Supporting Information

Highly Diastereoselective Hydrostannylation of Allyl and Homoallyl Alcohols with Dibutyl(trifluoromethanesulfoxy)stannane

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1. General Method

Unless otherwise noted, all reactions and distillation of solvents were carried out under N₂. Solvents were dried by distillation from sodium metal/benzophenone ketyl (THF, Et₂O) and CaH₂ (hexane, CH₂Cl₂). Allyl and homoallyl alcohols were distilled simply. Boiling points determined with Kugelrohr distillation apparatus are indicated by air-bath temperature (bath temp.). ¹H and ¹³C NMR were recorded in CDCl₃ or C₆D₆ at 270 and 67.7 MHz, respectively. The chemical shifts (δ) are reported with reference at 0.00 ppm (Me₄Si), 7.15 ppm (C₆HD₅) or 7.26 ppm (CHCl₃) for the proton and at 77.00 ppm (centered on the signal of CDCl₃) or 128.00 ppm (centered on the signal of C₆D₆) for the carbon.

2. Substrates and Reagents

2. 1. Hydrostannanes

Bu₂Sn(OTf)H (**1a**) was prepared from Bu₂SnH₂ and TfOH.¹ According to the reported procedure,² Bu₂SnClH (**1b**) was prepared from Bu₂SnCl₂ and Bu₂SnH₂, and directly used for the hydrostannylation in the same flask.

2. 2. Allyl and Homoallyl Alcohols, and Allyl Ether **2a'**

Allyl alcohols **2a** and **2c-f** were prepared by the reactions of isopropenylmagnesium bromide with aldehydes. For the synthesis of **2b**, methacrolein was methylated with MeMgBr. Allyl alcohol **2g** was obtained by the Morita-Baylis-Hillman reaction of methyl acrylate with benzaldehyde.^{3,4} The synthesis of **2h** was carried out by the reaction of phenylmagnesium bromide with 1-cyclohexenecarbaldehyde. Allyl ether **2a'** was prepared from **2a** by deprotonation with BuLi followed by methylation with MeI. Homoallyl alcohols **4a-d** were obtained by the reactions of methallylmagnesium chloride with aldehydes. For the synthesis of **4e**, the reductive coupling of benzaldehyde and methyl 2-

(bromomethyl)acrylate was carried out with zinc powder.⁵

2-Methyl-1-phenyl-2-propen-1-ol (2a) [4383-08-8]⁶

Isopropenylmagnesium bromide was prepared from 2-bromopropene and magnesium turning in Et₂O at room temperature. Benzaldehyde (4.0 mL, 40 mmol) was dropwise added to the ethereal solution of isopropenylmagnesium bromide (0.66 M, 70 mL, 46 mmol) at 0 °C. The mixture was stirred at 0 °C for 1 h and warmed to room temperature. After being stirred overnight, the reaction mixture was poured into iced aqueous HCl (1 M, 50 mL) and extracted with *t*-BuOMe (3 × 30 mL). The extract was washed with saturated aqueous NaHCO₃, dried over Na₂SO₄, and evaporated. Purification of the residual oil by silica gel column chromatography (hexane-AcOEt 10:1) gave the title compound (4.7 g, 32 mmol, 80%). ¹H NMR (CDCl₃) δ 1.61 (s, 3H), 1.83 (br s, 1H), 4.94–4.97 (m, 1H), 5.14 (s, 1H), 5.20–5.21 (m, 1H), 7.24–7.40 (m, 5H); ¹³C NMR (CDCl₃) δ 18.18 (CH₃), 77.78 (CH), 111.09 (CH₂), 126.41 (CH $\times$ 2), 127.57 (CH), 128.31 (CH $\times$ 2), 141.91(C), 146.76 (C); MS *m/z* (relative intensity) 148 (M⁺, 19), 133 (M⁺ – CH₃, 24), 51 (100). These NMR data for **2a** agree with those reported in ref 6.

3-Methyl-3-buten-2-ol (2b) [10473-14-0]⁷

The title compound was synthesized by the reaction of methacrolein with MeMgI in 53% yield. For the NMR data for **2b**, see ref 7.

2,5-Dimethyl-1-penten-3-ol (2c) [19781-54-5]⁸

The title compound was prepared from isobutyraldehyde and isopropenylmagnesium bromide in 37% yield. IR (neat) 3392 (br, OH), 2958, 1650 (weak, C=C), 1022 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.86 (d, $J = 6.9$ Hz, 3H), 0.96 (d, $J = 6.8$ Hz, 3H), 1.60 (br s, 1H), 1.70–1.72 (m, 3H), 1.79 (sep, $J = 6.8$ Hz, 1H), 3.71 (d, $J = 7.3$ Hz, 1H), 4.86–4.88 (m, 1H), 4.91–4.93 (m, 1H); ^{13}C NMR (CDCl_3) δ 17.48 (CH_3), 17.65 (CH_3), 19.36 (CH_3), 30.81 (CH), 81.68 (CH), 112.04 (CH_2), 146.70 (C).

2,5,5-Trimethyl-1-penten-3-ol (2d) [7432-48-6]⁹

The title compound was prepared from pivalaldehyde and isopropenylmagnesium bromide in 58% yield. IR (neat) 3446 (br, OH), 2954, 1645 (weak, C=C), 1059, 1001 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.94 (s, 9H), 1.58 (br s, 1H), 1.79–1.80 (m, 3H), 3.79 (s, 1H), 4.91–4.92 (m, 1H), 4.95–4.97 (m, 1H); ^{13}C NMR (CDCl_3) δ 20.74 (CH_3), 26.42 (CH_3 $\times$ 3), 35.22 (C), 83.31 (CH), 113.56 (CH_2), 146.67 (C). Anal. Calcd for $\text{C}_8\text{H}_{16}\text{O}$: C, 74.94; H, 12.58. Found: C, 74.67; H, 12.88.

1-(4-Methoxyphenyl)-2-methyl-2-propen-1-ol (2e) [139711-00-5]¹⁰

The title compound was prepared from 4-methoxybenzaldehyde and isopropenylmagnesium bromide in 65% yield. Bp 90 $^{\circ}\text{C}$ (0.9 Torr, bath temp.). IR (neat) 3419 (br, OH), 1652 (weak, C=C), 1510, 1248, 1036 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.60 (br s, 3H), 1.89 (br s, 1H), 3.80 (s, 3H), 4.94 (br s, 1H), 5.08 (br s, 1H), 5.20 (br s, 1H), 6.85–6.90 (m, 2H), 7.26–7.31 (m, 2H). ^{13}C NMR (CDCl_3) δ 18.44 (CH_3), 55.23

(CH₃), 77.31 (CH), 110.60 (CH₂), 113.75 (CH □ 2), 127.73 (CH □ 2), 134.16 (C), 146.96 (C), 159.11 (C); MS *m/z* (relative intensity) 178 (M⁺, 37), 137 (100). Anal. Calcd for C₁₁H₁₄O₂: C, 74.13; H, 7.92. Found: C, 74.26; H, 7.92.

1-(1-Naphthyl)-2-methyl-2-propen-1-ol (2f) [257865-43-3] for one enantiomer^{10b}

The title compound was prepared from 1-naphthalenecarboxaldehyde and isopropenylmagnesium bromide in 68% yield. Bp 102 °C (0.42 Torr, bath temp.). IR (neat) 3354 (br, OH), 1653 (weak, C=C), 1056, 798, 781 cm⁻¹; ¹H NMR (CDCl₃) □ 1.65 (s, 3H), 2.13 (br s, 1H), 5.08 (br s, 1H), 5.29 (br s, 1H), 5.82 (br s, 1H), 7.43–7.53 (m, 3H), 7.61 (d, *J* = 6.8 Hz, 1H), 7.78–7.87 (m, 2H), 8.15–8.20 (m, 1H); ¹³C NMR (CDCl₃) □ 19.15 (CH₃), 74.57 (CH), 112.27 (CH₂), 123.64 (CH), 124.37 (CH), 125.24 (CH), 125.48 (CH), 125.91 (CH), 128.32 (CH), 128.66 (CH), 131.02 (C), 133.80 (C), 137.14 (C), 146.28 (C); MS *m/z* (relative intensity) 198 (M⁺, 46), 129 (100). Anal. Calcd for C₁₄H₁₄O: C, 84.81; H, 7.12. Found: C, 84.84; H, 7.35.

Methyl 2-Hydroxy(phenyl)methyl-2-propenoate (2g) [18020-59-2]^{4b,c}

A mixture of benzaldehyde (12.0 mL, 118 mmol), DABCO (1.3 g, 12 mmol), and methyl acrylate (7.0 mL, 78 mmol) was stirred at 60 °C for 2 days. The resultant mixture was poured into 0.1 M HCl (150 mL) and extracted with *t*-BuOMe (3 □ 50 mL). The extract was washed with saturated aqueous NaHCO₃, dried over Na₂SO₄, and evaporated. Purification of the residual oil by silica gel column

chromatography (hexane-AcOEt 3:1) gave the title compound (7.1g, 37 mmol, 47%). For the characterization data for **2g**, see ref. 4c.

1-Cyclohexenyl(phenyl)methanol (2h) [36306-47-5]¹¹

The title compound was prepared from 1-cyclohexenecarboxaldehyde and phenylmagnesium bromide in 95% yield. Bp 105 °C (3.0 Torr, bath temp.). IR (neat) 3352 (br, OH), 2927, 1450, 1016 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.49–1.62 (m, 4H), 1.68–1.98 (m, 3H), 2.04–2.13 (m, 2H), 5.08 (s, 1H), 5.84–5.88 (m, 1H), 7.22–7.38 (m, 5H); ^{13}C NMR (CDCl_3) δ 22.42 (CH_2), 22.48 (CH_2), 23.97 (CH_2), 24.99 (CH_2), 78.21 (CH), 123.46 (CH), 126.28 (CH $\times$ 2), 127.19 (CH), 128.14 (CH $\times$ 2), 139.58 (C), 142.54 (C); MS m/z (relative intensity) 188 (M^+ , 66), 105 (100). Anal. Calcd for $\text{C}_{13}\text{H}_{16}\text{O}$: C, 82.94; H, 8.57. Found: C, 82.71; H, 8.43.

3-Methoxy-2-methyl-3-phenyl-1-propene (2a') [142024-56-4]¹²

BuLi (1.64 M in hexane, 7.3 mL, 12.0 mmol) was dropwise added to a solution of **2a** (1.78 g, 12.0 mmol) in THF (30 mL) at 0 °C. After 10 min MeI (0.78 mL, 12.6 mmol) was added to the mixture, then the reaction mixture was warmed to room temperature. After 6 h the resultant mixture was poured into water and extracted with *t*-BuOMe (3 $\times$ 30 mL). The extract was dried over Na_2SO_4 , and evaporated. Purification of the residual oil by silica gel column chromatography (hexane-AcOEt 20:1) gave the title compound (0.80g, 4.9 mmol, 41%). IR (neat) 1653 (weak, C=C), 1450, 1097, 698 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.56–1.57 (m, 3H), 3.33 (s, 3H), 4.59 (s, 1H), 4.96–4.98 (m, 1H), 5.12–5.14 (m, 1H), 7.22–

7.34 (m, 5H); ^{13}C NMR (CDCl_3) δ 17.45 (CH_3), 56.47 (CH_3), 87.02 (CH), 112.87 (CH_2), 126.51 (CH δ 2), 127.33 (CH), 128.13 (CH δ 2), 140.42 (C), 144.92 (C); MS m/z (relative intensity) 162 (M^+ , 16), 161 ($\text{M}^+ - \text{H}$, 24), 121 (100). Anal. Calcd for $\text{C}_{11}\text{H}_{14}\text{O}$: C, 81.44; H, 8.70. Found: C, 81.11; H, 8.99.

3-Methyl-1-phenyl-3-buten-1-ol (4a) [23092-23-1]¹³

The title compound was prepared from benzaldehyde and methallylmagnesium chloride in 69% yield. Bp 76 °C (1.2 Torr, bath temp.); IR (neat) 3392 (br, OH), 2935, 1647 ($\text{C}=\text{C}$), 1454, 1055, 756, 700 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.79–1.81 (m, 3H), 1.92 (br s, 1H), 2.43 (dd, $J = 6.8, 1.0$ Hz, 2H), 4.82 (t, $J = 6.8$ Hz, 1H), 4.85–4.87 (m, 1H), 4.91–4.94 (m, 1H), 7.23–7.40 (m, 5H); ^{13}C NMR (CDCl_3) δ 22.35 (CH_3), 48.39 (CH_2), 71.42 (CH), 114.10 (CH_2), 125.77 (CH δ 2), 127.50 (CH), 128.42 (CH δ 2), 142.41 (C), 144.07 (C); MS m/z (relative intensity) 162 (M^+ , 1.3), 145 ($\text{M}^+ - \text{OH}$, 1.3), 107 (100). These characterization data for **4a** agree with those reported in ref. 13.

4-Methyl-4-penten-2-ol (4b) [2004-67-3]¹⁴

The title compound was prepared from acetaldehyde and methallylmagnesium chloride in 63% yield. For the characterization data for **4b**, see ref. 14.

2,5-Dimethyl-5-hexen-3-ol (4c) [19550-91-5]¹⁵

The title compound was prepared from isobutyraldehyde and methallylmagnesium chloride in 47% yield. Bp 100 °C (255 Torr, bath temp.). IR (neat) 3431 (br, OH), 2962, 1647 ($\text{C}=\text{C}$), 1055 cm^{-1} ; ^1H

NMR (CDCl₃) δ 0.94 (d, J = 6.8 Hz, 3H), 0.96 (d, J = 6.8 Hz, 3H), 1.64–1.77 (m, 5H), 2.06 (ddd, J = 13.7, 10.0, 0.7 Hz, 1H), 2.22 (dd, J = 13.7, 3.1 Hz, 1H), 3.48 (ddd, J = 10.0, 5.3, 3.1 Hz, 1H), 4.80–4.82 (m, 1H), 4.87–4.90 (m, 1H); ¹³C NMR (CDCl₃) δ 17.59 (CH₃), 18.60 (CH₃), 22.22 (CH₃), 33.33 (CH), 42.88 (CH₂), 73.05 (CH), 113.41 (CH₂), 143.22 (C). Anal. Calcd for C₈H₁₆O: C, 74.94; H, 12.58. Found: C, 74.61; H, 12.76. These characterization data for **4c** agree with those reported in ref. 13.

2,2,5-Trimethyl-5-hexen-3-ol (4d) [294675-15-3]^{15a,16}

The title compound was prepared from pivalaldehyde and methallylmagnesium chloride in 52% yield. Bp 60 °C (30 Torr); IR (neat) 3494 (br, OH), 2956, 1645 (C=C) cm⁻¹; ¹H NMR (CDCl₃) δ 0.93 (s, 9H), 1.65 (s, 1H), 1.77 (d, J = 0.7 Hz, 3H), 2.00 (dd, J = 13.7, 10.9, Hz, 1H), 2.25 (br d, J = 13.7 Hz, 1H), 3.34 (dd, J = 10.9, 2.1 Hz, 1H), 4.82 (br s, 1H), 4.90 (br s, 1H); ¹³C NMR (CDCl₃) δ 22.06 (CH₃), 25.74 (CH₃ $\times$ 3), 34.39 (C), 40.53 (CH₂), 75.61 (CH), 113.43 (CH₂), 143.69 (C). Anal. Calcd for C₉H₁₈O: C, 76.00; H, 12.76. Found: C, 75.71; H, 12.94.

Methyl 2-(2-Hydroxy-2-phenylethyl)-2-propenoate (4e) [138832-36-7]¹⁷

A mixture of benzaldehyde (3.13 mL, 30.8 mmol), methyl 2-(bromomethyl)acrylate (6.71 g, 37.5 mmol), saturated aqueous NH₄Cl (150 mL), THF (30 mL), and zinc powder (>90% pure, 2.7 g, >37 mmol) was stirred at room temperature for 9 h. The resultant mixture was poured into water (100 mL) and extracted with *t*-BuOMe (3 $\times$ 30 mL). The extract was dried over Na₂SO₄ and evaporated. Purification of the residual oil by silica gel column chromatography (hexane-AcOEt 4:1) gave the title compound (6.36g, 30.8 mmol, 100%). Bp 130 °C (1.2 Torr, bath temp.). IR (neat) 3442 (br, OH), 1716

(C=O), 1631 (C=C), 1441, 1203, 1144, 702 cm^{-1} ; ^1H NMR (CDCl_3) δ 2.64 (br s, 1H), 2.69 (ddd, J = 14.0, 8.4, 1.0 Hz, 1H), 2.78 (ddd, J = 14.0, 4.3, 1.2 Hz, 1H), 3.78 (s, 3H), 4.89 (dd, J = 8.2, 4.3 Hz, 1H), 5.60–5.62 (m, 1H), 6.24 (d, J = 1.3 Hz, 1H), 7.23–7.40 (m, 5H); ^{13}C NMR (CDCl_3) δ 42.17 (CH_2), 51.89 (CH_3), 72.75 (CH), 125.62 (CH $\times$ 2), 127.26 (CH), 128.13 (CH $\times$ 2), 128.24 (CH_2), 136.61 (C), 143.80 (C), 167.93 (C); MS m/z (relative intensity) 174 (M^+ – MeOH, 27), 68 (100). Anal. Calcd for $\text{C}_{12}\text{H}_{14}\text{O}_3$: C, 69.88; H, 6.84. Found: C, 70.14; H, 6.83.

3. Hydrostannylation

3. 1. Hydrostannylation of Allyl Alcohols (Typical Procedure)

Hydrostannane **1a** (0.42 g, 1.10 mmol), Et_3B (1.0 M, 0.05 mL, 0.05 mmol), and dry air (5 mL) were successively added to a solution of allyl alcohol **2a** (148 mg, 1.00 mmol) in Et_2O (2 mL) at 0 $^\circ\text{C}$. After being stirred for 3 h, the resultant mixture was diluted with Et_2O (2 mL). BuLi (1.72 M in hexane, 1.4 mL, 2.4 mmol) was dropwise added to the diluted mixture. After 20 min the reaction was quenched with aqueous NH_4Cl (saturated aqueous NH_4Cl (5 mL) and water (5 mL)). The extract with *t*-BuOMe (3 $\times$ 10 mL) was dried over Na_2SO_4 and evaporated. Purification of the residual oil by silica gel column chromatography (hexane-AcOEt 20:1) gave β -stannylated alcohol **3a** (320 mg, 0.73 mmol, 73%, *syn:anti* = 97:3).

(1*S**, 2*S**)-2-Methyl-1-phenyl-3-tributylstannyl-1-propanol (*syn*-**3a**)

Bp 160 $^\circ\text{C}$ (1.2 Torr, bath temp.). IR (neat) 3390 (br, OH), 2956, 2924, 1454 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.57–0.96 (m, 19H), 1.10–1.48 (m, 13H), 1.82 (d, J = 3.1 Hz, 1H), 1.99–2.11 (m, 1H), 4.42 (dd, J =

5.6, 3.3 Hz, 1H), 7.26–7.37 (m, 5H); ^{13}C NMR (CDCl_3) δ 9.34 (CH_2 $\times$ 3), 13.68 (CH_3 $\times$ 3), 14.14 (CH_2), 18.47 (CH_3), 27.39 (CH_2 $\times$ 3), 29.18 (CH_2 $\times$ 3), 38.61 (CH), 80.99 (CH), 126.54 (CH $\times$ 2), 127.25 (CH), 128.11 (CH $\times$ 2), 143.74 (C); MS m/z (relative intensity) 383 ($\text{M}^+ - \text{Bu}$, 18), 381 ($\text{M}^+ - 2 - \text{Bu}$, 15), 379 ($\text{M}^+ - 4 - \text{Bu}$, 8.7), 251 (100). Anal. Calcd for $\text{C}_{22}\text{H}_{40}\text{OSn}$: C, 60.15; H, 9.18. Found: C, 60.09; H, 9.07

(1*S, 2*R**)-2-Methyl-1-phenyl-3-tributylstannyl-1-propanol (*anti*-3a)**

Bp 160 °C (1.2 Torr, bath temp.). IR (neat) 3417 (br, OH), 2956, 2924, 1454 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.63–0.94 (m, 19H), 1.14 (dd, $J = 12.6, 3.5$ Hz, 1H), 1.22–1.56 (m, 12H), 1.88 (d, $J = 3.3$ Hz, 1H), 2.00–2.10 (m, 1H), 4.31 (dd, $J = 6.8, 3.5$ Hz, 1H), 7.26–7.37 (m, 5H); ^{13}C NMR (CDCl_3) δ 9.54 (CH_2 $\times$ 3), 13.27 (CH_2), 13.70 (CH_3 $\times$ 3), 20.19 (CH_3), 27.43 (CH_2 $\times$ 3), 29.22 (CH_2 $\times$ 3), 38.83 (CH), 81.41 (CH), 126.62 (CH $\times$ 2), 127.35 (CH), 128.14 (CH $\times$ 2), 143.75 (C); MS m/z (relative intensity) 383 ($\text{M}^+ - \text{Bu}$, 17), 381 ($\text{M}^+ - 2 - \text{Bu}$, 13), 379 ($\text{M}^+ - 4 - \text{Bu}$, 8.0), 251 (100). Anal. Calcd for $\text{C}_{22}\text{H}_{40}\text{OSn}$: C, 60.15; H, 9.18. Found: C, 60.27; H, 9.32.

(1*S, 2*S**)-3-Dibutyl(trifluoromethanesulfoxy)stannyl-2-methyl-1-phenyl-1-propanol (crude)**

To determine the structure of the hydrostannylation product prior to butylation with BuLi, the concentrated reaction mixture obtained from **1a** and **2a** was subjected to ^1H , ^{13}C , and ^{119}Sn NMR analyses. In the ^1H NMR spectrum, a singlet with an integral value of one proton appeared at 6.74 ppm. Upon treatment of CD_3OD , the signal was changed into a broad singlet at 4.8 ppm. Accordingly, the original singlet at 6.74 ppm can be assigned to the hydroxy proton. Judging from the fact that the

hydroxy proton of *syn*-**3a** appears at 1.82 ppm (*vide supra*), the considerable downfield shift in the product prior to butylation is indicative of the presence of a tin-oxygen coordinate bond. The ^{13}C NMR analysis revealed that one bond coupling constants between ^{119}Sn and ^{13}C nuclei ($^1J(^{119}\text{Sn}-^{13}\text{C})$) are 401 Hz (Sn-Bu) and 417 Hz (Sn-CH₂CHMe-). The reported value for $^1J(^{119}\text{Sn}-^{13}\text{C})$ in Bu₃SnOTf is 376 ppm.¹⁸ The larger coupling constants suggest a five-coordinated bipyramidal arrangement around the tin center. The ^{119}Sn signal (94.4 ppm in CDCl₃) appeared in a higher field compared with that of Bu₃SnOTf (168.4 ppm in CDCl₃).¹⁸ This observation also supports the tin-oxygen coordination. Thus we concluded the hydrostannylation product prior to butylation to be a trialkylstannyl triflate intramolecularly coordinated by a hydroxy group.

Since free CF₃SO₃H could not be observed in the reaction mixture, it is unlikely that the product prior to butylation is a free oxastannacyclopentane. There is a possibility of the formation of an oxastannacyclopentane protonated at the oxygen atom. The ^{119}Sn signal of such a cationic complex is expected to appear in a lower field than that of alkoxytributylstannanes (Bu₃SnOMe, 83 ppm (neat) and 108 ppm in CD₂Cl₂; Bu₃SnOEt (neat), 86 ppm).^{19, 20} Sakurai et al. have demonstrated that the Et₂O complex of tributylstannyl cation has the ^{119}Sn signal at 165 ppm.²¹ Additionally, Yamago, Yoshida, and co-workers have reported that the treatment of Bu₃SnOMe with 0.2 equiv of CF₃SO₃H in CD₂Cl₂ results in a lower field shift of the ^{119}Sn signal (from 108 ppm to 112 ppm).¹⁹ However, the ^{119}Sn signal (94.4 ppm) of the product in CDCl₃ appeared in a higher field compared with that of Bu₃SnOMe in CD₂Cl₂. The above-mentioned values of $^1J(^{119}\text{Sn}-^{13}\text{C})$ are also inconsistent with a four-coordinated tetrahedral arrangement.

The NMR sample of the title compound was prepared by the reaction of **1a** (0.50 mmol) with **2a** (0.55 mmol), concentration of the resultant mixture in vacuo, and addition of CDCl₃ (1.0 mL) to the residue. ^1H NMR (CDCl₃) for the major product δ 0.69 (d, J = 6.8 Hz, 3H), 0.92 (t, J = 7.3 Hz, >6H), 1.15–1.90 (m, >14H), 2.42–2.55 (m, $^3J(\text{Sn}-^1\text{H})$ = 136 Hz, 1H), 4.82 (d, J = 3.6 Hz, 1H), 6.74 (s, 1H), 7.14–7.36 (m, >5H). Integral values of the butyl and phenyl groups were larger than the expected

values due to byproducts and the use of a small excess of **2a**. ^{13}C NMR (CDCl_3) for the major product 13.53 ($\text{CH}_3 \square 2$), 16.71 (CH_3 , $^3J(\text{Sn}-^{13}\text{C}) = 39.1$ Hz), 19.69 ($\text{CH}_2 \square 2$, $^1J(^{119}\text{Sn}-^{13}\text{C}) = 401$ Hz, $^1J(^{117}\text{Sn}-^{13}\text{C}) = 383$ Hz), 21.55 (CH_2 , $^1J(^{119}\text{Sn}-^{13}\text{C}) = 417$ Hz, $^1J(^{117}\text{Sn}-^{13}\text{C}) = 398$ Hz), 26.75 ($\text{CH}_2 \square 2$), 27.47 ($\text{CH}_2 \square 2$), 36.75 (CH , $^2J(\text{Sn}-^{13}\text{C}) = 27.9$ Hz), 79.39 (CH , $^3J(\text{Sn}-^{13}\text{C}) = 17.3$ Hz), 119.05 (CF_3 , $^1J(^{19}\text{F}-^{13}\text{C}) = 318$ Hz), 125.58 ($\text{CH} \square 2$), 127.80 (CH), 128.09 ($\text{CH} \square 2$), 138.30 (C); ^{119}Sn NMR (CDCl_3) for the major product $\square 94.4$.

3-Methyl-4-tributylstannyl-2-butanol (**3b**, *syn:anti* = 84:16)

IR (neat) 3357 (br, OH), 2956, 2925, 1462, 1375 cm^{-1} ; ^1H NMR (CDCl_3) $\square 0.58\text{--}0.95$ (m, 20H), 1.13–1.16 (m, 3H), 1.24–1.61 (m, 13H), 1.65–1.78 (m, 1H), 3.50–3.64 (m, 1H); ^{13}C NMR (CDCl_3) for the major isomer $\square 9.36$ ($\text{CH}_2 \square 3$), 13.14 (CH_2), 13.64 ($\text{CH}_3 \square 3$), 18.36 (CH_3), 19.80 (CH_3), 27.40 ($\text{CH}_2 \square 3$), 29.20 ($\text{CH}_2 \square 3$), 38.27 (CH), 73.79 (CH) for the minor isomer (only well resolved signals) $\square 12.98$ (CH_2), 18.85 (CH_3), 19.34 (CH_3), 38.46 (CH), 73.98 (CH); MS m/z (relative intensity) 321 ($\text{M}^+ - \text{Bu}$, 59), 319 ($\text{M}^+ - 2 - \text{Bu}$, 45), 317 ($\text{M}^+ - 4 - \text{Bu}$, 28), 251 (100). Anal. Calcd for $\text{C}_{17}\text{H}_{38}\text{OSn}$: C, 54.13; H, 10.15. Found: C, 53.96; H, 10.01.

(1*R**, 2*S**)-2,4-Dimethyl-1-tributylstannyl-3-pentanol (*syn-3c*)

IR (neat) 3390 (br, OH), 2956, 2925, 1464 cm^{-1} ; ^1H NMR (CDCl_3) $\square 0.66\text{--}0.96$ (m, 26H), 1.24–1.56 (m, 13H), 1.71–1.93 (m, 2H), 2.94–3.00 (m, 1H); ^{13}C NMR (CDCl_3) $\square 9.44$ ($\text{CH}_2 \square 3$), 13.70 ($\text{CH}_3 \square 3$), 15.32 (CH_2), 17.30 (CH_3), 17.83 (CH_3), 19.60 (CH_3), 27.43 ($\text{CH}_2 \square 3$), 29.24 ($\text{CH}_2 \square 3$), 30.78 (CH),

33.66 (CH), 83.20 (CH); MS m/z (relative intensity) 349 ($M^+ - \text{Bu}$, 44), 347 ($M^+ - 2 - \text{Bu}$, 34), 345 ($M^+ - 4 - \text{Bu}$, 20), 251 (100). Anal. Calcd for $\text{C}_{19}\text{H}_{42}\text{OSn}$: C, 56.31; H, 10.45. Found: C, 56.53; H, 10.68.

(1*R, 2*R**)-2,4-Dimethyl-1-tributylstannyl-3-pentanol (*anti*-3c)**

IR (neat) 3390 (br, OH), 2956, 2925, 1464 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.59–1.00 (m, 26H), 1.24–1.53 (m, 13H), 1.71–1.93 (m, 2H), 2.94–3.01 (m, 1H); ^{13}C NMR (CDCl_3) δ 9.57 ($\text{CH}_2 \times 3$), 11.24 (CH_2), 13.70 ($\text{CH}_3 \times 3$), 17.12 (CH_3), 19.94 (CH_3), 21.04 (CH_3), 27.45 ($\text{CH}_2 \times 3$), 29.25 ($\text{CH}_2 \times 3$), 30.71 (CH), 33.76 (CH), 83.07 (CH); MS m/z (relative intensity) 349 ($M^+ - \text{Bu}$, 100), 347 ($M^+ - 2 - \text{Bu}$, 74), 345 ($M^+ - 4 - \text{Bu}$, 43). Anal. Calcd for $\text{C}_{19}\text{H}_{42}\text{OSn}$: C, 56.31; H, 10.45. Found: C, 56.49; H, 10.62.

2,4,4-Trimethyl-1-tributylstannyl-3-pentanol (3d, *syn:anti* = 97:3)

IR (neat) 3504 (br, OH), 2956, 2924, 1464 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.71–1.01 (m, 29H), 1.24–1.59 (m, 13H), 1.97–2.17 (m, 1H), 3.07 (dd, $J = 7.5, 1.8$ Hz, 0.03H), 3.14 (dd, $J = 5.8, 1.6$ Hz, 0.97H); ^{13}C NMR (CDCl_3) for the major isomer δ 9.39 ($\text{CH}_2 \times 3$), 13.69 ($\text{CH}_3 \times 3$), 17.47 (CH_3), 19.72 (CH_2), 26.89 ($\text{CH}_3 \times 3$), 27.42 ($\text{CH}_2 \times 3$), 29.27 ($\text{CH}_2 \times 3$), 32.28 (CH), 35.79 (C), 85.41 (CH) for the minor isomer (only well resolved signals) δ 9.51 (CH_2), 24.60 (CH_3), 26.74 (CH_3), 84.16 (CH); MS m/z (relative intensity) 363 ($M^+ - \text{Bu}$, 43), 361 ($M^+ - 2 - \text{Bu}$, 36), 359 ($M^+ - 4 - \text{Bu}$, 18), 251 (100). Anal. Calcd for $\text{C}_{20}\text{H}_{44}\text{OSn}$: C, 57.29; H, 10.58. Found: C, 57.02; H, 10.71.

(1*S, 2*S**)-1-(4-Methoxyphenyl)-2-methyl-3-tributylstannyl-1-propanol (*syn*-3e)**

Bp 160 °C (1.4 Torr, bath temp). IR (neat) 3419 (br, OH), 2954, 2925, 1512, 1248 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.57 (dd, $J = 12.8, 10.9$ Hz, 1H), 0.67–0.90 (m, 16H), 0.96 (d, $J = 6.8$ Hz, 3H), 1.19–1.55 (m, 12H), 1.76 (d, $J = 3.1$ Hz, 1H), 1.94–2.12 (m, 1H), 3.81 (s, 3H), 4.35 (dd, $J = 6.3, 3.1$ Hz, 1H), 6.85–6.90 (m, 2H), 7.21–7.26 (m, 2H); ^{13}C NMR (CDCl_3) δ 9.33 (CH_2 $\times$ 3), 13.67 (CH_3 $\times$ 3), 13.99 (CH_2), 18.80 (CH_3), 27.39 (CH_2 $\times$ 3), 29.17 (CH_2 $\times$ 3), 38.58 (CH), 55.20 (CH_3), 80.72 (CH), 113.48 (CH $\times$ 2), 127.70 (CH $\times$ 2), 135.95 (C), 158.79 (C); MS m/z (relative intensity) 413 ($\text{M}^+ - \text{Bu}$, 14), 411 ($\text{M}^+ - 2 - \text{Bu}$, 20), 409 ($\text{M}^+ - 4 - \text{Bu}$, 13), 251 (100). Anal. Calcd for $\text{C}_{23}\text{H}_{42}\text{O}_2\text{Sn}$: C, 58.86; H, 9.02. Found: C, 58.96; H, 8.89.

(1S*, 2R*)-1-(4-Methoxyphenyl)-2-methyl-3-tributylstannyl-1-propanol (*anti*-3e)

Bp 160 °C (1.4 Torr, bath temp). IR (neat) 3452 (br, OH), 2954, 2924, 1512, 1248 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.67 (dd, $J = 12.7, 10.2$ Hz, 1H), 0.71–0.97 (m, 18H), 1.15 (dd, $J = 12.8, 3.8$ Hz, 1H), 1.23–1.58 (m, 12H), 1.84 (d, $J = 3.3$ Hz, 1H), 1.94–2.13 (m, 1H), 3.80 (s, 3H), 4.25 (dd, $J = 7.1, 3.3$ Hz, 1H), 6.84–6.89 (m, 2H), 7.20–7.26 (m, 2H); ^{13}C NMR (CDCl_3) δ 9.54 (CH_2 $\times$ 3), 13.55 (CH_2), 13.70 (CH_3 $\times$ 3), 20.13 (CH_3), 27.43 (CH_2 $\times$ 3), 29.22 (CH_2 $\times$ 3), 38.82 (CH), 55.22 (CH_3), 81.05 (CH), 113.50 (CH $\times$ 2), 127.74 (CH $\times$ 2), 135.94 (C), 158.87 (C); MS m/z (relative intensity) 413 ($\text{M}^+ - \text{Bu}$, 16), 411 ($\text{M}^+ - 2 - \text{Bu}$, 15), 409 ($\text{M}^+ - 4 - \text{Bu}$, 9.4), 251 (100).

(1S*, 2S*)-2-Methyl-1-(1-naphthyl)-3-tributylstannyl-1-propanol (*syn*-3f)

Bp 180 °C (1.4 Torr, bath temp). IR (neat) 3417 (br, OH), 2954, 2923, 1462, 795, 779, cm^{-1} ; ^1H NMR (CDCl_3) δ 0.69–0.97 (m, 20H), 1.19–1.54 (m, 12H), 1.95 (d, $J = 3.3$ Hz, 1H), 2.20–2.50 (m, 1H), 5.26 (dd, $J = 4.8, 3.5$ Hz, 1H), 7.44–7.53 (m, 3H), 7.63 (d, $J = 6.6$ Hz, 1H), 7.78 (d, $J = 8.1$ Hz, 1H), 7.83–7.90 (m, 1H), 8.05–8.10 (m, 1H); ^{13}C NMR (CDCl_3) δ 9.37 ($\text{CH}_2 \times 3$), 13.67 ($\text{CH}_3 \times 3$), 15.41 (CH_2), 17.69 (CH_3), 27.37 ($\text{CH}_2 \times 3$), 29.18 ($\text{CH}_2 \times 3$), 37.47 (CH), 77.64 (CH), 123.36 (CH), 123.71 (CH), 125.20 (CH), 125.29 (CH), 125.68 (CH), 127.61 (CH), 128.86 (CH), 130.71 (C), 133.78 (C), 139.60 (C); MS m/z (relative intensity) 433 ($\text{M}^+ - \text{Bu}$, 13), 431 ($\text{M}^+ - 2 - \text{Bu}$, 11), 429 ($\text{M}^+ - 4 - \text{Bu}$, 6.2), 251 (100). Anal. Calcd for $\text{C}_{26}\text{H}_{42}\text{OSn}$: C, 63.82; H, 8.65. Found: C, 63.97; H, 8.34.

(1S*, 2R*)-2-Methyl-1-(1-naphthyl)-3-tributylstannyl-1-propanol (*anti*-3f)

Bp 180 °C (1.4 Torr, bath temp). IR (neat) 3431 (br, OH), 2954, 2924, 1462, 796, 779 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.66–0.95 (m, 20H), 1.16–1.50 (m, 12H), 2.01 (d, $J = 3.8$ Hz, 1H), 2.30–2.45 (m, 1H), 5.11 (dd, $J = 6.4, 3.9$ Hz, 1H), 7.43–7.59 (m, 4H), 7.77 (d, $J = 8.1$ Hz, 1H), 7.84–7.88 (m, 1H), 8.13–8.17 (m, 1H); ^{13}C NMR (CDCl_3) δ 9.53 ($\text{CH}_2 \times 3$), 12.42 (CH_2), 13.67 ($\text{CH}_3 \times 3$), 21.29 (CH_3), 27.39 ($\text{CH}_2 \times 3$), 29.17 ($\text{CH}_2 \times 3$), 38.19 (CH), 78.16 (CH), 123.65 (CH), 124.07 (CH), 125.20 (CH), 125.38 (CH), 125.71 (CH), 127.83 (CH), 128.86 (CH), 130.85 (C), 133.88 (C), 139.88 (C); Anal. Calcd for $\text{C}_{26}\text{H}_{42}\text{OSn}$: C, 63.82; H, 8.65. Found: C, 63.69; H, 8.27.

(1S*, 2S*)-Methyl 3-Hydroxy-3-phenyl-2-(tributylstannylmethyl)propanoate (*syn*-3g)

IR (neat) 3452 (br, OH), 2954, 2924, 1738 (C=O), 1454, 1165 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.67–0.99 (m, 17H), 1.18–1.56 (m, 12H), 2.90 (ddd, $J = 10.7, 7.6, 4.8$ Hz, 1H), 2.93 (d, $J = 5.6$ Hz, 1H), 3.68 (s, 3H), 4.69 (dd, $J = 7.8, 5.3$ Hz, 1H), 7.26–7.39 (m, 5H); ^{13}C NMR (CDCl_3) δ 8.53 (CH_2), 9.06 (CH_2 $\times$ 3), 13.63 (CH_3 $\times$ 3), 27.27 (CH_2 $\times$ 3), 28.95 (CH_2 $\times$ 3), 50.42 (CH), 51.74 (CH_3), 78.11 (CH), 126.62 (CH $\times$ 2), 127.87 (CH), 128.38 (CH $\times$ 2), 141.99 (C), 176.61 (C); Anal. Calcd for $\text{C}_{23}\text{H}_{40}\text{O}_3\text{Sn}$: C, 57.16; H, 8.34. Found: C, 57.23; H, 8.12.

(1S*, 2R*)-Methyl 3-Hydroxy-3-phenyl-2-(tributylstannylmethyl)propanoate (*anti*-3g)

IR (neat) 3479 (br, OH), 2954, 2924, 1734 (C=O), 1454, 1198 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.63–0.98 (m, 16H), 1.06 (dd, $J = 13.0, 10.9$ Hz, 1H), 1.18–1.48 (m, 12H), 2.71 (d, $J = 3.5$ Hz, 1H), 2.89 (dt, $J = 10.9, 4.9$ Hz, 1H), 3.60 (s, 3H), 4.96 (dd, $J = 4.9, 3.3$ Hz, 1H), 7.23–7.37 (m, 5H); ^{13}C NMR (CDCl_3) δ 5.27 (CH_2), 9.29 (CH_2 $\times$ 3), 13.69 (CH_3 $\times$ 3), 27.35 (CH_2 $\times$ 3), 29.01 (CH_2 $\times$ 3), 50.44 (CH), 51.73 (CH_3), 75.76 (CH), 126.09 (CH $\times$ 2), 127.54 (CH), 128.21 (CH $\times$ 2), 141.67 (C), 176.16 (C).

Phenyl(2'-tributylstannyl)cyclohexyl)methanol (3h)

Bp 180 $^{\circ}\text{C}$ (2.4 Torr, bath temp). IR (neat) 3444 (br, OH), 2925, 2924, 1454, 1070 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.63–0.95 (m, 16H), 1.05–1.56 (m, 15H), 1.63–2.12 (m, 7H), 4.46 (dd, $J = 5.2, 4.0$ Hz, 1H), 7.23–7.38 (m, 5H); ^{13}C NMR (CDCl_3) δ 11.24 (CH_2 $\times$ 3), 13.70 (CH_3 $\times$ 3), 25.57 (CH_2), 26.60 (CH_2),

26.64 (CH₂), 27.73 (CH₂ □ 3), 29.42 (CH₂ □ 3), 31.59 (CH₂), 32.02 (CH), 49.57 (CH), 78.60 (CH), 126.42 (CH □ 2), 127.33 (CH), 128.25 (CH □ 2), 143.83 (C). Anal. Calcd for C₂₅H₄₄OSn: C, 62.64; H, 9.25. Found: C, 62.68; H, 9.32.

3. 2. Hydrostannylation of Homoallyl Alcohols

For the reaction protocol, see the above section.

3-Methyl-1-phenyl-4-tributylstannyl-1-butanol (**5a**, *syn:anti* = **94:6**)

Bp 175 °C (0.9 Torr, bath temp.). IR (neat) 3352 (br, OH), 2954, 2924, 1456 cm⁻¹; ¹H NMR (CDCl₃) □ 0.64–1.15 (m, 20H), 1.22–1.59 (m, 13H), 1.68–1.87 (m, 3H), 4.73–4.80 (m, 1H), 7.26–7.36 (m, 5H); ¹³C NMR (CDCl₃) for the major isomer □ 9.44 (CH₂ □ 3), 13.66 (CH₃ □ 3), 18.34 (CH₂), 24.37 (CH₃), 27.39 (CH₂ □ 3), 28.45 (CH), 29.22 (CH₂ □ 3), 50.37 (CH₂), 72.99 (CH), 125.90 (CH □ 2), 127.46 (CH), 128.41 (CH □ 2), 145.14 (C) for the minor isomer (only well resolved signals) □ 19.25 (CH₂), 23.46 (CH₃), 50.66 (CH₂), 72.82 (CH), 125.79 (CH □ 2), 127.39 (CH), 145.32 (C); MS *m/z* (relative intensity) 397 (M⁺ – Bu, 28), 395 (M⁺ – 2 – Bu, 28), 393 (M⁺ – 4 – Bu, 16), 177 (100). Anal. Calcd for C₂₃H₄₂OSn: C, 60.94; H, 9.34. Found: C, 60.91; H, 9.03.

4-Methyl-5-tributylstannyl-2-pentanol (**5b**, *syn:anti* = **90:10**)

IR (neat) 3377 (br, OH), 2956, 2924, 1462, 1375 cm⁻¹; ¹H NMR (CDCl₃) □ 0.69–1.04 (m, 20H), 1.14–1.59 (m, 18H), 1.77–1.95 (m, 1H), 3.83–3.96 (m, 1H); ¹³C NMR (CDCl₃) for the major isomer □

9.46 (CH₂ □ 3), 13.67 (CH₃ □ 3), 18.49 (CH₂), 23.79 (CH₃), 24.30 (CH₃), 27.41 (CH₂ □ 3), 28.63 (CH), 29.24 (CH₂ □ 3), 50.72 (CH₂), 66.33 (CH) for the minor isomer □ 19.36 (CH₂), 23.50 (CH₃), 24.12 (CH₃), 28.29 (CH), 50.89 (CH₂), 66.19 (CH); MS *m/z* (relative intensity): 335 (M⁺ – Bu, 100), 333 (M⁺ – 2 – Bu, 74), 331 (M⁺ – 4 – Bu, 47). Anal. Calcd for C₁₈H₄₀OSn: C, 55.26; H, 10.31. Found: C, 55.19; H, 10.34.

2,5-Dimethyl-6-tributylstannyl-3-hexanol (5c, *syn:anti* = 90:10)

IR (neat) 3367 (br, OH), 2956, 2923, 1464, 1375 cm⁻¹; ¹H NMR (CDCl₃) □ 0.63–1.16 (m, 26H), 1.18–1.69 (m, 16H), 1.84–2.01 (m, 1H), 3.40–3.54 (m, 1H); ¹³C NMR (CDCl₃) for the major isomer □ 9.49 (CH₂ □ 3), 13.68 (CH₃ □ 3), 16.81 (CH₃), 17.75 (CH₂), 18.89 (CH₃), 24.91 (CH₃), 27.44 (CH₂ □ 3), 28.59 (CH), 29.26 (CH₂ □ 3), 33.83 (CH), 45.55 (CH₂), 74.65 (CH) for the minor isomer (only well resolved signals) □ 17.16 (CH₃), 18.64 (CH₃), 20.12 (CH₂), 23.08 (CH₃), 28.10 (CH), 34.07 (CH), 45.48 (CH₂), 74.51 (CH); MS *m/z* (relative intensity) 363 (M⁺ – Bu, 87), 361 (M⁺ – 2 – Bu, 69), 359 (M⁺ – 4 – Bu, 32), 177 (100). Anal. Calcd for C₂₀H₄₄OSn: C, 57.29; H, 10.58. Found: C, 56.93; H, 10.58.

2,2,5-Trimethyl-6-tributylstannyl-3-hexanol (5d, *syn:anti* = 93:7)

IR (neat) 3417 (br, OH), 2954, 2925, 1464, 1070 cm⁻¹; ¹H NMR (CDCl₃) □ 0.60 (dd, *J* = 12.9, 9.6 Hz, 0.93H), 0.66–0.99 (m, 27.1H), 1.09 (dd, *J* = 12.9, 3.6 Hz, 0.93H), 1.22–1.62 (m, 15H), 1.85–2.02 (m, 1H), 3.22–3.37 (m, 1H); ¹³C NMR (CDCl₃) for the major isomer □ 9.48 (CH₂ □ 3), 13.69 (CH₃ □ 3), 17.14 (CH₂), 25.29 (CH₃), 25.69 (CH₃ □ 3), 27.45 (CH₂ □ 3), 28.91 (CH), 29.27 (CH₂ □ 3), 34.87 (C),

42.94 (CH₂), 77.84 (CH) for the minor isomer (only well resolved signals) δ 20.52 (CH₂), 22.82 (CH₃), 28.28 (CH), 34.74 (C); MS m/z (relative intensity) 377 (M⁺ – Bu, 84), 375 (M⁺ – 2 – Bu, 66), 373 (M⁺ – 4 – Bu, 39), 177 (100). Anal. Calcd for C₂₁H₄₆OSn: C, 58.21; H, 10.70. Found: C, 57.90; H, 10.87.

***cis*-4-Phenyl-2-tributylstannyl-4-butanolide (*cis*-6)**

Bp 169 °C (0.5 Torr, bath temp.). IR (neat) 2954, 2924, 1770 (C=O), 1159 cm⁻¹; ¹H NMR (CDCl₃) δ 0.76–0.98 (m, 16H), 1.20–1.66 (m, 13H), 1.81 (q, J = 11.4 Hz 1H), 2.73–3.03 (m, 2H), 5.32 (dd, J = 10.6, 5.5 Hz, 1H), 7.30–7.42 (m, 5H); ¹³C NMR (CDCl₃) δ 9.60 (CH₂ $\times$ 3), 9.92 (CH₂), 13.68 (CH₃ $\times$ 3), 27.38 (CH₂ $\times$ 3), 29.14 (CH₂ $\times$ 3), 40.15 (CH), 42.03 (CH₂), 78.65 (CH), 125.49 (CH $\times$ 2), 128.41 (CH), 128.69 (CH $\times$ 2), 139.22 (C), 179.99 (C); MS m/z (relative intensity) 409 (M⁺ – Bu, 100), 407 (M⁺ – 2 – Bu, 76), 405 (M⁺ – 4 – Bu, 44). Anal. Calcd for C₂₃H₃₈O₂Sn: C, 59.37; H, 8.23. Found: C, 59.28; H, 8.00.

***trans*-4-Phenyl-2-tributylstannyl-4-butanolide (*trans*-6)**

Bp 169 °C (0.5 Torr, bath temp.). IR (neat) 2954, 2924, 1776 (C=O), 1174, 1025 cm⁻¹; ¹H NMR (CDCl₃) δ 0.76–1.01 (m, 15H), 1.02 (dd, J = 13.2, 9.4 Hz, 1H), 1.21–1.55 (m, 13H), 2.29 (dt, J = 12.7, 7.8 Hz, 1H), 2.42 (ddd, J = 12.6, 8.4, 4.9 Hz, 1H), 2.67–2.91 (m, 1H), 5.56 (dd, J = 7.4, 4.9 Hz, 1H), 7.26–7.42 (m, 5H); ¹³C NMR (CDCl₃) δ 9.54 (CH₂ $\times$ 3), 10.38 (CH₂), 13.68 (CH₃ $\times$ 3), 27.36 (CH₂ $\times$ 3), 29.13 (CH₂ $\times$ 3), 37.24 (CH), 40.34 (CH₂), 78.01 (CH), 124.97 (CH $\times$ 2), 128.13 (CH), 128.72 (CH $\times$ 2), 139.87 (C), 180.70 (C); MS m/z (relative intensity) 409 (M⁺ – Bu, 87), 407 (M⁺ – 2 – Bu, 65), 405 (M⁺ –

4 – Bu, 41), 57 (100).

4. Transformation of Stannylated Products

4. 1. Etherification of Alcohol 3a

N,N-Diisopropylethylamine (0.25 mL, 1.5 mmol) and 2-methoxyethoxymethyl chloride (MEM-Cl, 0.17 mL, 1.5 mmol) were successively added to a solution of *syn*-**3a** (439 mg, 1.00 mmol) in CH₂Cl₂ (2.0 mL) at room temperature. After being stirred for 3 h, the reaction mixture was poured into water (15 mL) and extracted with *t*-BuOMe (3 × 15 mL). The extract was dried over Na₂SO₄ and evaporated. Purification of the crude product by silica gel column chromatography (hexane-AcOEt 15:1) gave MEM ether **9** (485 mg, 0.920 mmol, 92%).

1-[(2-Methoxyethoxy)methoxy]-2-methyl-1-phenyl-3-tributylstannylpropane (**9**)

Bp 235 °C (0.2 Torr, bath temp.); IR (neat) 2954, 2925, 1109, 1076, 1038 cm⁻¹; ¹H NMR (CDCl₃) δ 0.49 (dd, *J* = 12.7, 11.4 Hz, 1H), 0.58–0.90 (m, 16H), 1.02 (d, *J* = 6.2 Hz, 3H), 1.17–1.51 (m, 12H), 2.00–2.17 (m, 1H), 3.36 (s, 3H), 3.41–3.58 (m, 3H), 3.78–3.86 (m, 1H), 4.24 (d, *J* = 7.3 Hz, 1H), 4.57 (d, *J* = 6.9 Hz, 1H), 4.63 (d, *J* = 6.9 Hz, 1H), 7.24–7.65 (m, 5H); ¹³C NMR (CDCl₃) δ 8.91 (CH₂ $\times$ 3), 13.64 (CH₃ $\times$ 3), 13.74 (CH₂), 19.88 (CH₃), 27.33 (CH₂ $\times$ 3), 29.11 (CH₂ $\times$ 3), 37.81 (CH), 58.92 (CH₃), 66.96 (CH₂), 71.68 (CH₂), 85.31 (CH), 93.49 (CH₂), 127.27 (CH), 127.87 (CH $\times$ 4), 141.03 (C); MS *m/z* (relative intensity) 471 (M⁺ – Bu, 21), 469 (M⁺ – 2 – Bu, 16), 467 (M⁺ – 4 – Bu, 8.1), 309 (100). Anal. Calcd for C₂₆H₄₈O₃Sn: C, 59.21; H, 9.17. Found: C, 59.20; H, 9.02.

4. 2. Transmetalation of Ether **9** Followed by Addition to Benzaldehyde

BuLi (1.58 M in hexane, 0.63 mL, 1.00 mmol) was dropwise added to a solution of **9** (264 mg, 0.500 mmol) in THF (1.0 mL) at $-25\text{ }^{\circ}\text{C}$. After being stirred for 6 h, benzaldehyde (0.10 mL, 1.00 mmol) was added to the reaction mixture. After 1 h the resultant mixture was warmed to room temperature and stirred for 0.5 h. The reaction was quenched with saturated aqueous NH_4Cl (10 mL). The extract with AcOEt (3 $\times$ 15 mL) was dried over Na_2SO_4 and evaporated. Purification of the residual oil by silica gel column chromatography (hexane-AcOEt 2:1) gave **11** (124 mg, 0.360 mmol, 72%, *dr* = 61:39).

4-[(2-Methoxyethoxy)methoxy]-3-methyl-1,4-diphenyl-1-butanol (**10**, *dr* = 61:39)

Bp $240\text{ }^{\circ}\text{C}$ (0.2 Torr, bath temp.); IR (neat) 3438 (br, OH), 2933, 2833, 1454, 1105, 1039 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.95 (d, $J = 6.8\text{ Hz}$, 1.2H), 0.97 (d, $J = 6.4\text{ Hz}$, 1.8H), 1.42–1.67 (m, 2H), 1.78–1.97 (m, 1H), 2.10–2.13 (m, 0.6H), 2.32–2.35 (m, 0.4H), 3.348 and 3.352 (2 $\times$ s, 3H), 3.40–3.61 (m, 3H), 3.78–3.95 (m, 1H), 4.54–4.80 (m, 4H), 7.17–7.34 (m, 10H); ^{13}C NMR (CDCl_3) for the major isomer δ 15.95 (CH_3), 35.97 (CH), 41.66 (CH_2), 58.79 (CH_3), 67.06 (CH_2), 71.52 (CH_2), 72.53 (CH), 81.81 (CH), 93.39 (CH_2), 125.95 (CH $\times$ 2), 127.22 (CH), 127.31 (CH), 127.43 (CH $\times$ 2), 128.23 (CH $\times$ 4), 140.28 (C), 144.24 (C) for the minor isomer δ 14.98 (CH_3), 36.35 (CH), 42.53 (CH_2), 58.79 (CH_3), 67.14 (CH_2), 71.59 (CH_2), 71.96 (CH), 81.76 (CH), 93.55 (CH_2), 125.71 (CH $\times$ 2), 127.14 (CH), 127.17 (CH), 127.31 (CH $\times$ 2), 127.88 (CH $\times$ 4), 140.23 (C), 145.20 (C); MS m/z (relative intensity) for the major isomer 255 ($\text{M}^+ - \text{C}_4\text{H}_9\text{O}_2$, 0.4), 59 (100). for the minor isomer 255 ($\text{M}^+ - \text{C}_4\text{H}_9\text{O}_2$, 0.3), 59 (100). Anal. Calcd for $\text{C}_{21}\text{H}_{28}\text{O}_4$: C, 73.23; H, 8.19. Found: C, 73.07; H, 8.27.

4. 3. Cyclopropanation of Alcohols **3** (Typical Procedure)

Pyridine (0.29 mL, 3.6 mmol) and SOCl_2 (>90% pure, 0.15 mL, >1.8 mmol) were successively added to a solution of *syn*-**3a** (395 mg, 0.900 mmol) in THF (9.0 mL) at room temperature. After being stirred for 10 h, the reaction mixture was treated with saturated aqueous NaHCO_3 (10 mL). The resultant mixture was extracted with hexane (3 $\times$ 10 mL). The extract was washed with brine, dried over Na_2SO_4 , and evaporated. Purification of the residual oil by silica gel column chromatography (hexane) gave cyclopropane *trans*-**11a** (101 mg, 0.764 mmol, 85%).

***trans*-1-Methyl-2-phenylcyclopropane (*trans*-11a)** [5070-01-9]²²

For the characterization data for *trans*-**11a**, see ref. 22a.

***trans*-1-(4-Methoxyphenyl)-2-methylcyclopropane (*trans*-11e)** [77525-88-3]²³

IR (neat) 2952, 1516, 1248, 1038 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.67 (dt, $J = 8.6, 4.8$ Hz, 1H), 0.80 (dt, $J = 8.2, 4.8$ Hz, 1H), 0.90–1.03 (m, 1H), 1.16 (d, $J = 5.8$ Hz, 3H), 1.52 (dt, $J = 8.4, 4.8$ Hz, 1H), 3.77 (s, 3H), 6.77–6.81 (m, 2H), 6.94–6.98 (m, 2H); ^{13}C NMR (CDCl_3) δ 16.94 (CH_2), 17.27 (CH), 19.08 (CH_3), 23.50 (CH), 55.31 (CH_3), 113.70 (CH $\times$ 2), 126.53 (CH $\times$ 2), 136.01 (C), 157.40 (C); MS m/z (relative intensity) 162 (M^+ , 70), 147 ($\text{M}^+ - \text{Me}$, 100). These ^1H NMR data for *trans*-**11e** agree with those reported in ref. 19.

***trans*-1-Methyl-2-(1-naphthyl)cyclopropane (*trans*-11f)**

IR (neat) 2951, 1594, 1508, 796, 775 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.75–0.85 (m, 1H), 0.98–1.12 (m, 2H), 1.34–1.40 (m, 3H), 2.00–2.10 (m, 1H), 7.20–7.25 (m, 1H), 7.33–7.39 (m, 1H), 7.45–7.57 (m, 2H), 7.68 (d, $J = 8.2$ Hz, 1H), 7.82–7.86 (m, 1H), 8.30–8.33 (m, 1H); ^{13}C NMR (CDCl_3) δ 14.34 (CH_2), 15.30 (CH), 19.25 (CH_3), 21.96 (CH), 123.49 (CH), 124.48 (CH), 125.47 (CH), 125.50 (CH), 125.67 (CH), 126.32 (CH), 128.45 (CH), 133.45 (C), 133.54 (C), 139.31 (C); MS m/z (relative intensity) 182 (M^+ , 59), 167 ($\text{M}^+ - \text{Me}$, 100). Anal. Calcd for $\text{C}_{14}\text{H}_{14}$: C, 92.26; H, 7.74. Found: C, 91.89; H, 7.50.

***trans*-2-Phenylcyclopropanecarboxylate (*trans*-**11g**)** [5861-31-4]²⁴

For the characterization data for *trans*-**11g**, see ref. 24.

5. Stereochemical Assignment of Stannylated Products

5. 1. $\square$ -Stannylated Alcohols

The major isomer of **3g** was converted into diol **12** by reduction with LiAlH_4 (Scheme S1). The acid-catalyzed reaction of **12** with 2,2-dimethoxypropane gave acetonide **13** in high yield. ^1H NMR analysis of **13** showed that the coupling constant between protons on the stereogenic centers (J_{ab}) is 10.1 Hz, which is corresponding to an axial-axial coupling. The spectroscopic result disclosed that **13** has *trans* configuration, and that **12** and the major isomer of **3g** have *syn* configuration. The ^1H - ^1H coupling constant between the stereogenic centers in the major isomer of **3g** (7.7 Hz) is larger than that in the minor isomer (4.9 Hz). Additionally, the proton $\square$ to the hydroxy group in the major isomer (4.69 ppm)

appears in a higher field than that in the major isomer (4.96 ppm). These facts also support the *syn* configuration ((1*S**, 2*S**)-configuration) of the major isomer of **3g**.²⁵

Site-selective tosylation of *syn*-**12** followed by reduction with LiAlH₄ afforded the major isomer of **3a** in good yield. This transformation proved the major isomer of **3a** to be *syn*.

Scheme S1

The cyclopropanation reaction of β -stannylated alcohols via 1,3-elimination (Scheme 3 in the text) is known to proceed with double inversion at both reaction sites.^{26,27} Accordingly, the formation of *trans*-**10** indicates that the major isomers of **3a,e-g** have *syn* configuration. The major isomers of **3b-d** were assigned to *syn* configuration by analogy with the stereochemical outcomes of the hydrostannylation of **2a,e-g**.

To determine the relative configuration of **3h**, the reaction mixture obtained from **1a** and **2h** was subjected to Bu₄NF, KHCO₃, and H₂O₂ (Scheme S2).²⁸ This oxidation gave a mixture containing the corresponding 1,3-diol. After the TsOH-catalyzed reaction of the mixture with 2,2-dimethoxypropane, acetonide **14** was isolated as a single isomer but in a low total yield (15%) from **2h**. The formation of other isomers of **14** was not observed. The acetonide was determined to have (1*S**, 5*S**, 6*R**)-configuration (4,5-*cis*-5,6-*cis*-configuration with respect to the 1,3-dioxane ring) by comparison with the authentic sample prepared from the *syn*-aldol obtained from cyclohexanone and benzaldehyde. Since

oxidative cleavage of tin-carbon bond is known to proceed with stereochemical retention, **3h** is likely to have (1*S**, 1'*S**, 2'*S**)-configuration (*syn,syn*-configuration) (Scheme S2).

Scheme S2

(1*S, 2*S**)-1-Phenyl-2-tributylstannylmethyl-1,3-propanediol (**12**)**

A solution of the major isomer of **3g** (522 mg, 1.08 mmol) in Et₂O (2.0 mL) was dropwise added to a suspension of LiAlH₄ (>92% pure, 62 mg, >1.50 mmol) in Et₂O (3.0 mL) at 0 °C. After being stirred for 2 h, the reaction mixture was poured into water (20 mL) and extracted with *t*-BuOMe (3 × 10 mL). The extract was dried over Na₂SO₄ and evaporated. Purification of the crude product by silica gel column chromatography (hexane-AcOEt 3:1) gave the title compound (419 mg, 0.920 mmol, 85%). IR (neat) 3336 (br, OH), 2954, 2924, 1454, 1022 cm⁻¹; ¹H NMR (CDCl₃) δ 0.49–0.89 (m, 17H), 1.18–1.50 (m, 12H), 2.08–2.20 (m, 1H), 2.83 (br s, 1H), 2.90 (dd, *J* = 6.6, 3.8 Hz, 1H), 3.64 (ddd, *J* = 10.9, 7.1, 3.7 Hz,

1H), 3.74 (ddd, $J = 10.9, 7.2, 3.0$ Hz, 1H), 4.57 (dd, $J = 7.6, 3.0$ Hz, 1H), 7.29–7.40 (m, 5H); ^{13}C NMR (CDCl_3) δ 7.76 (CH_2), 9.29 (CH_2 $\times$ 3), 13.64 (CH_3 $\times$ 3), 27.30 (CH_2 $\times$ 3), 29.04 (CH_2 $\times$ 3), 44.57 (CH), 68.02 (CH_2), 82.12 (CH), 126.74 (CH $\times$ 2), 127.64 (CH), 128.32 (CH $\times$ 2), 143.60 (C).

Conversion of Diol **12** into Stannylated Alcohol *syn*-**3a**

BuLi (1.72 M in hexane, 0.55 mL, 0.95 mmol) was dropwise added to a solution of **12** (419 mg, 0.920 mmol) in Et_2O (3 mL) at 0 °C. After 20 min a solution of TsCl (181 mg, 0.95 mmol) was added. The reaction was carried out at room temperature for 2 h. The reaction mixture was cooled to 0 °C again. After addition of LiAlH_4 (>92% pure, 36 mg, >0.87 mmol), the reaction mixture was warmed to room temperature and stirred for another 2 h. The resultant mixture was subjected to an aqueous work up as shown in the preparation of **12**. Purification of the crude product by silica gel column chromatography (hexane-AcOEt 10:1) gave *syn*-**3a** (351 mg, 0.799 mmol, 87%).

trans-2,2-Dimethyl-4-phenyl-5-tributylstannylmethyl-1,3-dioxane (**13**)

2,2-Dimethoxypropane (0.69 mL, 5.6 mmol) and $\text{TsOH} \cdot \text{H}_2\text{O}$ (10.7 mg, 0.056 mmol) were successively added to a solution of **12** (255 mg, 0.560 mmol) in DMF (1.0 mL) at room temperature. After being stirred for 2 h, the resultant mixture was subjected to an aqueous work up as shown in the preparation of **12**. Purification of the crude product by silica gel column chromatography (hexane-AcOEt 20:1) gave the title compound (257 mg, 0.519 mmol, 93%). IR (neat) 2956, 2925, 1377, 1198, 1109 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.26 (dd, $J = 13.2, 11.4$ Hz, 1H), 0.51 (dd, $J = 13.2, 3.0$ Hz, 1H), 0.62–0.87 (m, 15H) including 0.84 (t, $J = 7.2$ Hz), 1.15–1.43 (m, 12H), 1.48 (s, 3H), 1.56 (s, 3H), 1.92–2.08

(m, 1H), 3.61 (dd, $J = 11.6, 11.2$ Hz, 1H), 3.83 (dd, $J = 11.6, 4.9$ Hz, 1H), 4.38 (d, $J = 10.1$ Hz, 1H), 7.25–7.40 (m, 5H); ^{13}C NMR (CDCl_3) δ 6.28 (CH_2), 9.30 (CH_2 $\times$ 3), 13.62 (CH_3 $\times$ 3), 19.04 (CH_3), 27.27 (CH_2 $\times$ 3), 29.00 (CH_2 $\times$ 3), 29.89 (CH_3), 39.19 (CH), 67.25 (CH_2), 80.77 (CH), 98.62 (C), 127.82 (CH $\times$ 2), 127.96 (CH), 128.25 (CH $\times$ 2), 140.40 (C); MS m/z (relative intensity) 439 ($\text{M}^+ - \text{Bu}$, 1.7), 437 ($\text{M}^+ - 2 - \text{Bu}$, 1.6), 435 ($\text{M}^+ - 4 - \text{Bu}$, 0.8), 351 (100). Anal. Calcd for $\text{C}_{25}\text{H}_{44}\text{O}_2\text{Sn}$: C, 60.62; H, 8.95. Found: C, 60.54; H, 9.21.

Hydrostannylation of Allyl Alcohol **2h** Followed by Oxidation and Protection

According to the typical procedure described above (Section 3.1), the hydrostannylation of **2h** (1.00 mmol) was performed at -78 °C for 6 h. The resultant mixture was warmed to room temperature and concentrated under reduced pressure. Bu_4NF (1.0 M in THF, 4.0 mL, 4.0 mmol), KHCO_3 (200 mg, 2.0 mmol), MeOH (2 mL), and H_2O_2 (35% in H_2O , 1.0 mL, 10 mmol) were added to the residue. The oxidation reaction was performed at 40 °C for 24 h. The reaction mixture was poured into water. The extract with *t*-BuOMe was dried over MgSO_4 and evaporated. Purification of the crude product by silica gel column chromatography (hexane-AcOEt 1:1) gave a mixture of the desired 1,3-diol and unidentified impurities (58 mg). The impure 1,3-diol was converted into acetonide **14** (36 mg, 15% yield from **2h**) by the same procedure that be used for the synthesis of **13** (2,2-dimethoxypropane (0.35 mL), $\text{TsOH} \cdot \text{H}_2\text{O}$ (2.7 mg), and DMF (5 mL)).

(1S*, 5S*, 6R*)-3,3-Dimethyl-5-phenyl-2,4-dioxabicyclo[4.4.0]decane (14) [162660-49-3]²⁹

IR (CDCl_3) 1254, 1200, 1189, 1164, 1012 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.79–0.85 (m, 1H), 0.95–1.10

(m, 1H), 1.35–1.69 (m, 12H), 1.82–1.91 (m, 1H), 4.26 (br s, 1H), 5.06 (d, $J = 2.0$ Hz, 1H), 7.19–7.36 (m, 5H); ^{13}C NMR (CDCl_3) δ 19.09 (CH_2), 19.63 (CH_3), 20.16 (CH_2), 25.20 (CH_2), 30.07 (CH_3), 31.88 (CH_2), 40.61 (CH), 68.03 (CH), 74.11 (CH), 98.92 (C), 125.57 ($\text{CH} \times 2$), 126.70 (CH), 127.94 ($\text{CH} \times 2$), 140.72 (C); MS m/z (relative intensity) 231 ($\text{M}^+ - \text{Me}$, 6.9), 59 (100).

Synthesis of Authentic Sample of **14**

Under the atmosphere, a MeOH (1 mL) solution of the *syn*-aldol (109 mg, 0.534 mmol) derived from benzaldehyde and cyclohexanone was added to a suspension of NaBH_4 (80 mg, 2.1 mmol) in MeOH (2 mL) at room temperature. After being stirred for 30 min, the reaction mixture was poured into saturated aqueous NH_4Cl and extracted with *t*-BuOMe. The extract was dried over Na_2SO_4 and evaporated. Purification of the crude product by silica gel column chromatography (hexane-AcOEt 2:1) gave the corresponding *syn,syn*-1,3-diol (81 mg, 0.39 mmol) in 74% yield. The 1,3-diol was converted into acetone **14** (72 mg, 75%) by the same procedure that be used for the synthesis of **13** (2,2-dimethoxypropane (0.50 mL), $\text{TsOH} \cdot \text{H}_2\text{O}$ (2 mg), and DMF (2 mL)).

5. 2. α -Stannylated Alcohols

The stereochemical assignment of **6** is based on the results of ^1H NMR analysis and NOE experiments (Scheme S3). In the major isomer of **6**, the methylene proton H_b is much more shielded than the geminal proton H_c . Such a significant difference between the methylene protons in chemical shift was not observed in the minor isomer. The strong shielding of H_b indicates that the phenyl group, H_b , and the stannylmethyl group exist on the same side of the lactone ring in the major isomer.³⁰ In addition, NOE experiments of the minor isomer proved the *trans* relationship between the phenyl and stannylmethyl groups.

Reduction of *cis*-**6** with LiAlH_4 followed by site-selective deoxygenation gave the major isomer of

5a. This transformation disclosed that the major isomer of **5a** has *syn* configuration. The low yield of **5a** was due to the formation of disubstituted tetrahydrofuran **16**.

Scheme S3

(1*S, 3*S**)-1-Phenyl-3-tributylstannylmethyl-1,4-butanediol (15)**

The title compound was obtained from the major isomer of **6** in 88% yield. The reduction of **6** was carried out by the same procedure as used for the reduction of **3g**. IR (neat) 3273 (br, OH), 2954, 2924, 1454, 1026 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.61–0.92 (m, 17H), 1.21–1.50 (m, 12H), 1.68 (ddd, $J = 14.5, 4.0, 3.2$ Hz, 1H), 1.84 (ddd, $J = 14.5, 9.4, 8.1$ Hz, 1H), 1.97–2.11 (m, 1H), 3.03 (br s, 2H), 3.44 (dd, $J = 10.6, 7.9$ Hz, 1H), 3.65 (dd, $J = 10.6, 3.8$ Hz, 1H), 4.78 (dd, $J = 9.5, 2.9$ Hz, 1H), 7.22–7.38 (m, 5H); ^{13}C NMR (CDCl_3) δ 9.43 (CH_2 $\times$ 3), 13.65 (CH_3 $\times$ 3), 27.36 (CH_2 $\times$ 3), 29.14 (CH_2 $\times$ 3), 38.86 (CH), 47.97 (CH_2), 69.76 (CH_2), 74.03 (CH), 125.57 (CH $\times$ 2), 127.27 (CH), 128.34 (CH $\times$ 2), 145.37 (C).

Conversion of Diol **15** into Stannylated Alcohol *syn*-**5a**

Site-selective deoxygenation of **15** was carried out by the same procedure as used for the conversion of **12** into *syn*-**3a**. The desired product, *syn*-**5a**, was obtained in only 11% yield. Tetrahydrofuran *cis*-**16** was formed as the major product (53%).

cis-2-Phenyl-4-(tributylstannylmethyl)tetrahydrofuran (**16**)

IR (neat) 2956, 2924, 1456, 1041 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.71–1.03 (m, 17H), 1.23–1.53 (m, 13H), 2.40–2.69 (m, 2 H), 3.51 (dd, $J = 8.8, 8.0$ Hz, 1H), 4.10 (t, $J = 7.6$ Hz, 1H), 4.94 (dd, $J = 10.1, 5.8$ Hz, 1H), 7.20–7.34 (m, 5H); ^{13}C NMR (CDCl_3) δ 9.20 (CH_2 $\times$ 3), 11.68 (CH_2), 13.67 (CH_3 $\times$ 3), 27.38 (CH_2 $\times$ 3), 29.18 (CH_2 $\times$ 3), 39.34 (CH), 46.11 (CH_2), 77.06 (CH_2), 81.67 (CH), 125.57 (CH $\times$ 2), 127.10 (CH), 128.28 (CH $\times$ 2), 143.53 (C); MS m/z (relative intensity) 395 ($\text{M}^+ - \text{Bu}$, 100), 393 ($\text{M}^+ - 2 - \text{Bu}$, 62), 391 ($\text{M}^+ - 4 - \text{Bu}$, 45). Anal. Calcd for $\text{C}_{23}\text{H}_{40}\text{OSn}$: C, 61.21; H, 8.93. Found: C, 61.16; H, 9.00.

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