Supporting Information

Aerobic Allylation of Alcohols with Non-Activated Alkenes Enabled by Light-Driven Selenium-π-Acid Catalysis

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1 General Remarks

Unless stated otherwise, all catalytic reactions were carried out under an atmosphere of air. Chemicals were obtained from commercial sources and were used without further purification. Yields correspond to isolated compounds unless indicated otherwise. Purity is estimated to be ≥95% based on $^1$H-NMR spectroscopic analysis. Irradiation experiments were performed at $\lambda = 465$ nm using commercially available blue LED strips (see experimental setup picture below). The light intensity applied was in the range of 9000 lx. TLC: Merck Silica Gel 60 F$_{254}$. Visualization of the developed chromatogram was performed by fluorescence quenching at 254 nm and staining with $p$-anisaldehyde, or potassium permanganate. Chromatography: Separations were carried out on Merck Silica 60 (0.063–0.200 mm, 70–230 mesh ASTM) using forced flow. IR: Bruker FT-IR Alpha-spectrometer with ATR sampling module. High resolution mass spectrometry (HR-MS): APEX IV 7T FTICR, BRUKER Daltonic. M.p.: KRÜSS M5000 capillary melting point apparatus, values are uncorrected. NMR ($^1$H, $^{13}$C) spectra were recorded at 300, 400, 500 MHz ($^1$H), 101, 126 MHz ($^{13}$C, APT (Attached Proton Test)) and 76, respectively, on VARIAN Unity-300, AMX 300 and Inova 500 instruments in CDCl$_3$ solution sat 298 K, if not specified otherwise. Chemical shifts (δ) are given in ppm. Multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sex = sextet, sept = septet, m = multiplet). In case of isomeric mixtures, integrals in $^1$H-NMR-spectra are given as a fraction of the combined integrals of both isomers. Fluorescence measurements: CLARIOstar well-plate reader by BMG LABTECH using 96 well-plates by Eppendorf (F-bottom, chimney-well, PP).
2 Optimization of the reaction conditions

To a solution of (Z)-dec-4-en-1-ol (78 mg, 0.5 mmol, 1.0 equiv) in an appropriate solvent (0.2 M) are added a base, the respective diselenide catalyst and the respective photosensitizer. The resulting mixture is vigorously stirred at ambient temperature under irradiation at $\lambda = 465$ nm for the noted time. The solvent is removed under reduced pressure and the yield is determined by $^1$H-NMR-spectroscopy using 1,3,5-trimethoxybenzene as the internal standard.

Table S1: Catalyst optimization.

<table>
<thead>
<tr>
<th>entry</th>
<th>diselenide</th>
<th>photosensitizer</th>
<th>solvent</th>
<th>yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ph$_2$Se$_2$ (10 mol%)</td>
<td>TAPT</td>
<td>MeCN</td>
<td>27</td>
</tr>
<tr>
<td>2</td>
<td>Ph$_2$Se$_2$ (10 mol%)</td>
<td>TAPT</td>
<td>DCE</td>
<td>27</td>
</tr>
<tr>
<td>3</td>
<td>(2-anisyl-Se)$_2$</td>
<td>TAPT</td>
<td>MeCN</td>
<td>51</td>
</tr>
<tr>
<td>4</td>
<td>(2-anisyl-Se)$_2$</td>
<td>TAPT</td>
<td>DCE</td>
<td>43</td>
</tr>
<tr>
<td>5</td>
<td>(2-anisyl-Se)$_2$</td>
<td>DMTA</td>
<td>MeCN-$d_3$</td>
<td>31$^a$</td>
</tr>
<tr>
<td>8</td>
<td>(2-anisyl-Se)$_2$</td>
<td>Ru(bpz)$_3$(PF$_6$)$_2$</td>
<td>MeCN-$d_3$</td>
<td>9$^a$</td>
</tr>
</tbody>
</table>

[a] Standard 1,1,2,2-tetrachlorethane.
Table S2: Catalyst loading optimization and control experiments.

<table>
<thead>
<tr>
<th>entry</th>
<th>x mol%</th>
<th>y mol%</th>
<th>time (h)</th>
<th>yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10</td>
<td>5</td>
<td>16</td>
<td>46</td>
</tr>
<tr>
<td>2</td>
<td>10</td>
<td>3</td>
<td>16</td>
<td>40</td>
</tr>
<tr>
<td>3</td>
<td>10</td>
<td>1</td>
<td>16</td>
<td>35</td>
</tr>
<tr>
<td>4</td>
<td>10</td>
<td>3</td>
<td>7</td>
<td>68\textsuperscript{a}</td>
</tr>
<tr>
<td>5</td>
<td>5</td>
<td>5</td>
<td>17</td>
<td>45</td>
</tr>
<tr>
<td>6</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>51</td>
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<tr>
<td>7</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>69\textsuperscript{a}</td>
</tr>
<tr>
<td>8</td>
<td>5</td>
<td>3</td>
<td>5</td>
<td>65\textsuperscript{a}</td>
</tr>
<tr>
<td>9\textsuperscript{b}</td>
<td>5</td>
<td>3</td>
<td>5</td>
<td>67\textsuperscript{a}</td>
</tr>
<tr>
<td>10</td>
<td>-</td>
<td>3</td>
<td>5</td>
<td>0\textsuperscript{c}</td>
</tr>
<tr>
<td>11</td>
<td>5</td>
<td>-</td>
<td>5</td>
<td>0\textsuperscript{c}</td>
</tr>
<tr>
<td>12\textsuperscript{d}</td>
<td>5</td>
<td>3</td>
<td>5</td>
<td>0\textsuperscript{c}</td>
</tr>
<tr>
<td>13\textsuperscript{e}</td>
<td>5</td>
<td>3</td>
<td>5</td>
<td>0\textsuperscript{c}</td>
</tr>
<tr>
<td>14\textsuperscript{f}</td>
<td>5</td>
<td>3</td>
<td>5</td>
<td>0\textsuperscript{c}</td>
</tr>
<tr>
<td>15\textsuperscript{g}</td>
<td>5</td>
<td>3</td>
<td>5</td>
<td>57 (57)</td>
</tr>
<tr>
<td>16\textsuperscript{h}</td>
<td>5</td>
<td>3</td>
<td>5</td>
<td>0\textsuperscript{c}</td>
</tr>
</tbody>
</table>

\textsuperscript{[a]} Reaction on 1.0 mmol scale in MeCN-\textsubscript{d}3 with internal standard 1,1,2,2-tetrachloroethane. \textsuperscript{[b]} Reaction with O\textsubscript{2} and molecular sieves instead of air. \textsuperscript{[c]} Standard 1,1,2,2-tetrachloroethane. \textsuperscript{[d]} No irradiation. \textsuperscript{[e]} No irradiation, flask in aluminum foil. \textsuperscript{[f]} Reaction was conducted under an argon atmosphere using degassed solvents. \textsuperscript{[g]} Reaction was conducted at 60 °C. \textsuperscript{[h]} Reaction was conducted at 0 °C.
Table S3: Solvent optimization.

\[
\begin{align*}
\text{entry} & \quad \text{solvent} & \quad \text{yield (\%)} \\
1 & \quad \text{MeCN} & \quad 47 \\
2 & \quad \text{DCE} & \quad 29 \\
3 & \quad \text{acetone} & \quad 9 \\
4 & \quad \text{THF} & \quad 0 \\
5 & \quad 1,4\text{-dioxane} & \quad 0 \\
6 & \quad \text{nitromethane} & \quad 42
\end{align*}
\]
**Table S4:** Base optimization.

<table>
<thead>
<tr>
<th>entry</th>
<th>base</th>
<th>x equiv</th>
<th>yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Na$_2$HPO$_4$</td>
<td>0.8</td>
<td>47</td>
</tr>
<tr>
<td>2</td>
<td>NaH$_2$PO$_4$</td>
<td>0.8</td>
<td>53</td>
</tr>
<tr>
<td>3</td>
<td>KH$_2$PO$_4$</td>
<td>0.8</td>
<td>14</td>
</tr>
<tr>
<td>4</td>
<td>K$_2$HPO$_4$</td>
<td>0.8</td>
<td>12</td>
</tr>
<tr>
<td>5</td>
<td>K$_3$PO$_4$</td>
<td>0.8</td>
<td>0</td>
</tr>
<tr>
<td>6</td>
<td>KF</td>
<td>0.8</td>
<td>0</td>
</tr>
<tr>
<td>7</td>
<td>CaF$_2$</td>
<td>0.8</td>
<td>51</td>
</tr>
<tr>
<td>8</td>
<td>NaHCO$_3$</td>
<td>0.8</td>
<td>28</td>
</tr>
<tr>
<td>9</td>
<td>Na$_2$CO$_3$</td>
<td>0.8</td>
<td>14</td>
</tr>
<tr>
<td>10</td>
<td>Li$_2$CO$_3$</td>
<td>0.8</td>
<td>50</td>
</tr>
<tr>
<td>11</td>
<td>LiOAc·2H$_2$O</td>
<td>0.8</td>
<td>0</td>
</tr>
<tr>
<td>12</td>
<td>NaOAc</td>
<td>0.8</td>
<td>9</td>
</tr>
<tr>
<td>13</td>
<td>KOAc</td>
<td>0.8</td>
<td>0</td>
</tr>
<tr>
<td>14</td>
<td>-</td>
<td>-</td>
<td>33</td>
</tr>
<tr>
<td>15</td>
<td>Na$_2$HPO$_4$</td>
<td>0.5</td>
<td>47</td>
</tr>
<tr>
<td>16</td>
<td>Na$_2$HPO$_4$</td>
<td>1.0</td>
<td>41</td>
</tr>
<tr>
<td>17</td>
<td>Na$_2$HPO$_4$</td>
<td>1.5</td>
<td>41</td>
</tr>
</tbody>
</table>
3 Fluorescence quenching experiments

For fluorescence quenching measurements, 2 mM stock solutions of the diselenide and the photocatalyst in methanol were prepared. From these stock solutions, samples were prepared with a final diselenide concentration of 10 µM and photocatalyst concentrations in the range of 0-700 µM (0-70 equiv.). Every measurement was conducted four times and an average value of the fluorescence intensity was used for analysis. The obtained intensities $I_0 - I$ were plotted against the diselenide concentration $c_{Se}$, where $I_0$ equals the fluorescence intensity of the unquenched photocatalyst derived from the sample containing no diselenide and $I$ equals the intensity of the quenched sample. The STERN-VOLMER constants $K_{SV}$ of the diselenides were obtained from the slopes of these plots following the STERN-VOLMER equation

$$\frac{I_0}{I} - 1 = K_{SV} \cdot c_{Se}$$

(eq. 1)

Fluorescence quenching was conducted at the respective absorption and emission wavelengths for every combination of the photocatalysts and diselenides as depicted in figure S1. The resulting STERN-VOLMER constants are summarized in table S5. The acridinium salt DMTA was synthesized following a procedure by NICEWICZ and DiROCCO et al.[1] The ruthenium catalyst, proflavin and rhodamine 6G were obtained from commercial sources. The syntheses of the diselenides are detailed in chapter 4.

---

Photocatalysts:

- TAUP
  - $\lambda_{abs} = 443 \text{ nm}$
  - $\lambda_{em} = 591 \text{ nm}$
- DMTA
  - $\lambda_{abs} = 411 \text{ nm}$
  - $\lambda_{em} = 553 \text{ nm}$
- Ru(bpy)$_3$(PF$_6$)$_2$
  - $\lambda_{abs} = 443 \text{ nm}$
  - $\lambda_{em} = 591 \text{ nm}$
- Proflavin-H
  - $\lambda_{abs} = 456 \text{ nm}$
  - $\lambda_{em} = 496 \text{ nm}$
- Rhodamine 6G
  - $\lambda_{abs} = 528 \text{ nm}$
  - $\lambda_{em} = 553 \text{ nm}$

Diselenides:

- G
- H
- I
- J
- K
- L
- M
- N
- O
Table S5: STERN-VOLMER constants for diselenide/photocatalyst combinations.

<table>
<thead>
<tr>
<th></th>
<th>$p$-CF$_3$</th>
<th>H</th>
<th>$p$-Me</th>
<th>$p$-OMe</th>
<th>$o$-OMe</th>
</tr>
</thead>
<tbody>
<tr>
<td>TAPT</td>
<td>85.1±4.70 M$^{-1}$</td>
<td>116±2.52 M$^{-1}$</td>
<td>-$^a$</td>
<td>162±6.33 M$^{-1}$</td>
<td>-$^a$</td>
</tr>
<tr>
<td>DMTA</td>
<td>96.3±6.00 M$^{-1}$</td>
<td>76.9±4.12 M$^{-1}$</td>
<td>187±3.50 M$^{-1}$</td>
<td>301±4.15 M$^{-1}$</td>
<td>103±5.61 M$^{-1}$</td>
</tr>
<tr>
<td>[Ru(bpz)$_3$(PF$_6$)$_2$</td>
<td>-$^b$</td>
<td>140±29.6 M$^{-1}$</td>
<td>870±9.71 M$^{-1}$</td>
<td>2020±60.0 M$^{-1}$</td>
<td>992±18.8 M$^{-1}$</td>
</tr>
<tr>
<td>Proflavin-H</td>
<td>48.2±2.67 M$^{-1}$</td>
<td>53.1±1.15 M$^{-1}$</td>
<td>76.6±2.99 M$^{-1}$</td>
<td>79.9±1.78 M$^{-1}$</td>
<td>43.9±4.05 M$^{-1}$</td>
</tr>
<tr>
<td>Rhodamine 6G$^c$</td>
<td>25.1±5.15 M$^{-1}$</td>
<td>23.2±1.88 M$^{-1}$</td>
<td>25.9±2.14 M$^{-1}$</td>
<td>44.4±9.22 M$^{-1}$</td>
<td>25.7±1.51 M$^{-1}$</td>
</tr>
</tbody>
</table>

$^a$Value was not determined; $^b$a negative value was obtained, the origin of which is unclear; $^c$all values obtained for rhodamine 6G exhibit large spreadings and therefore large errors and should be considered with care.
4 Syntheses of diselenides and TAPT

General procedure A: Syntheses of diselenides
To a solution of the aryl bromide (1.00 equiv.) in dry THF (100 mL) under an argon atmosphere is added tBuLi (1.7 M solution in n-pentane, 2.10 equiv.) dropwise at -78 °C. After complete addition, the solution is stirred at -78 °C for 15 min. and at 0 °C for 45 min. Selenium powder (1.10 equiv.) is added in one portion and stirring is continued for 15 min. at 0 °C and 45 min. at ambient temperature. The reaction is quenched by the addition of 1 M HCl (20 mL) and diluted with H2O (100 mL). The mixture is extracted with Et2O (3 × 100 mL) and the combined organic extracts are washed with brine (100 mL). The organic phase is dried over Na2SO4, the solid is filtered off and the solvent is removed under reduced pressure. The residue is redissolved in EtOH (50 mL), two pellets of NaOH are added and the mixture is vigorously stirred open to air for 2 h. The solvent is removed under reduced pressure and the residue is purified by column chromatography on silica gel.

1,2-Bis(4-(trifluoromethyl)phenyl)diselenide
Following general procedure A: 4-bromobenzotrifluoride (2.83 mL, 4.55 g, 20.2 mmol, 1.00 equiv.); eluting with n-pentane; yield: 1.98 g, 4.41 mmol, 44%, yellow solid.

TLC: Rf = 0.32 (n-pentane); m.p.: 58 °C; IR (neat): ν = 1597, 1494, 1397, 1320, 1161, 1101, 1067, 1008, 947, 819, 771, 718, 683, 586 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃): δ (ppm) = 7.47-7.53 (m, 4 H), 7.66-7.72 (m, 4 H); ¹³C-NMR (101 MHz, CDCl₃): δ (ppm) = 123.8, (q, J = 272.2 Hz), 126.1 (q, J = 3.7 Hz), 130.0 (q, J = 32.8 Hz), 130.7, 134.8 (q, J = 1.7 Hz); ⁷⁷Se-NMR (76 MHz, CDCl₃): δ (ppm) = 453.1; HR-MS (EI): [C₁₁H₇F₆Se₂]⁺ ([M]⁺): obs.: 449.8847; calcd.: 449.8863.

1,2-Di-p-tolyldiselenide
Following general procedure A: 4-bromotoluene (2.49 mL, 3.46 g, 20.2 mmol, 1.00 equiv.); eluting with n-pentane; yield: 1.17 g, 3.44 mmol, 34%, yellow solid.

TLC: Rf = 0.36 (n-pentane); m.p.: 38-40 °C; IR (neat): ν = 2913, 1485, 1393, 1300, 1205, 1177, 1037, 1012, 796, 480 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃): δ (ppm) = 2.35
(s, 6 H), 7.08 (dd, J = 8.1, 4.0 Hz, 4 H), 7.50 (dd, J = 8.1, 4.0 Hz); 13C-NMR (101 MHz, CDCl3): δ (ppm) = 21.1, 127.6, 129.9, 132.3, 137.9; 77Se-NMR (76 MHz, CDCl3): δ (ppm) = 474.2; HR-MS (EI): [C14H14Se2]·+ ([M]+): obs.: 341.9430; calcd.: 341.9429.

1,2-Bis(4-methoxyphenyl)diselenide

Following general procedure A: 4-bromoanisole (2.53 mL, 3.78 g, 20.2 mmol, 1.00 equiv.); eluting with n-pentane/EtOAc (10:1); yield: 1.80 g, 4.84 mmol, 48%, yellow solid.

TLC: Rf = 0.39 (n-pentane/EtOAc, 10:1); m.p.: 50-52 °C; IR (neat): ν = 3015, 2964, 2938, 2839, 2029, 1579, 1568, 1485, 1454, 1440, 1284, 1236, 1171, 1099, 1017, 1002, 817, 807, 787, 594, 512, 502 cm⁻¹; 1H-NMR (400 MHz, CDCl3): δ (ppm) = 3.79 (s, 6 H), 6.79 (dd, J = 8.9, 5.2 Hz, 2 H), 7.49 (dd, J = 8.9, 5.2 Hz, 2 H); 13C-NMR (101 MHz, CDCl3): δ (ppm) = 55.3, 114.7, 121.9, 135.4, 160.0; 77Se-NMR (76 MHz, CDCl3): δ (ppm) = 504.3; HR-MS (ESI): [C14H14NaO2Se2]·+ ([M + Na]·): obs.: 396.9194; calcd.: 396.9219.

1,2-Bis(2-methoxyphenyl)diselenide

Following general procedure A: 2-bromoanisole (2.52 mL, 3.78 g, 20.2 mmol, 1.00 equiv.); eluting with n-pentane/EtOAc (10:1); yield: 3.46 g, 9.30 mmol, 92%, yellow solid.

TLC: Rf = 0.41 (n-pentane/EtOAc, 10:1); m.p.: 77-78 °C; IR (neat): ν = 3000, 2936, 2834, 1571, 1464, 1429, 1266, 1233, 1180, 1158, 1121, 1050, 1015, 785, 746, 652, 568 cm⁻¹; 1H-NMR (400 MHz, CDCl3): δ (ppm) = 3.89 (s, 6 H), 6.80 (dd, J = 8.1, 1.2 Hz, 2 H), 6.86 (td, J = 7.6, 1.2 Hz, 2 H), 7.19 (ddd, J = 8.1, 7.4, 1.6 Hz, 2 H), 7.55 (dd, J = 7.7, 1.6 Hz, 2 H); 13C-NMR (101 MHz, CDCl3): δ (ppm) = 55.9, 110.1, 118.7, 121.9, 128.1, 130.6, 156.8; 77Se-NMR (76 MHz, CDCl3): δ (ppm) = 332.6; HR-MS (ESI): [C14H14NaO2Se2]·+ ([M + Na]·): obs.: 396.9228; calcd.: 396.9219.
2,4,6-Tris(4-methoxyphenyl)pyrylium tetrafluoroborate[2]

To a solution of 4’-methoxyacetophenone (15.0 g, 100 mmol, 2.0 equiv) and freshly distilled 4-methoxy-benzaldehyde (6.08 mL, 6.81 g, 50.0 mmol, 1.0 equiv) in dry toluene (5 mL) under an argon atmosphere BF$_3$·Et$_2$O (14.8 mL, 17.0 g, 120 mmol, 2.4 equiv) is slowly added and the resulting mixture is stirred at 100 °C for 2 h. The formed Et$_2$O is removed under reduced pressure and the residue is dissolved in acetone. Et$_2$O is added and the formed precipitate is filtered off and recrystallized from acetone. The title product is obtained as a red solid (5.53 g, 11.4 mmol, 23%).

IR (neat): $\nu = 2941, 2841, 1585, 1482, 1457, 1434, 1258, 1235, 1174, 1016, 829, 562, 518 \text{ cm}^{-1}$; $^1$H-NMR (300 MHz, DMSO-$d_6$): $\delta$ (ppm) = 3.91 (s, 6 H), 3.94 (s, 3 H), 7.04-7.21 (m, 6 H), 8.29 (d, $J = 9.0$ Hz, 4 H), 8.43 (d, $J = 9.1$ Hz, 2 H), 8.54 (s, 2 H); $^{13}$C-NMR (126 MHz, DMSO-$d_6$): $\delta$ (ppm) = 55.8, 55.9, 110.3, 115.1, 115.2, 121.0, 124.2, 130.4, 132.2, 161.5, 164.4, 165.2, 167.4; HR-MS (ESI): [C$_{26}$H$_{23}$O$_4$]$^+$ ([M - BF$_4$]$^+$): obs.: 399.1587; calcd.: 399.1591.

5 Synthesis and analytical data of precursors

2-(But-3-en-1-yl)isoindoline-1,3-dione

4-Bromo-1-butene (743 mg, 5.50 mmol, 1.1 equiv), phthalimide (736 mg, 5.01 mmol, 1.0 equiv) and CsCO$_3$ (1.79 g, 5.49 mmol, 1.1 equiv) were dissolved in DMF (3.5 mL) and the solution was stirred at 70 °C for 4 h. The mixture was poured in H$_2$O and filtered. The resulting precipitate was washed with water and dried. The desired compound was obtained as a white solid (740 mg, 3.68 mmol, 74%).

TLC: $R_f = 0.48$ (n-pentane/Et$_2$O, 2:1); m.p.: 50 °C; IR (neat): $\nu = 2942, 1769, 1694, 1642, 1612, 1466, 1450, 1436, 1396, 1361, 1332, 1291, 1271, 1255, 1188, 1176, 1055, 1015, 999, 983, 964, 935, 867, 798, 721, 712, 651, 611, 530, 445 \text{ cm}^{-1}$; $^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ (ppm) = 8.03-7.77 (m, 2 H), 7.77-7.54 (m, 2 H), 5.79 (ddt, $J = 17.1, 10.1, 6.9$ Hz, 1 H), 5.11-4.98 (m, 2 H), 3.77 (t, $J = 7.1$ Hz, 2 H), 2.45 (qt, $J = 7.0, 1.3$ Hz, 2 H); $^{13}$C-NMR (126 MHz, CDCl$_3$): $\delta$ (ppm) = 168.3, 134.5, 133.9,
132.2, 123.3, 117.6, 37.5, 33.0; HR-MS (ESI): [C_{12}H_{12}NO_2]^+ ([M + H]^+): obs.: 202.0855, calcd.: 202.0863.

**Hept-6-ynal**

A solution of Dess-Martin-periodinane (5.09 g, 12.0 mmol, 2.0 equiv) in dry DCM (27 mL) was cooled to 0 °C, 6-heptyn-1-ol (673 mg, 6.00 mmol, 1.0 equiv) in DCM (3 mL) was added and the mixture was stirred at 23 °C for 5 h. DCM (5 mL) was added, the mixture was washed with sat. aq. Na_2S_2O_3-sol. (30 mL), 1 M NaOH (30 mL), and sat. aq. NaCl (30 mL). The aq. phase was extracted with DCM (90 mL), the combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered and the solvent was removed under reduced pressure. The residue was purified on silica gel (n-pentane/Et_2O, 9:1) to yield the title compound as a colourless oil (337 mg, 3.06 mmol, 51%).

**TLC:** \( R_f = 0.29 \) (n-pentane/Et_2O, 9:1); **IR** (neat): \( \nu = \text{cm}^{-1}; \) \( ^1H\)-NMR (400 MHz, CDCl_3): \( \delta (ppm) = 9.77 \) (t, \( J = 1.7 \text{ Hz, 1 H} \), 2.46 (td, \( J = 7.3, 1.7 \text{ Hz, 2 H} \), 2.22 (td, \( J = 7.0, 2.7 \text{ Hz, 2 H} \), 1.95 (t, \( J = 2.7 \text{ Hz, 1 H} \), 1.83-1.66 (m, 2 H), 1.65-1.42 (m, 2 H);

\( ^{13}C\)-NMR (101 MHz, CDCl_3): \( \delta (ppm) = 202.3, 83.9, 68.9, 43.4, 27.9, 21.2, 18.3; \) **MS** (ESI): [C_7H_10O]^+ ([M]^+): obs.: 110.1, calcd.: 110.1.

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**6 Synthesis and analytical data of carboxylic acid substrates**

**General procedure B: Wittig Reaction**

Under an atmosphere of argon, the carboxyalkyl triphenyl phosphonium bromide (2.0 equiv) is dissolved in anhydrous THF (0.6 m) The suspension is cooled to 0 °C and KOt-Bu (powder or 1 m in THF; 4.0 equiv) is added dropwise. After 30 min of stirring at room temperature, a solution of the aldehyde (1.0 equiv) in anhydrous THF (2 m) is added dropwise at 0 °C. The reaction is stirred at room temperature and after the aldehyde is consumed, the mixture is quenched with 1 m aq. HCl solution (20 mL), extracted with Et_2O (3 × 30 mL) and washed with H_2O (2 × 60 mL). The combined organic layers are washed with brine, dried over anhydrous Na_2SO_4, filtered and the solvent is removed under reduced pressure. The residue is purified on silica gel to yield the title compound.
(Z)-7-Phenylhept-4-enoic acid

Following general procedure B: 3-phenylpropanaldehyde (402 mg, 3.00 mmol, 1.00 equiv), (3-carboxypropyl)triphenylphosphonium bromide (2.57 g, 6.00 mmol, 2.00 equiv), KOt-Bu (1.30 g, 12.0 mmol, 4.00 equiv), THF (15 mL); the reaction is stirred for 5 h; eluting with n-pentane/EtOAc, 95:5 to 80:20; yield: 590 mg, 2.90 mmol, 96%, yellow liquid.

TLC: $R_f = 0.63$ (n-pentane/EtOAc, 70:30); IR (neat): $\nu = 3324, 2921, 2857, 1704, 1603, 1495, 1452, 1279, 1209, 1086, 934, 725, 696, 481 \text{ cm}^{-1}$; $^1\text{H-NMR}$ (300 MHz, CDCl$_3$): $\delta$ (ppm) = 10.73 (s, 1 H), 7.42-7.30 (m, 2 H), 7.30-7.15 (m, 3 H), 5.49-5.35 (m, 2 H), 2.73 (t, $J = 7.5$ Hz, 2 H), 2.28-2.52 (m, 6 H); $^{13}\text{C-NMR}$ (76 MHz, CDCl$_3$): $\delta$ (ppm) = 179.9, 141.9, 130.6, 128.6, 128.4, 127.9, 125.9, 35.9, 34.1, 29.2, 22.6; HR-MS (ESI): [C$_{13}$H$_{16}$NaO$_2$]$: \text{[M + Na]}^+$: obs.: 227.1048, calcd.: 227.1043.

Methyl 2,2-diphenylhex-4-enoate$[^3]$

A 2.5 m solution of n-BuLi in hexane (2.04 mL, 5.10 mmol, 1.02 equiv) is added dropwise to a solution of diisopropylamine (0.74 mL, 5.25 mmol, 1.05 equiv) in anhydrous THF (5 mL) at $-78 \degree C$ under Ar atmosphere and the reaction mixture is allowed to stir for 30 min at $-78 \degree C$. After stirring an additional 30 min at $-5 \degree C$, a solution of methyl 2,2-diphenylacetate (1.13 g, 5.00 mmol, 1.00 equiv) in anhydrous THF (2 mL) is added dropwise at $-78 \degree C$, and the reaction mixture is allowed to stir for 10 min. Finally, crotyl bromide (trans, 85% pure; 810 mg, 6.00 mmol, 1.20 equiv) is added dropwise at $-78 \degree C$ and the mixture is slowly warmed to rt. After stirring for 16 h at rt, the reaction mixture is cooled to 0 $\degree C$, quenched by the addition of saturated aq. NH$_4$Cl (10 mL) then extracted with Et$_2$O (4 $\times$ 10 mL). The combined organic extracts are washed with brine, dried over anhydrous Na$_2$SO$_4$, filtered and concentrated under reduced pressure; yield: 1.00 g, 3.57 mmol, 71%, yellow liquid. The product is obtained as inseparable mixture of E/Z isomers (90:10; cf. NMR spectra).

TLC: $R_f = 0.28$ (n-pentane/EtOAc, 6:4); IR (neat): $\nu = 3028, 2949, 1728, 1598, 1495, 1445, 1217, 1114, 1034, 1018, 966, 851, 748, 728, 660, 500, 399 \text{ cm}^{-1}$; $^1\text{H-NMR}$ (300 MHz, CDCl$_3$): $\delta$ (ppm) = 7.37-7.10 (m, 10 H), 5.36-5.11 (m, 2 H), 3.70 (s, 3 H), 3.10-3.02 (m, 2 H), 1.53-1.45 (m, 3 H); $^{13}\text{C-NMR}$ (126 MHz, CDCl$_3$): major isomer: $\delta$ (ppm) = 174.6, 142.7, 129.0, 127.8, 126.8, 126.4, 60.8, 52.5, 41.8, 18.2; minor
isomer: $\delta$ (ppm) = 174.6, 142.7, 129.1, 127.8, 127.6, 126.9, 125.8, 60.5, 52.4, 36.0, 12.9; HR-MS (ESI): [C_{13}H_{21}O_{2}]^+ ([M + H]^+): obs.: 281.1537; calcd.: 281.1536.

(Z)-5-Cyclohexylpent-4-enoic acid

Following general procedure B: cyclohexanecarbaldehyde (393 mg, 3.50 mmol, 1.00 equiv), (3-carboxypropyl)triphenylphosphoniumbromide (3.00 g, 7.00 mmol, 2.00 equiv), KOt-Bu (1.60 g, 14.00 mmol, 4.00 equiv), THF (15 mL); the reaction is stirred for 5 h; eluting with n-pentane/EtOAc, 90:10 to 70:30; yield: 480 mg, 2.60 mmol, 75%, yellow liquid.

TLC: $R_f = 0.73$ (n-pentane/EtOAc, 70:30); IR (neat): $\nu = 2921, 2849, 1706, 1447, 1411, 1277, 1277, 1208, 1131, 945, 889, 736, 479, 397$ cm$^{-1}$; $^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ (ppm) = 11.11 (s, 1 H), 5.32-5.13 (m, 2 H), 2.44-2.14 (m, 5 H), 1.75-1.47 (m, 5 H), 1.34-0.91 (m, 5 H); $^{13}$C-NMR (76 MHz, CDCl$_3$): $\delta$ (ppm) = 180.0, 138.0, 125.1, 36.5, 34.6, 33.4, 26.2, 26.0, 22.9; HR-MS (ESI): [C_{11}H_{17}O_{2}]^+ ([M - H]^+): obs.: 181.1235; calcd.: 181.1234.

(Z)-11-Hydroxy-7,11-dimethylidodec-4-enoic acid

Following general procedure B: 7-hydroxy-3,7-dimethyloctanal (603 mg, 3.50 mmol, 1.00 equiv), (3-carboxypropyl)triphenylphosphonium bromide (3.00 g, 7.00 mmol, 2.00 equiv), KOt-Bu (1.60 g, 14.00 mmol, 4.00 equiv), THF (15 mL); the reaction is stirred for 6 h; eluting with n-pentane/EtOAc, 6:4 to 4:6; yield: 838 mg, 3.45 mmol, 99%, yellow liquid.

TLC: $R_f = 0.89$ (n-pentane/EtOAc, 4:6); IR (neat): $\nu = 3433, 2934, 1709, 1460, 1376, 1199, 1159, 933, 903, 762, 715, 479$ cm$^{-1}$; $^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ (ppm) = 6.00 (s, 1 H), 5.51-5.24 (m, 2 H), 2.43-2.23 (m, 4 H), 2.05 (m, 1 H), 1.91 (m, 1 H), 1.54-0.99 (m, 13 H), 0.87 (d, $J = 6.7$ Hz, 3 H); $^{13}$C-NMR (76 MHz, CDCl$_3$): $\delta$ (ppm) = 178.5, 150.3, 128.1, 71.6, 64.2, 37.0, 34.3, 34.2, 33.3, 29.3, 22.9, 21.7, 19.8; HR-MS (ESI): [C_{14}H_{26}NaO_{3}]^+ ([M + Na]^+): obs.: 265.1776, calcd.: 265.1774.

(E)-6-Cyclohexylhex-5-enoic acid

Following general procedure B: (4-carboxybutyl)triphenylphosphonium bromide (3.10 g, 7.00 mmol, 2.00 equiv), KOtBu (1.57 g, 14.0 mmol, 4.00 equiv), cyclohexanecarbaldehyde
(393 mg, 3.50 mmol, 1.00 equiv), THF (14 mL), 4 h; eluting with n-pentane/Et₂O, 1:1; yield: 531 mg, 2.70 mmol, 77%, colourless oil.

**TLC:** \( R_f = 0.36 \) (n-pentane/Et₂O, 1:1); **IR** (neat): \( \nu = 2999, 2922, 2849, 1704, 1447, 1411, 1289, 1241, 1203, 1164, 930, 889, 732, 487 \) cm⁻¹; **¹H-NMR** (500 MHz, CDCl₃): \( \delta \) (ppm) = 5.30-5.16 (m, 2 H), 2.37 (t, \( J = 7.5 \) Hz, 2 H), 2.22 (m, 1 H), 2.16-2.07 (m, 2 H), 1.76-1.65 (m, 5 H), 1.63-1.54 (m, 2 H), 1.39-1.12 (m, 3 H), 1.11-0.99 (m, 2 H); **¹³C-NMR** (76 MHz, CDCl₃): \( \delta \) (ppm) = 180.2, 137.5, 126.4, 36.5, 33.6, 33.5, 26.8, 26.2, 26.1, 24.9; **HR-MS** (ESI): [C₁₂H₁₉O₃⁻] (M - H⁻): obs.: 195.1394; calcd.: 195.1391.

**Z-8-Methylnon-5-enoic acid**

Following general procedure **B**: (4-carboxybutyl)triphenylphosphonium bromide (3.10 g, 7.00 mmol, 2.00 equiv), KOtBu (1 M in THF; 14 mL, 14.0 mmol, 4.00 equiv), isovaleraldehyde (301 mg, 3.50 mmol, 1.00 equiv), THF (6 mL), 3 h; eluting with n-pentane/Et₂O, 3:1; yield: 501 mg, 2.95 mmol, 84%, colourless oil.

**TLC:** \( R_f = 0.50 \) (n-pentane/Et₂O, 1:1); **IR** (neat): \( \nu = 3008, 2954, 2870, 1706, 1461, 1412, 1384, 1366, 1314, 1241, 1167, 1122, 928, 826, 703, 481 \) cm⁻¹; **¹H-NMR** (300 MHz, CDCl₃): \( \delta \) (ppm) = 6.40 (s, 2 H), 5.53-5.30 (m, 2 H), 2.34 (t, \( J = 7.3 \) Hz, 2 H), 2.14-2.01 (m, 3 H), 1.82 (m, 1 H), 1.70 (quin, \( J = 7.3 \) Hz, 2 H), 1.52-1.25 (m, 6 H), 1.21 (s, 6 H), 1.12 (m, 1 H), 0.87 (d, 6 H); **¹³C-NMR** (126 MHz, CDCl₃): \( \delta \) (ppm) = 179.9, 130.1, 128.9, 36.6, 33.6, 28.9, 26.8, 24.8, 22.6; **HR-MS** (ESI): [C₁₀H₁₇O₂⁻] (M - H⁻): obs.: 169.1234; calcd.: 169.1234.

**Z-12-Hydroxy-8,12-dimethyltridec-5-enoic acid**

Following general procedure **B**: (4-carboxybutyl)triphenylphosphonium bromide (3.10 g, 7.00 mmol, 2.00 equiv), KOtBu (1.57 g, 14.0 mmol, 4.00 equiv), 3,7-dimethyl-7-hydroxyoctanal (603 mg, 3.50 mmol, 1.00 equiv), THF (12 mL), 17 h; eluting with Et₂O; yield: 709 mg, 2.77 mmol, 79%, colourless oil.

**TLC:** \( R_f = 0.20 \) (Et₂O); **IR** (neat): \( \nu = 2935, 2869, 1707, 1459, 1377, 1198, 1158, 934, 906, 763, 699, 495 \) cm⁻¹; **¹H-NMR** (300 MHz, CDCl₃): \( \delta \) (ppm) = 6.40 (s, 2 H), 5.53-5.30 (m, 2 H), 2.34 (t, \( J = 7.3 \) Hz, 2 H), 2.14-2.01 (m, 3 H), 1.82 (m, 1 H), 1.70 (quin, \( J = 7.3 \) Hz, 2 H), 1.52-1.25 (m, 6 H), 1.21 (s, 6 H), 1.12 (m, 1 H), 0.87 (d, 6 H).
$J = 6.6 \text{ Hz, } 3 \text{ H}$; $^{13}$C-NMR (76 MHz, CDCl$_3$): $\delta$ (ppm) = 178.9, 129.8, 129.3, 71.6, 44.1, 37.2, 34.3, 33.6, 33.5, 29.3, 29.2, 26.7, 24.8, 21.8, 19.9; HR-MS (ESI): [C$_{15}$H$_{28}$NaO$_3$]$^+$ ([M + Na]$^+$): obs.: 279.1929; calcd.: 279.1931.

(Z)-8-Phenyloct-5-enoic acid

Following general procedure B: (4-carboxybutyl)triphenyl-phosphonium bromide (3.10 g, 7.00 mmol, 2.00 equiv), KO'Bu (1.57 g, 14.0 mmol, 4.00 equiv), 3-phenylpropanal (470 mg, 3.50 mmol, 1.00 equiv), THF (12 mL), 3 h; eluting with n-pentane/Et$_2$O, 4:1; yield: 629 mg, 2.88 mmol, 82%, colourless oil.

TLC: $R_f = 0.20$ (n-pentane/Et$_2$O, 1:1); IR (neat): $\nu$ = 2924, 2856, 1704, 1495, 1453, 1411, 1239, 1029, 931, 747, 697, 582, 478 cm$^{-1}$; $^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ (ppm) = 10.53 (s, 1 H), 5.59-5.26 (m, 2 H), 2.67 (t, $J = 7.7 \text{ Hz}$, 2 H), 2.35 (q, $J = 7.5 \text{ Hz}$, 2 H), 2.30 (t, $J = 7.5 \text{ Hz}$, 2 H), 2.05 (q, $J = 7.1 \text{ Hz}$, 2 H), 1.64 (quin, $J = 7.5 \text{ Hz}$, 2 H); $^{13}$C-NMR (76 MHz, CDCl$_3$): $\delta$ (ppm) = 180.2, 142.1, 130.6, 129.8, 128.6, 128.4, 125.9, 36.0, 33.5, 29.3, 26.6, 24.6; HR-MS (ESI): [C$_{14}$H$_{18}$NaO$_2$]$^+$ ([M + Na]$^+$): obs.: 279.1926; calcd.: 279.1931.

(Z)-10-Chlorodec-5-enoic acid

Following general procedure B: (4-carboxybutyl)triphenyl-phosphonium bromide (3.10 g, 7.00 mmol, 2.00 equiv), KO'Bu (1.57 g, 14.0 mmol, 4.00 equiv), 3-phenylpropanal (470 mg, 3.50 mmol, 1.00 equiv), THF (12 mL), 3 h; eluting with n-pentane/Et$_2$O, 4:1; yield: 629 mg, 2.88 mmol, 82%, colourless oil.

TLC: $R_f = 0.20$ (n-pentane/Et$_2$O, 1:1); IR (neat): $\nu$ = 2924, 2856, 1704, 1495, 1453, 1411, 1239, 1029, 931, 747, 697, 582, 478 cm$^{-1}$; $^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ (ppm) = 10.53 (s, 1 H), 5.59-5.26 (m, 2 H), 2.67 (t, $J = 7.7 \text{ Hz}$, 2 H), 2.35 (q, $J = 7.5 \text{ Hz}$, 2 H), 2.30 (t, $J = 7.5 \text{ Hz}$, 2 H), 2.05 (q, $J = 7.1 \text{ Hz}$, 2 H), 1.64 (quin, $J = 7.5 \text{ Hz}$, 2 H); $^{13}$C-NMR (76 MHz, CDCl$_3$): $\delta$ (ppm) = 180.2, 142.1, 130.6, 129.8, 128.6, 128.4, 125.9, 36.0, 33.5, 29.3, 26.6, 24.6; HR-MS (ESI): [C$_{14}$H$_{18}$NaO$_2$]$^+$ ([M + Na]$^+$): obs.: 279.1926; calcd.: 279.1931.

(Z)-Hexadeca-5,15-dienoic acid

Following general procedure B: (4-carboxybutyl)triphenyl-phosphonium bromide (3.10 g, 7.00 mmol, 2.00 equiv), KO'Bu (1.57 g, 14.0 mmol, 4.00 equiv), 3-phenylpropanal (470 mg, 3.50 mmol, 1.00 equiv), THF (12 mL), 3 h; eluting with n-pentane/Et$_2$O, 4:1; yield: 629 mg, 2.88 mmol, 82%, colourless oil.

TLC: $R_f = 0.29$ (n-pentane/Et$_2$O, 1:1); IR (neat): $\nu$ = 2924, 2856, 1704, 1495, 1453, 1411, 1239, 1029, 931, 747, 697, 582, 478 cm$^{-1}$; $^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ (ppm) = 5.53-5.29 (m, 2 H), 3.55 (t, $J = 6.7 \text{ Hz}$, 2 H), 2.38 (t, $J = 7.5 \text{ Hz}$, 2 H), 2.21-1.96 (m, 4 H), 1.76 (dp, $J = 22.4, 7.1 \text{ Hz}$, 4 H), 1.51 (quint, $J = 7.5 \text{ Hz}$, 2 H); $^{13}$C-NMR (106 MHz, CDCl$_3$): $\delta$ (ppm) = 179.8, 130.5, 129.1, 45.1, 33.5, 32.3, 27.0, 26.6, 24.7; HR-MS (ESI): [C$_{10}$H$_{16}$ClO$_2$]$^-$ ([M - H]$^-$): obs.: 203.0846; calcd.: 203.0844.
(1 mL in THF; 16 mL, 16.0 mmol, 4.00 equiv), 10-undecenal (673 mg, 4.00 mmol, 1.00 equiv), THF (6 mL), 17 h; eluting with n-pentane/Et₂O, 3:1; yield: 733 mg, 2.90 mmol, 73%, colourless oil.

**TLC:** $R_f = 0.25$ (n-pentane/Et₂O, 3:1); **IR** (neat): $\nu = 3005, 2924, 2853, 2359, 2341, 1707, 1640, 1413, 1240, 1205, 1170, 992, 908, 721, 687 \text{ cm}^{-1}$; **¹H-NMR** (300 MHz, CDCl₃): $\delta$ (ppm) = 5.81 (ddt, $J = 16.9, 10.2, 6.7 \text{ Hz}$, 1 H), 5.59-5.17 (m, 2 H), 4.99 (ddt, $J = 17.1, 2.3, 1.2 \text{ Hz}$, 1 H), 2.36 (td, $J = 7.6, 4.3 \text{ Hz}$, 2 H), 2.15-1.86 (m, 6 H), 1.85-1.58 (m, 2 H), 1.49-1.10 (m, 12 H); **¹³C-NMR** (126 MHz, CDCl₃): $\delta$ (ppm) = 179.6, 139.3, 131.4, 128.2, 114.2, 34.0, 33.6, 29.9, 29.7, 29.7, 29.5, 29.4, 29.2, 27.5, 26.7, 24.8; **HR-MS** (ESI): [C₁₆H₂₉O₂]⁺ ([M + H]⁺): obs.: 253.2154; calcd.: 253.2162.

(Z)-dodec-5-en-11-ynoic acid

Following general procedure B: (4-carboxybutyl)triphenylphosphonium bromide (2.66 g, 6.00 mmol, 2.00 equiv), KOtBu (1 mL in THF; 12 mL, 12.0 mmol, 4.00 equiv), (300 mg, 3.00 mmol, 1.00 equiv), THF (5 mL), 17 h; eluting with n-pentane/Et₂O, 3:1; yield: 317 mg, 1.63 mmol, 54%, yellow oil. Contains allene (10%).

**TLC:** $R_f = 0.14$ (n-pentane/Et₂O, 3:1); **IR** (neat): $\nu = 3303, 3006, 2935, 2860, 1705, 1457, 1432, 1412, 1267, 1239, 1206, 1098, 1040, 932 \text{ cm}^{-1}$; **¹H-NMR** (400 MHz, CDCl₃): $\delta$ (ppm) = 9.25 (s, 1 H), 5.47-5.29 (m, 2 H), 2.37 (t, $J = 7.4 \text{ Hz}$, 2 H), 2.19 (td, $J = 6.9, 2.7 \text{ Hz}$, 2 H), 2.15-2.00 (m, 4 H), 1.94 (t, $J = 2.7 \text{ Hz}$, 1 H), 1.70 (p, $J = 7.4 \text{ Hz}$, 2 H), 1.60-1.36 (m, 4 H); **¹³C-NMR** (101 MHz, CDCl₃): $\delta$ (ppm) = 179.8, 130.8, 128.8, 84.7, 68.4, 33.6, 28.8, 28.2, 26.8, 26.6, 24.7, 18.4; **HR-MS** (ESI): [C₁₂H₁₈O₂Na]⁺ ([M + Na]⁺): obs.: 217.1198; calcd.: 217.1199.

### 7 Synthesis and analytical data of alcohol substrates

**General procedure C: Reduction with LiAlH₄**

A solution of acid or ester (1.0 equiv) in anhydrous THF (7 mL) is added dropwise with stirring to a suspension of LiAlH₄ (2.4 M in THF) (1.5 equiv) in anhydrous THF (3 mL) at 0 °C. After stirring at rt, the reaction mixture was cooled to 0 °C, diluted with Et₂O (15 mL), quenched with sodium sulfate decahydrate, and filtered. The filter cake
is washed with a saturated aqueous solution of potassium sodium tartrate (Rochelle salt) and extracted with Et₂O (5 × 10 mL). The combined organic layers are washed with brine, dried over anhydrous Na₂SO₄, filtered and the solvent is removed under reduced pressure. The product is used without further purification.

**General procedure D: Cross Metathesis Reaction**

Under an atmosphere of argon, 4-penten-1-ol (3 equiv) and alkene (1 equiv) are dissolved in degassed, anhydrous CH₂Cl₂ (0.5 M). Grubbs catalyst 2nd generation (0.05 equiv) is added and the reaction mixture is allowed to stir at 40 °C. The solvent is removed under reduced pressure and the residue is purified on silica gel to yield the title compound.

(\(Z\))-7-Phenylhept-4-en-1-ol 6b

Following general procedure C: (\(Z\))-7-Phenylhept-4-enoic acid (540 mg, 2.60 mmol, 1.00 equiv), LiAlH₄ (1.60 mL, 3.90 mmol, 1.50 equiv), THF (10 mL); the reaction is stirred overnight; yield: 197 mg, 1.00 mmol, 40%, colorless liquid. The product is obtained as inseparable mixture of \(Z/E\) isomers (87/13).

**TLC:** \(R_f = 0.47\) (n-pentane/EtOAc, 50:50); **IR** (neat): \(\nu = 3334, 3025, 3006, 2929, 2858, 1603, 1495, 1452, 1057, 1029, 968, 905, 696, 583, 487, 470 \text{ cm}^{-1}\); **\(^1\)H-NMR** (300 MHz, CDCl₃): \(\delta\) (ppm) = 7.33-7.15 (m, 5 H), 5.51-5.34 (m, 2 H), 3.58 (t, \(J = 6.5 \text{ Hz}, 2 \text{ H}), 2.70 (t, \(J = 7.6 \text{ Hz}, 2 \text{ H}), 2.41 (td, \(J = 7.9, 6.2 \text{ Hz}, 2 \text{ H}), 2.11-2.00 (m, 2 \text{ H}), 1.60-1.48 (m, 2 \text{ H}); **\(^{13}\)C-NMR** (76 MHz, CDCl₃): \(\delta\) (ppm) = major isomer: 142.1, 129.8, 129.5, 128.5, 128.3, 125.8, 62.4, 36.0, 32.5, 29.2, 23.6; minor isomer: 130.3, 130.1, 128.5, 125.8, 62.3, 36.1, 34.4, 32.4, 28.9; **HR-MS** (ESI): [C₁₃H₁₈NaO]⁺ ([M + Na]⁺): obs.: 213.1251; calcd.: 213.1250.

2,2-Diphenylhex-4-en-1-ol 6c

Following general procedure C: Methyl 2,2-diphenylhex-4-enoate (1.00 g, 3.57 mmol, 1.00 equiv), LiAlH₄ (2.00 mL, 4.90 mmol, 1.50 equiv), THF (10 mL); 2.5 h; yield: 804 mg, 3.20 mmol, 89%, colorless liquid. The product is obtained as mixture of \(E/Z\) isomers (90:10).

**TLC:** \(R_f = 0.39\) (n-pentane/EtOAc, 60:40); **IR** (neat): \(\nu = 3444, 3057, 3024, 2915, 2854, 1598, 1495, 1443, 1037, 1015, 968, 752, 696, 631, 611, 574, 551 \text{ cm}^{-1}\); **\(^1\)H-
**NMR** (400 MHz, CDCl$_3$): $\delta$ (ppm) = 7.36-7.27 (m, 4 H), 7.25-7.14 (m, 6 H), 5.52 (m, 1 H), 5.07 (m, 1 H), 4.14 (s, 2 H), 2.91 (dt, $J = 7.1$, 1.3 Hz, 2 H), 1.70-1.48 (m, 3 H), 1.28 (s, 1 H); $^{13}$C-NMR (100 MHz, CDCl$_3$): $\delta$ (ppm) = E isomer: 145.6, 128.8, 128.4, 128.3, 126.8, 126.4, 68.1, 51.9, 39.9, 18.1; Z isomer: 145.6, 128.8, 128.4, 127.0, 126.4, 68.3, 52.2, 33.9, 13.1; **HR-MS** (ESI): $[C_{18}H_{20}NaO]^+$ ([M + Na]$^+$): obs.: 275.1409; calcd.: 275.1406.

**Z**-5-cyclohexylpent-4-en-1-ol 6d

Following general procedure C: (Z)-5-Cyclohexylpent-4-enoic acid (480 mg, 2.60 mmol, 1.00 equiv), LiAlH$_4$ (1.6 mL, 3.90 mmol, 1.50 equiv), THF (10 mL); 17 h; the product is purified by flash column chromatography eluting with $n$-pentane/EtOAc, 85:15 to 70:30; yield: 388 mg, 2.3 mmol, 88%, colorless liquid.

**TLC:** $R_f = 0.28$ ($n$-pentane/EtOAc, 70:30); **IR** (neat): $\nu = 3331, 2999, 2920, 2848, 1447, 1057, 946, 889, 730, 602, 397$ cm$^{-1}$; **$^1$H-NMR** (300 MHz, CDCl$_3$): $\delta$ (ppm) = 5.69-4.98 (m, 2 H), 3.64 (t, $J = 6.5$ Hz, 2 H), 2.25 (tdt, $J = 10.9$, 8.4, 3.8 Hz, 1 H), 2.17-2.04 (m, 2 H), 1.86-1.48 (m, 8 H), 1.43-0.86 (m, 5 H), $^{13}$C-NMR (126 MHz, CDCl$_3$): $\delta$ (ppm) = 136.9, 127.0, 62.8, 36.5, 33.5, 33.1, 26.3, 26.2, 24.1; **HR-MS** (ESI): $[C_{11}H_{21}O]^+$ ([M + H]$^+$): obs.: 169.1588; calcd.: 169.1587.

**Z/E**-9-Hydroxynon-5-enenitrile 6e

Following general procedure D: 5-penten-1-ol (904 mg, 10.5 mmol, 3.00 equiv), 6-hexenenitrile (333 mg, 3.50 mmol, 1.00 equiv), Grubbs Catalyst 2nd generation (148 mg, 0.17 mmol, 0.05 equiv) CH$_2$Cl$_2$ (7 mL); 20 h; yield: 289 mg, 1.89 mmol, 54%, orange liquid. The product is obtained as mixture of E/Z isomers (90:10).

**TLC:** $R_f = 0.74$ ($n$-pentane/EtOAc, 60:40); **IR** (neat): $\nu = 3404, 2934, 2868, 2246, 1439, 1054, 969, 916, 541$ cm$^{-1}$; **$^1$H-NMR** (400 MHz, CDCl$_3$): $\delta$ (ppm) = 5.50 (m, 1 H), 5.36 (dtt, $J = 15.0$, 6.8, 1.3 Hz, 1 H), 3.62 (t, $J = 6.5$ Hz, 2 H), 2.31 (t, $J = 7.2$ Hz, 2 H), 2.19-2.02 (m, 4 H), 1.78-1.66 (m, 3 H), 1.61 (ddt, $J = 8.3$, 7.3, 6.5 Hz, 2 H); $^{13}$C-NMR (101 MHz, CDCl$_3$): $\delta$ (ppm) = major isomer: 131.5, 127.4, 62.1, 32.4, 25.8, 25.1, 23.5, 16.3; minor isomer: 131.7, 127.6, 119.8, 62.3, 32.5, 25.9, 25.3, 23.7, 16.5; **HR-MS** (ESI): $[C_9H_{15}NNaO]^+$ ([M + Na]$^+$): obs.: 176.1050; calcd.: 176.1046.
Oct-4-ene-1,8-diol 6f

4-Penten-1-ol (5.00 g, 58.1 mmol, 1.0 equiv) was dissolved in degassed DCM (200 mL). Grubbs Catalyst 2nd generation (50 mg, 59 µmol, 0.1 mol-%) was added and the reaction was stirred for 22 h at room temperature. After 3 h, another portion of Grubbs Catalyst 2nd generation (50 mg, 59 µmol, 0.1 mol-%) was added, after 3 more hours, another portion (50 mg, 59 µmol, 0.1 mol-%) was added. After 3 more hours, Grubbs Catalyst 2nd generation (100 mg, 118 µmol, 0.2 mol-%) in DCM (6 mL) was added via syringe pump over 6 h. The solvent was removed in vacuum and the product afforded via column chromatography (n-pentane/EtOAc, 3:1 to 0:1) as a brown liquid (1.88 g, 13.0 mmol, 48%). The product is obtained as mixture of E/Z isomers.

**TLC:** $R_f = 0.06$ (n-pentane/EtOAc, 3:1); **IR** (neat): $\nu = 3316, 2931, 2865, 1440, 1375, 1351, 1051, 966, 915, 631 \text{ cm}^{-1}$; **$^1$H-NMR** (300 MHz, CDCl$_3$): $\delta$ (ppm) = 5.75-5.20 (m, 2 H), 3.72-3.46 (m, 4 H), 2.86-1.89 (m, 6 H), 1.74-1.27 (m, 4 H); **$^{13}$C-NMR** (126 MHz, CDCl$_3$): $\delta$ (ppm) = major isomer: 130.2, 62.4, 32.5, 29.0; minor isomer: 129.8, 61.9, 32.4, 23.5; **HR-MS** (ESI): [C$_8$H$_{16}$O$_2$Na]$^+$ ([M + Na]$^+$): obs.: 167.1043; calcd.: 167.1043.

8-Hydroxyoct-4-en-1-yl pivalate 6g

Oct-4-ene-1,8-diol (216 mg, 1.50 mmol, 1.0 equiv) and DMAP (202 mg, 1.65 mmol, 1.1 equiv) were dissolved in DCM (5 mL) and pivalic acid chloride (199 mg, 1.65 mmol, 1.1 equiv) was added dropwise. The solution was stirred for 23 h at room temperature, subsequently washed with aq. sat. Na$_2$CO$_3$ solution (5 mL) and aq. 1 M HCl (5 mL) and the comb. aq. phases were extracted with DCM (10 mL). The comb. org. phases were dried over Na$_2$SO$_4$, the solvent was removed in vacuum and the product was afforded via column chromatography (n-pentane/EtOAc, 4:1) as a colourless oil (113 mg, 520 µmol, 35%).

**TLC:** $R_f = 0.29$ (n-pentane/EtOAc, 4:1); **IR** (neat): $\nu = 2958, 2934, 2872, 1727, 1480, 1460, 1398, 1366, 1284, 1153, 1055, 1036, 968, 892, 772 \text{ cm}^{-1}$; **$^1$H-NMR** (300 MHz, CDCl$_3$): $\delta$ (ppm) = 5.83-5.22 (m, 2 H), 4.04 (t, $J = 6.5$ Hz, 2 H), 3.64 (t, $J = 6.5$ Hz, 2 H), 2.20-1.90 (m, 4 H), 1.87-1.50 (m, 4 H), 1.41 (s, 1 H), 1.20 (d, $J = 1.1$ Hz, 9 H); **$^{13}$C-NMR** (126 MHz, CDCl$_3$): $\delta$ (ppm) = 178.6, 130.6, 129.6, 63.9, 62.6, 39.0, 32.6.

**Ethyl (8-hydroxyoct-4-en-1-yl) carbonate 6h**

Oct-4-ene-1,8-diol (216 mg, 1.50 mmol, 1.0 equiv) and DMAP (4.6 mg, 38 µmol, 0.025 equiv) were dissolved in dry DCM (2 mL) and cooled to 0 °C. Pyridine (237 mg, 3.0 mmol, 2.0 equiv) was added dropwise, then ethyl chloroformate (163 mg, 1.5 mmol, 1.5 equiv) was added dropwise and the reaction was allowed to warm up to room temperature. The solution was stirred for 23 h, subsequently washed with H$_2$O (5 × 2 mL) and the comb. aq. phases were extracted with DCM (10 mL). The comb. org. phases were dried over Na$_2$SO$_4$, the solvent was removed in vacuum and the product was afforded via column chromatography (n-pentane/EtOAc, 4:1) as a colourless oil (113 mg, 520 µmol, 35%).

**TLC:** $R_f = 0.21$ (n-pentane/EtOAc, 4:1); **IR** (neat): $\nu = 2983, 2934, 2874, 1742, 1467, 1448, 1403, 1386, 1368, 1251, 1054, 1010, 969, 863, 791$ cm$^{-1}$; **$^1$H-NMR** (300 MHz, CDCl$_3$): $\delta$ (ppm) = 5.69-5.29 (m, 2 H), 4.18 (q, $J = 7.1$ Hz, 2 H), 4.14-4.07 (m, 2 H), 3.64 (t, $J = 6.5$ Hz, 2 H), 2.23-1.98 (m, 4 H), 1.83-1.67 (m, 2 H), 1.67-1.56 (m, 2 H), 1.51 (m, 1 H), 1.31 (t, $J = 7.1$ Hz, 3 H); **$^{13}$C-NMR** (126 MHz, CDCl$_3$): $\delta$ (ppm) = 155.3, 130.8, 129.4, 67.4, 64.0, 62.6, 32.6, 29.0, 28.8, 28.7, 14.5; **HR-MS** (ESI): [C$_{11}$H$_{20}$O$_4$Na]$^+$ ([M + Na]$^+$): obs.: 239.1256; calcd.: 239.1254.

**2-(7-Hydroxyhept-3-en-1-yl)isoindoline-1,3-dione 6i**

Following general procedure D: 5-Penten-1-ol (904 mg, 10.5 mmol, 3.00 equiv), 2-(but-3-en-1-yl)isoindoline-1,3-dion (704 mg, 3.50 mmol, 1.00 equiv), Grubbs Catalyst 2$^{nd}$ generation (148 mg, 0.175 mmol, 0.05 equiv) CH$_2$Cl$_2$ (7 mL); 5.5 h; eluting with n-pentane/EtOAc, 2:1; yield: 408 mg, 1.57 mmol, 45%, colourless liquid. The product is obtained as mixture of E/Z isomers.

**TLC:** $R_f = 0.25$ (n-pentane/EtOAc, 2:1); **IR** (neat): $\nu = 2935, 2864, 1771, 1700, 1614, 1467, 1436, 1393, 1359, 1187, 1130, 1057, 1015, 971, 870, 794, 718, 624, 530$ cm$^{-1}$; **$^1$H-NMR** (300 MHz, CDCl$_3$): $\delta$ (ppm) = 7.85-7.81 (m, 2 H), 7.72-7.68 (m, 2 H), 5.48-5.37 (m, 2 H), 3.73 (t, $J = 7.0$ Hz, 2 H), 3.53 (t, $J = 6.5$ Hz, 2 H), 2.43-2.31 (m, 2 H), 2.15-1.94 (m, 3 H), 1.66-1.40 (m, 2 H); **$^{13}$C-NMR** (126 MHz, CDCl$_3$): $\delta$ (ppm) = major
isomer: 168.4, 133.9, 132.8, 132.2, 126.7, 123.2, 62.3, 38.0, 32.3, 31.9, 28.9; **HR-MS (ESI)**: [C\textsubscript{15}H\textsubscript{18}NO\textsubscript{3}]\textsuperscript{+} ([M + H]\textsuperscript{+}): obs.: 260.1284; calcd.: 260.1281.

**Z)**-7,11-Dimethyldodec-4-ene-1,11-diol 6j

Following general procedure **C**: (Z)-11-Hydroxy-7,11-dimethyldodec-4-enoic acid (838 mg, 3.45 mmol, 1.00 equiv), LiAlH\textsubscript{4} (2.20 mL, 1.50 mmol, 4.00 equiv), THF (15 mL); 21 h; yield: 656 mg, 2.90 mmol, 83%, colorless liquid. The product is obtained as inseparable mixture Z/E.

**TLC**: $R_f = 0.92$ (n-pentane/EtOAc, 40:60); **IR** (neat): $\nu = 3345, 2933, 2868, 1460, 1376, 1198, 1158, 1058, 936, 907, 851, 763, 705, 382$ cm\textsuperscript{-1}; **\textsuperscript{1}H-NMR** (300 MHz, CDCl\textsubscript{3}): $\delta$ (ppm) = 5.43-5.31 (m, 2 H), 3.63 (t, $J = 6.5$ Hz, 2 H), 2.16-1.79 (m, 4 H), 1.72-1.05 (m, 19 H), 0.87 (d, $J = 6.6$ Hz, 3 H); **\textsuperscript{13}C-NMR** (126 MHz, CDCl\textsubscript{3}): $\delta$ (ppm) = major isomer: 129.8, 129.2, 71.2, 62.7, 44.3, 37.2, 34.5, 33.5, 32.8, 29.9, 29.4, 23.9, 21.9, 19.9; minor isomer: 130.8, 129.6, 62.6, 44.4, 40.2, 33.3, 32.7, 29.4, 29.1, 22.0, 19.7; **HR-MS** (ESI): [C\textsubscript{14}H\textsubscript{28}NaO\textsubscript{2}]\textsuperscript{+} ([M + Na]\textsuperscript{+}): obs.: 251.1983; calcd.: 251.1982.

**Z)**-1-Phenylhept-4-en-1-ol 6k

Phenylmagnesium bromide (1 M in THF; 6 mL, 6.00 mmol, 1.20 equiv) was added dropwise at 0°C to a solution of cis-4-hepten-1-al (561 mg, 5.00 mmol, 1.00 equiv) in dry Et\textsubscript{2}O (20 mL) and the solution was stirred at room temperature for 4 h. The reaction was quenched by the addition of 1 M aq. HCl (20 mL), the aq. phase was extracted with Et\textsubscript{2}O (3 x 20 mL) and the comb. org. phases were washed with H\textsubscript{2}O (50 mL) and sat. aq. NaCl (50 mL). They were dried over Na\textsubscript{2}SO\textsubscript{4}, the solvent was removed in vacuum and the product was afforded via column chromatography (n-pentane/EtOAc, 20:1) as a colourless oil (756 mg, 3.97 mmol, 79%).

**TLC**: $R_f = 0.20$ (n-pentane/EtOAc, 20:1); **IR** (neat): $\nu = 3347, 3006, 2961, 2933, 2870, 1493, 1453, 1202, 1060, 1027, 914, 754, 698$ cm\textsuperscript{-1}; **\textsuperscript{1}H-NMR** (300 MHz, CDCl\textsubscript{3}): $\delta$ (ppm) = 7.46-7.27 (m, 5 H), 5.56-5.22 (m, 2 H), 4.69 (dd, $J = 7.7$, 5.5 Hz, 1 H), 2.24-1.65 (m, 7 H), 0.96 (t, $J = 7.5$ Hz, 3 H); **\textsuperscript{13}C-NMR** (126 MHz, CDCl\textsubscript{3}): $\delta$ (ppm) = 144.7, 132.6, 128.5, 128.3, 127.6, 125.9, 74.3, 39.2, 23.8, 20.8, 14.5; **HR-MS** (ESI): [C\textsubscript{13}H\textsubscript{18}Na\textsubscript{+}]\textsuperscript{+} ([M + Na]\textsuperscript{+}): obs.: 213.1254; calcd.: 213.1250.
(Z)-1-(4-Fluorophenyl)hept-4-en-1-ol 6l

4-Fluorophenylmagnesium bromide (1 M in THF; 6 mL, 6.00 mmol, 1.20 equiv) was added dropwise at 0 °C to a solution of cis-4-hepten-1-ol (561 mg, 5.00 mmol, 1.00 equiv) in dry Et₂O (20 mL) and the solution was stirred at room temperature for 1 h. The reaction was quenched by the addition of 1 M aq. HCl (20 mL), the aq. phase was extracted with Et₂O (3 x 20 mL) and the comb. org. phases were washed with H₂O (50 mL) and sat. aq. NaCl (50 mL). They were dried over Na₂SO₄, the solvent was removed in vacuum and the product was afforded via column chromatography (n-pentane/EtOAc, 10:1) as a colourless oil (860 mg, 4.13 mmol, 83%).

TLC: \( R_f = 0.19 \) (n-pentane/EtOAc, 10:1); IR (neat): \( \nu = 3347, 3008, 2962, 2933, 2872, 1604, 1508, 1455, 1300, 1222, 1156, 1065, 1012, 833 \) cm⁻¹; \(^1\text{H-NMR} \) (300 MHz, CDCl₃): \( \delta \) (ppm) = 7.37-7.27 (m, 2 H), 7.10-6.96 (m, 2 H), 5.50-5.24 (m, 2 H), 4.67 (dd, \( J = 7.7, 5.5 \) Hz, 1 H), 2.21-1.93 (m, 5 H), 1.91-1.65 (m, 2 H), 0.95 (t, \( J = 7.5 \) Hz, 3 H); \(^{13}\text{C-NMR} \) (101 MHz, CDCl₃): \( \delta \) (ppm) = 162.3 (d, \( J = 245.2 \) Hz), 140.5 (d, \( J = 3.1 \) Hz), 132.8, 128.2, 127.6 (d, \( J = 8.0 \) Hz), 115.4 (d, \( J = 21.4 \) Hz), 73.7, 39.2, 23.7, 20.7, 14.4; \(^{19}\text{F-NMR} \) (282 MHz, CDCl₃): \( \delta \) (ppm) = -115.2 (tt, \( J = 8.6, 5.4 \) Hz); HR-MS (ESI): \([\text{C}_{13}\text{H}_{17}\text{OFNa}]^+ \) ([M + Na]⁺): obs.: 231.1158; calcd.: 231.1156.

(Z)-1-(4-Methoxyphenyl)hept-4-en-1-ol 6m

4-Methoxyphenylmagnesium bromide (0.5 M in THF; 12 mL, 6.00 mmol, 1.20 equiv) was added dropwise at 0 °C to a solution of cis-4-hepten-1-ol (561 mg, 5.00 mmol, 1.00 equiv) in dry Et₂O (20 mL) and the solution was stirred at room temperature for 1 h. The reaction was quenched by the addition of 1 M aq. HCl (20 mL), the aq. phase was extracted with Et₂O (3 x 20 mL) and the comb. org. phases were washed with H₂O (50 mL) and sat. aq. NaCl (50 mL). They were dried over Na₂SO₄, the solvent was removed in vacuum and the product was afforded via column chromatography (n-pentane/EtOAc, 5:1) as a colourless oil (837 mg, 3.80 mmol, 76%).

TLC: \( R_f = 0.24 \) (n-pentane/EtOAc, 5:1); IR (neat): \( \nu = 3379, 3006, 2962, 2932, 2870, 2839, 1611, 1584, 1510, 1461, 1303, 1243, 1173, 1033, 829 \) cm⁻¹; \(^1\text{H-NMR} \) (300 MHz, CDCl₃): \( \delta \) (ppm) = 7.45-7.09 (m, 2 H), 7.00-6.37 (m, 2 H), 5.62-4.98 (m,
\[ \text{Following general procedure C: (Z)-6-cyclohexylhex-5-enoic acid (530 mg, 2.70 mmol, 1.00 equiv), LiAlH}_4 \text{ (2.4 M in THF; 1.7 mL, 4.05 mmol, 1.50 equiv), THF (9 mL); 17.5 h; yield: 451 mg, 2.47 mmol, 91%, colorless liquid.} \]

**TLC:** \( R_f = 0.15 \) (n-pentane/Et\(_2\)O, 7:1); **IR** (neat): \( \nu = 3328, 2999, 2921, 2849, 1447, 1059, 993, 889, 729 \text{ cm}^{-1} \); **\(^1\)H-NMR** (400 MHz, CDCl\(_3\)): \( \delta \text{ (ppm)} = 5.53-4.73 \) (m, 2 H), 3.65 (t, \( J = 6.6 \text{ Hz, 2 H})\), 2.23 (tdq, \( J = 11.1, 7.8, 3.8 \text{ Hz, 1 H})\), 2.14-2.01 (m, 2 H), 1.82-1.64 (m, 2 H), 1.64-1.49 (m, 5 H), 1.49-1.37 (m, 2 H), 1.35-1.12 (m, 4 H), 1.10-0.97 (m, 2 H); **\(^13\)C-NMR** (101 MHz, CDCl\(_3\)): \( \delta \text{ (ppm)} = 136.6, 127.6, 63.1, 36.5, 33.5, 32.5, 27.3, 26.2, 26.1 \); **GC-MS** (EI): \([\text{C}_{12}\text{H}_{20}\text{O}]^+ \text{ (M}^+\text{): obs.}: 182.3; calcd.: 182.1671.**

\[ \text{Following general procedure C: (Z)-8-methylnon-5-enoic acid (500 mg, 2.94 mmol, 1.00 equiv), LiAlH}_4 \text{ (1.8 mL, 4.4 mmol, 1.5 equiv), THF (9 mL); 4 h; yield: 334 mg, 2.1 mmol, 71%, colorless liquid. The product is obtained as a mixture of E/Z isomers.} \]

**TLC:** \( R_f = 0.22 \) (n-pentane/Et\(_2\)O, 9:1); **IR** (neat): \( \nu = 3318, 3006, 2953, 2931, 2868, 1464, 1383, 1366, 1336, 1064, 989, 968, 937, 828, 707, 580 \text{ cm}^{-1} \); **\(^1\)H-NMR** (300 MHz, CDCl\(_3\)): \( \delta \text{ (ppm)} = 5.53-5.04 \) (m, 2 H), 3.61 (t, \( J = 6.5 \text{ Hz, 2 H})\), 2.04 (ddddd, \( J = 7.9, 6.5, 4.9, 0.6 \text{ Hz, 2 H})\), 1.95-1.76 (m, 3 H), 1.67-1.48 (m, 3 H), 1.48-1.22 (m, 2 H), 0.87 (d, \( J = 6.6 \text{ Hz, 6 H})\); **\(^13\)C-NMR** (126 MHz, CDCl\(_3\)): \( \delta \text{ (ppm)} = \text{major isomer: 130.1, 129.0, 62.9, 36.6, 32.6, 28.8, 27.2, 26.0, 22.6; minor isomer: 130.9, 129.3, 42.0, 32.3, 32.2, 28.5, 25.8, 22.3; GC-MS (EI): [C\(_{10}\)H\(_{20}\)O]\(^+ \text{ ([M}^+\text{]): obs.}: 156.2; calcd.: 156.2.} \]
(Z)-8,12-Dimethyltridec-5-ene-1,12-diol 6p

Following general procedure C: (Z)-12-Hydroxy-8,12-dimethyltridec-5-enoic acid (710 mg, 2.77 mmol, 1.00 equiv), LiAlH₄ (1.73 mL, 4.16 mmol, 1.50 equiv), THF (9 mL); 4.5 h; yield: 454 mg, 1.9 mmol, 68%, colorless liquid. The product is obtained as a mixture of E/Z isomers.

TLC: $R_f = 0.21$ (n-pentane/Et₂O, 1:1); IR (neat): $\nu = 3327, 2933, 2866, 1459, 1377, 1198, 1160, 1065, 937, 907, 688 \text{ cm}^{-1}$; $^1\text{H-NMR}$ (300 MHz, CDCl₃): $\delta$ (ppm) = 5.61-5.20 (m, 2 H), 3.63 (td, $J = 6.5, 0.6 \text{ Hz}$, 2 H), 2.03 (dt, $J = 13.5, 6.4, 5.4, 4.0 \text{ Hz}$, 3 H), 1.86 (m, 1 H), 1.67 (d, $J = 0.8 \text{ Hz}$, 2 H), 1.63-1.24 (m, 11 H), 1.20 (d, $J = 0.5 \text{ Hz}$, 6 H), 0.87 (dd, $J = 6.6, 0.5 \text{ Hz}$, 3 H); $^{13}\text{C-NMR}$ (126 MHz, CDCl₃): $\delta$ (ppm) = major isomer: 130.3, 128.8, 71.2, 63.0, 44.4, 37.3, 34.6, 33.6, 32.6, 29.5, 29.4, 27.2, 26.0, 22.0, 19.9; minor isomer: 131.3, 129.3, 63.0, 40.2, 37.2, 33.3, 32.5, 32.4, 25.9, 22.0, 19.8; HR-MS (ESI): [C₁₅H₃₀NaO₂]^+ ([M + Na]^+): obs.: 265.2139; calcd.: 265.2138.

(Z)-8-phenyloct-5-en-1-ol 6q

Following general procedure C: (Z)-8-Phenyloct-5-enoic acid (610 mg, 2.79 mmol, 1.00 equiv), LiAlH₄ (1.7 mL, 4.19 mmol, 1.50 equiv), THF (9 mL); 4 h; yield: 293 mg, 1.4 mmol, 51%, colorless liquid.

TLC: $R_f = 0.11$ (n-pentane/Et₂O, 9:1); IR (neat): $\nu = 3332, 3026, 3005, 2930, 2857, 1495, 1453, 1059, 1031, 746, 722, 696, 583 \text{ cm}^{-1}$; $^1\text{H-NMR}$ (300 MHz, CDCl₃): $\delta$ (ppm) = 7.32-7.20 (m, 2 H), 7.17 (d, $J = 7.0 \text{ Hz}$, 3 H), 5.62-5.01 (m, 2 H), 3.59 (t, $J = 6.5 \text{ Hz}$, 1 H), 2.64 (dd, $J = 8.7, 6.7 \text{ Hz}$, 2 H), 2.48-2.14 (m, 2 H), 2.07-1.83 (m, 2 H), 1.63-1.38 (m, 2 H), 1.32 (dddd, $J = 14.7, 11.0, 6.2, 2.2 \text{ Hz}$, 3 H); $^{13}\text{C-NMR}$ (126 MHz, CDCl₃): $\delta$ (ppm) = 142.2, 130.3, 129.2, 128.6, 128.4, 125.9, 63.0, 36.1, 32.5, 29.4, 27.0, 25.8, HR-MS (ESI): [C₁₄H₃₀NaO]^+ ([M + Na]^+): obs.: 227.1408; calcd.: 227.1406.

(Z)-10-chlorodec-5-en-1-ol 6r

Following general procedure C: (Z)-10-chlorodec-5-enoic acid (477 mg, 2.34 mmol, 1.00 equiv), LiAlH₄ (2.4 M in THF; 1.46 mL, 3.51 mmol, 1.50 equiv), THF (8 mL); 16.5 h; yield: 161 mg, 842 µmol, 36%, colorless liquid. The product is obtained as a mixture of E/Z isomers.
**TLC:** $R_f = 0.29$ (n-pentane/Et$_2$O, 3:2); **IR** (neat): $\nu = 3342, 3004, 2933, 2859, 1455, 1363, 1311, 1058, 715, 651$ cm$^{-1}$; **$^1$H-NMR** (300 MHz, CDCl$_3$): $\delta$ (ppm) = 5.57-5.19 (m, 2 H), 3.65 (t, $J = 6.5$ Hz, 2 H), 3.54 (t, $J = 6.7$ Hz, 2 H), 2.26-1.94 (m, 4 H), 1.94-1.69 (m, 2 H), 1.69-1.34 (m, 6 H); **$^{13}$C-NMR** (101 MHz, CDCl$_3$): $\delta$ (ppm) = major isomer: 130.2, 129.4, 63.1, 45.2, 32.6, 32.4, 27.2, 27.1, 26.6, 26.0; minor isomer: 130.7, 130.0, 32.5, 32.3, 31.9, 27.8, 25.9; **GC-MS** (El): [C$_{10}$H$_{17}$Cl]$^+$ (M-[H$_2$O]$^+$): obs.: 172.2; calcd.: 172.1.

**Z-Hexadeca-5,15-dien-1-ol 6s**

Following general procedure C: (Z)-hexadeca-5,15-dienoic acid (719 mg, 2.85 mmol, 1.00 equiv), LiAlH$_4$ (2.4 m in THF; 1.78 mL, 4.27 mmol, 1.50 equiv), THF (9 mL); 5.5 h; yield: 578 mg, 2.42 mmol, 85%, colorless liquid.

**TLC:** $R_f = 036$. (n-pentane/Et$_2$O, 3:1); **IR** (neat): $\nu = 3697, 3322, 3076, 3004, 2923, 2853, 2360, 2341, 1726, 1640, 1460, 136, 1060, 991, 908, 721$ cm$^{-1}$; **$^1$H-NMR** (400 MHz, CDCl$_3$): $\delta$ (ppm) = 5.81 (ddt, $J = 16.9, 10.2, 6.7$ Hz, 1 H), 5.55-5.26 (m, 2 H), 4.99 (ddt, $J = 17.1, 2.1, 1.7$ Hz, 1 H), 4.93 (ddt, $J = 10.2, 2.4, 1.2$ Hz, 1 H), 3.65 (t, $J = 6.6$ Hz, 2 H), 2.26-1.91 (m, 6 H), 1.71-1.52 (m, 2 H), 1.48-1.12 (m, 15 H); **$^{13}$C-NMR** (101 MHz, CDCl$_3$): $\delta$ (ppm) = 139.4, 130.5, 129.4, 114.2, 63.1, 34.0, 32.5, 29.9, 29.6, 29.6, 29.4, 29.3, 29.1, 27.4, 27.1, 26.0; **HR-MS** (El): [C$_{18}$H$_{31}$O]$^+$ ([M + H]$^+$): obs.: 239.2368; calcd.: 239.2369.

**Z-dodec-5-en-11-yn-1-ol 6t**

Following general procedure C: (Z)-dodec-5-en-11-ynoic acid (310 mg, 1.60 mmol, 1.00 equiv), LiAlH$_4$ (2.4 m in THF; 1.00 mL, 2.40 mmol, 1.50 equiv), THF (8 mL); 4 h; yield: 262 mg, 1.45 mmol, 91%, colorless liquid. Contains allene (10%).

**TLC:** $R_f = 022$. (n-pentane/Et$_2$O, 4:1); **IR** (neat): $\nu = 3304, 3005, 2931, 2858, 2117, 1955, 1653, 1456, 1433, 1327, 1057, 972, 939, 843$ cm$^{-1}$; **$^1$H-NMR** (300 MHz, CDCl$_3$): $\delta$ (ppm) = 5.54-5.28 (m, 2 H), 3.65 (t, $J = 6.5$ Hz, 2 H), 2.27-2.14 (m, 2 H), 2.14-1.97 (m, 4 H), 1.94 (t, $J = 2.7$ Hz, 1 H), 1.67-1.35 (m, 9 H); **$^{13}$C-NMR** (126 MHz, CDCl$_3$): $\delta$ (ppm) = 129.9, 129.8, 84.7, 68.4, 63.1, 32.6, 28.9, 28.3, 27.2, 26.9, 26.1, 18.5; **HR-MS** (El): [C$_{12}$H$_{20}$ONa]$^+$ ([M + Na]$^+$): obs.: 203.1401; calcd.: 203.1406.
8 Synthesis and analytical data of alkene substrates

(E)-Benzyl hex-3-enoate 6u

A solution of (E)-hex-3-en-1-ylphosphonate (9.05 g, 66.0 mmol, 1.10 equiv) was dissolved in dry THF (200 mL) and cooled to −10 °C. n-BuLi (2.5 M in hexane; 26.4 mL, 66.0 mmol, 1.1 equiv) was added dropwise and the mixture was stirred for 5 min. A solution of (E)-crotyl bromide (8.10 g, 60.0 mmol, 1.00 equiv) in dry THF (24 mL) was added dropwise, the solution was stirred for 15 min at −10 °C, allowed to warm up to 23 °C and stirred for 18 h. The reaction was quenched by the addition of sat. aq. NH₄Cl-sol. (25 mL), the aq. phase was extracted with Et₂O (3 x 25 mL), the comb. org. phases were washed with sat. aq. NaCl-sol. (3 x 40 mL) and dried over Na₂SO₄. Removal of the solvent in vacuum and vacuum distillation afforded the product as a colourless oil (7.06 g, 36.7 mmol, 61%).

Diethyl (E)-but-2-en-1-ylphosphonate 6v

Diethyl phosphite (9.05 g, 66.0 mmol, 1.10 equiv) was dissolved in dry THF (200 mL) and cooled to −10 °C. n-BuLi (2.5 M in hexane; 26.4 mL, 66.0 mmol, 1.1 equiv) was added dropwise and the mixture was stirred for 5 min. A solution of (E)-crotyl bromide (8.10 g, 60.0 mmol, 1.00 equiv) in dry THF (24 mL) was added dropwise, the solution was stirred for 15 min at −10 °C, allowed to warm up to 23 °C and stirred for 18 h. The reaction was quenched by the addition of sat. aq. NH₄Cl-sol. (25 mL), the aq. phase was extracted with Et₂O (3 x 25 mL), the comb. org. phases were washed with sat. aq. NaCl-sol. (3 x 40 mL) and dried over Na₂SO₄. Removal of the solvent in vacuum and vacuum distillation afforded the product as a colourless oil (7.06 g, 36.7 mmol, 61%).
$J = 7.8, 7.1, 3.9$ Hz, $4$ H), $5.41$ (m, $1$ H), $5.61$ (dddt, $J = 15.2, 6.4, 5.1, 1.3$ Hz, $1$ H);
$^{13}$C-NMR (126 MHz, CDCl$_3$): $\delta$ (ppm) = $16.6$ (d, $J = 6.0$ Hz), $18.2$ (d, $J = 2.6$ Hz), $30.6$ (d, $J = 140.0$ Hz), $61.9$ (d, $J = 6.8$ Hz), $119.7$ (d, $J = 11.4$ Hz), $130.8$ (d, $J = 14.7$ Hz);
$^{31}$P-NMR (203 MHz, CDCl$_3$): $\delta$ (ppm) = $28.1$; HR-MS (ESI): [C$_8$H$_{18}$O$_3$P]$^+ ([M + H]^+)$: obs.: 193:0992; calcd.: 193:0988.

9 References


10 $^1$H-NMR/$^{13}$C-NMR/IR-spectra

1,2-Bis(4-(trifluoromethyl)phenyl)diselenide $^1$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz), $^{77}$Se-NMR (76 MHz): CDCl$_3$
1,2-Di-p-tolyl diselenide $^1$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz), $^{77}$Se-NMR (76 MHz): CDCl$_3$
1,2-Bis(4-methoxyphenyl)diselenide $^1$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz), $^{77}$Se-NMR (76 MHz): CDCl$_3$
1,2-Bis(2-methoxyphenyl)diselenide $^{1}$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz), $^{77}$Se-NMR (76 MHz): CDCl$_3$
2,4,6-Tris(4-methoxyphenyl)pyrylium tetrafluoroborate $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
2-(But-3-en-1-yl)isoindoline-1,3-dione, $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz):

CDCl$_3$
Hept-6-ynal, $^1$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz): CDCl$_3$
(Z)-7-Phenylhept-4-enoic acid, $^1$H-NMR (300 MHz), $^{13}$C-NMR (76 MHz): CDCl$_3$
Methyl 2,2-diphenylhex-4-enoate. $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(Z)-5-Cyclohexylpent-4-enoic acid, $^1$H-NMR (300 MHz), $^{13}$C-NMR (76 MHz): CDCl$_3$
(Z)-11-Hydroxy-7,11-dimethyldodec-4-enoic acid, $^1$H-NMR (300 MHz), $^{13}$C-NMR (76 MHz): CDCl$_3$
(E)-6-Cyclohexylhex-5-enoic acid $^1$H-NMR (500 MHz), $^{13}$C-NMR (76 MHz): CDCl$_3$
(Z)-8-Methylnon-5-enoic acid, $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(Z)-12-Hydroxy-8,12-dimethyltridec-5-enoic acid, $^1\text{H-NMR}$ (300 MHz), $^{13}\text{C-NMR}$ (76 MHz): CDCl$_3$
(Z)-8-Phenylcho-5-enoic acid $^1$H-NMR (300 MHz), $^{13}$C-NMR (76 MHz): CDCl$_3$
(Z)-10-Chlorodec-5-enoic acid, $^1$H-NMR (500 MHz), $^{13}$C-NMR (101 MHz): CDCl$_3$
(Z)-Hexadeca-5,15-dienoic acid $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(Z)-Dodec-5-en-11-ynoic acid $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(Z)-7-Phenylhept-4-en-1-ol 6b, $^1$H-NMR (300 MHz), $^{13}$C-NMR (76 MHz): CDCl$_3$
2,2-Diphenylhex-4-en-1-ol 6c \( ^1\text{H-NMR} \) (400 MHz), \( ^{13}\text{C-NMR} \) (100 MHz): CDC\textsubscript{3}
(Z)-5-Cyclohexylpent-4-en-1-ol 6d \(^1\)H-NMR (300 MHz), \(^{13}\)C-NMR (126 MHz): CDCl\(_3\)
(Z/E)-9-Hydroxynon-5-enenitrile 6e $^1$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz): CDCl$_3$
(E)-Oct-4-ene-1,8-diol 6f $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$

HO-\(\overset{\cdot}{\overset{\cdot}{\cdot}}\)OH

$^1$H-NMR spectrum

$^{13}$C-NMR spectrum
8-Hydroxyoct-4-en-1-yl pivalate 6g \( ^1H\)-NMR (300 MHz), \( ^{13}C\)-NMR (126 MHz): C\(\text{DCl}_3\)
Ethyl (8-hydroxyoct-4-en-1-yl) carbonate 6h $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl₃
2-(7-Hydroxyhept-3-en-1-yl)isoindoline-1,3-dione 6i  \( ^1\)H-NMR (300 MHz), \( ^{13}\)C-NMR (126 MHz): CDCl₃
(Z)-7,11-Dimethyldodec-4-ene-1,11-diol 6j, $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(Z)-1-Phenyleth-4-en-1-ol 6k $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(Z)-1-(4-Fluorophenyl)hept-4-en-1-ol 6l

$^1$H-NMR (300 MHz), $^{19}$F-NMR (282 MHz), $^{13}$C-NMR (101 MHz): CDCl$_3$
(Z)-1-(4-Methoxyphenyl)hept-4-en-1-ol 6m ¹H-NMR (300 MHz), ¹³C-NMR (101 MHz): CDCl₃
(Z)-6-cyclohexylhex-5-en-1-ol 6n $^1$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz): CDCl₃
(Z)-8-Methylnon-5-en-1-ol 6o \( ^1H\text{-NMR} \) (300 MHz), \( ^{13}C\text{-NMR} \) (126 MHz): CDCl\(_3\)
(Z)-8,12-Dimethyltridec-5-ene-1,12-diol 6p \( ^1H\)-NMR (300 MHz), \( ^{13}C\)-NMR (126 MHz): CDCl₃
(Z)-8-Phenyloct-5-en-1-ol 6q \(^1\)H-NMR (300 MHz), \(^{13}\)C-NMR (126 MHz): CDCl\(_3\)
(Z)-10-chlorodec-5-en-1-ol 6r $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(Z)-Hexadeca-5,15-dien-1-ol 6s \(^1\)H-NMR (300 MHz), \(^{13}\)C-NMR (126 MHz): CDCl\(_3\)
(Z)-dodec-5-en-11-yn-1-ol 6\textsuperscript{1}\textsuperscript{H}-NMR (300 MHz), 13\textsuperscript{C}-NMR (126 MHz): CDCl\textsubscript{3}
(E)-Benzyl hex-3-enoate 6u $^1$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz): CDCl$_3$
Diethyl (E)-but-2-en-1-ylphosphonate 6v $^1$H-NMR (500 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(E)-2-(Hex-1-en-1-yl)tetrahydrofuran 7a \(^1\text{H-NMR}\) (400 MHz), \(^{13}\text{C-NMR}\) (101 MHz): CDCl\(_3\)
(E)-2-(3-Phenylprop-1-en-1-yl)tetrahydrofuran 7b \(^1\)H-NMR (300 MHz), \(^{13}\)C-NMR (126 MHz): CDCl\(_3\)
4,4-Diphenyl-2-vinyltetrahydrofuran 7c $^1$H-NMR (300 MHz), $^{13}$C-NMR (76 MHz): CDCl₃
2-(Dyclohexylidenemethyl)tetrahydrofuran 7d $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(E)-5-(Tetrahydrofuran-2-yl)pent-4-enenitrile 7e $^1$H-NMR (500 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(E)-4-(Tetrahydrofuran-2-yl)but-3-en-1-ol 7f $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(E)-4-(Tetrahydrofuran-2-yl)but-3-en-1-yl pivalate 7g  $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(E)-Ethyl (4-(tetrahydrofuran-2-yl)but-3-en-1-yl) carbonate 7h $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(E)-2-(3-(Tetrahydrofuran-2-yl)allyl)isoindoline-1,3-dione 7i ¹H-NMR (500 MHz), ¹³C-NMR (126 MHz): CDCl₃
(E)-2,6-Dimethyl-8-(tetrahydrofuran-2-yl)oct-7-en-2-ol 7j \[^1H\text{-NMR} (300 \text{ MHz}), \[^{13}C\text{-NMR} (126 \text{ MHz})\]: CDCl\text{3}
(E)-2-Phenyl-5-(prop-1-en-1-yl)tetrahydrofuran 7k \textsuperscript{1}H-NMR (300 MHz), \textsuperscript{13}C-NMR (126 MHz): CDCl\textsubscript{3}
(E)-2-(4-Fluorophenyl)-5-(prop-1-en-1-yl)tetrahydrofuran 71 \(^1\)H-NMR (300 MHz).

\(^{19}\)F-NMR (282 MHz), \(^{13}\)C-NMR (126 MHz): CDCl\(_3\)
(E)-2-(4-Methoxyphenyl)-5-(prop-1-en-1-yl)tetrahydrofuran 7m \(^1\)H-NMR (300 MHz), \(^{13}\)C-NMR (126 MHz): CDCl\(_3\)
2-(Cyclohexylidenemethyl)tetrahydro-2H-pyran 7n \(^1\)H-NMR (300 MHz), \(^{13}\)C-NMR (126 MHz): CDCl\(_3\)
(E)-2-(3-Methylbut-1-en-1-yl)tetrahydro-2H-pyran 7o $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCls
(E)-2,6-Dimethyl-8-(tetrahydro-2H-pyran-2-yl)oct-7-en-2-ol 7p $^1$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz): CDCl$_3$
(E)-2-(3-Phenylprop-1-en-1-yl)tetrahydro-2H-pyran 7q \textsuperscript{1}H-NMR (300 MHz), \textsuperscript{13}C-NMR (126 MHz): CDCl\textsubscript{3}
(E)-2-(5-Chloropent-1-en-1-yl)tetrahydro-2H-pyran 7r $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(E)-2-(undeca-1,10-dien-1-yl)tetrahydro-2H-pyran 7s $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(E)-2-(Hept-1-en-6-yn-1-yl)tetrahydro-2H-pyran 7t \(^1\)H-NMR (300 MHz), \(^{13}\)C-NMR (126 MHz): CDCl\(_3\)
Benzyl (E)-4-methoxyhex-2-enoate 7ua $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl₃
Benzyl (E)-4-ethoxyhex-2-enoate 7ub $^1$H-NMR (300 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
Benzyl (E)-4-isopropoxyhex-2-enoate 7uc $^1$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz): CDCl$_3$
Diethyl (E)-(3-methoxybut-1-en-1-yl)phosphonate 7va $^1$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz): CDCl$_3$
Diethyl (E)-(3-ethoxybut-1-en-1-yl)phosphonate 7vb $^1$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz): CDCl$_3$
Diethyl (E)-(3-isopropoxybut-1-en-1-yl)phosphonate 7vc $^1$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz): CDCl$_3$
Diethyl (E)-(3-((4-(trifluoromethyl)benzyl)oxy)but-1-en-1-yl)phosphonate 7vd ¹H-NMR (400 MHz), ¹³C-NMR (101 MHz): CDCl₃
Diethyl (E)-(3-((perfluorophenyl)methoxy)but-1-en-1-yl)phosphonate 7ve $^1$H-NMR (400 MHz), $^{13}$C-NMR (126 MHz): CDCl$_3$
(E/Z)-4-((4-(Trifluoromethyl)benzyl)oxy)pent-2-enenitrile 7wd $^1$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz): CDCl$_3$

Spectra with $E$ as the major isomer:
(E/Z)-4-((4-(Trifluoromethyl)benzyl)oxy)pent-2-enenitrile 7wd $^1$H-NMR (400 MHz), $^{13}$C-NMR (101 MHz): CDCl$_3$

Spectra with Z as the major isomer: