), 2.31 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.2, 148.6, 135.5, 133.6, 129.9, 129.3, 128.7, 127.3, 121.1, 41.4, 20.9. FTIR (thin film): 1740 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{15}$H$_{14}$O$_2$ [M+Na$^+$] 249.0891, found 249.0893.

4-isopropylphenyl 2-phenylacetate (7am), this compound was prepared according to General Procedure C for 10 h, Yield: 89% (45.2 mg), colorless liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.43 – 7.25 (m, 5H), 7.19 (d, $J = 8.5$ Hz, 2H), 6.96 (d, $J = 8.5$ Hz, 2H), 3.83 (s, 2H), 2.94 –2.82 (m, 1H), 1.22 (d, $J = 6.9$ Hz, 6H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.2, 148.7, 146.4, 133.6, 129.3, 128.7, 127.3, 121.1, 41.5, 33.6, 24.0. FTIR (thin film): 1762 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{17}$H$_{18}$O$_2$ [M+Na$^+$] 277.1204, found 277.1199.
4-(tert-butyl)phenyl 2-phenylacetate (7an), this compound was prepared according to General Procedure C for 8 h, Yield: 84% (44.9 mg), colorless liquid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.40 – 7.32 (m, 6H), 7.31 – 7.26 (m, 1H), 7.00 – 6.95 (m, 2H), 3.84 (s, 2H), 1.29 (s, 9H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 170.2, 148.7, 148.5, 133.7, 129.3, 128.7, 127.3 126.3, 120.8, 41.5, 34.5, 31.4. FTIR (thin film): 1759 cm\(^{-1}\) (CH\(_2\)COOR); HRMS(ESI\(^+\)) calculated for C\(_{18}\)H\(_{20}\)O\(_2\) [M+Na\(^+\)] 291.1361, found 291.1356.

4-methoxyphenyl 2-phenylacetate (7ao), this compound was prepared according to General Procedure C for 10 h, Yield: 87% (42.1 mg), colorless liquid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.40 – 7.34 (m, 4H), 7.33 – 7.27 (m, 1H), 6.97 (d, \(J = 9.2\) Hz, 2H), 6.85(d, \(J = 9.2\) Hz, 2H), 3.83 (s, 2H), 3.77 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 170.4, 157.3, 144.3, 133.6, 129.3, 128.7, 127.3, 122.2, 114.4, 55.6, 41.4. FTIR (thin film): 1739 cm\(^{-1}\) (CH\(_2\)COOR); HRMS(ESI\(^+\)) calculated for C\(_{15}\)H\(_{14}\)O\(_3\) [M+Na\(^+\)] 265.0841, found 265.0835.

O-tolyl 2-phenylacetate (7ap), this compound was prepared according to General Procedure C for 8 h, Yield: 76% (34.3 mg), colorless liquid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.42 –7.33 (m, 4H), 7.29 (t, \(J = 7.2\) Hz, 1H), 7.17 (t, \(J = 7.2\) Hz, 2H), 7.13 – 7.07 (m, 1H), 6.97 (d, \(J = 7.8\) Hz, 1H), 3.86 (s, 2H), 2.02 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 169.7, 149.4, 133.6, 131.2, 130.2, 129.4, 128.8, 127.4, 126.9, 126.1, 121.8, 41.5, 16.0. FTIR (thin film): 1754 cm\(^{-1}\) (CH\(_2\)COOR); HRMS(ESI\(^+\)) calculated for C\(_{15}\)H\(_{14}\)O\(_2\) [M+Na\(^+\)] 249.0891, found 249.0886

4-fluorophenyl 2-phenylacetate (7aq), this compound was prepared according to General Procedure C for 12 h, Yield: 80% (36.7 mg), colorless liquid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.41 – 7.28 (m, 5H), 7.06 – 6.99 (m, 4H), 3.85 (s, 2H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 170.1, 160.3 (d, \(J_{C-F} = 244.2\) Hz), 146.6 (d, \(J_{C-F} = 2.7\) Hz), 133.4, 129.3, 128.8, 127.5, 122.9 (d, \(J_{C-F} = 8.5\) Hz), 116.0 (d, \(J_{C-F} = 23.5\) Hz), 41.3. FTIR (thin film): 1759 cm\(^{-1}\) (CH\(_2\)COOR); HRMS(ESI\(^+\)) calculated for C\(_{14}\)H\(_{11}\)FO\(_2\) [M+Na\(^+\)] 253.0641, found 253.0635.
4-chlorophenyl 2-phenylacetate (7ar), this compound was prepared according to General Procedure C for 8 h, Yield: 89% (43.7 mg), colorless liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.36 (d, $J$ = 4.4 Hz, 4H), 7.33 – 7.27 (m, 3H), 7.02 – 6.97 (m, 2H), 3.84 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.8, 149.3, 133.3, 131.3, 129.5, 129.3, 128.8, 127.5, 122.9, 41.4. FTIR (thin film): 1747 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{14}$H$_{11}$ClO$_2$ [M+Na$^+$] 269.0345, found 269.0340.

4-cyanophenyl 2-phenylacetate (7as), this compound was prepared according to General Procedure C for 10 h, Yield: 92% (43.7 mg), colorless liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.66 (d, $J$ = 8.8 Hz, 2H), 7.42 – 7.28 (m, 5H), 7.20 (d, $J$ = 8.7 Hz, 2H), 3.88 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.2, 154.0, 133.7, 132.8, 129.3, 128.9, 127.7, 122.7, 118.2, 109.8, 41.3. FTIR (thin film): 1766 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{15}$H$_{11}$NO$_2$ [M+Na$^+$] 260.0687, found 260.0682.

4-(trifluoromethyl)phenyl 2-phenylacetate (7at), this compound was prepared according to General Procedure C for 8 h, Yield: 91% (50.9 mg), yellow liquid; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.62 (d, $J$ = 8.8 Hz, 2H), 7.39 – 7.30 (m, 5H), 7.19 (d, $J$ = 8.4 Hz, 2H), 3.87 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.5, 153.3, 133.1, 129.4, 128.9, 128.2 (q, $J_{C-F}$ = 32.9 Hz), 127.6, 126.8 (q, $J_{C-F}$ = 3.7 Hz), 123.9 (d, $J_{C-F}$ = 271.9 Hz), 122.0, 41.4. FTIR (thin film): 1750 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{15}$H$_{11}$F$_3$O$_2$ [M+Na$^+$] 303.0609, found 303.0603.

Methyl 4-(2-phenyl acetox)benzoate (7au), this compound was prepared according to General Procedure C for 10 h, Yield: 58% (31.3 mg), white solid; Mp: 88-89 ℃. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.04 (d, $J$ = 8.8 Hz, 2H), 7.38 (d, $J$ = 4.3 Hz, 3H), 7.32 (t, $J$ = 4.2 Hz 1H), 7.14 (d, $J$ = 8.8 Hz, 2H), 3.90 (s, 3H), 3.87 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.5, 166.3, 154.4, 133.1, 131.1, 129.3, 128.8, 127.8, 127.5, 121.5, 52.2, 41.4. FTIR(KBr): 1745 cm$^{-1}$ (CH$_2$COOR); HRMS(ESI$^+$) calculated for C$_{16}$H$_{14}$O$_4$ [M+H$^+$] 271.0970, found 271.0965.
Naphthalen-2-yl 2-phenylacetate (7av), this compound was prepared according to General Procedure C for 10 h, Yield: 58% (30.2 mg), white solid; Mp: 82-83 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.82 (d, J = 8.9 Hz, 2H), 7.79 – 7.75 (m, 1H), 7.53 (d, J = 2.1 Hz, 1H), 7.50 – 7.36 (m, 6H), 7.35 – 7.29 (m, 1H), 7.19 (dd, J = 8.9, 2.3 Hz, 1H), 3.91 (s, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 170.2, 148.5, 133.8, 133.5, 131.5, 129.4, 129.4, 128.9, 127.8, 127.7, 127.5, 126.6, 125.8, 121.0, 118.5, 41.5. FTIR(KBr): 1760 cm⁻¹ (CH₂COOR); HRMS(ESI⁺) calculated for C₁₈H₁₄O₂ [M+Na⁺] 285.0891, found 285.0886.

3,5-dimethylphenyl 2-phenylacetate (7aw), this compound was prepared according to General Procedure C for 10 h, Yield: 71% (34.1 mg), colorless liquid; ¹H NMR (400 MHz, CDCl₃) δ 7.42 – 7.33 (m, 4H), 7.32 – 7.26 (m, 1H), 6.84 (s, 1H), 6.66 (s, 2H), 3.83 (s, 2H), 2.28 (s, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 170.2, 150.7, 139.3, 133.6, 129.3, 128.7, 127.6, 127.3, 119.0, 41.5, 21.2. FTIR(thin film): 1758 cm⁻¹ (CH₂COOR); HRMS(ESI⁺) calculated for C₁₆H₁₆O₂ [M+Na⁺] 263.1048, found 263.1043.

2,4-difluorophenyl 2-phenylacetate (7ax), this compound was prepared according to General Procedure C for 10 h, Yield: 73% (36.2 mg), colorless liquid; ¹H NMR (400 MHz, CDCl₃) δ 7.39 – 7.27 (m, 5H), 7.08 – 7.00 (m, 1H), 6.93 – 6.78 (m, 2H), 3.89 (s, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 169.1, 161.5 (dd, J_C-F = 247.4, 10.5 Hz), 155.4 (dd, J_C-F = 252.1, 12.5 Hz), 134.6 (dd, J_C-F = 13.0, 4.0 Hz), 133.0, 129.3, 128.8, 127.5, 124.2 (dd, J_C-F = 9.9, 2.0 Hz), 111.2 (dd, J_C-F = 23.1, 3.9 Hz), 105.1 (dd, J_C-F = 26.9, 22.4 Hz), 40.8. FTIR(thin film): 1752 cm⁻¹ (CH₂COOR); HRMS(ESI⁺) calculated for C₁₄H₁₀F₂O₂ [M+Na⁺] 271.0547, found 271.0548.

Perfluorophenyl 2-phenylacetate (7ay), this compound was prepared according to General Procedure C for 12 h, Yield: 48% (28.7 mg), white solid; ¹H NMR (400 MHz, CDCl₃) δ 7.46 – 7.29 (m, 4H), 3.97 (s, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 167.5, 132.1, 129.3, 129.0, 127.9, 127.6, 127.3, 119.0, 41.5, 21.2. FTIR(thin film): 1748 cm⁻¹ (CH₂COOR); HRMS(ESI⁺) calculated for C₁₄H₈F₅NO [M + Na⁺] 324.0424, found 324.0430.
Part 8: References:


(2) The reductive aminations were carried out according to literature procedure, except that Me₂NH was formed in situ from Me₂N·HCl and Et₃N. See: Bhattacharyya, S. Synth. Commun. 2000, 30, 2001.


Part 9. $^1$H NMR and $^{13}$C NMR Spectra

$^1$H NMR Spectra of 3aa
$^{13}$C NMR Spectra of 3aa

$^1$H NMR Spectra of 3ab
$^{13}$C NMR Spectra of 3ab

$^1$H NMR Spectra of 3ac
$^{13}$C NMR Spectra of 3ac

$^1$H NMR Spectra of 3ad
$^{13}\text{C NMR Spectra of 3ad}$

$^{1}\text{H NMR Spectra of 3ae}$
\( ^{13}\text{C} \) NMR Spectra of 3ae

\( ^{1}\text{H} \) NMR Spectra of 3af
13C NMR Spectra of 3af

1H NMR Spectra of 3ag
$^{13}$C NMR Spectra of 3ag

$^1$H NMR Spectra of 3ah
**13C NMR Spectra of 3ah**

**1H NMR Spectra of 3ai**
$^{13}$C NMR Spectra of 3ai

$^1$H NMR Spectra of 3aj
$^{13}$C NMR Spectra of 3aj

$^1$H NMR Spectra of 3ak
$^{13}$C NMR Spectra of 3ak

$^1$H NMR Spectra of 3al
$^{13}$C NMR Spectra of 3al

$^1$H NMR Spectra of 3am
$^{13}\text{C NMR Spectra of 3am}$

$^1\text{H NMR Spectra of 3an}$
$^{13}$C NMR Spectra of 3am

$^1$H NMR Spectra of 3ao
$^{13}$C NMR Spectra of 3ao

$^1$H NMR Spectra of 3ap
$^{13}$C NMR Spectra of 3ap

$^1$H NMR Spectra of 3aq
$^{13}$C NMR Spectra of 3aq

$^1$H NMR Spectra of 3ar
$^{13}$C NMR Spectra of 3ar

$^1$H NMR Spectra of 3as
$^{13}$C NMR Spectra of 3as

$^1$H NMR Spectra of 3at
$^{13}$C NMR Spectra of 3at

$^1$H NMR Spectra of 3au
$^{13}$C NMR Spectra of 3au

$^1$H NMR Spectra of 3av
$^{13}$C NMR Spectra of 3av

$^1$H NMR Spectra of 3aw
$^{13}\text{C}$ NMR Spectra of 3aw

$^1\text{H}$ NMR Spectra of 3ax
$^{13}$C NMR Spectra of 3ax

$^1$H NMR Spectra of 3ay
\[ ^{13}\text{C NMR Spectra of 3ay} \]

\[ ^{1}\text{H NMR Spectra of 3az} \]
$^{13}$C NMR Spectra of 3az

$^1$H NMR Spectra of 3ba

S67
$^{13}$C NMR Spectra of 3ba

$^1$H NMR Spectra of 3bb
$^{13}$C NMR Spectra of 3bb

$^1$H NMR Spectra of 3bc
$^{13}$C NMR Spectra of 3bc

$^1$H NMR Spectra of 3bd
$^{13}$C NMR Spectra of 3bd

$^1$H NMR Spectra of 3be
$^{13}$C NMR Spectra of 3be

$^1$H NMR Spectra of 3bf
$^1$H NMR Spectra of 3bg

$^{13}$C NMR Spectra of 3bf
\( ^{13}\text{C} \text{ NMR Spectra of 3bg} \)

\( ^{1}\text{H} \text{ NMR Spectra of 3bh} \)
$^{13}$C NMR Spectra of 3bh

$^1$H NMR Spectra of 3bi
$^{13}$C NMR Spectra of 3bi

$^1$H NMR Spectra of 3bj
$^{13}$C NMR Spectra of 3bj

$^1$H NMR Spectra of 5aa
$^{13}$C NMR Spectra of 5aa

$^1$H NMR Spectra of 5ab
$^{13}$C NMR Spectra of 5ab

$^1$H NMR Spectra of 5ac
$^{13}$C NMR Spectra of 5ac

$^1$H NMR Spectra of 5ad
$^{13}$C NMR Spectra of $5\text{ad}$

$^1$H NMR Spectra of $5\text{ae}$
$^{13}$C NMR Spectra of 5ae

$^1$H NMR Spectra of 5af
$^{13}$C NMR Spectra of 5af

$^1$H NMR Spectra of 5ag

S83
$^{13}$C NMR Spectra of 5ag

$^1$H NMR Spectra of 5ah
$^{13}$C NMR Spectra of 5ah

$^1$H NMR Spectra of 5ai
$^{13}$C NMR Spectra of 5ai

$^1$H NMR Spectra of 5aj
$^{13}$C NMR Spectra of 5aj

$^1$H NMR Spectra of 5ak
$^1$H NMR Spectra of 5al

$^{13}$C NMR Spectra of 5ak
$^{13}$C NMR Spectra of 5al

$^1$H NMR Spectra of 5am
$^{13}$C NMR Spectra of 5am

$^1$H NMR Spectra of 5an
$^{13}$C NMR Spectra of 5an

$^1$H NMR Spectra of 5ao
$^{13}$C NMR Spectra of 5ao

$^1$H NMR Spectra of 5ap
$^{13}$C NMR Spectra of 5ap

$^1$H NMR Spectra of 5aq
$^{13}$C NMR Spectra of 5aq

$^1$H NMR Spectra of 5ar
$^{13}$C NMR Spectra of 5ar

$^1$H NMR Spectra of 5as
$^{13}$C NMR Spectra of 5as

$^1$H NMR Spectra of 5at
$^{13}$C NMR Spectra of 5at

$^1$H NMR Spectra of 5au
$^{13}$C NMR Spectra of 5au

$^1$H NMR Spectra of 5av
$^{13}$C NMR Spectra of 5av

$^1$H NMR Spectra of 5aw
$^{13}$C NMR Spectra of 5aw

$^1$H NMR Spectra of 5ax
$^{13}$C NMR Spectra of 5ax

$^1$H NMR Spectra of 5ay
$^{13}$C NMR Spectra of 5ay

$^1$H NMR Spectra of 7aa
\[ \text{\textsuperscript{13}C NMR Spectra of 7aa} \]

\[ \text{\textsuperscript{1}H NMR Spectra of 7ab} \]
\[ \text{\(^{13}C\) NMR Spectra of 7ab} \]

\[ \text{\(^{1}H\) NMR Spectra of 7ac} \]
$^{13}$C NMR Spectra of 7ac

$^1$H NMR Spectra of 7ad
$^{13}$C NMR Spectra of 7ad

$^1$H NMR Spectra of 7ae
$^{13}$C NMR Spectra of 7ae

$^1$H NMR Spectra of 7af
$^{13}$C NMR Spectra of 7af

$^1$H NMR Spectra of 7ag
$^{13}$C NMR Spectra of 7ag

$^1$H NMR Spectra of 7ah
\textbf{\( ^{13} \text{C} \) NMR Spectra of 7ah}

\textbf{\( ^{1} \text{H} \) NMR Spectra of 7ai}

S110
$^{13}$C NMR Spectra of 7ai

$^1$H NMR Spectra of 7aj
$^{13}$C NMR Spectra of 7aj

$^1$H NMR Spectra of 7ak
$^{13}$C NMR Spectra of 7ak

$^1$H NMR Spectra of 7al
$^{13}$C NMR Spectra of 7al

$^1$H NMR Spectra of 7am
$^{13}$C NMR Spectra of 7am

$^1$H NMR Spectra of 7an
$^{13}$C NMR Spectra of 7an

$^1$H NMR Spectra of 7ao
$^{13}$C NMR Spectra of 7ao

$^1$H NMR Spectra of 7ap

S117
$^1$H NMR Spectra of 7aq

$^{13}$C NMR Spectra of 7ap
$^{13}$C NMR Spectra of 7aq

$^1$H NMR Spectra of 7ar
$^{13}$C NMR Spectra of 7ar

$^1$H NMR Spectra of 7as
$^1$H NMR Spectra of 7at

$^{13}$C NMR Spectra of 7as
$^{13}$C NMR Spectra of 7at

$^1$H NMR Spectra of 7au
\textbf{13C NMR Spectra of 7au}
$^1$H NMR Spectra of 7av

$^{13}$C NMR Spectra of 7av
$^1$H NMR Spectra of 7ax

$^{13}$C NMR Spectra of 7ax
**\( ^1\)H NMR Spectra of 7ay**

![1H NMR Spectra of 7ay](image1)

**\( ^{13}\)C NMR Spectra of 7ay**

![13C NMR Spectra of 7ay](image2)