Supporting Information
for
Gold(I)-catalyzed formation of furans from γ-acyloxyalkynyl ketones

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General procedures, characterization data and NMR spectra for compounds 1a–l, 2a–l and 3

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General Information:

Proton ($^1$H NMR) and carbon ($^{13}$C NMR) nuclear magnetic resonance spectra were recorded on 300, 400 or 500 MHz instruments. The chemical shifts are given in part per million (ppm) on the delta scale. The solvent peak was used as reference value. For $^1$H NMR: CDCl$_3$ = 7.26 ppm. For $^{13}$C NMR: CDCl$_3$ = 77.16 ppm. Data are presented as follow: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, m = multiplet), coupling constants (J/Hz) and integration. Assignments were determined either on the basis of unambiguous chemical shifts or coupling patterns, COSY, HMQC, HMBC, NOESY experiments to fully interpreted spectra for related compounds. Infrared spectra were recorded neat with Hewlett-Packard spectrometer. Wavelengths of maximum absorbance ($\nu_{max}$) are quoted in wave numbers (cm$^{-1}$). High resolution mass spectra (HRMS) data were recorded on a microTOF spectrometer equipped with orthogonal electrospray interface (ESI). The parent ions [M]+, [M + H]+, [M + Li]+ or [M + Na]⁺ are quoted. Analytical thin-layer chromatography (TLC) was carried out on silica gel 60 F254 plates or basic alumina (63–200 μm) with visualization by ultraviolet light, cerium ammonium molybdate (CAM) or potassium permanganate dip. Flash-column chromatography was carried out using silica gel 60 (40–63 μm) or basic Al$_2$O$_3$ (63–200 μm) and the procedure included the subsequent evaporation of solvents in vacuo. Reagents and solvents were purified using standard means. Dichloroethane ([CH$_2$Cl]$_2$), and triethylamine (NET$_3$) were distilled from CaH$_2$ under an argon atmosphere; pyridine was distilled from potassium carbonate; diisopropylamine (DIPA) was distilled from KOH; and tetrahydrofuran (THF) was dried using Glasstechnology GT S100 device. Anhydrous reactions were carried out in flame-dried glassware and under an argon atmosphere.

CuI (98%) and DMA (≥99.5%) was purchased from Sigma Aldrich. AgSbF$_6$ (98%) was purchased from STREM Chemicals. AgNTf$_2$ was prepared from commercially available HNTf$_2$ (Aldrich) and Ag$_2$CO$_3$ [1]. Triphenylphosphinegold(I) chloride was prepared by reduction of NaAuCl$_4$ with thiodiethanol and subsequent addition of triphenylphosphine [2]. PPh$_3$AuNTf$_2$ catalyst was prepared from the corresponding triphenylphosphinegold(I) chloride and AgNTf$_2$ [3]. Cu(MeCN)$_2$NTf$_2$ was prepared from commercially available HNTf$_2$ (Aldrich) and Cu$_2$O (Acros) [4]. All other chemicals were used as received. All extractive procedures were performed using non distilled solvents and all aqueous solutions used were saturated unless details are given.


To an oven-dried flask under argon were successively added copper iodide (4 mol %), dichlorobis(triphenylphosphine)palladium (2 mol %) and NET$_3$ [0.4 M]. The acyloxyalkyne (1 equiv) previously dissolved in NET$_3$ [0.5 M] was added to the catalyst mixture via cannula.

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and briefly stirred. The acid chloride (1.1 equiv) was then added dropwise. The reaction was stirred at room temperature and monitored by TLC until completion. The mixture was quenched with HCl (1 N) and extracted with Et₂O. The organic layers were washed with HCl (1 N), NaHCO₃ solution and brine, dried over MgSO₄ and concentrated under vacuum. The resulting crude product was purified by flash-column chromatography on silica gel (cyclohexane/EtOAc).

Procedure B: preparation of γ-acyloxyalkynyl ketone by condensation [5]

To a stirred solution of propargyl alcohol (1 equiv) in THF [0.5 M] under argon at −78 °C, was dropwise added n-BuLi ([1.6 M] in hexanes, 2.2 equiv). The resulting solution was then allowed to warm to room temperature and stirred for 0.5 to 1 hour. A solution of acid chloride (2.5 equiv) in THF [1.8 M] under argon at −78 °C was added via cannula to the mixture. The reaction was then allowed to warm to room temperature and stirred for 2 to 7 hours (monitored by TLC). The reaction was quenched with NH₃ (aqueous solution) and extracted with Et₂O. The organic layers were washed with HCl (1 N), NaHCO₃ and brine, dried over MgSO₄ and concentrated under vacuum. The resulting crude product was purified by column chromatography on silica gel (cyclohexane/EtOAc).

5-Oxo-5-phenylpent-3-yn-2-yl pivalate (1a) [5]: Prepared following the Procedure A in 63% yield (1.06 g, 4.10 mmol) from 1.00 g (6.48 mmol) of but-3-yn-2-yl pivalate. Colorless oil; TLC Rf 0.60 (cyclohexane/EtOAc 25%); ¹H NMR (400 MHz, CDCl₃) δ 1.26 (s, 9H), 1.63 (d, J = 6.8 Hz, 3H), 5.66 (q, J = 6.8 Hz, 2H), 7.48 (dd, J = 8.1, 7.5 Hz, 2H), 7.62 (t, J = 7.5 Hz, 1H), 8.10 (d, J = 8.1 Hz, 2H); ¹³C NMR (125.8 MHz, CDCl₃) δ 20.5, 27.2, 38.9, 59.8, 82.0, 92.0, 128.7, 129.7, 134.4, 136.6, 177.3, 177.6.

6,6-Dimethyl-5-oxohept-3-yn-2-yl pivalate (1b): Prepared following the Procedure B in 38% yield (1.30 g, 5.45 mmol) from 1.00 g (14.30 mmol) of but-3-yn-2-yl pivalate. Colorless oil; TLC Rf 0.40 (pentane/Et₂O 5%); IR (neat) νmax 1007, 1035, 1079, 1133, 1274, 1366, 1396, 1460, 1479, 1675, 1736, 2217, 2872, 2936, 2971 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 1.18 (s, 9H), 1.22 (s, 9H), 1.55 (d, J = 6.8 Hz, 3H), 5.55 (q, J = 6.8 Hz, 1H); ¹³C NMR (125.8 MHz, CDCl₃) δ 20.4, 26.0, 27.1, 38.9, 45.0, 59.7, 81.3, 91.4, 177.2, 193.7; HRMS 261.148 (C₁₄H₂₁O₃ + Na calcd 261.146).

5-Methyl-1-oxo-1-phenyltetradec-2-yn-4-yl pivalate (1c): Prepared following the Procedure A in 56% yield (229 mg, 0.57 mmol, mixture 50/50 of 2 diastereoisomers) from 300 mg (1.02 mmol) of 4-methyltridec-1-yn-3-yl pivalate. Colorless oil; TLC Rf 0.56 (cyclohexane/EtOAc 10%); IR (neat) νmax 977, 1031, 1133, 1259, 1312, 1365, 1396, 1450, 1582, 1598, 1650, 1736, 2208, 2226, 2854, 2924, 2958, 3067 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 0.83–0.92 (m, 6H), 1.10 (d, J = 6.9 Hz, 3H, Diastereoisomer 1), 1.11 (d, J = 6.9 Hz, 3H, Diastereoisomer 2), 1.21–1.33 (m, 28H), 1.26–1.27 (m, 18H), 1.35–1.45 (m, 2H), 1.52–1.64 (m, 2H), 1.95–2.05 (m, 2H), 5.50 (d, J = 5.0 Hz, 1H, Diastereoisomer 2), 5.52 (d, J = 5.4 Hz, 1H, Diastereoisomer 1), 7.50–7.45 (m, 4H), 7.59–7.64 (m, 2H), 8.08–8.12 (m, 4H); ¹³C NMR (125.8 MHz, CDCl₃) δ
14.27 (x2), 15.3, 15.6, 22.8 (x2), 27.0, 27.1, 27.2 (x2), 29.5 (x2), 29.6 (x2), 29.7 (x2), 29.8, 29.9, 32.0 (x2), 32.4, 32.7, 37.2, 37.5, 39.1, 39.2, 67.4, 67.8, 83.2, 83.5, 90.5, 91.1, 128.7 (x2), 129.7 (x2), 134.4 (x2), 136.7 (x2), 177.3, 177.4, 177.50, 177.53; HRMS 421.270 (C26H38O3 + Na calc 421.271).

2,2-Dimethyl-6-oxo-6-phenylhex-4-yn-3-yl pivalate (1d): Prepared following the Procedure A in 70% yield (1.30 g, 4.33 mmol) from 1.20 g (6.12 mmol) of 4,4-dimethylpent-1-yn-3-yl pivalate. White solid; mp 54 °C; TLC Rf 0.40 (cyclohexane/EtOAc 10%); IR (neat) νmax 1110, 1266, 1313, 1366, 1395, 1448, 1478, 1580, 1596, 1638, 1739, 2238, 2871, 2935, 2972, 3071 cm⁻¹; 1H NMR (500 MHz, CDCl3) δ 1.11 (s, 9H), 1.28 (s, 9H), 5.30 (s, 1H), 7.48 (dd, J = 7.5, 7.3 Hz, 2H), 7.61 (t, J = 7.2 Hz, 1H), 8.10 (d, J = 7.5 Hz, 2H); 13C NMR (125.8 MHz, CDCl3) δ 25.9, 27.2, 35.9, 39.2, 71.5, 83.3, 90.6, 128.8, 129.7, 134.4, 136.8, 177.3, 177.5; HRMS 323.163 (C19H23O3 + Na calc 323.162).

7,7-Dimethyl-6-oxo-1-phenyloct-4-yn-3-yl pivalate (1e): Prepared following the Procedure A in 84% yield (553 mg, 1.68 mmol) from 489 mg (2.00 mmol) of 5-phenylpent-1-yn-3-yl pivalate. Colorless oil; TLC Rf 0.50 (cyclohexane/CH2Cl2 50%); IR (neat) νmax 699, 745, 1032, 1130, 1275, 1365, 1478, 1496, 1674, 1736, 2214, 2870, 2933, 2969, 3029 cm⁻¹; 1H NMR (500 MHz, CDCl3) δ 1.19 (s, 9H), 1.24 (s, 9H), 2.11–2.23 (m, 2H), 2.79 (t, J = 8.0 Hz, 2H), 5.74 (t, J = 6.8 Hz, 1H), 7.18 (d, J = 7.2 Hz, 2H), 7.21 (t, J = 7.2 Hz, 1H), 7.30 (dd, J = 7.7, 7.2 Hz, 2H); 13C NMR (125.8 MHz, CDCl3) δ 26.0, 27.2, 31.3, 35.7, 39.0, 45.0, 62.8, 82.2, 90.3, 126.5, 128.5, 128.8, 140.3, 177.2, 193.7; HRMS 351.193 (C21H28O3 + Na calc 351.194).

7,7-Dimethyl-6-oxo-1-phenyloct-4-yn-3-yl benzoate (1f): Prepared following the Procedure A in 70% yield (478 mg, 1.37 mmol) from 518 mg (1.96 mmol) of 5-phenylpent-1-yn-3-yl benzoate. Colorless oil; TLC Rf 0.23 (pentane/Et2O 5%); IR (neat) νmax 711, 745, 1025, 1067, 1094, 1141, 1261, 1476, 1672, 1723, 2214, 2868, 2931, 2968, 63.82 cm⁻¹; 1H NMR (300 MHz, CDCl3) δ 1.21 (s, 9H), 2.23–2.42 (m, 2H), 2.89 (t, J = 7.9 Hz, 2H), 4.54 (t, J = 6.6 Hz, 1H), 7.18–7.25 (m, 3H), 7.27–7.34 (m, 2H), 7.47 (dd, J = 8.3, 7.5 Hz, 2H), 7.60 (t, J = 7.5 Hz, 1H), 8.04 (d, J = 8.3 Hz, 2H); 13C NMR (125.8 MHz, CDCl3) δ 26.0, 31.5, 35.9, 45.0, 63.5, 82.6, 90.0, 126.5, 128.5, 128.6, 128.8, 129.4, 129.9, 133.6, 140.2, 165.4, 193.6; HRMS 371.158 (C23H24O4 + Na calc 371.163).

7,7-Dimethyl-6-oxo-1-phenyloct-4-yn-3-yl acetate (1g): Prepared following the Procedure A in 57% yield (331 mg, 1.16 mmol) from 410 mg (2.03 mmol) of 5-phenylpent-1-yn-3-yl acetate. Colorless oil; TLC Rf 0.28 (pentane/Et2O 10%); IR (neat) νmax 699, 745, 1023, 1222, 1369, 1476, 1673, 1745, 2213, 2869, 2933, 2969, 3028 cm⁻¹; 1H NMR (500 MHz, CDCl3) δ 1.20 (s, 9H), 2.10 (s, 3H), 2.11–2.23 (m, 2H), 2.79 (t, J = 7.9 Hz, 2H), 5.47 (t, J = 6.7 Hz, 1H), 7.18 (d, J = 7.8 Hz, 2H), 7.21 (t, J = 7.5 Hz, 1H), 7.30 (dd, J = 7.8, 7.5 Hz, 2H); 13C NMR (125.8 MHz, CDCl3) δ 21.0, 26.0, 31.4, 35.7, 45.0, 63.0, 82.4, 89.9, 126.5, 128.5, 128.8, 140.23, 169.8, 193.6; HRMS 309.149 (C18H22O3 + Na calc 309.147).
7,7-Dimethyl-6-oxo-1-phenyloct-4-yn-3-yl 2-phenylacetate (1h): Prepared following the Procedure A in 66% yield (204 mg, 0.56 mmol) from 489 mg (0.86 mmol) of 5-phenylpent-1-yn-3-yl 2-phenylacetate. Colorless oil; TLC \( R_f \) 0.27 (pentane/\( \text{Et}_2\text{O} \) 15%); IR (neat) \( \nu_{\text{max}} \) 1131, 1240, 1455, 1476, 1496, 1672, 2213, 2868, 2931, 2968, 3029, 3063, 3087 cm\(^{-1}\); \(^1\)H NMR (300 MHz, \( \text{CDCl}_3 \)) \( \delta \) 1.54 (s, 9H), 2.06–2.24 (m, 2H), 2.71 (t, \( J = 7.8 \) Hz, 2H), 3.65 (s, 2H), 5.47 (t, \( J = 6.6 \) Hz, 1H), 7.10 (d, \( J = 7.6 \) Hz, 2H), 7.20 (t, \( J = 7.3 \) Hz, 1H), 7.25–7.37 (m, 7H); \(^{13}\)C NMR (125.8 MHz, \( \text{CDCl}_3 \)) \( \delta \) 26.0, 31.2, 35.7, 41.3, 45.0, 63.3, 82.5, 89.7, 126.5, 127.5, 128.5, 128.7, 128.8, 129.4, 133.5, 140.2, 170.3, 193.6; HRMS 385.176 (\( \text{C}_{24}\text{H}_{28}\text{O}_3 + \text{Na} \) calc 385.177).

2-Oxodec-3-yn-5-yl acetate (1i) [6]: Prepared following the Procedure B in 52% yield (2.11 g 10.03 mmol) from 2.8 mL (19.68 mmol) of oct-1-yn-3-ol. Colorless oil; TLC \( R_f \) 0.50 (cyclohexane/\( \text{EtOAc} \) 20%); \(^1\)H NMR (300 MHz, \( \text{CDCl}_3 \)) \( \delta \) 0.90 (t, \( J = 6.9 \) Hz, 3H), 1.27–1.37 (m, 4H), 1.38–1.50 (m, 2H), 1.76–1.86 (m, 2H), 2.10 (s, 3H), 2.35 (s, 3H), 5.46 (t, \( J = 6.7 \) Hz, 1H); \(^{13}\)C NMR (75.5 MHz, \( \text{CDCl}_3 \)) \( \delta \) 14.1, 21.0, 22.6, 24.7, 31.3, 32.7, 34.1, 63.4, 84.3, 88.5, 169.9, 184.

5-Oxooct-3-yn-2-butylbpyrate (1j): Prepared following the Procedure B in 58% yield (876 mg, 4.17 mmol) from 0.5 mL (7.13 mmol) of but-3-yn-2-ol. Yellowish oil; TLC \( R_f \) 0.38 (pentane/\( \text{EtOAc} \) 20%); IR (neat) \( \nu_{\text{max}} \) 1023, 1078, 1159, 1245, 1350, 1456, 1678, 1742, 2216, 2876, 2937, 2965 cm\(^{-1}\); \(^1\)H NMR (300 MHz, \( \text{CDCl}_3 \)) \( \delta \) 0.93 (t, \( J = 7.4 \) Hz, 3H), 0.96 (t, \( J = 7.4 \) Hz, 3H), 1.53 (d, \( J = 6.8 \) Hz, 3H), 1.67 (q, \( J = 7.4 \), 7.4 Hz, 2H), 1.68 (q, \( J = 7.4 \), 7.4 Hz, 2H), 2.32 (t, \( J = 7.4 \) Hz, 2H), 2.53 (t, \( J = 7.4 \) Hz, 2H), 2.56 (q, \( J = 6.8 \) Hz, 1H); \(^{13}\)C NMR (125.8 MHz, \( \text{CDCl}_3 \)) \( \delta \) 13.6, 13.7, 17.5, 18.5, 20.6, 36.1, 47.3, 59.4, 83.1, 89.3, 172.4, 187.6; HRMS 233.116 (\( \text{C}_{12}\text{H}_{13}\text{O}_3 + \text{Na} \) calc 233.115).

5-Oxo-7-phenylhept-3-yn-2-yl 3-phenylpropanoate (1k): Prepared following the Procedure B in 45% yield (1.89 g, 5.69 mmol) from 1 mL (12.76 mmol) of but-3-yn-2-ol. Yellowish oil; TLC \( R_f \) 0.31 (pentane/\( \text{EtOAc} \) 20%); IR (neat) \( \nu_{\text{max}} \) 698, 748, 1030, 1076, 1087, 1138, 1229, 1453, 1496, 1678, 1740, 2220, 2934, 3028 cm\(^{-1}\); \(^1\)H NMR (300 MHz, \( \text{CDCl}_3 \)) \( \delta \) 1.05 (d, \( J = 6.9 \) Hz, 3H), 2.67 (t, \( J = 7.6 \) Hz, 2H), 2.89 (t, \( J = 7.6 \) Hz, 2H), 2.97 (m, 4H), 5.54 (q, \( J = 6.9 \) Hz, 1H), 7.16–7.23 (m, 6H), 7.26–7.32 (m, 4H); \(^{13}\)C NMR (125.8 MHz, \( \text{CDCl}_3 \)) \( \delta \) 20.5, 29.7, 30.9, 35.8, 47.0, 59.7, 83.1, 89.6, 126.5, 126.5, 128.4, 128.5, 128.7, 128.7, 140.1, 140.2, 171.6, 186.3; HRMS 357,144 (\( \text{C}_{22}\text{H}_{22}\text{O}_3 + \text{Na} \) calc 357,146).

9-(Benzyloxy)-6-oxo-1-phenylnon-4-yn-3-yl acetate (II): To a stirred solution of 1-(benzyloxy)-7-hydroxy-9-phenylnon-5-yn-4-one [7] (500 mg, 1.31 mmol) in dichloromethane was added at once a mixture of pyridinium chlorochromate (430 mg, 2 mmol) and SiO\(_2\) (430 mg). After 5 h of stirring at room temperature, the reaction mixture was filtered through a pad of Celite\(^{6}\) (\( \text{CH}_3\text{Cl}_2 \)) and the filtrate was concentrated in vacuo. The crude product was purified by flash chromatography (cyclohexane/\( \text{EtOAc} \) 10-20%) to afford II as a pale yellow oil (460 mg, 1.22 mmol, 93%). TLC \( R_f \) 0.45 (cyclohexane/\( \text{EtOAc} \) 25%); IR (neat) \( \nu_{\text{max}} \) 698, 737, 1025, 1101, 1155, 1220, 1369, 1454, 1496, 1677, 1743, 2217, 2859, 2931, 3028 cm\(^{-1}\); \(^1\)H NMR (300 MHz, \( \text{CDCl}_3 \)) \( \delta \) 1.97 (tt, \( J = 6.0 \), 7.3 Hz, 2H), 2.09 (s, 3H), 2.10–2.19 (m, 2H).

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\(^{7}\) This compound was synthesized according to the sequence described by Singh, M.; Argade, N. P. J. Org. Chem. 2010, 75, 3121–3124.
Procedure C: gold(I)-catalyzed formation of furans from γ-acyloxyalkynyl ketones

In an oven-dried flask, γ-acyloxyalkynyl ketone (0.4 mmol) was dissolved in dichloroethane (0.1 M) and heated to 70 °C under argon atmosphere. Ph3PdAuNTf2 (2.5 mol %) was then added to the stirred solution at 70 °C. The reaction was monitored by thin-layer chromatography until completion. The solvent was then removed in vacuo and the crude residue was purified by flash chromatography on silica gel (pentane/EtO).

2-Methyl-5-phenylfuran-3-yl pivalate (2a) [5]: Prepared following the Procedure C in 95% yield (99 mg, 0.38 mmol) from 102 mg (0.40 mmol) of 1a. White solid; mp 57 °C; TLC Rf 0.60 (Pentane/EtO 5%); 1H NMR (400 MHz, CDCl3) δ 8.4, 8.2, 5.8 (s, 1H), 7.23 (t, J = 7.3 Hz, 2H), 5.95 (s, 1H), 6.10 (s, 1H), 6.30 (s, 1H), 7.46 (dd, J = 7.5, 7.1 Hz, 2H), 7.54 (t, J = 7.1 Hz, 1H), 7.86 (d, J = 7.5 Hz, 2H), 13C NMR (125.8 MHz, CDCl3) δ 10.8, 27.3, 39.2, 102.1, 123.4, 127.3, 128.7, 131.0, 135.9, 139.8, 150.1, 176.2.

3-Hydroxy-5-oxo-5-phenylpent-3-en-2-yl pivalate (3): By-product obtained following the Procedure C (at room temperature instead of 70 °C) in 22% yield (22 mg, 0.09 mmol) from 101 mg (0.39 mmol) of 1a. Yellowish oil; TLC Rf 0.31 (pentane/EtO 5%); IR (neat) νmax 521, 633, 685, 761, 879, 1036, 1060, 1116, 1150, 1278, 1477, 1569, 1598, 1731, 2903, 2958 cm−1; 1H NMR (300 MHz, CDCl3) δ 1.30 (s, 9H), 1.50 (d, J = 7.0 Hz, 3H), 5.28 (q, J = 7.0 Hz, 1H), 6.30 (s, 1H), 7.46 (dd, J = 7.5, 7.1 Hz, 2H), 7.54 (t, J = 7.1 Hz, 1H), 7.86 (d, J = 7.5 Hz, 2H), 13C NMR (125.8 MHz, CDCl3) δ 17.9, 27.3, 38.9, 71.9, 92.4, 127.1, 128.9, 132.7, 134.4, 177.6, 182.6, 196.1; HRMS 276.137 (C16H20O + Na calcld 276.136).

5-(3-Butyl)-2-methylfuran-3-yl pivalate (2b): Prepared following the Procedure C in 93% yield (101 mg, 0.42 mmol) from 108 mg (0.45 mmol) of 1b. Colorless oil; TLC Rf 0.77 (pentane/EtO 5%); IR (neat) νmax 939, 1028, 1117, 1190, 1241, 1277, 1363, 1395, 1460, 1479, 1652, 1735, 2871, 2914, 2967 cm−1; 1H NMR (300 MHz, CDCl3) δ 1.23 (s, 9H), 1.31 (s, 9H), 2.14 (s, 3H), 5.95 (s, 1H); 13C NMR (125.8 MHz, CDCl3) δ 10.7, 27.3, 29.0, 32.8, 39.1, 99.3, 134.2, 137.6, 160.5, 176.5; HRMS 261.146 (C14H22O3 + Na calcld 261.146).

5-Phenyl-2-(undecan-2-yl)furan-3-yl pivalate (2c): Prepared following the Procedure C in 90% yield (45 mg, 0.11 mmol) from 49 mg (0.12 mmol) of 1c. Colorless oil; TLC Rf 0.38 (pentane/EtO 5%); IR (neat) νmax 1113, 1175, 1275, 1397, 1456, 1604, 1754, 2838, 2924, 2971 cm−1; 1H NMR (300 MHz, CDCl3) δ 0.86 (t, J = 7.0 Hz, 3H), 1.20–1.27 (m, 14H), 1.29 (d, J = 7.2 Hz, 3H), 1.34 (s, 9H), 1.51–1.59 (m, 1H), 1.65–1.74 (m, 1H), 2.83 (dq, J = 8.4, 7.2, 5.8 Hz, 1H), 6.65 (s, 1H), 7.22 (t, J = 7.4 Hz, 1H), 7.35 (dd, J = 8.4, 7.4 Hz, 2H), 7.59 (d, J = 8.4 Hz, 2H); 13C NMR (125.8 MHz, CDCl3) δ
2-(tert-Butyl)-5-phenylfuran-3-yl pivalate (2d): Prepared following the Procedure C in 61% yield (63 mg, 0.21 mmol) from 104 mg (0.35 mmol) of 1d. Colorless oil; TLC Rf 0.64 (pentane/Et2O 10%); IR (neat) νmax 1079, 1117, 1156, 1175, 1275, 1460, 1477, 1601, 1745, 2873, 2905, 2963, 2991, 3058 cm⁻¹; 1H NMR (500 MHz, CDCl3) δ 1.35 (s, 9H), 1.37 (s, 9H), 6.59 (s, 1H), 7.23 (t, J = 7.3 Hz, 1H), 7.35 (dd, J = 8.0, 7.3 Hz, 2H), 7.59 (d, J = 8.0 Hz, 2H); 13C NMR (125.8 MHz, CDCl3) δ 27.3, 29.0, 33.2, 39.1, 102.7, 123.4, 127.3, 128.7, 131.0, 134.0, 148.9, 149.1, 176.4; HRMS 323.161 (C10H13O3 + Na calcd 323.162).

5-(tert-Butyl)-2-phenylfuran-3-yl pivalate (2d'): isolated as a mixture of 2d/2d' using Procedure C from 104 mg (0.35 mmol) of 1d. TLC Rf 0.71 (pentane/Et2O 10%); 1H NMR (400 MHz, CDCl3) δ 1.32 (s, 9H), 1.39 (s, 9H), 6.24 (s, 1H), 7.21 (t, J = 7.3 Hz, 1H), 7.38 (dd, J = 8.0, 7.3 Hz, 2H), 7.68 (d, J = 8.0 Hz, 2H); 13C NMR (125.8 MHz, CDCl3) δ 27.4, 29.0, 33.1, 39.3, 101.2, 123.9, 126.7, 128.6, 130.3, 135.7, 138.4, 161.6, 176.0.

5-(tert-Butyl)-2-phenylfuran-3-yl pivalate (2e): Prepared following the Procedure C in 78% yield (71 mg, 0.22 mmol) from 91 mg (0.28 mmol) of 1e. Colorless oil; TLC Rf 0.52 (pentane/Et2O 5%); IR (neat) νmax 942, 1029, 1117, 1189, 1273, 1397, 1454, 1748, 2871, 2934, 2966, 3030, 3066, 3088 cm⁻¹; 1H NMR (500 MHz, CDCl3) δ 1.25 (s, 9H), 1.28 (s, 9H), 2.82 (t, J = 7.6 Hz, 2H), 2.92 (t, J = 7.6 Hz, 2H), 5.98 (s, 1H), 7.15 (d, J = 7.7 Hz, 2H), 7.20 (t, J = 7.4 Hz, 1H), 7.28 (dd, J = 7.7, 7.4 Hz, 2H); 13C NMR (125.8 MHz, CDCl3) δ 27.3, 27.5, 28.9, 32.9, 34.2, 39.1, 99.3, 126.1, 128.4, 128.5, 134.3, 140.3, 141.4, 160.6, 176.3; HRMS 351.191 (C12H18O3 + Na calcd 351.193).

5-(tert-Butyl)-2-phenylfuran-3-yl benzoate (2f): Prepared following the Procedure C in 81% yield (75 mg, 0.21 mmol) from 100 mg (0.29 mmol) of 1f. Colorless oil; TLC Rf 0.45 (pentane/Et2O 10%); IR (neat) νmax 698, 1024, 1057, 1096, 1192, 1260, 1396, 1452, 1741, 2867, 2904, 2930, 2965, 3027 cm⁻¹; 1H NMR (500 MHz, CDCl3) δ 1.27 (s, 9H), 2.90 (t, J = 7.5 Hz, 2H), 2.96 (t, J = 7.5 Hz, 2H), 6.08 (s, 1H), 7.14–7.19 (m, 3H), 7.23–7.27 (m, 2H), 7.48 (dd, J = 8.3, 7.5 Hz, 2H), 7.62 (t, J = 7.5 Hz, 1H), 8.07 (d, J = 8.3 Hz, 2H); 13C NMR (125.8 MHz, CDCl3) δ 27.6, 29.0, 33.0, 34.1, 99.3, 126.1, 128.4, 128.6, 129.4, 130.2, 133.6, 134.3, 140.8, 141.4, 160.8, 164.4; HRMS 371.165 (C13H14O3 + Na calcd 371.163).

5-(tert-Butyl)-2-phenylfuran-3-yl acetate (2g): Prepared following the Procedure C in 70% yield (71 mg, 0.48 mmol) from 101 mg (0.35 mmol) of 1g. Colorless oil; TLC Rf 0.56 (pentane/Et2O 10%); IR (neat) νmax 1030, 1094, 1206, 1289, 1366, 1455, 1645, 1759, 2868, 2930, 2964, 3027, 3063, 3073 cm⁻¹; 1H NMR (500 MHz, CDCl3) δ 1.23 (s, 9H), 2.16 (s, 3H), 2.82 (t, J = 7.8 Hz, 2H), 2.91 (t, J = 7.8 Hz, 2H), 5.93 (s, 1H), 7.16 (d, J = 7.0 Hz, 2H), 7.19 (t, J = 7.5 Hz, 1H), 7.27 (dd, J = 7.5, 7.0 Hz, 2H); 13C NMR (125.8 MHz, CDCl3) δ 20.9, 27.4, 28.9, 32.9, 34.1, 99.2, 126.1, 128.4, 128.5, 134.1, 140.6, 141.4, 160.6, 168.8; HRMS 309.145 (C11H12O2 + Na calcd 309.146).

5-(tert-Butyl)-2-phenylfuran-3-yl 2-phenyl acetate (2h): Prepared following the Procedure C in 75% yield (64 mg, 0.18 mmol) from 85 mg (0.23 mmol) of 1h. Colorless oil; TLC Rf 0.42 (pentane/Et2O 10%); IR (neat) νmax 946, 1121, 1234, 1289, 1397, 1496, 1645, 1757, 2868, 2930, 2965, 3028, 3063, 3086 cm⁻¹; 1H NMR (500 MHz, CDCl3) δ 1.21 (s, 9H), 2.72 (t, J = 7.7 Hz, 2H), 2.82 (t, J =
5-Methyl-2-pentylfuran-3-yl acetate (2i) [6]: Prepared following the Procedure C in 77% yield (77 mg, 0.37 mmol) from 100 mg (0.48 mmol) of 1i. Colorless oil; TLC Rf 0.35 (pentane/Et2O 5%); 1H NMR (300 MHz, CDCl3) δ 0.89 (t, J = 7.0 Hz, 3H), 1.25–1.36 (m, 4H), 1.54–1.62 (m, 2H), 2.22 (d, J = 1.0 Hz, 3H), 2.23 (s, 3H), 2.48 (t, J = 7.7 Hz, 2H), 5.94 (q, J = 0.9 Hz, 1H); 13C NMR (125.8 MHz, CDCl3) δ 14.1, 20.9, 22.5, 25.2, 27.6, 31.4, 102.7, 133.9, 142.3, 148.6, 169.1.

2-Methyl-5-propylfuran-3-yl butyrate (2j): Prepared following the Procedure C in 68% yield (71 mg, 0.33 mmol) from 104 mg (0.49 mmol) of 1j. Colorless oil; TLC Rf 0.58 (pentane/Et2O 10%); IR (neat) νmax 943, 1149, 1240, 1372, 1456, 1652, 1761, 2874, 2933, 2962 cm⁻¹; 1H NMR (400 MHz, CDCl3) δ 0.95 (t, J = 7.5 Hz, 3H), 1.01 (t, J = 7.5 Hz, 3H), 1.62 (qt, J = 7.5, 7.5 Hz, 2H), 1.75 (qt, J = 7.5, 7.5 Hz, 2H), 2.14 (s, 3H), 2.47 (t, J = 7.5 Hz, 2H), 2.49 (t, J = 7.5 Hz, 2H), 5.96 (s, 1H); 13C NMR (125.8 MHz, CDCl3) δ 10.7, 13.8, 13.9, 18.6, 21.3, 30.6, 36.0, 102.1, 134.1, 137.9, 152.8, 171.6; HRMS 233.115 (C12H18O3 + Na calcld 233.115).

2-Methyl-5-phenethylfuran-3-yl 3-phenylpropanoate (2k): Prepared following the Procedure C in 74% yield (76 mg, 0.23 mmol) from 103 mg (0.31 mmol) of 1k. Colorless oil; TLC Rf 0.51 (pentane/Et2O 20%); IR (neat) νmax 697, 747, 789, 933, 1075, 1135, 1235, 1371, 1453, 1495, 1651, 1758, 2860, 2921, 3027 cm⁻¹; 1H NMR (400 MHz, CDCl3) δ 2.09 (s, 3H), 2.83 (t, J = 7.8 Hz, 4H), 2.92 (t, J = 7.8 Hz, 2H), 3.04 (t, J = 7.8 Hz, 2H), 5.93 (s, 1H); 7.18–7.26 (m, 6H), 7.27–7.34 (m, 4H); 13C NMR (125.8 MHz, CDCl3) δ 10.7, 30.5, 31.1, 34.2, 35.7, 102.5, 126.2, 126.6, 128.5, 128.5, 128.7, 134.0, 138.7, 140.2, 141.2, 151.8, 170.9; HRMS 357,145 (C22H22O5 + Na calcld 357,146).

5-(3-(Benzylxoy)propyl)-2-phenethylfuran-3-yl acetate (2l): Prepared following the Procedure C in 65% yield (68 mg, 0.18 mmol) from 105 mg (0.28 mmol) of 1l. Colorless oil; TLC Rf 0.43 (cyclohexane/EtOAc 20%); IR (neat) νmax 578, 697, 734, 1071, 1099, 1206, 1367, 1453, 1495, 1759, 2856, 2925, 3027 cm⁻¹; 1H NMR (400 MHz, CDCl3) δ 1.84 (tt, J = 7.5, 6.3 Hz, 2H), 2.08 (s, 3H), 2.58 (t, J = 7.5 Hz, 2H), 2.72 (t, J = 7.7 Hz, 2H), 2.81 (t, J = 7.7 Hz, 2H), 3.43 (t, J = 6.3 Hz, 2H), 4.23 (s, 2H), 5.89 (s, 1H), 7.06–7.30 (m, 10H); 13C NMR (125.8 MHz, CDCl3) δ 20.8, 25.3, 27.4, 28.1, 34.1, 69.4, 73.1, 102.4, 126.2, 127.7, 127.8, 128.5, 128.5, 134.2, 138.7, 141.1, 141.4, 152.4, 168.7; HRMS 402,175 (C24H26O4 + Na calcld 402,176).