Intermolecular Vicinal Diaminative Assembly of Tetrahydroquinoxalines via Metal-free Oxidative [4+2] Cycloaddition Strategy

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Supporting Information
**Table of contents**

1. General information.................................................................................................................................... S3
2. Reaction Condition Optimizations......................................................................................................... S3
3. Experimental procedure and characterization data for products ...................................................... S4
4. Synthetic Transformations of products .............................................................................................. S31
5. X-Ray crystallographic data of 3gq ................................................................................................... S33
6. Copies of $^1$H and $^{13}$C NMR spectra............................................................................................ S35
1. General information

Unless otherwise noted, all reagents were obtained from commercially suppliers and were used without further purification. All products were purified by flash chromatography on silica gel. The chemical yields referred are isolated products. $^1$H NMR and $^{13}$C NMR spectra were recorded on 400 MHz or 600 MHz Bruker spectrometers. Chemical shifts of $^1$H were reported in part per million (ppm) relative to the CDCl$_3$ residual peak ($\delta$ 7.26). Chemical shifts of $^{13}$C NMR were reported relative to CDCl$_3$ ($\delta$ 77.16). The used abbreviations are as follows: s (singlet), d (doublet), t (triplet), quart. (quartet), m (multiplet), br (broad). High resolution mass spectra (HRMS) data were measured on a FT-ICR-MS SolariX 7T. Melting points were measured on a SGW® X-4B and are not corrected. Reactions were monitored by TLC analysis using silica gel 60 Å F-254 thin layer plates and compounds were visualized with a UV light at 254 nm. Flash column chromatography was performed on silica gel 60 Å, 10 – 40 μm.

o-Phenylenediamine derivatives were synthesized according to the literature procedure.1-2 o-Benzoquinone diimide 6a was synthesized according to the literature procedure.3

2. Reaction Condition Optimizations

Table S1. The effect of solvents

<table>
<thead>
<tr>
<th>Entry</th>
<th>Solvent</th>
<th>Yieldb (%)</th>
<th>Entry</th>
<th>Solvent</th>
<th>Yieldb (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>DCE</td>
<td>67</td>
<td>8</td>
<td>CH$_3$OH</td>
<td>27</td>
</tr>
<tr>
<td>2</td>
<td>CHCl$_3$</td>
<td>59</td>
<td>9</td>
<td>THF</td>
<td>82</td>
</tr>
<tr>
<td>3</td>
<td>DCM</td>
<td>56</td>
<td>10</td>
<td>HFIP</td>
<td>22</td>
</tr>
<tr>
<td>4</td>
<td>MTBE</td>
<td>60</td>
<td>11</td>
<td>CH$_3$CN</td>
<td>45</td>
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<tr>
<td>5</td>
<td>EtOAc</td>
<td>59</td>
<td>12</td>
<td>Acetone</td>
<td>62</td>
</tr>
<tr>
<td>6</td>
<td>Toluene</td>
<td>60</td>
<td>13</td>
<td>Hexane</td>
<td>67</td>
</tr>
<tr>
<td>7</td>
<td>Et$_2$O</td>
<td>71</td>
<td>14</td>
<td>H$_2$O</td>
<td>20</td>
</tr>
</tbody>
</table>

$^a$The reactions were carried out with 1a (0.1 mmol, 1 equiv) and 2a (0.2 mmol, 2 equiv) in the presence of Ph(I(OAc)$_2$ (0.105 mmol, 1.05 equiv.) in 1 mL solvent at room temperature for 5h in a sealed tube. $^b$Isolated yields are given.
Table S2. The effect of the ratio of reactants

<table>
<thead>
<tr>
<th>Entry</th>
<th>1a</th>
<th>2a</th>
<th>Yieldb (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.1 mmol</td>
<td>0.3 mmol</td>
<td>83</td>
</tr>
<tr>
<td>2</td>
<td>0.1 mmol</td>
<td>0.2 mmol</td>
<td>82</td>
</tr>
<tr>
<td>3</td>
<td>0.1 mmol</td>
<td>0.15 mmol</td>
<td>85</td>
</tr>
<tr>
<td>4</td>
<td>0.12 mmol</td>
<td>0.1 mmol</td>
<td>82</td>
</tr>
<tr>
<td>5</td>
<td>0.15 mmol</td>
<td>0.1 mmol</td>
<td>81</td>
</tr>
</tbody>
</table>

Note: The reactions were carried out with 1a and 2a in the presence of PIDA (0.105 mmol, 1.05 equiv.) in 1 mL THF at room temperature for 5h in a sealed tube. Isolated yields are given.

Table S3. The effect of oxidants

<table>
<thead>
<tr>
<th>Entry</th>
<th>Oxidant</th>
<th>Yieldb (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.05 equiv. PIFA</td>
<td>72</td>
</tr>
<tr>
<td>2</td>
<td>1.05 equiv. IBX</td>
<td>trace</td>
</tr>
<tr>
<td>3</td>
<td>1.05 equiv. PhIO</td>
<td>17</td>
</tr>
<tr>
<td>4</td>
<td>1 equiv. PIDA</td>
<td>80</td>
</tr>
<tr>
<td>5</td>
<td>1.05 equiv. PIDA</td>
<td>85</td>
</tr>
<tr>
<td>6</td>
<td>1.2 equiv. PIDA</td>
<td>85</td>
</tr>
<tr>
<td>7</td>
<td>1.5 equiv. PIDA</td>
<td>86</td>
</tr>
</tbody>
</table>

Note: The reactions were carried out with 1a (0.1 mmol, 1 equiv) and 2a (0.15 mmol, 1.5 equiv) in the presence of oxidant in 1 mL THF at room temperature for 5h in a sealed tube. Isolated yields are given.

3. Experimental procedure and characterization data for products

A typical experimental procedure:

To a sealed tube was added 1 (0.1 mmol, 1 equiv), PIDA (0.105 mmol, 1.05 equiv.) and 1 mL THF, then added 2 (0.15 mmol, 1.5 equiv), the reaction mixture was stirred at room temperature for 5 hours. Afterword, the solvent was evaporated in vacuo. The residue was purified by column chromatography on silica gel to give the product 3.
Scale-up synthesis of 3aa and 3ua:

A round bottom flask equipped with a magnetic stir bar was charged with \( N,N'-(4,5\text{-dichloro-1,2-phenylene})\text{bis}(4\text{-methylbenzenesulfonamide}) \) (1a) (972 mg, 2.0 mmol), PIDA (680 mg, 2.1 mmol) and 20 mL THF, then added styrene (2a) (348 ul, 3.0 mmol), the reaction mixture was stirred at room temperature for 5 hours. Afterword, the solvent was evaporated in vacuo. The residue was purified by column chromatography on silica gel (petroleum ether/dichloromethane = 2/1) to give the product 3aa as a white solid (926 mg, 94% yield).

2,3-Dichloro-6-methyl-5,11-ditosyl-5a,6,10b,11-tetrahydro-5H-indolo[2,3-b]quinoxaline (3ax) was synthesized analogously as a white solid (969 mg, 79% yield).

6,7-Dichloro-2-phenyl-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3aa)

Product 3aa was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2a (0.15 mmol, 15.6 mg) by following the general procedure.

A white solid, 49.8 mg, 85% yield.

\textbf{m.p.}: 188 – 190 °C.

\textbf{TLC}: \( R_f = 0.80 \) (dichloromethane/petroleum ether = 2:1) [UV].

\textbf{\( ^1\text{H NMR} \)} (600 MHz, CDCl\(_3\)) \( \delta \)

- 8.01 (s, 1H),
- 7.61 (s, 1H),
- 7.47 (d, \( J = 8.1 \) Hz, 2H),
- 7.32 – 7.28 (m,
- 7.26 – 7.19 (m, 6H),
- 7.12 (d, \( J = 8.2 \) Hz, 2H),
- 5.61 (t, \( J = 4.7 \) Hz, 1H),
- 4.29 (dd, \( J = 13.4, 5.0 \) Hz, 1H),
- 3.41 (dd, \( J = 13.4, 4.6 \) Hz, 1H),
- 2.43 (s, 3H),
- 2.37 (s, 3H).

\textbf{\( ^{13}\text{C NMR} \)} (101 MHz, CDCl\(_3\)) \( \delta \)

- 145.0 (s),
- 144.8 (s),
- 137.1 (s),
- 135.0 (s),
- 134.9 (s),
- 130.2 (s),
- 130.1 (s),
- 130.0 (s),
- 129.2 (s),
- 129.1 (s),
- 128.2 (s),
- 127.5 (s),
- 127.5 (s),
- 126.6 (s),
- 126.5 (s),
- 121.0 (s),
- 57.4 (s),
- 48.3 (s),
- 21.8 (s),
- 21.7 (s). (The deficiency of carbon signals is probably because some of them coincide with identical chemical shift)

\textbf{HRMS (ESI)}: \text{C}_{28}\text{H}_{25}\text{Cl}_2\text{N}_2\text{O}_4\text{S}_2 [(\text{M+H})^+]$: calcd.: 587.0627; found: 587.0626.

\textbf{IR (ATR/cm\(^{-1}\))}: 2921, 1595, 1475, 1357, 1166, 1066, 899.
6,7-Dichloro-2-(o-tolyl)-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3ab)

Product 3ab was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2b (0.15 mmol, 17.7 mg) by following the general procedure.
A white solid, 48.0 mg, 80% yield.

m.p.: 119 – 121 °C.

TLC: \( R_f = 0.55 \) (dichloromethane/petroleum ether = 1:1) [UV].

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.89 (s, 1H), 7.56 (s, 1H), 7.45 (d, \( J = 8.4 \) Hz, 2H), 7.34 (d, \( J = 8.4 \) Hz, 2H), 7.24 (d, \( J = 8.1 \) Hz, 2H), 7.22 – 7.16 (m, 4H), 7.06 – 6.95 (m, 2H), 5.66 (dd, \( J = 8.5, 5.7 \) Hz, 1H), 3.86 (dd, \( J = 14.0, 5.7 \) Hz, 1H), 3.57 (dd, \( J = 14.0, 8.5 \) Hz, 1H), 2.46 (s, 3H), 2.40 (s, 6H).

\(^{13}\)C NMR (151 MHz, CDCl\(_3\)) \( \delta \) 144.9 (s), 144.9 (s), 136.3 (s), 135.8 (s), 135.0 (s), 134.8 (s), 131.5 (s), 131.1 (s), 130.4 (s), 130.3 (s), 130.0 (s), 128.9 (s), 128.8 (s), 128.3 (s), 127.6 (s), 127.3 (s), 126.8 (s), 126.7 (s), 125.8 (s), 123.6 (s), 58.0 (s), 50.4 (s), 21.8 (s), 21.7 (s), 19.3 (s).

HRMS (ESI): \( \text{C}_{29}\text{H}_{27}\text{Cl}_{2}\text{N}_{2}\text{O}_{4}\text{S}_{2}^{+}\) [M+H]*: calcd.: 601.0784; found: 601.0785.

IR (ATR/cm\(^{-1}\)) 2922, 1597, 1473, 1355, 1162, 1063, 964, 891, 815.

6,7-Dichloro-2-(m-tolyl)-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3ac)

Product 3ac was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2c (0.15 mmol, 17.7 mg) by following the general procedure.
A white solid, 44.4 mg, 74% yield.

m.p.: 181 – 183 °C.

TLC: \( R_f = 0.52 \) (dichloromethane/petroleum ether = 1:1) [UV].
**1H NMR** (400 MHz, CDCl₃) δ 8.02 (s, 1H), 7.62 (s, 1H), 7.45 (d, J = 8.3 Hz, 2H), 7.26 – 7.20 (m, 4H), 7.17 (t, J = 7.5 Hz, 1H), 7.14 – 7.07 (m, 3H), 7.03 – 6.98 (m, 2H), 5.55 (t, J = 4.9 Hz, 1H), 4.22 (dd, J = 13.4, 5.3 Hz, 1H), 3.42 (dd, J = 13.4, 4.7 Hz, 1H), 2.43 (s, 3H), 2.37 (s, 3H), 2.28 (s, 3H).

**13C NMR** (101 MHz, CDCl₃) δ 145.0 (s), 144.8 (s), 138.8 (s), 137.2 (s), 135.1 (s), 135.0 (s), 130.3 (s), 130.2 (s), 130.0 (s), 129.2 (s), 129.0 (s), 127.6 (s), 127.6 (s), 127.5 (s), 127.2 (s), 127.0 (s), 126.5 (s), 123.6 (s), 121.3 (s), 57.8 (s), 48.7 (s), 21.8 (s), 21.7 (s), 21.6 (s). (the deficiency of carbon signals is probably because some of them coincide with identical chemical shift)

**HRMS (ESI)**: C₂₉H₂₇Cl₂N₂O₄S₂ [(M+H)+]: calcd.: 601.0784; found: 601.0786.

**IR** (ATR/cm⁻¹) 2918, 2848, 1645, 1596, 1469, 1359, 1166, 1088, 1064, 905, 809.

**6,7-Dichloro-2-(3-chlorophenyl)-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3ad)**

![image of 3ad]

Product 3ad was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2d (0.15 mmol, 20.7 mg) by following the general procedure.

A white solid, 45.3 mg, 73% yield.

**m.p.:** 189 – 191 °C.

**TLC:** Rₚ = 0.55 (dichloromethane/petroleum ether = 1:1) [UV].

**1H NMR** (600 MHz, CDCl₃) δ 8.03 (s, 1H), 7.65 (s, 1H), 7.46 (d, J = 8.3 Hz, 2H), 7.31 (d, J = 8.3 Hz, 2H), 7.27 – 7.24 (m, 3H), 7.23 (t, J = 7.6 Hz, 1H), 7.19 – 7.12 (m, 4H), 5.54 (t, J = 5.1 Hz, 1H), 4.12 (dd, J = 13.5, 5.5 Hz, 1H), 3.45 (dd, J = 13.5, 4.8 Hz, 1H), 2.44 (s, 3H), 2.39 (s, 3H).

**13C NMR** (151 MHz, CDCl₃) δ 145.2 (s), 145.0 (s), 139.4 (s), 135.0 (s), 135.0 (s), 134.5 (s), 130.4 (s), 130.3 (s), 130.2 (s), 130.1 (s), 129.5 (s), 128.5 (s), 127.9 (s), 127.5 (s), 127.3 (s), 126.8 (s), 126.8 (s), 126.6 (s), 124.7 (s), 121.4 (s), 57.6 (s), 48.6 (s), 21.8 (s), 21.7 (s).

**HRMS (ESI)**: C₂₈H₂₇Cl₂N₂O₄S₂ [(M+H)+]: calcd.: 621.0238; found: 621.0238.

**IR** (ATR/cm⁻¹) 2920, 1590, 1470, 1360, 1160, 1090, 1060, 901, 820.
6,7-Dichloro-2-(p-tolyl)-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3ae)

Product 3ae was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2e (0.15 mmol, 17.7 mg) by following the general procedure.

A white solid, 45.0 mg, 75% yield.

**m.p.:** 231 – 233 °C.

**TLC:** $R_f = 0.49$ (dichloromethane/petroleum ether = 1:1) [UV].

**$^1$H NMR** (400 MHz, CDCl$_3$) $\delta$ 7.99 (s, 1H), 7.63 (s, 1H), 7.46 (d, $J = 8.3$ Hz, 2H), 7.26 – 7.20 (m, 4H), 7.14 – 7.06 (m, 6H), 5.56 (t, $J = 4.6$ Hz, 1H), 4.28 (dd, $J = 13.3$, 4.9 Hz, 1H), 3.38 (dd, $J = 13.3$, 4.6 Hz, 1H), 2.43 (s, 3H), 2.38 (s, 3H), 2.33 (s, 3H).

**$^{13}$C NMR** (151 MHz, CDCl$_3$) $\delta$ 144.9 (s), 144.7 (s), 138.1 (s), 135.0 (s), 134.0 (s), 130.2 (s), 129.7 (s), 129.2 (s), 127.5 (s), 127.4 (s), 126.7 (s), 126.6 (s), 126.5 (s), 120.9 (s), 57.2 (s), 48.3 (s), 21.8 (s), 21.8 (s), 21.2 (s).

(The deficiency of carbon signals is probably because some of them coincide with identical chemical shift)

**HRMS (ESI):** C$_{29}$H$_{27}$Cl$_2$N$_2$O$_4$S$_2$ [(M+H)$^+$]: calcld.: 601.0784; found: 601.0787.

**IR (ATR/cm$^{-1}$)** 2920, 1596, 1354, 1166, 1065, 1032, 890, 806.

2-(4-(tert-Butyl)phenyl)-6,7-dichloro-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3af)

Product 3af was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2f (0.15 mmol, 24.0 mg) by following the general procedure.

A white solid, 50.1 mg, 78% yield.

**m.p.:** 208 – 210 °C.

**TLC:** $R_f = 0.58$ (dichloromethane/petroleum ether = 1:1) [UV].
$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.99 (s, 1H), 7.60 (s, 1H), 7.44 (d, $J = 8.3$ Hz, 2H), 7.32 (d, $J = 8.4$ Hz, 2H), 7.24 (t, $J = 8.9$ Hz, 4H), 7.17 (d, $J = 8.3$ Hz, 2H), 7.13 (d, $J = 8.2$ Hz, 2H), 5.56 (t, $J = 4.8$ Hz, 1H), 4.27 (dd, $J = 13.3$, 5.1 Hz, 1H), 3.42 (dd, $J = 13.3$, 4.7 Hz, 1H), 2.43 (s, 3H), 2.37 (s, 3H), 1.30 (s, 9H).

$^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 151.3 (s), 144.9 (s), 144.8 (s), 135.0 (s), 135.0 (s), 133.9 (s), 130.2 (s), 130.2 (s), 129.2 (s), 127.5 (s), 127.5 (s), 127.5 (s), 126.7 (s), 126.7 (s), 126.3 (s), 126.0 (s), 121.0 (s), 57.3 (s), 48.3 (s), 34.7 (s), 31.5 (s), 21.8 (s), 21.7 (s).

HRMS (ESI): C$_{32}$H$_{36}$Cl$_2$N$_3$O$_4$S$_2$ [(M+NH$_4$)$^+$]: calcd.: 660.1519; found: 660.1522.

IR (ATR/cm$^{-1}$) 2964, 1595, 1469, 1354, 1167, 1088, 1065, 889, 811.

6,7-Dichloro-2-(4-methoxyphenyl)-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3ag)

![Chemical Structure](image)

Product 3ag was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2g (0.3 mmol, 40.2 mg) by following the general procedure.

A yellow solid, 43.1 mg, 70% yield.

m.p.: 217 – 218 oC.

TLC: $R_f = 0.34$ (dichloromethane/petroleum ether = 1:1) [UV].

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.97 (s, 1H), 7.67 (s, 1H), 7.46 (d, $J = 8.3$ Hz, 2H), 7.27 – 7.21 (m, 4H), 7.13 (d, $J = 8.8$ Hz, 4H), 6.83 – 6.74 (m, 2H), 5.55 (t, $J = 4.6$ Hz, 1H), 4.26 (dd, $J = 13.3$, 4.8 Hz, 1H), 3.78 (s, 3H), 3.37 (dd, $J = 13.3$, 4.6 Hz, 1H), 2.43 (s, 3H), 2.38 (s, 3H).

$^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 159.5 (s), 144.9 (s), 144.8 (s), 135.1 (s), 135.1 (s), 130.2 (s), 130.1 (s), 130.0 (s), 129.2 (s), 129.0 (s), 127.8 (s), 127.5 (s), 127.5 (s), 127.4 (s), 126.7 (s), 126.6 (s), 121.0 (s), 114.4 (s), 56.9 (s), 55.4 (s), 48.5 (s), 21.8 (s), 21.7 (s).

HRMS (ESI): C$_{29}$H$_{27}$Cl$_2$N$_2$O$_5$S$_2$ [(M+H)$^+$]: calcd.: 617.0733; found: 617.0736.

IR (ATR/cm$^{-1}$) 2919, 1594, 1513, 1468, 1354, 1262, 1166, 1065, 1029, 886, 809.
6,7-Dichloro-2-(4-fluorophenyl)-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3ah)

Product 3ah was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2h (0.15 mmol, 18.3 mg) by following the general procedure.

A white solid, 52.0 mg, 86% yield.

m.p.: 136 – 138 °C.

TLC: $R_t = 0.48$ (dichloromethane/petroleum ether = 1:1) [UV].

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.01 (s, 1H), 7.67 (s, 1H), 7.46 (d, $J = 8.3$ Hz, 2H), 7.30 – 7.22 (m, 4H), 7.22 – 7.18 (m, 2H), 7.16 (d, $J = 8.1$ Hz, 2H), 7.00 – 6.92 (m, 2H), 5.57 (t, $J = 4.7$ Hz, 1H), 4.23 (dd, $J = 13.4$, 5.0 Hz, 1H), 3.38 (dd, $J = 13.4$, 4.6 Hz, 1H), 2.44 (s, 3H), 2.39 (s, 3H).

$^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 162.5 (d, $J = 247.6$ Hz), 145.1 (s), 144.9 (s), 135.0 (s), 134.7 (s), 132.9 (d, $J = 3.1$ Hz), 130.3 (s), 130.0 (s), 129.4 (s), 128.4 (d, $J = 8.3$ Hz), 127.7 (s), 127.5 (s), 127.3 (s), 126.6 (s), 126.4 (s), 121.1 (s), 116.1 (s), 115.9 (s), 56.9 (s), 48.4 (s), 21.8 (s), 21.7 (s).

HRMS (ESI): C$_{28}$H$_{24}$Cl$_2$FN$_2$O$_4$S$_2$ [(M+H)$^+$]: calcd.: 605.0533; found: 605.0534.

IR (ATR/cm$^{-1}$) 2922, 1596, 1469, 1359, 1166, 1066, 904, 810.

6,7-Dichloro-2-(4-chlorophenyl)-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3ai)

Product 3ai was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2i (0.15 mmol, 20.7 mg) by following the general procedure.

A white solid, 51.5 mg, 83% yield.

m.p.: 204 – 205 °C.

TLC: $R_t = 0.52$ (dichloromethane/petroleum ether = 1:1) [UV].
$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.03 (s, 1H), 7.70 (s, 1H), 7.47 (d, $J = 8.3$ Hz, 2H), 7.29 – 7.20 (m, 6H), 7.18 – 7.11 (m, 4H), 5.57 (t, $J = 4.5$ Hz, 1H), 4.26 (dd, $J = 13.5$, 4.8 Hz, 1H), 3.33 (dd, $J = 13.5$, 4.4 Hz, 1H), 2.44 (s, 3H), 2.39 (s, 3H).

$^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 145.2 (s), 145.0 (s), 135.7 (s), 134.9 (s), 134.6 (s), 134.2 (s), 130.3 (s), 130.0 (s), 129.8 (s), 129.4 (s), 129.2 (s), 128.0 (s), 127.7 (s), 127.5 (s), 127.3 (s), 126.5 (s), 126.2 (s), 121.0 (s), 56.8 (s), 48.1 (s), 21.8 (s), 21.8 (s).

HRMS (ESI): C$_{28}$H$_{24}$Cl$_3$N$_2$O$_4$S$_2$ [(M+H)$^+$]: calcd.: 621.0238; found: 621.0236.

IR (ATR/cm$^{-1}$) 2920, 1595, 1469, 1356, 1167, 1066, 891, 809.

2-(4-Bromophenyl)-6,7-dichloro-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3aj)

Product 3aj was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2j (0.15 mmol, 27.3 mg) by following the general procedure.

A white solid, 48.5 mg, 73% yield.

m.p.: 236 – 238 °C.

TLC: $R_f = 0.60$ (dichloromethane/petroleum ether = 1:1) [UV].

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.06 (s, 1H), 7.72 (s, 1H), 7.49 (d, $J = 8.3$ Hz, 2H), 7.41 (d, $J = 8.5$ Hz, 2H), 7.30 – 7.24 (m, 4H), 7.17 (d, $J = 8.3$ Hz, 2H), 7.10 (d, $J = 8.4$ Hz, 2H), 5.58 (t, $J = 4.5$ Hz, 1H), 4.29 (dd, $J = 13.5$, 4.8 Hz, 1H), 3.35 (dd, $J = 13.5$, 4.4 Hz, 1H), 2.46 (s, 3H), 2.42 (s, 3H).

$^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 145.2 (s), 145.0 (s), 136.2 (s), 135.0 (s), 134.7 (s), 132.2 (s), 130.3 (s), 130.1 (s), 129.8 (s), 129.5 (s), 128.3 (s), 127.7 (s), 127.6 (s), 127.3 (s), 126.5 (s), 126.2 (s), 122.4 (s), 121.1 (s), 56.9 (s), 48.1 (s), 21.8 (s), 21.8 (s).

HRMS (ESI): C$_{28}$H$_{23}$BrCl$_2$N$_2$NaO$_4$S$_2$ [(M+Na)$^+$]: calcd.: 686.9552; found: 686.9556.

HRMS (ESI): C$_{28}$H$_{23}$BrCl$_2$N$_2$NaO$_4$S$_2$ [(M+Na)$^+$]: calcd.: 688.9531; found: 688.9535.

IR (ATR/cm$^{-1}$) 2919, 1594, 1469, 1356, 1166, 1065, 890, 809.
4-(6,7-Dichloro-1,4-ditosyl-1,2,3,4-tetrahydroquinoxalin-2-yl)phenyl acetate (3ak)

Product 3ak was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2k (0.15 mmol, 24.3 mg) by following the general procedure.

A yellow solid, 48.3 mg, 75% yield.

m.p.: 223 – 224 °C.

TLC: Rf = 0.26 (dichloromethane/petroleum ether = 1:1) [UV].

1H NMR (400 MHz, CDCl3) δ 7.99 (s, 1H), 7.62 (s, 1H), 7.45 (d, J = 8.3 Hz, 2H), 7.30 – 7.22 (m, 6H), 7.18 (d, J = 8.2 Hz, 2H), 7.02 (d, J = 8.6 Hz, 2H), 5.58 (t, J = 4.8 Hz, 1H), 4.22 (dd, J = 13.4, 5.2 Hz, 1H), 3.43 (dd, J = 13.4, 4.7 Hz, 1H), 2.43 (s, 3H), 2.38 (s, 3H), 2.30 (s, 3H).

13C NMR (101 MHz, CDCl3) δ 169.3 (s), 150.6 (s), 145.1 (s), 145.0 (s), 134.9 (s), 134.8 (s), 134.6 (s), 130.3 (s), 130.2 (s), 129.4 (s), 127.8 (s), 127.7 (s), 127.5 (s), 127.4 (s), 126.7 (s), 126.6 (s), 122.3 (s), 121.2 (s), 57.3 (s), 48.4 (s), 21.8 (s), 21.7 (s), 21.2 (s).

HRMS (ESI): C30H27Cl2N2O6S2 [(M+H)+]: calcd.: 645.0682; found: 645.0683.

IR (ATR/cm−1) 2921, 1759, 1596, 1472, 1356, 1330, 1207, 1161, 1069, 911, 808.

6,7-Dichloro-2-(4-nitrophenyl)-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3al)

Product 3al was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2l (0.3 mmol, 44.7 mg) by following the general procedure.

A light yellow oil, 38.5 mg, 61% yield.

TLC: Rf = 0.46 (dichloromethane/petroleum ether = 2:1) [UV].

1H NMR (400 MHz, CDCl3) δ 8.11 (d, J = 8.8 Hz, 2H), 8.07 (s, 1H), 7.66 (s, 1H), 7.48 (d, J = 8.3 Hz, 2H), 7.44 (d, J = 8.7 Hz, 2H), 7.35 (d, J = 8.3 Hz, 2H), 7.26 (d, J = 8.1 Hz, 2H), 7.16 (d, J = 8.2
S13 Hz, 2H), 5.68 (t, J = 4.9 Hz, 1H), 4.20 (dd, J = 13.6, 5.3 Hz, 1H), 3.44 (dd, J = 13.6, 4.6 Hz, 1H), 2.44 (s, 3H), 2.38 (s, 3H).

$^{13}$C NMR (101 MHz, CDCl$_3$) δ 147.7 (s), 145.5 (s), 145.2 (s), 144.4 (s), 135.2 (s), 134.2 (s), 130.4 (s), 130.1 (s), 130.0 (s), 129.8 (s), 128.2 (s), 127.6 (s), 127.6 (s), 127.1 (s), 126.5 (s), 126.5 (s), 124.2 (s), 121.4 (s), 57.6 (s), 48.3 (s), 21.8 (s), 21.7 (s).

HRMS (ESI): C$_{28}$H$_{23}$Cl$_2$N$_3$NaO$_6$S$_2$ [(M+Na)$^+$]: calcld.: 654.0298; found: 654.0297.

IR (ATR/cm$^{-1}$) 2920, 1600, 1520, 1470, 1350, 1170, 1060, 895.

6,7-Dichloro-1,4-ditosyl-2-(4-(trifluoromethyl)phenyl)-1,2,3,4-tetrahydroquinoxaline (3am)

Product 3am was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2m (0.3 mmol, 51.6 mg) by following the general procedure.

A white solid, 47.1 mg, 72% yield.

m.p.: 217 – 219 °C.

TLC: $R_f = 0.69$ (dichloromethane/petroleum ether = 2:1) [UV].

$^1$H NMR (400 MHz, CDCl$_3$) δ 8.08 (s, 1H), 7.67 (s, 1H), 7.54 (d, J = 8.2 Hz, 2H), 7.48 (d, J = 8.3 Hz, 2H), 7.36 (d, J = 8.1 Hz, 2H), 7.28 – 7.22 (m, 4H), 7.12 (d, J = 8.2 Hz, 2H), 6.65 (t, J = 4.5 Hz, 1H), 4.28 (dd, J = 13.5, 5.0 Hz, 1H), 3.40 (dd, J = 13.5, 4.5 Hz, 1H), 2.44 (s, 3H), 2.37 (s, 3H).

$^{13}$C NMR (151 MHz, CDCl$_3$) δ 145.3 (s), 145.1 (s), 141.3 (s), 135.0 (s), 134.5 (s), 130.6 (q, J = 32.7 Hz), 130.4 (s), 130.1 (s), 129.9 (s), 129.6 (s), 127.9 (s), 127.6 (s), 127.3 (s), 127.0 (s), 126.5 (s), 126.3 (s), 126.1 (q, J = 3.6 Hz), 124.8 (s), 123.0 (s), 121.2 (s), 57.3 (s), 48.2 (s), 21.8 (s), 21.7 (s). (the deficiency of carbon signals is probably because some of them coincide with identical chemical shift)

HRMS (ESI): C$_{29}$H$_{24}$Cl$_2$F$_3$N$_2$O$_4$S$_2$ [(M+H)$^+$]: calcld.: 655.0501; found: 655.0501.

IR (ATR/cm$^{-1}$) 2918, 1469, 1361, 1319, 1166, 1066, 891, 810.
6,7-Dichloro-2-(naphthalen-2-yl)-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3an)

Product 3an was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2n (0.15 mmol, 23.1 mg) by following the general procedure. A white solid, 51.5 mg, 81% yield.

**m.p.:** 190 – 192 °C.

**TLC:** $R_f = 0.50$ (dichloromethane/petroleum ether = 1:1) [UV].

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.13 (s, 1H), 7.81 (t, $J = 8.0$ Hz, 2H), 7.73 (s, 1H), 7.65 (d, $J = 7.6$ Hz, 1H), 7.53 – 7.43 (m, 5H), 7.36 (dd, $J = 8.6$, 1.8 Hz, 1H), 7.25 (d, $J = 9.5$ Hz, 2H), 7.04 (d, $J = 8.4$ Hz, 2H), 6.73 (d, $J = 8.1$ Hz, 2H), 5.77 (t, $J = 4.3$ Hz, 1H), 4.47 (dd, $J = 13.6$, 4.6 Hz, 1H), 3.40 (dd, $J = 13.6$, 4.4 Hz, 1H), 2.43 (s, 3H), 2.21 (s, 3H).

$^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 145.1 (s), 144.6 (s), 144.6 (s), 134.6 (s), 134.4 (s), 133.3 (s), 133.0 (s), 130.3 (s), 129.7 (s), 129.2 (s), 129.1 (s), 128.4 (s), 127.7 (s), 127.5 (s), 127.3 (s), 126.7 (s), 126.6 (s), 126.4 (s), 126.3 (s), 125.8 (s), 124.1 (s), 121.0 (s), 57.3 (s), 48.1 (s), 21.8 (s), 21.6 (s). (the deficiency of carbon signals is probably because some of them coincide with identical chemical shift)

**HRMS (ESI):** C$_{32}$H$_{27}$Cl$_2$N$_2$O$_4$S$_2$ [(M+H)$^+$]: calcd.: 637.0784; found: 637.0783.

**IR** (ATR/cm$^{-1}$) 2922, 1595, 1474, 1356, 1152, 1065, 893, 821.

6,7-Dichloro-2-methyl-3-phenyl-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3ao)

Product 3ao was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2o (0.15 mmol, 17.7 mg) by following the general procedure. A white solid, 53.4 mg, 89% yield.

**m.p.:** 223 – 225 °C.
TLC: $R_f = 0.46$ (dichloromethane/petroleum ether = 1:1) [UV].

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.88 (s, 1H), 7.69 (d, $J = 8.3$ Hz, 2H), 7.65 (s, 1H), 7.34 (t, $J = 7.3$ Hz, 1H), 7.29 – 7.23 (m, 4H), 7.08 (d, $J = 7.6$ Hz, 2H), 6.91 (d, $J = 8.2$ Hz, 2H), 6.78 (d, $J = 8.3$ Hz, 2H), 5.90 (d, $J = 2.0$ Hz, 1H), 5.28 (qd, $J = 6.5$, 2.4 Hz, 1H), 2.40 (s, 3H), 2.28 (s, 3H), 1.37 (d, $J = 6.6$ Hz, 3H).

$^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 145.2 (s), 144.4 (s), 138.6 (s), 136.3 (s), 134.8 (s), 130.0 (s), 129.7 (s), 129.2 (s), 128.0 (s), 127.9 (s), 126.7 (s), 126.3 (s), 125.4 (s), 124.8 (s), 120.6 (s), 120.5 (s), 61.0 (s), 53.7 (s), 21.7 (s), 21.6 (s), 20.4 (s). (the deficiency of carbon signals is probably because some of them coincide with identical chemical shift)

HRMS (ESI): C$_{29}$H$_{27}$Cl$_2$N$_2$O$_4$S$_2$ [(M+H)$^+$]: calcd.: 601.0784; found: 601.0786.

IR (ATR/cm$^{-1}$) 2920, 1595, 1489, 1344, 1155, 1078, 1006, 912, 805.

$(6,7$-Dichloro-$3$-phenyl-1,4-ditosyl-$1,2,3,4$-tetrahydroquinoxalin-$2$-yl)methyl acetate (3ap)

Product 3ap was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2p (0.15 mmol, 26.4 mg) by following the general procedure.

A light yellow solid, 34.8 mg, 53% yield.

m.p.: 146 – 148 °C.

TLC: $R_f = 0.35$ (dichloromethane) [UV].

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.95 (s, 1H), 7.70 (s, 1H), 7.68 (d, $J = 8.4$ Hz, 2H), 7.37 (t, $J = 7.3$ Hz, 1H), 7.31 – 7.27 (m, 3H), 7.11 (d, $J = 7.6$ Hz, 2H), 6.87 (d, $J = 8.1$ Hz, 2H), 6.69 (d, $J = 8.4$ Hz, 2H), 6.12 (d, $J = 1.7$ Hz, 1H), 5.47 – 5.40 (m, 1H), 4.21 (dd, $J = 11.3$, 5.9 Hz, 1H), 3.85 (dd, $J = 11.3$, 8.4 Hz, 1H), 2.51 – 2.36 (m, 4H), 2.27 (s, 3H), 2.19 (s, 3H).

$^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 170.7 (s), 145.5 (s), 144.6 (s), 137.9 (s), 135.8 (s), 134.2 (s), 130.2 (s), 129.7 (s), 129.5 (s), 128.3 (s), 128.2 (s), 127.8 (s), 127.1 (s), 126.8 (s), 126.4 (s), 125.0 (s), 124.4 (s), 120.7 (s), 120.6 (s), 63.1 (s), 56.2 (s), 56.0 (s), 21.7 (s), 21.6 (s), 21.0 (s).

HRMS (ESI): C$_{31}$H$_{29}$Cl$_2$N$_2$O$_6$S$_2$ [(M+H)$^+$]: calcd.: 659.0839; found: 659.0842.

IR (ATR/cm$^{-1}$) 2919, 1744, 1596, 1479, 1343, 1229, 1161, 1072, 816.
2-Butoxy-6,7-dichloro-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3aq)

![Chemical Structure](image)

Product 3aq was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2q (0.15 mmol, 15.0 mg) by following the general procedure.

A white solid, 54.1 mg, 93% yield.

**m.p.:** 152 – 154 °C.

**TLC:** \( R_f = 0.81 \) (dichloromethane/petroleum ether = 2:1) [UV].

**\(^1\)H NMR** (600 MHz, CDCl\(_3\)) \( \delta \) 7.96 (s, 1H), 7.87 (s, 1H), 7.68 (d, \( J = 8.2 \) Hz, 2H), 7.45 (d, \( J = 8.2 \) Hz, 2H), 7.26 (d, \( J = 8.0 \) Hz, 2H), 7.19 (d, \( J = 8.2 \) Hz, 2H), 5.53 (t, \( J = 2.4 \) Hz, 1H), 4.41 (dd, \( J = 13.6, 2.5 \) Hz, 1H), 3.60 (dt, \( J = 9.5, 6.8 \) Hz, 1H), 3.52 (dt, \( J = 9.6, 6.4 \) Hz, 1H), 2.87 (dd, \( J = 13.6, 2.6 \) Hz, 1H), 2.42 (s, 3H), 2.38 (s, 3H), 1.40 – 1.32 (m, 2H), 1.17 – 1.09 (m, 2H), 0.84 (t, \( J = 7.4 \) Hz, 3H).

**\(^{13}\)C NMR** (151 MHz, CDCl\(_3\)) \( \delta \) 145.3 (s), 144.7 (s), 134.9 (s), 134.6 (s), 130.4 (s), 129.6 (s), 129.3 (s), 128.7 (s), 128.3 (s), 127.2 (s), 126.9 (s), 126.5 (s), 125.3 (s), 120.5 (s), 79.7 (s), 67.7 (s), 47.3 (s), 31.2 (s), 21.8 (s), 21.7 (s), 19.2 (s), 13.9 (s).

**HRMS (ESI):** \( \text{C}_{26}\text{H}_{28}\text{Cl}_{2}\text{N}_{2}\text{O}_{5}\text{S}_{2} \left[ (\text{M}+\text{Na})^+ \right] \): calcd.: 605.0709; found: 605.0710.

**IR (ATR/cm\(^{-1}\))** 2921, 1596, 1478, 1356, 1165, 1058, 912, 809.

6,7-Dichloro-2-(phenylthio)-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3ar)

![Chemical Structure](image)

Product 3ar was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2r (0.3 mmol, 40.8 mg) by following the general procedure.

A white solid, 39.6 mg, 64% yield.

**m.p.:** 165 – 167 °C.

**TLC:** \( R_f = 0.59 \) (dichloromethane/petroleum ether = 2:1) [UV].
**1H NMR** (600 MHz, CDCl₃) δ 7.82 (s, 1H), 7.69 – 7.64 (m, 3H), 7.42 (d, \(J = 7.5\) Hz, 2H), 7.39 (d, \(J = 8.1\) Hz, 2H), 7.37 – 7.35 (m, 1H), 7.35 – 7.31 (m, 2H), 7.29 (d, \(J = 8.1\) Hz, 2H), 7.19 (d, \(J = 8.0\) Hz, 2H), 5.81 (t, \(J = 3.6\) Hz, 1H), 4.23 (dd, \(J = 13.1, 3.1\) Hz, 1H), 3.49 (dd, \(J = 13.1, 4.3\) Hz, 1H), 2.42 (s, 3H), 2.41 (s, 3H).

**13C NMR** (151 MHz, CDCl₃) δ 145.2 (s), 145.1 (s), 135.2 (s), 135.0 (s), 134.8 (s), 130.4 (s), 130.3 (s), 130.2 (s), 130.1 (s), 129.4 (s), 129.3 (s), 127.6 (s), 127.5 (s), 127.4 (s), 127.1 (s), 124.2 (s), 120.5 (s), 61.6 (s), 48.7 (s), 21.8 (s), 21.8 (s).

**HRMS (ESI):** C₂₈H₂₄Cl₂N₂NaO₄S₃ [(M+Na)^+]: calcd.: 641.0167; found: 641.0171.

**IR (ATR/cm⁻¹):** 2920, 1590, 1480, 1360, 1170, 1060, 1030, 899.

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7,8-Dichloro-5,10-ditosyl-5,10,10a,11-tetrahydro-4bH-indeno[1,2-b]quinoxaline (3at)

Product 3at was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2t (0.15 mmol, 17.4 mg) by following the general procedure.

A white solid, 50.8 mg, 85% yield.

**m.p.:** 203 – 205 °C.

**TLC:** \(R_f = 0.60\) (dichloromethane/petroleum ether = 1:1) [UV].

**1H NMR** (400 MHz, CDCl₃) δ 7.70 (d, \(J = 8.3\) Hz, 2H), 7.62 (d, \(J = 8.3\) Hz, 2H), 7.45 (d, \(J = 7.5\) Hz, 2H), 7.35 (d, \(J = 8.1\) Hz, 2H), 7.32 (d, \(J = 8.2\) Hz, 2H), 7.19 – 7.08 (m, 3H), 7.04 (d, \(J = 7.3\) Hz, 1H), 5.94 (d, \(J = 8.6\) Hz, 1H), 5.36 – 5.26 (m, 1H), 3.13 (dd, \(J = 17.1, 7.5\) Hz, 1H), 2.95 (dd, \(J = 17.1, 3.7\) Hz, 1H), 2.46 (s, 3H), 2.44 (s, 3H).

**13C NMR** (101 MHz, CDCl₃) δ 144.7 (s), 139.6 (s), 138.0 (s), 136.3 (s), 136.1 (s), 132.7 (s), 131.1 (s), 130.4 (s), 130.2 (s), 129.3 (s), 129.2 (s), 127.9 (s), 127.5 (s), 127.5 (s), 125.4 (s), 125.1 (s), 125.0 (s), 65.9 (s), 62.2 (s), 38.4 (s), 21.8 (s), 21.8 (s). (the deficiency of carbon signals is probably because some of them coincide with identical chemical shift)

**HRMS (ESI):** C₂₉H₂₅Cl₂N₂O₄S₂ [(M+H)^+]: calcd.: 599.0627; found: 599.0627.

**IR (ATR/cm⁻¹):** 2918, 1596, 1477, 1354, 1160, 1088, 1008, 945, 817.
6,7-Dichloro-4,9-ditosyl-2,3,3a,4,9,9a-hexahydrofuro[2,3-b]quinoxaline (3au)

![Structure of 3au](image)

Product 3au was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2u (0.15 mmol, 10.5 mg) by following the general procedure.

A pink solid, 31.5 mg, 57% yield.

**m.p.:** 194 – 196 °C.

**TLC:** $R_t = 0.51$ (dichloromethane/petroleum ether = 2:1) [UV].

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.79 (d, $J = 8.4$ Hz, 2H), 7.70 (s, 1H), 7.59 (d, $J = 8.3$ Hz, 2H), 7.33 (d, $J = 2.7$ Hz, 2H), 7.31 (d, $J = 2.9$ Hz, 2H), 7.09 (s, 1H), 5.96 (d, $J = 7.8$ Hz, 1H), 5.32 – 5.23 (m, 1H), 3.65 (t, $J = 7.0$ Hz, 2H), 2.44 (s, 3H), 2.43 (s, 3H), 2.36 – 2.24 (m, 1H), 1.88 – 1.76 (m, 1H).

$^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 145.1 (s), 144.7 (s), 137.7 (s), 135.5 (s), 132.8 (s), 131.4 (s), 130.6 (s), 130.2 (s), 129.5 (s), 129.2 (s), 129.1 (s), 127.5 (s), 127.1 (s), 123.1 (s), 87.9 (s), 66.4 (s), 61.1 (s), 30.9 (s), 21.8 (s), 21.7 (s).

**HRMS (ESI):** C$_{24}$H$_{23}$Cl$_2$N$_2$O$_5$S$_2$ [(M+H)$^+$]: calcd.: 553.0420; found: 553.0421.

**IR** (ATR/cm$^{-1}$) 2919, 1597, 1483, 1355, 1162, 1087, 811.

8,9-Dichloro-6,11-ditosyl-5a,6,11,11a-tetrahydrobenzofuro[2,3-b]quinoxaline (3av)

![Structure of 3av](image)

Product 3av was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2v (0.3 mmol, 35.4 mg) by following the general procedure.

A yellow solid, 30.0 mg, 50% yield.

**m.p.:** 213 – 215 °C.

**TLC:** $R_t = 0.62$ (dichloromethane/petroleum ether = 2:1) [UV].
$^1$H NMR (400 MHz, CDCl$_3$) δ 7.84 (d, $J = 8.4$ Hz, 2H), 7.66 (d, $J = 8.3$ Hz, 2H), 7.56 (s, 1H), 7.40 – 7.34 (m, 4H), 7.30 (d, $J = 7.5$ Hz, 1H), 7.13 (t, $J = 7.8$ Hz, 1H), 7.02 (t, 1H), 6.91 (t, $J = 7.5$ Hz, 1H), 6.69 (d, $J = 9.0$ Hz, 1H), 6.62 (d, $J = 8.1$ Hz, 1H), 6.37 (d, $J = 9.0$ Hz, 1H), 2.46 (s, 6H).

$^{13}$C NMR (101 MHz, CDCl$_3$) δ 157.8 (s), 145.3 (s), 145.0 (s), 137.5 (s), 135.5 (s), 132.0 (s), 131.4 (s), 130.9 (s), 130.7 (s), 130.3 (s), 129.9 (s), 129.7 (s), 127.7 (s), 127.3 (s), 125.7 (s), 123.5 (s), 122.5 (s), 122.2 (s), 110.1 (s), 90.5 (s), 63.2 (s), 21.8 (s), 21.8 (s). (the deficiency of carbon signals is probably because some of them coincide with identical chemical shift)

HRMS (ESI): C$_{26}$H$_{23}$Cl$_2$N$_2$O$_5$S$_2$ [(M+H)$^+$]: calcd.: 601.0420; found: 601.0426.

IR (ATR/cm$^{-1}$) 2920, 1598, 1477, 1362, 1163, 1072, 921, 811.

6-Benzyl-2,3-dichloro-5,11-ditosyl-5a,6,10b,11-tetrahydro-5H-indolo[2,3-b]quinoxaline (3aw)

![3aw]

Product 3aw was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2w (0.3 mmol, 62.1 mg) by following the general procedure.

A white solid, 62.0 mg, 90% yield.

m.p.: 78 – 80 °C.

TLC: $R_f = 0.39$ (dichloromethane/petroleum ether = 1:1) [UV].

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.75 (d, $J = 8.3$ Hz, 2H), 7.56 (d, $J = 8.3$ Hz, 2H), 7.39 (s, 1H), 7.35 (d, $J = 8.2$ Hz, 2H), 7.29 (s, 1H), 7.27 – 7.23 (m, 5H), 7.13 – 7.07 (m, 2H), 6.98 – 6.91 (m, 1H), 6.45 (d, $J = 4.3$ Hz, 2H), 6.26 – 6.20 (m, 2H), 6.08 (d, $J = 9.3$ Hz, 1H), 4.52 (d, $J = 16.6$ Hz, 1H), 4.35 (d, $J = 16.6$ Hz, 1H), 2.45 (s, 3H), 2.41 (s, 3H).

$^{13}$C NMR (101 MHz, CDCl$_3$) δ 149.5 (s), 144.8 (s), 144.7 (s), 137.8 (s), 137.1 (s), 135.7 (s), 133.5 (s), 131.9 (s), 131.0 (s), 130.4 (s), 130.2 (s), 130.0 (s), 128.7 (s), 128.6 (s), 127.7 (s), 127.6 (s), 127.3 (s), 127.1 (s), 126.1 (s), 124.8 (s), 123.4 (s), 118.6 (s), 106.7 (s), 80.6 (s), 62.9 (s), 47.8 (s), 21.8 (s), 21.8 (s).

HRMS (ESI): C$_{35}$H$_{30}$Cl$_2$N$_2$O$_4$S$_2$ [(M+H)$^+$]: calcd.: 690.1049; found: 690.1048.

IR (ATR/cm$^{-1}$) 2920, 1596, 1474, 1352, 1159, 1087, 934, 813.
2,3-Dichloro-6-methyl-5,11-ditosyl-5a,6,10b,11-tetrahydro-5H-indolo[2,3-b]quinoxaline (3ax)

![Structure of 3ax](image)

Product 3ax was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2x (0.3 mmol, 39.3 mg) by following the general procedure. A white solid, 50.3 mg, 82% yield.

**m.p.:** 168 – 170 °C.

**TLC:** \( R_f = 0.46 \) (dichloromethane/petroleum ether = 1:1) [UV].

**\( ^1H \) NMR** (400 MHz, CDCl3) \( \delta 7.76 \text{ (d, } J = 8.3 \text{ Hz, } 2\text{H}), 7.66 – 7.60 \text{ (m, } 3\text{H}), 7.36 \text{ (d, } J = 8.1 \text{ Hz, } 2\text{H}), 7.30 \text{ (d, } J = 8.1 \text{ Hz, } 2\text{H}), 7.22 \text{ (s, } 1\text{H}), 7.00 \text{ (t, } J = 7.6 \text{ Hz, } 1\text{H}), 6.43 \text{ (t, } J = 7.4 \text{ Hz, } 1\text{H}), 6.34 \text{ (d, } J = 7.4 \text{ Hz, } 1\text{H}), 6.29 \text{ (d, } J = 7.9 \text{ Hz, } 1\text{H}), 6.09 \text{ (d, } J = 8.9 \text{ Hz, } 1\text{H}), 5.99 \text{ (d, } J = 8.9 \text{ Hz, } 1\text{H}), 2.84 \text{ (s, } 3\text{H}), 2.47 \text{ (s, } 3\text{H}), 2.42 \text{ (s, } 3\text{H}).

**\( ^{13}C \) NMR** (101 MHz, CDCl3) \( \delta 150.1 \text{ (s)}, 144.8 \text{ (s)}, 144.8 \text{ (s)}, 137.1 \text{ (s)}, 135.4 \text{ (s)}, 133.1 \text{ (s)}, 132.0 \text{ (s)}, 130.7 \text{ (s)}, 130.4 \text{ (s)}, 130.3 \text{ (s)}, 130.2 \text{ (s)}, 130.0 \text{ (s)}, 128.0 \text{ (s)}, 127.8 \text{ (s)}, 127.5 \text{ (s)}, 126.0 \text{ (s)}, 124.4 \text{ (s)}, 123.4 \text{ (s)}, 118.5 \text{ (s)}, 106.5 \text{ (s)}, 81.8 \text{ (s)}, 62.6 \text{ (s)}, 30.7 \text{ (s)}, 21.8 \text{ (s)}, 21.8 \text{ (s}).

**HRMS (ESI):** \( \text{C}_{29}\text{H}_{26}\text{Cl}_{2}\text{N}_{3}\text{O}_{4}\text{S}_{2} \left[(\text{M+H})^+\right]: \text{calcd.}: 614.0736; \text{found}: 614.0738.

**IR (ATR/cm\(^{-1}\)):** 2920, 1740, 1600, 1470, 1360, 1160, 1090, 935, 810.

2,3-Dichloro-6,8-dimethyl-5,11-ditosyl-5a,6,10b,11-tetrahydro-5H-indolo[2,3-b]quinoxaline (3ay)

![Structure of 3ay](image)

Product 3ay was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2y (0.3 mmol, 43.5 mg) by following the general procedure. A brown solid, 54.6 mg, 87% yield.

**m.p.:** 165 – 167 °C.
TLC: $R_f = 0.68$ (dichloromethane/petroleum ether = 2:1) [UV].

$^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.75 (d, $J = 8.3$ Hz, 2H), 7.65 (s, 1H), 7.63 (d, $J = 8.3$ Hz, 2H), 7.35 (d, $J = 8.2$ Hz, 2H), 7.30 (d, $J = 8.2$ Hz, 2H), 7.24 (s, 1H), 6.27 – 6.21 (m, 2H), 6.12 (s, 1H), 6.08 (d, $J = 8.9$ Hz, 1H), 5.96 (d, $J = 8.9$ Hz, 1H), 2.82 (s, 3H), 2.46 (s, 3H), 2.42 (s, 3H), 2.16 (s, 3H).

$^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 150.2 (s), 144.7 (s), 140.6 (s), 137.1 (s), 135.5 (s), 133.2 (s), 132.0 (s), 130.6 (s), 130.4 (s), 130.2 (s), 129.8 (s), 128.0 (s), 127.7 (s), 127.5 (s), 125.9 (s), 124.1 (s), 120.5 (s), 119.3 (s), 107.5 (s), 82.1 (s), 62.5 (s), 30.6 (s), 21.8 (s), 21.8 (s), 21.8 (s).

HRMS (ESI): C$_{30}$H$_{28}$Cl$_2$N$_3$O$_4$S$_2$ [(M+H)$^+$]: calcd.: 628.0893; found: 628.0895.

IR (ATR/cm$^{-1}$) 2920, 1598, 1478, 1354, 1163, 1088, 930, 811.

2,3-Dichloro-9-fluoro-6-methyl-5,11-ditosyl-5a,6,10b,11-tetrahydro-5H-indolo[2,3-b]quinoxaline (3az)

![Structure of 3az](image.png)

Product 3az was obtained from the reaction mixture with substrate 1a (0.1 mmol, 48.4 mg) and 2z (0.3 mmol, 44.7 mg) by following the general procedure.

A yellow solid, 57.4 mg, 91% yield.

m.p.: 125 – 127 °C.

TLC: $R_f = 0.68$ (dichloromethane/petroleum ether = 2:1) [UV].

$^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.74 (d, $J = 8.3$ Hz, 2H), 7.64 (s, 1H), 7.62 (d, $J = 8.3$ Hz, 2H), 7.37 (d, $J = 8.2$ Hz, 2H), 7.30 (d, $J = 8.2$ Hz, 2H), 7.27 (s, 1H), 6.70 (td, $J = 8.9, 2.4$ Hz, 1H), 6.19 (dd, $J = 8.6, 3.9$ Hz, 1H), 6.11 – 6.04 (m, 2H), 5.94 (d, $J = 8.9$ Hz, 1H), 2.79 (s, 3H), 2.47 (s, 3H), 2.42 (s, 3H).

$^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 157.3 (s), 155.7 (s), 146.3 (s), 145.0 (d, $J = 33.4$ Hz), 136.8 (s), 135.3 (s), 132.9 (s), 131.9 (s), 130.8 (s), 130.5 (s), 130.2 (s), 130.1 (s), 128.0 (s), 127.7 (s), 127.4 (s), 126.0 (s), 124.8 (d, $J = 7.8$ Hz), 116.6 (d, $J = 23.4$ Hz), 112.0 (d, $J = 25.0$ Hz), 106.9 (d, $J = 7.9$ Hz), 82.2 (s), 62.2 (s), 31.2 (s), 21.8 (s), 21.8 (s).
2-Phenyl-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3ba)

![Structure of 3ba]

Product 3ba was obtained from the reaction mixture with substrate 1b (0.1 mmol, 41.6 mg) and 2a (0.15 mmol, 15.6 mg) by following the general procedure.

A white solid, 18.6 mg, 36% yield.

**m.p.:** 130 – 132°C.

**TLC:** \( R_f = 0.63 \) (dichloromethane/petroleum ether = 2:1) [UV].

**\(^1\)H NMR** (400 MHz, CDCl₃) \( \delta \) 7.87 (dd, \( J = 8.1, 1.6 \) Hz, 1H), 7.42 – 7.32 (m, 5H), 7.32 – 7.26 (m, 5H), 7.20 (d, \( J = 8.1 \) Hz, 2H), 7.14 (d, \( J = 8.1 \) Hz, 2H), 7.09 (td, \( J = 7.8, 1.6 \) Hz, 1H), 7.06 – 7.01 (m, 1H), 5.62 (dd, \( J = 6.7, 5.5 \) Hz, 1H), 3.96 (dd, \( J = 13.3, 6.8 \) Hz, 1H), 3.74 (dd, \( J = 13.4, 5.4 \) Hz, 1H), 2.40 (s, 3H), 2.36 (s, 3H).

**\(^{13}\)C NMR** (101 MHz, CDCl₃) \( \delta \) 144.4 (s), 144.2 (s), 138.3 (s), 136.4 (s), 135.4 (s), 132.2 (s), 130.0 (s), 129.9 (s), 129.0 (s), 128.6 (s), 128.1 (s), 127.5 (s), 127.2 (s), 126.7 (s), 126.1 (s), 125.8 (s), 124.6 (s), 120.4 (s), 59.8 (s), 50.4 (s), 21.8 (s), 21.7 (s).

**HRMS (ESI):** C₂₈H₂₇N₂O₄S₂ [(M+H)+]: calcd.: 519.1407; found: 519.1410.

**IR** (ATR/cm⁻¹) 2920, 1596, 1464, 1354, 1165, 1063.

6,7-Dibromo-2-phenyl-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3ca)

![Structure of 3ca]

Product 3ca was obtained from the reaction mixture with substrate 1c (0.1 mmol, 57.2 mg) and 2a
(0.15 mmol, 15.6 mg) by following the general procedure.

A white solid, 57.9 mg, 86% yield.

**m.p.:** 197 – 199 °C.

**TLC:** $R_f = 0.70$ (dichloromethane/petroleum ether = 2:1) [UV].

**$^1$H NMR** (400 MHz, CDCl$_3$) $\delta$ 8.16 (s, 1H), 7.77 (s, 1H), 7.46 (d, $J = 8.3$ Hz, 2H), 7.32 – 7.27 (m, 3H), 7.26 – 7.20 (m, 4H), 7.18 (d, $J = 8.4$ Hz, 2H), 7.10 (d, $J = 8.2$ Hz, 2H), 5.61 (t, $J = 4.6$ Hz, 1H), 4.32 (dd, $J = 13.4$, 4.9 Hz, 1H), 3.37 (dd, $J = 13.4$, 4.5 Hz, 1H), 2.43 (s, 3H), 2.36 (s, 3H).

**$^{13}$C NMR** (101 MHz, CDCl$_3$) $\delta$ 145.0 (s), 144.8 (s), 137.1 (s), 135.0 (s), 134.9 (s), 130.6 (s), 130.2 (s), 130.0 (s), 129.4 (s), 129.1 (s), 128.2 (s), 127.6 (s), 127.5 (s), 127.2 (s), 126.6 (s), 124.9 (s), 121.0 (s), 119.0 (s), 57.4 (s), 48.3 (s), 21.8 (s), 21.7 (s).

**HRMS (ESI):** C$_{28}$H$_{24}$Br$_2$N$_2$NaO$_4$S$_2$ [(M+Na)$^+$]: calcd.: 696.9436; found: 696.9438.

**HRMS (ESI):** C$_{28}$H$_{24}$Br$_2$N$_2$NaO$_4$S$_2$ [(M+Na)$^+$]: calcd.: 700.9396; found: 700.9398.

**IR** (ATR/cm$^{-1}$) 2919, 1463, 1354, 1165, 1059, 899.

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6,7-Dimethyl-2-phenyl-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3da)

![3da](image)

Product 3da was obtained from the reaction mixture with substrate 1d (0.1 mmol, 44.4 mg) and 2a (0.15 mmol, 15.6 mg) by following the general procedure.

A white solid, 29.5 mg, 54% yield.

**m.p.:** 144 – 146 °C.

**TLC:** $R_f = 0.53$ (dichloromethane/petroleum ether = 2:1) [UV].

**$^1$H NMR** (400 MHz, CDCl$_3$) $\delta$ 7.61 (s, 1H), 7.41 (d, $J = 8.3$ Hz, 2H), 7.35 (d, $J = 8.3$ Hz, 2H), 7.30 – 7.25 (m, 5H), 7.19 (d, $J = 8.0$ Hz, 2H), 7.17 (s, 1H), 7.14 (d, $J = 8.1$ Hz, 2H), 5.54 (dd, $J = 6.6$, 5.5 Hz, 1H), 3.91 (dd, $J = 13.3$, 6.8 Hz, 1H), 3.66 (dd, $J = 13.4$, 5.4 Hz, 1H), 2.40 (s, 3H), 2.36 (s, 3H), 2.21 (s, 3H), 2.13 (s, 3H).
$^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 144.2 (s), 144.0 (s), 138.4 (s), 136.5 (s), 135.6 (s), 134.4 (s), 133.2 (s), 129.9 (s), 129.8 (s), 129.6 (s), 128.9 (s), 127.9 (s), 127.6 (s), 127.2 (s), 126.8 (s), 126.7 (s), 126.1 (s), 121.4 (s), 59.5 (s), 50.3 (s), 21.7 (s), 21.7 (s), 19.8 (s), 19.6 (s).

HRMS (ESI): C$_{30}$H$_{30}$N$_2$NaO$_4$S$_2$ [(M+Na)$^+$]: calcd.: 569.1539; found: 569.1542.

IR (ATR/cm$^{-1}$) 2919, 1352, 1333, 1159, 1087, 997, 809.

6-Methyl-5,11-ditosyl-5a,6,10b,11-tetrahydro-5H-indolo[2,3-b]quinoxaline (3bx)

Product 3bx was obtained from the reaction mixture with substrate 1b (0.1 mmol, 41.6 mg) and 2x (0.15 mmol, 19.7 mg) by following the general procedure.

A yellow solid, 27.8 mg, 51% yield.

m.p.: 148 – 150 °C.

TLC: $R_f$ = 0.70 (dichloromethane/petroleum ether = 2:1) [UV].

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.80 (d, $J$ = 8.3 Hz, 2H), 7.59 (d, $J$ = 8.3 Hz, 2H), 7.57 – 7.52 (m, 1H), 7.35 (d, $J$ = 8.1 Hz, 2H), 7.26 (d, $J$ = 8.1 Hz, 2H), 7.15 – 7.09 (m, 1H), 7.05 – 6.98 (m, 2H), 6.93 (t, $J$ = 7.6 Hz, 1H), 6.33 (t, $J$ = 7.4 Hz, 1H), 6.25 (d, $J$ = 7.9 Hz, 1H), 6.20 (d, $J$ = 7.4 Hz, 1H), 6.16 (d, $J$ = 8.8 Hz, 1H), 6.00 (d, $J$ = 8.8 Hz, 1H), 2.88 (s, 3H), 2.46 (s, 3H), 2.39 (s, 3H).

$^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 150.3 (s), 144.3 (s), 144.3 (s), 138.1 (s), 135.8 (s), 134.0 (s), 132.5 (s), 130.3 (s), 130.0 (s), 129.9 (s), 127.7 (s), 127.5 (s), 127.3 (s), 127.1 (s), 126.2 (s), 124.5 (s), 124.2 (s), 123.9 (s), 118.0 (s), 106.1 (s), 82.3 (s), 62.7 (s), 30.6 (s), 21.8 (s), 21.8 (s).

HRMS (ESI): C$_{29}$H$_{28}$N$_3$O$_4$S$_2$ [(M+H)$^+$]: calcd.: 546.1516; found: 546.1516.

IR (ATR/cm$^{-1}$) 2918, 1596, 1490, 1349, 1155, 1089, 920.

2,3,6-Trimethyl-5,11-ditosyl-5a,6,10b,11-tetrahydro-5H-indolo[2,3-b]quinoxaline (3dx)

Product 3dx was obtained from the reaction mixture with substrate 1d (0.1 mmol, 44.4 mg) and 2x
(0.15 mmol, 19.7 mg) by following the general procedure.

A white solid, 43.6 mg, 76% yield.

**m.p.**: 185 – 186 °C.

**TLC**: \( R_f = 0.73 \) (dichloromethane/petroleum ether = 2:1) [UV].

**\(^1\text{H NMR}\)** (400 MHz, CDCl\(_3\)) \( \delta \): 7.79 (d, \( J = 8.3 \) Hz, 2H), 7.63 (d, \( J = 8.3 \) Hz, 2H), 7.34 (d, \( J = 8.0 \) Hz, 2H), 7.29 (d, \( J = 9.5 \) Hz, 2H), 7.26 (s, 1H), 6.99 – 6.91 (m, 1H), 6.87 (s, 1H), 6.36 (dt, \( J = 7.3 \), 3.7 Hz, 1H), 6.32 (d, \( J = 7.3 \) Hz, 1H), 6.24 (d, \( J = 7.9 \) Hz, 1H), 6.10 (d, \( J = 9.0 \) Hz, 1H), 5.96 (d, \( J = 9.0 \) Hz, 1H), 2.85 (s, 3H), 2.46 (s, 3H), 2.40 (s, 3H), 2.11 (s, 3H), 2.07 (s, 3H).

**\(^{13}\text{C NMR}\)** (101 MHz, CDCl\(_3\)) \( \delta \): 150.3 (s), 144.1 (s), 138.2 (s), 136.2 (s), 135.7 (s), 135.0 (s), 131.1 (s), 130.1 (s), 129.9 (s), 129.7 (s), 127.9 (s), 127.8 (s), 127.4 (s), 125.6 (s), 124.4 (s), 124.2 (s), 117.8 (s), 105.9 (s), 81.6 (s), 62.5 (s), 30.5 (s), 21.8 (s), 21.8 (s), 19.9 (s), 19.7 (s). (the deficiency of carbon signals is probably because some of them coincide with identical chemical shift)

**HRMS (ESI)**: \( \text{C}_{31}\text{H}_{31}\text{N}_3\text{NaO}_4\text{S}_2 \) \([\text{M+Na}^+]\): calcd.: 596.1648; found: 596.1654.

**IR** (ATR/cm\(^{-1}\)) 3375, 2921, 1509, 1331, 1159, 1089, 917, 812.

**2,3-Difluoro-6-methyl-5,11-ditosyl-5a,6,10b,11-tetrahydro-5H-indolo[2,3-b]quinoxaline (3ex)**

![Chemical structure of 3ex](attachment:image.png)

Product 3ex was obtained from the reaction mixture with substrate 1e (0.1 mmol, 45.2 mg) and 2x (0.15 mmol, 19.7 mg) by following the general procedure.

A white solid, 43.6 mg, 75% yield.

**m.p.**: 167 – 169 °C.

**TLC**: \( R_f = 0.74 \) (dichloromethane/petroleum ether = 2:1) [UV].

**\(^1\text{H NMR}\)** (400 MHz, CDCl\(_3\)) \( \delta \): 7.77 (d, \( J = 8.3 \) Hz, 2H), 7.62 (d, \( J = 8.3 \) Hz, 2H), 7.43 (dd, \( J = 10.8 \), 8.0 Hz, 1H), 7.37 (d, \( J = 8.1 \) Hz, 2H), 7.29 (d, \( J = 8.1 \) Hz, 2H), 7.02 – 6.93 (m, 2H), 6.40 (t, \( J = 7.4 \) Hz, 1H), 6.28 (t, \( J = 8.1 \) Hz, 2H), 6.12 (d, \( J = 8.9 \) Hz, 1H), 6.00 (d, \( J = 8.9 \) Hz, 1H), 2.86 (s, 3H), 2.47 (s, 3H), 2.42 (s, 3H).
$^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 150.1 (s), 148.5 (dd, $J = 249.7$, 13.2 Hz), 147.9 (dd, $J = 248.7$, 13.0 Hz), 144.7 (d, $J = 4.6$ Hz), 137.4 (s), 135.4 (s), 130.4 (s), 130.3 (m), 130.2 (s), 130.2 (s), 129.0 (dd, $J = 8.6$, 3.5 Hz), 127.7 (s), 127.4 (s), 124.2 (s), 123.4 (s), 118.3 (s), 116.1 (d, $J = 20.9$ Hz), 113.9 (d, $J = 20.6$ Hz), 106.4 (s), 82.1 (s), 62.8 (s), 30.6 (s), 21.8 (s), 21.8 (s). (the deficiency of carbon signals is probably because some of them coincide with identical chemical shift)

**HRMS (ESI):** C$_{29}$H$_{26}$F$_2$N$_3$O$_4$S$_2$ [(M+H)$^+$]: calcd.: 582.1327; found: 582.1333.

**IR (ATR/cm$^{-1}$)** 2919, 1611, 1489, 1360, 1163, 1086, 930.

2-Butoxy-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3bq)

![Chemical Structure](image)

**Product 3bq** was obtained from the reaction mixture with substrate 1b (0.1 mmol, 41.6 mg) and 2q (0.15 mmol, 15.0 mg) by following the general procedure.

A white solid, 26.7 mg, 52% yield.

**m.p.:** 91 – 93 $^\circ$C.

**TLC:** $R_f = 0.79$ (dichloromethane/petroleum ether = 2:1) [UV].

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.82 – 7.74 (m, 1H), 7.67 (d, $J = 8.3$ Hz, 2H), 7.61 – 7.55 (m, 1H), 7.42 (d, $J = 8.3$ Hz, 2H), 7.20 (d, $J = 8.1$ Hz, 2H), 7.17 (d, $J = 8.1$ Hz, 2H), 7.07 – 6.97 (m, 2H), 5.61 (t, $J = 3.4$ Hz, 1H), 4.19 (dd, $J = 13.4$, 3.3 Hz, 1H), 3.67 (dt, $J = 9.6$, 6.8 Hz, 1H), 3.55 (dt, $J = 9.6$, 6.3 Hz, 1H), 3.21 (dd, $J = 13.4$, 3.6 Hz, 1H), 2.39 (s, 3H), 2.35 (s, 3H), 1.44 – 1.34 (m, 2H), 1.20 – 1.09 (m, 2H), 0.82 (t, $J = 7.4$ Hz, 3H).

$^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 144.7 (s), 144.1 (s), 136.2 (s), 135.3 (s), 130.5 (s), 130.1 (s), 129.5 (s), 127.9 (s), 127.2 (s), 126.0 (s), 126.0 (s), 125.1 (s), 123.9 (s), 119.6 (s), 81.4 (s), 67.6 (s), 48.4 (s), 31.3 (s), 21.7 (s), 21.7 (s), 19.2 (s), 13.9 (s).

**HRMS (ESI):** C$_{26}$H$_{30}$N$_2$NaO$_5$S$_2$ [(M+Na)$^+$]: calcd.: 537.1488; found: 537.1489.

**IR (ATR/cm$^{-1}$)** 2919, 1486, 1349, 1163, 1045, 999, 814.
6,7-Dibromo-2-butoxy-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3cq)

Product 3cq was obtained from the reaction mixture with substrate 1c (0.1 mmol, 57.2 mg) and 2q (0.15 mmol, 15.0 mg) by following the general procedure.

A white solid, 62.3 mg, 93% yield.

**m.p.:** 144 – 146 °C.

**TLC:** \( R_f = 0.54 \) (dichloromethane/petroleum ether = 1:1) [UV].

**\(^1\)H NMR** (400 MHz, CDCl\(_3\)) \( \delta \) 8.09 (s, 1H), 8.01 (s, 1H), 7.68 (d, \( J = 8.4 \) Hz, 2H), 7.44 (d, \( J = 8.3 \) Hz, 2H), 7.26 (d, \( J = 8.1 \) Hz, 2H), 7.19 (d, \( J = 8.1 \) Hz, 2H), 5.52 (t, \( J = 2.5 \) Hz, 1H), 4.43 (dd, \( J = 13.7 \), 2.6 Hz, 1H), 3.63 – 3.56 (m, 1H), 3.55 – 3.48 (m, 1H), 2.83 (dd, \( J = 13.7 \), 2.6 Hz, 1H), 2.42 (s, 3H), 2.38 (s, 3H), 1.41 – 1.32 (m, 2H), 1.18 – 1.08 (m, 2H), 0.84 (t, \( J = 7.4 \) Hz, 3H).

**\(^{13}\)C NMR** (101 MHz, CDCl\(_3\)) \( \delta \) 145.3 (s), 144.8 (s), 134.9 (s), 134.6 (s), 130.4 (s), 129.6 (s), 129.4 (s), 129.2 (s), 128.4 (s), 127.2 (s), 124.1 (s), 123.5 (s), 121.2 (s), 118.4 (s), 79.6 (s), 67.7 (s), 47.2 (s), 31.2 (s), 21.8 (s), 21.7 (s), 19.2 (s), 13.9 (s).

**HRMS (ESI):** C\(_{26}\)H\(_{28}\)\(_79^{79}\)Br\(_2\)N\(_2\)NaO\(_5\)S\(_2\) [(M+Na){sup+}]: calcd.: 692.9699; found: 692.9699.

**HRMS (ESI):** C\(_{26}\)H\(_{28}\)\(_{81}\)Br\(_2\)N\(_2\)NaO\(_5\)S\(_2\) [(M+Na){sup+}]: calcd.: 696.9658; found: 696.9658.

**IR** (ATR/cm\(^{-1}\)) 2921, 1470, 1354, 1164, 1088, 1049, 906, 899.

2-Butoxy-6,7-dimethyl-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3dq)

Product 3dq was obtained from the reaction mixture with substrate 1d (0.1 mmol, 44.4 mg) and 2q (0.15 mmol, 15.0 mg) by following the general procedure.

A white solid, 50.9 mg, 94% yield.

**m.p.:** 107 – 109 °C.

**TLC:** \( R_f = 0.76 \) (dichloromethane/petroleum ether = 2:1) [UV].
\[ ^1H\text{ NMR}\ (400\text{ MHz, CDCl}_3)\ \delta\ 7.66\ (d, \ J = 8.3\text{ Hz, 2H}),\ 7.53\ (s, 1H),\ 7.43\ (d, \ J = 8.3\text{ Hz, 2H}),\ 7.36\ (s, 1H),\ 7.21\ (d, \ J = 8.1\text{ Hz, 2H}),\ 7.17\ (d, \ J = 8.1\text{ Hz, 2H}),\ 5.55\ (t, \ J = 3.5\text{ Hz, 1H}),\ 4.10\ (dd, \ J = 13.5,\ 3.4\text{ Hz, 1H}),\ 3.65\ (dt, \ J = 9.6,\ 6.8\text{ Hz, 1H}),\ 3.52\ (dt, \ J = 9.6,\ 6.5\text{ Hz, 1H}),\ 3.17\ (dd, \ J = 13.5,\ 3.7\text{ Hz, 1H}),\ 2.39\ (s, 3H),\ 2.36\ (s, 3H),\ 2.17\ (s, 3H),\ 2.13\ (s, 3H),\ 1.43 - 1.34\ (m, 2H),\ 1.23 - 1.12\ (m, 2H),\ 0.83\ (t, \ J = 7.4\text{ Hz, 3H}).\]

\[ ^{13}C\text{ NMR}\ (101\text{ MHz, CDCl}_3)\ \delta\ 144.4\ (s),\ 143.9\ (s),\ 136.4\ (s),\ 135.4\ (s),\ 134.6\ (s),\ 132.4\ (s),\ 130.0\ (s),\ 129.5\ (s),\ 128.1\ (s),\ 127.9\ (s),\ 127.2\ (s),\ 126.8\ (s),\ 122.6\ (s),\ 120.6\ (s),\ 81.6\ (s),\ 67.5\ (s),\ 48.5\ (s),\ 31.3\ (s),\ 21.7\ (s),\ 19.8\ (s),\ 19.4\ (s),\ 19.2\ (s),\ 13.9\ (s).\]

HRMS (ESI): C\textsubscript{28}H\textsubscript{34}N\textsubscript{2}NaO\textsubscript{5}S\textsubscript{2} [(M+Na)+]: calcd.: 565.1801; found: 565.1803.

IR (ATR/cm\textsuperscript{-1}) 2921, 1600, 1450, 1351, 1160, 1090, 806.

6-Bromo-2-butoxy-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3fq) and 7-bromo-2-butoxy-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3fq*)

Product 3fq and 3fq* was obtained from the reaction mixture with substrate 1f (0.1 mmol, 49.4 mg) and 2q (0.15 mmol, 15.0 mg) by following the general procedure.

A mixture of two isomers. Colorless oil, 45.8 mg, 77% yield.

TLC: \( R_f = 0.70\) (dichloromethane/petroleum ether = 2:1) [UV].

\[ ^1H\text{ NMR for the mixture}\ (400\text{ MHz, CDCl}_3)\ \delta\ 7.98\ (d, \ J = 2.3\text{ Hz, 1H, major}),\ 7.85\ (d, \ J = 2.2\text{ Hz, 1H, minor}),\ 7.72 - 7.64\ (m, 2H, mixture),\ 7.55\ (d, \ J = 9.0\text{ Hz, 1H, major}),\ 7.45\ (d, \ J = 8.3\text{ Hz, 2H, major}),\ 7.41\ (d, \ J = 8.3\text{ Hz, 2H, minor}),\ 7.25 - 7.21\ (m, 2H, mixture),\ 7.21 - 7.15\ (m, 2H, mixture),\ 7.15 - 7.09\ (m, 1H, mixture),\ 5.61 - 5.51\ (m, 1H, mixture),\ 4.39 - 4.28\ (m, 1H, mixture),\ 3.67 - 3.58\ (m, 1H, mixture),\ 3.58 - 3.48\ (m, 1H, mixture),\ 3.03 - 2.91\ (m, 1H, mixture),\ 2.40\ (s, 3H, mixture),\ 2.36\ (s, 3H, mixture),\ 1.43 - 1.33\ (m, 2H, mixture),\ 1.20 - 1.07\ (m, 2H, mixture),\ 0.86 - 0.79\ (m, 3H, mixture).

\[ ^{13}C\text{ NMR for the mixture}\ (151\text{ MHz, CDCl}_3)\ \delta\ 145.0\ (s),\ 145.0\ (s),\ 144.5\ (s),\ 144.4\ (s),\ 135.5\ (s),\ 135.2\ (s),\ 134.8\ (s),\ 134.8\ (s),\ 130.7\ (s),\ 130.3\ (s),\ 130.2\ (s),\ 129.5\ (s),\ 128.8\ (s),\ 128.6\ (s),\ 128.2\]
(s), 128.1 (s), 128.1 (s), 127.2 (s), 127.1 (s), 127.0 (s), 126.6 (s), 125.6 (s), 123.3 (s), 122.1 (s), 120.7 (s), 119.0 (s), 116.2 (s), 80.3 (s), 80.2 (s), 67.7 (s), 67.6 (s), 47.7 (s), 47.5 (s), 31.2 (s), 31.2 (s), 21.8 (s), 21.7 (s), 21.7 (s), 19.2 (s), 13.9 (s). (the deficiency of carbon signals is probably because some of them coincide with identical chemical shift)

**HRMS (ESI)**: C_{26}H_{33}^{39}BrN_{3}O_{5}S_{2} [(M+NH_{4})^{+}]: calcd.: 610.1040; found: 610.1044.

**HRMS (ESI)**: C_{26}H_{33}^{31}BrN_{3}O_{5}S_{2} [(M+NH_{4})^{+}]: calcd.: 612.1019; found: 612.1023.

**IR** (ATR/cm\(^{-1}\)) 2918, 1481, 1353, 1162, 1090, 1043, 1001, 812.

---

2-Butoxy-5-methyl-1,4-ditosyl-1,2,3,4-tetrahydroquinoxaline (3gq)

![Chemical Structure](image)

Product 3gq was obtained from the reaction mixture with substrate 1g (0.1 mmol, 43.0 mg) and 2q (0.15 mmol, 15.0 mg) by following the general procedure.

A white solid, 42.2 mg, 80% yield.

**m.p.:** 146 – 148 °C.

**TLC:** \( R_t = 0.37 \) (dichloromethane/petroleum ether = 2:1) [UV].

**\(^1\)H NMR** (600 MHz, CDCl\(_3\)) \( \delta \) 7.78 (d, \( J = 8.4 \) Hz, 2H), 7.76 (d, \( J = 8.3 \) Hz, 2H), 7.44 (d, \( J = 7.6 \) Hz, 1H), 7.33 (d, \( J = 8.1 \) Hz, 2H), 7.19 (d, \( J = 8.2 \) Hz, 2H), 7.08 (t, \( J = 7.9 \) Hz, 1H), 6.98 (d, \( J = 7.5 \) Hz, 1H), 5.88 (t, \( J = 7.5 \) Hz, 1H), 4.43 (dd, \( J = 14.7, 7.6 \) Hz, 1H), 3.83 (dt, \( J = 9.5, 6.6 \) Hz, 1H), 3.57 (dt, \( J = 9.5, 6.6 \) Hz, 1H), 3.16 (dd, \( J = 14.7, 7.5 \) Hz, 1H), 2.44 (s, 3H), 2.34 (s, 3H), 2.23 (s, 3H), 1.53 – 1.47 (m, 2H), 1.34 – 1.29 (m, 2H), 0.88 (t, \( J = 7.4 \) Hz, 3H).

**\(^{13}\)C NMR** (151 MHz, CDCl\(_3\)) \( \delta \) 144.3 (s), 144.0 (s), 137.4 (s), 136.7 (s), 136.5 (s), 134.4 (s), 132.8 (s), 130.1 (s), 129.4 (s), 128.5 (s), 128.2 (s), 127.6 (s), 127.4 (s), 122.0 (s), 86.8 (s), 68.8 (s), 53.0 (s), 31.9 (s), 21.7 (s), 21.7 (s), 19.5 (s), 19.3 (s), 13.9 (s).

**HRMS (ESI):** C_{27}H_{32}N_{2}NaO_{5}S_{2} [(M+Na)^{+}]: calcd.: 551.1645; found: 551.1643.

**IR** (ATR/cm\(^{-1}\)) 2924, 1595, 1471, 1323, 1151, 978, 812.
6,7-Dichloro-1,4-bis(methylsulfonyl)-2-phenyl-1,2,3,4-tetrahydroquinoxaline (3ha)

![Image of 3ha]

Product 3ha was obtained from the reaction mixture with substrate 1h (0.1 mmol, 33.2 mg) and 2a (0.15 mmol, 15.6 mg) by following the general procedure.
A white solid, 35.2 mg, 81% yield.

**m.p.:** 186 – 188 °C.

**TLC:** \( R_f \) = 0.23 (dichloromethane/petroleum ether = 2:1) [UV].

**\(^1\)H NMR** (400 MHz, CDCl\(_3\)) \( \delta \): 8.01 (s, 1H), 7.86 (s, 1H), 7.40 – 7.24 (m, 5H), 5.74 (t, \( J = 5.2 \) Hz, 1H), 4.37 (dd, \( J = 14.3, 5.9 \) Hz, 1H), 3.90 (dd, \( J = 14.3, 4.6 \) Hz, 1H), 2.98 (s, 3H), 2.45 (s, 3H).

**\(^{13}\)C NMR** (101 MHz, CDCl\(_3\)) \( \delta \): 137.0 (s), 129.7 (s), 129.6 (s), 129.4 (s), 129.3 (s), 128.7 (s), 128.4 (s), 126.5 (s), 124.9 (s), 123.3 (s), 58.1 (s), 50.5 (s), 40.4 (s), 38.9 (s).

**HRMS (ESI):** \( \text{C}_{16}\text{H}_{17}\text{Cl}_{2}\text{N}_{2}\text{O}_{4}\text{S}_{2} \) [(M+H)+]: calcd.: 435.0001; found: 435.0004.

**IR** (ATR/cm\(^{-1}\)) 2930, 1472, 1334, 1152, 1069, 957, 883.

Di-tert-butyl 6,7-dichloro-2-phenyl-2,3-dihydroquinoxaline-1,4-dicarboxylate (3ia)

![Image of 3ia]

Product 3ia was obtained from the reaction mixture with substrate 1i (0.1 mmol, 37.6 mg) and 2a (0.3 mmol, 31.2 mg) by following the general procedure.
A white solid, 39.2 mg, 82% yield.

**m.p.:** 186 – 188 °C.

**TLC:** \( R_f \) = 0.48 (dichloromethane/petroleum ether = 2:1) [UV].

**\(^1\)H NMR** (400 MHz, CDCl\(_3\)) \( \delta \): 8.42 (s, 1H), 7.69 (s, 1H), 7.32 – 7.22 (m, 3H), 7.16 – 7.11 (m, 2H), 5.46 (t, \( J = 4.3 \) Hz, 1H), 4.25 – 3.95 (m, 1H), 3.80 – 3.55 (m, 1H), 1.26 (s, 18H).

**\(^{13}\)C NMR** (101 MHz, CDCl\(_3\)) \( \delta \): 152.6 (s), 152.1 (s), 141.0 (s), 131.2 (s), 128.8 (s), 127.7 (s), 127.6 (s), 125.7 (s), 125.3 (s), 123.6 (s), 82.5 (s), 81.9 (s), 60.4 (s), 48.5 (s), 28.0 (s), 28.0 (s). (the
deficiency of carbon signals is probably because some of them coincide with identical chemical shift)

**HRMS (ESI):** \( \text{C}_{24}\text{H}_{29}\text{Cl}_{2}\text{N}_{2}\text{O}_{4} \left([\text{M+H}]^+\right) \): calcd.: 479.1499; found: 479.1502.

**IR (ATR/cm\(^{-1}\))** 2977, 2925, 1715, 1482, 1388, 1333, 1149, 1056, 884.

Dibenzyl 6,7-dichloro-2-phenyl-2,3-dihydroquinoxaline-1,4-dicarboxylate (3ja)

![Diagram of 3ja]

Product 3ja was obtained from the reaction mixture with substrate 1j (0.1 mmol, 44.4 mg) and 2a (0.3 mmol, 31.2 mg) by following the general procedure.

A white solid, 35.5 mg, 65% yield.

**m.p.:** 155 – 157 °C.

**TLC:** \( R_f = 0.39 \) (dichloromethane/petroleum ether = 2:1) [UV].

**\(^1\)H NMR** (400 MHz, CDCl\(_3\)) \( \delta \) 8.38 (s, 1H), 7.76 (s, 1H), 7.35 – 7.30 (m, 3H), 7.27 – 7.17 (m, 8H), 7.13 – 7.08 (m, 2H), 7.05 – 6.98 (m, 2H), 5.57 (t, J = 4.9 Hz, 1H), 5.18 – 4.92 (m, 4H), 4.25 – 4.00 (m, 1H), 3.95 – 3.80 (m, 1H).

**\(^{13}\)C NMR** (101 MHz, CDCl\(_3\)) \( \delta \) 153.5 (s), 153.2 (s), 139.5 (s), 135.5 (s), 135.3 (s), 130.6 (s), 130.0 (s), 128.9 (s), 128.7 (s), 128.6 (s), 128.5 (s), 128.4 (s), 128.2 (s), 128.0 (s), 128.0 (s), 126.8 (s), 125.8 (s), 125.2 (s), 124.2 (s), 68.4 (s), 68.2 (s), 59.7 (s), 49.0 (s).

**HRMS (ESI):** \( \text{C}_{30}\text{H}_{25}\text{Cl}_{2}\text{N}_{2}\text{O}_{4} \left([\text{M+H}]^+\right) \): calcd.: 547.1186; found: 547.1188.

**IR (ATR/cm\(^{-1}\))** 2940, 1700, 1480, 1380, 1330, 1270, 1200, 1060, 880.

### 4. Synthetic Transformations of products

Under a N\(_2\) atmosphere, naphthalene (1.00 g, 7.8 mmol) was dissolved in 15 mL THF, then sliced Na (0.18 g, 7.8 mmol) was gradually added to the mixture, then stirred at room temperature for 3h.
Then 2 mL solution was transferred to compound 3aa (58.6 mg, 0.1 mmol) in 2 mL THF, and the mixture was stirred at room temperature for 36 h. After the reaction is completed, Sat. NaCl (aq.) was added to quench the reaction, and the aqueous phase was extracted with EtOAc. The combined organic layers were washed with sat. NaCl (aq.), dried over MgSO₄, filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1:1) to afford 6,7-dichloro-2-phenylquinoxaline (4aa) as a light yellow solid.

6,7-Dichloro-2-phenylquinoxaline (4aa)

A light yellow solid, 14.2 mg, 52% yield.

**TLC:** \( R_f = 0.70 \) (dichloromethane/petroleum ether = 2:1) [UV].

**\( ^1H \) NMR** (400 MHz, CDCl₃) \( \delta \) 9.31 (s, 1H), 8.27 (s, 1H), 8.23 (s, 1H), 8.20 – 8.15 (m, 2H), 7.62 – 7.52 (m, 3H).

The spectra data are matched with those reported.⁴

Compound 3ia (47.8 mg, 0.1 mmol) was dissolved in 2 mL DCM, then gradually added 0.5 mL CF₃COOH, the mixture was stirred at room temperature for 3h. After the reaction is completed, Sat. Na₂CO₃ (aq.) was added to the mixture to adjust \( \text{PH} \approx 7 \), the aqueous phase then extracted with ethyl acetate. The organic layer was dried over anhydrous Na₂SO₄, filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1:1) to afford 6,7-dichloro-2-phenyl-1,2,3,4-tetrahydroquinoxaline (5ia) as a light yellow solid.

6,7-Dichloro-2-phenyl-1,2,3,4-tetrahydroquinoxaline (5ia)
A light yellow solid, 26.4 mg, 95% yield.

**TLC:** $R_t = 0.50$ (dichloromethane/petroleum ether = 2:1) [UV].

**$^1$H NMR** (400 MHz, CDCl$_3$) $\delta$ 7.41 – 7.30 (m, 5H), 6.59 (d, $J = 3.8$ Hz, 2H), 4.43 (dd, $J = 7.9$, 3.1 Hz, 1H), 3.45 (dd, $J = 11.2$, 3.1 Hz, 1H), 3.27 (dd, $J = 11.1$, 8.0 Hz, 1H).

The spectra data are matched with those reported.

5. **X-Ray crystallographic data of 3gq.**

Table S1 Crystal data and structure refinement for 3gq.

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References

6. Copies of $^1$H and $^{13}$C NMR spectra

600 MHz, CDCl$_3$

101 MHz, CDCl$_3$
600 MHz, CDCl₃

151 MHz, CDCl₃
3ae

400 MHz, CDCl₃

3ae

151 MHz, CDCl₃
400 MHz, CDCl₃

151 MHz, CDCl₃
600 MHz, CDCl₃

151 MHz, CDCl₃
3ar

600 MHz, CDCl₃

151 MHz, CDCl₃
400 MHz, CDCl₃

101 MHz, CDCl₃
400 MHz, CDCl₃

101 MHz, CDCl₃
600 MHz, CDCl₃

151 MHz, CDCl₃
400 MHz, CDCl$_3$

101 MHz, CDCl$_3$
400 MHz, CDCl₃

101 MHz, CDCl₃
3bx

400 MHz, CDCl₃

3bx

101 MHz, CDCl₃
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400 MHz, CDCl₃

3dx

101 MHz, CDCl₃
400 MHz, CDCl₃

151 MHz, CDCl₃
400 MHz, CDCl₃

151 MHz, CDCl₃
600 MHz, CDCl₃

151 MHz, CDCl₃
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400 MHz, CDCl₃

3la

101 MHz, CDCl₃
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4aa

5ia

400 MHz, CDCl₃