CHEMISTRY
A European Journal

Supporting Information

Asymmetric Palladium-Catalyzed Alkene Carboamination Reactions for the Synthesis of Cyclic Sulfamides

Zachary J. Garlets, Kaia R. Parenti, and John P. Wolfe*[a]

chem_201600887_sm_miscellaneous_information.pdf

Asymmetric Palladium-Catalyzed Carboamination Reactions for the Synthesis of Cyclic Sulfamides

Zachary J. Garlets, Kaia R. Parenti, and John P. Wolfe*

Department of Chemistry, University of Michigan, 930 N. University Avenue, Ann Arbor, Michigan 48109-1055

Supporting Information

Experimental procedures and characterization data for new compounds.

Table of Contents

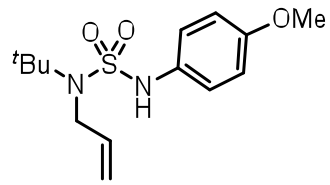
General: Reactions were carried out under nitrogen in flame-dried or oven-dried glassware unless otherwise specified. Tris(dibenzylideneacetone)dipalladium and (*S*)-SIPHOS-PE were purchased from Strem Chemical Co. and used without further purification. Dichloromethane and toluene were purified using a GlassContour solvent

system. Xylenes were purified by distillation over CaH_2 prior to use in reactions. 1-Allyl-1,3-bisbenzylsulfamide (**1f**)[1] was prepared according to published procedures. All other solvents and aryl halides were purchased from commercial sources and used as received. Yields refer to isolated yields of compounds that are estimated to be ≥95% pure as judged by ^{1}H NMR or GC analysis. Unless otherwise noted, yields reported in the supporting information describe the result of a single experiment, whereas yields reported in Tables 1-3 and eq 2-3 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:

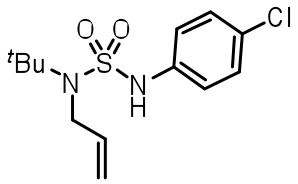
General procedure 1 for the synthesis of substrates:

A flame dried two neck round bottom flask equipped with a stir bar, condenser, and septum was cooled under a stream of nitrogen and charged with DMAP (0.20 equiv.) and oxazolidin-2-one substrate (1.0 equiv.). Anhydrous acetonitrile (5 mL/mmol) was added followed by triethylamine (3.0 equiv.). The reaction mixture was heated at 80 °C for 15 min, and then the allyl amine was added dropwise. The resulting mixture was stirred for 16-18 h at 80 °C The reaction mixture was cooled to rt, and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel using hexanes/ethyl acetate as eluant.

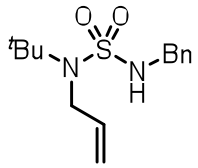


***N*-Allyl-*N'*-(4-methoxyphenyl)-*N*-*tert*-butylsulfamide (1a).** General procedure 1 was

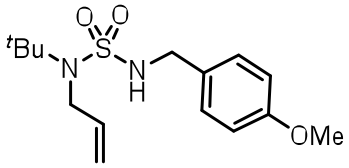
used to sulfonylate *N*-(*tert*-butyl)prop-2-en-1-amine (1.2 g, 10.5 mmol) with 3-[(4-methoxyphenyl)sulfonyl]oxazolidin-2-one (2.6 g, 9.5 mmol) to afford the title compound (2.2 g, 79%) as a yellow solid, mp 77–81 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.17–7.11 (m, 2 H), 6.87–6.82 (m, 2 H), 6.22 (s, br, 1 H), 5.78 (m, 1 H), 5.12–5.06 (m, 1 H), 5.02 (dd, *J* = 10.3, 1.2 Hz, 1 H), 3.89 (dd, *J* = 6.0, 1.3 Hz, 2 H), 3.80 (s, 3 H), 1.40 (s, 9 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 157.6, 137.4, 130.1, 124.7, 116.9, 114.5, 59.7, 55.6, 50.1, 30.0; IR (neat) 3289, 2934, 1510, 1128 cm^{-1}; MS (ESI+) 299.1422 (299.1424 calcd for $C_{14}H_{22}N_2O_3S$, M + H+).



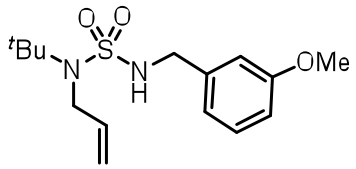
***N*-Allyl-*N'*-(4-chlorophenyl)-*N*-*tert*-butylsulfamide (1b).** General procedure 1 was used to acylate *N*-(*tert*-butyl)prop-2-en-1-amine (1.2 g, 10.5 mmol) with 3-((4-chlorophenyl)sulfonyl)oxazolidin-2-one (2.6 g, 9.5 mmol) to afford the title compound (2.0 g, 68%) as a colorless solid, mp 74–78 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.22–7.35 (m, 2 H), 7.04–7.14 (m, 2 H), 6.58 (s, 1 H), 5.71–5.84 (m, 1 H), 5.14 (dd, *J* = 17.2, 0.7 Hz, 1 H), 5.07 (dd, *J* = 10.3, 0.5 Hz, 1 H), 3.95 (d, *J* = 5.9 Hz, 2 H), 1.40 (s, 9 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 136.9, 136.1, 130.0, 129.5, 121.8, 116.9, 60.0, 50.1, 29.9; IR (neat) 3327, 3253, 2978, 1317, 1132 cm^{-1}; MS (ESI+) 325.0747 (325.0748 calcd for $C_{13}H_{19}ClN_2O_2S$, M + Na+).



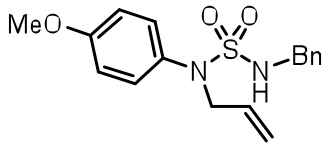
***N*-Allyl-*N'*-benzyl-*N*-*tert*-butylsulfamide (1c).** General procedure 1 was used to sulfonylate *N*-(*tert*-butyl)prop-2-en-1-amine (3.1 mL, 20.9 mmol) with 3-(benzylsulfonyl)oxazolidin-2-one (4.87 g, 19 mmol) to afford the title compound (3.3 g, 61%) as a colorless solid. Spectra were identical to those previously reported.[2]



***N*-Allyl-*N'*-(4-methoxybenzyl)-*N*-*tert*-butylsulfamide (1d).** General procedure 1 was used to sulfonylate *N*-(*tert*-butyl)prop-2-en-1-amine (1.2 g, 10.5 mmol) with 3-[(4-methoxybenzyl)sulfonyl]oxazolidin-2-one (2.7 g, 9.5 mmol) to afford the title compound (2.6 g, 87%) as a colorless solid, mp 66–69 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27–7.22 (m, 2 H), 6.89–6.85 (m, 2 H), 6.03–5.92 (m, 1 H), 5.22 (dd, *J* = 17.4, 1.5 Hz, 1 H), 5.13 (dd, *J* = 10.3, 1.5 Hz, 1 H), 4.20–4.14 (m, 1 H), 4.08 (d, *J* = 6.1 Hz, 2 H), 3.95 (dt, *J* = 5.9, 1.4 Hz, 2 H), 3.80 (s, 3 H), 1.46 (s, 9 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 159.5, 137.5, 129.6, 128.9, 116.6, 114.3, 59.4, 55.5, 49.5, 47.0, 29.9; IR (neat) 3325, 1513, 1313, 1248 cm^{-1}; MS (ESI+) 313.1581 (313.158 calcd for $C_{15}H_{24}N_2O_3S$, M + H+).

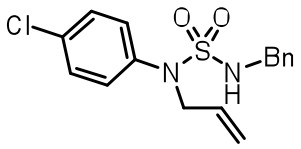


***N*-Allyl-*N'*-(3-methoxybenzyl)-*N*-*tert*-butylsulfamide (1e).** General procedure 1 was used to sulfonylate *N*-(*tert*-butyl)prop-2-en-1-amine (1.2 g, 10.5 mmol) with 3-[(3-methoxybenzyl)sulfonyl]oxazolidin-2-one (2.7 g, 9.5 mmol) to afford the title compound (1.7 g, 58%) as a yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25 (t, *J* = 7.8 Hz, 1 H), 6.92–6.86 (m, 2 H), 6.83 (dd, *J* = 8.3, 2.4 Hz, 1 H), 6.02–5.92 (m, 1 H), 5.22 (dd, *J* = 17.2, 1.3 Hz, 1 H), 5.13 (dd, *J* = 10.3, 1.2 Hz, 1 H), 4.35 (t, *J* = 5.0 Hz, 1 H), 4.11 (d, *J* = 6.1 Hz, 2 H), 3.95 (dt, *J* = 6.0, 1.3 Hz, 2 H), 3.80 (s, 3 H), 1.46 (s, 9 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 160.0, 138.5, 137.5, 129.9, 120.4, 116.7, 113.7, 113.6, 59.4, 55.4, 49.5, 47.4, 29.8; IR (neat) 3320, 3250, 1317, 1135 cm^{-1}; MS (ESI+) 313.1576 (313.158 calcd for $C_{15}H_{24}N_2O_3S$, M + H+).

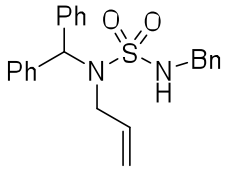


***N*-Allyl-*N'*-benzyl-*N*-(4-methoxyphenyl)sulfamide (1g).** General procedure 1 was used to sulfonylate *N*-allyl-4-methoxyaniline (1.71 g, 10.5 mmol) with 3-(benzylsulfonyl)oxazolidin-2-one (2.5 g, 9.5 mmol) to afford the title compound (2.2 g, 70%) as a light brown solid, mp 58–61 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36–7.32 (m, 2 H), 7.31–7.27 (m, 3 H), 7.27–7.23 (m, 2 H), 6.89–6.85 (m, 2 H), 5.86–5.77 (m, 1 H), 5.13–5.10 (m, 1 H), 5.08 (t, *J* = 1.2 Hz, 1 H), 4.52 (t, *J* = 6.0 Hz, 1 H), 4.23 (d, *J* = 6.1 Hz, 2 H), 4.18 (dt, *J* = 6.5, 1.1 Hz, 2 H), 3.80 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ

159.2, 136.8, 133.4, 132.8, 130.2, 128.9, 128.1, 128.1, 119.0, 114.5, 55.6, 55.3, 47.8; IR (neat) 3289, 1510, 1331, 1146 cm^{-1}; MS (ESI+) 333.1624 (333.1627 calcd for $C_{17}H_{20}N_2O_3S$, M + H+).

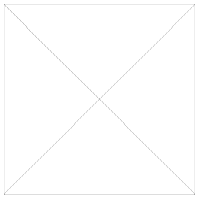


***N*-Allyl-*N'*-benzyl-*N*-(4-chlorophenyl)sulfamide (1h).** General procedure 1 was used to sulfonylate *N*-allyl-4-chloroaniline (1.76 g, 10.5 mmol) with 3-(benzylsulfonyl)oxazolidin-2-one (2.5 g, 9.5 mmol) to afford the title compound (1.39 g, 43%) as a colorless solid, mp 61–63 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37–7.28 (m, 5 H), 7.28–7.22 (m, 4 H), 5.83–5.73 (m, 1 H), 5.12 (s, 1 H), 5.11–5.07 (m, 1 H), 4.58 (s, br, 1 H), 4.22 (d, *J* = 5.9 Hz, 2 H), 4.19 (dd, *J* = 6.5, 1.1 Hz, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 138.8, 136.6, 133.6, 133.0, 129.8, 129.5, 129.0, 128.3, 128.1, 119.4, 54.9, 47.8; IR (neat) 3294, 1488, 1338, 1145 cm^{-1}; MS (ESI+) 337.0769 (337.0772 calcd for $C_{16}H_{17}ClN_2O_2S$, M + H+).



***N*-Allyl-*N*-benzhydryl-*N'*-benzylsulfamide (1i).** General procedure 1 was used to sulfonylate *N*-benzhydrylprop-2-en-1-amine (1.52 g, 6.8 mmol) with 3-(benzylsulfonyl)oxazolidin-2-one (1.59 g, 6.2 mmol) to afford the title compound (1.2 g, 48%) as a colorless solid, mp 100–102 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39–7.27 (m,

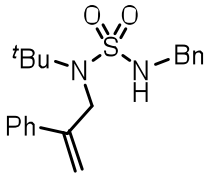
12 H), 7.19–7.15 (m, 2 H), 6.39 (s, 1 H), 5.40 (ddt, *J* = 17.0, 10.3, 6.5 Hz, 1 H), 4.98 (dd, *J* = 17.2, 1.3 Hz, 1 H), 4.92 (dd, *J* = 10.1, 1.1 Hz, 1 H), 4.23 (t, *J* = 6.0 Hz, 1 H), 4.05 (d, *J* = 6.1 Hz, 2 H), 3.95 (d, *J* = 6.4 Hz, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 139.5, 136.6, 134.6, 129.2, 128.9, 128.7, 128.2, 128.1, 128.0, 118.2, 65.9, 49.5, 47.4; IR (neat) 3300, 1433, 1324, 1142 cm^{-1}; MS (ESI+) 393.1628 (393.1631 calcd for $C_{23}H_{24}N_2O_2S$, M + H+).



***N'*-Benzyl-*N*-(*tert*-butyl)-*N*-(2-methylallyl)sulfamide (4a).** A flame-dried round-bottom flask equipped with a stirbar and a septum was cooled under a stream of nitrogen and charged with *tert*-butylamine (6.3 mL, 60 mmol), 3-bromo-2-methylprop-1-ene (1.51, 15 mmol), and solid potassium carbonate (2.5 g, 18 mmol). The resulting mixture was stirred at rt for 24 h, then the mixture was filtered through celite. The celite was washed 3 x 20 mL diethyl ether. The organic layers were combined, and the solvent was removed under reduced pressure to afford crude *N*-(tert-butyl)-2-methylprop-2-en-1-amine (1.5 g, 80%) as a colorless oil which was carried on without further purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.86 (s, 1 H), 4.79 (s, 1 H), 3.10 (s, 2 H), 1.77 (s, 3 H), 1.11 (s, 9 H)

General procedure 1 was used to sulfonylate *N*-(*tert*-butyl)-2-methylprop-2-en-1-amine (1.34 g, 10.5 mmol) with 3-(benzylsulfonyl)oxazolidin-2-one (2.5 g, 9.5 mmol) to afford the title compound (1.3 g, 47%) as a colorless solid, mp 76–80 °C. ^{1}H NMR (400

MHz, $CDCl_3$) δ 7.38–7.27 (m, 5 H), 5.07 (*d*, J = 0.7 Hz, 1 H), 4.93 (d, *J* = 1.2 Hz, 1 H), 4.21 (s, 3 H), 3.86 (s, 2 H), 1.77 (s, 3 H), 1.49–1.43 (m, 9 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 144.0, 137.1, 128.9, 128.3, 128.1, 111.2, 59.6, 52.4, 47.8, 29.6, 20.5; IR (neat) 3327, 2977, 1321, 1133 cm^{-1}; MS (ESI+) 297.1629 (297.1631 calcd for $C_{15}H_{24}N_2O_2S$, M + H+).



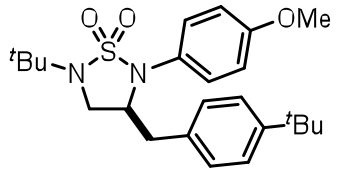
***N'*-Benzyl-*N*-(*tert*-butyl)-*N*-(2-phenylallyl)sulfamide (4b).** The alkylation of *tert*-butylamine (6.3 mL, 60 mmol) with (3-bromoprop-1-en-2-yl)benzene (2.96 g, 15 mmol) in the presence of potassium carbonate (2.5 g, 18 mmol) was accomplished using a procedure analogous to that described above for the formation of **4a**. This procedure afforded *N*-(*tert*-butyl)-2-phenylprop-2-en-1-amine (2.4 g, 83%), which was carried on without further purification. ^1H NMR (500 MHz, $CDCl_3$) δ 7.46 (d, *J* = 7.3 Hz, 2 H), 7.37–7.31 (m, 2 H), 7,29 (d, *J* = 7.3 Hz, 1 H), 5.38 (s, 1 H), 5.28 (s, 1 H), 3.62 (s, 2 H), 1.15 (s, 9 H).

General procedure 1 was used to sulfonylate *N*-(*tert*-butyl)-2-phenylprop-2-en-1-amine (2.0 g, 10.5 mmol) with 3-(benzylsulfonyl)oxazolidin-2-one (2.5 g, 9.5 mmol) to afford the title compound (2.4 g, 71%) as a colorless solid, mp 64–68 °C. ^1H NMR (500 MHz, $CDCl_3$) δ 7.44–7.25 (m, 10 H), 5.55–5.43 (m, 2 H), 4.31 (s, 2 H), 4.24–4.17 (m, 3 H), 1.53 (s, 9 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 146.7, 140.0, 137.0, 128.9, 128.6, 128.2, 128.1, 128.1, 126.4, 113.2, 59.9, 50.4, 47.9, 29.2; IR (neat) 3330, 2969, 1320,

1137 cm^{-1}; MS (ESI+) 359.1783 (359.1788 calcd for $C_{20}H_{26}N_2O_2S$, M + H+).

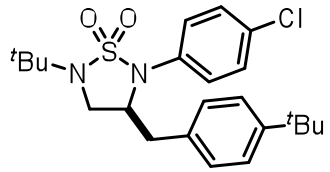
General procedure 2 for asymmetric Pd-catalyzed carboamination reactions.

An oven-dried test tube equipped with a stir bar and a rubber septum was cooled under a stream of nitrogen and charged with $Pd_2(dba)_3$ (1 mol %), *S*-SIPHOS-PE (5 mol %), the sulfamide substrate (1.0 equiv), and NaO^{t}Bu (2.0 equiv). The septum-capped tube was purged with N_2, and then the aryl or alkenyl halide (2.0 equiv), water (0 or 2.0 equiv.), and xylenes (0.125 M) were added. The resulting mixture was heated to 120 °C with stirring for 18 h. The reaction mixture was then cooled to rt and saturated aqueous ammonium chloride (6 mL/mmol substrate) was added. The mixture was extracted with ethyl acetate (3 x 2 mL) and then the combined organic layers were dried over anhydrous Na_2SO_4, filtered through a plug of celite, and concentrated *in vacuo*. The crude product was purified by flash chromatography on silica gel using hexanes/ethyl acetate as eluant.



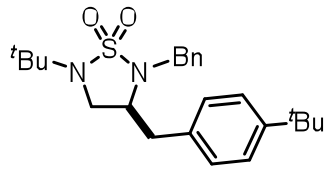
(–)-(*S*)-5-(*tert*-Butyl)-3-[4-(*tert*-butyl)benzyl]-2-(4-methoxyphenyl)-1,2,5-thiadiazolidine-1,1-dioxide (2a). The general procedure 2 was employed for the coupling of *N*-allyl-*N'*-(4-methoxyphenyl)-*N*-*tert*-butylsulfamide (89.0 mg, 0.30 mmol) and 1-bromo-4-(*tert*-butyl)benzene (104 µL, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction

time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (61 mg, 47%) as a yellow oil: $[\alpha]^{23}_{D}$ –21.5 (*c* 1.9, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37–7.33 (m, 2 H), 7.32–7.28 (m, 2 H), 7.02 (d, *J* = 8.3 Hz, 2 H), 6.96–6.92 (m, 2 H), 4.12–4.05 (m, 1 H), 3.82 (s, 3 H), 3.32 (dd, *J* = 8.7, 6.0 Hz, 1 H), 3.24–3.18 (m, 1 H), 2.95 (dd, *J* = 13.7, 4.2 Hz, 1 H), 2.63 (dd, *J* = 13.8, 9.8 Hz, 1 H), 1.42 (s, 9 H), 1.30 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.2, 150.1, 132.9, 128.9, 128.7, 128.4, 125.8, 115.0, 57.4, 56.6, 55.7, 46.4, 38.0, 34.6, 31.5, 27.5; IR (neat) 2963, 1509, 1149 cm^{-1}; MS (ESI+) 431.2359 (431.2363 calcd for $C_{24}H_{34}N_2O_3S$, M + H^+). The enantiopurity was determined to be 81:19 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 7.2 and 9.9 min).



(–)-(*S*)-5-(*tert*-Butyl)-3-[4-(*tert*-butyl)benzyl]-2-(4-chlorophenyl)-1,2,5-thiadiazolidine 1,1-dioxide (2b). The general procedure 2 was employed for the coupling of *N*-allyl-*N'*-(4-chlorophenyl)-*N*-*tert*-butylsulfamide (76.0 mg, 0.25 mmol) and 1-bromo-4-(*tert*-butyl)benzene (87 μL, 0.50 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (S)-SIPHOS-PE (7.6 mg, 0.015 mmol), sodium *tert*-butoxide (48.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (12 mg, 11%) yellow oil: $[\alpha]^{23}_{D}$ –10.0 (*c* 0.1, CH_2Cl_2); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.5 Hz, 2 H), 7.30 (t, *J* = 7.9 Hz, 4 H), 7.03 (d, *J* = 8.0 Hz, 2 H), 4.20–4.14 (m, 1 H), 3.36–3.40 (m, 1 H), 3.25 (t, *J* = 8.2 Hz, 1 H), 3.00 (dd, *J* = 13.9, 3.8 Hz, 1 H), 2.70 (dd, *J*

= 13.8, 9.7 Hz, 1 H), 1.43 (s, 9 H), 1.31 (s, 9 H); ^{13}C NMR (176 MHz, $CDCl_3$) δ 150.3. 135.3, 132.7, 132.3, 129.8, 128.9, 125.9, 125.7, 57.0, 56.4, 46.0, 37.8, 34.7, 31.5, 27.6; IR (film) 2966, 1491, 1150 cm^{-1}. MS (ESI+) 435.1865 (435.1868 calcd for $C_{23}H_{31}ClN_2O_2S$, M + H$^+$). The enantiopurity was determined to be 73:27 er by chiral HPLC analysis (Chiralcel ADH, 25 cm x 4.6 mm, 10% IPA/Hexanes, 0.8 mL/min, λ 254 nm, RT= 8.8 and 18.0 min).

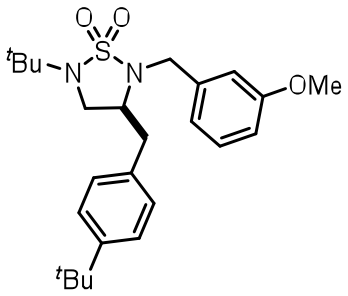


(–)-(*S*)-2-Benzyl-5-(*tert*-butyl)-3-[4-(*tert*-butyl)benzyl]-1,2,5-thiadiazolidine-1,1-dioxide (2c). The general procedure 2 was employed for the coupling of *N*-allyl-*N*'-benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and 1-bromo-4-(*tert*-butyl)benzene (104 μL, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 μL, 0.60 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (112 mg, 90%) as a colorless solid, mp 114–118 °C: $[\alpha]^{23}_D$ –2.5 (*c* 10.1, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43–7.38 (m, 2 H), 7.37–7.32 (m, 2 H), 7.31–7.23 (m, 3 H), 6.91 (d, *J* = 8.2 Hz, 2 H), 4.38 (d, *J* = 14.9 Hz, 1 H), 4.21 (d, *J* = 14.9 Hz, 1 H), 3.47–3.38 (m, 1 H), 3.18 (dd, *J* = 8.8, 6.5 Hz, 1 H), 3.06 (dd, *J* = 8.9, 6.2 Hz, 1 H), 2.93 (dd, *J* = 13.5, 4.3 Hz, 1 H), 2.61 (dd, *J* = 13.4, 10.1 Hz, 1 H), 1.40 (s, 9 H), 1.28 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.0, 136.0, 133.5, 129.0, 128.8, 128.7, 128.0, 125.8, 56.6, 56.3, 50.0, 45.8, 38.0, 34.6, 31.5, 27.5; IR (neat) 2962, 1455, 1309, 1148 cm^{-1}; MS (ESI+) 437.2233 (437.2233

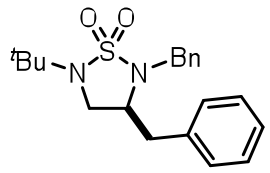
calcd for $C_{24}H_{34}N_2O_2S$, M + Na^+). The enantiopurity was determined to be 94:6 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 205 nm, RT= 4.1 and 5.0 min).

(–)-(*S*)-5-(*tert*-Butyl)-3-[4-(*tert*-butyl)benzyl]-2-(4-methoxybenzyl)-1,2,5-thiadiazolidine-1,1-dioxide (2d). The general procedure 2 was employed for the coupling of *N*-allyl-*N'*-(4-methoxybenzyl)-*N*-*tert*-butylsulfamide (94.0 mg, 0.30 mmol) and 1-bromo-4-(*tert*-butyl)benzene (104 µL, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (103 mg, 77%) as a colorless solid, mp 82–86 °C: $[\alpha]^{23}_D$ –9.6 (*c* 7.5, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.6 Hz, 2 H), 7.30–7.27 (m, 2 H), 6.94 (d, *J* = 8.3 Hz, 2 H), 6.90–6.86 (m, 2 H), 4.30 (d, *J* = 14.7 Hz, 1 H), 4.20 (d, *J* = 14.7 Hz, 1 H), 3.81 (s, 3 H), 3.46–3.39 (m, 1 H), 3.18 (dd, *J* = 8.9, 6.5 Hz, 1 H), 3.05 (dd, *J* = 8.9, 6.0 Hz, 1 H), 2.95 (dd, *J* = 13.5, 4.3 Hz, 1 H), 2.62 (dd, *J* = 13.4, 10.0 Hz, 1 H), 1.40 (s, 9 H), 1.30 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.5, 150.0, 133.6, 130.4, 128.9, 127.8, 125.8, 114.3, 56.3, 56.2, 55.4, 49.4, 45.7, 38.0, 34.6, 31.5, 27.6; IR (neat) 2966, 1514, 1150 cm^{-1}; MS (ESI+) 445.2510 (445.2519 calcd for $C_{25}H_{36}N_2O_3S$, M + H^+). The enantiopurity was determined to be 91:9 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7%

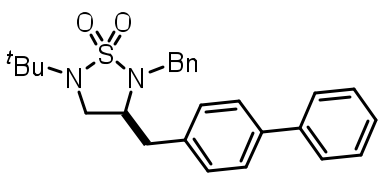
IPA/Hexanes, 1.00 mL/min, λ 215 nm, RT= 5.0 and 6.2 min).



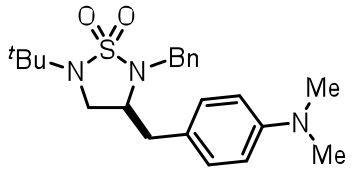
(–)-(*S*)-5-(*tert*-Butyl)-3-[4-(*tert*-butyl)benzyl]-2-(3-methoxybenzyl)-1,2,5-thiadiazolidine-1,1-dioxide (2e). The general procedure 2 was employed for the coupling of *N*-allyl-*N'*-(3-methoxybenzyl)-*N*-*tert*-butylsulfamide (94.0 mg, 0.30 mmol) and 1-bromo-4-(tert-butyl)benzene (104 µL, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (80 mg, 60%) as a yellow oil: $[\alpha]^{23}_{D}$ –4.4 (*c* 6.3, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29–7.22 (m, 3 H), 7.00–6.92 (m, 2 H), 6.92 (d, *J* = 8.2 Hz, 2 H), 6.85–6.81 (m, 1 H), 4.35 (d, *J* = 14.7 Hz, 1 H), 4.18 (d, *J* = 14.7 Hz, 1 H), 3.80 (s, 3 H), 3.47–3.39 (m, 1 H), 3.18 (dd, *J* = 8.8, 6.5 Hz, 1 H), 3.05 (dd, *J* = 8.9, 6.2 Hz, 1 H), 2.93 (dd, *J* = 13.5, 4.3 Hz, 1 H), 2.61 (dd, *J* = 13.4, 10.1 Hz, 1 H), 1.39 (s, 9 H), 1.27 (s, 9 H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.0, 150.0, 137.6, 133.5, 129.7, 128.9, 125.8, 121.2, 114.2, 113.8, 56.6, 56.3, 55.4, 50.0, 45.8, 37.9, 34.6, 31.5, 27.3; IR (neat) 2963, 1600, 1150 cm^{-1}; MS (ESI+) 445.2513 (445.2519 calcd for $C_{25}H_{36}N_2O_3S$, M + H^+). The enantiopurity was determined to be 91:9 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 230 nm, RT= 5.2 and 7.3 min).



(–)-(*S*)-2,3-Dibenzyl-5-(*tert*-butyl)-1,2,5-thiadiazolidine-1,1-dioxide (3a). The general procedure 2 was employed for the coupling of *N*-allyl-*N*'-benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and bromobenzene (63 μL, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 μL, 0.60 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (101 mg, 94%) as a colorless solid, mp 108–111 °C: $[\alpha]^{23}_{D}$ – 8.5 (*c* 4.6, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (d, *J* = 7.3 Hz, 2 H), 7.36 (t, *J* = 7.3 Hz, 2 H), 7.33–7.19 (m, 4 H), 7.00 (d, *J* = 7.3 Hz, 2 H), 4.39 (d, *J* = 14.7 Hz, 1 H), 4.22 (d, *J* = 15.2 Hz, 1 H), 3.49–3.42 (m, 1 H), 3.19–3.13 (m, 1 H), 3.05 (dd, *J* = 8.1, 7.1 Hz, 1 H), 2.98 (dd, *J* = 13.7, 4.4 Hz, 1 H), 2.64 (dd, *J* = 13.2, 10.3 Hz, 1 H), 1.40 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.6, 136.0, 129.2, 129.0, 128.9, 128.8, 128.1, 127.1, 56.6, 56.4, 50.1, 45.7, 38.6, 27.5; IR (neat) 2962, 1314, 1150 cm^{-1}; MS (ESI+) 381.1607 (381.1607 calcd for $C_{20}H_{26}N_2O_2S$, M + Na^+). The enantiopurity was determined to be 93:7 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 4% IPA/Hexanes, 0.5 mL/min, λ 210 nm, RT= 18.6 and 20.7 min).

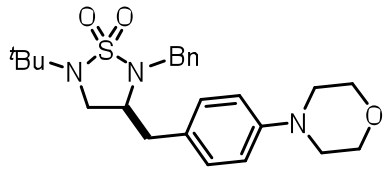


(+)-(*S*)-3-[(1,1'-Biphenyl)-4-ylmethyl]-2-benzyl-5-(*tert*-butyl)-1,2,5-thiadiazolidine-1,1-dioxide (3b). The general procedure 2 was employed for the coupling of *N*-allyl-*N*'-benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and 4-bromobiphenyl (140.0 mg, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 μL, 0.60 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (122 mg, 94%) as a colorless solid, mp 109–113 °C: $[\alpha]^{23}_D$ +0.51 (*c* 3.1, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58–7.54 (m, 2 H), 7.50 (d, *J* = 8.1 Hz, 2 H), 7.46–7.41 (m, 4 H), 7.40–7.30 (m, 4 H), 7.08 (d, *J* = 8.1 Hz, 2 H), 4.43 (d, *J* = 14.9 Hz, 1 H), 4.24 (d, *J* = 14.9 Hz, 1 H), 3.51 (d, *J* = 5.1 Hz, 1 H), 3.23 (dd, *J* = 8.9, 6.5 Hz, 1 H), 3.10 (dd, *J* = 8.8, 6.1 Hz, 1 H), 3.03 (dd, *J* = 13.4, 4.6 Hz, 1 H), 2.70 (dd, *J* = 13.4, 10.0 Hz, 1 H), 1.42 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.7, 140.0, 136.0, 135.6, 129.6, 129.0, 129.0, 128.8, 128.1, 127.6, 127.5, 127.1, 56.6, 56.4, 50.2, 45.8, 38.3, 27.6; IR (neat) 2974, 1487, 1311, 1151 cm^{-1}; MS (ESI+) 435.2094 (435.2101 calcd for $C_{26}H_{30}N_2O_2S$, M + H^+). The enantiopurity was determined to be 94:6 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 14.8 and 24.7 min).



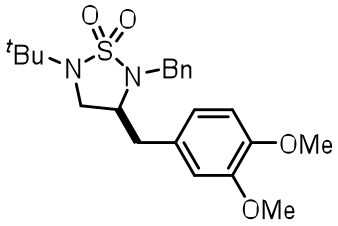
(+)-(*S*)-2-Benzyl-5-(*tert*-butyl)-3-[4-(dimethylamino)benzyl]-1,2,5-thiadiazolidine-1,1-dioxide (3c). The general procedure 2 was employed for the coupling of *N*-allyl-*N*'-

benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and 4-bromo-*N,N*-dimethylaniline (120.0 mg, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 μL, 0.60 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (91 mg, 76%) as a red solid, mp 118–122 °C: $[\alpha]^{23}_D$ +17.3 (*c* 5.1, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (d, *J* = 7.8 Hz, 2 H), 7.37 (t, *J* = 7.7 Hz, 2 H), 7.34–7.29 (m, 1 H), 6.88 (d, *J* = 8.1 Hz, 2 H), 6.64 (d, *J* = 7.8 Hz, 2 H), 4.42 (d, *J* = 14.9 Hz, 1 H), 4.23 (d, *J* = 15.1 Hz, 1 H), 3.40 (d, *J* = 5.9 Hz, 1 H), 3.17 (dd, *J* = 8.2, 7.2 Hz, 1 H), 3.04–3.10 (m, 1 H), 2.88–2.95 (m, 7 H), 2.54 (dd, *J* = 13.3, 10.4 Hz, 1 H), 1.41 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.7, 136.2, 129.8, 128.9, 128.7, 128.0, 124.0, 113.0, 57.0, 56.3, 49.9, 45.8, 40.8, 37.6, 27.5; IR (neat) 2900, 1615, 1524, 1303 cm^{-1}; MS (ESI+) 402.2206 (402.2210 calcd for $C_{22}H_{31}N_3O_2S$, M + H^+). The enantiopurity was determined to be 95:5 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 7.4 and 9.5 min).



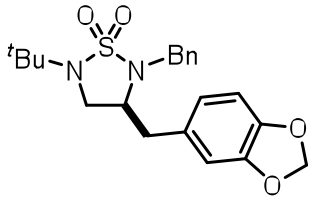
(–)-(*S*)-2-Benzyl-5-(*tert*-butyl)-3-(4-morpholinobenzyl)-1,2,5-thiadiazolidine-1,1-dioxide (3d). The general procedure 2 was employed for the coupling of *N*-allyl-*N*'-benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and 4-(4-bromophenyl)morpholine (145.0 mg, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol)

and (S)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 μL, 0.60 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (105 mg, 79%) as a yellow solid, mp 95–99 °C: $[\alpha]^{23}_{D}$ –11.5 (*c* 5.5, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44–7.41 (m, 2 H), 7.38–7.33 (m, 2 H), 7.31 (d, *J* = 7.1 Hz, 1 H), 6.90 (d, *J* = 8.6 Hz, 2 H), 6.80 (d, *J* = 8.6 Hz, 2 H), 4.40 (d, *J* = 14.9 Hz, 1 H), 4.20 (d, *J* = 14.9 Hz, 1 H), 3.87–3.82 (m, 4 H), 3.40 (d, *J* = 5.6 Hz, 1 H), 3.15 (dd, *J* = 8.8, 6.6 Hz, 1 H), 3.13–3.09 (m, 4 H), 3.03 (dd, *J* = 8.9, 6.5 Hz, 1 H), 2.91 (dd, *J* = 13.6, 4.5 Hz, 1 H), 2.56 (dd, *J* = 13.4, 10.0 Hz, 1 H), 1.39 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.3, 136.1, 129.9, 129.0, 128.7, 128.0, 127.7, 116.1, 67.0, 56.8, 56.3, 50.1, 49.5, 45.8, 37.7, 27.5; IR (neat) 2814, 1598, 1516, 1151 cm^{-1}; MS (ESI+) 444.2311 (444.2315 calcd for $C_{24}H_{33}N_3O_3S$, M + H^+). The enantiopurity was determined to be 94:6 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 18.2 and 22.6 min).



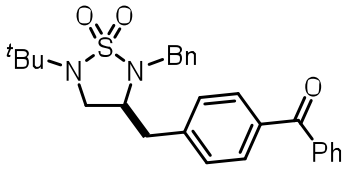
(–)-(*S*)-2-Benzyl-5-(*tert*-butyl)-3-(3,4-dimethoxybenzyl)-1,2,5-thiadiazolidine-1,1-dioxide (3e). The general procedure 2 was employed for the coupling of *N*-allyl-*N*'-benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and 4-bromo-1,2-dimethoxybenzene (86 μL, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 μL, 0.60 mmol), sodium *tert*-butoxide

(58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (99 mg, 79%) as a yellow oil: $[\alpha]^{23}_{D}$ –5.8 (*c* 5.3, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41–7.36 (m, 2 H), 7.36–7.23 (m, 3 H), 6.73 (d, *J* = 8.2 Hz, 1 H), 6.54 (dd, *J* = 8.1, 1.9 Hz, 1 H), 6.42 (d, *J* = 1.8 Hz, 1 H), 4.36 (d, *J* = 14.9 Hz, 1 H), 4.18 (d, *J* = 14.9 Hz, 1 H), 3.82 (s, 3 H), 3.76 (s, 3 H), 3.44–3.36 (m, 1 H), 3.16 (dd, *J* = 8.8, 6.5 Hz, 1 H), 3.02 (dd, *J* = 8.9, 6.2 Hz, 1 H), 2.90 (dd, *J* = 13.4, 4.6 Hz, 1 H), 2.56 (dd, *J* = 13.5, 9.6 Hz, 1 H), 1.38 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.2, 148.1, 136.0, 129.1, 129.0, 128.7, 128.0, 121.2, 112.2, 111.5, 56.7, 56.3, 56.1, 56.0, 50.2, 45.8, 38.3, 27.5; IR (neat) 2976, 1661, 1514, 1150 cm^{-1}; MS (ESI+) 419.1997 (419.1999 calcd for $C_{22}H_{30}N_2O_4S$, M + H^+). The enantiopurity was determined to be 94:6 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 233 nm, RT= 15.1 and 20.8 min).



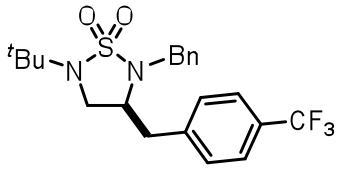
(–)-(*S*)-3-(Benzo[*d*][1,3]dioxol-5-ylmethyl)-2-benzyl-5-(*tert*-butyl)-1,2,5-thiadiazolidine-1,1-dioxide (3f). The general procedure 2 was employed for the coupling of *N*-Allyl-*N*'-benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and 5-bromobenzo[*d*][1,3]dioxole (72 µL, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 µL, 0.60 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title

compound (88 mg, 73%) as a colorless solid, mp 99–102 °C: $[\alpha]^{23}_D$ –14.2 (*c* 4.7, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44–7.39 (m, 2 H), 7.36 (t, *J* = 7.3 Hz, 2 H), 7.31 (d, *J* = 7.3 Hz, 1 H), 6.69 (d, *J* = 7.8 Hz, 1 H), 6.48–6.42 (m, 2 H), 5.92 (s, 2 H), 4.40 (d, *J* = 14.9 Hz, 1 H), 4.19 (d, *J* = 14.9 Hz, 1 H), 3.42–3.34 (m, 1 H), 3.21–3.15 (m, 1 H), 3.03 (dd, *J* = 8.6, 6.4 Hz, 1 H), 2.88 (dd, *J* = 13.6, 4.5 Hz, 1 H), 2.54 (dd, *J* = 13.4, 10.0 Hz, 1 H), 1.40 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 148.0, 146.7, 136.0, 130.2, 129.0, 128.8, 128.1, 122.2, 109.3, 108.7, 101.2, 56.8, 56.4, 50.3, 45.7, 38.4, 27.6; IR (neat) 2976, 1493, 1313, 1150 cm^{-1}; MS (ESI+) 403.1688 (403.1686 calcd for $C_{21}H_{26}N_2O_4S$, M + H^+). The enantiopurity was determined to be 92:8 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 10% IPA/Hexanes, 1.00 mL/min, λ 280 nm, RT= 7.0 and 11.6 min).



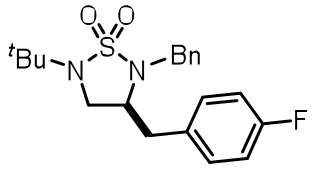
(–)-(S)-(4-{[2-benzyl-5-(*tert*-butyl)-1,1-dioxido-1,2,5-thiadiazolidin-3-yl]methyl}phenyl)(phenyl)methanone (3g). The general procedure 2 was employed for the coupling of *N*-allyl-*N*'-benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and 4-bromobenzophenone (157 mg, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 μL, 0.60 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (106 mg, 76%) as a colorless solid, mp 119–121 °C: $[\alpha]^{23}_D$ –3.3 (*c* 10.0, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (dd, *J* = 8.1, 1.2 Hz, 2 H), 7.70 (d, *J* = 8.1 Hz, 2 H), 7.61–

7.56 (m, 1 H), 7.49–7.45 (m, 2 H), 7.42–7.38 (m, 2 H), 7.37–7.28 (m, 3 H), 7.11 (d, *J* = 8.1 Hz, 2 H), 4.40 (d, *J* = 14.9 Hz, 1 H), 4.21 (d, *J* = 14.9 Hz, 1 H), 3.51 (d, *J* = 4.4 Hz, 1 H), 3.23 (dd, *J* = 8.8, 6.6 Hz, 1 H), 3.08–3.02 (m, 2 H), 2.75 (dd, *J* = 13.4, 9.5 Hz, 1 H), 1.40 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.2, 141.5, 137.6, 136.4, 135.7, 132.6, 130.7, 130.1, 129.2, 128.9, 128.8, 128.4, 128.1, 56.4, 56.3, 50.5, 45.7, 38.7, 27.5; IR (neat) 2976, 1661, 1311, 1151 cm^{-1}; MS (ESI+) 463.2048 (463.2050 calcd for $C_{27}H_{30}N_2O_3S$, M + H^+). The enantiopurity was determined to be 90:10 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 210 nm, RT= 32.7 and 68.3 min).



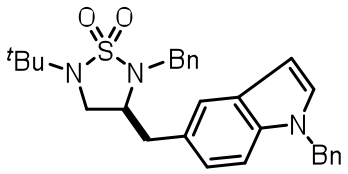
(–)-(*S*)-2-Benzyl-5-(*tert*-butyl)-3-[4-(trifluoromethyl)benzyl]-1,2,5-thiadiazolidine-1,1-dioxide (3h). The general procedure 2 was employed for the coupling of *N*-allyl-*N*'-benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and 4-bromobenzotrifluoride (84 µL, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 µL, 0.60 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (95 mg, 74%) as a colorless solid, mp 139–142 °C: $[\alpha]^{23}_D$ –16.5 (*c* 8.7, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.1 Hz, 2 H), 7.37–7.28 (m, 5 H), 7.10 (d, *J* = 8.1 Hz, 2 H), 4.39 (d, *J* = 14.7 Hz, 1 H), 4.17 (d, *J* = 14.7 Hz, 1 H), 3.50–3.43 (m, 1 H), 3.23 (dd, *J* = 9.0, 6.6 Hz, 1 H), 3.05–2.99 (m, 2 H), 2.72 (dd, *J* = 13.4, 9.3 Hz, 1 H), 1.40 (s, 9 H); ^{13}C NMR (125

MHz, $CDCl_3$) δ 140.8, 135.7, 129.6, 129.6 (q, *J* = 65.4 Hz), 129.0, 128.8, 128.2, 125.8 (q, *J* = 3.77 Hz), 124.2 (q, *J* = 271.6 Hz), 56.4, 56.3, 50.8, 45.8, 38.7, 27.6; ^{19}F NMR (471 MHz, $CDCl_3$) δ –62.56; IR (neat) 2975, 1314, 1152 cm^{-1}; MS (ESI+) 427.1660 (427.1662 calcd for $C_{21}H_{25}F_3N_2O_2S$, M + H^+). The enantiopurity was determined to be 88:12 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 210 nm, RT= 6.0 and 8.3 min).



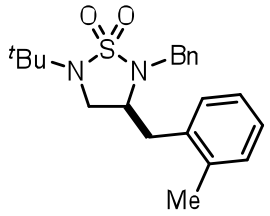
(–)-(*S*)-2-Benzyl-5-(*tert*-butyl)-3-(4-fluorobenzyl)-1,2,5-thiadiazolidine-1,1-dioxide (3i). The general procedure 2 was employed for the coupling of *N*-allyl-*N*'-benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and 1-bromo-4-fluorobenzene (66 µL, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 µL, 0.60 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (81 mg, 72%) as a colorless solid, mp 100–102 °C: $[\alpha]^{23}_D$ –15.6 (*c* 7.8, CH_2Cl_2); ^1H NMR (500 MHz, $CDCl_3$) δ 7.41–7.37 (m, 2 H), 7.35 (t, *J* = 7.2 Hz, 2 H), 7.31 (d, *J* = 7.1 Hz, 1 H), 6.97–6.92 (m, 4 H), 4.38 (d, *J* = 14.7 Hz, 1 H), 4.18 (d, *J* = 14.9 Hz, 1 H), 3.45–3.38 (m, 1 H), 3.18 (dd, *J* = 8.8, 6.6 Hz, 1 H), 3.02 (dd, *J* = 8.8, 6.1 Hz, 1 H), 2.94 (dd, *J* = 13.7, 4.9 Hz, 1 H), 2.62 (dd, *J* = 13.6, 9.7 Hz, 1 H), 1.40 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.0 (d, *J* = 246.5 Hz), 135.9, 132.3 (d,

J = 2.5 Hz), 130.7 (d, *J* = 8.8 Hz), 128.9 (d, *J* = 18.9 Hz), 128.1, 115.9, 115.7, 56.6, 56.4, 50.4, 45.7, 38.0, 27.5; ^{19}F NMR (471 MHz, $CDCl_3$) δ –115.7; IR (neat) 2974, 1509, 1315, 1152 cm^{-1}; MS (ESI+) 377.1694 (377.1694 calcd for $C_{20}H_{25}FN_2O_2S$, M + H^+). The enantiopurity was determined to be 90:10 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 220 nm, RT= 6.1 and 10.5 min).

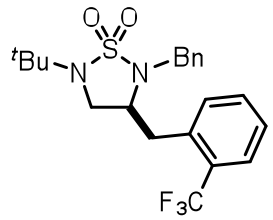


(–)-(*S*)-2-Benzyl-3-[(1-benzyl-1*H*-indol-6-yl)methyl]-5-(*tert*-butyl)-1,2,5-thiadiazolidine-1,1-dioxide (3j). The general procedure 2 was employed for the coupling of *N*-allyl-*N*'-benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and 1-benzyl-5-bromo-1*H*-indole (172.0 mg, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 μL, 0.60 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (122 mg, 83%) as a colorless solid, mp 158–161 °C: $[\alpha]^{23}_D$ –1.6 (*c* 8.6, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (d, *J* = 7.8 Hz, 2 H), 7.40–7.34 (m, 2 H), 7.34–7.25 (m, 5 H), 7.20–7.09 (m, 4 H), 6.79 (d, *J* = 8.5 Hz, 1 H), 6.49 (d, *J* = 2.4 Hz, 1 H), 5.31 (s, 2 H), 4.44 (d, *J* = 14.9 Hz, 1 H), 4.26 (d, *J* = 14.9 Hz, 1 H), 3.55–3.48 (m, 1 H), 3.19–3.08 (m, 3 H), 2.72 (dd, *J* = 13.3, 10.4 Hz, 1 H), 1.41 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 137.5, 136.2, 135.5, 129.2, 129.0, 129.0, 128.9, 128.7, 128.0, 127.8, 127.5, 126.9, 123.0, 121.3, 110.1, 101.5, 57.2, 56.3, 50.4, 50.0, 45.8, 38.7, 27.6; IR (neat) 2974,

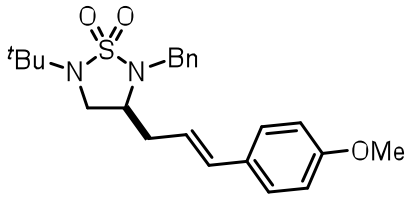
1314, 1152 cm^{-1}; MS (ESI+) 488.2366 (488.2366 calcd for $C_{29}H_{33}N_3O_2S$, M + H^+). The enantiopurity was determined to be 92:8 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 220 nm, RT= 31.9 and 74.6 min).



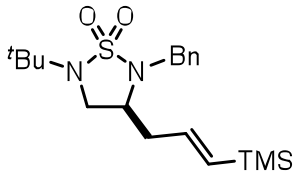
(+)-(*S*)-2-Benzyl-5-(*tert*-butyl)-3-(2-methylbenzyl)-1,2,5-thiadiazolidine-1,1-dioxide (3k). The general procedure 2 was employed for the coupling of *N*-allyl-*N*'-benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and 2-bromotoluene (72 μL, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 μL, 0.60 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (61 mg, 54%) as a colorless solid, mp 75–78 °C: $[\alpha]^{23}_D$ +19.3 (*c* 4.0, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44–7.39 (m, 2 H), 7.37–7.32 (m, 2 H), 7.30 (d, *J* = 6.8 Hz, 1 H), 7.15–7.07 (m, 3 H), 6.99 (d, *J* = 6.8 Hz, 1 H), 4.40–4.34 (m, 1 H), 4.27–4.21 (m, 1 H), 3.51–3.43 (m, 1 H), 3.20–3.14 (m, 1 H), 3.10 (dd, *J* = 8.8, 5.9 Hz, 1 H), 3.02 (dd, *J* = 13.6, 4.5 Hz, 1 H), 2.69 (dd, *J* = 13.6, 10.1 Hz, 1 H), 2.05 (s, 3 H), 1.41 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.5, 136.0, 134.9, 130.8, 130.2, 128.9, 128.8, 128.1, 127.3, 126.3, 56.4, 54.9, 50.1, 45.8, 36.1, 27.6, 19.4; IR (neat) 2977, 1290, 1148 cm^{-1}; MS (ESI+) 373.1946 (373.1944 calcd for $C_{21}H_{28}N_2O_2S$, M + H^+). The enantiopurity was determined to be 68:32 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 5% IPA/Hexanes, 1.00 mL/min, λ 225 nm, RT= 6.5 and 7.7 min).



(+)-(*S*)-2-Benzyl-5-(*tert*-butyl)-3-[2-(trifluoromethyl)benzyl]-1,2,5-thiadiazolidine-1,1-dioxide (3l). The general procedure 2 was employed for the coupling of *N*-allyl-*N*'-benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and 2-bromobenzotrifluoride (82 µL, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 µL, 0.60 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (30 mg, 23%) as a yellow oil: $[\alpha]^{23}_D$ +18.2 (*c* 4.1, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.8 Hz, 1 H), 7.48–7.42 (m, 1 H), 7.38–7.32 (m, 1 H), 7.31–7.22 (m, 6 H), 4.33 (d, *J* = 14.9 Hz, 1 H), 4.14 (d, *J* = 14.9 Hz, 1 H), 3.63–3.54 (m, 1 H), 3.28–3.19 (m, 2 H), 3.10 (dd, *J* = 8.9, 5.7 Hz, 1 H), 2.91–2.84 (m, 1 H), 1.41 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 135.8, 135.1, 132.8, 132.0, 128.9, 128.9 (q, *J* = 28.8 Hz), 128.7, 127.9, 127.3, 126.6 (q, *J* = 4.4 Hz), 124.5 (q, *J* = 273.8 Hz), 56.4, 56.0, 51.3, 46.1, 36.1, 27.6; ^{19}F NMR (471 MHz, $CDCl_3$) δ –59.4; IR (neat) 2978, 1312, 1150, 1113 cm^{-1}; MS (ESI+) 427.1664 (427.1662 calcd for $C_{21}H_{25}F_3N_2O_2S$, M + H^+). The enantiopurity was determined to be 62:38 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 230 nm, RT= 3.6 and 5.7 min).



(+)-(*S,E*)-2-Benzyl-5-(*tert*-butyl)-3-[3-(4-methoxyphenyl)allyl]-1,2,5-thiadiazolidine-1,1-dioxide (3m). The general procedure 2 was employed for the coupling of *N*-allyl-*N*'-benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and (*E*)-1-(2-bromovinyl)-4-methoxybenzene (128 mg, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 μL, 0.60 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (56 mg, 45%) as a yellow oil: $[\alpha]^{23}_{D}$ +31.6 (*c* 4.4, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 (d, *J* = 7.3 Hz, 2 H), 7.37–7.31 (m, 2 H), 7.29 (d, *J* = 7.3 Hz, 1 H), 7.22 (d, *J* = 8.6 Hz, 2 H), 6.83 (d, *J* = 8.6 Hz, 2 H), 6.31 (d, *J* = 15.9 Hz, 1 H), 5.84–5.77 (m, 1 H), 4.48 (d, *J* = 15.2 Hz, 1 H), 4.13 (d, *J* = 15.2 Hz, 1 H), 3.80 (s, 3 H), 3.42–3.36 (m, 2 H), 3.15–3.08 (m, 1 H), 2.51–2.43 (m, 1 H), 2.35–2.27 (m, 1 H), 1.41 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.4, 136.3, 133.3, 129.8, 128.8, 128.7, 128.0, 127.5, 121.5, 114.2, 56.4, 55.6, 55.5, 50.2, 45.8, 36.2, 27.5; IR (neat) 2975, 1606, 1510, 1246, 1148 cm^{-1}; MS (ESI+) 415.2050 (415.205 calcd for $C_{23}H_{30}N_2O_3S$, M + H^+). The enantiopurity was determined to be 94:6 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 13.5 and 26.3 min).

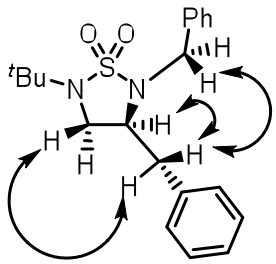


(+)-(*S,E*)-2-Benzyl-5-(*tert*-butyl)-3-[3-(trimethylsilyl)allyl]-1,2,5-thiadiazolidine-1,1-dioxide (3n). The general procedure 2 was employed for the coupling of *N*-allyl-*N*'-benzyl-*N*-*tert*-butylsulfamide (85.0 mg, 0.30 mmol) and (*E*)-(2-bromovinyl)trimethylsilane (92 µL, 0.60 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.8 mg, 0.003 mmol) and (*S*)-SIPHOS-PE (7.6 mg, 0.015 mmol), water (11 µL, 0.60 mmol), sodium *tert*-butoxide (58.0 mg, 0.60 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.4 mL of xylenes. This procedure afforded the title compound (46 mg, 40%) as a yellow oil: $[\alpha]^{23}_{D}$ +22.3 (*c* 2.1, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (d, *J* = 7.1 Hz, 2 H), 7.36–7.31 (m, 2 H), 7.28 (d, *J* = 7.3 Hz, 1 H), 5.82–5.67 (m, 2 H), 4.42 (d, *J* = 15.2 Hz, 1 H), 4.10 (d, *J* = 15.2 Hz, 1 H), 3.39–3.30 (m, 2 H), 3.08–3.04 (m, 1 H), 2.43–2.37 (m, 1 H), 2.28–2.21 (m, 1 H), 1.42 (s, 9 H), 0.02 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.1, 136.3, 135.9, 128.7, 128.7, 127.9, 56.4, 55.0, 50.0, 45.6, 39.6, 27.5, –1.2; IR (neat) 2954, 1617, 1293, 1247, 1149 cm^{-1}; MS (ESI+) 381.2029 (381.2027 calcd for $C_{19}H_{32}N_2O_2SSi$, M + H^+). The enantiopurity was determined to be 95:5 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 220 nm, RT= 2.9 and 7.5 min).

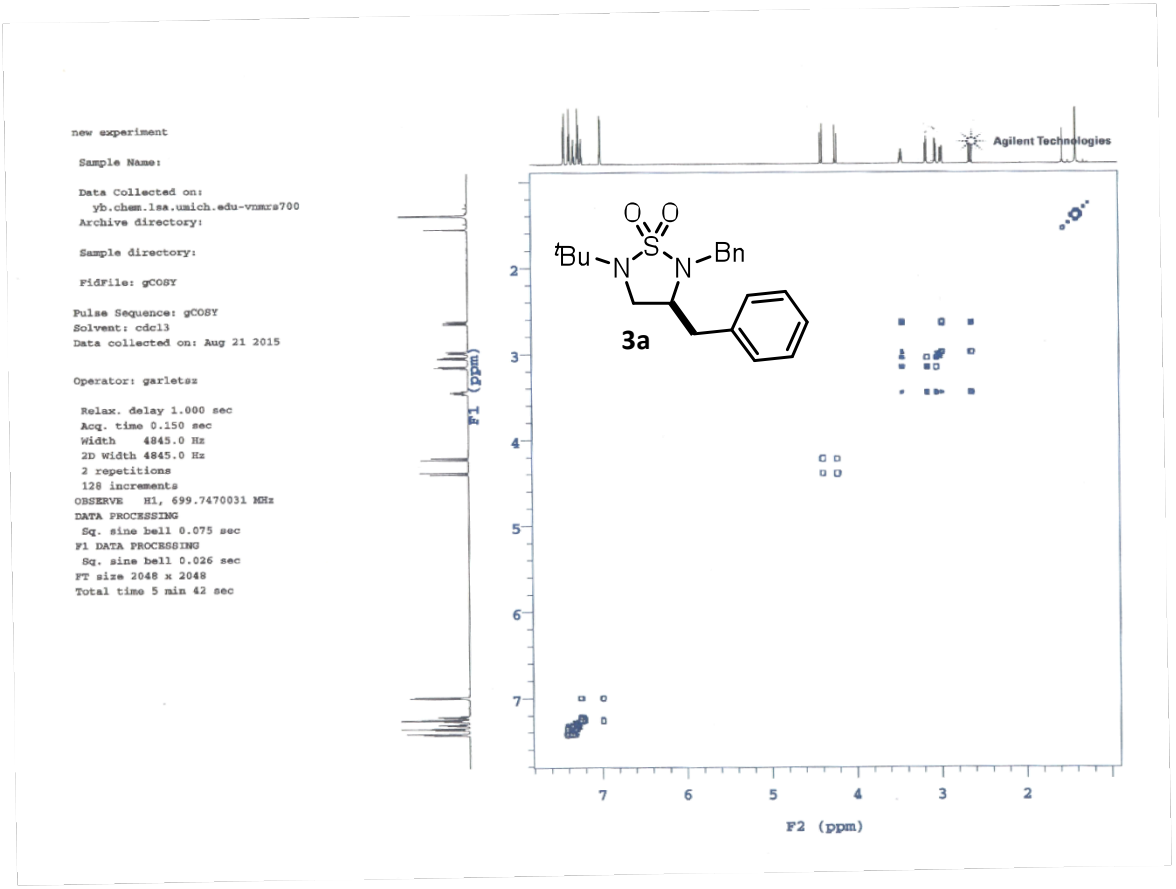
Deuterium Labeling Studies

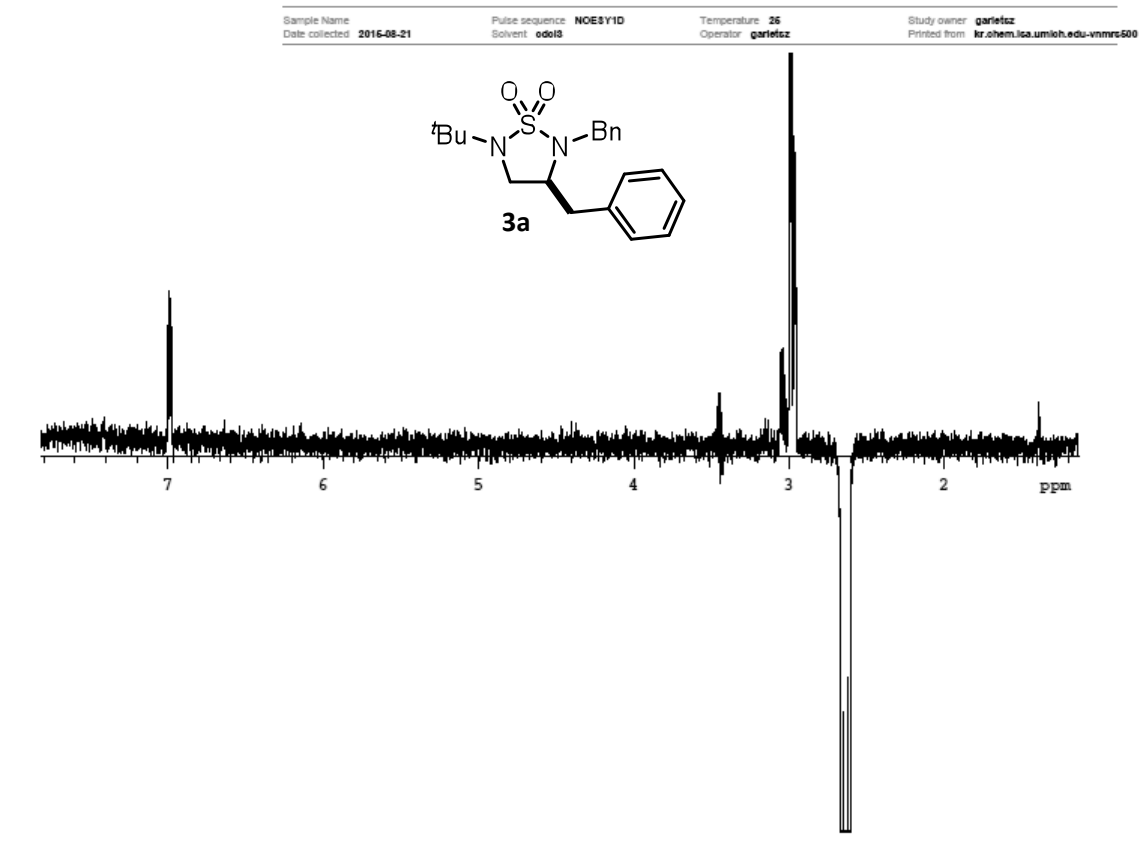
The configuration of deuterated products **9a-d** were assigned on the basis of 1D NOESY experiments carried out with the all-proteo analogs of these compounds. The

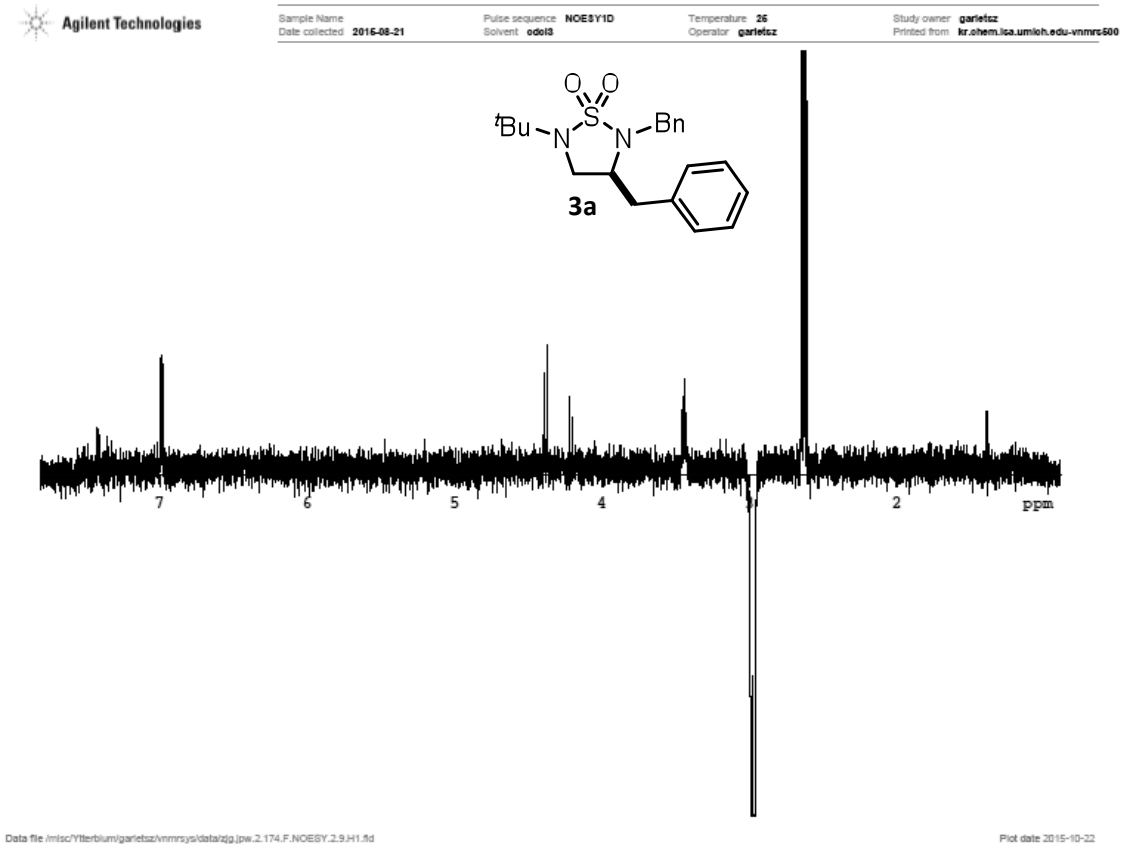
key nOe signals are shown below.



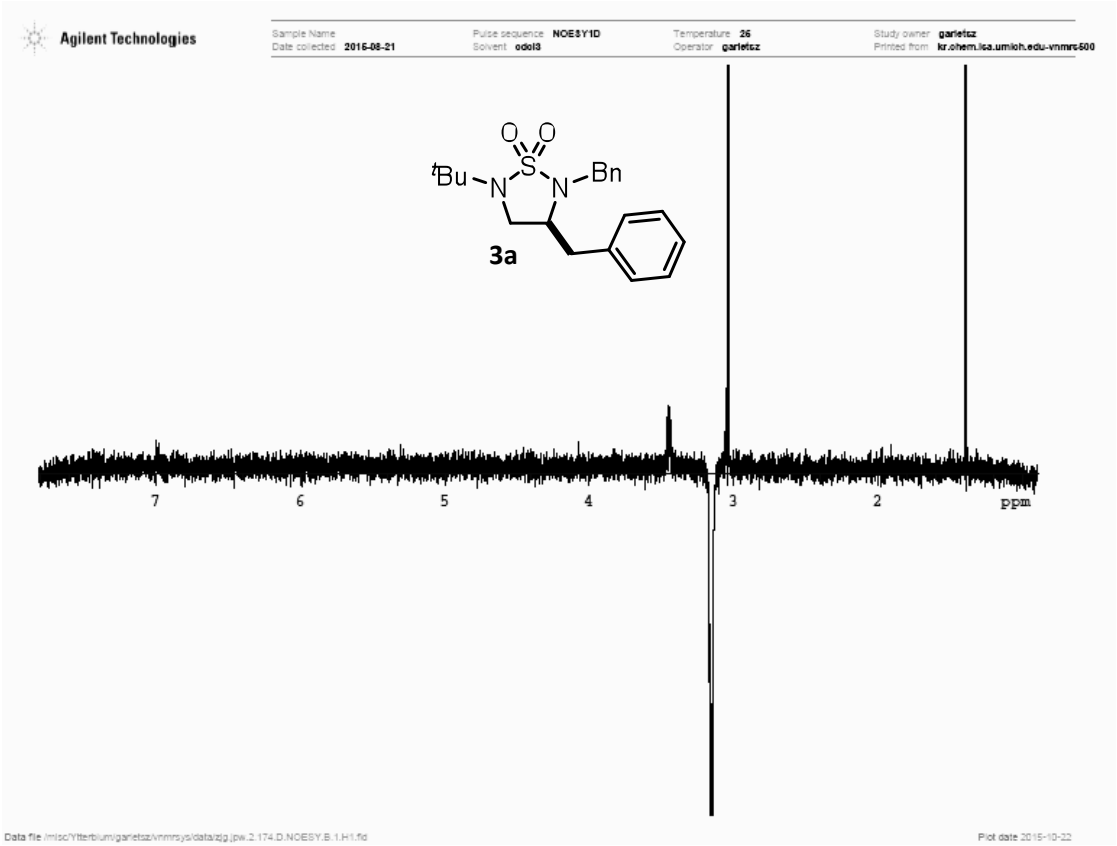
The configuration of the deuterated products was then assigned by examining which signal was absent from the ^{1}H NMR.

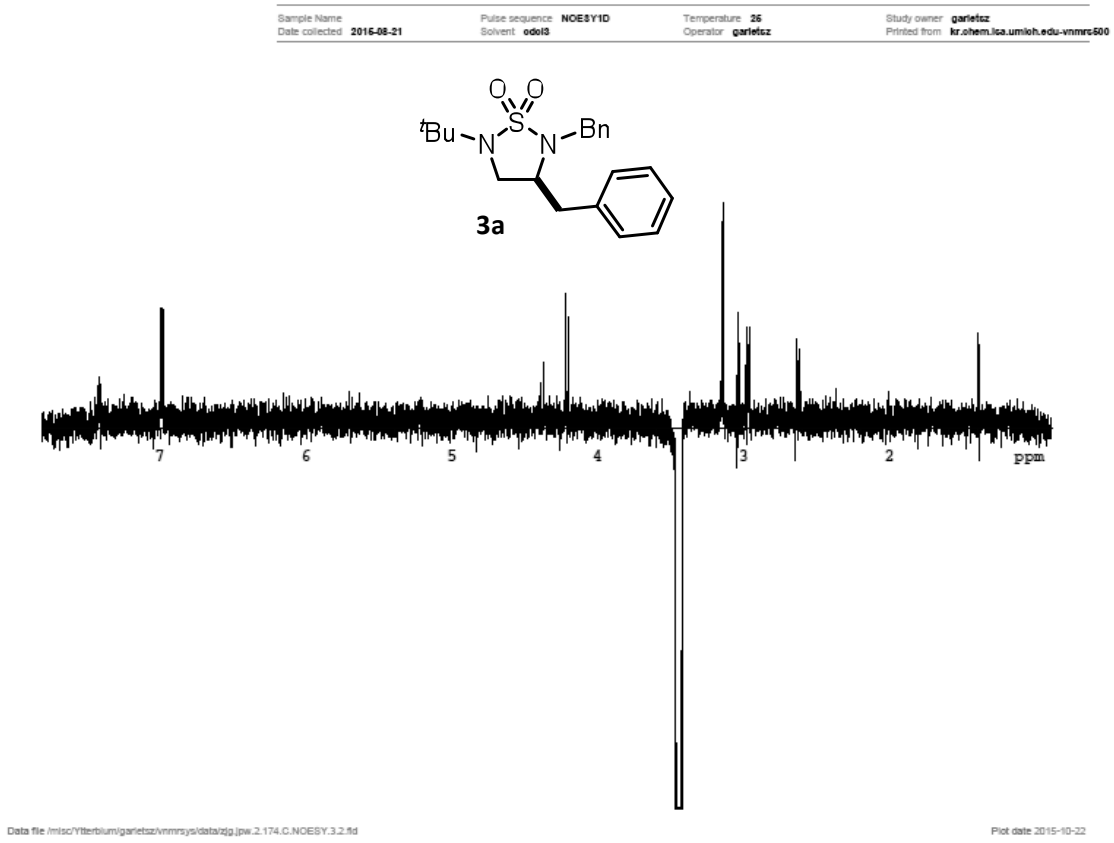



Sample Name
Date collected 2016-08-21
Pulse sequence NOESY1D
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs500
tBu
N
S
O
O
N
Bn
3a
7
6
5
4
3
2
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.2.174.NOESY.2.6.fid
Plot date 2015-10-22

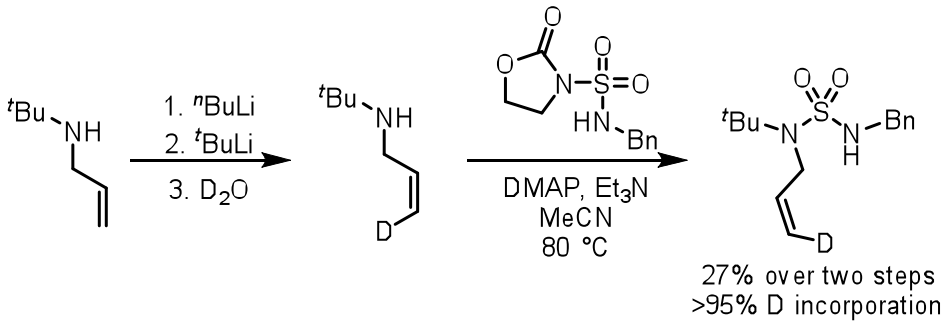

Agilent Technologies
Sample Name
Date collected 2016-08-21
Pulse sequence NOESY1D
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs500
tBu
N
S
O
O
N
Bn
3a
7
6
5
4
3
2
ppm
Data file /misc/Ytterbium/garletsz/vnmrsys/data/zjg.jpw.2.174.F.NOESY.2.9.H1.fid
Plot date 2015-10-22

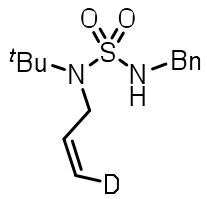

Agilent Technologies
Sample Name
Date collected 2016-08-21
Pulse sequence NOESY1D
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.isa.umich.edu-vnmrs500
tBu
N
S
O
O
N
Bn
3a
7
6
5
4
3
2
ppm
Data file /misc/Ytterbium/garletsz/vnmrsys/data/zjg.jpw.2.174.E.NOESY.C.3.0.H1.fid
Plot date 2015-10-22


Agilent Technologies
Sample Name
Date collected 2016-08-21
Pulse sequence NOESY1D
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from kr.ohem.isa.umich.edu-vnmrs500
tBu
N
S
O
O
N
Bn
3a
7
6
5
4
3
2
ppm
Data file /misc/Ytterbium/garletsz/vnmrsys/data/zjg.jpw.2.174.D.NOESY.B.1.H1.fid
Plot date 2015-10-22



Reaction Sequence for Deuterium Labeled Substrate



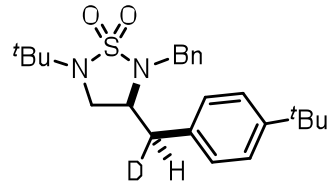


(*Z*)-1-(3-*d*-Allyl)-1-benzyl-3-*tert*-butylsulfamide (8). The procedure previously described

by our group for the preparation of (*Z*)-1-(3-*d*-Allyl)-1,3-dibenzylsulfamide was used for the synthesis of **8**.[2,3] A flame dried round bottom flask equipped with a stir bar was cooled to rt under a stream of nitrogen and charged with *N*-(*tert*-butyl)prop-2-en-1-amine (10.0 mmol, 1.13 g) and Et_2O (20 mL). The resulting solution was cooled to –42 °C using a CO_2/CH_3CN bath and stirred for 5 min. A solution of *n*-BuLi in hexanes (4.8 mL, 2.5 M, 12 mmol) was added slowly, and the resulting mixture was stirred at –42 °C for 20 min. A solution of *t*-BuLi in pentanes (13.0 mL, 1.7 M, 22 mmol) was added slowly and the resulting solution was stirred at –42 °C for 30 min. The CO_2/CH_3CN bath was replaced with a brine/ice bath, and the reaction mixture was allowed to slowly warm to rt as the ice melted. The bath was removed and the mixture was stirred at rt for 1 h. The reaction mixture was then cooled to –78 °C, and D_2O (3.6 mL, 200 mmol) was added dropwise. The resulting mixture was warmed to rt and stirred overnight. The reaction mixture was cooled to 0 °C, quenched with H_2O (15 mL) and transferred to a separatory funnel. The mixture was extracted with Et_2O (3 x 10 mL). The combined organic layers were dried with anhydrous Na_2SO_4, filtered, and concentrated *in vacuo* to yield crude (*Z*)-*N*-(*tert*-butyl)prop-2-en-3-d-1-amine.

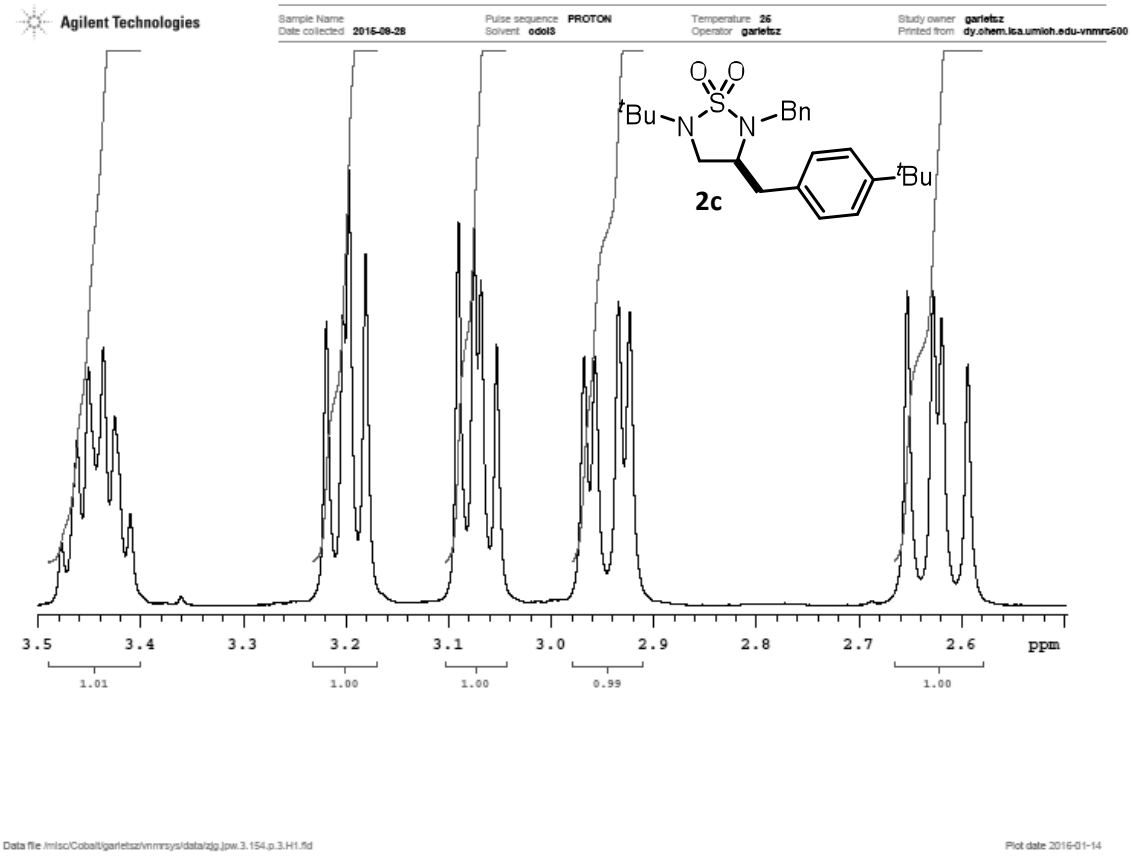
A flame dried two necked flask equipped with stir bar and condenser was then charged with *N*-benzyl-2-oxooxazolidine-3-sulfonamide (2.56 g, 10 mmol), 4-dimethylaminopyridine (244 mg, 2 mmol), acetonitrile (50mL) and Et_3N (4.2 mL, 30 mmol). The reaction vessel was placed in an oil bath at 80 ºC and the mixture was stirred for 15 min. Neat (*Z*)-*N*-(*tert*-butyl)prop-2-en-3-d-1-amine was added, and the resulting mixture was stirred at 80 °C for ca. 6 h. The mixture was cooled to rt, and the solvent was removed under reduced pressure. The resulting crude product was purified

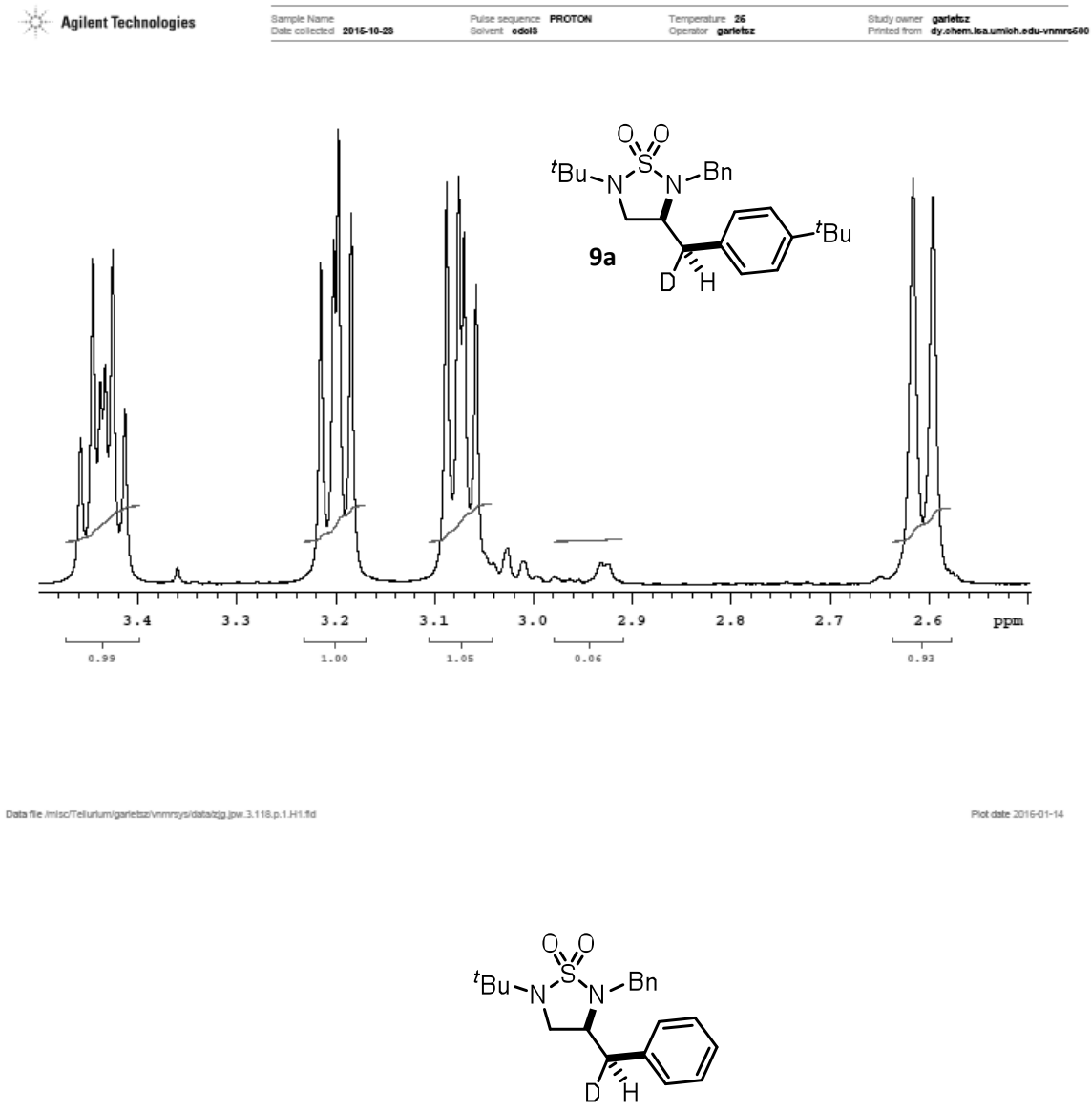
by flash chromatography on silica gel using hexanes/ethyl acetate as eluant to yield 770 mg (27%) of a yellow solid, mp 73–75 °C: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44–7.28 (m, 5 H), 6.01–5.91 (m, 1 H), 5.12 (d, *J* = 10.3 Hz, 1 H), 4.27–4.19 (m, 1 H), 4.15 (d, *J* = 6.1 Hz, 2 H), 3.96 (dd, *J* = 6.0, 1.1 Hz, 2 H), 1.47 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 137.4, 136.9, 128.9, 128.3, 128.1, 116.5 (t, *J* = 23.9 Hz), 59.4, 49.5, 47.5, 29.9; IR (neat) 3328, 2970, 1317, 1137 cm^{-1}; MS (ESI+) 306.1358 (306.1357 calcd for $C_{14}H_{21}DN_2O_2S$, M + Na^+).



(–)-(1'*R*,3*S*)-2-Benzyl-5-(*tert*-butyl)-3-{[4-(*tert*-butyl)phenyl]methyl-d}-1,2,5-thiadiazolidine-1,1-dioxide (9a). The general procedure 2 was employed for the coupling of (*Z*)-1-(3-*d*-allyl)-1-benzyl-3-*tert*-butylsulfamide (71.0 mg, 0.25 mmol) and 1-bromo-4-*tert*-butylbenzene (87 μL, 0.50 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.3 mg, 0.0025 mmol) and (*S*)-SIPHOS-PE (6.3 mg, 0.0125 mmol), sodium *tert*-butoxide (48.0 mg, 0.50 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.0 mL of xylenes. This procedure afforded the title compound (60 mg, 58%) as a colorless solid, mp 86–90 °C: $[\alpha]^{23}_D$ –2.4 (*c* 5.4, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43–7.40 (m, 2 H), 7.36 (t, *J* = 7.3 Hz, 2 H), 7.33–7.25 (m, 3 H), 6.92 (d, *J* = 8.1 Hz, 2 H), 4.39 (d, *J* = 14.9 Hz, 1 H), 4.22 (d, *J* = 14.9 Hz, 1 H), 3.44 (d, *J* = 10.0 Hz, 1 H), 3.20 (dd, *J* = 8.8, 6.6 Hz, 1 H), 3.08 (dd, *J* = 8.8, 6.1 Hz, 1 H), 2.98–2.91 (m, 0.06 H), 2.61 (d, *J* = 10.0 Hz, 1 H), 1.41 (s, 9 H), 1.30 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.0,

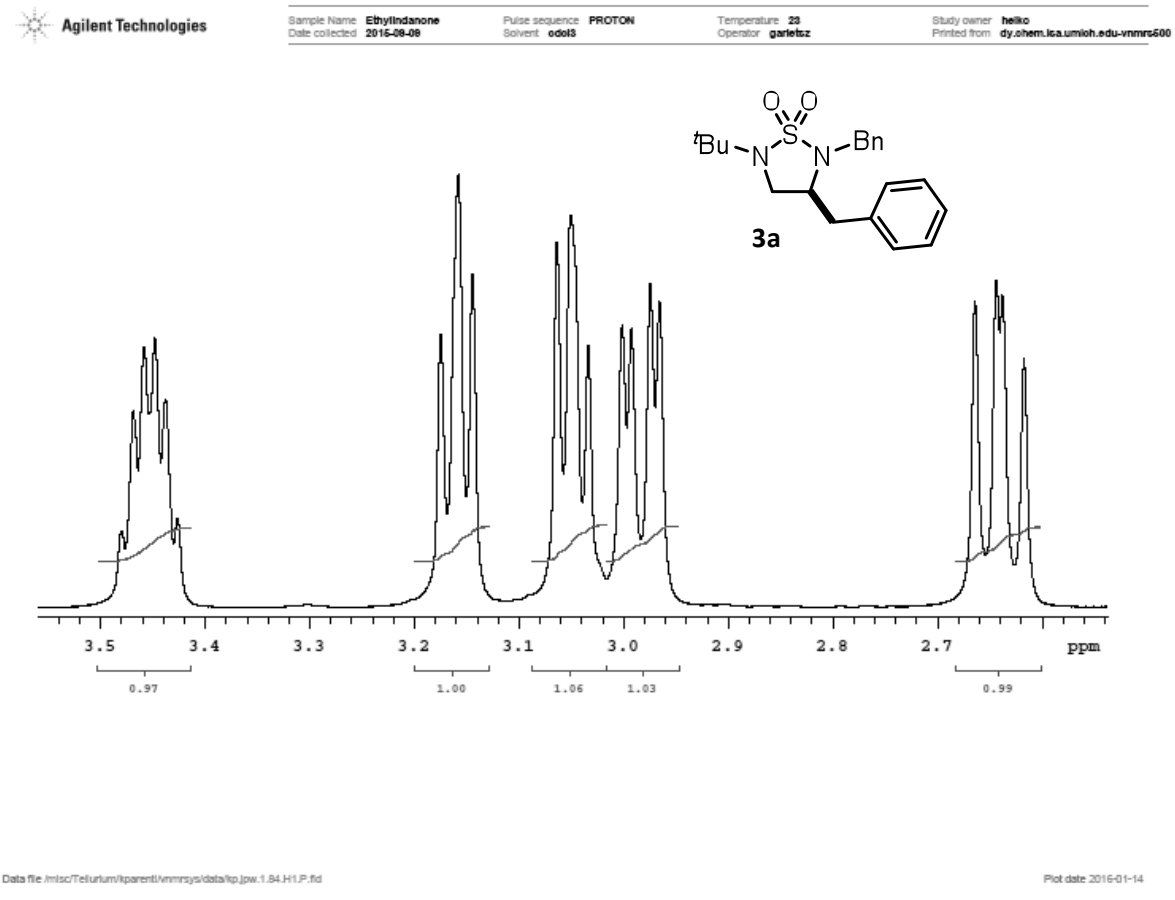
136.0, 133.5, 129.0, 128.9, 128.7, 128.0, 125.8, 56.5, 56.3, 50.0, 45.8, 37.7 (t, *J* = 20.0 Hz), 34.6, 31.5, 27.6; IR (neat) 2971, 1325, 1151 cm^{-1}; MS (ESI+) 416.2478 (416.2477 calcd for $C_{24}H_{33}DN_2O_2S$, M + H^+). The diastereoselectivity was determined to be 17:1 by comparing the products obtained from separate reactions of the deuterated and non-deuterated substrates. The enantiopurity was determined to be 94:6 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 205 nm, RT= 4.1 and 5.0 min).

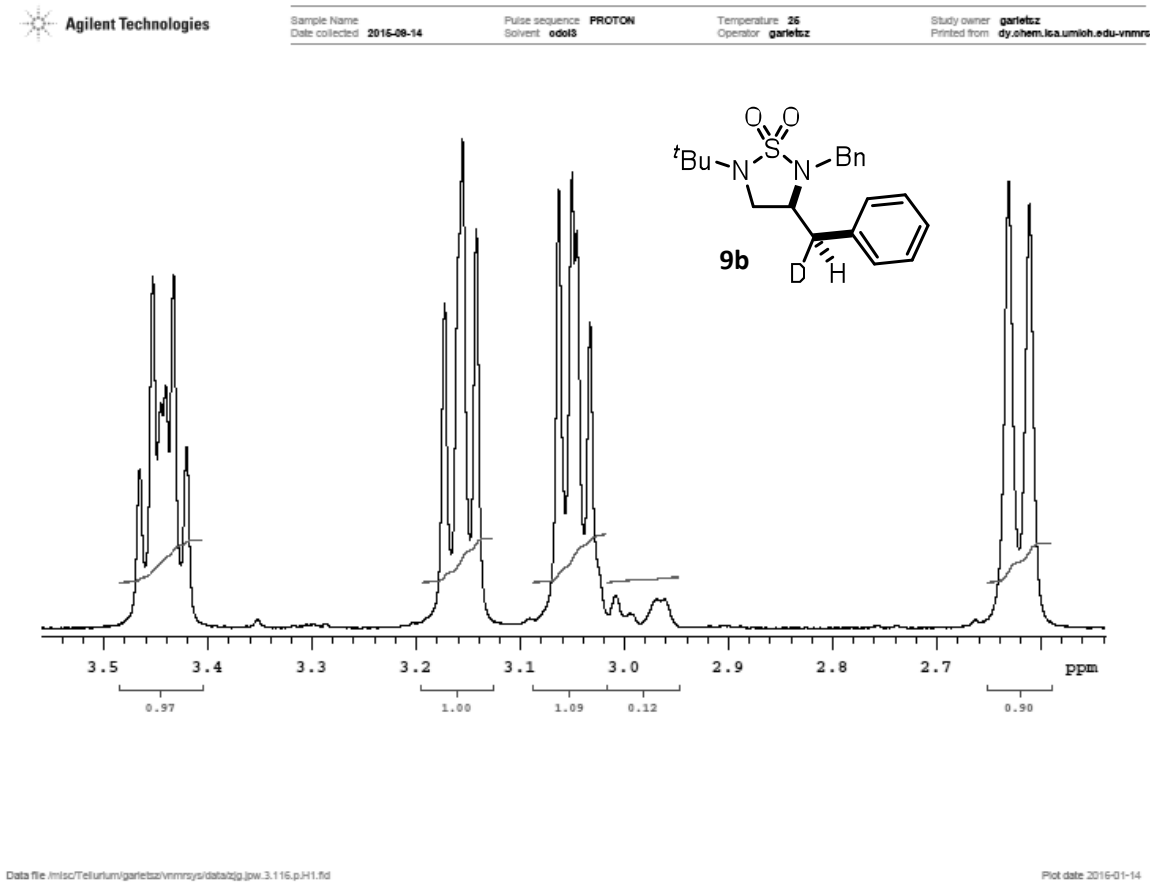


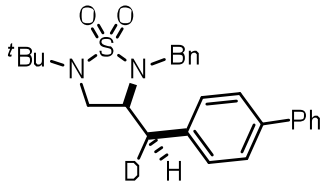


(–)-(1'*R*,3*S*)-2-Benzyl-5-(*tert*-butyl)-3-(-phenylmethyl-d)-1,2,5-thiadiazolidine-1,1-dioxide (9b). The general procedure 2 was employed for the coupling of (*Z*)-1-(3-*d*-allyl)-1-benzyl-3-*tert*-butylsulfamide (71.0 mg, 0.25 mmol) and bromobenzene (53 μL, 0.50 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.3 mg, 0.0025 mmol) and (*S*)-SIPHOS-PE (6.3 mg, 0.0125 mmol), sodium *tert*-butoxide (48.0 mg, 0.50 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.0 mL of xylenes. This procedure afforded the title compound (60 mg, 67%) as a colorless solid, mp 108–111

°C: $[\alpha]^{23}_D$ –10.0 (*c* 4.7, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45–7.40 (m, 2 H), 7.36 (t, *J* = 7.5 Hz, 2 H), 7.33–7.19 (m, 4 H), 7.00 (d, *J* = 7.8 Hz, 2 H), 4.39 (d, *J* = 14.9 Hz, 1 H), 4.21 (d, *J* = 14.9 Hz, 1 H), 3.48–3.41 (m, 1 H), 3.16 (dd, *J* = 8.4, 6.7 Hz, 1 H), 3.05 (dd, *J* = 8.7, 6.5 Hz, 1 H), 3.02–2.95 (m, 0.12 H), 2.62 (d, *J* = 10.0 Hz, 1 H), 1.40 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.6, 136.0, 129.2, 129.0, 128.9, 128.8, 128.1, 127.1, 56.6, 56.4, 50.1, 45.7, 38.4 (t, *J* = 21.0 Hz), 27.6; IR (neat) 2976, 1325, 1153 cm^{-1}; MS (ESI+) 360.1852 (360.1851 calcd for $C_{20}H_{25}DN_2O_2S$, M + Na^+). The diastereoselectivity was determined to be 8:1 by comparing the products obtained from separate reactions of the deuterated and non-deuterated substrates. The enantiopurity was determined to be 95:5 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 4% IPA/Hexanes, 0.500 mL/min, λ 210 nm, RT= 18.7 and 20.6 min).

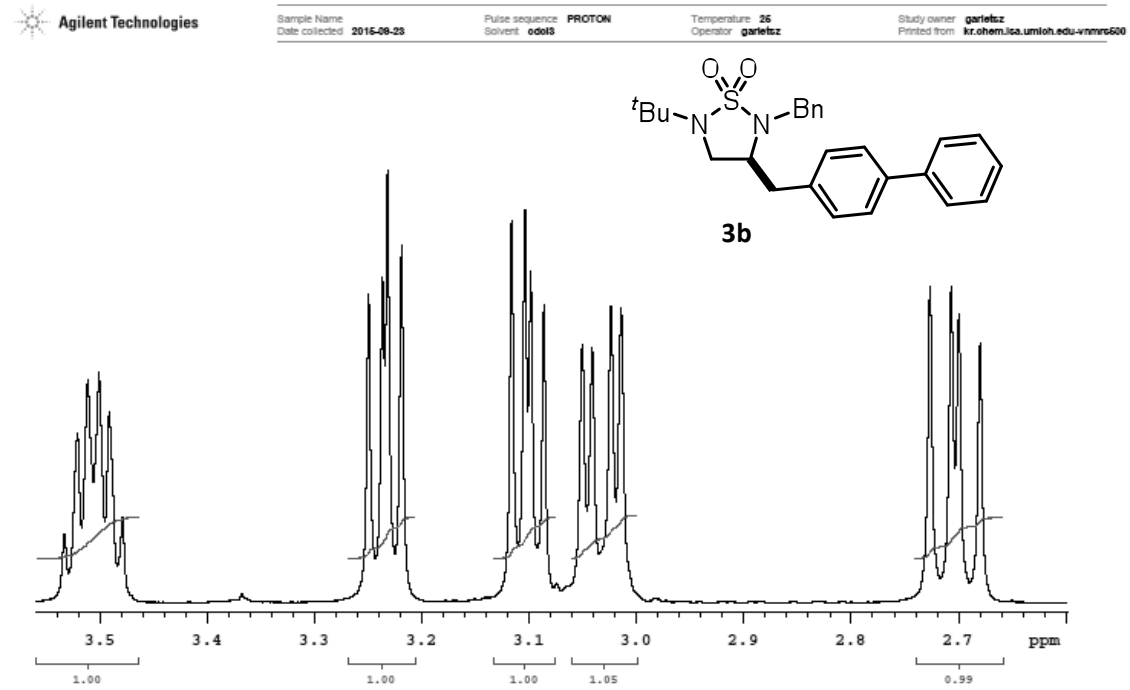


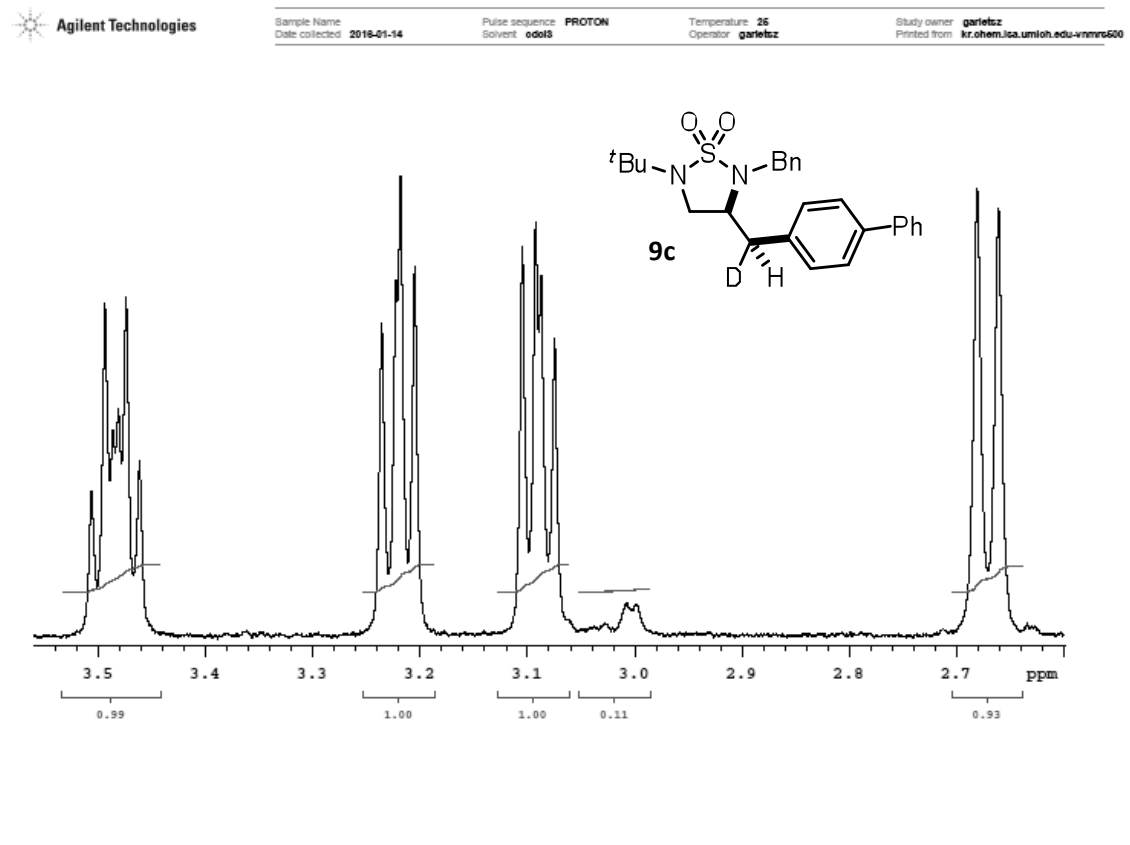


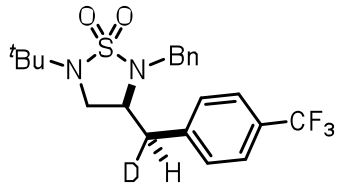


(+)-(1'*R*,3*S*)-3-[(1,1'-Biphenyl)-4-ylmethyl-d]-2-benzyl-5-(*tert*-butyl)-1,2,5-thiadiazolidine 1,1-dioxide (9c). The general procedure 2 was employed for the coupling of (*Z*)-1-(3-*d*-allyl)-1-benzyl-3-*tert*-butylsulfamide (71.0 mg, 0.25 mmol) and 4-bromobiphenyl (117 mg, 0.50 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.3 mg, 0.0025 mmol) and (*S*)-SIPHOS-PE (6.3 mg, 0.0125 mmol), sodium *tert*-butoxide (48.0 mg, 0.50 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.0 mL of xylenes. This procedure afforded the title compound (71 mg, 65%) as a yellow solid,

mp 116–121 °C: $[\alpha]^{23}_{D}$ +0.2 (*c* 5.7, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58–7.53 (m, 2 H), 7.51–7.47 (m, 2 H), 7.46–7.40 (m, 4 H), 7.40–7.29 (m, 4 H), 7.09–7.04 (m, 2 H), 4.42 (d, *J* = 14.9 Hz, 1 H), 4.23 (d, *J* = 14.9 Hz, 1 H), 3.52–3.45 (m, 1 H), 3.23 (dd, *J* = 8.8, 6.6 Hz, 1 H), 3.12–3.07 (m, 1 H), 3.06–2.99 (m, 0.11 H), 2.67 (d, *J* = 9.8 Hz, 1 H), 1.42 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.7, 140.0, 136.0, 135.6, 129.6, 129.0, 128.8, 128.1, 127.6, 127.5, 127.1, 56.6, 56.4, 50.3, 45.8, 38.0 (t, *J* = 20.0 Hz), 27.6, 1 carbon signal is missing due to incidental equivalence; IR (neat) 2974, 1488, 1320, 1151 cm^{-1}; MS (ESI+) 436.2162 (436.2164 calcd for $C_{26}H_{29}DN_2O_2S$, M + H^+). The diastereoselectivity was determined to be 9:1 by comparing the products obtained from separate reactions of the deuterated and non-deuterated substrates. The enantiopurity was determined to be 92:8 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 254 nm, RT= 14.6 and 24.0 min).

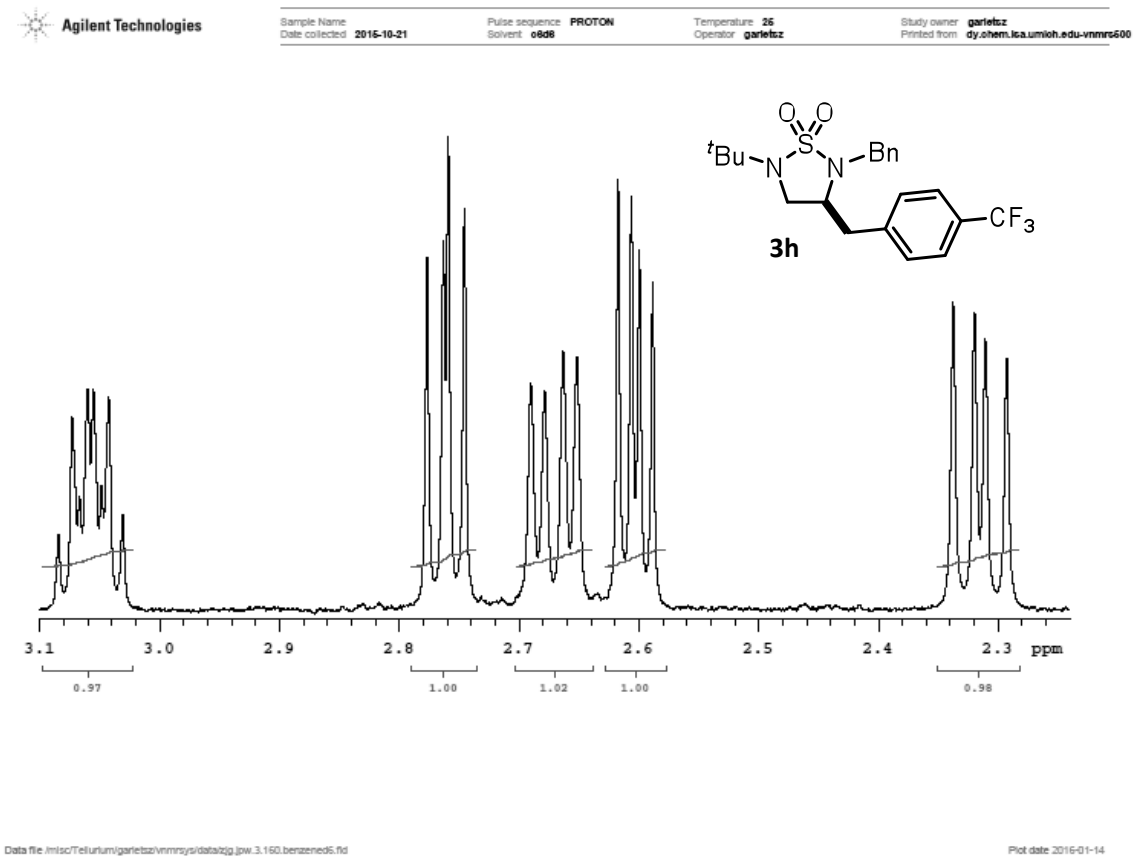

Agilent Technologies
Sample Name
Date collected 2015-08-23
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs500
tBu
N
S
O
O
N
Bn
3b
3.5
3.4
3.3
3.2
3.1
3.0
2.9
2.8
2.7
ppm
1.00
1.00
1.00
1.05
0.99
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.150.p.1.H1.fid
Plot date 2016-01-14

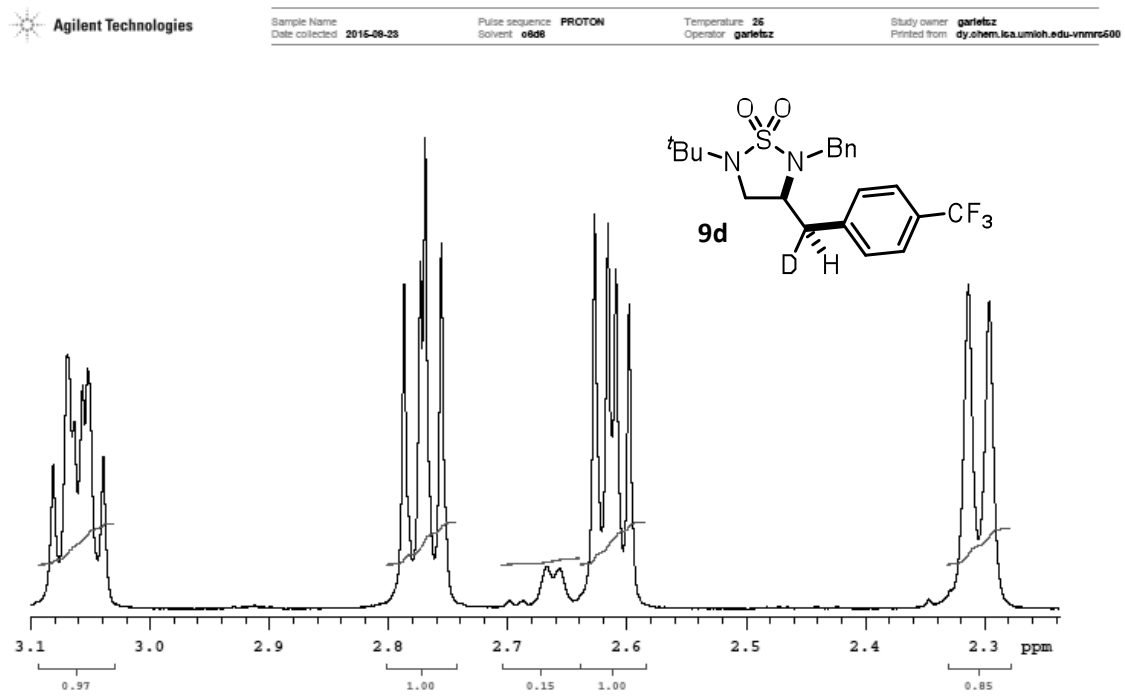

Agilent Technologies
Sample Name
Date collected 2016-01-14
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs500
tBu
N
S
O
O
N
Bn
Ph
9c
D
H
3.5
3.4
3.3
3.2
3.1
3.0
2.9
2.8
2.7
ppm
0.99
1.00
1.00
0.11
0.93
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.117.p.g.H1.fid
Plot date 2016-01-14

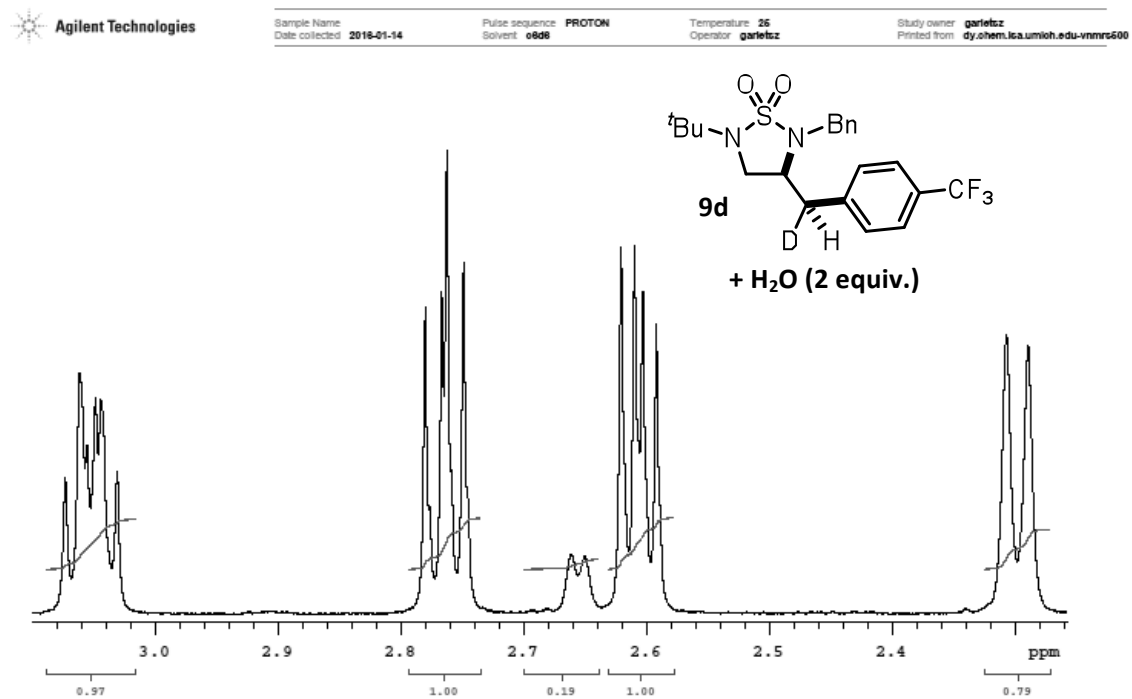


(–)-(1'*R*,3*S*)-2-Benzyl-5-(*tert*-butyl)-3-{[4-(trifluoromethyl)phenyl]methyl-d}-1,2,5-thiadiazolidine-1,1-dioxide (9d). The general procedure 2 was employed for the coupling of (*Z*)-1-(3-*d*-allyl)-1-benzyl-3-*tert*-butylsulfamide (71.0 mg, 0.25 mmol) and 4-bromobenzotrifluoride (70 µL, 0.50 mmol) using a catalyst composed of $Pd_2(dba)_3$ (2.3 mg, 0.0025 mmol) and (*S*)-SIPHOS-PE (6.3 mg, 0.0125 mmol), sodium *tert*-butoxide (48.0 mg, 0.50 mmol), a reaction temperature of 120 °C, and a reaction time of 18 h in 2.0 mL of xylenes. This procedure afforded the title compound (60.0 mg, 56%) as a colorless solid, mp 128–132 °C: $[\alpha]^{23}_{D}$ –16.6 (*c* 5.6, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.1 Hz, 2 H), 7.39–7.28 (m, 4 H), 7.09 (d, *J* = 7.8 Hz, 2 H), 4.39 (d, *J* = 14.7 Hz, 1 H), 4.16 (d, *J* = 14.9 Hz, 1 H), 3.46 (d, *J* = 9.0 Hz, 1 H), 3.23 (dd, *J* = 8.8, 6.6 Hz, 1 H), 3.03 (dd, *J* = 8.9, 5.7 Hz, 1 H), 2.70 (d, *J* = 9.3 Hz, 1 H), 1.40 (s, 9 H); ^{1}H NMR (500 MHz, C_6D_6) δ 7.13–7.21 (m, 4 H), 6.99–7.09 (m, 3 H), 6.57 (d, *J* = 8.1 Hz, 2 H), 4.28 (d, *J* = 14.9 Hz, 1 H), 3.91–3.97(d, *J* = 14.9 Hz, 1 H), 3.02–3.10 (m, 1 H), 2.76 (dd, *J* = 8.8, 6.8 Hz, 1 H), 2.64–2.70 (m, 0.15 H), 2.61 (dd, *J* = 8.8, 5.6 Hz, 1 H), 2.30 (d, *J* = 8.8 Hz, 1 H), 1.24 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.8, 135.7, 129.6, 129.4, 129.0, 128.8, 128.2, 125.8 (q, *J* = 3.90 Hz), 124.2 (q, *J* = 272.8 Hz), 56.5, 56.2, 50.8, 45.8, 38.4 (t, *J* = 19.1 Hz), 27.6; IR (neat) 2972, 1322, 1124 cm^{-1}; MS (ESI+) 428.1723 (428.1724 calcd for $C_{21}H_{24}DF_3N_2O_2S$, M + H^+). The diastereoselectivity was determined to be 7:1 by comparing the products obtained from separate reactions of the deuterated and non-deuterated substrates. The enantiopurity was determined to be 88:12 er by

chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.00 mL/min, λ 210 nm, RT= 6.0 and 8.3 min). When the reaction was conducted in the presence of water (2 equivalents), the desired product **9d** was obtained with 78% yield 5:1 dr and 89:11 er.

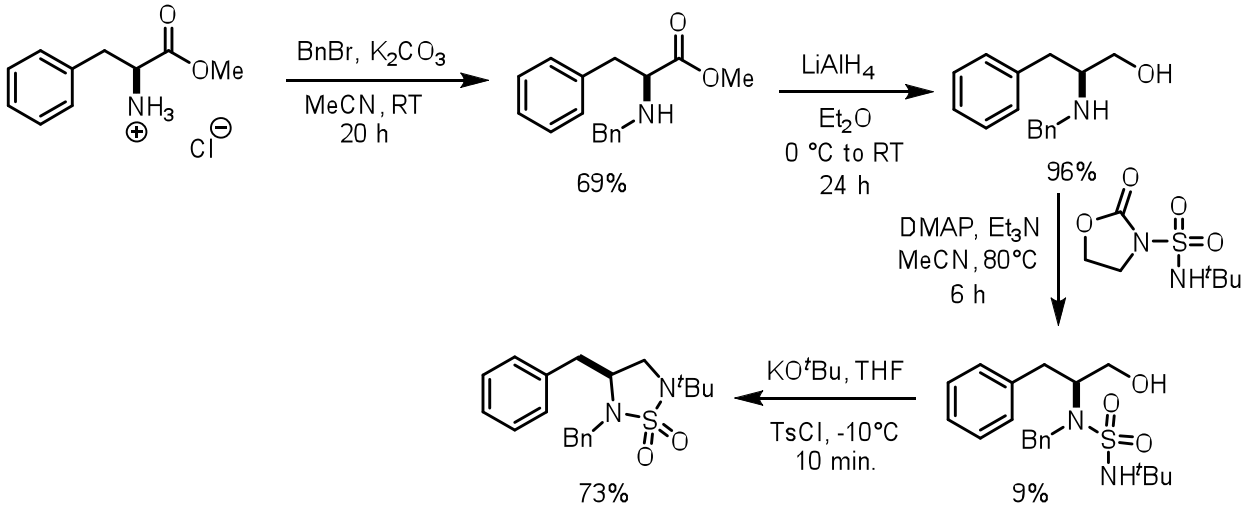



Agilent Technologies
Sample Name
Date collected 2016-08-23
Pulse sequence PROTON
Solvent o6d8
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs600
9d
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
ppm
0.97
1.00
0.15
1.00
0.85
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.143.p.benzened6.H1.fid
Plot date 2016-01-14

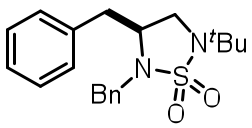

Agilent Technologies
Sample Name
Date collected 2018-01-14
Pulse sequence PROTON
Solvent o6d8
Temperature 26
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs600
9d
+ H2O (2 equiv.)
3.0
2.9
2.8
2.7
2.6
2.5
2.4
ppm
0.97
1.00
0.19
1.00
0.79
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.4.68.p.2.H1.fid
Plot date 2016-01-14

Determination of absolute configuration:

The absolute configuration of the cyclic sulfamide products were assigned by preparing an authentic sample of **3a** from L-phenylalanine methyl ester hydrochloride as described below. The optical rotation of **S1** was of the same sign **(–)** as that of the separate authentic sample prepared from L-phenylalanine methyl ester hydrochloride.



Reaction Sequence for Authentic Sample



(–)-(*S*)-2,3-Dibenzyl-5-(*tert*-butyl)-1,2,5-thiadiazolidine 1,1-dioxide (S1): A 250 mL round bottomed flask equipped with stir bar was flame dried, back filled with nitrogen, and then charged with methyl benzyl-L-phenylalaninate[4] (8.4 mmol, 2.3 g). Anhydrous ether (25 mL) was added, and the reaction mixture was cooled to 0 °C. Lithium aluminum hydride (25 mL of a 1 M solution in ether) was added dropwise then the reaction was warmed to rt and stirred at rt for 24 h. The mixture was then cooled to 0 °C, water (1 mL) was added dropwise followed by sodium hydroxide (1.3 mL of 3 M solution), and additional water (3 mL). The resulting mixture was filtered through celite

and concentrated to afford (*S*)-2-(benzylamino)-3-phenylpropan-1-ol as a white solid that was carried onto the next step.

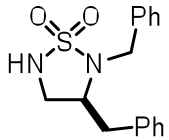
The sulfonylation was performed using a modification of the procedure described by Wolfe and co-workers.[2] A 100 mL 2-necked round bottom flask equipped with a stir bar, condenser and septum was flame dried and charged with N-(*tert*-butyl)-2-oxooxazolidine-3-sulfonamide (890 mg, 4 mmol) and 4-dimethylaminopyridine (100 mg, 0.8 mmol). Anhydrous acetonitrile (20 mL) and triethylamine (1.7 mL, 12 mmol) were added and the resulting mixture was heated to 80 °C with stirring for 15 min. A solution of (*S*)-2-(benzylamino)-3-phenylpropan-1-ol (1.0 g, 4 mmol) in anhydrous acetonitrile (5 mL) was added dropwise, and the mixture was stirred at 80 °C for 6 h. The mixture was then cooled to rt, the solvent was removed under reduced pressure, and the crude product was purified by flash chromatography on silica gel to afford 130 mg (9%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 6.8 Hz, 2 H), 7.35–7.15 (m, 6 H), 7.06 (d, *J* = 6.8 Hz, 2 H), 4.41 (s, 2 H), 4.19–4.10 (m, 1 H), 3.99 (s, 1 H), 3.73–3.65 (m, 1 H), 3.53 (d, *J* = 4.1 Hz, 1 H), 2.83 (d, *J* = 6.5 Hz, 1 H), 2.62 (dd, *J* = 14.0, 8.5 Hz, 1 H), 1.88 (t, *J* = 5.5 Hz, 1 H), 1.27 (s, 9 H).

A 10 mL flame-dried round bottom flask equipped with a stir bar was cooled under a stream of nitrogen and charged with the sulfamide prepared above (130.0 mg, 0.35 mmol) and THF (3 mL). The solution was cooled to -10 °C in a brine/ice bath then potassium *tert*-butoxide (80.0 mg, 0.70 mmol) was added in one portion, and the resulting mixture was stirred at –10 °C for 5 min. A solution of tosyl chloride (73.0 mg, 0.39 mmol) THF (1.3 mL) was then added dropwise and the mixture was stirred at –10 °C for 10 min at which time TLC analysis indicated the starting material had been

completely consumed. The mixture was warmed to rt, quenched with 5 mL water, and extracted with ether (2 x 5 mL). The organic layers were combined, filtered through a plug of celite, and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel using hexanes/ethyl acetate as eluant. This procedure afforded the title compound (91.0 mg, 73 %) as a colorless solid. $[\alpha]^{23}_{D}$ –10.7 (*c* 8.4, $CHCl_3$); The spectroscopic properties of this compound were identical to that of compound **3a**. The enantiopurity was determined to be >99:1 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 4.0% IPA/Hexanes, 0.5 mL/min, λ 210 nm, RT= 18.4 and 20.7 min).

Deprotection Methods:

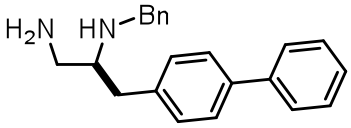
Cleavage of *N-tert*-butyl group



(–)-(*S*)-2,3-Dibenzyl-1,2,5-thiadiazolidine-1,1-dioxide (6). A round bottom flask equipped with a stirbar and a septum was charged with **3a** (55.2 mg, 0.15 mmol), hexanes (1.0 mL) and trifluoroacetic acid (0.75 mL). The resulting mixture was stirred at rt for 24 h, and then the solvent was removed under reduced pressure. The crude material was purified by flash chromatography on silica gel to afford the title compound (44.0 mg, 98%) as a colorless solid, mp 69–73 °C: $[\alpha]^{23}_{D}$ –9.1 (*c* 3.9, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44–7.21 (m, 8 H), 7.07–7.02 (m, 2 H), 4.35–4.25 (m, 2 H), 4.22 (t, *J* = 7.5 Hz, 1 H), 3.62 (ddt, *J* = 8.8, 6.7, 4.6 Hz, 1 H), 3.35 (ddd, *J* = 11.9, 7.9, 6.6 Hz, 1 H), 3.19 (ddd, *J* = 11.7, 7.2, 4.3 Hz, 1 H), 2.87 (dd, *J* = 13.6, 5.0 Hz, 1 H), 2.62 (dd, *J* =

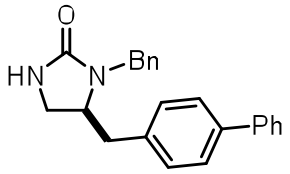
13.6, 8.7 Hz, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.1, 135.7, 129.4, 129.1, 129.0, 128.9, 128.4, 127.3, 61.6, 49.8, 45.1, 39.3; IR (neat) 3302, 2921, 1287, 1145 cm^{-1}; MS (ESI+) 303.1161 (303.1162 calcd for $C_{16}H_{18}N_2O_2S$, M + H^+). The enantiopurity was determined to be 6:94 er by chiral HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 0.5 mL/min, λ 210 nm, RT= 51.5 and 54.9 min).

Cleavage of *N-tert*-butyl group and sulfonyl group



(+)-(*S*)-3-([1,1'-Biphenyl]-4-yl)-*N*^{2}-benzylpropane-1,2-diamine (7). A 10 mL round bottom flask equipped with a reflux condenser and a stir bar was charged with (*S*)-3-([1,1'-biphenyl]-4-ylmethyl)-2-benzyl-5-(*tert*-butyl)-1,2,5-thiadiazolidine-1,1-dioxide (74.1 mg, 0.17 mmol) and phenol (50.0 mg, 0.53 mmol). Aqueous 2 M hydrobromic acid (2 mL, 4 mmol) was added, the reaction mixture was heated at 130 °C for 24 h, then was cooled to rt. The mixture was diluted with water (5 mL) and ether (5 mL) then solid sodium hydroxide (~2.3 g) was added until the mixture reached pH ≥ 10. The layers were separated, and the aqueous layer was extracted with ether (3 x 5 mL). The combined organic layers were dried with sodium sulfate, and then passed through a plug of celite. The solvent was removed under reduced pressure to afford the title compound (46 mg, 85%) as a yellow oil: $[\alpha]^{23}_D$ +1.6 (*c* 4.4, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61–7.55 (m, 2 H), 7.52 (d, *J* = 8.2 Hz, 2 H), 7.43 (t, *J* = 7.6 Hz, 2 H), 7.36–7.19 (m, 8 H), 3.86–3.77 (m, 2 H), 2.90–2.79 (m, 3 H), 2.78–2.69 (m, 1 H), 2.63–2.54 (m, 1 H), 1.53–1.38 (m, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 141.1, 140.8, 139.3, 138.4,

129.8, 128.9, 128.5, 128.2, 127.3, 127.3, 127.1, 127.0, 60.5, 51.5, 44.6, 38.7; IR (neat) 3292, 3025, 1486, 1451, 696 cm^{-1}; MS (ESI+) 317.2015 (317.2012 calcd for $C_{22}H_{24}N_2$, M + H^+). The enantiopurity was determined by further elaborating diamine **7** to urea **S2** by reaction with CDI as outlined below.



(+)-(*S*)-5-([1,1'-Biphenyl]-4-ylmethyl)-1-benzylimidazolidin-2-one (S2). A flame-dried round bottom flask equipped with a stirbar and reflux condenser was charged with **7** (46 mg, 0.15 mmol) and THF (1 mL). A solution of 1,1'-carbonyldiimidazole (35 mg, 0.22 mmol) THF (1 mL) was added the resulting mixture was heated to reflux with stirring for 24 h. The mixture was then cooled to rt and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel using dichloromethane/methanol as eluant to afford the title compound (46 mg, 89%) as a colorless solid, mp 110–114 °C: $[\alpha]^{23}_D$ +29.4 (*c* 3.6, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57–7.51 (m, 2 H), 7.48 (d, *J* = 8.2 Hz, 2 H), 7.45–7.39 (m, 2 H), 7.37–7.25 (m, 6 H), 7.10 (d, *J* = 8.2 Hz, 2 H), 4.99 (s, br, 1 H), 4.87 (d, *J* = 15.3 Hz, 1 H), 4.09 (d, *J* = 15.5 Hz, 1 H), 3.74 (tdd, *J* = 8.8, 6.9, 4.5 Hz, 1 H), 3.28 (t, *J* = 8.6 Hz, 1 H), 3.18–3.05 (m, 2 H), 2.65 (dd, *J* = 13.5, 9.2 Hz, 1 H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.5, 140.8, 139.9, 137.3, 135.9, 129.7, 128.9, 128.8, 128.3, 127.7, 127.5, 127.5, 127.1, 56.0, 45.5, 43.8, 38.4; IR (neat) 3215, 1690, 1486, 1487, 1449 cm^{-1}; MS (ESI+) 343.1804 (343.1805 calcd for $C_{23}H_{22}N_2O$, M + H^+). The enantiopurity was determined to be 94:6 er by chiral

HPLC analysis (Chiralcel ODH, 15 cm x 4.6 mm, 7% IPA/Hexanes, 1.0 mL/min, λ 254 nm, RT= 26.7 and 49.0 min).

References

[1] R. I. McDonald, S. S. Stahl, *Angew. Chem.* **2010**, *122*, 5661; *Angew. Chem. Int. Ed.* **2010**, *49*, 5529.

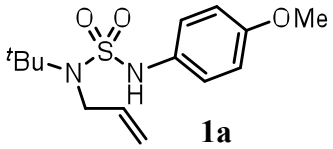
[2] R. M. Fornwald, J. A. Fritz, J. P. Wolfe, *Chem. Eur. J.* **2014**, *20*, 8782.

[3] B. A. Hopkins, J. P. Wolfe, *Angew. Chem. Int. Ed*. **2012**, *51*, 9886; *Angew Chem.* **2012**, *124*, 10024.

[4] M. Ordóñez, R. de la Cruz-Cordero, C. Quiñónes, A. González-Morales, *Chem. Commun*. **2004**, 672.

Agilent Technologies

Sample Name		Pulse sequence	PROTON	Temperature	25	Study owner	garletsz
Date collected	2015-10-20	Solvent	cdcl3	Operator	garletsz	Printed from	te.chem.lsa.umich.edu-vnmrs500



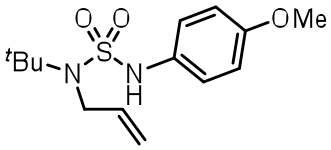
1a

Data file /home/garletsz/vnmrsys/data/zjg.jpw.2.122.A.p.1.H1.fid

Plot date 2015-10-20

Agilent Technologies

Sample Name		Pulse sequence	CARBON	Temperature	25	Study owner	garletsz
Date collected	2015-10-20	Solvent	cdcl3	Operator	garletsz	Printed from	te.chem.lsa.umich.edu-vnmrs500



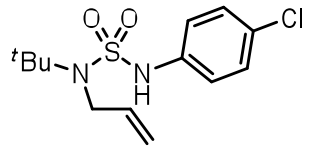
1a

Data file /home/garletsz/vnmrsys/data/zjg.jpw.2.122.A.p.2.C13.fid

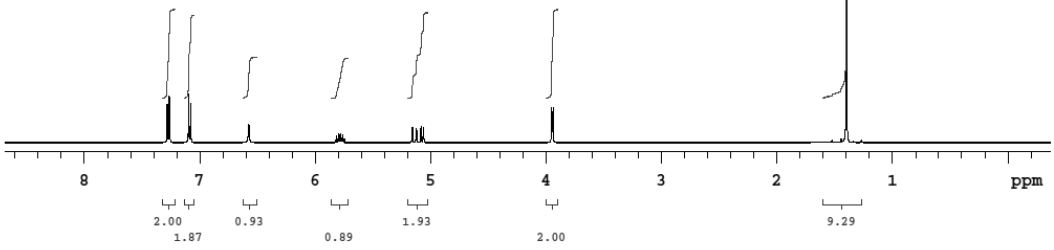
Plot date 2015-10-20

Agilent Technologies

Sample Name
Date collected 2015-10-20
Pulse sequence PROTON
Solvent cdcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs500



1b

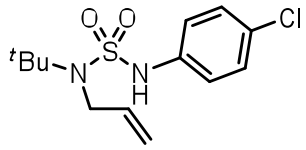


Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.2.114.p.1.H1.fid

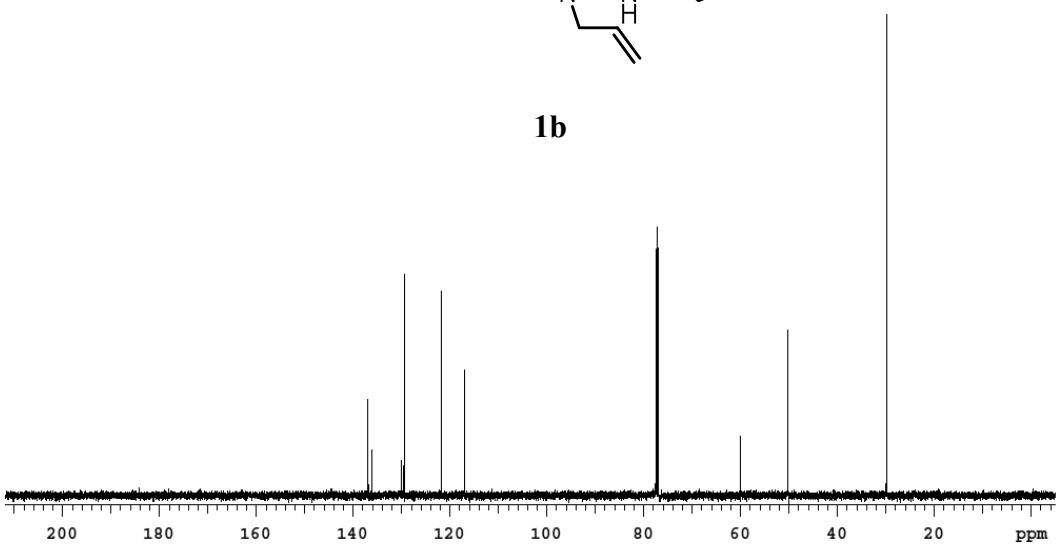
Plot date 2015-10-20

Agilent Technologies

Sample Name
Date collected 2015-10-20
Pulse sequence CARBON
Solvent cdcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs500

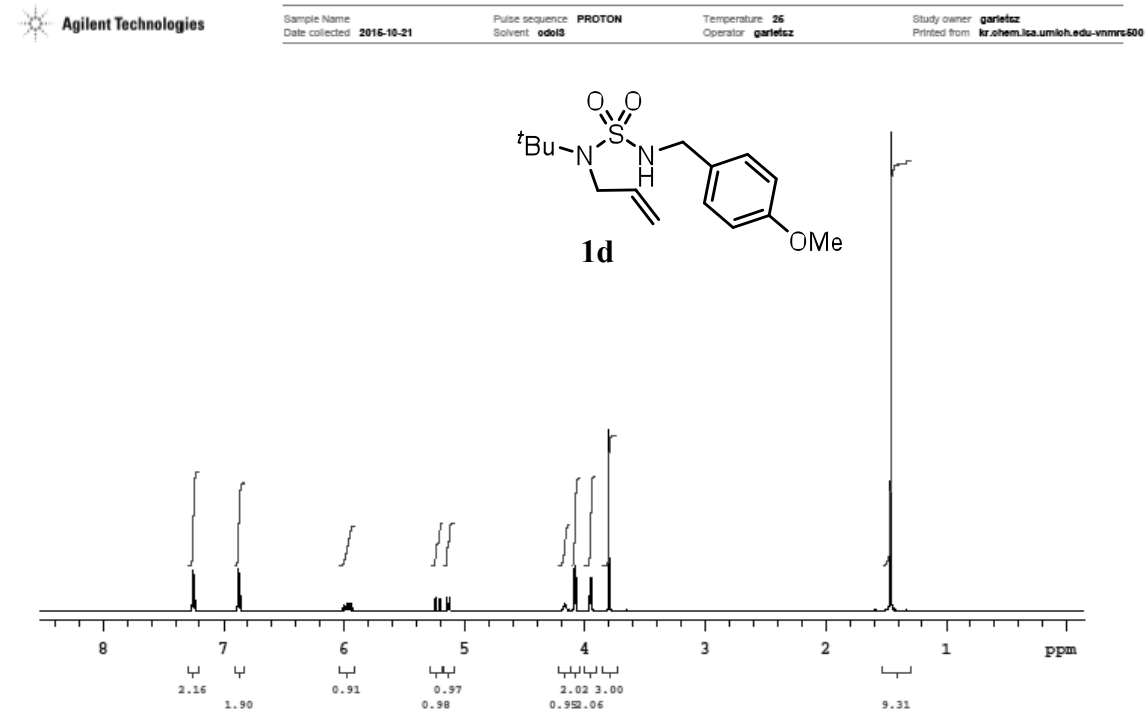


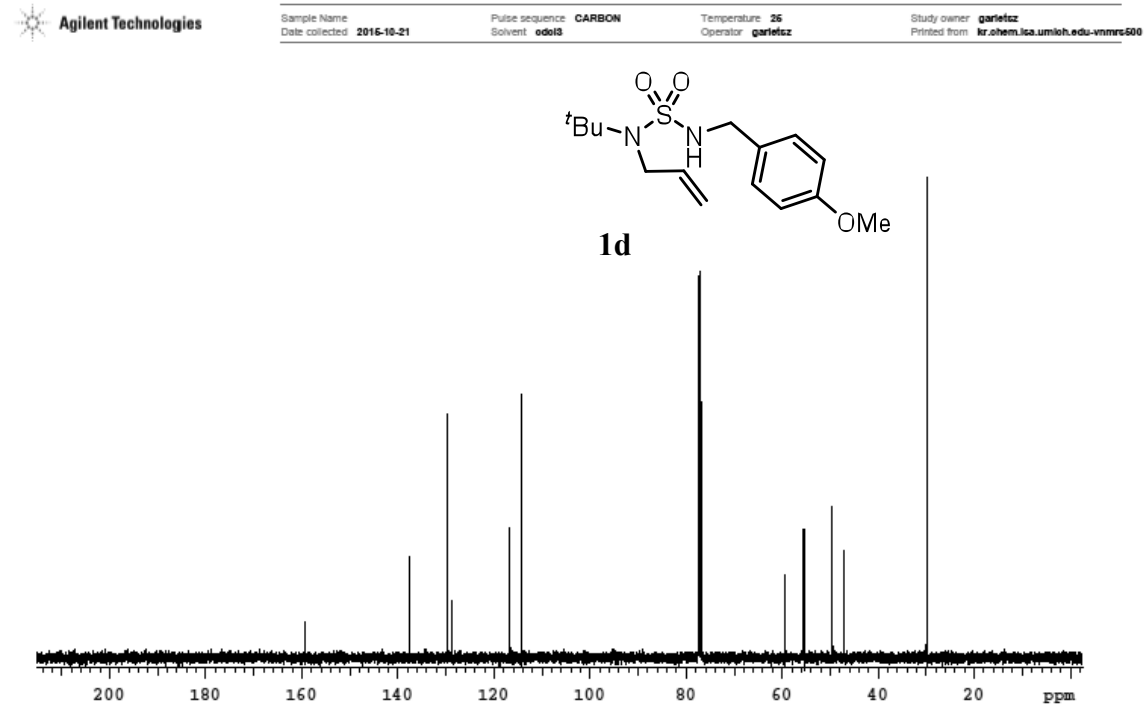
1b



Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.2.114.p.2.C13.fid

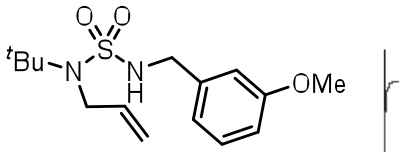
Plot date 2015-10-20


Agilent Technologies
Sample Name
Date collected 2016-10-21
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs500
1d
OMe
8
7
6
5
4
3
2
1
ppm
2.16
1.90
0.91
0.97
0.98
2.02
3.00
0.95
2.06
9.31
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.2.112.B.p.1.H1.fid
Plot date 2015-10-21

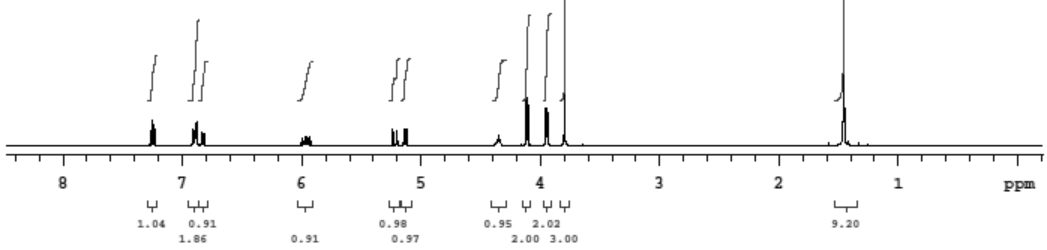

Agilent Technologies
Sample Name
Date collected 2016-10-21
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs500
1d
OMe
200
180
160
140
120
100
80
60
40
20
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.2.112.B.p.2.C13.fid
Plot date 2015-10-21

Agilent Technologies

Sample Name
Date collected 2015-10-21
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs500



1e

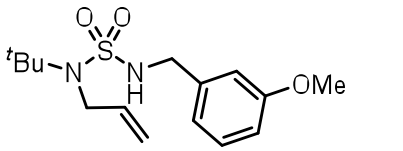


Data file /misc/Tellurium/garletsz/vnmrsys/data/zdg.jpw.2.168.p.1.H1.fid

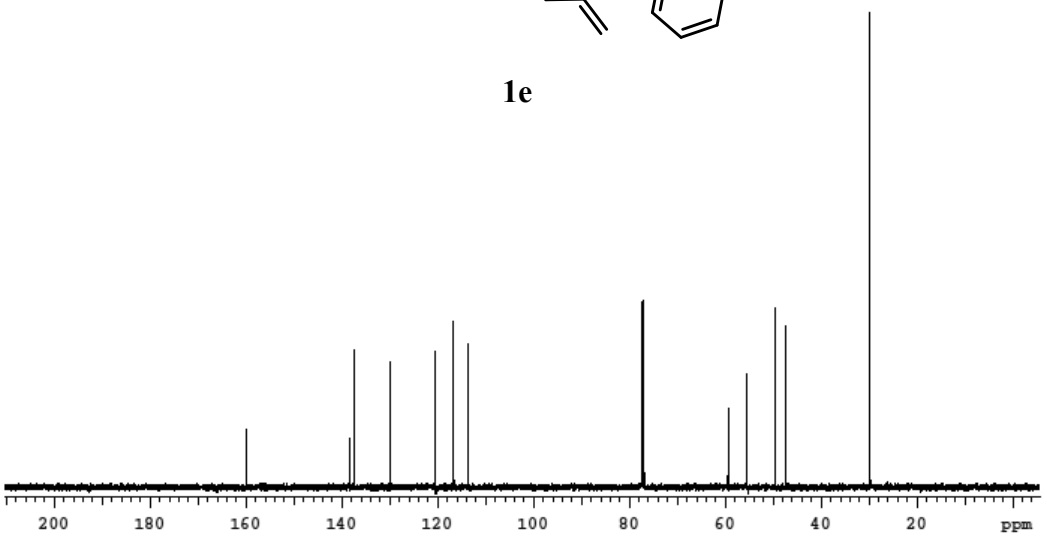
Plot date 2015-10-21

Agilent Technologies

Sample Name
Date collected 2015-10-21
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs500



1e

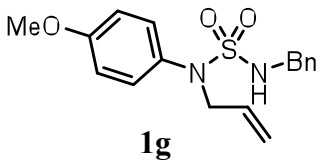


Data file /misc/Tellurium/garletsz/vnmrsys/data/zdg.jpw.2.168.p.2.C13.fid

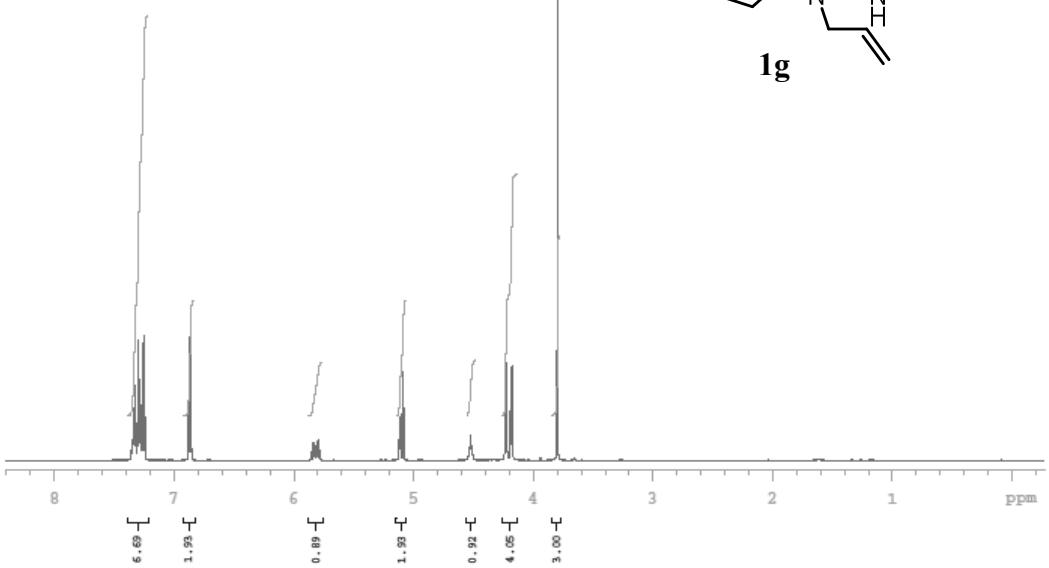
Plot date 2015-10-21

Agilent Technologies

Sample Name
Date collected 2016-10-20
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from te.ohem.lca.umich.edu-vnmrs600



1g

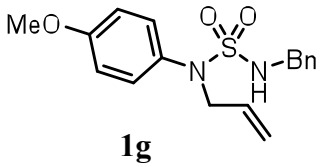


Data file /home/garletsz/vnmrsys/data/kp.jpw.1.13.p.1.H1.fid

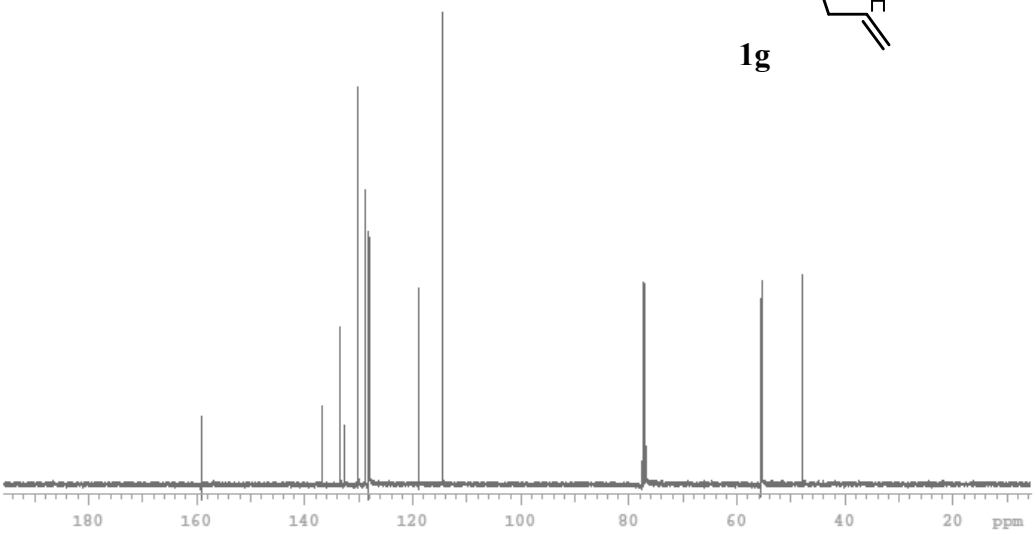
Plot date 2015-10-20

Agilent Technologies

Sample Name
Date collected 2016-10-20
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from te.ohem.lca.umich.edu-vnmrs600



1g

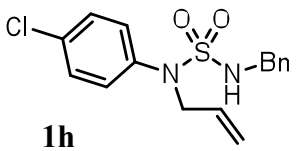


Data file /home/garletsz/vnmrsys/data/kp.jpw.1.13.p.2.C13.fid

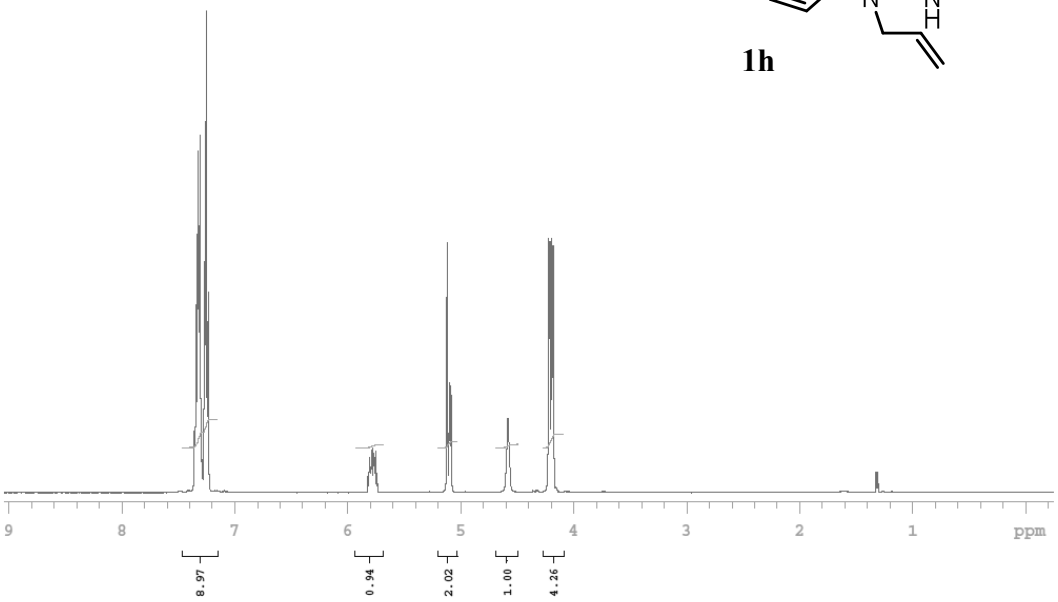
Plot date 2015-10-20

Agilent Technologies

Sample Name
Date collected 2015-10-20
Pulse sequence PROTON
Solvent cdcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from te.chem.lsa.umich.edu-vnmrs500



1h

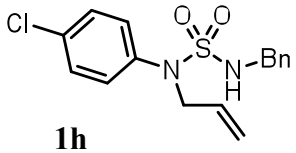


Data file /home/garletsz/vnmrsys/data/kp.jpw.1.15.p.1.H1.fid

Plot date 2015-10-20

Agilent Technologies

Sample Name
Date collected 2015-10-20
Pulse sequence CARBON
Solvent cdcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from te.chem.lsa.umich.edu-vnmrs500



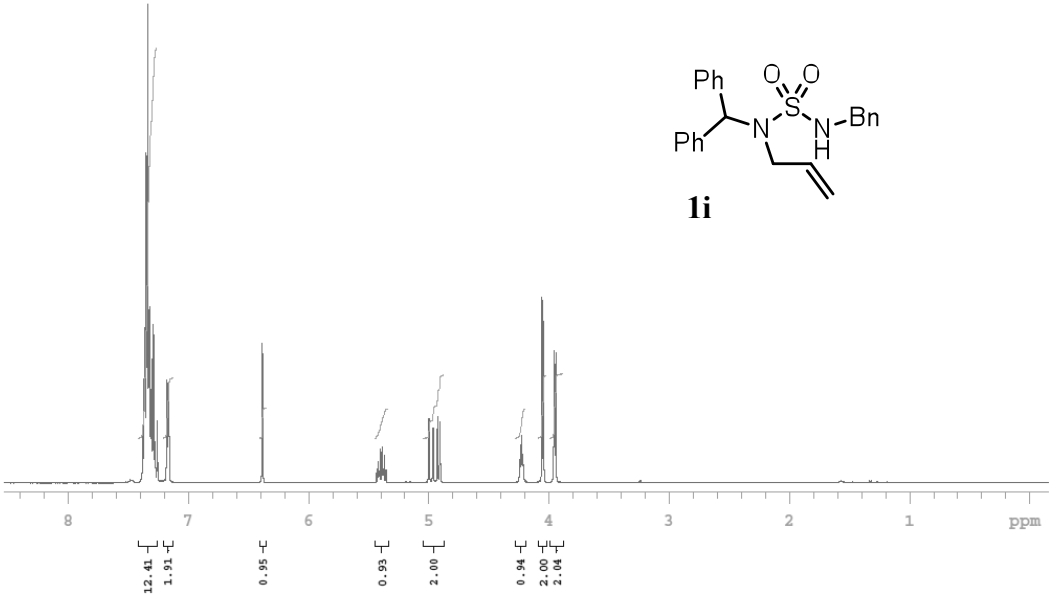
1h

Data file /home/garletsz/vnmrsys/data/kp.jpw.1.15.p.2.C13.fid

Plot date 2015-10-20

Agilent Technologies

Sample Name		Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**garletsz**
Date collected	**2015-10-20**	Solvent	**cdcl3**	Operator	**garletsz**	Printed from	**te.chem.lsa.umich.edu-vnmrs500**



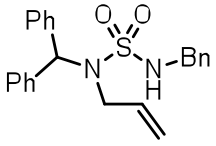
1i

Data file /home/garletsz/vnmrsys/data/kp.jpw.1.24.p.1.H1.fid

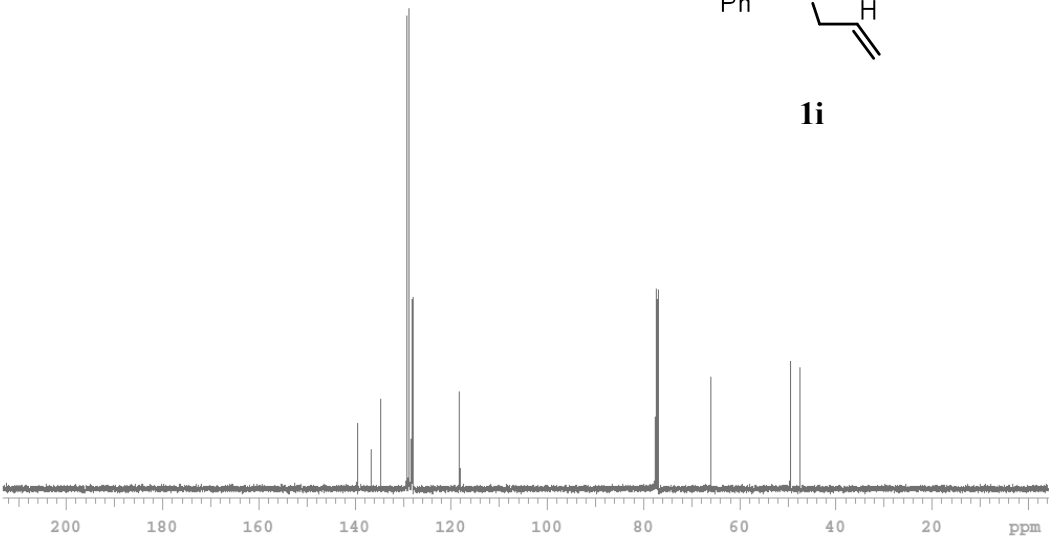
Plot date 2015-10-20

Agilent Technologies

Sample Name		Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**garletsz**
Date collected	**2015-10-20**	Solvent	**cdcl3**	Operator	**garletsz**	Printed from	**te.chem.lsa.umich.edu-vnmrs500**

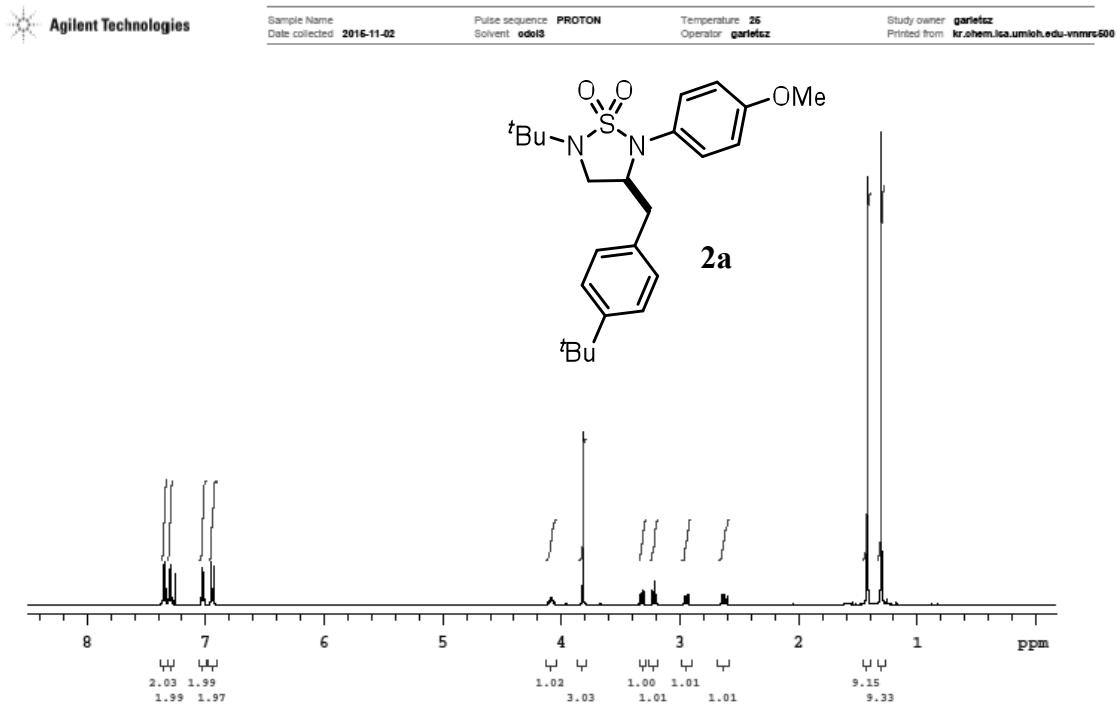


1i



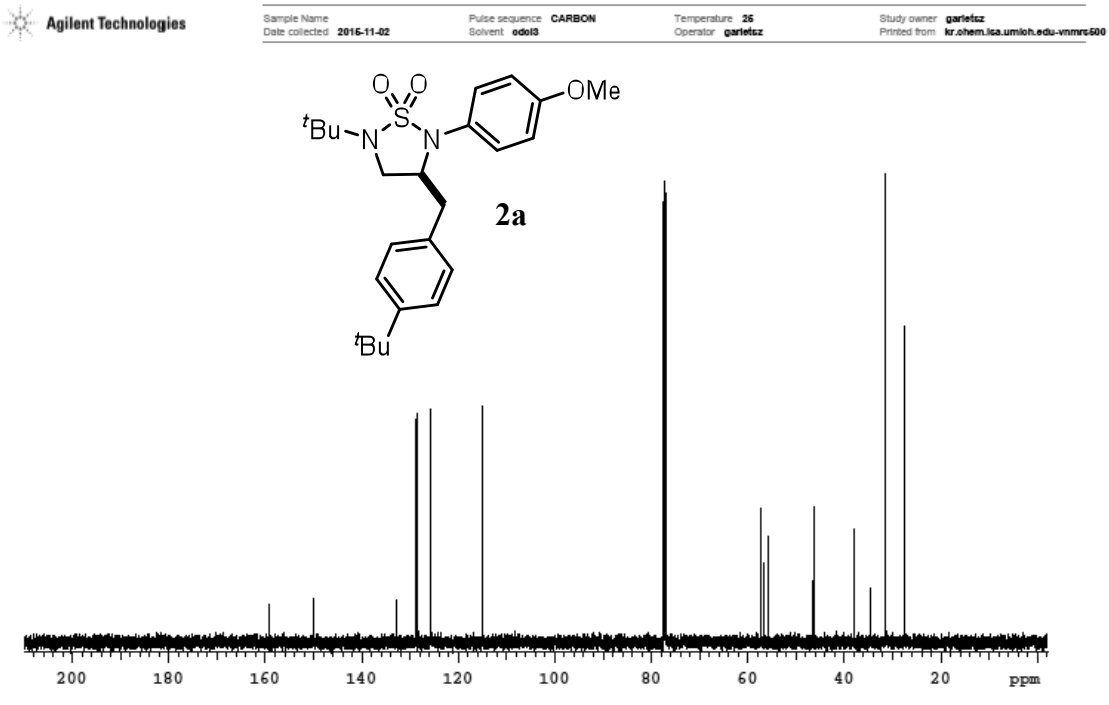
Data file /home/garletsz/vnmrsys/data/kp.jpw.1.24.p.2.C13.fid

Plot date 2015-10-20



Data file /misc/Telurium/garletsz/vnmrsys/data/kp.1.106.p.1.H1.fid

Plot date 2015-11-02



Data file /misc/Telurium/garletsz/vnmrsys/data/kp.jpw.1.106.p.C13.fid

Plot date 2015-11-02

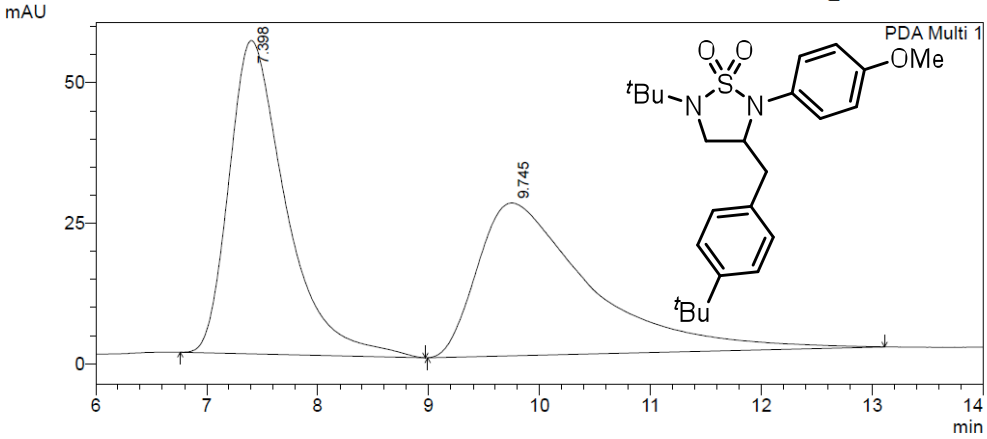
==== Shimadzu LCsolution Analysis Report ====

C:\...\Zac G\Chiral 5 Membered Sulfonamides\RAC-ZJG-2-139B-PMP-XPhos-PhtBu-7.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-2-139B-PMP-XPhos-PhtBu-7.00%IPA-1.0mL_min-ODH1.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-2-139B-PMP-XPhos-PhtBu-7.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/5/2015 12:42:08 PM
Data Processed	: 11/5/2015 1:09:00 PM

<Chromatogram>

C:\...\Chiral 5 Membered Sulfonamides\RAC-ZJG-2-139B-PMP-XPhos-PhtBu-7.00%IPA-1.0mL_min-ODH1.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

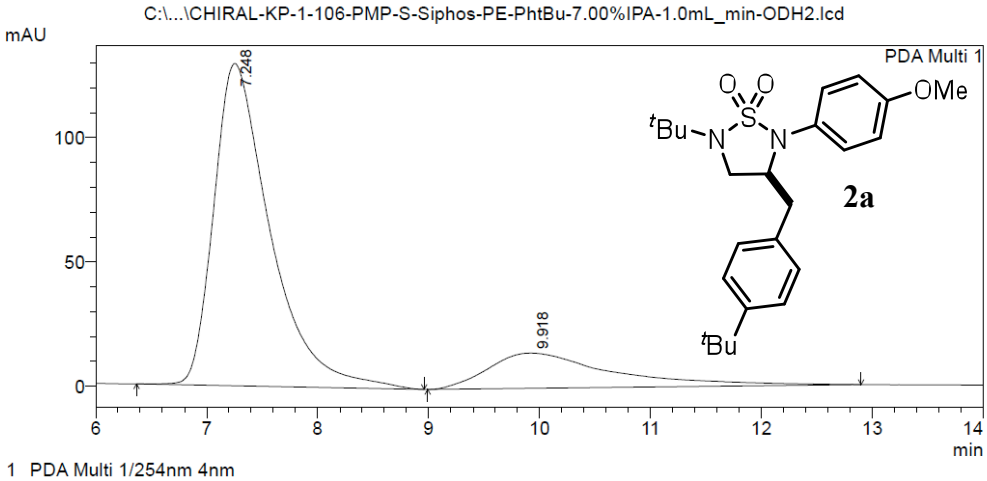
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.398	1982335	55769	50.831	67.233
2	9.745	1917485	27179	49.169	32.767
Total		3899820	82948	100.000	100.000

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

C:\...\Chiral 5 Membered Sulfonamides\CHIRAL-KP-1-106-PMP-S-Siphos-PE-PhtBu-7.00%IPA-1.0mL_min-ODH2.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-KP-1-106-PMP-S-Siphos-PE-PhtBu-7.00%IPA-1.0mL_min-ODH2.l
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-KP-1-106-PMP-S-Siphos-PE-PhtBu-7.00%IPA-1.0mL_min-ODH2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/5/2015 1:12:03 PM
Data Processed	: 11/5/2015 1:58:45 PM

<Chromatogram>



1 PDA Multi 1/254nm 4nm

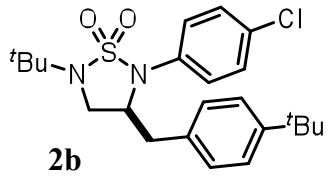
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.248	4577723	129624	80.623	90.181
2	9.918	1100248	14114	19.377	9.819
Total		5677971	143738	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2018-01-16
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs600



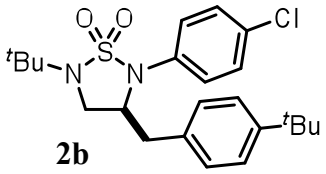
2b

Data file /misc/Ytterbium/garletsz/vnmrsys/data/zjg.jpw.2.12S.p.1.H1.fid

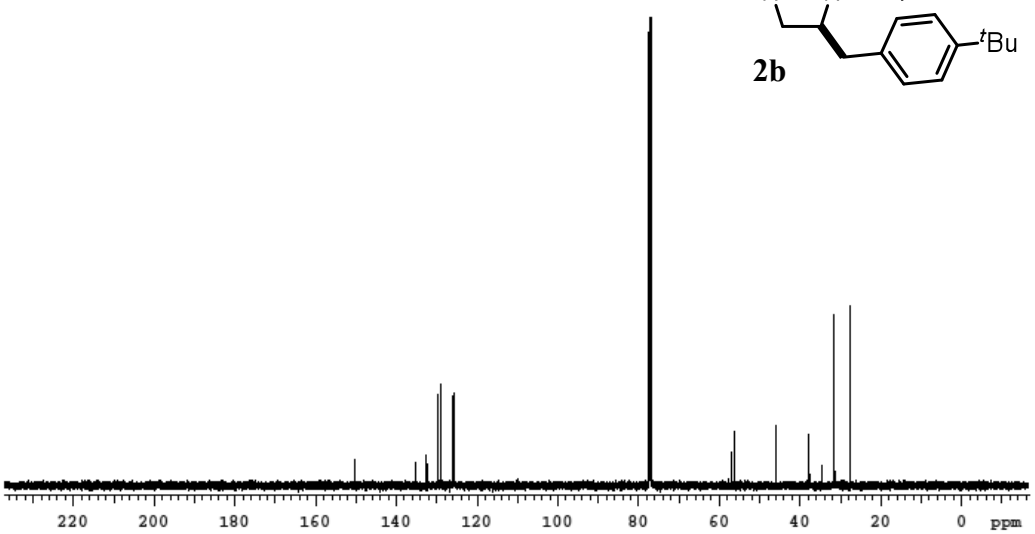
Plot date 2016-01-15

Agilent Technologies

Sample Name
Date collected 2018-01-16
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs600



2b



Data file /misc/Ytterbium/garletsz/vnmrsys/data/zjg.jpw.2.12S.p.2.C13.fid

Plot date 2016-01-15

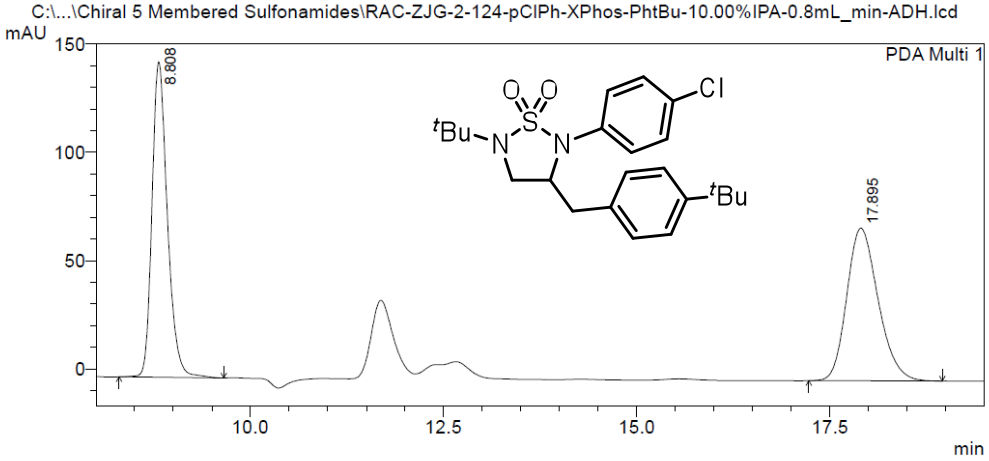
==== Shimadzu LCsolution Analysis Report ====

C:\...\Zac G\Chiral 5 Membered Sulfonamides\RAC-ZJG-2-124-pClPh-XPhos-PhtBu-10.00%IPA-0.8mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-2-125-pClPh-S-Siphos-10.00%IPA-0.8mL_min-ODH.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-2-124-pClPh-XPhos-PhtBu-10.00%IPA-0.8mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/6/2015 9:29:59 AM
Data Processed	: 3/6/2015 9:53:02 AM

<Chromatogram>

C:\...\Chiral 5 Membered Sulfonamides\RAC-ZJG-2-124-pClPh-XPhos-PhtBu-10.00%IPA-0.8mL_min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.808	2019951	145837	50.391	67.409
2	17.895	1988589	70509	49.609	32.591
Total		4008540	216346	100.000	100.000

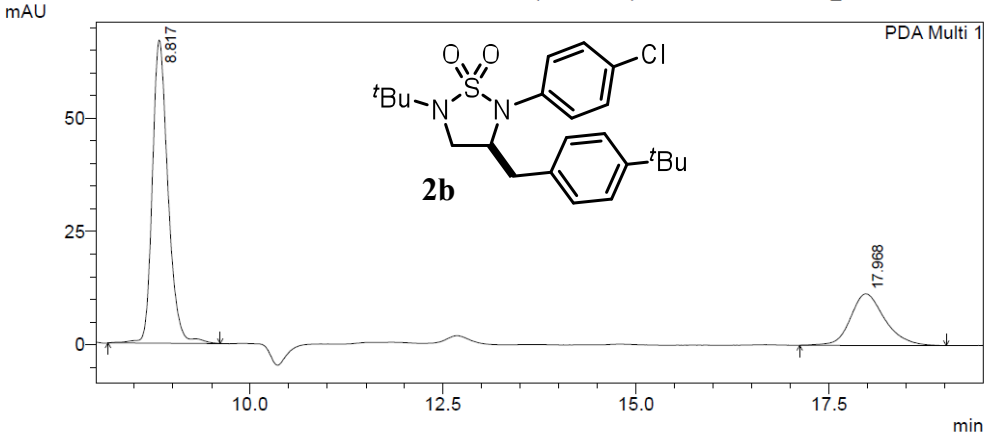
==== Shimadzu LCsolution Analysis Report ====

C:\...\Zac G\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-2-125-pClPh-S-Siphos-10.00%IPA-0.8mL_min-ADH2.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-2-125-pClPh-S-Siphos-10.00%IPA-0.8mL_min-ADH2.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-2-125-pClPh-S-Siphos-10.00%IPA-0.8mL_min-ADH2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/6/2015 10:25:02 AM
Data Processed	: 3/6/2015 10:53:56 AM

<Chromatogram>

C:\...\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-2-125-pClPh-S-Siphos-10.00%IPA-0.8mL_min-ADH2.lcd

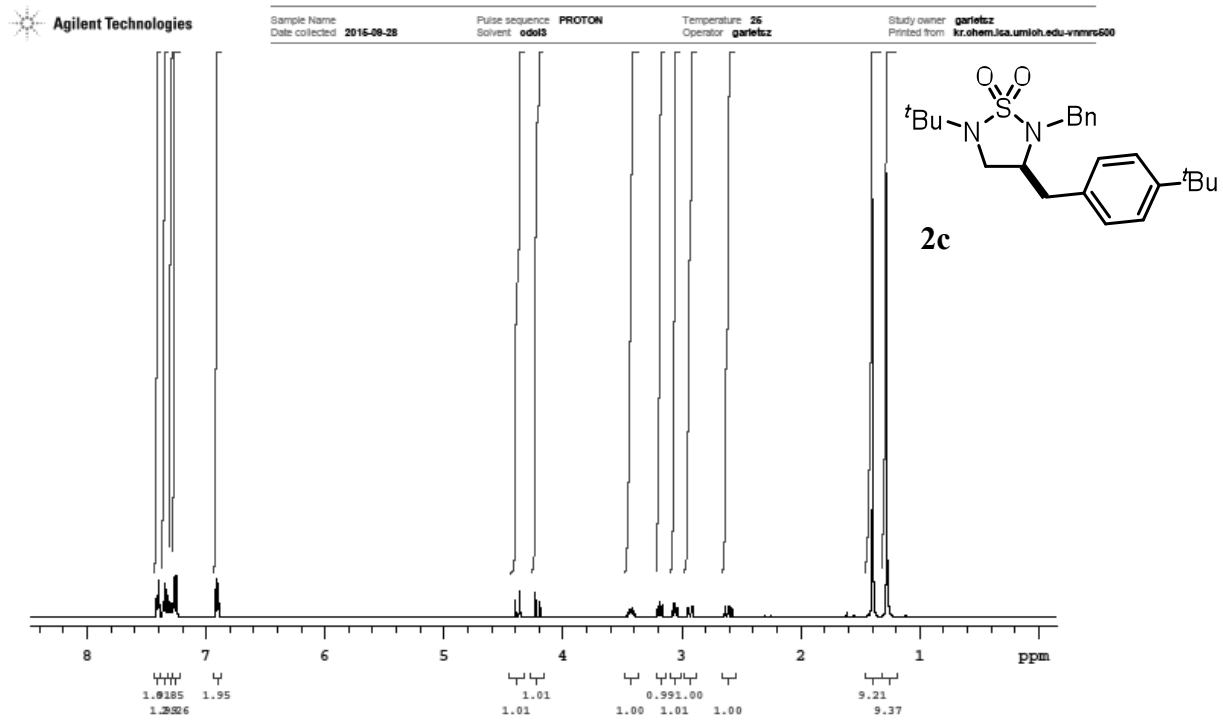


1 PDA Multi 1/254nm 4nm

PeakTable

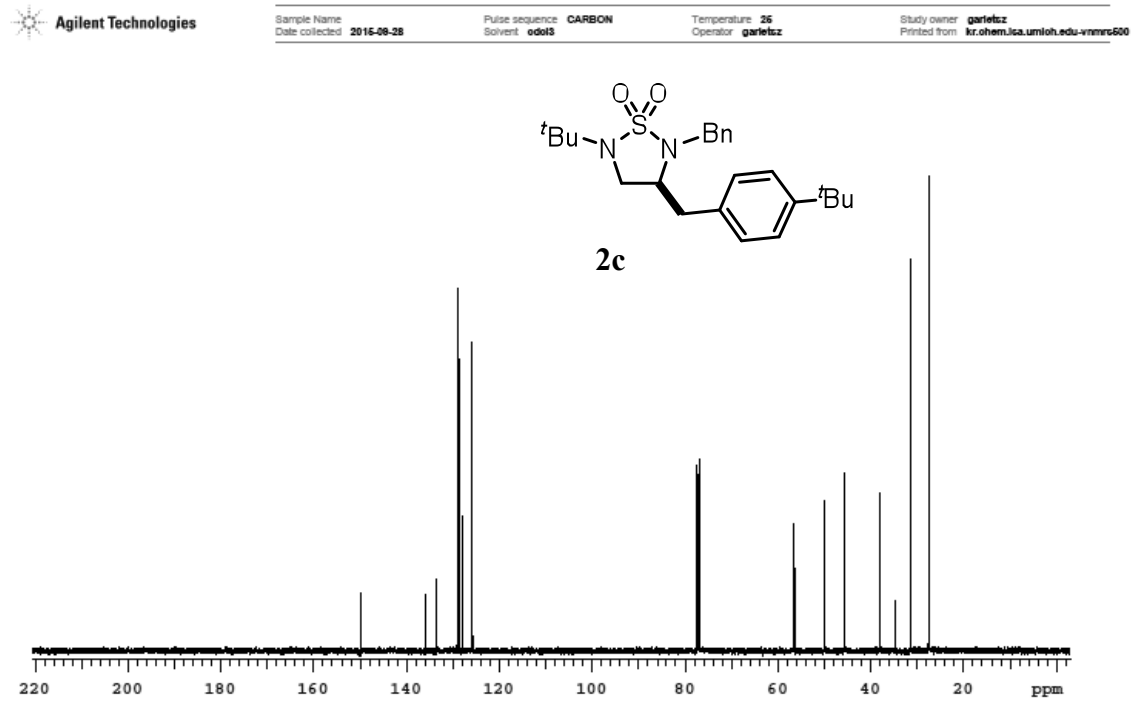
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.817	945205	66930	72.825	85.447
2	17.968	352707	11400	27.175	14.553
Total		1297912	78330	100.000	100.000



Data file /misc/Cobalt/garletsz/vnmrsys/data/zjg.jpw.3.154.p.3.H1.fid

Plot date 2015-09-28



Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.154.p.2.C13.fid

Plot date 2015-09-28

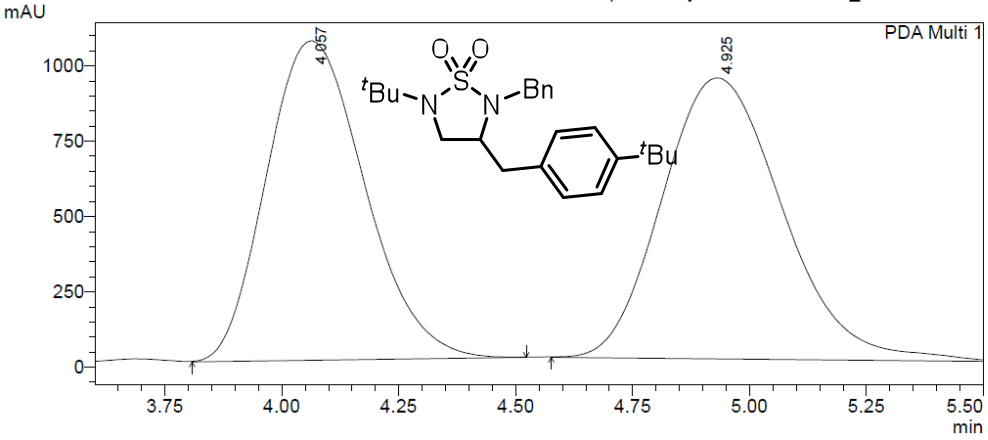
==== Shimadzu LCsolution Analysis Report ====

C:\...\Kaia Parenti\5 Membered Sulfonamides\RAC-KP-1-43-Bn-XPhos-p-tert-butyl-7.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: RAC-KP-1-43-Bn-XPhos-p-tert-butyl-7.00%IPA-1.0mL_min-ODH1.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-KP-1-43-Bn-XPhos-p-tert-butyl-7.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/31/2015 1:41:48 PM
Data Processed	: 10/31/2015 1:51:53 PM

<Chromatogram>

C:\...\Kaia Parenti\5 Membered Sulfonamides\RAC-KP-1-43-Bn-XPhos-p-tert-butyl-7.00%IPA-1.0mL_min-ODH1.lcd



1 PDA Multi 1/205nm 4nm

PeakTable

PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.057	15552282	1061540	48.182	53.212
2	4.925	16726211	933380	51.818	46.788
Total		32278493	1994920	100.000	100.000

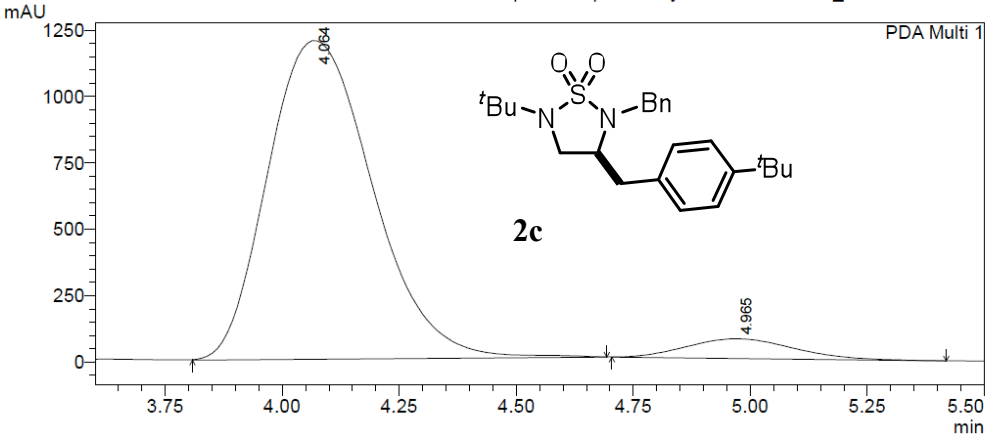
==== Shimadzu LCsolution Analysis Report ====

C:\...\5 Membered Sulfonamides\CHIRAL-ZJG-3-171-S-Siphos-PE-p-tert-butyl-7.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-171-S-Siphos-PE-p-tert-butyl-7.00%IPA-1.0mL_min-OD
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-171-S-Siphos-PE-p-tert-butyl-7.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/31/2015 1:53:53 PM
Data Processed	: 10/31/2015 2:12:23 PM

<Chromatogram>

C:\...\5 Membered Sulfonamides\CHIRAL-ZJG-3-171-S-Siphos-PE-p-tert-butyl-7.00%IPA-1.0mL_min-ODH1.lcd

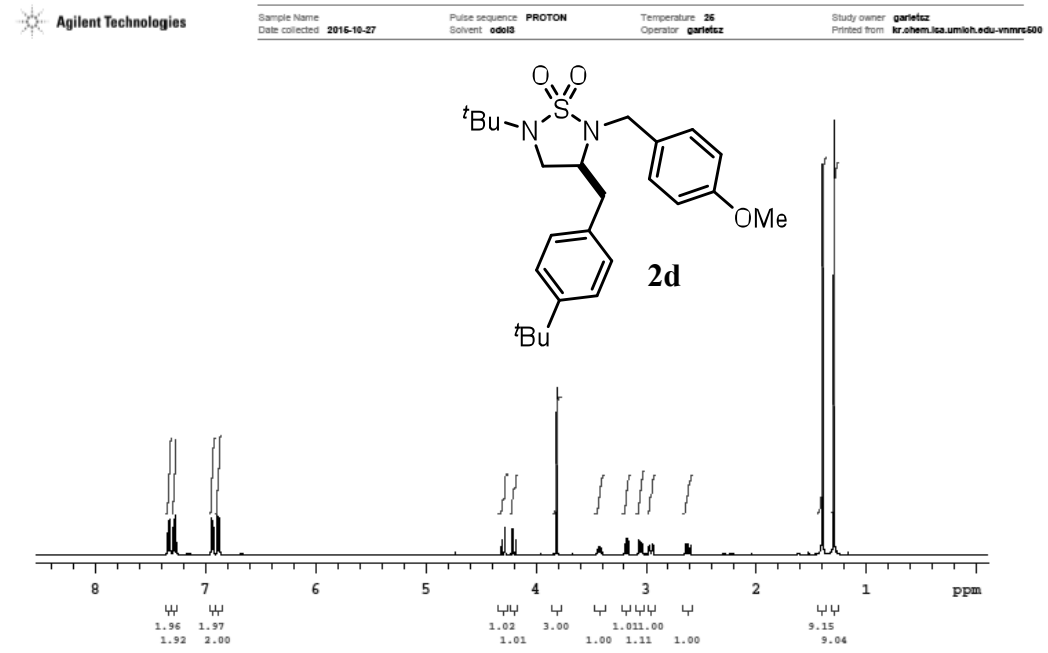


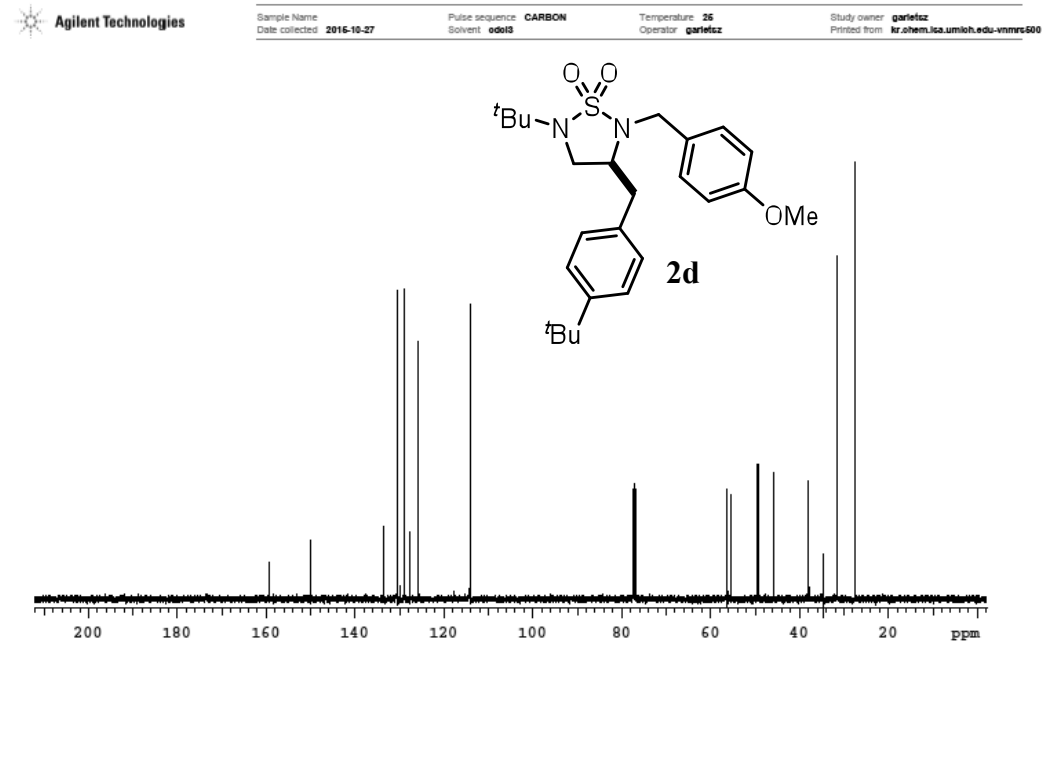
1 PDA Multi 1/205nm 4nm

PeakTable

PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.064	18734172	1199562	94.078	94.163
2	4.965	1179374	74356	5.922	5.837
Total		19913546	1273917	100.000	100.000


Agilent Technologies
Sample Name
Date collected 2016-10-27
Pulse sequence PROTON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from kr.ohem.ksa.umich.edu-vnmrs500
2d
ppm
1.96 1.97
1.92 2.00
1.02 3.00
1.01
1.00 1.11 1.00
1.011.00
9.15
9.04
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.2.140.c.p.1.H1.fid
Plot date 2015-10-27


Agilent Technologies
Sample Name
Date collected 2016-10-27
Pulse sequence CARBON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from kr.ohem.ksa.umich.edu-vnmrs500
2d
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.2.140.c.p.2.C13.fid
Plot date 2015-10-27

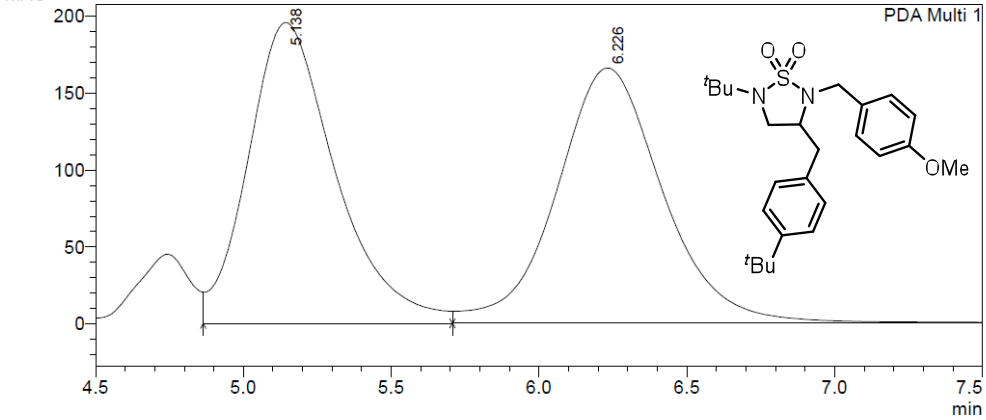
==== Shimadzu LCsolution Analysis Report ====

C:\...\Zac G\Chiral 5 Membered Sulfonamides\RAC-ZJG-2-139A-BnOMe-ptertbutyl-7.00%IPA-1.0mL_min-ODH2.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-2-139A-BnOMe-ptertbutyl-7.00%IPA-1.0mL_min-ODH2.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-2-139A-BnOMe-ptertbutyl-7.00%IPA-1.0mL_min-ODH2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/2/2015 12:08:43 PM
Data Processed	: 11/2/2015 12:29:02 PM

<Chromatogram>

C:\...\Zac G\Chiral 5 Membered Sulfonamides\RAC-ZJG-2-139A-BnOMe-ptertbutyl-7.00%IPA-1.0mL_min-ODH2.lcd



1 PDA Multi 1/215nm 4nm

PeakTable

PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.138	4028594	195872	49.169	54.147
2	6.226	4164686	165873	50.831	45.853
Total		8193280	361745	100.000	100.000

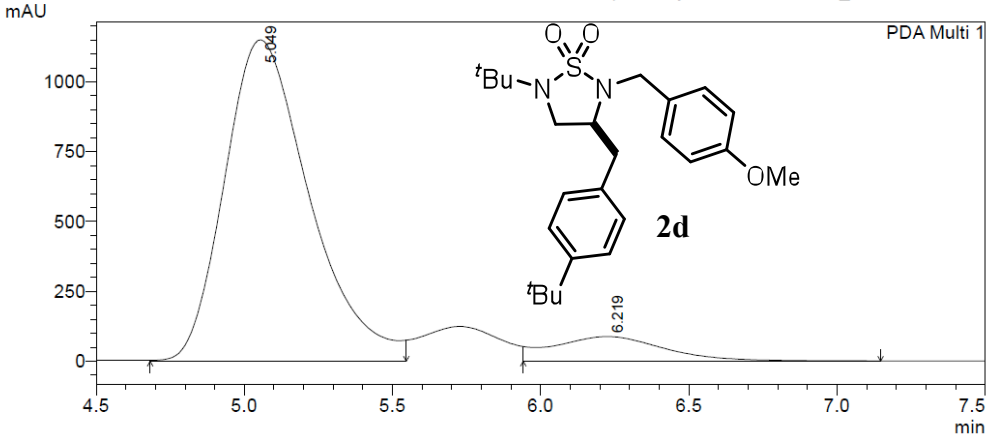
==== Shimadzu LCsolution Analysis Report ====

C:\...\Zac G\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-2-140C-BnOMe-ptertbutyl-7.00%IPA-1.0mL_min-ODH3.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-2-140C-BnOMe-ptertbutyl-7.00%IPA-1.0mL_min-ODH3.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-2-140C-BnOMe-ptertbutyl-7.00%IPA-1.0mL_min-ODH3.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/2/2015 12:31:24 PM
Data Processed	: 11/2/2015 12:44:01 PM

<Chromatogram>

C:\...\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-2-140C-BnOMe-ptertbutyl-7.00%IPA-1.0mL_min-ODH3.lcd



1 PDA Multi 1/215nm 4nm

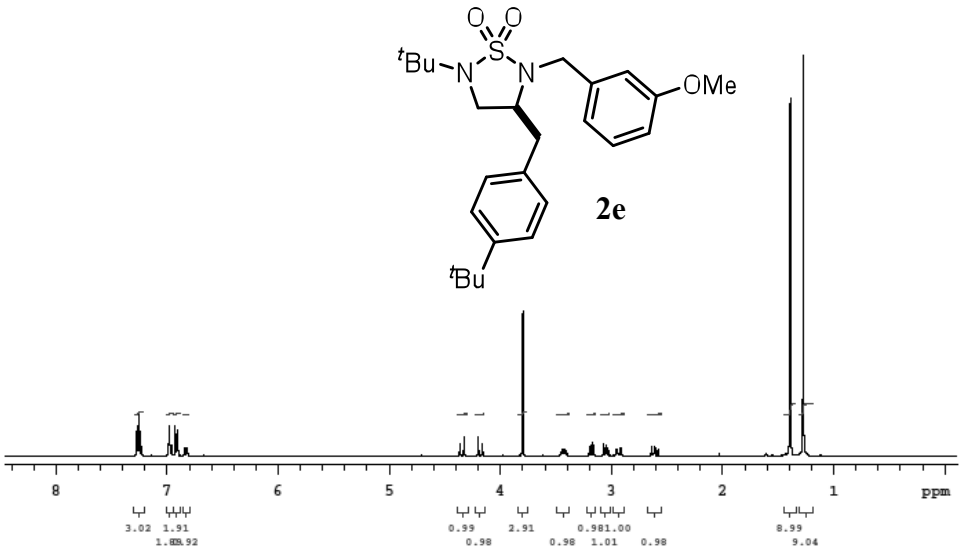
PeakTable

PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.049	22943307	1150159	90.827	92.916
2	6.219	2317251	87685	9.173	7.084
Total		25260558	1237844	100.000	100.000

Agilent Technologies

Sample Name	Pulse sequence PROTON	Temperature 25	Study owner garletsz
Date collected 2016-10-28	Solvent odcl3	Operator garletsz	Printed from kr.ohem.lsa.umich.edu-vnmrs600

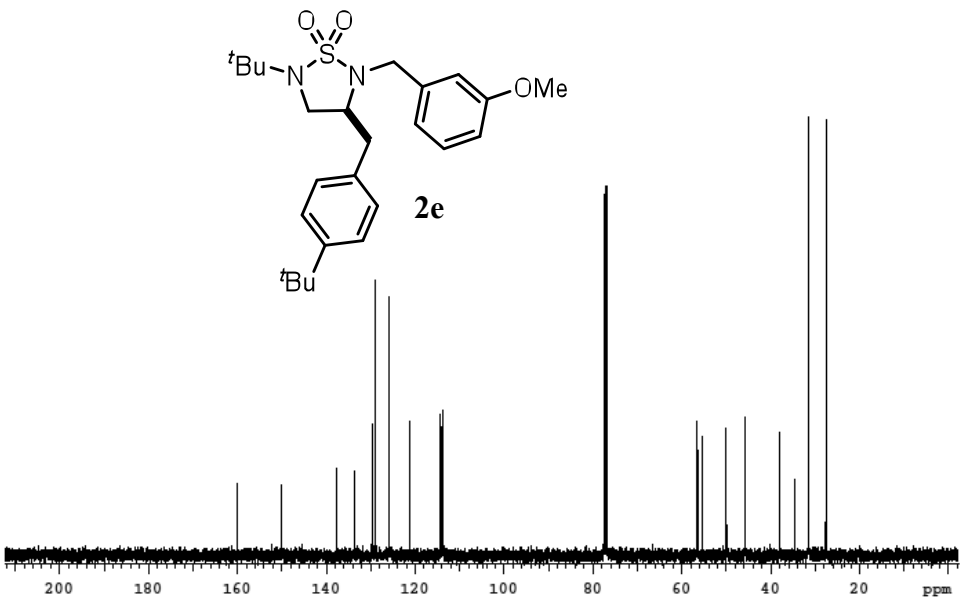


Data file /misc/Cobalt/garletsz/vnmrsys/data/zjg.jpw.2.170.p.1.H1.fid

Plot date 2015-10-28

Agilent Technologies

Sample Name	Pulse sequence CARBON	Temperature 25	Study owner garletsz
Date collected 2016-10-28	Solvent odcl3	Operator garletsz	Printed from kr.ohem.lsa.umich.edu-vnmrs600



Data file /misc/Cobalt/garletsz/vnmrsys/data/zjg.jpw.2.170.p.2.C13.fid

Plot date 2015-10-28

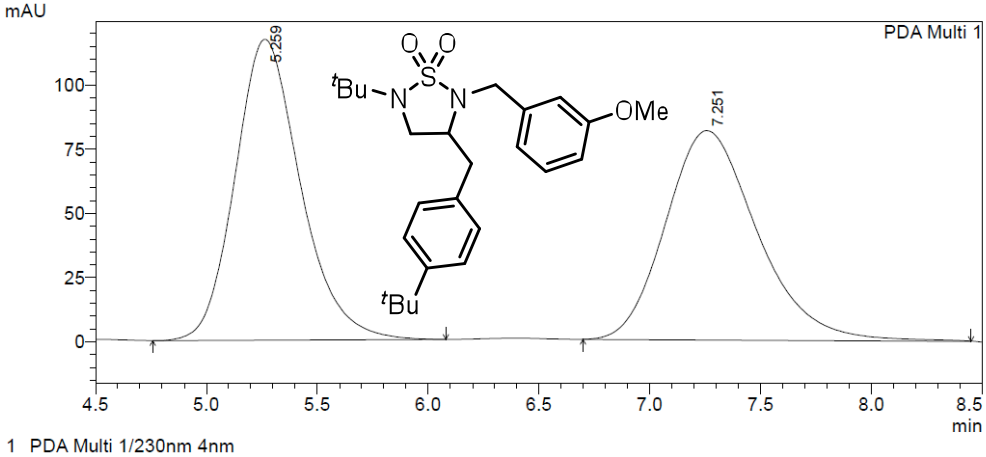
==== Shimadzu LCsolution Analysis Report ====

C:\...\Data\Zac G\Chiral 5 Membered Sulfonamides\RAC-ZJG-2-169-Bn-ptertbutyl-7.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-2-169-Bn-ptertbutyl-7.00%IPA-1.0mL_min-ODH.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-2-169-Bn-ptertbutyl-7.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/2/2015 11:24:11 AM
Data Processed	: 11/2/2015 11:43:05 AM

<Chromatogram>

C:\...\Data\Zac G\Chiral 5 Membered Sulfonamides\RAC-ZJG-2-169-Bn-ptertbutyl-7.00%IPA-1.0mL_min-ODH1.lcd



1 PDA Multi 1/230nm 4nm

PeakTable

PDA Ch1 230nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.259	2407126	117068	50.622	58.953
2	7.251	2347997	81511	49.378	41.047
Total		4755123	198579	100.000	100.000

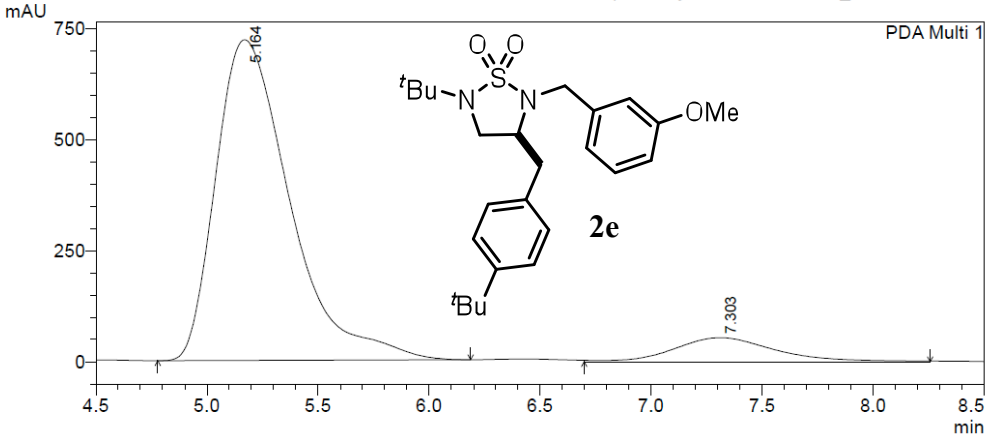
==== Shimadzu LCsolution Analysis Report ====

C:\...\Data\Zac G\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-2-170-Bn-ptertbutyl-7.00%IPA-1.0mL_min-ODH2.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-2-170-Bn-ptertbutyl-7.00%IPA-1.0mL_min-ODH2.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-2-170-Bn-ptertbutyl-7.00%IPA-1.0mL_min-ODH2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/2/2015 11:45:22 AM
Data Processed	: 11/2/2015 11:59:06 AM

<Chromatogram>

C:\...\Zac G\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-2-170-Bn-ptertbutyl-7.00%IPA-1.0mL_min-ODH2.lcd



1 PDA Multi 1/230nm 4nm

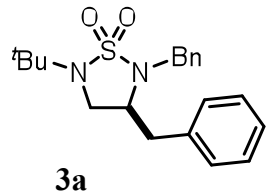
PeakTable

PDA Ch1 230nm 4nm

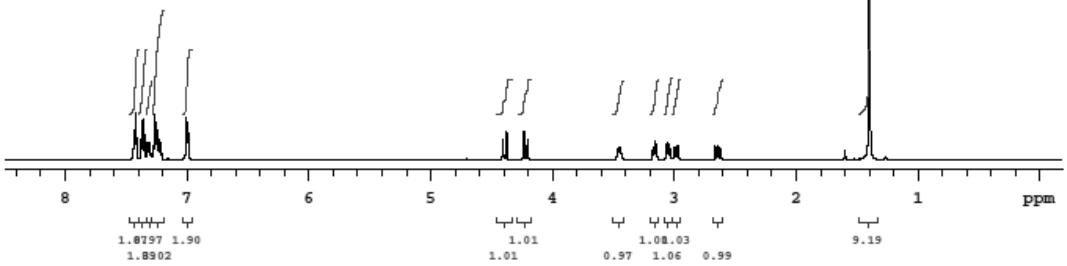
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.164	16739376	722017	90.548	92.985
2	7.303	1747380	54469	9.452	7.015
Total		18486756	776486	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2016-08-09
Pulse sequence PROTON
Solvent odcl3
Temperature 23
Operator garletsz
Study owner heiko
Printed from dy.chem.lsa.umich.edu-vnmrs500



3a

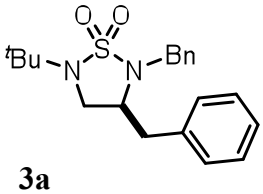


Data file /misc/Tellurium/kparenti/vnmrsys/data/kp.jpw.1.84.H1.P.fid

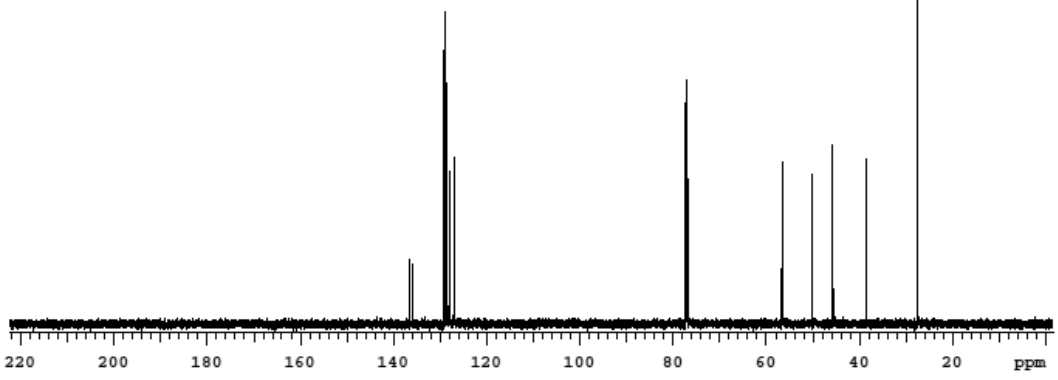
Plot date 2016-01-14

Agilent Technologies

Sample Name
Date collected 2016-08-09
Pulse sequence CARBON
Solvent odcl3
Temperature 23
Operator garletsz
Study owner kparenti
Printed from kr.chem.lsa.umich.edu-vnmrs500



3a



Data file /misc/Tellurium/kparenti/vnmrsys/data/kp.jpw.1.84.C13.P.fid

Plot date 2015-09-28

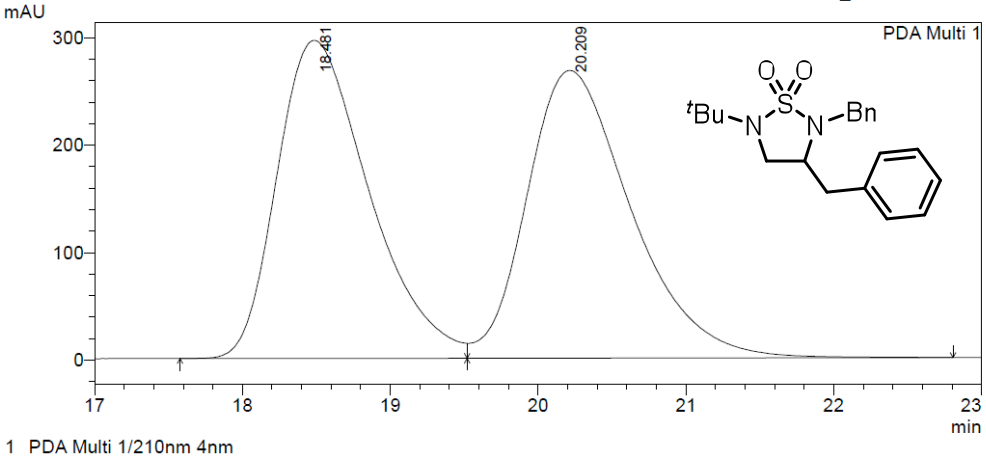
==== Shimadzu LCsolution Analysis Report ====

C:\...\Data\Zac G\Chiral 5 Membered Sulfonamides\RAC-ZJG-2-174-1-Bn-XPhos-Ph-4.00%IPA-0.5mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-2-174-1-Bn-XPhos-Ph-4.00%IPA-0.5mL_min-ODH.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-2-174-1-Bn-XPhos-Ph-4.00%IPA-0.5mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/3/2015 1:28:41 PM
Data Processed	: 9/3/2015 1:59:34 PM

<Chromatogram>

C:\...\Zac G\Chiral 5 Membered Sulfonamides\RAC-ZJG-2-174-1-Bn-XPhos-Ph-4.00%IPA-0.5mL_min-ODH.lcd



1 PDA Multi 1/210nm 4nm

PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.481	12803077	295938	49.430	52.505
2	20.209	13098442	267704	50.570	47.495
Total		25901519	563642	100.000	100.000

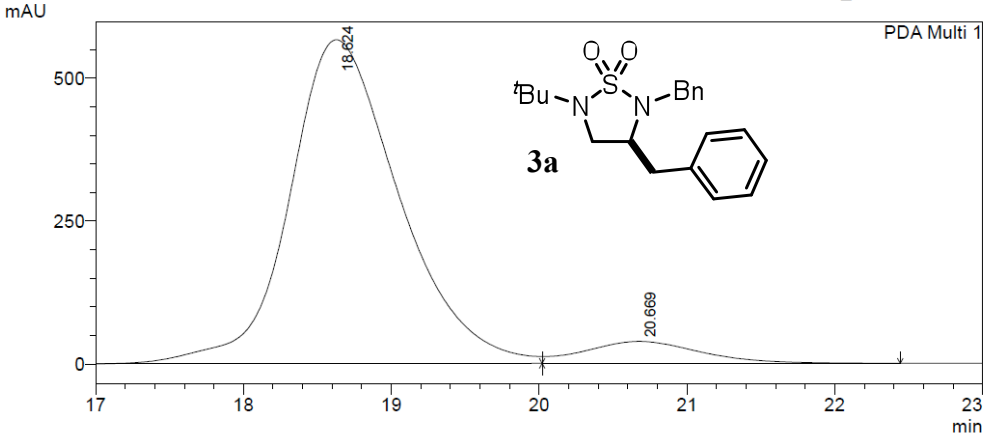
==== Shimadzu LCsolution Analysis Report ====

C:\...\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-3-158-Bn-S-SiPhos-PE-Ph-4.00%IPA-0.5mL_min-ODH2.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-158-Bn-S-SiPhos-PE-Ph-4.00%IPA-0.5mL_min-ODH2.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-158-Bn-S-SiPhos-PE-Ph-4.00%IPA-0.5mL_min-ODH2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/5/2015 4:43:02 PM
Data Processed	: 10/5/2015 5:19:29 PM

<Chromatogram>

C:\...\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-3-158-Bn-S-SiPhos-PE-Ph-4.00%IPA-0.5mL_min-ODH2.lcd



1 PDA Multi 1/210nm 4nm

PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.624	29143532	566132	93.310	93.643
2	20.669	2089504	38434	6.690	6.357
Total		31233036	604566	100.000	100.000

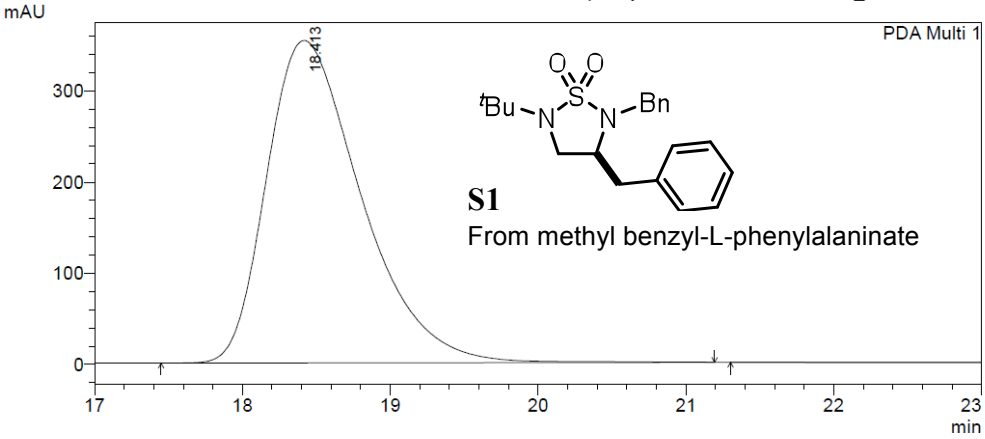
==== Shimadzu LCsolution Analysis Report ====

C:\...\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-3-130-Bn-Ph-Lphenylalanine-4.00%IPA-0.5mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-130-Bn-Ph-Lphenylalanine-4.00%IPA-0.5mL_min-ODH.lc
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-130-Bn-Ph-Lphenylalanine-4.00%IPA-0.5mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/3/2015 12:55:13 PM
Data Processed	: 9/3/2015 1:26:06 PM

<Chromatogram>

C:\...\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-3-130-Bn-Ph-Lphenylalanine-4.00%IPA-0.5mL_min-ODH.lcd



1 PDA Multi 1/210nm 4nm

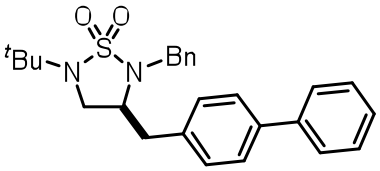
PeakTable

PDA Ch1 210nm 4nm

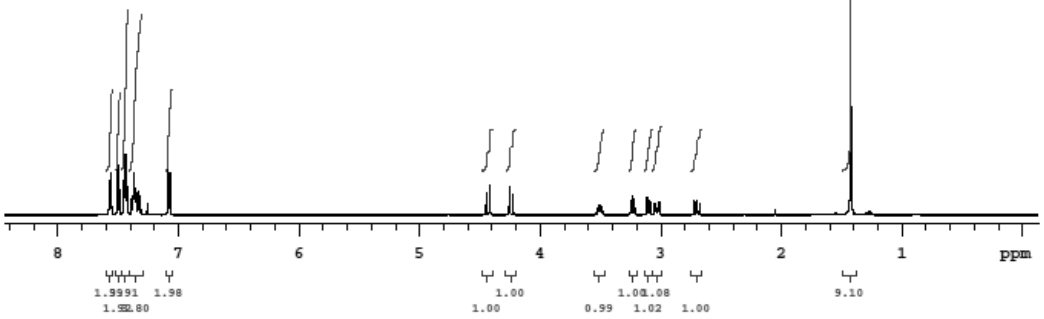
Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.413	15743426	353897	99.608	99.815
2	24.956	61927	656	0.392	0.185
Total		15805352	354552	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2015-08-23
Pulse sequence PROTON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs600



3b

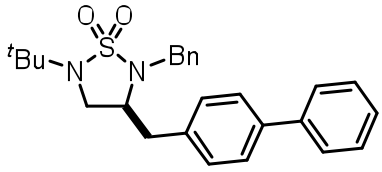


Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.150.p.1.H1.fid

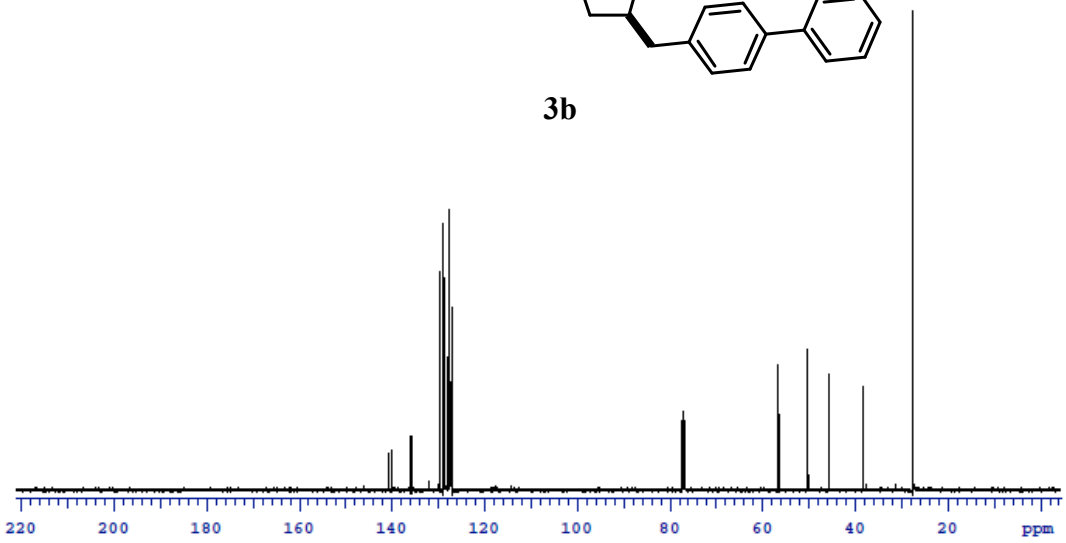
Plot date 2015-09-24

Agilent Technologies

Sample Name
Date collected 2015-08-23
Pulse sequence CARBON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs600



3b



Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.150.p.2.C13.fid

Plot date 2015-09-24

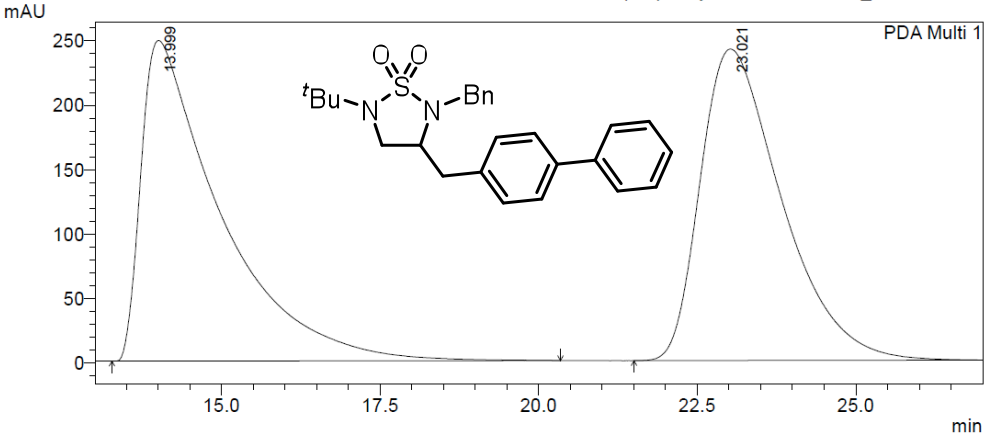
==== Shimadzu LCsolution Analysis Report ====

C:\...\Kaia Parenti\5 Membered Sulfonamides\RAC-KP-1-35-Bn-XPhos-2-p-biphenyl-7.00%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-KP-1-35-Bn-XPhos-2-p-biphenyl-7.00%IPA-1.0mL_min-ODH.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-KP-1-35-Bn-XPhos-2-p-biphenyl-7.00%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/29/2015 3:12:31 PM
Data Processed	: 6/29/2015 3:43:23 PM

<Chromatogram>

C:\...\Kaia Parenti\5 Membered Sulfonamides\RAC-KP-1-35-Bn-XPhos-2-p-biphenyl-7.00%IPA-1.0mL_min-ODH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.999	21685510	248926	50.105	50.725
2	23.021	21594352	241814	49.895	49.275
Total		43279862	490740	100.000	100.000

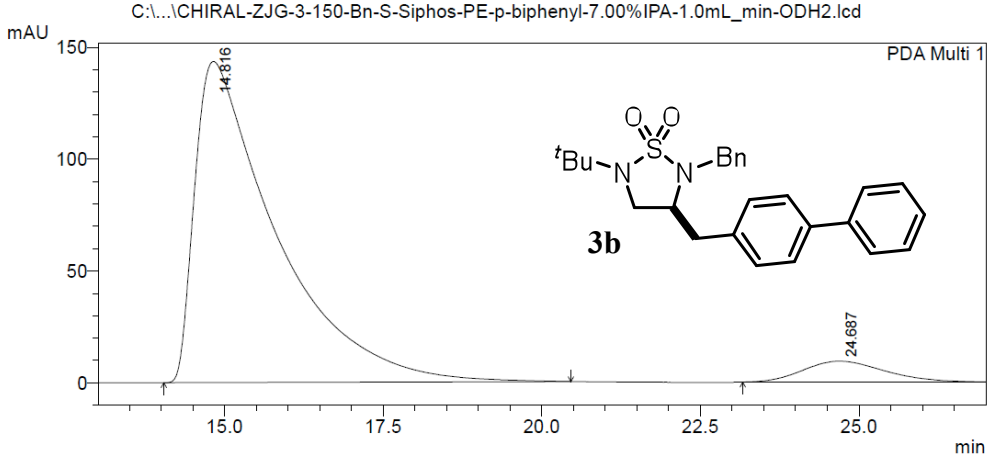
==== Shimadzu LCsolution Analysis Report ====

C:\...\CHIRAL-ZJG-3-150-Bn-S-Siphos-PE-p-biphenyl-7.00%IPA-1.0mL_min-ODH2.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-150-Bn-S-Siphos-PE-p-biphenyl-7.00%IPA-1.0mL_min-O
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-150-Bn-S-Siphos-PE-p-biphenyl-7.00%IPA-1.0mL_min-ODH2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/23/2015 3:53:48 PM
Data Processed	: 9/23/2015 5:17:00 PM

<Chromatogram>

C:\...\CHIRAL-ZJG-3-150-Bn-S-Siphos-PE-p-biphenyl-7.00%IPA-1.0mL_min-ODH2.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.816	12784169	143572	93.816	93.876
2	24.687	842740	9365	6.184	6.124
Total		13626909	152937	100.000	100.000

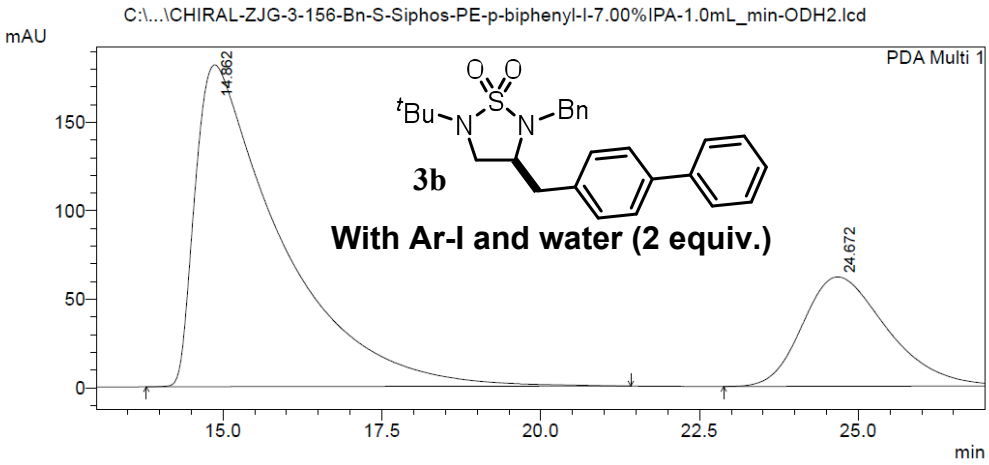
C:\LabSolutions\Data\Zac G\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-3-150-Bn-S-Siphos-PE-p-biphenyl-7.00%IPA-1.0mL_min-ODH2.lcd

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

C:\...\CHIRAL-ZJG-3-156-Bn-S-Siphos-PE-p-biphenyl-I-7.00%IPA-1.0mL_min-ODH2.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-156-Bn-S-Siphos-PE-p-biphenyl-I-7.00%IPA-1.0mL_min
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-156-Bn-S-Siphos-PE-p-biphenyl-I-7.00%IPA-1.0mL_min-ODH2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/30/2015 12:29:25 PM
Data Processed	: 9/30/2015 1:49:22 PM

<Chromatogram>



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.862	17070784	181873	74.773	74.623
2	24.672	5759373	61848	25.227	25.377
Total		22830157	243721	100.000	100.000

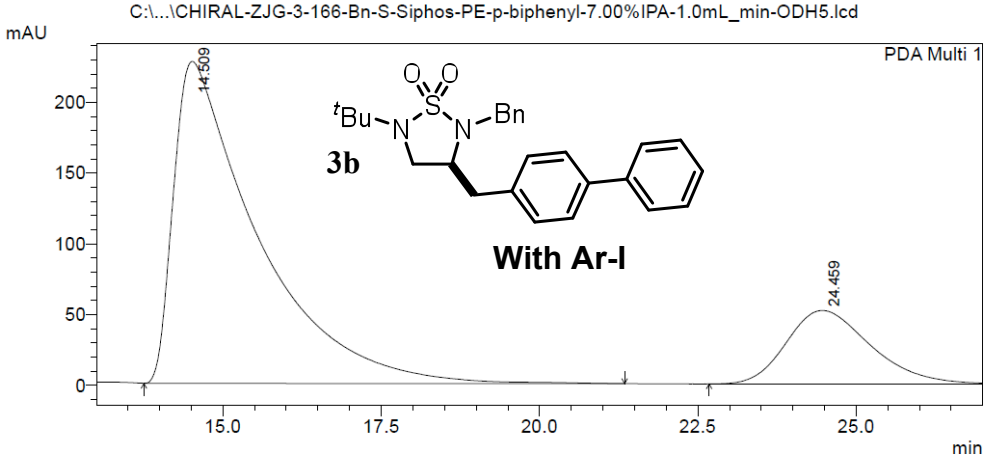
==== Shimadzu LCsolution Analysis Report ====

C:\...\CHIRAL-ZJG-3-166-Bn-S-Siphos-PE-p-biphenyl-7.00%IPA-1.0mL_min-ODH5.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-166-Bn-S-Siphos-PE-p-biphenyl-7.00%IPA-1.0mL_min-O
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-166-Bn-S-Siphos-PE-p-biphenyl-7.00%IPA-1.0mL_min-ODH5.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/7/2015 2:33:30 PM
Data Processed	: 10/7/2015 3:20:27 PM

<Chromatogram>

C:\...\CHIRAL-ZJG-3-166-Bn-S-Siphos-PE-p-biphenyl-7.00%IPA-1.0mL_min-ODH5.lcd

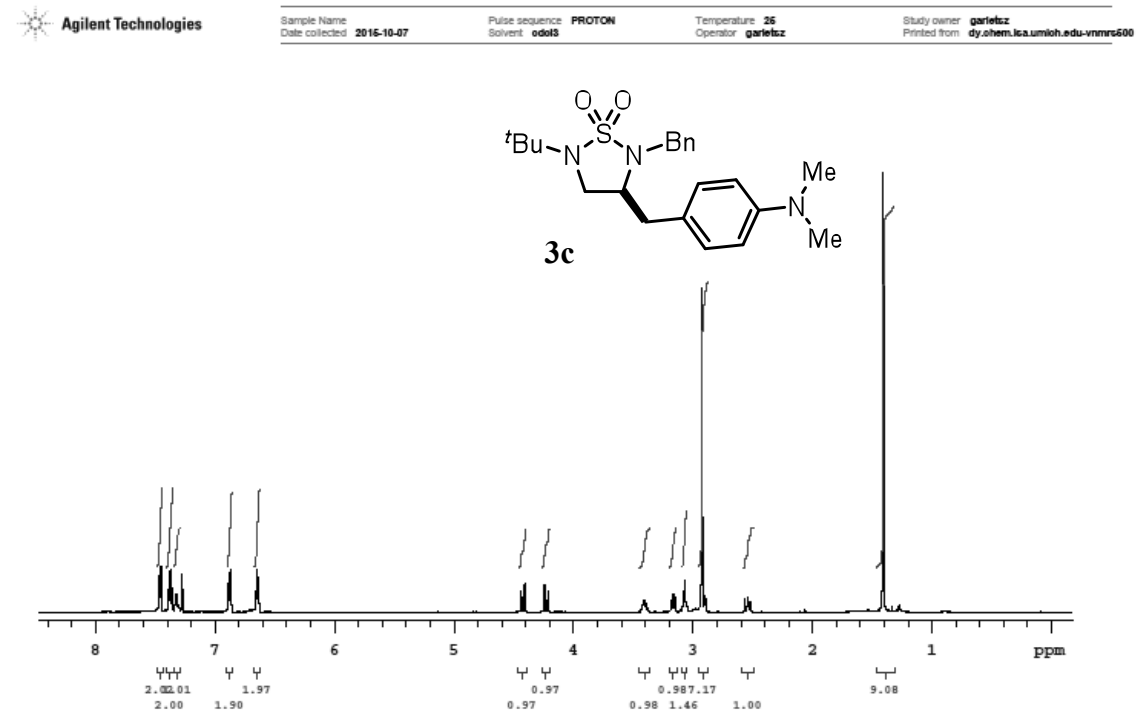


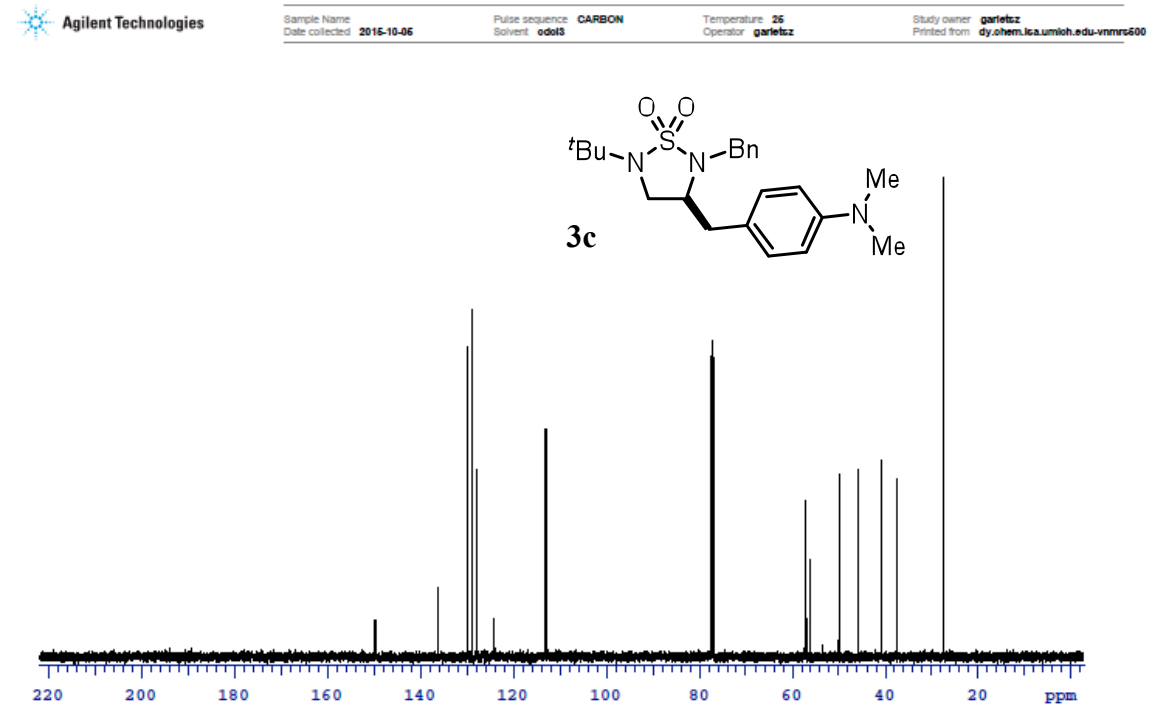
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.509	20927785	227330	81.319	81.400
2	24.459	4807481	51946	18.681	18.600
Total		25735266	279277	100.000	100.000


Agilent Technologies
Sample Name
Date collected 2016-10-07
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs500
tBu
N
S
O
O
N
Bn
Me
N
Me
3c
8
7
6
5
4
3
2
1
ppm
2.00
2.01
2.00
1.97
1.90
0.97
0.97
0.98
7.17
1.46
1.00
9.08
Data file /misc1500/garletsz/vnmrsys/data/zdq.jpw.3.167.p.1.H1.fid
Plot date 2015-10-07


Agilent Technologies
Sample Name
Date collected 2016-10-06
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs500
tBu
N
S
O
O
N
Bn
Me
N
Me
3c
220
200
180
160
140
120
100
80
60
40
20
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.139.p.2.C13.fid
Plot date 2015-10-07

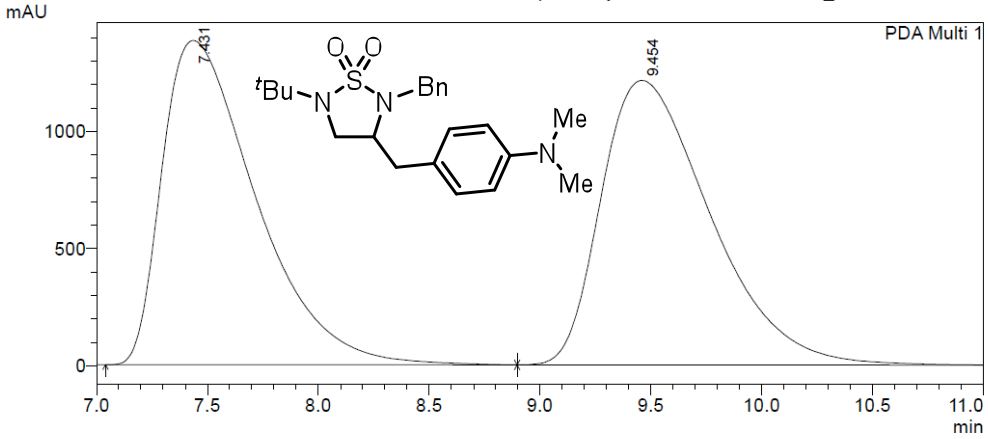
==== Shimadzu LCsolution Analysis Report ====

C:\...\Kaia Parenti\5 Membered Sulfonamides\RAC-KP-1-66-Bn-XPhos-pdimethylaniline-7.00%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-KP-1-66-Bn-XPhos-pdimethylaniline-7.00%IPA-1.0mL_min-ODH.lc
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-KP-1-66-Bn-XPhos-pdimethylaniline-7.00%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 8/8/2015 12:48:26 PM
Data Processed	: 8/8/2015 2:11:31 PM

<Chromatogram>

C:\...\5 Membered Sulfonamides\RAC-KP-1-66-Bn-XPhos-pdimethylaniline-7.00%IPA-1.0mL_min-ODH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

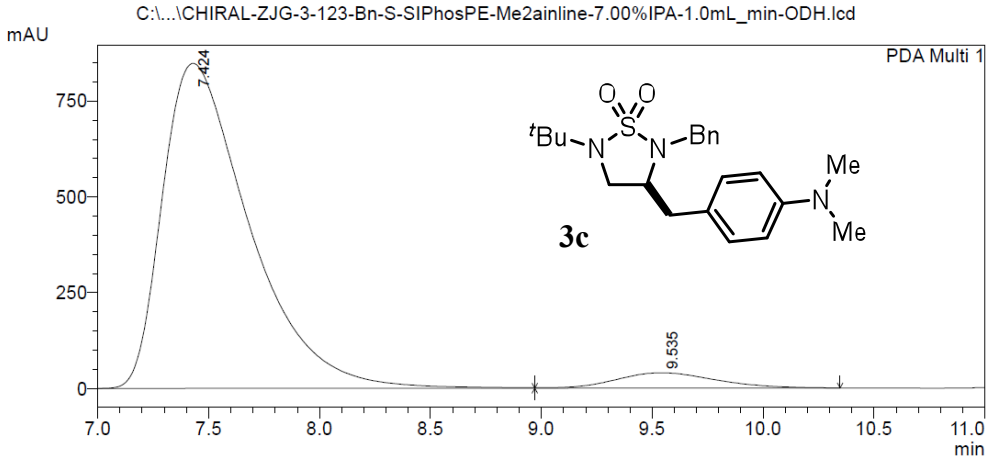
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.431	40835508	1384650	49.423	53.262
2	9.454	41789050	1215064	50.577	46.738
Total		82624558	2599714	100.000	100.000

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

C:\...\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-3-123-Bn-S-SIPhosPE-Me2ainline-7.00%IPA-1.0mL_min-ODH.lcd
Acquired by : Admin
Sample Name : CHIRAL-ZJG-3-123-Bn-S-SIPhosPE-Me2ainline-7.00%IPA-1.0mL_min-OD
Sample ID :
Tray# : 1
Vail # : 1
Injection Volume : 1 uL
Data File Name : CHIRAL-ZJG-3-123-Bn-S-SIPhosPE-Me2ainline-7.00%IPA-1.0mL_min-ODH.lcd
Method File Name : Cyclic Urea Method.lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 9/3/2015 3:53:01 PM
Data Processed : 9/3/2015 4:10:13 PM

<Chromatogram>

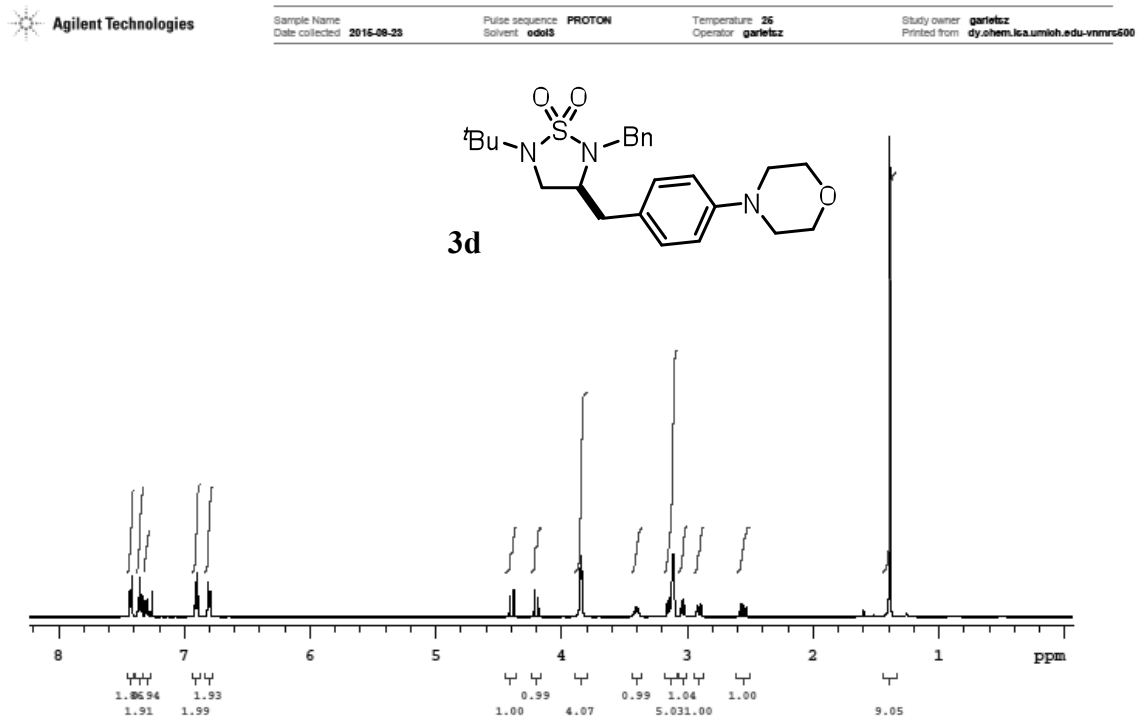


1 PDA Multi 1/254nm 4nm

PeakTable

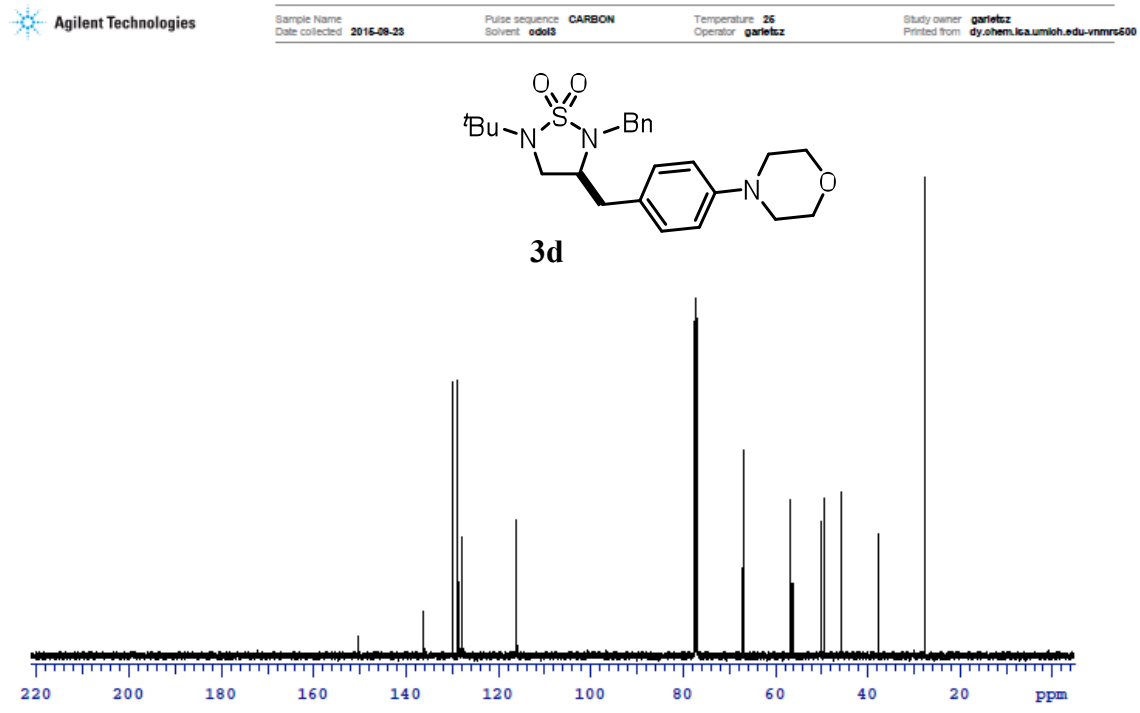
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.424	23109882	847062	94.984	95.524
2	9.535	1220371	39693	5.016	4.476
Total		24330253	886755	100.000	100.000



Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.149.p.1.H1.fid

Plot date 2015-09-24



Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.149.p.2.C13.fid

Plot date 2015-09-24

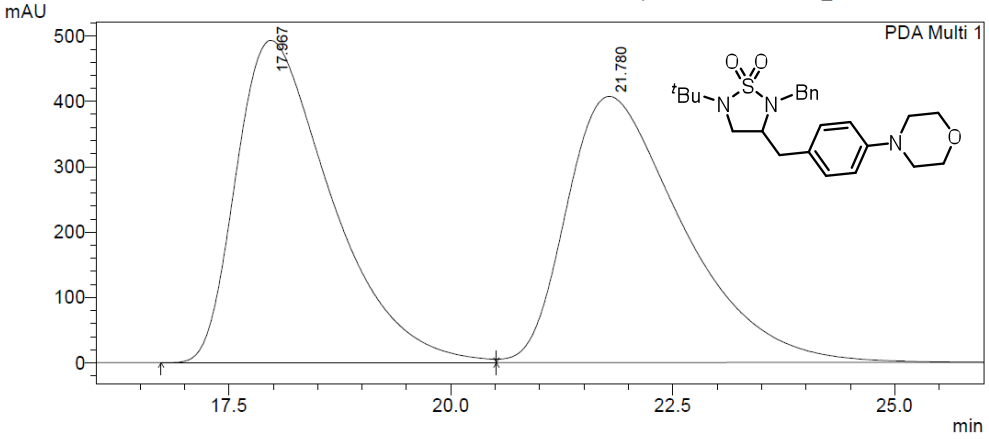
==== Shimadzu LCsolution Analysis Report ====

C:\...\Kaia Parenti\5 Membered Sulfonamides\RAC-KP-1-29-Bn-XPhos-2-benzomorpho-7.00%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-KP-1-29-Bn-XPhos-2-benzomorpho-7.00%IPA-1.0mL_min-ODH.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-KP-1-29-Bn-XPhos-2-benzomorpho-7.00%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/19/2015 9:32:41 AM
Data Processed	: 6/19/2015 10:42:44 AM

<Chromatogram>

C:\...\5 Membered Sulfonamides\RAC-KP-1-29-Bn-XPhos-2-benzomorpho-7.00%IPA-1.0mL_min-ODH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.967	36873517	492900	49.792	54.769
2	21.780	37181855	407056	50.208	45.231
Total		74055372	899956	100.000	100.000

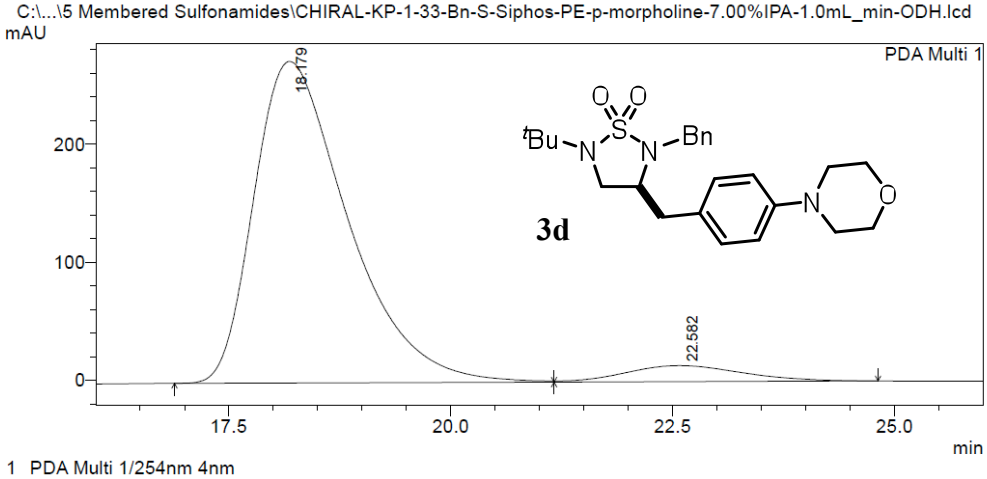
==== Shimadzu LCsolution Analysis Report ====

C:\...\5 Membered Sulfonamides\CHIRAL-KP-1-33-Bn-S-Siphos-PE-p-morpholine-7.00%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-KP-1-33-Bn-S-Siphos-PE-p-morpholine-7.00%IPA-1.0mL_min-O
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-KP-1-33-Bn-S-Siphos-PE-p-morpholine-7.00%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/3/2015 2:42:41 PM
Data Processed	: 7/3/2015 3:21:44 PM

<Chromatogram>

C:\...\5 Membered Sulfonamides\CHIRAL-KP-1-33-Bn-S-Siphos-PE-p-morpholine-7.00%IPA-1.0mL_min-ODH.lcd

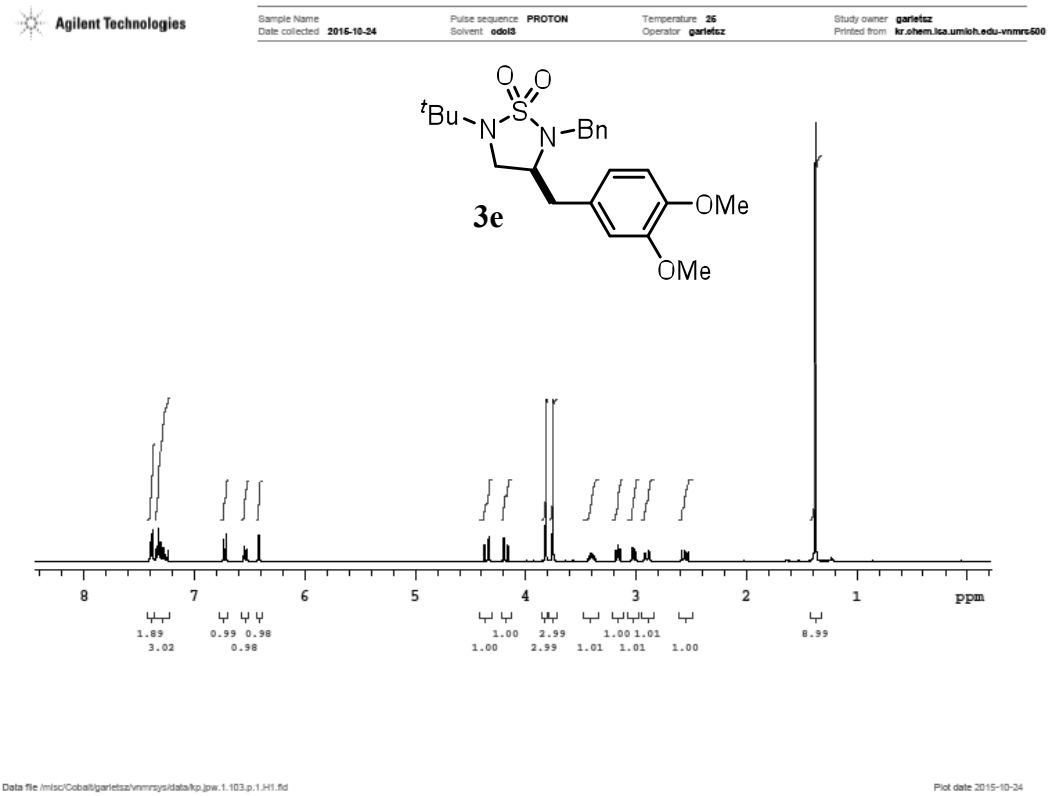


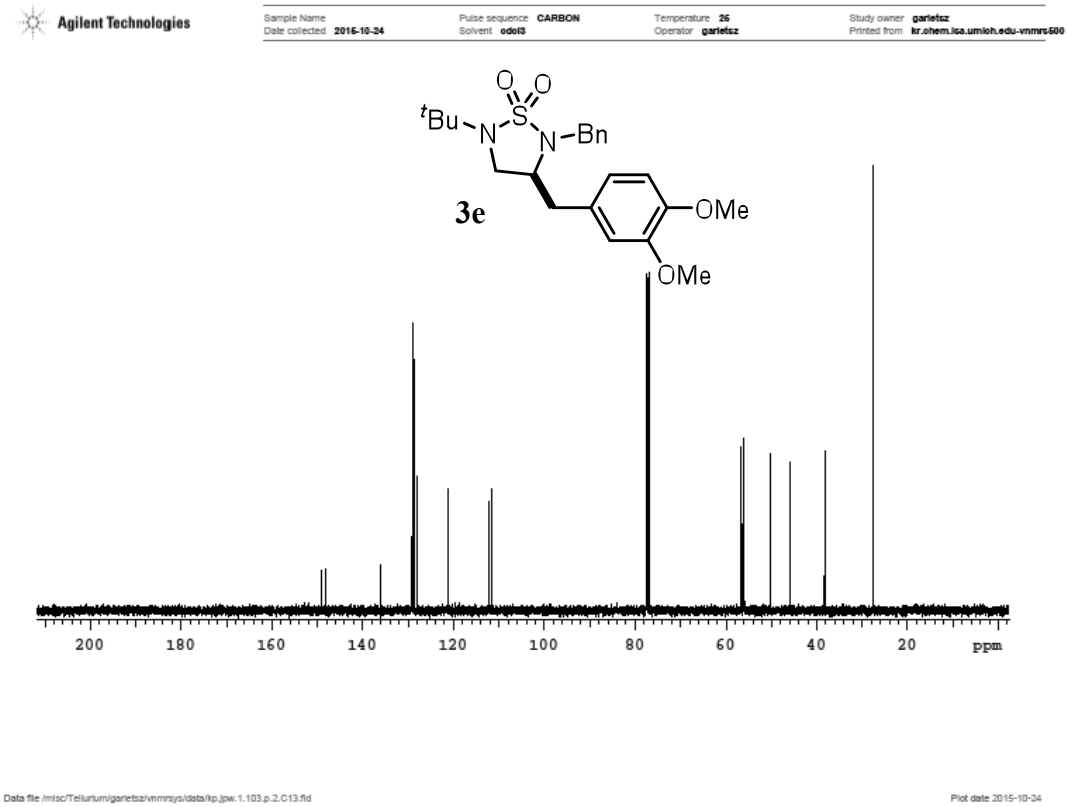
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.179	20051639	272367	94.152	95.249
2	22.582	1245532	13587	5.848	4.751
Total		21297171	285954	100.000	100.000


Agilent Technologies
Sample Name
Date collected 2016-10-24
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs500
3e
OMe
OMe
8
7
6
5
4
3
2
1
ppm
1.89
3.02
0.99
0.98
0.98
1.00
1.00
2.99
2.99
1.00
1.01
1.01
1.01
1.00
8.99
Data file /misc/Cobalt/garletsz/vnmrsys/data/kp.jpw.1.103.p.1.H1.fid
Plot date 2015-10-24


Agilent Technologies
Sample Name
Date collected 2016-10-24
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs500
3e
OMe
OMe
200
180
160
140
120
100
80
60
40
20
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.103.p.2.C13.fid
Plot date 2015-10-24

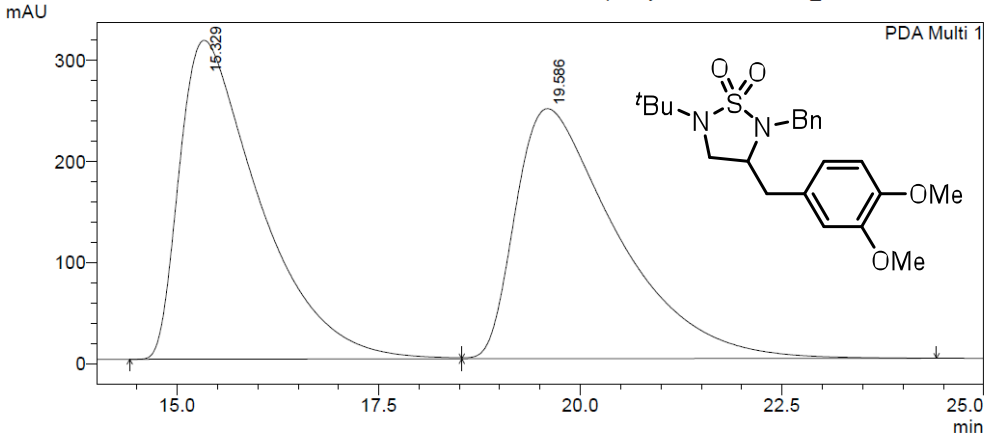
==== Shimadzu LCsolution Analysis Report ====

C:\...\Kaia Parenti\5 Membered Sulfonamides\RAC-KP-1-89-Bn-XPhos-di-o-mephenyl-7.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: RAC-KP-1-89-Bn-XPhos-di-o-mephenyl-7.00%IPA-1.0mL_min-ODH1.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-KP-1-89-Bn-XPhos-di-o-mephenyl-7.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/15/2015 4:01:15 PM
Data Processed	: 9/15/2015 4:31:46 PM

<Chromatogram>

C:\...\5 Membered Sulfonamides\RAC-KP-1-89-Bn-XPhos-di-o-mephenyl-7.00%IPA-1.0mL_min-ODH1.lcd



1 PDA Multi 1/233nm 4nm

PeakTable

PDA Ch1 233nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.329	21740650	314869	49.984	56.033
2	19.586	21754400	247063	50.016	43.967
Total		43495049	561932	100.000	100.000

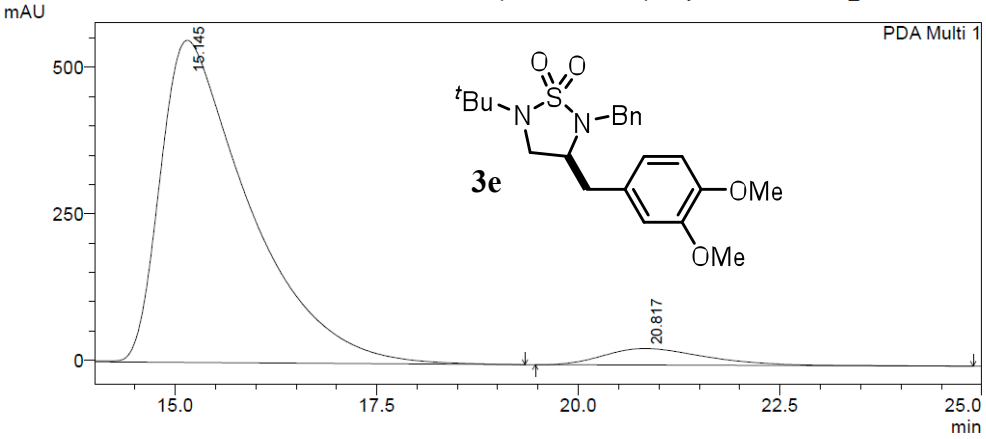
==== Shimadzu LCsolution Analysis Report ====

C:\...\5 Membered Sulfonamides\CHIRAL-KP-1-93-Bn-SiSiphos-PE-di-o-mephenyl-7.00%IPA-1.0mL_min-ODH3.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-KP-1-93-Bn-SiSiphos-PE-di-o-mephenyl-7.00%IPA-1.0mL_min-
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-KP-1-93-Bn-SiSiphos-PE-di-o-mephenyl-7.00%IPA-1.0mL_min-ODH3.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/9/2015 10:12:57 AM
Data Processed	: 10/9/2015 10:52:35 AM

<Chromatogram>

C:\...\5 Membered Sulfonamides\CHIRAL-KP-1-93-Bn-SiSiphos-PE-di-o-mephenyl-7.00%IPA-1.0mL_min-ODH3.lcd



1 PDA Multi 1/233nm 4nm

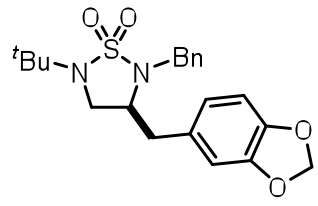
PeakTable

PDA Ch1 233nm 4nm

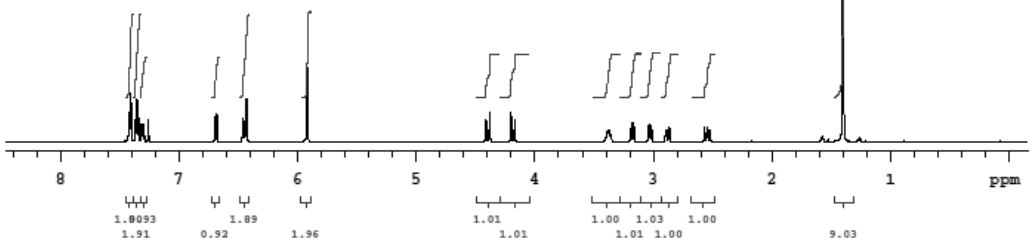
Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.145	42084475	549687	94.396	95.097
2	20.817	2498291	28341	5.604	4.903
Total		44582767	578027	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2016-10-23
Pulse sequence PROTON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs600



3f

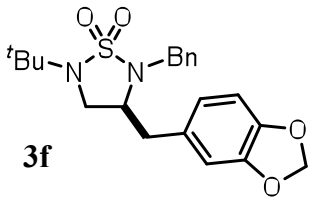


Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.97.p.1.H1.fid

Plot date 2015-10-23

Agilent Technologies

Sample Name
Date collected 2016-10-23
Pulse sequence CARBON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs600



3f

Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.97.p.2.C13.fid

Plot date 2015-10-23

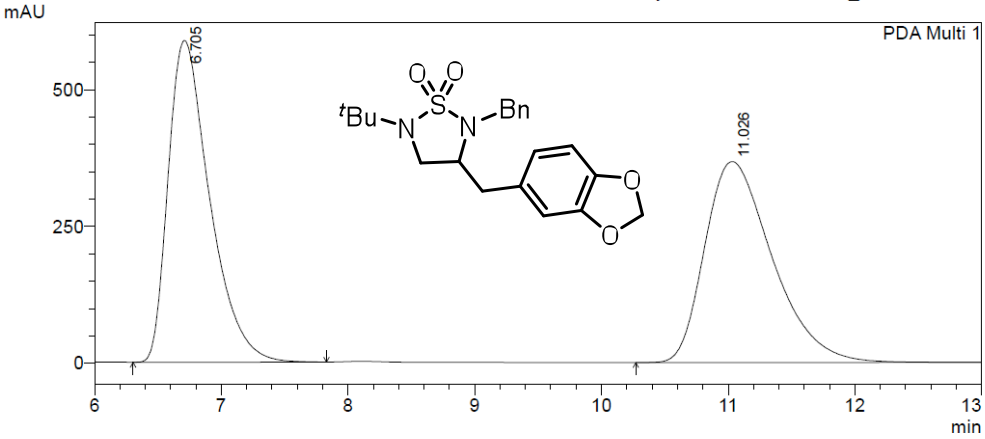
==== Shimadzu LCsolution Analysis Report ====

C:\...\Zac G\Chiral 5 Membered Sulfonamides\RAC-ZJG-3-65-1-Bn-XPhos-heterocycle-10.00%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-3-65-1-Bn-XPhos-heterocycle-10.00%IPA-1.0mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-3-65-1-Bn-XPhos-heterocycle-10.00%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/18/2015 4:42:16 PM
Data Processed	: 7/18/2015 5:01:24 PM

<Chromatogram>

C:\...\Chiral 5 Membered Sulfonamides\RAC-ZJG-3-65-1-Bn-XPhos-heterocycle-10.00%IPA-1.0mL_min-ODH.lcd



1 PDA Multi 1/280nm 4nm

PeakTable

PDA Ch1 280nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.705	13414130	588459	49.628	61.548
2	11.026	13615140	367632	50.372	38.452
Total		27029270	956092	100.000	100.000

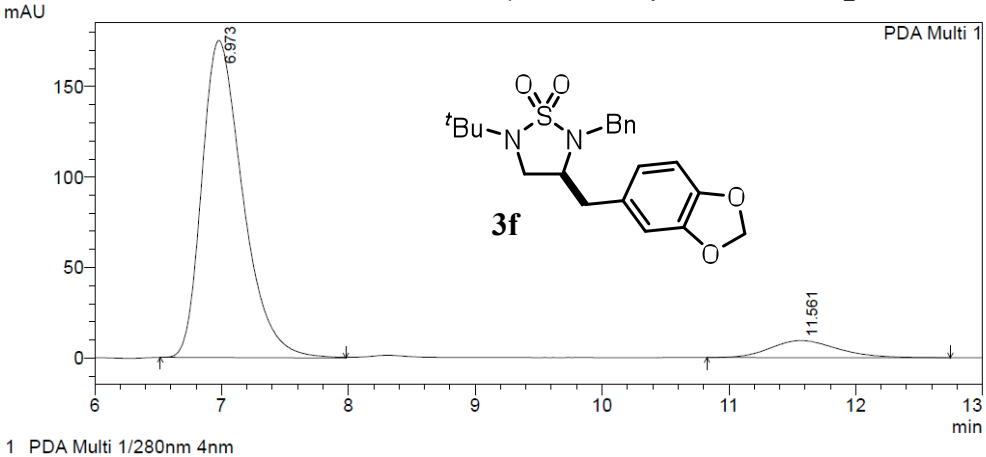
==== Shimadzu LCsolution Analysis Report ====

C:\...\5 Membered Sulfonamides\CHIRAL-KP-1-94-Bn-S-Siphos-PE-heterocycle-10.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-KP-1-94-Bn-S-Siphos-PE-heterocycle-10.00%IPA-1.0mL_min-O
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-KP-1-94-Bn-S-Siphos-PE-heterocycle-10.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/23/2015 11:23:11 AM
Data Processed	: 9/23/2015 11:38:30 AM

<Chromatogram>

C:\...\5 Membered Sulfonamides\CHIRAL-KP-1-94-Bn-S-Siphos-PE-heterocycle-10.00%IPA-1.0mL_min-ODH1.lcd

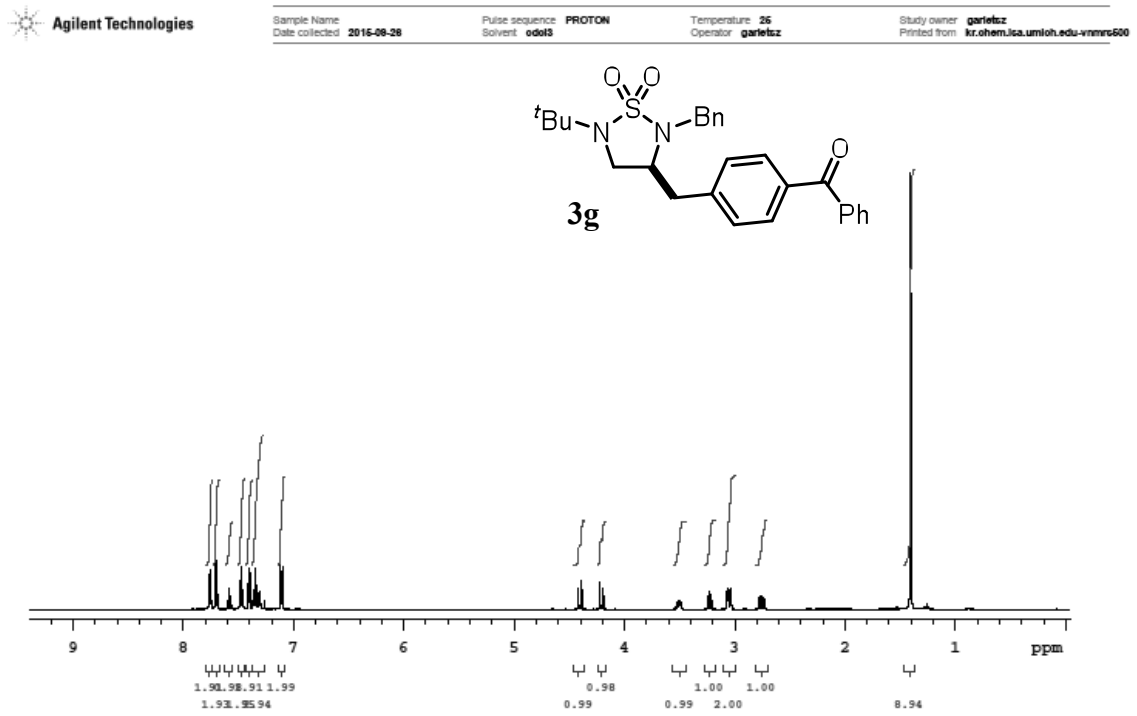


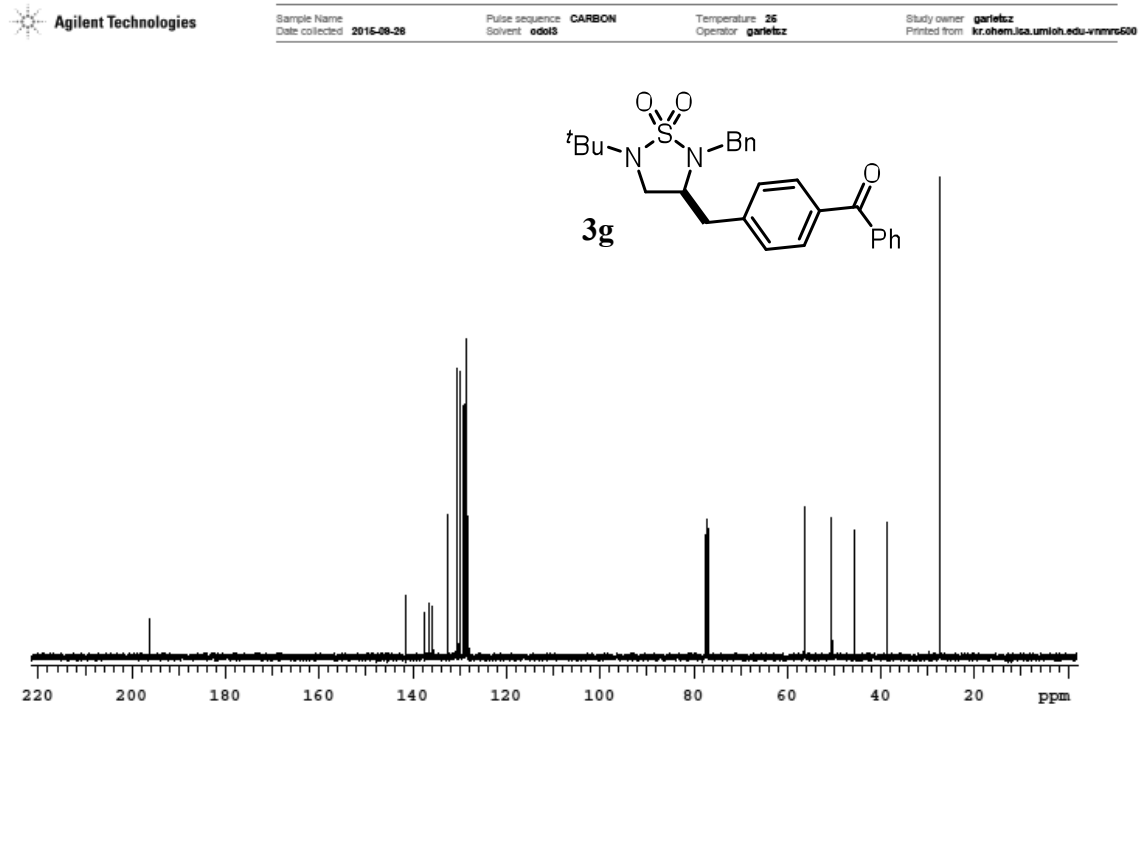
1 PDA Multi 1/280nm 4nm

PeakTable

PDA Ch1 280nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.973	3932631	175296	91.894	94.897
2	11.561	346879	9427	8.106	5.103
Total		4279510	184723	100.000	100.000


Agilent Technologies
Sample Name
Date collected 2015-09-26
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs500
3g
ppm
1.90 1.98 1.91 1.99
1.93 1.95 1.94
0.98
0.99
1.00
0.99
1.00
2.00
8.94
Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.95.p.1.H1.fid
Plot date 2015-09-26


Agilent Technologies
Sample Name
Date collected 2015-09-26
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs500
3g
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.95.p.2.C13.fid
Plot date 2015-09-26

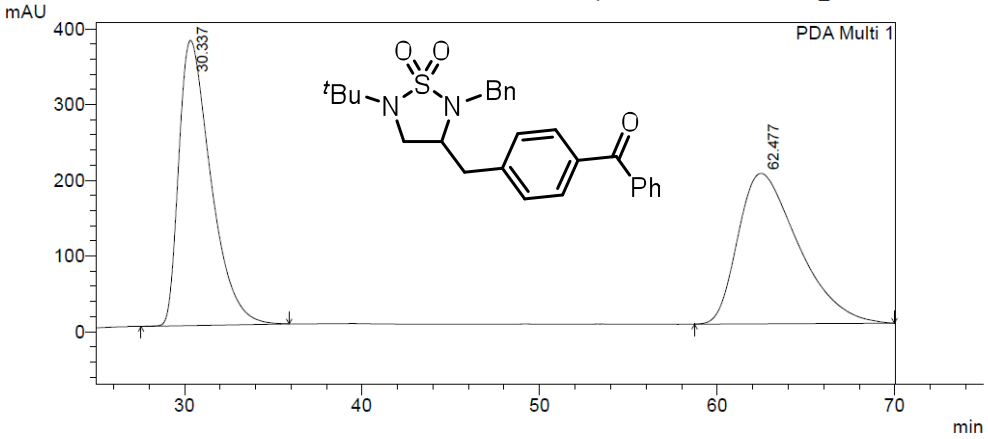
==== Shimadzu LCsolution Analysis Report ====

C:\...\Kaia Parenti\5 Membered Sulfonamides\RAC-KP-1-21-Bn-XPhos-Benzophenone-7.00%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-KP-1-21-Bn-XPhos-Benzophenone-7.00%IPA-1.0mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-KP-1-21-Bn-XPhos-Benzophenone-7.00%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/17/2015 3:10:47 PM
Data Processed	: 6/17/2015 4:20:50 PM

<Chromatogram>

C:\...\5 Membered Sulfonamides\RAC-KP-1-21-Bn-XPhos-Benzophenone-7.00%IPA-1.0mL_min-ODH.lcd



1 PDA Multi 1/210nm 4nm

PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.337	47222400	376568	50.138	65.473
2	62.477	46963048	198585	49.862	34.527
Total		94185448	575153	100.000	100.000

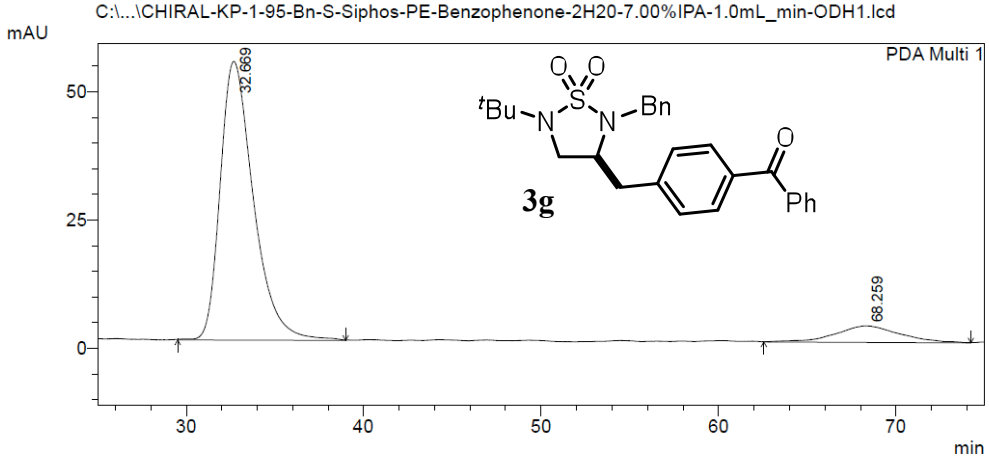
==== Shimadzu LCsolution Analysis Report ====

C:\...\CHIRAL-KP-1-95-Bn-S-Siphos-PE-Benzophenone-2H20-7.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-KP-1-92-Bn-S-Siphos-PE-p-fluoro-H2O-7.00%IPA-1.0mL_min-O
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-KP-1-95-Bn-S-Siphos-PE-Benzophenone-2H20-7.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/26/2015 8:17:38 AM
Data Processed	: 9/26/2015 9:57:42 AM

<Chromatogram>

C:\...\CHIRAL-KP-1-95-Bn-S-Siphos-PE-Benzophenone-2H20-7.00%IPA-1.0mL_min-ODH1.lcd



1 PDA Multi 1/210nm 4nm

PeakTable

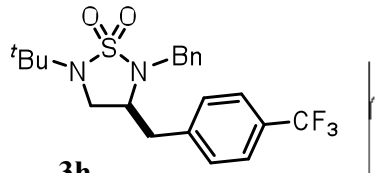
PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	32.669	7106082	54283	89.700	94.409
2	68.259	815986	3215	10.300	5.591
Total		7922068	57498	100.000	100.000

C:\LabSolutions\Data\Kaia Parenti\5 Membered Sulfonamides\CHIRAL-KP-1-95-Bn-S-Siphos-PE-Benzophenone-2H20-7.00%IPA-1.0mL_min-ODH1.lcd

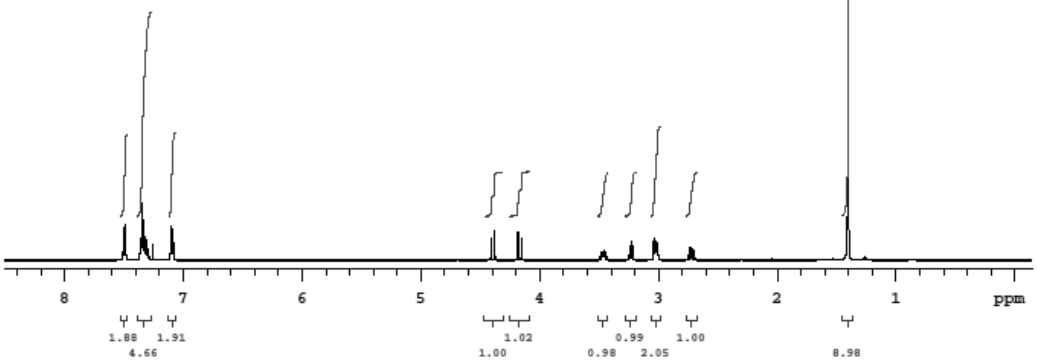
Agilent Technologies

Sample Name
Date collected 2015-10-01
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.ohem.ksa.umich.edu-vnmrs600



3h

$CDCl_3$

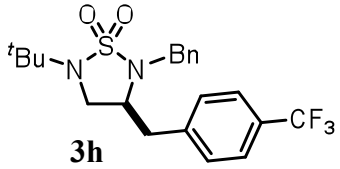


Data file /misc/Telurium/garletsz/vnmrsys/data/zjg.jpw.3.160.p.1.H1.fid

Plot date 2015-10-01

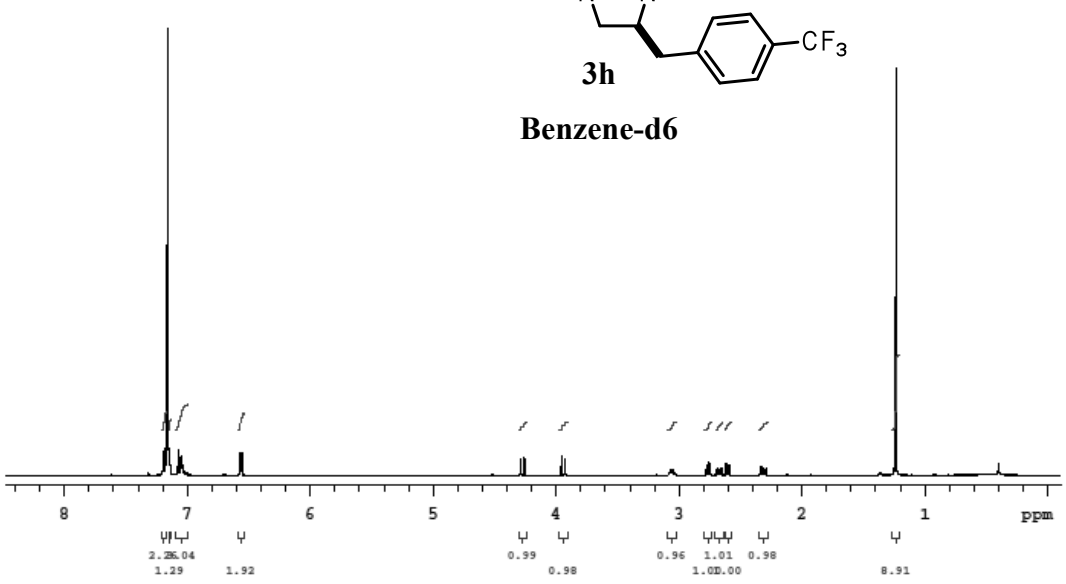
Agilent Technologies

Sample Name
Date collected 2016-10-21
Pulse sequence PROTON
Solvent o6d6
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.ohem.ksa.umich.edu-vnmrs600



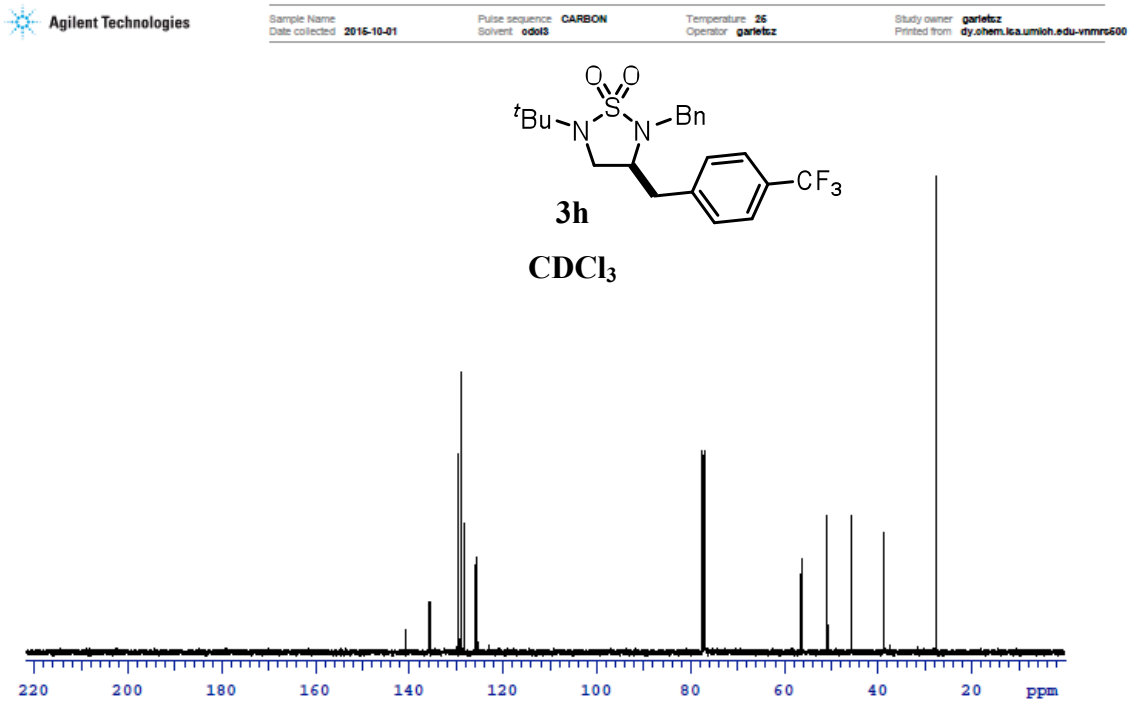
3h

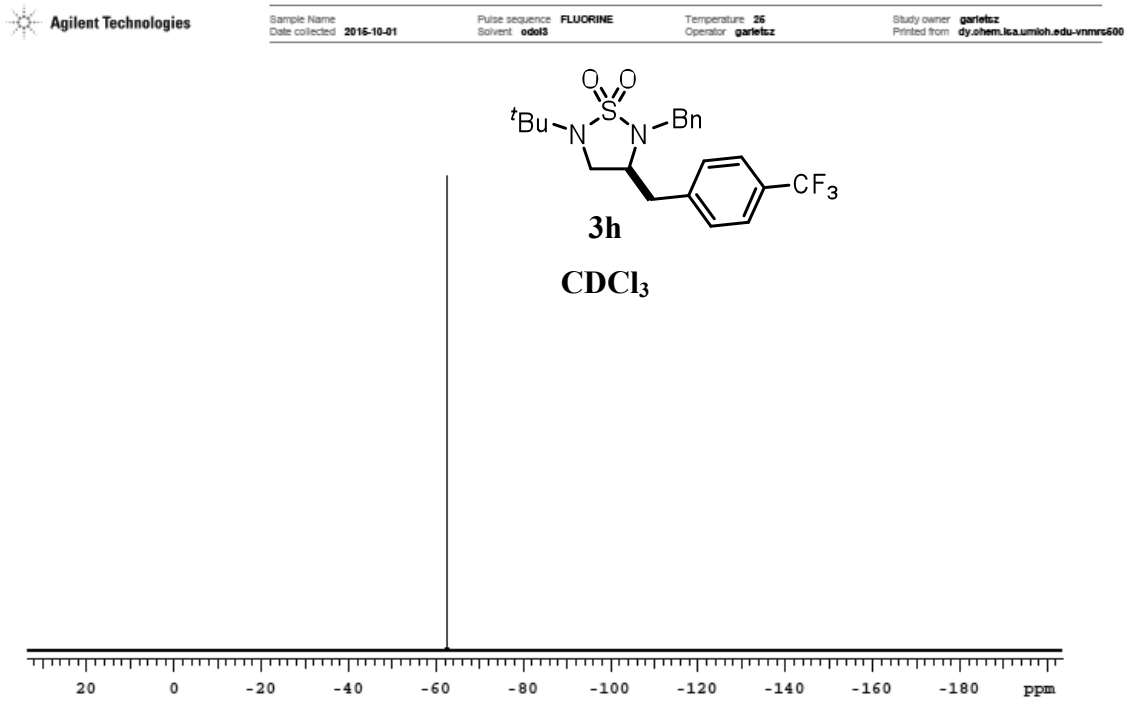
Benzene-d6



Data file /misc/Telurium/garletsz/vnmrsys/data/zjg.jpw.3.160.benzened6.fid

Plot date 2016-01-15


Agilent Technologies
Sample Name
Date collected 2015-10-01
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.ohem.lca.umich.edu-vnmrs600
tBu
N
S
O
O
N
Bn
CF3
3h
CDCl3
220
200
180
160
140
120
100
80
60
40
20
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.160.p.2.C13.fid
Plot date 2015-10-01


Agilent Technologies
Sample Name
Date collected 2015-10-01
Pulse sequence FLUORINE
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.ohem.lca.umich.edu-vnmrs600
tBu
N
S
O
O
N
Bn
CF3
3h
CDCl3
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm
Data file /misc/Cobalt/garletsz/vnmrsys/data/zjg.jpw.3.160.p.3.F19.fid
Plot date 2015-10-01

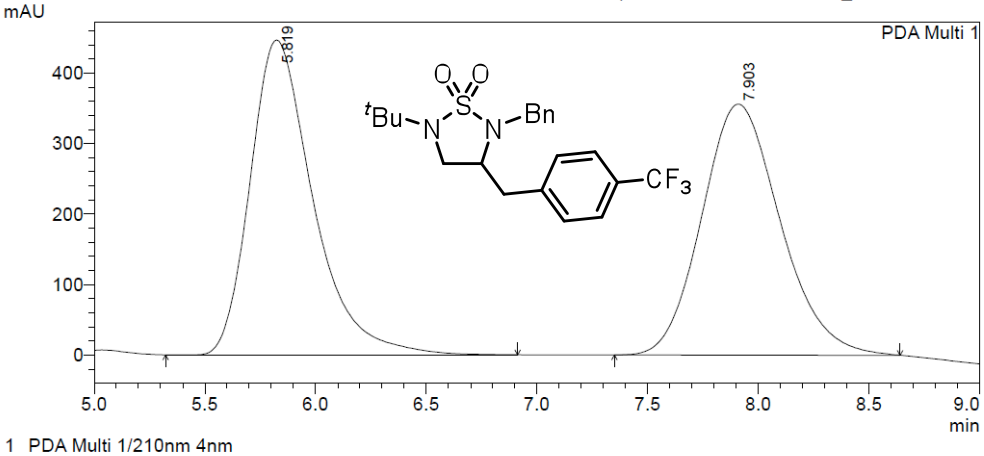
==== Shimadzu LCsolution Analysis Report ====

C:\...\Chiral 5 Membered Sulfonamides\RAC-ZJG-2-174-1-Bn-XPhos-Phptrifluoro-7.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-2-174-1-Bn-XPhos-Phptrifluoro-7.00%IPA-1.0mL_min-ODH1.l
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-2-174-1-Bn-XPhos-Phptrifluoro-7.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/24/2015 9:37:51 AM
Data Processed	: 10/24/2015 9:56:51 AM

<Chromatogram>

C:\...\Chiral 5 Membered Sulfonamides\RAC-ZJG-2-174-1-Bn-XPhos-Phptrifluoro-7.00%IPA-1.0mL_min-ODH1.lcd



1 PDA Multi 1/210nm 4nm

PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.819	8833032	446674	50.130	55.660
2	7.903	8787356	355836	49.870	44.340
Total		17620388	802510	100.000	100.000

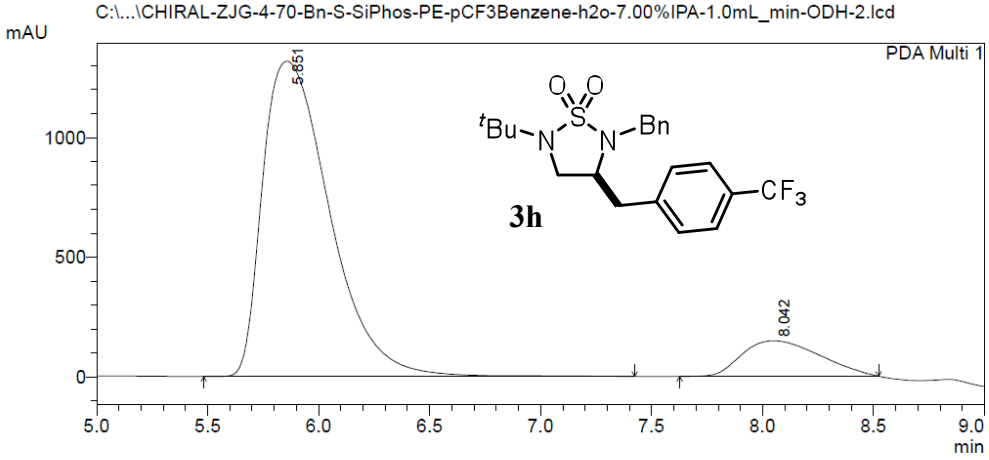
==== Shimadzu LCsolution Analysis Report ====

C:\...\CHIRAL-ZJG-4-70-Bn-S-SiPhos-PE-pCF3Benzene-h2o-7.00%IPA-1.0mL_min-ODH-2.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-4-70-Bn-S-SiPhos-PE-pCF3Benzene-h2o-7.00%IPA-1.0mL_m
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-4-70-Bn-S-SiPhos-PE-pCF3Benzene-h2o-7.00%IPA-1.0mL_min-ODH-2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/16/2016 3:31:57 PM
Data Processed	: 1/16/2016 3:46:42 PM

<Chromatogram>

C:\...\CHIRAL-ZJG-4-70-Bn-S-SiPhos-PE-pCF3Benzene-h2o-7.00%IPA-1.0mL_min-ODH-2.lcd

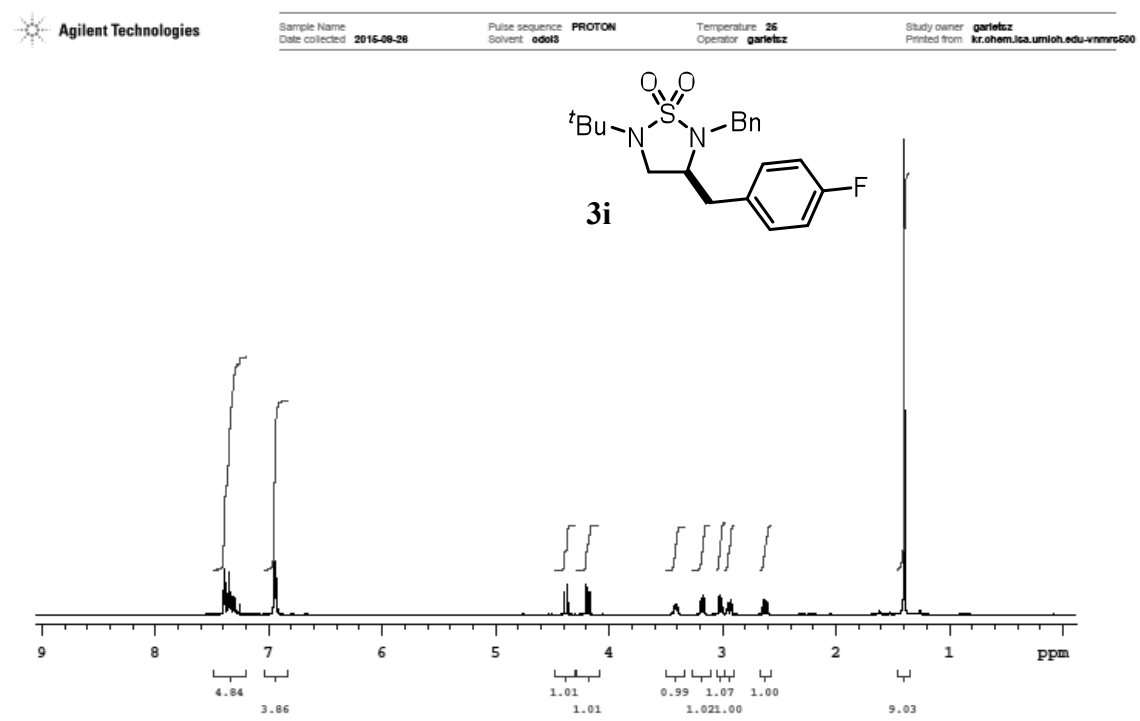


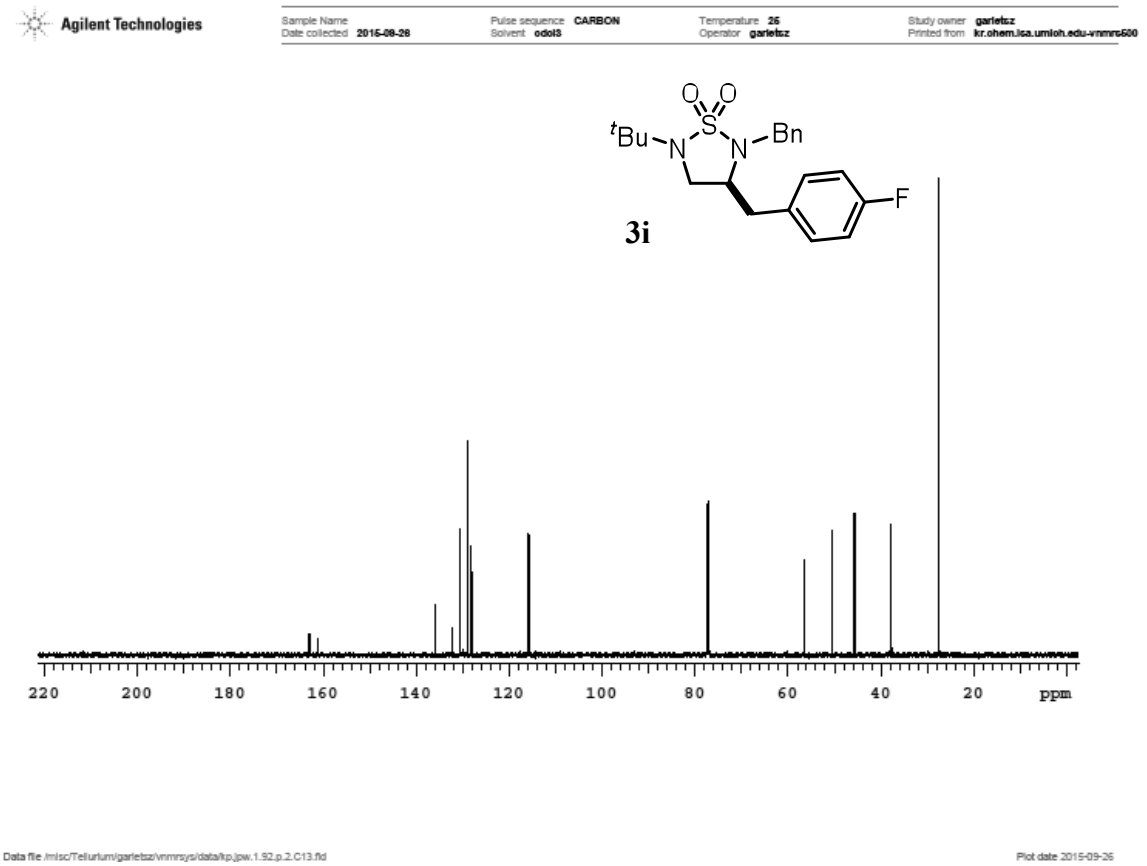
1 PDA Multi 1/210nm 4nm

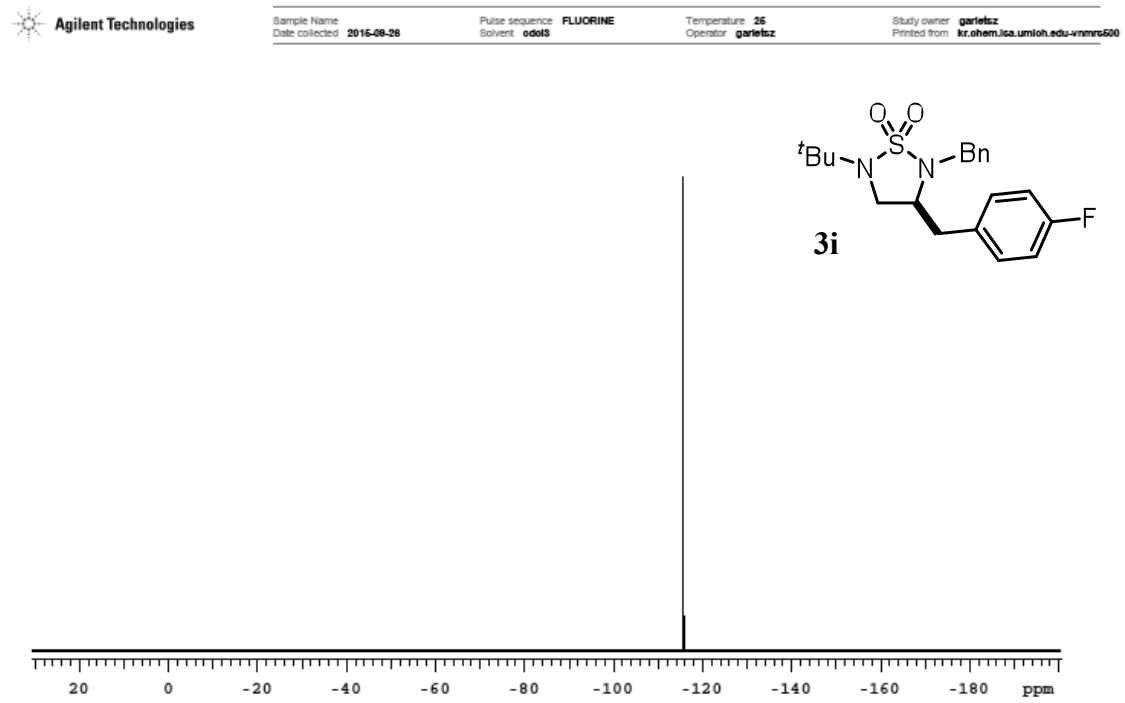
PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.851	27700472	1318235	88.334	89.855
2	8.042	3658444	148829	11.666	10.145
Total		31358916	1467064	100.000	100.000


Agilent Technologies
Sample Name
Date collected 2015-09-26
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs600
3i
9
8
7
6
5
4
3
2
1
ppm
4.84
3.86
1.01
1.01
0.99
1.07
1.00
1.02
1.00
9.03
Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.92.p.1.H1.fid
Plot date 2015-09-26


Agilent Technologies
Sample Name
Date collected 2015-09-26
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs600
3i
220
200
180
160
140
120
100
80
60
40
20
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.92.p.2.C13.fid
Plot date 2015-09-26


Agilent Technologies
Sample Name
Date collected 2015-09-26
Pulse sequence FLUORINE
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs500
tBu
N
S
O
O
N
Bn
F
3i
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.92.p.3.F19.fid
Plot date 2015-09-26

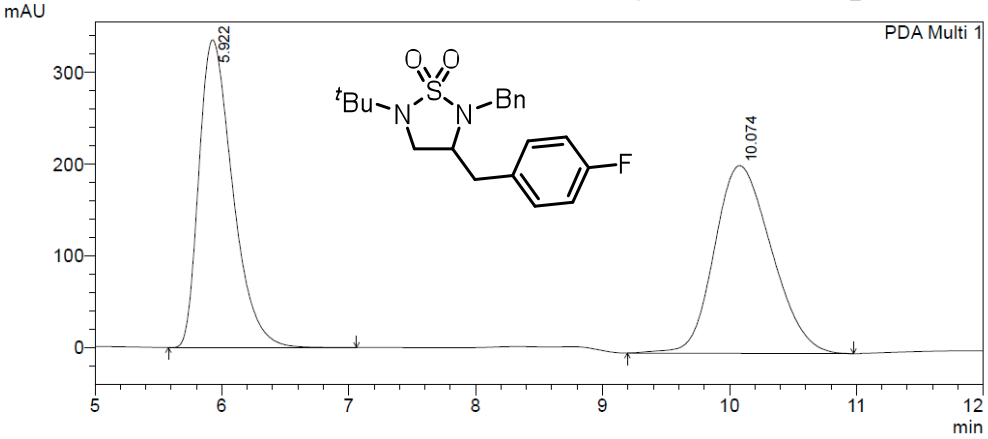
==== Shimadzu LCsolution Analysis Report ====

C:\...\Data\Kaia Parenti\5 Membered Sulfonamides\RAC-KP-1-37-Bn-XPhos-2-p-fluoro-7.00%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-KP-1-37-Bn-XPhos-2-p-fluoro-7.00%IPA-1.0mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-KP-1-37-Bn-XPhos-2-p-fluoro-7.00%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/30/2015 3:33:54 PM
Data Processed	: 6/30/2015 4:09:57 PM

<Chromatogram>

C:\...\Kaia Parenti\5 Membered Sulfonamides\RAC-KP-1-37-Bn-XPhos-2-p-fluoro-7.00%IPA-1.0mL_min-ODH.lcd



1 PDA Multi 1/220nm 4nm

PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.922	6193945	335253	49.174	62.119
2	10.074	6401944	204438	50.826	37.881
Total		12595889	539691	100.000	100.000

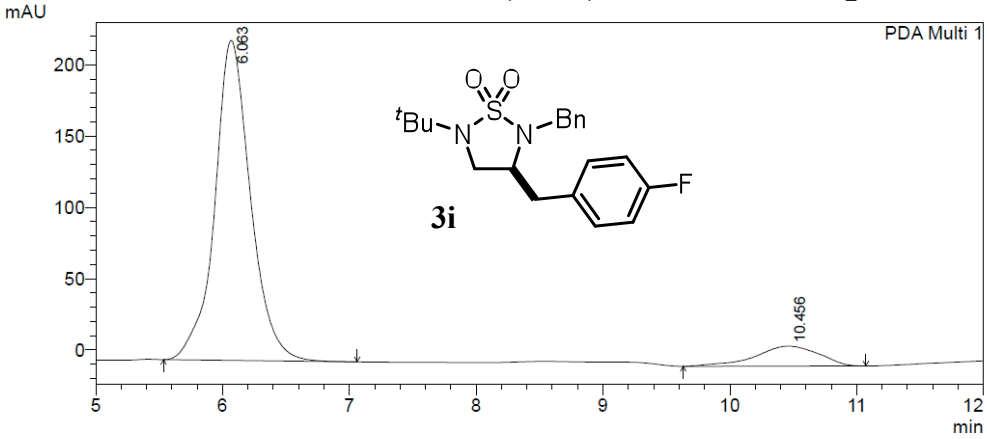
==== Shimadzu LCsolution Analysis Report ====

C:\...\5 Membered Sulfonamides\CHIRAL-KP-1-92-Bn-S-Siphos-PE-p-fluoro-H2O-7.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-KP-1-92-Bn-S-Siphos-PE-p-fluoro-H2O-7.00%IPA-1.0mL_min-O
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-KP-1-92-Bn-S-Siphos-PE-p-fluoro-H2O-7.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/26/2015 7:48:29 AM
Data Processed	: 9/26/2015 8:13:28 AM

<Chromatogram>

C:\...\5 Membered Sulfonamides\CHIRAL-KP-1-92-Bn-S-Siphos-PE-p-fluoro-H2O-7.00%IPA-1.0mL_min-ODH1.lcd

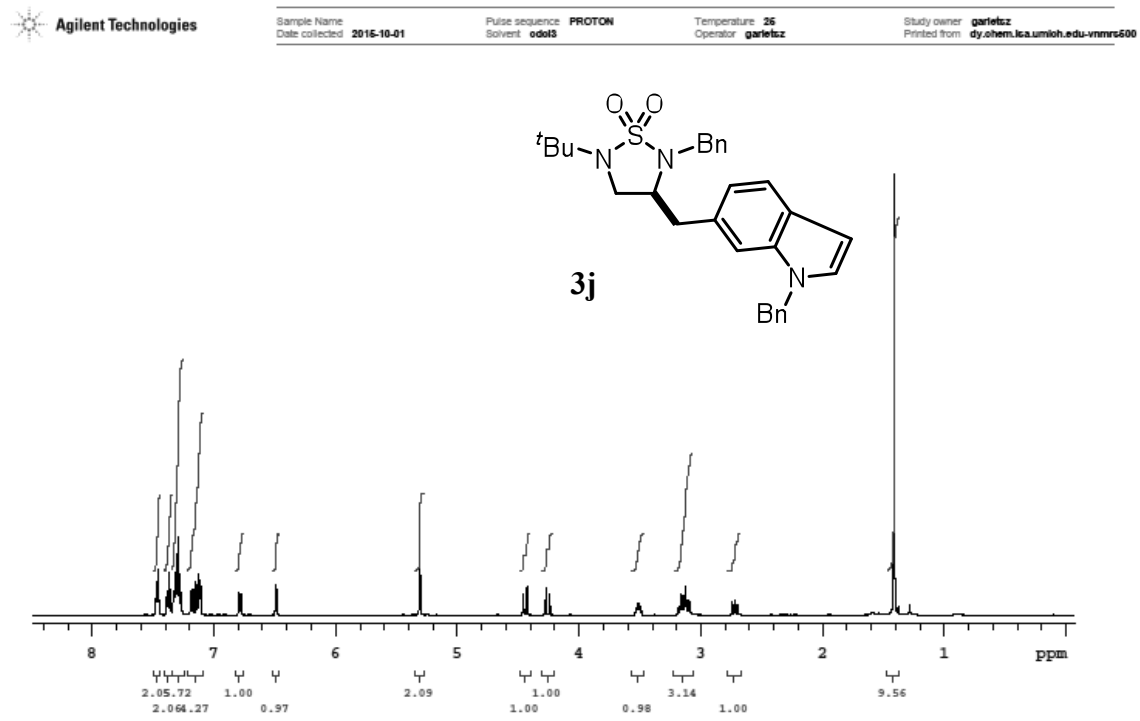


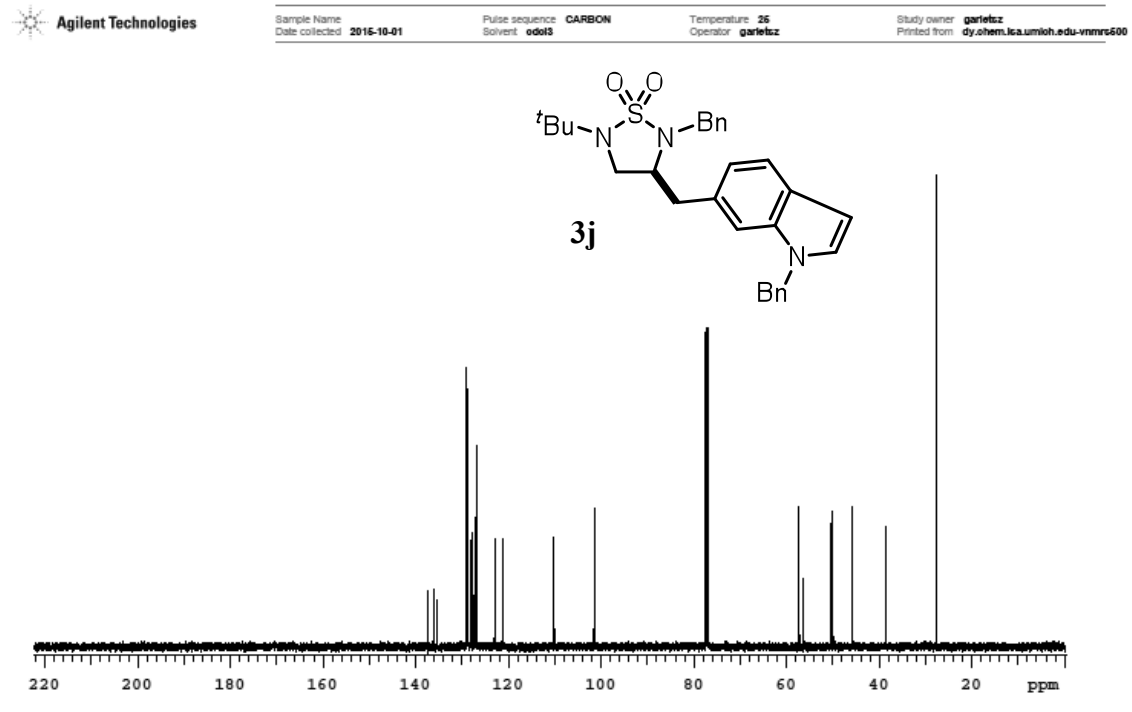
1 PDA Multi 1/220nm 4nm

PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.063	4407032	224393	89.994	94.126
2	10.456	489995	14004	10.006	5.874
Total		4897026	238397	100.000	100.000


Agilent Technologies
Sample Name
Date collected 2015-10-01
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs500
3j
8
7
6
5
4
3
2
1
ppm
2.05
5.72
1.00
2.09
1.00
3.14
9.56
2.06
4.27
0.97
1.00
0.98
1.00
Data file /misc/500/garletsz/vnmrsys/data/zjg.jpw.3.161.p.1.H1.fid
Plot date 2015-10-01


Agilent Technologies
Sample Name
Date collected 2015-10-01
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs500
3j
220
200
180
160
140
120
100
80
60
40
20
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.161.p.2.C13.fid
Plot date 2015-10-01

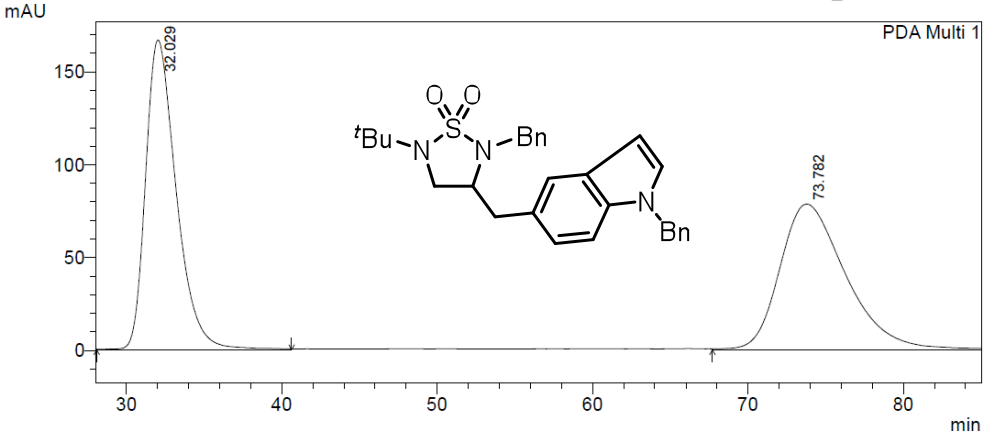
==== Shimadzu LCsolution Analysis Report ====

C:\...\Zac G\Chiral 5 Membered Sulfonamides\RAC-ZJG-3-61-1-Bn-XPhos-BnIndole-10.00%IPA-1.0mL_min-ODH4.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-3-61-1-Bn-XPhos-BnIndole-10.00%IPA-1.0mL_min-ODH4.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-3-61-1-Bn-XPhos-BnIndole-10.00%IPA-1.0mL_min-ODH4.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/5/2015 11:04:13 AM
Data Processed	: 10/5/2015 12:37:33 PM

<Chromatogram>

C:\...\Chiral 5 Membered Sulfonamides\RAC-ZJG-3-61-1-Bn-XPhos-BnIndole-10.00%IPA-1.0mL_min-ODH4.lcd



1 PDA Multi 1/220nm 4nm

PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	32.029	22940331	166749	49.755	68.014
2	73.782	23166710	78421	50.245	31.986
Total		46107041	245170	100.000	100.000

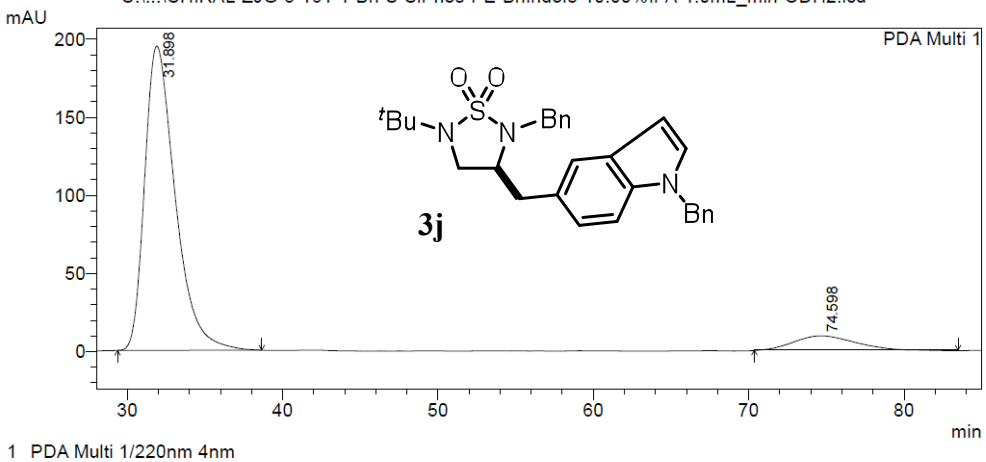
==== Shimadzu LCsolution Analysis Report ====

C:\...\CHIRAL-ZJG-3-161-1-Bn-S-SiPhos-PE-BnIndole-10.00%IPA-1.0mL_min-ODH2.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-161-1-Bn-S-SiPhos-PE-BnIndole-10.00%IPA-1.0mL_min-
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-161-1-Bn-S-SiPhos-PE-BnIndole-10.00%IPA-1.0mL_min-ODH2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/5/2015 12:47:23 PM
Data Processed	: 10/5/2015 2:26:58 PM

<Chromatogram>

C:\...\CHIRAL-ZJG-3-161-1-Bn-S-SiPhos-PE-BnIndole-10.00%IPA-1.0mL_min-ODH2.lcd

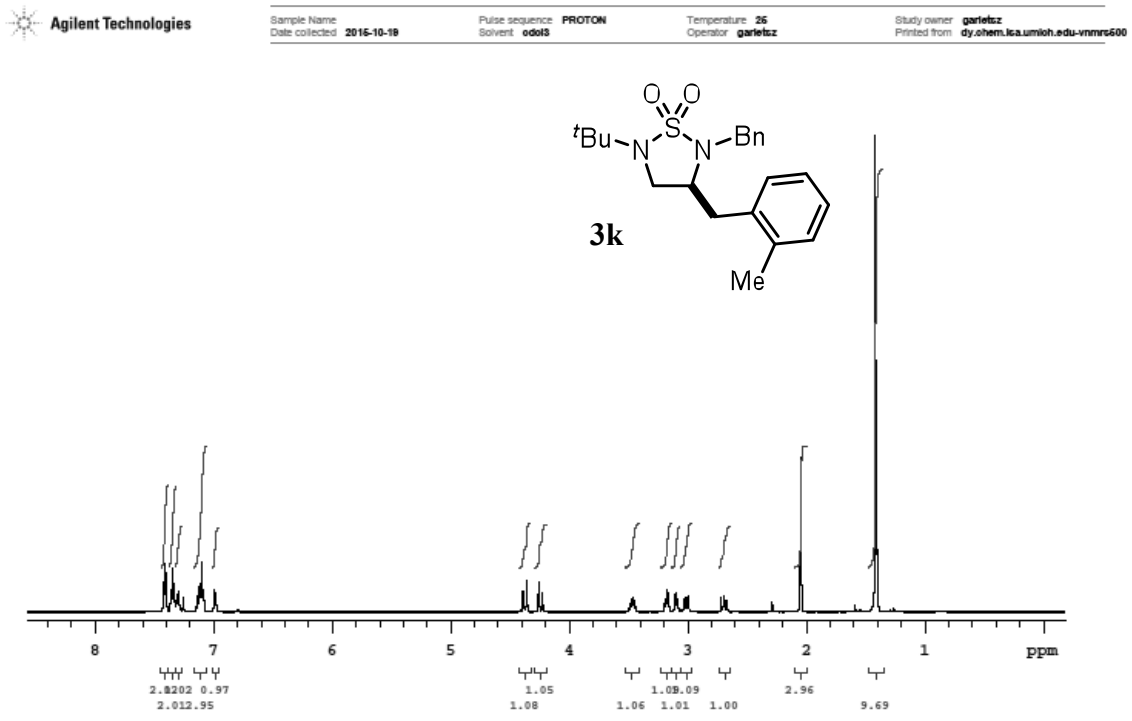


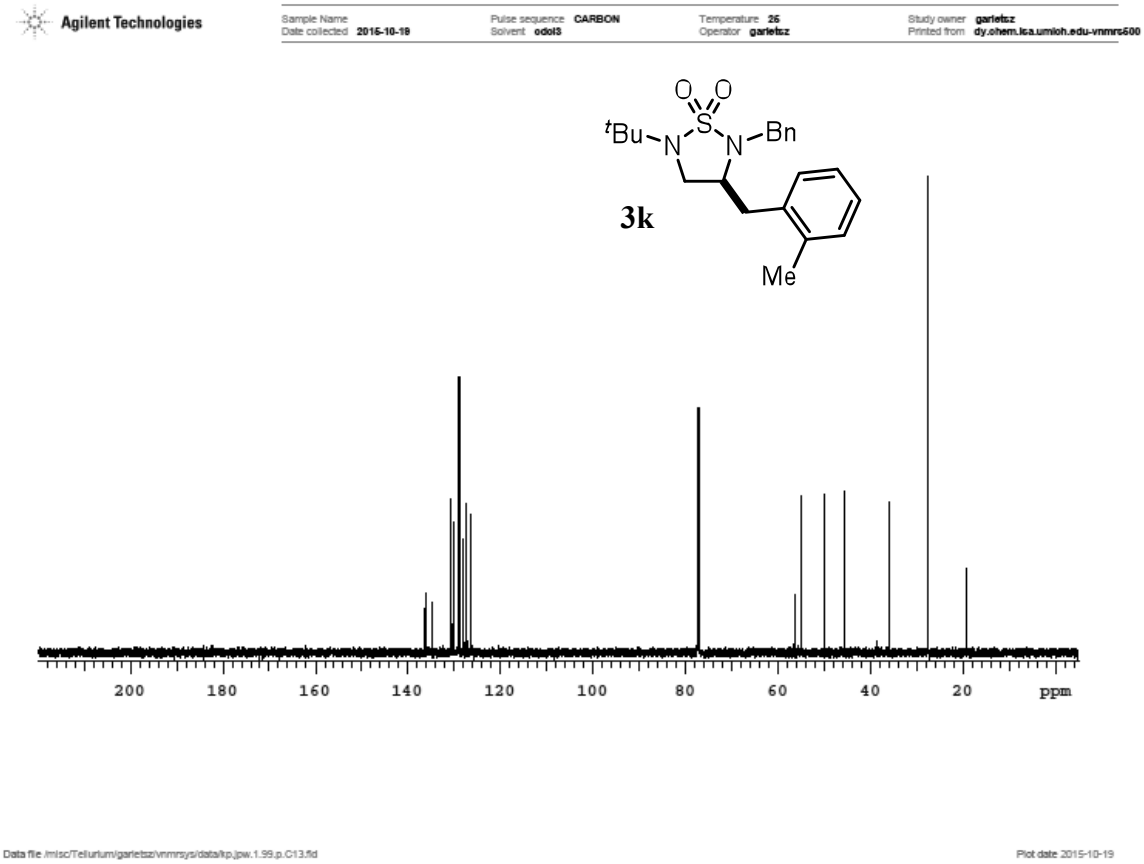
1 PDA Multi 1/220nm 4nm

PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	31.898	26668329	194906	91.672	95.554
2	74.598	2422658	9069	8.328	4.446
Total		29090986	203975	100.000	100.000


Agilent Technologies
Sample Name
Date collected 2016-10-19
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs500
3k
8
7
6
5
4
3
2
1
ppm
2.12
0.02
0.97
2.01
2.95
1.05
1.08
1.06
1.09
1.01
1.09
1.00
2.96
9.69
Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.99.p.1.H1.fid
Plot date 2015-10-19


Agilent Technologies
Sample Name
Date collected 2016-10-19
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs500
3k
200
180
160
140
120
100
80
60
40
20
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.99.p.C13.fid
Plot date 2015-10-19

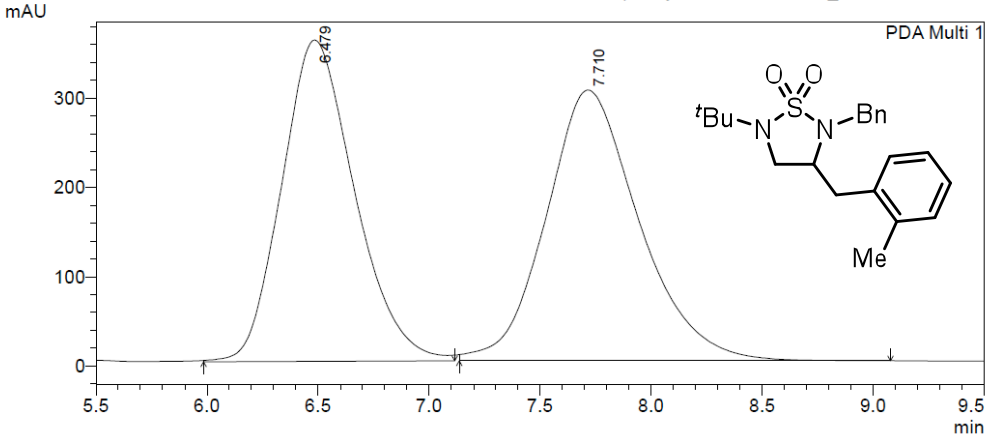
==== Shimadzu LCsolution Analysis Report ====

C:\...\Zac G\Chiral 5 Membered Sulfonamides\RAC-KP-1-76-Bn-XPhos-o-mephenyl-7.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: RAC-KP-1-76-Bn-XPhos-o-mephenyl-7.00%IPA-1.0mL_min-ODH1.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-KP-1-76-Bn-XPhos-o-mephenyl-7.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/3/2015 12:49:03 PM
Data Processed	: 11/3/2015 1:02:30 PM

<Chromatogram>

C:\...\Chiral 5 Membered Sulfonamides\RAC-KP-1-76-Bn-XPhos-o-mephenyl-7.00%IPA-1.0mL_min-ODH1.lcd



1 PDA Multi 1/225nm 4nm

PeakTable

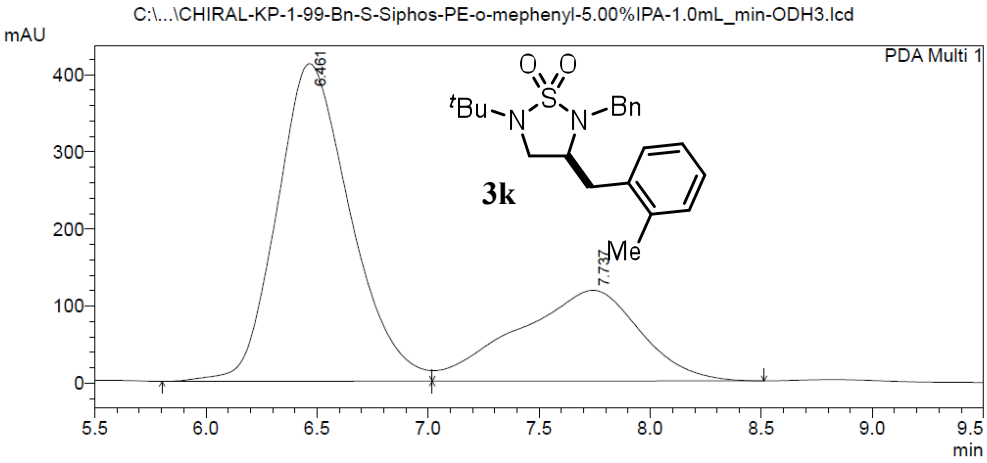
PDA Ch1 225nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.479	8235707	360393	48.168	54.268
2	7.710	8862105	303710	51.832	45.732
Total		17097812	664103	100.000	100.000

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

C:\...\Chiral 5 Membered Sulfonamides\CHIRAL-KP-1-99-Bn-S-Siphos-PE-o-mephenyl-5.00%IPA-1.0mL_min-ODH3.lcd
Acquired by : Admin
Sample Name : CHIRAL-KP-1-99-Bn-S-Siphos-PE-o-mephenyl-5.00%IPA-1.0mL_min-ODH
Sample ID :
Tray# : 1
Vail # : 1
Injection Volume : 1 uL
Data File Name : CHIRAL-KP-1-99-Bn-S-Siphos-PE-o-mephenyl-5.00%IPA-1.0mL_min-ODH3.lcd
Method File Name : Cyclic Urea Method.lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 11/3/2015 1:04:47 PM
Data Processed : 11/3/2015 1:22:54 PM

<Chromatogram>

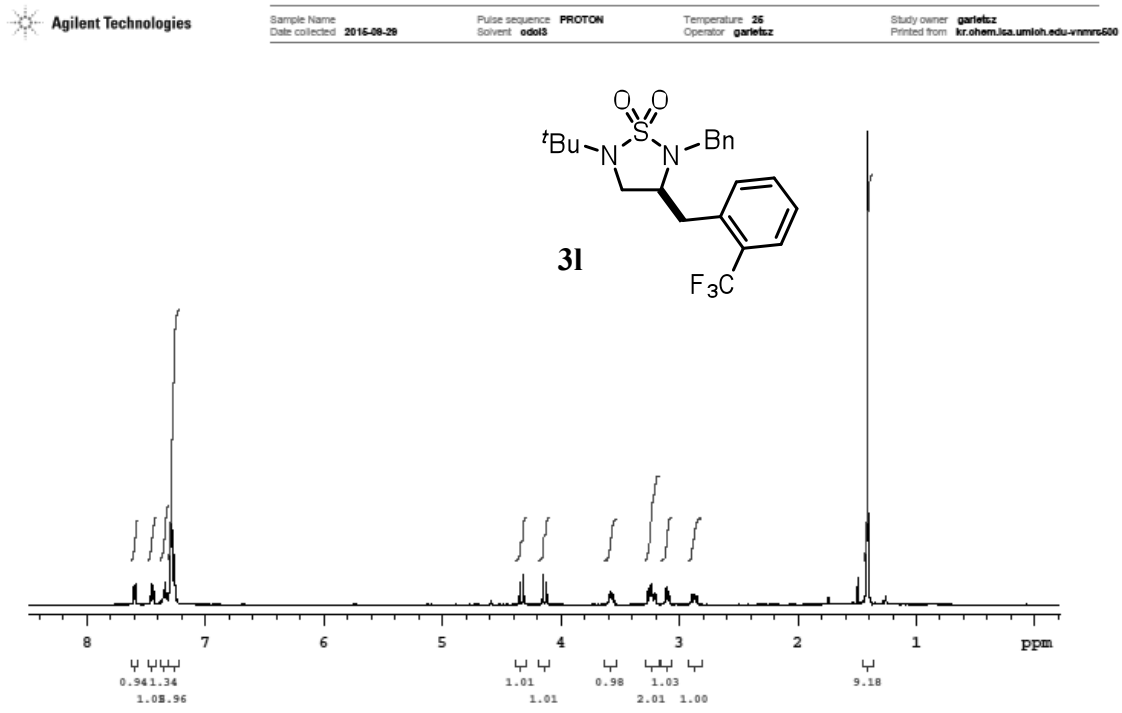


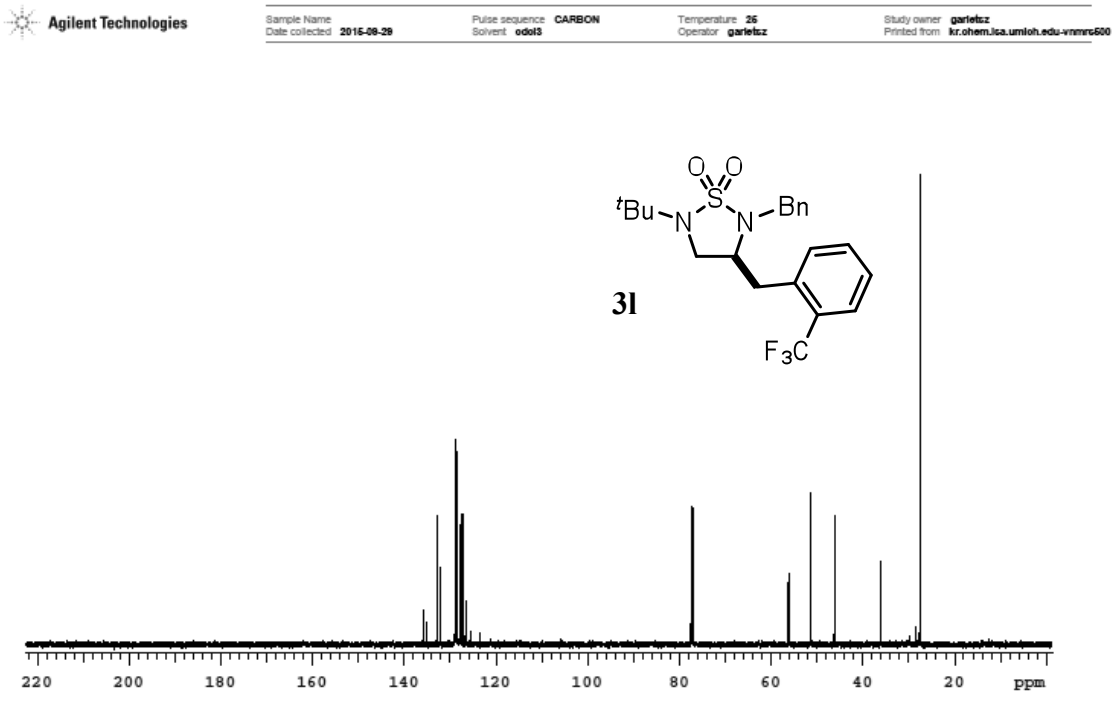
1 PDA Multi 1/225nm 4nm

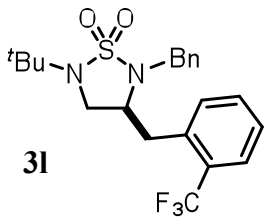
PeakTable

PDA Ch1 225nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.461	9467670	411735	67.666	77.785
2	7.737	4524157	117592	32.334	22.215
Total		13991827	529326	100.000	100.000


Agilent Technologies
Sample Name
Date collected 2015-08-29
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs600
O
O
S
tBu
N
N
Bn
F3C
3l
8
7
6
5
4
3
2
1
ppm
0.94
1.34
1.05
3.96
1.01
1.01
0.98
1.03
2.01
1.00
9.18
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.153.p.1.H1.fid
Plot date 2015-09-29


Agilent Technologies
Sample Name
Date collected 2015-08-29
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs600
O
O
S
tBu
N
N
Bn
F3C
3l
220
200
180
160
140
120
100
80
60
40
20
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.153.p.2.C13.fid
Plot date 2015-09-29



3l

Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.153.p.3.F19.fid

Plot date 2015-09-29

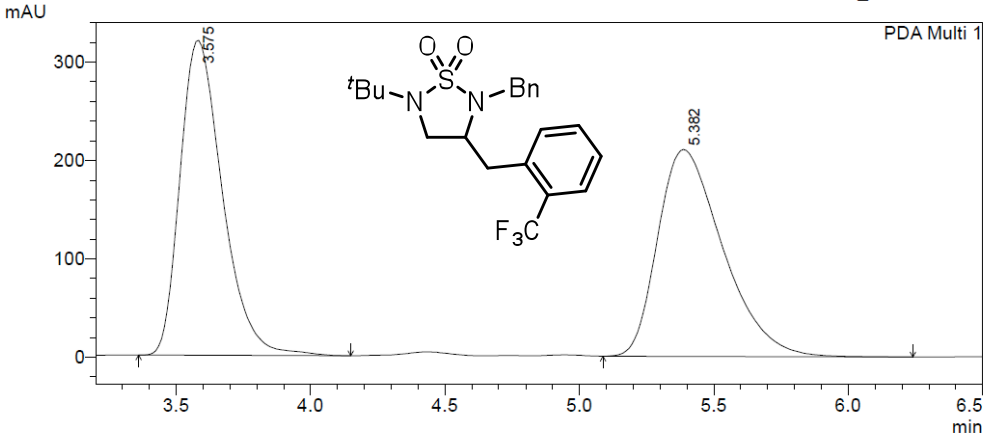
==== Shimadzu LCsolution Analysis Report ====

C:\...\Kaia Parenti\5 Membered Sulfonamides\RAC-KP-1-44-Bn-XPhos-2-o-trifluoro-7.00%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-KP-1-44-Bn-XPhos-2-o-trifluoro-7.00%IPA-1.0mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-KP-1-44-Bn-XPhos-2-o-trifluoro-7.00%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/22/2015 3:23:38 PM
Data Processed	: 7/22/2015 3:37:31 PM

<Chromatogram>

C:\...\Kaia Parenti\5 Membered Sulfonamides\RAC-KP-1-44-Bn-XPhos-2-o-trifluoro-7.00%IPA-1.0mL_min-ODH.lcd



1 PDA Multi 1/230nm 4nm

PeakTable

PDA Ch1 230nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	3.575	3519979	320240	49.825	60.351
2	5.382	3544721	210386	50.175	39.649
Total		7064700	530625	100.000	100.000

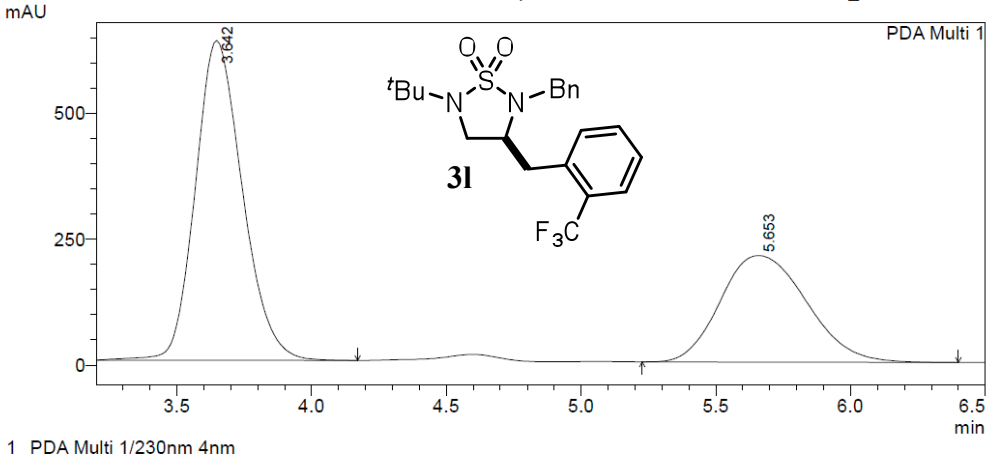
==== Shimadzu LCsolution Analysis Report ====

C:\...\5 Membered Sulfonamides\CHIRAL-KP-1-105-Bn-S-Siphos-PE-o-trifluorotol-7.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-KP-1-105-Bn-S-Siphos-PE-o-trifluorotol-7.00%IPA-1.0mL_mi
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-KP-1-105-Bn-S-Siphos-PE-o-trifluorotol-7.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/30/2015 10:44:59 AM
Data Processed	: 10/30/2015 10:56:16 AM

<Chromatogram>

C:\...\5 Membered Sulfonamides\CHIRAL-KP-1-105-Bn-S-Siphos-PE-o-trifluorotol-7.00%IPA-1.0mL_min-ODH1.lcd

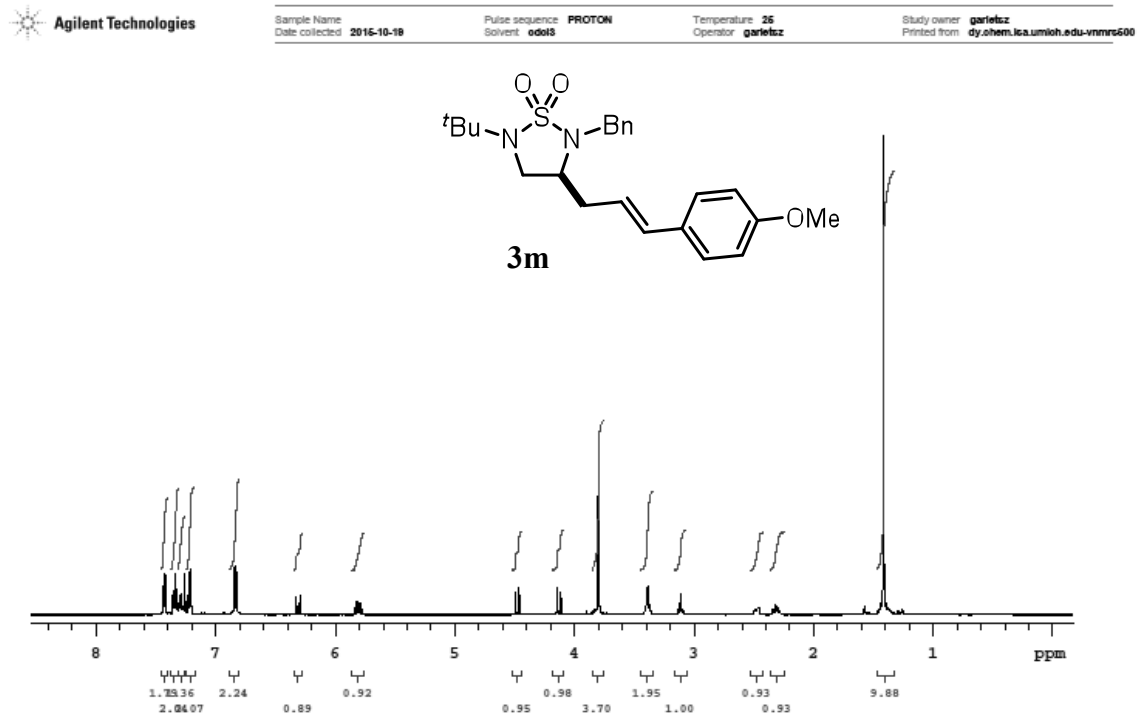


1 PDA Multi 1/230nm 4nm

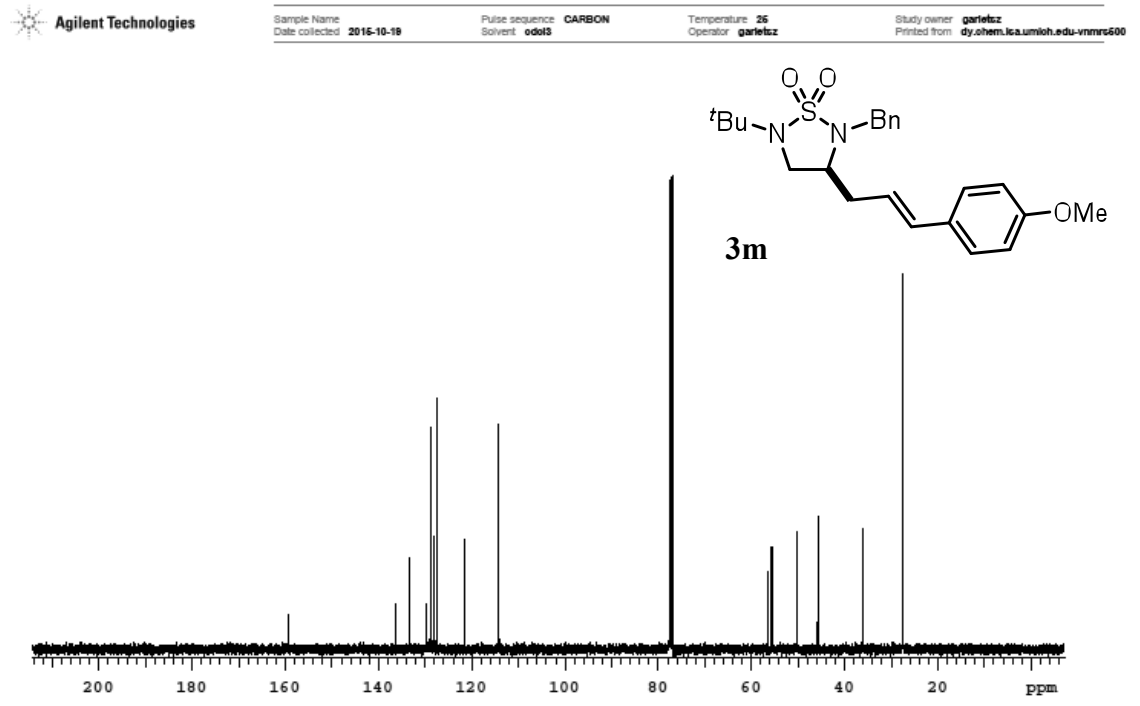
PeakTable

PDA Ch1 230nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	3.642	7729676	634952	62.125	75.019
2	5.653	4712453	211442	37.875	24.981
Total		12442129	846393	100.000	100.000



Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.155.p.1.H1.fid Plot date 2015-10-19



Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.155.p.2.C13.fid Plot date 2015-10-19

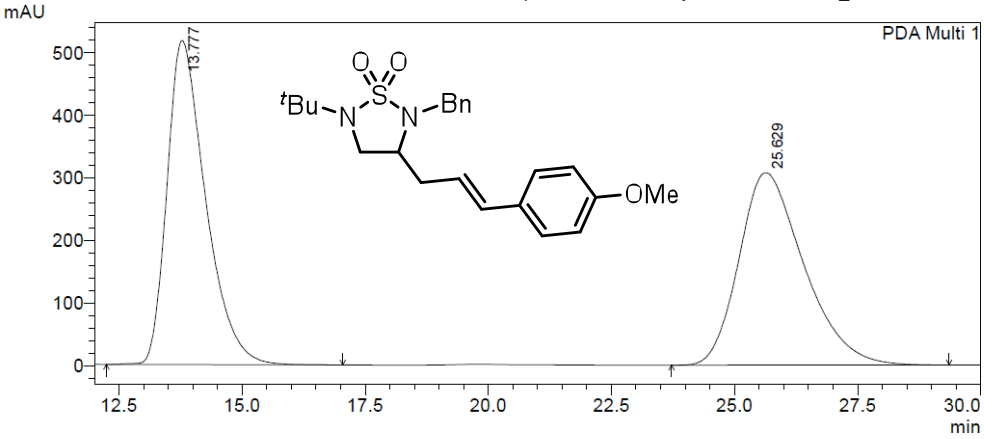
==== Shimadzu LCsolution Analysis Report ====

C:\...\5 Membered Sulfonamides\RAC-KP-1-46-Bn-XPhos-2-pOMebetabromosty-7.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: RAC-KP-1-46-Bn-XPhos-2-pOMebetabromosty-7.00%IPA-1.0mL_min-ODH2
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-KP-1-46-Bn-XPhos-2-pOMebetabromosty-7.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/24/2015 8:42:31 AM
Data Processed	: 10/24/2015 9:23:04 AM

<Chromatogram>

C:\...\5 Membered Sulfonamides\RAC-KP-1-46-Bn-XPhos-2-pOMebetabromosty-7.00%IPA-1.0mL_min-ODH1.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.777	28171525	517359	50.060	62.745
2	25.629	28104015	307186	49.940	37.255
Total		56275541	824546	100.000	100.000

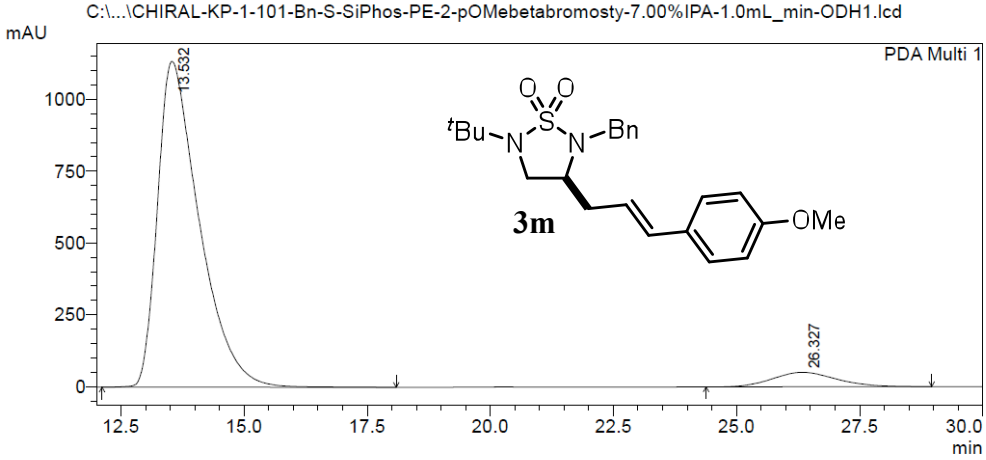
==== Shimadzu LCsolution Analysis Report ====

C:\...\CHIRAL-KP-1-101-Bn-S-SiPhos-PE-2-pOMebetabromosty-7.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-KP-1-101-Bn-S-SiPhos-PE-2-pOMebetabromosty-7.00%IPA-1.0m
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-KP-1-101-Bn-S-SiPhos-PE-2-pOMebetabromosty-7.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/24/2015 8:10:28 AM
Data Processed	: 10/24/2015 8:41:39 AM

<Chromatogram>

C:\...\CHIRAL-KP-1-101-Bn-S-SiPhos-PE-2-pOMebetabromosty-7.00%IPA-1.0mL_min-ODH1.lcd

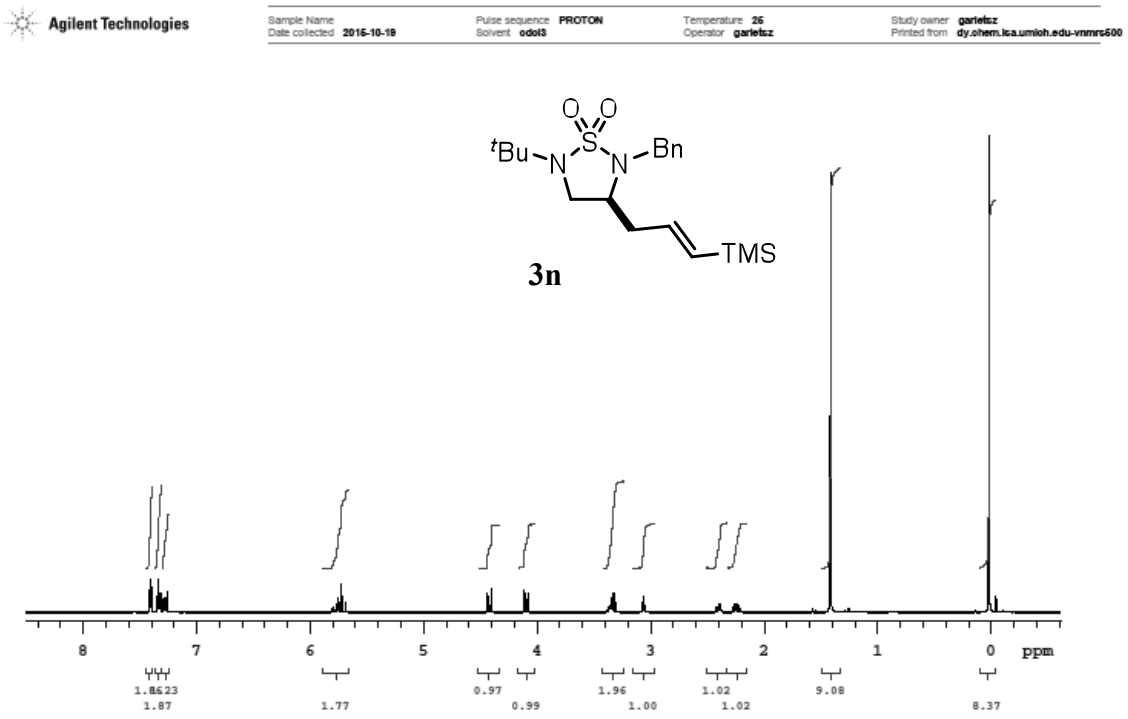


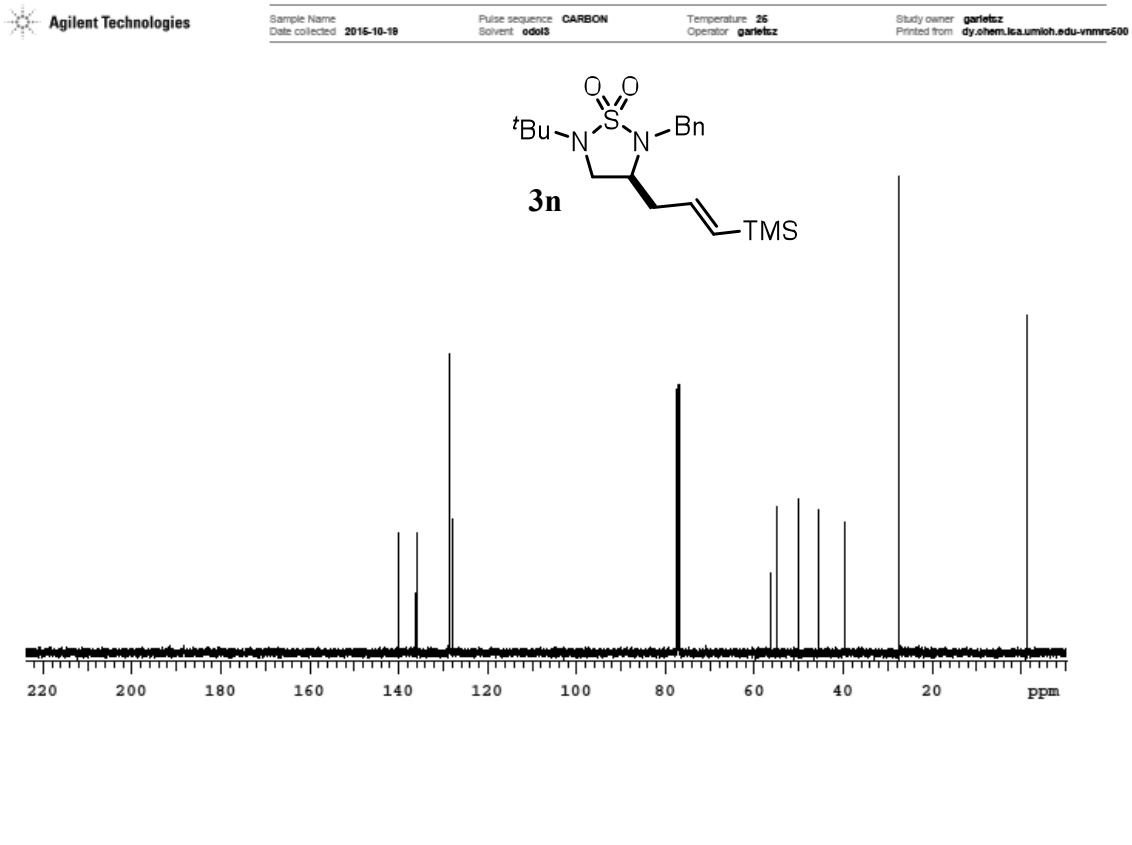
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.532	67008574	1134379	93.673	95.770
2	26.327	4525909	50107	6.327	4.230
Total		71534483	1184486	100.000	100.000


Agilent Technologies
Sample Name
Date collected 2015-10-18
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs600
O
O
S
tBu
N
N
Bn
TMS
3n
8
7
6
5
4
3
2
1
0
ppm
1.8623
1.87
1.77
0.97
0.99
1.96
1.00
1.02
1.02
9.08
8.37
Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.low.100.p.1.H1.fid
Plot date 2015-10-19


Agilent Technologies
Sample Name
Date collected 2015-10-18
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs600
O
O
S
tBu
N
N
Bn
TMS
3n
220
200
180
160
140
120
100
80
60
40
20
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.100.p.C13.fid
Plot date 2015-10-19

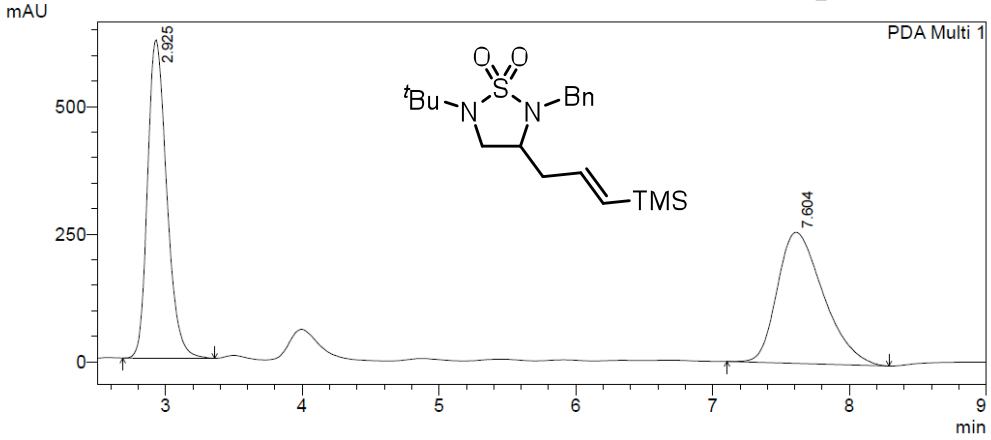
==== Shimadzu LCsolution Analysis Report ====

C:\...\Kaia Parenti\5 Membered Sulfonamides\RAC-KP-1-54-Bn-XPhos-BrAlkeneTMS-7.00%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-KP-1-54-Bn-XPhos-BrAlkeneTMS-7.00%IPA-1.0mL_min-ODH.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-KP-1-54-Bn-XPhos-BrAlkeneTMS-7.00%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/31/2015 9:59:49 AM
Data Processed	: 7/31/2015 10:37:59 AM

<Chromatogram>

C:\...\5 Membered Sulfonamides\RAC-KP-1-54-Bn-XPhos-BrAlkeneTMS-7.00%IPA-1.0mL_min-ODH.lcd



1 PDA Multi 1/220nm 4nm

PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	2.925	6077830	622938	50.288	70.848
2	7.604	6008303	256323	49.712	29.152
Total		12086133	879260	100.000	100.000

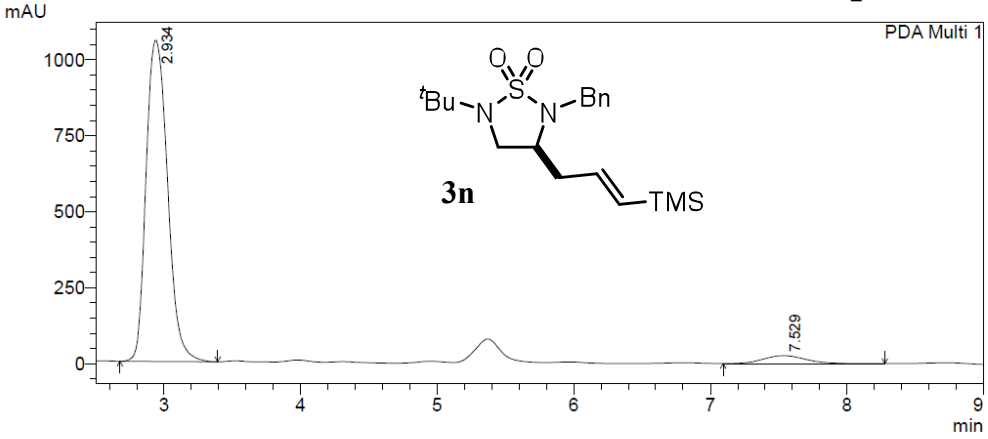
==== Shimadzu LCsolution Analysis Report ====

C:\...\5 Membered Sulfonamides\CHIRAL-KP-1-98-Bn-S-SIPhos-PE-BrAlkeneTMS-7.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-177-Bn-S-Siphos-PE-p-morpholine-7.00%IPA-1.0mL_min
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-KP-1-98-Bn-S-SIPhos-PE-BrAlkeneTMS-7.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/19/2015 9:37:58 AM
Data Processed	: 10/19/2015 9:55:48 AM

<Chromatogram>

C:\...\5 Membered Sulfonamides\CHIRAL-KP-1-98-Bn-S-SIPhos-PE-BrAlkeneTMS-7.00%IPA-1.0mL_min-ODH1.lcd



1 PDA Multi 1/220nm 4nm

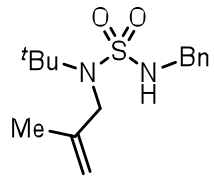
PeakTable

PDA Ch1 220nm 4nm

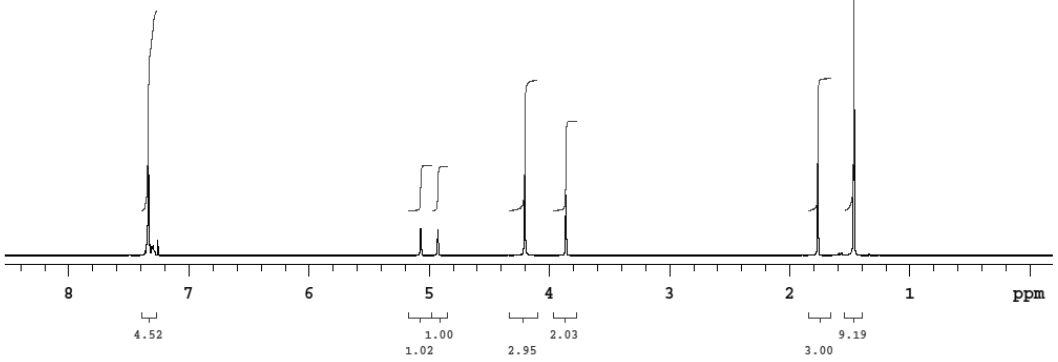
Peak#	Ret. Time	Area	Height	Area %	Height %
1	2.934	11570498	1056804	95.375	97.582
2	7.529	561033	26183	4.625	2.418
Total		12131530	1082987	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2015-10-20
Pulse sequence PROTON
Solvent cdcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs500



4a

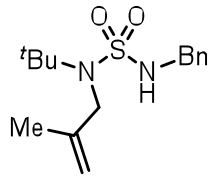


Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.57.p.1.H1.fid

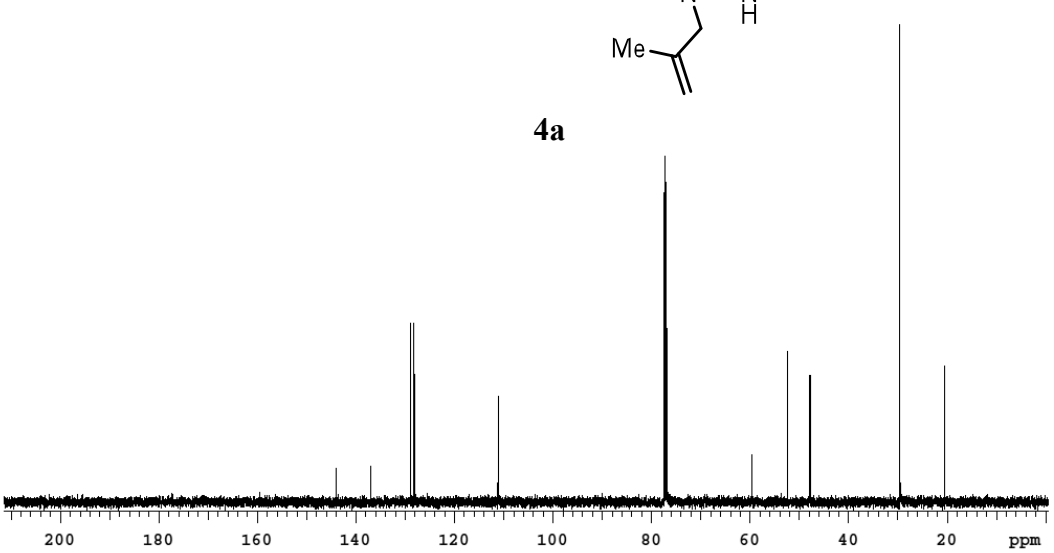
Plot date 2015-10-20

Agilent Technologies

Sample Name
Date collected 2015-10-20
Pulse sequence CARBON
Solvent cdcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs500



4a

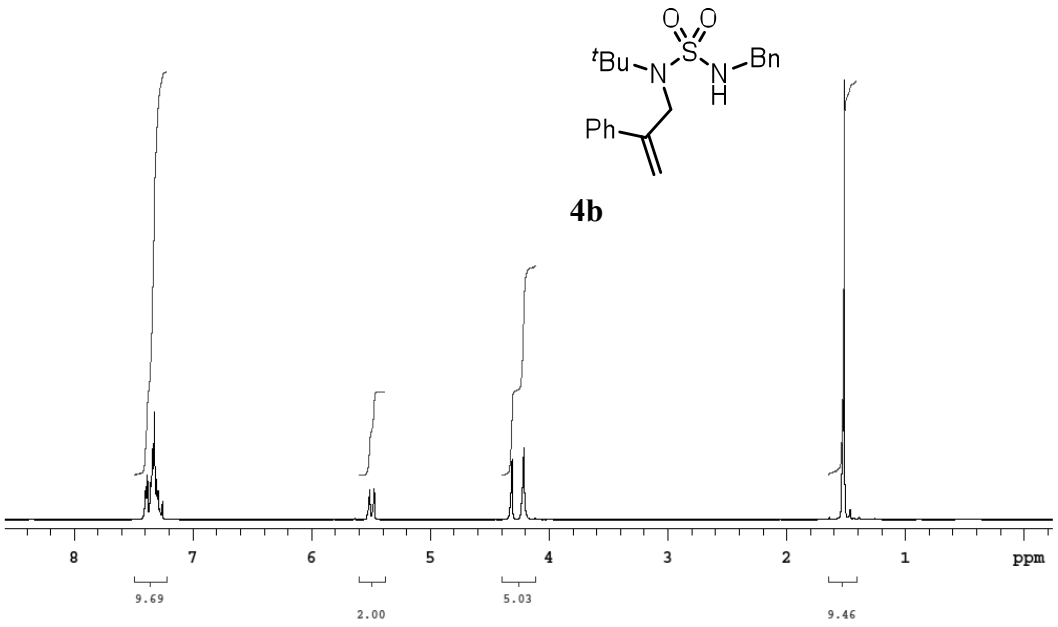


Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.57.p.2.C13.fid

Plot date 2015-10-20

Agilent Technologies

Sample Name
Date collected 2015-10-20
Pulse sequence PROTON
Solvent cdcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs500



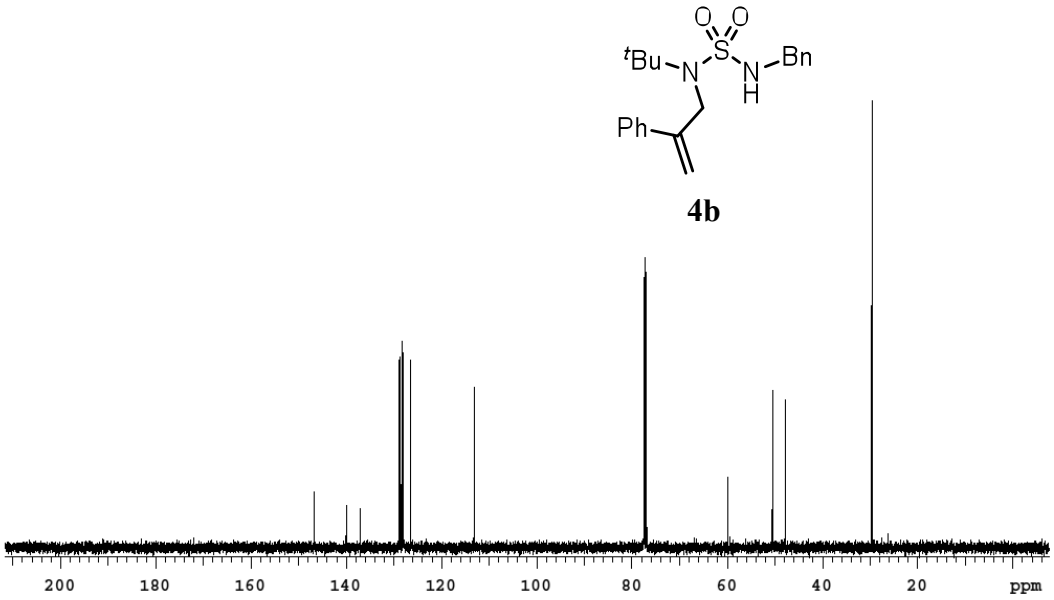
4b

Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.51.p.1.H1.fid

Plot date 2015-10-20

Agilent Technologies

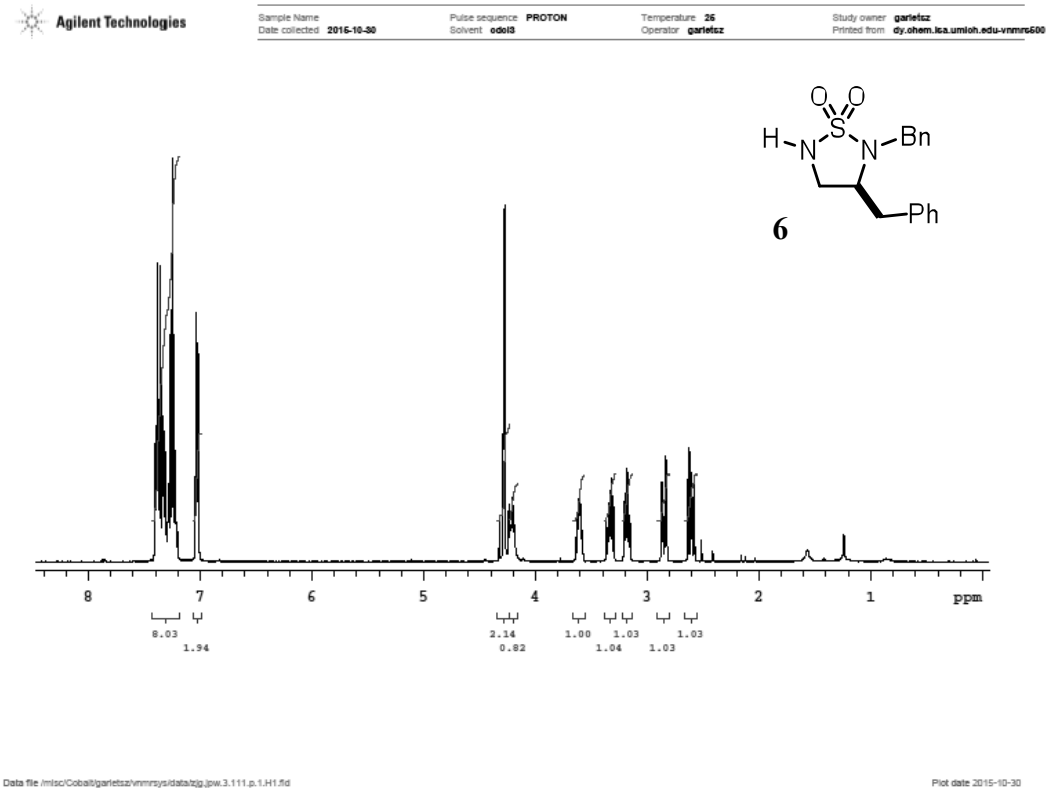
Sample Name
Date collected 2015-10-20
Pulse sequence CARBON
Solvent cdcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs500

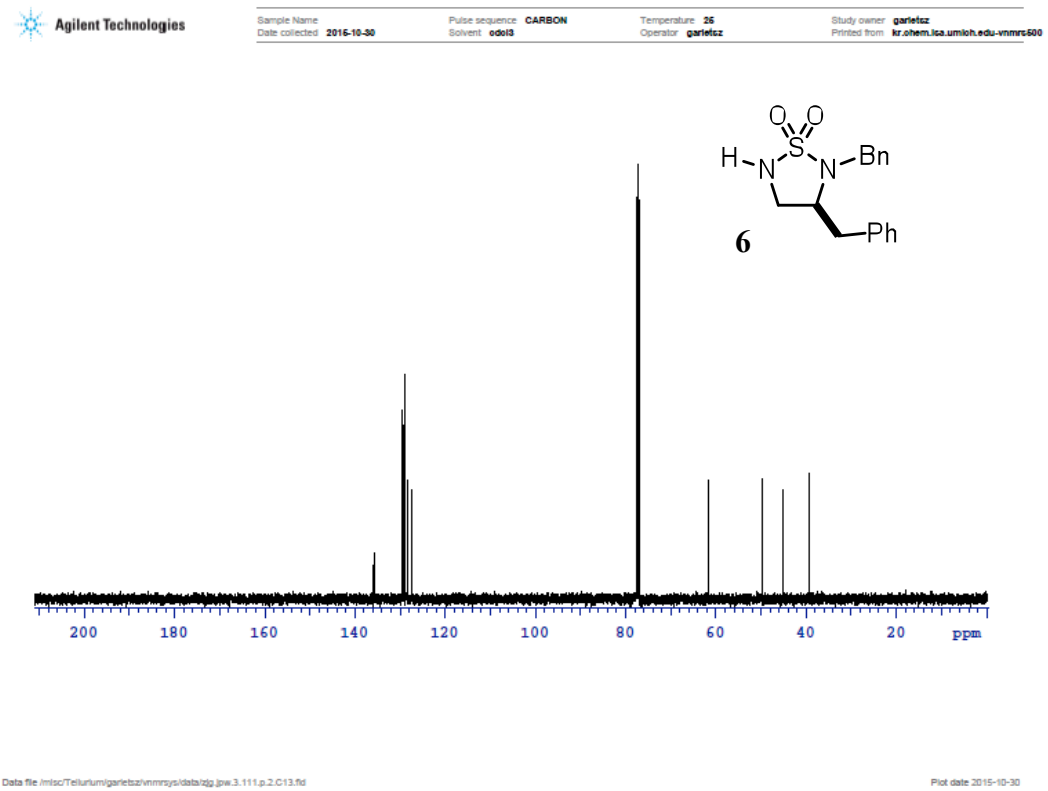


4b

Data file /misc/Tellurium/garletsz/vnmrsys/data/kp.jpw.1.51.p.2.C13.fid

Plot date 2015-10-20


Agilent Technologies
Sample Name
Date collected 2016-10-30
Pulse sequence PROTON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from dy.ohem.lsa.umich.edu-vnmrs600
O
O
S
H
N
N
Bn
Ph
6
8
7
6
5
4
3
2
1
ppm
8.03
1.94
2.14
0.82
1.00
1.04
1.03
1.03
1.03
Data file /misc/Cobalt/garletsz/vnmrsys/data/zjg.jpw.3.111.p.1.H1.fid
Plot date 2015-10-30


Agilent Technologies
Sample Name
Date collected 2016-10-30
Pulse sequence CARBON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs600
O
O
S
H
N
N
Bn
Ph
6
200
180
160
140
120
100
80
60
40
20
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.111.p.2.C13.fid
Plot date 2015-10-30

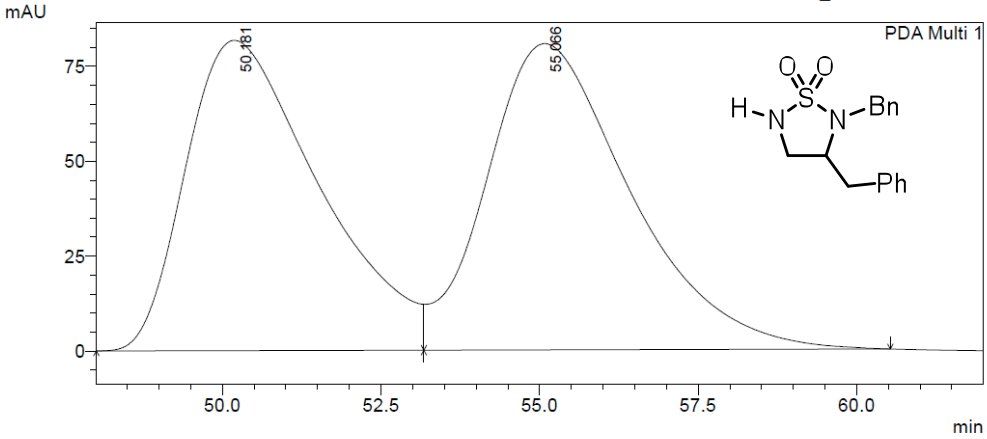
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Zac G\Chiral 5 Membered Sulfonamides\RAC-ZJG-3-138-Bn-Ph-7.00%IPA-0.5mL_min-ODH2.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-3-138-Bn-Ph-7.00%IPA-0.5mL_min-ODH2.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-3-138-Bn-Ph-7.00%IPA-0.5mL_min-ODH2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/16/2015 10:14:41 AM
Data Processed	: 9/16/2015 11:20:39 AM

<Chromatogram>

C:\...\Data\Zac G\Chiral 5 Membered Sulfonamides\RAC-ZJG-3-138-Bn-Ph-7.00%IPA-0.5mL_min-ODH2.lcd



1 PDA Multi 1/210nm 4nm

PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	50.181	11931851	81757	48.384	50.330
2	55.066	12728869	80686	51.616	49.670
Total		24660720	162443	100.000	100.000

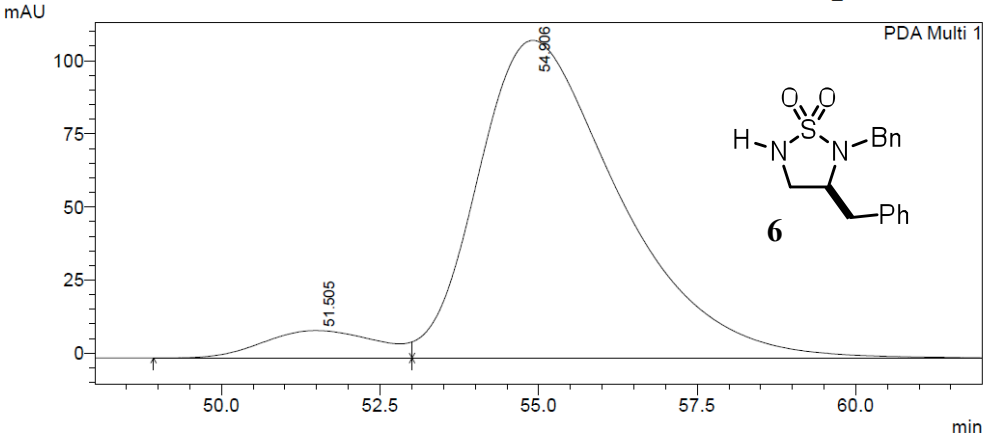
==== Shimadzu LCsolution Analysis Report ====

C:\...\Data\Zac G\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-3-111-Bn-Ph-7.00%IPA-0.5mL_min-ODH3.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-111-Bn-Ph-7.00%IPA-0.5mL_min-ODH3.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-111-Bn-Ph-7.00%IPA-0.5mL_min-ODH3.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/16/2015 11:21:32 AM
Data Processed	: 9/16/2015 12:29:01 PM

<Chromatogram>

C:\...\Data\Zac G\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-3-111-Bn-Ph-7.00%IPA-0.5mL_min-ODH3.lcd



1 PDA Multi 1/210nm 4nm

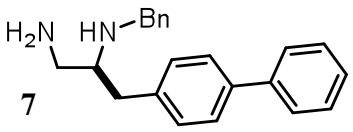
PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	51.505	1170311	9364	6.488	7.939
2	54.906	16868488	108583	93.512	92.061
Total		18038799	117947	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2016-10-24
Pulse sequence PROTON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs500



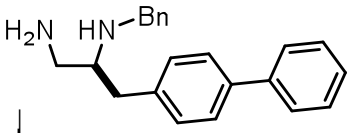
7

Data file /misc/Cobalt/garletsz/vnmrsys/data/zjg.jpw.3.184.p.3.H1.fid

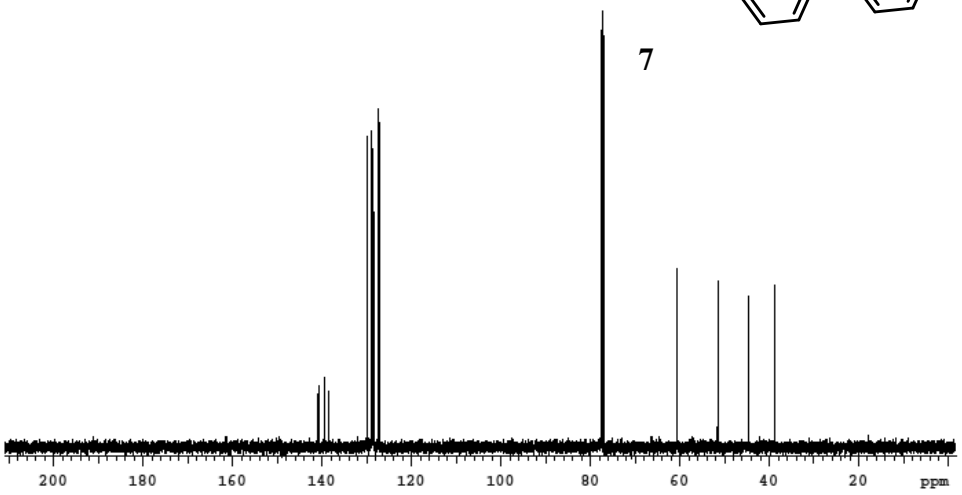
Plot date 2015-10-26

Agilent Technologies

Sample Name
Date collected 2016-10-24
Pulse sequence CARBON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs500

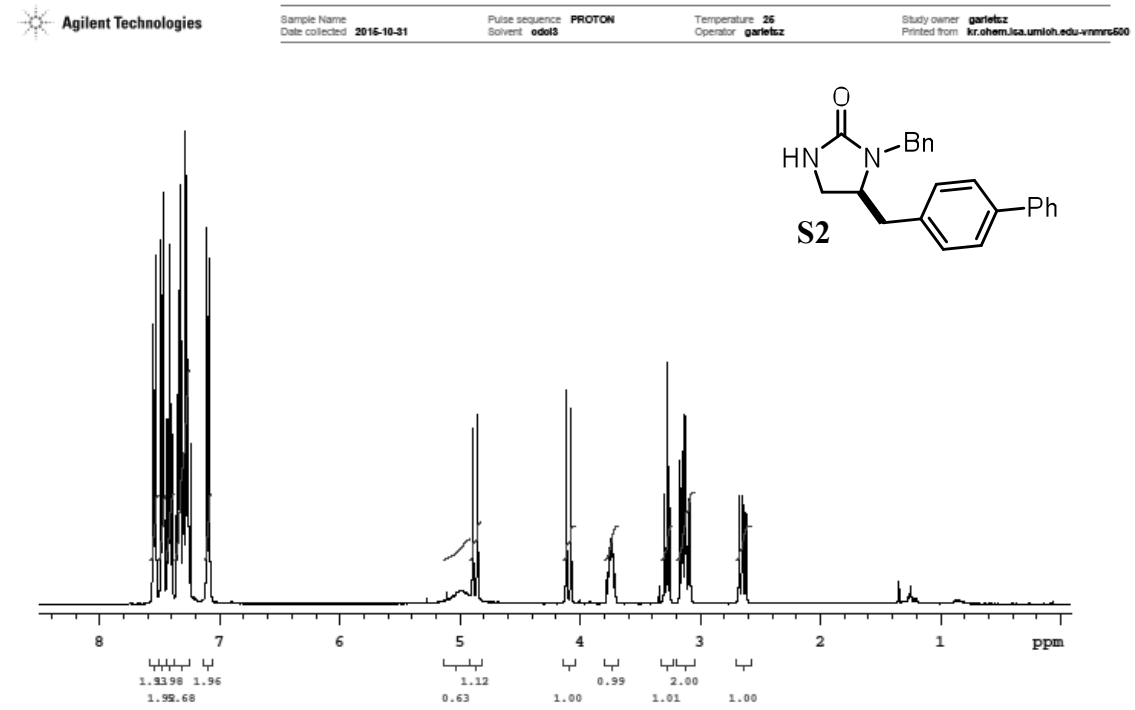


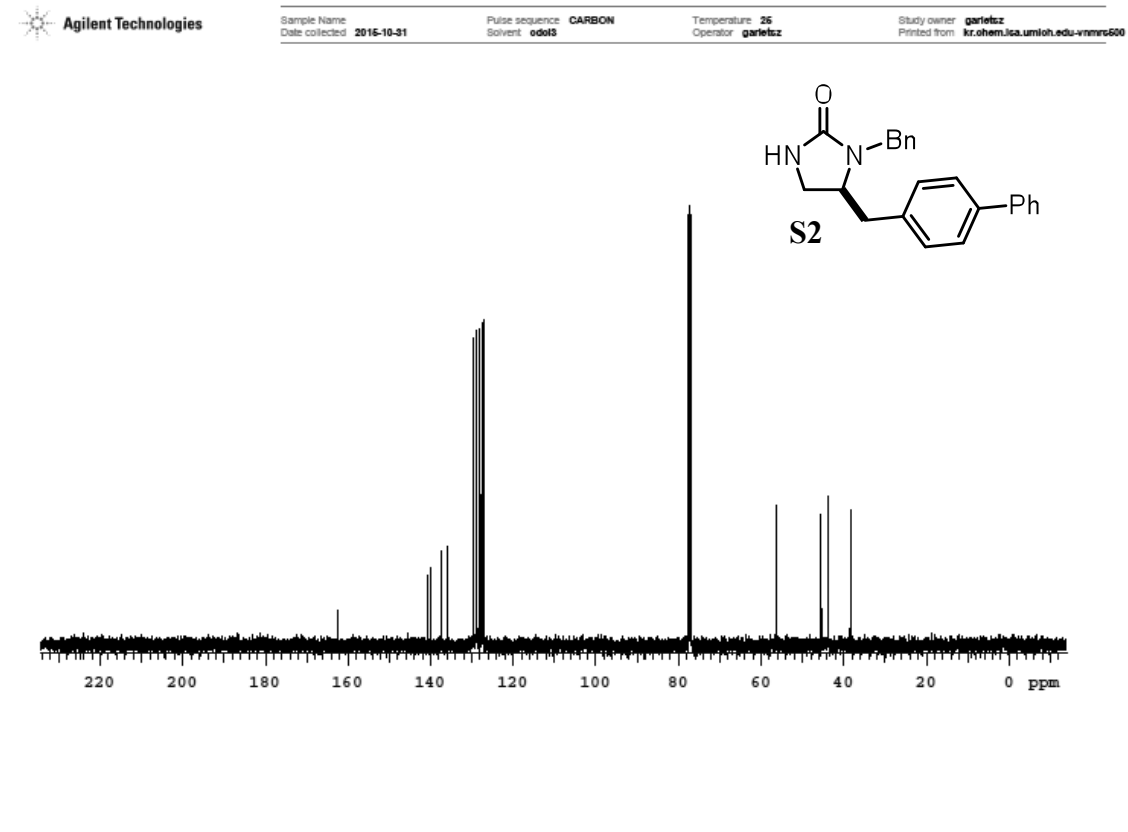
7



Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.184.p.2.C13.fid

Plot date 2015-10-26


Agilent Technologies
Sample Name
Date collected 2015-10-31
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs500
O
HN
N
Bn
Ph
S2
8
7
6
5
4
3
2
1
ppm
1.95
3.98
1.96
2.68
1.12
0.63
1.00
0.99
2.00
1.01
1.00
Data file /misc/Cobalt/garletsz/vnmrsys/data/zjg.jpw.3.190.p.1.H1.fid
Plot date 2015-10-31


Agilent Technologies
Sample Name
Date collected 2015-10-31
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.ohem.lsa.umich.edu-vnmrs500
O
HN
N
Bn
Ph
S2
220
200
180
160
140
120
100
80
60
40
20
0 ppm
Data file /misc/Cobalt/garletsz/vnmrsys/data/zjg.jpw.3.190.2.C13.fid
Plot date 2015-10-31

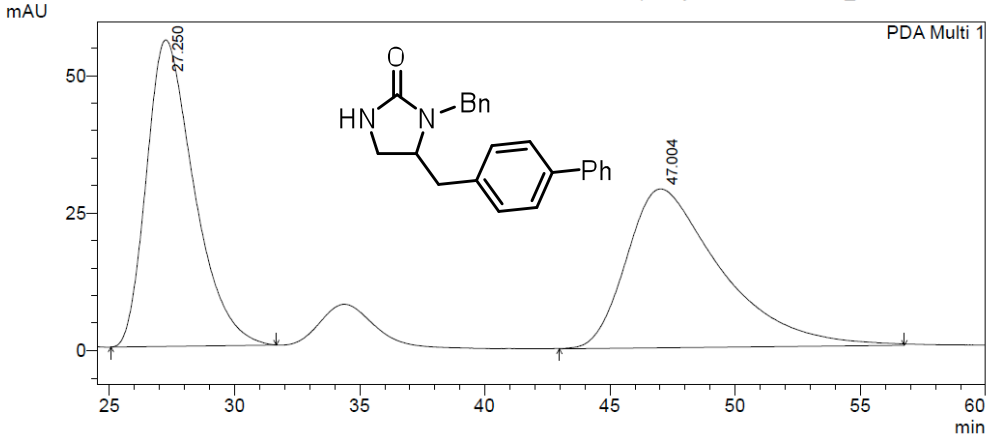
==== Shimadzu LCsolution Analysis Report ====

C:\...\Data\Zac G\Chiral 5 Membered Sulfonamides\RAC-ZJG-3-188-Bn-urea-biphenyl-7.00%IPA-1.0mL_min-ODH3.lcd

Acquired by	: Admin
Sample Name	: RAC-ZJG-3-188-Bn-urea-biphenyl-7.00%IPA-1.0mL_min-ODH3.lcd
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-ZJG-3-188-Bn-urea-biphenyl-7.00%IPA-1.0mL_min-ODH3.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/30/2015 11:45:48 AM
Data Processed	: 10/30/2015 1:25:51 PM

<Chromatogram>

C:\...\Zac G\Chiral 5 Membered Sulfonamides\RAC-ZJG-3-188-Bn-urea-biphenyl-7.00%IPA-1.0mL_min-ODH3.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	27.250	7380836	55749	49.303	65.854
2	47.004	7589674	28906	50.697	34.146
Total		14970510	84655	100.000	100.000

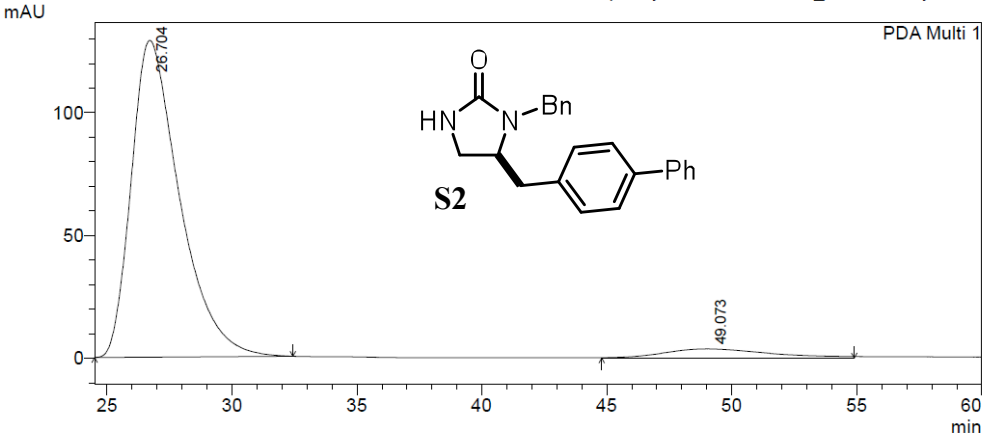
==== Shimadzu LCsolution Analysis Report ====

C:\...\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-3-190-Bn-urea-biphenyl-7.00%IPA-1.0mL_min-ODH-try2.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-190-Bn-urea-biphenyl-7.00%IPA-1.0mL_min-ODH-try2.l
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-190-Bn-urea-biphenyl-7.00%IPA-1.0mL_min-ODH-try2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/2/2015 9:09:36 AM
Data Processed	: 11/2/2015 10:27:29 AM

<Chromatogram>

C:\...\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-3-190-Bn-urea-biphenyl-7.00%IPA-1.0mL_min-ODH-try2.lcd

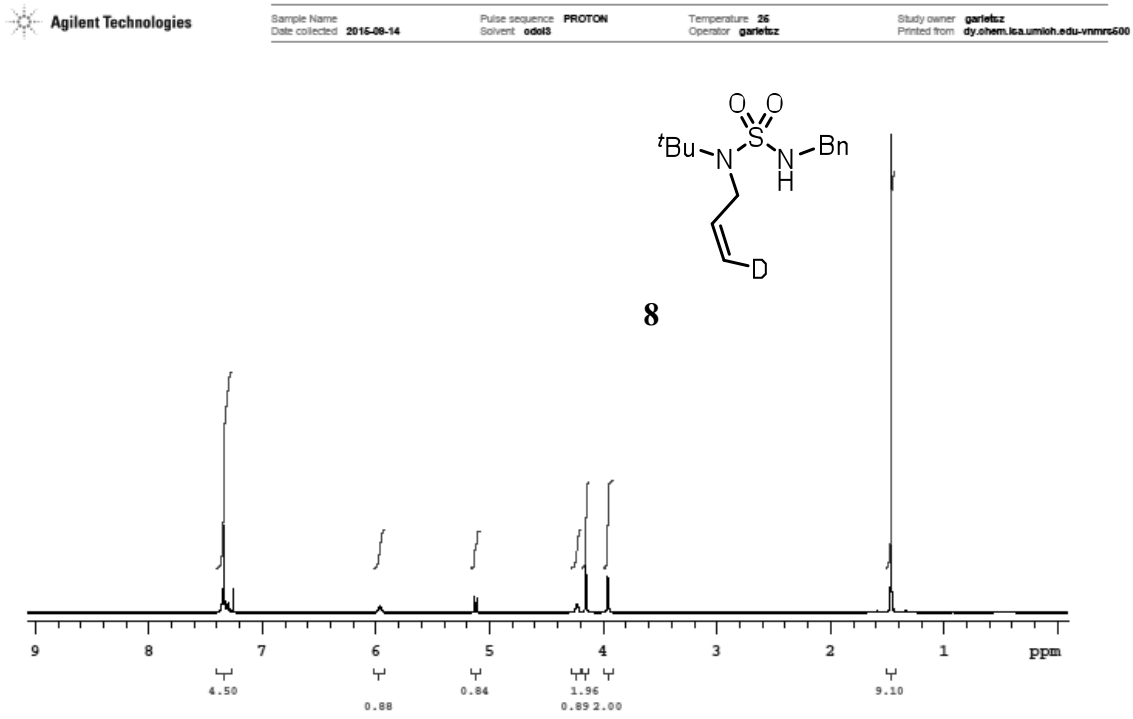


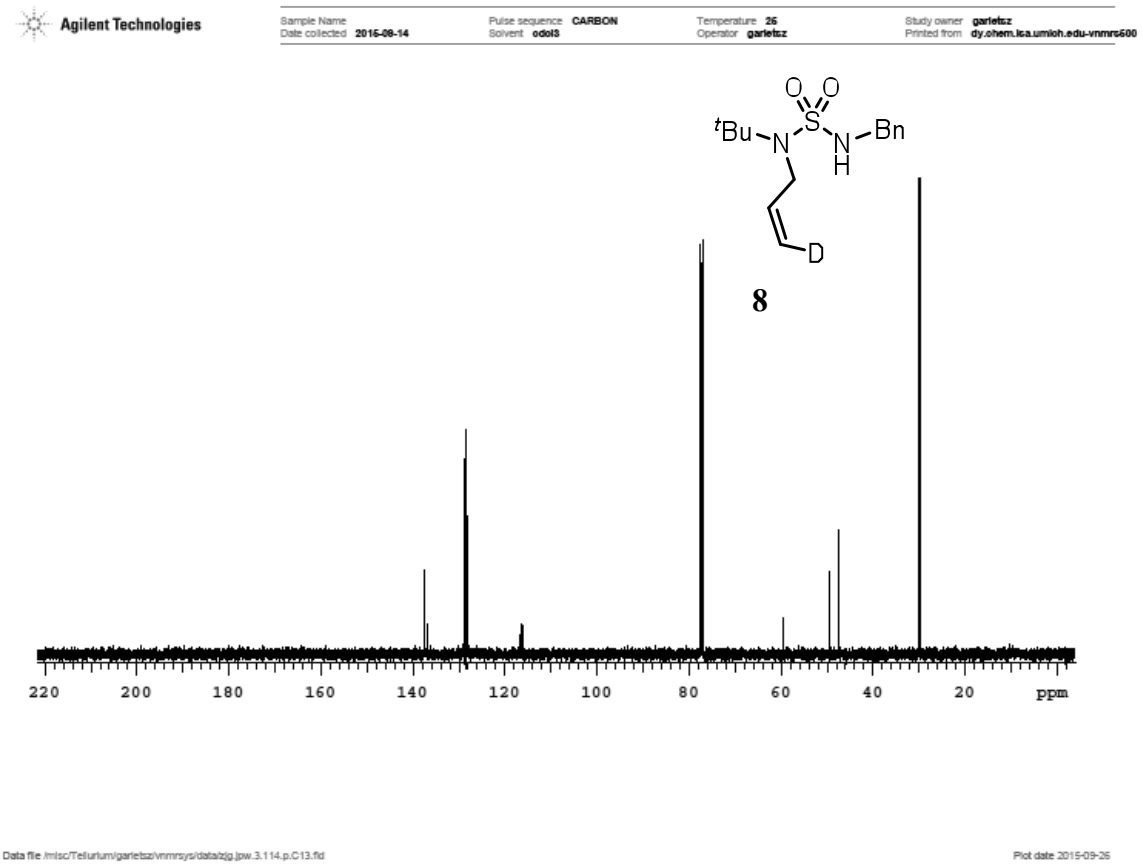
1 PDA Multi 1/254nm 4nm

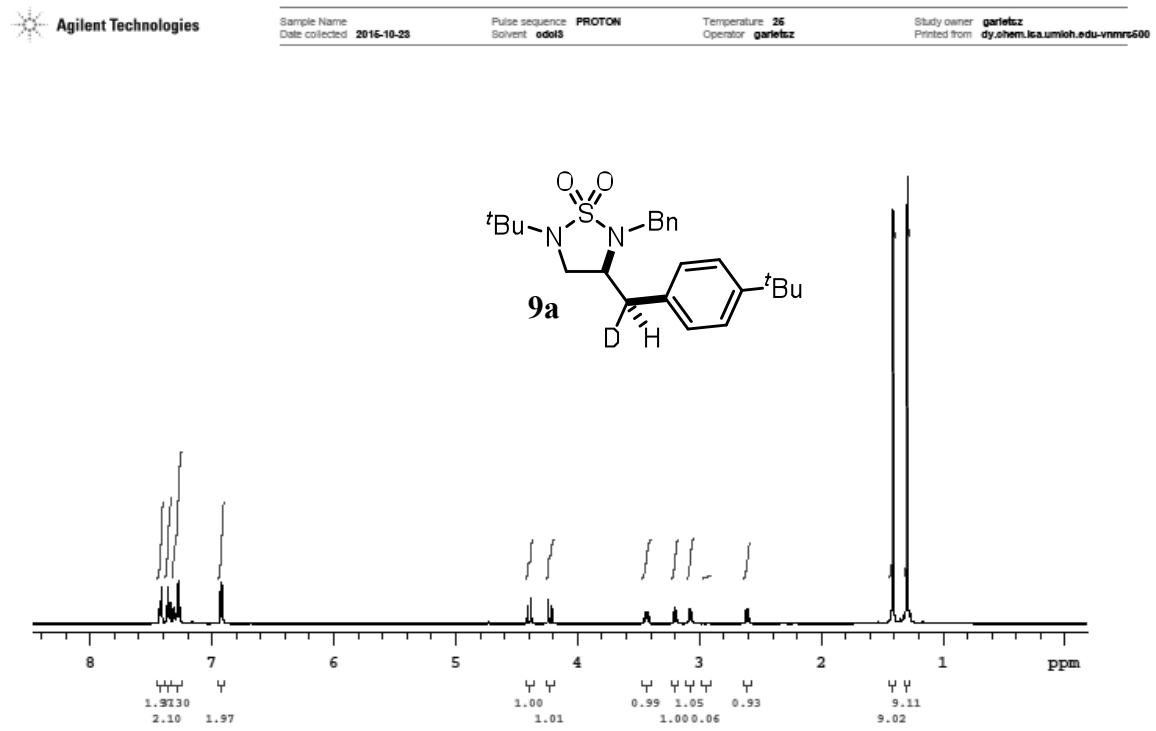
PeakTable

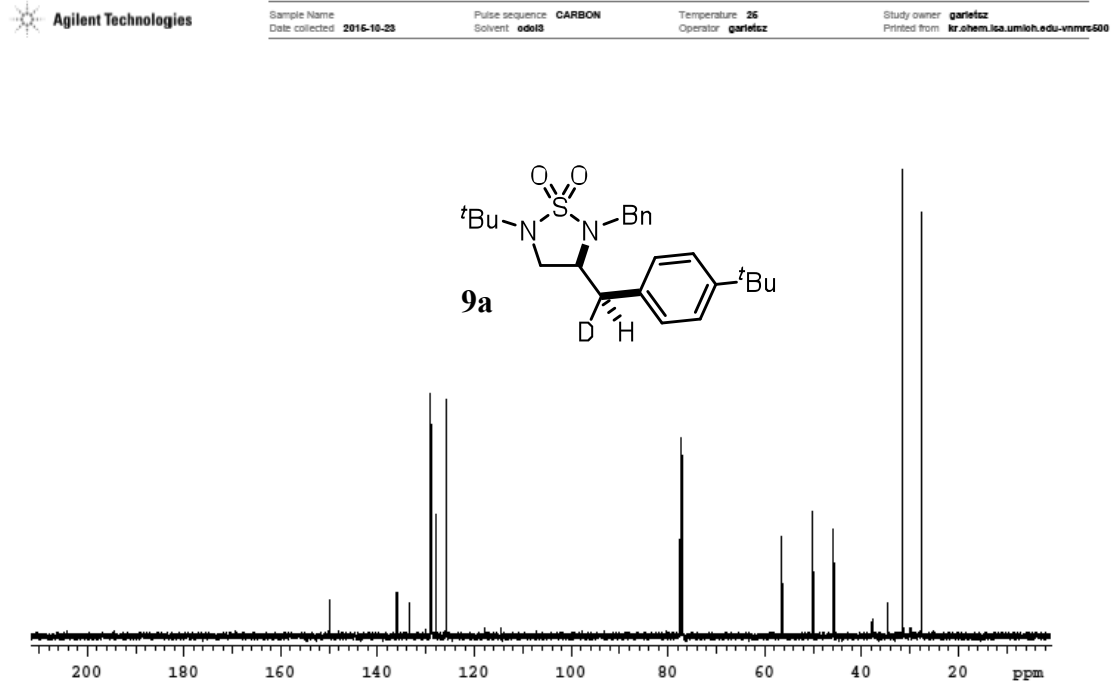
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.704	17352458	128883	94.161	97.195
2	49.073	1076039	3719	5.839	2.805
Total		18428497	132603	100.000	100.000


Agilent Technologies
Sample Name
Date collected 2015-09-14
Pulse sequence PROTON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from dy.ohem.isa.umich.edu-vnmrs600
O O
S
tBu N N Bn
H
D
8
9 8 7 6 5 4 3 2 1 ppm
4.50 0.88 0.84 1.96 0.89 2.00 9.10
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.114.p.H1.fid
Plot date 2015-09-26


Agilent Technologies
Sample Name
Date collected 2015-09-14
Pulse sequence CARBON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from dy.ohem.isa.umich.edu-vnmrs600
O O
S
tBu N N Bn
H
D
8
220 200 180 160 140 120 100 80 60 40 20 ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.114.p.C13.fid
Plot date 2015-09-26


Agilent Technologies
Sample Name
Date collected 2016-10-23
Pulse sequence PROTON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs500
9a
ppm
1.97 1.30
2.10 1.97
1.00
1.01
0.99 1.05 0.93
1.00 0.06
9.11
9.02
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.118.p.1.H1.fid
Plot date 2016-01-14


Agilent Technologies
Sample Name
Date collected 2016-10-23
Pulse sequence CARBON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs500
9a
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.118.p.2.C13.fid
Plot date 2015-10-23

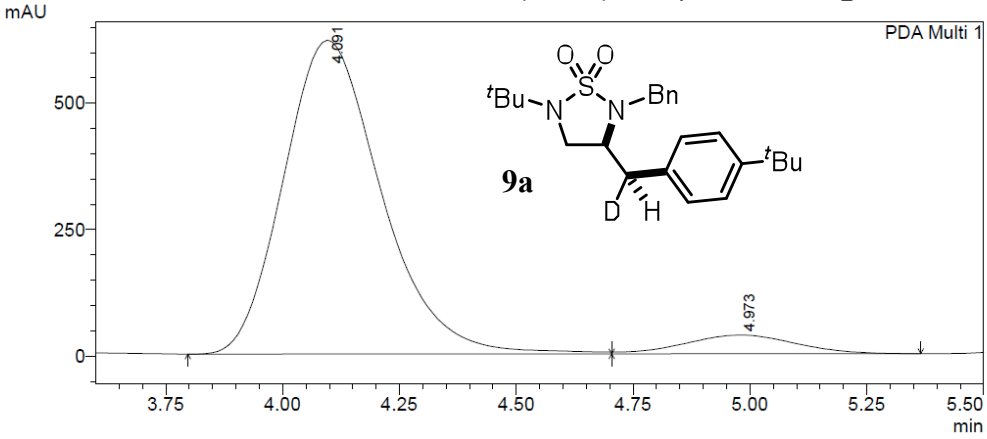
==== Shimadzu LCsolution Analysis Report ====

C:\...\5 Membered Sulfonamides\CHIRAL-ZJG-3-118-D-S-Siphos-PE-p-tert-butyl-7.00%IPA-1.0mL_min-ODH2.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-118-D-S-Siphos-PE-p-tert-butyl-7.00%IPA-1.0mL_min-
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-118-D-S-Siphos-PE-p-tert-butyl-7.00%IPA-1.0mL_min-ODH2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/31/2015 2:20:07 PM
Data Processed	: 10/31/2015 2:30:54 PM

<Chromatogram>

C:\...\5 Membered Sulfonamides\CHIRAL-ZJG-3-118-D-S-Siphos-PE-p-tert-butyl-7.00%IPA-1.0mL_min-ODH2.lcd



1 PDA Multi 1/205nm 4nm

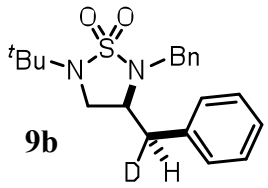
PeakTable

PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.091	9020083	620701	93.727	94.395
2	4.973	603683	36856	6.273	5.605
Total		9623766	657556	100.000	100.000

Agilent Technologies

Sample Name
Date collected 2015-08-14
Pulse sequence PROTON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs600



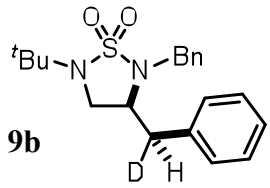
9b

Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.116.p.H1.fid

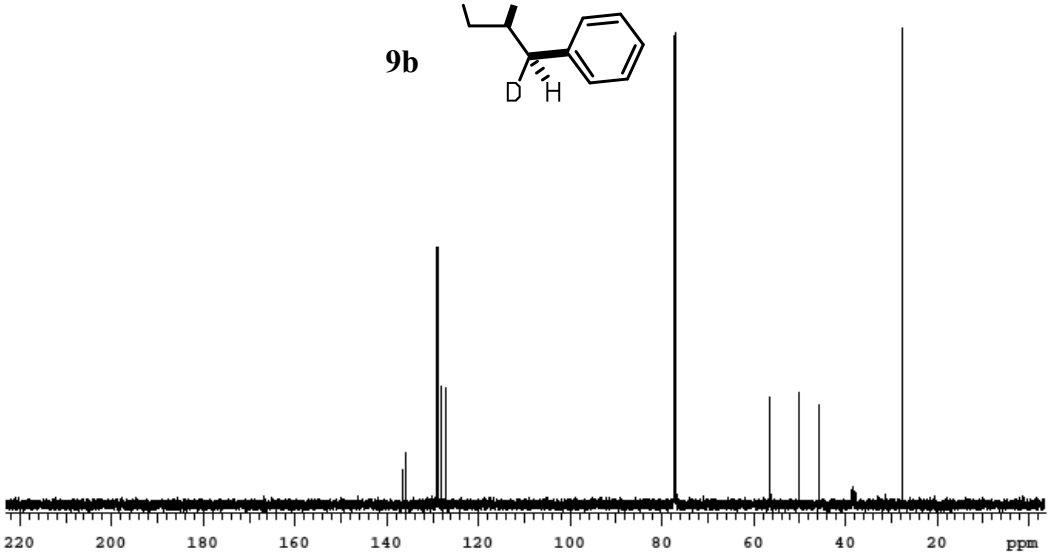
Plot date 2016-01-14

Agilent Technologies

Sample Name
Date collected 2015-08-14
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs600



9b



Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.116.p.C13.fid

Plot date 2015-09-26

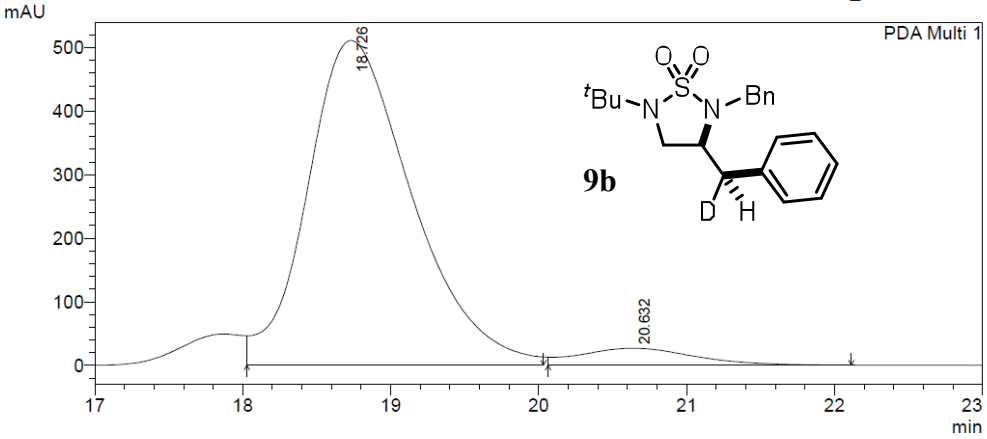
==== Shimadzu LCsolution Analysis Report ====

C:\...\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-3-116-Bn-S-SiPhos-PE-Ph-D-4.00%IPA-0.5mL_min-ODH2.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-116-Bn-S-SiPhos-PE-Ph-D-4.00%IPA-0.5mL_min-ODH2.lc
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-116-Bn-S-SiPhos-PE-Ph-D-4.00%IPA-0.5mL_min-ODH2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/26/2015 11:56:37 AM
Data Processed	: 9/26/2015 12:28:51 PM

<Chromatogram>

C:\...\Chiral 5 Membered Sulfonamides\CHIRAL-ZJG-3-116-Bn-S-SiPhos-PE-Ph-D-4.00%IPA-0.5mL_min-ODH2.lcd

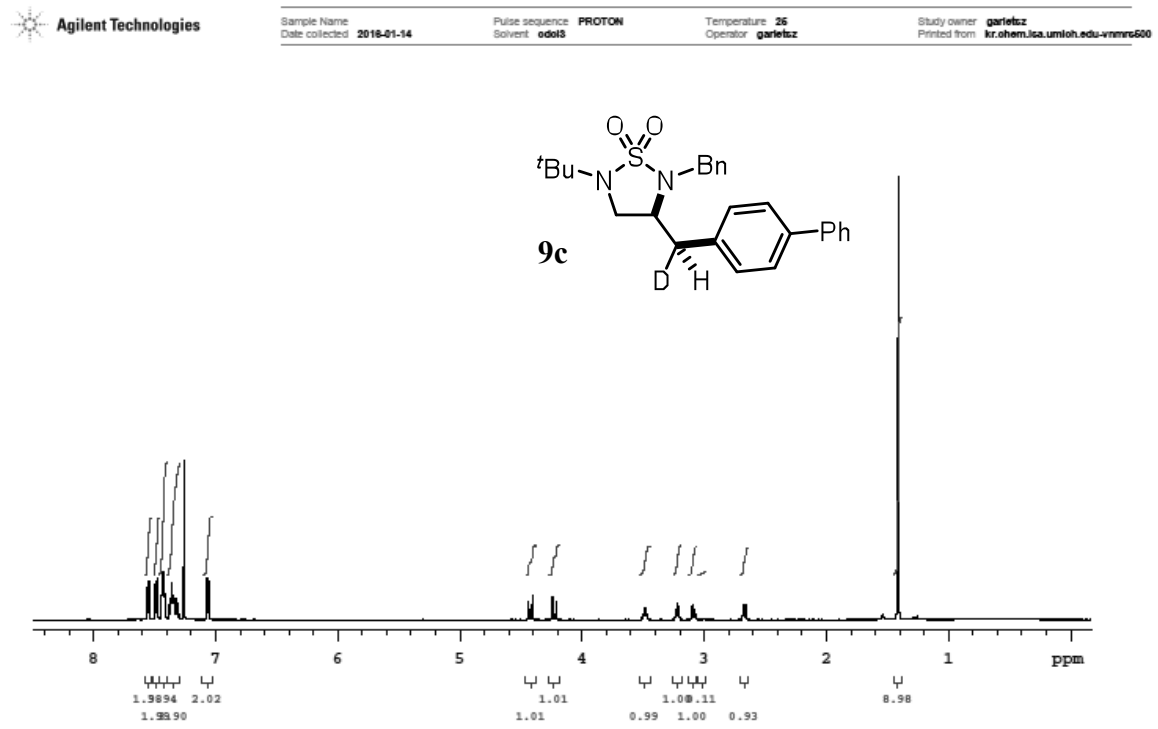


1 PDA Multi 1/210nm 4nm

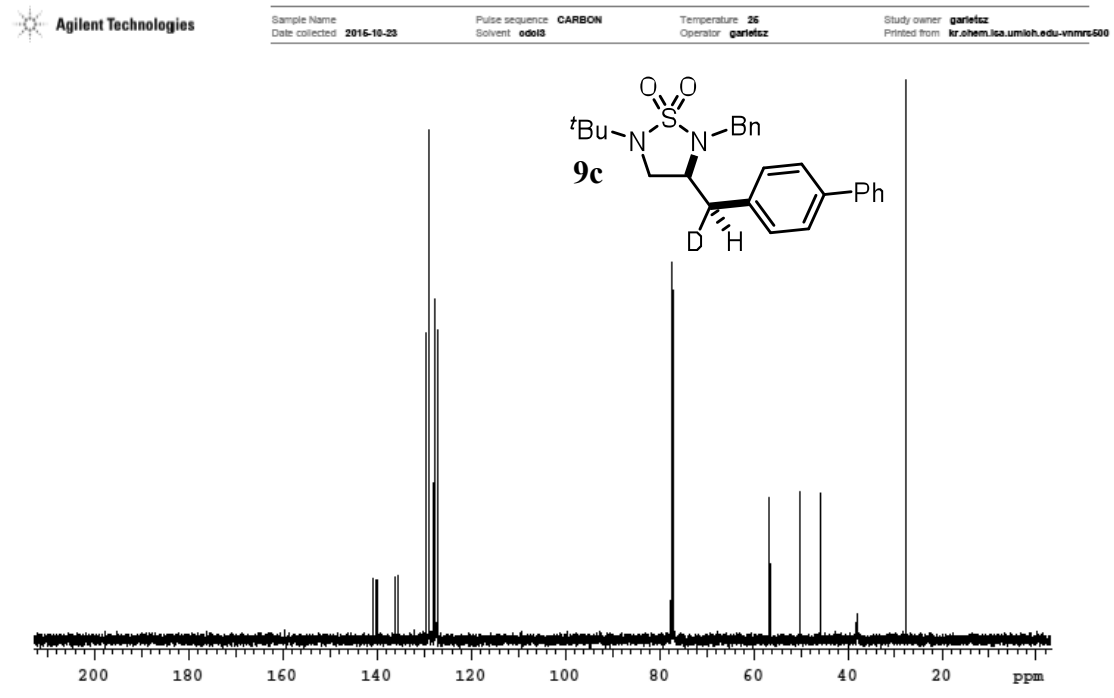
PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.726	25060681	510704	94.537	95.004
2	20.632	1448139	26855	5.463	4.996
Total		26508820	537559	100.000	100.000


Agilent Technologies
Sample Name
Date collected 2018-01-14
Pulse sequence PROTON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs500
9c
Ph
D H
8
7
6
5
4
3
2
1
ppm
1.98
94
2.02
1.01
1.01
0.99
1.00
0.93
8.98
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.117.p.g.H1.fid
Plot date 2016-01-14

y


Agilent Technologies
Sample Name
Date collected 2015-10-23
Pulse sequence CARBON
Solvent odcl3
Temperature 26
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs500
9c
Ph
D H
200
180
160
140
120
100
80
60
40
20
ppm
Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.117.p.2.C13.fid
Plot date 2015-10-23

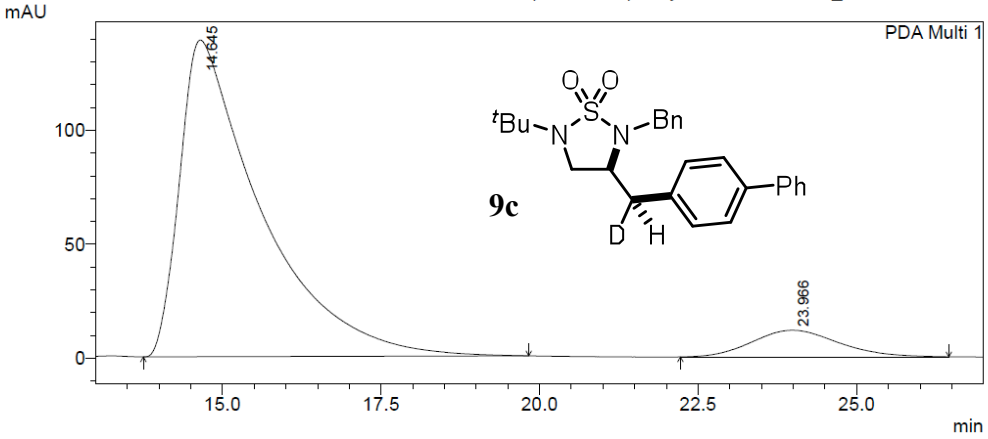
==== Shimadzu LCsolution Analysis Report ====

C:\...\5 Membered Sulfonamides\CHIRAL-ZJG-3-117-D-S-Siphos-PE-biphenyl-7.00%IPA-1.0mL_min-ODH3.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-117-D-S-Siphos-PE-biphenyl-7.00%IPA-1.0mL_min-ODH3
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-117-D-S-Siphos-PE-biphenyl-7.00%IPA-1.0mL_min-ODH3.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/31/2015 3:02:41 PM
Data Processed	: 10/31/2015 3:43:29 PM

<Chromatogram>

C:\...\5 Membered Sulfonamides\CHIRAL-ZJG-3-117-D-S-Siphos-PE-biphenyl-7.00%IPA-1.0mL_min-ODH3.lcd



1 PDA Multi 1/254nm 4nm

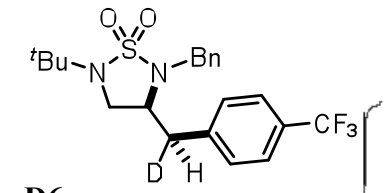
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.645	12360879	139007	91.778	92.234
2	23.966	1107406	11705	8.222	7.766
Total		13468285	150711	100.000	100.000

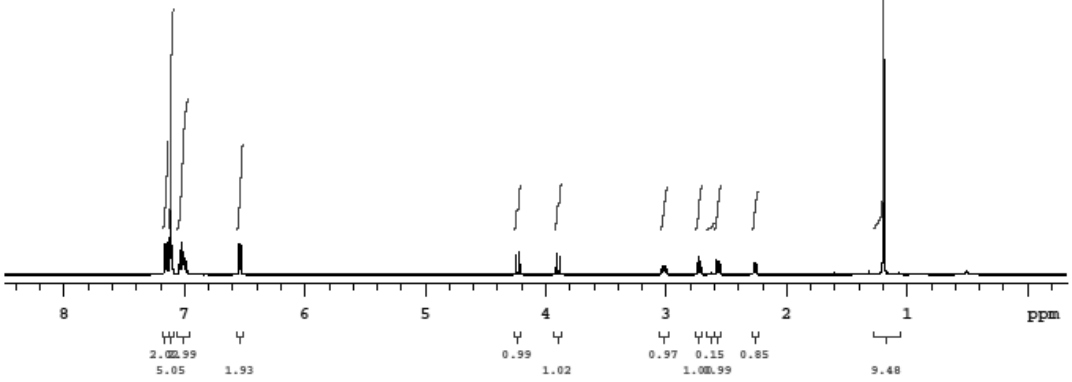
Agilent Technologies

Sample Name
Date collected 2015-08-23
Pulse sequence PROTON
Solvent o6d8
Temperature 25
Operator garletsz
Study owner garletsz
Printed from dy.chem.lsa.umich.edu-vnmrs500



9d

Benzene-D6

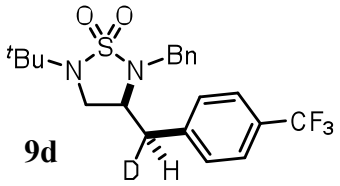


Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.143.p.benzened6.H1.fid

Plot date 2016-01-14

Agilent Technologies

Sample Name
Date collected 2016-09-19
Pulse sequence CARBON
Solvent odcl3
Temperature 25
Operator garletsz
Study owner garletsz
Printed from kr.chem.lsa.umich.edu-vnmrs500



9d

$CDCl_3$

Data file /misc/Tellurium/garletsz/vnmrsys/data/zjg.jpw.3.143.p.C13.fid

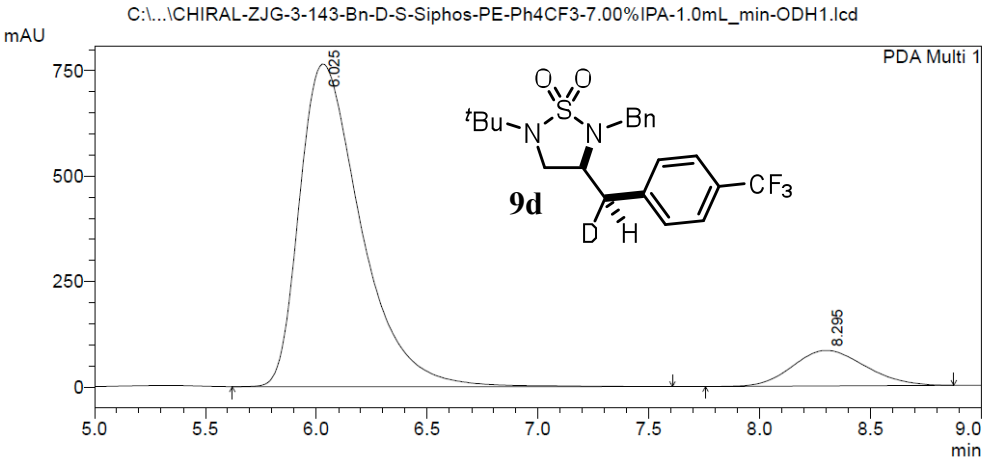
Plot date 2015-10-21

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

C:\...\CHIRAL-ZJG-3-143-Bn-D-S-Siphos-PE-Ph4CF3-7.00%IPA-1.0mL_min-ODH1.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-3-143-Bn-D-S-Siphos-PE-Ph4CF3-7.00%IPA-1.0mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-3-143-Bn-D-S-Siphos-PE-Ph4CF3-7.00%IPA-1.0mL_min-ODH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/21/2015 10:19:59 AM
Data Processed	: 9/21/2015 10:33:42 AM

<Chromatogram>



1 PDA Multi 1/210nm 4nm

PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.025	14750417	764558	88.443	90.058
2	8.295	1927470	84404	11.557	9.942
Total		16677886	848962	100.000	100.000

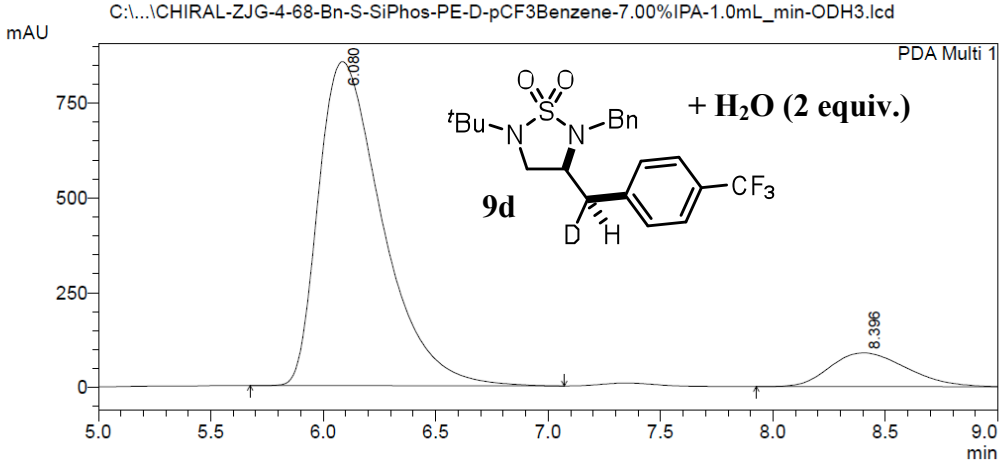
==== Shimadzu LCsolution Analysis Report ====

C:\...\CHIRAL-ZJG-4-68-Bn-S-SiPhos-PE-D-pCF3Benzene-7.00%IPA-1.0mL_min-ODH3.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-ZJG-4-68-Bn-S-SiPhos-PE-D-pCF3Benzene-7.00%IPA-1.0mL_min
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-ZJG-4-68-Bn-S-SiPhos-PE-D-pCF3Benzene-7.00%IPA-1.0mL_min-ODH3.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/14/2016 9:15:47 AM
Data Processed	: 1/14/2016 9:46:40 AM

<Chromatogram>

C:\...\CHIRAL-ZJG-4-68-Bn-S-SiPhos-PE-D-pCF3Benzene-7.00%IPA-1.0mL_min-ODH3.lcd



1 PDA Multi 1/210nm 4nm

PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.080	16873172	854096	88.916	90.573
2	8.396	2103313	88896	11.084	9.427
Total		18976485	942992	100.000	100.000