

General Procedures: All reactions were performed using oven dried glassware under an atmosphere of dry nitrogen. Toluene was distilled and dried before use (≤ 20 ppm H_2O as determined by Karl Fischer titration). Reagents were purchased from either Aldrich or Fluka chemical companies and used without prior purification except aldehydes which were distilled before use. Zinc triflate was purchased from Fluka chemical company at $\geq 98\%$ purity. Chromatographic purification of products was accomplished using forced flow chromatography on Fluka Silica Gel 60 according to the method of Still.¹ NMR spectra were recorded on a Varian Mercury 300 operating at 300 MHz and 75 MHz for ^1H and ^{13}C , respectively, and referenced to the internal solvent signals. IR spectra were recorded on a Perkin Elmer Spectrum RX I FT-IR spectrometer as thin film unless otherwise noted. Optical rotations were measured on a JASCO DID-1000 digital polarimeter. Thin layer chromatography was performed using Merck Silica Gel 60 F₂₅₄ TLC plates and visualized either with ultraviolet light or stained with CAM-Stain. HPLC analysis were carried out on a Merck Hitachi D-7000 system. Melting points were measured on a Büchi 510 apparatus and are uncorrected. Combustion analysis was performed by the Mikroelementaranalytisches Laboratorium at the ETH, Zürich.

General Procedure for the Nucleophilic Addition of Alkynes to Aldehydes in Toluene: A 10 mL flask was charged with $\text{Zn}(\text{OTf})_2$ (200 mg, 0.550 mmol, 1.1 eq) and (+)-*N*-Methylephedrine (108 mg, 0.602 mmol, 1.2 eq) and purged with nitrogen for 15 min. To the flask was added toluene (1.5 mL) and triethylamine (61 mg, 0.602 mmol, 1.2 eq). The resulting mixture was stirred at 23°C for 2 hours before the alkyne (0.600 mmol, 1.2 eq) was added by syringe in one portion. After 15 min of stirring the aldehyde was added in one portion by syringe. The reaction was quenched by the addition of saturated aqueous NH_4Cl solution (3 mL). The reaction mixture was poured into a separatory funnel containing diethyl ether (10 mL). The layers were separated and the aqueous layer was extracted with diethyl ether (3 x 10 mL). The combined organic layers were washed with brine (10 mL), dried over anhydrous MgSO_4 and concentrated in vacuo.

Purification of the material by chromatography on silica gel using a 95:5 mixture of pentane / diethyl ether afforded the secondary alcohol.

¹ W. C. Still, H. L. Ammon, P. DeShong, P. J. Am. Chem. Soc. **1995**, *117*, 5166.

(R)-1-Cyclohexyl-3-phenyl-2-propyn-1-ol²: Isolated in 99% yield and 96% ee as determined by HPLC analysis (Chiralcel OD, 10% *i*-PrOH in hexane, 254 nm) *t*_r 8.1 (major), 18.8 (minor); $[\alpha]_{\text{D}}^{26}$ -9.2° (*c* = 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.38-7.45 (m, 2H), 7.30-7.24 (m, 3H), 4.40-4.30 (t, 1H, *J* = 6.0 Hz), 2.00-1.58 (m, 1H), 1.36-1.05 (m, 5H); ¹³C NMR (75 MHz, CDCl₃) δ 131.7, 128.2, 122.7, 89.2, 85.6, 47.6, 44.3, 28.6, 28.2, 26.3, 25.9; FTIR (thin film) 3350, 3055, 2922, 2850, 2233, 1489, 1450, 1082, 1071, 1028 cm⁻¹; Anal. Calcd. For C₁₅H₁₈O: C, 84.07; H, 8.47. Found: C, 83.82; H, 8.56.

(R)-1-Cyclohexyl-5-phenyl-2-pentyn-1-ol: Isolated in 98% yield and 99% ee as determined by HPLC analysis (Chiralcel OD, 10% *i*-PrOH in hexane, 254 nm) *t*_r 11.2 (major), 26.0 (minor); $[\alpha]_{\text{D}}^{28}$ -1.7° (*c* = 1.1, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.30-7.18 (m, 5H), 4.11-4.05 (m, 1H), 2.83-2.78 (t, 2H, *J* = 7.5 Hz), 2.58-2.42 (dt, 2H, *J*₁ = 7.5 Hz, *J*₂ = 2.0 Hz), 1.81-1.57 (m, 6H), 1.50-0.90 (m, 6H); ¹³C NMR (75 MHz, CDCl₃) δ 140.6, 128.5, 128.3, 124.3, 85.4, 80.9, 67.4, 44.3, 35.0, 28.5, 27.9, 26.4, 25.9, 20.8; FTIR (thin film) 3367, 3026, 2924, 2851, 2233, 1604, 1497, 1452, 1078, 1011 cm⁻¹; Anal. Calcd. For C₁₇H₂₂O: C, 84.25; H, 9.15. Found: C, 83.99; H, 9.35.

(S)-1-Cyclohexyl-5-phenyl-2-pentyn-1-ol: Isolated in 97% yield and 99% ee as determined by HPLC analysis (Chiralcel OD, 10% *i*-PrOH in hexane, 254 nm) *t*_r 10.6 (minor), 23.1 (major); $[\alpha]_{\text{D}}^{26}$ +1.5° (*c* = 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.30-7.18 (m, 5H), 4.11-4.05 (m, 1H), 2.83-2.78 (t, 2H, *J* = 7.5 Hz), 2.58-2.42 (dt, 2H, *J*₁ = 7.5 Hz, *J*₂ = 2.0 Hz), 1.81-1.57 (m, 6H), 1.50-0.90 (m, 6H); ¹³C NMR (75 MHz, CDCl₃) δ 140.6, 128.5, 128.3, 124.3, 85.4, 80.9, 67.4, 44.3, 35.0, 28.5, 27.9, 26.4, 25.9, 20.8; FTIR (thin film) 3367, 3026, 2924, 2851, 2233, 1604, 1497, 1452, 1078, 1011 cm⁻¹; Anal. Calcd. For C₁₇H₂₂O: C, 84.25; H, 9.15. Found: C, 84.08; H, 9.39.

(R)-2-Methyl-7-phenyl-4-heptyn-3-ol: Isolated in 90% yield and 99% ee as determined by HPLC analysis (Chiralcel OD, 10% *i*-PrOH in hexane, 254 nm) *t*_r 8.4 (major), 15.2 (minor); $[\alpha]_{\text{D}}^{23}$ +1.7° (*c* = 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.30-7.17 (m, 5H), 4.18-4.05 (m, 1H), 2.85-2.78 (t, 2H, *J* = 7.5 Hz), 2.55-2.45 (dt, 2H, *J*₁ = 7.5

² E. J. Corey, K. A. Cimprich, *J. Am. Chem. Soc.* **1994**, *116*, 3151.

Hz, $J_2 = 1.9$ Hz), 1.90-1.70 (m, 1H), 1.70-1.62 (br, 1H), 1.00-0.90 (2d, 6H, $J = 4.2$ Hz); ^{13}C NMR (75 MHz, CDCl_3) δ 140.6, 128.4, 128.3, 126.3, 85.3, 80.6, 68.1, 35.0, 34.6, 20.8, 18.0, 17.3; FTIR (thin film) 3390, 3027, 2959, 2928, 2870, 2236, 1603, 1496, 1366, 1073 cm^{-1} ; Anal. Calcd. For $\text{C}_{14}\text{H}_{18}\text{O}$: C, 83.12; H, 8.97. Found: C, 82.89; H, 9.04.

(R)-4-Methyl-1-phenyl-1-pentyn-3-ol³: Isolated in 95% yield and 90% ee as determined by HPLC analysis (Chiralcel OD, 10% *i*-PrOH in hexane, 254 nm) t_r 7.9 (major), 16.0 (minor); $[\alpha]_D^{23} +3.2^\circ$ ($c = 6.8$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.42-7.38 (m, 2H), 7.30-7.21 (m, 3H), 4.40-4.35 (t, 1H, $J = 6.0$ Hz), 2.09-2.00 (d, 1H, $J = 5.7$ Hz), 2.01-1.90 (m, 1H), 1.10-1.00 (dd, 6H, $J = 6.7$ Hz); ^{13}C NMR (75 MHz, CDCl_3) δ 131.7, 128.3, 128.2, 122.7, 88.9, 85.5, 63.3, 34.6, 18.1, 17.5; FTIR (thin film) 3367, 3057, 2961, 2928, 2221, 1548, 1489, 1028 cm^{-1} ; Anal. Calcd. For $\text{C}_{12}\text{H}_{14}\text{O}$: C, 82.72; H, 8.10. Found: C, 82.48; H, 8.13.

(R)-1,7-Diphenylhept-1-en-4-yn-3-ol: Isolated in 39% yield and 80% ee as determined by HPLC analysis (Chiralcel OD-H, 15% *i*-PrOH in hexane, 254 nm) t_r 47.2 (minor), 53.8 (major); $[\alpha]_D^{23} +7.6^\circ$ ($c = 0.4$, CHCl_3), mp: 53-55°C, ^1H NMR (300 MHz, CDCl_3) δ 7.43-7.18 (m, 10H), 6.71 (d, 1H, $J = 15.9$ Hz), 6.27 (dd, 1H, $J_1 = 15.9$ Hz, $J_2 = 5.9$ Hz), 5.06-4.99 (m, 1H), 2.87 (t, 2H, $J = 7.5$ Hz), 2.58 (dt, 2H, $J_1 = 7.5$ Hz, $J_2 = 2.2$ Hz), 1.85 (d, 1H, $J = 6.2$ Hz); ^{13}C NMR (75 MHz, CDCl_3) δ 140.5, 136.2, 131.6, 128.63, 128.60, 128.5, 128.4, 128.0, 126.8, 126.4, 86.7, 80.0, 63.2, 35.0, 21.0; FTIR (CHCl_3) 4213, 3619, 3438, 3010, 2975, 2400, 1521, 1419, 1219, 1045, 930, 670 cm^{-1} ; Anal. Calcd. For $\text{C}_{19}\text{H}_{18}\text{O}$: C, 86.99; H, 6.92. Found: C, 86.93; H, 6.99.

(R)-2,2-Dimethyl-7-phenyl-4-heptyn-3-ol: Isolated in 84% yield and 99% ee as determined by HPLC analysis (Chiralcel OD, 10% *i*-PrOH in hexane, 254 nm) t_r 7.6 (major), 19.7 (minor); $[\alpha]_D^{28} +4.4^\circ$ ($c = 1.4$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.30-7.18 (m, 5H), 3.98-3.90 (br, 1H), 2.85-2.78 (t, 2H, $J = 7.7$ Hz), 2.56-2.45 (dt, 2H, $J_1 = 7.7$ Hz, $J_2 = 2.3$ Hz), 1.76-1.70 (br, 1H), 0.95-0.90 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 140.5, 128.4, 128.3, 126.2, 85.3, 80.6, 71.5, 35.7, 35.0, 25.2, 20.8; FTIR (thin film)

³ K. Matsumura, S. Hashiguchi, T. Ikariya, R. Noyori, *J. Am. Chem. Soc.* **1997**, *119*, 8738.

3419, 3028, 2954, 2868, 2224, 1603, 1496, 1479, 1454, 1394, 1363, 1133, 1041, 1004 cm^{-1} ; Anal. Calcd. For $\text{C}_{15}\text{H}_{20}\text{O}$: C, 83.29; H, 9.32. Found: C, 83.35; H, 9.21.

(R)-4,4-Dimethyl-1-phenyl-1-pentyn-3-ol²: Isolated in 99% yield and 94% ee as determined by HPLC analysis (Chiralcel OD, 10% *i*-PrOH in hexane, 254 nm) t_r 7.4 (major), 10.1 (minor); $[\alpha]_D^{30} +2.4^\circ$ ($c = 4.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.46-7.38 (m, 2H), 7.31-7.27 (m, 3H), 4.24-4.20 (d, 1H, $J = 6.3$ Hz), 2.00-1.90 (br, 1H), 1.07-1.02 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 131.7, 128.3, 128.2, 122.8, 88.9, 85.7, 71.8, 36.1, 25.3; FTIR (thin film) 3391, 3058, 2965, 2849, 2222, 1548, 1489, 1055, 1007 cm^{-1} ; Anal. Calcd. For $\text{C}_{13}\text{H}_{16}\text{O}$: C, 82.94; H, 8.57. Found: C, 82.78; H, 8.47.

(R)-1,5-Diphenyl-2-pentyn-1-ol: Isolated in 52% yield and 96% ee as determined by HPLC analysis (Chiralcel OD, 10% *i*-PrOH in hexane, 254 nm) t_r 21.8 (major), 35.6 (minor); $[\alpha]_D^{29} -5.9^\circ$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.50-7.43 (m, 2H), 7.40-7.20 (m, 8H), 5.45-5.39 (br, 1H), 2.90-2.80 (t, 2H, $J = 7.5$ Hz), 2.62-2.58 (dt, 2H, $J_1 = 7.5$ Hz, $J_2 = 2.0$ Hz), 2.25-2.18 (br, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 141.0, 140.4, 128.5, 128.4, 128.2, 126.6, 126.3, 86.7, 80.8, 64.7, 34.8, 20.9; FTIR (thin film) 3369, 3061, 3027, 2927, 2225, 1603, 1496, 1456 cm^{-1} ; Anal. Calcd. For $\text{C}_{17}\text{H}_{16}\text{O}$: C, 86.41; H, 6.82. Found: C, 86.35; H, 6.99.

(R)-1,3-Diphenyl-2-propyn-1-ol²: Isolated in 53% yield and 94% ee as determined by HPLC analysis (Chiralcel OD-H, 10% *i*-PrOH in hexane, 254 nm) t_r 13.1 (major), 23.0 (minor); $[\alpha]_D^{25} +7.4^\circ$ ($c = 1.4$, CHCl_3); ^1H NMR, ^{13}C NMR and FTIR data match with the published literature²; Anal. Calcd. For $\text{C}_{15}\text{H}_{12}\text{O}$: C, 86.51; H, 5.81. Found: C, 86.34; H, 5.98.

(R)-1-Cyclohexyl-3-(trimethylsilyl)-2-propyn-1-ol⁴: Isolated in 93% yield and 98% ee as determined by HPLC analysis of the benzoate ester (Chiralcel OD, hexane, 254 nm) t_r 6.2 (minor), 7.0 (major). $[\alpha]_D^{27} -6.7^\circ$ ($c = 1.2$, CHCl_3), $[\alpha]_D^{28} -6.0^\circ$ ($c = 3.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 4.16-4.09 (t, 1H, $J = 5.4$ Hz), 1.90-1.72 (m,

⁴ J. Bach, R. Berenguer, J. Garcia, T. Loscertales, J. Vilarrasa, *J. Org. Chem* **1996**, 61, 9021.

5H), 1.71-1.61 (m, 1H), 1.60-1.46 (m, 1H), 1.34-0.96 (m, 5H), 0.17 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 105.9, 90.2, 67.6, 44.0, 28.5, 28.1, 26.4, 25.9, -0.1; FTIR (thin film) 3350, 2926, 2853, 2360, 2170, 1451, 1405, 1250, 1083, 1027, 984, 843 cm^{-1} ; Anal. Calcd. For $\text{C}_{12}\text{H}_{22}\text{OSi}$: C, 68.51; H, 10.54. Found: C, 68.28; H, 10.73;

A small amount of the alcohol was converted into the corresponding benzoate ester (benzoyl chloride, pyridine, DMAP, CH_2Cl_2 , 23°C , 14h)² and the product purified by chromatography on silica gel prior to analysis by HPLC.

(R)-2,2-Dimethyl-8-phenyl-5-octyn-4-ol: Isolated in 72% yield and 99% ee as determined by HPLC analysis (Chiralcel OD, 10% *i*-PrOH in hexane, 254 nm) t_r 8.5 (major), 25.5 (minor); $[\alpha]_D^{28} +13.7^\circ$ ($c = 0.6$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.35-7.10 (m, 5H), 4.45-4.30 (m, 1H), 2.83-2.70 (t, 2H, $J = 7.3$ Hz), 2.50-2.40 (dt, 2H, $J_1 = 7.3$ Hz, $J_2 = 2.1$ Hz), 1.65-1.55 (d, 2H, $J = 6.3$ Hz), 1.60-1.50 (br, 1H), 1.00-0.85 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 140.7, 128.5, 128.4, 126.4, 84.5, 83.5, 60.3, 51.7, 34.9, 29.9, 29.8, 20.8; FTIR (thin film) 3367, 3081, 2953, 2867, 2229, 1599, 1490, 1366, 1070, 1060, 1024, 1004 cm^{-1} ; Anal. Calcd. For $\text{C}_{16}\text{H}_{22}\text{O}$: C, 88.43; H, 9.63. Found: C, 83.40; H, 9.66.

(R)-5,5-Dimethyl-1-phenyl-1-hexyn-3-ol: Isolated in 90% yield and 97% ee as determined by HPLC analysis (Chiralcel OD, 10% *i*-PrOH in hexane, 254 nm) t_r 8.2 (major), 17.2 (minor); $[\alpha]_D^{25} +16.8^\circ$ ($c = 1.3$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.47-7.36 (m, 2H), 7.30-7.20 (m, 3H), 4.70-4.60 (q, 1H, $J = 5.8$ Hz), 1.90-1.80 (d, 1H, $J = 5.8$ Hz), 1.80-1.70 (dd, 2H, $J_1 = 7.5$ Hz, $J_2 = 1.5$ Hz), 1.05-0.90 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 131.6, 128.8, 122.9, 91.5, 84.7, 60.7, 51.6, 30.0, 29.9; FTIR (thin film) 3367, 3001, 2953, 2867, 2229, 1599, 1490, 1366, 1070, 1024 cm^{-1} ; Anal. Calcd. For $\text{C}_{14}\text{H}_{18}\text{O}$: C, 83.12; H, 8.97. Found: C, 82.85; H, 8.92.

(R)-1-Cyclohexyl-4-(trimethylsilyl)-2-butyn-1-ol: Isolated in 84% yield and 98% ee as determined by HPLC analysis of the corresponding 3,5-dinitrobenzoate ester (Chiralcel OD, 10% *i*-PrOH in hexane, 254 nm) t_r 11.7 (major), 17.1 (minor); $[\alpha]_D^{27} +1.6^\circ$ ($c = 1.3$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 4.16-4.11 (m, 1H), 1.88-1.60 (m, 6H), 1.51 (s, 1H), 1.50 (s, 1H), 1.33-0.97 (m, 5H), 0.10 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 84.0, 67.7, 44.5, 28.0, 26.5, 26.0, 25.9, 7.1, -2.0; FTIR (thin film)

3391, 2925, 2852, 2339, 2215, 1646, 1451, 1249, 1140, 1008, 851 cm^{-1} ; Anal. Calcd. For $\text{C}_{13}\text{H}_{24}\text{OSi}$: C, 69.58; H, 10.78. Found: C, 69.39; H, 10.64;

A small amount of the alcohol was converted into the corresponding 3,5-dinitrobenzoate ester (3,5-dinitrobenzoyl chloride, pyridine, DMAP, CH_2Cl_2 , 23°C , 14h) and the product purified by chromatography on silica gel prior to analysis by HPLC.

(R)-1-Cyclohexyl-4-(tert-butyldimethylsilyloxy)-2-butyn-1-ol: Isolated in 83% yield and 98% ee as determined by HPLC analysis of the corresponding Mosher ester (Chiralcel OD, 2% *i*-PrOH in hexane, 254 nm) t_r 5.5 (major), 7.4 (minor); $[\alpha]_D^{25}$ -2.6° ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 4.35 (d, 2H, $J = 0.9$ Hz), 4.21-4.15 (m, 1H), 1.90-1.72 (m, 4H), 1.71-1.61 (m, 1H), 1.61-1.47 (m, 1H), 1.33-0.98 (m, 5H), 0.91 (s, 9H), 0.12 (s, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 84.8, 67.2, 51.7, 44.1, 28.5, 28.1, 26.4, 25.9, 25.8, 18.3, -5.1; FTIR (thin film) 3392, 2928, 2855, 2360, 1472, 1451, 1362, 1255, 1128, 1085, 837, 778 cm^{-1} ; Anal. Calcd. For $\text{C}_{16}\text{H}_{30}\text{O}_2\text{Si}$: C, 68.03; H, 10.70. Found: C, 68.02; H, 10.62.

A small amount of the alcohol was converted into the corresponding Mosher ester⁵ ((R)-MTPA chloride, pyridine, DMAP, CH_2Cl_2 , 23°C , 14h) and the product purified by chromatography on silica gel prior to analysis by HPLC.

(R)-1-Cyclohexyl-4,4-diethoxy-2-butyn-1-ol: Isolated in 90% yield and 98% ee as determined by HPLC analysis of the benzoate ester (Chiralcel OD, 2% *i*-PrOH in hexane, 254 nm) t_r 8.3 (major), 10.1 (minor); $[\alpha]_D^{29}$ -5.4° ($c = 1.5$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 5.25 (s, 1H), 4.20-4.10 (t, 1H, $J = 5.8$), 3.75-3.62 (m, 2H), 3.60-3.50 (m, 2H), 2.12-2.05 (d, 1H, $J = 6.1$ Hz), 1.90-1.45 (m, 6H), 1.22-1.10 (t, 6H, $J = 6.8$ Hz), 1.20-0.95 (m, 4H); ^{13}C NMR (75 MHz, CDCl_3) δ 91.2, 85.5, 80.9, 66.9, 60.9, 60.8, 43.8, 28.4, 28.1, 26.3, 25.8, 15.0; FTIR (thin film) 3436, 2976, 2929, 2240, 1451, 1343, 1328, 1143, 1049 cm^{-1} ; Anal. Calcd. For $\text{C}_{14}\text{H}_{24}\text{O}_3$: C, 69.96; H, 10.06. Found: C, 69.70; H, 10.20.

A small amount of the alcohol was converted into the corresponding benzoate ester (benzoyl chloride, pyridine, DMAP, CH_2Cl_2 , 23°C , 14h)² and the product purified by chromatography on silica gel prior to analysis by HPLC.

⁵ J. A. Dale, D.L. Dull, H.S. Mosher, *J. Org. Chem.* **1969**, *34*, 2543.

(R)-1-Cyclohexyl-4-methyl-4-penten-2-yn-1-ol: Isolated in 94% yield and 98% ee as determined by HPLC analysis (Chiralcel AD-C, 2% *i*-PrOH in hexane, 254 nm) *t*_r 10.7 (minor), 11.9 (major); $[\alpha]_D^{25}$ -13.0° (*c* = 0.9, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 5.28 (s, 1H), 5.22 (s, 1H), 4.29-4.22 (t, 1H, *J* = 6.0 Hz), 1.89 (s, 3H), 1.87-1.72 (m, 4H), 1.72-1.63 (m, 1H), 1.63-1.49 (m, 1H), 1.34-0.98 (m, 5H); ¹³C NMR (75 MHz, CDCl₃) δ 126.4, 121.9, 88.3, 67.6, 44.3, 28.6, 28.2, 26.4, 25.9, 23.5; FTIR (thin film) 3350, 2925, 2852, 2667, 2360, 1792, 1615, 1451, 1373, 1287, 1011, 893 cm⁻¹; Anal. Calcd. For C₁₂H₁₈O: C, 80.85; H, 10.18. Found: C, 80.74; H, 10.12.

(R)-2,6-Dimethylhept-3-yn-2,5-diol: Isolated in 97% yield and 98% ee as determined by HPLC analysis of the corresponding 3,5-dinitrobenzoate ester (Chiralcel OD-H, 10% *i*-PrOH in hexane, 254 nm) *t*_r 43.4 (major), 53.9 (minor); $[\alpha]_D^{23}$ -1.5° (*c* = 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 4.18 (t, 1H, *J* = 5.2 Hz), 3.00 (s, 1H), 2.83 (d, 1H, *J* = 5.1 Hz), 1.92-1.80 (m, 1H), 1.52 (s, 6H), 0.99 (d, 3H, *J* = 6.7 Hz), 0.97 (d, 3H, *J* = 6.7 Hz); ¹³C NMR (75 MHz, CDCl₃) δ 90.4, 81.8, 67.7, 65.1, 34.4, 31.4, 18.2, 17.5; FTIR (thin film) 3338, 2980, 2932, 2874, 2342, 1654, 1459, 1365, 1236, 1166, 1021, 952, 861 cm⁻¹; Anal. Calcd. For C₉H₁₆O₂: C, 69.19%; H, 10.32%. Found: C, 69.24%; H, 10.21%. A small amount of the alcohol was converted into the corresponding 3,5-dinitrobenzoate ester (3,5-dinitrobenzoyl chloride, pyridine, DMAP, CH₂Cl₂, 23°C, 14h) and the product purified by chromatography on silica gel prior to analysis by HPLC.