Supporting Information

Selective oxidation of terminal aryl and aliphatic alkenes to aldehydes catalyzed by iron(III) porphyrins with triflate as counter anion

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**General information**: Unless otherwise indicated, all reactions were performed under argon atmosphere and all the solvents were dried and freshly distilled. Alkenes were obtained commercially and distilled under reduced pressure before used. $^1$H NMR spectra were measured on Varian Mercury 300 spectrometer. EI mass were determined on a HP5989A mass spectrometer. IR spectra were measured on Bio-Rad FTS-185 spectrometer. UV-Vis were determined on a CARY100 spectrometer. Elemental analyses were determined by Elementar Vario EL. GC analyses were performed on a Varian CP-3800, SPB$^1$T-5, FID and $n$-dodecane was used as the internal standard.

**Experimental procedures and characterizations:**

**PhIO$^{[1]}$**:

\[
\text{PhI(OAc)}_2 \xrightarrow{\text{NaOH}} \xrightarrow{\text{H}_2\text{O}} \text{PhIO}
\]

To finely ground PhI(OAc)$_2$ (8.05 g, 25.0 mmol) placed in a 100 mL conical flask was added 45 mL of a 3M NaOH aqueous solution over a period of 10 mins. The resulting yellow heterogeneous mixture was then stirred for 90 mins at rt. The reaction medium was diluted with 40 mL of H$_2$O and vigorously stirred for 1 h. The yellow solid was collected on a funnel, washed with H$_2$O ($2 \times 100$ mL) and CHCl$_3$ (100 mL) before being dried under vacuum for 12 h. PhIO was obtained as a yellow powder (4.8 g, 87% yield).

Anal. Calcd. for C$_6$H$_5$IO: C, 32.76; H, 2.29. Found: C, 32.91; H, 2.20.

**[Fe(2,6-Cl$_2$TPP)OTf]$^{[2]}$**:

A mixture of [Fe(2,6-Cl$_2$TPP)Cl] (2.00 g, 2.04 mmol) and AgOTf (524 mg, 2.04 mmol) in THF (200 mL) was refluxed gently for 1 h and then filtered through a coarse frit. Heptane (200 mL) was added gradually with continuous swirling and set aside overnight for crystallization. The product was collected by filtration, washed with heptane and dried under vacuum (1.99 g, 88% yield).
IR (KBr): \(\nu_{\text{max}}\) 1652, 1523, 1488, 1336, 1082, 988, 939, 760 cm\(^{-1}\);
UV-Vis (CH\(_2\)Cl\(_2\), nm): \(\lambda_{\text{max}}\) = 407, 514, 648.

**Typical procedure for [Fe(2,6-Cl\(_2\)TPP)OTf]-catalyzed aryl alkenes to aldehydes:**

To a solution of [Fe(2,6-Cl\(_2\)TPP)OTf] (4.4 mg, 0.004 mmol), styrene (62.4 mg, 0.6 mmol) and DCM (2 mL) was added PhIO (44.0 mg, 0.2 mmol). The resulting heterogeneous mixture was stirred at room temperature for 6 h and then filtered through a short column of silica (2-3 cm), washed with DCM. The yield was determined by \(^1\)H NMR with PhTMS as an internal standard.

**Typical procedure for [Fe(2,6-Cl\(_2\)TPP)OTf]-catalyzed aliphatic alkenes to aldehydes:**

To a solution of [Fe(2,6-Cl\(_2\)TPP)OTf] (6.6 mg, 0.006 mmol), dodec-1-ene (33.7 mg, 0.2 mmol) in DCM (1 mL) was added PhIO (52.8 mg, 0.24 mmol). The resulting heterogeneous mixture was stirred at room temperature for 1 h. Then dioxane (3 mL) was added and the reaction mixture was stirred at 80 °C for 3 h. After cooling to room temperature, the mixture was filtered through a short column of silica (2-3 cm), washed with DCM. The yield was determined by \(^1\)H NMR with PhTMS as the internal standard.

**2-phenylacetaldehyde**\(^{[3]}\)

![2-phenylacetaldehyde structure](image)

\(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) 9.75 (t, \(J = 2.4\) Hz, 1H), 7.41-7.21 (m, 5H), 3.69 (d, \(J = 2.4\) Hz, 2H);
EI-MS m/z(relative intensity): 120(M\(^+\), 70), 91(100), 44(54), 107(40), 77(39), 79(31), 105(31), 45(25).

**2-p-tolylacetaldehyde**\(^{[3]}\)

![2-p-tolylacetaldehyde structure](image)

\(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) 9.73 (t, \(J = 2.1\) Hz, 1H), 7.18 (d, \(J = 7.8\) Hz, 2H), 7.11 (d, \(J = 7.8\) Hz, 2H), 3.65 (d, \(J = 2.1\) Hz, 2H), 2.35 (s, 3H);
EI-MS m/z(relative intensity): 134(M\(^+\), 32), 105(100), 119(93), 84(91), 91(88), 86(57), 120(55), 77(45).

**2-(4-methoxyphenyl)acetaldehyde**\(^{[3]}\)
2-(4-chlorophenyl)acetaldehyde \[4\]

\[
\begin{array}{c}
\text{CHO} \\
\text{Cl}
\end{array}
\]

$^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 9.71 (t, $J = 2.1$ Hz, 1H), 7.85 (d, $J = 8.7$ Hz, 2H), 7.32 (d, $J = 8.7$ Hz, 2H), 3.88 (d, $J = 2.1$ Hz, 2H);
EI-MS m/z(relative intensity): 154(M$^+$, 18), 84(100), 139(93), 140(65), 86(61), 77(53), 125(50), 111(43).

2-(3-chlorophenyl)acetaldehyde \[5\]

\[
\begin{array}{c}
\text{CHO} \\
\text{Cl}
\end{array}
\]

$^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 9.75 (t, $J = 1.8$ Hz, 1H), 7.34-7.28 (m, 2H), 7.23 (s, 1H), 7.12-7.10 (m, 1H), 3.69 (d, $J = 1.8$ Hz, 2H);
EI-MS m/z(relative intensity): 154(M$^+$, 40), 84(100), 139(86), 125(82), 86(68), 140(67), 91(60), 77(53).

2-(2-chlorophenyl)acetaldehyde \[6\]

\[
\begin{array}{c}
\text{CHO} \\
\text{Cl}
\end{array}
\]

$^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 9.76 (t, $J = 1.5$ Hz, 1H), 7.45-7.42 (m, 1H), 7.29-7.22 (m, 3H), 3.85 (s, 2H);
EI-MS m/z(relative intensity): 154(M$^+$, 6), 84(100), 86(64), 47(17), 49(14), 125(14), 88(10), 139(9).

2-(4-fluorophenyl)acetaldehyde \[3\]

\[
\begin{array}{c}
\text{CHO} \\
\text{F}
\end{array}
\]

$^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 9.75 (t, $J = 1.5$ Hz, 1H), 7.21-7.16 (m, 2H), 7.09-7.04 (m, 2H), 3.69 (d, $J = 1.5$ Hz, 2H);
EI-MS m/z(relative intensity): 138(M$^+$, 9), 109(100), 123(38), 95(20), 83(14), 125(13), 97(10), 75(8).
2-(3-bromophenyl)acetaldehyde \[^7\]

\[
\begin{align*}
\text{Br} & \quad \text{CHO} \\
\end{align*}
\]

\(^{1}\text{H} \text{ NMR (300 MHz, CDCl}\text{\textsubscript{3}}\): \delta 9.78 (t, \(J = 2.1\ \text{Hz}, 1\text{H})
, 7.48 (d, \(J = 8.1\ \text{Hz}, 1\text{H})
, 7.42 (s, 1\text{H})
, 7.26-7.23 (m, 1\text{H})
, 7.18 (d, \(J = 7.8\ \text{Hz}, 1\text{H})
, 3.71 (d, \(J = 2.1\ \text{Hz}, 2\text{H})
;
\text{EI-MS m/z(relative intensity): 198(M\text{\textsuperscript{+}}, 6), 84(100), 86(64), 184(43), 77(40), 183(33), 185(33), 186(24).}
\]

2-phenylpropanal \[^8\]

\[
\begin{align*}
\text{CHO} \\
\end{align*}
\]

\(^{1}\text{H} \text{ NMR (300 MHz, CDCl}\text{\textsubscript{3}}\): \delta 9.69 (s, 1\text{H})
, 7.42-7.21 (m, 5\text{H})
, 3.64 (q, \(J = 7.2\ \text{Hz}, 1\text{H})
, 1.45 (d, \(J = 7.2\ \text{Hz}, 3\text{H})
;
\text{EI-MS m/z(relative intensity): 134(M\text{\textsuperscript{+}}, 3), 43(30), 105(28), 77(10), 58(10), 121(8), 117(8), 118(7).}
\]

2-(naphthalen-2-yl)acetaldehyde \[^8\]

\[
\begin{align*}
\end{align*}
\]

\(^{1}\text{H} \text{ NMR (300 MHz, CDCl}\text{\textsubscript{3}}\): \delta 9.75 (t, \(J = 2.1\ \text{Hz}, 1\text{H})
, 7.86-7.79 (m, 3\text{H})
, 7.69 (s, 1\text{H})
, 7.50-7.47 (m, 2\text{H})
, 7.52 (d, \(J = 8.4\ \text{Hz}, 1\text{H})
, 3.76 (d, \(J = 2.1\ \text{Hz}, 2\text{H})
;
\text{EI-MS m/z(relative intensity): 170(M\text{\textsuperscript{+}}, 18), 154(100), 153(60), 152(37), 155(23), 141(18), 127(14), 151(12).}
\]

1-phenylcyclopentanecarbaldehyde \[^9\]

\[
\begin{align*}
\end{align*}
\]

\(^{1}\text{H} \text{ NMR (300 MHz, CDCl}\text{\textsubscript{3}}\): \delta 9.36 (s, 1\text{H})
, 7.28-7.24 (m, 2\text{H})
, 7.19-7.15 (m, 3\text{H})
, 2.54-2.48 (m, 2\text{H})
, 1.90-1.84 (m, 2\text{H})
, 1.75-1.71 (m, 2\text{H})
, 1.67-1.61 (m, 2\text{H})
;
\text{EI-MS m/z(relative intensity): 174(M\text{\textsuperscript{+}}, 27), 105(100), 77(85), 115(38), 51(35), 173(33), 133(30), 120(28).}
\]

\(n\)-dodecanal \[^{10}\]

\[
\begin{align*}
\end{align*}
\]

\(^{1}\text{H} \text{ NMR (300 MHz, CDCl}\text{\textsubscript{3}}\): \delta 9.77 (t, \(J = 1.5\ \text{Hz}, 1\text{H})
, 2.42 (td, \(J = 7.2, 1.5\ \text{Hz}, 2\text{H})
, 1.66-1.58 (m, 2\text{H})
, 1.31-1.26 (m, 16\text{H})
, 0.88 (t, \(J = 6.6\ \text{Hz}, 3\text{H})
;
\text{EI-MS m/z(relative intensity): 184(M\text{\textsuperscript{+}}, 1), 43(100), 57(81), 41(80), 71(58),}
\]

S5
55(58), 82(53), 69(40).

<n-undecanal>\[^{[11]}\]

\[
\begin{align*}
\text{CHO} & & 4b \\
\end{align*}
\]

\(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) 9.77 (s, 1H), 2.42 (td, \(J = 7.2, 1.5 \text{ Hz}, 2\)H), 1.65-1.60 (m, 2H), 1.31-1.26 (m, 14H), 0.88 (t, \(J = 6.6 \text{ Hz}, 3\)H);
EI-MS m/z(relative intensity): 170(M\(^+\), 1), 57(100), 82(93), 43(87), 55(75), 41(72), 68(61), 71(60).

<n-palmitaldehyde>\[^{[12]}\]

\[
\begin{align*}
\text{CHO} & & 4c \\
\end{align*}
\]

\(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) 9.76 (t, \(J = 2.1 \text{ Hz}, 1\)H), 2.44-2.39 (m, 2H), 1.64-1.58 (m, 2H), 1.31-1.26 (m, 24H), 0.89 (t, \(J = 6.6 \text{ Hz}, 3\)H);
EI-MS m/z(relative intensity): 240(M\(^+\), 2), 43(25), 188(10), 58(8), 57(6), 242(6), 173(5), 160(5).

3-phenylpropanal\[^{[13]}\]

\[
\begin{align*}
\text{CHO} & & 4d \\
\end{align*}
\]

\(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) 9.83 (d, \(J = 0.9 \text{ Hz}, 1\)H), 7.33-7.19 (m, 5H), 2.96 (t, \(J = 7.5 \text{ Hz}, 2\)H), 2.79 (t, \(J = 7.5 \text{ Hz}, 2\)H);
EI-MS m/z(relative intensity): 134(M\(^+\), 1), 43(100), 58(45), 42(9), 91(7), 92(7), 44(3), 41(3).

4-phenylbutanal\[^{[13]}\]

\[
\begin{align*}
\text{CHO} & & 4e \\
\end{align*}
\]

\(^1\)H NMR (300 MHz, CDCl\(_3\)): \(\delta\) 9.74 (s, 1H), 7.31-7.27 (m, 2H), 7.23-7.16 (m, 3H), 2.65 (t, \(J = 7.2 \text{ Hz}, 2\)H), 2.44 (t, \(J = 7.2 \text{ Hz}, 2\)H), 1.95 (quintet, \(J = 7.2 \text{ Hz}, 2\)H);
EI-MS m/z(relative intensity): 148(M\(^+\), 4), 104(24), 91(19), 43(13), 105(8), 130(6), 92(6), 51(6).

ethyl 4-phenylbut-2-enoate (5)\[^{[14]}\]

\[
\begin{align*}
\text{Ph} & & \text{PhIO} & & 1)2 \text{ mol}\% \text{Fe(2,6-Cl}_2\text{TPP})\text{OTf, DCM, r.t.} & & \text{Ph} & & \text{COOEt} \\
\text{Ph} & & \text{Ph} & & 2)1.1 \text{ eq PPh}_3, 1.1 \text{ eq EDA / DCM, r.t.} & & \text{Ph} & & \\
\end{align*}
\]

To a solution of [Fe(2,6-Cl\(_2\)TPP)OTf] (4.4 mg, 0.004 mmol), styrene (62.4 mg, 0.6 mmol) and DCM (2 mL) was added PhIO (44.0 mg, 0.2 mmol). The resulting
A heterogeneous mixture was stirred at room temperature for 6 h and then the solvent and excess styrene were removed under reduced pressure. PPh₃ (57.6 mg, 0.22 mmol) and DCM (1 mL) were added to the mixture. To the mixture the solution of ethyl diazoacetate (25.1 mg, 0.22 mmol) in 1 mL DCM was added for 1 h and stirred at room temperature for additional 1 h. The crude product was purified by silica gel flash column chromatography (PE:EtOAc = 50:1) to give the product as a colorless oil (32.1 mg, 84% yield).

**1H NMR (300 MHz, CDCl₃):** δ 7.36-7.15 (m, 5H), 7.10 (dt, J = 15.8, 7.2 Hz, 1H), 5.81 (d, J = 15.8 Hz, 1H), 4.17 (q, J = 7.2 Hz, 2H), 3.52 (d, J = 7.2 Hz, 2H), 1.27 (t, J = 7.2 Hz, 3H);

EI-MS m/z(relative intensity): 190(M⁺, 56), 117(100), 115(70), 145(40), 116(34), 144(26), 91(25), 127(17);

IR (film): v_max 3028, 2981, 1719, 1652, 1496, 1270, 1163, 1042, 984, 699 cm⁻¹.

**1-phenylethane-1,2-diol (6)**

4.4 mg Fe(TDCPP)OTf (0.004 mmol), 62.4 mg styrene (0.6 mmol), 22 μL formic acid (0.6 mmol) were dissolved in 1 mL dichloromethane, followed by 44.0 mg PhIO (0.2 mmol) and additional 1 mL dichloromethane. The reaction mixture were stirred at room temperature for 10 h, after which the volatile were evaporated and 41.4 mg potassium carbonate (0.3 mmol) together with 6 mL methanol was added. After stirring at room temperature for 6 h, the reaction mixture was subjected to flash chromatography (ethyl acetate: petrol ether = 2:3) to afford the desired diol as a white solid (20.2 mg, 73%).

**1H NMR (300 MHz, CDCl₃):** δ 7.39-7.27 (m, 5H), 4.83-4.79 (m, 1H), 3.76-3.61 (m, 2H), 3.12 (brs, 1H), 2.70 (brs, 1H);

 EI-MS m/z(relative intensity): 138(M⁺, 11), 107(100), 79(56), 77(35), 108(8), 51(7), 78(6), 105(6);

IR (KBr): v_max 3208, 3061, 2963, 2933, 1494, 1448, 1343, 1101, 1053, 887, 832, 699 cm⁻¹.
References
Table SI Effect of different oxidants on $[\text{Fe(2,6-Cl}_2\text{TPP})\text{OTf}]$ catalyzed E-I reaction of styrene to phenylacetaldehyde:

![Diagram of reaction](image)

<table>
<thead>
<tr>
<th>Entry$^a$</th>
<th>Oxidant</th>
<th>Yield (%)$^b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>PhIO</td>
<td>45</td>
</tr>
<tr>
<td>2</td>
<td>TBHP</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>urea peroxide</td>
<td>1</td>
</tr>
<tr>
<td>4</td>
<td>m-CPBA</td>
<td>0</td>
</tr>
<tr>
<td>5</td>
<td>H$_2$O$_2$ (30% aq.)</td>
<td>0</td>
</tr>
<tr>
<td>6</td>
<td>NaClO (aq.)</td>
<td>0</td>
</tr>
<tr>
<td>7</td>
<td>oxone</td>
<td>6</td>
</tr>
</tbody>
</table>

$^a$ 0.3 mmol styrene, 0.2 mmol oxidant in 2 mL CH$_2$Cl$_2$ were stirred for 8 h; $^b$ yield based on GC with n-dodecane as the internal standard.
Table SII Optimization of [Fe(2,6-Cl<sub>2</sub>TPP)OTf] catalyzed E-I reaction of styrene to phenylacetaldehyde

<table>
<thead>
<tr>
<th>Entry&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Solvent</th>
<th>Temp.</th>
<th>Styrene:PhIO</th>
<th>Conversion(&lt;sup&gt;b&lt;/sup&gt;)</th>
<th>Yield(&lt;sup&gt;c&lt;/sup&gt;)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>DCM</td>
<td>rt</td>
<td>1:1.5</td>
<td>100</td>
<td>59</td>
</tr>
<tr>
<td>2</td>
<td>DCE</td>
<td>rt</td>
<td>1:1.5</td>
<td>100</td>
<td>45</td>
</tr>
<tr>
<td>3</td>
<td>dioxane</td>
<td>rt</td>
<td>1:1.5</td>
<td>56</td>
<td>57</td>
</tr>
<tr>
<td>4</td>
<td>Toluene</td>
<td>rt</td>
<td>1:1.5</td>
<td>59</td>
<td>49</td>
</tr>
<tr>
<td>5</td>
<td>EtOAc</td>
<td>rt</td>
<td>1:1.5</td>
<td>97</td>
<td>39</td>
</tr>
<tr>
<td>6</td>
<td>MeCN</td>
<td>rt</td>
<td>1:1.5</td>
<td>100</td>
<td>4</td>
</tr>
<tr>
<td>7</td>
<td>THF</td>
<td>rt</td>
<td>1:1.5</td>
<td>15</td>
<td>13</td>
</tr>
<tr>
<td>8</td>
<td>DCM</td>
<td>40</td>
<td>1:1.5</td>
<td>100</td>
<td>53</td>
</tr>
<tr>
<td>9</td>
<td>DCM</td>
<td>0</td>
<td>1:1.5</td>
<td>100</td>
<td>49</td>
</tr>
<tr>
<td>10</td>
<td>DCM</td>
<td>-20</td>
<td>1:1.5</td>
<td>100</td>
<td>35</td>
</tr>
<tr>
<td>11</td>
<td>DCM</td>
<td>rt</td>
<td>1:1.1</td>
<td>75</td>
<td>60</td>
</tr>
<tr>
<td>12</td>
<td>DCM</td>
<td>rt</td>
<td>1.5:1</td>
<td>-</td>
<td>45&lt;sup&gt;d&lt;/sup&gt;</td>
</tr>
<tr>
<td>13</td>
<td>DCM</td>
<td>rt</td>
<td>3:1</td>
<td>-</td>
<td>83&lt;sup&gt;d&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>a</sup> 0.2 mmol styrene; 0.3 mmol PhIO; 2 mL solvent; <sup>b</sup> determined by GC with n-dodecane as internal standard. <sup>c</sup> base on conversion. <sup>d</sup> based on oxidant.
Table SIII Optimization of [Fe(2,6-Cl₂TPP)OTf] catalyzed E-I reaction of aliphatic alkene

<table>
<thead>
<tr>
<th>Entry</th>
<th>X</th>
<th>Y</th>
<th>Solvent</th>
<th>Temp.</th>
<th>Conversion^b</th>
<th>Epoxide</th>
<th>Aldehyde</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2</td>
<td>1.5</td>
<td>2 mL DCE</td>
<td>r.t.-80</td>
<td>93</td>
<td>12</td>
<td>38</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>1.5</td>
<td>2 mL DCE</td>
<td>80</td>
<td>83</td>
<td>14</td>
<td>25</td>
</tr>
<tr>
<td>3</td>
<td>4</td>
<td>2</td>
<td>2 mL DCE</td>
<td>r.t.-80</td>
<td>&gt;99</td>
<td>trace</td>
<td>40</td>
</tr>
<tr>
<td>4</td>
<td>2</td>
<td>1.5</td>
<td>2 mL DCM</td>
<td>r.t.-40</td>
<td>&gt;99</td>
<td>51</td>
<td>17</td>
</tr>
<tr>
<td>5</td>
<td>4</td>
<td>2</td>
<td>1 mL DCE</td>
<td>r.t. 80</td>
<td>&gt;99</td>
<td>Not detected</td>
<td>47</td>
</tr>
<tr>
<td>6</td>
<td>2</td>
<td>1.5</td>
<td>1 mL DCM</td>
<td>r.t. 80</td>
<td>&gt;99</td>
<td>22</td>
<td>36</td>
</tr>
<tr>
<td>7</td>
<td>4</td>
<td>1.5</td>
<td>1 mL DCE</td>
<td>r.t. 80</td>
<td>&gt;99</td>
<td>Not detected</td>
<td>68</td>
</tr>
<tr>
<td>8</td>
<td>3</td>
<td>1.5</td>
<td>1 mL DCE</td>
<td>r.t. 80</td>
<td>&gt;99</td>
<td>Not detected</td>
<td>63</td>
</tr>
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<td>1 mL DCE</td>
<td>r.t. 80</td>
<td>&gt;99</td>
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<td>&gt;99</td>
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<td>r.t. 80</td>
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<td>r.t. 80</td>
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</tr>
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</table>

^a 0.2 mmol alkene were used; ^b Conversion and yield based on ^1H NMR with PhTMS as internal standard. ^c The alkene was treated with corresponding amount of catalyst and oxidant in 1 mL DCM or DCE at room temperature, after the oxidation was complete, additional dioxane were added and the reaction was run at 80°C till completion.