Chemoselective C(α)-C(β) bond cleavage of saturated aryl ketones with amines leading to α-ketoamides: A copper-catalyzed aerobic oxidation process with air
Chengkou Liu, Zhao Yang, Yu Zeng, Zheng Fang* and Kai Guo*

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1. General information

Reagents: Commercially available reagents were used without any further purification. Commercially unavailable ketones in Table 2 have been prepared according to the literatures: 3-hydroxy-1-phenyl-1-propanone (entry 25), 3-methoxy-1-phenyl-1-propanone (entry 26), 3-benzoyl-propionitrile (entry 27).

Solvents: All organic solvents were also of reagent grade quality without any further purification.

Chromatography: Flash column chromatography was performed using silicycle silica gel (200-300 mesh).

Analytical thin-layer chromatography (TLC) was performed on 0.2 mm coated silica gel plates (HSGF 254) and visualized using a UV lamp (254 nm or 365 nm).

Nuclear Magnetic Resonance Spectroscopy:

\(^1\)H NMR was recorded on magnet system 400'54 ascend purchased from Bruker Biospin AG.

\(^1\)H NMR spectra chemical shifts (δ) are reported in parts per million (ppm) referenced to TMS (0 ppm).

\(^{13}\)C NMR spectra chemical shifts (δ) are reported in parts per million (ppm) were referenced to carbon resonances in the NMR solvent.

ESI-MS spectra were recorded on Agilent Q-TOF 6520.

2. Optimization data

2.1. Optimization data of selective cleavage of C(α)-C(β) bond of propiophenone

2.1.1. The base screening

Table S1. The base screening.a

<table>
<thead>
<tr>
<th>Entry</th>
<th>Catalyst</th>
<th>Base (equiv)</th>
<th>Yieldb (%)</th>
<th>Yield ratio (3a/4a)</th>
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<td>3a</td>
<td>4a</td>
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<td>K(_2)CO(_3)</td>
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<td>Cu(OAc)(_2)</td>
<td>Cs(_2)CO(_3)</td>
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aReaction conditions: 1a (1 mmol), 2a (3 mmol), Cu(OAc)\(_2\) (0.2 mmol), base (2 mmol), toluene (1 mL), 70 °C, \(O_2\) (O\(_2\) balloon), 18h. bIsolated yield.
## 2.1.2. The temperature and solvent screening

Table S2. The temperature and solvent screening.

<table>
<thead>
<tr>
<th>Entry</th>
<th>T (℃)</th>
<th>Solvent</th>
<th>Yield (%)</th>
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<td>H₂O</td>
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<td>PhCN</td>
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<td>15</td>
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*aReaction conditions: 1a (1 mmol), 2a (3 mmol), CuSCN (0.2 mmol), DMAP (2 mmol), solvent (1 mL), temperature, O₂ (O₂ balloon), 18h. bIsolated yield.

## 2.1.3 The equiv. of catalyst, amine and base screening

Table S3. The equiv. of catalyst, amine and base screening.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Equiv. of catalyst</th>
<th>Equiv. of amine</th>
<th>Equiv. of DMAP</th>
<th>Yield (%)</th>
<th>Yield ratio (3a/4a)</th>
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<td>4a</td>
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<td>1</td>
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<td>3.00</td>
<td>0.5</td>
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<td>4.00</td>
<td>1.5</td>
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<td>11</td>
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<tr>
<td>6</td>
<td>0.20</td>
<td>3.50</td>
<td>1.5</td>
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<td>10</td>
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<tr>
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<td>3.00</td>
<td>1.5</td>
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<td>2.50</td>
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*aReaction conditions: 1a (1 mmol), 2a, CuSCN, DMAP, solvent (1 mL), 70 ℃, O₂ (O₂ balloon), 18h. bIsolated yield.
2.2. Optimization data of selective cleavage of C(O)-C(α) bond of propiophenone

As for the formation of C(O)-C(α) bond cleavage product 4a, solvent screening indicated that H₂O was best choice with 9.75 yield ratio of 4a to 3a (Table S4, entries 1-6). However, the conversion was unsatisfactory (Table S4, entry 5). To our delight, 89% 4a was isolated with only trace amounts of 3a when the reaction was performed in H₂O using Cu(hfacac)₂ as the catalyst (Table S4, entry 7).

Table S4. The selective cleavage of C(O)-C(α) bond of propiophenone.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Catalyst</th>
<th>Solvent</th>
<th>Yield (%)</th>
<th>Yield ratio (4a/3a)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>CuCl₂•2H₂O</td>
<td>Neat</td>
<td>50</td>
<td>43</td>
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<td>CuCl₂•2H₂O</td>
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<td>37</td>
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<td>CuCl₂•2H₂O</td>
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<tr>
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<td>CuCl₂•2H₂O</td>
<td>H₂O</td>
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</tr>
<tr>
<td>6</td>
<td>CuCl₂•2H₂O</td>
<td>PhCN</td>
<td>49</td>
<td>30</td>
</tr>
<tr>
<td>7</td>
<td>Cu(hfacac)₂</td>
<td>H₂O</td>
<td>trace</td>
<td>89</td>
</tr>
</tbody>
</table>

*aReaction conditions: 1a (1 mmol), 2a (3 mmol), catalyst (0.2 mmol), solvent (1 mL), 70 ℃, O₂ (O₂ balloon), 36h. bIsolated yield.

3. General experimental details for the synthesis of C(α)-C(β) bond cleavage products

To a solution of the specific ketone (1 mmol, 1eq) in toluene (1 mL) was added CuSCN (0.05 mmol, 0.05 eq), DMAP (1.5 mmol, 1.5 eq) and amine (3 mmol, 3eq). The mixture was stirred at 70 ℃ under the open air for about 5-48 h. After the TLC revealed that the full conversion of corresponding ketone was completed, the reaction mixture was cooled and diluted with ethyl acetate (30 mL). The crude product was washed with saturated NH₄Cl solution (30 mL) and water (30 mL). The separated organic layer was dried over by anhydrous Na₂SO₄ and filtered. The filtrate was concentrated under reduced pressure and the residue was chromatographed on silica gel using hexanes/ethyl acetate or dichloromethane/methanol to afford the desired product.
4. Control experiments

To a solution of propiophenone (1 mmol, 1 eq) in toluene (1 mL) was added CuSCN (0.05 mmol, 0.05 eq), DMAP (1.5 mmol, 1.5 eq) and morpholine (3 mmol, 3 eq). The mixture was stirred at 70 °C under N₂ for 24 h. The TLC revealed that no desired product was generated.

To a solution of propiophenone (1 mmol, 1 eq) in toluene (1 mL) was added CuSCN (0.05 mmol, 0.05 eq), DMAP (1.5 mmol, 1.5 eq), DABCO (3 mmol, 3 eq) and morpholine (3 mmol, 3 eq). The mixture was stirred at 70 °C under the open air for 9 h. Then, the reaction mixture was cooled and diluted with ethyl acetate (30 mL). The crude product was washed with saturated NH₄Cl solution (30 mL) and water (30 mL). The separated organic layer was dried over by anhydrous Na₂SO₄ and filtered. The filtrate was concentrated under reduced pressure and the residue was chromatographed on silica gel using hexane/ethyl acetate (6:1) to afford the desired product (3a, 168.7 mg, 77%; 4a, 28.7 mg, 15%).

To a solution of propiophenone (1 mmol, 1 eq) in toluene (1 mL) was added CuSCN (0.05 mmol, 0.05 eq), DMAP (1.5 mmol, 1.5 eq) and morpholine (3 mmol, 3 eq). The mixture was stirred at 70 °C under the open air in the dark for 9 h. Then, the reaction mixture was cooled and diluted with ethyl acetate (30 mL). The crude product was washed with saturated NH₄Cl solution (30 mL) and water (30 mL). The separated organic layer was dried over by anhydrous Na₂SO₄ and filtered. The filtrate was concentrated under reduced pressure and the residue was chromatographed on silica gel using hexane/ethyl acetate (6:1) to afford the desired product (3a, 157.7 mg, 72%; 4a, 17.2 mg, 9%).
To a solution of propiophenone (1 mmol, 1 eq) in toluene (1 mL) was added CuSCN (0.05 mmol, 0.05 eq), DMAP (1.5 mmol, 1.5 eq), TEMPO (3 mmol, 3 eq) and morpholine (3 mmol, 3eq). The mixture was stirred at 70 °C under the open air for 9 h. Then, the reaction mixture was cooled and diluted with ethyl acetate (30 mL). The crude product was washed with saturated NH₄Cl solution (30 mL) and water (30 mL). The separated organic layer was dried over anhydrous Na₂SO₄ and filtered. The filtrate was concentrated under reduced pressure and the residue was chromatographed on silica gel using hexanes/ethyl acetate (200:1) to afford the Intermediate A (274.7 mg, 95%).

**1-Phenyl-2-(2,2,6,6-tetramethylpiperidin-1-yl)propan-1-one:**

![1-Phenyl-2-(2,2,6,6-tetramethylpiperidin-1-yl)propan-1-one](image)

$^1$H NMR (300 MHz, DMSO) δ 8.01 (dt, $J = 7.2, 1.5$ Hz, 2H), 7.64 (tt, $J = 7.5, 1.2$ Hz, 1H), 7.53 (tt, $J = 7.2, 1.5$ Hz, 2H), 5.00 (q, $J = 7.0$ Hz, 1H), 1.58 – 1.30 (m, 8H), 1.25 (s, 4H), 1.11 (s, 3H), 0.97 (s, 3H), 0.78 (s, 3H); $^{13}$C NMR (75 MHz, DMSO) δ 201.30, 135.21, 133.83, 129.25, 129.15, 85.36, 59.65, 40.16, 34.07, 33.62, 20.48, 20.40, 19.29, 17.05; HRMS (TOF) m/z [M + H]$^+$ Calcd for C₁₈H₂₇NO₂ 290.2115 found 290.2100.

To a solution of propiophenone (1 mmol, 1 eq) in toluene (1 mL) was added CuSCN (0.05 mmol, 0.05 eq), DMAP (1.5 mmol, 1.5 eq) and TEMPO (3 mmol, 3 eq). The mixture was stirred at 70 °C under the open air for 9 h. Then, the reaction mixture was cooled and diluted with ethyl acetate (30 mL). The crude product was washed with saturated NH₄Cl solution (30 mL) and water (30 mL). The separated organic layer was dried over anhydrous Na₂SO₄ and filtered. The filtrate was concentrated under reduced pressure and the residue was chromatographed on silica gel using hexanes/ethyl acetate (200:1) to afford the Intermediate A (14.5 mg, 5%).

To a solution of propiophenone (1 mmol, 1 eq) in toluene (1 mL) was added CuSCN (0.05 mmol, 0.05 eq), DMAP (1.5 mmol, 1.5 eq) and morpholine (3 mmol, 3eq). The mixture was
stirred at 70℃ under the open air for 9 h. Then, the reaction mixture was cooled and diluted with ethyl acetate (30 mL). The crude product was washed with saturated NH₄Cl solution (30 mL) and water (30 mL). The separated organic layer was dried over anhydrous Na₂SO₄ and filtered. The filtrate was concentrated under reduced pressure and the residue was chromatographed on silica gel using hexanes/ethyl acetate (6:1) to afford the byproduct B (6.0 mg, 2%).

**2,3-di-4-morpholinyl-1-phenyl-2-propen-1-one:**

\[ \text{H NMR (400 MHz, DMSO) } \delta \text{ 7.46 – 7.36 (m, 5H), 6.52 (s, 1H), 3.75 (s, 4H), 3.64 – 3.55 (m, 8H), 3.03 (s, 4H); } \]

\[ \text{13C NMR (101 MHz, DMSO) } \delta \text{ 193.87, 150.23, 142.25, 130.03, 128.65, 128.41, 122.19, 66.93, 66.78, 50.98, 50.25; HRMS (TOF) m/z [M + Na] + Calcd for C}_{17}H_{22}N_{2}O_{3} 325.1523 \text{ found 325.1532.} \]

To a solution of propiophenone (1 mmol, 1eq) in toluene (1 mL) was added CuSCN (0.05 mmol, 0.05 eq), DMAP (1.5 mmol, 1.5 eq) and dibenzylamine (3 mmol, 3eq). The mixture was stirred at 70℃ under the open air for 9 h. Then, the reaction mixture was cooled and diluted with ethyl acetate (30 mL). The crude product was washed with saturated NH₄Cl solution (30 mL) and water (30 mL). The separated organic layer was dried over anhydrous Na₂SO₄ and filtered. The filtrate was concentrated under reduced pressure and the residue was chromatographed on silica gel using hexanes/ethyl acetate (9:1) to afford the desired product (82.3 mg, 25%) and the corresponding formamide C (4.5 mg, 2%).

**N,N-Dibenzyl-2-oxo-2-phenylacetamide:**

\[ \text{H NMR (500 MHz, DMSO) } \delta \text{ 7.93 (d, J = 7.3 Hz, 2H), 7.78 (t, J = 7.4 Hz, 1H), 7.63 (t, J = 7.7 Hz, 2H), 7.43 (t, J = 7.3 Hz, 2H), 7.38-7.31 (m, 5H), 7.30 – 7.23 (m, 3H), 4.62 (s, 2H), 4.38 (s, 2H); } \]

\[ \text{13C NMR (126 MHz, DMSO) } \delta \text{ 191.78, 167.54, 136.58, 135.63, 135.60, 133.09, 129.76, 129.19, 129.05, 128.53, 128.33, 128.24, 128.08, 50.60, 47.05; HRMS (TOF) m/z [M + Na] + Calcd for C}_{22}H_{19}NO_{2} 352.1308 \text{ found 352.1318.} \]

**N,N-Bis(phenylmethyl)formamide:**

\[ \text{H NMR (500 MHz, DMSO) } \delta \text{ 8.50 (s, 1H), 7.40 (t, J = 7.3 Hz, 2H), 7.35 (q, J = 6.8 Hz, 3H), 7.31 – 7.24 (m, 3H), 7.20 (d, J = 7.3 Hz, 2H), 4.39 (s, 2H), 4.32 (s, 2H); } \]

\[ \text{13C NMR (126 MHz, DMSO) } \delta \text{ 163.66, 137.14, 137.01, 129.12, 128.95, 128.28, 128.26, 128.14, 127.68, 50.16, 44.70; HRMS (TOF) m/z [M + Na] + Calcd for C}_{15}H_{15}NO 248.1046 \text{ found 248.1046.} \]
5. Characterization data

1-Morpholino-2-phenylethane-1,2-dione:¹

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow solid; ¹H NMR (400 MHz, CDCl₃) δ 7.95 (d, J = 7.3 Hz, 2H), 7.65 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.7 Hz, 2H), 3.78 (brs, 4H), 3.67–3.60 (m, 2H), 3.40–3.33 (m, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 191.29, 165.59, 135.08, 133.20, 129.82, 129.24, 66.88, 66.81, 46.41, 41.76; HRMS (TOF) m/z [M + Na]+ Calcd for C₁₂H₁₃NO₂ 242.0788 found 242.0783.

1-Morpholino-2-(o-tolyl)ethane-1,2-dione:²

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow solid; ¹H NMR (400 MHz, CDCl₃) δ 7.72 (d, J = 6.8 Hz, 1H), 7.50 (t, J = 6.9 Hz, 1H), 7.33 (dd, J = 10.0, 7.7 Hz, 2H), 3.79 (d, J = 3.1 Hz, 4H), 3.69–3.65 (m, 2H), 3.42–3.37 (m, 2H), 2.66 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 193.21, 166.30, 141.75, 134.00, 132.82, 131.80, 131.58, 126.33, 66.77, 66.75, 46.38, 41.72, 21.94; HRMS (TOF) m/z [M + H]+ Calcd for C₁₃H₁₅NO₃ 234.1125 found 234.1122.

1-Morpholino-2-(m-tolyl)ethane-1,2-dione:³

Purified by column chromatography (hexanes/ethyl acetate 6:1); White solid; ¹H NMR (300 MHz, DMSO) δ 7.71 (s, 2H), 7.55 (dd, J = 19.2, 7.1 Hz, 2H), 3.68 (dd, J = 14.2, 3.5 Hz, 4H), 3.52 (t, J = 4.2 Hz, 2H), 3.28 (t, J = 5.1 Hz, 2H), 2.41 (s, 3H); ¹³C NMR (75 MHz, DMSO) δ 191.70, 164.97, 138.98, 135.83, 132.69, 129.25, 129.23, 126.72, 66.03, 65.83, 45.67, 40.99, 20.71; HRMS (TOF) m/z [M + H]+ Calcd for C₁₃H₁₅NO₃ 234.1125 found 234.1112.

1-Morpholino-2-(p-tolyl)ethane-1,2-dione:⁴

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow solid; ¹H NMR (400 MHz, CDCl₃) δ 7.83 (d, J = 8.1 Hz, 2H), 7.30 (d, J = 8.2 Hz, 2H), 3.73 (brs, 4H), 3.64–3.60 (m, 2H), 3.37–3.33 (m, 2H), 2.42 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 190.98, 165.75, 146.36, 130.73, 129.90, 129.86, 66.82, 66.74, 46.34, 41.64, 22.00; HRMS (TOF) m/z [M + H]+ Calcd for C₁₃H₁₃NO₃ 234.1125 found 234.1106.

1-(4-Methoxyphenyl)-2-morpholinoethane-1,2-dione:⁵

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow solid; ¹H NMR (400 MHz, CDCl₃) δ 7.89 (t, J = 12.9 Hz, 2H), 6.94 (t, J = 12.3 Hz, 2H), 3.88–3.79 (m, 3H), 3.73 (d, J = 25.3 Hz, 4H), 3.60 (t, J = 10.4 Hz, 2H), 3.33 (t, J = 10.7 Hz, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 189.91, 165.89, 165.11, 132.23, 126.24, 114.51, 66.86, 66.76, 55.76, 46.38, 41.64; HRMS (TOF) m/z [M + H]+ Calcd for C₁₃H₁₃NO₄ 250.1074 found 250.1072.

1-(4-fluorophenethyl)-2-morpholinoethane-1,2-dione:⁶
Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.06 – 7.95 (m, 2H), 7.19 (t, \(J = 8.5\) Hz, 2H), 3.79 (s, 4H), 3.70 – 3.62 (m, 2H), 3.43 – 3.34 (m, 2H); \(^13\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 189.50, 166.94 (d, \(J = 256.7\)), 165.22, 132.69 (d, \(J = 9.8\)), 129.75 (d, \(J = 2.8\)), 116.70, 116.48, 66.89, 66.79, 46.44, 41.84; HRMS (TOF) m/z [M + H]\(^+\) Calcd for C\(_{12}\)H\(_{12}\)FNO\(_3\) 238.0874 found 238.0876.

**1-(2-Chlorophenyl)-2-morpholinoethane-1,2-dione:**

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow solid; \(^1\)H NMR (300 MHz, DMSO) \(\delta\) 7.85 (dd, \(J = 7.7\), 1.7 Hz, 1H), 7.69 (t, \(J = 7.7\), 1.7 Hz, 1H), 3.74 – 3.67 (m, 2H), 3.67 – 3.61 (m, 2H), 3.54 (t, \(J = 4.5\) Hz, 2H), 3.32 (t, \(J = 4.8\) Hz, 2H); \(^13\)C NMR (75 MHz, DMSO) \(\delta\) 190.04, 165.26, 135.70, 133.27, 132.88, 132.74, 131.50, 128.38, 66.10, 65.97, 46.12, 41.78; HRMS (TOF) m/z [M + Na]\(^+\) Calcd for C\(_{12}\)H\(_{12}\)ClNO\(_3\) 276.0398 found 276.0366.

**1-(3-Chlorophenyl)-2-morpholinoethane-1,2-dione:**

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow solid; \(^1\)H NMR (300 MHz, DMSO) \(\delta\) 7.92 – 7.79 (m, 3H), 7.66 (t, \(J = 7.7\) Hz, 1H), 3.74 – 3.67 (m, 2H), 3.67 – 3.61 (m, 2H), 3.54 (t, \(J = 4.5\) Hz, 2H), 3.32 (t, \(J = 4.8\) Hz, 2H); \(^13\)C NMR (75 MHz, DMSO) \(\delta\) 190.48, 164.64, 135.31, 134.91, 134.68, 131.88, 128.81, 66.52, 66.24, 46.13, 41.62; HRMS (TOF) m/z [M + Na]\(^+\) Calcd for C\(_{12}\)H\(_{12}\)ClNO\(_3\) 276.0398 found 276.0394.

**1-(4-Chlorophenyl)-2-morpholinoethane-1,2-dione:**

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.98 – 7.85 (m, 2H), 7.56 – 7.46 (m, 2H), 3.82 – 3.74 (m, 4H), 3.69 – 3.63 (m, 2H), 3.42 – 3.34 (m, 2H); \(^13\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 188.83, 164.04, 140.76, 130.61, 130.18, 128.64, 65.88, 65.79, 45.43, 40.85; HRMS (TOF) m/z [M + Na]\(^+\) Calcd for C\(_{12}\)H\(_{12}\)ClNO\(_3\) 276.0398 found 276.0391.

**1-(4-bromophenethyl)-2-morpholinoethane-1,2-dione:**

Purified by column chromatography (hexanes/ethyl acetate 6:1); White solid; \(^1\)H NMR (300 MHz, DMSO) \(\delta\) 7.84 (s, 4H), 3.74-3.68 (m, 2H), 3.67-3.62 (m, 2H), 3.53 (t, \(J = 4.2\) Hz, 2H), 3.30 (t, \(J = 4.5\) Hz, 2H); \(^13\)C NMR (75 MHz, DMSO) \(\delta\) 190.51, 164.42, 132.53, 131.65, 131.16, 129.52, 66.05, 65.77, 45.66, 41.09; HRMS (TOF) m/z [M + Na]\(^+\) Calcd for C\(_{12}\)H\(_{12}\)BrNO\(_3\) 319.9893 found 319.9872.

**1-(4-iodophenyl)-2-morpholinoethane-1,2-dione:**

Purified by column chromatography (hexanes/ethyl acetate 6:1); White solid; \(^1\)H NMR (300 MHz, DMSO) \(\delta\) 8.02 (d, \(J = 8.2\) Hz, 2H), 7.65 (d, \(J = 8.1\) Hz, 2H), 3.72 – 3.67 (m, 2H), 3.65 – 3.60 (m, 2H), 3.52 (t, \(J = 5.1\) Hz, 2H), 3.27 (t, \(J = 4.5\) Hz, 2H); \(^13\)C NMR (75 MHz, DMSO) \(\delta\) 191.41, 164.94, 138.87, 132.37, 131.19, 105.07, 66.52, 66.25, 46.12, 41.54; HRMS (TOF) m/z [M + Na]\(^+\) Calcd
Methyl 4-(2-morpholino-2-oxoacyl)benzoate: \(^9\)

Purified by column chromatography (hexanes/ethyl acetate 4:1); Yellow solid; \(^1^H\) NMR (300 MHz, DMSO) \(\delta\) 8.15 (d, \(J = 8.1\) Hz, 2H), 8.04 (d, \(J = 8.1\) Hz, 2H), 3.90 (s, 3H), 3.76 – 3.59 (m, 4H), 3.53 (t, \(J = 4.2\) Hz, 2H), 3.30 (t, \(J = 5.8\) Hz, 2H); \(^1^C\) NMR (75 MHz, DMSO) \(\delta\) 191.26, 165.76, 164.82, 136.30, 135.26, 130.42, 130.10, 66.51, 66.24, 53.10, 46.14, 41.62; HRMS (TOF) m/z [M + Na]^+ Calcd for C\(_{14}\)H\(_{15}\)NO5 300.0844 found 300.0844.

1-Morpholino-2-(4-(trifluoromethyl)phenyl)ethane-1,2-dione: \(^8\)

Purified by column chromatography (hexanes/ethyl acetate 5:1); White solid; \(^1^H\) NMR (300 MHz, DMSO) \(\delta\) 8.12 (d, \(J = 8.4\) Hz, 2H), 7.98 (d, \(J = 8.4\) Hz, 2H), 3.74 – 3.69 (m, 2H), 3.68 – 3.63 (m, 2H), 3.54 (t, \(J = 5.1\) Hz, 2H), 3.33 (t, \(J = 4.5\) Hz, 2H); \(^1^C\) NMR (75 MHz, DMSO) \(\delta\) 190.79, 164.63, 136.18, 134.74, 134.30, 130.68, 126.79 (q, \(J = 3.4\) Hz), 125.79, 66.54, 66.24, 46.13, 41.65; HRMS (TOF) m/z [M + Na]^+ Calcd for C\(_{13}\)H\(_{12}\)NO3F3 310.0661 found 310.0672.

1-Morpholino-2-(2-nitrophenyl)ethane-1,2-dione: \(^1^3\)

Purified by column chromatography (hexanes/ethyl acetate 4:1); Yellow solid; \(^1^H\) NMR (400 MHz, DMSO) \(\delta\) 8.18 (d, \(J = 8.4\) Hz, 2H), 7.95 (td, \(J = 7.5, 1.1\) Hz, 1H), 7.91 – 7.82 (m, 2H), 7.74 (t, \(J = 8.0\) Hz, 1H), 3.83 – 3.79 (m, 4H), 3.72 – 3.67 (m, 2H), 3.46 – 3.42 (m, 2H); \(^1^C\) NMR (101 MHz, DMSO) \(\delta\) 187.03, 161.75, 147.07, 134.83, 133.48, 131.71, 131.14, 124.15, 66.01 (d, \(J = 16.8\) Hz), 45.98, 42.11; HRMS (TOF) m/z [M + Na]^+ Calcd for C\(_{12}\)H\(_{12}\)N\(_2\)O5 287.0638 found 287.0637.

1-Morpholino-2-(3-nitrophenyl)ethane-1,2-dione: \(^9\)

Purified by column chromatography (hexanes/ethyl acetate 4:1); Yellow solid; \(^1^H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.78 (t, \(J = 1.8\) Hz, 1H), 8.48 (ddd, \(J = 8.2, 2.2, 1.0\) Hz, 1H), 8.34 – 8.27 (m, 1H), 7.74 (t, \(J = 8.0\) Hz, 1H), 3.83 – 3.79 (m, 4H), 3.72 – 3.67 (m, 2H), 3.46 – 3.42 (m, 2H); \(^1^C\) NMR (101 MHz, CDCl\(_3\)) \(\delta\) 188.25, 164.00, 148.73, 135.24, 134.66, 130.48, 128.96, 124.64, 66.85, 66.74 46.48, 42.10; HRMS (TOF) m/z [M + Na]^+ Calcd for C\(_{12}\)H\(_{12}\)N\(_2\)O5 287.0638 found 287.0623.

1-Morpholino-2-(4-nitrophenyl)ethane-1,2-dione: \(^8\)

Purified by column chromatography (hexanes/ethyl acetate 4:1); Yellow solid; \(^1^H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.41 (d, \(J = 8.4\) Hz, 2H), 8.18 (d, \(J = 8.4\) Hz, 2H), 3.74 (t, \(J = 4.4\) Hz, 2H), 3.67 (t, \(J = 5.2\) Hz, 2H), 3.56 (t, \(J = 4.4\) Hz, 2H), 3.36 (t, \(J = 4.8\) Hz, 2H); \(^1^C\) NMR (101 MHz, DMSO) \(\delta\) 189.78, 163.89, 150.90, 136.96, 130.84, 124.37, 66.05, 65.73, 45.66, 41.23; HRMS (TOF) m/z [M + K]^+ Calcd for C\(_{12}\)H\(_{12}\)N\(_2\)O5 303.0378 found 303.0330.
**1-Morpholino-2-(naphthalen-2-yl)ethane-1,2-dione:**\(^{13}\)

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.46 (s, 1H), 8.04 – 7.86 (m, 4H), 7.61 (dt, \(J = 28.4, 7.3\) Hz, 2H), 3.84 (s, 4H), 3.72 – 3.61 (m, 2H), 3.47 – 3.36 (m, 2H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 191.34, 165.70, 136.54, 133.16, 132.53, 130.53, 130.02, 129.66, 129.30, 128.08, 127.35, 126.68, 66.87, 66.82, 46.47, 41.83; HRMS (TOF) m/z [M + H]\(^+\) Calcd for C\(_{16}\)H\(_{15}\)NO\(_3\) 270.1125 found 270.1140.

**1-Morpholino-2-(thiophen-2-yl)ethane-1,2-dione:**

Purified by column chromatography (hexane/ethyl acetate 6:1); Yellow oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.81 (ddd, \(J = 5.9, 4.4, 1.0\) Hz, 2H), 7.17 (dd, \(J = 4.8, 4.0\) Hz, 1H), 3.78 – 3.71 (m, 4H), 3.69 – 3.62 (m, 2H), 3.51 – 3.44 (m, 2H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 182.90, 164.41, 140.36, 136.87, 136.37, 128.82, 66.89, 66.70, 46.52, 42.03; HRMS (TOF) m/z [M + H]\(^+\) Calcd for C\(_{10}\)H\(_{11}\)NO\(_3\)S 226.0532 found 226.0552.

**1-Morpholino-2-(pyridine-3-yl)ethane-1,2-dione:**

Purified by column chromatography (hexane/ethyl acetate 4:1); Yellow solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 9.07 (s, 1H), 8.81 – 8.75 (m, 1H), 8.19 (dd, \(J = 8.0, 2.0\) Hz, 1H), 7.46 – 7.37 (m, 1H), 3.72 (s, 4H), 3.63 – 3.58 (m, 2H), 3.31 – 3.23 (m, 2H), 1.71 – 1.64 (m, 4H), 1.57 – 1.47 (m, 2H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 189.45, 164.13, 154.80, 151.17, 136.73, 128.77, 123.92, 66.68, 66.55, 46.25, 41.79; HRMS (TOF) m/z [M + H]\(^+\) Calcd for C\(_{11}\)H\(_{12}\)N\(_2\)O\(_3\) 221.0921 found 221.0928.

**1-Phenyl-2-(piperidin-1-yl)ethane-1,2-dione:**

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.92 (d, \(J = 1.4\) Hz, 2H), 7.65 – 7.59 (m, 1H), 7.49 (t, \(J = 7.7\) Hz, 2H), 3.69 (s, 2H), 3.31 – 3.23 (m, 2H), 2.51 – 2.45 (m, 2H), 2.36 – 2.32 (m, 2H), 2.28 (s, 3H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 192.05, 165.54, 134.74, 133.36, 129.65, 129.09, 47.12, 42.24, 26.29, 25.54, 24.47; HRMS (TOF) m/z [M + H]\(^+\) Calcd for C\(_{13}\)H\(_{15}\)NO\(_2\) 218.1184 found 218.1184.

**1-(4-Methylpiperazin-1-yl)-2-phenylethane-1,2-dione:**

Purified by column chromatography (dichloromethane/methanol 30:1); Yellow solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.96 – 7.87 (m, 2H), 7.61 (t, \(J = 7.4\) Hz, 1H), 7.48 (t, \(J = 7.7\) Hz, 2H), 3.82 – 3.71 (m, 2H), 3.47 – 3.38 (m, 2H), 2.51 – 2.45 (m, 2H), 2.36 – 2.32 (m, 2H), 2.28 (s, 3H); \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 191.54, 165.44, 134.88, 133.16, 129.69, 129.11, 54.95, 54.49, 46.04, 45.81, 41.20; HRMS (TOF) m/z [M + H]\(^+\) Calcd for C\(_{14}\)H\(_{16}\)N\(_2\)O\(_2\) 233.1285 found 233.1281.

**1-Phenyl-2-(pyrrolidin-1-yl)ethane-1,2-dione:**

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow solid; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.05 – 7.94 (m, 2H), 7.62 (t, \(J = 8.0\) Hz, 1H), 7.48 (t, \(J = 7.7\) Hz, 2H), 3.64 (t, \(J = 6.8\) Hz, 2H), 3.40 (t, \(J =...
6.2 Hz, 2H), 2.00 – 1.87 (m, 4H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 191.59, 164.93, 134.59, 132.86, 129.80, 128.91, 46.62, 45.19, 25.85, 23.96; HRMS (TOF) m/z [M + H]$^+$ Calcd for C$_{12}$H$_{13}$NO$_2$ 204.1019 found 204.1026.

**N,N-Diethyl-2-oxo-2-phenylacetamide:**

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow solid; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.97 – 7.85 (m, 2H), 7.60 (t, $J = 7.4$ Hz, 1H), 7.47 (t, $J = 7.7$ Hz, 2H), 3.53 (q, $J = 7.2$ Hz, 2H), 3.21 (q, $J = 7.1$ Hz, 2H), 1.25 (t, $J = 7.2$ Hz, 3H), 1.12 (t, $J = 7.1$ Hz, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 191.66, 166.80, 134.64, 133.28, 129.64, 129.01, 42.16, 38.85, 14.14, 12.87; HRMS (TOF) m/z [M + H]$^+$ Calcd for C$_{12}$H$_{15}$NO$_2$ 206.1176 found 206.1191.

**N-Butyl-2-oxo-2-phenylacetamide:**

Purified by column chromatography (hexanes/ethyl acetate 7:1); Yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.60 (t, $J = 7.4$ Hz, 1H), 7.40 (t, $J = 7.8$ Hz, 2H), 7.07 (s, 1H), 3.32 (dd, $J = 13.4$, 7.0 Hz, 2H), 1.56 – 1.47 (m, 2H), 1.38 – 1.28 (m, 2H), 0.88 (t, $J = 7.3$ Hz, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 186.93, 160.75, 133.32, 132.35, 130.17, 127.44, 38.13, 30.30, 19.03, 12.68; HRMS (TOF) m/z [M + H]$^+$ Calcd for C$_{12}$H$_{15}$NO$_2$ 206.1176 found 206.1193.

**Morpholino(phenyl)methanone:**

Purified by column chromatography (hexanes/ethyl acetate 6:1); White solid; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.44 – 7.38 (m, 5H), 3.90 – 3.36 (m, 8H); $^{13}$C NMR (101 MHz, CDCl$_3$) 169.4, 134.3, 128.9, 127.5, 126.1, 65.9, 47.3; HRMS (TOF) m/z [M + H]$^+$ Calcd for C$_{11}$H$_{13}$NO$_2$ 191.1019 found 191.1039.

**(2-methylphenyl)(morpholino)methanone:**

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.23 – 7.17 (m, 1H), 7.17 – 7.10 (m, 2H), 7.10-7.55 (m, 1H), 3.79 – 3.65 (m, 4H), 3.49 (s, 2H), 3.16 (d, $J = 4.5$ Hz, 2H), 2.24 (s, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 169.07, 134.60, 133.15, 129.48, 128.03, 124.98, 124.80, 65.96, 65.91, 46.23, 40.88, 17.99; HRMS (TOF) m/z [M + H]$^+$ Calcd for C$_{12}$H$_{15}$NO$_2$ 206.1136 found 206.1153.

**(3-methylphenyl)(morpholino)methanone:**

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.23 – 7.18 (m, 1H), 7.17-7.13 (m, 2H), 7.09 (d, $J = 7.4$ Hz, 1H), 3.74 – 3.31 (m, 8H), 2.29 (s, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 169.59, 137.47, 134.28, 129.54, 127.34, 126.67, 122.95, 65.87, 47.15, 41.50, 20.34; HRMS (TOF) m/z [M + H]$^+$ Calcd for C$_{12}$H$_{13}$NO$_2$ 206.1149.

**(4-methylphenyl)(morpholino)methanone:**

Purified by column chromatography (hexanes/ethyl acetate 6:1);
Yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.30 (d, $J$ = 8.1 Hz, 2H), 7.20 (d, $J$ = 8.0 Hz, 2H), 3.89-3.41 (m, 8H), 2.37 (s, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 169.64, 139.08, 131.31, 128.13, 126.21, 65.90, 20.37; HRMS (TOF) m/z [M + H]$^+$ Calcd for C$_{12}$H$_{15}$NO$_2$ 206.1136 found 206.1147.

(4-methoxyphenyl)(morpholino)methanone$^{14}$

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.39 (dt, $J$ = 8.8, 2.0 Hz, 2H), 6.92 (dt, $J$ = 8.8, 2.0 Hz, 2H), 3.84 (s, 3H), 3.76 – 3.54 (m, 8H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 169.43, 159.90, 128.19, 126.32, 112.78, 65.92, 54.35; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{12}$H$_{15}$NO$_3$ 222.1085 found 222.1094.

(4-hydroxyphenyl)(morpholino)methanone$^{16}$

Purified by column chromatography (hexanes/ethyl acetate 3:1); white solid; $^1$H NMR (500 MHz, DMSO) $\delta$ 9.85 (s, 1H), 7.29 (d, $J$ = 8.1 Hz, 2H), 6.83 (d, $J$ = 8.3 Hz, 2H), 3.60 (t, $J$ = 4.2 Hz, 4H), 3.51 (s, 4H); $^{13}$C NMR (126 MHz, DMSO) $\delta$ 169.83, 159.24, 129.72, 126.24, 115.36, 66.60; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{11}$H$_{13}$NO$_2$ 230.0788 found 230.0794.

(4-fluorophenyl)(morpholino)methanone$^{17}$

Purified by column chromatography (hexanes/ethyl acetate 6:1); Yellow solid; $^1$H NMR (500 MHz, DMSO) $\delta$ 7.51-7.25 (m, 2H), 7.27 (tt, $J$ = 9.1, 2.1 Hz, 2H), 3.70 – 3.31 (m, 8H); $^{13}$C NMR (126 MHz, DMSO) $\delta$ 168.11, 163.47, 161.51, 131.92, 131.90, 129.58, 129.51, 115.37, 115.20, 65.96; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{11}$H$_{12}$FNO$_2$ 232.0744 found 232.0752.

(2-chlorophenyl)(morpholino)methanone$^{18}$

Purified by column chromatography (hexanes/ethyl acetate 6:1); white solid; $^1$H NMR (500 MHz, DMSO) $\delta$ 7.58 – 7.33 (m, 1H), 7.50 – 7.39 (m, 3H), 3.74-3.64 (m, 4H), 3.56 (t, $J$ = 4.7 Hz, 2H), 3.15 (t, $J$ = 4.5 Hz, 2H); $^{13}$C NMR (126 MHz, DMSO) $\delta$ 168.11, 163.47, 161.51, 131.92, 131.90, 129.33, 126.76, 125.42, 65.84, 46.58, 41.47; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{11}$H$_{12}$ClNO$_2$ 248.0449 found 248.0459.

(3-chlorophenyl)(morpholino)methanone$^{18}$

Purified by column chromatography (hexanes/ethyl acetate 6:1); colorless liquid; $^1$H NMR (500 MHz, DMSO) $\delta$ 7.54 – 7.46 (m, 3H), 7.42 – 7.38 (m, 1H), 3.63 (s, 6H), 3.36 (s, 2H); 13C NMR (126 MHz, DMSO) $\delta$ 167.38, 137.62, 133.22, 130.22, 129.33, 126.76, 125.42, 65.92, 47.47, 42.02; HRMS (TOF) m/z [M + Na]$^+$ Calcd for C$_{11}$H$_{12}$ClNO$_2$ 248.0449 found 248.0455.

(4-chlorophenyl)(morpholino)methanone$^{14}$

Purified by column chromatography (hexanes/ethyl acetate 6:1); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.38 (q, $J$ = 8.5 Hz, 4H), 3.89-3.36 (m, 8H);
\(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 168.37, 135.03, 132.61, 127.86, 127.65, 65.83; HRMS (TOF) \(m/z\) [M + Na\(^+\)] Calcd for C\(_{11}\)H\(_{12}\)ClNO\(_2\) 248.0449 found 248.0462.

(4-bromophenethyl)(morpholino)methanone:

Purified by column chromatography (hexanes/ethyl acetate 6:1);
\(\text{\textsuperscript{1}}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.56 (dt, \(J = 8.4, 2.0\) Hz, 2H), 7.29 (dt, \(J = 8.4, 1.6\) Hz, 2H), 3.85-3.40 (m, 8H);
\(\text{\textsuperscript{13}}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 168.38, 133.08, 130.82, 127.83, 123.25, 65.81;
HRMS (TOF) \(m/z\) [M + H\(^+\)] Calcd for C\(_{11}\)H\(_{12}\)BrNO\(_2\) 271.1260 found 271.1282.

(4-iodophenyl)(morpholino)methanone:

Purified by column chromatography (hexane/ethyl acetate 6:1);
White solid;
\(\text{\textsuperscript{1}}\)H NMR (500 MHz, DMSO) \(\delta\) 7.85 (d, \(J = 8.2\) Hz, 2H), 7.24 (d, \(J = 8.2\) Hz, 2H), 3.71-3.34 (m, 8H);
\(\text{\textsuperscript{13}}\)C NMR (126 MHz, DMSO) \(\delta\) 168.19, 137.16, 134.94, 129.02, 96.25, 65.94;
HRMS (TOF) \(m/z\) [M + Na\(^+\)] Calcd for C\(_{11}\)H\(_{12}\)INO\(_2\) 339.9805 found 339.9811.

4-(4-Morpholinylcarbonyl)benzoic acid methyl ester:

Purified by column chromatography (hexane/ethyl acetate 4:1); white solid;
\(\text{\textsuperscript{1}}\)H NMR (500 MHz, DMSO) \(\delta\) 8.04 (d, \(J = 7.9\) Hz, 2H), 7.57 (d, \(J = 7.9\) Hz, 2H), 3.90 (s, 3H), 3.78-3.52 m, 6H), 3.33 (s, 2H);
\(\text{\textsuperscript{13}}\)C NMR (126 MHz, DMSO) \(\delta\) 168.55, 166.14, 140.56, 130.86, 129.76, 127.76, 66.47, 52.76; HRMS (TOF) \(m/z\) [M + Na\(^+\)] Calcd for C\(_{13}\)H\(_{15}\)NO\(_4\) 272.0896 found 272.0890.

(4-(trifluoromethyl)phenyl)(morpholino)methanone:

Purified by column chromatography (hexane/ethyl acetate 5:1);
White solid; \(\text{\textsuperscript{1}}\)H NMR (500 MHz, DMSO) \(\delta\) 8.04 (d, \(J = 7.9\) Hz, 2H), 7.67 (d, \(J = 7.7\) Hz, 2H), 3.79-3.49 (m, 6H), 3.39-3.25 (m, 2H); \(\text{\textsuperscript{13}}\)C NMR (126 MHz, DMSO) \(\delta\) 168.17, 140.20, 130.21 (q, \(J = 32.2\) Hz), 128.27, 127.62, 125.91 (q, \(J = 3.7\) Hz), 125.45, 123.27, 121.12, 66.45, 47.67, 42.32; HRMS (TOF) \(m/z\) [M + Na\(^+\)] Calcd for C\(_{12}\)H\(_{12}\)F\(_3\)NO\(_2\) 282.0712 found 282.0722.

4-Morpholinyl-2-naphthalenylmethanone:

Purified by column chromatography (hexane/ethyl acetate 6:1);
Yellow solid; \(\text{\textsuperscript{1}}\)H NMR (500 MHz, DMSO) \(\delta\) 8.05 – 7.97 (m, 4H), 7.64 – 7.58 (m, 2H), 7.55 (d, \(J = 8.3\) Hz, 1H), 3.65 (s, 8H); \(\text{\textsuperscript{13}}\)C NMR (126 MHz, DMSO) \(\delta\) 169.00, 133.04, 132.88, 132.16, 128.26, 127.97, 127.58, 127.02, 126.63, 126.47, 124.40, 66.05; HRMS (TOF) \(m/z\) [M + H\(^+\)] Calcd for C\(_{15}\)H\(_{13}\)NO\(_2\) 242.1176 found 242.1191.

Phenyl(piperidin-1-yl)methanone:

Purified by column chromatography (hexane/ethyl acetate 6:1);
Colourless oil; \(\text{\textsuperscript{1}}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.31 (s, 5H), 3.63 (s, 2H), 3.27
(s, 2H), 1.70–1.35 (m, 6H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 169.29, 135.51, 128.32, 127.37, 125.76, 47.75, 42.11, 25.49, 24.63, 23.57; HRMS (TOF) m/z [M + H]$^+$ Calcd for C$_{12}$H$_{15}$NO 190.1187 found 190.1190.

(4-Methyl-1-piperazinyl)phenylmethanone: $^{19}$

Purified by column chromatography (dichloromethane/methanol 30:1); Yellow oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.45 – 7.34 (m, 5H), 3.78 (s, 2H), 3.39 (s, 2H), 2.53 – 2.27 (m, 7H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 169.33, 134.73, 128.68, 127.46, 126.01, 54.16, 53.63, 46.51, 44.89, 40.92; HRMS (TOF) m/z [M + H]$^+$ Calcd for C$_{12}$H$_{16}$N$_2$O 205.1296 found 205.1304.

Phenyl(pyrrolidin-1-yl)methanone: $^{19}$

Purified by column chromatography (hexane/ethyl acetate 6:1); Colourless oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.46 – 7.41 (m, 2H), 7.35 – 7.28 (m, 3H), 3.46 (d, $^J = 78.4$ Hz, 4H), 1.84 (s, 4H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 168.74, 136.17, 128.76, 127.22, 126.05, 48.60, 45.18, 25.36, 23.46; HRMS (TOF) m/z [M + H]$^+$ Calcd for C$_{11}$H$_{13}$NO 176.1031 found 176.1042.

$^{N,N}$-Diethylbenzamide: $^{19}$

Purified by column chromatography (hexane/ethyl acetate 6:1); Colourless oil; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.59 – 7.31 (m, 5H), 3.54 (s, 2H), 3.25 (s, 2H), 1.27 – 0.99 (m, 6H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 170.32, 136.24, 128.08, 127.37, 125.26, 42.29, 38.21, 13.18, 11.91; HRMS (TOF) m/z [M + H]$^+$ Calcd for C$_{11}$H$_{15}$NO 178.1187 found 178.1189.

$^N$-$n$-Butylbenzamide: $^{20}$

Purified by column chromatography (hexane/ethyl acetate 7:1); White solid; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.85 – 7.51 (m, 2H), 7.48 – 7.09 (m, 3H), 6.40-6.07 (m, 1H), 3.53 – 3.11 (m, 2H), 1.65-1.50 (m, 2H), 1.45-1.31 (m, 2H), 0.99-0.82 (m, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 167.57, 134.89, 131.25, 128.49, 126.85, 39.81, 31.74, 20.16, 13.77; HRMS (TOF) m/z [M + H]$^+$ Calcd for C$_{11}$H$_{15}$NO 178.1187 found 178.1191.
6. NMR spectra of products in Table 2, Table 3 and Scheme 2
7. References