Electronic Supplementary Information (ESI)

Electrode instead of catalyst and enzyme. A greener protocol for the synthesis of new 2-hydroxyacetamides derivatives containing \( \gamma \)-lactone ring

Abbas Maleki,* Davood Nematollahi, * Fereshteh Rasouli and Azam Zeinodini-Meimand
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**Apparatus**

Cyclic voltammetry, controlled-potential coulometry and preparative electrolysis were performed using an Autolab model PGSTAT302N potentiostat/galvanostat. The working electrode used in the voltammetry experiments was a glassy carbon disc (1.8 mm$^2$ area) and platinum wire was used as a counter electrode. The working electrode used in controlled-potential coulometry and macroscale electrolysis was an assembly of four carbon rods (6 mm diameter and 4 cm length), placed as single rods in the edges of a square, and large stainless steely gauze constitutes the counter electrode. The working electrode potentials were measured versus Ag/AgCl (all electrodes from AZAR electrode). The electrolysis was performed in a simple cell (a narrow beaker type cell, 100 ml), equipped with a magnetic stirrer.

![Arrangement of the electrodes and cell](image)

**Reagens**

3,5-di-tert-butylcatechol, $n$-butylamine, $n$-propylamine, ethylamine, methylamine, benzylamine, cyclopentylamine, cyclohexylamine, cycloheptylamine, cyclooctylamine were reagent-grade materials and carbonate salts were of pro-analysis grade, from E. Merck. These chemicals were used without further purification.
**Electrochemical synthesis 9a-17a: General procedure**

**Controlled-potential method**

In a typical procedure, four carbon rods as working electrodes, a stainless steely gauze as auxiliary electrode along with an Ag/AgCl reference electrode were immersed into an undivided cell containing a mixture (60 mL) of water (carbonate buffer, \( c = 0.2 \) M, pH = 11)/acetonitrile (40/60 v/v). This mixture was pre-electrolyzed at the 0.05 V versus Ag/AgCl, then 1 mmol of 3,5-di-tert-butylcatechol and 1 mmol of 9-17 were added to the cell and the mixture was stirred until homogeneity was achieved. The electrolysis was terminated when the decay of the current became more than 95% (within about 3-4 hours). The process was interrupted during the electrolysis and the carbon anode was washed in acetone in order to reactivate it. At the end of electrolysis, after evaporation of acetonitrile, the residue was transferred to a separating funnel and extracted with cyclohexane or n-hexane. The extracted portion was recrystallized in n-hexane or chloroform. After purification, all products were characterized by: IR, \(^1\)H NMR, \(^{13}\)C NMR and MS. Moreover, product 9a was also characterized by single crystal X-ray diffraction.

**Constant-current method (Galvanostatic method)**

A mixture (60 mL) of water (carbonate buffer, \( c = 0.2 \) M, pH = 11)/acetonitrile (40/60 v/v) containing 1 mmol of 3,5-di-tert-butylcatechol and 1 mmol of 9-17 was electrolyzed in an undivided cell equipped with a carbon anode (an assembly of four rods, with 30 cm²) and a large stainless steely gauze cathode at 25 °C under a constant-current density of 1.0 mA cm⁻². The other steps are similar to those described above in the controlled-potential method.
Table S1. Electrochemical synthesis of 9a-17a at constant current conditiona

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</tr>
<tr>
<td>2</td>
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<td>54</td>
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<tr>
<td>3</td>
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<tr>
<td>9</td>
<td>17a</td>
<td>70</td>
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</table>

aGeneral procedure: 1 (1 mmol), 9-17 (1 mmol), acetonitrile (24ml), carbonat buffer (36ml), current density 1 mA cm⁻².
bYield of isolated product.

Characterization of Products

9a: N-butyl-2-(2,4-di-tert-butyl-5-oxo-2,5-dihydropyran-2-yl)-2-hydroxyacetamide

![Chemical structure of 9a](image-url)

¹H NMR (300 MHz, acetone-d₆), δ (ppm): 0.88 (t, 3H), 1.06 (s, 9H), 1.21 (s, 9H), 1.32 (m, 2H), 1.44 (m, 2H), 3.20 (m, 2H), 4.49 (d, 1H), 4.61 (d, 1H), 7.12 (NH, 1H), 7.31 (ring, 1H); ¹³C NMR (75 MHz, acetone-d₆), δ (ppm): 13.2, 19.8, 25.7, 27.4, 31.3, 31.39, 39.3, 37.7, 39.2, 73.6, 89.3, 142.7, 146.5, 169.9, 170.7; IR (KBr): 3371, 3323, 1741, 1658, 1313 cm⁻¹; MS (EI) m/z (relative intensity): 326 [M+H⁺] (5), 269 (4), 196 (95), 181 (100), 169 (45), 130 (30), 57(18).
**10a**: N-propyl-2-(2,4-di-tert-butyl-5-oxo-2,5-dihydrofuran-2-yl)-2-hydroxyacetamide

![Chemical structure of 10a](image)

$^1$H NMR (300 MHz, acetone-$d_6$), $\delta$ (ppm): 0.86 (t, 3H), 1.05 (s, 9H), 1.20 (s, 9H), 1.46 (t, 2H), 3.07 (m, CH$_2$), 4.50 (d, 1H), 4.73 (d, 1H), 7.10 (NH, 1H), 7.32 (ring, 1H); $^{13}$C NMR (75 MHz, acetone-$d_6$), $\delta$ (ppm): 10.9, 22.3, 25.6, 27.4, 31.3, 37.7, 41.2, 73.7, 89.4, 142.7, 146.57, 170.0, 170.8; IR (KBr): 3381, 3327, 1743, 1658, 1315 cm$^{-1}$; MS (EI) $m/z$ (relative intensity): 313 [M+2H$^+$] (3), 312 [M+H$^+$] (10), 196 (55), 181 (63), 169 (42), 116 (42), 57 (100), 43 (98), 41 (90).

**11a**: N-ethyl-2-(2,4-di-tert-butyl-5-oxo-2,5-dihydrofuran-2-yl)-2-hydroxyacetamide

![Chemical structure of 11a](image)

$^1$H NMR (300 MHz, acetone-$d_6$), $\delta$ (ppm): 1.07 (s, 12H), 1.21 (s, 9H), 3.19 (t, 2H), 4.50 (q, 2H), 7.13 (NH,1H), 7.31 (ring,H); $^{13}$C NMR (75 MHz, acetone-$d_6$), $\delta$ (ppm): 13.9, 25.6, 27.4, 31.3, 34.2, 37.6, 73.5, 89.2, 142.8, 146.4, 169.7, 170.7; IR (KBr): 3385, 3321, 1741, 1663, 1317 cm$^{-1}$; MS (EI) $m/z$ (relative intensity): 300 [M+3] (1), 299 [M+2] (4), 298 [M+1] (40), 296 (1), 196 (12), 181(20), 102 (8), 72 (25), 57 (100), 41 (61), 29 (75).

**12a**: N-methyl-2-(2,4-di-tert-butyl-5-oxo-2,5-dihydrofuran-2-yl)-2-hydroxyacetamide

![Chemical structure of 12a](image)
\[^1\text{H}\text{ NMR}\ (300 \text{ MHz, acetone-}d_6), \delta (\text{ppm}): 1.07 (s, 9\text{H}), 1.21 (s, 9\text{H}), 2.70 (d, \text{CH}_3), 4.47 (d, 1\text{H}), 4.60 (d, 1\text{H}), 7.10 (\text{NH, 1H}), 7.30(\text{ring, 1H}); \ ^{13}\text{C NMR}\ (75 \text{ MHz, acetone-}d_6), \delta (\text{ppm}): 25.5, 25.6, 27.4, 31.2, 37.5, 73.7, 89.3, 142.8, 146.4, 170.4, 170.7; \ IR(\text{KBr}): 3362, 3319, 1740, 1664, 1313 \text{ cm}^{-1}; \ MS (\text{EI}) m/z \ (\text{relative intensity}): 285 \ [\text{M+2}] \ (1), 284 \ [\text{M+1}] \ (6), 196 (39), 181 (65), 169 (55), 125 (32), 57 (100), 41 (54), 29 (63).\]

\textbf{13a:} \textit{N-}benzyl-2-(2,4-di-tert-butyl-5-oxo-2,5-dihydrofuran-2-yl)-2-hydroxyacetamide

\[\text{(H}_3\text{C)}_2\text{C}\]

\[\text{O}\]

\[\text{C}_{(\text{CH}_3)}\text{OH}\]

\[\text{NH}\]

\[\text{C}\text{H}_2\text{CH}\text{C}(\text{CH}_3)_3\]

\[\text{O}\]

\[\text{C}(\text{CH}_3)_3\]

\[\text{O}\]

\[^1\text{H}\text{ NMR}\ (300 \text{ MHz, acetone-}d_6), \delta (\text{ppm}): 1.01 (s, 9\text{H}), 1.22 (s, 9\text{H}), 2.97(s, 2\text{H}), 4.28 (d, 1\text{H}), 4.40 (d, 1\text{H}), 4.60 (d, 1\text{H}), 4.74 (d, 1\text{H}), 7.27 (m, 6\text{H, aromatic}), 7.61 (\text{NH, 1H}); \ ^{13}\text{C NMR}\ (75 \text{ MHz, acetone-}d_6), \delta (\text{ppm}): 170.8, 170.2, 146.5, 142.8, 138.6, 128.29, 127.8, 127.0, 89.4, 74.1, 43.20, 37.7, 31.3, 27.4, 25.7; \ IR(\text{KBr}): 3377, 3292, 3086, 2960, 2872, 1739, 1662, 1315 \text{ cm}^{-1}; \ MS (\text{EI}) m/z \ (\text{relative intensity}): 361 \ [\text{M+2}] \ (2), 360 \ [\text{M+1}] \ (12), 285 (5), 196 (40), 181 (50), 164 (30), 106 (40), 91 (100), 57 (80), 41 (55), 29 (33).\]

\textbf{14a:} \textit{N-cyclopentyl-2-(2,4-di-tert-butyl-5-oxo-2,5-dihydrofuran-2-yl)-2-hydroxyacetamide}

\[\text{(H}_3\text{C)}_2\text{C}\]

\[\text{O}\]

\[\text{C}_{(\text{CH}_3)}\text{OH}\]

\[\text{NH}\]

\[\text{C}\text{H}_2\text{CH}_{(\text{C})}\text{C}(\text{CH}_3)_3\]

\[\text{O}\]

\[^1\text{H}\text{ NMR}\ (500 \text{ MHz, CDCl}_3), \delta (\text{ppm}): 1.12 (s, 9\text{H}), 1.25 (s, 9\text{H}), 1.40 (m, 2\text{H}), 1.57 (m, 2\text{H}), 1.69 (m, 2\text{H}), 1.94 (m, 2\text{H}), 4.11(m, 2\text{H}), 4.47 (s, 1\text{H}), 5.75 (d, lactone ring, 1\text{H}), 7.13 (s, \text{NH, 1H}); \ ^{13}\text{C NMR}\ (125 \text{ MHz, CDCl}_3), \delta (\text{ppm}): 24.1, 24.1, 26.6, 28.5, 32.2, 32.9, 33.7, 38.8, 52.4, 73.0, 89.9, 144.6, 146.4, 169.8; \ IR(\text{KBr}): 1636, 1733, 2958, 3309\]
cm⁻¹; MS (EI) m/z (relative intensity): 339 [M+2] (20), 338 [M+1] (80), 337 [M] (10), 263 (18), 197 (75), 181 (90), 142 (30), 84 (45).

15a. N-cyclohexyl-2-(2,4-di-tert-butyl-5-oxo-2,5-dihydrofuran-2-yl)-2-hydroxyacetamide

1H NMR (500 MHz, CDCl₃), δ (ppm): 1.12 (s, 9H), 1.18 (m, 2H), 1.24 (s, 9H), 1.33 (m, 2H), 1.61 (q, 1H), 1.71 (q, 1H), 1.83, 1.92 (d, 1H), 3.63 (m, 1H), 4.47 (s, 1H), 5.71 (lactone ring, 1H), 7.14 (NH, 1H); 13C NMR (125 MHz, CDCl₃), δ (ppm): 171.6, 169.4, 146.5, 144.4, 89.9, 73.0, 50.0, 38.8, 33.3, 32.9, 32.2, 32.0, 28.5, 26.6, 25.7, 25.2; IR (KBr): 3439, 3320, 2959, 2937, 2857, 1753, 1649, 1551 cm⁻¹; MS (EI) m/z (relative intensity): 352 [M+1] (2), 295 (1), 277 (2), 197 (5), 196 (39), 181 (50), 83 (48), 67 (48), 57(100), 41(77).

16a. N-cycloheptyl-2-(2,4-di-tert-butyl-5-oxo-2,5-dihydrofuran-2-yl)-2-hydroxyacetamide

1H NMR (500 MHz, CDCl₃), δ (ppm): 7.14 (s, 1H), 5.78 (d, 1H), 4.45 (s, 1H), 4.14 (s, 1H), 3.81 (m, 1H), 1.88 (m, 2H), 1.61 (m, 4H), 1.47 (m, 2H), 1.43 (m, 4H), 1.39 (s, 9H), 1.11 (s, 9H); 13C NMR (125 MHz, CDCl₃), δ (ppm): 171.6, 169.1, 146.5, 144.4, 89.9, 73.1, 52.2, 38.8, 35.3, 35.0, 32.2, 28.5, 28.4, 28.4, 26.6, 24.4, 24.4; IR (KBr): 3310, 2957,
2932, 2866, 1736, 1632 cm⁻¹; MS (EI) m/z (relative intensity): 367 [M+2] (1), 366 [M+1] (2), 269 (0.5), 196 (2), 181 (2), 57 (100), 41 (17).

17a: N-cyclooctyl-2-(2,4-di-tert-butyl-5-oxo-2,5-dihydrofuran-2-yl)-2-hydroxyacetamide

\[
\begin{align*}
\text{NH} & \\
& \\
O & \text{C} \\
& \text{CH-OH} \\
& \text{C(CH}_3)_3 \\
& \text{C} \\
\end{align*}
\]

\(^1\)H NMR (500 MHz, CDCl₃), δ (ppm): 7.14 (s, 1H), 5.77 (d, 1H), 4.44 (d, 1H), 4.14 (s, 1H), 3.87 (t, 1H), 1.77 (m, 2H), 1.65 (t, 2H), 1.55 (m, 10H), 1.24 (s, 9H), 1.12 (s, 9H); \(^13\)C NMR (125 MHz, CDCl₃), δ (ppm): 171.6, 169.1, 146.5, 144.4, 89.9, 73.0, 51.2, 38.9, 32.6, 32.3, 32.2, 28.5, 27.4, 27.3, 26.6, 25.9, 24.0; IR (KBr): 3316, 2926, 1737, 1633, 1533 cm⁻¹; MS (EI) m/z (relative intensity): 382 [M+3] (1), 380 [M+1] (6), 364 (2), 323 (6), 290 (10), 226 (12), 195 (100), 184 (50), 181 (85), 169 (80), 125 (40), 83 (53), 69 (55), 56 (98), 41 (42).
FTIR, $^1$H NMR, $^{13}$C NMR and mass spectra

FTIR of 9a
$^1$H NMR of 9a
Expanded $^1$H NMR of 9a
$^{13}$C NMR of 9a
Expanded $^{13}$C NMR of 9a
MS of 9a
FTIR of 10a
$^1$H NMR of 10a
Expanded $^1$H NMR of 10a
$^{13}$C NMR of 10a
Expanded $^{13}$C NMR of 10a
MS of 10a
IR of 11a
$^1$H NMR of 11a
Expanded $^1$H NMR of 11a
$^{13}$C NMR of 11a
Expanded $^{13}$C NMR of 11a
MS of 11a
IR of 12a
\(^1\text{H} \text{NMR of 12a}\)
Expanded $^1$H NMR of 12a
$^{13}$C NMR of 12a
Expanded $^{13}$C NMR of 12a
MS of 12a
IR of 13a
$^1$H NMR of 13a
Expanded $^1$H NMR of 13a
$^{13}$C NMR of 13a
Expanded $^{13}$C NMR of 13a
MS of 13a
IR of 14a
$^1$H NMR of 14a
Expanded 1H NMR of 14a

TBCyda 1H NMR in CDCl3 at 298 K 89/6/13

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POLYNOD 1

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13C NMR of 14a
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MS of 15a
IR of 16a
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Expanded $^1$H NMR of 17a
$^{13}$C NMR of 17a
Expanded $^{13}$C NMR of 17a
VI. Crystallography of 9a

Table S2. Selected bond lengths (Å) and bond Angles (°) of compound 9a

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### Table S3: Crystal data and structure refinement for compound 9a

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<td>Largest diff. peak and hole (e Å&lt;sup&gt;-3&lt;/sup&gt;)</td>
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Figure S1. X-ray crystal structure of compound 9a.
Figure S2. X-ray crystal structure of compound 9a. The hydrogen atoms are omitted for the reason of clarity.