Silver Oxide Mediated Novel SET Oxidative Cyclization: Stereoselective Synthesis of 3-Azabicyclo[n.1.0]alkanes

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Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 1i S91
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 1j S97
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 1k S103
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 1l S109
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 1m S115
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 1n S121
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 1o S127
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 1p S133
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2b S139
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2a S145
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2c S151
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2d S157
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2e  S163
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2f  S169
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2g  S175
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2h  S181
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2i  S187
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2j  S193
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2k  S199
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2l  S205
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2m  S211
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2n  S217
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2o  S223
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 2p  S229
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 3q  S235
Copy of $^1$H, $^{13}$C and DEPT-135 NMR spectra of compound 3j  S241
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 3k  S245
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 3o  S251
Copy of $^1$H, $^{13}$C, DEPT-135, $^1$H-$^1$H COSY & $^1$H-$^{13}$C HSQC NMR spectra of compound 4r  S257
1. General Information:

**General Methods:** All the solvents were distilled prior to use. Dry solvents were prepared according to the standard procedures. All other reagents were used as received from either Aldrich or Lancaster chemical companies. Reactions requiring inert atmosphere were carried out under argon atmosphere. Infrared (IR) spectra were recorded on a JASCO 4100 FT-IR spectrometer. $^1$H NMR spectra were measured on Bruker AVANCE 400 MHz and 500 MHz spectrometers. Chemical shifts were reported in ppm relative to solvent signals. $^{13}$C NMR spectra were recorded on Bruker 100 MHz and 125 MHz spectrometers with complete proton decoupling. Chemical shifts were reported in ppm from the residual solvent as an internal standard. The high-resolution mass spectra (HRMS) were performed on Micromass QTOF micro mass spectrometer equipped with a Harvard apparatus syringe pump. X-ray crystallographic data were recorded using Bruker-AXS Kappa CCD-Diffractometer with graphite monochromator MoKα radiation ($\lambda=0.7107$ Å). The
structures were solved by direct methods (SHELXS-97) and refined by full-matrix least squares techniques against \( F^2 \) (SHELXL-97). Hydrogen atoms were inserted from geometry consideration using the HFIX option of the program. For thin layer chromatography (TLC) analysis throughout this work, E-merck precoated TLC plates (silica gel 60 F254 grade, 0.25 mm) were used. Acme (India) silica gel (100-200 mesh) was used for column chromatography.

2. Optimization of reaction conditions:

2A. General procedure: To a stirred solution of \( \alpha \)-amidinoester 1a (1 equiv) in dry acetonitrile (3 mL/ mmol), were added DBU, I\(_2\), SET oxidant and co-oxidant and reaction mixture was stirred at room temperature until the completion of reaction as indicated by TLC. The reaction mixture was diluted with saturated Na\(_2\)S\(_2\)O\(_3\) (20 mL/ mmol) and extracted with DCM (2 X 30 mL/ mmol). The combined organic solvent was washed successively with water (2 X 20 mL) and brine solution (30 mL). The organic layers were collected, dried over anhydrous Na\(_2\)SO\(_4\), filtered and concentrated under reduced pressure. Purification of the crude product using column chromatography yielded the cyclic amidine 2a.

2B. Screening of SET oxidants

The screening of SET oxidants were performed using general procedure 2A and the results are summarized in Table 1.

Table 1. Optimization of SET oxidant for synthesis of cyclic amidine
<table>
<thead>
<tr>
<th>Entry</th>
<th>SET Oxidant (equiv)</th>
<th>Time (h)</th>
<th>Yield (%)a</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ag₂O (2)</td>
<td>24</td>
<td>48</td>
</tr>
<tr>
<td>2</td>
<td>CAN (2)</td>
<td>24</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>Mn(OAc)₃ (2)</td>
<td>24</td>
<td>5</td>
</tr>
<tr>
<td>4</td>
<td>FeCl₃ (2)</td>
<td>24</td>
<td>-</td>
</tr>
<tr>
<td>5</td>
<td>Hg(OAc)₂ (2)</td>
<td>24</td>
<td>5</td>
</tr>
<tr>
<td>6</td>
<td>AgNO₃ (2)</td>
<td>24</td>
<td>-</td>
</tr>
<tr>
<td>7</td>
<td>Ag₂CO₃ (2)</td>
<td>24</td>
<td>-</td>
</tr>
<tr>
<td>8</td>
<td>AgOAc (2)</td>
<td>24</td>
<td>-</td>
</tr>
<tr>
<td>9</td>
<td>AgBF₄ (2)</td>
<td>24</td>
<td>-</td>
</tr>
<tr>
<td>10</td>
<td>Ag₂O (3)</td>
<td>24</td>
<td>54</td>
</tr>
</tbody>
</table>

aIsolated yields.

2C. Screening of solvents

The screening of solvents were performed using general procedure 2A and the results are summarized in Table 2.

Table 2. Optimization of solvent system for synthesis of cyclic amidine

α-amidinoester

\[ \text{EtO}_2\text{C} \quad \text{N} \quad \text{Ts} \]

\[ \text{Ag}_2\text{O} \text{ (2 equiv), DBU (2.1 equiv)} \]

Solvent, rt

\[ \text{CO}_2\text{Et} \quad \text{N} \quad \text{Ts} \]

cyclic amidine
<table>
<thead>
<tr>
<th>Entry</th>
<th>Solvent</th>
<th>Time (h)</th>
<th>Yield (%)&lt;sup&gt;a&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>THF</td>
<td>24</td>
<td>37</td>
</tr>
<tr>
<td>2</td>
<td>DCM</td>
<td>24</td>
<td>5</td>
</tr>
<tr>
<td>3</td>
<td>DCE</td>
<td>24</td>
<td>10</td>
</tr>
<tr>
<td>4</td>
<td>Et&lt;sub&gt;2&lt;/sub&gt;O</td>
<td>24</td>
<td>6</td>
</tr>
<tr>
<td>5</td>
<td>1,4-dioxane</td>
<td>24</td>
<td>5</td>
</tr>
<tr>
<td>6</td>
<td>toluene</td>
<td>24</td>
<td>-</td>
</tr>
<tr>
<td>7</td>
<td>DMSO</td>
<td>24</td>
<td>30</td>
</tr>
<tr>
<td>8</td>
<td>DMF</td>
<td>24</td>
<td>25</td>
</tr>
<tr>
<td>9</td>
<td>CH&lt;sub&gt;3&lt;/sub&gt;CN</td>
<td>24</td>
<td>48</td>
</tr>
<tr>
<td>10</td>
<td>CH&lt;sub&gt;3&lt;/sub&gt;CN : H&lt;sub&gt;2&lt;/sub&gt;O (1:1)</td>
<td>24</td>
<td>10</td>
</tr>
<tr>
<td>11</td>
<td>CH&lt;sub&gt;3&lt;/sub&gt;CN : DMF (1:1)</td>
<td>24</td>
<td>8</td>
</tr>
<tr>
<td>12</td>
<td>CH&lt;sub&gt;3&lt;/sub&gt;CN: DMSO (1:1)</td>
<td>24</td>
<td>35</td>
</tr>
</tbody>
</table>

<sup>a</sup>Isolated yields.

**2D. Screening of base**

The screening of base was performed using general procedure **2A** and the results are summarized in Table 3.

**Table 3. Optimization of base for synthesis of cyclic amidine**
<table>
<thead>
<tr>
<th>Entry</th>
<th>Base (equiv)</th>
<th>Time (h)</th>
<th>Yield (%)(^a)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>potassium carbonate (2.1)</td>
<td>24</td>
<td>35</td>
</tr>
<tr>
<td>2</td>
<td>2,2(^1)-bipyridine (2.1)</td>
<td>24</td>
<td>20</td>
</tr>
<tr>
<td>3</td>
<td>pyridine (2.1)</td>
<td>24</td>
<td>16</td>
</tr>
<tr>
<td>4</td>
<td>DABCO (2.1)</td>
<td>24</td>
<td>10</td>
</tr>
<tr>
<td>5</td>
<td>DMAP (2.1)</td>
<td>24</td>
<td>5</td>
</tr>
<tr>
<td>6</td>
<td>ethylenediamine (2.1)</td>
<td>24</td>
<td>21</td>
</tr>
<tr>
<td>7</td>
<td>2,6-lutidine (2.1)</td>
<td>24</td>
<td>-</td>
</tr>
<tr>
<td>8</td>
<td>(N^1,N^1,N^2,N^2)-tetramethylthene-1,2-diamine (2.1)</td>
<td>24</td>
<td>30</td>
</tr>
<tr>
<td>9</td>
<td>4,4(^1)-bipyridine (2.1)</td>
<td>24</td>
<td>20</td>
</tr>
<tr>
<td>10</td>
<td>triethylamine (2.1)</td>
<td>24</td>
<td>-</td>
</tr>
<tr>
<td>11</td>
<td>DBN (2.1)</td>
<td>24</td>
<td>28</td>
</tr>
<tr>
<td>12</td>
<td>DBU (2.1)</td>
<td>24</td>
<td>48</td>
</tr>
<tr>
<td>13</td>
<td>DBU (3)</td>
<td>24</td>
<td>57</td>
</tr>
<tr>
<td>14</td>
<td>DBU (4)</td>
<td>24</td>
<td>69</td>
</tr>
</tbody>
</table>
2E. Screening of SET oxidant, Co-oxidant and additive

The screening of equivalents of Ag₂O, co-oxidant and I₂ were performed using general procedure 2A and the results are summarized in Table 4.

Table 4. Optimization of equivalents of Ag₂O and co-oxidant for synthesis of cyclic amidine

<table>
<thead>
<tr>
<th>Entry</th>
<th>Ag₂O (equiv)</th>
<th>Co-oxidant (equiv)</th>
<th>I₂ (equiv)</th>
<th>Time (h)</th>
<th>Yield (%)a</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.1</td>
<td>-</td>
<td>-</td>
<td>24</td>
<td>trace</td>
</tr>
<tr>
<td>2</td>
<td>0.1</td>
<td>K₂S₂O₈ (2.0)</td>
<td>-</td>
<td>24</td>
<td>14</td>
</tr>
<tr>
<td>3</td>
<td>0.1</td>
<td>K₂S₂O₈ (4.0)</td>
<td>-</td>
<td>24</td>
<td>34</td>
</tr>
<tr>
<td>4</td>
<td>0.1</td>
<td>Na₂S₂O₈ (4.0)</td>
<td>-</td>
<td>24</td>
<td>16</td>
</tr>
<tr>
<td>5</td>
<td>0.1</td>
<td>(NH₄)₂S₂O₈ (4.0)</td>
<td>-</td>
<td>24</td>
<td>11</td>
</tr>
<tr>
<td>6</td>
<td>0.1</td>
<td>O₂</td>
<td>-</td>
<td>24</td>
<td>trace</td>
</tr>
<tr>
<td>7</td>
<td>0.2</td>
<td>K₂S₂O₈ (4.0)</td>
<td>-</td>
<td>24</td>
<td>58</td>
</tr>
</tbody>
</table>
3. Experimental procedures and spectral data of all compounds

3A. Preparation of starting materials

The amine derivatives b, c, d, e, f, g, h, i, j, k, l, m, n, o, and p were prepared according to literature report (Table 5). The sulfonyl and phosphoryl azides were prepared using the reported procedure.\(^\text{11}\)

3B. General procedure for the preparation of α-amidinoester 1:

\[
\text{CO}_2\text{R}^1 \xrightarrow{\text{CuCN (15 mol %), TsN}_3 (1.2 equiv), THF, rt, 1h}} \xrightarrow{\text{amine (1.2 equiv), rt, 2h}} \text{R}^1\text{O}_2\text{C} = \text{NTs} \quad \alpha\text{-amidinoester}
\]

To a stirred solution of ethyl propiolate (1 equiv) in dry THF (3 mL/mmol), was added CuCN (15 mol %) followed by TsN\(_3\) (1.2 equiv) and the mixture was vigorously stirred at room temperature for 1h under N\(_2\) atmosphere. To this reaction mixture, amine 

\[\text{Table 5:}
\]

<p>| | | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>8</td>
<td>0.3</td>
<td>K(_2)S(_2)O(_8) (4.0)</td>
<td>-</td>
<td>24</td>
</tr>
<tr>
<td>9</td>
<td>0.4</td>
<td>K(_2)S(_2)O(_8) (4.0)</td>
<td>-</td>
<td>24</td>
</tr>
<tr>
<td>10</td>
<td>0.3</td>
<td>K(_2)S(_2)O(_8) (3.5)</td>
<td>-</td>
<td>24</td>
</tr>
<tr>
<td>11</td>
<td>0.3</td>
<td>K(_2)S(_2)O(_8) (4.5)</td>
<td>-</td>
<td>24</td>
</tr>
<tr>
<td>12</td>
<td>0.3</td>
<td>K(_2)S(_2)O(_8) (4.0)</td>
<td>0.6</td>
<td>18</td>
</tr>
<tr>
<td>13</td>
<td>0.3</td>
<td>K(_2)S(_2)O(_8) (4.0)</td>
<td>0.9</td>
<td>12</td>
</tr>
<tr>
<td>14</td>
<td>0.3</td>
<td>K(_2)S(_2)O(_8) (4.0)</td>
<td>1.2</td>
<td>18</td>
</tr>
</tbody>
</table>

\(^a\)Isolated yields.
derivative (1.2 equiv) was added and the resultant mixture was stirred at room temperature until the completion of reaction as indicated by TLC. The reaction mixture was diluted with saturated NH₄Cl (20 mL) and extracted with dichloromethane (2 X 30 mL). The combined organic solvent was washed successively with water (2 X 20 mL) and brine solution (30 mL). The organic layers were collected, dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. Purification of the crude product using column chromatography yielded the α-amidinoester 1 in quantitative yields.

Table 5. Preparation of α-amidinoesters:

<table>
<thead>
<tr>
<th>Entry</th>
<th>Amine</th>
<th>Time (h)</th>
<th>α-amidinoester</th>
<th>Isolated Yield (%)</th>
<th>Entry</th>
<th>Amine</th>
<th>Time (h)</th>
<th>α-amidinoester</th>
<th>Isolated Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>a</td>
<td>2</td>
<td>EtO₂C-N⁺Ts≡N⁺</td>
<td>94</td>
<td>9</td>
<td>b</td>
<td>2</td>
<td>EtO₂C-N⁺Ts≡N⁺</td>
<td>72</td>
</tr>
<tr>
<td>2</td>
<td>b</td>
<td>2</td>
<td>EtO₂C-N⁺Ts≡N⁺</td>
<td>81</td>
<td>10</td>
<td>c</td>
<td>2</td>
<td>EtO₂C-N⁺Ts≡N⁺</td>
<td>80</td>
</tr>
<tr>
<td>3</td>
<td>c</td>
<td>2</td>
<td>EtO₂C-N⁺Ts≡N⁺</td>
<td>80</td>
<td>11</td>
<td>d</td>
<td>2</td>
<td>EtO₂C-N⁺Ts≡N⁺</td>
<td>81</td>
</tr>
<tr>
<td>4</td>
<td>d</td>
<td>2</td>
<td>EtO₂C-N⁺Ts≡N⁺</td>
<td>81</td>
<td>12</td>
<td>e</td>
<td>3</td>
<td>EtO₂C-N⁺Ts≡N⁺</td>
<td>77</td>
</tr>
<tr>
<td>5</td>
<td>e</td>
<td>2</td>
<td>EtO₂C-N⁺Ts≡N⁺</td>
<td>82</td>
<td>13</td>
<td>f</td>
<td>3</td>
<td>EtO₂C-N⁺Ts≡N⁺</td>
<td>72</td>
</tr>
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</table>
Preparation of Compound 1a: The coupling reaction of ethyl propiolate (1 g, 10.19 mmol), tosyl azide (2.4 g, 12.23 mmol) and amine a (1.18 g, 12.23 mmol) was carried out using general procedure 3B, the product 1a obtained in 94% yield (3.48 g) as a colourless crystals; m.p 57-59 °C; IR (neat): 3073, 2982, 2934, 1737, 1599, 1564, 1471, 1425, 1328, 1148, 1092, 1035, 934, 829 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.76(d, J = 8.4 Hz, 2.01H), 7.21(d, J = 8.0 Hz, 2.03H), 5.79-5.67(m, 2.09H), 5.27-5.14(m, 4.26H), 4.15-4.09(m, 4.24H), 4.07(s, 2.12H), 3.91-3.89(m, 2.08H), 2.37(s, 3.04H), 1.23(t, J = 7.2 Hz, 3.07H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 166.7, 160.8, 142.1, 140.8, 131.4, 130.9, 129.1, 126.4, 118.2, 118.1, 62.0, 51.2, 50.7, 36.3, 21.5, 14.1; HRMS(ESI): m/z calcd for C₁₈H₂₅N₂O₄S[M+H]⁺: 365.1530, found: 365.1521.

Preparation of Compound 1b: The coupling reaction of ethyl propiolate (500 mg, 5.09 mmol), tosyl azide (1.2 g, 6.11 mmol) and amine b (691 mg, 6.11 mmol) was carried out using general procedure 3B, the product 1b obtained in 81% yield (1.56 g) as a yellow oil; IR (neat): 2969, 2924, 2857, 2361, 1732, 1642, 1577, 1457, 1277, 1157, 1086, 1020, 890 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.77(d, J = 8.0 Hz, 2.02H), 7.22(d, J = 8.0 Hz, 2.03H), 5.87-5.78(m, 1.04H), 5.27-5.15(m, 2.12H), 4.14(q, J = 7.2 Hz, 2.16H), 4.01-3.98(m, 3.90H), 2.36(s, 3.00H), 1.43(s,
9.07H), 1.24(t, J = 7.2 Hz, 3.28H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ ppm 167.5, 160.5, 141.9, 140.7, 134.4, 129.1, 126.2, 116.9, 61.7, 60.9, 48.4, 38.9, 28.4, 21.5, 14.0; HRMS(ESI): m/z calcd for C$_{19}$H$_{28}$N$_2$O$_4$NaS [M+Na]$^+$: 403.1662, found: 403.1652.

**Preparation of Compound 1c:** The coupling reaction of ethyl propiolate (615 mg, 6.27 mmol), tosyl azide (1.48 g, 7.52 mmol) and amine c (1.7 g, 7.52 mmol) was carried out using general procedure 3B, the product 1c obtained in 80% yield (2.47 g) as a pale yellow semi solid; $\text{IR ( }$\text{neat}$\text{): }$3059, 2985, 2930, 1736, 1602, 1552, 1421, 1269, 1191, 1147, 1090, 1026, 959, 847 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ ppm 7.79-7.71(m, 0.41H), 7.59-7.54(m, 2.57H), 7.48-7.46(m, 1.10H), 7.34-7.05(m, 8.3H), 5.80-5.68(m, 1.53H), 5.28-5.24(m, 1.06H), 5.22-5.09(m, 2.27H), 4.79(s, 1.93H), 4.73(s, 0.22H), 4.53(s, 1.10H), 4.18-4.07(m, 6.40H), 4.02(s, 0.88H), 3.91-3.84(m, 2.18H), 3.84-3.82(m, 0.23H), 2.36(s, 1.98H), 2.31(s, 3.00H), 1.26-1.18(m, 4.46H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ ppm 166.4, 161.3, 161.2, 159.6, 142.1, 142.0, 140.5, 140.3, 134.4, 133.8, 133.6, 133.3, 132.6, 131.2, 130.9, 130.4, 130.3, 129.8, 129.7, 129.3, 129.0, 128.9, 128.8, 128.8, 128.2, 128.1, 127.9, 127.7, 127.1, 126.5, 126.4, 126.3, 126.2, 123.0, 122.5, 118.4, 118.3, 62.0, 61.9, 52.0, 51.9, 51.6, 51.2, 36.4, 36.2, 21.4, 21.3, 14.0, 13.9; HRMS(ESI): m/z calcd for C$_{22}$H$_{26}$BrN$_2$O$_4$S [M+H]$^+$: 493.0797, found: 493.0773.

**Preparation of Compound 1d:** The coupling reaction of ethyl propiolate (1.03 g, 10.49 mmol), tosyl azide (2.4 g, 12.28 mmol) and amine d (2.74 g, 12.28 mmol) was carried out using general procedure 3B, the product 1d obtained in 81% yield (4.17 g) as a pale yellow semi solid; $\text{IR ( }$\text{neat}$\text{): }$3059, 2986, 2873, 2360, 2338, 1961, 1909, 1736, 1598, 1539, 1450, 1419, 1327, 1271, 1150, 1088, 1028, 965, 860 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ ppm 7.72-7.70(m, 0.34H), 7.52-7.46(m, 2.60H), 7.41-7.39(m, 1.09H), 7.24-7.229(m, 1.71H), 7.226-7.08(m, 3.84H), 5.73-5.58(m, 1.58H), 5.21-5.06(m, 3.44H), 4.72(s, 1.99H), 4.66(s, 0.168H), 4.47-4.45(m, 1.05H), 4.13-4.00(m, 6.16H), 3.95(s, 0.87H), 3.85-3.84(m, 2.17H), 3.77-3.75(m, 0.16H), 2.29(s, 1.86H), 2.24(s, 3.00H), 1.19-1.11(m, 4.76H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ ppm 167.5, 167.0, 162.0, 160.7, 159.1, 142.0, 141.8, 140.6, 137.8, 137.8, 137.6, 137.1, 132.7, 131.3, 130.9, 129.2, 129.2,
Preparation of Compound 1e: The coupling reaction of ethyl propiolate (630 mg, 6.42 mmol), tosyl azide (1.52 g, 7.71 mmol) and amine e (1.47 g, 7.71 mmol) was carried out using general procedure 3B, the product 1e obtained in 82% yield (2.41 g) as a yellow semi solid; IR (neat): 3059, 2984, 2928, 2362, 1736, 1642, 1601, 1551, 1493, 1444, 1370, 1327, 1278, 1145, 1090, 1036, 930, 842 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.79-7.71(m, 2.29H), 7.23-7.18(m, 2.062H), 6.76-6.71(m, 1.04H), 6.66-6.59(m, 2.23H), 5.92-5.88(m, 2.17H), 5.77-5.70(m, 1.06H), 5.47(s, 0.11H), 5.27-5.13(m, 2.19H), 4.66(s, 1.32H), 4.51-4.36(m, 0.79H), 4.13-4.08(m, 4.47H), 3.94-3.82(m, 1.32H), 2.36(s, 3.37H), 1.22-1.18(m, 3.00H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 166.2, 166.1, 160,8, 160.6, 148.1, 147.6, 147.3, 146.8, 141.8, 140.4, 140.3, 130.9, 130.3, 129.1, 129.0, 128.7, 128.1, 125.9, 120.9, 119.6, 117.8, 117.7, 108.3, 107.9, 107.7, 106.7, 101.1, 100.7, 61.6, 51.0, 50.8, 50.6, 50.1, 36.1, 35.8, 21.1, 13.6; HRMS(ESI): m/z calcd for C₂₈H₃₁N₂O₄S [M+H]⁺: 491.2005, found: 491.2026.

Preparation of Compound 1f: The coupling reaction of ethyl propiolate (1.3 g, 13.33 mmol), tosyl azide (3.13 g, 15.9 mmol) and amine f (2 g, 15.9 mmol) was carried out using general procedure 3B, the product 1f obtained in 86% yield (4.46 g) as a greenish yellow oil; IR (neat): 2962, 2918, 2868, 1737, 1643, 1539, 1455, 1423, 1323, 1275, 1197, 1145, 1090, 1024, 974, 813 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.73-7.67(m, 2.10H), 7.17-7.13(m, 2.01H), 5.77-5.67(m, 1.04H), 5.18-4.80(m, 2.34H), 4.16-4.06(m, 0.96H), 4.06-3.97(m, 2.80H), 3.95-3.94(m, 2.13H), 3.798-3.795(m, 0.98H), 2.31-2.29(m, 3.25H), 1.78-1.76(m, 2.08H), 1.57-1.42(m, 6.32H), 1.19-1.11(m, 3.12H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 166.8, 166.7, 161.5, 159.6, 141.9, 141.8, 140.9, 140.8, 133.2, 132.7, 129.0, 128.9, 126.3, 126.2, 117.2, 116.4, 61.8, 61.7, 59.9, 58.8, 46.9, 46.7, 37.0, 36.4, 29.8, 28.8, 23.9, 23.9, 21.4, 14.0, 13.9; HRMS(ESI): m/z calcd for C₂₀H₂₉N₂O₄S [M+H]⁺: 393.1848, found: 393.1863.
Preparation of Compound 1g: The coupling reaction of ethyl propiolate (623 mg, 6.35 mmol), tosyl azide (1.5 g, 7.62 mmol) and amine g (1.45 g, 7.62 mmol) was carried out using general procedure 3B, the product 1g obtained in 76% yield (2.21 g) as a pale yellow semi solid; IR (neat): 3059, 2980, 2911, 2856, 2661, 1738, 1643, 1600, 1536, 1452, 1415, 1323, 1278, 1145, 1090, 1029, 966, 916, 845, 814 cm⁻¹; \(^1\)H NMR (400 MHz, CDCl₃): \(\delta\) ppm 7.63(d, \(J = 8.0\) Hz, 2.0H), 7.07(d, \(J = 8.0\) Hz, 2.03H), 5.76-5.69(m, 0.88H), 5.08(d, \(J = 10.4\) Hz, 0.82H), 4.98(d, \(J = 17.2\) Hz, 1.09H), 4.26(s, 1.11H), 4.07-3.96(m, 5.86H), 2.21(s, 3.07H), 2.01.91(m, 2.23H), 1.72-1.68(m, 4.06H), 1.62-1.44(m, 8.16H), 1.08(t, \(J = 7.2\) Hz, 3.06H); \(^1\)C NMR (100 MHz, CDCl₃): \(\delta\) ppm 166.5, 160.7, 141.4, 140.4, 134.0, 128.4, 125.7, 116.4, 62.4, 61.2, 46.8, 38.2, 37.2, 36.8, 32.0, 30.0, 27.0, 26.2, 20.9, 13.5; HRMS(ESI): \(m/z\) calcd for C₂₅H₃₅N₂O₄S [M+H]^+: 459.2318, found: 459.2315.

Preparation of Compound 1h: The coupling reaction of ethyl propiolate (842 mg, 8.58 mmol), tosyl azide (2.03 g, 10.3 mmol) and amine h (1.5 g, 10.3 mmol) was carried out using general procedure 3B, the product 1h obtained in 79% yield (2.85 mg) as a brown semi solid; IR (neat): 3065, 2983, 2927, 2873, 2361, 2337, 1737, 1600, 1551, 1457, 1425, 1327, 1278, 1184, 1145, 1088, 1026, 969, 844 cm⁻¹; \(^1\)H NMR (400 MHz, CDCl₃): \(\delta\) ppm 7.82-7.76(m, 2.75H), 7.23-7.17(m, 4.15H), 6.94(s, 1.66H), 6.85(s, 1.01H), 5.78-5.71(m, 1.35H), 5.25-5.14(m, 2.69H), 4.82(s, 1.81H), 4.73-4.62(m, 0.64H), 4.21-4.14(m, 1.37H), 4.12-4.05(m, 4.57H), 3.94(s, 1.98H), 2.36(s, 4.14H), 1.21(t, \(J = 6.8\) Hz, 0.97H), 1.16(t, \(J = 6.8\) Hz, 3.00H); \(^1\)C NMR (100 MHz, CDCl₃): \(\delta\) ppm 165.9, 165.8, 160.2, 160.1, 141.7, 140.2, 140.1, 137.1, 136.8, 130.9, 130.1, 128.6, 127.1, 126.8, 126.2, 126.0, 125.9, 125.8, 125.7, 125.6, 117.8, 117.7, 61.4, 61.3, 50.3, 50.1, 46.6, 46.1, 35.9, 35.7, 20.9, 13.5, 13.5; HRMS(ESI): \(m/z\) calcd for C₂₀H₂₅N₂O₄S₂ [M+H]^+: 421.1256, found: 421.1259.

Preparation of Compound 1i: The coupling reaction of ethyl propiolate (244 mg, 2.49 mmol), tosyl azide (589 mg, 2.98 mmol) and amine i (598 mg, 2.98 mmol) was carried out using general procedure 3B, the product 1i obtained in 72% yield (823 mg) as a brown semi solid; IR (neat):
3369, 2921, 2856, 2362, 2339, 1734, 1599, 1551, 1457, 1427, 1328, 1270, 1192, 1141, 1087, 1021, 849, 813, 747 cm⁻¹; 1H NMR (400 MHz, CDCl₃): δ ppm 8.42(s, 0.56H), 8.32(s, 0.97H), 7.73(d, J = 8.0 Hz, 2.00H), 7.67(d, J = 8.4 Hz, 1.15H), 7.37(d, J = 7.6 Hz, 0.63H), 7.28-7.23(m, 2.68H), 7.16-7.10(m, 3.45H), 7.08-6.99(m, 2.48H), 6.89-6.83(m, 2.60H), 5.70-5.52(m, 1.62H), 5.13-5.04(m, 1.97H), 5.00-4.96(m, 1.32H), 4.10-4.04(m, 3.26H), 4.00(s, 1.69H), 3.99-3.93(m, 1.56H), 3.75(s, 1.16H), 3.59(t, J = 7.2 Hz, 2.07H), 3.46(t, J = 7.2 Hz, 1.16H), 2.93-2.88(m, 3.20H), 2.29-2.20(m, 4.89H), 1.17(t, J = 6.8 Hz, 3.11H), 1.08(t, J = 7.2 Hz, 1.67H); 13C NMR (100 MHz, CDCl₃): δ ppm 166.7, 160.5, 160.3, 142.2, 142.1, 140.8, 140.7, 136.3, 136.3, 131.3, 131.0, 129.2, 129.1, 127.1, 126.6, 126.4, 126.3, 122.8, 122.7, 122.3, 121.9, 119.6, 119.2, 118.6, 118.0, 117.9, 117.8, 111.9, 111.8, 111.3, 110.6, 61.9, 61.8, 52.3, 51.2, 50.7, 49.2, 36.5, 35.8, 24.4, 22.4, 21.5, 21.5, 14.1, 13.9. HRMS(ESI): m/z calcd for C₂₅H₃₅N₃O₄S [M+H]+: 468.1957, found: 468.1958.

Preparation of Compound 1j: The coupling reaction of ethyl propiolate (774 mg, 7.9 mmol), tosyl azide (1.86 g, 9.48 mmol) and amine j (1.79 g, 9.48 mmol) was carried out using general procedure 3B, the product 1j obtained in 80% yield (2.9 g) as a pale yellow semi solid; IR (neat): 3058, 2959, 2927, 2871, 2363, 2342, 1739, 1642, 1634, 1604, 1550, 1493, 1454, 1425, 1365, 1326, 1276, 1195, 1144, 1089, 1024, 971, 848 cm⁻¹; 1H NMR (400 MHz, CDCl₃): δ ppm 7.84(d, J = 8.4 Hz, 0.79H), 7.70(d, J = 8.4 Hz, 1.91H), 7.36-7.30(m, 1.39H), 7.30-6.25(m, 5.97H), 7.17(d, J = 8.0 Hz, 2.93H), 5.63-5.33(m, 1.34H), 5.41-5.33(m, 1.34H), 4.78(s, 1.92H), 4.57(s, 0.79H), 4.21(s, 1.94H), 4.16-4.11(m, 4.61H), 3.87(d, J = 4.8 Hz, 1.83H), 2.39(s, 1.35H), 2.35(s, 3.0H), 2.05-2.00(m, 2.16H), 1.95(q, J = 6.8 Hz, 0.81H), 1.44-1.29(m, 3.05), 1.27-1.20(m, 4.57H), 0.92-0.85(m, 4.43H); 13C NMR (100 MHz, CDCl₃): δ ppm 166.1, 166.1, 160.7, 160.4, 141.6, 141.5, 140.5, 140.3, 135.3, 134.9, 134.8, 134.5, 128.7, 128.6, 128.6, 128.1, 127.7, 127.2, 127.0, 126.0, 125.9, 125.9, 122.4, 122.0, 61.4, 61.4, 50.9, 50.8, 50.5, 50.0, 36.1, 35.8, 33.8, 33.7, 21.7, 21.6, 21.0, 21.0, 13.6, 13.3; HRMS(ESI): m/z calcd for C₂₅H₃₂N₂O₄NaS [M+Na]+: 479.1980, found: 479.1993.
Preparation of Compound 1k: The coupling reaction of ethyl propiolate (765 mg, 7.8 mmol), tosyl azide (1.84 g, 9.36 mmol) and amine k (2.08 g, 9.36 mmol) was carried out using general procedure 3B, the product 1k obtained in 81% yield (3.1 g) as a yellow semi solid; IR (neat): 3056, 2986, 2932, 2685, 2522, 2411, 2362, 2306, 1736, 1644, 1551, 1493, 1451, 1425, 1365, 1327, 1267, 1144, 1090, 1024, 970 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.76(d, J = 8.0 Hz, 1.30H), 7.66(d, J = 8.4 Hz, 2.11H), 7.32-7.07(m, 21.53H), 6.39(s, 0.73H), 6.359-6.356(m, 0.90H), 6.02-5.98(m, 0.92H), 5.98-5.95(m, 0.77H), 4.77(s, 1.99H), 4.53(s, 1.21H), 4.21-4.18(m, 3.27H), 4.11(s, 1.24H), 4.08-4.0(m, 5.60H), 2.27(s, 3.0H), 2.26(s, 1.90H), 1.16-1.11(m, 5.16H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 166.5, 160.9, 160.6, 141.8, 141.7, 140.4, 140.3, 136.0, 135.3, 134.4, 133.0, 132.8, 128.9, 128.8, 128.7, 128.4, 128.3, 128.2, 128.0, 127.9, 127.5, 127.4, 127.3, 126.2, 126.1, 126.0, 122.0, 121.8, 61.6, 61.6, 51.5, 51.2, 50.7, 50.3, 36.1, 36.0, 21.1, 13.7, 13.7; HRMS(ESI): m/z calcd for C₂₈H₃₁N₂O₄S [M+H]⁺: 491.2005, found: 491.1988.

Preparation of Compound 1l: The coupling reaction of ethyl propiolate (100 mg, 1.1 mmol), tosyl azide (241 mg, 1.22 mmol) and amine l (297 mg, 1.22 mmol) was carried out using general procedure 3B, the product 1l obtained in 77% yield (403 mg) as a pale yellow semi solid; IR (neat): 2922, 2850, 2798, 2367, 2339, 1735, 1648, 1547, 1491, 1453, 1428, 1364, 1276, 1145, 1090, 1023, 855, 813 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.83(d, J = 8.0 Hz, 0.765H), 7.72(d, J = 8.4 Hz, 2.062H), 7.38-7.29(m, 1.236H), 7.27-7.21(m, 6.315H), 7.18-7.14(m, 2.955H), 5.13(t, J = 6.8 Hz, 0.398H), 5.07-5.04(m, 2.471H), 4.76(s, 1.927H), 4.52(s, 0.738H), 4.20(s, 1.916H), 4.18-4.08(m, 4.541H), 3.86(d, J = 6.0 Hz, 1.950H), 2.38(s, 1.315H), 2.35(s, 3.0H), 2.08-1.95(m, 6.353H), 1.69-1.65 (m, 4.207H), 1.61-1.59 (m, 4.773H), 1.55-1.52(m, 2.925H), 1.45(s, 1.197H), 1.25-1.20(m, 4.267H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 166.4, 160.7, 160.5, 141.8, 141.8, 141.3, 141.0, 140.8, 140.6, 135.5, 134.8, 131.9, 131.5, 128.9, 128.9, 128.8, 128.4, 128.0, 127.6, 127.4, 126.3, 126.2, 126.2, 123.7, 123.3, 117.7, 117.4, 61.7, 61.6, 51.2, 51.2, 46.6, 46.5, 39.3, 39.2, 36.4, 36.1, 26.1, 25.9, 25.6, 25.6, 21.3, 17.6, 17.6, 16.1, 13.9, 13.8; HRMS(ESI): m/z calcd for C₂₉H₃₉N₂O₄S [M+H]⁺: 511.2631, found: 511.2642.
Preparation of Compound 1m: The coupling reaction of ethyl propiolate (300 mg, 3.05 mmol), phosphoryl azide (721 mg, 3.66 mmol) and amine a (355 mg, 3.66 mmol) was carried out using general procedure 3B, the product 1m obtained in 72% yield (973 mg) as a yellow semi solid; IR (neat): 3057, 2984, 2933, 2363, 2313, 1735, 1581, 1488, 1421, 1327, 1259, 1201, 1060, 997, 929, 740 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.28-7.19(m, 8.24H), 7.10-7.06(m, 1.95H), 5.75-5.66(m, 1.02H), 5.59-5.49(m, 1.00H), 5.20(dd, J = 10.4 Hz, J = 0.8 Hz, 1.01H), 5.12-5.03(m, 1.04H), 4.10(q, J = 7.2 Hz, 2.12H), 4.02(s, 2.00H), 3.95(d, J = 5.2 Hz, 1.99H), 3.87-3.85(m, 1.99H), 1.17(t, J = 7.2 Hz, 3.04H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 167.1, 162.5, 162.4, 151.6, 151.6, 131.4, 131.0, 124.2, 120.6, 117.6, 117.5, 61.7, 50.5, 50.4, 38.5, 38.4, 13.9; HRMS(ESI): m/z calcd for C₂₃H₂₈N₂O₅P [M+H]^+: 443.1730, found: 443.1725.

Preparation of Compound 1n: The coupling reaction of ethyl propiolate (1.26 g, 12.91 mmol), tosyl azide (3.06 g, 15.46 mmol) and amine n (2.5 g, 15.5 mmol) was carried out using general procedure 3B, the product 1n obtained in 76% yield (4.1 g) as a pale yellow oil; IR (neat): 3056, 2982, 1736, 1558, 1448, 1282, 1158, 1089, 1027, 1158, 1089, 1027, 961, 855 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.70-7.58(m, 1.16H), 7.52(d, J = 8.0 Hz, 1.04H), 7.24-7.17(m, 1.36H), 7.11-7.06(m, 1.48H), 7.01(d, J = 7.6 Hz, 2.07H), 5.62-5.44(m, 1.00H), 4.98-4.92(m, 1.00H), 4.87-4.76(m, 1.06H), 4.66(s, 1.00H), 4.50-4.40(m, 1.08H), 4.07(s, 1.00H), 4.02-3.95(m, 3.01H), 3.42(t, J = 7.2 Hz, 0.87H), 3.28-3.16(m, 1.14H), 2.29-2.10(m, 5.13H), 1.08(t, J = 7.2 Hz, 3.03H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 166.2, 160.4, 160.3, 141.8, 141.6, 140.5, 140.3, 135.4, 134.5, 134.2, 133.0, 128.8, 128.6, 128.3, 127.9, 127.2, 127.1, 126.1, 125.9, 118.2, 117.0, 61.6, 61.5, 52.6, 51.0, 49.0, 47.8, 36.1, 35.9, 32.1, 30.6, 21.1, 21.1, 13.7; HRMS(ESI): m/z calcd for C₂₃H₂₉N₂O₄S [M+H]^+: 429.1848, found: 429.1849.

Preparation of Compound 1o: The coupling reaction of ethyl propiolate (864 mg, 8.8 mmol), tosyl azide (2.08 g, 10.56 mmol) and amine o (1.85 g, 10.56 mmol) was carried out using general procedure 3B, the
product 1o obtained in 80% yield (3.11 g) as a colourless semi solid; IR (neat): 3059, 2983, 2935, 2362, 2308, 1736, 1648, 1601, 1553, 1487, 1450, 1367, 1269, 1148, 1089, 1027, 962, 865, 816, 737 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.72(d, J = 8.0 Hz, 1.05H), 7.55(d, J = 8.0 Hz, 0.98H), 7.26-7.19(m, 1.73H), 7.137-7.13(m, 3.72H), 7.04(brs, 2.11H), 4.69-4.68(m, 1.48H), 4.58(d, J = 6.8 Hz, 1.03H), 4.48-4.45(m, 1.59H), 4.08-4.00(m, 4.12H), 3.46(t, J = 7.6 Hz, 1.04H), 3.27(t, J = 7.6 Hz, 1.00H), 2.26-2.23(m, 3.23H), 2.17-2.11(m, 2.14H), 1.59(s, 1.47H), 1.47(s, 1.62H), 1.14-1.09(m, 3.16H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 166.2, 166.1, 160.1, 160.0, 141.8, 141.8, 141.6, 140.7, 140.5, 140.3, 135.4, 134.5, 128.8, 128.7, 128.7, 128.3, 127.9, 127.2, 126.1, 126.0, 112.9, 112.0, 61.6, 61.5, 52.3, 51.1, 48.2, 47.0, 36.2, 35.8, 35.8, 34.0, 22.2, 21.9, 21.1, 21.1, 13.7; HRMS(ESI): m/z calcd for C₂₄H₃₁N₂O₄S [M+H]^+: 443.1999, found: 443.1988.

**Preparation of Compound 1p:** The coupling reaction of ethyl propiolate (754 mg, 7.69 mmol), tosyl azide (1.81 g, 9.22 mmol) and amine p (1.48 g, 9.22 mmol) was carried out using general procedure 3B, the product 1p obtained in 81% yield (2.66 g) as a yellow semi solid; IR (neat): 3056, 2986, 2935, 2362, 2315, 1736, 1638, 1608, 1553, 1487, 1427, 1324, 1267, 1191, 1149, 1090, 1025, 964, 890, 817, 741 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.82(d, J = 8.0 Hz, 1.91H), 7.70(d, J = 8.0 Hz, 1.01H), 7.21-7.12(m, 7.63H), 7.06(d, J = 7.2 Hz, 1.13H), 7.01(d, J = 7.2 Hz, 2.14H), 5.73-5.60(m, 1.46H), 5.20-5.03(m, 3.01H), 4.13-4.08(m, 3.17H), 4.05-4.00(m, 2.76H), 3.76-3.75(m, 2.76H), 3.56(t, J = 7.2 Hz, 1.96H), 3.45(t, J = 7.2 Hz, 0.96H), 2.81-2.76(m, 2.91H), 2.32-2.30(m, 4.40H), 1.20(t, J = 7.2 Hz, 3.0H), 1.14(t, J = 7.2 Hz, 1.59H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 166.2, 166.1, 160.1, 159.8, 141.7, 141.6, 140.5, 140.4, 138.0, 136.9, 131.1, 130.6, 128.7, 128.6, 128.5, 128.4, 128.3, 128.1, 126.6, 126.1, 125.9, 117.4, 117.4, 61.4, 51.9, 51.3, 50.6, 49.8, 35.9, 35.2, 34.1, 32.4, 21.0, 21.0, 13.6, 13.5; HRMS(ESI): m/z calcd for C₂₃H₂₉N₂O₄S [M+H]^+: 429.1848, found: 429.1859.
3C. General procedure for the preparation of cyclic amidine 2 from α-amidinoester 1:

![Chemical Reaction Diagram]

To a stirred solution of α-amidinoester 1 (1 equiv) in dry acetonitrile (3 mL/ mmol), were added DBU (4 equiv), Ag₂O (30 mol%), I₂ (0.9 equiv), K₂S₂O₈ (4 equiv) and reaction mixture was stirred at room temperature until the completion of reaction as indicated by TLC. The reaction mixture was diluted with saturated Na₂S₂O₃ (20 mL/ mmol) and extracted with DCM (2 × 30 mL/ mmol). The combined organic solvent was washed successively with water (2 × 20 mL) and brine solution (30 mL). The organic layers were collected, dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. Purification of the crude product using column chromatography yielded the cyclic amidine 2.
Table S1. Substrate scope for silver(I)-catalyzed synthesis of cyclic amidines

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<th>Entry</th>
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<th>Product</th>
<th>Yield (%)$^a$</th>
<th>Entry</th>
<th>α-Amidinoester</th>
<th>Time (h)</th>
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<th>Yield (%)$^b$</th>
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Preparation of Compound 2a: The reaction of \( \alpha \)-amidinoester 1a (128 mg, 0.35 mmol), DBU (213 mg, 1.4 mmol), I\(_2\) (80 mg, 0.31 mmol), Ag\(_2\)O (24 mg, 0.1 mmol) and K\(_2\)S\(_2\)O\(_8\) (378 mg, 1.4 mmol) was carried out using general procedure 3C, the product 2a obtained in 88% yield (111 mg) as a pale yellow crystals; m.p 62-64 °C; IR (neat): 3057, 2976, 2931, 2870, 1728, 1571, 1485, 1294, 1192, 1147, 1081 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) ppm 7.75(d, \( J = 8.0 \) Hz, 2H), 7.20(d, \( J = 8.0 \) Hz, 2H), 5.60-5.50(m, 1H), 5.13-5.03(m, 2H), 4.29-4.14(m, 2H), 3.99-3.94(m, 1H), 3.85-3.79(m, 1H), 3.70(dd, \( J = 11.6 \) Hz, 4.4 Hz, 1H), 3.29(d, \( J = 11.6 \) Hz, 1H), 2.36(s, 3H), 2.23(brs, 2H), 1.27(t, \( J = 7.2 \) Hz, 3H), 0.91(t, \( J = 10 \) Hz, 1H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \) ppm 167.7, 164.2, 141.8, 141.2, 130.7, 129.0, 126.1, 119.4, 62.1, 49.6, 47.4, 34.9, 24.5, 21.5, 19.0, 13.9. HRMS(ESI): m/z calcd for C\(_{18}\)H\(_{22}\)N\(_2\)O\(_4\)NaS [M+Na]\(^+\): 385.1198, found: 385.1193.

Preparation of Compound 2b: The reaction of \( \alpha \)-amidinoester 1b (600 mg, 1.57 mmol), DBU (957 mg, 6.28 mmol), I\(_2\) (358 mg, 1.41 mmol), Ag\(_2\)O (109 mg, 0.47 mmol) and K\(_2\)S\(_2\)O\(_8\) (1.69 g, 6.28 mmol) was carried out using general procedure 3C, the product 2b obtained in 76% yield (454 mg) as a pale yellow crystals; m.p 106-108 °C; IR (neat): 2978, 2926, 1730, 1565, 1459, 1381, 1286, 1179, 1149, 1088, 1017, 965, 910, 860 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) ppm 7.71(d, \( J = 8.0 \) Hz, 2H), 7.18(d, \( J = 7.6 \) Hz, 2H), 4.25-4.09(m, 2H), 3.77(dd, \( J = 12.0 \) Hz, 6.0 Hz, 1H), 3.44(d, \( J = 12.0 \) Hz, 1H), 2.33(s, 3H), 2.17-2.08(m, 2H), 1.26(s, 9H), 1.24(t, \( J = 7.2 \) Hz, 3H), 0.79(t, \( J = 4.4 \) Hz, 1H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \) ppm 167.9, 163.5, 141.5, 141.3, 129.0, 125.9, 61.8, 56.7, 48.7, 35.8, 27.4, 23.5, 21.4, 18.2, 13.8; HRMS(ESI): m/z calcd for C\(_{19}\)H\(_{26}\)N\(_2\)O\(_4\)NaS [M+Na]\(^+\): 401.1511, found: 401.1500.
Preparation of Compound 2c: The reaction of α-amidinoester 1c (520 mg, 1.05 mmol), DBU (639 mg, 4.2 mmol), I₂ (240 mg, 0.94 mmol), Ag₂O (73 mg, 0.31 mmol) and K₂S₂O₈ (1.13 g, 4.2 mmol) was carried out using general procedure 3C, the product 2c obtained in 88% yield (457 mg) as a pale yellow semisolid; IR (neat): 3060, 2982, 2930, 2362, 2338, 1732, 1488, 1442, 1383, 1289, 1184, 1147, 1090, 1025, 903, 855 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.76(d, J = 8.0 Hz, 2H), 7.45-7.42 (m, 1H), 7.19(d, J = 8.0 Hz, 2H), 7.10-7.04 (m, 2H), 6.98-6.96 (m, 1H), 4.84(d, J = 14.8 Hz, 1H), 4.33(d, J = 14.8 Hz, 1H), 4.29-4.16 (m, 2H), 3.63-3.59 (m, 1H), 3.14 (d, J = 11.6 Hz, 1H), 2.36(s, 3H), 2.21-2.17 (m, 2H), 1.28(t, J = 7.2 Hz, 3H), 0.94(t, J = 3.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 167.6, 164.3, 141.9, 141.1, 134.2, 133.0, 130.5, 129.8, 129.0, 127.8, 126.2, 123.8, 62.0, 49.4, 48.2, 34.7, 24.6, 21.4, 18.7, 13.8; HRMS(ESI): m/z calcd for C₂₂H₂₂BrN₂O₄S [M+H]⁺: 491.0640, found: 491.0642.

Preparation of Compound 2d: The reaction of α-amidinoester 1d (500 mg, 1 mmol), DBU (620 mg, 4.07 mmol), I₂ (232 mg, 0.91 mmol), Ag₂O (71 mg, 0.3 mmol) and K₂S₂O₈ (1.1 g, 4 mmol) was carried out using general procedure 3C, the product 2d obtained in 83% yield (416 mg) as a colourless semisolid; IR (neat): 3060, 2979, 2927, 2360, 1732, 1637, 1563, 1454, 1391, 1290, 1153, 1089, 1021, 907, 851 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.52(d, J = 8.0 Hz, 2H), 7.35-7.30 (m, 3H), 7.23-7.19 (m, 1H), 7.17-7.14 (m, 4H), 7.06(d, J = 8.0 Hz, 2H), 6.85(d, J = 6.8 Hz, 2H), 6.60(s, 1H), 4.30-4.16 (m, 2H), 3.51(dd, J = 12.0 Hz, 4.8 Hz, 1H), 3.22(d, J = 11.6 Hz, 1H), 2.34(s, 1H), 2.22-2.21 (m, 2H), 1.30(t, J = 7.2 Hz, 3H), 0.93(t, J = 10.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 167.6, 164.6, 141.5, 141.1, 138.0, 136.5, 128.9, 128.8, 128.7, 128.6, 128.0, 127.8, 127.7, 125.8, 62.0, 61.1, 47.9, 34.9, 24.6, 21.3, 18.3, 13.8; HRMS(ESI): m/z calcd for C₂₈H₂₉N₂O₄S [M+H]⁺: 489.1848, found: 489.1839.

Preparation of Compound 2e: The reaction of α-amidinoester 1e (944 mg, 2.05 mmol), DBU (1.25 g, 8.23 mmol), I₂ (470 mg, 1.85 mmol), Ag₂O (143 mg, 0.61 mmol) and K₂S₂O₈ (2.2 g, 8.23 mmol) was carried out using general procedure 3C, the product 2e obtained in 82% yield (776 mg) as a colorless crystalline solid;
m.p 95.5-97 °C IR (neat): 3052, 2983, 2304, 1731, 1576, 1443, 1267, 1181, 1149, 1084, 897 cm⁻¹; ¹H NMR (400 MHz, CDCl₃):  δ ppm 7.73(d, J = 8.0 Hz, 2H), 7.18(d, J = 8.0 Hz, 2H), 6.51(d, J = 8.0 Hz, 1H), 6.38(dd, J = 8.0 Hz, 1.6 Hz, 1H), 6.27(d, J = 1.6 Hz, 1H), 5.83(q, J = 1.6 Hz, 2H), 4.60(d, J = 14.4 Hz, 1H), 4.241-4.11(m, 2H), 3.99(d, J = 14.4 Hz, 1H), 3.59(dd, J = 11.6 Hz, 5.6 Hz, 1H), 3.18(d, J = 11.6 Hz, 1H), 2.33(s, 3H), 2.18-2.10(m, 2H), 1.25(t, J = 7.2 Hz, 3H), 0.75(t, J = 4.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 167.5, 163.9, 147.9, 147.2, 141.9, 140.9, 129.0, 128.4, 125.9, 121.7, 108.2, 108.0, 101.0, 61.8, 49.0, 48.0, 34.6, 24.3, 21.3, 18.3, 13.7; HRMS(ESI): m/z calcd for C₂₃H₂₄N₂O₆NaS [M+Na]⁺: 479.1253, found: 479.1252.

Preparation of Compound 2f: The reaction of α-amidinoester 1f (400 mg, 1.01 mmol), DBU (620 mg, 4.07 mmol), I₂ (232 mg, 0.91 mmol), Ag₂O (71 mg, 0.3 mmol) and K₂S₂O₈ (1.1 g, 4.07 mmol) was carried out using general procedure 3C, the product 2f obtained in 88% yield (352 mg) as a brown semi solid; IR (neat): 2960, 2851, 2824, 2362, 1733, 1649, 1562, 1458, 1398, 1284, 1146, 1089, 1015, 910, 853, 752 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.75(d, J = 8.0 Hz, 2H), 7.20(d, J = 8.0 Hz, 2H), 4.49-4.45(m, 1H), 4.26-4.14(m, 2H), 3.72-3.68(m, 1H), 3.30(d, J = 11.6 Hz, 1H), 2.36(s, 3H), 2.24-2.18(m, 2H), 1.91-1.85(m, 1H), 1.68-1.47(m, 6H), 1.29-1.25(m, 3H), 1.20-1.07(m, 1H), 0.84-0.79(m, 1H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 167.8, 164.4, 141.7, 141.5, 129.0, 126.0, 62.0, 55.1, 46.3, 35.2, 29.6, 28.2, 24.1, 23.8, 21.5, 18.6, 13.9; HRMS(ESI): m/z calcd for C₂₀H₂₇N₂O₄S [M+H]⁺: 391.1692, found: 391.1689.

Preparation of Compound 2g: The reaction of α-amidinoester 1g (667 mg, 1.45 mmol), DBU (882 mg, 5.8 mmol), I₂ (331 mg, 1.30 mmol), Ag₂O (101 mg, 0.43 mmol) and K₂S₂O₈ (1.56 g, 5.8 mmol) was carried out using general procedure 3C, the product 2g obtained in 87% yield (576 mg) as a pale yellow semi solid; IR (neat): 3056, 2984, 2918, 2857, 1730, 1563, 1453, 1424, 1267, 1180, 1148, 1090, 1017, 913, 862, 738 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.52(d, J = 8.4 Hz, 2H), 6.96(d, J = 8.4 Hz, 2H), 4.05-3.90(m, 2H), 3.86(brs, 1H), 3.71(dd, J = 11.6 Hz, 6.0 Hz, 1H), 3.55(d, J = 11.2 Hz, 1H), 2.12(s, 3H), 2.07-2.02(m, 2H), 1.94-1.91(m, 1H), 1.66-1.59(brm, 5H) 1.48-1.43(brm, 7H), 1.28(d, J = 12.4 Hz, 1H), 1.05(t, J = 7.2 Hz, 3H), 0.59(t, J = 5.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ
ppm 167.6, 163.9, 141.3, 141.2, 128.5, 125.4, 61.4, 59.4, 49.7, 38.0, 37.2, 34.0, 32.1, 31.8, 30.9, 30.1, 27.0, 26.5, 24.6, 21.0, 18.3, 13.5; **HRMS(ESI):** m/z calcd for C$_{25}$H$_{33}$N$_2$O$_4$S [M+H]$^+$: 457.2161, found: 457.2189.

**Preparation of Compound 2h:** The reaction of α-amidinoester 1h (943 mg, 2.24 mmol), DBU (1.36 g, 8.96 mmol), I$_2$ (511 mg, 2 mmol), Ag$_2$O (155 mg, 0.67 mmol) and K$_2$S$_2$O$_8$ (2.42 g, 8.96 mmol) was carried out using general procedure 3C, the product 2h obtained in 76% yield (714 mg) as an orange-yellow oil; **IR (neat):** 3054, 2989, 2924, 2689, 2416, 2304, 1732, 1582, 1577, 1491, 1427, 1268, 1148, 1089, 902 cm$^{-1}$; **$^1$H NMR (400 MHz, CDCl$_3$):** δ ppm 7.77(d, $J = 8.0$ Hz, 2H), 7.20(d, $J = 8.4$ Hz, 2H), 7.09(dd, $J = 4.8$ Hz, 0.8 Hz, 1H), 6.77(dd, $J = 5.2$ Hz, 3.6 Hz, 1H), 4.93(d, $J = 14.8$ Hz, 1H), 4.28-4.13(m, 3H), 3.68(dd, $J = 11.2$ Hz, 5.6 Hz, 1H), 3.29(d, $J = 11.2$ Hz, 1H), 2.36(s, 3H), 2.22-2.14(m, 2H), 1.28(t, $J = 6.8$ Hz, 3H), 0.81(t, $J = 4.4$ Hz, 1H); **$^{13}$C NMR (100 MHz, CDCl$_3$):** δ ppm 167.5, 163.7, 141.9, 141.0, 136.6, 129.0, 127.4, 126.8, 126.3, 126.0, 62.0, 48.9, 42.7, 34.6, 24.3, 21.4, 18.2, 13.8; **HRMS(ESI):** m/z calcd for C$_{20}$H$_{23}$N$_2$O$_4$S$_2$ [M+H]$^+$: 419.1099, found: 419.1086.

**Preparation of Compound 2i:** The reaction of α-amidinoester 1i (350 mg, 0.74 mmol), DBU (455 mg, 2.99 mmol), I$_2$ (108 mg, 0.67 mmol), Ag$_2$O (52 mg, 0.22 mmol) and K$_2$S$_2$O$_8$ (808 mg, 2.99 mmol) was carried out using general procedure 3C, the product 2i obtained in 80% yield (277 mg) as a pale yellow semi-solid; **IR (neat):** 3410, 3056, 2925, 2856, 2361, 1729, 1642, 1577, 1457, 1276, 1144, 1088, 1018, 910, 746 cm$^{-1}$; **$^1$H NMR (400 MHz, CDCl$_3$):** δ ppm 8.40(s, 1H), 7.87(d, $J = 8.4$ Hz, 2H), 7.34(dd, $J = 3.6$ Hz, 7.6 Hz, 2H), 7.26(d, $J = 8.0$ Hz, 2H), 7.15(t, $J = 7.2$ Hz, 1H), 7.03(t, $J = 7.2$ Hz, 1H), 6.75(d, $J = 1.6$ Hz, 1H), 4.31-4.09(m, 2H), 3.80-3.73(m, 1H), 3.49(dd, $J = 11.6$ Hz, 4.8 Hz, 1H), 3.40(dt, $J = 13.6$ Hz, 7.2 Hz, 1H), 3.01(d, $J = 11.6$ Hz, 1H), 2.83(t, $J = 7.2$ Hz, 2H), 2.37(s, 3H), 2.06(s, 2H), 1.28(t, $J = 7.2$ Hz, 3H), 0.61(t, $J = 10.0$ Hz, 1H); **$^{13}$C NMR (100 MHz, CDCl$_3$):** δ ppm 167.9, 164.2, 142.1, 141.3, 136.2, 129.2, 127.0, 126.2, 122.5, 122.1, 119.4, 118.4, 111.5, 111.4, 62.1, 50.9, 45.2, 35.0, 24.6, 22.6, 21.5, 18.7, 13.9; **HRMS(ESI):** m/z calcd for C$_{25}$H$_{28}$N$_3$O$_4$S [M+H]$^+$: 466.1801, found: 466.1797.
Preparation of Compound 2j: The reaction of α-amidinoester 1j (743 mg, 1.62 mmol), DBU (990 mg, 6.5 mmol), I2 (372 mg, 1.46 mmol), Ag2O (113 mg, 0.48 mmol) and K2S2O8 (1.76 g, 6.5 mmol) was carried out using general procedure 3C, the product 2j obtained in 74% yield (551 mg) as a pale yellow crystal; m.p 108-110 °C; IR (neat): 3058, 2962, 2871, 2304, 1742, 1574, 1570, 1567, 1485, 1454, 1293, 1181, 1149, 1084, 891 cm⁻¹. 1H NMR (400 MHz, CDCl3): δ ppm 7.78(d, J = 7.6 Hz, 2H), 7.24-7.20(m, 3H), 7.17-7.13(m, 2H), 6.93(d, J = 7.6 Hz, 2H), 4.72(d, J = 14.4 Hz, 1H), 4.28-4.11(m, 3H), 3.63-3.59(m, 1H), 3.21(d, J = 11.6 Hz, 1H), 2.39(s, 3H), 2.06-2.02(m, 2H), 1.83-1.74(m, 1H), 1.71-1.64(m, 1H), 1.51-1.44(m, 1H), 1.29-1.26(m, 3H), 1.05(q, J = 6.0 Hz, 1H), 0.93(t, J = 7.6 Hz, 3H); 13C NMR (100 MHz, CDCl3): δ ppm 167.8, 165.4, 141.8, 141.4, 135.1, 129.1, 128.8, 128.2, 126.2, 61.4, 49.6, 48.4, 37.7, 34.3, 29.5, 29.2, 22.3, 21.5, 14.0, 13.9; HRMS(ESI): m/z calcd for C25H31N2O4S [M+H]+: 455.2005, found: 455.2027.

Preparation of Compound 2k: The reaction of α-amidinoester 1k (341 mg, 0.69 mmol), DBU (423 mg, 2.78 mmol), I2 (158 mg, 0.62 mmol), Ag2O (48 mg, 0.2 mmol) and K2S2O8 (751 mg, 2.78 mmol) was carried out using general procedure 3C, the product 2k obtained in 84% yield (286 mg) as a colorless crystals; m.p 126-128 °C; IR (neat): 3065, 2928, 2857, 1737, 1572, 1487, 1453, 1291, 1245, 1144, 1087, 916, 873 cm⁻¹. 1H NMR (400 MHz, CDCl3): δ ppm 7.89(d, J = 8.0 Hz, 2H), 7.49(d, J = 7.6 Hz, 2H), 7.35-7.31(m, 3H), 7.28-7.26(m, 3H), 7.22(d, J = 7.2 Hz, 1H), 7.19-7.15(m, 2H), 6.98(d, J = 7.2 Hz, 2H), 4.86(d, J = 14.4 Hz, 1H), 4.20(d, J = 14.8 Hz, 1H), 4.14-4.06(m, 1H), 4.03-3.95(m, 1H), 3.83-3.78(m, 1H), 2.74(t, J = 6.0 Hz, 1H), 2.46(d, J = 6.0 Hz, 1H), 2.42(s, 3H), 1.09(t, J = 7.2 Hz, 3H); 13C NMR (100 MHz, CDCl3): δ ppm 166.5, 164.6, 141.9, 141.2, 134.9, 133.2, 129.5, 129.2, 128.8, 128.2, 128.1, 127.4, 126.2, 61.3, 49.3, 48.5, 39.5, 37.0, 27.6, 21.5, 13.6; HRMS(ESI): m/z calcd for C28H28N2O4NaS [M+Na]+: 511.1669, found: 511.1667.
**Preparation of Compound 2l:** The reaction of α-amidinoester 1l (477 mg, 0.93 mmol), DBU (568 mg, 3.73 mmol), I₂ (213 mg, 0.84 mmol), Ag₂O (64 mg, 0.28 mmol) and K₂S₂O₈ (1 g, 3.73 mmol) was carried out using general procedure 3C, the product 2l obtained in 63% yield (296 mg) as a brown semi solid; IR (neat): 3255, 3059, 2959, 2930, 2871, 1729, 1574, 1490, 1455, 1287, 1148, 1088, 1016, 942, 882, 813 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.80 (d, J = 8 Hz, 2H), 7.23 (d, J = 8 Hz, 3H), 7.16 (t, J = 7.6 Hz, 2H), 7.09 (d, J = 7.2 Hz, 2H), 4.70 (d, J = 14.4 Hz, 1H), 4.44 (t, J = 6.8 Hz, 1H), 4.30 (d, J = 14.4 Hz, 1H), 4.26-4.18 (m, 1H), 4.11-4.05 (m, 1H), 3.64-3.61 (m, 1H), 3.15 (d, J = 11.6 Hz, 1H), 2.40 (s, 3H), 2.05 (d, J = 6 Hz, 2H), 1.88-1.79 (m, 1H), 1.60 (s, 3H), 1.56 (s, 3H), 1.45 (s, 3H), 1.23 (t, J = 6.8 Hz, 3H), 1.18-1.13 (m, 1H), 0.73-0.65 (m, 1H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 168.3, 163.6, 141.8, 134.5, 131.8, 129.8, 129.1, 128.8, 128.2, 126.5, 126.5, 123.5, 61.6, 48.8, 47.1, 43.8, 35.6, 35.0, 30.0, 25.6, 24.4, 21.6, 19.3, 17.7, 13.8. HRMS (ESI): m/z calcd for C₂₉H₃₇N₂O₄S [M+H]⁺: 509.2474, found: 509.2458.

**Preparation of Compound 2m:** The reaction of α-amidinoester 1m (422 mg, 0.95 mmol), DBU (581 mg, 3.82 mmol), I₂ (218 mg, 0.85 mmol), Ag₂O (66 mg, 0.28 mmol) and K₂S₂O₈ (1 g, 3.82 mmol) was carried out using general procedure 3C, the product 2m obtained in 54% yield (226 mg) as a pale yellow oil; IR (neat): 3069, 2983, 2930, 2363, 2250, 1731, 1619, 1587, 1491, 1255, 1196, 1009, 929 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.26-7.16 (m, 8H), 7.15-7.01 (m, 2H), 5.43-5.32 (m, 1H), 5.07 (dd, J = 10.4 Hz, 1.2 Hz, 1H), 5.02-4.97 (m, 1H), 4.26-4.18 (m, 1H), 4.13-4.05 (m, 1H), 3.92 (dd, J = 15.2 Hz, 6.0 Hz, 1H), 3.67-3.58 (m, 2H), 3.22 (d, J = 11.2 Hz, 1H), 2.19-2.11 (m, 2H), 1.19 (t, J = 7.2 Hz, 3H), 0.80 (t, J = 4.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 167.8, 166.2, 152.0, 151.9, 151.7, 151.6, 130.9, 129.2, 129.2, 124.1, 124.0, 120.6, 120.6, 120.4, 120.4, 118.9, 61.9, 49.2, 46.7, 35.0, 24.3, 19.5, 13.9; HRMS (ESI): m/z calcd for C₂₅H₂₆N₂O₅P [M+H]⁺: 441.1579, found: 441.1580.
Preparation of Compound 2n: The reaction of α-amidinoester 1n (474 mg, 1.1 mmol), DBU (674 mg, 4.42 mmol), I2 (253 mg, 0.99 mmol), Ag2O (77 mg, 0.33 mmol) and K2S2O8 (1.08 g, 4.42 mmol) was carried out using general procedure 3C, the product 2n obtained in 72% yield (334 mg) as a pale yellow oil and 10% recovered starting material 27; IR (neat): 2930, 2861, 1731, 1547, 1492, 1448, 1282, 1149, 1089, 1022, 941, 820 cm⁻¹; ¹H NMR (400 MHz, CDCl3): δ ppm 7.67(d, J = 8.0 Hz, 2H), 7.18-7.17(m, 3H), 7.12-7.10(m, 4H), 4.97(d, J = 14.4 Hz, 1H), 4.49(d, J = 14.8 Hz, 1H), 4.17-4.06(m, 2H), 3.42(t, J = 12.8 Hz, 1H), 2.98(d, J = 12.8 Hz, 1H), 2.30(s, 3H), 2.24-2.18(m, 1H), 2.11-2.08(m, 1H) 1.87-1.80(m, 1H), 1.13(t, J = 7.2 Hz, 3H), 1.04(t, J = 6.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl3): δ ppm 170.0, 161.3, 141.7, 141.2, 135.6, 129.0, 128.7, 127.8, 127.7, 126.3, 61.8, 53.5, 48.4, 27.7, 26.0, 25.8, 25.6, 21.4, 13.9; HRMS (ESI): m/z calcd for C23H26N2O4NaS [M+Na]⁺: 449.1511, found: 449.1511.

Preparation of Compound 2o: The reaction of α-amidinoester 1o (398 mg, 0.9 mmol), DBU (547 mg, 3.6 mmol), I2 (205 mg, 0.81 mmol), Ag2O (62 mg, 0.27 mmol) and K2S2O8 (973 mg, 3.6 mmol) was carried out using general procedure 3C, the product 2o obtained in 65% yield (260 mg) as a pale yellow oil and 8% recovered starting material 28; IR (neat): 3058, 2983, 2935, 2865, 2304, 1731, 1544, 1485, 1427, 1267, 1154, 1084, 891,753 cm⁻¹; ¹H NMR (400 MHz, CDCl3): δ ppm 7.67(d, J = 8.0 Hz, 2H), 7.21-7.19(m, 3H), 7.16-7.13(m, 2H), 7.11(d, J = 8.0 Hz, 2H), 5.11(d, J = 14.8 Hz, 1H), 4.37(d, J = 14.8 Hz, 1H), 4.17-4.03(m, 2H), 3.44(td, J = 12.8 Hz, 2.0 Hz, 1H), 2.97(dt, J = 12.8 Hz, 2.8 Hz, 1H), 2.30(s, 3H), 1.96(d, J = 5.6 Hz, 1H), 1.94-1.90(m, 1H), 1.39-1.32(m, 1H), 1.20-1.18(m, 3H), 1.14(t, J = 7.2 Hz, 3H), 0.80(t, J = 6.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl3): δ ppm 167.5, 162.4, 141.7, 141.2, 135.7, 129.0, 128.7, 127.8, 126.3, 61.7, 53.8, 47.4, 36.4, 32.0, 31.2, 29.8, 21.5, 18.7, 14.1; HRMS (ESI): m/z calcd for C24H29N2O4S [M+H]⁺: 441.1848, found: 441.1866.

Preparation of Compound 2p: The reaction of α-amidinoester 1p (9.16 g, 21.3 mmol), DBU (13 g, 85 mmol), I2 (4.88 g, 19.2 mmol), Ag2O (1.48 g, 6.4 mmol) and K2S2O8 (23.1 g, 85 mmol) was carried out using general
procedure 3C, the product 2p obtained in 84% yield (7.62 g) as a colourless semi solid; **IR (neat):** 3057, 2984, 2930, 2874, 1731, 1577, 1490, 1455, 1271, 1184, 1148, 1090, 1018, 907, 854 cm⁻¹; **^1H NMR (400 MHz, CDCl₃):** δ ppm 7.82(d, J = 8.0 Hz, 2H), 7.25(d, J = 7.6 Hz, 2H), 7.16(brs, 3H), 6.91(brs, 2H), 4.29-4.12(m, 2H), 3.77-3.71(m, 1H), 3.48(dd, J = 11.2 Hz, 5.2 Hz, 1H), 3.36-3.29(m, 1H), 3.05(d, J = 11.6 Hz, 1H), 2.68(t, J = 6.8 Hz, 2H), 2.37(s, 3H), 2.09-2.04(m, 2H), 1.28(t, J = 7.2 Hz, 3H), 0.58(t, J = 3.6 Hz, 1H); **^13C NMR (100 MHz, CDCl₃):** δ ppm 167.6, 164.1, 141.8, 141.3, 137.4, 129.0, 128.5, 128.4, 126.6, 126.0, 61.7, 50.6, 45.6, 34.7, 32.7, 24.4, 21.3, 18.4, 13.7; **HRMS(ESI):** m/z calcd for C₂₃H₂₆N₂O₄NaS [M+Na⁺]: 449.1505, found: 449.1493.

3D. Plausible mechanism for the formation of cyclic amidine 2a from α-amidinoester 1a

A plausible mechanism for the formation of cyclic amidine 2a from α-amidinoester 1a is shown in below scheme. The key precursor 1a was derived from coupling reaction of alkyne, azide and amine. Treatment of α-amidinoester 1a with DBU would generate carbanion A, which on exposed with SET oxidant Ag₂O would lead to the formation of amidyl-radical B. Subsequent 5-exo-trig radical cyclization of amidyl-radical B would lead to the corresponding cyclic amidyl-radical C. Notably, potassium persulfate would readily oxidize Ag(I) to Ag(II) and promotes the catalytic process. The intermediate C would then undergo iodination followed by carbanion cyclization furnish the cyclic amidine 2a.
3E. Procedure for an efficient reduction of cyclic amidine to 3-azabicyclo[n.1.0]alkanes:

A round bottomed flask charged with dry THF and lithium aluminium hydride was allowed to reflux for 15 minutes. To this reaction mixture, was added cyclic amidine 2 in dry THF (3 mL) over five minutes and the reaction mixture was stirred at reflux until the completion of reaction as indicated by TLC. The reaction mixture was quenched by addition of moist Na₂SO₄ and extracted with EtOAc (2 X 30 mL). The combined EtOAc solvent was washed successively with water (2 X 20 mL) and brine solution (30 mL). The organic layers were collected, dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. Purification of the crude product using column chromatography yielded the 3-azabicyclo[n.1.0]alkanes 3.

Preparation of Compound 3q: The reaction of cyclic amidine 2q (4.43 g, 10.75 mmol) with LiAlH₄ (3.17 g, 86 mmol), was carried out using general procedure 3E, the product 3q obtained in 78% yield (1.69 g) as a colourless oil; IR (neat): 3398, 3055, 2904, 2792, 1724, 1608, 1454, 1373, 1265, 1149, 1030, 891, 775, 702 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.33-7.24(m, 5H), 3.74-3.68(m, 1H), 3.64-3.59(m, 3H), 3.03(d, J = 8.4 Hz, 1H), 2.96(d, J = 8.8 Hz, 1H), 2.44(d, J = 8.4 Hz, 2H), 1.70(brs, 1H), 1.28-1.26(m, 1H), 1.13(t, J = 4.4 Hz, 1H), 0.47(dd, J = 4 Hz, 7.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 139.1, 128.6, 128.1, 126.8, 66.1, 59.2, 56.6, 54.9, 29.8, 20.3, 12.5. HRMS(ESI): m/z calcd for C₁₃H₁₈NO [M+H]⁺: 204.1310, found: 204.1314.

Preparation of Compound 3j: The reaction of cyclic amidine 2j (1.93 g, 4.24 mmol) with LiAlH₄ (1.25 g, 34 mmol), was carried out using general procedure 3E, the product 3j obtained in 74% yield (770 mg) as a
colourless oil; **IR (neat):** 3418, 3081, 3029, 2955, 2928, 2859, 2787, 1740, 1652, 1494, 1456, 1379, 1289, 1249, 1156, 1027, 783 cm\(^{-1}\); **\(^1\)H NMR (400 MHz, CDCl\(_3\)):** δ ppm 7.26-7.17(m, 5H), 3.70(s, 2H), 3.55(q, J = 13.2 Hz, 2H), 2.97(d, J = 8.8 Hz, 1H), 2.89(d, J = 8.8 Hz, 1H), 2.40-2.35(m, 2H), 1.43-1.34(m, 4H), 1.25-1.14(m, 2H), 0.94(s, 1H), 0.87(t, J = 7.2 Hz, 3H); **\(^{13}\)C NMR (100 MHz, CDCl\(_3\)):** δ ppm 139.4, 129.8, 128.7, 128.2, 126.9, 126.5, 63.4, 59.3, 58.5, 55.5, 33.6, 30.3, 27.3, 24.7, 23.2, 14.2. **HRMS(ESI):** m/z calcd for C\(_{16}\)H\(_{24}\)NO [M+H]\(^+\): 246.1852, found: 246.1854.

**Preparation of Compound 3k:** The reaction of cyclic amidine 2k (708 mg, 1.45 mmol) with LiAlH\(_4\) (428 mg, 11.6 mmol), was carried out using general procedure 3E, the product 3k obtained in 81% yield (327 mg) as a colourless oil; **IR (neat):** 3421, 3086, 3033, 2956, 2921, 2869, 2789, 1738, 1651, 1499, 1451, 1370, 1279, 1254, 1159, 1031, 782 cm\(^{-1}\); **\(^1\)H NMR (400 MHz, CDCl\(_3\)):** δ ppm 7.19-7.15(m, 3H), 7.19-7.15(m, 3H), 7.19-7.15(m, 3H), 7.19-7.15(m, 3H), 3.67(s, 2H), 3.54(q, J = 12 Hz, 2H), 3.25(d, J = 8.8 Hz, 1H), 3.10(d, J = 8.8 Hz, 1H), 2.81(d, J = 4 Hz, 1H), 2.56-2.51(m, 2H), 1.87(t, J = 3.6 Hz, 1H), 1.17(brs, 1H); **\(^{13}\)C NMR (100 MHz, CDCl\(_3\)):** δ ppm 139.4, 138.3, 128.7, 128.6, 128.4, 128.3, 127.0, 126.1, 62.4, 59.0, 58.4, 55.1, 37.2, 28.6, 25.4. **HRMS(ESI):** m/z calcd for C\(_{19}\)H\(_{22}\)NO [M+H]\(^+\): 280.1696, found: 280.1696.

**Preparation of Compound 3o:** The reaction of cyclic amidine 2o (1.2 g, 2.72 mmol) with LiAlH\(_4\) (805 mg, 21.8 mmol), was carried out using general procedure 3E, the product 3o obtained in 77% yield (485 mg) as a colourless oil; **IR (neat):** 3392, 3061, 3030, 2977, 2924, 2873, 2810, 2769, 1653, 1605, 1493, 1451, 1362, 1286, 1251, 1159, 1125, 1078, 1030 cm\(^{-1}\); **\(^1\)H NMR (400 MHz, CDCl\(_3\)):** δ ppm 7.33-7.21(m, 5H), 3.63(d, J = 8.4 Hz, 1H), 3.60(d, J = 10.4 Hz, 1H), 3.48(d, J = 4.4 Hz, 1H), 3.45(d, J = 2.8 Hz, 1H), 3.00(d, J = 11.2 Hz, 1H), 2.62(d, J = 11.6 Hz, 1H), 2.42-2.36(m, 1H), 2.15-2.09(m, 1H), 1.86-1.77(m, 2H), 1.20(s, 3H), 0.68(d, J = 4.4 Hz, 1H), 0.31(d, J = 4 Hz, 1H); **\(^{13}\)C NMR (100 MHz, CDCl\(_3\)):** δ ppm 136.9, 129.4, 128.3, 127.4, 66.7, 62.1, 56.4, 49.0, 31.2, 27.1, 22.1, 21.8, 19.4. **HRMS(ESI):** m/z calcd for C\(_{15}\)H\(_{22}\)NO [M+H]\(^+\): 232.1701, found: 232.1697.
**3F. Procedure for unusual dialkylation of amidine using Grignard reagent:**

To a stirred solution of cyclic amidine 2 (1 equiv) in dry THF (3 mL/ mmol) at room temperature under N₂ atmosphere was added MeMgBr (2.1 equiv). The reaction mixture was stirred at room temperature until the completion of reaction as indicated by TLC. Then reaction mixture was cooled to 0 °C, diluted with saturated Na₂SO₄ (20 mL/ mmol) and extracted with EtOAc (2 X 30 mL/ mmol). The combined EtOAc solvent was washed successively with water (2 X 20 mL) and brine solution (30 mL). The organic layers were collected, dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. Purification of the crude product using column chromatography yielded the dialkylated 3-azabicyclo[n.1.0]alkane 4.
Table S2. Substrate scope for nucleophilic addition of Grignard reagent to amidine

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\(^a\)Isolated yield.
Preparation of Compound 4q: The reaction of cyclic amidine 2q (610 mg, 1.53 mmol) with Grignard reagent generated \textit{in situ} from C$_6$H$_{13}$Br (531 mg, 3.21 mmol) and Mg (78 mg, 3.21 mmol) was carried out using general procedure 3F, the product 4q obtained in 58% yield (358 mg) as a colourless oil; \textbf{IR (neat)}: 3066, 3026, 2929, 2859, 2701, 1722, 1600, 1533, 1363, 1298, 1198, 1159, 1116, 1039, 955, 727 cm$^{-1}$; \textbf{1H NMR (400 MHz, CDCl$_3$)}: $\delta$ ppm 7.29-7.18 (m, 5H), 3.93 (d, $J = 13.6$ Hz, 1H), 3.63 (s, 3H), 3.33 (d, $J = 13.6$ Hz, 1H), 2.70 (s, 2H), 2.11-2.05 (m, 1H), 1.99 (brs, 1H), 1.88-1.80 (m, 1H), 1.64-1.60 (m, 1H), 1.53-1.44 (m, 4H), 1.27 (brs, 14H), 1.13 (dd, $J = 3.2$ Hz, 8 Hz, 1H), 0.91-0.85 (m, 6H); \textbf{13C NMR (100 MHz, CDCl$_3$)}: $\delta$ ppm 173.4, 140.8, 128.2, 128.1, 126.6, 64.4, 52.1, 51.4, 51.2, 38.2, 35.9, 34.8, 32.1, 31.8, 30.6, 30.5, 29.8, 25.7, 25.1, 24.4, 22.8, 22.8, 18.7, 14.2. \textbf{HRMS(ESI)}: m/z calcd for C$_{26}$H$_{42}$NO$_2$ [M+H]$^+$: 400.3216, found: 400.3217.

Preparation of Compound 4r: The reaction of cyclic amidine 2r (723 mg, 1.69 mmol) with MeMgBr (1.18 mL, 3.56 mmol), was carried out using general procedure 3F, the product 4r obtained in 47% yield (463 mg) as a colourless oil; \textbf{IR (neat)}: 3057, 2982, 2931, 2903, 2799, 2684, 1713, 1603, 1490, 1454, 1426, 1374, 1314, 1263, 1205, 1166, 1093, 1030, 897, 732 cm$^{-1}$; \textbf{1H NMR (400 MHz, CDCl$_3$)}: $\delta$ ppm 7.29-7.17 (m, 5H), 4.16-4.04 (m, 2H), 3.82 (d, $J = 13.6$ Hz, 1H), 3.11 (d, $J = 13.6$ Hz, 1H), 2.68 (d, $J = 8.8$ Hz, 1H), 2.41 (dd, $J = 3.2$ Hz, 8.8 Hz, 1H), 1.97-1.93 (m, 1H), 1.47-1.45 (m, 1H), 1.33 (s, 3H), 1.24 (t, $J = 6.8$ Hz, 3H), 1.18 (s, 3H), 0.94 (dd, $J = 3.6$ Hz, 8.4 Hz, 1H); \textbf{13C NMR (100 MHz, CDCl$_3$)}: $\delta$ ppm 173.0, 140.8, 128.2, 128.2, 126.7, 60.1, 59.6, 51.0, 49.9, 37.9, 23.6, 22.7, 16.8, 16.3, 14.3. \textbf{HRMS(ESI)}: m/z calcd for C$_{17}$H$_{24}$NO$_2$ [M+H]$^+$: 274.1802, found: 274.1803.

Preparation of Compound 4rx: The reaction of cyclic amidine 2r (595 mg, 1.44 mmol) with Grignard reagent generated \textit{in situ} from 1,4-dibromobutane (654 mg, 3.03 mmol) and Mg (147 mg, 6.06 mmol) was carried out using general procedure 3F, the product 4rx obtained in 41% yield (169 mg) as a colourless oil; \textbf{IR (neat)}: 3057, 2953, 2930, 2856, 2792, 1723, 1635, 1625, 1452, 1440, 1365, 1264, 1199, 1165, 1106, 1039, 1029, 965, 732 cm$^{-1}$; \textbf{1H NMR (400 MHz, CDCl$_3$)}: $\delta$ ppm 7.29-7.17 (m, 5H), 4.16-4.04 (m, 2H), 3.82 (d, $J = 13.6$ Hz, 1H), 3.11 (d, $J = 13.6$ Hz, 1H), 2.68 (d, $J = 8.8$ Hz, 1H), 2.41 (dd, $J = 3.2$ Hz, 8.8 Hz, 1H), 1.97-1.93 (m, 1H), 1.47-1.45 (m, 1H), 1.33 (s, 3H), 1.24 (t, $J = 6.8$ Hz, 3H), 1.18 (s, 3H), 0.94 (dd, $J = 3.6$ Hz, 8.4 Hz, 1H); \textbf{13C NMR (100 MHz, CDCl$_3$)}: $\delta$ ppm 173.0, 140.8, 128.2, 128.2, 126.7, 60.1, 59.6, 51.0, 49.9, 37.9, 23.6, 22.7, 16.8, 16.3, 14.3. \textbf{HRMS(ESI)}: m/z calcd for C$_{17}$H$_{24}$NO$_2$ [M+H]$^+$: 274.1802, found: 274.1803.
891, 717 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) ppm 7.22-7.12(m, 5H), 4.09-3.98(m, 2H), 3.79(d, J = 13.2 Hz, 1H), 3.08(d, J = 13.6 Hz, 1H), 2.56(d, J = 8.8 Hz, 1H), 2.27-2.19(m, 2H), 1.85-1.82(m, 2H), 1.64-1.61(m, 1H), 1.58-1.49(m, 2H), 1.21-1.15(m, 5H), 0.91-0.88(m, 1H), 0.82-0.79(m, 2H); \(^1\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) ppm 173.3, 140.6, 128.2, 128.2, 126.7, 71.3, 60.1, 51.3, 50.5, 38.7, 34.8, 27.9, 27.8, 26.4, 23.9, 18.2, 14.3. HRMS(ESI): m/z calcd for C\(_{19}\)H\(_{26}\)NO\(_2\) [M+H]\(^+\): 300.1807, found: 300.1815.

**Preparation of Compound 4p:** The reaction of cyclic amidine 2p (342 mg, 0.8 mmol) with MeMgBr (0.8 mL, 2.4 mmol), was carried out using general procedure 3F, the product 4p obtained in 48% yield (108 mg) as a pale yellow oil; IR (neat): 3061, 3028, 2968, 2931, 2859, 2800, 1715, 1605, 1493, 1459, 1372, 1316, 1265, 1172, 1101, 1039, 730, 724 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) ppm 7.40-7.36(m, 2H), 7.31-7.27(m, 3H), 4.28-4.14(m, 2H), 3.17(d, J = 8.4 Hz, 1H), 2.86-2.79(m, 2H), 2.74-2.65(m, 1H), 2.61(dd, J = 2.8 Hz, 8.4 Hz, 1H), 2.53-2.43(m, 1H), 2.12-2.08(m, 1H), 1.42-1.40(m, 1H), 1.34(t, J = 7.2 Hz, 3H), 1.21(s, 3H), 1.15(s, 3H), 1.02-1.00(m, 1H); \(^1\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) ppm 172.9, 141.0, 128.8, 128.2, 125.9, 60.0, 59.6, 50.0, 48.6, 37.9, 35.9, 23.5, 22.5, 16.7, 15.9, 14.2. HRMS(ESI): m/z calcd for C\(_{18}\)H\(_{26}\)NO\(_2\) [M+H]\(^+\): 288.2005, found: 288.2009.

**Preparation of Compound 4o:** The reaction of cyclic amidine 2o (702 mg, 1.59 mmol) with MeMgBr (1.1 mL, 3.34 mmol), was carried out using general procedure 3F, the product 4o obtained in 43% yield (206 mg) as a colourless oil; IR (neat): 3060, 2979, 2927, 2866, 2829, 2800, 1712, 1602, 1497, 1451, 1365, 1296, 1255, 1174, 1138, 1103, 1022, 769, 704 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) ppm 7.31-7.25(m, 4H), 7.21-7.18(m, 1H), 4.14(q, J = 6.8 Hz, 2H), 3.92(d, J = 14.4 Hz, 1H), 3.03(d, J = 14 Hz, 1H), 2.45-2.38(m, 1H), 2.25-2.19(m, 1H), 1.78-1.70(m, 1H), 1.54-1.49(m, 1H), 1.37(d, J = 3.6 Hz, 1H), 1.29(s, 6H), 1.27(s, 3H), 1.04(s, 3H), 0.81(d, J = 4 Hz, 1H); \(^1\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) ppm 172.6, 141.0, 128.8, 128.0, 125.9, 60.0, 59.6, 50.0, 48.6, 37.9, 35.9, 23.5, 22.5, 16.7, 15.9, 14.2. HRMS(ESI): m/z calcd for C\(_{19}\)H\(_{26}\)NO\(_2\) [M+H]\(^+\): 302.2115, found: 302.2110.
3G. Procedure for regioselective ring opening of cyclopropane using organocopper reagent:

To a stirred solution of MeMgBr (2.1 equiv) and CuCN (1.1 equiv) in dry THF at room temperature for about 30 minutes under N\textsubscript{2} atmosphere was added cyclic amidine 2 (1 equiv) in dry THF (3 mL/ mmol). The reaction mixture was stirred at room temperature until the completion of reaction as indicated by TLC. Then reaction mixture was cooled to 0 °C, diluted with saturated Na\textsubscript{2}SO\textsubscript{4} (20 mL/ mmol) and extracted with EtOAc (2 X 30 mL/ mmol). The combined EtOAc solvent was washed successively with water (2 X 20 mL) and brine solution (30 mL). The organic layer was collected, dried over anhydrous Na\textsubscript{2}SO\textsubscript{4}, filtered and concentrated under reduced pressure. Purification of the crude product using column chromatography yielded the substituted 2-iminopyrrolidine 5.

**Preparation of Compound 5a:** The reaction of cyclic amidine 2a (755 mg, 1.83 mmol), MeMgBr (1.28 mL, 3.84 mmol) and CuCN (180 mg, 2 mmol) was carried out using general procedure 3G, the product 5a obtained in 83% yield (574 mg) (dr = 3:1) as a pale yellow oil; IR (neat): 3061, 2975, 2933, 2875, 1733, 1592, 1487, 1428, 1370, 1337, 1264, 1184, 1149, 1093, 1024, 993, 889, 816 cm\textsuperscript{-1}; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}): δ ppm 7.79(d, J = 8.0 Hz, 0.67H), 7.75(d, J = 8.0 Hz, 2.13H), 7.28(d, J = 8.0 Hz, 0.67H), 7.21(d, J = 8.0 Hz, 2.15H) 5.74-5.64(m, 1.07H), 5.27-5.19(m, 2.19H), 4.95-4.93(brm, 0.58H), 4.41(d, J = 8.8 Hz, 0.44H), 4.21-4.06(m, 4.02H), 3.99-3.90(m, 1.15H), 3.67(dd, J = 10.4 Hz, 7.2 Hz, 0.65H), 3.40(t, J = 10 Hz, 0.45H), 3.24(t, J = 10.4 Hz, 0.46H), 3.05(d, J = 10.4 Hz, 0.65H), 2.53-2.47(m, 0.49H), 2.41(s, 1.04H), 2.37(s, 3.21H), 2.34-2.31(m, 1.01H), 1.59-1.42(m, 1.81H), 1.31-1.27(m, 0.44H), 1.26-1.22(m, 3.43H), 0.96(t, J = 7.6 Hz, 1.36H), 0.91(t, J = 7.6 Hz, 2.01H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}): δ ppm 169.7, 168.0, 165.6, 164.2, 142.1, 142.1, 140.5, 140.4, 130.6, 130.5, 129.7, 129.7, 129.1, 129.1, 126.5, 126.4, 119.2, 118.9, 61.7, 61.5, 54.6, 52.9, 52.8, 52.5, 47.9,
Preparation of Compound 5q: The reaction of cyclic amidine 2q (684 mg, 1.71 mmol), MeMgBr (1.2 mL, 3.6 mmol) and CuCN (169 mg, 1.89 mmol) was carried out using general procedure 3G, the product 5q obtained in 87% yield (618 mg) (dr = 4:1) as a pale yellow oil; IR (neat): 3061, 2962, 2934, 2875, 1740, 1579, 1489, 1457, 1439, 1336, 1295, 1169, 1093, 1024, 999, 892, 817, 722 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.78 (d, J = 8.0 Hz, 2.46H), 7.29 - 7.27 (m, 3.63H), 7.24 - 7.19 (m, 5.04H), 4.84 (d, J = 14.4 Hz, 0.23H), 4.75 (d, J = 14.8 Hz, 1.02H), 4.46 (d, J = 14.4 Hz, 0.36H), 4.18 (d, J = 2 Hz, 1.00H), 3.70 (s, 3.00H), 3.68 (s, 0.74H), 3.58 (dd, J = 10.8 Hz, 7.2 Hz, 1.04H), 3.35-3.31 (m, 0.23H), 3.14 (t, J = 10.4 Hz, 0.24H), 2.98-2.95 (m, 1.04H), 2.39 (s, 3.73H), 2.34-2.29 (m, 1.05H), 1.53-1.30 (m, 2.15H), 1.25-1.18 (m, 0.42H), 0.91 (t, J = 7.2 Hz, 0.70H), 0.85 (t, J = 7.6 Hz, 3.17H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 170.2, 164.3, 142.2, 140.5, 134.7, 129.2, 128.9, 128.3, 128.1, 126.5, 54.3, 52.7, 49.1, 40.2, 27.8, 21.6, 12.2, 11.0. HRMS(ESI): m/z calcd for C₂₂H₂₇N₂O₄S [M+H]⁺: 415.1692, found: 415.1685.

Preparation of Compound 5g: The reaction of cyclic amidine 2g (304 mg, 0.66 mmol), MeMgBr (0.46 mL, 1.4 mmol) and CuCN (65 mg, 0.73 mmol) was carried out using general procedure 3G, the product 5g obtained in 84% yield (264 mg) (dr = 5:1) as a pale yellow oil; IR (neat): 3056, 2983, 2967, 2856, 1736, 1578, 1479, 1456, 1371, 1274, 1255, 1179, 1147, 1092, 1023, 916, 891 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ ppm 7.74 (d, J = 8.0 Hz, 2.35H), 7.20 (d, J = 8.0 Hz, 2.42H), 5.29 (s, 1.59H), 4.41 (d, J = 8.4 Hz, 0.20H), 4.23 (s, 0.99H), 4.20-4.03 (m, 4.31H), 3.96-3.92 (m, 1.01H), 3.81 (t, J = 8.4 Hz, 0.21H), 3.48 (d, J = 10 Hz, 0.17H), 3.44 (d, J = 10.4 Hz, 1.04H), 2.37 (s, 3.66H), 2.35-2.31 (m, 0.89H), 2.26 (s, 1.12H), 2.10 (s, 1.18H), 2.03 (s, 0.92H), 1.98-1.61 (m, 16.90H), 1.51-1.42 (m, 1.99H), 1.24 (t, J = 7.2 Hz, 1.37H), 1.19 (t, J = 7.2 Hz, 3.49H), 0.99 (t, J = 7.6 Hz, 0.69H), 0.92 (t, J = 7.6 Hz, 3.00H); ¹³C NMR (100 MHz, CDCl₃): δ ppm 169.7, 164.1, 141.8, 140.9, 129.0, 126.2, 61.6, 61.3, 60.5, 60.3, 60.3, 54.1,
53.5, 53.3, 52.2, 41.2, 40.7, 38.6, 38.5, 37.8, 37.7, 33.1, 32.5, 30.8, 30.7, 30.7, 30.6, 27.6, 27.6, 27.1, 22.4, 21.5, 14.4, 14.2, 12.5, 11.3.

**HRMS(ESI):** m/z calcd for C_{26}H_{37}N_{2}O_{4}S [M+H]^+: 473.2469, found: 473.2465.

**Preparation of Compound 5p (data of major diastereomer):** The reaction of cyclic amidine 2p (542 mg, 1.27 mmol), MeMgBr (0.9 mL, 2.67 mmol) and CuCN (125 mg, 1.39 mmol) was carried out using general procedure 3G, the product 5p obtained in 85\% yield (477 mg) (dr = 4:1) as a pale yellow oil; **IR (neat):** 3063, 3030, 2969, 2935, 2873, 1734, 1584, 1575, 1488, 1460, 1371, 1295, 1282, 1261, 1222, 1183, 1148, 1092, 1026, 901 cm\(^{-1}\); **\(^1\)H NMR (400 MHz, CDCl\(_3\)):** δ ppm 7.79(d, J = 8.4 Hz, 2H), 7.26-7.19(m, 5H), 7.13(d, J = 7.6 Hz, 2H), 4.21-4.07(m, 2H), 4.04(d, J = 1.6 Hz, 1H), 3.74-3.61(m, 2H), 3.51(dd, J = 7.2 Hz, 10.4 Hz, 1H), 2.91-2.81(m, 3H), 2.39(s, 3H), 2.25-2.20(m, 1H), 1.44-1.33(m, 1H), 1.30-1.27(m, 1H), 1.24(t, J = 7.2 Hz, 3H), 0.82(t, J = 7.2 Hz, 3H); **\(^{13}\)C NMR (100 MHz, CDCl\(_3\)):** δ ppm 169.6, 164.1, 142.0, 140.8, 138.2, 129.1, 128.8, 128.7, 126.7, 126.4, 61.6, 54.6, 54.1, 46.9, 40.5, 32.9, 27.7, 21.5, 14.1, 11.2; **HRMS(ESI):** m/z calcd for C_{24}H_{31}N_{2}O_{4}S [M+H]^+: 443.1999, found: 443.2010;

**1D NOESY data (500 MHz, CDCl\(_3\))** of major diastereomer of 5p:

**Preparation of Compound 5k: The reaction of cyclic amidine 2k (1.1 g, 2.25 mmol), MeMgBr (1.57 mL, 4.73 mmol) and CuCN (221 mg, 2.47 mmol) was carried out using general procedure 3G, the product 5k obtained in 81\% yield (915 mg) (dr = 2:1) as a pale...
yellow oil; **IR (neat):** 3057, 2985, 2926, 2854, 1729, 1589, 1491, 1452, 1370, 1271, 1186, 1147, 1092, 1027, 896, 815, 764, 719 cm\(^{-1}\); **\(^1\)H NMR (400 MHz, CDCl\(_3\)):** \(\delta\) ppm 7.74-7.70 (m, 3.02H), 7.35-7.25 (m, 8.45H), 7.23-7.17 (m, 7.47H), 7.09 (d, \(J = 7.2\) Hz, 1.16H), 7.02 (d, \(J = 6.8\) Hz, 1.96H), 4.86 (d, \(J = 14.8\) Hz, 0.55H) 4.78 (d, \(J = 14.4\) Hz, 0.95H), 4.49 (d, \(J = 14.4\) Hz, 0.55H), 4.32 (d, \(J = 8.0\) Hz, 0.57H), 4.26 (d, \(J = 14.8\) Hz, 0.98H), 4.20 (s, 0.98H), 4.15-4.01 (m, 2.56H), 3.89-3.84 (m, 0.57H), 3.54-3.50 (m, 1.00H), 3.46 (d, \(J = 3.6\) Hz, 0.48H), 3.43 (s, 0.58H), 3.14 (d, \(J = 11.2\) Hz, 0.93H), 2.99-2.90 (m, 0.56H), 2.73-2.62 (m, 2.51H), 2.39 (s, 3.00), 2.36 (s, 1.75H), 1.17 (t, \(J = 6.8\) Hz, 3.05H), 1.14-1.05 (m, 6.37H); **\(^13\)C NMR (100 MHz, CDCl\(_3\)):** \(\delta\) ppm 169.4, 168.0, 165.7, 164.4, 144.0, 142.3, 142.1, 142.0, 140.5, 140.4, 134.7, 134.5, 129.1, 129.0, 128.8, 128.8, 128.7, 128.2, 128.2, 128.0, 127.4, 127.1, 126.8, 126.6, 126.4, 126.3, 61.7, 61.3, 52.9, 51.9, 51.5, 50.4, 49.1, 48.8, 44.8, 44.2, 42.5, 39.8, 22.6, 21.5, 21.4, 17.8, 13.9, 13.7. **HRMS (ESI):** m/z calcd for C\(_{29}\)H\(_{33}\)N\(_2\)O\(_4\)S [M+H]\(^+\): 505.2156, found: 505.2149.

4. Crystal data and structure refinement for compound 2b

Single crystal X-ray analysis of ethyl (Z)-3-(tert-butyl)-2-(tosylimino)-3-azabicyclo[3.1.0]hexane-1-carboxylate (2b)

ORTEP diagram of compound 2b with ellipsoids adjusted to 30% probability
Table S6. Crystal data and structure refinement for compound 2b.

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5. References:


1H NMR spectrum of compound 1b
$^1$H NMR spectrum of compound 1b
$^{13}$C NMR spectrum of compound 1b
DEPT-135 NMR spectrum of compound 1b
\(^1\)H-\(^1\)H COSY NMR spectrum of compound 1b
Current Data Parameters
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PROCNO                1

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D11          0.03000000 sec
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IN0          0.00000000 sec
ZGOPTNS

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P28             1000.00 usec
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======== CHANNEL f2 ========
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CPDPRG[2           garp
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PLW2        47.00000000 W
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====== GRADIENT CHANNEL ======
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F1 - Acquisition parameters
TD                  256
SFO1           100.6127609 MHz
FIDRES       130.208328 Hz
SW              165.639 ppm
FnMODE    Echo-Antiecho
F2 - Processing parameters
SI                 1024
SF          400.1316592 MHz
WDW               QSINE
SSB                   2
LB       0 Hz
PC       1.40

F1 - Processing parameters
SI                 1024
MC2         100.6127609 MHz
WDW               QSINE
GB       0 Hz
G8       0

1H-13C HSQC NMR spectrum of compound 1b

S48
\[ ^1H \text{ NMR spectrum of compound 1a} \]
$^1$H NMR spectrum of compound 1a
$^{13}$C NMR spectrum of compound 1a
DEPT-135 NMR spectrum of compound 1a
$^1$H-$^1$H COSY NMR spectrum of compound 1a
$^1$H-$^{13}$C HSQC NMR spectrum of compound 1a
\[ ^1H \text{NMR spectrum of compound 1c} \]
\[ \text{lab sb-kkd-527} \]

\[ \text{iitm-Proton(-5to15) CDCl}_3 /\text{opt/topspin nmr 12} \]

\[ \text{NMR spectrum of compound 1c} \]
$^{13}$C NMR spectrum of compound 1c
DEPT-135 NMR spectrum of compound 1c
$^1$H-$^1$H COSY NMR spectrum of compound 1c
$^1$H-$^1$C HSQC NMR spectrum of compound 1c
lab sb-kkd-528

tilm-Proton(-5to15) CDCl3 /opt/topspin nmr 13

Current Data Parameters
NAME sb-kkd-528
EXPNO 152
PROCNO 1

F2 - Acquisition Parameters
Date 20171001
Time 7.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.089465 sec
RG 31.9
DW 62.400 usec
DE 6.50 usec
TE 300.9 K
D1 0.50000000 sec
TD0 1

CHANNEL f1
SFO1 400.1320007 MHz
NUC1 1H
P1 15.70 usec
PLW1 7.75000000 W

F2 - Processing parameters
SI 65536
SF 400.1300316 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 1.00
PC 1.00

^H NMR spectrum of compound 1d


$^1$H NMR spectrum of compound 1d
$^{13}$C NMR spectrum of compound 1d

Current Data Parameters
NAME sb-kkd-528
EXPNO 147
PROCNO 1

F2 - Acquisition Parameters
Date 20171001
Time 7.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 16540
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 1.453353 Hz
AQ 0.3440320 sec
RG 200.34
DW 20.800 usec
DE 6.50 usec
TE 301.6 K
D1 1.00000000 sec
D11 0.00000000 sec
TD0 1

-------- CHANNEL f1 --------
SFO1 100.6228289 MHz
NUC1 13C
P1 9.25 usec
PLW1 47.00000000 W

-------- CHANNEL f2 --------
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 7.75000000 W
PLW12 0.23539999 W
PLW13 0.11863000 W

F2 - Processing parameters
SF 32768
SF 100.6127646 MHz
WDW EM
SSB 0
LB 0
GB 1.00 Hz
PC 1.40
DEPT-135 NMR spectrum of compound 1d
$^{1}H-^{1}H$ COSY NMR spectrum of compound 1d
\textbf{S66}

\textbf{1H-13C HSQC NMR spectrum of compound 1d}
"\textbf{\textscatter{\textsuperscript{1}H NMR spectrum of compound 1e}}\"
$^1$H NMR spectrum of compound 1e
$^{13}$C NMR spectrum of compound 1e
DEPT-135 NMR spectrum of compound 1e
\(^1\)H-\(^1\)H COSY NMR spectrum of compound 1e
"1H-13C HSQC NMR spectrum of compound 1e"
**Current Data Parameters**

**NAME**  sb-dvk-595  
**EXPNO**  143  
**PROCNO**  1  

**F2 - Acquisition Parameters**

Date_  20171112  
Time  21.45  
INSTRUM  spect  
PROBHD  5 mm PABBO BB-  
PULPROG  zg30  
TD  65536  
SOLVENT  CDCl3  
NS  16  
DS  2  
SWH  8012.820 Hz  
FIDRES  0.122266 Hz  
AQ  4.0894465 sec  
RG  31.9  
DW  62.400 usec  
DE  6.50 usec  
TE  298.3 K  
D1  0.50000000 sec  
TD0  1  

**F2 - Processing parameters**

SI  65536  
SF  400.1300094 MHz  
WDW  EM  
SSB  0  
LB  0.30 Hz  
GB  0  
PC  1.00  

--- CHANNEL f1 ---

**SFO1**  400.1320007 MHz  
**NUC1**  1H  
P1  15.70 usec  
PLW1  7.75000000 W  

--- CHANNEL f1 ---

**1H NMR spectrum of compound 1f**
$^1$H NMR spectrum of compound 1f
\[13\text{C NMR spectrum of compound 1f}\]
DEPT-135 NMR spectrum of compound 1f
$^1$H-$^1$H COSY NMR spectrum of compound 1f
\( ^{1}\text{H}-^{13}\text{C} \) HSQC NMR spectrum of compound 1f
1H NMR spectrum of compound 1g
$^{1}H$ NMR spectrum of compound 1g
$^{13}$C NMR spectrum of compound 1g
DEPT-135 NMR spectrum of compound 1g
$^1$H-$^1$H COSY NMR spectrum of compound 1g
$^1$H-$^{13}$C HSQC NMR spectrum of compound 1g
$^1$H NMR spectrum of compound 1h
$^{1}$H NMR spectrum of compound 1h
$^{13}$C NMR spectrum of compound 1h
DEPT-135 NMR spectrum of compound 1h
$^1$H-$^1$H COSY NMR spectrum of compound 1h
$^1$H-$^{13}$C HSQC NMR spectrum of compound 1h
\[ ^1H \text{ NMR spectrum of compound 1i} \]
$^1$H NMR spectrum of compound 1i
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**F2 - Acquisition Parameters**

- **Date:** 20171223
- **Time:** 10:01
- **INSTRUM:** spect
- **PROBHD:** 5 mm PABBO BB-
- **PULPROG:** zgpg30
- **TD:** 16540
- **SOLVENT:** CDCl3
- **NS:** 256
- **DG:** 4
- **SWH:** 24038.461 Hz
- **FIDRES:** 1.453535 Hz
- **AO:** 0.3440320 sec
- **RG:** 200.34
- **DW:** 20.800 usec
- **DE:** 6.50 usec
- **TE:** 293.9 K
- **D1:** 1.00000000 sec
- **D11:** 0.03000000 sec
- **TD0:** 1

** CHANNEL f1 **

- **SFO1:** 100.6228289 MHz
- **NUC1:** 13C
- **P1:** 9.25 usec
- **PLW1:** 47.00000000 W

** CHANNEL f2 **

- **SFO2:** 400.1316005 MHz
- **NUC2:** 1H
- **CPDPRG[2:** waltz16
- **PCPD2:** 90.00 usec
- **PLW2:** 7.75000000 W
- **PLW12:** 0.23583999 W
- **PLW13:** 0.11863000 W

**F2 - Processing parameters**

- **SI:** 32768
- **SF:** 100.6127666 MHz
- **WDW:** EM
- **SSR:** 0
- **LB:** 1.00 Hz
- **GB:** 0
- **PC:** 1.40

---

**13C NMR spectrum of compound 1i**

![13C NMR spectrum of compound 1i](image)
DEPT-135 NMR spectrum of compound 1i
$^1$H-$^1$H COSY NMR spectrum of compound 1i
\textsuperscript{1}H-\textsuperscript{13}C HSQC NMR spectrum of compound 1i
Current Data Parameters
NAME           sb-kkd-562
EXPNO               314
PROCNO                1

F2 - Acquisition Parameters
Date_          20171016
Time              17.03
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                15.17
DW               62.400 usec
DE                 6.50 usec
TE                292.4 K
D1           0.50000000 sec
TD0                   1

--------- CHANNEL f1 --------
SFO1        400.1320007 MHz
NUC1                 1H
P1                15.70 usec
PLW1         7.75000000 W

F2 - Processing parameters
SI                65536
SF       400.1299638 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00

\( ^1\text{H} \) NMR spectrum of compound 1j
$^1$H NMR spectrum of compound 1j
\[ 1^3 \text{C NMR spectrum of compound 1j} \]
DEPT-135 NMR spectrum of compound 1j
\(^{1}\text{H}-^{1}\text{H}\) COSY NMR spectrum of compound 1j
$^1$H-$^{13}$C HSQC NMR spectrum of compound 1j
1H NMR spectrum of compound 1k


$^1$H NMR spectrum of compound 1k
$^{13}$C NMR spectrum of compound 1k
DEPT-135 NMR spectrum of compound 1k
$^1$H-$^1$H COSY NMR spectrum of compound 1k
$^1$H-$^{13}$C HSQC NMR spectrum of compound 1k
$^1$H NMR spectrum of compound 1l
$^1$H NMR spectrum of compound II

**Current Data Parameters**

- **NAME**: sb-kkd-530
- **EXPNO**: 197
- **PROCNO**: 1

**F2 - Acquisition Parameters**

- **Date**: 20171008
- **Time**: 21:13
- **INSTRUM**: spect
- **PROBHD**: 5 mm PABBO BB-
- **PULPGR**: zgpg30
- **TD**: 16540
- **SOLVENT**: CDCl3
- **NS**: 256
- **DS**: 4
- **SWH**: 24038.461 Hz
- **FIDRES**: 1.453353 Hz
- **AQ**: 0.3440320 sec
- **RG**: 200.34
- **DW**: 20.800 usec
- **DE**: 6.50 usec
- **TE**: 295.1 K
- **D1**: 1.00000000 sec
- **D11**: 0.03000000 sec
- **TD0**: 1

**F2 - Processing parameters**

- **SI**: 32768
- **SF**: 100.6127786 MHz
- **WDW**: EM
- **SSB**: 0
- **LB**: 1.00 Hz
- **GB**: 0
- **PC**: 1.40

---

**13C NMR spectrum of compound 1l**

---

S111
DEPT-135 NMR spectrum of compound 1l

lab sb-kkd-530

Current Data Parameters
NAME sb-kkd-530
EXPNO 198
PROCNO 1

F2 - Acquisition Parameters
Date 2017/10/08
Time 21.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG deptsp135
TD 32768
SOLVENT CDCl3
NS 64
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 0.8126464 sec
RG 200.34
DW 24.800 usec
DE 6.50 usec
TE 295.0 K
CNST2 145.0000000
D1 1.00000000 sec
D2 0.00044628 sec
D12 0.00002000 sec
TD0 1

-------- CHANNEL f1 --------
SFO1 100.6208166 MHz
NUC1 13C
P1 9.25 usec
P13 2000.00 usec
PLW0 0 W
PLW1 47.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPW5 6.14429998 W

-------- CHANNEL f2 --------
SFO2 400.1312797 MHz
NUC2 1H
CPDPROI2 waltz16
P3 15.70 usec
P4 31.40 usec
PCPD2 90.00 usec
PLW2 7.75000000 W
PLW12 0.23589999 W
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
PC 1.40
$^1$H-$^1$H COSY NMR spectrum of compound 1l
$^1$H-$^{13}$C HSQC NMR spectrum of compound 11
Current Data Parameters
NAME      sb-kkd-583
EXPNO               532
PROCNO                1

F2 - Acquisition Parameters
Date_          20171029
Time              11.11
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TO                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                 31.9
DW               62.400 usec
DE                 6.50 usec
TE                292.5 K
D1           0.50000000 sec
TD0                   1

-------- CHANNEL f1 --------
SFO1        400.1320007 MHz
NUC1                 1H
P1                15.70 usec
PLW1         7.5000000 W

F2 - Processing parameters
SI                65536
SF          400.1300061 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00

$^1$H NMR spectrum of compound 1m
$^1$H NMR spectrum of compound 1m
\(^{13}\text{C}\) NMR spectrum of compound 1m
DEPT-135 NMR spectrum of compound 1m
$^1$H-$^1$H COSY NMR spectrum of compound 1m
$^1$H-$^{13}$C HSQC NMR spectrum of compound 1m
$^1$H NMR spectrum of compound 1n
$^1$H NMR spectrum of compound 1n
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**F2 - Acquisition Parameters**

- **Date:** 20180128
- **Time:** 13.14
- **INSTRUMENT:** spect
- **PROBHD:** 5 mm PABBO BB-
- **PULPROG:** zgpg30
- **TD:** 16540
- **SOLVENT:** CDCl3
- **NS:** 256
- **DS:** 4
- **SWH:** 240.08 Hz
- **FIDRES:** 1.453353 Hz
- **AQ:** 0.3440320 sec
- **RG:** 200.34
- **DW:** 20.800 usec
- **DE:** 6.50 usec
- **TE:** 296.8 K
- **D1:** 1.00000000 sec
- **D11:** 0.03000000 sec
- **TD0:** 1

**--- CHANNEL f1 ---**

- **SFO1:** 100.6127943 MHz
- **NUC1:** 13C
- **P1:** 9.25 usec
- **PLW1:** 47.00000000 W

**--- CHANNEL f2 ---**

- **SFO2:** 400.1316005 MHz
- **NUC2:** 1H
- **CPDPB2:** waltz16
- **PCPD2:** 90.00 usec
- **PLW2:** 7.75000000 W
- **PLW12:** 0.23583999 W
- **PLW13:** 0.11863000 W

**F2 - Processing parameters**

- **SI:** 32768
- **SF:** 100.6127943 MHz
- **WDW:** EM
- **SSB:** 0
- **LB:** 1.00 Hz
- **GB:** 0
- **PC:** 1.40

**1³C NMR spectrum of compound 1n**
DEPT-135 NMR spectrum of compound 1n
lab sb-kkd-633
COSYGPSW CDCl3 /opt/topspin nmr 2

^{1}H-^{1}H\text{ COSY NMR spectrum of compound 1n}
### $^1$H-$^{13}$C HSQC NMR spectrum of compound 1n

![HSQC NMR Spectrum](image)

- **NAME**: sb-kkd-633
- **EXPNO**: 293
- **PROCNO**: 1
- **Current Data Parameters**
  - **INSTRUM**: spect
  - **PROBHD**: 5 mm PABBO BB-
  - **PULPROG**: hsqcetgp
  - **TD**: 1024
  - **SOLVENT**: CDCl3
  - **NS**: 2
  - **DS**: 16
  - **SWH**: 3472.222 Hz
  - **FIDRES**: 3.390842 Hz
  - **AQ**: 0.1474560 sec
  - **RG**: 200.34
  - **DW**: 144.000 usec
  - **DE**: 6.60 usec
  - **TE**: 296.7 K
  - **CNST2**: 145.0000000
  - **D0**: 0.00000300 sec
  - **D1**: 1.45105302 sec
  - **D4**: 0.00172414 sec
  - **D11**: 0.00000000 sec
  - **D16**: 0.00020000 sec
  - **IN0**: 0.00003000 sec
  - **ZGOPTNS**:

#### CHANNEL f1
- **SFO1**: 400.1317687 MHz
- **NUC1**: $^1$H
- **P1**: 15.70 usec
- **P2**: 31.40 usec
- **P3**: 9.25 usec
- **P4**: 18.50 usec
- **PCPD1**: 80.00 usec
- **PLW1**: 7.75000000 W
- **PLW12**: 0.62835002 W

#### CHANNEL f2
- **SFO2**: 100.6202727 MHz
- **NUC2**: $^{13}$C
- **CPDPRG[2]**: garp
- **P3**: 9.25 usec
- **P4**: 18.50 usec
- **PCPD2**: 80.00 usec
- **PLW2**: 47.00000000 W
- **PLW12**: 0.62835002 W

#### GRADIENT CHANNEL
- **GPZ1**: 80.00 %
- **GPZ2**: 20.10 %
- **P16**: 1000.00 usec

- **lab sb-kkd-633**
- **HSQCETGP CDCl3 /opt/topspin nmr 2**

---

S126
1H NMR spectrum of compound 1o
$^{1}$H NMR spectrum of compound 1o
$^{13}$C NMR spectrum of compound 1o
DEPT-135 NMR spectrum of compound 1o
$^1$H-$^1$H COSY NMR spectrum of compound 1o
$^1$H-$^{13}$C HSQC NMR spectrum of compound 10
Current Data Parameters
NAME       sb-kkd-582
EXPNO               538
PROCNO                1

F2 - Acquisition Parameters
Date_          20171029
Time              11.50
INSTRUM           spect
PROBHD   5 mm PABB BO BB- 
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                15.17
DW               62.400 usec
DE                6.50 usec
TE                292.6 K
D1           0.50000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1320007 MHz
NUC1                 1H
P1                15.70 usec
PLW1         7.75000000 W

F2 - Processing parameters
SI                65536
SF          400.1300000 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00

--- CHANNEL f1 ---

1H NMR spectrum of compound 1p
1H NMR spectrum of compound 1p
\(^{13}\)C NMR spectrum of compound 1p
DEPT-135 NMR spectrum of compound 1p
$^1$H-$^1$H COSY NMR spectrum of compound 1p
$^1$H-$^{13}$C HSQC NMR spectrum of compound 1p
1H NMR spectrum of compound 2b
$^{1}$H NMR spectrum of compound 2b
13C NMR spectrum of compound 2b
DEPT-135 NMR spectrum of compound 2b
**1H-1H COSY NMR spectrum of compound 2b**

---

**Current Data Parameters**

- **NAME**: sb-kkd-567
- **EXPNO**: 275
- **PROCNO**: 1

**F2 - Acquisition Parameters**

- **Date**: 20171014
- **Time**: 14.42
- **INSTRUM**: spect
- **PROBHD**: 5 mm PABBO BB-
- **PULPROG**: cosygpppqf
- **TD**: 2048
- **SOLVENT**: CDCl3
- **NS**: 1
- **DS**: 8
- **SWH**: 3448.276 Hz
- **FIDRES**: 1.683728 Hz
- **AQ**: 0.2398500 sec
- **DE**: 6.50 usec
- **DW**: 145.000 usec
- **TE**: 292.2 K
- **D0**: 0.00000300 sec
- **D1**: 1.89473295 sec
- **D11**: 0.03000000 sec
- **D12**: 0.00002000 sec
- **D13**: 0.00000400 sec
- **D16**: 0.00003990 sec
- **IN0**: 0.00002900 sec

**== CHANNEL f1 ========**

- **SFO1**: 400.1316093 MHz
- **NUC1**: 1H
- **P0**: 15.70 usec
- **P1**: 15.70 usec
- **P17**: 2500.00 usec
- **PLW1**: 7.7500000 W
- **PLW10**: 2.12260008 W

**== GRADIENT CHANNEL =====**

- **GPNAM[1]**: SMSQ10.100
- **GPZ1**: 10.00 %
- **P16**: 1000.00 usec

**F1 - Acquisition parameters**

- **TD**: 128
- **SFO1**: 400.1316 MHz
- **FIDRES**: 8.619 ppm
- **FMODE**: OF

**F2 - Processing parameters**

- **SI**: 0.024
- **SF**: 400.100004 MHz
- **WZW**: 0.80 QINE
- **GB**: 1.40
- **PC**: 1.40

**F1 - Processing parameters**

- **SI**: 10.04
- **MC2**: OF
- **SF**: 400.130002 MHz
- **WZW**: 0.80 QINE
- **LB**: 0 Hz
- **GB**: 0

---

S143
$^1$H-$^{13}$C HSQC NMR spectrum of compound 2b
$^1$H NMR spectrum of compound 2a
\(^1\)H NMR spectrum of compound 2a
13C NMR spectrum of compound 2a
DEPT-135 NMR spectrum of compound 2a
$^1$H-$^1$H COSY NMR spectrum of compound 2a
\[ ^1H^{13}C \] HSQC NMR spectrum of compound 2a
\[ \text{\textsuperscript{1}H NMR spectrum of compound 2c} \]
$^1$H NMR spectrum of compound 2c
13C NMR spectrum of compound 2c
Current Data Parameters
NAME sb-kkd-531
EXPN0 63
PROCNO 1

F2 - Acquisition Parameters
Date 20171004
Time 8.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG deptsp135
TD 32768
SOLVENT CDCl3
NS 64
DS 4
SWH 20161.291 Hz
AQ 0.8126464 sec
DW 24.800 usec
DE 6.50 usec
CNST2 145.0000000
D1 1.00000000 sec
D2 0.00044828 sec
D12 0.00002000 sec

DEPT-135 NMR spectrum of compound 2c
\( ^1H-^1H \text{ COSY NMR spectrum of compound 2c} \)
$^1$H-$^{13}$C HSQC NMR spectrum of compound 2c
$^1$H NMR spectrum of compound 2d
$^{1}$H NMR spectrum of compound 2d
$^{13}$C NMR spectrum of compound 2d
DEPT-135 NMR spectrum of compound 2d
lab sb-kkd-532
COSYGPSW CDCl3 /opt/topspin nmr 3

1H-1H COSY NMR spectrum of compound 2d
$^1$H-$^{13}$C HSQC NMR spectrum of compound 2d
1H NMR spectrum of compound 2e
$^1$H NMR spectrum of compound 2e
$^{13}$C NMR spectrum of compound 2e
DEPT-135 NMR spectrum of compound 2e
$^1$H-$^1$H COSY NMR spectrum of compound 2e
$^1$H-$^{13}$C HSQC NMR spectrum of compound 2e
$^1$H NMR spectrum of compound 2f
$^1$H NMR spectrum of compound 2f
$^{13}$C NMR spectrum of compound 2f
DEPT-135 NMR spectrum of compound 2f
\( ^1\text{H}-^1\text{H} \) COSY NMR spectrum of compound 2f
$^1$H-$^{13}$C HSQC NMR spectrum of compound 2f
1H NMR spectrum of compound 2g
IHM NMR spectrum of compound 2g
$^{13}$C NMR spectrum of compound 2g
DEPT-135 NMR spectrum of compound 2g
\(^1\)H-\(^1\)H COSY NMR spectrum of compound 2g
\(^1\)H-\(^{13}\)C HSQC NMR spectrum of compound 2g
$^1$H NMR spectrum of compound 2h
$^1$H NMR spectrum of compound 2h
13C NMR spectrum of compound 2h
DEPT-135 NMR spectrum of compound 2h
$^1$H-$^1$H COSY NMR spectrum of compound 2h

S185
$^1$H-$^{13}$C HSQC NMR spectrum of compound 2h
1H NMR spectrum of compound 2i
$^1$H NMR spectrum of compound 2i
$^{13}$C NMR spectrum of compound 2i
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#### F2 - Acquisition Parameters

- **Date:** 20171014  
- **Time:** 5.37  
- **INSTRUM:** spect  
- **PROBHD:** 5 mm PABBO BB-PULPROG  
- **TD:** 32768  
- **SOLVENT:** CDCl3  
- **NS:** 64  
- **DS:** 4  
- **SWH:** 20161.291 Hz  
- **AQ:** 0.8125464 sec  
- **RG:** 200.34  
- **DW:** 24.800 usec  
- **DE:** 6.50 usec  
- **TE:** 292.8 K  
- **CNST2:** 145.0000000  
- **D1:** 1.00000000 sec  
- **D2:** 0.00344828 sec  
- **D12:** 0.00002000 sec  
- **TD0:** 1

#### CHANNEL f1

- **SFO1:** 100.6208166 MHz  
- **NUC1:** 13C  
- **P1:** 9.25 usec  
- **T1:** 2000.00 usec  
- **PLW0:** 0 W  
- **PLW1:** 47.00000000 W  
- **SPW1:** 6.14429998 W  

#### CHANNEL f2

- **SFO2:** 400.1312797 MHz  
- **NUC2:** 1H  
- **CPDPRG2:** waltz16  
- **P3:** 15.70 usec  
- **P4:** 31.40 usec  
- **PCPD2:** 90.00 usec  
- **PLW2:** 7.75000000 W  
- **PLW12:** 0.23583999 W  

#### F2 - Processing parameters

- **SI:** 32768  
- **SF:** 100.6127690 MHz  
- **WDW:** EM  
- **SSB:** 0  
- **LB:** 1.00 Hz  
- **GB:** 0  
- **PC:** 1.40

---

DEPT-135 NMR spectrum of compound 2i
\[ ^1H-^1H \text{ COSY NMR spectrum of compound 2i} \]
\[ ^1\text{H}-^{13}\text{C} \text{ HSQC NMR spectrum of compound 2i} \]
$^1$H NMR spectrum of compound 2j
$^1$H NMR spectrum of compound 2j
$^{13}$C NMR spectrum of compound 2j
DEPT-135 NMR spectrum of compound 2j
$^1$H-$^1$H COSY NMR spectrum of compound 2j
$^1$H-$^{13}$C HSQC NMR spectrum of compound 2j
Current Data Parameters
NAME  cynna-cy
EXPNO  451
PROCNO  1

F2 - Acquisition Parameters
Date  20171021
Time  23.35
INSTRUM  spect
PROBHD  5 mm PABBO BB-
PULPROG  zg30
TD  65536
SOLVENT  CDCl3
NS  32
DS  2
SWH  8012.820 Hz
FIDRES  0.122266 Hz
AQ  4.0894465 sec
RG  67.99
DW  62.400 usec
DE  6.50 usec
TE  292.5 K
D1  1.00000000 sec
TD0  1

-------- CHANNEL f1 --------
SFO1  400.1324710 MHz
NUC1  1H
P1  15.70 usec
PLW1  7.75000000 W

F2 - Processing parameters
SI  65536
SF  400.1300098 MHz
WDW  EM
SSB  0
LB  0.30 Hz
GB  0
PC  1.00

1H NMR spectrum of compound 2k
1H NMR spectrum of compound 2k
$^{13}$C NMR spectrum of compound 2k
DEPT-135 NMR spectrum of compound 2k
1H-1H COSY NMR spectrum of compound 2k
$^1$H-$^1$C HSQC NMR spectrum of compound 2k
**lab sb-kkd-547**

PROTON CDCl₃ /opt/topspin nmr 5

Current Data Parameters
NAME sb-kkd-547
EXPNO 413
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171019
Time 7.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 88.51
DW 62.400 usec
DE 6.50 usec
TE 292.5 K
D1 1.00000000 sec
TDD 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 15.70 usec
PLW1 7.75000000 W

F2 - Processing parameters
SI 65536
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
PC 1.00

--- CHANNEL f1 -----------
SFO1 400.1324710 MHz
NUC1 1H
P1 15.70 usec
PLW1 7.75000000 W

**1H NMR spectrum of compound 2l**
$^1$H NMR spectrum of compound 2l
13C NMR spectrum of compound 2l
DEPT-135 NMR spectrum of compound 2l
1H-1H COSY NMR spectrum of compound 2l
$^1$H-$^{13}$C HSQC NMR spectrum of compound 2l
\textsuperscript{1}H NMR spectrum of compound 2m
$^1$H NMR spectrum of compound 2m
13C NMR spectrum of compound 2m
DEPT-135 NMR spectrum of compound 2m
1H-1H COSY NMR spectrum of compound 2m
$^1$H-$^{13}$C HSQC NMR spectrum of compound 2m
\(^1\)H NMR spectrum of compound 2n
$^1$H NMR spectrum of compound $2n$
$^{13}$C NMR spectrum of compound 2n
DEPT-135 NMR spectrum of compound 2n
\(^1\text{H}\)⁻\(^1\text{H}\) COSY NMR spectrum of compound 2n
\(^{1}\text{H}-^{13}\text{C}\) HSQC NMR spectrum of compound 2n
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#### F2 - Acquisition Parameters

- **Date:** 20171021
- **Time:** 23.14
- **INSTRUM:** spect
- **PROBHD:** 5 mm PABBO BB-PULPROG zg30
- **TD:** 65536
- **SOLVENT:** CDCl3
- **NS:** 16
- **DS:** 2
- **SWH:** 8012.820 Hz
- **FIDRES:** 0.122266 Hz
- **AQ:** 4.084465 sec
- **RG:** 79.6
- **DW:** 62.400 usec
- **DE:** 6.50 usec
- **TE:** 292.5 K
- **D1:** 0.50000000 sec

**CHANNEL f1**

- **SFO1:** 400.1320007 MHz
- **NUC1:** 1H
- **P1:** 15.70 usec
- **PLW1:** 7.75000000 W

#### F2 - Processing parameters

- **SI:** 65536
- **SF:** 400.1300354 MHz
- **WDW:** EM
- **SSB:** 0
- **LB:** 0.30 Hz
- **GB:** 0
- **PC:** 1.00

---

**1H NMR spectrum of compound 2o**

---

S223
$^1$H NMR spectrum of compound 2o
$^{13}$C NMR spectrum of compound 2o
DEPT-135 NMR spectrum of compound 2o
$^{1}$H-$^{1}$H COSY NMR spectrum of compound 2o
Current Data Parameters
NAME         kkd-573
EXPNO               459
PROCNO                1
F2 - Acquisition Parameters
Date: 20171022
Time: 0.59
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG        hsqcetgp
TD                 1024
SOLVENT           CDCl3
NS                    2
DS                   16
SWH            4032.258 Hz
FIDRES         3.937752 Hz
AQ            0.1269760 sec
RG               200.34
TE                292.6 K
CNST2       145.0000000
D0           0.00000300 sec
D1           1.47153294 sec
D4           0.00172414 sec
D11          0.03000000 sec
D16          0.00020000 sec
ZGOPTNS
======== CHANNEL f1 ========
SFO1        400.1314623 MHz
NUC1                 1H
P1                15.70 usec
P2                31.40 usec
P28             1000.00 usec
PLW1         7.75000000 W
======== CHANNEL f2 ========
SFO2        100.6202727 MHz
NUC2                13C
CPDP2             garp
P3                 9.25 usec
P4                18.50 usec
PCPD2             80.00 usec
PLW2        47.00000000 W
PLW12        0.62835002 W
====== GRADIENT CHANNEL ======
GPNAM[1]     SMSQ10.100
GPNAM[2]     SMSQ10.100
GPZ1              80.00 %
GPZ2              20.10%
PL16             1000.00 usec

F1 - Acquisition parameters
TD                 256
SFO1           100.6203 MHz
FIDRES       130.208328 Hz
SW              165.639 ppm
FnMODE    Echo-Antiecho
F2 - Processing parameters
SI                 1024
SF          400.1000339 MHz
WDW               QSINE
SSB           2
LB             0 Hz
GB       0 %
F1 - Processing parameters
SI                 1024
MC2       echo-echo
SF          100.612753 MHz
WDW               QSINE
SSB           2
LB             0 Hz
GB       0 %

1H-13C HSQC NMR spectrum of compound 2o

S228
$^1$H NMR spectrum of compound 2p
$^1$H NMR spectrum of compound 2p
$^{13}$C NMR spectrum of compound 2p
DEPT-135 NMR spectrum of compound 2p
$^1$H-$^1$H COSY NMR spectrum of compound 2p
$^1$H-$^{13}$C HSQC NMR spectrum of compound 2p
$^1$H NMR spectrum of compound 3q
1H NMR spectrum of compound 3q
$^{13}$C NMR spectrum of compound 3q
DEPT-135 NMR spectrum of compound 3q
\(^1\text{H}-\text{H}\) COSY NMR spectrum of compound 3q
1H-13C HSQC NMR spectrum of compound 3q
Current Data Parameters
NAME           sb-dvk-661
EXPNO          72
PROCNO          1

F2 - Acquisition Parameters
Date_          20180305
Time            10.03
INSTRUM         spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD               65536
SOLVENT         CDCl3
NS                16
DS                 2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ               4.0894465 sec
RG              95.73
DW            62.400 usec
DE              6.50 usec
TE             299.5 K
D1       0.50000000 sec
TD0             1

======== CHANNEL f1 ========
SFO1        400.1320007 MHz
NUC1                 1H
P1                15.70 usec
PLW1         7.75000000 W

F2 - Processing parameters
SI                65536
SF          400.1300301 MHz
WDW                  EM
SSB             0
LB              0.30 Hz
PC              1.00

1H NMR spectrum of compound 3j
$^1$H NMR spectrum of compound 3j
$^{13}$C NMR spectrum of compound 3j
### Current Data Parameters

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#### F2 - Acquisition Parameters

- **Date**: 20180305
- **Time**: 10.13
- **INSTRUM**: spect
- **PROBHD**: 5 mm PABBO BB-
- **PULPROG**: deptsp135
- **TD**: 32768
- **SOLVENT**: CDCl3

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#### F2 - Processing parameters

- **SI**: 32768
- **SF**: 100.6127690 MHz
- **WDW**: EM
- **SSB**: 0
- **LB**: 1.00 Hz
- **PC**: 1.40

---

**DEPT-135 NMR spectrum of compound 3j**

![DEPT-135 NMR spectrum of compound 3j](image-url)
$^1$H NMR spectrum of compound 3k
$^1$H NMR spectrum of compound 3k
Current Data Parameters
NAME  sb-dvk-660
EXPNO  165
PROCNO  1

F2 - Acquisition Parameters
Date  20180311
Time  0.28
INSTRUM  spect
PROBHD  5 mm PABBO BB-
PULPROG  zpg30
TD  16540
SOLVENT  CDCl3
NS  256
DS  4
SWH  24038.461 Hz
FIDRES  1.453353 Hz
AQ  0.3440320 sec
RG  200.34
DW  20.800 usec
DE  6.50 usec
TE  299.3 K
D1  1.00000000 sec
D11  0.00000000 sec
TD0  1

-------- CHANNEL f1 --------
SFO1  100.6228289 MHz
NUC1  13C
P1  9.25 usec
PLW1  47.00000000 W

-------- CHANNEL f2 --------
SFO2  400.1316005 MHz
NUC2  1H
CPDPROJ  waltz16
CPD2  90.00 usec
PLW2  7.75000000 W
PLW12  0.23589999 W
PLW13  0.11863000 W

F2 - Processing parameters
SI  32768
SF  100.6127583 MHz
WDW  EM
SSB  0
LB  1.00 Hz
PC  1.40

$^{13}$C NMR spectrum of compound 3k
NMR spectrum of compound 3k

DEPT-135 NMR spectrum of compound 3k
$^1$H-$^1$H COSY NMR spectrum of compound 3k
$^1$H-$^{13}$C HSQC NMR spectrum of compound 3k
Current Data Parameters
NAME  sb-kkd-reduction
EXPNO  157
PROCNO  1

F2 - Acquisition Parameters
Date_  20180310
Time  23.25
INSTRUM  spect
PROBHD  5 mm PABBO BB-
PULPROG  zg30
TD  65536
SOLVENT  CDCl3
NS  16
DS  2
SWH  8012.820 Hz
FIDRES  0.122266 Hz
AQ  4.0894465 sec
RG  50.04
DW  62.400 usec
DE  6.50 usec
TE  298.5 K
D1  0.50000000 sec
TD0  1

== CHANNEL f1 ==
SFO1  400.1320007 MHz
NUC1  1H
P1  15.70 usec
PLW1  7.75000000 W

F2 - Processing parameters
SI  65536
SF  400.1300102 MHz
WDW  EM
SSB  0
LB  0.30 Hz
GB  0
PC  1.00

^1H NMR spectrum of compound 3o
$^1$H NMR spectrum of compound 3o
lab sb-kkd-reduction
iitm_carbonshort CDCl3 /opt/topspin nmr 1

13C NMR spectrum of compound 3o
DEPT-135 NMR spectrum of compound 3o
$^1$H-$^1$H COSY NMR spectrum of compound 3o
\[ ^1H-^13C \text{ HSQC NMR spectrum of compound 3o} \]
1H NMR spectrum of compound 4r
$^1$H NMR spectrum of compound 4r
$^{13}$C NMR spectrum of compound 4r
DEPT-135 NMR spectrum of compound 4r
$^1$H-$^1$H COSY NMR spectrum of compound 4r
\[^{1}H^{13}C\] HSQC NMR spectrum of compound 4r
$^1$H NMR spectrum of compound 4q
\( ^1 \text{H NMR spectrum of compound 4q} \)
Current Data Parameters
NAME: sb-dvk-819
EXPNO: 289
PROCNO: 1

F2 - Acquisition Parameters
Date: 20180821
Time: 0.48
INSTRUM: spect
PROBHD: 5 mm PABBO BB-
PULPROG: zgpg30
TG: 1650
SOLVENT: CDCl3
NS: 256
DS: 4
SWH: 24038.461 Hz
FIDRES: 1.453353 Hz
AQ: 0.3440320 sec
RG: 200.34
DW: 20.800 usec
DE: 6.50 usec
TE: 296.0 K
D1: 1.00000000 sec
D11: 0.03000000 sec
D20: 1

------ CHANNEL f1 ------
SFO1: 100.6228289 MHz
NUC1: 13C
P1: 9.25 usec
PLW1: 47.00000000 W

------ CHANNEL f2 ------
SFO2: 400.1316005 MHz
NUC2: 1H
CPDPRG[2: waltz16
PCPD2: 90.00 usec
PLW2: 7.75000000 W
PLW12: 0.23833999 W
PLW13: 0.11863000 W

F2 - Processing parameters
SI: 32768
SF: 100.6127565 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

\[^{13}\text{C}\] NMR spectrum of compound 4q
Current Data Parameters
NAME       sb-dvk-819
EXPNO               290
PROCNO                1
F2 - Acquisition Parameters
Date_          20180821
Time               0.51
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG       deptsp135
TD                32768
SOLVENT           CDCl3
NS                   64
DS                    4
SWH           20161.291 Hz
FIDRES         0.615274 Hz
AQ            0.8126464 sec
RG               200.34
DW                24.800 usec
DE                6.50 usec
TE                295.8 K
CNST2       145.0000000
D1           1.00000000 sec
D2            0.00344828 sec
D12          0.00002000 sec
TD0                   1
-------- CHANNEL f1 --------
SFO1        100.6208166 MHz
NUC1                13C
P1                 9.25 usec
P13              2000.00 usec
PLW0      0 W
PLW1        47.0000000 W
SPNAM[5]    Crp60comp.4
SPOAL5           0.500
SPOFFS5  0 Hz
SPW5         6.1449998 W
-------- CHANNEL f2 --------
SFO2        400.1312797 MHz
NUC2                  1H
CPDPRG[2]  waltz16
P3                 15.70 usec
P4               31.40 usec
PCPD2     90.00 usec
PLW2        7.7500000 W
PLW12      0.23583999 W
F2 - Processing parameters
SF            32768
SF          100.6127690 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB            0
PC             1.40

DEPT-135 NMR spectrum of compound 4q
**1H-1H COSY NMR spectrum of compound 4q**
$^{1}H$-$^{13}C$ HSQC NMR spectrum of compound 4q
Current Data Parameters
NAME            sb-dvk-642
EXPNO               224
PROCNO                1

F2 - Acquisition Parameters
Date_          20180226
Time              15.01
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG               124.58
DW       62.400 usec
DE                 6.50 usec
TE                299.5 K
D1           0.50000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1320007 MHz
NUC1                 1H
P1                15.70 usec
PLW1         7.75000000 W

F2 - Processing parameters
SI                65536
SF          400.1300400 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00

\[^1\text{H}\] NMR spectrum of compound 4rx
\[ ^1H \text{ NMR spectrum of compound 4rx} \]
$^{13}$C NMR spectrum of compound 4rx
DEPT-135 NMR spectrum of compound 4rx
Current Data Parameters
NAME        Sb-DVK-604
EXPNO               31
PROCNO              1

F2 - Acquisition Parameters
Date_          20180105
Time              11.33
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                 31.9
DW               62.400 usec
DE                 6.50 usec
TE                291.9 K
D1           0.50000000 sec
TD0                   1

-------- CHANNEL f1 --------
SFO1        400.1320007 MHz
NUC1                 1H
P1                15.70 usec
PLW1         7.75000000 W

F2 - Processing parameters
SI                65536
SF        400.1299676 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00

1H NMR spectrum of compound 4p
$^1$H NMR spectrum of compound 4p
$^{13}$C NMR spectrum of compound 4p
**Current Data Parameters**

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**F2 - Acquisition Parameters**

- Date_          20180105
- Time              11.44
- INSTRUM           spect
- PROBHD   5 mm PABBO BB-
- PULPROG       deptsp135
- TD                32768
- SOLVENT           CDCl3
- NS                   64
- DS                    4
- SWH           20161.291 Hz
- FIRES5         0.615274 Hz
- AQ               0.8156464 sec
- RG               200.34
- DW                24.800 usec
- DE               6.50 usec
- TE                292.3 K
- CNST2       145.0000000
- D1           1.00000000 sec
- D2          0.00344828 sec
- D12        0.00002000 sec
- TD0                   1

**SFO1**

- NUC1                13C
- P1                 9.25 usec
- P13             2000.00 usec
- PLW0     0 W
- PLW1        47.00000000 W
- SPNM[5]    Crp60comp.4
- SPOALS5  0.500
- SPOFF5s  0 Hz
- SPW5         6.14429998 W

**SFO2**

- NUC2                1H
- CPDPRG[2        waltz16
- P3                15.70 usec
- P4                31.40 usec
- PCPD2             90.00 usec
- PLW2        7.75000000 W
- PLW12       0.23583999 W

**F2 - Processing parameters**

- SI                32768
- SF                100.6127690 MHz
- WDW                  EM
- SSB                  0
- LB                1.00 Hz
- GB                  0
- PC                 1.40

---

**DEPT-135 NMR spectrum of compound 4p**

---
$^1$H-$^1$H COSY NMR spectrum of compound 4p
\[ ^1H\text{-}^{13}C \] HSQC NMR spectrum of compound 4p
H NMR spectrum of compound 4o
$^1$H NMR spectrum of compound 4o
$^{13}$C NMR spectrum of compound 4o
Current Data Parameters
NAME     sb-dvk-832
EXPNO    383
PROCNO   1

F2 - Acquisition Parameters
Date_          20180829
Time               4.35
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG       deptsp135
TD                32768
SOLVENT           CDCl3
NS                   64
DS                    4
SWH           20161.291 Hz
FIDRES         0.615274 Hz
AQ            0.8126464 sec
RG               200.34
DW               24.800 usec
DE                6.50 usec
TE                300.5 K
CNST2       145.000000
D1           1.00000000 sec
D2          0.00344828 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6208166 MHz
NUC1                13C
P1                 9.25 usec
P13              2000.00 usec
PLW0          0 W
PLW1        47.00000000 W
SPNM[5](Spn60comp.4)
SPW5         6.14429998 W

======== CHANNEL f2 ========
SFO2        400.1312797 MHz
NUC2                1H
CPDPROG[2]  waltz16
P3               15.70 usec
P4                31.40 usec
PLW2        7.75000000 W
PLW12       0.23583999 W

F2 - Processing parameters
SI                32708
SI         100.8127690 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB                 0
PC                 1.40

DEPT-135 NMR spectrum of compound 4o
$^1$H-$^1$H COSY NMR spectrum of compound 4o
**Current Data Parameters**

**NAME**       sb-dvk-832
**EXPNO**       386
**PROCNO**      1

**F2 - Acquisition Parameters**

**Date**       20180829
**Time**        4.44
**INSTRUM**     spect
**PROBHD**      5 mm PABBO BB-
**PULPROG**     hsqcetgp
**TD**          1024
**SOLVENT**     CDCl3
**NS**          2
**DS**          16
**SWH**         3496.503 Hz
**FIDRES**      3.414554 Hz
**AQ**          0.1664320 sec
**RG**          203.24
**DG**          143.000 usec
**DE**          6.650 usec
**TE**          300.0 K
**CNST2**       145.0000000
**D0**          0.00000300 sec
**D1**          1.45207703 sec
**D4**          0.00172414 sec
**D11**         0.03000000 sec
**D16**         0.00020000 sec
**IN0**         0.00003000 sec

**ZDQPTNS**

======== CHANNEL f1 ========
**SFO1**         400.1315087 MHz
**NUC1**         1H
**P1**           15.70 usec
**P2**           31.40 usec
**P3**           9.25 usec
**P4**           18.50 usec
**PLW1**         7.75000000 W

======== CHANNEL f2 ========
**SFO2**         100.6202727 MHz
**NUC2**         13C
**CPDPRG[2]**    garp
**P5**           9.25 usec
**P6**           18.50 usec
**PLW2**         47.00000000 W
**PLW12**        0.62835002 W

====== GRADIENT CHANNEL =====
**GPNAME[1]**    SMSQ10.100
**GPNAME[2]**    SMSQ10.100
**GPZ1**         80.00 %
**GPZ2**         20.10 %
**P16**          1000.00 usec

**F1 - Acquisition parameters**
**TD**           256
**SFO1**         100.6127550 MHz
**FIDRES**       165.639 ppm
**FnMODE**       Echo-Antiecho

**F2 - Processing parameters**
**SI**           1024
**SF**           400.1300137 MHz
**SFCL**         100-500 Hz
**WDW**          QSINE
**SSB**          2
**LB**           0 Hz
**GB**           0

**F1 - Processing parameters**
**SI**           1024
**MC2**          echo-antiecho
**SF**           100.613750 MHz
**SFCL**         100-500 Hz

---

**1H-13C HSQCNMR spectrum of compound 4o**

---
**1H NMR spectrum of crude compound 5q**
$^1$H NMR spectrum of crude compound 5q
C NMR spectrum of crude compound 5q
DEPT-135 NMR spectrum of crude compound 5q
$^1$H-$^1$H COSY NMR spectrum of crude compound 5q
$^1$H-$^{13}$C HSQC NMR spectrum of crude compound 5q
$^1$H NMR spectrum of crude compound 5a
^{1}H NMR spectrum of crude compound 5a
$^{13}$C NMR spectrum of crude compound 5a

---

S293
DEPT-135 NMR spectrum of crude compound 5a
\textbf{current Data Parameters}

- **NAME**: sb-dvk-820
- **EXPNO**: 329
- **PROCNO**: 1

\textbf{F2 - Acquisition Parameters}

- **Date**: 20180825
- **Time**: 11.52
- **INSTRUM**: spect
- **PROBHD**: 5 mm PABBO BB-
- **PULPROG**: cosygppqf
- **TD**: 2048
- **SOLVENT**: CDCl3
- **NS**: 1
- **DS**: 8
- **SWH**: 3289.474 Hz
- **FIDRES**: 1.606188 Hz
- **AQ**: 0.3112960 sec
- **RG**: 60.89
- **DW**: 152.000 usec
- **DE**: 6.50 usec
- **TE**: 295.4 K
- **D0**: 0.00000300 sec
- **D1**: 1.88039696 sec
- **D11**: 0.00000400 sec
- **D12**: 0.00002000 sec
- **D13**: 0.00000400 sec
- **D16**: 0.00020000 sec
- **IN0**: 0.00000400 sec

\textbf{======== CHANNEL f1 ========}

- **SFO1**: 400.1317163 MHz
- **NUC1**: 1H
- **P0**: 15.70 usec
- **P1**: 15.70 usec
- **P17**: 2500.00 usec
- **PLW1**: 7.75000000 W
- **PLW10**: 2.12260008 W

\textbf{====== GRADIENT CHANNEL ======}

- **GPNAME[1]**: SMSQ10.100
- **GPZ1**: 10.00 %
- **P16**: 1000.00 usec

\textbf{F1 - Acquisition parameters}

- **TD**: 128
- **SFO1**: 400.1317 MHz
- **P2RES**: 51.398026 Hz
- **SW**: 8.221 ppm
- **FmMODE**: QF

\textbf{F2 - Processing parameters}

- **SI**: 128
- **SF**: 400.1300097 MHz
- **WDW**: QSPINE
- **SSB**: 0
- **LB**: 0 Hz
- **GB**: 0
- **PC**: 1.40

\textbf{F1 - Processing parameters}

- **SI**: 128
- **MC2**: QF
- **SF**: 400.1300097 MHz
- **WDW**: QSPINE
- **SSB**: 0
- **LB**: 0 Hz
- **GB**: 0

\text{\textbf{1H-1H COSY NMR spectrum of crude compound 5a}}
$^1$H-$^{13}$C HSQC NMR spectrum of crude compound 5a
H NMR spectrum of crude compound 5g
$^{1}$H NMR spectrum of crude compound 5g
13C NMR spectrum of crude compound 5g
DEPT-135 NMR spectrum of crude compound 5g
$^1$H-$^1$H COSY NMR spectrum of crude compound 5g
**1H-13C HSQC NMR spectrum of crude compound 5g**
$^1$H NMR spectrum of major diastereomer 5p
$^{1}$H NMR spectrum of major diastereomer 5p
$^{13}$C NMR spectrum of major diastereomer 5p
DEPT-135 NMR spectrum of major diastereomer 5p
$^1$H-$^1$H COSY NMR spectrum of major diastereomer 5p
1H-13C HSQC NMR spectrum of major diastereomer 5p
$^1$H NMR spectrum of crude compound 5k
$^1$H NMR spectrum of crude compound 5k
$^{13}$C NMR spectrum of crude compound 5k
DEPT-135 NMR spectrum of crude compound 5k
1H-1H COSY NMR spectrum of crude compound 5k
$^1$H-$^{13}$C HSQC NMR spectrum of crude compound 5k