Supporting Information

Highly Enantioselective Additions of Diethylzinc to Aldehydes using 2-Triflamidomethyl-2'-hydroxy-1,1'-binaphthyl

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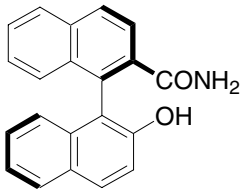
General

IR spectra were recorded on a Bomem MB-104 spectrophotometer. Optical rotations were measured with a Rudolph Research Autopol III polarimeter. ^{1}H NMR spectra were recorded on a Varian Germini 300 (300 MHz) with TMS as an internal reference. ^{13}C NMR spectra were recorded on a Varian Germini 300 (75 MHz) with TMS or $CDCl_3$ as an internal reference. Elemental analyses were obtained from Sogang Organic Chemistry Research Center, Seoul. HRMS (FAB+) sprectra were recorded on JEOL JMS-AX505WA mass spectrometer. Chiral HPLC analysis was performed on a Jasco LC-1500 Series HPLC system with a UV detector. GC analysis was performed on a Hewlett Packard 5890 series II plus GAS Chromatograph with a FID detector.

All reactions were carried out in oven-dried glassware under an argon atmosphere. Toluene (CaH_2), THF (Na, benzophenone) and CH_2Cl_2 (CaH_2) were dried by distillation before use. Diethylzinc was purchased from Aldrich.

(*R*)-2-Carbamoyl-2'-hydroxy-1,1'-binaphthyl (8) : A mixture of ester **7**[1] (500 mg, 1.09 mmol) and NaCN (5.3 mg, 0.11 mmol) in 4 ml of 7 N NH_3 in methanol was heated to 70-80 ^{o}C in a sealed pressure tube for 48h. The solvent was removed in vacuo, and the residue was dissolved in 50ml of CH_2Cl_2 and washed with 20ml H_2O. The aqueous layer was extracted with CH_2Cl_2 (30 ml × 3). The combined organic layers were washed with brine (30 ml × 2), dried ($MgSO_4$), and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel with CH_2Cl_2/MeOH (30:1) to give the amide **8** (318 mg, 94 % yield) as a white solid.



$[\alpha]_D{}^{25} = -22.7$ (c = 1.62, THF).

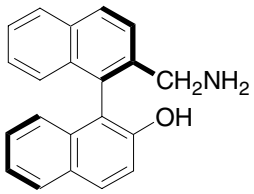
IR (KBr) ν 3444(br), 1652 cm^{-1}.

^1H NMR (300 MHz, CD_3OD) δ 6.84 (d, *J* = 8 Hz, 1H), 7.11-7.26 (m, 4H), 7.29 (d, *J* = 9 Hz, 1H), 7.49 (ddd, *J* = 8, 7, 2 Hz, 1H), 7.79 (d, *J* = 8 Hz, 1H), 7.82 (d, *J* = 7 Hz, 1H), 7.89 (d, *J* = 9 Hz, 1H), 7.95 (d, *J* = 8 Hz, 1H), 8.03 (d, *J* = 9 Hz, 1H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 117.25, 118.26, 122.64, 124.34, 125.14, 126.27, 126.39, 126.58, 126.61, 127.67, 127.89, 127.94, 128.05, 129.41, 131.73, 132.29, 133.63, 134.04, 135.61, 152.52, 170.34.

HRMS (FAB+): Calcd for $C_{21}H_{15}NO_2$: 314.1181 $[M+H]^+$. Found: 314.1178. Anal. Calcd for $C_{21}H_{15}NO_2$: C, 80.49; H, 4.82; N, 4.47. Found: C, 80.45; H, 4.70; N, 4.30.

(*R*)-2-Aminomethyl-2'-hydroxy-1,1'-binaphthyl (9) : To a solution of amide **8** (300 mg, 0.96 mmol) in THF (10 ml) was added $LiAlH_4$ (145 mg, 3.83 mmol) in small portions at room temperature and the mixture was heated at reflux for 24h. The mixture was cooled to 0 °C and quenched with H_2O (20 ml). The resulting mixture was filtered through Celite pad which was washed with CH_2Cl_2. The filtrate was extracted with CH_2Cl_2 (30 ml × 3), and the combined organic layers were washed with brine (30 ml × 2), dried ($MgSO_4$), and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel with CH_2Cl_2/MeOH (10:1) to give the amine **9** (138 mg, 48% yield) as a white solid.



$[\alpha]_D{}^{25} = -31.94$ (c = 1.33, EtOH).

IR (KBr) ν 3439 (br) cm^{-1}.

^1H NMR (300 MHz, CD_3OD) δ 3.63 (d, *J* = 14 Hz, 1H), 3.71 (d, *J* = 14 Hz, 1H), 6.75 (d, *J* = 8 Hz, 1H), 7.08-7.25 (m, 4H), 7.30 (d, *J* = 9 Hz, 1H), 7.42 (ddd, *J* = 8, 7, 2 Hz, 1H), 7.70 (d, *J* = 8 Hz, 1H), 7.83 (d, *J* = 8 Hz, 1H), 7.88 (d, *J* = 9 Hz, 1H), 7.93 (d, *J* = 8 Hz, 1H), 8.01 (d, *J* = 8 Hz, 1H).

^{13}C NMR (75 MHz, CD_3OD) δ 45.17, 117.39, 118.76, 120.38, 123.73, 125.29, 126.80, 127.11, 127.39, 127.55, 127.67, 129.03, 129.11, 129.46, 129.84, 130.77, 134.66, 134.85, 135.80, 137.64, 155.40.

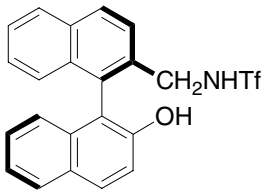
HRMS (FAB+): Calcd for $C_{21}H_{17}NO$: 300.1388 $[M+H]^+$. Found: 300.1388.

Synthesis of sulfonamide (10a-c): general procedure

To a solution of amine **9** (130mg, 0.43 mmol) in THF (8 ml) at −78 °C was added n-BuLi (0.54 ml of 1.6M solution in hexane, 0.87 mmol). After 30min of stirring at −78 °C, the mixture was added with the corresponding sulfonic anhydride or sulfonyl chloride (0.87 mmol), stirred at −78 °C for 1h, and then allowed to warm to room temperature. The mixture was quenched with 1N HCl (10 ml), and extracted with ethyl acetate (30 ml × 2). The combined organic layers were washed with brine (30 ml), dried ($MgSO_4$), and concentrated. The crude bissulfonated residue was used directly in the next step.

The crude product was dissolved in THF (10 ml) and a solution of $LiOH·H_2O$ (219 mg, 5.21 mmol) in H_2O (3 ml) was added. The mixture was stirred at room temperature for 12h. The reaction mixture was quenched with 1N HCl (10 ml) and the mixture was extracted with ethyl acetate (30 ml × 3). The combined organic layers were washed with washed with brine (30 ml × 2), dried ($MgSO_4$), and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel eluted with ethyl acetate/hexane (1:5) to give the sulfonamide **10** as a white solid.

(*R*)-2-Triflouromethanesulfonylamidomethyl-2'-hydroxy-1,1'-binaphthyl (10a)



yield: 89%

$[\alpha]_D^{25}$ = −5.4 (c = 1.0, $CHCl_3$).

IR ($CHCl_3$) ν 3511 (br) cm^{-1}.

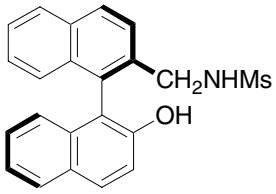
^{1}H NMR (300 MHz, $CDCl_3$) δ 4.10 (d, *J* = 14 Hz, 1H), 4.24 (d, *J* = 14 Hz, 1H), 5.27 (br, 2H), 6.86 (d, *J* = 8 Hz, 1H), 7.19-7.37 (m, 5H), 7.51 (td, *J* = 8, 1 Hz, 1H), 7.71 (d, *J* = 8 Hz, 1H), 7.89 (d, *J* = 8 Hz, 1H), 7.94 (d, *J* = 9 Hz, 1H), 8.03 (d, *J* = 8 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 46.90, 116.34, 119.46 (q, J_{C-F} = 320 Hz), 117.63, 124.05, 124.14, 125.99, 126.94, 127.04, 127.37, 127.45, 128.32, 128.44, 129.27, 130.02, 130.76,

131.49, 132.84, 133.34, 133.48, 133.74, 150.54.

HRMS (FAB+): Calcd for $C_{22}H_{16}F_3NO_3S$: 431.0803 $[M]^+$. Found: 431.0807. Anal. Calcd for $C_{22}H_{16}F_3NO_3S$: C, 61.25; H, 3.74; N, 3.25; O, 11.13, S, 7.43. Found: C, 61.28, H, 3.91; N, 2.97; S, 7.47.

(*R*)-2-Methanesulfonylamidomethyl-2'-hydroxy-1,1'-binaphthyl (10b)



yield: 74%

$[\alpha]_D^{25} = -12.0$ (c = 1.0, $CHCl_3$).

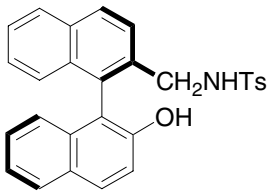
IR ($CHCl_3$) ν 3329 (br) cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$) δ 2.49 (s, 3H), 3.39 (dd, *J* = 13, 3 Hz, 1H), 4.06 (dd, *J* = 13, 6 Hz, 1H), 4.71 (br, 1H), 5.58 (br, 1H), 6.88 (d, *J* = 8 Hz, 1H), 7.20-7.36 (m, 5H), 7.49 (t, *J* = 8 Hz, 1H), 7.73 (d, *J* = 9 Hz, 1H), 7.87 (d, *J* = 8 Hz, 1H), 7.91 (d, *J* = 9 Hz, 1H), 7.93 (d, *J* = 8 Hz, 1H), 7.99 (d, *J* = 8 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 39.40, 45.82, 116.54, 117.80, 123.75, 124.20, 125.92, 126.50, 126.94, 127.05, 127.12, 128.13, 128.19, 128.95, 129.35, 130.28, 131.33, 132.90, 133.42, 133.64, 134.17, 150.95.

HRMS (FAB+): Calcd for $C_{22}H_{19}NO_3S$: 377.1086 $[M]^+$. Found: 377.1081. Anal. Calcd for $C_{22}H_{19}NO_3S$: C, 70.00; H, 5.07; N, 3.71; S, 8.50. Found: C, 70.03; H, 5.27; N, 3.51; S, 8.16.

(*R*)-2-(4-Toluenesulfonyl)amidomethyl-2'-hydroxy-1,1'-binaphthyl (10c)



yield: 81%

$[\alpha]_D^{25} = -4.2$ (c = 1.0, $CHCl_3$).

IR ($CHCl_3$) ν 3382 (br) cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$) δ 2.32 (s, 3H), 3.72 (dd, *J* = 13, 3 Hz, 1H), 3.82 (dd, *J* = 13, 6 Hz, 1H), 5.05 (br, 1H), 5.66 (br, 1H), 6.78 (d, *J* = 8 Hz, 1H), 7.02 (d, *J* = 8 Hz, 2H),

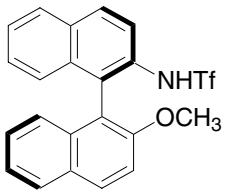
7.10-7.30 (m, 5H), 7.37 (d, J = 8 Hz, 2H), 7.44 (t, J = 7 Hz, 1H), 7.62 (d, J = 8 Hz, 1H), 7.76 (d, J = 9 Hz, 1H), 7.79 (d, J = 7 Hz, 1H), 7.86 (d, J = 8 Hz, 1H), 7.87 (d, J = 8 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 21.44, 45.81, 116.43, 117.75, 123.62, 124.14, 125.94, 126.48, 126.82, 126.91, 127.02, 127.34, 128.13, 128.18, 128.93, 129.40, 129.51, 130.03, 130.95, 132.84, 133.45, 133.60, 134.40, 135.72, 143.18, 150.90.

HRMS (FAB+): Calcd for $C_{28}H_{23}NO_3S$: 453.1399 $[M]^+$. Found: 453.1400. Anal. Calcd for $C_{28}H_{23}NO_3S$: C, 74.15; H, 5.11; N, 3.09; S, 7.07. Found: C, 74.34; H, 5.33; N, 2.87; S, 7.06.

(*R*)-2-Triflouromethanesulfonylamino-2'-methoxy-1,1'-binaphthyl (12)

To a cooled (−78 °C) solution of **11**[2] (150 mg, 0.50 mmol) in CH_2Cl_2 (5 ml) was added triflic anhydride (0.17 ml, 1.00 mmol). The reaction mixture was stirred for 1h at –78 °C. The reaction was quenched with 1N HCl (10 ml) and the mixture was extracted with ethyl acetate (30 ml × 2). The combined organic layers were washed with $NaHCO_3$ (20 ml) and brine (20 ml), dried ($MgSO_4$), and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel eluted with ethyl acetate/hexane (1:8) to give the triflamide **12** (198 mg, 92% yield) as a white solid.



$[\alpha]_D^{25}$ = –92.8 (c = 1.0, $CHCl_3$).

IR ($CHCl_3$) ν 3285(NH) cm^{-1}.

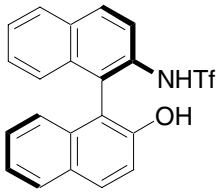
^{1}H NMR (300 MHz, $CDCl_3$) δ 3.82 (s, 3H), 6.63 (br, 1H), 6.95 (d, J = 8 Hz, 1H), 7.08 (d, J = 8 Hz, 1H), 7.24-7.50 (m, 5H), 7.89-7.94 (m, 3H), 8.01 (d, J = 9 Hz, 1H), 8.09 (d, J = 9 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 56.20, 112.95, 115.33, 119.34 (q, $J_{C\text{-}F}$ = 320 Hz), 120.20, 124.20, 124.44, 125.46, 125.95, 126.27, 126.94, 127.44, 128.10, 128.27, 129.13, 129.51, 131.04, 131.59, 131.88, 132.92, 133.38, 154.71.

HRMS (FAB+): Calcd for $C_{22}H_{16}F_3NO_3S$: 431.0803 $[M]^+$. Found: 431.0801. Anal. Calcd for $C_{22}H_{16}F_3NO_3S$: C, 61.25; H, 3.74; N, 3.25; S, 7.43. Found: C, 61.27; H, 3.66; N, 3.15; S, 7.36.

(*R*)-2-Triflouromethanesulfonylamino-2'-hydroxy-1,1'-binaphthyl (13)

A solution of **12** (198 mg, 0.46 mmol) in CH_2Cl_2 (5 ml) at 0 °C was treated with BBr_3 (1.38 ml of 1.0 M solution in CH_2Cl_2, 1.38 mmol). After the addition, the solution was stirred for 2h, and then allowed to warm to room temperature. To the reaction mixture was added H_2O (5 ml) and with 1N HCl (5 ml). The mixture was extracted with ethyl acetate (30 ml × 2) and combined organic layers were washed with H_2O (20 ml) and brine (20 ml), dried ($MgSO_4$), and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel eluted with ethyl acetate/hexane (1:5) to give the product **13** (132 mg, 69% yield) as a white solid.



$[\alpha]_D^{25}$ = +43.4 (c = 1.12, $CHCl_3$).

IR ($CHCl_3$) ν 3511 (OH), 3323(NH) cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$) δ 4.97 (br, 1H), 6.53 (br, 1H), 6.96 (d, *J* = 9 Hz, 1H), 7.23 (d, *J* = 8 Hz, 1H), 7.29-7.43 (m, 4H), 7.53 (ddd, *J* = 8, 7, 2 Hz, 1H), 7.92 (d, *J* = 9 Hz, 1H), 7.97 (d, *J* = 8 Hz, 1H), 7.98 (d, *J* = 9 Hz, 1H), 8.01 (d, *J* = 9 Hz, 1H), 8.09 (d, *J* = 9 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 111.93, 117.86, 119.32 (q, J_{C-F} = 320 Hz), 120.03, 122.54, 123.61, 124.21, 125.75, 126.57, 127.69, 127.81, 128.30, 128.55, 129.28, 130.64, 131.71, 132.01, 132.11, 132.72, 132.86, 151.70.

HRMS (FAB+): Calcd for $C_{21}H_{14}F_3NO_3S$: 417.0646 $[M]^+$. Found: 417.0637.

General Procedure for the Addition of Et2Zn to aldehyde

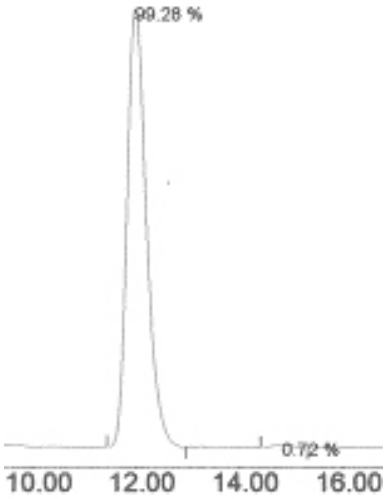
To a solution of chiral ligand **10a** (6.5 mg, 0.015 mmol) in dichloromethane (2 ml) was added $Ti(O^iPr)_4$ (17.7 ml, 0.60 mmol) at room temperature. After stirring of the mixture for 15min, aldehyde (0.50 mmol) was added into the reaction solution and reaction mixture was cooled to –25 °C. Diethylzinc (1.0 M solution in hexane, 0.9 ml, 0.90 mmol) was added into the solution and the reaction mixture was stirred for 2h at –25 °C. The reaction was quenched by the addition of 1N HCl solution (10ml) and the mixture was extracted with ethyl acetate (30 ml x 2). The combined organic extracts were washed with brine (30 ml x 2), dried ($MgSO_4$), and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel with eluted with ethyl acetate/hexanes (1:8) to give the alcohol product.

Conditions for the determination of enantiomeric excess are listed below. All alcohols

were compared with racemic samples which prepared via Grignard reaction.

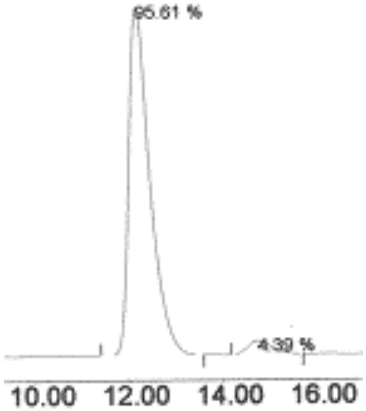
1-Phenyl-1-propanol

$[\alpha]_D^{25}$ = +42.4 (c = 2.50, $CHCl_3$) {lit.[3] $[\alpha]_D^{22}$ = -47.6 (c = 6.11, $CHCl_3$) for 98% ee (*S*) }; 99% ee by HPLC analysis (Chiralcel OD column, hexane:2-propanol = 98:2, 1 ml/min, 254 nm UV detector), t_R = 11.99 min for (*R*) and t_R = 14.83 min for (*S*).



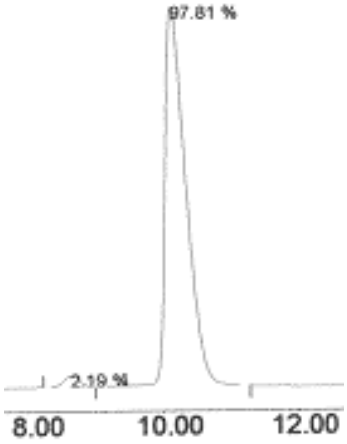
1-(4'-Methoxyphenyl)-1-propanol

$[\alpha]_D^{25}$ = +30.41 (c = 3.57, C_6H_6) {lit.[3] $[\alpha]_D^{22}$ = –32.1 (c = 1.25, C_6H_6) for 93% ee (*S*) }; 91% ee by HPLC analysis (Chiralcel OD column, hexane:2-propanol = 97:3, 1 ml/min, 254 nm UV detector), t_R = 12.20 min for (*R*) and t_R = 14.74 min for (*S*).



1-(2'-Chlorophenyl)-1-propanol

$[\alpha]_D^{25}$ = +52.31 (c = 3.46, $CHCl_3$) {lit.[5] $[\alpha]_D$ = +37.1 (c = 4, $CHCl_3$) for 79% ee (*S*) }; 96% ee by HPLC analysis (Chiralcel OB-H column, hexane:2-propanol = 99:1, 1 ml/min, 254 nm UV detector), t_R = 10.16 min for (*R*) and t_R = 8.63 min for (*S*).

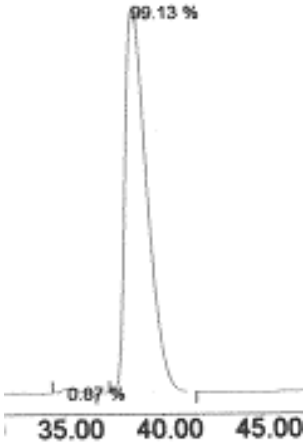


1-(3'-Chlorophenyl)-1-propanol

$[\alpha]_D^{25}$ = +29.27 (c = 3.17, C_6H_6) {lit.[4] $[\alpha]_D^{20}$ = +26.6 (c = 2.36, C_6H_6) for 97% ee (*R*) }; 99% ee by HPLC analysis (Chiralcel OD column, hexane:2-propanol = 99:1, 0.5 ml/min, 254 nm UV detector), t_R = 41.58 min for (*R*) and t_R = 36.90 min for (*S*).

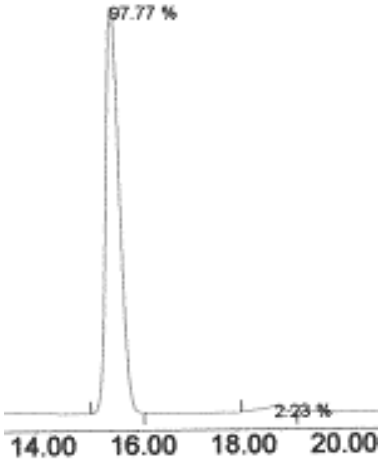
1-(4'-Chlorophenyl)-1-propanol

$[\alpha]_D^{25}$ = +27.32 (c = 2.91, C_6H_6) {lit.[3] $[\alpha]_D^{22}$ = –23.5 (c = 0.82, C_6H_6) for 93% ee (*S*) }; 98% ee by HPLC analysis (Chiralcel OD column, hexane:2-propanol = 99:1, 0.5 ml/min, 254 nm UV detector), t_R = 38.57 min for (*R*) and t_R = 35.21 min for (*S*).



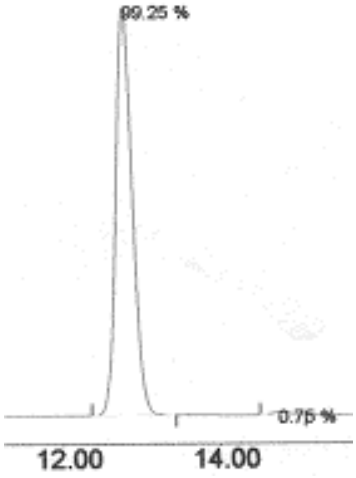
1-(2'-Tolyl)-1-propanol

$[\alpha]_D^{25}$ = +64.10 (c = 2.12 , C_6H_6) {lit.[7] $[\alpha]_D^{RT}$ = +58.5 (c = 2.0 , C_6H_6) for 99% ee (*R*) } 96% ee by HPLC analysis (Chiralpak AD-H column, hexane:2-propanol = 99:1, 1ml/min, 254 nm UV detector), t_R = 15.57 min for (*R*) and t_R = 18.75 min for (*S*).



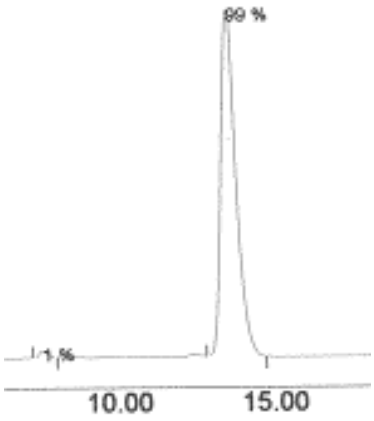
1-(4'-Tolyl)-1-propanol

$[\alpha]_D^{25}$ = +41.62 (c = 2.53 , C_6H_6) {lit.[4] $[\alpha]_D^{20}$ = +39.3 (c = 3.65, C_6H_6) for 95.7% ee (*R*) } 99% ee by HPLC analysis (Chiralpak AD-H column, hexane:2-propanol = 98:2, 1ml/min, 254 nm UV detector), t_R = 12.79 min for (*R*) and t_R = 14.74 min for (*S*).



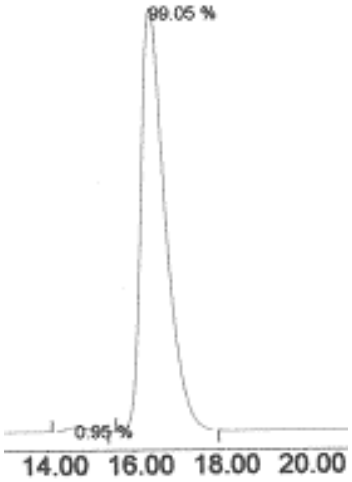
1-(1'-Napthyl)-1-propanol

$[\alpha]_D^{25}$ = +51.10 (c = 4.10, $CHCl_3$) {lit.[4] $[\alpha]_D^{20}$ = +52.6 (c = 2.55, $CHCl_3$) for 93.5% ee (*R*) }; 98% ee by HPLC analysis (Chiralcel OD column, hexane:2-propanol = 9:1, 1 ml/min, 254 nm UV detector), t_R = 13.69 min for (*R*) and t_R = 7.74 min for (*S*).



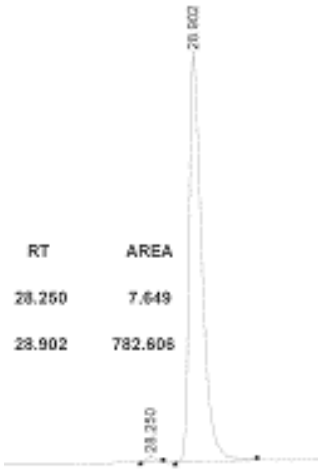
1-(2'-Napthyl)-1-propanol

$[\alpha]_D^{25}$ = +28.83 (c = 4.11, C_6H_6) {lit.[4] $[\alpha]_D^{20}$ = +27.5 (c = 3.80, C_6H_6) for 96.1% ee (*R*) }; 98% ee by HPLC analysis (Chiralcel OD column, hexane:2-propanol = 95:5, 1 ml/min, 254 nm UV detector), t_R = 16.45 min for (*R*) and t_R = 14.63 min for (*S*).



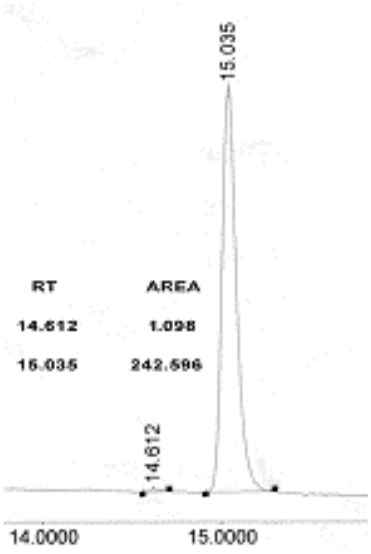
3-Nonanol

$[\alpha]_D^{25}$ = -7.50 (c = 0.56, $CHCl_3$) {lit.[3] $[\alpha]_D^{22}$ = +5.1 (c = 1.31, $CHCl_3$) for 61% ee (*S*)}; 98% ee by GC analysis of the corresponding acetate derivative. (Chiraldex G-TA column, 50 to 100 ^{O}C at 1 ^{O}C /min, N_2, 1 ml/min) t_R = 28.90 min for (*R*) and t_R = 25.25 min for (*S*).



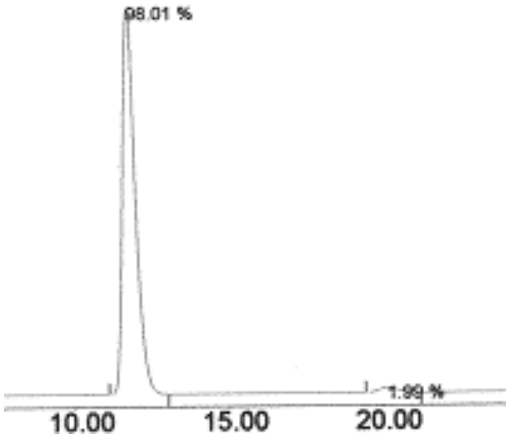
1-Cyclohexyl-1-propanol

$[\alpha]_D^{25}$ = +30.64 (c = 0.47, $CHCl_3$) {lit.[4] $[\alpha]_D^{20}$ = +6.35 (c = 3.0, $CHCl_3$) for 94.8% ee (*R*) }; 99% ee by GC analysis of the corresponding acetate derivative. (Chiraldex G-TA column, 50 to 100 ^{O}C at 5 ^{O}C /min, N_2, 1 ml/min) t_R = 15.03 min for (*R*) and t_R = 14.61 min for (*S*).



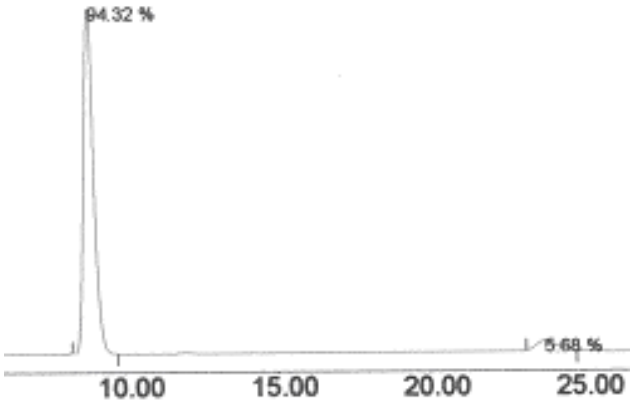
(*E*)-1- Phenyl-1-penten-3-ol

$[\alpha]_D^{25}$ = +14.74 (c = 3.46, $CHCl_3$) {lit.[3] $[\alpha]_D^{22}$ = –5.7 (c = 100, $CHCl_3$) for 96% ee (*S*)}; 96% ee by HPLC analysis (Chiralcel OD column, hexane:2-propanol = 95:5, 1 ml/min, 254 nm UV detector), t_R = 11.76 min for (*R*) and t_R = 20.15 min for (*S*).



1-Phenyl-1-pentyn-3-ol

$[\alpha]_D^{25}$ = +6.32 (c = 3.72, $CHCl_3$) {lit.[6] $[\alpha]_D^{RT}$ = -59.32 (neat) for 99% ee (*S*)}; 89% ee by HPLC analysis (Chiralcel OD column, hexane:2-propanol = 95:5, 1 ml/min, 254 nm UV detector), t_R = 8.83 min for (*R*) and t_R = 23.71 min for (*S*).



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