Electronic Supplementary Material (ESI) for ChemComm.

Supplementary Information

Copper-catalyzed α-selective hydrostannylation of alkynes for synthesis of branched alkenylstannanes

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Contents

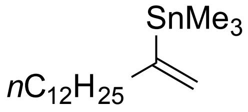
General remarks.

All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique under a purified argon atmosphere. Nuclear magnetic resonance spectra were taken on a Varian 400-MR (^{1}H, 400 MHz; ^{13}C, 100 MHz) spectrometer or a Varian System 500 (^{1}H, 500 MHz; ^{13}C, 125 MHz; ^{119}Sn, 186 MHz) spectrometer using residual chloroform (^{1}H, $\delta = 7.26$) or $CDCl_3$ (^{13}C, $\delta = 77.0$) as an internal standard, and tetramethyltin (^{119}Sn, $\delta = 0$) as an external standard. ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, quint = quintet, sext = sextet, m = multiplet), coupling constants (Hz), integration. High-resolution mass spectra (ESI or APCI/FTMS; Negative Mode) were obtained with Thermo Fisher Scientific LTQ Orbitrap XL spectrometer. Preparative recycling gel permeation chromatography was performed with JAI LC-908 or JAI LC-9201 equipped with JAI GEL-1H and -2H columns (chloroform or toluene as an eluent). Column chromatography was carried out using Merck Kieselgel 60. Unless otherwise noted, commercially available reagents were used without purification. Toluene was distilled from sodium/benzophenone ketyl. DMF was distilled from calcium hydride.

Hydrostannylation of alkynes with $Me_3SnSnMe_3$: a general procedure.

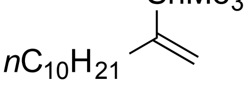
A Schlenk tube equipped with a magnetic stirring bar was charged with $Cu(OAc)_2$ (0.015 mmol), tri-*tert*-butylphosphine (20 wt% solution in toluene, 0.053 mmol) and MeOH (2.5 mL). After the mixture was stirred at 80 °C for 0.5 h, the solvent was removed in vacuo at room temperature. To the residue were added toluene (0.2 mL), hexamethyldistannane (0.39 mmol), an alkyne (0.30 mmol) and water (0.90 mmol), and the resulting mixture was stirred at 110 °C for the period as specified in Table 1. The mixture was diluted with ethyl acetate and filtered through a Celite plug before evaporation of the solvent. Then the residue was diluted with toluene and filtered through neutral alumina (activity = IV). Evaporation of the solvent followed by gel permeation chromatography (chloroform as an eluent) gave the corresponding product.

Trimethyl(tetradec-1-en-2-yl)stannane (2a)



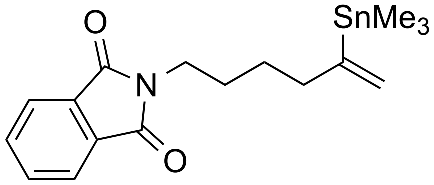
A colorless oil: ^{1}H NMR ($CDCl_3$) δ 0.13 (s, $^{2}J_{^{119}\mathrm{Sn\text{-}H}}$ = 53.9 Hz, $^{2}J_{^{117}\mathrm{Sn\text{-}H}}$ = 51.6 Hz, 9H), 0.88 (t, J = 6.7 Hz, 3H), 1.20-1.42 (m, 20H), 2.26 (t, $^{3}J_{\mathrm{Sn\text{-}H}}$ = 53.2 Hz, J =7.2 Hz, 2H), 5.12 (dt, $^{3}J_{\mathrm{Sn\text{-}H}}$ = 71.9 Hz, J = 2.8, 1.0 Hz 1H), 5.64 (dt, $^{3}J_{\mathrm{Sn\text{-}H}}$ = 155.0 Hz, J = 2.8, 1.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ -9.48, 14.13, 22.70, 29.21, 29.36, 29.48, 29.63, 29.65, 29.67, 31.93, 124.15, 156.17; ^{119}Sn NMR ($CDCl_3$) δ -35.6; HRMS Calcd for $C_{16}H_{33}Sn$: $[M\text{-}Me]^+$, 345.15987. Found: *m/z* 345.15985.

Dodec-1-en-2-yltrimethylstannane (2b)



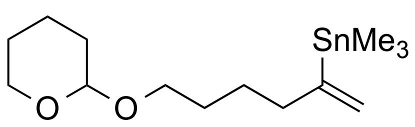
A colorless oil: ^{1}H NMR ($CDCl_3$) δ 0.13 (s, $^{2}J_{^{119}\mathrm{Sn\text{-}H}}$ = 53.9 Hz, $^{2}J_{^{117}\mathrm{Sn\text{-}H}}$ = 51.5 Hz, 9H), 0.88 (t, J = 6.5 Hz, 3H), 1.20-1.41 (m, 16H), 2.27 (t, $^{3}J_{\mathrm{Sn\text{-}H}}$ = 53.2 Hz, J =7.4 Hz, 2H), 5.12 (dt, $^{3}J_{\mathrm{Sn\text{-}H}}$ = 73.3 Hz, J = 2.9, 1.0 Hz, 1H), 5.64 (dt, $^{3}J_{\mathrm{Sn\text{-}H}}$ = 155.0 Hz, J = 2.9, 1.0 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ -9.48, 14.14, 22.70, 29.21, 29.36, 29.49, 29.64, 29.67, 31.93, 124.16, 156.15; ^{119}Sn NMR ($CDCl_3$) δ -35.6; HRMS Calcd for $C_{14}H_{29}Sn$: $[M\text{-}Me]^+$, 317.12857. Found: *m/z* 317.12854.

2-[5-(Trimethylstannyl)hex-5-enyl]isoindoline-1,3-dione (2c)



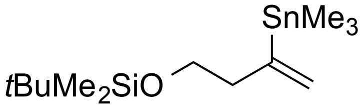
A green oil: ^{1}H NMR (CDCl$_3$) δ 0.11 (s, $^{2}J_{^{119}\mathrm{Sn\text{-}H}}$ = 54.0 Hz,$^{2}J_{^{117}\mathrm{Sn\text{-}H}}$ = 51.6 Hz, 9H), 1.43 (quint, J = 7.5 Hz, 2H), 1.66 (quint, J = 7.5 Hz, 2H), 2.31 (t, $^{3}J_{\mathrm{Sn\text{-}H}}$ = 51.7 Hz, J = 1.3 Hz, 2H), 3.68 (t, J = 7.5 Hz, 2H) 5.13 (dt, $^{3}J_{\mathrm{Sn\text{-}H}}$ = 71.0 Hz, J = 2.7, 1.3 Hz, 1H), 5.78 (dt, $^{3}J_{\mathrm{Sn\text{-}H}}$ = 151.7 Hz, J = 2.7, 1.4 Hz, 1H), 7.68-7.73 (m, 2H), 7.81-7.86 (m, 2H); ^{13}C NMR (CDCl$_3$) δ -9.53, 26.66, 28.03, 37.80, 40.16, 123.14, 124.93, 132.14, 144.84, 155.08, 168.41; ^{119}Sn NMR (CDCl$_3$) δ -34.6; HRMS Calcd for $C_{17}H_{24}O_2NSn$: [M+H]$^+$, 374.08235. Found: *m/z* 394.08188.

Trimethyl[6-(tetrahydro-2H-pyran-2-yloxy)hex-1-en-2-yl]stannane (2d)



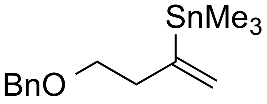
A colorless oil: ^{1}H NMR (CDCl$_3$) δ 0.13 (s, $^{2}J_{^{119}\mathrm{Sn\text{-}H}}$ = 54.0 Hz, $^{2}J_{^{117}\mathrm{Sn\text{-}H}}$ = 51.7,9H), 1.41-1.63 (m, 8H), 1.66-1.75 (m, 1H), 1.77-1.88 (m, 1H), 2.30 (t, $^{3}J_{^{119}\mathrm{Sn\text{-}H}}$ = 53.4 Hz, $^{3}J_{^{117}\mathrm{Sn\text{-}H}}$ = 51.5, J =7.5 Hz, 2H), 3.38 (dt, J = 9.6 Hz, 6.4 Hz, 1 H), 3.47-3.54 (m, 1H), 3.73 (dt, J = 9.6, 6.6 Hz, 1H), 3.86 (ddd, J = 11.2, 7.9, 3.5 Hz, 1H), 4.58 (t, J = 2.7 Hz, 1H), 5.14 (s, $^{3}J_{\mathrm{Sn\text{-}H}}$ = 71.4 Hz, 1H), 5.65 (s, $^{3}J_{\mathrm{Sn\text{-}H}}$ = 153.15 Hz, 1H); ^{13}C NMR (CDCl$_3$) δ -9.53, 19.52, 25.46, 26.17, 29.19, 30.69, 40.54, 62.12, 67.23, 98.67, 124.53, 155.61; ^{119}Sn NMR (CDCl$_3$) δ -35.1; HRMS Calcd for $C_{14}H_{28}O_2NaSn$: [M+Na]$^+$, 371.1004. Found: *m/z* 371.1005.

***tert*-Butyldimethyl[3-(trimethylstannyl)but-3-enyloxy]silane (2e)**



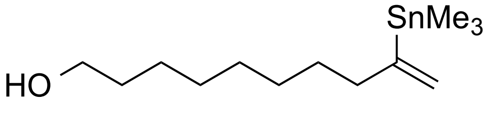
A colorless oil: ^{1}H NMR (CDCl$_3$) δ 0.06 (s, 6H), 0.14 (s, $^{2}J_{^{119}\mathrm{Sn\text{-}H}}$ = 54.2 Hz, $^{2}J_{^{117}\mathrm{Sn\text{-}H}}$ = 51.8 Hz, 9H), 0.89 (s, 9H), 2.49 (t, $^{3}J_{^{119}\mathrm{Sn\text{-}H}}$ = 53.1 Hz, $^{3}J_{^{117}\mathrm{Sn\text{-}H}}$ = 51.0 Hz, J =7.3 Hz, 2H), 3.63 (t, J = 7.2 Hz, 2H), 5.21 (s, $^{3}J_{\mathrm{Sn\text{-}H}}$ = 71.0 Hz, 1H), 5.76 (s, $^{3}J_{\mathrm{Sn\text{-}H}}$ = 151.1 Hz, 1H); ^{13}C NMR (CDCl$_3$) δ -9.34, -5.18, 18.46, 26.02, 44.15, 63.41, 126.72, 151.65; ^{119}Sn NMR (CDCl$_3$) δ -33.2; HRMS Calcd for $C_{12}H_{27}OSiSn$: [M-Me]$^+$, 335.08477. Found: *m/z* 335.08469.

[4-(Benzyloxy)but-1-en-2-yl]trimethylstannane (2f)



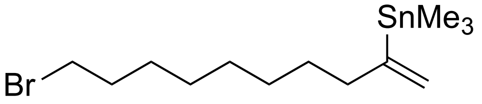
A yellow oil: ^{1}H NMR ($CDCl_3$) δ 0.13 (s, ${}^{2}J_{Sn\text{-}H}$ = 53.1 Hz, 9H), 2.61 (t, ${}^{3}J_{Sn\text{-}H}$ = 50.9 Hz, J =6.4 Hz, 2H), 3.52 (t, J = 6.9 Hz, 2H), 4.52 (s, 2H), 5.25 (dt, ${}^{3}J_{Sn\text{-}H}$ = 70.13 Hz, J = 2.7, 1.4 Hz,, 1H), 5.76 (dt, ${}^{3}J_{Sn\text{-}H}$ = 129.75 Hz, J = 2.8, 1.4 Hz, 1H), 7.27-7.39 (m, 5H); ^{13}C NMR ($CDCl_3$) δ -9.24, 40.88, 70.16, 72.95, 126.41, 127.51, 127.73, 128.29, 138.29, 152.26; ^{119}Sn NMR ($CDCl_3$) δ -33.7; HRMS Calcd for $C_{13}H_{19}OSn$: [M-Me]$^+$, 311.04524. Found: *m/z* 311.04517.

9-(Trimethylstannyl)dec-9-en-1-ol (2g)



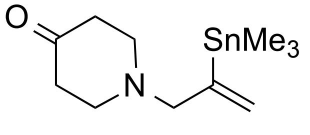
A brown oil: ^{1}H NMR ($CDCl_3$) δ 0.12 (s, ${}^{2}J_{Sn\text{-}H}$ = 52.8 Hz, 9H), 1.21 (m, 12H), 2.25 (t, ${}^{3}J_{Sn\text{-}H}$ = 53.1 Hz, J =7.6 Hz, 2H), 3.62 (dt, J = 6.4 Hz, 5.3 Hz 2H), 5.11 (s, ${}^{3}J_{Sn\text{-}H}$ = 72.1 Hz, 1H), 5.76 (s, ${}^{3}J_{Sn\text{-}H}$ = 154.2 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ -9.50, 25.69, 29.07, 29.36, 29.39, 29.59, 32.75, 40.80, 63.00, 124.20, 156.04; ^{119}Sn NMR ($CDCl_3$) δ -35.5; HRMS Calcd for $C_{12}H_{25}OSn$: [M-Me]$^+$, 305.09219. Found: *m/z* 305.0921.

(10-Bromodec-1-en-2-yl)trimethylstannane (2h)



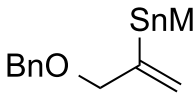
A yellow oil: ^{1}H NMR ($CDCl_3$) δ 0.13 (s, ${}^{2}J^{119}{}_{Sn\text{-}H}$ = 53.9 Hz, ${}^{2}J^{117}{}_{Sn\text{-}H}$ = 51.6 Hz, 9H), 1.20-1.48 (m, 12H), 1.85 (quint, J = 7.1 Hz), 2.26 (t, ${}^{3}J^{119}{}_{Sn\text{-}H}$ = 53.9 Hz, ${}^{3}J^{117}{}_{Sn\text{-}H}$ = 51.8, J =7.1 Hz, 2H), 5.12 (s, ${}^{3}J_{Sn\text{-}H}$ = 71.8 Hz, 1H), 5.76 (s, ${}^{3}J_{Sn\text{-}H}$ = 154.2 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ -9.48, 28.14, 28.71, 29.03, 29.23, 29.55, 32.80, 34.02, 40.78, 124.25, 156.00; ^{119}Sn NMR ($CDCl_3$) δ -35.5; HRMS Calcd for $C_{12}H_{24}BrSn$: [M-Me]$^+$, 367.00779. Found: *m/z* 367.00677.

1-[2-(Trimethylstannyl)allyl]piperidin-4-one (2i)



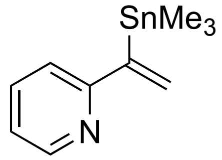
A yellow oil: ^{1}H NMR ($CDCl_3$) δ 0.16 (s, ${}^{2}J^{119}{}_{Sn\text{-}H}$ = 54.8 Hz, ${}^{2}J^{117}{}_{Sn\text{-}H}$ = 52.4 Hz, 9H), 2.41 (t, J = 6.2 Hz, 4H), 2.68 (t, J = 5.8 Hz, 4H), 3.19 (t, ${}^{3}J_{Sn\text{-}H}$ = 49.8 Hz, J = 1.3 Hz, 2H), 5.28 (dt, ${}^{3}J_{Sn\text{-}H}$ = 70.0 Hz, J = 2.5, 1.4 Hz, 1H), 5.78 (dt, ${}^{3}J_{Sn\text{-}H}$ = 149.6 Hz, J = 2.5, 1.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ -9.38, 41.25, 53.00, 67.14, 125.77, 154,92, 209.41; ^{119}Sn NMR ($CDCl_3$) δ -38.5; HRMS Calcd for $C_{11}H_{22}ONSn$: [M+H]$^+$, 304.07169. Found: *m/z* 304.07166.

[3-(Benzyloxy)prop-1-en-2-yl]trimethylstannane (2j)



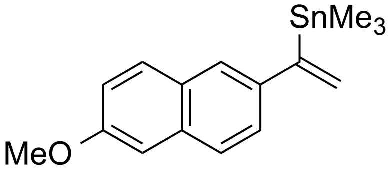
A brown oil: ^{1}H NMR (CDCl$_3$) δ 0.17 (s, $^{2}J^{119}{}_{\text{Sn-H}}$ = 55.4 Hz, $^{2}J^{117}{}_{\text{Sn-H}}$ = 52.9 Hz, 9H), 4.20 (t, $^{3}J_{\text{Sn-H}}$ = 39.5 Hz, J =1.4 Hz, 2H), 4.50(s, 2H), 5.33 (dt, $^{3}J_{\text{Sn-H}}$ = 70.2 Hz, J = 2.8, 0.9 Hz, 1H), 5.87 (dt, $^{3}J_{\text{Sn-H}}$ = 144.96 Hz, J = 2.5, 1.4 Hz, , 1H), 7.27-7.39 (m, 5H); ^{13}C NMR (CDCl$_3$) δ -9.47, 72.02. 77.05, 124.65, 127.44, 127.64, 128.26, 138.38, 153.32; ^{119}Sn NMR (CDCl$_3$) δ -35.0; HRMS Calcd for $C_{12}H_{17}OSn$: [M-Me]$^+$, 297.02959. Found: *m/z* 297.02939.

2-[1-(Trimethylstannyl)vinyl]pyridine (2k)



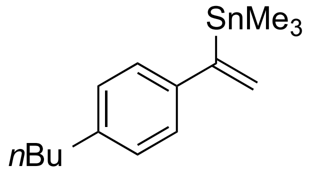
A brown oil: ^{1}H NMR (CDCl$_3$) δ 0.22 (s, $^{3}J^{119}{}_{\text{Sn-H}}$ = 55.7 Hz,$^{3}J^{117}{}_{\text{Sn-H}}$ = 53.2 Hz, 9H), 7.51 (d, $^{3}J_{\text{Sn-H}}$ = 72.0 Hz, J = 1.7 Hz, 1H), 6.48 (d, $^{3}J_{\text{Sn-H}}$ = 145.1 Hz, J = 1.8 Hz, 1H), 7.07 (ddd, J = 7.3, 4.9, 1.1 Hz, 1H), 7.50 (dt, J = 8.0, 1.1 Hz, 1H), 7.60 (td, J = 7.7, 1.8 Hz, 1H), 8.50 (ddd, J = 4.9, 1.8, 0.9 Hz, 1H); ^{13}C NMR (CDCl$_3$) δ -8.22, 117.85, 121.36, 126.59, 136.09, 148.76, 156.83; ^{119}Sn NMR (CDCl$_3$) δ -35.8; HRMS Calcd for $C_9H_{12}NSn$: [M-Me]$^+$, 253.99862. Found: *m/z* 253.99835.

[1-(6-Methoxynaphthalen-2-yl)vinyl]trimethylstannane (2l)



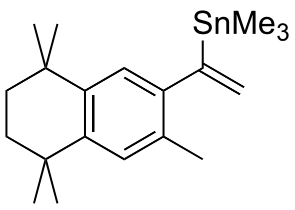
A yellow needle: mp = 64.2 °C; ^{1}H NMR (CDCl$_3$) δ 0.27 (s, $^{3}J^{119}{}_{\text{Sn-H}}$ = 54.4 Hz, $^{2}J^{117}{}_{\text{Sn-H}}$ = 52.0 Hz, 9H), 3.92 (s, 3H), 5.51 (d, $^{3}J_{\text{Sn-H}}$ = 69.2 Hz, J = 2.3 Hz 1H), 6.16 (d, $^{3}J_{\text{Sn-H}}$ = 142.0 Hz, J = 2.3 Hz 1H), 7.10-7.16 (m, 2H), 7.39 (dd, J = 8.5, 1.9 Hz, 1H), 7.53-7.56 (m, 1H), 7.64-7.63 (m, 2H); ^{13}C NMR (CDCl$_3$) δ -9.56. -8.54, 55.31, 105.61, 105.80, 123.70, 124.90, 125.34, 125.93, 126.08, 126.79, 126.94, 129.03, 129.08, 129.44, 129.66, 133.40, 140.64, 145.71, 154.40, 157.42; ^{119}Sn NMR (CDCl$_3$) δ -28.1; HRMS Calcd for C16H21OSn: [M+H]$^+$, 349.06089. Found: *m/z* 349.06079.

[1-(4-Butylphenyl)vinyl]trimethylstannane (2m)



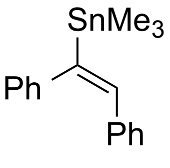
A yellow oil: ^{1}H NMR (CDCl$_3$) δ 0.24 (s, $^3J_{^{119}\mathrm{Sn\text{-}H}}$ = 54.3 Hz, $^2J_{^{117}\mathrm{Sn\text{-}H}}$ = 52.1 Hz, 9H), 0.94 (t, J = 7.3 Hz, 3H), 1.38 (sext, J = 7.2 Hz, 2H), 1.56-1.66 (m, 2H), 2.60 (t, J = 7.7 Hz, 2H), 5.42 (d, $^3J_{\mathrm{Sn\text{-}H}}$ = 69.8 Hz, J = 2.1 Hz, 1H), 6.07 (d, $^3J_{\mathrm{Sn\text{-}H}}$ = 144.4 Hz, J = 2.2 Hz, 1H), 7.10-7.18 (m, 4H); ^{13}C NMR (CDCl$_3$) δ -8.62, 13.97, 22.39, 33.59, 35.26, 125.45, 126.14, 128.43, 141.28, 142.47, 154.11; ^{119}Sn NMR (CDCl$_3$) δ -28.8; HRMS Calcd for $C_{15}H_{25}Sn$: [M+H]$^+$, 325.09727. Found: *m/z* 325.09729.

Trimethyl[1-(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)vinyl]stannane (2n)



A white powder: mp = 47.4 °C; ^{1}H NMR (CDCl$_3$) δ 0.15 (s, $^3J_{^{119}\mathrm{Sn\text{-}H}}$ = 54.1 Hz, $^2J_{^{117}\mathrm{Sn\text{-}H}}$ = 51.8 Hz, 9H), 1.25 (s, 6H), 1.27 (s, 6H), 1.66 (m, 4H), 2.16 (s, 3H), 5.56 (d, $^3J_{\mathrm{Sn\text{-}H}}$ = 71.9 Hz, J = 3.2 Hz 1H), 5.77 (d $^3J_{\mathrm{Sn\text{-}H}}$ = 148.2 Hz, J = 3.0 Hz 1H), 6.78 (s, 1H), 7.04 (s, 1H); ^{13}C NMR (CDCl$_3$) δ -8.89, 20.11, 31.88, 31.93, 33.80, 35.23, 35.26, 124.82, 127.38, 127.69, 130.08, 141.71, 141.84, 143.17, 156.05; ^{119}Sn NMR (CDCl$_3$) δ -28.1; HRMS Calcd for $C_{20}H_{33}Sn$: [M+H]$^+$, 393.15987. Found: *m/z* 393.15988.

(*E*)-(1,2-Diphenylvinyl)trimethylstannane (2o)[1]

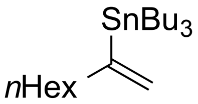


A green oil: ^{1}H NMR (CDCl$_3$) δ 0.18 (s, $^3J_{^{119}\mathrm{Sn\text{-}H}}$ = 54.2 Hz, $^2J_{^{117}\mathrm{Sn\text{-}H}}$ = 51.8 Hz, 9H), 6.67 (s, $^3J_{\mathrm{Sn\text{-}H}}$ = 76.1 Hz, 1H), 6.95-7.03 (m, 4H), 7.06-7.18 (m, 4H), 7.24-7.31 (m, 2H); ^{13}C NMR (CDCl$_3$) δ -9.23, 125.17, 126.15, 126.73, 127.90, 128.70, 129.13, 137.29, 137.74, 145.02, 149.75; ^{119}Sn NMR (CDCl$_3$) δ -17.4.

Hydrostannylation of alkynes with $Me_3SiSnBu_3$: a general procedure.

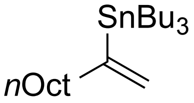
A Schlenk tube equipped with a magnetic stirring bar was charged with Cu(OAc)$_2$ (0.030 mmol), tricyclohexylphosphine (20 wt% solution in toluene, 0.105 mmol) and MeOH (5 mL). After the mixture was stirred at 80 °C for 0.5 h, the solvent was removed in vacuo at room temperature. To the residue were added toluene (0.2 mL), tributyl(trimethylsilyl)stannane (0.30 mmol), an alkyne (0.45 mmol) and water (0.45 mmol), and the resulting mixture was stirred at 30 °C for the period as specified in Table 2. The mixture was diluted with ethyl acetate and filtered through a Celite plug before drying over $MgSO_4$. Evaporation of the solvent followed by gel permeation chromatography (chloroform as an eluent) gave the corresponding product.

Tributyl(oct-1-en-2-yl)stannane (3p)



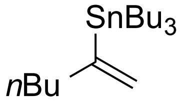
A colorless oil: ^{1}H NMR ($CDCl_3$) δ 0.79-0.98 (m, 16H), 1.21-1.62 (m, 22H), 2.23 (t, $^3J_{Sn\text{-}H}$ = 47.9 Hz, J = 7.1 Hz, 2H), 5.09 (s, $^3J_{Sn\text{-}H}$ = 63.5 Hz, 1H), 5.66 (s, $^3J_{Sn\text{-}H}$ = 141.8 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 9.56, 13.69, 14.11, 22.66, 27.43, 29.00, 29.14, 29.67, 31.77, 41.46, 124.50, 155.84; ^{119}Sn NMR ($CDCl_3$) δ -47.3; HRMS Calcd for $C_{20}H_{42}Sn$: $[M]^+$, 402.23085. Found: *m*/*z* 402.23115.

Tributyl(dec-1-en-2-yl)stannane (3q)



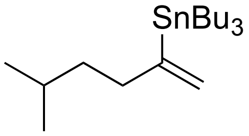
A pale yellow oil: ^{1}H NMR ($CDCl_3$) δ 0.77-1.00 (m, 18H), 1.20-1.40 (m, 18H), 1.41-1.57 (m, 6H), 2.23 (t, $^3J_{Sn\text{-}H}$ = 47.8 Hz, J = 7.3 Hz, 2H), 5.08 (d, $^3J_{Sn\text{-}H}$ = 63.8 Hz, J = 3.1 Hz, 1H), 5.65 (dt, $^3J_{Sn\text{-}H}$ = 141.3 Hz, J = 3.1, 1.3 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 9.57, 13.71, 14.12, 22.72, 27.45, 29.16, 29.35, 29.52, 29.74, 31.93, 41.48, 124.53, 155.78; ^{119}Sn NMR ($CDCl_3$) δ -47.3; HRMS Calcd for $C_{22}H_{46}Sn$: $[M]^+$, 430.26215. Found: *m*/*z* 430.26294.

Tributyl(hex-1-en-2-yl)stannane (3r)



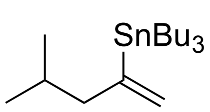
A colorless oil: ^{1}H NMR ($CDCl_3$) δ 0.81-0.97 (m, 18H), 1.25-1.40 (m, 10H), 1.41-1.58 (m, 6H), 2.25 (t, $^3J_{Sn\text{-}H}$ = 48.2 Hz, J = 7.1 Hz, 2H), 5.09 (d, $^3J_{Sn\text{-}H}$ = 64.9 Hz, J = 2.9 Hz, 1H), 5.66 (dt, $^3J_{Sn\text{-}H}$ = 141.1 Hz, J = 2.9, 1.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 9.56, 13.70, 13.99, 22.35, 27.43, 29.15, 31.88, 41.11, 124.53, 155.73; ^{119}Sn NMR ($CDCl_3$) δ -47.2; HRMS Calcd for $C_{18}H_{38}Sn$: $[M]^+$, 374.19955. Found: *m*/*z* 374.19984.

Tributyl(5-methylhex-1-en-2-yl)stannane (3s)



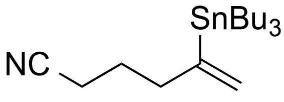
A colorless oil: ^{1}H NMR (CDCl$_3$) δ 0.79-0.98 (m, 18H), 1.18-1.37 (m, 8H), 1.38-1.59 (m, 7H), 2.24 (t, $^{3}J_{\text{Sn-H}}$ = 48.3 Hz, J = 8.1 Hz, 2H), 5.07 (d, $^{3}J_{\text{Sn-H}}$ = 64.7 Hz, J = 2.9 Hz, 1H), 5.66 (dt, $^{3}J_{\text{Sn-H}}$ = 141.8 Hz, J = 2.9, 1.5 Hz, 1H); ^{13}C NMR (CDCl$_3$) δ 9.58, 13.70, 22.53, 27.42, 27.62, 29.14, 39.03, 39.22, 124.38, 155.95; ^{119}Sn NMR (CDCl$_3$) δ -46.9; HRMS Calcd for $C_{19}H_{40}Sn$: [M]$^+$, 388.21520. Found: *m/z* 388.21594.

Tributyl(4-methylpent-1-en-2-yl)stannane (3t)



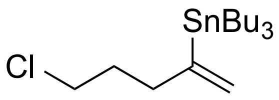
A colorless oil: ^{1}H NMR (CDCl$_3$) δ 0.78-0.98 (m, 23H), 1.25-1.37 (m, 6H), 1.39-1.65 (m, 7H), 2.12 (d, $^{3}J_{\text{Sn-H}}$ = 52.1 Hz, J = 7.1 Hz, 2H), 5.13 (d, $^{3}J_{\text{Sn-H}}$ = 64.3 Hz, J = 3.1 Hz, 1H), 5.62 (dt, $^{3}J_{\text{Sn-H}}$ = 141.7 Hz, J = 3.1, 1.6 Hz, 1H); ^{13}C NMR (CDCl$_3$) δ 9.59, 13.70, 22.45, 27.42, 27.85, 29.11, 51.39, 125.72, 154.98; ^{119}Sn NMR (CDCl$_3$) δ -47.2; HRMS Calcd for $C_{18}H_{38}Sn$: [M]$^+$, 374.19955. Found: *m/z* 374.20060.

5-(Tributylstannyl)hex-5-enenitrile (3u)



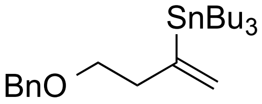
A colorless oil: ^{1}H NMR (CDCl$_3$) δ 0.80-1.00 (m, 15H), 1.22-1.38 (m, 6H), 1.39-1.58 (m, 6H), 1.67-1.81 (m, 2H), 2.31 (t, J = 7.2 Hz, 2H), 2.38 (t, $^{3}J_{\text{Sn-H}}$ = 44.6 Hz, J = 7.3 Hz, 2H), 5.20 (s, $^{3}J_{\text{Sn-H}}$ = 61.5 Hz, 1H), 5.72 (s, $^{3}J_{\text{Sn-H}}$ = 133.6 Hz, 1H); ^{13}C NMR (CDCl$_3$) δ 9.53, 13.64, 16.32, 24.86, 27.33, 29.06, 39.64, 119.56, 126.87, 152.70; ^{119}Sn NMR (CDCl$_3$) δ -45.5; HRMS Calcd for $C_{14}H_{26}NSn$: [M-Bu]$^+$, 328.10872. Found: *m/z* 328.10805.

Tributyl(5-chloropent-1-en-2-yl)stannane (3v)



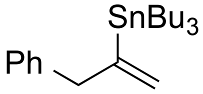
A pale yellow oil: ^{1}H NMR (CDCl$_3$) δ 0.82-0.98 (m, 15H), 1.26-1.36 (m, 6H), 1.40-1.57 (m, 6H), 1.85 (quint, J = 7.5 Hz, 2H), 2.38 (t, $^{3}J_{\text{Sn-H}}$ = 45.0 Hz, J = 7.5 Hz, 2H), 3.52 (t, J = 6.7 Hz, 2H), 5.16 (dt, $^{3}J_{\text{Sn-H}}$ = 63.1 Hz, J = 2.7, 1.1 Hz, 1H), 5.71 (dt, $^{3}J_{\text{Sn-H}}$ = 136.9 Hz, J = 2.7, 1.5 Hz, 1H); ^{13}C NMR (CDCl$_3$) δ 9.54, 13.69, 27.39, 29.10, 32.20, 38.09, 44.41, 126.04, 153.51; ^{119}Sn NMR ($CDCl_3$) δ -45.9; HRMS Calcd for $C_{13}H_{26}ClSn$: $[M-Bu]^+$, 337.07450. Found: *m*/*z* 337.07361.

[4-(Benzyloxy)but-1-en-2-yl]tributylstannane (3f)



A pale yellow oil: ^{1}H NMR ($CDCl_3$) δ 0.80-1.00 (m, 15H), 1.25-1.35 (m, 6H), 1.39-1.55 (m, 6H), 2.58 (t, $^3J_{Sn\text{-}H}$ = 45.1 Hz, J = 7.0 Hz, 2H), 3.50 (t, J = 7.2 Hz, 2H), 4.52 (s, 2H), 5.20 (d, $^3J_{Sn\text{-}H}$ = 63.0 Hz, J = 2.8 Hz, 1H), 5.76 (dt, $^3J_{Sn\text{-}H}$ = 137.1 Hz, J = 2.8, 1.5 Hz, 1H), 7.26-7.38 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 9.61, 13.69, 27.39, 29.08, 41.28, 70.21, 72.97, 126.86, 127.51, 127.72, 128.30, 138.38, 151.48; ^{119}Sn NMR ($CDCl_3$) δ -45.1; HRMS Calcd for $C_{19}H_{31}OSn$: $[M-Bu]^+$, 395.13969. Found: *m*/*z* 395.14076.

Tributyl(3-phenylprop-1-en-2-yl)stannane (3w)



A colorless oil: ^{1}H NMR ($CDCl_3$) δ 0.54-0.71 (m, 6H), 0.77 (t, J = 7.2 Hz, 9H), 1.10-1.20 (m, 6H), 1.21-1.35 (m, 6H), 3.50 (s, $^3J_{Sn\text{-}H}$ = 46.0 Hz, 2H), 5.15 (s, $^3J_{Sn\text{-}H}$ = 60.9 Hz, 1H), 5.65 (s, $^3J_{Sn\text{-}H}$ = 134.3 Hz, 1H), 7.04-7.14 (m, 3H), 7.19 (t, J = 7.7 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 9.45, 13.69, 27.36, 28.97, 47.72, 126.00, 126.05, 128.22, 129.08, 140.23, 154.20; ^{119}Sn NMR ($CDCl_3$) δ -44.1; HRMS Calcd for $C_{17}H_{27}Sn$: $[M-Bu]^+$, 351.11347. Found: *m*/*z* 351.11257.

Dideuteriostannylation of 1-tetradecyne.

A Schlenk tube equipped with a magnetic stirring bar was charged with $Cu(OAc)_2$ (0.015 mmol), tri-*tert*-butylphosphine (20 wt% solution in toluene, 0.053 mmol) and MeOH (2.5 mL). After the mixture was stirred at 80 °C for 0.5 h, the solvent was removed in vacuo at room temperature. To the residue were added toluene (0.2 mL), hexamethyldistannane (0.39 mmol), 1-tetradeyne (0.30 mmol) and deuterium oxide (0.90 mmol), and the resulting mixture was stirred at 110 °C for 2 h. The mixture was diluted with ethyl acetate and filtered through a Celite plug before evaporation of the solvent. Then the residue was diluted with toluene and filtered through neutral alumina (activity = IV). Evaporation of the solvent followed by gel permeation chromatography (chloroform as an eluent) gave the products. The ratio of the products was determined by ^{1}H NMR.

Hydrogen–deuterium exchange of 1-tetradecyne.

A Schlenk tube equipped with a magnetic stirring bar was charged with $Cu(OAc)_2$ (0.015 mmol), tri-*tert*-butylphosphine (20 wt% solution in toluene, 0.053 mmol) and MeOH (2.5 mL). After the mixture was stirred at 80 °C for 0.5 h, the solvent was removed in vacuo at room temperature. To the residue were added toluene (0.2 mL), 1-tetradeyne (0.30 mmol) and deuterium oxide (0.90 mmol), and the resulting mixture was stirred at 110 °C for 12 h. The mixture was diluted with ethyl acetate and filtered through a Celite plug before evaporation of the solvent. The hydrogen–deuterium exchange ratio was determined by ^1H NMR.

Cross-coupling of 2n: Synthesis of ethyl 4-(1-(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)vinyl)benzoate (4).

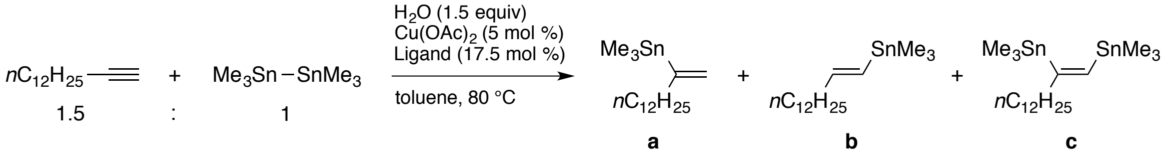
A Schlenk tube equipped with a magnetic stirring bar was charged with CsF (0.60 mmol). After CsF was dried with a heat gun in vacuo, $Pd(dba)_2$ (0.030 mmol), XPhos (0.060 mmol), CuI (0.060 mmol), **2n** (0.30 mmol) and DMF (5.1 mL) were added. To this solution was added a DMF solution (1.5 mL) of ethyl 4-iodobenzoate (0.36 mmol) over 15 min, and the resulting mixture was stirred at 100 °C for 15 h before dilution with ethyl acetate. The solution was washed with sat.NH_4Claq (twice) and brine (twice), and the organic layer was dried over $MgSO_4$. Evaporation of the solvent followed by gel permeation chromatography (chloroform as an eluent) gave **4**.[2]

Hydrolysis of 4: Synthesis of 4-(1-(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)vinyl)benzoic acid (bexarotene, 5).

A MeOH solution (5 mL) of **4** (0.270 mmol) and 5M NaOHaq (125 mL) was stirred at reflux temperature overnight before the mixture was acidified with 5N HCl and extracted with ethyl acetate. After the organic extract was washed with water, the solvent was removed in vacuo to give **5**.[3]

Optimization of reaction conditions for hydrostannylation with $Me_3SnSnMe_3$ (Table S1).

Table S1

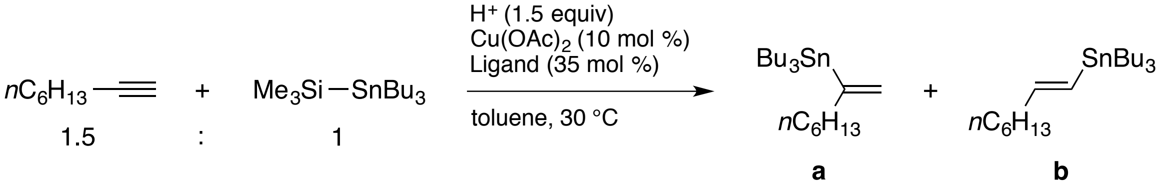


entry	Ligand	time (h)	NMR yield (%)		a:b
			a+b	c	
1	PCy_3	2	89	3	90:10
2[a]	PCy_3	16	69	8	88:12
3[b]	PCy_3	1	55	29	91:9
4[c]	PCy_3	0.5	84	6	90:10
5[d]	PCy_3	0.5	58	29	88:12
6[e]	PCy_3	0.5	64	29	81:19
7[f]	PPh_3	24	34	36	91:9
8	$PnBuAd_2$[g]	3.5	88	8	95:5
9	$PtBu_3$	22	63	0	95:5
10[h]	$PtBu_3$	0.5	80	0	96:4
11[h,i]	$PtBu_3$	1.5	95	0	97:3

[a] H_2O = 10 equiv. [b] MeOH was used instead of H_2O. [c] Solvent = THF. [d] Solvent = MeCN. [e] Solvent = DMF. [f] $(PPh_3)_3CuOAc$ (10 mol %) was used. Temperature = 60 °C. [g] Ad = 1-adamantyl. [h] Temperature = 110 °C. [i] alkyne:distannane:H_2O = 1:1.3:3.

Optimization of reaction conditions for hydrostannylation with $Me_3SiSnBu_3$ (Table S2).

Table S2

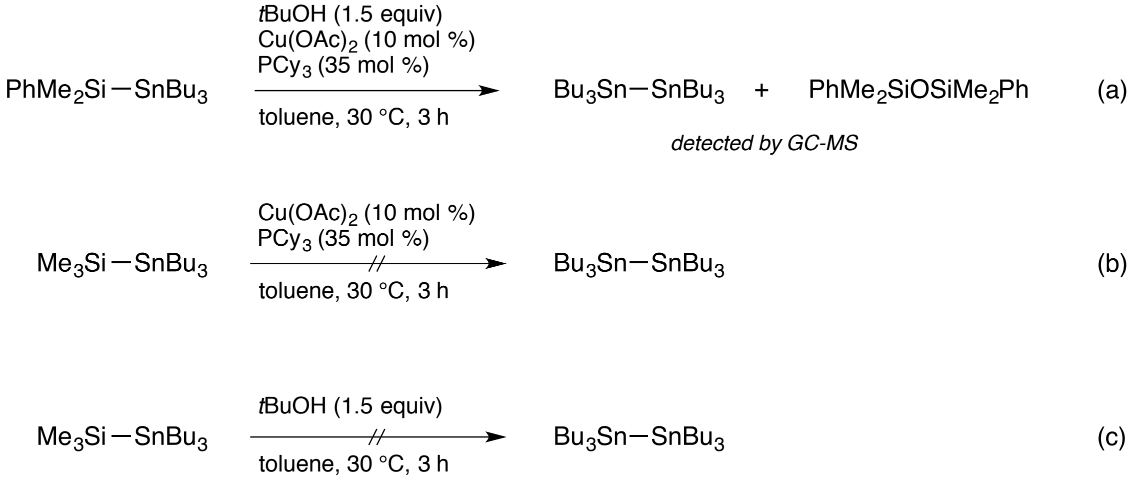


entry	Ligand	H^+	time (h)	NMR yield (%)	**a**:**b**
1	PCy_3	H_2O	1.5	75	86:14
2	$PCyp_3$[a]	H_2O	2	67	86:14
3	P*i*Pr_3	H_2O	50	43	92:8
4	P*n*$BuAd_2$[b]	H_2O	2	41	93:7
5[c]	P*t*Bu_3	H_2O	16	trace	–
6[d]	P*t*Bu_3	H_2O	16	23	>99:1
7	PCy_3	MeOH	13	6	–
8	PCy_3	*t*BuOH	2.5	64	89:11
9	PCy_3	PhOH	96	13	92:8
10	PCy_3	Ph_3COH	2	39	87:13
11	PCy_3	AcOH	2	8	–
12[e]	PCy_3	H_2O	44	trace	–

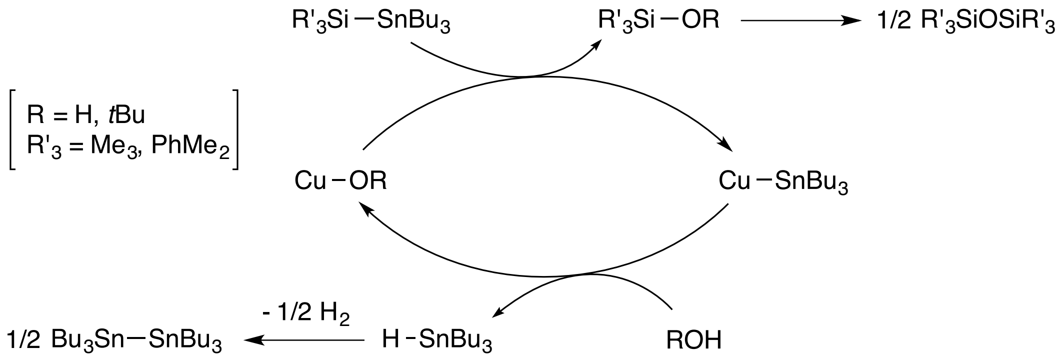
[a] Cyp = cyclopentyl. [b] Ad = 1-admantyl. [c] Temperature = 50 °C. [d] Temperature = 80 °C. [e] *t*$BuMe_2Si–SnBu_3$ was used instead of $Me_3Si–SnBu_3$. Temperature = 80 °C.

Formation of a distannane from a silylstannane (Schemes S1 and S2)

Treatment of a silylstannane ($PhMe_2SiSnBu_3$) with a proton source (*t*BuOH) in the presence of $Cu(OAc)_2$–PCy_3 catalyst was found to produce a distannane ($Bu_3SnSnBu_3$) and a disiloxane ($PhMe_2SiOSiMe_2Ph$) (Scheme S1, eq. a), whereas the reaction without the proton source (eq. b) or the copper catalyst (eq. c) did not give the distannane at all. Accordingly, this transformation should proceed through a pathway, which includes dehydrogenative coupling of tributyltin hydride, as depicted in Scheme S2.



Scheme S1



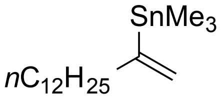
Scheme S2

References

1 E. F. Córsico and R. A. Rossi, *J. Org. Chem.*, 2004, **69**, 6427.

2 Y. Zou, L. Qin, X. Ren, Y. Lu, Y. Li and J. Zhou, *Chem. Eur. J.*, 2013, **19**, 3504.

3 H. Yoshida, Y. Takemoto and K. Takaki, *Chem. Commun.*, 2014, **50**, 8299.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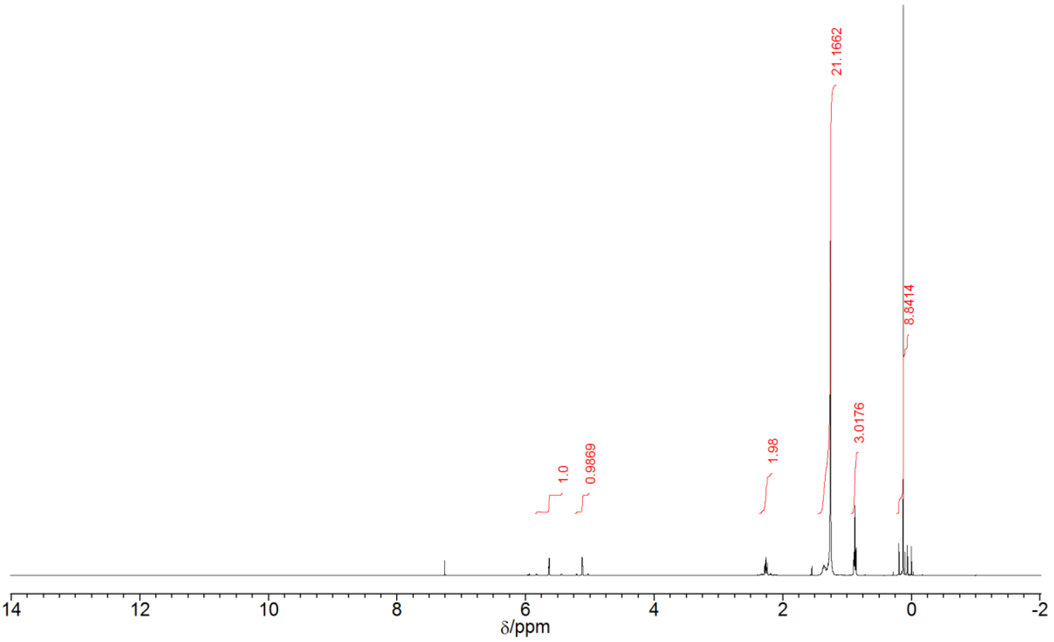
nC$_{12}$H$_{25}$

2a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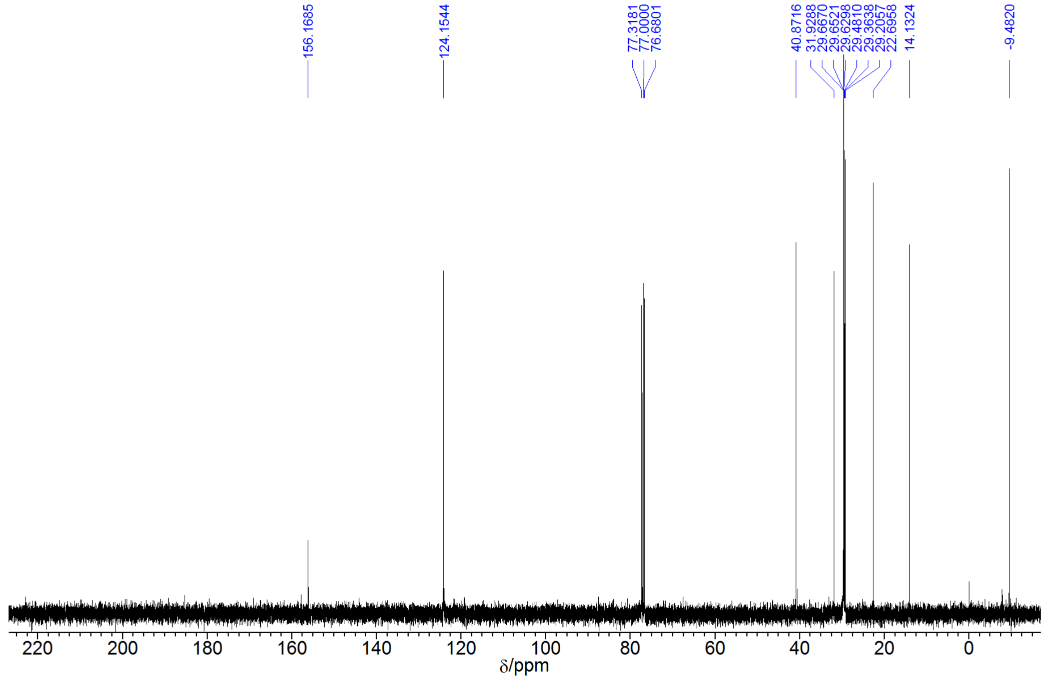
1.98
1.0
0.9869
14
12
10
8
6
4
2
0
-2
δ/ppm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29.6521
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29.3638
29.2057
22.6958
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-9.4820
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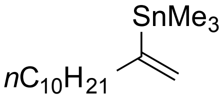
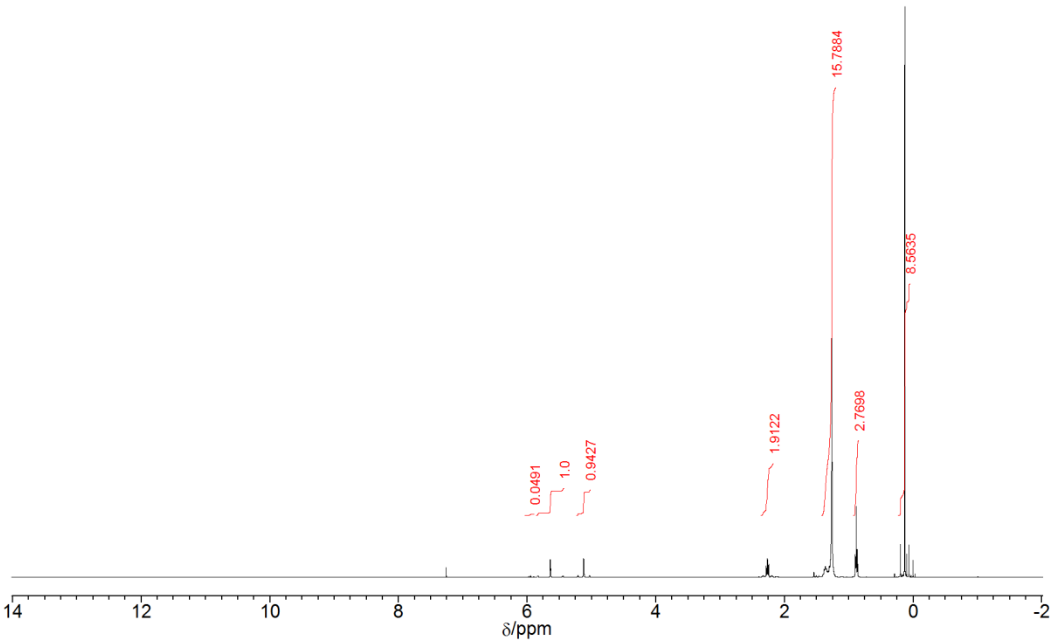
200
180
160
140
120
100
80
60
40
20
0
δ/ppm


SnMe3
nC10H21

2b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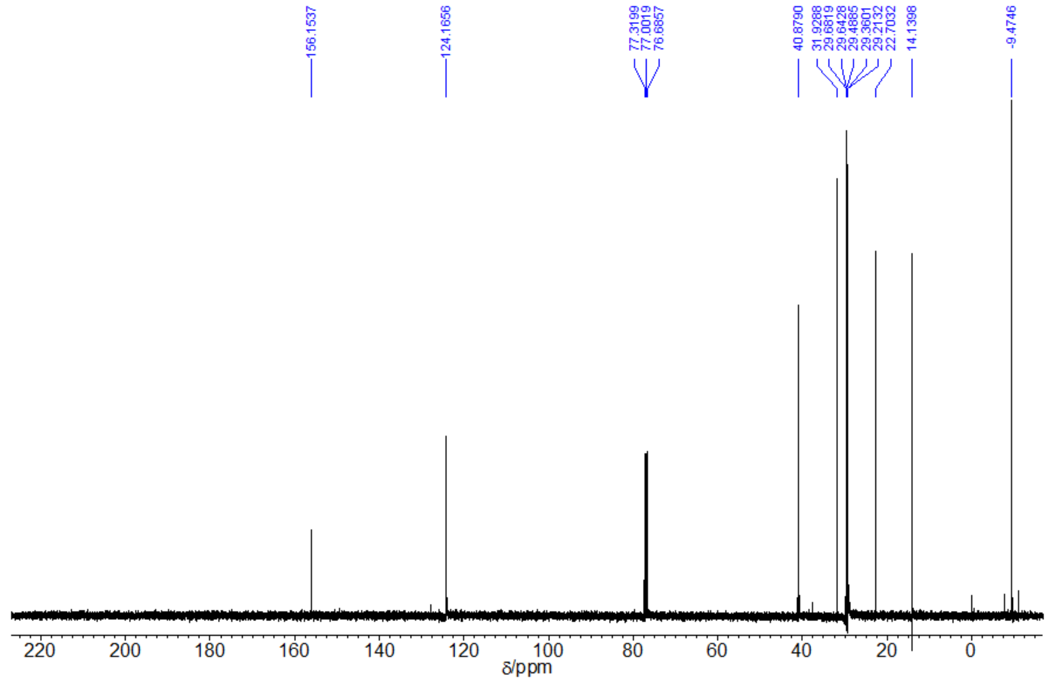
δ/ppm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29.2132
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200
180
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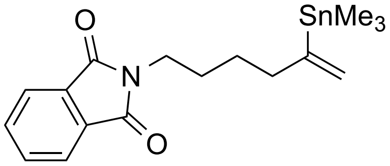
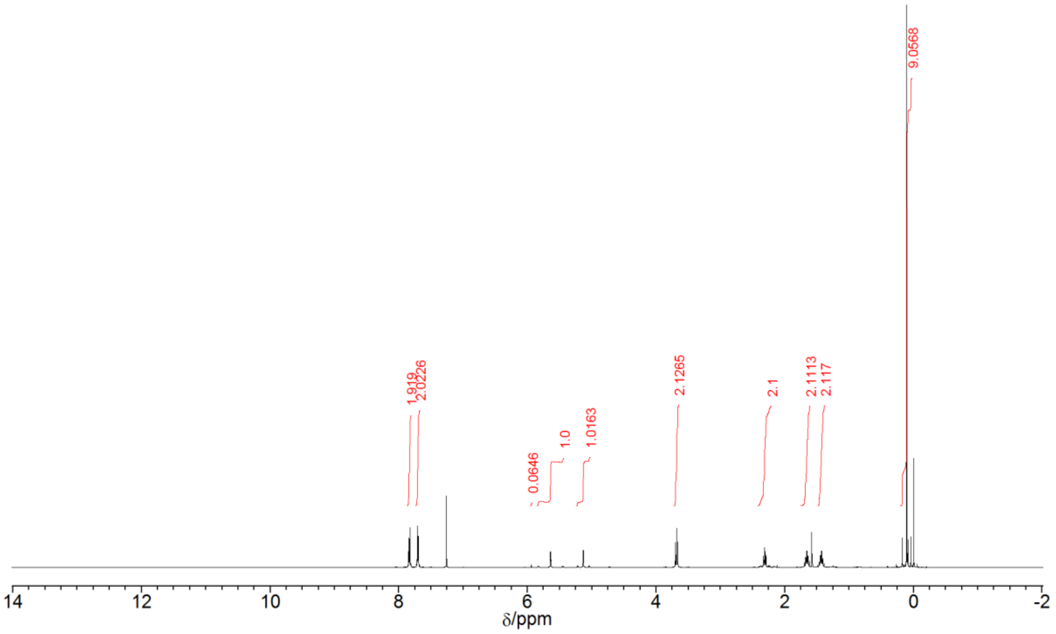
120
100
80
60
40
20
0
δ/ppm


O
N
O
SnMe3

2c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8
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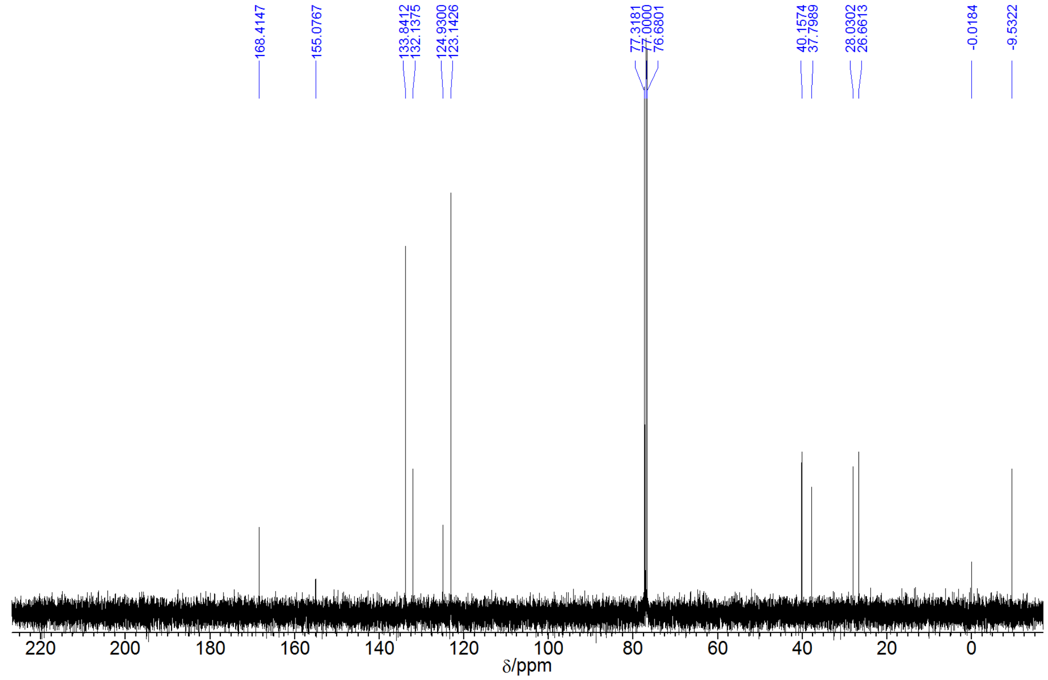
δ/ppm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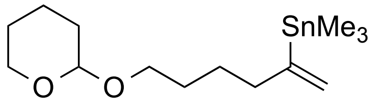
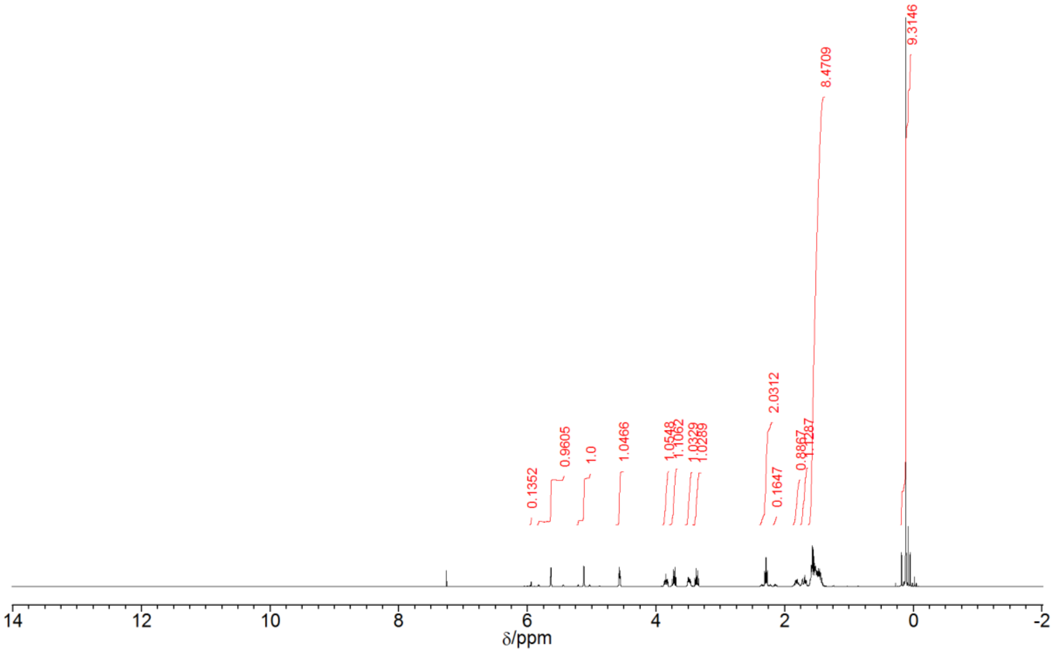
δ/ppm


SnMe3
O
O

2d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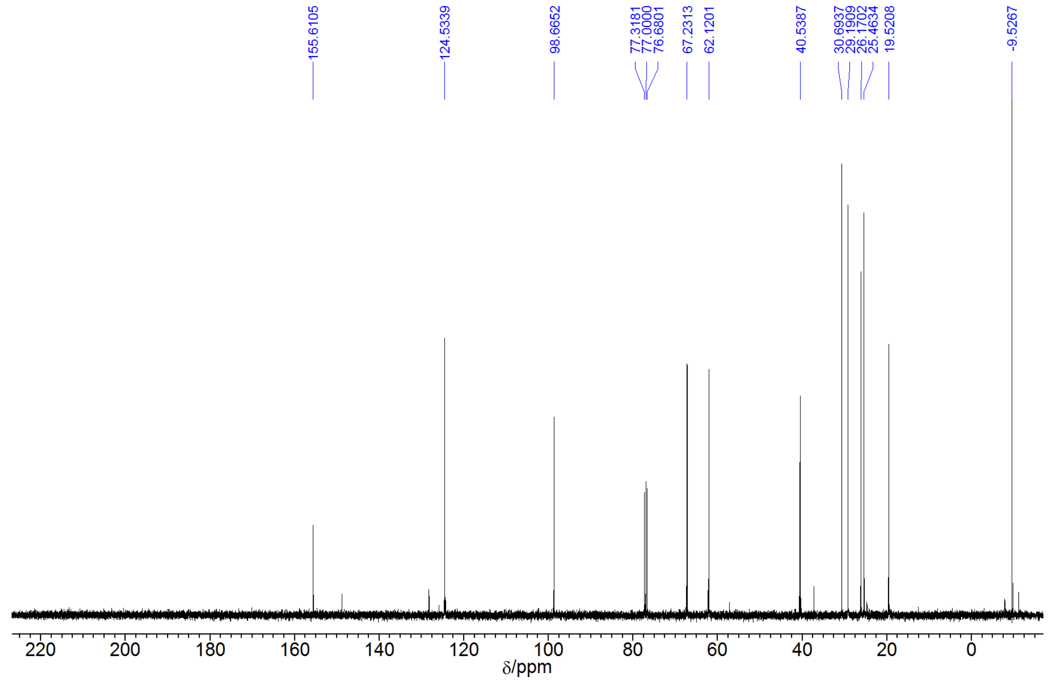
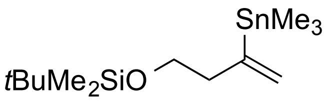
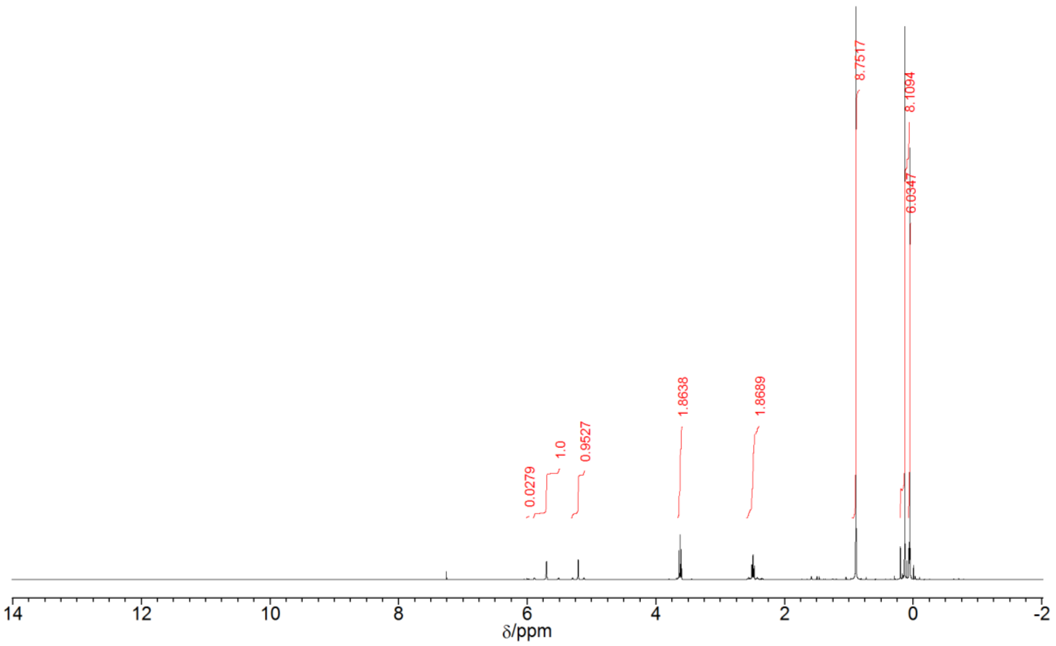
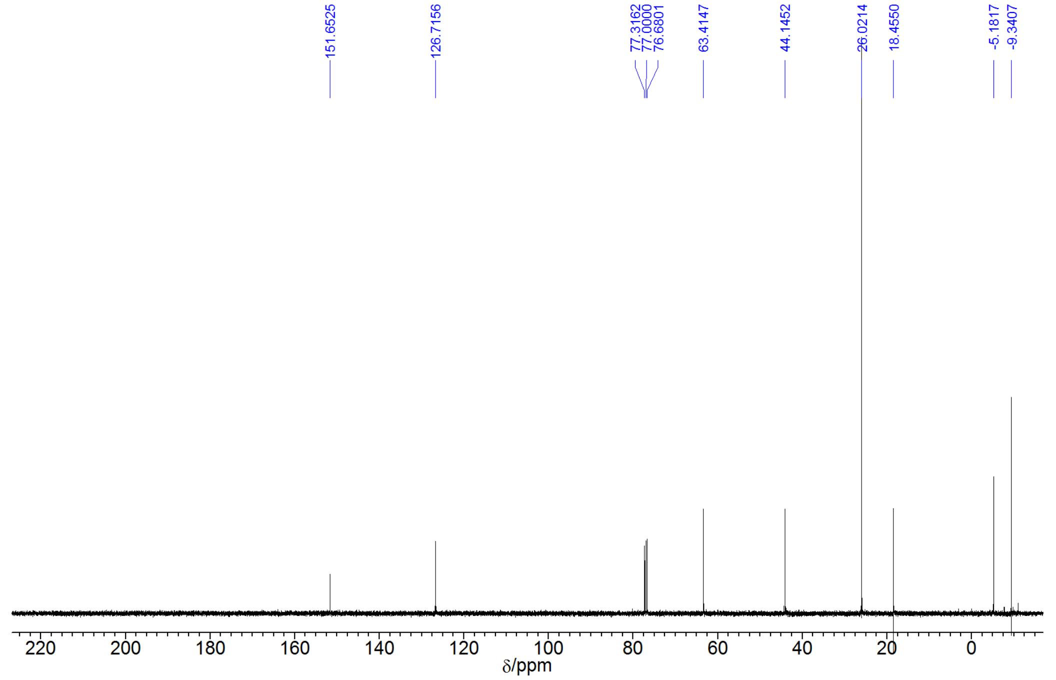
14
12
10
8
6
4
2
0
-2
δ/ppm

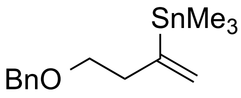

155.6105
124.5339
98.6652
77.3181
77.0000
76.6801
67.2313
62.1201
40.5387
30.6937
29.1909
26.1702
25.4634
19.5208
-9.5267
220
200
180
160
140
120
100
80
60
40
20
0
δ/ppm


SnMe$_3$
tBuMe$_2$SiO

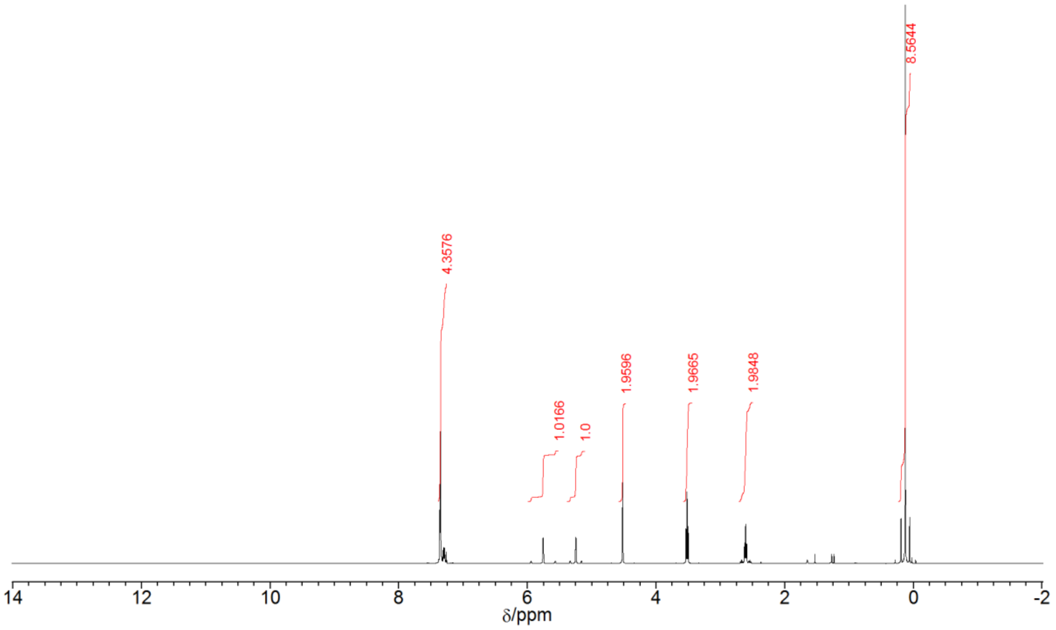
2e

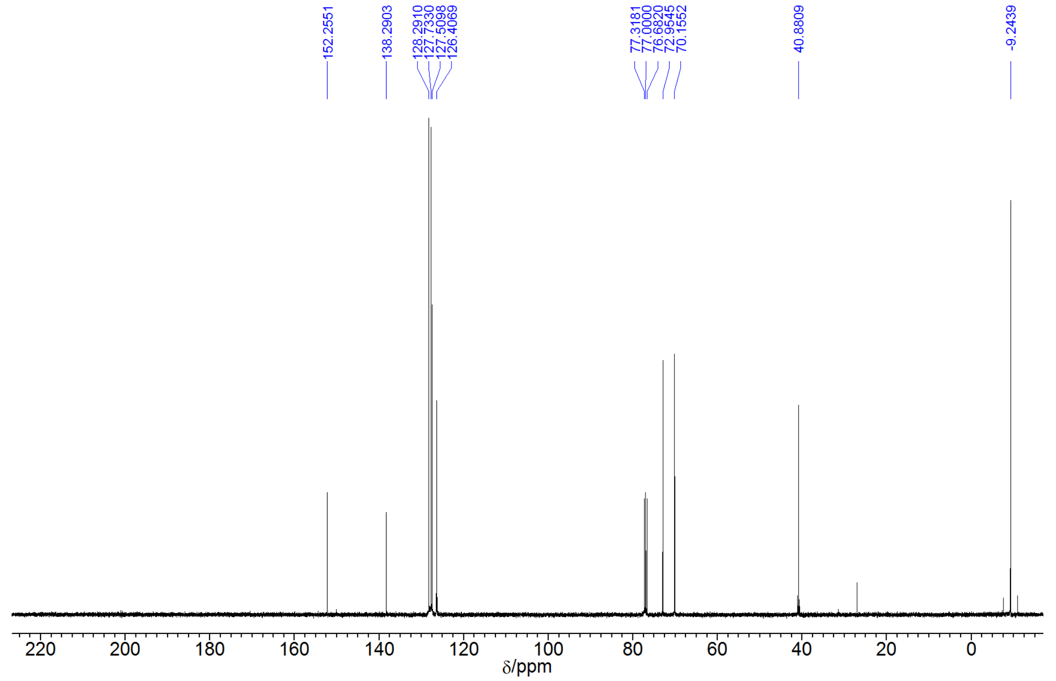

0.0279
1.0
0.9527
1.8638
1.8689
8.7517
8.1094
6.0347
14
12
10
8
6
4
2
0
-2
δ/ppm

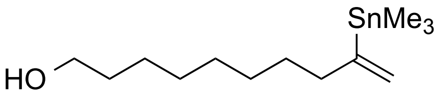

151.6525
126.7156
77.3162
77.0000
76.6801
63.4147
44.1452
26.0214
18.4550
-5.1817
-9.3407
220
200
180
160
140
120
100
80
60
40
20
0
δ/ppm


SnMe$_3$
BnO

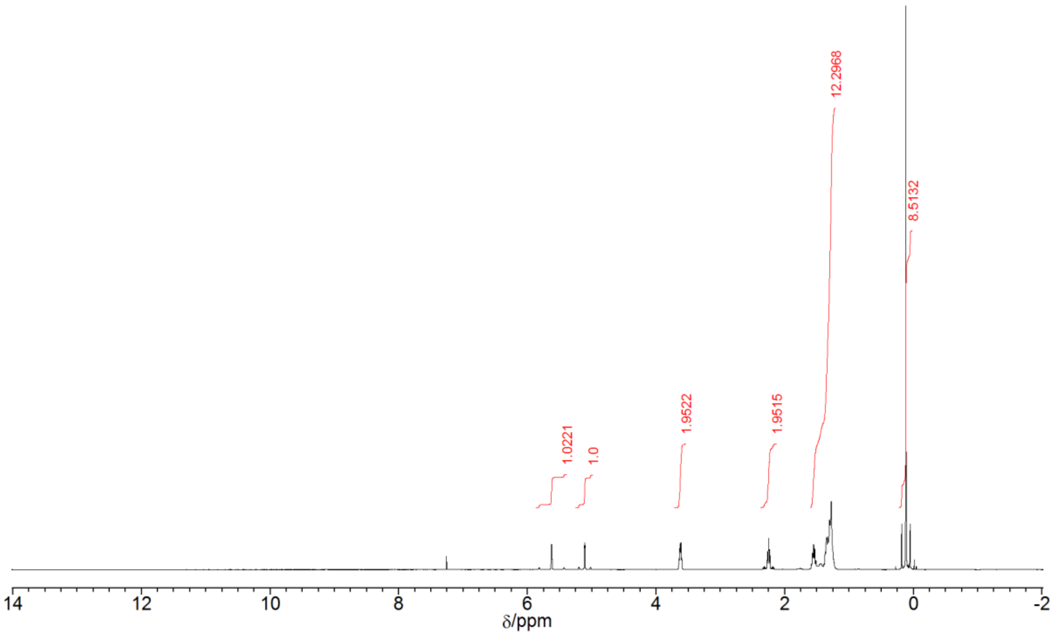
2f

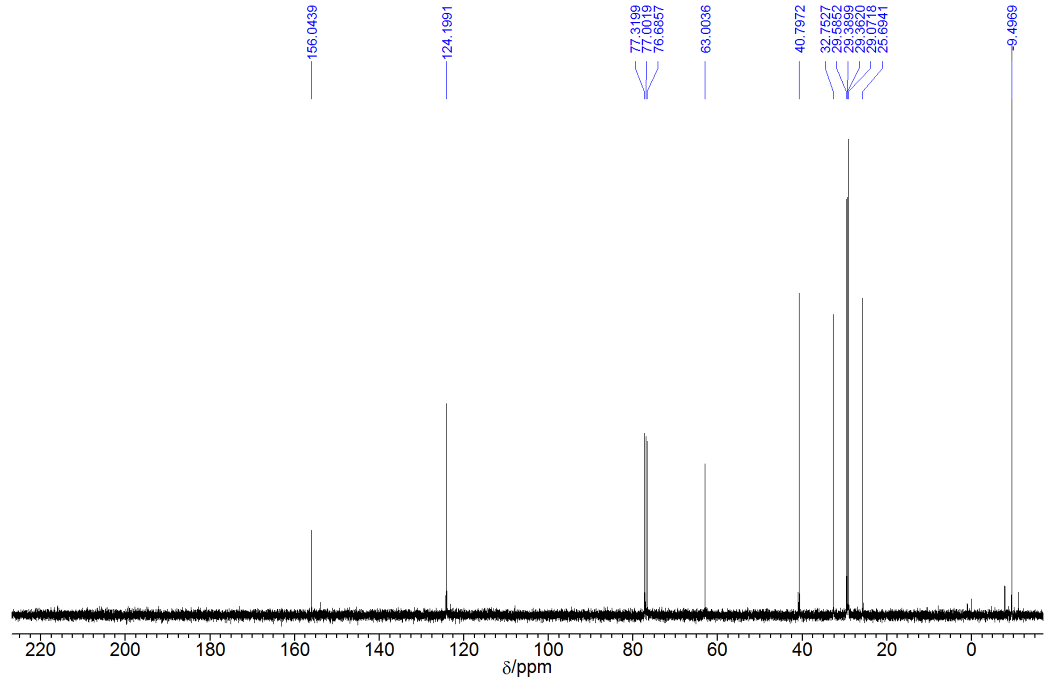

4.3576
1.0166
1.0
1.9596
1.9665
1.9848
8.5644
14
12
10
8
6
4
2
0
-2
δ/ppm

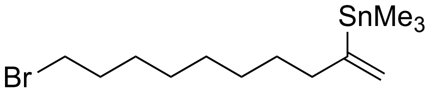

152.2551
138.2903
128.2910
127.7330
127.5098
126.4069
77.3181
77.0000
76.6820
72.9545
70.1552
40.8809
-9.2439
220
200
180
160
140
120
100
80
60
40
20
0
δ/ppm


SnMe3
HO

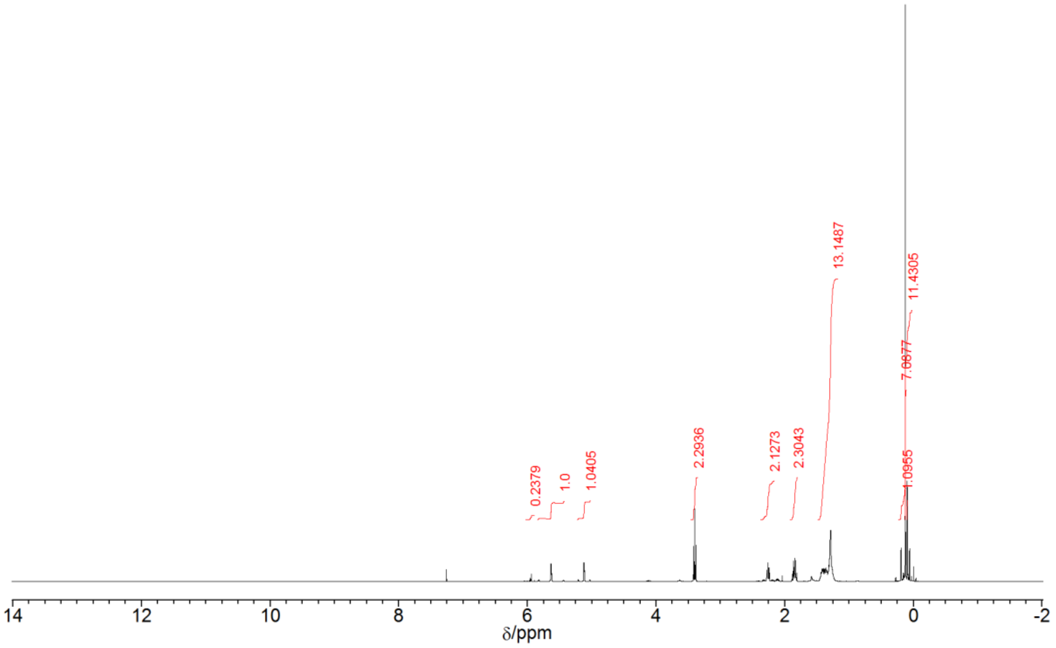
2g

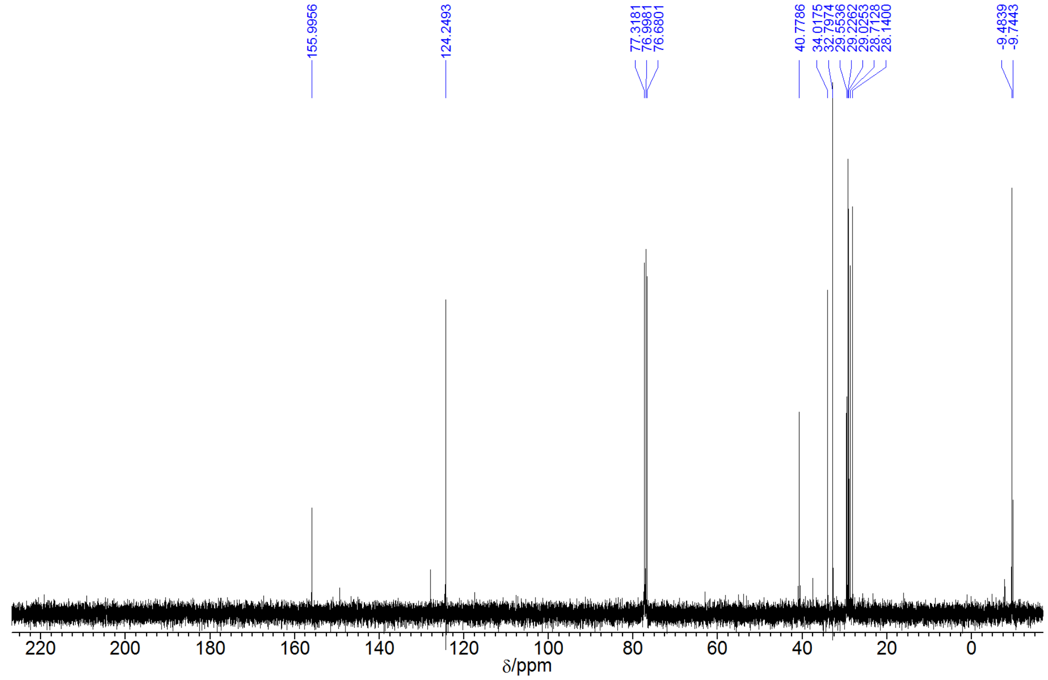

1.0221
1.0
1.9522
1.9515
12.2968
8.5132
14
12
10
8
6
4
2
0
-2
δ/ppm

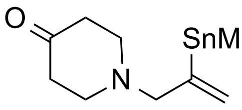

156.0439
124.1991
77.3199
77.0019
76.6857
63.0036
40.7972
32.7527
29.5852
29.3899
29.3620
29.0718
25.6941
-9.4969
220
200
180
160
140
120
100
80
60
40
20
0
δ/ppm


SnMe3
Br

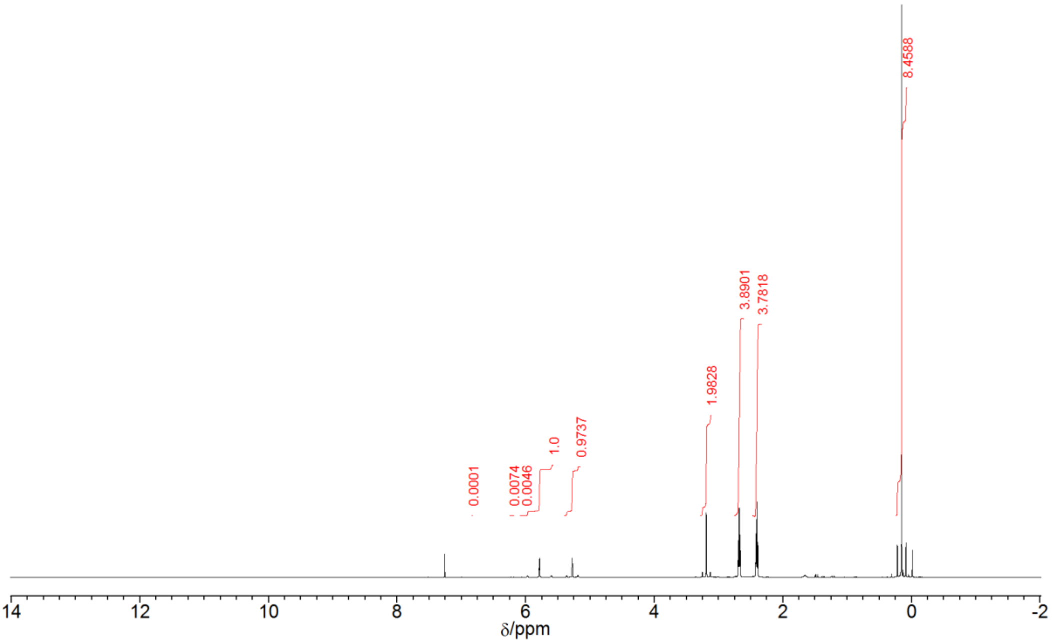
2h

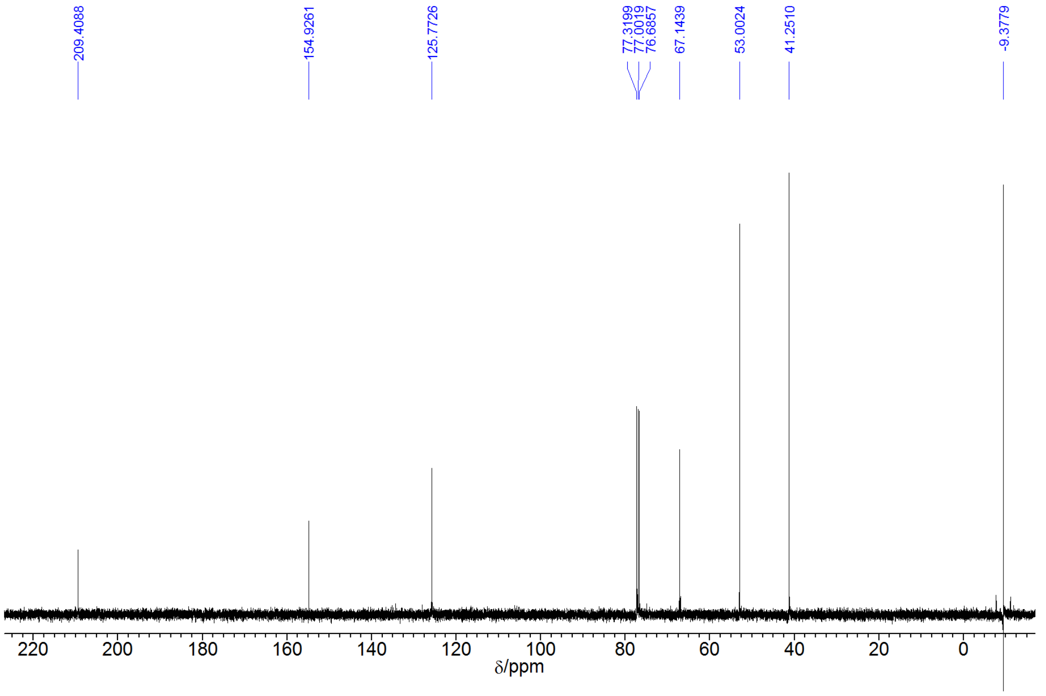

0.2379
1.0
1.0405
2.2936
2.1273
2.3043
13.1487
11.4305
7.0877
1.0955
14
12
10
8
6
4
2
0
-2
δ/ppm

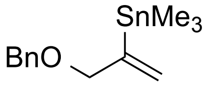

155.9956
124.2493
77.3181
76.9981
76.6801
40.7786
34.0175
32.7974
29.5536
29.2262
29.0253
28.7128
28.1400
-9.4839
-9.7443
220
200
180
160
140
120
100
80
60
40
20
0
δ/ppm


O
N
SnMe3

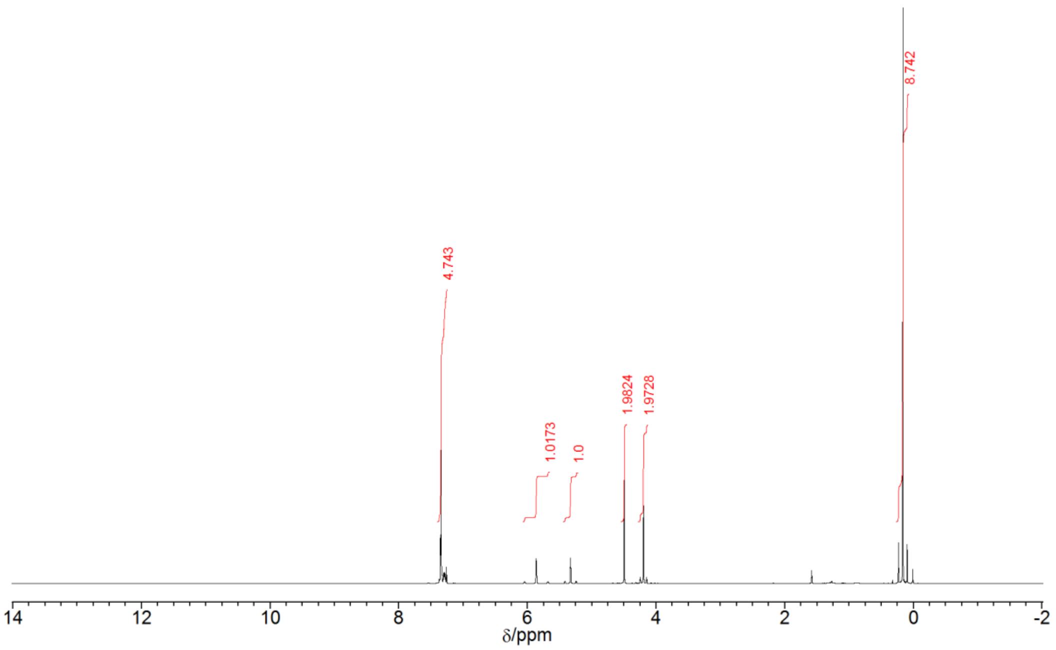
2i

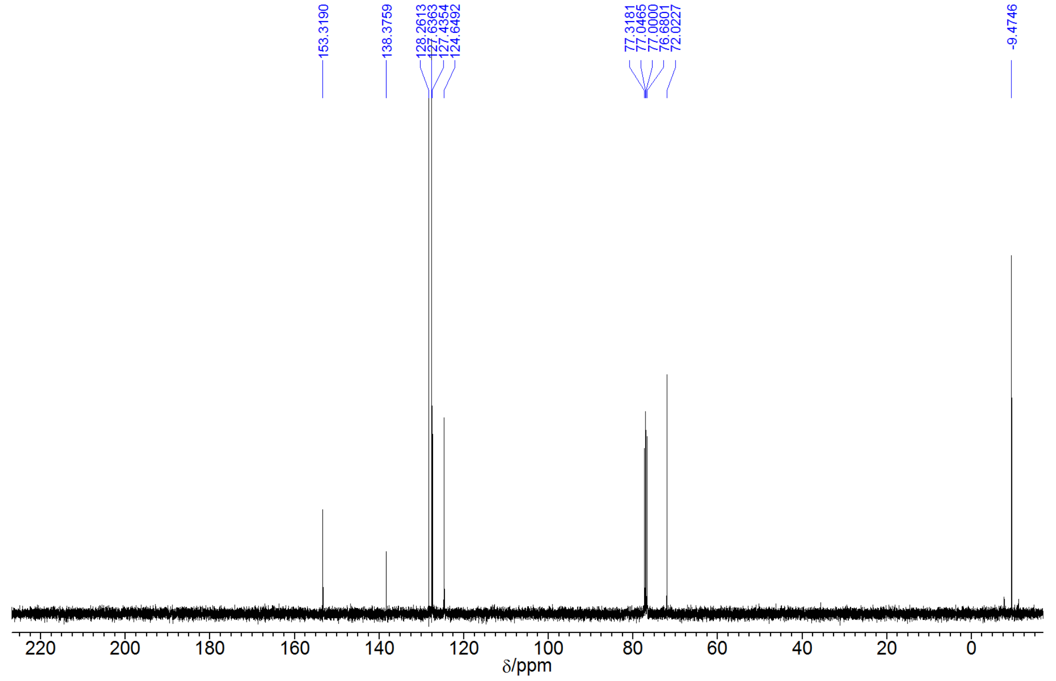

8.4588
3.8901
3.7818
1.9828
0.9737
1.0
0.0074
0.0046
0.0001
14
12
10
8
6
4
2
0
-2
δ/ppm

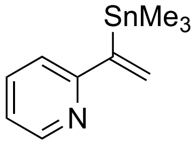

209.4088
154.9261
125.7726
77.3199
77.0019
76.6857
67.1439
53.0024
41.2510
-9.3779
220
200
180
160
140
120
100
80
60
40
20
0
δ/ppm


SnMe3
BnO

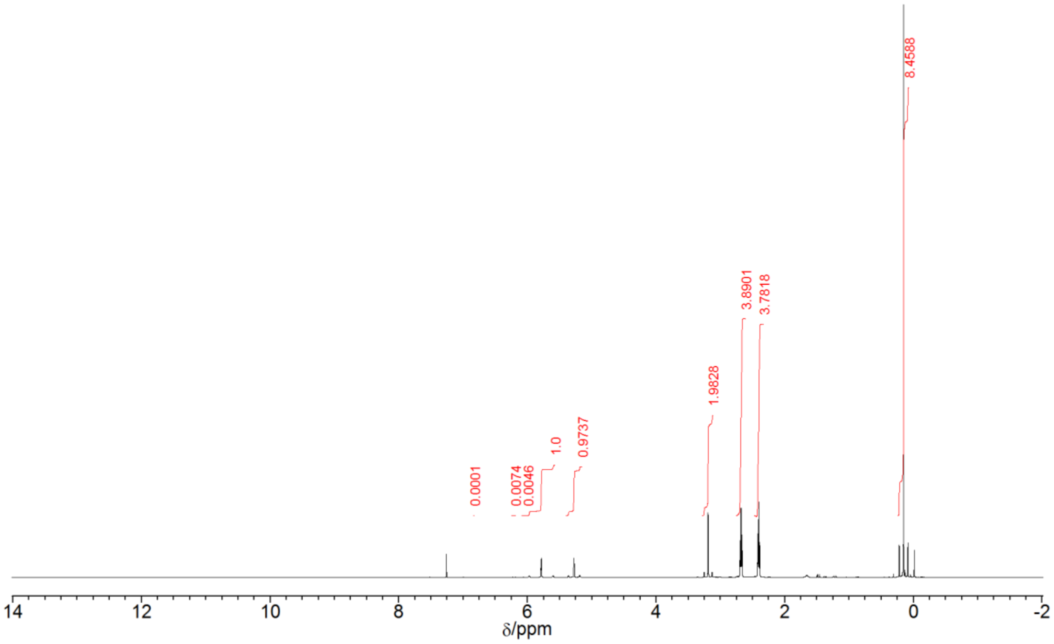
2j

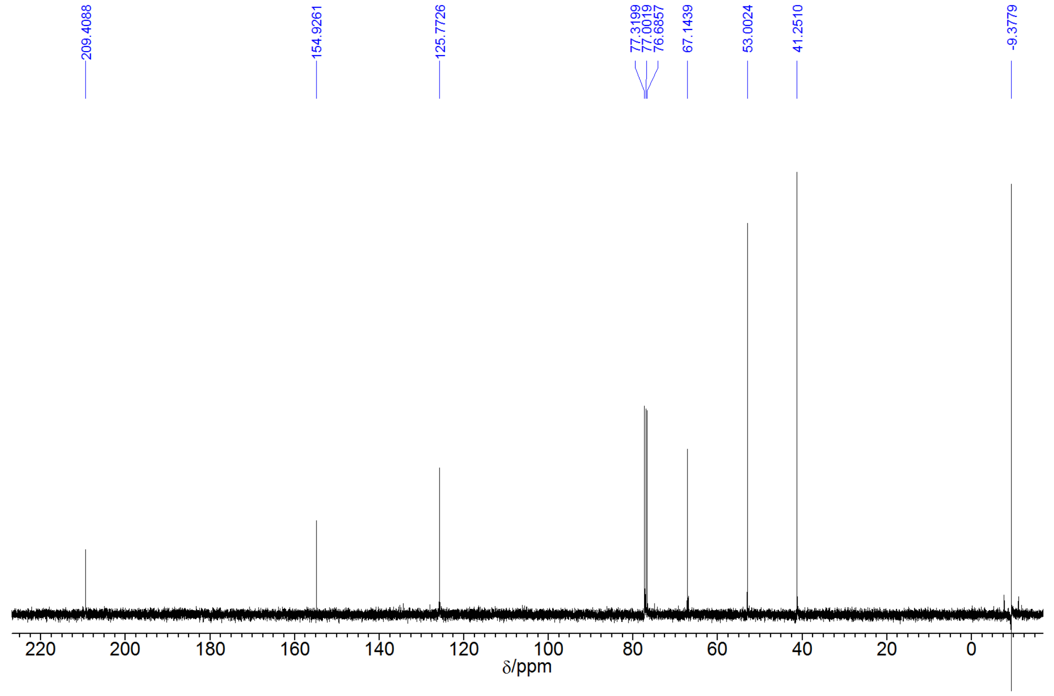

4.743
1.0173
1.0
1.9824
1.9728
8.742
14
12
10
8
6
4
2
0
-2
δ/ppm

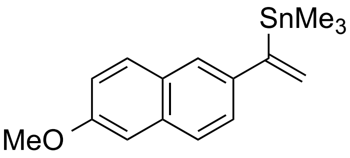

153.3190
138.3759
128.2613
127.6363
127.4354
124.6492
77.3181
77.0465
77.0000
76.6801
72.0227
-9.4746
220
200
180
160
140
120
100
80
60
40
20
0
δ/ppm


SnMe3
N

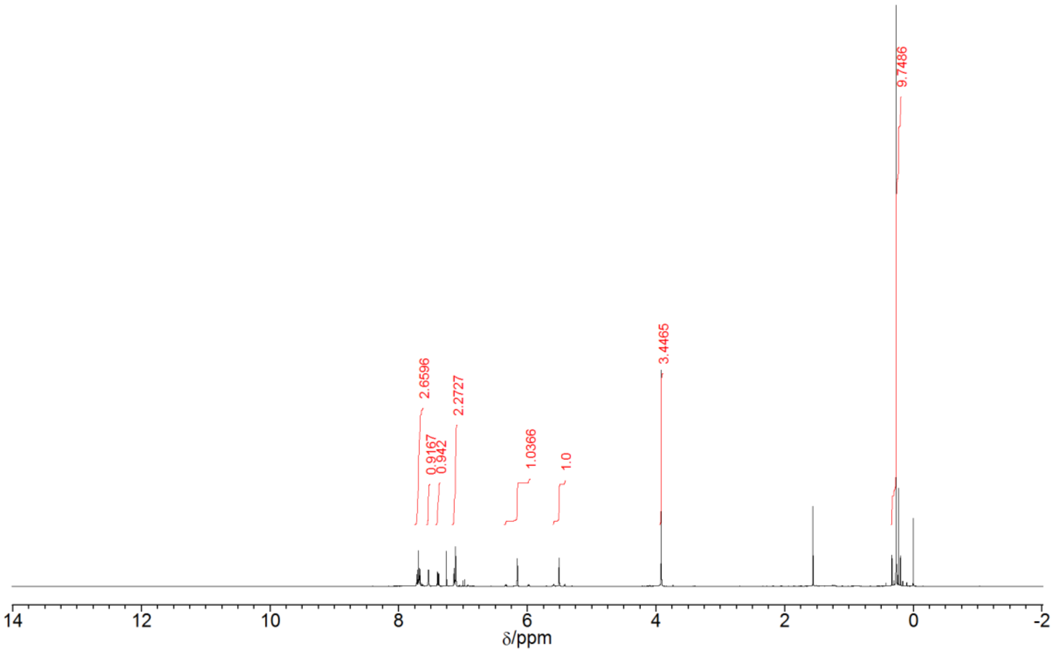
2k


0.0001
0.0074
0.0046
1.0
0.9737
1.9828
3.8901
3.7818
8.4588
14
12
10
8
6
4
2
0
-2
δ/ppm


209.4088
154.9261
125.7726
77.3199
77.0019
76.6857
67.1439
53.0024
41.2510
-9.3779
220
200
180
160
140
120
100
80
60
40
20
0
δ/ppm


SnMe3
MeO

2l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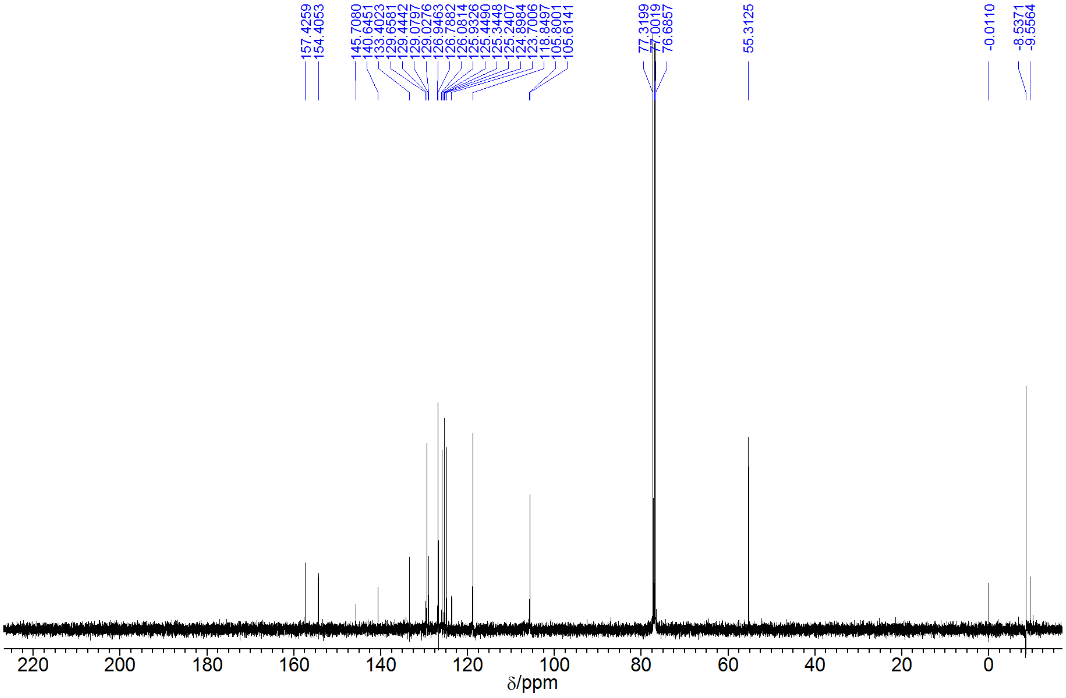
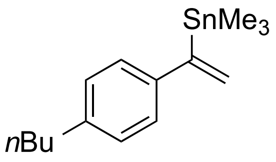
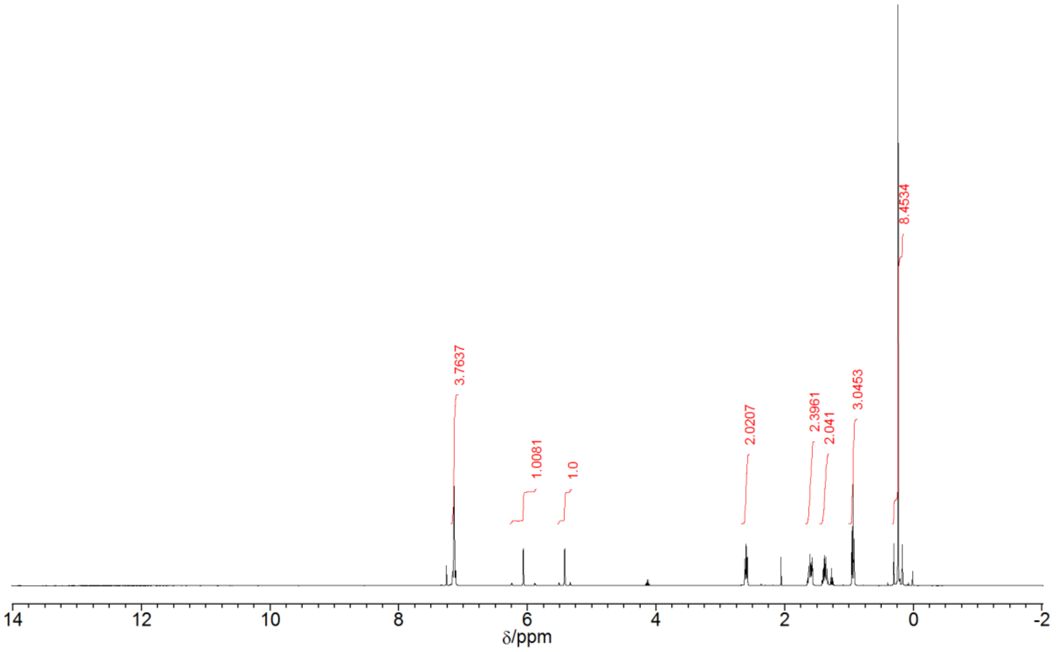
2.6596
0.9167
0.942
2.2727
1.0366
1.0
14
12
10
8
6
4
2
0
-2
δ/ppm


157.4259
154.4053
145.7080
140.6451
133.4023
129.6581
129.4442
129.0797
129.0276
126.9463
126.7882
126.0814
125.9326
125.4490
125.3448
125.2407
124.8984
123.7006
118.8497
105.8001
105.6141
77.3199
77.0019
76.6857
55.3125
-0.0110
-8.5371
-9.5564
220
200
180
160
140
120
100
80
60
40
20
0
δ/ppm


SnMe3
nBu

2m


3.7637
1.0081
1.0
2.0207
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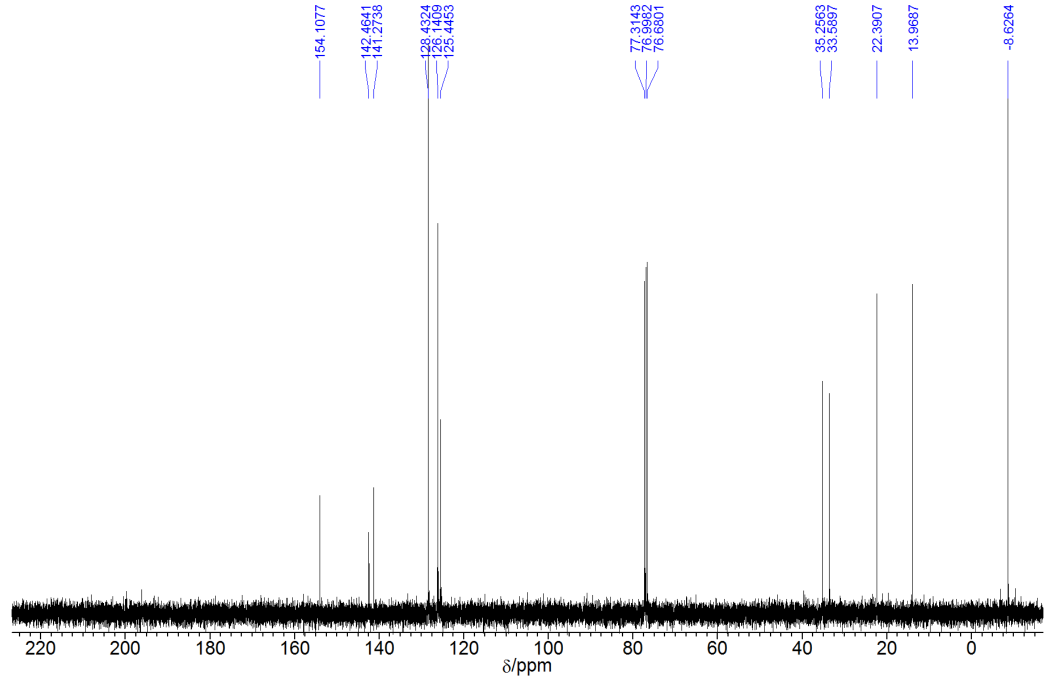
2.041
3.0453
8.4534
14
12
10
8
6
4
2
0
-2
δ/ppm


154.1077
142.4641
141.2738
128.4324
126.1409
125.4453
77.3143
76.9982
76.6801
35.2563
33.5897
22.3907
13.9687
-8.6264
220
200
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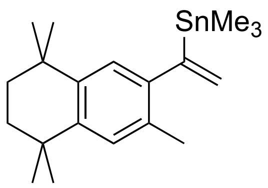
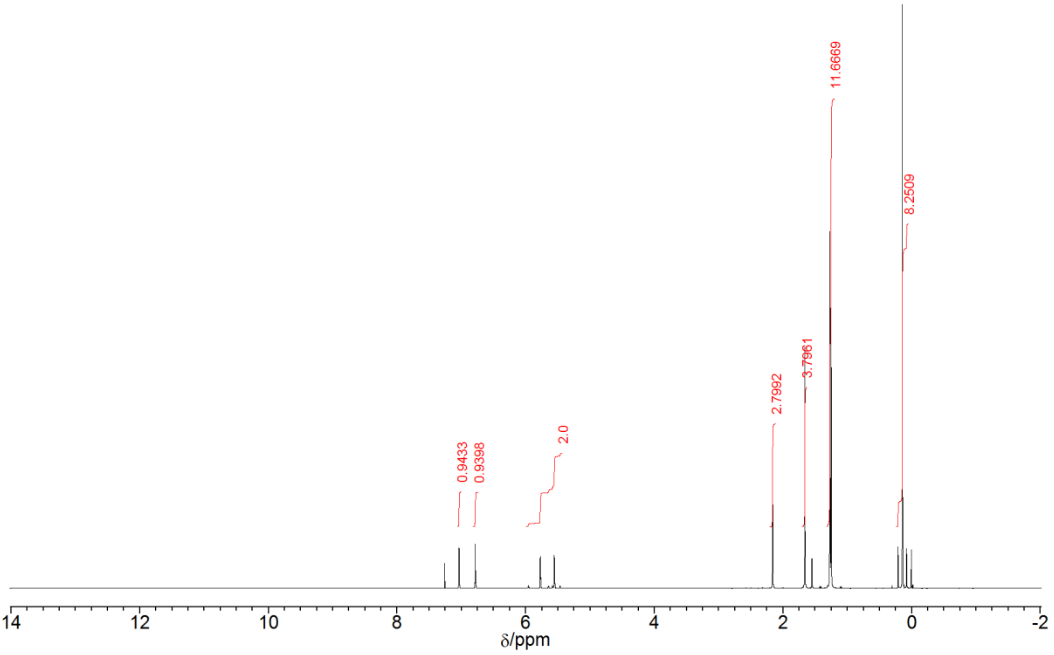
160
140
120
100
80
60
40
20
0
δ/ppm


SnMe3

2n


0.9433
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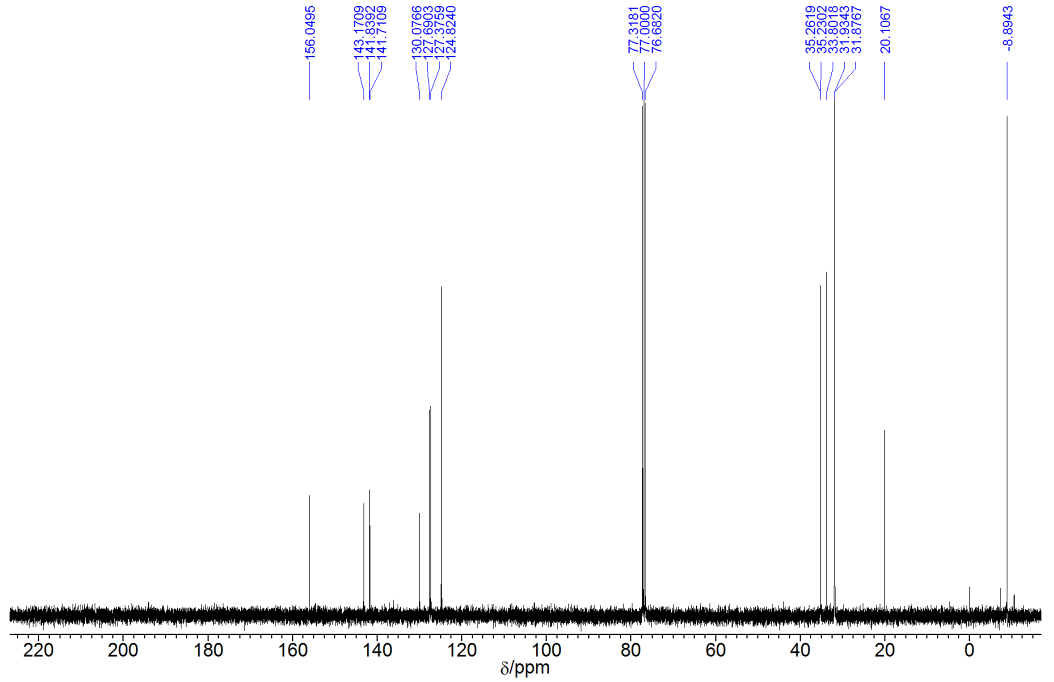
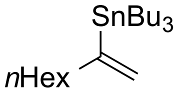
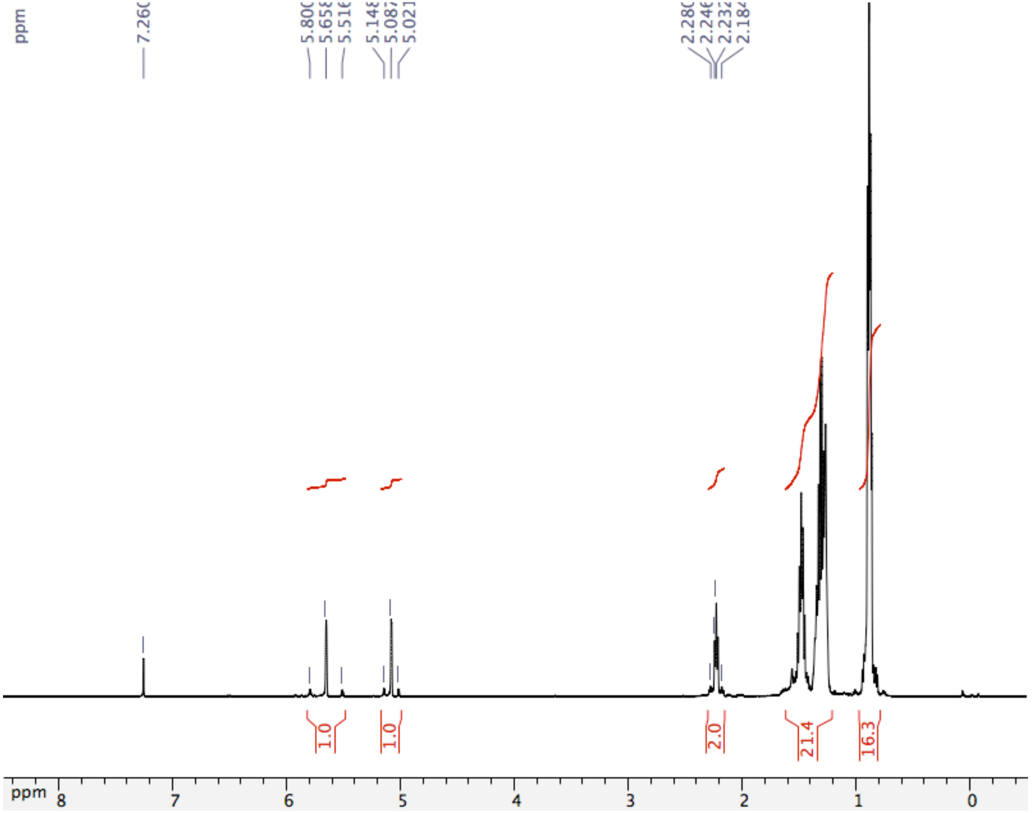
2.7992
3.7961
11.6669
8.2509
14
12
10
8
6
4
2
0
-2
δ/ppm


156.0495
143.1709
141.8392
141.7109
130.0766
127.6903
127.3759
124.8240
77.3181
77.0000
76.6820
35.2619
35.2302
33.8018
31.9343
31.8767
20.1067
-8.8943
220
200
180
160
140
120
100
80
60
40
20
0
δ/ppm


SnBu$_3$
nHex

3p


ppm
7.26
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5.65
5.51
5.14
5.08
5.02
2.28
2.24
2.23
2.18
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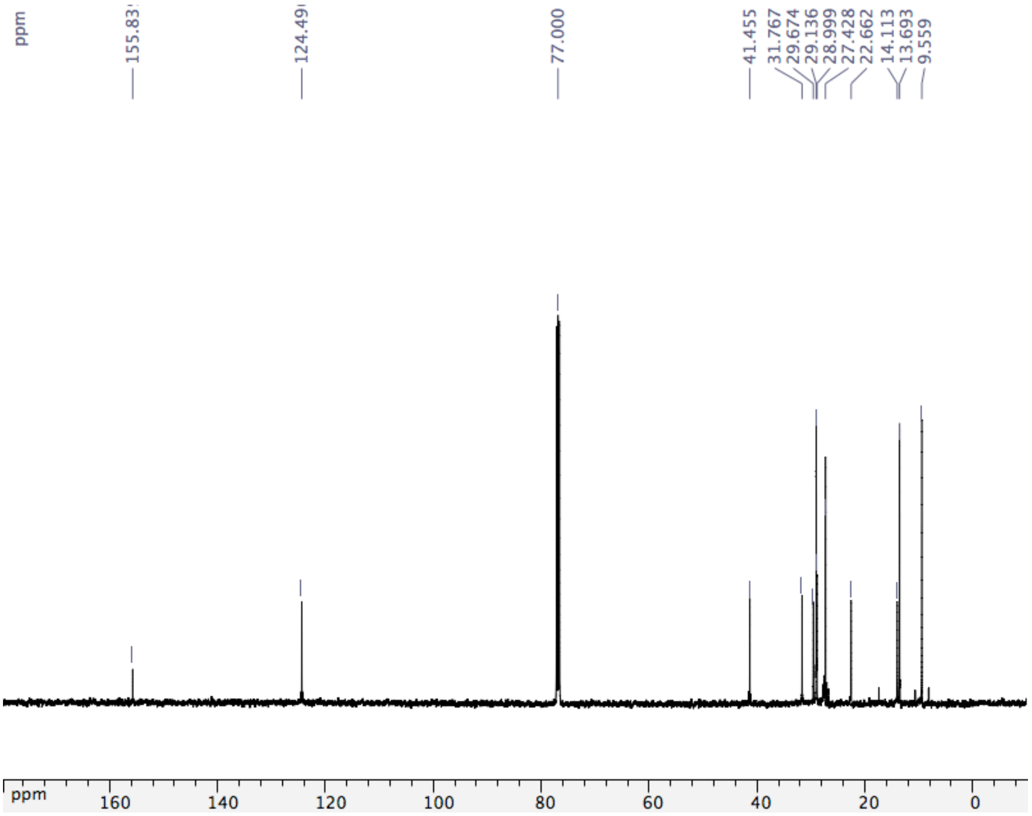
5
4
3
2
1
0


ppm
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41.455
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29.674
29.136
28.999
27.428
22.662
14.113
13.693
9.559
ppm
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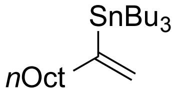
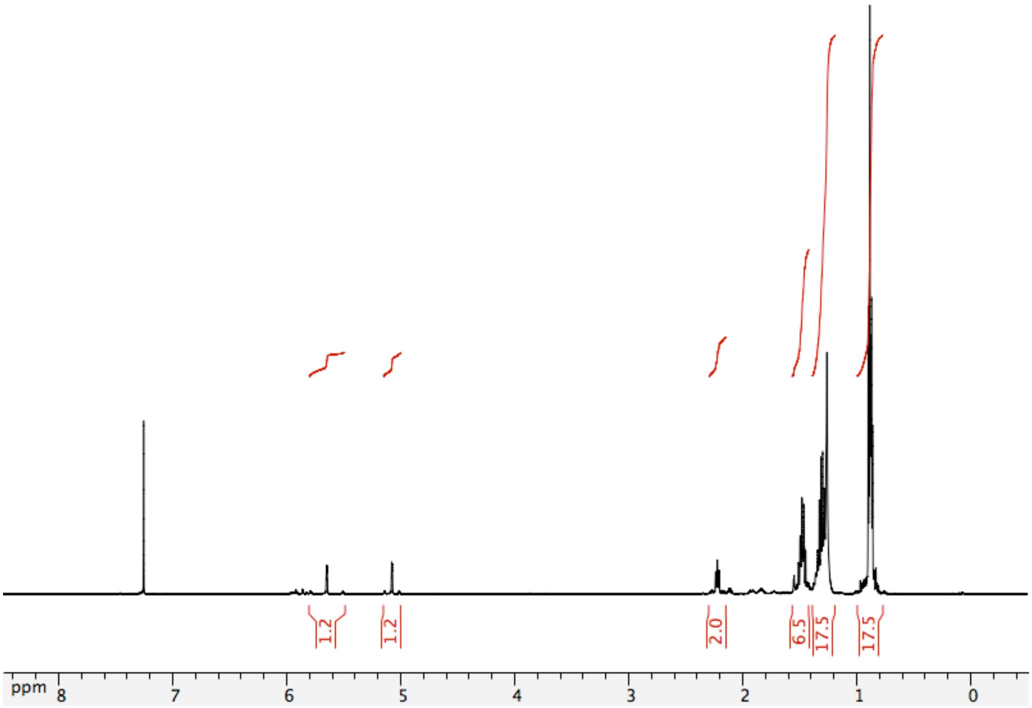
140
120
100
80
60
40
20
0


SnBu3
nOct

3q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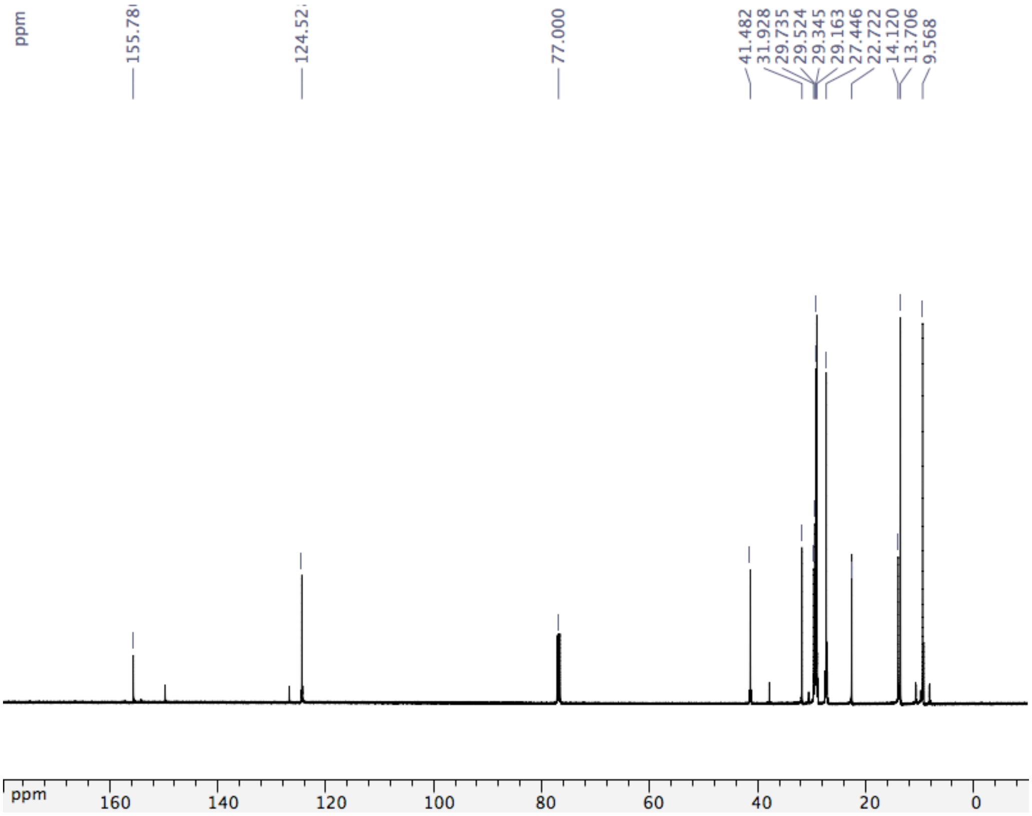
1.2
2.0
6.5
17.5
17.5
ppm
8
7
6
5
4
3
2
1
0


ppm
155.78
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77.000
41.482
31.928
29.735
29.524
29.345
29.163
27.446
22.722
14.120
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9.568
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140
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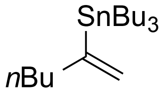
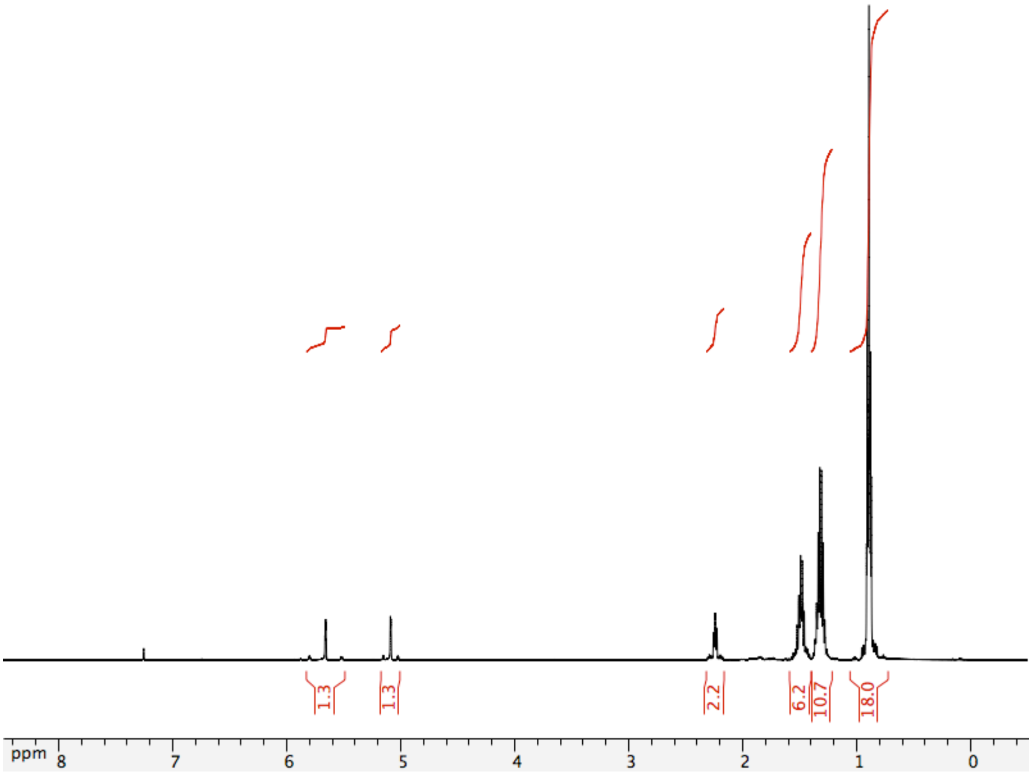
100
80
60
40
20
0


SnBu3
nBu

3r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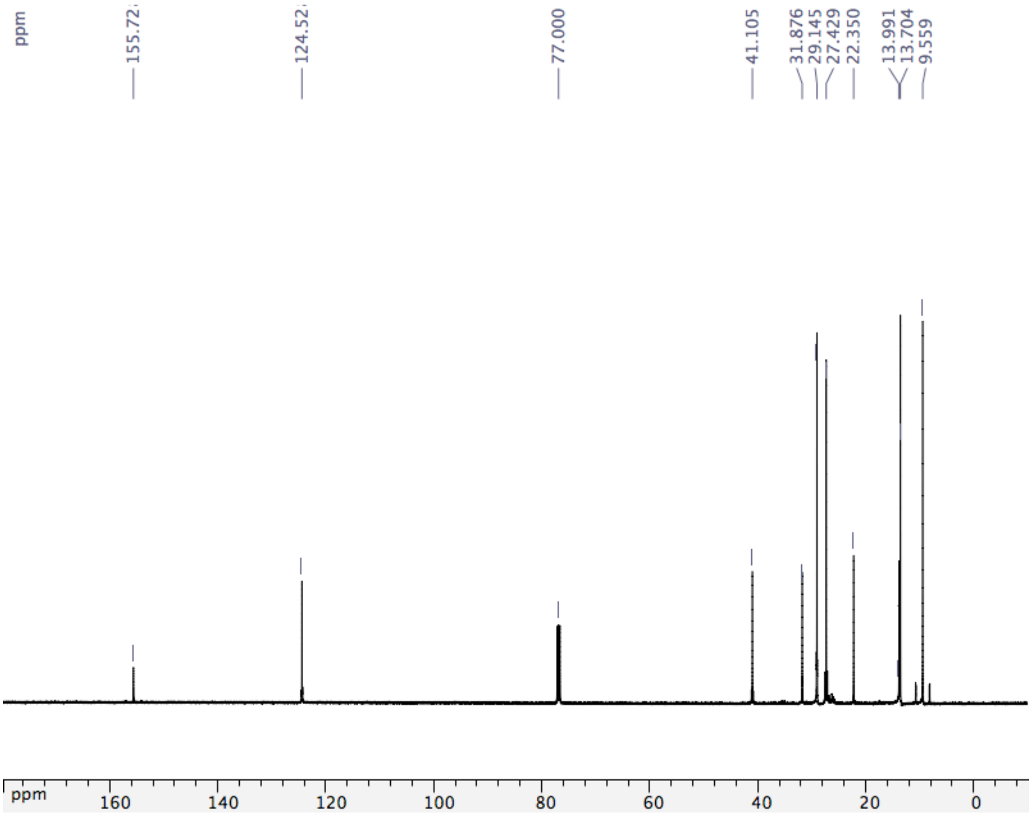
1.3
2.2
6.2
10.7
18.0
ppm
8
7
6
5
4
3
2
1
0


ppm
155.72
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77.000
41.105
31.876
29.145
27.429
22.350
13.991
13.704
9.559
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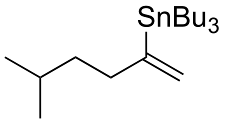
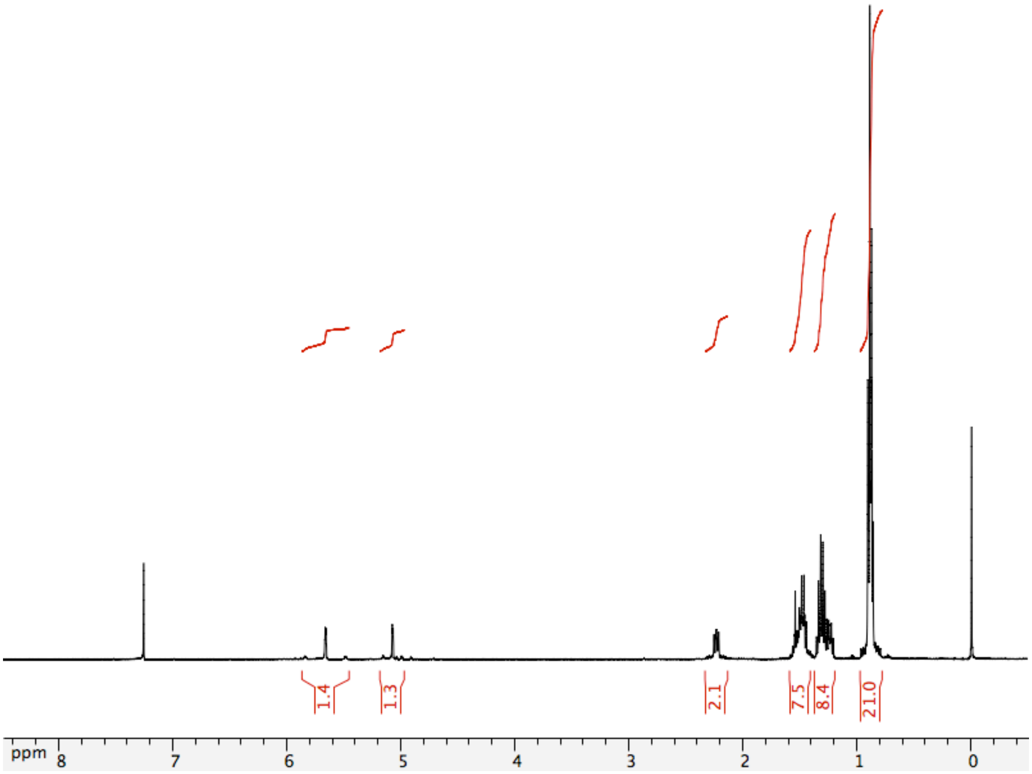
100
80
60
40
20
0


SnBu$_3$

3s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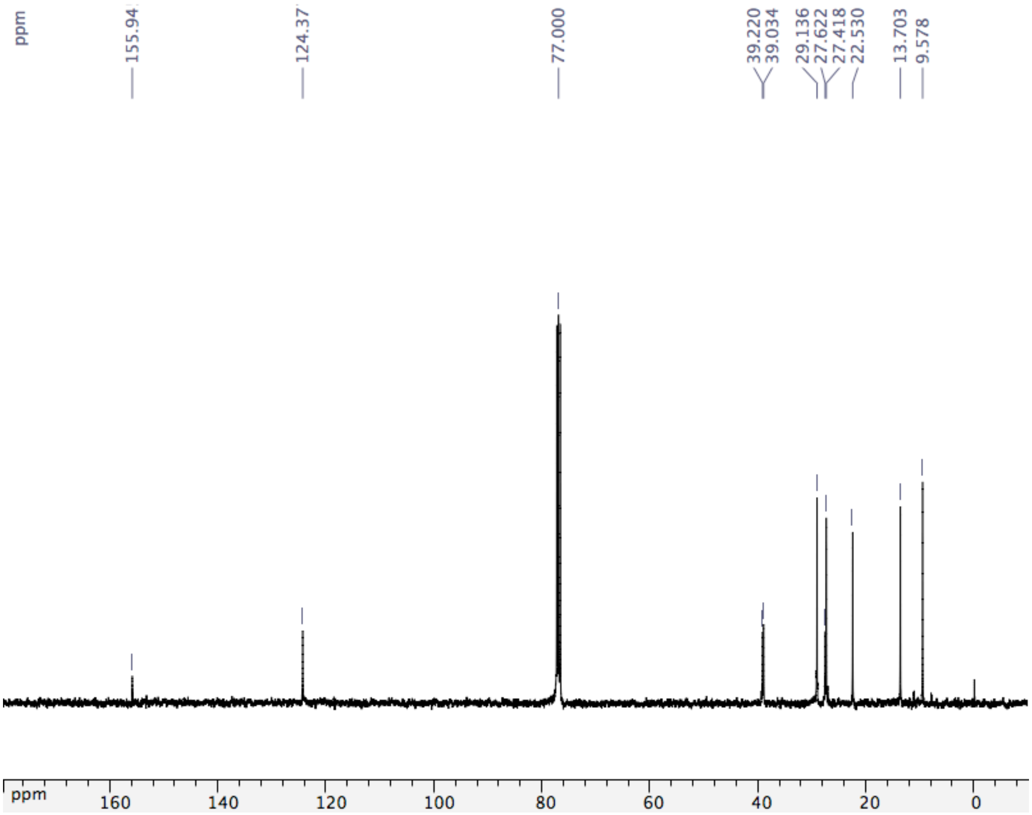
8.4
21.0
ppm
8
7
6
5
4
3
2
1
0


ppm
155.94
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77.000
39.220
39.034
29.136
27.622
27.418
22.530
13.703
9.578
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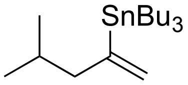
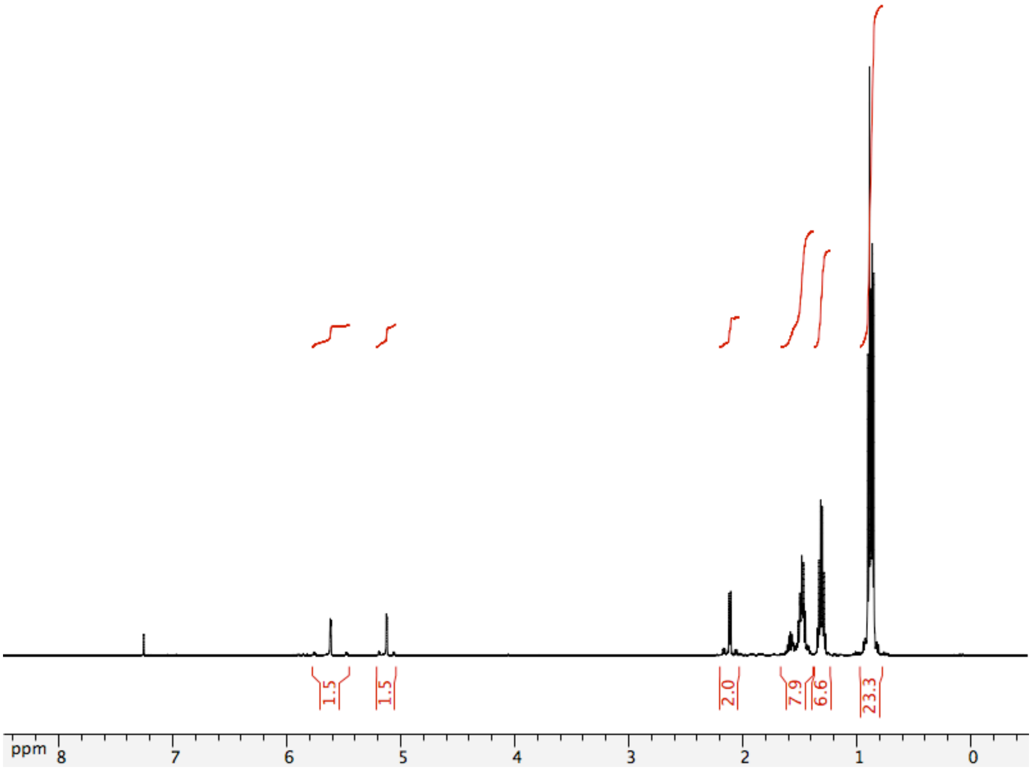
100
80
60
40
20
0


SnBu$_3$

3t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1.5
2.0
7.9
6.6
23.3
ppm
8
7
6
5
4
3
2
1
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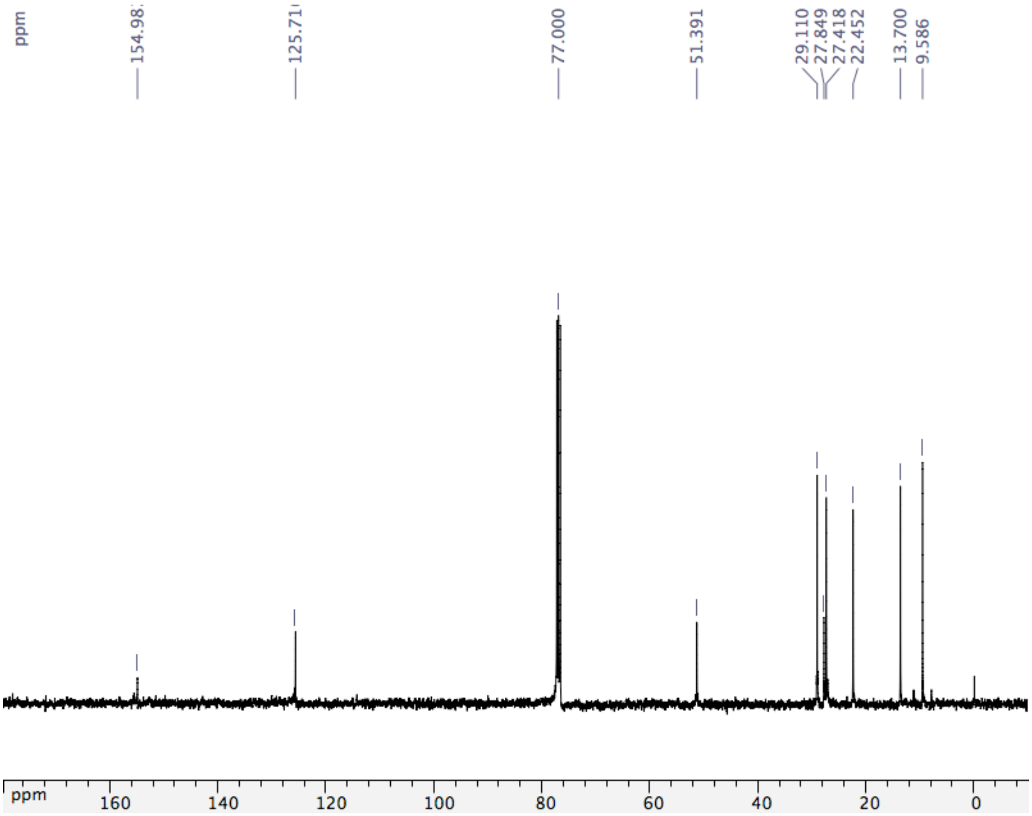
ppm
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77.000
51.391
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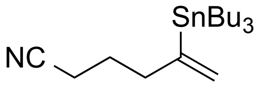
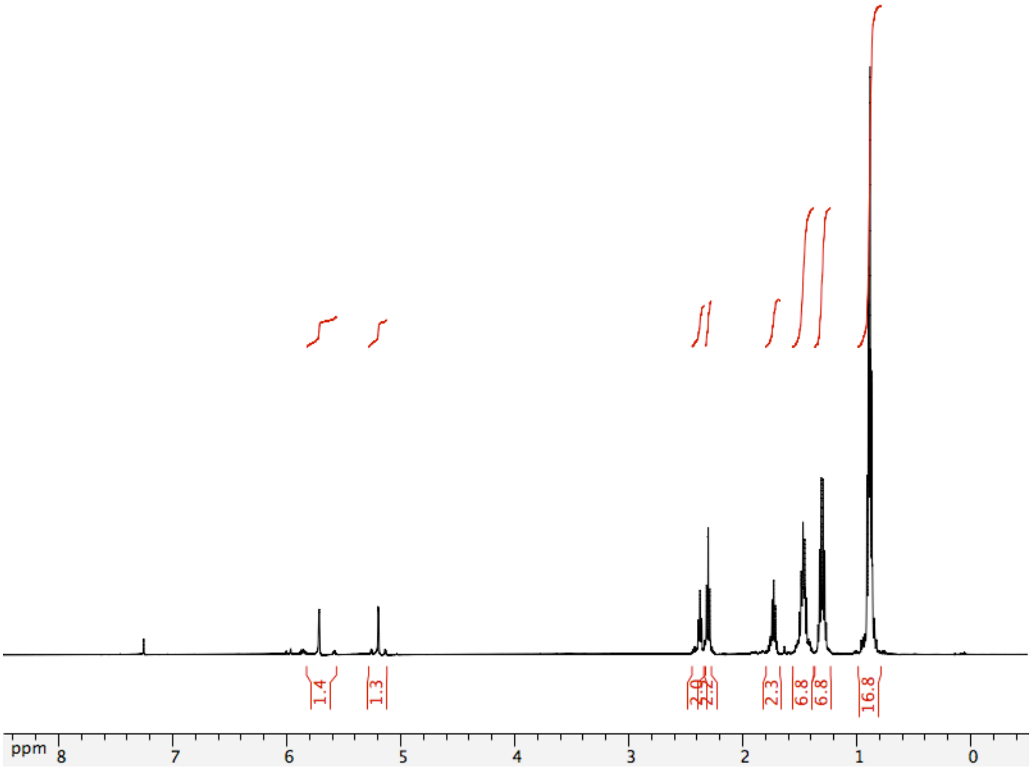
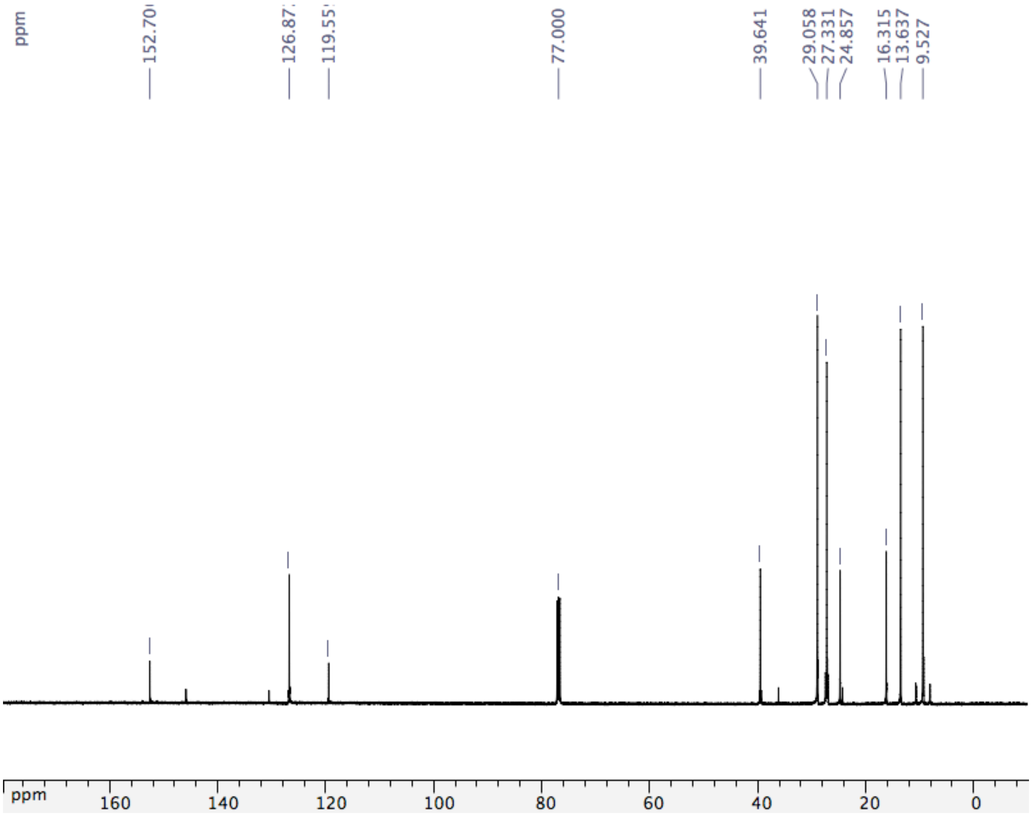
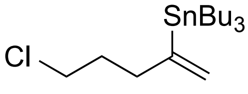
27.849
27.418
22.452
13.700
9.586
ppm
160
140
120
100
80
60
40
20
0


SnBu3
NC

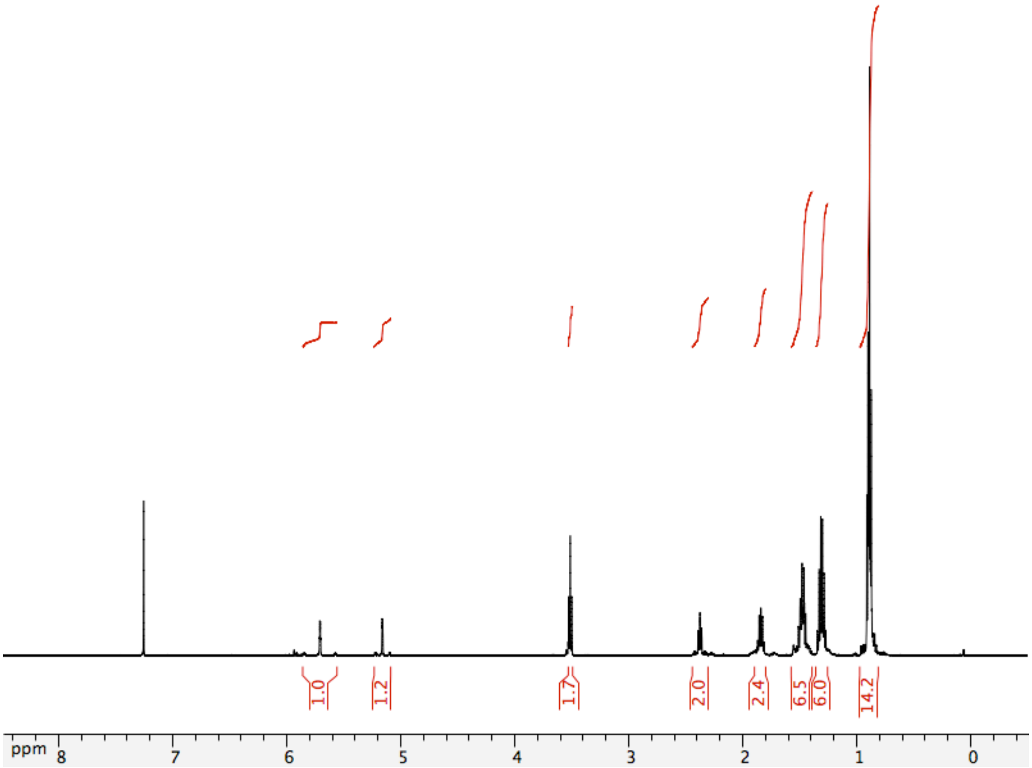
3u


1.4
1.3
2.0
2.2
2.3
6.8
6.8
16.8
ppm
8
7
6
5
4
3
2
1
0


ppm
152.70
126.87
119.55
77.000
39.641
29.058
27.331
24.857
16.315
13.637
9.527
ppm
160
140
120
100
80
60
40
20
0


SnBu3
Cl

3v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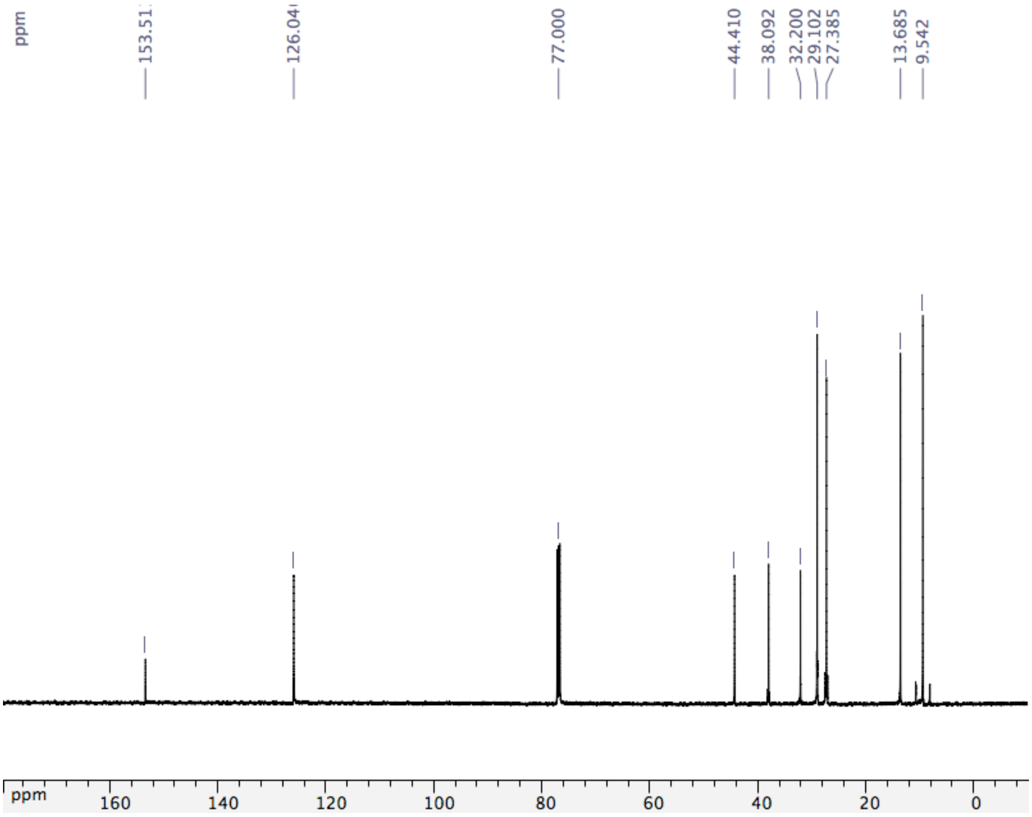
1.2
1.7
2.0
2.4
6.5
6.0
14.2
ppm
8
7
6
5
4
3
2
1
0


ppm
153.51
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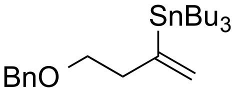
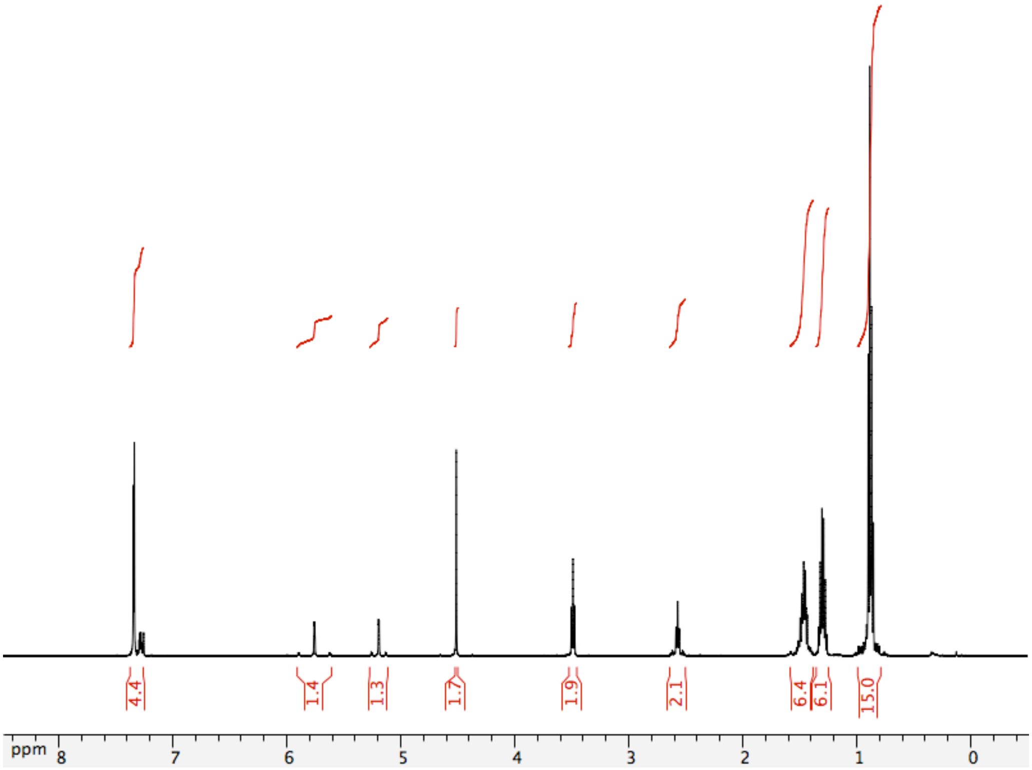
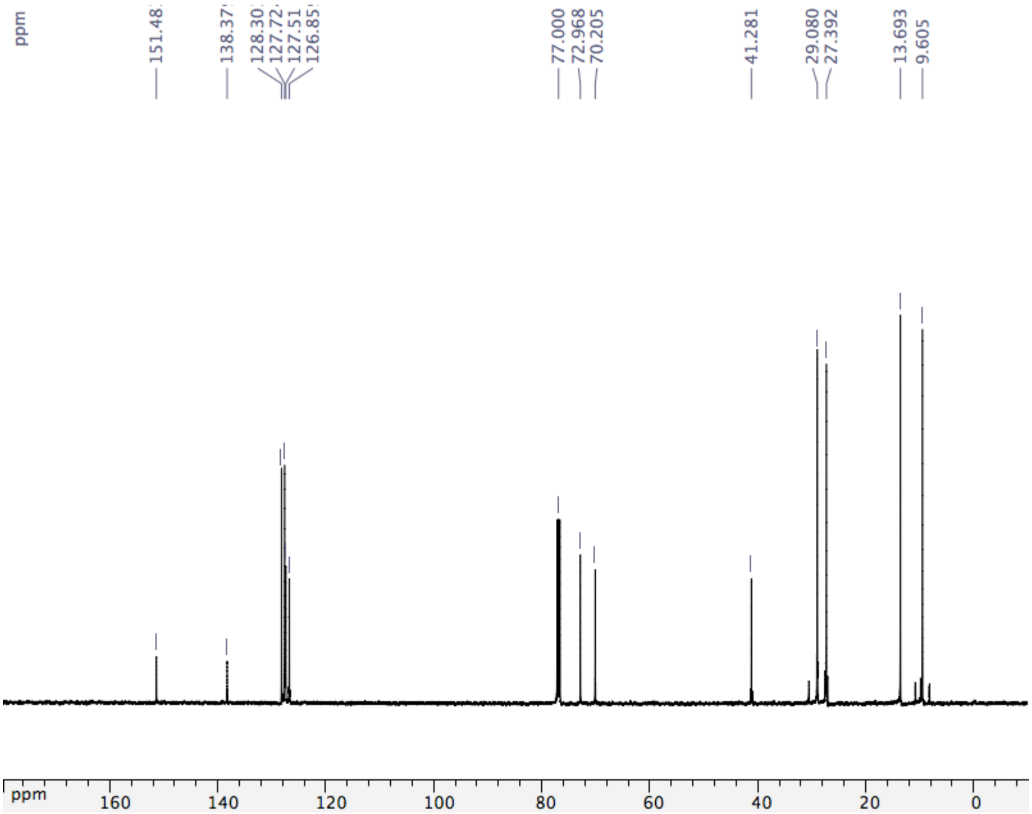
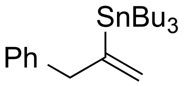
77.000
44.410
38.092
32.200
29.102
27.385
13.685
9.542
ppm
160
140
120
100
80
60
40
20
0


SnBu3
BnO

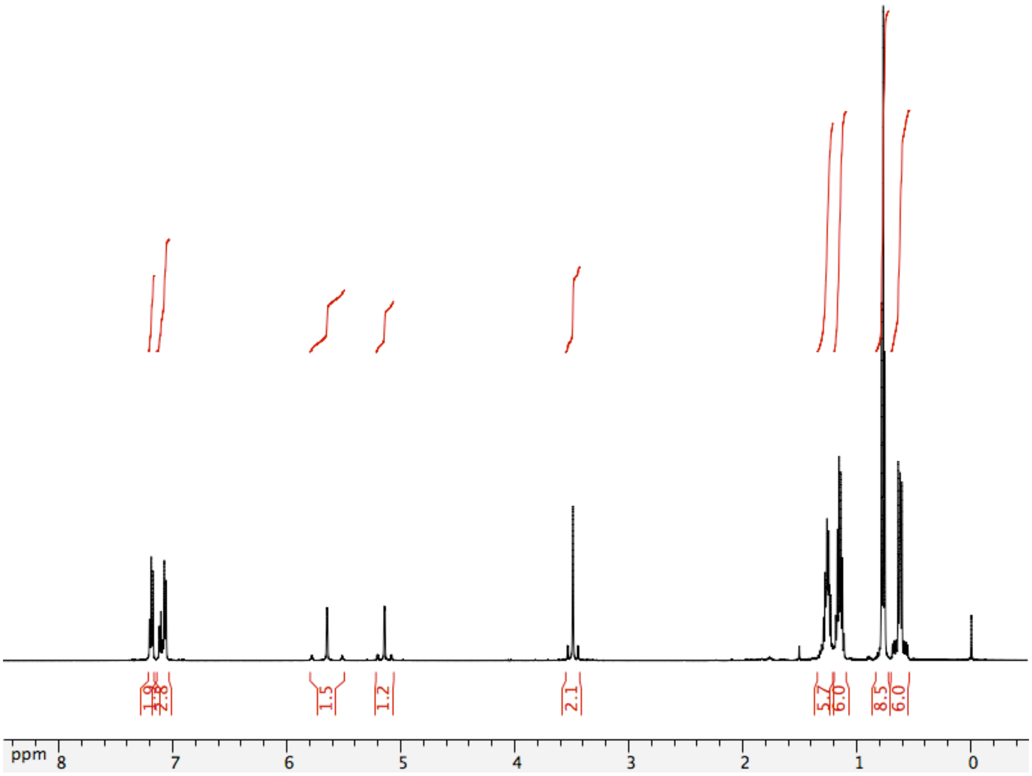
3f

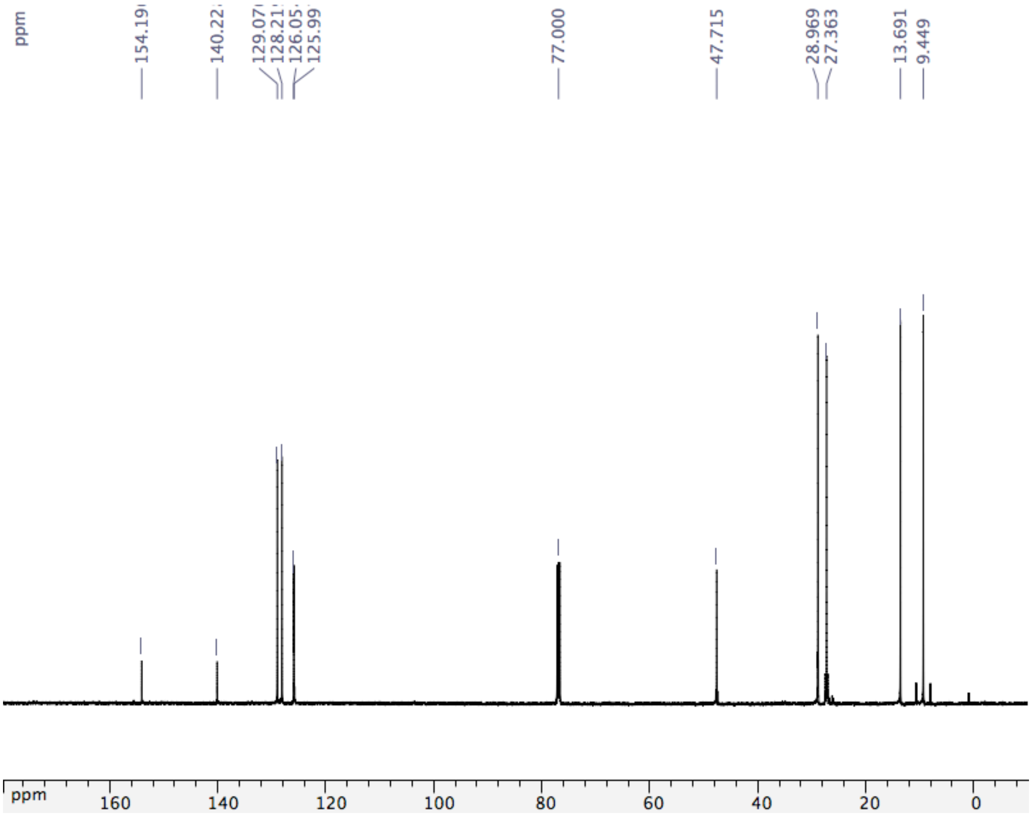

4.4
1.4
1.3
1.7
1.9
2.1
6.4
6.1
15.0
ppm
8
7
6
5
4
3
2
1
0


ppm
151.48
138.37
128.30
127.72
127.51
126.85
77.000
72.968
70.205
41.281
29.080
27.392
13.693
9.605
ppm
160
140
120
100
80
60
40
20
0


SnBu3
Ph

3w


1.9
1.8
1.5
1.2
2.1
5.7
6.0
8.5
6.0
ppm
8
7
6
5
4
3
2
1
0


ppm
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129.07
128.21
126.05
125.99
77.000
47.715
28.969
27.363
13.691
9.449
ppm
160
140
120
100
80
60
40
20
0