Electronic Supplementary Information

Gallium-Catalyzed Reductive Lactonization of Keto Acids using a Hydrosilane

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General
All reactions were carried out under a N\textsubscript{2} atmosphere, unless otherwise noted. Benzene and toluene were distilled from a Na/benzophenone ketyl. All gallium compounds and other metal compounds were commercially available and were used without further purification. Hydrosilanes were used without further purification. 3-Benzoylpropionic acid (1a) is commercial available and purified by recrystallization prior to use. Reactions were monitored by TLC analysis of reaction aliquots. Column chromatography was performed using a silica gel. \textsuperscript{1}H NMR spectra were measured at 500 (or 300) MHz using tetramethylsilane as an internal standard (0.00 ppm). \textsuperscript{13}C NMR spectra were measured at 125 (or 75) MHz using the center peak of chloroform (77.0 ppm). High-resolution mass spectra (HRMS) were measured using NBA (3-nitrobenzylalcohol) as a matrix.

Synthesis of keto acid derivatives
Keto acid derivatives were synthesized by either Method A or Method B described in the corresponding literatures, except for 1a.

Method A: To a solution of succinic anhydride (10 mmol) and AlCl\textsubscript{3} (22 mmol, 2.9 g) in CH\textsubscript{2}Cl\textsubscript{2} (20 mL) was added aromatic hydrocarbon derivatives (11 mmol). The reaction mixture was stirred at room temperature over 6 h. After the reaction, the mixture was quenched with an aqueous solution of 1N HCl (10 mL). The aqueous layer was extracted with CH\textsubscript{2}Cl\textsubscript{2} (15 mL x 3) and the organic phase was combined, dried with anhydrous Na\textsubscript{2}SO\textsubscript{4} and evaporated under reduced pressure. The crude product was purified by recrystallization (hexane/CH\textsubscript{2}Cl\textsubscript{2} or toluene) to give the corresponding keto acid.

Method B: To a solution of Mg (13.0 mmol, 316 mg) in THF (5 mL) was added dropwise a solution of aryl bromide derivatives (12 mmol) and THF (15 mL) for 24 h. The mixture was then added to a solution of succinic anhydride (10 mmol) in THF (10 mL) at -90 °C. The mixture was stirred at room temperature over 6 h. After the reaction, the mixture was quenched with an aqueous solution of 1N HCl (10 mL). The aqueous layer was extracted with Et\textsubscript{2}O (15 mL x 3) and the organic phase was combined, dried with anhydrous Na\textsubscript{2}SO\textsubscript{4} and evaporated under reduced pressure. The crude product was purified by recrystallization (hexane/CH\textsubscript{2}Cl\textsubscript{2} or toluene) to give the corresponding keto acids.

3-(2-Methylbenzoyl)propionic acid (1b)

The preparation is reported in our previous report, and the spectra data are in agreement with the literature. 

S2
3-(3-Methylbenzoyl)propionic acid\(^1\) (1c)

\[
\begin{align*}
\text{Method B; a white solid: } & \quad ^1\text{H NMR (CDCl}_3, 500 \text{ MHz}) \delta 2.41 (\text{s, 3 H, CH}_3), 2.81 (t, J = 6.5 \text{ Hz, 2 H, CH}_2), 3.30 (t, J = 6.5 \text{ Hz, 2 H, CH}_2), 7.34 - 7.40 (m, 2 H, ArH), 7.77 - 7.79 (m, 2 H, ArH); \quad ^{13}\text{C NMR (CDCl}_3, 125 \text{ MHz}) \delta 21.3, 28.0, 33.2, 125.2, 128.5, 128.6, 134.1, 136.4, 138.4, 178.8, 198.1; \quad \text{MS (EI) } m/z \quad (%) 192 (M^+ , 22), 119 (100). \end{align*}
\]

3-(4-Methylbenzoyl)propionic acid\(^1\) (1d)

\[
\begin{align*}
\text{Method A; a white solid: } & \quad ^1\text{H NMR (CDCl}_3, 300 \text{ MHz}) \delta 2.41 (\text{s, 3 H, CH}_3), 2.80 (t, J = 6.5 \text{ Hz, 2 H, CH}_2), 3.29 (t, J = 6.5 \text{ Hz, 2 H, CH}_2), 7.26 (d, J = 7.4 \text{ Hz, 2 H, ArH}), 7.87 (d, J = 7.4 \text{ Hz, 2 H, ArH}); \quad ^{13}\text{C NMR (CDCl}_3, 75 \text{ MHz}) \delta 21.6, 28.0, 33.0, 128.1, 129.3, 133.9, 144.2, 178.6, 197.5; \quad \text{MS (EI) } m/z \quad (%) 192 (M^+ , 17), 119 (100). \end{align*}
\]

3-(2, 5-Dimethylbenzoyl)propionic acid\(^1\) (1e)

\[
\begin{align*}
\text{Method A; a white solid: } & \quad ^1\text{H NMR (CDCl}_3, 500 \text{ MHz}) \delta 2.37 (\text{s, 3 H, CH}_3), 2.45 (s, 3 H, CH}_3), 2.79 (t, J = 6.5 \text{ Hz, 2 H, CH}_2), 3.22 (t, J = 6.5 \text{ Hz, 2 H, CH}_2), 7.13 (d, J = 7.5 \text{ Hz, 1 H, ArH}), 7.20 (d, J = 7.5 \text{ Hz, 1 H, ArH}), 7.50 (s, 1 H, ArH); \quad ^{13}\text{C NMR (CDCl}_3, 125 \text{ MHz}) \delta 20.9, 20.9, 28.2, 35.8, 129.2, 131.9, 132.3, 135.2, 137.1, 178.5, 201.7; \quad \text{MS (EI) } m/z \quad (%) 206 (M^+ , 50), 133 (100). \end{align*}
\]

3-(5,6,7,8-Tetrahydro-2-naphthoyl)propionic acid\(^1\) (1f)

\[
\begin{align*}
\text{The preparation is reported in our previous report, and the spectra data are in agreement with the literature.} \quad ^1\end{align*}
\]
3-(4-Methoxybenzoyl)propionic acid\(^1\) (1g)

Method A; a white solid: \(^1\)H NMR (CDCl\(_3\), 500 MHz) \(\delta 2.80 (t, J = 6.5 \text{ Hz}, 2 \text{ H, } CH_2), 3.28 (t, J = 6.5 \text{ Hz}, 2 \text{ H, } CH_2), 3.88 (s, 3 \text{ H, } CH_3), 6.94 (d, J = 9.0 \text{ Hz}, 2 \text{ H, ArH}), 7.97 (d, J = 9.0 \text{ Hz}, 2 \text{ H, ArH}); ^{13}\text{C NMR (CDCl}_3\text{, 125 MHz) }\delta 28.1, 32.8, 55.5, 113.8, 129.5, 130.3, 163.7, 178.2, 196.4; \text{ MS (EI) } m/z (\%) 208 (M^+ , 9), 135 (100).

3-(4-Phenoxybenzoyl)propionic acid\(^1\) (1h)

The preparation is reported in our previous report, and spectra data are in agreement with the literature.\(^1\)

3-(3-Methoxybenzoyl)propionic acid\(^2\) (1i)

Method B; a white solid: mp 108–109 °C; \(^1\)H NMR (CDCl\(_3\), 500 MHz) \(\delta 2.81 (t, J = 6.5 \text{ Hz}, 2 \text{ H, } CH_2), 3.30 (t, J = 6.5 \text{ Hz}, 2 \text{ H, } CH_2), 3.85 (s, 3 \text{ H, } CH_3), 7.12 (d, J = 8.0 \text{ Hz}, 1 \text{ H, ArH}), 7.38 (d, J = 8.0 \text{ Hz}, 7.5 \text{ Hz}, 1 \text{ H, ArH}), 7.50 (s, 1 \text{ H, ArH}), 7.56 (d, J = 7.5 \text{ Hz}, 1 \text{ H, ArH}); ^{13}\text{C NMR (CDCl}_3\text{, 125 MHz) }\delta 28.0, 33.2, 55.4, 112.2, 119.9, 120.7, 129.6, 137.7, 159.8, 178.8, 197.6; \text{ IR (ATR, cm}^{-1}) \ 1686 \text{ s; MS (EI) } m/z (\%) 208 (M^+ , 21), 135 (100); \text{ HRMS (FAB-Magnetic Sector): calcd. for } C_{11}H_{13}O_4[M+H]^+ : 209.080, \text{ found: 209.0818.}

3-(4-Fluorobenzoyl)propionic acid\(^1\) (1j)

Method A; a white solid: \(^1\)H NMR (CDCl\(_3\), 300 MHz) \(\delta 2.82 (t, J = 6.5 \text{ Hz}, 2 \text{ H, } CH_2), 3.29 (t, J = 6.5 \text{ Hz}, 2 \text{ H, } CH_2), 7.14 (dd, J = 8.3 \text{ Hz}, 8.3 \text{ Hz}, 2 \text{ H, ArH}), 8.01 (dd, J = 8.3 \text{ Hz}, 5.7 \text{ Hz}, 2 \text{ H, ArH}); ^{13}\text{C NMR (CDCl}_3\text{, 75 MHz) }\delta 28.0, 33.0, 115.8 (J_{C,F} = 22.6 \text{ Hz}), 130.7 (J_{C,F} = 8.8 \text{ Hz}), 132.8 (J_{C,F} = 2.5 \text{ Hz}), 165.9 (J_{C,F} = 255.3 \text{ Hz}), 178.7, 196.2; \text{ MS (EI) } m/z (\%) 196 (M^+ , 9), 123 (100).
3-(4-Chlorobenzoyl)propionic acid<sup>1</sup> (1k)

![Structural formula of 3-(4-Chlorobenzoyl)propionic acid](image)

Method A; a white solid: <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 2.81 (t, J = 6.5 Hz, 2 H, CH₂), 3.28 (t, J = 6.5 Hz, 2 H, CH₂), 7.45 (d, J = 8.0 Hz, 2 H, ArH), 7.92 (d, J = 8.0 Hz, 2 H, ArH); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ 27.9, 33.1, 129.0, 129.5, 134.7, 139.8, 178.5, 196.6; MS (EI) m/z (%) 214 (M<sup>+</sup>+2, 2), 212 (M<sup>+</sup>, 7), 139 (100).

3-(4-Bromobenzoyl)propionic acid<sup>1</sup> (1l)

![Structural formula of 3-(4-Bromobenzoyl)propionic acid](image)

Method A; a white solid: <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 2.81 (t, J = 6.5 Hz, 2 H, CH₂), 3.27 (t, J = 6.5 Hz, 2 H, CH₂), 7.62 (d, J = 8.3 Hz, 2 H, ArH), 7.84 (d, J = 8.3 Hz, 2 H, ArH); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 27.9, 33.1, 128.5, 129.5, 132.0, 135.1, 178.5, 196.8; MS (EI) m/z (%) 258 (M<sup>+</sup>+2, 8), 256 (M<sup>+</sup>, 8), 183 (100).

3-(4-Trifluoromethylbenzoyl)propionic acid<sup>3</sup> (1m)

![Structural formula of 3-(4-Trifluoromethylbenzoyl)propionic acid](image)

Method B; a white solid: mp 129–130 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 2.85 (t, J = 6.5 Hz, 2 H, CH₂), 3.33 (t, J = 6.5 Hz, 2 H, CH₂), 7.75 (d, J = 8.0 Hz, 2 H, ArH), 8.09 (d, J = 8.0 Hz, 2 H, ArH); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ 27.8, 33.4, 123.5 (J<sub>CF</sub> = 272.9 Hz), 125.7 (J<sub>CF</sub> = 3.8 Hz), 128.4, 134.6 (J<sub>CF</sub> = 32.7 Hz), 139.0 (J<sub>CF</sub> = 1.3 Hz), 178.5, 196.9; IR (ATR, cm<sup>−1</sup>) 1715 m, 1688 s; MS (FAB) m/z (%) 247 (M+H<sup>+</sup>); HRMS (FAB-Magnetic Sector): calcd. for C<sub>11</sub>H<sub>10</sub>F<sub>3</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 247.0577, found: 247.0592.

4-([1,1'-Biphenyl]-4-yl)-4-oxobutanoic acid<sup>1</sup> (1n)

![Structural formula of 4-([1,1'-Biphenyl]-4-yl)-4-oxobutanoic acid](image)

The preparation is reported in our previous report, and spectra data are in agreement with the literature.<sup>1</sup>
4-(1-Naphthyl)-4-oxobutanoic acid\(^1\) (1o)

![Structure](image)

Method B; a white solid: \(^1\)H NMR (CDCl\(_3\), 300 MHz) \(\delta 2.90\) (t, \(J = 6.5\) Hz, 2 H, CH\(_2\)), \(3.40\) (t, \(J = 6.5\) Hz, 2 H, CH\(_2\)), 7.50-7.60 (m, 3 H, ArH), 7.88 (d, \(J = 8.5\) Hz, 1 H, ArH), 7.95 (d, \(J = 8.5\) Hz, 1 H, ArH), 8.00 (d, \(J = 8.5\) Hz, 1 H, ArH); \(^{13}\)C NMR (CDCl\(_3\), 75 MHz) \(\delta 28.4, 36.3, 124.3, 125.8, 126.5, 127.7, 128.0, 128.4, 130.1, 132.9, 133.9, 135.2, 178.9, 201.8\); MS (EI) m/z (\%): 228 (M\(^+\), 74), 155 (100).

4-(2-Thienyl)-4-oxobutanoic acid\(^4\) (1p)

![Structure](image)

Method A; a green solid: \(^1\)H NMR (CDCl\(_3\), 500 MHz) \(\delta 2.81\) (t, \(J = 6.5\) Hz, 2 H, CH\(_2\)), \(3.26\) (t, \(J = 6.5\) Hz, 2 H, CH\(_2\)), 7.14 (t, \(J = 4.5\) Hz, 1 H, ArH), 7.65 (d, \(J = 4.5\) Hz, 1 H, ArH), 7.77 (d, \(J = 4.5\) Hz, 1 H, ArH); \(^{13}\)C NMR (CDCl\(_3\), 125 MHz) \(\delta 28.0, 33.6, 128.1, 132.1, 133.8, 143.4, 178.5, 190.7\); MS (EI) m/z (\%): 184 (M\(^+\), 14), 110 (100); HRMS (EI-Quadrupole): calcd. for C\(_8\)H\(_8\)O\(_3\)S [M\(^+\)]: 184.0194, found: 184.0177.

\(\gamma\)-Oxo-benzenehexanoic acid\(^5\) (3)

![Structure](image)

Method B; a white solid: mp 90–91 °C; \(^1\)H NMR (CDCl\(_3\), 500 MHz) \(\delta 2.63\) (t, \(J = 6.5\) Hz, 2 H, CH\(_2\)), 2.70 (t, \(J = 6.5\) Hz, 2 H, CH\(_2\)), 2.79 (t, \(J = 7.5\) Hz, 2 H, CH\(_2\)), 2.92 (t, \(J = 7.5\) Hz, 2 H, CH\(_2\)), 7.17-7.21 (m, 3 H, ArH), 7.26-7.29 (m, 2 H, ArH); \(^{13}\)C NMR (CDCl\(_3\), 125 MHz) \(\delta 27.7, 29.6, 36.9, 44.1, 126.1, 128.3, 128.5, 140.8, 178.5, 207.8\); IR (ATR, cm\(^{-1}\)) 1703 w; MS (EI) m/z (\%): 206 (M\(^+\), 30), 91 (100); HRMS (EI-Quadrupole): calcd. for C\(_{12}\)H\(_{14}\)O\(_3\) [M\(^+\)]: 206.0943, found: 206.0948.

2-Benzoylbenzoic acid\(^1\) (5)

![Structure](image)

The preparation is reported in our previous report, and the spectra data are in agreement with the literature.\(^1\)
5-Oxo-5-phenylpentanoic acid\(^6\) (7)

![Structure](image)

Method B; a white solid: \(^1\)H NMR (CDCl\(_3\), 500 MHz) \(\delta\) 2.09 (quint, \(J = 7.0\) Hz, 2 H, CH\(_2\)), 2.51 (t, \(J = 7.0\) Hz, 2 H, CH\(_2\)), 3.08 (t, \(J = 7.0\) Hz, 2 H, CH\(_2\)), 7.46 (t, \(J = 7.0\) Hz, 1 H, ArH), 7.56 (d, \(J = 7.0\) Hz, 1 H, ArH); \(^{13}\)C NMR (CDCl\(_3\), 125 MHz) \(\delta\) 18.9, 33.0, 37.3, 128.0, 128.6, 133.1, 136.7, 179.4, 199.3; MS (EI) m/z (\%) 192 (M\(^+\), 7), 105 (100).

General procedure for the synthesis of the \(\gamma\)-lactone derivatives: To a screw-capped tube, GaCl\(_3\) (0.010 mmol, 8.8 mg), benzene (2 mL), a keto acid (1.0 mmol), and PhSiH\(_3\) (1.00 mmol, 108 mg) were successively added. After the tube was sealed with a cap that contained a PTFE septum, the mixture was heated at 60 °C for the reaction time shown in Table 2. After the reaction, H\(_2\)O (2 mL) was added to the reaction mixture, which of the organic layer was extracted with EtOAc (5 mL x 3). The combined organic phases were evaporated under reduced pressure. To remove the siloxane residue, MeOH (15 mL) was rinsed to the crude material. The formed precipitate was filtered, and the filtrate was concentrated under reduced pressure. The crude product was purified by a silica gel column chromatography (hexane/EtOAc) to afford the lactone derivative.

5-Phenyl-dihydro-furan-2-one\(^7\) (2a)

![Structure](image)

General procedure was followed with 3-benzoylpropionic acid (1a, 178.4 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded 2a as a colorless oil (152.8 mg, 94%): \(^1\)H NMR (CDCl\(_3\), 300 MHz) \(\delta\) 2.12-2.28 (m, 1 H, CH), 2.62-2.73 (m, 3 H, CH\(_2\), CH\(_2\)), 5.49-5.54 (m, 1 H, CH), 7.32-7.42 (m, 5 H, ArH); \(^{13}\)C NMR (CDCl\(_3\), 75 MHz) \(\delta\) 28.9, 30.9, 81.2, 125.2, 128.4, 128.7, 139.4, 176.9; IR (ATR, cm\(^{-1}\)) 1772 s; MS (EI) m/z (%) 162 (M\(^+\), 100); HRMS (EI-Quadrupole): calcd. for C\(_{10}\)H\(_{10}\)O\(_2\)[M\(^+\)]: 162.0681, found: 162.0685.

5-o-Tolyldihydro-furan-2-one\(^7\) (2b)

![Structure](image)

General procedure was followed with 3-(2-methylbenzoyl)propionic acid (1b, 192.6 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded 2b as a colorless oil (151.8 mg, 86%): \(^1\)H
NMR (CDCl₃, 500 MHz) δ 2.06-2.14 (m, 1 H, CH₂), 2.34 (s, 3 H, CH₃), 2.64-2.71 (m, 3 H, CH₂, CH₂), 5.69-5.72 (m, 1 H, CH), 7.18 (dd, J = 4.5 Hz, 4.5 Hz, 1 H, ArH), 7.23-7.25 (m, 2 H, ArH), 7.35 (dd, J = 5.0 Hz, 4.5 Hz, 1 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 18.9, 28.6, 29.5, 78.8, 124.1, 126.4, 128.1, 130.7, 134.1, 137.5, 177.1; IR (ATR, cm⁻¹) 1774 s; MS (EI) m/z (%): 176 (M⁺, 100); HRMS (EI-Quadrupole): calcd. for C₁₁H₁₂O₂ [M⁺]: 176.0837, found: 176.0844.

5-m-Tolyl-dihydro-furan-2-one⁷ (2c)

General procedure was followed with 3-(3-methylbenzoyl)propionic acid (1c, 192.4 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded 2c as a colorless oil (166.7 mg, 95%): ¹H NMR (CDCl₃, 500 MHz) δ 2.13-2.23 (m, 1 H, CH₂), 2.36 (s, 3 H, CH₃), 2.60-2.67 (m, 3 H, CH₂, CH₂), 5.46-5.49 (m, 1 H, CH), 7.11-7.15 (m, 3 H, ArH), 7.25-7.29 (m, 1 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 21.3, 28.9, 30.9, 81.2, 122.3, 125.8, 128.6, 129.1, 138.5, 139.3, 176.9; IR (ATR, cm⁻¹) 1774 s; MS (EI) m/z (%): 176 (M⁺, 100); HRMS (EI-Quadrupole): calcd. for C₁₁H₁₂O₂ [M⁺]: 176.0837, found: 176.0839.

5-p-Tolyl-dihydro-furan-2-one⁷ (2d)

General procedure was followed with 3-(4-methylbenzoyl)propionic acid (1d, 192.0 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded 2d as a white solid (157.7 mg, 90%): mp 70-71 °C; ¹H NMR (CDCl₃, 500 MHz) δ 2.15-2.21 (m, 1 H, CH₂), 2.36 (s, 3 H, CH₃), 2.61-2.66 (m, 3 H, CH₂, CH₂), 5.47-5.50 (m, 1 H, CH), 7.19-7.24 (m, 4 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 21.1, 29.0, 30.9, 81.3, 125.3, 129.3, 136.2, 138.3, 177.0; IR (ATR, cm⁻¹) 1764 s; MS (EI) m/z (%) 176 (M⁺, 87), 121 (100); HRMS (EI-Quadrupole): calcd. for C₁₁H₁₂O₂ [M⁺]: 176.0837, found: 176.0839.

5-(2, 5-Dimethylphenyl)-dihydro-furan-2-one⁸ (2e)

General procedure was followed with 3-(2,5-dimethylbenzoyl)propionic acid (1e, 206.7 mg) for 24
h. Column chromatography (7/3 = hexane/EtOAc) afforded 2e as a pale yellow solid (147.4 mg, 78%): mp 76–77 °C; ^1^H NMR (CDCl₃, 500 MHz) δ 2.07-2.12 (m, 1 H, CH₂), 2.28 (s, 3 H, CH₃), 2.32 (s, 3 H, CH₃), 2.64-2.66 (m, 3 H, CH₂, CH₂), 5.67-5.69 (m, 1 H, CH), 7.03-7.08 (m, 2 H, ArH), 7.16 (s, 1 H, ArH); ^1^C NMR (CDCl₃, 125 MHz) δ 18.4, 21.0, 28.6, 29.6, 78.9, 124.7, 128.7, 130.6, 130.8, 135.9, 137.3, 177.1; IR (ATR, cm⁻¹) 1768 s; MS (EI) m/z (%) 190 (M⁺, 100); HRMS (EI-Quadrupole): calcd. for C₁₂H₁₄O₂ [M⁺]: 190.0994, found: 199.0990.

5-(5,6,7,8-Tetrahydronaphthalen-2-yl)dihydrofuran-2(3H)-one (2f)

General procedure was followed with 4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid (1f, 232.3 mg) for 24 h. Column chromatography (8/2 = hexane/EtOAc) afforded 2f as a pale yellow oil (210.4 mg, 97%): ^1^H NMR (CDCl₃, 500 MHz) δ 1.78-1.81 (m, 4 H, CH₂), 2.15-2.22 (m, 1 H, CH₂), 2.58-2.66 (m, 3 H, CH₂), 2.76 (m, 4 H, CH₂), 5.43-5.46 (m, 1 H, CH), 7.03-7.04 (m, 2 H, ArH), 7.07-7.09 (m, 1 H, ArH); ^1^C NMR (CDCl₃, 125 MHz) δ 23.0, 29.0, 29.1, 29.4, 30.9, 81.4, 122.4, 126.0, 129.5, 136.3, 137.6, 177.1; IR (ATR, cm⁻¹) 1774 s; MS (EI) m/z (%) 216 (M⁺, 100); HRMS (EI-Quadrupole): calcd. for C₁₄H₁₆O₂ [M⁺]: 216.1150, found: 216.1137.

5-(4-Phenoxypyphenyl)dihydrofuran-2-one (2h)

General procedure was followed with 3-(4-phenoxypybenzoyl)propionic acid (1h, 270.1 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded 2h as a white solid (182.0 mg, 72%): mp 60–62 °C; ^1^H NMR (CDCl₃, 500 MHz) δ 2.16-2.25 (m, 1 H, CH₂), 2.60-2.68 (m, 3 H, CH₂, CH₂), 5.46-5.49 (m, 1 H, CH), 7.00-7.02 (m, 4 H, ArH), 7.12 (t, J = 7.5 Hz, 1 H, ArH), 7.29-7.36 (m, 4 H, ArH); ^1^C NMR (CDCl₃, 125 MHz) δ 29.1, 30.9, 81.0, 118.7, 119.1, 123.6, 127.0, 129.8, 133.7, 156.7, 157.5, 176.7; IR (ATR, cm⁻¹) 1753 s; MS (EI) m/z (%) 254 (M⁺, 100); HRMS (EI-Quadrupole): calcd. for C₁₆H₁₄O₃ [M⁺]: 254.0943, found: 254.0949.

5-(3-Methoxyphenyl)dihydrofuran-2-one (2i)

General procedure was followed with 3-(3-methoxybenzoyl)propionic acid (1i, 110.6 mg) for 24 h.
Column chromatography (8/2 = hexane/EtOAc) afforded 2i as a colorless oil (160.5 mg, 84%): \(^1\)H NMR (CDCl\(_3\), 500 MHz) \(\delta\) 2.14-2.22 (m, 1 H, CH\(_2\)), 2.62-2.68 (m, 3 H, CH\(_2\), CH\(_2\)), 3.81 (s, 3 H, OCH\(_3\)), 5.47-5.50 (m, 1 H, CH), 6.86-6.91 (m, 3 H, ArH); \(^13\)C NMR (CDCl\(_3\), 125 MHz) \(\delta\) 28.8, 30.8, 55.2, 80.9, 110.7, 113.7, 117.3, 129.8, 141.0, 159.8, 176.8; IR (ATR, cm\(^{-1}\)) 1774 s; MS (EI) \(m/z\) 192 (M\(^+\), 100); HRMS (FAB-Magnetic Sector): calcd. for C\(_{11}\)H\(_{13}\)O\(_3\) [M+H]\(^+\): 193.0859, found: 193.0864.

5-(4-Fluorophenyl)-dihydro-furan-2-one\(^7\) (2j)

General procedure was followed with 3-(4-fluorobenzoyl)propionic acid (1j, 192.6 mg) for 7 days. Column chromatography (7/3 hexane/EtOAc) afforded 2j as a pale yellow oil (136.2 mg, 76%): \(^1\)H NMR (CDCl\(_3\), 500 MHz) \(\delta\) 2.14-2.22 (m, 1 H, CH\(_2\)), 2.62-2.68 (m, 3 H, CH\(_2\), CH\(_2\)), 5.47-5.50 (m, 1 H, CH), 7.08 (t, \(J = 8.5\) Hz, 2 H, ArH), 7.32 (dd, \(J = 8.5\) Hz, 5.5 Hz, 2 H, ArH); \(^13\)C NMR (CDCl\(_3\), 125 MHz) \(\delta\) 29.0, 31.0, 80.6, 115.7 \((J_{C,F} = 21.1\) Hz), 127.2 \((J_{C,F} = 8.6\) Hz), 135.0 \((J_{C,F} = 3.8\) Hz), 162.6 \((J_{C,F} = 246.6\) Hz), 176.6; IR (ATR, cm\(^{-1}\)) 1773 s; MS (EI) \(m/z\) 180 (M\(^+\), 95), 125 (100); HRMS (EI-Quadrupole): calcd. for C\(_{10}\)H\(_8\)O\(_2\)F [M\(^+\)]: 180.0587, found: 180.0587.

5-(4-Chlorophenyl)-dihydro-furan-2-one\(^7\) (2k)

General procedure was followed with 3-(4-chlorobenzoyl)propionic acid (1k, 212.8 mg) for 7 days. Column chromatography (7/3 = hexane/EtOAc) afforded 2k as a colorless oil (123.2 mg, 63%): \(^1\)H NMR (CDCl\(_3\), 500 MHz) \(\delta\) 2.13-2.18 (m, 1 H, CH\(_2\)), 2.64-2.69 (m, 3 H, CH\(_2\), CH\(_2\)), 5.46-5.49 (m, 1 H, CH), 7.28 \((d, J = 9.0\) Hz, 2 H, ArH), 7.36 \((d, J = 9.0\) Hz, 2 H, ArH); \(^13\)C NMR (CDCl\(_3\), 125 MHz) \(\delta\) 28.8, 30.9, 80.4, 126.6, 128.9, 134.2, 137.8, 176.5; IR (ATR, cm\(^{-1}\)) 1776 s; MS (EI) \(m/z\) (%) 198 (M\(^+\)+2, 17), 196 (M\(^+\), 56), 58 (100); HRMS (EI-Quadrupole): calcd. for C\(_{10}\)H\(_8\)O\(_2\)Cl [M\(^+\)]: 196.0291, found: 196.0295.

5-(4-Bromophenyl)-dihydro-furan-2-one\(^7\) (2l)

General procedure was followed with 3-(4-bromobenzoyl)propionic acid (1l, 161.4 mg) for 7 days.
Column chromatography (7/3 = hexane/EtOAc) afforded 2l as a white solid (161.4 mg, 67%): mp 81–82 °C; 1H NMR (CDCl₃, 500 MHz) δ 2.13-2.18 (m, 1 H, CH₂), 2.64-2.69 (m, 3 H, CH₃, CH₂), 5.45-5.48 (m, 1 H, CH), 7.22 (d, J = 9.0 Hz, 2 H, ArH), 7.52 (d, J = 9.0 Hz, 2 H, ArH); 13C NMR (CDCl₃, 125 MHz) δ 28.8, 30.9, 80.4, 122.4, 126.9, 131.9, 138.4, 176.5; IR (ATR, cm⁻¹) 1760 s; MS (EI) m/z (%): 238 (M⁺+2, 32), 240 (M⁺, 33) 58 (100); HRMS (EI-Quadrupole): calcd. for C₁₀H₉O₂Br [M+2]⁺: 241.9765, found: 241.9757.

5-(4-Trifluoromethylphenyl)-dihydro-furan-2-one⁹ (2m)

General procedure was followed with 3-(4-trifluoromethylbenzoyl)propionic acid (1m, 246.2 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded 2m as a pale orange oil (144.6 mg, 63%): 1H NMR (CDCl₃, 500 MHz) δ 2.13-2.21 (m, 1 H, CH₂), 2.62-2.77 (m, 3 H, CH₃, CH₂), 5.55-5.58 (m, 1 H, CH), 7.47 (d, J = 8.0 Hz, 2 H, ArH), 7.66 (d, J = 8.0 Hz, 2 H, ArH); 13C NMR (CDCl₃, 125 MHz) δ 28.6, 30.9, 80.1, 123.8 (J_C-F = 272.0 Hz), 125.4, 125.7 (J_C-F = 3.9 Hz), 130.6 (J_C-F = 32.6 Hz), 143.4, 176.4; IR (ATR) 1782 s; MS (EI) m/z (%) 230 (M⁺, 52), 58 (100); HRMS (FAB-Magnetic Sector): calcd. for C₁₁H₁₀O₂F₃ [M+H]⁺: 231.0627, found: 231.0633.

5-(1, 1'-Biphenyl)-4-yl-dihydro-furan-2-one¹⁰ (2n)

General procedure was followed with 4-([1,1'-biphenyl]-4-yl)-4-oxobutanoic acid (1n, 254.6 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded 2n as a pale yellow solid (171.0 mg, 72%): mp 103–105 °C; 1H NMR (CDCl₃, 500 MHz) δ 2.19-2.29 (m, 1 H, CH₂), 2.66-2.72 (m, 3 H, CH₃, CH₂), 5.54-5.57 (m, 1 H, CH), 7.35-7.46 (m, 5 H, ArH), 7.58-7.62 (m, 2 H, ArH); 13C NMR (CDCl₃, 125 MHz) δ 29.0, 30.9, 81.0, 125.8, 127.1, 127.5, 127.5, 128.8, 138.3, 140.4, 141.4, 176.8; IR (ATR, cm⁻¹) 1766 s; MS (EI) m/z (%) 238 (M⁺, 100); HRMS (EI-Quadrupole): calcd. for C₁₆H₁₄O₂ [M⁺]: 238.0994, found: 238.1000.

5-Naphthalen-1-yl-dihydro-furan-2-one (2o)

General procedure was followed with 4-(1-naphthyl)-4-oxobutanoic acid (1o, 228.3 mg) for 24 h.
Column chromatography (7/3 = hexane/EtOAc) afforded 2o as a colorless oil (165.4 mg, 78%): $^1$H NMR (CDCl$_3$, 500 MHz) $\delta$ 2.23-2.31 (m, 1 H, CH$_2$), 2.60-2.73 (m, 2 H, CH$_2$), 2.83-2.91 (m, 1 H, CH$_2$), 6.23-6.26 (m, 1 H, CH), 7.45-7.56 (m, 4 H, Ar$H$), 7.81-7.90 (m, 3 H, Ar$H$); $^{13}$C NMR (CDCl$_3$, 125 MHz) $\delta$ 28.2, 29.9, 78.6, 121.5, 122.4, 125.3, 125.9, 126.5, 128.7, 129.1, 129.4, 133.7, 135.0, 177.1; IR (ATR, cm$^{-1}$) 1774 s; MS (EI) m/z (%) 212 (M$^+$, 100); HRMS (EI-Quadrupole): calcd. for C$_{14}$H$_{12}$O$_2$ [M$^+$]: 212.0837, found: 212.0835.

5-Phenylethylidihydrofuran-2(3H)-one$^{11}$ (4)

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General procedure was followed with $\gamma$-oxo-benzenehexanoic acid (3, 206.6 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded 4 as a colorless oil (181.8 mg, 96%): $^1$H NMR (CDCl$_3$, 500 MHz) $\delta$ 1.83-1.95 (m, 2 H, CH$_2$), 2.01-2.09 (m, 1 H, CH$_2$), 2.27-2.34 (m, 1 H, CH$_2$), 2.51-2.55 (m, 2 H, CH$_2$), 2.70-2.76 (m, 1 H, CH$_2$), 2.80-2.86 (m, 1 H, CH$_2$), 4.44-4.50 (m, 1 H, CH), 7.19-7.22 (m, 3 H, Ar$H$), 7.28-7.31 (m, 2 H, Ar$H$); $^{13}$C NMR (CDCl$_3$, 125 MHz) $\delta$ 27.9, 28.8, 31.6, 37.3, 79.8, 126.1, 128.4, 128.5, 140.7, 177.1; IR (ATR, cm$^{-1}$) 1770 s; MS (EI) m/z (%) 190 (M$^+$, 51), 130 (100); HRMS (EI-Quadrupole): calcd. for C$_{12}$H$_{14}$O$_2$ [M$^+$]: 190.0994, found: 190.0994.

3-Phenylisobenzofuran-1-one$^{12}$ (6)

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General procedure was followed with 2-benzoylbenzoic acid (5, 226.1 mg) for 24 h. Column chromatography (9/1 = hexane/EtOAc) afforded 6 as a white solid (103.9 mg, 49%): mp 116–117 °C; $^1$H NMR (CDCl$_3$, 500 MHz) $\delta$ 6.41 (s, 1 H, C=H), 7.27-7.39 (m, 6 H, Ar$H$), 7.56 (t, $J = 7.5$ Hz, 1 H, Ar$H$), 7.63 (t, $J = 7.5$ Hz, 1 H, Ar$H$), 7.96 (d, $J = 7.5$ Hz, 1 H, Ar$H$); $^{13}$C NMR (CDCl$_3$, 125 MHz) $\delta$ 82.7, 122.8, 125.6, 125.6, 126.9, 128.9, 128.9, 129.3, 129.3, 134.3, 136.4, 149.6, 170.5; IR (ATR, cm$^{-1}$) 1746 s; MS (EI) m/z (%) 210 (M$^+$, 85) 105 (100); HRMS (EI-Quadrupole): calcd. for C$_{14}$H$_{10}$O$_2$ [M$^+$]: 210.0681, found: 210.0689.

4-(4-Methoxyphenyl)butanoic acid$^{13}$ (2g$^*$)

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General procedure was followed with 3-(4-methoxybenzoyl)propionic acid (1g, 208.2 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded 2g$^*$ as a white solid (41.0 mg, 21%): mp
57–59 °C 1H NMR (CDCl3, 500 MHz) δ 1.93 (quint, J = 7.5 Hz, 2 H, CH2), 2.36 (t, J = 7.5 Hz, 2 H, CH2), 2.61 (t, J = 7.5 Hz, 2 H, CH2), 6.83 (d, J = 8.5 Hz, 2 H, ArH), 7.10 (d, J = 8.5 Hz, 2 H, ArH); 13C NMR (CDCl3, 125 MHz) δ 26.4, 33.2, 34.0, 55.2, 113.8, 129.4, 133.2, 157.9, 179.7; IR (ATR, cm⁻¹) 1694 s; MS (EI) m/z (%) 194 (M⁺, 33), 121 (100); HRMS (EI-Quadrupole): calcd. for C11H14O3 [M⁺]: 190.0943, found: 190.0947.

4-(4-Phenoxyphenyl)butanoic acid¹⁴ (2h*)

General procedure was followed with 3-(4-phenoxybenzoyl)propionic acid (1h, 270.1 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded 2h* as a white solid (11.3 mg, 4%): mp 41–43 °C; 1H NMR (CDCl3, 500 MHz) δ 1.96 (quint, J = 7.5 Hz, 2 H, CH2), 2.39 (t, J = 7.5 Hz, 2 H, CH2), 2.61 (t, J = 7.5 Hz, 2 H, CH2), 6.94 (d, J = 8.0 Hz, 2 H, ArH), 6.99 (dd, J = 9.0 Hz, 1.0 Hz 2 H, ArH), 7.08 (t, J = 8.0 Hz, 1 H, ArH), 7.14 (d, J = 9.0 Hz, 2 H, ArH), 7.32 (dd, J = 8.0 Hz, 1.0 Hz 2 H, ArH); 13C NMR (CDCl3, 125 MHz) δ 26.3, 33.2, 34.2, 118.6, 119.0, 123.0, 129.7, 136.1, 155.4, 157.5, 179.4; IR (ATR, cm⁻¹) 1690 s; MS (EI) m/z (%) 256 (M⁺, 41), 183 (100); HRMS (EI-Quadrupole): calcd. for C16H18O3 [M⁺]: 256.10995, found: 256.10940.

(E)-5-Phenylpent-4-enoic acid¹⁵ (8)

General procedure was followed with 5-oxo-5-phenylpentanoic acid (7, 192.3 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded 8* as a white solid (12.0 mg, 8%): mp 88–90 °C; 1H NMR (CDCl3, 500 MHz) δ 2.55 (m, 4 H, CH2), 6.18-6.24 (m, 1 H, CH), 6.45 (d, J = 16.0 Hz, 1 H, CH), 7.21 (t, J = 7.5 Hz, 1 H, ArH), 7.29 (t, J = 7.5 Hz, 2 H, ArH), 7.34 (d, J = 7.5 Hz, 2 H, ArH); 13C NMR (CDCl3, 125 MHz) δ 27.9, 33.7, 126.1, 127.2, 128.0, 128.5, 131.2, 137.2, 178.9; IR (ATR, cm⁻¹) 1694 s; MS (EI) m/z (%) 176 (M⁺, 39), 117 (100); HRMS (EI-Quadrupole): calcd. for C11H12O2 [M⁺]: 176.0837, found: 176.0838.

6-Phenyltetrahydro-2H-pyran-2-one¹⁰ (9)

General procedure was followed with 5-oxo-5-phenylpentanoic acid (7, 192.6 mg) and triethylsilane for 4 h. Column chromatography (7/3 = hexane/EtOAc) afforded 8 as a colorless oil (14.5 mg, 8%): 1H NMR (CDCl3, 500 MHz) δ 1.83-1.90 (zm, 1 H, CH2), 1.97-2.02 (m, 2 H, CH2),
2.15-2.19 (m, 1 H, CH$_2$), 2.55-2.61 (m, 1 H, CH$_2$), 2.69-2.74 (m, 1 H, CH$_2$), 2.51-2.55 (m, 2 H, CH$_2$), 2.70-2.76 (m, 1 H, CH$_2$), 2.80-2.86 (m, 1 H, CH$_2$), 5.36 (dd, 1 H, J = 10.5, 3.5 Hz, CH), 7.31-7.40 (m, 5 H, ArH); $^{13}$C NMR (CDCl$_3$, 125 MHz) $\delta$ 18.6, 29.5, 30.5, 81.6, 125.7, 128.2, 128.6, 139.7, 171.3; IR (ATR, cm$^{-1}$) 1732 s; MS (EI) $m/z$ (%) 176 (M$^+$, 51), 104 (100); HRMS (EI-Quadrupole): calcd. for C$_{11}$H$_{12}$O$_2$ [M]$^+$: 176.0837, found: 176.0834.

2-Phenyltetrahydropyran$^{16}$ (9’)

General procedure was followed with 5-oxo-5-phenylpentanoic acid (7, 192.6 mg) and triethylsilane for 4 h. Gel permeation chromatography (chloroform) afforded 9’ as a colorless oil (34.6 mg, 21%): $^1$H NMR (CDCl$_3$, 500 MHz) $\delta$ 1.57-1.72 (m, 5 H, CH$_2$), 1.82-1.84 (m, 1 H, CH$_2$), 1.93-1.95 (m, 1 H, CH$_2$), 3.59-3.64 (m, 1 H, CH$_2$), 4.12-4.15 (m, 1 H, CH$_2$), 4.32 (dd, J = 11.0 Hz, 2.0 Hz, 1 H, CH$_2$), 7.23-7.25 (m, 1 H, ArH), 7.31-7.36 (m, 4 H, ArH); $^{13}$C NMR (CDCl$_3$, 125 MHz) $\delta$ 24.0, 25.9, 34.0, 69.0, 80.1, 125.8, 127.2, 128.2, 143.3; MS (EI) $m/z$ (%) 162 (M$^+$, 93), 105 (100); HRMS (EI-Quadrupole): calcd. for C$_{11}$H$_{14}$O [M]$^+$: 162.1045, found: 162.1042.
References and Notes

2h