Supporting Information

Nickel-Catalyzed Electrochemical Reductive Relay Cross-Coupling of Alkyl Halides with Alkyl Carboxylic Acids

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1 General Information

All the electrochemical reduction were performed in an undivided cell equipped with Nickel foam (1.5×2.5 cm²) and an iron rod as sacrificial anode unless otherwise noted. Solvents and commercially available reagents were used without purification. Column chromatography was performed using either 100-200 Mesh or 300-400 Mesh silica gel. Visualization of spots on TLC plate was accomplished with UV light (254 nm). All the nickel foams were purchased from T-mall, China. The potentiostat was purchased from Shiqiang Telecom Co., Ltd, Shengzhen, China. The All commercial reagents were purchased from TCI, Sigma-Aldrich, Adamas-beta chemistry and Energy Chemical of the highest purity grade. They were used without further purification unless specified. 1H NMR and 13C NMR spectra were recorded on Agilent AV 400, Varian Inova 400 (400 MHz and 101 MHz, respectively). 19F NMR spectra were recorded on Agilent AV 400, Varian Inova 400 (376 MHz) instrument. The peaks were internally referenced to TMS (0.00 ppm) or residual undeuterated solvent signal. The following abbreviations were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br = broad. Infrared spectra were obtained on a Bio-Rad FTS-185 instrument. High resolution mass spectra were recorded at the Center for Mass Spectrometry, Shanghai Institute of Organic Chemistry. Analytical and spectral data of all those known compounds are exactly matching with the reported values.
2 Structures of Starting Materials

Alkyl acids

1a

1b

1c

1d

1e

1f

1g

1h

1i

1j

1k

1l

1m

1n

1o

1p

1q

1r

1s

1t

1u

1v

1w

1x

1y

1z

1aa

1ab

1ac

1ad

1ae

1af

1ag
Alkyl bromides

Failed substrates
3 Preparation of alkyl bromide substrates

General procedure for the synthesis of 2b, 2e-2g, 2j, 2l and 2m.

Procedure A: LiAlH₄ (1.2 equiv.) and anhydrous THF (c = 1.25 M) were added to the flask. Then a solution of acid (1.0 equiv.) in anhydrous THF (c = 1.25 M) was added dropwise under the ice bath. The mixture was stirred at room temperature for 1 h. then the reaction was quenched with MeOH and NaOH (10% in aqueous), followed by workup with hydrochloric acid (1 M). The mixture was extracted with EtOAc (3 equal volume) and the organic layer was washed with brine, dried with Na₂SO₄, and concentrated by flash evaporation for the next step without further purification.

Procedure B: To a stirred solution of alcohol (1.0 equiv.) and CBr₄ (1.2 equiv.) in DCM (0.3 M) was added triphenylphosphine (1.2 equiv.). After stirring at room temperature for 5 h, the mixture was concentrated by flash evaporation. The residue was added with petroleum ether and filtered. The solution was concentrated by flash evaporation and the crude product was purified by flash column chromatography on silica to afford the product.

1-(2-bromoethyl)-4-methoxybenzene (2b) [1]

From 2-(4-methoxyphenyl)acetic acid (3.32 g, 20 mmol), following the general procedure A and B, the title compound (2b) was obtained (2.6 g, 60% in two steps) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.18 (d, J = 8.8 Hz, 2H), 6.91 (d, J = 8.8 Hz, 2H), 3.84 (s, 3H), 3.58 (t, J = 7.6 Hz, 2H), 3.15 (t, J = 7.6 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 158.51, 130.98, 129.66, 113.97, 55.23, 38.56, 33.47.

4-(2-bromoethyl)-1,1'-biphenyl (2e) [1]

From 2-([1,1'-biphenyl]-4-yl)acetic acid (4.25 g, 20 mmol), following the general procedure A and B, the title compound (2e) was obtained (4.06 g, 78% in two steps) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.64 (d, J = 7.2 Hz, 2H), 7.60 (d, J = 8.0 Hz, 2H), 7.49 (t, J = 7.6 Hz, 2H), 7.43 – 7.37 (m, 1H), 7.33 (d, J = 8.0 Hz, 2H), 3.64 (t, J = 7.6 Hz, 2H), 3.25 (t, J = 7.6 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 140.91, 140.02, 138.08, 129.26, 128.95, 127.50, 127.44, 127.20, 39.19, 33.08.
1-(2-bromoethyl)-4-(trifluoromethyl)benzene (2f) [2]

From 2-(4-(trifluoromethyl)phenyl)ethanol (796 mg, 4.2 mmol), following the general procedure B, the title compound (2f) was obtained (780 mg, 73%) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.62 (d, J = 8.0 Hz, 2H), 7.37 (d, J = 8.0 Hz, 2H), 3.62 (t, J = 7.6 Hz, 2H), 3.26 (t, J = 7.6 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 142.81 (br m), 129.29 (q, J = 32.0 Hz), 129.06, 125.55 (q, J = 3.8 Hz), 124.21 (q, J = 270.1 Hz), 38.88, 32.15. ¹⁹F NMR (376 MHz, CDCl₃) δ -62.46.

1-(2-bromoethyl)-4-(trifluoromethoxy)benzene (2g) [3]

From 2-(4-(trifluoromethoxy)phenyl)acetic acid (2.20 g, 10 mmol), following the general procedure A and B, the title compound (2g) was obtained (1.56 g, 58% in two steps) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.27 (d, J = 8.4 Hz, 2H), 7.21 (d, J = 8.4 Hz, 2H), 3.59 (t, J = 7.6 Hz, 2H), 3.20 (t, J = 7.6 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 148.19, 137.56, 130.05, 121.15, 120.49 (q, J = 256.1 Hz), 38.52, 32.58. ¹⁹F NMR (376 MHz, CDCl₃) δ -57.91.

1-(benzyloxy)-3-(2-bromoethyl)benzene (2j)

From 2-(3-(benzyloxy)phenyl)acetic acid (2.42 g, 10 mmol), following the general procedure A and B, the title compound (2j) was obtained (2.08 g, 71% in two steps) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.52 – 7.37 (m, 5H), 7.30 (t, J = 7.6 Hz, 1H), 6.97 – 6.86 (m, 3H), 5.11 (s, 2H), 3.61 (t, J = 7.6 Hz, 2H), 3.19 (t, J = 7.6 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 159.16, 140.65, 137.11, 129.83, 128.79, 128.18, 127.71, 121.44, 115.61, 113.25, 70.12, 39.60, 32.99. HRMS (EI) calculated for C₁₅H₁₅BrO [M]⁺ 290.0301, measured: 290.0302. IR (neat) 3031, 1592, 1491, 1259, 1018, 740, 696, 626, 540, 448 cm⁻¹.

1-(2-bromoethyl)-2-methylbenzene (2l) [4]

S6
From 2-(o-tolyl)acetic acid (3.00 g, 20 mmol), following the general procedure A and B, the title compound (2l) was obtained (2.20 g, 55% in two steps) as a yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.29 – 7.22 (m, 4H), 3.60 (t, $J$ = 7.6 Hz, 2H), 3.26 (t, $J$ = 7.6 Hz, 2H), 2.42 (s, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 137.21, 136.17, 130.62, 129.40, 127.20, 126.32, 37.04, 31.76, 19.37.

4-(2-bromoethyl)-1,2-dimethoxybenzene (2m) $^5$

From 2-(3,4-dimethoxyphenyl)ethan-1-ol (1.82 g, 10 mmol), following the general procedure B, the title compound (2m) was obtained (2.25 g, 91%) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 6.84 (d, $J$ = 8.0 Hz, 1H), 6.79 – 6.74 (m, 2H), 3.90 (s, 3H), 3.89 (s, 3H), 3.57 (t, $J$ = 7.6 Hz, 2H), 3.12 (t, $J$ = 7.6 Hz, 2H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 148.94, 147.97, 131.51, 120.69, 111.86, 111.23, 55.91, 55.88, 39.10, 33.31.

(4-(2-bromoethyl)phenoxy)triisopropylsilane (2h) $^6$

To a solution of 4-(2-bromoethyl)phenol (1.0 g, 4.97 mmol, 1.0 equiv.) in anhydrous CH$_2$Cl$_2$ (30 mL) was added imidazole (0.36 g, 5.22 mmol, 1.05 equiv.), followed by dropwise addition of TIPSCl (1.06 mL, 4.97 mmol, 1.0 equiv.). After stirring for 16 h, the cloudy reaction mixture was transferred to a separatory funnel and washed with saturated aqueous NH$_4$Cl and H$_2$O. The aqueous phase was extracted twice with CH$_2$Cl$_2$, and the combined extracts were dried over Na$_2$SO$_4$ and concentrated. The crude product was purified by flash column chromatography on silica to afford the title product (2h) (1.30 g, 73%) as a colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.08 (d, $J$ = 8.4 Hz, 2H), 6.86 (d, $J$ = 8.4 Hz, 2H), 3.55 (t, $J$ = 7.6 Hz, 2H), 3.11 (t, $J$ = 7.6 Hz, 2H), 1.33 – 1.23 (m, 3H), 1.13 (d, $J$ = 7.2 Hz, 18H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 155.02, 131.38, 129.59, 119.99, 38.81, 33.36, 17.95, 12.67.
4 Conditions Screening of the Reaction

Table S1

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<th>yield(%)&lt;sup&gt;b&lt;/sup&gt;</th>
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<td>77(72)&lt;sup&gt;c&lt;/sup&gt;</td>
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<td>27</td>
<td>ZnI₂ (1.0 equiv.) as additive</td>
<td>4.5</td>
<td>25</td>
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<sup>a</sup> Reaction conditions: 1a (0.3 mmol), 2a (0.3 mmol), NiCl₂·glyme (10 mol%), Ligand 1 (12 mol%), MgBr₂ (0.3 mmol, 1.0 equiv.) and DMA (4 mL) in an undivided cell with a Ni foam cathode and an iron rod as sacrificial anode. <sup>b</sup> The yields were determined by ¹H-NMR with CH₂Br₂ as the internal standard. <sup>c</sup> Isolated yield of 3a.
<p>| | | | |</p>
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<tr>
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<td>iPr</td>
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5 Photographic Guide for Electrochemical Reductive Relay Cross-Couplings

1 Easily hand-made electrochemical cell
Step 0. Overview of materials used.
From left to right: 1) The iron rod 2) The nickel foam cathode 3) The electrode clamp.
4) copper wire

Step 1. Preparation of two electrodes.
Cut an iron rod about 0.5 × 1.5 cm² with a scissors.
Attach the iron with the electrode clamp.
Strip the protective skin of the copper wire with a tweezer.
Wrap the Ni foam cathode (1.5 × 2.5 cm²) with copper wire.

Materials used in the reaction.

Step 1. Weight the ligand and the hydrocinnamic acid in a 10 mL hydrogenation tube.
Step 2. Transfer the vial to a Nitrogen-filled glove box. Weight the MgBr$_2$ and Nickel catalyst, then install the two electrodes. Remove the tube from the glove box.
Step 3. The alkyl bromide and Boc$_2$O dissolved in anhydrous DMA (4.0 mL) was injected into the tube with a 5 mL syringe. And the tube was sealed with parafilm.

Step 4. After stirring for 30 minutes, attached to electrode (the red (+) to the electrode clamp, the black (-) to the copper wire). The reaction was electrolyzed for 6 h under a constant-current electrolysis at 6 mA.
6 General Procedure for the Electrolysis

To a 10 mL hydrogenation tube charged with a stir bar was added the acids and ligand 1 (6.6 mg, 12 mol%). The vial was then introduced in a Nitrogen-filled glove box. Weight the NiCl$_2$·glyme (6.6 mg, 10 mol%) and MgBr$_2$ (55.2 mg, 1.0 equiv) into the tube and install a Ni foam cathode (2.5 x 1.5 cm$^2$) and an iron rod anode. The tube was then removed from the glove box. Next the Boc$_2$O and the alkyl bromide dissolved in anhydrous DMA (4.0 mL) was injected into the tube with a 5 mL syringe. The reaction mixture was stirred at about 1000 rpm for 30 minutes. After that, the reaction mixture was electrolyzed for 6 h under a constant-current electrolysis at 6 mA. After the reaction was completed, the mixture was diluted with EtOAc (about 40 mL) and washed with sat. NH$_4$Cl (3 x equal volume), dried over Na$_2$SO$_4$, filtered, and concentrated in vacuo.

The crude product was purified by column chromatography to furnish the desired product.

Characterization Data for the Products

1,4-diphenylpentan-3-one (3a) [7]

![Structure of 3a](image)

The title product was prepared according to the general procedure with 3-phenylpropionic acid (1a) (45.0 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3a (51.5 mg, 72%) as a colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.30 (t, $J = 7.2$ Hz, 2H), 7.24 – 7.18 (m, 3H), 7.17 – 7.14 (m, 3H), 7.06 (d, $J = 7.2$ Hz, 2H), 3.70 (q, $J = 6.8$ Hz, 1H), 2.89 – 2.71 (m, 2H), 2.70 – 2.61 (m, 2H), 1.37 (d, $J = 6.8$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 209.9, 141.1, 140.5, 128.9, 128.4, 128.3, 127.9, 127.2, 126.0, 53.2, 42.6, 30.0, 17.3.

4-phenyl-1-(p-tolyl)pentan-3-one (3b)

![Structure of 3b](image)

The title product was prepared according to the general procedure with 3-(p-
(1b) (49.3 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3b ((35.4 mg, 52%) as a colorless oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.21 (t, \(J = 7.2\) Hz, 2H), 7.18 – 7.12 (m, 1H), 7.08 (d, \(J = 6.8\) Hz, 2H), 6.94 (d, \(J = 8.0\) Hz, 2H), 6.87 (d, \(J = 8.0\) Hz, 2H), 3.61 (q, \(J = 6.8\) Hz, 1H), 2.77 – 2.60 (m, 2H), 2.59 – 2.52 (m, 2H), 2.20 (s, 3H), 1.29 (d, \(J = 6.8\) Hz, 3H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 209.0, 139.5, 136.9, 134.4, 128.0, 127.9, 127.1, 126.8, 126.1, 52.2, 41.7, 28.5, 19.9, 16.3. HRMS (EI) calculated for C\(_{18}\)H\(_{20}\)O \([M]^{+}\) 252.1509, measured: 252.1507. IR (neat) 2927, 1711, 1514, 1451, 1259, 1020, 808, 699, 528, 481 cm\(^{-1}\).

1-(4-methoxyphenyl)-4-phenylpentan-3-one (3c) \[^{[8]}\]

\[
\begin{array}{c}
\text{MeO} \\
\text{Ph}
\end{array}
\]

The title product was prepared according to the general procedure with 3-(4-methoxyphenyl)propanoic acid (1c) (54.1 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3c (56.4 mg, 70%) as a pale yellow oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.34 – 7.22 (m, 3H), 7.16 (d, \(J = 7.2\) Hz, 2H), 6.99 (d, \(J = 8.8\) Hz, 2H), 6.77 (d, \(J = 8.4\) Hz, 2H), 3.77 (s, 3H), 3.70 (q, \(J = 6.8\) Hz, 1H), 2.84 – 2.69 (m, 2H), 2.67 – 2.56 (m, 2H), 1.38 (d, \(J = 6.8\) Hz, 3H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 210.0, 157.9, 140.5, 133.1, 129.2, 128.9, 127.9, 127.1, 113.8, 55.2, 53.2, 42.9, 29.1, 17.3.

1-(4-fluorophenyl)-4-phenylpentan-3-one (3d)

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

The title product was prepared according to the general procedure with 3-(4-fluorophenyl)propanoic acid (1d) (50.5 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3d (50.0 mg, 65%) as a yellow oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\)
7.32 – 6.86 (m, 9H), 3.70 (q, \( J = 6.8 \) Hz, 1H), 2.86 – 2.71 (m, 2H), 2.69 – 2.56 (m, 2H), 1.38 (d, \( J = 6.8 \) Hz, 3H). \(^{13}\text{C NMR}\) (101 MHz, CDCl\(_3\)) \( \delta \) 209.7, 161.4 (d, \( J = 243.7 \) Hz), 140.4, 136.7 (d, \( J = 3.2 \) Hz), 129.8 (d, \( J = 7.8 \) Hz), 129.1, 128.0, 127.3, 115.2 (d, \( J = 21.1 \) Hz), 53.3, 42.6, 29.2, 17.4. \(^{19}\text{F NMR}\) (376 MHz, CDCl\(_3\)) \( \delta \) -117.45. HRMS (EI) calculated for C\(_{17}\)H\(_{17}\)FO [M]\(^+\) 256.1258, measured: 256.1257. IR (neat) 2930, 1711, 1508, 1219, 1156, 825, 699, 530, 481 cm\(^{-1}\).

1-(3-fluorophenyl)-4-phenylpentan-3-one (3e)

![Structure 3e](image)

The title product was prepared according to the general procedure with 3-(3-fluorophenyl)propanoic acid (1e) (50.5 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3e (45.0 mg, 58%) as a pale yellow oil. \(^1\text{H NMR}\) (400 MHz, CDCl\(_3\)) \( \delta \) 7.34 – 7.21 (m, 3H), 7.20 – 7.11 (m, 3H), 6.86 – 6.79 (m, 2H), 6.76 – 6.70 (m, 1H), 3.70 (q, \( J = 6.8 \) Hz, 1H), 2.88 – 2.72 (m, 2H), 2.71 – 2.57 (m, 2H), 1.38 (d, \( J = 6.8 \) Hz, 3H). \(^{13}\text{C NMR}\) (101 MHz, CDCl\(_3\)) \( \delta \) 209.4, 162.8 (d, \( J = 245.4 \) Hz), 143.6 (d, \( J = 7.3 \) Hz), 140.3, 129.8 (d, \( J = 8.3 \) Hz), 129.0, 127.9, 127.2, 124.0 (d, \( J = 2.8 \) Hz), 115.1 (d, \( J = 21.0 \) Hz), 112.9 (d, \( J = 21.0 \) Hz), 53.2, 42.1, 29.6, 17.3. \(^{19}\text{F NMR}\) (376 MHz, CDCl\(_3\)) \( \delta \) -113.64. HRMS (EI) calculated for C\(_{17}\)H\(_{17}\)FO [M]\(^+\) 256.1258, measured: 256.1257. IR (neat) 2931, 1712, 1587, 1489, 1449, 1247, 1139, 782, 699, 537 cm\(^{-1}\).

4-phenyl-1-(m-tolyl)pentan-3-one (3f)

![Structure 3f](image)

The title product was prepared according to the general procedure with 3-(m-tolyl)propanoic acid (1f) (49.3 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3f (45.5 mg, 60%) as a yellow oil. \(^1\text{H NMR}\) (400 MHz, CDCl\(_3\)) \( \delta \) 7.32 – 7.27 (m, 2H), 7.25 – 7.21 (m, 1H), 7.17 – 7.15 (m, 2H), 7.12 – 7.08 (m, 1H), 6.96 (d,
$J = 7.2 \text{ Hz, 1H), 6.86 – 6.85 (m, 2H), 3.70 (q, } J = 6.8 \text{ Hz, 1H), 2.85 – 2.70 (m, 2H), 2.69 – 2.61 (m, 2H), 2.27 (s, 3H), 1.37 (d, } J = 6.8 \text{ Hz, 3H).}^{13}\text{C NMR} (101 \text{ MHz, CDCl}_3) \delta 210.0, 141.0, 140.5, 138.0, 129.1, 128.9, 128.3, 127.9, 127.2, 122.8, 125.3, 53.2, 42.7, 29.9, 21.4, 17.4. \text{ HRMS (EI) calculated for C}_{18}\text{H}_{20}\text{O } [\text{M}]^+ 252.1509, \text{ measured: 252.1509. IR (neat) 2928, 1712, 1608, 1492, 1451, 1122, 782, 699, 548 cm}^{-1}.$

1-(benzo[d][1,3]dioxol-5-yl)-4-phenylpentan-3-one (3g)

![1-(benzo[d][1,3]dioxol-5-yl)-4-phenylpentan-3-one](image)

The title product was prepared according to the general procedure with 3-(benzo[d][1,3]dioxol-5-yl)propanoic acid (1g) (58.3 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3g (52.5 mg, 62%) as a pale yellow oil. $^1\text{H NMR} (400 \text{ MHz, CDCl}_3) \delta 7.36 – 7.31 (m, 2H), 7.30 – 7.25 (m, 1H), 7.22 – 7.16 (m, 2H), 6.69 (d, $ J = 7.6 \text{ Hz, 1H}), 6.57 (s, 1H), 6.54 (d, $ J = 8.0 \text{ Hz, 1H}), 5.91 (s, 2H), 3.73 (q, $ J = 6.8 \text{ Hz, 1H}), 2.84 – 2.59 (m, 4H), 1.41 (d, $ J = 6.8 \text{ Hz, 3H}).^{13}\text{C NMR} (101 \text{ MHz, CDCl}_3) \delta 209.8, 147.5, 145.8, 140.4, 134.9, 129.0, 127.9, 127.2, 121.1, 108.8, 108.2, 100.8, 53.3, 42.8, 29.7, 17.3. \text{ HRMS (ESI) calculated for C}_{18}\text{H}_{18}\text{O}_3\text{Na } [\text{M+Na}]^+ 305.1148, \text{ measured: 305.1148. IR (neat) 2892, 1710, 1488, 1442, 1243, 1037, 928, 808, 731, 699, 545 cm}^{-1}.$

1-(4-chloro-3-fluorophenyl)-4-phenylpentan-3-one (3h)

![1-(4-chloro-3-fluorophenyl)-4-phenylpentan-3-one](image)

The title product was prepared according to the general procedure with 3-(4-chloro-3-fluorophenyl)propanoic acid (1h) (60.8 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3h (43.6 mg, 50%) as a pale yellow oil. $^1\text{H NMR} (400 \text{ MHz, CDCl}_3)$
δ 7.34 – 7.25 (m, 3H), 7.21 (t, J = 8.0 Hz, 1H), 7.18 – 7.12 (m, 2H), 6.83 – 6.77 (m, 2H), 3.73 (q, J = 6.8 Hz, 1H), 2.93 – 2.51 (m, 4H), 1.40 (d, J = 6.8 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 209.0, 157.8 (d, J = 248.3 Hz), 142.0 (d, J = 6.5 Hz), 140.0, 130.3, 129.0, 127.8, 127.3, 124.8 (d, J = 3.5 Hz), 118.3 (d, J = 17.6 Hz), 116.5 (d, J = 20.7 Hz), 53.4, 41.8, 29.1, 17.2. ¹⁹F NMR (376 MHz, CDCl₃) δ -115.93. HRMS (EI) calculated for C₁₇H₁₆ClFO [M]+ 290.0868, measured: 290.0870. IR (neat) 2932, 1712, 1580, 1490, 1373, 1242, 1062, 867, 699, 500 cm⁻¹.

1-(furan-2-yl)-4-phenylpentan-3-one (3i)

The title product was prepared according to the general procedure with 3-(furan-2-yl)propanoic acid (1i) (42.0 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3i (43.2 mg, 63%) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.32 (t, J = 7.2 Hz, 2H), 7.27 – 7.24 (m, 1H), 7.23 – 7.21 (m, 1H), 7.21 – 7.16 (m, 2H), 6.26 – 6.17 (m, 1H), 5.87 (d, J = 3.2 Hz, 1H), 3.74 (q, J = 6.8 Hz, 1H), 2.92 – 2.77 (m, 2H), 2.72 – 2.65 (m, 2H), 1.39 (d, J = 6.8 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 209.4, 154.6, 141.0, 140.4, 129.0, 127.9, 127.2, 110.1, 105.1, 53.1, 39.2, 22.3, 17.4. HRMS (EI) calculated for C₁₅H₁₆O₂ [M]+ 228.1145, measured: 228.1143. IR (neat) 2930, 1712, 1598, 1493, 1359, 1147, 1009, 729, 699, 598, 484 cm⁻¹.

4-phenyl-1-(thiophen-2-yl)pentan-3-one (3j)

The title product was prepared according to the general procedure with 3-(thiophen-2-yl)propanoic acid (1j) (46.9 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3j (40.0 mg, 54%) as a pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.34 (t, J = 7.2 Hz, 2H), 7.32 – 7.25 (m, 1H), 7.21 (d, J = 6.8 Hz, 2H), 7.09 (d, J = 5.2 Hz, 1H), 6.93 – 6.84 (m, 1H), 6.71 (d, J = 4.0 Hz, 1H), 3.76 (q, J = 6.8 Hz, 1H), 3.16 – 2.97
(m, 2H), 2.80 – 2.72 (m, 2H), 1.42 (d, $J = 6.8$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 209.3, 143.7, 140.4, 129.0, 127.9, 127.2, 126.8, 124.5, 123.3, 53.2, 42.7, 24.1, 17.4. HRMS (EI) calculated for C$_{15}$H$_{16}$OS [M]$^+$ 244.0916, measured: 244.0916. IR (neat) 2929, 1711, 1493, 1451, 1121, 1030, 847, 824, 694, 544 cm$^{-1}$.

6-(4-methoxyphenyl)-2-phenylhexan-3-one (3k)

![Chemical structure of 6-(4-methoxyphenyl)-2-phenylhexan-3-one (3k)]

The title product was prepared according to the general procedure with 4-(4-methoxyphenyl)butanoic acid (1k) (58.3 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3k (70.3 mg, 83%) as a pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.36 (t, $J = 7.2$ Hz, 2H), 7.32 – 7.26 (m, 1H), 7.26 – 7.21 (m, 2H), 6.98 (d, $J = 8.4$ Hz, 2H), 6.80 (d, $J = 8.4$ Hz, 2H), 3.80 (s, 3H), 3.75 (q, $J = 6.8$ Hz, 3H), 2.54 – 2.35 (m, 4H), 1.90 – 1.73 (m, 2H), 1.41 (d, $J = 6.8$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 210.7, 157.8, 140.7, 133.8, 129.3, 128.9, 127.9, 127.1, 113.7, 55.3, 53.0, 40.2, 34.0, 25.6, 17.5. HRMS (ESI) calculated for C$_{19}$H$_{22}$O$_2$Na [M+Na]$^+$ 305.1512, measured: 305.1511. IR (neat) 2931, 1710, 1510, 1452, 1243, 1176, 1034, 810, 700, 544 cm$^{-1}$.

6-(4-chlorophenyl)-2-phenylhexan-3-one (3l)

![Chemical structure of 6-(4-chlorophenyl)-2-phenylhexan-3-one (3l)]

The title product was prepared according to the general procedure with 4-(4-methoxyphenyl)butanoic acid (1l) (59.6 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3l (61.1 mg, 71%) as a pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.36 (t, $J = 7.2$ Hz, 2H), 7.32 – 7.28 (m, 1H), 7.25 – 7.18 (m, 4H), 6.97 (d, $J = 8.4$ Hz, 2H), 3.75 (q, $J = 6.8$ Hz, 1H), 2.55 – 2.31 (m, 4H), 1.86 – 1.77 (m, 2H), 1.41 (d, $J = 6.8$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 210.4, 140.6, 140.1, 131.5, 129.8, 129.0, 128.4, 127.9, 127.2, 53.1, 39.9, 34.2, 25.1, 17.4. HRMS (EI) calculated for C$_{19}$H$_{19}$ClO [M]$^+$ 286.1119, measured: 286.1119. IR (neat) 2929, 1711,
1491, 1451, 1089, 799, 759, 699, 522 cm\(^{-1}\).

**2,7-diphenylheptan-3-one (3m)**

![2,7-diphenylheptan-3-one](image)

The title product was prepared according to the general procedure with 5-phenylpentanoic acid (1m) (53.5 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3m (58.3 mg, 73%) as a yellow oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.36 (t, J = 7.2 \text{ Hz}, 2\text{H}), 7.31 – 7.27 (\text{m, 3H}), 7.26 – 7.17 (\text{m, 3H}), 7.14 (d, J = 7.2 \text{ Hz}, 2\text{H}), 3.77 (q, J = 6.8 \text{ Hz}, 1\text{H}), 2.55 (t, J = 7.2 \text{ Hz}, 2\text{H}), 2.43 – 2.38 (\text{m, 2H}), 1.62 – 1.47 (\text{m, 4H}), 1.42 (d, J = 6.8 \text{ Hz}, 3\text{H}).\(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta 210.8, 142.3, 140.7, 128.9, 128.4, 128.3, 127.9, 127.2, 125.7, 53.0, 35.7, 30.8, 23.5, 17.5.\) HRMS (EI) calculated for C\(_{19}\)H\(_{22}\)O \([M]^+\) 266.1665, measured: 266.1669. IR (neat) 2930, 1711, 1600, 1493, 1451, 1028, 745, 697, 701, 545 cm\(^{-1}\).

**6-(4-(bis(2-chloroethyl)amino)phenyl)-2-phenylhexan-3-one (3n)**

![6-(4-(bis(2-chloroethyl)amino)phenyl)-2-phenylhexan-3-one](image)

The title product was prepared according to the general procedure with chlorambucil (1n) (91.3 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3n (70.6 mg, 60%) as a yellow oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.37 (t, J = 7.2 \text{ Hz}, 2\text{H}), 7.34 – 7.28 (\text{m, 1H}), 7.27 – 7.22 (\text{m, 2H}), 6.98 (d, J = 8.4 \text{ Hz}, 2\text{H}), 6.61 (d, J = 8.8 \text{ Hz}, 2\text{H}), 3.82 – 3.75 (\text{m, 1H}), 3.72 (t, J = 6.4 \text{ Hz}, 4\text{H}), 3.64 (t, J = 6.4 \text{ Hz}, 4\text{H}), 2.52 – 2.34 (\text{m, 4H}), 1.86 – 1.76 (\text{m, 2H}), 1.42 (d, J = 6.8 \text{ Hz}, 3\text{H}).\(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta 210.7, 144.1, 140.7, 131.0, 129.7, 129.0, 128.0, 127.2, 112.2, 53.7, 53.0, 40.5, 40.3, 33.8, 25.6, 17.5.\) HRMS (EI) calculated for C\(_{22}\)H\(_{27}\)Cl\(_2\)NO \([M]^+\) 391.1464, measured: 391.1467. IR (neat) 2930, 1710, 1614, 1516, 1353, 1179, 802, 741, 701, 545 cm\(^{-1}\).
1-cyclohexyl-4-phenylpentan-3-one (3o)

\[
\begin{align*}
&\text{O} \\
&\text{Ph}
\end{align*}
\]

The title product was prepared according to the general procedure with 3-cyclohexylpropanoic acid (1o) (46.9 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3o (36.7 mg, 50%) as a colorless oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.32 (t, \(J = 7.2\) Hz, 2H), 7.28 – 7.23 (m, 1H), 7.23 – 7.19 (m, 2H), 3.75 (q, \(J = 7.2\) Hz, 1H), 2.46 – 2.25 (m, 2H), 1.68 – 1.49 (m, 5H), 1.38 (d, \(J = 7.2\) Hz, 3H), 1.41 – 1.34 (m, 2H), 1.19 – 0.99 (m, 4H), 0.84 – 0.68 (m, 2H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 211.3, 140.8, 128.9, 127.9, 127.1, 53.0, 38.6, 37.1, 33.2, 32.9, 31.3, 26.5, 26.2, 26.2, 17.5. HRMS (El) calculated for C\(_{17}\)H\(_{24}\)O \([M]^+\) 244.1822, measured: 244.1823. IR (neat) 2920, 2849, 1712, 1493, 1449, 1070, 757, 699, 542 cm\(^{-1}\).

3-phenyl-1-(tetrahydro-2H-pyran-4-yl)butan-2-one (3p)

\[
\begin{align*}
&\text{O} \\
&\text{Ph}
\end{align*}
\]

The title product was prepared according to the general procedure with 2-(tetrahydro-2H-pyran-4-yl)acetic acid (1p) (43.3 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3p (48.7 mg, 70%) as a white solid. m.p. 51-53 °C. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.38 – 7.32 (m, 2H), 7.30 – 7.27 (m, 1H), 7.21 (d, \(J = 6.8\) Hz, 2H), 3.92 – 3.79 (m, 2H), 3.73 (q, \(J = 6.8\) Hz, 1H), 3.42 – 3.30 (m, 2H), 2.30 (d, \(J = 6.4\) Hz, 2H), 2.10 – 1.98 (m, 1H), 1.56 – 1.50 (m, 1H), 1.44 – 1.42 (m, 1H), 1.40 (d, \(J = 6.8\) Hz, 3H), 1.26 – 1.12 (m, 1H), 1.11 – 0.97 (m, 1H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 209.7, 140.3, 129.0, 127.9, 127.3, 67.8, 53.6, 47.8, 32.8, 32.4, 30.7, 17.2. HRMS (ESI) calculated for C\(_{15}\)H\(_{21}\)O\(_2\) \([M+H]^+\) 233.1536, measured: 233.1536. IR (neat) 2927, 2839, 1710, 1493, 1451, 1092, 1013, 852, 760, 700, 545 cm\(^{-1}\).
1-((3r,5r,7r)-adamantan-1-yl)-3-phenylbutan-2-one (3q)

The title product was prepared according to the general procedure with 2-((3r,5r,7r)-adamantan-1-yl)acetic acid (1q) (58.3 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3q (53.0 mg, 63%) as a colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.32 (t, $J = 7.2$ Hz, 2H), 7.27 – 7.22 (m, 1H), 7.21 – 7.16 (m, 2H), 3.70 (q, $J = 6.8$ Hz, 1H), 2.22 (d, $J = 14.4$ Hz, 1H), 1.98 (d, $J = 14.4$ Hz, 1H), 1.92 (brs, 3H), 1.73 – 1.51 (m, 12H), 1.34 (d, $J = 6.8$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 210.2, 140.6, 128.9, 128.1, 127.0, 55.0, 54.5, 42.4, 36.8, 33.6, 28.6, 17.4. HRMS (ESI) calculated for C$_{22}$H$_{26}$ONa [M+Na]$^+$ 305.1876, measured: 305.1871. IR (neat) 2898, 2846, 1707, 1450, 1029, 755, 698, 537 cm$^{-1}$.

6-methyl-2-phenylheptan-3-one (3r)

The title product was prepared according to the general procedure with 4-methylpentanoic acid (1r) (34.9 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3r (42.9 mg, 70%) as a colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.25 (t, $J = 7.2$ Hz, 2H), 7.20 – 7.16 (m, 1H), 7.15 – 7.11 (m, 2H), 3.69 (q, $J = 6.8$ Hz, 1H), 2.27 (t, $J = 8.0$ Hz, 2H), 1.40 – 1.23 (m, 6H), 0.71 (d, $J = 6.0$ Hz, 3H), 0.68 (d, $J = 6.0$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 211.2, 140.8, 128.9, 127.9, 127.1, 53.0, 39.1, 32.8, 27.5, 22.4, 22.1, 17.5. HRMS (EI) calculated for C$_{14}$H$_{20}$O [M]$^+$ 204.1509, measured: 204.1508. IR (neat) 2956, 2870, 1712, 1453, 1136, 757, 700, 548 cm$^{-1}$.
methyl 5-oxo-6-phenylheptanoate (3s)

The title product was prepared according to the general procedure with 5-methoxy-5-oxopentanoic acid (1s) (43.8 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3s (45.6 mg, 65%) as a pale yellow oil. **1H NMR** (400 MHz, CDCl₃) δ 7.32 (t, J = 7.2 Hz, 2H), 7.28 – 7.23 (m, 1H), 7.20 (d, J = 7.2 Hz, 2H), 3.75 (q, J = 6.8 Hz, 1H), 3.60 (s, 3H), 2.42 (t, J = 7.2 Hz, 2H), 2.29 – 2.12 (m, 2H), 1.90 – 1.74 (m, 2H), 1.39 (d, J = 6.8 Hz, 3H). **13C NMR** (101 MHz, CDCl₃) δ 210.0, 173.6, 140.5, 129.0, 127.8, 127.2, 53.0, 51.5, 39.7, 32.9, 18.9, 17.4. **HRMS** (ESI) calculated for C₁₄H₁₈O₃Na [M+Na]⁺ 257.1148, measured: 257.1150. **IR** (neat) 2951, 1733, 1711, 1493, 1451, 1372, 1171, 761, 700, 545 cm⁻¹.

2-phenyloctan-3-one (3t) [¹]

The title product was prepared according to the general procedure with hexanoic acid (1t) (34.9 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3t (33.0 mg, 54%) as a pale yellow oil. **1H NMR** (400 MHz, CDCl₃) δ 7.35 (t, J = 7.6 Hz, 2H), 7.28 (d, J = 7.2 Hz, 1H), 7.23 (d, J = 7.2 Hz, 2H), 3.77 (q, J = 6.8 Hz, 1H), 2.36 (t, J = 7.2 Hz, 2H), 1.59 – 1.45 (m, 2H), 1.41 (d, J = 6.8 Hz, 3H), 1.29 – 1.12 (m, 4H), 0.85 (t, J = 7.2 Hz, 3H). **13C NMR** (101 MHz, CDCl₃) δ 211.1, 140.8, 128.9, 127.9, 127.1, 53.0, 41.0, 31.3, 23.6, 22.4, 17.5, 13.9.

2-phenyloctan-3-one (3u)

The title product was prepared according to the general procedure with decanoic acid
(1u) (51.7 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3u (41.5 mg, 53%) as a yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.25 (t, $J$ = 7.2 Hz, 2H), 7.20 – 7.16 (m, 1H), 7.13 (d, $J$ = 7.2 Hz, 2H), 3.67 (q, $J$ = 7.2 Hz, 1H), 2.26 (t, $J$ = 8.0 Hz, 2H), 1.47 – 1.35 (m, 2H), 1.31 (d, $J$ = 7.2 Hz, 3H), 1.22 – 1.07 (m, 12H), 0.79 (t, $J$ = 6.8 Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 211.1, 140.8, 128.9, 127.9, 127.1, 53.0, 41.1, 31.9, 29.4, 29.3, 29.2, 29.1, 23.9, 22.7, 17.5, 14.1. HRMS (EI) calculated for C$_{18}$H$_{28}$O [M]$^+$ 260.2135, measured: 260.2134. IR (neat) 2923, 2853, 1713, 1493, 1453, 1373, 759, 699, 546 cm$^{-1}$.

1-cyclopropyl-2-phenylpropan-1-one (3v)

![1-cyclopropyl-2-phenylpropan-1-one](image)

The title product was prepared according to the general procedure with cyclopropanecarboxylic acid (1v) (25.8 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3v (26.0 mg, 50%) as a pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.34 (t, $J$ = 7.2 Hz, 2H), 7.30 – 7.21 (m, 3H), 3.90 (q, $J$ = 7.2 Hz, 1H), 1.90 – 1.81 (m, 1H), 1.41 (d, $J$ = 7.2 Hz, 3H), 1.09 – 0.90 (m, 2H), 0.83 – 0.74 (m, 1H), 0.73 – 0.66 (m, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 210.9, 140.9, 128.9, 128.1, 127.0, 53.8, 19.7, 17.6, 11.4, 11.3. HRMS (EI) calculated for C$_{12}$H$_{14}$O [M]$^+$ 174.1039, measured: 174.1039. IR (neat) 2975, 2931, 1689, 1452, 1378, 1041, 1016, 796, 699 cm$^{-1}$.

2-phenyl-1-(2,2,3,3-tetramethylcyclopropyl)propan-1-one (3w)

![2-phenyl-1-(2,2,3,3-tetramethylcyclopropyl)propan-1-one](image)

The title product was prepared according to the general procedure with chrysanthemum acid (1w) (42.7 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3w (47.7 mg, 69%) as a colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.24 (t, $J$ = 7.2 Hz,
2H), 7.18 – 7.12 (m, 1H), 7.11 – 7.06 (m, 2H), 3.70 (q, \( J = 6.8 \) Hz, 1H), 1.30 – 1.26 (m, 4H), 1.17 (s, 3H), 1.00 (s, 3H), 0.90 (s, 3H), 0.74 (s, 3H). \(^{13}\text{C} \text{NMR} \) (101 MHz, CDCl\(_3\)) \( \delta \) 207.7, 140.1, 127.6, 126.9, 125.7, 54.3, 43.1, 33.4, 32.9, 22.8, 21.9, 15.9, 15.4, 15.1. \text{HRMS} \) (ESI) calculated for C\(_{16}\)H\(_{23}\)O [M]^+ 231.1743, measured: 231.1740. \text{IR} \) (neat) 2925, 1683, 1492, 1451, 1378, 1106, 1010, 698, 524 cm\(^{-1}\).

1-cyclobutyl-2-phenylpropan-1-one (3x)

![1-cyclobutyl-2-phenylpropan-1-one](image)

The title product was prepared according to the general procedure with cyclobutanecarboxylic acid (1x) (30.0 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3x (41.8mg, 74%) as a yellow oil. \(^{1}\text{H} \text{NMR} \) (400 MHz, CDCl\(_3\)) \( \delta \) 7.33 (t, \( J = 7.2 \) Hz, 2H), 7.29 – 7.25 (m, 1H), 7.24 – 7.18 (m, 2H), 3.76 (q, \( J = 7.2 \) Hz, 1H), 3.30 (m, 1H), 2.22 – 2.13 (m, 2H), 2.09 (m, 1H), 1.94 – 1.71 (m, 3H), 1.40 (d, \( J = 7.2 \) Hz, 3H). \(^{13}\text{C} \text{NMR} \) (101 MHz, CDCl\(_3\)) \( \delta \) 211.8, 140.7, 128.8, 128.0, 127.0, 50.8, 44.3, 25.4, 24.4, 17.7, 17.6. \text{HRMS} \) (EI) calculated for C\(_{13}\)H\(_{16}\)O [M]^+ 188.1196, measured: 188.1196. \text{IR} \) (neat) 2928, 1706, 1453, 1130, 968, 699, 504 cm\(^{-1}\).

1-(2,3-dihydro-1H-inden-2-yl)-2-phenylpropan-1-one (3y)

![1-(2,3-dihydro-1H-inden-2-yl)-2-phenylpropan-1-one](image)

The title product was prepared according to the general procedure with 2,3-dihydro-1H-indene-2-carboxylic acid (1y) (48.7 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3y (41.3 mg, 55%) as a pale yellow oil. \(^{1}\text{H} \text{NMR} \) (400 MHz, CDCl\(_3\)) \( \delta \) 7.35 (t, \( J = 7.2 \) Hz, 2H), 7.30 – 7.25 (m, 3H), 7.17 – 7.09 (m, 4H), 3.95 (q, \( J = 6.8 \) Hz, 1H), 3.50 (p, \( J = 8.8 \) Hz, 1H), 3.21 – 3.09 (m, 3H), 2.79 – 2.67 (m, 1H), 1.45 (d, \( J = 6.8 \) Hz, 3H). \(^{13}\text{C} \text{NMR} \) (101 MHz, CDCl\(_3\)) \( \delta \) 211.8, 142.0, 141.1, 140.4, 129.0, 128.0, 127.2, 126.6, 126.4, 124.3, 124.2, 52.3, 49.8, 36.5, 35.5, 17.9. \text{HRMS} \) (EI) calculated for C\(_{16}\)H\(_{18}\)O [M]^+ 250.1352, measured: 250.1352. \text{IR} \) (neat)
The title product was prepared according to the general procedure with cyclohexanecarboxylic acid (1z) (38.5 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3z (38.9 mg, 60%) as a pale yellow oil. 

\[ {^1H\text{ NMR}} \ (400 \text{ MHz, CDCl}_3) \delta 7.31 \ (t, J = 7.2 \text{ Hz, } 2\text{H}), \ 7.28 - 7.23 \ (m, 1\text{H}), \ 7.23 - 7.19 \ (m, 2\text{H}), \ 3.90 \ (q, J = 6.8 \text{ Hz, } 1\text{H}), \ 2.45 - 2.35 \ (m, 1\text{H}), \ 1.87 - 1.81 \ (m, 1\text{H}), \ 1.77 - 1.72 \ (m, 1\text{H}), \ 1.69 - 1.58 \ (m, 2\text{H}), \ 1.52 - 1.41 \ (m, 1\text{H}), \ 1.36 \ (d, J = 6.8 \text{ Hz, } 3\text{H}), \ 1.31 - 1.03 \ (m, 5\text{H}). \]

\[ {^{13}C\text{ NMR}} \ (101 \text{ MHz, CDCl}_3) \delta 212.8, \ 139.7, \ 127.8, \ 127.0, \ 126.0, \ 50.1, \ 48.5, \ 28.4, \ 27.3, \ 24.9, \ 24.7, \ 24.3, \ 17.2. \]

\[ \text{HRMS (ESI) calculated for C}_{20}\text{H}_{30}\text{ONa} \ [\text{M+Na}]^+ \ 309.2189, \text{ measured: } 309.2183. \]

\[ \text{IR (neat) 2922, 2852, 1706, 1449, 1374, 951, 729, 698, 555 cm}^{-1}. \]
methyl (1r,4r)-4-(2-phenylpropanoyl)cyclohexane-1-carboxylate (3ab)

The title product was prepared according to the general procedure with trans-4-(methoxycarbonyl)cyclohexane-1-carboxylic acid (1ab) (55.9 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3ab (57.6 mg, 70%) as a yellow oil.

\[ \text{1H NMR (400 MHz, CDCl}_3 \text{ δ 7.34 (t, J = 7.2 Hz, 2H), 7.25 – 7.23 (m, 1H), 7.25 – 7.19 (m, 2H), 3.91 (q, J = 6.8 Hz, 1H), 3.65 (s, 3H), 2.50 – 2.36 (m, 1H), 2.34 – 2.20 (m, 1H), 2.05 – 2.01 (m, 1H), 1.99 – 1.91 (m, 2H), 1.58 – 1.49 (m, 1H), 1.44 – 1.24 (m, 7H).} \]

\[ \text{13C NMR (101 MHz, CDCl}_3 \text{ δ 213.2, 176.0, 140.4, 128.9, 128.0, 127.2, 51.6, 51.5, 48.4, 42.4, 28.5, 28.3, 27.8, 27.4, 18.0. HRMS (ESI) calculated for C}_{17}\text{H}_{22}\text{O}_3\text{Na [M+Na}^+ \text{] 297.1461, measured: 297.1462. IR (neat) 2933, 2861, 1731, 1704, 1451, 1247, 1017, 896, 730, 699, 555 cm}^{-1}. \]

1-(4-(4-chlorophenyl)cyclohexyl)-2-phenylpropan-1-one (3ac)

The title product was prepared according to the general procedure with 4-(4-chlorophenyl)cyclohexane-1-carboxylic acid (1ac) (71.6 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3ac (78.4 mg, 80%) as a pale yellow solid. m.p. 59-61 °C. \[ \text{1H NMR (400 MHz, CDCl}_3 \text{ δ 7.37 (t, J = 7.2 Hz, 2H), 7.31 – 7.23 (m, 5H), 7.10 (d, J = 8.4 Hz, 2H), 3.96 (q, J = 6.8 Hz, 1H), 2.57 – 2.41 (m, 2H), 2.08 – 1.99 (m, 1H), 1.98 – 1.91 (m, 1H), 1.89 – 1.79 (m, 1H), 1.70 – 1.55 (m, 3H), 1.51 – 1.39 (m, 5H).} \]

\[ \text{13C NMR (101 MHz, CDCl}_3 \text{ δ 213.5, 145.4, 140.5, 131.6, 128.9, 128.4, 128.1, 128.0, 127.2, 51.5, 48.8, 43.0, 33.6, 33.0, 29.7, 28.6, 18.1. HRMS (EI) \]

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calculated for C$_{21}$H$_{23}$ClO $[\text{M}]^+$ 326.1432, measured: 326.1432. \textbf{IR} (neat) 2927, 2854, 1704, 1492, 1449, 1091, 972, 822, 699, 530 cm$^{-1}$.

2-phenyl-1-(tetrahydro-2H-pyran-4-yl)propan-1-one (3ad)

![Structure of 2-phenyl-1-(tetrahydro-2H-pyran-4-yl)propan-1-one (3ad)](image)

The title product was prepared according to the general procedure with tetrahydro-2H-pyran-4-carboxylic acid (1ad) (39.0 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3ad (37.9 mg, 58%) as a pale yellow oil. \textbf{\textsuperscript{1}H NMR} (400 MHz, CDCl$_3$) $\delta$ 7.53 – 7.11 (m, 5H), 4.00 – 3.85 (m, 3H), 3.42 – 3.31 (m, 1H), 3.31 – 3.20 (m, 1H), 2.70 – 2.57 (m, 1H), 1.79 – 1.58 (m, 3H), 1.39 (d, $J = 6.8$ Hz, 3H), 1.37 – 1.29 (m, 1H).

\textbf{\textsuperscript{13}C NMR} (101 MHz, CDCl$_3$) $\delta$ 211.8, 140.3, 129.0, 127.9, 127.2, 67.3, 67.0, 51.0, 46.1, 29.0, 28.1, 18.2. \textbf{HRMS} (ESI) calculated for C$_{14}$H$_{19}$O$_2$ [M+H]$^+$ 219.1380, measured: 219.1380. \textbf{IR} (neat) 2950, 2844, 1705, 1449, 1115, 1088, 1016, 732, 699, 559, 506 cm$^{-1}$.

\textit{tert}-butyl 4-(2-phenylpropanoyl)piperidine-1-carboxylate (3ae)

![Structure of tert-butyl 4-(2-phenylpropanoyl)piperidine-1-carboxylate (3ae)](image)

The title product was prepared according to the general procedure with 1-(\textit{tert}-butoxycarbonyl)piperidine-4-carboxylic acid (1ae) (68.8 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3ae (57.2 mg, 60%) as a yellow solid. m.p. 77-79 $^\circ$C. \textbf{\textsuperscript{1}H NMR} (400 MHz, CDCl$_3$) $\delta$ 7.35 – 7.30 (m, 2H), 7.29 – 7.25 (m, 1H), 7.23 – 7.17 (m, 2H), 4.20 – 3.96 (m, 2H), 3.90 (q, $J = 6.8$ Hz, 1H), 2.68 (t, $J = 11.8$ Hz, 1H), 2.59 – 2.47 (m, 2H), 1.84 – 1.71 (m, 1H), 1.59 – 1.46 (m, 3H), 1.43 (s, 9H), 1.37 (d, $J = 6.8$ Hz, 3H). \textbf{\textsuperscript{13}C NMR} (101 MHz, CDCl$_3$) $\delta$ 212.2, 154.7, 140.3, 129.1, 128.0, 127.3, 79.6, 51.3, 47.2, 43.2 (br, NCH$_2$), 28.5, 27.6, 18.2. \textbf{HRMS} (ESI) calculated for C$_{19}$H$_{27}$NO$_3$Na [M+Na]$^+$ 340.1883, measured: 340.1881. \textbf{IR} (neat) 2930, 1688, 1450, 1374, 1095, 1017, 857, 682 cm$^{-1}$. 

\textbf{IR} (neat) 2930, 2857, 1705, 1504, 1475, 1449, 1380, 1238, 1123, 1088, 1020, 762, 699, 532 cm$^{-1}$. 

\textbf{IR} (neat) 2927, 2854, 1704, 1492, 1449, 1091, 972, 822, 699, 530 cm$^{-1}$. 

\textbf{IR} (neat) 2950, 2844, 1705, 1449, 1115, 1088, 1016, 732, 699, 559, 506 cm$^{-1}$.
1421, 1233, 1168, 1012, 972, 701, 555 cm⁻¹.

1-(1-benzylopyrrolidine-4-yl)-2-phenylpropan-1-one (3af)

The title product was prepared according to the general procedure with 1-benzylopyrrolidine-4-carboxylic acid (1af) (70.0 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3af (61.7 mg, 64%) as a yellow oil.

$^1$H NMR (400 MHz, CDCl₃) δ 7.34 – 7.23 (m, 7H), 7.21 – 7.16 (m, 1H), 7.15 – 7.11 (m, 2H), 4.51 (br, 1H), 3.84 – 3.81 (m, 1H), 3.59 (br, 1H), 2.76 – 2.55 (br m, 3H), 1.86 – 1.74 (m, 1H), 1.52 (br, 3H), 1.31 (d, $J = 6.8$ Hz, 3H).

$^{13}$C NMR (101 MHz, CDCl₃) δ 210.6, 169.3, 139.1, 135.0, 128.6, 128.0, 127.4, 126.9, 126.3, 125.8, 50.4, 45.9, 40.6 (br m), 27.3 (br m), 17.1. HRMS (ESI) calculated for C₂₁H₂₄NO₂ [M+H]⁺ 322.1802, measured: 322.1796. IR (neat) 2947, 2859, 1704, 1626, 1430, 1278, 972, 789, 700, 556 cm⁻¹.

2-phenyl-1-(1-tosylpyrrolidine-4-yl)propan-1-one (3ag)

The title product was prepared according to the general procedure with 1-tosylpyrrolidine-4-carboxylic acid (1ag) (85.0 mg, 0.3 mmol) and (2-bromoethyl)benzene (2a) (55.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 3ag (71.3 mg, 64%) as a white solid. m.p. 151-153 °C. $^1$H NMR (400 MHz, CDCl₃) δ 7.60 (d, $J = 8.4$ Hz, 2H), 7.36 – 7.21 (m, 5H), 7.14 (d, $J = 6.8$ Hz, 2H), 3.84 (q, $J = 6.8$ Hz, 1H), 3.75 – 3.68 (m, 1H), 3.66 – 3.59 (m, 1H), 2.43 (s, 3H), 2.36 – 2.24 (m, 2H), 2.22 – 2.13 (m, 1H), 1.93 – 1.85 (m, 1H), 1.78 – 1.61 (m, 2H), 1.51 – 1.45 (m, 1H), 1.34 (d, $J = 6.8$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl₃) δ 211.4, 143.6, 140.1, 133.0, 129.7, 129.1, 127.9, 127.7, 127.3, 51.3, 45.8, 45.7, 45.3, 27.9, 27.0, 21.6, 18.1. HRMS (DART) calculated for C₂₁H₂₆NSO₂
The title product was prepared according to the general procedure with 3-phenylpropionic acid (1a) (45.0 mg, 0.3 mmol) and 1-(2-bromoethyl)-4-methoxybenzene (2b) (64.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 4a (33.8 mg, 42%) as a pale yellow oil. **1H NMR** (400 MHz, CDCl₃) δ 7.29 – 7.23 (m, 2H), 7.22 – 7.16 (m, 1H), 7.13 – 7.07 (m, 4H), 6.86 (d, J = 8.4 Hz, 2H), 3.82 (s, 3H), 3.68 (q, J = 7.2 Hz, 1H), 2.94 – 2.61 (m, 4H), 1.38 (d, J = 7.2 Hz, 3H). **13C NMR** (101 MHz, CDCl₃) δ 210.2, 158.7, 141.1, 132.5, 128.9, 128.4, 128.3, 126.0, 114.3, 55.3, 52.3, 42.5, 30.0, 17.4.

**4-(4-chlorophenyl)-1-phenylpentan-3-one (4b)**

The title product was prepared according to the general procedure with 3-phenylpropionic acid (1a) (45.0 mg, 0.3 mmol) and 1-(2-bromoethyl)-4-chlorobenzene (2c) (65.9 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 4b (49.8 mg, 61%) as a colorless oil. **1H NMR** (400 MHz, CDCl₃) δ 7.33 – 7.17 (m, 5H), 7.13 – 7.07 (m, 4H), 3.71 (q, J = 7.2 Hz, 1H), 2.93 – 2.78 (m, 2H), 2.73 – 2.64 (m, 2H), 1.39 (d, J = 7.2 Hz, 3H). **13C NMR** (101 MHz, CDCl₃) δ 209.4, 140.9, 138.8, 133.1, 129.2, 129.1, 128.5, 128.3, 126.1, 52.5, 42.7, 29.9, 17.4. **HRMS** (EI) calculated for C₁₇H₁₇ClO [M]+ 272.0962, measured: 272.0963. **IR** (neat) 2930, 1712, 1490, 1453, 1091, 1013, 830, 748, 698, 505 cm⁻¹.
4-(4-fluorophenyl)-1-phenylpentan-3-one (4c)\textsuperscript{[7]}

![Chemical structure of 4-(4-fluorophenyl)-1-phenylpentan-3-one (4c)](image)

The title product was prepared according to the general procedure with 3-phenylpropionic acid (1a) (45.0 mg, 0.3 mmol) and 1-(2-bromoethyl)-4-fluorobenzene (2d) (60.9 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 4c (43.8 mg, 57%) as a pale yellow oil. \textbf{1H NMR} (400 MHz, CDCl\textsubscript{3}) \(\delta\) 7.19 – 7.12 (m, 2H), 7.10 – 7.06 (m, 1H), 7.05 – 6.96 (m, 4H), 6.93 – 6.86 (m, 2H), 3.61 (q, \(J = 7.2\) Hz, 1H), 2.81 – 2.66 (m, 2H), 2.63 – 2.55 (m, 2H), 1.28 (d, \(J = 7.2\) Hz, 3H). \textbf{13C NMR} (101 MHz, CDCl\textsubscript{3}) \(\delta\) 209.6, 162.0 (d, \(J = 245.7\) Hz), 140.9, 136.1 (d, \(J = 3.3\) Hz), 129.4 (d, \(J = 8.0\) Hz), 128.4, 128.3, 126.1, 115.8 (d, \(J = 21.4\) Hz), 52.3, 42.6, 29.9, 17.5. \textbf{19F NMR} (376 MHz, CDCl\textsubscript{3}) \(\delta\) -115.52.

4-([1,1'-biphenyl]-4-yl)-1-phenylpentan-3-one (4d)\textsuperscript{[7]}

![Chemical structure of 4-([1,1'-biphenyl]-4-yl)-1-phenylpentan-3-one (4d)](image)

The title product was prepared according to the general procedure with 3-phenylpropionic acid (1a) (45.0 mg, 0.3 mmol) and 4-(2-bromoethyl)-1,1'-biphenyl (2e) (78.4 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 4d (58.4 mg, 62%) as a white solid. \textbf{1H NMR} (400 MHz, CDCl\textsubscript{3}) \(\delta\) 7.62 (d, \(J = 7.2\) Hz, 2H), 7.57 (d, \(J = 8.4\) Hz, 2H), 7.48 (t, \(J = 7.6\) Hz, 2H), 7.39 (t, \(J = 7.2\) Hz, 1H), 7.31 – 7.23 (m, 4H), 7.23 – 7.17 (m, 1H), 7.12 (d, \(J = 7.2\) Hz, 2H), 3.79 (q, \(J = 6.8\) Hz, 1H), 2.99 – 2.62 (m, 4H), 1.46 (d, \(J = 6.8\) Hz, 3H). \textbf{13C NMR} (101 MHz, CDCl\textsubscript{3}) \(\delta\) 209.9, 141.1, 140.7, 140.1, 139.4, 128.8, 128.4, 128.3, 128.3, 127.7, 127.4, 127.1, 126.1, 52.9, 42.7, 30.0, 17.4.
1-phenyl-4-(4-(trifluoromethyl)phenyl)pentan-3-one (4e) \[\text{[7]}\]

![Chemical Structure]

The title product was prepared according to the general procedure with 3-phenylpropionic acid (1a) (45.0 mg, 0.3 mmol) and 1-(2-bromoethyl)-4-(trifluoromethyl)benzene (2f) (75.9 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 4e (51.0 mg, 64%) as a pale yellow oil. 

1\text{H NMR} (400 MHz, CDCl\textsubscript{3}) \(\delta 7.45\) (d, \(J = 8.4\) Hz, 2H), \(7.20 – 7.05\) (m, 5H), \(6.97\) (d, \(J = 7.2\) Hz, 2H), 3.69 (q, \(J = 6.8\) Hz, 1H), 2.83 – 2.68 (m, 2H), 2.60 (t, \(J = 7.6\) Hz, 2H), 1.31 (d, \(J = 6.8\) Hz, 3H). 

13\text{C NMR} (101 MHz, CDCl\textsubscript{3}) \(\delta 208.8, 144.2, 140.7, 129.5\) (q, \(J = 32.5\) Hz), 128.4, 128.3, 128.2, 126.1, 125.9 (q, \(J = 3.8\) Hz), 124.1 (q, \(J = 272.0\) Hz), 53.0, 42.8, 29.8, 17.4. 

19\text{F NMR} (376 MHz, CDCl\textsubscript{3}) \(\delta -62.52\). 

HRMS (EI) calculated for C\textsubscript{18}H\textsubscript{17}F\textsubscript{3}O\textsubscript{2} [M]\textsuperscript{+} 322.1175, measured: 322.1176. 

IR (neat) 2932, 1706, 1509, 1449, 1263, 1244, 1148, 1025, 811, 690, 552 cm\textsuperscript{-1}.

1-phenyl-4-(4-(trifluoromethoxy)phenyl)pentan-3-one (4f)

![Chemical Structure]

The title product was prepared according to the general procedure with 3-phenylpropionic acid (1a) (45.0 mg, 0.3 mmol) and 1-(2-bromoethyl)-4-(trifluoromethoxy)benzene (2g) (80.7 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 4f (54.7 mg, 56%) as a colorless oil. 

1\text{H NMR} (400 MHz, CDCl\textsubscript{3}) \(\delta 7.28 – 7.23\) (m, 2H), \(7.22 – 7.13\) (m, 5H), 7.09 (d, \(J = 7.2\) Hz, 2H), 3.75 (q, \(J = 6.8\) Hz, 1H), 2.91 – 2.79 (m, 2H), 2.75 – 2.67 (m, 2H), 1.40 (d, \(J = 6.8\) Hz, 3H). 

13\text{C NMR} (101 MHz, CDCl\textsubscript{3}) \(\delta 209.3, 148.3\) (d, \(J = 1.8\) Hz), 140.8, 139.0, 129.2, 128.4, 128.3, 126.1, 121.4, 120.5 (q, \(J = 257.1\) Hz), 52.4, 42.7, 29.9, 17.5. 

19\text{F NMR} (376 MHz, CDCl\textsubscript{3}) \(\delta -57.86\). 

HRMS (EI) calculated for C\textsubscript{18}H\textsubscript{17}F\textsubscript{3}O\textsubscript{2} [M]\textsuperscript{+} 322.1175, measured: 322.1176.
1-phenyl-4-((triisopropylsilyl)oxy)phenylpentan-3-one (4g)

The title product was prepared according to the general procedure with 3-phenylpropionic acid (1a) (45.0 mg, 0.3 mmol) and (4-(2-bromoethyl)phenoxy)triisopropylsilane (2h) (107.2 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 4g (67.8 mg, 55%) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.30 – 7.23 (m, 2H), 7.18 (t, J = 7.2 Hz, 1H), 7.09 (d, J = 7.2 Hz, 2H), 7.03 (d, J = 8.4 Hz, 2H), 6.84 (d, J = 8.4 Hz, 2H), 3.67 (q, J = 6.8 Hz, 1H), 2.91 – 2.74 (m, 2H), 2.73 – 2.58 (m, 2H), 1.38 (d, J = 6.8 Hz, 3H), 1.33 – 1.22 (m, 3H), 1.13 (d, J = 7.2 Hz, 18H). ¹³C NMR (101 MHz, CDCl₃) δ 210.3, 155.2, 142.3, 140.8, 134.7, 130.2, 128.5, 128.3, 128.1, 127.4, 126.1, 126.1, 52.8, 42.8, 29.9, 17.3. HRMS (EI) calculated for C₂₆H₃₈O₂Si [M]⁺ 410.2636, measured: 410.2635. IR (neat) 2943, 2866, 1713, 1508, 1262, 882, 837, 683, 553 cm⁻¹.

4-(3-chlorophenyl)-1-phenylpentan-3-one (4h)

The title product was prepared according to the general procedure with 3-phenylpropionic acid (1a) (45.0 mg, 0.3 mmol) and 1-(2-bromoethyl)-3-chlorobenzene (2i) (65.9 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 4h (49.0 mg, 60%) as a pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.29 – 7.23 (m, 4H), 7.22 – 7.17 (m, 2H), 7.11 (d, J = 6.8 Hz, 2H), 7.07 – 7.01 (m, 1H), 3.70 (q, J = 6.8 Hz, 1H), 2.94 – 2.79 (m, 2H), 2.74 – 2.67 (m, 2H), 1.39 (d, J = 6.8 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 209.1, 142.3, 140.8, 134.7, 130.2, 128.5, 128.3, 128.1, 127.4, 126.1, 126.1, 52.8, 42.8, 29.9, 17.3. HRMS (EI) calculated for C₁₇H₁₇ClO [M]⁺ 272.0962, measured: 272.0964. IR (neat) 2929, 1713, 1593, 1453, 1080, 780, 748, 695, 553, 443 cm⁻¹.
4-(3-(benzyloxy)phenyl)-1-phenylpentan-3-one (4i)

![Chemical structure of 4-(3-(benzyloxy)phenyl)-1-phenylpentan-3-one](image)

The title product was prepared according to the general procedure with 3-phenylpropionic acid (1a) (45.0 mg, 0.3 mmol) and 1-(benzyloxy)-3-(2-bromoethyl)benzene (2j) (87.4 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 4i (51.7 mg, 50%) as a pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.51 (d, $J = 7.2$ Hz, 2H), 7.46 (t, $J = 7.2$ Hz, 2H), 7.42 – 7.37 (m, 1H), 7.35 – 7.26 (m, 3H), 7.26 – 7.20 (m, 1H), 7.15 (d, $J = 7.2$ Hz, 2H), 6.95 (d, $J = 8.0$ Hz, 1H), 6.86 (d, $J = 7.6$ Hz, 2H), 5.09 (s, 2H), 3.75 (q, $J = 7.2$ Hz, 1H), 2.98 – 2.83 (m, 2H), 2.80 – 2.66 (m, 2H), 1.45 (d, $J = 7.2$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 209.7, 159.2, 142.1, 141.1, 136.9, 130.1, 128.7, 128.5, 128.4, 128.1, 127.7, 126.1, 120.7, 114.5, 113.5, 70.0, 53.2, 42.5, 30.0, 17.3. HRMS (EI) calculated for C$_{24}$H$_{24}$O$_2$ [M$^+$] $^{344.1771}$, measured: 344.1772. IR (neat) 2930, 1710, 1581, 1451, 1258, 1156, 1025, 735, 695, 554, 456 cm$^{-1}$.

4-(2,3-dihydrobenzofuran-5-yl)-1-phenylpentan-3-one (4j)

![Chemical structure of 4-(2,3-dihydrobenzofuran-5-yl)-1-phenylpentan-3-one](image)

The title product was prepared according to the general procedure with 3-phenylpropionic acid (1a) (45.0 mg, 0.3 mmol) and 5-(2-bromoethyl)-2,3-dihydrobenzofuran (2k) (68.1 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 4j (38.0 mg, 39%) as a colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.21 – 7.12 (m, 2H), 7.07 (t, $J = 7.2$ Hz, 1H), 6.99 (d, $J = 7.2$ Hz, 2H), 6.87 (s, 1H), 6.82 (d, $J = 8.0$ Hz, 1H), 6.62 (d, $J = 8.0$ Hz, 1H), 4.47 (t, $J = 8.8$ Hz, 2H), 3.55 (q, $J = 7.2$ Hz, 1H), 3.07 (t, $J = 8.8$ Hz, 2H), 2.82 – 2.68 (m, 2H), 2.67 – 2.51 (m, 2H), 1.26 (d, $J = 7.2$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 210.3,
6-(4-methoxyphenyl)-2-(4-(trifluoromethyl)phenyl)hexan-3-one (4k)

The title product was prepared according to the general procedure with 4-(4-methoxyphenyl)butanoic acid (1k) (58.3 mg, 0.3 mmol) and 1-(2-bromoethyl)-4-(trifluoromethyl)benzene (2f) (75.9 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 4k (51.0 mg, 60%) as a pale yellow oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.61 (d, $J = 8.0$ Hz, 2H), 7.35 (d, $J = 8.0$ Hz, 2H), 6.97 (d, $J = 8.8$ Hz, 2H), 6.80 (d, $J = 8.8$ Hz, 2H), 3.86 – 3.81 (m, 1H), 3.80 (s, 3H), 2.57 – 2.42 (m, 2H), 2.39 (t, $J = 7.2$ Hz, 2H), 1.88 – 1.78 (m, 2H), 1.43 (d, $J = 6.8$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 209.7, 157.9, 144.6, 133.5, 129.5 (q, $J = 32.4$ Hz), 129.3, 128.3, 125.8 (q, $J = 3.7$ Hz), 124.1 (q, $J = 272.1$ Hz), 113.8, 55.2, 52.7, 40.4, 33.9, 25.3, 17.5. $^{19}$F NMR (376 MHz, CDCl$_3$) $\delta$ -62.47. HRMS (EI) calculated for C$_{20}$H$_{21}$F$_3$O$_2$ [M]$^+$ 350.1488, measured: 350.1486. IR (neat) 2925, 1714, 1613, 1511, 1323, 1244, 1163, 1119, 1068, 840, 605 cm$^{-1}$.

6-(4-methoxyphenyl)-2-(o-tolyl)hexan-3-one (4l)

The title product was prepared according to the general procedure with 4-(4-methoxyphenyl)butanoic acid (1k) (58.3 mg, 0.3 mmol) and 1-(2-bromoethyl)-2-methylbenzene (2l) (59.7 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 4l (43.0 mg, 48%) as a colorless oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.24 – 7.17 (m, 3H), 7.10 – 7.03 (m, 1H), 6.99 (d, $J = 8.8$ Hz, 2H), 6.80 (d, $J = 8.8$ Hz, 2H), 3.94 (q, $J = 7.2$ Hz, 1H), 3.80 (s, 3H), 2.61 – 2.46 (m, 1H), 2.45 – 2.40 (m, 1H), 2.38 (s, 3H), 2.35 – 2.27 (m, 2H), 1.87 – 1.74 (m, 2H), 1.36 (d, $J = 7.2$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 211.1, 157.8, 139.2, 135.8,
133.8, 130.9, 129.3, 126.99, 126.98, 126.7, 113.7, 55.3, 48.9, 40.1, 34.1, 25.7, 19.8, 16.9. **HRMS (EI)** calculated for C_{20}H_{24}O_{2} [M]+ 296.1771, measured: 296.1775. **IR** (neat) 2930, 1710, 1611, 1510, 1454, 1243, 1176, 1034, 811, 758, 728, 554, 455 cm^{-1}.

2-(3,4-dimethoxyphenyl)-6-(4-methoxyphenyl)hexan-3-one (4m)

![Structure of 2-(3,4-dimethoxyphenyl)-6-(4-methoxyphenyl)hexan-3-one](image)

The title product was prepared according to the general procedure with 4-(4-methoxyphenyl)butanoic acid (1k) (58.3 mg, 0.3 mmol) and 4-(2-bromoethyl)-1,2-dimethoxybenzene (2m) (73.5 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 4m (41.5 mg, 40%) as a white solid. m.p. 75-77 °C. **^1H NMR** (400 MHz, CDCl₃) δ 6.98 (d, J = 8.4 Hz, 2H), 6.86 – 6.75 (m, 4H), 6.70 (s, 1H), 3.89 (s, 3H), 3.87 (s, 3H), 3.78 (s, 3H), 3.68 (q, J = 7.2 Hz, 1H), 2.54 – 2.30 (m, 4H), 1.88 – 1.77 (m, 2H), 1.38 (d, J = 7.2 Hz, 3H). **^13C NMR** (101 MHz, CDCl₃) δ 211.0, 157.8, 149.2, 148.2, 133.7, 133.1, 129.3, 120.1, 113.7, 111.4, 110.7, 55.9, 55.2, 52.5, 39.9, 34.0, 25.6, 17.5. **HRMS (DART)** calculated for C_{21}H_{27}O_{4} [M]+ 343.1904, measured: 343.1902. **IR** (neat) 2921, 1714, 1507, 1454, 1255, 1209, 1158, 1018, 747, 698, 549 cm⁻¹.

1-(4-methoxyphenyl)-5-phenylheptan-4-one (4n)

![Structure of 1-(4-methoxyphenyl)-5-phenylheptan-4-one](image)

The title product was prepared according to the general procedure with 4-(4-methoxyphenyl)butanoic acid (1k) (58.3 mg, 0.3 mmol) and (3-bromopropyl)benzene (2n) (59.7 mg, 0.3 mmol). The crude material was purified by flash column chromatography on silica to afford 4n (46.3 mg, 52%) as a yellow oil. **^1H NMR** (400 MHz, CDCl₃) δ 7.35 (t, J = 7.2 Hz, 2H), 7.32 – 7.28 (m, 1H), 7.22 (d, J = 6.8 Hz, 2H), 6.97 (d, J = 8.4 Hz, 2H), 6.80 (d, J = 8.4 Hz, 2H), 3.80 (s, 3H), 3.52 (t, J = 7.2 Hz, 1H), 2.54 – 2.32 (m, 4H), 2.18 – 2.01 (m, 1H), 1.88 – 1.77 (m, 2H), 1.77 – 1.68 (m, 1H), ...
0.84 (t, $J = 7.2$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 210.5, 157.8, 139.0, 133.8, 129.3, 128.8, 128.4, 127.2, 113.7, 60.9, 55.3, 41.0, 34.0, 25.5, 25.3, 12.2. HRMS (EI) calculated for C$_{20}$H$_{24}$O$_2$ [M]$^+$ 296.1771, measured: 296.1772. IR (neat) 2925, 1708, 1611, 1510, 1453, 1243, 1033, 807, 755, 699, 544 cm$^{-1}$. 
7 Mechanism Experiments

7.1 Cyclic Voltammetry

Cyclic voltammograms were recorded with a CHI660E potentiostat at room temperature in DMAc. \( \text{Bu}_4\text{NBr} \) (0.1 M) was used as the supporting electrolyte, and a Glass Carbon electrode was used as the working electrode. The auxiliary electrode was a Pt sheet. All potentials are referenced against the Ag/AgNO\(_3\) redox couple. The scan rate was 100 mV s\(^{-1}\).

**Figure S1:** Photograph of setup used for cyclic voltammetry.

**Figure S2:** Cyclic voltammograms recorded on a glassy carbon electrode at 100 mVs\(^{-1}\) in: (a) DMA containing 0.1 M of \( \text{Bu}_4\text{NBr} \); (b) solution (a) with 10 mM of NiCl\(_2\)-glyme added; (c) solution (a) with 10 mM of 2,9-Dimethyl-1,10-phenanthroline (Ligand 2) added; (d) solution (a) with 10 mM of (2-Bromoethyl)benzene (2a) added; (e) solution (a) with 10 mM of 3-phenylpropanoic anhydride.
added.

**Figure S3.** Cyclic voltammograms recorded on a glassy carbon electrode at 100 mVs$^{-1}$ in: (a) DMA containing 0.1 M of $n$Bu$_4$NBr; (b) solution (a) with 7.5 mM of NiCl$_2$·glyme and 2,9-Dimethyl-1,10-phenanthroline (Ni/L = 1/1) added; (c) solution (b) with 10 mM of 2a added;

**Figure S4.** Cyclic voltammograms recorded on a glassy carbon electrode at 100 mVs$^{-1}$ in: (a) DMA containing 0.1 M of $n$Bu$_4$NBr; (b) solution (a) with 7.5 mM of NiCl$_2$·glyme and 2,9-Dimethyl-1,10-phenanthroline (Ni/L = 1/1) added; (c) solution (b) with 10 mM of 3-phenylpropanoic anhydride added.
7.2 Deuterium-labeled Experiments

To gain further insight into this electrochemical reductive relay cross-coupling system, deuterium-labeled 2a–D and 2a–D’ were prepared and subjected to the reactions. As shown in Scheme S1a, 91% deuterium incorporation was observed in the methyl group of 3a–D and the H/D scrambling between methyl and benzyl groups was not observed in Scheme S1b. These results indicate that the styrene intermediate may be generated in the migratory process and the β–H elimination/reductive elimination sequence is irreversible in the formation of product 3a–D or 3a–D’.

**Scheme S1.** Deuterium-labeled Experiments
8 Reference

9 Spectra of Compounds

$^1$H NMR Spectrum of 3a (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 3a (CDCl$_3$, 101 MHz)
$^1$H NMR Spectrum of 3a-D (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 3a-D (CDCl$_3$, 101 MHz)
$^{1}H$ NMR Spectrum of 3a-D’ (CDCl$_{3}$, 400 MHz)

$^{13}C$ NMR Spectrum of 3a-D’ (CDCl$_{3}$, 101 MHz)
$^1$H NMR Spectrum of 3b (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 3b (CDCl$_3$, 101 MHz)
$^1$H NMR Spectrum of 3c (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 3c (CDCl$_3$, 101 MHz)
$^1$H NMR Spectrum of 3d (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 3d (CDCl$_3$, 101 MHz)
$^{19}$F NMR Spectrum of 3d (CDCl$_3$, 376 MHz)

$^1$H NMR Spectrum of 3e (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3e (CDCl$_3$, 101 MHz)

$^{19}$F NMR Spectrum of 3e (CDCl$_3$, 376 MHZ)
$^1$H NMR Spectrum of 3f (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 3f (CDCl$_3$, 101 MHz)
$^1$H NMR Spectrum of 3g (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 3g (CDCl$_3$, 101 MHz)
$^1$H NMR Spectrum of 3h (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 3h (CDCl$_3$, 101 MHz)
$^{19}$F NMR Spectrum of 3h (CDCl$_3$, 376 MHZ)

$^1$H NMR Spectrum of 3i (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3i (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3j (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3j (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3k (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3k (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3l (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3l (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3m (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3m (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3n (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3n (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3o (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3o (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3p (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3p (CDCl$_3$, 101 MHz)

$^{1}$H NMR Spectrum of 3q (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3q (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3r (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3r (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3s (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3s (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3t (CDCl$_3$, 400 MHz)
$^{13}\text{C NMR Spectrum of 3t (CDCl}_3\text{, 101 MHz)}$

![13C NMR Spectrum of 3t](image)

$^{1}\text{H NMR Spectrum of 3u (CDCl}_3\text{, 400 MHz)}$

![1H NMR Spectrum of 3u](image)
$^{13}$C NMR Spectrum of 3u (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3v (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3v (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3w (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3w (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3x (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3x (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3y (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3y (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3z (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3z (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3aa (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3aa (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3ab (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3ab (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3ac (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3ac (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3ad (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3ad (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3ae (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3ae (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3af (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3af (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 3ag (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 3ag (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 4a (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 4a (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 4b (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 4b (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 4c (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 4c (CDCl$_3$, 101 MHz)

$^{19}$F NMR Spectrum of 4c (CDCl$_3$, 376 MHz)
$^1$H NMR Spectrum of 4d (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 4d (CDCl$_3$, 101 MHz)
$^1$H NMR Spectrum of 4e (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 4e (CDCl$_3$, 101 MHz)
$^{19}$F NMR Spectrum of 4e (CDCl₃, 376 MHz)

$^1$H NMR Spectrum of 4f (CDCl₃, 400 MHz)
$^{13}$C NMR Spectrum of 4f (CDCl$_3$, 101 MHz)

$^{19}$F NMR Spectrum of 4f (CDCl$_3$, 376 MHz)
$^1$H NMR Spectrum of 4g (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 4g (CDCl$_3$, 101 MHz)
$^1$H NMR Spectrum of 4h (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 4h (CDCl$_3$, 101 MHz)
$^1$H NMR Spectrum of 4i (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 4i (CDCl$_3$, 101 MHz)
$^1$H NMR Spectrum of 4j (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 4j (CDCl$_3$, 101 MHz)
$^1$H NMR Spectrum of 4k (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 4k (CDCl$_3$, 101 MHz)
$^{19}$F NMR Spectrum of 4k (CDCl$_3$, 376 MHZ)

$^1$H NMR Spectrum of 4l (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 4l (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 4m (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 4m (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 4n (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 4n (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 2b (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 2b (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 2e (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 2e (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 2f (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 2f (CDCl$_3$, 101 MHz)

$^{19}$F NMR Spectrum of 2f (CDCl$_3$, 376 MHZ)
$^1$H NMR Spectrum of 2g (CDCl$_3$, 400 MHz)

$^{13}$C NMR Spectrum of 2g (CDCl$_3$, 101 MHz)
$^{19}$F NMR Spectrum of 2g (CDCl$_3$, 376 MHz)

$^{1}$H NMR Spectrum of 2h (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 2h (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 2j (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 2j (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 2l (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 2l (CDCl$_3$, 101 MHz)

$^1$H NMR Spectrum of 2m (CDCl$_3$, 400 MHz)
$^{13}$C NMR Spectrum of 2m (CDCl$_3$, 101 MHz)