A chemoselective α-aminoxylation of aryl ketones: A cross dehydrogenative coupling reaction catalysed by Bu4NI

Yogesh Siddaraju and Kandikere Ramaiah Prabhu*

Department of Organic Chemistry, Indian Institute of Science, Bangalore 560 012, Karnataka, India

*e-mail: prabhu@orgchem.iisc.ernet.in

Fax: (+)91-80-23600529
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**General experimental**

NMR spectra were recorded on a BRUKER-AV400 spectrometer in CDCl$_3$, Tetramethylsilane (TMS; $\delta = 0.00$ ppm) served as an internal standard for $^1$H NMR. The corresponding residual non-deuterated solvent signal (CDCl$_3$; $\delta = 77.00$ ppm) was used as internal standard for $^{13}$C NMR. IR spectra were measured using Perkin-Elmer FT/IR Spectrum BX, GX. Mass spectra were measured with Micromass Q-Tof (ESI-HRMS). Column chromatography was carried out on Silica gel 100-200 mesh (commercial suppliers) and thin-layer chromatography was carried out using SILICA GEL GF-254.
Experimental Section

Typical experimental procedure for Friedel Crafts acylation of arenes, starting material synthesis: To a solution of anhydrous aluminiumtrichloride (1.1 equiv) in DCE (10 mL) was added acid chloride (1.1 equiv), slowly at 0°C, the reaction mixture was stirred at 0°C for 10 min and was added a solution of arene in DCE (500 mg – 1g in 10 mL solvent, 1equiv) during 10 min, the reaction mixture was slowly warmed to 25°C- 50°C and allowed to stir at 25°C - 50 °C. After the completion of the reaction (monitored by TLC), the reaction mixture was cooled to 0°C and quenched with ice cold water (20 mL). The reaction mixture was extracted with ethyl acetate (3 x 20 mL), and combined organic layer was washed with dilute HCl (25 mL), dilute sodium carbonate solution (25 mL) and finally with water. The organic layer was dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The crude product was purified on a silica gel column using hexane/EtOAc to get the pure product.
Characterization data for ketones

1-Phenylbutan-1-one.\(^1\)

Yellow oily liquid; Yield 90%; \(R_f\) (Hexane) 0.2. Prepared as shown in general experimental procedure (Reaction completion time: 2 h at 50 °C): \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.96 (d, \(J = 7.2\) Hz, 2H), 7.55 (t, \(J = 7.2\) Hz, 1H), 7.46 (t, \(J = 7.6\) Hz, 2H), 2.95 (t, \(J = 7.2\) Hz, 2H), 1.81 – 1.72 (m, 2H), 1.01 (t, \(J = 7.2\) Hz, 3H).

1-Phenylpentan-1-one.\(^1\)

Yellow oily liquid; Yield 90%; \(R_f\) (Hexane) 0.2. Prepared as shown in general experimental procedure (Reaction completion time 2 h at 50 °C):

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.96 (d, \(J = 7.2\) Hz, 2H), 7.55 (t, \(J = 7.2\) Hz, 1H), 7.46 (t, \(J = 7.6\) Hz, 2H), 2.97 (t, \(J = 7.2\) Hz, 2H), 1.76 – 1.69 (m, 2H), 1.46 – 1.37 (m, 2H), 0.95 (t, \(J = 7.2\) Hz, 3H).

6-Methyl-1-phenylheptan-1-one.\(^2\)

Colorless oily liquid; Yield 76%; \(R_f\) (Hexane) 0.3. Prepared as shown in general experimental procedure (Reaction completion time: 2 h at 50 °C):

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.96 (d, \(J = 7.2\) Hz, 2H), 7.55 (t, \(J = 7.2\) Hz, 1H), 7.46 (t, \(J = 7.6\) Hz, 2H), 2.97 (t, \(J = 7.2\) Hz, 2H), 1.76 – 1.68 (m, 2H), 1.60 – 1.50 (m, 1H), 1.42 – 1.34 (m, 2H), 1.25 – 1.19 (m, 2H), 0.87 (d, \(J = 6.4\) Hz, 6H).

1-Phenyldecan-1-one.\(^3\)

Colorless oily liquid; Yield 70%; \(R_f\) (Hexane) 0.3. Prepared as shown in general experimental procedure (Reaction completion time: 2 h at 50 °C):

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.96 (d, \(J = 7.2\) Hz, 2H), 7.55 (t, \(J = 7.2\) Hz, 1H), 7.46 (t, \(J = 7.6\) Hz, 2H), 2.96 (t, \(J = 7.2\) Hz, 2H), 1.77 – 1.70 (m, 2H), 1.34 – 1.27(m, 12H), 0.88 (t, \(J = 6.4\) Hz, 3H).

1-(4-ethylphenyl)propan-1-one.\(^4\)

Yellow oily liquid; Yield 91%; \(R_f\) (Hexane) 0.2. Prepared as shown in general experimental procedure (Reaction completion time: 6 h at RT):

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.89 (d, \(J = 8.0\) Hz, 2H), 7.27 (d, \(J = 8.0\) Hz, 2H), 2.98 (q, \(J = 7.2\) Hz, 2H), 2.70 (q, \(J = 7.6\) Hz, 2H), 1.27-1.19 (m, 6H).

1-(4-decylphenyl)propan-1-one. Colourless oily liquid; Yield 90%; \(R_f\) (Hexane) 0.2. Prepared as shown in general experimental procedure (Reaction completion time: 6 h at RT):

\textbf{IR}(Neat, cm\(^{-1}\)): 3356, 3030, 2926, 2854, 1805, 1686, 1607, 1569, 1460; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.88 (d, \(J = 8.0\) Hz, 2H), 7.25 (d, \(J = 8.0\) Hz, 2H), 2.98 (q, \(J = 7.2\) Hz, 2H), 2.65 (t, \(J = 7.6\) Hz, 2H), 1.64 – 1.58 (m, 2H), 1.31 – 1.25 (m, 13H), 1.22 (t, \(J = 7.2\) Hz, 3H), 0.88 (t, \(J = 6.8\) Hz, 3H). \(^1\)C NMR (100
MHz, CDCl₃ δ 200.52, 148.53, 134.66, 128.56, 128.10, 35.97, 31.88, 31.64, 31.11, 29.58, 29.54, 29.44, 29.30, 29.25, 22.66, 14.08, 8.33; HRESI-MS (m/z): Calculated for C₁₀H₃₀O (M⁺ + H): 275.2375, found (M⁺ + H): 275.2377.

1-(4-(tert-butyl)phenyl)propan-1-one. Yellow oily liquid; Yield 93%; Rᵣ (Hexane) 0.2. Prepared as shown in general experimental procedure (Reaction completion time: 6 h at RT): ¹H NMR (400 MHz, CDCl₃) δ 7.91 (d, J = 8.4 Hz, 2H), 7.47 (d, J = 8.4 Hz, 2H), 2.98 (q, J = 7.2 Hz, 2H), 1.34 (s, 9H), 1.22 (t, J = 7.2 Hz, 3H).

1-(5-methylthiophen-2-yl)propan-1-one. Brown oily liquid; Yield 88%; Rᵣ (Hexane) 0.2. Prepared as shown in general experimental procedure (Reaction completion time: 6 h at RT): IR (Neat, cm⁻¹): 3581, 3072, 2977, 2935, 2877, 1781, 1659, 1533, 1455; ¹H NMR (400 MHz, CDCl₃) δ 7.52 (d, J = 3.6 Hz, 1H), 6.78 (dd, J = 3.6, 0.8 Hz, 1H), 2.87 (q, J = 7.2 Hz, 2H), 2.53 (s, 3H), 1.21 (t, J = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 193.54, 149.14, 141.94, 132.07, 126.58, 32.07, 15.94, 8.67; HRESI-MS (m/z): Calculated for C₈H₁₀OS (M⁺ + Na): 177.0350, found (M⁺ + Na): 177.0351.

1-(5-bromothiophen-2-yl)propan-1-one. Brown oily liquid; Yield 81%; Rᵣ (Hexane) 0.2. Prepared as shown in general experimental procedure (Reaction completion time: 6 h at RT): ¹H NMR (400 MHz, CDCl₃) δ 7.44 (d, J = 4.0 Hz, 1H), 7.10 (d, J = 4.0 Hz, 1H), 2.80 (t, J = 7.2 Hz, 2H), 1.80 – 1.71 (m, 2H), 0.99 (t, J = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 192.20, 145.94, 131.67, 131.10, 122.19, 40.54, 18.04, 13.74; HRESI-MS (m/z): Calculated for C₈H₉BrOS (M⁺ + Na): 254.9455, found (M⁺ + Na): 254.9458.

1-(thiophen-2-yl)propan-1-one. Brown oily liquid; Yield 88%; Rᵣ (Hexane) 0.2. Prepared as shown in general experimental procedure (Reaction completion time: 6 h at RT): ¹H NMR (400 MHz, CDCl₃) δ 7.71 (d, J = 3.6 Hz, 1H), 7.61 (d, J = 4.8 Hz, 1H), 7.12 (t, J = 4.8 Hz, 1H), 2.94 (q, J = 7.2 Hz, 2H), 1.24 (t, J = 7.3 Hz, 3H).
Optimization of solvent

Optimization of catalyst

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<th>Isolated yield</th>
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<tr>
<td>3</td>
<td>EtoAc</td>
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<td>Toluene</td>
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<td>5</td>
<td>THF</td>
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<tr>
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<td>Ethanol</td>
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</tr>
<tr>
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<td>Trifluoroethanol</td>
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<td>Benzene</td>
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<tr>
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<td>TBAI (10)</td>
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<tr>
<td>2</td>
<td>KI (10)</td>
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</tr>
<tr>
<td>3</td>
<td>NaI (10)</td>
<td>60%</td>
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<td>[Bmim]I (10)</td>
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<td>5</td>
<td>I₂ (10)</td>
<td>NR</td>
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<td>6</td>
<td>NIS (10)</td>
<td>NR</td>
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<tr>
<td>7</td>
<td>NCS (10)</td>
<td>NR</td>
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<td>8</td>
<td>TEBA (10)</td>
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<tr>
<td>9</td>
<td>TBACl (10)</td>
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<td>NR</td>
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<tr>
<td>11</td>
<td>NBS</td>
<td>NR</td>
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[Bmim]I= 1-butyl-3-methylimidazolium iodide
NCS= N-Chlorosuccinimide
TEBA= Benzyltriethylammonium chloride
TBACl= Tetrabutylammonium chloride
TBAB= Tetrabutylammonium bromide
NBS= N-Bromosuccinimide
Optimization of oxidant

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<td>Ditertiarybutylperoxide</td>
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<td>7</td>
<td>m-CPBA</td>
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Optimization of equiv or mol% of 1a, 2a, TBAI and aq.TBHP

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<th>Ketone (equiv)</th>
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<th>TBAI (mol%)</th>
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<th>Isolated yield</th>
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<td>2</td>
<td>1</td>
<td>3</td>
<td>TBAI (10)</td>
<td>aq.TBHP (1 equiv)</td>
<td>64%</td>
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<td>2</td>
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<td>aq.TBHP (1 equiv)</td>
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<td>3</td>
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<td>3</td>
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Addition of acid and basic additives

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<td>1</td>
<td>PTSA (20 mol%)</td>
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<td>2</td>
<td>Acetic acid (100 mol%)</td>
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<tr>
<td>2</td>
<td>Piperidine (10 mol%)</td>
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<td>3</td>
<td>Triethylamine (100 mol%)</td>
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<td>Potassium carbonate (100 mol%)</td>
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Optimization of Temperature

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<tr>
<td>3</td>
<td>100 °C</td>
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Typical Experimental Procedure for CDC reactions

**Typical experimental procedure for CDC reaction of **NH**SI**: 
A mixture of propiophenone (0.5 mmol), *N*-hydroxysuccinimide (1.5 mmol), TBAI (0.05 mmol) and aq TBHP 70% (0.5mmol) in DMA (1 mL) were stirred at 80 °C temperature in a reaction vial (0.5h - 3h). After completion of the reaction (monitored by TLC, if reaction is incomplete terminate after 3h, continuation of more time leads to decreasing in yield), was added water (20 mL) and extracted with ethylacetate (3x50mL). The combined organic layer was dried over Na$_2$SO$_4$ and concentrated under reduced pressure. The crude product was purified on a silica gel column using hexane/ EtOAc to get the pure product.

**Typical experimental procedure for CDC reaction of **NH**PI**: 
A mixture of propiophenone (0.5 mmol), *N*-hydroxyphthalimide (1.5 mmol), TBAI (0.05 mmol) and TBHP (0.5mmol, in decane, 5.5 M) in DMA (1 mL) were stirred at 100 °C temperature in a reaction vial (0.5 - 2h). After completion of the reaction (monitored by TLC, if reaction is incomplete terminate after 3h, continuation of more time leads to decreasing in yield), was added water (20 mL) and extracted with ethylacetate (3x50mL). The combined organic layer was dried over Na$_2$SO$_4$ and concentrated under reduced pressure. The crude product was purified on a silica gel column using hexane/ EtOAc to get the pure product.

**Typical experimental procedure for CDC reaction of **HOB**t**: 
A mixture of propiophenone (0.5 mmol), hydroxybenzotriazole (1.5 mmol), TBAI (0.05mmol) and TBHP (0.5 mmol, in decane, 5.5 M) in DMSO (1 mL) were stirred at 80 °C in a reaction vial (1 - 3h). After completion of the reaction (monitored by TLC, if reaction is incomplete terminate after 3h, continuation of more time leads to decreasing in yield), was added water (20 mL) and extracted with ethylacetate (3x50mL). The combined organic layer was dried over Na$_2$SO$_4$ and concentrated under reduced pressure. The crude product was purified on a silica gel column using hexane/ EtOAc to get the pure product.
Characterization data for products

1-((1-oxo-1-phenylpropan-2-yl)oxy)pyrrolidine-2,5-dione (3a).

White solid; Yield 64% (79 mg); mp 104–106 °C; Rf (50% EtOAc/hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 1 h at 80 °C). IR (KBr, cm⁻¹): 3834, 3733, 3612, 2919, 2850, 1789, 1716, 1685, 1593, 1507; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.07 (d, J = 7.6 Hz, 2H), 7.59 (t, J = 7.6 Hz, 1H), 7.48 (t, J = 8.0 Hz, 2H), 5.68 (q, J = 6.8 Hz, 1H), 2.69 (s, 4H), 1.59 (d, J = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) 195.17, 171.14, 134.48, 133.65, 128.95, 128.55, 82.36, 25.29, 16.14; HRESI-MS (m/z): Calculated for C₁₃H₁₃NO₄ (M⁺ + Na): 270.0742, found (M⁺ + Na): 270.0742.

1-((1-oxo-1-phenylbutan-2-yl)oxy)pyrrolidine-2,5-dione (3b).

Pale yellow solid; Yield 63% (82 mg); mp 125–127 °C; Rf (50% EtOAc/hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 2h at 80 °C). IR (KBr, cm⁻¹): 2963, 1785, 1709, 1689, 1596, 1448; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.01 (d, J = 7.2 Hz, 2H), 7.59 (t, J = 7.2 Hz, 1H), 5.46 (t, J = 6.4 Hz, 1H), 2.66 (s, 4H), 2.07 – 1.99 (m, 2H), 1.08 (t, J = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) 195.64, 170.84, 134.97, 133.65, 128.78, 128.63, 88.10, 25.33, 24.76, 9.49; HRESI-MS (m/z): Calculated for C₁₄H₁₅NO₄ (M⁺ + Na): 284.0899, found (M⁺ + Na): 284.0897.

1-((1-oxo-1-phenylpentan-2-yl)oxy)pyrrolidine-2,5-dione (3c).

Pale yellow solid; Yield 66% (91 mg); mp 127–129 °C; Rf (30% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 2h at 80 °C). IR (Neat, cm⁻¹): 3789, 3775, 3697, 3664, 3640, 3463, 2962, 2847, 1754, 1726, 1712, 1597; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.01 (d, J = 8.0 Hz, 2H), 7.60 (t, J = 7.2 Hz, 1H), 7.48 (t, J = 7.6 Hz, 2H), 5.44 (t, J = 6.8 Hz, 1H), 2.65 (s, 4H), 2.03 – 1.87 (m, 2H), 1.61 – 1.52 (m, 2H), 0.96 (t, J = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) 195.76, 170.83, 134.93, 133.58, 128.71, 128.59, 86.75, 33.45, 25.27, 18.41, 13.67; HRESI-MS (m/z): Calculated for C₁₅H₁₇NO₄ (M⁺ + Na): 298.1055, found (M⁺ + Na): 298.1057.
1-((6-methyl-1-oxo-1-phenylheptan-2-yl)oxy)pyrrolidine-2,5-dione (3d).

White solid; Yield 60% (96 mg); mp 135–137 °C; Rf (30% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 3h at 80 °C). IR (KBr, cm⁻¹): 2955, 2276, 1784, 1713, 1595, 1454; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.01 (d, J = 8.0 Hz, 2H), 7.60 (t, J = 7.2 Hz, 1H), 7.48 (t, J = 7.6 Hz, 2H), 5.54 (t, J = 6.4 Hz, 1H), 2.65 (s, 4H), 2.02 – 1.90 (m, 2H), 1.60 – 1.48 (m, 3H), 1.28 – 1.13 (m, 2H), 0.85 (dd, J = 6.4, 1.6 Hz, 6H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) 195.82, 170.79, 134.93, 133.63, 128.78, 128.64, 86.99, 38.42, 31.70, 27.63, 25.32, 22.88, 22.41; HRESI-MS (m/z): Calculated for C₁₈H₂₃NO₄ (M⁺ + Na): 340.1525, found (M⁺ + Na): 340.1522.

1-((1-oxo-1-phenyldecan-2-yl)oxy)pyrrolidine-2,5-dione (3e).

Pale yellow solid; Yield 61% (105 mg); mp 113–115 °C; Rf (30% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 3h at 80 °C). IR (KBr, cm⁻¹): 3726, 2920, 1784, 1724, 1532, 1446; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.01 (d, J = 7.2 Hz, 2H), 7.60 (t, J = 7.2 Hz, 1H), 7.48 (t, J = 7.6 Hz, 2H), 5.53 (t, J = 6.4 Hz, 1H), 2.64 (s, 4H), 2.02 – 1.91 (m, 2H), 1.54 – 1.48 (m, 2H), 1.35 – 1.23 (m, 10H), 0.86 (t, J = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) 195.81, 170.80, 134.90, 133.60, 128.75, 128.61, 86.98, 31.68, 31.47, 29.19, 29.13, 29.03, 25.30, 24.98, 22.52, 13.99; HRESI-MS (m/z): Calculated for C₂₀H₂₇NO₄ (M⁺ + Na): 368.1838, found (M⁺ + Na): 368.1835.

1-((1-(4-ethylphenyl)-1-oxopropan-2-yl)oxy)pyrrolidine-2,5-dione (3f).

Pale yellow solid; Yield 62% (86 mg); mp 75–77 °C; Rf (30% EtOAc/hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 3h at 80 °C). IR (KBr, cm⁻¹): 3936, 3492, 3063, 2968, 2919, 2876, 2851, 1790, 1728, 1688, 1606; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.01 (d, J = 8.4 Hz, 2H), 7.30 (d, J = 8.4 Hz, 2H), 5.71 (q, J = 6.8 Hz, 1H), 2.74 – 2.70 (m, 2H), 1.60 (d, J = 6.8 Hz, 3H), 1.26 (t, J = 7.6 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) 194.81, 171.06, 150.89, 132.24, 129.26, 128.17, 82.34, 28.95, 25.36, 16.34, 15.05; HRESI-MS (m/z): Calculated for C₁₅H₁₇NO₄ (M⁺ + Na): 298.1055, found (M⁺ + Na): 298.1055.

1-((1-(4-decylphenyl)-1-oxopropan-2-yl)oxy)pyrrolidine-2,5-dione (3g).

Pale yellow solid; Yield 61% (118 mg); mp 67–69 °C; Rf (30% EtOAc/hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 3h at 3fO

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80 °C). IR (KBr, cm⁻¹): 3489, 3359, 3068, 2921, 2120, 2016, 1779, 1715, 1684, 1603; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.00 (d, _J_ = 8.0 Hz, 2H), 7.28 (d, _J_ = 7.6 Hz, 2H), 5.71 (q, _J_ = 6.8 Hz, 1H), 2.69 (s, 4H), 2.65 (t, _J_ = 7.6 Hz, 2H), 1.61 – 1.59 (m, 5H), 1.31 – 1.25 (m, 15H), 0.88 (t, _J_ = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) 194.77, 171.05, 149.70, 132.22, 129.16, 128.70, 82.28, 36.01, 31.82, 30.97, 29.48, 29.38, 29.25, 29.20, 25.36, 22.61, 16.33, 14.06; HRESI-MS (m/z): Calculated for C₂₃H₃₃NO₄ (M⁺ + Na): 410.2307, found (M⁺ + Na): 410.2307.

1-((1-oxo-1,3-diphenylpropan-2-yl)oxy)pyrrolidine-2,5-dione (3h).

White solid; Yield 68% (110 mg); mp 131–133 °C; R₇ (30% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 2h at 80 °C). IR (KBr, cm⁻¹): 3486, 3064, 3033, 2990, 2942, 2920, 2225, 2104, 1918, 1782, 1715, 1690, 1594; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.06 (d, _J_ = 7.6 Hz, 2H), 7.57 (t, _J_ = 7.6 Hz, 1H), 7.45 (t, _J_ = 7.6 Hz, 2H), 7.29 – 7.23 (m, 4H), 7.22 – 7.18 (m, 1H), 6.03 (dd, _J_ = 8.0, 5.2 Hz, 1H), 3.43 (dd, _J_ = 15.2, 5.2 Hz, 1H), 3.29 (dd, _J_ = 15.2, 8.0 Hz, 1H), 2.38 (s, 4H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) 194.39, 170.80, 135.75, 135.07, 133.73, 129.07, 128.80, 128.59, 128.51, 126.85, 84.98, 37.01, 25.20; HRESI-MS (m/z): Calculated for C₁₉H₁₇NO₄ (M⁺ + Na): 346.1055, found (M⁺ + Na): 346.1059.

1-((3-(naphthalen-1-yl)-1-oxo-1-phenylpropan-2-yl)oxy)pyrrolidine-2,5-dione (3i).

Pale yellow oily; Yield 67% (125 mg); R₇ (30% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 2h at 80 °C). IR (Neat, cm⁻¹): 3059, 2359, 1787, 1725, 1690, 1596, 1510, 1448, 1364; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.06 (d, _J_ = 8.4 Hz, 1H), 7.87 (d, _J_ = 7.6 Hz, 2H), 7.81 (d, _J_ = 8.0 Hz, 1H), 7.67 (d, _J_ = 8.0 Hz, 1H), 7.56 – 7.52 (m, 1H), 7.50 – 7.44 (m, 2H), 7.35 – 7.25 (m, 4H), 6.07 (t, _J_ = 6.8 Hz, 1H), 3.88 (dd, _J_ = 14.8, 6.8 Hz, 1H), 3.78 (dd, _J_ = 14.8, 6.8 Hz, 1H), 2.40 (s, 4H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) 195.17, 170.69, 135.25, 133.69, 131.60, 131.05, 128.80, 128.43, 127.77, 127.70, 126.42, 125.69, 125.27, 123.20, 84.77, 34.28, 25.13; HRESI-MS (m/z): Calculated for C₂₃H₁₉NO₄ (M⁺ + Na): 396.1212, found (M⁺ + Na): 396.1216.

1-((3-(4-chlorophenyl)-1-oxo-1-phenylpropan-2-yl)oxy)pyrrolidine-2,5-dione (3j).

White solid; Yield 64% (115 mg); mp 139–141 °C; R₇ (30% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 3h at 80 °C). IR (KBr, cm⁻¹): 3749, 3489, 2954, 2562, 2118, 1778, 1716, 1685, 1595, 1489; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.02 (d, _J_ = 7.6 Hz, 2H), 7.59 (t, _J_ = 7.2 Hz,
1H), 7.47 (t, J = 7.6 Hz, 2H), 7.27 – 7.18 (m, 4H), 5.95 (t, J = 6.4 Hz, 1H), 3.38 (dd, J = 14.8, 5.2 Hz, 1H), 3.27 (dd, J = 14.8, 7.6 Hz, 1H), 2.50 (s, 4H); 13C NMR (100 MHz, CDCl₃) δ (ppm) 194.25, 170.77, 134.95, 133.95, 133.86, 132.74, 130.41, 128.90, 128.67, 128.56, 85.03, 36.34, 25.23; HRESI-MS (m/z): Calculated for C₁₉H₁₆ClNO₄ (M⁺ + Na): 380.0663, found (M⁺ + Na): 380.0666.

1-((1,3-bis(4-fluorophenyl)-1-oxopropan-2-yl)oxy)pyrrolidine-2,5-dione (3k).

Pale yellow solid; Yield 70% (126 mg); mp 79–82 °C; Rₚ (30% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 2h at 80 °C): IR(KBr, cm⁻¹): 3789, 3698, 3661, 3638, 3020, 2360, 1726, 1597; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.08 (dd, J = 8.4, 5.2 Hz, 2H), 7.28 – 7.18 (m, 2H), 7.12 (t, J = 8.4 Hz, 2H), 6.94 (t, J = 8.4 Hz, 2H), 5.81 (t, J = 6.4 Hz, 1H), 3.37 (dd, J = 14.8, 6.0 Hz, 1H), 3.26 (dd, J = 14.8, 7.2 Hz, 1H), 2.51 (s, 4H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) 192.82, 170.81, 165.74 (d, Jₐᵣₑ₉₉(C-F) = 255 Hz), 161.73 (d, Jₐᵣₑ₉₉(C-F) = 244 Hz), 131.90 (d, Jₐᵣₑ₉₉(C-F) = 10 Hz), 131.35 (d, Jₐᵣₑ₉₉(C-F) = 3 Hz), 131.01 (d, Jₐᵣₑ₉₉(C-F) = 4 Hz), 130.54 (d, Jₐᵣₑ₉₉(C-F) = 8 Hz), 115.80 (d, Jₐᵣₑ₉₉(C-F) = 22 Hz), 115.33 (d, Jₐᵣₑ₉₉(C-F) = 21 Hz), 85.78, 36.20, 25.23; ¹⁹F NMR (376 MHz, CDCl₃) δ (ppm) -103.20, -115.38; HRESI-MS (m/z): Calculated for C₁₉H₁₁F₂NO₄ (M⁺ + Na): 382.0867, found (M⁺ + Na): 382.0863.

1-((1-(4-(tert-butyl)phenyl)-1-oxopropan-2-yl)oxy)pyrrolidine-2,5-dione (3l).

Pale yellow solid; Yield 68% (103 mg); mp 125–128 °C; Rₚ (30% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 3h at 80 °C): IR(KBr, cm⁻¹): 3904, 3821, 3805, 3770, 3502, 2964, 2871, 1790, 1728, 1694, 1605; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.03 (d, J = 8.0 Hz, 2H), 7.50 (d, J = 8.4 Hz, 2H), 5.73 (q, J = 6.8 Hz, 1H), 2.69 (s, 4H), 1.61 (d, J = 6.8 Hz, 3H), 1.34 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) 194.76, 171.05, 157.66, 131.95, 129.03, 125.66, 82.34, 35.18, 30.98, 25.39, 16.35; HRESI-MS (m/z): Calculated for C₁₇H₂₁NO₄ (M⁺ + Na): 326.1368, found (M⁺ + Na): 326.1368.

1-((1-(3-fluorophenyl)-1-oxopropan-2-yl)oxy)pyrrolidine-2,5-dione (3m).

Yellow oily; Yield 73% (97 mg); Rₚ (50% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 1h at 80 °C): IR(Neat, cm⁻¹): 3071, 2920, 2851, 2123, 1790, 1722, 1587, 1486, 1446; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 7.92 (d, J = 7.6 Hz, 1H), 7.83 (d, J = 9.2 Hz, 1H), 7.50 – 7.45 (m, 1H), 7.33 – 7.28 (m, 1H), 5.58 (q, J = 6.8 Hz, 1H), 2.73 (s, 4H), 1.61 (d, J = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) 193.90, 171.25, 162.42 (d, Jₐᵣₑ₉₉(C-F) = 246 Hz), 136.29 (d, Jₐᵣₑ₉₉(C-F) = 6 Hz), 130.17 (d, Jₐᵣₑ₉₉(C-F) = 8 Hz), 124.79 (d, Jₐᵣₑ₉₉(C-F) = 3 Hz), 115.33 (d, Jₐᵣₑ₉₉(C-F) = 21 Hz), 85.78, 129.03, 125.66, 82.34, 35.18, 30.98, 25.39, 16.35; HRESI-MS (m/z): Calculated for C₁₉H₁₆ClNO₄ (M⁺ + Na): 380.0663, found (M⁺ + Na): 380.0666.

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120.55 (d, $J_{C-F} = 21$ Hz), 115.69 (d, $J_{C-F} = 22$ Hz), 82.70, 25.20, 15.81; $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ (ppm) -111.46; HRESI-MS ($m/z$): Calculated for C$_{13}$H$_{12}$FNO$_4$ (M$^+$ + Na): 288.0648, found (M$^+$ + Na): 288.0645.

1-((1-(3-chlorophenyl)-1-oxopropan-2-yl)oxy)pyrrolidine-2,5-dione (3n).

Yellow oily; Yield 65% (92 mg); (1g, 65% (1087 mg); $R_f$ (50% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 1h at 80 °C): IR (Neat, cm$^{-1}$): 3398, 2939, 1791, 1727, 1570, 1427, 1374; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm) 8.11 (s, 1H), 8.01 (d, $J = 7.6$ Hz, 1H), 7.56 (d, $J = 8.0$ Hz, 1H), 7.43 (t, $J = 8.0$ Hz, 1H), 5.55 (q, $J = 6.8$ Hz, 1H), 2.72 (s, 4H), 1.59 (d, $J = 6.4$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm) 193.95, 171.07, 135.98, 134.80, 133.53, 129.89, 129.16, 127.28, 82.92, 25.32, 15.91; HRESI-MS ($m/z$): Calculated for C$_{13}$H$_{12}$ClNO$_4$ (M$^+$ + Na): 304.0353, found (M$^+$ + Na): 304.0350.

1-((1-(4-chlorophenyl)-1-oxopropan-2-yl)oxy)pyrrolidine-2,5-dione (3o).

Yellow solid; Yield 67% (95 mg); (1g, 67% (1128 mg); mp 88–91 °C; $R_f$ (50% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 3h at 80 °C): IR (KBr, cm$^{-1}$): 3735, 3368, 2931, 1785, 1725, 1686, 1589, 1375; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm) 8.07 (d, $J = 8.4$ Hz, 2H), 7.44 (d, $J = 8.4$ Hz, 2H), 5.55 (q, $J = 6.8$ Hz, 1H), 2.72 (s, 4H), 1.57 (d, $J = 6.8$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm) 193.92, 171.16, 140.00, 132.69, 130.54, 128.79, 82.78, 25.26, 15.84; HRESI-MS ($m/z$): Calculated for C$_{13}$H$_{12}$ClNO$_4$ (M$^+$ + Na): 304.0353, found (M$^+$ + Na): 304.0353.

1-((1-(3-bromophenyl)-1-oxopropan-2-yl)oxy)pyrrolidine-2,5-dione (3p).

Yellow oily; Yield 63% (103 mg); $R_f$ (50% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 2h at 80 °C): IR (Neat, cm$^{-1}$): 3904, 3495, 3066, 2988, 2941, 2362, 2343, 1787, 1727, 1699, 1566; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ (ppm) 8.28 (s, 1H), 8.07 (d, $J = 8.0$ Hz, 1H), 7.72 (d, $J = 7.6$ Hz, 1H), 7.37 (t, $J = 8.0$ Hz, 1H), 5.56 (q, $J = 6.8$ Hz, 1H), 2.73 (s, 4H), 1.60 (d, $J = 6.8$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ (ppm) 193.90, 171.04, 136.52, 136.22, 132.16, 130.18, 127.78, 122.86, 82.96, 25.36, 15.95; HRESI-MS ($m/z$): Calculated for C$_{13}$H$_{12}$BrNO$_4$ (M$^+$ + Na): 347.9847, found (M$^+$ + Na): 347.9842.
1-((1-(4-bromophenyl)-1-oxopropan-2-yl)oxy)pyrrolidine-2,5-dione (3q).

White solid; Yield 60% (98 mg); mp 98–102 °C; \( R_f \) (50% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 3h at 80 °C): \( \text{IR (KBr, cm}^{-1}) \): 3856, 3747, 3505, 3063, 2990, 2925, 2853, 2558, 1717, 1585, 1430; \( \text{H NMR (400 MHz, CDCl}_3 \text{)} \delta \text{(ppm)} \): 8.01 (d, \( J = 8.8 \text{ Hz, 2H} \)), 7.63 (d, \( J = 8.4 \text{ Hz, 2H} \)), 5.55 (q, \( J = 6.8 \text{ Hz, 1H} \)), 2.72 (s, 4H), 1.59 (d, \( J = 6.8 \text{ Hz, 3H} \)); \( \text{C NMR (100 MHz, CDCl}_3 \text{)} \delta \text{(ppm)} \): 194.25, 171.00, 133.26, 131.95, 130.79, 129.06, 83.06, 125.38, 15.96; \( \text{HRESI-MS (m/z): Calculated for C}_{13}\text{H}_{12}\text{BrNO}_4 \text{(M}^+ + \text{Na): 347.9847, found (M}^+ + \text{Na): 347.9847.} \)

1-((1-oxo-1-(3-(trifluoromethyl)phenyl)propan-2-yl)oxy)pyrrolidine-2,5-dione (3r).

Yellow oily; Yield 64% (99 mg); \( R_f \) (50% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 3 h at 80 °C): \( \text{IR (Neat, cm}^{-1}) \): 3786, 3658, 2921, 2171, 1790, 1724, 1611, 1435; \( \text{H NMR (400 MHz, CDCl}_3 \text{)} \delta \text{(ppm)} \): 8.45 (s, 1H), 8.36 (d, \( J = 8.0 \text{ Hz, 1H} \)), 7.85 (d, \( J = 7.6 \text{ Hz, 1H} \)), 7.64 (t, \( J = 8.0 \text{ Hz, 1H} \)), 5.58 (q, \( J = 6.8 \text{ Hz, 1H} \)), 2.74 (s, 4H), 1.62 (d, \( J = 6.4 \text{ Hz, 3H} \)); \( \text{C NMR (100 MHz, CDCl}_3 \text{)} \delta \text{(ppm)} \): 193.90, 171.12, 134.98, 133.04 – 132.43 (m), 130.99 (q, \( J_{C-F} = 32 \text{ Hz} \)), 129.87 (q, \( J_{C-F} = 3.6 \text{ Hz} \)), 129.18 – 129.02 (m), 126.08 (q, \( J_{C-F} = 3.6 \text{ Hz} \)), 123.49 (q, \( J_{C-F} = 270 \text{ Hz} \)), 83.1, 25.26, 15.63; \( \text{F NMR (376 MHz, CDCl}_3 \text{)} \delta \text{(ppm)} \): -62.81; \( \text{HRESI-MS (m/z): Calculated for C}_{14}\text{H}_{12}\text{F}_3\text{NO}_4 \text{(M}^+ + \text{Na): 338.0616, found (M}^+ + \text{Na): 338.0619.} \)

1-((1-oxo-1-(5-methylthiophen-2-yl)-1-oxopropan-2-yl)oxy)pyrrolidine-2,5-dione (3s).

Brown solid; Yield 53% (71 mg); mp 113–116 °C; \( R_f \) (50% EtOAc/hexane) 0.4. Prepared as shown in general experimental procedure (Reaction time: 3h at 80 °C): \( \text{IR (KBr, cm}^{-1}) \): 3734, 2946, 2357, 1786, 1716, 1656, 1541, 1454; \( \text{H NMR (400 MHz, CDCl}_3 \text{)} \delta \text{(ppm)} \): 7.94 (d, \( J = 4.0 \text{ Hz, 1H} \)), 6.84 (d, \( J = 3.6 \text{ Hz, 1H} \)), 5.39 (q, \( J = 6.8 \text{ Hz, 1H} \)), 2.71 (s, 4H), 2.55 (s, 3H), 1.61 (d, \( J = 6.8 \text{ Hz, 3H} \)); \( \text{C NMR (100 MHz, CDCl}_3 \text{)} \delta \text{(ppm)} \): 187.62, 171.00, 151.51, 138.94, 135.05, 127.19, 83.52, 25.36, 16.70, 16.03; \( \text{HRESI-MS (m/z): Calculated for C}_{12}\text{H}_{13}\text{NO}_4 \text{S (M}^+ + \text{Na): 290.0463, found (M}^+ + \text{Na): 290.0465.} \)

1-((1-(5-bromothiophen-2-yl)-1-oxopropan-2-yl)oxy)pyrrolidine-2,5-dione (3t).

Brown solid; Yield 50% (83 mg); mp 146–148 °C; \( R_f \) (50% EtOAc/hexane) 0.4. Prepared as shown in general experimental procedure (Reaction time:3h
at 80 °C): \textbf{IR} (KBr, cm$^{-1}$): 3854, 3736, 3619, 2919, 2851, 1786, 1711, 1663, 1528, 1406; \textbf{H NMR} (400 MHz, CDCl$_3$) \(\delta\) (ppm) 7.96 (d, \(J = 3.6\) Hz, 1H), 7.15 (d, \(J = 3.6\) Hz, 1H), 5.28 (q, \(J = 6.8\) Hz, 1H), 2.74 (s, 4H), 1.61 (d, \(J = 6.8\) Hz, 3H); \textbf{C NMR} (100 MHz, CDCl$_3$) \(\delta\) (ppm) 187.12, 170.97, 142.34, 135.04, 131.56, 124.48, 84.14, 25.37, 16.47; \textbf{HRESI-MS} (m/z): Calculated for C$_{11}$H$_{10}$BrNO$_4$S (M$^+$ + Na): 353.9412, found (M$^+$ + Na): 353.9410.

\textbf{1-}((1-(5-bromothiophen-2-yl)-1-oxobutan-2-yl)oxy)pyrrolidine-2,5-dione (3u).

Brown solid; Yield 58% (100 mg); mp 102–104 °C; \(R_f\) (50% EtOAc/hexane) 0.4. Prepared as shown in general experimental procedure (Reaction time: 3h at 80 °C): \textbf{IR} (KBr, cm$^{-1}$): 3856, 3747, 3619, 2919, 2851, 1772, 1718, 1731, 1527, 1399; \textbf{H NMR} (400 MHz, CDCl$_3$) \(\delta\) (ppm) 7.81 (d, \(J = 4.0\) Hz, 1H), 7.14 (d, \(J = 4.0\) Hz, 1H), 4.98 (t, \(J = 6.8\) Hz, 1H), 2.70 (s, 4H), 2.12 – 1.98 (m, 2H), 1.08 (t, \(J = 6.8\) Hz, 3H); \textbf{C NMR} (100 MHz, CDCl$_3$) \(\delta\) (ppm) 187.80, 170.67, 142.65, 134.55, 131.47, 124.29, 90.14, 25.54, 25.25, 9.51; \textbf{HRESI-MS} (m/z) Calculated for C$_{12}$H$_{12}$BrNO$_4$S (M$^+$ + Na): 367.9568, found (M$^+$ + Na): 367.9569.

\textbf{1-}((1-(5-methylfuran-2-yl)-1-oxopropan-2-yl)oxy)pyrrolidine-2,5-dione (3v).

White solid; Yield 44% (55 mg); mp 140–142 °C; \(R_f\) (50% EtOAc/hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 2 h at 80 °C). \textbf{IR} (KBr, cm$^{-1}$) 3485, 3118, 2988, 2926, 1714, 1659, 1514; \textbf{H NMR} (400 MHz, CDCl$_3$) \(\delta\) 7.51 (d, \(J = 3.6\) Hz, 1H), 6.22 (d, \(J = 3.6\) Hz, 1H), 5.33 (q, \(J = 6.8\) Hz, 1H), 2.72 (s, 4H), 2.41 (s, 3H), 1.59 (d, \(J = 6.8\) Hz, 3H); \textbf{C NMR} (100 MHz, CDCl$_3$) \(\delta\) 182.76, 171.00, 159.15, 149.00, 122.66, 109.36, 82.79, 25.28, 16.41, 13.96; \textbf{HRESI-MS} (m/z) Calculated for C$_{12}$H$_{13}$NO$_5$ (M$^+$ + Na) 274.0691, found (M$^+$ + Na) 274.0692.

\textbf{1-}((1-(isoquinolin-1-yl)-1-oxobutan-2-yl)oxy)pyrrolidine-2,5-dione (3w).

White solid; Yield 42% (64 mg); mp 161–163 °C; \(R_f\) (50% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 2 h at 80 °C). \textbf{IR} (KBr, cm$^{-1}$) 2961, 2925, 2852, 2111, 1784, 1701, 1578; \textbf{H NMR} (400 MHz, CDCl$_3$) \(\delta\) 9.05 (d, \(J = 7.2\) Hz, 1H), 8.54 (d, \(J = 5.2\) Hz, 1H), 7.89 – 7.84 (m, 2H), 7.77 – 7.71 (m, 2H), 6.19 (t, \(J = 6.2\) Hz, 1H), 2.70 – 2.54 (m, 4H), 2.17 – 2.07 (m, 2H), 1.16 (t, \(J = 7.6\) Hz, 3H); \textbf{C NMR} (100 MHz, CDCl$_3$) \(\delta\) 198.31, 170.79, 151.01, 140.80, 136.91, 130.62, 129.61, 126.97, 126.55, 126.41, 125.22, 87.27, 25.31, 24.68, 9.63; \textbf{HRESI-MS} (m/z) Calculated for C$_{17}$H$_{16}$N$_2$O$_4$ (M$^+$ + Na) 335.1008, found (M$^+$ + Na) 335.1010.
1-((1-(naphthalen-2-yl)-1-oxo-3-phenylpropan-2-yl)oxy)pyrrolidine-2,5-dione (3x).

White solid; Yield 66% (125 mg); mp 128–130 °C; R₇ (50% EtOAc/hexane) 0.5. Prepared as shown in general experimental procedure (Reaction time: 2 h at 80 °C). **IR** (KBr, cm⁻¹) 3617, 3539, 3063, 3023, 2949, 2918, 2849, 2173, 1706, 1623; **¹H NMR** (400 MHz, CDCl₃) δ 8.70 (s, 1H), 8.05 (d, J = 8.4 Hz, 1H), 7.95 (d, J = 8.0 Hz, 1H), 7.85 (t, J = 8.4 Hz, 2H), 7.56 (t, J = 6.7 Hz, 1H), 7.53 (t, J = 6.7 Hz, 1H), 7.27 – 7.24 (m, 4H), 7.20 – 7.18 (m, 1H), 6.15 (t, J = 6.2 Hz, 1H), 3.49 (dd, J = 14.8, 5.2 Hz, 1H), 3.35 (dd, J = 14.8, 8.0 Hz, 1H), 2.36 (s, 4H); **¹³C NMR** (100 MHz, CDCl₃) δ 194.15, 170.86, 135.87, 135.73, 132.36, 132.30, 131.54, 129.83, 128.89, 128.86, 128.54, 128.45, 127.67, 126.87, 126.79, 124.24, 85.15, 37.08, 25.22; **HRESI-MS** (m/z) Calculated for C₂₃H₁₉NO₄ (M⁺ + Na) 396.1212, found (M⁺ + Na) 396.1213.

1-(1-cyclopropyl-2-oxo-2-phenylethoxy)pyrrolidine-2,5-dione (3y).

White solid; Yield 56% (76 mg); mp 110–112 °C; R₇ (50% EtOAc/hexane) 0.5. Prepared as shown in general experimental procedure (Reaction time: 2 h at 80 °C). **IR** (KBr, cm⁻¹) 3481, 3070, 3009, 2918, 2853, 1817, 1777, 1711, 1595; **¹H NMR** (400 MHz, CDCl₃) δ 8.19 (d, J = 7.6 Hz, 2H), 7.60 (t, J = 7.2 Hz, 1H), 7.49 (t, J = 7.6 Hz, 2H), 4.65 (d, J = 6.2 Hz, 1H), 2.70 (s, 4H), 1.46 – 1.39 (m, 1H), 0.81 – 0.73 (m, 1H), 0.68 – 0.61 (m, 1H), 0.60 – 0.53 (m, 1H), 0.39 – 0.32 (m, 1H); **¹³C NMR** (100 MHz, CDCl₃) δ 194.04, 171.03, 134.86, 133.66, 129.47, 128.54, 91.55, 25.34, 11.46, 4.79, 2.05; **HRESI-MS** (m/z) Calculated for C₁₅H₁₅NO₄ (M⁺ + Na) 296.0889, found (M⁺ + Na) 296.0897.

2-((1-oxo-1-phenylpropan-2-yl)oxy)isoindoline-1,3-dione (4a).

White Solid; Yield 64% (95 mg); mp 86-88 °C; R₇ (50% EtOAc/Hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 20min at 100 °C): **IR** (KBr, cm⁻¹): 3834, 3733, 3612, 2919, 2850, 1852, 1786, 1728, 1677, 1594; **¹H NMR** (400 MHz, CDCl₃) δ 8.16 (d, J = 7.6 Hz, 2H), 7.84 – 7.81 (m, 2H), 7.76 – 7.74 (m, 2H), 6.19 (d, J = 6.4 Hz, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ 195.28, 163.61, 134.66, 134.60, 133.71, 129.16, 128.73, 128.62, 123.62, 83.53, 16.14; **HRESI-MS** (m/z): Calculated for C₁₇H₁₃NO₄ (M⁺ + Na): 318.0742, found (M⁺ + Na): 318.0743.

2-((1-oxo-1-phenylpentan-2-yl)oxy)isoindoline-1,3-dione (4b).

Yellow oily; Yield 62% (100 mg); R₇ (20% EtOAc/hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 1h at 100 °C): **IR** (Neat, cm⁻¹): 3512,
3065, 2963, 2934, 2875, 1791, 1731, 1694, 1597, 1580, 1467; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) δ (ppm) 8.07 (d, J = 8.0 Hz, 2H), 7.80 – 7.78 (m, 2H), 7.75 – 7.71 (m, 2H), 7.59 (t, J = 7.2 Hz, 1H), 7.48 (t, J = 7.6 Hz, 2H), 5.62 – 5.59 (m, 1H), 2.13 – 1.96 (m, 2H), 1.68 – 1.59 (m, 2H), 1.00 (t, J = 7.2 Hz, 3H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}) δ (ppm) 195.85, 163.30, 134.84, 134.46, 133.56, 128.80, 128.63, 128.55, 123.46, 87.95, 33.38, 18.45, 13.70; HRESI-MS (m/z): Calculated for C\textsubscript{19}H\textsubscript{17}NO\textsubscript{4} (M\textsuperscript{+} + Na): 346.1055, found (M\textsuperscript{+} + Na): 346.1051

2-((1-(4-ethylphenyl)-1-oxopropan-2-yl)oxy)isoindoline-1,3-dione (4c).

Yellow Solid; Yield 60% (97 mg); mp 101–103 °C; R\textsubscript{f} (20% EtOAc/hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 1 h at 100°C): IR (KBr, cm\textsuperscript{-1}): 3496, 3059, 2967, 2920, 2851, 1791, 1735, 1687, 1606; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) δ (ppm) 8.09 (d, J = 8.0 Hz, 2H), 7.84 – 7.82 (m, 2H), 7.77 – 7.73 (m, 2H), 7.32 (d, J = 8.0 Hz, 2H), 5.75 (q, J = 6.8 Hz, 1H), 2.71 (q, J = 7.6 Hz, 2H), 1.68 (d, J = 6.8 Hz, 3H), 1.26 (t, J = 7.6 Hz, 3H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}) δ (ppm) 194.83, 163.60, 150.81, 134.56, 132.32, 129.33, 128.71, 128.14, 123.58, 83.39, 28.93, 16.22, 15.01; HRESI-MS (m/z): Calculated for C\textsubscript{19}H\textsubscript{17}NO\textsubscript{4} (M\textsuperscript{+} + Na): 346.1055, found (M\textsuperscript{+} + Na): 346.1053.

2-((1-oxo-1,3-diphenylpropan-2-yl)oxy)isoindoline-1,3-dione (4d).

White Solid; Yield 52% (97 mg); mp 103–105 °C; R\textsubscript{f} (10% EtOAc/hexane) 0.1. Prepared as shown in general experimental procedure (Reaction time: 1 h at 100°C): IR (KBr, cm\textsuperscript{-1}): 3854, 3751, 3524, 3066, 2964, 2871, 1792, 1772, 1733, 1699, 1605; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) δ (ppm) 8.04 (d, J = 7.6 Hz, 2H), 7.76 – 7.73 (m, 2H), 7.71 – 7.68 (m, 2H), 7.55 (t, J = 7.6 Hz, 1H), 7.43 (t, J = 8.0 Hz, 2H), 7.27 (d, J = 7.2 Hz, 2H), 7.21 (t, J = 7.6 Hz, 2H), 7.12 (t, J = 7.2 Hz, 1H), 5.96 (t, J = 6.4 Hz, 1H), 3.47 (dd, J = 14.4, 6.4 Hz, 1H), 3.39 (dd, J = 14.4, 6.8 Hz, 1H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}) δ (ppm) 194.76, 163.31, 135.30, 135.17, 134.49, 133.67, 129.18, 128.96, 128.59, 128.57, 128.43, 126.76, 123.49, 86.96, 37.04; HRESI-MS (m/z): Calculated for C\textsubscript{23}H\textsubscript{17}NO\textsubscript{4} (M\textsuperscript{+} + Na): 394.1055, found (M\textsuperscript{+} + Na): 394.1052.

2-((1-(4-(tert-butyl)phenyl)-1-oxopropan-2-yl)oxy)isoindoline-1,3-dione (4e).

Yellow oily; Yield 65% (114mg); R\textsubscript{f} (20% EtOAc/hexane) 0.3. Prepared as shown in general experimental procedure (Reaction time: 1 h at 100°C): IR (KBr, cm\textsuperscript{-1}): 3854, 3751, 3524, 3066, 2964, 2871, 1792, 1772, 1733, 1699, 1605; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) δ (ppm) 8.10 (d, J = 8.0 Hz, 2H), 7.84 – 7.81 (m, 2H), 7.77 – 7.73 (m, 2H), 7.51 (d, J = 8.4 Hz, 2H), 5.76 (q, J = 6.8 Hz, 2H); 13C NMR (100 MHz, CDCl\textsubscript{3}) δ (ppm) 194.76, 163.31, 135.30, 135.17, 134.49, 133.67, 129.18, 128.96, 128.59, 128.57, 128.43, 126.76, 123.49, 86.96, 37.04; HRESI-MS (m/z): Calculated for C\textsubscript{23}H\textsubscript{17}NO\textsubscript{4} (M\textsuperscript{+} + Na): 394.1055, found (M\textsuperscript{+} + Na): 394.1052.
Hz, 1H), 1.68 (d, J = 6.8 Hz, 3H), 1.34 (s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm) 194.83, 163.66, 157.61, 134.59, 132.11, 129.15, 128.81, 125.67, 123.64, 83.44, 35.19, 31.00, 16.24; HRESI-MS (m/z): Calculated for C$_{21}$H$_{21}$NO$_4$ (M$^+$ + Na): 374.1368, found (M$^+$ + Na): 374.1370.

2-((1-(3-fluorophenyl)-1-oxopropan-2-yl)oxy)isoindoline-1,3-dione (4f).

White Solid; Yield 65% (102 mg); mp 120–122 °C; R$_f$ (20% EtOAc/hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 20min at 100 °C): IR (KBr, cm$^{-1}$): 3508, 3074, 2988, 2935, 1791, 1733, 1696, 1608, 1587; $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm) 8.01 (d, J = 8.0 Hz, 1H), 7.90 (d, J = 9.6 Hz, 1H), 7.86 – 7.82 (m, 2H), 7.79 – 7.56 (m, 2H), 7.52 – 7.46 (m, 1H), 7.33 – 7.28 (m, 1H), 5.65 (q, J = 6.8 Hz, 1H), 1.69 (d, J = 6.8 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm) 194.12, 163.63, 162.71 (d, J$_{C-F}$ = 246 Hz), 136.63 (d, J$_{C-F}$ = 6 Hz), 134.71, 130.30 (d, J$_{C-F}$ = 7 Hz), 128.70, 125.11 (d, J$_{C-F}$ = 3 Hz), 123.72, 120.78 (d, J$_{C-F}$ = 21 Hz), 116.06 (d, J$_{C-F}$ = 22 Hz), 83.90, 15.92; $^{19}$F NMR (376 MHz, CDCl$_3$) δ (ppm) -111.487; HRESI-MS (m/z): Calculated for C$_{17}$H$_{12}$NO$_4$F (M$^+$ + Na): 336.0648, found (M$^+$ + Na): 336.0649.

2-((1-(3-chlorophenyl)-1-oxopropan-2-yl)oxy)isoindoline-1,3-dione (4g).

White Solid; Yield 64% (105 mg); mp 125–128 °C; R$_f$ (20% EtOAc/hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 30min at 100°C): IR (KBr, cm$^{-1}$): 3400, 3066, 2919, 1791, 1735, 1699, 1690, 1570; $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm) 8.19 (s, 1H), 8.10 (d, J = 8.0 Hz, 1H), 7.86 – 7.83 (m, 2H), 7.78 – 7.75 (m, 2H), 7.57 (d, J = 8.0 Hz, 1H), 7.46 (t, J = 8.0 Hz, 1H), 5.63 (q, J = 6.8 Hz, 1H), 1.69 (d, J = 6.8 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm) 194.03, 163.56, 136.12, 134.86, 134.67, 133.55, 129.90, 129.28, 128.63, 127.40, 123.66, 83.85, 15.81; HRESI-MS (m/z): Calculated for C$_{17}$H$_{12}$ClNO$_4$ (M$^+$ + Na): 352.0353, found (M$^+$ + Na): 352.0352

2-((1-(4-chlorophenyl)-1-oxopropan-2-yl)oxy)isoindoline-1,3-dione (4h).

White Solid; Yield 60% (99 mg); mp 127–130 °C; R$_f$ (20% EtOAc/hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 1.5h at 100 °C): IR(KBr, cm$^{-1}$): 3751, 3102, 3045, 2939, 2345, 1788, 1730, 1688, 1612, 1591, 1465; $^1$H NMR (400 MHz, CDCl$_3$) δ (ppm) 8.17 (d, J = 8.4 Hz, 2H), 7.85 – 7.82 (m, 2H), 7.78 – 7.75 (m, 2H), 7.48 (d, J = 8.0 Hz, 2H), 5.63 (q, J = 6.8 Hz, 1H), 1.68 (d, J = 6.8 Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ (ppm) 194.13, 163.64, 140.23, 134.71, 133.01, 130.81, 128.97,
128.72, 123.71, 83.98, 15.88; **HRESI-MS** (m/z): Calculated for C₁₇H₁₂ClNO₄ (M⁺ + Na): 352.0353, found (M⁺ + Na): 352.0352.

2-((1-(4-bromophenyl)-1-oxopropan-2-yl)oxy)isoindoline-1,3-dione (4i).

White Solid; Yield 58% (109 mg); mp 130–132 °C; R_f (20% EtOAc/hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 1.5 h at 100 °C): **IR** (KBr, cm⁻¹): 3840, 3737, 3620, 3101, 2852, 1772, 1734, 1718, 1700, 1542; **¹H NMR** (400 MHz, CDCl₃) δ (ppm) 8.09 (d, J = 8.4 Hz, 2H), 7.85 – 7.81 (m, 2H), 7.79 – 7.75 (m, 2H), 7.65 (d, J = 8.4 Hz, 2H), 5.62 (q, J = 6.8 Hz, 1H), 1.68 (d, J = 6.8 Hz, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm) 194.34, 163.63, 134.72, 133.41, 131.96, 130.88, 129.05, 128.70, 123.72, 83.96, 15.86; **HRESI-MS** (m/z): Calculated for C₁₇H₁₂BrNO₄ (M⁺ + Na): 395.9847, found (M⁺ + Na): 395.9845.

2-((1-oxo-1-(thiophen-2-yl)propan-2-yl)oxy)isoindoline-1,3-dione (4j).

Pale yellow Solid; Yield 51% (77 mg); mp 103–105 °C; R_f (20% EtOAc/hexane) 0.4. Prepared as shown in general experimental procedure (Reaction time: 2h at 100 °C): **IR** (KBr, cm⁻¹): 3815, 3722, 3493, 2918, 1737, 1663, 1373; **¹H NMR** (400 MHz, CDCl₃) δ (ppm) 8.23 (d, J = 3.6 Hz, 1H), 7.85 – 7.82 (m, 2H), 7.78 – 7.75 (m, 2H), 7.73 (d, J = 4.8 Hz, 1H), 7.20 (t, J = 4.4 Hz, 1H), 5.49 (q, J = 6.8 Hz, 1H), 1.72 (d, J = 6.4 Hz, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm) 188.39, 163.60, 141.27, 135.09, 134.69, 134.51, 128.75, 128.44, 123.72, 85.05, 16.59; **HRESI-MS** (m/z): Calculated for C₁₅H₁₁NO₄S (M⁺ + Na): 324.0306, found (M⁺ + Na): 324.0307.

2-((1-(5-methylthiophen-2-yl)-1-oxopropan-2-yl)oxy)isoindoline-1,3-dione (4k).

White Solid; Yield 58% (91 mg); mp 146–148 °C; R_f (20% EtOAc/hexane) 0.4. Prepared as shown in general experimental procedure (Reaction time: 2h at 100 °C): **IR** (KBr, cm⁻¹): 2938, 1782, 1734, 1653, 1605, 1451; **¹H NMR** (400 MHz, CDCl₃) δ (ppm) 8.03 (d, J = 3.6 Hz, 1H), 7.85 – 7.81 (m, 2H), 7.78 – 7.75 (m, 2H), 7.73 (d, J = 4.8 Hz, 1H), 7.20 (t, J = 4.4 Hz, 1H), 5.46 (q, J = 6.8 Hz, 1H), 2.55 (s, 3H), 1.69 (d, J = 6.8 Hz, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm) 187.82, 163.59, 151.45, 139.13, 135.18, 134.63, 128.76, 127.22, 123.67, 84.74, 16.66, 16.08; **HRESI-MS** (m/z): Calculated for C₁₆H₁₃NO₄S (M⁺ + Na): 338.0463, found (M⁺ + Na): 338.0464.

2-((1-(5-bromothiophen-2-yl)-1-oxobutan-2-yl)oxy)isoindoline-1,3-dione (4l).
Pale yellow Solid; Yield 53% (104 mg); mp 102–104 °C; R$_f$ (20% EtOAc/hexane) 0.4. Prepared as shown in general experimental procedure (Reaction time: 2h at 100 °C):

**IR** (KBr, cm$^{-1}$): 3856, 3747, 3620, 3098, 2920, 2627, 1757, 1735, 1718, 1707, 1528, 1406; **$^1$H NMR** (400 MHz, CDCl$_3$) $\delta$ (ppm) 7.89 (d, $J$ = 4.0 Hz, 1H), 7.83 – 7.80 (m, 2H), 7.78 – 7.74 (m, 2H), 7.15 (d, $J$ = 4.4 Hz, 1H), 5.07 (t, $J$ = 6.8 Hz, 1H), 2.21 – 2.03 (m, 2H), 1.13 (t, $J$ = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, CDCl$_3$) $\delta$ (ppm) 188.00, 163.30, 142.84, 134.69, 134.58, 131.46, 128.36, 124.30, 123.67, 91.13, 25.06, 9.53; **HRESI-MS** (m/z): Calculated for C$_{16}$H$_{12}$BrNO$_4$S (M$^+$ + Na): 415.9568, found (M$^+$ + Na): 415.9570.

2-((1H-benzo[d][1,2,3]triazol-1-yl)oxy)-1-phenylpropan-1-one (5a).

White solid; Yield 42% (56 mg); mp 68–70 °C; R$_f$ (20% EtOAc/hexane) 0.5. Prepared as shown in general experimental procedure (Reaction time: 2 h at 80 °C):

**IR** (KBr, cm$^{-1}$): 3401, 2989, 2359, 1697, 1592, 1448, 1375; **$^1$H NMR** (400 MHz, CDCl$_3$) $\delta$ (ppm) 7.97 – 7.93 (m, 3H), 7.82 (d, $J$ = 8.4 Hz, 1H), 7.58 (t, $J$ = 7.2 Hz, 1H), 7.51 (t, $J$ = 8.0 Hz, 1H), 7.46 (t, $J$ = 7.6 Hz, 2H), 7.35 (t, $J$ = 7.6 Hz, 1H), 6.36 (q, $J$ = 6.8 Hz, 1H), 1.79 (d, $J$ = 6.8 Hz, 3H); **$^{13}$C NMR** (100 MHz, CDCl$_3$) $\delta$ (ppm) 195.37, 143.20, 134.12, 134.01, 128.87, 128.54, 128.14, 128.06, 124.63, 119.73, 110.07, 85.32, 16.95; **HRESI-MS** (m/z): Calculated for C$_{15}$H$_{13}$N$_3$O$_2$ (M$^+$ + Na): 290.0905, found (M$^+$ + Na): 290.0906.

2-((1H-benzo[d][1,2,3]triazol-1-yl)oxy)-1-(3-fluorophenyl)propan-1-one (5b).

White solid; Yield 41% (58 mg); mp 87–89 °C; R$_f$ (20% EtOAc/hexane) 0.5. Prepared as shown in general experimental procedure (Reaction time: 2 h at 80 °C): **IR** (KBr, cm$^{-1}$): 3403, 2983, 2359, 1697, 1592, 1448, 1376; **$^1$H NMR** (400 MHz, CDCl$_3$) $\delta$ (ppm) 7.96 (d, $J$ = 8.4 Hz, 1H), 7.82 – 7.76 (m, 2H), 7.67 (d, $J$ = 8.4 Hz, 1H), 7.53 (t, $J$ = 8.0 Hz, 1H), 7.49 – 7.43 (m, 1H), 7.37 (t, $J$ = 7.6 Hz, 1H), 7.32 – 7.27 (m, 1H), 6.30 (q, $J$ = 6.8 Hz, 1H), 1.79 (d, $J$ = 6.8 Hz, 3H); **$^{13}$C NMR** (100 MHz, CDCl$_3$) $\delta$ (ppm) 194.23 (d, $J_{CF}$ = 2 Hz), 162.77, (d, $J_{CF}$ = 248 Hz), 143.19, 135.96 (d, $J_{CF}$ = 7 Hz), 130.66 (d, $J_{CF}$ = 8 Hz), 128.12 (d, $J_{CF}$ = 27 Hz), 124.73, 124.39 (d, $J_{CF}$ = 3 Hz), 121.25 (d, $J_{CF}$ = 21 Hz), 119.80, 115.47, 115.24, 109.91, 85.39, 16.81; **$^{19}$F NMR** (376 MHz, CDCl$_3$) $\delta$ (ppm) -110.649; **HRESI-MS** (m/z): Calculated for C$_{15}$H$_{12}$N$_3$O$_2$F (M$^+$ + Na): 308.0811, found (M$^+$ + Na): 308.0812.
Yellow solid; Yield 41% (62 mg); mp 89–91 °C; \( R_f \) (20% EtOAc/hexane) 0.5. Prepared as shown in general experimental procedure (Reaction time: 2 h at 80 °C): \( \text{IR} (\text{KBr}, \text{cm}^{-1}) \): 3418, 3069, 2989, 2935, 2352, 1698, 1573, 1427; \( ^1\text{H NMR} \) (400 MHz, CDCl\(_3\)) \( \delta \) (ppm) 7.96 – 7.92 (m, 2H), 7.86 (d, \( J = 8.0 \) Hz, 1H), 7.80 (d, \( J = 8.4 \) Hz, 1H), 7.56 – 7.51 (m, 2H), 7.44 – 7.34 (m, 2H), 6.29 (q, \( J = 6.8 \) Hz, 1H), 1.78 (d, \( J = 6.8 \) Hz, 3H); \( ^{13}\text{C NMR} \) (100 MHz, CDCl\(_3\)) \( \delta \) (ppm) 194.24, 143.18, 135.46, 135.29, 134.04, 130.21, 128.60, 128.26, 127.97, 126.73, 124.73, 119.80, 109.89, 109.86, 85.33, 16.79; HRESI-MS (m/z): Calculated for C\(_{15}\)H\(_{12}\)N\(_3\)O\(_2\)Cl (M\(^+\) + Na): 324.0516, found (M\(^+\) + Na): 324.0513.

\( \text{2-((1H-benzo[d][1,2,3]triazol-1-yl)oxy)-1-(4-chlorophenyl)propan-1-one (5d).} \)

White solid; Yield 44% (67 mg); mp 96–98 °C; \( R_f \) (20% EtOAc/hexane) 0.5. Prepared as shown in general experimental procedure (Reaction time: 3 h at 80 °C): \( \text{IR} (\text{KBr}, \text{cm}^{-1}) \): 3734, 2985, 2359, 1933, 1696, 1586, 1567; \( ^1\text{H NMR} \) (400 MHz, CDCl\(_3\)) \( \delta \) (ppm) 7.97 – 7.93 (m, 3H), 7.78 (d, \( J = 8.4 \) Hz, 1H), 7.53 (t, \( J = 7.6 \) Hz, 1H), 7.45 (d, \( J = 8.8 \) Hz, 2H), 7.37 (t, \( J = 8.0 \) Hz, 1H), 6.29 (q, \( J = 6.8 \) Hz, 1H), 1.77 (d, \( J = 6.8 \) Hz, 3H); \( ^{13}\text{C NMR} \) (100 MHz, CDCl\(_3\)) \( \delta \) (ppm) 194.15, 143.19, 140.73, 132.33, 130.04, 129.25, 128.60, 128.01, 124.73, 119.82, 109.86, 85.30, 16.73; HRESI-MS (m/z): Calculated for C\(_{15}\)H\(_{12}\)N\(_3\)O\(_2\)Cl (M\(^+\) + Na): 324.0516, found (M\(^+\) + Na): 324.0518.

\( \text{2-((1H-benzo[d][1,2,3]triazol-1-yl)oxy)-1-(3-bromophenyl)propan-1-one (5e).} \)

White solid; Yield 40% (70 mg); mp 64–66 °C; \( R_f \) (20% EtOAc/hexane) 0.5. Prepared as shown in general experimental procedure (Reaction time: 3 h at 80 °C): \( \text{IR} (\text{KBr}, \text{cm}^{-1}) \): 3394, 2987, 3066, 2929, 2870, 2371, 1698, 1589, 1567, 1444; \( ^1\text{H NMR} \) (400 MHz, CDCl\(_3\)) \( \delta \) (ppm) 8.10 (s, 1H), 7.95 (d, \( J = 8.4 \) Hz, 1H), 7.90 (d, \( J = 7.6 \) Hz, 1H), 7.45 (d, \( J = 8.8 \) Hz, 2H), 7.37 (t, \( J = 8.0 \) Hz, 1H), 6.29 (q, \( J = 6.8 \) Hz, 1H), 1.77 (d, \( J = 6.8 \) Hz, 3H); \( ^{13}\text{C NMR} \) (100 MHz, CDCl\(_3\)) \( \delta \) (ppm) 194.17, 143.15, 136.97, 135.67, 131.55, 130.45, 130.04, 129.25, 128.60, 128.01, 124.73, 119.83, 109.91, 85.31, 16.81; HRESI-MS (m/z): Calculated for C\(_{15}\)H\(_{12}\)N\(_3\)O\(_2\)Br (M\(^+\) + Na): 368.0011, found (M\(^+\) + Na): 368.0010.

\( \text{2-((3H-[1,2,3]triazolo[4,5-b]pyridin-3-yl)oxy)-1-phenylpropan-1-one (5f).} \)

White solid; Yield 44% (62 mg); mp 82–84 °C; \( R_f \) (30% EtOAc/hexane) 0.1. Prepared as shown in general experimental procedure (Reaction time: 2 h at 80 °C): \( \text{IR} (\text{KBr}, \text{cm}^{-1}) \): 3059, 3000, 2922, 2851, 2465, 2386, 2290, 2230, 1976, 1938, 1688, 1591; \( ^1\text{H NMR} \) (400 MHz, CDCl\(_3\)) \( \delta \) (ppm) 8.77 (d, \( J = 4.0 \) Hz, 1H), 8.35 (d, \( J = 8.4 \) Hz, 1H), 7.96 – 7.84 (m, 3H), 7.45 – 7.33 (m, 2H), 7.30 – 7.16 (m, 7H), 7.07 (d, \( J = 8.0 \) Hz, 1H), 6.34 (q, \( J = 6.8 \) Hz, 1H), 1.78 (d, \( J = 6.8 \) Hz, 3H); HRESI-MS (m/z): Calculated for C\(_{15}\)H\(_{12}\)N\(_3\)O\(_2\)Br (M\(^+\) + Na): 368.0011, found (M\(^+\) + Na): 368.0010.

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Hz, 1H), 8.11 (d, J = 8.0 Hz, 2H), 7.61 (t, J = 7.2 Hz, 1H), 7.50 (t, J = 7.6 Hz, 2H), 7.43 – 7.39 (m, 1H), 6.31 (q, J = 6.8 Hz, 1H), 1.81 (d, J = 6.8 Hz, 3H); ^13C NMR (100 MHz, CDCl₃) δ (ppm) 194.42, 151.49, 139.88, 134.84, 134.17, 133.98, 129.10, 128.89, 128.78, 120.70, 85.73, 16.46; HRESI-MS (m/z): Calculated for C₁₄H₁₂N₄O₂ (M⁺ + Na): 291.0853, found (M⁺ + Na): 291.0858.

2-((3H-[1,2,3]triazolo[4,5-b]pyridin-3-yl)oxy)-1-(3-chlorophenyl)propan-1-one (5g).

Pale yellow oily; Yield 47% (75 mg); Rₓ (30% EtOAc/hexane) 0.1. Prepared as shown in general experimental procedure (Reaction time: 2 h at 80 °C): IR (Neat, cm⁻¹): 3778, 3701, 3583, 3377, 3068, 2991, 2927, 2853, 2381, 2315, 1698, 1589; ^1H NMR (400 MHz, CDCl₃) δ (ppm) 8.77 (d, J = 4.0 Hz, 1H), 8.37 (d, J = 8.4 Hz, 1H), 8.14 (s, 1H), 8.05 (d, J = 8.0 Hz, 1H), 7.59 (d, J = 7.6 Hz, 1H), 7.49 – 7.41 (m, 2H), 6.20 (q, J = 6.8 Hz, 1H), 1.80 (d, J = 6.8 Hz, 3H); ^13C NMR (100 MHz, CDCl₃) δ (ppm) 193.32, 151.54, 139.82, 135.69, 135.13, 134.84, 133.89, 130.11, 129.21, 129.06, 127.11, 120.80, 85.87, 16.18; HRESI-MS (m/z): Calculated for C₁₄H₁₁ClN₄O₂ (M⁺ + Na): 325.0468, found (M⁺ + Na): 325.0468.

2-((3H-[1,2,3]triazolo[4,5-b]pyridin-3-yl)oxy)-1-(3-bromophenyl)propan-1-one (5h).

White solid; Yield 50% (92 mg); mp87–89 °C; Rₓ (30% EtOAc/hexane) 0.1. Prepared as shown in general experimental procedure (Reaction time: 3 h at 80 °C): IR (KBr, cm⁻¹): 3350, 3072, 2928, 2851, 2598, 2550, 2332, 2290, 1686, 1565; ^1H NMR (400 MHz, CDCl₃) δ (ppm) 8.78 (d, J = 4.0 Hz, 1H), 8.37 (d, J = 8.4 Hz, 1H), 8.31 (s, 1H), 8.10 (d, J = 7.6 Hz, 1H), 7.74 (d, J = 8.0 Hz, 1H), 7.45 – 7.38 (m, 2H), 6.19 (q, J = 6.8 Hz, 1H), 1.80 (d, J = 6.8 Hz, 3H); ^13C NMR (100 MHz, CDCl₃) δ (ppm) 193.22, 151.54, 139.83, 136.81, 135.90, 133.89, 130.11, 129.21, 127.56, 123.10, 120.81, 85.84, 16.16; HRESI-MS (m/z): Calculated for C₁₄H₁₁N₂O₂Br (M⁺ + Na): 368.9979, found (M⁺ + Na): 368.9963.
General procedure for synthesis of vinylphosphates

A solution of ketone (0.5 mmol, 1 equiv), and triethyl phosphate (5 equiv), in toluene (2 mL) in a screw capped reaction vial was heated at 110°C for 12 h. The reaction mixture was cooled to RT and diluted with H_2O (5 mL) and extracted with ethyl acetate (10 mL x 3). The combined organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was purified on a silica gel column using hexane/EtOAc to get the pure product.

Diethyl (1-phenylprop-1-en-1-yl) phosphate (6a).^9

Colourless oily; Yield 92% (124 mg); R_f (30% EtOAc/hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 12 h at 110 °C): IR (Neat, cm⁻¹): 3673, 3476, 3312, 3060, 2985, 2922, 2859, 2740, 1732, 1666, 1575; \(^1\)H NMR (400 MHz, CDCl_3) δ 7.50 (d, J = 7.6 Hz, 2H), 7.36 – 7.25 (m, 3H), 5.70 – 5.64 (m, 1H), 4.17 – 3.99 (m, 4H), 1.90 – 1.75 (m, 3H), 1.26 – 1.22 (m, 6H); \(^13\)C NMR (100 MHz, CDCl_3) δ 146.80, 146.71, 135.72, 128.51, 128.43, 128.14, 128.03, 128.01, 125.29, 112.22, 112.15, 64.26, 64.20, 15.99, 15.92, 11.72, 11.70; \(^31\)P NMR (162 MHz, CDCl_3) δ -5.72; HRESI-MS (m/z): Calculated for C_{13}H_{19}O_4P (M + Na): 293.0919, found (M + Na): 293.0919;

1-(3-chlorophenyl)prop-1-en-1-yl diethyl phosphate (6n).

Colourless oily; Yield 83% (126 mg); R_f (30% EtOAc/hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 12 h at 110 °C): IR (Neat, cm⁻¹): 3487, 3064, 2985, 2920, 2863, 2373, 1945, 1742, 1664, 1594, 1566, 1477; \(^1\)H NMR (400 MHz, CDCl_3) δ 7.49 (S, 1H), 7.41 – 7.33 (s, 1H), 7.31 – 7.24 (m, 2H), 5.74 – 5.67 (m, 1H), 4.19 – 4.03 (m, 4H), 1.89 (dd, J = 6.8, 3.2 Hz, 3H), 1.27 (t, J = 6.8 Hz, 6H); \(^13\)C NMR (100 MHz, CDCl_3) δ 145.43, 145.34, 137.41, 137.39, 134.07, 129.36, 127.91, 125.29, 123.29, 113.47, 113.40, 64.29, 64.23, 15.92, 15.85, 11.68, 11.67; \(^31\)P NMR (162 MHz, CDCl_3) δ -5.76; HRESI-MS (m/z): Calculated for C_{13}H_{18}ClO_4P (M + Na): 327.0529, found (M + Na): 327.0530;

1-(4-chlorophenyl)prop-1-en-1-yl diethyl phosphate (6o).

Colourless oily; Yield 87% (132 mg); R_f (30% EtOAc/hexane) 0.2. Prepared as shown in general experimental procedure (Reaction time: 12 h at 110 °C): IR (Neat, cm⁻¹): 3667, 3555, 3490, 3050, 2985, 2920, 2864, 2740, 2572, 1903, 1841, 1743, 1664, 1595; \(^1\)H NMR (400 MHz, CDCl_3) δ 7.45 – 7.39 (m, 2H), 7.36 – 7.28 (m, 2H), 5.67 – 5.65 (m, 1H), 4.18 – 4.02 (m, 4H), 1.89 – 1.73 (m, 6H), 1.26 – 1.22 (m, 6H); \(^13\)C NMR (100 MHz, CDCl_3) δ 144.43, 144.34, 137.41, 137.39, 134.07, 129.36, 127.91, 125.29, 123.29, 113.47, 113.40, 64.29, 64.23, 15.92, 15.85, 11.68, 11.67; \(^31\)P NMR (162 MHz, CDCl_3) δ -5.76; HRESI-MS (m/z): Calculated for C_{13}H_{18}ClO_4P (M + Na): 327.0529, found (M + Na): 327.0530;
3H), 1.27 (t, J = 6.8 Hz, 6H); $^1$H NMR (100 MHz, CDCl$_3$) δ 145.77, 145.68, 134.16, 134.14, 133.78, 129.81, 128.29, 128.25, 126.51, 112.76, 112.70, 112.27, 64.31, 64.25, 15.99, 15.92, 11.71, 11.69; $^{31}$P NMR (162 MHz, CDCl$_3$) δ -5.67; HRESI-MS (m/z): Calculated for C$_{13}$H$_{18}$ClO$_4$P (M + Na): 327.0529, found (M + Na): 327.0528;
References


The image appears to be a 13C NMR spectrum of a compound. The spectrum shows various peaks at different ppm values, indicating the chemical shifts of the carbon nuclei in the compound. The peaks at specific ppm values suggest the presence of certain functional groups and chemical environments within the molecule.

The compound is labeled as 3g, and the NMR spectrum was recorded in CDCl3 using a 100 MHz instrument. The peaks are marked with their corresponding chemical shifts, which are crucial for identifying the structure of the compound.
ESI 48

$\text{CDCl}_3^{13}\text{C NMR 100MHz}$
CDCl$_3$ $^{19}$F NMR 376 MHz

ESI 49
$\text{CDCl}_3 \ ^1\text{H NMR 400MHz}$
CDCl$_3$ $^{13}$C NMR 100MHz
\[
\text{CDCl}_3{}^{19}\text{F NMR 376 MHz}
\]
$\text{CDCl}_3 \ \ ^1\text{H NMR} \ 400\text{MHz}$
CDCl₃ ¹H NMR 400MHz
Yogesh and Prabhu/Organic Chemistry/IISc

ESI 65

CDCl₃ ¹⁹F NMR 376 MHz

 ppm
CDCl₃ ¹H NMR 400MHz
$\text{CDCl}_3^{13}\text{C} \text{ NMR 100MHz}$

- 182.76
- 171.00
- 159.15
- 149.00
- 122.66
- 109.36
- 82.79
- 77.32
- 77.00
- 76.68
- 25.28
- 16.41
- 13.96
The diagram depicts an NMR spectrum of a compound with the chemical formula 3y. The spectrum is recorded in CDCl₃ and has a frequency of 400 MHz. The peaks are labeled with their respective chemical shifts, indicating the presence of various functional groups and protons in the molecule. The spectrum shows distinct peaks at different ppm values, which correspond to different chemical environments within the molecule.
CDCl$_3$ $^{13}$C NMR 100MHz
CDCl₃ $^1$H NMR 400MHz
CDCl₃ ¹H NMR 400MHz
$\text{CDCl}_3\ ^{19}\text{F NMR 376MHz}$
Yogesh and Prabhu/Organic Chemistry/IISc

ESI 96

CDCl₃¹³C NMR 100MHz
CDCl₃ ¹H NMR 400MHz

5a

ESI 105
$^{19}$F NMR in CDCl$_3$ 376 MHz
$$\text{CDCl}_3^{13}\text{C NMR 100MHz}$$
$\text{CDCl}_3 \quad ^{31}\text{P NMR 162 MHz}$
CDCl$_3$ $^{31}$P NMR 162 MHz
CDCl$_3$ $^{31}$P NMR 162MHz