

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 ($\lambda = 254$ 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 (40–63 μ m) 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 Brücker 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.

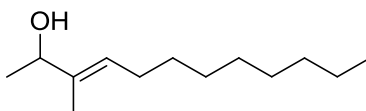
Mass spectra were obtained by field ionisation (FI; Micromass GCT) or by electrospray ionisation (ESI; LCT Premier Reflectron TOF and Brücker MicroTOF) using tetraoctylammonium bromide or sodium dodecyl sulfate as lock mass; values are quoted as ratios of mass:charge (*m/z*) in Daltons, and relative intensities of assignable peaks observed are quoted as a percentage value.

2. Synthesis of trisubstituted *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 $-78\text{ }^{\circ}\text{C}$. PhLi (2.0 M in Bu₂O, 1.0 equiv) was then added dropwise at $-78\text{ }^{\circ}\text{C}$. 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 $-78\text{ }^{\circ}\text{C}$ 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 Bu₂O, 1.05 equiv) was added dropwise resulting in a cherry-red solution. This solution was stirred for 30 min at $-78\text{ }^{\circ}\text{C}$, after which time a solution of the second aldehyde (1.05 equiv) in THF (0.5 mL/mmol aldehyde) was added dropwise at $-78\text{ }^{\circ}\text{C}$. The reaction mixture was stirred at $-78\text{ }^{\circ}\text{C}$ 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 Et₂O (3 × 15 mL), dried (MgSO₄) and evaporated under reduced pressure. Purification of the crude product by flash chromatography in the solvent system indicated gave the trisubstituted *E*-allylic alcohol.

(*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% Et₂O/petrol);

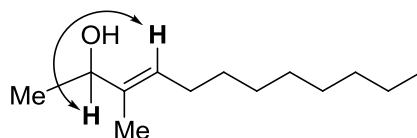
IR (film) / cm^{-1} : 3340w, 2922m, 2854m, 2360w, 1717w, 1457w, 1368w, 1077m, 887w.

¹H (400 MHz) δ = 5.40 (1 H, t, J = 7.1, C=CH), 4.20 (1 H, q, J = 6.3, CH(OH)), 2.00 (2 H, q, J = 7.1, =CHCH₂), 1.62 (3 H, s, =CCH₃), 1.56 (1 H, br. s, OH), 1.38-1.21 (15 H, m, 6 × CH₂ and CH₃CH(OH)), 0.88 (3 H, t, J = 6.8, CH₂CH₃).

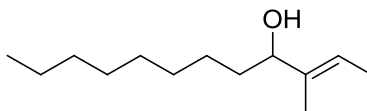
¹³C (100 MHz) δ = 138.2 (=CCH₃), 125.4 (C=CH), 73.5 (CH(OH)), 31.9 (CH₂), 29.5 (2 × CH₂), 29.3 (CH₂), 29.2 (CH₂), 27.5 (=CHCH₂), 22.65 (CH₂), 21.6 (=CCH₃), 14.1 (CH₂CH₃), 11.4 (CH₃CH(OH)).

LRMS (ESI+): 221.3 (15, M+Na), 253.3 (45), 413.3 (100); HRMS (ESI+): (M+Na) 221.1876 calculated for C₁₃H₂₆ONa; found 221.1879.

NOEs observed for **4a**:



(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 μ L, 1.05 mmol) to give allylic alcohol **4b**¹ (126 mg, 64%) as a pale yellow oil; R_f 0.35 (20% Et₂O/petrol);

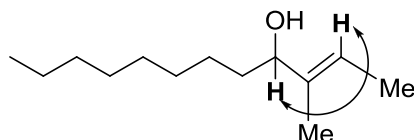
IR (neat) /cm⁻¹: 3343w, 2923m, 2855m, 1457w, 1378w, 1000w, 828w.

¹H (400 MHz) δ = 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 CH₃), 1.55-1.46 (2 H, m, CH(OH)CH₂), 1.35-1.14 (12 H, m, 6 \times CH₂), 0.87 (3 H, t, J = 6.8, CH₂CH₃).

¹³C (100 MHz) δ = 138.0 (=CCH₃), 120.7 (=CH), 78.1 (CH(OH)), 34.8 (CH₂CH(OH)), 31.8 (CH₂), 29.6 (CH₂), 29.5 (CH₂), 29.3 (CH₂), 25.8 (CH₂), 22.6 (CH₂), 14.1 (CH₂CH₃), 13.0 (CH₃), 10.7 (CH₃).

LRMS (ESI⁺): 142.3 (20), 235.3 (45), 413.3 (100); HRMS (FI⁺): 198.1984 calculated for C₁₃H₂₆O; found 198.1991.

NOEs observed for **4b**:

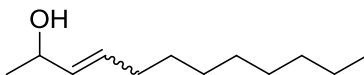


3. Synthesis of disubstituted *E*-allylic alcohols 7 - 14

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 $-78\text{ }^{\circ}\text{C}$. PhLi (2.0 M in Bu₂O, 1.0 equiv) was then added dropwise at $-78\text{ }^{\circ}\text{C}$. 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 $-78\text{ }^{\circ}\text{C}$ 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 Bu₂O, 1.05 equiv) was added dropwise resulting in a cherry-red solution. This solution was stirred for 30 min at $-78\text{ }^{\circ}\text{C}$, after which time a solution of the second aldehyde (1.05 equiv) in THF (0.5 mL/mmol aldehyde) was added dropwise at $-78\text{ }^{\circ}\text{C}$. The reaction mixture was stirred at $-78\text{ }^{\circ}\text{C}$ 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 Et₂O (3 × 15 mL), dried (MgSO₄) 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 μL , 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 ¹H NMR integration of CH(OH)) as a pale yellow oil; R_f 0.32 (25% Et₂O/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 C₁₂H₂₄ONa; found 207.1719.

Data for **7E**:

¹H (400 MHz) δ = 5.63 (1 H, dt, J = 15.4, 6.8, =CHCH₂), 5.50 (1 H, dd, J = 15.4, 6.8, CH(OH)CH), 4.25 (1 H, app. quin, J = 6.4, CH(OH)), 2.01, (2 H, quin, J = 6.9, =CHCH₂), 1.60 (1 H, br. s, OH), 1.40-1.21 (15 H, m, 6 × CH₂ and CH(OH)CH₃), 0.88 (3 H, t, J = 6.7, CH₂CH₃).

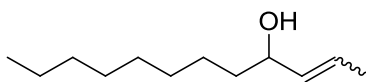
^{13}C (100 MHz) δ = 134.0 (CH(OH)CH), 131.2 (=CHCH₂), 69.0 (CH(OH)), 32.1 (CH₂), 31.9 (CH₂), 29.4 (CH₂), 29.2 (CH₂), 29.2 (CH₂), 23.4 (CH₃CH(OH)), 22.6 (CH₂), 14.1 (CH₃).

Discernible data for **7Z**:

^1H (400 MHz, CDCl₃) δ = 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, =CHCH₂), 1.60 (1 H, br. s, OH), 1.40-1.21 (15 H, m, 6 × CH₂ and CH(OH)CH₃), 0.88 (3 H, t, J = 6.7, CH₂CH₃).

^{13}C (100 MHz, CDCl₃) δ = 133.7 (CH(OH)CH), 131.4 (=CHCH₂), 63.9 (CH(OH)), 31.9 (CH₂), 29.7(CH₂), 29.4 (CH₂), 29.2 (CH₂), 29.2 (CH₂), 27.5 (CH₂), 23.6 (CH₃CH(OH)), 22.6 (CH₂), 14.1 (CH₃).

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 μL , 1.05 mmol) to give allylic alcohols **8** and **7** as a mixture of chromatographically inseparable isomers (106 mg, 58%, **8E**⁴:**8Z**:**7E**²:**7Z**³ 46:13:4:37 by ^1H NMR integration of CH(OH)) as a pale yellow oil; R_f 0.32 (25% Et₂O/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 C₁₂H₂₄ONa; found 207.1718.

Data for **8E**:

^1H (400 MHz) δ = 5.64 (1 H, dq, J = 15.5, 6.0, =CHCH₃), 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, =CHCH₃, OH), 1.60-1.40 (2 H, m, CH(OH)CH₂), 1.39-1.17 (12 H, m, 6 × CH₂), 0.87 (3 H, t, J = 6.7, CH₃).

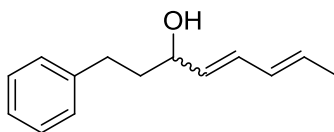
^{13}C (100 MHz) δ = 134.4 (CH(OH)CH), 126.6 (=CHCH₃), 73.1 (CH(OH)), 37.2 (CH₂CH(OH)), 31.9 (CH₂), 29.6 (CH₂), 29.4 (CH₂), 25.5 (CH₂), 22.6 (CH₂), 17.6 (=CHCH₃), 14.1 (CH₃).

Discernible data for **8Z**:

^1H (400 MHz, CDCl₃) δ = 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, CHCH₃, OH), 1.59-1.40 (2 H, m, CH(OH)CH₂), 1.39-1.17 (12 H, m, 6 × CH₂), 0.87 (3 H, t, J = 6.7, (CH₃)).

^{13}C (100 MHz, CDCl_3) δ = 133.6 (CH(OH)CH), 126.1 (=CHCH₃), 67.3 (CH(OH)), 37.4 (CH₂CH(OH)), 29.6 (CH₂), 27.5 (CH₂), 25.3 (CH₂), 17.6 (CH₃), 13.2 (=CHCH₃).

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 ^1H NMR integration of CH(OH)) as a pale yellow oil; R_f 0.23 (20% Et₂O/petrol);

IR (neat) /cm⁻¹: 3366br. w, 3024w, 2915w, 1495w, 1453w, 986s, 745m, 697s.

LRMS (ESI⁺): 225.14 (55, M+Na), 301.09 (100), 579.19 (67); HRMS (ESI⁺): (M+Na) 225.1250 calculated for C₁₄H₁₈ONa; found 225.1244.

Data for **9aE**:

^1H (400 MHz) δ = 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=CHCH₃), 5.74 (1 H, dq, J = 15.0, 6.8, =CHCH₃), 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, PhCH₂), 2.02-1.82 (2 H, m, CH(OH)CH₂), 1.79 (3 H, d, J = 6.6, CH₃), 1.77 (1 H, br. s, OH).

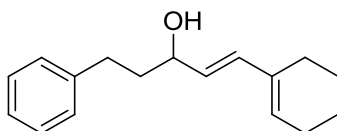
^{13}C (100 MHz) δ = 141.9 (Ph), 133.0 (CH(OH)CH), 131.1 (CH(OH)CH=CH), 130.7 (CH=CHCH₃), 130.0 (=CHCH₃), 128.4 (2 × Ph), 128.3 (2 × Ph), 125.7 (Ph), 72.0 (CH(OH)), 38.7 (CH(OH)CH₂), 31.7 (PhCH₂), 18.1 (CH₃)

Discernible data for **9aZ**:

^1H (400 MHz) δ = 7.34-7.17 (5 H, m, Ph), 6.33-6.23 (1 H, m, CH=CHCH₃), 6.14-6.02 (1 H, m, CH(OH)CH=CH), 5.78 (1 H, dq, J = 15.0, 6.8, =CHCH₃), 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, PhCH₂), 2.02-1.82 (2 H, m, CH(OH)CH₂), 1.79 (3 H, d, J = 6.6, CH₃), 1.77 (1 H, br. s, OH).

^{13}C (100 MHz) δ = 141.8 (Ph), 131.8 (=CHCH₃), 130.8 (CH(OH)CH=CH), 130.5 (CH(OH)CH), 128.4 (Ph), 128.3 (Ph), 126.5 (CH=CHCH₃), 125.9 (Ph), 67.3 (CH(OH)), 38.9 (CH(OH)CH₂), 31.6 (PhCH₂), 18.2 (CH₃).

(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% Et₂O/petrol);

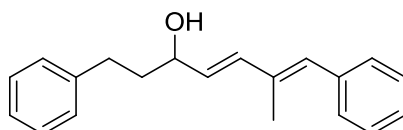
IR (neat) /cm⁻¹: 3334br. w, 2926w, 1649w, 1495w, 1453w, 1027m, 964m, 744m, 698s.

¹H (400 MHz) δ = 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=CHCH₂), 5.60 (1 H, dd, J = 15.7, 7.2, CH(OH)CH), 4.18 (1 H, app. q, J = 6.7, CH(OH)), 2.82-2.66 (2 H, m, PhCH₂), 2.21-2.11 (4 H, m, 2 × CH₂ cyclohexenyl), 2.03-1.81 (3 H, m, CH(OH)CH₂ and OH), 1.76-1.67 (2 H, m, CH₂ cyclohexenyl), 1.67-1.59 (2 H, m, CH₂ cyclohexenyl).

¹³C (100 MHz) δ = 141.9 (Ph), 134.9 (C=CH), 134.4 (CH(OH)CH=CH), 130.0 (C=CHCH₂), 128.4 (2 × Ph), 128.2 (2 × Ph), 128.0 (CH(OH)CH), 125.6 (Ph), 72.5 (CH(OH)), 38.8 (CH(OH)CH₂), 31.7 (PhCH₂), 25.8 (=CCH₂), 24.4 (CH₂), 22.4 (CH₂), 22.3 (CH₂).

LRMS (ESI+): 265.15 (100, M+Na), 266.18 (70), 389.24 (48), 507.35 (63, 2M+Na); HRMS (ESI+): (M+Na) 265.1568 calculated for C₁₇H₂₂ONa; 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 α -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% Et₂O/petrol);

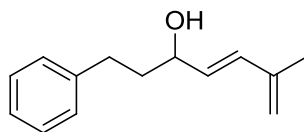
IR (neat) /cm⁻¹: 3024w, 2919w, 2858w, 1601w, 1494m, 1453m, 1101w, 1030m, 1004m, 964s, 916m, 826w, 746s, 696s.

¹H (400 MHz) δ = 7.41-7.15 (10 H, m, Ph), 6.55 (1 H, s, PhCH=C), 6.44 (1 H, d, J = 15.5, CCH=CH), 5.82 (1 H, dd, J = 15.5, 6.8, CH(OH)CH), 4.28 (1 H, app. q, J = 6.5, CH(OH)), 2.85-2.70 (2 H, m, CH(OH)CH₂), 2.02 (3 H, s, CH₃), 2.06-1.88 (2 H, m, PhCH₂), 1.69 (1 H, br. s, OH).

¹³C (100 MHz) δ = 141.8 (Ph), 137.6 (Ph), 136.0 (CCH=CH), 135.0 (C(CH₃)), 131.7 (PhCH=C), 131.4 (CH(OH)CH), 129.1 (Ph), 128.4 (2 × Ph), 128.4 (2 × Ph), 128.1 (2 × Ph), 126.6 (Ph), 125.8 (Ph), 72.4 (CH(OH)), 38.9 (PhCH₂), 31.7 (CH(OH)CH₂), 13.9 (CH₃).

LRMS (ESI⁺): 301.17 (100, M+Na); HRMS (ESI⁺): (M+Na) 301.1563 calculated for C₂₀H₂₂ONa; found 301.1561.

(E)-6-Methyl-1-phenylhepta-4,6-dien-3-ol (9d)



Following **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% Et₂O/petrol);

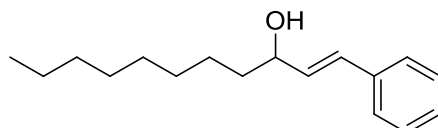
IR (neat) /cm⁻¹: 3354br. w, 2922w, 2360w, 1608w, 1495w, 1453w, 966w, 907m, 729m, 698m.

¹H (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), 5.09-5.01 (2 H, m, C=CH₂), 4.23 (1 H, app. q, *J* = 6.6, CH(OH)), 2.85-2.67 (2 H, m, PhCH₂), 2.13 (1 H, br. s, OH), 2.03-1.84 (5 H, m, CH(OH)CH₂ and CH₃).

¹³C (125 MHz) δ = 141.8 (Ph), 141.3 (C=CH₂), 133.5 (=CCH), 132.1 (CH(OH)CH), 128.4 (2 × Ph), 128.3 (Ph), 125.8 (Ph), 116.9 (Ph), 72.2 (CH(OH)), 38.8 (CH(OH)CH₂), 31.7 (PhCH₂), 18.6 (CH₃).

LRMS (ESI⁻): 112.99 (100), 203.11 (25, M+Na); HRMS (ESI⁺): (M+Na) 255.1250 calculated for C₁₄H₁₈ONa; found 255.1244.

(E)-1-Phenylundec-1-en-3-ol (10a)



Following **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% Et₂O/petrol);

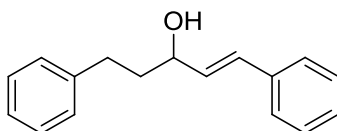
IR (neat) /cm⁻¹: 3351br. w, 2924m, 2853m, 1494w, 1450w, 964m, 746s, 691s.

¹H (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), 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, CH₂CH(OH)), 1.50-1.22 (12 H, m, 6 × CH₂), 0.94-0.87 (3 H, t, *J* = 1.1, CH₃).

¹³C (100 MHz) δ = 136.7 (Ph), 132.6 (CH(OH)CH=CH), 130.1 (CH(OH)CH), 128.5 (2 × Ph), 127.5 (Ph), 126.4 (2 × Ph), 73.1 (CH(OH)), 37.4 (CH(OH)CH₂), 31.8 (CH₂), 29.6 (CH₂), 29.5 (CH₂), 29.2 (CH₂), 25.4 (CH₂), 22.6 (CH₂), 14.1 (CH₃).

LRMS (ESI⁺): 269.19 (80, M+Na), 301.22 (55), 413.32 (55), 515.39 (100); HRMS (ESI⁺): (M+Na) 269.1876 calculated for C₁₇H₂₆ONa; 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**⁶ (190 mg, 40%) as a pale yellow oil; R_f 0.25 (30% Et₂O/petrol);

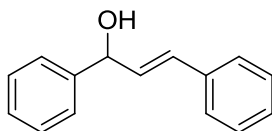
IR (neat) /cm⁻¹: 3346br. w, 3025w, 2924w, 1601w, 1494w, 1452w, 965m, 745s, 692s.

¹H (400 MHz) δ = 7.50-7.13 (10 H, m, 2 \times 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, PhCH₂), 2.12-1.91 (2 H, m, CH(OH)CH₂), 1.86 (1 H, br. s, OH).

¹³C (100 MHz) δ = 141.7 (Ph), 136.6 (Ph), 132.1 (CH(OH)CH), 130.5 (CH(OH)CH=CH), 128.6 (2 \times Ph), 128.4 (2 \times Ph), 128.4 (Ph), 127.7 (Ph), 126.4 (Ph), 125.8 (Ph), 72.3 (CH(OH)), 38.7 (PhCH₂), 31.6 (CH(OH)CH₂).

LRMS (ESI⁺): 261.14 (100, M+Na), 493.24 (80); HRMS (ESI⁺): (M+Na) 261.1250 calculated for C₁₇H₁₈ONa; 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**⁶ (274 mg, 65%) as a pale yellow oil; R_f 0.26 (30% Et₂O/petrol);

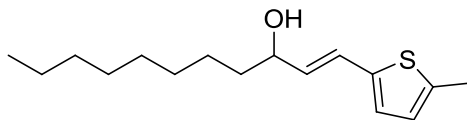
IR (neat) /cm⁻¹: 3329br. w, 3027w, 1493w, 1449w, 1494w, 964m, 742s, 692s.

¹H (400 MHz) δ = 7.53-7.20 (10 H, m, 2 \times 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).

¹³C (100 MHz) δ = 142.7 (Ph), 136.5 (Ph), 131.5 (CH(OH)CH), 130.5 (PhCH), 128.6 (2 \times Ph), 128.5 (2 \times Ph), 127.7 (Ph), 126.6 (Ph), 126.3 (2 \times Ph), 75.1 (CH(OH)).

LRMS (ESI⁺): 233.09 (100, M+Na), 265.13 (75), 443.23 (55); HRMS (ESI⁺): (M+Na) 233.0937 calculated for C₁₅H₁₄ONa; found 233.0943.

(E)-1-(5-Methylthiophen-2-yl)-undec-1-en-3-ol (10d)



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% Et₂O/petrol);

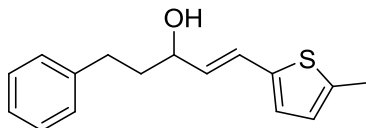
IR (neat) /cm⁻¹: 3340br. w, 2922s, 2853m, 2360s, 1645s, 1464w, 1040w, 952s, 789s, 721w.

¹H (400 MHz) δ = 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, SCCH₃), 2.11 (1 H, br. s, OH), 1.69-1.50 (2 H, m, CH(OH)CH₂), 1.50-1.18 (12 H, m, 6 × CH₂), 0.97-0.83 (3 H, m, CH₃).

¹³C (100 MHz) δ = 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)CH₂), 31.8 (CH₂), 29.5 (CH₂), 29.2 (CH₂), 25.4 (CH₂), 22.6 (CH₂), 15.5 (SC(CH₃)), 14.1 (CH₃).

LRMS (ESI⁺): 289.18 (25, M+Na); HRMS (ESI⁺): (M+Na) 289.1597 calculated for C₁₆H₂₆OSNa; found 289.1589.

(E)-1-(5-Methylthiophen-2-yl)-5-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% Et₂O/petrol);

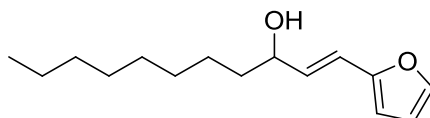
IR (neat) /cm⁻¹: 3371br. w, 3025w, 2918w, 2858w, 1644w, 1602w, 1543w, 1495m, 1453m, 1156m, 1029s, 954s, 793s, 744s, 697s.

¹H (400 MHz) δ = 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, PhCH₂), 2.48 (3 H, d, J = 0.8, CH₃), 2.06-1.88 (2 H, m, CH(OH)CH₂), 1.85 (1 H, br. s, OH).

¹³C (100 MHz) δ = 141.7 (Ph), 139.6 (thiophenyl), 139.2 (thiophenyl), 130.4 (CH(OH)CH), 128.4 (2 × Ph), 128.3 (2 × Ph), 126.1 (thiophenyl), 125.8 (Ph), 125.4 (thiophenyl), 124.1 (CH(OH)CH=CH), 72.0 (CH(OH)), 38.7 (CH(OH)CH₂), 31.6 (PhCH₂), 15.5 (CH₃).

LRMS (ESI⁺): 281.11 (100, M+Na); HRMS (ESI⁺): (M+Na) 281.0971 calculated for C₁₆H₁₈OSNa; 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% Et₂O/petrol);

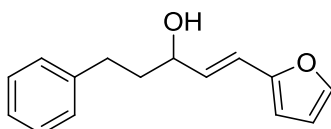
IR (neat) /cm⁻¹: 3352br. w, 2924w, 2854w, 1465w, 1011w, 960w, 793w, 728w.

¹H (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)CH₂), 1.48-1.18 (12 H, m, 6 × CH₂), 0.89 (3 H, t, *J* = 6.7, CH₃).

¹³C (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)CH₂), 31.8 (CH₂), 29.6 (CH₂), 29.5 (CH₂), 29.3 (CH₂), 25.4 (CH₂), 22.6 (CH₂), 14.1 (CH₃).

LRMS (ESI⁻): 251.18 (45), 469.36 (40) 533.37 (100); (ESI⁺): 477.36 (100), 511.37 (40); HRMS (FI⁺): 236.1776 calculated for C₁₅H₂₄O₂; 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* 0.32 (30% Et₂O/petrol);

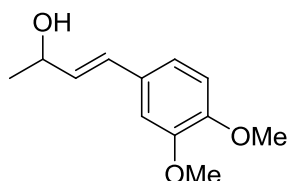
IR (neat) /cm⁻¹: 3363br. w, 2923w, 1602w, 1493w, 1454w, 1254w, 1150w, 1011m, 960m, 926m, 883w, 798w, 732s, 697s.

¹H (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, PhCH₂), 2.04-1.88 (2 H, m, CH(OH)CH₂), 1.77 (1 H, br. s, OH).

^{13}C (100 MHz) δ = 152.3 (furanlyl), 141.9 (furanlyl), 141.7 (Ph), 130.8 (CH(OH)CH), 128.4 (2 \times Ph), 128.3 (2 \times Ph), 125.8 (Ph), 118.7 (CH(OH)CH=CH), 111.3 (furanlyl), 108.1 (furanlyl), 71.8 (CH(OH)), 38.7 (CH(OH)CH₂), 31.6 (PhCH₂).

LRMS (ESI+): 251.12 (20, M+Na); HRMS (ESI+): (M+Na) 251.1043 calculated for C₁₅H₁₆O₂Na found 251.1038.

(E)-4-(3,4-Dimethoxyphenyl)-but-3-en-2-ol (10h)



Following **General Procedure B**, methyltriphenylphosphonium bromide (714 mg, 2.0 mmol) was first reacted with acetaldehyde (112 μL , 2.0 mmol) and then 3,4-dimethoxybenzaldehyde (349 mg, 2.1 mmol) to give allylic alcohol **10h**⁷ (271 mg, 65%) as a pale yellow oil; R_f 0.16 (30% Et₂O/petrol);

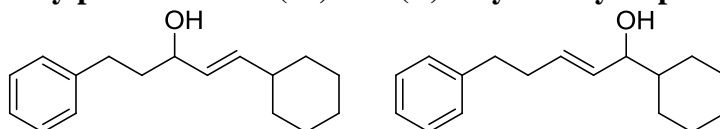
IR (neat) /cm⁻¹: 3372br. w, 2967w, 2836w, 1513m, 1463w, 1262m, 1136m, 1024m, 965w, 911w, 800w, 728m.

^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).

^{13}C (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**⁸ 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; R_f 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 C₁₇H₂₄ONa; found 267.1720.

Data for **11**:

¹H (400 MHz) δ = 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, (PhCH₂)), 2.10-1.58 (8 H, m, CH cyclohexyl, 2 × CH₂ cyclohexyl, CH(OH)CH₂ and OH), 1.42-1.05 (6 H, m, 3 × CH₂ cyclohexyl).

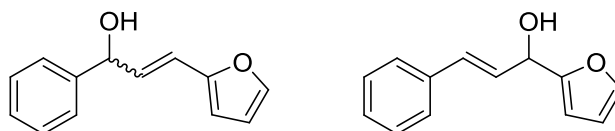
¹³C (100 MHz) δ = 142.0 (Ph), 138.0 (CH(OH)CH=CH), 130.1 (CH(OH)CH), 128.3 (2 × Ph), 128.2 (2 × Ph), 125.6 (Ph), 72.4 (CH(OH)), 40.2 (CH=CHCH), 38.7 (CH(OH)CH₂), 32.8 (CH₂ cyclohexyl), 32.7 (CH₂ cyclohexyl), 31.7 (PhCH₂), 26.0 (CH₂ cyclohexyl), 25.9 (2 × CH₂ cyclohexyl).

Data for **12**:

¹H (400 MHz) δ = 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, PhCH₂), 2.42 (2 H, q, *J* = 7.4, PhCH₂CH₂), 2.10-1.58 (5 H, m, CH cyclohexyl, CH₂ cyclohexyl, OH), 1.42-1.05 (5 H, m, CH₂ cyclohexyl), 1.03-0.88 (2 H, m, CH₂ cyclohexyl).

¹³C (100 MHz) δ = 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 (PhCH₂), 33.9 (PhCH₂CH₂), 28.6 (CH₂ cyclohexyl), 28.5 (CH₂ cyclohexyl), 26.4 (CH₂ cyclohexyl), 25.9 (CH₂ 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%, **13E**⁹:**13Z**:**14** 82:9:9 by ¹H NMR integration of CH(OH)) as a red-orange oil; R_f 0.29 (20% Et₂O/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 C₁₃H₁₂O₂; found 200.0829.

Data for **13E**:

¹H (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).

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

Discernible data for **13Z**:

¹H (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).

¹³C (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)).

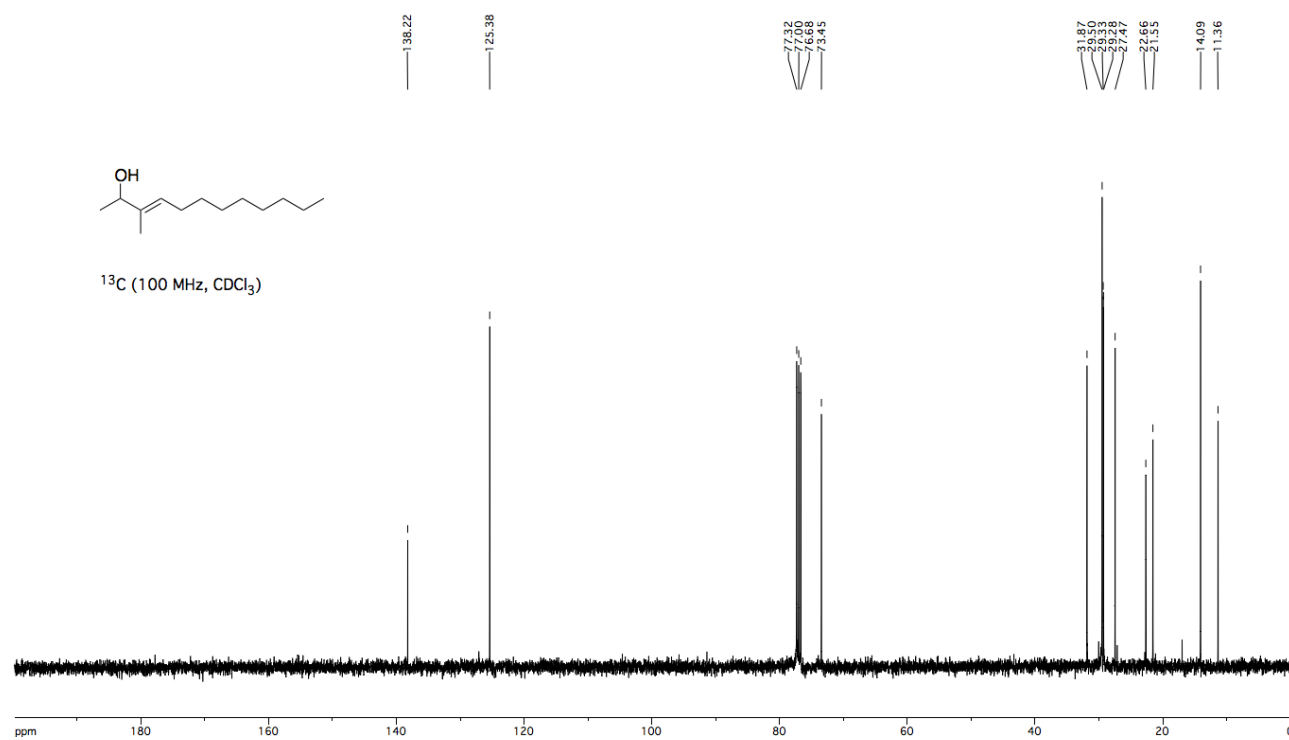
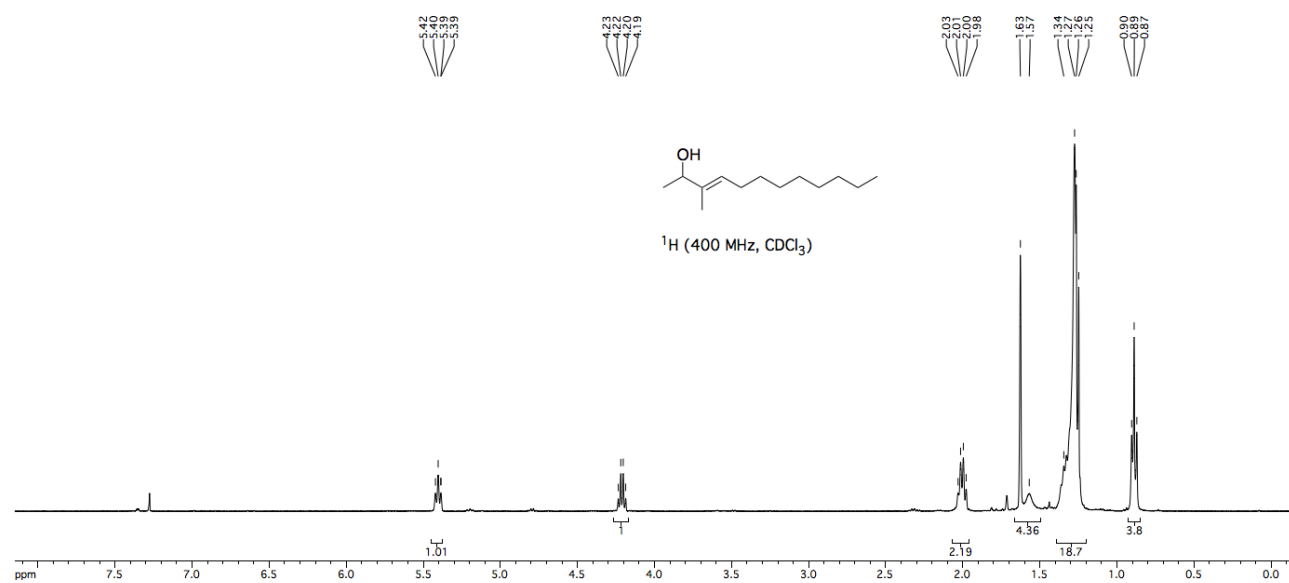
Discernible data for **14**:

¹H (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)).

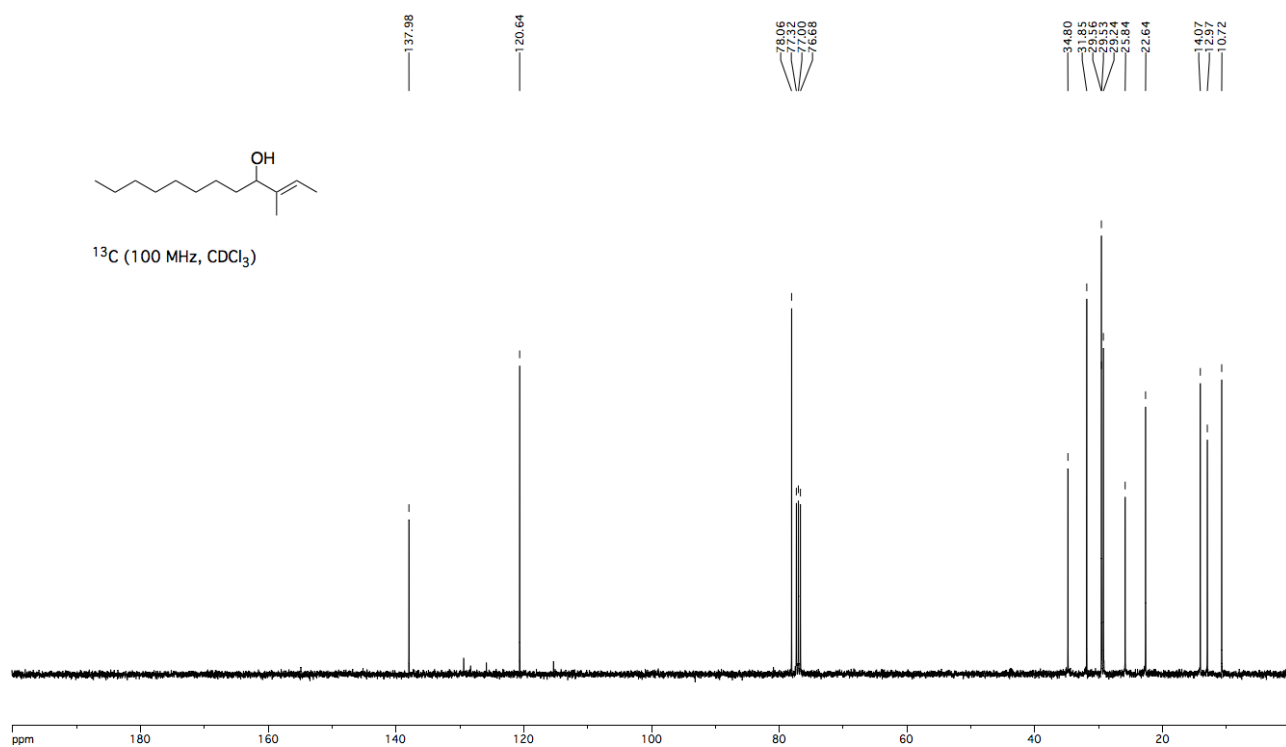
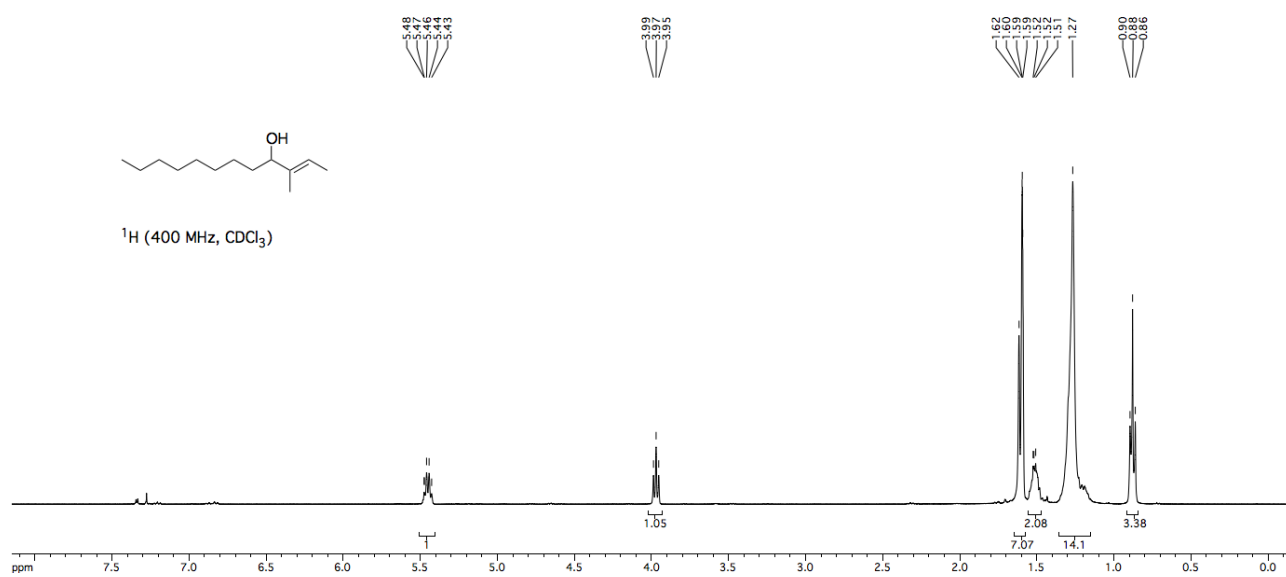
¹³C (100 MHz) δ = 142.5 (Ph), 142.5 (Ph), 131.2 (PhCH=CH), 70.5 (CH(OH)).

4. ^1H and ^{13}C NMR spectra of 4a-b, 7, 8, 9a-d, 10a-h, and 11-14.

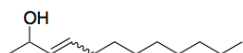
(*E*)-3-Methyldodec-3-en-2-ol (4a)



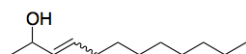
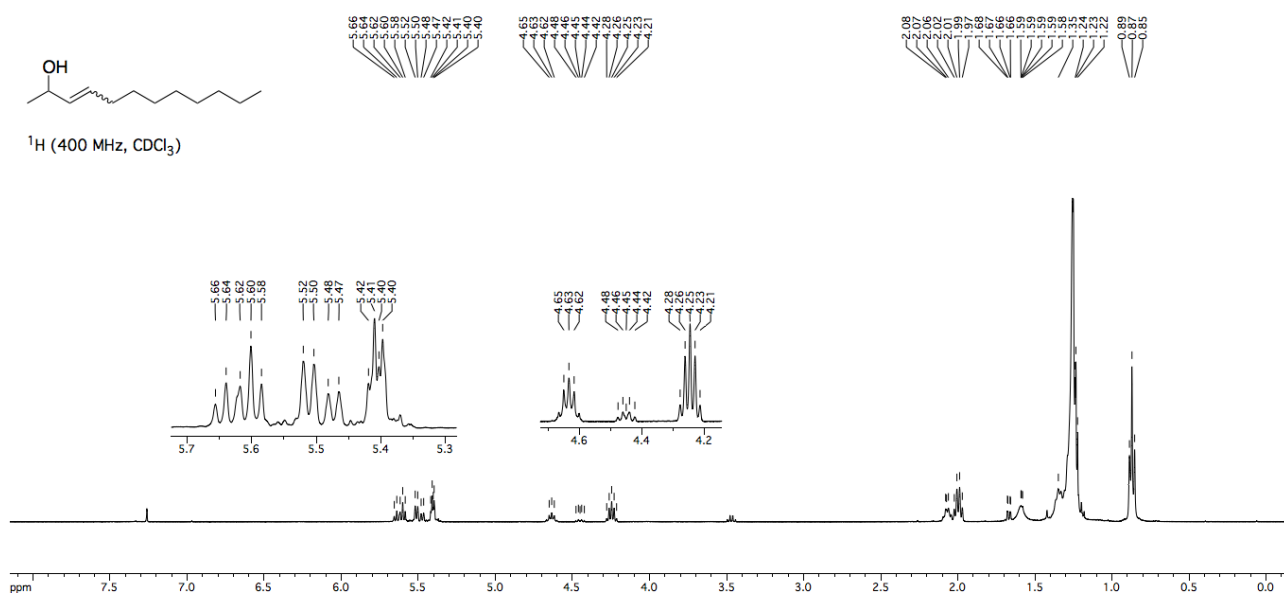
(E)-3-Methyldodec-2-en-4-ol (4b)



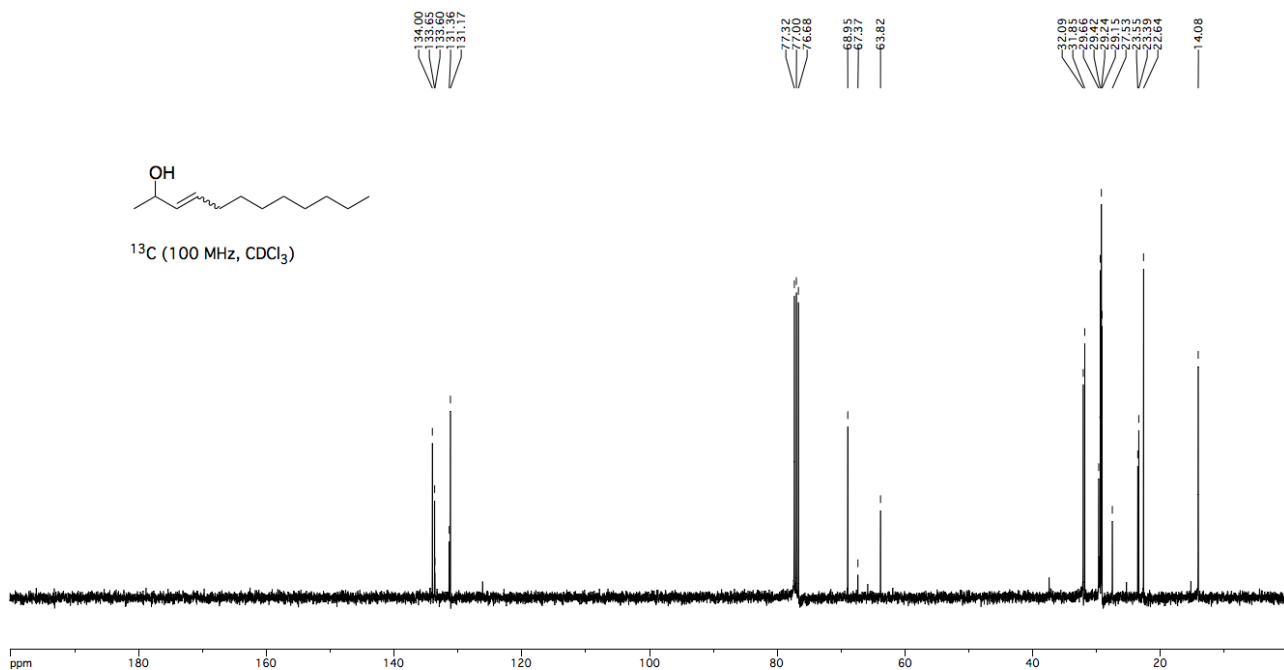
Dodec-3-en-2-ol (7)



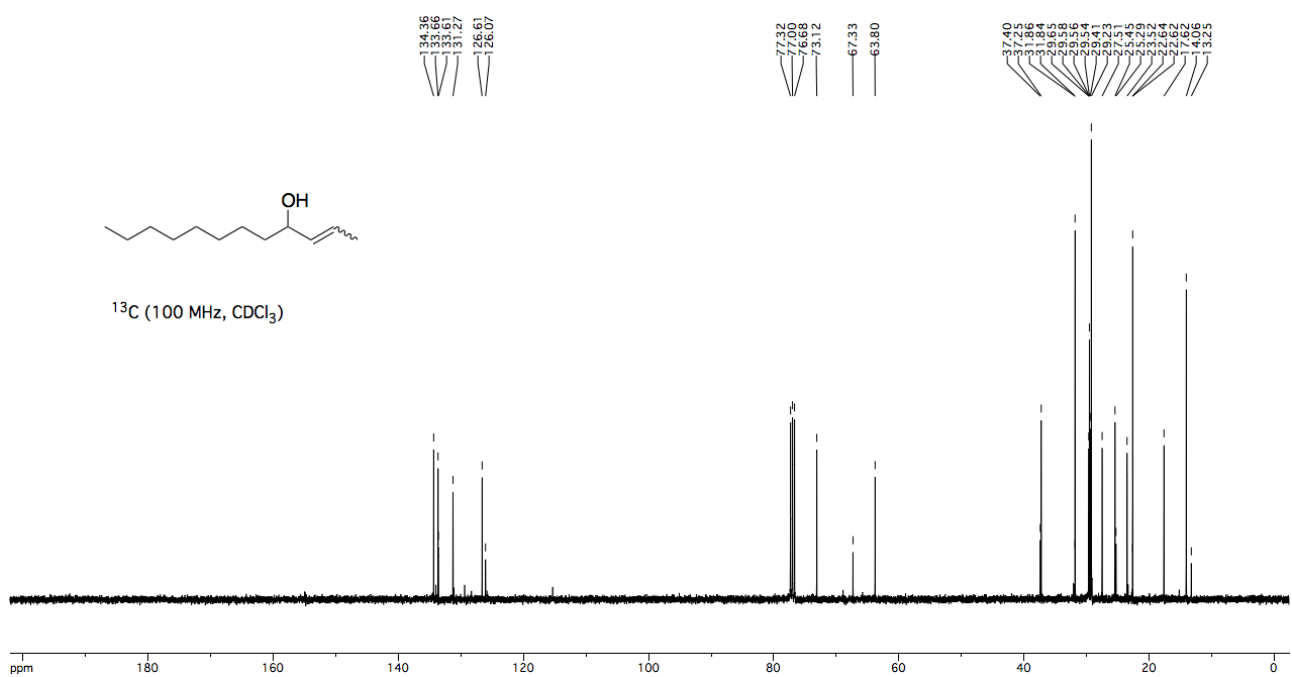
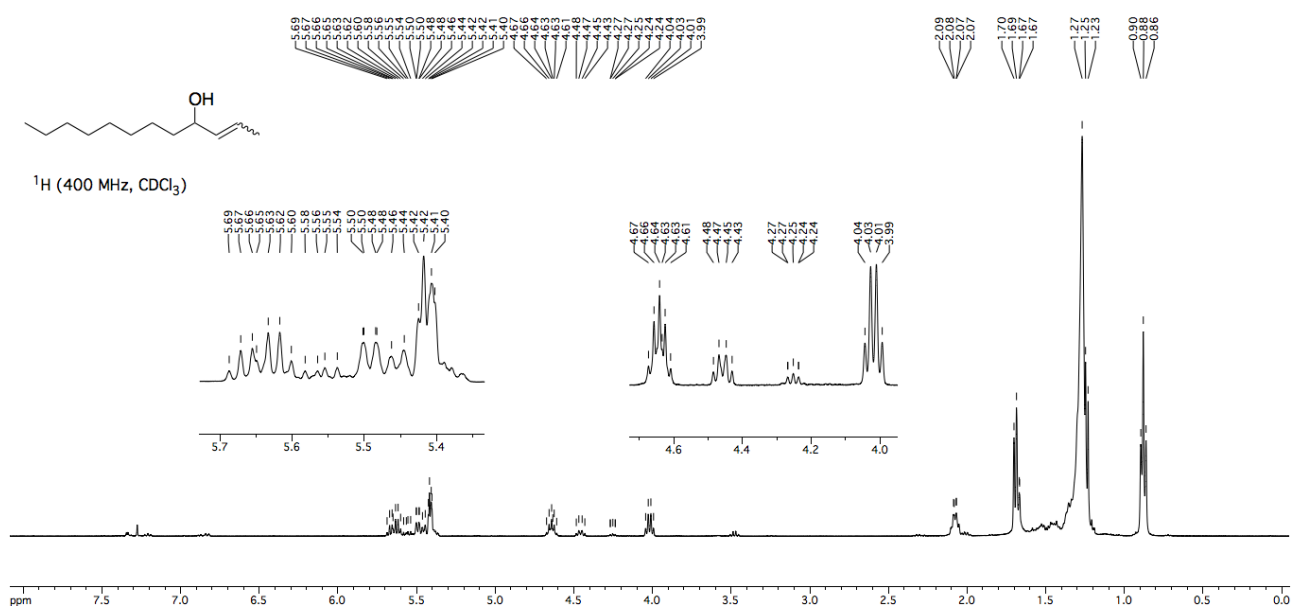
^1H (400 MHz, CDCl_3)



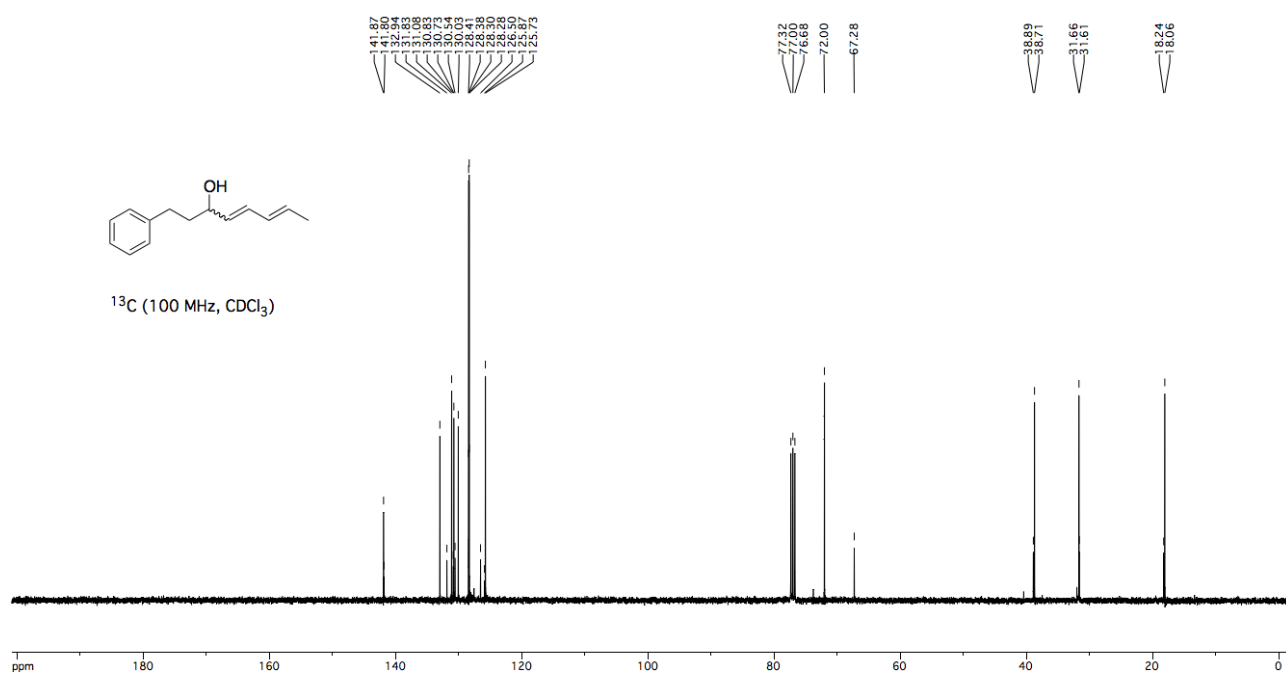
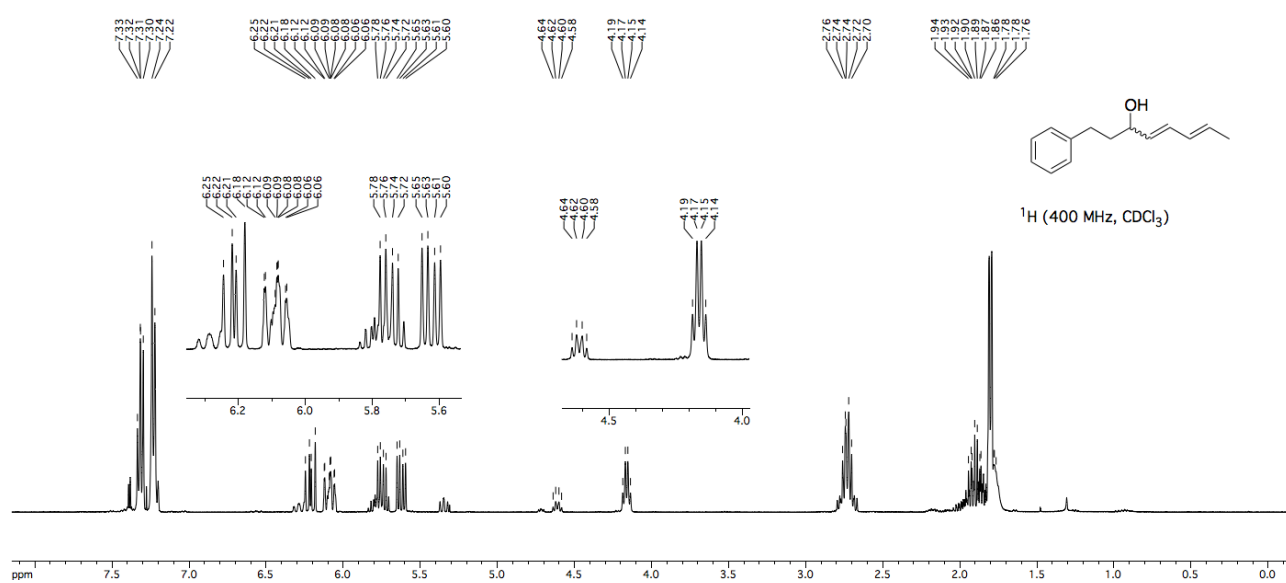
^{13}C (100 MHz, CDCl_3)



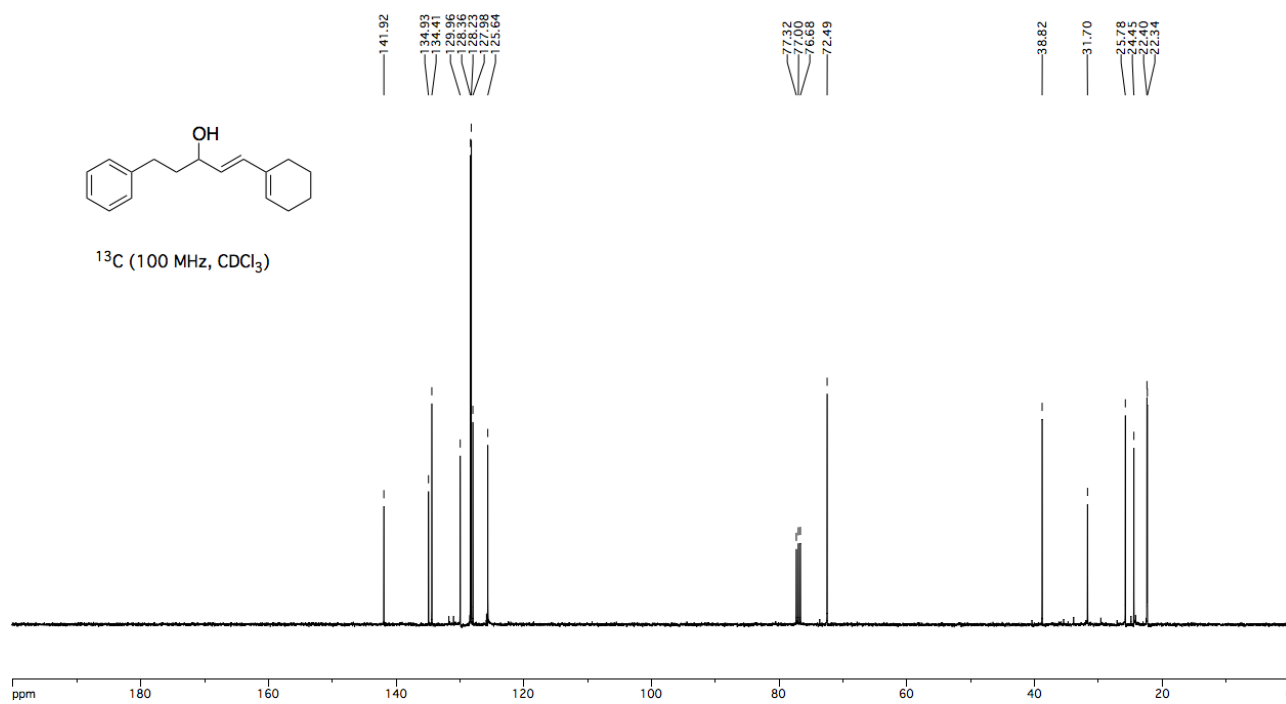
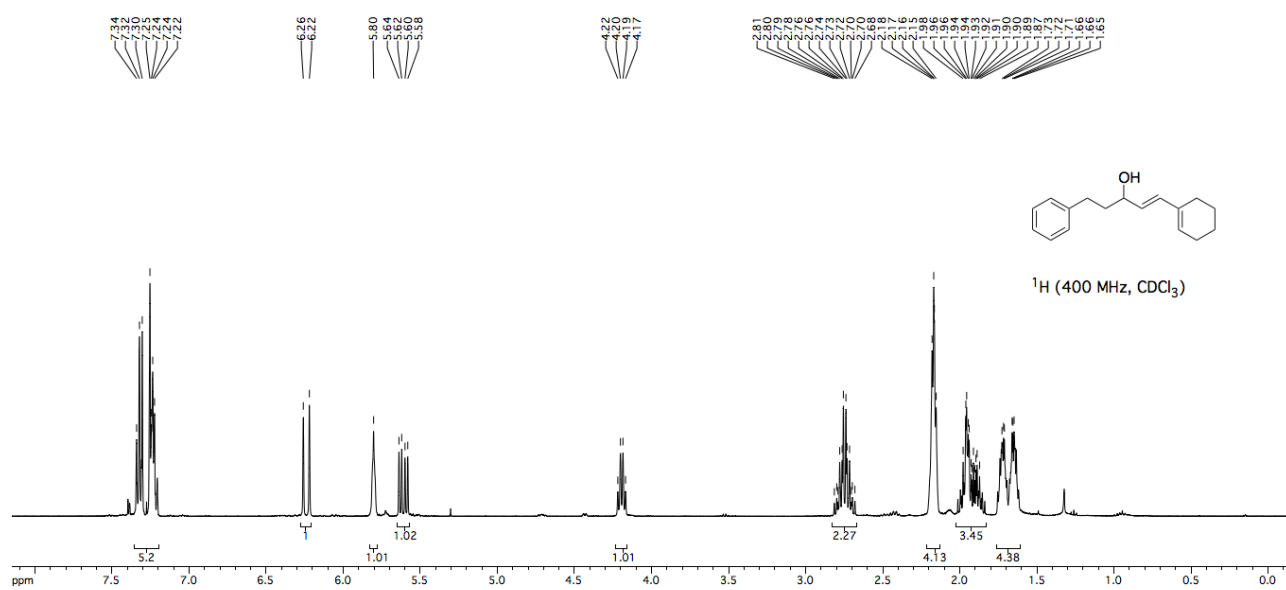
Dodec-2-en-4-ol (8)



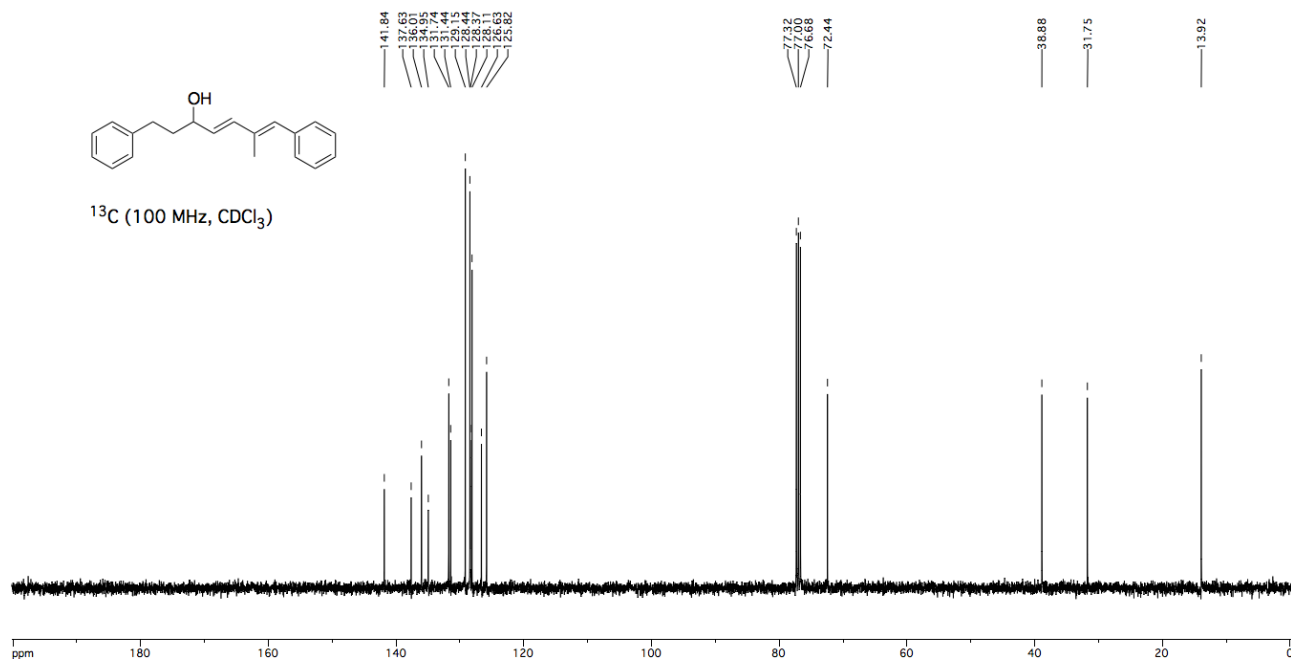
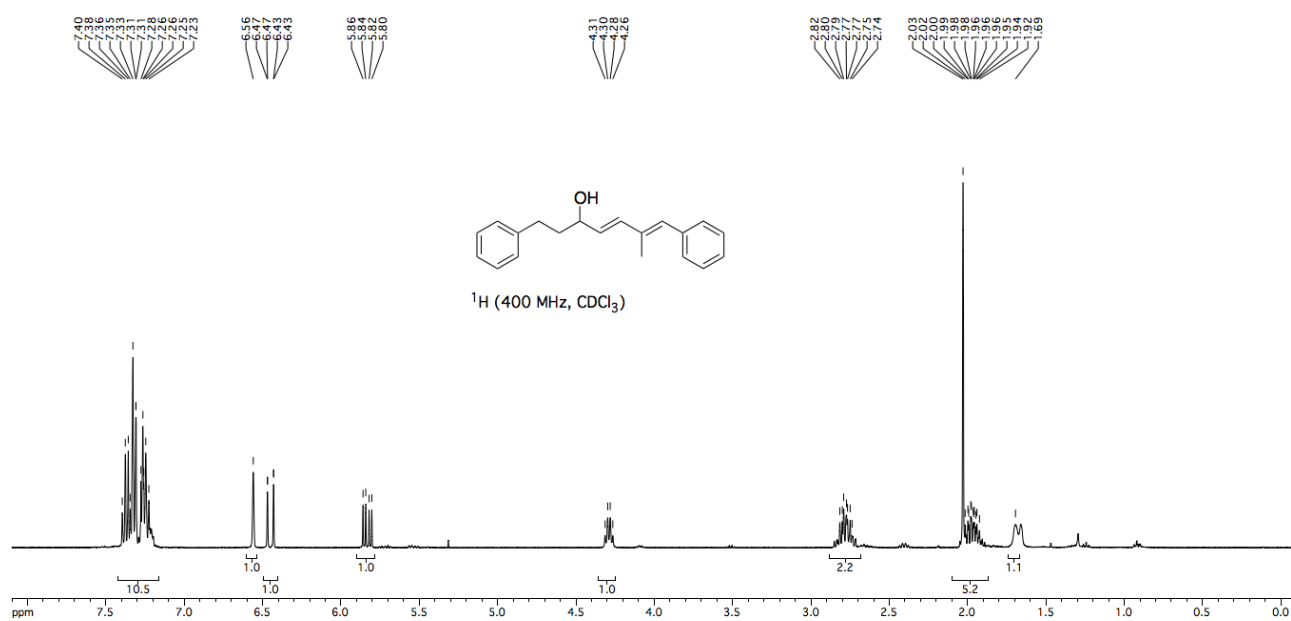
1-Phenyllocta-4,6-dien-3-ol (9a)



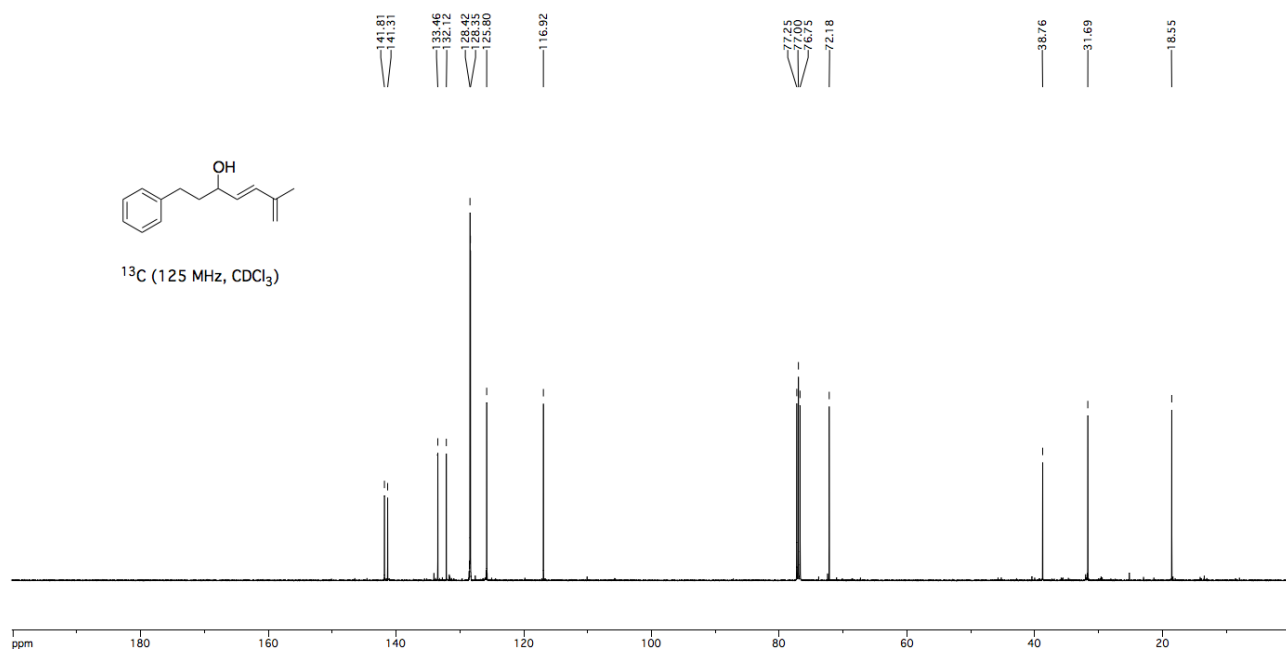
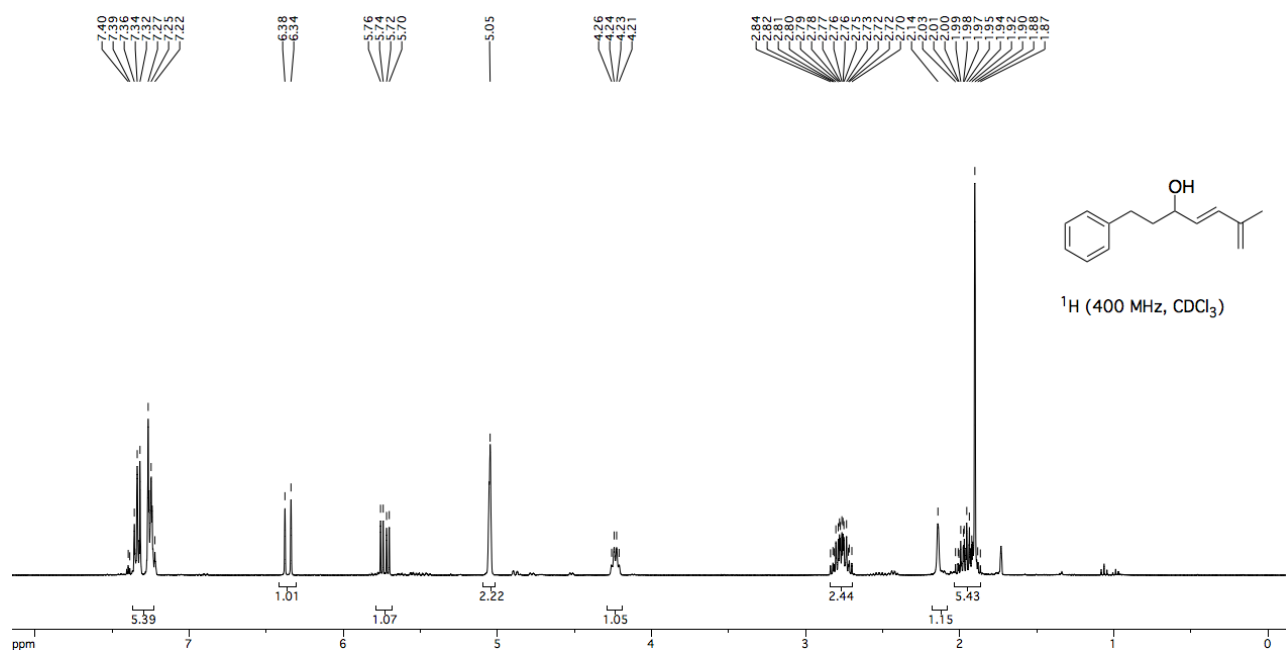
(E)-1-(Cyclohex-1-en-1-yl)-5-phenylpent-1-en-3-ol (9b)



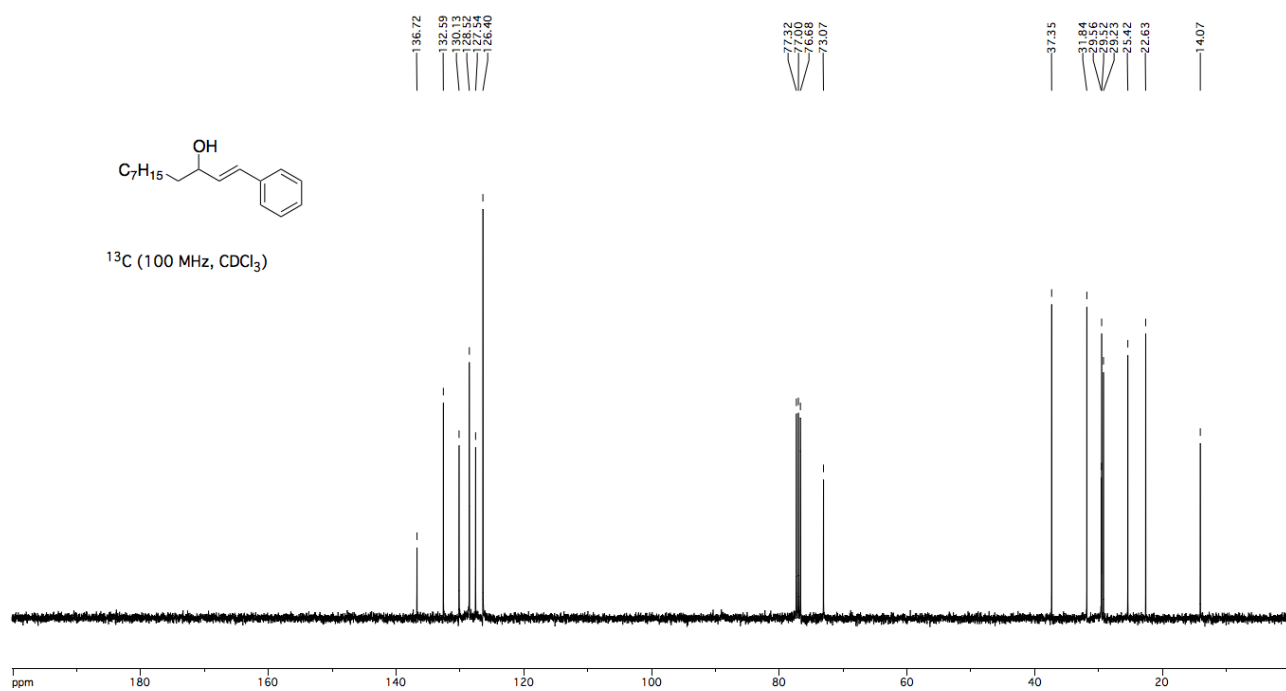
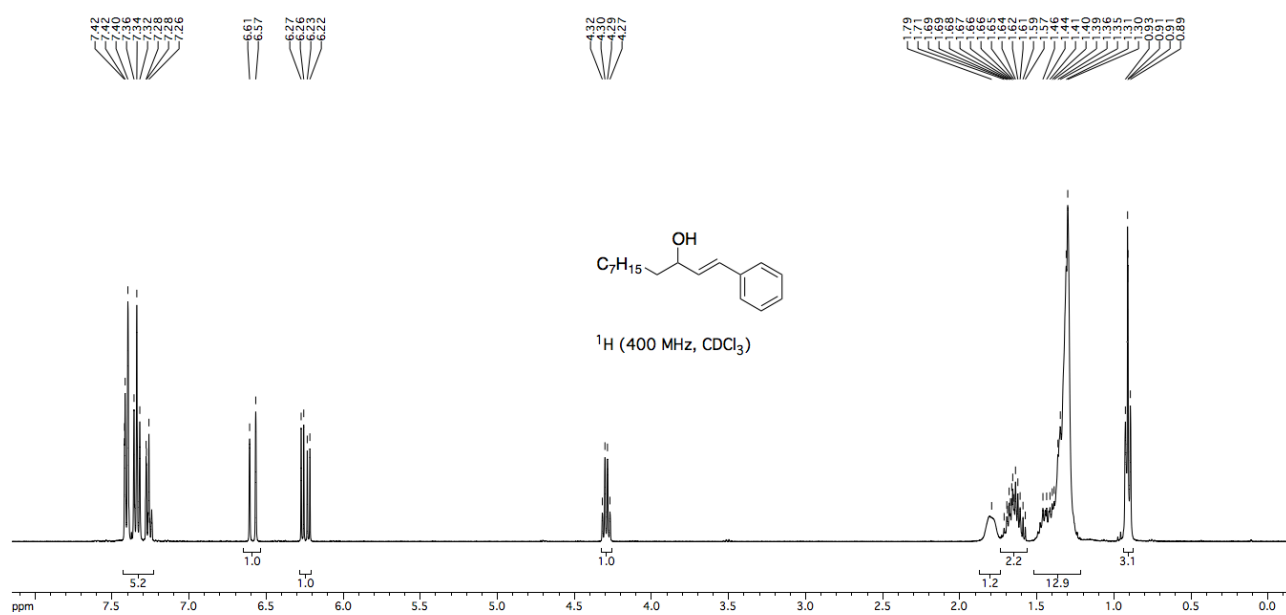
(4E,6E)-6-Methyl-1,7-diphenylhepta-4,6-dien-3-ol (9c)



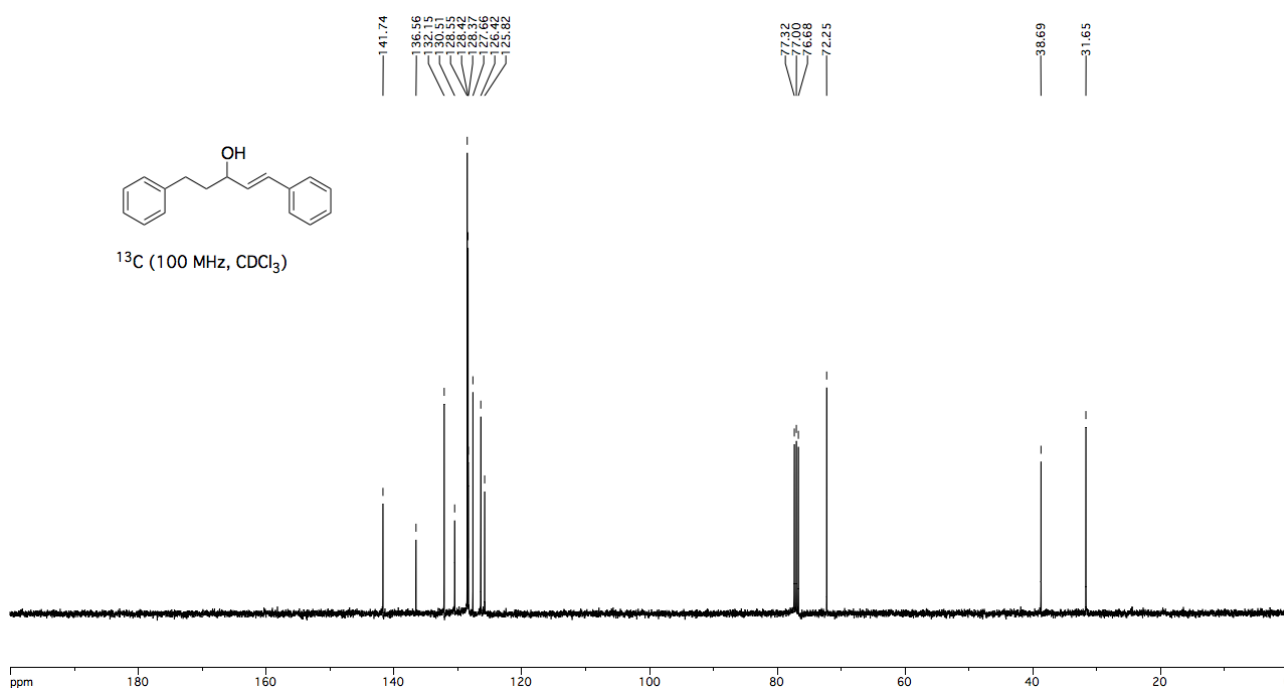
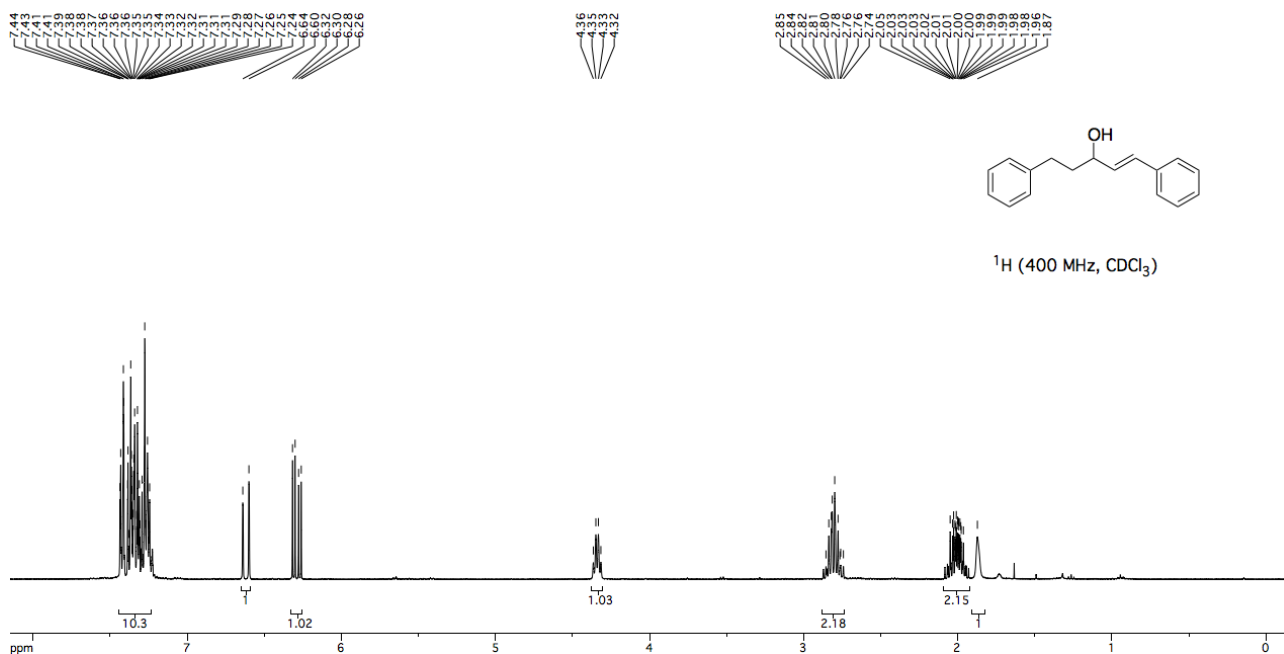
(E)-6-Methyl-1-phenylhepta-4,6-dien-3-ol (9d)



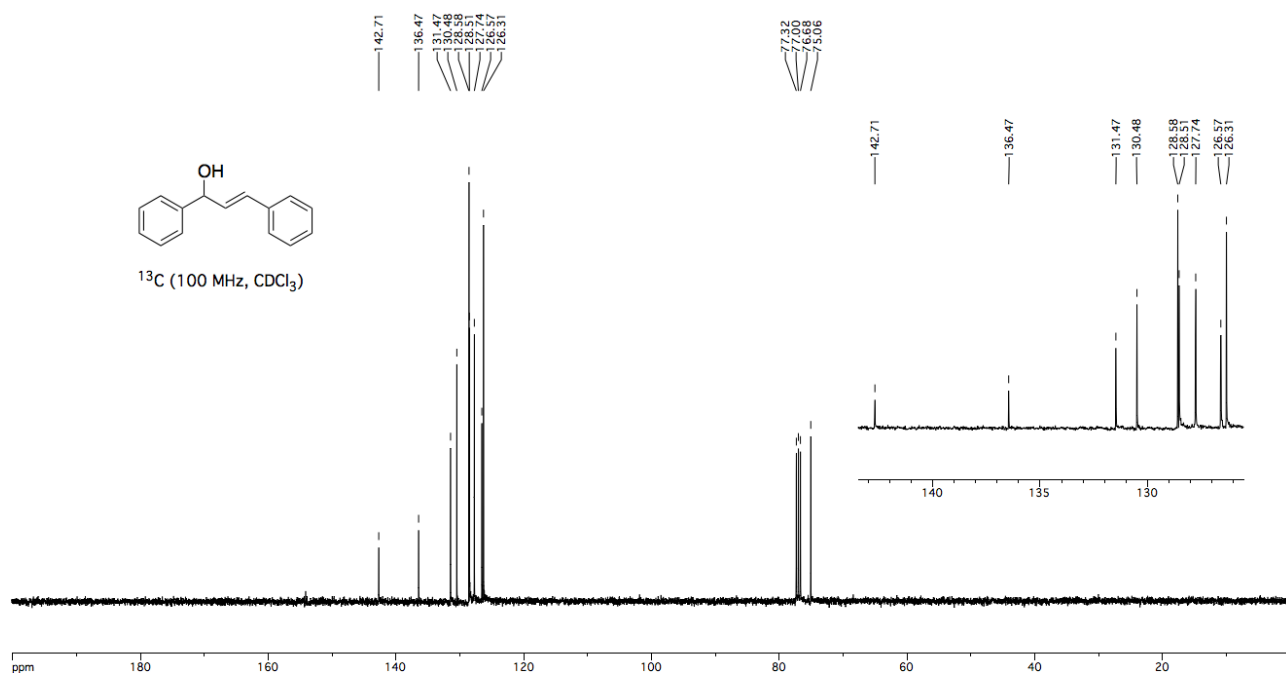
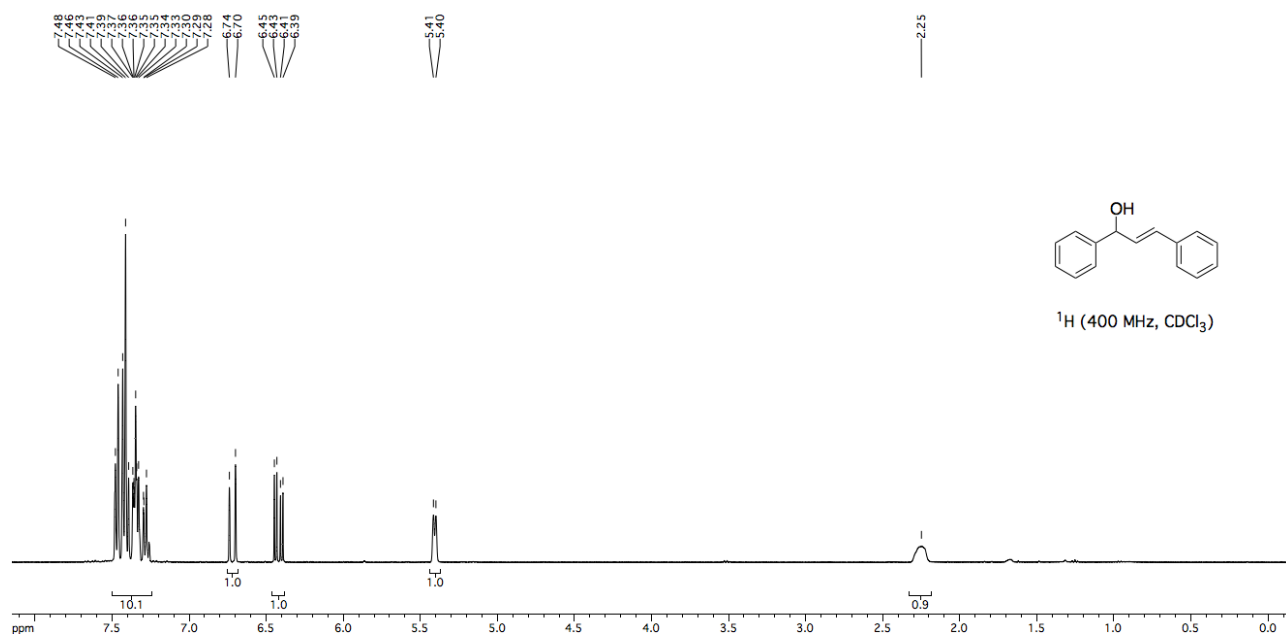
(E)-1-Phenylundec-1-en-3-ol (10a)



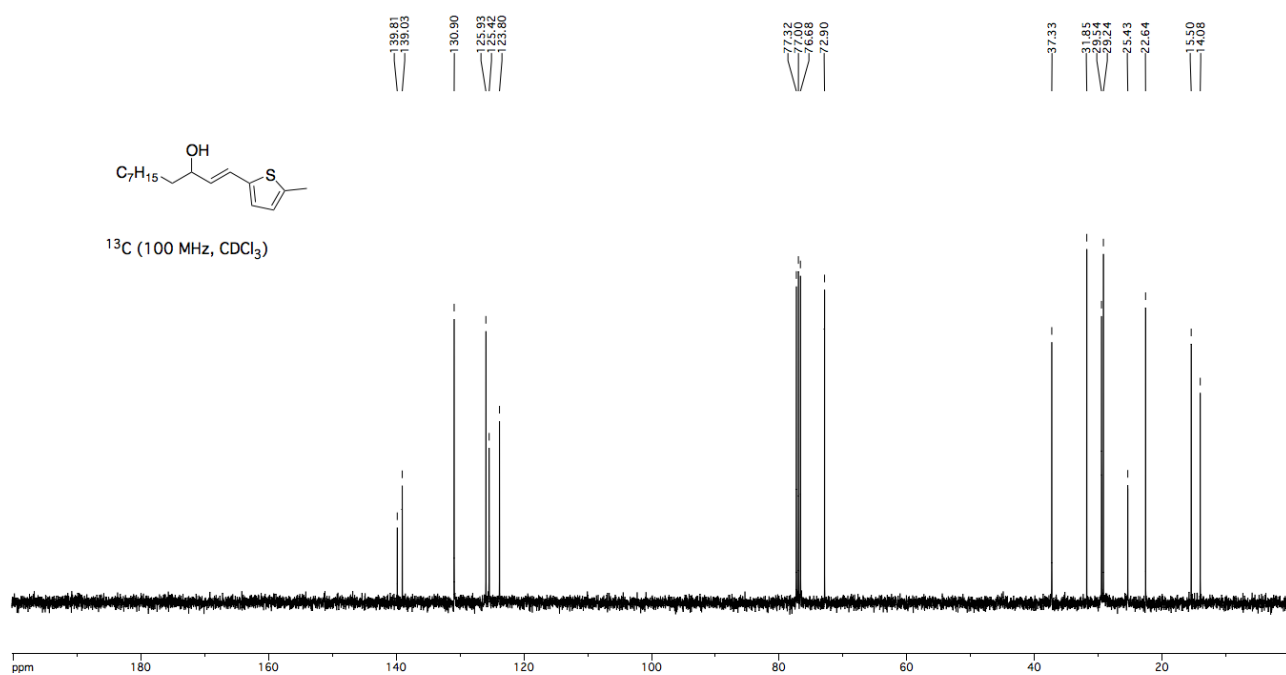
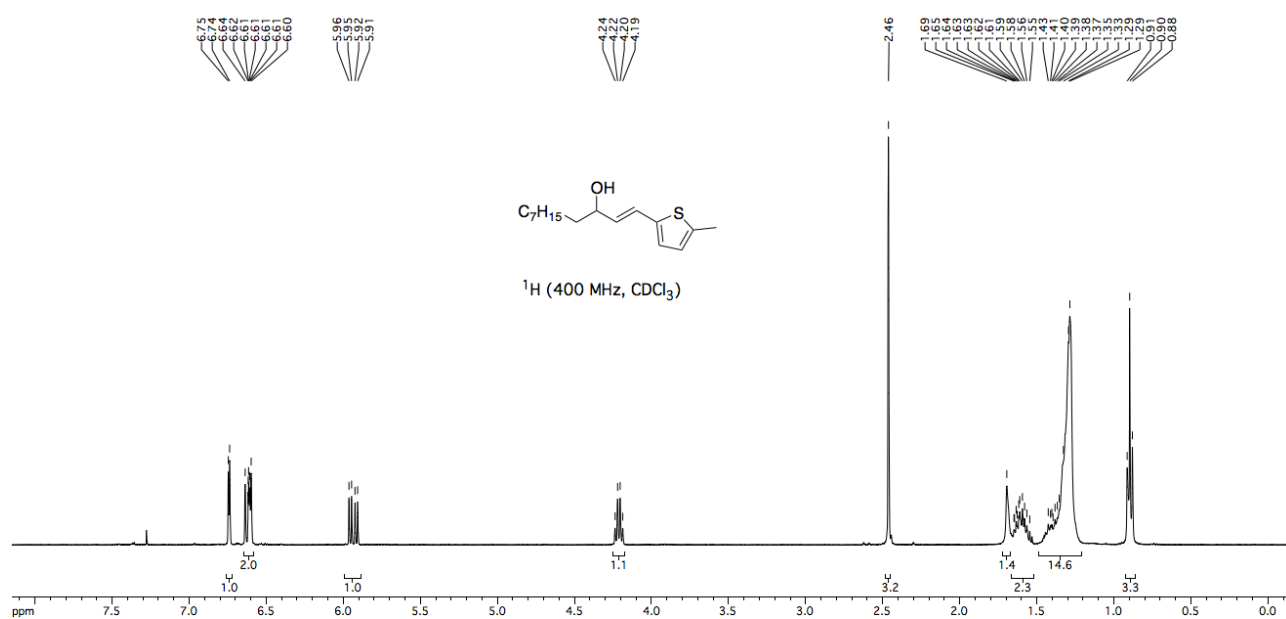
(E)-1,5-Diphenylpent-1-en-3-ol (10b)



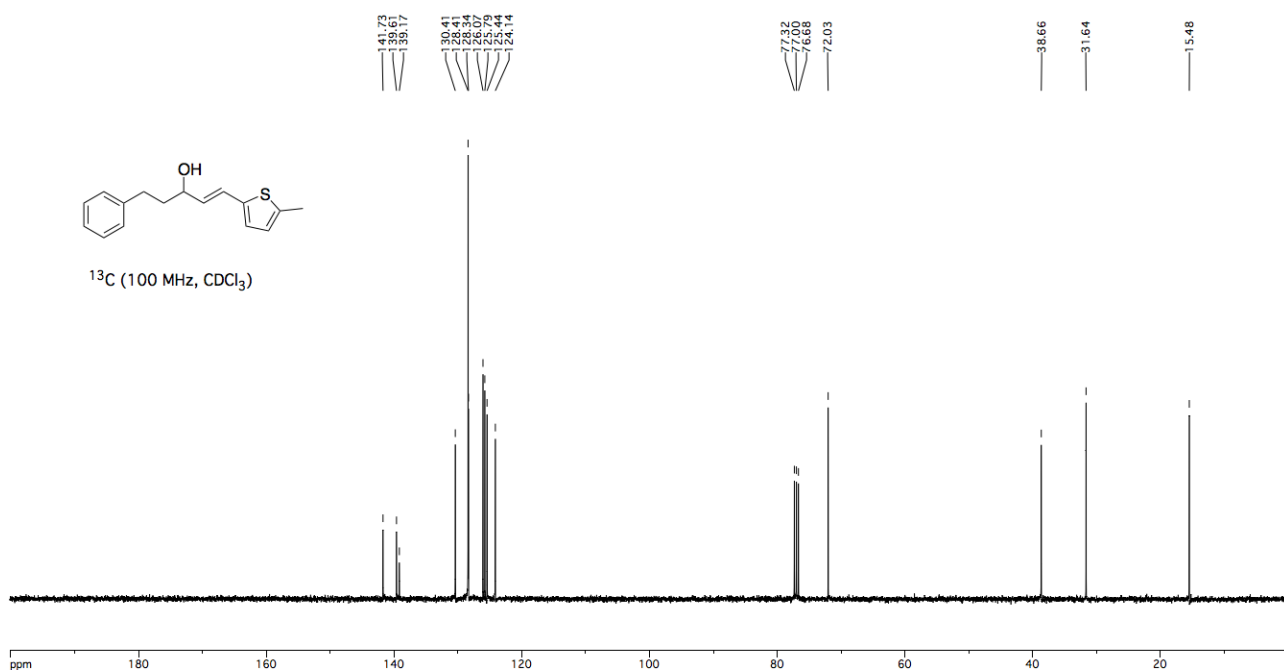
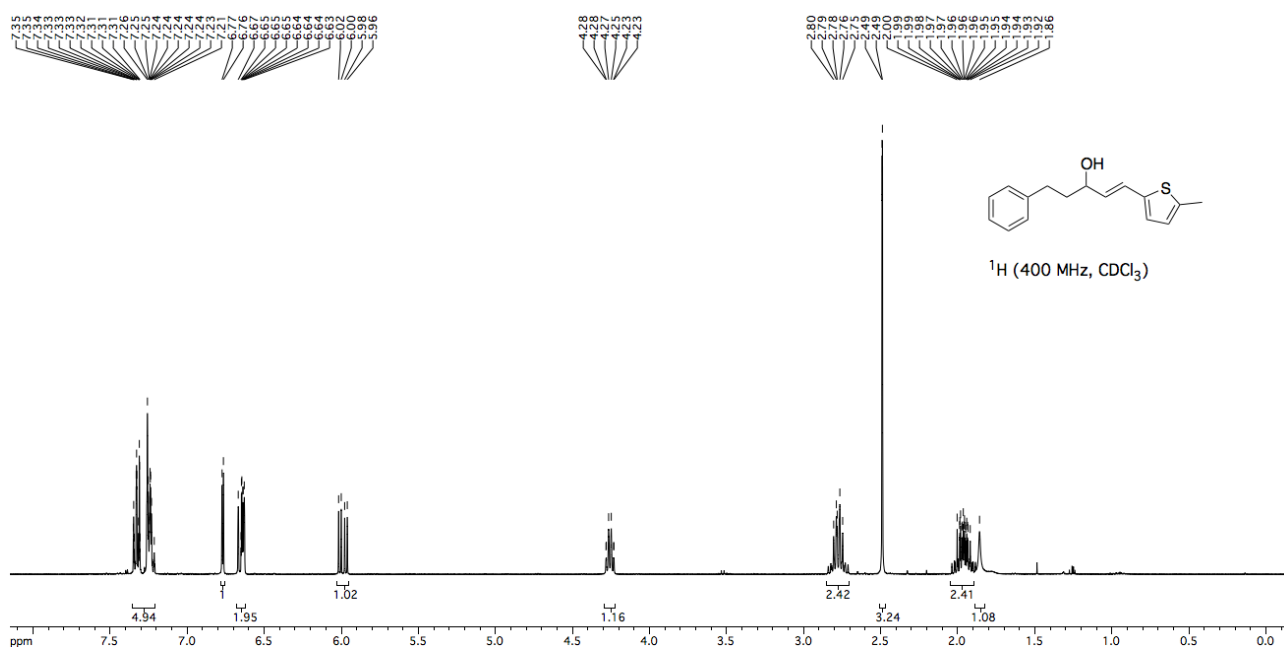
(E)-1,3-Diphenylprop-2-en-1-ol (10c)



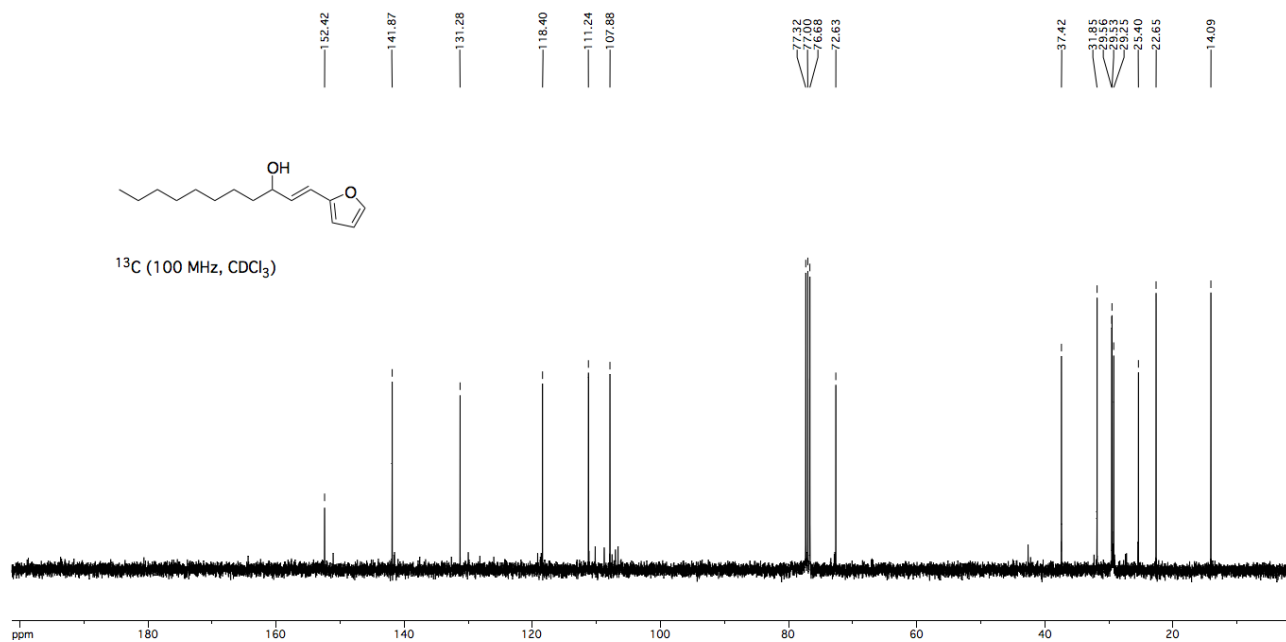
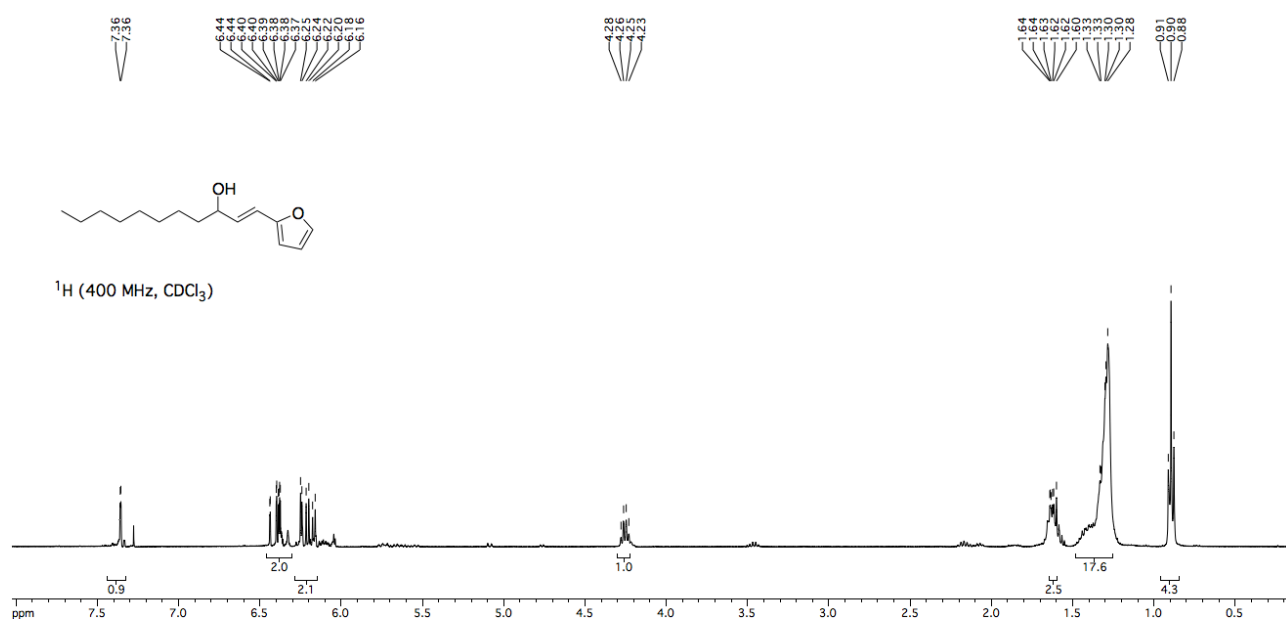
(E)-1-(5-Methylthiophen-2-yl)-undec-1-en-3-ol (10d)



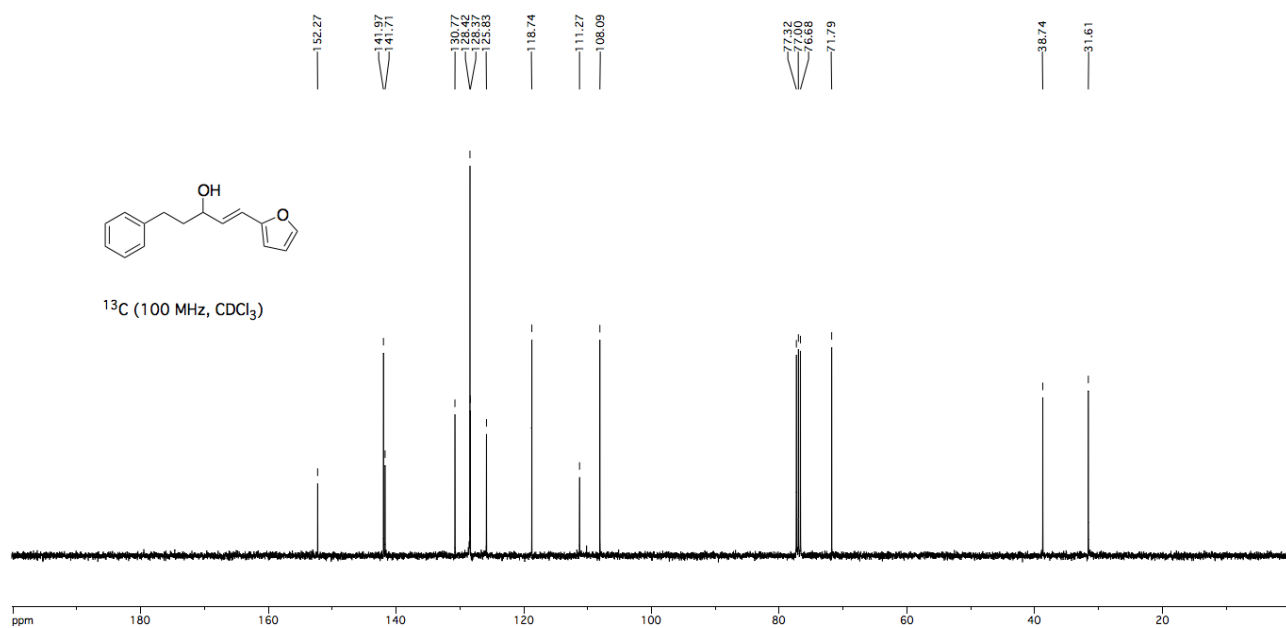
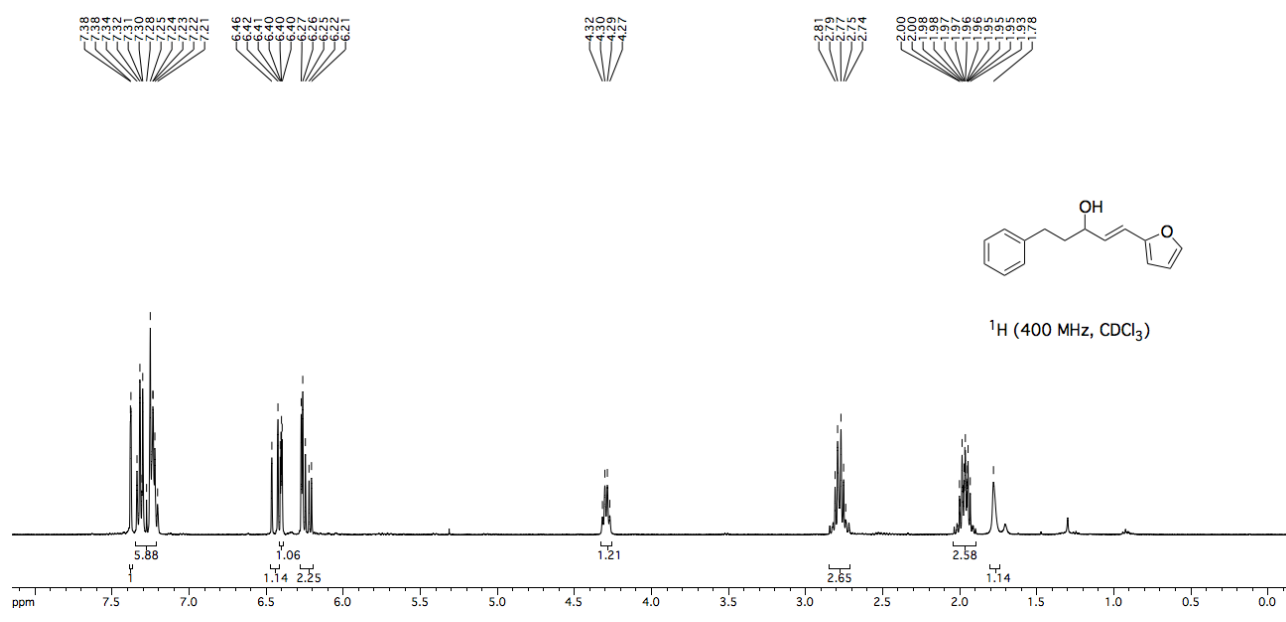
(E)-1-(5-Methylthiophen-2-yl)-5-phenylpent-1-en-3-ol (10e)



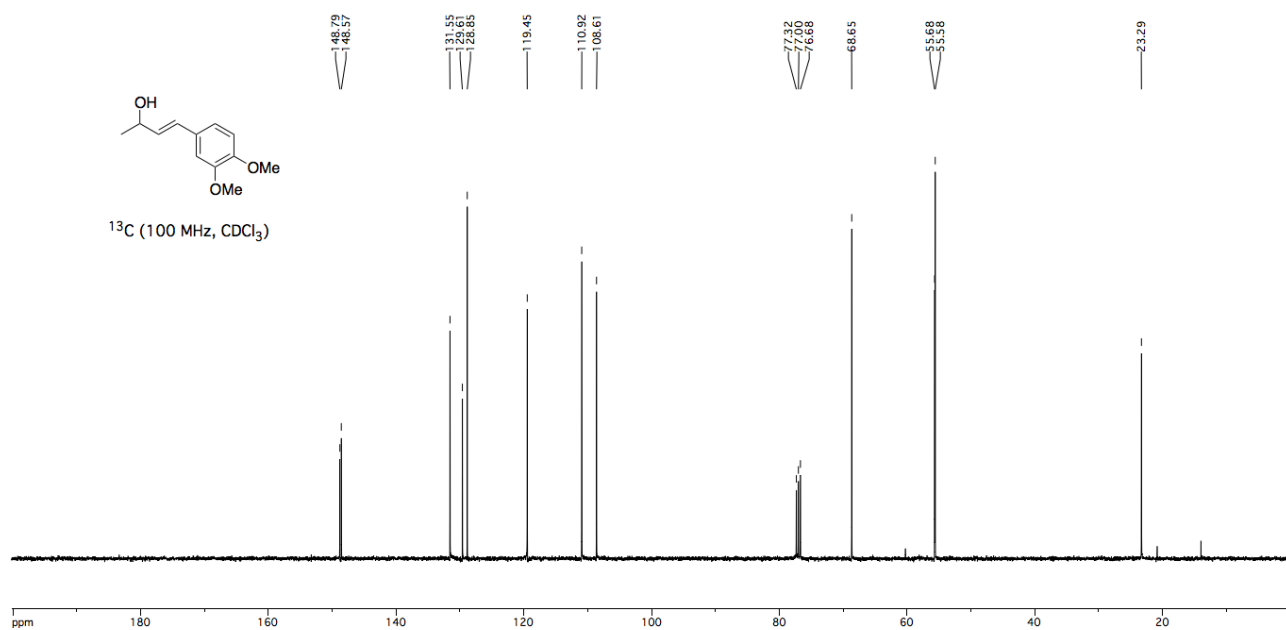
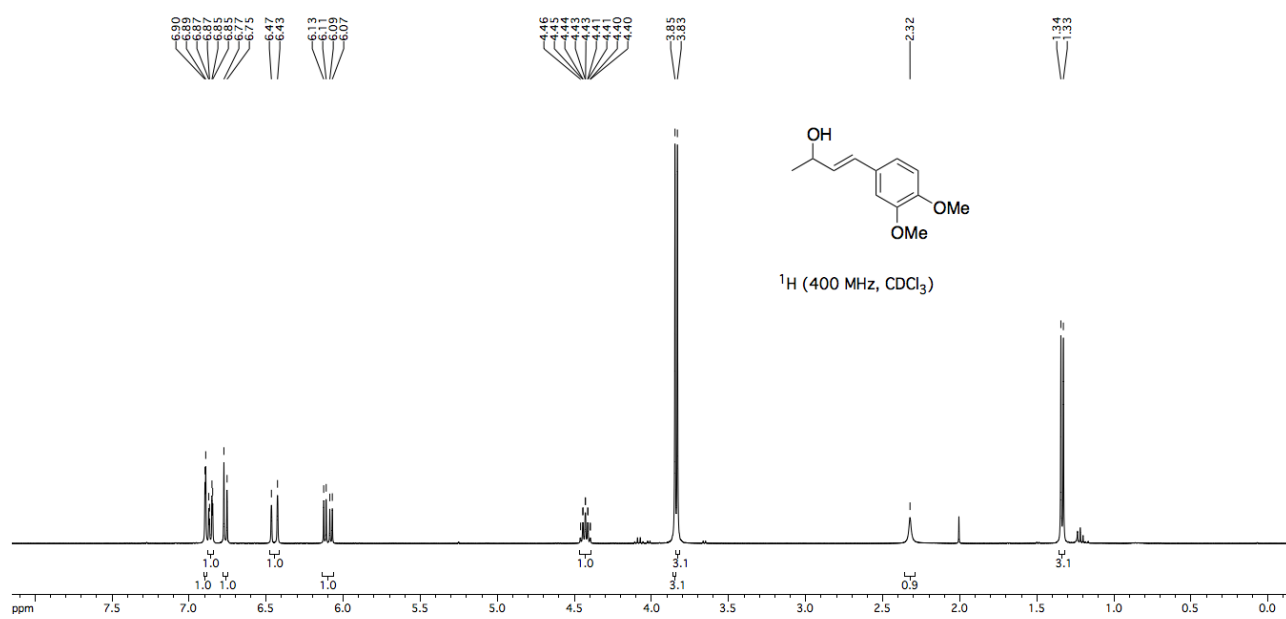
(E)-1-(Furan-2-yl)-undec-1-en-3-ol (10f)



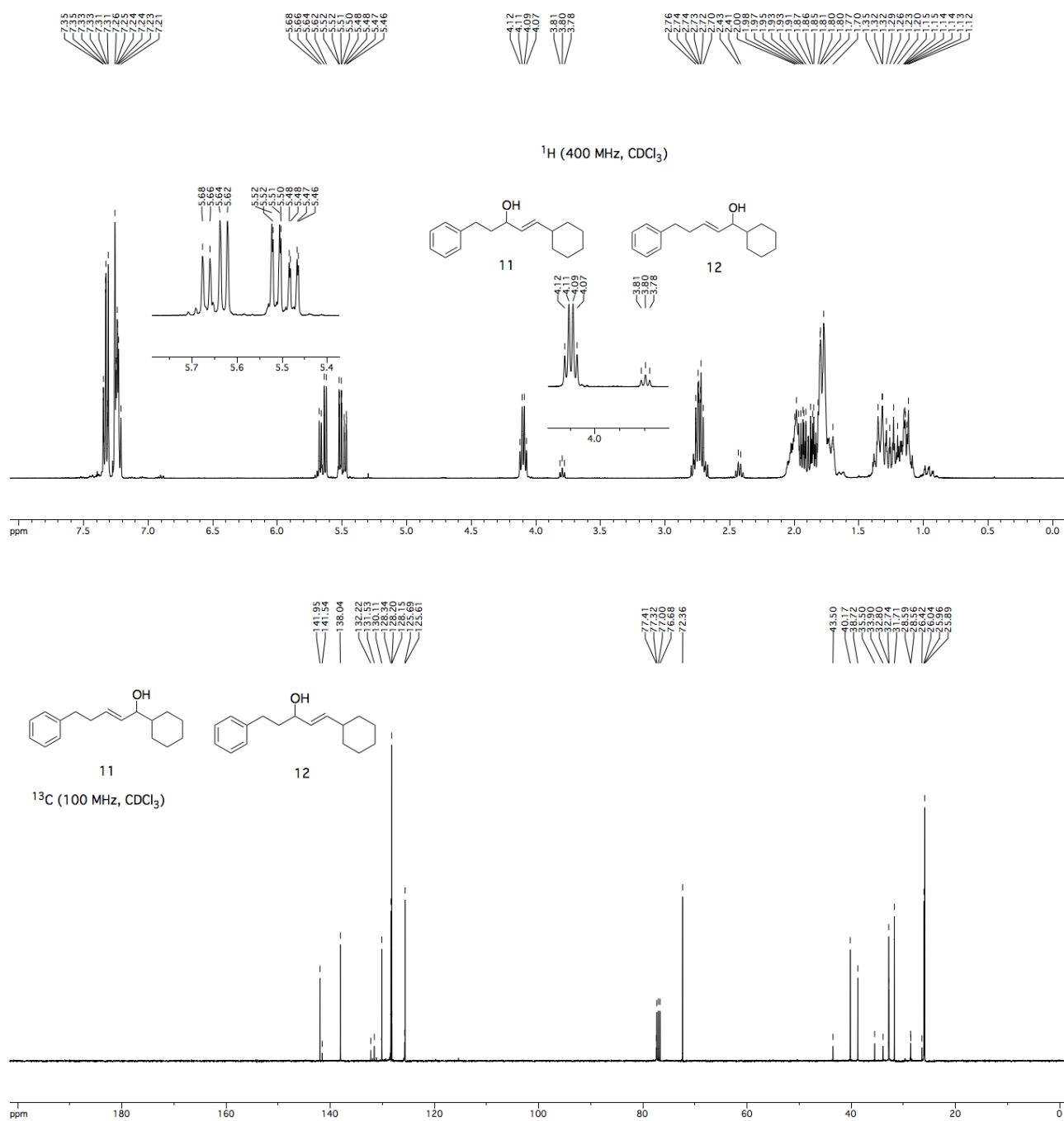
(E)-1-(Furan-2-yl)-5-phenylpent-1-en-3-ol (10g)



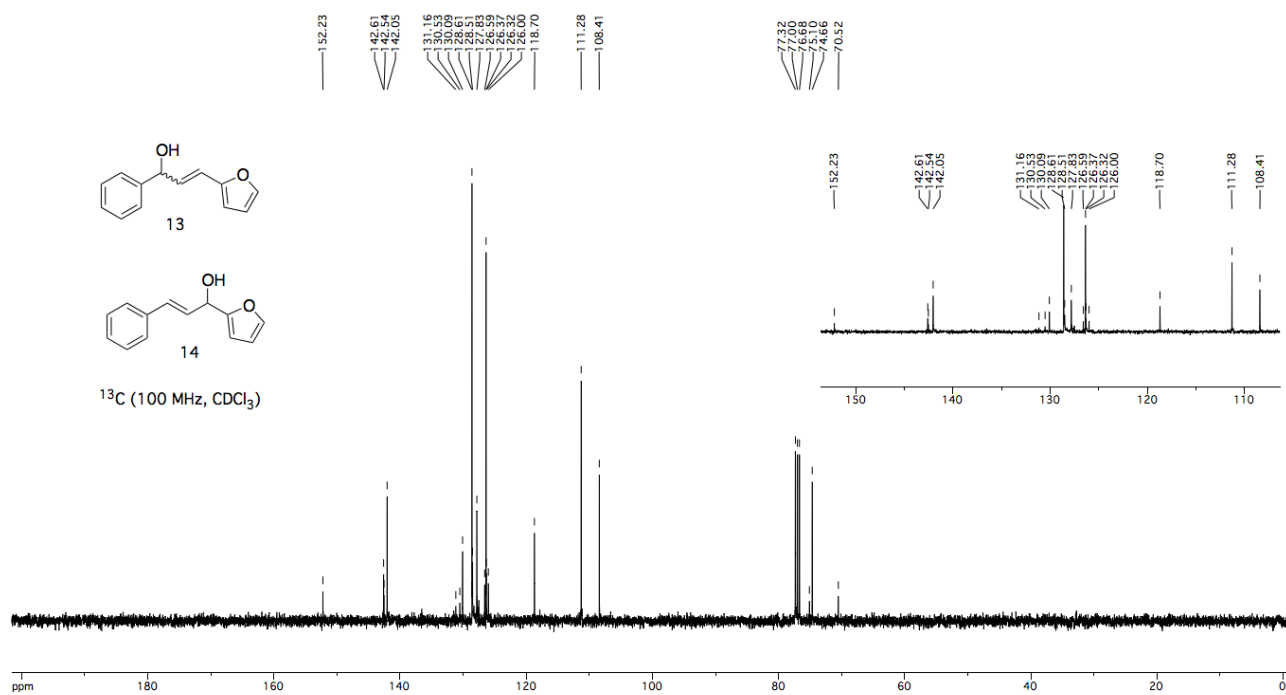
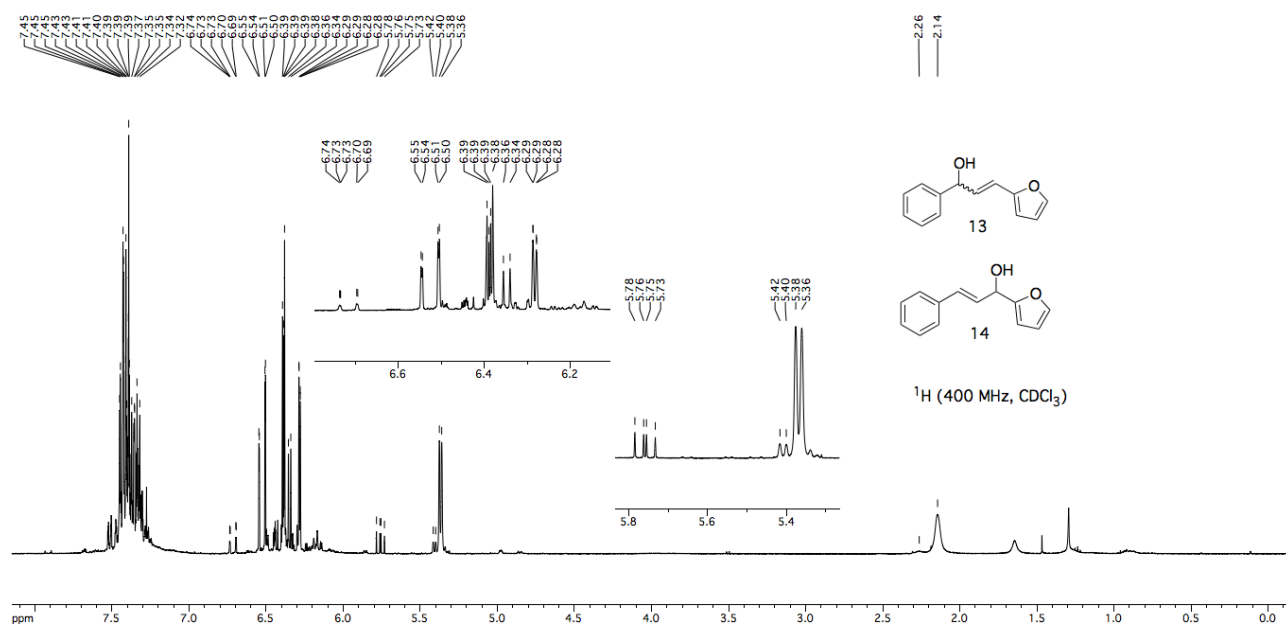
(E)-4-(3,4-Dimethoxyphenyl)-but-3-en-2-ol (10h)



(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

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