**One-Pot Asymmetric Synthesis of Acyclic Chiral Epoxy Alcohols via
Tandem Vinylation-Epoxidation with Dioxygen**

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General Methods. All reactions were carried out under a nitrogen atmosphere with oven-dried glassware. The progress of all reactions was monitored by thin-layer chromatography to ensure the reactions had reached completion. All manipulations involving dicyclohexylborane and dialkylzinc reagents were carried out using an inert atmosphere in a drybox or by using standard Schlenk or vacuum line techniques. Dichloromethane and hexanes were dried through alumina columns. All aldehydes were distilled or recrystallized and sublimed prior to use and stored under N₂. 230-400 mesh silica gel was used for air-flashed chromatography. The ¹H NMR and ¹³C{¹H} NMR spectra were obtained on an AM-500 Fourier transform NMR spectrometer at 500 and 125 MHz, respectively. Chemical shifts are reported in units of parts per million downfield from tetramethylsilane, and all coupling constants are reported in Hertz. Analysis of enantiomeric excess was performed using chiral HPLC with a Chiralcel OD-H column or by chiral capillary gas chromatography with a Beta-DEX column. Absolute configuration was determined by comparison of optical rotation to literature data for known compounds.

(S)-1-(2-Bromo-phenyl)-hept-2-en-1-ol (4). General

Procedure A. A 10 mL Schlenk flask was charged with Cy₂BH (98 mg, 0.55 mmol), prepared according to Oppolzer's procedure,¹ and 1.5 mL of hexanes. 1-Hexyne (64 μ L, 0.55 mmol) was added dropwise, and the homogenous reaction mixture was stirred for 30 min at room temp. After cooling the reaction flask to -10 °C, (-)-MIB (4.8 mg, 0.02 mmol) was added, followed by Et₂Zn (0.55 mL, 2.0 M), then *o*-bromobenzaldehyde (58 μ L, 0.50 mmol) dropwise. The reaction was stirred at -10 °C for 2 h and quenched with 2 mL H₂O. The organic and aqueous layers were separated, and the aqueous layer was extracted with hexanes (3 X 5 mL). The combined organic layers were then washed with 5 mL H₂O and dried over MgSO₄. The filtrate was concentrated *in vacuo*, and the residue was chromatographed on silica (5% ethyl acetate in hexanes) to afford the title compound in 77% yield (104 mg, 0.39 mmol) as a colorless oil. [α]_D²⁰ = -31.3 (*c* = 3.2, CHCl₃, 93% ee); ¹H NMR (CDCl₃, 500 MHz): δ 0.86 (t, 3H, *J* = 7.1 Hz), 1.25-1.37 (m, 4H), 2.03 (dt, 2H, *J* = 7.0, 6.7 Hz), 2.11 (br s, 1H), 5.51 (d, 1H, *J* = 6.1 Hz), 5.58 (dd, 1H, *J* = 15.2, 6.4 Hz), 5.78 (dt, 1H, *J* = 15.2, 6.7 Hz), 7.10 (t, 1H, *J* = 7.6 Hz), 7.31 (t, 1H, *J* = 7.4 Hz), 7.50 (d, 1H, *J* = 7.9 Hz), and 7.53 (d, 1H, *J* = 7.7 Hz) ppm; ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 13.9, 22.2, 31.3, 31.9, 73.5, 122.4, 127.70, 127.74, 128.8, 130.2, 132.7, 133.5, and 142.2 ppm; IR (neat): 3360, 3060, 2956, 2926, 2857, 1664, 1567, 1466, 1438, 1378, and 1193 cm⁻¹; HRMS-Cl *m/z* 268.0471 [*M*⁺; calcd for C₁₃H₁₇BrO: 268.0463].

(S)-4,4-Dimethyl-1-phenyl-pent-2-en-1-ol (1). The product was prepared by General Procedure A using 1.07 g (6.0 mmol) Cy₂BH, 739 μ L (6.0 mmol) 2,2-dimethyl-1-butyne, 3.0 mL (6.0 mmol, 2.0 M in toluene) Me₂Zn, 24.0 mg (0.1 mmol) (-)-MIB, and 510 μ L (5.0 mmol) benzaldehyde. The crude product was purified by column chromatography on silica (20% ethyl acetate in hexanes) to afford the title compound as a colorless oil in 86% yield (819 mg, 4.3 mmol). [α]_D²⁰ = +40.6 (*c* = 1.00, CHCl₃, 96% ee); ¹H NMR (CDCl₃, 500 MHz): δ 1.14 (s, 9H), 2.54 (s, 1H), 5.19 (d, 1H, *J* = 6.8 Hz), 5.65 (dd, 1H, *J*

= 15.4, 6.9 Hz), 5.86 (d, 1H, J = 15.9 Hz), and 7.34-7.45 (m, 5H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 30.0, 33.3, 75.7, 126.7, 127.8, 128.7, 128.9, 143.7, and 144.1 ppm; IR (neat): 3346, 3086, 3063, 3029, 2959, 2902, 2866, 1494, 1476, and 1453 cm^{-1} ; HRMS-CI m/z 190.1356 [M^+ ; calcd for $\text{C}_{13}\text{H}_{18}\text{O}$: 190.1358].

(S)-1-Phenyl-hept-2-en-1-ol (2). The product was prepared by General Procedure A using 2.14 g (12.0 mmol) Cy_2BH , 1.4 mL (12.0 mmol) 1-hexyne, 6 mL (12.0 mmol, 2.0 M in toluene) Me_2Zn , 48 mg (0.2 mmol) (–)-MIB, and 1.01 mL (10.0 mmol) benzaldehyde. The crude product was purified by column chromatography on silica (5% ethyl acetate in hexanes) to afford the title compound as a colorless oil in 85% yield (1.62 g, 8.52 mmol). $[\alpha]_{\text{D}}^{20}$ = +38.3 (c = 1.01, CHCl_3 , 95% ee); ^1H NMR (CDCl_3 , 500 MHz): δ 0.85 (t, 3H, J = 7.1 Hz), 1.22-1.37 (m, 4H), 1.81 (br s, 1H), 2.02 (dd, 2H, J = 7.0, 6.5 Hz), 5.13 (d, 1H, J = 6.6 Hz), 5.63 (dd, 1H, J = 15.3, 6.7 Hz), 5.73 (dt, 1H, J = 15.3, 6.6 Hz), and 7.22-7.34 (m, 5H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 13.9, 22.2, 31.2, 31.8, 75.1, 126.1, 127.3, 128.4, 132.2, 132.7, and 143.4 ppm; IR (neat): 3354, 3068, 3027, 2926, 1667, and 1454 cm^{-1} ; HRMS-CI m/z 190.1350 [M^+ ; calcd for $\text{C}_{13}\text{H}_{18}\text{O}$: 190.1358].

(S)-1-(2-Bromo-phenyl)-4,4-dimethyl-pent-2-en-1-ol (3). The product was prepared by General Procedure A using 50 mg (0.28 mmol) Cy_2BH , 35 μL (0.28 mmol) 3,3-dimethyl-1-butyne, 0.26 mL (0.53 mmol, 2.0 M in hexanes) Et_2Zn , 2.4 mg (0.01 mmol) (–)-MIB, and 29 μL (0.25 mmol) *o*-bromobenzaldehyde. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound as a colorless oil in 76% yield (51 mg, 0.19 mmol). $[\alpha]_{\text{D}}^{20}$ = –31.8 (c = 0.33, CHCl_3 , 93% ee); ^1H NMR (CDCl_3 , 500 MHz): δ 1.02 (s, 9H), 2.03 (s, 1H), 5.46-5.53 (m, 2H), 5.86 (d, 1H, J = 15.0 Hz), 7.13 (t, 1H, J = 7.6 Hz), 7.33 (t, 1H, J = 7.5 Hz), 7.52 (d, 1H, J = 8.9 Hz), and 7.54 (d, 1H, J = 8.2 Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 29.4, 33.0, 73.8, 122.6, 125.0, 127.7, 127.8, 128.9, 132.8, 142.3, and 144.3 ppm; IR (neat): 3353, 3064, 3013, 2958, 2865, 1721, 1661, 1590, 1568, 1465, 1439, 1363, and 1267 cm^{-1} ; HRMS-CI m/z 251.0432 [$(\text{M} - \text{OH})^+$; calcd for $\text{C}_{13}\text{H}_{16}\text{Br}$: 251.0435].

(S)-4,4-Dimethyl-1-(4-trifluoromethyl-phenyl)-pent-2-en-1-ol (5). The product was prepared by General Procedure A using 98 mg (0.55 mmol) Cy_2BH , 68 μL (0.55 mmol) 3,3-dimethyl-1-butyne, 0.55 mL (1.1 mmol, 2.0 M in hexanes) Et_2Zn , 4.8 mg (0.02 mmol) (–)-MIB, and 68 μL (0.50 mmol) *p*-trifluoromethylbenzaldehyde. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound as a white solid in 89% yield (115 mg, 0.45 mmol). m.p.: 30-32 $^{\circ}\text{C}$; $[\alpha]_{\text{D}}^{20}$ = –10.0 (c = 0.27, CHCl_3 , 95% ee); ^1H NMR (CDCl_3 , 500 MHz): δ 0.98 (s, 9H), 2.03 (br s, 1H), 5.15 (d, 1H, J = 7.0 Hz), 5.47 (dd, 1H, J = 15.5, 7.2 Hz), 5.78 (d, 1H, J = 15.6 Hz), 7.43 (d, 2H, J = 8.0 Hz), and 7.55 (d, 2H, J = 8.0 Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 29.3, 33.0, 74.9, 123.1, 125.28, 125.31, 125.34, 126.4, 126.7, 129.4, 129.7, 144.6, and 147.3

ppm; IR (film): 3335, 3025, 2962, 2905, 2868, 1661, 1620, 1477, 1463, 1414, 1364, and 1327 cm^{-1} ; HRMS-CI m/z 239.1247 [$(M - F)^+$; calcd for $\text{C}_{14}\text{H}_{17}\text{F}_2\text{O}$: 239.1247].

(S)-1-(4-Trifluoromethyl-phenyl)-hept-2-en-1-ol (6).

The product was prepared by General Procedure A using 98 mg (0.55 mmol) Cy_2BH , 64 μL (0.55 mmol) 1-hexyne, 0.55 mL (1.1 mmol, 2.0 M in hexanes) Et_2Zn , 4.8 mg (0.02 mmol) (-)-MIB, and 68 μL (0.50 mmol) *p*-trifluoromethylbenzaldehyde. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound as a colorless oily solid in 94% yield (121 mg, 0.47 mmol). $[\alpha]_{\text{D}}^{20} = +69.3$ ($c = 0.88$, CHCl_3 , 95% ee); ^1H NMR (CDCl_3 , 500 MHz): δ 0.88 (t, 3H, $J = 7.1$ Hz), 1.25-1.39 (m, 4H), 1.61 (br s, 1H), 2.05 (dt, 2H, $J = 6.9, 6.7$ Hz), 5.19 (d, 1H, $J = 7.0$ Hz), 5.59 (dd, 1H, $J = 15.3, 7.1$ Hz), 5.77 (dt, 1H, $J = 15.3, 6.7$ Hz), 7.46 (d, 2H, $J = 8.0$ Hz), and 7.58 (d, 2H, $J = 8.0$ Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 13.9, 22.2, 31.3, 31.8, 74.7, 125.3, 125.4, 126.4, 129.4, 129.7, 131.7, 133.9, and 147.2 ppm; IR (neat): 3370, 3037, 2959, 2930, 2859, 2353, 1666, 1620, 1415, 1327, and 1165 cm^{-1} ; HRMS-ESI m/z 258.1239 [M^+ ; calcd for $\text{C}_{14}\text{H}_{17}\text{F}_3\text{O}$: 258.1232].

(S)-1-(4-Fluoro-phenyl)-4,4-dimethyl-pent-2-en-1-ol (7).

The product was prepared by General Procedure A using 98 mg (0.55 mmol) Cy_2BH , 68 μL (0.55 mmol) 3,3-dimethyl-1-butyne, 0.55 mL (1.1 mmol, 2.0 M in hexanes) Et_2Zn , 4.8 mg (0.02 mmol) (-)-MIB, and 54 μL (0.50 mmol) *p*-fluorobenzaldehyde. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound as a white solid in 82% yield (85 mg, 0.41 mmol). m.p.: 38-40 $^{\circ}\text{C}$; $[\alpha]_{\text{D}}^{20} = +21.3$ ($c = 1.2$, CHCl_3 , 93% ee); ^1H NMR (CDCl_3 , 500 MHz): δ 1.01 (s, 9H), 1.84 (br s, 1H), 5.13 (d, 1H, $J = 6.9$ Hz), 5.52 (dd, 1H, $J = 15.6, 6.9$ Hz), 5.77 (d, 1H, $J = 15.6$ Hz), 7.01 (t, 2H, $J = 8.7$ Hz), and 7.31 (dd, 2H, $J = 8.3, 5.6$ Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 29.4, 32.9, 74.7, 115.1, 115.3, 127.1, 127.77, 127.83, 139.18, 139.20, 143.7, 161.1, and 163.1 ppm; IR (film): 3344, 3025, 2959, 2866, 1725, 1661, 1604, 1509, 1463, 1363, 1294, 1225, and 1156 cm^{-1} ; HRMS-CI m/z 208.1267 [M^+ ; calcd for $\text{C}_{13}\text{H}_{17}\text{FO}$: 208.1263].

(S)-4,4-Dimethyl-1-*p*-tolyl-pent-2-en-1-ol (8).

The product was prepared by General Procedure A using 98 mg (0.55 mmol) Cy_2BH , 68 μL (0.55 mmol) 3,3-dimethyl-1-butyne, 0.55 mL (1.1 mmol, 2.0 M in hexanes) Et_2Zn , 4.8 mg (0.02 mmol) (-)-MIB, and 59 μL (0.50 mmol) *p*-tolualdehyde. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound as a white solid in 86% yield (88 mg, 0.43 mmol). m.p.: 38-40 $^{\circ}\text{C}$; $[\alpha]_{\text{D}}^{20} = +52.7$ ($c = 0.97$, CHCl_3 , 95% ee); ^1H NMR (CDCl_3 , 500 MHz): δ 0.95 (s, 9H), 1.83 (br s, 1H), 2.26 (s, 3H), 5.03 (d, 1H, $J = 6.7$ Hz), 5.47 (dd, 1H, $J = 15.6, 6.8$ Hz), 5.70 (d, 1H, $J = 15.6$ Hz), 7.07 (d, 2H, $J = 7.7$ Hz), and 7.17 (d, 2H, $J = 7.9$ Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 21.1, 29.4, 32.8, 75.2, 126.1, 127.2, 129.1, 137.1, 140.6, and 143.2 ppm; IR (film): 3346, 3023, 2959, 2865, 2361, 2334, 1655,

1613, 1513, 1478, 1461, 1416, 1388, 1362, and 1268 cm^{-1} ; HRMS-CI m/z 204.1509 [M^+ ; calcd for $\text{C}_{14}\text{H}_{20}\text{O}$: 204.1514].

(S)-1-*p*-Tolyl-hept-2-en-1-ol (9). The product was prepared by General Procedure A using 98 mg (0.55 mmol) Cy_2BH , 64 μL (0.55 mmol) 1-hexyne, 0.55 mL (1.1 mmol, 2.0 M in hexanes) Et_2Zn , 4.8 mg (0.02 mmol) (–)-MIB, and 59 μL (0.50 mmol) *p*-tolualdehyde. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound as a colorless oil in 87% yield (89 mg, 0.44 mmol). $[\alpha]_{\text{D}}^{20} = +43.1$ ($c = 1.3$, CHCl_3 , 95% ee); ^1H NMR (CDCl_3 , 500 MHz): δ 0.81 (t, 3H, $J = 7.1$ Hz), 1.20–1.32 (m, 4H), 1.81 (br s, 1H), 1.97 (dt, 2H, $J = 7.0, 6.6$ Hz), 2.26 (s, 3H), 5.04 (d, 1H, $J = 6.5$ Hz), 5.57 (dd, 1H, $J = 15.3, 6.6$ Hz), 5.66 (dt, 1H, $J = 15.3, 6.5$ Hz), 7.07 (d, 2H, $J = 7.8$ Hz), and 7.17 (d, 2H, $J = 7.8$ Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 13.9, 21.1, 22.2, 31.2, 31.9, 75.1, 126.1, 129.1, 132.3, 132.5, 137.1, and 140.5 ppm; IR (neat): 3346, 3017, 2957, 2925, 2858, 1663, 1613, 1513, 1456, 1377, and 1198 cm^{-1} ; HRMS-CI m/z 203.1440 [$(\text{M} - \text{H})^+$; calcd for $\text{C}_{14}\text{H}_{19}\text{O}$: 203.1436].

(S)-1-Cyclohexyl-4,4-dimethyl-pent-2-en-1-ol (10). The product was prepared by General Procedure A using 98 mg (0.55 mmol) Cy_2BH , 68 μL (0.55 mmol) 3,3-dimethyl-1-butyne, 0.55 mL (1.1 mmol, 2.0 M in hexanes) Et_2Zn , 4.8 mg (0.02 mmol) (–)-MIB, and 61 μL (0.50 mmol) cyclohexane-carboxaldehyde. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound as a colorless oil in 80% yield (78 mg, 0.40 mmol). $[\alpha]_{\text{D}}^{20} = -27.7$ ($c = 0.13$, CHCl_3 , 96% ee); ^1H NMR (CDCl_3 , 500 MHz): δ 0.92–0.97 (m, 2H), 1.01 (s, 9H), 1.11–1.26 (m, 4H), 1.41 (m, 1H), 1.64 (m, 2H), 1.73 (m, 2H), 1.86 (br d, 1H, $J = 12.6$ Hz), 3.75 (t, 1H, $J = 7.0$ Hz), 5.35 (dd, 1H, $J = 15.6, 7.5$ Hz), and 5.62 (d, 1H, $J = 15.7$ Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 26.1, 26.2, 26.6, 28.76, 28.84, 29.6, 32.9, 43.8, 77.9, 126.2, and 143.9 ppm; IR (neat): 3351, 2953, 2926, 2853, 1655, 1473, 1450, 1387, 1362, 1305, 1260, and 1204 cm^{-1} ; HRMS-CI m/z 195.1743 [$(\text{M} - \text{H})^+$; calcd for $\text{C}_{13}\text{H}_{23}\text{O}$: 195.1749].

(S)-2,7,7-Trimethyl-oct-5-en-4-ol (11). The product was prepared by General Procedure A using 196 mg (1.1 mmol) Cy_2BH , 137 μL (1.1 mmol) 3,3-dimethyl-1-butyne, 1.1 mL (2.1 mmol, 2.0 M in hexanes) Et_2Zn , 9.6 mg (0.04 mmol) (–)-MIB, and 107 μL (1.0 mmol) isovaleraldehyde. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound as a colorless oil in 86% yield (146 mg, 0.86 mmol). $[\alpha]_{\text{D}}^{20} = +16.7$ ($c = 3.0$, CHCl_3 , 85% ee); ^1H NMR (CDCl_3 , 500 MHz): δ 0.88 (t, 6H, $J = 7.0$ Hz), 0.97 (s, 9H), 1.26 (m, 1H), 1.43 (m, 1H), 1.50 (br s, 1H), 1.65 (m, 1H), 4.07 (dt, 1H, $J = 7.0, 6.8$ Hz), 5.31 (dd, 1H, $J = 15.6, 7.2$ Hz), and 5.61 (d, 1H, $J = 15.7$ Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 22.5, 22.9, 24.6, 29.5, 32.5, 46.6, 71.5, 128.1, and 142.6 ppm; IR (neat): 3334, 3013, 2956, 2869, 2354, 2320, 1662, 1466, 1386, 1363, 1308, 1268, 1220, and 1202 cm^{-1} ; HRMS-CI m/z 170.1666 [M^+ ; calcd for $\text{C}_{11}\text{H}_{22}\text{O}$: 170.1671].

(S)-6,6-Dimethyl-hept-4-en-3-ol (12). The product was prepared by General Procedure A using 196 mg (1.1 mmol) Cy_2BH , 137 μL (1.1 mmol) 3,3-dimethyl-1-butyne, 1.1 mL (2.1 mmol, 2.0 M in hexanes) Et_2Zn , 9.6 mg (0.04 mmol) (–)-MIB, and 72 μL (1.0 mmol) propanal.

The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound as a colorless oil in 68% yield (96 mg, 0.68 mmol). $[\alpha]_{\text{D}}^{20} = -60.3$ ($c = 0.63$, CHCl_3 , 77% ee); ^1H NMR (CDCl_3 , 500 MHz): δ 0.86 (t, 3H, $J = 7.4$ Hz), 0.98 (s, 9H), 1.26 (br s, 1H), 1.44–1.59 (m, 2H), 3.93 (dt, 1H, $J = 6.6$, 6.6 Hz), 5.31 (dd, 1H, $J = 15.7$, 7.1 Hz), and 5.62 (d, 1H, $J = 15.7$ Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 9.7, 22.6, 29.5, 30.2, 74.7, 127.4, and 143.2 ppm; IR (neat): 3360, 3025, 2960, 2866, 1660, 1478, 1463, 1414, 1384, 1363, 1331, and 1262 cm^{-1} ; HRMS-CI m/z 141.1274 $[(\text{M} - \text{H})^+]$; calcd for $\text{C}_9\text{H}_{17}\text{O}$: 141.1279].

(S)-Non-4-en-3-ol (13). The product was prepared by General Procedure A using 50 mg (0.28 mmol) Cy_2BH , 32 μL (0.28 mmol) 1-hexyne, 0.39 mL (0.78 mmol, 2.0 M in hexanes) Et_2Zn ,

2.4 mg (0.01 mmol) (–)-MIB, and 18 μL (0.25 mmol) propanal. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound as a colorless oil in 74% yield (26 mg, 0.18 mmol). $[\alpha]_{\text{D}}^{20} = +58.5$ ($c = 0.97$, CHCl_3 , 79% ee); ^1H NMR (CDCl_3 , 500 MHz): δ 0.87 (m, 6H), 1.22–1.36 (m, 4H), 1.39 (br s, 1H), 1.43–1.57 (m, 2H), 2.00 (dt, 2H, $J = 7.0$, 6.6 Hz), 3.94 (dt, 1H, $J = 6.6$, 6.6 Hz), 5.41 (dd, 1H, $J = 15.3$, 7.0 Hz), and 5.60 (dt, 1H, $J = 15.3$, 6.7 Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 9.7, 13.9, 22.2, 30.2, 31.4, 31.9, 74.6, 132.4, and 132.7 ppm; IR (neat): 3421, 2955, 2921, 2854, 2361, 2337, 1725, 1663, 1601, 1455, 1405, and 1259 cm^{-1} ; HRMS-CI m/z 141.1273 $[(\text{M} - \text{H})^+]$; calcd for $\text{C}_9\text{H}_{17}\text{O}$: 141.1279].

(S)-2-Methyl-non-4-en-3-ol (14). The product was prepared by General Procedure A using 98 mg (0.55 mmol) Cy_2BH , 64 μL (0.55 mmol) 1-hexyne, 0.55 mL (1.1 mmol, 2.0 M in hexanes) Et_2Zn , 4.8 mg (0.02 mmol) (–)-MIB, and 45 μL (0.50 mmol)

isobutyraldehyde. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound as a colorless oil in 88% yield (69 mg, 0.44 mmol). $[\alpha]_{\text{D}}^{20} = -12.5$ ($c = 1.3$, CHCl_3 , 92% ee); ^1H NMR (CDCl_3 , 500 MHz): δ 0.82 (d, 3H, $J = 6.7$ Hz), 0.84 (t, 3H, $J = 7.2$ Hz), 0.87 (d, 3H, $J = 6.7$ Hz), 1.23–1.32 (m, 4H), 1.48 (br s, 1H), 1.64 (m, 1H), 1.99 (dt, 2H, $J = 6.8$, 6.5 Hz), 3.72 (t, 1H, $J = 6.5$ Hz), 5.40 (dd, 1H, $J = 15.3$, 7.2 Hz), and 5.57 (dt, 1H, $J = 15.3$, 6.7 Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 13.9, 18.1, 18.2, 22.2, 31.4, 32.0, 33.8, 78.3, 131.1, and 133.2 ppm; IR (neat): 3221, 2953, 2925, 2862, 1488, 1466, 1381, 1331, 1260, and 1194 cm^{-1} ; LRMS-CI m/z 139.14 $[(\text{M} - \text{OH})^+]$; calcd for $\text{C}_{10}\text{H}_{19}\text{O}$: 139.15].

(2-Bromo-phenyl)-(3-butyl-oxiranyl)-methanol (18).

General Procedure B. A 10 mL Schlenk flask (A) was charged with Cy_2BH (98 mg, 0.55 mmol), prepared according to Oppolzer's procedure,¹ and 1.2 mL of hexanes. 1-Hexyne (64 μL , 0.55 mmol) was added dropwise, and the homogenous reaction mixture was stirred for 30 min at room temp. After cooling the reaction flask to $-10\text{ }^\circ\text{C}$, (–)-MIB (4.8

mg, 0.02 mmol) was added, followed by Et_2Zn (0.78 mL, 2.0 M), then *o*-bromobenzaldehyde (58 μL , 0.50 mmol) dropwise. The reaction was stirred at $-10\text{ }^\circ\text{C}$ for 4 h until vinyl addition was complete by TLC. The reaction flask was then capped with a balloon of oxygen and allowed to stir at $-10\text{ }^\circ\text{C}$ for 1 h. In a separate 10 mL Schlenk flask (B), 1 mL of dichloromethane, $\text{Ti}(\text{OiPr})_4$ (100 μL , 1.0 M in hexanes), and (+)-DIPT (21 μL , 0.10 mmol) were combined. After stirring at room temp for 45 min, flask B was evacuated for 1 h to strip off the solvent. The contents of flask B were then redissolved in 1 mL of dichloromethane and stripped again for 1 h. Finally, the contents of flask B were taken up in 1 mL of dichloromethane and transferred to reaction flask A. The reaction continued to stir at $-10\text{ }^\circ\text{C}$ under an O_2 atmosphere for 18 h. It was then quenched with 2 mL 15% tartaric acid solution and allowed to stir for 45 min before the organic and aqueous layers were separated, and the aqueous layer was extracted with hexanes (3 X 5 mL). The combined organic layers were then washed with 5 mL H_2O and dried over MgSO_4 . The filtrate was concentrated *in vacuo* and the residue was chromatographed on silica (5% ethyl acetate in hexanes) to afford the title compound in 77% yield (110 mg, 0.39 mmol). *threo*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = -40.5$ ($c = 0.39$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.87 (t, 3H, $J = 7.1$ Hz), 1.27-1.44 (m, 4H), 1.53-1.61 (m, 2H), 2.51 (d, 1H, $J = 5.4$ Hz), 2.98 (dd, 1H, $J = 4.5, 2.1$ Hz), 3.24 (dt, 1H, $J = 5.6, 2.0$ Hz), 5.02 (t, 1H, $J = 4.9$ Hz), 7.18 (t, 1H, $J = 7.6$ Hz), 7.37 (t, 1H, $J = 7.5$ Hz), 7.56 (d, 1H, $J = 7.9$ Hz), and 7.61 (d, 1H, $J = 7.7$ Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 13.9, 22.4, 27.9, 31.1, 57.7, 61.1, 71.6, 122.1, 127.9, 128.0, 129.5, 132.8, and 139.8 ppm; IR (neat): 3424, 3063, 2962, 2930, 2861, 1562, 1468, 1434, 1383, and 1260 cm^{-1} ; HRMS-CI m/z 285.0496 [MH^+ ; calcd for $\text{C}_{13}\text{H}_{18}\text{BrO}_2$: 285.0490]. *erythro*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = -55.9$ ($c = 1.2$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.82 (t, 3H, $J = 7.1$ Hz), 1.20-1.33 (m, 4H), 1.44 (m, 1H), 1.55 (m, 1H), 2.51 (s, 1H), 3.03 (dt, 1H, $J = 5.6, 1.8$ Hz), 3.20 (br t, 1H), 5.37 (br s, 1H), 7.17 (t, 1H, $J = 7.1$ Hz), 7.34 (t, 1H, $J = 7.5$ Hz), 7.50 (d, 1H, $J = 7.6$ Hz), and 7.54 (d, 1H, $J = 8.0$ Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 13.8, 22.2, 27.9, 31.0, 54.8, 59.6, 69.1, 122.1, 127.7, 127.9, 129.4, 132.6, and 138.8 ppm; IR (neat): 3434, 3072, 2957, 2931, 2859, 1590, 1568, 1468, 1438, and 1378 cm^{-1} ; HRMS-CI m/z 285.0496 [MH^+ ; calcd for $\text{C}_{13}\text{H}_{18}\text{BrO}_2$: 285.0490].

(3-*tert*-Butyl-oxiranyl)-phenyl-methanol (15). The product was prepared by General Procedure B using 50 mg (0.28 mmol) Cy_2BH , 35 μL (0.28 mmol) 3,3-dimethyl-1-butyne, 0.39 mL (0.78 mmol, 2.0 M in hexanes) Et_2Zn , 2.4 mg (0.01 mmol) (–)-MIB, 25 μL (0.25 mmol) benzaldehyde, 50 μL (0.05 mmol, 1.0 M in hexanes) $\text{Ti}(\text{OiPr})_4$, and 10.6 μL (0.05 mmol) (+)-DIPT. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound in 77% yield (40 mg, 0.19 mmol). *threo*-diastereomer: White solid. m.p.: $70\text{--}73\text{ }^\circ\text{C}$. $[\alpha]_{\text{D}}^{20} = -25.6$ ($c = 0.16$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.83 (s, 9H), 2.32 (br s, 1H), 2.82 (d, 1H, $J = 1.6$ Hz), 3.01 (dd, 1H, $J = 5.3, 1.9$ Hz), 4.46 (t, 1H, $J = 4.4$ Hz), 7.26 (t, 1H, $J = 6.9$ Hz), and 7.31-7.36 (m, 4H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 25.7, 30.6, 59.3, 65.1, 74.2, 126.0, 128.0, 128.6, and 140.5 ppm; IR (film): 3413, 3049, 3025, 2955, 2919, 2861, 2355, 1719, 1602, 1478, 1455, 1390, 1360, 1273, 1237, and 1196 cm^{-1} ; HRMS-CI m/z 189.1288 [(M –

OH^+ ; calcd for $\text{C}_{13}\text{H}_{17}\text{O}$: 189.1279]. *erythro*-diastereomer: White solid. m.p.: 57-59 °C; $[\alpha]_{\text{D}}^{20} = -27.1$ ($c = 0.31$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.82 (s, 9H), 2.20 (br s, 1H), 2.96 (d, 1H, $J = 2.0$ Hz), 3.01 (t, 1H, $J = 2.7$ Hz), 4.82 (br s, 1H), and 7.26-7.32 (m, 5H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 25.8, 30.4, 58.3, 62.8, 71.2, 126.5, 128.3, 128.6, and 139.8 ppm; IR (film): 3413, 3037, 2955, 2931, 2861, 1719, 1643, 1602, 1449, 1367, and 1327 cm^{-1} ; HRMS-CI m/z 189.1288 $[(\text{M} - \text{OH})^+]$; calcd for $\text{C}_{13}\text{H}_{17}\text{O}$: 189.1279].

(3-Butyl-oxiranyl)-phenyl-methanol (16). The product was prepared by General Procedure B using 50 mg (0.28 mmol) Cy_2BH , 32 μL (0.28 mmol) 1-hexyne, 0.39 mL (0.78 mmol, 2.0 M in hexanes) Et_2Zn , 2.4 mg (0.01 mmol) (–)-MIB, 25 μL

(0.25 mmol) benzaldehyde, 50 μL (0.05 mmol, 1.0 M in hexanes) $\text{Ti}(\text{OiPr})_4$, and 10.6 μL (0.05 mmol) (+)-DIPT. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound in 83% yield (43 mg, 0.21 mmol). *threo*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = -12.9$ ($c = 2.5$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.83 (t, 3H, $J = 7.1$ Hz), 1.22-1.41 (m, 4H), 1.46-1.56 (m, 2H), 2.31 (br s, 1H), 2.93 (dt, 1H, $J = 5.2, 2.0$ Hz), 3.01 (dt, 1H, $J = 5.7, 5.4$ Hz), 4.48 (d, 1H, $J = 2.0$ Hz), and 7.27-7.38 (m, 5H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 14.0, 22.5, 28.1, 31.3, 57.7, 62.6, 74.4, 126.9, 128.8, 129.4, and 141.2 ppm; IR (neat): 3420, 3060, 2957, 2931, 2859, 1713, 1692, 1600, 1578, 1490, 1452, 1408, 1384, and 1314 cm^{-1} ; HRMS-ESI m/z 189.1275 $[(\text{M} - \text{OH})^+]$; calcd for $\text{C}_{13}\text{H}_{17}\text{O}$: 189.1279]. *erythro*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = -10.7$ ($c = 0.43$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.83 (t, 3H, $J = 7.1$ Hz), 1.22-1.38 (m, 4H), 1.44-1.56 (m, 2H), 2.22 (br s, 1H), 2.94 (t, 1H, $J = 2.4$ Hz), 3.13 (dt, 1H, $J = 5.8, 5.4$ Hz), 4.84 (d, 1H, $J = 2.6$ Hz), and 7.28-7.35 (m, 5H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 14.0, 22.5, 28.2, 31.3, 55.5, 61.7, 71.4, 127.2, 129.0, 129.4, and 139.6 ppm; IR (neat): 3420, 3060, 3025, 2957, 2930, 2859, 1725, 1605, 1496, 1453, 1378, 1284, 1237, and 1190 cm^{-1} ; HRMS-ESI m/z 189.1275 $[(\text{M} - \text{OH})^+]$; calcd for $\text{C}_{13}\text{H}_{17}\text{O}$: 189.1279].

(2-Bromo-phenyl)-(3-tert-butyl-oxiranyl)-methanol (17). The product was prepared by General Procedure B using 50 mg (0.28 mmol) Cy_2BH , 35 μL (0.28 mmol) 3,3-dimethyl-1-butyne, 0.39 mL (0.78 mmol, 2.0 M in hexanes) Et_2Zn , 2.4 mg (0.01 mmol) (–)-

MIB, 29 μL (0.25 mmol) *o*-bromobenzaldehyde, 0.25 mL (0.25 mmol, 1.0 M in hexanes) $\text{Ti}(\text{OiPr})_4$, and 63 μL (0.30 mmol) (+)-DIPT. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound in 78% yield (55 mg, 0.19 mmol). *threo*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = -65.8$ ($c = 0.40$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.90 (s, 9H), 2.55 (d, 1H, $J = 5.2$ Hz), 3.08 (m, 2H), 5.00 (t, 1H, $J = 4.6$ Hz), 7.18 (t, 1H, $J = 7.6$ Hz), 7.37 (t, 1H, $J = 7.5$ Hz), 7.55 (d, 1H, $J = 7.9$ Hz), and 7.61 (d, 1H, $J = 7.3$ Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 25.7, 30.6, 58.2, 65.2, 71.7, 122.0, 127.88, 127.94, 129.4, 132.8, and 140.0 ppm; IR (neat): 3418, 3060, 2957, 2872, 2343, 1568, 1469, 1437, 1396, 1365, and 1190 cm^{-1} ; HRMS-CI m/z 267.0368 $[(\text{M} - \text{OH})^+]$; calcd for $\text{C}_{13}\text{H}_{16}\text{BrO}$: 267.0385]. *erythro*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = -55.5$ ($c = 0.53$, CHCl_3); ^1H NMR (CDCl_3 , 500

MHz): δ 0.84 (s, 9H), 2.42 (br d, 1H, J = 2.1 Hz), 2.85 (br d, 1H, J = 2.3 Hz), 3.29 (t, 1H, J = 2.4 Hz), 5.40 (br s, 1H), 7.17 (t, 1H, J = 7.4 Hz), 7.34 (t, 1H, J = 7.4 Hz), 7.51 (d, 1H, J = 7.7 Hz), and 7.54 (d, 1H, J = 8.0 Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 25.7, 30.3, 56.5, 62.3, 69.1, 122.1, 127.7, 127.9, 129.4, 132.6, and 138.9 ppm; IR (neat): 3426, 3060, 2958, 2861, 1631, 1590, 1586, 1467, 1439, 1390, and 1364 cm^{-1} ; HRMS-Cl m/z 267.0368 $[(\text{M} - \text{OH})^+]$; calcd for $\text{C}_{13}\text{H}_{16}\text{BrO}$: 267.0385].

(3-*tert*-Butyl-oxiranyl)-(4-trifluoromethyl-phenyl)-methanol (19). The product was prepared by General Procedure B using 50 mg (0.28 mmol) Cy_2BH , 35 μL (0.28 mmol) 3,3-dimethyl-1-butyne, 0.39 mL (0.78 mmol, 2.0 M in hexanes) Et_2Zn , 2.4 mg (0.01 mmol) (–)-MIB, 34 μL (0.25 mmol) *p*-trifluoromethylbenzaldehyde, 50 μL (0.05 mmol, 1.0 M in hexanes) $\text{Ti}(\text{OiPr})_4$, and 10.6 μL (0.05 mmol) (+)-DIPT. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound in 85% yield (58 mg, 0.21 mmol). *threo*-diastereomer: Colorless solid. m.p.: 76–79 $^\circ\text{C}$; $[\alpha]_{\text{D}}^{20} = -10.0$ (c = 0.27, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.90 (s, 10H), 2.89 (d, 1H, J = 2.2 Hz), 3.03 (dd, 1H, J = 5.5, 2.2 Hz), 4.58 (d, 1H, J = 5.4 Hz), 7.53 (d, 2H, J = 8.1 Hz), and 7.64 (d, 2H, J = 8.1 Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 25.7, 30.6, 59.0, 65.3, 73.6, 125.54, 125.58, 125.59, and 126.3 ppm; IR (film): 3411, 3028, 2969, 2927, 2871, 2367, 2332, 1622, 1477, 1460, 1420, 1365, 1331, and 1241 cm^{-1} ; HRMS-Cl m/z 255.1192 $[(\text{M} - \text{F})^+]$; calcd for $\text{C}_{14}\text{H}_{17}\text{F}_2\text{O}_2$: 255.1197]. *erythro*-diastereomer: Colorless solid. m.p.: 74–75 $^\circ\text{C}$; $[\alpha]_{\text{D}}^{20} = -32.0$ (c = 0.10, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.83 (s, 9H), 2.30 (br s, 1H), 2.93 (d, 1H, J = 2.0 Hz), 3.03 (t, 1H, J = 2.6 Hz), 4.90 (d, 1H, J = 2.2 Hz), 7.48 (d, 2H, J = 8.0 Hz), and 7.60 (d, 2H, J = 8.0 Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 25.7, 31.6, 57.9, 62.8, 75.6, 125.50, 125.51, 125.53, and 126.6 ppm; IR (film): 3350, 3037, 2955, 2868, 1661, 1620, 1477, 1463, 1414, 1365, and 1327 cm^{-1} ; HRMS-Cl m/z 255.1192 $[(\text{M} - \text{F})^+]$; calcd for $\text{C}_{14}\text{H}_{17}\text{F}_2\text{O}_2$: 255.1197].

(3-Butyl-oxiranyl)-(4-trifluoromethyl-phenyl)-methanol (20). The product was prepared by General Procedure B using 98 mg (0.55 mmol) Cy_2BH , 64 μL (0.55 mmol) 1-hexyne, 0.78 mL (1.55 mmol, 2.0 M in hexanes) Et_2Zn , 4.8 mg (0.02 mmol) (–)-MIB, 68 μL (0.50 mmol) *p*-trifluoromethylbenzaldehyde, 100 μL (0.10 mmol, 1.0 M in hexanes) $\text{Ti}(\text{OiPr})_4$, and 21 μL (0.10 mmol) (+)-DIPT. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound in 76% yield (104 mg, 0.38 mmol). *threo*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = -22.4$ (c = 0.25, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.81 (t, 3H, J = 7.1 Hz), 1.19–1.35 (m, 3H), 1.45–1.53 (m, 3H), 2.37 (br d, 1H, J = 3.7 Hz), 2.87 (dt, 1H, J = 5.1, 1.8 Hz), 3.00 (dt, 1H, J = 5.3, 1.3 Hz), 4.53 (br t, 1H), 7.46 (d, 2H, J = 8.0 Hz), and 7.57 (d, 2H, J = 8.0 Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 13.9, 22.4, 27.9, 31.1, 57.4, 61.8, 73.2, 125.56, 125.59, 126.4, and 144.2 ppm; IR (neat): 3432, 2960, 2931, 2860, 2352, 2326, 1621, 1469, 1456, 1417, 1380, 1326, and 1260 cm^{-1} ; HRMS-ESI m/z 255.1187 $[(\text{M} - \text{F})^+]$; calcd for $\text{C}_{14}\text{H}_{17}\text{F}_2\text{O}_2$: 255.1197]. *erythro*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = -26.0$

($c = 0.80$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.77 (t, 3H, $J = 7.2$ Hz), 1.15-1.32 (m, 3H), 1.38-1.52 (m, 3H), 2.27 (br s, 1H), 2.91 (t, 1H, $J = 2.6$ Hz), 3.04 (dt, 1H, $J = 5.8$, 2.0 Hz), 4.87 (br s, 1H), 7.44 (d, 2H, $J = 8.0$ Hz), and 7.57 (d, 2H, $J = 8.0$ Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 13.8, 22.3, 28.0, 31.0, 55.2, 61.0, 70.5, 125.49, 125.52, 126.5, and 143.7 ppm; IR (neat): 3425, 3013, 2966, 2932, 2861, 1619, 1457, 1414, 1384, 1326, and 1261 cm^{-1} ; HRMS-ESI m/z 255.1187 $[(\text{M} - \text{F})^+]$; calcd for $\text{C}_{14}\text{H}_{17}\text{F}_2\text{O}_2$: 255.1197].

(3-*tert*-Butyl-oxiranyl)-(4-fluorophenyl)-methanol (21). The product was prepared by General Procedure B using 98 mg (0.55 mmol) Cy_2BH , 68 μL (0.55 mmol) 3,3-dimethyl-1-butyne, 0.78 mL (1.55 mmol, 2.0 M in hexanes) Et_2Zn , 4.8 mg (0.02 mmol) (–)-MIB, 54 μL (0.50 mmol) *p*-fluorobenzaldehyde, 100 μL (0.10 mmol, 1.0 M in hexanes) $\text{Ti}(\text{OiPr})_4$, and 21 μL (0.10 mmol) (+)-DIPT. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound in 92% yield (103 mg, 0.46 mmol). *threo*-diastereomer: Colorless solid. m.p.: 83-85 $^\circ\text{C}$; $[\alpha]_{\text{D}}^{20} = -14.8$ ($c = 0.23$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.82 (s, 9H), 2.31 (br s, 1H), 2.78 (d, 1H, $J = 2.1$ Hz), 2.95 (dd, 1H, $J = 5.5$, 2.2 Hz), 4.42 (d, 1H, $J = 5.2$ Hz), 6.99 (t, 2H, $J = 8.6$ Hz), and 7.31 (dd, 2H, $J = 8.3$, 5.5 Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 25.7, 30.6, 59.2, 65.1, 73.6, 115.4, 115.6, 127.68, 127.74, 136.2, and 163.5 ppm; IR (neat): 3403, 3002, 2963, 2919, 2872, 1725, 1661, 1637, 1603, 1511, 1461, 1390, 1361, and 1226 cm^{-1} ; HRMS-CI m/z 224.1206 $[\text{M}^+]$; calcd for $\text{C}_{13}\text{H}_{17}\text{FO}_2$: 224.1213]. *erythro*-diastereomer: Colorless solid. m.p.: 56-59 $^\circ\text{C}$; $[\alpha]_{\text{D}}^{20} = -34.3$ ($c = 0.40$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.85 (s, 9H), 2.28 (br s, 1H), 2.96 (d, 1H, $J = 2.1$ Hz), 3.01 (t, 1H, $J = 2.7$ Hz), 4.84 (br d, 1H, $J = 2.8$ Hz), 7.04 (t, 2H, $J = 8.5$ Hz), and 7.34 (dd, 2H, $J = 8.3$, 5.5 Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 25.8, 30.4, 58.2, 62.7, 70.5, 115.4, 115.6, 128.08, 128.15, 135.6, and 163.6 ppm; IR (KBr): 3409, 3015, 2964, 2871, 1606, 1510, 1463, 1392, 1369, 1331, and 1316 cm^{-1} ; HRMS-CI m/z 224.1206 $[\text{M}^+]$; calcd for $\text{C}_{13}\text{H}_{17}\text{FO}_2$: 224.1213].

(3-*tert*-Butyl-oxiranyl)-*p*-tolyl-methanol (22). The product was prepared by General Procedure B using 98 mg (0.55 mmol) Cy_2BH , 68 μL (0.55 mmol) 3,3-dimethyl-1-butyne, 0.78 mL (1.55 mmol, 2.0 M in hexanes) Et_2Zn , 4.8 mg (0.02 mmol) (–)-MIB, 59 μL (0.50 mmol) *p*-tolualdehyde, 100 μL (0.10 mmol, 1.0 M in hexanes) $\text{Ti}(\text{OiPr})_4$, and 21 μL (0.10 mmol) (+)-DIPT. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound in 85% yield (93 mg, 0.42 mmol). *threo*-diastereomer: White solid. m.p.: 36-39 $^\circ\text{C}$; $[\alpha]_{\text{D}}^{20} = -40.0$ ($c = 0.26$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.81 (s, 9H), 2.28 (s, 3H), 2.35 (br d, 1H, $J = 3.9$ Hz), 2.78 (d, 1H, $J = 2.2$ Hz), 2.97 (dd, 1H, $J = 5.5$, 2.2 Hz), 4.39 (br t, 1H), 7.10 (d, 2H, $J = 7.8$ Hz), and 7.22 (d, 2H, $J = 7.9$ Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 21.1, 25.7, 30.6, 59.4, 65.1, 74.1, 125.9, 129.2, 137.5, and 137.7 ppm; IR (film): 3416, 3095, 2957, 2867, 1614, 1514, 1482, 1463, 1417, 1393, 1365, 1260, and 1239 cm^{-1} ; HRMS-CI m/z 203.1431 $[(\text{M} - \text{OH})^+]$; calcd for $\text{C}_{14}\text{H}_{19}\text{O}$: 203.1436]. *erythro*-diastereomer: White solid. m.p.: 65-68 $^\circ\text{C}$; $[\alpha]_{\text{D}}^{20} = -58.2$ ($c =$

0.57, CHCl₃); ¹H NMR (CDCl₃, 500 MHz): δ 0.82 (s, 9H), 2.16 (br s, 1H), 2.28 (s, 3H), 2.95 (d, 1H, *J* = 2.1 Hz), 2.97 (t, 1H, *J* = 2.7 Hz), 4.76 (br s, 1H), 7.10 (d, 2H, *J* = 7.7 Hz), and 7.19 (d, 2H, *J* = 8.0 Hz) ppm; ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 21.2, 25.8, 30.4, 58.3, 62.8, 71.1, 126.5, 129.2, 136.8, and 137.9 ppm; IR (film): 3418, 3018, 2965, 2923, 2867, 1919, 1515, 1478, 1458, 1412, 1381, and 1322 cm⁻¹; HRMS-CI *m/z* 203.1431 [(M – OH)⁺; calcd for C₁₄H₁₉O: 203.1436].

(3-Butyl-oxiranyl)-*p*-tolyl-methanol (23). The product was prepared by General Procedure B using 89 mg (0.55 mmol) Cy₂BH, 64 μL (0.55 mmol) 1-hexyne, 0.78 mL (1.55 mmol, 2.0 M in hexanes) Et₂Zn, 4.8 mg (0.02 mmol) (–)-MIB, 59 μL (0.50 mmol) *p*-tolualdehyde, 100 μL (0.10 mmol, 1.0 M in hexanes) Ti(OiPr)₄, and 21 μL (0.10 mmol) (+)-DIPT. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound in 83% yield (92 mg, 0.42 mmol). *threo*-diastereomer: Colorless oil. [α]_D²⁰ = –18.7 (*c* = 0.31, CHCl₃); ¹H NMR (CDCl₃, 500 MHz): δ 0.81 (t, 3H, *J* = 7.1 Hz), 1.20–1.34 (m, 4H), 1.46–1.51 (m, 2H), 2.25 (br d, 1H, *J* = 4.1 Hz), 2.30 (s, 3H), 2.90 (dd, 1H, *J* = 5.2, 2.0 Hz), 2.97 (dt, 1H, *J* = 5.5, 1.8 Hz), 4.42 (br t, 1H), 7.13 (d, 2H, *J* = 7.7 Hz), and 7.23 (d, 2H, *J* = 7.9 Hz) ppm; ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 13.9, 21.1, 22.4, 28.0, 31.2, 57.3, 62.2, 73.8, 126.1, 126.4, 129.3, and 137.5 ppm; IR (neat): 3428, 3051, 2961, 2928, 2860, 2356, 1646, 1512, 1462, 1383, 1260, and 1192 cm⁻¹; HRMS-CI *m/z* 202.1362 [(M – H₂O)⁺; calcd for C₁₄H₁₈O: 202.1358]. *erythro*-diastereomer: Colorless oil. [α]_D²⁰ = –58.4 (*c* = 0.25, CHCl₃); ¹H NMR (CDCl₃, 500 MHz): δ 0.79 (t, 3H, *J* = 7.1 Hz), 1.19–1.33 (m, 4H), 1.43–1.50 (m, 2H), 2.15 (br s, 1H), 2.28 (s, 3H), 2.89 (t, 1H, *J* = 2.5 Hz), 3.10 (dt, 1H, *J* = 5.6, 1.5 Hz), 4.77 (br s, 1H), 7.11 (d, 2H, *J* = 7.8 Hz), and 7.19 (d, 2H, *J* = 8.1 Hz) ppm; ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 13.9, 21.2, 22.4, 28.0, 31.2, 55.2, 61.3, 70.9, 126.4, 129.3, 136.7, and 138.0 ppm; IR (neat): 3430, 3028, 2950, 2916, 2860, 2356, 2334, 1680, 1607, 1512, 1461, 1383, and 1265 cm⁻¹; HRMS-CI *m/z* 202.1362 [(M – H₂O)⁺; calcd for C₁₄H₁₈O: 202.1358].

(3-*tert*-Butyl-oxiranyl)-cyclohexyl-methanol (24). The product was prepared by General Procedure B using 98 mg (0.55 mmol) Cy₂BH, 68 μL (0.55 mmol) 3,3-dimethyl-1-butyne, 0.78 mL (1.55 mmol, 2.0 M in hexanes) Et₂Zn, 4.8 mg (0.02 mmol) (–)-MIB, 61 μL (0.50 mmol) cyclohexane carboxaldehyde, 100 μL (0.10 mmol, 1.0 M in hexanes) Ti(OiPr)₄, and 21 μL (0.10 mmol) (+)-DIPT. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound in 74% yield (79 mg, 0.37 mmol). *threo*-diastereomer: White solid. m.p.: 48–50 °C; [α]_D²⁰ = +70.7 (*c* = 0.68, CHCl₃); ¹H NMR (CDCl₃, 500 MHz): δ 0.92 (s, 9H), 1.00–1.19 (m, 6H), 1.47 (m, 1H), 1.64–1.87 (m, 5H), 2.79 (d, 1H, *J* = 2.1 Hz), 2.92 (t, 1H, *J* = 2.5 Hz), and 3.57 (t, 1H, *J* = 3.1 Hz) ppm; ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 25.9, 26.0, 26.2, 26.5, 28.5, 28.8, 30.5, 41.8, 56.5, 62.4, and 72.3 ppm; IR (KBr): 3392, 2922, 2853, 1643, 1449, 1391, 1363, 1262, 1245, and 1211 cm⁻¹; HRMS-CI *m/z* 213.1865 [MH⁺; calcd for C₁₃H₂₅O₂: 213.1855]. *erythro*-diastereomer: White solid. m.p.: 38–48 °C; [α]_D²⁰ = –38.9 (*c* = 0.27, CHCl₃); ¹H NMR (CDCl₃, 500 MHz): δ 0.92 (s, 9H), 1.03–1.28 (m, 6H),

1.66 (m, 1H), 1.73 (m, 3H), 1.89 (m, 2H), 2.67 (d, 1H, $J = 2.0$ Hz), 2.87 (dd, 1H, $J = 5.4$, 2.1 Hz), and 3.13 (m, 1H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 25.8, 26.0, 26.1, 26.4, 28.6, 28.8, 30.7, 42.2, 57.5, 65.0, and 75.9 ppm; IR (KBr): 3503, 2961, 2929, 2852, 1481, 1447, 1391, 1368, 1312, and 1284 cm^{-1} ; HRMS-Cl m/z 213.1865 [MH^+ ; calcd for $\text{C}_{13}\text{H}_{25}\text{O}_2$: 213.1855].

1-(3-*tert*-Butyl-oxiranyl)-3-methyl-butan-1-ol (25). The product was prepared by General Procedure B using 50 mg (0.28 mmol) Cy_2BH , 35 μL (0.28 mmol) 3,3-dimethyl-1-butyne, 0.39 mL (0.78 mmol, 2.0 M in hexanes) Et_2Zn , 2.4 mg (0.01 mmol) (–)-MIB, 27 μL (0.25 mmol) isovaleraldehyde, 50 μL (0.05 mmol, 1.0 M in hexanes) $\text{Ti}(\text{OiPr})_4$, and 10.6 μL (0.05 mmol) (+)-DIPT. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound in 89% yield (42 mg, 0.23 mmol). *threo*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = +72.5$ ($c = 0.67$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.90 (s, 9H), 0.91 (d, 3H, $J = 6.7$ Hz), 0.93 (d, 3H, $J = 6.7$ Hz), 1.24–1.29 (m, 1H), 1.36–1.42 (m, 1H), 1.78 (br s, 1H), 1.83 (m, 1H), 2.77 (d, 1H, $J = 2.3$ Hz), 2.83 (t, 1H, $J = 2.7$ Hz), and 3.86 (m, 1H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 22.0, 23.5, 24.5, 25.8, 31.6, 42.6, 58.0, 62.3, and 66.5 ppm; IR (neat): 3410, 2957, 2923, 2862, 1730, 1483, 1467, 1387, 1365, 1285, 1261, and 1209 cm^{-1} ; HRMS-Cl m/z 169.1585 [$(\text{M} - \text{OH})^+$; calcd for $\text{C}_{11}\text{H}_{21}\text{O}$: 169.1592]. *erythro*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = +60.6$ ($c = 0.53$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.90 (s, 9H), 0.93 (t, 6H, $J = 6.8$ Hz), 1.27–1.31 (m, 1H), 1.49–1.53 (m, 1H), 1.55 (br s, 1H), 1.79–1.82 (m, 1H), 2.68 (d, 1H, $J = 2.2$ Hz), 2.78 (dd, 1H, $J = 5.4$, 2.3 Hz), and 3.47 (m, 1H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 22.1, 23.2, 24.3, 25.8, 31.6, 43.2, 59.1, 64.9, and 70.1 ppm; IR (neat): 3379, 2959, 2923, 2872, 1726, 1483, 1463, 1417, 1361, and 1255 cm^{-1} ; HRMS-Cl m/z 169.1585 [$(\text{M} - \text{OH})^+$; calcd for $\text{C}_{11}\text{H}_{21}\text{O}$: 169.1592].

1-(3-*tert*-Butyl-oxiranyl)-propan-1-ol (26). The product was prepared by General Procedure B using 196 mg (1.1 mmol) Cy_2BH , 137 μL (1.1 mmol) 3,3-dimethyl-1-butyne, 1.1 mL (2.1 mmol, 2.0 M in hexanes) Et_2Zn , 9.6 mg (0.04 mmol) (–)-MIB, 72 μL (1.0 mmol) propanal, 200 μL (0.20 mmol, 1.0 M in hexanes) $\text{Ti}(\text{OiPr})_4$, and 42 μL (0.20 mmol) (+)-DIPT. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound in 69% yield (109 mg, 0.69 mmol). *threo*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = +80.3$ ($c = 0.79$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.90 (s, 9H), 0.96 (t, 3H, $J = 7.5$ Hz), 1.56–1.62 (m, 2H), 1.84 (br d, 1H, $J = 4.6$ Hz), 2.67 (d, 1H, $J = 2.2$ Hz), 2.80 (dd, 1H, $J = 5.1$, 1.8 Hz), and 3.32 (m, 1H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 22.6, 25.8, 27.4, 31.6, 58.5, 64.7, and 73.2 ppm; IR (neat): 3401, 2955, 2851, 1728, 1602, 1578, 1462, 1370, 1367, and 1273 cm^{-1} ; LRMS-Cl m/z 141.08 [$(\text{M} - \text{OH})^+$; calcd for $\text{C}_9\text{H}_{17}\text{O}$: 141.13]. *erythro*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = +28.0$ ($c = 0.79$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.90 (s, 9H), 0.99 (t, 3H, $J = 7.5$ Hz), 1.47–1.60 (m, 2H), 1.79 (br s, 1H), 2.78 (d, 1H, $J = 2.2$ Hz), 2.85 (t, 1H, $J = 2.7$ Hz), and 3.72 (m, 1H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 22.6, 25.8, 26.6, 31.6, 57.5, 62.4, and 69.6 ppm; IR (neat): 3366, 2955, 2922, 2849, 2367, 2332,

1725, 1596, 1455, and 1373 cm^{-1} ; LRMS-CI m/z 141.08 $[(M - OH)^+]$; calcd for $\text{C}_9\text{H}_{17}\text{O}$: 141.13].

1-(3-Butyl-oxiranyl)-propan-1-ol (27). The product was prepared by General Procedure B using 50 mg (0.28 mmol) Cy_2BH , 32 μL (0.28 mmol) 1-hexyne, 0.39 mL (0.78 mmol, 2.0 M in hexanes) Et_2Zn , 2.4 mg (0.01 mmol) (–)-MIB, 18 μL (0.25 mmol) propanal, 50 μL (0.05 mmol, 1.0 M in hexanes) $\text{Ti}(\text{OiPr})_4$, and 21 μL (0.10 mmol) (+)-DIPT. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound in 68% yield (27 mg, 0.17 mmol). *threo*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = -12.5$ ($c = 0.12$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.88 (t, 3H, $J = 7.0$ Hz), 0.99 (t, 3H, $J = 7.5$ Hz), 1.31–1.41 (m, 4H), 1.48–1.62 (m, 4H), 1.76 (br s, 1H), 2.73 (br t, 1H, $J = 2.7$ Hz), 2.96 (dt, 1H, $J = 5.7$, 2.0 Hz), and 3.70 (m, 1H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 9.6, 13.9, 22.5, 26.5, 28.1, 31.3, 54.9, 60.7, and 69.7 ppm; IR (neat): 3356, 2960, 2922, 2852, 2355, 2332, 1729, 1463, 1414, 1378, and 1261 cm^{-1} ; HRMS-CI m/z 141.1273 $[(M - OH)^+]$; calcd for $\text{C}_9\text{H}_{17}\text{O}$: 141.1279]. *erythro*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = +9.2$ ($c = 0.41$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.88 (t, 3H, $J = 7.0$ Hz), 0.97 (t, 3H, $J = 7.5$ Hz), 1.22–1.27 (m, 2H), 1.30–1.43 (m, 4H), 1.48–1.56 (m, 2H), 1.82 (br s, 1H), 2.70 (dd, 1H, $J = 4.9$, 2.1 Hz), 2.87 (dt, 1H, $J = 5.6$, 2.0 Hz), and 3.35 (m, 1H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 9.7, 13.9, 22.5, 27.5, 28.0, 31.3, 56.9, 61.5, and 72.6 ppm; IR (neat): 3424, 2959, 2922, 2851, 2362, 2332, 1731, 1463, 1408, and 1373 cm^{-1} ; HRMS-CI m/z 141.1273 $[(M - OH)^+]$; calcd for $\text{C}_9\text{H}_{17}\text{O}$: 141.1279].

1-(3-Butyl-oxiranyl)-2-methyl-propan-1-ol (28). The product was prepared by General Procedure B using 98 mg (0.55 mmol) Cy_2BH , 64 μL (0.55 mmol) 1-hexyne, 0.78 mL (1.55 mmol, 2.0 M in hexanes) Et_2Zn , 4.8 mg (0.02 mmol) (–)-MIB, 45 μL (0.50 mmol) isobutyraldehyde, 100 μL (0.10 mmol, 1.0 M in hexanes) $\text{Ti}(\text{OiPr})_4$, and 21 μL (0.10 mmol) (+)-DIPT. The crude product was purified by column chromatography (5% ethyl acetate in hexanes) to afford the title compound in 77% yield (66 mg, 0.38 mmol). *threo*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = -26.3$ ($c = 0.79$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.85 (t, 3H, $J = 7.1$ Hz), 0.91 (d, 3H, $J = 6.8$ Hz), 0.94 (d, 3H, $J = 6.8$ Hz), 1.27–1.41 (m, 4H), 1.47–1.52 (m, 2H), 1.75 (m, 1H), 1.84 (d, 1H, $J = 5.7$ Hz), 2.70 (dd, 1H, $J = 5.0$, 2.1 Hz), 2.82 (dt, 1H, $J = 5.7$, 1.6 Hz), and 3.10 (q, 1H, $J = 5.7$ Hz) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 13.9, 18.1, 18.4, 22.5, 28.0, 31.3, 32.5, 57.1, 60.3, and 76.0 ppm; IR (neat): 3414, 2959, 2930, 2862, 1466, 1381, 1366, 1280, and 1255 cm^{-1} ; LRMS-CI m/z 155.17 $[(M - OH)^+]$; calcd for $\text{C}_{10}\text{H}_{19}\text{O}$: 155.14]. *erythro*-diastereomer: Colorless oil. $[\alpha]_{\text{D}}^{20} = +8.1$ ($c = 0.36$, CHCl_3); ^1H NMR (CDCl_3 , 500 MHz): δ 0.85 (t, 3H, $J = 7.0$ Hz), 0.93 (d, 3H, $J = 6.2$ Hz), 0.94 (d, 3H, $J = 6.0$ Hz), 1.29–1.40 (m, 4H), 1.50 (dt, 2H, $J = 7.2$, 6.2 Hz), 1.75 (m, 1H), 1.78 (br s, 1H), 2.74 (t, 1H, $J = 2.4$ Hz), 2.94 (dt, 1H, $J = 5.4$, 2.0 Hz), and 3.51 (br t, 1H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 13.9, 17.8, 18.5, 22.5, 28.1, 31.3, 31.7, 54.9, 59.6, and 73.0 ppm; IR (neat): 3412, 2963, 2929, 2872, 1640, 1534, 1458, and 1381 cm^{-1} ; LRMS-CI m/z 155.17 $[(M - OH)^+]$; calcd for $\text{C}_{10}\text{H}_{19}\text{O}$: 155.14].

Conditions for the Determination of Enantiomeric Excess. The enantiomeric excess values for the following compounds were determined by chiral HPLC analysis. The conditions for the resolution of the racemates are described below.

(S)-4,4-Dimethyl-1-phenyl-pent-2-en-1-ol (1): Chiralcel OD-H; $t_1 = 9.2$ min, $t_2 = 11.5$ min (hexanes / 2-propanol: 95/5, 0.8 mL/min).

(S)-1-Phenyl-hept-2-en-1-ol (2): Chiralcel OD-H; $t_1 = 20.3$ min, $t_2 = 22.5$ min (hexanes / 2-propanol: 98/2, 0.5 mL/min).

(S)-1-(2-Bromo-phenyl)-4,4-dimethyl-pent-2-en-1-ol (3): Chiralcel OD-H; $t_1 = 13.4$ min, $t_2 = 16.4$ min (hexanes / 2-propanol: 97/3, 0.5 mL/min).

(S)-1-(2-Bromo-phenyl)-hept-2-en-1-ol (4): Chiralcel OD-H; $t_1 = 15.9$ min, $t_2 = 19.8$ min (hexanes / 2-propanol: 97/3, 0.5 mL/min).

(S)-1-(4-Trifluoromethyl-phenyl)-hept-2-en-1-ol (6): Chiralcel OB-H; $t_1 = 23.1$ min, $t_2 = 31.3$ min (hexanes / 2-propanol: 98/2, 0.5 mL/min).

(S)-4,4-Dimethyl-1-*p*-tolyl-pent-2-en-1-ol (8): Chiralcel OD-H; $t_1 = 18.8$ min, $t_2 = 21.8$ min (hexanes / 2-propanol: 98/2, 0.5 mL/min).

(S)-1-*p*-Tolyl-hept-2-en-1-ol (9): Chiralcel OD-H; $t_1 = 22.1$ min, $t_2 = 25.0$ min (hexanes / 2-propanol: 98/2, 0.5 mL/min).

(3-*tert*-Butyl-oxiranyl)-(4-trifluoromethyl-phenyl)-methanol (19): Chiralcel OD-H; $t_1 = 20.8$ min, $t_2 = 26.3$ min (hexanes / 2-propanol: 97/3, 0.5 mL/min).

The enantiomeric excess values for the following compounds were determined by chiral capillary GC analysis using a Supelco β -Dex 120 fused silica column (30m x 0.25 mm x 0.25 μ m film thickness). The carrier gas was nitrogen; inlet temperature was 250 °C; FID detector at 270 °C. The conditions for the resolution of the racemates are described below.

(S)-1-(4-Fluoro-phenyl)-4,4-dimethyl-pent-2-en-1-ol (7): $t_1 = 371.1$ min, $t_2 = 376.6$ min (110 °C, 0.5 mL/min).

(S)-1-Cyclohexyl-4,4-dimethyl-pent-2-en-1-ol (10): $t_1 = 98.4$ min, $t_2 = 101.5$ min (110 °C, 1.2 mL/min).

(S)-2,7,7-Trimethyl-oct-5-en-4-ol (11): $t_1 = 22.6$ min, $t_2 = 24.6$ min (110 °C, 0.8 mL/min).

(S)-6,6-Dimethyl-hept-4-en-3-ol (12): $t_1 = 48.0$ min, $t_2 = 49.3$ min (80 °C, 0.8 mL/min).

(S)-Non-4-en-3-ol (13): $t_1 = 79.6$ min, $t_2 = 81.7$ min (80 °C, 0.8 mL/min).

(S)-2-Methyl-non-4-en-3-ol (14): $t_1 = 35.6$ min, $t_2 = 37.0$ min (100 °C, 0.8 mL/min).

References

- (1) Oppolzer, W.; Radinov, R. N. *Helv. Chim. Acta* **1992**, 75, 170-173.