A chemo- and regioselective C6-functionalization of 2,3-disubstituted indoles: highly efficient synthesis of diarylindol-6-ylmethanes

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1. NMR spectra of products 3 (S2-S23)

2. HPLC spectra of product 3aa (S24)

3. X-ray single crystal data for compound 3ae (S25-S26)
1. NMR spectra of products 3

$^1$H NMR of compound 3aa

$^{13}$C NMR of compound 3aa
$^1$H NMR of compound 3ba

$^{13}$C NMR of compound 3ba
$^1$H NMR of compound 3ca

\begin{figure}
\centering
\includegraphics[width=\textwidth]{hnmr}
\caption{$^1$H NMR spectrum of compound 3ca.}
\end{figure}

$^{13}$C NMR of compound 3ca

\begin{figure}
\centering
\includegraphics[width=\textwidth]{cnmr}
\caption{$^{13}$C NMR spectrum of compound 3ca.}
\end{figure}
$^1$H NMR of compound 3da

$^{13}$C NMR of compound 3da
$^1$H NMR of compound 3ea

$^{13}$C NMR of compound 3ea
$^1$H NMR of compound 3ga

$^{13}$C NMR of compound 3ga
$^1$H NMR of compound 3ha

$^{13}$C NMR of compound 3ha
$^{1}H$ NMR of compound 3ia

$^{13}C$ NMR of compound 3ia
**$^1$H NMR of compound 3ja**

**$^{13}$C NMR of compound 3ja**
$^1$H NMR of compound 3la

$^{13}$C NMR of compound 3la
$^1$H NMR of compound 3na

$^1$C NMR of compound 3na
\[ ^1\text{H NMR of compound 3oa} \]

\[ ^{13}\text{C NMR of compound 3oa} \]
$^1$H NMR of compound 3ab

$^{13}$C NMR of compound 3ab
\(^1\)H NMR of compound 3ac

\(^{13}\)C NMR of compound 3ac
$^1$H NMR of compound 3ad

$^{13}$C NMR of compound 3ad
$^1$H NMR of compound 3ae

$^{13}$C NMR of compound 3ae
$^{1}{H}$ NMR of compound 3af

$^{13}{C}$ NMR of compound 3af
$^1$H NMR of compound 3ag

$^{13}$C NMR of compound 3ag
$^1$H NMR of compound 3ah

$^{13}$C NMR of compound 3ah
2. HPLC spectra of product 3aa

### Chromatogram

![HPLC spectra of product 3aa](image)

### Integration Results

<table>
<thead>
<tr>
<th>No.</th>
<th>Peak Name</th>
<th>Retention Time (min)</th>
<th>Area (mAU:min)</th>
<th>Height (mAU)</th>
<th>Relative Area (%)</th>
<th>Relative Height (%)</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td>8.760</td>
<td>149,840</td>
<td>578,367</td>
<td>50.31</td>
<td>55.10</td>
<td>n.a.</td>
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<td>2</td>
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<td>8.073</td>
<td>139,129</td>
<td>471,216</td>
<td>49.69</td>
<td>44.90</td>
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<tr>
<td>Total</td>
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<td></td>
<td>279,969</td>
<td>1,040,582</td>
<td>100.00</td>
<td>100.00</td>
<td></td>
</tr>
</tbody>
</table>

---

### Chromatogram

![HPLC spectra of product 3aa](image)

### Integration Results

<table>
<thead>
<tr>
<th>No.</th>
<th>Peak Name</th>
<th>Retention Time (min)</th>
<th>Area (mAU:min)</th>
<th>Height (mAU)</th>
<th>Relative Area (%)</th>
<th>Relative Height (%)</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
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<td>6.810</td>
<td>60,843</td>
<td>274,985</td>
<td>62.69</td>
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<td>7.857</td>
<td>36,215</td>
<td>130,887</td>
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<tr>
<td>Total</td>
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<td>97,058</td>
<td>405,873</td>
<td>100.00</td>
<td>100.00</td>
<td></td>
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</tbody>
</table>
3. X-ray single crystal data for compound 3ae

The thermal ellipsoid was drawn at the 30% probability level.

**Empirical formula**: C_{24}H_{22}ClN_{1}O

**Formula weight**: 375.87

**Temperature**: 296 K

**Wavelength**: 0.71073 Å

**Crystal system**: Monoclinic

**Space group**: C 1 2/c 1

**Unit cell dimensions**:
- \(a = 17.424(3)\) Å
- \(\alpha = 90^\circ\)
- \(b = 12.100(2)\) Å
- \(\beta = 101.189(4)^\circ\)

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
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</thead>
<tbody>
<tr>
<td>Volume</td>
<td>3935.4(12) Å³</td>
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<tr>
<td>Z</td>
<td>8</td>
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<tr>
<td>Density (calculated)</td>
<td>1.269 Mg/m³</td>
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<tr>
<td>Absorption coefficient</td>
<td>0.207 mm⁻¹</td>
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<tr>
<td>F(000)</td>
<td>1584</td>
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<tr>
<td>Crystal size</td>
<td>0.25 x 0.2 x 0.15 mm³</td>
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<tr>
<td>Theta range for data collection</td>
<td>2.062 to 30.714°</td>
</tr>
<tr>
<td>Index ranges</td>
<td>-16&lt;=h&lt;=25, -17&lt;=k&lt;=17, -27&lt;=l&lt;=26</td>
</tr>
<tr>
<td>Reflections collected</td>
<td>19722</td>
</tr>
<tr>
<td>Independent reflections</td>
<td>6111 [R(int) = 0.0432]</td>
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<tr>
<td>Completeness to theta = 25.242°</td>
<td>100.0 %</td>
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<td>Absorption correction</td>
<td>Semi-empirical from equivalents</td>
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<td>Max. and min. transmission</td>
<td>0.7461 and 0.6912</td>
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<tr>
<td>Refinement method</td>
<td>Full-matrix least-squares on F²</td>
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<td>Data / restraints / parameters</td>
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<tr>
<td>Goodness-of-fit on F²</td>
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<tr>
<td>Final R indices [I&gt;2sigma(I)]</td>
<td>R₁ = 0.0478, wR² = 0.1126</td>
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<td>R indices (all data)</td>
<td>R₁ = 0.1218, wR² = 0.1450</td>
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<td>Extinction coefficient</td>
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<tr>
<td>Largest diff. peak and hole</td>
<td>0.181 and -0.307 e.Å⁻³</td>
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</tbody>
</table>