Supporting Information

Electrochemical Benzylic Oxidation of C-H Bonds

Jason A. Marko, Anthony Durgham, Stacey Lowery Bretz and Wei Liu*

Department of Chemistry and Biochemistry, Miami University, Oxford, Ohio, 45046, United States

Corresponding Author
wei.liu@miamioh.edu

Table of Contents

I. General Information ................................................................. S2
II. General Procedure for Benzylic C-H Oxidation .................... S2
III. Photograph guide for divided cell preparation ....................... S3
IV. Photographic guide for undivided cell preparation ............... S17
V. Supplementary Figures .......................................................... S25
VI. Characterization and Spectral Data ........................................ S29
I. General Information

All oxidation reactions were run under normal atmospheric conditions and at 50°C in both divided and undivided cells. Solvents were used as is and required no further purification. All purchased chemicals were of highest purity available from Sigma-Aldrich, Oakwood Chemical, AK Scientific, TCI America, or Fisher Scientific and were used without further purification. All synthetized chemicals were made according to their published procedure. Electrolysis was performed using C-H Instruments Model 1000C Series Multi-Potentiostat. GC/MS analyses were performed on a Thermo-Fisher Scientific Trace 1300 gas chromatograph fitted with a Thermo-Fischer Scientific ISQ QD single quadrupole mass spectrometer. 1H and 13C NMR spectra were obtained on a Bruker 500 Ultra Shield (500 MHz) spectrometer. Data is reported in ppm using CDCl₃ as solvent. Data reported as: chemical shifts (δ), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant (Hz); integrated intensity.

II. General Procedure for Benzylic C-H Oxidation

Without any provisions to ensure air and moisture were expelled, an undivided or divided cell was occupied with a stir bar and 0.1M NaHCO₃ solution (10 mL). Substrate (1 equiv., 2 mmol) and acetonitrile (2 mL) was added and stirred until most of the suspension was uniformly mixed. 70% aqueous tert-butyl hydroperoxide (6 equiv., 12 mmol) was added and heated to 50°C. The anode and cathode (100 PPI RVC) were placed into the solution, along with an aqueous Ag/AgCl reference electrode, and the reaction mixture was electrolyzed at a constant voltage of 1.1V for 15 hrs. The reaction mixtures were then extracted, and electrodes rinsed, with dichloromethane. The crude product mixture were then subjected to GCMS analysis and purified on SiO₂ to provide the desired oxidized product.
II. Photographic Guide for Divided Cell Preparation

1.) Below are the pieces of equipment needed: 2 sides of an undivided cell, rubber o-ring, a stir bar, 100 PPI RVC electrodes (2 – for anode and cathode) and graphite rods. The working electrode’s dimensions are approximately $\frac{1}{2}$” x 2”. The counter electrode is approximately 1”x1”. Electrodes were purchased from https://ergaerospace.com/.
2.) Place the rubber o-ring in the notch at the mouth of one of the divided cells and press it onto a piece of membrane and cut it out. The membrane was purchased from www.fuelcellstore.com.
3.) Once the membrane has been cut, place the rubber ring into the notch and put the membrane on top. Then, press the other side of the divided cell on top of the membrane and clamp together using a metal bracket.
4.) After the cell has be constructed, pipet 0.1M NaHCO₃ into the anode.
5.) After the NaHCO₃ solution, pipet 1 equivalent of substrate into the anode.
6.) To help with the solubility of the substrates, add acetonitrile into the anode.
7.) Place the cell on a stir plate to help the components of the solution to uniformly mix.
8.) While stirring, add 6 equivalents of *tert*-Butyl hydroperoxide.
9.) While mixing the solution, insert graphite rods into the RVC electrodes.
10.) After the electrodes are made, fill the cathode with 0.1M NaHCO₃ to match the liquid level on the anode so that there is no pressure differential that could cause forced solvent passage through the membrane.
11.) Insert the counter electrode into cathode and parafilm the top.
12.) Place the working and reference electrodes in the anode and parafilm.
13.) Submerge the divided cell into a sand bath and heat to 50°C.
14.) Connect the electrodes to the potentiostat and electrolyze at 1.1V for 15 hrs. In the picture, the green lead is the working electrode, the white lead is the reference electrode and the red lead is the counter electrode.
III. Photographic Guide for Undivided Cell Preparation

1.) Below are the pieces of equipment needed: 20mL scintillation vial, a stir bar, 100 PPI RVC electrodes, 16-gauge needles with stainless steel wire wrapped around the non-sharp end and a plastic cap with a hole cut into the middle for the reference electrode. The working and counter electrode’s dimensions are approximately ½” x 2”. The Electrodes were purchased from https://ergaerospace.com/.
2.) A stir bar is placed in the scintillation vial and 0.1M NaHCO is pipetted into the cell.
3.) Pipet, 1 equivalent of substrate into the anode.
4.) To help with the solubility of the substrates, add acetonitrile into the cell and placed onto a stir plate to help the components mix.
5.) While stirring, add 6 equivalents of \textit{tert}-Butyl hydroperoxide.
6.) While mixing the solution, the electrodes were put together by inserting the needle into the top of the electrode.
7.) Place the electrodes into the cell and parafilm the wires to help keep them in place. *Careful* care needs to be taken so that the electrodes do not touch.
8.) Place the reference electrode into the hole of the plastic cap and set the cap on top of the vial and place the electrode into the cell. The undivided cell is heated to 50°C and the electrodes are connected to the potentiostat and electrolyzed at 1.1V for 15 hrs.
IV. Supplementary Figures

Fig. S1. Additional optimization conditions using ethyl benzene measuring the effect of temperature and pH on product yield. \textsuperscript{a}Yields determined via Thermo-Fischer GC-MS; \textsuperscript{b}Phosphate Buffer used to obtain desired pH; \textsuperscript{c}Carbonate Buffer used to obtain desired pH
Fig. S2. Cyclic Voltammetry of Reaction Parameters vs. Ag/AgCl (0.1M NaHCO$_3$/MeCN, 0.17 mM ethyl benzene, 1 mM tBuOOH)

A: CV Potential range 0 – 0.8V.
B: CV Potential range 0.3-0.8V.
C: CV Potential range 0-1.3V.
<table>
<thead>
<tr>
<th>Substrate</th>
<th>Ref.</th>
<th>Literature Yield (%)</th>
<th>This Study's Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>55</td>
<td>70</td>
<td>64</td>
</tr>
<tr>
<td>3</td>
<td>56</td>
<td>49</td>
<td>56</td>
</tr>
<tr>
<td>5</td>
<td>41</td>
<td>84</td>
<td>81</td>
</tr>
<tr>
<td>6</td>
<td>41</td>
<td>56</td>
<td>51</td>
</tr>
<tr>
<td>19</td>
<td>17</td>
<td>65</td>
<td>75</td>
</tr>
<tr>
<td>22</td>
<td>57</td>
<td>67</td>
<td>81</td>
</tr>
</tbody>
</table>

**Table S1** Comparison of literature and this study's yields. The yields beneath the structures in Scheme 1 are those determined in the present study via Thermo-Fischer GC-MS.
V. Characterization and Spectral Data

**Compound 3**, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.13 g).  

**¹H NMR** (500 MHz, Chloroform-d) δ 7.93 (d, J = 7.9 Hz, 2H), 7.53 (t, J = 6.9 Hz, 1H), 7.43 (t, J = 7.6 Hz, 2H), 2.57 (s, 3H).  

**¹³C NMR** (126 MHz, Chloroform-d) δ 198.09, 137.08, 133.09, 128.56, 128.28, 26.57.  

**MS (EI):** m/z calc’d C₈H₈O [M]⁺ 120.05, found 120.05
Compound 11. Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as colorless solid (0.138 g). $^1$H NMR (500 MHz, Chloroform-d) δ 7.99 – 7.93 (m, 2H), 6.99 – 6.92 (m, 2H), 3.89 (s, 3H), 2.58 (s, 3H). $^{13}$C NMR (126 MHz, Chloroform-d) δ 196.81, 163.49, 130.61, 130.35, 113.69, 55.48, 26.36. MS (EI): m/z calc'd C$_9$H$_{10}$O$_2$ [M]$^+$ 150.07, found 150.07
Compound 13, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.184 g). $^1$H NMR (500 MHz, Chloroform-$d$) $\delta$ 7.91 (d, $J$ = 8.5 Hz, 2H), 7.45 (d, $J$ = 8.5 Hz, 2H), 2.61 (s, 3H). $^{13}$C NMR (126 MHz, Chloroform-$d$) $\delta$ 196.83, 139.57, 135.44, 129.74, 128.90, 26.57. MS (EI): $m/z$ calc’d C$_6$H$_7$ClO [M]$^+$ 156.01, found 156.01
**Compound 14**, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.145 g). $^1$H NMR (500 MHz, Chloroform-$d$) $\delta$ 8.09 – 8.03 (m, 3H), 7.82 – 7.76 (m, 3H), 2.66 (s, 4H). $^{13}$C NMR (126 MHz, Chloroform-$d$) $\delta$ 196.58, 139.92, 132.54, 128.72, 117.94, 116.41, 26.79. **MS (EI):** $m/z$ calc'd C$_9$H$_7$NO [M]$^+$ 145.05, found 145.05
**Compound 12**, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a pink solid (0.23 g). \textit{\textsuperscript{1}H NMR} (500 MHz, Chloroform-\textit{d}) $\delta$ 8.00 – 7.94 (m, 2H), 7.64 (d, $J$ = 8.5 Hz, 2H), 2.60 (s, 3H), 2.25 (s, 3H). \textit{\textsuperscript{13}C NMR} (126 MHz, Chloroform-\textit{d}) $\delta$ 196.88, 142.14, 132.95, 129.78, 118.78, 26.46, 24.87. \textbf{MS (EI)}: \textit{m/z} calc’d C\textsubscript{10}H\textsubscript{11}NO\textsubscript{2} [M]\textsuperscript{+} 177.08, found 177.08.
Compound 15, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a white solid (0.239 g). $^1$H NMR (500 MHz, Chloroform-d) $\delta$ 8.37 (dd, $J = 8.0$, 1.7 Hz, 2H), 7.75 (ddd, $J = 8.7$, 7.1, 1.7 Hz, 2H), 7.54 – 7.49 (m, 2H), 7.44 – 7.38 (m, 2H). $^{13}$C NMR (126 MHz, Chloroform-d) $\delta$ 177.23, 156.19, 134.83, 126.75, 123.92, 121.87, 118.00. MS (EI): m/z calc’d C$_{13}$H$_8$O$_2$ [M]$^+$ 196.05, found 196.05
Compound 16, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as colorless oil (0.151 g). $^1$H NMR (500 MHz, Chloroform-$d$) $\delta$ 7.54 (d, $J = 1.9$ Hz, 1H), 7.49 (dd, $J = 8.2$, 1.8 Hz, 1H), 7.04 (d, $J = 8.2$ Hz, 1H), 3.82 (s, 3H), 2.92 (q, $J = 7.2$ Hz, 2H), 2.26 (s, 3H), 1.15 (t, $J = 7.2$ Hz, 3H). $^{13}$C NMR (126 MHz, Chloroform-$d$) $\delta$ 199.65, 168.56, 151.38, 143.60, 135.74, 122.76, 121.30, 111.45, 56.03, 31.72, 20.67, 8.33. MS (EI): $m/z$ calc'd C$_{12}$H$_{14}$O$_4$ [M]$^+$ 222.09, found 222.09
**Compound 17**, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.169 g). **1H NMR** (500 MHz, Chloroform-d) δ 7.84 (dt, J = 8.4, 1.5 Hz, 2H), 7.65 – 7.58 (m, 1H), 7.54 – 7.48 (m, 2H). **13C NMR** (126 MHz, Chloroform-d) δ 196.78, 137.62, 132.44, 130.08, 128.30. **MS (EI):** m/z calc’d C_{13}H_{10}O [M]^{+} 182.07, found 182.07
**Compound 18.** Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a yellow solid (0.238 g). $^1$H NMR (500 MHz, Chloroform-$d$) $\delta$ 7.64 (d, $J = 7.4$ Hz, 2H), 7.50 – 7.44 (m, 4H), 7.30 – 7.25 (m, 2H). $^{13}$C NMR (126 MHz, Chloroform-$d$) $\delta$ 193.88, 144.40, 134.68, 134.12, 129.06, 124.26, 120.32. **MS (EI):** $m/z$ calc'd C$_{13}$H$_8$O [M]$^+$ 180.06, found 180.06
Compound 19, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.198 g). $^1$H NMR (500 MHz, Chloroform-d) $\delta$ 7.78 (d, $J$ = 7.7 Hz, 1H), 7.60 (td, $J$ = 7.6, 1.1 Hz, 1H), 7.50 (d, $J$ = 7.7 Hz, 1H), 7.41–7.35 (m, 1H), 3.19–3.14 (m, 2H), 2.73–2.68 (m, 2H). $^{13}$C NMR (126 MHz, Chloroform-d) $\delta$ 207.12, 155.19, 137.10, 134.62, 127.30, 126.72, 123.73, 36.24, 25.82. MS (EI): m/z calc’d C$_9$H$_8$O [M]$^+$ 132.05, found 132.05
**Compound 20**, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.14 g). $^1$H NMR (500 MHz, Chloroform-$d$) $\delta$ 8.50 (s, 1H), 8.07 (dd, $J = 8.6, 1.8$ Hz, 1H), 8.00 (d, $J = 8.1$ Hz, 1H), 7.92 (t, $J = 8.3$ Hz, 2H), 7.64 (ddd, $J = 8.2, 6.9, 1.4$ Hz, 1H), 7.59 (ddd, $J = 8.1, 6.9, 1.4$ Hz, 1H), 2.76 (s, 3H) $^1$C NMR (126 MHz, Chloroform-$d$) $\delta$ 198.14, 135.62, 134.52, 132.54, 130.22, 129.57, 128.49, 128.45, 127.81, 126.80, 123.92, 26.73. **MS (EI)**: m/z calc'd C$_{12}$H$_{10}$O [M]$^+$ 170.07, found 170.03
Compound 21, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.227 g). $^1$H NMR (500 MHz, Chloroform-$d$) $\delta$ 8.07 (d, $J = 8.4$ Hz, 2H), 7.72 (d, $J = 8.4$ Hz, 2H), 7.66 (d, $J = 7.0$ Hz, 2H), 7.50 (t, $J = 7.5$ Hz, 2H), 7.43 (t, $J = 7.4$ Hz, 1H), 2.67 (s, 3H). $^{13}$C NMR (126 MHz, Chloroform-$d$) $\delta$ $^{13}$C NMR (126 MHz, Chloroform-$d$) $\delta$ 197.78, 145.81, 139.90, 135.87, 128.98, 128.93, 128.25, 127.30, 127.25, 26.70. MS (EI): $m/z$ calc’d C$_{14}$H$_{12}$O [M]$^+$ 196.09, found 196.09
**Compound 24.** Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as light yellow powder (0.337 g). \(^1\)H NMR (500 MHz, Chloroform-\(d\)) \(\delta\) 8.39 – 8.32 (m, 2H), 7.87 – 7.81 (m, 2H). \(^13\)C NMR (126 MHz, Chloroform-\(d\)) \(\delta\) 183.20, 134.15, 133.54, 127.26. **MS (EI):** \(m/z\) calc'd C\(_{14}\)H\(_8\)O\(_2\) [M]+ 208.05, found 208.05
Compound 25, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a yellow oil (0.204 g). \( ^1H \) NMR (500 MHz, Chloroform-\( d \)) \( \delta \) 7.99 (d, \( J = 8.7 \) Hz, 2H), 7.59 (d, \( J = 8.7 \) Hz, 2H), 4.42 (h, \( J = 6.1 \) Hz, 1H), 2.69 (ddd, \( J = 16.7, 9.3, 7.0 \) Hz, 1H), 2.59 (s, 3H), 2.58 – 2.52 (m, 1H), 2.39 (ddt, \( J = 15.1, 9.3, 7.4 \) Hz, 1H), 1.84 – 1.75 (m, 1H), 1.27 (d, \( J = 6.2 \) Hz, 3H). \( ^{13}C \) NMR (126 MHz, Chloroform-\( d \)) \( \delta \) 196.08, 173.39, 141.05, 132.48, 128.31, 120.98, 53.96, 30.43, 25.51, 25.40, 18.85. MS (EI): m/z calc’d C_{13}H_{15}NO_{2} [M]+ 217.1, found 217.1.
Compound 23, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a yellow oil (0.294 g). $^1$H NMR (500 MHz, Chloroform-$d$) $\delta$ 8.04 (d, $J$ = 7.8 Hz, 1H), 7.58 (t, $J$ = 7.7 Hz, 1H), 7.31 (t, $J$ = 7.7 Hz, 2H), 4.28 (t, $J$ = 6.1 Hz, 2H), 2.83 (t, $J$ = 6.2 Hz, 2H), 2.37 (s, 3H). $^{13}$C NMR (126 MHz, Chloroform-$d$) $\delta$ 194.00, 169.36, 144.00, 134.13, 127.80, 126.13, 125.63, 124.13, 39.56, 23.14. MS (EI): $m/z$ calc’d C$_{11}$H$_{11}$NO$_2$ [M]$^+$ 189.08, found 189.08
Compound 4, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.225 g). $^1$H NMR (500 MHz, Chloroform-d) $\delta$ 8.72 (d, $J = 4.5$ Hz, 1H), 8.08 (d, $J = 7.9$ Hz, 1H), 7.86 (td, $J = 7.7, 1.6$ Hz, 1H), 7.53 – 7.48 (m, 1H), 2.76 (s, 3H). $^{13}$C NMR (126 MHz, Chloroform-d) $\delta$ 200.17, 153.62, 149.01, 136.86, 127.11, 121.68, 25.82. MS (EI): $m/\epsilon$ calc’d C$_7$H$_7$NO [M]$^-$ 121.05, found 121.05
**Compound 5**, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.196g). $^1$H NMR (500 MHz, Chloroform-$d$) δ 9.21 (s, 1H), 8.82 (s, 1H), 8.26 (d, $J = 8.0$ Hz, 1H), 7.46 (dd, $J = 7.9$, 4.8 Hz, 1H), 2.67 (s, 3H). $^{13}$C NMR (126 MHz, Chloroform-$d$) δ 196.76, 153.60, 150.00, 135.47, 132.34, 123.70, 26.76. **MS (EI):** $m/z$ calc’d C$_7$H$_7$NO [M]$^+$ 121.05, found 121.05
Compound 6, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.123 g). $^1$H NMR (500 MHz, Chloroform-$d$) $\delta$ 8.67 – 8.63 (m, 2H), 7.58 (dd, $J = 4.5, 1.4$ Hz, 2H), 2.48 (s, 3H). $^{13}$C NMR (126 MHz, Chloroform-$d$) $\delta$ 197.20, 150.82, 142.56, 121.09, 26.51. MS (EI): $m/z$ calc’d C$_7$H$_7$NO [M]$^+$ 121.05, found 121.05
Compound 7, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.177 g). $^1$H NMR (500 MHz, Chloroform-$d$) $\delta$ 8.54 (d, $J = 4.4$ Hz, 1H), 8.04 – 7.98 (m, 2H), 7.61 (td, $J = 7.7$, 1.8 Hz, 1H), 7.59 – 7.54 (m, 1H), 7.47 (t, $J = 7.7$ Hz, 2H), 7.30 – 7.26 (m, 1H), 7.13 (dd, $J = 7.0$, 5.2 Hz, 1H), 3.54 (t, $J = 7.3$ Hz, 2H), 3.26 (t, $J = 7.3$ Hz, 2H). $^{13}$C NMR (126 MHz, Chloroform-$d$) $\delta$ 199.31, 160.72, 149.22, 136.89, 136.42, 133.03, 128.56, 128.10, 123.41, 121.26, 37.84, 32.06. MS (EI): $m/z$ calc’d C$_{14}$H$_{13}$NO [M]+ 211.09, found 211.1
**Compound 8**, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.194 g). \(^1\)H NMR (500 MHz, Chloroform-\(d\)) \(\delta\) 8.74 (ddd, \(J = 4.7, 1.6, 0.9\) Hz, 1H), 8.07 (ddt, \(J = 15.3, 7.8, 0.8\) Hz, 3H), 7.91 (td, \(J = 7.7, 1.7\) Hz, 1H), 7.61 (tt, \(J = 6.9, 1.3\) Hz, 1H), 7.53 – 7.48 (m, 3H). \(^{13}\)C NMR (126 MHz, Chloroform-\(d\)) \(\delta\) 193.90, 155.10, 148.57, 137.08, 136.28, 132.95, 131.00, 128.19, 126.19, 124.64. **MS (EI)**: \(m/z\) calc’d C\(_{12}\)H\(_9\)NO \([M]^+\) 183.07, found 183.07.
Compound 2, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.234 g). $^1$H NMR (500 MHz, Chloroform-d) $\delta$ 8.86 – 8.81 (m, 2H), 7.84 (dd, $J$ = 8.3, 1.2 Hz, 2H), 7.70 – 7.64 (m, 1H), 7.63 – 7.59 (m, 2H), 7.54 (t, $J$ = 7.8 Hz, 2H). $^{13}$C NMR (126 MHz, Chloroform-d) $\delta$ 195.18, 150.37, 144.40, 135.90, 133.57, 130.16, 128.68, 122.90. MS (EI): $m/z$ calc’d C$_{12}$H$_9$NO [M]$^+$ 183.06, found 183.06
**Compound 22**, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as white powder (0.11 g). 

$^1$H NMR (500 MHz, Chloroform-$d$) $\delta$ 10.01 (s, 1H), 7.85 (d, $J = 8.1$ Hz, 1H), 7.47 (d, $J = 8.2$ Hz, 1H), 7.34 (dt, $J = 30.6, 7.4$ Hz, 2H), 2.76 (s, 3H). 

$^{13}$C NMR (126 MHz, Chloroform-$d$) $\delta$ 191.79, 147.68, 143.49, 126.62, 123.89, 122.07, 112.06, 25.89. 

MS (EI): $m/z$ calc’d C$_9$H$_8$N$_2$O [M]$^+$ 160.06, found 160.06
**Compound 9.** Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.109 g). \(^1H\) NMR (500 MHz, Chloroform-d) \(\delta\) 8.83 (dd, \(J = 4.8, 1.6\) Hz, 1H), 8.04 (dd, \(J = 7.7, 1.6\) Hz, 1H), 7.34 (dd, \(J = 7.7, 4.8\) Hz, 1H), 3.34 – 3.28 (m, 2H), 2.83 – 2.77 (m, 2H). \(^{13}C\) NMR (126 MHz, Chloroform-d) \(\delta\) 204.99, 174.39, 155.76, 131.96, 130.34, 122.51, 35.79, 28.75. MS (EI): \(m/z\) calc’d C\(_8\)H\(_7\)NO [M\(^+\)] 133.05, found 133.05
2D NOESY for Compound 9
**Compound 10**, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.194 g). $^1$H NMR (500 MHz, Chloroform-d) $\delta$ 8.71 (d, $J = 4.0$ Hz, 1H), 7.67 (d, $J = 7.8$ Hz, 1H), 7.39 (dd, $J = 7.8$, 4.5 Hz, 1H), 3.04 (t, $J = 6.1$ Hz, 2H), 2.85 – 2.78 (m, 2H), 2.21 (p, $J = 6.4$ Hz, 2H). $^{13}$C NMR (126 MHz, Chloroform-d) $\delta$ 196.79, 149.15, 148.23, 140.77, 137.63, 126.98, 39.67, 29.16, 22.68. MS (EI): m/z calc’d C$_9$H$_9$NO [M]$^+$ 147.07, found 147.07
2D NOESY for Compound 10

Additional information or context related to the image is not provided in the image.
Compound 26, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.186 g). $^1$H NMR (500 MHz, Chloroform-d) $\delta$ 8.05 (dd, $J = 2.8, 1.1$ Hz, 1H), 7.55 (d, $J = 5.1$ Hz, 1H), 7.32 (dd, $J = 5.1, 2.9$ Hz, 1H), 2.54 (s, 3H). $^{13}$C NMR (126 MHz, Chloroform-d) $\delta$ 192.28, 142.64, 132.39, 126.97, 126.42, 27.56. MS (EI): $m/z$ calc'd C$_6$H$_6$OS [M]$^+$ 126.01, found 126.01
Compound 27, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as colorless oil (0.155 g). $^1$H NMR (500 MHz, Chloroform-$d$) $\delta$ 8.06 (dd, $J = 2.8$, 1.1 Hz, 1H), 7.57 (dd, $J = 5.1$, 1.0 Hz, 1H), 7.33 (dd, $J = 5.1$, 2.9 Hz, 1H), 2.89 (t, $J = 7.4$ Hz, 2H), 1.74 (p, $J = 7.4$ Hz, 2H), 1.43 – 1.29 (m, 8H), 0.91 (t, $J = 6.7$ Hz, 3H). $^{13}$C NMR (126 MHz, Chloroform-$d$) $\delta$ 195.03, 142.49, 131.67, 127.02, 126.24, 39.98, 31.72, 29.35, 29.15, 24.44, 22.64, 14.10. MS (EI): $m/z$ calc’d C$_{12}$H$_{18}$OS [M]$^+$ 210.1, found 210.1.
**Compound 28**, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as colorless oil (0.145 g). $^1$H NMR (500 MHz, Chloroform-$d$) $\delta$ 7.36 (d, $J = 5.8$ Hz, 1H), 7.26 (d, $J = 5.8$ Hz, 1H), 2.98 – 2.93 (m, 2H), 1.73 (p, $J = 7.5$ Hz, 2H), 1.41 – 1.29 (m, 8H), 0.91 (t, $J = 6.9$ Hz, 3H). $^{13}$C NMR (126 MHz, Chloroform-$d$) $\delta$ 194.26, 138.01, 127.81, 125.06, 116.39, 40.82, 30.68, 28.20, 28.12, 22.98, 21.60, 13.07. MS (EI): $m/z$ calc’d $C_{12}H_{17}BrOS$ [M]$^+$ 288.02, found 288.02
**Compound 29**, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a white solid (0.381 g). ¹H NMR (500 MHz, Chloroform-δ) δ 7.57 (d, J = 1.6 Hz, 1H), 7.36 (d, J = 1.6 Hz, 1H), 2.66 (s, 3H), 2.64 (s, 2H), 1.46 (s, 6H), 1.39 (s, 9H). ¹³C NMR (126 MHz, Chloroform-δ) δ 204.77, 204.10, 164.60, 159.42, 139.23, 129.37, 123.28, 121.77, 53.25, 38.71, 35.79, 31.15, 30.98, 30.05. **MS (EI):** m/z calc’d C₁₇H₂₂O₂ [M]⁺ 258.2, found 258.2
**Compound 30**, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a colorless oil (0.187 g). $^1$H NMR (500 MHz, Chloroform-$_d$) $\delta$ 7.95 (d, $J$ = 8.3 Hz, 2H), 7.42 (d, $J$ = 8.3 Hz, 2H), 3.82 (q, $J$ = 7.2 Hz, 1H), 3.70 (s, 3H), 3.56 (hept, $J$ = 6.8 Hz, 1H), 1.55 (d, $J$ = 7.2 Hz, 3H), 1.24 (d, $J$ = 6.8 Hz, 6H). $^{13}$C NMR (126 MHz, Chloroform-$_d$) $\delta$ 203.98, 174.33, 145.46, 135.15, 128.77, 127.82, 52.23, 45.42, 35.35, 19.16, 18.43. MS (EI): $m/z$ calc’d C$_{14}$H$_{18}$O$_3$ [M$^+$] 234.1,
**Compound 31**, Synthesized according to the general procedure for benzylic electroorganic oxidation, and isolated as a beige solid (0.409 g). $^1$H NMR (500 MHz, Chloroform-$d$) $\delta$ 8.48 (d, $J = 5.5$ Hz, 1H), 7.73 (d, $J = 1.8$ Hz, 1H), 7.67 (d, $J = 5.5$ Hz, 1H), 7.57 (s, 1H), 7.4 (dd, $J = 8.4, 1.9$ Hz, 1H), 7.17 (s, 1H), 6.89 (d, $J = 8.4$ Hz, 1H), 4.08 (s, 3H), 3.99 (s, 6H), 3.97 (s, 3H). $^{13}$C NMR (126 MHz, Chloroform-$d$) $\delta$ 194.02, 153.83, 153.76, 153.22, 151.05, 149.08, 140.11, 133.99, 129.98, 126.94, 122.91, 121.27, 111.95, 109.96, 104.86, 104.13, 56.17, 56.15, 56.12, 56.06. **MS (EI):** $m/z$ calc’d $C_{20}H_{19}NO_5$ [M]$^+$ 353.1, found 353.1