Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Efficient Synthesis of (*S*,*R*)- Bn-Yanphos and Rh/ (*S*,*R*)- Bn-Yanphos Catalyzed Asymmetric Hydroformylation of Vinyl Heteroarenes

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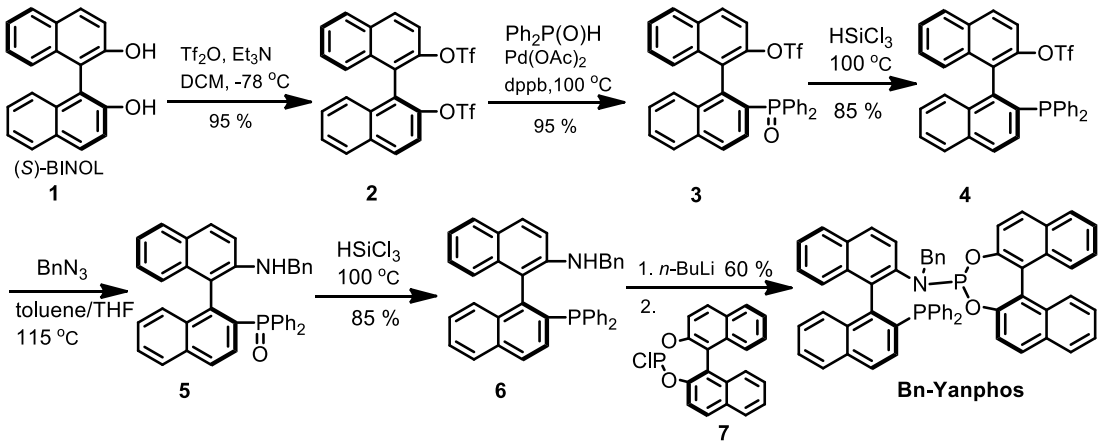
1. General Information

All reactions and manipulations that were sensitive to moisture or air were performed in a nitrogen-filled glovebox or using standard Schlenk techniques, unless otherwise noted. Solvents were dried with standard procedures, degassed with N_2 and transferred by syringe. NMR spectra were recorded on Bruker ADVANCE III (400 MHz) spectrometers for ^{1}H NMR and ^{13}C NMR. $CDCl_3$ was the solvent used for the NMR analysis, with tetramethylsilane as the internal standard. Chemical shifts were reported upfield to TMS (0.00 ppm) for ^{1}H NMR and relative to $CDCl_3$ (77.3 ppm) for ^{13}C NMR. Optical rotation was determined using a Perkin Elmer 343 polarimeter. HPLC analysis was conducted on an Agilent 1260 Series instrument. GC analysis was carried out on Angilent 1200 Series instrument using chiral capillary columns. Column Chromatography was performed with silica gel Merck 60 (300-400 mesh). Thin layer chromatography (TLC) was performed on EM reagents 0.25 mm silica 60-F plates. All new products were further characterized by HRMS. A positive ion mass spectrum of sample was acquired on a Thermo LTQ-FT mass spectrometer with an electrospray ionization source.

2. Procedures for the preparation of (*S,R*)- Bn-Yanphos



(*S*)-[1,1'-binaphthalene]-2,2'-diyl bis(trifluoromethanesulfonate) （2）

A single necked flask charged with a solution of (*S*)-BINOL (**1**) (14.3 g, 50 mmol) and Et_3N (15.2 g, 20.9 mL, 150 mmol) in CH_2Cl_2 (125 mL) was cooled to -78 °C, and Tf_2O (31.0 g, 18.5 mL, 110 mmol) was added dropwise. Then the resulting mixture was warmed to 0 °C for 2 h. After removal of the solvent under vacuum, the resulting thick residue was filtrated through a short silica gel column and flushed with petroleum ether/ethyl acetate (5:1, v/v). The filtrate was concentrated under reduce pressure, and the product **2** was obtained as a white solid (27.5 g, >99% yield) and used for the next step without further purification.[1] ^{1}H NMR (400 MHz，$CDCl_3$) δ/ppm = 7.27(d, *J* = 8.5 Hz, 2H), 7.42(ddd, *J* = 1.1 Hz, 6.8 Hz, 8.2 Hz, 2H), 7.59(ddd, *J* = 1.0 Hz , 7.0 Hz, 8.1 Hz, 2H), 7.63(d, *J* = 9.1 Hz, 2H), 8.02(d, *J* = 8.2 Hz, 2H), 8.15(d, *J* = 9.1 Hz, 2H); ^{13}C NMR(100 MHz，$CDCl_3$) δ：118.3, 119.4, 123.6, 126.8, 127.4, 128.1, 132.1, 132.5, 133.2, 145.5.

(*S*)-2'-(diphenylphosphoryl)-[1,1'-binaphthalen]-2-yl trifluoromethanesulfonate (3)

To a schlenk flask charged with **2** (1.10 g, 2.0 mmol), diphenylphosphine oxide (0.81 g, 4 mmol), $Pd(OAc)_2$ (22.4 mg, 0.1 mmol) and dppb (42.7 mg, 0.1 mmol) in DMSO (8.8 mL) was added DIEA (1.03 g, 1.4 mL, 8.0 mmol) under argon. The resulting mixture was stirred at 100 °C for 12 h. Then the mixture was cooled to room temperature, diluted with EtOAc (50 mL), washed with water (20 mL x 3), brine (20 mL), successively. The organic phase was dried over anhydrous Na_2SO_4, filtered, concentrated, and the crude residue was further purified by flash chromatography on silica gel (petroleum ether/ethyl acetate = 1:1, v/v) to afford the desired product **3** as a white solid (1.08 g, 90% yield).[2] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48–7.59 (m, 6H), 7.67–7.72 (m, 4H), 8.07 (d, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ: 129.0 (d, $J_{C,P}$ = 13.5 Hz), 130.8 (d, $J_{C,P}$ = 11.5 Hz), 131.5 (d, $J_{C,P}$ = 101.6 Hz), 132.7 (d, $J_{C,P}$ = 2.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 21.5.

(*S*)-2'-(diphenylphosphino)-[1,1'-binaphthalen]-2-yl trifluoromethanesulfonate (4)

To a dried sealed tube charged with **3** (0.48 g, 0.81 mmol) in dry toluene (20 mL), Et_3N (0.57 g, 0.79 mL, 5.67 mmol) and $HSiCl_3$ (0.54 g, 0.41 mL, 4.05 mmol)

were added successively under argon at 0 °C. The resulting mixture was stirring at 100 °C for 12 h. After cooled to 0 °C, diluted by Et_2O (10 mL), quenched with several drops of saturated Na_2CO_3, the mixture was filtered by a short celite column, and washed with Et_2O (10 mL x 3). The filtrate was dried over anhydrous Na_2SO_4, filtered, concentrated, and the residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate = 20:1, v/v) to afford the desired product **4** as a white solid (0.394 g, 83% yield).[3] ^{1}H NMR (400 MHz，$CDCl_3$) δ：6.96-8.11 (m, 22H); ^{31}P NMR(162 MHz, $CDCl_3$) δ：-13.3.

(*S*)-(2'-(benzylamino)-[1,1'-binaphthalen]-2-yl)diphenylphosphine oxide (5)

Phosphine **4** (150 mg, 0.20 mmol) was added to a solution of benzyl azide (33 mg, 0.24 mmol) in toluene/THF 1:1 (0.1 M). The reaction was stirred at 115 °C. After 17 h the reaction mixture was concentrated in vacuo yielding the phosphonium salt. The remaining phosphonium salt was stirred for 2 h at 65 °C in a mixture of THF (2 mL), EtOH (2 mL) and aqueous 0.1 M NaOH (2 mL). After cooling the mixture, Et_2O was added and the organic phase was washed with H_2O (25 mL) and brine (25 mL). The organic layer was dried (Na_2SO_4) and concentrated in vacuo. Purification by column chromatography (Et_2O: pentane = 4:1→15:1) afforded **5** (0.11 g, 0.20 mmol, 99%) as a yellow foam.[4] ^{1}H NMR (400 MHz): δ 8.02 (d, J = 8.5 Hz, 1H), 7.96 (d, J = 8.0 Hz, 1H), 7.90 (dd, J = 9.0 Hz, 1H), 7.73 (d, J = 11.0 Hz, 1H), 7.72 (d, J= 11.5 Hz, 1H), 7.58 (t, J = 6.5 Hz, 1H), 7.42-7.50 (m, 3H), 7.25-7.36 (m, 10H), 7.22 (m, 1H), 7.03-7.08 (m, 2H), 6.97 (t, J = 6.5 Hz, 1H), 6.78-6.88 (m, 2H), 6.86 (d, J = 9.0 Hz, 1H), 6.60 (d, J = 8.5 Hz, 1H), 4.42 (s, 2H), 4.19 (br. s, 1H). ^{13}C NMR (100 MHz, nonaromatic only): δ 48.2. ^{31}P NMR (162 MHz): δ 28.4. IR (cm^{-1}): 3342, 1624, 1526, 1468. HRMS: calcd for $C_{39}H_{31}NOP$ $[M+H]^+$: 560.2143, found: 560.2139.

(*S*)-N-benzyl-2'-(diphenylphosphino)-[1,1'-binaphthalen]-2-amine (6)

To a dried sealed tube charged with **5** (0.45 g, 0.81 mmol) in dry toluene (20 mL), Et_3N (0.57 g, 0.79 mL, 5.67 mmol) and $HSiCl_3$ (0.55 g, 0.41 mL, 4.05 mmol) were added successively under argon at 0 °C. The resulting mixture was stirring at 100 °C for 12 h. After cooled to 0 °C, diluted by Et_2O (10 mL), quenched with several drops of saturated Na_2CO_3, the mixture was filtered by a short celite column, and

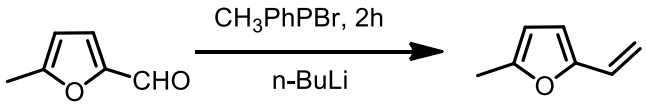
washed with Et_2O (10 mL x 3). The filtrate was dried over anhydrous Na_2SO_4, filtered, concentrated, and the residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate = 20:1, v/v) to afford the desired product **6** as a yellow foam. (0.39 g, 83% yield). ^{1}H NMR (400 MHz，$CDCl_3$) δ 7.90-7.88 (m, 2H), 7.82 (d, *J* = 8.9 Hz, 1H), 7.71 (d, *J* = 8.9 Hz, 1H), 7.65-7.53 (m, 2H), 7.51-7.49 (m, 2H), 7.47-7.03 (m, 17H), 7.00-6.97 (m, 1H), 6.62 (d, *J* = 8.4 Hz, 1H), 4.18 (dd, *J* = 6.1 Hz, 1H), 4.01 (dd, *J* = 5.8 Hz, 1H), 3.70 (br. s, 1H). ^{31}P NMR (162 MHz): δ -13.9.

(11b*R*)-N-benzyl-N-((*S*)-2'-(diphenylphosphino)-[1,1'-binaphthalen]-2-yl)dinapth o[2,1-d:1',2'-f][1,3,2]dioxaphosphepin-4-amine (Yanphos)

To a solution of **6** (0.27 g, 0.5 mmol) in THF (5 mL) at -78°C was added dropwise *n*-BuLi (0.65 mmol, 0.26mL of 2.5 M hexane solution). The reaction mixture was stirred for 4h to give a deep red solution, and **7** (262 mg, 0.75 mmol) in THF (5 mL) was added dropwise. After addition, the cooling bath was removed and the mixture was stirred at room temperature overnight. The volatiles were evaporated under reduced pressure. To the residue was added CH_2Cl_2(10 mL), and the mixture was filtered to remove the salt. The filtration was concentrated and subjected to chromatography on silica gel (eluted with hexane/EtOAc 10:1) to afford pure ligand (*S,R*)- Bn-Yanphos (145 mg) in 60% yield.[5] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, *J* = 8.5 Hz, 1 H), 8.15 (d, *J*= 8.0 Hz, 1 H), 7.97 (d, *J* = 8.0 Hz, 1 H), 7.92 (d, *J* = 8.0 Hz, 1 H), 7.74–7.60 (m, 5 H), 7.43–7.01 (m, 23 H), 6.87–6.83 (m, 2 H), 6.79–6.75 (m, 2 H), 6.45–6.42 (m, 1 H), 6.24 (d, *J* = 8.5 Hz, 1 H), 5.93 (d, *J* = 8.5 Hz, 1 H), 3.82 (d, *J* =14.5 Hz, 1 H), 3.21 (d, *J* =14.5 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.1, 150.0, 149.8, 142., 141.9, 138.7, 128.2, 127.9, 137.8, 135.6, 135.4, 133.9, 133.5, 133.4, 132.0, 131.8, 131.7, 131.6, 130.7, 130.5, 130.3, 129.8, 129.8, 128.8, 128.7, 128.6, 128.5, 128.4, 128.4, 128.3, 128.2, 128.1, 128.0, 127.9, 127.8, 127.7, 127.4, 127.2, 127.2, 127.0, 126.8, 126.7, 126.1, 126.0, 125.3, 124.8, 124.6, 122.7, 122.5, 122.1, 51.3 ppm; ^{31}P NMR (162 MHz, $CDCl_3$) δ：138.41 (d, *J* = 78.6 Hz), 11.86 ppm (d, *J* = 78.6 Hz).

3. Procedures for the preparation of substrates

General procedure 1 for the synthesis of styrenes *via* Wittig olefination of aldehydes

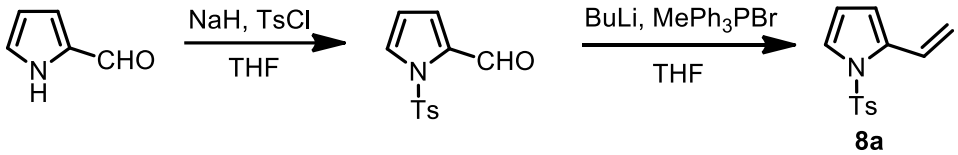


Synthesis of 2-methyl-5-vinylfurane (**8c**)

Methyl triphenyl phosphonium bromide (3.53 g, 9.894 mmol) was dissolved in dry THF (78.5 mL) under nitrogen atmosphere. At 23°C *n*-BuLi (3.95 mL, 9.894 mmol, 2.5 M in hexane) was added dropwise and the mixture was stirred for 15 min. Afterwards a solution of 5-methylfuran-2-carbaldehyde (1.03 g, 9.423 mmol) in dry THF (5 mL) was added. The reaction mixture was stirred at 23°C for 2 h, before it was quenched by addition of saturated aqueous ammonium chloride solution (50 mL) and extracted with methylene chloride (3x 30 mL). The combined organic layers were dried over sodium sulfate and the solvent was removed. The oily residue was purified by flash-chromatography through silica gel. 2-methyl-5-vinylfurane 8c was obtained as a colorless oil (356 mg, 35%). The analytical data were in complete agreement with the previously published data.[6]^{1}H-NMR (400 MHz, $CDCl_3$): δ 6.42 (dd, J = 17.5, 11.3 Hz, 1H), 6.12 (d, J = 3.2 Hz, 1H), 5.94 (ddd, J = 2.9 Hz, 1.9 Hz, 0.9 Hz, 1H), 5.56 (dd, J = 17.5 Hz, 1.3 Hz, 1H), 5.05 (dd, J = 11.3 Hz, 1.3 Hz, 1H), 2.29 (s, 3H).

Following the general procedure 1, **8d**, **8i** were obtained as a colorless oil.

General procedure 2 for the synthesis of 1-(Toluene-4-sulfonyl)-1H-pyrrole-2-carbaldehyde (**8a**)



8a

Synthesis of 1-(Toluene-4-sulfonyl)-1H-pyrrole-2-carbaldehyde

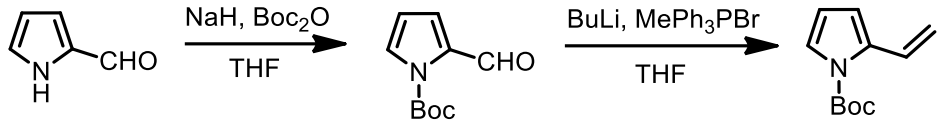
A solution of pyrrole-2-carbaldehyde (1.0 g, 11 mmol, 1 equiv.) in THF (10 mL)

was added over 10 min to a suspension of NaH (0.30 g, 13 mmol, 1.2 equiv.) in THF (60 mL) at 23 °C under argon. After 1 h, tosyl chloride (2.2 g, 12 mmol, 1.1 equiv.) was added. After stirring for 13 h at 23 °C, the reaction mixture was quenched with sat. NH_4Cl (10 mL), diluted with water (200 mL) and extracted with Et_2O (3x100 mL). The combined organic layer were washed with brine (50 mL), dried over Na_2SO_4 and the solvent was removed under reduced pressure. The crude product was purified by column chromatography (AcOEt/hexane 1:10) to give desired product (2.5 g, 9.9 mmol, 94%) as a colorless solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ/ppm 9.96 (s, 1H, aldehyde H), 7.79 (d, *J* = 8.4 Hz, 2H, phenyl H), 7.61 (m, 1H, pyrrole H), 7.31 (d, *J* = 8.1 Hz, 2H, phenyl H), 7.14 (m, 1H, pyrrole H), 6.40 (m, 1H, pyrrole H), 2.40 (s, 3H, CH_3).

Synthesis of 1-(Toluene-4-sulfonyl)-2-vinyl-1H-pyrrole (**8a**)

n-BuLi (1.6M in hexane, 5.5 mL, 8.8 mmol, 1.1 equiv.) was added to a suspension of methyltriphenylphosphonium bromide (3.4 g, 9.5 mmol, 1.2 equiv.) in THF (70 mL) at 0 °C under argon. After stirring for 2 h, the reaction mixture was cooled to -78 °C and a solution of 1-(Toluene-4-sulfonyl)-1H-pyrrole-2-carbaldehyde (2.0 g, 8.0 mmol, 1.0 equiv.) in THF (10 mL) was added dropwise. The reaction was warm up to 23°C over 12 h, quenched with water (150 mL) and extracted with Et_2O (3x100 mL). The combined organic layer were washed with water (50 mL) and brine (50 mL), dried over Na_2SO_4 and the solvent was removed under reduced pressure. The crude product was purified by column chromatography (AcOEt/hexane 1:15) to give **8a** (1.8 g, 7.3 mmol, 91%) as a colorless solid. ^{1}H NMR ($CDCl_3$, 400 MHz): *δ* 7.69 (d, *J* = 8.4 Hz, 2H, phenyl H), 7.31-7.28 (m, 1H, pyrrole H), 7.27 (d, *J* = 8.1 Hz, 2H, phenyl H), 7.10 (dd, *J* = 17.4 Hz, 11.2 Hz, 1H, vinyl H), 6.44 (m, 1H, pyrrole H), 6.24 (m, 1H, pyrrole H), 5.48 (dd, *J* = 17.4 Hz, 1.2 Hz, 1H, vinyl H), 5.15 (dd, *J* = 11.2 Hz, 1.5 Hz, 1H, vinyl H), 2.39 (s, 3H, CH_3);

General procedure 3 for the synthesis of 2-Vinyl-pyrrole-1-carboxylic acid *tert*-butyl ester (**8b**)



8b

2-Formyl-pyrrole-1-carboxylic acid tert-butyl ester

A solution of pyrrole-2-carbaldehyde (1.0 g, 11 mmol, 1.0 equiv) in THF (10 mL) was added over 10 min to a suspension of NaH (0.30 g, 13 mmol, 1.2 equiv.) in THF (60 mL) at 23 °C under argon. After 1 h, Boc anhydride (2.5 g, 12 mmol, 1.1 equiv) was added. After stirring for 13 h at 23 °C, the reaction mixture was quenched with sat. NH_4Cl (10 mL), diluted with water (200 mL) and extracted with Et_2O (3x100 mL). The combined organic layer were washed with brine (50 mL), dried over Na_2SO_4 and the solvent was removed under reduced pressure. The crude product was purified by column chromatography (AcOEt/hexane 1:30) to give 2-Formyl-pyrrole-1-carboxylic acid *tert*-butyl ester (2.0 g, 10 mmol, 97%) as an oil. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 10.29 (s, 1H, aldehyde H), 7.41 (m, 1H, pyrrole H), 7.14 (m, 1H, pyrrole H), 6.24 (m, 1H, pyrrole H), 1.61 (s, 9H, CH_3).

2-Vinyl-pyrrole-1-carboxylic acid tert-butyl ester (**8b**)

n-BuLi (1.6M in hexane, 5.5 mL, 8.8 mmol, 1.1 equiv.) was added to a suspension of methyltriphenylphosphonium bromide (3.4 g, 9.5 mmol, 1.2 equiv.) in THF (70 mL) at 0 °C under argon. After stirring for 2 h, the reaction mixture was cooled to -78 °C and a solution of 91 (1.6 g, 8.0 mmol, 1.0 equiv) in THF (10 mL) was added dropwise. The reaction was warm up to 23 °C over 12 h, quenched with water (150 mL) and extracted with Et_2O (3x100 mL). The combined organic layer were washed with water (50 mL) and brine (50 mL), dried over Na_2SO_4 and the solvent was removed under reduced pressure. The crude product was purified by column chromatography (AcOEt/hexane 1:30) to give **8b** (1.22 g, 6.33 mmol, 79%) as a yellow oil. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.25 (m, 1H, pyrrole H), 7.23 (dd, J = 17.4 Hz, 11.2 Hz, 1H, vinyl H), 6.43 (m, 1H, pyrrole H), 6.14 (m, 1H, pyrrole H), 5.53 (dd, J = 17.4 Hz, 1.6 Hz, 1H, vinyl H), 5.12 (dd, J = 11.2 Hz, 1.6 Hz, 1H, vinyl H), 1.61 (s, 9H, CH_3).

Following the general procedure 2, **8e** was obtained as a colorless oil (1.04 g,

3.5 mmol, 85%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.94 (d, 2H), 7.74 (d, *J* = 8.7 Hz, 2H, phenyl H), 7.40 (d, 2H), 7.33 (dd, 1H, indole H), 6.87 (dd, 1H), 6.66 (1H, indole H), 6.63 (dd, 1H, vinyl H), 5.80 (d, *J* = 17.7 Hz, 1H, vinyl H), 5.35 (d, *J* = 11.2 Hz, 1H, vinyl H), 2.31 (s, 3H, CH_3) and **8g** was obtained as a colorless oil (0.98 g, 3.3 mmol, 85%). ^{1}H NMR ($CDCl_3$, 400 MHz) *δ* 8.01 (m, *J* = 8.1 Hz, 1H, indole H), 7.82-7.73 (m, 1H, indole H), 7.78 (d, *J* = 8.7 Hz, 2H, phenyl H), 7.62 (s, 1H, indole H), 7.37-7.24 (m, 2H, indole H), 7.20 (d, *J* = 7.8 Hz, 2H, phenyl H), 6.77 (m, *J* = 17.7, 11.2 Hz, 1H, vinyl H), 5.80 (m, *J* = 18.0 Hz, 1H, vinyl H), 5.35 (m, *J* = 11.2 Hz, 1H, vinyl H), 2.31 (s, 3H, CH_3).

Following the general procedure 3, **8f** was obtained as a yellow oil (0.84g, 3.5 mmol, 80%). ^{1}H NMR ($CDCl_3$, 400 MHz): *δ* 8.11 (d, *J* = 7.9 Hz, 1H, indole H), 7.93 (d, *J* = 7.6 Hz, 1H, indole H), 7.33 (s, 1H, indole H), 6.87 (1H, indole H), 6.66 (1H, indole H), 6.63 (vinyl H), 5.84 (d, *J* = 17.8 Hz, 1H, vinyl H), 5.35 (d, *J* = 11.3 Hz, 1H, vinyl H), 1.70 (s, 9H, CH_3); and **8h** was obtained as a yellow oil (0.72 g, 3.0 mmol, 80%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.21 (d, *J* = 7.9 Hz, 1H, indole H), 7.81 (d, *J* = 7.6 Hz, 1H, indole H), 7.65 (s, 1H, indole H), 7.39-7.25 (m, 2H, indole H), 6.83 (dd, *J* = 17.8 Hz, 11.3 Hz, 1H, vinyl H), 5.84 (d, *J* = 17.8 Hz, 1H, vinyl H), 5.35 (d, *J* = 11.3 Hz, 1H, vinyl H), 1.70 (s, 9H, CH_3).

Synthesis of N-vinylindole (**8j**)

N-vinylindole was obtained by the N-alkylation of indole. Under the nitrogen atmosphere, indole (5.0 g, 38 mmol) was added to anintensely stirred mixture of 1,2-dichloroethane (100 g, 1.0 mol), tetrabutylammonium bromide (0.25 g, 0.8 mmol), KOH (14 g,250 mmol), and K_2CO_3 (11 g, 80 mmol) at room temperature. The stirring was continued at 50 °C for 72 h. After cooling, the inorganic material was filtered off, and the organic solvent was removed by evaporation. A mixture of the condensate, which corresponds to crude 3-chloroethylindole (5.0 g, 26 mmol), KOH (2.2 g, 240 mmol), and hydroquinone (30 mg, 0.27 mmol), was placed in toluene (100 mL) and refluxed for 3 h. After the toluene solution was evaporated under reduced pressure, the organic material was extracted from the reaction mixture by means of methylene

chloride (30 mL) with water (50 mL). The extract was then dried over anhydrous $MgSO_4$ and filtered, and the solvent was evaporated to give a dark red liquid. The crude product was purification by silica gel chromatography eluted with n-hexane/ethyl acetate 5/1 (volume ratio) to give N-vinylindole as a pale yellow liquid (2.8 g, yield 73%).

Synthesis of allyl-1H-indole (**8k**)

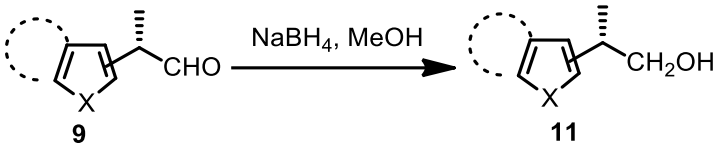
Allyl-1H-indole (8k): A mixture of indole (0.59 g, 5.0 mmol), allyl chloride (0.38 g, 5.0 mmol), NaOH (0.40 g, 10.0 mmol) in DMSO (10 mL) was vigorously stirred at room temperature under nitrogen atmosphere for 2.5 h. The reaction mixture was diluted with EtOAc (40 mL) and washed with H_2O (2×30 mL). The aqueous phase was extracted with EtOAc (2×30 mL), and the combined organic phase was dried over Na_2SO_4. After filtration and evaporation of the solvents in vacuo, the crude product was purified by column chromatography on silica gel (petroleum ether/EtOAc= 30/1) to give 8k (0.78 g, 99%) as a brown oil. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.67 (dd, J = 8.0 Hz, 4.0 Hz, 1H), 7.35 (dd, J = 8.0 Hz, 3.0 Hz, 1H), 7.25-7.22 (m, 1H), 7.16-7.12 (m, 2H), 6.55 (s, 1H), 6.06-5.98 (m, 1H), 5.24-5.21 (m, 1H), 5.13-5.09 (m, 1H), 4.76-4.75 (m, 2H).

4. General procedure for asymmetric hydroformylation

In a glovebox filled with nitrogen, to a 5 ml vial equipped with a magnetic bar was added ligand (*S,R*)- Bn-Yanphos (0.005 mmol) and $Rh(acac)(CO)_2$ (0.0025 mmol in 0.20 mL solvent). After stirring for 10 min, substrate (0.5 mmol) and additional solvent was charged to bring the total volume of the reaction mixture to 1.0 mL. The vial was transferred into an autoclave and taken out of the glovebox. Carbon monoxide (10 bar) and hydrogen (10 bar) were charged in sequence. The reaction mixture was stirred at 70 °C (oil bath) for 24 h. The reaction was cooled and the pressure was carefully released in a well-ventilated hood. The conversion and b/l were

determined by ^{1}H NMR spectroscopy from the crude reaction mixture. The enantiomeric excesses of **9a-9k** were determined by HPLC.

5. General procedure for reduction of aldehydes

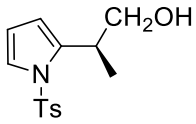


9 **11**

The crude mixture of **9** was treated with $NaBH_4$ (40 mg) in MeOH (2 mL) at 0°C. After stirring at 0°C for 2h, the reaction mixture was quenched with saturated aqueous H_2O (5 mL) and extracted 2 times with ethyl acetate (5 mL). The combined organic layers were washed with brine and dried over Na_2SO_4, the solvents were removed under reduced pressure, and the residue was purified by flash chromatography on silica gel (petroleum ether/ethyl acetate = 10:1, v/v) to afford the desired alcohols **11**.

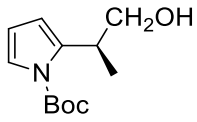
6. Characterizations of compounds

(*S*)-2-(1-tosyl-1*H*-pyrrol-2-yl)propan-1-ol (11a)



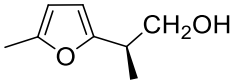
White solid, 114 mg, 82% yield; 94% *ee*; $[\alpha]_{25}D$ = 14.3 (c = 0.3, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, 2H), 7.33 (d, 2H), 6.29 (s, 1H), 6.15 (s, 1H), 3.67-3.63 (m, 1H), 3.59-3.55 (m, 1H), 3.49-3.40 (m, 1H) 2.43 (s, 3H), 1.15 (d, 3H). Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel AD-H, hexane/i-PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 32.2 min, t_{minor} = 40.2 min;

(*S*)-*tert*-butyl 2-(1-hydroxypropan-2-yl)-1*H*-pyrrole-1-carboxylate (11b)



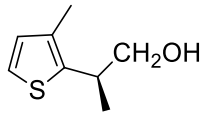
White solid, 90 mg, 80% yield, 91% *ee*, $[\alpha]^{25}_D$ = 3.3 (c = 0.3, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.24 (q, 1H), 6.16-6.11 (m, 2H), 3.82-3.67(m, 3H), 1.62 (s, 9H), 1.31 (d, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 149.7, 137.8, 121.6, 110.2, 110.1, 83.7, 67.7, 34.7, 28.0, 17.2. Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel AD-H, hexane/*i*-PrOH = 90:10, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 5.1 min, t_{minor} = 5.5 min; HRMS calculated $[M+H]^+$ for $C_{12}H_{20}NO_3$: 226.1437, found: 226.1435.

(*R*)-2-(5-methylfuran-2-yl)propan-1-ol (11c)



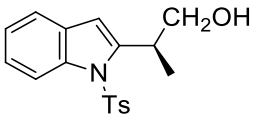
White solid, 54 mg, 78% yield, 94% *ee*, $[\alpha]^{25}_D$ = -3.3 (c = 0.3, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 5.99 (s, 1H), 5.91 (s, 1H), 3.74 (d, 2H), 3.06-2.97 (m, 1H), 2.29 (s, 3H), 1.28 (d, 3H). Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel AS-H, hexane/*i*-PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 7.2 min, t_{minor} = 7.6 min.

(*R*)-2-(3-methylthiophen-2-yl)propan-1-ol (11d)



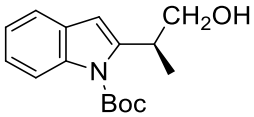
I White solid, 64 mg, 82% yield, 94% *ee*, $[\alpha]^{25}_D$ = -3.3 (c = 0.3, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.15 (d, 1H), 6.85 (d, 1H), 3.72-3.65 (m, 2H), 3.41-3.33 (m, 1H), 2.24 (s, 3H), 1.34 (d, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 140.6, 133.6, 130.1, 121.9, 68.9, 36.1, 18.9, 13.8. Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel AS-H, hexane/*i*-PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 7.2 min, t_{minor} = 8.2 min. HRMS calculated $[M+H]^+$ for $C_9H_{13}OS$: 157.0681, found: 157.0680.

(*S*)-2-(1-tosyl-1*H*-indol-2-yl)propan-1-ol (11e)



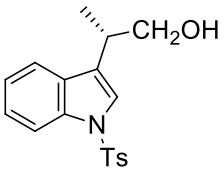
White solid, 130 mg, 79% yield, 95% *ee*, $[\alpha]^{25}_D$ = -98.7 (c = 0.3, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, 1H), 7.63 (d, 2H), 7.47 (d, 1H), 7.33-7.19 (m, 4H), 6.55 (s, 1H), 3.92-3.84 (m, 2H), 3.81-3.77 (q, 1H), 2.35 (s, 3H), 1.39 (d, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.8, 144.1, 137.4, 135.8, 129.8, 129.7, 126.1, 124.3, 123.7, 120.3, 115.3, 108.8, 67.7, 35.5, 21.6, 17.5. Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel AD-H, hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 13.8 min, t_{minor} = 29.3 min; HRMS calculated $[M+H]^+$ for $C_{18}H_{20}NO_3S$: 330.1158, found: 330.1152.

(*S*)-*tert*-butyl 2-(1-hydroxypropan-2-yl)-1*H*-indole-1-carboxylate (11f)



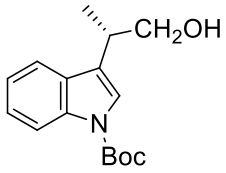
White solid, 110 mg, 80% yield, 95% *ee*, $[\alpha]^{25}_D$ = 1.0 (c = 0.3, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, 1H), 7.52 (d, 1H), 7.28-7.21 (m, 2H), 6.53 (s, 1H), 4.01-3.84 (m, 2H), 3.82 (q, 1H), 1.73 (s, 9H), 1.41 (d, 3H).Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel IB-H, hexane/*i*-PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{minor} = 6.9 min, t_{major} = 7.3 min.

(*S*)-2-(1-tosyl-1*H*-indol-3-yl)propan-1-ol (11g)



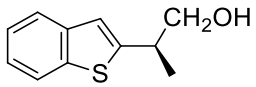
White solid, 123 mg, 75% yield, 93% *ee*, $[\alpha]^{25}_D$ = 14.7 (c = 0.3, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, 1H), 7.80 (d, 2H), 7.58 (d, 1H), 7.43 (s, 1H), 7.37 (t, 1H), 7.25 (m, 3H), 3.83-3.76 (m, 2H), 3.18-3.26 (m, 1H), 2.35 (s, 3H), 1.41 (d, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.9, 135.4, 135.1, 130.3, 129.8, 126.8, 124.9, 124.8, 123.1, 122.7, 119.7, 113.8, 67.2, 33.5, 21.6, 16.7. Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel IB -H, hexane/*i*-PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 22.7 min, t_{minor} = 25.3 min; HRMS calculated $[M+H]^+$ for $C_{18}H_{20}NO_3S$: 330.1158, found: 330.1152.

(*S*)-*tert*-butyl 3-(1-hydroxypropan-2-yl)-1*H*-indole-1-carboxylate (11h)



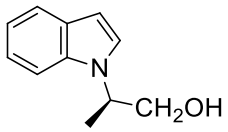
White solid, 105 mg, 76% yield, 96% *ee*, $[\alpha]^{25}_D$ = 1.0 (c = 0.3, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (s, 1H), 7.63 (d, 1H), 7.47 (s, 1H), 7.38-7.34 (t, 1H), 7.27-7.25 (m, 1H), 3.91-3.79 (m, 2H), 3.28 (m, 1H), 1.71 (s, 9H), 1.43 (d, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 149.7, 136.6, 129.9, 124.6, 124.5, 122.6, 122.4, 119.2, 115.4, 83.6, 67.3, 33.5, 28.2, 16.8. Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel IB-H, hexane/*i*-PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 7.4 min, t_{minor} = 8.2 min; HRMS calculated $[M+H]^+$ for $C_{16}H_{22}NO_3$:267.1594, found: 267.1591.

(*R*)-2-(benzo[*b*]thiophen-2-yl)propan-1-ol (11i)



White solid, 78 mg, 82% yield, 92% *ee*. $[\alpha]^{25}_D$ = -4.0 (c = 0.3, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, 1H), 7.75 (d, 1H), 7.38-7.32 (m, 2H), 7.16 (s, 1H), 3.82 (m, 2H), 3.37 (m, 1H), 1.46 (d, 3H). Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel AS-H, hexane/*i*-PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{minor} = 11.5 min, t_{major} = 12.4 min.

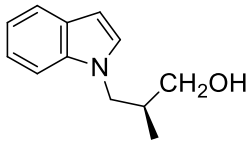
(*R*)-2-(1*H*-indol-1-yl)propan-1-ol (11j)



White solid, 76 mg, 87% yield, 95% *ee*, $[\alpha]^{25}_D$ = 47.0 (c = 0.3, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.69 (d, 1H), 7.46 (d, 1H), 7.27-7.23 (m, 2H), 7.17 (t, 1H), 6.60 (d, 1H), 4.74 (m, 1H), 3.92 (t, 2H), 1.60 (d, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 136.1, 128.5, 124.2, 121.6, 121.6, 121.1, 119.6, 109.5, 102.1, 66.5, 53.1, 16.9. Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel AS-H, hexane/*i*-PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 13.0 min,

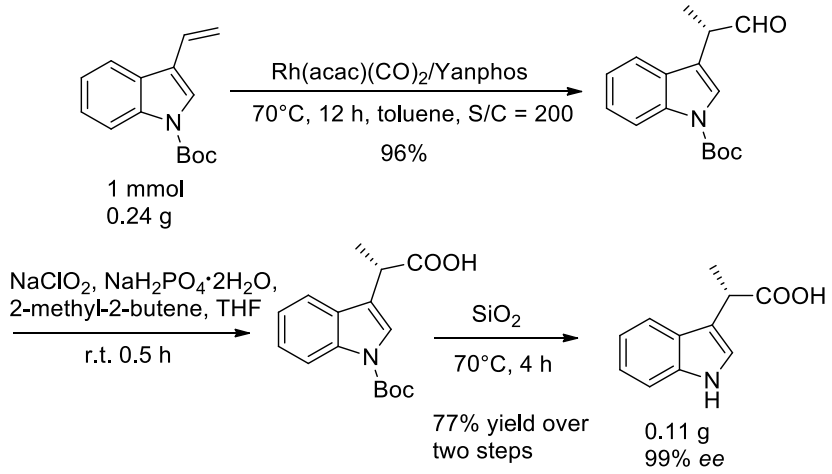
t_{minor} = 15.3 min; HRMS calculated $[M+H]^+$ for $C_{11}H_{14}NO$:176.1069, found: 176.1067.

(*S*)-3-(1*H*-indol-1-yl)-2-methylpropan-1-ol (11k)



White solid, 63 mg, 67% yield, 90% *ee*, $[\alpha]^{25}_D$ = 2.0 (c = 0.3, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, 1H), 7.43 (d, 1H), 7.26 (t, 1H), 7.15-7.11 (m, 2H), 6.54 (d, 1H), 4.31 (q, 1H), 4.06 (q, 1H), 3.54 (d, 2H), 2.34 (m, 1H), 1.01 (d, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 136.2, 128.6, 128.4, 121.4, 120.9, 119.2, 109.5, 101.1, 65.0, 48.9, 36.7, 14.9. Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel OD-H, hexane/*i*-PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{minor} = 26.6 min, t_{major} = 32.9 min; HRMS calculated $[M+H]^+$ for $C_{12}H_{16}NO$:190.1226, found: 190.1223.

7. Procedures for the preparation of (*S*)- α-Methyl-3-indolylacetic acid



In a glovebox filled with nitrogen, to a 5 ml vial equipped with a magnetic bar was added ligand (*S,R*)- Bn-Yanphos (0.01 mmol in 1 mL solvent) and $Rh(acac)(CO)_2$ (0.005 mmol in 0.25 mL solvent) After stirring for 10 min, substrate **8h** (1 mmol) and additional solvent was charged to bring the total volume of the reaction mixture to 2 mL. The vial was transferred into an autoclave and taken out of the glovebox. Carbon

monoxide (10 bar) and hydrogen (10 bar) were charged in sequence. The reaction mixture was stirred at 70 °C (oil bath) for 12 h. The reaction was cooled and the pressure was carefully released in a well-ventilated hood. The reaction mixture was transferred into a 20 mL Schlenk tube, then *t*-BuOH (10 mL), 2-methyl-2-butene (2.0 M in THF, 5.5 mL, 11 mmol), and NaH_2PO_4 (276 mg, 1.77 mmol) in H_2O (2.0 mL) were added. The mixture was cooled to 0°C, then NaOClO (994 mg, 11.0 mmol) in 2 mL of H_2O was added. After being stirred for 30 min at room temperature. The reaction mixture was poured into saturated aq NH_4Cl (5 mL), and whole was extracted with EtOAc (5 mL).The combined organic layers were washed with brine (5 mL) and dried over anhydrous Na_2SO_4. Filtration and evaporation in vacuo furnished the crude product, the crude product was used directly in next step.

The crude product was transferred into a 50 mL bottom flask, then EtOAc (10 mL), silica gel (300 mg) were added, evaporation in vacuo, then the reaction mixture was stirred at 70 °C in vacuo (oil bath) for 4 h. The crude product was purified by column chromatography (silica gel, 1:1 hexane/EtOAc) to provide **12** (0.11 g, 77%) as a dark yellow oil: {$[\alpha]^{25}_D$ = +37.0 (c = 0.1, $CHCl_3$) for 99% ee} was the same as that reported in the literature {lit.7 $[\alpha]^{25}_D$ = +41.5 (c = 0.13, CH_2Cl_2) for >99% ee}.^{1}H NMR (400 MHz, $CDCl_3$) : 1.63 (d, *J* = 7.3 Hz, 3H, CH_3CHAr), 4.05 (q, *J* = 7.3 Hz, 1H, CH_3CHAr), 7.13 (dd, *J* = 7.3 Hz, 8.0 Hz, 1H, Ar), 7.15 (s, 1H, Ar), 7.20 (dd, *J* = 7.3 Hz, 7.3 Hz, 1H, Ar), 7.36 (d, *J* = 8.0 Hz, 1H, Ar), 7.69 (d, *J* = 7.6 Hz, 1H, Ar), 8.07 (br s, 1H, NH). Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel IC, hexane/*i*-PrOH = 90:10, flow rate = 0.5 mL/min, λ = 220 nm, 40℃, t_{major} = 18.3 min, t_{minor} = 26.7 min

8. References

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[2] Y. Uozumi, A. Tanahashi, S.Y. Lee, T. Hayashi, *J. Org. Chem.* 1993, **58**, 1945.

[3] Y. Uozumi, A. Tanahashi, S.Y. Lee, T. Hayashi, *J. Org. Chem.* 1993, **58**, 1945.

[4] P. N. M. Botman, O. David, A. Amore, J. Dinkelaar, M. T. Vlaar, K. Goubitz, J. Fraanje, H. Schenk, H. Hiemstra, J. H. van Maarseveen, *Angew. Chem. Int. Ed*. 2004, **43**, 3471 –3473.

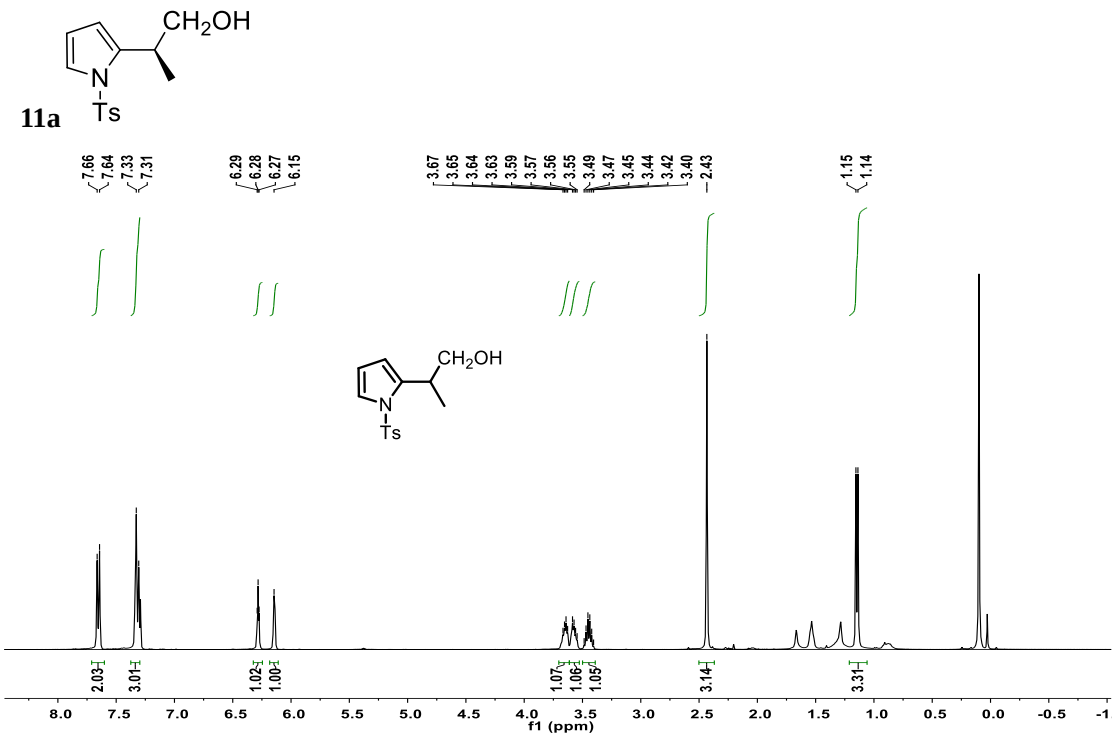
[5] Y. Yan, X. Zhang, *J. Am. Chem. Soc*. 2006**, 128,** 7198-7202.

[6] T. Mitsudome, A. Noujima, Y. Mikami, T. Mizugaki, K. Jitsukawa, K. Kaneda, *Chem. Eur. J*. 2010, **16**, 11818.

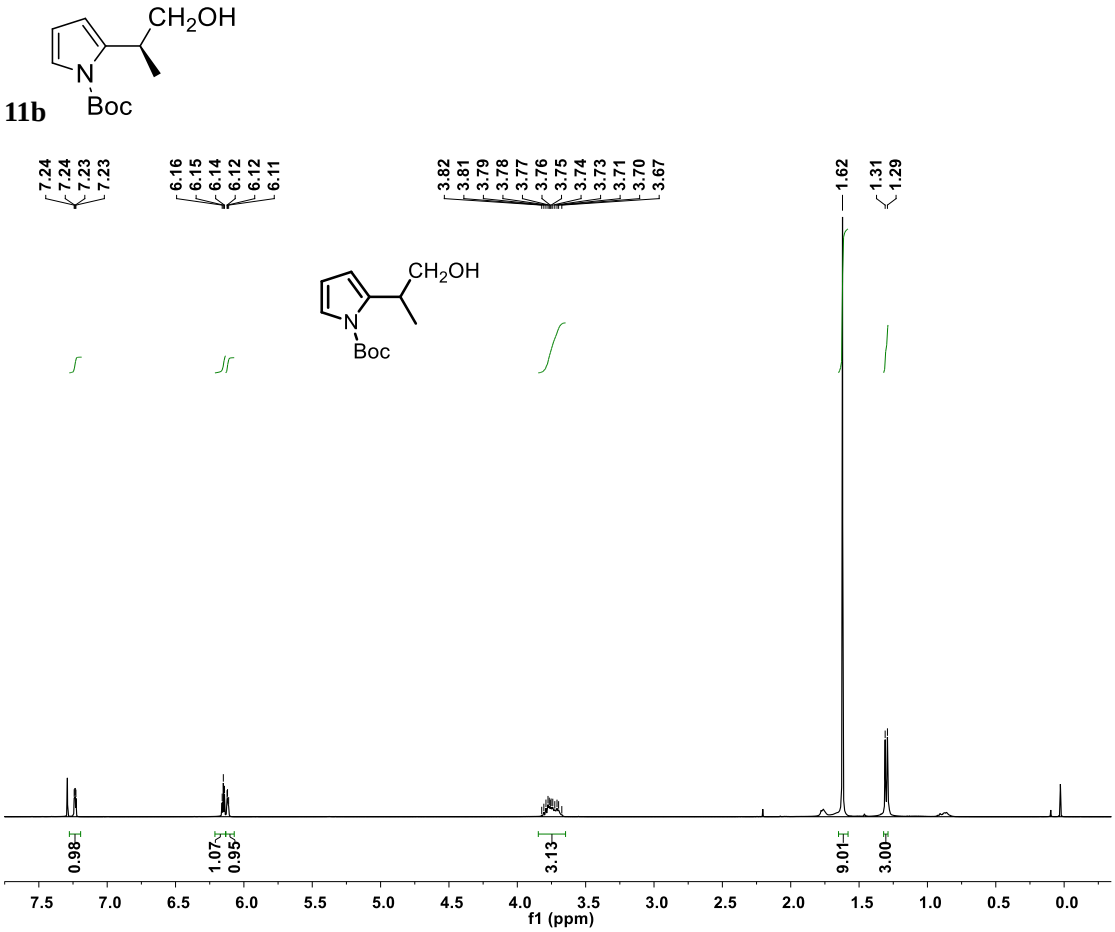
[7] M. Richter, W. Whitefield, J. Maimone, W. Lin, P. Castroviejo, S. Baran, *J. Am. Chem. Soc*. 2007, **129**, 12857–12869.

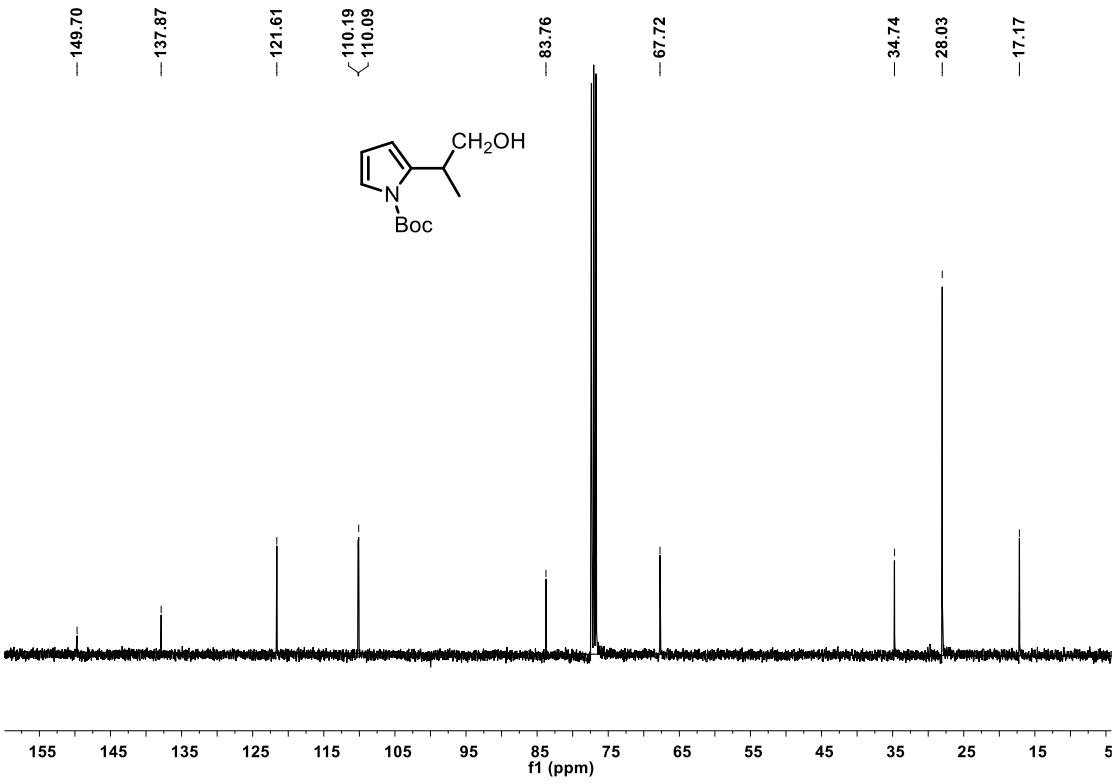
9. NMR spectra of compound

11a

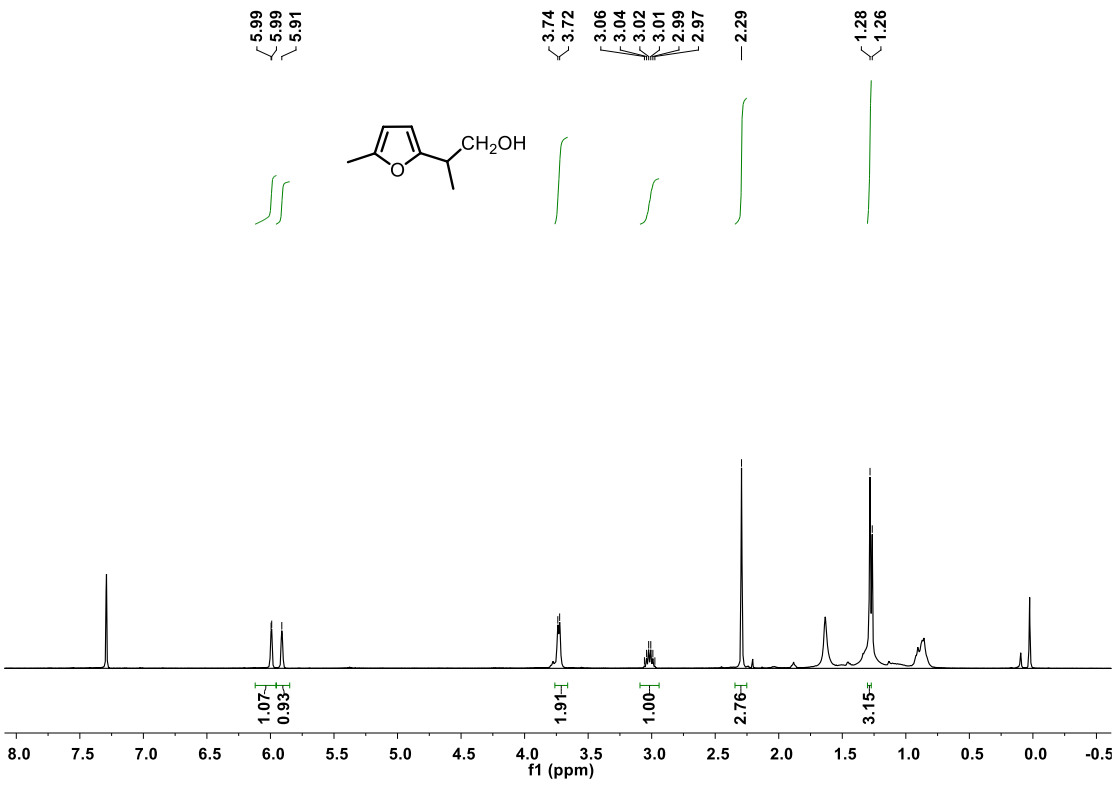


11b


7.24
7.24
7.23
7.23
6.16
6.15
6.14
6.12
6.12
6.11
3.82
3.81
3.79
3.78
3.77
3.76
3.75
3.74
3.73
3.71
3.70
3.67
1.62
1.31
1.29
CH2OH
N
Boc
0.98
1.07
0.95
3.13
9.01
3.00
f1 (ppm)


149.70
137.87
121.61
110.19
110.09
83.76
67.72
34.74
28.03
17.17
CH2OH
N
Boc
f1 (ppm)

11c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5.99
5.91
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3.72
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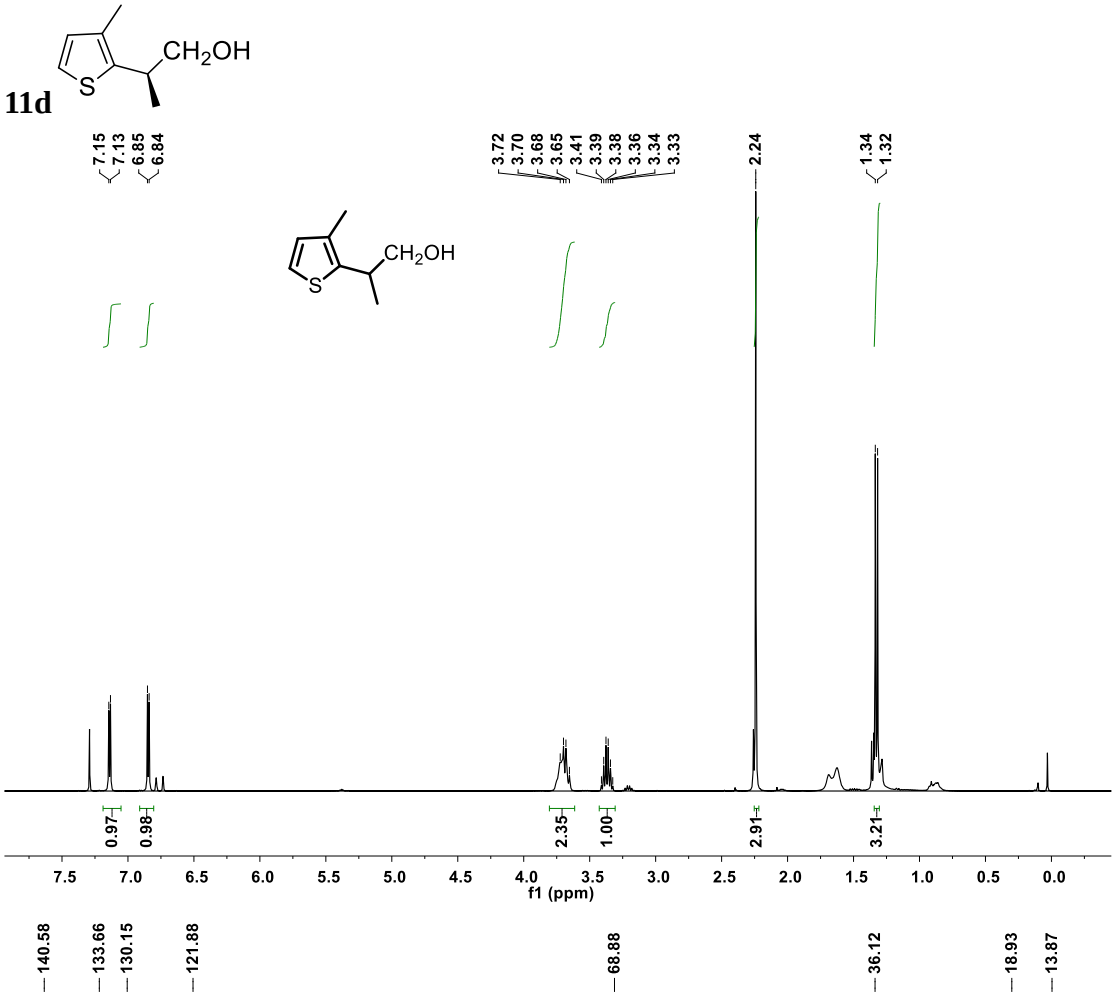
3.04
3.02
3.01
2.99
2.97
2.29
1.28
1.26
CH2OH
1.07
0.93
1.91
1.00
2.76
3.15
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

11d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7.13
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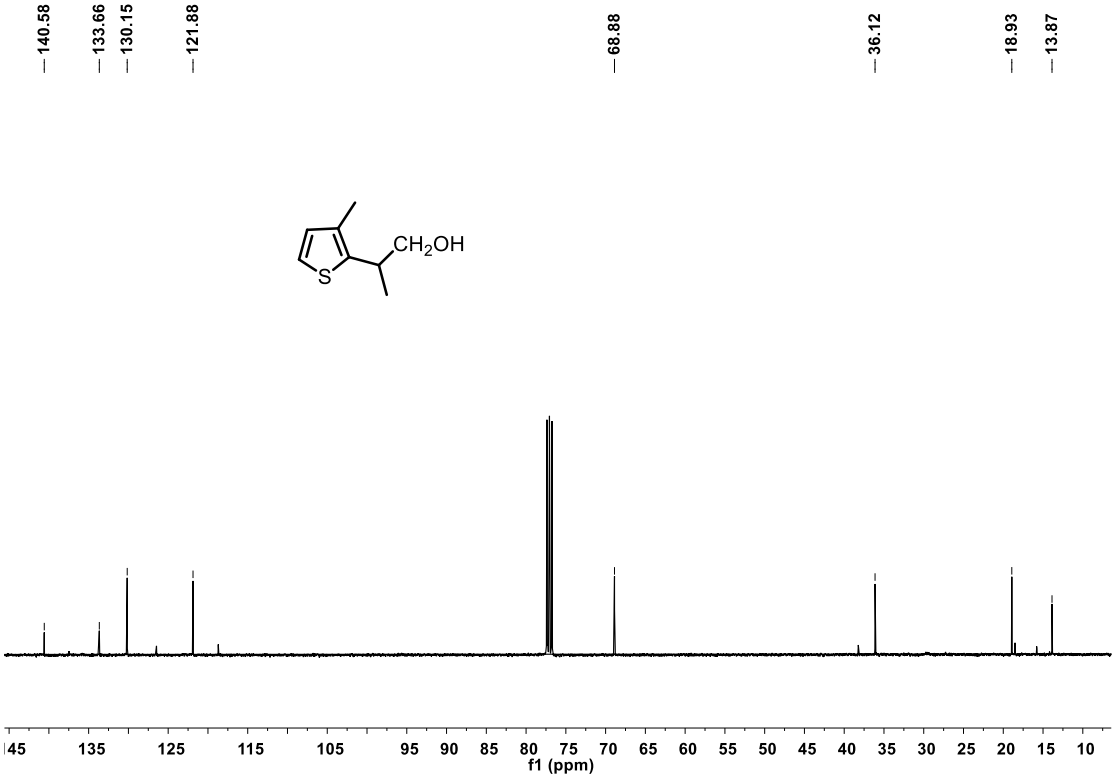
6.84
3.72
3.70
3.68
3.65
3.41
3.39
3.38
3.36
3.34
3.33
2.24
1.34
1.32
CH2OH
S
0.97
0.98
2.35
1.00
2.91
3.21
f1 (ppm)


140.58
133.66
130.15
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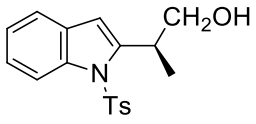
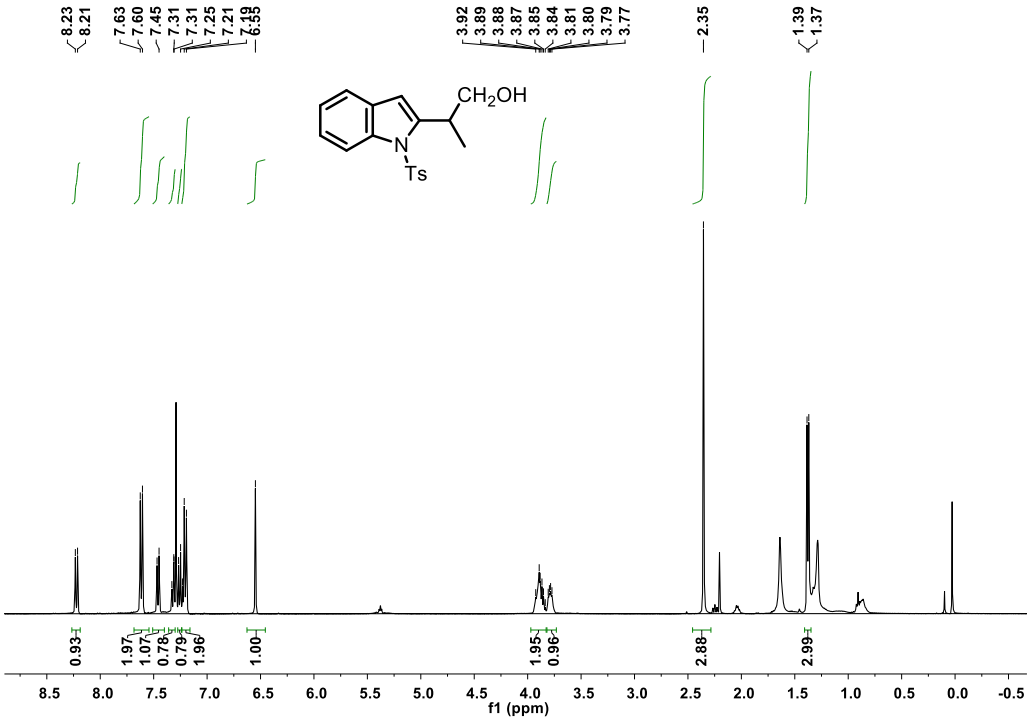
68.88
36.12
18.93
13.87
CH2OH
S
f1 (ppm)


CH2OH
N
Ts

11e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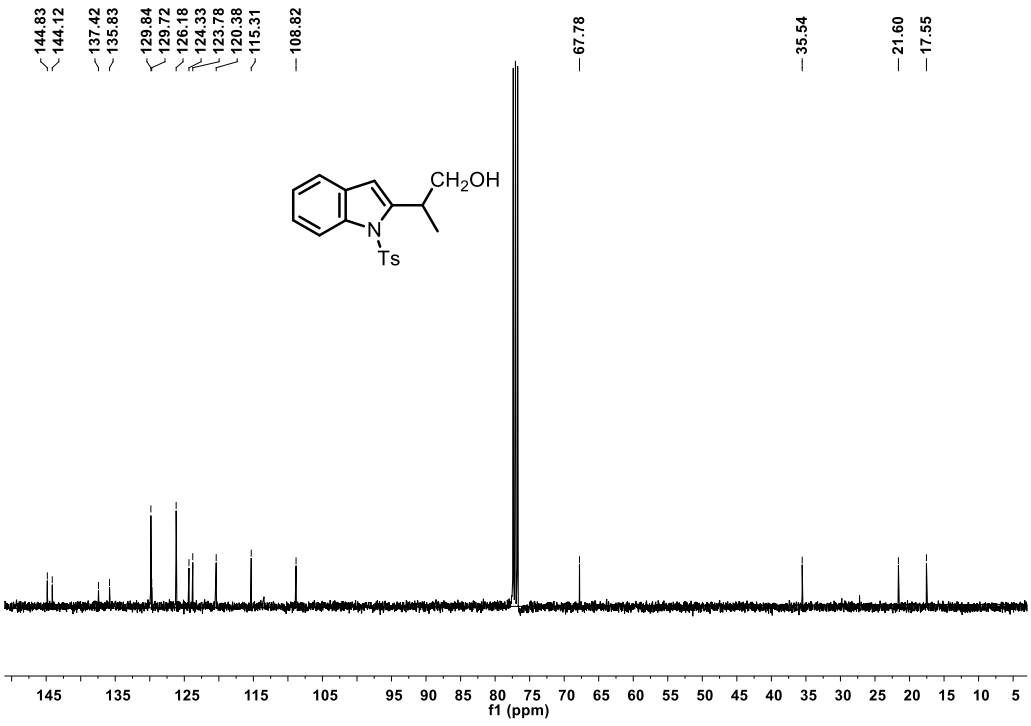
7.60
7.45
7.31
7.31
7.25
7.21
7.19
6.35
3.92
3.89
3.88
3.87
3.85
3.84
3.81
3.80
3.79
3.77
2.35
1.39
1.37
CH2OH
N
Ts
0.93
1.97
1.07
0.78
0.79
1.96
1.00
1.95
0.96
2.88
2.99
f1 (ppm)


144.83
144.12
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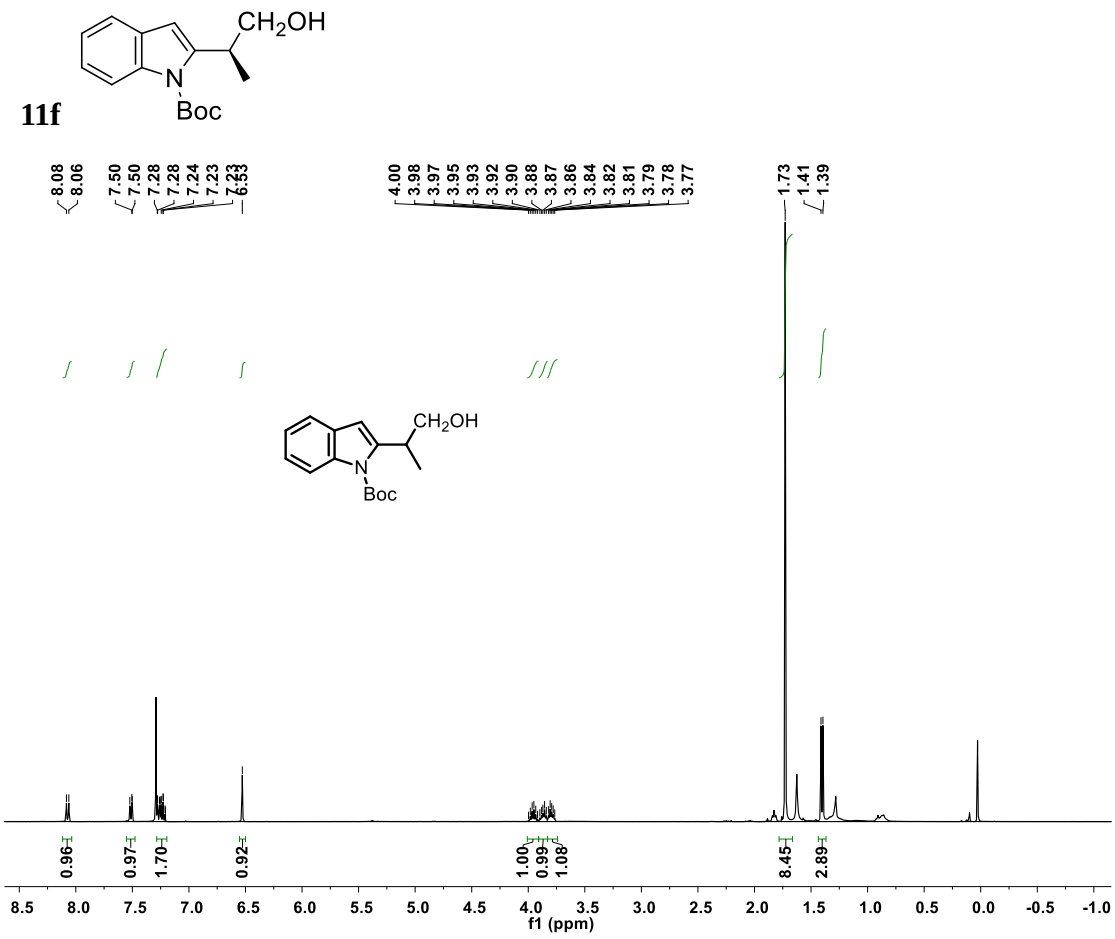
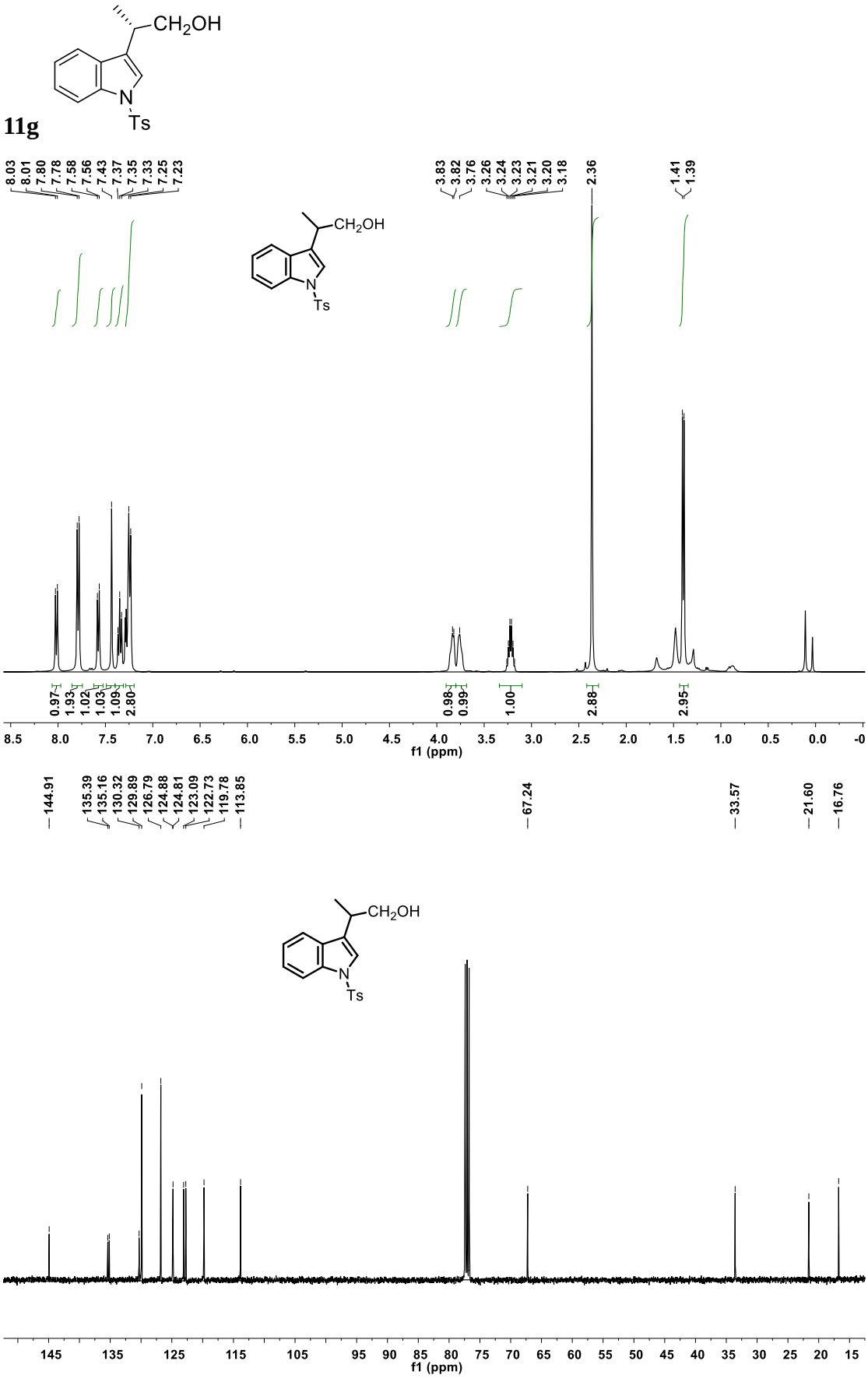
135.83
129.84
129.72
126.18
124.33
123.78
120.38
115.31
108.82
67.78
35.54
21.60
17.55
CH2OH
N
Ts
f1 (ppm)

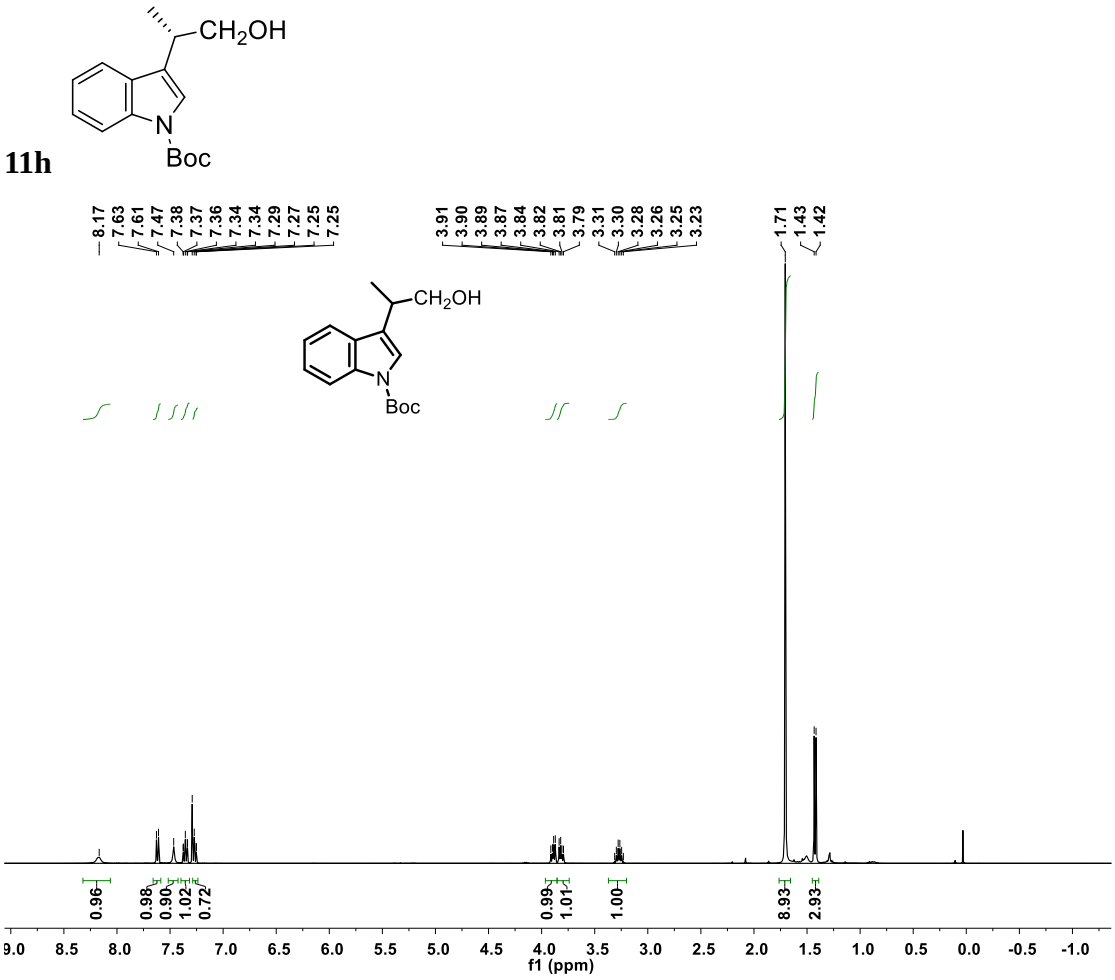
11f


8.08
8.06
7.50
7.50
7.28
7.28
7.24
7.23
6.53
4.00
3.98
3.97
3.95
3.93
3.92
3.90
3.88
3.87
3.86
3.84
3.82
3.81
3.79
3.78
3.77
1.73
1.41
1.39
CH2OH
N
Boc
0.96
0.97
1.70
0.92
1.00
0.99
1.08
8.45
2.89
f1 (ppm)

11g


CH2OH
N
Ts
8.03
8.01
7.80
7.78
7.58
7.56
7.43
7.37
7.35
7.33
7.25
7.23
3.83
3.82
3.76
3.26
3.24
3.23
3.21
3.20
3.18
2.36
1.41
1.39
0.97
1.93
1.02
1.03
1.09
2.80
0.98
0.99
1.00
2.88
2.95
f1 (ppm)
144.91
135.39
135.16
130.32
129.89
126.79
124.88
124.81
123.09
122.73
119.78
113.85
67.24
33.57
21.60
16.76
f1 (ppm)

11h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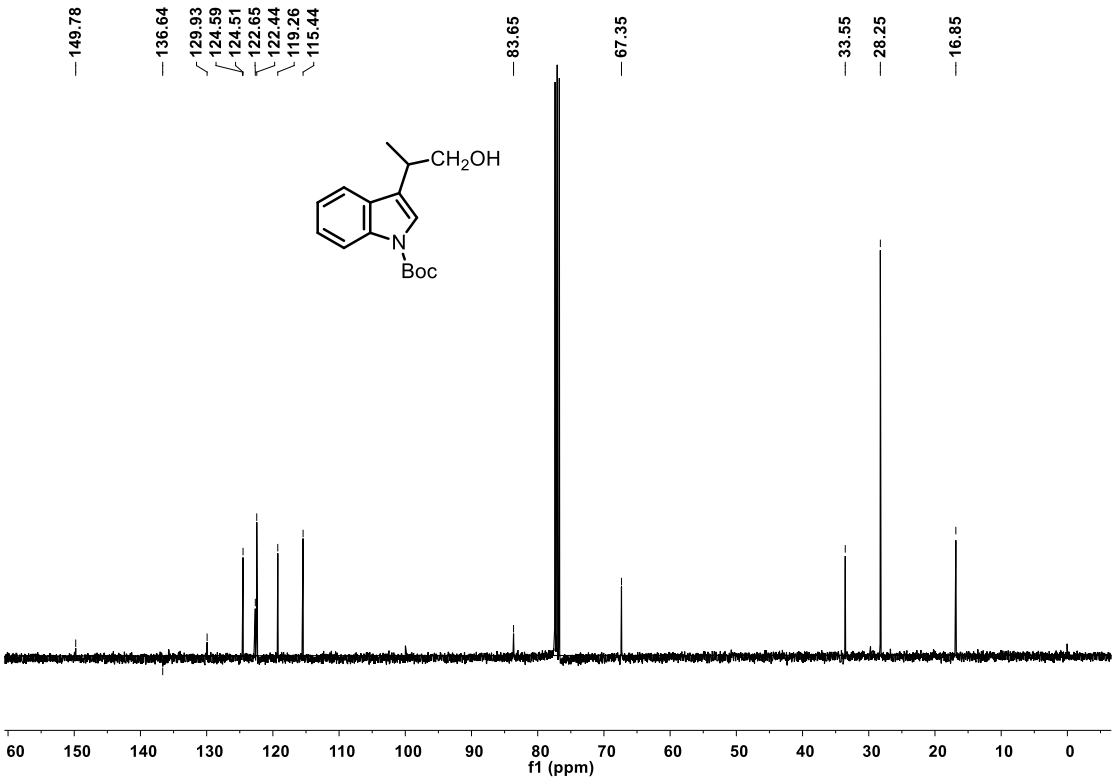
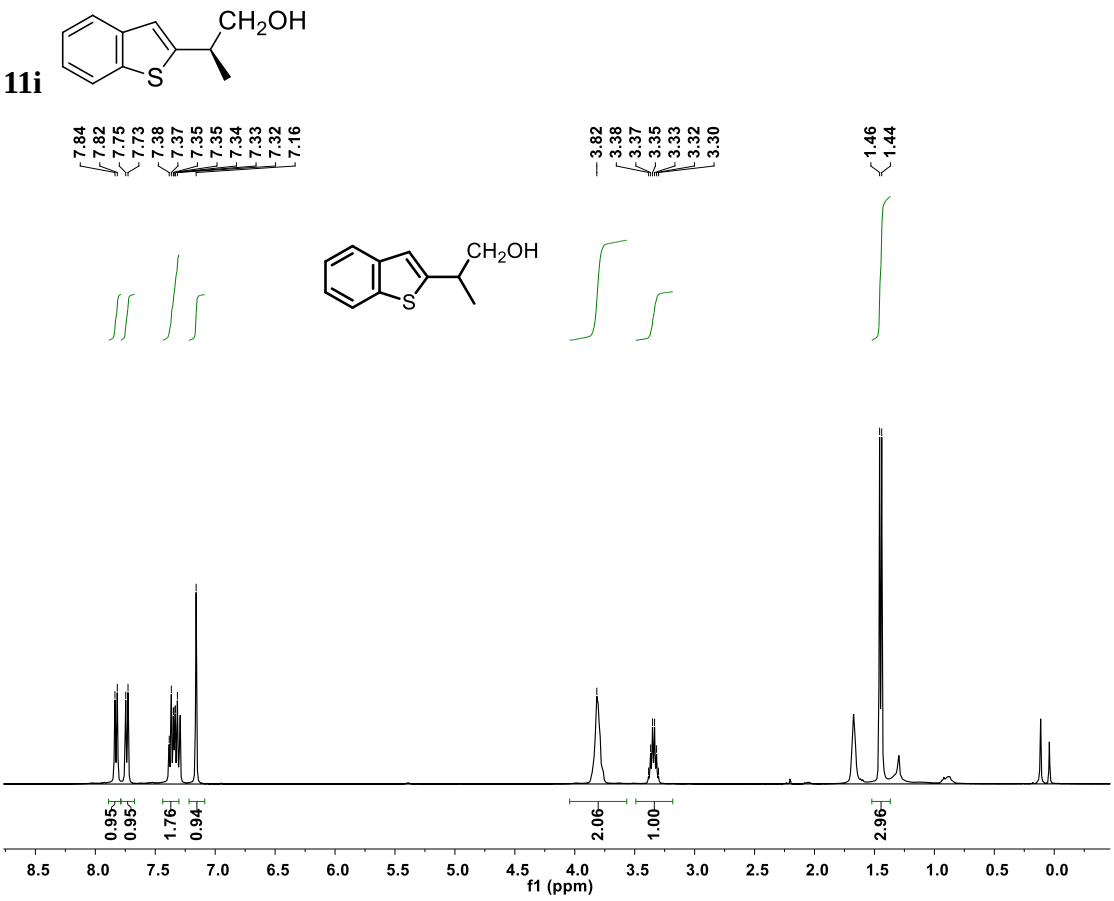
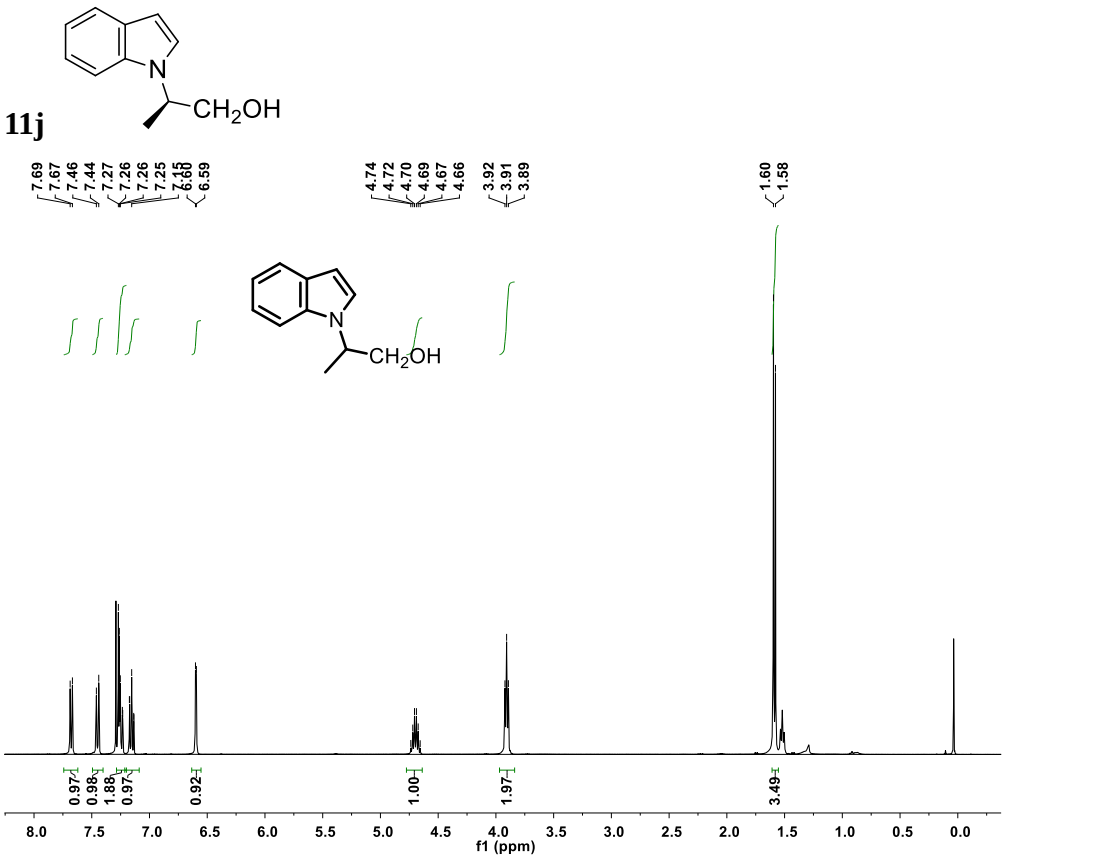
7.63
7.61
7.47
7.38
7.37
7.36
7.34
7.34
7.29
7.27
7.25
7.25
3.91
3.90
3.89
3.87
3.84
3.82
3.81
3.79
3.31
3.30
3.28
3.26
3.25
3.23
1.71
1.43
1.42
CH2OH
N
Boc
0.96
0.98
0.90
1.02
0.72
0.99
1.01
1.00
8.93
2.93
f1 (ppm)

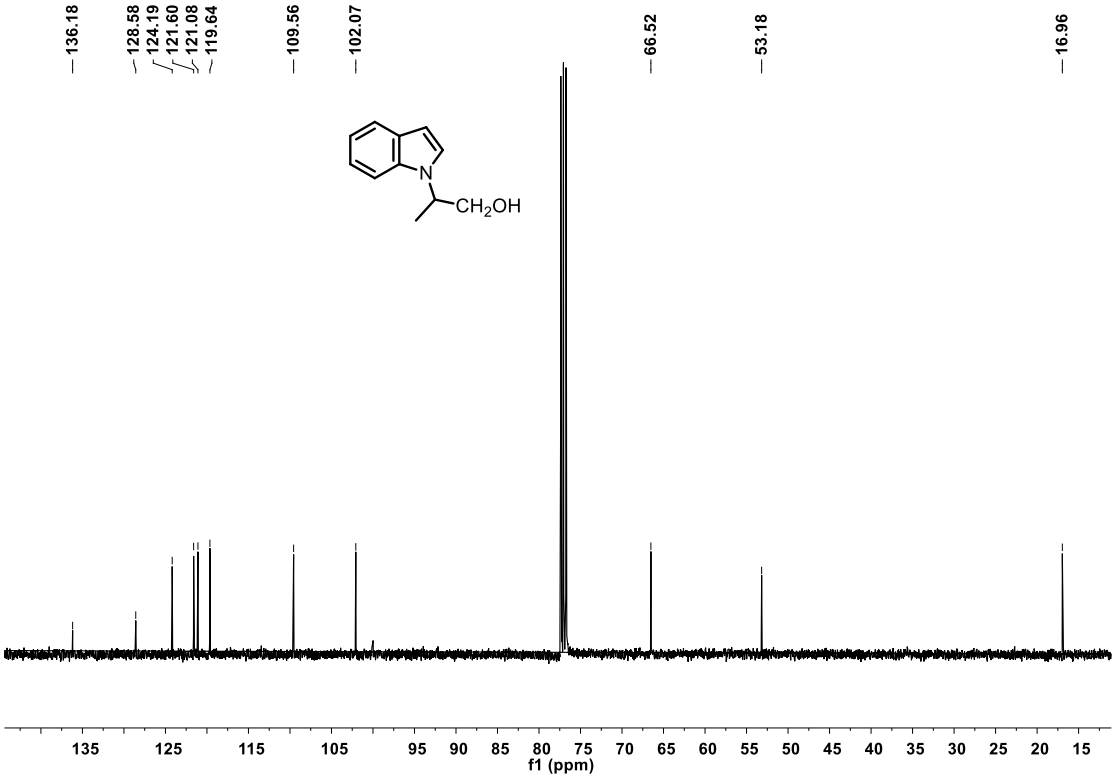

149.78
136.64
129.93
124.59
124.51
122.65
122.44
119.26
115.44
83.65
67.35
33.55
28.25
16.85
CH2OH
N
Boc
f1 (ppm)

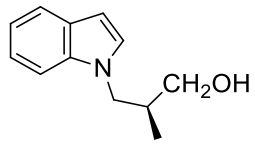
11i


7.84
7.82
7.75
7.73
7.38
7.37
7.35
7.35
7.34
7.33
7.32
7.16
3.82
3.38
3.37
3.35
3.33
3.32
3.30
1.46
1.44
CH2OH
S
0.95
0.95
1.76
0.94
2.06
1.00
2.96
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

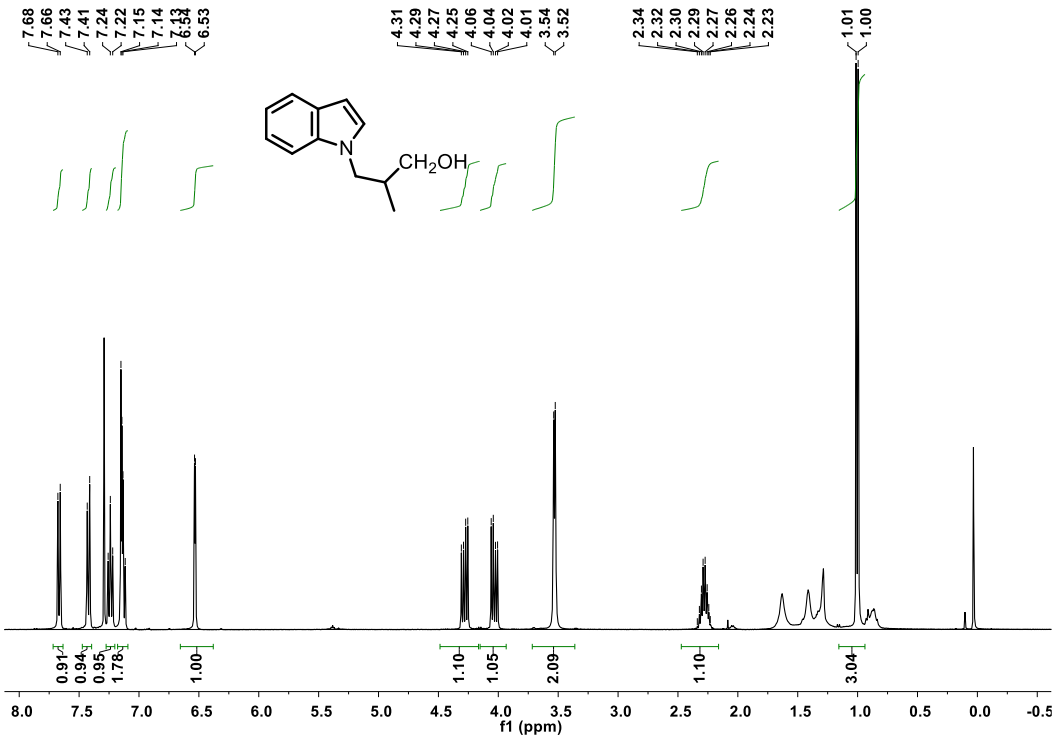
11j

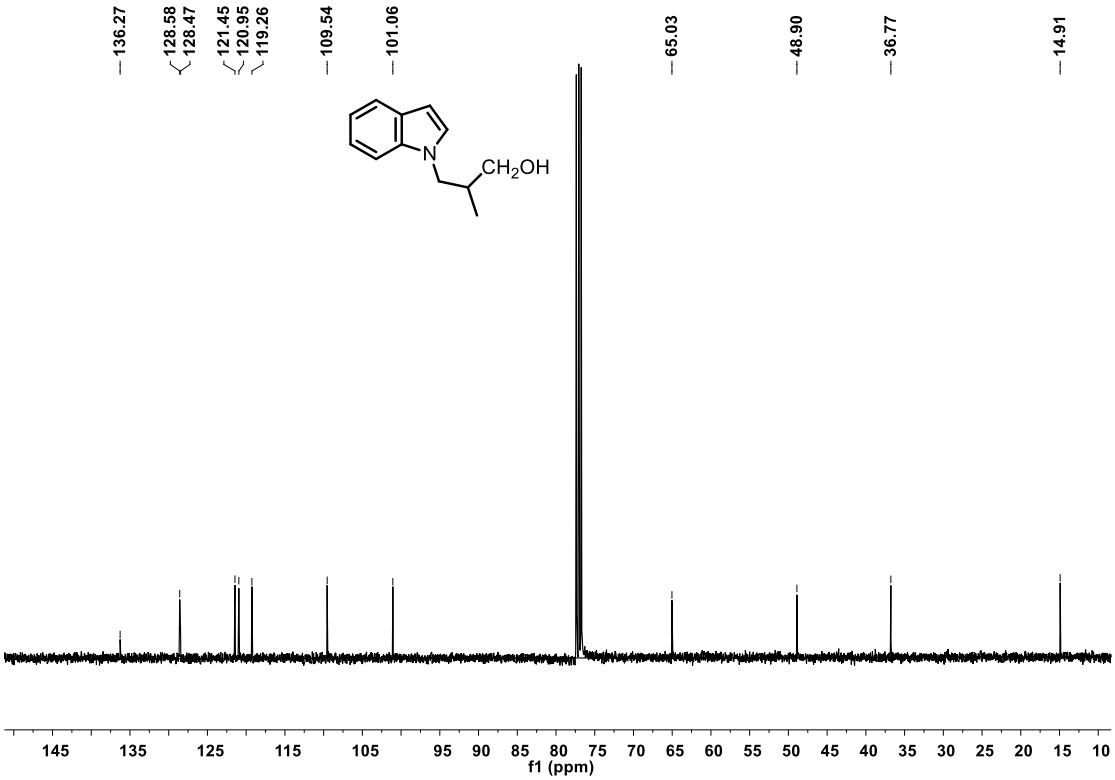

7.69
7.67
7.46
7.44
7.27
7.26
7.26
7.25
7.15
6.60
6.59
4.74
4.72
4.70
4.69
4.67
4.66
3.92
3.91
3.89
1.60
1.58
0.97
0.98
1.88
0.97
0.92
1.00
1.97
3.49
f1 (ppm)


136.18
128.58
124.19
121.60
121.08
119.64
109.56
102.07
66.52
53.18
16.96
f1 (ppm)

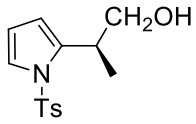

N
CH2OH

11k


7.68
7.66
7.43
7.41
7.24
7.22
7.15
7.14
6.54
6.53
4.31
4.29
4.27
4.25
4.06
4.04
4.02
4.01
3.54
3.52
2.34
2.32
2.30
2.29
2.27
2.26
2.24
2.23
1.01
1.00
N
CH2OH
0.91
0.94
0.95
1.78
1.00
1.10
1.05
2.09
1.10
3.04
f1 (ppm)


136.27
128.58
128.47
121.45
120.95
119.26
109.54
101.06
65.03
48.90
36.77
14.91
N
CH2OH
f1 (ppm)

10. HPLC spectra for ee determination



Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel AD-H, hexane/*i*PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 32.2 min, t_{minor} = 40.2 min

```
Data File E:\DATA\WB\WB-20150912\VWD-AD-95-5-WB-20150912 2015-09-12 18-01-03\011-0201.D
Sample Name: weib-20150912-1

    ================================================================
    Acq. Operator    : SYSTEM                        Seq. Line :   2
    Acq. Instrument  : 1260HPLC-VWD                   Location : Vial 11
    Injection Date   : 9/12/2015 6:12:38 PM               Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method      : E:\DATA\WB\WB-20150912\VWD-AD-95-5-WB-20150912 2015-09-12 18-01-03\VWD-
                       ADH(1-2)-95-5-1ML-220NM-50MIN.M
    Last changed     : 9/12/2015 6:01:03 PM by SYSTEM
    Analysis Method  : E:\DATA\WB\WB-20150912\VWD-AD-95-5-WB-20150912 2015-09-12 18-01-03\VWD-
                       ADH(1-2)-95-5-1ML-220NM-50MIN.M (Sequence Method)
    Last changed     : 3/27/2016 2:18:06 PM by SYSTEM
                       (modified after loading)
    Additional Info  : Peak(s) manually integrated
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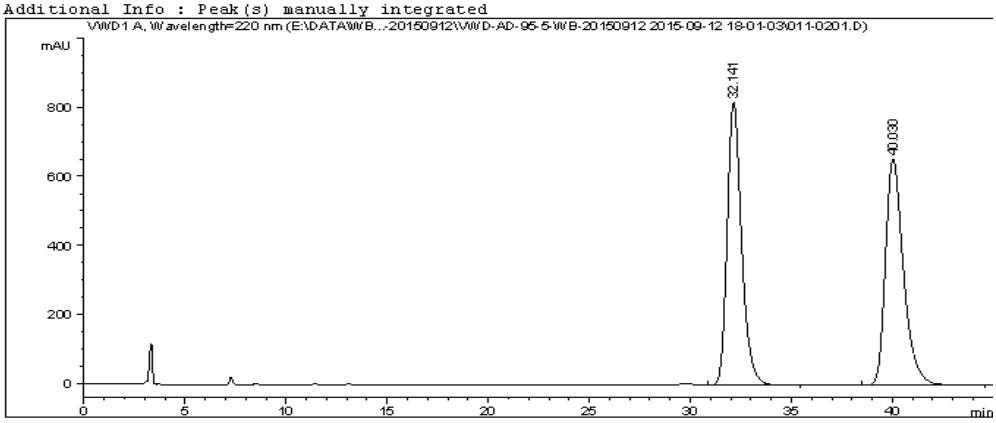



```
    ================================================================
                            Area Percent Report
    ================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  32.141 BB    0.7547 4.04313e4   819.35242  49.9533
       2  40.030 BB    0.9375 4.05069e4   654.07281  50.0467

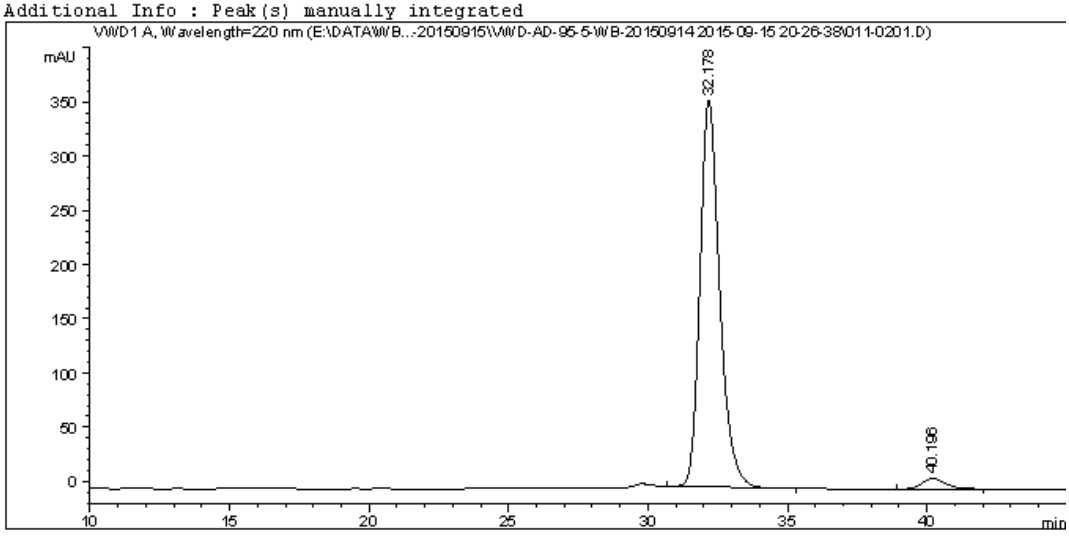
    Totals :                  8.09382e4  1473.42523


    ================================================================
                            *** End of Report ***


1260HPLC-DAD 3/27/2016 2:18:12 PM SYSTEM                    Page 1 of 1
```

Data File E:\DATA\WB\WB-20150915\VWD-AD-95-5-WB-20150914 2015-09-15 20-26-38\011-0201.D
Sample Name: weib-20150915-1

```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                    Location : Vial 11
Injection Date  : 9/15/2015 8:38:14 PM                 Inj :   1
                                                 Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WB\WB-20150915\VWD-AD-95-5-WB-20150914 2015-09-15 20-26-38\VWD-
                  ADH(1-2)-95-5-1ML-220NM-50MIN.M
Last changed    : 9/15/2015 8:26:39 PM by SYSTEM
Analysis Method : E:\DATA\WB\WB-20150915\VWD-AD-95-5-WB-20150914 2015-09-15 20-26-38\VWD-
                  ADH(1-2)-95-5-1ML-220NM-50MIN.M (Sequence Method)
Last changed    : 3/27/2016 2:20:07 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                          Area Percent Report
==========================================================================

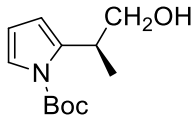
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  32.178 BB    0.7444 1.74604e4   355.87524  96.8814
   2  40.196 BB    0.8831  562.05292    9.52958   3.1186

Totals :                  1.80225e4   365.40483

==========================================================================
                          *** End of Report ***
```



Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel AD-H, hexane/*i*PrOH = 90:10, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 5.1 min, t_{minor} = 5.5 min

```
Data File E:\DATA\WB\WB-20160303\VWD-AD-90-10-WB-20160303 2016-03-03 08-45-25\049-0201.D
Sample Name: weib-20160303-1

    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 49
    Injection Date  : 3/3/2016 8:57:05 AM                   Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20160303\VWD-AD-90-10-WB-20160303 2016-03-03 08-45-25\VWD-
                      ADH(1-6)-90-10-1ML-220NM-50MIN.M
    Last changed    : 3/3/2016 8:45:25 AM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20160303\VWD-AD-90-10-WB-20160303 2016-03-03 08-45-25\VWD-
                      ADH(1-6)-90-10-1ML-220NM-50MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:00:52 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
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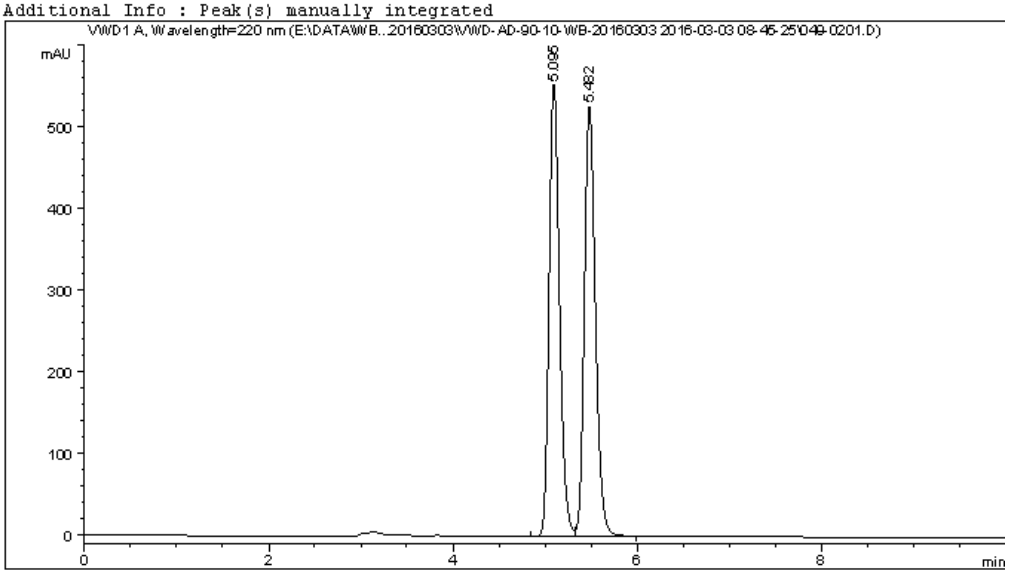
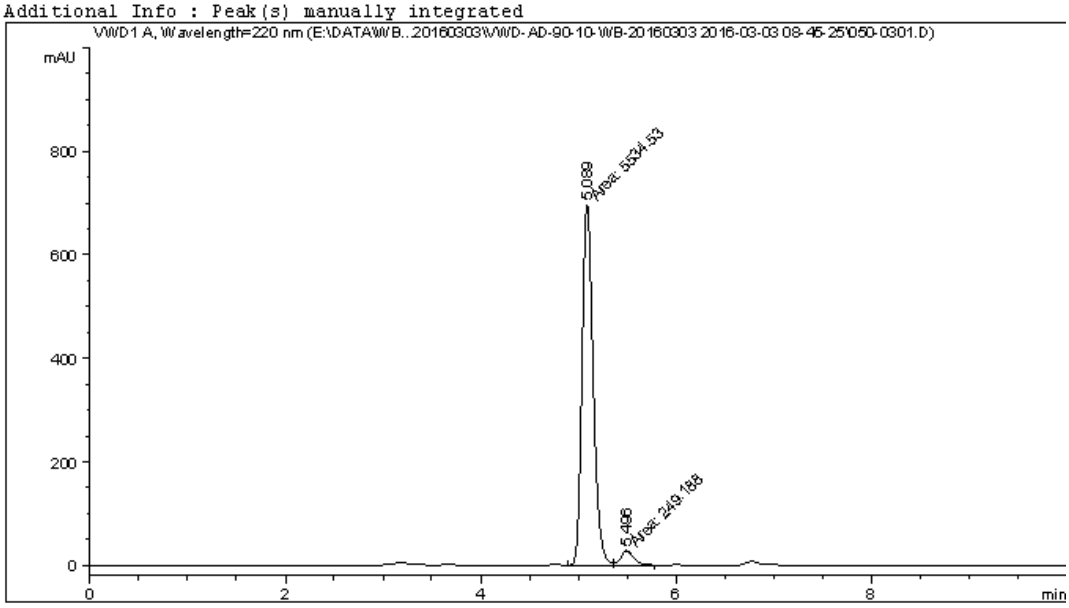



```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   5.095 VV    0.1210 4373.16553  554.48450  49.9110
       2   5.482 VB    0.1274 4388.75342  525.80615  50.0890

    Totals :                   8761.91895 1080.29065


    ======================================================================
                              *** End of Report ***

1260HPLC-DAD 3/27/2016 2:01:05 PM SYSTEM                       Page 1 of 1
```

Data File E:\DATA\WB\WB-20160303\VWD-AD-90-10-WB-20160303 2016-03-03 08-45-25\050-0301.D
Sample Name: weib-20160303-1

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   3
    Acq. Instrument : 1260HPLC-VWD                    Location : Vial 50
    Injection Date  : 3/3/2016 9:47:52 AM                 Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20160303\VWD-AD-90-10-WB-20160303 2016-03-03 08-45-25\VWD-
                      ADH(1-6)-90-10-1ML-220NM-50MIN.M
    Last changed    : 3/3/2016 10:08:01 AM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\WB\WB-20160303\VWD-AD-90-10-WB-20160303 2016-03-03 08-45-25\VWD-
                      ADH(1-6)-90-10-1ML-220NM-50MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:04:11 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   5.089 MF    0.1324 5534.52783  696.87225  95.6916
       2   5.496 FM    0.1478  249.18770   28.09358   4.3084

    Totals :                  5783.71553  724.96583

    ======================================================================
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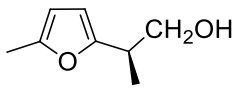

Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel AS-H, hexane/*i*PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 7.2 min, t_{minor} = 7.6 min

```
Data File E:\DATA\WB\WB-20151216-4\VWD-AD-95-5-WB-20151216S 2015-12-16 18-34-07\096-0501.D
Sample Name: weib-20151216-za-5

    ======================================================================
    Acq. Operator   : SYSTEM                       Seq. Line :   5
    Acq. Instrument : 1260HPLC-VWD                  Location : Vial 96
    Injection Date  : 12/16/2015 8:48:00 PM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20151216-4\VWD-AD-95-5-WB-20151216S 2015-12-16 18-34-07
                      \VWD-AS(1-6)-95-5-1ML-5U-220NM-40MIN.M
    Last changed    : 12/16/2015 6:34:08 PM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20151216-4\VWD-AD-95-5-WB-20151216S 2015-12-16 18-34-07
                      \VWD-AS(1-6)-95-5-1ML-5U-220NM-40MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:33:13 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
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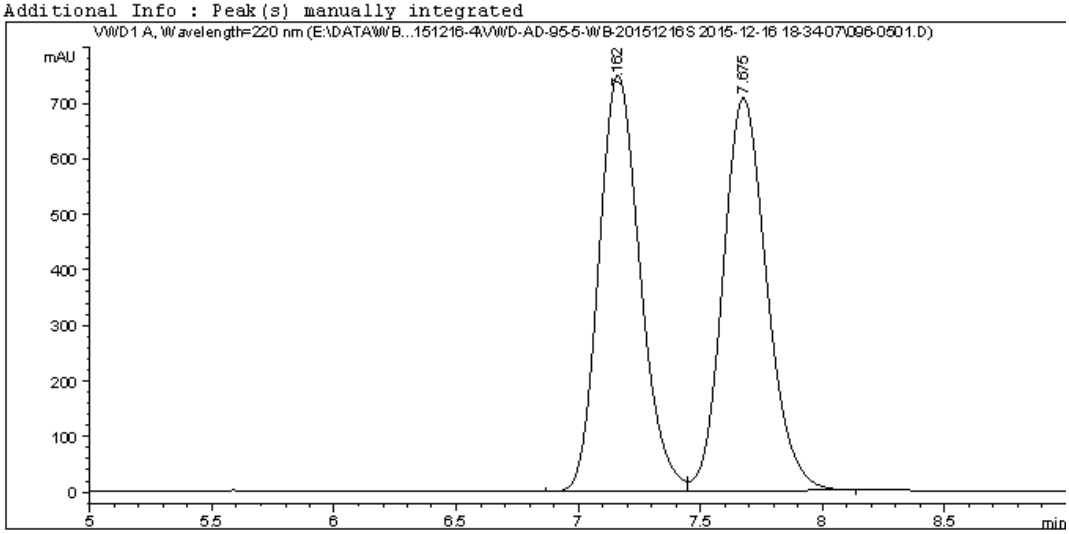
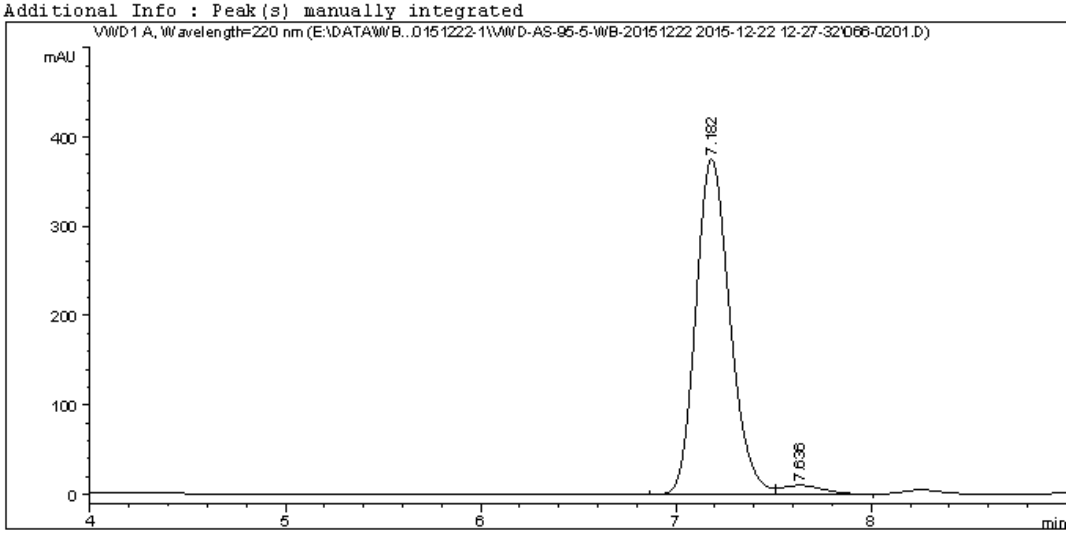


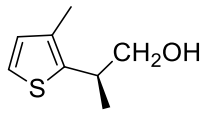

```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   7.162 BV    0.1813 8739.21191  748.31024  49.8280
       2   7.675 VB    0.1933 8799.54492  706.72589  50.1720

    Totals :                   1.75388e4  1455.03613

    ======================================================================
                            *** End of Report ***


1260HPLC-DAD 3/27/2016 2:33:23 PM SYSTEM                    Page 1 of 1
```

Data File E:\DATA\WB\WB-20151222-1\VWD-AS-95-5-WB-20151222 2015-12-22 12-27-32\066-0201.D
Sample Name: weib-20151222-6

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 66
    Injection Date  : 12/22/2015 12:40:02 PM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20151222-1\VWD-AS-95-5-WB-20151222 2015-12-22 12-27-32\VWD
                      -AS(1-6)-95-5-1ML-5U-220NM-15MIN.M
    Last changed    : 12/22/2015 12:27:32 PM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20151222-1\VWD-AS-95-5-WB-20151222 2015-12-22 12-27-32\VWD
                      -AS(1-6)-95-5-1ML-5U-220NM-15MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:35:05 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

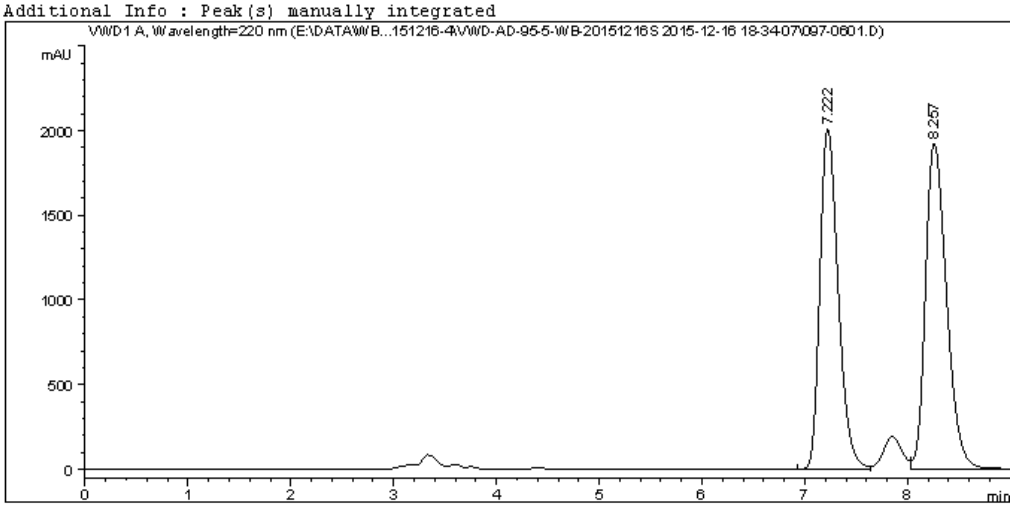



```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   7.182 BV    0.1873 4541.74219  374.91458  96.9673
       2   7.636 VV    0.2204  142.04477    9.83837   3.0327

    Totals :                   4683.78696  384.75295


    ======================================================================
                            *** End of Report ***
```



Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel AS-H, hexane/*i*PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 7.2 min, t_{minor} = 8.2 min

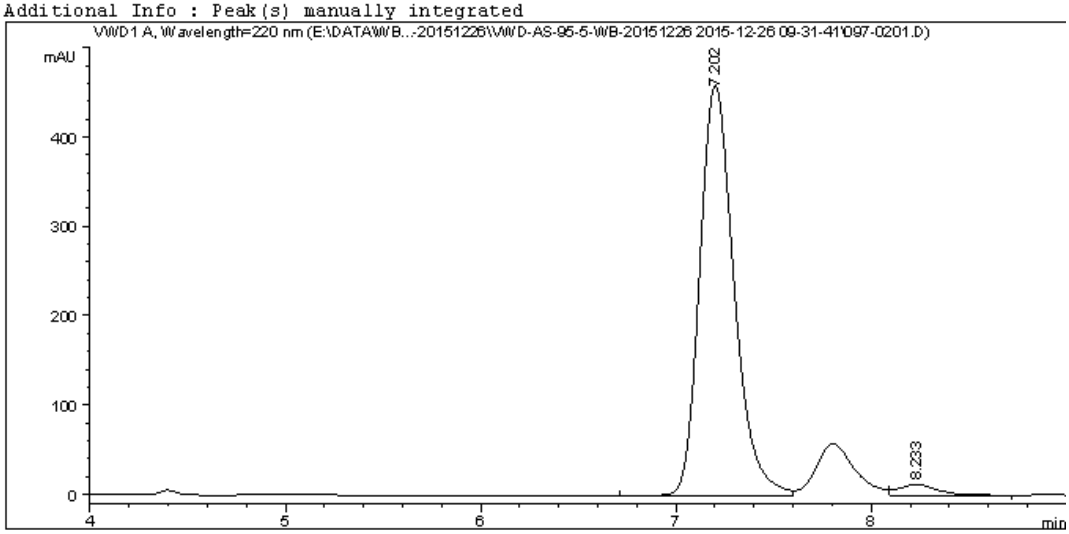
```
Data File E:\DATA\WB\WB-20151216-4\VWD-AD-95-5-WB-20151216S 2015-12-16 18-34-07\097-0601.D
Sample Name: weib-20151216-za-6

    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   6
    Acq. Instrument : 1260HPLC-VWD                    Location : Vial 97
    Injection Date  : 12/16/2015 9:28:46 PM              Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20151216-4\VWD-AD-95-5-WB-20151216S 2015-12-16 18-34-07
                      \VWD-AS(1-6)-95-5-1ML-5U-220NM-40MIN.M
    Last changed    : 12/16/2015 6:34:08 PM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20151216-4\VWD-AD-95-5-WB-20151216S 2015-12-16 18-34-07
                      \VWD-AS(1-6)-95-5-1ML-5U-220NM-40MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:12:48 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



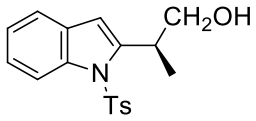

```
    ======================================================================
                           Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   7.222 BV    0.1920 2.47679e4  2006.74084  47.6247
       2   8.257 VB    0.2204 2.72385e4  1921.49268  52.3753

    Totals :                   5.20065e4  3928.23352


    ======================================================================
                           *** End of Report ***


1260HPLC-DAD 3/27/2016 2:13:05 PM SYSTEM                    Page 1 of 1
```

Data File E:\DATA\WB\WB-20151226\VWD-AS-95-5-WB-20151226 2015-12-26 09-31-41\097-0201.D
Sample Name: weib-20151226-1

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                  Location : Vial 97
    Injection Date  : 12/26/2015 9:43:45 AM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20151226\VWD-AS-95-5-WB-20151226 2015-12-26 09-31-41\VWD-
                      AS(1-6)-95-5-1ML-5U-220NM-15MIN.M
    Last changed    : 12/26/2015 9:31:41 AM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20151226\VWD-AS-95-5-WB-20151226 2015-12-26 09-31-41\VWD-
                      AS(1-6)-95-5-1ML-5U-220NM-15MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:15:06 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

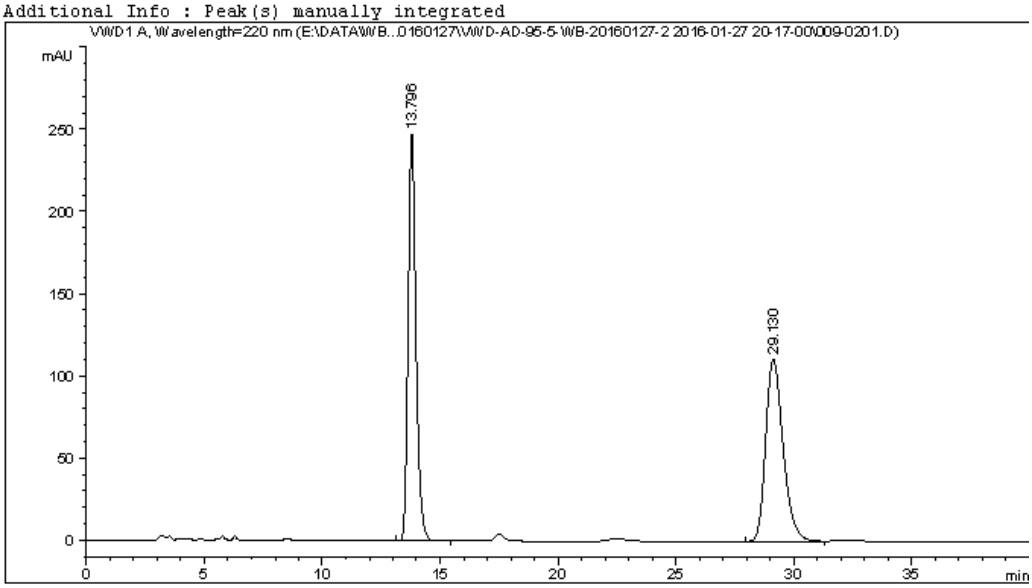



```
    ======================================================================
                           Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   7.202 BV    0.1872 5591.02051  458.65384  97.1526
       2   8.233 VB    0.2087  163.86201   11.82037   2.8474

    Totals :                   5754.88252  470.47421


    ======================================================================
                           *** End of Report ***
```



Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel AD-H, hexane/*i*PrOH = 80:20, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 13.8 min, t_{minor} = 29.3 min

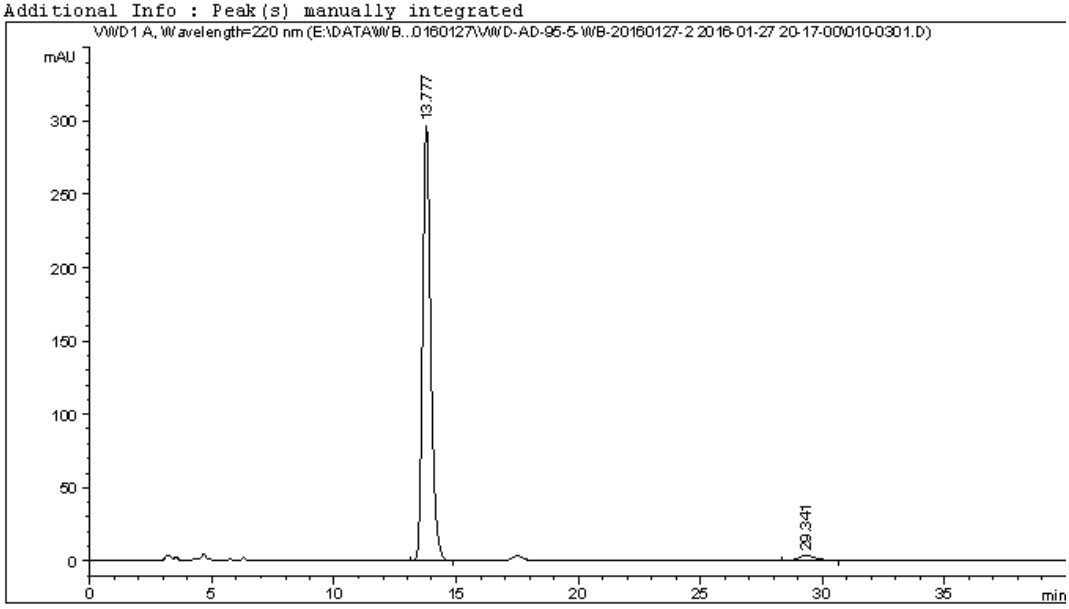
```
Data File E:\DATA\WB\WB-20160127\VWD-AD-95-5-WB-20160127-2 2016-01-27 20-17-00\009-0201.D
Sample Name: weib-20160127-9

    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 9
    Injection Date  : 1/27/2016 8:28:35 PM               Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20160127\VWD-AD-95-5-WB-20160127-2 2016-01-27 20-17-00\VWD
                      -AD(1-6)-80-20-1ML-5U-220NM-40MIN.M
    Last changed    : 1/27/2016 8:17:00 PM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20160127\VWD-AD-95-5-WB-20160127-2 2016-01-27 20-17-00\VWD
                      -AD(1-6)-80-20-1ML-5U-220NM-40MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:42:38 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



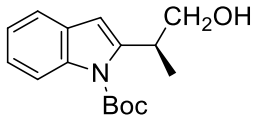

```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]       [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  13.796 BB    0.3530 5748.76221  247.45482  50.2644
       2  29.130 BB    0.7869 5688.29297  110.98508  49.7356

    Totals :                  1.1437le4   358.43990


    ======================================================================
                            *** End of Report ***

1260HPLC-DAD 3/27/2016 2:42:48 PM SYSTEM                    Page 1 of 1
```

Data File E:\DATA\WB\WB-20160127\VWD-AD-95-5-WB-20160127-2 2016-01-27 20-17-00\010-0301.D
Sample Name: weib-20160127-10

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260HPLC-VWD                  Location : Vial 10
    Injection Date  : 1/27/2016 9:09:24 PM             Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20160127\VWD-AD-95-5-WB-20160127-2 2016-01-27 20-17-00\VWD
                      -AD(1-6)-80-20-1ML-5U-220NM-40MIN.M
    Last changed    : 1/27/2016 8:17:00 PM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20160127\VWD-AD-95-5-WB-20160127-2 2016-01-27 20-17-00\VWD
                      -AD(1-6)-80-20-1ML-5U-220NM-40MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:44:25 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

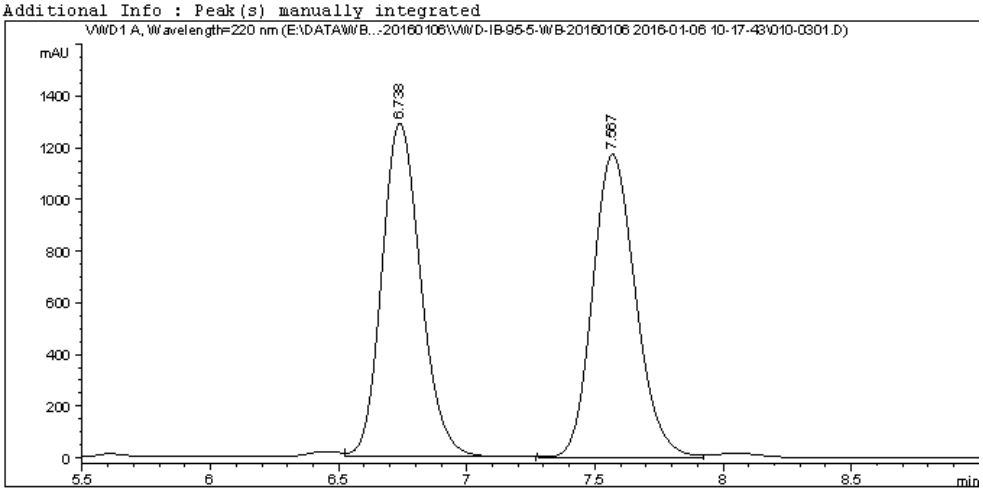



```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  13.777 BB    0.3542 6893.48730  296.52197  97.5910
       2  29.341 BB    0.7313  170.16338    3.45696   2.4090

    Totals :                  7063.65068  299.97893

    ======================================================================
                              *** End of Report ***
```



Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel IB-H, hexane/*i*PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{minor} = 6.9 min, t_{major} = 7.3 min

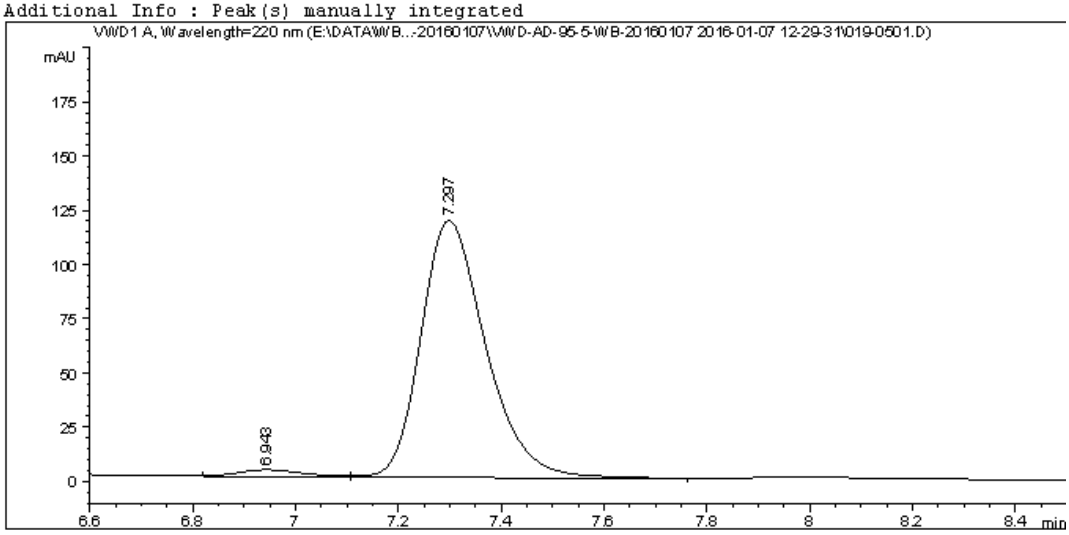
```
Data File E:\DATA\WB\WB-20160106\VWD-IB-95-5-WB-20160106 2016-01-06 10-17-43\010-0301.D
Sample Name: weib-20160106-2

    ================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 10
    Injection Date  : 1/6/2016 11:10:40 AM               Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20160106\VWD-IB-95-5-WB-20160106 2016-01-06 10-17-43\VWD-
                      IB(1-6)-95-5-1ML-220NM-40MIN.M
    Last changed    : 1/6/2016 10:17:44 AM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20160106\VWD-IB-95-5-WB-20160106 2016-01-06 10-17-43\VWD-
                      IB(1-6)-95-5-1ML-220NM-40MIN.M (Sequence Method)
    Last changed    : 3/27/2016 1:47:44 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



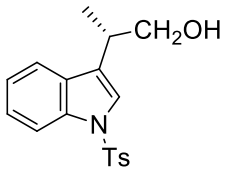

```
    ================================================================
                          Area Percent Report
    ================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   6.738 VB    0.1641 1.36225e4  1290.94263  49.2567
       2   7.567 BV    0.1804 1.36274e4  1173.98914  49.2743
       3  17.822 BB    0.3877   59.50492    2.33989   0.2152
       4  19.271 BB    0.5792  182.17918    4.87208   0.6587
       5  21.465 BB    0.3794   54.21317    2.16345   0.1960
       6  26.824 BB    0.6550  110.35899    2.45699   0.3990

    Totals :                  2.76562e4  2476.76417


    ================================================================

1260HPLC-DAD 3/27/2016 1:47:52 PM SYSTEM                    Page 1 of 1
```

Data File E:\DATA\WB\WB-20160107\VWD-AD-95-5-WB-20160107 2016-01-07 12-29-31\019-0501.D
Sample Name: weib-20160107-4

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   5
    Acq. Instrument : 1260HPLC-VWD                    Location : Vial 19
    Injection Date  : 1/7/2016 2:14:11 PM                 Inj :   1
                                                     Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20160107\VWD-AD-95-5-WB-20160107 2016-01-07 12-29-31\VWD-
                      IB(1-6)-95-5-1ML-220NM-40MIN.M
    Last changed    : 1/7/2016 12:29:32 PM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20160107\VWD-AD-95-5-WB-20160107 2016-01-07 12-29-31\VWD-
                      IB(1-6)-95-5-1ML-220NM-40MIN.M (Sequence Method)
    Last changed    : 3/27/2016 1:42:26 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

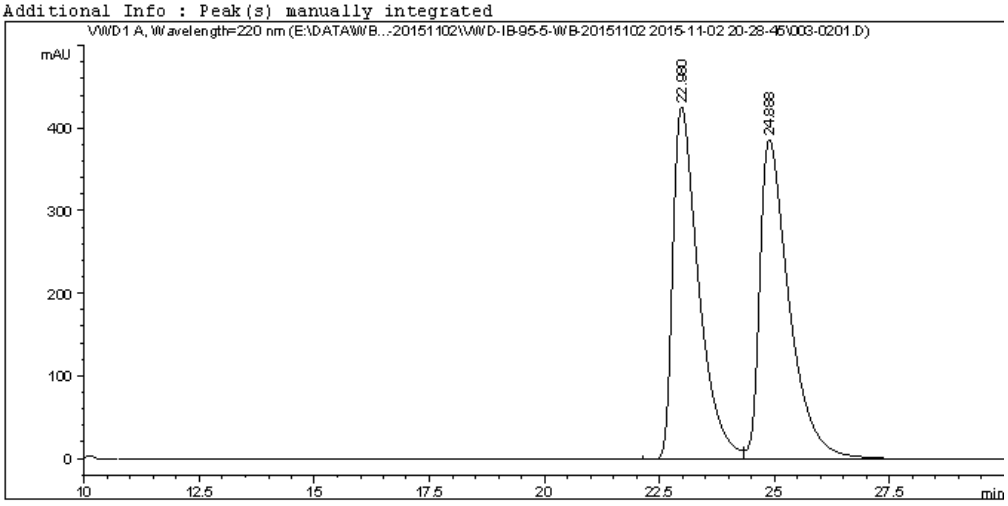



```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   6.943 VV    0.1311   26.34415    3.06968   2.4710
       2   7.297 VB    0.1343 1039.78674  118.53698  97.5290

    Totals :                   1066.13090  121.60666


    ======================================================================
                            *** End of Report ***
```



Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel IB -H, hexane/*i*PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 22.7 min, t_{minor} = 25.3 min

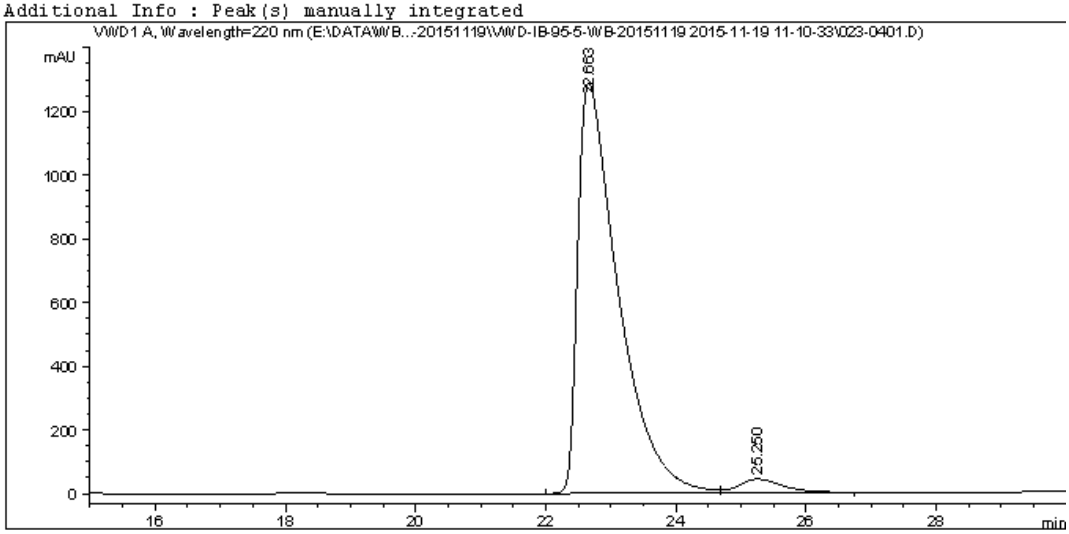
```
Data File E:\DATA\WB\WB-20151102\VWD-IB-95-5-WB-20151102 2015-11-02 20-28-45\003-0201.D
Sample Name: weib-20151102-3

    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 3
    Injection Date  : 11/2/2015 8:40:18 PM                  Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20151102\VWD-IB-95-5-WB-20151102 2015-11-02 20-28-45\VWD-
                      IB(1-6)-95-5-1ML-220NM-40MIN.M
    Last changed    : 11/2/2015 8:28:45 PM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20151102\VWD-IB-95-5-WB-20151102 2015-11-02 20-28-45\VWD-
                      IB(1-6)-95-5-1ML-220NM-40MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:30:49 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



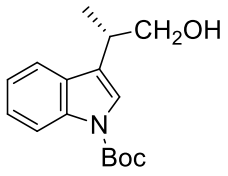

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  22.980 BV    0.5831 1.66978e4   427.14056  49.0558
       2  24.888 VB    0.6662 1.73406e4   387.38220  50.9442

    Totals :                  3.40385e4   814.52277


    ======================================================================
                              *** End of Report ***

1260HPLC-DAD 3/27/2016 2:30:56 PM SYSTEM                    Page 1 of 1
```

Data File E:\DATA\WB\WEIB-20151119\VWD-IB-95-5-WB-20151119 2015-11-19 11-10-33\023-0401.D
Sample Name: WEIB-20151119-3

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   4
    Acq. Instrument : 1260HPLC-VWD                  Location : Vial 23
    Injection Date  : 11/19/2015 12:43:54 PM             Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WEIB-20151119\VWD-IB-95-5-WB-20151119 2015-11-19 11-10-33\VWD
                      -IB(1-6)-95-5-1ML-220NM-40MIN.M
    Last changed    : 11/19/2015 11:10:33 AM by SYSTEM
    Analysis Method : E:\DATA\WB\WEIB-20151119\VWD-IB-95-5-WB-20151119 2015-11-19 11-10-33\VWD
                      -IB(1-6)-95-5-1ML-220NM-40MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:28:54 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

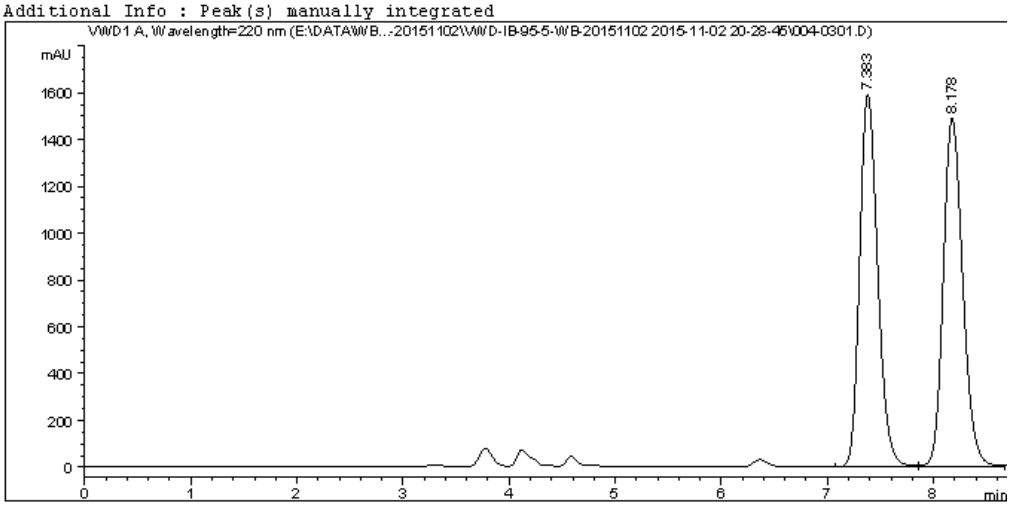



```
    ======================================================================
                           Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  22.663 BV    0.6340 5.55000e4  1287.30933  96.4829
       2  25.250 VB    0.6953 2023.12036   43.28545   3.5171

    Totals :                  5.7523le4  1330.59478

    ======================================================================
                           *** End of Report ***
```



Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel IB-H, hexane/*i*PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 7.4 min, t_{minor} = 8.2 min

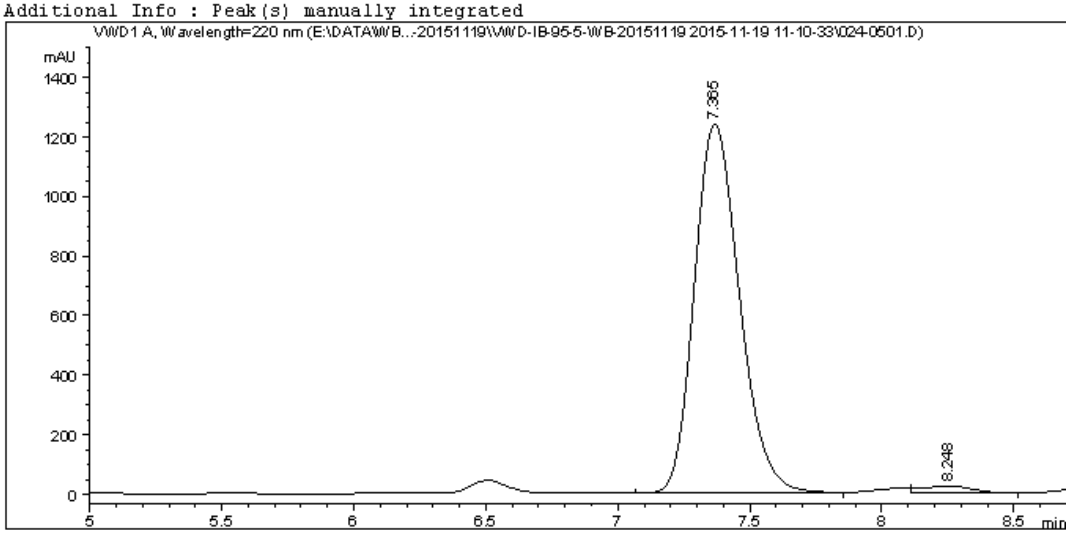
```
Data File E:\DATA\WB\WB-20151102\VWD-IB-95-5-WB-20151102 2015-11-02 20-28-45\004-0301.D
Sample Name: weib-20151102-4

    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260HPLC-VWD                  Location : Vial 4
    Injection Date  : 11/2/2015 9:21:03 PM               Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20151102\VWD-IB-95-5-WB-20151102 2015-11-02 20-28-45\VWD-
                      IB(1-6)-95-5-1ML-220NM-40MIN.M
    Last changed    : 11/2/2015 8:28:45 PM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20151102\VWD-IB-95-5-WB-20151102 2015-11-02 20-28-45\VWD-
                      IB(1-6)-95-5-1ML-220NM-40MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:23:03 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



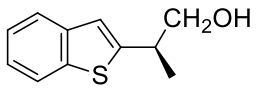

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   7.383 BB    0.1832 1.87011e4  1589.85840  49.8082
       2   8.178 BV    0.1968 1.88451e4  1487.36462  50.1918

    Totals :                   3.75462e4  3077.22302


    ======================================================================
                              *** End of Report ***

1260HPLC-DAD 3/27/2016 2:23:09 PM SYSTEM                    Page 1 of 1
```

Data File E:\DATA\WB\WEIB-20151119\VWD-IB-95-5-WB-20151119 2015-11-19 11-10-33\024-0501.D
Sample Name: WEIB-20151119-4

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   5
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 24
    Injection Date  : 11/19/2015 1:24:35 PM             Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WEIB-20151119\VWD-IB-95-5-WB-20151119 2015-11-19 11-10-33\VWD
                      -IB(1-6)-95-5-1ML-220NM-40MIN.M
    Last changed    : 11/19/2015 11:10:33 AM by SYSTEM
    Analysis Method : E:\DATA\WB\WEIB-20151119\VWD-IB-95-5-WB-20151119 2015-11-19 11-10-33\VWD
                      -IB(1-6)-95-5-1ML-220NM-40MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:25:21 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

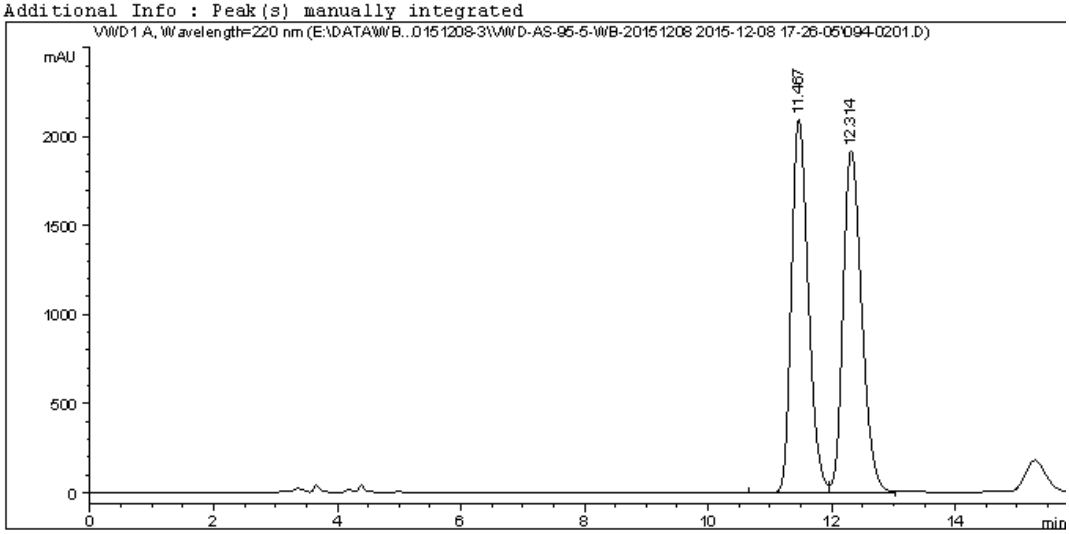



```
    ======================================================================
                           Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   7.365 BB    0.1848 1.47444e4  1239.19934  97.9761
       2   8.248 VB    0.1963  304.57535   23.48474   2.0239

    Totals :                  1.50490e4  1262.68408


    ======================================================================
                           *** End of Report ***
```



Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel AS-H, hexane/*i*PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{minor} = 11.5 min, t_{major} = 12.4 min

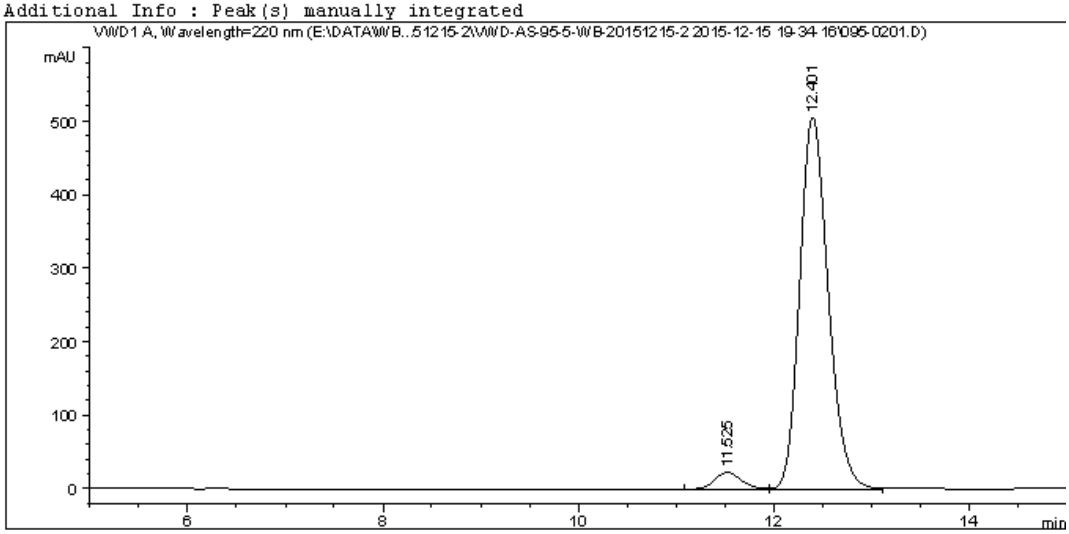
```
Data File E:\DATA\WB\WB-20151208-3\VWD-AS-95-5-WB-20151208 2015-12-08 17-26-05\094-0201.D
Sample Name: weib-20151208za-1

    ======================================================================
    Acq. Operator   : SYSTEM                       Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                  Location : Vial 94
    Injection Date  : 12/8/2015 6:08:17 PM               Inj :   1
                                                  Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20151208-3\VWD-AS-95-5-WB-20151208 2015-12-08 17-26-05\VWD
                      -AS(1-6)-95-5-1ML-5U-220NM-40MIN.M
    Last changed    : 12/8/2015 5:26:05 PM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20151208-3\VWD-AS-95-5-WB-20151208 2015-12-08 17-26-05\VWD
                      -AS(1-6)-95-5-1ML-5U-220NM-40MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:37:44 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



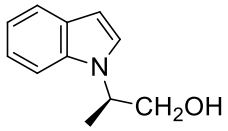

```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  11.467 BV    0.2952 3.97259e4  2094.88696  49.6959
       2  12.314 VV    0.3252 4.02121e4  1921.38354  50.3041

    Totals :                  7.99381e4  4016.27051

    ======================================================================
                            *** End of Report ***
```

Data File E:\DATA\WB...20151215-2\VWD-AS-95-5-WB-20151215-2 2015-12-15 19-34-16\095-0201.D
Sample Name: weib-20151215-za-1

```
=====================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                    Location : Vial 95
Injection Date  : 12/15/2015 7:45:49 PM                Inj :   1
                                                 Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WB\WB-20151215-2\VWD-AS-95-5-WB-20151215-2 2015-12-15 19-34-16
                  \VWD-AS(1-6)-95-5-1ML-5U-220NM-40MIN.M
Last changed    : 12/15/2015 7:34:17 PM by SYSTEM
Analysis Method : E:\DATA\WB\WB-20151215-2\VWD-AS-95-5-WB-20151215-2 2015-12-15 19-34-16
                  \VWD-AS(1-6)-95-5-1ML-5U-220NM-40MIN.M (Sequence Method)
Last changed    : 3/27/2016 2:39:57 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

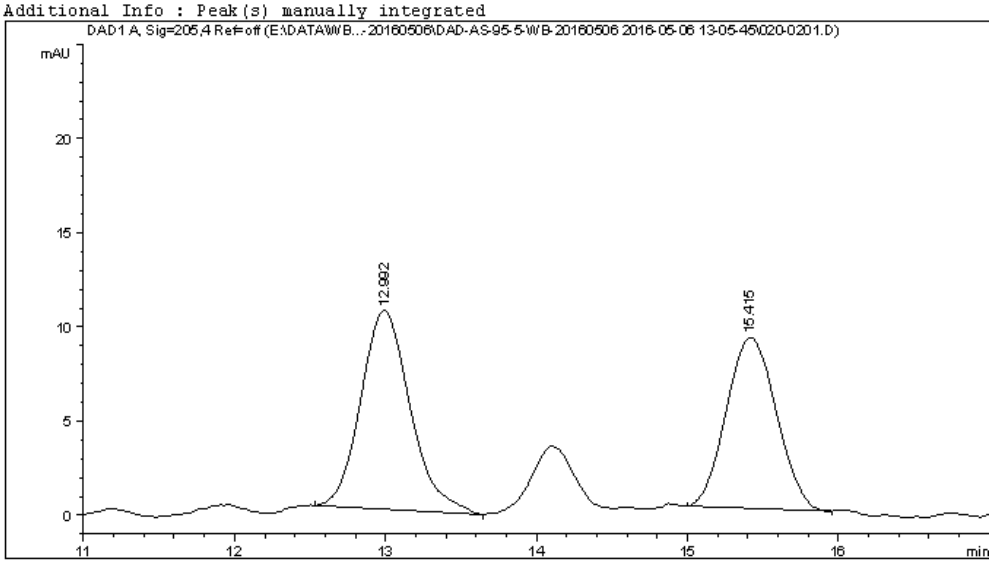



```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.525 BV    0.2849  423.10434   22.86581   3.9524
   2  12.401 VV    0.3138 1.02819e4   506.55368  96.0476

Totals :                  1.07050e4   529.41949

=====================================================================
                        *** End of Report ***
```



Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel AS-H, hexane/*i*PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{major} = 13.0 min, t_{minor}=15.3min

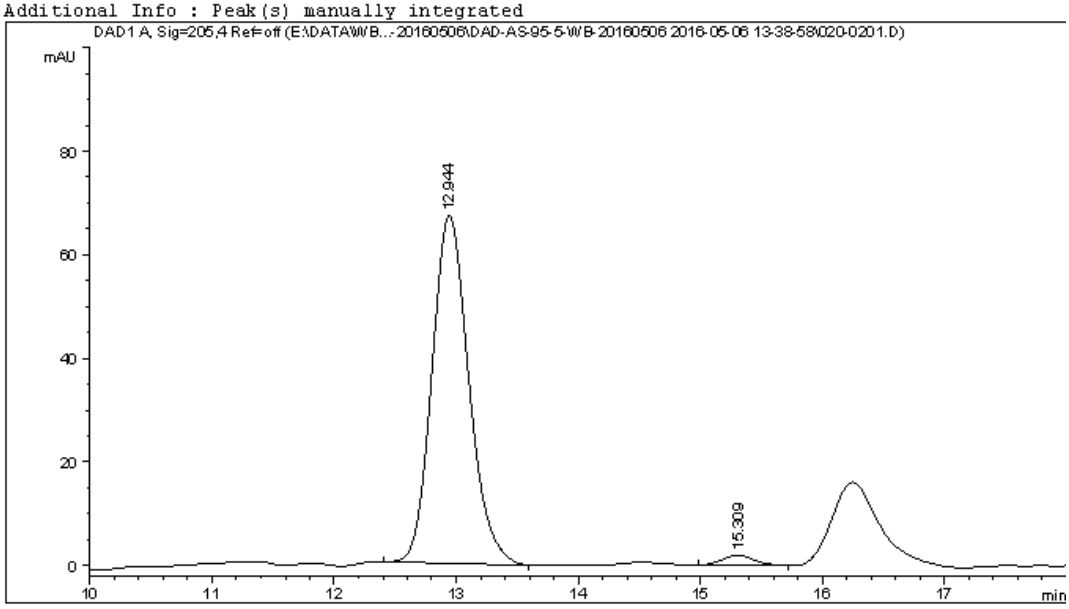
```
Data File E:\DATA\WB\WB-20160506\DAD-AS-95-5-WB-20160506 2016-05-06 13-05-45\020-0201.D
Sample Name: weib-20160506-6

    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 20
    Injection Date  : 5/6/2016 1:17:48 PM                 Inj :   1
                                                  Inj Volume : 8.000 µl
    Acq. Method     : E:\DATA\WB\WB-20160506\DAD-AS-95-5-WB-20160506 2016-05-06 13-05-45\DAD-
                      AS(1-6)-95-5-1ML-254-8U-40MIN.M
    Last changed    : 5/6/2016 1:31:30 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\WB\WB-20160506\DAD-AS-95-5-WB-20160506 2016-05-06 13-05-45\DAD-
                      AS(1-6)-95-5-1ML-254-8U-40MIN.M (Sequence Method)
    Last changed    : 5/7/2016 10:55:53 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



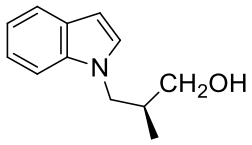

```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 A, Sig=205,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  12.992 BB    0.3334  239.86397   10.54427  53.5207
       2  15.415 BB    0.3145  208.30688    9.05233  46.4793

    Totals :                   448.17085   19.59660


    ======================================================================

1260HPLC-DAD 5/7/2016 10:56:01 AM SYSTEM                       Page 1 of 1
```

Data File E:\DATA\WB\WB-20160506\DAD-AS-95-5-WB-20160506 2016-05-06 13-38-58\020-0201.D
Sample Name: weib-20160506-8

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-DAD                  Location : Vial 20
    Injection Date  : 5/6/2016 1:51:00 PM              Inj :   1
                                                   Inj Volume : 8.000 µl
    Acq. Method     : E:\DATA\WB\WB-20160506\DAD-AS-95-5-WB-20160506 2016-05-06 13-38-58\DAD-
                      AS(1-6)-95-5-1ML-254-8U-40MIN.M
    Last changed    : 5/6/2016 1:38:59 PM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20160506\DAD-AS-95-5-WB-20160506 2016-05-06 13-38-58\DAD-
                      AS(1-6)-95-5-1ML-254-8U-40MIN.M (Sequence Method)
    Last changed    : 5/7/2016 10:47:22 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

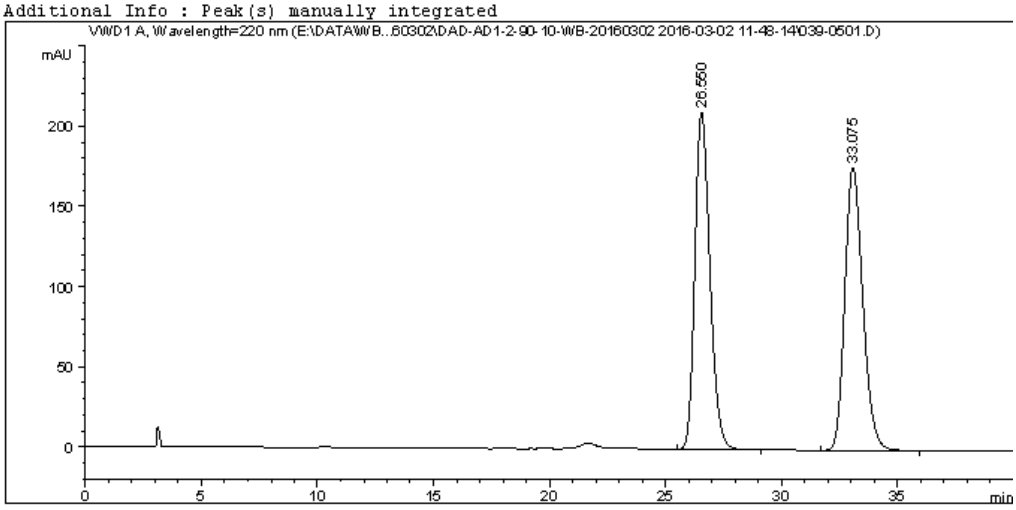



```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 A, Sig=205,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  12.944 BB    0.3205 1408.20715   67.19543  97.4761
       2  15.309 BB    0.2303   36.46145    2.00360   2.5239

    Totals :                  1444.66860   69.19904


    ======================================================================
                            *** End of Report ***
```



Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel OD-H, hexane/*i*PrOH = 95:5, flow rate = 1.0 mL/min, λ = 220 nm, t_{minor} = 26.6 min, t_{major} = 32.9 min

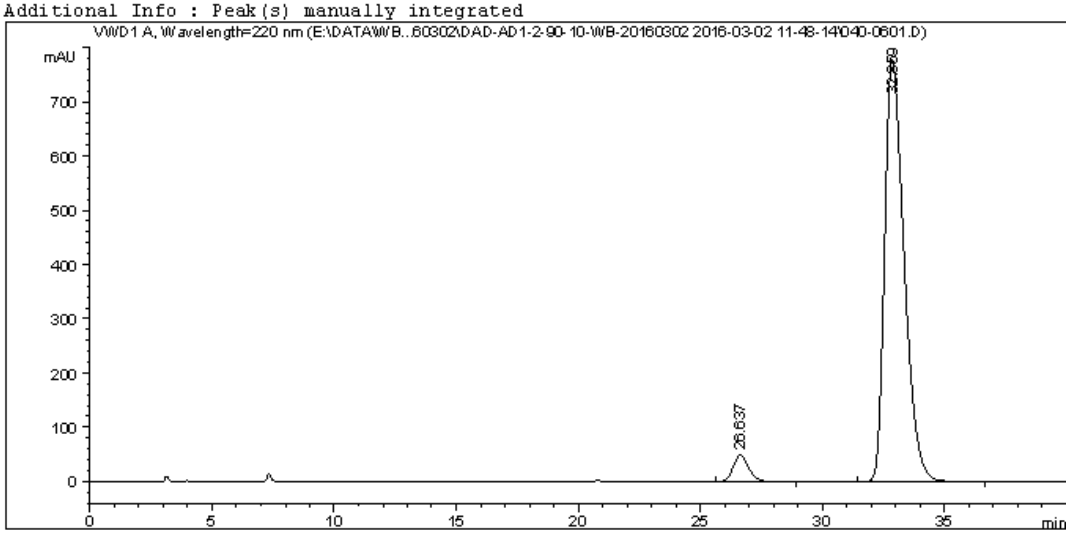
```
Data File E:\DATA\WB...20160302\DAD-AD1-2-90-10-WB-20160302 2016-03-02 11-48-14\039-0501.D
Sample Name: weib-20160302-3

    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   5
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 39
    Injection Date  : 3/2/2016 1:52:17 PM                Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20160302\DAD-AD1-2-90-10-WB-20160302 2016-03-02 11-48-14
                      \VWD-OD(1-2)-95-5-1ML-220NM-40MIN.M
    Last changed    : 3/2/2016 11:48:15 AM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20160302\DAD-AD1-2-90-10-WB-20160302 2016-03-02 11-48-14
                      \VWD-OD(1-2)-95-5-1ML-220NM-40MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:08:01 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



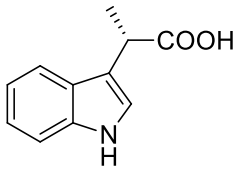

```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  26.550 BB    0.6942 9477.07715  210.51416  49.8747
       2  33.075 BB    0.8310 9524.70605  176.67365  50.1253

    Totals :                  1.90018e4   387.18781

    ======================================================================
                            *** End of Report ***


1260HPLC-DAD 3/27/2016 2:08:16 PM SYSTEM                    Page 1 of 1
```

Data File E:\DATA\WB...20160302\DAD-AD1-2-90-10-WB-20160302 2016-03-02 11-48-14\040-0601.D
Sample Name: weib-20160302-4

```
    ==================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   6
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 40
    Injection Date  : 3/2/2016 2:33:04 PM                Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20160302\DAD-AD1-2-90-10-WB-20160302 2016-03-02 11-48-14
                      \VWD-OD(1-2)-95-5-1ML-220NM-40MIN.M
    Last changed    : 3/2/2016 11:48:15 AM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20160302\DAD-AD1-2-90-10-WB-20160302 2016-03-02 11-48-14
                      \VWD-OD(1-2)-95-5-1ML-220NM-40MIN.M (Sequence Method)
    Last changed    : 3/27/2016 2:09:57 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

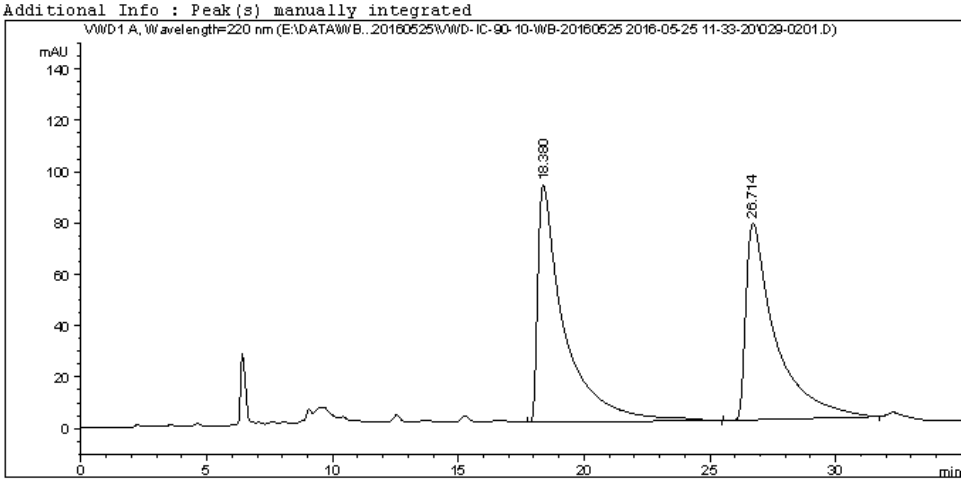



```
    ==================================================================
                          Area Percent Report
    ==================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  26.637 BB    0.6864 2193.65039   49.26874   4.8387
       2  32.859 BB    0.8560 4.31416e4   777.94818  95.1613

    Totals :                  4.53353e4   827.21692


    ==================================================================
                          *** End of Report ***
```



Enantiomeric excess was determined by HPLC analysis: Daicel Chiralcel IC, hexane/*i*-PrOH = 90:10, flow rate = 0.5 mL/min, λ = 220 nm, 40℃, t_{major} = 18.3 min, t_{minor} = 26.7 min

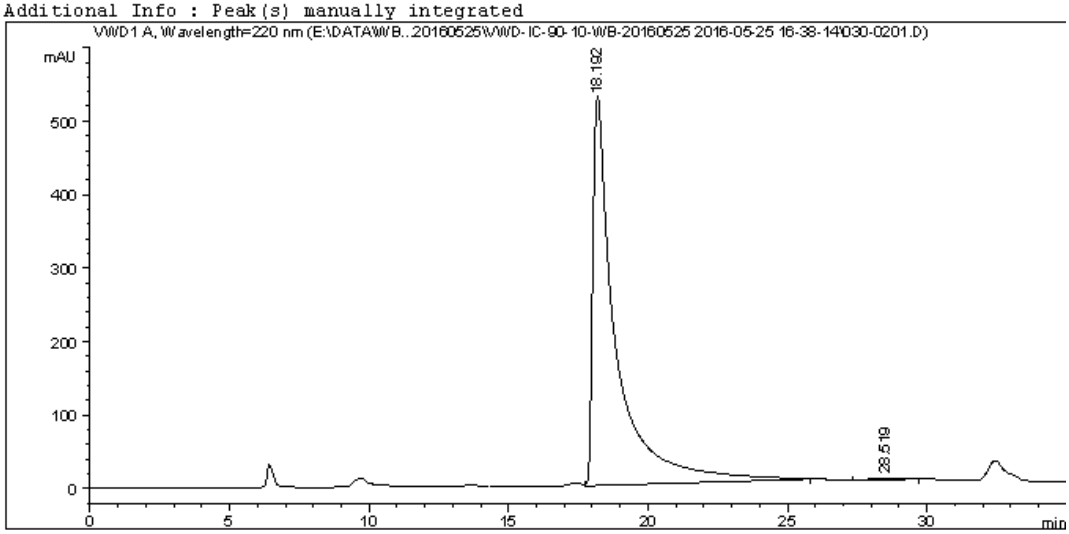
```
Data File E:\DATA\WB\WB-20160525\VWD-IC-90-10-WB-20160525 2016-05-25 11-33-20\029-0201.D
Sample Name: weib-20160525-3

    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 29
    Injection Date  : 5/25/2016 11:45:02 AM                 Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WB\WB-20160525\VWD-IC-90-10-WB-20160525 2016-05-25 11-33-20\VWD-
                      IC-90-10-0.5ML-40MIN-40DU-(1-2).M
    Last changed    : 5/25/2016 11:33:20 AM by SYSTEM
    Analysis Method : E:\DATA\WB\WB-20160525\VWD-IC-90-10-WB-20160525 2016-05-25 11-33-20\VWD-
                      IC-90-10-0.5ML-40MIN-40DU-(1-2).M (Sequence Method)
    Last changed    : 5/28/2016 11:15:57 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  18.380 BB    1.0032 6739.29395   92.25677  51.8738
       2  26.714 BB    1.1316 6252.40527   76.81479  48.1262

    Totals :                  1.29917e4   169.07156

    ======================================================================
                            *** End of Report ***


1260HPLC-DAD 5/28/2016 11:16:10 AM SYSTEM                    Page 1 of 1
```

Data File E:\DATA\WB\WB-20160525\VWD-IC-90-10-WB-20160525 2016-05-25 16-38-14\030-0201.D
Sample Name: weib-20160525-6

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 30
Injection Date  : 5/25/2016 4:53:00 PM               Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WB\WB-20160525\VWD-IC-90-10-WB-20160525 2016-05-25 16-38-14\VWD-
                  IC-90-10-0.5ML-40MIN-40DU-(1-2).M
Last changed    : 5/25/2016 4:38:15 PM by SYSTEM
Analysis Method : E:\DATA\WB\WB-20160525\VWD-IC-90-10-WB-20160525 2016-05-25 16-38-14\VWD-
                  IC-90-10-0.5ML-40MIN-40DU-(1-2).M (Sequence Method)
Last changed    : 5/28/2016 11:09:55 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  18.192 VB    0.7872 3.0794le4   529.73083  99.5989
   2  28.519 BB    0.8131  124.02528    1.81080   0.4011

Totals :                  3.0918le4   531.54163


==================================================================
                        *** End of Report ***
```