Supporting Information

Diversity-Oriented Synthesis of Disubstituted Alkenes using Masked Silanols

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1. General Procedures

All experiments were carried out under an atmosphere of nitrogen, using anhydrous solvents, unless otherwise stated. All chemicals were purchased from Sigma-Aldrich, Strem or Fluorochem and used without further purification. ^{n}Butyl lithium was titrated against *N*-benzyl benzamide before use.[1] Reaction temperatures of ~0^{o}C were obtained using an ice-water bath, temperatures of -78^{o}C were obtained using an acetone-dry ice bath. Room temperature refers to 20-25^{o}C. Analytical thin layer chromatography was carried out on Merck Kieselgel 60 F_{254} plates with visualisation by ultraviolet light or staining with potassium permanganate or ninhydrin dips made using standard procedures. Retention factors (R_f) are quoted to 0.01. Flash column chromatography was performed using Merck Kieselgel 60 (230-400 mesh) or biotage silica columns under a positive pressure of nitrogen.

Infra-red spectra were recorded on a Perkin Elmer Spectrum One FT-IR spectrometer fitted with an Attenuated Total Reflectance (ATR) sampling accessory as neat films. Maximum absorbance (ν_{max}) are quoted in wavenumbers (cm^{-1}) and the abbreviations used to describe the absorbance intensity are: w, weak; m, medium; s, strong.

Proton nuclear magnetic resonance (^{1}H-NMR), carbon nuclear magnetic resonance (^{13}C-NMR) and fluorine nuclear magnetic resonance (^{19}F-NMR) were recorded using ambient probe temperatures on the following instruments: Bruker DXP 400, Bruker Avance DXP 400, Bruker Avance 500 BB-ATM, Bruker Avance Cyro 500. The following deuterated solvents were used: chloroform ($CDCl_3$), dichloromethane (CD_2Cl_2) and methanol (MeOD). ^{1}H-NMR chemical shifts (δ) are quoted in ppm relative to the residual non-deuterated solvent peak and coupling constants (*J*) are quoted to the nearest 0.1 Hertz (Hz). Spectral data is reported as follows: chemical shift, integration, multiplicity [s, singlet; d, doublet; t, triplet; q, quartet; sept, septuplet; m, multiplet; br, broad; or as a combination of these eg br s, dd etc.], coupling constant(s) and assignment. Proton assignment is supported by COSY (2D ^{1}H-^{1}H) spectra where necessary. Carbon assignment is supported by DEPT editing and HMQC (2D one bond ^{1}H-^{13}C) correlations where necessary.

High resolution mass spectrometry (HRMS) was carried out with a Micromass Q-TOF or a Micromass LCT premier spectrometer using electrospray ionisation (ESI) or electron ionisation (EI) and the calculated mass value relative to found mass value is within the error limits of ±5 ppm mass units.

Low resolution mass spectrometry (LCMS) was carried out using either method LCMS (A) or LCMS (B). LCMS (A): Acquity UPLC BEH C18 column (50mmx2.1mm i.d. 1.7µm packing diameter) at 40°C. Solvent A = 0.1% v/v solution of formic acid in water. Solvent B = 0.1% v/v solution of formic acid in acetonitrile. The gradient employed was over 2 minutes going from 100% A to 100% B at a flow rate of 1 ml/min. LCMS (B): ABZ++ colunm. Solvent A = 1% v/v solution of formic acid and 10 mM ammonium acetate in water. Solvent B = 0.05% v/v solution of formic acid and 5% water in acetonitrile. The gradient was employed over 8 minutes going from 100% A to 100% B at a flow rate of 5 ml/min. The UV detection was an averaged signal from wavelength of 210nm to 350nm and mass spectra were recorded on a mass spectrometer using alternate-scan positive and negative mode electrospray ionisation.

Melting points were carried out using a Buchi melting point B545 apparatus and are uncorrected.

General Method 1.1 – (*E*)-hydrosilylation of terminal alkynes with pentafluorophenyldimethylsilane

A solution of tri-*tert*-butylphosphine (0.1 mol%, 1.0 M in toluene) was added to a solution of platinum(0)-1,3-divinyl-1,1,3,3-tetramethyldisiloxane complex (0.1 mol%, ~2% Pt in xylenes) under nitrogen and stirred for 5 minutes, forming a white paste. On cooling the reaction to 0°C, pentafluorophenyldimethylsilane (1.00 g, 4.40 mmol, 1 equiv.) in anhydrous toluene (0.5 ml) was added followed by terminal alkyne (1.1 equiv.) in anhydrous toluene (0.5 ml). The reaction mixture was stirred at room temperature for 16 hours. The solvent was removed and the crude residue purified by flash silica chromatography to give the desired product.

General Method 1.2 – (*Z*)-hydrosilylation of terminal alkynes with pentafluorophenyldimethylsilane

To an oven dried flask under nitrogen was added the terminal alkyne (1 equiv.) in anhydrous dichloromethane (1 ml) and pentafluorophenyldimethylsilane (200 mg, 0.881 mmol, 1 equiv.) in anhydrous dichloromethane (1 ml). The reaction mixture was degassed by bubbling nitrogen through solution before addition of the ruthenium complex $(PCy_3)_2(CO)(Cl)RuH$ (5 mol%).[2] The reaction was stirred at room temperature for 2 hours, filtered immediately through a plug of silica eluting with petroleum ether 40-60 and the filtrate concentrated under reduced pressure. The crude residue was purified by flash silica chromatography to give the desired product.

General Method 1.3 – α-hydrosilylation of terminal alkynes with pentafluorophenyldimethylsilane

To a solution of the terminal alkyne (1 equiv.) in anhydrous dichloromethane (0.5 ml) was added pentafluorophenyldimethylsilane (446 mg, 1.97 mmol, 1.05 equiv.) in anhydrous dichloromethane (0.5 ml). The reaction was cooled to 0°C before the addition of pentamethylcyclopentadienyltris (acetonitrile)ruthenium(II) hexafluoro-phosphate (1 mol%). The reaction was immediately allowed to warm to room temperature and stirred for 16 hours. The reaction mixture was concentrated under reduced pressure and the residue purified by flash silica chromatography to give the desired product.

General Method 1.4 – Fluoride induced cross coupling of pentafluorophenyldimethylvinylsilanes with aryl iodides

To a solution of pentafluorophenyldimethylvinylsilane (200 mg, 0.650 mmol, 1 equiv.) and aryl iodide (0.75 equiv.) in tetrahydrofuran (2 ml) was added a tetrabutylammonium fluoride solution (2 equiv., 1.0M in tetrahydrofuran) and bis(dibenzylideneacetone)palladium(0) (2.5 mol%). The reaction mixture was stirred at room temperature for 1 hour, then filtered through silica eluting with dichloromethane and the filtrate was concentrated under reduced pressure. The crude residue was purified by flash silica chromatography to give the desired product.

General Method 1.5 – Base induced cross coupling of pentafluorophenyldimethylvinylsilanes with aryl iodides

Pentafluorophenyldimethylvinylsilane (200 mg, 0.610 mmol, 1 equiv.), aryl iodide (0.75 equiv.), potassium hydroxide (3 equiv.) and bis(dibenzylideneacetone)palladium(0) (2.5 mol%) in methanol (1 ml) were stirred at room temperature for 3 hours. The reaction mixture was partitioned between water and dichloromethane, separated and the aqueous layer extracted further with dichloromethane. The organic extracts were combined, dried ($MgSO_4$), concentrated under reduced pressure and the crude residue purified by flash silica chromatography to give the desired product.

General Method 1.6 – Telescoping the synthesis of alkynylsilanes, reduction and cross coupling reactions to produce (*Z*)-disubstituted alkenes

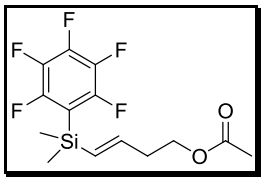
To a stirring solution of the terminal alkyne (0.500 g, 2.21 mmol, 1 equiv.) in anhydrous tetrahydrofuran (2.5 ml) at -78^{o}C under a nitrogen atmosphere, was added ^{n}butyllithium (1.05 equiv., 1.6 m in hexane) dropwise. The reaction mixture was stirred at -78^{o}C for 30 minutes before dropwise addition of chlorodimethyl(pentafluorophenyl)silane (1.05 equiv.). The reaction mixture was stirred for an additional 30 minutes at -78^{o}C before warming to room temperature for a further 3 hours. The reaction was poured into iced-water and extracted with diethyl ether. The organic extracts were combined, dried ($MgSO_4$) and concentrated under reduced pressure. The crude residue was purified by flash silica chromatography to give the pentafluorophenyldimethylalkynylsilane.

A solution of pentafluorophenyldimethylalkynylsilane (100 mg, 0.310 mmol, 1 equiv.) in anhydrous diethyl ether (3 ml) under nitrogen, was cooled to 0^{o}C, diisobutylaluminum hydride solution (5 equiv., 1M in toluene) was added dropwise. The reaction mixture was allowed to warm to room temperature and stirred for 16 hours. The solution was carefully poured into 1N aqueous hydrochloric acid and extracted with dichloromethane. The organic extracts were combined, dried ($MgSO_4$) and concentrated to yield crude (*Z*)-pentafluorophenyldimethylvinylsilane.

The crude (*Z*)-pentafluorophenyldimethylvinylsilane (0.310 mmol, 1 equiv.) was dissolved in tetrahydrofuran (1 ml), aryl iodide (0.75 equiv) was added, followed by tetrabutylammonium fluoride solution (2 equiv., 1.0M in tetrahydrofuran) and bis(dibenzylideneacetone)palladium(0) (5 mol%). The reaction mixture was stirred at room temperature for 1 hour, then filtered through silica eluting with dichloromethane and the filtrate was concentrated under reduced pressure. The crude residue was purified by flash silica chromatography to give the desired product.

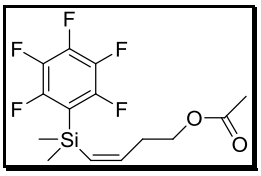
2. Characterisation of compounds synthesised

(*E*)-4-(Dimethyl(perfluorophenyl)silyl)but-3-enyl acetate (Table 1, Entry 1)



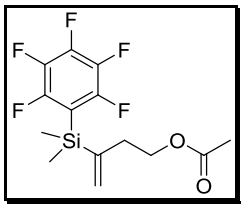
General method 1.1 followed. Purification by flash silica chromatography eluting with a stepped gradient of diethyl ether: petroleum ether 40-60 (0:100 → 1:99 → 3:97) to yield the product as a colourless oil (1.97 g, 66%). $\boldsymbol{R_f}$ 0.21 [diethyl ether: petroleum ether 40-60 (1:99)]. $\boldsymbol{\nu_{max}}$ (neat)/ cm^{-1} 2965 (m, C-H), 1742 (s, C=O ester), 1642 (w, C=C), 1620 (w, C=C), 1456 (s). δ_H (400 MHz, $CDCl_3$) 6.12 (1H, dt, *J*=19.0, 6.2 Hz, SiCH=C<u>H</u>CH_2), 5.91 (1H, dt, *J*=19.0, 1.0 Hz, SiC<u>H</u>=CH), 4.13 (2H, t, *J*=6.2 Hz, $CH_2C\underline{H_2}O$), 2.47 (2H, qd, *J*=6.2, 1.0 Hz, SiCH=CHC<u>H_2</u>), 2.02 (3H, s, C<u>H_3</u>CO_2), 0.45 (6H, t, *J*=1.2 Hz, Si(C<u>H_3</u>)$_2$). δ_F (376 MHz, $CDCl_3$) -127.13 (2F, m), -152.29 (1F, m), -161.93 (2F, m). δ_C (125 MHz, $CDCl_3$) 171.0 (<u>C</u>=O), 148.9 (dm, *J*=240.5 Hz, Ar <u>C</u>F), 144.7 (SiCH=<u>C</u>H), 141.9 (d sept, *J*=252.9, 8.0 Hz, Ar <u>C</u>F), 137.1 (dm, *J*=250.0 Hz, Ar <u>C</u>F), 129.0 (Si<u>C</u>H=CH), 109.9 (t, *J*=32.0 Hz, Ar <u>C</u>), 62.9 (<u>C</u>H_2O), 35.7 (SiCH=CH<u>C</u>H_2), 20.8 (<u>C</u>H_3CO_2), -1.6 (t, *J*=3.0 Hz, Si(<u>C</u>H_3)$_2$). **HRMS** (ESI+) *m/z* found 339.0851, $C_{14}H_{16}F_5O_2Si$ (MH^+) requires 339.0840 (Δ = 3.2 ppm).

(*Z*)-4-(Dimethyl(perfluorophenyl)silyl)but-3-enyl acetate (Table 1, Entry 2)



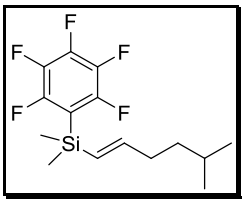
General method 1.2 followed. Purification by flash silica chromatography eluting with a stepped gradient of dichloromethane: iso-hexane (0:100 → 10:90→20:80 → 30:70 → 40:60) to yield the product as a colourless oil (0.142 g, 48%, (*Z*):(*E*):α, 68:19:13). $\boldsymbol{R_f}$ 0.14 [iso-hexane: dichloromethane (80:20)]. $\boldsymbol{\nu_{max}}$ (neat)/ cm^{-1} 2963 (w, C-H), 1742 (s, C=O ester), 1642 (w, C=C), 1615 (w, C=C), 1516 (s, C=C). δ_H (400 MHz, $CDCl_3$) 6.36 (1H, dt, *J*=14.1, 7.4 Hz, SiCH=C<u>H</u>CH_2), 5.77 (1H, d, *J*=14.1 Hz, SiC<u>H</u>=CH), 4.06 (2H, t, *J*=6.6 Hz, $CH_2C\underline{H_2}O$), 2.41 (2H, qd, *J*=7.3, 1.1 Hz, SiCH=CHC<u>H_2</u>), 2.01 (3H, s, C<u>H_3</u>CO_2), 0.52 (6H, t, *J*=1.7 Hz, Si(C<u>H_3</u>)$_2$). δ_F (376 MHz, $CDCl_3$) -127.11 (2F, m), -152.05 (1F, m), -161.70 (2F, m). δ_C (125 MHz, $CDCl_3$) 170.9 (<u>C</u>=O), 148.9 (dm, *J*=240.5 Hz, Ar <u>C</u>F), 145.9 (SiCH=<u>C</u>H), 141.9 (d sept, *J*=252.9, 8.0 Hz, Ar <u>C</u>F), 137.1 (dm, *J*=250.0 Hz, Ar <u>C</u>F), 128.2 (Si<u>C</u>H=CH), 110.1 (t, *J*=32.0, Ar <u>C</u>), 63.2 (<u>C</u>H_2O), 32.7 (SiCH=CH<u>C</u>H_2), 20.8 (<u>C</u>H_3CO_2), 0.1 (t, *J*=3.0 Hz, Si(<u>C</u>H_3)$_2$). **LCMS** (A) R_t 1.44 min, $[M+H]^+$ 339.3. **HRMS** (ESI+) *m/z* found 361.0665, $C_{14}H_{15}F_5O_2SiNa$ (MNa^+) requires 361.0654 (Δ = -3.2 ppm).

3-(Dimethyl(perfluorophenyl)silyl)but-3-enyl acetate (Table 1, Entry 3)



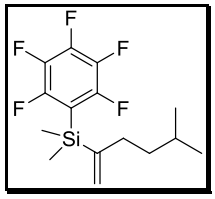
General method 1.3 followed. Purification by flash silica chromatography eluting with a stepped gradient of diethyl ether: petroleum ether 40-60 (0:100 → 0.5:99.5 → 1:99) to yield the product as a colourless oil (539 mg, 89%). R_f 0.61 [diethyl ether: petroleum ether 40-60 (1:99)]. ν_{max} (neat)/ cm^{-1} 2958 (br w, C-H), 1742 (s, C=O ester), 1642 (m, C=C), 1516 (s, C=C). δ_H (400 MHz, $CDCl_3$) 5.78 (1H, d, *J*=2.0 Hz, $\underline{H}_2C$=C), 5.55 (1H, d, *J*=2.0 Hz, $\underline{H}_2C$=C), 4.09 (2H, t, *J*=7.0 Hz, $CH_2C\underline{H}_2O$), 2.45 (2H, t, *J*=7.0 Hz, H_2C=C(Si)$C\underline{H}_2$), 2.00 (3H, s, $C\underline{H}_3CO_2$), 0.50 (6H, t, *J*=1.2 Hz, Si($C\underline{H}_3$)$_2$). δ_F (376 MHz, $CDCl_3$) -126.69 (2F, m), -151.59 (1F, m), -161.54 (2F, m). δ_C (400 MHz, $CDCl_3$) 170.9 ($\underline{C}$=O), 149.0 (dm, *J*=241.0 Hz, Ar $\underline{C}$F), 143.8 (H_2C=$\underline{C}$(Si)CH_2), 142.1 (dm, *J*=253.5 Hz, Ar $\underline{C}$F), 137.1 (dm, *J*=251.9 Hz, Ar $\underline{C}$F), 129.2 ($H_2\underline{C}$=C(Si)CH_2), 109.7 (tq, *J*=32.1, 3.4 Hz, Ar $\underline{C}$), 63.3 ($\underline{C}H_2O$), 33.9 (H_2C=C(Si)$\underline{C}H_2$), 20.8 ($\underline{C}H_3CO_2$), -1.7 (t, *J*=3.5 Hz, Si($\underline{C}H_3$)$_2$). **LCMS** (A) R_t 1.43 min, $[M+H]^+$ 339.0. **HRMS** (ESI+) *m/z* found 361.0644, $C_{14}H_{15}F_5O_2SiNa$ (MNa^+) requires 361.0654 (Δ = 2.5 ppm).

(*E*)-Dimethyl(5-methylhex-1-enyl)(perfluorophenyl)silane (Table 1, Entry 4)



General method 1.1 followed. Purificiation by flash silica chromatography eluting with of cyclohexane (100%) to yield the product as a colourless oil (3.12 g, 97%). R_f 0.53 [cyclohexane]. ν_{max} (neat)/ cm^{-1} 2962 (m, C-H), 2902 (m, C-H), 1641 (w, C=C), 1617 (w, C=C), 1516 (m, C=C). δ_H (400 MHz, $CDCl_3$) 6.19 (1H, dt, *J*=18.6, 6.3 Hz, SiCH=C$\underline{H}CH_2$), 5.80 (1H, dt, *J*=18.6, 1.5 Hz, SiC$\underline{H}$=CH), 2.12-2.19 (2H, m, SiCH=CHC$\underline{H}_2$), 1.49-1.60 (1H, m, C$\underline{H}$(CH_3)$_2$), 1.26-1.33 (2H, m, C$\underline{H}_2$CH(CH_3)$_2$), 0.89 (6H, d, *J*=6.5 Hz, CH($C\underline{H}_3$)$_2$), 0.46 (6H, t, *J*=1.5 Hz, Si($C\underline{H}_3$)$_2$). δ_F (376 MHz, $CDCl_3$) -127.20 (2F, m), -152.73 (1F, m), -162.11 (2F, m). δ_C (101 MHz, $CDCl_3$) 150.3 (SiCH=$\underline{C}$H), 124.7 (Si$\underline{C}$H=CH), 37.2 ($\underline{C}H_2$CH(CH_3)$_2$), 34.2 (SiCH=CH$\underline{C}H_2$), 27.3 ($\underline{C}$H(CH_3)$_2$), 22.2 (CH($\underline{C}H_3$)$_2$), -1.8 (Si($\underline{C}H_3$)$_2$). **Elemental analysis** (CHN) calculated for $C_{15}H_{19}F_5Si$ (%) C 55.88, H 5.94, N 0.00; found C 55.97, H 5.93, N 0.00.

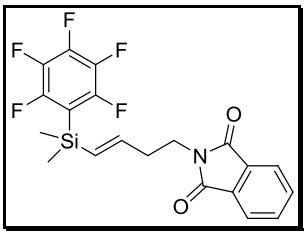
Dimethyl(5-methylhex-1-en-2-yl)(perfluorophenyl)silane (Table 1, Entry 5)



General method 1.3 followed. Purification by flash silica chromatography eluting with cyclohexane (100%) to yield the product as a colourless oil (3.16 g, 98%). R_f 0.50 [cyclohexane]. ν_{max} (neat)/ cm^{-1} 2958 (w, C-H), 2927 (w, C-H), 1641 (w, C=C), 1516 (m, C=C). δ_H (400 MHz, $CDCl_3$) 5.71 (1H, d, *J*=2.3 Hz, $\underline{H}_2$C=C), 5.43-5.45 (1H, m, $\underline{H}_2$C=C), 2.10-2.18 (2H, m, SiC(=CH_2)C$\underline{H}_2$),

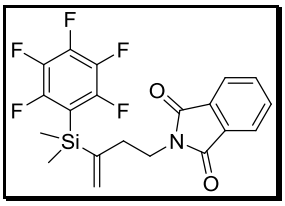
1.50 (1H, sept, *J*=6.5 Hz, C<u>H</u>(CH$_3$)$_2$), 1.21-1.30 (2H, m, C<u>H</u>$_2$CH(CH$_3$)$_2$), 0.85 (6H, d, *J*=6.5 Hz, CH(C<u>H</u>$_3$)$_2$), 0.50 (6H, t, *J*=1.8 Hz, Si(C<u>H</u>$_3$)$_2$). δ_F (376 MHz, $CDCl_3$) -126.73 (2F, m), -152.16 (1F, m), -161.89 (2F, m). δ_C (101 MHz, $CDCl_3$) 148.1 (Si<u>C</u>(=CH$_2$)CH$_2$), 126.2 (SiC(=<u>C</u>H$_2$)CH$_2$), 37.8 (<u>C</u>H$_2$CH(CH$_3$)$_2$), 33.0 (SiC(=CH$_2$)<u>C</u>H$_2$), 27.4 (<u>C</u>H(CH$_3$)$_2$), 22.1 (CH(<u>C</u>H$_3$)$_2$), -1.7 (Si(<u>C</u>H$_3$)$_2$). **Elemental analysis** (CHN) calculated for $C_{15}H_{19}F_5Si$ (%) C 55.88, H 5.94, N 0.00; found C 56.20, H 6.06, N 0.00.

(*E*)-2-(4-(Dimethyl(perfluorophenyl)silyl)but-3-enyl)isoindoline-1,3-dione (Table 1, Entry 6)



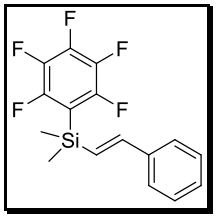
General method 1.1 followed. Purification by flash silica chromatography eluting with a stepped gradient of ethyl acetate: petroleum ether 40-60 (0:100 → 4:96) to yield the product as a colourless oil (0.920 g, 86%). R_f 0.16 [ethyl acetate: petroleum ether 40-60 (4:96)]. ν_{max} (neat)/ cm^{-1} 2948 (w, C-H), 1774 (m, C=O), 1710 (s, C=O), 1642 (m, C=C), 1617 (m, C=C), 1515 (m, C=C). δ_H (500 MHz, $CDCl_3$) 7.78 (2H, m Ar C<u>H</u>), 7.68 (2H, m Ar C<u>H</u>), 6.12 (1H, dt, *J*=18.6, 6.7 Hz, SiCH=C<u>H</u>CH$_2$), 5.80 (1H, dt, *J*=18.6, 1.4 Hz, SiC<u>H</u>=CH), 3.78 (2H, t, *J*=6.8 Hz, CH$_2$C<u>H</u>$_2$N), 2.47 (2H, qd, *J*=6.8, 1.3 Hz, SiCH=CHC<u>H</u>$_2$), 0.36 (6H, t, *J*=1.6 Hz, Si(C<u>H</u>$_3$)$_2$). δ_F (376 MHz, $CDCl_3$) -127.10 (2F, m), -152.41 (1F, m), -161.83 (2F, m). δ_C (125 MHz, $CDCl_3$) 168.2 (<u>C</u>=O), 148.7 (dm, *J*=241.1 Hz, Ar <u>C</u>F), 145.4 (SiCH=<u>C</u>H), 141.8 (dm, *J*=250.0 Hz, Ar <u>C</u>F), 137.0 (dm, *J*=251.8 Hz, Ar <u>C</u>F), 133.8 (Ar <u>C</u>H), 131.9 (Ar <u>C</u>), 129.5 (Si<u>C</u>H=CH), 123.0 (Ar <u>C</u>H), 109.7 (t, *J*=32.1 Hz, Ar <u>C</u>), 36.7 (<u>C</u>H$_2$N), 35.6 (SiCH=CH<u>C</u>H$_2$), -1.8 (t, *J*=3.0 Hz, Si(<u>C</u>H$_3$)$_2$). **LCMS** (A) R$_t$ 1.49min, [M+H]$^+$ 425.9. **HRMS** (ESI+) *m/z* found 426.0967, $C_{20}H_{17}F_5NO_2Si$ (MH$^+$) requires 426.0949 (Δ = 4.2 ppm).

2-(3-(Dimethyl(perfluorophenyl)silyl)but-3-enyl)isoindoline-1,3-dione (Table 1, Entry 7)



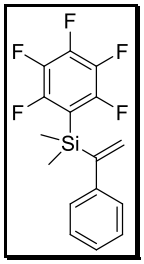
General method 1.3 followed. Purification by flash silica chromatography eluting with dichloromethane to yield the product as a white crystalline solid (2.12 g, 99%). R_f 0.58 [dichloromethane]. ν_{max} (neat)/ cm^{-1} 2948 (w, C-H), 1767 (m, C=O), 1703 (s, C=O), 1642 (w, C=C), 1611 (w, C=C), 1518 (m, C=C). δ_H (400 MHz, $CDCl_3$) 7.81 (2H, m Ar C<u>H</u>), 7.69 (2H, m Ar C<u>H</u>), 5.81 (1H, d, *J*=1.6 Hz, <u>H</u>$_2$C=C), 5.56 (1H, d, *J*=1.7 Hz, <u>H</u>$_2$C=C), 3.71 (2H, t, *J*=7.6 Hz, CH$_2$C<u>H</u>$_2$N), 2.51 (2H, t, *J*=7.7 Hz, C<u>H</u>$_2$CH$_2$N), 0.54 (6H, t, *J*=1.8 Hz, Si(C<u>H</u>$_3$)$_2$). δ_F (376 MHz, $CDCl_3$) -126.53 (2F, m), -151.65 (1F, m), -161.51 (2F, m). δ_C (125 MHz, $CDCl_3$) 168.1 (<u>C</u>=O), 148.7 (dm, *J*=241.1 Hz, Ar <u>C</u>F), 144.3 (H$_2$C=<u>C</u>), 141.8 (dm, *J*=250.0 Hz, Ar <u>C</u>F), 137.0 (dm, *J*=251.8 Hz, Ar <u>C</u>F), 133.9 (Ar <u>C</u>H), 132.0 (Ar <u>C</u>), 129.4 (H$_2$<u>C</u>=C), 123.2 (Ar <u>C</u>H), 109.7 (t, *J*=32.1 Hz, Ar <u>C</u>), 37.4 (<u>C</u>H$_2$N), 33.4 (<u>C</u>H$_2$CH$_2$N), -1.7 (t, *J*=3.0 Hz, Si(<u>C</u>H$_3$)$_2$). **Mpt.** 91.1-93.3°C. **LCMS** (A) R$_t$ 1.50 min, [M+H]$^+$ 425.8. **HRMS** (ESI+) *m/z* found 448.0753, $C_{20}H_{16}F_5NO_2SiNa$ (MNa$^+$) requires 448.0763 (Δ = 2.2 ppm).

(*E*)-Dimethyl(perfluorophenyl)(styryl)silane (Table 1, Entry 8)



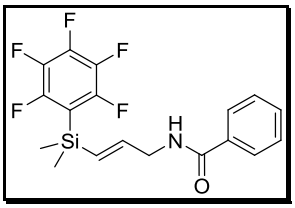
General method 1.1 followed. Purification by flash silica chromatography eluting with petroleum ether 40-60 (100 %) to yield the product as a white solid (2.58 g, 89%). R_f 0.44 [petroleum ether 40-60]. ν_{max} (neat)/ cm^{-1} 2967 (w, C-H), 1642 (m, C=C), 1606 (w, C=C), 1575 (w, C=C), 1515 (m, C=C). δ_H (400 MHz, $CDCl_3$) 7.45 (2H, d, *J*=8.0 Hz, o-Ar C<u>H</u>), 7.36-7.24 (3H, m, m,p-Ar C<u>H</u>), 6.99 (1H, d, *J*=19.0 Hz, SiCH=C<u>H</u>Ar), 6.58 (1H, dt, *J*=19.0, 1.2 Hz, SiC<u>H</u>=CH), 0.57 (6H, t, *J*=1.2 Hz, Si(C<u>H</u>$_3$)$_2$). δ_F (376 MHz, $CDCl_3$) -127.04 (2F, m), -152.11 (1F, m), -161.75 (2F, m). δ_C (125 MHz, $CDCl_3$) 148.9 (dm, *J*=240.5 Hz, Ar <u>C</u>F), 146.0 (SiCH=<u>C</u>H), 142.0 (d sept, *J*=253.0, 6.0 Hz, Ar <u>C</u>F), 137.6 (Ar <u>C</u>), 137.1 (dm, *J*=251.5 Hz, Ar <u>C</u>F), 128.7 (Ar <u>C</u>H), 128.6 (Ar <u>C</u>H), 126.8 (Ar <u>C</u>H), 124.5 (Si<u>C</u>H=CH), 109.8 (tq, *J*=32.4, 3.5, Ar <u>C</u>), -1.5 (t, *J*=3.0 Hz, Si(<u>C</u>H$_3$)$_2$). **Mpt.** 45.2-45.7°C (no lit. value reported). **Elemental analysis** (CHN) calculated for $C_{16}H_{13}F_5Si$ (%) C 58.53, H 3.99, N 0.00; found C 58.50, H 3.94, N 0.00. Data consistent with that reported in the literature.[3]

Dimethyl(perfluorophenyl)(1-phenylvinyl)silane (Table 1, Entry 9)



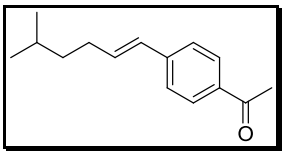
General method 1.3 followed, except 10 mol% Ru catalyst used. Purification by flash silica chromatography eluting with petroleum ether 40-60 (100%) to yield the product as a colourless oil (1.24 g, 86%). R_f 0.48 [petroleum ether 40-60]. ν_{max} (neat)/ cm^{-1} 2962 (w, C-H), 1641 (m, C=C), 1515 (m, C=C). δ_H (400 MHz, $CDCl_3$) 7.29-7.19 (3H, m, Ar C<u>H</u>), 7.13-7.09 (2H, m, Ar C<u>H</u>), 5.99 (1H, d, *J*=2.4 Hz, <u>H</u>$_2$C=C), 5.71 (1H, d, *J*=2.4 Hz, <u>H</u>$_2$C=C), 0.56 (6H, t, *J*=1.2 Hz, Si(C<u>H</u>$_3$)$_2$). δ_F (376 MHz, $CDCl_3$) -126.57 (2F, m), -151.55 (1F, m), -161.60 (2F, m). δ_C (125 MHz, $CDCl_3$) 150.0 (Ar <u>C</u>), 149.8 (dm, *J*=241.9 Hz, Ar <u>C</u>F), 143.8 (H$_2$C=<u>C</u>(Si)CH$_2$), 142.9 (dm, *J*=250.0 Hz, Ar <u>C</u>F), 138.0 (dm, *J*=250.0 Hz, Ar <u>C</u>F), 130.4 (H$_2$<u>C</u>=C(Si)CH$_2$), 129.3 (Ar <u>C</u>H), 127.6 (Ar <u>C</u>H), 127.5 (Ar <u>C</u>H), 110.3 (tq, *J*=31.6, 3.5, Ar <u>C</u>), 0.0 (t, *J*=3.4 Hz, Si(<u>C</u>H$_3$)$_2$). **HRMS** (EI) *m/z* found 328.0693, $C_{16}H_{13}F_5Si$ (M^+) requires 328.0701 (Δ = 2.5 ppm). No analytical data found, compound referenced.[3]

(*E*)-*N*-(3-(Dimethyl(perfluorophenyl)silyl)allyl)benzamide (Table 1, Entry 10)



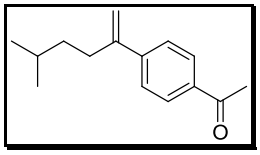
General method 1.1 followed, pentafluorophenyldimethylvinylsilane was subjected to further modifications. After the synthesis of the pentafluorophenyldimethylvinylsilane the crude reaction mixture was cooled to 0°C, anhydrous tetrahydrofuran (5 ml), triethylamine (1.2 equiv.), then benzoyl chloride (1.2 equiv.) were added dropwise. The reaction was stirred at room temperature for 3 hours, before partitioning between water and ethyl acetate. The aqueous layer was extracted further with ethyl acetate; all organic extracts were combined, dried ($MgSO_4$) and concentrated under reduced pressure. The crude residue was purified by flash silica chromatography eluting with a stepped gradient of dichloromethane: petroleum ether 40-60 (50:50 → 75:25) to yield the product as a colourless oil (475 mg, 68%). $\boldsymbol{R_f}$ 0.14 [dichloromethane]. $\boldsymbol{\nu_{max}}$ (neat)/ cm^{-1} 3321 (m, N-H amide), 2990 (w, C-H), 2919 (w, C-H), 1643 (s, C=O amide), 1540 (s, C=C), 1517 (s, C=C). δ_H (400 MHz, $CDCl_3$) 7.79 (2H, m, Ar C<u>H</u>), 7.51 (1H, m, Ar C<u>H</u>), 7.43 (2H, m, Ar C<u>H</u>), 6.37 (1H, br s, NH), 6.25 (1H, dt, *J*=18.7, 4.8 Hz, SiCH=C<u>H</u>CH_2), 6.03 (1H, dt, *J*=18.7, 1.5 Hz, SiC<u>H</u>=CH), 4.17 (2H, m, SiCH=CHC<u>H</u>$_2$), 0.48 (6H, t, *J*=1.6 Hz, Si(C<u>H</u>$_3$)$_2$). δ_F (376 MHz, $CDCl_3$) -127.03 (2F, m), -151.98 (1F, m), -161.68 (2F, m). δ_C (101 MHz, $CDCl_3$) 167.3 (<u>C</u>=O), 148.8 (dm, *J*=242.0 Hz, Ar <u>C</u>F), 144.5 (SiCH=<u>C</u>H), 142.0 (dm, *J*=254.4 Hz, Ar <u>C</u>F), 137.1 (dm, *J*=251.3 Hz, Ar <u>C</u>F), 134.6 (Ar <u>C</u>), 131.6 (Ar C<u>H</u>), 128.6 (Ar C<u>H</u>), 126.9 (Si<u>C</u>H=CH and Ar C<u>H</u>), 109.5 (t, *J*=32.7 Hz, Ar <u>C</u>), 44.1 (<u>C</u>H_2N), -1.6 (Si(<u>C</u>H$_3$)$_2$). **LCMS** (B) R_t 4.91 min, $[M+H]^+$ 386.10. **HRMS** (ESI+) *m/z* found 408.0836, $C_{18}H_{16}F_5NOSiNa$ (MNa^+) requires 408.0819 (Δ = 4.2 ppm).

(*E*)-1-(4-(5-Methylhex-1-enyl)phenyl)ethanone (Table 2, Entry 1)



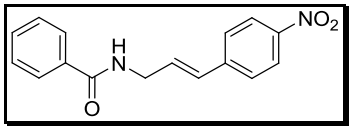
General method 1.4 followed. Purification by flash silica chromatography eluting with a gradient of dichloromethane: cyclohexane (0:100 → 100:0) to yield the product as a colourless oil (96.0 mg, 91%). $\boldsymbol{R_f}$ 0.55 [dichloromethane]. $\boldsymbol{\nu_{max}}$ (neat)/ cm^{-1} 2955 (w, C-H), 2927 (w, C-H), 2869 (w, C-H), 2849 (w, C-H), 1679 (s, C=O ketone), 1648 (w, C=C), 1601 (s, C=C), 1563 (w, C=C). δ_H (400 MHz, $CDCl_3$) 7.89 (2H, d, *J*=8.3 Hz, Ar C<u>H</u>), 7.41 (2H, d, *J*=8.6 Hz, Ar C<u>H</u>), 6.33-6.47 (2H, m, ArC<u>H</u>=C<u>H</u>CH_2), 2.59 (3H, s, COC<u>H</u>$_3$), 2.25 (2H, td, *J*=7.7, 5.8 Hz, ArCH=CHC<u>H</u>$_2$), 1.62 (1H, sept, *J*=6.5 Hz, C<u>H</u>(CH$_3$)$_2$), 1.34-1.42 (2H, m, C<u>H</u>$_2$CH(CH$_3$)$_2$), 0.93 (6H, d, *J*=6.8 Hz, CH(C<u>H</u>$_3$)$_2$). δ_C (101 MHz, $CDCl_3$) 197.6 (<u>C</u>=O), 142.7 (Ar <u>C</u>COCH$_3$), 135.3 (Ar <u>C</u>), 134.7 (<u>C</u>H=CH), 128.7 (<u>C</u>H=CH and Ar <u>C</u>H), 125.9 (Ar <u>C</u>H), 38.2 (<u>C</u>H_2CH(CH$_3$)$_2$), 31.0 (ArCH=CH<u>C</u>H_2), 27.6 (<u>C</u>H(CH$_3$)$_2$), 26.5 (CO<u>C</u>H$_3$), 22.5 (CH(<u>C</u>H$_3$)$_2$). **LCMS** (B) R_t 5.20 min, $[M+H]^+$ 217.28. Data consistent with that reported in the literature.[4]

1-(4-(5-Methylhex-1-en-2-yl)phenyl)ethanone (Table 2, Entry 2)



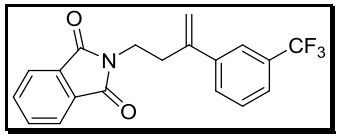
General method 1.4 followed. Purification by flash silica chromatography eluting with a gradient of dichloromethane: cyclohexane (0:100 → 100:0) to yield the product as a colourless oil (91.0 mg, 87%). R_f 0.55 [dichloromethane]. ν_{max} (neat)/ cm^{-1} 2955 (w, C-H), 2928 (w, C-H), 2870 (w, C-H), 1682 (s, C=O ketone), 1625 (w, C=C), 1604 (s, C=C), 1559 (w, C=C). δ_H (400 MHz, $CDCl_3$) 7.93 (2H, d, *J*=8.8 Hz, Ar C<u>H</u>), 7.49 (2H, d, *J*=8.5 Hz, Ar C<u>H</u>), 5.36 (1H, s, <u>H</u>$_2$C=C), 5.18 (1H, d, *J*=1.3 Hz, <u>H</u>$_2$C=C), 2.61 (3H, s, COC<u>H</u>$_3$), 2.49-2.56 (2H, m, C<u>H</u>$_2$CH$_2$CH(CH$_3$)$_2$), 1.54-1.63 (1H, m, C<u>H</u>(CH$_3$)$_2$), 1.29-1.37 (2H, m, C<u>H</u>$_2$CH(CH$_3$)$_2$), 0.91 (6H, d, *J*=6.8 Hz, CH(C<u>H</u>$_3$)$_2$). δ_C (101 MHz, $CDCl_3$) 197.7 (<u>C</u>=O), 148.1 (H$_2$C=<u>C</u>), 146.2 (Ar <u>C</u>), 135.9 (Ar <u>C</u>), 128.4 (Ar <u>C</u>H), 126.2 (Ar <u>C</u>H), 114.0 (H$_2$<u>C</u>=C), 37.4 (<u>C</u>H$_2$CH(CH$_3$)$_2$), 33.0 (<u>C</u>H$_2$CH$_2$CH(CH$_3$)$_2$), 27.8 (<u>C</u>H(CH$_3$)$_2$), 26.6 (CO<u>C</u>H$_3$), 22.5 (CH(<u>C</u>H$_3$)$_2$). **LCMS** (B) R_t 5.09 min, $[M+H]^+$ 217.28. **HRMS** (ESI+) *m/z* found 217.1602, $C_{15}H_{21}O$ (MH^+) requires 217.1592 (Δ = 4.6 ppm). Data consistent with that reported in the literature, only proton NMR data described.[4]

(*E*)-*N*-(3-(4-Nitrophenyl)allyl)benzamide (Table 2, Entry 3)



General method 1.4 followed. Purification by flash silica chromatography eluting with dichloromethane (100%) to yield the product as an amorphous brown solid (47.0 mg, 85%). R_f 0.48 [dichloromethane: methanol (95:5)]. ν_{max} (neat)/ cm^{-1} 3312 (m, N-H), 2919 (m, C-H), 1637 (s, C=O amide), 1595 (m, C=C), 1580 (m, C=C), 1542 (s, C=C), 1507 (s, C=C). δ_H (400 MHz, $CDCl_3$) 8.18 (2H, d, *J*=8.9 Hz, Ar C<u>H</u>), 7.81 (2H, d, *J*=8.5 Hz, Ar C<u>H</u>), 7.55-7.44 (5H, m, Ar C<u>H</u>), 6.66 (1H, d, *J*=15.9 Hz, ArC<u>H</u>=CH), 6.49 (1H, dt, *J*=15.9, 5.9 Hz, ArCH=C<u>H</u>), 6.33 (1H, br s, N<u>H</u>), 4.31 (2H, td, *J*=5.9, 1.4 Hz, ArCH=CHC<u>H</u>$_2$). δ_C (101 MHz, $CDCl_3$) 167.4 (<u>C</u>=O), 147.0 (Ar <u>C</u>NO$_2$), 142.9 (Ar <u>C</u>), 134.1 (Ar <u>C</u>), 131.8 (Ar <u>C</u>H), 130.7 (ArCH=<u>C</u>H), 129.9 (Ar<u>C</u>H=CH), 128.7 (Ar <u>C</u>H), 126.9 (Ar <u>C</u>H), 126.9 (Ar <u>C</u>H), 124.0 (Ar <u>C</u>H), 41.8 (ArCH=CH<u>C</u>H$_2$). **LCMS** (A) R_t 4.37 min, $[M+H]^+$ 283.14. **HRMS** (ESI+) *m/z* found 283.1088, $C_{16}H_{15}N_2O_3$ (MH^+) requires 283.1083 (Δ = 1.8 ppm).

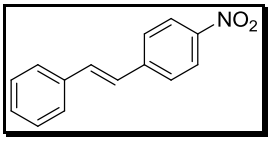
2-(3-(3-(Trifluoromethyl)phenyl)but-3-enyl)isoindoline-1,3-dione (Table 2, Entry 4)



General method 1.4 followed. Purification by flash silica chromatography eluting with a stepped gradient of dichloromethane: petroleum ether 40-60 (0:100 → 20:80 → 40:60) to yield the product as a colourless oil (121 mg, 99%). R_f 0.39 [dichloromethane]. ν_{max} (neat)/ cm^{-1} 2921 (w, C-H), 1773 (m, C=O), 1708 (s, C=O), 1615 (m, C=C). δ_H (400 MHz, $CDCl_3$) 7.80-7.78 (2H, m, Ar Phtalamide C<u>H</u>), 7.70-7.67 (2H, m, Ar Phtalamide C<u>H</u>), 7.79 (1H, br s, Ar C<u>H</u>), 7.61-7.60 (1H, m, Ar C<u>H</u>), 7.45-7.38 (2H, m, Ar C<u>H</u>), 5.39 (1H, br s, <u>H</u>$_2$C=C), 5.23 (1H, br s, <u>H</u>$_2$C=C), 3.83 (2H, t, *J*=7.4 Hz, CH$_2$C<u>H</u>$_2$N), 2.93 (2H, t, *J*=7.5 Hz, C<u>H</u>$_2$CH$_2$N). δ_C (101 MHz, $CDCl_3$) 168.1 (<u>C</u>=O), 143.9 (H$_2$C=<u>C</u>), 141.0 (Ar <u>C</u>), 133.9 (Ar Phtalamide <u>C</u>H), 131.9 (Ar Phtalamide <u>C</u>),

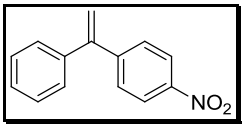
130.7 (d, *J*=31.9 Hz, Ar $\underline{C}CF_3$), 129.4 (Ar $\underline{C}H$), 128.3 (Ar $\underline{C}H$), 124.2 (d, *J*=3.7 Hz, Ar $\underline{C}H$), 124.1 (d, *J*=272.7 Hz, Ar $C\underline{C}F_3$), 123.1 (Ar Phtalamide $\underline{C}H$), 122.8 (d, *J*=3.6 Hz, Ar $\underline{C}H$), 116.3 ($H_2\underline{C}$=C), 37.2 ($\underline{C}H_2N$), 33.8 ($\underline{C}H_2CH_2N$). **LCMS** (B) R_t 4.86 min, $[M+H]^+$ 346.14. **HRMS** (ESI+) *m/z* found 346.1040, $C_{19}H_{15}F_3NO_2$ (MH^+) requires 346.1055 (Δ = -4.3 ppm).

(*E*)-1-Nitro-4-styrylbenzene (Table 2, Entry 5)



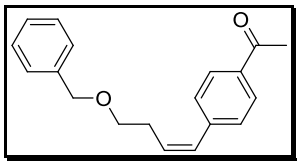
General method 1.5 followed. Purification by flash silica chromatography eluting with a gradient of dichloromethane: cyclohexane (0:100 → 50:50) to yield the product as a yellow solid (77.0 mg, 75%). $\boldsymbol{R_f}$ 0.35 [dichloromethane: hexane (50:50)]. $\boldsymbol{\nu_{max}}$ (neat)/ cm^{-1} 3080 (w, C-H), 3025 (w, C-H), 2921 (w, C-H), 1633 (w, C=C), 1595 (w, C=C), 1504 (w, C=C). δ_H (400 MHz, $CDCl_3$) 8.24-8.20 (2H, m, Ar $\underline{C}H$), 7.65-7.62 (2H, m, Ar $\underline{C}H$), 7.57-7.55 (2H, m, Ar $\underline{C}H$), 7.42-7.39 (2H, m, Ar $\underline{C}H$), 7.36-7.32 (1H, m, Ar $\underline{C}H$), 7.27 (1H, d, *J*=16.3 Hz, Ar$\underline{C}H$=CHAr), 7.14 (1H, d, *J*=16.3 Hz, Ar$\underline{C}H$=CHAr). δ_C (101 MHz, $CDCl_3$) 146.7 (Ar $\underline{C}NO_2$), 143.8 (Ar $\underline{C}$), 136.1 (Ar $\underline{C}$), 133.3 (Ar$\underline{C}H$=CHAr), 128.9 (Ar $\underline{C}H$), 128.8 (Ar $\underline{C}H$), 127.0 (Ar $\underline{C}H$), 126.8 (Ar $\underline{C}H$), 126.3 (Ar$\underline{C}H$=CHAr), 124.1 (Ar $\underline{C}H$). **Mpt.** 156.7-157.5°C (lit. value 158-159°C).[5] **Elemental analysis** (CHN) calculated for $C_{14}H_{11}NO_2$ (%) C 74.65, H 4.92, N 6.22; found C 74.51, H 5.08, N 6.07. Data consistent with that reported in the literature.[5]

1-Nitro-4-(1-phenylvinyl)benzene (Table 2, Entry 6)



General method 1.4 followed. Purification by flash silica chromatography eluting with a stepped gradient of dichloromethane: petroleum ether 40-60 (0:100 → 10:90 → 20:80) to yield product as an yellow amorphous solid (44.0 mg, 86%). $\boldsymbol{R_f}$ 0.29 [dichloromethane: petroleum ether 40-60 (30:70)]. $\boldsymbol{\nu_{max}}$ (neat)/ cm^{-1} 3080 (w, C-H), 1595 (m, C=C), 1512 (m, C=C). δ_H (400 MHz, $CDCl_3$) 8.20 (2H, d, *J*=8.9 Hz, Ar $\underline{C}H$), 7.50 (2H, d, *J*=8.9 Hz, Ar $\underline{C}H$), 7.38-7.36 (3H, m, Ar $\underline{C}H$), 7.30-7.28 (2H, m, Ar $\underline{C}H$), 5.63 (1H, s, $\underline{H}_2C$=C), 5.59 (1H, s, $\underline{H}_2C$=C). δ_C (101 MHz, $CDCl_3$) 148.4 (Ar $\underline{C}NO_2$), 148.0 (H_2C=$\underline{C}$), 147.3 (Ar $\underline{C}$), 140.1 (Ar $\underline{C}$), 129.0 (Ar $\underline{C}H$), 128.5 (Ar $\underline{C}H$), 128.3 (Ar $\underline{C}H$), 128.1 (Ar $\underline{C}H$), 123.5 (Ar $\underline{C}H$), 117.2 ($H_2\underline{C}$=C). **Mpt.** 64.5-66.9°C (lit. value 66-66.5°C).[6] **HRMS** (EI) *m/z* found 225.0795, $C_{14}H_{11}NO_2$ (M^+) requires 225.0784 (Δ = -4.8 ppm). Data consistent with that reported in the literature, only melting point described.[6]

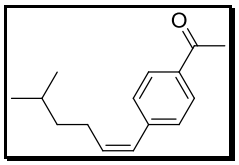
(*Z*)-1-(4-(4-(Benzyloxy)but-1-enyl)phenyl)ethanone (Table 3, Entry 1)



General method 1.6 followed. Purification by flash silica chromatography eluting with a stepped gradient of dichloromethane: petroleum ether 40-60 (50:50 → 75:25) to yield the product as a colourless oil (50.0 mg, 66%). $\boldsymbol{R_f}$ 0.12 [dichloromethane]. $\boldsymbol{\nu_{max}}$ (neat)/ cm^{-1} 2959 (w, C-H), 1716 (m, C=O), 1680 (s, C=O ketone), 1608 (m, C=C), 1572 (w, C=C), 1503 (w, C=C). δ_H (400 MHz, $CDCl_3$) 7.91 (2H, d, *J*=8.4 Hz, Ar $\underline{C}H$), 7.38 (2H, d, *J*=8.3 Hz, Ar $\underline{C}H$), 7.35-7.26 (5H, m, Ar $\underline{C}H$), 6.54

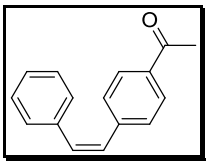
(1H, d, *J*=11.7 Hz, ArC<u>H</u>=CHCH$_2$), 5.85 (1H, dt, *J*=11.7, 7.3 Hz, ArCH=C<u>H</u>CH$_2$), 4.53 (2H, s, OC<u>H</u>$_2$Ar), 3.59 (2H, t, *J*=6.6 Hz, CH$_2$C<u>H</u>$_2$O), 2.66 (2H, qd, *J*=6.7, 1.9 Hz, ArCH=CHC<u>H</u>$_2$), 2.60 (3H, s, C=OC<u>H</u>$_3$). δ_C (101 MHz, $CDCl_3$) 198.1 (<u>C</u>=O), 142.7 (Ar <u>C</u>), 138.7 (Ar <u>C</u>), 135.7 (Ar <u>C</u>), 131.7 (ArCH=<u>C</u>H), 130.1 (Ar<u>C</u>H=CH), 129.3 (Ar <u>C</u>H), 128.8 (Ar <u>C</u>H), 128.7 (Ar <u>C</u>H), 128.0 (2H Ar <u>C</u>H), 73.4 (O<u>C</u>H$_2$Ar), 70.0 (CH$_2$<u>C</u>H$_2$O), 29.9 (ArCH=CH<u>C</u>H$_2$), 27.0 (C=O<u>C</u>H$_3$). **LCMS** (B) R_t 4.80 min, $[M+H]^+$ 281.17.

(*Z*)-1-(4-(5-Methylhex-1-enyl)phenyl)ethanone (Table 3, Entry 2)



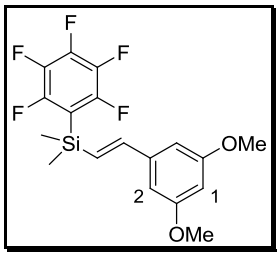
General method 1.6 followed. Purification by flash silica chromatography eluting with a stepped gradient of dichloromethane: petroleum ether 40-60 (0:100 → 25:75 → 50:50) to yield the product as a colourless oil (41.0 mg, 79%). R_f 0.29 [dichloromethane: petroleum ether 40-60 (25:75)]. ν_{max} (neat)/ cm^{-1} 2959 (w, C-H), 1680 (s, C=O ketone), 1609 (w, C=C), 1571 (s, C=C), 1506 (w, C=C). δ_H (400 MHz, $CDCl_3$) 7.92 (2H, d, *J*=8.4 Hz, Ar C<u>H</u>), 7.36 (2H, d, *J*=8.3 Hz, Ar C<u>H</u>) 6.42 (1H, d, *J*=11.7 Hz, ArC<u>H</u>=CHCH$_2$), 5.78 (1H, dt, *J*=11.7, 7.4 Hz, ArCH=C<u>H</u>CH$_2$), 2.60 (3H, s, COC<u>H</u>$_3$), 2.34 (2H, qd, *J*=7.6, 1.7 Hz, ArCH=CHC<u>H</u>$_2$), 1.59 (1H, sept, *J*=6.7 Hz, C<u>H</u>(CH$_3$)$_2$), 1.36 (2H, m, C<u>H</u>$_2$CH(CH$_3$)$_2$), 0.89 (6H, d, *J*=6.6 Hz, CH(C<u>H</u>$_3$)$_2$). δ_C (101 MHz, $CDCl_3$) 197.7 (<u>C</u>=O), 142.7 (Ar <u>C</u>), 135.8 (ArCH=<u>C</u>H), 135.1 (Ar <u>C</u>), 128.8 (Ar <u>C</u>H), 128.3 (Ar <u>C</u>H), 127.8 (Ar<u>C</u>H=CH), 38.9 (ArCH=CH<u>C</u>H$_2$), 27.7 (<u>C</u>H(CH$_3$)$_2$), 26.7 (<u>C</u>H$_2$CH(CH$_3$)$_2$), 26.6 (C=O<u>C</u>H$_3$), 22.4 (CH(<u>C</u>H$_3$)$_2$). **LCMS** (B) R_t 5.06 min, $[M+H]^+$ 217.22.

(*Z*)-1-(4-styrylphenyl)ethanone (Table 3, Entry 3)



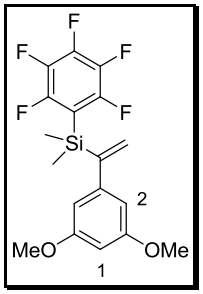
General method 1.6 followed. Purification by flash silica chromatography eluting with a stepped gradient of dichloromethane: petroleum ether 40-60 (0:100 → 25:75 → 50:50) to yield the product as a white solid (18 mg, 70%). R_f 0.15 [dichloromethane: petroleum ether 40-60 (50:50)]. ν_{max} (neat)/ cm^{-1} 3015 (w, C-H), 2920 (w, C-H), 2850 (w, C-H), 1678 (s, C=O α,β-unsaturated ketone), 1602 (m, C=C), 1560 (w, C=C). δ_H (400 MHz, $CDCl_3$) 7.81 (2H, d, *J*=8.4 Hz, Ar C<u>H</u>), 7.32 (2H, d, *J*=8.2 Hz, Ar C<u>H</u>), 7.24-7.20 (5H, m, Ar C<u>H</u>), 6.73 (1H, d, *J*=12.3 Hz, ArC<u>H</u>=CHAr), 6.61 (1H, d, *J*=12.3 Hz, ArCH=C<u>H</u>Ar), 2.57 (3H, s, C=OC<u>H</u>$_3$). δ_C (101 MHz, $CDCl_3$) 197.6 (C=O), 142.3 (Ar <u>C</u>COCH$_3$), 136.7 (Ar <u>C</u>), 135.6 (Ar <u>C</u>), 132.4 (Ar<u>C</u>H=CHAr), 129.1 (Ar <u>C</u>H), 129.0 (Ar <u>C</u>H), 128.8 (Ar <u>C</u>H), 128.4 (Ar <u>C</u>H), 128.3 (Ar <u>C</u>H), 127.5 (Ar<u>C</u>H=CHAr), 26.5 (C=O<u>C</u>H$_3$). **LCMS** (B) R_t 4.78 min, $[M+H]^+$ 223.14. **HRMS** (ESI+) *m/z* found 223.1125, $C_{16}H_{15}O$ (MH^+) requires 223.1123 (Δ = 0.9 ppm). No analytical data found, compound referenced.[7]

(*E*)-(3,5-dimethoxystyryl)dimethyl(perfluorophenyl)silane (Scheme 1, 8-(*E*))



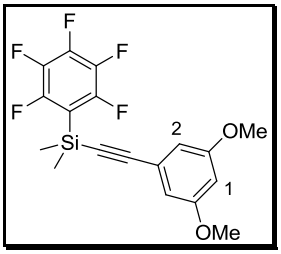
General method 1.1 followed. Purification by flash silica chromatography eluting with a stepped gradient of dichloromethane: petroleum ether 40-60 (5:95 → 10:90) to yield the product as a colourless oil (433 mg, 90%). R_f 0.19 [dichloromethane: petroleum ether 40-60 (20:80)]. ν_{max} (neat)/ cm^{-1} 2961 (w, C-H), 2840 (w, C-H), 1642 (w, C=C), 1582 (m, C=C), 1515 (m, C=C). δ_H (400 MHz, $CDCl_3$) 6.91 (1H, d, *J*=19.1 Hz, SiCH=CHAr), 6.60 (2H, d, *J*=2.3 Hz, Ar CH2), 6.55 (1H, dt, *J*=19.1, 1.3 Hz, SiCH=CH), 6.42 (1H, t, *J*=2.3 Hz, Ar CH1), 3.81 (6H, s, OCH_3), 0.57 (6H, t, *J*=1.6 Hz, $Si(CH_3)_2$). δ_F (376 MHz, $CDCl_3$) -127.00 (2F, m), -152.10 (1F, m), -161.75 (2F, m). δ_C (101 MHz, $CDCl_3$) 161.0 (Ar $COCH_3$), 145.9 (SiCH=CH), 139.6 (Ar C), 125.0 (SiCH=CH), 104.6 (Ar CH), 101.1 (Ar CH), 55.4 (OCH_3), -1.5 ($Si(CH_3)_2$). **LCMS** (B) R_t 5.33 min, $[M+H]^+$ 389.06. **HRMS** (ESI+) *m/z* found 389.0994, $C_{18}H_{18}F_5O_2Si$ (MH^+) requires 389.0996 (Δ = -0.5 ppm).

(1-(3,5-Dimethoxyphenyl)vinyl)dimethyl(perfluorophenyl)silane (Scheme 1, 8-α)



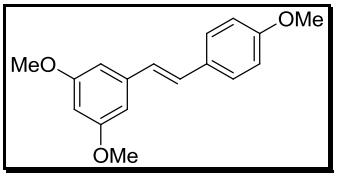
General method 1.3 followed. Purification by flash silica chromatography eluting with a stepped gradient of dichloromethane: petroleum ether 40-60 (0:100 → 10:90) to yield the product as an amorphous white solid (273 mg, 76%). R_f 0.31 [dichloromethane: petroleum ether 40-60 (20:80)]. ν_{max} (neat)/ cm^{-1} 3006 (w, C-H), 2938 (w, C-H), 2907 (w, C-H), 2835 (w, C-H), 1640 (m, C=C), 1603 (s, C=C), 1592 (s, C=C), 1515 (s, C=C). δ_H (400 MHz, $CDCl_3$) 6.34 (1H, t, *J*=2.3 Hz, Ar CH1), 6.26 (2H, d, *J*=2.3 Hz, Ar CH2), 6.00 (1H, d, *J*=2.3 Hz, H_2C=C), 5.69 (1H, d, *J*=2.3 Hz, H_2C=C), 3.74 (6H, s, OCH_3), 0.57 (6H, t, *J*=1.8 Hz, $Si(CH_3)_2$). δ_F (376 MHz, $CDCl_3$) -126.48 (2F, m), -151.50 (1F, m), -161.58 (2F, m). δ_C (101 MHz, $CDCl_3$) 160.6 (Ar $COCH_3$), 149.2 (H_2C=C(Si)CH_2), 145.1 (Ar C), 129.6 (H_2C=C(Si)CH_2), 104.8 (Ar CH), 98.9 (Ar CH), 55.2 (OCH_3), -0.8 ($Si(CH_3)_2$). **LCMS** (B) R_t 5.28 min, $[M+H]^+$ 389.10. **HRMS** (ESI+) *m/z* found 389.1004, $C_{18}H_{18}F_5O_2Si$ (MH^+) requires 389.0996 (Δ = 2.1 ppm).

((3,5-Dimethoxyphenyl)ethynyl)dimethyl(perfluorophenyl)silane (Scheme 1, 9)



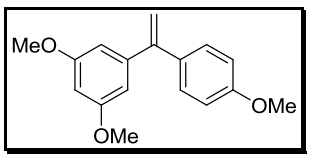
General method 1.6 followed. Purification by flash silica chromatography eluting with a stepped gradient of dichloromethane: petroleum ether 40-60 (0:100 → 5:95→ 10:90) to yield the product as a colourless oil (635 mg, 89%). R_f 0.50 [dichloromethane: petroleum ether 40-60 (30:70)]. ν_{max} (neat)/ cm^{-1} 2963 (w, C-H), 2842 (w, C-H), 2164 (m, C≡C), 1644 (m, C=C), 1587 (s, C=C), 1517 (s, C=C). δ_H (400 MHz, $CDCl_3$) 6.61 (2H, d, *J*=2.3 Hz, Ar CH2), 6.46 (1H, t, *J*=2.3 Hz, Ar CH1), 3.77 (6H, s, OCH_3), 0.62 (6H, t, *J*=1.6 Hz). δ_F (376 MHz, $CDCl_3$) -126.92 (2F, m), -151.41 (1F, m), -161.65 (2F, m). δ_C (101 MHz, $CDCl_3$) 160.5 (Ar $COCH_3$), 123.5 (Ar C), 109.8 (Ar CH), 107.2 (SiC≡CAr), 102.8 (Ar CH), 89.1 (SiC≡CAr), 55.4 (OCH_3), 0.2 ($Si(CH_3)_2$). **LCMS** (B) R_t 5.33 min, $[M+H]^+$ 387.06. **HRMS** (ESI+) *m/z* found 387.0853, $C_{18}H_{16}F_5O_2Si$ (MH^+) requires 387.0840 (Δ = 3.4 ppm).

(*E*)-1,3-Dimethoxy-5-(4-methoxystyryl)benzene (Scheme 1, 10-(*E*))



General method 1.4 followed. Purification by flash silica chromatography eluting with a stepped gradient of petroleum ether: dichloromethane (100: 0 → 90:10 → 80:20 → 70:30) to yield product as an white solid (38.0 mg, 73%). R_f 0.25 [dichloromethane: petroleum ether (50:50)]. ν_{max} (neat)/ cm^{-1} 3074 (w, C-H), 2990 (m, C-H), 2932 (m, C-H), 2832 (m, C-H), 1588 (s, C=C), 1510 (m, C=C). δ_H (400 MHz, $CDCl_3$) 7.45 (2H, d, *J*=8.7 Hz, Ar CH), 7.04 (1H, d, *J*=16.2 Hz, ArCH=CHAr), 6.91 (1H, d, *J*=16.1 Hz, ArCH=CHAr), 6.90 (2H, d, *J*=8.8 Hz, Ar CH), 6.65 (2H, d, *J*=2.2 Hz, Ar CH), 6.38 (1H, t, *J*=2.2 Hz, Ar CH), 3.83 (9H, s, $ArOCH_3$). δ_C (101 MHz, $CDCl_3$) 161.0 (Ar $COCH_3$), 159.4 (Ar $COCH_3$), 139.7 (Ar C), 129.9 (Ar C), 128.7 (ArCH=CHAr), 127.8 (Ar CH), 126.6 (ArCH=CHAr), 114.1 (Ar CH), 104.3 (Ar CH), 99.6 (Ar CH), 55.4 (OCH_3). **Mpt.** 55.3-56.6°C (lit. value 55°C).[8] **LCMS** (B) R_t 4.91 min, $[M+H]^+$ 271.17. Data consistent with that reported in the literature.[8]

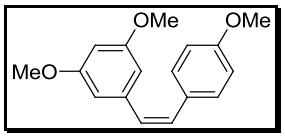
1,3-Dimethoxy-5-(1-(4-methoxyphenyl)vinyl)benzene (Scheme 1, 10-α)



General method 1.4 followed. Purification by flash silica chromatography eluting with a stepped gradient of dichloromethane: petroleum ether 40-60 (5:95 → 10:90 → 20:80) to yield the product as a colourless oil (40.0 mg, 77%). R_f 0.35 [dichloromethane: petroleum ether 40-60 (50:50)]. ν_{max} (neat)/ cm^{-1} 3001 (w, C-H), 2936 (w, C-H), 2837 (w, C-H), 1652 (w, C=C), 1591 (s, C=C), 1509 (s, C=C). δ_H (400 MHz, $CDCl_3$) 7.28 (2H, d, *J*=8.9 Hz, Ar CH), 6.86 (2H, d, *J*=8.9 Hz, Ar CH), 6.49

(2H, d, *J*=2.3 Hz, Ar C<u>H</u>), 6.44 (1H, t, *J*=2.3 Hz, Ar C<u>H</u>), 6.39 (1H, d, *J*=1.3 Hz, <u>H</u>$_2$C=C), 6.35 (1H, d, *J*=1.3 Hz, <u>H</u>$_2$C=C), 3.83 (3H, s, OC<u>H</u>$_3$), 3.77 (6H, s, OC<u>H</u>$_3$). δ_C (101 MHz, $CDCl_3$) 160.5 (Ar <u>C</u>OCH$_3$), 159.3 (Ar <u>C</u>OCH$_3$), 149.4 (H$_2$C=<u>C</u>), 144.0 (Ar <u>C</u>), 133.7 (Ar <u>C</u>), 129.3 (Ar <u>C</u>H), 113.5 (Ar <u>C</u>H), 113.0 (H$_2$<u>C</u>=C), 106.6 (Ar <u>C</u>H), 99.6 (Ar <u>C</u>H), 55.4 (O<u>C</u>H$_3$), 55.3 (O<u>C</u>H$_3$). **LCMS** (B) R_t 4.85 min, $[M+H]^+$ 271.17. **HRMS** (ESI+) *m/z* found 271.1340, $C_{17}H_{19}O_3$ (MH^+) requires 271.1334 (Δ = 2.2 ppm).

(*Z*)-1,3-dimethoxy-5-(4-methoxystyryl)benzene (Scheme 1, 10-(*Z*))



General method 1.6 followed. Purification by flash silica chromatography eluting with a stepped gradient of dichloromethane: petroleum ether 40-60 (0:100 → 10:90 → 20:80 → 30:70 → 40:60) to yield product as a colourless oil (30.0 mg, 56%). R_f 0.23 [dichloromethane: petroleum ether 40-60 (50:50)]. ν_{max} (neat)/ cm^{-1} 3001 (w, C-H), 2934 (w, C-H), 2836 (w, C-H), 1587 (s, C=C), 1509 (s, C=C). δ_H (400 MHz, $CDCl_3$) 7.22 (2H, d, *J*=8.8 Hz, Ar C<u>H</u>), 6.77 (2H, d, *J*=8.8 Hz, Ar C<u>H</u>), 6.53 (1H, d, *J*=12.3 Hz, ArC<u>H</u>=CHAr), 6.44 (1H, d, *J*=12.3 Hz, ArCH=C<u>H</u>Ar), 6.44 (2H, d, *J*=2.3 Hz, Ar C<u>H</u>), 6.32 (1H, t, *J*=2.3 Hz, Ar C<u>H</u>), 3.78 (3H, s, OC<u>H</u>$_3$), 3.67 (6H, s, OC<u>H</u>$_3$). δ_C (101 MHz, $CDCl_3$) 160.6 (Ar <u>C</u>OCH$_3$), 158.7 (Ar <u>C</u>OCH$_3$), 139.5 (Ar <u>C</u>), 130.3 (Ar <u>C</u>H), 130.1 (ArCH=<u>C</u>HAr), 129.6 (Ar <u>C</u>), 128.7 (Ar<u>C</u>H=CHAr), 113.5 (Ar <u>C</u>H), 106.6 (Ar <u>C</u>H), 99.7 (Ar <u>C</u>H), 55.2 (O<u>C</u>H$_3$). **LCMS** (B) R_t 4.87 min, $[M+H]^+$ 271.17. **HRMS** (ESI+) *m/z* found 271.1339, $C_{17}H_{19}O_3$ (MH^+) requires 271.1334 (Δ = 1.8 ppm). Data consistent with that reported in the literature.[9]

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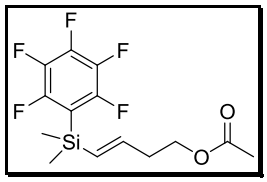
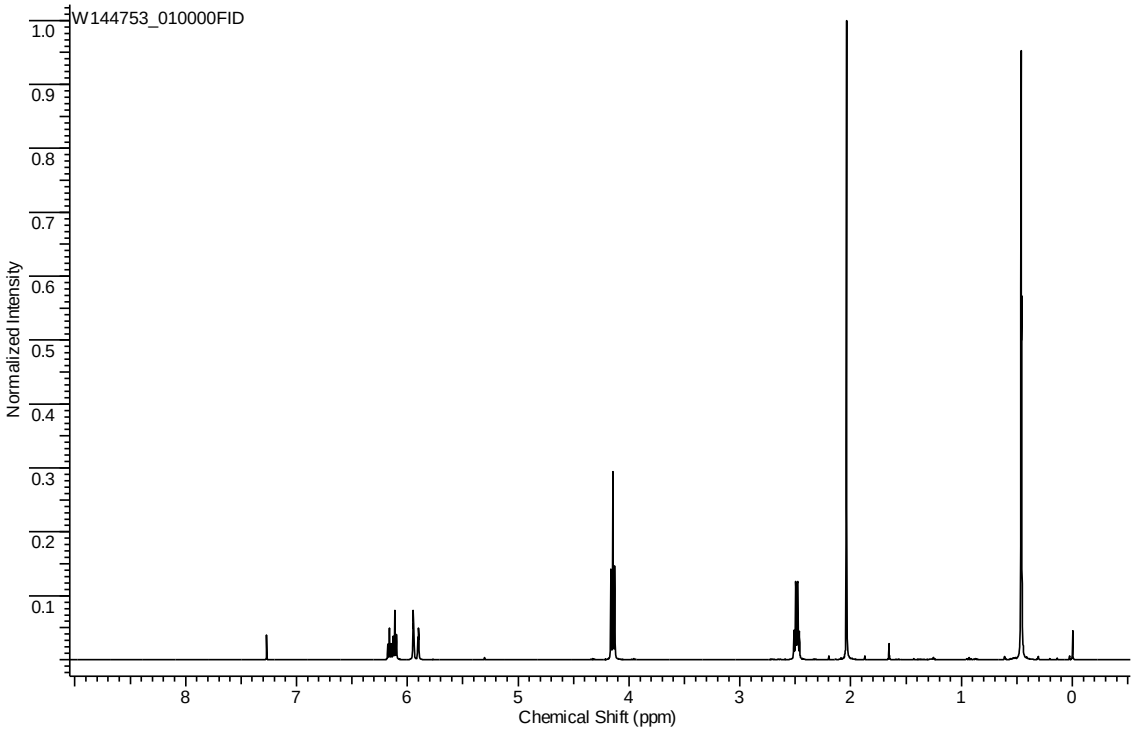
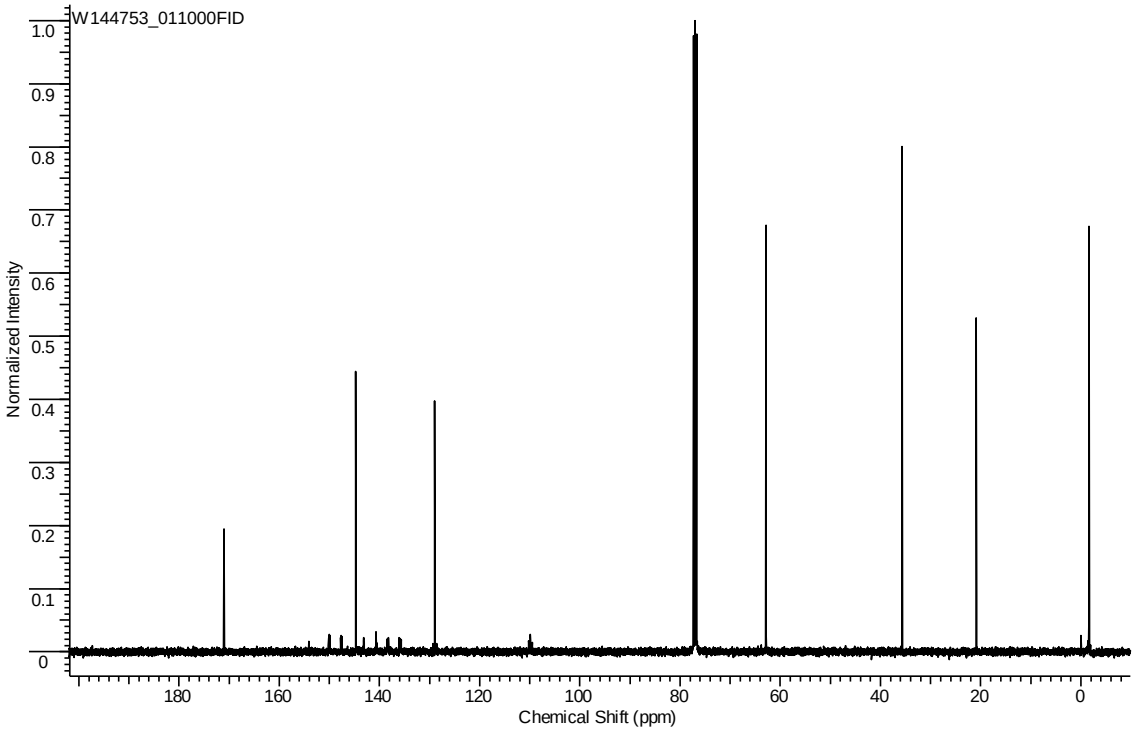
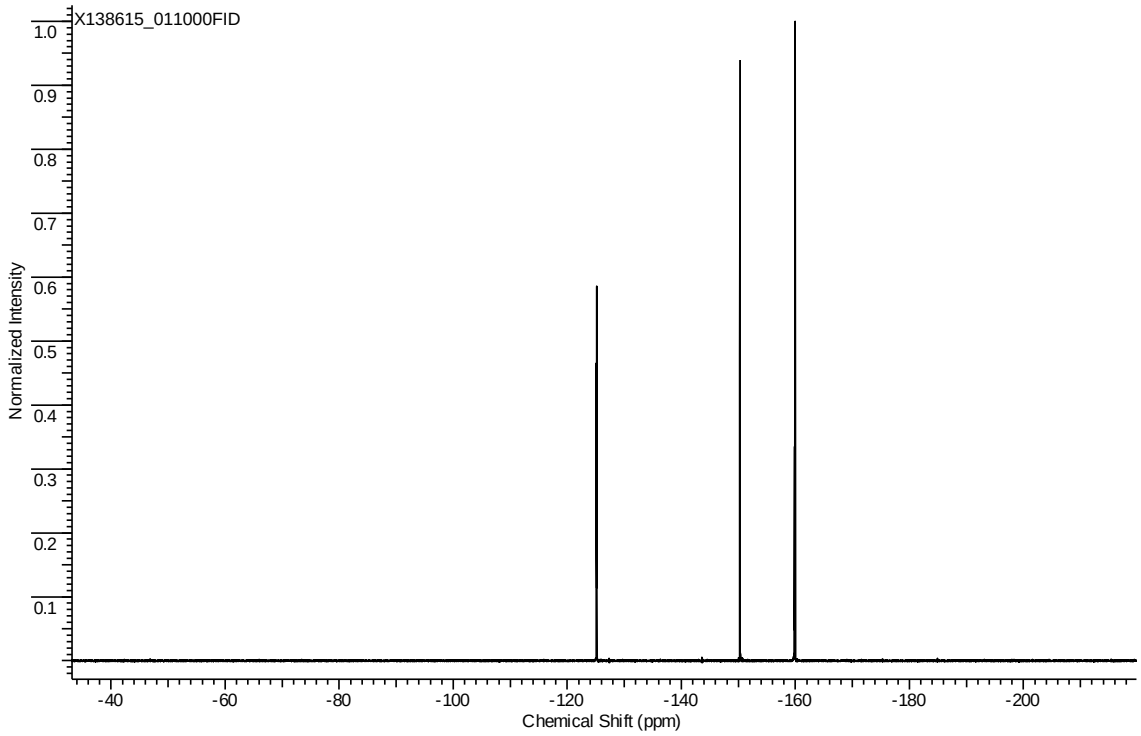
(*E*)-4-(Dimethyl(perfluorophenyl)silyl)but-3-enyl acetate (Table 1, Entry 1)

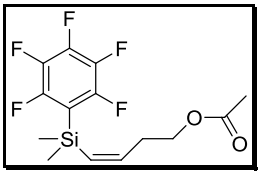


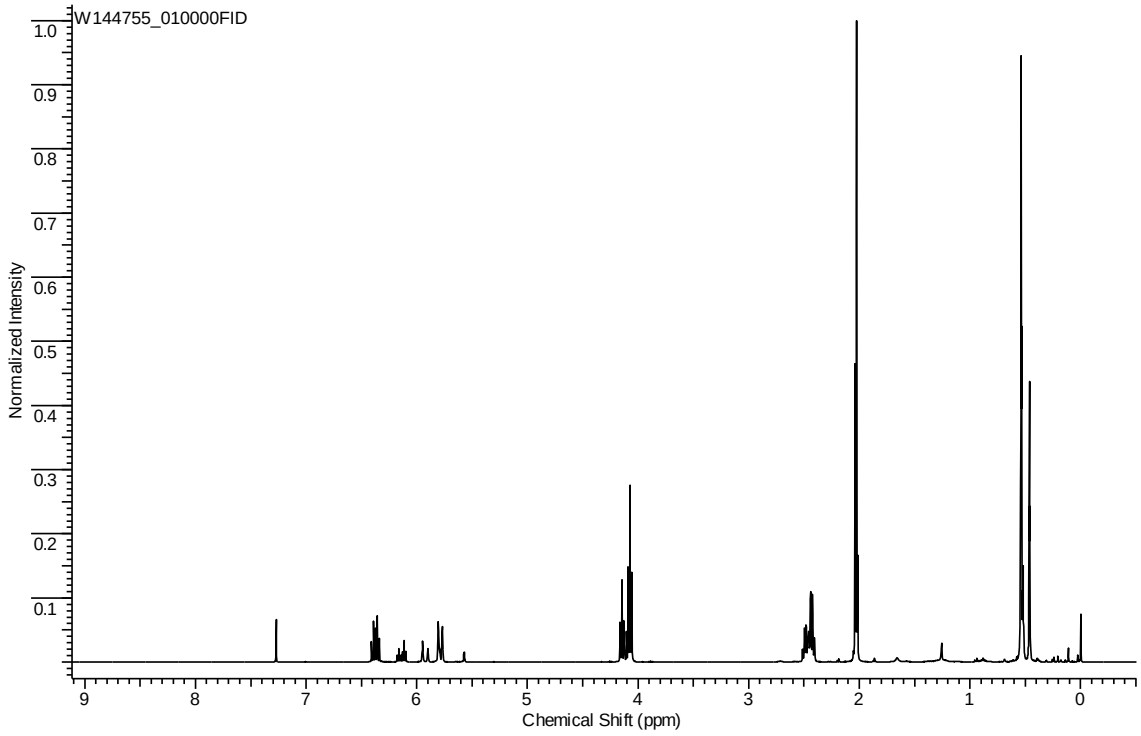


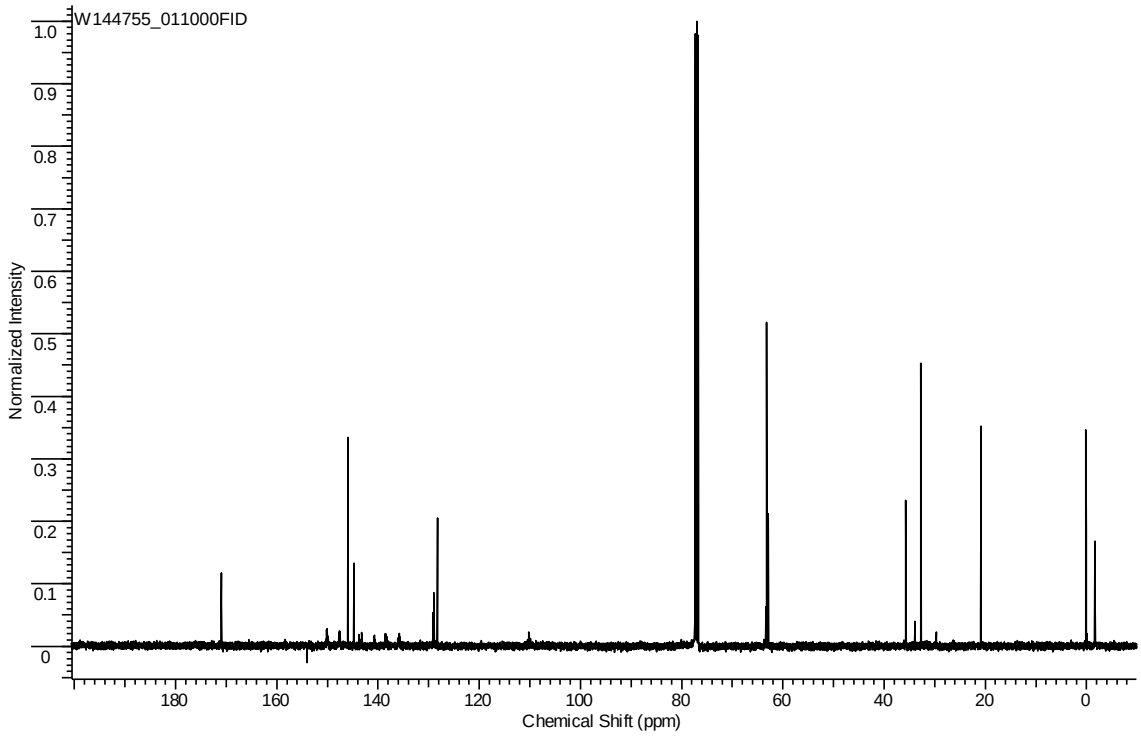




(*Z*)-4-(Dimethyl(perfluorophenyl)silyl)but-3-enyl acetate (Table 1, Entry 2)



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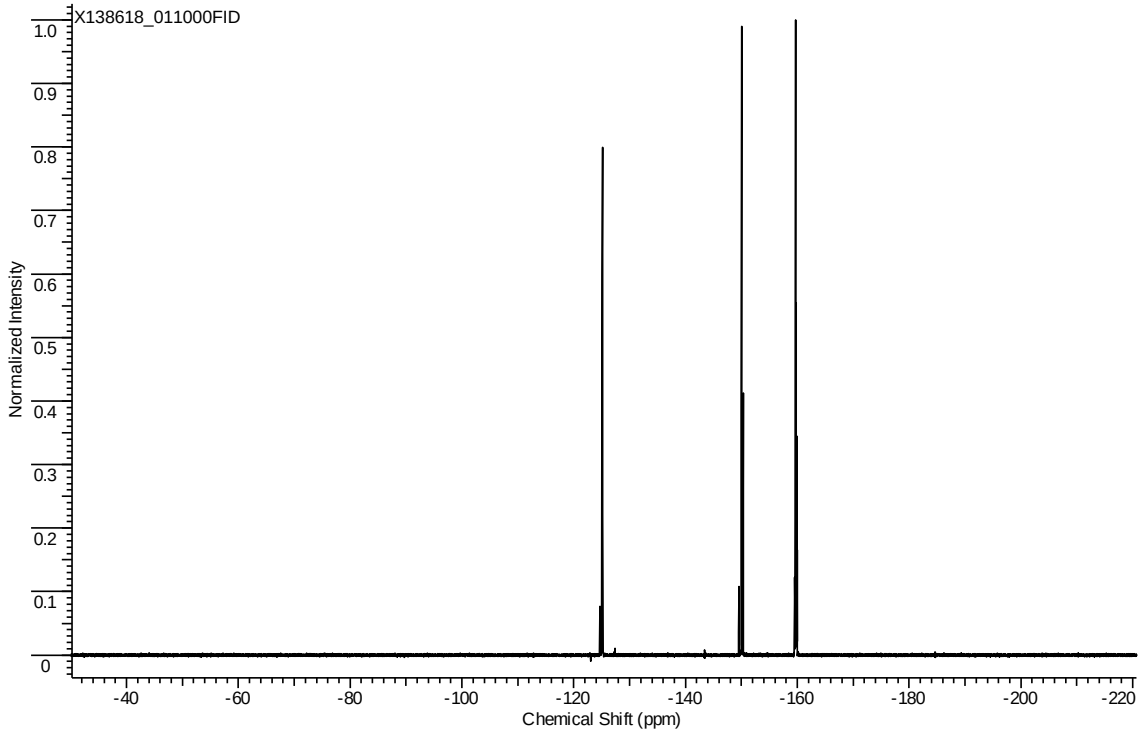
0.6
0.5
0.4
0.3
0.2
0.1
0
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


X138618_011000FID
Normalized Intensity
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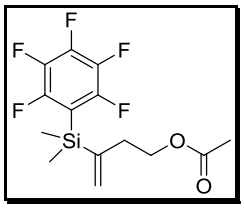
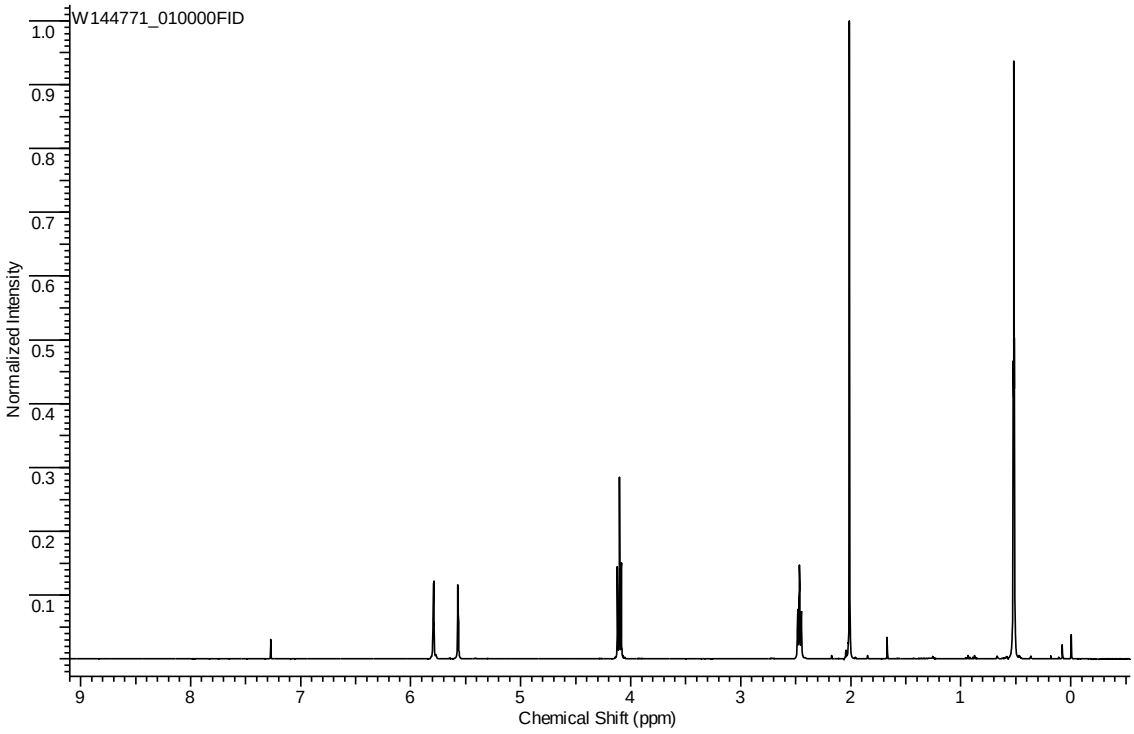
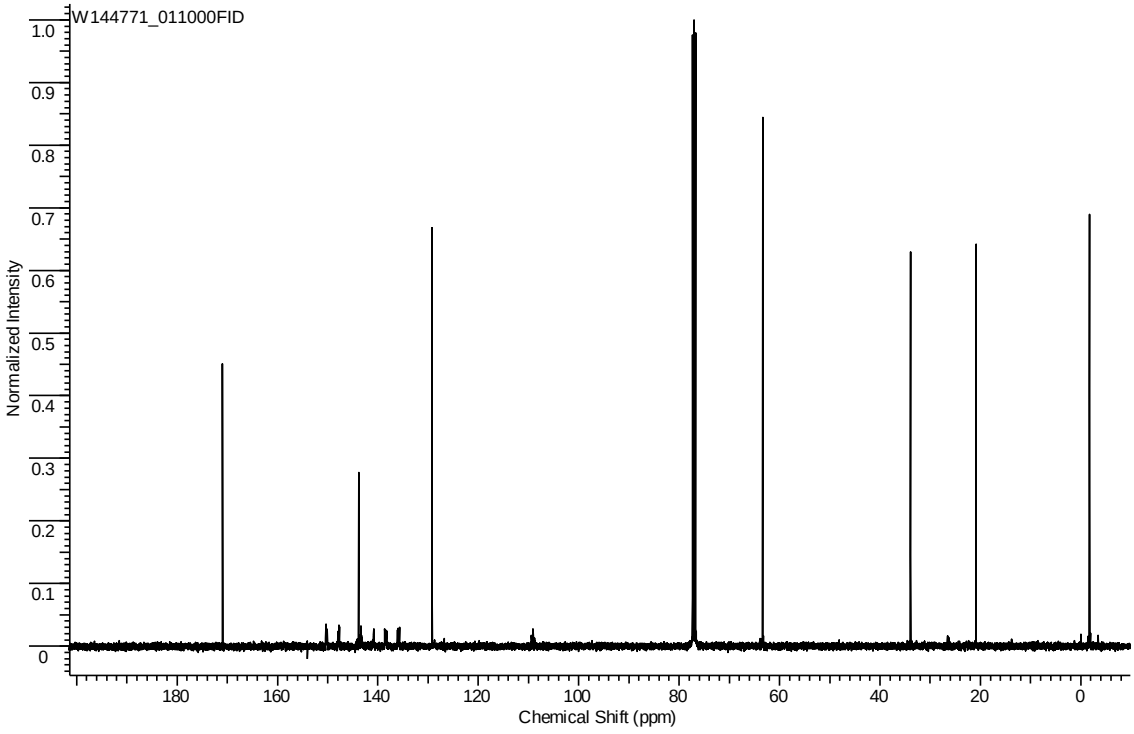
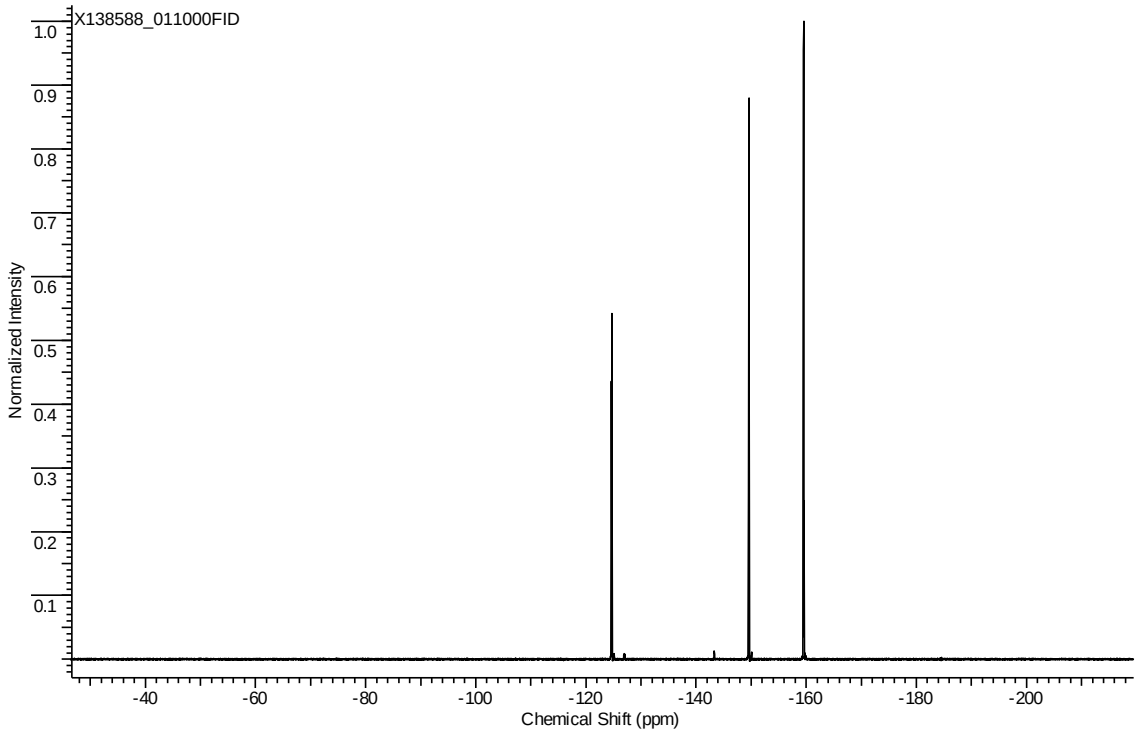
0.6
0.5
0.4
0.3
0.2
0.1
0
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
Chemical Shift (ppm)

3-(Dimethyl(perfluorophenyl)silyl)but-3-enyl acetate (Table 1, Entry 3)

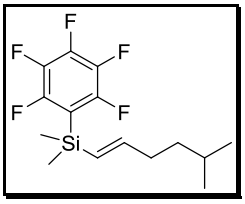


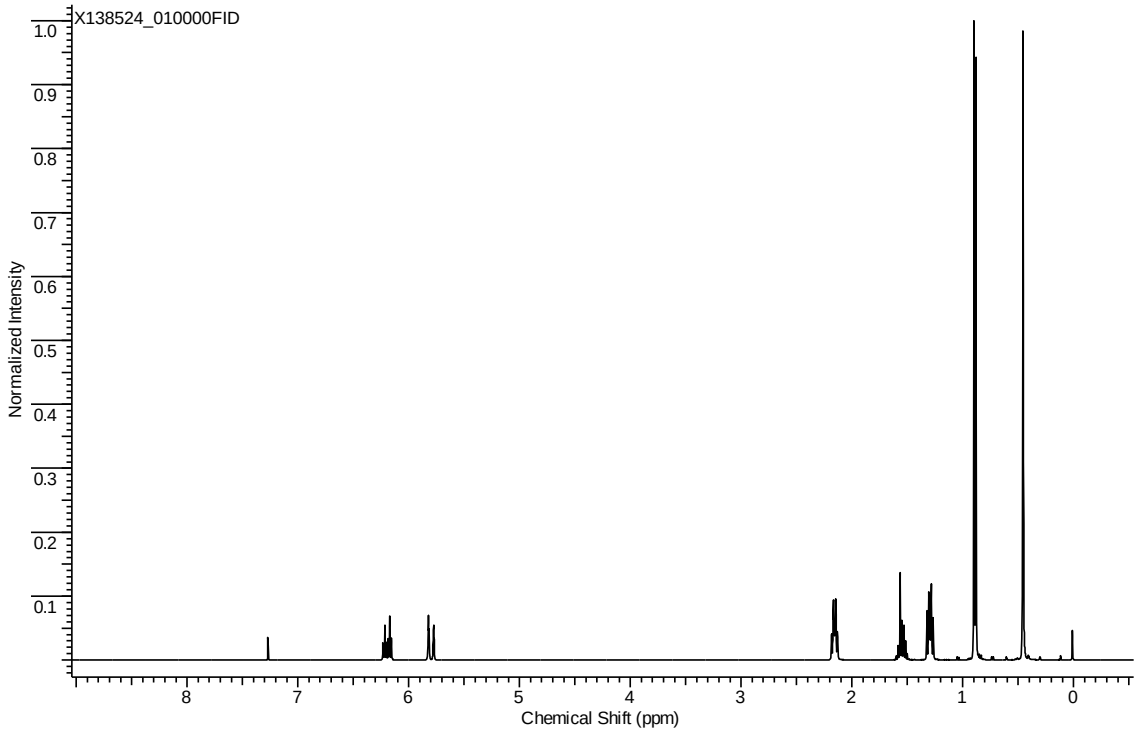


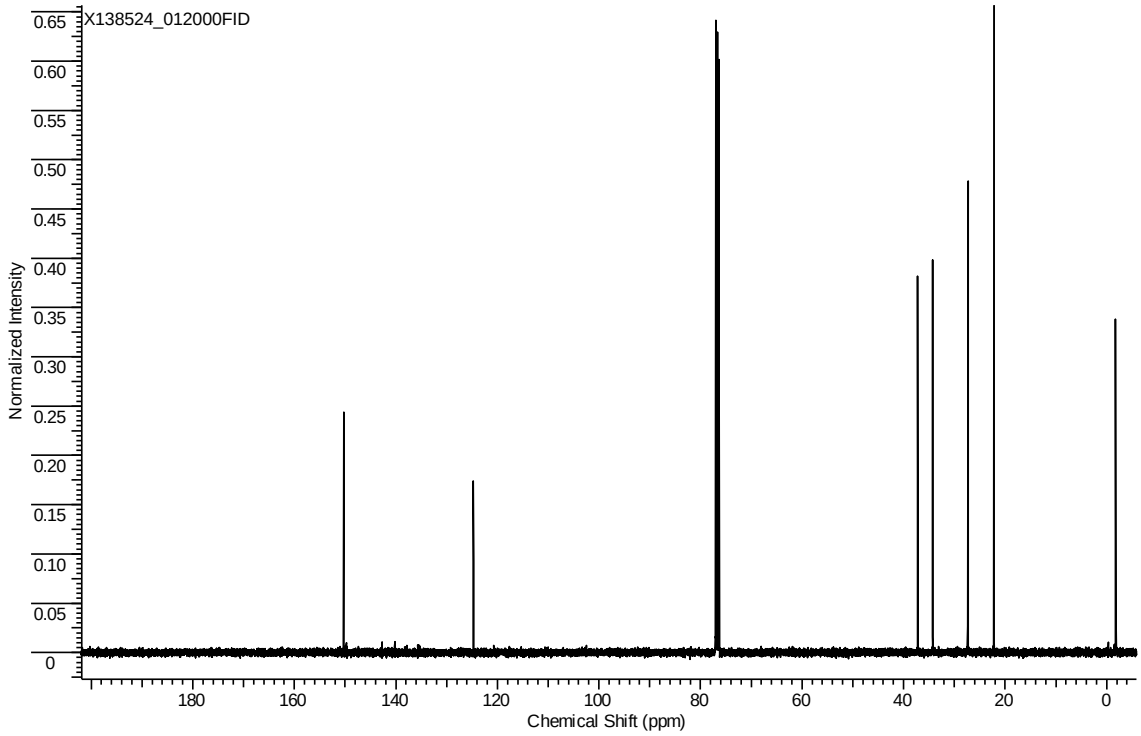




(*E*)-Dimethyl(5-methylhex-1-enyl)(perfluorophenyl)silane (Table 1, Entry 4)



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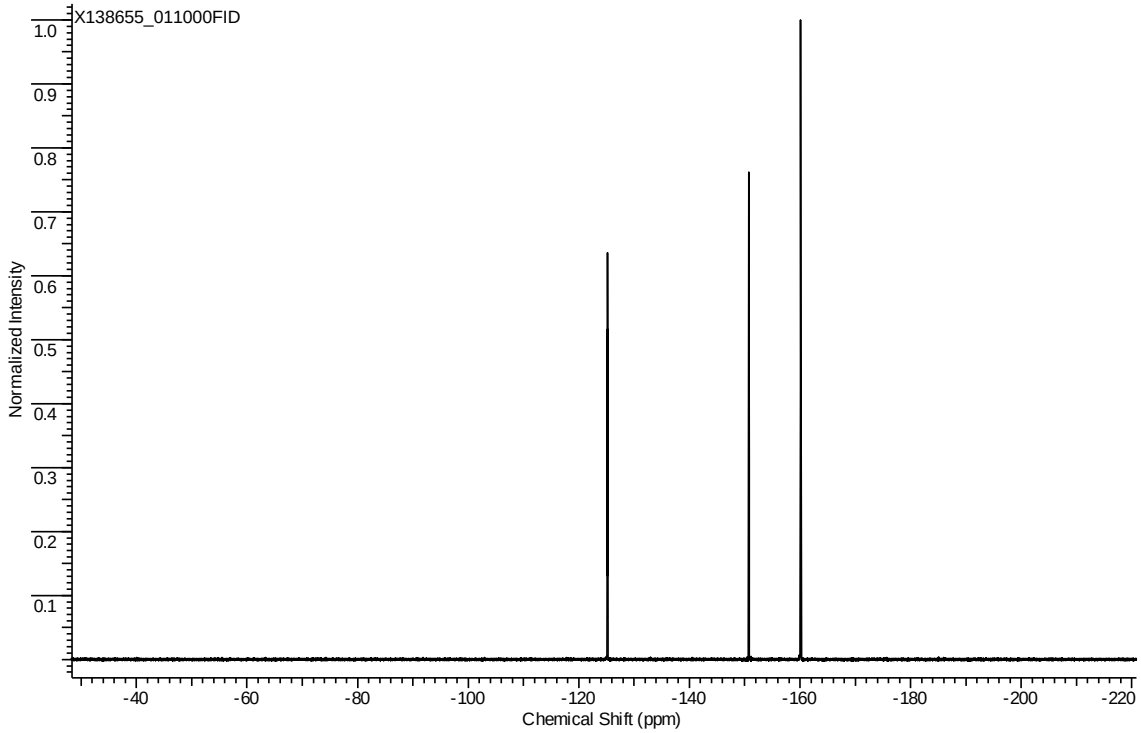
0.15
0.10
0.05
0
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


X138655_011000FID
Normalized Intensity
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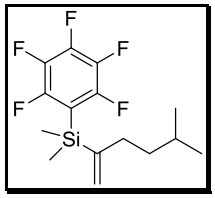
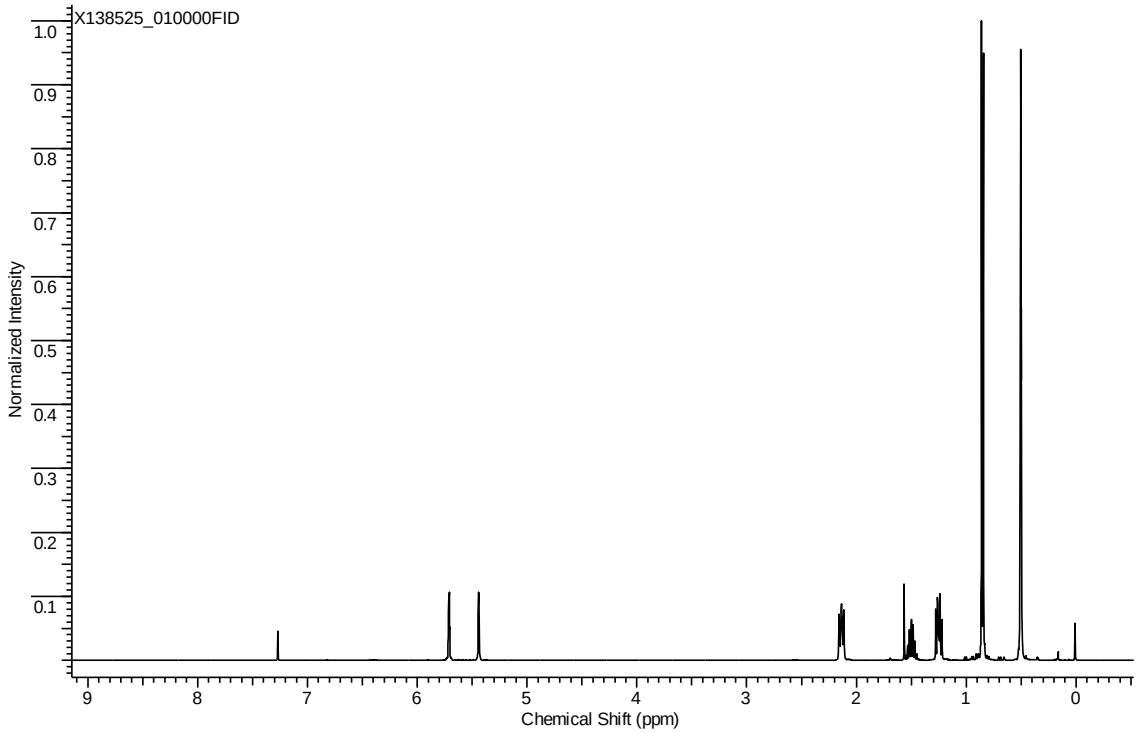
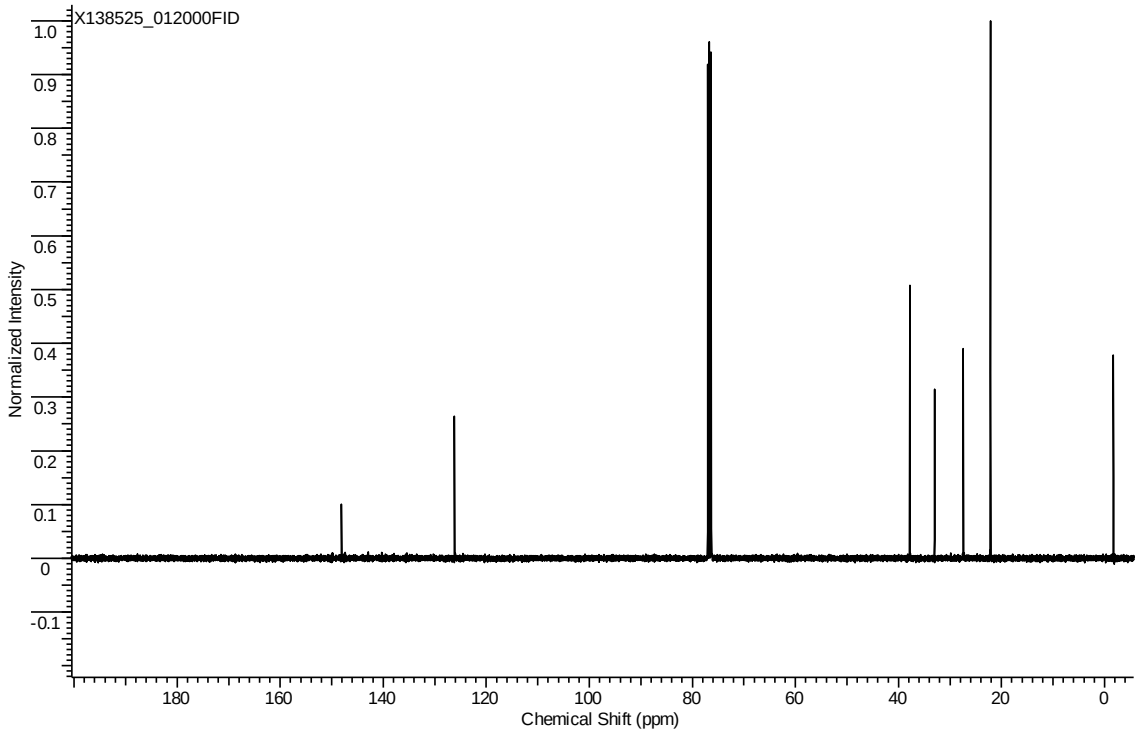
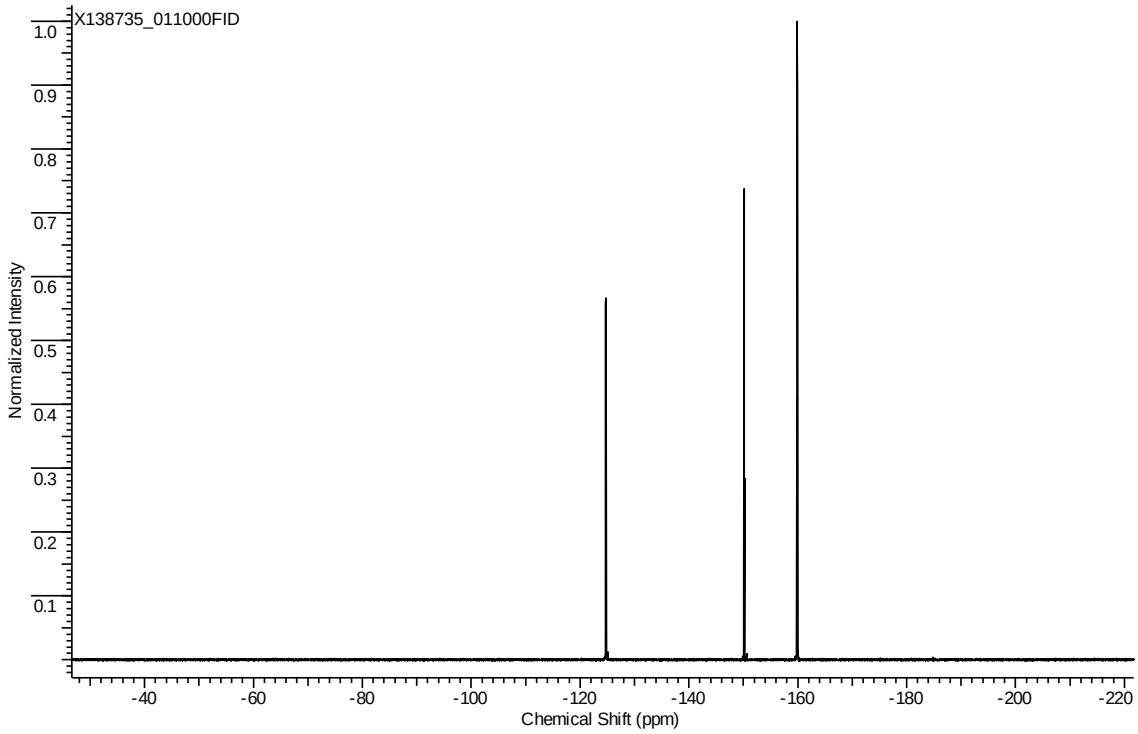
0.5
0.4
0.3
0.2
0.1
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
Chemical Shift (ppm)

Dimethyl(5-methylhex-1-en-2-yl)(perfluorophenyl)silane (Table 1, Entry 5)

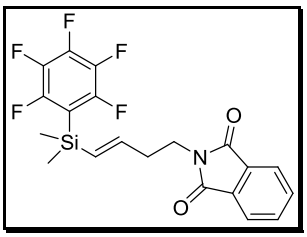


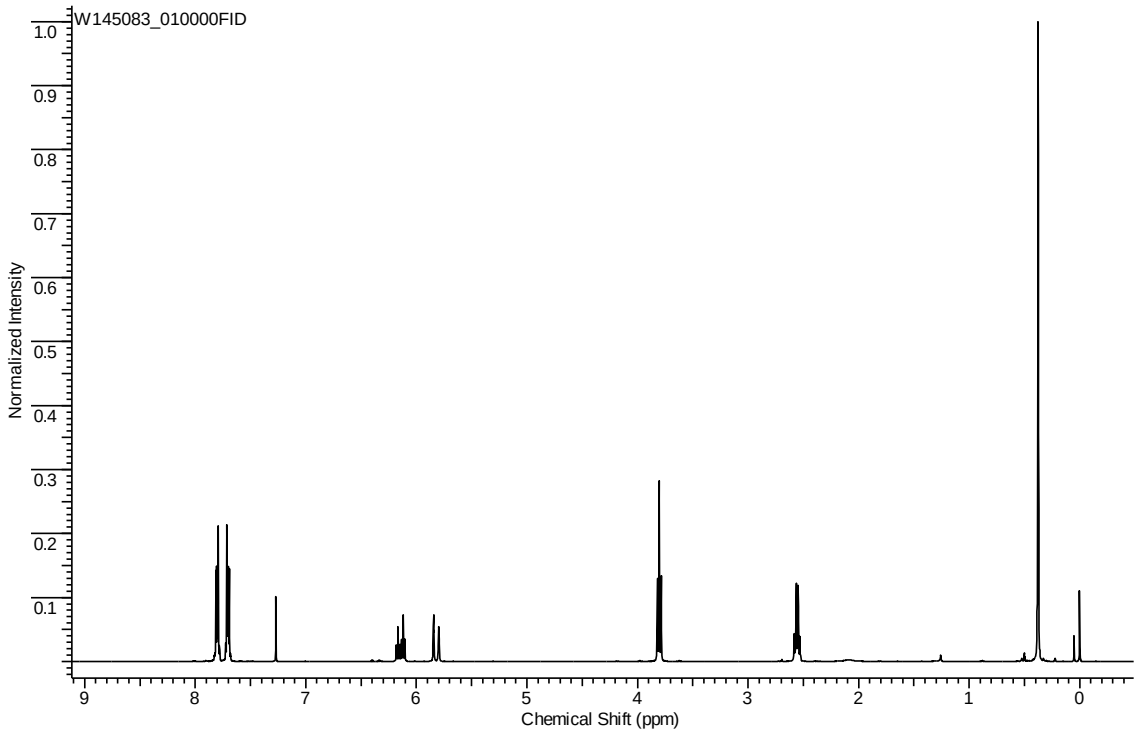


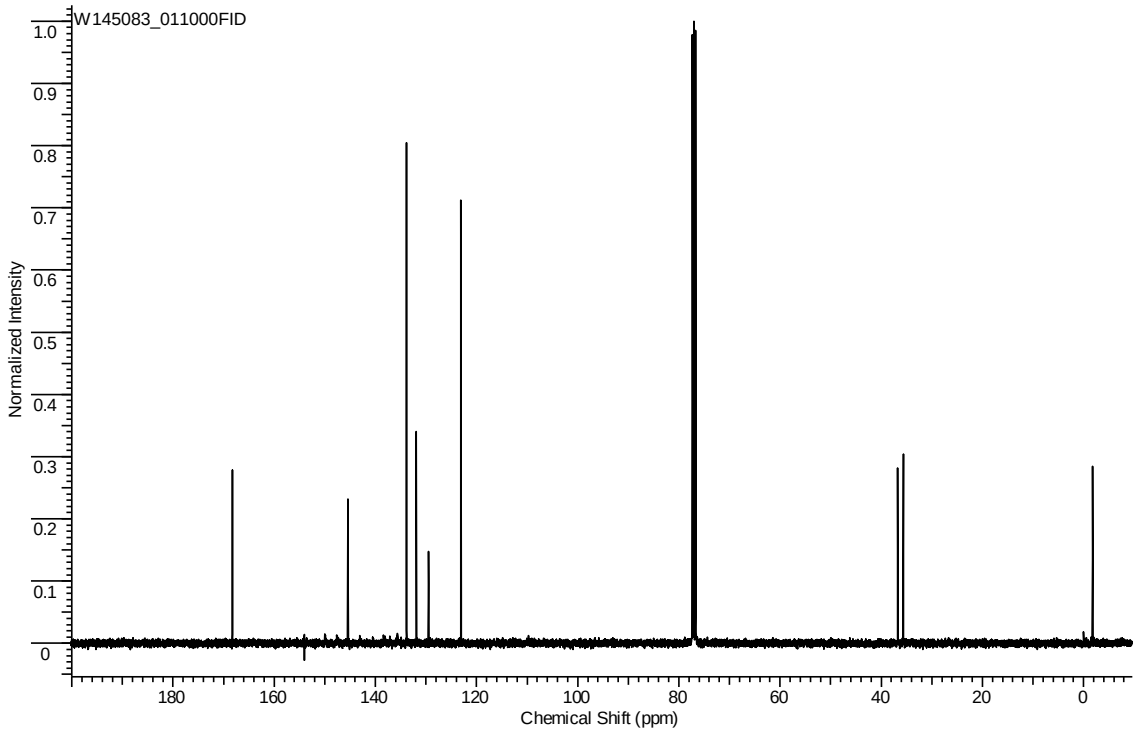




(*E*)-2-(4-(Dimethyl(perfluorophenyl)silyl)but-3-enyl)isoindoline-1,3-dione (Table 1, Entry 6)



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Normalized Intensity
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0.6
0.5
0.4
0.3
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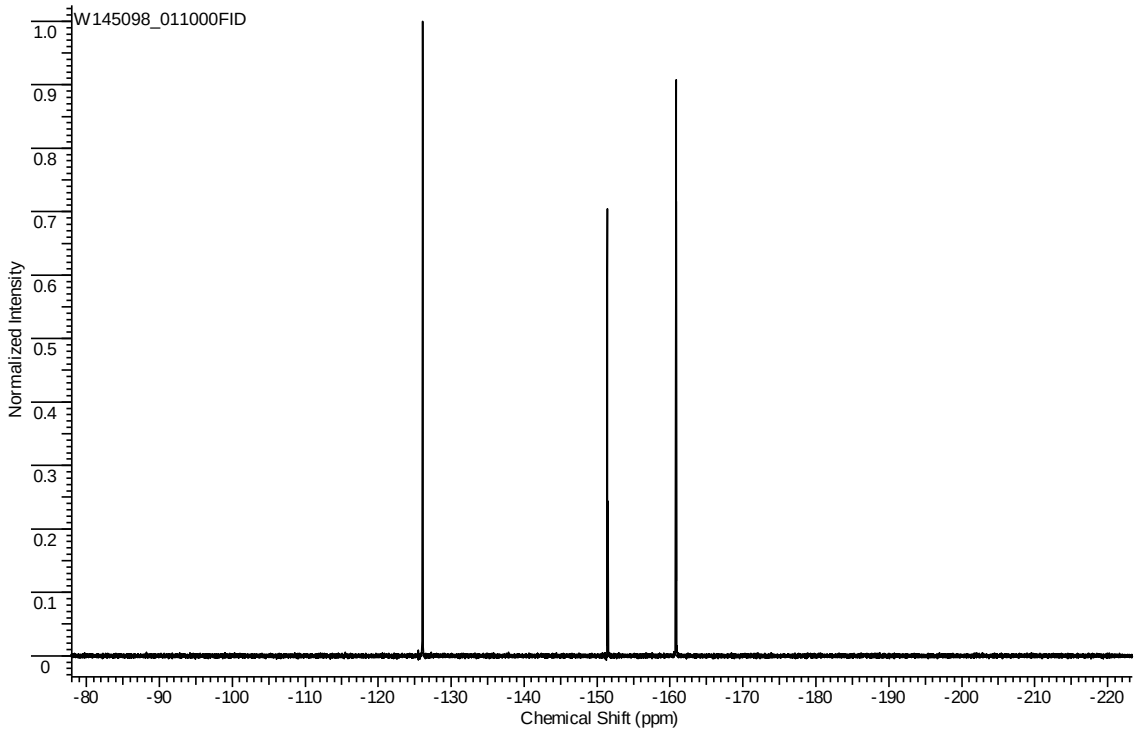
0.1
0
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


W145098_011000FID
Normalized Intensity
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0.7
0.6
0.5
0.4
0.3
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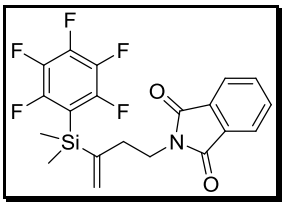
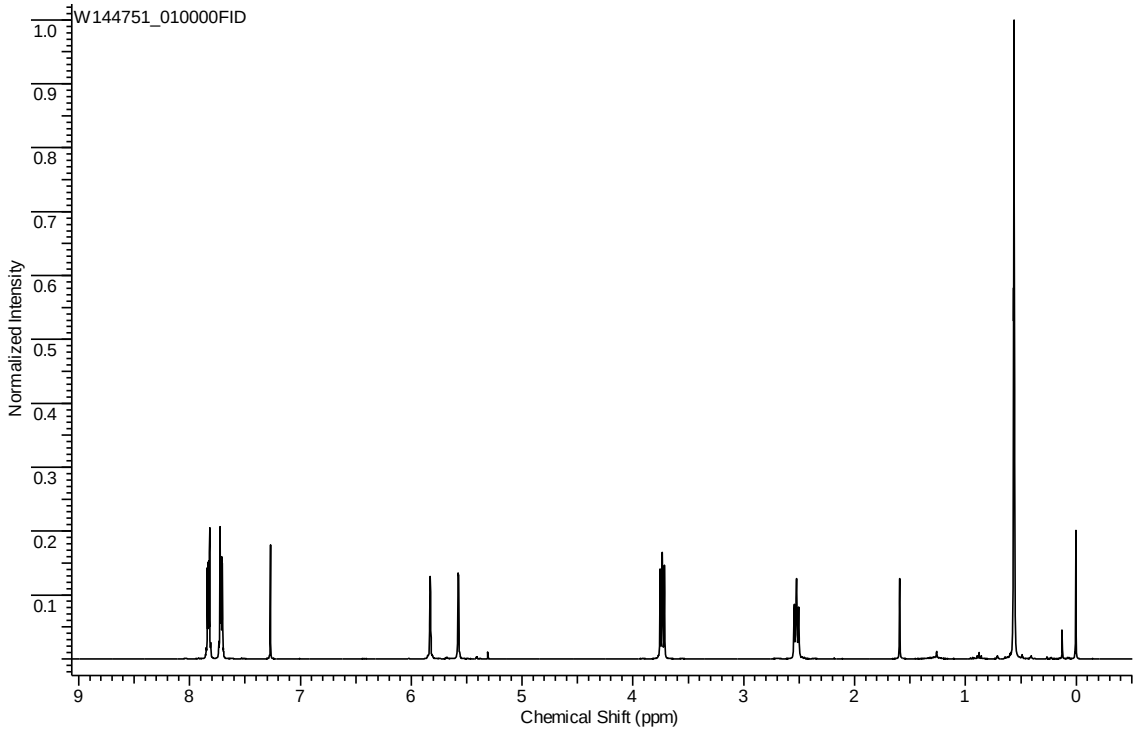
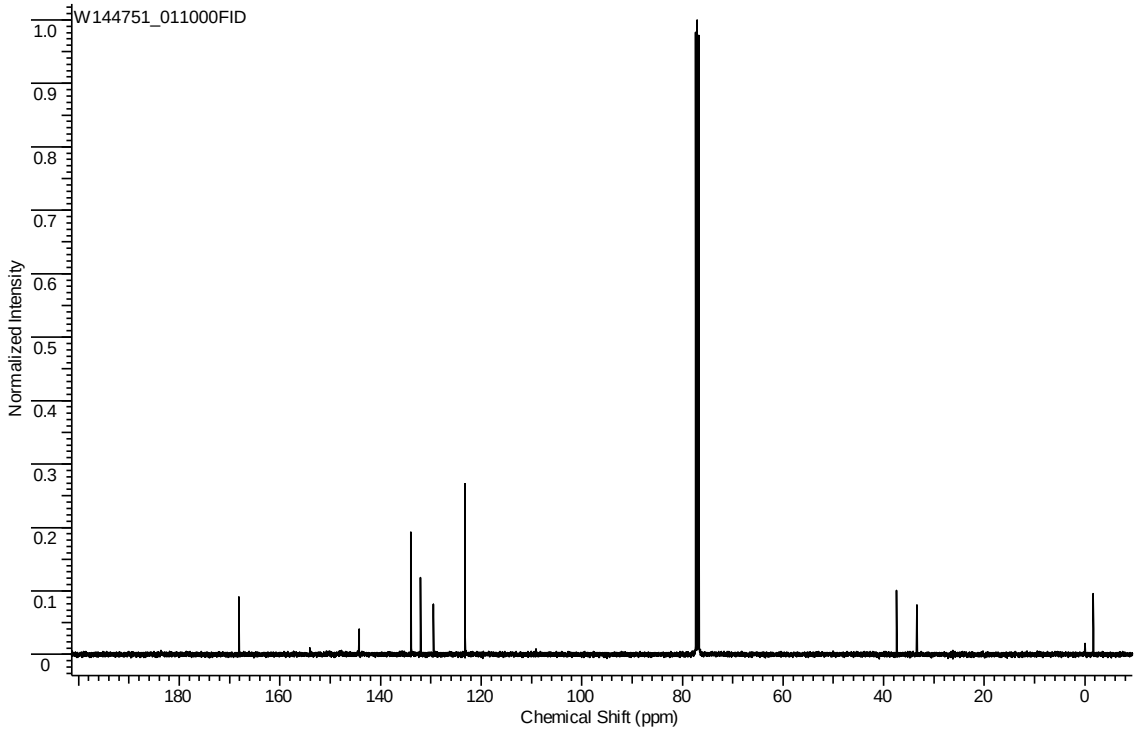
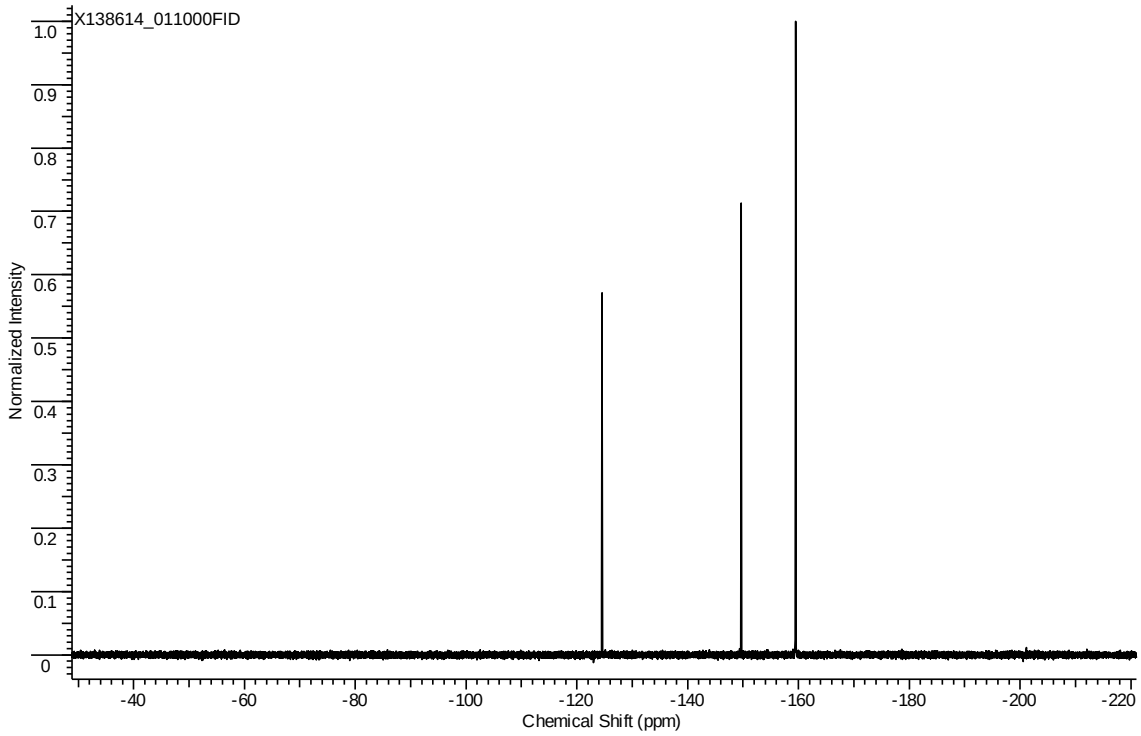
0
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
-220
Chemical Shift (ppm)

2-(3-(Dimethyl(perfluorophenyl)silyl)but-3-enyl)isoindoline-1,3-dione (Table 1, Entry 7)

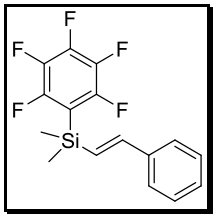


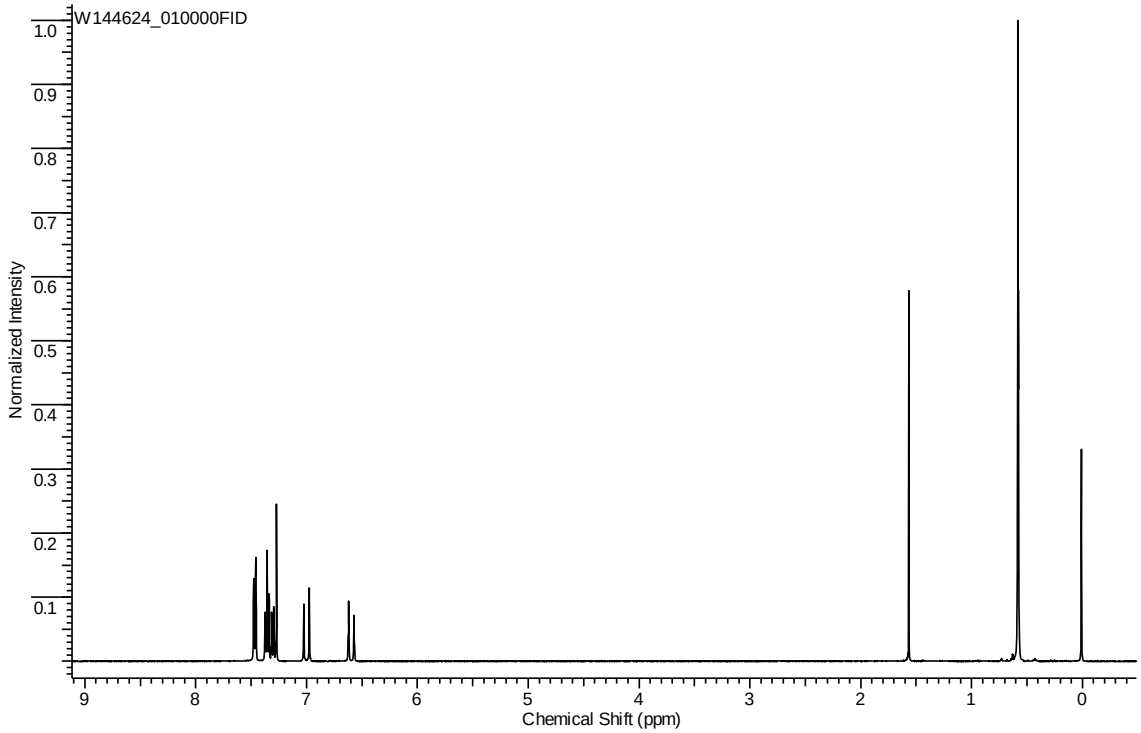


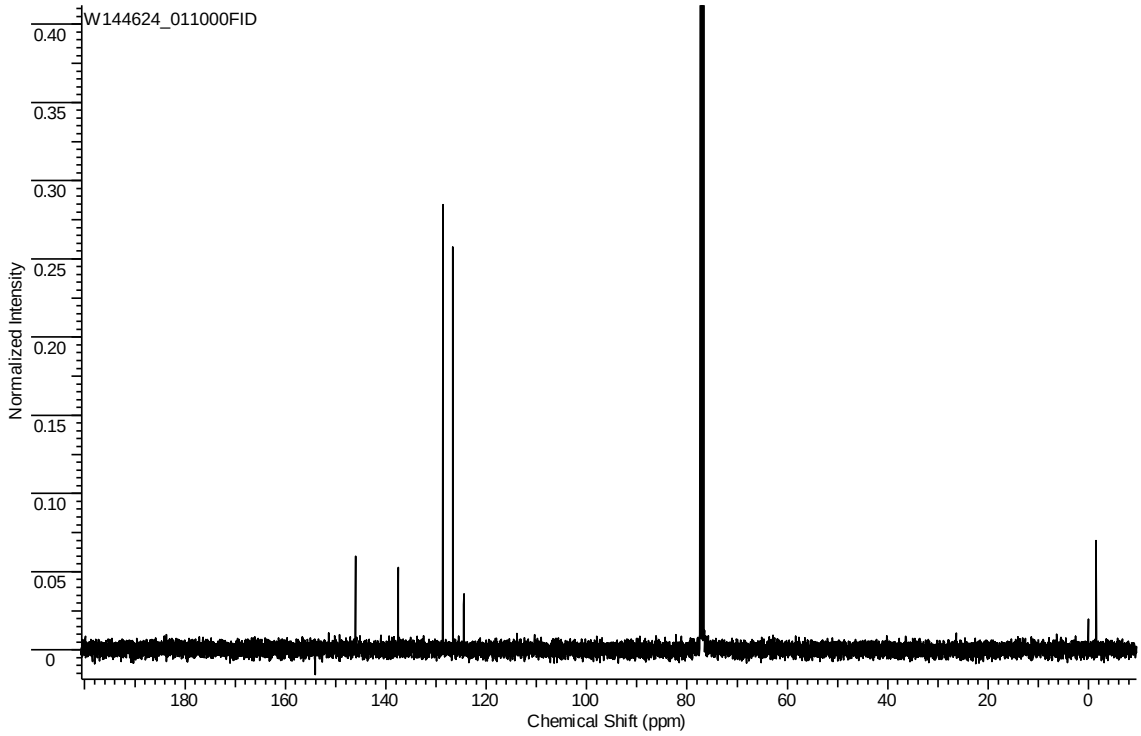




(*E*)-Dimethyl(perfluorophenyl)(styryl)silane (Table 1, Entry 8)






W144624_011000FID
Normalized Intensity
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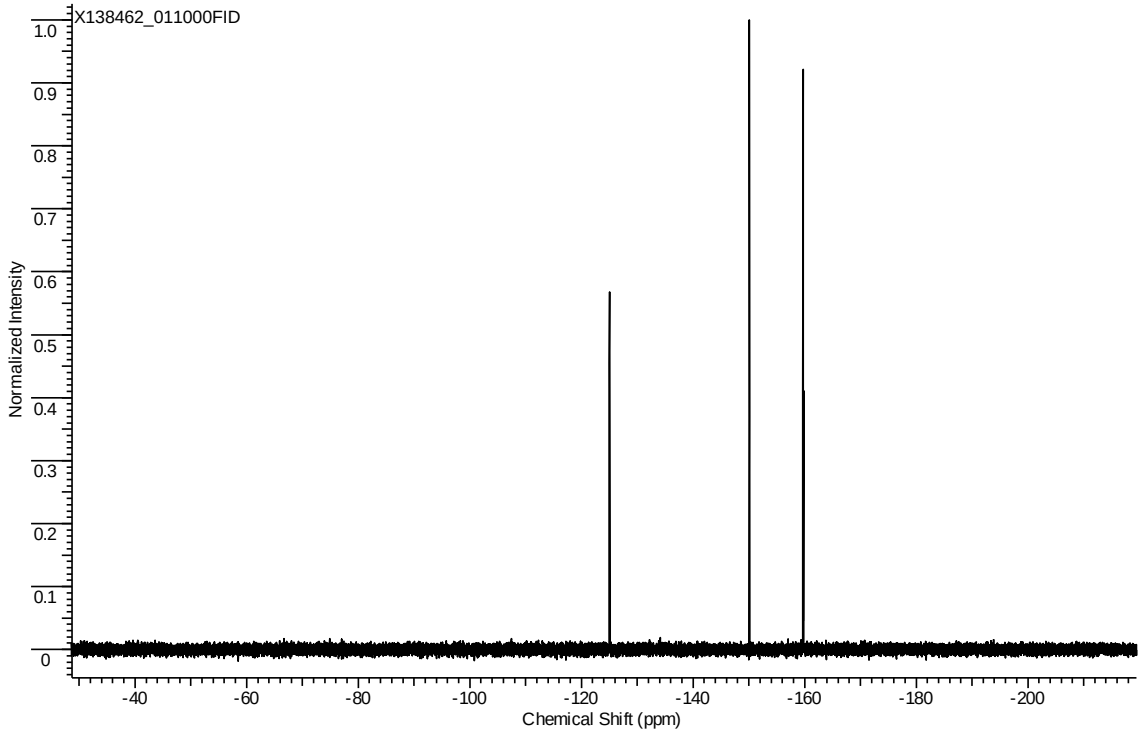
0.15
0.10
0.05
0
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


X138462_011000FID
Normalized Intensity
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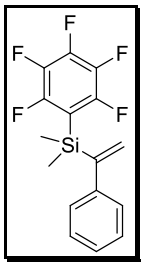
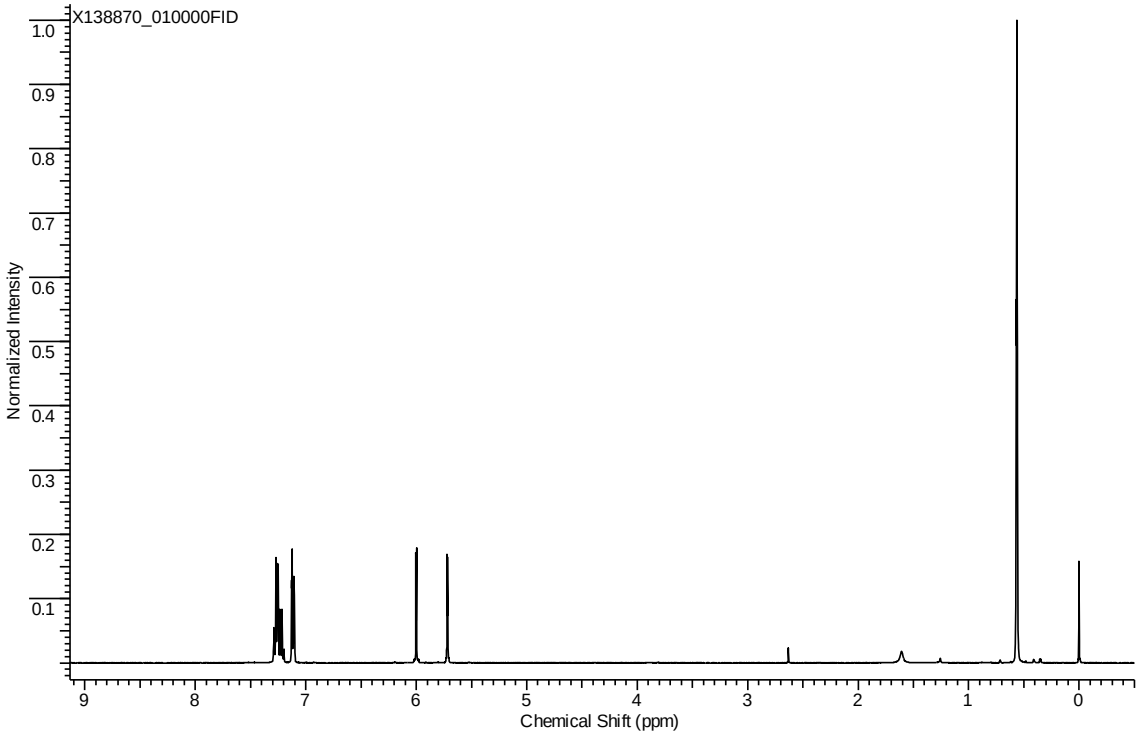
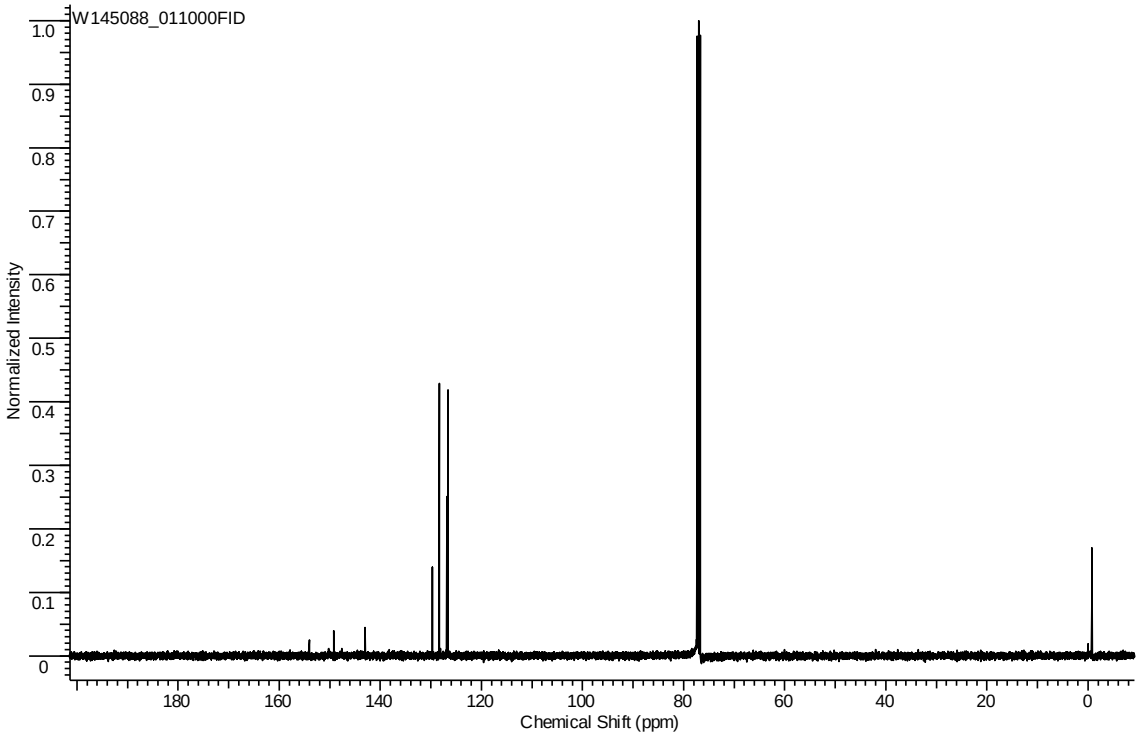
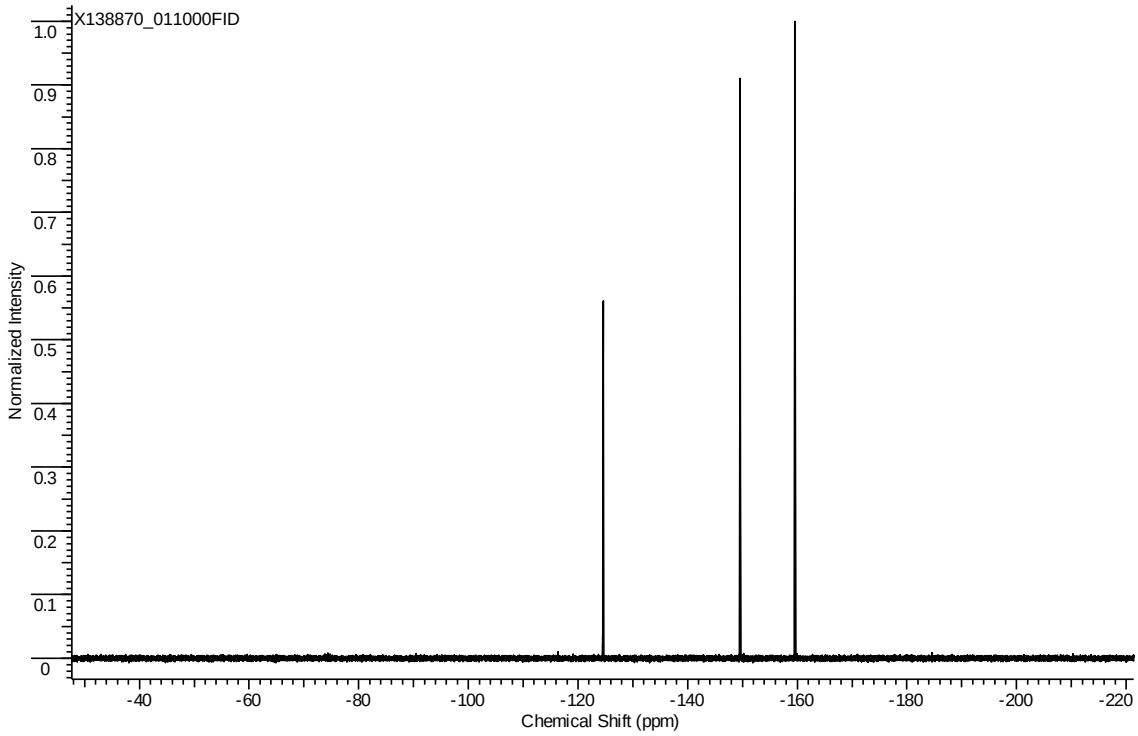
0.4
0.3
0.2
0.1
0
-40
-60
-80
-100
-120
-140
-160
-180
-200
Chemical Shift (ppm)

Dimethyl(perfluorophenyl)(1-phenylvinyl)silane (Table 1, Entry 9)

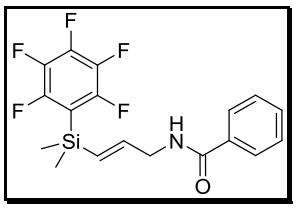


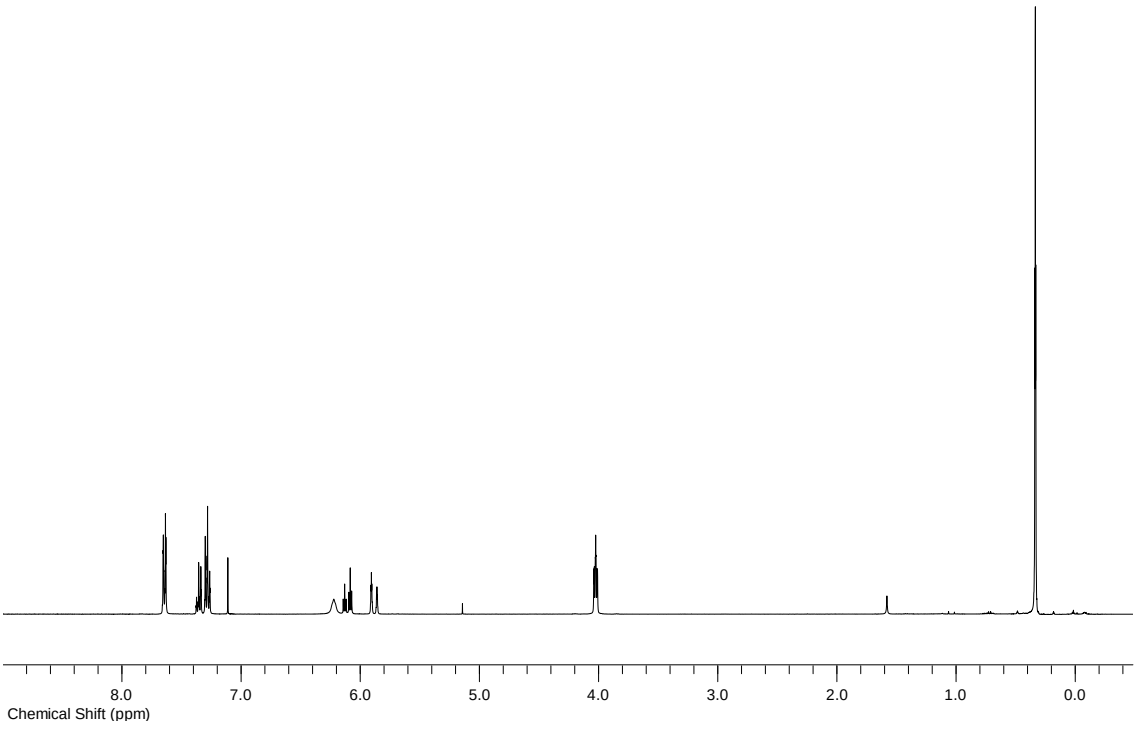


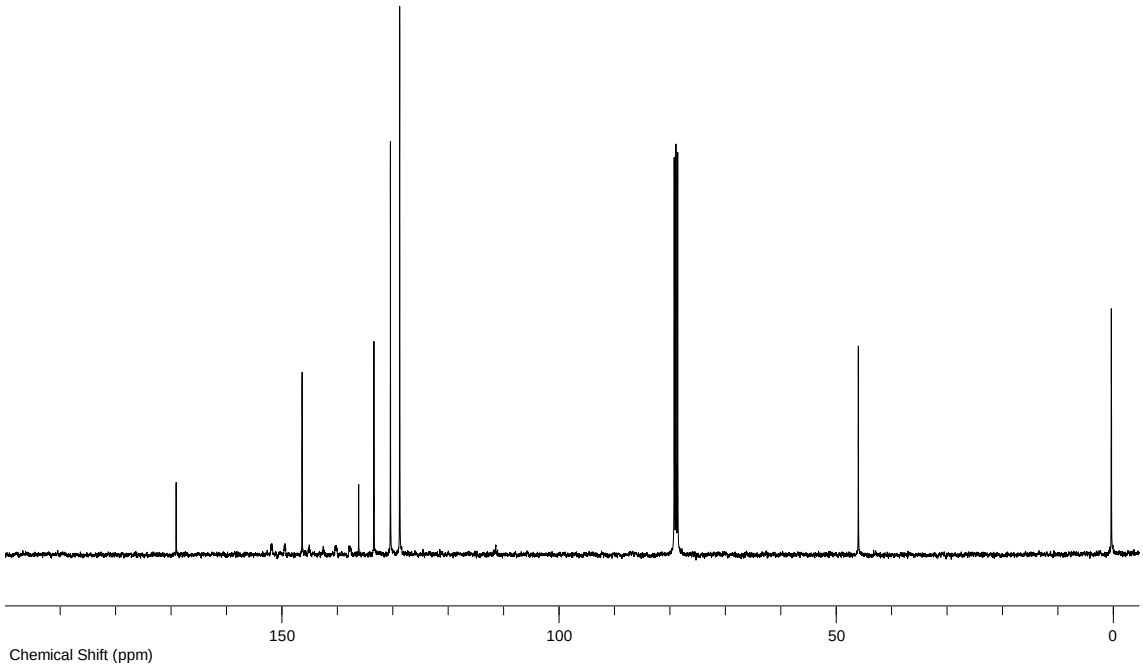


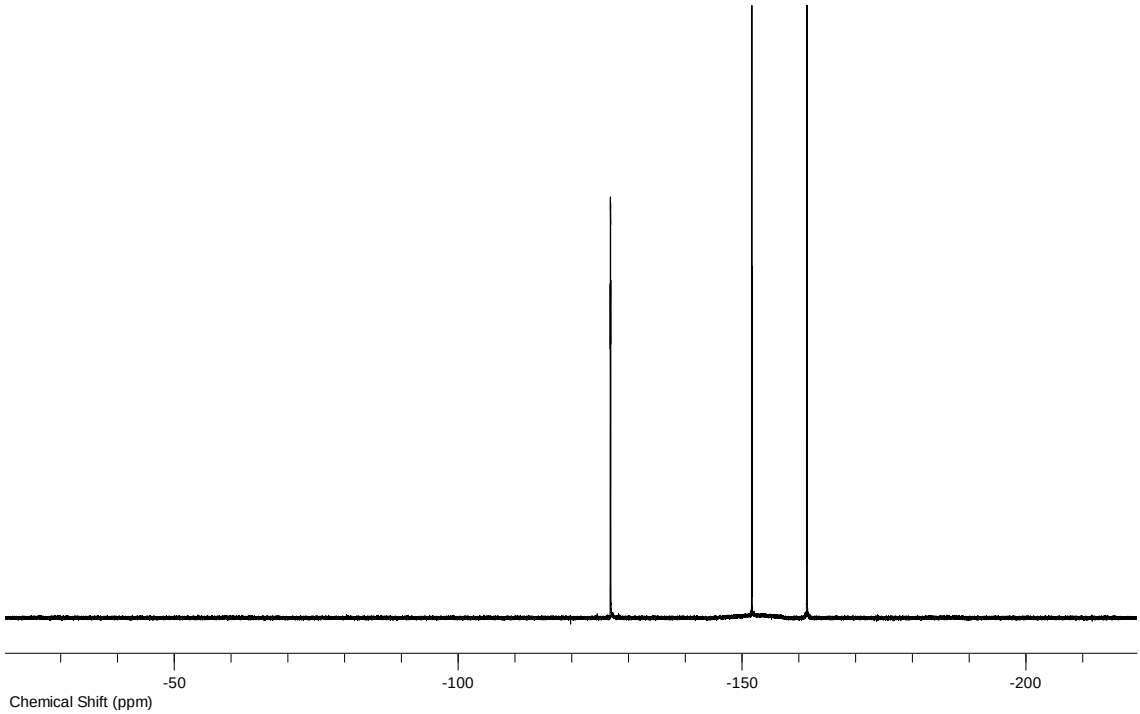


(*E*)-*N*-(3-(Dimethyl(perfluorophenyl)silyl)allyl)benzamide (Table 1, Entry 10)

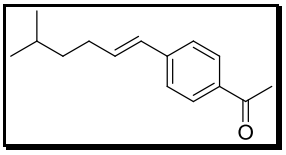


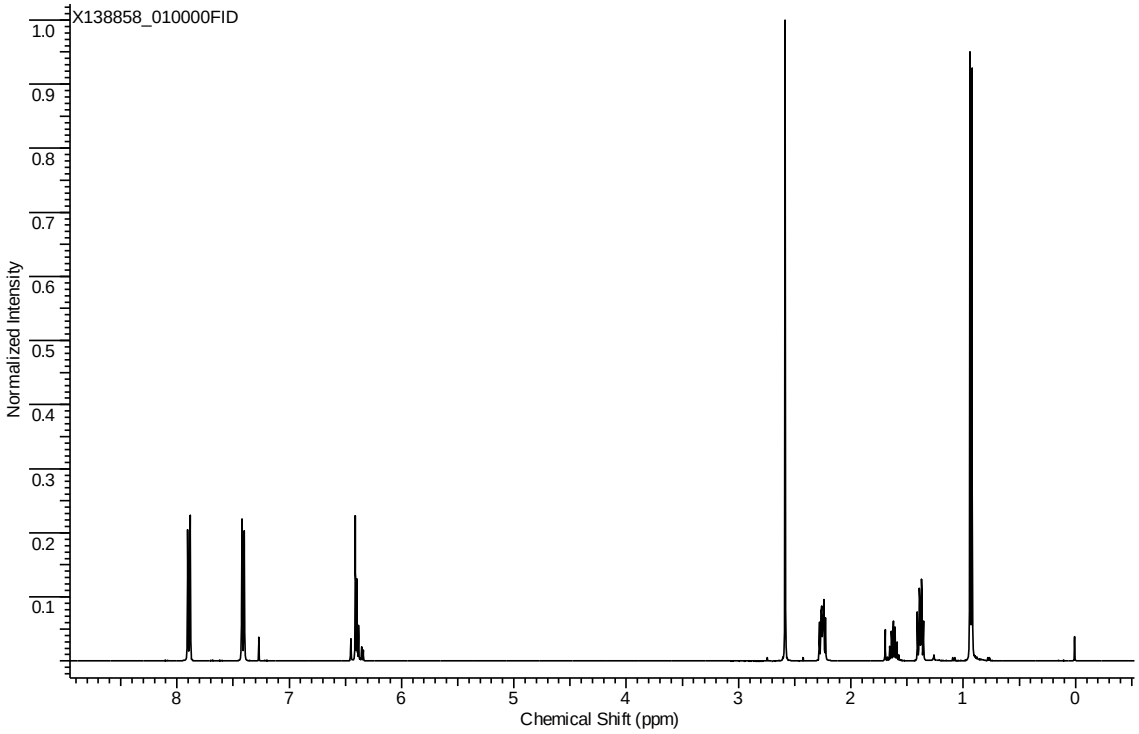


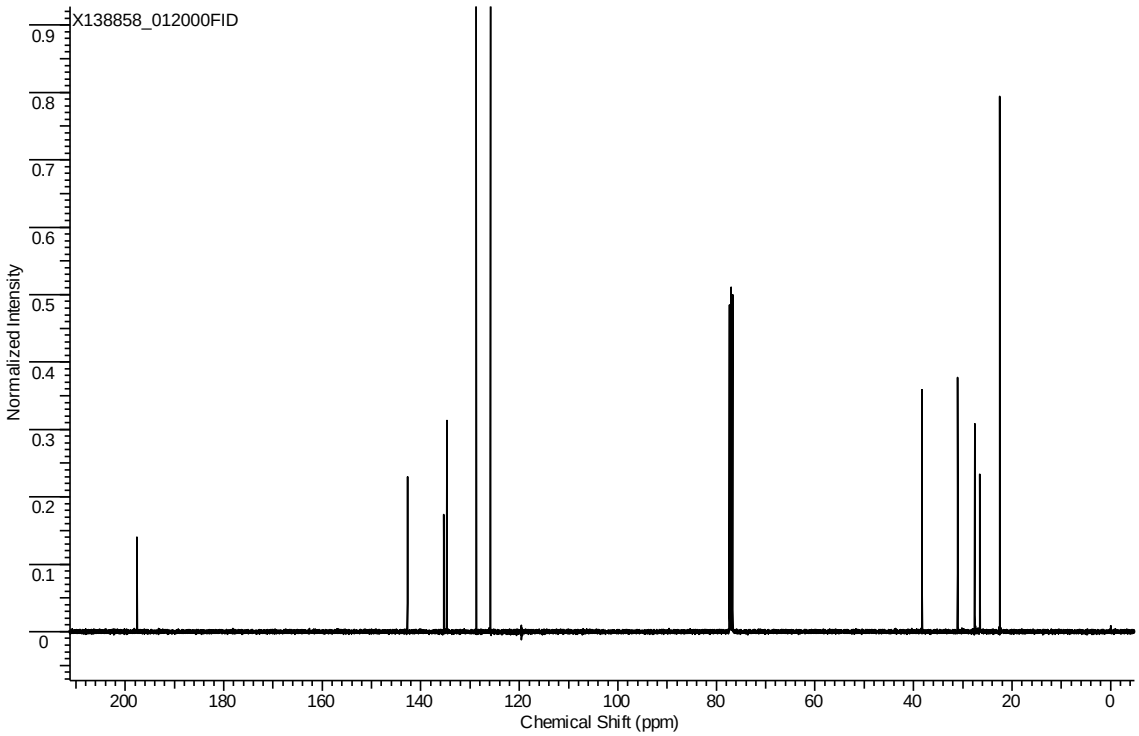

150
100
50
0
Chemical Shift (ppm)


-50
-100
-150
-200
Chemical Shift (ppm)

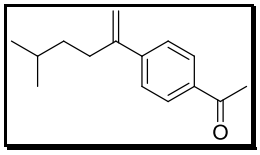
(*E*)-1-(4-(5-Methylhex-1-enyl)phenyl)ethanone (Table 2, Entry 1)

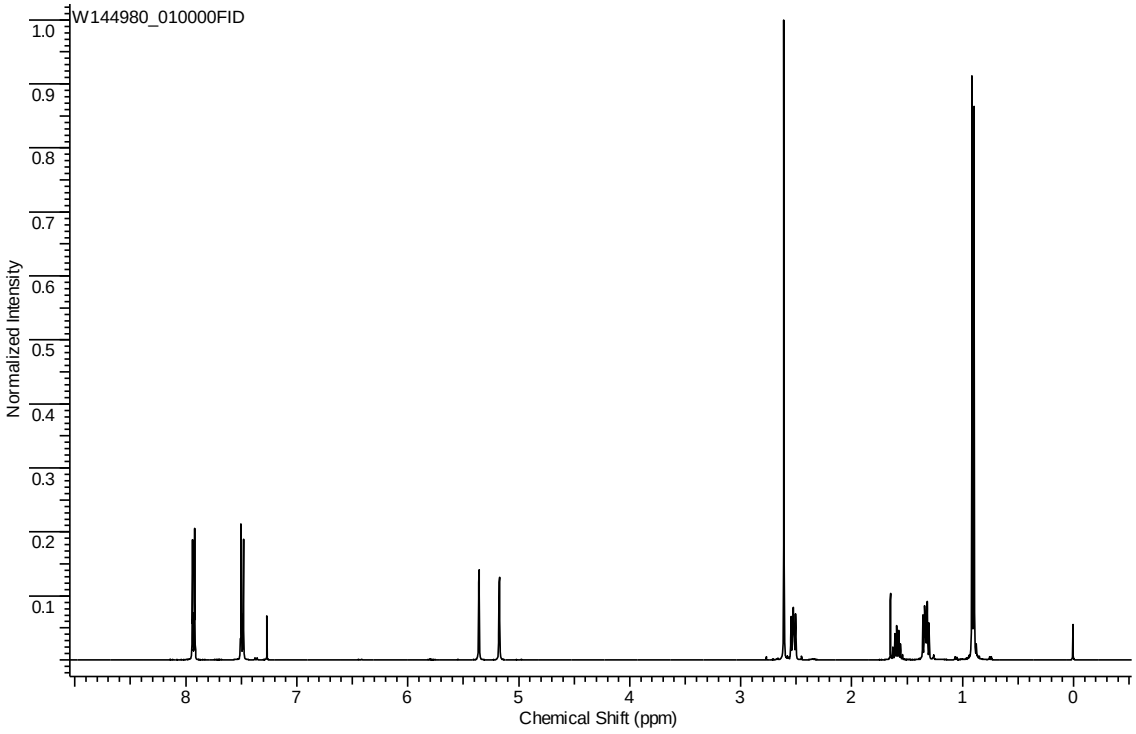


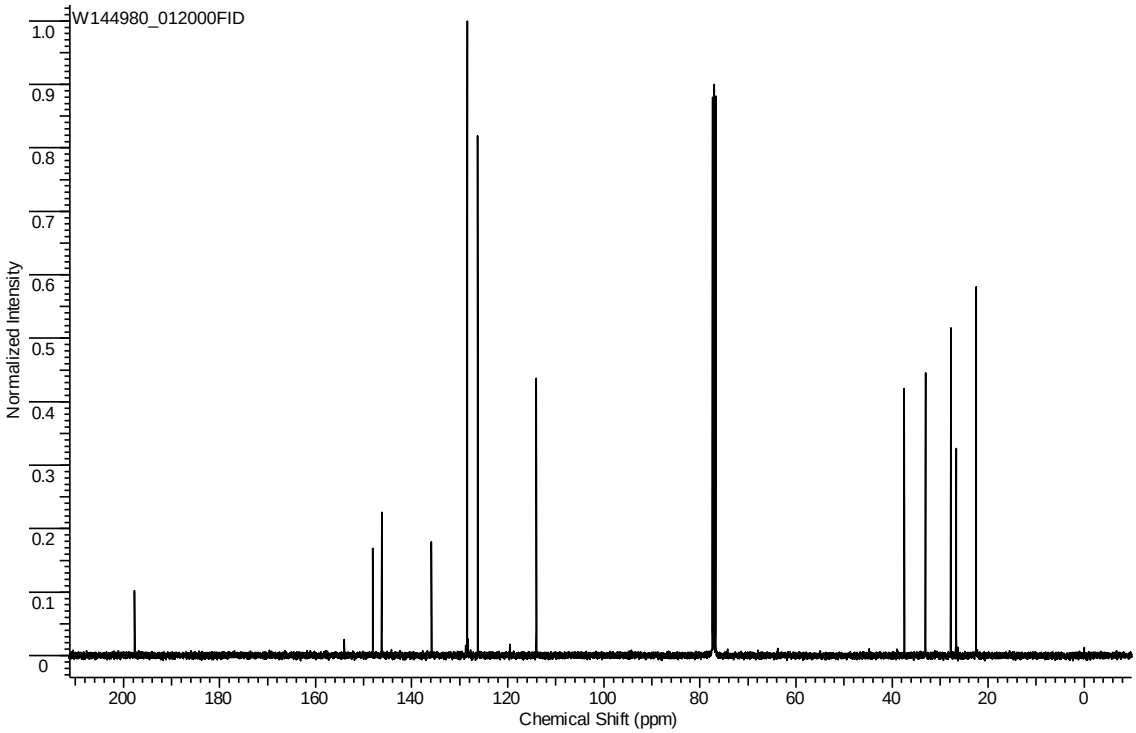




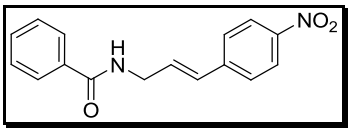
1-(4-(5-Methylhex-1-en-2-yl)phenyl)ethanone (Table 2, Entry 2)

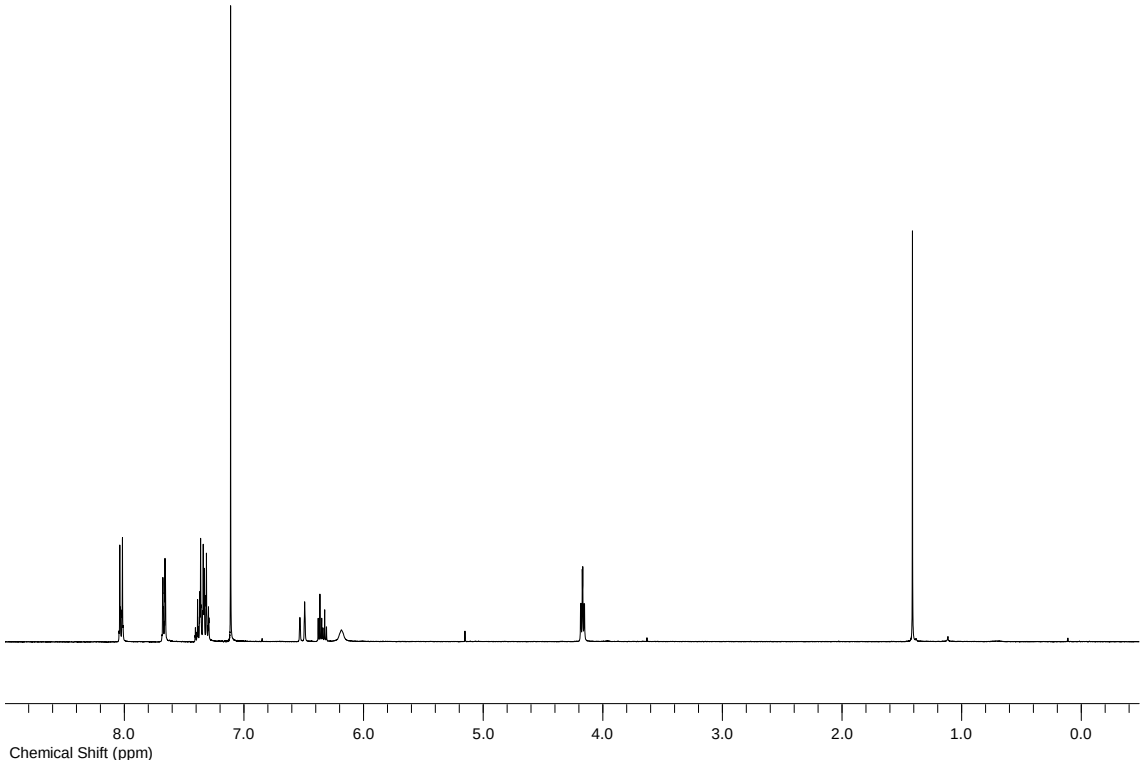


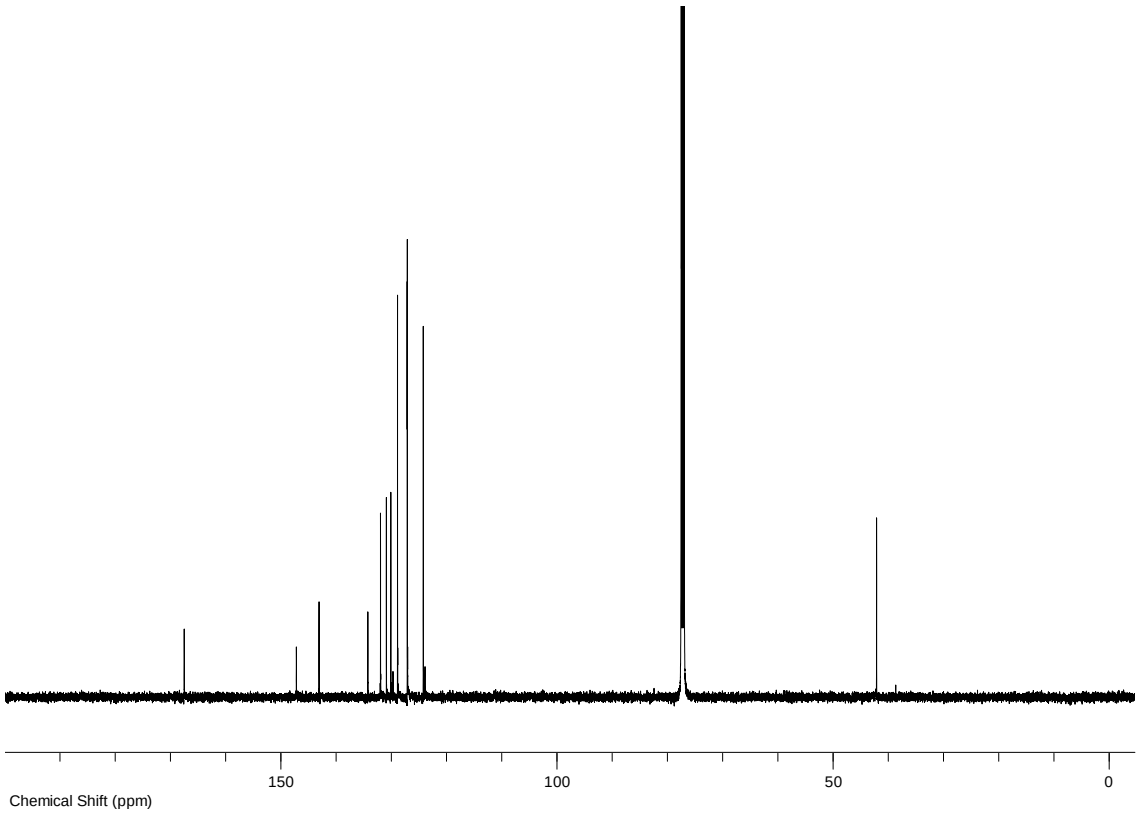




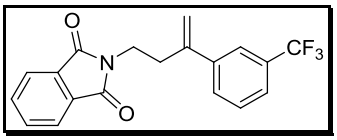
(*E*)-*N*-(3-(4-Nitrophenyl)allyl)benzamide (Table 2, Entry 3)

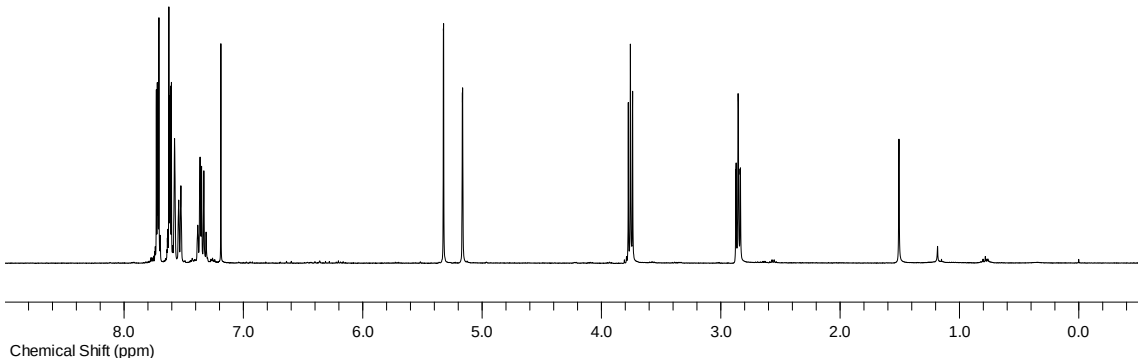


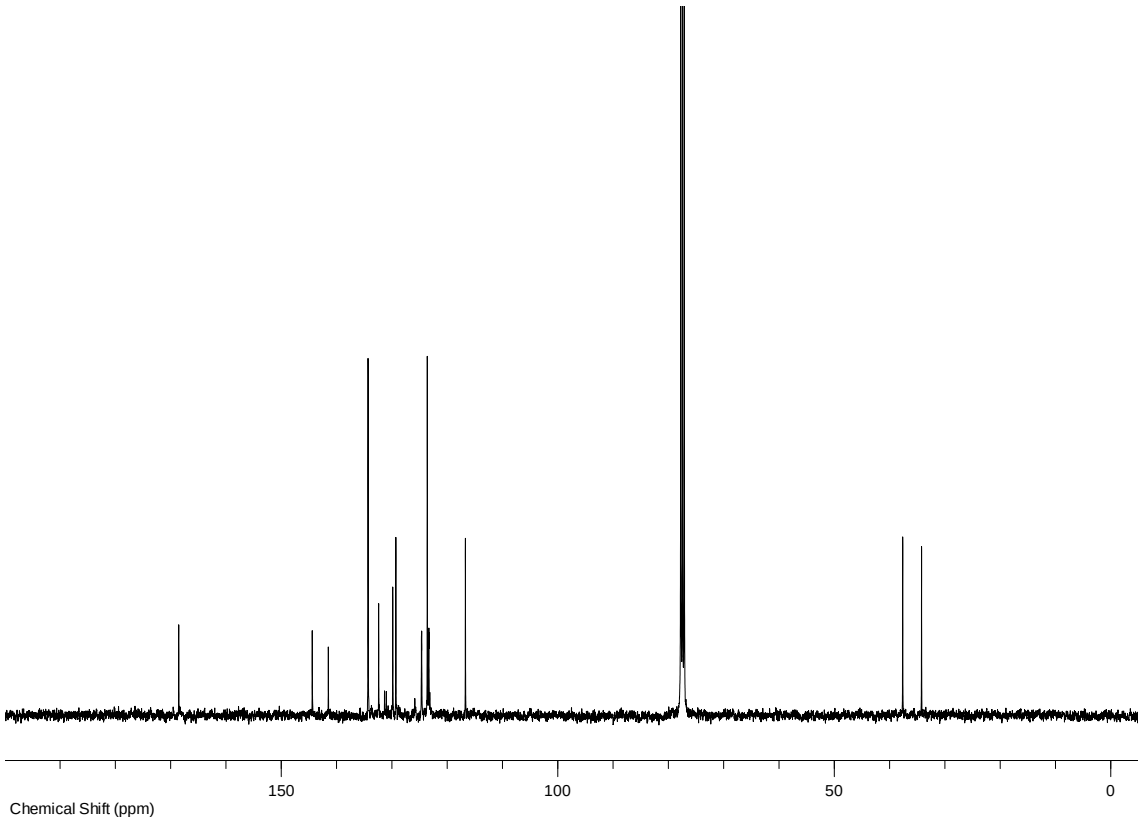




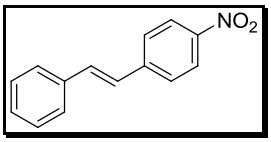
2-(3-(3-(Trifluoromethyl)phenyl)but-3-enyl)isoindoline-1,3-dione (Table 2, Entry 4)

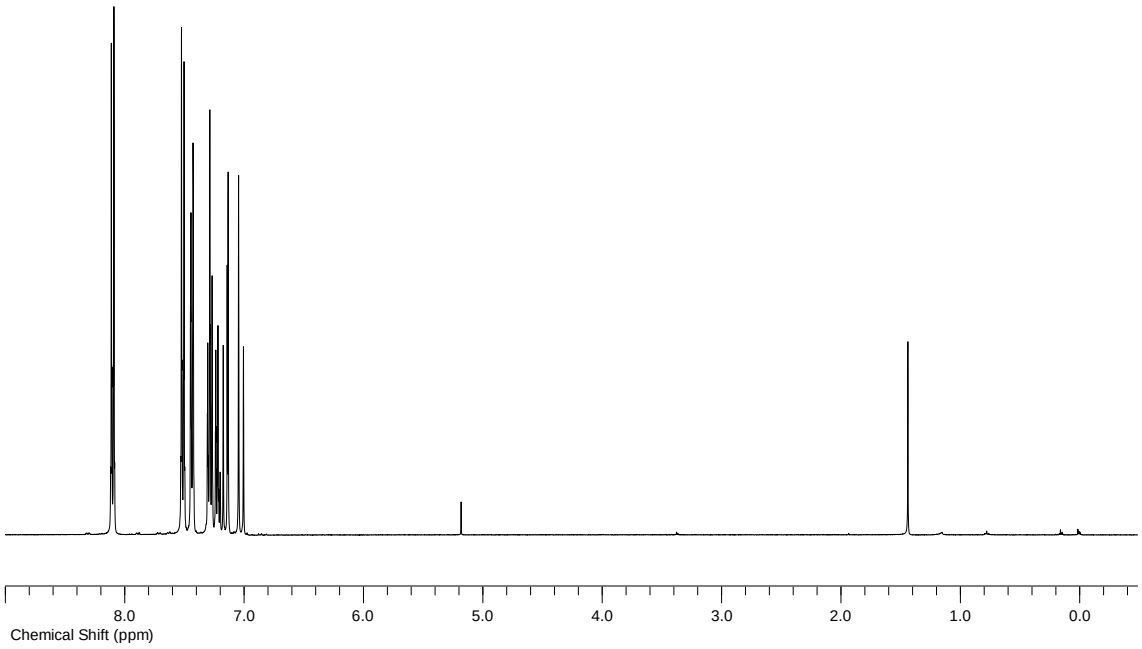


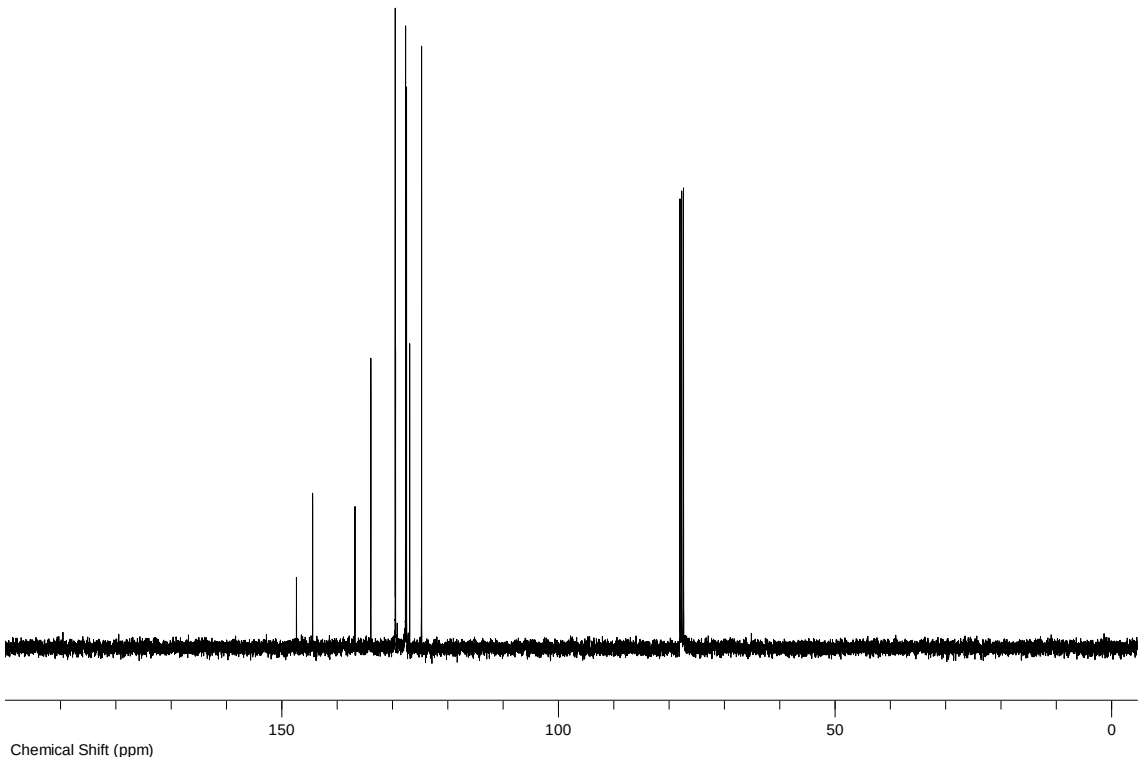




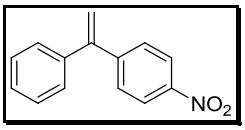
(*E*)-1-Nitro-4-styrylbenzene (Table 2, Entry 5)

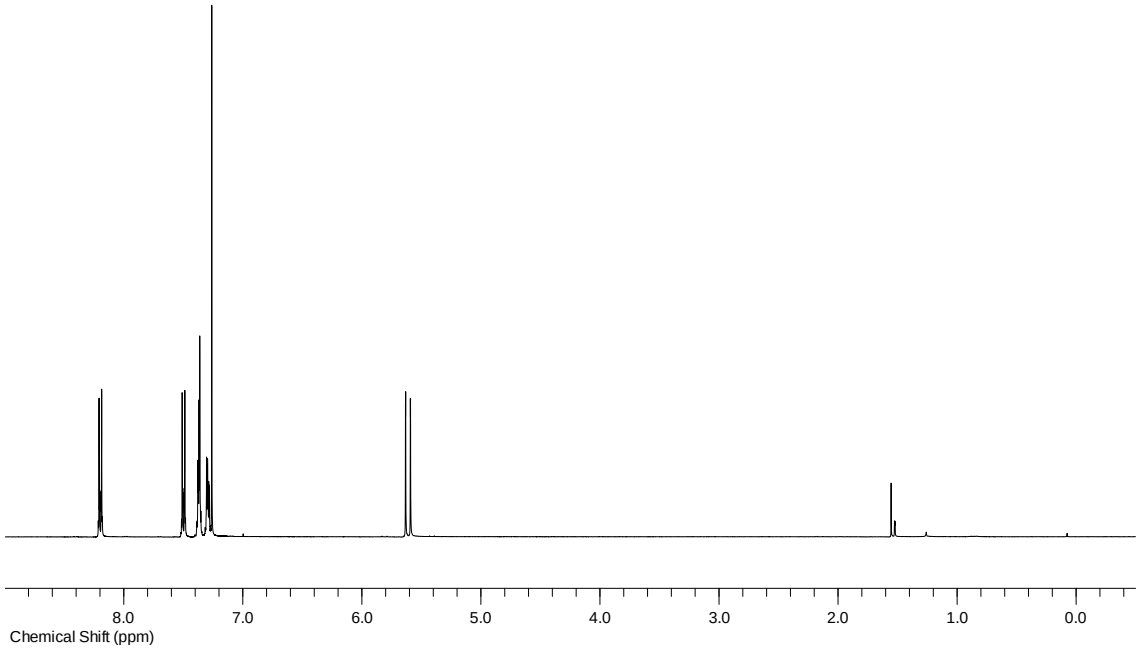


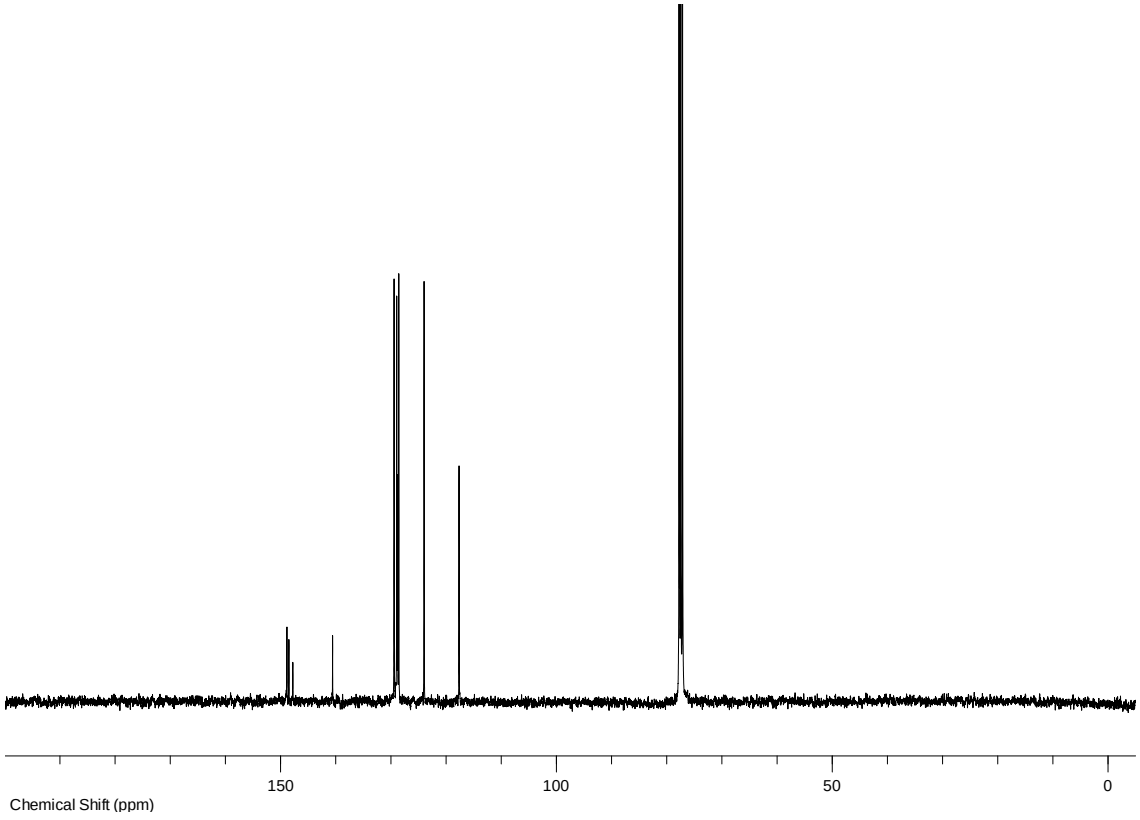




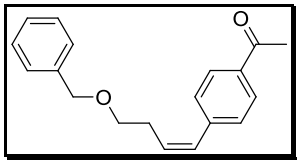
1-Nitro-4-(1-phenylvinyl)benzene (Table 2, Entry 6)

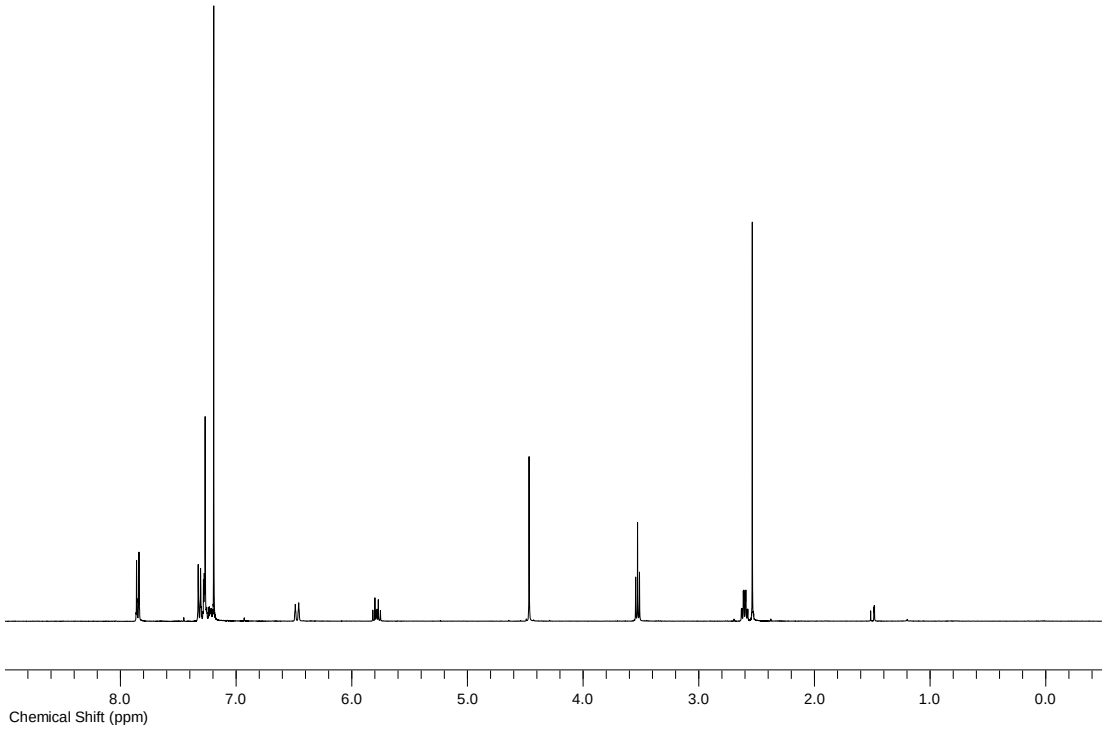


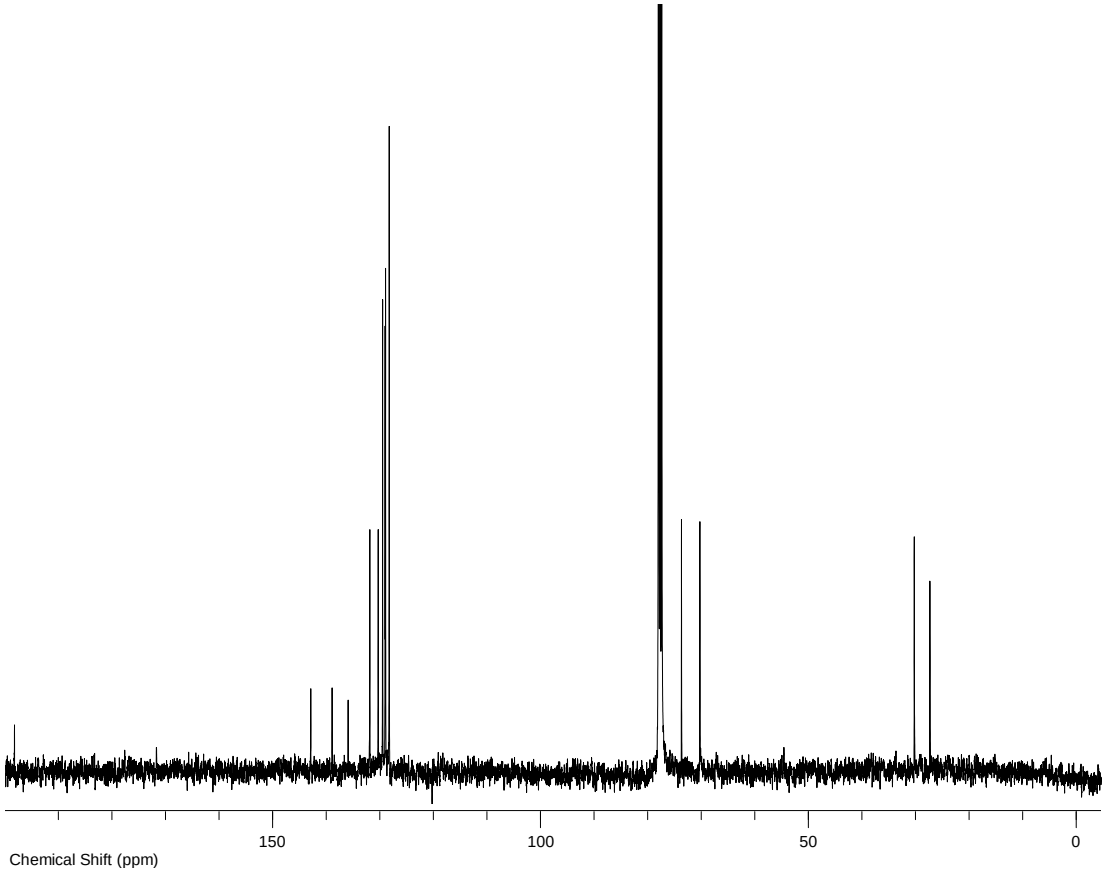




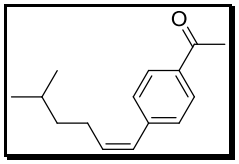
(*Z*)-1-(4-(4-(Benzyloxy)but-1-enyl)phenyl)ethanone (Table 3, Entry 1)

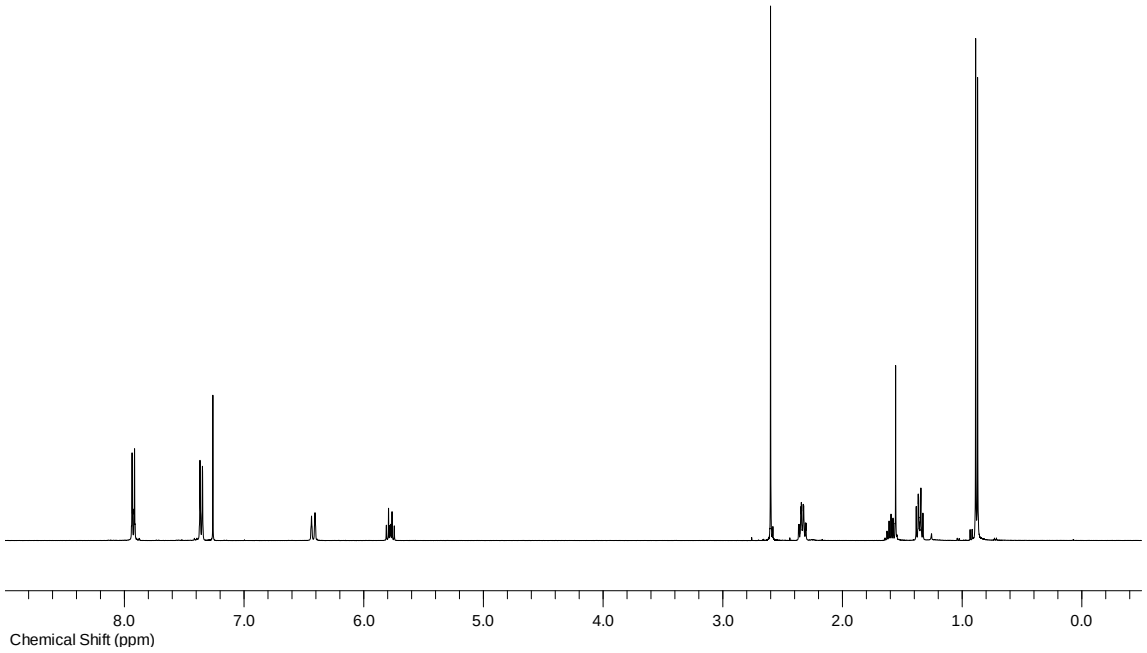


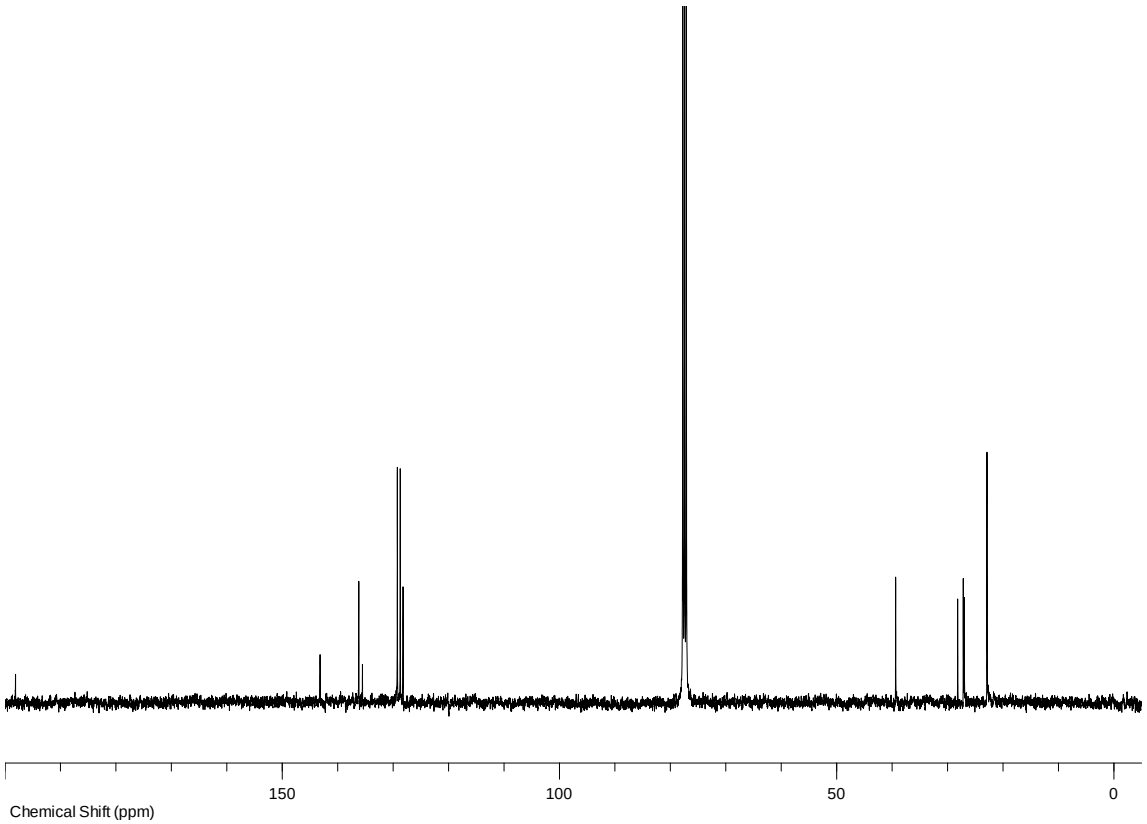




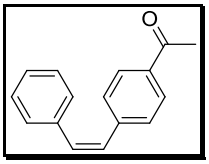
(*Z*)-1-(4-(5-Methylhex-1-enyl)phenyl)ethanone (Table 3, Entry 2)

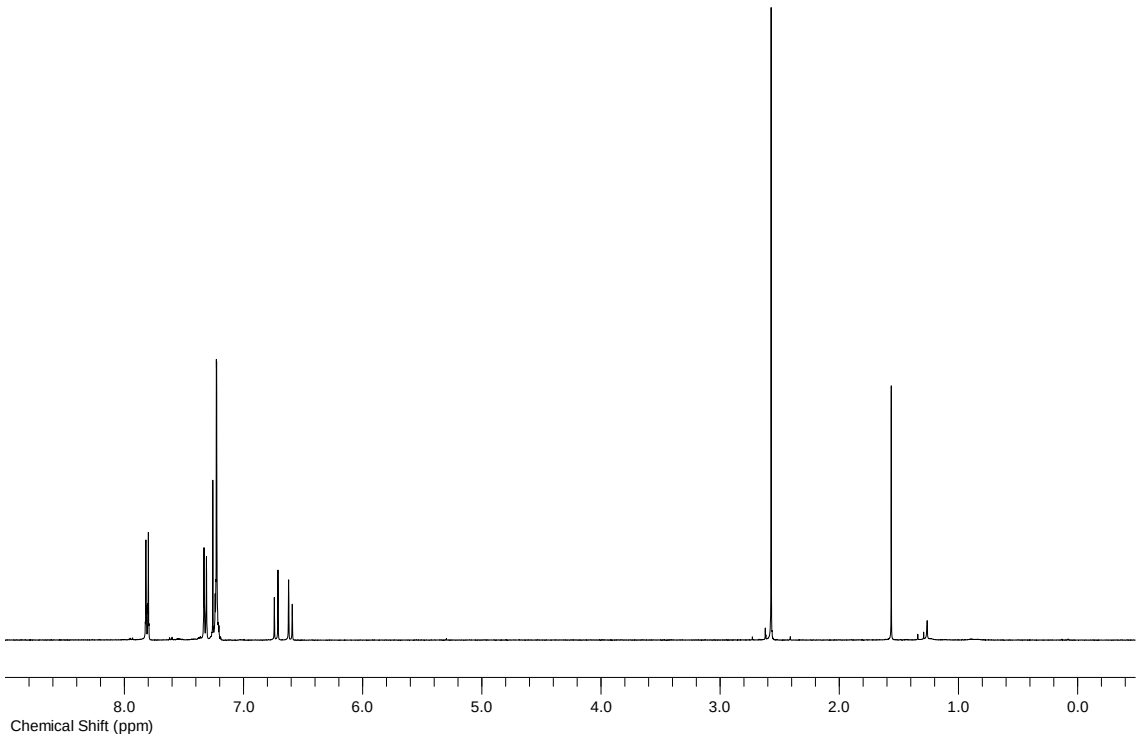


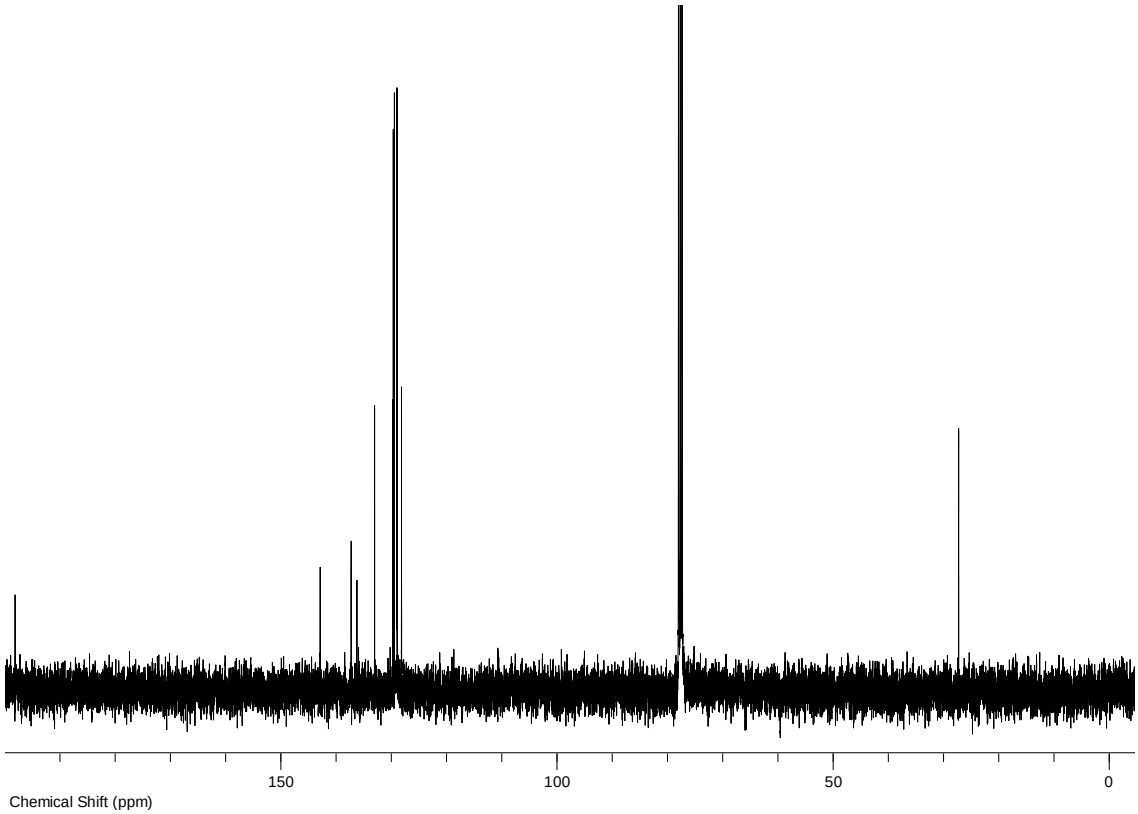




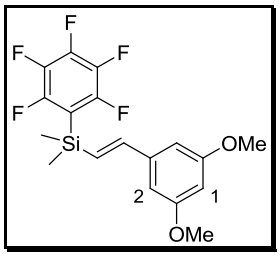
(*Z*)-1-(4-styrylphenyl)ethanone (Table 3, Entry 3)

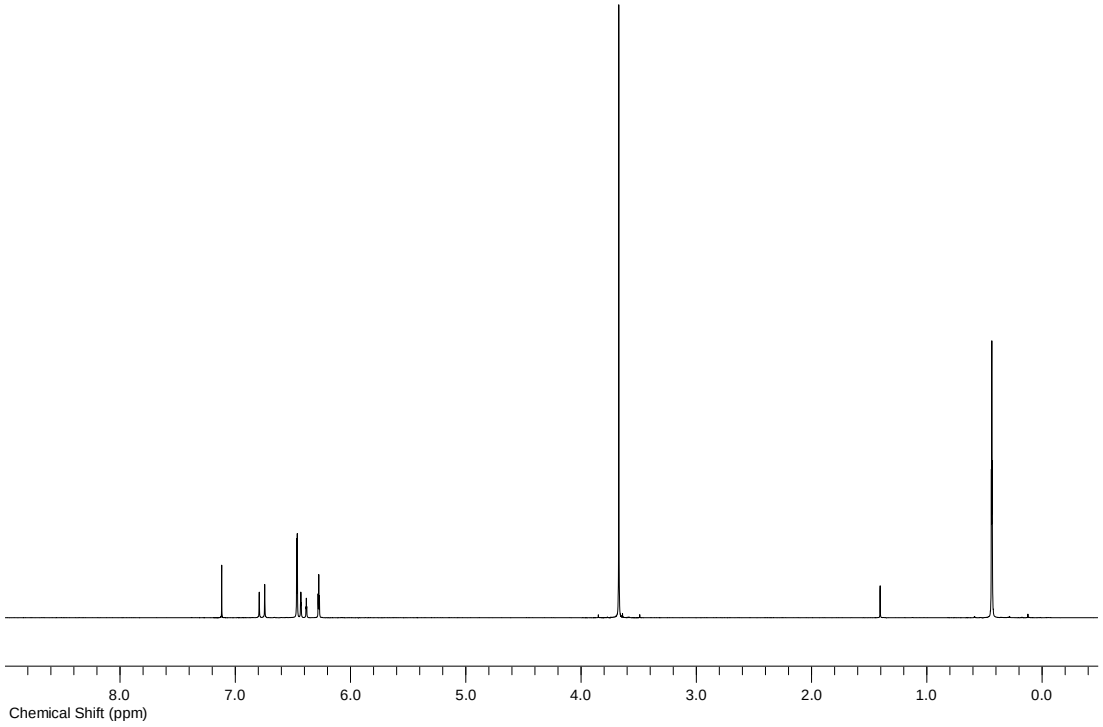


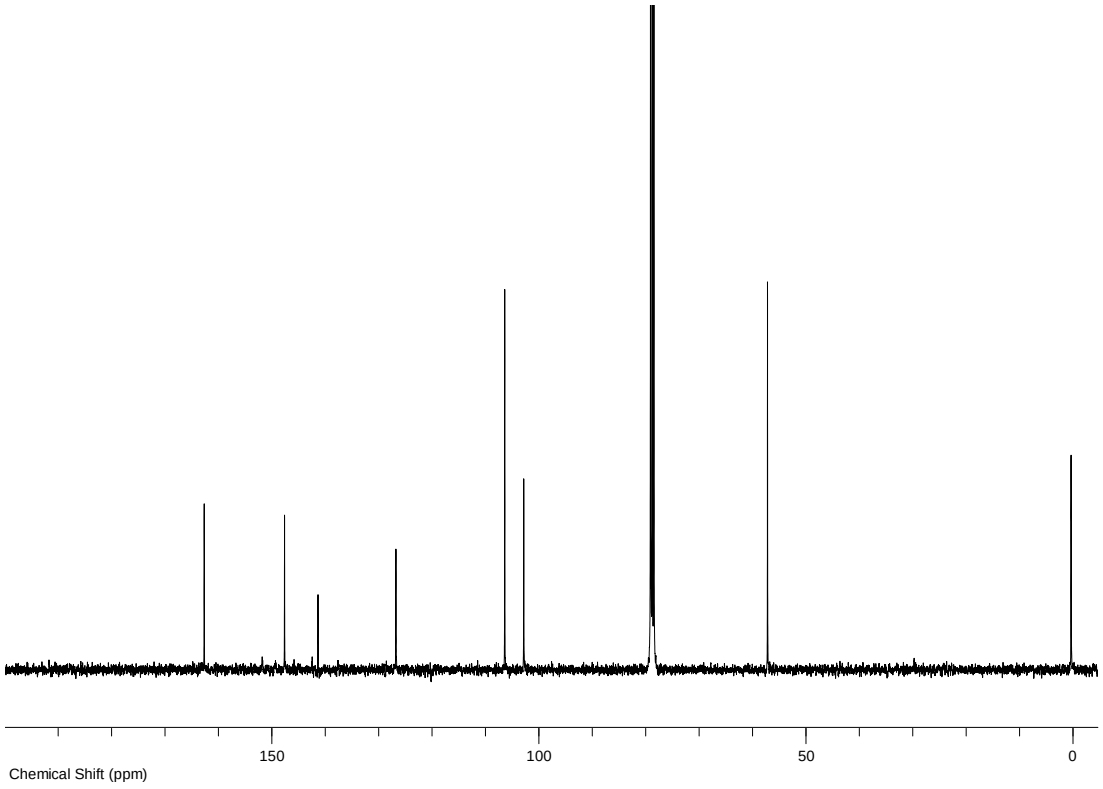


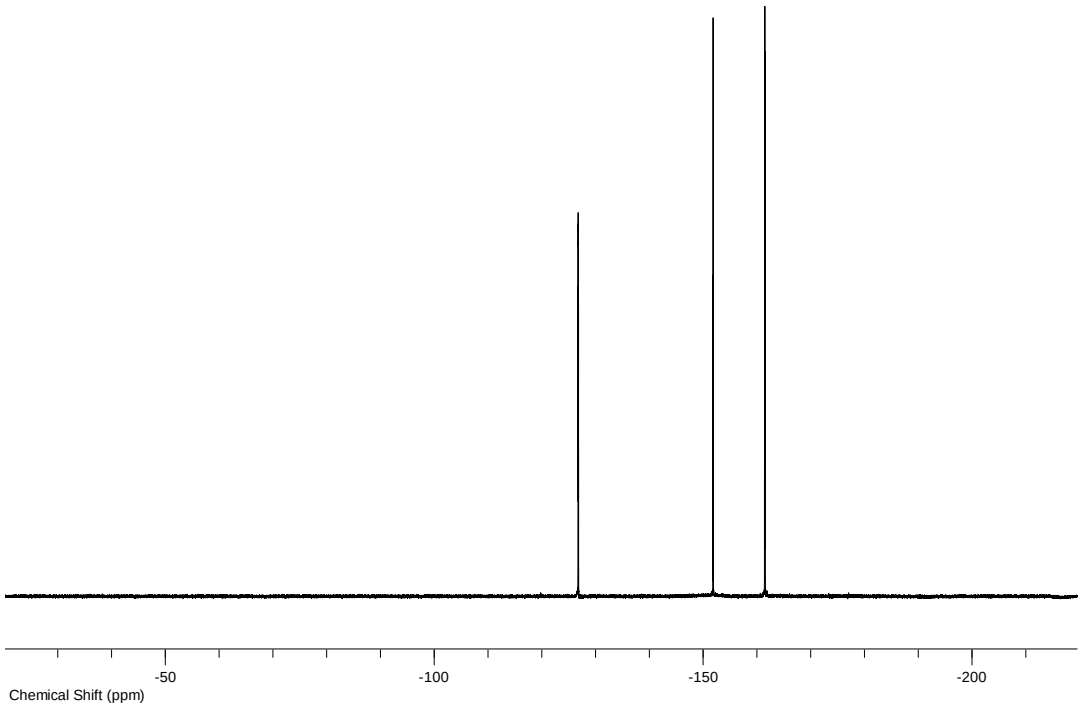


(*E*)-(3,5-dimethoxystyryl)dimethyl(perfluorophenyl)silane (Scheme 1, 8-(*E*))

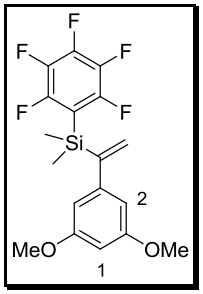


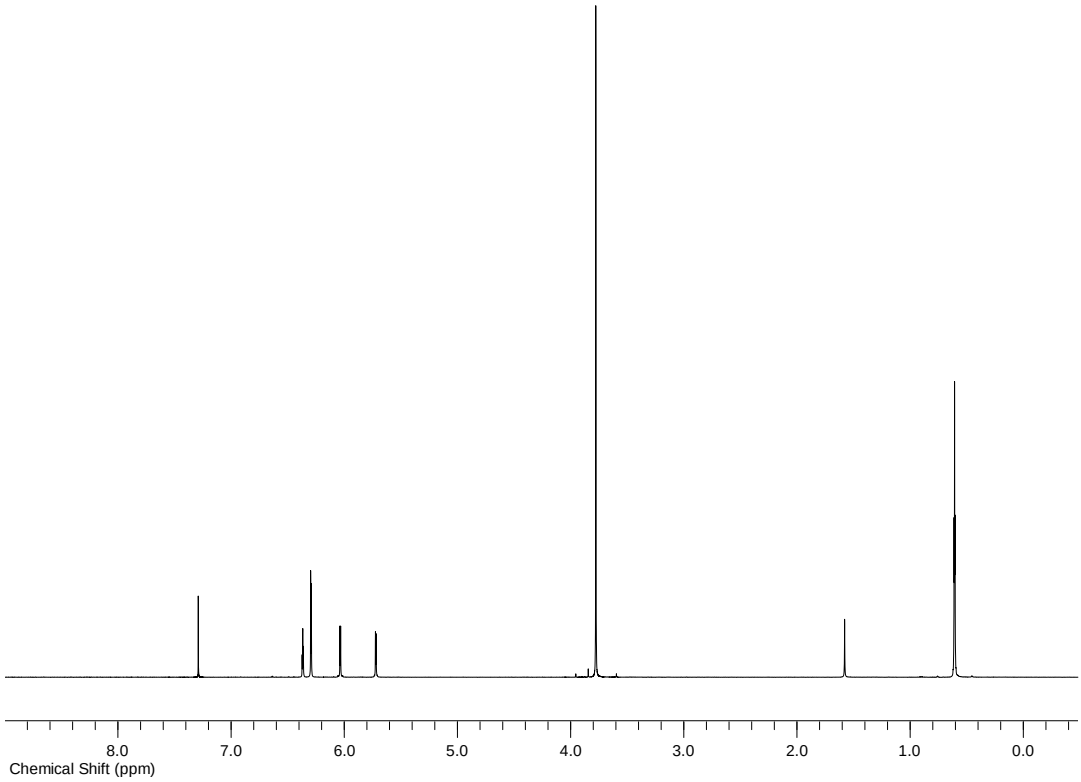


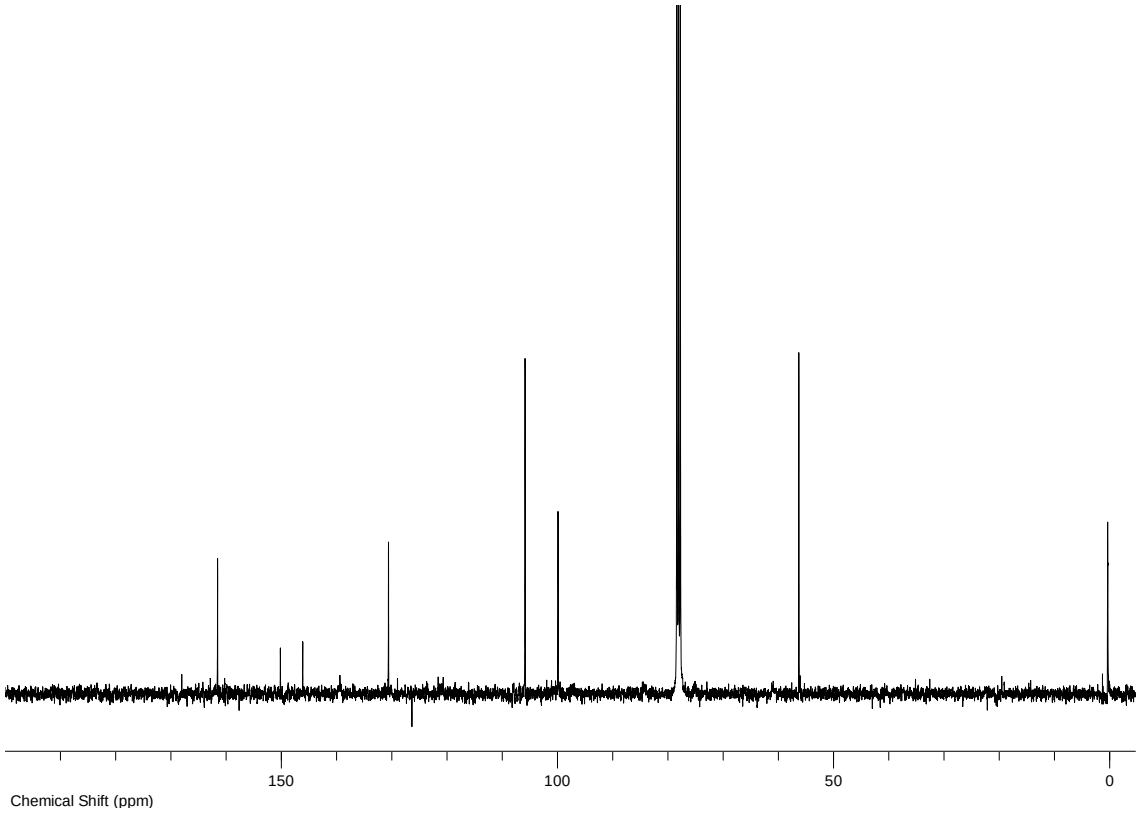


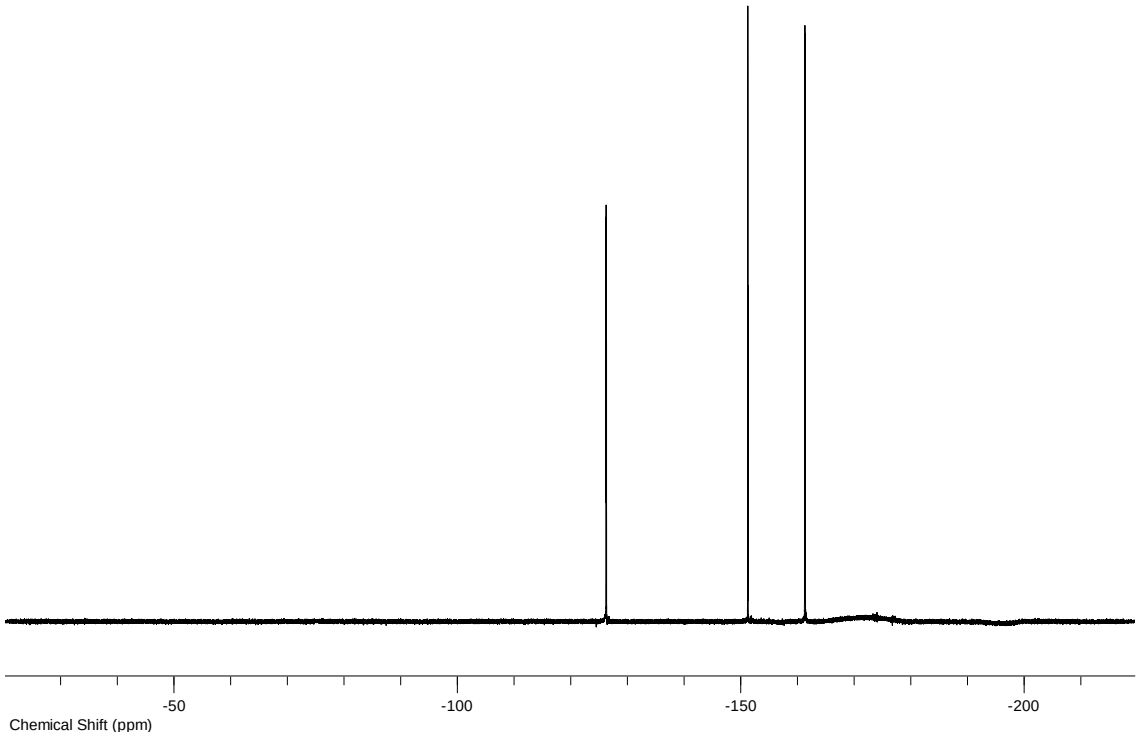


(1-(3,5-Dimethoxyphenyl)vinyl)dimethyl(perfluorophenyl)silane (Scheme 1, 8-α)

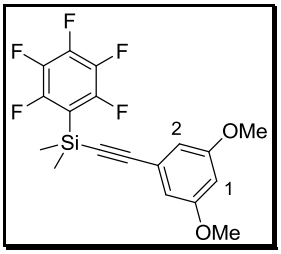


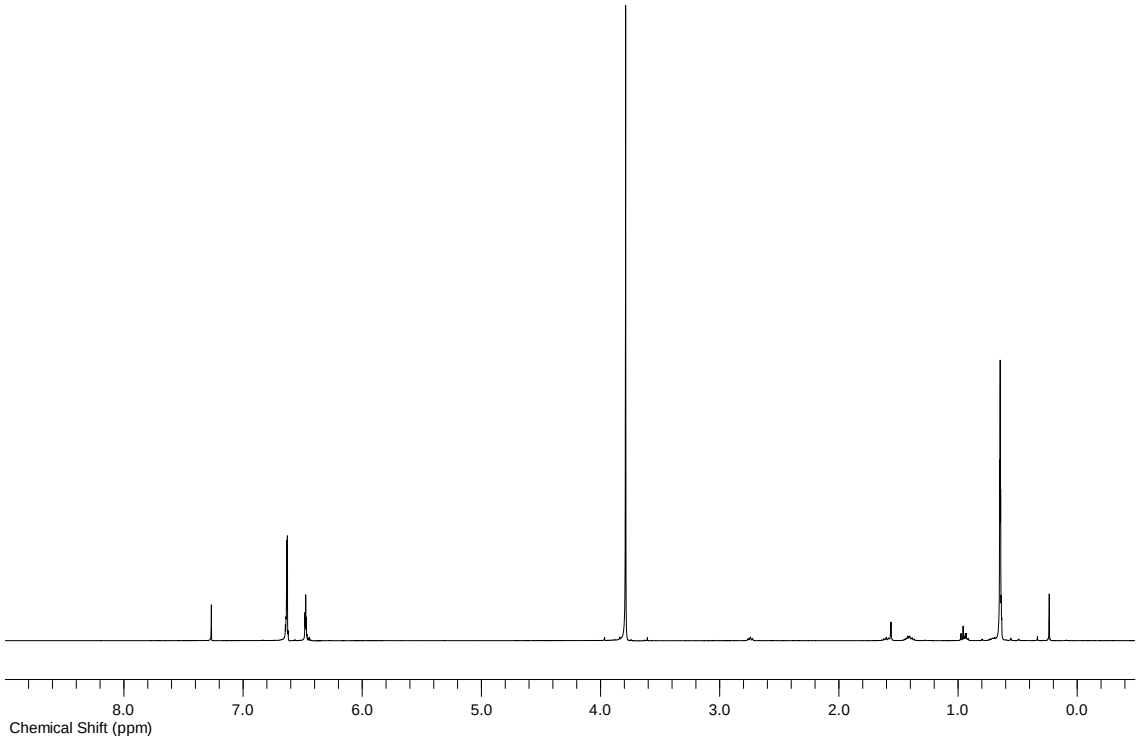

8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
Chemical Shift (ppm)

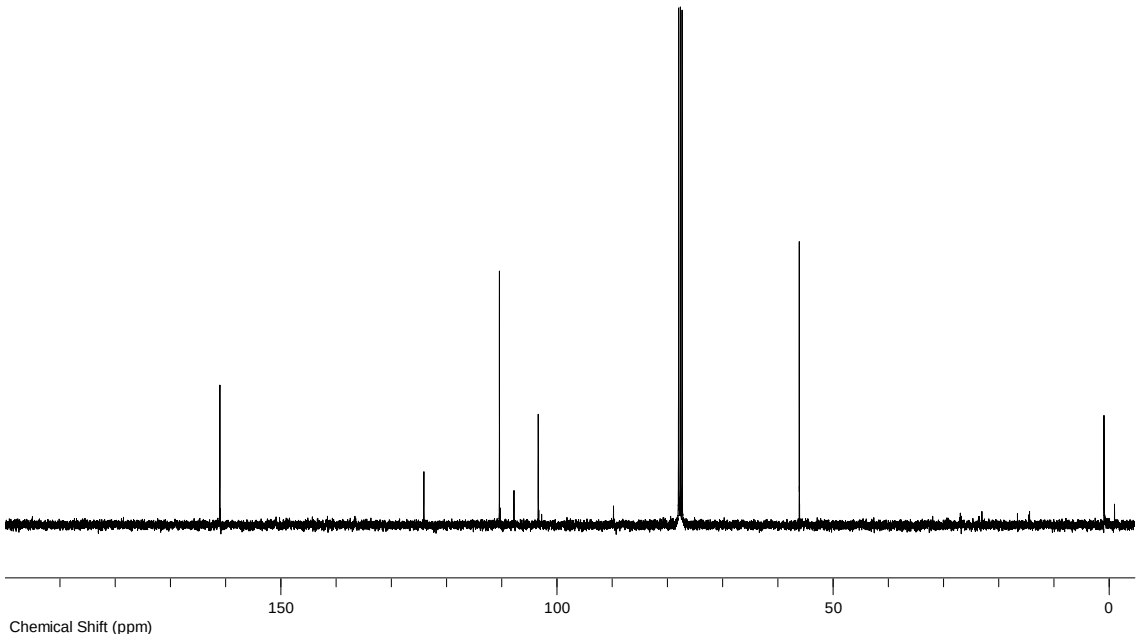

150
100
50
0
Chemical Shift (ppm)

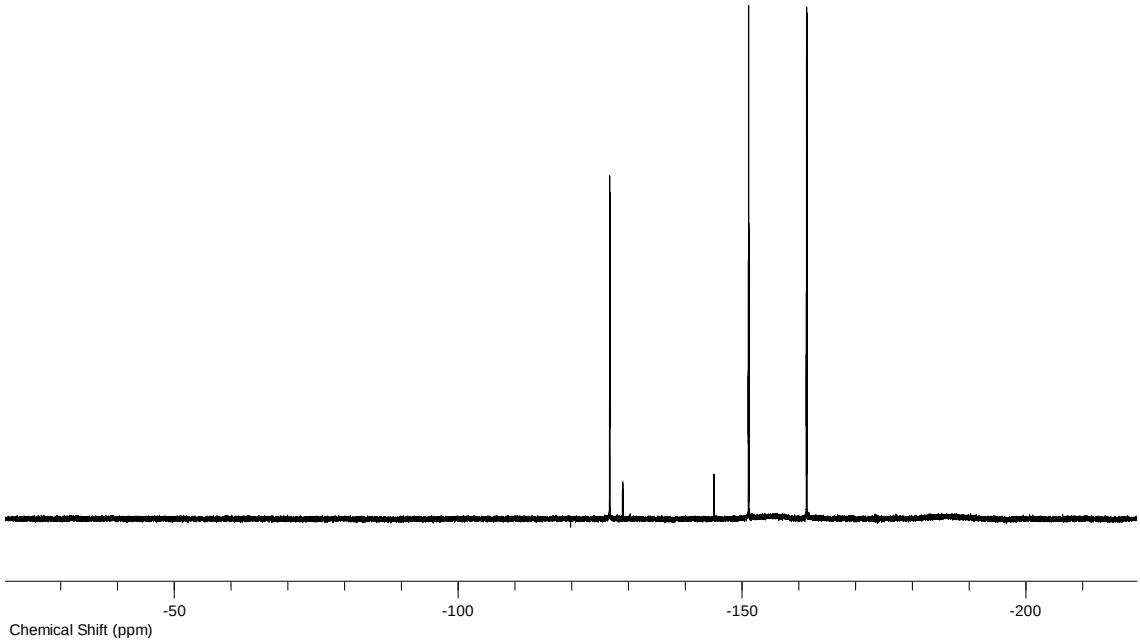


((3,5-Dimethoxyphenyl)ethynyl)dimethyl(perfluorophenyl)silane (Scheme 1, 9)

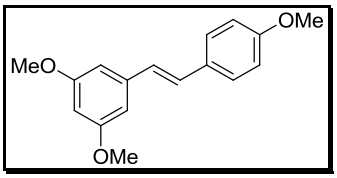


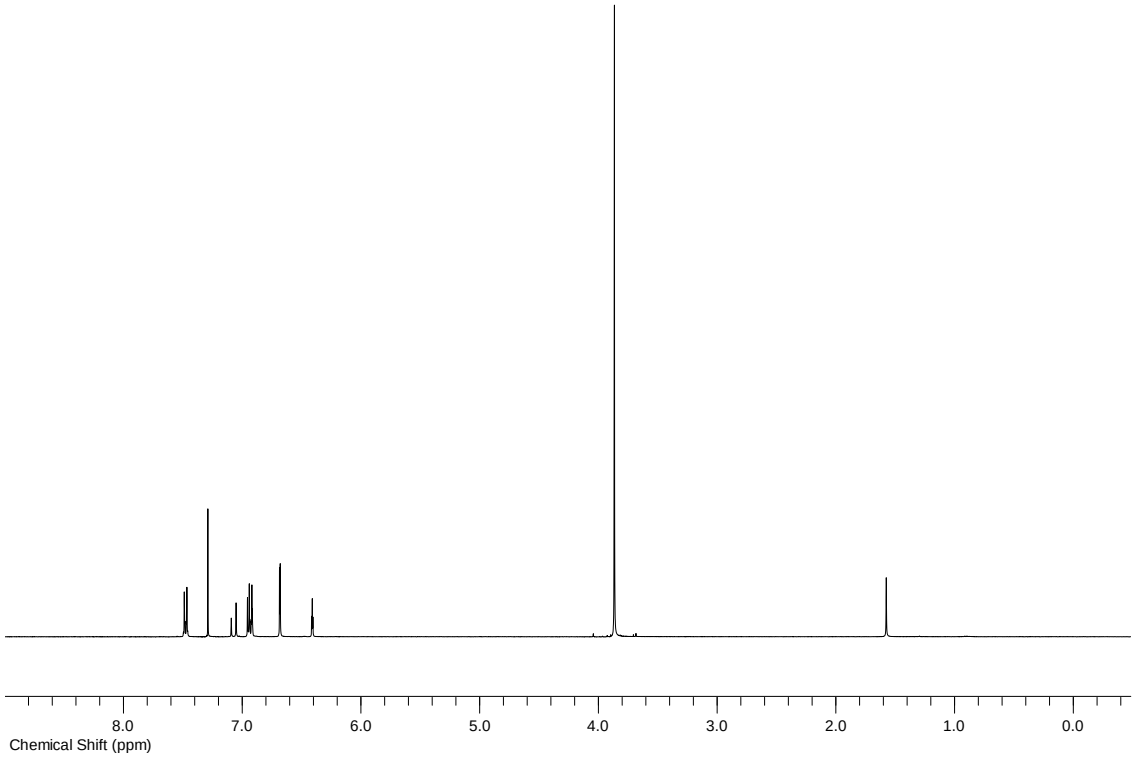


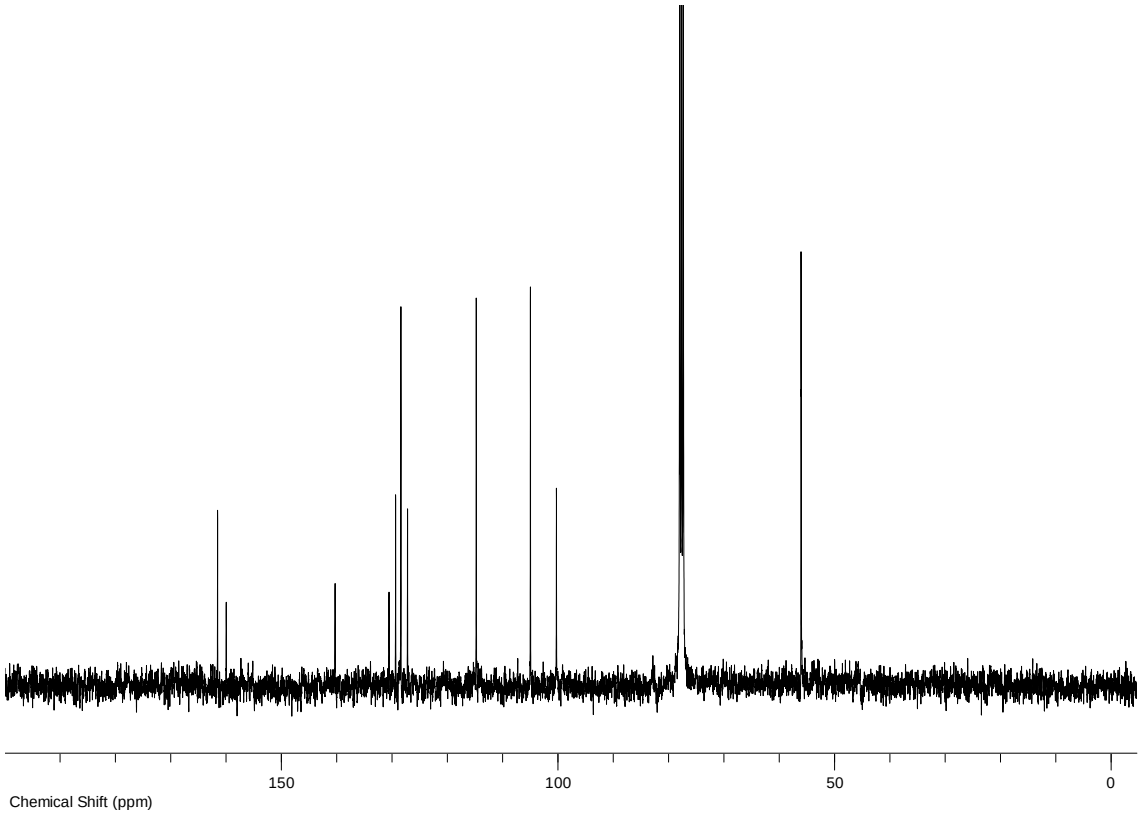

150
100
50
0
Chemical Shift (ppm)


-50
-100
-150
-200
Chemical Shift (ppm)

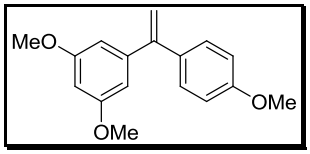
(*E*)-1,3-Dimethoxy-5-(4-methoxystyryl)benzene (Scheme 1, 10-(*E*))

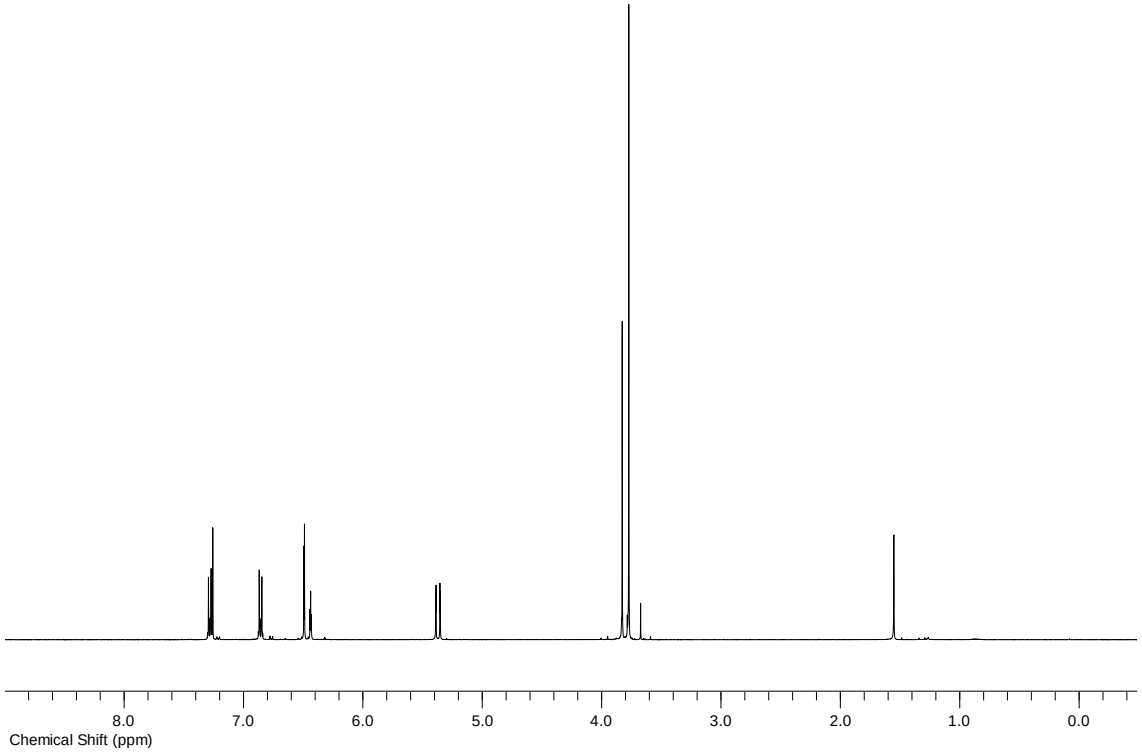


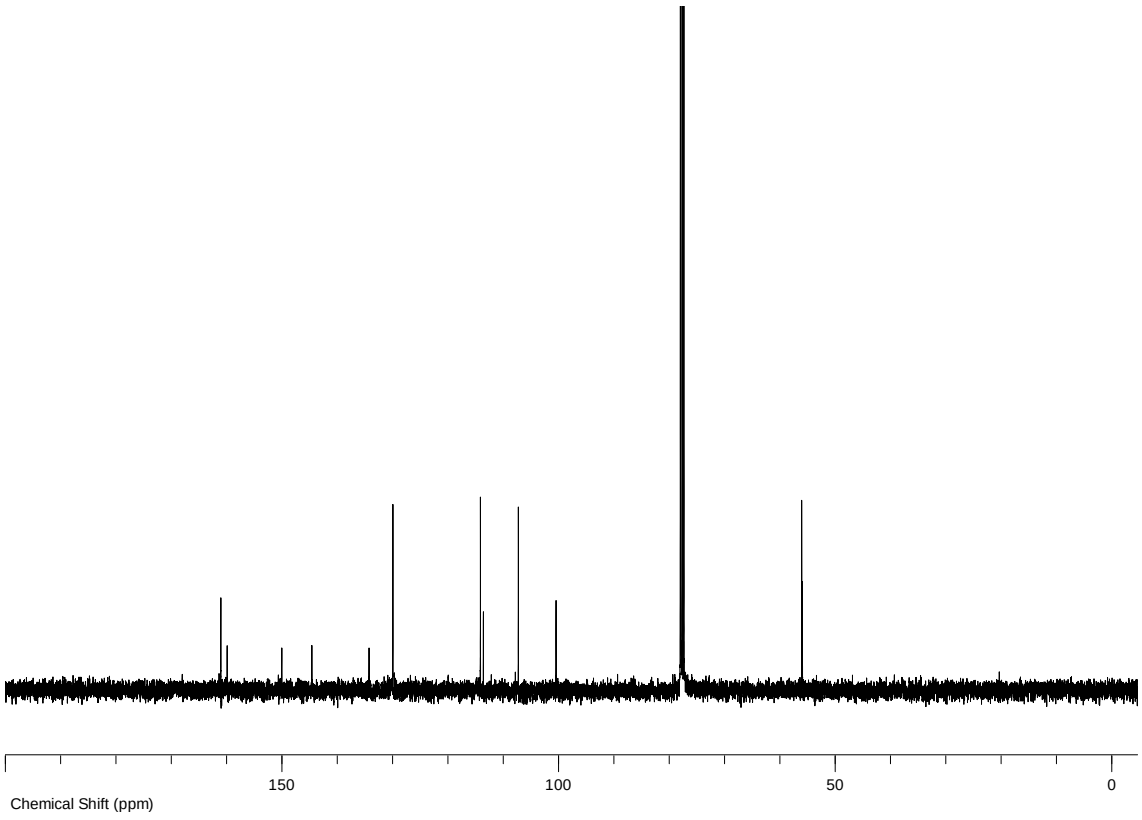




1,3-Dimethoxy-5-(1-(4-methoxyphenyl)vinyl)benzene (Scheme 1, 10-α)







(*Z*)-1,3-dimethoxy-5-(4-methoxystyryl)benzene (Scheme 1, 10-(*Z*))