Supplementary Information

Convergent synthesis of conjugated 1,2-disubstituted E-allylic alcohols from two aldehydes and methylenetriphenylphosphorane

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1. General Details

All reactions requiring anhydrous conditions were carried out under an atmosphere of nitrogen in flame-dried glassware.

**Materials:** THF was distilled from sodium and benzophenone under nitrogen. Petrol refers to the fraction of petroleum ether that boils at 30–40 °C. All other solvents were used as obtained. PhLi (2.0 M in Bu₂O) was obtained from Acros Organics®. Benzaldehyde and furfural were distilled under reduced pressure (64 °C, 20 mbar and 61 °C, 33 mbar, respectively) prior to use. LiBr was made anhydrous by heating under nitrogen until it melted then cooled and dissolved in THF before using. Methyltriphenylphosphonium bromide was dried overnight (~12 h) under high vacuum (~1 mbar). Other starting materials were obtained commercially and were used without further purification unless otherwise indicated.

**Chromatography:** Thin Layer Chromatography was carried out on aluminum-backed plates pre-coated with silica (60 F₂₅₄, Merck) and visualised by irradiation under UV light (λ = 254 nm) and by immersion in phosphomolybdic acid (PMA) or KMnO₄ solutions followed by heating. Purification of reaction products was carried out by flash chromatography using silica gel (40-63μm) in the solvent systems indicated.

**IR spectra** were recorded as thin films using a 1750 Perkin-Elmer Paragon Fourier Transform spectrometer. The strength of absorbance is designated by the following abbreviations: br, s, m, and w, which refer to broad, strong, medium and weak, respectively.

**1H and 13C NMR spectra** were recorded using Brüker AV400 and AVC500 spectrometers. Chemical shifts are reported in ppm and referenced to internal residual CHCl₃ at 7.27 ppm for 1H NMR spectra, and to the central line of the CDCl₃ triplet at 77.0 for 13C NMR spectra. Coupling constants, J, are given in Hz to the nearest 0.1 Hz. The 13C NMR peaks were assigned by standard methods using HSQC and DEPT experiments. E:Z ratios of allylic alcohols were determined by 1H NMR analysis of crude products.

**Mass spectra** were obtained by field ionisation (FI; Micromass GCT) or by electrospray ionisation (ESI; LCT Premier Reflectron TOF and Brüker MicroTOF) using tetraoctylammonium bromide or sodium dodecyl sulfate as lock mass; values are quoted as ratios of mass:charge (m/z) in Daltons, and relative intensities of assignable peaks observed are quoted as a percentage value.
2. Synthesis of trisubstituted \( \text{E-allylic} \) alcohols 4

**General Procedure A**

A solution of anhydrous LiBr (2 equiv) in THF (7.5 mL/mmol LiBr) was added to anhydrous ethyltriphenylphosphonium bromide (1.0 equiv) at room temperature. The mixture was stirred for 10 min before cooling to \(-78^\circ \text{C}\). PhLi (2.0 M in Bu\(_2\)O, 1.0 equiv) was then added dropwise at \(-78^\circ \text{C}\). The cooling bath was removed, and the mixture allowed to warm to rt over 15 min during which time a clear-yellow solution formed. After 30 min at rt, the mixture was re-cooled to \(-78^\circ \text{C}\) and a solution of the first aldehyde (1.0 equiv) in THF (0.5 mL/mmol aldehyde) was added dropwise. After 20 min, when complete decolourisation had occurred, PhLi (2.0 M in Bu\(_2\)O, 1.05 equiv) was added dropwise resulting in a cherry-red solution. This solution was stirred for 30 min at \(-78^\circ \text{C}\), after which time a solution of the second aldehyde (1.05 equiv) in THF (0.5 mL/mmol aldehyde) was added dropwise at \(-78^\circ \text{C}\). The reaction mixture was stirred at \(-78^\circ \text{C}\) for 2 h. The reaction mixture was then warmed to rt over 1 h and stirring continued for a further 1 h. The mixture was quenched with saturated aq ammonium chloride (20 mL), extracted with Et\(_2\)O (3 × 15 mL), dried (MgSO\(_4\)) and evaporated under reduced pressure. Purification of the crude product by flash chromatography in the solvent system indicated gave the trisubstituted \( \text{E-allylic} \) alcohol.

\( \text{(E)-3-Methyldodec-3-en-2-ol (4a)} \)

Following **General Procedure A**, ethyltriphenylphosphonium bromide (371 mg, 1.0 mmol) was first reacted with acetaldehyde (56 µL, 1.0 mmol) and then nonanal (149 mg, 1.05 mmol) to give allylic alcohol 4a (115 mg, 58%) as a pale yellow oil; \( R_f \) 0.35 (20% Et\(_2\)O/petrol); IR (film) /cm\(^{-1}\): 3340w, 2922m, 2854m, 2360w, 1717w, 1457w, 1368w, 1077m, 887w.  

\[^1\text{H} (400 \text{ MHz}) \delta = 5.40 \ (1 \text{ H, t, } J = 7.1, \text{C=CH}), 4.20 \ (1 \text{ H, q, } J = 6.3, \text{CH(OH)}), 2.00 \ (2 \text{ H, q, } J = 7.1, =\text{CHCH}_2\), 1.62 \ (3 \text{ H, s, } =\text{CCH}_3\), 1.56 \ (1 \text{ H, br. s, OH}), 1.38-1.21 \ (15 \text{ H, m, } 6 \times \text{CH}_2 \text{ and CH}_3\text{CH(OH)}), 0.88 \ (3 \text{ H, t, } J = 6.8, \text{CH}_2\text{CH}_3\).  

\[^{13}\text{C} (100 \text{ MHz}) \delta = 138.2 \ (=\text{CCH}_3\), 125.4 \ (=\text{CH}), 73.5 \ (=\text{CH(OH)})), 31.9 \ (\text{CH}_2\), 29.5 \ (2 \times \text{CH}_2\), 29.3 \ (\text{CH}_2\), 29.2 \ (\text{CH}_2\), 27.5 \ (=\text{CHCH}_2\), 22.65 \ (\text{CH}_2\), 21.6 \ (=\text{CCH}_3\), 14.1 \ (\text{CH}_2\text{CH}_3\), 11.4 \ (\text{CH}_3\text{CH(OH)}). 

LRMS (ESI+): 221.3 \ (15, \text{M+Na}), 253.3 \ (45), 413.3 \ (100); HRMS (ESI+): \text{(M+Na)} 221.1876 calculated for \( \text{C}_{13}\text{H}_{26}\text{ONa} \); found 221.1879.
NOEs observed for 4a:

![Diagram](image)

(E)-3-Methyldodec-2-en-4-ol (4b)

Following General Procedure A, ethyltriphenylphosphonium bromide (371 mg, 1.0 mmol) was first reacted with nonanal (142 mg, 1.0 mmol) and then acetaldehyde (59 µL, 1.05 mmol) to give allylic alcohol 4b\(^1\) (126 mg, 64%) as a pale yellow oil; \(R_f\) 0.35 (20% Et\(_2\)O/petrol);

IR (neat)/\(\text{cm}^{-1}\): 3343w, 2923m, 2855m, 1457w, 1378w, 1000w, 828w.

\(^1\)H (400 MHz) \(\delta = 5.44\) (1 H, q, \(J = 6.3\), C=CH), 3.96 (1 H, app. t, \(J = 6.8\), CH(OH)), 1.56-1.65 (6 H, m, 2 \(\times\) CH\(_3\)), 1.55-1.46 (2 H, m, CH(OH)CH\(_2\)), 1.35-1.14 (12 H, m, 6 \(\times\) CH\(_2\)), 0.87 (3 H, t, \(J = 6.8\), CH\(_2\)CH\(_3\)).

\(^{13}\)C (100 MHz) \(\delta = 138.0\) (=CCH\(_3\)), 120.7 (=CH), 78.1 (CH(OH)), 34.8 (CH\(_2\)CH(OH)), 31.8 (CH\(_2\)), 29.6 (CH\(_2\)), 29.5 (CH\(_2\)), 29.3 (CH\(_2\)), 25.8 (CH\(_2\)), 22.6 (CH\(_2\)), 14.1 (CH\(_2\)CH\(_3\)), 13.0 (CH\(_3\)), 10.7 (CH\(_3\)).

LRMS (ESI+): 142.3 (20), 235.3 (45), 413.3 (100); HRMS (FI+): 198.1984 calculated for C\(_{13}\)H\(_{26}\)O; found 198.1991.

NOEs observed for 4b:

![Diagram](image)
3. Synthesis of disubstituted \(E\)-allylic alcohols 7 - 14

**General Procedure B**

A solution of anhydrous LiBr (2 equiv) in THF (7.5 mL/mmol LiBr) was added to anhydrous methyltriphenylphosphonium bromide (1.0 equiv) at room temperature. The mixture was stirred for 10 min before cooling to \(\sim 78 ^\circ C\). PhLi (2.0 M in Bu\(_2\)O, 1.0 equiv) was then added dropwise at \(\sim 78 ^\circ C\). The cooling bath was removed, and the mixture allowed to warm to rt over 15 min during which time a clear-yellow solution formed. After 30 min at rt, the mixture was re-cooled to \(\sim 78 ^\circ C\) and a solution of the first aldehyde (1.0 equiv) in THF (0.5 mL/mmol aldehyde) was added dropwise. After 20 min, when complete decolourisation had occurred, PhLi (2.0 M in Bu\(_2\)O, 1.05 equiv) was added dropwise resulting in a cherry-red solution. This solution was stirred for 30 min at \(\sim 78 ^\circ C\), after which time a solution of the second aldehyde (1.05 equiv) in THF (0.5 mL/mmol aldehyde) was added dropwise at \(\sim 78 ^\circ C\). The reaction mixture was then warmed to rt over 1 h and stirring continued for a further 1 h. The mixture was quenched with saturated aq ammonium chloride (20 mL), extracted with Et\(_2\)O (3 × 15 mL), dried (MgSO\(_4\)) and evaporated under reduced pressure. Purification of the crude product by flash chromatography in the solvent system indicated gave the disubstituted \(E\)-allylic alcohol.

**Dodec-3-en-2-ol (7) and Dodec-2-en-4-ol (8)**

Following **General Procedure B**, methyltriphenylphosphonium bromide (357 mg, 1.0 mmol) was first reacted with acetaldehyde (56 \(\mu\)L, 1.0 mmol) and then nonanal (149 mg, 1.05 mmol) to give allylic alcohols 7 and 8 as a mixture of chromatographically inseparable isomers (102 mg, 55\% \(7E^2:7Z^3:8Z\) 60:33:7 by \(^1\)H NMR integration of \(CH(OH)\)) as a pale yellow oil; R\(_f\) 0.32 (25\% Et\(_2\)O/petrol);

IR (neat) /cm\(^{-1}\): 3339w, 2923m, 2854m, 2360w, 1457w, 1367w, 1059m, 967m, 721w.

LRMS (ESI+): 207.1 (25, M+Na), 281.3 (100), 413.3 (100); HRMS (ESI+): (M+Na) 207.1723 calculated for C\(_{12}\)H\(_{24}\)ONa; found 207.1719.

Data for \(7E\):

\(^1\)H (400 MHz) \(\delta = 5.63 (1 \)H, dt, \(J = 15.4, 6.8, =CHCH\(_2\))\), 5.50 (1 H, dd, \(J = 15.4, 6.8, CH(OH)CH\(_2\))\), 4.25 (1 H, app. quin, \(J = 6.4, CH(OH)\)), 2.01, (2 H, quin, \(J = 6.9, =CHCH\(_2\))\), 1.60 (1 H, br. s, OH), 1.40-1.21 (15 H, m, 6 × CH\(_2\) and CH(OH)CH\(_3\))\), 0.88 (3 H, t, \(J = 6.7, CH\(_2\)CH\(_3\))\).
$^{13}$C (100 MHz) $\delta$ = 134.0 (CH(OH)CH), 131.2 (\(=\text{CHCH}_2\)), 69.0 (CH(OH)), 32.1 (CH$_2$), 31.9 (CH$_2$), 29.4 (CH$_2$), 29.2 (CH$_2$), 29.2 (CH$_2$), 23.4 (CH$_3$CH(OH)), 22.6 (CH$_2$), 14.1 (CH$_3$).

Discernible data for 7Z:
$^1$H (400 MHz, CDCl$_3$) $\delta$ = 5.44-5.37 (2 H, m, CH=CH), 4.64 (1 H, app. quin, $J$ = 6.5, CH(OH)), 2.07 (2 H, ddd, $J$ = 12.8, 6.3, 2.1, \(=\text{CHCH}_2\)), 1.60 (1 H, br, s, OH), 1.40-1.21 (15 H, m, 6 $\times$ CH$_2$, and CH(OH)CCH$_3$), 0.68 (3 H, t, $J$ = 6.7, CH$_2$CH$_3$).

$^{13}$C (100 MHz, CDCl$_3$) $\delta$ = 133.7 (CH(OH)CH), 131.4 (\(=\text{CHCH}_2\)), 63.9 (CH(OH)), 31.9 (CH$_2$), 29.7 (CH$_2$), 29.4 (CH$_2$), 29.2 (CH$_2$), 27.5 (CH$_2$), 23.6 (CH$_3$CH(OH)), 22.6 (CH$_2$), 14.1 (CH$_3$).

Dodec-2-en-4-ol (8) and Dodec-3-en-2-ol (7)

Following General Procedure B, methyltriphenylphosphonium bromide (357 mg, 1.0 mmol) was first reacted with nonanal (142 mg, 1.0 mmol) and then acetaldehyde (59 $\mu$L, 1.05 mmol) to give allylic alcohols 8 and 7 as a mixture of chromatographically inseparable isomers (106 mg, 58%, 8$^E$:8$^Z$:7$^E$:7$^Z$ 46:13:4:37 by $^1$H NMR integration of CH(OH)) as a pale yellow oil; R$_f$ 0.32 (25% Et$_2$O/petrol);

IR (neat) /cm$^{-1}$: 3355w, 2924m, 2854m, 2360w, 1456w, 1377w, 1109w, 908m, 732s.

LRMS (ESI+): 207.1 (15, M+Na), 253.3 (30), 413.3 (100); HRMS (ESI+): (M+Na) 207.1723 calculated for C$_{12}$H$_{24}$ONa; found 207.1718.

Data for 8$^E$:
$^1$H (400 MHz) $\delta$ = 5.64 (1 H, dq, $J$ = 15.5, 6.0, \(=\text{CHCH}_3\)), 5.47 (1 H, dd, $J$ = 15.5, 6.9, CH(OH)CH), 4.01 (1 H, app. q, $J$ = 6.6, CH(OH)), 1.72-1.64 (4 H, m, \(=\text{CHCH}_3\), OH), 1.60-1.40 (2 H, m, CH(OH)CH$_2$), 1.39-1.17 (12 H, m, 6 $\times$ CH$_2$), 0.87 (3 H, t, $J$ = 6.7, CH$_3$).

$^{13}$C (100 MHz) $\delta$ = 134.4 (CH(OH)CH), 126.6 (\(=\text{CHCH}_3\)), 73.1 (CH(OH)), 37.2 (CH$_2$CH(OH)), 31.9 (CH$_2$), 29.6 (CH$_2$), 29.4 (CH$_2$), 25.5 (CH$_2$), 22.6 (CH$_2$), 17.6 (\(=\text{CHCH}_3\)), 14.1 (CH$_3$).

Discernible data for 8$^Z$:
$^1$H (400 MHz, CDCl$_3$) $\delta$ = 5.42-5.34 (2 H, m, CH=CH), 4.49-4.41 (1 H, m, CH(OH)), 1.72-1.64 (4 H, m, CH$_2$CH$_3$, OH), 1.59-1.40 (2 H, m, CH(OH)CH$_2$), 1.39-1.17 (12 H, m, 6 $\times$ CH$_2$), 0.87 (3 H, t, $J$ = 6.7, CH$_3$).
$^{13}$C (100 MHz, CDCl$_3$) $\delta$ = 133.6 (CH(OH)CH), 126.1 ($\equiv$CHCH$_3$), 67.3 (CH(OH)), 37.4 (CH$_2$CH(OH)), 29.6 (CH$_2$), 27.5 (CH$_2$), 25.3 (CH$_2$), 17.6 (CH$_3$), 13.2 ($\equiv$CHCH$_3$).

1-Phenylocta-4,6-dien-3-ol (9a)

Following **General Procedure B**, methyltriphenylphosphonium bromide (357 mg, 1.0 mmol) was first reacted with 3-phenylpropanal (134 mg, 1.0 mmol) and then crotonaldehyde (74 mg, 1.05 mmol) to give allylic alcohol 9a as a mixture of chromatographically inseparable isomers (103 mg, 51%). **9aE:9aZ** 81:19 by $^1$H NMR integration of CH(OH)) as a pale yellow oil; R$_f$ 0.23 (20% Et$_2$O/petrol);

IR (neat) /cm$^{-1}$: 3366 br. w, 3024w, 2915w, 1495w, 1453w, 986s, 745s.

LRMS (ESI+): 225.14 (55, M+Na), 301.09 (100), 579.19 (67); HRMS (ESI+): (M+Na) 225.1250 calculated for C$_{14}$H$_{18}$ONa; found 225.1244.

**Data for 9aE:**

$^1$H (400 MHz) $\delta$ = 7.34-7.17 (5 H, m, Ph), 6.21 (1 H, dd, $J$ = 15.2, 10.6, CH(OH)CH=CH), 6.14-6.02 (1 H, m, CH=CHCH$_3$), 5.74 (1 H, dq, $J$ = 15.0, 6.8, $\equiv$CHCH$_3$), 5.62 (1 H, dd, $J$ = 15.2, 7.1, CH(OH)CH=CH), 4.15 (1 H, app. q, $J$ = 6.6, CH(OH)), 2.80-2.64 (2 H, m, PhCH$_2$), 2.02-1.82 (2 H, m, CH(OH)CH$_2$), 1.79 (3 H, d, $J$ = 6.6, CH$_3$), 1.77 (1 H, br. s, OH).

$^1$H (400 MHz) $\delta$ = 7.34-7.17 (5 H, m, Ph), 6.33-6.23 (1 H, m, CH=CHCH$_3$), 6.14-6.02 (1 H, m, CH(OH)CH=CH), 5.78 (1 H, dq, $J$ = 15.0, 6.8, $\equiv$CHCH$_3$), 5.38-5.29 (1 H, m, CH(OH)CH=CH), 4.60 (1 H, app. q, $J$ = 7.5, CH(OH)), 2.80-2.64 (2 H, m, PhCH$_2$), 2.02-1.82 (2 H, m, CH(OH)CH$_2$), 1.79 (3 H, d, $J$ = 6.6, CH$_3$), 1.77 (1 H, br. s, OH).

Discernible data for **9aZ**:

$^1$H (400 MHz) $\delta$ = 7.34-7.17 (5 H, m, Ph), 6.14-6.02 (1 H, m, CH(OH)CH=CH), 5.78 (1 H, dq, $J$ = 15.0, 6.8, $\equiv$CHCH$_3$), 5.38-5.29 (1 H, m, CH(OH)CH=CH), 4.60 (1 H, app. q, $J$ = 7.5, CH(OH)), 2.80-2.64 (2 H, m, PhCH$_2$), 2.02-1.82 (2 H, m, CH(OH)CH$_2$), 1.79 (3 H, d, $J$ = 6.6, CH$_3$), 1.77 (1 H, br. s, OH).

$^{13}$C (100 MHz) $\delta$ = 141.8 (Ph), 131.8 ($\equiv$CHCH$_3$), 130.8 (CH(OH)CH=CH), 130.5 (CH(OH)CH), 128.4 (Ph), 128.3 (Ph), 126.5 ($\equiv$CHCH$_3$), 125.9 (Ph), 72.0 (CH(OH)), 38.7 (CH(OH)CH$_2$), 31.7 (PhCH$_2$), 18.1 (CH$_3$).
(E)-1-(Cyclohex-1-en-1-yl)-5-phenylpent-1-en-3-ol (9b)

Following General Procedure B, methyltriphenylphosphonium bromide (714 mg, 2.0 mmol) was first reacted with 3-phenylpropanal (268 mg, 2.0 mmol) and then 1-cyclohexene-1-carboxaldehyde (231 mg, 2.1 mmol) to give allylic alcohol 9b (215 mg, 44%) as a pale yellow oil; \( R_f \) 0.31 (20% Et\(_2\)O/petrol);

IR (neat) /cm\(^{-1}\): 3334br. w, 2926w, 1649w, 1495w, 1453w, 1027m, 964m, 744m, 698s.

\(^1\)H (400 MHz) \( \delta = 7.35-7.18 \) (5 H, m, Ph), 6.23 (1 H, d, \( J = 15.7 \), CH(OH)CH=CH), 5.79 (1 H, app. br. s, C=CHCH\(_2\)), 5.60 (1 H, dd, \( J = 15.7, 7.2 \), CH(OH)CH\(_2\)), 4.18 (1 H, app. q, \( J = 6.7 \), CH(OH)), 2.82-2.66 (2 H, m, PhC\(_2\)H\(_2\)), 2.21-2.11 (4 H, m, 2 \times CH\(_2\) cyclohexenyl), 2.03-1.81 (3 H, m, CH(OH)CH\(_2\) and OH), 1.76-1.67 (2 H, m, CH\(_2\) cyclohexenyl), 1.67-1.59 (2 H, m, CH\(_2\) cyclohexenyl).

\(^{13}\)C (100 MHz) \( \delta = 141.9 \) (Ph), 134.9 (C=CH), 134.4 (CH(OH)CH=CH), 130.0 (C=CHCH\(_2\)), 128.4 (2 \times Ph), 128.2 (2 \times Ph), 128.0 (CH(OH)CH), 125.6 (Ph), 72.5 (CH(OH)CH\(_2\)), 38.9 (PhCH\(_2\)), 31.7 (CH(OH)C\(_2\)H\(_2\)), 25.8 (=CCH\(_2\)), 2.02 (3 H, s, CH\(_3\)), 2.06-1.88 (2 H, m, PhCH\(_2\)), 1.69 (1 H, br. s, OH).

LRMS (ESI+): 265.15 (100, M+Na), 266.18 (70), 389.24 (48), 507.35 (63, 2M+Na); HRMS (ESI+): (M+Na) calculated for C\(_{17}\)H\(_{22}\)ONa; found 265.1567.

(4E,6E)-6-Methyl-1,7-diphenylhepta-4,6-dien-3-ol (9c)

Following General Procedure B, methyltriphenylphosphonium bromide (357 mg, 1.0 mmol) was first reacted with 3-phenylpropanal (134mg, 1.0 mmol) and then \( \alpha \)-methyl-trans-cinnamaldehyde (154 mg, 1.05 mmol) to give allylic alcohol 9c (110 mg, 39%) as a pale yellow oil; \( R_f \) 0.21 (20% Et\(_2\)O/petrol);

IR (neat) /cm\(^{-1}\): 3024w, 2919w, 2858w, 1601w, 1494m, 1453m, 1101w, 1030m, 1004m, 964s, 916m, 826w, 746s, 698s.

\(^1\)H (400 MHz) \( \delta = 7.41-7.15 \) (10 H, m, Ph), 6.55 (1 H, s, PhCH=H), 6.44 (1 H, d, \( J = 15.5 \), CCH=CH), 5.82 (1 H, dd, \( J = 15.5, 6.8 \), CH(OH)CH\(_2\)), 4.28 (1 H, app. q, \( J = 6.5 \), CH(OH)), 2.85-2.70 (2 H, m, CH(OH)CH\(_2\)), 2.02 (3 H, s, CH\(_3\)), 2.06-1.88 (2 H, m, PhCH\(_2\)), 1.69 (1 H, br. s, OH).

\(^{13}\)C (100 MHz) \( \delta = 141.8 \) (Ph), 137.6 (Ph), 136.0 (CCH=CH), 135.0 (C(CH\(_3\))), 131.7 (PhCH=H), 131.4 (CH(OH)CH), 129.1 (Ph), 128.4 (2 \times Ph), 128.4 (2 \times Ph), 128.1 (2 \times Ph), 126.6 (Ph), 125.8 (Ph), 72.4 (CH(OH)), 38.9 (PhCH\(_2\)), 31.7 (CH(OH)CH\(_2\)), 13.9 (CH\(_3\)).
LRMS (ESI+): 301.17 (100, M+Na); HRMS (ESI+): (M+Na) 301.1563 calculated for C_{20}H_{22}ONa; found 301.1561.

\textbf{(E)-6-Methyl-1-phenylhepta-4,6-dien-3-ol (9d)}

Following \textbf{General Procedure B}, methyltriphenylphosphonium bromide (714 mg, 2.0 mmol) was first reacted with 3-phenylpropanal (268 mg, 2.0 mmol) and then methacrylaldehyde (147 mg, 2.1 mmol) to give allylic alcohol 9d (254 mg, 63%) as a pale yellow oil; R_f 0.29 (20% Et_2O/petrol); IR (neat) /cm\(^{-1}\): 3354br. w, 2922w, 2360w, 1608w, 1495w, 1453w, 966w, 907m, 729m, 698m.

\(^1\)H (400 MHz) δ = 7.37-7.19 (5 H, m, Ph), 6.35 (1 H, d, J = 15.8, =CCH), 5.72 (1 H, dd, J = 15.8, 6.9, CH(OH)CH\(_2\)), 5.09-5.01 (2 H, m, C=CH\(_2\)), 4.23 (1 H, app. q, J = 6.6, CH(OH)), 2.85-2.67 (2 H, m, PhCH\(_2\)), 2.13 (1 H, br. s, OH), 2.03-1.84 (5 H, m, CH(OH)CH\(_2\) and CH\(_3\)).

\(^{13}\)C (125 MHz) δ = 141.8 (Ph), 141.3 (C=CH\(_2\)), 133.5 (=CCH), 132.1 (CH(OH)CH), 128.4 (2 \times Ph), 128.3 (Ph), 125.8 (Ph), 116.9 (Ph), 72.2 (CH(OH)), 38.8 (CH(OH)CH\(_2\)), 31.7 (PhCH\(_2\)), 18.6 (CH\(_3\)).

LRMS (ESI−): 112.99 (100), 203.11 (25, M+Na); HRMS (ESI+): (M+Na) 255.1250 calculated for C\(_{14}\)H\(_{18}\)ONa; found 255.1244.

\textbf{(E)-1-Phenylundec-1-en-3-ol (10a)}

Following \textbf{General Procedure B}, methyltriphenylphosphonium bromide (714 mg, 2 mmol) was reacted with nonanal (284 mg, 2 mmol) and benzaldehyde (223 mg, 2.1 mmol) to give allylic alcohol 10a\(^5\) (197 mg, 40%) as a pale yellow oil; R_f 0.41 (30% Et\(_2\)O/petrol);

IR (neat) /cm\(^{-1}\): 3351br. w, 2922w, 2360w, 1608w, 1495w, 1453w, 966w, 907m, 729m, 698m.

\(^1\)H (400 MHz) δ = 7.43-7.22 (5 H, m, Ph), 6.58 (1H, d, J = 15.8, CH(OH)CH=CH), 6.24 (1 H, dd, J = 15.8, 6.8, CH(OH)CH\(_2\)), 4.29 (1 H, app. q, J = 6.6, CH(OH)), 1.80 (1 H, br. s, OH), 1.71-1.56 (2 H, m, CH\(_2\)CH(OH)), 1.50-1.22 (12 H, m, 6 \times CH\(_2\)), 0.94-0.87 (3 H, t, J = 1.1, CH\(_3\)).

\(^{13}\)C (100 MHz) δ = 136.7 (Ph), 132.6 (CH(OH)CH=CH), 130.1 (CH(OH)CH\(_2\)), 128.5 (2 \times Ph), 127.5 (Ph), 126.4 (2 \times Ph), 73.1 (CH(OH)), 37.4 (CH(OH)CH\(_2\)), 31.8 (CH\(_2\)), 29.6 (CH\(_2\)), 29.5 (CH\(_2\)), 29.2 (CH\(_2\)), 25.4 (CH\(_2\)), 22.6 (CH\(_2\)), 14.1 (CH\(_3\)).

LRMS (ESI+): 269.19 (80, M+Na), 301.22 (55), 413.32 (55), 515.39 (100); HRMS (ESI+): (M+Na) 269.1876 calculated for C\(_{17}\)H\(_{26}\)ONa; found 269.1868.
(E)-1,5-Diphenylpent-1-en-3-ol (10b)

Following **General Procedure B**, methyltriphenylphosphonium bromide (714 mg, 2.0 mmol) was first reacted with 3-phenylpropanal (268 mg, 2.0 mmol) and then benzaldehyde (223 mg, 2.1 mmol) to give allylic alcohol **10b** (190 mg, 40%) as a pale yellow oil; R$_f$ 0.25 (30% Et$_2$O/petrol);

IR (neat) /cm$^{-1}$: 3346br. w, 3025w, 2924w, 1601w, 1494w, 1452w, 965m, 745s, 692s.

$^1$H (400 MHz) δ = 7.50-7.13 (10 H, m, 2 × Ph), 6.61 (1H, d, J = 15.9, PhCH), 6.28 (1 H, dd, J = 15.9, 6.8, CH(OH)CH), 4.33 (1 H, app. q, J = 6.5, CH(OH)), 2.91-2.68 (2 H, m, PhCH$_2$), 2.12-1.91 (2 H, m, CH(OH)CH$_2$), 1.86 (1 H, br. s, OH).

$^{13}$C (100 MHz) δ = 141.7 (Ph), 136.6 (Ph), 132.1 (CH(OH)CH), 130.5 (CH(OH)CH=CH), 128.6 (2 × Ph), 128.4 (2 × Ph), 128.4 (Ph), 127.7 (Ph), 126.4 (Ph), 125.8 (Ph), 72.3 (CH(OH)), 38.7 (PhCH$_2$), 31.6 (CH(OH)CH$_2$).

LRMS (ESI+): 261.14 (100, M+Na), 493.24 (80); HRMS (ESI+): (M+Na) 261.1250 calculated for C$_{17}$H$_{18}$ONa; found 261.1248.

(E)-1,3-Diphenylprop-2-en-1-ol (10c)

Following **General Procedure B**, methyltriphenylphosphonium bromide (714 mg, 2.0 mmol) was first reacted with benzaldehyde (212 mg, 2.0 mmol) and then benzaldehyde (223 mg, 2.1 mmol) to give allylic alcohol **10c** (274 mg, 65%) as a pale yellow oil; R$_f$ 0.26 (30% Et$_2$O/petrol);

IR (neat) /cm$^{-1}$: 3329br. w, 3027w, 1493w, 1449w, 1494w, 964m, 742s, 692s.

$^1$H (400 MHz) δ = 7.53-7.20 (10 H, m, 2 × Ph), 6.71 (1H, d, J = 15.9, PhCH), 6.41 (1 H, dd, J = 15.9, 6.6, CH(OH)CH), 5.40 (1 H, d, J = 6.3, CH(OH)), 2.24 (1 H, br. s, OH).

$^{13}$C (100 MHz) δ = 142.7 (Ph), 136.5 (Ph), 131.5 (CH(OH)CH), 130.5 (PhCH), 128.6 (2 × Ph), 128.5 (2 × Ph), 127.7 (Ph), 126.6 (Ph), 126.3 (2 × Ph), 75.1 (CH(OH)).

LRMS (ESI+): 233.09 (100, M+Na), 493.24 (80); HRMS (ESI+): (M+Na) 233.0937 calculated for C$_{13}$H$_{14}$ONa; found 233.0943.
Following **General Procedure B**, methyltriphenylphosphonium bromide (714 mg, 2.0 mmol) was first reacted with nonanal (284 mg, 2.0 mmol) and then 5-methylthiophene-2-carboxaldehyde (225 mg, 2.1 mmol) to give allylic alcohol **10d** (317 mg, 59%) as a pale yellow oil; R_f 0.36 (20% Et_2O/petrol);

IR (neat) /cm\(^{-1}\): 3340br. w, 2922s, 2853m, 2360s, 1645s, 1464w, 1040w, 952s, 789s, 721w.

\(^1\)H (400 MHz) \(\delta = 6.73\) (1 H, d, \(J = 3.3\), Ar), 6.60 (1 H, d, \(J = 15.7\), CH(OH)CH=CH), 6.60 (1 H, dq, \(J = 3.4\), 1.1, Ar), 5.93 (1 H, dd, \(J = 15.7\), 6.8, (CH(OH)CH)), 4.19 (1 H, app. q, \(J = 6.7\), CH(OH)), 2.51-2.42 (3 H, m, SCC\(\text{H}_3\)), 2.11 (1 H, br. s, OH), 1.69-1.50 (2 H, m, CH(OH)CH\(\text{H}_2\)), 1.50-1.18 (12 H, m, 6 \times \text{CH}_2), 0.97-0.83 (3 H, m, CH\(_3\)).

\(^{13}\)C (100 MHz) \(\delta = 139.8\) (Ar), 139.0 (Ar), 130.9 (CH(OH)CH), 125.9 (Ar), 125.4 (Ar), 123.8 (CH(OH)CH=CH), 72.9 (CH(OH)), 37.3 (CH(OH)CH\(_2\)), 31.8 (CH\(_3\)), 29.5 (CH\(_2\)), 29.2 (CH\(_2\)), 25.4 (CH\(_2\)), 22.6 (CH\(_2\)), 15.5 (SC(CH\(_3\))), 14.1 (CH\(_3\)).

LRMS (ESI+): 289.18 (25, M+Na); HRMS (ESI+): (M+Na) 289.1597 calculated for C\(_{16}\)H\(_{26}\)OSNa; found 289.1589.

**Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry**
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**(E)-1-(5-Methylthiophen-2-yl)-8-phenylpent-1-en-3-ol (10e)**

Following **General Procedure B**, methyltriphenylphosphonium bromide (714 mg, 2.0 mmol) was first reacted with 3-phenylpropanal (268 mg, 2.0 mmol) and then 5-methylthiophene-2-carboxaldehyde (225 mg, 2.1 mmol) to give allylic alcohol **10e** (309 mg, 60%) as a yellow oil; R_f 0.20 (20% Et\(_2\)O/petrol);

IR (neat) /cm\(^{-1}\): 3371br. w, 3025w, 2918w, 2858w, 1644w, 1602w, 1543w, 1495m, 1453m, 1156m, 1029s, 954s, 793s, 744s, 697s.

\(^1\)H (400 MHz) \(\delta = 7.36-7.19\) (5 H, m, Ph), 6.76 (1 H, d, \(J = 3.5\), CH thiophenyl), 6.64 (1 H, d, \(J = 15.7\), CH(OH)CH=CH), 6.64 (1 H, dq, \(J = 3.6\), 1.2, CH thiophenyl), 5.98 (1 H, dd, \(J = 15.7\), 6.8, CH(OH)CH), 4.25 (1 H, app. q, \(J = 6.3\), CH(OH)), 2.85-2.68 (2 H, m, PhCH\(_2\)), 2.48 (3 H, d, \(J = 0.8\), CH\(_3\)), 2.06-1.88 (2 H, m, CH(OH)CH\(_2\)), 1.85 (1 H, br. s, OH).

\(^{13}\)C (100 MHz) \(\delta = 141.7\) (Ph), 139.6 (thiophenyl), 139.2 (thiophenyl), 130.4 (CH(OH)CH), 128.4 (2 \(\times\) Ph), 128.3 (2 \(\times\) Ph), 126.1 (thiophenyl), 125.8 (Ph), 125.4 (thiophenyl), 124.1 (CH(OH)CH=CH), 72.0 (CH(OH)), 38.7 (CH(OH)CH\(_2\)), 31.6 (PhCH\(_2\)), 15.5 (CH\(_3\)).
LRMS (ESI+): 281.11 (100, M+Na); HRMS (ESI+): (M+Na) 281.0971 calculated for C_{16}H_{18}OSNa; found 281.0964.

**(E)-1-(Furan-2-yl)-undec-1-en-3-ol (10f)**

Following **General Procedure B**, methyltriphenylphosphonium bromide (714 mg, 2.0 mmol) was first reacted with nonanal (284 mg, 2.0 mmol) and then furfuraldehyde (202 mg, 2.1 mmol) to give allylic alcohol **10f** (235 mg, 49%) as a red-orange oil; R_f 0.37 (25% Et_2O/petrol);

IR (neat) /cm^{-1}: 3352br. w, 2924w, 2854w, 1465w, 1011w, 960w, 793w, 728w.

^1^H (400 MHz) δ = 7.35 (1 H, d, J = 1.5, Ar), 6.41 (1 H, d, J = 15.9, CH(OH)CH=CH), 6.37 (1 H, dd, J = 3.3, 1.8, Ar), 6.24 (1 H, d, J = 3.0, Ar), 6.18 (1 H, dd, J = 15.9, 6.6, CH(OH)CH), 4.24 (1 H, app. q, J = 6.4, CH(OH)), 1.76 (1 H, br. s, OH), 1.67-1.53 (2 H, m, CH(OH)CH_2), 1.48-1.18 (12 H, m, 6 × CH_2), 0.89 (3 H, t, J = 6.7, CH_3).

^1^3^C (100 MHz) δ = 152.4 (Ar), 141.9 (Ar), 131.0 (CH(OH)CH), 118.4 (CH(OH)CH=CH), 111.2 (Ar), 107.9 (Ar), 72.6 (CH(OH)), 37.4 (CH(OH)CH_2), 31.8 (CH_2), 29.6 (CH_2), 29.5 (CH_2), 29.3 (CH_2), 25.4 (CH_2), 22.6 (CH_2), 14.1 (CH_3).

LRMS (ESI−): 251.18 (45), 469.36 (40) 533.37 (100); (ESI+): 477.36 (100), 511.37 (40); HRMS (FI+): 236.1776 calculated for C_{15}H_{24}O_2; found 236.1774.

**(E)-1-(Furan-2-yl)-5-phenylpent-1-en-3-ol (10g)**

Following **General Procedure B**, methyltriphenylphosphonium bromide (714 mg, 2.0 mmol) was first reacted with 3-phenylpropanal (268 mg, 2.0 mmol) and then furfuraldehyde (202 mg, 2.1 mmol) to give allylic alcohol **10g** (258 mg, 56%) as a red-orange oil; R_f 0.37 (30% Et_2O/petrol);

IR (neat) /cm^{-1}: 3363br. w, 2923w, 1602w, 1493w, 1454w, 1254w, 1150w, 1011m, 960m, 926m, 883w, 798w, 732s, 697s.

^1^H (400 MHz) δ = 7.39-7.35 (1 H, app. d, CH furanyl), 7.35-7.17 (5 H, m, Ph), 6.44 (1 H, d, J = 15.9, CH(OH)CH=CH), 6.40 (1 H, dd, J = 3.2, 1.9, CH furanyl), 6.26 (1 H, d, J = 4.0, CH furanyl), 6.23 (1 H, dd, J = 15.9, 6.6, CH(OH)CH), 4.29 (1 H, app. q, J = 6.3, CH(OH)), 2.85-2.69 (2 H, m, PhCH_2), 2.04-1.88 (2 H, m, CH(OH)CH_2), 1.77 (1 H, br. s, OH).
1H (400 MHz) δ = 6.89 (1 H, d, J = 1.8, Ar), 6.85 (1 H, dd, J = 8.4, 1.8, Ar), 6.76 (1 H, d, J = 8.4, Ar), 6.44 (1 H, d, J = 15.9, CH(OH)CH=CH), 6.09 (1 H, dd, J = 15.9, 6.3, CH(OH)CH), 4.42 (1 H, app. quin d, J = 6.3, 0.8, CH(OH)), 3.84 (3 H, s, OCH3), 3.82 (3 H, s, OCH3), 2.32 (1 H, br. s, OH), 1.33 (3 H, d, J = 6.3, CH3).

13C (100 MHz) δ = 148.8 (OCH3), 148.6 (OCH3), 131.6 (CH(OH)CH), 129.6 (Ar), 128.9 (CH(OH)CH=CH), 119.5 (Ar), 110.9 (Ar), 108.6 (Ar), 68.7 (CH(OH)), 55.7 (Ar), 55.6 (Ar), 23.3 (CH3).

LRMS (ESI+): 231.10 (25, M+Na), 391.28 (30), 413.24 (91), 803.44 (96); HRMS (ESI+): (M+Na) 231.0992 calculated for C12H16O3Na; found 231.0997.

(E)-1-cyclohexyl-5-phenylpent-1-en-3-ol (11) and (E)-1-cyclohexyl-5-phenylpent-2-en-1-ol (12)

Following General Procedure B, methyltriphenylphosphonium bromide (714 mg, 2.0 mmol) was first reacted with 3-phenylpropanal (268 mg, 2.0 mmol) and then cyclohexanecarboxaldehyde (236 mg, 2.1 mmol) to give E-allylic alcohols 11 and 12 as a mixture of chromatographically inseparable isomers (206 mg, 42%, 11:12 90:10 by 1H NMR integration of CH(OH)) as a pale yellow oil; Rf 0.28 (20% Et2O/petrol);
IR (neat) /cm$^{-1}$: 3364br. w, 2922m, 2851m, 1495w, 1449m, 1030w, 969m, 907m, 730s, 697s.

LRMS (ESI+): 267.17 (100, M+Na), 268.20 (38), 511.38 (24, 2M+Na); HRMS (ESI+): (M+Na) 267.1725 calculated for C$_{17}$H$_{24}$ONa; found 267.1720.

Data for 11:
$^1$H (400 MHz) $\delta = 7.35$-$7.19$ (5 H, m, Ph), 5.64 (1 H, dd, $J = 15.7, 6.6$, CH(OH)CH=CH), 5.49 (1 H, ddd, $J = 15.7, 7.0, 1.1$, CH(OH)CH), 4.09 (1 H, app. q, $J = 6.7$, CH(OH)), 2.80-2.65 (2 H, m, PhCH$_2$), 2.10-1.58 (8 H, m, CH cyclohexyl, 2 × CH$_2$ cyclohexyl, CH(OH)CH=CH$_2$ and OH), 1.42-1.05 (6 H, m, 3 × CH$_2$ cyclohexyl).

$^{13}$C (100 MHz) $\delta = 142.0$ (Ph), 138.0 (CH(OH)CH=CH), 130.1 (CH(OH)CH), 128.3 (2 × Ph), 128.2 (2 × Ph), 125.6 (Ph), 72.4 (CH(OH)), 40.2 (CH=CHCH), 38.7 (CH(OH)CH$_2$), 32.8 (CH$_2$ cyclohexyl), 32.7 (CH$_2$ cyclohexyl), 31.7 (PhCH$_2$), 26.0 (CH$_2$ cyclohexyl), 25.9 (2 × CH$_2$ cyclohexyl).

Data for 12:
$^1$H (400 MHz) $\delta = 7.35$-$7.19$ (5 H, m, Ph), 5.66 (1 H, dt, $J = 15.5, 6.4$, CH(OH)CH=CH), 5.49 (1 H, ddt, $J = 15.5, 7.3, 1.2$, CH(OH)CH), 3.79 (1 H, app. t, $J = 6.9$, CH(OH)), 2.80-2.65 (2 H, m, PhCH$_2$), 2.42 (2 H, q, $J = 7.4$, PhCH$_2$CH$_2$), 2.10-1.58 (5 H, m, CH cyclohexyl, CH$_2$ cyclohexyl, OH), 1.42-1.05 (5 H, m, CH$_2$ cyclohexyl), 1.03-0.88 (2 H, m, CH$_2$ cyclohexyl).

$^{13}$C (100 MHz) $\delta = 141.5$ (Ph), 132.2 (CH(OH)CH=CH), 131.5 (CH(OH)CH=CH), 128.2 (2 × Ph), 128.1 (2 × Ph), 125.7 (Ph), 77.4 (CH(OH)), 43.5 (CH cyclohexyl), 35.5 (PhCH$_2$), 33.9 (PhCH$_2$CH$_2$), 28.6 (CH$_2$ cyclohexyl), 28.5 (CH$_2$ cyclohexyl), 26.4 (CH$_2$ cyclohexyl), 25.9 (CH$_2$ cyclohexyl).

3-(furan-2-yl)-1-phenylprop-2-en-1-ol (13) and (E)-1-(furan-2-yl)-3-phenylprop-2-en-1-ol (14)

Following General Procedure B, methyltriphenylphosphonium bromide (714 mg, 2.0 mmol) was first reacted with benzaldehyde (212 mg, 2.0 mmol) and then furfuraldehyde (202 mg, 2.1 mmol) to give allylic alcohols 13 and 14$^{10}$ as a mixture of chromatographically inseparable isomers (160 mg, 40%, 13$^{E}$:13$^{Z}$:14 82:9:9 by $^1$H NMR integration of CH(OH)) as a red-orange oil; $R_f$ 0.29 (20% Et$_2$O/petrol).

IR (neat) /cm$^{-1}$: 3366br. w, 3028w, 2923w, 2854w, 1491w, 1452w, 1150w, 1067w, 1011m, 960w, 926w, 883m, 735m, 698m.
LRMS (ESI+): 405.17 (100), 406.17 (30), 787.35 (40), (ESI–): 215.09 (70), 381.17 (100), 413.19 (35); HRMS (FI+): 200.0837 calculated for C_{13}H_{12}O_{2}; found 200.0829.

Data for 13E:
^{1}H (400 MHz) δ = 7.53-7.29 (6 H, m, Ph and CH furanyl), 6.52 (1 H, dd, J = 16.0, 1.3, CH(OH)CH=CH), 6.38 (1 H, dd, J = 3.4, 1.9, CH furanyl), 6.36 (1 H, dd, J = 16.0, 5.8, (CH(OH)CH), 6.28 (1 H, d, J = 3.3, CH furanyl), 5.36 (1 H, d, J = 6.3, CH(OH)), 2.14 (1 H, br. s, OH).

^{13}C (100 MHz) δ = 152.2 (furanyl), 142.5 (Ph), 142.0 (furanyl), 130.1 (CH(OH)CH), 128.6 (2 × Ph), 127.8 (2 × Ph), 126.4 (Ph), 118.7 (CH(OH)CH=CH), 111.3 (furanyl), 108.4 (furanyl), 74.7 (CH(OH)).

Discernible data for 13Z:

^{1}H (400 MHz) δ = 7.53-7.29 (6 H, m, Ph and CH furanyl), 6.31 (1 H, d, J = 11.9, (CH(OH)CH=CH), 6.17 (1 H, d, J = 9.1, CH(OH)), 5.57 (1 H, dd, J = 11.9, 9.0, CH(OH)CH).

^{13}C (100 MHz) δ = 152.2 (furanyl), 142.6 (Ph), 142.1 (furanyl), 131.2 (CH(OH)CH), 128.5 (Ph), 126.4 (Ph), 128.5 (CH(OH)CH=CH), 75.1 (CH(OH)).

Discernible data for 14:

^{1}H (400 MHz) δ = 7.53-7.29 (6 H, m, Ph, CH furanyl), 6.71 (1 H, d, J = 16.0, PhCH), 6.41 (1 H, dd, J = 16.0, 6.6, PhCH=CH), 5.40 (1 H, d, J = 6.6, CH(OH)).

^{13}C (100 MHz) δ = 142.5 (Ph), 142.5 (Ph), 131.2 (PhCH=CH), 70.5 (CH(OH)).
4. $^1$H and $^{13}$C NMR spectra of 4a-b, 7, 8, 9a-d, 10a-h, and 11-14.

*(E)-3-Methyldodec-3-en-2-ol (4a)*
(E)-3-Methyldodec-2-en-4-ol (4b)
Dodec-3-en-2-ol (7)
Dodec-2-en-4-ol (8)
1-Phenylocta-4,6-dien-3-ol (9a)
(E)-1-(Cyclohex-1-en-1-yl)-5-phenylpent-1-en-3-ol (9b)
(4E,6E)-6-Methyl-1,7-diphenylhepta-4,6-dien-3-ol (9c)
(E)-6-Methyl-1-phenylhepta-4,6-dien-3-ol (9d)
(E)-1-Phenylundec-1-en-3-ol (10a)
(E)-1,5-Diphenylpent-1-en-3-ol (10b)
(E)-1,3-Diphenylprop-2-en-1-ol (10c)
(E)-1-(5-Methylthiophen-2-yl)-undec-1-en-3-ol (10d)
(E)-1-(5-Methylthiophen-2-yl)-5-phenylpent-1-en-3-ol (10e)
(E)-1-(Furan-2-yl)-undec-1-en-3-ol (10f)
(E)-1-(Furan-2-yl)-5-phenylpent-1-en-3-ol (10g)
(E)-4-(3,4-Dimethoxyphenyl)-but-3-en-2-ol (10h)
(E)-1-cyclohexyl-5-phenylpent-1-en-3-ol (11) and (E)-1-cyclohexyl-5-phenylpent-2-en-1-ol (12)
3-(furan-2-yl)-1-phenylprop-2-en-1-ol (13) and (E)-1-(furan-2-yl)-3-phenylprop-2-en-1-ol (14)
5. References