Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

An Alkyne Hydrosilylation-Hiyama Coupling Approach to Highly Functionalised 1,3-Dienes

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General Methods

All reactions were carried out under an atmosphere of argon in oven-dried glassware with magnetic stirring. All reactions were monitored by thin layer chromatography (TLC) using Merck TLC silica gel 60 sheets, which were visualised with ultraviolet light and then developed with iodine and basic potassium permanganate solution. Flash chromatography was performed on Sigma-Aldrich silica gel 60 or Flurochem silica gel as the stationary phase and the solvents employed were of analytical grade. 1H NMR spectra were recorded on a Bruker AVX300 (300 MHz) spectrometer at ambient temperature. Data are reported as follows: chemical shift in parts per million (δ, ppm) from deuterated chloroform (CDCl3) taken as 7.26 ppm, integration, multiplicity (s = singlet; d = doublet; t = triplet; dd = double doublets, qd = quartet/quintet of doublets, m = multiplet), and coupling constant (Hz). 13C NMR spectra were recorded on either a Bruker AVX300 (75 MHz) spectrometer. Chemical shifts are reported in ppm from CDCl3 taken as 77.0 ppm. Infrared spectra were recorded on a Perkin Elmer RX I FT-IR spectrometer as liquid films or as dilute solutions between two KBr discs. Mass spectra were recorded on either a Micromass GCT Premier or a Waters Micromass LCT Premier spectrometer using electron ionisation (EI) at 70 eV or electrospray (ES) techniques, respectively. Unless stated otherwise, all commercially available reagents were used as received. When necessary, THF was dried prior to use according to standard laboratory practices. Dioxane was used without drying in all cases.

General Procedure A: Hydrosilylation of Propargyl Alcohols

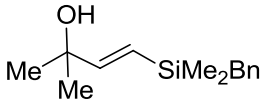
To an oven dried 5 mL round bottom flask equipped with a reflux condenser and magnetic stirrer was added $PtCl_2$ (1 mol %) and 2-Dicyclohexylphosphino-2′,4′,6′-triisopropylbiphenyl (2 mol %) (XPhos). The flask was then flushed quickly with argon and dry THF was added. The mixture was then stirred at 50 ^{o}C for 30 minutes until a yellow homogenous mixture was obtained. The corresponding propargyl alcohol (1 eq.) was added followed by benzyldimethylsilane (1.2 equivalents) via syringe (CAUTION: Rapid evolution of hydrogen gas) and the solution was stirred at 50^{o}C overnight. The solvent was evaporated and the crude mixture was applied to the top of a column and chromatographed to afford the requisite (*E*)-vinyl silane.

General Procedure B: Hiyama coupling

Vinylsilane **2** (1.0 equiv.) was added to an oven dried reaction vessel and placed under argon. Dioxane (used without drying) (1 mL) was added, followed by tetra-n-butylammonium fluoride (1M solution in THF) (2.2 equiv.). The orange solution was stirred at room temperature for 30 mins. A solution of vinyl iodide (1.1 equiv.) in dioxane (1 mL) was then added. $Pd_2dba.CHCl_3$ (5 mol%) was immediately added as a single portion.

Reaction was stirred for 4 – 6 hours (as determined by thin layer chromatography), at which time it was filtered through a pad of silica and concentrated under reduced pressure. Column chromatography (silica gel) afforded the requisite diene.

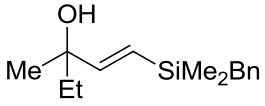
(*E*)-4-(Benzyldimethylsilyl)-2-methylbut-3-en-2-ol (2a)[1]



The title compound was prepared according to general procedure A, from 2-methyl-3-butyn-2-ol **1a** (84.1 mg, 1.00 mmol) and benzyldimethyl silane (165 mg, 1.10 mmol) using $PtCl_2$ (1.3 mg, 25.0 μmol) and XPhos (4.8 mg, 50.0 μmol) in THF (1 mL) which following conversion to the vinyl silane and column chromatography (19:1 Hexane/EtOAc) afforded **2a** (173 mg, 84%) as a colourless oil.

R_f (9:1 hexane – ethyl acetate) = 0.29; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.19 (2H, t, J = 7.5 Hz), 7.06 (1H, t, J = 7.3 Hz), 6.99 (2H, d, J = 7.5 Hz), 6.06 (1H, d, J = 19.1 Hz), 5.74 (1H, d, J = 19.1 Hz), 2.13 (2H, s), 1.27 (6H, s), 0.05 (6H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.6, 139.9, 128.3, 128.0, 124.0, 122.6, 72.0, 29.3, 26.1, -3.4; Characterisation in accordance with that previously reported.

(*E*)-1-(Benzyldimethylsilyl)-3-methylpent-1-en-3-ol (2b)

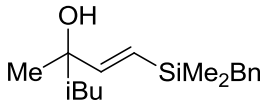


The title compound was prepared according to general procedure A, from 3-methylpent-1-yn-3-ol **1b** (500 mg, 5.10 mmol) and benzyldimethyl silane (735 mg, 5.61 mmol) using

$PtCl_2$ (6.8 mg, 25.5 μmol) and XPhos (24 mg, 51.0 μmol) in THF (3 mL) which following conversion to the vinyl silane and column chromatography (9:1 Hexane/EtOAc) afforded **2b** (1.13 g, 97%) as a colourless oil.

R_f (9:1 hexane – ethyl acetate) = 0.28; IR: ν_{max} (thin film) / cm^{-1} 3362, 2953, 2928, 2856, 1616, 1470, 1361, 1390, 1247, 1147, 963, 908, 830; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.19 (2H, t, J = 7.3 Hz), 7.05 (1H, t, J = 7.3 Hz), 6.99 (2H, d, *J* = 7.3 Hz), 5.99 (1H, d, *J* = 19.1 Hz), 5.78 (1H, d, *J* = 19.1 Hz), 2.13 (2H, s), 1.55 – 1.47 (2H, m), 1.40 (1H, br s), 1.22 (3H, s), 0.82 (3H, t, *J* = 7.5 Hz, 0.06 (6H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ153.6, 136.0, 128.3, 128.1, 124.0, 123.7, 74.3, 53.4, 34.7, 27.2, 26.2, -3.3, -3.3; HRMS (ES^+) Calcd. for $C_{15}H_{25}OSi$ $[M+H]^+$ 249.1675. Found 249.1679

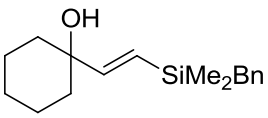
(*E*)-1-(Benzyldimethylsilyl)-3,5-dimethylhex-1-en-3-ol (2c)



The title compound was prepared according to general procedure A, from 3,5-dimethylhex-1-yn-3-ol **1c** (500 mg, 3.97 mmol) and benzyldimethyl silane (572 mg, 4.37 mmol) using $PtCl_2$ (5.3 mg, 19.8 μmol) and XPhos (18.9 mg, 39.7 μmol) in THF (3 mL) which following conversion to the vinyl silane and column chromatography (9:1 Hexane/EtOAc) afforded **2c** (1.02 g, 99%) as a colourless oil.

R_f (9:1 hexane – ethyl acetate) = 0.34; IR: ν_{max} (thin film) / cm^{-1} 3500, 2976, 1769, 1637, 1604, 1443, 1382, 1151, 1064, 1032, 859; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.21 (2H, t, *J* = 7.5 Hz), 7.07 (1H, t, *J* = 7.3 Hz), 6.99 (2H, d, *J* = 7.3 Hz), 6.06 (1H, d, *J* = 19.1 Hz), 5.76 (1H, d, *J* = 19.1 Hz), 2.14 (2H, s), 1.64 (1H, dq, *J* = 6.8, 6.2 Hz), 1.44 (2 H, t, *J* = 5.5 Hz), 1.24 (3H, s), 0.92 (3H, t, *J* = 6.5), 0.07 (6H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ154.4, 140.0, 128.3, 128.0, 124.0, 122.8, 74.6, 50.8, 28.7, 26.1, 24.6, 24.3, -3.3, -3.3; HRMS (ES^+) Calcd. for $C_{17}H_{29}OSi$ $[M+H]^+$ 277.1988. Found 277.1991

(*E*)-1-(2-(Benzyldimethylsilyl)vinyl)cyclohexanol (2d)

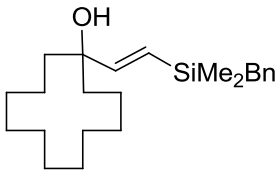


The title compound was prepared according to general procedure A, from 1-

ethynylcyclohexanol **1d** (500 mg, 4.03 mmol) and benzyldimethyl silane (581 mg, 4.4 mmol) using $PtCl_2$ (5.4 mg, 20.2 μmol) and XPhos (19 mg, 40.4 μmol) in THF (3 mL) which following conversion to the vinyl silane and column chromatography (9:1 Hexane/EtOAc) afforded **2d** (1.01 g, 99%) as a yellow oil.

R_f (9:1 hexane – ethyl acetate) = 0.32; IR: υ_{max} (thin film) / cm^{-1}3431, 2972, 2831, 1571, 1492, 1092, 1058; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.20 (2H, t, *J* = 7.3 Hz), 7.07 (1H, t, *J* = 7.3 Hz), 6.99 (2H, d, *J* = 7.3 Hz), 6.08 (1H, d, *J* = 19.1 Hz), 5.82 (1H, d, *J* = 19.1 Hz), 2.14 (2H, s), 1.70 - 1.55 (4H, m), 1.54 – 1.47 (6H, m), 1.29 (1H, br s), 1.22 (3H, s), 0.82 (3H, t, *J* = 7.5 Hz), 0.07 (6H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.6, 140.0, 128.3, 128.1, 124.0, 123.3, 72.6, 37.4, 26.1, 25.5, 22.0, -3.3; HRMS (ES^+) Calcd. for $C_{17}H_{27}OSi$ $[M+H]^+$ 275.1831. Found 275.1842

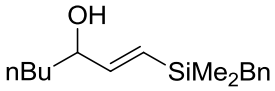
(E)-1-(2-(Benzyldimethylsilyl)vinyl)cyclododecanol (2e)



The title compound was prepared according to general procedure A, 1-ethynylcyclododecanol[1] **1e** (177 mg, 0.849 mmol) and benzyl dimethylsilane (140 mg 0.934 mmol) using $PtCl_2$ (1.1 mg, 4.24 μmol) and XPhos (4.0 mg, 8.49 μmol) in THF (3 mL) which following conversion to the vinyl silane and column chromatography (39:1 Hexane/EtOAc) afforded **2e** (210 mg, 69 %) as a colourless oil.

R_f (9:1 hexane – ethyl acetate) = 0.55; IR: ν_{max} (thin film) / cm^{-1} 3459, 2771, 2746, 1483, 1471, 1221, 1026, 1014, 907; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.14 (2H, t, *J* = 7.5 Hz), 7.00 (1H, t, *J* = 7.3 Hz), 6.94 (2H, d, *J* = 7.3 Hz), 6.02 (1H, d, *J* = 19.0 Hz), 5.71 (1H, d, *J* = 19.0 Hz), 2.12 (2H, s), 1.59 – 1.48 (2H, m), 1.46 – 1.25 (20H, m), 0.05 (6H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.4, 140.0, 128.3, 128.0, 124.0, 123.0, 76.2, 34.6, 26.5, 26.2, 22.6, 22.2, 19.6, -3.3; HRMS (ES^+) Calcd. for $C_{23}H_{38}NaOSi$ $[M+Na^+]^+$ 381.2590. Found 381.2601

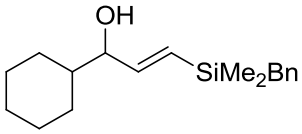
(*E*)-1-(Benzyldimethylsilyl)hept-1-en-3-ol (2f)



The title compound was prepared according to general procedure A, from hept-1-yn-3-ol **1f** (262 mg, 2.34 mmol) and benzyldimethyl silane (460 mg, 3.51 mmol) using $PtCl_2$ (6.2 mg, 23.4 μmol) and XPhos (22.3 mg, 46.8 μmol) in THF (2 mL) which following conversion to the vinyl silane and column chromatography (9:1 Hexane/EtOAc) afforded **2f** (413 mg, 76%) as a colourless oil.

R_f (9:1 hexane – ethyl acetate) = 0.41; IR: ν_{max} (thin film) / cm^{-1}3541, 2849, 2807, 1366, 1341, 1167, 1049, 1031; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.21 (2H, t, *J* = 7.5 Hz), 7.07 (1H, t, *J* = 7.3 Hz), 7.00 (2H, d, *J* = 7.3 Hz), 6.02 (1H, dd, *J* = 19.1, 5.3 Hz), 5.81 (1H, d, *J* = 19.1 Hz), 4.07 (1H, dd, *J* = 5.5, 5.3 Hz), 2.14 (2H, s), 1.51 (2H, dd, *J* = 12.3, 6.7), 1.46 (1 H, br s), 140 – 1.25 (4H, m), 0.92 (3H, t, *J* = 6.5 Hz), 0.05 (6H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.4, 128.6, 128.5, 127.4, 124.4, 75.1 37.0, 27.9, 26.4, 23.1, 14.4, -3.0; HRMS (ES^+) Calcd. for $C_{16}H_{27}OSi$ $[M+H]^+$ 263.1831 Found 263.1843

(*E*)-3-(Benzyldimethylsilyl)-1-cyclohexylprop-2-en-1-ol (2g)

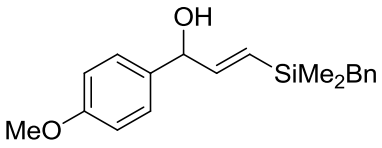


The title compound was prepared according to general procedure A, from 1-cyclohexylprop-2-yn-1-ol **1g** (189 mg, 1.37 mmol) and benzyldimethyl silane (270 mg, 2.06 mmol) using $PtCl_2$ (3.6 mg, 13.7 μmol) and XPhos (13.0 mg, 27.4 μmol) in THF (2 mL) which following conversion to the vinyl silane and column chromatography (9:1 Hexane/EtOAc) afforded **2g** (328 mg, 93%) as a colourless oil.

R_f (9:1 hexane – ethyl acetate) = 0.34; IR: ν_{max} (thin film) / cm^{-1} 3457, 2986, 2733, 1506, 1482, 1221, 1058, 963; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.19 (2H, t, *J* = 7.5 Hz), 7.06 (1H, t, *J* = 7.3 Hz), 6.99 (2H, d, *J* = 7.5 Hz), 5.99 (1H, dd, *J* = 18.8, 5.7 Hz), 5.77 (1H, dd, *J* = 18.8, 1.1 Hz), 3.82 (1H, dd, *J* = 9.0, 5.8 Hz), 2.14 (2H, s), 1.81 – 1.69 (3H, m), 1.62 (1H, s), 1.35 (1H, dtt , *J* = 11.6, 5.0, 3.2 Hz), 1.04 – 0.86 (2H, m), 0.06 (6H, s) 1.26 – 1.09 (3H, m), 0.08 (6H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ149.0, 140.3, 128.6, 128.5, 124.4, 43.8, 29.3, 28.6, 26.9, 26.4, 11.0, -1.4, -2.9; HRMS (ES^+) Calcd. for $C_{18}H_{29}OSi$

$[M+H]^+$ 289.1988. Found 289.1974

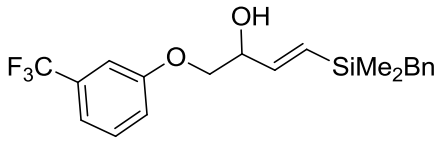
(*E*)-3-(Benzyldimethylsilyl)-1-(4-methoxyphenyl)prop-2-en-1-ol (2h)



The title compound was prepared according to general procedure A, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1h** (150 mg, 0.926 mmol) and benzyldimethyl silane (153 µmg 1.02 mmol) using $PtCl_2$ (2.5 mg, 9.26 µmol) and XPhos (8.8 mg,18.5 µmol) in THF (2 mL) which following conversion to the vinyl silane and column chromatography (9:1 Hexane/EtOAc) afforded **2h** (222 mg, 77%) as a yellow oil.

R_f (9:1 hexane – ethyl acetate) = 0.24; IR: ν_{max} (thin film) / cm^{-1} 3362, 2953, 2928, 2856, 1616, 1470, 1361, 1390, 1247, 1147, 963, 908, 830; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.24 – 7.16 (4H, m), 7.09 - 7.02, 6.99 – 6.94 (2H, m), 6.89 (2H, d, *J* = 8.2 Hz), 6.12 (1H, dd, *J* = 18.8, 4.9 Hz), 5.93 (1H, dd, *J* = 18.8, 1.3 Hz), 5.13 (1H, t, *J* = 4.1 Hz), 3.82 (3H, s), 2.14 (2H, s), 1.85 (1H, d, *J* =13.8 Hz), 0.06 (6H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.2, 148.6, 139.8, 128.3, 128.2, 127.8, 127.3, 124.0, 114.0, 76.3, 55.3,30.9, 26.0, -3.4; HRMS (ES^+) Calcd. for $C_{19}H_{24}NaOSi$ $[M+Na]^+$ 335.1443. Found 335.1451

(*E*)-4-(Benzyldimethylsilyl)-1-(3-(trifluoromethyl)phenoxy)but-3-en-2-ol (2i)

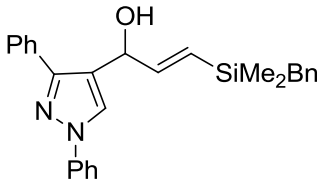


The title compound was prepared according to general procedure A, from 1-(3-(trifluoromethyl)phenoxy)but-3-yn-2-ol **1i** (244 mg, 1.06 mmol) and benzyldimethyl silane (238 mg, 1.59 mmol) using $PtCl_2$ (2.8 mg, 10.6 µmol) and XPhos (10.1 mg, 21.2 µmol) in THF (2 mL) which following conversion to the vinyl silane and column chromatography (9:1 Hexane/EtOAc) afforded **2i** (333 mg, 81%) as a yellow oil.

R_f (9:1 hexane – ethyl acetate) = 0.35; IR: ν_{max} (thin film) / cm^{-1} 3563, 2901, 2885, 1254, 1187, 1154, 1058, 1035, 910, 850; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.40 (1H, t, *J* = 8.0 Hz), 7.23 - 7.17 (3H, m), 7.14 (1H, s), 7.08 (1H, dd, *J* = 13.3, 7.3 Hz), 6.98 (2H, d, *J* =

7.3 Hz), 6.08 (1H, d, *J* = 18.8 Hz), 6.01 (1H, dd, *J* = 18.8, 3.8 Hz), 4.53 (1H, dt, *J* = 10.8, 3.8 Hz), 3.99 (1H, dd, *J* = 9.8, 3.2 Hz), 3.85 (1H, dd, *J* =9.8, 7.3 Hz), 2.16 (2H, s), 1.54 (1H, br s), 0.09 (6H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.4, 144.8, 140.4, 133.8 (q, *J* = 32.8 Hz), 131.4, 130.7, 129.0, 128.8, 124.8, 118.9 (q, *J* = 1.5 Hz), 118.6 (q, *J* = 3.7 Hz), 112.1 (q, *J* = 3.7 Hz), 26.5, -2.7, -2.8 ; HRMS (ES$^+$) Calcd. for $C_{20}H_{27}NF_3O_2Si$ $[M+NH_4^+]^+$ 398.1763. Found 398.1752

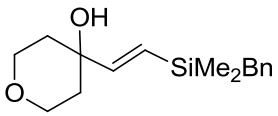
(*E*)-3-(Benzyldimethylsilyl)-1-(1,3-diphenyl-1H-pyrazol-4-yl)prop-2-en-1-ol (2j)



The title compound was prepared according to general procedure A, from 1-(1,3-diphenyl-1H-pyrazol-4-yl)prop-2-yn-1-ol[1] **1j** (180 mg, 0.657 mmol) and benzyldimethyl silane (118 mg, 0.788 mmol) using $PtCl_2$ (1.8 mg, 6.57 µmol) and XPhos (6.3 mg, 13.1 µmol) in THF (2 mL) which following conversion to the vinyl silane and column chromatography (9:1 Hexane/EtOAc) afforded **2j** (164 mg, 76%) as a colourless oil.

R_f (9:1 hexane – ethyl acetate) = 0.22; IR: ν_{max} (thin film) / cm^{-1} 3362, 2953, 2928, 2856, 1616, 1470, 1361, 1390, 1247, 1147, 963, 908, 830; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.81 (2H, d, *J* = 7.3 Hz), 7.79 (1H, s), 7.72 (1H, d, *J* = 8.3 Hz), 7.47 (3H, dd, *J* = 15.1, 8.0 Hz), 7.40 (1H, dd, *J* = 14.6, 8.0 Hz), 7.32 – 7.28 (1H, m), 7.17 (1H, t, *J* = 7.3 Hz), 7.09 – 7.01 (12H, m), 6.96 (2H, d, *J* = 7.5 Hz), 6.25 (1H, dd, *J* = 19.1, 4.3 Hz), 6.02 (1H, d, *J* = 18.8 Hz), 5.37 (1H, dd, *J* = 4.3, 3.5 Hz), 2.14 (2H, s), 1.92 (1H, d, 4.3 Hz), 0.07 (6H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.3, 140.6, 133.7, 130.1, 129.3, 129.1, 128.9, 128.8, 128.5, 127.3, 124.8, 119.9, 114.6, 68.9, 52.7, 38.6, 28.7, 26.6, -2.6, -2.8; HRMS (ES$^+$) Calcd. for $C_{27}H_{29}N_2OSi$ $[M+H]^+$ 425.2049. Found 425.2043

(*E*)-4-(2-(Benzyldimethylsilyl)vinyl)tetrahydro-2H-pyran-4-ol (2k)

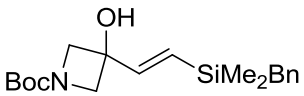


The title compound was prepared according to general procedure A, from 4-

ethynyltetrahydro-2H-pyran-4-ol **1j** (450 mg, 3.57 mmol) and benzyldimethyl silane (589 mg, 3.93 mmol) using $PtCl_2$ (4.8 mg, 17.9 µmol) and XPhos (17.0 mg, 35.7 µmol) in THF (3 mL) which following conversion to the vinyl silane and column chromatography (9:1 Hexane/EtOAc) afforded **2k** (789 mg, 86%) as a colourless solid.

R_f (9:1 hexane – ethyl acetate) = 0.35; IR: ν_{max} (thin film) / cm^{-1} 3564, 2086, 2895, 1206, 1198, 1154, 1058; ^1H NMR: (300 MHz, $CDCl_3$) δ 7.19 (2H, t, *J* = 7.5 Hz), 7.06 (1H, t, *J* = 7.3 Hz), 6.97 (2H, d, *J* = 7.5 Hz), 5.99 (1H, d, *J* = 18.8 Hz), 5.81 (1H, d, *J* = 18.8 Hz), 3.84 – 3.67 (4H, m), 2.15 (2H, s), 1.78 (2H, ddd, *J* = 13.3, 11.3, 4.8 Hz), 1.43 (2H, d, *J* = 13.1 Hz), 1.29 (1H, s), 0.08 (6H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.7, 140.2, 128.7, 128.5, 124.9, 124.5, 70.8, 70.5, 64.1, 37.8, 26.4, -3.0; HRMS (ES^+) Calcd. for $C_{16}H_{28}NO_2Si$ $[M+NH_4^+]^+$ 294.1889. Found 294.1880

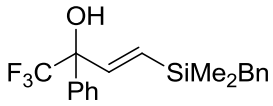
(*E*)-*tert*-Butyl 3-(2-(benzyldimethylsilyl)vinyl)-3-hydroxyazetidine-1-carboxylate (2l)



The title compound was prepared according to general procedure A, from *tert*-butyl 3-ethynyl-3-hydroxyazetidine-1-carboxylate[1] **1l** (143 mg, 0.726 mmol) and benzyldimethyl silane (120 mg, 0.799 mmol) using $PtCl_2$ (1.9 mg, 7.26 µmol) and XPhos (6.9 mg,14.5 µmol) in THF (2 mL) which following conversion to the vinyl silane and column chromatography (15:1 Hexane/EtOAc) afforded **2l** (194 mg, 79%) as a colourless oil.

R_f (7:1 hexane – ethyl acetate) = 0.17; IR: ν_{max} (thin film) / cm^{-1} 3498, 2977, 1786, 1649, 1601, 1467, 1396, 1063; ^1H NMR: (300 MHz, $CDCl_3$) δ 7.21 (2H, t, *J* = 7.5 Hz), 7.07 (1H, t, *J* = 7.3 Hz), 6.97 (2H, d, *J* = 7.5 Hz), 6.23 (1H, d, *J* = 19.0 Hz), 5.95 (1H, dd, *J* = 19.0 Hz), 3.96 (2H, d, *J* = 9.6 Hz), 3.92 (2H, d, *J* = 9.6 Hz), 2.15 (2H, s), 1.45 (9H, s), 0.08 (6H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.1, 148.5, 140.2, 128.9, 128.9, 127.1, 124.9, 80.4, 71.4, 29.1, 26.5, -2.8; HRMS (ES^+) Calcd. for $C_{19}H_{20}ONSi$ $[M+H]^+$ 348.1995. Found 348.2006.

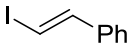
(E)-4-(benzyldimethylsilyl)-1,1,1-trifluoro-2-phenylbut-3-en-2-ol (2m)



The title compound was prepared according to general procedure A, from 1,1,1-trifluoro-2-phenylbut-3-yn-2-ol[1] **1m** (130 mg, 0.650 mmol) and benzyldimethyl silane (107 mg, 0.715 mmol) using $PtCl_2$ (1.7 mg, 6.50 µmol) and XPhos (5.7 mg, 12.0 µmol) in THF (2 mL) which following conversion to the vinyl silane and column chromatography (9:1 Hexane/EtOAc) afforded **2m** (218 mg, 83%) as a colourless oil.

R_f (9:1 hexane – ethyl acetate) = 0.42; IR: υ_{max} (thin film) / cm^{-1} 3360, 2983, 2904, 2741, 1616, 1470, 1360, 1247, 1147, 963; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.64 – 7.56 (4H, m), 7.43 – 7.35 (3H, m), 7.34 – 7.25 (1H, m), 7.20 – 7.13 (2H, m), 6.69 (1H, d, *J* = 18.8 Hz), 6.44 (1H, d, J = 18.8 Hz), 2.38 (2H, s), 0.20 (6H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.9, 139.8, 139.3, 136.7 (d, J = 56.7 Hz), 131.8, 128.4, 127.6, 126.6, 124.4, 26.7, 6.4, -3.4, -3.5; HRMS (ES^+) Calcd. for $C_{19}H_{22}F_3OSi$ $[M+H]^+$ 351.1392. Found 351.1384

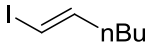
(*E*)-(2-Iodovinyl)benzene[2] (3a)



The title compound was prepared according to the general procedure outlined by Zhao.[2] Phenyl acetylene (100 mg, 0.980 mmol) and Cp_2ZrCl_2 (408mg, 1.40 mmol) were added to an oven dried round bottom flask and placed under argon. Dry THF (1 mL) was added. A separate solution of $LiAlH(O^tBu)_3$ (355 mg, 1.40 mmol) in THF (1.5 mL) was prepared and added to zirconium solution. Evolution of gas was observed and the colour of the reaction changed to orange. Reaction was stirred for 15 mins. Iodine (355 mg, 1.4 mmol) in THF (1 mL) was added dropwise then stirred for 15 mins. Reaction was quenched with 1M HCl (5mL), extracted with diethyl ether (3x 10mL) and the organic layers were dried over Na_2SO_4. Column chromatography (9:1 Hexane/EtOAc) provided the desired product (156 mg, 68%) as a colourless oil.

^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.43 (2H, d, *J* = 15.1 Hz), 7.33 (3H, m), 7.25 (1H, d, *J* = 1.0 Hz), 6.93 (1H, d, *J* = 14.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 130.5,k 129.2, 129.0, 128.4, 127.8, 125.5; All characterization in accordance with literature data.

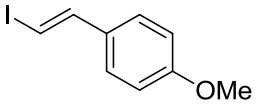
(*E*)-1-Iodohex-1-ene (3b)[2]



The title compound was prepared according to the general procedure outlined by Zhao.[2] 1-Hexyne (39.0, 0.457 mmol) and Cp_2ZrCl_2 (187 mg, 0.640 mmol) were added to an oven dried round bottom flask and placed under argon. Dry THF (0.5 mL) was added. A separate solution of $LiAlH(O^tBu)_3$ (163 mg, 0.640 mmol) in THF (1 mL) was prepared and added to zirconium solution. Evolution of gas was observed and the colour of the reaction changed to orange. Reaction was stirred for 15 mins. Iodine (161 mg, 0.640 mmol) in THF (0.5 mL) was added dropwise then stirred for 15 mins. Reaction was quenched with 1M HCl (5 mL), extracted with diethyl ether (3x 10mL) and the organic layers were dried over Na_2SO_4. Column chromatography (9:1 Hexane/EtOAc) provided the desired product (153 mg, 73%) as a dark green oil. (Caution! Product highly volatile. Isolated with ethyl acetate. Yield corrected accordingly).

(300 MHz, $CDCl_3$) δ 6.51 (1H, dt, *J* = 14.5, 7.2 Hz), 5.97 (1H, dt, *J* = 14.3, 1.3 Hz), 2.09 – 1.98 (2H, m), 1.40-1.28 (4H, m), 0.87 (3H, t, *J* = 7.8 Hz); All characterization in accordance with literature data.[2]

(*E*)-1-(2-Iodovinyl)-4-methoxybenzene (3c)[3]

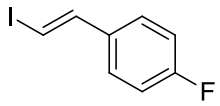


The title compound as prepared according to a modified version of the procedure used by Kabir.[4] $CrCl_2$ (152 mg, 1.24 mmol) was added to an oven dried round bottom flask and placed under argon. Dry THF (0.5 mL) was added and the stirred vigorously. The temperature was lowered to 0 ºC. Freshly prepared iodoform (163 mg, 0.413 mmol) and anisaldehyde (28.1, 0.207 mmol) were added to a separate flask and dissolved in THF (0.5 mL). This solution was then transferred via syringe to the flask containing $CrCl_2$. Reaction was stirred for 6 hours, then quenched with H_2O and extracted with diethyl (3x 5mL) ether. The combined organic layers were dries over Na_2SO_4 and concentrated. Column chromatography (99:1 hexane- ethyl acetate) provided desired vinyl iodide (28.9 mg, 54%) as a colourless oil.

R_f (9:1 hexane – ethyl acetate) = 0.76; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.36 (1H, d, *J* = 14.9 Hz), 7.23 (2H, d, *J* = 8.9 Hz), 6.83 (2H, d, *J* = 8.9 Hz), 6.65 (1H, d, *J* = 14.9 Hz),

3.80 (3H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.1, 144.7, 131.1, 128.1, 127.7, 114.5, 55.7; All characterisation in accordance with literature data.[3]

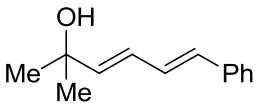
(*E*)-1-Fluoro-4-(2-iodovinyl)benzene (3d)[5]



The title compound was prepared following the procedure outlined for **3c**, using 4-fluorobenzaldehyde (25 µL, 0.23 mmol), iodoform (183 mg, 0.466 mmol) and $CrCl_2$ (172 mg, 1.40 mmol) in THF (1 mL).

R_f (9:1 hexane – ethyl acetate) = 0.83; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.36 (1H, d, $J_{C\text{-}F}$ = 14.9 Hz), 7.25 – 7.21 (2H, m), 7.02 – 6.97 (2H, m), 6.77 (1H, dd, J = 14.9, 0.7 Hz) ^{13}C NMR (75 MHz, CDCl3) δ 162.5 (d, $J_{C\text{-}F}$ = 249.3 Hz), 143.7, 133.9 (d, $J_{C\text{-}F}$ = 3.7 Hz), 129.1, 127.6 (d, $J_{C\text{-}F}$ = 8.4 Hz), 115.7 (d, $J_{C\text{-}F}$ = 22.6 Hz); All characterisation in accordance with literature data.[5]

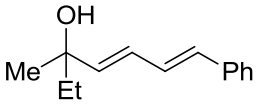
(3*E*,5*E*)-2-Methyl-6-phenylhexa-3,5-dien-2-ol (4a)[6]



The title compound was prepared following general procedure B, from **2a** (36.1 mg, 0.153 mmol) and (*E*)-(2-iodovinyl)benzene **3a** (35.2 mg, 0.168 mmol) using TBAF (1M solution in THF) (0.337 mL, 0.337 mmol) and $Pd_2dba_3.CHCl_3$ (7.9 mg, 7.65 µmol) in dioxane (1.5 mL). Following conversion to the diene and column chromatography (19:1 Hexane/EtOAc), this afforded **4a** (24.4 mg, 85%) as a colourless solid.

R_f (9:1 hexane – ethyl acetate) = 0.11; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.39 (2H, d, J = 7.2 Hz), 7.31 (2H, t, J = 7.2 Hz), 7.25 – 7.18 (1H, m), 6.76 (1H, dd, J = 15.6, 10.3 Hz), 6.55 (1H, d, J = 15.6 Hz), 6.39 (1H, dd, J = 15.6, 10.5 Hz), 5.96 (1H, d, J = 15.6 Hz),1.52 (1H, s), 1.38 (6H, s);^{13}C NMR (100 MHz, $CDCl_3$) δ 141.8, 137.3, 132.3, 128.6, 128.5, 127.6, 127.4, 126.3, 70.9, 29.8; All characterisation in accordance with literature data.[6]

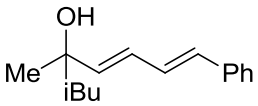
(4*E*,6*E*)-3-Methyl-7-phenylhepta-4,6-dien-3-ol (4b)[7]



The title compound was prepared following general procedure B, from **2b** (37.6 mg, 0.152 mmol) and (*E*)-(2-iodovinyl)benzene **3a** (35.1 mg, 0.182 mmol) using TBAF (1M solution in THF) (0.337 mL, 0.337 mmol) and $Pd_2dba_3.CHCl_3$ (7.9 mg, 7.60 µmol) in dioxane (1.5 mL). Following conversion to the diene and column chromatography (19:1 Hexane/EtOAc), this afforded **4b** (24.8 mg, 81%) as a colourless solid.

R_f (9:1 hexane – ethyl acetate) = 0.19; ^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.39 (2H, d, *J* = 7.28 Hz), 7.30 (2H, t, *J* = 7.28 Hz), 7.24 – 7.19 (1H, m), 6.78 (1H, dd, *J* = 15.8, 10.5 Hz), 6.55 (1H, d, *J* = 15.6 Hz), 6.41 (1H, dd, *J* = 15.6, 10.5 Hz), 5.88 (1H, d, *J* = 15.3 Hz), 1.62 (2H, q, *J* = 7.8 Hz), 1.44 (1H, s), 1.33 (3H, s), 0.98 (3H, t, *J* = 7.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.8, 137.4, 132.0, 128.6, 128.6, 127.9, 127.4, 126.3, 73.3, 35.3, 27.6, 8.3; All characterisation in accordance with literature data.[7]

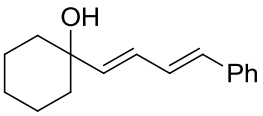
(5*E*,7*E*)-2,4-Dimethyl-8-phenylocta-5,7-dien-4-ol (4c)[6]



The title compound was prepared following general procedure B, from **2c** (41.6 mg, 0.159 mmol) and (*E*)-(2-iodovinyl)benzene **3a** (41.0 mg, 0.175 mmol) using TBAF (1M solution in THF) (0.350 mL, 0.350 mmol) and $Pd_2dba_3.CHCl_3$ (8.2 mg, 7.95 µmol) in dioxane (1.5 mL). Following conversion to the diene and column chromatography (15:1 Hexane/EtOAc), this afforded **4c** (32.1 mg, 88%) as a yellow oil.

R_f (9:1 hexane – ethyl acetate) = 0.22; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.47 – 7.38 (2H, m), 7.32 (2H, t, *J* = 7.28 Hz), 7.25 – 7.18 (1H, m), 6.79 (1H, dd, *J* = 15.5, 10.6 Hz), 6.53 (1H, d, *J* = 15.6 Hz), 6.41 (1H, dd, *J* = 15.6, 10.4 Hz), 5.89 (1H, d, *J* = 15.4 Hz), 1.79 – 1.65 (1H, m), 1.25 (3H, s), 0.96 (3H, d, J = 6.6 Hz, 0.93 (3H, d, J = 6.6 Hz);^{13}C NMR (75 MHz, $CDCl_3$) δ144.3, 139.0, 134.8, 128.0, 124.9, 121.2, 120.5, 70.8, 58.1, 51.4, 34.1, 25.7, 16.5; All characterisation in accordance with literature data.[6]

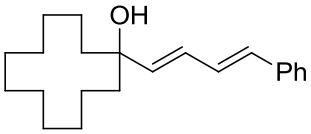
1-((1*E*,3*E*)-4-Phenylbuta-1,3-dien-1-yl)cyclohexanol (2d)[6]



The title compound was prepared following general procedure B, from **2d** (35.7 mg, 0.130 mmol) and (*E*)-(2-iodovinyl)benzene **3a** (45.1 mg, 0.196 mmol) using TBAF (1M solution in THF) (0.286 mL, 0.286 mmol) and $Pd_2dba_3.CHCl_3$ (6.7 mg, 6.50 µmol) in dioxane (1.5 mL). Following conversion to the diene and column chromatography (19:1 Hexane/EtOAc), this afforded **4d** (24.8 mg, 81 %) as a colourless solid.

R_f (9:1 hexane – ethyl acetate) = 0.29; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.39 (2H, d, *J* = 7.28 Hz), 7.31 (2H, t, *J* = 7.28 Hz), 7.24 – 7.17 (1H, m), 6.78 (1H, dd, *J* = 15.6, 10.6 Hz), 6.55 (1H, d, *J* = 15.6 Hz), 6.45 (1H, dd, *J* = 15.5, 10.4 Hz), 5.94 (1H, d, *J* = 15.3 Hz), 1.73 – 1.59 (6H, m), 1.57 – 1.48 (4H, m), 1.38 (1H, br s), 0.92 – 0.82 (1H, m);^{13}C NMR (75 MHz, $CDCl_3$) δ137.4, 132.2, 129.2, 128.8, 128.6, 127.8, 127.4, 126.3, 84.6, 38.0, 25.5, 22.1; All characterisation in accordance with literature data.[6]

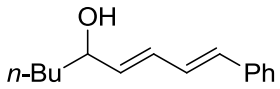
1-((1*E*,3*E*)-4-Phenylbuta-1,3-dien-1-yl)cyclododecanol (4e)



The title compound was prepared following general procedure B, from **2e** (47.6 mg, 0.133 mmol) and (E)-(2-iodovinyl)benzene **3a** (33.5 mg, 0.146 mmol) using TBAF (1M solution in THF) (0.293 mL, 0.293 mmol) and $Pd_2dba_3.CHCl_3$ (6.9 mg, 6.70 µmol) in dioxane (1.5 mL). Following conversion to the diene and column chromatography (19:1 Hexane/EtOAc), this afforded **4e** (26.1 mg, 63 %) as a colourless solid.

R_f (9:1 hexane – ethyl acetate) = 0.29; IR: ν_{max} (thin film) / cm^{-1} 2870, 2803, 1771, 1592, 1432, 1403, 1356, 1290, 974; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.54 – 7.77 (2H, m), 7.35 – 7.28 (2H, m), 7.25 – 7.19 (1H, m), 6.76 (2H, dd, *J* = 16.0, 10.0 Hz), 6.54 (1H, d, *J* = 15.8 Hz), 6.41 (1H, dd, *J* = 16.0, 10.0 Hz), 5.95 (1H, d, *J* = 15.6 Hz), 1.70 (1H, ddd, *J* = 14.3, 9.2, 4.0 Hz), 1.46 – 1.31 (17 H, m), 1.29 -1.18 (3H, m), 0.95 – 0.78 (2H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ141.4, 128.9, 128.9, 128.6, 128.3, 127.4, 126.3, 75.3, 53.4, 35.2, 26.4, 22.6, 22.2, 19.7; HRMS (ES) Calcd. for $C_{22}H_{33}O$ $[M+H]^+$ 313.2531 Found 313.2525

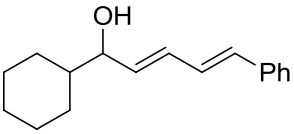
(1*E*,3*E*)-1-Phenylnona-1,3-dien-5-ol (4f)[8]



The title compound was prepared following general procedure B, from **2f** (39.3 mg, 0.159 mmol) and (*E*)-(2-iodovinyl)benzene **3a** (40.0 mg, 0.175 mmol) using TBAF (1M solution in THF) (0.349 mL, 0.349 mmol) and $Pd_2dba_3.CHCl_3$ (8.2 mg, 7.95 µmol) in dioxane (1.5 mL). Following conversion to the diene and column chromatography (19:1 Hexane/EtOAc), this afforded **4f** (26.8 mg, 78%) as a colourless solid.

R_f (9:1 hexane – ethyl acetate) = 0.21; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.39 (2H, d, *J* = 7.2 Hz), 7.31 (2H, t, *J* = 7.2 Hz), 7.24 – 7.17 (1H, m), 6.78 (1H, dd, *J* = 15.5, 10.4 Hz), 6.55 (1H, d, *J* = 15.6 Hz), 6.45 (1H, dd, *J* = 15.5, 10.4 Hz), 5.94 (1H, dd, *J* = 15.3, 6.8 Hz), 4.21 (1H, dd, *J* = 10.2, 6.8 Hz), 1.51 (2H, d, *J* = 4.5 Hz), 1.41 – 1.27 (4H, m), 0.94 – 0.86 (3H, m);^{13}C NMR (75 MHz, $CDCl_3$) δ 130.1, 129.1, 128.6, 128.6, 128.5, 128.4, 128.2, 126.3, 53.4, 30.5, 29.7, 22.7, 14.1; All characterisation in accordance with literature data.[8]

(2*E*,4*E*)-1-Cyclohexyl-5-phenylpenta-2,4-dien-1-ol (4g)

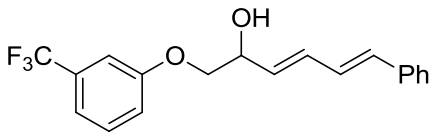


The title compound was prepared following general procedure B, from **2g** (24.9 mg, 86.5 µmol) and (*E*)-(2-iodovinyl)benzene **3a** (21.8 mg, 95.2 µmol) using TBAF (1M solution in THF) (0.190 mL, 0.190 mmol) and $Pd_2dba_3.CHCl_3$ (4.5 mg, 4.33 µmol) in dioxane (1.5 mL). Following conversion to the diene and column chromatography (19:1 Hexane/EtOAc), this afforded **4g** (17.1 mg, 81%) as a colourless oil.

R_f (9:1 hexane – ethyl acetate) = 0.19; IR: ν_{max} (thin film) / cm^{-1} 3241, 2995, 2961, 1752, 1658, 1501, 1497, 1058, 934 ; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.39 (2H, d, *J* = 7.2 Hz), 7.31 (2H, t, *J* = 7.2 Hz), 7.24 – 7.16 (1H, m), 6.79 (1H, dd, *J* = 15.3, 10.0 Hz), 6.54 (1H, d, *J* = 15.8 Hz), 6.37 (1H, dd, *J* = 14.9, 10.0 Hz), 5.82 (1H, dd, *J* = 15.3, 7.2 Hz), 3.94 (1H, ddd, *J* = 7.3, 4.1, 2.0 Hz), 1.93 – 1.83 (1H, m), 1.82 -1.70 (3H, m), 1.51 (1H, s),

1.29 – 1.22 (3H, m), 1.10 – 0.97 (3H, m), 0.95 – 0.86 (1H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.4, 132.2, 129.2, 128.8, 128.6, 127.8, 127.4, 126.3, 84.6, 71.6, 38.0, 25.5, 22.1; HRMS (ES^+) Calcd. for $C_{17}H_{23}O$ $[M+H]^+$ 243.1749 Found 243.1750

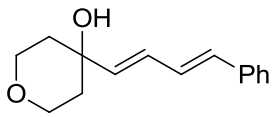
(3*E*,5*E*)-6-Phenyl-1-(3-(trifluoromethyl)phenoxy)hexa-3,5-dien-2-ol (4i)



The title compound was prepared following general procedure B, from **2i** (30.1 mg, 81.8 µmol) and (*E*)-(2-iodovinyl)benzene **3a** (20.4 mg, 89.2 µmol) using TBAF (1M solution in THF) (0.178 mL, 0.178 mmol) and $Pd_2dba_3.CHCl_3$ (4.2 mg, 4.05 µmol) in dioxane (1.5 mL). Following conversion to the diene and column chromatography (15:1 Hexane/EtOAc), this afforded **4i** (20.4 mg, 78 %) as a yellow solid.

R_f (19:1 hexane – ethyl acetate) = 0.30; IR: ν_{max} (thin film) / cm^{-1} 3107, 3021, 2974, 1741, 1605, 1504, 1491, 1452, 1058, 851; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.45 – 7.37 (3H, m), 7.39 – 7.27 (3H, m), 2.24 – 7.14 (2H, m), 7.14 – 7.07 (1H, m), 6.81 (1H, dd, *J* = 15.1, 10.0 Hz), 6.63 (1H, d, *J* = 15.6 Hz), 6.60 (1H, dd, *J* = 15.4, 10.1 Hz), 5.89 (1H, dd, *J* = 15.3, 7.0 Hz), 4.72 – 4.63 (1H, m), 4.09 (1H, dd, *J* = 9.4, 3.4 Hz), 3.98 (2H, dd, *J* = 9.4, 7.7 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.3, 134.2, 133.4, 130.6 (q, J = 32.2 Hz), 129.1, 128.3, 128.1, 126.9, 124.2, 123.4, 121.7,118.4 (q, J = 271.9 Hz), 72.4, 71.7, 30.1; HRMS (ES^+) Calcd. for $C_{19}H_{17}NaF_3O_2$ $[M+Na]^+$ 357.1078 Found 357.1085

4-((1*E*,3*E*)-4-Phenylbuta-1,3-dien-1-yl)tetrahydro-2*H*-pyran-4-ol (4k)

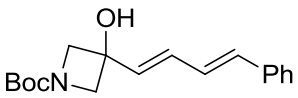


The title compound was prepared following general procedure B, from **2k** (51.3 mg, 0.198 mmol) and (*E*)-(2-iodovinyl)benzene **3a** (56.1 mg, 0.238 mmol) using TBAF (1M solution in THF) (0.486 mL, 0.486 mmol) and $Pd_2dba_3.CHCl_3$ (9.1 mg, 9.0 µmol) in dioxane (1.5 mL). Following conversion to the diene and column chromatography (5:1 Hexane/EtOAc), this afforded **4k** (37.0 mg, 89%) as a colourless solid.

R_f (3:1 hexane – ethyl acetate) = 0.15; IR: ν_{max} (thin film) / cm^{-1} 3162, 2953, 2774, 1502, 1486, 1308, 1247, 1147, 1058; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.39 (2H, d, *J* = 7.28 Hz),

7.32 (2H, t, *J* = 7.28 Hz), 7.25 – 7.21 (1H, m), 6.79 (1H, dd, *J* = 15.6, 10.5 Hz), 6.57 (1H, d, J = 15.6 Hz), 6.46 (1H, dd, *J* = 15.3, 10.3 Hz), 5.92 (1H, d, *J* = 15.3 Hz), 3.85 (2H, td, *J* = 11.1, 2.5 Hz), 3.75 (2H, dt, *J* = 11.0, 4.8 Hz), 1.90 (1H, dd, *J* = 10.8, 5.0 Hz), 1.85 (1H, dd, *J* = 10.8, 4.8 Hz), 1.62 – 1.56 (2H, m), 1.41 (1H, br s);^{13}C NMR (75 MHz, $CDCl_3$) δ140.3, 137.1, 133.1, 128.7, 128.4, 128.2, 127.7, 126.3, 69.2, 63.8, 37.9; HRMS (ES^+) Calcd. for $C_{15}H_{19}O_2$ $[M+H]^+$ 231.1385. Found 231.1374

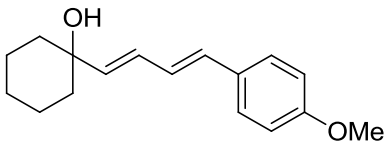
tert-Butyl 3-hydroxy-3-((1*E*,3*E*)-4-phenylbuta-1,3-dien-1-yl)azetidine-1-carboxylate (4l)



The title compound was prepared following general procedure B, from **2l** (30.0 mg, 86.4 µmol) and (E)-(2-iodovinyl)benzene **3a** (21.8 mg, 95.1 µmol) using TBAF (1M solution in THF) (0.190 mL, 0.190 mmol) and $Pd_2dba_3.CHCl_3$ (4.5 mg, 4.32 µmol) in dioxane (1.5 mL). Following conversion to the diene and column chromatography (5:1 Hexane/EtOAc), this afforded **4l** (19.1 mg, 74 %) as a colourless solid.

R_f (3:1 hexane – ethyl acetate) = 0.16; IR: ν_{max} (thin film) / cm^{-1} 3167, 3076, 2961, 1743, 1619, 1492, 1457, 1058, 974; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.41 (2H, d, *J* = 7.2 Hz), 7.33 (2H, t, *J* = 7.2 Hz), 7.26 – 7.21 (1H, m), 6.79 (2H, dd, *J* = 15.8, 9.8 Hz), 6.63 (1H, d, *J* = 15.4 Hz), 6.53 (1H, dd, *J* = 15.8, 10.5 Hz), 6.08 (1H, d, *J* = 15.4 Hz), 4.06 (2H, d, *J* = 9.6 Hz), 3.98 (2H, d, *J* = 9.6 Hz), 1.46 (9H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.0, 139.0, 129.1, 127.3, 126.9, 126.7, 126.4, 107.7, 103.1, 83.5, 71.7, 28.8, 11.0; HRMS (ES^+) Calcd. for $C_{18}H_{23}NaNO$ $[M+Na]^+$ 324.1576 Found 324.1584

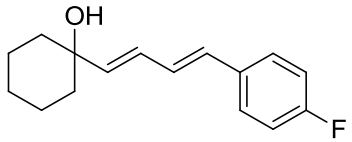
1-((1*E*,3*E*)-4-(4-Methoxyphenyl)buta-1,3-dien-1-yl)cyclohexanol (5)



The title compound was prepared following general procedure B, from **2d** (18.2 mg, 66.7 µmol) and (*E*)-1-(2-iodovinyl)-4-methoxybenzene **3b** (19.2 mg, 73.0 µmol) using TBAF (1M solution in THF) (0.146 mL, 0.146 mmol) and $Pd_2dba_3.CHCl_3$ (3.5 mg, 3.3

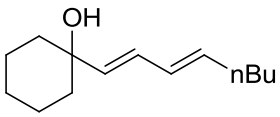
μmol) in dioxane (1.5 mL). Following conversion to the diene and column chromatography (19:1 Hexane/EtOAc), this afforded **5** (15.1 mg, 87%) as a yellow solid R_f (9:1 hexane – ethyl acetate) = 0.16; IR: ν_{max} (thin film) / cm^{-1} 2904, 2823, 1732, 1489, 1412, 1377, 1290, 1058; ^1H NMR: (300 MHz, $CDCl_3$) δ 7.31 (2H, d, *J* = 8.7 Hz), 6.84 (1H, d, *J* = 8.7 Hz), 6.67 (1H, dd, *J* = 15.6 Hz), 6.48 (1H, d, *J* = 15.6 Hz), 6.41 (1H, dd, *J* = 15.3, 10.2 Hz), 5.87 (1H, d, *J* = 15.3 Hz), 3.81 (3H, s), 1.61 (1H, d, *J* = 3.6 Hz), 1.55 – 1.48 (4H, m), 1.40 – 1.21 (5H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.0, 132.1, 130.6, 128.3, 127.9, 127.2, 114.5, 80.0, 71.2, 70.8, 55.7, 33.9; HRMS (ES^+) Calcd. for $C_{17}H_{22}NaO_2$ $[M+Na]^+$ 281.1517. Found 281.1511

1-((1*E*,3*E*)-4-(4-Fluorophenyl)buta-1,3-dien-1-yl)cyclohexanol (6)



The title compound was prepared following general procedure B, from **2d** (30.2 mg, 0.110 mmol) and (*E*)-1-fluoro-4-(2-iodovinyl)benzene **3c** (29.9 mg, 0.146 mmol) using TBAF (1M solution in THF) (0.242 mL, 0.242 mmol) and $Pd_2dba_3.CHCl_3$ (5.7 mg, 5.5 μmol) in dioxane (1.5 mL). Following conversion to the diene and column chromatography (19:1 Hexane/EtOAc), this afforded **6** (21.2 mg, 79 %) as a yellow oil.
R_f (9:1 hexane – ethyl acetate) = 0.23; IR: υ_{max} (thin film) / cm^{-1}2870, 2803, 1771, 1592, 1432, 1403, 1356, 1290, 974; ^1H NMR: (300 MHz, $CDCl_3$) δ 7.34 (2H, dd, *J* = 8.9, 5.8 Hz), 6.96 (2H, t, *J* = 8.7 Hz), 6.69 (1H, dd, *J* = 15.5, 10.2 Hz), 6.44 (1H, d, *J* = 15.5 Hz), 6.28 (1H, dd, *J* = 15.1, 10.4 Hz), 5.78 (1H, d, *J* = 15.3 Hz),1.61 (1H, d, *J* = 3.6 Hz), 1.55 – 1.48 (4H, m), 1.40 – 1.21 (5H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.0 (d, J = 249.3 Hz), 128.8 (d, J = 3.0 Hz), 128.1, 127.9, 127.6 (d, J = 8.7 Hz), 116.1, 115.8, 112.3 (d, J = 22.5 Hz), 67.6, 38.4, 25.9, 22.5; HRMS (ES^+) Calcd. for $C_{16}H_{20}FO$ $[M+H]^+$ 247.1498 Found 247.1486

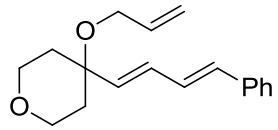
1-((1*E*,3*E*)-octa-1,3-dien-1-yl)cyclohexanol (7)



The title compound was prepared following general procedure B, from **2d** (34.5 mg, 0.126 mmol) and (*E*)-1-iodohex-1-ene **3d** (29.3 mg, 0.139 mmol) using TBAF (1M solution in THF) (0.277 mL, 0.277 mmol) and $Pd_2dba_3.CHCl_3$ (6.5 mg, 6.3 µmol) in dioxane (1.5 mL). Following conversion to the diene and column chromatography (19:1 Hexane/EtOAc), this afforded **7** (26.6 mg, 76 %) as a colourless solid.

Rf (9:1 hexane – ethyl acetate) = 0.19; IR: υ_{max} (thin film) / cm^{-1} 3308, 2978, 2843, 1876, 1739, 1520, 1442, 1297, 1184, 1058, 954; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 6.03 (1H, dd, *J* = 15.1, 6.8 Hz), 5.95 (1H, d, *J* = 15.2 Hz), 5.70 (1H, dd, *J* = 15.1, 8.0 Hz), 5.87 (1H, d, *J* = 15.1 Hz), 2.18 (2H, dt, *J* = 7.1, 6.8 Hz), 1.69 – 1.58 (2H, m), 1.54 – 1.41 (10 H, m), 1.37 – 1.29 (2H, m), 0.87 (1H, t, J = 7.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ147.0, 140.2, 126.4, 70.0, 65.0, 51.2, 37.2, 36.5, 24.7, 21.3, 21.2, 16.3; HRMS (ES^+) Calcd. for $C_{14}H_{24}NaO[M+Na]^+$ 231.1725. Found 231.1729

4-(allyloxy)-4-((1E,3E)-4-phenylbuta-1,3-dien-1-yl)tetrahydro-2H-pyran (8)

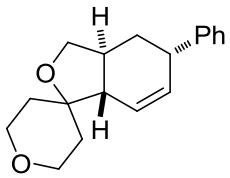


4k (11.4 mg, 45.0 µmol) was added to an oven dried round bottom flask and placed under argon. Dry THF (0.5 mL) was added, followed by allyl bromide (11.6 µL, 0.135 mmol). NaH (60% dispersion in mineral oil) (5.4 mg, 0.135 mmol) was added as a single portion and the temperature was raised to 60 ºC. Reaction was stirred at this temperature overnight, at which point it was quenched with sat. NH_4Cl. Extracted with Et_2O, dried over Na_2SO_4 and concentrated to provide desired product (11.8 mg, 89%) as a colourless oil. No further purification carried out.

R_f (9:1 hexane – ethyl acetate) = 0.43; IR: ν_{max} (thin film) / cm^{-1} 3162, 3098, 2953, 2774, 2548, 1507, 1392, 1308, 1247, 1153, 1058, 954; ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.39 (2H, d, *J* = 7.2 Hz), 7.32 (2H, t, *J* = 7.2 Hz), 7.25 – 7.21 (1H, m), 6.80 (1H, dd, *J* = 15.6, 10.5 Hz), 6.57 (1H, d, *J* = 15.6 Hz), 6.34 (1H, dd, *J* = 15.3, 10.3 Hz), 5.98 (1H, ddd, *J* = 11.2, 6.8, 5.0 Hz), 5.74 (1H, d, *J* = 15.3 Hz), 5.31 (1H, ddd, *J* = 11.2, 5.6, 4.0 Hz), 5.15 (1H, ddd, *J* = 11.2, 5.6, 4.0 Hz), 3.87 (2H, d, *J* = 5.1 Hz), 3.82 (2H, td *J* = 11.1, 2.5 Hz), 3.75 (2H, dt, *J* = 11.0, 4.8 Hz), 1.84 – 1.82 (4H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 133.4,

129.1, 128.7, 128.1, 126.8, 116.4, 94.2, 74.0, 64.1, 63.6, 35.4, 30.1, 14.5, 11.0; HRMS (ES^+) Calcd. for $C_{18}H_{23}O_2$ $[M+H]^+$ 271.1698. Found 271.1704

(3a*S,5*S**,7a*S**)-5-Phenyl-2',3a,3',4,5,5',6',7a-octahydro-3*H*-spiro[isobenzofuran-1,4'-pyran] (9)**



8 (11.8 mg, 0.04 mmol) was added to an oven dried pressure tube along with toluene (0.5 mL). Tube was sealed and heated to 150ºC overnight. When reaction was complete by TLC, it was cooled to room temperature and solvent was then evaporated under reduced pressure. Crude product passed through a short silica column (19:1 hexane-ethyl acetate) to provide **9** (11.0 mg, 93%, 1.7:1 *exo:endo,* inseparable mix*)* as a colourless oil.

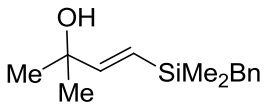
Major : ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.37 – 7.28 (2H. m), 7.25 – 7.19 (2H, m), 7.19 – 7.24 (1H, m), 6.01 (1H, d, *J* = 10.4 Hz), 5.79 (1H, dt, *J* = 9.8, 3.8 Hz), 3.90 – 3.79 (2H, m), 3.79 – 3.70 (2H, m), 3.65 (1H, dd, *J* = 8.4, 2.6 Hz), 3.40 (1H, dd, *J* = 10.6, 7.5 Hz), 2.22 (1H, ddd, *J* = 14.3, 6.8, 3.2 Hz), 2.11 – 1.90 (4H, m), 1.71 (2H, td, *J* = 12.4, 4.9 Hz), 1.39 (2H, d, *J* = 12.1 Hz), 0.91 – 0.78 (1H, br m); ^{13}C NMR (100 MHz, $CDCl_3$) δ145.7. 133.2, 128.4, 128.1, 127.3, 70.2, 65.6, 64.0,m 53.4, 48.1, 41.2, 36.2, 35.5, 33.1;

Minor : ^{1}H NMR: (300 MHz, $CDCl_3$) δ 7.37 – 7.28 (2H. m), 7.25 – 7.19 (2H, m), 7.19 – 7.24 (1H, m), 5.93 (1H, d, *J* = 10.7 Hz), 5.74 (1H, ddd, *J* = 9.7, 4.3, 3.3 Hz), 3.99 (1H, dd, *J* = 8.7, 6.0 Hz), 3.90- 3.79 (2H, m), 3.79 – 3.70 (2H, m), 3.67 (1H, dd, *J* = 8.4, 2.6 Hz), 2.53 (1H, ddd, *J* = 14.3, 6.8, 3.2 Hz), 2.11 – 1.90 (4H, m), 1.86 (2H, td, *J* = 12.6, 5.8 Hz), 1.49 (2H, d, *J* = 12.1 Hz), 0.91 – 0.78 (1H, br m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.0, 130.8,. 128.6, 126.3, 126.1, 71.0, 65.1, 64.5, 53.4, 48.1, 41.7, 36.2, 35.7, 33.4;

Combined: R_f (9:1 hexane – ethyl acetate) = 0.26; IR: ν_{max} (thin film) / cm^{-1} 3479, 3068, 3023, 2960, 1685, 1446, 1427, 1254, 1118, 1048, 998, 832, 795, 729, 699, 469; HRMS (ES^+) Calcd. for $C_{18}H_{23}O_2$ $[M+H]^+$ 271.1698. Found 271.1707

Spectral

Appendix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SiMe2Bn

2a

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1.01
1.00
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8
7
6
5
4
3
2
1
0
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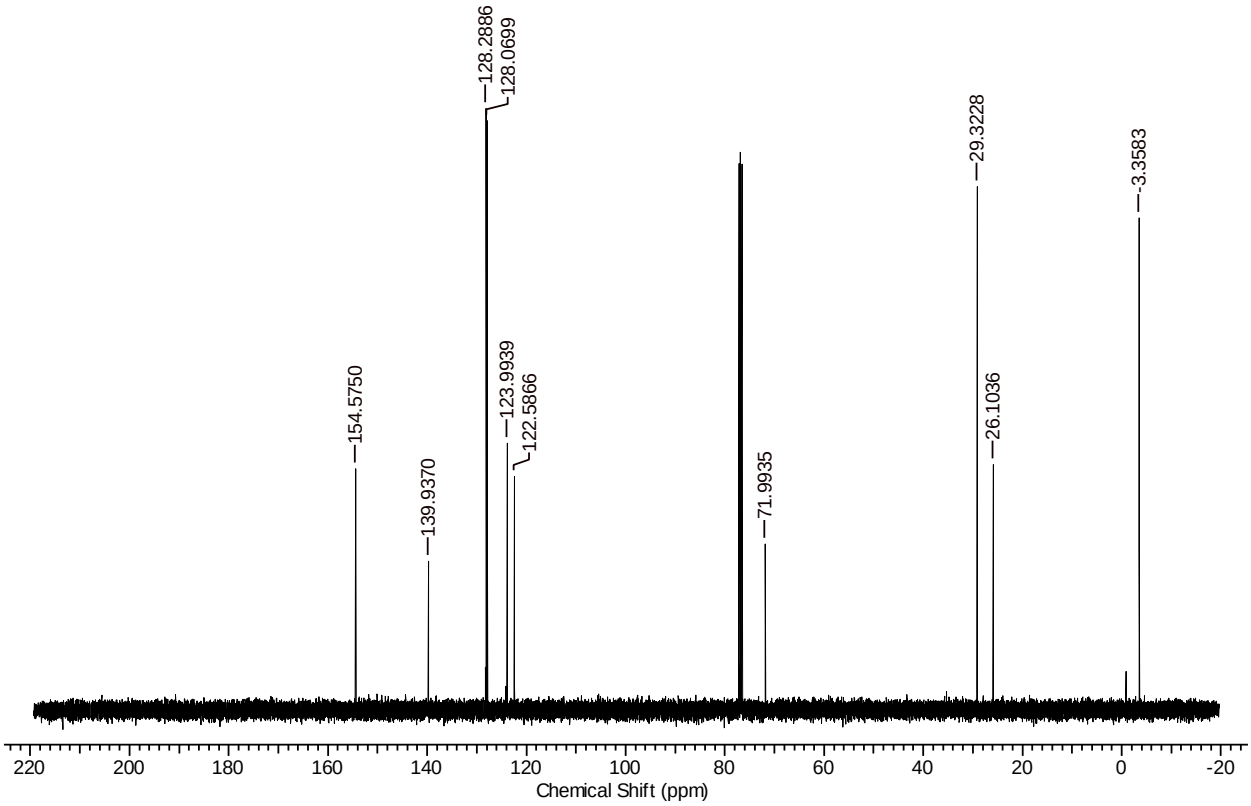
Chemical Shift (ppm)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140
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60
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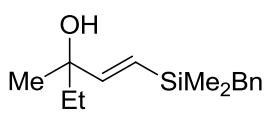
Chemical Shift (ppm)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Et
SiMe2Bn

2b

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0.99
1.00
1.96
1.93
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2.94
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7
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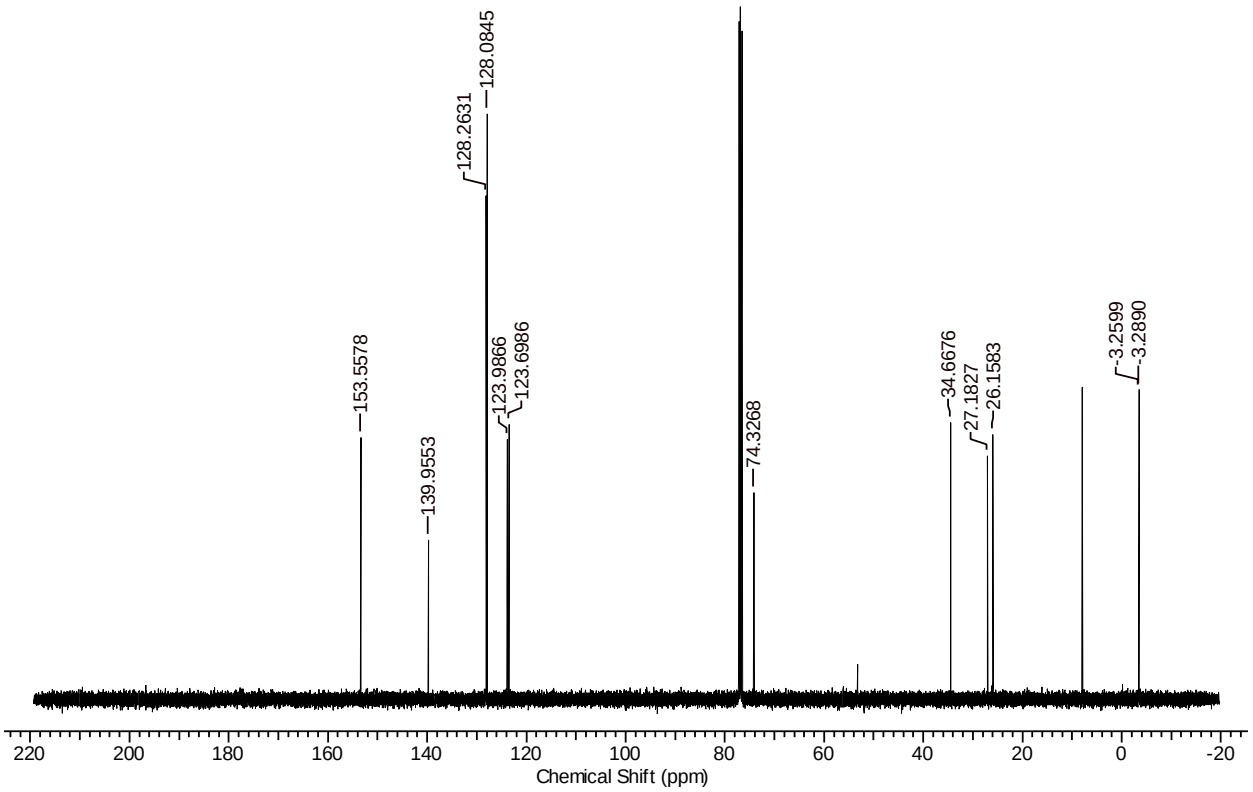
5
4
3
2
1
0
-1
Chemical Shift (ppm)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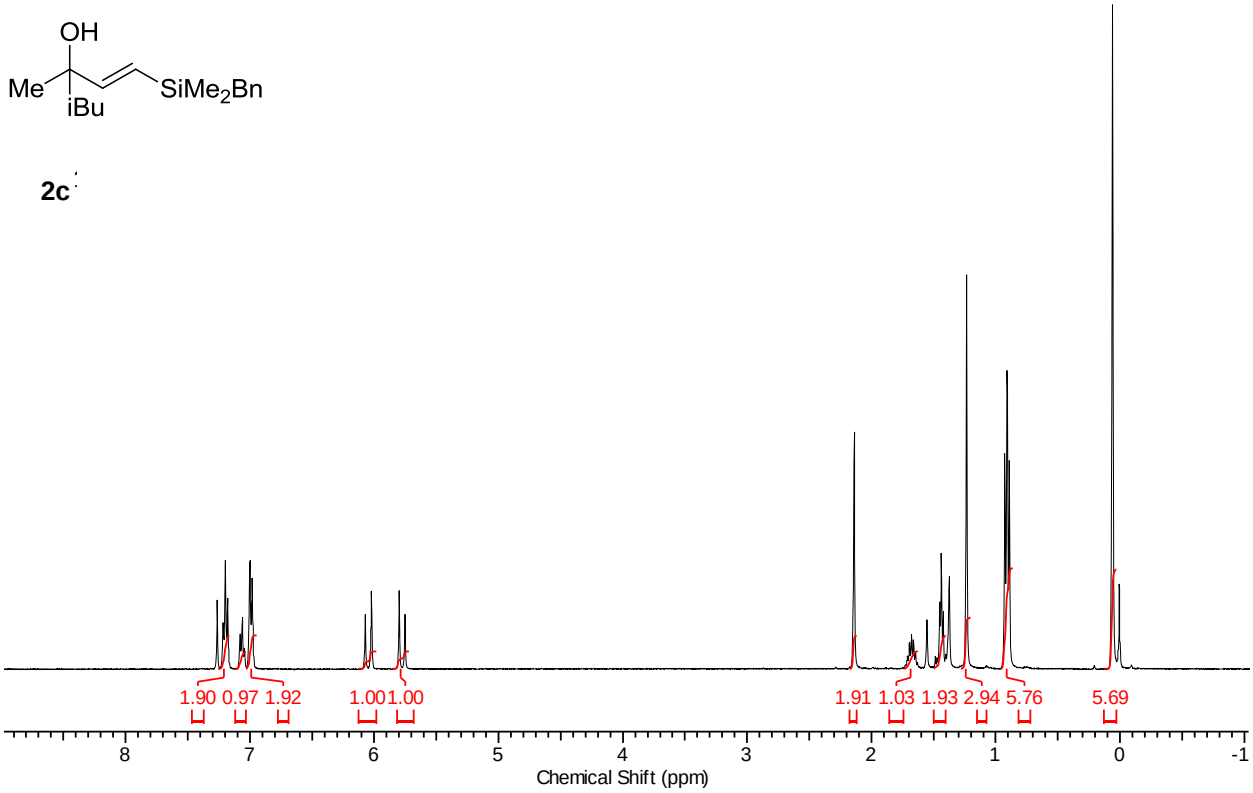
Chemical Shift (ppm)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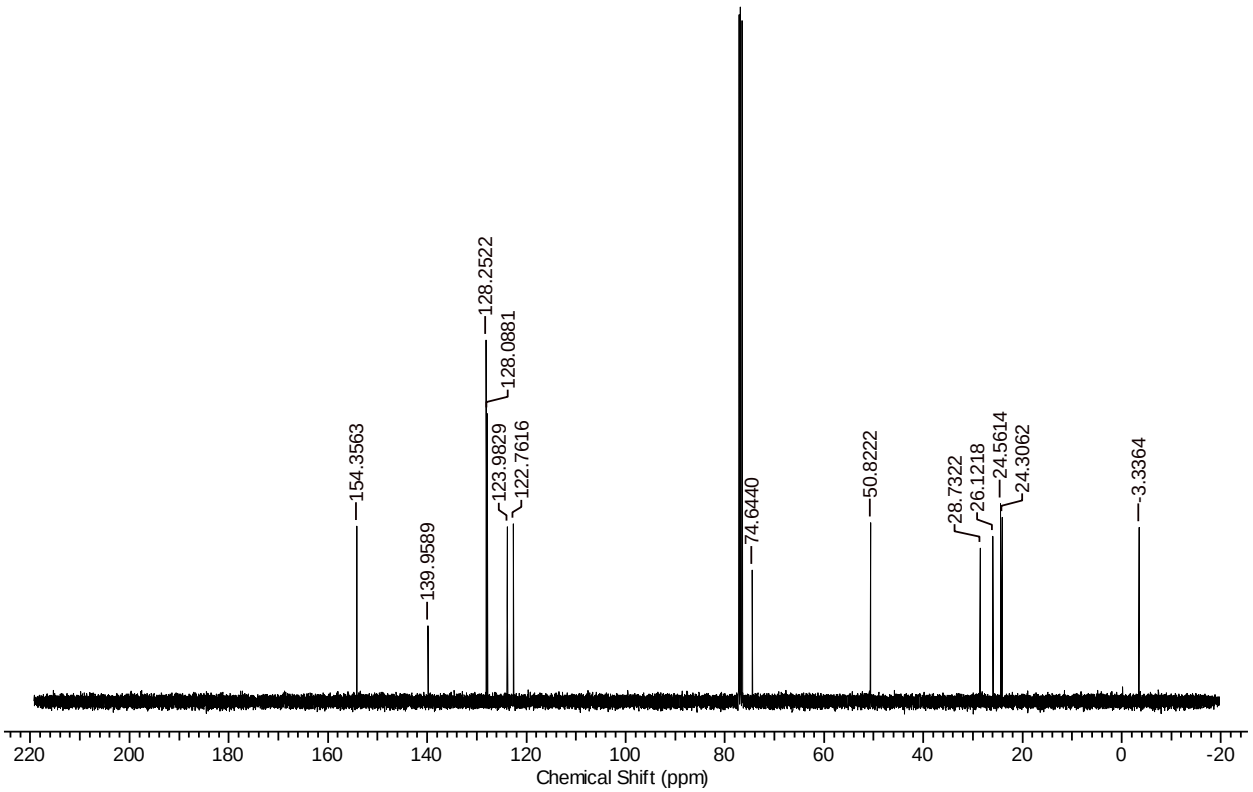
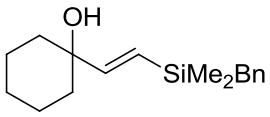
Chemical Shift (ppm)

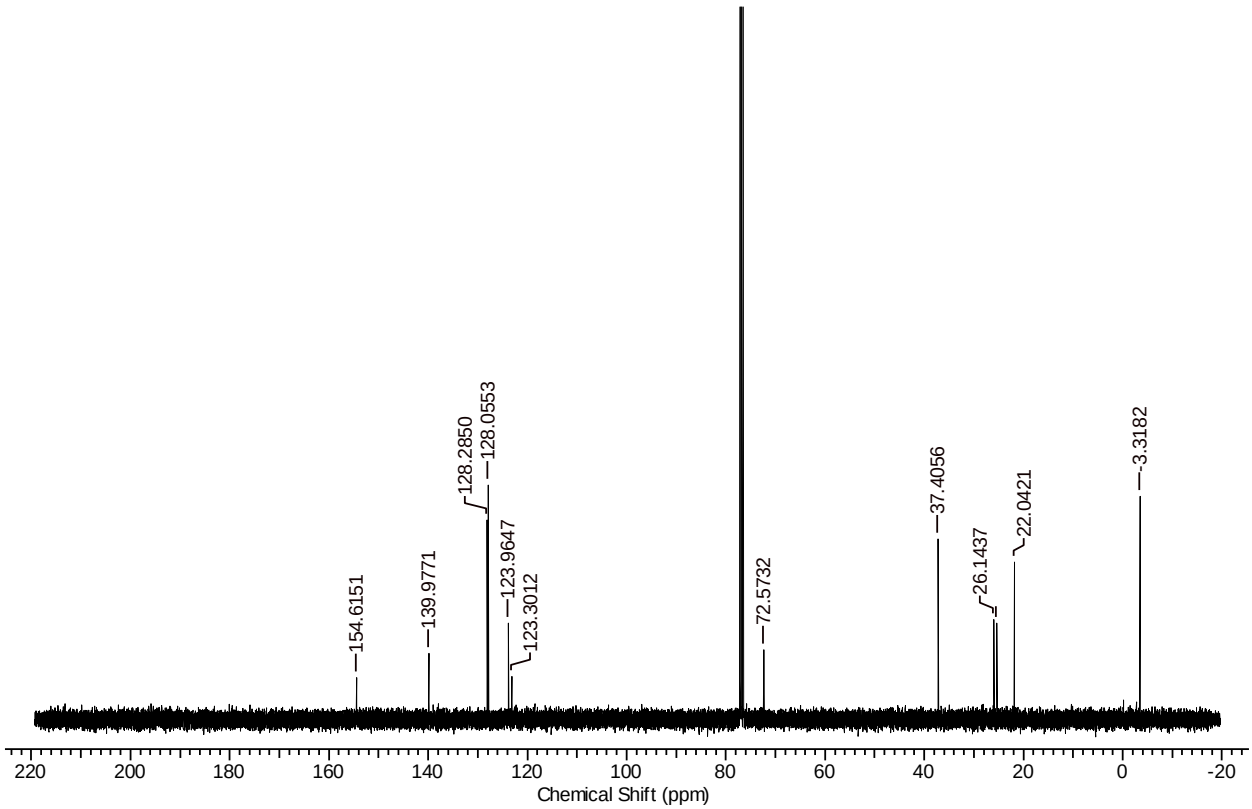
2c

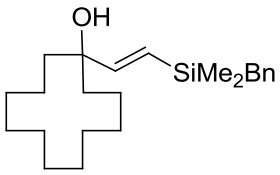

154.3563
139.9589
128.2522
128.0881
123.9829
122.7616
74.6440
50.8222
28.7322
26.1218
24.5614
24.3062
3.3364
220 200 180 160 140 120 100 80 60 40 20 0 -20
Chemical Shift (ppm)


OH
SiMe2Bn

2d

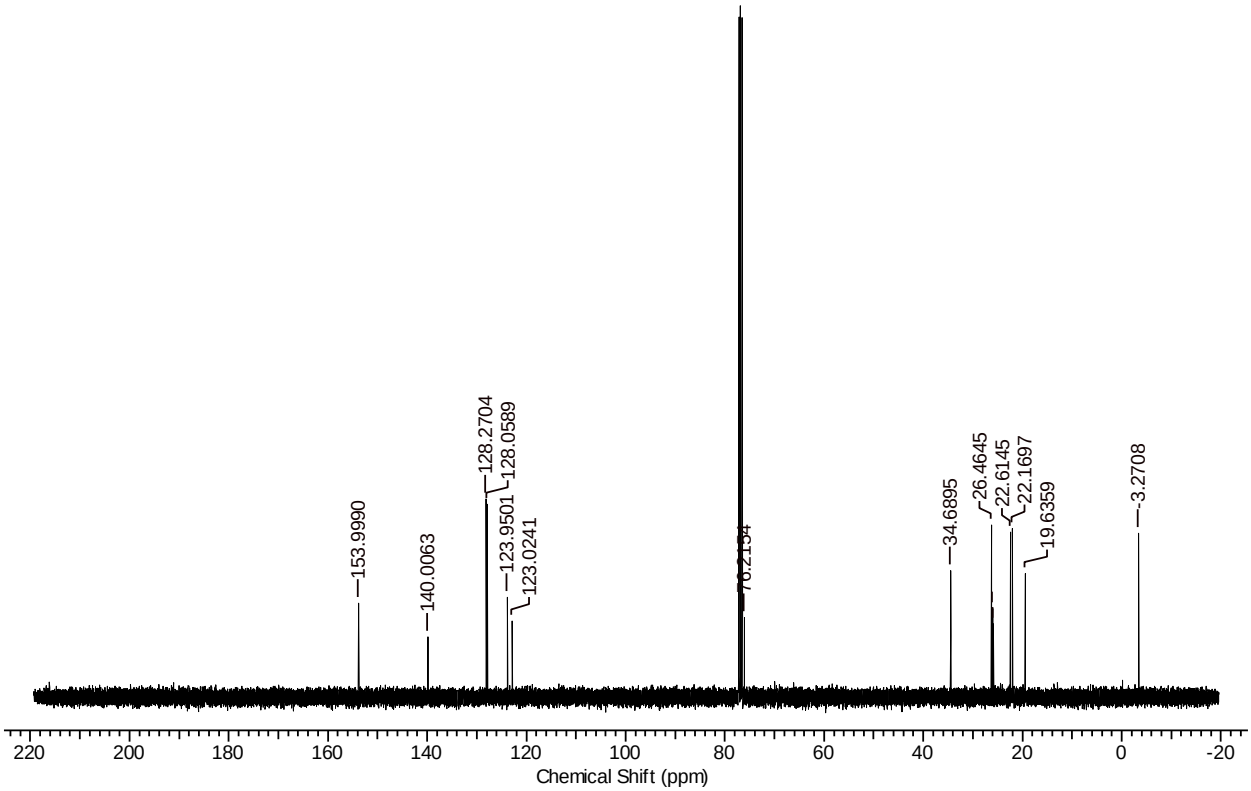
2.01
1.02
2.01
1.00
1.00
2.02
3.94
5.99
1.26
5.80
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

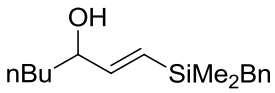

154.6151
139.9771
128.2850
128.0553
123.9647
123.3012
72.5732
37.4056
26.1437
22.0421
-3.3182
220
200
180
160
140
120
100
80
60
40
20
0
-20
Chemical Shift (ppm)


OH
SiMe2Bn

2e

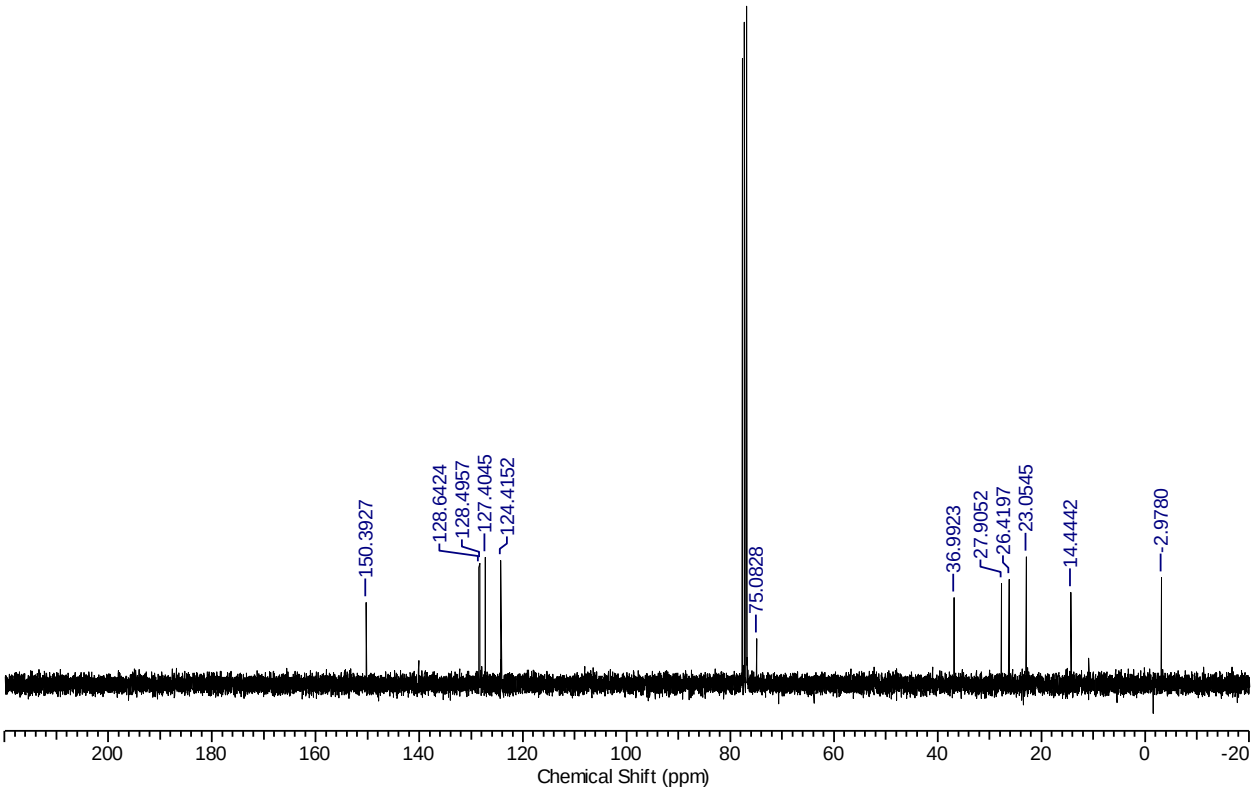
1.95 0.98 1.95
1.00 1.00
1.98
2.46 20.00
5.78
9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)

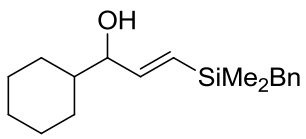

153.9990
140.0063
128.2704
128.0589
123.9501
123.0241
77.2154
34.6895
26.4645
22.6145
22.1697
19.6359
-3.2708
220 200 180 160 140 120 100 80 60 40 20 0 -20
Chemical Shift (ppm)


OH
nBu
SiMe2Bn

2f

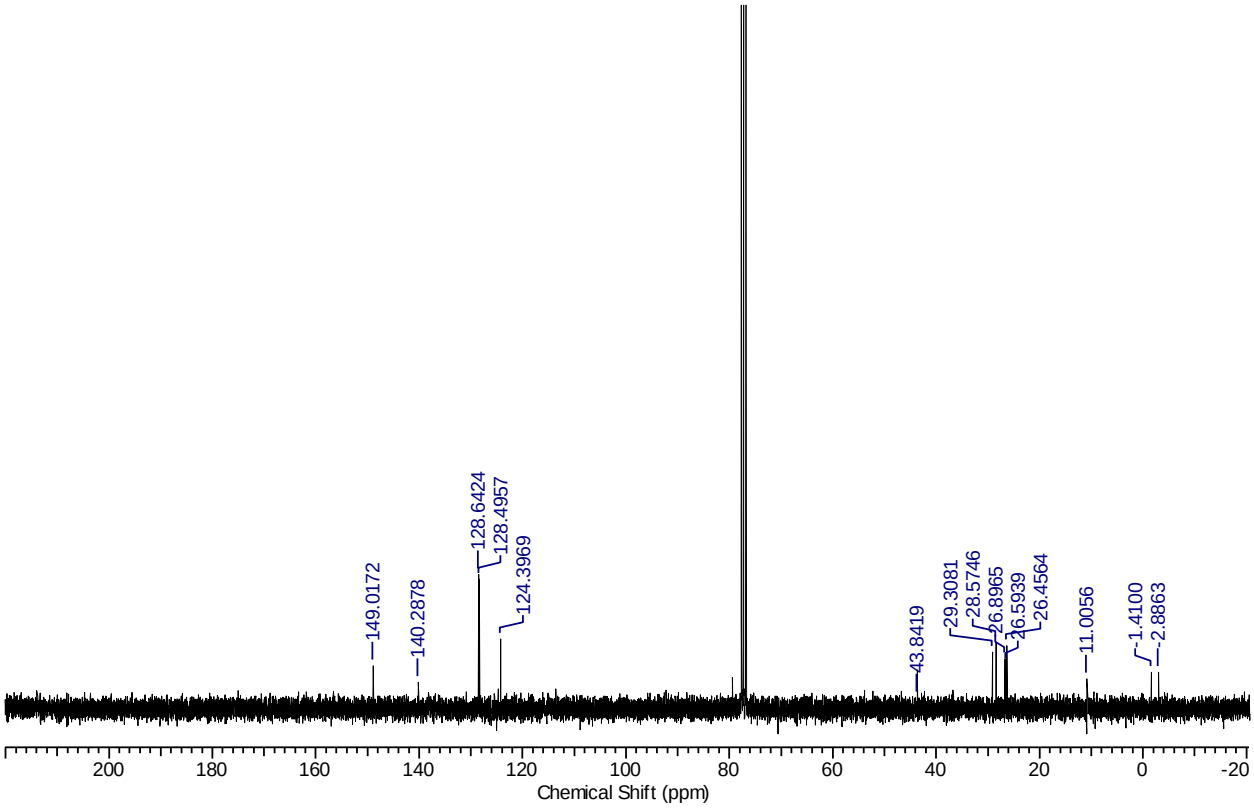
2.15 1.24 2.03
1.02 1.00
0.99
1.90 2.23 0.74 3.85 3.46
5.52
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0 -0.5 -1.0
Chemical Shift (ppm)

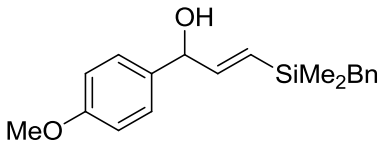

150.3927
128.6424
128.4957
127.4045
124.4152
75.0828
36.9923
27.9052
26.4197
23.0545
14.4442
-2.9780
200 180 160 140 120 100 80 60 40 20 0 -20
Chemical Shift (ppm)


OH
SiMe2Bn

2g

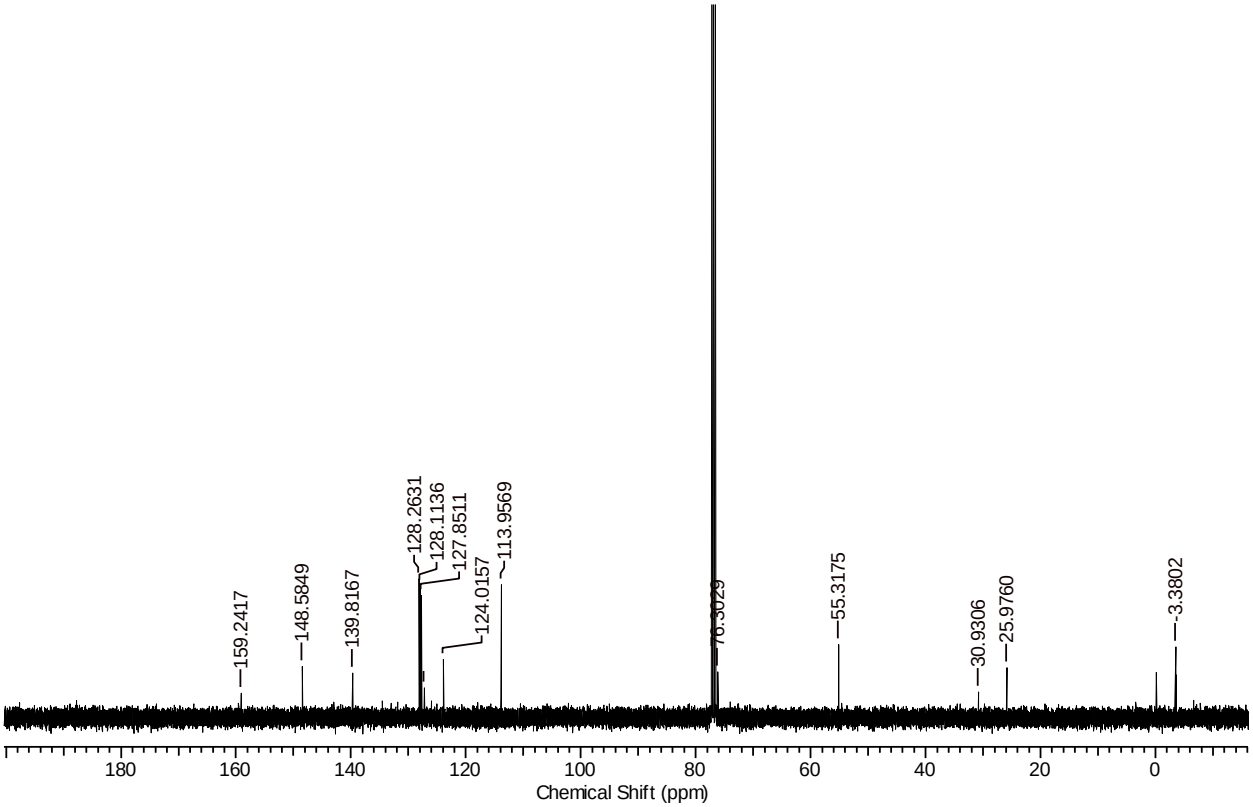
1.88 0.96 1.94 0.94 1.00 0.93 1.91 2.98 0.93 0.91 1.09 3.14 2.12 5.53
8 7 6 5 4 3 2 1 0 -1
Chemical Shift (ppm)

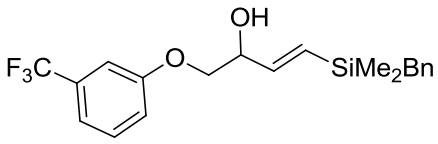

149.0172
140.2878
128.6424
128.4957
124.3969
43.8419
29.3081
28.5746
26.8965
26.5939
26.4564
11.0056
-1.4100
-2.8863
200 180 160 140 120 100 80 60 40 20 0 -20
Chemical Shift (ppm)


OH
SiMe2Bn
MeO

2h

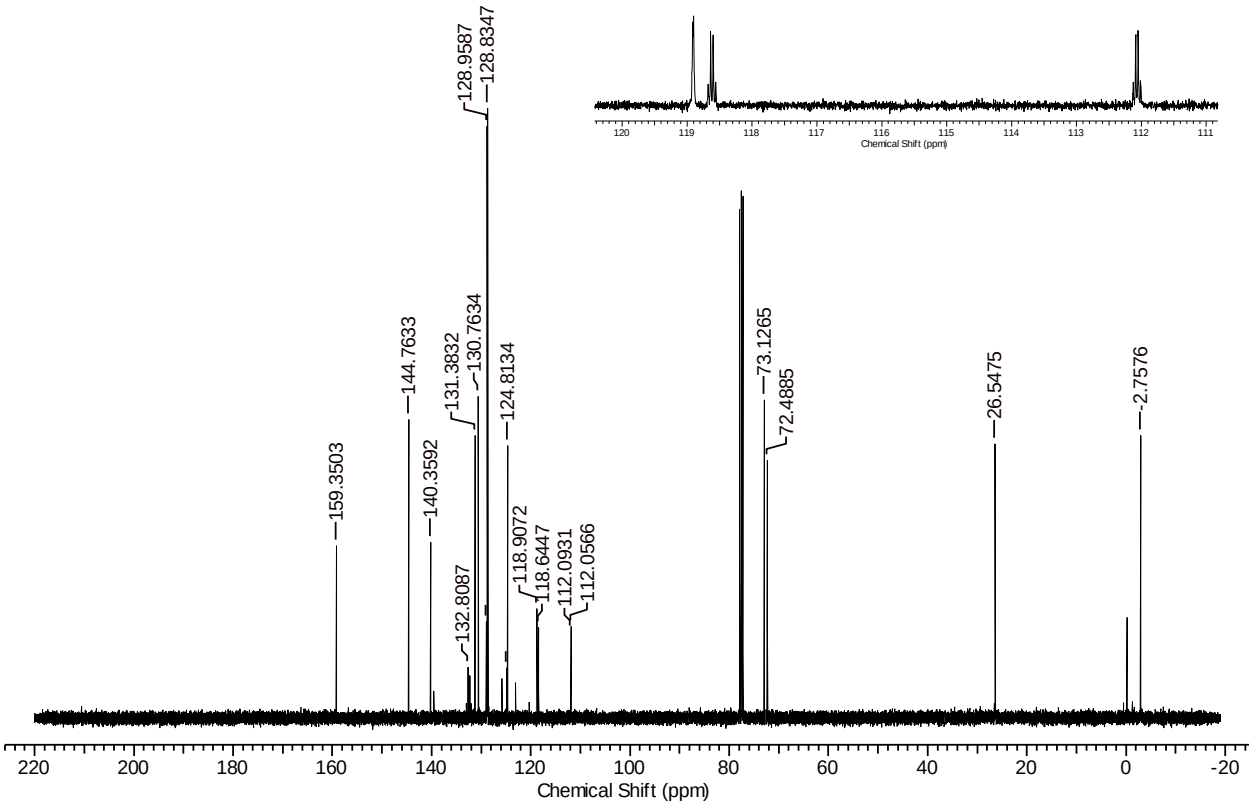
4.10 1.42 2.03 2.24 1.02 1.00 0.99 3.24 2.08 0.96 5.88
8 7 6 5 4 3 2 1 0 -1
Chemical Shift (ppm)

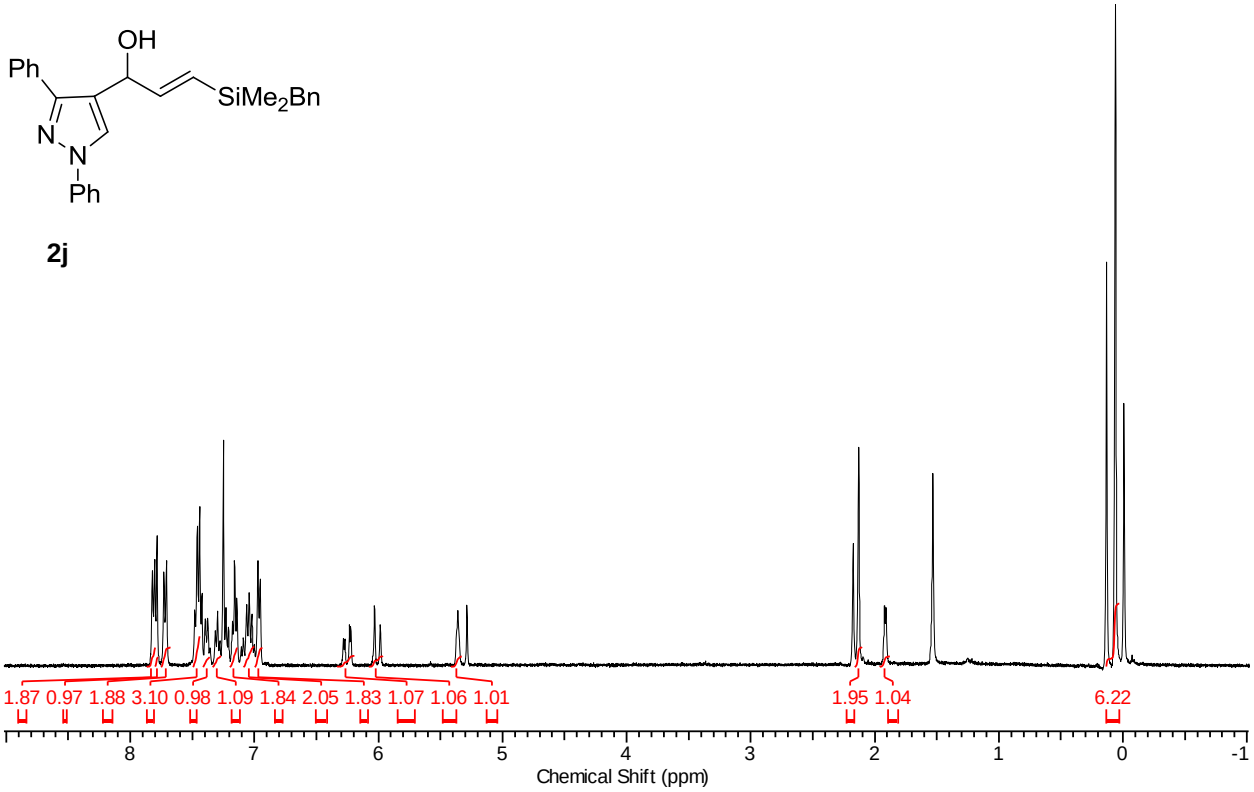

159.2417
148.5849
139.8167
128.2631
128.1136
127.8511
124.0157
113.9569
77.3029
55.3175
30.9306
25.9760
-3.3802
180 160 140 120 100 80 60 40 20 0
Chemical Shift (ppm)


OH
F3C
O
SiMe2Bn

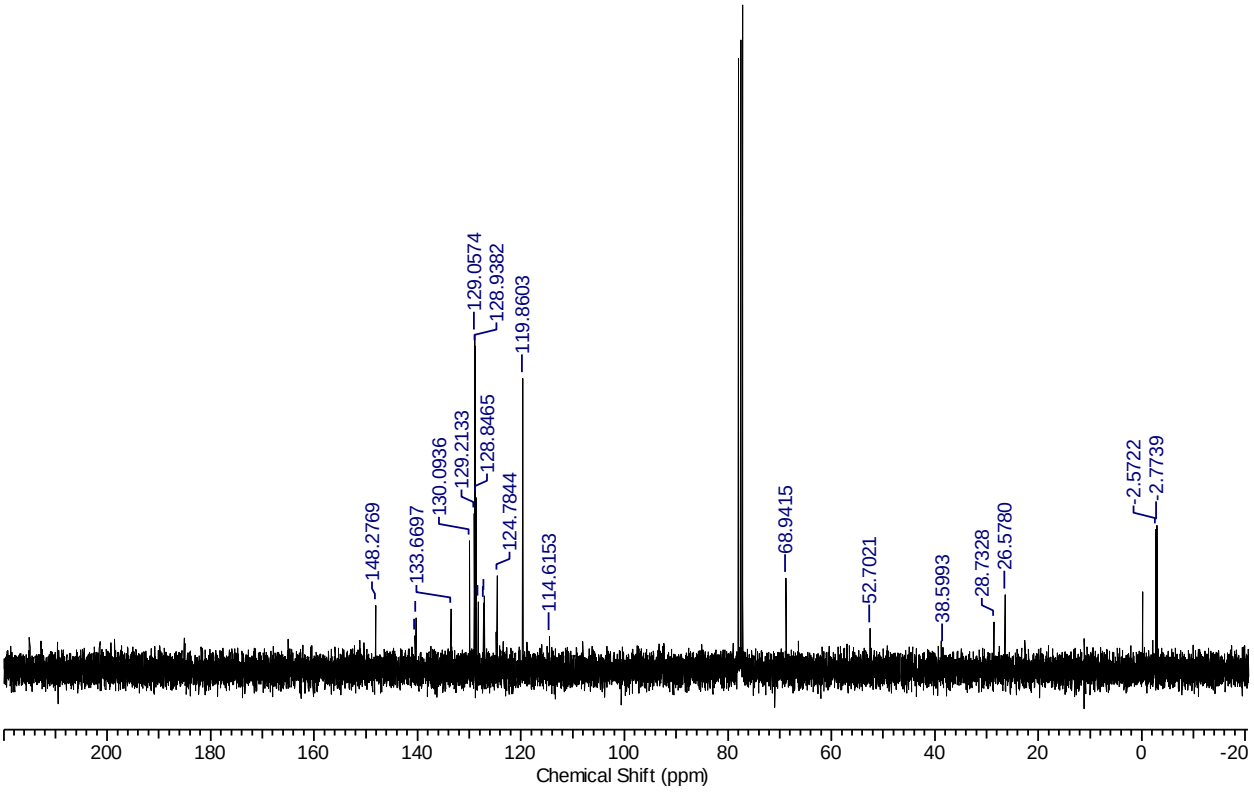
2i

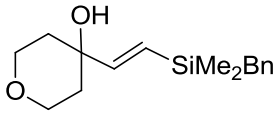
1.09
3.06
2.63
1.94
2.00
1.03
0.98
1.12
1.98
5.80
Chemical Shift (ppm)


128.9587
128.8347
144.7633
131.3832
130.7634
124.8134
140.3592
159.3503
132.8087
118.9072
118.6447
112.0931
112.0566
73.1265
72.4885
26.5475
2.7576
Chemical Shift (ppm)


OH
Ph
SiMe2Bn
N
N
Ph
1.87 0.97 1.88 3.10 0.98 1.09 1.84 2.05 1.83 1.07 1.06 1.01
1.95 1.04
6.22
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

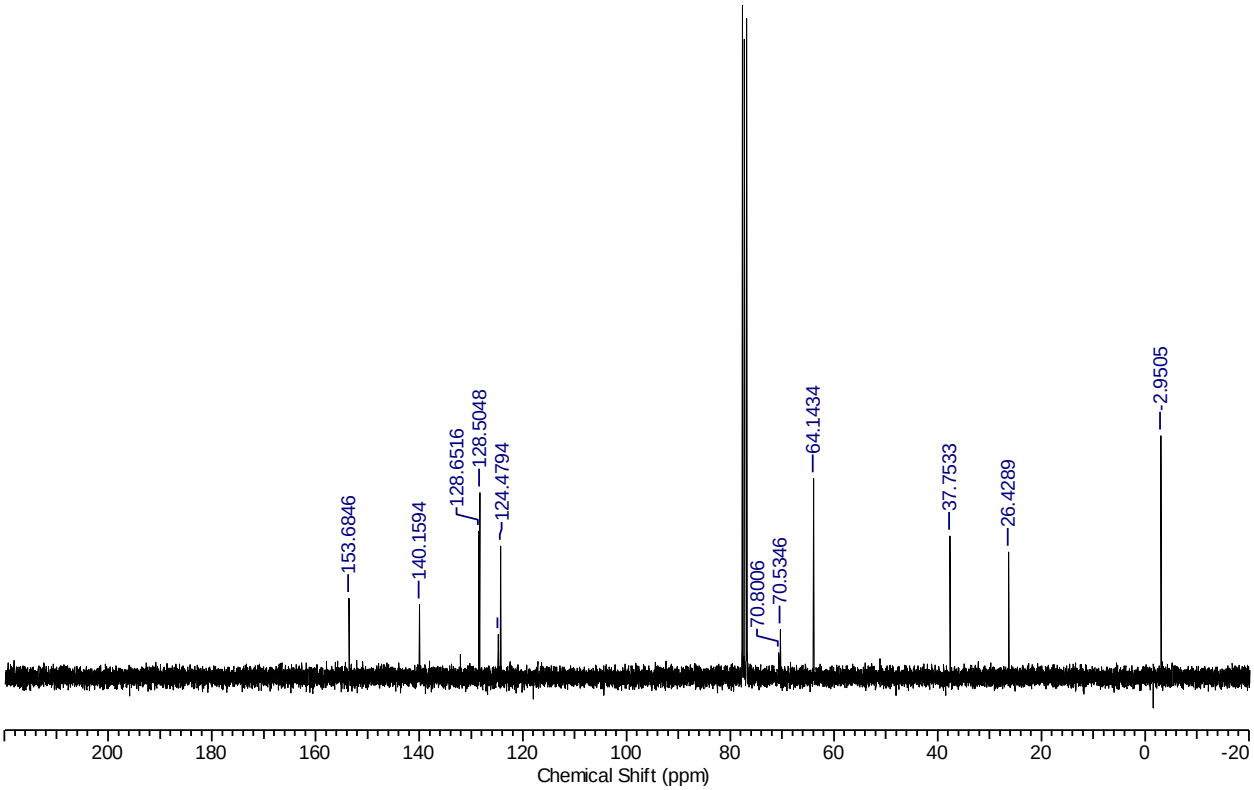
2j

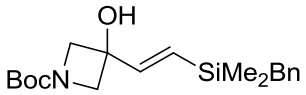

148.2769
133.6697
130.0936
129.2133
129.0574
128.9382
128.8465
124.7844
119.8603
114.6153
68.9415
52.7021
38.5993
28.7328
26.5780
2.5722
2.7739
200
180
160
140
120
100
80
60
40
20
0
-20
Chemical Shift (ppm)


OH
O
SiMe2Bn

2k

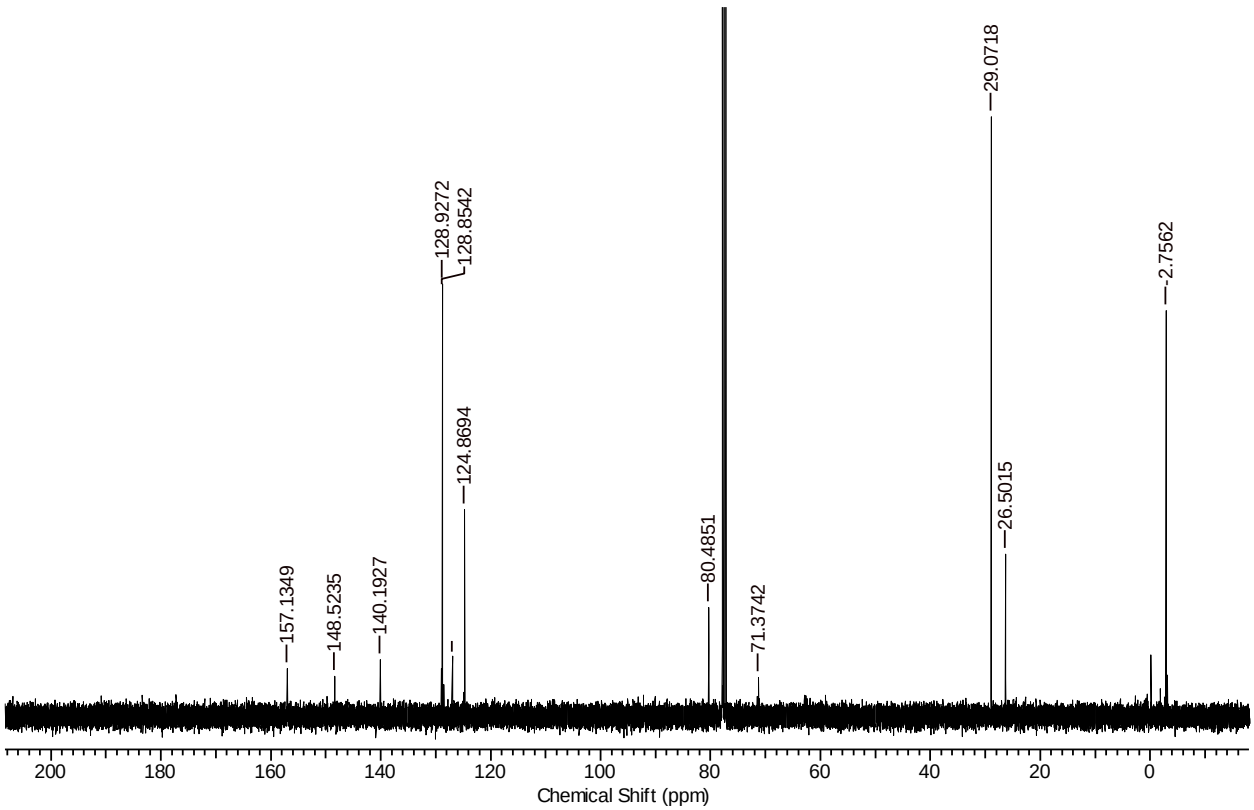
1.91
0.99
1.93
0.97
1.00
4.00
1.99
2.03
2.11
5.71
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

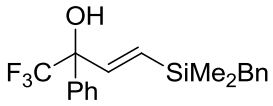

153.6846
140.1594
128.6516
128.5048
124.4794
70.8006
70.5346
64.1434
37.7533
26.4289
2.9505
200
180
160
140
120
100
80
60
40
20
0
-20
Chemical Shift (ppm)


OH
BocN
SiMe2Bn

2l

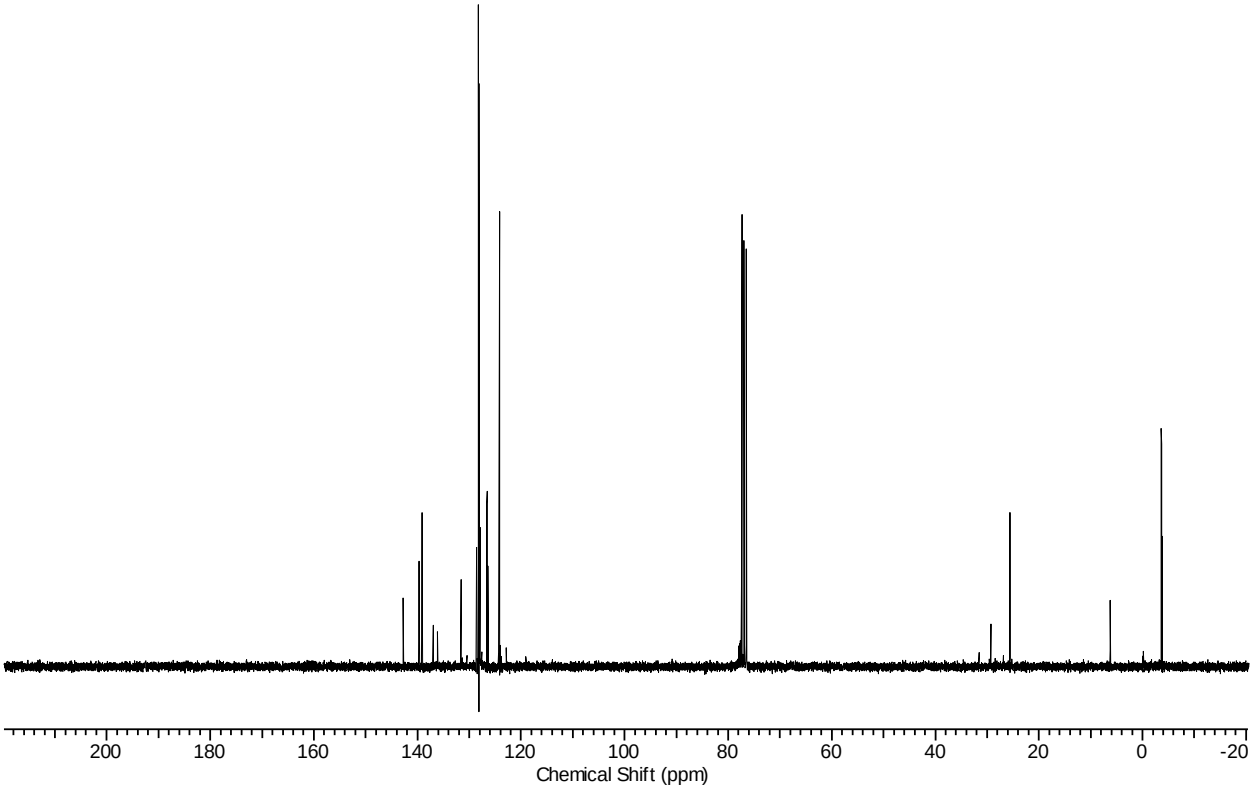
2.31
1.19
2.29
1.00
1.09
3.91
2.27
9.70
6.02
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

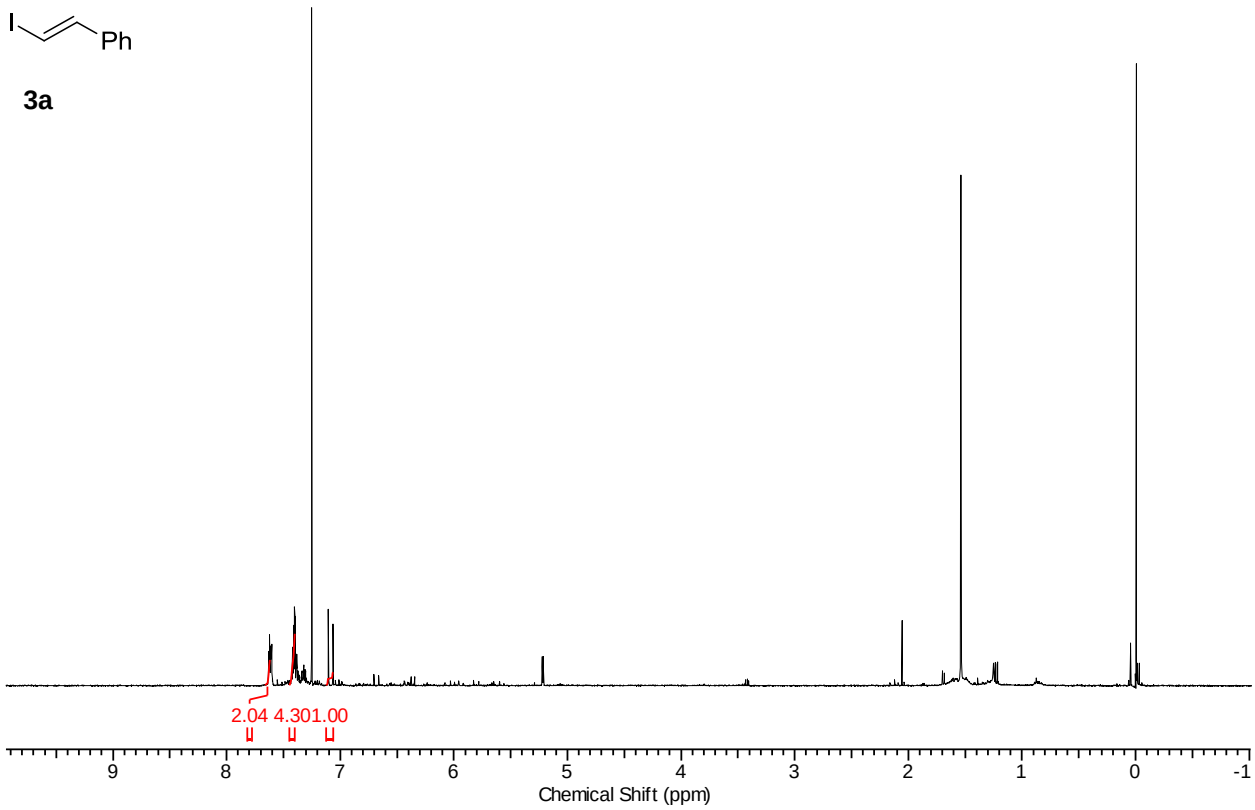

157.1349
148.5235
140.1927
128.9272
128.8542
124.8694
80.4851
71.3742
29.0718
26.5015
2.7562
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

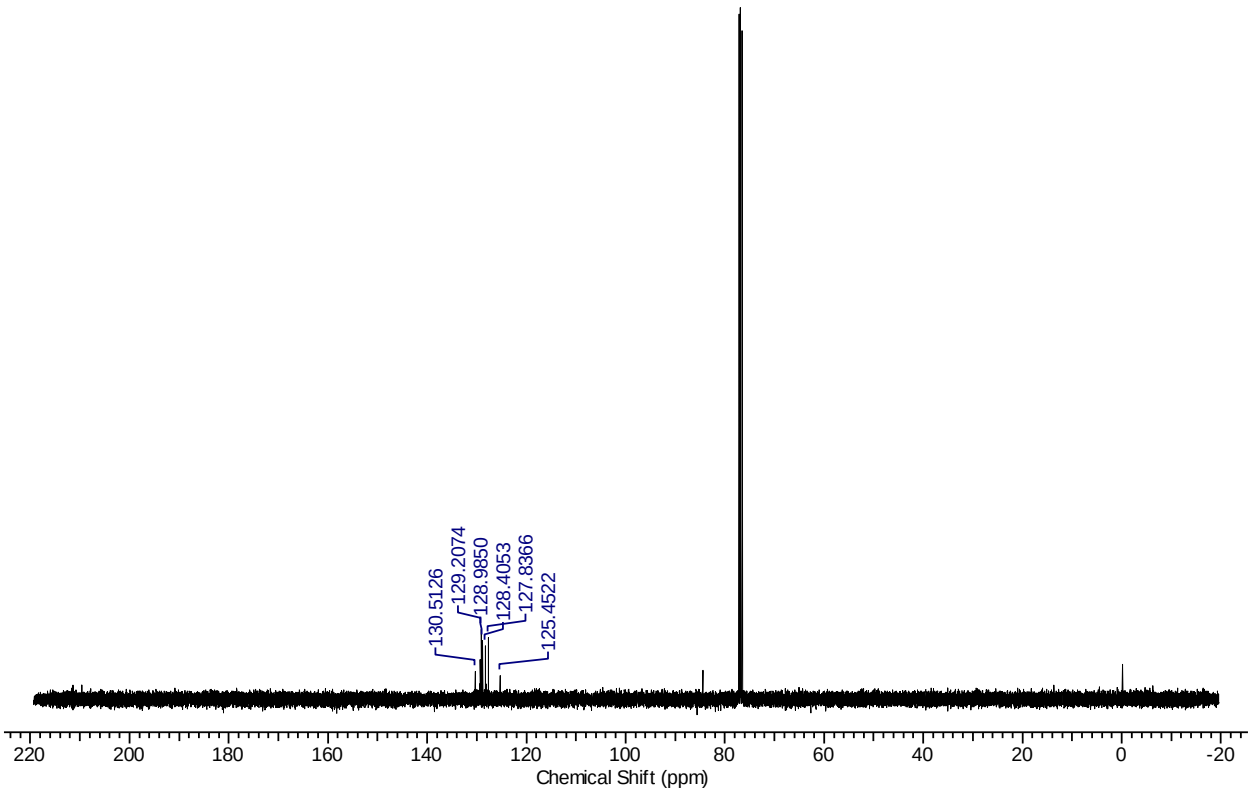

OH
F3C
Ph
SiMe2Bn

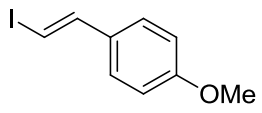
2m

8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)


200
180
160
140
120
100
80
60
40
20
0
-20
Chemical Shift (ppm)

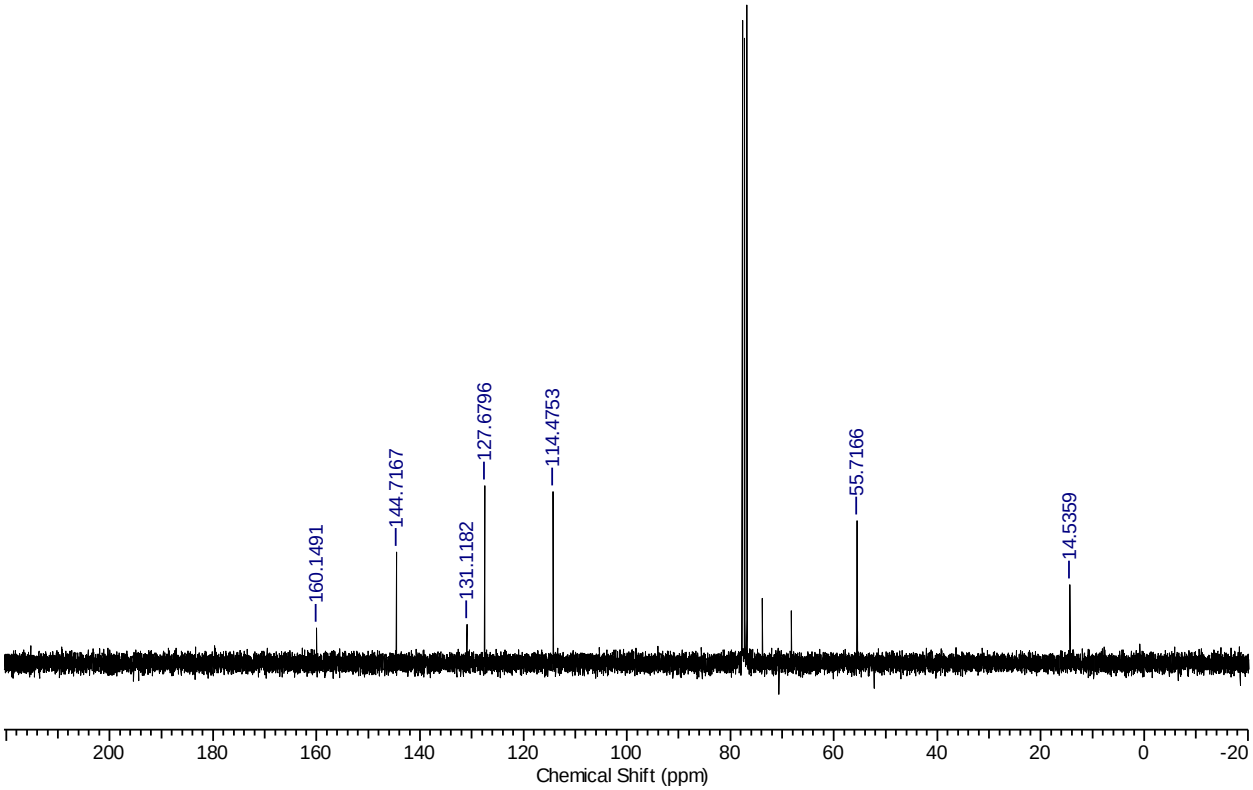

I
Ph
3a
2.04 4.30 1.00
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

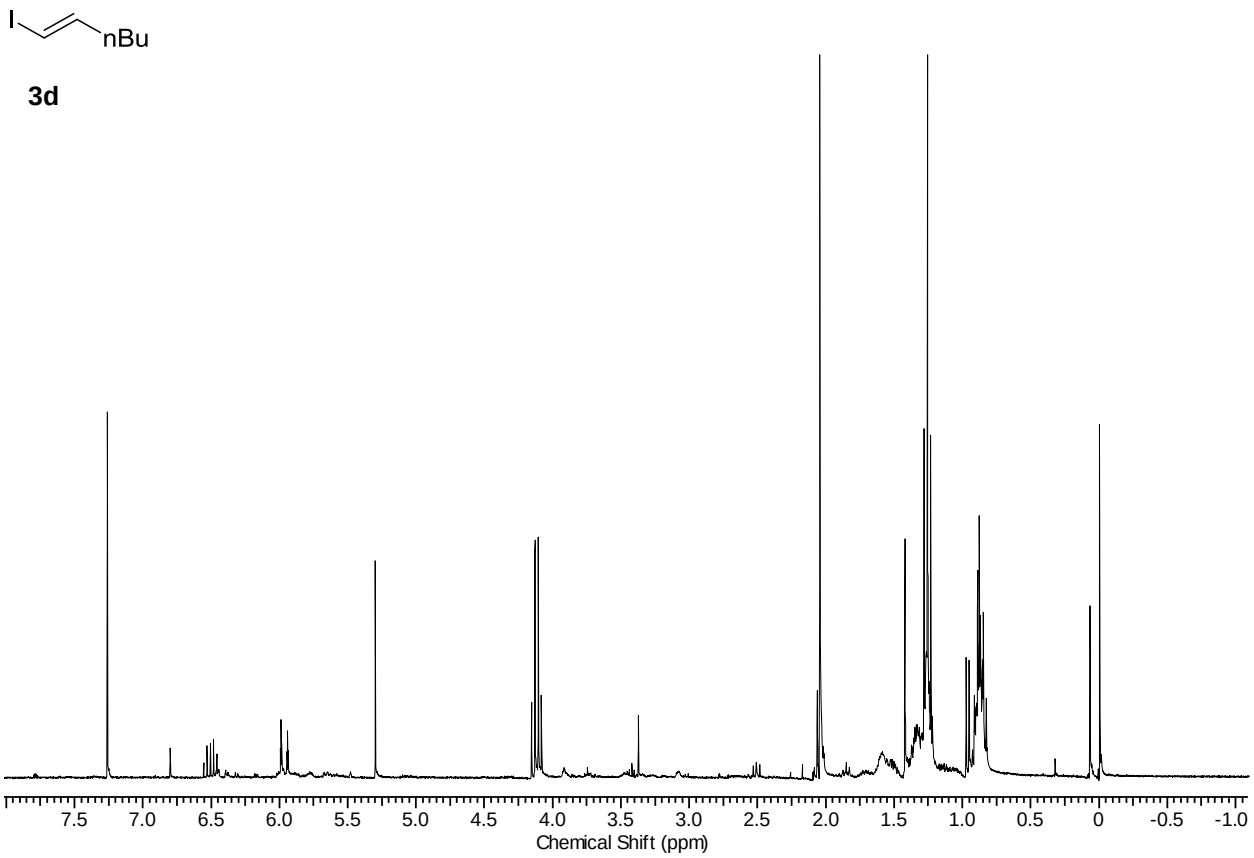

130.5126
129.2074
128.9850
128.4053
127.8366
125.4522
220
200
180
160
140
120
100
80
60
40
20
0
-20
Chemical Shift (ppm)

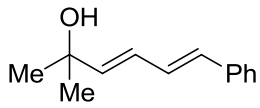

I
OMe

3b

0.91
1.87
1.94
1.00
2.83
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

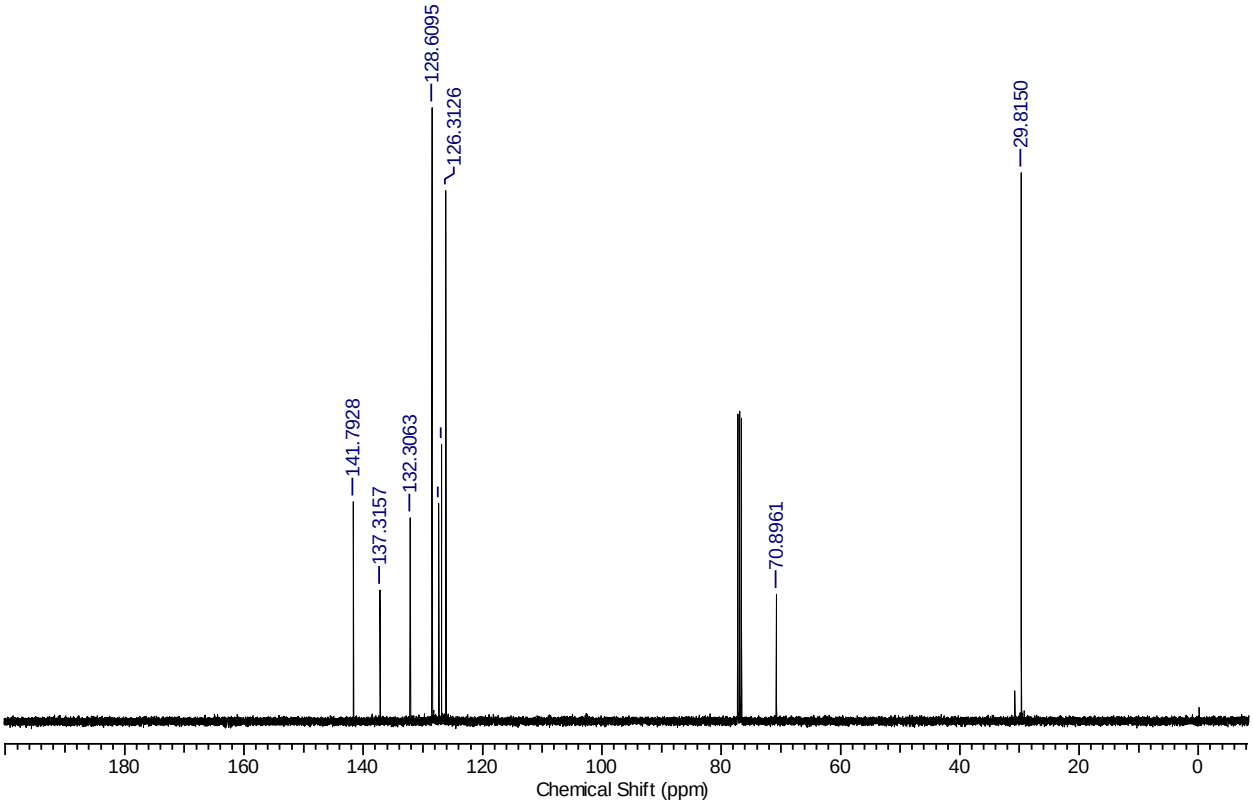

160.1491
144.7167
131.1182
127.6796
114.4753
55.7166
14.5359
200
180
160
140
120
100
80
60
40
20
0
-20
Chemical Shift (ppm)

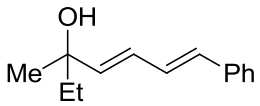

I
nBu
3d
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
-0.5
-1.0
Chemical Shift (ppm)


OH
Me
Me
Ph

4a

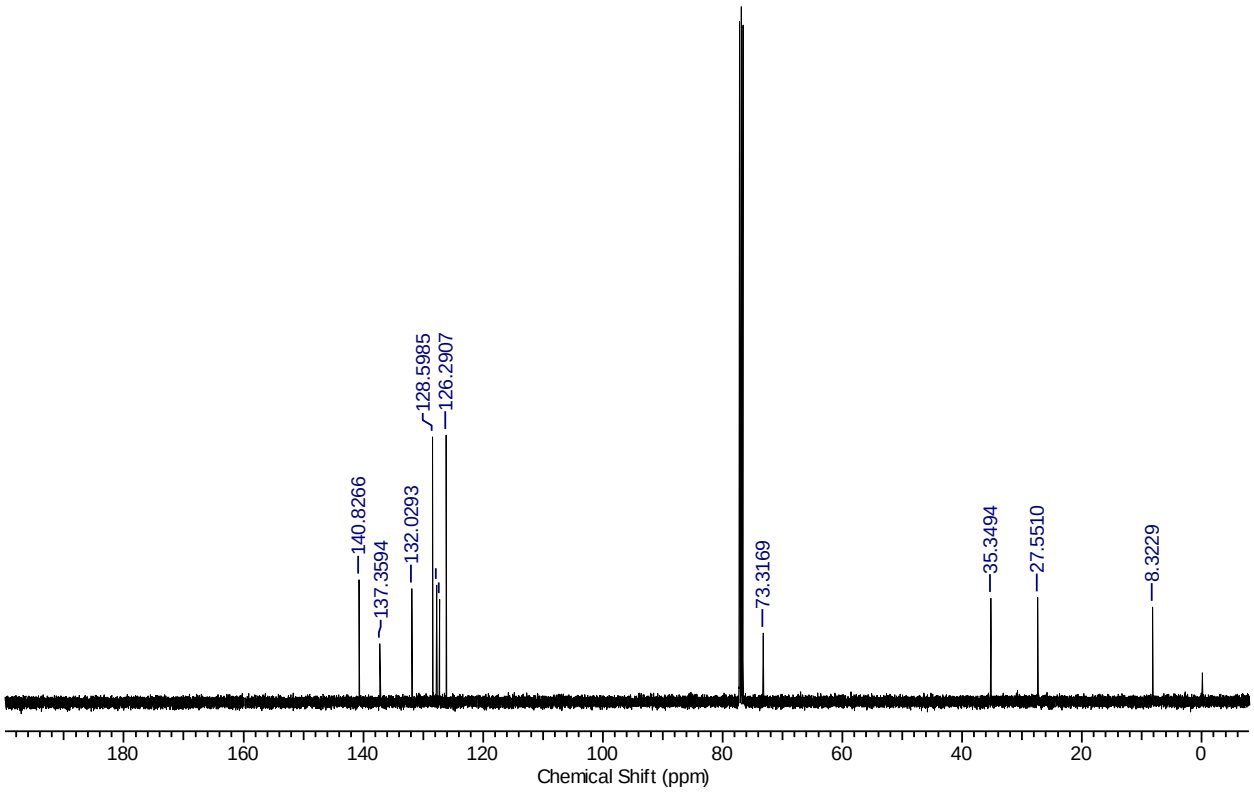
2.10 2.23 1.10 1.03 1.00 1.05 1.00
0.96 6.15
8 7 6 5 4 3 2 1 0 -1
Chemical Shift (ppm)

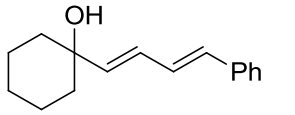

128.6095
126.3126
29.8150
141.7928
137.3157
132.3063
70.8961
180 160 140 120 100 80 60 40 20 0
Chemical Shift (ppm)


OH
Me
Et
Ph

4b

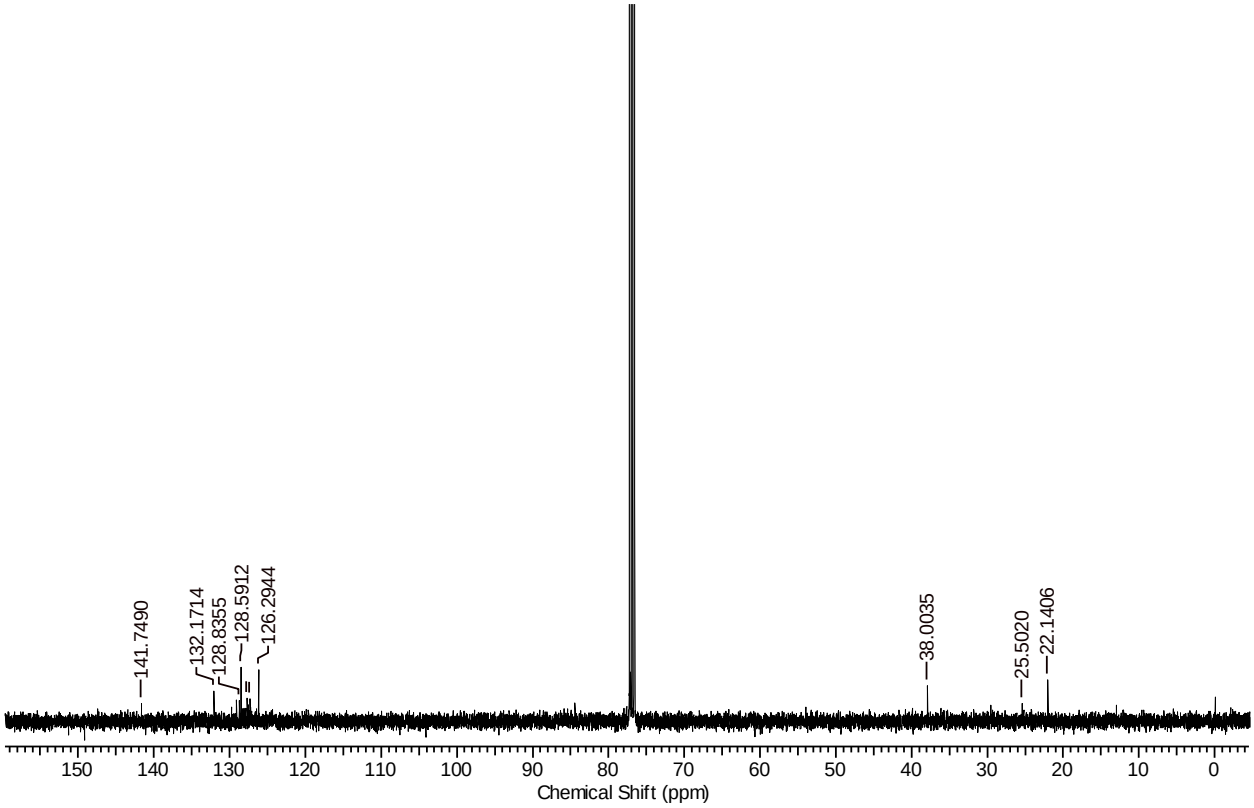
2.00 2.28 1.23 1.02 1.00 1.02 1.00
2.08 0.89 2.95 3.17
8 7 6 5 4 3 2 1 0 -1
Chemical Shift (ppm)

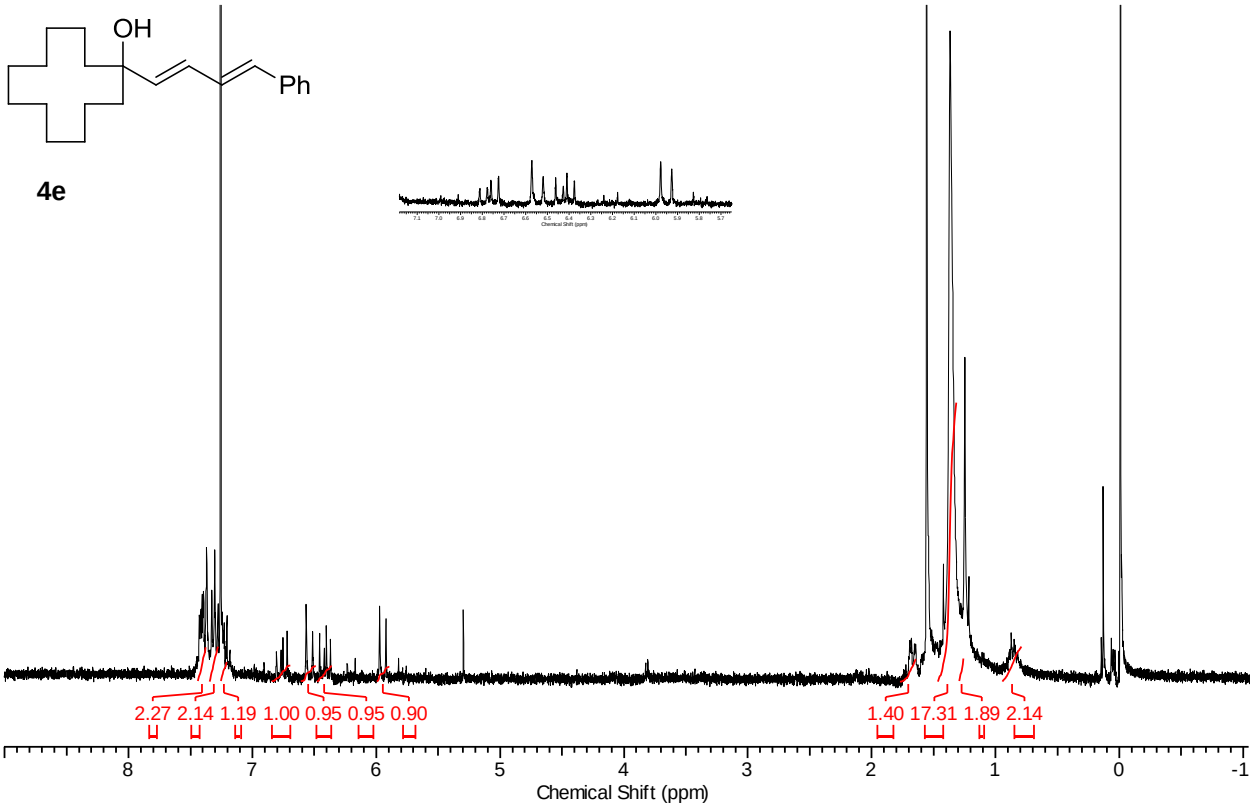

140.8266
137.3594
132.0293
128.5985
126.2907
73.3169
35.3494
27.5510
8.3229
180 160 140 120 100 80 60 40 20 0
Chemical Shift (ppm)

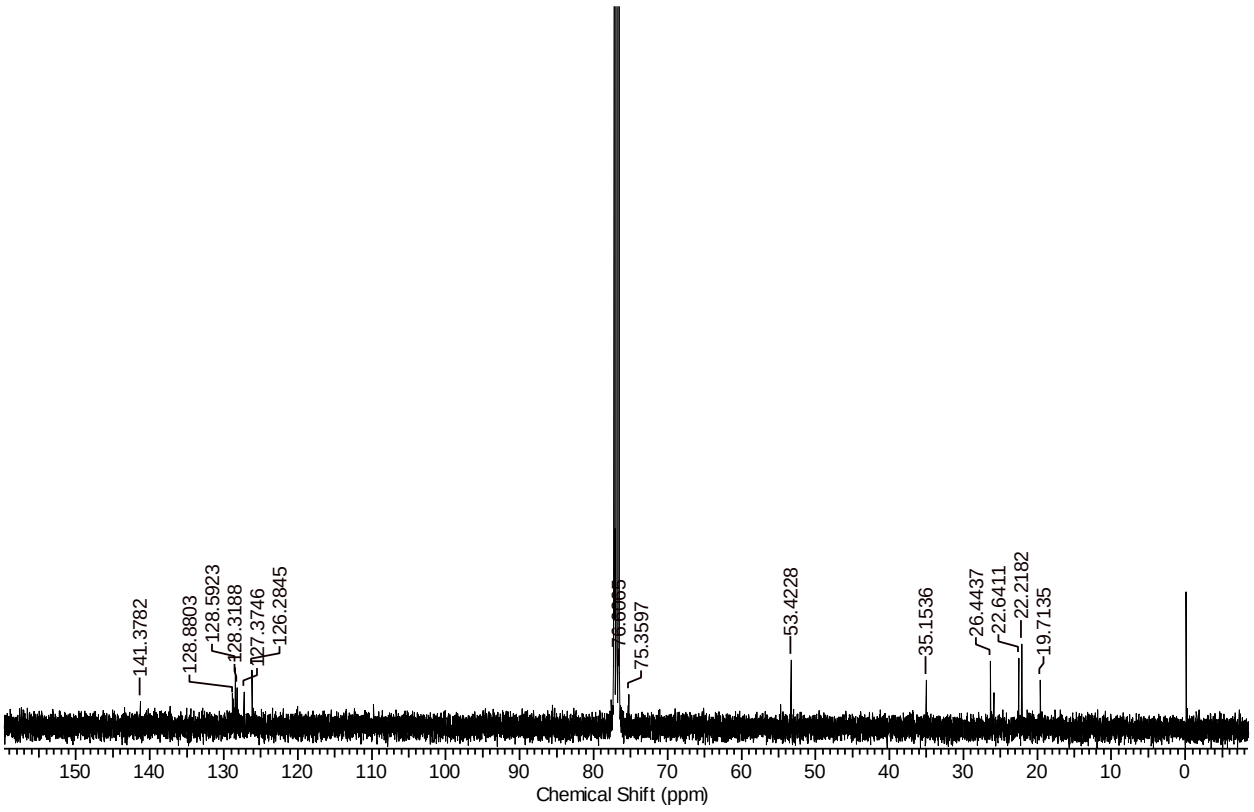

OH
Ph

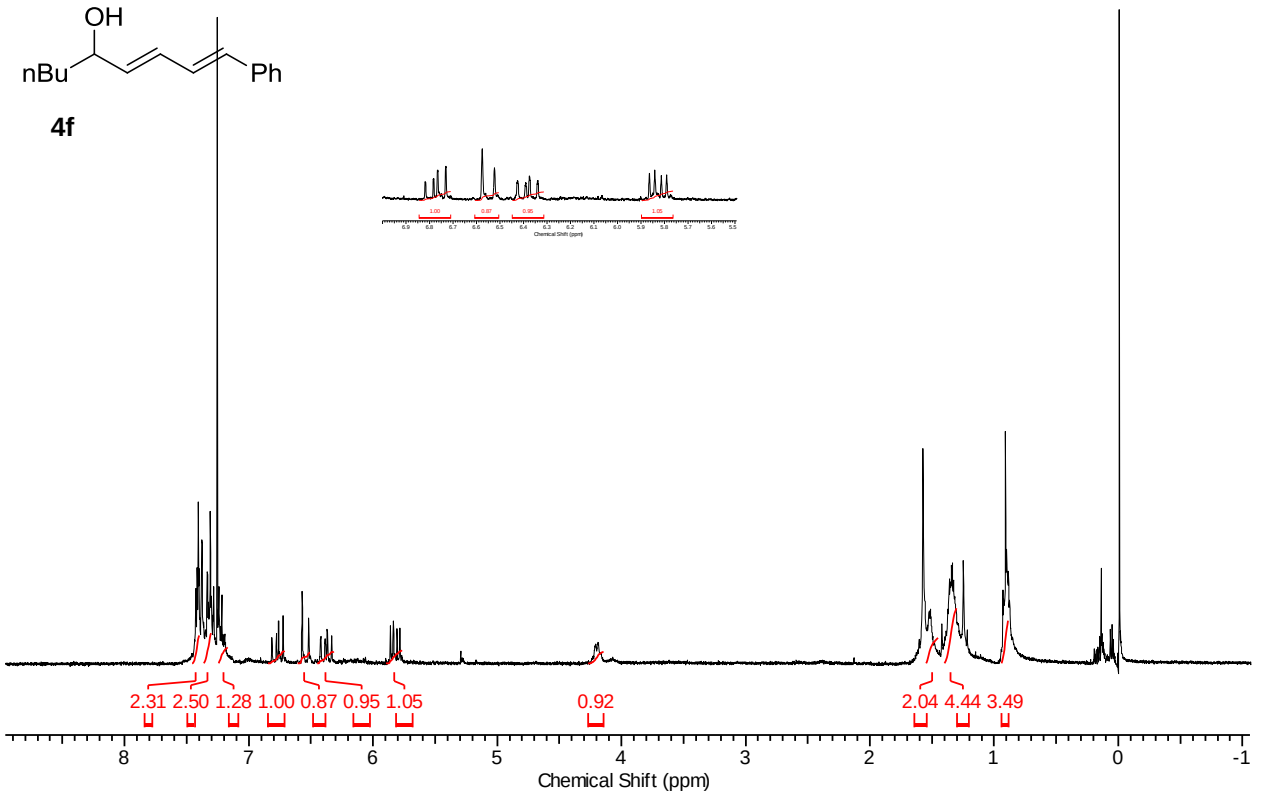
4d

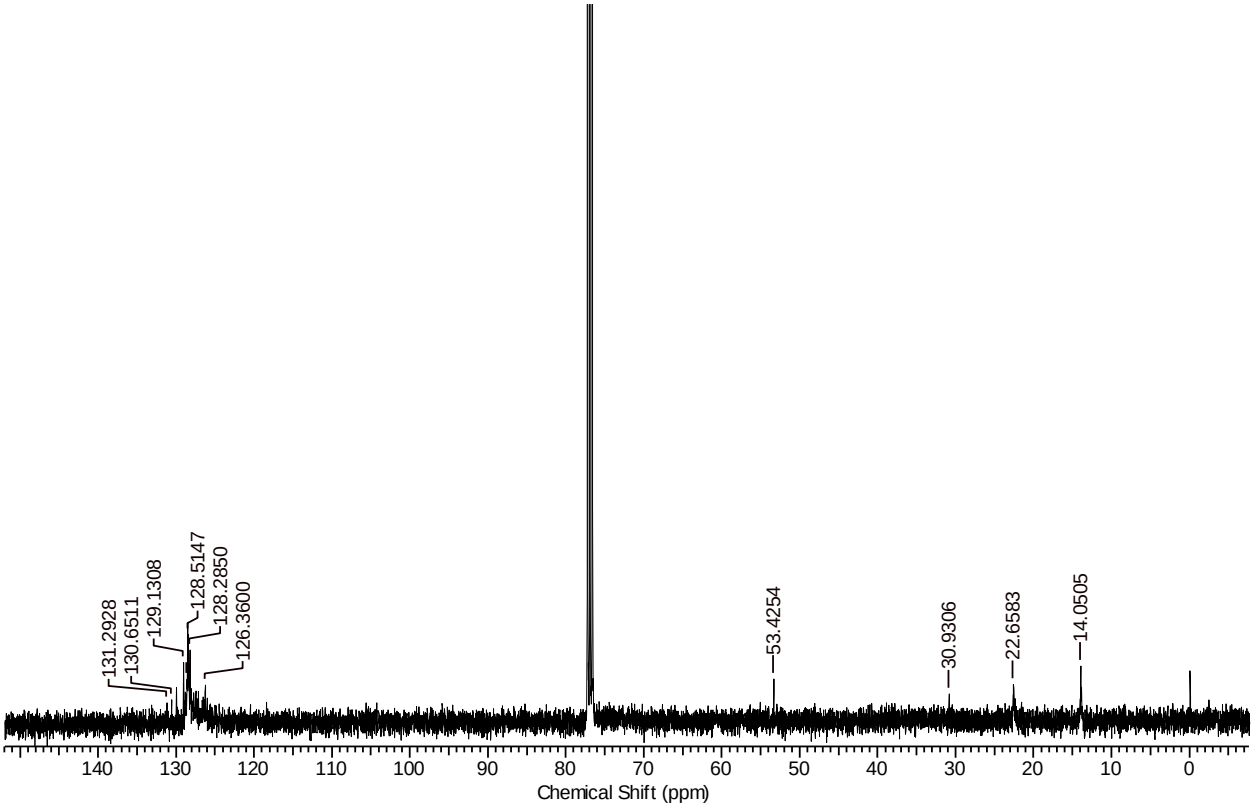
2.17 2.75 1.17 1.11 1.01 1.03 0.99
5.92 3.32 0.97
8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)

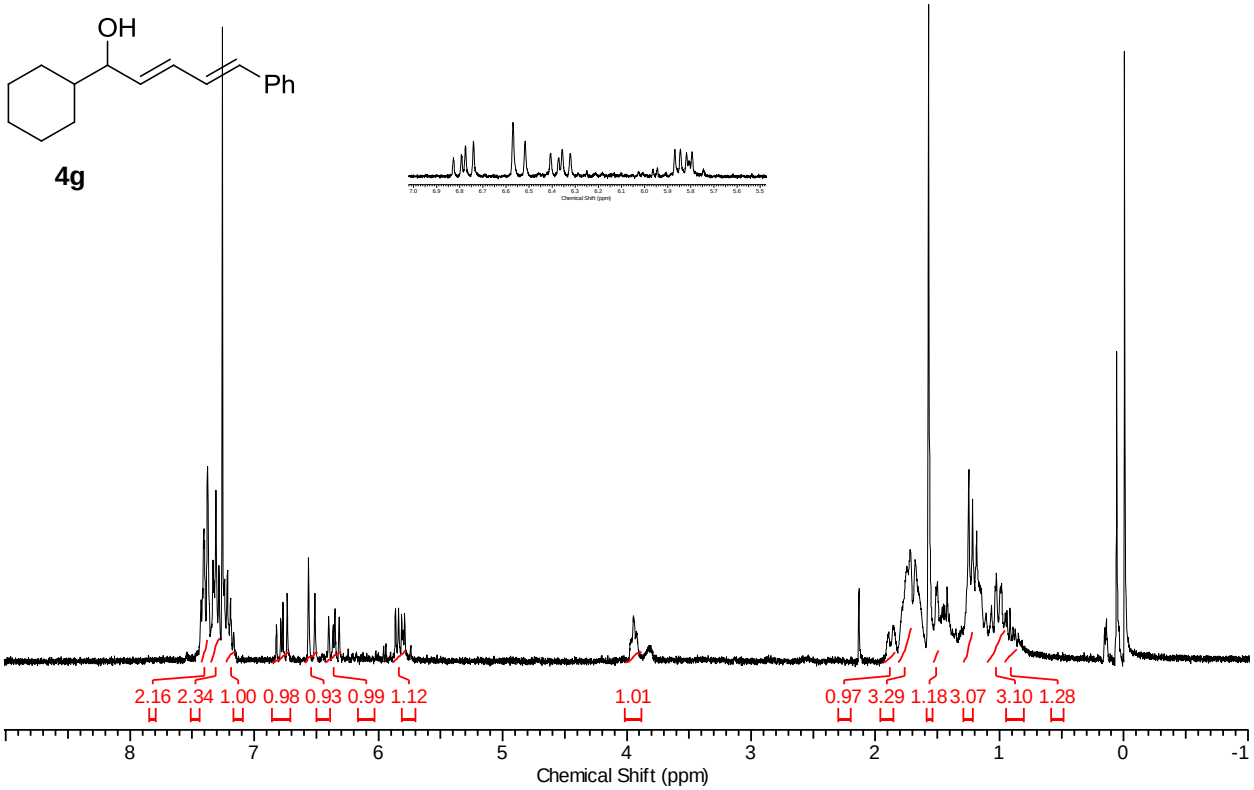

141.7490
132.1714
128.8355
128.5912
126.2944
38.0035
25.5020
22.1406
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
Chemical Shift (ppm)

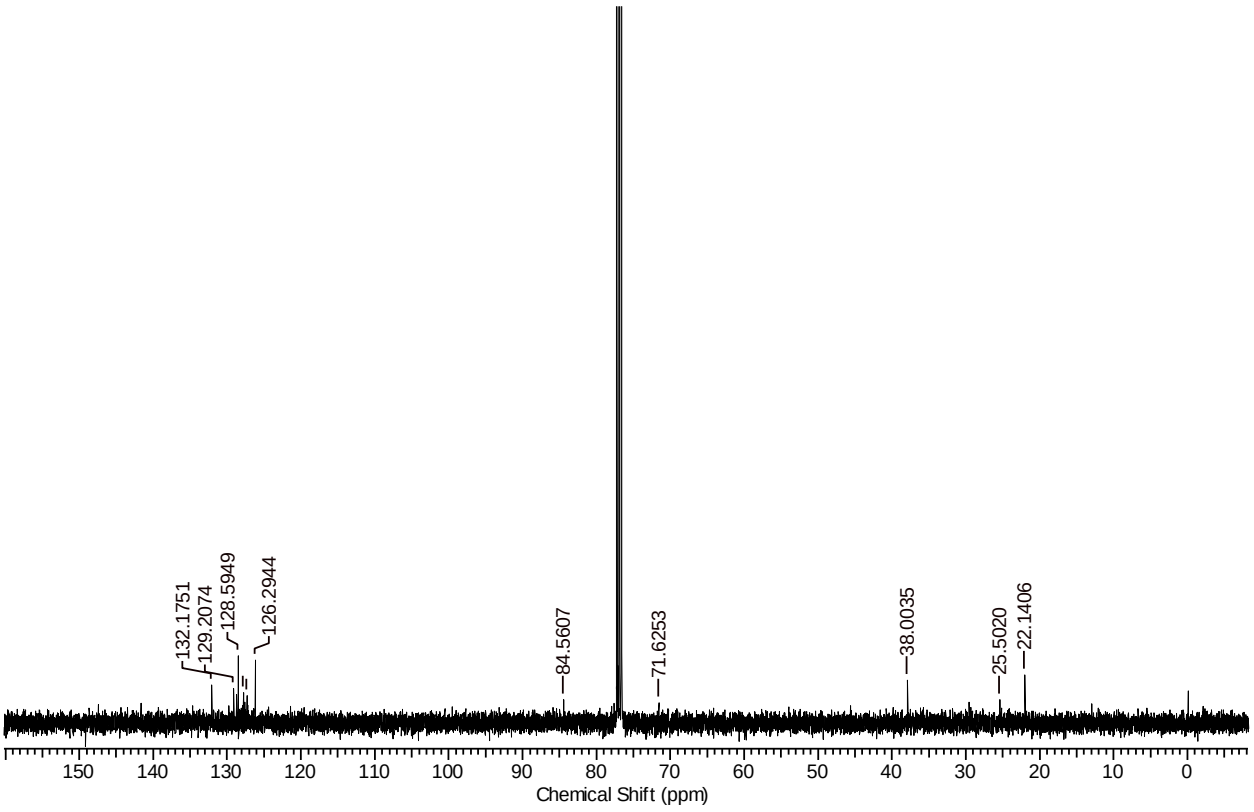

OH
Ph
4e
2.27 2.14 1.19 1.00 0.95 0.95 0.90
1.40 17.31 1.89 2.14
8 7 6 5 4 3 2 1 0 -1
Chemical Shift (ppm)

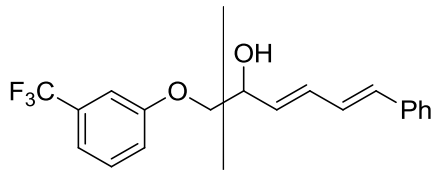

141.3782
128.8803
128.5923
128.3188
127.3746
126.2845
77.0000
75.3597
53.4228
35.1536
26.4437
22.6411
22.2182
19.7135
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
Chemical Shift (ppm)


OH
nBu
Ph
4f
2.31 2.50 1.28 1.00 0.87 0.95 1.05
0.92
2.04 4.44 3.49
Chemical Shift (ppm)

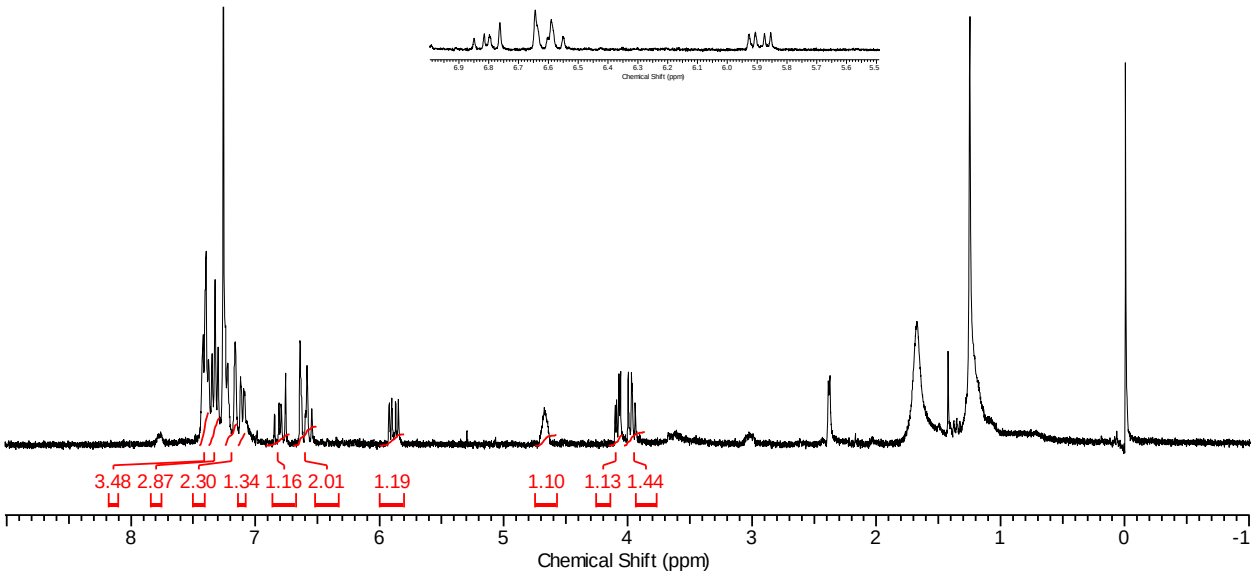

131.2928
130.6511
129.1308
128.5147
128.2850
126.3600
53.4254
30.9306
22.6583
14.0505
Chemical Shift (ppm)

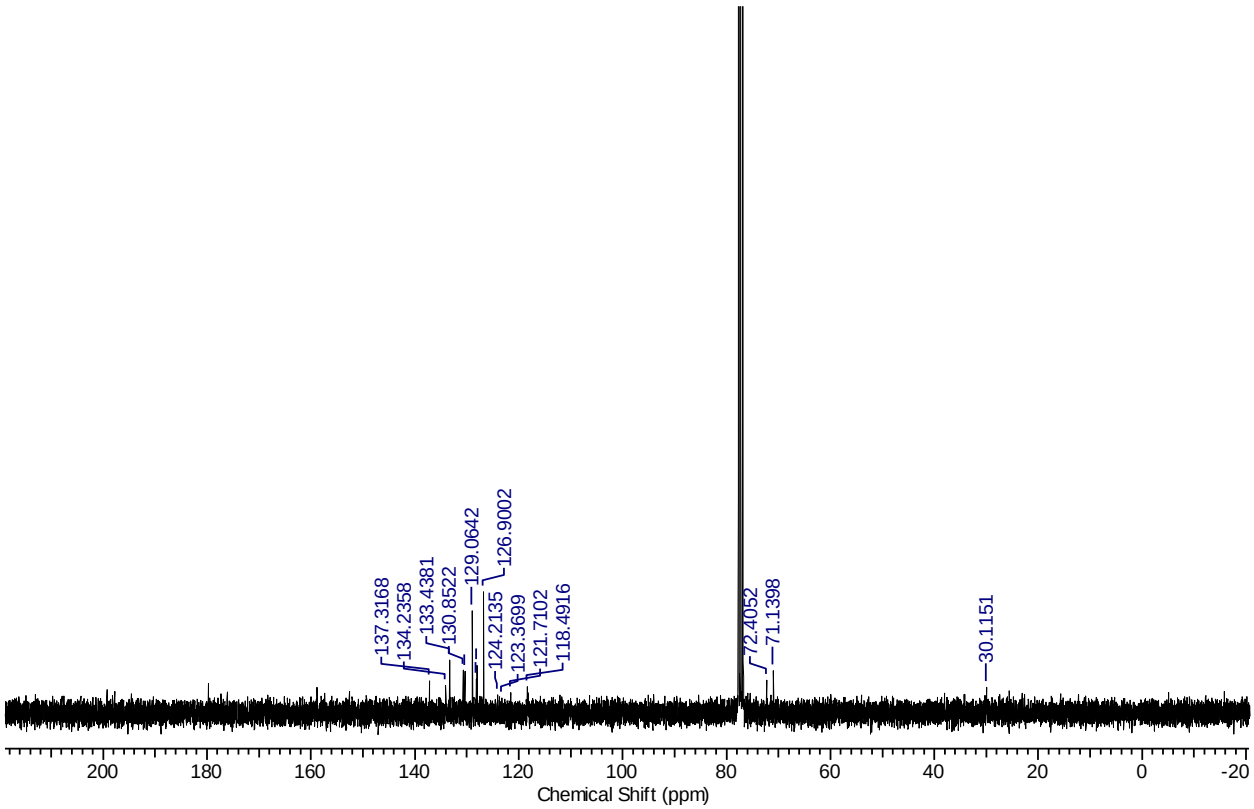

OH
Ph
4g
2.16 2.34 1.00 0.98 0.93 0.99 1.12
1.01
0.97 3.29 1.18 3.07 3.10 1.28
8 7 6 5 4 3 2 1 0 -1
Chemical Shift (ppm)

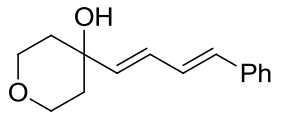

132.1751
129.2074
128.5949
126.2944
84.5607
71.6253
38.0035
25.5020
22.1406
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
Chemical Shift (ppm)


OH
F3C
O
Ph

4i

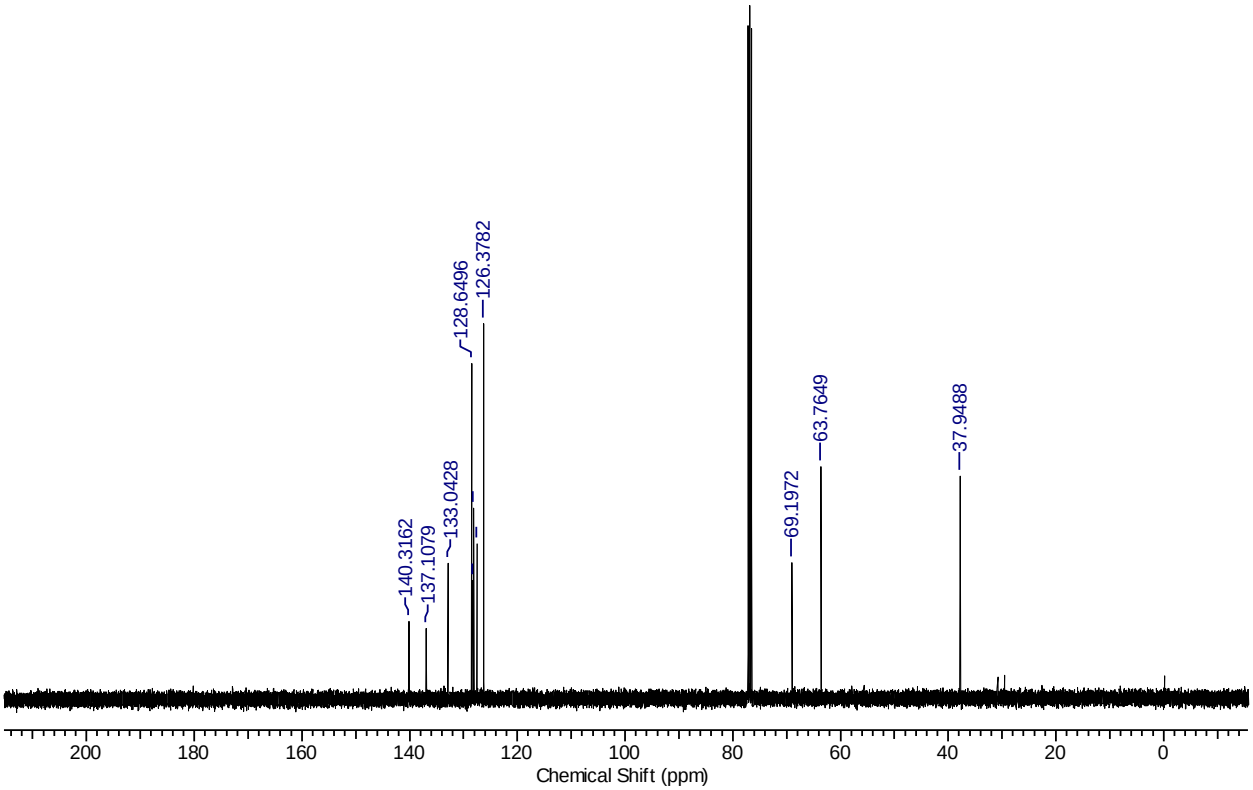

3.48 2.87 2.30 1.34 1.16 2.01 1.19 1.10 1.13 1.44
Chemical Shift (ppm)

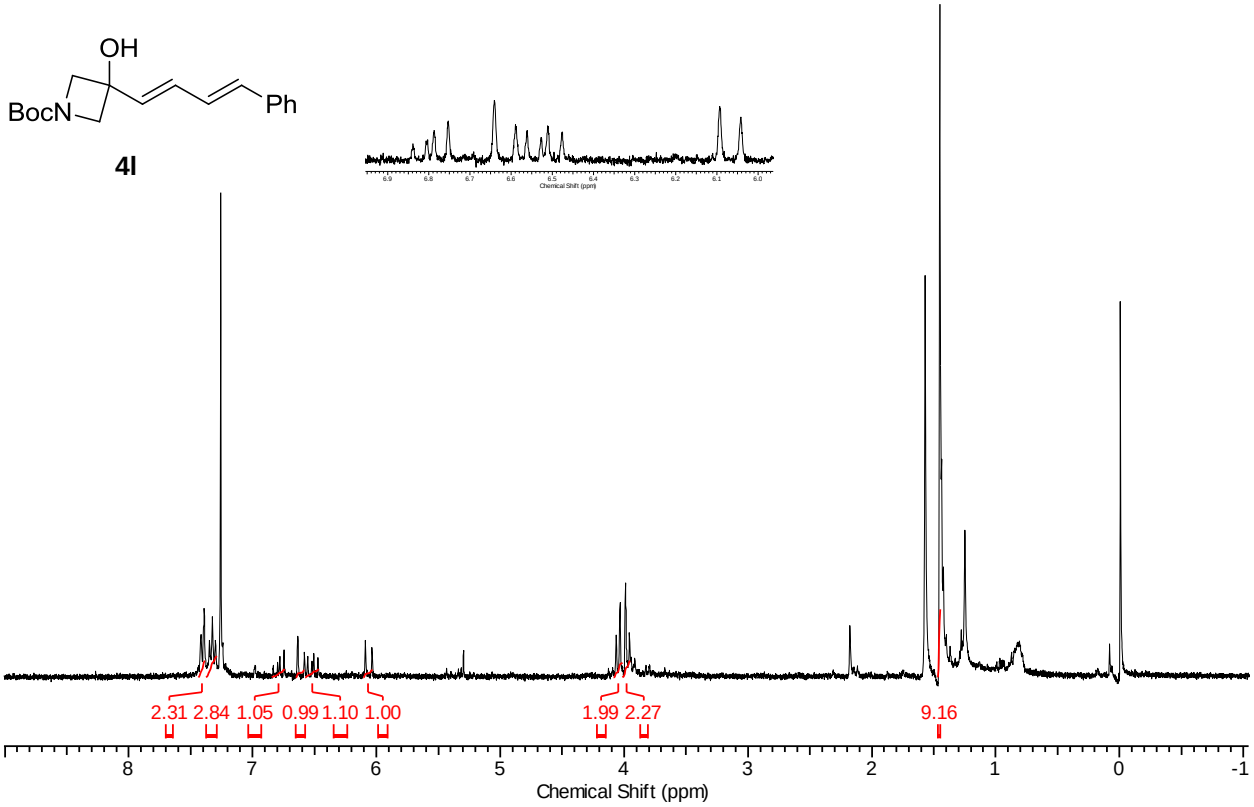

137.3168
134.2358
133.4381
130.8522
129.0642
126.9002
124.2135
123.3699
121.7102
118.4916
72.4052
71.1398
30.1151
Chemical Shift (ppm)

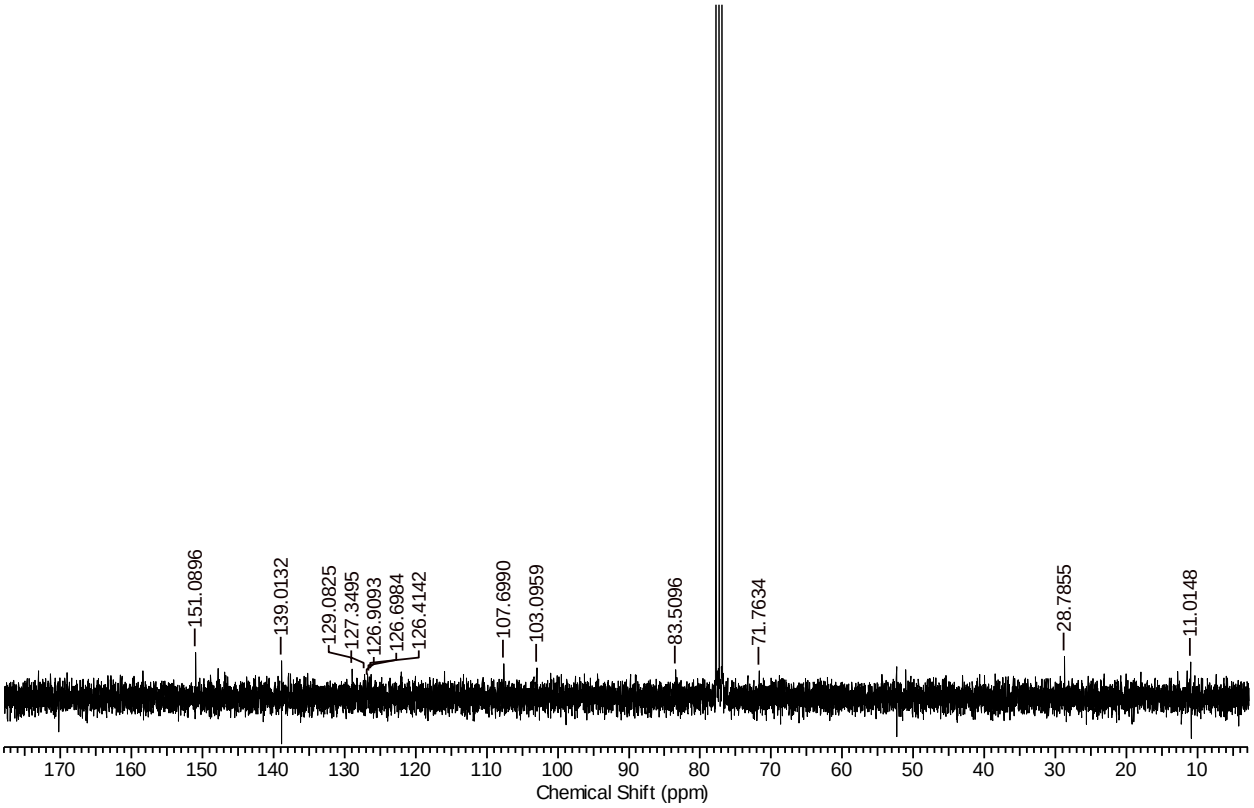

OH
O
Ph

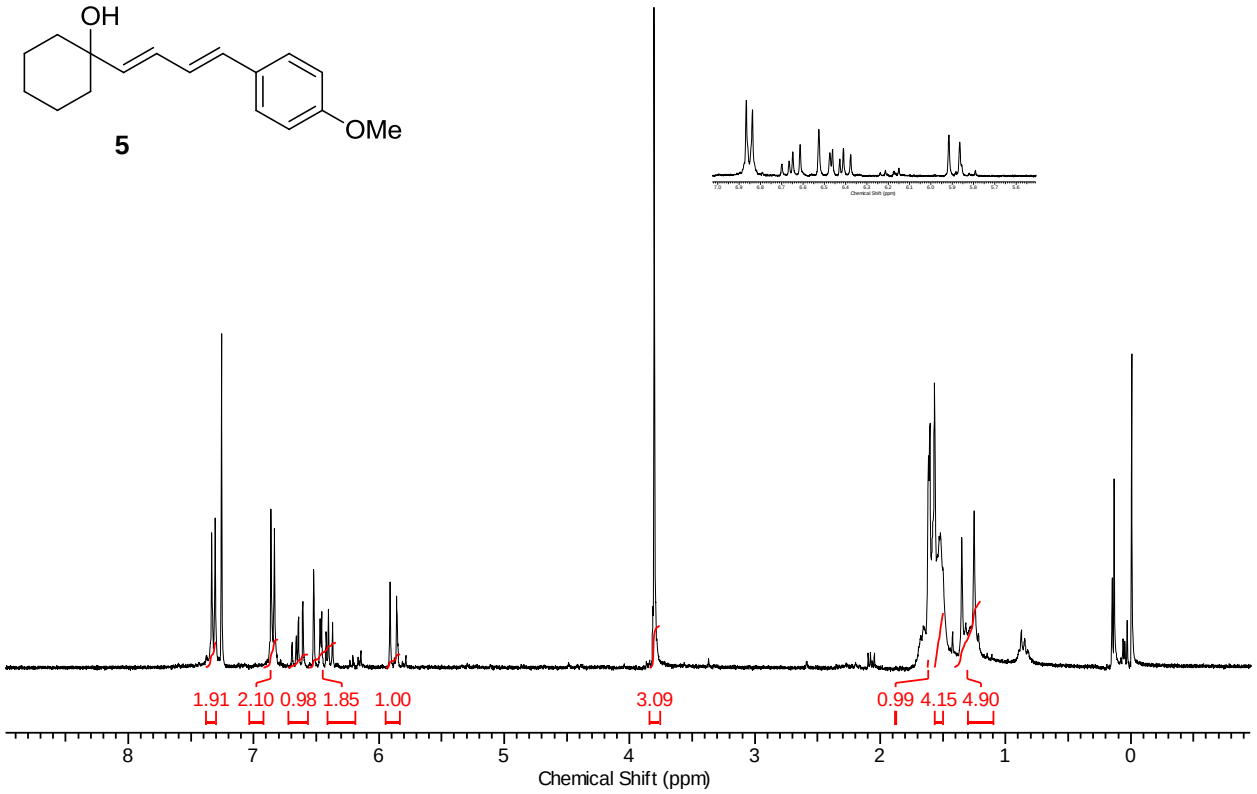
4k

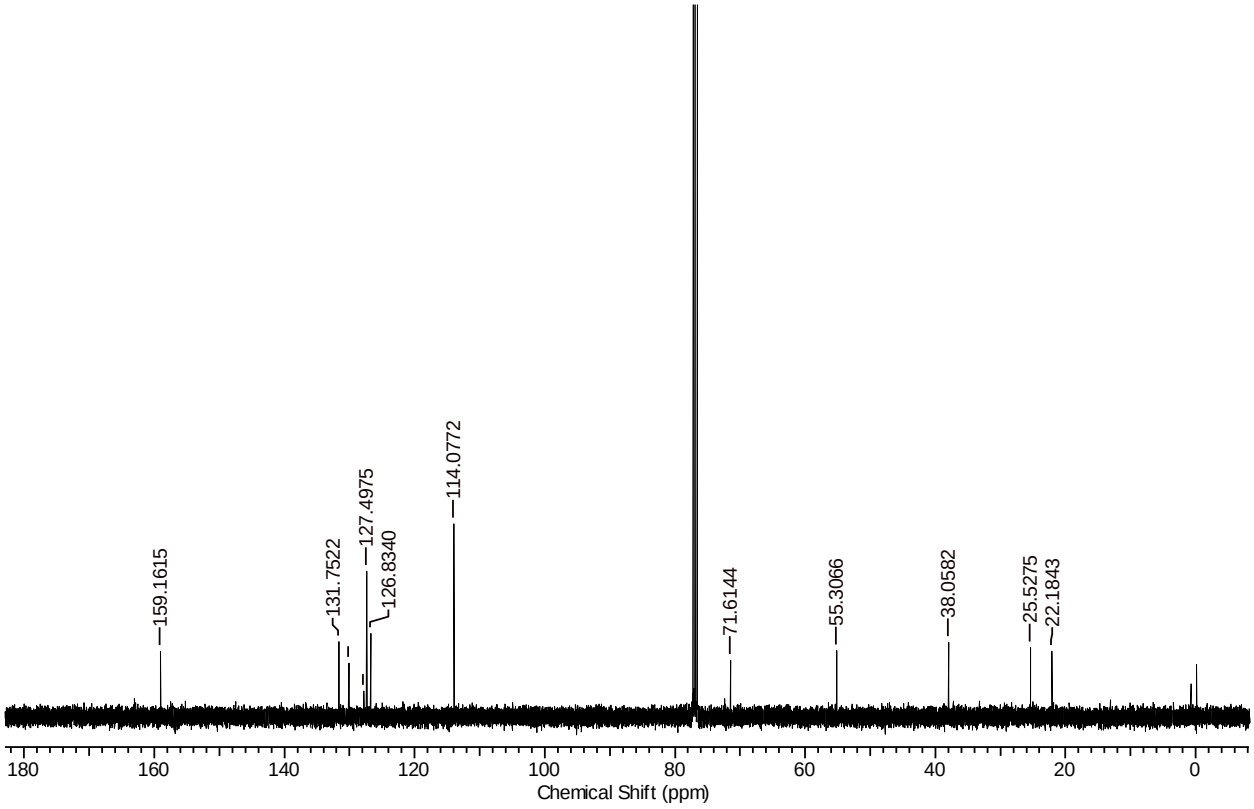
2.15 2.29 0.99 1.03 0.98 1.01 0.99
2.10 2.19
2.06 2.53 0.93
8 7 6 5 4 3 2 1 0 -1
Chemical Shift (ppm)

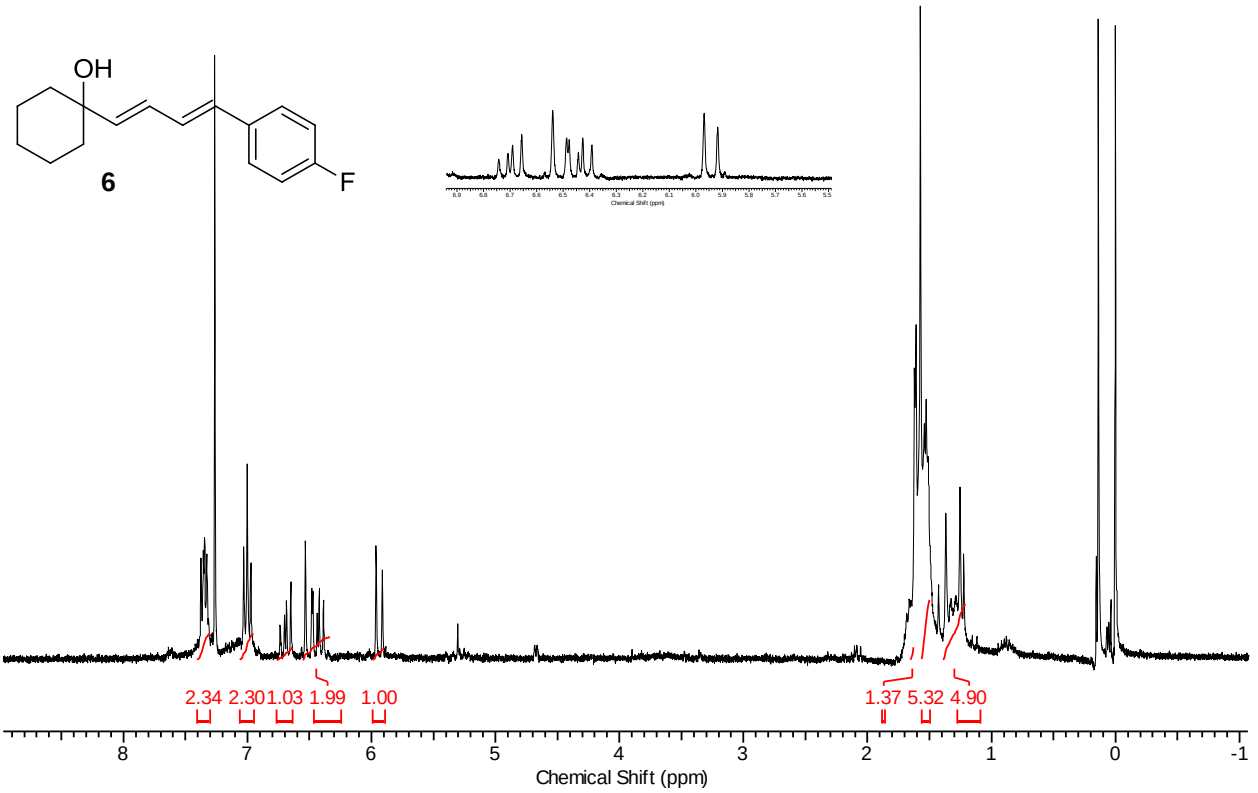

140.3162
137.1079
133.0428
128.6496
126.3782
69.1972
63.7649
37.9488
200 180 160 140 120 100 80 60 40 20 0
Chemical Shift (ppm)

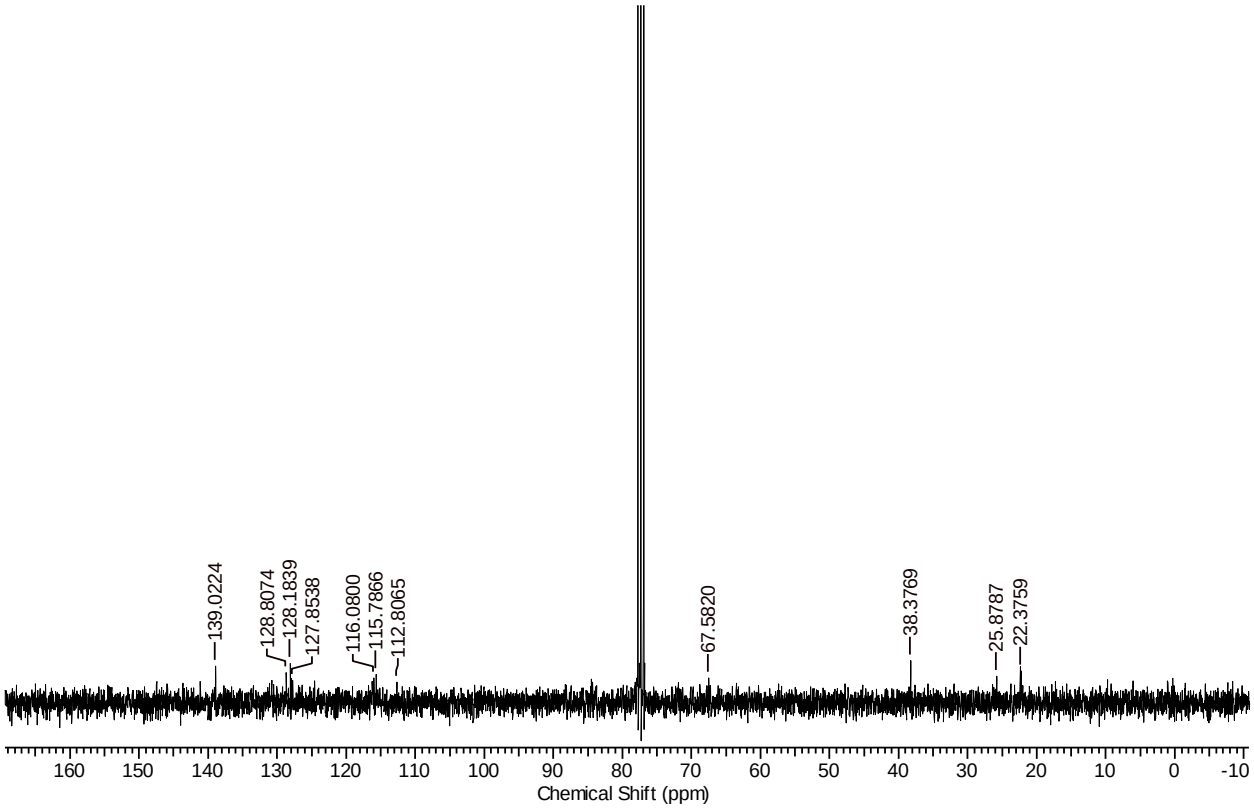

OH
BocN
Ph
4l
2.31 2.84 1.05 0.99 1.10 1.00
1.99 2.27
9.16
Chemical Shift (ppm)

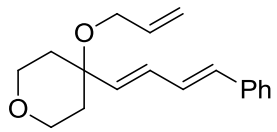

151.0896
139.0132
129.0825
127.3495
126.9093
126.6984
126.4142
107.6990
103.0959
83.5096
71.7634
28.7855
11.0148
Chemical Shift (ppm)


OH
OMe
5
1.91 2.10 0.98 1.85 1.00
3.09
0.99 4.15 4.90
8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)


159.1615
131.7522
127.4975
126.8340
114.0772
71.6144
55.3066
38.0582
25.5275
22.1843
180 160 140 120 100 80 60 40 20 0
Chemical Shift (ppm)

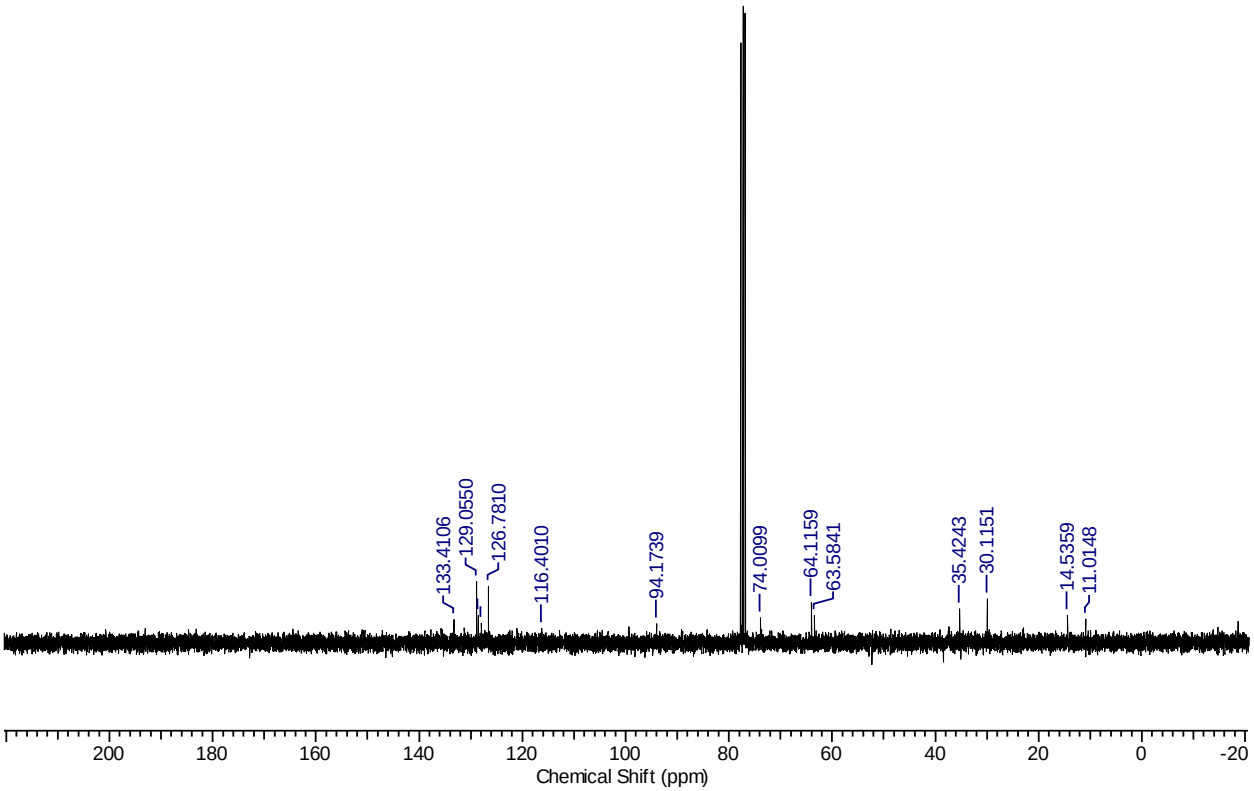

OH
F
6
2.34 2.30 1.03 1.99 1.00
1.37 5.32 4.90
8 7 6 5 4 3 2 1 0 -1
Chemical Shift (ppm)

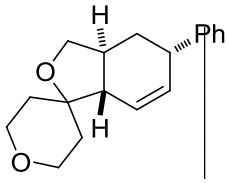

139.0224
128.8074
128.1839
127.8538
116.0800
115.7866
112.8065
67.5820
38.3769
25.8787
22.3759
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
Chemical Shift (ppm)


O
O
Ph

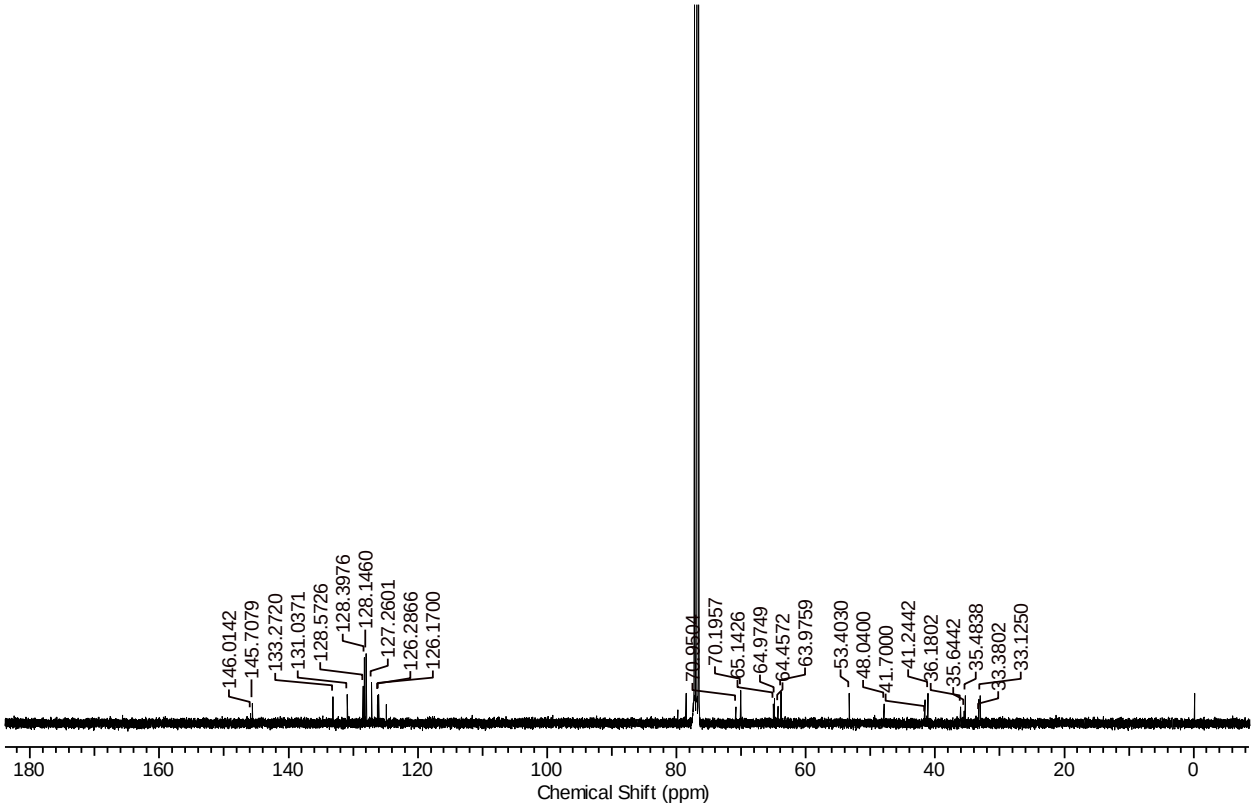
8

7.0
6.5
6.0
5.5
5.0
Chemical Shift (ppm)
2.04 2.53 0.92 1.00 0.98 1.02 1.29 1.32 1.23 1.44
2.48 2.62
4.02
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)


133.4106
129.0550
126.7810
116.4010
94.1739
74.0099
64.1159
63.5841
35.4243
30.1151
14.5359
11.0148
200
180
160
140
120
100
80
60
40
20
0
-20
Chemical Shift (ppm)



9- Mix exo:endo



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