

Supplementary Information for

Copper-catalyzed distannylation of alkynes

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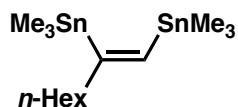
General remarks.

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 (^1H , 400 MHz; ^{13}C , 100 MHz) spectrometer or a Varian System 500 (^1H , 500 MHz; ^{13}C , 125 MHz; ^{119}Sn , 186 MHz) spectrometer using residual chloroform (^1H , $\delta = 7.26$) or CDCl_3 (^{13}C , $\delta = 77.0$) as an internal standard, and tetramethyltin (^{119}Sn , $\delta = 0$) as an external standard. ^1H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constants (Hz), integration. High-resolution mass spectra (ESI or APCI/FTMS) 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 Aluminium oxide 90 (activity IV). Hexamethyldistannane (**2**)¹ and tris(triphenylphosphine)copper acetate² were synthesized according to literature procedures. Alkynes were purified prior to use by distillation. DMF was distilled from calcium hydride and stored over activated molecular sieves 4A.

Cu-catalyzed distannylation of alkynes: a general procedure.

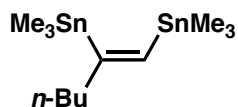
A Schlenk tube equipped with a magnetic stirring bar was charged with Cs_2CO_3 (0.060 mmol) before flame-drying under vacuum. To this were added $\text{Cu}(\text{OAc})(\text{PPh}_3)_3$ (6.0 μmol) and DMF (0.2 mL), and the resulting mixture was stirred at room temperature for 1 h. Then tetramethyldistannane (0.30 mmol) and an alkyne (0.45 mmol) were added, and the resulting mixture was stirred at 60 °C for the period as specified in Table 2. The mixture was diluted with ethyl acetate and filtered through a Celite plug. The organic solution was washed twice with water and dried over MgSO_4 . Evaporation of the solvent followed by preparative recycling gel permeation chromatography (chloroform or toluene as an eluent) or alumina-column chromatography (hexane as an eluent) gave the corresponding product.

(Z)-Oct-1-ene-1,2-diylbis(trimethylstannane) (3a)



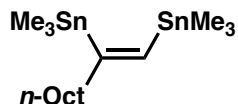
Isolated in 80% yield as a colorless oil: ^1H NMR (CDCl_3) δ 0.16 (s, $^2J_{\text{Sn-H}} = 53.0$ Hz, 9H), 0.17 (s, $^2J_{\text{Sn-H}} = 51.5$ Hz, 9H), 0.89 (t, $J = 5.3$ Hz, 3H), 1.27 (m, 4H), 2.33 (t, $^3J_{\text{Sn-H}} = 52.0$ Hz, $J = 7.5$ Hz, 2H), 6.61 (s, $^3J_{^{119}\text{Sn-H}} = 203.4$ Hz, $^3J_{^{117}\text{Sn-H}} = 194.3$ Hz, $^2J_{\text{Sn-H}} = 88.9$ Hz, 1H); ^{13}C NMR (CDCl_3) δ -7.71, -7.47, 14.09, 22.62, 28.88, 29.88, 31.72, 47.67, 142.47, 169.16; ^{119}Sn NMR (CDCl_3) δ -63.48, -50.93; HRMS Calcd for $\text{C}_{13}\text{H}_{29}\text{Sn}_2$: $[\text{M-Me}]^-$, 425.03077. Found: m/z 425.03107.

(Z)-Hex-1-ene-1,2-diylbis(trimethylstannane) (3b)



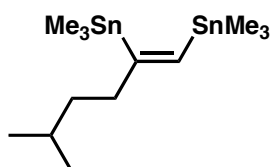
Isolated in 80% yield as a colorless oil: ^1H NMR (CDCl_3) δ 0.16 (s, $^2J_{\text{Sn-H}} = 52.8$ Hz, 9H), 0.17 (s, $^2J_{\text{Sn-H}} = 51.5$ Hz, 9H), 0.90 (t, $J = 7.2$ Hz, 3H), 1.32 (m, 4H), 2.34 (t, $^3J_{\text{Sn-H}} = 50.6$ Hz, $J = 6.4$ Hz, 2H), 6.61 (s, $^3J_{^{119}\text{Sn-H}} = 202.7$ Hz, $^3J_{^{117}\text{Sn-H}} = 193.7$ Hz, $^2J_{\text{Sn-H}} = 88.2$ Hz, 1H); ^{13}C NMR (CDCl_3) δ -7.69, -7.47, 14.00, 22.27, 32.10, 47.35, 142.51, 169.21; ^{119}Sn NMR (CDCl_3) δ -63.55, -50.90; HRMS Calcd for $\text{C}_{11}\text{H}_{25}\text{Sn}_2$: $[\text{M-Me}]^-$, 396.99947. Found: m/z 397.00031.

(Z)-Dec-1-ene-1,2-diylbis(trimethylstannane) (3c)



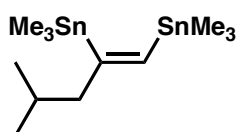
Isolated in 90% yield as a colorless oil: ^1H NMR (CDCl_3) δ 0.16 (s, $^2J_{\text{Sn-H}} = 53.4$ Hz, 9H), 0.18 (s, $^2J_{\text{Sn-H}} = 52.3$ Hz, 9H), 0.89 (t, $J = 7.0$ Hz, 3H), 1.23–1.37 (m, 8H), 2.33 (t, $^3J_{\text{Sn-H}} = 51.1$ Hz, $J = 6.7$ Hz, 2H), 6.61 (s, $^3J_{\text{H}^{119}\text{Sn-H}} = 203.1$ Hz, $^3J_{\text{H}^{117}\text{Sn-H}} = 194.2$ Hz, $^2J_{\text{Sn-H}} = 89.6$ Hz, 1H); ^{13}C NMR (CDCl_3) δ -7.70, -7.47, 14.12, 22.67, 29.19, 29.27, 31.87, 29.91, 29.44, 47.66, 142.46, 169.27; ^{119}Sn NMR (CDCl_3) δ -63.50, -50.93; HRMS Calcd for $\text{C}_{15}\text{H}_{33}\text{Sn}_2$: $[\text{M-Me}]^-$, 453.06207. Found: m/z 453.06271.

(Z)-(5-Methylhex-1-ene-1,2-diyl)bis(trimethylstannane) (3d)



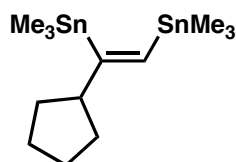
Isolated in 85% yield as a colorless oil: ^1H NMR (CDCl_3) δ 0.16 (s, $^2J_{\text{H}^{119}\text{Sn-H}} = 53.8$ Hz, $^2J_{\text{H}^{117}\text{Sn-H}} = 51.7$ Hz, 9H), 0.17 (s, $J_{\text{H}^{119}\text{Sn-H}} = 52.5$ Hz, $^3J_{\text{H}^{117}\text{Sn-H}} = 50.3$ Hz, 9H), 0.88 (d, $J = 6.7$ Hz, 6H), 1.22 (q, $J = 6.7$ Hz, 2H), 1.53 (nonet, $J = 6.7$ Hz, 1H), 2.25–2.40 (m, 2H), 6.62 (t, $^3J_{\text{H}^{119}\text{Sn-H}} = 202.7$ Hz, $^3J_{\text{H}^{117}\text{Sn-H}} = 193.6$ Hz, $^2J_{\text{H}^{119}\text{Sn-H}} = 89.8$ Hz, $^2J_{\text{H}^{117}\text{Sn-H}} = 86.1$ Hz, $J = 1.2$ Hz); ^{13}C NMR (CDCl_3) δ -7.70, -7.41, 22.60, 27.75, 39.33, 45.58, 142.37, 169.39; ^{119}Sn NMR (CDCl_3) δ -63.41, -50.60; HRMS Calcd for $\text{C}_{12}\text{H}_{27}\text{Sn}_2$: $[\text{M-Me}]^-$, 411.01512. Found: m/z 411.01593.

(Z)-(4-Methylpent-1-ene-1,2-diyl)bis(trimethylstannane) (3e)



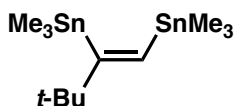
Isolated in 62% yield as a colorless oil: ^1H NMR (CDCl_3) δ 0.14 (s, $^2J_{\text{Sn-H}} = 52.9$ Hz, 9H), 0.14 (s, $^2J_{\text{Sn-H}} = 51.5$ Hz, 9H), 0.13 (d, $J = 6.6$ Hz, 6H), 1.55 (nonet, $J = 6.6$ Hz, 2H), 2.19 (dd, $^3J_{\text{Sn-H}} = 53.9$ Hz, $J = 7.4, 1.2$ Hz, 2H), 6.52 (t, $^3J_{\text{H}^{119}\text{Sn-H}} = 203.4$ Hz, $^3J_{\text{H}^{117}\text{Sn-H}} = 194.4$ Hz, $^2J_{\text{H}^{119}\text{Sn-H}} = 91.1$ Hz, $^2J_{\text{H}^{117}\text{Sn-H}} = 87.3$ Hz, $J = 1.2$ Hz, 1H); ^{13}C NMR (CDCl_3) δ -7.70, -7.43, 22.19, 27.69, 57.52, 143.90, 168.37; ^{119}Sn NMR (CDCl_3) δ -64.50, -50.93; HRMS Calcd for $\text{C}_{11}\text{H}_{25}\text{Sn}_2$: $[\text{M-Me}]^-$, 396.99947. Found: m/z 397.00095.

(Z)-(1-Cyclopentylethene-1,2-diyl)bis(trimethylstannane) (3f)



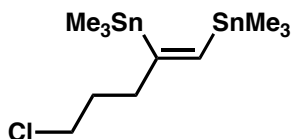
Isolated in 78% yield as a colorless oil: ^1H NMR (CDCl_3) δ 0.16 (s, $^2J_{\text{Sn-H}}^{119} = 53.8$ Hz, $^2J_{\text{Sn-H}}^{117} = 51.5$ Hz, 9H), 0.18 (s, $^2J_{\text{Sn-H}}^{119} = 52.1$ Hz, $^2J_{\text{Sn-H}}^{117} = 50.0$ Hz, 9H), 1.33–1.78 (m, 8H), 2.57–2.73 (m, 1H), 6.63 (d, $^3J_{\text{Sn-H}}^{119} = 210.0$ Hz, $^3J_{\text{Sn-H}}^{117} = 200.1$ Hz, $^2J_{\text{Sn-H}} = 88.2$ Hz, $J = 1.3$ Hz, 1H); ^{13}C NMR (CDCl_3) δ -7.38, -6.66, 32.13, 24.88, 55.93, 138.80, 172.21; ^{119}Sn NMR (CDCl_3) δ -61.72, -53.93; HRMS Calcd for $\text{C}_{12}\text{H}_{25}\text{Sn}_2$: $[\text{M-Me}]^-$, 408.99947. Found: m/z 408.99896.

(Z)-(3,3-Dimethylbut-1-ene-1,2-diyl)bis(trimethylstannane) (3g)



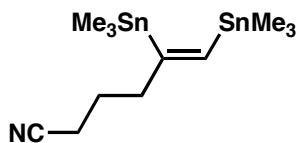
Isolated in 10% yield as a colorless oil: ^1H NMR (CDCl_3) δ 0.17 (s, $^2J_{\text{Sn-H}} = 52.8$ Hz, 9H), 0.21 (s, $^2J_{\text{Sn-H}} = 50.3$ Hz, 9H), 1.06 (s, 9H), 6.56 (s, $^3J_{\text{Sn-H}}^{119} = 218.7$ Hz, $^3J_{\text{Sn-H}}^{117} = 209.0$ Hz, $^2J_{\text{Sn-H}} = 69.6$ Hz, 1H); ^{13}C NMR (CDCl_3) δ -6.87, -4.59, 30.02, 135.85, 178.85; ^{119}Sn NMR (CDCl_3) δ -60.50, -57.91; HRMS Calcd for $\text{C}_{11}\text{H}_{25}\text{Sn}_2$: $[\text{M-Me}]^-$, 396.99947. Found: m/z 396.99942.

(Z)-(5-Chloropent-1-ene-1,2-diyl)bis(trimethylstannane) (3h)



Isolated in 76% yield as a yellow oil: ^1H NMR (CDCl_3) δ 0.16 (s, $^2J_{\text{Sn-H}}^{119} = 54.3$ Hz, $^2J_{\text{Sn-H}}^{117} = 51.6$ Hz, 9H), 0.18 (s, $^2J_{\text{Sn-H}}^{119} = 53.0$ Hz, $^2J_{\text{Sn-H}}^{117} = 50.1$ Hz, 9H), 1.82 (quint, $J = 6.6$ Hz, 2H), 2.48 (t, $^3J_{\text{Sn-H}} = 48.2$ Hz, $J = 7.7$ Hz, 2H), 3.50 (t, $J = 7.0$ Hz, 2H), 6.67 (s, $^3J_{\text{Sn-H}}^{119} = 195.4$ Hz, $^3J_{\text{Sn-H}}^{117} = 189.6$ Hz, $^2J_{\text{Sn-H}} = 84.5$ Hz, 1H); ^{13}C NMR (CDCl_3) δ -7.70, -7.50, 32.45, 44.15, 44.36, 144.75, 166.61; ^{119}Sn NMR (CDCl_3) δ -62.44, -49.22; HRMS Calcd for $\text{C}_{10}\text{H}_{22}\text{ClSn}_2$: $[\text{M-Me}]^-$, 416.94485. Found: m/z 416.94467.

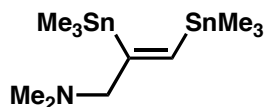
(Z)-5,6-Bis(trimethylstannyl)hex-5-enenitrile (3i)



Isolated in 51% yield as a brown oil: ^1H NMR (CDCl_3) δ 0.17 (s, $^2J_{\text{Sn-H}}^{119} = 54.0$ Hz, $^2J_{\text{Sn-H}}^{117} = 52.1$ Hz, 9H), 0.19 (s, $^2J_{\text{Sn-H}}^{119} = 52.9$ Hz, $^2J_{\text{Sn-H}}^{117} = 50.5$ Hz, 9H), 1.72 (quint, $J = 7.4$ Hz, 2H), 2.30 (t, $J = 7.4$ Hz, 2H), 2.38–2.55 (m, 2H), 6.69 (s, $^3J_{\text{Sn-H}}^{119} = 194.1$ Hz, $^3J_{\text{Sn-H}}^{117} = 185.8$ Hz, $^2J_{\text{Sn-H}}^{119} = 83.3$ Hz, $^2J_{\text{Sn-H}}^{117} = 80.8$ Hz, 1H); ^{13}C NMR (CDCl_3) δ -7.69, -7.50,

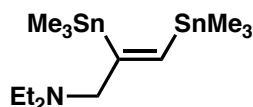
16.29, 25.00, 45.69, 119.67, 146.03, 165.67; ^{119}Sn NMR (CDCl_3) δ -61.85, -48.64; HRMS Calcd for $\text{C}_{11}\text{H}_{22}\text{NSn}_2$: $[\text{M-Me}]^-$, 407.97907. Found: m/z 407.98001.

(Z)-N,N-Dimethyl-2,3-bis(trimethylstannyl)prop-2-en-1-amine (3j)



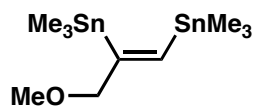
Isolated in 54% yield as a colorless oil: ^1H NMR (CDCl_3) δ 0.13 (s, $^2J_{\text{Sn-H}}^{119} = 53.5$ Hz, $^2J_{\text{Sn-H}}^{117} = 51.6$ Hz, 9H), 0.17 (s, $^2J_{\text{Sn-H}}^{119} = 54.1$ Hz, $^2J_{\text{Sn-H}}^{117} = 52.1$ Hz, 9H), 2.12 (s, 6H), 3.01 (d, $^3J_{\text{Sn-H}} = 48.1$ Hz, $J = 1.4$ Hz, 2H), 6.73 (t, $^3J_{\text{Sn-H}}^{119} = 201.3$ Hz, $^3J_{\text{Sn-H}}^{117} = 192.4$ Hz, $^2J_{\text{Sn-H}}^{119} = 90.5$ Hz, $^2J_{\text{Sn-H}}^{117} = 87.1$ Hz, $J = 1.4$ Hz, 1H); ^{13}C NMR (CDCl_3) δ -7.71, -7.23, 45.03, 75.52, 142.83, 169.79; ^{119}Sn NMR (CDCl_3) δ -62.24, -54.24; HRMS Calcd for $\text{C}_{10}\text{H}_{24}\text{NSn}_2$: $[\text{M-Me}]^-$, 397.99472. Found: m/z 397.99518.

(Z)-N,N-Diethyl-2,3-bis(trimethylstannyl)prop-2-en-1-amine (3k)



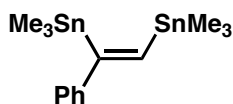
Isolated in 54% yield as a colorless oil: ^1H NMR (CDCl_3) δ 0.18 (s, $^2J_{\text{Sn-H}} = 53.2$ Hz, 18H), 3.29 (s, 3H), 4.02 (s, $^3J_{\text{Sn-H}} = 39.3$ Hz, 2H), 6.89 (s, $^3J_{\text{Sn-H}}^{119} = 190.3$ Hz, $^3J_{\text{Sn-H}}^{117} = 183.1$ Hz, $^2J_{\text{Sn-H}} = 84.1$ Hz, 1H); ^{13}C NMR (CDCl_3) δ -7.61, -7.13, 10.50, 44.92, 70.14, 142.75, 169.83; ^{119}Sn NMR (CDCl_3) δ -63.05, -55.94; HRMS Calcd for $\text{C}_{12}\text{H}_{28}\text{NSn}_2$: $[\text{M-Me}]^-$, 426.02602. Found: m/z 426.02637.

(Z)-(3-Methoxyprop-1-ene-1,2-diyl)bis(trimethylstannane) (3l)



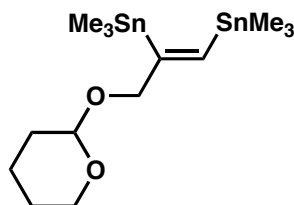
Isolated in 41% yield as a colorless oil: ^1H NMR (CDCl_3) δ 0.17 (s, $^2J_{\text{Sn-H}} = 52.8$ Hz, 9H), 0.21 (s, $^2J_{\text{Sn-H}} = 50.3$ Hz, 9H), 1.06 (s, 9H), 6.56 (s, $^3J_{\text{Sn-H}}^{119} = 218.7$ Hz, $^3J_{\text{Sn-H}}^{117} = 209.0$ Hz, $^2J_{\text{Sn-H}} = 69.6$ Hz, 1H); ^{13}C NMR (CDCl_3) δ -7.78, -7.68, 57.66, 84.37, 144.71, 165.17; ^{119}Sn NMR (CDCl_3) δ -59.42, -48.53; ^{119}Sn NMR (CDCl_3) δ -62.24, -54.24; HRMS Calcd for $\text{C}_9\text{H}_{21}\text{OSn}_2$: $[\text{M-Me}]^-$, 384.96309. Found: m/z 384.96249.

(Z)-(1-Phenylethene-1,2-diyl)bis(trimethylstannane) (3m)



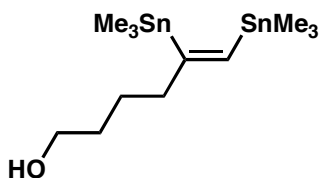
Isolated in 64% yield as a yellow oil: ^1H NMR (CDCl_3) δ 0.19 (s, $^2J_{\text{Sn-H}}^{119} = 53.4$ Hz, $^2J_{\text{Sn-H}}^{117} = 51.1$ Hz, 9H), 0.24 (s, $^2J_{\text{Sn-H}}^{119} = 54.5$ Hz, $^2J_{\text{Sn-H}}^{117} = 52.0$ Hz, 9H), 6.95 (s, $^3J_{\text{Sn-H}}^{119} = 188.1$ Hz, $^3J_{\text{Sn-H}}^{117} = 179.9$ Hz, $^2J_{\text{Sn-H}}^{119} = 82.7$ Hz, $^2J_{\text{Sn-H}}^{117} = 79.0$ Hz, 1H), 7.02–7.03 (m, 2H), 7.13–7.18 (m, 1H), 7.24–7.30 (m, 2H); ^{13}C NMR (CDCl_3) δ -7.47, -6.70, 125.79, 125.85, 128.07, 148.97, 150.30, 168.43; ^{119}Sn NMR (CDCl_3) δ -58.47, -44.84; HRMS Calcd for $\text{C}_{13}\text{H}_{21}\text{Sn}_2$: $[\text{M-Me}]^-$, 416.96817. Found: m/z 416.96741.

(Z)-(3-((Tetrahydro-2H-pyran-2-yl)oxy)prop-1-ene-1,2-diyl)bis(trimethylstannane)
(3n)



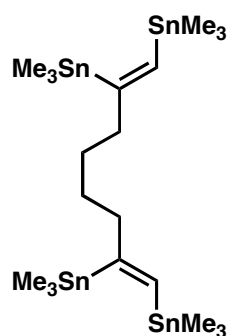
Isolated in 58% yield as a yellow green oil: ^1H NMR (CDCl_3) δ 0.18 (s, $^2J_{\text{Sn-H}} = 53.2$ Hz, 9H), 0.18 (s, $^2J_{\text{Sn-H}} = 52.9$ Hz, 9H), 1.47–1.65 (m, 4H), 1.66–1.75 (m, 1H), 1.70 (tt, $J = 13.1, 3.3$ Hz, 1H), 3.47–3.54 (m, 1H), 3.86 (ddd, $J = 11.8, 8.7, 3.0$ Hz, 1H), 4.06 (dd, $^3J_{\text{Sn-H}}^{119} = 34.2$ Hz, $J = 12.1, 1.6$ Hz, 1H), 4.36 (dd, $^3J_{\text{Sn-H}}^{119} = 39.6$ Hz, $J = 12.0, 1.6$ Hz, 1H), 4.62 (t, $J = 3.3$ Hz, 1H), 6.69 (t, $^3J_{\text{Sn-H}}^{119} = 192.6$ Hz, $^3J_{\text{Sn-H}}^{117} = 184.1$ Hz, $^2J_{\text{Sn-H}}^{119} = 85.2$ Hz, $^2J_{\text{Sn-H}}^{117} = 81.7$ Hz, $J = 1.4$ Hz, 1H); ^{13}C NMR (CDCl_3) δ -7.65, 7.50, 19.29, 25.42, 25.45, 30.55, 61.93, 78.74, 97.81, 144.00, 164.75; ^{119}Sn NMR (CDCl_3) δ -59.66, -46.49; HRMS Calcd for $\text{C}_{14}\text{H}_{30}\text{O}_2\text{NaSn}_2$: $[\text{M}+\text{Na}]^+$, 493.01820. Found: m/z 493.01810.

(Z)-5,6-Bis(trimethylstannyl)hex-5-en-1-ol (3o)



Isolated in 13% yield as a colorless oil: ^1H NMR (CDCl_3) δ 0.15 (s, $^2J_{\text{Sn-H}} = 52.8$ Hz, 9H), 0.17 (s, $^2J_{\text{Sn-H}} = 51.5$ Hz, 9H), 1.22 (t, $J = 4.2$ Hz, 1H), 1.41 (quint, $J = 7.3$ Hz, 2H), 1.55 (quint, $J = 7.3$ Hz, 2H), 2.35 (t, $^3J_{\text{Sn-H}} = 50.1$, $J = 7.6$ Hz, 2H), 3.64 (q, $J = 5.4$ Hz, 2H), 6.62 (s, $^3J_{\text{Sn-H}}^{119} = 199.8$ Hz, $^3J_{\text{Sn-H}}^{117} = 193.7$ Hz, $^2J_{\text{Sn-H}}^{119} = 86.6$ Hz, 1H); ^{13}C NMR (CDCl_3) δ -7.72, -7.49, 25.82, 32.23, 47.21, 62.84, 143.22, 168.51; ^{119}Sn NMR (CDCl_3) δ -63.09, -50.40; HRMS Calcd for $\text{C}_{11}\text{H}_{25}\text{OSn}_2$: $[\text{M-Me}]^-$, 412.99439. Found: m/z 412.99545.

(1Z,7Z)-Octa-1,7-diene-1,2,7,8-tetrayltetrakis(trimethylstannane) (3p)



Isolated in 66% yield as a colorless oil: ^1H NMR (CDCl_3) δ 0.15 (s, $^2J_{^{119}\text{Sn-H}} = 54.1$ Hz, $^2J_{^{117}\text{Sn-H}} = 51.7$ Hz, 18H), 0.16 (s, $^2J_{^{119}\text{Sn-H}} = 52.7$ Hz, $^2J_{^{117}\text{Sn-H}} = 50.3$ Hz, 18H), 1.33 (quint, $J = 3.5$ Hz, 4H), 2.33 (t, $^3J_{\text{Sn-H}} = 50.7$ Hz, $J = 6.2$ Hz, 4H), 6.60 (s, $^3J_{^{119}\text{Sn-H}} = 202.2$ Hz, $^3J_{^{117}\text{Sn-H}} = 193.3$ Hz, $^2J_{^{119}\text{Sn-H}} = 89.7$ Hz, $^2J_{^{117}\text{Sn-H}} = 86.2$ Hz, 2H); ^{13}C NMR (CDCl_3) δ -7.68, -7.41, 29.45, 47.44, 142.84, 168.91; ^{119}Sn NMR (CDCl_3) δ -63.33, -50.65; HRMS Calcd for $\text{C}_{19}\text{H}_{43}\text{Sn}_4$: $[\text{M-Me}]^-$, 750.94472. Found: m/z 750.94637.

References

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