Synthesis of Allylic and Homoallylic Alcohols from Unsaturated Cyclic Ethers Using a Mild and Selective C-O Reduction Approach

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Supporting Information

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General Information: Commercial reagents were purchased and used without further purification. All glassware was flame dried under vacuum and reactions were performed under a nitrogen atmosphere, unless otherwise stated. Toluene, dichloromethane, diethyl ether, and THF were dried over a column of alumina. Benzene was distilled from sodium/benzophenone ketyl. Flash chromatography was done with Grace Davisil F60 40-63μm 60Å silica, and thin layer chromatography (TLC) was performed with EMD 250 μm silica gel 60-F_{254} plates. ^{1}H and ^{13}C NMR data was acquired on a Varian Inova 600, Bruker DRX 500, or Bruker DRX 600 and referenced to residual protic solvent ($CDCl_3$ ^{1}H [7.26 ppm], $CDCl_3$ ^{13}C [77.00 ppm], C_6F_6 ^{19}F [-164.90 ppm]) or tetramethylsilane (0.00 ppm). Infrared spectrum were acquired on a Shimadzu Prestige FT-IR. Optical rotations were measured on a Rudolph Instruments Autopol IV polarimeter. High-resolution mass spectrometry was performed at The University of Arizona Mass Spectral Facility.

General Procedure for $B(C_6F_5)_3$-catalyzed reduction of cyclic ethers

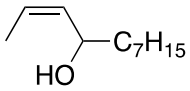
To a flame-dried vial containing a stir bar was added cyclic ether (1 eq) and dichloromethane (0.1 M), and $B(C_6F_5)_3$ (0.05 eq). Triethylsilane (1.1 eq) was added to this solution, at room temperature, over 1 hour, and the reaction was allowed to stir at room temperature until TLC indicated completion of the reduction. TBAF (1.0 M in THF, 1.5 eq) was added in one portion, and the reaction was stirred until TLC indicated complete consumption of the triethylsilyl ether. The reaction was diluted with DI water, the organic layers were separated, and the organic layer was washed with brine, dried over $MgSO_4$ and concentrated. Compounds were purified via column chromatography, yielding the desired alcohol. For all alcohol products we used either 20-35% Ethyl acetate/hexanes or 7% methanol/methylene chlorides as eluents.

(*Z*)-but-2-en-1-ol (1)

1

^{1}H NMR (499 MHz, Chloroform-d) δ 5.64 – 5.56 (m, 2H), 4.19 (d, *J* = 4.4 Hz, 2H), 2.43 (s, 1H), 1.66 (d, *J* = 5.2 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 129.26, 126.73, 57.95, 12.85; FTIR (thin film) 3343, 3022, 2922, 2874, 1449, 1032, 980 cm^{-1}; HRMS (EI^{+}) *m/z* 72.0572 [calculated mass for C_4H_8O $(M)^{+}$ 72.0575].

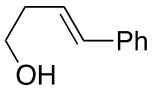
(*Z*)-undec-2-en-4-ol (2)



2

^{1}H NMR (499 MHz, Chloroform-d) δ 5.60 (dq, *J* = 11.0, 7.1 Hz, 1H), 5.43 (ddt, *J* = 10.8, 7.0, 1.8 Hz, 1H), 4.50 (dtd, *J* = 9.7, 6.8, 3.7 Hz, 1H), 1.72 (dd, *J* = 6.9, 1.7 Hz, 3H), 1.68 – 1.59 (m, 1H), 1.52 – 1.42 (m, 1H), 1.43 – 1.24 (m, 11H), 0.91 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 133.62, 126.23, 67.44, 37.48, 31.82, 29.58, 29.29, 25.35, 22.66, 14.10, 13.32; FTIR (thin film) 3350, 2926, 2857, 1456, 1001, 725 cm^{-1}; HRMS (EI^{+}) *m/z* 170.1668 [calculated mass for $C_{11}H_{22}O$ $(M)^{+}$ 170.1671].

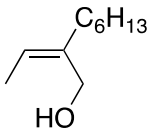
(*E*)-4-phenylbut-3-en-1-ol (3)



3

^{1}H NMR (499 MHz, Chloroform-d) δ 7.40 – 7.18 (m, 5H), 6.50 (d, *J* = 15.9 Hz, 1H), 6.21 (dt, *J* = 15.9, 7.2 Hz, 1H), 3.76 (q, *J* = 6.2 Hz, 2H), 2.49 (dtd, *J* = 7.7, 6.4, 1.4 Hz, 2H), 1.44 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.20, 132.84, 128.51, 127.26, 126.29, 126.06, 62.03, 36.43; FTIR (thin film) 3347, 3080, 3057, 3024, 2926, 2876, 1493, 1040, 962, 743, 692 cm^{-1}; HRMS (EI^{+}) *m/z* 148.0885 [calculated mass for $C_{10}H_{12}O$ $(M)^{+}$ 148.0888].

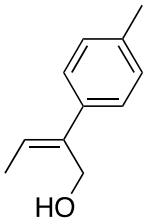
(*Z*)-2-ethylideneoctan-1-ol (4)



4

^{1}H NMR (499 MHz, Chloroform-d) δ 5.39 (q, *J* = 7.0 Hz, 1H), 4.15 (d, *J* = 5.0 Hz, 2H), 2.10 (ddt, *J* = 9.1, 6.9, 1.3 Hz, 2H), 1.67 (d, *J* = 7.0 Hz, 3H), 1.47 – 1.36 (m, 2H), 1.36 – 1.25 (m, 6H), 1.17 (t, *J* = 5.4 Hz, 1H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 139.31, 122.46, 59.99, 35.21, 31.77, 29.14, 28.30, 22.64, 14.09, 13.10; FTIR (thin film) 3327, 2957, 2928, 2859, 1456, 1379, 1242, 1005, 833, 743, 725 cm^{-1}; HRMS (EI^{+}) *m/z* 156.1511 [calculated mass for $C_{10}H_{20}O$ $(M)^{+}$ 156.1514].

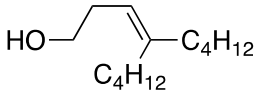
(*Z*)-2-(*p*-tolyl)but-2-en-1-ol (5)



5

^{1}H NMR (499 MHz, Chloroform-d) δ 7.32 (d, *J* = 8.1 Hz, 2H), 7.14 (d, *J* = 7.9 Hz, 2H), 5.93 (q, *J* = 7.0 Hz, 1H), 4.58 (d, *J* = 5.7 Hz, 2H), 2.34 (s, 3H), 1.88 (d, *J* = 7.0 Hz, 3H), 1.30 (t, *J* = 5.8 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 139.31, 137.99, 136.72, 129.18, 126.08, 125.78, 59.50, 21.05, 14.02; FTIR (thin film) 3350, 3022, 2920, 2862, 1512, 1437, 1024, 982, 810 cm^{-1}; HRMS (EI$^+$) *m/z* 162.1042 [calculated mass for $C_{11}H_{14}O$ $(M)^+$ 162.1045].

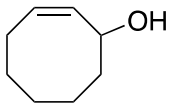
4-butyloct-3-en-1-ol (6)



6

^{1}H NMR (499 MHz, Chloroform-d) δ 5.09 (t, *J* = 7.4 Hz, 1H), 3.62 (q, *J* = 6.3 Hz, 2H), 2.30 (q, *J* = 6.8 Hz, 2H), 2.02 (dt, *J* = 14.5, 7.5 Hz, 4H), 1.41 – 1.24 (m, 9H), 0.94 – 0.87 (m, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.78, 119.44, 62.71, 36.74, 31.32, 30.84, 30.48, 29.96, 22.90, 22.55, 14.05, 14.03; FTIR (thin film) 3331, 2957, 2930, 2872, 2860, 1456, 1049, 735 cm^{-1}; HRMS (EI$^+$) *m/z* 184.1830 [calculated mass for $C_{12}H_{24}O$ $(M)^+$ 184.1827].

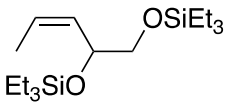
(*Z*)-cyclooct-2-enol (7)



7

^{1}H NMR (499 MHz, Chloroform-d) δ 5.61 (dddd, *J* = 10.7, 8.8, 7.2, 1.6 Hz, 1H), 5.52 (ddd, *J* = 10.8, 6.7, 1.2 Hz, 1H), 4.70 – 4.60 (m, 1H), 2.24 – 2.02 (m, 2H), 1.95 – 1.86 (m, 1H), 1.71 – 1.32 (m, 8H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 134.94, 128.68, 69.50, 38.67, 29.10, 26.34, 25.94, 23.73; FTIR (thin film) 3325, 3017, 2926, 2857, 1136, 1055, 986, 849, 752, 667 cm^{-1}; HRMS (EI$^+$) *m/z* 126.1039 [calculated mass for $C_8H_{14}O$ $(M)^+$ 126.1045].

TES-Protected Diol (8)

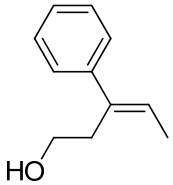


8

^{1}H NMR (499 MHz, Chloroform-d) δ 5.53 (dqd, *J* = 11.0, 6.9, 1.2 Hz, 1H), 5.37 – 5.28 (m, 1H), 4.49 (dddd, *J* = 8.6, 6.8, 5.8, 1.2 Hz, 1H), 3.57 (dd, *J* = 10.1, 6.5 Hz, 1H), 3.42 (dd, *J* = 10.1, 5.8 Hz, 1H), 1.66 (dd, *J* = 6.9, 1.8 Hz, 3H), 0.95 (td, *J* = 7.9, 2.0 Hz, 18H), 0.59 (q, *J* = 8.1 Hz, 12H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 132.03, 125.54, 69.38, 67.40, 13.56, 6.78, 6.77, 4.89, 4.45; FTIR

(thin film) 3019, 2955, 2913, 2878, 1460, 1414, 1238, 1121, 1092, 1005, 964, 785, 743 cm^{-1}; HRMS (EI^+) *m/z* 330.2415 [calculated mass for $C_{17}H_{38}O_2Si_2$ $(M)^+$ 330.2410].

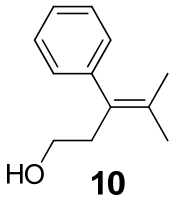
(*E*)-3-phenylpent-3-en-1-ol (**9**)



9

^1H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.27 (m, 4H), 7.24 – 7.20 (m, 1H), 5.91 (qt, *J* = 6.9, 0.6 Hz, 1H), 3.64 (t, *J* = 6.8 Hz, 2H), 2.86 – 2.80 (m, 2H), 1.85 (dt, *J* = 6.9, 0.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.64, 136.83, 128.35, 126.79, 126.24, 125.79, 61.22, 32.87, 14.37; IR (neat) 3338, 3028, 2956, 2933, 2877, 1492, 1442, 1045, 1020, 758, 742, 698 cm^{-1}; HRMS (EI^+) m/z 162.1042 [(M^+) calcd for $C_{11}H_{14}O$ 162.1045].

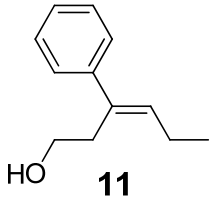
4-methyl-3-phenylpent-3-en-1-ol (**10**)



10

^1H NMR (600 MHz, $CDCl_3$) δ 7.33 – 7.29 (m, 2H), 7.23 – 7.20 (m, 1H), 7.11 – 7.08 (m, 2H), 3.54 (td, *J* = 6.8, 5.9 Hz, 2H), 2.67 (t, *J* = 6.8 Hz, 2H), 1.87 (s, 3H), 1.57 (s, 3H), 1.26 (t, *J* = 5.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.24, 131.16, 130.81, 128.88, 128.12, 126.14, 60.91, 37.52, 22.32, 20.39; IR (neat) 3342, 3323, 3307, 2954, 2914, 2875, 1465, 1255, 1039, 702 cm^{-1}; HRMS (EI^+) m/z 176.1201 [(M^+); calcd for $C_{12}H_{16}O$: 176.1201].

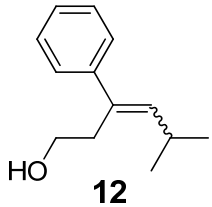
(*E*)-3-phenylhex-3-en-1-ol (**11**)



11

^1H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.28 (m, 4H), 7.25 – 7.20 (m, 1H), 5.82 (t, *J* = 7.3 Hz, 1H), 3.62 (td, *J* = 6.9, 5.9 Hz, 2H), 2.81 (t, *J* = 6.9 Hz, 2H), 2.25 (p, *J* = 7.5 Hz, 2H), 1.33 (t, *J* = 5.9 Hz, 1H), 1.07 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.57, 135.28, 133.64, 128.33, 126.81, 126.30, 61.34, 33.11, 22.02, 14.40; IR (neat) 3338, 3331, 3024, 2962, 2931, 2872, 1492, 1456, 1444, 1043, 1029, 759, 696 cm^{-1}; HRMS (EI^+) m/z 176.1208 [(M^+); calcd for $C_{12}H_{16}O$: 176.1201].

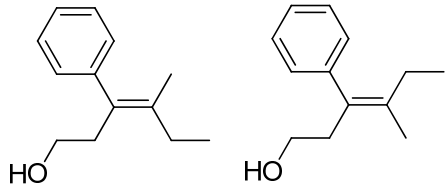
(*Z*/*E*)-5-methyl-3-phenylhex-3-en-1-ol (12)



12

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.26 (m, 4H), 7.25 – 7.12 (m, 1H), 5.63 (d, *J* = 9.7 Hz, 1H), 3.61 (t, *J* = 6.9 Hz, 2H), 2.81 (dd, *J* = 7.1, 6.7 Hz, 2H), 1.05 (dd, *J* = 6.6, 2.4 Hz,6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.57, 140.65, 139.55, 138.18, 133.41, 128.34, 128.28, 128.23, 126.83, 126.72, 126.41, 61.47, 60.47, 42.45, 33.29, 28.00, 27.89, 23.37, 23.31; IR (neat) 3354, 3319, 2958, 2931, 2868, 1492, 1463, 1444, 1361, 1045, 1029, 761, 698 cm^{-1}; HRMS (EI^{+}) m/z 190.1364 [(M^{+}) calcd for $C_{13}H_{18}O$ 190.1358]

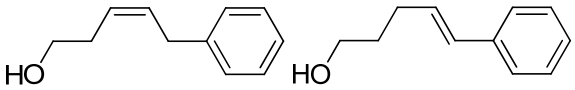
(*E*/*Z*)-4-methyl-3-phenylhex-3-en-1-ol (13)



13

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.33 – 7.28 (m, 4H), 7.24 – 7.19 (m, 2H), 7.08 (tt, *J* = 8.0, 1.7 Hz, 4H), 3.56 – 3.49 (m, 4H), 2.64 (dt, *J* = 13.8, 6.5 Hz, 4H), 2.27 – 2.21 (m, 2H), 1.90 – 1.85 (m, 2H), 1.84 (s, 4H), 1.54 (d, *J* = 0.9 Hz, 2H), 1.27 (dt, *J* = 8.7, 5.9 Hz, 2H), 1.08 (t, *J* = 7.5 Hz, 2H), 0.90 (t, *J* = 7.5 Hz, 4H);^{13}C NMR (125 MHz, $CDCl_3$) δ 143.20, 133.52, 128.87, 128.68, 128.66, 128.12, 128.03, 126.13, 61.01, 60.74, 58.11, 40.36, 37.72, 36.99, 28.65, 27.06, 19.51, 17.27, 13.28, 13.21; IR (neat) 3355 3338, 3331, 3039, 3024, 2962, 2930, 2872, 1492, 1446, 1456, 1444, 1043, 1029, 759, 696 cm^{-1}; HRMS (EI^{+}) m/z 190.1356 [(M^{+}) calcd for $C_{13}H_{18}O$ 190.1358].

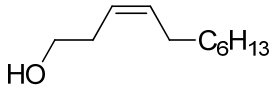
(*Z*)-5-phenylpent-3-en-1-ol/(*E*)-5-phenylpent-4-en-1-ol (14)



14

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.29 (m, 7H), 7.25 – 7.20 (m, 5H), 6.46 (dt, *J* = 15.8, 1.4 Hz, 1H), 6.26 (dt, *J* = 15.8, 6.9 Hz, 1H), 5.79 (dtt, *J* = 10.6, 7.5, 1.6 Hz, 1.5H), 5.56 (dtt, *J* = 10.7, 7.4, 1.6 Hz, 1.5H), 3.78 – 3.69 (m, 5H), 3.47 (dd, *J* = 7.5, 0.6 Hz, 3H), 2.52 – 2.45 (m, 3H), 2.38 – 2.31 (m, 2H), 1.79 (ddd, *J* = 14.7, 7.4, 6.5 Hz, 2H), 1.01 – 0.94 (m, 1.5), 0.59 – 0.52 (m, 1H);^{13}C NMR (100 MHz, $CDCl_3$) δ 140.71, 137.60, 131.41, 130.37, 130.02, 128.48, 128.47, 128.29, 126.94, 126.19, 125.96, 125.93, 62.39, 62.26, 33.58, 32.23, 30.81, 29.30, 6.77, 6.40; IR (neat) 3377, 3358, 3340, 3323, 3026, 2935, 2912, 1494, 1462, 1454, 1068, 1060, 1006, 740, 694 cm^{-1}; HRMS (EI^{+}) m/z 162.1051 [(M^{+}) calcd for $C_{11}H_{14}O$ 162.1045].

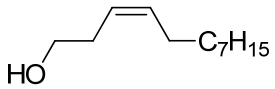
(*Z*)-undec-3-en-1-ol (15)



15

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.56 (dtt, *J* = 10.4, 7.3, 1.5 Hz, 1H), 5.35 (dtt, *J* = 10.6, 7.4, 1.6 Hz, 1H), 3.64 (dd, *J* = 12.3, 6.5 Hz, 2H), 2.33 (dtdt, *J* = 7.1, 6.5, 1.4, 0.6 Hz, 2H), 2.06 (dd, *J* = 13.3, 6.9 Hz, 2H), 1.40 – 1.20 (m, 12H), 0.91 – 0.85 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.62, 124.90, 62.35, 31.84, 30.80, 29.70, 29.26, 29.19, 27.37, 22.65, 14.09; IR(neat) 3331, 3007, 2954, 2924, 2854, 1465, 1458, 1049, 1020, 970, 723 cm^{-1}; HRMS (EI$^+$) m/z 170.1676 [(M^+) calcd for $C_{11}H_{22}O$ 170.1671].

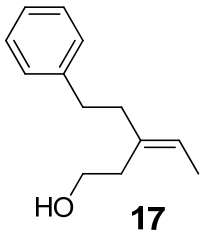
(*Z*)-dodec-3-en-1-ol (16)



16

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.61 – 5.50 (m, 1H), 5.41 – 5.30 (m, 1H), 3.63 (ddd, *J* = 12.2, 7.8, 6.3 Hz, 2H), 2.33 (dtdt, *J* = 7.1, 6.5, 1.4, 0.6 Hz, 2H), 2.10 – 1.98 (m, 2H), 1.39 – 1.20 (m, 13H), 0.92 – 0.83 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.63, 124.90, 62.36, 31.88, 30.80, 29.70, 29.49, 29.31, 29.29, 27.38, 22.67, 14.10; IR (neat) 3323, 3007, 2954, 2924, 2854, 1467, 1456, 1377, 1049, 1024, 968, 721 cm^{-1}; HRMS (EI+) m/z 166.1717 [$(M-H_2O)^+$ calcd for $C_{12}H_{22}$ 166.1722].

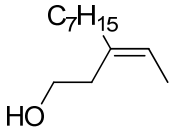
(*Z*)-3-phenethylpent-3-en-1-ol (17)



17

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 – 7.24 (m, 2H), 7.20 – 7.14 (m, 3H), 5.45 (q, *J* = 6.8 Hz, 1H), 3.68 (t, *J* = 6.8 Hz, 2H), 2.71 (dt, *J* = 18.8, 8.3 Hz, 2H), 2.39 (t, *J* = 6.8 Hz, 2H), 2.35 – 2.26 (m, 2H), 1.64 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.13, 135.33, 128.31, 128.26, 125.75, 122.40, 60.88, 38.82, 34.96, 33.27, 13.48; IR(neat) 3342, 3323, 3305, 3026, 2935, 2875, 2860, 1602, 1494, 1454, 1043, 1029, 744, 698 cm^{-1}; HRMS (EI$^+$) m/z 190.1349 [(M^+) calcd for $C_{13}H_{18}O$ 190.1358].

(*Z*)-3-ethylidenedecan-1-ol (18)

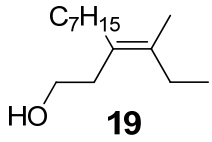


18

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.39 (qd, *J* = 6.8, 6.1 Hz, 1H), 3.65 (td, *J* = 6.8, 5.9 Hz, 2H), 2.34 (t, *J* = 6.8 Hz, 2H), 2.03 – 1.91 (m, 2H), 1.63 (dtd, *J* = 6.8, 1.2, 0.6 Hz, 3H), 1.46 – 1.13 (m, 10H), 0.97 (t, *J* = 7.9 Hz, 1H), 0.91 – 0.85 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.10,

121.70, 60.89, 36.94, 33.00, 31.85, 29.38, 29.21, 28.31, 22.66, 14.08, 13.42; IR (neat) 3342, 2954, 2926, 2873, 2856, 1457, 1454, 1257, 1039, 848, 740 cm^{-1}; HRMS (EI^+) m/z 184.1821 [(M^+) calcd for C12H24O 184.1827].

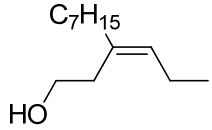
(*Z*)-3-(butan-2-ylidene) decan-1-ol (19)



19

^1H NMR (400 MHz, $CDCl_3$) δ 3.62 (t, *J* = 7.0 Hz, 2H), 2.32 (td, *J* = 7.0, 0.6 Hz, 2H), 2.07 (q, *J* = 7.5 Hz, 2H), 2.01 – 1.94 (m, 2H), 1.66 (s, 3H), 1.43 (s, 1H), 1.37 – 1.20 (m, 11H), 0.96 (t, *J* = 7.5 Hz, 3H), 0.92 – 0.84 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.04, 127.87, 61.41, 35.02, 32.31, 31.89, 29.81, 29.27, 28.76, 27.24, 22.66, 17.54, 14.10, 13.28; IR (neat) 3360, 3342, 2956, 2926, 2872, 2854, 1460, 1443, 1029, 1016, 760 cm^{-1}; HRMS (EI^+) m/z 212.2145 [(M^+) calcd for $C_{14}H_{28}O$ 212.2140].

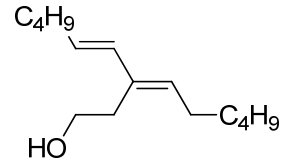
(*Z*)-3-propylidenedecan-1-ol (20)



20

^1H NMR (400 MHz, $CDCl_3$) δ 5.31 (t, *J* = 7.2 Hz, 1H), 3.64 (t, *J* = 6.8 Hz, 2H), 2.32 (t, *J* = 6.8 Hz, 2H), 2.05 (p, *J* = 7.5 Hz, 2H), 2.00 – 1.94 (m, 2H), 1.56 (s, 1H), 1.43 – 1.20 (m, 12H), 0.95 (t, *J* = 7.5 Hz, 3H), 0.92 – 0.84 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 134.39, 129.89, 60.98, 36.84, 33.30, 31.87, 29.36, 29.22, 28.35, 22.68, 21.16, 14.72, 14.11; IR (neat) 3327, 2958, 2926, 2872, 2854, 1456, 1043, 759 cm^{-1}; HRMS (EI^+) m/z 198.1978 [(M^+); calcd for $C_{13}H_{26}O$ 198.1984].

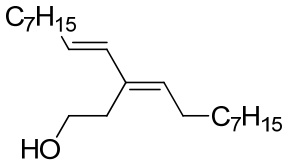
(*E*)-3-((*E*)-hex-1-en-1-yl)non-3-en-1-ol (21)



21

^1H NMR (600 MHz, $CDCl_3$) δ 5.98 (dd, *J* = 15.8, 0.7 Hz, 1H), 5.62 (dt, *J* = 15.7, 6.9 Hz, 1H), 5.51 (t, *J* = 7.4 Hz, 1H), 3.66 (dd, *J* = 12.9, 6.9 Hz, 2H), 2.54 (t, *J* = 6.9 Hz, 2H), 2.13 (dd, *J* = 14.9, 7.4 Hz, 2H), 2.08 (dt, *J* = 7.9, 4.0 Hz, 2H), 1.42 – 1.24 (m, 9H), 0.91 – 0.87 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.56, 133.38, 133.28, 127.96, 61.73, 32.56, 31.77, 31.59, 30.24, 29.53, 28.23, 22.57, 22.26, 14.03, 13.95; IR (neat) 3327, 3311, 3018, 2956, 2926, 2872, 2856, 1463, 1454, 1377, 1041, 1020, 964 cm^{-1}; HRMS (EI^+) m/z 224.2144 [(M+); calcd for $C_{15}H_{28}O$ 224.2140].

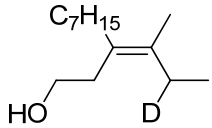
(*E*)-3-((*E*)-non-1-en-1-yl)dodec-3-en-1-ol (22)



22

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.97 (dd, *J* = 15.8, 0.7 Hz, 1H), 5.61 (dt, *J* = 15.7, 6.9 Hz, 1H), 5.50 (t, *J* = 7.4 Hz, 1H), 3.66 (t, *J* = 6.9 Hz, 2H), 2.54 (t, *J* = 6.9 Hz, 2H), 2.18 – 2.03 (m, 4H), 1.45 – 1.18 (m, 23H), 0.88 (t, *J* = 6.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.55, 133.36, 133.25, 128.00, 61.71, 32.91, 31.87, 31.81, 30.24, 29.87, 29.62, 29.54, 29.51, 29.42, 29.35, 29.27, 29.20, 29.17, 28.27, 22.65, 14.08; IR (neat) 3315, 3016, 2954, 2924, 2852, 1465, 1377, 1041, 1020, 964, 721 cm^{-1}; HRMS (EI$^+$) m/z 308.3085 [(M$^+$); cacld for $C_{21}H_{40}O$ 308.3079].

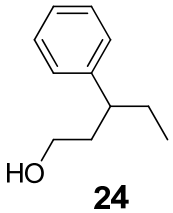
(*Z*)-3-(butan-2-ylidene)undecan-1-ol (23)[1]



23

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.62 (t, *J* = 7.0 Hz, 2H), 2.32 (td, *J* = 7.0, 0.7 Hz, 2H), 2.08 – 2.00 (m, 1H), 2.00 – 1.95 (m, 2H), 1.66 (s, 3H), 1.38 – 1.20 (m, 11H), 0.95 (d, *J* = 7.5 Hz, 3H), 0.92 – 0.85 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.01, 127.86, 61.41, 35.02, 32.30, 31.88, 29.81, 29.69, 29.27, 28.76, 22.66, 17.52, 14.10, 13.18;IR (neat) 3361, 3344, 2956, 2926, 2872, 2854, 1456, 1435, 1377, 1043, 1016, 740 cm^{-1}; HRMS (EI$^+$) m/z 213.2208 [(M$^+$) cacld for $C_{24}H_{27}DO$ 213.2203].

3-phenylpentan-1-ol (24)[2]

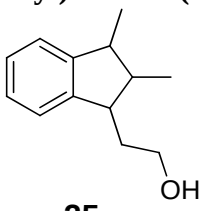


24

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.26 (m, 2H), 7.21 – 7.13 (m, 3H), 3.59 – 3.39 (m, 2H), 2.59 (tt, *J* = 10.1, 5.2 Hz, 1H), 2.00 – 1.91 (m, 1H), 1.80 (dddd, *J* = 13.7, 10.0, 6.5, 5.4 Hz, 1H), 1.74 – 1.57 (m, 2H), 1.11 (t, *J* = 5.3 Hz, 1H), 0.78 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.95, 128.39, 127.67, 126.14, 61.27, 44.29, 39.26, 29.79, 12.08.

[1] 97% Et_3SiD from Aldrich was used, >95% of compound was found to be labeled according to HRMS
[2] Known compound, see *J. Org. Chem*, **1992**, V57, 1237-1241

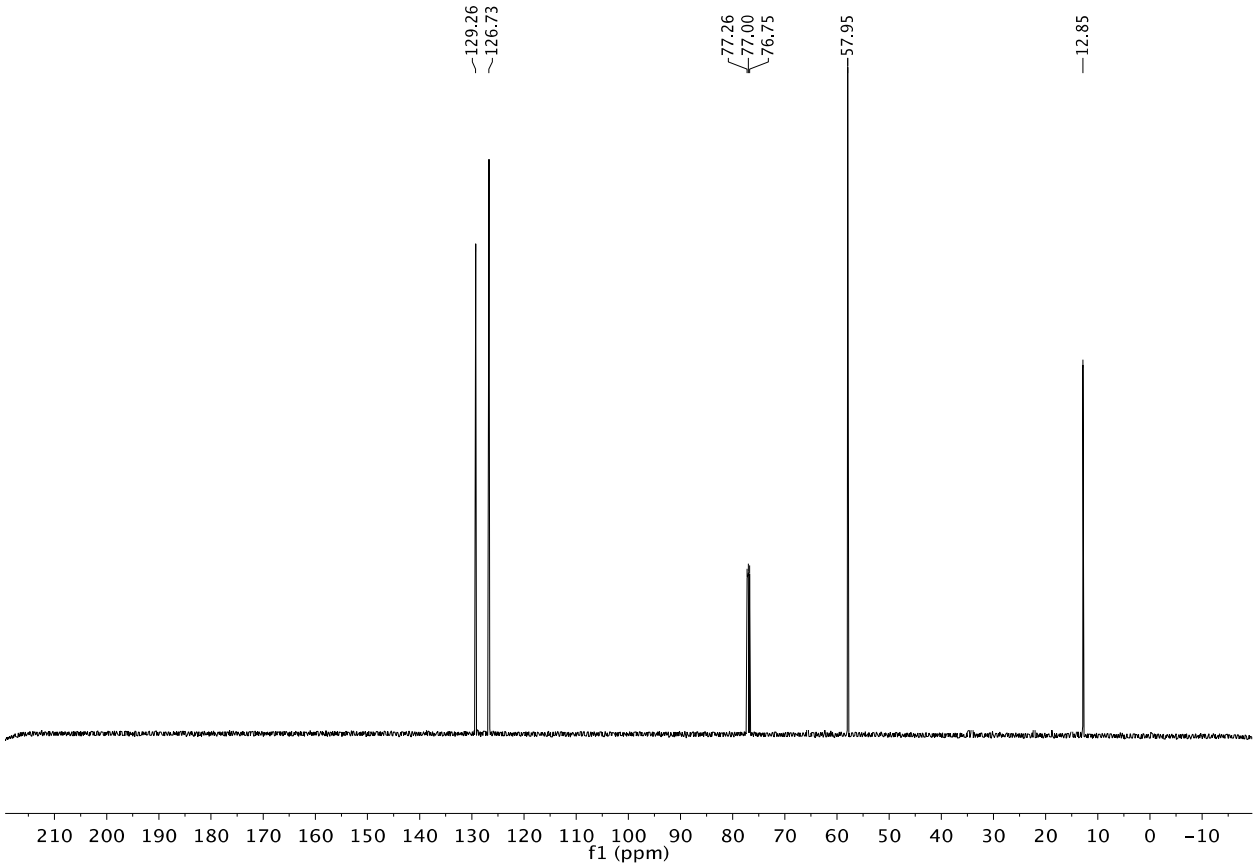
2-(2,3-dimethyl-2,3-dihydro-1H-inden-1-yl)ethanol (25)

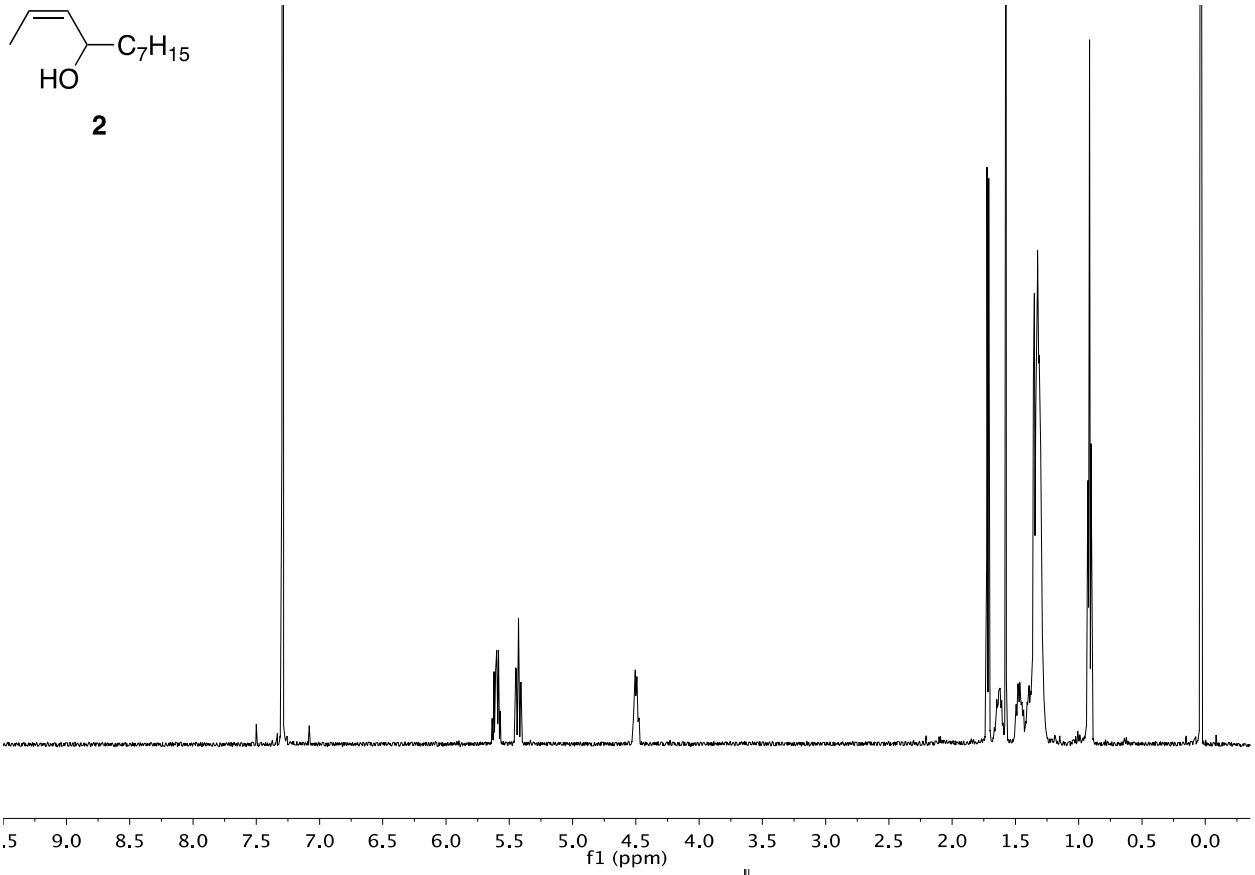


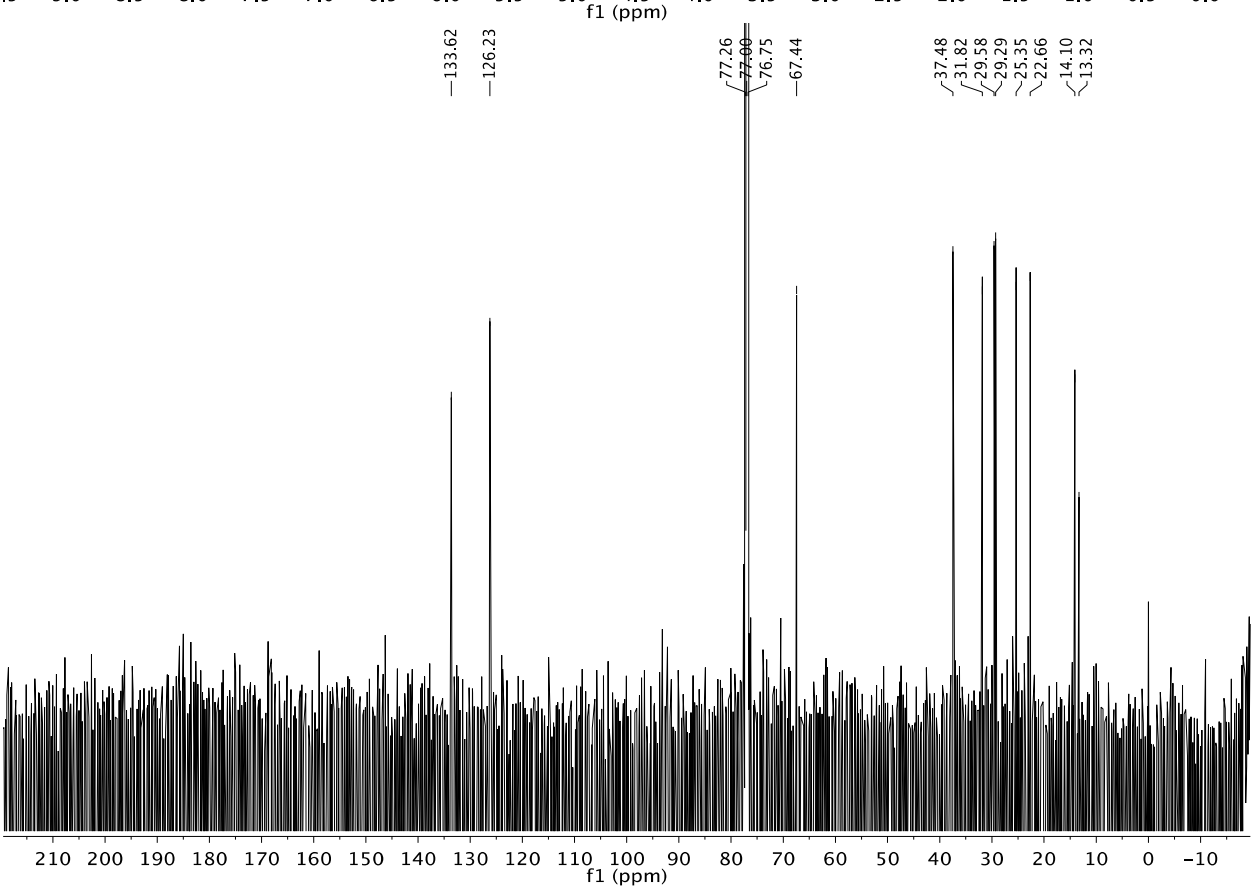
25

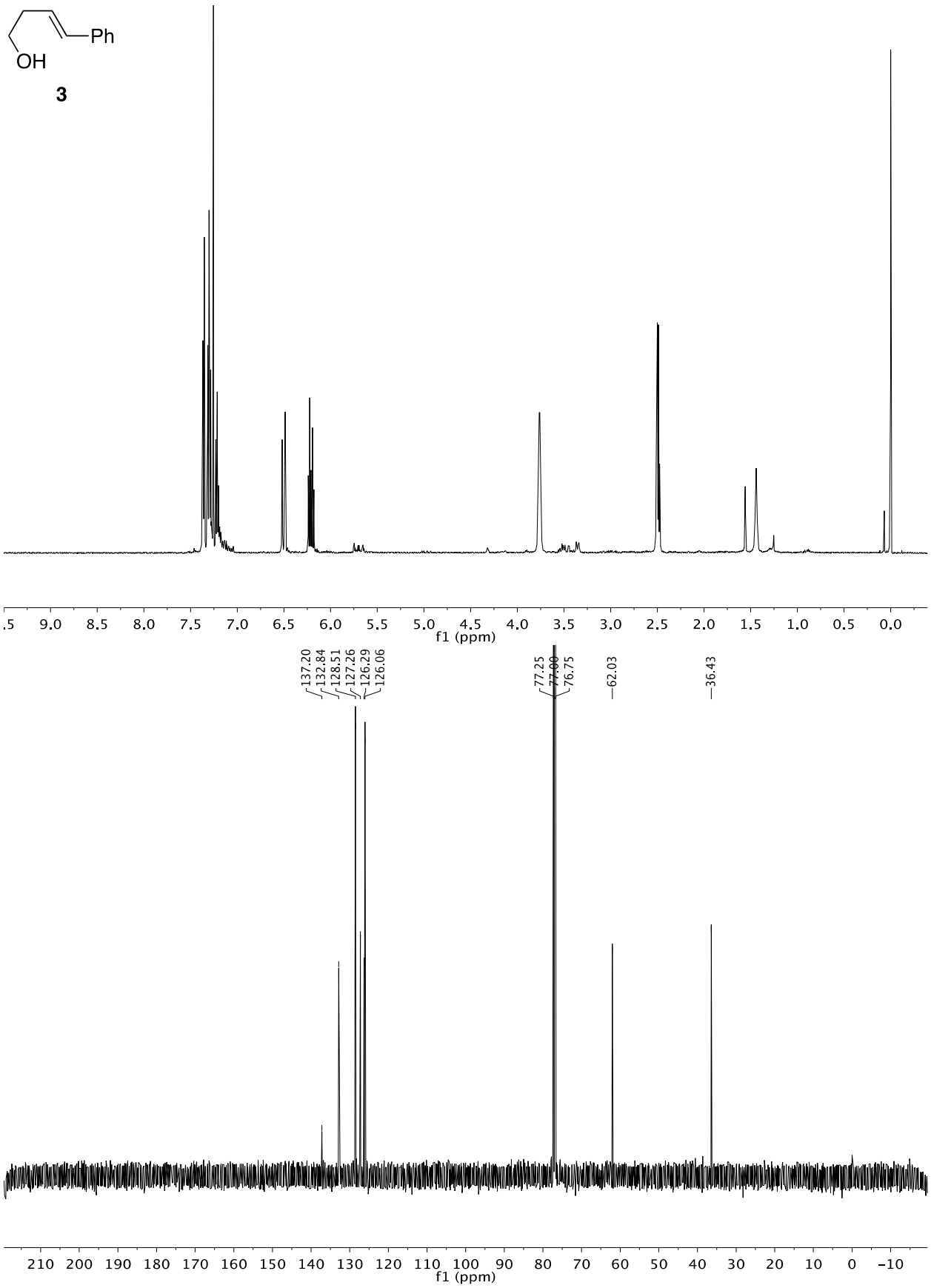
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 – 7.11 (m, 4H), 3.85 (td, *J* = 7.2, 4.6 Hz, 2H), 2.74 (dt, *J* = 9.3, 5.9 Hz, 1H), 2.63 (tt, *J* = 13.6, 6.8 Hz, 1H), 2.14 (dtd, *J* = 13.9, 7.3, 5.6 Hz, 1H), 1.92 (dtd, *J* = 13.8, 6.7, 3.2 Hz, 1H), 1.68 – 1.56 (m, 1H), 1.35 (q, *J* = 5.1 Hz, 1H), 1.30 (dd, *J* = 6.8, 2.4 Hz, 3H), 1.22 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.81, 146.11, 126.44, 126.35, 123.00, 122.92, 61.42, 50.09, 47.86, 45.97, 36.34, 17.75, 17.56; IR (neat) 3338, 3330, 3024, 2962, 2931, 2872, 1043, 1029, 962, 759, 696 cm^{-1}; HRMS (EI^+) m/z 190.1362 [(M^+) calcd for $C_{13}H_{18}O$ 190.1358].

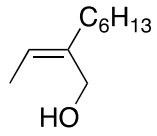
1











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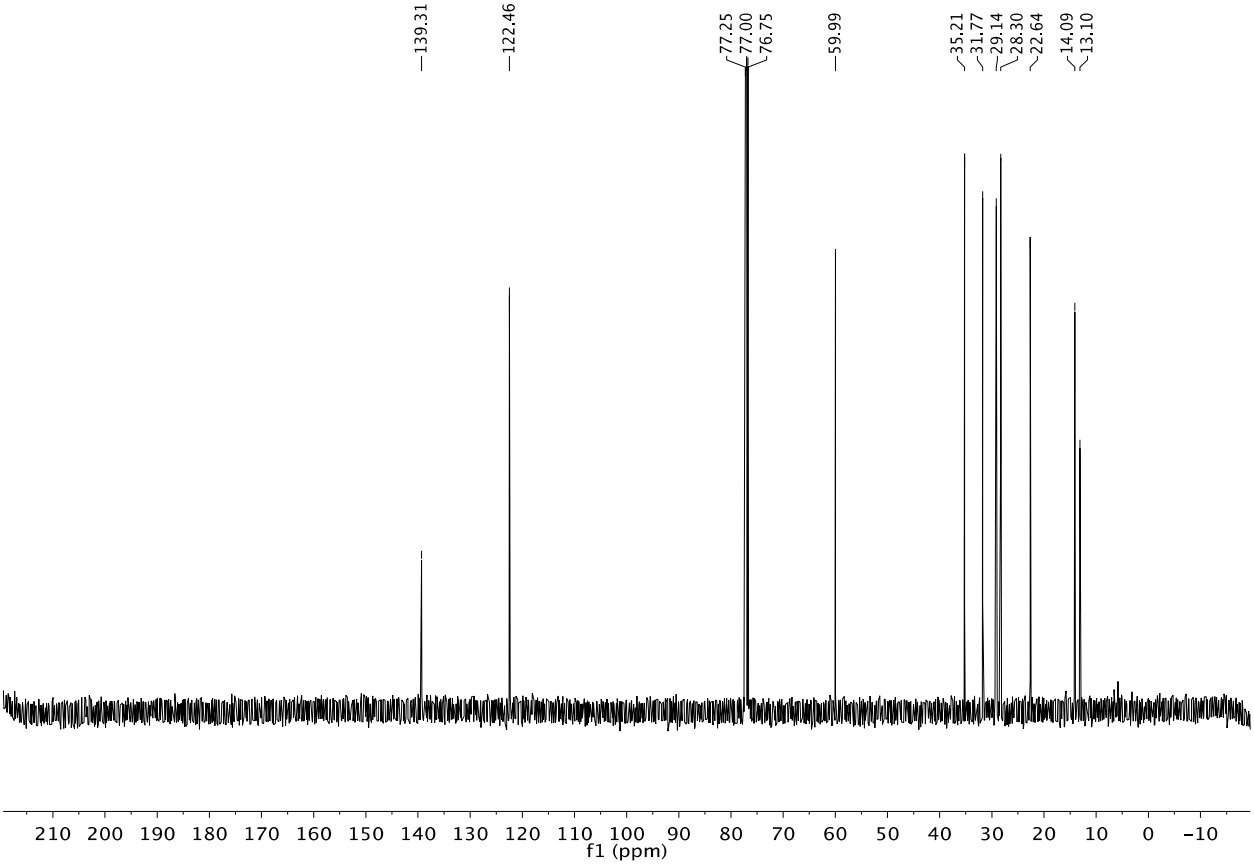

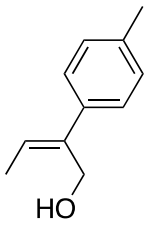

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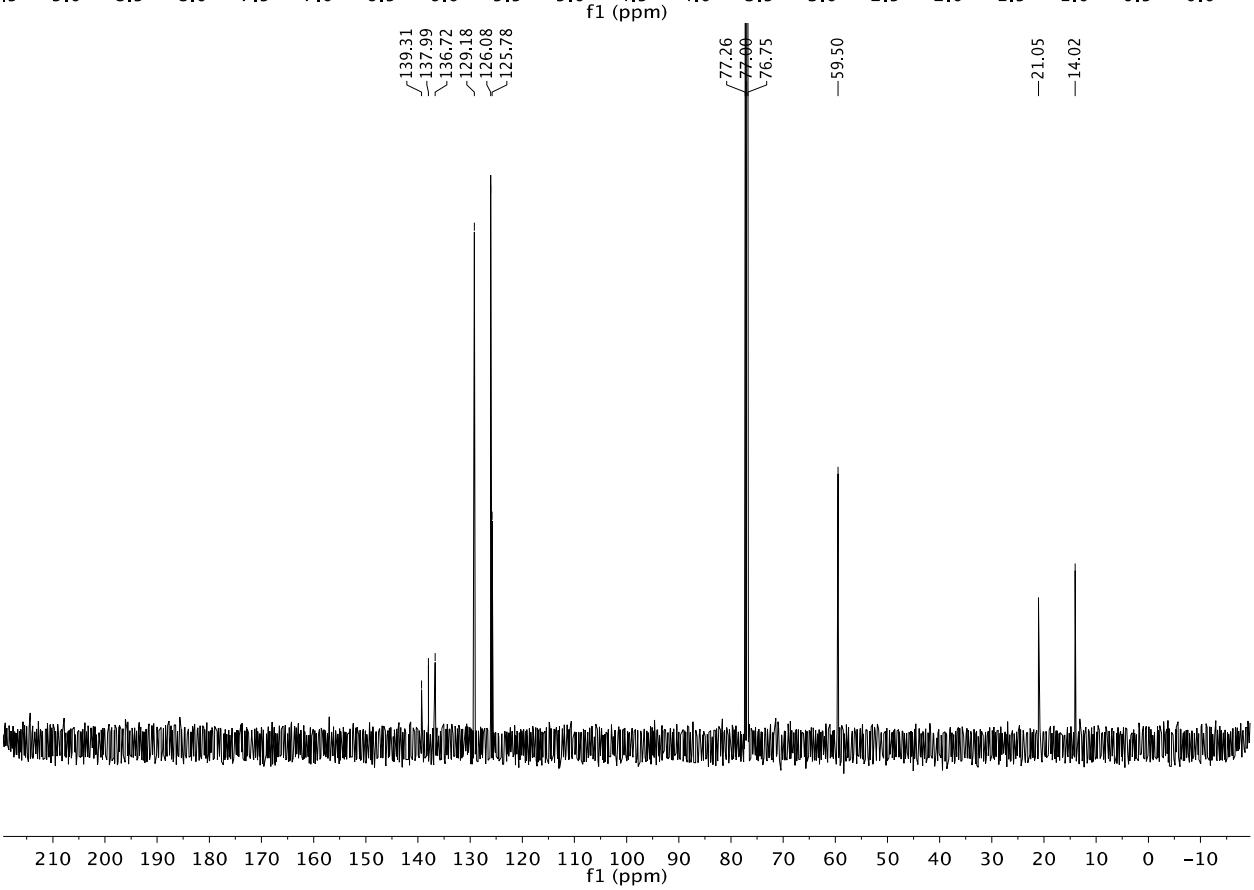
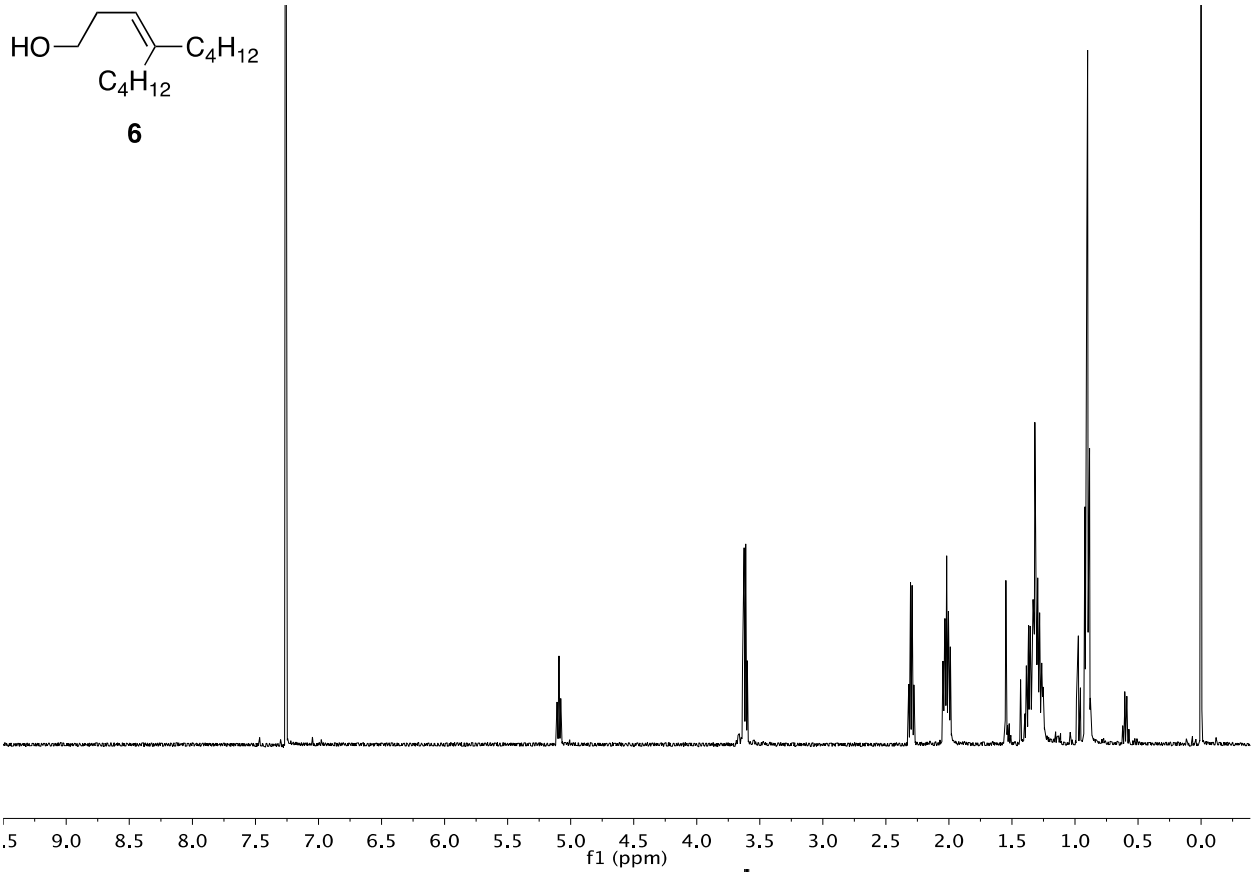
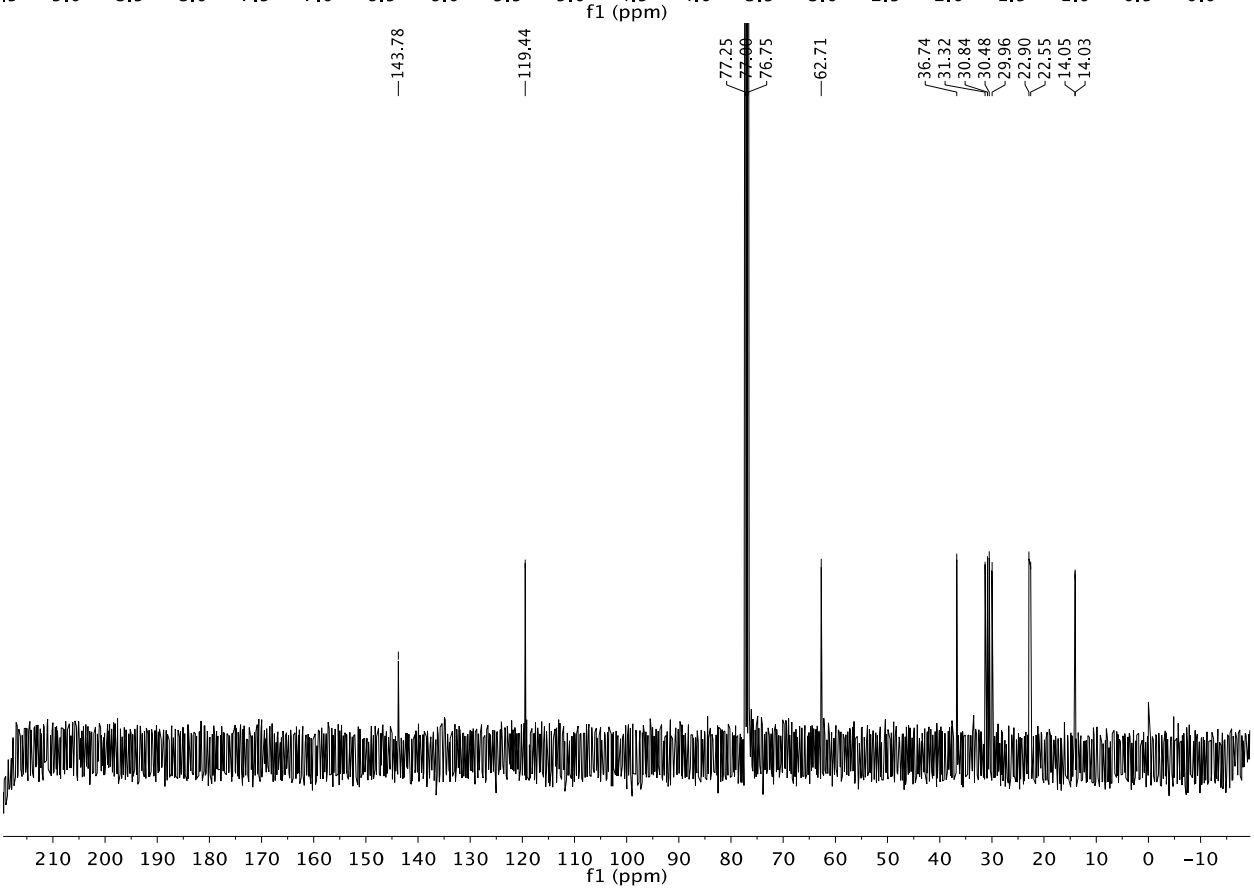
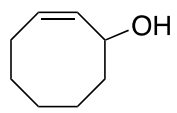
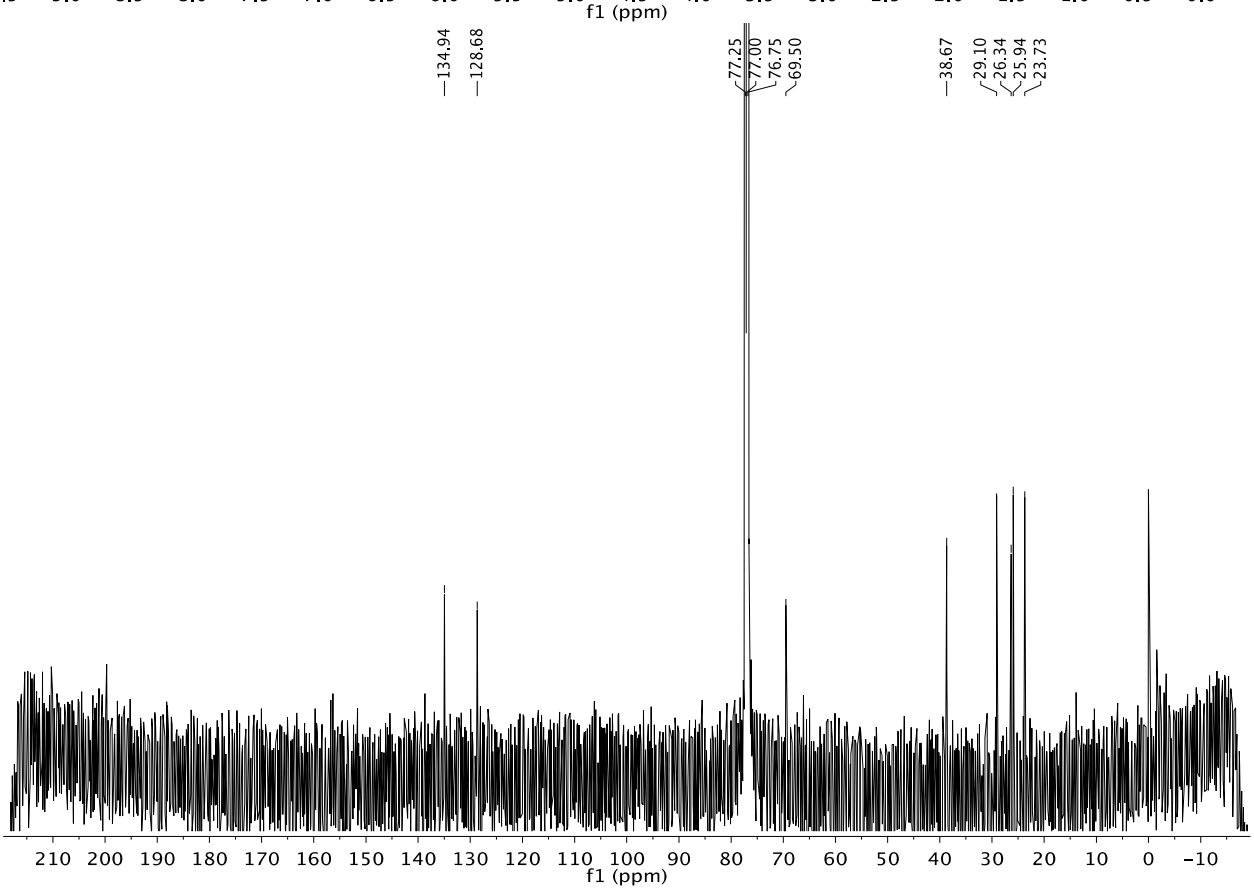


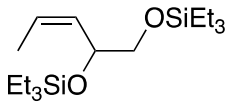




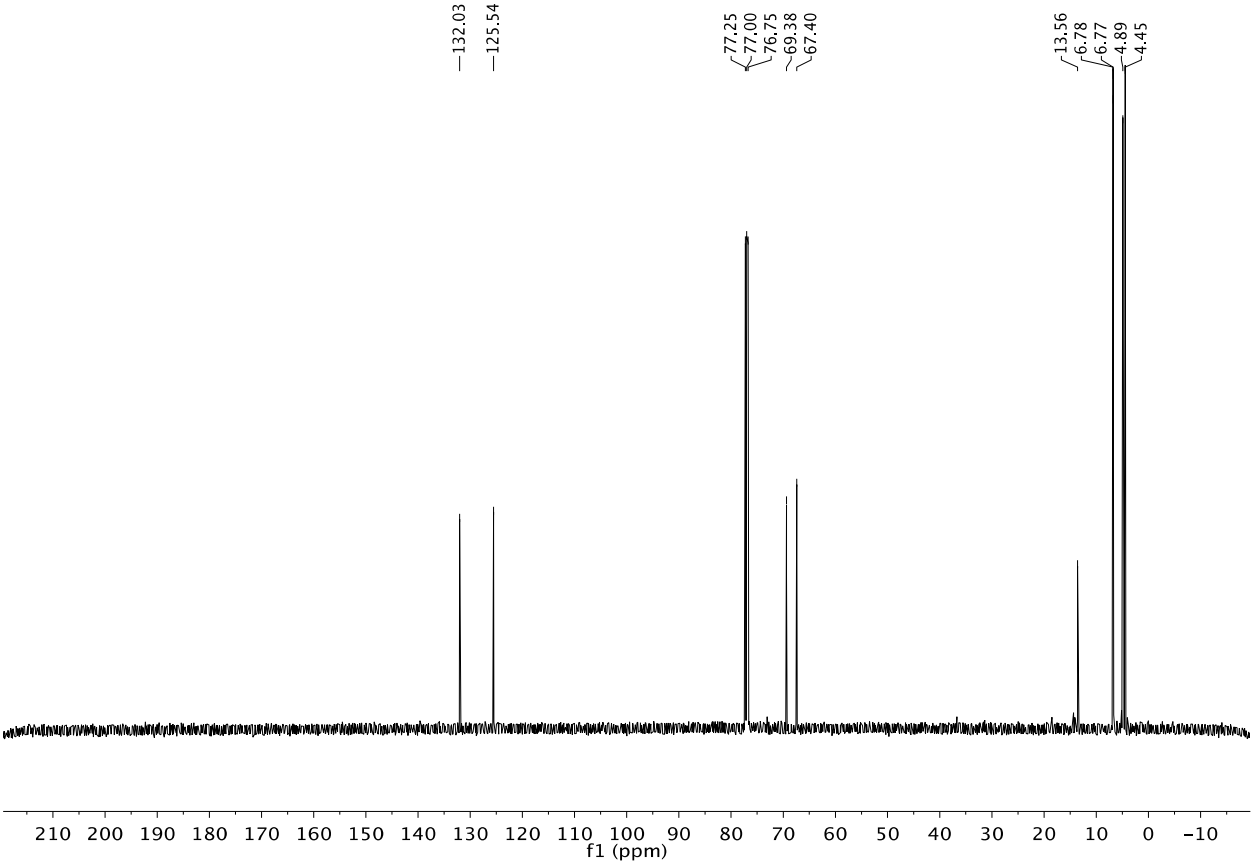


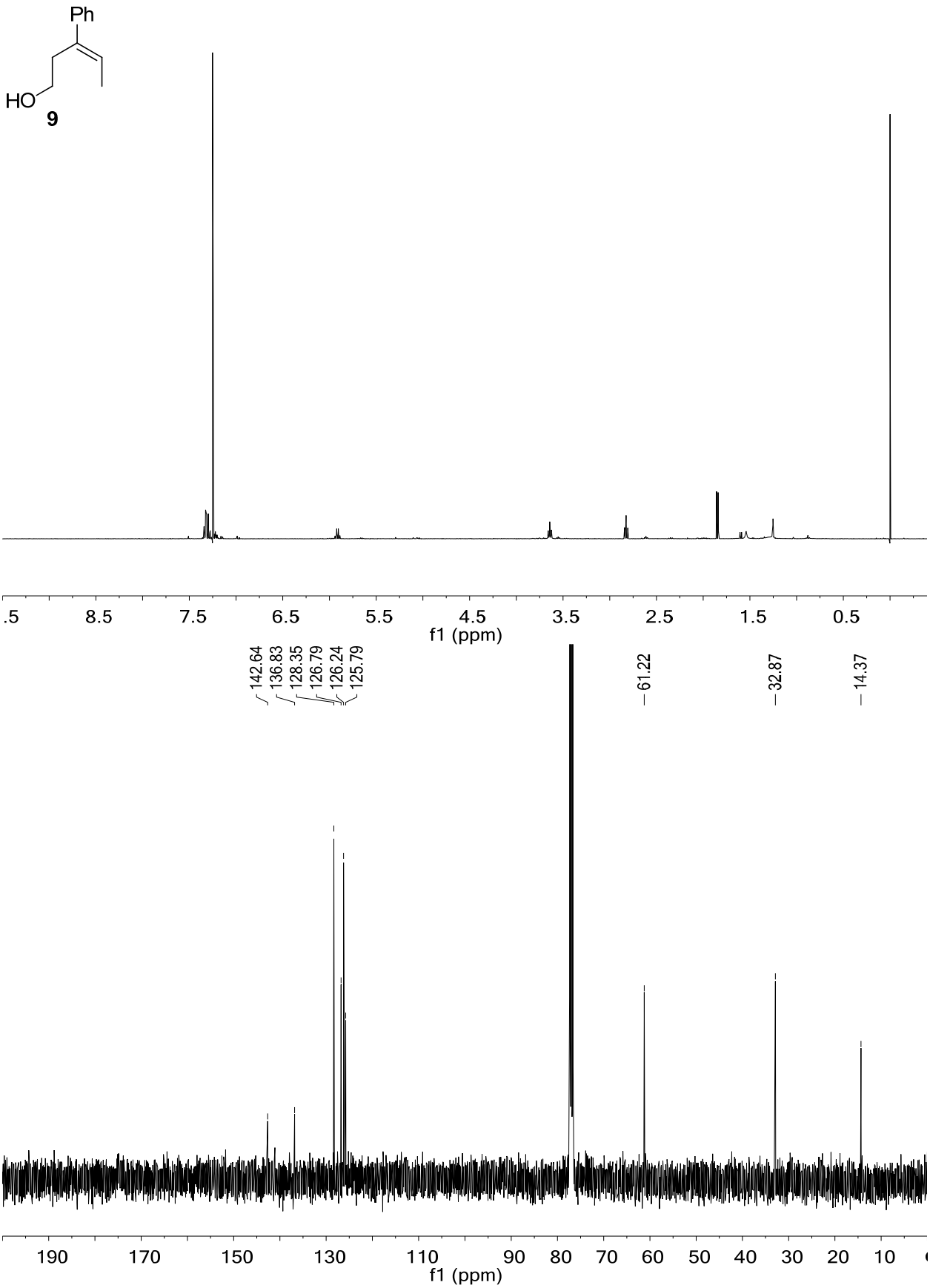
7

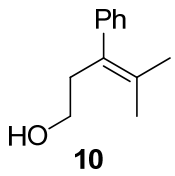




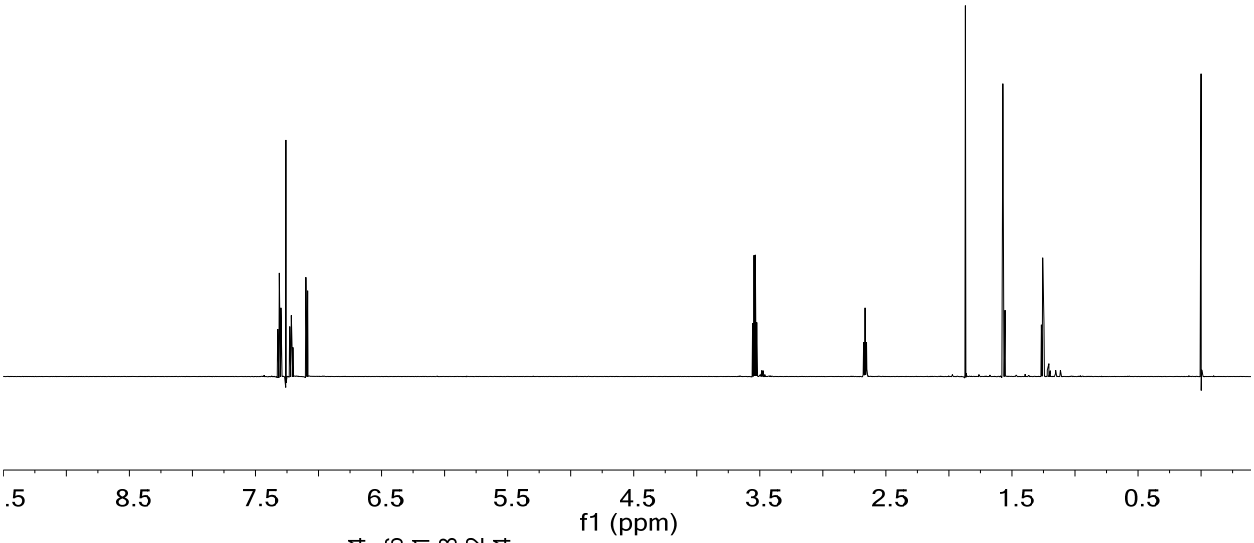
8

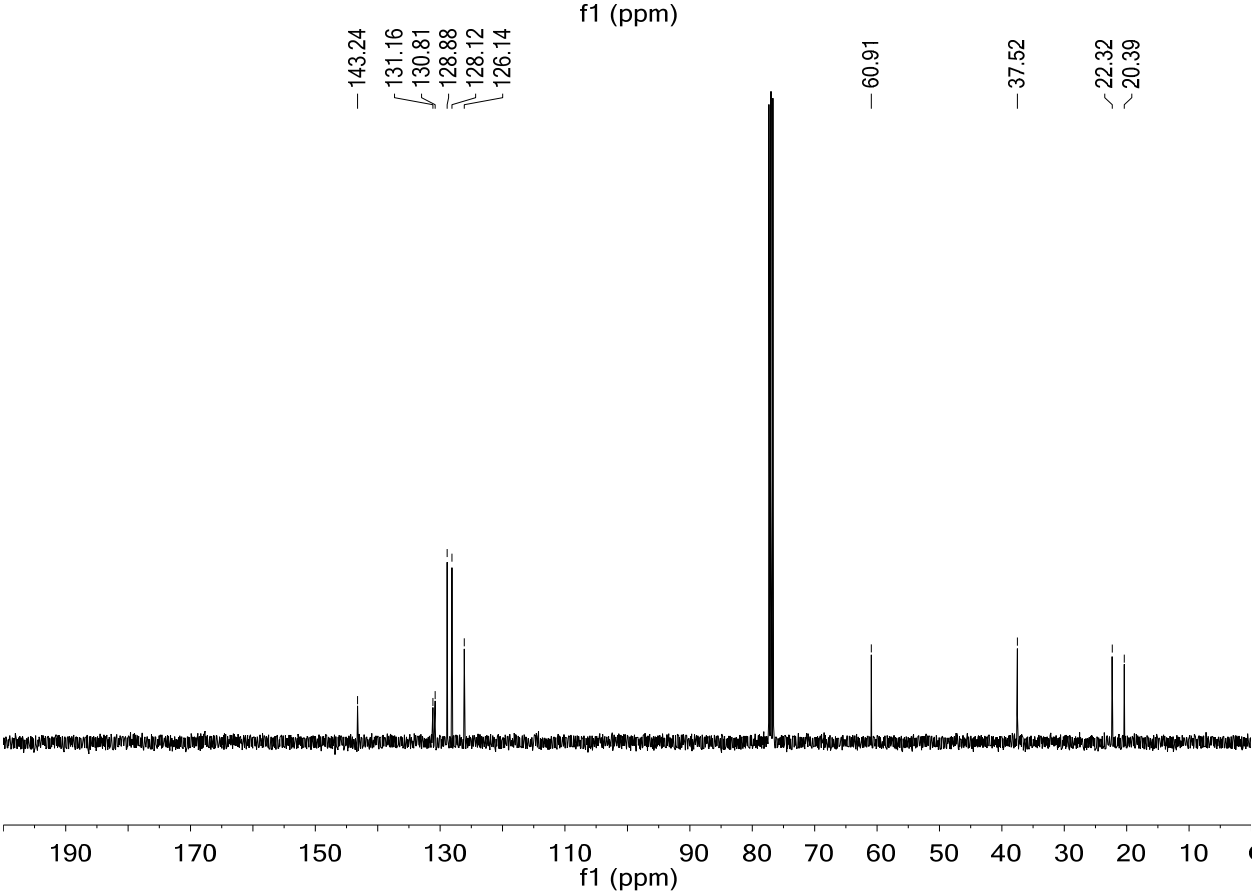


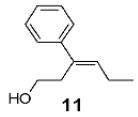




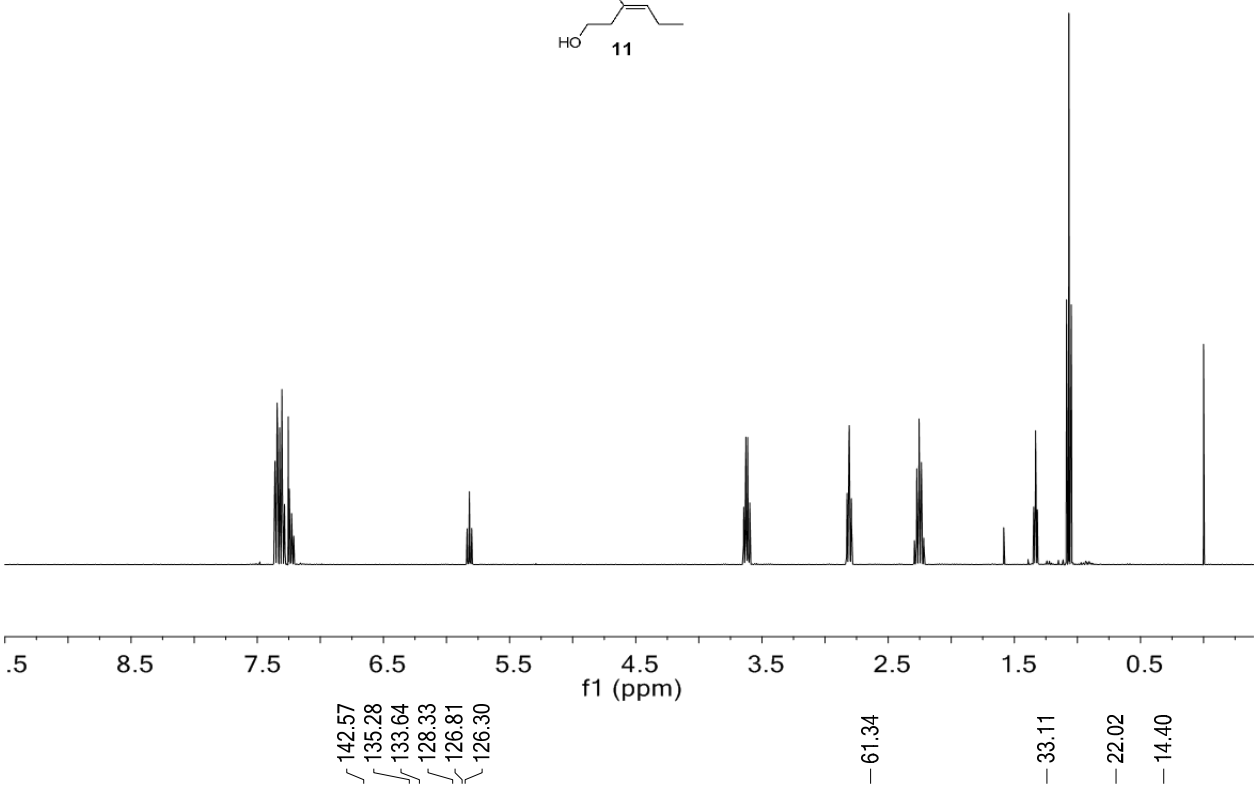
10

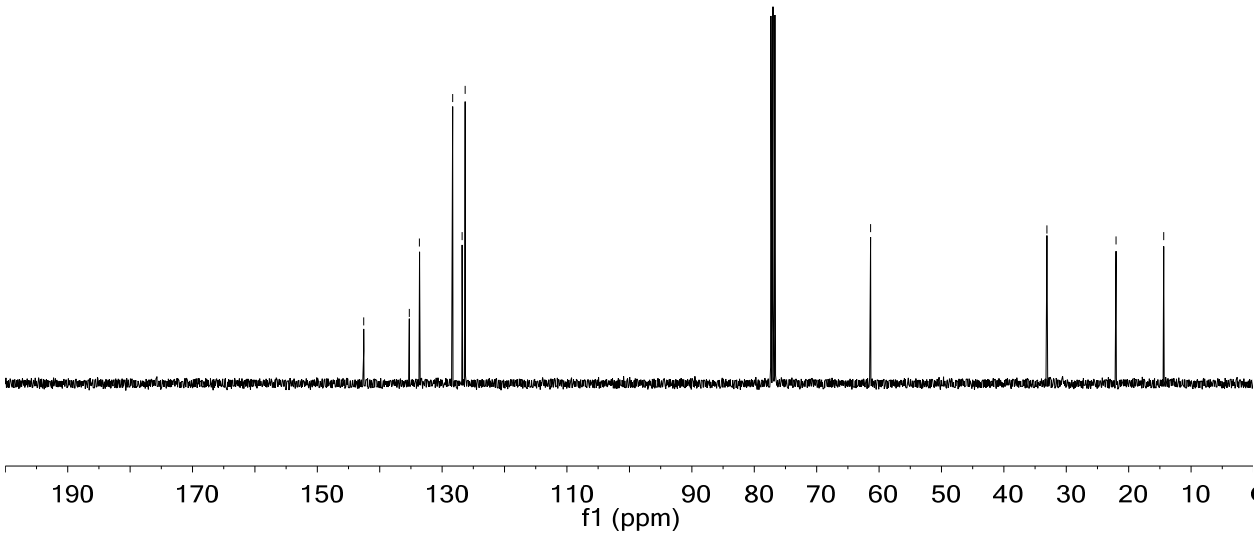


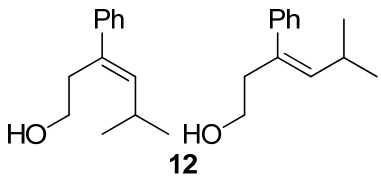




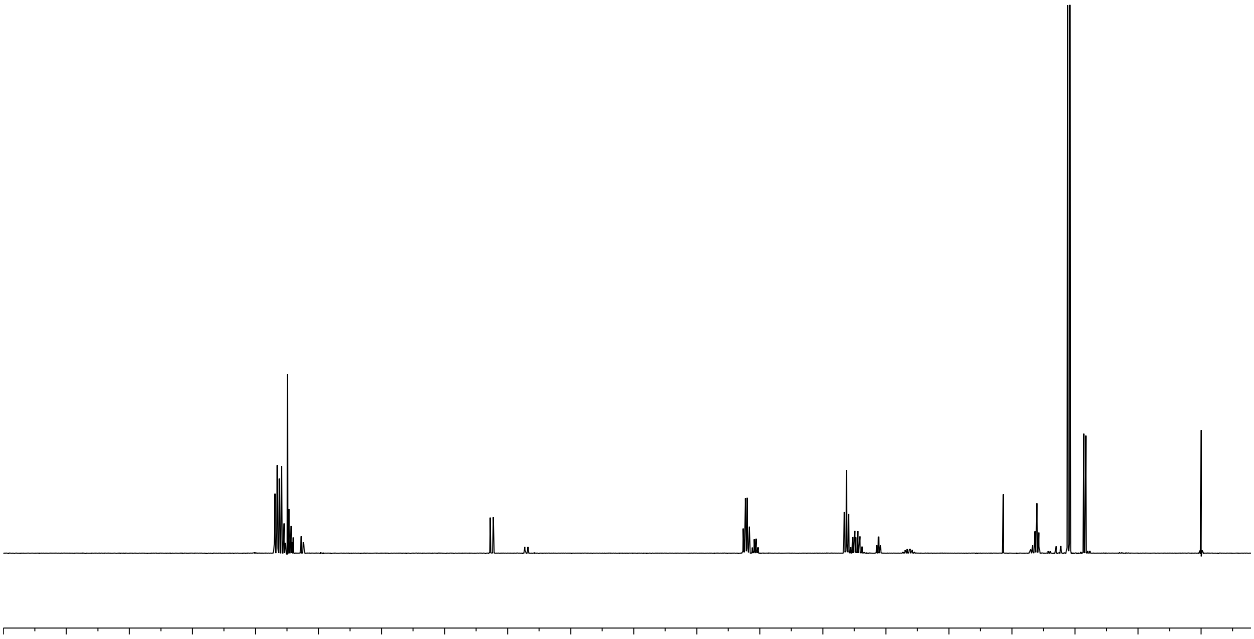
11

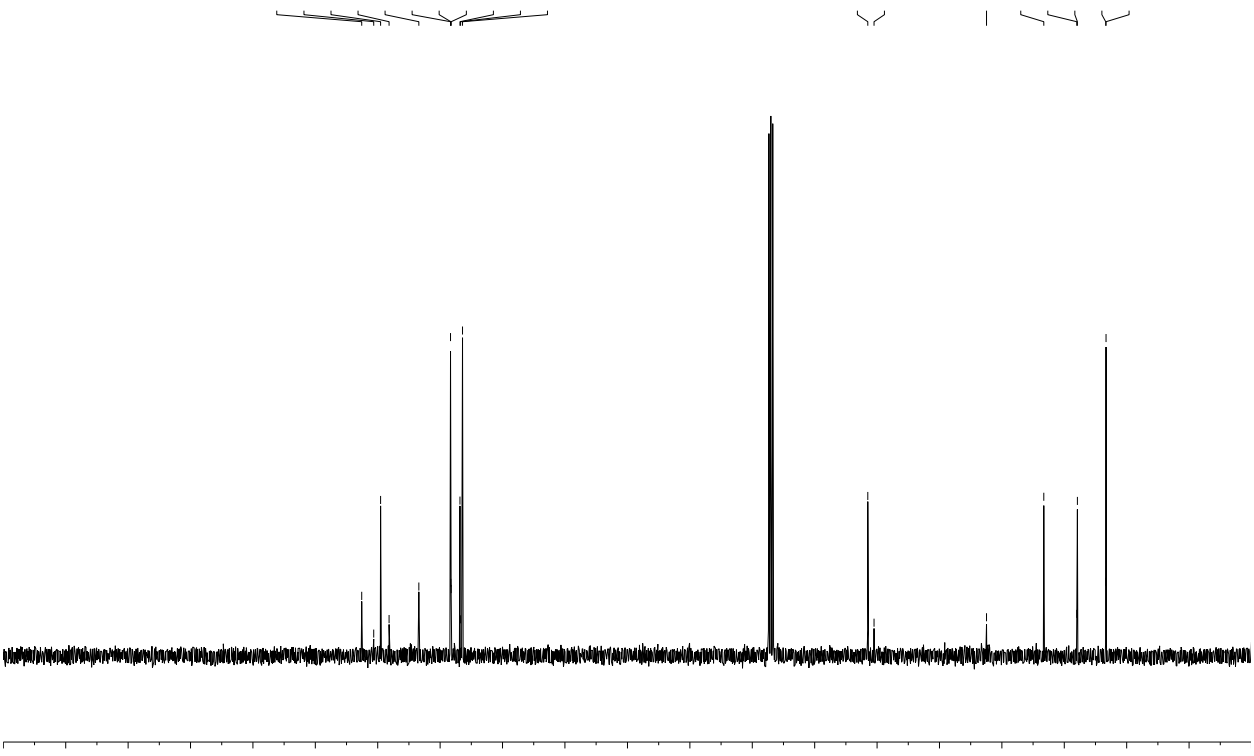


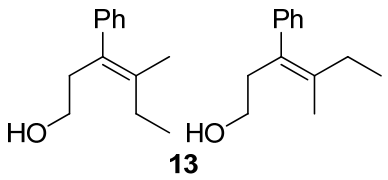




12







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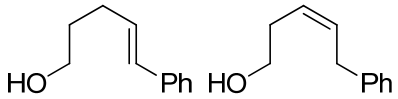

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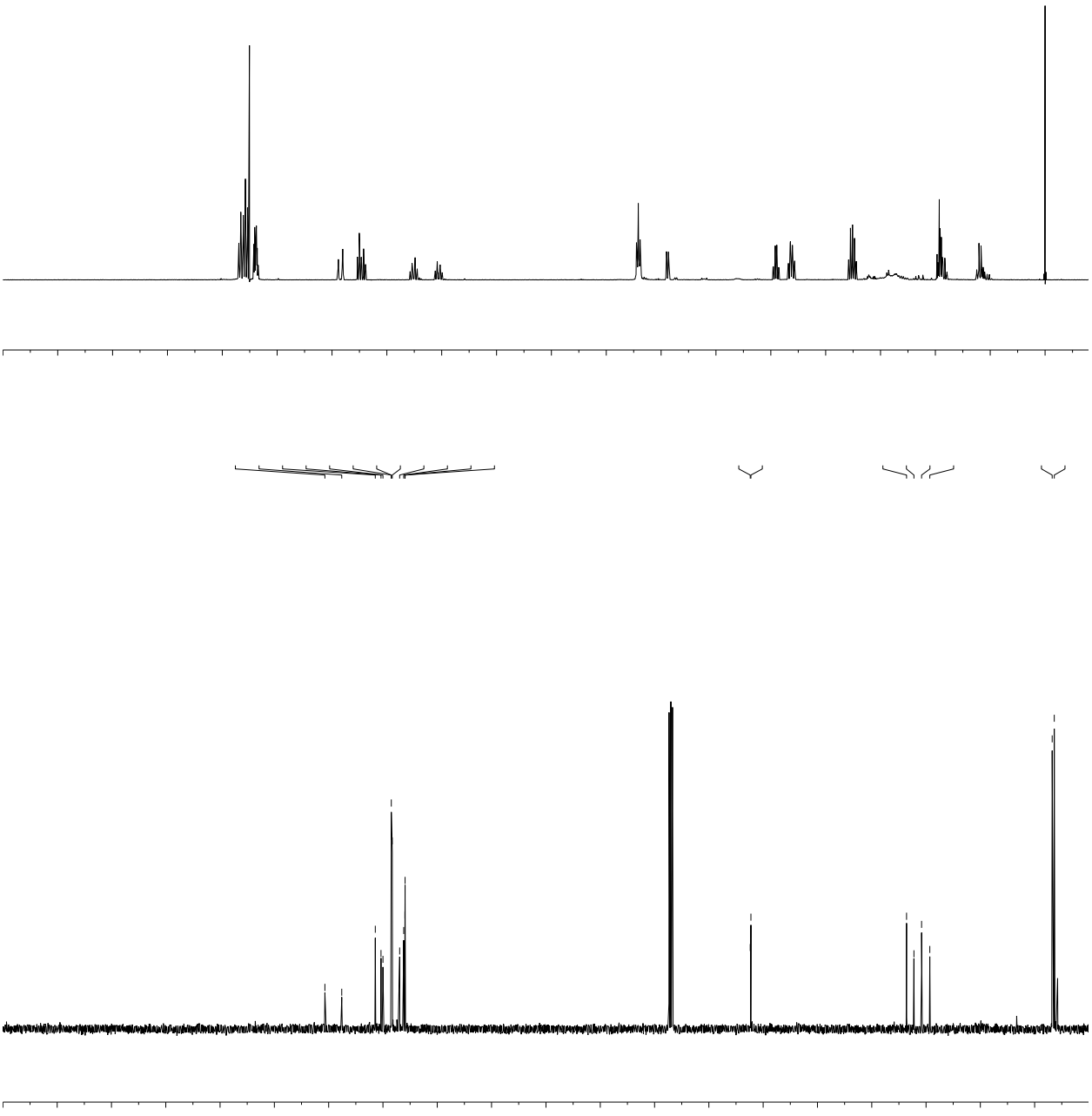
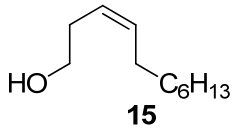
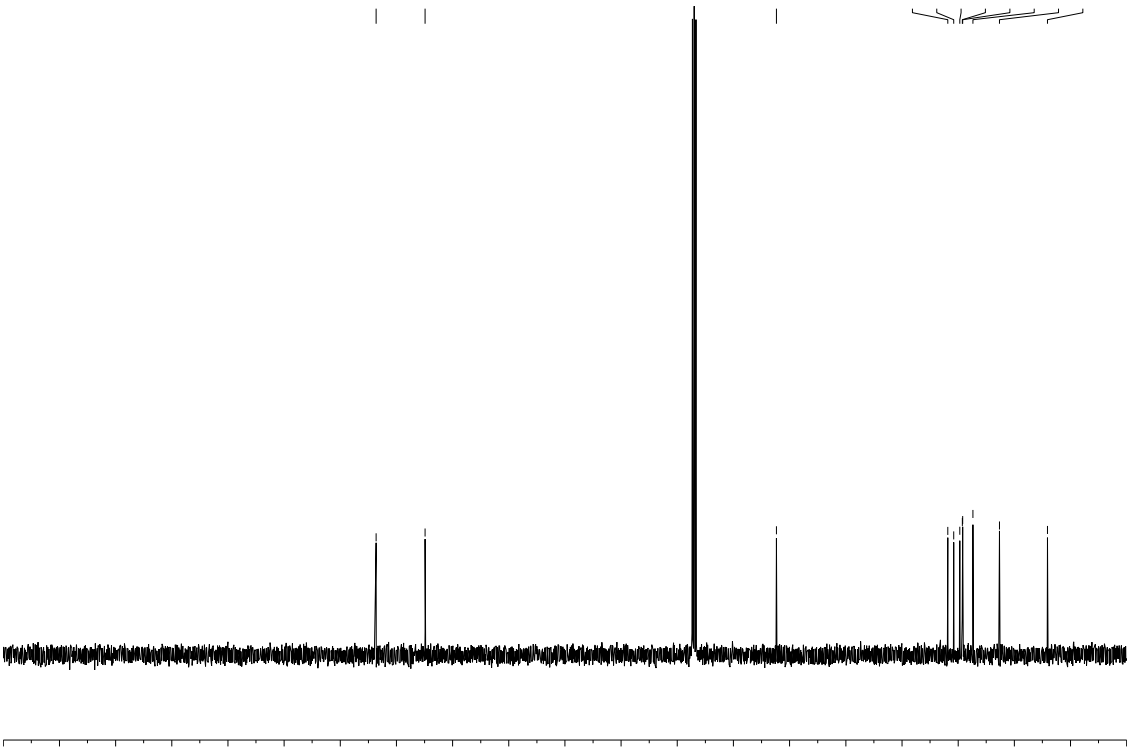
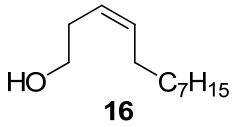




15





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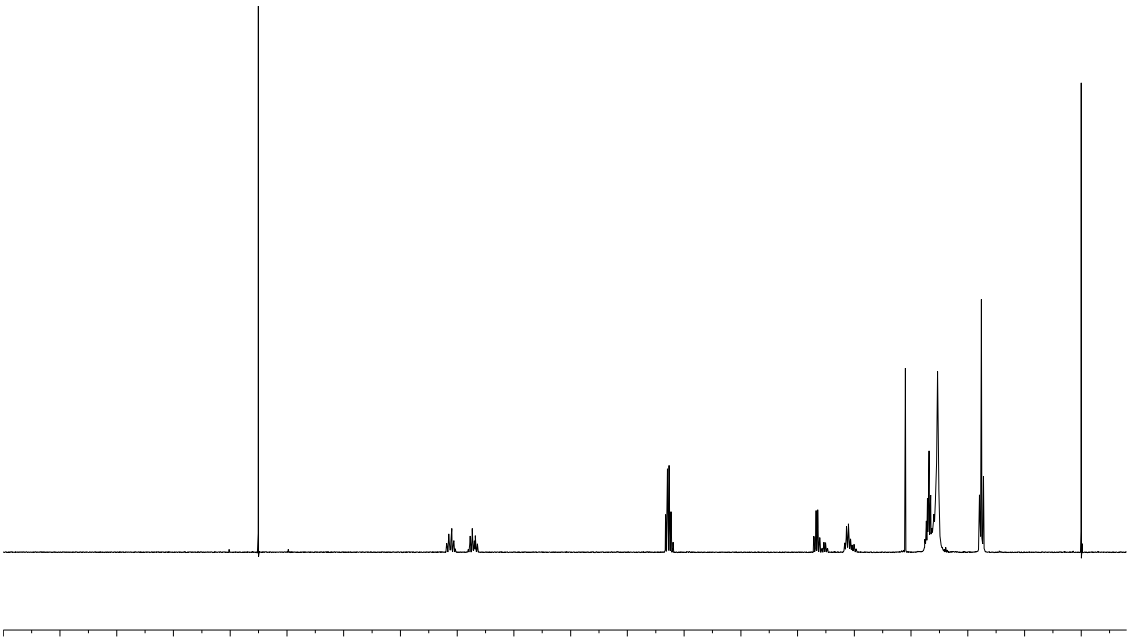

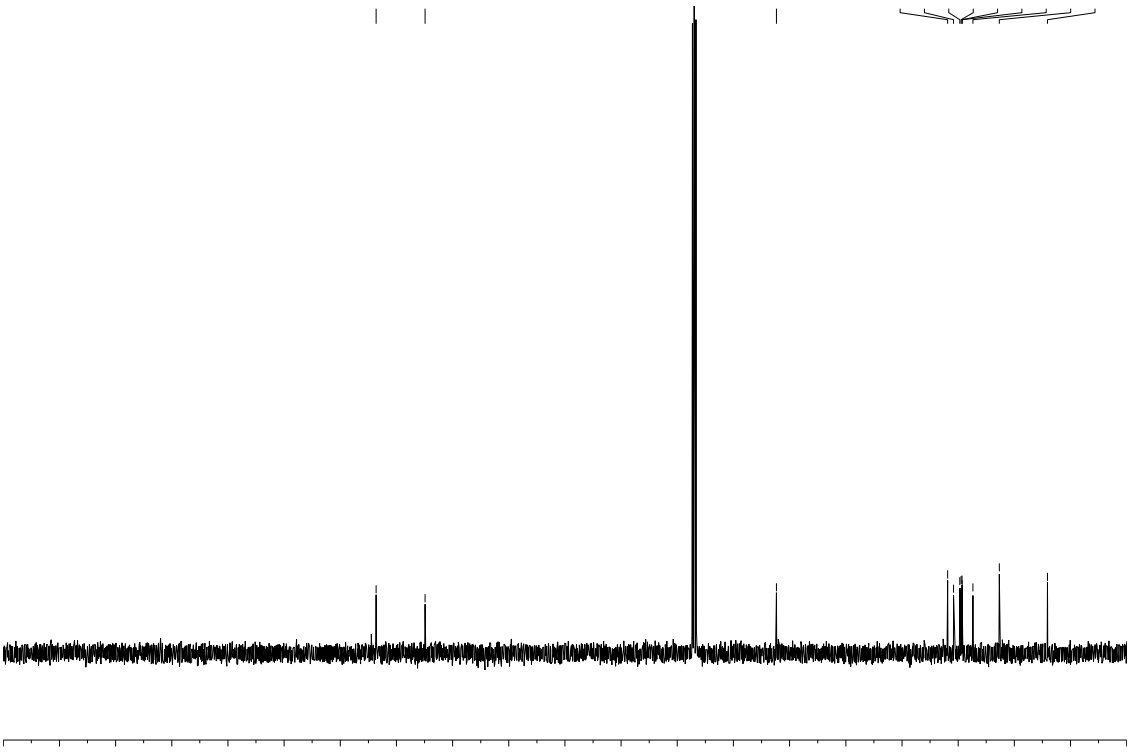

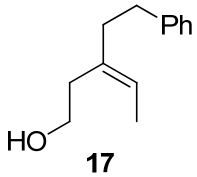

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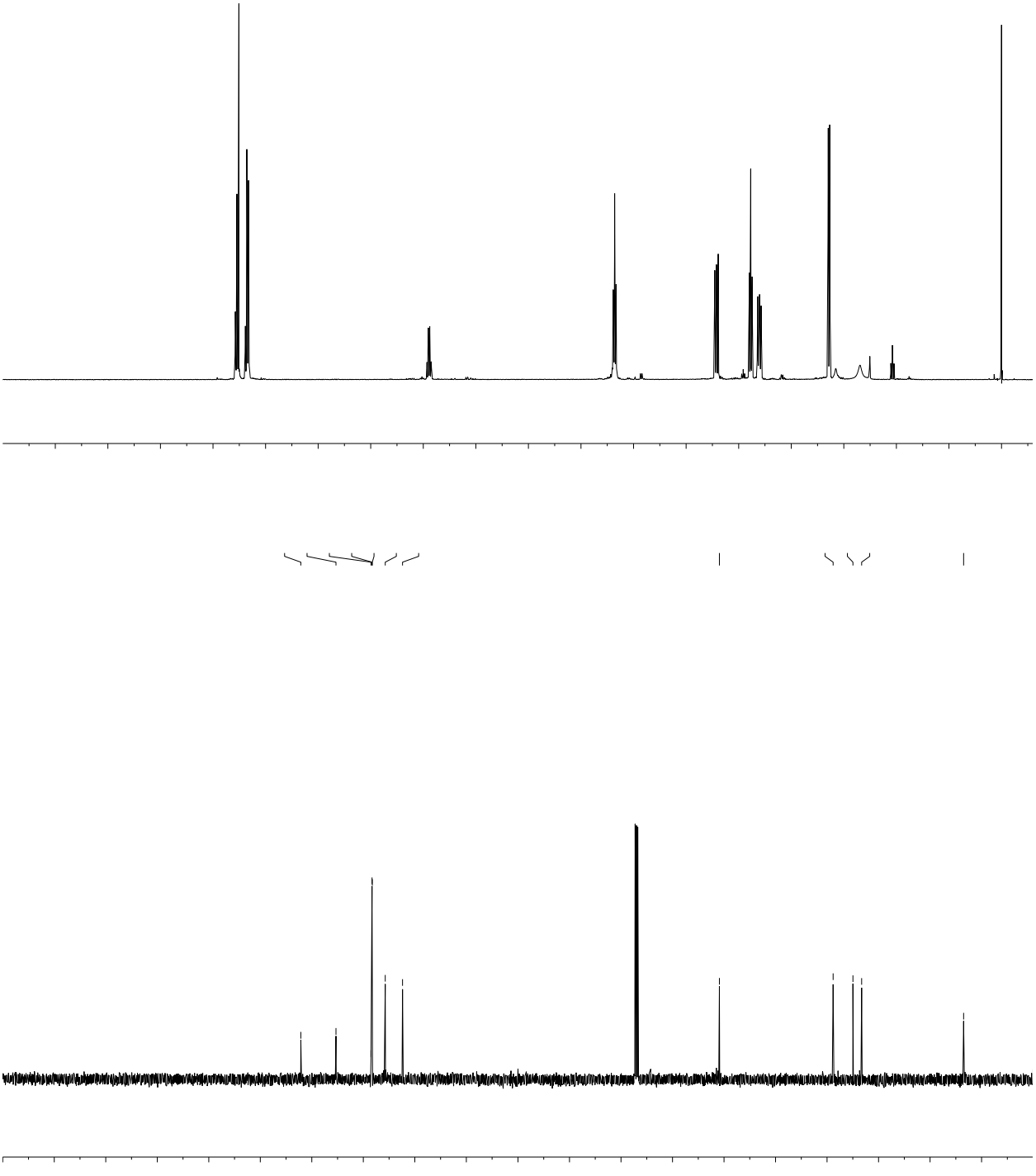

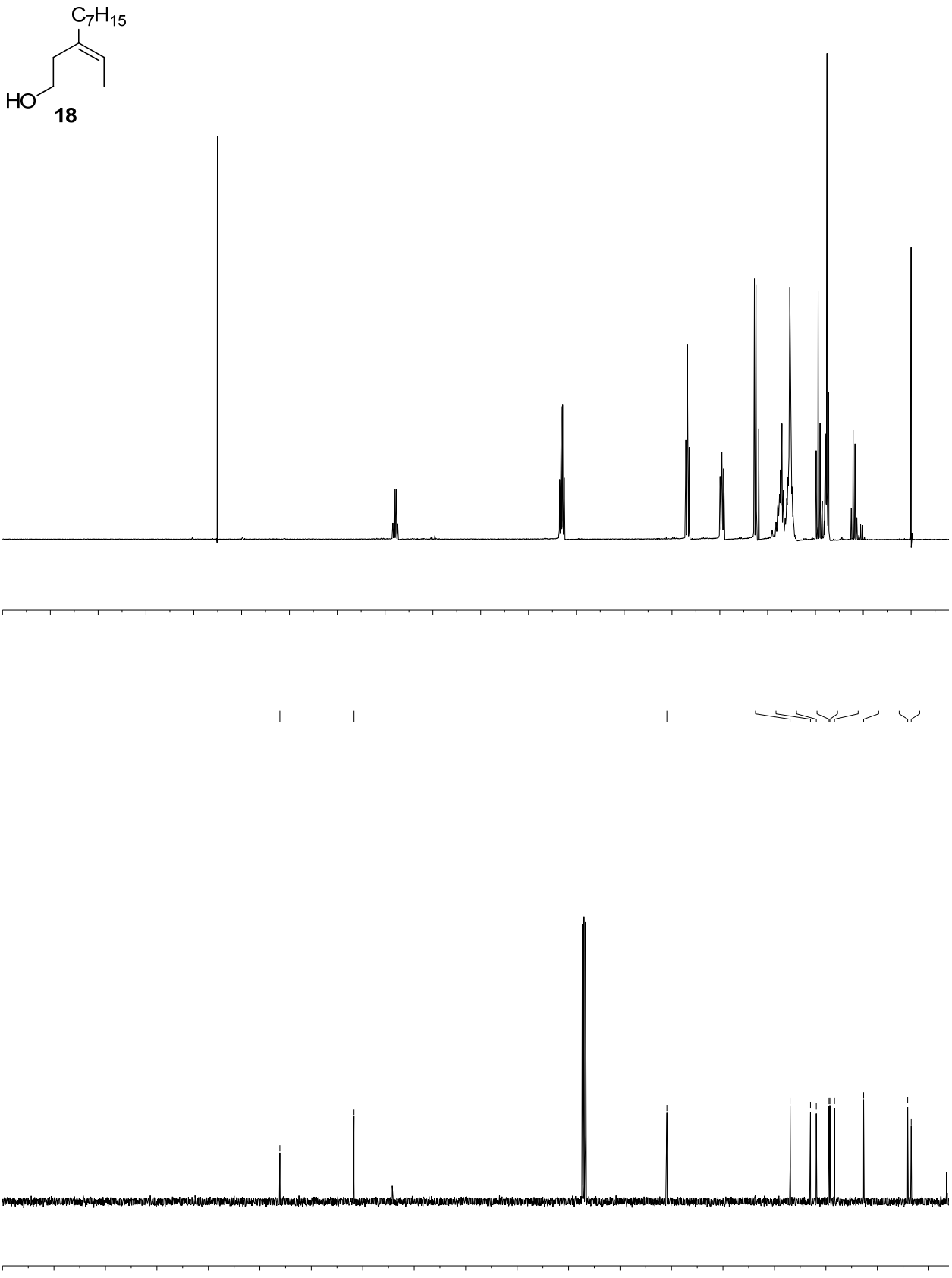

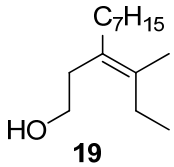

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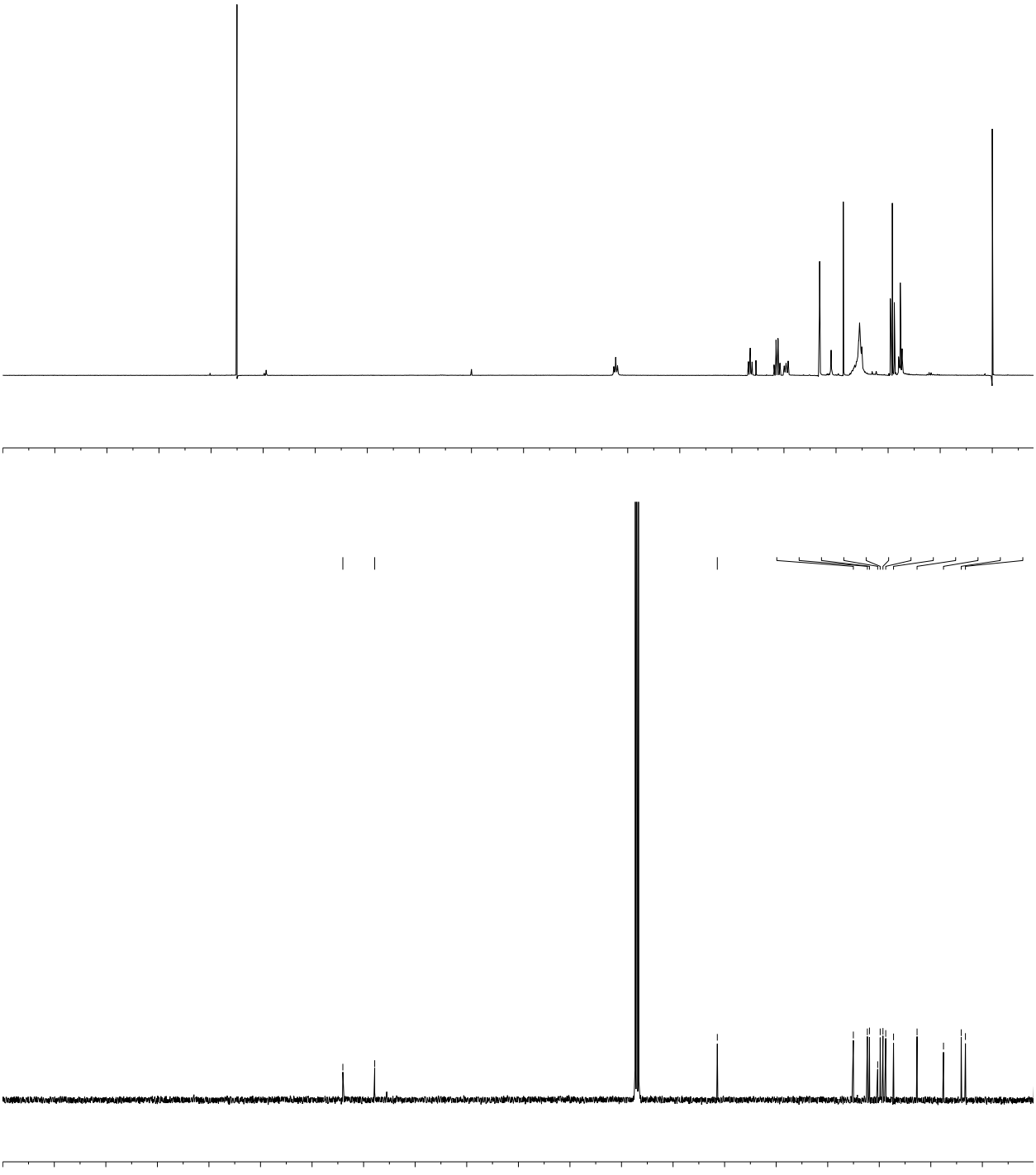

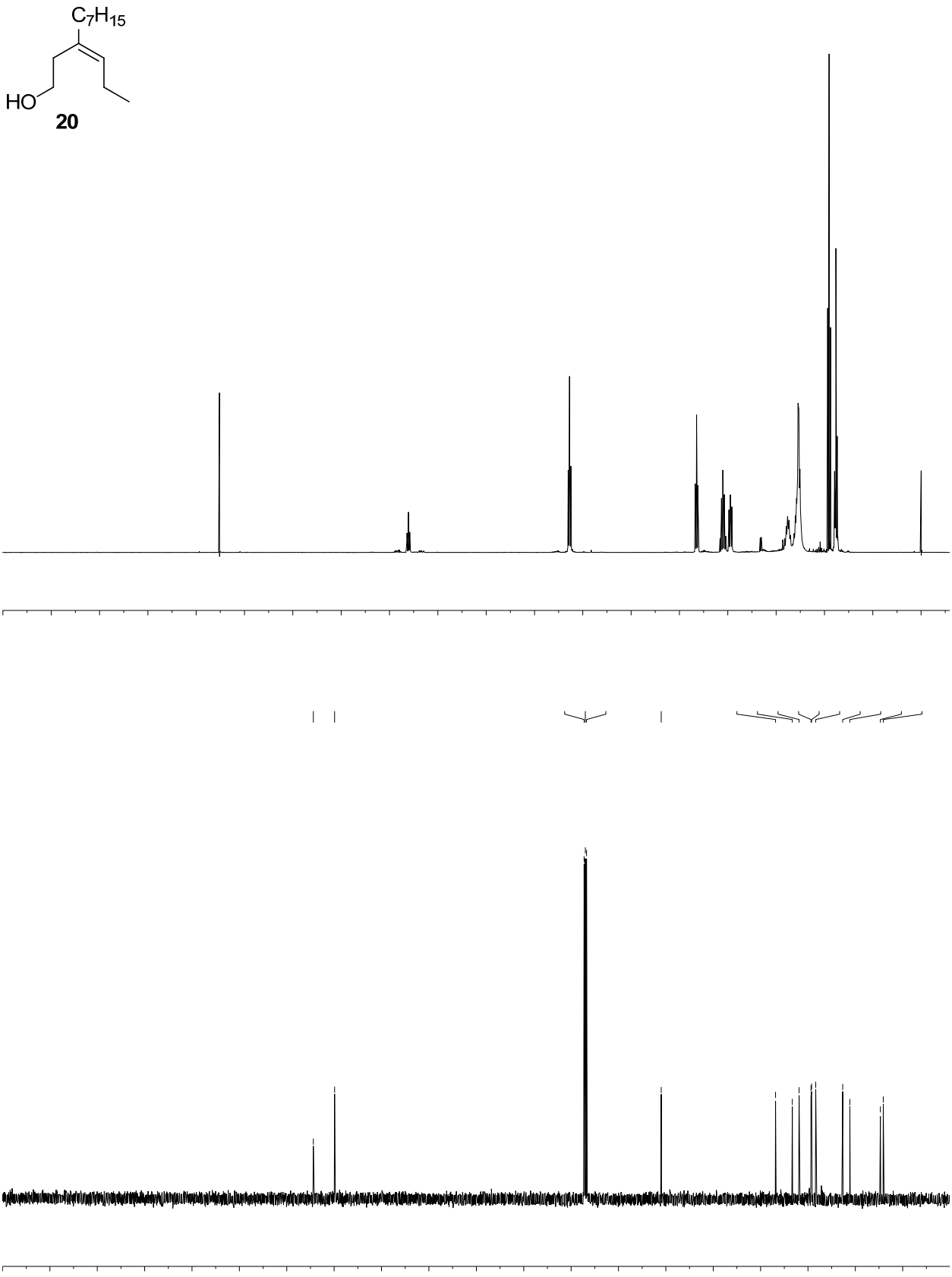

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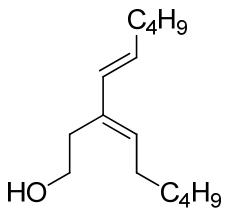

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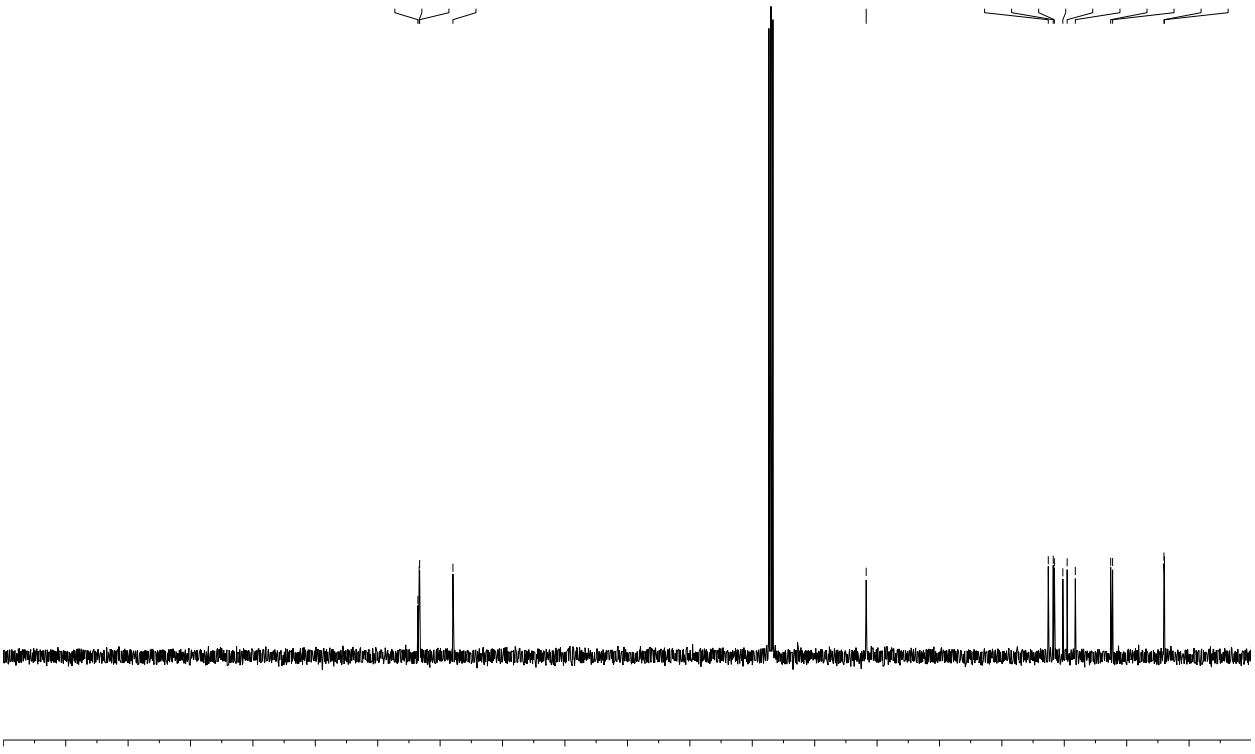

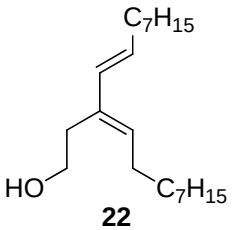

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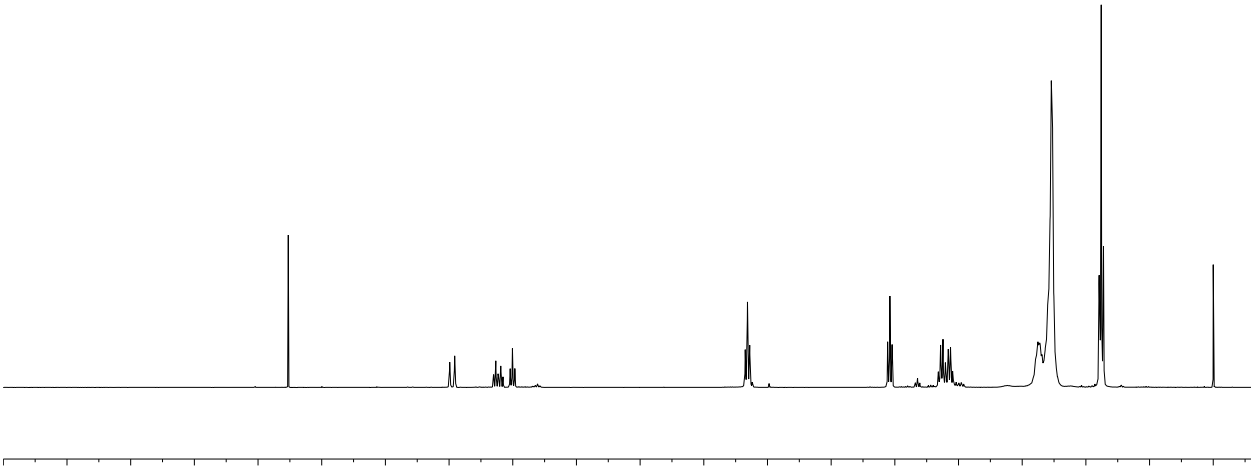

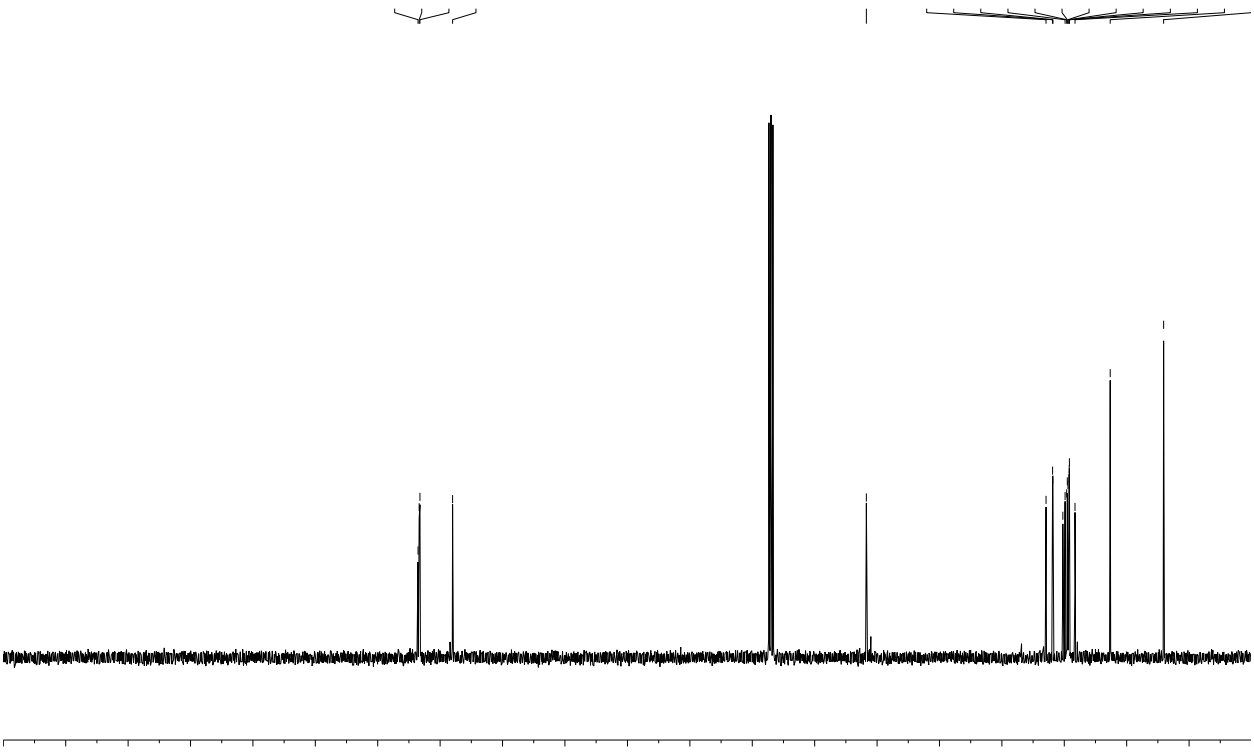

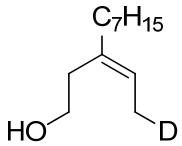

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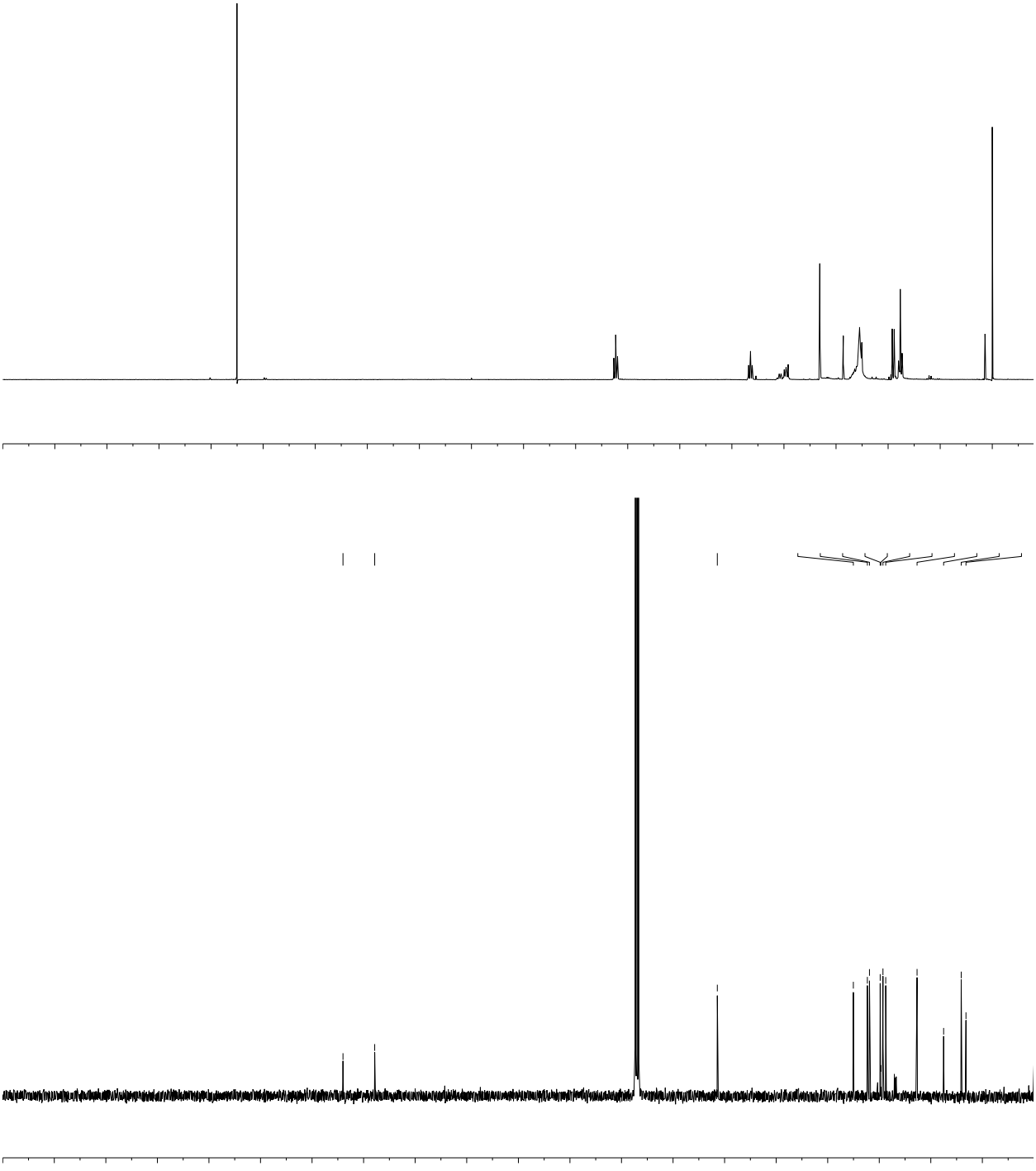

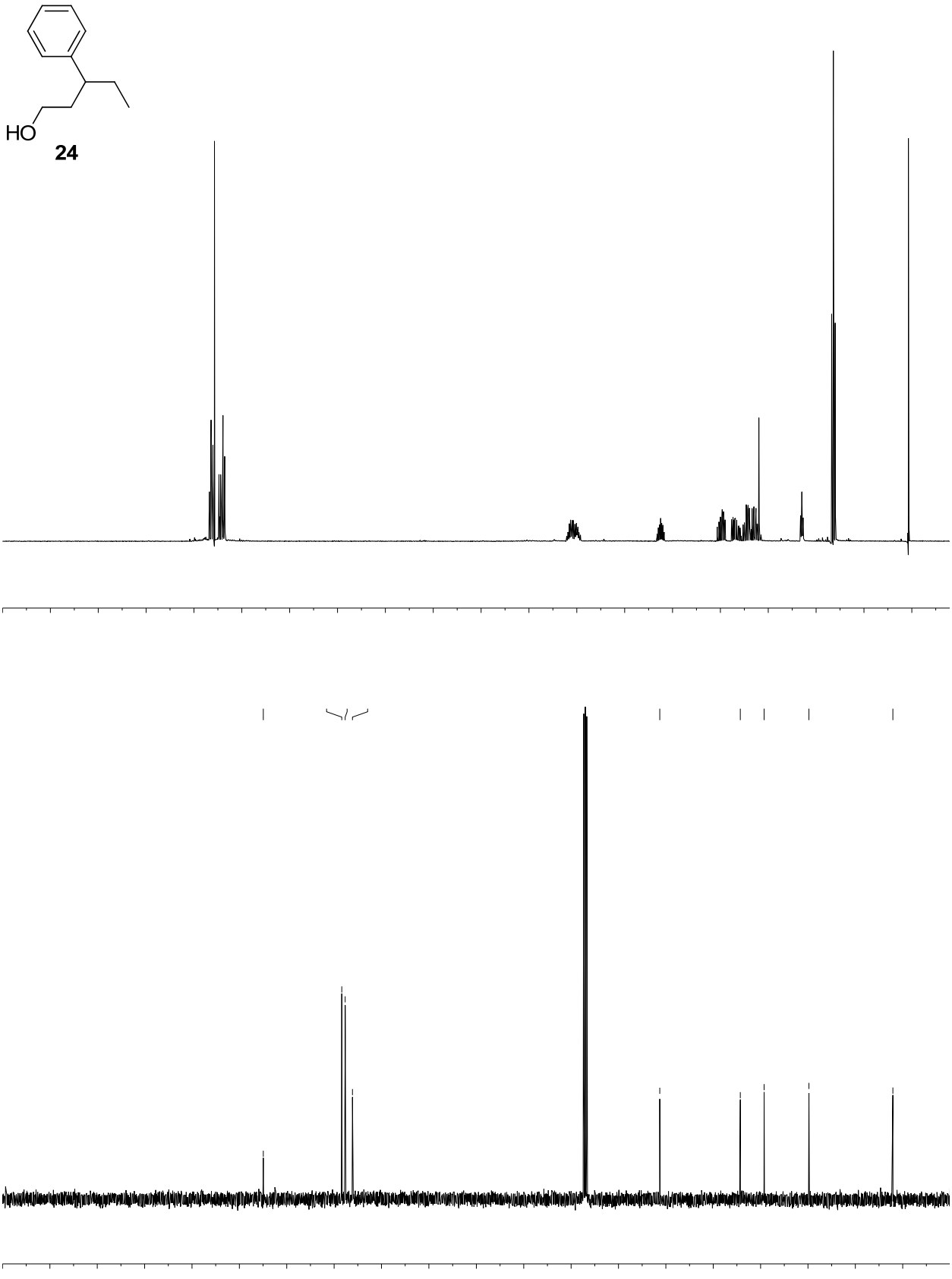

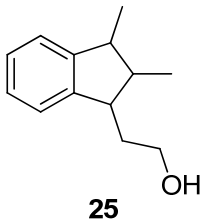

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