Development of a Pd-Catalyzed Enantioselective Carboalkoxylation Reaction for the Synthesis of Tetrahydrofurans

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Supporting Information

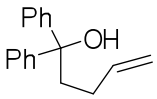
Experimental procedures and characterization data for new compounds.

Table of Contents

General: Reactions were carried out under nitrogen in flame-dried glassware. Tris(dibenzylideneacetone)dipalladium was purchased from Strem Chemical Co. and used without further purification. Dichloromethane and toluene were purified using a GlassContour solvent system. Anhydrous dioxane was purchased from Acros Organics

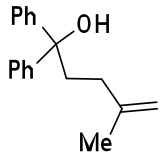
in a sure seal bottle and used as received. All other solvents and aryl halides were purchased from commercial sources and used as received. 1-(But-3-en-1-yl)cyclopentan-1-ol (**1a**),[1] 2,5-dimethylhex-5-en-2-ol (**1d**),[1] and (+)-(1*S*,2*R*)-2-phenylcyclohexan-1-ol,[2] 4-methyl-2,2-diphenylpent-4-en-1-ol (**5**),[3] and ligands **L1**–**L6**[4] were synthesized according to literature procedures. 4-Penten-1-ol (**1b**) was purchased from commercial sources and was used without further purification. Yields refer to isolated compounds that are estimated to be ≥95% pure as judged by ^{1}H NMR or GC analysis unless stated otherwise. The yields reported in the supporting information describe the result of a single experiment, whereas yields reported in Tables 2 and 3 are average yields of two or more experiments. Thus, the yields reported in the supporting information may differ from those in the manuscript.

Synthesis of Substrates:



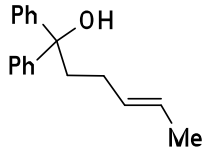
1,1-Diphenylpent-4-en-1-ol (1c).[5] A flame dried round bottom flask equipped with a stir bar was cooled under a stream of nitrogen and charged with 4-pentenoyl chloride (5 mmol, 0.55 mL) and diethyl ether (50 mL). The mixture was cooled to 0 °C in an ice bath for five min and then PhMgBr (20 mL, 20 mmol, 1M in THF) was added dropwise to the flask. The resulting mixture was warmed to rt and stirred for 12 h, then the flask was cooled to 0 °C in an ice bath and slowly quenched with saturated aqueous ammonium chloride (10 mL). The mixture was transferred to a separatory funnel, the layers were separated, and the aqueous layer was extracted with ethyl acetate (3 x 25 mL. The organic layers were combined, dried over anhydrous sodium sulfate, filtered,

and concentrated in vacuo. The crude product was then purified by flash chromatography on silica gel to afford the title compound (864 mg, 72%) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (d, *J* = 7.4 Hz, 4 H), 7.33 (t, *J* = 8.1 Hz, 4 H), 7.24 (t, *J* = 6.6 Hz, 2 H), 6.85–6.78 (m, 2 H), 5.06–4.96 (m, 2 H), 2.44–2.38 (m, 2 H), 2.18 (s, 1 H), 2.12–2.04 (m, 2 H). Spectroscopic data was consistent with that previously reported in the literature.[5]

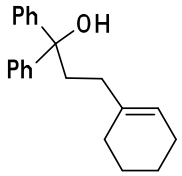


4-Methyl-1,1-diphenylpent-4-en-1-ol (1e). A flame dried round bottom flask equipped with a stir bar was cooled under a stream of nitrogen and charged with PhMgBr (25 mL, 25 mmol, 1M in THF). The solution was cooled to 0 °C in an ice bath for five min. In a separate flask ethyl 4-methylpent-4-enoate[6] (1.0 g, 7 mmol) was dissolved in 20 mL anhydrous THF, and the resulting solution was added dropwise to the flask containing the cooled PhMgBr solution. The reaction mixture was then warmed to rt, stirred for 12 h, then was cooled to 0 °C in an ice bath and slowly quenched with saturated aqueous ammonium chloride (20 mL). The resulting mixture was transferred to a separatory funnel, the layers were separated, and the aqueous layer was extracted with diethyl ether (3x25 mL). The organic layers were combined, dried over anhydrous sodium sulfate, filtered, and concentrated in vacuo. The crude product was then purified by flash chromatography on silica gel to afford the title compound (1.54 g, 88%) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (dd, *J* = 8.31, 0.98 Hz, 4 H), 7.34–7.31 (m, 4H), 7.25–7.20 (m, 2 H), 4.73 (s, 1 H), 4.70 (s, 1 H), 2.48–2.42 (m, 2 H), 2.25 (s, br,

1 H), 2.06–1.99 (m, 2 H), 1.74 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 147.1, 146.4, 128.4, 127.0, 126.2, 110.1, 78.5, 40.0, 32.2, 23.0; IR (film) 3469, 2932, 1446 cm^{-1}; MS (EI) 252.1515 (252.1514 calcd for $C_{18}H_{20}O$, M +).



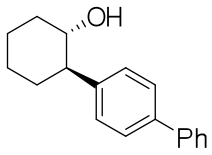
(*E*)-1,1-Diphenylhex-4-en-1-ol (1f). The title compound was prepared from PhMgBr (50 mL, 50 mmol, 1M in THF) and (*E*)-ethyl hex-4-enoate[7] (2.28 g, 16.0 mmol) using a procedure analogous to that described above for the synthesis of **1e**. This procedure afforded the title compound (1.23 g, 30%) as a colorless solid, mp 53–54 °C: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40–7.43 (m, 4 H), 7.28–7.33 (m, 4 H), 7.20–7.24 (m, 2 H), 5.37–5.51 (m, 2 H), 2.33–2.38 (m, 2 H), 2.23 (s, 1 H), 1.96–2.03 (m, 2 H), 1.63 (dd, *J* = 5.9, 1.0 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 147.2, 131.3, 128.3, 127.0, 126.2, 125.7, 78.6, 41.7, 27.3, 18.1; IR (film) 3556, 2958, 1446 cm^{-1}; MS (EI) 252.1510 (252.1514 calcd for $C_{18}H_{20}O$, M +).



3-(Cyclohex-1-en-1-yl)-1,1-diphenylpropan-1-ol (1g). The title compound was prepared from PhMgBr (11 mL, 11 mmol, 1M in THF) and 3-(cyclohex-1-en-1-yl)-1-phenylpropan-1-one[8] (1.2 g, 5.5 mmol) using a procedure analogous to that described above for the synthesis of **1e**. This procedure afforded the title compound (600 mg,

37%) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39–7.49 (m, 4 H), 7.28–7.34 (m, 4 H), 7.18–7.24 (m, 2 H), 5.41 (s, 1 H), 2.38–2.45 (m, 2 H), 2.37 (s, 1 H), 1.88–2.03 (m, 6 H), 1.48–1.67 (m, 4 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 147.2, 138.2, 128.3, 126.9, 126.2, 121.6, 78.7, 39.8, 32.5, 28.7, 25.4, 23.1, 22.7; IR (film) 3467, 2923, 1446 cm^{-1}; MS (EI) 292.1823 (292.1827 calcd for $C_{21}H_{24}O$, M +).

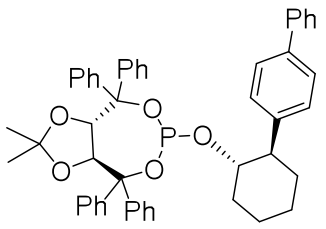
Synthesis of ligand L7.



(–)-(1*S*,2*R*)-2-[(1,1'-Biphenyl)-4-yl]cyclohexan-1-ol (S1). A flame-dried 2-neck round bottom flask equipped with a stirbar and a reflux condenser was cooled under a stream of nitrogen and charged with magnesium turnings (1.76 g, 72 mmol) and THF (50 mL). A solution of 4-bromobiphenyl (11.65 g, 50 mmol) in THF (15 mL) was slowly added. The reaction mixture began to rapidly reflux, and the reaction temperature was controlled by placing the flask in an ice bath until reflux subsided. Once the magnesium turnings had disappeared, the reaction mixture was cooled to -20 °C for 10 min then CuCl (8 mol%) was added to the reaction mixture immediately followed by the addition of cyclohexene oxide (3.36 mL, 33.3 mmol) as a solution in THF (7 mL). The resulting mixture was allowed to slowly warm to rt and stirred for 4 h. The mixture was then cooled to 0 °C and quenched with saturated ammonium chloride (1mL/mmol cyclohexene oxide). The mixture was filtered through a pad of celite, and transferred to a separatory funnel. The layers were separated and the aqueous layer was extracted with ethyl acetate (3x). The combined organic layers were then dried over sodium

sulfate, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel to afford 4.00 g (48 %) of (±)-**S1** as a white solid.

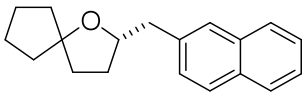
A flame dried round bottom flask equipped with a stirbar was cooled under a stream of nitrogen and charged with PS 30 Amano Lipase (14.8 mg), (±)-**S1** (3.74 g, 14.8 mmol) and *tert*-butyl methyl ether (45 mL). Neat vinyl acetate (13.6 mL, 148 mmol) was then added and the resulting mixture was stirred at rt until one enantiomer of the alcohol had been consumed as judged by chiral HPLC analysis (3 days). The mixture was then filtered through a fritted funnel and the enzyme was washed with diethyl ether and then recycled for future use (if desired). The resulting solution was concentrated in vacuo and the crude product was purified by flash chromatography on silica gel to afford 1.72 g (46%) of the title compound as a white solid, mp 122–125 °C. This material was judged to be >99:1 er by chiral HPLC analysis (Chiracel OJH, 25 cm x 4.6 mm, 4% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 21.8 and 25.0 min). $[\alpha]^{23}_{D}$ –13.99 (*c* 3.38, CH_2Cl_2); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.62–7.57 (m, 4 H), 7.45 (t, *J* = 7.7 Hz, 2 H), 7.37–7.34 (m, 3 H), 3.72 (td, *J* = 10.0, 4.2 Hz, 1 H), 2.51 (td, *J* = 11.1, 3.6 Hz, 1 H), 2.1–2.15 (m, 1 H), 1.95–1.88 (m, 2 H), 1.81 (app. d, *J* = 13.2 Hz, 1 H), 1.64–1.35 (m, 5 H); ^{13}C NMR (175 MHz $CDCl_3$) δ 142.6, 141.1, 140.0, 128.9, 128.5, 127.7, 127.3, 127.2, 74.6, 53.1, 34.5, 33.5, 26.2, 25.3; IR (film) 3548, 2919, 1490 cm^{-1}; MS (ESI+) 270.1850 (270.1852 calcd for $C_{18}H_{20}O$, M + NH_4^+).



(+)-(1*S*,2*R*,3a*S*,8a*S*)-6-{[-2-([1,1'-Biphenyl]-4-yl)cyclohexyl]oxy}-2',2'-dimethyl-4,4,8,8-Tetraphenyltetrahydro-[1,3]dioxolo[4,5-*e*][1,3,2]dioxaphosphepine (L7). The ligand was prepared according to a previously reported procedure for the synthesis of chiral phosphites.[4] A flame dried round bottom flask equipped with a stirbar was cooled under a stream of nitrogen and charged with (1*S*,2*R*)-2-([1,1'-biphenyl]-4-yl)cyclohexan-1-ol (255 mg, 1.01 mmol), and dry dichloromethane (2 mL). Neat PCl_3 (86 µL, 1.01 mmol), was added and the resulting mixture was allowed to stir for 1 h at rt. After this time, anhydrous NEt_3 (0.56 mL, 4.04 mmol) was added dropwise and the mixture was stirred at rt for 30 min. A solution of (*S*,*S*)-TADDOL (450 mg, 0.963 mmol) in dichloromethane (2 mL) was added, and the reaction mixture was stirred at rt for 12 h. The mixture was then diluted with diethyl ether (20 mL) and then filtered through celite. The solvent was evaporated in vacuo and the crude product was purified by flash chromatography on silica gel to afford 520 mg (72%) of the title compound as a white foamy solid, mp 115–118 °C. $[\alpha]^{23}_{D}$ +130.0 (*c* 5.81, CH_2Cl_2); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.51 (d, *J* = 7.9 Hz, 2 H), 7.38–7.02 (m, 27 H), 4.94–4.90 (m, 1 H), 4.88 (d, *J* = 8.4 Hz, 1 H), 4.56 (app. qd, *J* = 9.9, 3.7 Hz, 1 H), 2.71–2.66 (m, 1 H), 2.28 (app. d, *J* = 13.8 Hz, 1 H), 1.95 (app. d, *J* = 13.2 Hz, 1 H), 1.81–1.73 (m, 2 H), 1.69–1.50 (m, 2 H), 1.41–1.31 (m, 2 H), 1.19 (s, 3 H), 0.30 (s, 3 H); ^{13}C NMR (175 MHz $CDCl_3$) δ 146.6, 146.1, 143.0, 141.8, 141.25, 141.23, 139.4, 129.4, 129.0, 128.92, 128.89, 128.6, 128.0, 127.8, 127.54, 127.48, 127.45, 127.38, 127.34, 127.30, 127.18, 127.15, 127.12, 127.06, 126.9, 112.0, 82.9, 82.7, 82.6, 82.12, 82.10, 81.92, 81.88, 78.10, 78.09, 51.39, 51.37, 35.7, 33.8, 27.6, 26.0, 25.5, 25.3 (due to the complexity of the spectra all the peaks are listed without assigning C-P couplings); ^{31}P NMR (202 MHz $CDCl_3$) δ 140.6; IR (film)

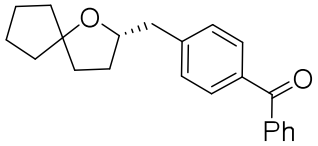
2932, 1486, 1447 cm^{-1}; MS (ESI+) 747.3224 (747.3234 calcd for $C_{49}H_{47}O_5P$, M + H^+).

General procedure for asymmetric Pd-catalyzed carboalkoxylation reactions. A flame-dried Schlenk tube equipped with a stirbar was cooled under a stream of nitrogen and charged with $Pd_2(dba)_3$ (2 mol %), **L7** (5 mol %), the alcohol substrate (1.0 equiv), and NaO*t*Bu (1.50–2.0 equiv). The flask was purged with N_2 then the aryl or alkenyl halide (1.40–2.0 equiv), and dioxane or toluene (0.10 M) was added. The resulting mixture was heated to 90 °C with stirring until the starting material had been consumed as judged by TLC analysis (ca. 12 h). The reaction mixture was then cooled to rt, saturated aqueous ammonium chloride (6 mL/mmol substrate) was added, and the mixture was transferred to a separatory funnel. The mixture was extracted with ethyl acetate (3 x 5 mL) then the combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel.



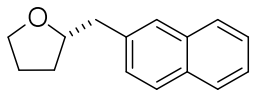
(+)-(*S*)-2-(Naphthalen-2-ylmethyl)-1-oxaspiro[4.4]nonane (2a). The general procedure was employed for the coupling of 1-(but-3-en-1-yl)cyclopentan-1-ol (28.0 mg, 0.20 mmol) and 2-bromonaphthalene (75.0 mg, 0.36 mmol) using a catalyst composed of $Pd_2(dba)_3$ (3.7 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of 90 °C and a reaction time of 12 h in 2mL of dioxane. This procedure afforded the title compound (31.1 mg, 58%, 10:1 regioselectivity) as a colorless oil: $[\alpha]^{23}_D$ +12.4(*c* 2.1, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85–7.79 (m, 3 H), 7.67 (s, 1 H), 7.49–7.35 (m, 3

H), 4.29–4.22 (m, 1 H), 3.15 (dd, *J* = 13.6, 5.0 Hz, 1 H), 2.87 (dd, *J* = 13.3, 7.7 Hz, 1 H), 1.95–1.87(m, 1 H), 1.86–1.49 (m, 11 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.6, 133.7, 132.3, 128.4, 127.84, 127.79, 127.75, 127.7, 91.6, 79.2, 43.0, 39.4, 38.6, 36.6, 24.2; IR (film) 2953, 2361, 2338, 1508 cm^{-1}. MS (CI) 267.1743 (267.1743 calcd for $C_{19}H_{22}O$, M + H^+). The enantiopurity was determined to be 89:11 er by chiral HPLC analysis (Chiralcel OJH, 25 cm x 4.6 mm, 1% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 10.5 and 12.8 min).

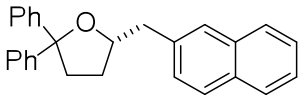


(+)-(S)-2-(Naphthalen-2-ylmethyl)-1-oxaspiro[4.4]nonane (2b). The general procedure was employed for the coupling of 1-(but-3-en-1-yl)cyclopentan-1-ol (28 mg, 0.20 mmol) and 4-bromobenzophenone (94 mg, 0.36 mmol) using a catalyst composed of $Pd_2(dba)_3$ (3.7 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of 90 °C, and a reaction time of 12 h in 2mL of dioxane. This procedure afforded the title compound (34.6 mg, 53%, 6:1 regioselectivity) as a clear oil: $[\alpha]^{23}_D$ +21.9 (*c* 1.77, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80–7.77 (m, 2 H), 7.74 (d, *J* = 8.1 Hz, 2 H), 7.57 (t, *J* = 7.3 Hz, 1 H), 7.49–7.45 (m, 2 H), 7.34 (d, *J* = 8.3 Hz, 2 H), 4.19 (app. quint, *J* = 6.6 Hz, 1 H), 3.01 (dd, *J* = 13.4, 5.6 Hz, 1 H), 2.80 (dd, *J* = 13.4, 6.8 Hz, 1 H), 1.96–1.90 (m, 1 H), 1.83–1.48 (m, 11 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.5, 144.1, 137.9, 132.2, 130.1, 129.9, 129.4, 128.3, 128.2, 127.6, 91.5. 78.5, 42.7, 39.2, 38.4, 36.4, 31.3, 24.0, 23.9; IR (film) 2959, 1655, 1606, 1277 cm^{-1}. MS (CI) 321.1848 (321.1849 calcd for $C_{22}H_{24}O_2$, M + H^+). The enantiopurity was determined to be 82:18 er by chiral HPLC

analysis (Chiralcel ADH, 25 cm x 4.6 mm, 1% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 17.4 and 18.4 min).

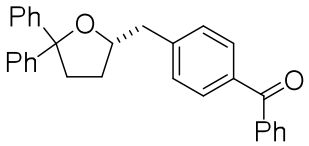


(+)-(*S*)-2-(Naphthalen-2-ylmethyl)tetrahydrofurantetrahydrofuran (2c). The general procedure was employed for the coupling of pent-4-en-1-ol (17 mg, 0.20 mmol) and 2-bromonaphthalene (58 mg, 0.28 mmol) using a catalyst composed of $Pd_2(dba)_3$ (3.7 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of 90 °C, and a reaction time of 12 h in 2mL of dioxane. This procedure afforded the title compound (9.9 mg, 23%) as a light yellow oil. $[\alpha]^{23}_{D}$= +2.1 (*c* 0.95, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83–7.75 (m, 3 H), 7.68 (s, 1 H), 7.48–7.36 (m, 3 H), 4.18 (app. quint, *J* = 6.5 Hz, 1 H), 3.95–3.89 (m, 1 H), 3.79–3.73 (m, 1 H), 3.08 (dd, *J* = 13.5, 6.4 Hz, 1 H), 2.92 (dd, *J* = 13.7, 6.4 Hz, 1 H), 1.98–1.82 (m, 3 H), 1.66–1.57 (m, 1 H). Other spectroscopic data matched those previously reported.[1] The enantiopurity was determined to be 58:42 er by chiral HPLC analysis (Chiralcel OJH, 25 cm x 4.6 mm, 1% IPA/Hexanes, 1.50 mL/min, λ 254 nm, RT= 19.8 and 26.1 min).



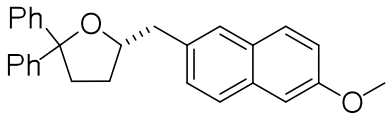
(+)-(*S*)-5-(Naphthalen-2-ylmethyl)-2,2-diphenyltetrahydrofuran (2d). The general procedure was employed for the coupling of 1,1-diphenylpent-4-en-1-ol (48 mg, 0.20 mmol) and 2-bromonaphthalene (58 mg, 0.28 mmol) using a catalyst composed of $Pd_2(dba)_3$ (3.7 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of

90 °C and a reaction time of 12 h in 2mL of dioxane. This procedure afforded the title compound (48.7mg, 67%) as a white solid, mp 83–86 °C. $[\alpha]^{23}_{D}$ +29.6 (*c* 4.24, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.2 Hz, 1 H), 7.81 (d, *J* = 8.6 Hz, 2 H), 7.74 (s, 1 H), 7.55–7.44 (m, 7 H), 7.37–7.31 (m, 4 H), 7.28–7.21 (m, 2 H), 4.53 (app. quint, *J* = 6.7 Hz, 1 H), 3.34 (dd, *J* = 13.6, 6.0 Hz, 1 H), 3.02 (dd, *J* = 13.6, 7.0 Hz, 1 H), 2.71–2.64 (m, 1 H), 2.58–2.51 (m, 1 H), 2.03–1.95 (m, 1 H), 1.89–1.81 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 147.4, 146.9, 136.6, 133.7, 132.3, 128.31, 128.25, 128.2, 127.88, 127.87, 127.8, 127.6, 126.76, 126.74, 126.1, 126.0, 125.4, 88.5, 79.9, 42.8, 38.8, 31.0; IR (film) 2934, 1601, 1446 cm^{-1}. MS (CI) 365.1899 (365.1900 calcd for $C_{27}H_{24}O$, M + H^+). The enantiopurity was determined to be 95:5 er by chiral HPLC analysis (Chiralcel ADH, 25 cm x 4.6 mm, 2% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 5.2 and 6.3 min). When 2.0 equiv of H_2O was added with toluene as solvent the enantiopurity was determined to be 96:4 er.



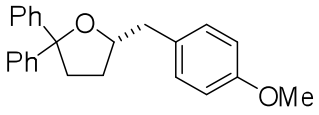
(+)-(*S*)-{4-[(5,5-diphenyltetrahydrofuran-2-yl)methyl]phenyl}(phenyl)methanone (2e). The general procedure was employed for the coupling of 1,1-diphenylpent-4-en-1-ol (48 mg, 0.20 mmol) and 4-bromobenzophenone (94 mg, 0.36 mmol) using a catalyst composed of $Pd_2(dba)_3$ (3.7 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of 90 °C and a reaction time of 12 h in 2mL of dioxane. This procedure afforded the title compound (52 mg, 62%) as a colorless oil. $[\alpha]^{23}_{D}$ +18.9 (*c* 2.40, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82–7.78 (m, 2 H), 7.75 (d, *J* = 8.1 Hz, 2 H),

7.59 (t, *J* = 7.2 Hz, 1 H), 7.51–7.40 (m, 6 H), 7.38 (d, *J* = 8.1 Hz, 2 H), 7.31–7.25 (m, 4 H), 7.22–7.17 (m, 2 H), 4.42 (app. quint, *J* = 6.6 Hz, 1 H), 3.16 (dd, *J* = 13.6, 6.6 Hz, 1 H), 2.91 (dd, *J* = 13.7, 6.4 Hz, 1 H), 2.68–2.61 (m, 1 H), 2.54–2.47 (m, 1 H), 2.03–1.96 (m, 1 H), 1.81–1.73 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.7, 147.2, 146.7, 144.2, 138.0, 135.7, 132.4, 130.4, 130.2, 129.5, 128.40, 128.35, 128.2, 126.83, 126.80, 126.0, 125.9, 88.6, 79.5, 42.7, 38.7, 31.2; IR (film) 2362, 1654, 1446 cm^{-1}. MS (CI) 419.2006 (419.2006 calcd for $C_{30}H_{26}O_2$, M + H^+). The enantiopurity was determined to be 92:8 er by chiral HPLC analysis (Chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 9.8 and 11.7 min). When 2.0 equiv of H_2O was added with toluene as solvent the enantiopurity was determined to be 95:5 er (an unknown product co-eluted when water was used with this reaction see spectra for product **2e** below.)



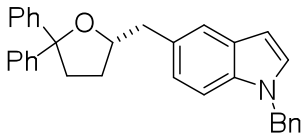
(+)-(*S*)-5-[(6-methoxynaphthalen-2-yl)methyl]-2,2-diphenyltetrahydrofuran (2f). The general procedure was employed for the coupling of 1,1-diphenylpent-4-en-1-ol (48 mg, 0.20 mmol) and 2-bromo-6-methoxynaphthalene (85 mg, 0.36 mmol) using a catalyst composed of $Pd_2(dba)_3$ (3.7 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of 90 °C and a reaction time of 12 h in 2mL of dioxane. This procedure afforded the title compound (52 mg, 66%) as a white solid, mp 93–96 °C. $[\alpha]^{23}_D$ +29.8 (*c* 5.19, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (app. dd, *J* = 8.6, 3.2 Hz, 2 H), 7.65 (s, 1 H), 7.53–7.46 (m, 4 H), 7.69 (d, *J* = 8.4 Hz, 1 H), 7.36–7.28 (m, 4 H), 7.27–7.13 (m, 4 H), 4.50 (app. quint, *J* = 6.7 Hz, 1 H), 3.94 (s, 3H), 3.29 (dd, *J* = 13.7, 5.9 Hz, 1 H), 2.97

(dd, *J* = 13.7, 7.1 Hz, 1 H), 2.69–2.61 (m, 1 H), 2.55–2.48 (m, 1 H), 2.00–1.93 (m, 1 H), 1.87–1.79 (m, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 157.4, 147.4, 146.9, 134.2, 133.3, 129.2, 128.6, 128.3, 128.2, 127.7, 126.74, 126.72, 126.1, 126.0, 118.8, 105.8, 88.5, 80.0, 55.4, 42.6, 38.8, 30.9; IR (film) 2937, 1605, 1448 cm^{-1}. MS (CI) 395.2004 (395.2006 calcd for $C_{28}H_{26}O_2$, M + H^+). The enantiopurity was determined to be 95:5 er by chiral HPLC analysis (Chiralcel ADH, 25 cm x 4.6 mm, 2% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 7.5 and 8.9 min).

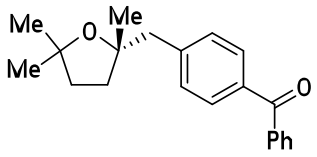


(+)-(*S*)-5-(4-Methoxybenzyl)-2,2-diphenyltetrahydrofuran (2g). The general procedure was employed for the coupling of 1,1-diphenylpent-4-en-1-ol (48 mg, 0.20 mmol) and 4-bromoanisole (46 μL, 0.36 mmol) using a catalyst composed of $Pd_2(dba)_3$ (3.7 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of 90 °C and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound (45.0 mg, 65%) as a colorless oil: $[\alpha]^{23}_D$ +24.5 (*c* 2.00, CH_2Cl_2); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.47–7.42 (m, 4 H), 7.30–7.26 (m, 4 H), 7.21–7.15 (m, 4 H), 6.82 (d, *J* = 8.6 Hz, 2 H), 4.33 (app. quint, *J* = 6.6 Hz, 1 H), 3.79 (s, 3 H), 3.08 (dd, *J* = 13.6, 5.8 Hz, 1 H), 2.74 (dd, *J* = 13.7, 7.2 Hz, 1 H), 2.64–2.59 (m, 1 H), 2.50–2.45 (m, 1 H), 1.95–1.89 (m, 1 H), 1.76–1.70 (m, 1 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 158.2, 147.4, 146.9, 131.1, 130.5, 128.3, 128.1, 126.73, 126.70, 126.1, 126.0, 113.9, 88.4, 80.2, 55.4, 41.8, 38.8, 30.9; IR (film) 2936, 1606, 1512 cm^{-1}. MS (CI) 345.1847 (345.1855 calcd for $C_{24}H_{24}O_2$, M + H^+). The enantiopurity was determined to be 94:6 er by chiral HPLC

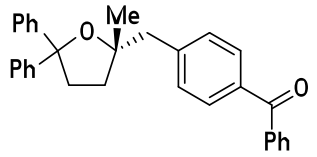
analysis (Chiralcel ADH, 25 cm x 4.6 mm, 2% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 7.5 and 8.9 min).



(+)-(*S*)-1-Benzyl-5-[(5,5-diphenyltetrahydrofuran-2-yl)methyl]-1*H*-indole (2h). The general procedure was employed for the coupling of 1,1-diphenylpent-4-en-1-ol (48 mg, 0.20 mmol) and 1-benzyl-5-bromo-1*H*-indole (103 mg, 0.36 mmol) using a catalyst composed of $Pd_2(dba)_3$ (3.7 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of 90 °C and a reaction time of 12 h in 2mL of dioxane. This procedure afforded the title compound (63.5 mg, 72%) as a colorless oil: $[\alpha]^{23}_{D}$ +14.7 (*c* 6.00, CH_2Cl_2); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.54–7.46 (m, 5 H), 7.33–7.25 (m, 7 H), 7.23–7.18 (m, 3 H), 7.15–7.08 (m, 4 H), 6.50 (d, *J* = 3.1 Hz, 1 H), 5.30 (s ,2 H), 4.44 (app. quint, *J* = 6.7 Hz, 1 H), 3.30 (dd, *J* = 13.5, 5.4, Hz 1 H), 2.88 (dd, *J* = 13.5, 7.9 Hz, 1 H), 2.65–2.60 (m, 1 H), 2.55–2.50 (m, 1 H), 1.95–1.89 (m, 1 H), 1.84–1.78 (m, 1 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 147.5, 147.0, 137.8, 135.3, 130.0, 129.0, 128.9, 128.3, 128.1, 127.7, 127.0, 126.68, 126.66, 126.13, 126.08, 123.7, 121.4, 109.5, 101.5, 88.4, 80.9, 50.3, 42.8, 38.9, 30.9; IR (film) 2923, 1485, 1446 cm^{-1}. MS (CI) 444.2319 (444.2322 calcd for $C_{32}H_{29}NO$, M + H^+). The enantiopurity was determined to be 93:7 er by chiral HPLC analysis (Chiralcel ADH, 25 cm x 4.6 mm, 2% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 10.8 and 21.3 min).

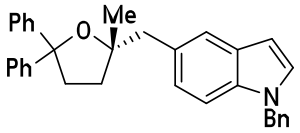


(+)-(*S*)-Phenyl{4-[(2,5,5-trimethyltetrahydrofuran-2-yl)methyl]phenyl}methanone (2j). The general procedure was employed for the coupling of 2,5-dimethylhex-5-en-2-ol[1] (26 mg, 0.20 mmol) and 4-bromobenzophenone (94 mg, 0.36 mmol) using a catalyst composed of $Pd_2(dba)_3$ (3.7 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of 90 °C and a reaction time of 12 h in 2mL of dioxane. This procedure afforded the title compound (51.0 mg, 77%) as a colorless oil: $[\alpha]^{23}_{D}$ +3.48 (*c* 7.10, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76–7.82 (m, 2 H), 7.73 (d, *J* = 8.1 Hz, 2 H), 7.54–7.60 (m, 1 H), 7.47 (t, *J* = 10.0 Hz, 2 H), 7.35 (d, *J* = 8.1 Hz, 2 H), 2.85 (s, 2 H), 1.95–2.03 (m, 1 H), 1.76–1.85 (m, 2 H), 1.56–1.66 (m, 1 H), 1.25 (d, *J* = 2.9 Hz, 6 H), 1.14 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 196.8, 143.9, 138.1, 135.6, 132.3, 130.8, 130.1, 129.9, 128.4, 83.3, 81.7, 48.5, 38.6, 36.7, 29.9, 29.4, 28.6; IR (film) 2966, 1654, 1277 cm^{-1}; MS (ESI+) 309.1847 (309.1849 calcd for $C_{21}H_{24}O_2$, M + H^+). The enantiopurity was determined to be 38:62 er by chiral HPLC analysis (Chiralcel ADH, 25 cm x 4.6 mm, 2% IPA/Hexanes, 1.00 mL/min, λ 195 nm, RT= 10.1 and 10.8 min).



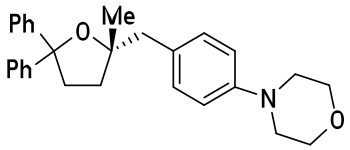
(+)-(*S*)-{4-[(2-Methyl-5,5-diphenyltetrahydrofuran-2 yl)methyl]phenyl}(phenyl)methanone (2k). The general procedure was employed for the coupling of 4-methyl-1,1-diphenylpent-4-en-1-ol (51 mg, 0.20 mmol) and 4-

bromobenzophenone (94 mg, 0.36 mmol) using a catalyst composed of $Pd_2(dba)_3$ (3.7 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of 90 °C and a reaction time of 12 h in 2mL of dioxane. This procedure afforded the title compound (74.8 mg, 86%) as a colorless solid, mp 89–91 °C: $[\alpha]^{23}_D$ +20.9 (*c* 6.30, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79–7.86 (m, 2 H), 7.72 (d, *J* = 8.1 Hz, 2 H), 7.58–7.64 (m, 1 H), 7.46–7.54 (m, 6 H), 7.26–7.40 (m, 6 H), 7.16–7.25 (m, 2 H), 3.03 (d, *J* = 13.2 Hz, 1 H), 2.91 (d, *J* = 13.2 Hz, 1 H), 2.62–2.75 (m, 2 H), 2.09 (m, 1 H), 1.86 (m 1 H), 1.31 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 196.7, 148.2, 147.7, 143.8, 138.0, 135.6, 132.3, 130.6, 130.1, 129.9, 128.4, 128.1, 128.1, 126.7, 126.5, 126.0, 125.8, 88.7, 84.6, 48.5, 38.4, 37.4, 27.2; IR (film) 2966, 1654, 1277 cm^{-1}; MS (ESI+) 433.2160 (433.2162 calcd for $C_{31}H_{28}O_2$, M + H^+). The enantiopurity was determined to be 95:5 er by chiral HPLC analysis (Chiralcel ADH, 25 cm x 4.6 mm, 2% IPA/Hexanes, 1.00 mL/min, λ 275 nm, RT= 15.4 and 18.1 min).



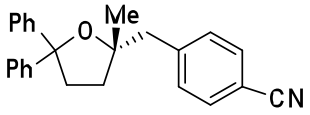
(+)-(*S*)-1-Benzyl-5-[(2-methyl-5,5-diphenyltetrahydrofuran-2-yl)methyl]-1H-indole (2l). The general procedure was employed for the coupling of 4-methyl-1,1-diphenylpent-4-en-1-ol (51 mg, 0.20 mmol) and 1-benzyl-5-bromo-1*H*-indole (103 mg, 0.36 mmol) using a catalyst composed of $Pd_2(dba)_3$ (3.7 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of 90 °C and a reaction time of 12 h. This procedure afforded the title compound (80.5 mg, 88%) as a colorless solid, mp 127–128 °C : $[\alpha]^{23}_D$ +22.6 (*c* 6.91, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.43–7.51 (m, 5 H),

7.22–7.33 (m, 10 H), 7.10–7.20 (m, 5 H), 7.08 (d, *J* = 3.2 Hz, 1 H), 7.04 (dd, *J* = 8.4, 1.6 Hz, 1 H), 6.46 (d, *J* = 3.2 Hz, 1 H), 5.29 (s, 2 H), 3.04 (d, *J* = 13.4 Hz, 1 H), 2.90 (d, *J* = 13.4 Hz, 1 H), 2.62–2.66 (m, 2 H), 2.06–2.13 (m, 1 H), 1.67–1.75 (m, 1 H), 1.26 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 148.5, 148.1, 137.8, 135.3, 129.8, 128.9, 128.7, 128.3, 128.1, 128.0, 127.7, 127.0, 126.5, 126.4, 126.2, 126.0, 124.8, 122.5, 109.1, 101.5, 88.4, 85.4, 50.2, 48.5, 38.8, 36.9, 27.1; IR (film) 2924, 1485, 1447 cm^{-1}; MS (ESI+) 458.2478 (458.2478 calcd for $C_{33}H_{31}NO$, M + H^+). The enantiopurity was determined to be 96:4 er by chiral HPLC analysis (Chiralcel ADH, 25 cm x 4.6 mm, 1% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 11.6 and 30.3 min).

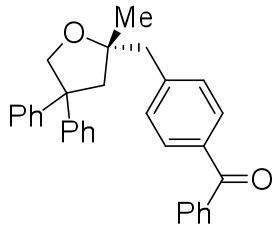


(+)-(*S*)-4-{4-[(2-Methyl-5,5-diphenyltetrahydrofuran-2-yl)methyl]phenyl}morpholine (2m). The general procedure was employed for the coupling of 4-methyl-1,1-diphenylpent-4-en-1-ol (51 mg, 0.20 mmol) and 4-(4-bromophenyl)morpholine (87 mg, 0.36 mmol) using a catalyst composed of $Pd_2(dba)_3$ (3.7 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of 90 °C and a reaction time of 12 h in 2 mL of dioxane. This procedure afforded the title compound (68.0 mg, 82%) as a colorless solid, mp 143–145 °C : $[\alpha]^{23}_D$ +28.8 (*c* 6.70, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (dd, *J* = 12.8, 7.5 Hz, 4 H), 7.22–7.33 (m, 4 H), 7.13–7.19 (m, 2 H), 7.12 (d, *J* = 8.3 Hz, 2 H), 6.79 (d, *J* = 8.3 Hz, 2 H), 3.87 (t, *J* = 4.7 Hz, 4 H), 3.09–3.14 (m, 4 H), 2.88 (d, *J* = 13.5 Hz, 1 H), 2.74 (d, *J* = 13.5 Hz, 1 H), 2.59–2.68 (m, 2 H), 1.98–2.03 (m, 1 H), 1.69–1.74 (m, 1 H), 1.23 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.7, 148.4, 148.0, 131.3,

130.3, 128.1, 128.0, 126.5, 126.5, 126.1, 125.9, 115.4, 88.4, 85.1, 67.1, 49.7, 47.6, 38.7, 37.0, 27.0; IR (film) 2966, 1515, 1446 cm^{-1}; MS (ESI+) 414.2427 (414.2428 calcd for $C_{28}H_{31}NO_2$, M + H^+). The enantiopurity was determined to be 93:7 er by chiral HPLC analysis (Chiralcel ADH, 25 cm x 4.6 mm, 2% IPA/Hexanes, 1.00 mL/min, λ 210 nm, RT= 8.7 and 10.7 min).



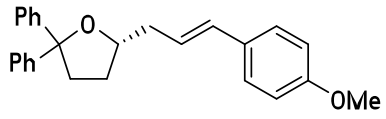
(+)-(*S*)-4-[(2-Methyl-5,5-diphenyltetrahydrofuran-2-yl)methyl]benzonitrile (2n). The general procedure was employed for the coupling of 4-methyl-1,1-diphenylpent-4-en-1-ol (51 mg, 0.20 mmol) and 4-bromobenzonitrile (66 mg, 0.36 mmol) using a catalyst composed of $Pd_2(dba)_3$ (3.7 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of 90 °C and a reaction time of 12 h in 2mL of dioxane. This procedure afforded the title compound (27.0 mg, 38%) as a colorless solid, mp 100–104 °C: $[\alpha]^{23}_D$ +23.9 (*c* 2.00, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (d, *J* = 8.1 Hz, 2 H), 7.38–7.42 (m, 4 H), 7.27–7.30 (m, 4 H), 7.22–7.25 (m, 2 H), 7.13–7.22 (m, 2 H), 2.90–2.95 (d, *J* = 13.5 Hz, 1 H), 2.79–2.85 (d, *J* = 13.5 Hz, 1 H), 2.55–2.67 (m, 2 H), 1.96–2.02 (m, 1 H), 1.79–1.86 (m, 1 H), 1.23 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 148.1, 147.4, 144.2, 131.7, 131.4, 128.2, 128.2, 126.8, 126.6, 126.0, 125.7, 119.4, 110.1, 88.8, 84.3, 48.5, 38.2, 37.5, 27.3; IR (film) 2925, 2223, 1607 cm^{-1}; MS (ESI+) 376.1670 (376.1670 calcd for $C_{25}H_{23}NO$, M + Na^+). The enantiopurity was determined to be 87:13 er by chiral HPLC analysis (Chiralcel ADH, 25 cm x 4.6 mm, 2% IPA/Hexanes, 1.00 mL/min, λ 195 nm, RT= 7.5 and 8.4 min).



(+)-(*R*)-{4-[(2-Methyl-4,4-diphenyltetrahydrofuran-2-yl)methyl]phenyl}(phenyl)methanone (6). The general procedure was employed for the coupling of 4-methyl-2,2-diphenylpent-4-en-1-ol[3] (51 mg, 0.20 mmol) and (4-bromophenyl)(phenyl)methanone (94 mg, 0.36 mmol) using a catalyst composed of $Pd_2(dba)_3$ (3.7 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of 90 °C, and a reaction time of 12 h in 2mL of dioxane. This procedure afforded the title compound (73 mg, 84%) as a light yellow oil. $[\alpha]^{23}_D$= +0.01 (*c* 5.9, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57–7.62 (m, 1 H), 7.46–7.52 (m, 2 H), 7.26–7.36 (m, 11 H), 7.16–7.22 (m, 2 H), 4.53 (d, *J* = 9.5 Hz, 1 H), 4.39 (d, *J* = 9.5 Hz, 1 H), 2.90 (d, *J* = 13.2 Hz, 1 H), 2.81 (d, *J* = 12.7 Hz, 1 H), 2.73 (d, *J* = 13.2 Hz, 1 H), 2.61 (d, *J* = 12.7 Hz, 1 H), 1.12 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.7, 146.5, 143.4, 138.0, 135.7, 132.4, 130.6, 130.1, 130.0, 128.6, 128.5, 128.4, 127.3, 126.4, 126.4, 83.7, 75.5, 56.5, 50.3, 47.8, 26.9; IR (film) 2926.7, 2247, 1654, 1276 cm^{-1}; MS (ESI+) 433.2164 (433.2162 calcd for $C_{31}H_{28}O_2$, M + H^+). The enantiopurity was determined to be 51:49 er by chiral HPLC analysis (Chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 15.6 and 21.3 min).

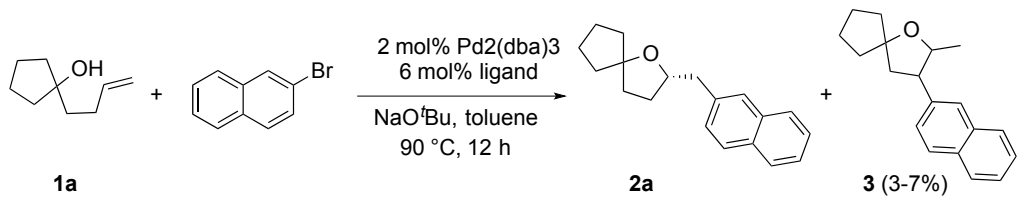
Determination of absolute configuration:

Product **2i** was synthesized according to general procedure D. The optical rotation of this compound ($[\alpha]^{23}_{D}$ +4.54 (*c* 0.22, $CHCl_3$)); was compared with that in the literature[9] (lit$[\alpha]^{23}_{D}$ +8.30 (*c* 0.6, $CHCl_3$)). Both compounds were dextrorotatory, thus **2i** was assigned the (*S*) configuration on this basis.

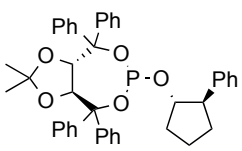


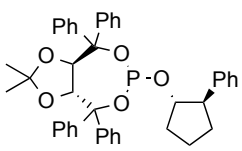
(+)-(*S,E*)-5-[3-(4-Methoxyphenyl)allyl]-2,2-diphenyltetrahydrofuran (2i): The general procedure was employed for the coupling of 1,1-diphenylpent-4-en-1-ol (48 mg, 0.20 mmol) and (*E*)-1-(2-bromovinyl)-4-methoxybenzene (85 mg, 0.40 mmol) using a catalyst composed of 2 mol % $Pd_2(dba)_3$ (3.6 mg, 0.004 mmol) and **L7** (7.5 mg, 0.010 mmol), a reaction temperature of 90 °C and a reaction time of 12 h. This procedure afforded the title compound (14 mg, 18 %) as a colorless oil. $[\alpha]^{23}_{D}$ +4.54 (*c* 0.22, $CHCl_3$); lit[9]$[\alpha]^{23}_{D}$ +8.30 (*c* 0.6, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 7.5 Hz, 4 H), 7.33–7.23 (m, 6 H), 7.22–7.15 (m, 2 H), 6.83 (d, *J* = 8.8 Hz, 2 H), 6.40 (d, *J* = 16.0 Hz, 1 H), 6.83 (dt, *J* = 15.7, 7.0 Hz, 1 H), 4.31–4.21 (m, 1 H), 3.80 (s, 3 H), 2.70–2.58 (m, 2 H), 2.57–2.40 (m, 2 H), 2.06–1.86 (m, 1 H), 1.81–1.70 (m, 1 H). Other spectroscopic data matched that of the literature.[9] The enantiopurity was determined to be 79:21 er by chiral HPLC analysis (Chiralcel ADH, 25 cm x 4.6 mm, 0.5% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 17.5 and 18.8 min).

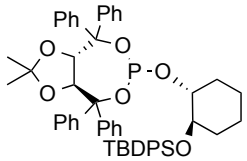
Screen of Chiral TADDOL-Derived Phosphite Ligands:

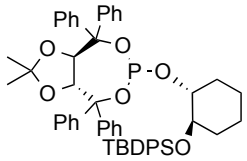


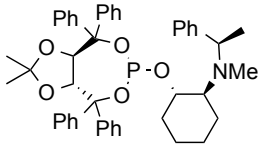
1a **2a** **3** (3-7%)

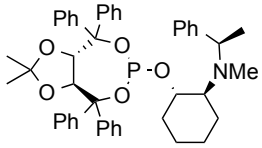

50%, 73:27 er

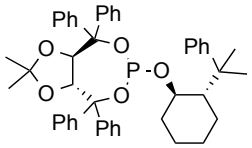

43%, 32:68

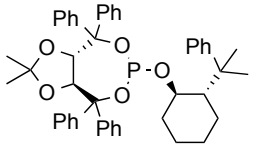

55%, 65:35 er

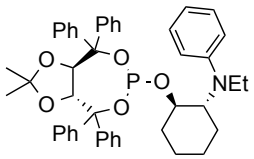

50%, 33:67

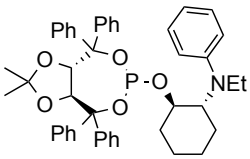

75%, 17:83

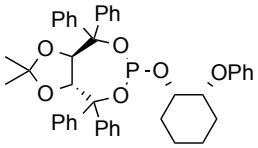

58%, 82:18

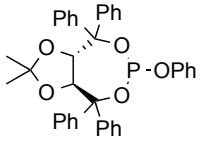

48%, 18:82

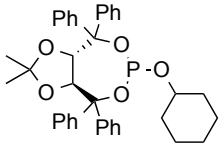

58%, 82:18

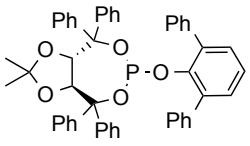

55%, 14:86

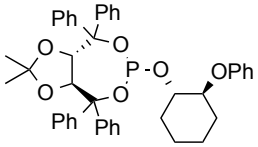

75%, 85:15 er

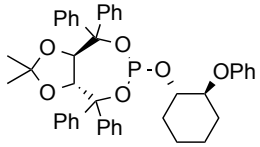

25%, 27:73

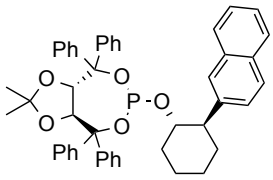

25%, 27:73 er

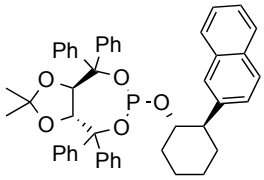

52%, 59:41 er

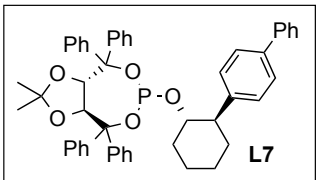

20%, 66:33 er


52%, 79:21

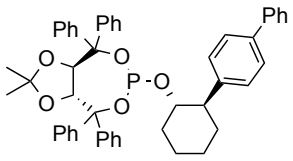

46%, 41:59


58%, 86:14


70%, 17:83


L7

60%, 88:12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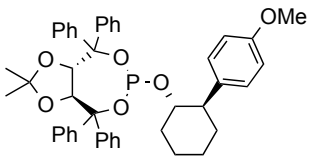
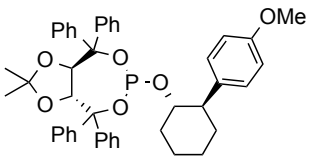
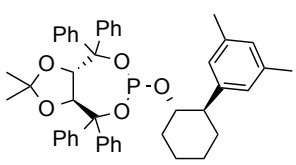
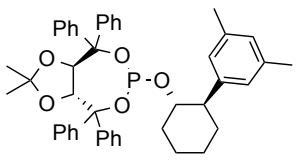

62%, 86:14


70%, 15:85


70%, 86:14


73%, 14:86

References

[1] M. B. Hay, A. R. Hardin, J. P. Wolfe, *J. Org. Chem.* **2005**, *70*, 3099.

[2] B. E. Carpenter, I. R. Hunt, B. A. Keay, *Tetrahedron: Asymmetry* **1996**, *7*, 3107.

[3] Y. Jeong, D. -Y. Kim, Y. Choi, J. -S. Ryu, *Org. Biomol. Chem*, **2011**, *9*, 374.

[4] A. Alexakis, J. Burton, J. Vastra, C. Benhaim, X. Fournioux, A. van den Heuvel, J.-M. Levêque, F. Mazé, S. Rosset, *Eur. J. Org. Chem.* **2000**, 4011.

[5] M. Hatano, O. Ito, S. Suzuki, K. Ishihara, *J. Org. Chem.* **2010**, *75*, 5008.

[6] Z. Cai, N. Yongpruksa, M. Harmata, *Org. Lett.* **2012**, *14*, 1661.

[7] M. B. Hay, J. P. Wolfe, *J. Am. Chem. Soc.* **2005**, *127*, 16468.

[8] A. Faulkner, J. S. Scott, J. F. Bower, *Chem. Comm.* **2013**, *49*, 1521.

[9] M. T. Bovino, T. W. Liwosz, N. E. Kendel, Y. Miller, N. Tyminska, E. Zurek, S. R. Chemler, *Angew. Chem.* **2014**, *126*, 6501; *Angew. Chem. Int. Ed.* **2014**, *53*, 6383.

Agilent Technologies

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Date collected 2016-05-08
Pulse sequence PROTON
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Operator garletsz
Study owner garletsz
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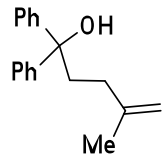

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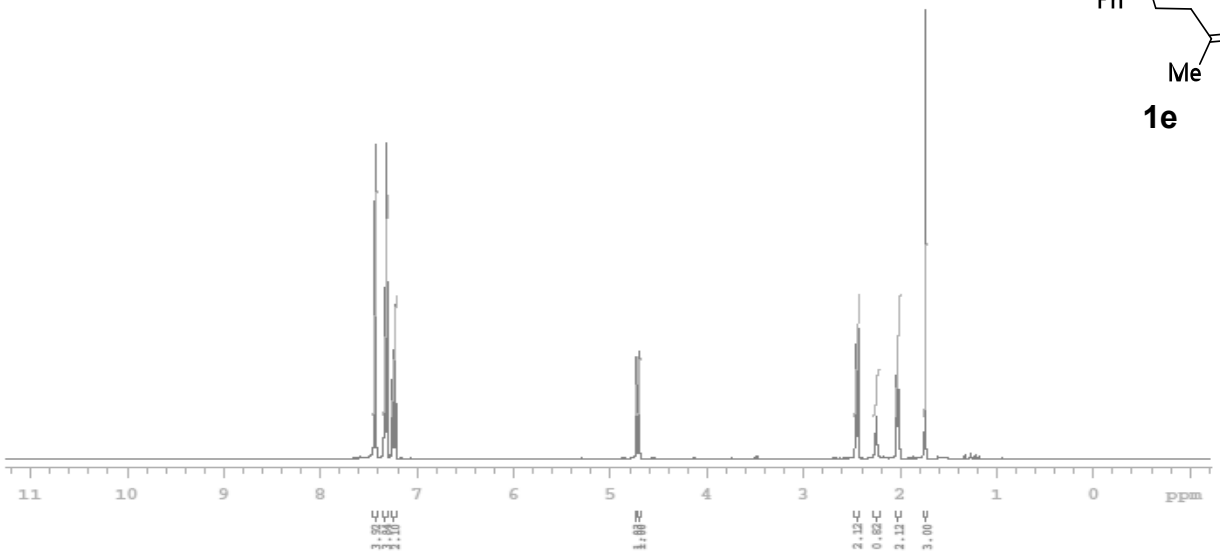

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Plot date 2015-05-14

Agilent Technologies

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Date collected 2016-05-08
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Solvent odcl3
Temperature 24
Operator garletsz
Study owner garletsz
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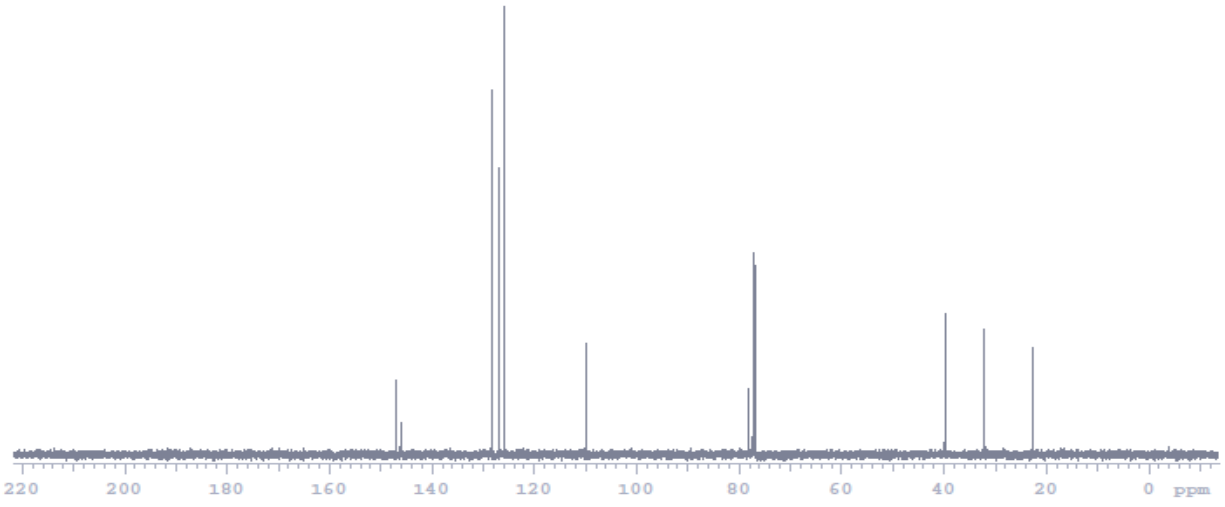

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Agilent Technologies

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Solvent odcl3
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Study owner garletsz
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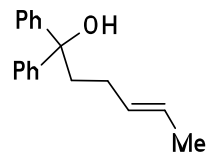

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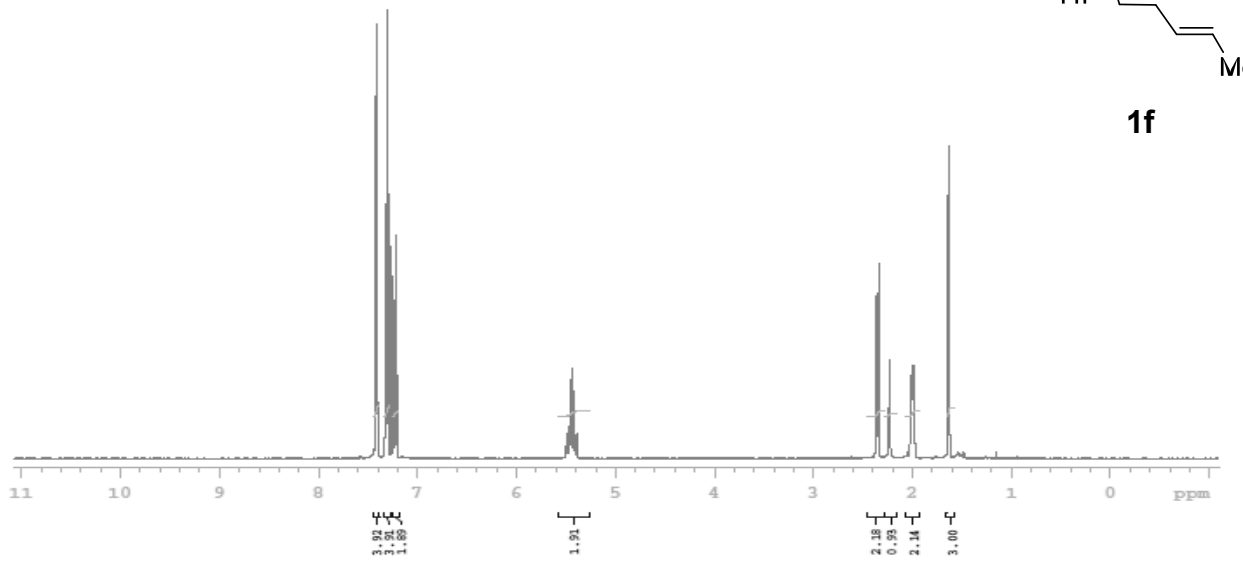

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Agilent Technologies

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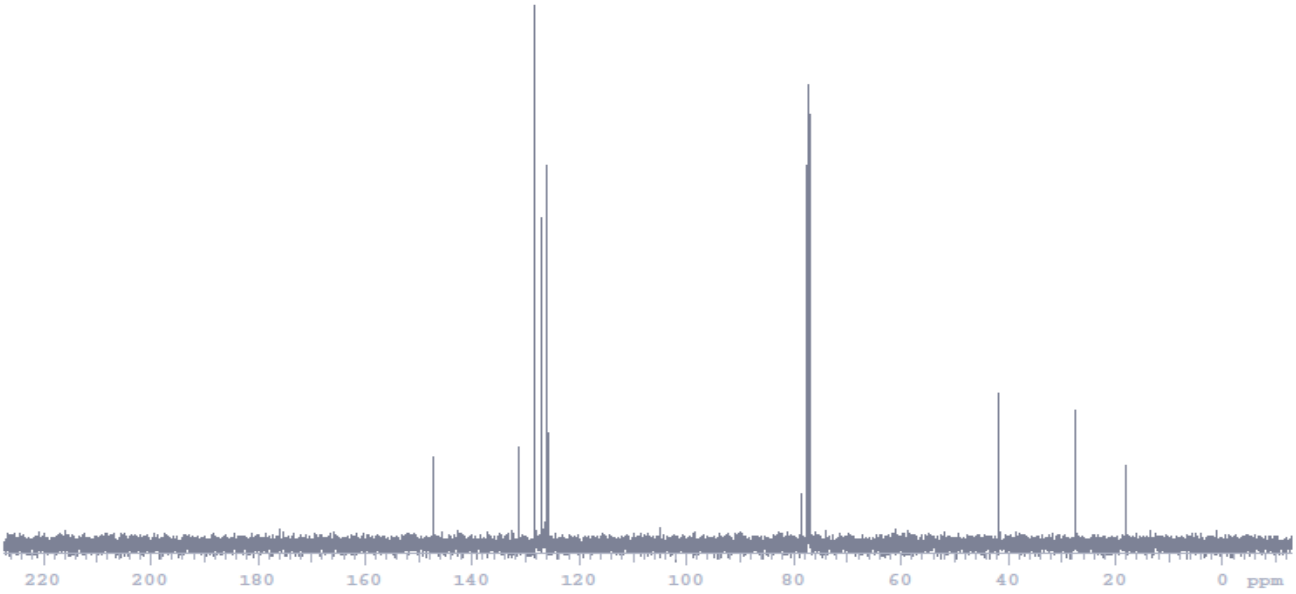

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Temperature 24
Operator garletsz
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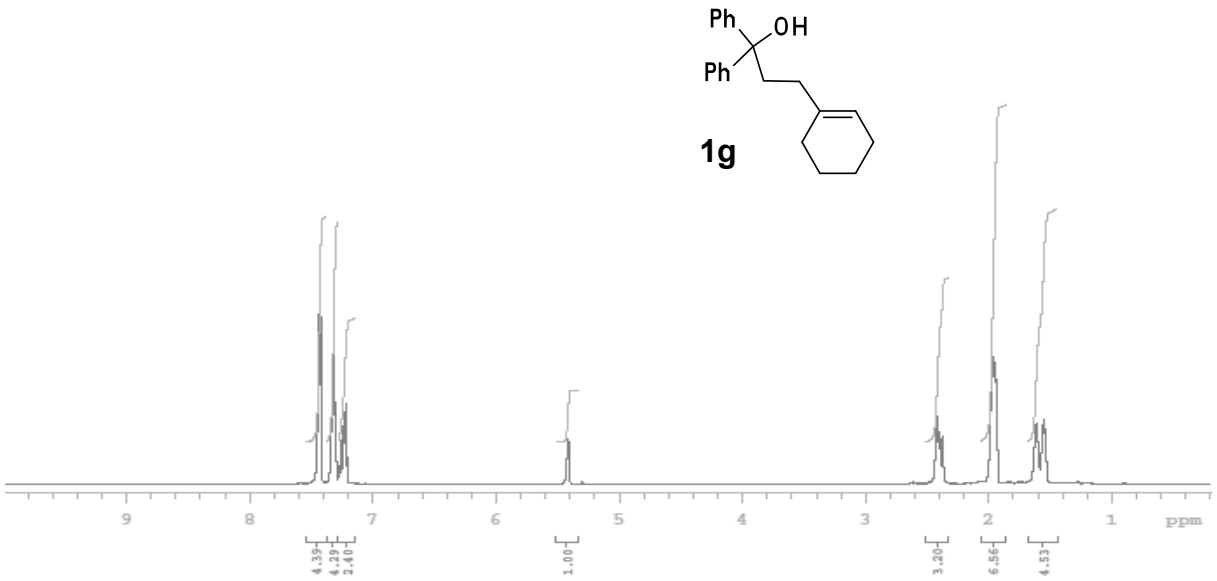

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Study owner garletsz
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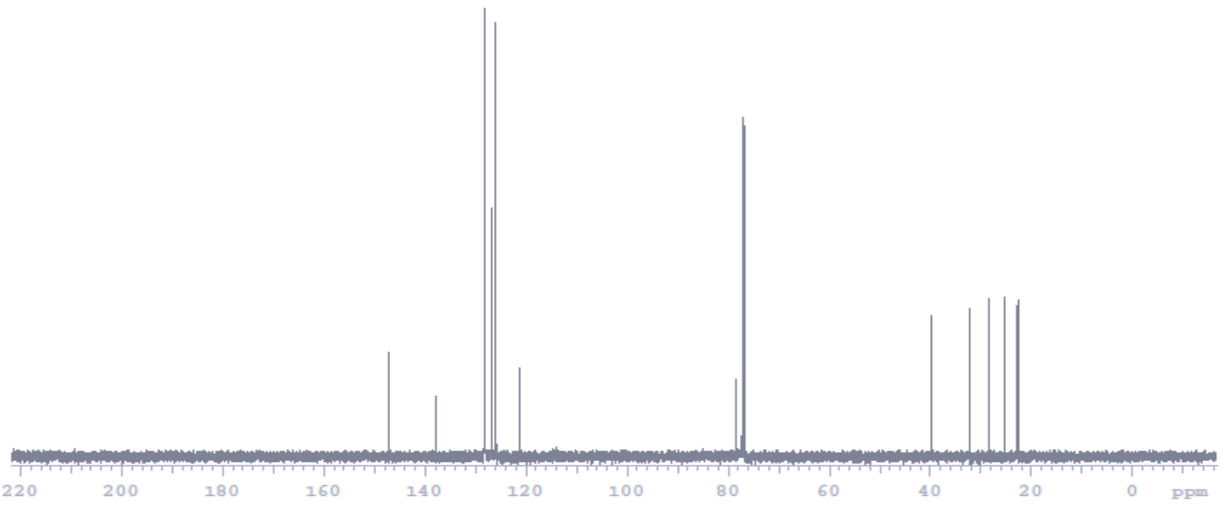

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Plot date 2015-05-14

Agilent Technologies

Sample Name	Pulse sequence PROTON	Temperature 23	Study owner bahopkl
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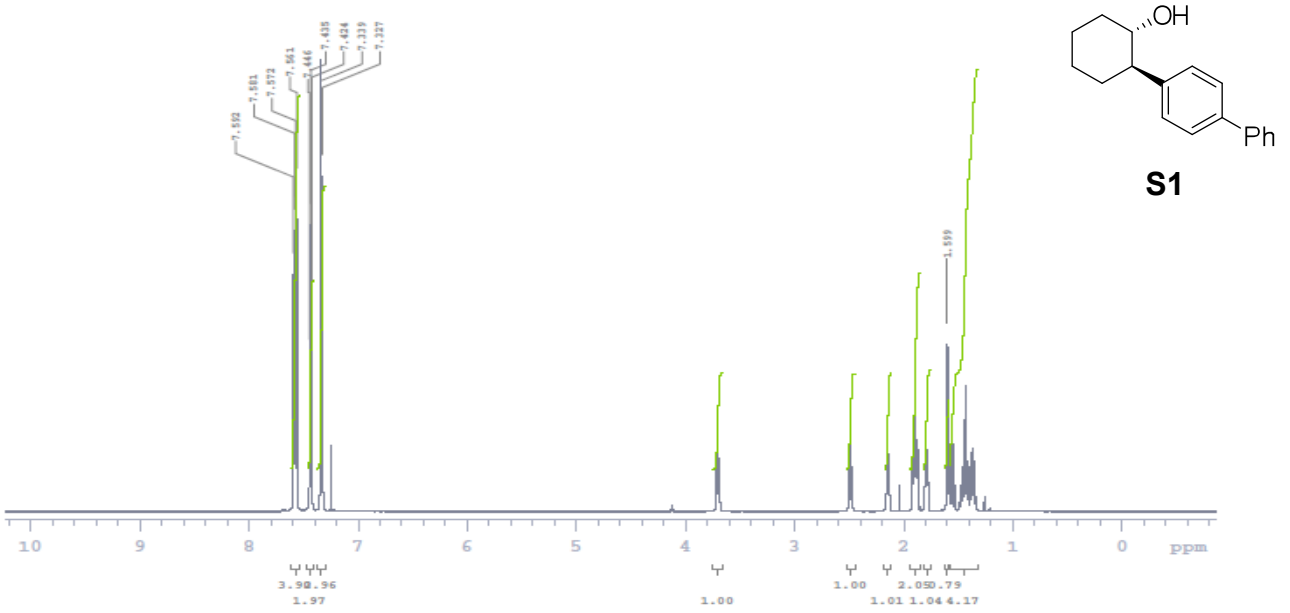

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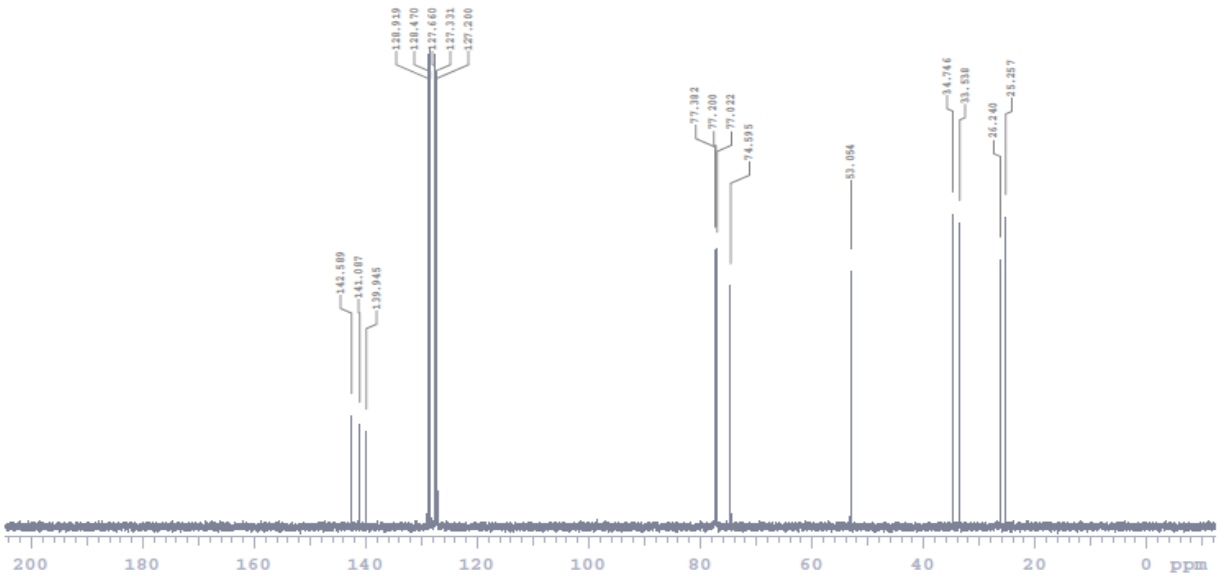
Plot date 2015-03-16

Agilent Technologies

Sample Name	Pulse sequence CARBON	Temperature 23	Study owner bahopkl
Date collected 2016-03-16	Solvent odcl3	Operator bahopkl	Printed from dy.chem.lsa.umich.edu-vnmrs600



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Plot date 2015-03-16

==== Shimadzu LCsolution Analysis Report ====

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Sample ID :
Tray# : 1
Vail # : 1
Injection Volume : 1 uL
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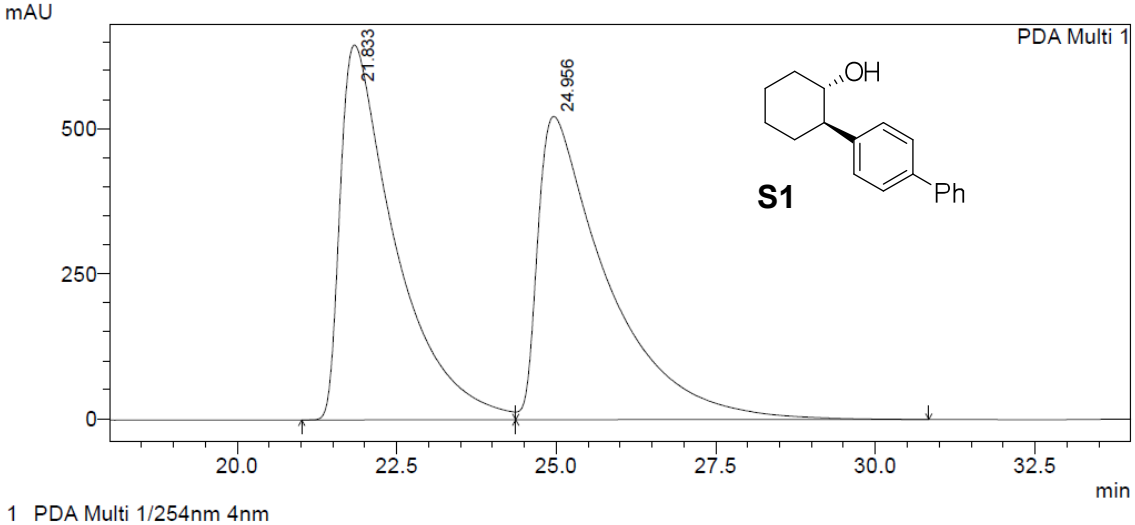

1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
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2	24.956	39788133	522646	50.561	44.726
Total		78693453	1168542	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

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Sample ID	:
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Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-cyclohexanol-BAH-9-62-1.00mL_min-4.00%IPA-OJH-3days.lcd
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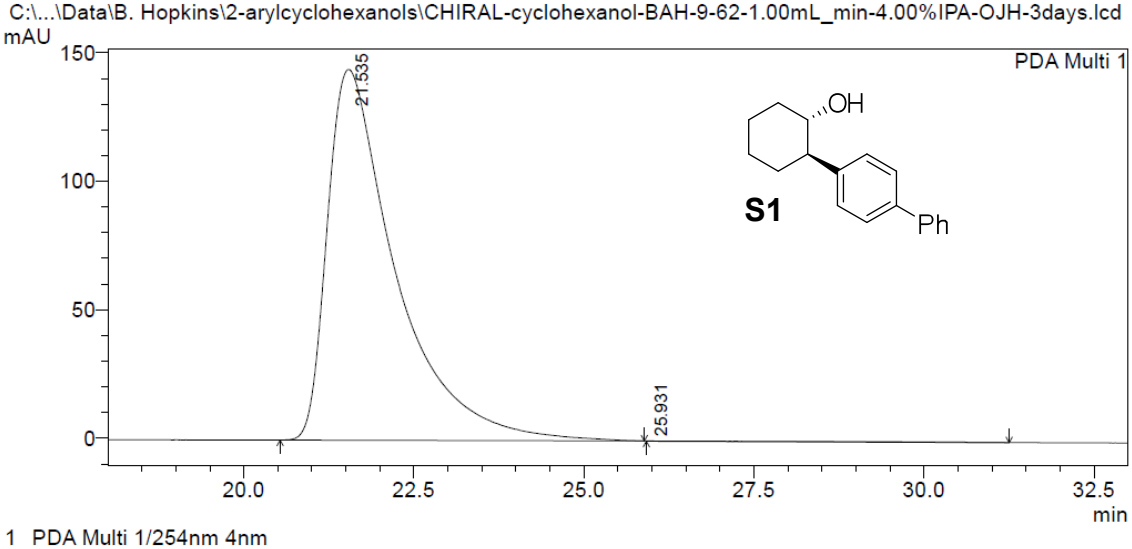

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PeakTable

PDA Ch1 254nm 4nm

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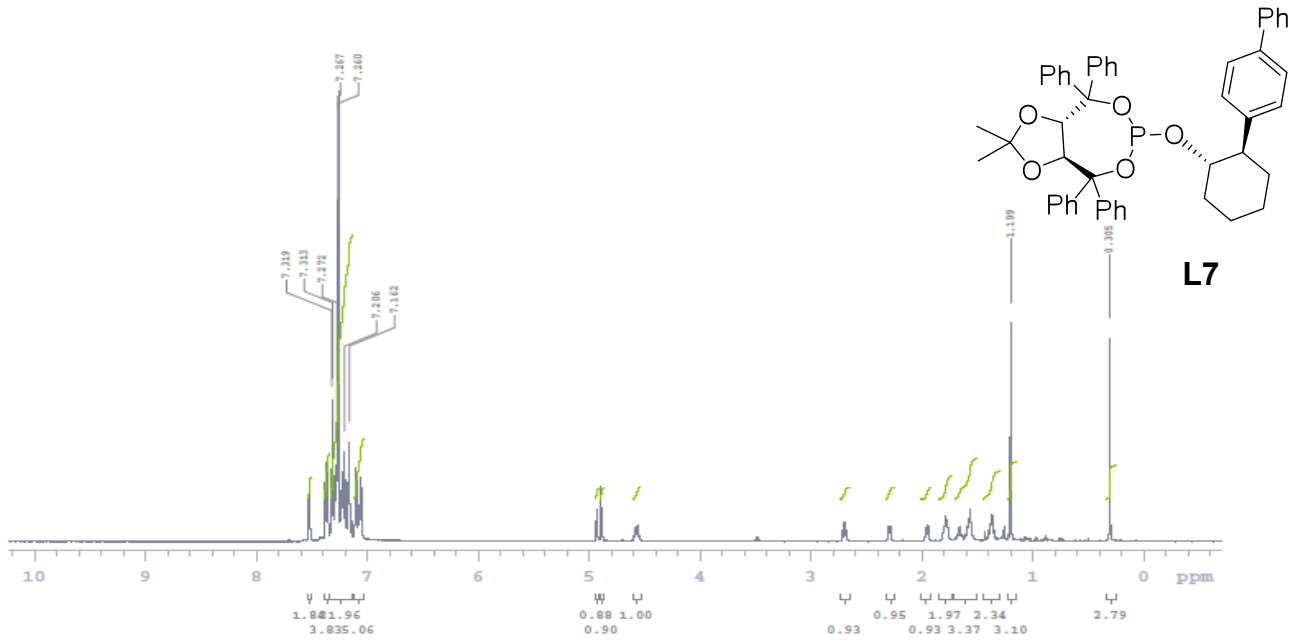
Agilent Technologies

Sample Name		Pulse sequence	PROTON	Temperature	23	Study owner	bahopkl
Date collected	2015-03-16	Solvent	cdcl3	Operator	bahopkl	Printed from	dy.chem.lsa.umich.edu-vnmrs500

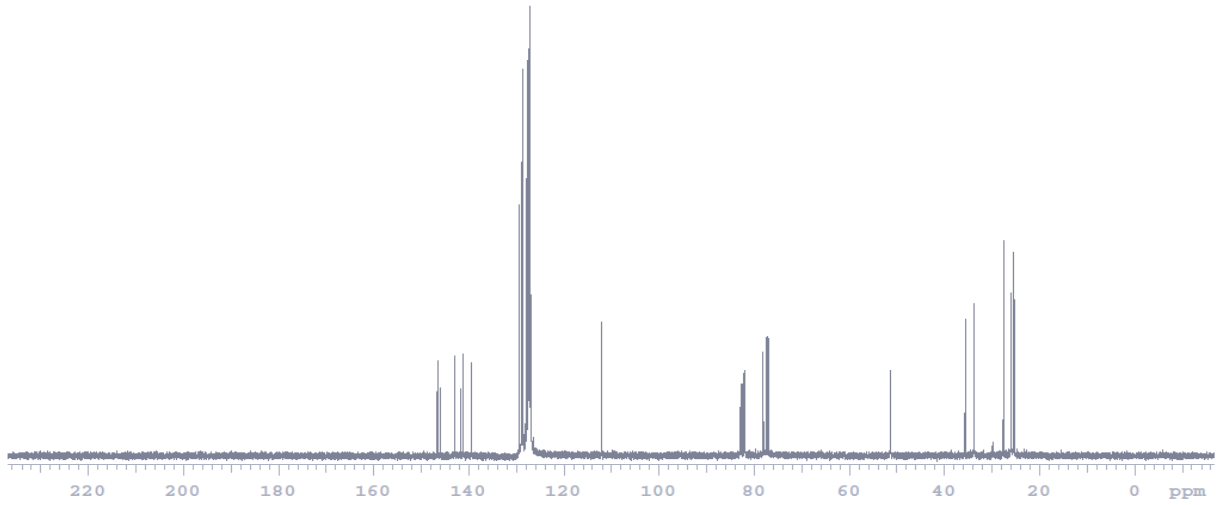


Data file /misc/Ytterbium/bahopkl/vnmrsys/data/BAH-8-21proton.fid

Plot date 2015-03-16

Agilent Technologies

Sample Name		Pulse sequence	CARBON	Temperature	22	Study owner	bahopkl
Date collected	2015-03-16	Solvent	cdcl3	Operator	bahopkl	Printed from	kr.chem.lsa.umich.edu-vnmrs500

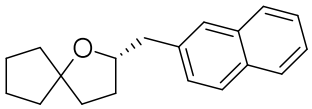


Data file /misc/Ytterbium/bahopkl/vnmrsys/data/BAH-8-21carbon.fid

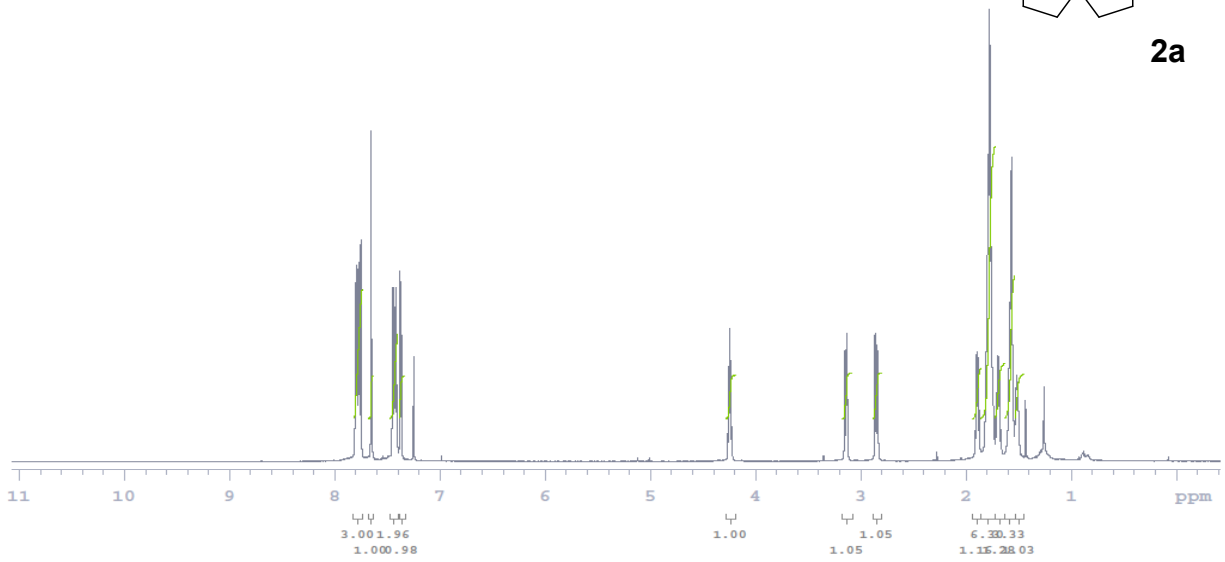
Plot date 2015-05-04

Agilent Technologies

Sample Name	Pulse sequence PROTON	Temperature 22	Study owner bahopki
Date collected 2015-03-21	Solvent cdcl3	Operator bahopki	Printed from kr.chem.lsa.umich.edu-vnmrs500



2a

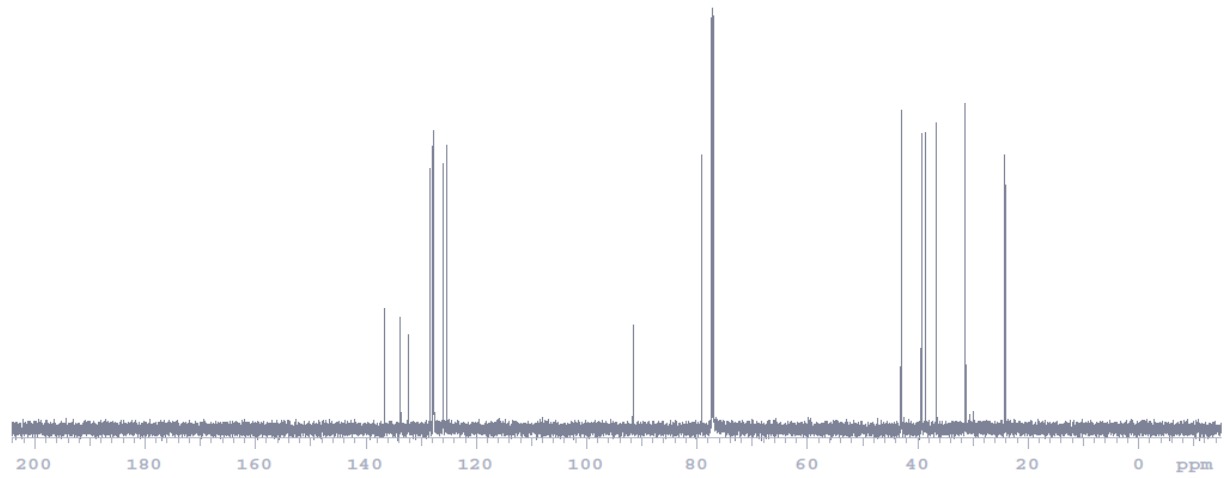


Data file /misc/Ytterbium/bahopki/vnmrsys/data/BAH-9-119proton.fid

Plot date 2015-05-04

Agilent Technologies

Sample Name	Pulse sequence CARBON	Temperature 22	Study owner bahopki
Date collected 2015-03-21	Solvent cdcl3	Operator bahopki	Printed from kr.chem.lsa.umich.edu-vnmrs500



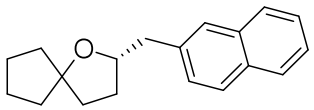
Data file /misc/Ytterbium/bahopki/vnmrsys/data/BAH-9-119carbon.fid

Plot date 2015-05-04

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Sprio-RAC-BAH-8-92(1)-1.00%IPA-1.00mL_min-OJH1.lcd

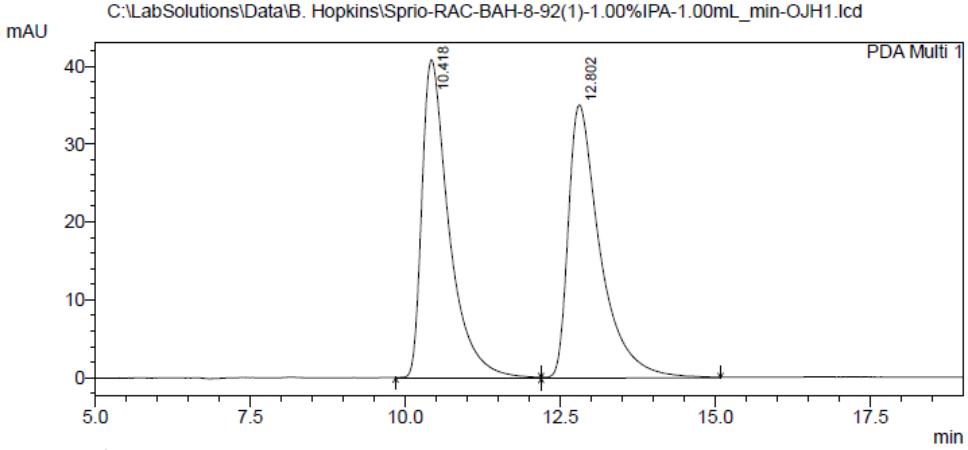
Acquired by	: Admin
Sample Name	: Sprio-RAC-BAH-8-92(1)-1.00%IPA-1.00mL_min-OJH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: Sprio-RAC-BAH-8-92(1)-1.00%IPA-1.00mL_min-OJH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/16/2014 11:44:43 AM
Data Processed	: 10/16/2014 12:54:46 PM



2a

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Sprio-RAC-BAH-8-92(1)-1.00%IPA-1.00mL_min-OJH1.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.418	1241027	40824	49.700	53.782
2	12.802	1256030	35082	50.300	46.218
Total		2497057	75905	100.000	100.000

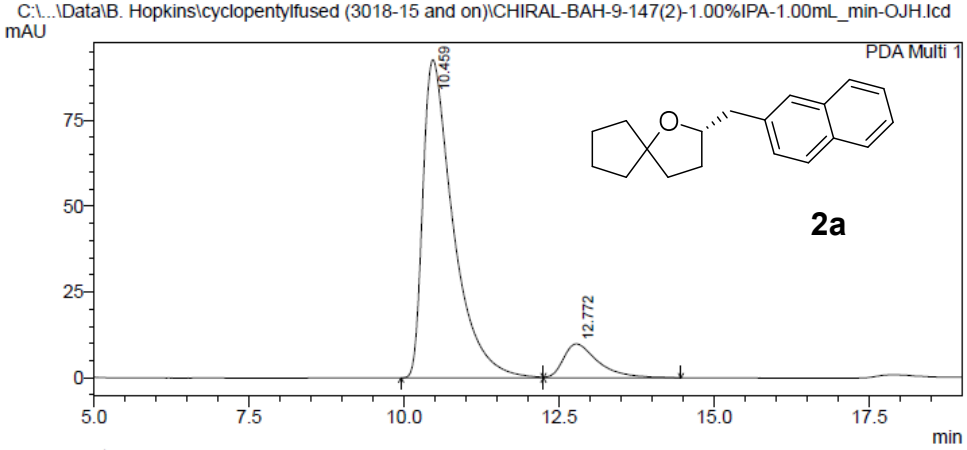
==== Shimadzu LCsolution Analysis Report ====

C:\...\Data\B. Hopkins\cyclopentylfused (3018-15 and on)\CHIRAL-BAH-9-147(2)-1.00%IPA-1.00mL_min-OJH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-9-147(2)-1.00%IPA-1.00mL_min-OJH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-9-147(2)-1.00%IPA-1.00mL_min-OJH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/10/2015 10:45:11 PM
Data Processed	: 4/10/2015 11:20:21 PM

<Chromatogram>

C:\...\Data\B. Hopkins\cyclopentylfused (3018-15 and on)\CHIRAL-BAH-9-147(2)-1.00%IPA-1.00mL_min-OJH.lcd



1 PDA Multi 1/254nm 4nm

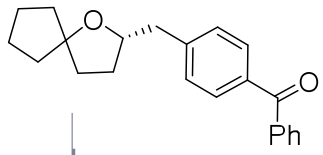
PeakTable

PDA Ch1 254nm 4nm

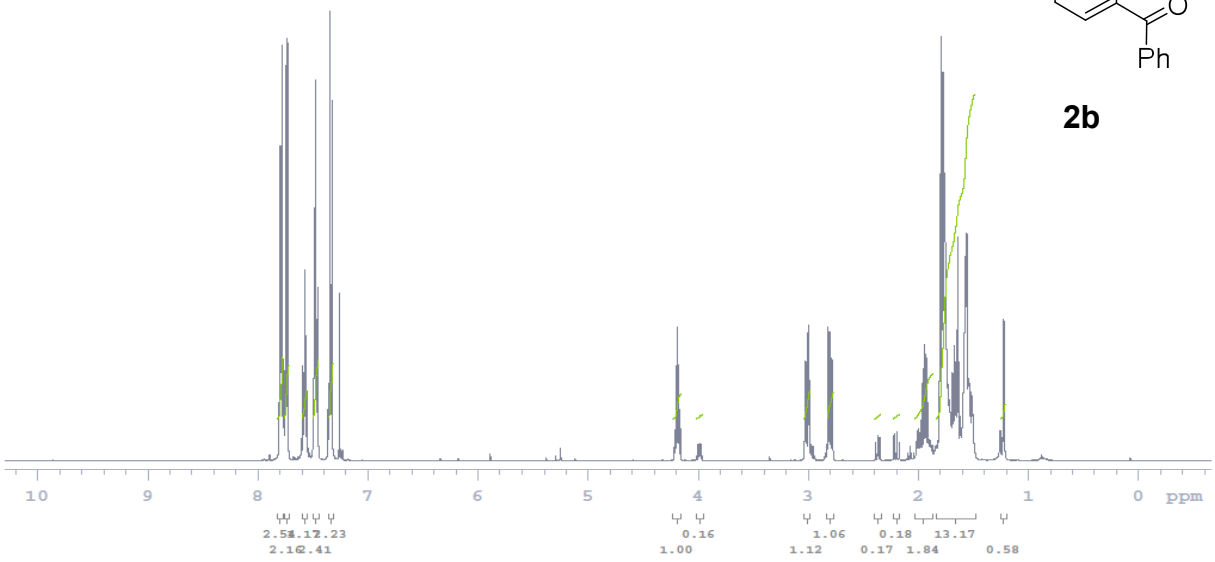
Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.459	3154717	92708	89.034	90.370
2	12.772	388542	9879	10.966	9.630
Total		3543259	102587	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2015-05-08
Pulse sequence PROTON
Solvent cdcl3
Temperature 25
Operator bahopki
Study owner bahopki
Printed from te.chem.lsa.umich.edu-vnmrs500



2b

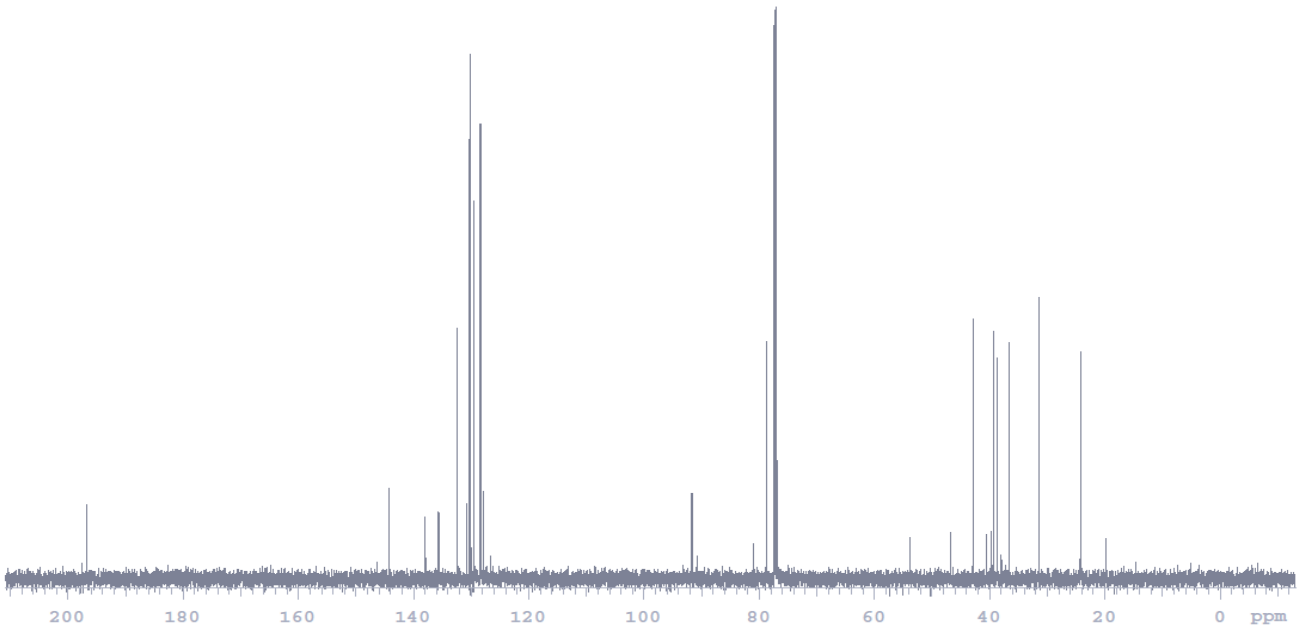


Data file /home/bahopki/vnmrsys/data/BAH-9-180-3proton.fid

Plot date 2015-05-08

Agilent Technologies

Sample Name
Date collected 2015-05-08
Pulse sequence CARBON
Solvent cdcl3
Temperature 25
Operator bahopki
Study owner bahopki
Printed from te.chem.lsa.umich.edu-vnmrs500



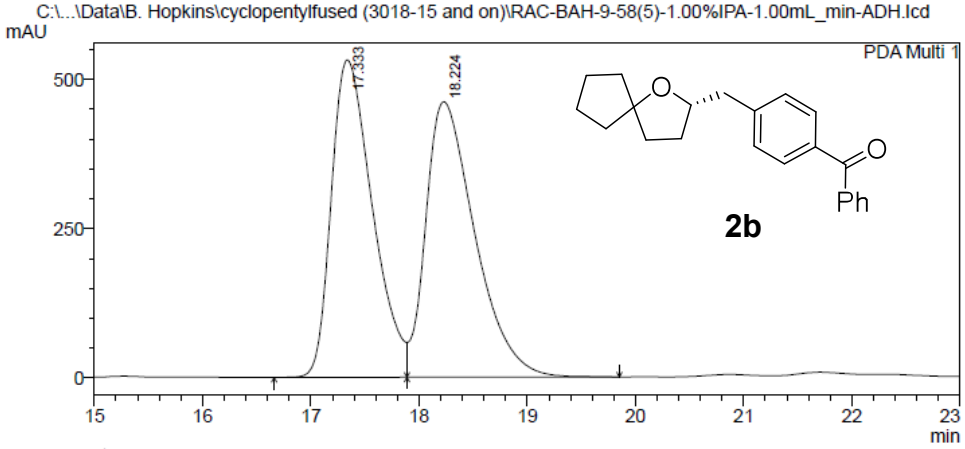
==== Shimadzu LCsolution Analysis Report ====

C:\...\Data\B. Hopkins\cyclopentylfused (3018-15 and on)\RAC-BAH-9-58(5)-1.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-9-58(5)-1.00%IPA-1.00mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-9-58(5)-1.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/8/2015 1:22:52 PM
Data Processed	: 5/8/2015 1:50:25 PM

<Chromatogram>

C:\...\Data\B. Hopkins\cyclopentylfused (3018-15 and on)\RAC-BAH-9-58(5)-1.00%IPA-1.00mL_min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.333	14045763	531427	49.132	53.520
2	18.224	14541794	461515	50.868	46.480
Total		28587558	992942	100.000	100.000

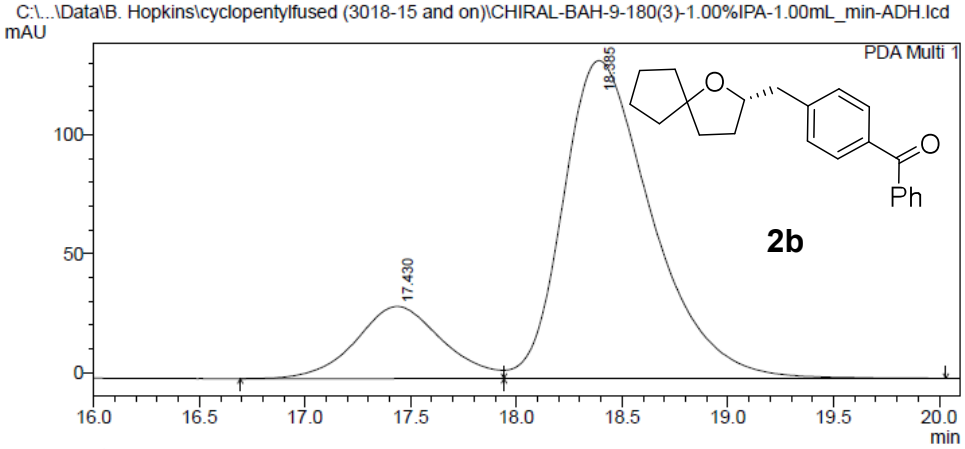
==== Shimadzu LCsolution Analysis Report ====

C:\...\Data\B. Hopkins\cyclopentylfused (3018-15 and on)\CHIRAL-BAH-9-180(3)-1.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-9-180(3)-1.00%IPA-1.00mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-9-180(3)-1.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/8/2015 2:54:37 PM
Data Processed	: 5/8/2015 3:18:40 PM

<Chromatogram>

C:\...\Data\B. Hopkins\cyclopentylfused (3018-15 and on)\CHIRAL-BAH-9-180(3)-1.00%IPA-1.00mL_min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.430	860939	30219	18.094	18.474
2	18.385	3897330	133358	81.906	81.526
Total		4758269	163577	100.000	100.000

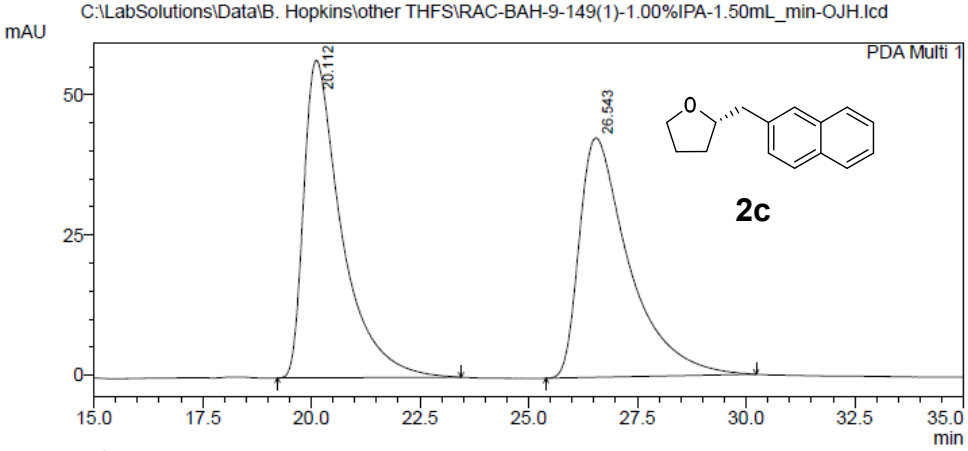
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\other THFS\RAC-BAH-9-149(1)-1.00%IPA-1.50mL_min-OJH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-9-149(1)-1.00%IPA-1.50mL_min-OJH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-9-149(1)-1.00%IPA-1.50mL_min-OJH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/12/2015 11:07:32 AM
Data Processed	: 4/12/2015 11:53:14 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\other THFS\RAC-BAH-9-149(1)-1.00%IPA-1.50mL_min-OJH.lcd



1 PDA Multi 1/230nm 4nm

PeakTable

PDA Ch1 230nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.112	3431556	56621	50.157	57.013
2	26.543	3410131	42691	49.843	42.987
Total		6841687	99312	100.000	100.000

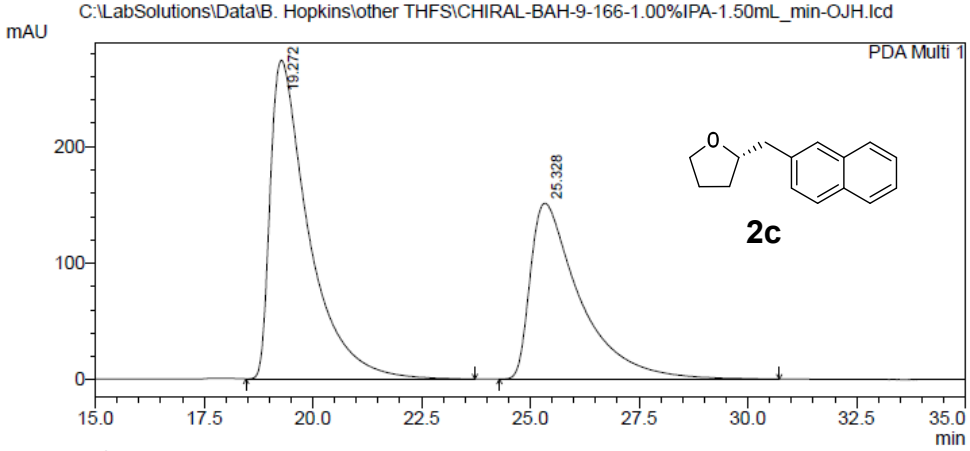
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\other THFS\CHIRAL-BAH-9-166-1.00%IPA-1.50mL_min-OJH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-9-166-1.00%IPA-1.50mL_min-OJH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-9-166-1.00%IPA-1.50mL_min-OJH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/5/2015 6:02:55 PM
Data Processed	: 5/5/2015 7:12:57 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\other THFS\CHIRAL-BAH-9-166-1.00%IPA-1.50mL_min-OJH.lcd



1 PDA Multi 1/230nm 4nm

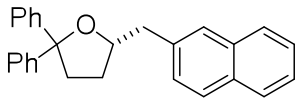
PeakTable

PDA Ch1 230nm 4nm

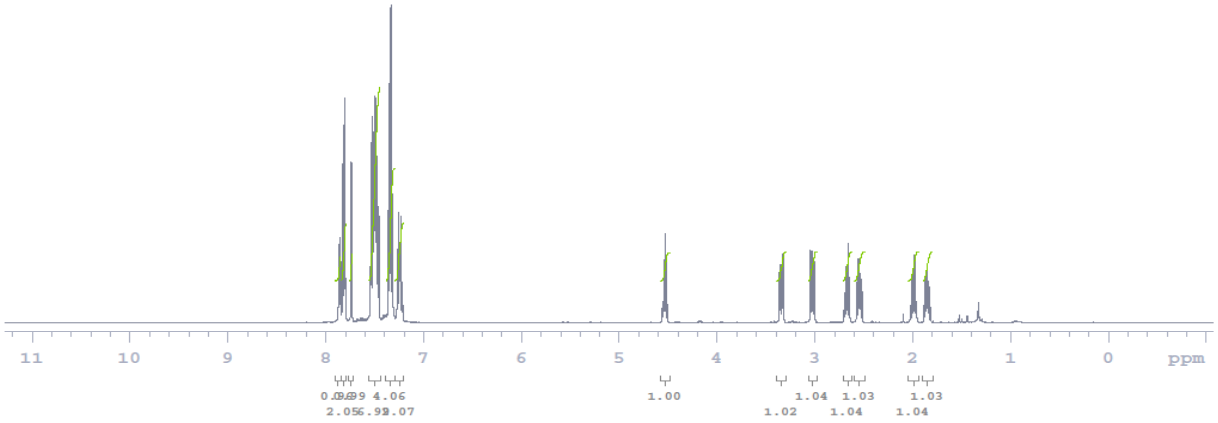
Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.272	16768556	273470	58.206	64.431
2	25.328	12040354	150966	41.794	35.569
Total		28808910	424436	100.000	100.000

Agilent Technologies

Sample Name		Pulse sequence PROTON	Temperature 25	Study owner bahopki
Date collected 2015-04-11		Solvent cdcl3	Operator bahopki	Printed from kr.chem.lsa.umich.edu-vnmrs500



2d

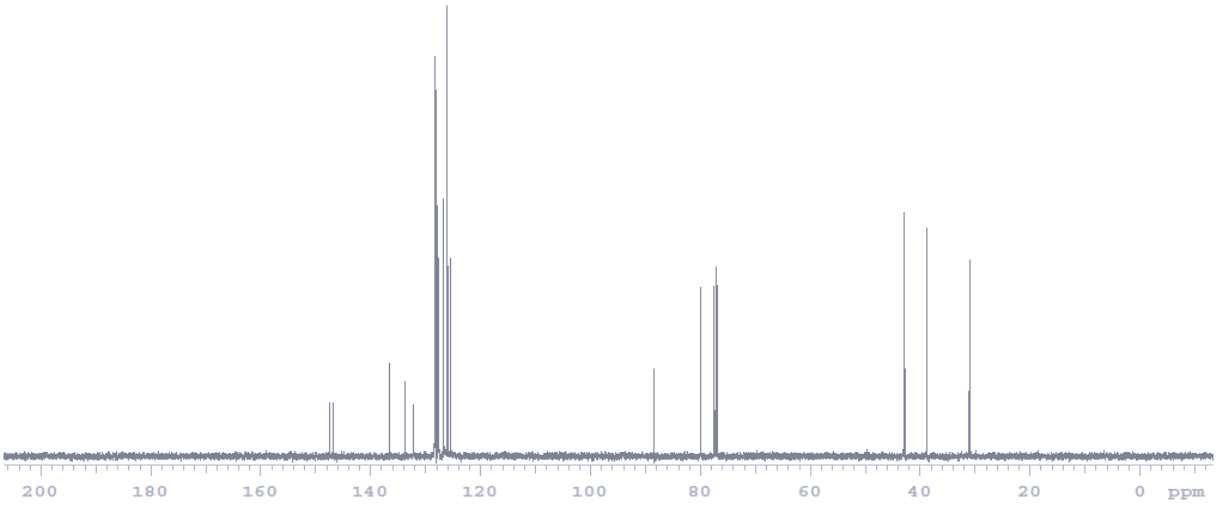


Data file /misc/Tellurium/bahopki/vnmrsys/data/BAH-9-144-1proton.fid

Plot date 2015-05-04

Agilent Technologies

Sample Name		Pulse sequence CARBON	Temperature 25	Study owner bahopki
Date collected 2015-04-11		Solvent cdcl3	Operator bahopki	Printed from kr.chem.lsa.umich.edu-vnmrs500



Data file /misc/Tellurium/bahopki/vnmrsys/data/BAH-9-144-1carbon.fid

Plot date 2015-05-04

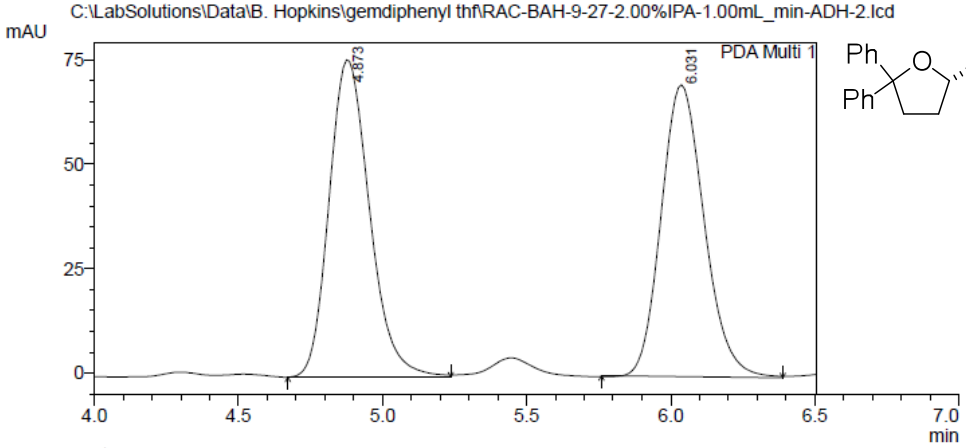
==== Shimadzu LCsolution Analysis Report ====

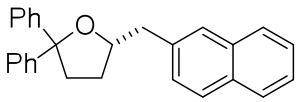
C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\RAC-BAH-9-27-2.00%IPA-1.00mL_min-ADH-2.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-9-27-2.00%IPA-1.00mL_min-ADH-2
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-9-27-2.00%IPA-1.00mL_min-ADH-2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/6/2015 11:36:45 AM
Data Processed	: 4/6/2015 11:43:16 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\RAC-BAH-9-27-2.00%IPA-1.00mL_min-ADH-2.lcd





2d

1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.873	742501	75958	50.586	52.078
2	6.031	725292	69896	49.414	47.922
Total		1467793	145854	100.000	100.000

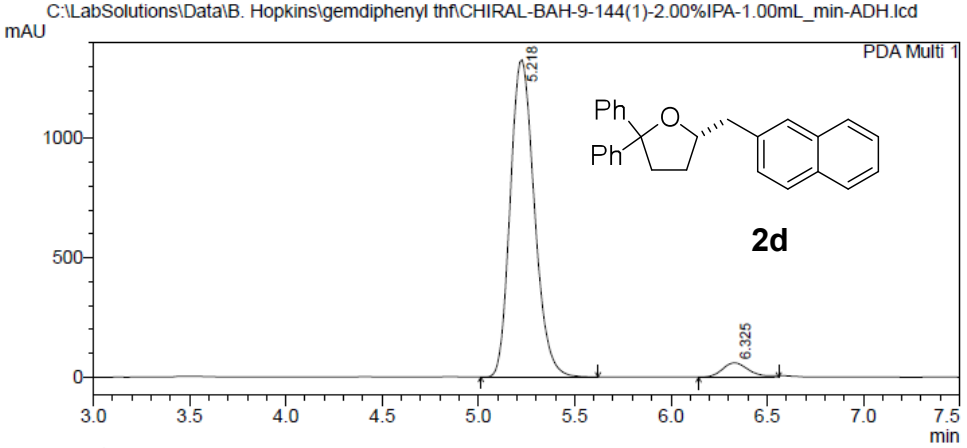
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\CHIRAL-BAH-9-144(1)-2.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-9-144(1)-2.00%IPA-1.00mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-9-144(1)-2.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/7/2015 2:28:04 PM
Data Processed	: 4/7/2015 2:43:36 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\CHIRAL-BAH-9-144(1)-2.00%IPA-1.00mL_min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.218	11628104	1328650	95.102	95.642
2	6.325	598911	60539	4.898	4.358
Total		12227015	1389189	100.000	100.000

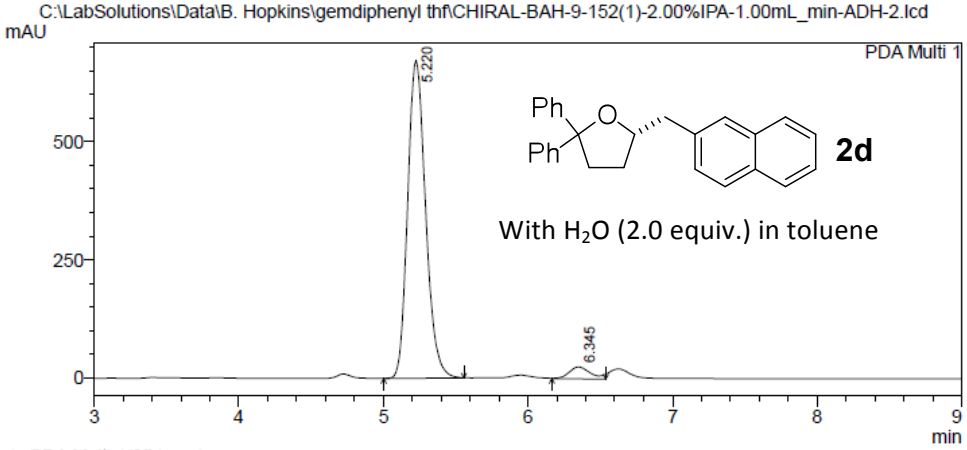
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\CHIRAL-BAH-9-152(1)-2.00%IPA-1.00mL_min-ADH-2.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-9-152(1)-2.00%IPA-1.00mL_min-ADH-2
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-9-152(1)-2.00%IPA-1.00mL_min-ADH-2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/16/2015 1:32:51 PM
Data Processed	: 4/16/2015 1:50:10 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\CHIRAL-BAH-9-152(1)-2.00%IPA-1.00mL_min-ADH-2.lcd



1 PDA Multi 1/254nm 4nm

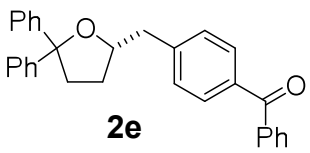
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.220	5795128	672196	95.568	96.400
2	6.345	268775	25101	4.432	3.600
Total		6063903	697297	100.000	100.000

Agilent Technologies

Sample Name	Pulse sequence PROTON	Temperature 24	Study owner bahopki
Date collected 2015-05-08	Solvent cdcl3	Operator bahopki	Printed from te.chem.lsa.umich.edu-vnmrs500



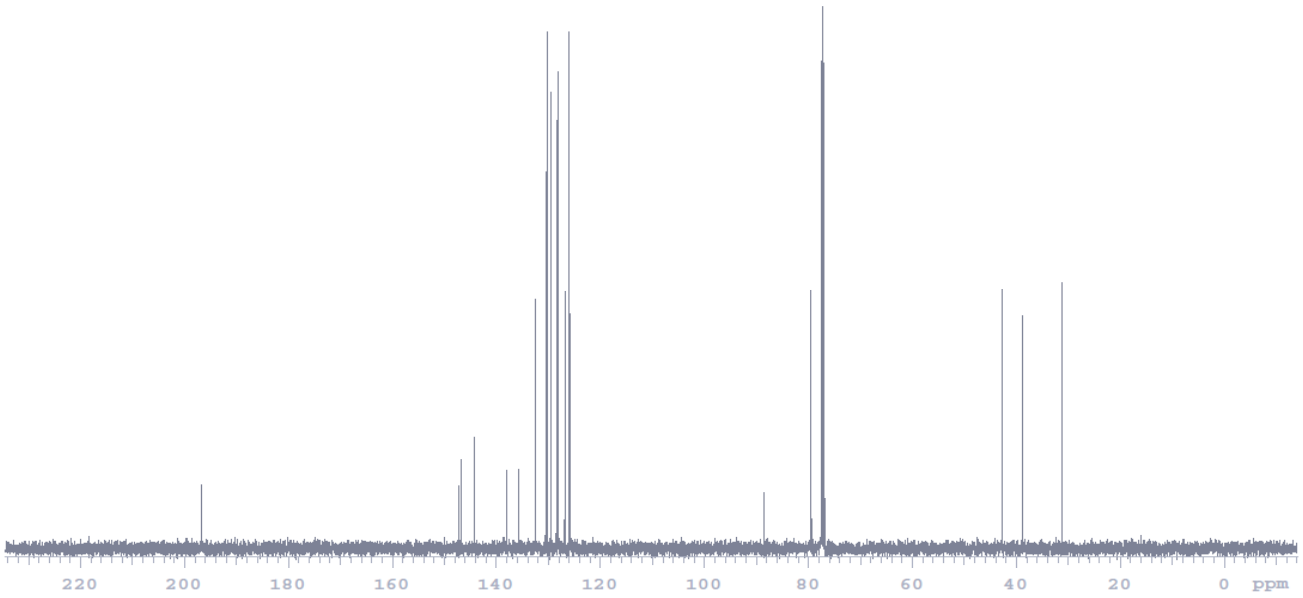
2e

Data file /home/bahopki/vnmrsys/data/BAH-9-178-2proton.fid

Plot date 2015-05-08

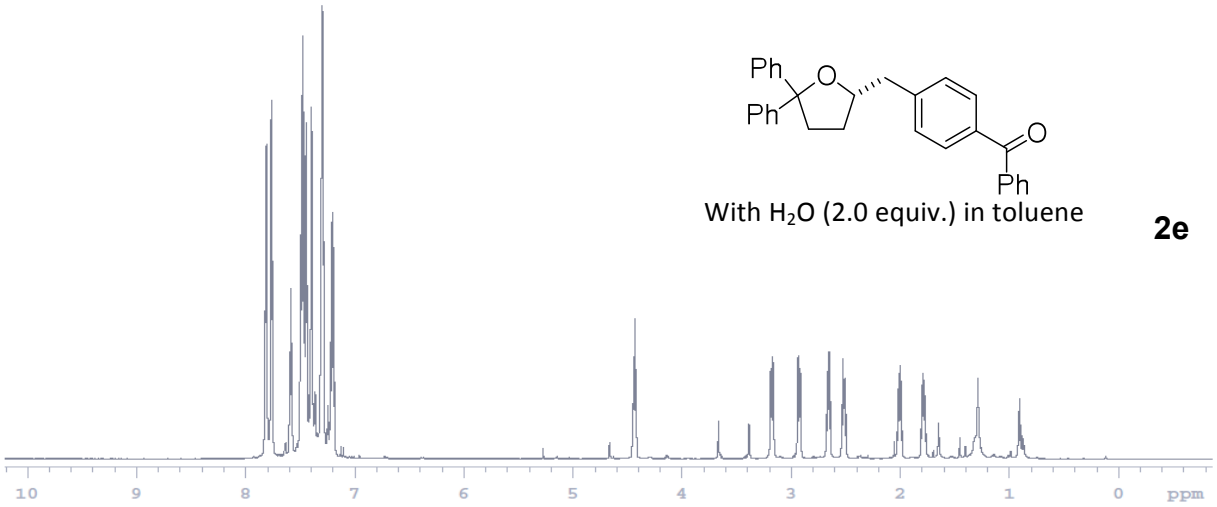
Agilent Technologies

Sample Name	Pulse sequence CARBON	Temperature 25	Study owner bahopki
Date collected 2015-05-08	Solvent cdcl3	Operator bahopki	Printed from te.chem.lsa.umich.edu-vnmrs500



Data file /home/bahopki/vnmrsys/data/BAH-9-178-2carbon.fid

Plot date 2015-05-08



With H_2O (2.0 equiv.) in toluene

2e

Data file /misc/Ytterbium/bahopki/vnmrsys/data/BAH-9-174-3proton.fid

Plot date 2015-05-08

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\RAC-BAH-9-163(2)-5.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-9-163(2)-5.00%IPA-1.00mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-9-163(2)-5.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
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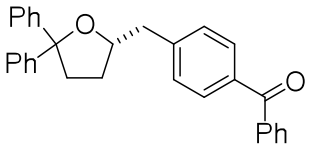
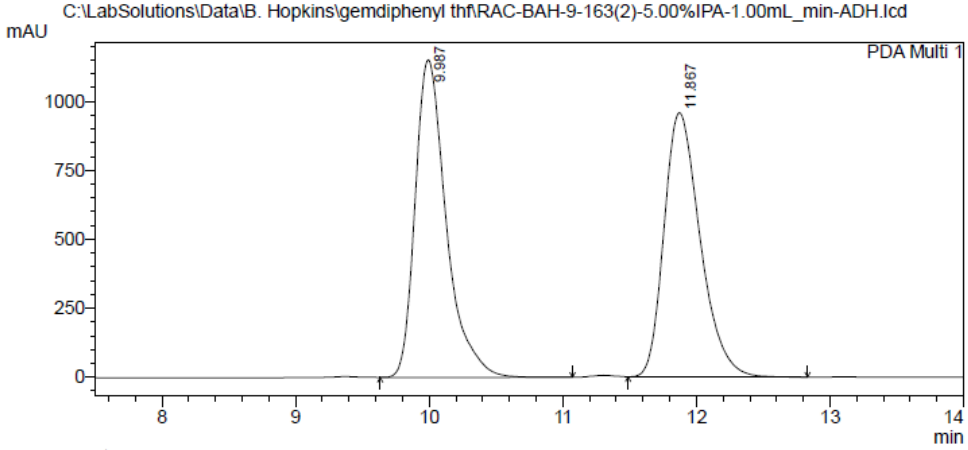
Data Processed	: 4/23/2015 12:14:45 AM



2e

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\RAC-BAH-9-163(2)-5.00%IPA-1.00mL_min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.987	18652646	1152095	50.859	54.585
2	11.867	18022436	958560	49.141	45.415
Total		36675082	2110655	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\CHIRAL-BAH-9-171(1)-5.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-9-171(1)-5.00%IPA-1.00mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-9-171(1)-5.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/2/2015 12:02:09 PM
Data Processed	: 5/2/2015 12:44:56 PM

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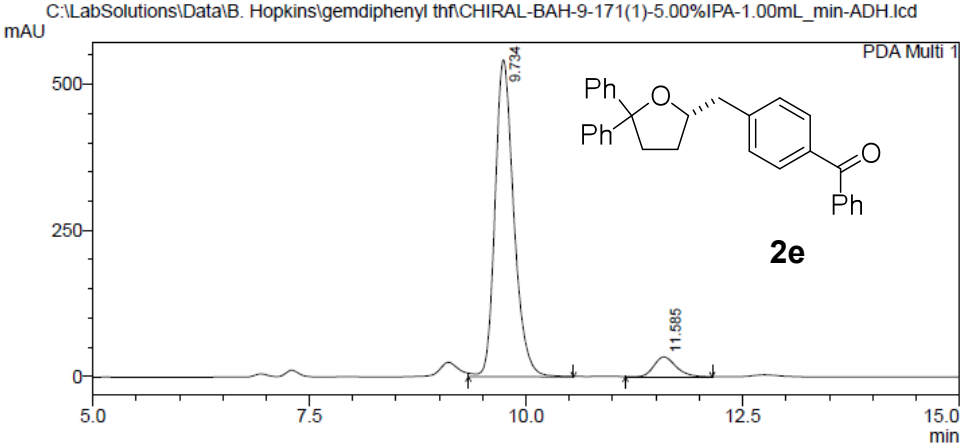

1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.734	8178331	542062	93.068	94.082
2	11.585	609165	34098	6.932	5.918
Total		8787496	576160	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\CHIRAL-BAH-9-165(4)-5.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-9-165(4)-5.00%IPA-1.00mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-9-165(4)-5.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/24/2015 7:47:58 AM
Data Processed	: 4/24/2015 8:04:49 AM

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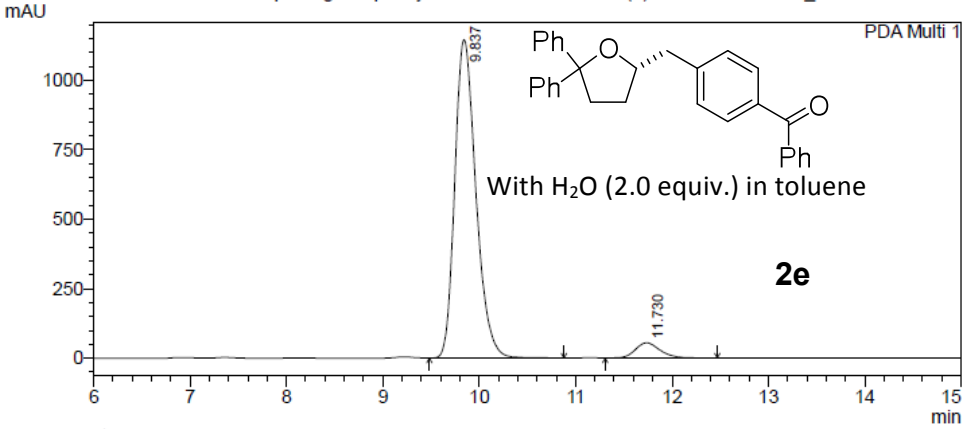


1 PDA Multi 1/254nm 4nm

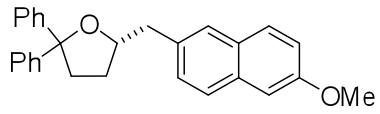
PeakTable

PDA Ch1 254nm 4nm

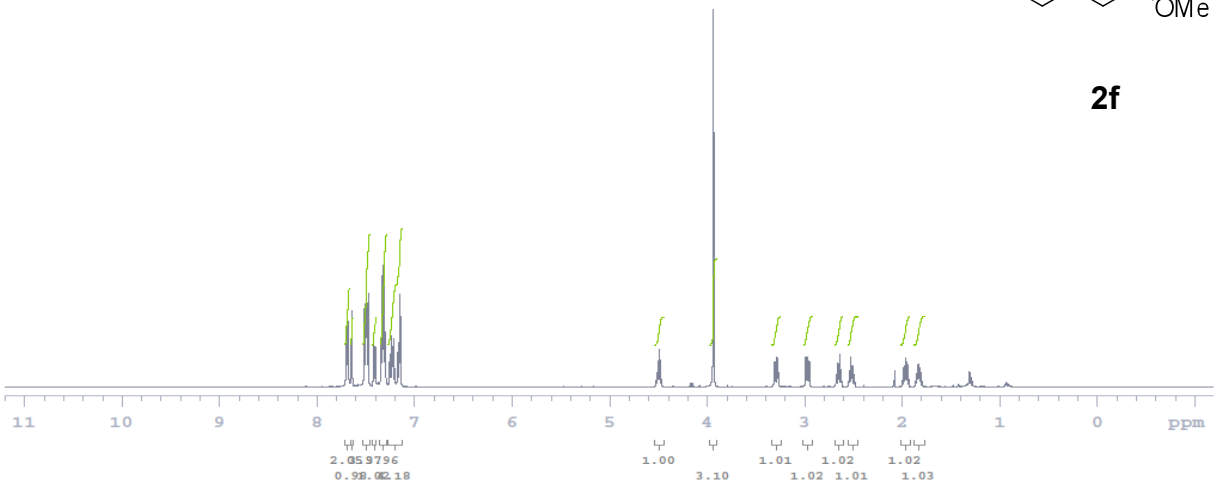
Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.837	17433060	1144879	94.713	95.438
2	11.730	973158	54726	5.287	4.562
Total		18406218	1199605	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2015-04-11
Pulse sequence PROTON
Solvent cdcl3
Temperature 25
Operator bahopki
Study owner bahopki
Printed from kr.chem.lsa.umich.edu-vnmrs500



2f

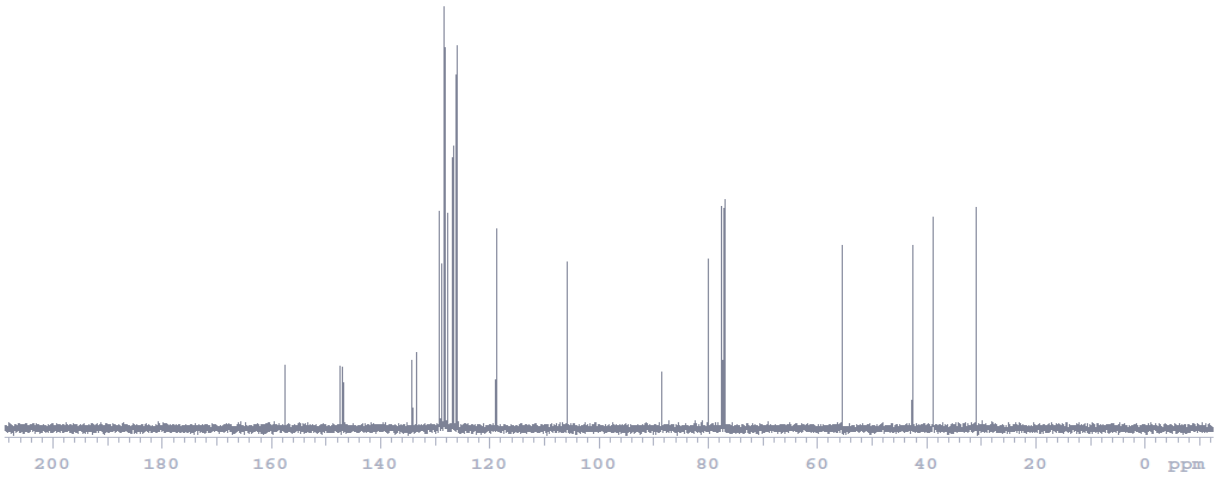


Data file /misc/Tellurium/bahopki/vnmrsys/data/BAH-9-144-2proton.fid

Plot date 2015-05-04

Agilent Technologies

Sample Name
Date collected 2015-04-11
Pulse sequence CARBON
Solvent cdcl3
Temperature 25
Operator bahopki
Study owner bahopki
Printed from kr.chem.lsa.umich.edu-vnmrs500



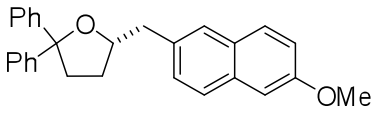
Data file /misc/Tellurium/bahopki/vnmrsys/data/BAH-9-144-2carbon.fid

Plot date 2015-05-04

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\RAC-BAH-9-142(1)-2.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-9-142(1)-2.00%IPA-1.00mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-9-142(1)-2.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/6/2015 1:02:20 PM
Data Processed	: 4/6/2015 1:28:36 PM



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<Chromatogram>

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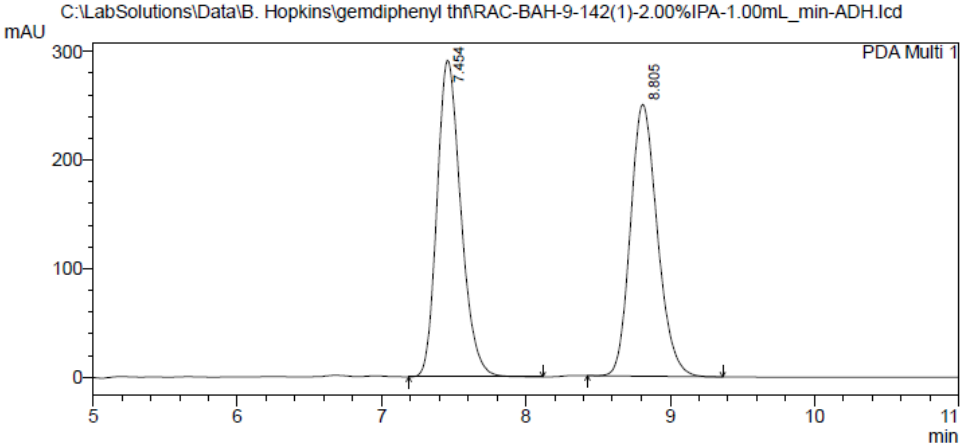

1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.454	3362119	291487	50.829	53.807
2	8.805	3252515	250237	49.171	46.193
Total		6614634	541724	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\CHIRAL-BAH-9-144(2)-2.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-9-144(2)-2.00%IPA-1.00mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-9-144(2)-2.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/7/2015 3:49:10 PM
Data Processed	: 4/7/2015 4:59:12 PM

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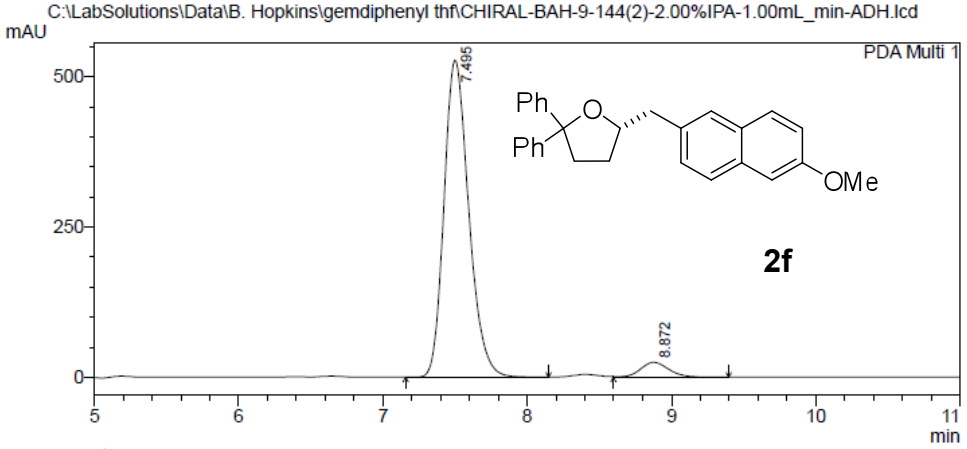


1 PDA Multi 1/254nm 4nm

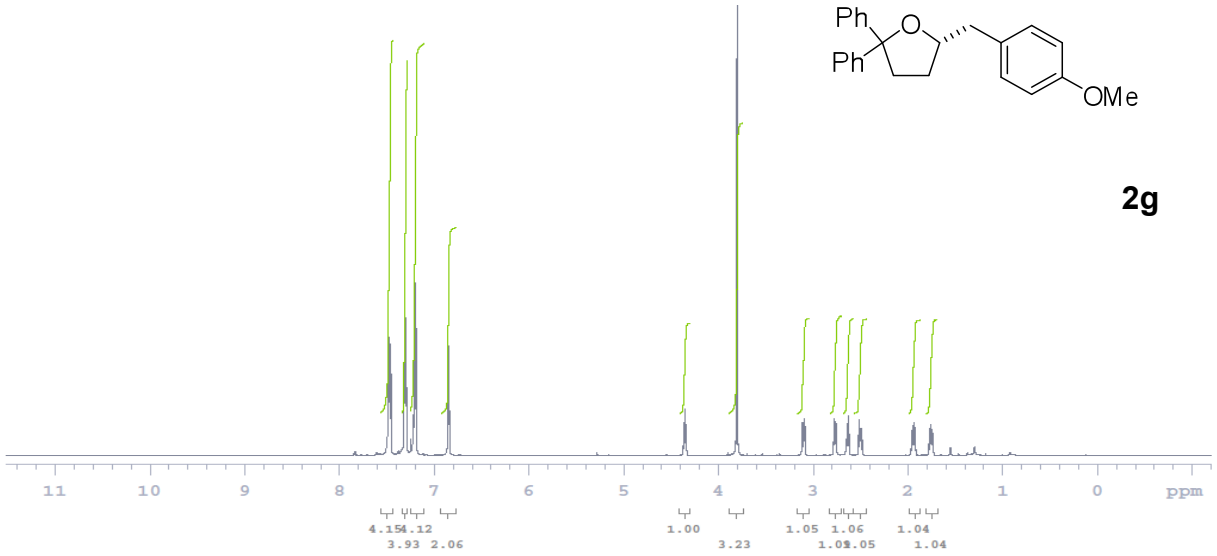
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.495	6299925	527840	94.803	95.510
2	8.872	345355	24817	5.197	4.490
Total		6645280	552657	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2015-05-08
Pulse sequence PROTON
Solvent cdcl3
Temperature 25
Operator bahopki
Study owner bahopki
Printed from yb.chem.lsa.umich.edu-vnmrs700



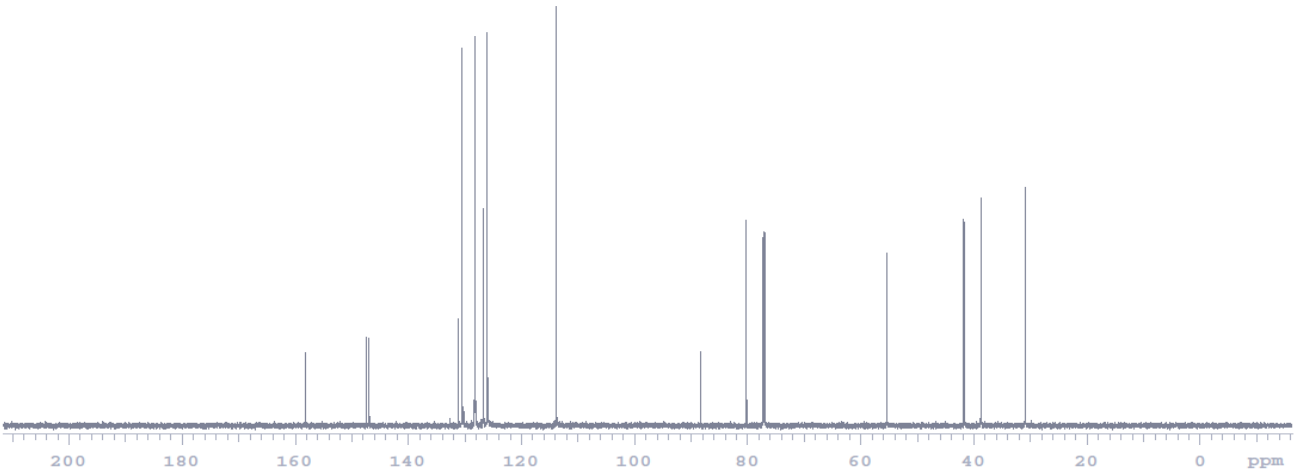
2g

Data file /home/bahopki/vnmrsys/data/BAH-9-178-4proton.fid

Plot date 2015-05-08

Agilent Technologies

Sample Name
Date collected 2015-05-08
Pulse sequence CARBON
Solvent cdcl3
Temperature 25
Operator bahopki
Study owner bahopki
Printed from yb.chem.lsa.umich.edu-vnmrs700



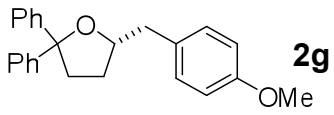
Data file /home/bahopki/vnmrsys/data/BAH-9-178-4carbon.fid

Plot date 2015-05-08

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\RAC-BAH-9-158(1)-1.00%IPA-1.00mL_min-ADH.lcd

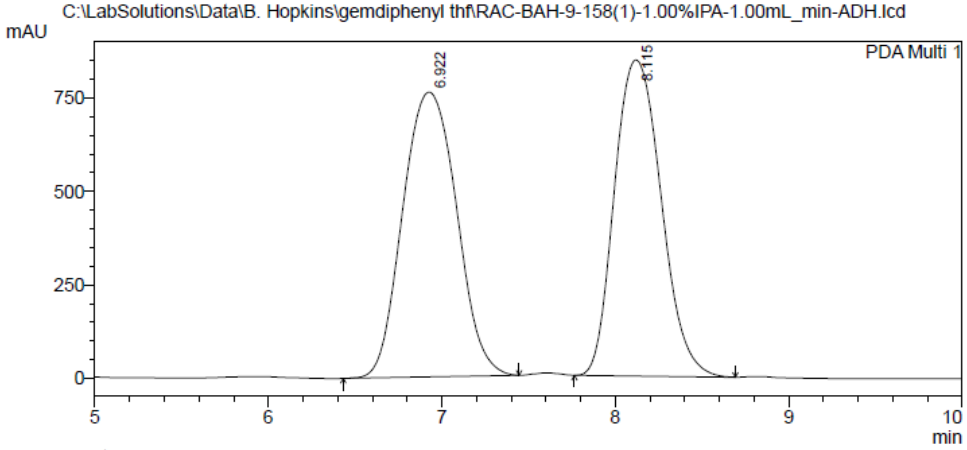
Acquired by	: Admin
Sample Name	: RAC-BAH-9-158(1)-1.00%IPA-1.00mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-9-158(1)-1.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/20/2015 11:12:08 PM
Data Processed	: 4/20/2015 11:25:00 PM



2g

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\RAC-BAH-9-158(1)-1.00%IPA-1.00mL_min-ADH.lcd



1 PDA Multi 1/230nm 4nm

PeakTable

PDA Ch1 230nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.922	16482730	762059	50.931	47.384
2	8.115	15880069	846210	49.069	52.616
Total		32362798	1608269	100.000	100.000

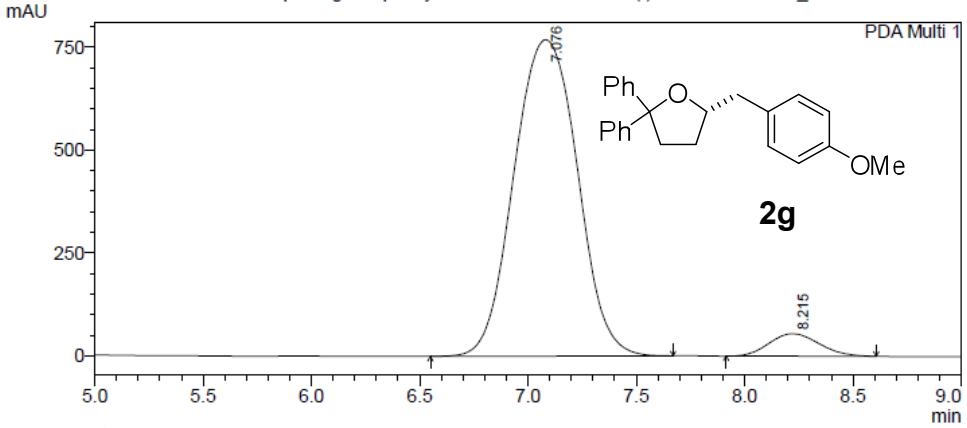
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\CHIRAL-BAH-9-170(I)-1.00%IPA-1.0mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-9-170(I)-1.00%IPA-1.0mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-9-170(I)-1.00%IPA-1.0mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/1/2015 6:05:32 PM
Data Processed	: 5/1/2015 6:46:19 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\CHIRAL-BAH-9-170(I)-1.00%IPA-1.0mL_min-ADH.lcd



1 PDA Multi 1/230nm 4nm

PeakTable

PDA Ch1 230nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.076	15706142	769699	94.696	93.385
2	8.215	879721	54526	5.304	6.615
Total		16585863	824224	100.000	100.000

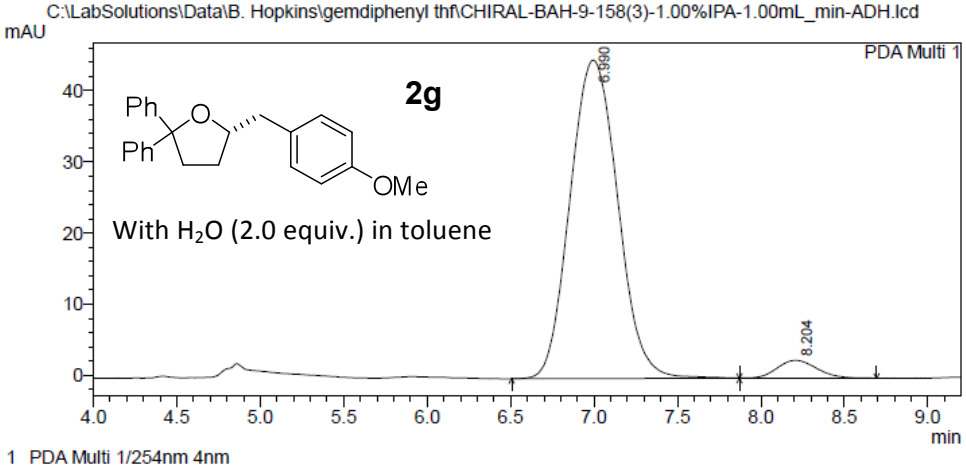
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\CHIRAL-BAH-9-158(3)-1.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-9-158(3)-1.00%IPA-1.00mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-9-158(3)-1.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/21/2015 12:21:25 AM
Data Processed	: 4/21/2015 12:31:07 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\CHIRAL-BAH-9-158(3)-1.00%IPA-1.00mL_min-ADH.lcd



1 PDA Multi 1/254nm 4nm

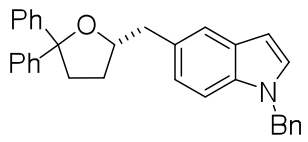
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.990	891515	44749	95.481	94.667
2	8.204	42192	2521	4.519	5.333
Total		933707	47270	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2015-05-06
Pulse sequence PROTON
Solvent cdcl3
Temperature 25
Operator bahopki
Study owner bahopki
Printed from dy.chem.lsa.umich.edu-vnmrs500



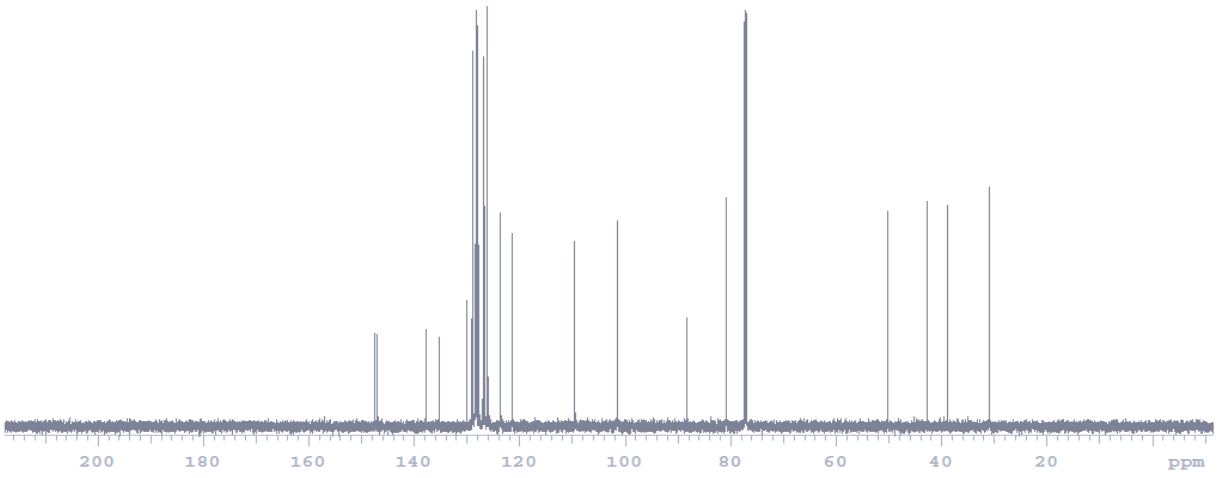
2h

Data file /misc/Ytterbium/bahopki/vnmrsys/data/BAH-9-175-2proton.fid

Plot date 2015-05-08

Agilent Technologies

Sample Name
Date collected 2015-05-06
Pulse sequence CARBON
Solvent cdcl3
Temperature 25
Operator bahopki
Study owner bahopki
Printed from dy.chem.lsa.umich.edu-vnmrs500



Data file /misc/Ytterbium/bahopki/vnmrsys/data/BAH-9-175-2carbon.fid

Plot date 2015-05-08

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\RAC-BAH-9-167(1)-2.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-9-167(1)-2.00%IPA-1.00mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-9-167(1)-2.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
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Data Processed	: 5/3/2015 10:28:12 PM

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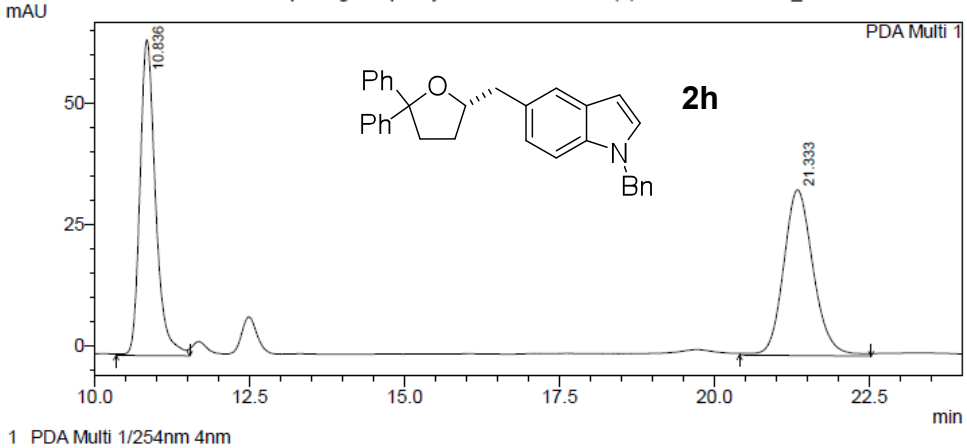
C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\RAC-BAH-9-167(1)-2.00%IPA-1.00mL_min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.836	1143534	65081	49.978	65.593
2	21.333	1144518	34138	50.022	34.407
Total		2288053	99219	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\CHIRAL-BAH-9-175(2)-2.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-9-175(2)-2.00%IPA-1.00mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-9-175(2)-2.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/6/2015 3:20:11 AM
Data Processed	: 5/6/2015 3:43:56 AM

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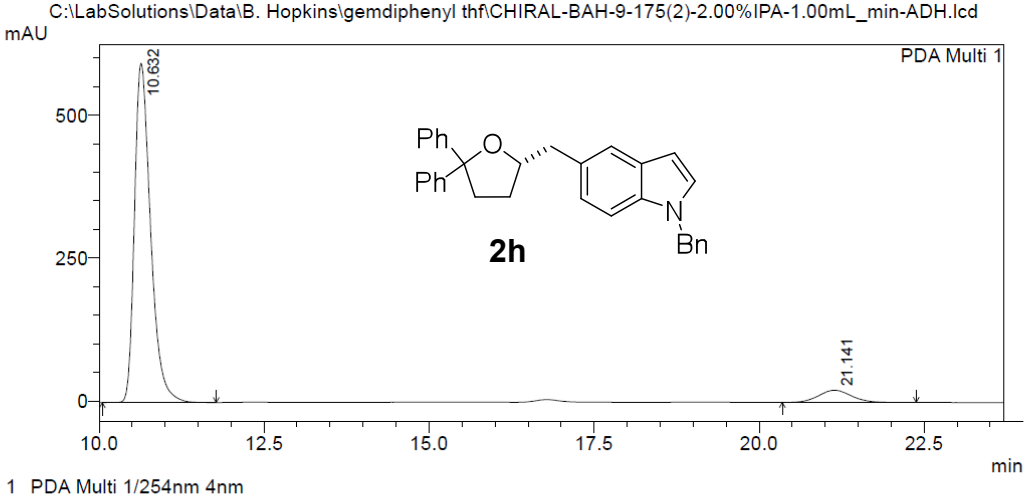
C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\CHIRAL-BAH-9-175(2)-2.00%IPA-1.00mL_min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.632	10096553	593722	93.160	96.529
2	21.141	741340	21347	6.840	3.471
Total		10837893	615069	100.000	100.000

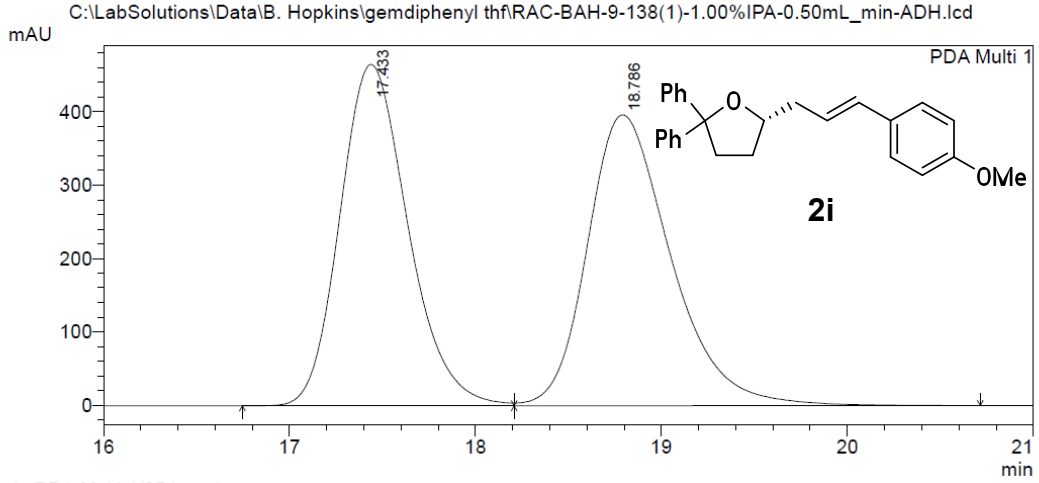
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\RAC-BAH-9-138(1)-1.00%IPA-0.50mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-9-138(1)-1.00%IPA-0.50mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-9-138(1)-1.00%IPA-0.50mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/3/2015 2:58:02 PM
Data Processed	: 4/3/2015 3:22:05 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\RAC-BAH-9-138(1)-1.00%IPA-0.50mL_min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.433	11809441	465170	48.852	54.004
2	18.786	12364382	396186	51.148	45.996
Total		24173823	861356	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\gemdiphenyl thf\CHIRAL-BAH-9-138(2)-1.00%IPA-0.50mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-9-138(2)-1.00%IPA-0.50mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-9-138(2)-1.00%IPA-0.50mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/3/2015 3:26:59 PM
Data Processed	: 4/3/2015 4:37:01 PM

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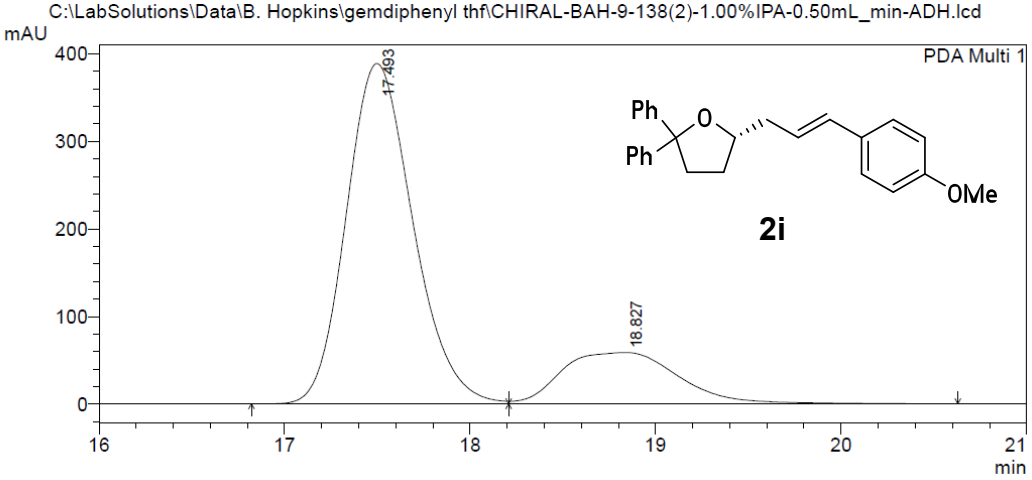


1 PDA Multi 1/254nm 4nm

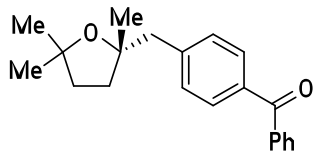
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.493	9690833	388542	79.180	86.888
2	18.827	2548227	58633	20.820	13.112
Total		12239059	447175	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2016-06-07
Pulse sequence PROTON
Solvent odcl3
Temperature 24
Operator garletsz
Study owner garletsz
Printed from zn.ohem.lsa.umich.edu-vnmrs400



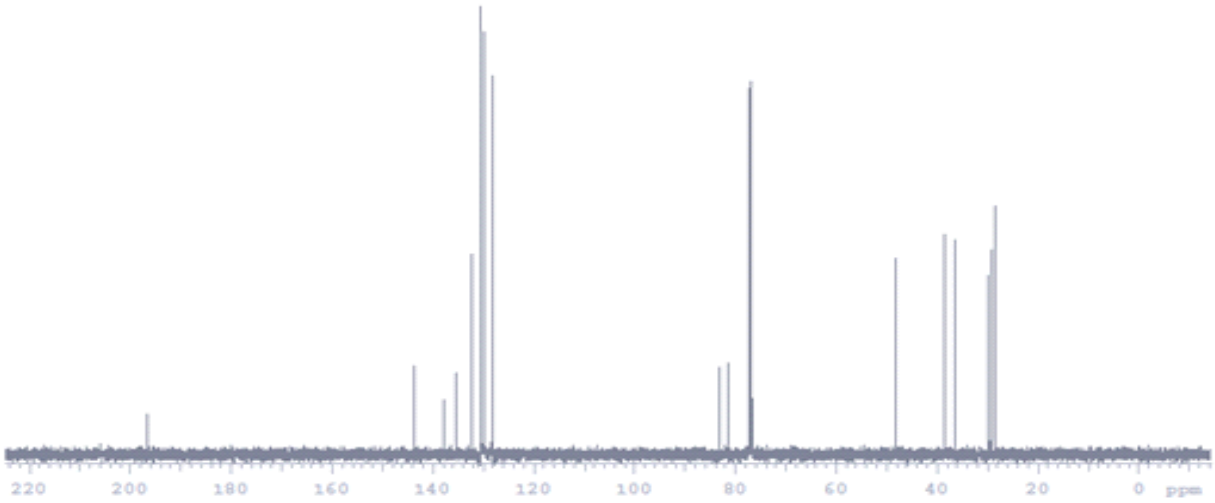
2j

Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.2.197.3.H1.fid

Plot date 2015-05-14

Agilent Technologies

Sample Name
Date collected 2016-06-07
Pulse sequence CARBON
Solvent odcl3
Temperature 24
Operator garletsz
Study owner garletsz
Printed from zn.ohem.lsa.umich.edu-vnmrs400



Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.2.197.4.C13.fid

Plot date 2015-05-14

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Zac G\RAC-ZJG-2-189C-1-DPEPhos-4bromobenzophenone-2.00%IPA-01.0mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-2-189C-1-DPEPhos-4bromobenzophenone-2.00%IPA-01.0mL_min
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-2-189C-1-DPEPhos-4bromobenzophenone-2.00%IPA-01.0mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/29/2015 2:38:55 PM
Data Processed	: 4/29/2015 2:57:12 PM

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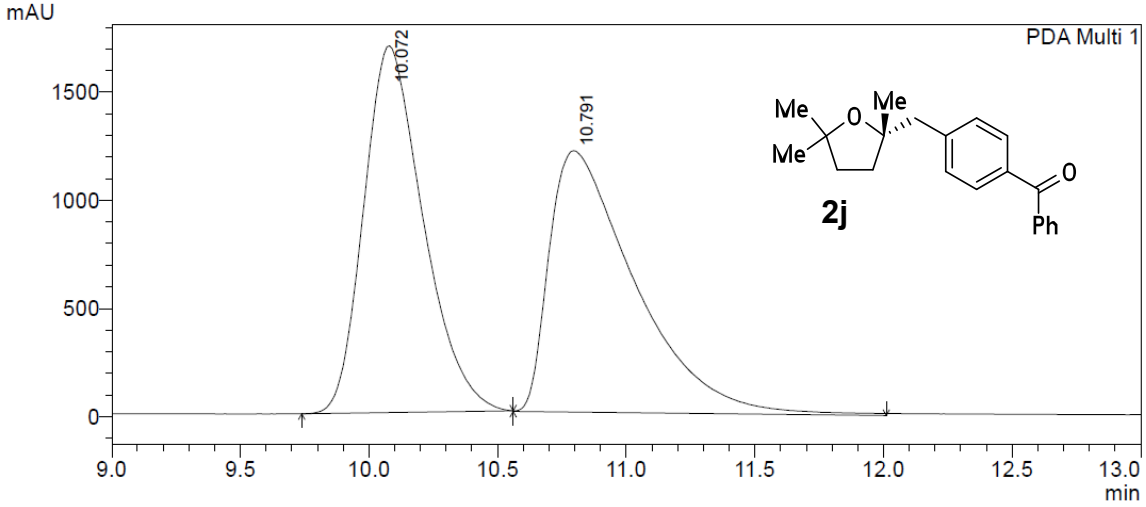

1 PDA Multi 1/195nm 4nm

PeakTable

PDA Ch1 195nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.072	27237637	1695761	49.332	58.394
2	10.791	27974754	1208227	50.668	41.606
Total		55212392	2903988	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Zac G\CHIRAL-ZJG-2-197-4bromobenzophenon-2.00%IPA-01.0mL_minADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-2-197-4bromobenzophenon-2.00%IPA-01.0mL_minADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-2-197-4bromobenzophenon-2.00%IPA-01.0mL_minADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/2/2015 11:08:59 AM
Data Processed	: 5/2/2015 11:24:17 AM

<Chromatogram>

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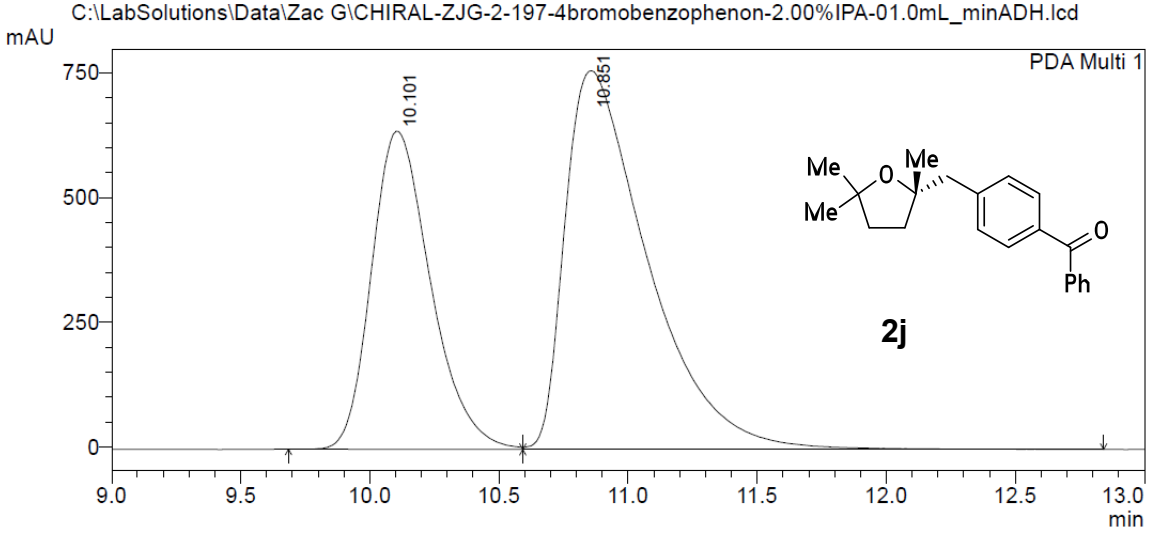


1 PDA Multi 1/195nm 4nm

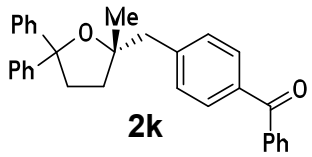
PeakTable

PDA Ch1 195nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.101	10171837	637898	37.522	45.663
2	10.851	16936870	759064	62.478	54.337
Total		27108707	1396962	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2016-05-16
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from oc.ohem.lca.umloh.edu-vnmrs400



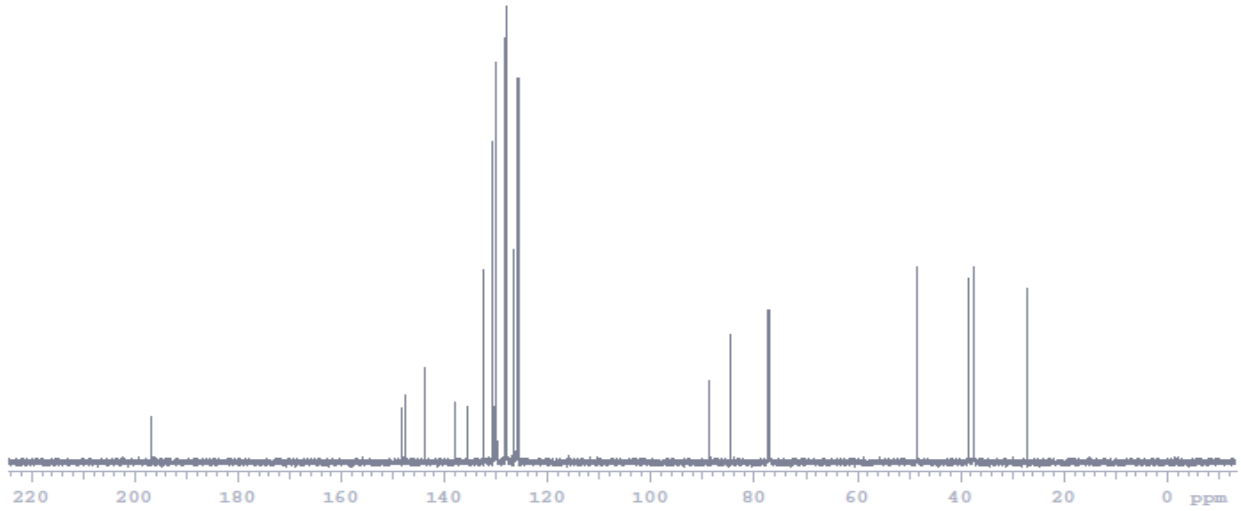
2k

Data file /home/garletsz/vnmrsys/data/zjg.jpw.3.6.5.H1.fid

Plot date 2015-05-16

Agilent Technologies

Sample Name
Date collected 2016-05-14
Pulse sequence CARBON
Solvent odcl3
Temperature 24
Operator garletsz
Study owner garletsz
Printed from dy.ohem.lca.umloh.edu-vnmrs600



Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.2.208.B.4.C13.fid

Plot date 2015-05-14

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Zac G\RAC-ZJG-2-201A-1-DPEPhos-4bromobenzophenone-2.00%IPA-01.0mL_min1.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-2-201-1-DPEPhos-4bromobenzophenone-2.00%IPA-01.0mL_min1
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-2-201A-1-DPEPhos-4bromobenzophenone-2.00%IPA-01.0mL_min1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/14/2015 5:38:49 PM
Data Processed	: 5/14/2015 6:13:17 PM

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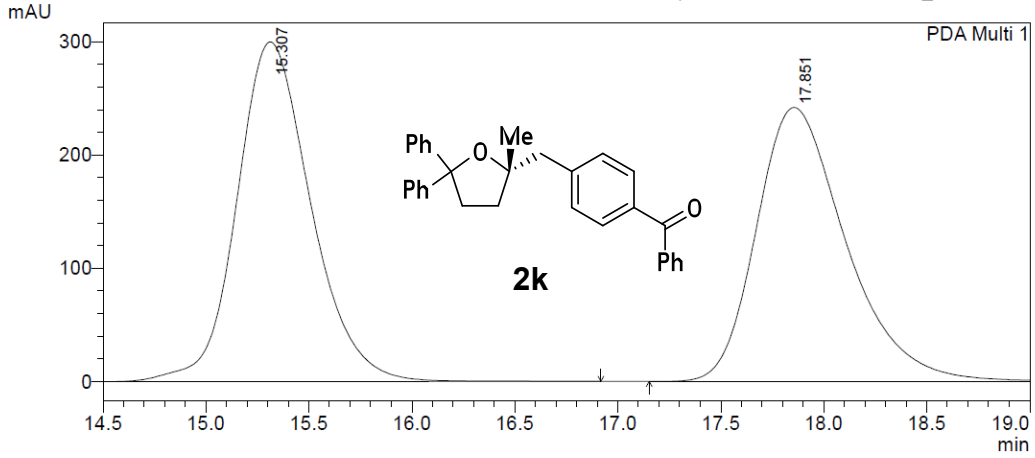
C:\LabSolutions\Data\Zac G\RAC-ZJG-2-201A-1-DPEPhos-4bromobenzophenone-2.00%IPA-01.0mL_min1.lcd



1 PDA Multi 1/275nm 4nm

PeakTable

PDA Ch1 275nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.307	7497473	299832	50.619	55.349
2	17.851	7314070	241882	49.381	44.651
Total		14811543	541715	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Zac G\CHIRAL-ZJG-2-208B-Bligand-4bromobenzophenone-2.00%IPA-01.0mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-2-208B-Bligand-4bromobenzophenone-2.00%IPA-01.0mL_mi
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-2-208B-Bligand-4bromobenzophenone-2.00%IPA-01.0mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/14/2015 4:44:49 PM
Data Processed	: 5/14/2015 5:15:41 PM

<Chromatogram>

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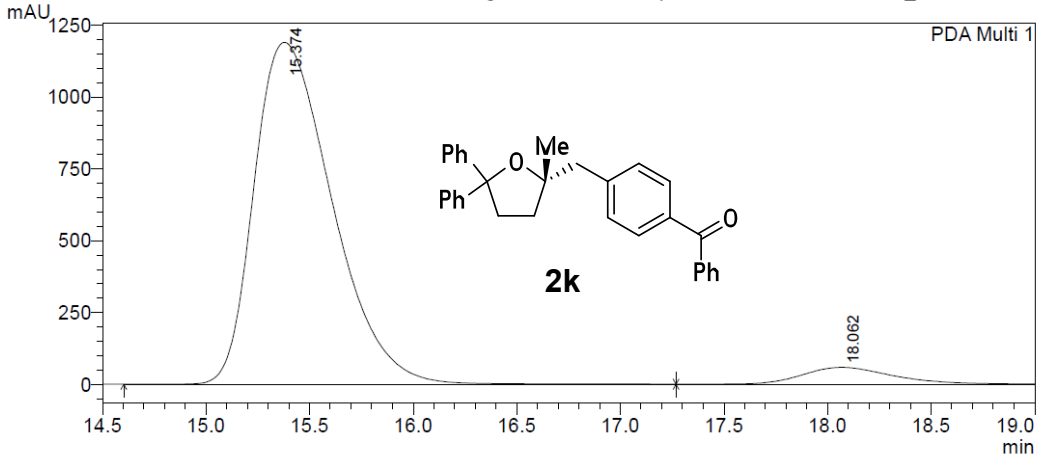
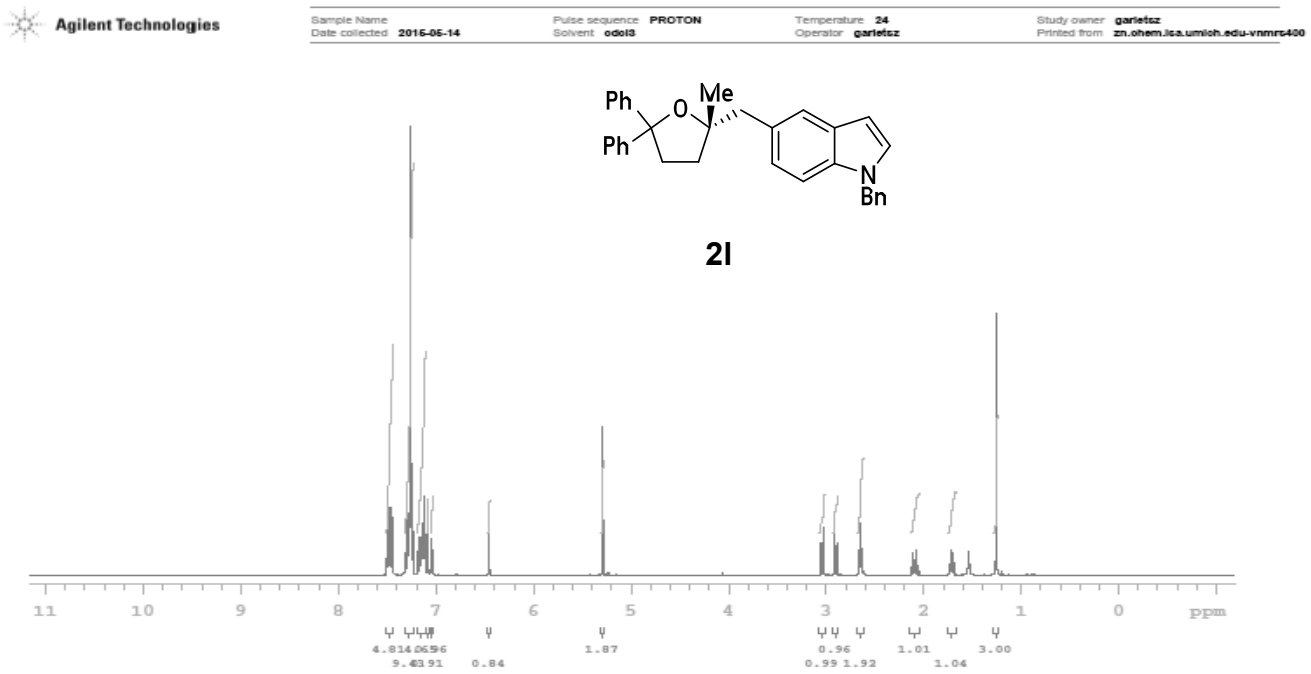


1 PDA Multi 1/275nm 4nm

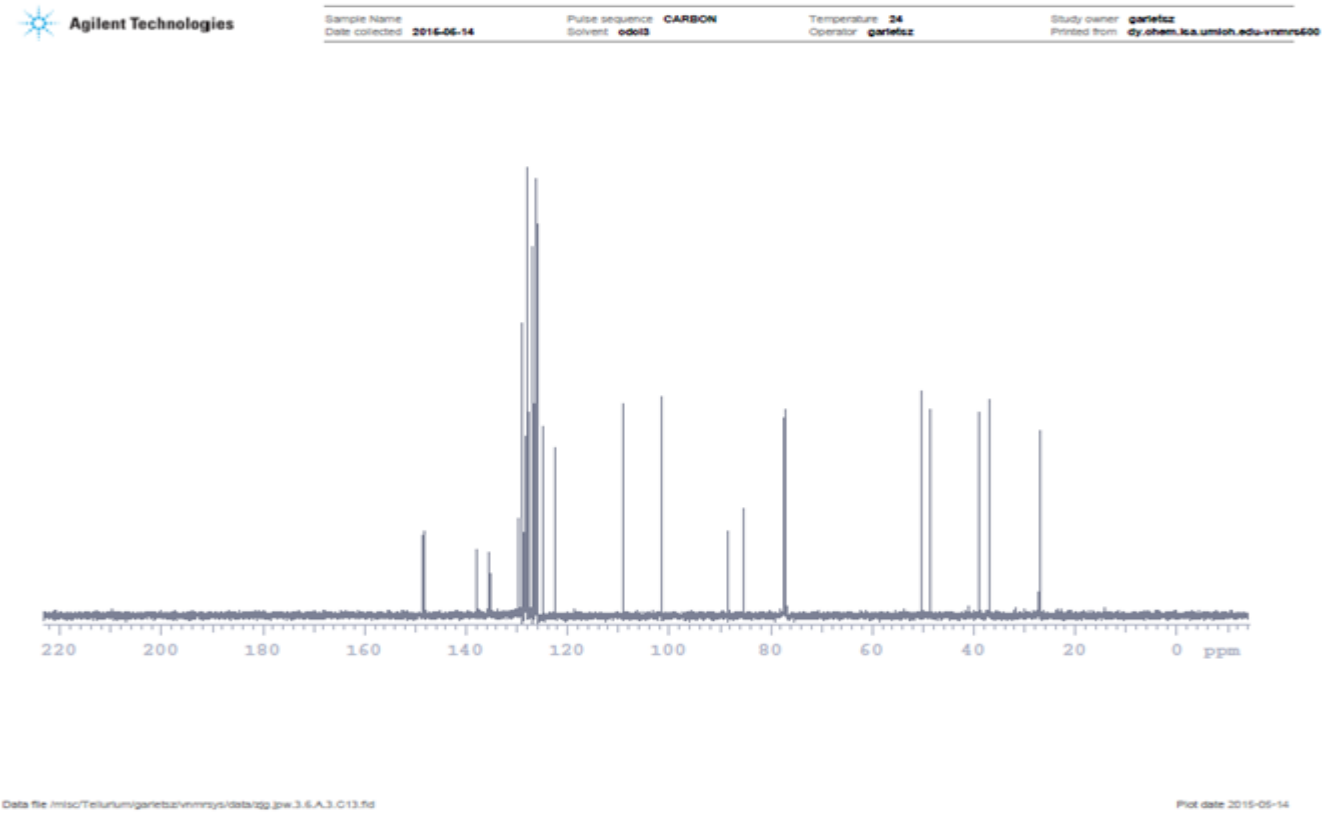
PeakTable

PDA Ch1 275nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.374	31151956	1189142	94.565	95.305
2	18.062	1790297	58578	5.435	4.695
Total		32942253	1247721	100.000	100.000


Agilent Technologies
Sample Name
Date collected 2015-05-14
Pulse sequence PROTON
Solvent odcl3
Temperature 24
Operator garletsz
Study owner garletsz
Printed from zn.chem.lsa.umich.edu-vnmrs400
Ph
Ph
O
Me
N
Bn
11 10 9 8 7 6 5 4 3 2 1 0 ppm
4.81 4.06 9.46 3.91
0.84
1.87
0.96 0.99 1.92
1.01 1.04
3.00
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.2.208.C.4.H1.fid
Plot date 2015-05-14

2l


Agilent Technologies
Sample Name
Date collected 2015-05-14
Pulse sequence CARBON
Solvent odcl3
Temperature 24
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs600
220 200 180 160 140 120 100 80 60 40 20 0 ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.6.A.3.C13.fid
Plot date 2015-05-14

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Zac G\RAC-ZJG-2-206-1-DPEPhos-bromobnindole-1.00%IPA-01.0mL_min1.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-2-206-1-DPEPhos-bromobnindole-1.00%IPA-01.0mL_min1
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-2-206-1-DPEPhos-bromobnindole-1.00%IPA-01.0mL_min1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/16/2015 11:18:00 AM
Data Processed	: 5/16/2015 12:05:19 PM

<Chromatogram>

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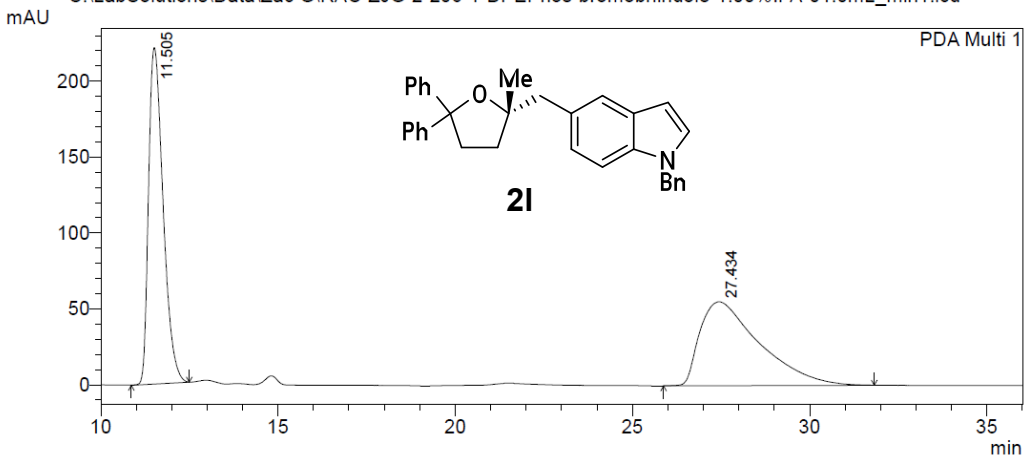

1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.505	6336323	221869	49.463	80.036
2	27.434	6473788	55341	50.537	19.964
Total		12810111	277211	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Zac G\CHIRAL-ZJG-3-6-A-Bligand-bromobnindole-1.0%IPA-01.0mL_minAD1.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-6-A-Bligand-bromobnindole-1.0%IPA-01.0mL_minAD1
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-6-A-Bligand-bromobnindole-1.0%IPA-01.0mL_minAD1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/16/2015 12:06:37 PM
Data Processed	: 5/16/2015 1:06:37 PM

<Chromatogram>

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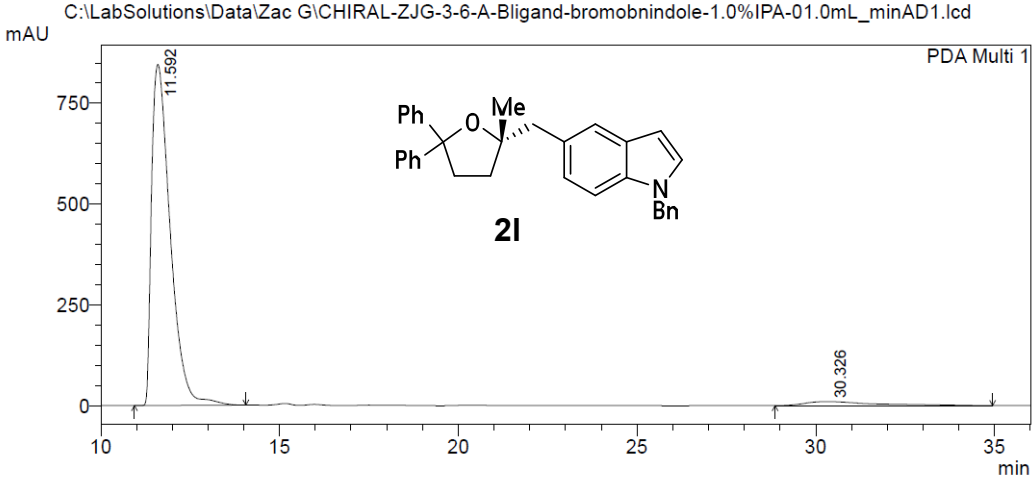


1 PDA Multi 1/254nm 4nm

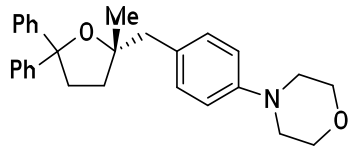
PeakTable

PDA Ch1 254nm 4nm

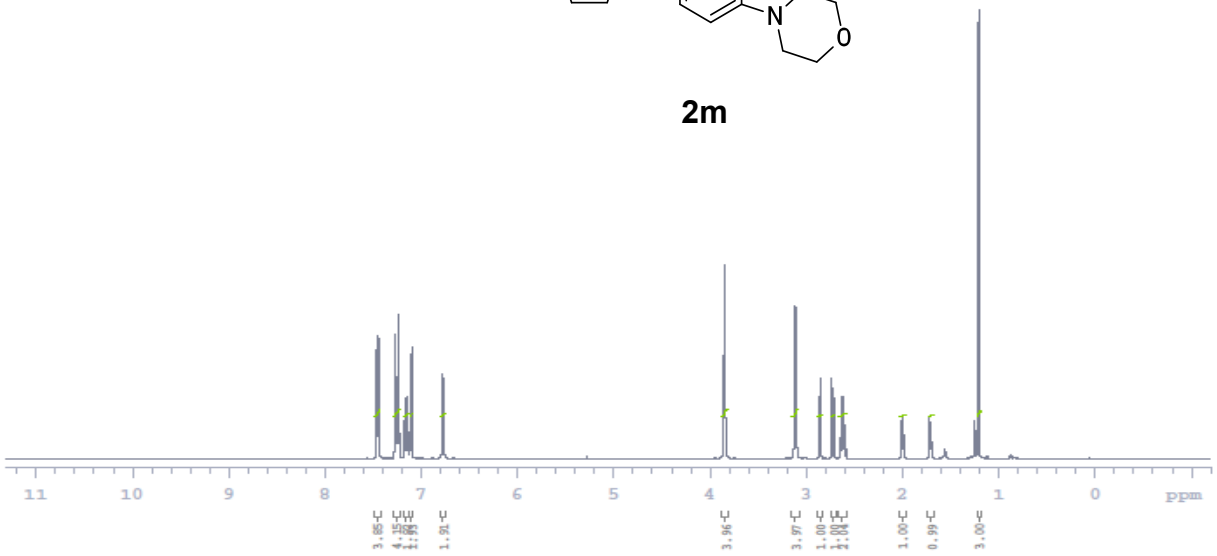
Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.592	30684412	846134	95.635	98.815
2	30.326	1400511	10144	4.365	1.185
Total		32084923	856277	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2016-05-11
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.chem.isa.umich.edu-vnmrs600



2m

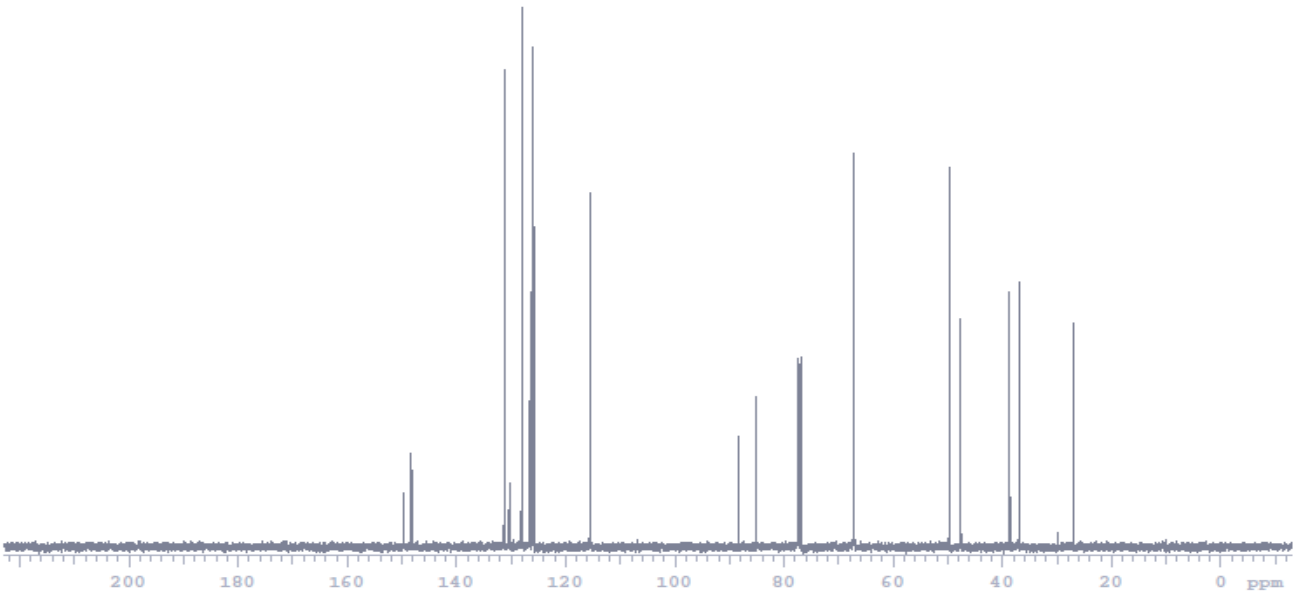


Data file /misc/Ytterbium/garletsz/vnmrsys/data/zjg.jpw.2.205.3.H1.fid

Plot date 2015-05-14

Agilent Technologies

Sample Name
Date collected 2016-05-14
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from te.chem.isa.umich.edu-vnmrs600



Data file /home/garletsz/vnmrsys/data/zjg.jpw.2.205.6.C13.fid

Plot date 2015-05-14

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Zac G\RAC-ZJG-2-201B-1-DPEPhos-4bromobenzomorpoli-2.00%IPA-01.0mL_min2.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-2-201B-1-DPEPhos-4bromobenzomorpoli-2.00%IPA-01.0mL_min
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-2-201B-1-DPEPhos-4bromobenzomorpoli-2.00%IPA-01.0mL_min2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/14/2015 5:17:10 PM
Data Processed	: 5/14/2015 5:34:30 PM

<Chromatogram>

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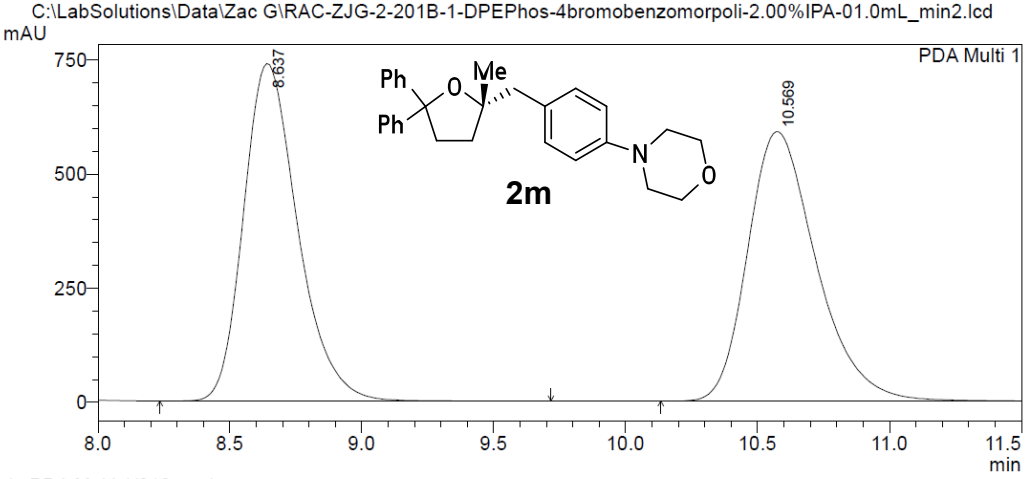

1 PDA Multi 1/210nm 4nm

PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.637	10540544	739139	49.688	55.597
2	10.569	10672771	590323	50.312	44.403
Total		21213314	1329462	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Zac G\CHIRAL-ZJG-2-205-1-4bromobenzomorpoli-2.00%IPA-01.0mL_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-2-205-1-4bromobenzomorpoli-2.00%IPA-01.0mL_min
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-2-205-1-4bromobenzomorpoli-2.00%IPA-01.0mL_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/11/2015 12:11:49 PM
Data Processed	: 5/11/2015 12:32:52 PM

<Chromatogram>

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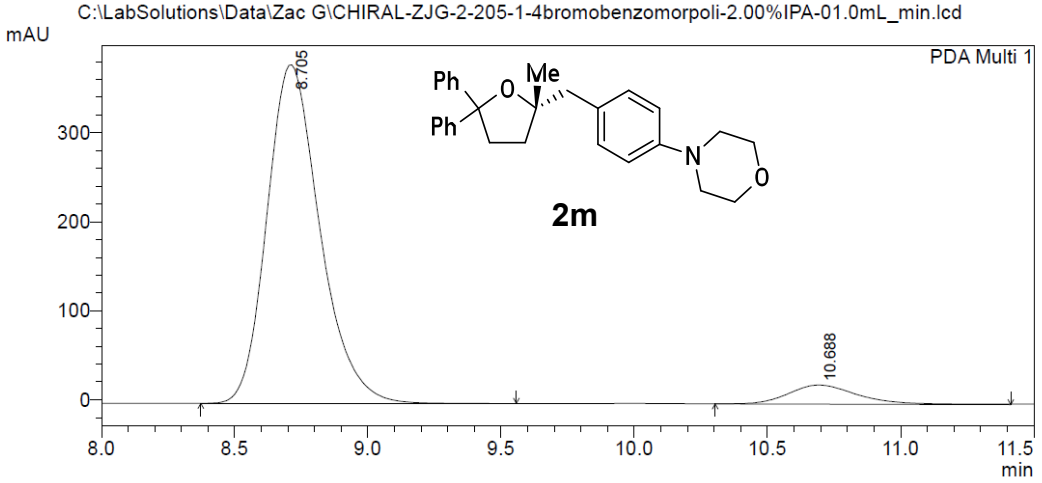


1 PDA Multi 1/210nm 4nm

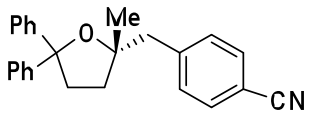
PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.705	5254484	380375	93.034	94.651
2	10.688	393455	21497	6.966	5.349
Total		5647939	401872	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2015-05-16
Pulse sequence PROTON
Solvent odcl3
Temperature 20
Operator garletsz
Study owner garletsz
Printed from dy.chem.lca.umich.edu-vnmrs600



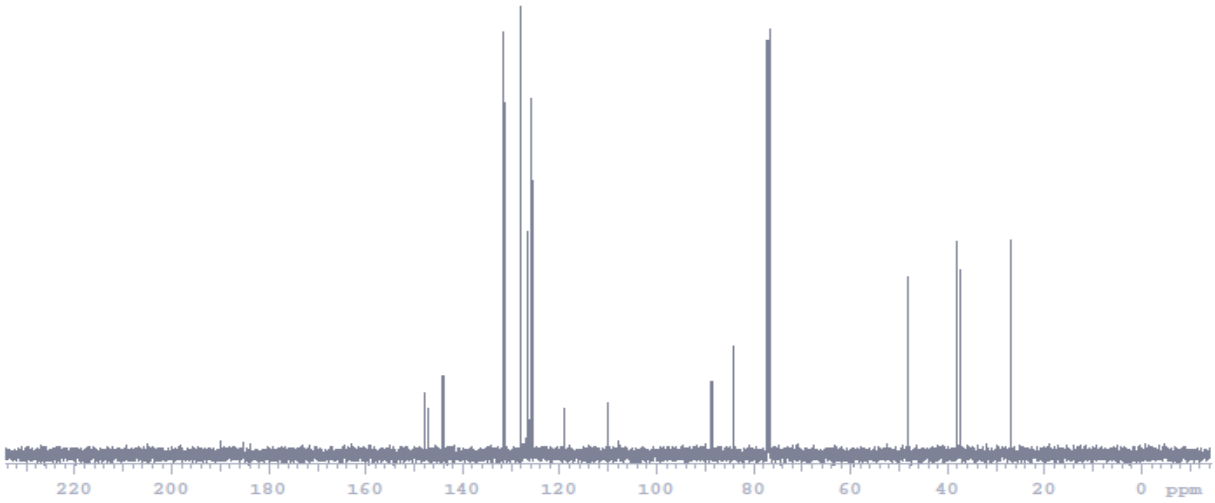
2n

Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.5.6.H1.fid

Plot date 2015-05-16

Agilent Technologies

Sample Name
Date collected 2015-05-16
Pulse sequence CARBON
Solvent odcl3
Temperature 60
Operator garletsz
Study owner garletsz
Printed from dy.chem.lca.umich.edu-vnmrs600



Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.5.4.C13.fid

Plot date 2015-05-16

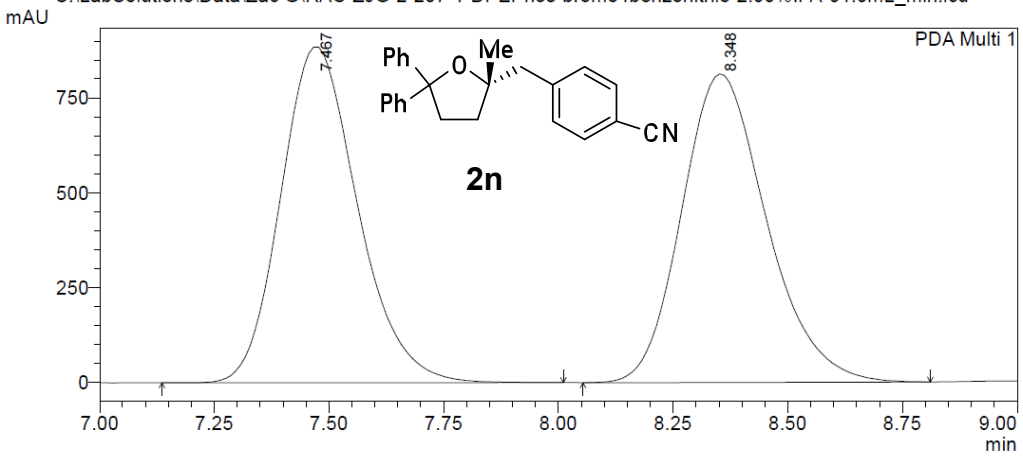
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Zac G\RAC-ZJG-2-207-1-DPEPhos-bromo4benzonitrile-2.00%IPA-01.0mL_min.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-2-207-1-DPEPhos-bromo4benzonitrile-2.00%IPA-01.0mL_min.
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-2-207-1-DPEPhos-bromo4benzonitrile-2.00%IPA-01.0mL_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/12/2015 3:11:17 PM
Data Processed	: 5/12/2015 3:20:54 PM

<Chromatogram>

C:\LabSolutions\Data\Zac G\RAC-ZJG-2-207-1-DPEPhos-bromo4benzonitrile-2.00%IPA-01.0mL_min.lcd



1 PDA Multi 1/195nm 4nm

PeakTable

PDA Ch1 195nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.467	10329364	884476	49.713	52.115
2	8.348	10448562	812688	50.287	47.885
Total		20777926	1697164	100.000	100.000

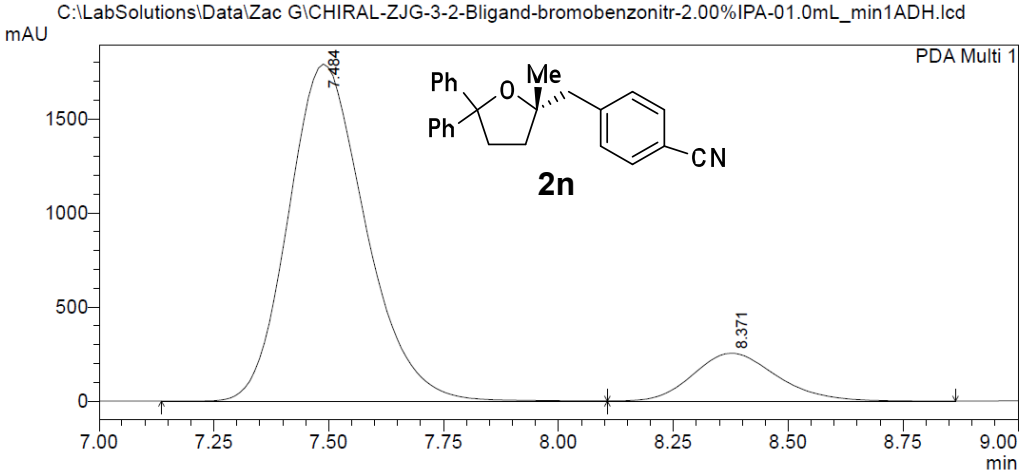
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Zac G\CHIRAL-ZJG-3-2-Bligand-bromobenzonitr-2.00%IPA-01.0mL_min1ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-3-2-DPEPhos-bromobenzonitr-2.00%IPA-01.0mL_min1ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-2-Bligand-bromobenzonitr-2.00%IPA-01.0mL_min1ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/13/2015 4:39:14 PM
Data Processed	: 5/13/2015 4:56:56 PM

<Chromatogram>

C:\LabSolutions\Data\Zac G\CHIRAL-ZJG-3-2-Bligand-bromobenzonitr-2.00%IPA-01.0mL_min1ADH.lcd

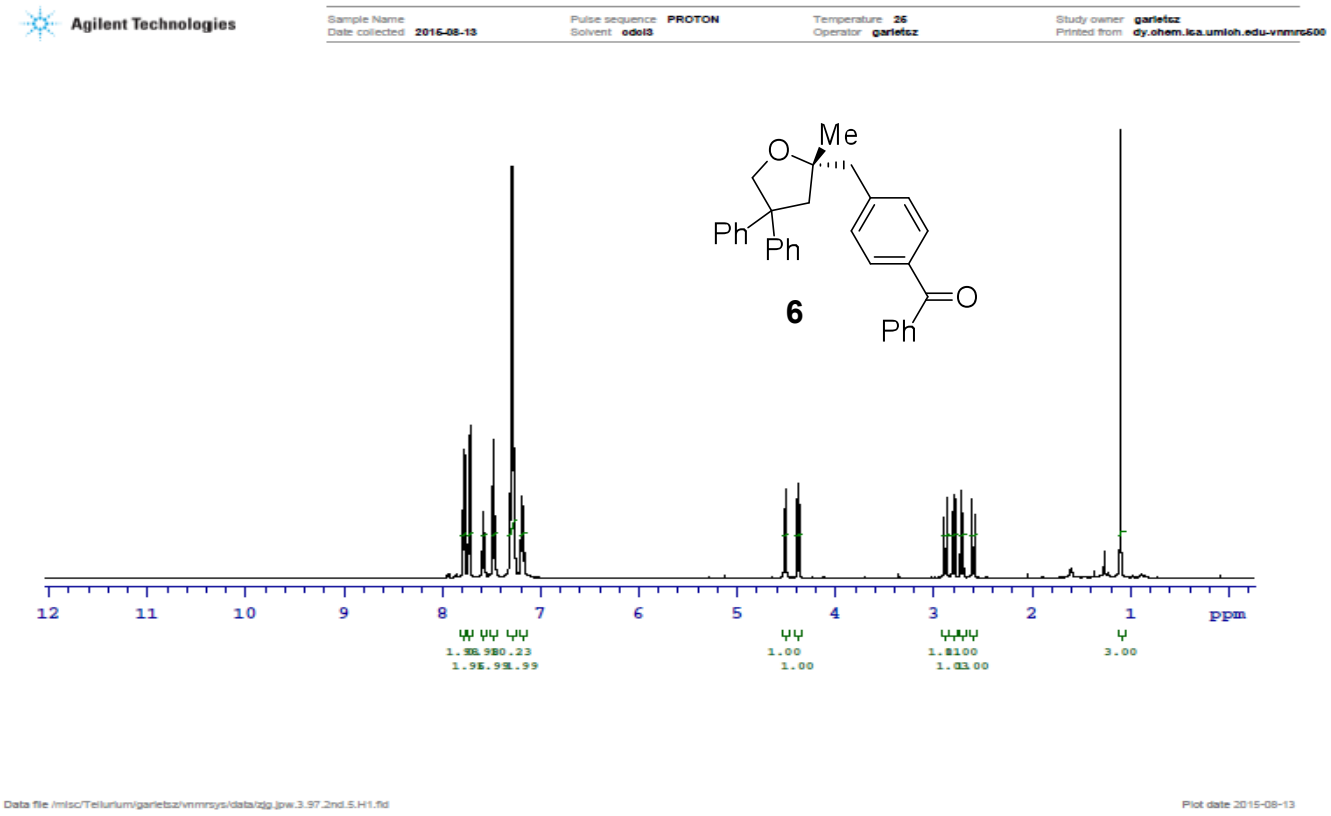


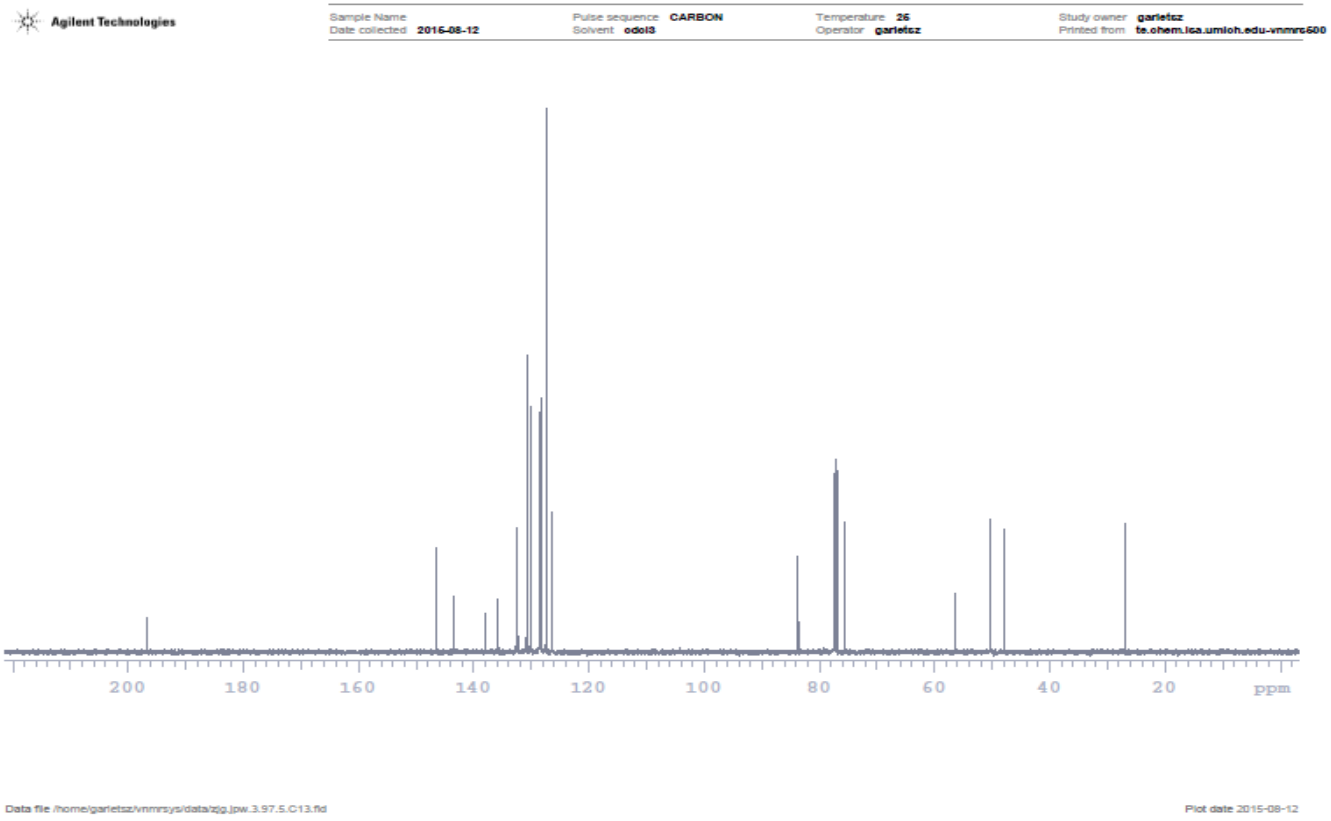
1 PDA Multi 1/195nm 4nm

PeakTable

PDA Ch1 195nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.484	20895001	1791445	86.563	87.549
2	8.371	3243362	254773	13.437	12.451
Total		24138363	2046218	100.000	100.000


Agilent Technologies
Sample Name
Date collected 2016-08-13
Pulse sequence PROTON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs600
Me
O
Ph
Ph
O
Ph
6
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.00
3.00
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.97.2nd.S.H1.fid
Plot date 2016-08-13


Agilent Technologies
Sample Name
Date collected 2016-08-12
Pulse sequence CARBON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from te.chem.lsa.umich.edu-vnmrs600
200
180
160
140
120
100
80
60
40
20
ppm
Data file /home/garletsz/vnmrsys/data/zjg.jpw.3.97.5.C13.fid
Plot date 2016-08-12

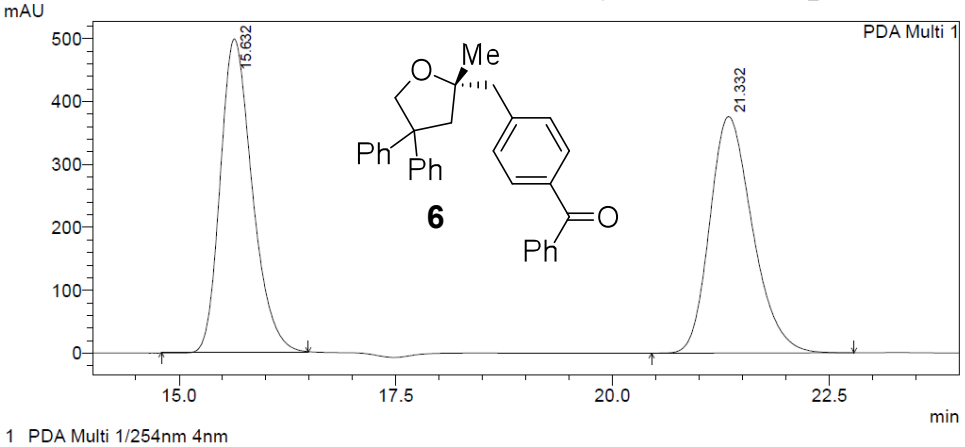
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Zac G\THFs\RAC-ZJG-3-96-DPEPhos-benzophenone-5.0%IPA-1.0mL_minADH.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-3-96-DPEPhos-benzophenone-5.0%IPA-1.0mL_minADH.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-3-96-DPEPhos-benzophenone-5.0%IPA-1.0mL_minADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 8/14/2015 11:04:13 AM
Data Processed	: 8/14/2015 11:29:30 AM

<Chromatogram>

C:\LabSolutions\Data\Zac G\THFs\RAC-ZJG-3-96-DPEPhos-benzophenone-5.0%IPA-1.0mL_minADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.632	12769339	498328	49.746	57.006
2	21.332	12899764	375837	50.254	42.994
Total		25669103	874165	100.000	100.000

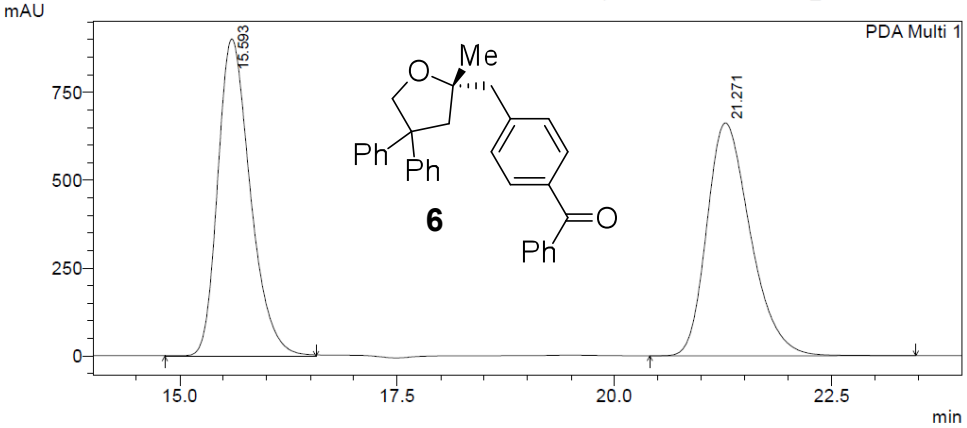
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Zac G\THFs\CHIRAL-ZJG-3-97-1st-L7-benzophenone-5.0%IPA-1.0mL_minADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-97-1st-L7-benzophenone-5.0%IPA-1.0mL_minADH.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-97-1st-L7-benzophenone-5.0%IPA-1.0mL_minADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 8/14/2015 11:33:50 AM
Data Processed	: 8/14/2015 11:59:52 AM

<Chromatogram>

C:\LabSolutions\Data\Zac G\THFs\CHIRAL-ZJG-3-97-1st-L7-benzophenone-5.0%IPA-1.0mL_minADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.593	23625033	903116	50.544	57.673
2	21.271	23116394	662804	49.456	42.327
Total		46741427	1565920	100.000	100.000