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Direct Asymmetric Catalytic 1,2-Addition of RZnX to Aldehydes promoted by $AlMe_3$ and Reversal of Expected Stereochemistry

Jonathan Shannon, David Bernier, Daniel Rawson and Simon Woodward*

**School of Chemistry, The University of Nottingham, Nottingham NG7 2RD United Kingdom. Fax: +44-(0)115-9513564; Tel: +44-(0)115-9513541; E-mail: simon.woodward@nottingham.ac.uk*

Supporting Experimental Data

General

All experiments were carried out under an argon atmosphere using standard Schlenk techniques. Solvents were dried prior to use: THF distilled from sodium-benzophenone ketyl and toluene dried over 4Å molecular sieves. Aldehydes were freshly distilled prior to use. Proton and ^{13}C NMR spectra were recorded on Bruker AM400 or JEOL EX270 spectrometers, details of the NMR monitoring experiments are given below. For all other samples δ values were referenced to residual $CHCl_3$. All *J* values are in Hz. Infrared spectra were recorded on a Perkin Elmer 1600 Series FTIR machine. Mass spectra were recorded using Electronspray (ES) or electron impact (EI) techniques using a Micromass 70E machine. GC analyses were performed on a Varian 3380 gas chromatograph using Lipodex A column with yield determination by an appropriately calibrated internal standard. Optical rotations were measured using a JASCO DIP370 Digital polarimeter at ambient temperature. Chiral HPLC analysis was preformed on a Hewlett Packard 1100LC chromatograph using Daicel Chiracel AD (250mm), OB (250mm) or OD (250mm) stationary phase column. Thin layer chromatography (TLC) was performed on Merck silica gel 60 $F_{254+366}$ pre-coated plates (0.25 mm) silica. The plates were visualised by the use of a combination of ultraviolet light (254 and 366 nm) and/or aqueous potassium permanganate with heating. Liquid chromatography was by forced flow (flash chromatography) with the solvent systems indicated using silica gel 60 (220-240 mesh) supplied by Fluka. The compound triphenylaluminium[1] was prepared by a literature method, all other compounds were commercial products, specifically: $AlMe_3$ (2.0 M toluene solution; Aldrich), (1*R*,2*S*)-(+)-2-(dibutylamino)-1-phenyl-1-

propanol 97% **L1** (Aldrich), $ZnMe_2$ (1.2 M toluene solution; Acros), $ZnEt_2$ (1.1 M toluene solution; Aldrich), ArZnX (0.5 M THF solution, Aldrich). Known compounds were characterised by comparison with reported literature data.

The ligands **L4**, **L5** and **L6** were prepared following a general procedure described by Soai[2] (alkylation using 2 eq. of alkyl monobromide or 1 eq. of alkyl dibromide in the presence of 3 eq. K_2CO_3 in refluxing acetonitrile). The analytical data was consistent with the following literature references: (1*R*,2*S*)-1-phenyl-2-(pyrrolidin-1-yl)propan-1-ol (**L4**),[3] (1*R*,2*S*)-2-(*N,N*-Dibenzylamino)-1-phenylpropan-1-ol (**L5**)[4] and (1*R*,2*S*)-2-(dibutylamino)-1,2-diphenylethanol (**L6**).[5]

General procedure: Racemic phenylation (arylation) of aldehydes

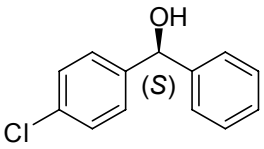
A flame dried Schlenk tube was charged with aldehyde (5.0 mmol) in dried THF (10 ml) at 0 ^oC was added PhMgBr (7.5 mmol, 5 ml, 3.0 M solution in Et_2O). The reaction mixture was stirred at 0 ^oC for 2 h after which time an aqueous solution of NH_4Cl (10 ml) was added. The product was extracted into dichloromethane (2 x 20 ml), the combined organic extracts were washed successively with water (30 ml) and brine (30 ml), dried over Na_2SO_4 and the solvent removed *in vacuo*. Purification by flash chromatography (silica; Et_2O:light petroleum b.p. 40-60 ^oC; 1:5 mixtures) gave the pure secondary alcohol. Non phenyl containing *sec*-alcohols were prepared using the procedure below but using racemic **L1**.

General procedure: Amino alcohol catalysed entantioselective 1,2-addition of ArZnBr to aldehyde

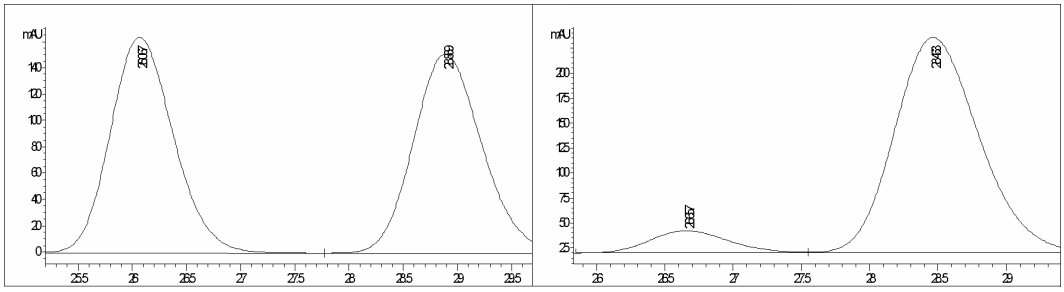
A flame-dried Schlenk tube was charged with ArZnBr (0.5 M in THF, 1 ml, 0.5 mmol) was added $AlMe_3$ (2.0 M solution in toluene, 7.5 mmol, 5.0 ml). After stirring at r.t. for 5 minutes the amino alcohol (10 mol %, 0.025 mmol) was added, followed by toluene (2.0 ml). The aldehyde (0.25mmol, 2 ml, 0.125M in toluene) was added by syringe pump over 1 hour. The reaction mixture was stirred at r.t. for 16 h after which time an aqueous solution of NH_4Cl (5 ml). The product was extracted into dichloromethane (2 x 20 ml), the combined organic extracts were washed successively with water (30 ml) and brine (30 ml), dried over Na_2SO_4 and the solvent

removed *in vacuo*. Purification by flash chromatography (silica; Et_2O:light petroleum b.p. 40-60 ^oC 1:5 mixtures for all but **3aj** and **3ak** where a 1:2 mixture was used) gave the pure secondary alcohol.

(*S*)-(4-Chlorophenyl)(phenyl)methanol (3ba)

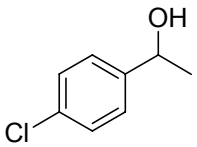


$[\alpha]_D$ = +19.1 (c = 0.83, $CHCl_3$, for 83 % *ee*; lit.[6] +22.0 for *S* antipode, c = 0.9, $CHCl_3$); **^{1}H NMR** (270 MHz, $CDCl_3$) δ_H = 7.37-7.23 (m, 9H, *Ar*), 5.81 (d, *J* = 3.4, 1H, C*H*), 2.20 (d, *J* = 3.4, 1H, O*H*); **^{13}C NMR** (100 MHz, $CDCl_3$) δ_C = 143.5 (q), 142.2 (q), 133.3 (q), 128.7 (2CH), 128.6 (2CH), 127.9 (3CH), 126.5 (2CH), 75.6 (CH); **HRMS** (EI) calcd. for $C_{13}H_{11}ClO^+$, [M^+] 218.0498, found 218.0500; **HPLC** Chiracel AD column, 95:5 *n*-hexane/2-propanol, 0.5 ml/min; t_R = 26.1 min for (*R*), t_S = 29.0 min for (*S*). HPLC trace for racemic and 83% *ee* sample below.



The minor byproduct (2-4% yield) was also fully characterized.

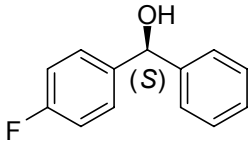
1-(4-Chlorophenyl)ethanol



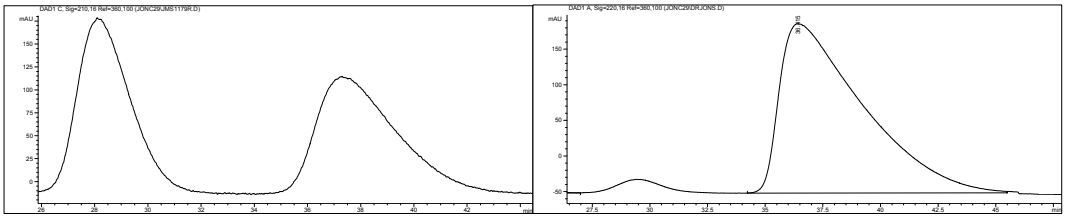
^{1}H NMR (270 MHz, $CDCl_3$) δ_H = 7.33-7.21 (m, 4H, *Ar*), 4.85 (q, *J* = 6.4, 1H, C*H*), 2.11 (br s, 1H, O*H*) 1.45 (d, *J* = 6.4, 3H); **^{13}C NMR** (67.8 MHz, $CDCl_3$) δ_C = 144.3 (q), 133.1 (q), 128.7 (2CH), 126.9 (2CH), 69.8 (CH), 25.3 (CH_3); **HRMS** (EI) calcd.

for $C_8H_9ClO^+$, [M^+] 156.0342, found 156.0340. No significant stereoselectivities were realized for this and the other methyl additions as determined by chiral GC.

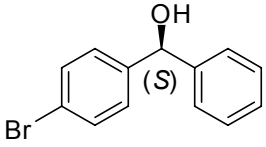
(*S*)-(4-Fluorophenyl)(phenyl)methanol (3ca)



$[\alpha]_D$ = +10.6 (c = 0.32, $CHCl_3$, for 90 % *ee*); **^{1}H NMR** (270 MHz, $CDCl_3$) δ_H = 7.40-7.21 (m, 7H, *Ar*), 7.04-6.97 (m, 2H, *Ar*), 5.81 (s, 1H, C*H*), 2.11 (br s, 1H, O*H*); **^{13}C NMR** (67.8 MHz, $CDCl_3$) δ_C = 162.3 (d, $^1J_{CF}$ = 252.5, q), 143.7 (q), 139.6 (d, $^4J_{CF}$ = 3.1, q), 128.7 (2CH), 128.3 (d, $^3J_{CF}$ = 8.3, 2CH), 127.8 (CH), 126.5 (2CH), 115.4 (d, $^2J_{CF}$ = 22.9, 2CH), 75.7 (CH); **HRMS** (EI) calcd. for $C_{13}H_{11}FO^+$, [M^+] 202.0794, found 202.0799; **HPLC** Chiracel OB column, 90:10 *n*-hexane/2-propanol, 1.0 ml/min; t_R = 28.8 min for (*R*), t_S = 36.7 min for (*S*). HPLC trace for racemic and 90% *ee* sample below.

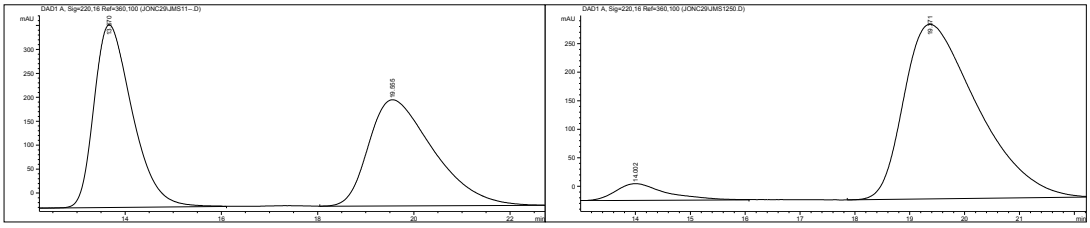


(*S*)-(4-Bromophenyl)(phenyl)methanol (3da)

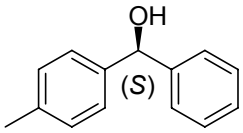


$[\alpha]_D$ = + 20.1 (c = 0.755, $CHCl_3$, for 88 % *ee*; lit.[6] +21.0 for *S* antipode, c = 0.9, C_6H_6); **^{1}H NMR** (270 MHz, $CDCl_3$) δ_H = 7.44 (d, *J* = 8.4, 2H, *Ar*), 7.36-7.27 (m, 5H, *Ar*), 7.22 (d, *J* = 8.5, 2H, *Ar*), 5.73 (s, 1H, C*H*), 2.53 (br s, 1H, O*H*); **^{13}C NMR** (68.7 MHz, $CDCl_3$) δ_C = 143.4 (q), 142.8 (q), 131.6 (2CH), 128.8 (2CH), 128.3 (2CH), 128.0 (CH), 126.6 (2CH), 121.5 (q), 75.6 (CH); **HRMS** (EI) calcd. for $C_{13}H_{11}BrO^+$, [M^+] 261.9993, found 261.9989; **HPLC** Chiracel OB column, 90:10 *n*-hexane/2-

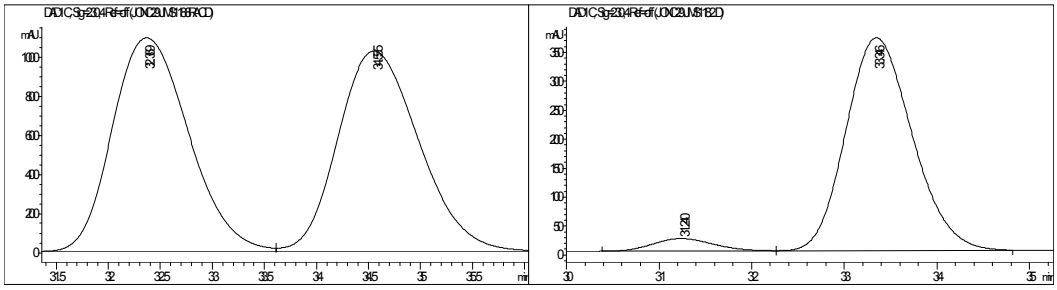
propanol, 1.0 ml/min; t_R = 14.0 min for (*R*), t_S = 19.4 min for (*S*). HPLC trace for racemic and 88% *ee* sample below.



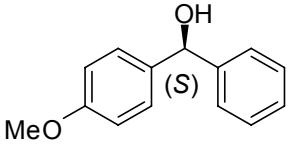
(*S*)-(4-Methylphenyl)(phenyl)methanol (3ea)



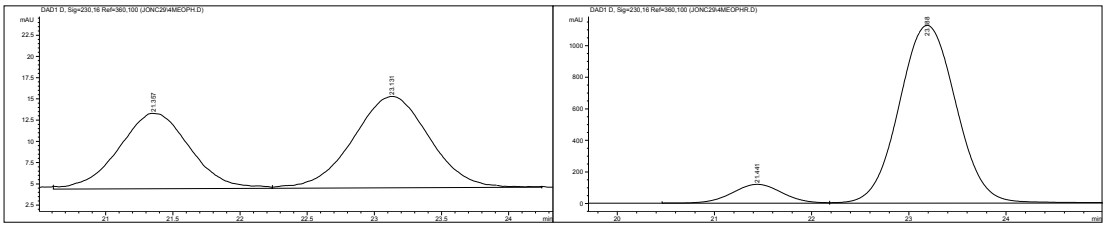
$[\alpha]_D$ = -12.0 (c = 0.3, $CHCl_3$, for 89 % *ee*; lit.[7] -9.7 for *S* antipode, c = 4.4, C_6H_6); **^{1}H NMR** (270 MHz, $CDCl_3$) δ_H = 7.40-7.30 (m, 5H, *Ar*), 7.26 (d, *J* = 8.9, 2H, *Ar*), 7.14 (d, *J* = 8.9, 2H, *Ar*), 5.79 (s, 1H, C*H*), 2.33 (s, 3H, *Me*), 2.19 (br s, 1H, O*H*); **^{13}C NMR** (67.8 MHz, $CDCl_3$) δ_C = 144.1 (q), 141.1 (q), 137.4 (q), 129.3 (2CH), 128.5 (2CH), 127.5 (CH), 126.6 (2CH), 126.5 (2CH), 76.2 (CH), 21.2 (CH_3); **HRMS** (EI) calcd. for $C_{14}H_{14}O^+$, [M^+] 198.1045, found 198.1050; **HPLC** Chiracel AD column, 95:5 *n*-hexane/2-propanol, 0.5 ml/min; t_R = 32.4 min for (*R*), t_S = 34.6 min for (*S*). HPLC trace for racemic and 89% *ee* sample below.



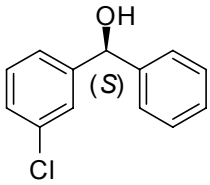
(S)-(4-Methoxyphenyl)(phenyl)methanol (3fa) and its enantiomer (*R*)-3af



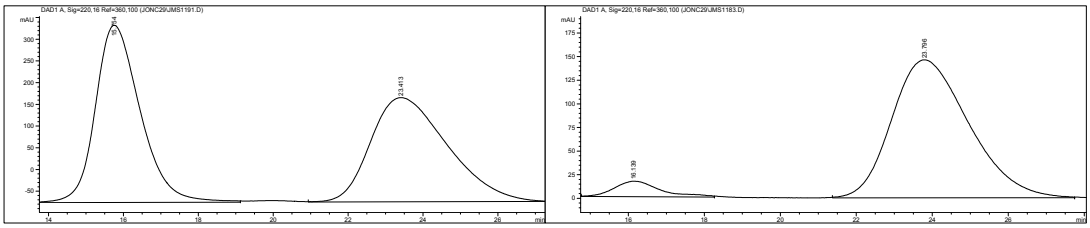
$[\alpha]_D$ = -13.2 (c = 0.55, $CHCl_3$, for 85 % *ee*; lit.[6] -18.8 for *S* antipode, c = 5.0, C_6H_6) for (*S*)-**3fa** and $[\alpha]_D$ = + 12.4 (c = 0.62, $CHCl_3$, for 84 % *ee*) for (*R*)-**3af**; **^{1}H NMR** (270 MHz, $CDCl_3$) δ_H = 7.40-7.25 (m, 6H, *Ar*), 6.85 (d, *J* = 8.9, 2H, *Ar*), 5.80 (s, 1H, C*H*), 3.78 (s, 3H, O*Me*) 2.13 (br s, 1H, O*H*); **^{13}C NMR** (67.8 MHz, $CDCl_3$) δ_C = 159.1 (q), 144.1 (q), 136.3 (q), 128.5 (2CH), 128.0 (2CH), 127.5 (CH), 126.5 (2CH), 114.0 (2CH), 75.9 (CH), 55.4 (CH_3); **HRMS** (EI) calcd. for $C_{14}H_{14}O_2^+$, [M^+] 214.0994, found 214.0992; **HPLC** chiracel AD column, 95:5 *n*-hexane/2-propanol, 0.5 ml/min; t_R = 21.4 min for (*R*), t_S = 23.4 min for (*S*). HPLC trace for racemic and 85% *ee* sample of (*S*)-**3fa** below.



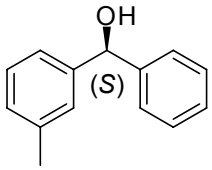
(*S*)-(3-Chlorophenyl)(phenyl)methanol (3ga)



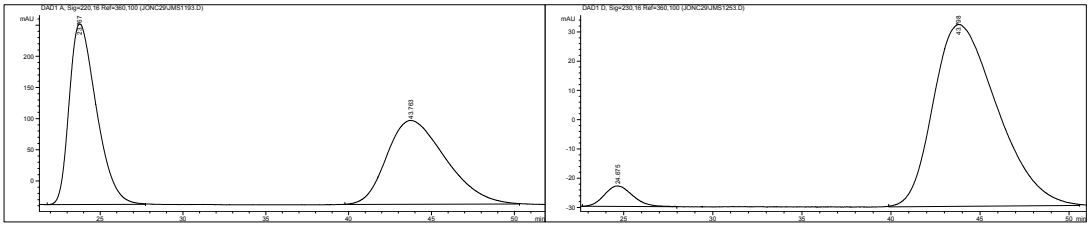
^{1}H NMR (270 MHz, $CDCl_3$) δ_H = 7.37-7.23 (m, 9H, *Ar*), 5.79 (s, 1H, C*H*), 2.29 (br s, 1H, O*H*); **^{13}C NMR** (67.8 MHz, $CDCl_3$) δ_C = 143.8 (q), 143.3 (q), 134.5 (q), 129.8 (CH), 128.8 (2CH), 128.0 (CH), 127.3 (CH), 126.7 (3CH), 124.7 (CH), 75.8 (CH); **HRMS** (EI) calcd. for $C_{13}H_{11}ClO^+$, [M^+] 218.0498, found 218.0492; **HPLC** Chiracel OB column, 90:10 *n*-hexane/2-propanol, 1.0 ml/min; t_R = 15.7 min for (*R*), t_S = 23.4 min for (*S*). HPLC trace for racemic and 91% *ee* sample below.



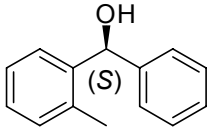
(*S*)-(3-Methylphenyl)(phenyl)methanol (3ha)



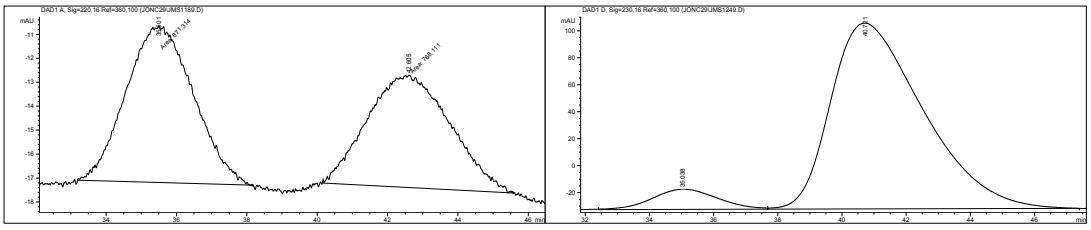
$[\alpha]_D$ = -1.1 (c = 0.45, $CHCl_3$, for 91 % *ee*); **^{1}H NMR** (270 MHz, $CDCl_3$) δ_H = 7.47-7.05 (m, 9H, *Ar*), 5.77 (s, 1H, C*H*), 2.55 (br s, 1H, O*H*), 2.37 (s, 3H, *Me*); **^{13}C NMR** (67.8 MHz, $CDCl_3$) δ_C = 144.0 (q), 143.9 (q), 138.3 (q), 128.6 (2CH), 128.5 (CH), 128.5 (CH), 127.6 (CH), 127.4 (CH), 126.7 (2CH), 123.8 (CH), 76.4 (CH), 21.6 (CH_3); **HRMS** (EI) calcd. for $C_{14}H_{14}O^+$, [M^+] 198.1045, found 198.1045; **HPLC** Chiracel OB column, 95:5 *n*-hexane/2-propanol, 1.0 ml/min; t_R = 24.7 min for (*R*), t_S = 43.7 min for (*S*). HPLC trace for racemic and 91% *ee* sample below.



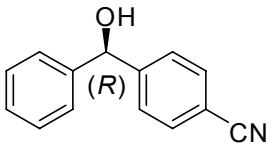
(*S*)-(2-Methylphenyl)(phenyl)methanol (3ia)



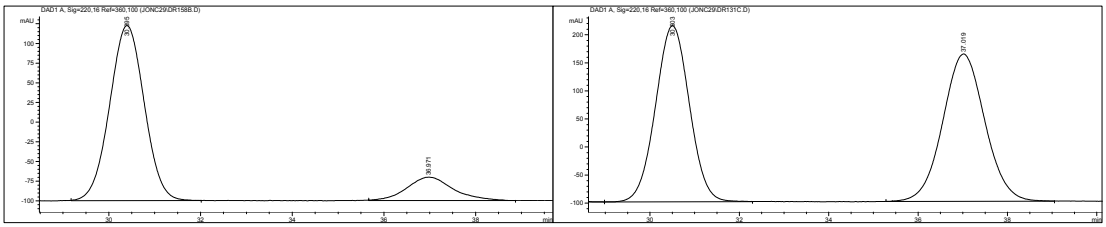
$[\alpha]_D$ = +3.0 (c = 0.65, $CHCl_3$, for 86 % *ee*; lit.[8] +5.9 for *S* antipode, c = 0.77, $CHCl_3$); **^{1}H NMR** (270 MHz, $CDCl_3$) δ_H = 7.54-7.48 (m, 1H, *Ar*), 7.36-7.12 (m, 8H, *Ar*), 5.98 (s, 1H, C*H*), , 2.29 (br s, 1H, O*H*), 2.25 (s, 3H, *Me*); **^{13}C NMR** (67.8 MHz, $CDCl_3$) δ_C = 143.0 (q), 141.6 (q), 135.5 (q), 130.6 (CH), 128.6 (2CH), 127.7 (CH), 127.6 (CH), 127.2 (2CH), 126.4 (CH), 126.2 (CH), 73.5 (CH), 19.5 (CH_3); **HRMS** (EI) calcd. for $C_{14}H_{14}O^+$, [M^+] 198.1045, found 198.1047; **HPLC** Chiracel OB column, 95:5 *n*-hexane/2-propanol, 0.5 ml/min; t_R = 35.1 min for (*R*), t_S = 40.7 min for (*S*). HPLC trace for racemic and 86% *ee* sample below.



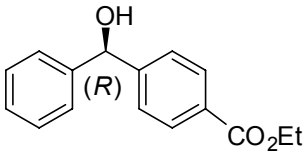
(*R*)-4-[Hydroxy(phenyl)methyl]benzonitrile (3aj)



$[\alpha]_D$ = -14.3 (c = 1.03, $CHCl_3$, for 79 % *ee*); **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.64 (d, 2H, *J* = 8.5, *Ar*), 7.54 (d, 2H, *J* = 8.0, *Ar*), 7.40–7.31 (m, 5H, *Ar*), 5.89 (s, 1H, C*H*), 2.43 (s, 1H, br, O*H*); **^{13}C NMR** (100 MHz, $CDCl_3$): δ_C = 148.8 (q), 140.3 (q), 134.3 (2CH), 128.9 (2CH), 128.3 (CH), 127.0 (2CH), 126.7 (2CH), 116.3 (q), 110.1 (*C*N), 75.7 (CH); **HRMS** (EI) calcd. for $C_{14}H_{11}NO^+$, [M^+] 209.0841, found 209.0849; **HPLC** Chiracel AD column, 95:5 *n*-hexane/2-propanol, 0.5 ml/min; t_R = 30.2 min for (*R*), t_S = 36.3 min for (*S*). HPLC trace for racemic and 79% *ee* sample below.

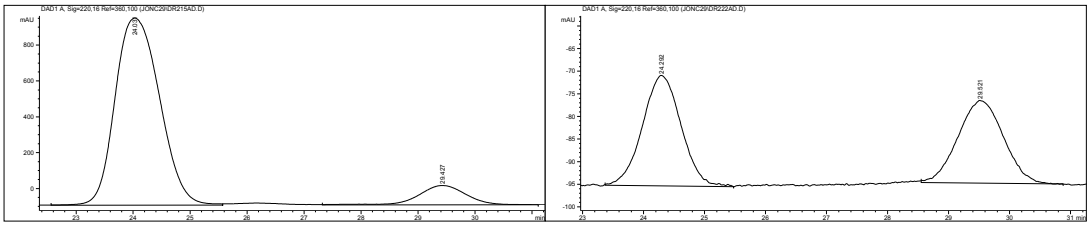


(*R*)-Ethyl 4-[hydroxy(phenyl)methyl]benzoate (3ak)

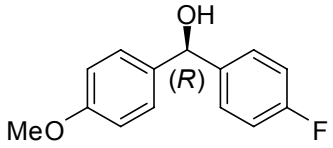


$[\alpha]_D$ = -38.3 (c = 0.42, $CHCl_3$, for 81 % *ee*); **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 8.01 (d, 2H, *J* = 8.2, *Ar*), 7.47 (d, 2H, *J* = 8.3, *Ar*), 7.36-7.28 (m, 4H, *Ar*), 5.87 (s, 1H, C*H*), 4.36 (q, 2H, *J* = 7.2, CH_2), 2.74 (s, 1H, O*H*), 1.39 (t, 3H, *J* = 7.2, CH_3); **^{13}C NMR**

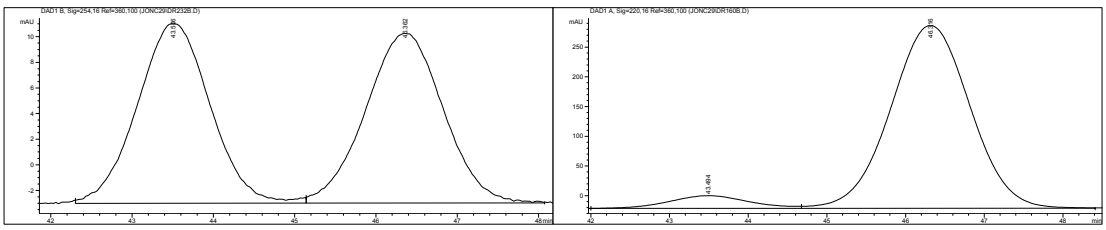
(100 MHz, $CDCl_3$): δ_C = 166.6 (q), 148.7 (q), 143.3 (q), 129.7 (2CH), 128.7 (2CH), 128.1 (CH), 126.9 (2CH), 126.0 (2CH), 75.7 (CH_2), 60.7 (CH_2), 14.3 (CH_3); **HRMS** (EI) calcd. for $C_{16}H_{16}O_3^+$, [M^+] 256.1099, found 256.1101; **HPLC** Chiracel AD column, 95:5 *n*-hexane/2-propanol, 1.0 ml/min; t_R = 24.0 min for (*R*), t_S = 29.4 min for (*S*). HPLC trace for racemic and 81% *ee* sample below.



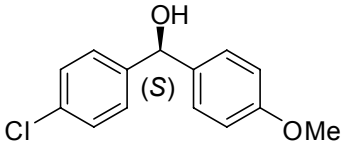
(*R*)-(4-Fluorophenyl)(4-methoxyphenyl)methanol (3fc)



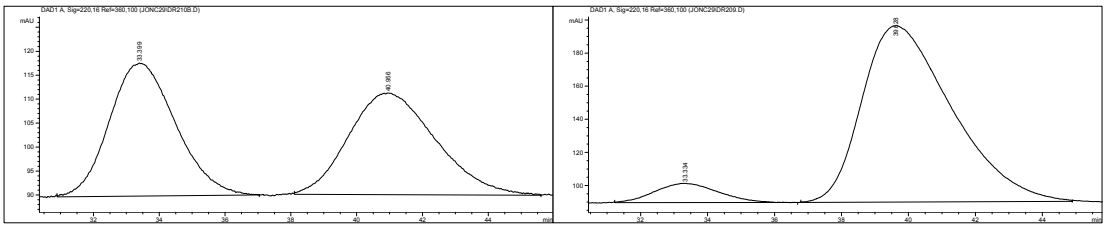
$[\alpha]_D$ = <+0.5 (c = 0.62, $CHCl_3$, for 87 % *ee*); **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.64 (d, 2H, *J* = 8.3, *Ar*), 7.51 (d, 2H, *J* = 8.6, *Ar*), 7.26 (m, 2H, *Ar*), 6.90 (d, 2H, *J* = 8.8, *Ar*), 5.84 (s, 1H, C*H*), 3.82 (s, 3H, O*Me*); **^{13}C NMR** (100 MHz, $CDCl_3$): δ_C = 159.6 (q), 149.1 (q), 137.9 (q), 134.1 (2CH), 128.7 (2CH), 126.9 (2CH), 118.9 (q), 114.2 (2CH), 75.2 (CH), 55.3 (CH_3); **HRMS** (EI) calcd. for $C_{14}H_{13}FO_2^+$, [M^+] 232.090, found 232.1012; **HPLC** Chiracel AD column, 95:5 *n*-hexane/2-propanol, 1.0 ml/min; t_R = 43.5 min for (*R*), t_S = 46.3 min for (*S*). HPLC trace for racemic and 87% *ee* sample below. The stereochemical assignment is made on the basis of model **C** in the paper (Scheme 2).



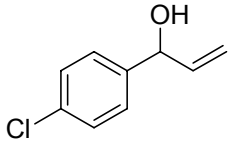
(*S*)-(4-Chlorophenyl)(4-methoxyphenyl)methanol (3bf)



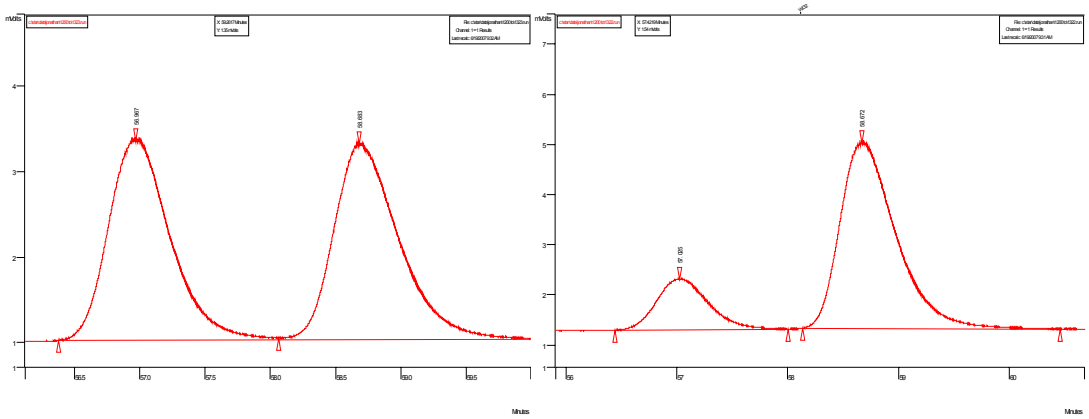
$[\alpha]_D$ = +4.5 (c = 0.21, $CHCl_3$, for 87 % *ee*); **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.34-7.33 (m, 4H, *Ar*), 7.26 (dd, 2H, *J* = 8.0, *J* = 1.4, *Ar*), 6.89 (dd, 2H, *J* = 8.6, *J* = 1.6, *Ar*), 5.80 (s, 1H, *CH*), 3.81 (s, 3H, *OMe*); **^{13}C NMR** (100 MHz, $CDCl_3$): δ_C = 159.3 (q), 142.4 (q), 135.8 (q), 133.1 (q), 128.6 (2CH), 127.9 (2CH), 126.8 (2CH), 114.0 (2CH), 75.2 (CH), 55.3 (CH_3); **HRMS** (EI) calcd. for $C_{14}H_{13}ClO_2^+$, [M^+] 248.0604, found 248.0605; **HPLC** Chiracel OB column, 90:10 *n*-hexane/2-propanol, 1.0 ml/min; t_R = 33.4 min for (*R*), t_S = 41.0 min for (*S*). HPLC trace for racemic and 87% *ee* sample below. The stereochemical assignment is made on the basis of model **C** in the paper (Scheme 2); the CIP rules result in the descriptor reversal compared to (*R*)-**3fc**.



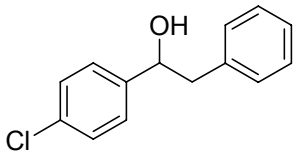
1-(4-Chlorophenyl)prop-2-en-1-ol



^{1}H NMR (270 MHz, $CDCl_3$) δ_H = 7.44-7.14 (m, 4H, *Ar*), 5.99 (ddd, *J* =17.1, 10.2, 6.1, 1H, =C*H*), 5.33 (app. dt, *J* =17.2, 1.3, 1H, $CH_{2\alpha}$), 5.20 (app. dt, *J* = 10.2, 1.3, 1H, $CH_{2\beta}$), 5.19-5.17 (m, 1H, C*H*OH), 1.71 (br s, 1H, O*H*); **^{13}C NMR** (125.7 MHz, $CDCl_3$) δ_C = 141.0 (q), 139.9 (CH), 133.5 (q), 128.7 (2CH), 127.7 (2CH), 115.0 (CH_2), 69.8 (CH); **HRMS** (EI) calcd. for $C_9H_9ClO^+$, [M^+] 168.0342, found 168.0341; **GC** Lipodex A, isothermal 100 ^oC; t_{E1} = 57.0 min for (E1), t_{E2} = 58.7 min for (E2). GC traces for racemic and 50% *ee* sample are given below.

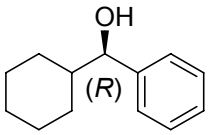


1-(4-Chlorophenyl)-2-phenylethanol

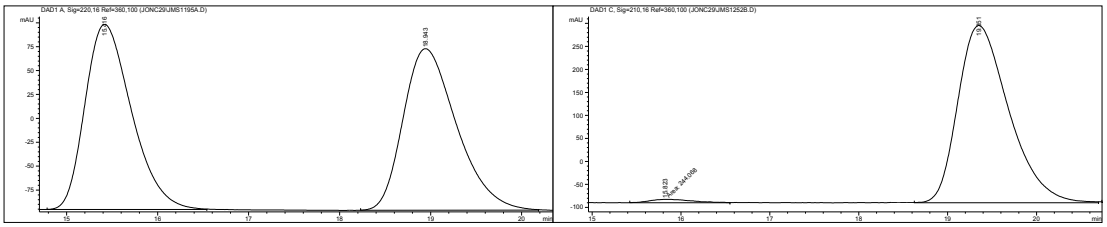


^{1}H NMR (270 MHz, $CDCl_3$) δ_H = 7.37-7.10 (m, 9H, *Ar*), 4.86 (dd, *J* = 8.0, 5.2, 1H, C*H*), 3.00 (dd, *J* = 13.5, 5.2, 1H, C$H_{2\alpha}$), 2.93 (dd, *J* = 13.5, 8.0, 1H, C$H_{2\beta}$), 1.89 (br s, 1H, O*H*); **^{13}C NMR** (67.8 MHz, $CDCl_3$) δ_C = 142.3 (q), 137.6 (q), 133.3 (q), 129.6 (2CH), 128.7 (2CH), 128.6 (2CH), 127.4 (2CH), 126.9 (2CH), 74.7 (CH), 46.2 (CH_2).

(*R*)-cyclohexyl(phenyl)methanol



$[\alpha]_D$ = +38.0 (c = 0.4, $CHCl_3$, for 96 % *ee*; lit.[9] +26.6 for *R* antipode, c = 1.76, C_6H_6); **^{1}H NMR** (270 MHz, $CDCl_3$) δ_H = 7.38-7.21 (m, 5H, *Ar*), 4.35 (d, *J*=7.2, 1H), 2.05-1.91 (m, 1 H), 1.84 - 1.54 (m, 5 H), 1.44 - 1.31 (m, 1 H), 1.27-0.83 (m, 5 H); **^{13}C NMR** (67.8 MHz, $CDCl_3$) δ_C = 143.7 (q), 128.3 (2CH), 127.5 (CH), 126.7 (2CH), 79.5 (CH), 45.0 (CH), 38.0 (CH_2), 29.4 (CH_2), 28.9 (CH_2), 26.5 (CH_2), 26.2 (CH_2), 26.1 (CH_2); **HRMS** (EI) calcd. for $C_{13}H_{18}O^+$, [M^+] 190.1358, found 190.1361. **HPLC** Chiracel OD column, 95:5 *n*-hexane/2-propanol, 0.5 ml/min; t_R = 15.4 min for (*R*), t_S = 18.9 min for (*S*). HPLC trace for racemic and 96% *ee* sample below.



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