Supplementary Material (ESI) for Chemical Communications

GENERAL

Reactions which required the use of anhydrous, inert atmosphere techniques were carried out under an atmosphere of argon. Reactions with ruthenium catalysts were performed under an atmosphere of argon using standard Schlenk line techniques. In all cases, solvents were distilled or obtained by passing through anhydrous alumina columns using an Innovative Technology Inc. PS-400-7 solvent purification system. Solvents were rigorously freeze/thaw degassed where required and stored in sealed Young's ampoules prior to use, apparatus was oven dried or flame dried under vacuum and purged with inert gas by three cycles of high vacuum and replacement with inert gas. Diethyl ether and tetrahydrofuran were distilled from the anion of benzophenone ketyl radical. Hexane and toluene were distilled from sodium wire. Acetonitrile, *iso*-propanol and dichloromethane were distilled from calcium hydride

TLC using plastic backed plates precoated with Macherey-Nagel Sil G/$UV_{254\ nm}$ neutral silica were used to monitor reactions. Visualisation of TLC plates was by 254 nm UV light and/or $KMnO_4$ dip followed by gentle warming. TLC data quoted for specific compounds indicate the most suitable method of visualisation. Organic layers were routinely dried with anhydrous $MgSO_4$ or Na_2SO_4 and evaporated using a Büchi evaporator, or high vacuum line. Where necessary, further drying was obtained by high vacuum. Flash chromatography was carried out using Davisil LC 60A silica gel (35-70 micron) purchased from Fluorochem. Purification by kugelrohr distillation refers to the use of kugelrohr distillation apparatus under high vacuum, at a pressure between 0.3 – 0.1 mmHg. Where products are separated from cyclohexanol, complete removal of cyclohexanol could be achieved between room temperature and 40 ºC within this pressure range. After complete removal of cyclohexanol further slow increases in the oven temperature to between 50 – 150 ºC were required for isolation of products.

NMR spectra were run in $CDCl_3$, C_6D_6, d_8-PhMe, d_6-DMSO or d_4-MeOH on either a Bruker Avance 300 (300 MHz) or Bruker Avance 400 (400 MHz) instrument and recorded at the following frequencies: proton (^1H – 300/400 MHz), carbon (^{13}C – 75.4/100.5 MHz), phosphorus ($^{31}P\{^1H\}$ – 121.4/161.9 MHz). Chemical shifts are reported relative to the residual solvent peak where possible or alternatively to $SiMe_4$

(δ = 0.00 ppm) as internal standard. Coupling constants (*J*) are given in Hz and multiplicities denoted as singlet (s), doublet (d), triplet (t), quartet (q), pentet (pent), sextet, septet (sep), unresolved multiplet (m), broad (br.) or apparent (app.) Signals are assigned relative to the numbered structure according to the numbered atoms (e.g. H_1 or C_1) or as general assignments classified as (Ar) aromatic, (C) quaternary carbon, (CH) methyne carbon, (CH_2) methylene carbon and (CH_3) methyl carbon. Structural assignments of both protons and carbons were achieved with the aid of COSY, HMQC and PENDANT experiments where required and with comparisons from analogous literature compounds.

IR spectra were recorded as either liquid films or KBr disks using a Nicolet NEXUS FT-IR spectrometer (with internal background scan). Absorption maxima (ν_{max}) are recorded in wavenumbers (cm^{-1}) and classified as strong (s), medium (m), weak (w) or broad (br.).

Mass spectra, including high resolution spectra, were recorded using electron impact (EI+) ionisation, chemical ionisation (CI+ using ammonia) at the EPSRC national mass spectrometry service centre, University of Wales Swansea.

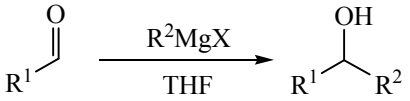
Melting points are recorded on a Büchi 535 series instrument and are uncorrected.

GC was performed using a Fisons Instruments 8000GC Series instrument where a detector temperature of 250 ºC, injector temperature of 250 ºC and flow rate of 1 ml/min were routinely used. Columns and oven temperatures are outlined in the relevant experiment. High pressure liquid chromatography was carried out using a spectra SERIES P200 using Chiralcel OD®, OD-H®, AD®, AD-H®, OB, OC and OJ columns obtained from Fisher Scientific Supplies; the column, solvent and flow rate used are detailed in the relevant experiment.

Specific rotations were determined on an Optical Activity LTD: AA-10 automatic polarimeter.

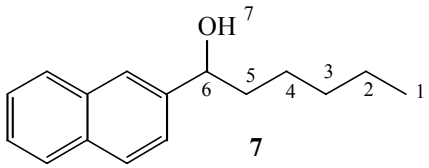
Unless preparative details are provided, all chemicals were commercially available and purchased from either Acros organics, Aldrich, Alfa Aesar, Avocado, Fluka, Lancaster or Strem chemical companies.

Procedure 1. Preparation of secondary alcohols from aldehydes



To an adequately sized (50 – 250 mL) oven dried round bottomed flask purged with argon was added the aldehyde (1 eq), followed by anhydrous and degassed THF (5 – 50 mL). The resulting solution was cooled to 0 ºC in an ice bath, and the round bottomed flask purged with argon. The required Grignard reagent (1-1.33 eq) was slowly added. The reaction was stirred for a further one hour at 0 ºC then allowed to warm to room temperature and stirred for a further one hour. The reaction was quenched by slow addition of a 2M solution of HCl until the mixture reached pH 6 unless stated otherwise. The resulting mixture was extracted three times with DCM, the organics were collected and dried over $MgSO_4$unless stated otherwise. Concentration *in vacuo* provided the crude alcohol product which was purified by distillation under vacuum using a kugelrohr distillation apparatus, or by column chromatography.

Preparation of 1-(2-naphthyl)hexanol ***7***

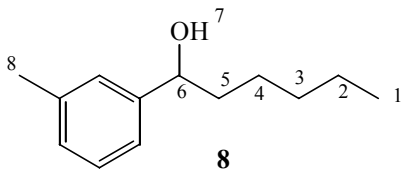


7

Following procedure *5.4.4*, using 2-naphthaldehyde (1.56 g, 10 mmol, 1 eq), THF (20 mL) and pentylmagnesium bromide (6 mL, 2M soln in diethylether, 12 mmol, 1.2 eq). The product alcohol was isolated by column chromatography (17:3 petroleum ether (b.p. 40–60 °C)/diethyl ether, $R_f = 0.16$) to yield 1-(2-naphthyl)hexanol **7**[1] as a white solid (1.98 g, 87%). ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): $\delta = 0.70 – 0.84$ (3H, m, H_1),

1.09 – 1.42 (6H, m, H_{2-4}), 1.59 – 1.83 (2H, m, H_5), 2.05 – 2.15 (1H, m, H_7), 4.68 (1H, t, J = 6.6 Hz, H_6), 7.29 – 7.49 (3H, m, H_{Ar}), 7.64 (1H, s, H_{Ar}), 7.67 – 7.77 (3H, m, H_{Ar}). ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C): δ = 14.5 (C_1), 23.0 (C_2), 26.0 (C_3), 32.2 (C_4), 39.4 (C_5), 75.2 (C_6), 124.6 (CH_{Ar}), 125.0 (CH_{Ar}), 126.2 (CH_{Ar}), 126.5 (CH_{Ar}), 128.1 (CH_{Ar}), 128.4 (CH_{Ar}), 128.7 (CH_{Ar}), 133.4 (C_{Ar}), 133.7 (C_{Ar}), 142.7 (C_{Ar}). IR (KBr Disk): ν_{max} (cm^{-1}) = 3273 (br. s, OH), 3055 (m), 2956 (s), 2930 (s), 2855 (s), 1923 (w), 1601 (m), 1507 (m), 1466 (m), 1369 (m), 1318 (m), 1271 (m), 1171 (w), 1144 (w), 1127 (m), 1103 (m), 1036 (m), 995 (w), 970 (w), 949 (m), 930 (w), 896 (m), 860 (m), 826 (s), 774 (m), 747 (s), 662 (w), 586 (w), 479 (m).

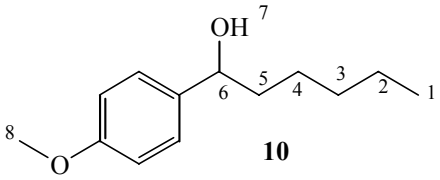
*Preparation of 1-(3-methylphenyl)hexanol **8***



8

Following procedure *5.4.4*, using 3-methylbenzaldehyde (1.20 g, 10 mmol, ρ = 1.019 gmL^{-1}, 1.18 mL, 1 eq), THF (20 mL) and pentylmagnesium bromide (6 mL, 2M soln in diethylether, 12 mmol, 1.2 eq). The product alcohol was isolated by distillation to yield 1-(3-methylphenyl)hexanol **8**[2] as a colourless liquid (1.73 g, 90%). ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 0.72 – 0.88 (3H, m, H_1), 1.11 – 1.40 (6H, m, H_{2-4}), 1.51 – 1.78 (2H, m, H_5), 1.99 (1H, s, H_7), 2.27 (3H, s, H_{11}), 4.50 (1H, dd, J = 7.4 and 6.0 Hz, H_6), 6.97 – 7.17 (4H, m, H_{Ar}). ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C): δ = 14.5 (C_1), 21.9 (C_8), 23.0 (C_2), 26.0 (C_3), 32.2 (C_4), 39.5 (C_5), 75.1 (C_6), 123.4 (CH_{Ar}), 127.0 (CH_{Ar}), 128.6 (CH_{Ar}), 128.7 (CH_{Ar}), 138.4 (C_{Ar}), 145.4 (C_{Ar}). IR (liquid film): ν_{max} (cm^{-1}) = 3350 (br. s, OH), 3104 (m), 3027 (s), 2952 (s), 2731 (m), 1937 (w), 1870 (w), 1786 (w), 1691 (w), 1608 (s), 1591 (m), 1489 (s), 1457 (s), 1379 (s), 1312 (m), 1258 (m), 1158 (m), 1112 (m), 1091 (m), 1031 (s), 938 (m), 903 (m), 882 (m), 847 (w), 832 (w), 787 (s), 704 (s).

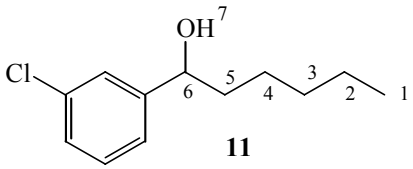
*Preparation of 1-(4-methoxyphenyl)hexanol **10***



10

Following procedure *5.4.4*, using 4-methoxybenzaldehyde (1.36 g, 10 mmol, ρ = 1.119 gmL^{-1}, 1.22 mL, 1 eq), THF (20 mL) and pentylmagnesium bromide (6 mL, 2M soln in diethylether, 12 mmol, 1.2 eq). The product alcohol was isolated by column chromatography (8:2 petroleum ether (b.p. 40–60 °C)/diethyl ether, R_f = 0.17) to yield 1-(4-methoxyphenyl)hexanol **10** [3] as a colourless liquid (2.04 g, 98%). ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 0.72 – 0.84 (3H, m, H_1), 1.10 – 1.39 (6H, m, $H_{2\text{-}4}$), 1.52 – 1.79 (3H, m, $H_{5,7}$), 3.73 (3H, s, H_{12}), 4.53 (1H, t, *J* = 6.8 Hz, H_6), 6.75 – 9.84 (2H, m, H_{Ar}), 7.14 – 7.22 (2H, m, H_{Ar}). ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C): δ = 14.4 (C_1), 23.0 (C_2), 26.0 (C_3), 32.1 (C_4), 39.4 (C_5), 55.7 (C_8), 74.7 (C_6), 114.2 ($2CH_{Ar}$), 127.5 ($2CH_{Ar}$), 137.5 (C_{Ar}), 159.4 (C_{Ar}). IR (liquid film): ν_{max} (cm^{-1}) = 3378 (br. m, OH), 2999 (m), 2956 (s), 2931 (s), 2859 (s), 2062 (w), 1886 (w), 1656 (w), 1612 (s), 1586 (m), 1513 (s), 1466 (m), 1442 (m), 1377 (m), 1303 (m), 1246 (s), 1203 (w), 1174 (s), 1114 (m), 1037 (m), 925 (m), 831 (s), 810 (m), 728 (w).

*Preparation of 1-(3-chlorophenyl)hexanol **11***

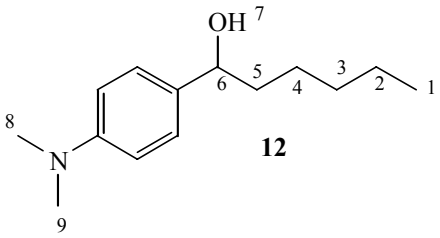


11

Following procedure *5.4.4*, using 3-chlorobenzaldehyde (1.40 g, 10 mmol, ρ = 1.241 gmL^{-1}, 1.128 mL, 1 eq), THF (50 mL) and pentylmagnesium bromide (6 mL, 2M soln in diethylether, 12 mmol, 1.2 eq). The product alcohol was isolated by column

chromatography (9:1 petroleum ether (b.p. 40–60 °C)/diethyl ether, $R_f = 0.12$) to yield 1-(3-chlorophenyl)hexanol **11** as a colourless liquid (1.47 g, 69%). ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 0.74 – 0.85 (3H, m, H_1), 1.11 – 1.41 (6H, m, H_{2-4}), 1.51 – 1.74 (2H, m, H_5), 1.98 (1H, br. s, H_7), 4.54 (1H, dd, J = 7.4 and 5.8 Hz, H_6), 7.08 – 7.28 (4H, m, H_{Ar}). ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C): δ = 14.4 (C_1), 22.9 (C_2), 25.8 (C_3), 32.0 (C_4), 39.5 (C_5), 74.4 (C_6), 124.4 (CH_{Ar}), 126.5 (CH_{Ar}), 127.9 (CH_{Ar}), 130.1 (CH_{Ar}), 134.7 (C_{Ar}), 147.4 (C_{Ar}). IR (liquid film): ν_{max} (cm^{-1}) = 3350 (br. s, OH), 3064 (w), 2957 (s), 2932 (s), 2859 (s), 1937 (w), 1868 (w), 1760 (w), 1685 (w), 1589 (m), 1575 (m), 1468 (s), 1433 (s), 1379 (m), 1342 (m), 1302 (m), 1265 (m), 1199 (m), 1117 (m), 1094 (m), 1078 (m), 1056 (m), 1029 (m), 1000 (m), 930 (m), 882 (m), 816 (w), 786 (s, C–Cl), 727 (w), 698 (s).

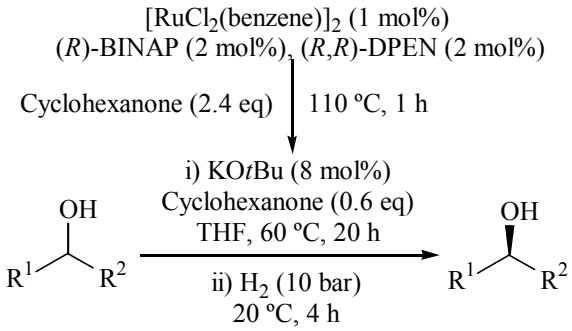
*Preparation of 1-(4-dimethylaminophenyl)hexanol **12***



12

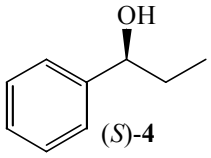
Following procedure *5.4.4*, using 4-(dimethylamino)benzaldehyde (1.49 g, 10 mmol, 1 eq), THF (20 mL) and pentylmagnesium bromide (6 mL, 2M soln in diethylether, 12 mmol, 1.2 eq). The product alcohol was isolated by distillation followed by column chromatography (40:1 petroleum ether (b.p. 40–60 °C)/diethyl ether, R_f = 0.16) to yield 1-(4-dimethylaminophenyl)hexanol **12**[4] as a white solid (1.38 g, 62%). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 0.88 – 1.06 (3H, m, H_1), 1.22 – 1.49 (6H, m, H_{2-4}), 1.64 – 1.74 (1H, m, $H_{5a/b}$), 1.78 – 1.88 (1H, m, $H_{5b/a}$), 1.92 – 2.56 (1H, br. s, H_7), 2.95 (6H, s, $H_{8,9}$), 4.54 (1H, dd, J = 6.6 and 6.6, H_6), 6.77 – 6.83 (2H, m, H_{Ar}), 7.26 – 7.31 (2H, m, H_{Ar}). ^{13}C NMR (100.5 MHz, $CDCl_3$, 25 °C): δ = 14.2 (C_1), 22.7 (C_2), 25.8 (C_3), 31.9 (C_4), 38.8 (C_5), 40.8 ($C_{12,13}$), 74.4 (C_6), 112.7 ($2CH_{Ar}$), 127.0 ($2CH_{Ar}$), 133.3 (C_{Ar}), 150.2 (C_{Ar}). IR (KBr Disk): ν_{max} (cm^{-1}) = 3261 (br. s, OH), 2954 (s), 2921 (s), 2857 (s), 2804 (m), 1616 (s), 1569 (w), 1559 (w), 1523 (s), 1468 (m), 1445 (m), 1356 (s), 1317 (m), 1231 (m), 1189 (m), 1167 (m), 1103 (m), 1068 (m), 1031 (m), 987 (w), 950 (w), 928 (w), 812 (s), 727 (w), 683 (w), 577 (w), 555 (w).

Procedure 2. Application of deracemisation II



To a flame dried and argon purged Schlenk tube containing $[RuCl_2(benzene)]_2$ (10 mg, 0.02 mmol, 0.01 eq), (*R*)-BINAP (*R*)-**1** (25.4 mg, 0.04 mmol, $[\alpha]_D^{23}$ + 221º (*c* = 0.326 g/100 mL, benzene), (lit.[5] $[\alpha]_D$ + 229 (*c* = 0.32 g/100 mL, benzene) (*R*)), 0.02 eq) and (*R*,*R*)-DPEN (*R*,*R*)-**3** (8.4 mg, 0.04 mmol, $[\alpha]_D^{23}$ + 102° (*c* = 1 g/100 mL, MeOH), (lit.[6] $[\alpha]_D$ +102° (*c* = 1 g/100 mL, MeOH) (*R*)), 0.02 eq) was added cyclohexanone (474 mg, 4.8 mmol, ρ = 0.947 gmL^{-1}, 500 µL, 2.4 eq). The reaction was heated for 1 hour at 110 ºC and allowed to cool to room temperature. To the reaction mixture was added, cyclohexanone (114 mg, 1.2 mmol, ρ = 0.947 gmL^{-1}, 120 µL, 0.6 eq), the alcohol substrate (2 mmol, 1 eq), and THF (2 mL) followed by potassium *tert*-butoxide (18 mg, 0.16 mmol, 0.08 eq) under argon. The reaction was heated at 60 ºC for 20 hours, after which it was allowed to cool, and was transferred via cannula under argon pressure into a Parr® hydrogen bomb reactor which had been purged with a rapid flow of argon for 15 minutes prior to use. The bomb was sealed and pressurised to 10 bar with hydrogen and the reaction was stirred at 20 ºC for 4 hours. After this time the pressure was released and the resulting dark brown solution filtered through silica and Celite and purified by distillation under vacuum using a kugelrohr distillation apparatus or chromatography as recorded below, to afford the product alcohol. The enantiomeric excess of the product alcohol was found by chiral HPLC analysis.

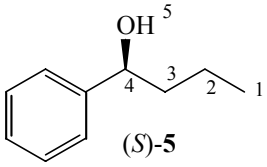
*Preparation of (S)-1-phenylpropanol (S)-**4***



(*S*)-**4**

Following procedure *5.4.5*, using *1-phenylpropanol **4*** (272 mg, 2 mmol, ρ = 0.994 gmL^{-1}, 274 μL, 1 eq). GC analysis using a 30 m x 0.32 mm Innowax column at an isothermal 185 ºC, showed >99% conversion to 1-phenylpropanone after oxidation, and 100% conversion back to 1-phenylpropanol **4** after hydrogenation (Retention time of 1-phenylpropanone = 3.7 min, and 1-phenylpropanol **4** = 4.8 min). Purification by kugelrohr distillation provided (*S*)-1-phenylpropanol (*S*)-**4**[7] as a colourless liquid (236 mg, 87%, 83% ee (*S*) using a Chiralcel OD-H® column, 3:97 ^{i}PrOH:Hexane solvent, 1 mL/min flow, retention times = 14.7 min (*R*), 16.7 min (*S*), $[\alpha]_D^{23}$ – 40º (*c* = 1 g/100 mL, $CHCl_3$), (lit.[7] $[\alpha]_D$ – 49.2º (*c* = 1.18 g/100 mL, $CHCl_3$) 98% ee (*S*)). The ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C) and ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C) data were consistent with the analytical data reported previously. IR (Liquid film): ν_{max} (cm^{-1}) = 3382 (br. s, OH), 3086 (m), 3063 (m), 3030 (m), 2965 (s), 2934 (m), 2876 (m), 1949 (w), 1881 (w), 1809 (w), 1755 (w), 1682 (w) 1604 (m), 1584 (w), 1493 (m), 1453 (s), 1411 (m), 1379 (m), 1359 (m), 1332 (m), 1262 (m) 1223 (m), 1201 (m), 1156 (w), 1096 (s), 1069 (m), 1046 (m), 1029 (m), 1014 (s), 975 (m), 917 (m), 898 (m), 835 (w), 809 (w), 745 (s), 700 (s).

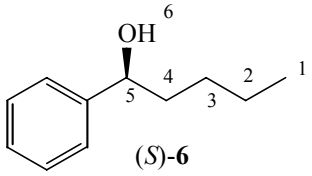
*Preparation of (S)-1-phenylbutanol (S)-**5***



(*S*)-**5**

Following procedure *5.4.5*, using *4-phenyl-1-butene-4-ol* (284 mg, 2 mmol, ρ = 0.992 gmL^{-1}, 286 μL, 1 eq). Purification by kugelrohr distillation provided (*S*)-1-phenylbutanol (*S*)-**5**[8] as a colourless liquid (232 mg, 82%, 87% ee (*S*) using a Chiralcel OB-H® column, 3:97 ^{i}PrOH:Hexane solvent, 1 mL/min flow, retention times = 14.5 min (*S*)-1-phenylbutanol (*S*)-**5**, 18.1 min (*R*)-1-phenylbutanol (*R*)-**5**). 4-phenyl-1-butene-4-ol **5** could not be resolved using the above chiral HPLC conditions, and so was resolved using a Chiralcel OD-H® column, 3:97 ^{i}PrOH:Hexane solvent, 1 mL/min flow, retention times = 14.1 min and 15.6 min (*R*) and (*S*)-4-phenyl-1-butene-4-ol **5**. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 0.86 (3H, t, *J* = 7.4 Hz, H_1), 1.00 – 1.47 (2H, m, H_2), 1.50 – 1.88 (3H, m, $H_{3,5}$), 4.60 (1H, dd, *J* = 7.5 and 5.8 Hz, H_4), 7.11 – 7.34 (5H, m, H_{Ar}). ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C): δ = 14.4 (C_1), 19.4 (C_2), 41.6 (C_3), 65.1 (C_4), 126.3 ($2CH_{Ar}$), 127.9 (CH_{Ar}), 128.8 ($2CH_{Ar}$), 145.3 (C_{Ar}). IR (Liquid film): ν_{max} (cm^{-1}) = 3445 (br. m, OH), 3087 (m), 3062 (m), 3028 (m), 2932 (s), 2865 (s), 1954 (w), 1812 (w), 1678 (m), 1597 (m), 1580 (m), 1494 (m), 1448 (m), 1379 (m), 1338 (m), 1261 (m), 1217 (m), 1135 (m), 1057 (m), 1030 (m), 1002 (m), 964 (m), 946 (m), 876 (w), 850 (w), 812 (w), 753 (m), 700 (m).

*Preparation of (S)-1-phenylpentanol (S)-**6***

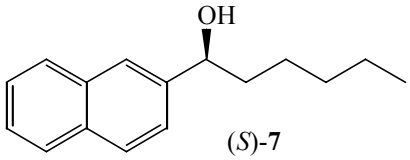


(*S*)-**6**

Following procedure *5.4.5*, using *1-phenylpentanol* **6** (328 mg, 2 mmol, ρ = 0.96 gmL^{-1}, 342 μL, 1 eq). GC analysis using a 30 m x 0.32 mm Innowax column at an isothermal 200 ºC, showed 100% conversion to 1-phenylpentanone after oxidation, and 100% conversion back to 1-phenylpentanol **6** after hydrogenation, (Retention time of 1-phenylpentanone = 4.1 min, and 1-phenylpentanol **6** = 5.4 min). Purification by kugelrohr distillation provided (*S*)-1-phenylpentanol (*S*)-**6**[9] as a colourless liquid (301 mg, 92%, 86% ee (*S*) using a Chiralcel OD-H® column, 3:97 ^{i}PrOH:Hexane solvent, 1 mL/min flow, retention times = 18.8 min (*R*), 21.0 min (*S*), $[\alpha]_D^{23}$ – 16º (*c* =

3 g/100 mL, MeOH), (lit.[10] $[\alpha]_D$ – 25.9º (*c* = 3.1 g/100 mL, MeOH) 99% ee (*S*)). ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 0.81 (3H, t, *J* = 7.0 Hz, H_1), 0.99 – 1.39 (4H, m, $H_{2,3}$), 1.55 – 1.87 (3H, m, $H_{4,6}$), 4.57 (1H, dd, *J* = 7.4 and 5.8 Hz, H_5), 7.13 – 7.33 (5H, m, H_{Ar}). ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C): δ = 14.4 (C_1), 23.0 (C_2), 28.4 (C_3), 39.2 (C_4), 75.1 (C_5), 126.3 ($2CH_{Ar}$), 127.9 (CH_{Ar}), 128.8 ($2CH_{Ar}$), 145.4 (C_{Ar}). IR (Liquid film): ν_{max} (cm^{-1}) = 3357 (br. s, OH), 3086 (m), 3063 (m), 3030 (m), 2964 (s), 2933 (s), 2876 (s), 1950 (w), 1883 (w), 1809 (w), 1604 (m), 1586 (w), 1493 (m), 1454 (s), 1412 (m), 1379 (m), 1360 (m), 1332 (m), 1271 (m), 1238 (m), 1202 (m), 1156 (w), 1096 (m), 1070 (m), 1046 (m), 1029 (m), 1014 (m), 975 (m), 917 (m), 898 (m), 835 (m), 763 (m), 699 (s).

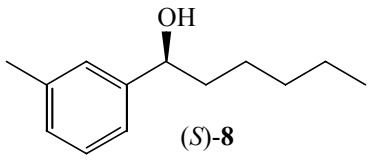
Preparation of (S)-1-(2-naphthyl)hexanol (S)-7



(*S*)-**7**

Following procedure *5.4.5*, using *1-(2-naphthyl)hexanol 7* (456 mg, 2 mmol, 1 eq). GC analysis using a 30 m x 0.32 mm Innowax column at an isothermal 250 ºC, showed 100% conversion to 1-(2-naphthyl)hexanone after oxidation, and 100% conversion back to 1-(2-naphthyl)hexanol **7** after hydrogenation, (Retention time of 1-(2-naphthyl)hexanone = 10.2 min, and 1-(2-naphthyl)hexanol **7** = 13.8 min). Purification by kugelrohr distillation provided (*S*)-1-(2-naphthyl)hexanol (*S*)-**7**[1] as a white solid (435 mg, 95%, 82% ee (*S*) using a Chiralcel OD-H® column, 2:98 ^{i}PrOH:Hexane solvent, 1 mL/min flow, retention times = 30.1 min (*S*), 35.5 min (*R*), $[\alpha]_D^{23}$ – 18º (*c* = 1 g/100 mL, MeOH). The ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C), ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C) and IR data were consistent with the analytical data reported previously.

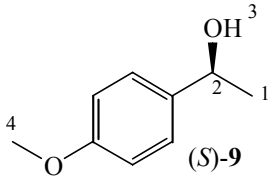
*Preparation of (S)-1-(3-methylphenyl)hexanol (S)-**8***



(*S*)-**8**

Following procedure *5.4.5*, using *1-(3-methylphenyl)hexanol* ***8*** (384 mg, 2 mmol, ρ = 0.94 gmL^{-1}, 409 μL, 1 eq). GC analysis using a 30 m x 0.32 mm Innowax column at an isothermal 190 ºC, showed 100% conversion to 1-(3-methylphenyl)hexanone after oxidation, and 100% conversion back to 1-(3-methylphenyl)hexanol **8** after hydrogenation, (Retention time of 1-(3-methylphenyl)hexanone = 6.0 min, and 1-(3-methylphenyl)hexanol **8** = 10.2 min). Purification by kugelrohr distillation provided (*S*)-1-(3-methylphenyl)hexanol (*S*)-**8**[2] as a colourless liquid (374 mg, 97%, 90% ee (*S*) using a Chiralcel OD-H® column, 2:98 ^{i}PrOH:Hexane solvent, 1 mL/min flow, retention times = 11.6 min (*R*), 13.3 min (*S*), $[\alpha]_D^{23}$ – 16º (*c* = 1.07 g/100 mL, MeOH). The ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C), ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C) and IR data were consistent with the analytical data reported previously.

*Preparation of (S)-1-(4-methoxyphenyl)ethanol (S)-**9***

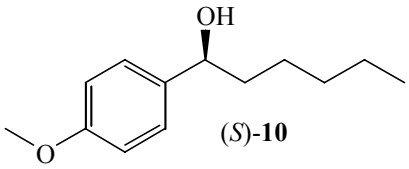


(*S*)-**9**

Following procedure *5.4.5*, using *1-(4-methoxyphenyl)ethanol* **9** (304 mg, 2 mmol, ρ = 1.09 gmL^{-1}, 279 μL, 1 eq). GC analysis using a 30 m x 0.32 mm Innowax column at an isothermal 190 ºC, showed 100% conversion to 1-(4-methoxyphenyl)ethanone after oxidation, and 100% conversion back to 1-(4-methoxyphenyl)ethanol **9** after hydrogenation, (Retention time of 1-(4-methoxyphenyl)ethanone = 4.8 min, and 1-(4-methoxyphenyl)ethanol **9** = 5.2 min). Purification by kugelrohr distillation provided (*S*)-1-(4-methoxyphenyl)ethanol (*S*)-**9**[11] as a colourless liquid (271 mg, 89%, 79% ee (*S*) using a Chiralcel OB-H® column, 10:90 ^{i}PrOH:Hexane solvent, 1 mL/min flow, retention times = 18.0 min (*S*), 24.4 min (*R*), $[\alpha]_D^{23}$ – 42.9º (*c* = 1.05 g/100 mL, MeOH), (lit.[12] $[\alpha]_D$ + 51.2º (*c* = 1.02 g/100 mL, MeOH) >99% ee (*R*)). ^{1}H NMR

(300 MHz, $CDCl_3$, 25 °C): δ = 1.33 (3H, d, *J* = 6.4 Hz, H_1), 1.84 (1H, br. s, H_3), 3.67 (3H, s, H_8), 4.71 (1H, q, *J* = 6.4 Hz, H_2), 6.71 – 6.78 (2H, m, H_{Ar}), 7.12 – 7.20 (2H, m, H_{Ar}). ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C): δ = 25.4 (C_1), 55.7 (C_4), 70.4 (C_2), 114.2 ($2CH_{Ar}$), 127.1 ($2CH_{Ar}$), 138.4 (C_{Ar}), 159.3 (C_{Ar}). IR (Liquid film): ν_{max} (cm^{-1}) = 3416 (br. m, OH), 2972 (m), 2931 (m), 2836 (m), 2246 (w), 2059 (w), 1889 (w), 1676 (w), 1612 (s), 1586 (m), 1513 (s), 1464 (m), 1445 (m), 1422 (m), 1369 (m), 1303 (m), 1289 (m), 1246 (s), 1206 (m), 1175 (s), 1090 (s), 1036 (s), 948 (w), 908 (m), 832 (s), 810 (m), 735 (m).

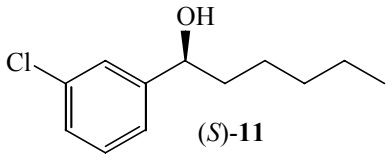
*Preparation of (S)-1-(4-methoxyphenyl)hexanol (S)-**10***



(*S*)-**10**

Following procedure *5.4.5*, using *1-(4-methoxyphenyl)hexanol **10*** (416 mg, 2 mmol, ρ = 1.00 gmL^{-1}, 416 μL, 1 eq). GC analysis using a 30 m x 0.32 mm Innowax column at an isothermal 250 ºC, showed 100% conversion to 1-(4-methoxyphenyl)hexanone after oxidation, and 100% conversion back to 1-(4-methoxyphenyl)hexanol **10** after hydrogenation, (Retention time of 1-(4-methoxyphenyl)hexanone = 4.9 min, and 1-(4-methoxyphenyl)hexanol **10** = 11.5 min). Purification by kugelrohr distillation provided (*S*)-1-(4-methoxyphenyl)hexanol (*S*)-**10** as a colourless liquid (389 mg, 94%, 88% ee (*S*) using a Chiralcel OB-H® column, 10:90 ^{i}PrOH:Hexane solvent, 1 mL/min flow, retention times = 14.9 min (*S*), 17.4 min (*R*), $[\alpha]_D^{23}$ – 17.8º (*c* = 1.13 g/100 mL, MeOH), (lit.[13] $[\alpha]_D$ + 20.8º (*c* = 1 g/100 mL, MeOH) 96% ee (*R*)). The ^1H NMR (300 MHz, $CDCl_3$, 25 °C), ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C) and IR data were consistent with the analytical data reported previously.

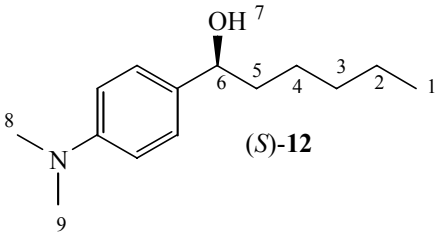
*Preparation of (S)-1-(3-chlorophenyl)hexanol (S)-**11***



(*S*)-**11**

Following procedure *5.4.5*, using *1-(3-chlorophenyl)hexanol* ***11*** (424 mg, 2 mmol, ρ = 1.07 gmL^{-1}, 396 μL, 1 eq). GC analysis using a 30 m x 0.32 mm Innowax column at an isothermal 230 ºC, showed 93% conversion to 1-(3-chlorophenyl)hexanone after oxidation, and 30% conversion back to 1-(3-chlorophenyl)hexanol **11** after hydrogenation, (Retention time of 1-(3-chlorophenyl)hexanone = 4.3 min, and 1-(3-chlorophenyl)hexanol **11** = 6.8 min). Purification by kugelrohr distillation followed by column chromatography (9:1 petroleum ether (b.p. 40–60 °C)/diethyl ether, R_f = 0.12) provided (*S*)-1-(3-chlorophenyl)hexanol (*S*)-**11** as a colourless liquid (110 mg, 26%, 57% ee (*S*) using a Chiralcel OD-H® column, 2:98 ^{i}PrOH:Hexane solvent, 1 mL/min flow, retention times = 14.4 min (*S*), 16.4 min (*R*), $[\alpha]_D^{23}$ – 25º (*c* = 1 g/100 mL, MeOH). The ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C), ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C) and IR data were consistent with the analytical data reported previously.

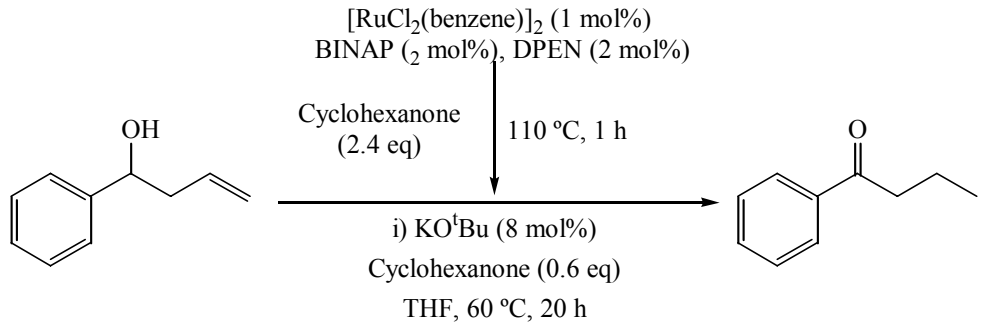
*Preparation of (S)-1-(4-dimethylaminophenyl)hexanol (S)-**12***



(*S*)-**12**

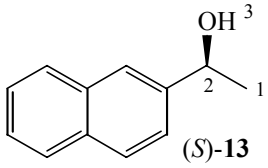
Following procedure *5.4.5*, using *1-(4-dimethylaminophenyl)hexanol* ***12*** (442 mg, 2 mmol, 1 eq). Purification by kugelrohr distillation provided (*S*)-1-(4-dimethylaminophenyl)hexanol (*S*)-**12**[4] as a white solid (423 mg, 96%, 92% ee (*S*) using a Chiralcel OB-H® column, 5:95 ^{i}PrOH:Hexane solvent, 1 mL/min flow, retention times = 18.0 min (*R*), 21.9 min (*S*), $[\alpha]_D^{23}$ – 16.6º (*c* = 1.03 g/100 mL, MeOH). ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 0.72 – 0.87 (3H, m, H_1), 1.08 – 1.40 (6H, m, $H_{2\text{-}4}$), 1.50 – 1.82 (3H, m, $H_{5,7}$), 2.87 (6H, s, $H_{8,9}$), 4.45 – 4.54 (1H, m, H_6), 6.65 (2H, d, *J* = 8.6 Hz, H_{Ar}), 7.15 (2H, d, *J* = 8.6 Hz, H_{Ar}). The ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C) and IR data were consistent with the analytical data reported previously.

Transfer hydrogenation of 4-phenyl-1-butene-4-ol



Following procedure *5.4.5*, using *4-phenyl-1-butene-4-ol* (284 mg, 2 mmol, ρ = 0.992 gmL^{-1}, 286 μL, 1 eq), the reaction was stopped after 20 hours oxidation, and was not transferred to the Parr® hydrogen bomb reactor. The reaction mixture was filtered through silica and Celite and concentrated *in vacuo*. Inspection of the ^1H NMR spectrum of the crude reaction mixture showed 100% conversion to phenylbutanone.

*Preparation of (S)-1-(2-naphthyl)ethanol (S)-**13** by stereoinversion of (R)-1-(2-naphthyl)ethanol (R)-**13***

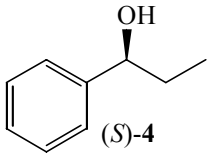


(*S*)-**13**

Following procedure *5.4.5*, using (*R*)-*1-(2-naphthyl)ethanol* (*R*)-**13** (344 mg, 2 mmol, 1 eq) and (*R*)-xylyl-BINAP (*R*)-**2** (29.4 mg, 0.04 mmol, $[\alpha]_D^{23}$ + 198º (*c* = 1 g/100 mL, $CHCl_3$), (lit.[14] $[\alpha]_D$ – 220.8 (*c* = 1 g/100 mL, $CHCl_3$) (*S*)), 0.02 eq). GC analysis using a 30 m x 0.32 mm Innowax column at an isothermal 250 ºC, showed 94% conversion to 1-(2-naphthyl)ethanone after oxidation, and 91% conversion back to 1-(2-naphthyl)ethanol **13** after hydrogenation, (Retention time of 1-(2-naphthyl)ethanone = 5.7 min, and 1-(2-naphthyl)ethanol **13** = 7.2 min). Purification by kugelrohr distillation followed by column chromatography (9:1 petroleum ether (b.p. 40–60 °C)/diethyl ether, R_f 1-(2-naphthyl)ethanol **13** = 0.05, R_f 1-(2-naphthyl)ethanone = 0.25, increasing the polarity of the solvent system to 8:2

petroleum ether (b.p. 40–60 °C)/diethyl ether, R_f 1-(2-naphthyl)ethanol **13** = 0.40 after isolation of the ketone) provided (*S*)-1-(2-naphthyl)ethanol (*S*)-**13**[11] as a white solid (279 mg, 81%, 80% ee (*S*) using a Chiralcel OB-H® column, 5:95 ^{i}PrOH:Hexane solvent, 1 mL/min flow, retention times = 19.6 min (*S*), 23.2 min (*R*), $[\alpha]_D^{23}$ – 31º (*c* = 1 g/100 mL, MeOH), (lit.[15] $[\alpha]_D$ – 31º (*c* = 3.5 g/100 mL, MeOH) 97% ee (*S*)). ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 1.49 (3H, d, *J* = 6.4 Hz, H_1), 1.90 (1H, br. s, H_3), 4.97 (1H, q, *J* = 6.4 Hz, H_2), 7.33 – 7.46 (3H, m, H_{Ar}), 7.68 – 7.80 (4H, m, H_{Ar}). ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C): δ = 25.6 (C_1), 70.9 (C_2), 124.2 (CH_{Ar}), 124.3 (CH_{Ar}), 126.2 (CH_{Ar}), 126.6 (CH_{Ar}), 128.1 (CH_{Ar}), 128.4 (CH_{Ar}), 128.7 (CH_{Ar}), 133.3 (C_{Ar}), 133.7 (C_{Ar}), 143.6 (C_{Ar}). IR (KBr Disk): ν_{max} (cm^{-1}) = 3355 (br. s, OH), 3053 (m), 2969 (m), 2921 (m), 2871 (m), 1701 (w), 1685 (w), 1654 (w), 1636 (w), 1600 (w), 1577 (w), 1559 (w), 1540 (w), 1507 (w), 1448 (w), 1406 (w), 1362 (m), 1273 (w), 1171 (w), 1125 (m), 1079 (m), 1024 (w), 950 (w), 911 (w), 895 (w), 861 (m), 826 (s), 743 (s), 484 (m).

Preparation of (S)-1-phenylpropanol (*S*)-***4*** *by stereoinversion of (R)-1-phenylpropanol* (*R*)-***4***

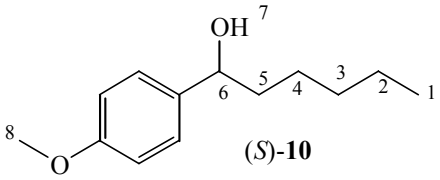


(*S*)-**4**

Following procedure *5.4.5*, using (*R*)-*1-phenylpropanol* (*R*)-***4*** (272 mg, 2 mmol, ρ = 0.994 gmL^{-1}, 274 μL, 1 eq), and (*R*)-xylyl-BINAP (*R*)-**4** (29.4 mg, 0.04 mmol, $[\alpha]_D^{23}$ + 198º (*c* = 1 g/100 mL, $CHCl_3$), (lit.[14] $[\alpha]_D$ – 220.8 (*c* = 1 g/100 mL, $CHCl_3$) (*S*)), 0.02 eq). GC analysis using a 30 m x 0.32 mm Innowax column at an isothermal 160 ºC, showed 95% conversion to 1-phenylpropanone after oxidation, and 72% conversion back to 1-phenylpropanol **4** after hydrogenation (Retention time of 1-phenylpropanone = 4.0 min, and 1-phenylpropanol **4** = 7.5 min). Purification by kugelrohr distillation followed by column chromatography (9:1 petroleum ether (b.p. 40–60 °C)/diethyl ether, R_f = 0.06) provided (*S*)-1-phenylpropanol (*S*)-**4**[7] as a colourless liquid (169 mg, 62%, 87% ee (*S*) using a Chiralcel OB-H® column, 3:97 ^{i}PrOH:Hexane solvent, 1 mL/min flow, retention times = 14.7 min (*S*), 18.5 min (*R*),

$[\alpha]_D^{23}$ – 42.6º (*c* = 1.01 g/100 mL, $CHCl_3$), (lit.[7] $[\alpha]_D$ – 49.2º (*c* = 1.18 g/100 mL, $CHCl_3$) 98% ee (*S*)). The ^1H NMR (300 MHz, $CDCl_3$, 25 °C), ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C) and IR data were consistent with the analytical data reported previously.

*Preparation of (S)-1-(4-methoxyphenyl)hexanol (S)-**10** using* (*R*)-xylyl-BINAP (*R*)-**2** *ligand*

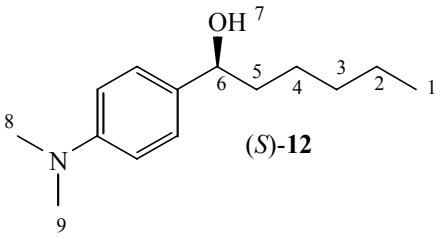


(*S*)-**10**

Following procedure *5.4.5*, using *1-(4-methoxyphenyl)hexanol **10*** (416 mg, 2 mmol, ρ = 1.00 gmL^{-1}, 416 μL, 1 eq), and (*R*)-xylyl-BINAP (*R*)-**2** (29.4 mg, 0.04 mmol, $[\alpha]_D^{23}$ + 198º (*c* = 1 g/100 mL, $CHCl_3$), (lit.[14] $[\alpha]_D$ – 220.8 (*c* = 1 g/100 mL, $CHCl_3$) (*S*)), 0.02 eq), oxidation length was extended to 24 hours, and hydrogenation length was extended to 16 hours. Purification by kugelrohr distillation provided (*S*)-1-(4-methoxyphenyl)hexanol (*S*)-**10**[3] as a colourless liquid (390 mg, 94%, 98.75% ee (*S*) using a Chiralcel OB-H® column, 5:95 ^{i}PrOH:Hexane solvent, 1 mL/min flow, retention times = 22.7 min (*S*), 27.0 min (*R*), $[\alpha]_D^{23}$ – 20º (*c* = 1.2 g/100 mL, MeOH), (lit.[13] $[\alpha]_D$ + 20.8º (*c* = 1 g/100 mL, MeOH) 96% ee (*R*)). ^1H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 0.71 – 0.84 (3H, m, H_1), 1.06 – 1.35 (6H, m, $H_{2\text{-}4}$), 1.50 – 1.81 (3H, m, $H_{5,7}$), 3.70 (3H, s, H_8), 4.45 – 4.54 (1H, m, H_6), 6.73 – 6.83 (2H, m, H_{Ar}), 7.11 – 7.21 (2H, m, H_{Ar}). ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C): δ = 14.5 (C_1), 23.0 (C_2), 26.0 (C_3), 32.2 (C_4), 39.4 (C_5), 55.7 (C_8), 74.7 (C_6), 114.1 ($2CH_{Ar}$), 127.6 ($2CH_{Ar}$), 137.6 (C_{Ar}), 159.3 (C_{Ar}). IR (KBr Disk): ν_{max} (cm^{-1}) = 3281 (br. s, OH), 3002 (s), 2930 (s), 2854 (s), 1895 (w), 1611 (s), 1587 (m), 1513 (s), 1466 (m), 1441 (m), 1377 (w), 1341 (w), 1303 (m), 1248 (s), 1180 (m), 1118 (w), 1100 (w), 1073 (w),

1038 (m), 1007 (m), 930 (m), 832 (s), 820 (m), 728 (w), 738 (w), 638 (w), 589 (w), 575 (w), 556 (m). MS (EI+): *m*/z (%) 208 (6) [$M^{\cdot+}$], 191 (2) [(M-($OH^{\cdot}$)$^+$], 190 (10) [(M-($H_2O^{\cdot}$)$^+$], 148 (3), 147 (25), 137 (100) [(M-(C_5H_{11})$^{\cdot}$)$^+$], 135 (6), 121 (8), 109 (59), 107 (10) [(M-($C_6H_{13}O$)$^{\cdot}$)$^+$], 94 (59), 91 (55) ($C_7H_7^{\cdot+}$), 89 (8), 79 (18), 78 (28), 77 (97) [$Ph^{\cdot+}$], 67 (5), 66 (23), 65 (29), 55 (15), 43 (16), 41 (23); HRMS (EI+) $C_{13}H_{20}O_2$ requires 208.1458 found 208.1455.

*Preparation of (S)-1-(4-dimethylaminophenyl)hexanol (S)-**12** using (R)-xylyl-BINAP (R)-**2** ligand*



(*S*)-**12**

Following procedure *5.4.5*, using *1-(4-dimethylaminophenyl)hexanol **12*** (442 mg, 2 mmol, 1 eq), and (*R*)-xylyl-BINAP (*R*)-**2** (29.4 mg, 0.04 mmol, $[\alpha]_D^{23}$ + 198º (*c* = 1 g/100 mL, $CHCl_3$), (lit.[14] $[\alpha]_D$ – 220.8 (*c* = 1 g/100 mL, $CHCl_3$) (*S*)), 0.02 eq), oxidation length was extended to 24 hours, and hydrogenation length was extended to 16 hours. Purification by kugelrohr distillation provided (*S*)-1-(4-dimethylaminophenyl)hexanol (*S*)-**12**[4] as a white solid (416 mg, 94%, 98% ee (*S*) using a Chiralcel OB-H® column, 5:95 ^{i}PrOH:Hexane solvent, 1 mL/min flow, retention times = 25.0 min (*R*), 31.4 min (*S*), $[\alpha]_D^{23}$ – 19º (*c* = 1 g/100 mL, MeOH). ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 0.68 – 0.82 (3H, m, H_1), 1.04 – 1.33 (6H, m, $H_{2\text{-}4}$), 1.48 – 1.79 (3H, m, $H_{5,7}$), 2.81 (6H, s, $H_{8,9}$), 4.37 – 4.47 (1H, m, H_6), 6.55 – 6.64 (2H, m, H_{Ar}), 7.03 – 7.15 (2H, m, H_{Ar}). ^{13}C NMR (75.4 MHz, $CDCl_3$, 25 °C): δ = 14.5 (C_1), 23.1 (C_2), 26.2 (C_3), 32.2 (C_4), 39.1 (C_5), 41.1 ($C_{8,9}$), 74.9 (C_6), 113.0 ($2CH_{Ar}$), 127.4 ($2CH_{Ar}$), 133.3 (C_{Ar}), 150.6 (C_{Ar}). IR (KBr Disk): ν_{max} (cm^{-1}) = 3269 (br. m, OH), 2934 (s), 2857 (s), 1616 (m), 1569 (w), 1524 (s), 1468 (m), 1446 (m), 1354 (m), 1316 (w), 1231 (w), 1190 (m), 1165 (m), 1130 (w), 1103 (w), 1068 (w) 1031 (m), 987 (w), 948 (w), 928 (w), 812 (s), 727 (w), 683 (w), 576 (w), 554 (w). MS

(EI+): *m*/z (%) 221 (10) [$M^{\cdot+}$], 203 (52) [$(M-(OH)^{\cdot})^{+}$], 160 (100) [$(M-(NMe_2)+(H_2O)^{\cdot})^{+}$], 151 (74) [$(M-(C_5H_{11})^{\cdot})^{+}$], 144 (25), 134 (20), 122 (13), 120 (27) [$(M-(C_6H_{13}O)^{\cdot})^{+}$], 115 (44), 107 (22) [$(M-(NMe_2)+(C_5H_{11})^{\cdot})^{+}$], 103 (17), 91 (46) ($C_7H_7^{\cdot+}$), 79 (28), 77 (67) [$Ph^{\cdot+}$], 65 (14), 51 (14), 43 (16), 41 (34); HRMS (CI, $[M+H]^{+}$) $C_{14}H_{24}NO$ requires 222.1852 found 222.1851.

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