Direct Asymmetric Catalytic 1,2-Addition of RZnX to Aldehydes promoted by AlMe₃ and Reversal of Expected Stereochemistry

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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 ¹³C 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 CHCl₃. 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 F₂₅₄-366 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 triphenylaluminium¹ was prepared by a literature method, all other compounds were commercial products, specifically: AlMe₃ (2.0 M toluene solution; Aldrich), (1R,2S)-(−)-2-(dibutylamino)-1-phenyl-1-
propanol 97% L1 (Aldrich), ZnMe₂ (1.2 M toluene solution; Acros), ZnEt₂ (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 Soai² (alkylation using 2 eq. of alkyl monobromide or 1 eq. of alkyl dibromide in the presence of 3 eq. K₂CO₃ in refluxing acetonitrile). The analytical data was consistent with the following literature references: (1R,2S)-1-phenyl-2-(pyrrolidin-1-yl)propan-1-ol (L4),³ (1R,2S)-2-(N,N-Dibenzylamino)-1-phenylpropan-1-ol (L5)⁴ and (1R,2S)-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 °C was added PhMgBr (7.5 mmol, 5 ml, 3.0 M solution in Et₂O). The reaction mixture was stirred at 0 °C for 2 h after which time an aqueous solution of NH₄Cl (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 Na₂SO₄ and the solvent removed *in vacuo*. Purification by flash chromatography (silica; Et₂O:light petroleum b.p. 40-60 °C; 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 AlMe₃ (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 NH₄Cl (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 Na₂SO₄ and the solvent
removed in vacuo. Purification by flash chromatography (silica; Et₂O:light petroleum b.p. 40-60 °C 1:5 mixtures for all but 3aj and 3ak where a 1:2 mixture was used) gave the pure secondary alcohol.

\((S)-(4\text{-Chlorophenyl})(\text{phenyl})\text{methanol (3ba)}\)

\[
\begin{align*}
\text{Cl} & \quad \text{(S)} \\
\text{OH} & \quad \text{(phenyl)}
\end{align*}
\]

\([\alpha]_D = +19.1 \ (c = 0.83, \text{CHCl}_3, \text{for } 83 \% ee); \text{lit.}^6 +22.0 \text{ for } S \text{ antipode, } c = 0.9, \text{CHCl}_3); \text{ } ^1\text{H NMR (270 MHz, CDCl}_3) \ \delta_H = 7.37-7.23 \ (m, 9H, Ar), 5.81 \ (d, J = 3.4, 1H, CH), 2.20 \ (d, J = 3.4, 1H, OH); \text{ } ^{13}\text{C NMR (100 MHz, CDCl}_3) \ \delta_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); \text{ HRMS (EL) calcd. for C}_{13}\text{H}_{11}\text{ClO}^+, [M^+] 218.0498, \text{found } 218.0500; \text{ HPLC} \text{ Chiracel AD column, 95:5 n-hexane/2-propanol, 0.5 ml/min; } t_R = 26.1 \text{ min for } (R), t_S = 29.0 \text{ min for } (S). \text{ HPLC trace for racemic and 83% ee sample below.}
\]

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

\(1\text{-}(4\text{-Chlorophenyl})\text{ethanol}\)

\[
\begin{align*}
\text{Cl} & \quad \text{(phenyl)} \\
\text{OH} & \quad \text{Ar}
\end{align*}
\]

\(^1\text{H NMR (270 MHz, CDCl}_3) \ \delta_H = 7.33-7.21 \ (m, 4H, Ar), 4.85 \ (q, J = 6.4, 1H, CH), 2.11 \ (br s, 1H, OH) 1.45 \ (d, J = 6.4, 3H); \text{ } ^{13}\text{C NMR (67.8 MHz, CDCl}_3) \ \delta_C = 144.3 \ (q), 133.1 \ (q), 128.7 \ (2CH), 126.9 \ (2CH), 69.8 \ (CH), 25.3 \ (CH}_3); \text{ HRMS (EL) calcd.}
for C₈H₉ClO⁺, [M⁺] 156.0342, found 156.0340. No significant stereoselectivities were realized for this and the other methyl additions as determined by chiral GC.

(5)-(4-Fluorophenyl)(phenyl)methanol (3ca)

[α]D = +10.6 (c = 0.32, CHCl₃, for 90 % ee); ¹H NMR (270 MHz, CDCl₃) δH = 7.40-7.21 (m, 7H, Ar), 7.04-6.97 (m, 2H, Ar), 5.81 (s, 1H, CH), 2.11 (br s, 1H, OH); ¹³C NMR (67.8 MHz, CDCl₃) δC = 162.3 (d, ¹JC = 252.5, q), 143.7 (q), 139.6 (d, ⁴JC = 3.1, q), 128.7 (2CH), 128.3 (d, ³JC = 8.3, 2CH), 127.8 (CH), 126.5 (2CH), 115.4 (d, ²JC = 22.9, 2CH), 75.7 (CH); HRMS (EI) calcd. for C₁₃H₁₁FO⁺, [M⁺] 202.0794, found 202.0799; HPLC Chiracel OB column, 90:10 n-hexane/2-propanol, 1.0 ml/min; tR = 28.8 min for (R), tS = 36.7 min for (S). HPLC trace for racemic and 90% ee sample below.

(5)-(4-Bromophenyl)(phenyl)methanol (3da)

[α]D = + 20.1 (c = 0.755, CHCl₃, for 88 % ee; lit. +21.0 for S antipode, c = 0.9, C₆H₆); ¹H NMR (270 MHz, CDCl₃) δH = 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, CH), 2.53 (br s, 1H, OH); ¹³C NMR (68.7 MHz, CDCl₃) δC = 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 C₁₃H₁₁BrO⁺, [M⁺] 261.9993, found 261.9989; HPLC Chiracel OB column, 90:10 n-hexane/2-
propanol, 1.0 ml/min; \( t_R = 14.0 \text{ min} \) for \((R)\), \( t_S = 19.4 \text{ min} \) for \((S)\). HPLC trace for racemic and 88\% ee sample below.

\[(S)-(4-Methylphenyl)(phenyl)methanol (3ea)\]

\[
\begin{align*}
\alpha_D &= -12.0 \text{ (c = 0.3, CHCl}_3, \text{ for 89 \% ee); lit.}^7 \text{ -9.7 for } S \text{ antipode, c = 4.4, C}_6\text{H}_6); \\
{^1}\text{H NMR} \text{ (270 MHz, CDCl}_3) \delta_H &= 7.40-7.30 \text{ (m, 5H, Ar)}, 7.26 \text{ (d, } J = 8.9, 2H, Ar), 7.14 \text{ (d, } J = 8.9, 2H, Ar), 5.79 \text{ (s, 1H, CH)}, 2.33 \text{ (s, 3H, Me)}, 2.19 \text{ (br s, 1H, OH); } \\
{^{13}}\text{C NMR} \text{ (67.8 MHz, CDCl}_3) \delta_C &= 144.1 \text{ (q), 141.1} \text{ (q), 137.4} \text{ (q), 129.3} \text{ (2CH), 128.5} \text{ (2CH), 127.5} \text{ (CH), 126.6} \text{ (2CH), 126.5} \text{ (2CH), 76.2} \text{ (CH), 21.2} \text{ (CH); HRMS (EI) calcd. for } C_{14}H_{14}O^+ \text{, [M*] 198.1045, found 198.1050; HPLC Chiracel AD column,} \\
95:5 \text{ n-hexane/2-propanol, 0.5 ml/min; } \text{HPLC trace for racemic and 89\% ee sample below.}\]

\[(S)-(4-Methoxyphenyl)(phenyl)methanol (3fa) and its enantiomer (R)-3af\]

\[
\begin{align*}
\text{(S)} &- \text{OH} \\
\text{(R)} &- \text{MeO}
\end{align*}
\]
$\alpha_D = -13.2$ (c = 0.55, CHCl$_3$, for 85 % ee; lit.$^6$ -18.8 for S antipode, c = 5.0, C$_6$H$_6$) 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$_3$) $\delta_H = 7.40$-$7.25$ (m, 6H, Ar), 6.85 (d, $J = 8.9$, 2H, Ar), 5.80 (s, 1H, CH), 3.78 (s, 3H, OMe) 2.13 (br s, 1H, OH); $^{13}$C NMR (67.8 MHz, CDCl$_3$) $\delta_C =$ 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)

1H NMR (270 MHz, CDCl$_3$) $\delta_H = 7.37$-$7.23$ (m, 9H, Ar), 5.79 (s, 1H, CH), 2.29 (br s, 1H, OH); $^{13}$C NMR (67.8 MHz, CDCl$_3$) $\delta_C =$ 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)
[α]_D = -1.1 (c = 0.45, CHCl₃, for 91% ee); \( ^1\)H NMR (270 MHz, CDCl₃) \( \delta_H = 7.47-7.05 \) (m, 9H, \( Ar \)), 5.77 (s, 1H, \( CH \)), 2.55 (br s, 1H, \( OH \)), 2.37 (s, 3H, \( Me \)); \( ^{13}\)C NMR (67.8 MHz, CDCl₃) \( \delta_C = 144.0 \) (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 (CH₃); HRMS (EI) calcd. for C₁₄H₁₀O⁺, [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)

[α]_D = +3.0 (c = 0.65, CHCl₃, for 86% ee; lit. \( +5.9 \) for \( S \) antipode, c = 0.77, CHCl₃); \( ^1\)H NMR (270 MHz, CDCl₃) \( \delta_H = 7.54-7.48 \) (m, 1H, \( Ar \)), 7.36-7.12 (m, 8H, \( Ar \)), 5.98 (s, 1H, \( CH \)), 2.29 (br s, 1H, \( OH \)), 2.25 (s, 3H, \( Me \)); \( ^{13}\)C NMR (67.8 MHz, CDCl₃) \( \delta_C = 143.0 \) (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 (CH₃); HRMS (EI) calcd. for C₁₄H₁₀O⁺, [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)

\[
\text{OH} \quad (R) \quad \text{CN}
\]

[\alpha]_D = -14.3 (c = 1.03, CHCl_3, for 79 % ee); \[^1\text{H} \text{NMR}\] (400 MHz, CDCl\_3): \(\delta_H = 7.64\) (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, CH), 2.43 (s, 1H, br, OH); \(^{13}\text{C} \text{NMR}\) (100 MHz, CDCl\_3): \(\delta_C = 148.8\) (q), 140.3 (q), 134.3 (2CH), 128.9 (2CH), 128.3 (CH), 127.0 (2CH), 126.7 (2CH), 116.3 (q), 110.1 (CN), 75.7 (CH); \(^{1}\text{HRMS}\) (EI) calcd. for C\(_{14}\)H\(_{11}\)NO\(^+\), [M\(^+\)] 209.0841, found 209.0849; \(^{1}\text{HPLC}\) 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)

\[
\text{OH} \quad (R) \quad \text{CO}_2\text{Et}
\]

[\alpha]_D = -38.3 (c = 0.42, CHCl\_3, for 81 % ee); \[^1\text{H} \text{NMR}\] (400 MHz, CDCl\_3): \(\delta_H = 8.01\) (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, CH), 4.36 (q, 2H, \(J = 7.2\), CH\_2), 2.74 (s, 1H, OH), 1.39 (t, 3H, \(J = 7.2\), CH\_3); \(^{13}\text{C} \text{NMR}\)
(100 MHz, CDCl₃): 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 (CH₂), 60.7 (CH₂), 14.3 (CH₃);

HRMS (EI) calcd. for C₁₆H₁₆O₃⁺, [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, CHCl₃, for 87% ee); \(^1\)H NMR (400 MHz, CDCl₃): 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, CH), 3.82 (s, 3H, OMe); \(^13\)C NMR (100 MHz, CDCl₃): 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 (CH₃); HRMS (EI) calcd. for C₁₄H₁₃FO₂⁺, [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)

\[
\text{OH} \quad \text{Cl} \quad (S) \quad \text{OMe}
\]

\[\alpha]_D = +4.5 \ (c = 0.21, \text{CHCl}_3, \text{for } 87 \% \text{ ee}) ; \ \ ^1\text{H NMR} \ (400 \text{ MHz, CDCl}_3) : \ \delta_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}\text{C NMR} \ (100 \text{ MHz, CDCl}_3) : \ \delta_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) ; \ \text{HRMS} \ (\text{EI}) \ \text{calcd. for } C_{14}H_{13}ClO_2^+, [M^+] 248.0604, \ \text{found} 248.0605 ; \ \text{HPLC} \ \text{Chiracel OB column, 90:10 } n\text{-hexane/2-propanol}, 1.0 \ \text{ml/min; } t_R = 33.4 \ \text{min for } (R), t_S = 41.0 \ \text{min for } (S). \ \text{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

\[
\text{OH} \quad \text{Cl}
\]

\[\text{H NMR} \ (270 \text{ MHz, CDCl}_3) \ \delta_H = 7.44-7.14 \ (m, 4H, Ar), 5.99 \ (ddd, J =17.1, 10.2, 6.1, 1H, =CH), 5.33 \ (\text{app. dt, } J=17.2, 1.3, 1H, CH_2\alpha), 5.20 \ (\text{app. dt, } J=10.2, 1.3, 1H, CH_2\beta), 5.19-5.17 \ (m, 1H, CHOH), 1.71 \ (\text{br s, } 1H, OH) ; \ \ ^{13}\text{C NMR} \ (125.7 \text{ MHz, CDCl}_3) \ \delta_C = 141.0 \ (q), 139.9 \ (CH), 133.5 \ (q), 128.7 \ (2CH), 127.7 \ (2CH), 115.0 \ (CH_2), 69.8 \ (CH) ; \ \text{HRMS} \ (\text{EI}) \ \text{calcd. for } C_9H_9ClO_+^-, [M^+] 168.0342, \ \text{found} 168.0341 ; \ \text{GC Lipodex A, isothermal } 100 ^\circ\text{C; } t_E1 = 57.0 \ \text{min for (E1), } t_{E2} = 58.7 \ \text{min for (E2). GC traces for racemic and 50% ee sample are given below.}
1-(4-Chlorophenyl)-2-phenylethanol

\[
\text{Cl} \quad \text{OH} \quad \text{Cl} \quad \text{Ar}
\]

\[\begin{align*}
\text{H NMR} & (270 \text{ MHz, CDCl}_3) \; \delta_H = 7.37-7.10 \; (m, \; 9H, \; Ar), \; 4.86 \; (dd, \; J = 8.0, \; 5.2, \; 1H, \; CH), \; 3.00 \; (dd, \; J = 13.5, \; 5.2, \; 1H, \; CH_{2z}), \; 2.93 \; (dd, \; J = 13.5, \; 8.0, \; 1H, \; CH_{2\beta}), \; 1.89 \; (br \; s, \; 1H, \; OH); \; ^{13}\text{C NMR} & (67.8 \text{ MHz, CDCl}_3) \; \delta_C = 142.3 \; (q), \; 137.6 \; (q), \; 133.3 \; (q), \; 129.6 \; (2CH), \; 128.7 \; (2CH), \; 128.6 \; (2CH), \; 127.4 \; (2CH), \; 126.9 \; (2CH), \; 74.7 \; (CH), \; 46.2 \; (CH_2). \n\end{align*}\]

(R)-cyclohexyl(phenyl)methanol

\[
\begin{align*}
\text{OH} \\
(R) \\
\text{Ph}
\end{align*}
\]

\[\begin{align*}
\left[\alpha\right]_D & = +38.0 \; (c = 0.4, \; \text{CHCl}_3, \; \text{for 96\% ee}; \; \text{lit.}^9 +26.6 \; \text{for } R \text{ antipode, } c = 1.76, \; \text{C}_8\text{H}_6); \; \\
\text{H NMR} & (270 \text{ MHz, CDCl}_3) \; \delta_H = 7.38-7.21 \; (m, \; 5H, \; Ar), \; 4.35 \; (d, \; J=7.2, \; 1H), \; 2.05-1.91 \; (m, \; 1 H), \; 1.84 - 1.54 \; (m, \; 5 H), \; 1.44 - 1.31 \; (m, \; 1 H), \; 1.27-0.83 \; (m, \; 5 H); \; ^{13}\text{C NMR} & (67.8 \text{ MHz, CDCl}_3) \; \delta_C = 143.7 \; (q), \; 128.3 \; (2CH), \; 127.5 \; (CH), \; 126.7 \; (2CH), \; 79.5 \; (CH), \; 45.0 \; (CH), \; 38.0 \; (CH_2), \; 29.4 \; (CH_2), \; 28.9 \; (CH_2), \; 26.5 \; (CH_2), \; 26.2 \; (CH_2), \; 26.1 \; (CH_2); \; \text{HRMS (EI) calcd. for C}_{13}\text{H}_{18}\text{O}^+, \left[M^+\right] 190.1358, \; \text{found } 190.1361. \; \text{HPLC} \; \text{Chiracel OD column, 95:5 n-hexane/2-propanol, 0.5 ml/min; } t_R = 15.4 \; \text{min for (R), } t_S = 18.9 \; \text{min for (S). HPLC trace for racemic and 96\% ee sample below.}
\end{align*}\]
References