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New Effective Modular (Imino)Oxazoline (IMOX) Ligands for Asymmetric Catalysis. Catalytic Enantioselective Reductions of Ketones by Cathecolborane Promoted by Zn(IMOX) Metal Complexes

Manuela Locatelli and Pier Giorgio Cozzi*

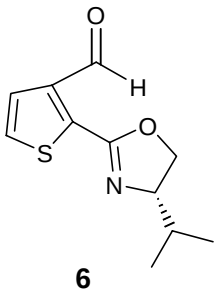
General: ^{1}H NMR spectra were recorded on Varian 200 MHz or Varian 300 MHz spectrometers. Chemical Shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard (deuterochloroform: δ 7.27 ppm). Data are reported as follows: chemical shifts, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet), coupling constants (Hz). ^{13}C NMR spectra were recorded on a Varian 50 MHz or Varian 75 MHz spectrometers with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent as the internal standard (deuterochloroform: δ 77.0 ppm). Mass spectra were performed at an ionizing voltage of 70 eV. Chromatographic purification was done with 240-400 mesh silica gel. Analytical gas chromatography (GC) was performed on a Hewlett-Packard HP 6890 gas chromatograph with a flame ionization detector and split mode capillary injection system, using a Crosslinked 5% PH ME Siloxane (30 m) column or a Megadex5 chiral (25 m) column. Analytical high performance liquid chromatograph (HPLC) was performed on a HP 1090 liquid chromatograph equipped with a variable wavelength UV detector (deuterium lamp 190-600 nm), using a Daicel Chiralcel^{TM} OD column (0.46 cm I.D. x 25 cm) (Daicel Inc.). HPLC grade isopropanol and hexane were used as the eluting solvents. Elemental analyses were carried out by using a EACE 1110 CHNOS analyzer. All the reactions were carried out under a nitrogen atmosphere in flame-dried glassware using standard inert techniques for introducing reagents and solvents. All ketones were distilled prior to use. All the other commercially obtained reagents were used as received. Neat commercially available cathecolborane (Aldrich)

stored 5°C was used for the catalytic reductions. Anhydrous CH_2Cl_2, THF, Et_2O, Toluene and CH_3CN were purchased from Fluka Co.

General procedure for the synthesis of the aldehydes 6 and 7.

In a oven dried flask cooled under vacuum and connected to a nitrogen line the oxazoline (1.03 mmol) was dissolved in anhydrous Et_2O (4 mL). The solution was cooled at – 78 °C and *n*BuLi 2.5 M in hexane (2.06 mmol) was added drop wise. The yellow solution was stirred 30 min. at – 78 °C and stirred another 30 min. at 0 °C, then cooled to –78°C. Anhydrous DMF (5.64 mmol) was added and the solution was allowed to warm to room temperature during 1 hour. After 4 additional hours the reaction mixture was quenched with water (5 mL) and the organic phase was separated. The aqueous phase was extracted with Et_2O (3 x 4 mL). The organic phases were reunited, dried over Na_2SO_4 and evaporated under reduced pressure to give an oil purified by chromatography.

Thiophen [(4*S*)-*iso*propyl-4,5-dihidro-oxazol-2yl]-3-carboxyaldehyde



6

$C_{11}H_{13}NO_2S$ Fw = 223

yield = 66 %

$[\alpha]_D$ = - 60 (c 1.01, $CHCl_3$)

^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 10.66 (s, 1 H); 7.58 (d, 1 H, J = 5.4 Hz); 7.38 (d, 1 H, J = 6 Hz); 4 .5-4.34 (m, 1 H); 4.25-4.14 (m, 2 H); 1.9-1.84 (sept, 1 H, J = 6.6 Hz); 1.05 (d, 3 H, J = 7 Hz); 0.97 (d, 3 H, J = 6.6 Hz).

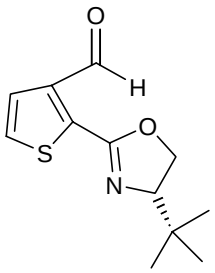
^{13}C-NMR ($CDCl_3$, 50 MHz) δ: 187.05, 157.5, 141.94, 138.75, 128.22, 127.0, 73.20, 70.91, 32.94, 18.90, 18.41.

GCMS: 57(9.4 %), 69(9.4 %), 83(23 %), 97(30 %), 111(58.3 %), 125(32 %), 152(71 %), 180(100 %), 195(58.3 %), 207(6.3 %), 223(1 %).

IR (Nujol): 2846, 1699, 1633, 1461, 1375, 1282, 1077, 725 (cm^{-1}).

Element. Anal. Calctd for $C_{11}H_{13}NO_2S$: C, 59.17; H, 5.87; N, 6.27; found: C, 59.22; H, 5.78; N, 6.24.

Thiophen [(4*S*)-*tert*-butyl-4,5-dihidro-oxazol-2yl]-3-carboxyaldehyde



7

$C_{12}H_{15}NO_2S$ Fw = 237

yield = 64 %

$[\alpha]_D$ = - 70.96 (c 1.03, $CHCl_3$)

^{1}H-NMR ($CDCl_3$, 200 MHz); δ 10.68 (s, 1 H); 7.59 (d, 1 H, J = 5.2 Hz); 7.38 (d, 1 H, J = 5.8 Hz); 4 .43 (dd, 1 H, J = 8.4, 10.2 Hz); 4.3 (t, 1 H, J = 7.6, 8.4 Hz); 4.13 (dd, 1 H, J = 7.6, 10.2 Hz); 0.98 (s, 9 H).

^{13}C-NMR ($CDCl_3$ 50 MHz) δ: 187.12; 169.91; 141.97; 136.03; 128.14; 127.10; 69.35; 69.30, 46.18; 34.18; 27.07; 25.94.

GC-MS: 57(62.5 %), 83(37.5 %), 111(85 %), 120(49 %), 152(94 %), 180(100 %), 209(59 %), 237(3 %).

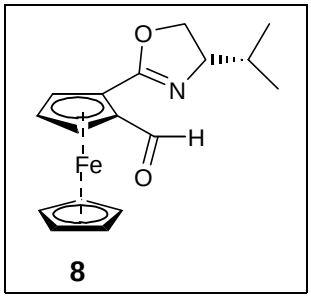
IR (Nujol): 2853, 1679 (C=O), 1646 (C=N), 1520, 1461, 1375, 1249, 1076, 738 (cm^{-1}).

Element. Anal. Calctd for $C_{12}H_{15}NO_2S$: C, 60.73; H, 6.37; N, 5.90; found: C, 60.80; H, 6.30; N, 5.88.

Synthetic procedure for the preparation of the aldehyde 8

In a oven dried flask cooled under vacuum and connected to a nitrogen line the oxazoline **3** (100 mg, 0.337 mmol) was dissolved in anhydrous Et_2O (4 mL). To the resulting solution TMEDA (0.067 mL, 0.438 mmol) was added then the reaction mixture was cooled at –78°C. *n*BuLi 2.5 M in hexane (0.175 mL, 0.438 mmol) was added drop wise. The yellow solution was stirred 30 min. at – 78 °C, then warmed at 0°C and stirred for another 10 min. The solution was cooled at –78°C then anhydrous DMF (0.434 mL, 5.64 mmol) was added and the solution was allowed to warm to room temperature during 4 hours. The reaction mixture was quenched with water (5 mL) and the organic phase was separated. The aqueous phase was extracted with Et_2O (3 x 4 mL). The organic phases were reunited, dried over Na_2SO_4 and evaporated under reduced pressure to give an oil purified by chromatography (ciclohexane:Et_2O, 4:6).

Ferrocen-[(4*S*,*Rp*)-*iso*propyl-4,5-dihydro-oxazol-2yl]-1-carboxyaldehyde



8

$C_{17}H_{19}NO_2Fe$ Fw = 325

Yield = 78 %

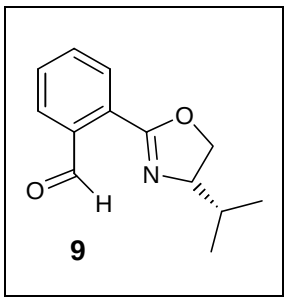
$[\alpha]_D$ = - 902 (c 0.96, $CHCl_3$)

^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 10.73 (s, 1 H); 5.11 (m, 1 H); 5.05 (m, 1 H); 4 .75 (m, 1 H); 4.32 (s, 5 H); 4.27-4.04 (m, 3 H); 1.91-1.81 (sept, 1 H, J = 6.6 Hz); 1.06 (d, 3 H, J = 6.6 Hz); 0.99 (d, 3 H, J = 6.2 Hz).

Synthetic procedure for the preparation of the aldehyde 9

In a oven dried flask cooled under vacuum and connected to a nitrogen line the oxazoline **4** (80 mg, 0.3 mmol) was dissolved in anhydrous Et_2O (4 mL). The solution is cooled at – 78 °C and *n*BuLi 2.5 M in hexane (0.18mL, 0.45 mmol) was added drop wise. The yellow solution was stirred 60 min. at – 78 °C. Anhydrous DMF (0.230 mL, 2.99 mmol) was added and the solution was allowed to warm to room temperature during 1 hour. The reaction mixture was quenched with water (5mL) and the organic phase was separated. The aqueous phase was extracted with Et_2O (3 x 4 mL). The organic phases were reunited, dried over Na_2SO_4 and evaporated under reduced pressure to give an oil purified by chromatography (ciclohexane:Et_2O, 7:3).

[(4*S*)-*iso*propyl-4,5-dihidro-oxazol-2yl]-Benzaldehyde



9

$C_{13}H_{15}NO_2$ Fw = 217

yield = 52 %

$[\alpha]_D$ = - 89.59 (c 0.97, $CHCl_3$)

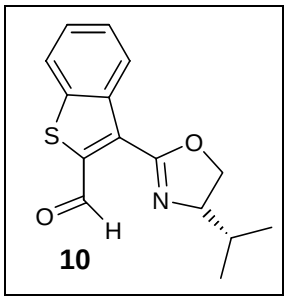
^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 10.80 (s, 1 H); 7.99-7.90 (m, 2 H); 7.63-7.58 (m, 2 H); 4.49-4.42 (m, 1 H); 4.25-4.13 (m, 2 H); 1.9-1.84 (sept, 1 H, J = 6.6 Hz); 1.06 (d, 3 H, J = 6.6 Hz); 0.98 (d, 3 H, J = 7 Hz).

GCMS: 51(26.6 %), 105(81 %), 146(100 %), 189(38.5 %), 216(0.52 %).

Synthetic procedure for the preparation of the aldehyde 10

In a oven dried flask cooled under vacuum and connected to a nitrogen line the oxazoline **5** (73 mg, 0.300 mmol) was dissolved in anhydrous Et_2O (4 mL). *n*BuLi 2.5 M in hexane (0.175 mL, 0.390 mmol) was added dropwise. The yellow solution was stirred 60 min. at – 78 °C then anhydrous DMF (0.390 mL, 0.5 mmol)) was added and the solution was allowed to warm to room temperature during 4 hours. The reaction mixture was quenched with water (5mL) and the organic phase was separated. The aqueous phase was extracted with Et_2O (3 x 4 mL). The organic phases were reunited, dried over Na_2SO_4 and evaporated under reduced pressure to give an oil purified by chromatography (cyclohexane:Et_2O, 4:6).

Benzo[b]thiophene[(4*S*)-*iso*propyl-4,5-dihydro-oxazol-3yl]-2-carboxyaldehyde



10

$C_{15}H_{15}NO_2S$ Fw = 273

yield = 64 %

$[\alpha]_D$ = - 20.0 (c 1.0, $CHCl_3$)

^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 10.78 (s, 1 H); 8.72-8.80 (m, 1 H); 7.85-7.91 (m, 1 H); 7.49-7.55 (m, 2 H); 4.61-4.48 (m, 1 H); 4.16-4.32 (m, 2 H); 1.94 (sept, 1 H, J = 6.6 Hz); 1.25 (d, 6 H, J = 6.6 Hz).

^{13}C-NMR ($CDCl_3$, 200 MHz) δ: 186.73; 158.29; 145.75; 141.12; 138.01; 129.09; 128.03; 127.28; 125.71; 122.67; 72.99; 70.10; 33.07; 19.01; 18.68.

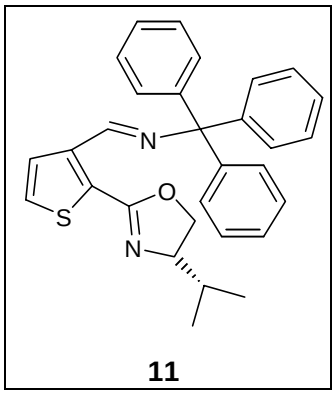
Element. Anal. Calctd for $C_{15}H_{15}NO_2S$: C, 65.91; H, 5.53; N, 5.12; found: C, 65.87; H, 5.65.; N, 5.10.

General procedure for the synthesis of the IMOX ligands 11-15

In a oven dried flask cooled under vacuum and connected to a nitrogen line the aldehyde (**6-10**) (1 mmol) was dissolved in anhydrous CH_2Cl_2 (10 mL). Anhydrous $MgSO_4$ (5mmol) and trytylamine (1mmol) were added and the reaction mixture stirred at room temperature for 120 hours. The reaction was filtered over a glass septum and the solvent was evaporated under reduced pressure. The crude product was purified by

chromatography (cyclohexane Et_2O, 9:1-8:2 on a deactivated SiO_2 (deactivated by adding 3% of Et_3N to the eluent during the preparation of the column).

Trityl-N-[(4*S*)-*iso*propyl-4,5-dihydro-oxazol-2-yl]-3-thienylmethyleneamine



11

$C_{30}H_{28}N_2OS$ Fw = 464

yield = 41 %

$[\alpha]_D$ = - 23.6 (c 0.93, $CHCl_3$)

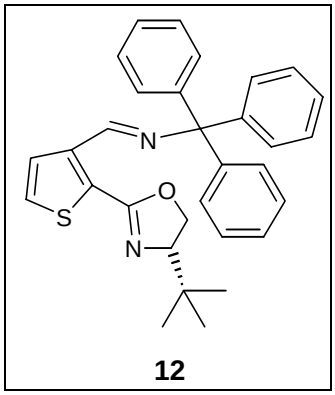
^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 8.6 (s, 1 H); 7.93 (d, 1 H, J = 4.8 Hz); 7.38 (d, 1 H, J = 5.2 Hz); 7.32-7.24 (m, 15 H); 4.26-4.12 (m, 1 H); 3.99-3.85 (m, 2 H); 1.75-1.6 (sept, 1 H, J = 6.6 Hz); 0.86 (d, 3 H, J = 6.6 Hz,); 0.8 (d, 3 H, J = 6.6 Hz).

^{13}C-NMR ($CDCl_3$, 200 MHz) δ: 158.12; 155.74; 145.73; 141.97; 130.65; 129.77; 127.75; 127.59; 126.6; 78.66; 72.85; 70.10; 32.72; 18.89; 18.36.

IR (Nujol): 2846, 1639, 1454 , 1441, 1375, 1242, 1050, 1010, 751, 698 (cm^{-1}).

Element. Anal. Calctd for $C_{30}H_{28}N_2OS$: C, 77.55; H, 6.07; N, 6.03; found: C, 77.50; H, 6.12.; N, 6.02.

Trityl-N-[(4*S*)-*tert*-butyl-4,5-dihydro-oxazol-2yl]-3-thienylmethyleneamine



12

$C_{31}H_{30}N_2OS$ Fw = 478

Yield = 47 %

$[\alpha]_D$ = - 21.3 (c 1.1, $CHCl_3$)

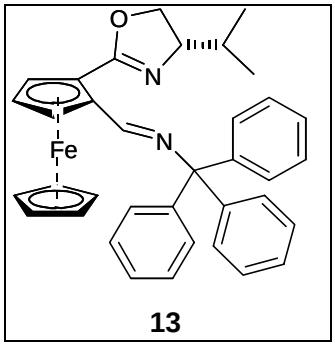
^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 8.68 (s, 1 H); 7.93 (d, 1 H, J = 5.1 Hz); 7.38 (d, 1 H, J = 5.1 Hz); 7.29-7.26 (m, 15 H); 4.15 (dd, 1 H, J = 7.8, 8 Hz); 4.00 (dd, 1 H, J = 7.8, 15.8 Hz); 3.89 (dd, 1 H, J = 8, 15.8 Hz); 0.79 (s, 9 H,).

^{13}C-NMR ($CDCl_3$, 50 MHz) δ: 157.92; 155.82; 145.78; 142.05; 130.56; 129.8; 127.84; 127.62; 126.61; 78.72; 76.37; 76.37; 68.51; 33.88; 25.94.

IR (Nujol): 2840, 1639, 1461, 1434, 1375, 1242, 1076, 1029, 751, 698 (cm^{-1}).

Element. Anal. Calctd for $C_{31}H_{30}N_2OS$: C, 77.79; H, 6.32; N, 5.85; found: C, 77.68; H, 6.30 ; N, 5.83.

Trityl-N-[(4*S*,*Rp*)-*iso*propyl-4,5-dihydro-oxazol-2yl]-1-ferrocenylmethyleneamine



13

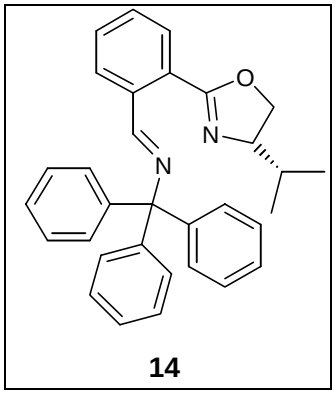
$C_{36}H_{34}FeN_2O$ Fw = 565

Yield = 65 %

$[\alpha]_D$ = - 386 (c 1.0, $CHCl_3$)

^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 8.46 (s, 1 H); 7.40-7.25 (m, 15 H); 5.36-5.34 (m, 1 H); 4.85-4.83 (m, 1 H); 4.57-4.54 (m, 1 H); 4.15 (s, 5 H); 4.10-3.86 (m, 3 H); 1.66-1.62 (sept, 1 H, J = 6.6 Hz); 0.81 (d, 3 H, J = 6.8 Hz); 0.77 (d, 3 H, J = 7 Hz).

Trityl-N-[(4*S*)-*iso*propyl-4,5-dihidro-oxazol-2yl]-1-phenylmethyleneamine



14

$C_{32}H_{30}N_2O$ Fw = 458

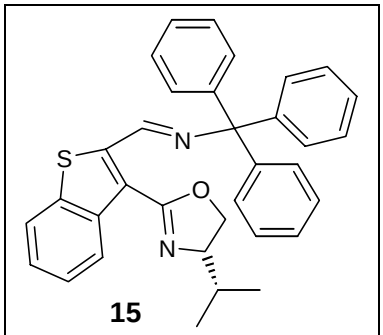
Yield = 17 %

$[\alpha]_D$ = - 33.1 (c 0.92, $CHCl_3$)

^{1}H-NMR ($CDCl_3$, 200 MHz) δ: 8.54 (s, 1 H); 8.35 (dd, 1 H, J = 1, 7.6 Hz); 7.8 (dd, 1 H, J = 1.8, 5.4 Hz); 7.56 (dt, 1 H, J = 1, 5.8 Hz); 7.46 (dt, 1 H, J = 1, 7.4 Hz); 7.38-7.24 (m, 15 H); 4.17 (dd, 1 H, J = 6.2, 7.6 Hz), 3.95 (dd, 1 H, J = 6.2, 14.2 Hz), 3.87 (dd, 1H, J = 7.6, 14.2 Hz), 1.71-1.64 (sept, 1 H, J = 7 Hz); 0.87 (d, 3 H, J = 7 Hz), 0.80 (d, 3 H, J = 6.6 Hz).

Trityl-N-[(4*S*)-*iso*propyl-4,5-dihydro-oxazol-3yl]-2

Benzo[b]thiophenemethyleneamine



15

$C_{34}H_{30}N_2OS$ Fw = 514

Yield = 48 %

$[\alpha]_D$ = - 23.0 (c 0.78, $CHCl_3$)

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 8.68 (s, 1 H); 7.93 (d, 1H, J = 5.1 Hz); 7.38 (d, 1 H, J = 5.1 Hz); 7.29-7.26 (m, 15 H); 4.15 (dd, 1 H, J = 7.8, 8 Hz); 4.00 (dd, 1 H, J = 7.8, 15.8 Hz); 3.89 (dd, 1 H, J = 8, 15.8 Hz); 0.79 (s, 9 H,).

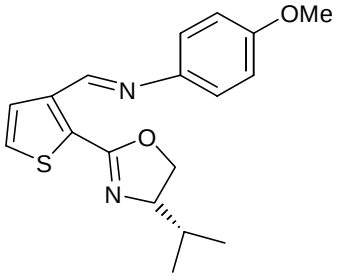
^{13}C-NMR ($CDCl_3$, 50 MHz) δ: 157.92; 155.82; 145.78; 142.05; 130.56; 129.8; 127.84; 127.62; 126.61; 78.72; 76.37; 76.37; 68.51; 33.88; 25.94.

IR (Nujol): 2840, 1639, 1461, 1434, 1375, 1242, 1076, 1029, 751, 698 (cm^{-1}).
Element. Anal. Calctd for $C_{34}H_{30}N_2OS$: C, 79.34; H, 5.88; N, 5.44; found: C, 79.30; H, 5.91; N, 5.42.

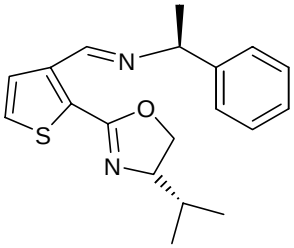
General procedure for the catalytic reduction

In a oven dried flask IMOX ligand (0.024 mmol) and $Zn(OTf)_2$ (0.024 mmol) were mixed and stirred under vacuum for 5-10 min. Anhydrous CH_2Cl_2 (2mL) was successively added and the resulting solution was stirred for 4 hours at room temperature. The freshly distilled ketone (0.6mol.) was added then the clear pale yellow solution was cooled at –20. Neat cathecolborane (0.9 mmol) was added dropwise. The reaction was left without stirring in a freezer at –15°C for 48 hours. The reaction mixture was diluted with Et_2O (5mL), then NaOH 2M (5mL) was carefully added (attention, gas evolution). The two phases were stirred 20-30minutes and during this time period the aqueous phase turning dark brown. The phases were separated and the aqueous phase extracted with Et_2O (2 x 3mL). The organic phases were collected and dried over Na_2SO_4, evaporated under reduced pressure and the resulting oil was purified by chromatography (cyclohexane: Et_2O, 9:1-7:3).

The following IMOX ligands derived from different imines were employed in the reduction of acetophenone performing the reduction at 0°C. The enantiomeric excesses recorded are indicated under the figures.



Ee = 0



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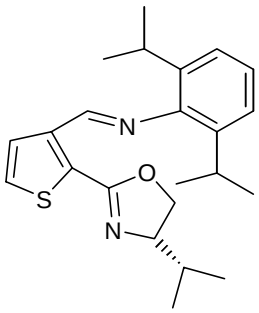
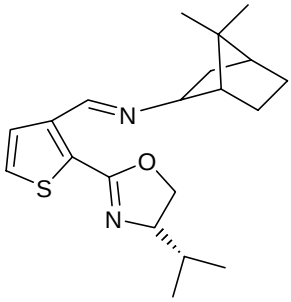
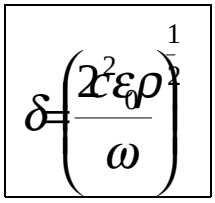


Ee = 0



Ee = 12

(*R*)-1-phenylethanol



$C_8H_{10}O$ Fw = 122.16

e.e. = 90 % yield = 75 %

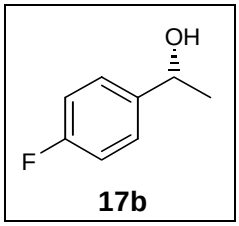
$[\alpha]_D$ = + 44.4 (c 2.4, $CHCl_3$). Lit.[1] (*R*)-1-phenylethanol $[\alpha]_D$ = + 45.2 (c = 1.01, CH_2Cl_2)

GC analysis: column temperature, 115 °C (isothermal)

t_r (major) (R)-1-phenylethanol = 12.61 min.

t_r (minor) (S)-1-phenylethanol = 15.77 min.

(*R*)-1-(4'fluorophenyl)ethanol



17b

C_8H_9FO Fw = 140.15

e.e. = 89 % yield = 76 %

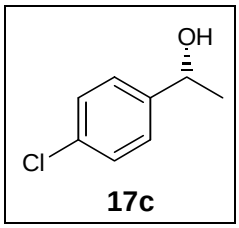
$[\alpha]_D$ = + 39 (c 1.16, $CHCl_3$) Lit.[2] (S)-1-(4'fluorophenyl)ethanol $[\alpha]_D$ = -37.2 (c 0.93, MeOH).

GC analysis: column temperature, 120 °C (isothermal)

t_r (major) (R)-1-(4'fluorophenyl)ethanol: 4.21 min.

t_r (minor) (S)-1-(4'fluorophenyl)ethanol: 5.5 min.

(*R*)-1-(4'chlorophenyl)ethanol



17c

C_8H_9ClO Fw = 156.03

e.e. = 88 % yield = 84 %

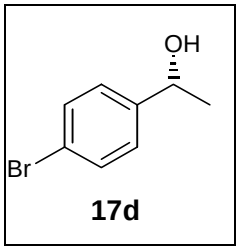
$[\alpha]_D$ = + 38.6 (c 1.01, $CHCl_3$). Lit.[3] (S)- 1-(4'chlorophenyl)ethanol $[\alpha]_D$ = -49.6 (c 1.8 , Et_2O).

GC analysis: column temperature, 120 °C (isothermal)

t_r (major) (R)-1-(4'chlorophenyl)ethanol: 19.93 min.

t_r (minor) (S)-1-(4'chlorophenyl)ethanol: 25.07 min.

(*R*)-1-(4'bromophenyl)ethanol



17d

C_8H_9BrO Fw = 201.06

e.e. = 87 % yield = 83 %

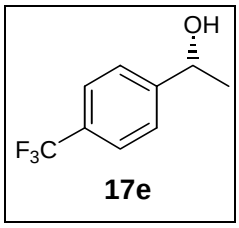
$[\alpha]_D$ = + 29 (c 1.08, $CHCl_3$).) Lit.[4] (S)-1-(4'bromophenyl)ethanol $[\alpha]_D$ = -35.1 (c 0.93, MeOH).

GC Analysis: column temperature, 130 °C (isothermal)

t_r (major) (R)-1-(4'bromophenyl)ethanol: 20.71 min.

t_r (minor) (S)-1-(4'bromophenyl)ethanol: 26.36 min.

(*R*)-1-(4'trifluorophenyl)ethanol



17e

$C_9H_9F_3O$ Fw = 189.97

e.e. = 86 % yield = 81 %

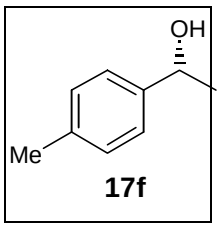
$[\alpha]_D$ = + 28 (c 1.02, $CHCl_3$). Lit.[5] (S)-1-(4'trifluorophenyl)ethanol $[\alpha]_D$ = -28.1 (c 1.13, MeOH).

GC analysis: column temperature, 120 °C (isothermal)

t_r (major) (R)-1-(4'trifluorometylphenyl)ethanol: 2.63 min.

t_r (minor) (S)-1-(4'trifluoromethylphenyl)ethanol: 3.61 min.

(*R*)-1-(4'methylphenyl)ethanol



17f

$C_9H_{12}O$ Fw = 136.09

e.e. = 87 % yield = 62 %

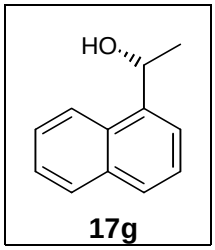
$[\alpha]_D$ = + 35 (c 1.0, $CHCl_3$). Lit.[4] (S)-1-(4'methylphenyl)ethanol $[\alpha]_D$ = -40.8 (c 1.3 , MeOH).

GC analysis: column temperature, 115 °C (isothermal)

t_r (major) (R)-1-(4'methylphenyl)ethanol: 11.81 min.

t_r (minor) (S)-1-(4'methylphenyl)ethanol: 15.87 min.

(*R*)-1-naphtylethanol



$C_{12}H_{12}O$ Fw = 172.09

e.e. = 86 % yield = 85 %

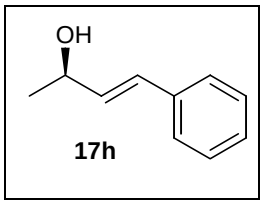
$[\alpha]_D$ = + 46.5 (c 1.06, $CHCl_3$). Lit.[6] (S)-1-naphtyletanol $[\alpha]_D$ = -54.8 (c 3.34 , $CHCl_3$).

GC analysis: column temperature, 150 °C (isothermal)

t_r (minor) (S)-1- naphtylethanol: 27.9 min.

t_r (major) (R)-1- naphtylethanol: 29.15 min.

(*R*)-*E*-4-Phenyl-but-3-en-2-ol



$C_{10}H_{12}O$ Fw = 148.09

e.e. = 92 % yield = 54 %

$[\alpha]_D$ = + 7.6 (c 0.83, $CHCl_3$). Lit. [1] (R)-1-*E*-4-phenyl-but-3-en-2-ol

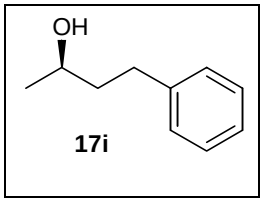
$[\alpha]_D$ = +16 (c 2.5 , $CHCl_3$)

HPLC analysis: column OD, hexane: *iso*propanol 90:10 (isocratic), flux 0.5ml/min.

t_r (major) (R)-E-4-phenyl-but-3-en-2-ol: 11.57 min.

t_r (minor) (S)-E-4-phenyl-but-3-en-2-ol: 15.31 min.

(*R*)-4-Phenyl-butan-2-ol



$C_{10}H_{14}O$ Fw = 150.10

e.e. = 53 % yield = 92 %

$[\alpha]_D$ = - 8 (c 1.07, $CHCl_3$). Lit.[7] (R)-4-phenyl-2-butanol

$[\alpha]_D$ = -21.2 (c 1 , benzene)

HPLC analysis: column OD, 80:20 hexane: *iso*propanol 80:20 (isocratic), flux 0.5ml/min.

t_r (R)-4-phenyl-2-butanol: 12.80 min. t_r (S)-4-phenyl-2-butanol: 18.03 min.

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