Supplementary Information for

A brisk and flexible synthetic approach to enureas (alkenyl ureas) via Pd-catalyzed C-N coupling reaction of alkenyl tosylates and mesylates

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1. Reagents

All reactions were carried out under a nitrogen atmosphere. Air- and moisture-sensitive solvents and solutions were transferred via syringe or stainless steel cannula. All chemicals were purchased from sigma Aldrich, merck and fluka. Solvents used were of analytical grade. Anhydrous potassium carbonate was stored in a nitrogen-filled glovebox, ground and was taken out in small quantities and stored in a desiccator. Aryl ureas were prepared by known methods\(^1\). All reactions were routinely checked by TLC. TLC was performed on aluminum-backed silica gel plates (silica gel 60 F\(\text{254}\) grade, Merck DC) with spots visualized by UV light. Column chromatography was performed on silica gel LC 60A (70-200 micron).

Instrumental

All compounds were characterized by \(^1\)H NMR, \(^{13}\)C NMR as well as elemental analysis. Melting points were determined in open capillaries on a Veego electronic apparatus VMP-D (Veego Instrument Corporation, Mumbai, India) and are uncorrected. \(^1\)HNMR and \(^{13}\)C NMR spectra were recorded on a Bruker 400 MHz model spectrometer using DMSO-d6 as a solvent and TMS as internal standard with \(^1\)H resonant frequency of 400 MHz and \(^{13}\)C resonant frequency of 100 MHz. The \(^1\)H NMR, \(^{13}\)C NMR chemical shifts were reported as parts per million (ppm) downfield from TMS (Me4Si). The splitting patterns are designated as follows; s, singlet; d, doublet; t, triplet; m, multiplet. Elemental analyses (C, H, N) were performed using a Heraeus CarloErba 1180 CHN analyzer (Hanau, Germany).

2. Preparation of Ligand, alkenyl tosylates and mesylates substrates
Ferrocene based triazine ligand L were synthesised according to the literature method without modification.\textsuperscript{2} Pyronyl tosylates and mesylates were prepared from their corresponding precursors with TsCl or MsCl in the presence of triethylamine in CH\textsubscript{2}Cl\textsubscript{2} according to the literature method without modifications.\textsuperscript{3} Other alkeny tosylates and mesylates were prepared from their corresponding species according to the literature method without modifications.\textsuperscript{4}

4. General procedure of reaction conditions screening

To an oven dried flat-bottomed flask which was equipped with a magnetic stir bar, was charged with phenyl urea (1.0 mmol), base (1.4 mmol), ligand (5 mol%), Pd (1.6 mol%), and alkeny tosylate 2 (1.0 mmol) in solvent (5.0 ml). The reaction was sparged with nitrogen for 15 minutes, stirred and heated to 60 °C for 10 hours. After completion of reaction, the reaction mixture was cooled to room temperature and filtered through a pad of Celite eluting with ethyl acetate. The filtrate was concentrated and purification of the residue by silica gel column chromatography.

Table 1. Screening of ligands\textsuperscript{a}

<table>
<thead>
<tr>
<th>Entry</th>
<th>Ligand</th>
<th>Yield\textsuperscript{b} (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Xphos</td>
<td>44</td>
</tr>
<tr>
<td>2</td>
<td>Sphos</td>
<td>50</td>
</tr>
<tr>
<td>3</td>
<td>Ruphos</td>
<td>55</td>
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<td>4</td>
<td>Dppf</td>
<td>38</td>
</tr>
<tr>
<td>5</td>
<td>Xantphos</td>
<td>49</td>
</tr>
<tr>
<td>6</td>
<td>DPEphos</td>
<td>35</td>
</tr>
<tr>
<td>7</td>
<td>Josiphos</td>
<td>78</td>
</tr>
<tr>
<td>8</td>
<td>Ligand L</td>
<td>93</td>
</tr>
</tbody>
</table>

\textsuperscript{a} Electronic Supplementary Material (ESI) for RSC Advances

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(ferrocene based triazine ligand)
Pd$_2$(dba)$_3$: 1.6 mol %, Ligand: 5 mol %, Phenyl Urea: 1.0 mmol, alkenyl tosylate: 1.0 mmol, K$_2$CO$_3$: 1.4 mmol, toluene: 5 ml per mmol.

Isolated yields.

**Table 2. Screening of the Pd-Catalysts**

<table>
<thead>
<tr>
<th>Entry</th>
<th>Pd Catalyst</th>
<th>Yield$^b$ (%)</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>Pd$_2$(dba)$_3$</td>
<td>93</td>
</tr>
<tr>
<td>2</td>
<td>Pd(OAC)$_2$</td>
<td>60</td>
</tr>
<tr>
<td>3</td>
<td>Pd(dppf)Cl$_2$</td>
<td>52</td>
</tr>
<tr>
<td>4</td>
<td>Pd(Ph$_3$P)$_2$Cl$_2$</td>
<td>33</td>
</tr>
</tbody>
</table>

$^a$ Pd: 1.6 mol %, ligand L: 5 mol %, Phenyl Urea: alkenyl tosylate: 1.0 mmol, K$_2$CO$_3$: 1.4 mmol, toluene: 5 ml per mmol.

Isolated yields.

**Table 3. Screening of bases**

<table>
<thead>
<tr>
<th>Entry</th>
<th>Base</th>
<th>Yield$^b$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Cs$_2$CO$_3$</td>
<td>67</td>
</tr>
<tr>
<td>2</td>
<td>NaO$t$Bu</td>
<td>33</td>
</tr>
<tr>
<td>3</td>
<td>K$_2$CO$_3$</td>
<td>93</td>
</tr>
<tr>
<td>4</td>
<td>K$_3$PO$_4$</td>
<td>56</td>
</tr>
<tr>
<td>5</td>
<td>N(C$_2$H$_5$)$_3$</td>
<td>0</td>
</tr>
</tbody>
</table>

$^a$ Pd$_2$(dba)$_3$: 1.6 mol %, ligand L: 5 mol %, Phenyl Urea: 1.0 mmol, alkenyl tosylate: 1.0 mmol, Base: 1.4 mmol, toluene: 5 ml per mmol.

Isolated yields.

**Table 4. Screening of solvents**

<table>
<thead>
<tr>
<th>Entry</th>
<th>Solvent</th>
<th>Yield$^b$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1,4-dioxane</td>
<td>75</td>
</tr>
<tr>
<td>2</td>
<td>THF</td>
<td>80</td>
</tr>
<tr>
<td>3</td>
<td>Toluene</td>
<td>93</td>
</tr>
<tr>
<td>4</td>
<td>DMF</td>
<td>39</td>
</tr>
<tr>
<td>5</td>
<td>t-$BuOH$</td>
<td>30</td>
</tr>
</tbody>
</table>

Isolated yields.
5. General procedures coupling reactions

To an oven dried flat-bottomed flask which was equipped with a magnetic stir bar, was charged with urea (1.0 mmol), K$_2$CO$_3$ (1.4 mmol), ligand L (5 mol %), Pd$_2$(dba)$_3$ (3.3 mol %), and alkenyl tosylate or mesylate (1.0 mmol) in Toluene (5.0 ml). The reaction was sparged with nitrogen for 15 minutes, stirred and heated to 60 °C (reactions were carried out at room temperature for the synthesis of tosyloxycoumarin, tosyloxyquinolinone, tosyloxypyrane and tosyloxyfuranone with different ureas) for 10 hours. The reaction mixture was cooled to room temperature and filtered through a pad of Celite eluting with ethyl acetate. The filtrate was concentrated and purification of the residue by silica gel column chromatography gave the desired product.

6. Characterization of coupling yield

$^1$H NMR (400 MHz, DMSO-d6) δ ppm: 9.44 (s, 1H), 8.05 (s, 1H), 7.39-7.22 (m, 14H), 7.10 (m, 1H), 2.10 (s, 3H), $^{13}$C NMR (100 MHZ, DMSO-d6) δ ppm: 156.55, 143.50, 141.08, 137.77,
129.02, 128.73, 128.35, 127.10, 122.21, 121.45, 118.19, 23.00 \textbf{Anal. Calcd. For } \text{C}_{22}\text{H}_{20}\text{N}_{2}O: \text{C}, 80.46; \text{H}, 6.14; \text{N}, 8.53 \textbf{ Found: } \text{C}, 80.50; \text{H}, 6.22; \text{N}, 8.44.  \textbf{mp } 130^\circ \text{C.}

![Chemical structure 1]

1\text{H NMR} (400 MHz, DMSO-d6) \delta \text{ ppm: } 9.42 (s, 1H), 7.80 (s, 1H), 7.32 (m, 8H), 7.05 (m, 1H), 6.83 (m, 1H), 2.19 (t, 2H), 1.91 (t, 2H), 1.69 (m, 4H) \textbf{13C NMR} (400 MHZ, DMSO-d6) \delta \text{ ppm: } 156.55, 140.88, 138.88, 129.06, 128.79, 128.31, 126.23, 126.00, 123.90, 121.15, 118.19, 29.00, 27.20, 24.14, 23.88. \textbf{Anal. Calcd. For } \text{C}_{19}\text{H}_{20}\text{N}_{2}O: \text{C}, 78.05; \text{H}, 6.89; \text{N}, 9.58 \textbf{ Found: } \text{C}, 78.00; \text{H}, 6.81; \text{N}, 9.64.  \textbf{mp } 151^\circ \text{C.}

![Chemical structure 2]

1\text{H NMR} (400 MHz, DMSO-d6) \delta \text{ ppm: } 9.47 (s, 1H), 8.32 (s, 1H), 7.45, (d, \text{J} = 7.6 \text{ Hz}, 2H), 7.24 (t, \text{J} = 7.6 \text{ Hz}, 2H), 7.12 (m, 3H), 7.07(t, \text{J} = 7.4 \text{ Hz}, 1H), 7.00(m, 1H), 5.07 (s, 1H), 3.12 (t, 2H), 2.72 (t, 2H). \textbf{13C NMR} (100 MHZ, DMSO-d6) \delta \text{ ppm: } 156.47, 141.17, 140.11, 136.00, 134.00, 129.04, 128.55, 127.10, 126.70, 126.25, 125.00, 121.78, 118.13, 33.11, 28.00 \textbf{Anal. Calcd. For } \text{C}_{17}\text{H}_{16}\text{N}_{2}O: \text{C}, 77.25; \text{H}, 6.10; \text{N}, 10.60. \textbf{ Found: } \text{C}, 77.29; \text{H}, 6.14; \text{N}, 10.50.  \textbf{mp } 143^\circ \text{C.}
\(^1\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) ppm : 9.45 (s, 1H), 9.00 (s, 1H), 7.49 (d, \(J = 7.6\) Hz, 2H), 7.31 (t, \(J = 7.6\) Hz, 2H), 7.04 (t, \(J = 7.4\) Hz, 1H), 4.20 (m, 2H), 2.62 (t, \(J = 6.2\) Hz, 2H), 2.34 (t, \(J = 6.2\) Hz, 2H), 1.82 (m, 4H), 1.38 (t, \(J = 5.8\) Hz, 3H), 13\(^\text{C} \) NMR (100 MHZ, DMSO-\(d_6\)) \(\delta\) ppm : 171.00, 156.37, 149.11, 140.94, 128.98, 121.00, 118.48, 104.12, 62.00, 28.28, 24.20, 23.05, 22.67, 16.03. Anal. Calcd. For \(C_{16}H_{20}N_2O_3\): C, 66.65; H, 6.99; N, 9.72. Found: C, 66.60; H, 6.91; N, 9.79. mp 171-172°C.

\(^1\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) ppm : 9.44 (s, 1H), 8.07 (s, 1H), 7.46 (d, \(J = 7.6\) Hz, 2H), 7.30 (t, \(J = 7.6\) Hz, 2H), 7.01 (t, \(J = 7.5\) Hz, 1H), 5.30 (tt, \(J = 6.1, 1.0\) Hz, 1H), 3.92 (t, \(J = 6.0\) Hz, 2H), 3.60 (d, \(J = 6.2\) Hz, 2H), 2.22 (t, \(J = 6.0\), 2H), 1.50 (s, 9H). 13\(^\text{C} \) NMR (100 MHZ, DMSO-\(d_6\)) \(\delta\) ppm : 156.21, 154.66, 141.00, 129.00, 124.12, 121.34, 118.15, 80.0, 43.57, 41.98, 28.44, 26.51. Anal. Calcd. For \(C_{17}H_{23}N_3O_3\): C, 64.33; H, 7.30; N, 13.24 Found: C, 64.30; H, 7.37; N, 13.31. mp 154°C.
**1H NMR** (400 MHz, DMSO-\(d_6\)) δ ppm: 9.49 (s, 1H), 8.35 (s, 1H), 7.64 (m, 2H), 7.40 (m, 3H), 7.21 (t, \(J=7.5\) Hz, 2H), 6.96 (tt, \(J=7.4, 2.0\) Hz, 1H), 6.08 (s, 1H).

**13C NMR** (100 MHz, DMSO-\(d_6\)) δ ppm: 163.00, 158.12, 156.55, 154.00, 141.08, 131.10, 129.02, 126.34, 125.00, 121.45, 118.19, 117.80, 115.15, 88.05.

**Anal. Calcd.** For C\(_{16}\)H\(_{12}\)N\(_2\)O\(_3\): C, 68.56; H, 4.32; N, 9.99.

**Found:** C, 68.60; H, 4.28; N, 9.93. **mp** 149°C.

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**1H NMR** (400 MHz, DMSO-\(d_6\)) δ ppm: 9.50 (s, 1H), 8.41 (s, 1H), 7.66 (dd, \(J = 7.6, 5.7\) Hz, 1H), 7.23 (m, 5H), 7.00 (td, \(J = 7.6, 5.8\) Hz, 1H), 6.79 (tdd, \(J = 7.7, 5.7, 2.0\) Hz, 1H), 6.10 (s, 1H).

**13C NMR** (100 MHz, DMSO-\(d_6\)) δ ppm: 163.21, 158.64, 155.49-155.31 (m), 154.24, 152.97, 131.36, 130.09 (d, \(J = 8.4\) Hz), 128.99 (d, \(J = 19.8\) Hz), 126.30, 125.95 (d, \(J = 2.9\) Hz), 125.08, 122.71 (d, \(J = 7.6\) Hz), 121.86 (d, \(J = 19.8\) Hz), 117.87, 115.43, 88.09. **Anal. Calcd.** For C\(_{16}\)H\(_{11}\)F\(_2\)N\(_2\)O\(_3\): C, 64.43; H, 3.72; N, 9.39. **Found:** C, 64.36; H, 3.79; N, 9.33. **mp** 178-180°C.
$^1$H NMR (400 MHz, DMSO-$d_6$) δ ppm: 9.52 (s, 1H), 9.46 (s, 1H), 7.87 (dt, $J = 9.0$, 2.0 Hz, 1H), 7.53 (m, 2H), 7.44 (m, 1H), 7.38 (td, $J = 7.6$, 5.8 Hz, 1H), 7.25 (m, 1H), 6.79 (ddt, $J = 11.0$, 7.8, 2.1 Hz, 2H), 6.20 (s, 1H), 3.50 (s, 3H).

$^{13}$C NMR (100 MHz, DMSO-$d_6$) δ ppm: 164.03, 163.29, 161.51, 158.22, 156.35, 154.11, 140.93 (d, $J = 7.6$ Hz), 131.27, 130.01 (d, $J = 7.6$ Hz), 126.24, 125.78, 117.90, 116.62 (d, $J = 2.9$ Hz), 115.50, 111.48 (d, $J = 19.8$ Hz), 106.60 (d, $J = 19.8$ Hz), 88.02, 31.00.

Anal. Calcd. For C$_{17}$H$_{14}$FN$_3$O$_2$: C, 65.59; H, 4.53; N, 13.50. Found: C, 65.52; H, 4.59; N, 13.56. mp 182°C.

$^1$H NMR (400 MHz, DMSO-$d_6$) δ ppm: 9.50 (s, 1H), 8.57 (s, 1H), 7.70 (dd, $J = 7.6$, 2.0 Hz, 1H), 7.56 (td, $J = 7.5$, 2.0 Hz, 1H), 7.30-7.22 (m, 4H), 7.00-7.09 (m, 2H), 6.15 (s, 1H). $^{13}$C NMR
(100 MHz, DMSO-$d_6$) $\delta$ ppm: 163.30, 161.51, 158.99, 158.19, 156.63, 154.43, 139.74 (d, $J = 2.9$ Hz), 131.17, 126.43, 125.21, 119.60 (d, $J = 8.4$ Hz), 117.74, 115.37, 114.55 & 114.34 (d, $J = 21$ Hz), 88.18. **Anal. Calcd.** For C$_{16}$H$_{11}$FN$_2$O$_3$: C, 64.43; H, 3.72; N, 9.39 **Found:** C, 64.40; H, 3.77; N, 9.37 **mp** 175°C.

![Structure 1](image1.png)

**$^1$H NMR** (400 MHz, DMSO-$d_6$) $\delta$ ppm: 9.38 (s, 1H), 9.04 (s, 1H), 7.73 (td, $J = 7.6$, 2.0 Hz, 1H), 7.50 (m, 2H), 7.36 (m, 1H), 7.21 (dd, $J = 7.2$, 2.1 Hz, 1H), 7.15-7.10 (m, 2H) 7.00 (dd, $J = 7.8$, 1.9 Hz, 1H), 6.12 (s, 1H), 3.44 (s, 3H), 3.85 (s, 3H). **$^{13}$C NMR** (100 MHz, DMSO-$d_6$) $\delta$ ppm: 163.03, 158.11, 156.60, 153.90, 153.07, 131.00, 129.41, 126.14, 125.33, 124.96, 124.19, 120.71, 117.43, 113.78, 115.04, 88.00, 56.63, 31.24. **Anal. Calcd.** For C$_{18}$H$_{17}$N$_3$O$_3$: C, 66.86; H, 5.30; N, 13.00 **Found:** C, 66.90; H, 5.35; N, 13.05. **mp** 158-159°C.

![Structure 2](image2.png)
$^{1}$H NMR (400 MHz, DMSO-$d_6$) δ ppm: 9.41 (s, 1H), 8.37 (s, 1H), 7.75 (dd, $J = 7.6$, 2.0 Hz, 1H) 7.48 (m, 2H), 7.37 (m, 2H), 7.22 (t, $J = 8.0$ Hz, 1H), 6.95 (dd, $J = 8.0$, 2.0 Hz, 1H), 6.62 (dd, $J = 8.0$, 2.0 Hz, 1H), 6.13 (s, 1H). 3.88 (s, 3H).

$^{13}$C NMR (100 MHz, DMSO-$d_6$) δ ppm: 162.98, 159.17, 158.04, 156.58, 153.87, 141.29, 131.00, 129.69, 126.10, 125.04, 117.38, 116.33, 115.09, 113.16, 107.45, 88.10, 56.57. Anal. Calcd. For C$_{17}$H$_{14}$N$_2$O$_4$: C, 65.80; H, 4.55; N, 9.03. Found: C, 65.84; H, 4.58; N, 9.01 mp 163°C

![Chemical Structure](image)

$^{1}$H NMR (400 MHz, DMSO-$d_6$) δ ppm: 9.40 (s, 1H), 8.90 (s, 1H), 7.70 (dd, $J = 7.6$, 2.0 Hz, 1H) 7.52 (m, 2H), 7.41 (d, $J = 7.6$ Hz, 2H), 7.27 (m, 1H), 6.83 (d, $J = 7.6$ Hz, 2H), 6.10 (s, 1H), 3.90 (s, 3H), 3.82 (s, 3H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ ppm: 163.06, 159.97, 158.05, 156.68, 154.02, 137.53, 131.05, 126.14, 125.10, 119.54, 117.33, 115.00, 114.36, 88.03, 56.56, 30.95. Anal. Calcd. For C$_{18}$H$_{17}$N$_3$O$_3$: C, 66.86; H, 5.30; N, 13.00 Found: C, 66.89; H, 5.37; N, 13.03. mp 167°C.
**1H NMR** (400 MHz, DMSO-$d_6$) $\delta$ ppm: 9.52 (s, 1H), 8.90 (s, 1H), 8.20 (d, $J = 8.6$ Hz, 2H), 7.69 (d, $J = 8.6$ Hz, 2H), 6.07 (s, 1H), 4.32 (m, 1H), 2.91 (ddd $J = 12.5$, 6.9, 1.0 Hz, 1H), 2.51 (ddd $J = 12.5$, 6.8, 1.0 Hz, 1H), 1.60 (d, 3H) 

**13C NMR** (100 MHZ, DMSO-$d_6$) $\delta$ ppm: 162.76, 158.10, 156.71, 142.44, 133.30, 119.13, 119.01, 107.68, 90.59, 76.09, 36.00, 22.08.  

**Anal. Calcd.** For C$_{14}$H$_{13}$N$_3$O$_3$: C, 61.99; H, 4.83; N, 15.49 **Found:** C, 61.94; H, 4.89; N, 15.44  

mp 138°C.

**1H NMR** (400 MHz, DMSO-$d_6$) $\delta$ ppm: 9.44 (s, 1H), 9.00 (s, 1H), 7.33 (d, $J = 7.8$ Hz, 2H), 7.00 (d, $J = 7.8$, 2H), 6.29 (s, 1H), 6.11 (s, 1H), 2.37 (s, 3H), 2.16 (s, 3H).  

**13C NMR** (100 MHZ, DMSO-$d_6$) $\delta$ ppm: 163.66, 162.95, 156.63, 152.27, 139.91, 137.54, 128.74, 118.98, 97.00, 91.00, 22.01, 21.00.  

**Anal. Calcd.** For C$_{14}$H$_{14}$N$_2$O$_3$: C, 65.11; H, 5.46; N, 10.85.  

**Found:** C, 65.16; H, 5.50; N, 10.80  

mp 125°C.
$^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ ppm: 9.69 (s, 1H), 7.96 (s, 1H), 7.30-7.16 (m, 5H), 5.67 (s, 1H), 5.01 (s, 2H), 4.33 (s, 2H). $^{13}$C NMR (100 MHZ, DMSO-$d_6$) $\delta$ ppm: 170.05, 155.53, 154.10, 141.17, 128.37, 128.19, 126.72, 90.00, 67.76, 45.01 Anal. Calcd. For C$_{12}$H$_{12}$N$_2$O$_3$: C, 62.06; H, 5.21; N, 12.06. Found: C, 62.08; H, 5.28; N, 12.00 mp 134-135°C.

$^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ ppm: 9.73 (s, 2H), 7.30 (dd, $J = 7.8$, 2.0 Hz, 2H), 7.24 (d, $J = 2.0$ Hz, 2H), 7.18 (d, $J = 7.8$, 2H), 5.96 (s, 2H), 2.24 (s, 6H). $^{13}$C NMR (100 MHZ, DMSO-$d_6$) $\delta$ ppm: 162.85, 159.00, 156.78, 152.76, 135.00, 131.78, 126.63, 117.20, 115.07, 88.98, 21.51, Anal. Calcd. For C$_{21}$H$_{16}$N$_2$O$_5$: C, 67.02; H, 4.28; N, 7.44 Found: C, 67.00; H, 4.34; N, 7.50. mp 187°C.
$^1$H NMR (400 MHz, DMSO-$d_6$) δ ppm: 7.18 (d, $J = 7.8$, 2H), 6.95 (d, $J = 2.0$ Hz, 2H), 6.75 (dd, $J = 7.8$, 2.0 Hz, 2H), 6.00 (s, 2H), 4.15 (m, 4H), 3.85 (s, 6H).

$^{13}$C NMR (100 MHz, DMSO-$d_6$) δ ppm: 161.88, 160.68, 157.14, 156.82, 153.00, 128.02, 111.00, 110.00, 103.55, 89.07, 56.23, 42.68.  

**Anal. Calcd.** For $C_{23}H_{18}N_2O_7$: C, 63.59; H, 4.18; N, 6.45. **Found:** C, 63.64; H, 4.16; N, 6.40. mp 190°C.

$^1$H NMR (400 MHz, DMSO-$d_6$) δ ppm: 9.48 (s, 1H), 8.43 (s, 1H), 7.73 (m, 2H), 7.62 (m, 3H), 7.44 (m, 2H), 7.31 (m, 2H), 7.09 (tt, $J = 7.4$, 2.0 Hz, 1H), 5.01 (m, 1H), 2.10 (d, 3H).  

$^{13}$C NMR (100 MHz, DMSO-$d_6$) δ ppm: 156.50, 141.10, 139.47, 136.67, 129.01, 129.46, 128.48, 127.12, 121.51, 118.23, 107.10, 12.22. **Anal. Calcd.** For $C_{16}H_{16}N_2O$: C, 76.16; H, 6.39; N, 11.10.  

**Found:** C, 76.19; H, 6.44; N, 11.14. mp 137°C.
\[ ^1H\text{ NMR} \text{ (400 MHz, DMSO-}d_6\text{)} \delta \text{ ppm: 9.76 (s, 1H), 9.42 (s, 1H), 8.15 (s, 1H), 7.73 (d, } J = 7.8 \text{ Hz, 2H), 7.58 (d, } J = 7.8 \text{ Hz, 2H), 5.30 (t, } J = 6.2 \text{ Hz, 1H), 3.92 (t, } J = 5.8 \text{ Hz, 2H), 3.60 (d, } J = 6.2 \text{ Hz, 2H), 2.22 (t, } J = 5.8 \text{ Hz, 2H), 2.11 (s, 3H) 1.50 (s, 9H).} \]

\[ ^13C\text{ NMR} \text{ (100 MHZ, DMSO-}d_6\text{)} \delta \text{ ppm: 170.04, 156.50, 154.66, 139.00, 137.87, 124.12, 119.13, 119.01, 80.0, 43.57, 41.98, 28.44, 26.51 23.51.} \]

**Anal. Calcd.** For C\textsubscript{19}H\textsubscript{26}N\textsubscript{4}O\textsubscript{4}: C, 60.95; H, 7.00; N, 14.96. **Found:** C, 60.91; H, 7.07; N, 14.92. **mp** 129°C.
$^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ ppm : 9.47 (s, 1H), 8.64 (s, 1H), 7.94 (d, 8.2 Hz, 2H), 7.85 (d, 8.2 Hz, 2H), 5.64 (s, 1H), 3.02 (t, $J = 6.1$ Hz, 2H), 2.75 (t, $J = 6.0$ Hz, 2H), 2.60 (s, 3H), 1.79 (m, 2H). $^{13}$C NMR (100 MHZ, DMSO-$d_6$) $\delta$ ppm : 197.86, 196.83, 156.70, 154.59, 141.54, 135.58, 128.76, 118.61, 108.86, 37.00, 28.44, 25.75, 22.89. Anal. Calcd. For C$_{13}$H$_{16}$N$_2$O$_3$ : C, 66.16; H, 5.92; N, 10.29. Found: C, 66.10; H, 5.99; N, 10.21. mp 122°C.

![Chemical Structure 1](image1)

$^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ ppm : 9.49 (s, 1H), 8.08 (s, 1H), 7.89 (d, $J = 8.8$ Hz, 2H), 7.55 (d, $J = 8.8$ Hz, 2H), 5.97 (t, $J = 6.2$ Hz, 1H), 2.20 (m, 2H), 1.87 (m, 2H), 1.60 (m, 1H), 1.52 (tt, $J = 8.2$, 7.0 Hz, 1H), 1.33 (m, 1H), 1.17 (s, 9H). $^{13}$C NMR (100 MHZ, DMSO-$d_6$) $\delta$ ppm : 156.79, 150.00, 143.44, 132.49, 132.17, 126.75 (m), 124.00, 119.69 (d, $J = 2.2$ Hz), 102.00, 45.11, 32.13, 28.00, 26.89, 26.26, 22.76. Anal. Calcd. For C$_{18}$H$_{23}$F$_3$N$_2$O : C, 63.51; H, 6.81; N, 8.23. Found: C, 63.55; H, 6.77; N, 8.28. mp 147°C.

![Chemical Structure 2](image2)
$^1$H NMR (400 MHz, DMSO-$d_6$) δ ppm: 9.51 (s, 1H), 8.70 (s, 1H), 7.73 (d, $J = 2.2$, 1H), 7.57 (dd, $J = 8.0$, 2.0 Hz, 1H), 7.40 (d, $J = 8.2$, 2H), 7.32 (d, $J = 7.8$, 1H), 7.21 (d, $J = 8.2$, 2H), 6.07 (s, 1H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ ppm: 162.86, 156.71, 153.83, 151.28, 138.90, 131.15, 130.18, 129.47, 128.35, 127.10, 121.55, 120.13, 117.84, 88.56. Anal. Calcd. For C$_{16}$H$_{10}$Cl$_2$N$_2$O$_3$: C, 55.04; H, 2.89; N, 8.02. Found: C, 55.00; H, 2.94; N, 8.05. mp 192-194°C.

$^1$H NMR (400 MHz, DMSO-$d_6$) δ ppm: 9.43 (s, 1H), 8.76 (s, 1H), 7.62 (m, 2H), 7.50 (d, $J = 7.6$ Hz, 2H), 7.44 (m, 1H), 7.25 (m, 1H), 6.90 (d, $J = 7.6$ Hz, 2H), 6.20 (s, 1H), 3.72 (t, $J = 4.8$ Hz, 4H), 3.50 (s, 3H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ ppm: 163.08, 158.00, 154.09, 151.75, 156.47, 154.09, 151.75, 136.00, 131.10, 126.21, 125.11, 120.56, 118.39, 117.35, 115.01, 88.09, 67.01, 49.79, 31.00. Anal. Calcd. For C$_{21}$H$_{22}$N$_4$O$_3$: C, 66.65; H, 5.86; N, 14.81. Found: C, 66.71; H, 5.90; N, 14.77. mp 190°C. mp 149°C.
$^1$H NMR (400 MHz, DMSO-$d_6$) δ ppm: 8.88 (s, 1H), 7.67 (d, $J = 7.6$ Hz, 2H), 7.41 (s, 1H), 7.00-7.17 (m, 3H), 4.17 (m, 2H), 2.73 (t, 2H), 2.42 (t, 2H), 2.00 (m, 2H), 1.32 (t, 3H). $^{13}$C NMR (100 MHZ, DMSO-$d_6$) δ ppm: 170.79, 152.07, 148.91, 141.00, 133.00, 129.05, 127.50, 105.12, 62.10, 31.24, 29.57, 25.19, 15.81. Anal. Calcd. For C$_{15}$H$_{18}$N$_2$O$_5$S: C, 53.24; H, 5.36; N, 8.28. Found: C, 53.31; H, 5.39; N, 8.33. mp 118°C.

7. $^1$H and $^{13}$C NMR Spectra
8. References


