Electronic Supplementary Information for

Acenaphthoimidazolium Chloride Enabled Nickel Catalyzed Amination of Bulky Aryl Tosylates

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1. General

All commercial reagents were used directly without further purification, unless otherwise stated. Dry dimethylsulfoxide (DMSO) was purchased from J & K chemical, stored over 4 Å molecular sieves and handled under N₂. Anhydrous methanol (MeOH) was distilled from anhydrous calcium chloride, Dioxane, Tetrahydrofuran (THF), toluene and m-Xylene were distilled from sodium/benzophenone, 1,2-Dimethoxyethane (DME) and 1,2-Dichloroethane (DCE) were distilled from calcium hydride prior to use. t-BuONa was purchased from J & K chemical. All schlenk tubes and sealed vessels (50 mL) were purchased from Beijing Synthware Glass. CDCl₃ was purchased from Cambridge Isotope Laboratories.¹H NMR and ¹³C NMR spectra were recorded on Jeol ECA-400 and Bruker 400 DRX spectrometers. ¹³C NMR spectra were referenced to the carbon signal of CDCl₃ (77.0 ppm). GC-MS spectra were recorded on Agilent Technologies 1890A GC system and 5975C inert MSD with Triple-Axis Detector.

2. Experimental sections

2.1 Synthesis of acenaphthoimidazolium chloride

\[
\begin{align*}
\text{S1} & \quad \text{MeCN}/\text{AcOH} \text{ reflux} \\
\text{NH}_2 & \quad \text{MeOCH}_2\text{Cl} \quad 100 \degree \text{C} \\
\text{S2} & \quad 1
\end{align*}
\]

**Scheme S1.** Synthesis of acenaphthoimidazolium chloride 1.

**Bis[N,N’-(2,6-diisopropylphenyl)imino]acenaphthene (S2):**

According to the literature procedure,⁵¹ acenaphthenequinone (3.5 g, 19.2 mmol) S1 was suspended in acetonitrile (125 mL) and heated under reflux for 1 hour. After that acetic acid (33 mL) was added, the solid was then completely dissolved. To the hot mixture,
2,6-diisopropylphenylaniline (8.16 g, 46 mmol) was added dropwise over 30 minutes and the obtained solution was continued refluxing for additional 5 hours. The resulting orange-yellow solid was then filtered after cooling to room temperature, washed with n-pentane (3 × 40 mL), and dried over under the vacuum. Yield: 8.5 g, 88 %. \(^1\)H NMR (CDCl\(_3\), 400 MHz, 298 K): \(\delta\) 7.87 (d, \(J = 8.0\) Hz, 2H), 7.36 (t, \(J = 7.6\) Hz, 2H), 7.27 (bs, 6H), 6.64 (d, \(J = 7.2\) Hz, 2H), 3.06-3.00 (m, 4H), 1.24 (d, \(J = 6.8\) Hz, 12H), 0.97 (d, \(J = 6.8\) Hz, 12H).

Acenaphthoimidazolium chloride (1):

According to the literature procedure,\(^{S1}\) S2 (3.00 g, 6 mmol) and methoxy(methyl)chloride (9.66 g, 120 mmol) were added to an nitrogen-flushed thick-walled sealed vessel. Then the reaction mixture was stirred at 100 °C for 16 hours, during the heating process, the mixture changed from a murky brown suspension to a clear red solution. After cooling to room temperature, a yellow precipitate was formed and 30 mL of diethyl ether was subsequently added. The precipitation was filtered and washed with 100 mL of diethyl ether and over under the vacuum to afford 1 as an analytically pure yellow powder. Yield: 2.3 g, 70%. \(^1\)H NMR (CDCl\(_3\), 400 MHz, 298 K): \(\delta\) 12.23 (s, 1H), 8.00 (d, \(J = 8.0\) Hz, 2H), 7.67 (t, \(J = 7.6\) Hz, 2H), 7.58 (t, \(J = 7.2\) Hz, 2H), 7.46 (d, \(J = 7.6\) Hz, 4H), 7.22 (d, \(J = 7.2\) Hz 2H), 2.78-2.68 (m, 4H), 1.40 (d, \(J = 6.8\) Hz, 12H), 1.16 (d, \(J = 6.8\) Hz, 12H).

2.2 General procedure for Ni-NHC catalyzed amination.

To a 50 mL schlenk tube containing base (1.5 mmol), NiCl\(_2\)(DME) (2 mol%) and acenaphthoimidazolium chloride 1 (4 mol%), aryl tosylate (0.5 mmol) and Ph-Bpin (1.0 mmol) were added and the tube was purged with N\(_2\) for 3 times. Then dioxane (2.5 mL) and amine (1.25 mmol) were injected via syringe, subsequently (Note, if amine was a solid, it was introduced to the tube before purging with N\(_2\)). The resulted mixture was allowed to stir for 1 hour at room temperature, then was heated at 100 °C for additional 3 hours. After cooling to the room temperature, the reaction mixture was concentrated under the vacuum and directly purified by flash chromatography.
3. Optimization of reaction conditions

Table S1. Base and solvent effects (excluded data in the Table 1)

<table>
<thead>
<tr>
<th>Entry</th>
<th>Solvent</th>
<th>Base (equiv)</th>
<th>Yield (%)&lt;sup&gt;b&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Dioxane</td>
<td>Et&lt;sub&gt;3&lt;/sub&gt;N (3.0)</td>
<td>NR</td>
</tr>
<tr>
<td>2</td>
<td>Dioxane</td>
<td>DBU (3.0)</td>
<td>NR</td>
</tr>
<tr>
<td>3</td>
<td>Dioxane</td>
<td>Na&lt;sub&gt;2&lt;/sub&gt;CO&lt;sub&gt;3&lt;/sub&gt; (3.0)</td>
<td>NR</td>
</tr>
<tr>
<td>4</td>
<td>Dioxane</td>
<td>Cs&lt;sub&gt;2&lt;/sub&gt;CO&lt;sub&gt;3&lt;/sub&gt; (3.0)</td>
<td>NR</td>
</tr>
<tr>
<td>5</td>
<td>Dioxane</td>
<td>CsF (3.0)</td>
<td>NR</td>
</tr>
<tr>
<td>6</td>
<td>MeOH</td>
<td>t-BuONa (3.0)</td>
<td>0</td>
</tr>
<tr>
<td>7</td>
<td>DMSO</td>
<td>t-BuONa (3.0)</td>
<td>0</td>
</tr>
<tr>
<td>8</td>
<td>DCE</td>
<td>t-BuONa (3.0)</td>
<td>NR</td>
</tr>
</tbody>
</table>

<sup>a</sup> 0.5 mmol scale for 1 hour at rt, then 3 hours at 100 °C, with 2 mol% NiCl<sub>2</sub>• (DME),<sup>b</sup> Isolated yield.

4. Data for the amination products

3<sup>S2</sup>

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 298 K): δ 7.76-7.69 (m, 3H), 7.41 (t, J = 7.6 Hz, 1H), 7.31 (t, J = 7.6 Hz, 1H), 7.27-7.24 (m, 1H), 7.12 (d, J = 2.0 Hz, 1H), 3.92 (t, J = 4.4 Hz, 4H), 3.27 (t, J = 4.4 Hz, 4H); GC-MS: m/z = 213.1 [M]<sup>+</sup>, 155.1, 127.1.

5a<sup>S2</sup>

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 298 K): δ 7.68 (t, J = 8.4 Hz, 2H), 7.62 (d, J = 8.4 Hz, 1H),
7.34 (t, $J = 7.2$ Hz, 1H), 7.15 (t, $J = 8.4$ Hz, 1H), 7.00 (dd, $J = 2.4$ Hz, $J = 8.8$ Hz, 1H), 6.75 (s, 1H), 3.41 (t, $J = 6.4$ Hz, 4H), 2.06 (t, $J = 6.4$ Hz, 4H); GC-MS: m/z = 196.1 [M-1]$^+$, 141.1, 127.1.

5b: $^2$H NMR (CDCl$_3$, 400 MHz, 298 K): δ 7.71-7.66 (m, 3H), 7.38 (t, $J = 7.2$ Hz, 1H), 7.30-7.23 (m, 2H), 7.13 (s, 1H), 3.26 (t, $J = 6.4$ Hz, 4H), 1.80-1.74 (m, 4H), 1.65-1.59 (m, 2H); GC-MS: m/z = 210.2 [M-1]$^+$, 135.9, 127.1.

5c: $^3$H NMR (CDCl$_3$, 400 MHz, 298 K): δ 7.66 (t, $J = 8.0$ Hz, 2H), 7.59 (d, $J = 8.4$ Hz, 1H), 7.32 (t, $J = 7.2$ Hz, 1H), 7.16-7.09 (m, 2H), 6.86 (d, $J = 2.0$ Hz, 1H), 3.58 (t, $J = 6.0$ Hz, 4H), 1.90-1.81 (m, 4H), 1.59-1.56 (m, 4H); GC-MS: m/z = 225.2 [M]$^+$, 196.1, 127.1.

6a: $^4$H NMR (CDCl$_3$, 400 MHz, 298 K): δ 7.75-7.70 (m, 3H), 7.42-7.38 (m, 1H), 7.31-7.26 (m, 2H), 7.13 (d, $J = 2.4$ Hz, 1H), 3.33 (t, $J = 5.0$ Hz, 4H), 2.65 (t, $J = 5.0$ Hz, 4H), 2.39 (s, 3H); $^{13}$C NMR (CDCl$_3$, 100 MHz, 298 K): δ = 148.98, 134.52, 128.60, 128.43, 127.33, 126.65, 126.17, 123.28, 119.27, 110.17, 55.00, 49.32, 46.02.

6b: $^5$H NMR (CDCl$_3$, 400 MHz, 298 K): δ 7.77-7.71 (m, 3H), 7.42 (t, $J = 7.6$ Hz, 1H), 7.34-7.30 (m, 4H), 7.19 (d, $J = 2.0$ Hz, 1H), 7.02 (d, $J = 8.0$ Hz, 2H), 6.91 (t, $J = 7.6$ Hz, 1H), 3.49-3.45 (m, 4H), 3.42-3.40 (m, 4H); GC-MS: m/z = 288.1 [M]$^+$, 155.1, 132.1.
**7a:**

$^1$H NMR (CDCl$_3$, 400 MHz, 298 K): δ 7.72-7.62 (m, 3H), 7.39 (t, $J = 7.2$ Hz, 1H), 7.30-7.28 (m, 1H), 7.22 (d, $J = 1.6$ Hz, 1H), 7.16-7.13 (m, 3H), 7.06 (d, $J = 8.4$ Hz, 2H), 3.40 (s, 3H), 2.35 (s, 3H); $^{13}$C NMR (CDCl$_3$, 100 MHz, 298 K): δ = 146.92, 146.62, 134.80, 132.76, 130.07, 128.63, 128.46, 127.56, 126.63, 126.29, 123.32, 123.17, 120.71, 112.24, 40.87, 20.85; GC-MS: m/z = 247.1 [M]$^+$, 231.1, 217.1.

**7b:**

$^1$H NMR (CDCl$_3$, 400 MHz, 298 K): δ 7.75-7.67 (m, 3H), 7.42 (t, $J = 7.2$ Hz, 1H), 7.33-7.29 (m, 4H), 7.23 (dd, $J = 2.4$ Hz, $J = 11.2$ Hz, 1H), 7.13 (d, $J = 7.6$ Hz, 2H), 7.04 (t, $J = 7.2$ Hz, 1H), 3.45 (s, 3H); GC-MS: m/z = 233.1 [M-1]$^+$, 217.1, 127.1.

**8a:**

$^1$H NMR (CDCl$_3$, 400 MHz, 298 K): δ 7.71 (d, $J = 8.4$ Hz, 2H), 7.61 (d, $J = 8.0$ Hz, 1H), 7.40-7.35 (m, 2H), 7.29-7.27 (m, 1H), 7.19-7.08 (m, 5H), 5.77 (s, 1H), 2.33 (s, 3H); $^{13}$C NMR (CDCl$_3$, 100 MHz, 298 K): δ = 141.74, 140.12, 134.76, 131.42, 130.00, 129.18, 128.92, 127.68, 126.45, 126.41, 123.22, 119.62, 119.39, 110.31, 20.81.

**8b:**

$^1$H NMR (CDCl$_3$, 400 MHz, 298 K): δ 7.69 (d, $J = 8.8$ Hz, 2H), 7.67 (d, $J = 8.0$ Hz, 1H), 7.46-7.41 (m, 2H), 7.35-7.31 (m, 1H), 7.27-7.21 (m, 2H), 7.01 (d, $J = 6.8$ Hz, 2H), 6.84 (d, $J = 7.6$ Hz, 1H), 5.86 (s, 1H), 2.37 (s, 3H); $^{13}$C NMR (CDCl$_3$, 100 MHz, 298 K): δ 142.83,
140.94, 139.27, 134.60, 129.20, 129.08, 127.60, 126.37, 123.39, 122.26, 120.06, 118.95, 115.33, 111.55, 29.69.

8c: 

$^1$H NMR (CDCl$_3$, 400 MHz, 298 K): δ 7.74 (d, $J = 8.0$ Hz, 2H), 7.62 (d, $J = 8.0$ Hz, 1H), 7.39 (t, $J = 6.8$ Hz, 1H), 7.33 (d, $J = 8.0$ Hz, 1H), 7.31-7.27 (m, 1H), 7.24-7.18 (m, 4H), 7.00 (t, $J = 7.2$ Hz, 1H), 5.56 (bs, 1H), 2.30 (s, 3H); GC-MS: m/z = 233.1 [M]$^+$, 217.1, 135.9.

9a: 

$^1$H NMR (DMSO, 400 MHz, 298 K): δ 8.35 (s, 1H), 7.77-7.72 (m, 2H), 7.65 (d, $J = 8.0$ Hz, 1H), 7.38-7.34 (m, 2H), 7.25-7.19 (m, 4H), 7.13 (t, $J = 8.4$ Hz, 2H); $^{13}$C NMR (CDCl$_3$, 100 MHz, 298 K): δ = 159.42, 157.02, 141.63, 138.68, 134.61, 129.23, 128.93, 127.62, 126.48, 126.34, 123.33, 121.02, 120.94, 119.32, 116.09, 115.87, 110.29. $^{19}$F NMR (CDCl$_3$, 400 MHz, 298 K): δ = -121.25.

9b: 

$^1$H NMR (CDCl$_3$, 400 MHz, 298 K): δ 7.79-7.77 (m, 2H), 7.68 (d, $J = 8.4$ Hz, 1H), 7.48-7.41 (m, 2H), 7.36-7.31 (m, 3H), 7.26 (dd, $J = 2.4$ Hz, $J = 8.8$ Hz, 1H), 7.21-7.19 (m, 2H), 7.03-6.99 (m, 1H), 5.91 (s, 1H); $^{13}$C NMR (CDCl$_3$, 100 MHz, 298 K): δ = 143.03, 140.96, 134.75, 129.54, 129.28, 127.77, 126.61, 126.57, 123.61, 121.51, 120.16, 118.38, 111.71.
$^9c$: \textsuperscript{S5}

\begin{align*}
\text{H NMR (CDCl}_3, \text{ 400 MHz, 298 K): } & \delta 7.71 \text{ (d, } J = 8.8 \text{ Hz, } 2\text{H}), 7.59 \text{ (d, } J = 8.4 \text{ Hz, } 2\text{H}), \\
7.39-7.35 \text{ (m, } 1\text{H}), 7.25-7.21 \text{ (m, } 2\text{H}), 7.19-7.15 \text{ (m, } 2\text{H}), 7.12 \text{ (dd, } J = 2.4 \text{ Hz, } J = 8.8 \text{ Hz, } 2\text{H}), 6.93-6.89 \text{ (m, } 2\text{H}), 3.83 \text{ (s, } 3\text{H}); \\
\text{C NMR (CDCl}_3, \text{ 100 MHz, 298 K): } & \delta = 155.42, 142.85, 135.44, 134.75, 129.06, 128.46, 127.57, 126.33, 126.16, 122.79, 122.46, 118.82, 114.67, 108.66, 55.49.
\end{align*}

\begin{align*}
10: \textsuperscript{S7}
\text{H NMR (CDCl}_3, \text{ 400 MHz, 298 K): } & \delta 7.72 \text{ (d, } J = 8.8 \text{ Hz, } 2\text{H}), 7.62 \text{ (d, } J = 8.4 \text{ Hz, } 2\text{H}), \\
7.40-7.36 \text{ (m, } 2\text{H}), 7.29-7.27 \text{ (m, } 1\text{H}), 7.20-7.18 \text{ (m, } 3\text{H}), 7.14-7.11 \text{ (m, } 2\text{H}), 5.80 \text{ (s, } 1\text{H}), 2.95-2.85 \text{ (m, } 1\text{H}), 1.26 \text{ (d, } J = 6.8 \text{ Hz, } 6\text{H}); \\
\text{C NMR (CDCl}_3, \text{ 100 MHz, 298 K): } & \delta = 142.34, 141.50, 140.36, 134.68, 129.04, 128.85, 127.58, 127.23, 126.33, 123.13, 119.62, 118.99, 110.43, 33.42, 24.11.
\end{align*}

\begin{align*}
11: \textsuperscript{S8}
\text{H NMR (CDCl}_3, \text{ 400 MHz, 298 K): } & \delta 7.73 \text{ (d, } J = 8.4 \text{ Hz, } 2\text{H}), 7.64 \text{ (d, } J = 8.4 \text{ Hz, } 1\text{H}), \\
7.40-7.37 \text{ (m, } 2\text{H}), 7.30-7.27 \text{ (m, } 1\text{H}), 7.24-7.19 \text{ (m, } 1\text{H}), 6.78 \text{ (s, } 2\text{H}), 6.63 \text{ (s, } 1\text{H}), 5.76 \text{ (s, } 1\text{H}), 2.29 \text{ (s, } 6\text{H}); \\
\text{C NMR (CDCl}_3, \text{ 100 MHz, 298 K): } & \delta = 142.85, 141.09, 139.09, 134.63, 129.05, 127.61, 126.44, 126.35, 123.35, 123.30, 120.15, 116.10, 111.65.
\end{align*}

\begin{align*}
12: \textsuperscript{S2}
\text{H NMR (CDCl}_3, \text{ 400 MHz, 298 K): } & \delta 7.70-7.66 \text{ (m, } 2\text{H}), 7.50 \text{ (d, } J = 8.0 \text{ Hz, } 1\text{H}), 7.32 \text{ (t, } J = 7.2 \text{ Hz, } 1\text{H}), 7.19 \text{ (t, } J = 7.2 \text{ Hz, } 1\text{H}), 7.00 \text{ (s, } 2\text{H}), 6.95 \text{ (dd, } J = 2.0 \text{ Hz, } J = 8.8 \text{ Hz, } 1\text{H)},
\end{align*}

s8
6.55 (d, J = 2.0 Hz, 1H), 5.26 (s, 1H), 2.35 (s, 3H), 2.21 (s, 6H); GC-MS: m/z = 261.3 [M]+, 246.3, 231.2.

\[ \text{13:} \]

\({ }^{1} \text{H NMR (CDCl}_3, 400 \text{ MHz, 298 K): } \delta 7.69-7.66 \text{ (m, 2H), 7.49 (d, } J = 8.0 \text{ Hz, 1H), 7.37-7.30 \text{ (m, 2H), 7.28-7.26 \text{ (m, 2H), 7.19 (t, } J = 7.6 \text{ Hz, 1H), 6.95 (dd, } J = 2.0 \text{ Hz, } J = 8.8 \text{ Hz, 1H), 6.56 \text{ (s, 1H), 5.29 (s, 1H), 3.29-3.19 \text{ (m, 2H), 1.15 (d, } J = 6.4 \text{ Hz, 12H); GC-MS: m/z = 303.1 [M]+, 288.1, 218.1.} \]

\[ \text{14:} \]

\({ }^{1} \text{H NMR (CDCl}_3, 400 \text{ MHz, 298 K): } \delta 8.08 \text{ (d, } J = 8.0 \text{ Hz, 1H), 7.90 (d, } J = 8.0 \text{ Hz, 1H), 7.76 \text{ (dd, } J = 2.8 \text{ Hz, } J = 8.4 \text{ Hz, 2H), 7.63 (dd, } J = 2.8 \text{ Hz, } J = 6.8 \text{ Hz, 1H), 7.60 (d, } J = 8.0 \text{ Hz, 1H), 7.55-7.44 \text{ (m, 4H), 7.41-7.37 \text{ (m, 1H), 7.32-7.24 \text{ (m, 3H), 6.12 (s, 1H). GC-MS: m/z = 269.3 [M]+, 133.8, 127.2.} \]

\[ \text{15:} \]

\({ }^{1} \text{H NMR (CDCl}_3, 400 \text{ MHz, 298 K): } \delta 7.66-7.56 \text{ (m, 3H), 7.40-7.25 \text{ (m, 6H), 7.18 (t, } J = 7.2 \text{ Hz, 1H), 6.87 (d, } J = 8.8 \text{ Hz, 1H), 6.81 (s, 1H), 4.39 (s, 2H); } ^{13} \text{C NMR (CDCl}_3, 100 \text{ MHz, 298 K): } \delta 145.72, 139.12, 135.12, 128.91, 128.64, 127.56, 127.28, 126.28, 125.95, 122.01, 117.81, 104.58, 48.28. \]

\[ \text{16:} \]

\({ }^{1} \text{H NMR (CDCl}_3, 400 \text{ MHz, 298 K): } \delta 8.08 (d, J = 8.0 \text{ Hz, 1H), 7.90 (d, } J = 8.0 \text{ Hz, 1H), 7.76 (dd, } J = 2.8 \text{ Hz, } J = 8.4 \text{ Hz, 2H), 7.63 (dd, } J = 2.8 \text{ Hz, } J = 6.8 \text{ Hz, 1H), 7.60 (d, } J = 8.0 \text{ Hz, 1H), 7.55-7.44 \text{ (m, 4H), 7.41-7.37 \text{ (m, 1H), 7.32-7.24 \text{ (m, 3H), 6.12 (s, 1H). GC-MS: m/z = 269.3 [M]+, 133.8, 127.2.} \]

\[ \text{s9} \]
$^1$H NMR (CDCl$_3$, 400 MHz, 298 K): δ 7.69-7.60 (m, 3H), 7.38-7.31 (m, 3H), 7.22-7.17 (m, 3H), 6.91 (dd, $J$ = 2.4 Hz, $J$ = 8.8 Hz, 1H), 6.86 (d, $J$ = 2.0 Hz, 1H), 4.40 (s, 2H), 4.16 (s, 1H), 2.37 (s, 3H); $^{13}$C NMR (CDCl$_3$, 100 MHz, 298 K): δ = 145.82, 136.96, 136.08, 135.17, 129.33, 128.90, 127.60, 126.27, 125.96, 121.97, 117.85, 104.58, 48.10, 21.08.

$^1$H NMR (CDCl$_3$, 400 MHz, 298 K): δ 7.63-7.57 (m, 2H), 7.48-7.46 (m, 1H), 7.41-7.39 (m, 2H), 7.33-7.28 (m, 3H), 7.24-7.21 (m, 1H), 7.16-7.12 (m, 1H), 6.86 (d, $J$ = 8.8 Hz, 1H), 6.62 (s, 1H), 4.63-4.58 (m, 1H), 4.18 (s, 1H), 1.55 (dd, $J$ = 1.2 Hz, $J$ = 6.8 Hz, 3H); $^{13}$C NMR (CDCl$_3$, 100 MHz, 298 K): δ = 144.87, 144.77, 135.00, 128.76, 128.64, 127.51, 127.33, 126.91, 126.12, 125.94, 125.83, 121.87, 117.93, 105.62, 53.40, 24.80.

$^1$H NMR (CDCl$_3$, 400 MHz, 298 K): δ 7.66-7.58 (m, 3H), 7.34 (t, $J$ = 7.6 Hz, 1H), 7.17 (t, $J$ = 7.6 Hz, 1H), 6.85 (dd, $J$ = 2.4 Hz, $J$ = 8.8 Hz, 1H), 6.80 (s, 1H), 3.72 (bs, 1H), 3.44-3.37 (m, 1H), 2.15-2.12 (m, 2H), 1.83-1.77 (m, 2H), 1.71-1.67 (m, 1H), 1.48-1.38 (m, 2H), 1.31-1.20 (m, 3H); GC-MS: m/z = 225.1 [M]$^+$, 182.1, 135.9.

$^1$H NMR (CDCl$_3$, 400 MHz, 298 K): δ 7.65 (d, $J$ = 8.4 Hz, 1H), 7.61-7.58 (m, 2H), 7.34 (t, $J$ = 7.2 Hz, 1H), 7.19 (t, $J$ = 7.2 Hz, 1H), 7.03 (d, $J$ = 2.0 Hz, 1H), 6.88 (dd, $J$ = 2.4 Hz, $J$ = 8.8 Hz, 1H), 3.45 (bs, 1H), 1.41 (s, 9H); $^{13}$C NMR (CDCl$_3$, 100 MHz, 298 K): δ = 144.33, 134.83, 128.53, 127.45, 126.10, 126.07, 122.19, 120.92, 109.34, 51.50, 29.87.
$^{20}$:  
\[
\begin{align*}
\text{H NMR (CDCl}_3, 400 MHz, 298 K): & \delta 7.57-7.49 (m, 3H), 7.25 (t, J = 7.2 Hz, 1H), 7.08 (t, J = 7.0 Hz, 1H), 6.74-6.68 (m, 2H), 3.08 (t, J = 6.8 Hz, 2H), 1.55-1.52 (m, 2H), 1.38-1.33 (m, 2H), 0.88 (t, J = 6.8 Hz, 3H); \\
\text{C NMR (CDCl}_3, 100 MHz, 298 K): & \delta = 146.26, 135.41, 128.91, 127.72, 127.49, 126.35, 125.95, 121.86, 118.08, 104.24, 43.78, 31.62, 20.47, 14.04.
\end{align*}
\]

$^{21}$:  
\[
\begin{align*}
\text{H NMR (CDCl}_3, 400 MHz, 298 K): & \delta 7.68-7.60 (m, 3H), 7.34 (t, J = 6.8 Hz, 1H), 7.15 (t, J = 7.2 Hz, 1H), 7.08 (d, J = 8.4 Hz, 1H), 6.83 (s, 1H), 3.35 (t, J = 7.6 Hz, 4H), 1.73-1.64 (m, 4H), 0.98 (t, J = 7.2 Hz, 6H); \\
\text{C NMR (CDCl}_3, 100 MHz, 298 K): & \delta = 146.22, 135.27, 128.71, 127.35, 126.07, 125.84, 121.38, 115.89, 105.25, 53.03, 20.55, 11.49.
\end{align*}
\]

$^{22}$:  
\[
\begin{align*}
\text{H NMR (CDCl}_3, 400 MHz, 298 K): & \delta 7.79-7.73 (m, 6H), 7.43 (td, J = 0.8 Hz, J = 7.6 Hz, 2H), 7.37-7.30 (m, 4H), 7.21 (d, J = 1.6 Hz, 2H), 3.51 (s, 8H); \\
\text{GC-MS: m/z} = 228.3 [M]^+, 182.1, 155.2, 135.9.
\end{align*}
\]

$^{23}$:  
\[
\begin{align*}
\text{H NMR (CDCl}_3, 400 MHz, 298 K): & \delta 8.23-8.21 (m, 1H), 7.83 (t, J = 4.4 Hz, 1H), 7.58 (d, J = 8.0 Hz, 1H), 7.51-7.46 (m, 2H), 7.42 (t, J = 8.0 Hz, 1H), 7.10 (d, J = 7.2 Hz, 1H), 3.99 (t, J = 4.4 Hz, 4H), 3.12 (t, J = 4.0 Hz, 4H); \\
\text{GC-MS: m/z} = 263.0 [M]^+, 204.0, 176.0.
\end{align*}
\]
1H NMR (CDCl₃, 400 MHz, 298 K): δ 8.70 (d, J = 8.4 Hz, 1H), 8.63-8.60 (m, 1H), 8.32 (d, J = 7.6 Hz, 1H), 7.82-7.80 (m, 1H), 7.68-7.61 (m, 2H), 7.58-7.54 (m, 2H), 7.30 (s, 1H), 4.03 (t, J = 4.4 Hz, 4H), 3.19 (bs, 4H); GC-MS: m/z = 263.1 [M]+, 204.2, 176.2.

25: S13

1H NMR (CDCl₃, 400 MHz, 298 K): δ 8.54 (d, J = 8.4 Hz, 2H), 8.34 (s, 1H), 8.02 (d, J = 7.6 Hz, 2H), 7.51-7.44 (m, 4H), 4.05 (t, J = 4.4 Hz, 4H), 3.54 (t, J = 4.4 Hz, 4H); GC-MS: m/z = 263.0 [M]+, 204.0, 176.0.

26: S14

1H NMR (CDCl₃, 400 MHz, 298 K): δ 8.73 (d, J = 2.4 Hz, 1H), 8.01-7.98 (m, 2H), 7.48 (dd, J = 9.6 Hz, J = 2.4 Hz, 1H), 7.34-7.31 (m, 1H), 7.03 (d, J = 2.4 Hz, 1H), 3.92 (t, J = 4.8 Hz, 4H), 3.29 (t, J = 4.8 Hz, 4H); GC-MS: m/z = 214.1 [M]+, 156.0, 128.0.

27: S12

1H NMR (CDCl₃, 400 MHz, 298 K): δ 7.97 (t, J = 8.8 Hz, 2H), 7.42-7.34 (m, 2H), 7.20 (t, J = 7.2 Hz, 1H), 6.91 (d, J = 7.6 Hz, 1H), 6.84 (s, 1H), 3.94 (t, J = 4.8 Hz, 4H), 3.81 (s, 3H), 3.30 (t, J = 4.8 Hz, 4H); GC-MS: m/z = 266.3 [M]+, 208.2, 180.2.
1H NMR (CDCl₃, 400 MHz, 298 K): δ 6.73 (d, J = 8.4 Hz, 1H), 6.55 (s, 1H), 6.35 (d, J = 8.0 Hz, 1H), 5.91 (s, 2H), 3.85 (t, J = 4.4 Hz, 4H), 3.04 (t, J = 4.4 Hz, 4H); GC-MS: m/z = 207.2 [M⁺], 149.2.

1H NMR (CDCl₃, 400 MHz, 298 K): δ 8.19 (d, J = 7.2 Hz, 1H), 7.81 (d, J = 8.4 Hz, 1H), 7.51 (d, J = 8.4 Hz, 1H), 7.47-7.43 (m, 2H), 7.39 (t, J = 8.0 Hz, 1H), 7.06 (d, J = 7.2 Hz, 1H), 3.06-3.03 (m, 4H), 1.86-1.83 (m, 4H), 1.68-1.64 (m, 2H); GC-MS: m/z = 210.2 [M-1]⁺, 154.1, 127.1.

1H NMR (CDCl₃, 400 MHz, 298 K): δ 7.90-7.88 (m, 1H), 7.80-7.76 (m, 1H), 7.44-7.41 (m, 2H), 7.32 (d, J = 4.4 Hz, 2H), 7.05 (t, J = 4.0 Hz, 1H), 4.04 (bs, 1H), 2.14 (s, 3H), 2.03 (s, 6H), 1.71 (s, 6H); GC-MS: m/z = 277.1 [M⁺], 220.1, 135.1.

1H NMR (CDCl₃, 400 MHz, 298 K): δ 6.90 (s, 4H), 3.86 (t, J = 4.8 Hz, 8H), 3.07 (t, J = 4.8 Hz, 8H); GC-MS: m/z = 248.2 [M⁺], 190.1, 132.1.
5. $^1$H NMR, $^{13}$C NMR and MS spectra for important compounds
6. References