Angewandte Chemie
Eine Zeitschrift der Gesellschaft Deutscher Chemiker

Supporting Information

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Synthesis of Enantiomerically Enriched Imidazolidin-2-Ones through Asymmetric Palladium-Catalyzed Alkene Carboamination Reactions**

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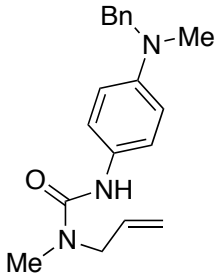
General: Reactions were carried out under nitrogen in flame-dried glassware. Tris(dibenzylideneacetone)dipalladium and (*S*)-Siphos-PE were purchased from Strem Chemical Co. and used without further purification. All other reagents including all aryl and alkenyl bromides were purchased from commercial sources and used as received unless otherwise noted. Xylenes were purified by distillation over CaH_2 prior to use in

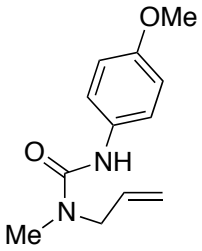
reactions. Methylene chloride and toluene were purified using a GlassContour solvent system. All yields refer to isolated compounds that are estimated to be ≥95% pure as judged by ^{1}H NMR analysis. The yields reported in the supporting information describe the result of a single experiment, whereas yields reported in Tables 1–2 and equations 2–4 are average yields of two or more experiments. Thus, the yields reported in the supporting information may differ from those shown in Table 2 and equations 2–4.

General procedure for the synthesis of *N*-allylurea substrates. A flame-dried round bottom flask equipped with a stirbar was cooled under a stream of nitrogen. The flask was charged with the appropriate isocyanate (1.0 equiv) and methylene chloride (0.60 M). The resulting solution was cooled to 0 °C and stirred for 5 min, then the allylic amine (1.1 equiv) was added dropwise. The solution was warmed to rt and stirred for five h. The mixture was then concentrated in vacuo and purified by flash chromatography on silica gel.



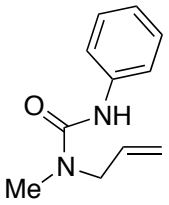
1-Allyl-3-{4-[benzyl(methyl)amino]phenyl}-1-methylurea (1a): A flame dried Schlenk flask equipped with a stirbar was cooled under a stream of nitrogen and charged with 1-allyl-3-(4-bromophenyl)-1-methylurea (1.00 g, 3.72 mmol), lithium bis(trimethylsilyl)amide (1.37 g, 4.46 mmol), $Pd_2(dba)_3$ (34.1 mg, 0.0372 mmol), and DavePhos (35.1 mg, 0.0893 mmol). The flask was purged with N_2 pressure for 30 s then THF (8.2 mL) and *N*-methyl benzylamine (0.58 mL, 4.46 mmol) were added. The

resulting mixture was heated to 65 °C with stirring for 15 h, then was cooled to rt. A solution of 1M HCl (8 mL) was added and the resulting mixture was stirred at rt for five min. A solution of saturated aqueous $NaHCO_3$ (8 mL) was slowly added and the mixture was transferred to a separatory funnel after bubbling ceased. The mixture was extracted with ethyl acetate (3 x 20 mL) then the combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel to afford 450 mg (40%) of the title compound as a light brown solid, mp 93–97 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31–7.14 (m, 7H), 6.68 (d, *J* = 9.0 Hz, 2H), 6.18 (s, br, 1H), 5.83 (ddt, *J* = 5.5, 5.2, 12.0 Hz, 1H), 5.26–5.19 (m, 2H), 4.46 (s, 2H), 3.93 (d, *J* = 5.5 Hz, 2H), 2.96 (s, 3H), 2.94 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.2, 146.5, 138.9, 133.6, 128.8, 128.4, 126.8, 126.7, 122.5, 116.7, 113.0, 57.0, 51.4, 38.7, 34.4; IR (film) 1637 cm^{-1}. MS (CI) 310.1916 (310.1914 calcd for $C_{19}H_{23}N_3O$, M + H^+).

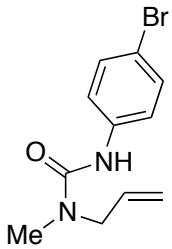


1-Allyl-3-(4-methoxyphenyl)-1-methylurea (1b): The reaction of *N*-allylmethylamine (0.47 mL, 4.92 mmol) with 4-methoxyphenyl isocyanate (0.58 mL, 4.47 mmol) according to the general procedure afforded 841 mg (85%) of the title compound as a white solid, mp 52–55 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (d, *J* = 9.0 Hz, 2H), 6.81 (d, *J* = 9.0 Hz, 2H), 6.61 (s, br, 1H), 5.90–5.78 (m, 1H), 5.25 (d, *J* = 5.5 Hz, 1H), 5.22 (s, 1H), 3.94 (d, *J* = 5.3 Hz, 2H), 3.76 (s, 3H), 2.97 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.8, 155.7,

133.5, 132.2, 122.1, 116.8, 114.0, 55.5, 51.5, 34.5; IR (film) 1638 cm^{-1}. MS (CI) 221.1280 (221.1285 calcd for $C_{12}H_{16}N_2O_2$, M + H^+).

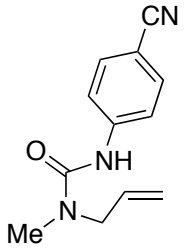


1-Allyl-1-methyl-3-phenylurea (1c): The reaction of *N*-allylmethylamine (0.37 mL, 3.85 mmol) with phenyl isocyanate (0.42 mL, 3.50 mmol) according to the general procedure afforded 644 mg (88%) of the title compound as a white solid, mp 71–74 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 7.8 Hz, 2H), 6.98 (t, *J* = 7.4 Hz, 1H), 6.48 (s, br, 1H), 5.88–5.78 (m, 1H), 5.26–5.19 (m, 2H), 3.93 (d, *J* = 5.5 Hz, 2H), 2.96 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.6, 139.2, 133.4, 128.8, 122.9, 119.8, 116.9, 51.5, 34.5; IR (film) 1639 cm^{-1}. MS (CI) 191.1180 (191.1179 calcd for $C_{11}H_{14}N_2O$, M + H^+).

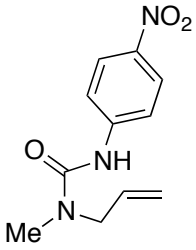


1-Allyl-3-(4-bromophenyl)-1-methylurea (1d): The reaction of *N*-allylmethylamine (0.80 mL, 8.44 mmol) with 4-bromophenyl isocyanate (1.51 g, 7.67 mmol) according to the general procedure afforded 1.88 g (91%) of the title compound as a white solid, mp 123–126 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.8 Hz, 2H), 7.23 (d, *J* = 8.9 Hz,

2H), 6.47 (s, br, 1H), 5.81 (ddt, *J* = 5.5, 5.6, 9.9 Hz, 1H), 5.27–5.19 (m, 2H), 3.92 (d, *J* = 5.3 Hz, 2H), 2.96 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.2, 138.3, 133.2, 131.7, 121.4, 117.0, 115.2, 51.5, 34.6; IR (film) 1634 cm^{-1}. MS (CI) 269.0282 (269.0284 calcd for $C_{11}H_{13}BrN_2O$, M + H^+).

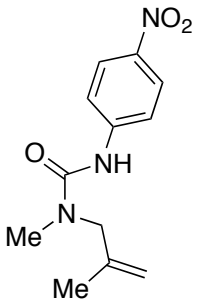


1-Allyl-3-(4-cyanophenyl)-1-methylurea (1e): The reaction of *N*-allylmethylamine (0.73 mL, 7.65 mmol) with 4-cyanophenyl isocyanate (1.00 g, 6.96 mmol) according to the general procedure afforded 1.17 g (78%) of the title compound as a white solid, mp 119–122 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (d, *J* = 8.8 Hz, 2H), 7.49 (d, *J* = 8.8 Hz, 2H), 6.69 (s, br, 1H), 5.86 (ddt, *J* = 5.1, 5.4, 11.9 Hz, 1H), 5.32–5.25 (m, 2H), 3.80 (d, *J* = 5.4 Hz, 2H), 3.03 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.6, 143.5, 133.1, 132.9, 119.2, 119.0, 117.4, 105.3, 51.6, 34.8; IR (film) 1664 cm^{-1}. MS (CI) 216.1135 (216.1131 calcd for $C_{12}H_{13}N_3O_3$, M + H^+).



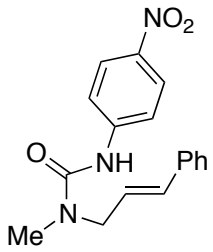
1-Allyl-1-methyl-3-(4-nitrophenyl)urea (1f): The reaction of *N*-allylmethylamine (0.77 mL, 8.09 mmol) with 4-nitrophenyl isocyanate (1.21 g, 7.35 mmol) according to the general procedure afforded 1.61 g (93%) of the title compound as a yellow solid, mp

78–81 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 9.2 Hz, 2H), 7.53 (d, *J* = 9.2 Hz, 2H), 7.00 (s, br, 1H), 5.84 (ddt, *J* = 5.3, 5.6, 11.4 Hz, 1H), 5.32–5.22 (m, 2H), 3.98 (d, *J* = 5.3 Hz, 2H), 3.02 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.7, 145.6, 142.2, 132.8, 125.0, 118.4, 117.4, 51.6, 34.8; IR (film) 1660 cm^{-1}. MS (CI) 236.1037 (236.1030 calcd for $C_{11}H_{13}N_3O_3$, M + H^+).



1-Methyl-1-(2-methylallyl)-3-(4-nitrophenyl)urea (3): A flame dried flask equipped with a stirbar was cooled under a stream of nitrogen and charged with 3-bromo-2-methylpropene (4.60 mL, 45 mmol). The flask was cooled to 0 °C and stirred for five min, then methylamine (27.2 mL, 225 mmol, 33% solution in EtOH) was added and the resulting mixture was warmed to rt and stirred for 15 h. A solution of 1M NaOH (20 mL) was added and the resulting mixture was transferred to a separatory funnel. The mixture was extracted with ether (3 x 20 mL) then the combined organic layers were washed with 1M NaOH (1x12 mL), dried over anhydrous Na_2SO_4, filtered, and partially concentrated *in vacuo* (to remove excess methylamine) to afford *N*,2-dimethylprop-2-en-1-ylamine as a solution in ethanol. The solution was transferred to a flask equipped with a stirbar and cooled to –10 °C. Neat 4-nitrophenyl isocyanate (1.64 g, 10 mmol) was added and the resulting solution and the reaction was slowly warmed to rt over the course of five h. The reaction mixture was then concentrated in vacuo and the crude

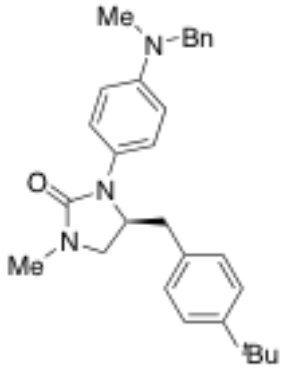
product was purified by flash column chromatography to yield 350 mg (14 %) of the title compound as a yellow solid, mp 79–82 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.13 (d, *J* = 9.3 Hz, 2H), 7.52 (d, *J* = 9.1 Hz, 2H), 6.92 (s, br, 1H), 5.03 (s, 1H) 4.96 (s, 1H), 3.90 (s, 2H), 3.05 (s, 3H), 1.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.7, 145.5, 142.3, 141.0, 125.0, 118.3, 112.5, 55.2, 35.3, 19.7; IR (film) 1658 cm^{-1}. MS (CI) 250.1191 (250.1186 calcd for $C_{12}H_{15}N_3O_3$, M + H^+).



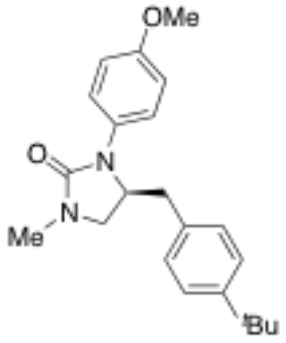
1-Cinnamyl-1-methyl-3-(4-nitrophenyl)urea (5): A flame dried flask equipped with a stirbar was cooled under a stream of nitrogen and charged with cinnamyl bromide (4.33 g, 22 mmol). The flask was cooled to 0 °C and stirred for five min, then methylamine (27.2 mL, 225 mmol, 33% solution in EtOH) was added and the resulting mixture was warmed to rt and stirred for 15 h. A solution of 1M NaOH (20 mL) was added and the resulting mixture was transferred to a separatory funnel. The mixture was extracted with ether (3 x 20 mL) then the combined organic layers were washed with 1M NaOH (1x12 mL), dried over anhydrous Na_2SO_4, filtered, and partially concentrated *in vacuo* (to remove excess methylamine) to afford *(E)-N*-methyl-3-phenylprop-2-en-1-ylamine as a solution in ethanol. The solution was transferred to a flask equipped with a stirbar and cooled to –10 °C. Neat 4-nitrophenyl isocyanate (1.44g, 8.8 mmol) was added and the resulting solution and the reaction was slowly warmed to rt over the course of five h. The reaction mixture was then concentrated in vacuo and the crude product was purified by flash column chromatography to yield 430 mg (16%) of the title compound as

a white solid, mp 120–124 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.14 (d, *J* = 9.1 Hz, 2H), 7.55 (d, *J* = 9.3 Hz, 2H), 7.40 (d, *J* = 7.3 Hz, 2H), 7.34 (t, *J* = 7.3 Hz, 2H), 7.28 (t, *J* = 7.1 Hz, 1H), 6.92 (s, br, 1H), 6.60 (d, *J* = 16.0 Hz, 1H), 6.23 (dt, *J* = 5.9, 15.9 Hz, 1H), 4.17 (d, *J*= 5.9 Hz, 2H), 3.10 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.5, 145.4, 142.4, 135.9, 132.8, 128.7, 128.2, 126.5, 125.1, 123.9, 118.4, 51.2, 34.8; IR (film) 1659 cm^{-1}. MS (CI) 312.1353 (312.1343 calcd for $C_{17}H_{17}N_3O_3$, M + H^+).

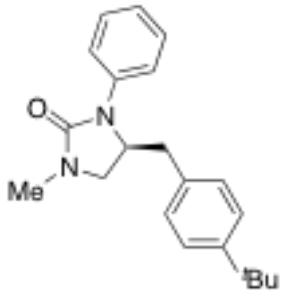
General procedure for asymmetric Pd-catalyzed carboamination reactions of *N*-allylurea derivatives. A flame-dried Schlenk tube equipped with a stirbar was cooled under a stream of nitrogen and then charged with $Pd_2(dba)_3$ (2 mol %), (*S*)-Siphos-PE (6 mol %), the urea substrate (1.0 equiv), and NaO^{t}Bu (2.0 equiv). The flask was purged with N_2, then the aryl or alkenyl halide (2.0 equiv), the additive (H_2O, 2.0 equiv; or TFA, 40 mol % if needed) and xylenes (0.20 M, for reactions at 120 °C) or toluene (0.20 M, for reactions at 90 °C) were added. The resulting mixture was heated to 90 °C or 120 °C with stirring until the starting material had been consumed as judged by TLC analysis. The reaction mixture was then cooled to rt, saturated aqueous ammonium chloride (6mL/mmol substrate) was added, and the mixture was transferred to a separatory funnel. The mixture was extracted with ethyl acetate (3 x 5 mL) then the combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel.



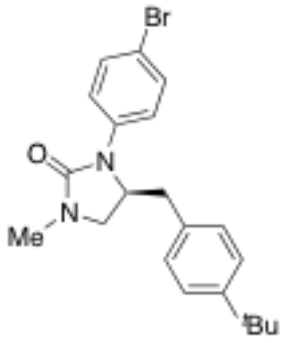
(–)-(4*S*)-3-{4-[Benzyl(methyl)amino]phenyl}-4-[4-(*tert*-butyl)benzyl]-1-methylimidazolidin-2-one (2a). The general procedure was employed for the coupling of 1-allyl-3-{4-[benzyl(methyl)amino]phenyl}-1-methylurea (0.10 mmol, 30.9 mg) and 4-bromo-*tert*-butylbenze (0.20 mmol, 42.6 mg), using a catalyst composed of $Pd_2(dba)_3$ (0.002 mmol, 1.8 mg) and (*S*)-Siphos-PE (0.006 mmol, 3.0 mg), a reaction temperature of 90 °C, and a reaction time of 12 h. This procedure afforded the title compound (28.7 mg**,** 65%) as an orange oil: $[\alpha]^{23}_{D}$ –23.2 (*c* 0.68, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34–7.23 (m, 9H), 7.05 (d, *J* = 8.2 Hz, 2H), 6.77 (d, *J* = 9.0 Hz, 2H), 4.52 (s, 2H), 4.28–4.20 (m, 1H), 3.30 (app. t, *J* = 8.6 Hz, 1H), 3.12 (dd, *J* = 6.1, 8.8 Hz, 1H), 3.06 (dd, *J* = 3.3, 13.7 Hz, 1H), 3.00 (s, 3H), 2.79 (s, 3H) 2.58 (dd, *J* = 10.0, 13.5 Hz, 1H), 1.30 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$); 159.5, 149.6, 147.3, 139.0, 133.8, 128.8, 128.6, 128.2, 126.9, 126.8, 125.5, 124.5, 112.9, 57.0, 55.9, 50.1, 38.7, 38.0, 34.4, 31.4, 31.3; IR (film) 1704 cm^{-1}; MS (CI) 442.2866 (442.2859 calcd for $C_{29}H_{35}N_3O$, M + H^+). The enantiopurity was determined to be 73% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.50 mL/min, λ 198 nm, RT= 6.1 and 9.2 min).



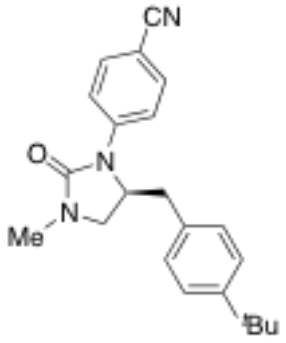
(–)-(4*S*)-4-[4-(*tert*-Butyl)benzyl]-3-(4-methoxyphenyl)-1-methylimidazolidin-2-one (2b). The general procedure was employed for the coupling of 1-allyl-3-(4-methoxyphenyl)-1-methylurea (0.10 mmol, 22.1 mg) and 4-bromo-*tert*-butylbenze (0.20 mmol, 42.6 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.002 mmol, 1.8 mg) and (*S*)-Siphos-PE (0.006 mmol, 3.0 mg), a reaction temperature of 90 °C, and a reaction time of 12 h. This procedure afforded the title compound (33.0 mg, 93%) as an orange oil: $[\alpha]^{23}_D$ –10.1 (*c* 0.76, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 9.0 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 7.06 (d, *J* = 8.3 Hz, 2H), 6.93 (d, *J* = 9.0 Hz, 2H), 4.37–4.29 (m, 1H), 3.82 (s, 3H), 3.36 (app. t, *J* = 8.7 Hz, 1H), 3.17 (dd, *J* = 5.9, 9.2 Hz, 1H), 3.05 (dd, *J* = 3.7, 13.7 Hz, 1H), 2.81 (s, 3H), 2.62 (dd, *J* = 9.8, 13.7 Hz, 1H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) 159.0, 156.4, 149.6, 133.5, 131.8, 128.8, 125.5, 123.8, 114.3, 55.5, 55.4, 49.8, 37.8, 34.4, 31.3, 31.2; IR (film) 1700 cm^{-1}; MS (CI) 353.2232 (353.2224 calcd for $C_{22}H_{28}N_2O_2$, M + H^+). The enantiopurity was determined to be 79% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.50 mL/min, λ 198 nm, RT= 5.3 and 8.1 min).



(–)-(4*S*)-4-[4-(*tert*-Butyl)benzyl]-1-methyl-3-phenylimidazolidin-2-one (2c). The general procedure was employed for the coupling of 1-allyl-1-methyl-3-phenylurea (0.10 mmol, 17.6 mg) and 4-bromo-*tert*-butylbenze (0.20 mmol, 42.6 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.002 mmol, 1.8 mg) and (*S*)-Siphos-PE (0.006 mmol, 3.0 mg), a reaction temperature of 90 °C and a reaction time of 12 h. This procedure afforded the title compound (29.0 mg**,** 90%) as white solid, mp 67–70 °C: $[\alpha]^{23}_D$ –19.4 (*c* 0.55, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.6 Hz, 2H), 7.37 (t, *J* = 7.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 7.12–7.06 (m, 3H), 4.48–4.40 (m, 1H), 3.37 (app. t, *J* = 8.6 Hz, 1H), 3.20 (dd, *J* = 4.9, 8.4 Hz, 1H), 3.11 (dd, *J* = 3.3, 13.8 Hz, 1H), 2.81 (s, 3H), 2.64 (dd, *J* = 9.8, 13.8 Hz, 1H), 1.30 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) 158.3, 149.7, 138.9, 133.5, 129.0, 128.8, 125.6, 123.4, 120.7, 54.4, 49.4, 37.5, 34.4, 31.3, 31.0; IR (film) 1707 cm^{-1}; MS (CI) 323.2125 (323.2118 calcd for $C_{21}H_{26}N_2O$, M + H^+). The enantiopurity was determined to be 78% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 8% IPA/Hexanes, 1.0 mL/min, λ 198 nm, RT= 9.5 and 10.1 min).

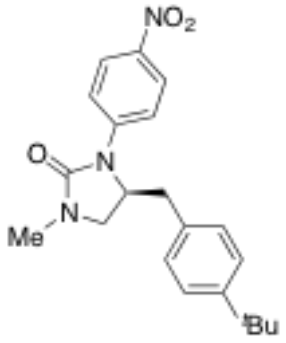


(–)-(4*S*)-3-(4-Bromophenyl)-4-[4-(*tert*-butyl)benzyl]-1-methylimidazolidin-2-one (2d). The general procedure was employed for the coupling of 1-allyl-3-(4-bromophenyl)-1-methylurea (0.10 mmol, 26.9 mg) and 4-bromo-*tert*-butylbenze (0.20 mmol, 42.6 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.002 mmol, 1.8 mg) and (*S*)-Siphos-PE (0.006 mmol, 3.0 mg), a reaction temperature of 90 °C and a reaction time of 12 h. This procedure afforded the title compound (18.0 mg**,** 45%) as an orange oil: $[\alpha]^{23}_D$ –41.6 (*c* 0.42, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49–7.43 (m, 4H), 7.32 (d, *J* = 8.3 Hz, 2H), 7.07 (d, *J* = 8.1 Hz, 2H), 4.44–4.38 (m, 1H), 3.39 (app. t, *J* = 8.8 Hz, 1H), 3.22 (dd, *J* = 4.6, 9.0 Hz, 1H), 3.07 (dd, *J* = 3.7, 13.8 Hz, 1H), 2.82 (s, 3H), 2.66 (dd, *J* = 9.5, 13.8 Hz, 1H), 1.32 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) 157.9, 150.0, 138.1, 133.3, 131.9, 128.8, 125.6, 121.9, 116.0, 54.2, 49.2, 37.4, 34.3, 31.3, 31.9; IR (film) 1708 cm^{-1}; MS (CI) 401.1230 (401.1223 calcd for $C_{21}H_{25}BrN_2O$, M + H^+). The enantiopurity was determined to be 83% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.50 mL/min, λ 198 nm, RT= 4.3 and 6.0 min).



(–)-(5*S*)-4-{5-[4-(*tert*-Butyl)benzyl]-3-methyl-2-oxoimidazolidin-1-yl}benzonitrile

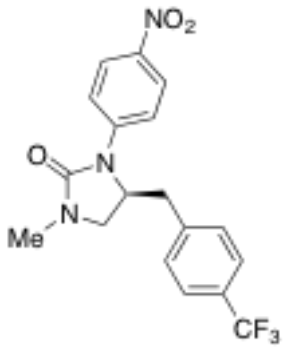
(2e): The general procedure was employed for the coupling of 1-allyl-3-(4-cyanophenyl)-1-methylurea (0.10 mmol, 21.5 mg) and 4-bromo-*tert*-butylbenze (0.20 mmol, 42.6 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.002 mmol, 1.8 mg) and (*S*)-Siphos-PE (0.006 mmol, 3.0 mg), a reaction temperature of 120 °C and a reaction time of 14 h. This procedure afforded the title compound (30.2 mg, 87%) as a light orange solid, mp 108–113 °C: $[\alpha]^{23}_{D}$ –73.6 (*c* 0.91, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 9.0 Hz, 2H), 7.63 (d, *J* = 9.0 Hz, 2H), 7.35 (d, *J* = 8.3 Hz, 2H), 7.10 (d, *J* = 8.3 Hz, 2H), 4.50 (m, 1H), 3.46 (app. t, *J* = 8.8 Hz, 1H), 3.28 (dd, *J* = 3.5, 9.2 Hz, 1H), 3.10 (dd, *J* = 3.5, 14.1 Hz, 1H), 2.83 (s, 3H) 2.73 (dd, *J* = 9.2, 14.0 Hz, 1H), 1.32 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.9, 150.2, 143.2, 133.1, 132.6, 128.8, 125.7, 119.2, 118.5, 105.0, 53.6, 48.7, 37.3, 34.5, 31.3, 30.8; IR (film) 1712 cm^{-1}; MS (CI) 348.2082 (348.2070 calcd for $C_{22}H_{25}N_3O$, M + H^+). The enantiopurity was determined to be 86% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 198 nm, RT= 5.4 and 8.6 min).



(–)-(4*S*)-4-[4-(*tert*-Butyl)benzyl]-1-methyl-3-(4-nitrophenyl)imidazolidin-2-one (2f). The general procedure was employed for the coupling of 1-allyl-1-methyl-3-(4-nitrophenyl)urea (0.20 mmol, 47.0 mg) and 4-bromo-tert-butylbenzene (0.40 mmol, 85.2 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.004 mmol, 3.7 mg) and (*S*)-Siphos-PE (0.012 mmol 6.1 mg), a reaction temperature of 115 °C and a reaction time of 18 h. This procedure afforded the title compound (59.6 mg, 81%) and 91% ee as a bright yellow solid, mp 115–118 °C: $[\alpha]^{23}_D$ –102.3 (*c* 1.49, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, *J* = 9.2 Hz, 2H), 7.78 (d, *J* = 9.4 Hz, 2H), 7.34 (d, *J* = 8.2 Hz, 2H), 7.9 (d, *J* = 8.2 Hz, 2H), 4.56–4.49 (m, 1H), 3.48 (app. t, *J* = 9.0 Hz, 1H), 3.29 (dd, *J* = 3.0, 9.3 Hz, 1H), 3.11 (dd, *J* = 3.5, 13.8 Hz, 1H), 2.83 (s, 3H), 2.75 (dd, *J* = 9.0, 13.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.7, 150.3, 145.2, 141.9, 132.5, 128.8, 125.8, 125.0, 117.7, 53.7, 48.6, 37.3, 34.5, 31.3, 30.8; IR (film) 1717 cm^{-1}. MS (CI) 368.1968 (368.1969 calcd for $C_{21}H_{25}N_3O_3$, M + H^+). The enantiopurity was determined to be 92% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.50 mL/min, λ 198 nm, RT= 6.1 and 9.4 min).

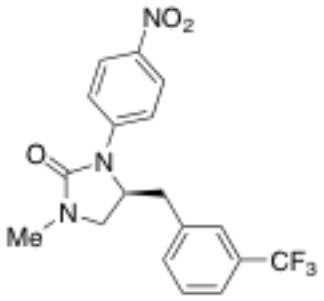
(–)-(4*S*)-4-[4-(*tert*-Butyl)benzyl]-1-methyl-3-(4-nitrophenyl)imidazolidin-2-one (X). The general procedure was employed for the coupling of 1-allyl-1-methyl-3-(4-nitrophenyl)urea (0.10 mmol, 23.5 mg) and 4-iodo-*tert*-butylbenzene (0.20 mmol, 52.0

mg) using a catalyst composed of $Pd_2(dba)_3$ (0.002 mmol, 1.8 mg) and (*S*)-Siphos-PE (0.006 mmol, 3.0 mg), a reaction temperature of 115 °C and a reaction time of 18 h. This procedure afforded the title compound (19.0 mg, 60%) as a bright yellow solid. This material contained ca 35% of an inseparable unidentified regioisomer. The enantiopurity was determined to be 47% ee by chiral HPLC analysis. Spectroscopic data were identical to those reported above.



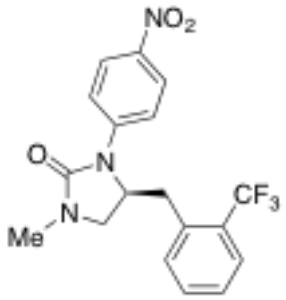
(–)-(4*S*)-1-Methyl-3-(4-nitrophenyl)-4-[4-(trifluoromethyl)benzyl]imidazolidin-2-one (2g). The general procedure was employed for the coupling of 1-allyl-1-methyl-3-(4-nitrophenyl)urea (0.20 mmol, 47.0 mg) and 4-bromobenzotrifluoride (0.40 mmol, 90.0 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.004 mmol, 3.7 mg) and (*S*)-Siphos-PE (0.012 mmol 6.1 mg), H_2O (0.40 mmol, 7 μL) as an additive, a reaction temperature of 115 °C and a reaction time of 18 h. This procedure afforded the title compound (54.6 mg**,** 72%) as a bright yellow solid, mp 161–164 °C: $[\alpha]^{23}_D$ –75.1 (*c* 11.5, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, *J* = 9.2 Hz, 2H), 7.78 (d, *J* = 9.3 Hz, 2H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 4.64–4.58 (m, 1H), 3.51 (app. t, *J* = 9.0 Hz, 1H), 3.24 (dd, *J* = 3.1, 9.3 Hz, 1H), 3.17 (dd, *J* = 3.1, 9.3 Hz, 1H), 3.01 (dd, *J* = 8.6, 14.0 Hz, 1H), 2.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.5, 144.8, 142.1, 139.6, 129.6 (q,

65.2 Hz), 129.5, 125.8 (q, 3.81), 125.1, 123.9 (q, 272.0 Hz), 117.8, 53.2, 48.3, 37.5, 30.7; IR (film) 1708 cm^{-1}. MS (CI) 380.1211 (380.1217 calcd for $C_{18}H_{16}F_3N_3O_3$, M + H^+). The enantiopurity was determined to be 95% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 195 nm, RT= 9.3 and 16.8 min).



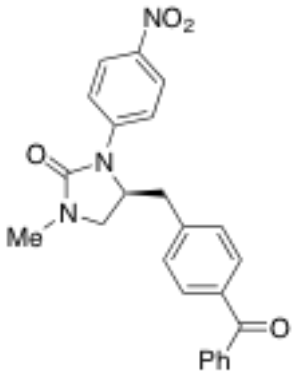
(–)-(4*S*)-1-Methyl-3-(4-nitrophenyl)-4-[3-(trifluoromethyl)benzyl]imidazolidin-2-one (2h). The general procedure was employed for the coupling of 1-allyl-1-methyl-3-(4-nitrophenyl)urea (0.20 mmol, 47.0 mg) and 3-bromobenzotrifluoride (0.40 mmol, 90.0 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.004 mmol, 3.7 mg) and (*S*)-Siphos-PE (0.012 mmol 6.1 mg), H_2O (0.40 mmol, 7 μL) as an additive, a reaction temperature of 115 °C and a reaction time of 18 h. This procedure afforded the title compound (53.1 mg, 70%) as a bright yellow solid, mp 145–148 °C: $[\alpha]^{23}_D$ –64.1 (*c* 1.16, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, *J* = 9.0 Hz, 2H), 7.77 (d, *J* = 9.0 Hz, 2H), 7.53 (d, *J* = 7.8 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 1H), 7.38 (s, 1H), 7.32 (d, *J* = 7.6 Hz, 1H), 4.65–4.58 (m, 1H), 3.53 (app. t, *J* = 9.0 Hz, 1H), 3.24 (dd, *J* = 2.7, 9.2 Hz, 1H), 3.15 (dd, *J* = 3.5, 14.1 Hz, 1H), 2.93 (dd, *J* = 8.4, 14.0 Hz, 1H), 2.79 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 156.5, 144.9, 142.1, 136.1, 132.7, 131.2 (q, *J* = 32.4 Hz), 129.4, 125.8 (q, *J* = 3.8 Hz), 125.1, 124.2 (q, *J* = 3.8 Hz), 123.9 (q, *J* = 271.8 Hz), 117.8, 53.1, 48.4, 37.6, 30.7; IR

(film) 1714 cm^{-1}. MS (CI) 380.1224 (380.1217 calcd for $C_{18}H_{16}F_3N_3O_3$, M + H^+). The enantiopurity was determined to be 88% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 195 nm, RT= 7.7 and 13.0 min).



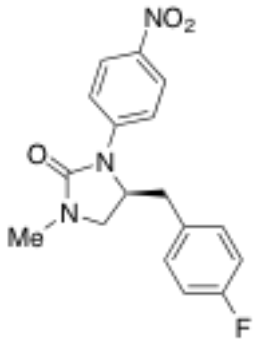
(–)-(4*S*)-1-Methyl-3-(4-nitrophenyl)-4-[2-(trifluoromethyl)benzyl]imidazolidin-2-one (2i). The general procedure was employed for the coupling of 1-allyl-1-methyl-3-(4-nitrophenyl)urea (0.20 mmol, 47.0 mg) and 2-bromobenzotrifluoride (0.40 mmol, 90.0 mg), using a catalyst composed of $Pd_2(dba)_3$ (0.004 mmol, 3.7 mg) and (*S*)-Siphos-PE (0.012 mmol 6.1 mg), H_2O (0.40 mmol, 7 μL) as an additive, a reaction temperature of 115 °C and a reaction time of 18 h. This procedure afforded the title compound (42.5 mg, 56%) as a bright yellow solid, mp 70–73 °C: $[\alpha]^{23}_D$ –98.7 (*c* 0.72, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 9.0 Hz, 2H), 7.72–7.64 (m, 3H), 7.44 (t, *J* = 7.4 Hz, 1H), 7.34 (t, *J* = 7.6 Hz, 1H), 7.26 (d, *J* = 8.6 Hz, 1H), 4.72–4.64 (m, 1H), 3.43 (app. t, *J* = 9.0 Hz, 1H), 3.36 (dd, *J* = 5.1, 14.4 Hz, 1H), 3.20 (dd, *J* = 2.0, 9.3 Hz, 1H), 2.96 (dd, *J* = 9.0, 14.3 Hz, 1H), 2.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.7, 145.1, 142.0, 134.4, 132.1, 129.2 (q, *J* = 30.0 Hz), 127.5, 126.6 (q, *J* = 5.7 Hz), 124.8, 124.4 (q, *J* = 272.3 Hz), 118.0, 53.1, 48.3, 35.0, 30.9 (one peak is missing due to incidental equivalence); IR (film) 1722 cm^{-1}. MS (CI) 380.1226 (380.1217 calcd for $C_{18}H_{16}F_3N_3O_3$, M + H^+). The

enantiopurity was determined to be 83% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 195 nm, RT= 7.9 and 12.5 min).



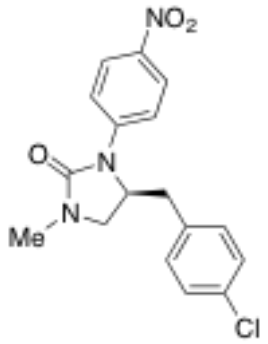
(–)-(4*S*)-4-(4-Benzoylbenzyl)-1-methyl-3-(4-nitrophenyl)imidazolidin-2-one (2j). The general procedure was employed for the coupling of 1-allyl-1-methyl-3-(4-nitrophenyl)urea (0.20 mmol, 47.0 mg) and 4-bromobenzophenone (0.40 mmol, 104.4 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.004 mmol, 3.7 mg) and (*S*)-Siphos-PE (0.012 mmol 6.1 mg), H_2O (0.40 mmol, 7 μL) as an additive, a reaction temperature of 115 °C and a reaction time of 18 h. This procedure afforded the title compound (68.1 mg**,** 82%) as a bright yellow solid, mp 115–118 °C: $[\alpha]^{23}_D$ –53.7 (*c* 0.97, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, *J* = 9.3 Hz, 2H), 7.80–7.72 (m, 6H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 2H), 7.26 (d, *J* = 8.2 Hz, 2H), 4.65–4.58 (m, 1H), 3.50 (app. t, *J* = 9.0 Hz, 1H), 3.26 (dd, *J* = 3.1, 9.2 Hz, 1H), 3.18 (dd, *J* = 3.5, 14.0 Hz, 1H), 2.91 (dd, *J* = 8.8, 13.9 Hz, 1H), 2.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.1, 156.6, 145.0, 142.1, 140.3, 137.3, 136.6, 132.6, 130.6, 130.0, 129.2, 128.4, 125.1, 117.8, 53.2, 48.4, 37.8, 34.4, 30.8; IR (film) 1715, 1657 cm^{-1}. MS (CI) 416.1620 (416.1605 calcd for $C_{24}H_{21}N_3O_4$, M + H^+). The enantiopurity was determined to be 86% ee by chiral HPLC

analysis (ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 195 nm, RT= 39.0 and 54.9 min.)

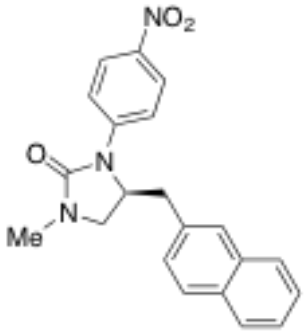


(–)-(4*S*)-4-(4-Fluorobenzyl)-1-methyl-3-(4-nitrophenyl)imidazolidin-2-one (2k). The general procedure was employed for the coupling of 1-allyl-1-methyl-3-(4-nitrophenyl)urea (0.20 mmol, 47.0 mg) and 4-bromofluorobenzene (0.40 mmol, 70.0 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.004 mmol, 3.7 mg) and (*S*)-Siphos-PE (0.012 mmol 6.1 mg), H_2O (0.40 mmol, 7 μL) as an additive, a reaction temperature of 115 °C and a reaction time of 18 h. This procedure afforded the title compound (43.0 mg**,** 65%) as a bright yellow solid, mp 153–157 °C: $[\alpha]^{23}_D$ –75.4 (*c* 1.10, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, *J* = 9.3 Hz, 2H), 7.88 (d, *J* = 9.3 Hz, 2H), 7.14–7.08 (m, 2H), 7.01 (app. t, *J* = 8.6 Hz, 2H), 4.58–4.51 (m, 1H), 3.49 (app. t, *J* = 9.0 Hz, 1H), 3.24 (dd, *J* = 3.1, 9.2 Hz, 1H), 3.06 (dd, *J* = 3.3, 14.0 Hz, 1H), 2.86–2.75 (m, 1H), 2.77 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.1 (d, *J* = 245.0 Hz), 156.6 145.0, 142.0, 131.1 (d, *J*= 3.5 Hz), 130.7 (d, *J* = 8.0 Hz), 125.1, 117.7, 115.8 (d, *J* = 21.3 Hz), 53.4, 48.2, 36.8, 30.7; IR (film) 1717 cm^{-1}. MS (CI) 330.1258 (330.1248 calcd for $C_{17}H_{16}FN_3O_3$, M + H^+). The enantiopurity was determined to be 94% ee by chiral HPLC

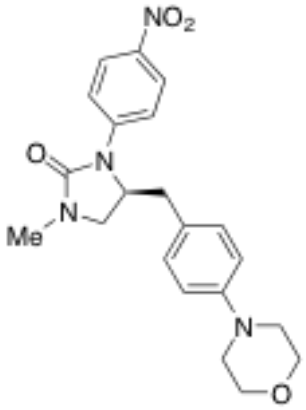
analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 198 nm, RT= 12.0 and 22.7 min).



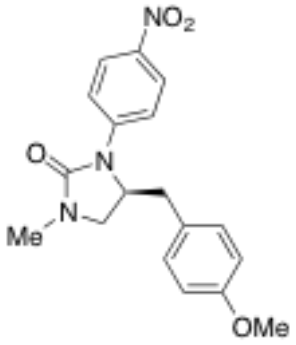
(–)-(4*S*)-4-(4-Chlorobenzyl)-1-methyl-3-(4-nitrophenyl)imidazolidin-2-one (2l). The general procedure was employed for the coupling of 1-allyl-1-methyl-3-(4-nitrophenyl)urea (0.20 mmol, 47.0 mg) and 4-bromochlorobenzene (0.40 mmol, 76.6 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.004 mmol, 3.7 mg) and (*S*)-Siphos-PE (0.012 mmol 6.1 mg), H_2O (0.40 mmol, 7 μL) as an additive, a reaction temperature of 115 °C and a reaction time of 18 h. This procedure afforded the title compound (51.2 mg**,** 74%) as a bright yellow solid, mp 144–147 °C: $[\alpha]^{23}_D$ –72.9 (*c* 0.81, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (d, *J* = 9.3 Hz, 2H), 7.78 (d, *J* = 9.3 Hz, 2H), 7.30 (d, *J* = 8.3 Hz, 2H), 7.08 (d, *J* = 8.2 Hz, 2H), 4.57–4.50 (m, 1H), 3.48 (app. t, *J* = 9.0 Hz, 1H), 3.23 (dd, *J* = 3.1, 9.1 Hz, 1H), 3.08 (dd, *J* = 3.3, 14.0 Hz, 1H), 2.85–2.78 (m, 1H), 2.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.6, 144.9, 142.1, 133.8, 133.3, 130.5, 129.0, 125.1, 117.7, 53.3, 48.2, 37.0, 34.4; IR (film) 1716 cm^{-1}. MS (CI) 346.0956 (346.0953 calcd for $C_{17}H_{16}ClN_3O_3$, M + H^+). The enantiopurity was determined to be 93% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 195 nm, RT= 12.5 and 22.7 min).



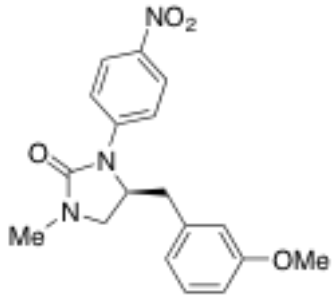
(–)-(4*S*)-1-Methyl-4-(naphthalen-2-ylmethyl)-3-(4-nitrophenyl)imidazolidin-2-one (2n). The general procedure was employed for the coupling of 1-allyl-1-methyl-3-(4-nitrophenyl)urea (0.20 mmol, 47.0 mg) and 2-bromonaphthalene (0.40 mmol, 82.8 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.004 mmol, 3.7 mg) and (*S*)-Siphos-PE (0.012 mmol 6.1 mg), a reaction temperature of 115 °C and a reaction time of 18 h. This procedure afforded the title compound (51.3 mg, 71%) as a bright yellow solid, mp 152–155 °C: $[\alpha]^{23}_D$ –116.7 (*c* 0.74, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (d, *J* = 9.1 Hz, 2H), 7.86–7.78 (m, 5H), 7.62 (s, 1H), 7.54–7.46 (m, 2H), 7.28 (d, *J* = 8.4 Hz, 1H), 4.68–4.61 (m, 1H), 3.46 (app. t, *J* = 9.2 Hz, 1H), 3.36–3.28 (m, 2H), 2.93 (dd, *J* = 9.3, 13.9 Hz, 1H), 2.81 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.7, 145.1, 142.0, 133.4, 133.0, 132.4, 128.7, 128.0, 127.7, 127.4, 126.9, 126.5, 126.0, 125.0, 117.8, 53.6, 48.5, 37.9, 30.8; IR (film) 1715 cm^{-1}. MS (CI) 362.1507 (362.1499 calcd for $C_{21}H_{19}N_3O_3$, M + H^+). The enantiopurity was determined to be 89% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 198 nm, RT= 9.4 and 17.4 min).



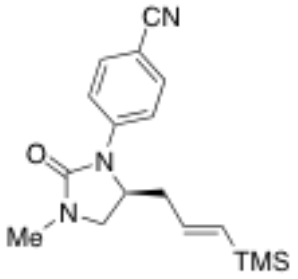
(–)-(4*S*)-1-Methyl-4-(4-morpholinobenzyl)-3-(4-nitrophenyl)imidazolidin-2-one (2o). The general procedure was employed for the coupling of 1-allyl-1-methyl-3-(4-nitrophenyl)urea (0.20 mmol, 47.0 mg) and 4-(4-bromophenyl)morpholine (0.40 mmol, 96.8 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.004 mmol, 3.7 mg) and (*S*)-Siphos-PE (0.012 mmol 6.1 mg), H_2O (0.40 mmol, 7 μL) as an additive, a reaction temperature of 115 °C and a reaction time of 18 h. This procedure afforded the title compound (63.4 mg, 80%) as a bright yellow solid, mp 125–129 °C: $[\alpha]^{23}_D$ –91.0 (*c* 0.97, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, *J* = 9.2 Hz, 2H), 7.78 (d, *J* = 9.2 Hz, 2H), 7.05 (d, *J* = 8.4 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 2H), 4.53–4.45 (m, 1H), 3.90–3.81 (m, 4H), 3.46 (app. t, *J* = 8.9 Hz, 1H), 3.27 (dd, *J* = 2.9, 9.2 Hz, 1H), 3.18–3.08 (m, 4H), 3.04 (dd, *J* = 3.2, 14.1 Hz, 1H), 2.81 (s, 3H), 2.72 (dd, *J* = 8.8, 14.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 156.7, 150.4, 145.2, 141.9, 130.0, 126.6, 125.0, 117.7, 115.9, 67.1, 53.8, 49.3, 48.4, 36.8, 30.8; IR (film) 1717 cm^{-1}. MS (CI) 397.1872 (397.1870 calcd for $C_{21}H_{24}N_4O_4$ $M+H^+$). The enantiopurity was determined to be 87% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 195 nm, RT= 25.7 and 33.0 min).



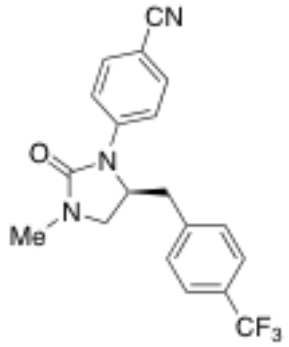
(–)-(4*S*)-4-(4-Methoxybenzyl)-1-methyl-3-(4-nitrophenyl)imidazolidin-2-one (2p). The general procedure was employed for the coupling of 1-allyl-1-methyl-3-(4-nitrophenyl)urea (0.20 mmol, 47.0 mg) and 4-bromoanisole (0.40 mmol, 74.8 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.004 mmol, 3.7 mg) and (*S*)-Siphos-PE (0.012 mmol 6.1 mg), TFA (0.08 mmol, 6 μL) as an additive, a reaction temperature of 115 °C and a reaction time of 18 h. This procedure afforded the title compound (55.3 mg, 81%) as a bright yellow solid, mp 110–113 °C: $[\alpha]^{23}_D$ –71.3 (*c* 0.98, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, *J* = 9.2 Hz, 2H), 7.79 (d, *J* = 9.4 Hz, 2H), 7.06 (d, *J* = 8.5 Hz, 2H), 6.85 (d, *J* = 8.5 Hz, 2H), 4.52–4.48 (m, 1H), 3.77 (s, 3H), 3.46 (app. t, *J* = 8.9 Hz, 1H), 3.26 (dd, *J* = 3.2, 9.0 Hz, 1H), 3.04 (dd, *J* = 3.4, 14.1 Hz, 1H), 2.79 (s, 3H), 2.74 (dd, *J* = 8.9, 14.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8, 156.7, 145.2, 141.9, 130.2, 127.3, 125.0, 117.7, 114.3, 55.3, 53.7, 48.3, 36.8, 30.8; IR (film) 1717 cm^{-1}. MS (CI) 342.1457 (342.1448 calcd for $C_{18}H_{19}N_3O_4$, M + H^+). The enantiopurity was determined to be 90% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 195 nm, RT= 14.1 and 25.9 min.)



(–)-(4*S*)-4-(3-Methoxybenzyl)-1-methyl-3-(4-nitrophenyl)imidazolidin-2-one (2q). The general procedure was employed for the coupling of 1-allyl-1-methyl-3-(4-nitrophenyl)urea (0.20 mmol, 47.0 mg) and 3-bromoanisole (0.40 mmol, 74.8 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.004 mmol, 3.7 mg) and (*S*)-Siphos-PE (0.012 mmol 6.1 mg), a reaction temperature of 115 °C and a reaction time of 18 h. This procedure afforded the title compound (51.2 mg, 75%) as a bright yellow solid, mp 110–114 °C: $[\alpha]^{23}_D$ –94.2 (*c* 0.73, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (d, *J* = 9.1 Hz, 2H), 7.77 (d, *J* = 9.3 Hz, 2H), 7.23 (t, *J* = 8.0 Hz, 1H), 6.78 (d, *J* = 8.4 Hz, 1H), 6.73 (d, *J* = 7.6 Hz, 1H), 6.65 (s, 1H), 4.56–4.48 (m, 1H), 3.76 (s, 1H), 3.44 (app. t, *J* = 9.0 Hz, 1H), 3.26 (dd, *J* = 2.9, 9.2 Hz, 1H), 3.08 (dd, *J* = 3.3, 13.9 Hz, 1H), 2.79 (s, 3H), 2.73 (dd, *J* = 9.0, 13.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.9, 156.7, 145.1, 141.9, 137.1, 129.9, 125.0, 121.4, 117.6, 115.4, 112.0, 55.2, 53.5, 48.4, 37.7, 30.8; IR (film) 1716 cm^{-1}. MS (CI) 342.1461 (342.1448 calcd for $C_{18}H_{19}N_3O_4$, M + H^+). The enantiopurity was determined to be 83% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 195 nm, RT= 12.6 and 19.6 min).



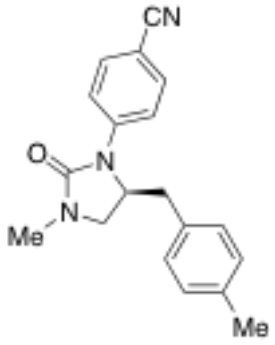
(+)-(*E*,5*S*)-4-(3-Methyl-2-oxo-5-(3-(trimethylsilyl)allyl)imidazolidin-1-yl)benzonitrile (2r). The general procedure was employed for the coupling of 1-allyl-3-(4-cyanophenyl)-1-methylurea (0.10 mmol, 21.5 mg) and 2-bromovinyltrimethylsilane (0.20 mmol, 35.8 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.002 mmol, 1.8 mg) and (*S*)-Siphos-PE (0.006 mmol, 3.0 mg), a reaction temperature of 120 °C and a reaction time of 14 h. This procedure afforded the title compound (20.0 mg, 64%) as a light orange solid, mp 130–133 °C: $[\alpha]^{23}_D$ +6.4 (*c* 0.50, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 9.0 Hz, 2H), 7.59 (d, *J* = 8.8 Hz, 2H), 5.87 (dt, *J* = 6.2, 18.5 Hz, 1H), 5.76 (d, *J* = 18.7 Hz, 1H), 4.35 (m, 1H), 3.56 (app. t, *J* = 9.0 Hz, 1H), 3.23 (dd, *J* = 3.4, 9.1 Hz, 1H), 2.88 (s, 3H) 2.53 (m, 1H), 2.38 (m, 1H) 0.04 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.6, 143.1, 138.8, 136.5, 133.0, 127.5, 119.1, 118.6, 105.0, 51.6, 48.8, 38.9, 30.8, –1.4; IR (film) 1702 cm^{-1}; MS (CI) 314.1685 (314.1683 calcd for $C_{17}H_{23}N_3OSi$, M + H^+). The enantiopurity was determined to be 86% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 200 nm, RT= 3.9 and 5.1 min).



(–)-(5*S*)-4-{3-Methyl-2-oxo-5-[4-(trifluoromethyl)benzyl]imidazolidin-1-yl}benzonitrile (2s). The general procedure was employed for the coupling of 1-allyl-3-(4-cyanophenyl)-1-methylurea (0.10 mmol, 21.5 mg) and 4-bromobenzotrifluoride (0.20 mmol, 45.0 mg), using a catalyst composed of $Pd_2(dba)_3$ (0.002 mmol, 1.8 mg) and (*S*)-Siphos-PE (0.006 mmol, 3.0 mg), water (3.5 µL, 0.20 mmol) as an additive, a reaction temperature of 120 °C and a reaction time of 14 h. This procedure afforded the title compound (20.7 mg**,** 58%) as a light orange solid, mp 120–124 °C: $[\alpha]^{23}_{D}$ –29.5 (*c* 1.12, CH_2Cl_2); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.72 (d, *J*= 8.9 Hz, 2H), 7.64 (d, *J*= 8.9 Hz, 2H), 7.57 (d, *J*= 8.0 Hz, 2H), 7.27 (d, *J*= 8.2 Hz, 2H), 4.56-4.52 (m, 1H), 3.46 (app. t, *J*= 9.0 Hz, 1H), 3.20 (dd, *J*= 3.4, 9.0 Hz, 1H), 3.13 (dd, *J*= 3.3, 14.1 Hz, 1H), 2.97 (dd, *J*= 8.7, 14.0 Hz, 1H), 2.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.7, 142.9, 139.7, 133.2, 129.7 (q, 32.4 Hz), 129.6, 125.8(q, 3.8 Hz), 124.0 (q, 272.0 Hz), 119.1, 118.7, 105.5, 53.0, 48.4, 37.5, 30.7 ; IR (film) 1717 cm^{-1}; MS (CI) 360.1322 (360.1318 calcd for $C_{19}H_{16}F_3N_3O$, M + H^+). The enantiopurity was determined to be 77% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 200nm, RT= 8.9 and 16.6 min).

(–)-5*S*-4-{3-Methyl-2-oxo-5-[4-(trifluoromethyl)benzyl]imidazolidin-1-yl}benzonitrile. (2s): The general procedure was employed for the coupling of 1-allyl-3-

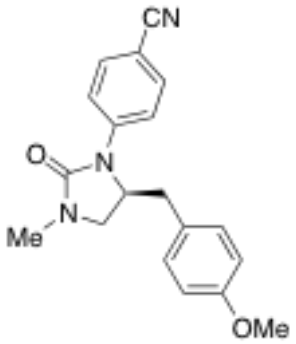
(4-cyanophenyl)-1-methylurea (0.10 mmol, 21.5 mg) and 4-iodobenzotrifluoride (0.20 mmol, 54.4 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.002 mmol, 1.8 mg) and (*S*)-Siphos-PE (0.006 mmol, 3.0 mg), water (3.5 µL, 0.20 mmol) as an additive, a reaction temperature of 120 °C and a reaction time of 14 h. This procedure afforded the title compound (27.9 mg, 77%) as a light orange oil. The enantiopurity was determined to be 80% ee by chiral HPLC analysis. Spectroscopic data were identical to those reported above.



(–)-(5*S*)-4-[3-Methyl-5-(4-methylbenzyl)-2-oxoimidazolidin-1-yl]benzonitrile (2t). The general procedure was employed for the coupling of 1-allyl-3-(4-cyanophenyl)-1-methylurea (0.10 mmol, 21.5 mg) and 4-bromotoluene (0.20 mmol, 34.2 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.002 mmol, 1.8 mg) and (*S*)-Siphos-PE (0.006 mmol, 3.0 mg), a reaction temperature of 120 °C and a reaction time of 14 h. This procedure afforded the title compound (26.3 mg, 86%) as a light orange solid, mp 123–126 °C: $[\alpha]^{23}_D$ –55.7 (*c* 0.90, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 (d, *J* = 9.0 Hz, 2H), 7.65 (d, *J* = 9.0 Hz, 2H), 7.15 (d, *J* = 7.8 Hz, 2H), 7.05 (d, *J* = 8.0 Hz, 2H), 4.51–4.45 (m, 1H), 3.44 (app. t, *J* = 9.0 Hz, 1H), 3.26 (dd, *J* = 3.7, 9.2 Hz, 1H), 3.09 (dd, *J* = 3.4, 13.9 Hz, 1H), 2.82 (s, 3H) 2.72 (dd, *J* = 9.3, 13.9 Hz, 1H), 2.35 (s, 3H); ^{13}C NMR (100 MHz,

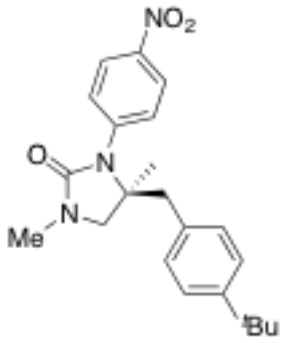
$CDCl_3$) δ 156.9, 143.2, 136.9, 133.1, 132.5, 129.5, 129.0, 119.2, 118.5, 105.0, 53.5, 48.5, 37.2, 30.8, 21.0; IR (film) 1712 cm^{-1}; MS (CI) 306.1608 (306.1601 calcd for $C_{19}H_{19}N_3O$, M + H^+). The enantiopurity was determined to be 85% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 198 nm, RT= 5.4 and 8.6 min).

(–)-(5*S*)-4-[3-Methyl-5-(4-methylbenzyl)-2-oxoimidazolidin-1-yl]benzonitrile (2t): The general procedure was employed for the coupling of 1-allyl-3-(4-cyanophenyl)-1-methylurea (0.10 mmol, 21.5 mg) and 4-iodotoluene (0.20 mmol, 43.6 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.002 mmol, 1.8 mg) and (*S*)-Siphos-PE (0.006 mmol, 3.0 mg), a reaction temperature of 120 °C and a reaction time of 14 h. This procedure afforded the title compound (27.2 mg, 89%) as a light orange oil. The enantiopurity was determined to be 73% ee by chiral HPLC analysis. Spectroscopic data were identical to those reported above.



(–)-(5*S*)-4-[5-(4-Methoxybenzyl)-3-methyl-2-oxoimidazolidin-1-yl]benzonitrile (2u). The general procedure was employed for the coupling of 1-allyl-3-(4-cyanophenyl)-1-methylurea (0.10 mmol, 21.5 mg) and 4-bromoanisole (0.20 mmol, 37.4 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.002 mmol, 1.8 mg) and (S)-Siphos-PE (0.006 mmol,

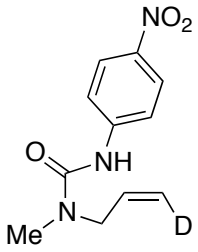
3.0 mg), a reaction temperature of 120 °C and a reaction time of 14 h. This procedure afforded the title compound in (23.5 mg, 73%) as a light orange solid, mp 87–91 °C: $[\alpha]^{23}_{D}$ –52.9 (*c* 0.37, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 9.0 Hz, 2H), 7.63 (d, *J* = 8.8 Hz, 2H), 7.06 (d, *J* = 8.6 Hz, 2H), 6.85 (d, *J* = 8.6 Hz, 2H), 4.46 (m, 1H), 3.79 (s, 3H), 3.43 (app. t, *J* = 9.0 Hz, 1H), 3.24 (dd, *J* = 3.3, 9.2 Hz, 1H), 3.04 (dd, *J* = 3.3, 14.0 Hz, 1H), 2.80 (s, 3H) 2.71 (dd, *J* = 9.0, 14.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.4, 156.9, 143.2, 133.1, 130.2, 127.5, 118.5, 114.2, 105.0, 55.3, 53.5, 48.5, 36.7, 30.8; IR (film) 1711 cm^{-1}; MS (CI) 322.1548 (322.1550 calcd for $C_{19}H_{19}N_3O_2$, M + H^+). The enantiopurity was determined to be 82% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 198 nm, RT= 12.3 and 23.3 min).



(+)-(4*S*)-4-[4-(*tert*-Butyl)benzyl]-1,4-dimethyl-3-(4-nitrophenyl)imidazolidin-2-one (5): The general procedure was employed for the coupling of 1-methyl-1-(2-methylallyl)-3-(4-nitrophenyl)urea (0.10 mmol, 24.9 mg) and 4-bromo-*tert*-butylbenzene (0.20 mmol, 42.6 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.002 mmol, 1.8 mg) and (*S*)-Siphos-PE (0.006 mmol, 3.0mg), a reaction temperature of 135 °C and a reaction time of 18 h. This procedure afforded the title compound (27.5 mg, 72%) as a yellow oil: $[\alpha]^{23}_{D}$ +70.2 (*c* 0.90, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.24 (d, *J* = 9.1 Hz, 2H), 7.54 (d, *J* = 9.1

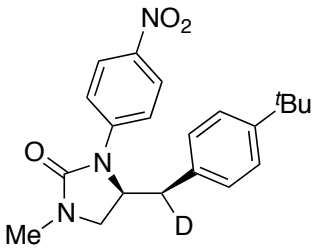
Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 7.03 (d, *J* = 8.1 Hz, 2H), 3.49 (d, *J* = 9.1 Hz, 1H), 3.11 (d, *J* = 13.5 Hz, 1H), 3.23 (d, *J* = 8.8 Hz, 1H), 2.81 (d, *J* = 14.4 Hz, 1H), 2.74 (s, 3H), 1.47, (s, 3H), 1.29 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.0, 150.2, 144.5, 144.3, 132.3, 129.7, 125.6, 125.4, 124.4, 61.5, 56.3, 44.2, 34.5, 313, 30.6, 25.1; IR (film) 1716 cm^{-1}; MS (CI) 382.2133 (382.2125 calcd for $C_{22}H_{27}N_3O_3$, M + H^+). The enantiopurity was determined to be 76% ee by chiral HPLC analysis (ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 198 nm, RT= 7.5 and 7.9 min.)

Deuterium Labeling Studies:



(*Z*)-1-(3-*d*-Allyl)-1-methyl-3-(4-nitrophenyl)urea ((*Z*)-*d*-1f):[2] A flame dried round bottom flask equipped with a stir bar was cooled to rt under a stream of N_2 and charged with *N*-methylallylamine (5.0 mmol, 0.47 mL) and Et_2O (10 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 (3.12 mL, 1.6 M, 5 mmol) was added slowly and the resulting mixture was stirred at –42 °C for 20 min. A solution of *t*-BuLi in pentane (3.50 mL, 1.4 M, 5 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 room temperature 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 (1.8 mL, 100 mmol) from freshly cracked ampoules was slowly added. The

resulting mixture was warmed to rt and stirred overnight. The reaction mixture was cooled to 0 °C, quenched with H_2O (2 mL) and transferred to a separatory funnel. The mixture was extracted with Et_2O (2 x 5 mL) and the combined organic layers were dried over anhydrous Na_2SO_4 and filtered to afford a solution of (*Z*)-*N*-methyl-3-deuterioallylamine. The solution was transferred to a round bottom flask and cooled to 0 °C. A solution of 4-nitrophenylisocyanate (3.63 mmol, 596 mg) in CH_2Cl_2 (4 mL) was slowly added and the resulting mixture was warmed to rt and stirred for 5 h. The reaction mixture was then concentrated in vacuo and the crude product was purified by flash chromatography on silica gel to afford the title compound as (315 mg, 37% yield, >95% deuterium incorporation) a yellow solid, mp 80–83 °C. ^{1}H NMR (700 MHz, $CDCl_3$) δ 8.12 (d, *J* = 9.2 Hz, 2H), 7.51 (d, *J* = 9.2 Hz, 2H), 6.88 (s, br, 1H), 5.87–5.82 (m, 1H), 5.27 (d, *J* = 10.4 Hz, 1H), 3.98 (d, *J* = 5.3 Hz, 2H), 3.03 (s, 3H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 154.5, 145.5, 142.3, 132.7, 125.0, 118.4, 117.2 (t, *J* = 23.8 Hz), 51.6, 34.8; IR (film) 1652 cm^{-1}. MS (CI) 237.1099 (237.1092 calcd for $C_{11}H_{12}DN_3O_3$, M + H^+).



(–)-(1'*R*,4*S*)-1'-Deuterio-4-[4-(*tert*-butyl)benzyl]-1-methyl-3-(4-nitrophenyl)imidazolidin-2-one (*d*-2f). The general procedure was employed for the coupling of (*Z*)-1-(3-*d*-allyl)-1-methyl-3-(4-nitrophenyl)urea (0.10 mmol, 23.6 mg) and 4-bromo-tert-butylbenzene (0.20 mmol, 42.6 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.002 mmol, 1.8 mg) and (*S*)-Siphos-PE (0.006 mmol 3.0 mg), a reaction temperature

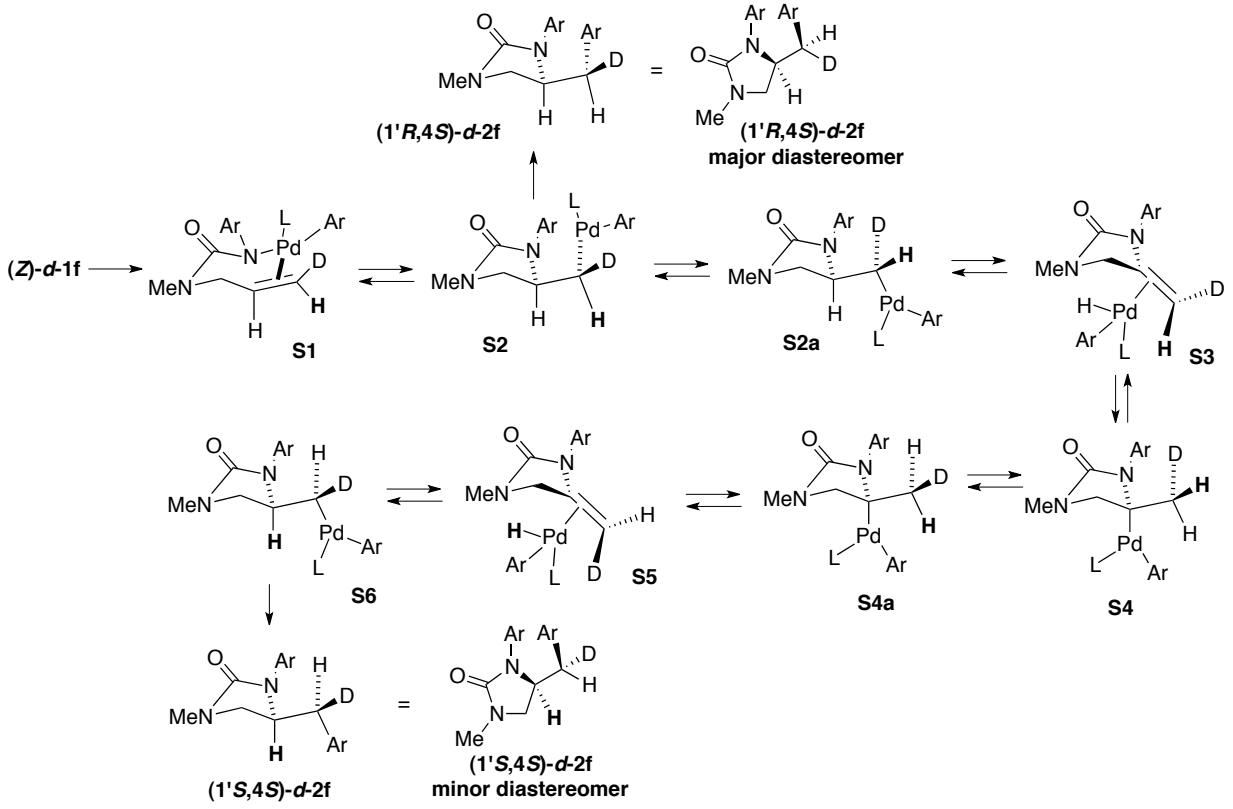
of 115 °C, and a reaction time of 18 h. This procedure afforded the title compound (32.1 mg, 85%) as a bright yellow solid, mp 110–113 °C, $[\alpha]^{23}_{D}$ –104 (*c* 1.00, CH_2Cl_2). This material was judged to be a 7:1 mixture of diastereomers by ^{1}H NMR analysis. Data are for the major isomer: ^{1}H NMR (700 MHz, $CDCl_3$) δ 8.22 (d, *J* = 8.9 Hz, 2H), 7.78 (d, *J* = 8.9 Hz, 2H), 7.34 (d, *J* = 7.8 Hz, 2H), 7.09 (d, *J* = 8.2 Hz, 2H), 4.54–4.50 (m, 1H), 3.48 (app. t, *J* = 8.7 Hz, 1H), 3.29 (dd, *J* = 2.6, 9.0 Hz, 1H), 3.09 (d, *J* = 3.2 Hz, 0.12 H), 2.74 (d, *J* = 9.0 Hz, 0.88 H), 2.83 (s, 3H), 1.30 (s, 9H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 156.7, 150.3, 145.2, 141.9, 132.4, 128.8, 125.8, 125.0, 117.7, 53.7, 48.6, 37.0 (t, *J* = 17.7 Hz), 34.5, 31.3, 30.8; IR (film) 1717 cm^{-1}. MS (CI) 369.2034 (369.2031 calcd for $C_{21}H_{24}DN_3O_3$, M + H^+). The enantiopurity was determined to be 91% ee by chiral HPLC analysis (ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 198 nm, RT= 6.3 and 9.5 min). The 1'*R*,4*S* relative stereochemistry was assigned on the basis of comparison of NMR data to those obtained for a sample of the title compound prepared using a catalyst composed of $Pd_2(dba)_3$ and DPE-Phos, which has previously been shown to effect the *syn*-carboamination of *N*-allylurea derivatives.[1]

Discussion of Mechanism of Diastereomer Formation in the Reaction of (*Z*)-*d*-1f

The formation of the minor diastereomer in the reaction of **(*Z*)-*d*-1f** is likely due to competing β-hydride elimination processes, as we have previously observed in related tetrahydrofuran-forming reactions.[4] As shown below in Scheme S1, intermediate **S1** (generated via oxidative addition of the aryl bromide to Pd(0) followed by deprotonation and transmetallation of the substrate) is formed from **(*Z*)-*d*-1f**. The *syn*-migratory insertion of the alkene into the Pd–N bond affords **S2**, which can undergo reductive

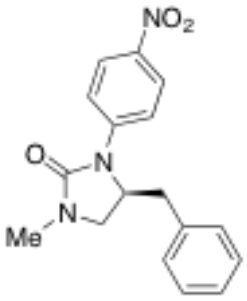
elimination to yield **(1'*R*,4*S*)-*d*-2m**. However, if this reductive elimination is relatively slow, **S2** can undergo sigma bond rotation to **S2a** followed by *syn*-β-hydride elimination to afford **S3**. Reinsertion of the alkene into the Pd–H bond with the opposite regiochemistry provides **S4**, which can undergo sigma bond rotation to **S4a**. The *syn*-β-hydride elimination of **H** from **S4a** provides **S5**, which can undergo reinsertion of the alkene into the Pd–**H** bond to give **S6**. Reductive elimination from **S6** then affords the minor diastereomer **(1'*S*,4*S*)-*d*-2m**. No migration of the deuterium atom was observed, which is presumably a result of a kinetic isotope effect coupled with the statistical probability for β-H vs. β-D elimination.

Scheme S1. Mechanism of diastereomer formation in the reaction of **(*Z*)-*d*-1f**.



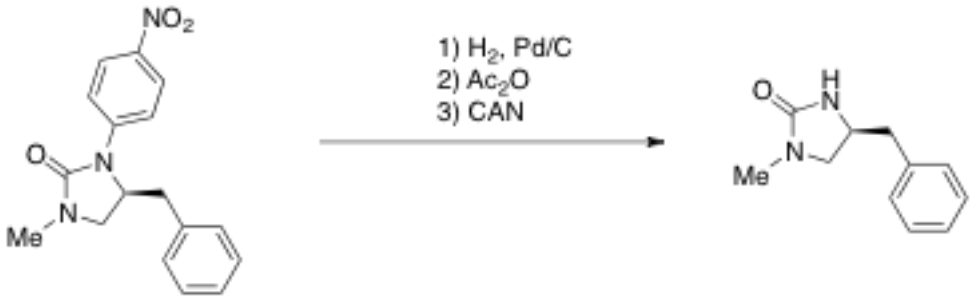
Deprotection of 2m and Assignment of Absolute Stereochemistry:

The absolute stereochemistry of the urea products was assigned by deprotection of **2m** (prepared via Pd-catalyzed carboamination of **1f**) to urea **6**. The optical rotation of **6** was of the same sign (–) as that of a separate sample of **6** prepared from L-phenylalanine as described below.



(–)-(4*S*)-4-Benzyl-1-methyl-3-(4-nitrophenyl)imidazolidin-2-one (2m): The general procedure was employed for the coupling of 1-allyl-1-methyl-3-(4-nitrophenyl)urea (1.0 mmol, 235.2 mg) and bromobenzene (1.2 mmol, 188.4 mg) using a catalyst composed of $Pd_2(dba)_3$ (0.02 mmol, 18.0 mg) and (*S*)-Siphos-PE (0.06 mmol, 30.0 mg), a reaction temperature of 115 °C, and a reaction time of 18 h. This procedure afforded the title compound (256.9 mg, 83%) as a bright yellow solid, mp 125–128 °C: $[\alpha]^{23}_{D}$ –108.9 (*c* 1.22, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.24 (d, *J* = 9.3 Hz, 2H), 7.80 (d, *J* = 9.3 Hz, 2H), 7.33 (t, *J* = 6.9 Hz, 2H), 7.28 (t, *J* = 7.3 Hz, 1H), 7.06 (d, *J* = 7.1 Hz, 2H), 4.58–4.52 (m, 1H), 3.47 (app. t, *J* = 8.8 Hz, 1H), 3.28 (dd, *J* = 2.9, 9.2 Hz, 1H), 3.14 (dd, *J* = 3.2, 13.9 Hz, 1H), 2.83–2.77 (m, 1H), 2.81 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 156.7, 145.1, 142.0, 135.5, 129.2, 128.9, 127.3, 125.0, 117.7, 53.7, 48.4, 37.7, 30.8; IR (film) 1717 cm^{-1}; MS (CI) 312.1347 (312.1343 calcd for $C_{17}H_{17}N_3O_3$, M + H^+). The

enantiopurity was determined to be 89% ee by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 15% IPA/Hexanes, 1.5 mL/min, λ 198 nm, RT= 10.5 and 17.1 min).



(–)-(4*S*)-4-Benzyl-1-methylimidazolidin-2-one (6). A glass microwave tube equipped with a stirbar was charged with 4-Benzyl-1-methyl-3-(4-nitrophenyl)imidazolidin-2-one (77.8 mg, 0.25 mmol,), 10% Pd/C (38.9 mg, 5% w/w Pd), ethyl acetate (2 mL) and methanol (1 mL). The tube was placed into a stainless steel bomb that was pressurized with H_2 to 50 psi and the reaction mixture was then stirred at rt for 12 h. The reaction vessel was then depressurized and the mixture was filtered through a pad of celite. The celite was washed with methanol (25 mL) and the combined organic solutions were concentrated in vacuo. The crude product from this reaction was dissolved in CH_2Cl_2 (0.7 mL) and transferred to a flame dried Schlenk tube equipped with a stir bar that had been cooled under a stream of nitrogen. Acetic anhydride (28 µL, 0.30 mmol) was added to the flask and the resulting solution was stirred at rt for 5 h. A solution of saturated aqueous Na_2CO_3 (5 mL) was added to the reaction vessel and the resulting mixture was transferred to a separatory funnel. The mixture was extracted with CH_2Cl_2 (3 x 10 mL) and the combined organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated in vacuo. The crude product from this reaction was placed into a round bottom flask equipped with a stir bar and dissolved in CH_3CN (3.5 mL) and

H_2O (0.70 mL). The mixture was cooled to 0 °C, stirred for 5 min, then ceric ammonium nitrate (1.13 mmol, 618.0 mg) was added in one portion. The resulting mixture was stirred at 0 °C for 25 min then saturated aqueous sodium sulfite (6 mL) was added. The mixture was transferred to a separatory funnel and extracted with ethyl acetate (3 x 10 mL). The combined organic layers were washed with saturated aqueous $NaHCO_3$ (5 mL) and brine (5 mL) then dried over anhydrous sodium sulfate, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel to afford the title compound (41.6 mg, 88% overall yield) as a brown oil, $[\alpha]^{23}_D$ –27.0 (*c* 1.0, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 (t, *J* = 7.1 Hz, 2H), 7.24 (t, *J* = 7.6 Hz, 1H), 7.17 (d, *J* = 7.1 Hz, 2H), 4.82–4.52 (s, br, 1H), 3.85 (m, 1H), 3.47 (app. t, *J* = 8.6 Hz, 1H), 3.13 (dd, *J* = 6.1, 8.8 Hz, 1H), 2.81 (app. d, *J* = 7.1 Hz, 2H), 2.76 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.9, 137.1, 129.0, 128.8, 126.9, 52.7, 51.1, 42.0, 30.5; IR (film) 1699 cm^{-1}; MS (CI) 191.1181 (191.1179 calcd for $C_{11}H_{14}N_2O$, M + H^+). The enantiopurity was determined to be 85% ee by chiral HPLC analysis (Lux Amylose-2, 25 cm x 4.6 mm, 10% IPA/Hexanes, 1.0 mL/min, λ 210 nm, RT= 21.3 and 22.7 min).

(–)-(4*S*)-4-Benzyl-1-methylimidazolidin-2-one (6) A flame-dried round bottomed flask equipped with a stirbar was cooled under a stream of nitrogen and charged with (*S*)-*N*^{1}-methyl-3-phenylpropane-1,2-diamine[3] (100.0 mg, 0.60 mmol) and THF (1 mL). Solid CDI (90.0 mg, 0.56 mmol) was added and the resulting mixture was heated to 60 °C with stirring for 12 h. The reaction mixture was then cooled to rt and the solvent was

removed in vacuo. The product was purified by flash chromatography on silica gel to afford the title compound (32.0 mg, 30% yield); $[\alpha]^{23}_{D}$ –37.2 (*c* 0.90, CH_2Cl_2). The spectroscopic properties of this compound were identical to that of compound **6.** The enantiopurity was determined to be 97% ee by chiral HPLC analysis (Lux Amylose-2, 25 cm x 4.6 mm, 10% IPA/Hexanes, 1.0 mL/min, λ 210 nm, RT= 21.3 and 22.7 min).

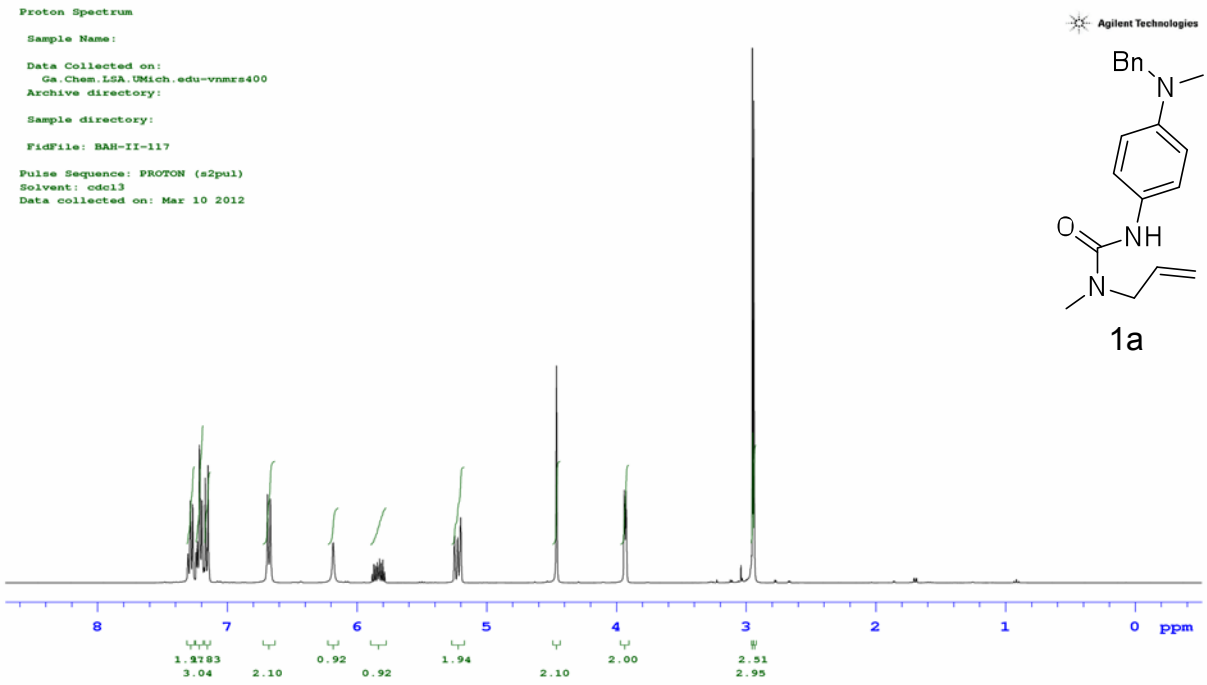
References

[1] J.A. Fritz, J.P. Wolfe, *Tetrahedron* **2008**, *64*, 6838-6852.

[2] J. Barluenga, F.J. Fananas, F. Foubelo, M. Yus, *J. Chem. Soc., Chem. Commun*. **1988**, 1135-1136.

[3] J. Li, S. Luo, J. Cheng, *J. Org. Chem*. **2008**, 1747-1750.

[4] M. B. Hay, J. P. Wolfe, *J. Am. Chem. Soc.* **2005**, *127*, 16468–16476.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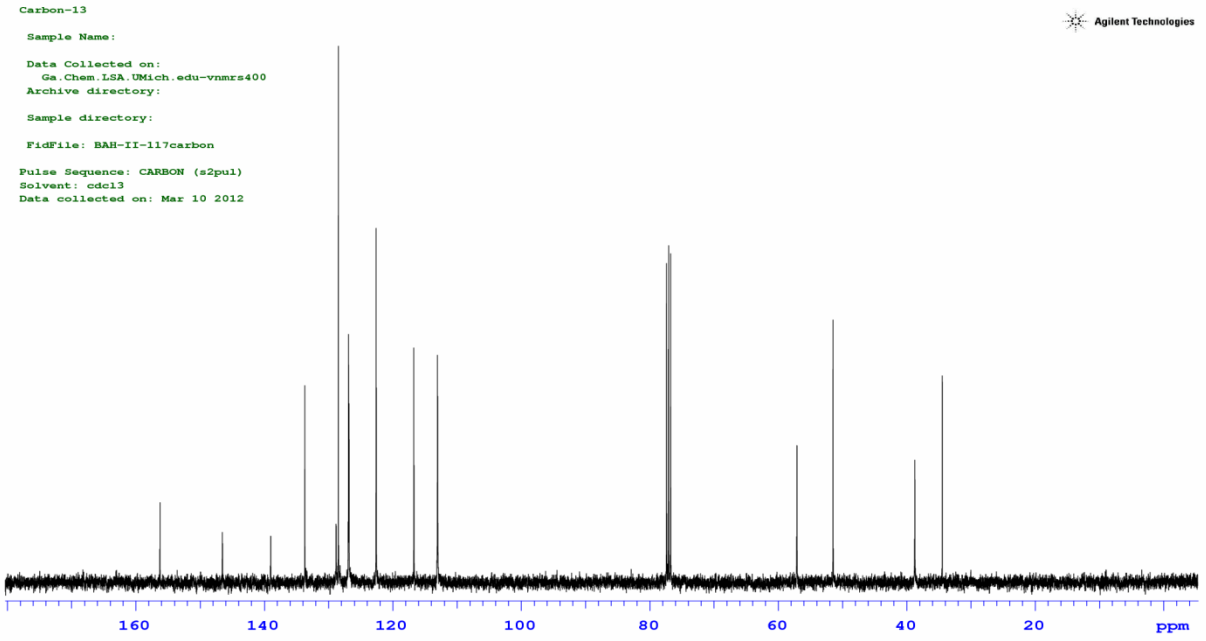
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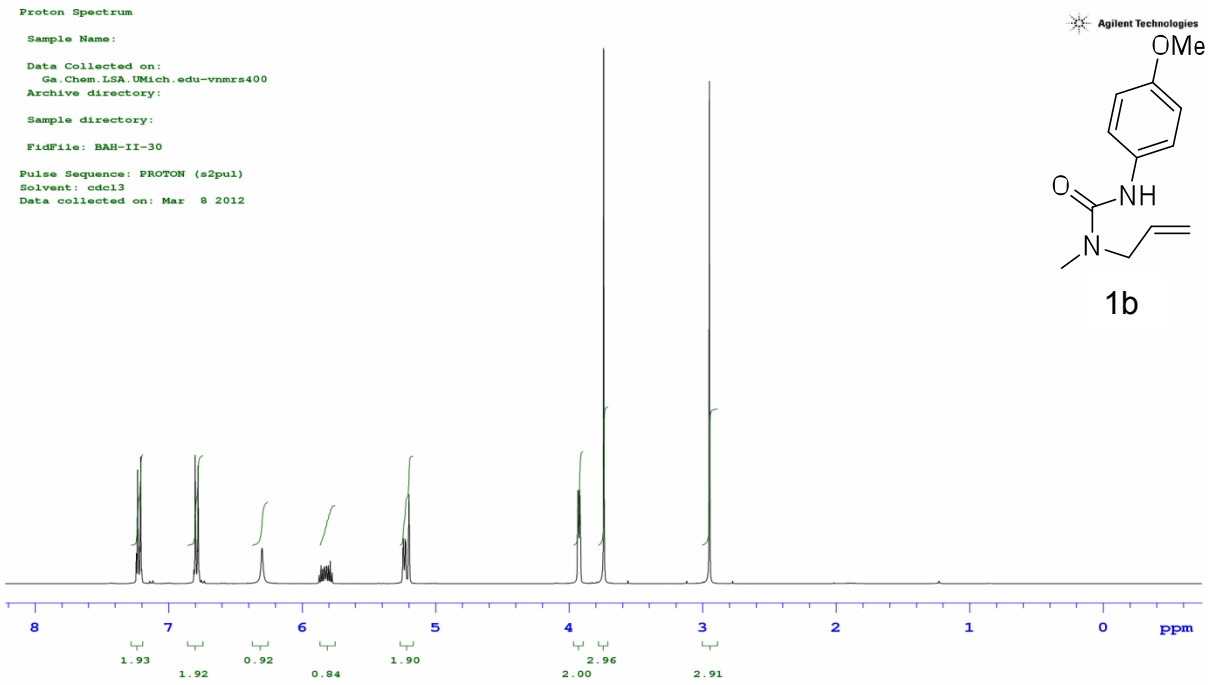
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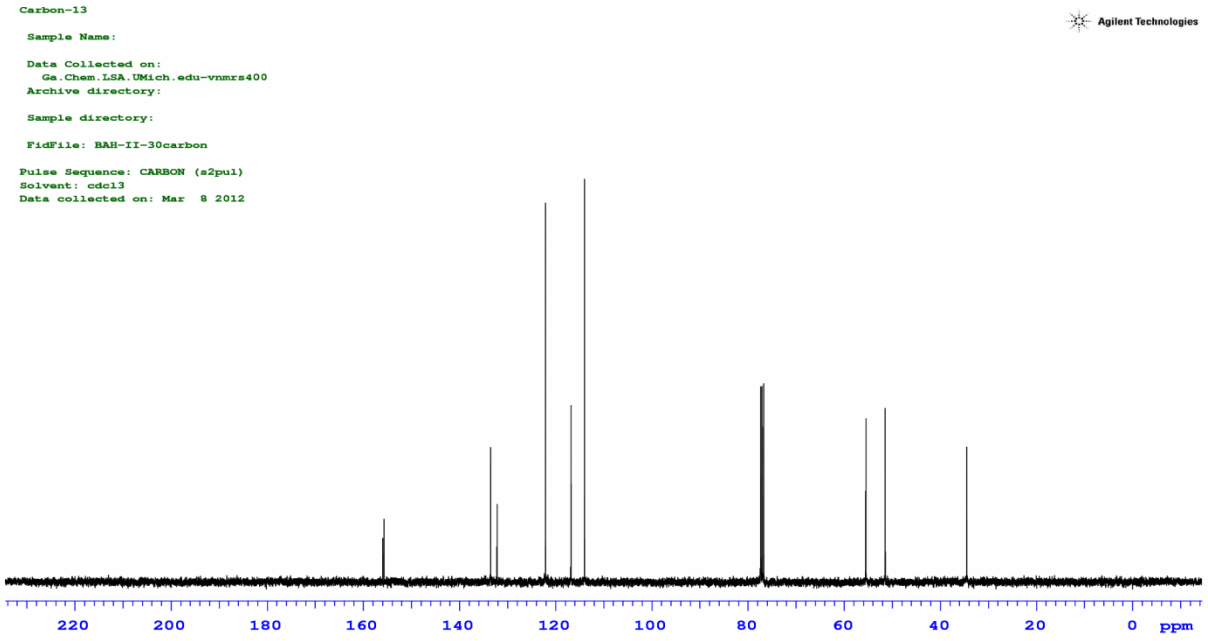
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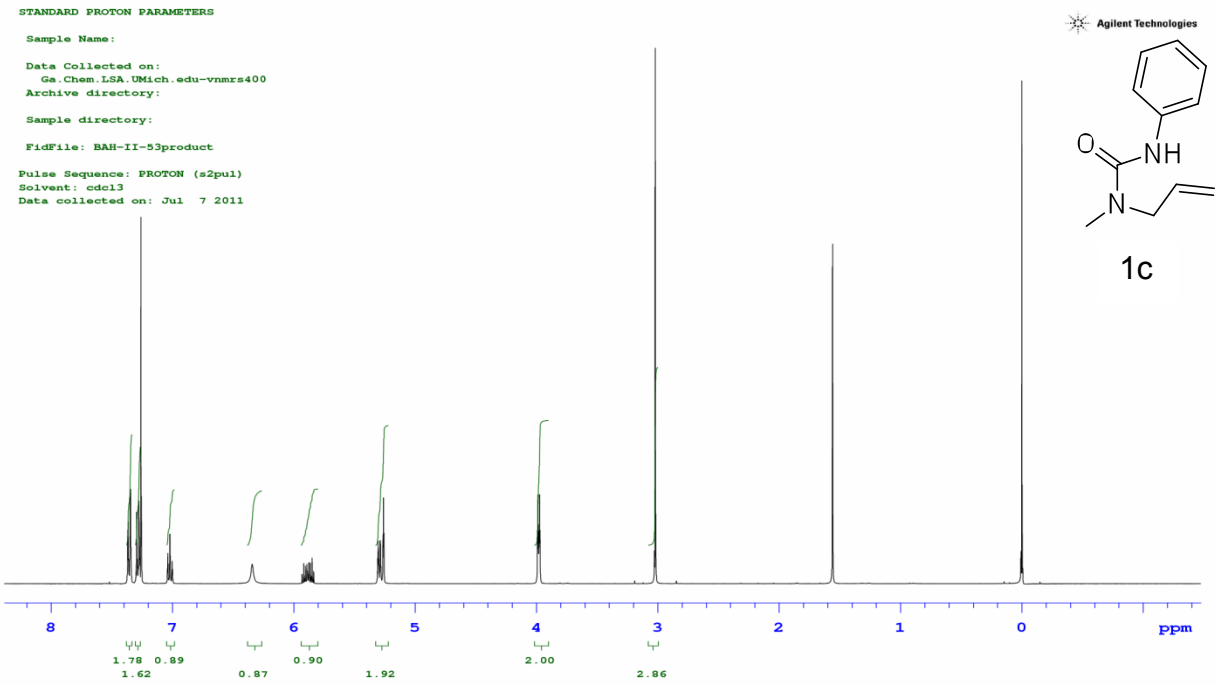
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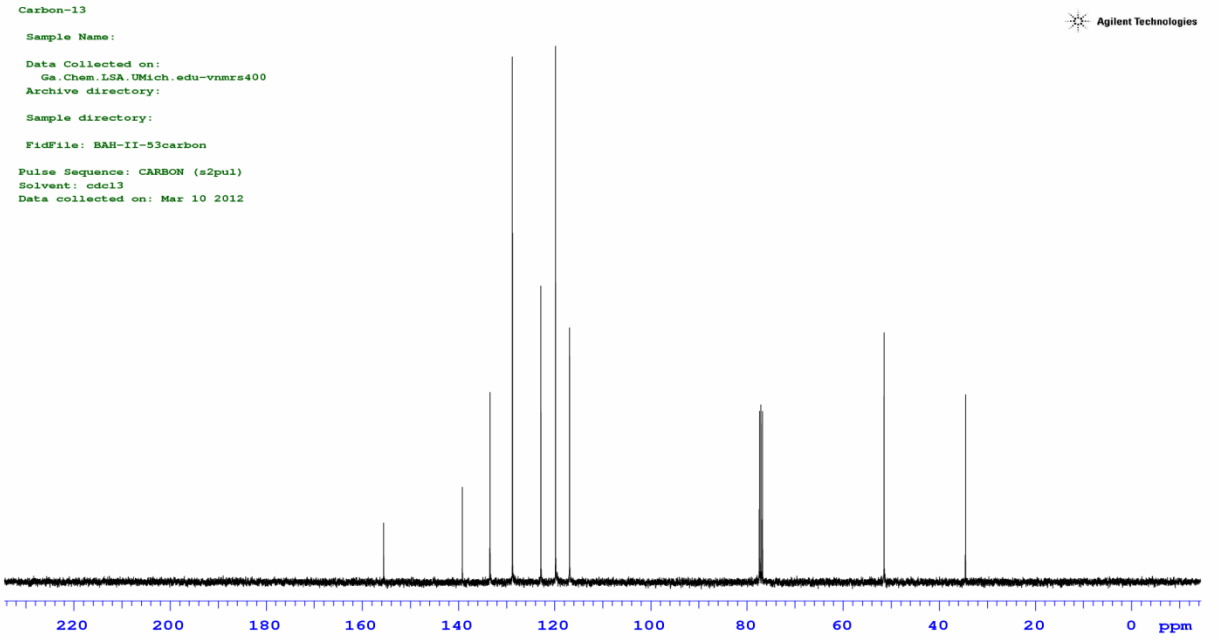
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Agilent Technologies
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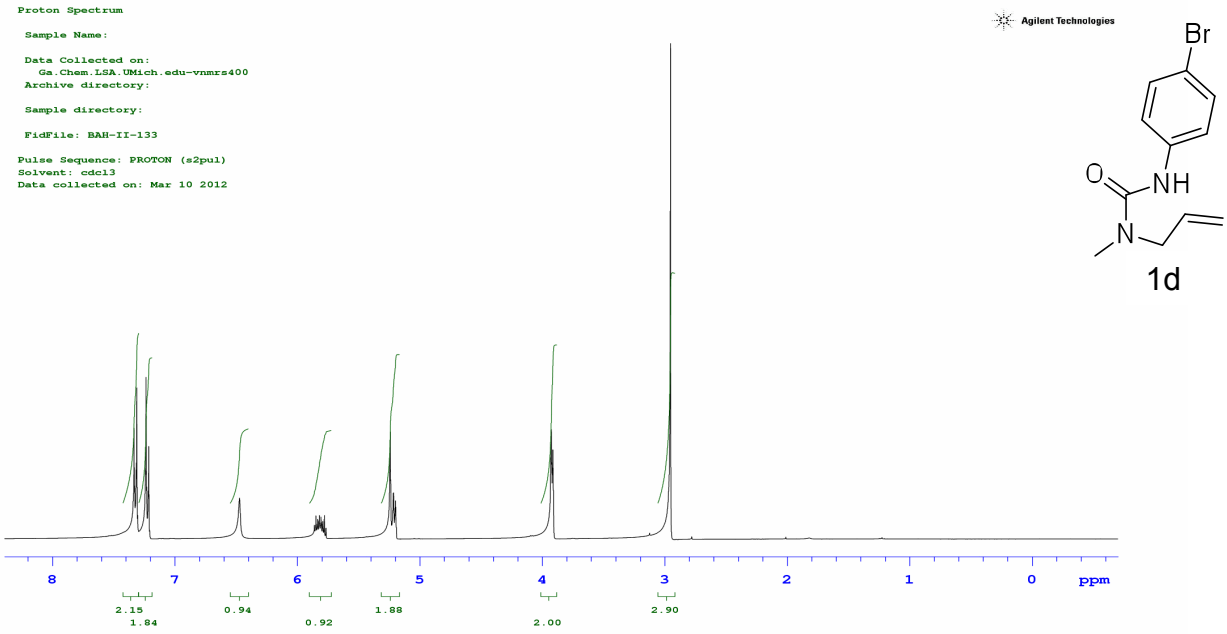
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N
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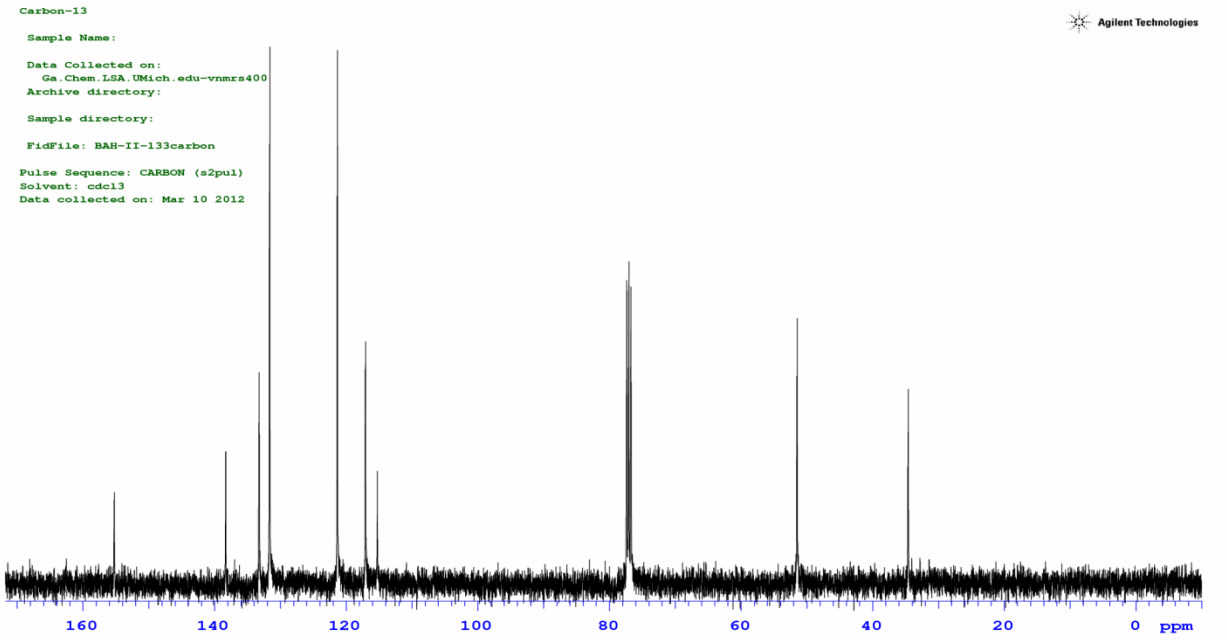
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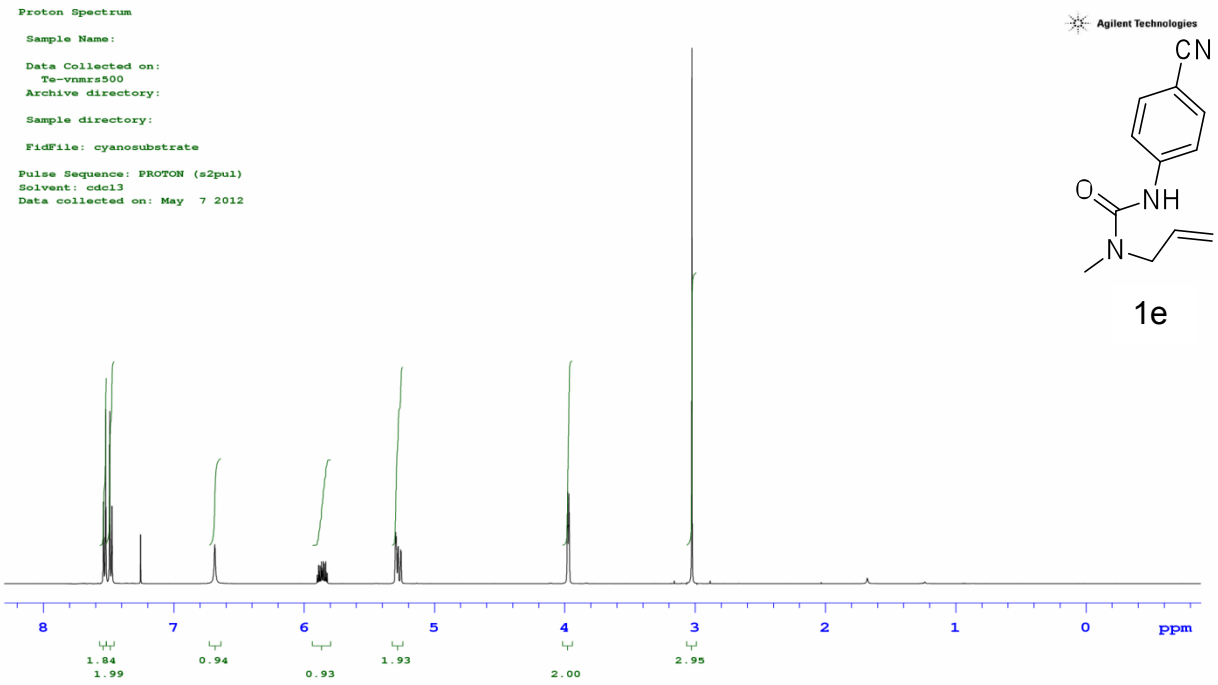
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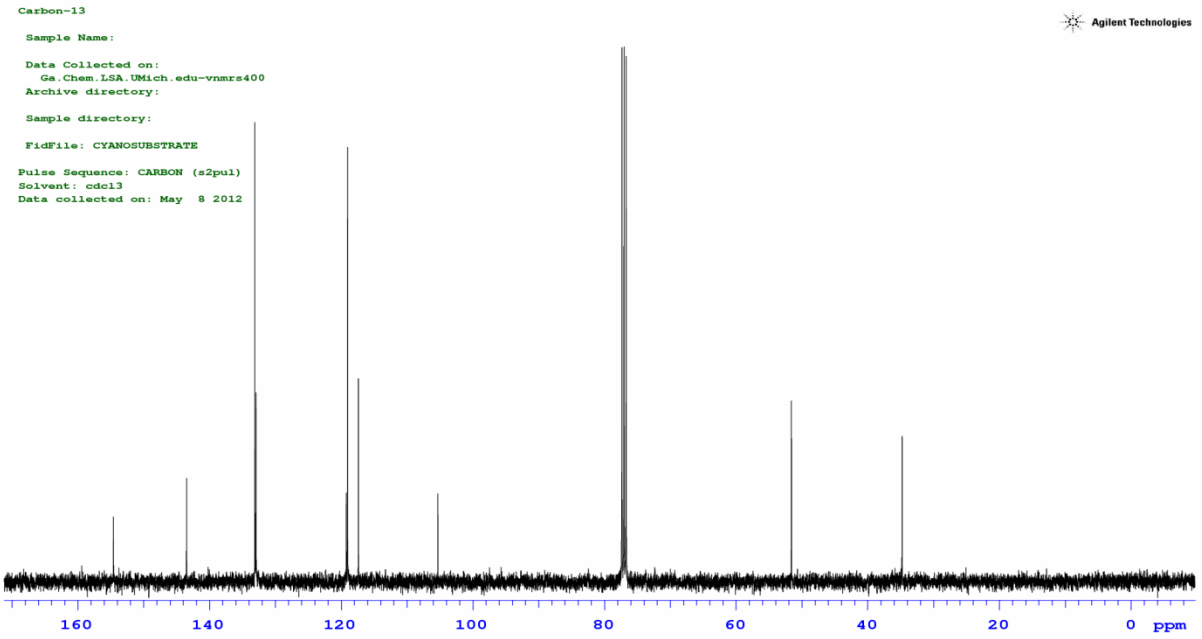
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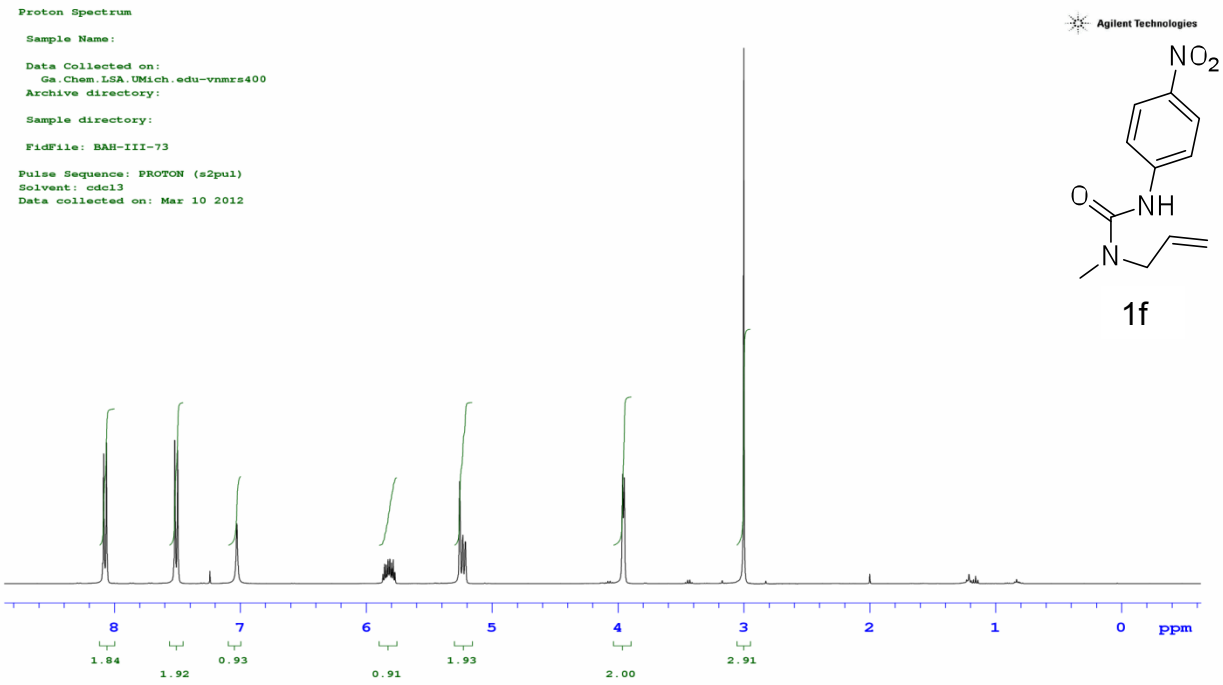
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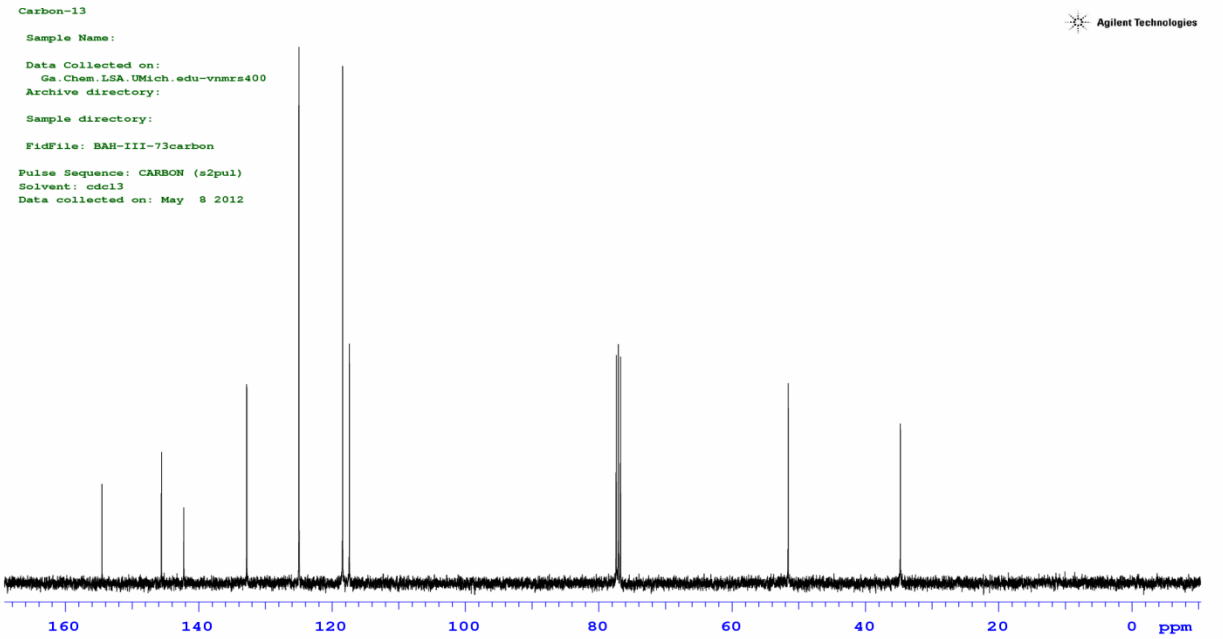
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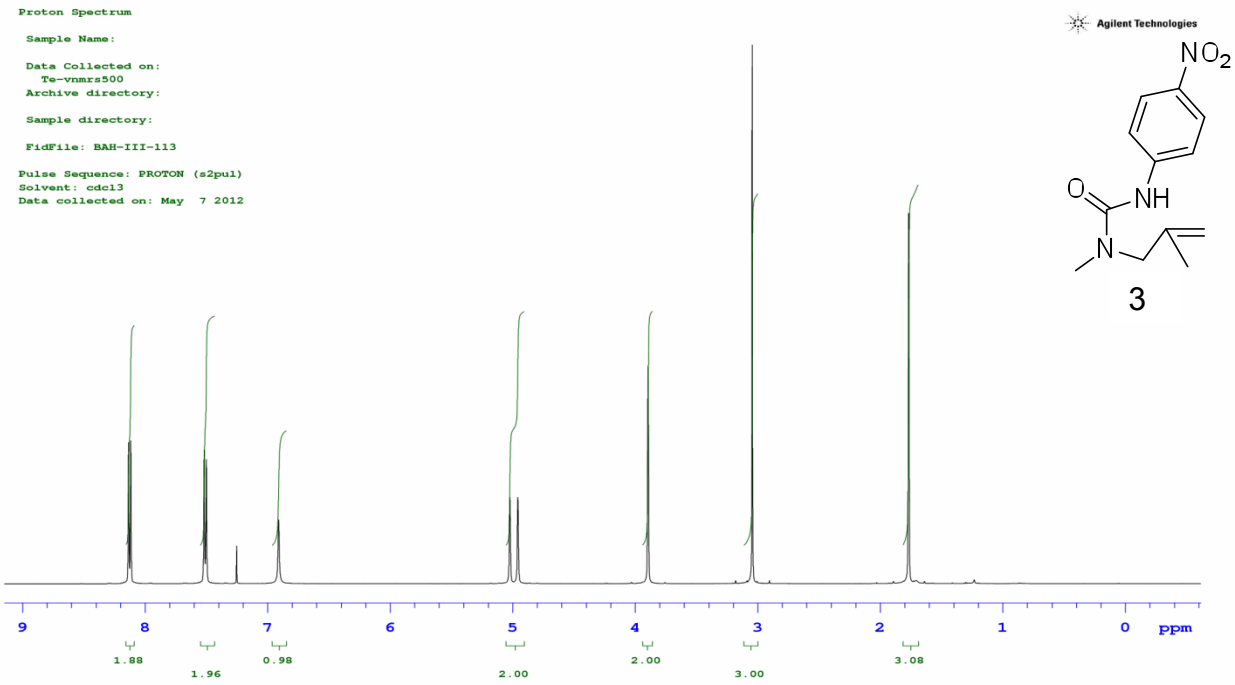
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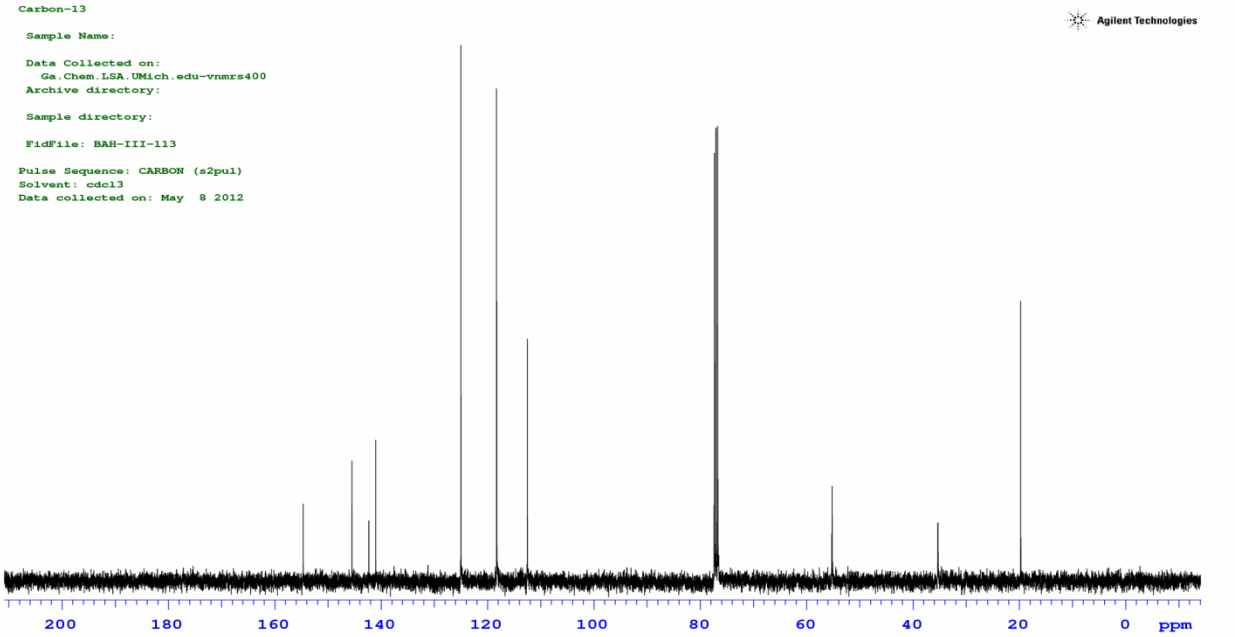
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Agilent Technologies
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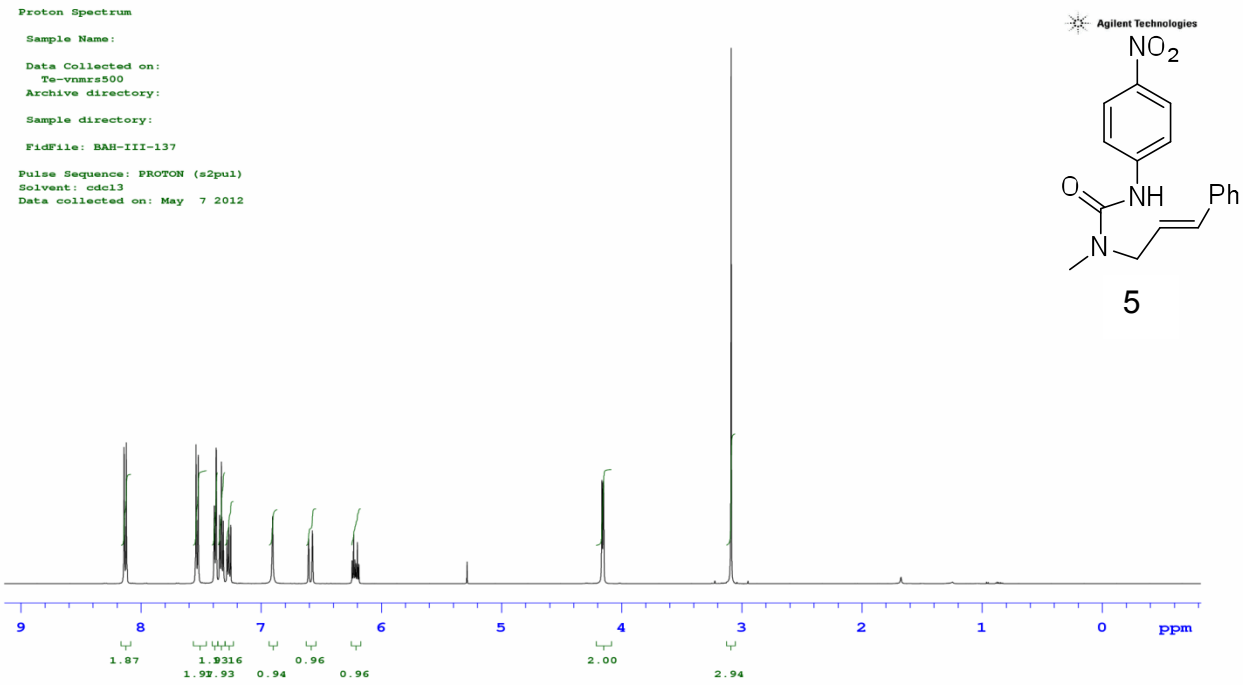
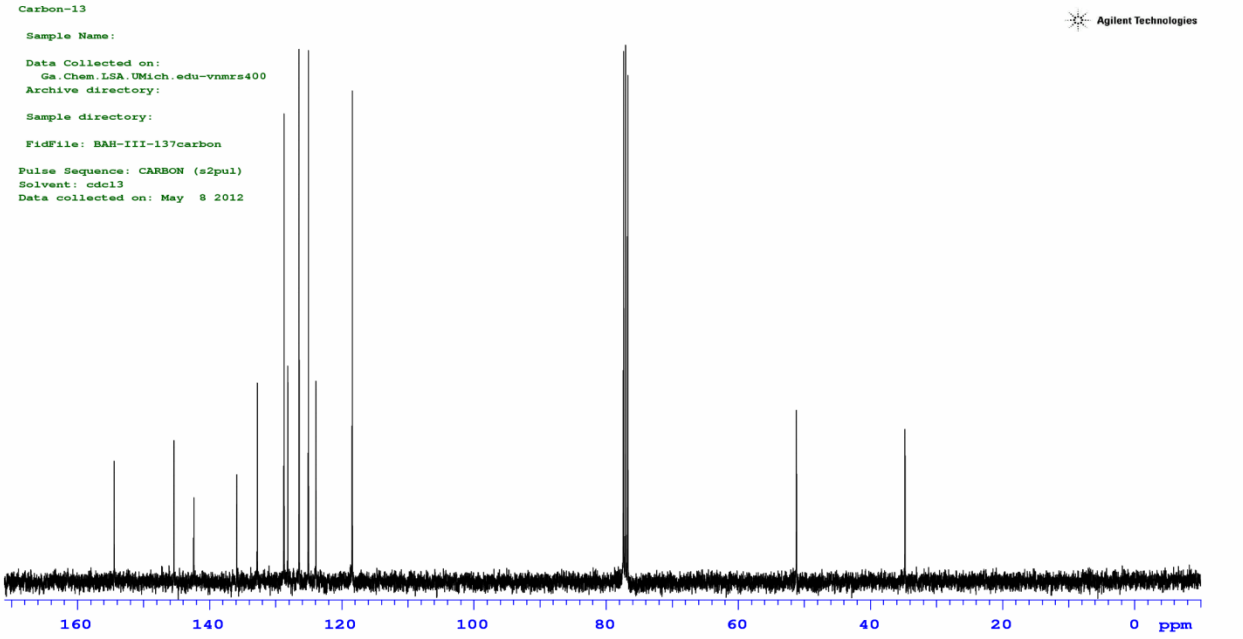
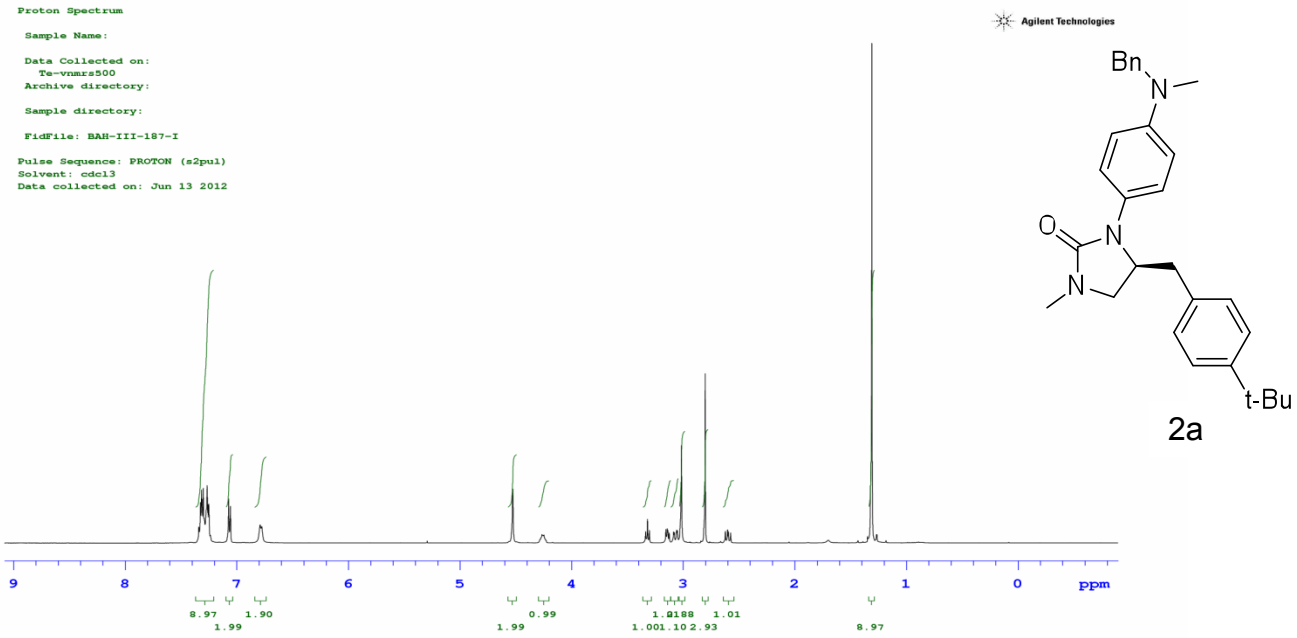
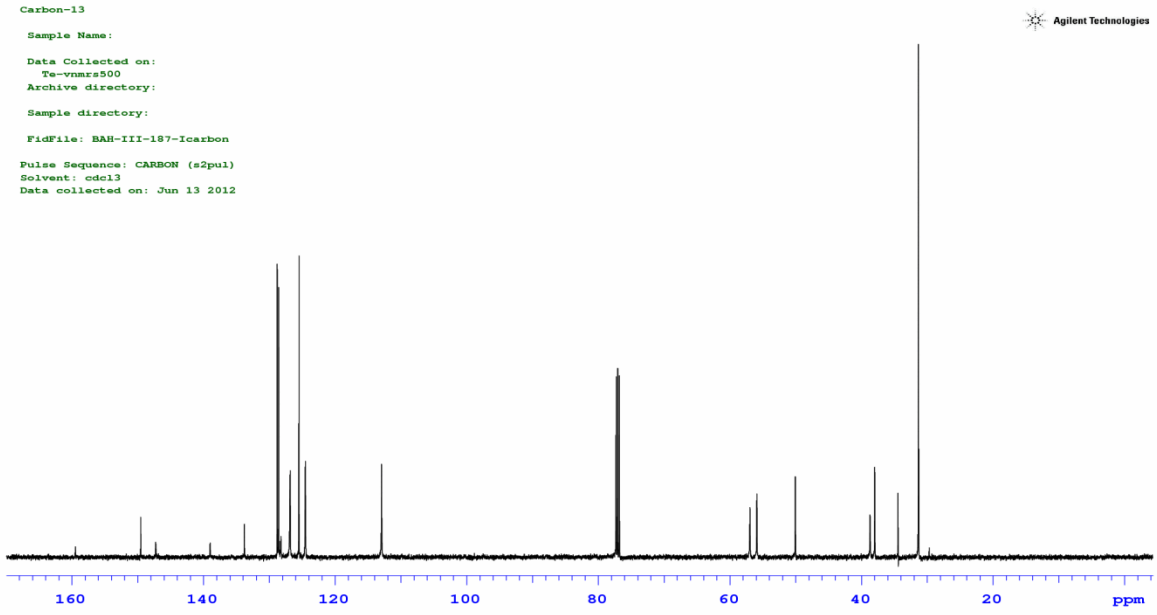
0
ppm


Proton Spectrum
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: BAH-III-137
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 7 2012
Agilent Technologies
NO2
NH
Ph
O
N
5
9
8
7
6
5
4
3
2
1
0
ppm
1.87
0.96
2.00
2.94
0.94
0.96


Carbon-13
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-137carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 8 2012
Agilent Technologies
160
140
120
100
80
60
40
20
0
ppm

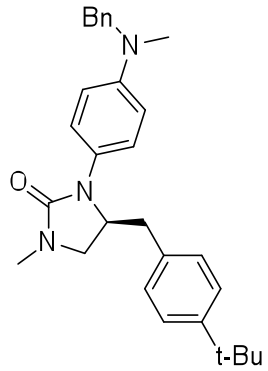

Proton Spectrum
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: BAH-III-187-I
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 13 2012
Agilent Technologies
Bn
N
O
N
N
t-Bu
9
8
7
6
5
4
3
2
1
0
ppm
8.97
1.90
1.99
0.99
1.99
1.00
1.02
2.93
1.01
8.97

2a


Carbon-13
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: BAH-III-187-Icarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 13 2012
Agilent Technologies
160
140
120
100
80
60
40
20
ppm

6/11/2012 18:12:31 1 / 1

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

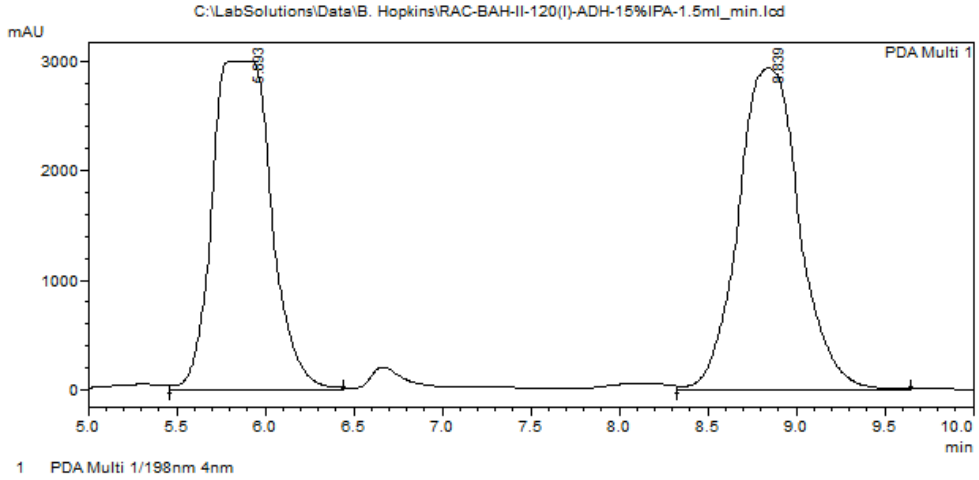


2a

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-II-120(I)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-II-120(I)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-II-120(I)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/7/2012 3:51:27 PM
Data Processed	: 6/7/2012 4:06:40 PM

<Chromatogram>



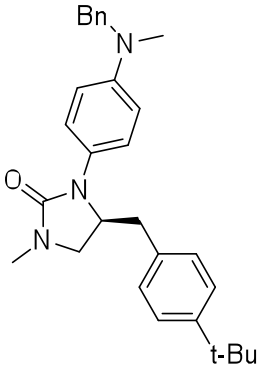
1 PDA Multi 1/198nm 4nm

PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.893	69331412	2993485	49.525	50.433
2	8.839	70661842	2942080	50.475	49.567
Total		139993254	5935565	100.000	100.000

6/18/2012 14:30:15 1 / 1



2a

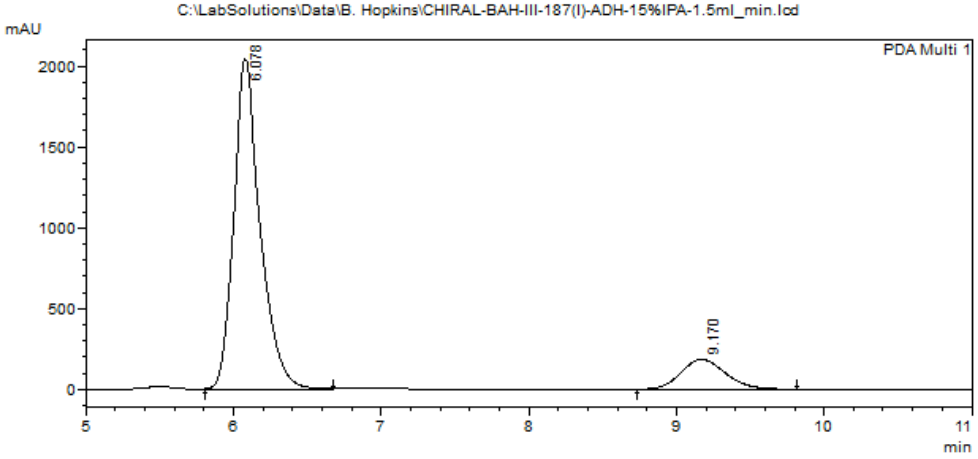
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-187(I)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-187(I)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-187(I)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/13/2012 1:15:18 PM
Data Processed	: 6/13/2012 1:41:24 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-187(I)-ADH-15%IPA-1.5ml_min.lcd

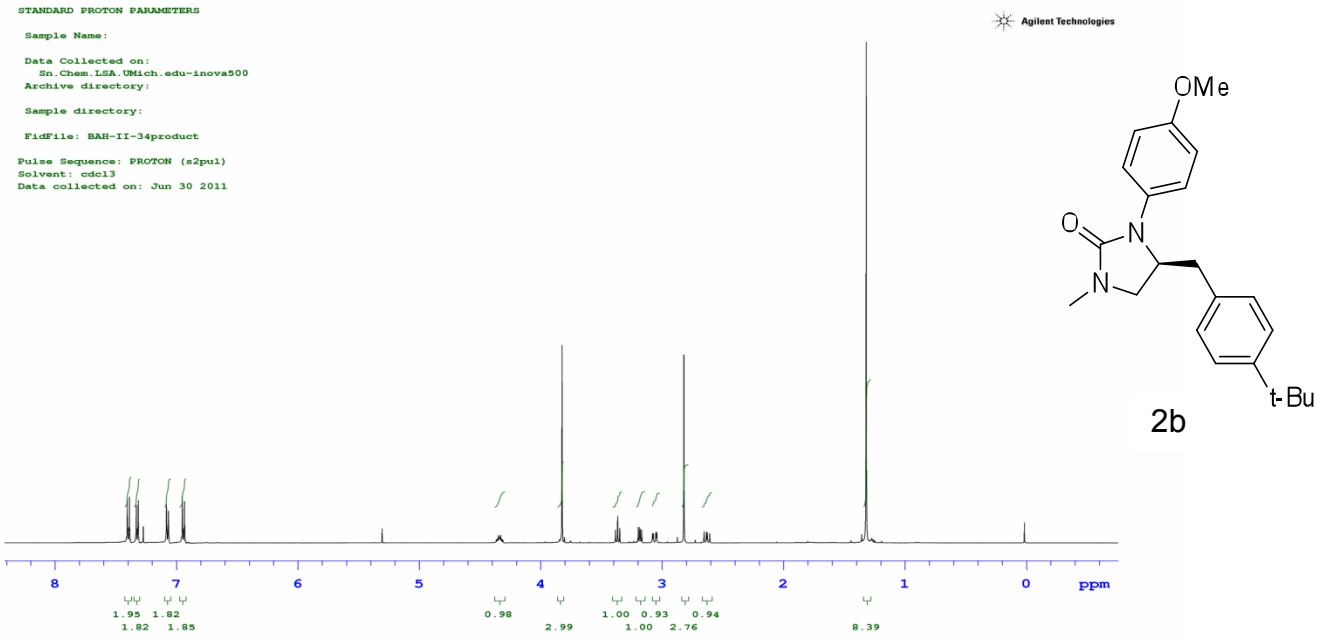


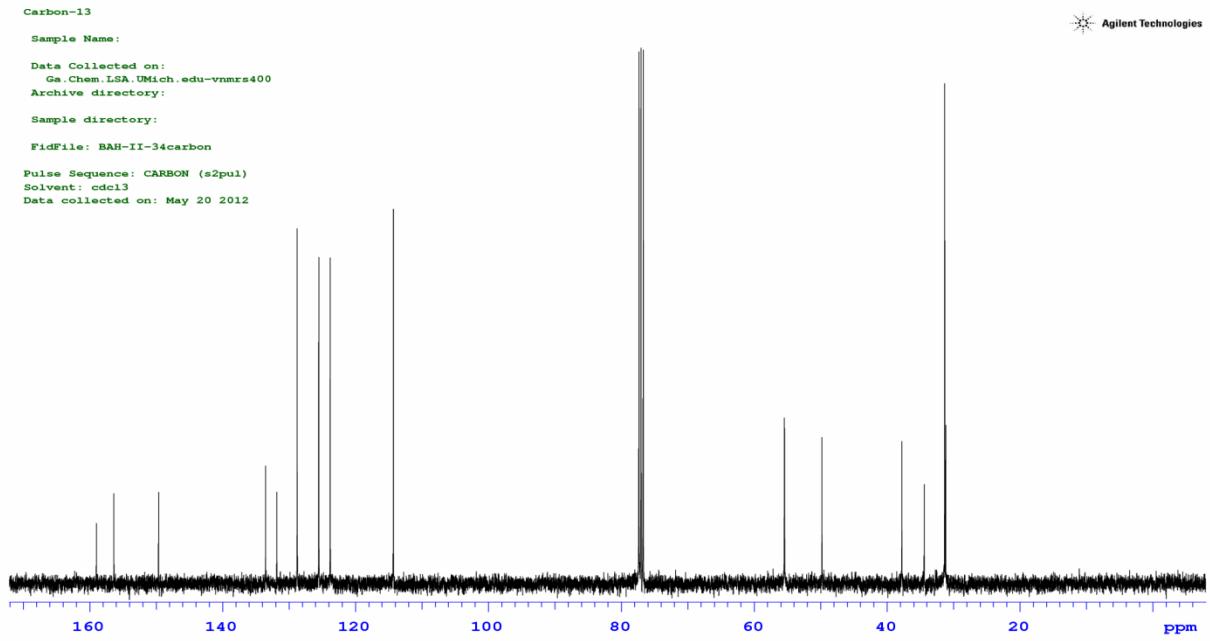
1 PDA Multi 1/198nm 4nm

PeakTable

PDA Ch1 198nm 4nm

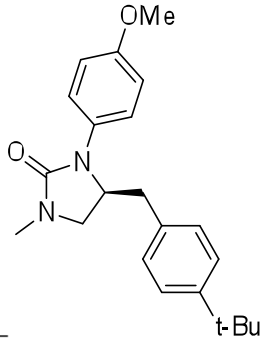
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.078	25236356	2043672	86.685	91.720
2	9.170	3876372	184483	13.315	8.280
Total		29112729	2228154	100.000	100.000


STANDARD PROTON PARAMETERS
Sample Name:
Data Collected on:
Sn.Chem.LSA.UMich.edu-inova500
Archive directory:
Sample directory:
FidFile: BAH-II-34product
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 30 2011
Agilent Technologies
OMe
O
N
N
t-Bu
2b
8
7
6
5
4
3
2
1
0
ppm
1.95 1.82
1.82 1.85
0.98
2.99
1.00 0.93 0.94
1.00 2.76
8.39


Carbon-13
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-II-34carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 20 2012
Agilent Technologies
160
140
120
100
80
60
40
20
ppm

7/1/2012 18:30:14 1 / 1

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



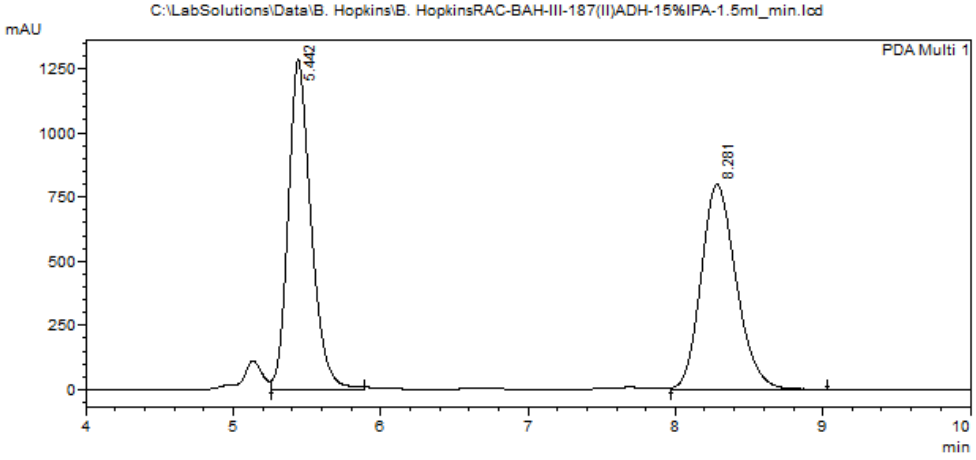
2b

C:\LabSolutions\Data\B. Hopkins\B. HopkinsRAC-BAH-III-187(II)ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-187(II)ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: B. HopkinsRAC-BAH-III-187(II)ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/13/2012 3:29:57 PM
Data Processed	: 6/13/2012 4:15:50 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\B. HopkinsRAC-BAH-III-187(II)ADH-15%IPA-1.5ml_min.lcd



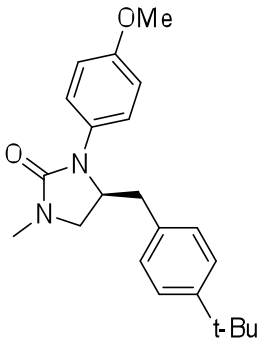
1 PDA Multi 1/198nm 4nm

PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.442	13776501	1286634	50.992	61.643
2	8.281	13240248	800590	49.008	38.357
Total		27016749	2087224	100.000	100.000

6/11/2012 18:19:32 1 / 1



2b

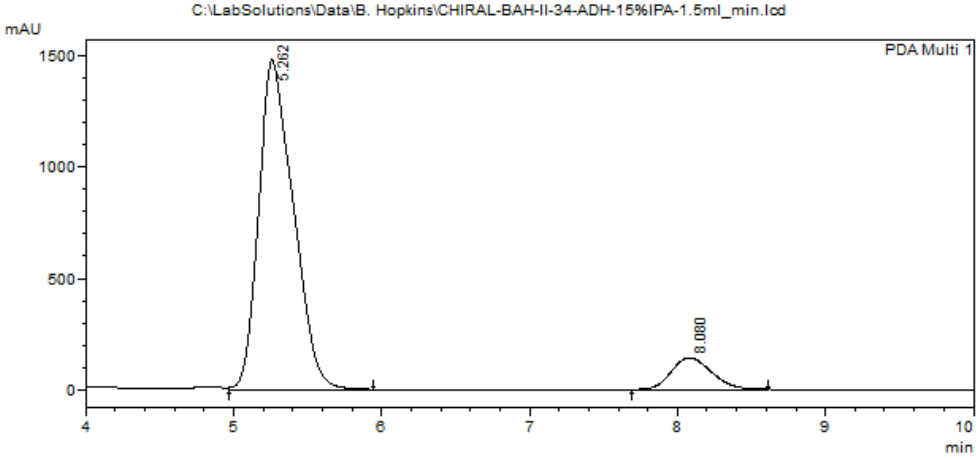
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-II-34-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-II-34-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-II-34-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/7/2012 10:29:07 AM
Data Processed	: 6/7/2012 10:58:14 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-II-34-ADH-15%IPA-1.5ml_min.lcd



1 PDA Multi 1/198nm 4nm

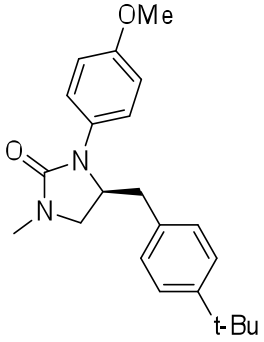
PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.262	24398002	1483328	89.839	91.144
2	8.080	2759497	144122	10.161	8.856
Total		27157499	1627450	100.000	100.000

6/11/2012 18:02:28 1 / 1

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

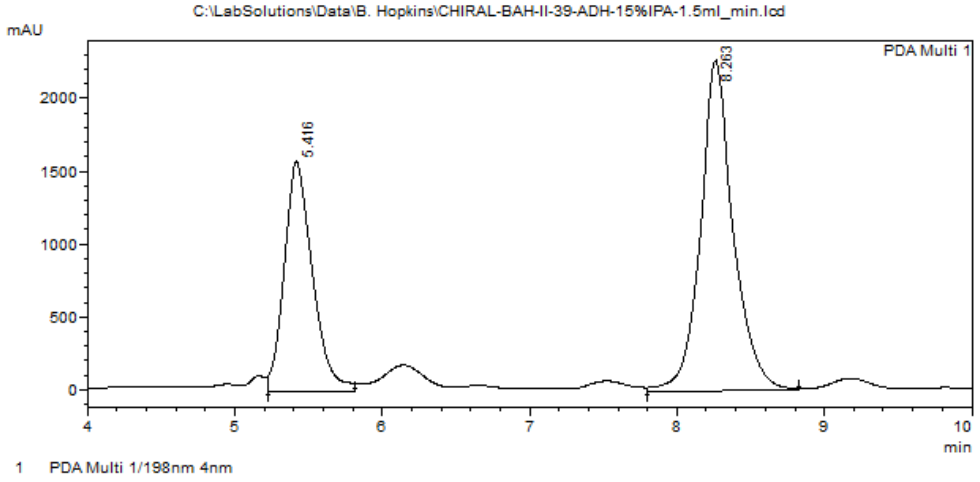


2b (w/ (R)-Siphos-PE)

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-II-39-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-II-39-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-II-39-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/11/2012 5:42:02 PM
Data Processed	: 6/11/2012 5:58:27 PM

<Chromatogram>

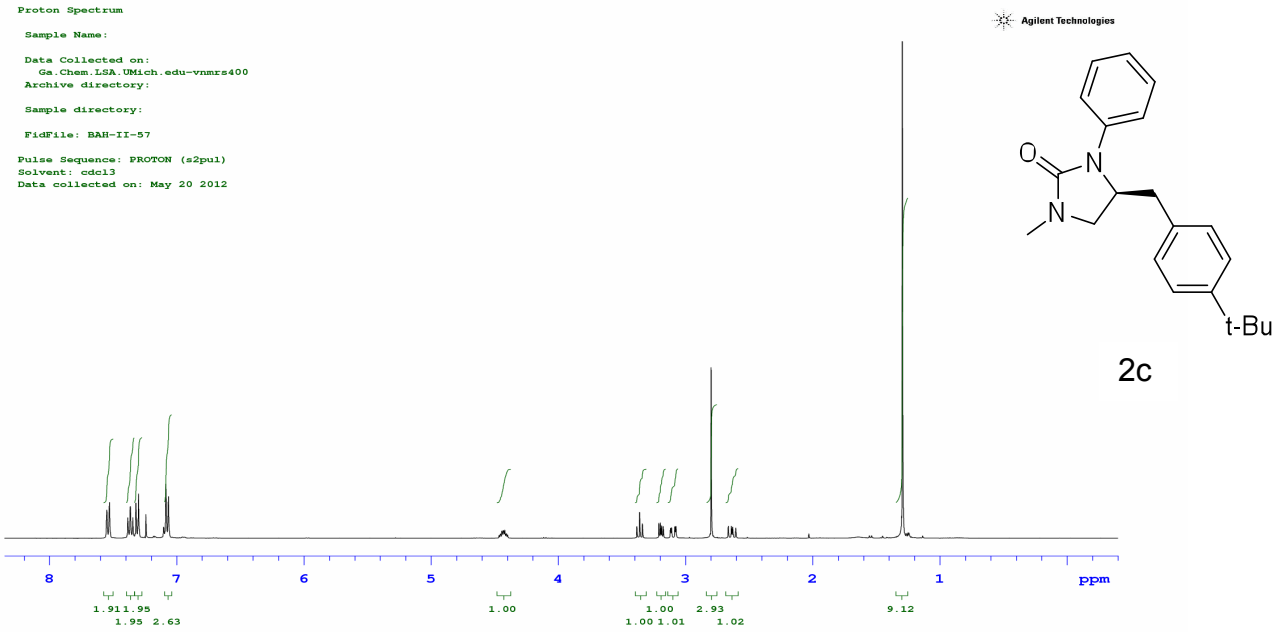


1 PDA Multi 1/198nm 4nm

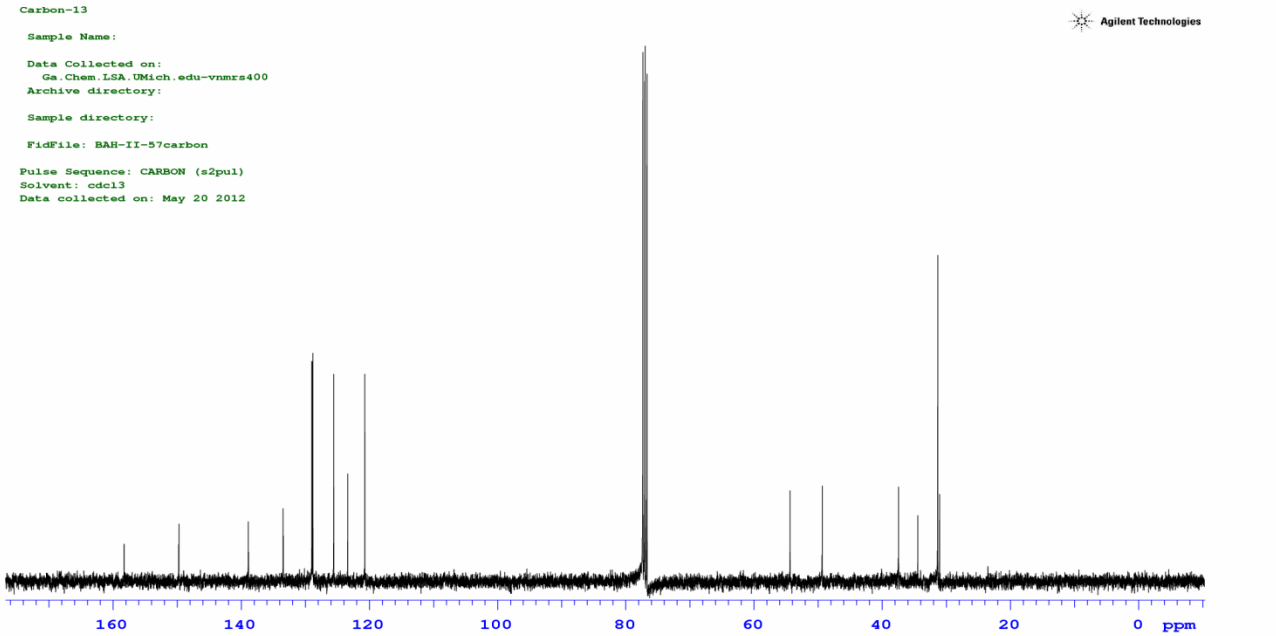
PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.416	20861745	1580948	37.536	41.085
2	8.263	34716376	2267026	62.464	58.915
Total		55578121	3847975	100.000	100.000

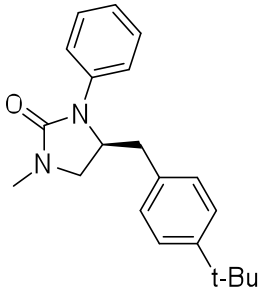

Proton Spectrum
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-II-57
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 20 2012
Agilent Technologies
O
N
N
t-Bu
8
7
6
5
4
3
2
1
ppm
1.91 1.95
1.95 2.63
1.00
1.00
1.00 1.01
2.93
1.02
9.12

2c


Carbon-13
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-II-57carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 20 2012
Agilent Technologies
160
140
120
100
80
60
40
20
0
ppm

6/6/2012 17:11:57 1 / 1

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

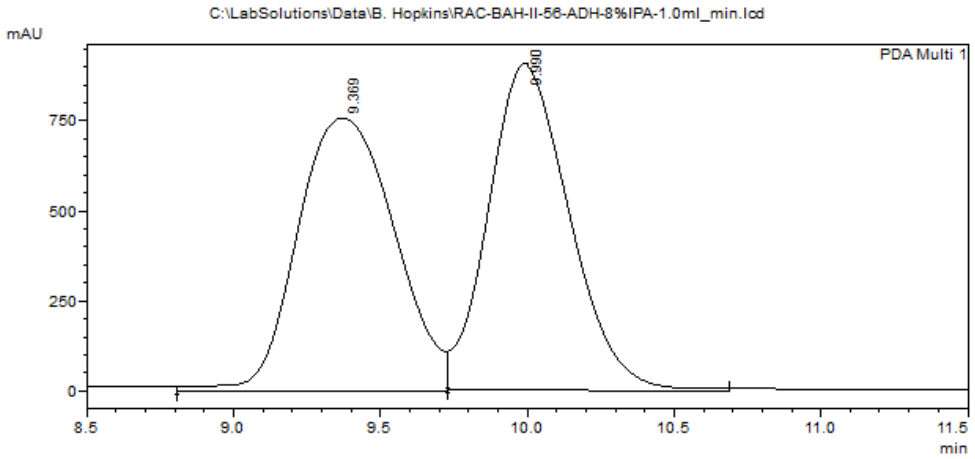


2c

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-II-56-ADH-8%IPA-1.0ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-II-56-ADH-8%IPA-1.0ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-II-56-ADH-8%IPA-1.0ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/5/2012 11:38:16 AM
Data Processed	: 6/5/2012 11:55:47 AM

<Chromatogram>



1 PDA Multi 1/198nm 4nm

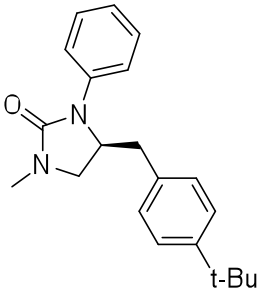
PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.369	17405197	757989	49.803	45.532
2	9.990	17542838	906760	50.197	54.468
Total		34948035	1664749	100.000	100.000

7/2/2012 14:40:29 1 / 1

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



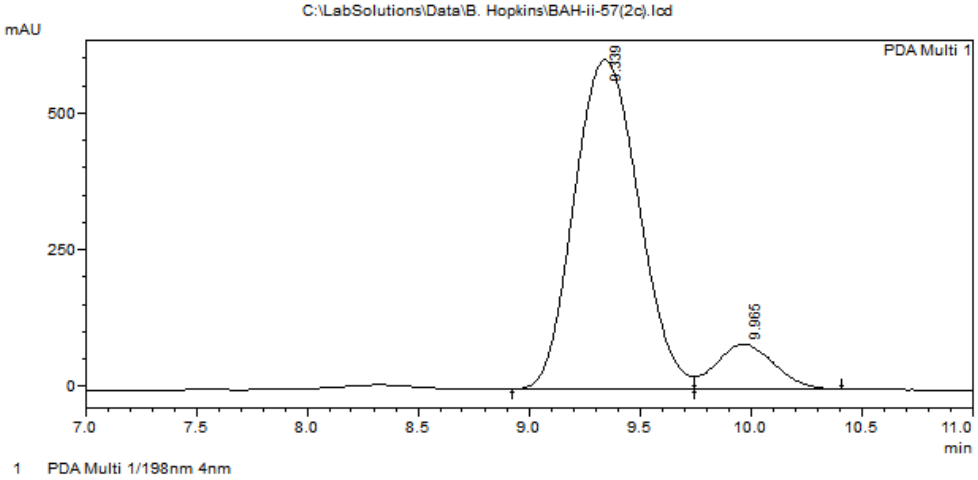
2c

C:\LabSolutions\Data\B. Hopkins\BAH-ii-57(2c).lcd

Acquired by	: Admin
Sample Name	: BAH-ii-57(2c)
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: BAH-ii-57(2c).lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/2/2012 2:24:18 PM
Data Processed	: 7/2/2012 2:37:21 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\BAH-ii-57(2c).lcd

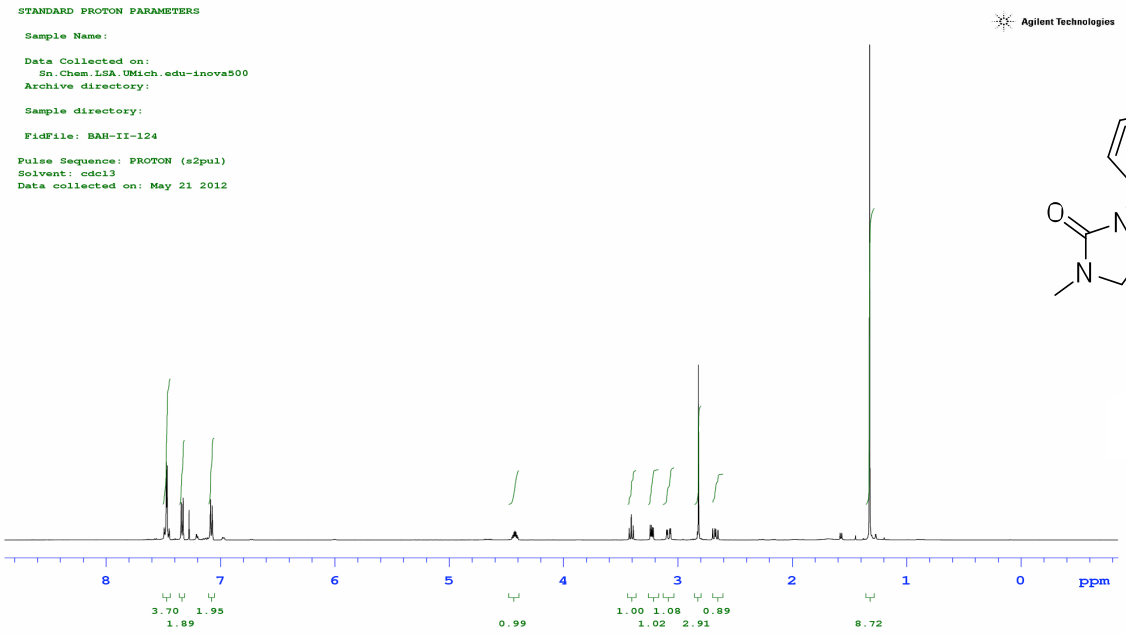


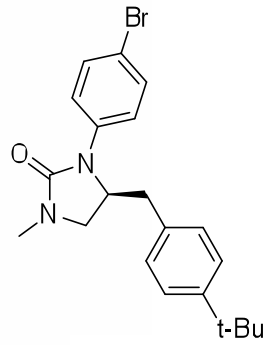
1 PDA Multi 1/198nm 4nm

PeakTable

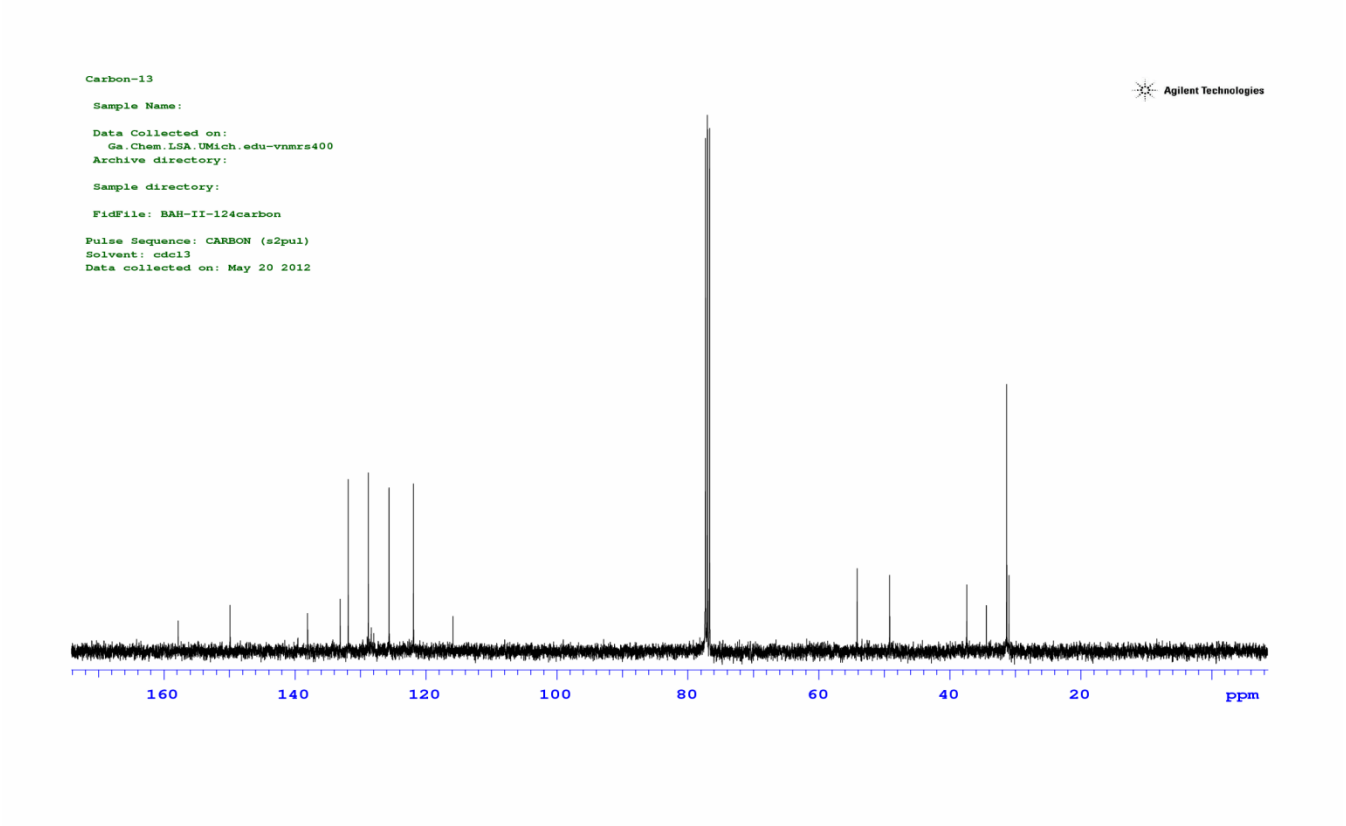
PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.339	12187152	603290	89.156	88.053
2	9.965	1482275	81851	10.844	11.947
Total		13669427	685141	100.000	100.000

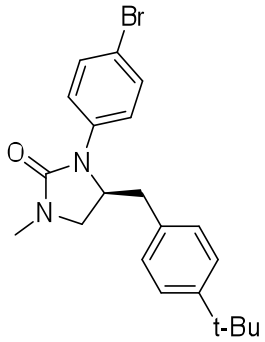

STANDARD PROTON PARAMETERS
Sample Name:
Data Collected on:
Sn.Chem.LSA.UMich.edu-inova500
Archive directory:
Sample directory:
FidFile: BAH-II-124
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 21 2012
Agilent Technologies
8
7
6
5
4
3
2
1
0
ppm
3.70 1.95
1.89
0.99
1.00 1.08 0.89
1.02 2.91
8.72


Br
O
N
N
t-Bu

2d


Carbon-13
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-II-124carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 20 2012
Agilent Technologies
160
140
120
100
80
60
40
20
ppm

6/11/2012 18:57:56 1 / 1



2d

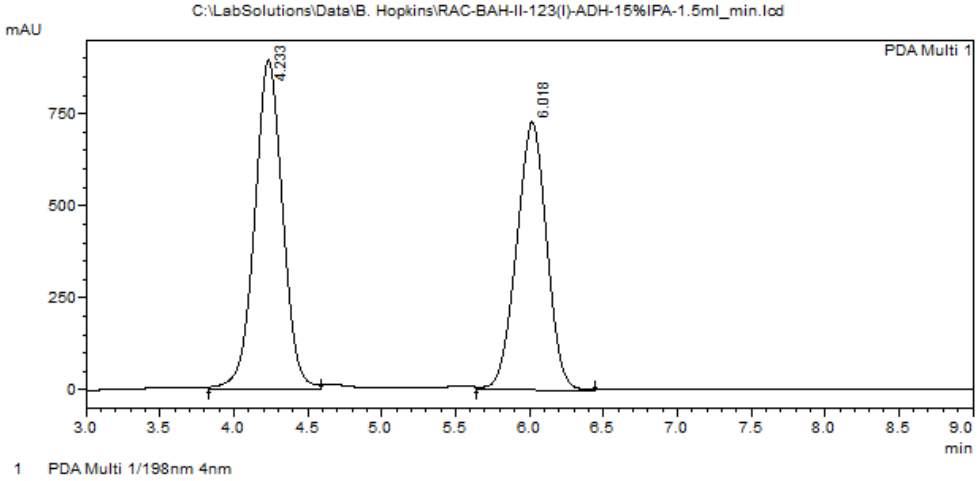
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-II-123(I)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-II-123(I)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-II-123(I)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/8/2012 1:04:51 PM
Data Processed	: 6/8/2012 2:14:54 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-II-123(I)-ADH-15%IPA-1.5ml_min.lcd



1 PDA Multi 1/198nm 4nm

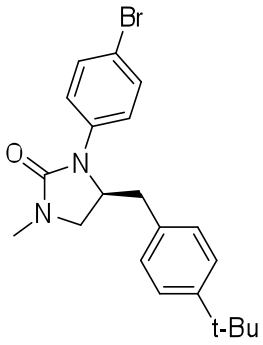
PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.233	11825411	896990	52.927	55.214
2	6.018	10517470	727586	47.073	44.786
Total		22342881	1624577	100.000	100.000

7/2/2012 15:04:56 1 / 1

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



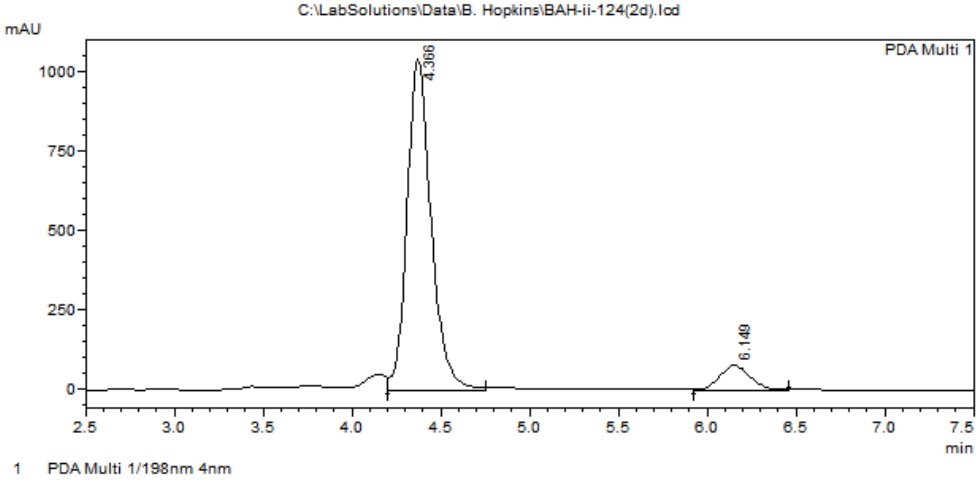
2d

C:\LabSolutions\Data\B. Hopkins\BAH-ii-124(2d).lcd

Acquired by	: Admin
Sample Name	: BAH-ii-124(2d)
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: BAH-ii-124(2d).lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/2/2012 2:45:57 PM
Data Processed	: 7/2/2012 3:00:29 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\BAH-ii-124(2d).lcd

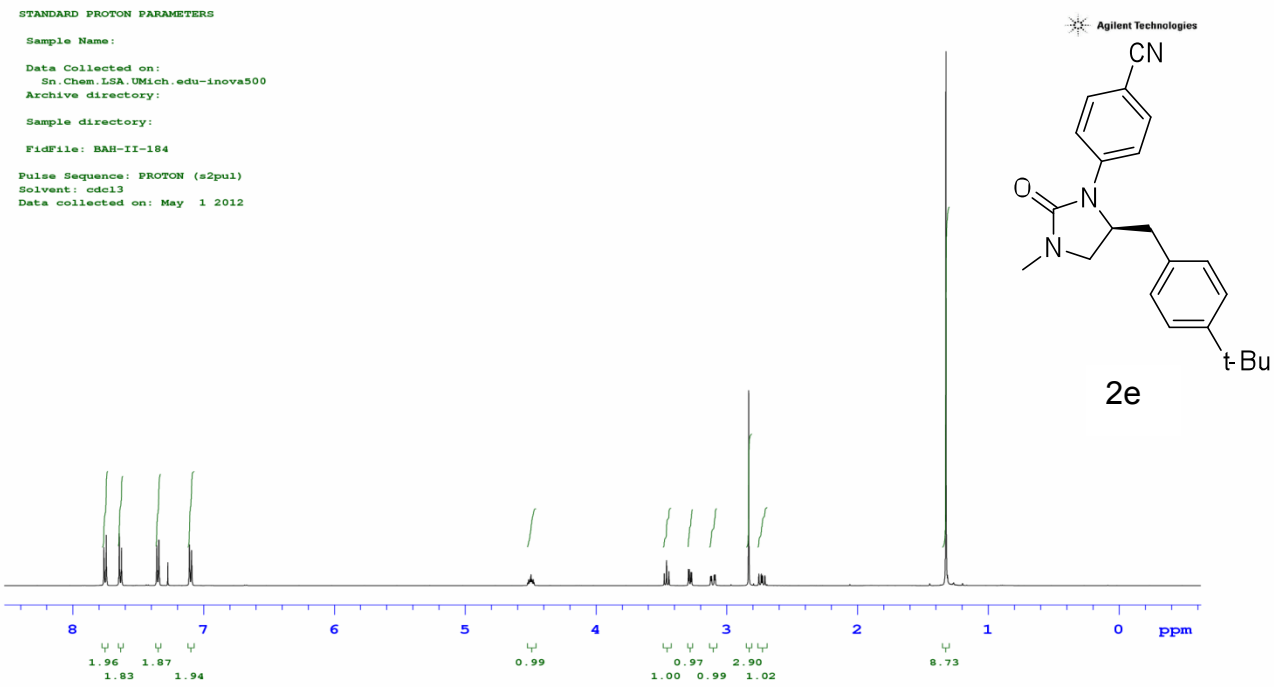


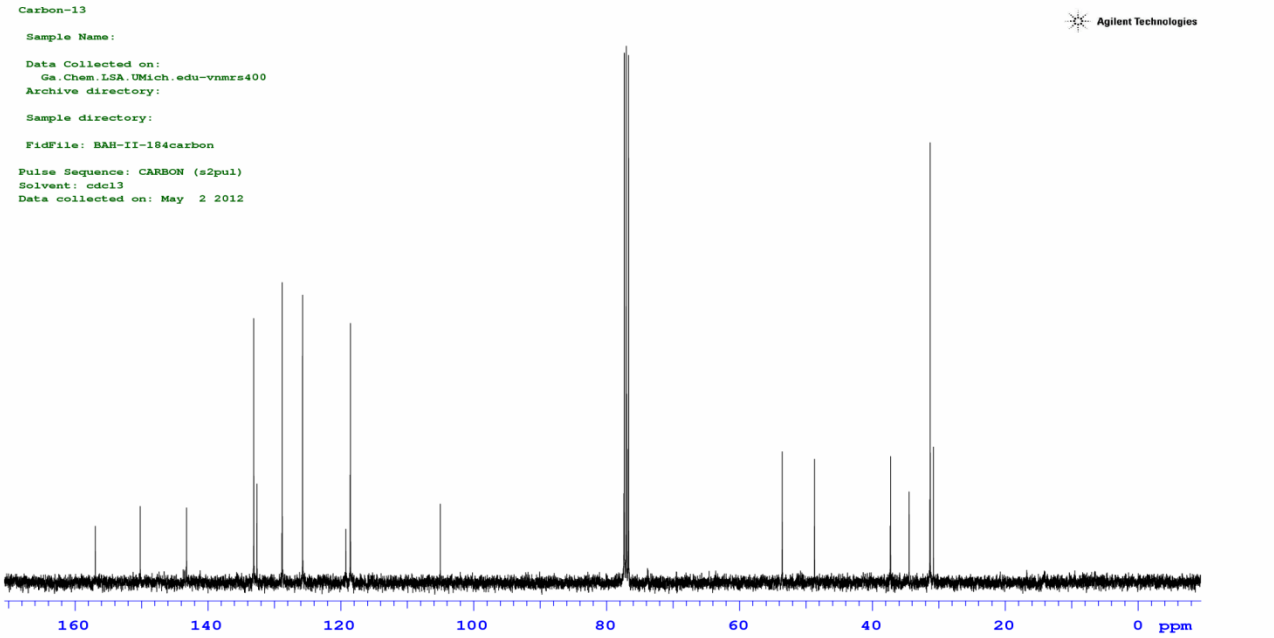
1 PDA Multi 1/198nm 4nm

PeakTable

PDA Ch1 198nm 4nm

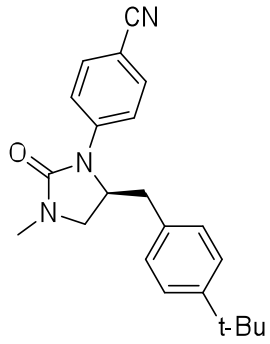
Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.366	9832740	1041963	91.726	93.193
2	6.149	886895	76108	8.274	6.807
Total		10719635	1118071	100.000	100.000


STANDARD PROTON PARAMETERS
Sample Name:
Data Collected on:
Sn.Chem.LSA.UMich.edu-inova500
Archive directory:
Sample directory:
FidFile: BAH-II-184
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 1 2012
Agilent Technologies
CN
O
N
N
t-Bu
2e
8
7
6
5
4
3
2
1
0
ppm
1.96
1.87
1.83
1.94
0.99
0.97
2.90
1.00
0.99
1.02
8.73


Carbon-13
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-II-184carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 2 2012
Agilent Technologies
160
140
120
100
80
60
40
20
0
ppm

6/6/2012 16:34:40 1 / 1

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

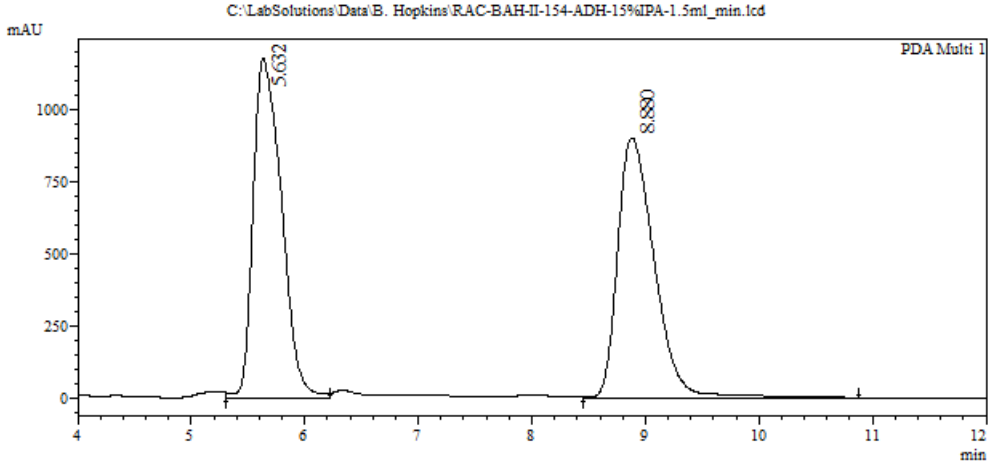


2e

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-II-154-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-II-154-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-II-154-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/6/2012 9:42:40 AM
Data Processed	: 6/6/2012 10:43:59 AM

<Chromatogram>



1 PDA Multi 1/198nm 4nm

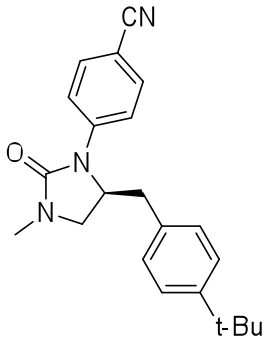
PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.632	20393415	1174136	50.274	56.635
2	8.880	20170873	899024	49.726	43.365
Total		40564288	2073160	100.000	100.000

6/6/2012 18:09:38 1 / 1

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

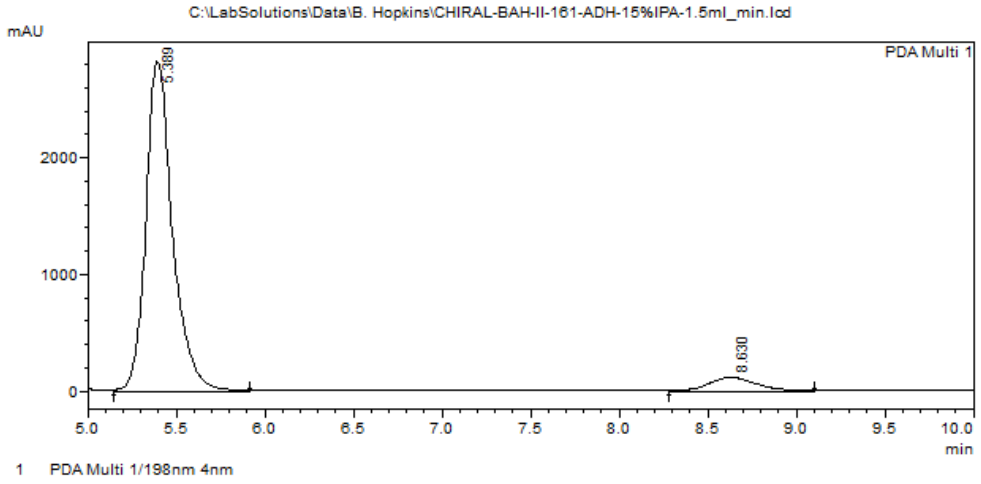


2e

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-II-161-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-II-161-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-II-161-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/6/2012 5:46:19 PM
Data Processed	: 6/6/2012 6:04:50 PM

<Chromatogram>

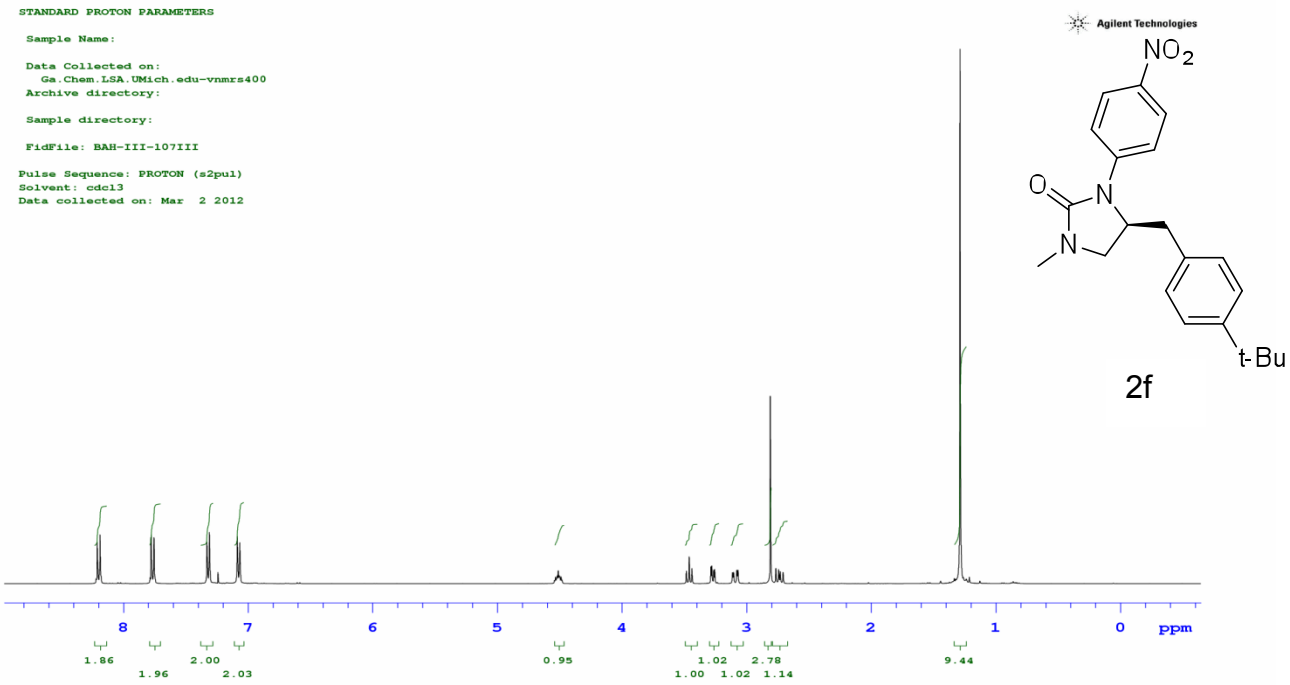


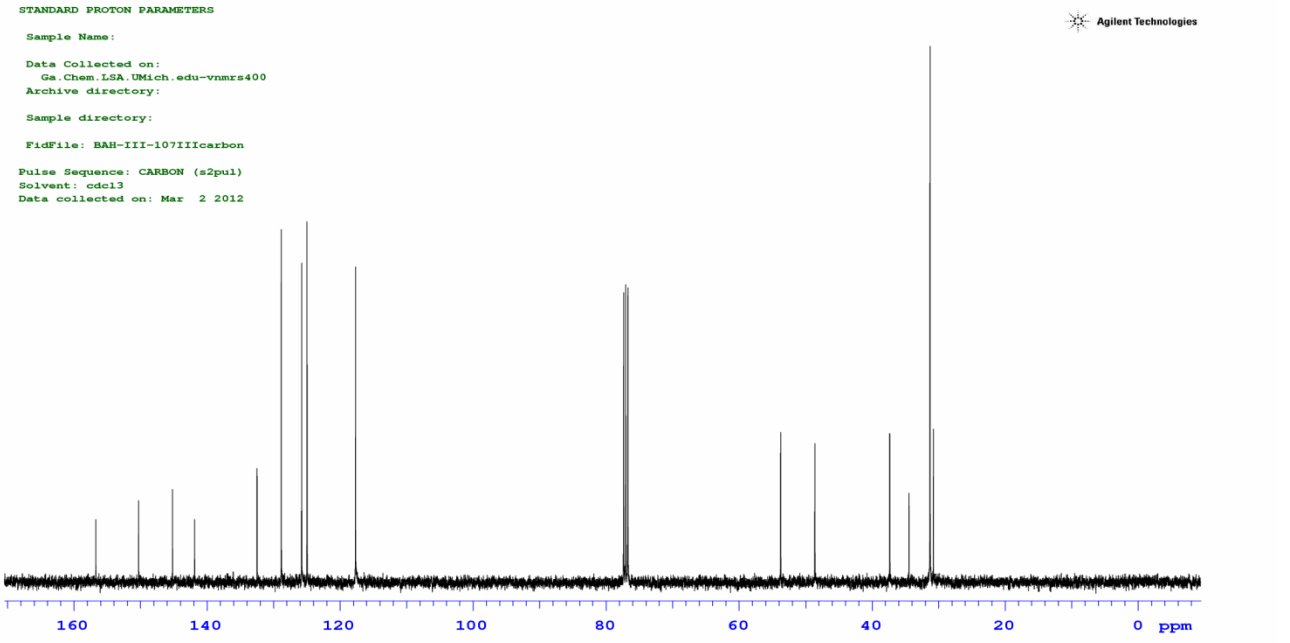
1 PDA Multi 1/198nm 4nm

PeakTable

PDA Ch1 198nm 4nm

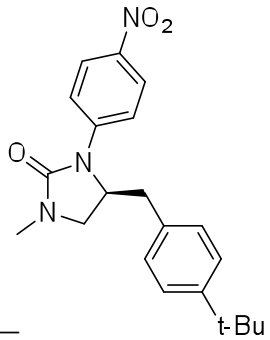
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.389	29555975	2828317	93.075	96.056
2	8.630	2199167	116134	6.925	3.944
Total		31755142	2944451	100.000	100.000


STANDARD PROTON PARAMETERS
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-107III
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 2 2012
Agilent Technologies
NO2
O
N
N
t-Bu
2f
8
7
6
5
4
3
2
1
0
ppm
1.86
1.96
2.00
2.03
0.95
1.00
1.02
2.78
1.02
1.14
9.44


STANDARD PROTON PARAMETERS
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-107IIIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 2 2012
Agilent Technologies
160
140
120
100
80
60
40
20
0
ppm

6/7/2012 15:59:44 1 / 1

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

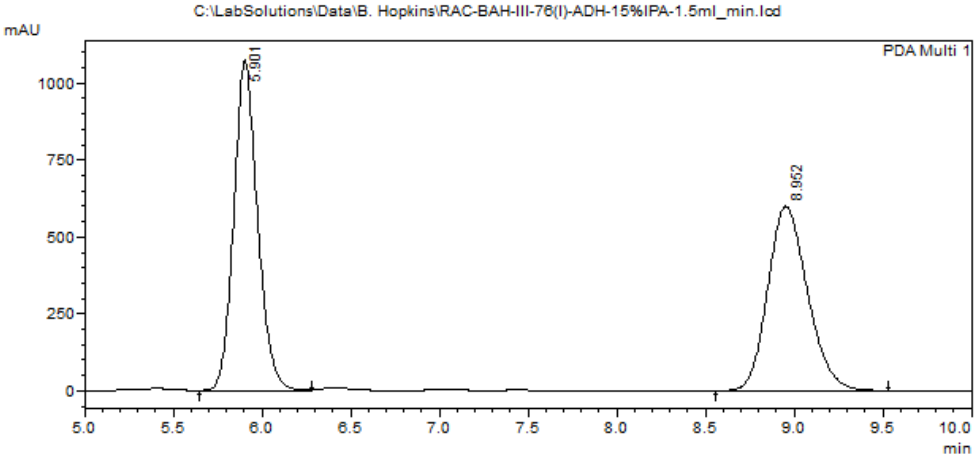


2f

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-76(I)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-76(I)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-76(I)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/30/2012 4:42:55 PM
Data Processed	: 1/30/2012 5:02:18 PM

<Chromatogram>



1 PDA Multi 1/198nm 4nm

PeakTable

PDA Ch1 198nm 4nm

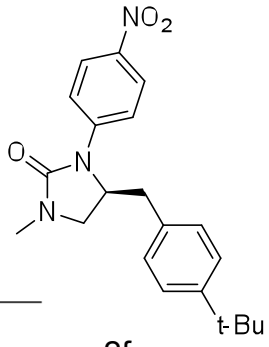
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.901	10040148	1075460	51.781	64.206
2	8.952	9349642	599557	48.219	35.794
Total		19389790	1675017	100.000	100.000

6/11/2012 12:52:09 1 / 1

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

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-107(III)-ADH-15%IPA-1.5ml_min.lcd

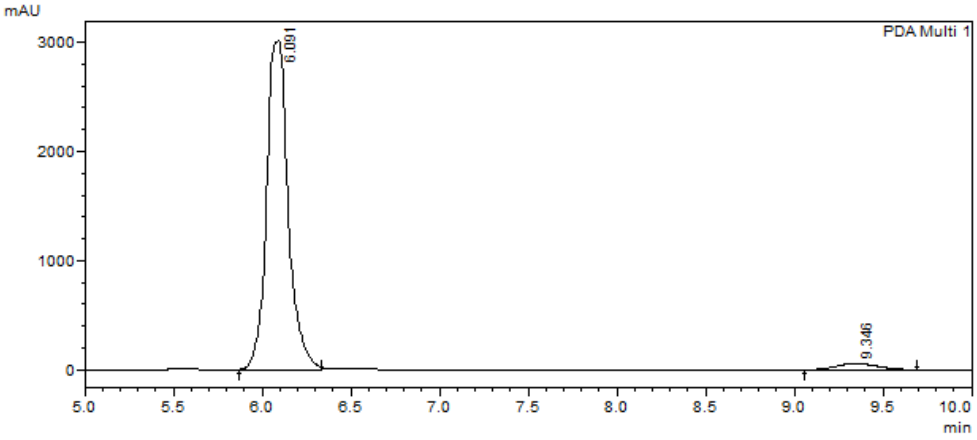
Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-107(III)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-107(III)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/29/2012 5:57:39 PM
Data Processed	: 2/29/2012 6:18:12 PM



2f

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-107(III)-ADH-15%IPA-1.5ml_min.lcd



1 PDA Multi 1/198nm 4nm

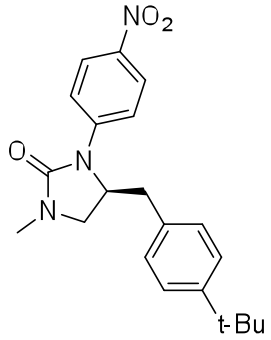
PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.091	25616432	3013402	96.193	98.044
2	9.346	1013941	60112	3.807	1.956
Total		26630373	3073513	100.000	100.000

6/12/2012 18:41:57 1 / 1

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

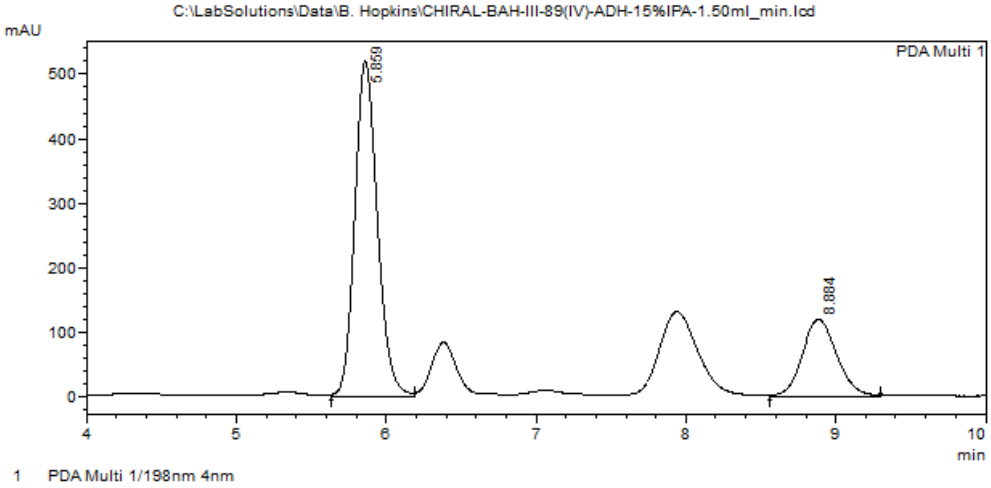


2f (from 4-iodo-*tert*-butylbenzene)

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-89(IV)-ADH-15%IPA-1.50ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-89(IV)-ADH-15%IPA-1.50ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-89(IV)-ADH-15%IPA-1.50ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/9/2012 6:08:03 PM
Data Processed	: 2/9/2012 6:39:31 PM

<Chromatogram>

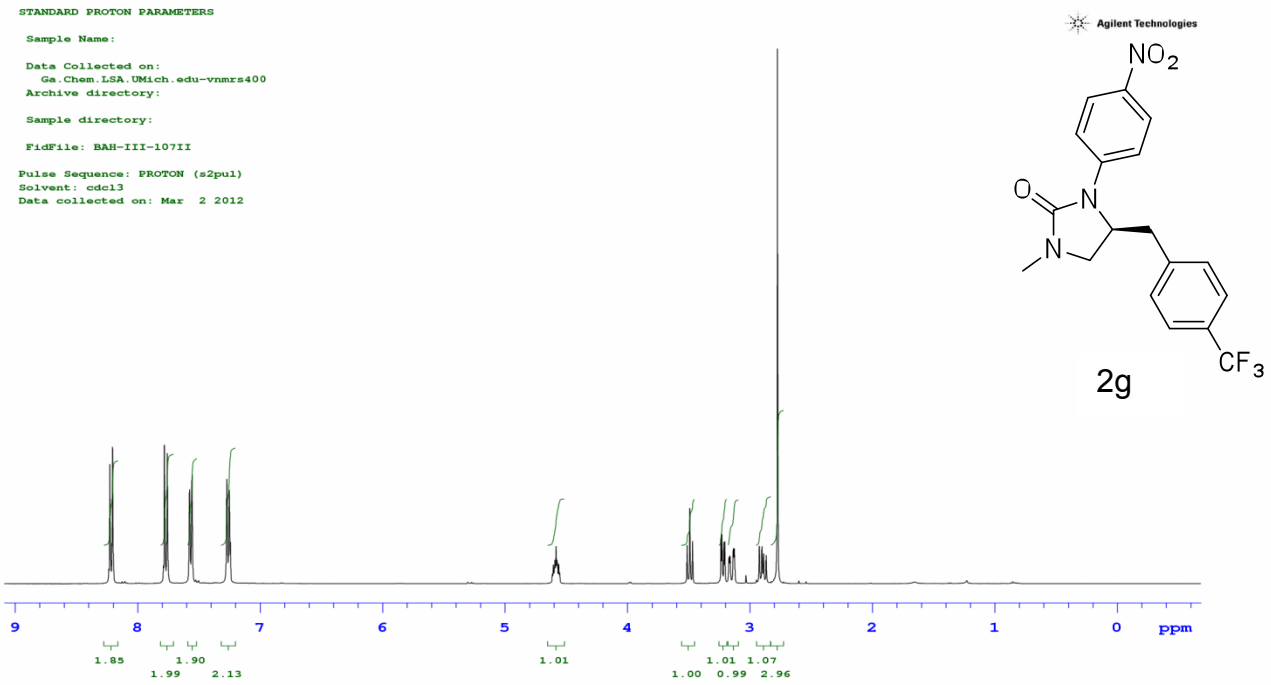


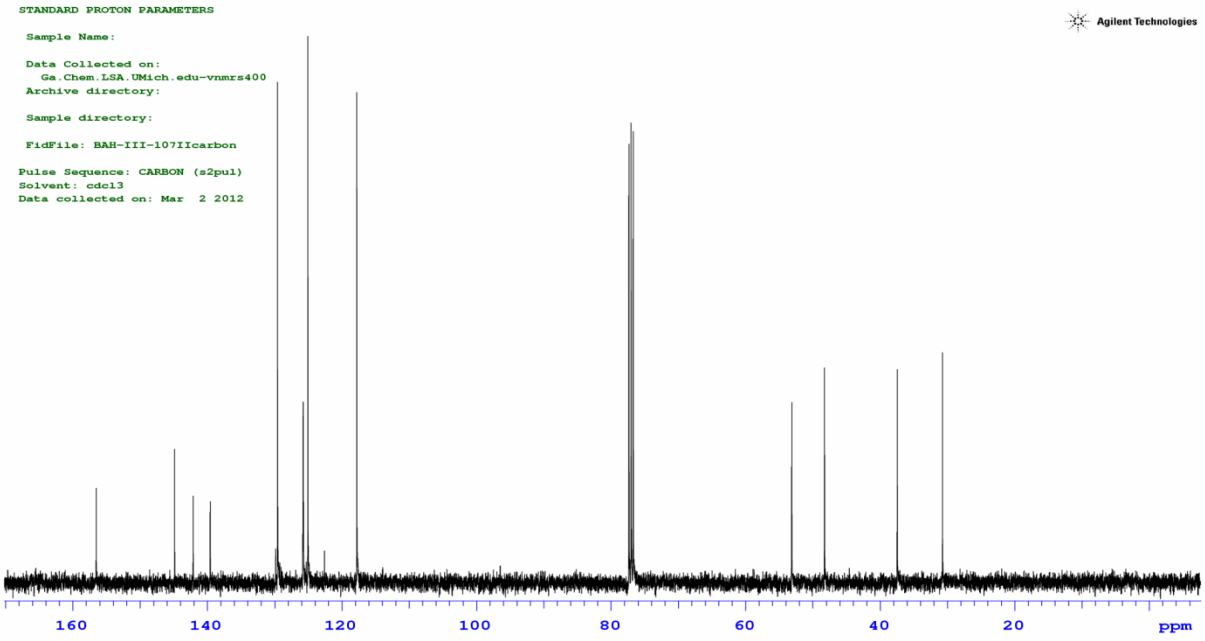
1 PDA Multi 1/198nm 4nm

PeakTable

PDA Ch1 198nm 4nm

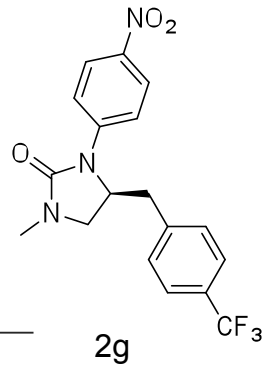
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.859	5434174	520821	73.536	81.277
2	8.884	1955621	119974	26.464	18.723
Total		7389795	640795	100.000	100.000


STANDARD PROTON PARAMETERS
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-107II
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 2 2012
Agilent Technologies
NO2
O
N
N
CF3
2g
9
8
7
6
5
4
3
2
1
0
ppm
1.85
1.90
1.99
2.13
1.01
1.01
1.07
1.00
0.99
2.96


STANDARD PROTON PARAMETERS
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-107IIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 2 2012
Agilent Technologies
160
140
120
100
80
60
40
20
ppm

6/12/2012 15:59:25 1 / 1

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

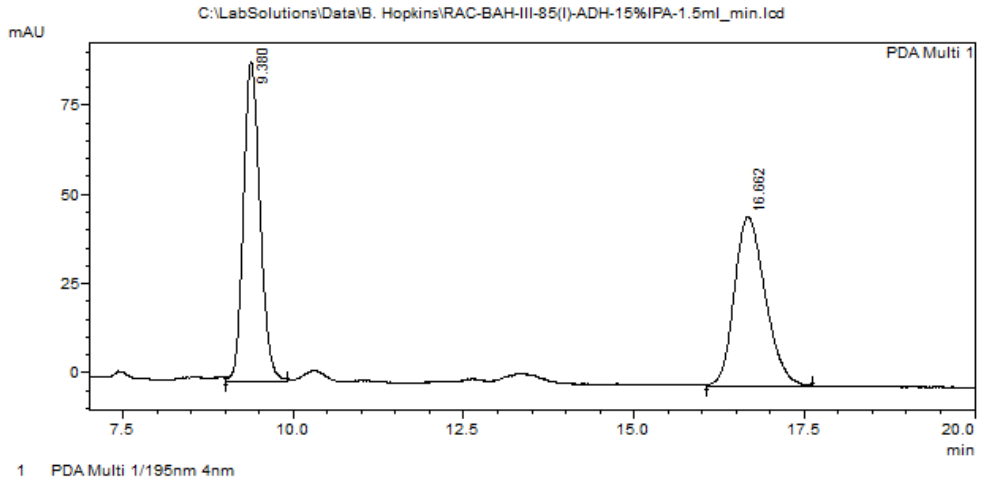


2g

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-85(I)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-85(I)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-85(I)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/7/2012 10:56:40 AM
Data Processed	: 2/7/2012 11:32:49 AM

<Chromatogram>



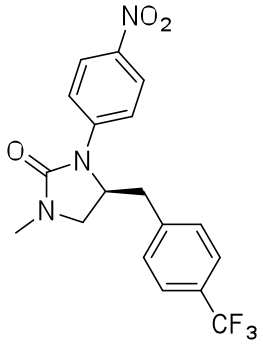
1 PDA Multi 1/195nm 4nm

PeakTable

PDA Ch1 195nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.380	1508512	89477	50.198	65.373
2	16.662	1496638	47394	49.802	34.627
Total		3005150	136871	100.000	100.000

6/12/2012 15:56:05 1 / 1



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

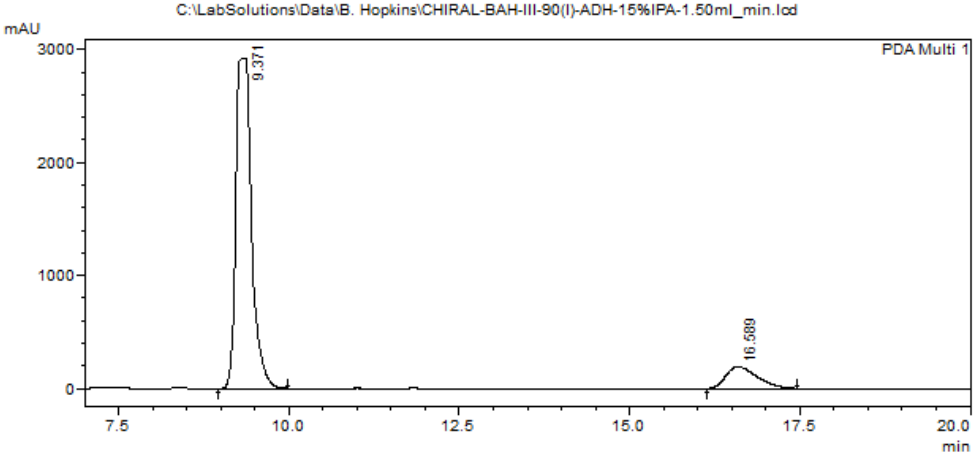
C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-90(I)-ADH-15%IPA-1.50ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-90(I)-ADH-15%IPA-1.50ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-90(I)-ADH-15%IPA-1.50ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/13/2012 4:04:52 PM
Data Processed	: 2/13/2012 4:35:11 PM

2g (without H_2O)

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-90(I)-ADH-15%IPA-1.50ml_min.lcd



1 PDA Multi 1/195nm 4nm

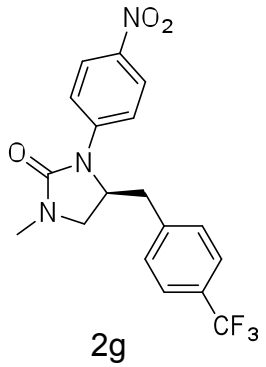
PeakTable

PDA Ch1 195nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.371	46823253	2916831	88.289	93.772
2	16.589	6211061	193724	11.711	6.228
Total		53034313	3110555	100.000	100.000

6/12/2012 15:49:38 1 / 1

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

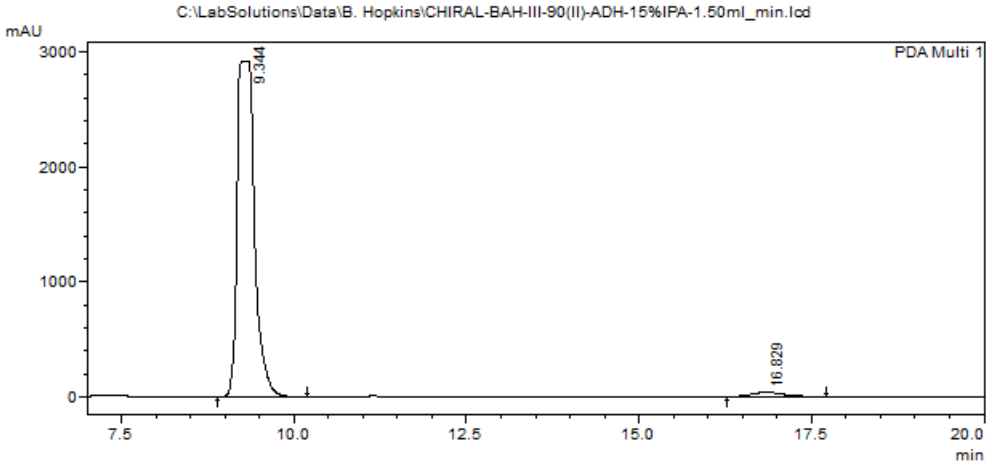


2g

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-90(II)-ADH-15%IPA-1.50ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-90(II)-ADH-15%IPA-1.50ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-90(II)-ADH-15%IPA-1.50ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/13/2012 4:36:35 PM
Data Processed	: 2/13/2012 5:03:59 PM

<Chromatogram>

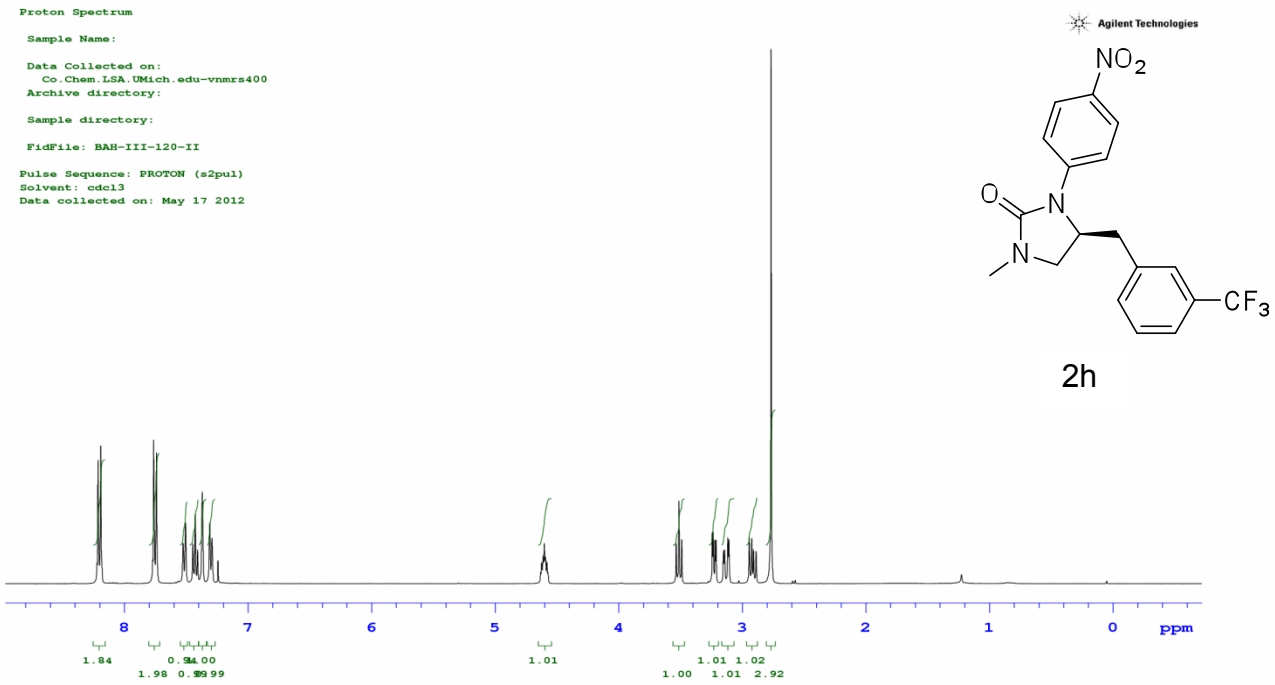


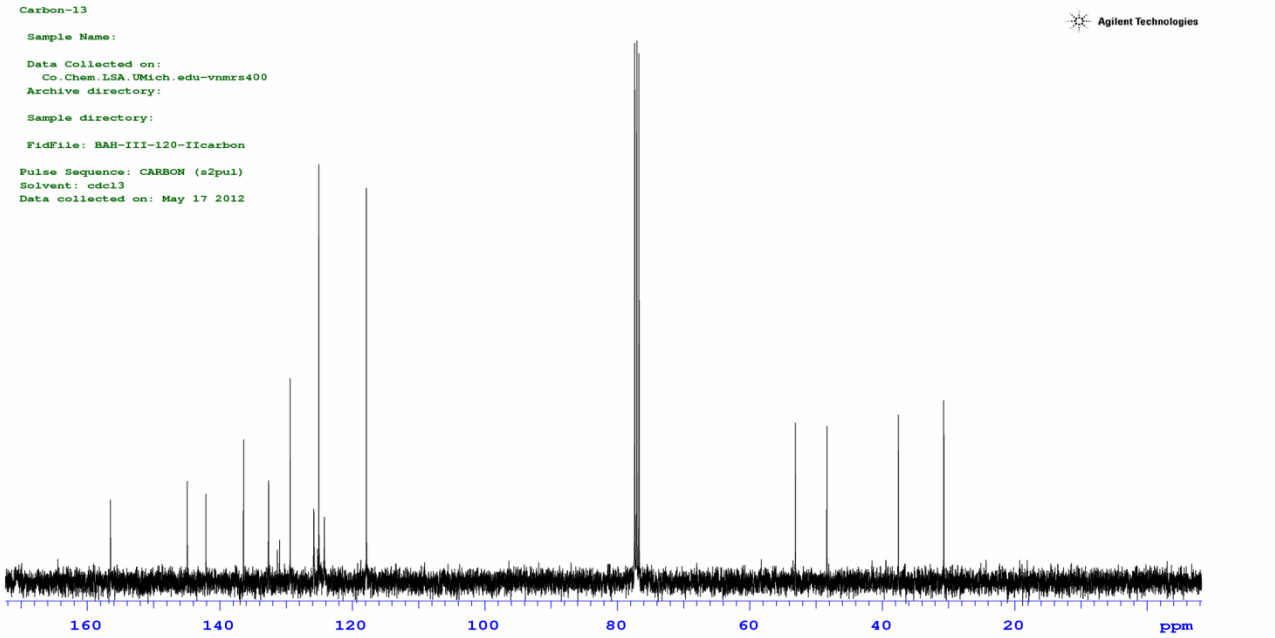
1 PDA Multi 1/195nm 4nm

PeakTable

PDA Ch1 195nm 4nm

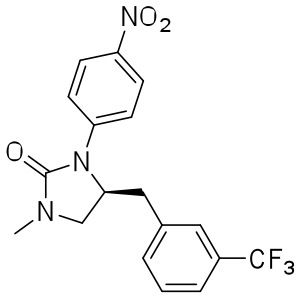
Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.344	51255265	2916724	97.507	98.710
2	16.829	1310197	38112	2.493	1.290
Total		52565462	2954836	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-120-II
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 17 2012
Agilent Technologies
NO2
O
N
N
CF3
2h
8
7
6
5
4
3
2
1
0
ppm
1.84
1.98
1.01
1.00
1.01
1.02
1.01
2.92


Carbon-13
Sample Name:
Data Collected on:
Co.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-120-IIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 17 2012
Agilent Technologies
160
140
120
100
80
60
40
20
ppm

6/12/2012 16:04:29 1 / 1

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

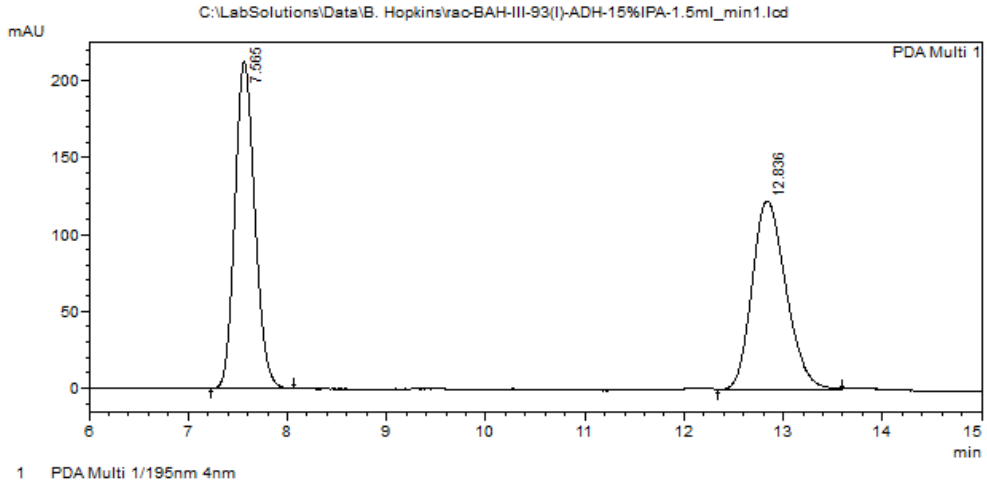


2h

C:\LabSolutions\Data\B. Hopkins\rac-BAH-III-93(I)-ADH-15%IPA-1.5ml_min1.lcd

Acquired by	: Admin
Sample Name	: rac-BAH-III-93(I)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: rac-BAH-III-93(I)-ADH-15%IPA-1.5ml_min1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/16/2012 3:35:31 PM
Data Processed	: 2/16/2012 4:40:03 PM

<Chromatogram>



1 PDA Multi 1/195nm 4nm

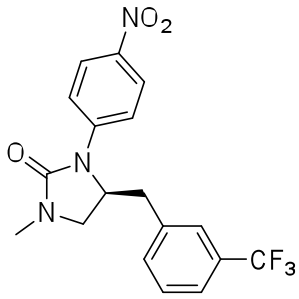
PeakTable

PDA Ch1 195nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.565	2958662	212608	50.414	63.420
2	12.836	2910088	122632	49.586	36.580
Total		5868750	335239	100.000	100.000

7/2/2012 17:01:35 1 / 1

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



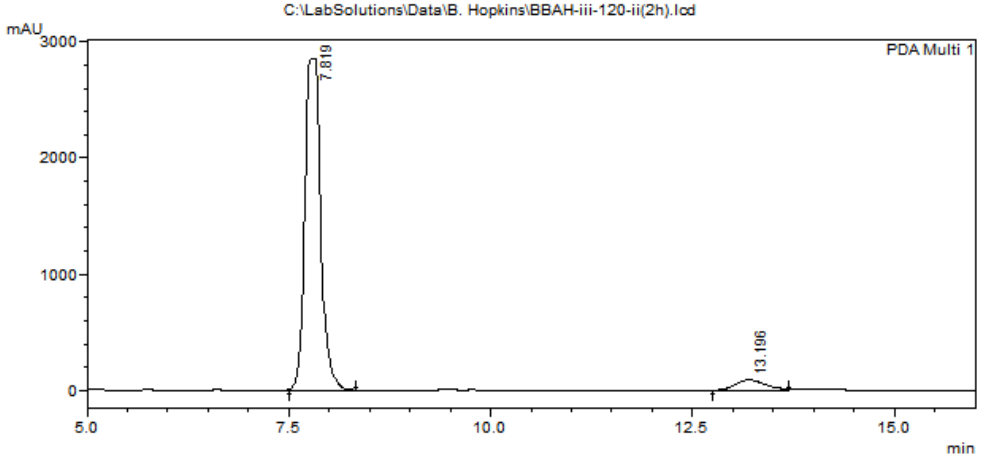
2h

C:\LabSolutions\Data\B. Hopkins\BBAH-iii-120-ii(2h).lcd

Acquired by	: Admin
Sample Name	: BAH-iii-120-ii(2h)
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: BBAH-iii-120-ii(2h).lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/2/2012 4:42:41 PM
Data Processed	: 7/2/2012 4:59:16 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\BBAH-iii-120-ii(2h).lcd

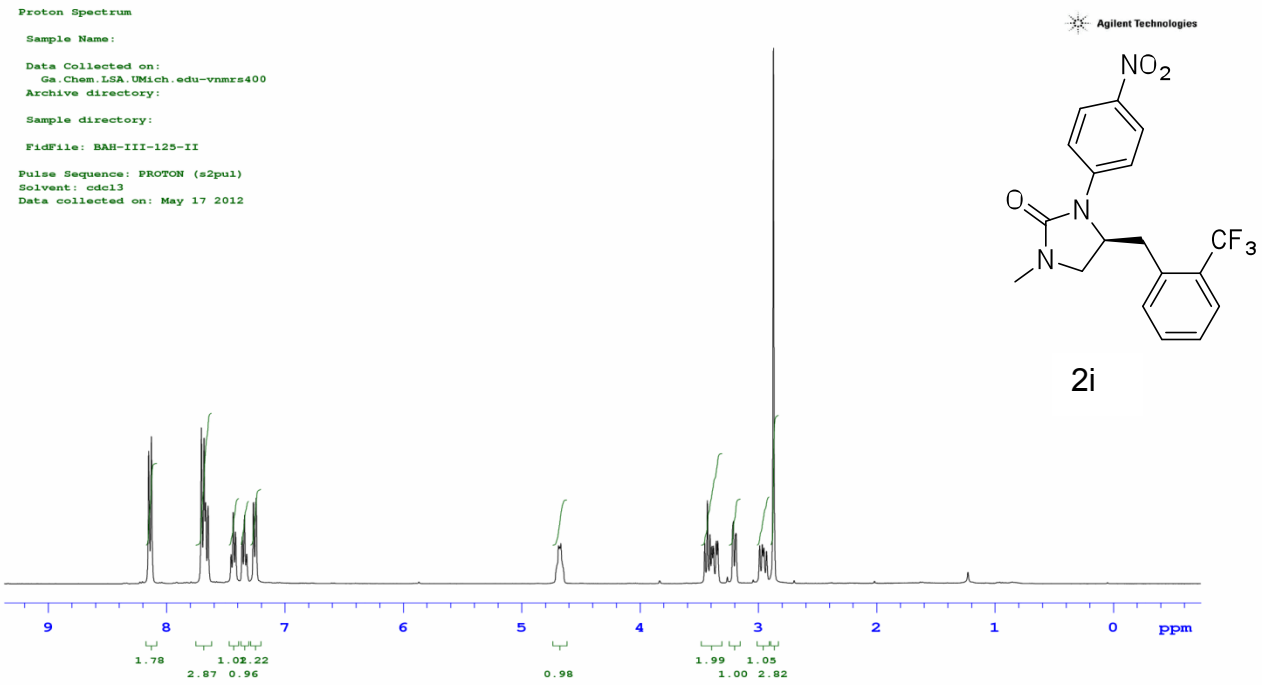


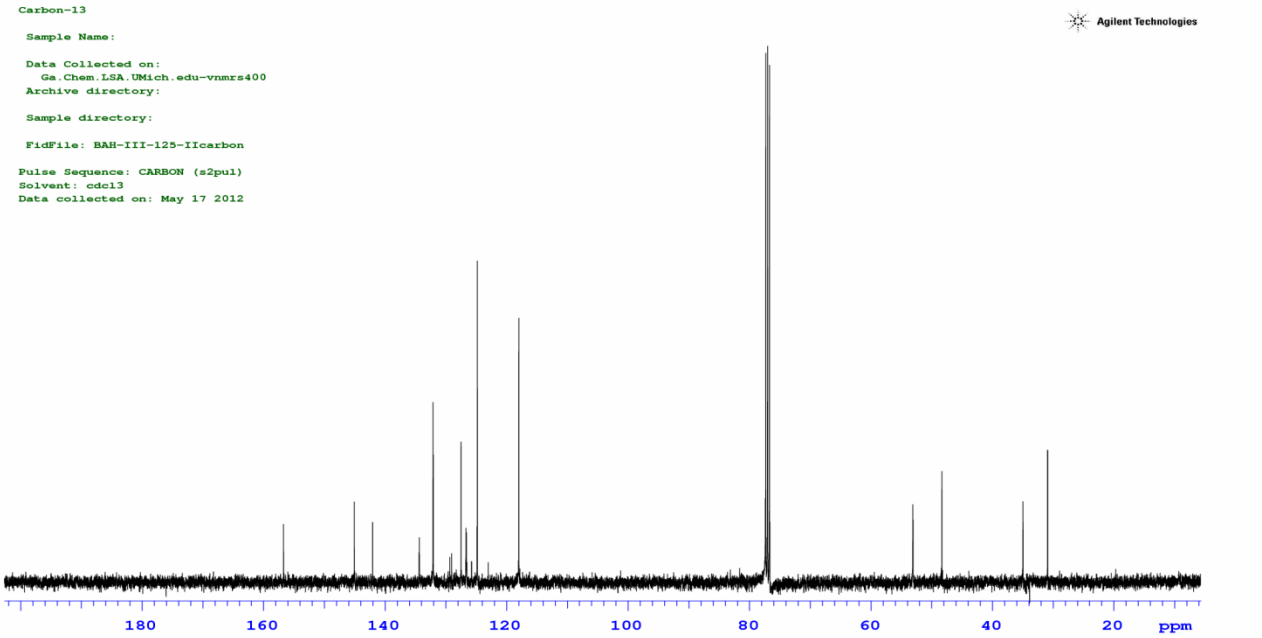
1 PDA Multi 1/195nm 4nm

PeakTable

PDA Ch1 195nm 4nm

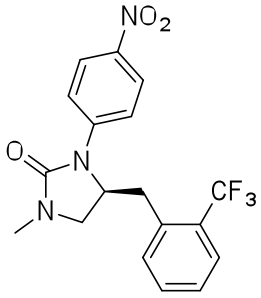
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.819	39038134	2849387	94.088	96.900
2	13.196	2452853	91156	5.912	3.100
Total		41490987	2940543	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-125-II
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 17 2012
Agilent Technologies
NO2
O
N
N
CF3
2i
9
8
7
6
5
4
3
2
1
0
ppm
1.78
1.02
2.22
2.87
0.96
0.98
1.99
1.05
1.00
2.82


Carbon-13
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-125-IIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 17 2012
Agilent Technologies
180
160
140
120
100
80
60
40
20
ppm

6/12/2012 16:13:15 1 / 1

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

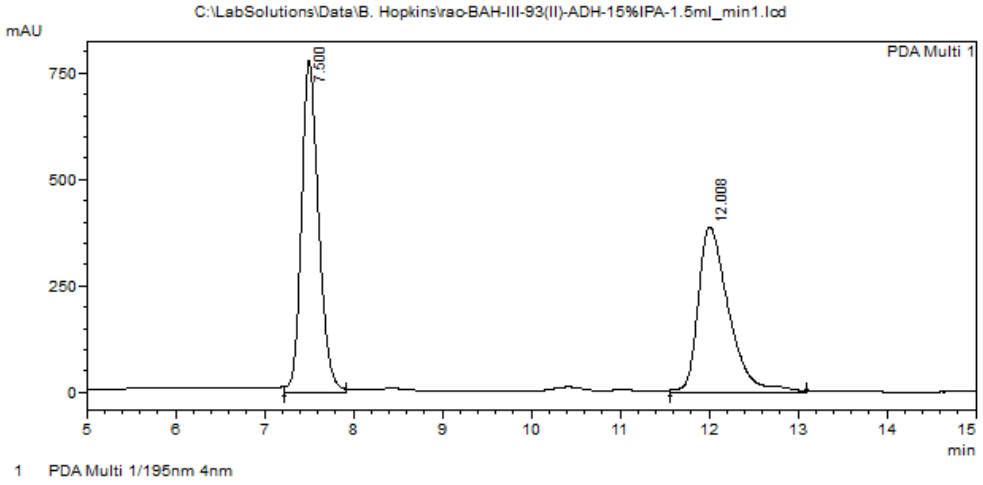


2i

C:\LabSolutions\Data\B. Hopkins\rac-BAH-III-93(II)-ADH-15%IPA-1.5ml_min1.lcd

Acquired by	: Admin
Sample Name	: rac-BAH-III-93(II)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: rac-BAH-III-93(II)-ADH-15%IPA-1.5ml_min1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/16/2012 5:53:49 PM
Data Processed	: 2/16/2012 6:11:50 PM

<Chromatogram>



1 PDA Multi 1/195nm 4nm

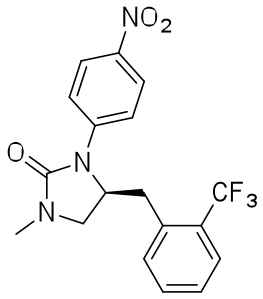
PeakTable

PDA Ch1 195nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.500	10438937	778284	52.514	66.610
2	12.008	9439517	390144	47.486	33.390
Total		19878454	1168428	100.000	100.000

7/2/2012 15:47:45 1 / 1

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



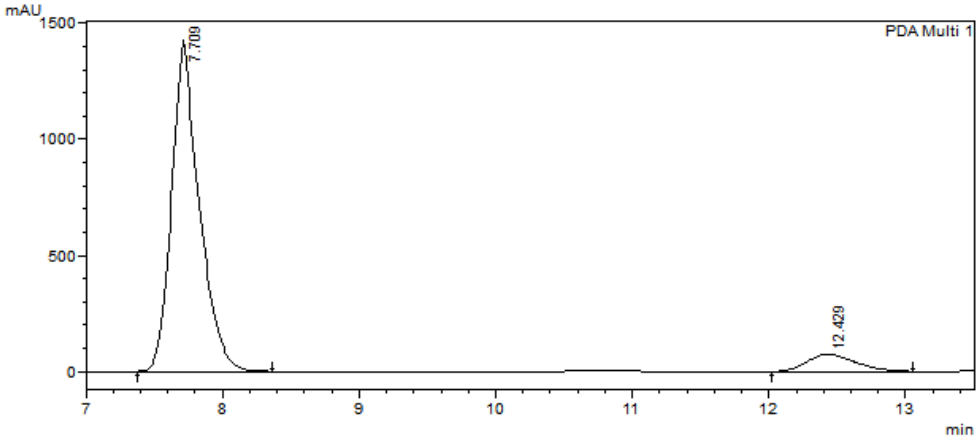
2i

C:\LabSolutions\Data\B. Hopkins\BAH-iii-125-ii(2i).lcd

Acquired by	: Admin
Sample Name	: BAH-iii-125-ii(2i)
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: BAH-iii-125-ii(2i).lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/2/2012 3:23:41 PM
Data Processed	: 7/2/2012 3:41:47 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\BAH-iii-125-ii(2i).lcd

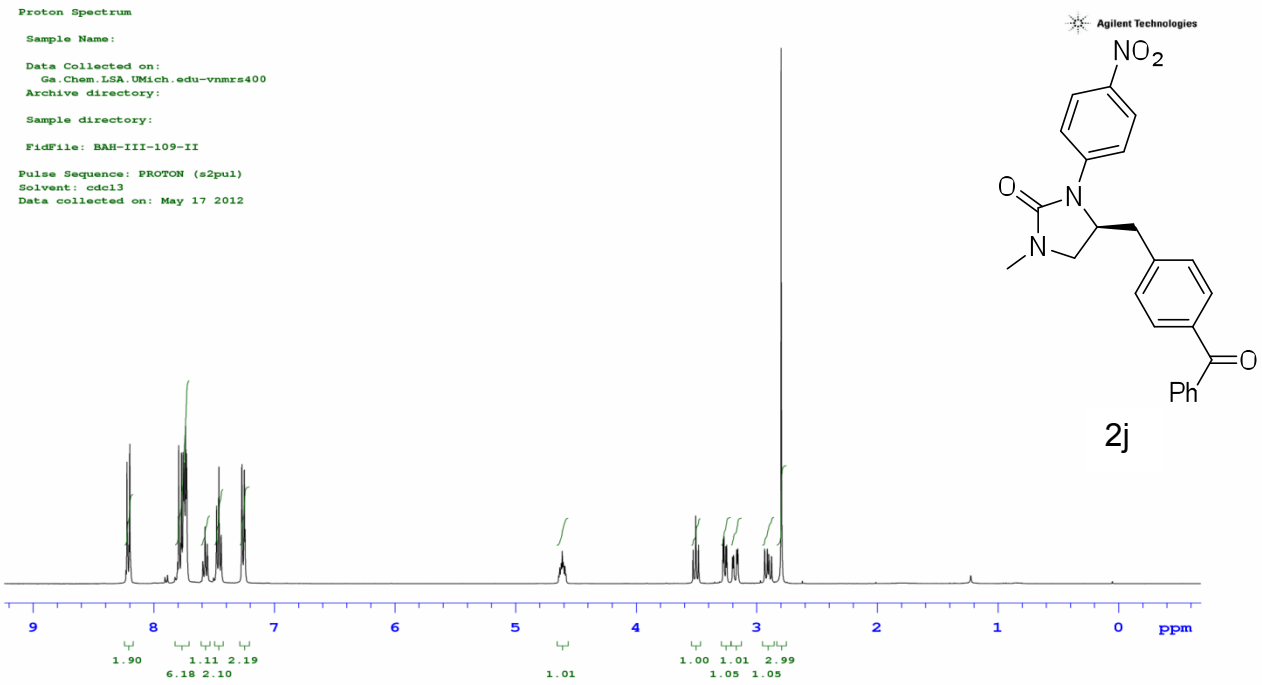


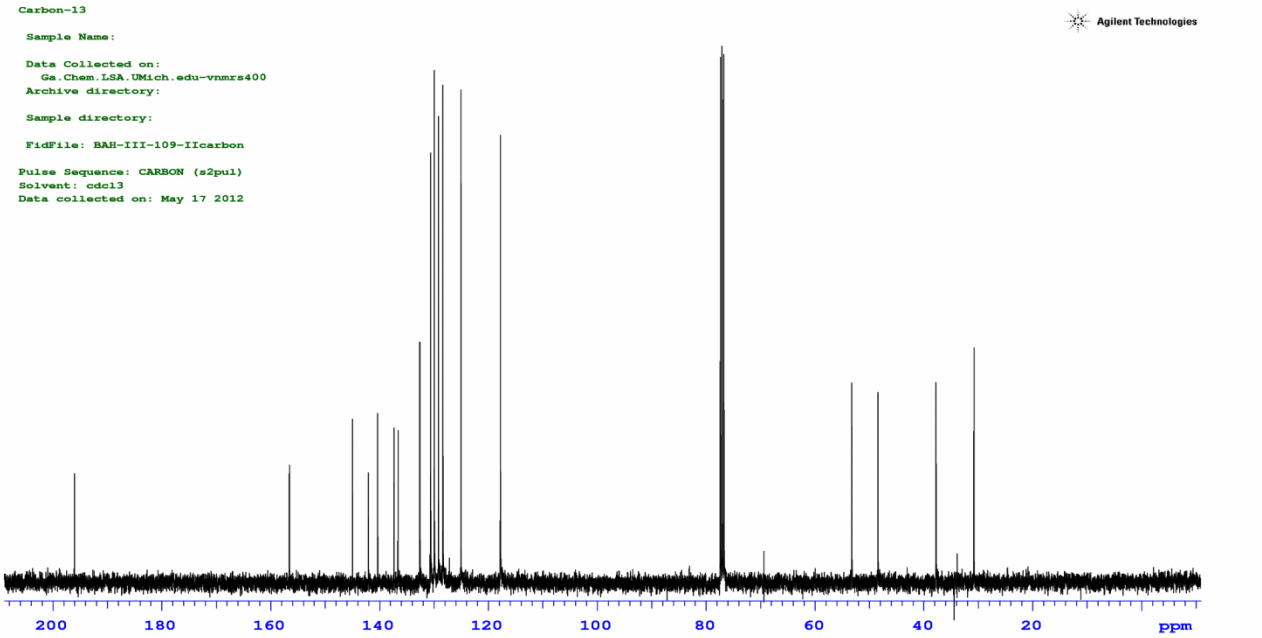
1 PDA Multi 1/195nm 4nm

PeakTable

PDA Ch1 195nm 4nm

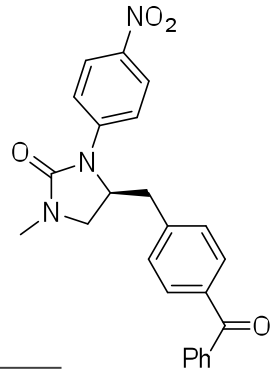
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.709	19948380	1426731	91.476	95.096
2	12.429	1858926	73572	8.524	4.904
Total		21807306	1500302	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-109-II
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 17 2012
Agilent Technologies
NO2
O
N
N
O
Ph
2j
9
8
7
6
5
4
3
2
1
0
ppm
1.90
1.11
2.19
6.18
2.10
1.01
1.00
1.01
2.99
1.05
1.05


Carbon-13
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-109-IIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 17 2012
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
ppm

6/18/2012 15:41:35 1 / 1

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

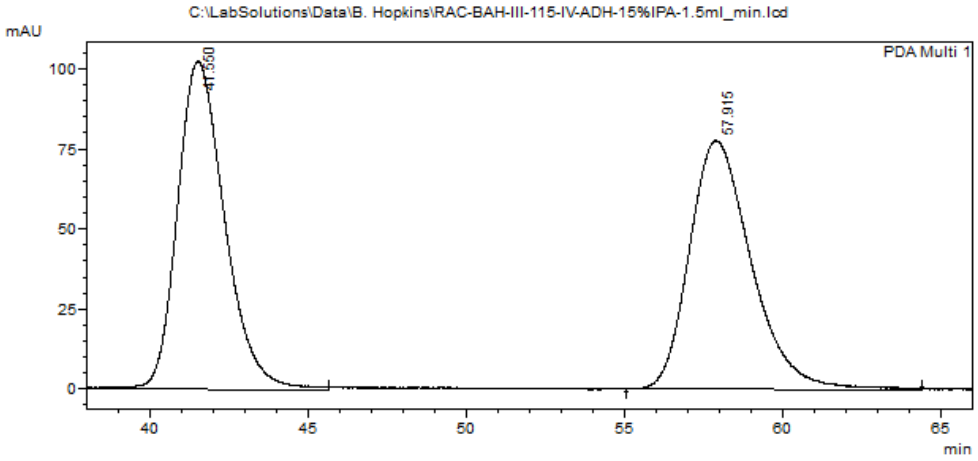


2j

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-115-IV-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-115-IV-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-115-IV-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/18/2012 2:11:20 PM
Data Processed	: 6/18/2012 3:21:23 PM

<Chromatogram>



1 PDA Multi 1/195nm 4nm

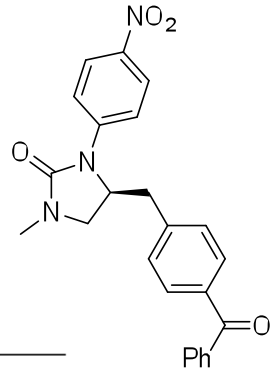
PeakTable

PDA Ch1 195nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	41.550	10651685	102441	50.245	56.903
2	57.915	10547826	77586	49.755	43.097
Total		21199511	180027	100.000	100.000

6/15/2012 17:14:00 1 / 1

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



2j

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-109(II)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-109(II)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-109(II)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/4/2012 2:44:19 PM
Data Processed	: 3/4/2012 3:54:22 PM

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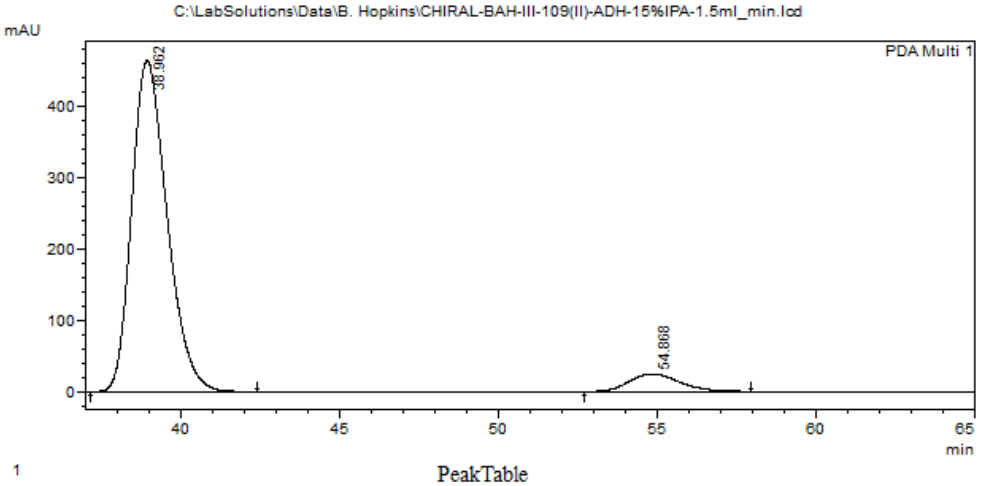
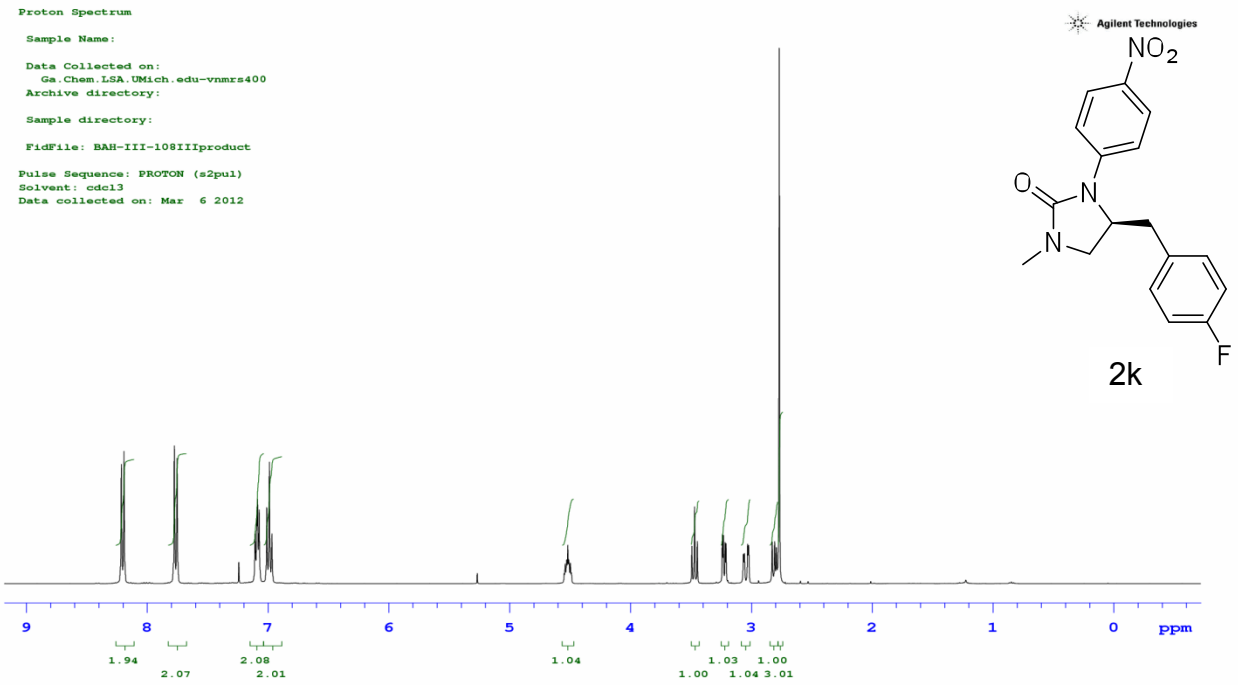


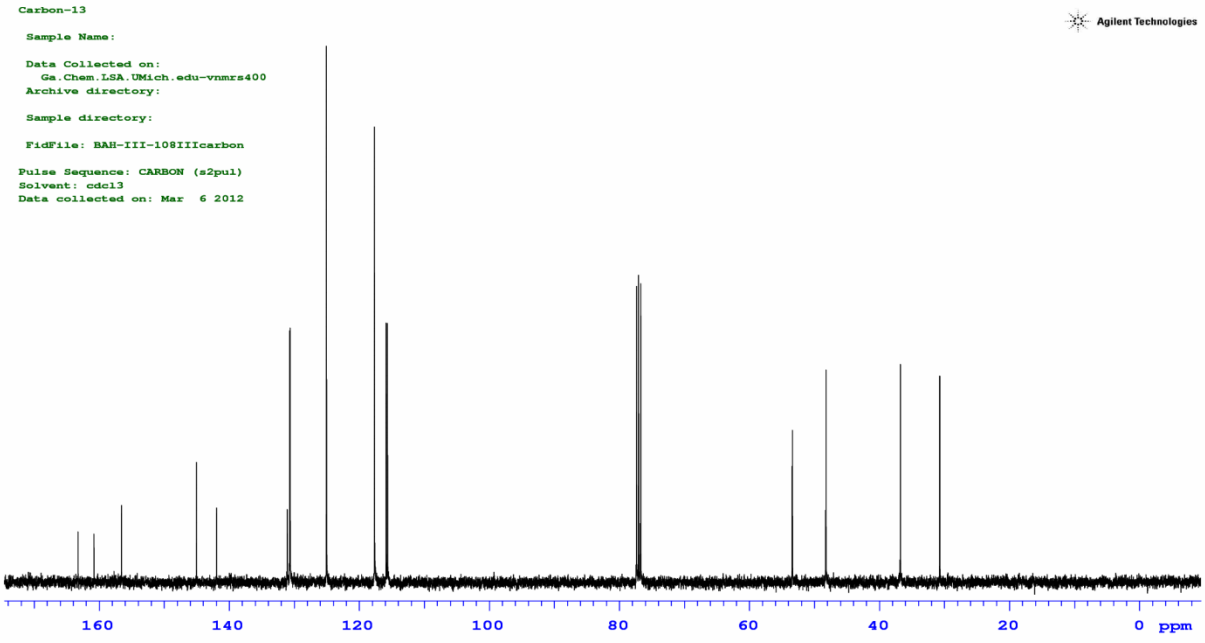
1

PeakTable

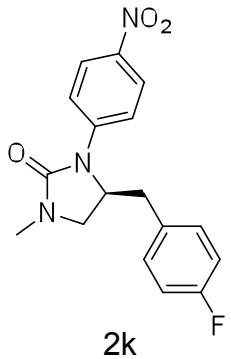
PDA Ch1 195nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	38.962	35917342	463475	92.764	94.869
2	54.868	2801554	25068	7.236	5.131
Total		38718896	488543	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-108IIIproduct
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 6 2012
Agilent Technologies
NO2
O
N
N
F
2k
9
8
7
6
5
4
3
2
1
0
ppm
1.94
2.07
2.08
2.01
1.04
1.00
1.03
1.04
1.00
3.01


Carbon-13
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-108IIIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 6 2012
Agilent Technologies
160
140
120
100
80
60
40
20
0
ppm

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

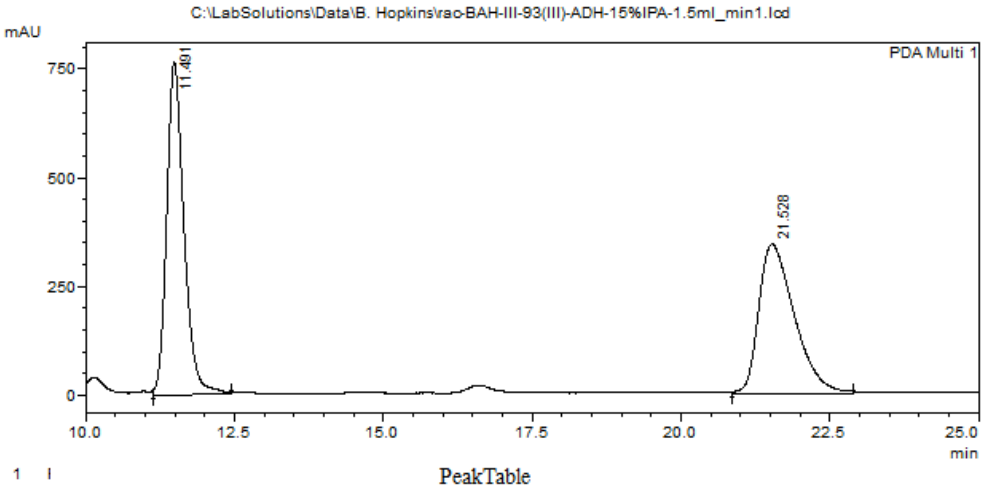


2k

C:\LabSolutions\Data\B. Hopkins\rac-BAH-III-93(III)-ADH-15%IPA-1.5ml_min1.lcd

Acquired by	: Admin
Sample Name	: rac-BAH-III-93(III)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: rac-BAH-III-93(III)-ADH-15%IPA-1.5ml_min1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/16/2012 6:20:23 PM
Data Processed	: 2/16/2012 6:50:06 PM

<Chromatogram>



1

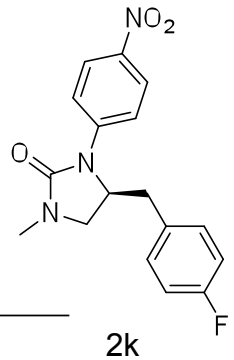
PeakTable

PDA Ch1 193nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.491	15416348	765454	51.389	68.899
2	21.528	14582919	345526	48.611	31.101
Total		29999267	1110980	100.000	100.000

6/12/2012 18:30:28 1 / 1

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



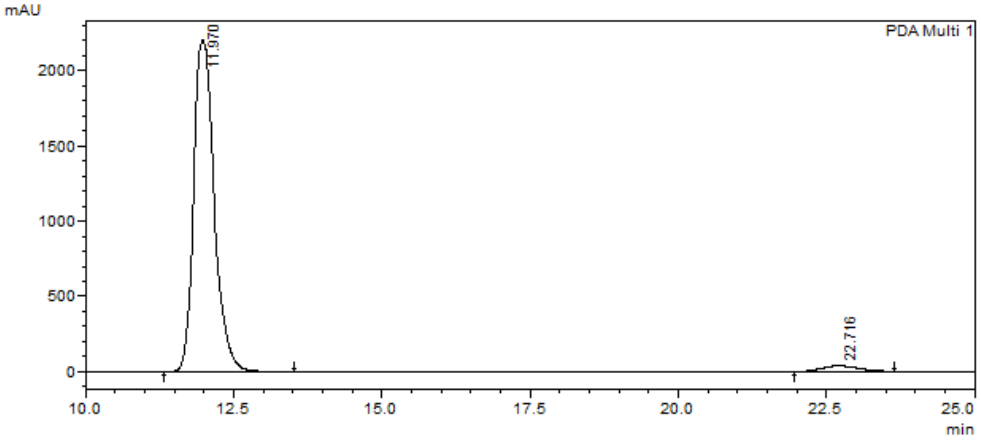
2k

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-122-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-122-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-122-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/19/2012 2:39:49 PM
Data Processed	: 3/19/2012 3:05:11 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-122-ADH-15%IPA-1.5ml_min.lcd

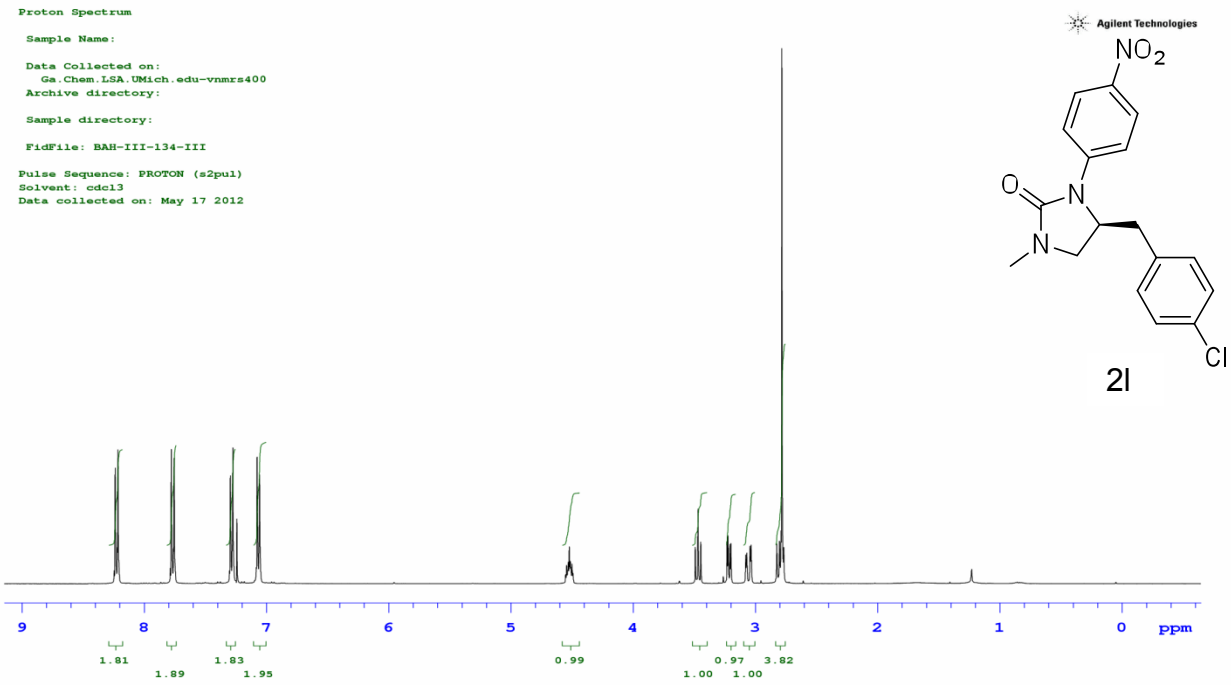


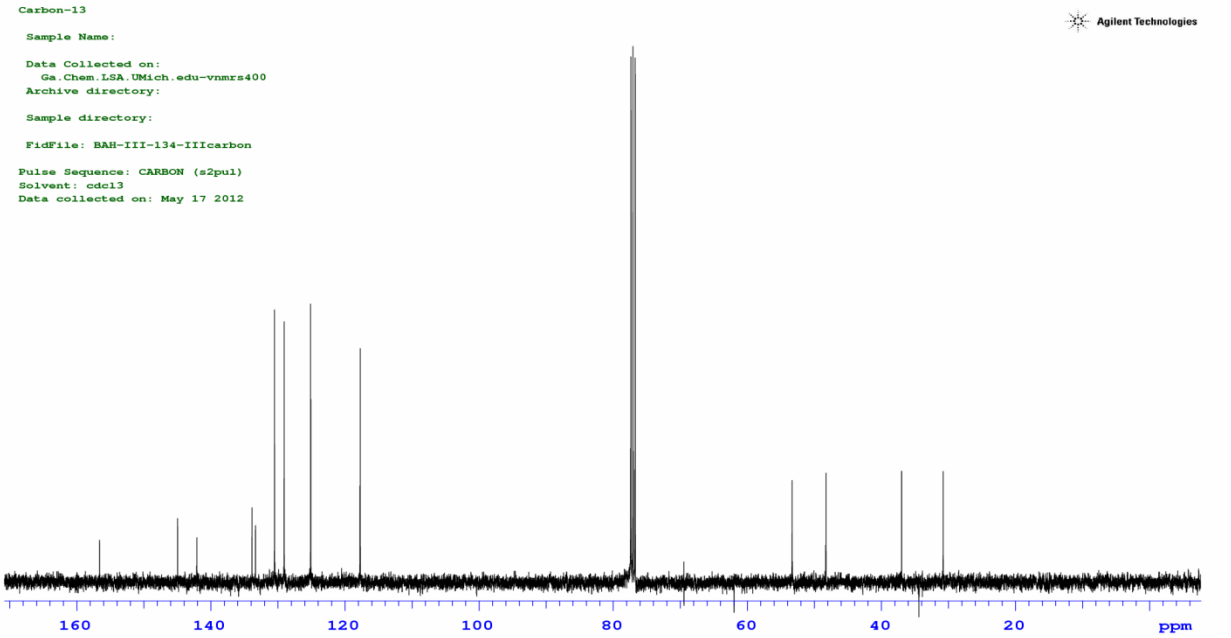
1 PDA Multi 1/193nm 4nm

PeakTable

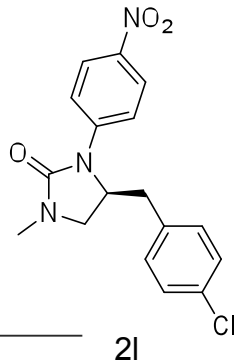
PDA Ch1 193nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.970	53026199	2203397	97.022	98.346
2	22.716	1627748	37050	2.978	1.654
Total		54653947	2240447	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-134-III
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 17 2012
Agilent Technologies
NO2
O
N
N
Cl
2l
9
8
7
6
5
4
3
2
1
0
ppm
1.81
1.89
1.83
1.95
0.99
1.00
0.97
1.00
3.82


Carbon-13
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-134-IIIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 17 2012
Agilent Technologies
160
140
120
100
80
60
40
20
ppm

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

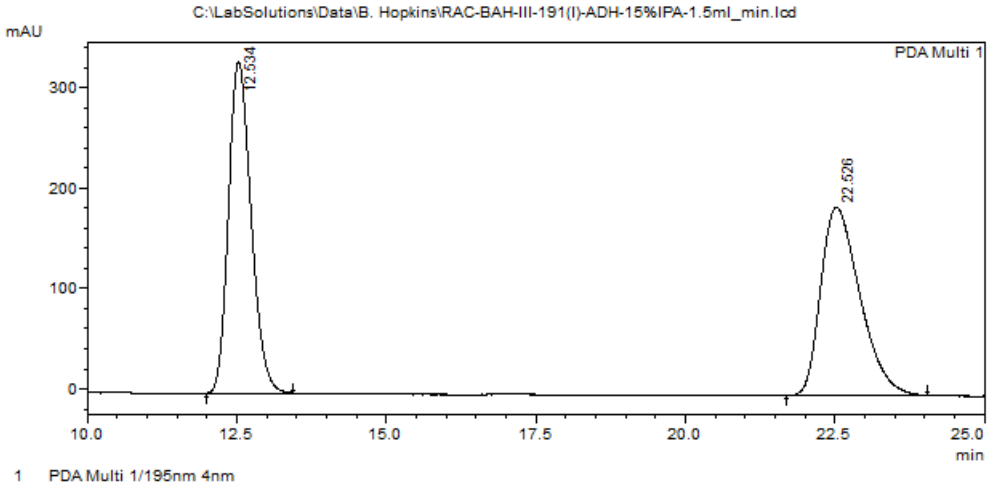


2l

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-191(I)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-191(I)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-191(I)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/15/2012 12:04:41 PM
Data Processed	: 6/15/2012 12:31:56 PM

<Chromatogram>



1 PDA Multi 1/195nm 4nm

PeakTable

PDA Ch1 195nm 4nm

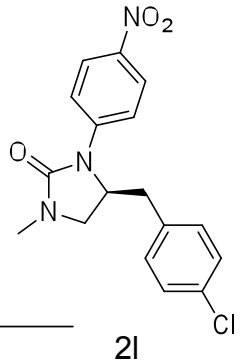
Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.534	8705719	329531	50.501	63.752
2	22.526	8532834	187365	49.499	36.248
Total		17238553	516896	100.000	100.000

7/2/2012 18:41:58 1 / 1

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

C:\LabSolutions\Data\B. Hopkins\BAH-iii-134-iii(2l).lcd

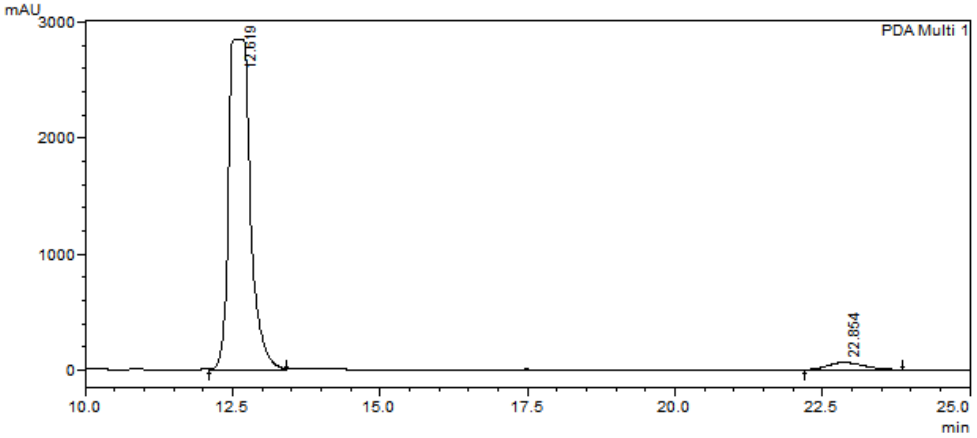
Acquired by	: Admin
Sample Name	: BAH-iii-134-iii(2l)
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: BAH-iii-134-iii(2l).lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/2/2012 5:50:23 PM
Data Processed	: 7/2/2012 6:15:34 PM



2l

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\BAH-iii-134-iii(2l).lcd

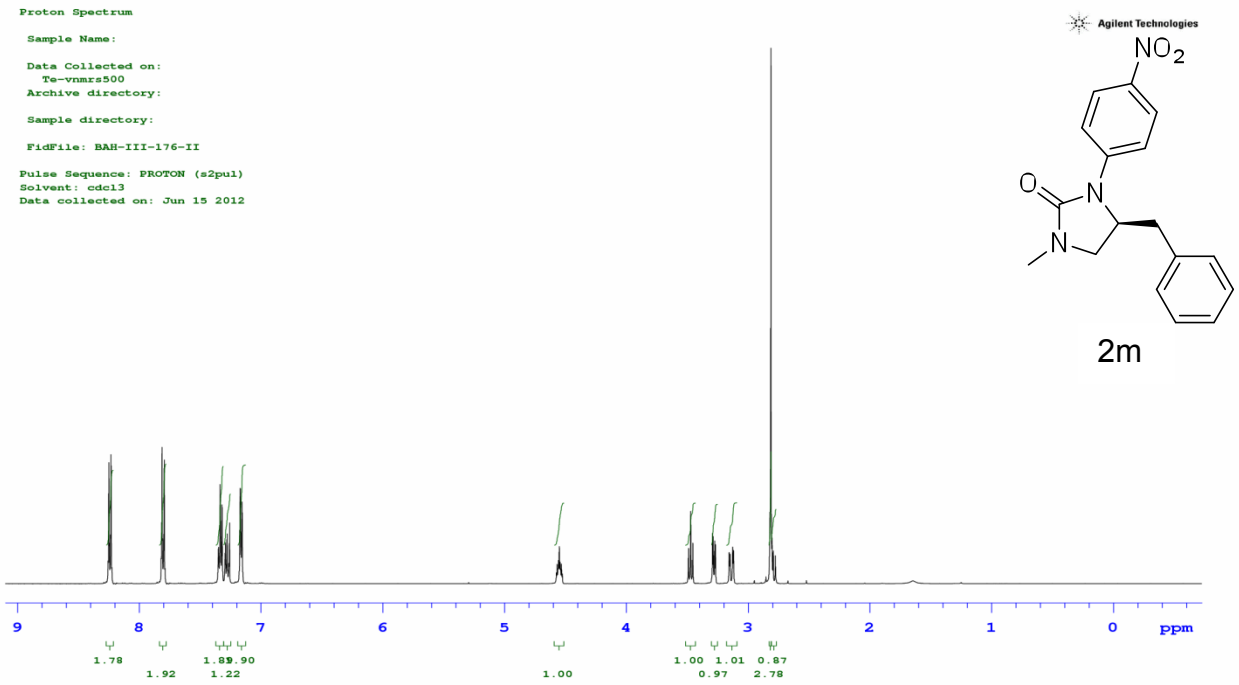


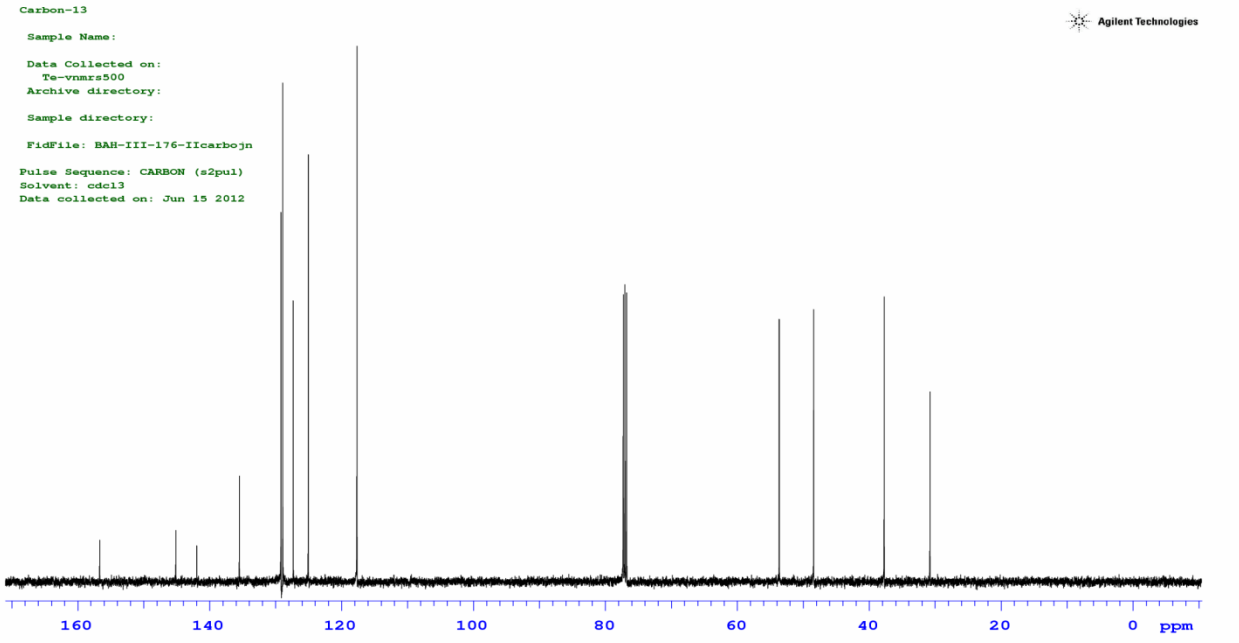
1 PDA Multi 1/195nm 4nm

PeakTable

PDA Ch1 195nm 4nm

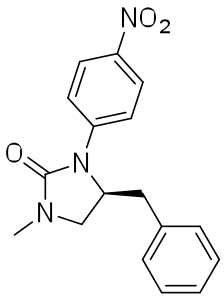
Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.619	73124560	2850512	96.311	97.808
2	22.854	2801215	63874	3.689	2.192
Total		75925775	2914386	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: BAH-III-176-II
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 15 2012
Agilent Technologies
NO2
O
N
N
2m
9
8
7
6
5
4
3
2
1
0
ppm
1.78
1.92
1.89
1.90
1.22
1.00
1.00
0.97
1.01
2.78
0.87


Carbon-13
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: BAH-III-176-IIcarbojn
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 15 2012
Agilent Technologies
160
140
120
100
80
60
40
20
0
ppm

6/18/2012 13:29:04 1 / 1

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

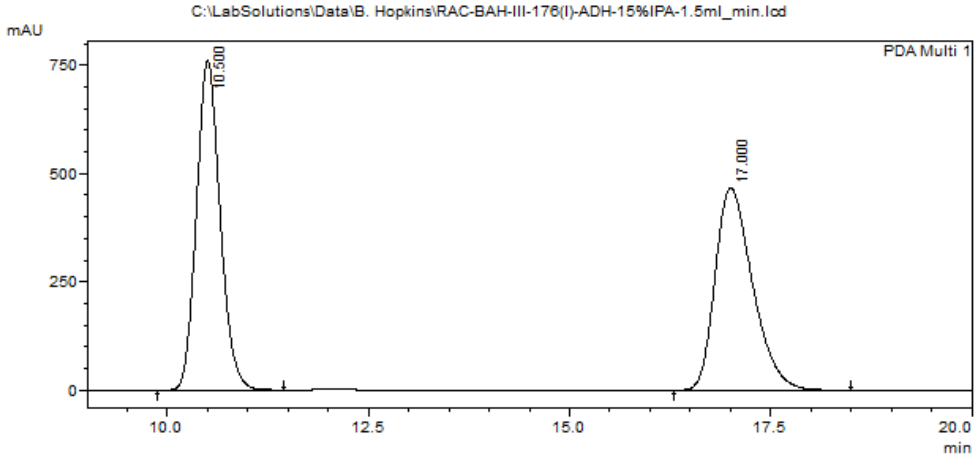


2m

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-176(I)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-176(I)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-176(I)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/6/2012 11:25:16 AM
Data Processed	: 6/6/2012 11:59:03 AM

<Chromatogram>



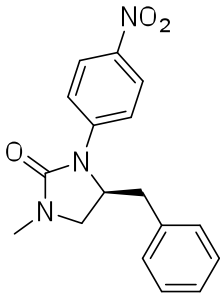
PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.500	15629577	761653	50.858	62.013
2	17.000	15102321	466566	49.142	37.987
Total		30731898	1228219	100.000	100.000

6/18/2012 13:31:29 1 / 1

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

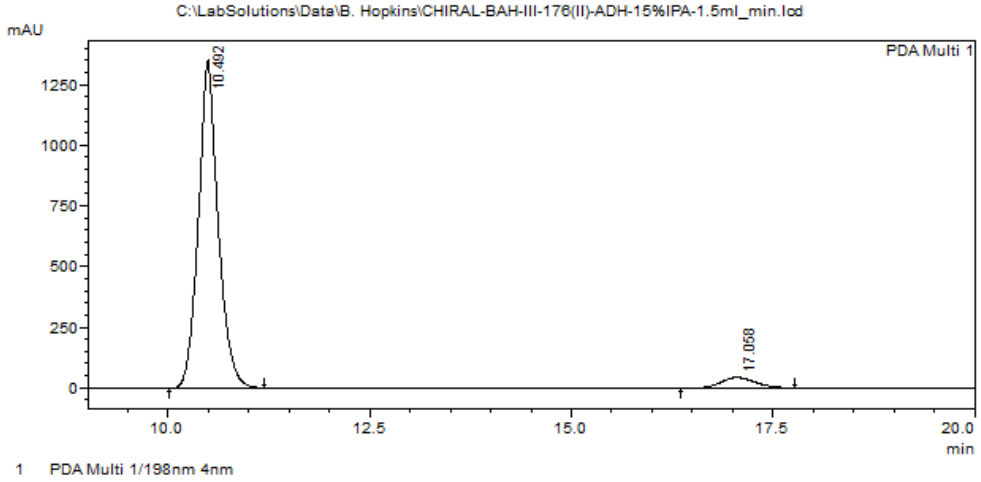


2m

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-176(II)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-176(II)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-176(II)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/6/2012 1:05:47 PM
Data Processed	: 6/6/2012 1:26:02 PM

<Chromatogram>

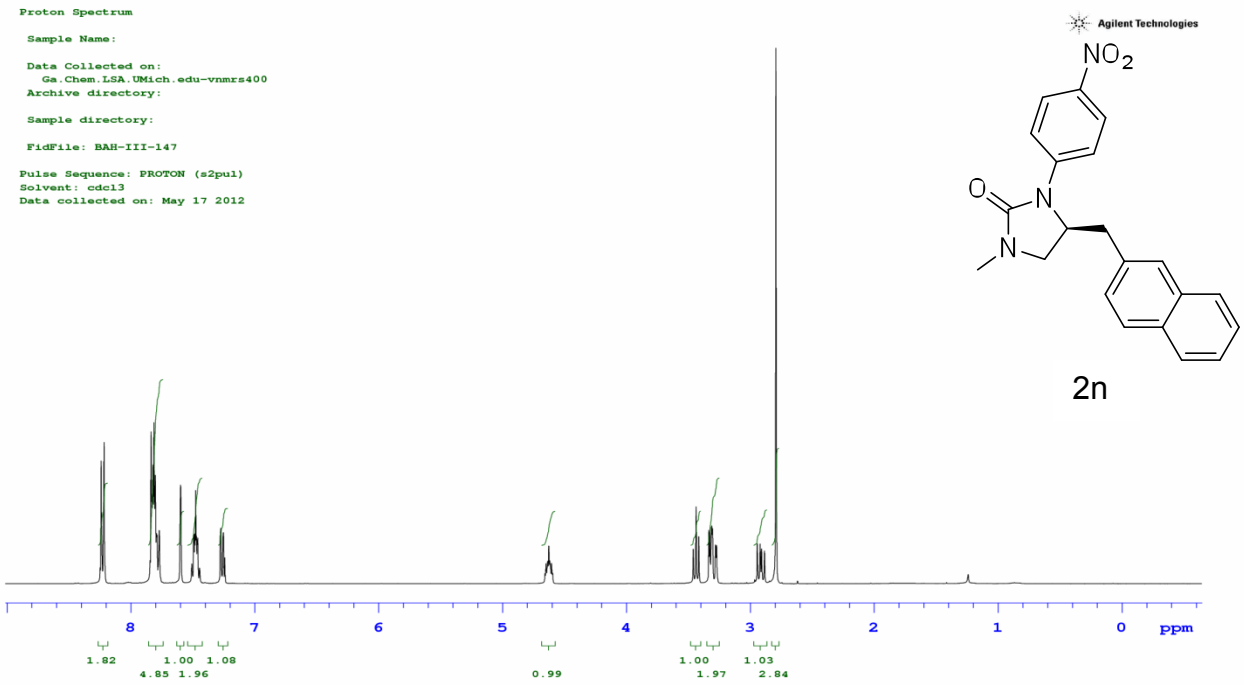


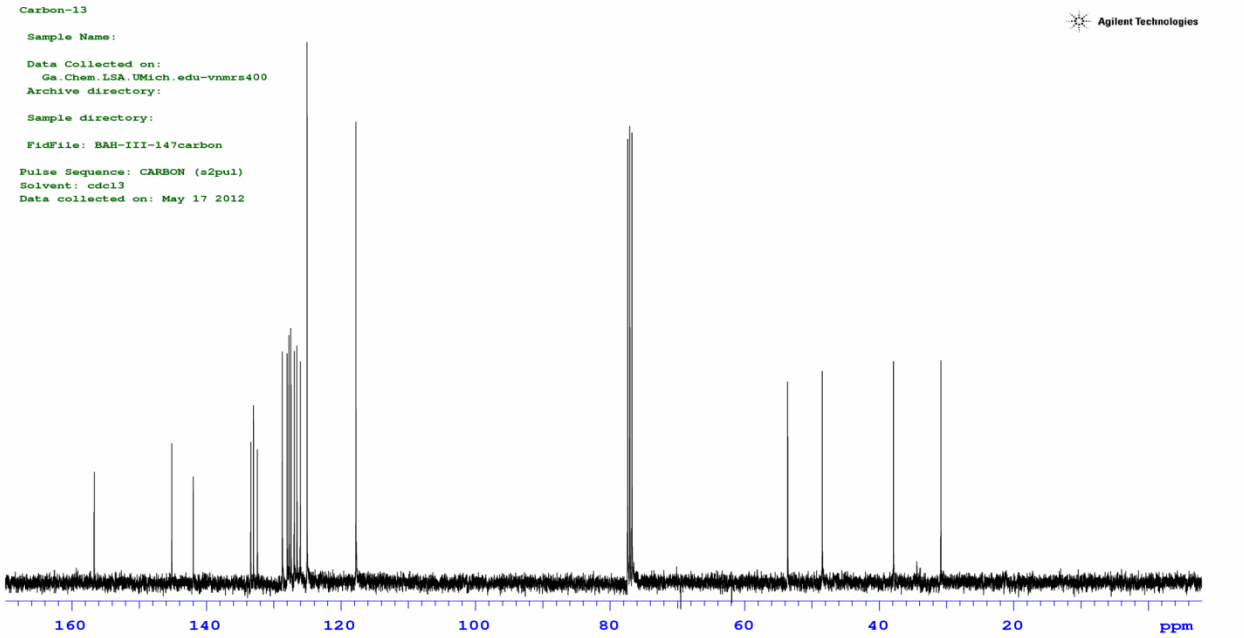
1 PDA Multi 1/198nm 4nm

PeakTable

PDA Ch1 198nm 4nm

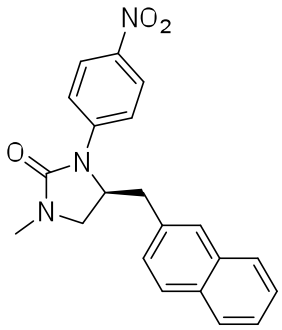
Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.492	23025310	1352429	94.456	96.859
2	17.058	1351374	43853	5.544	3.141
Total		24376684	1396282	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-147
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 17 2012
Agilent Technologies
NO2
O
N
N
2n
8
7
6
5
4
3
2
1
0
ppm
1.82
4.85
1.96
1.00
1.08
0.99
1.00
1.97
1.03
2.84


Carbon-13
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-147carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 17 2012
Agilent Technologies
160
140
120
100
80
60
40
20
ppm

6/28/2012 10:45:05 1 / 1

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



2n

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-191-2-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-191-2-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-191-2-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/18/2012 3:36:16 PM
Data Processed	: 6/18/2012 4:32:15 PM

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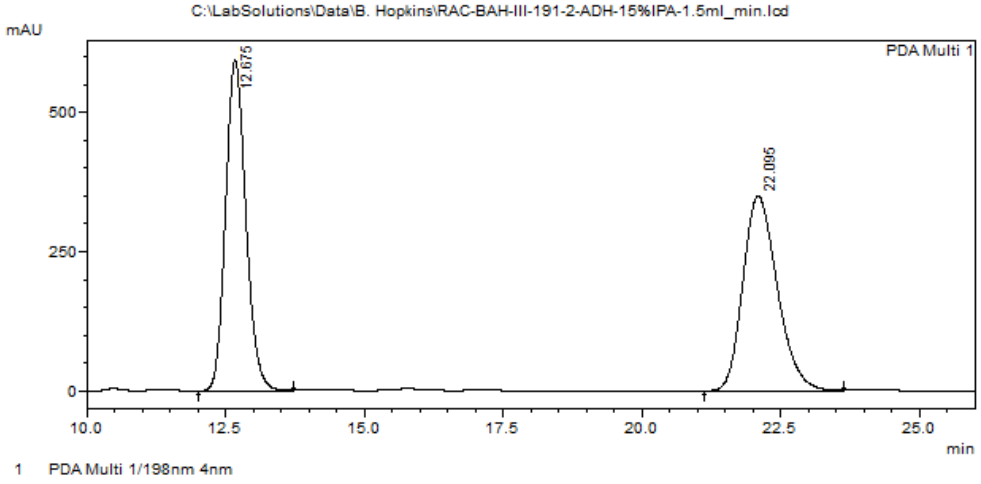


1 PDA Multi 1/198nm 4nm

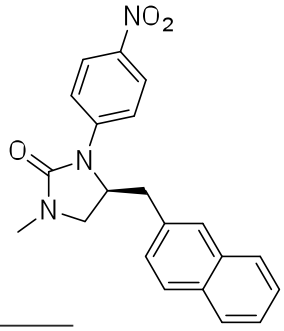
PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.675	15142562	594519	50.069	62.929
2	22.095	15100795	350226	49.931	37.071
Total		30243357	944745	100.000	100.000

7/2/2012 19:30:13 1 / 1

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

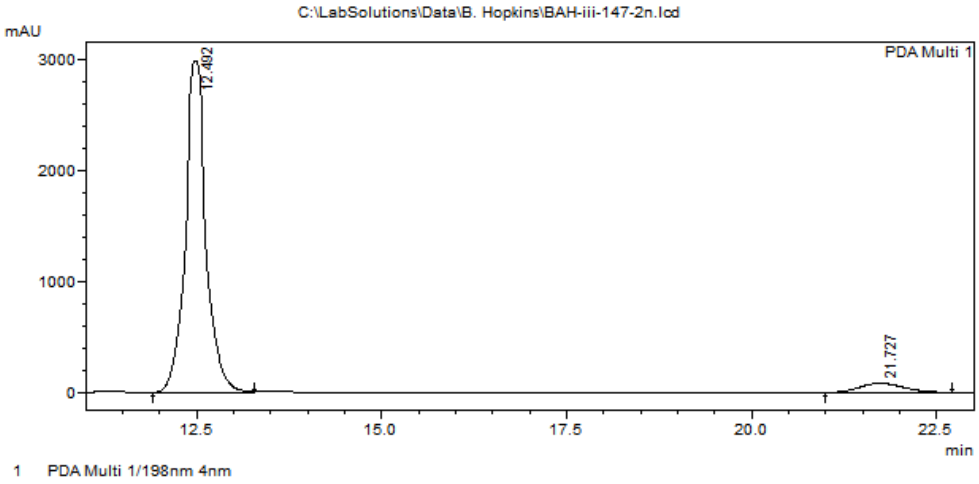


2n

C:\LabSolutions\Data\B. Hopkins\BAH-iii-147-2n.lcd

Acquired by	: Admin
Sample Name	: BAH-iii-147-2n
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: BAH-iii-147-2n.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/2/2012 7:01:39 PM
Data Processed	: 7/2/2012 7:25:37 PM

<Chromatogram>

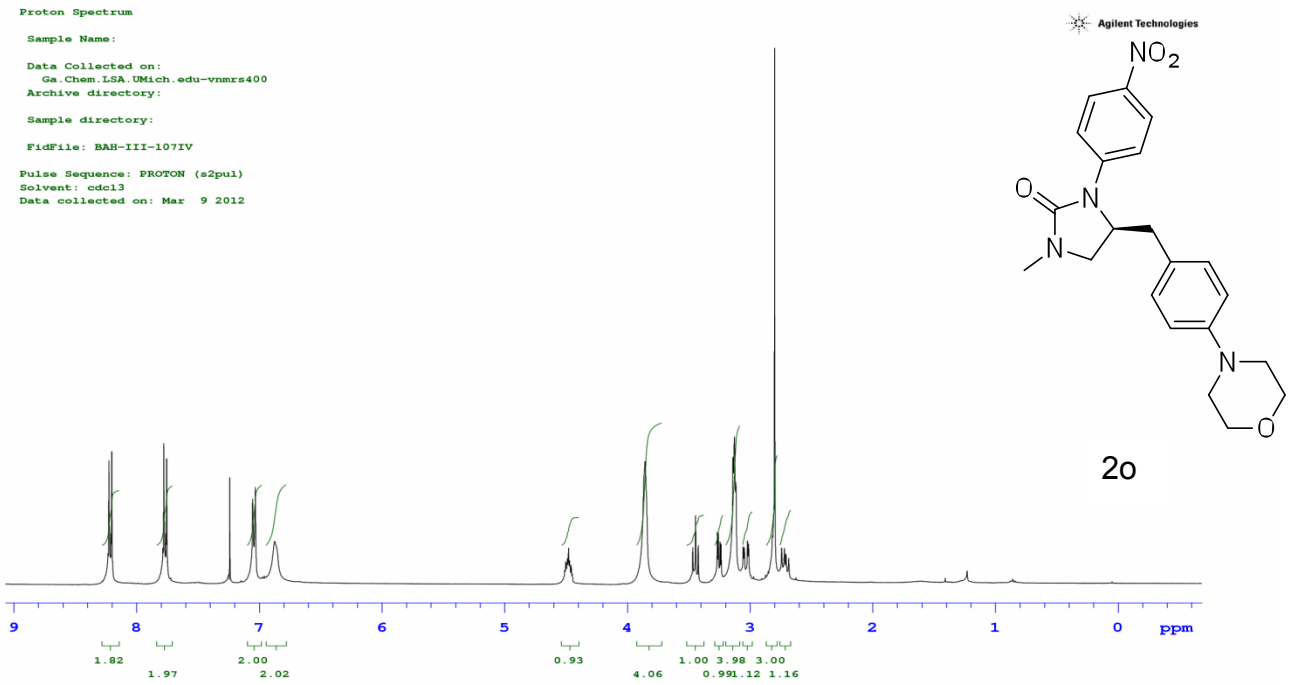


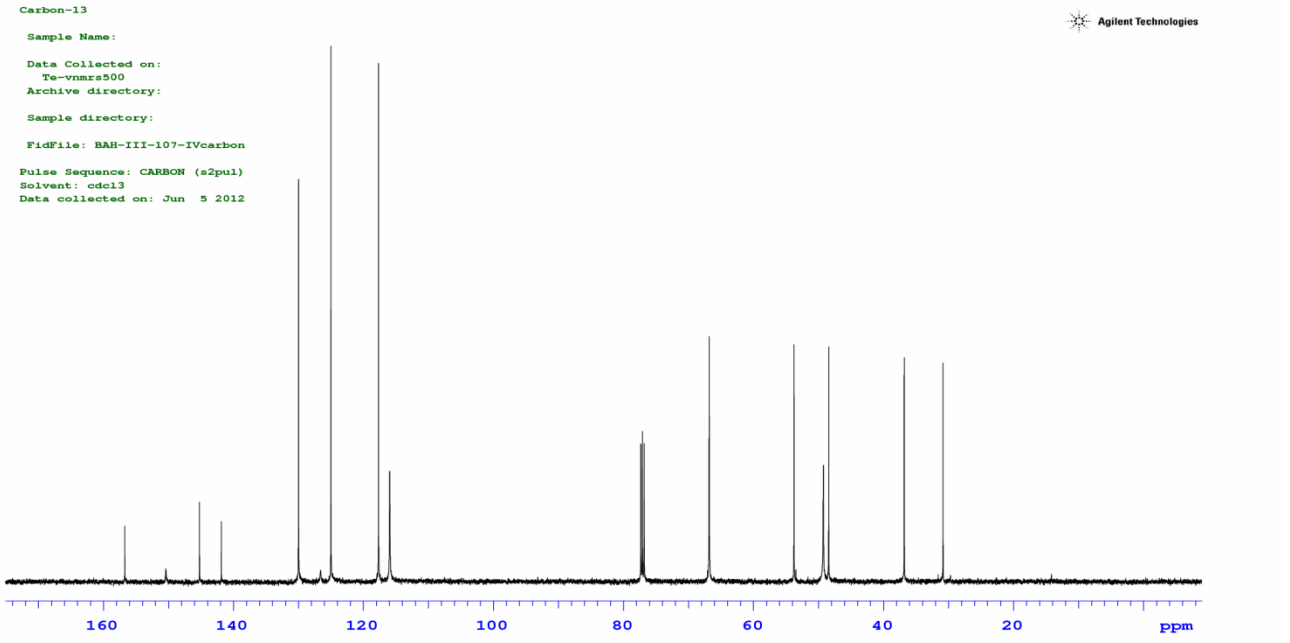
1 PDA Multi 1/198nm 4nm

PeakTable

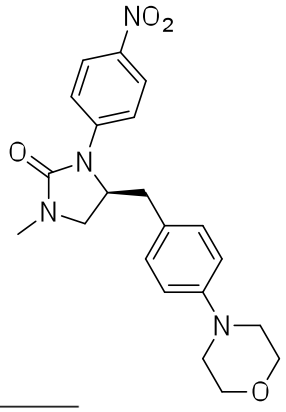
PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.492	58028359	2986559	94.475	97.297
2	21.727	3393857	82984	5.525	2.703
Total		61422216	3069542	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-107IV
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Mar 9 2012
Agilent Technologies
NO2
O
N
N
N
O
2o
9
8
7
6
5
4
3
2
1
0
ppm
1.82
1.97
2.00
2.02
0.93
4.06
1.00
3.98
0.99
1.12
3.00
1.16


Carbon-13
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: BAH-III-107-IVcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 5 2012
Agilent Technologies
160
140
120
100
80
60
40
20
ppm

6/18/2012 14:09:41 1 / 1



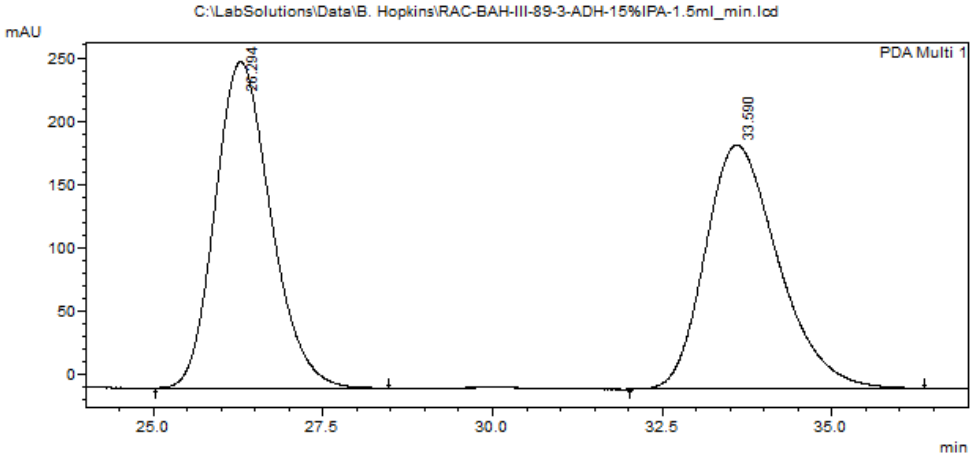
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-89-3-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-89-3-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-89-3-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/18/2012 1:26:13 PM
Data Processed	: 6/18/2012 2:06:51 PM

<Chromatogram>

2o



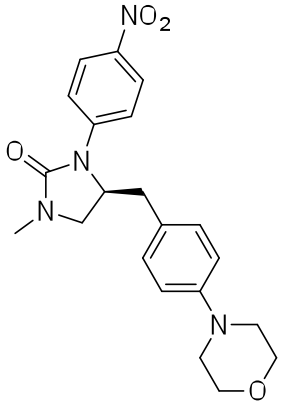
1 PDA Multi 1/195nm 4nm

PeakTable

PDA Ch1 195nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.294	14715240	257577	50.432	57.239
2	33.590	14463291	192428	49.568	42.761
Total		29178532	450005	100.000	100.000

6/15/2012 17:37:12 1 / 1



2o

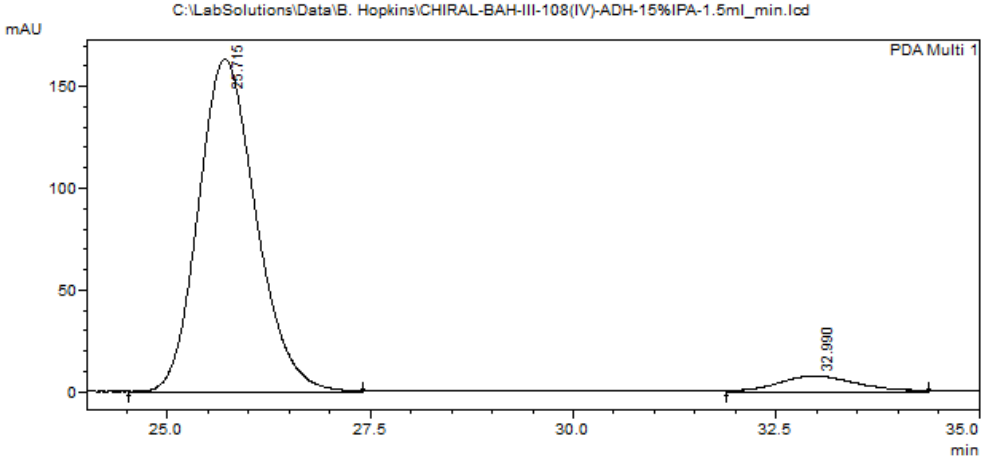
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-108(IV)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-108(IV)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-108(IV)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/2/2012 2:24:04 PM
Data Processed	: 3/2/2012 2:59:29 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-108(IV)-ADH-15%IPA-1.5ml_min.lcd

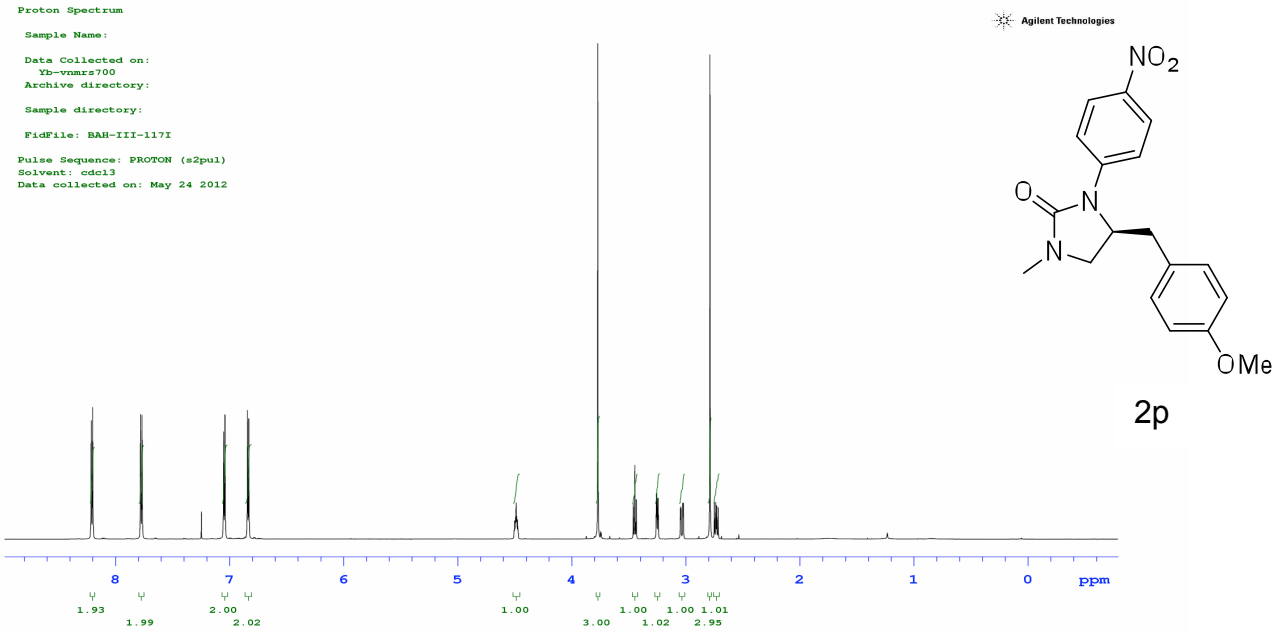


1 PDA Multi 1/195nm 4nm

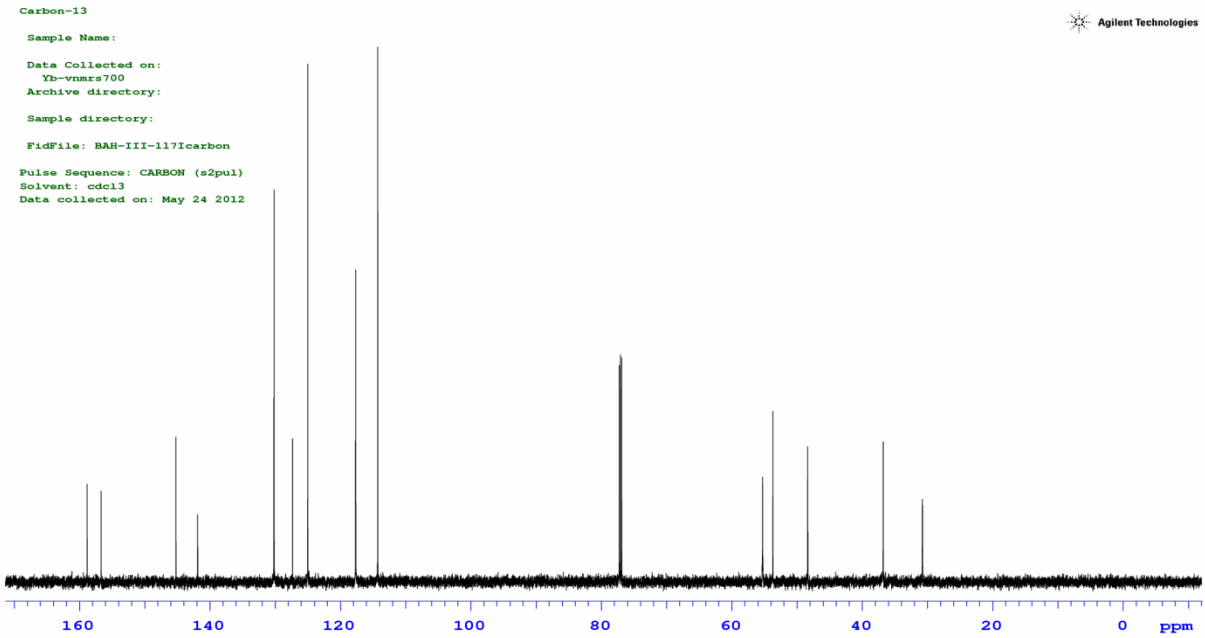
PeakTable

PDA Ch1 195nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.715	8058314	163227	93.720	95.363
2	32.990	540006	7937	6.280	4.637
Total		8598320	171164	100.000	100.000

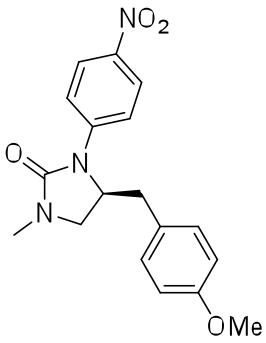

Proton Spectrum
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-III-117I
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 24 2012
Agilent Technologies
NO2
O
N
N
OMe
8
7
6
5
4
3
2
1
0
ppm
1.93
1.99
2.00
2.02
1.00
3.00
1.00
1.02
1.00
2.95
1.01

2p


Carbon-13
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-III-117Icarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 24 2012
Agilent Technologies
160
140
120
100
80
60
40
20
0
ppm

6/12/2012 18:59:17 1 / 1

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

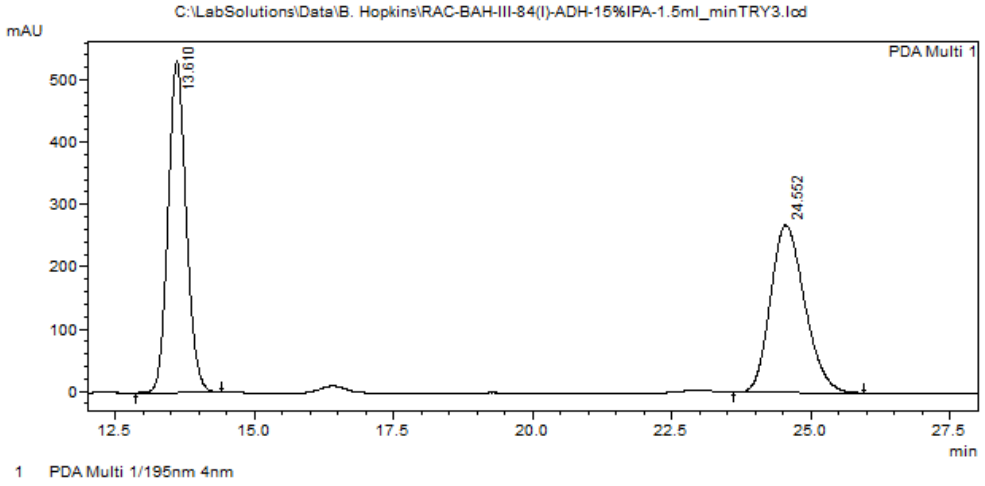


2p

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-84(I)-ADH-15%IPA-1.5ml_minTRY3.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-84(I)-ADH-15%IPA-1.5ml_minTRY3
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-84(I)-ADH-15%IPA-1.5ml_minTRY3.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/6/2012 5:55:14 PM
Data Processed	: 2/6/2012 6:30:02 PM

<Chromatogram>



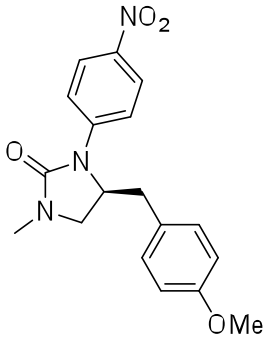
1 PDA Multi 1/195nm 4nm

PeakTable

PDA Ch1 195nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.610	12066213	532319	51.112	66.425
2	24.552	11541063	269068	48.888	33.575
Total		23607276	801386	100.000	100.000

6/12/2012 18:53:49 1 / 1



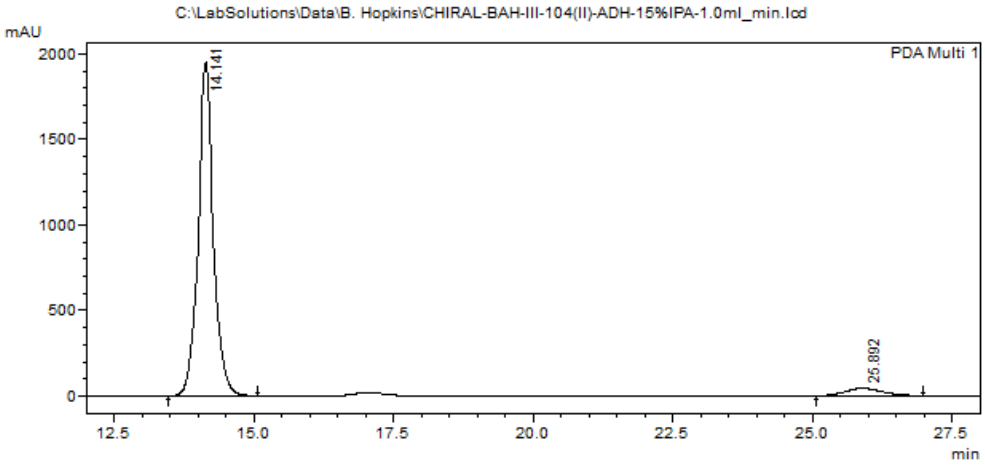
2p

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

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-104(II)-ADH-15%IPA-1.0ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-104(II)-ADH-15%IPA-1.0ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-104(II)-ADH-15%IPA-1.0ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/24/2012 4:03:50 PM
Data Processed	: 2/24/2012 4:38:03 PM

<Chromatogram>

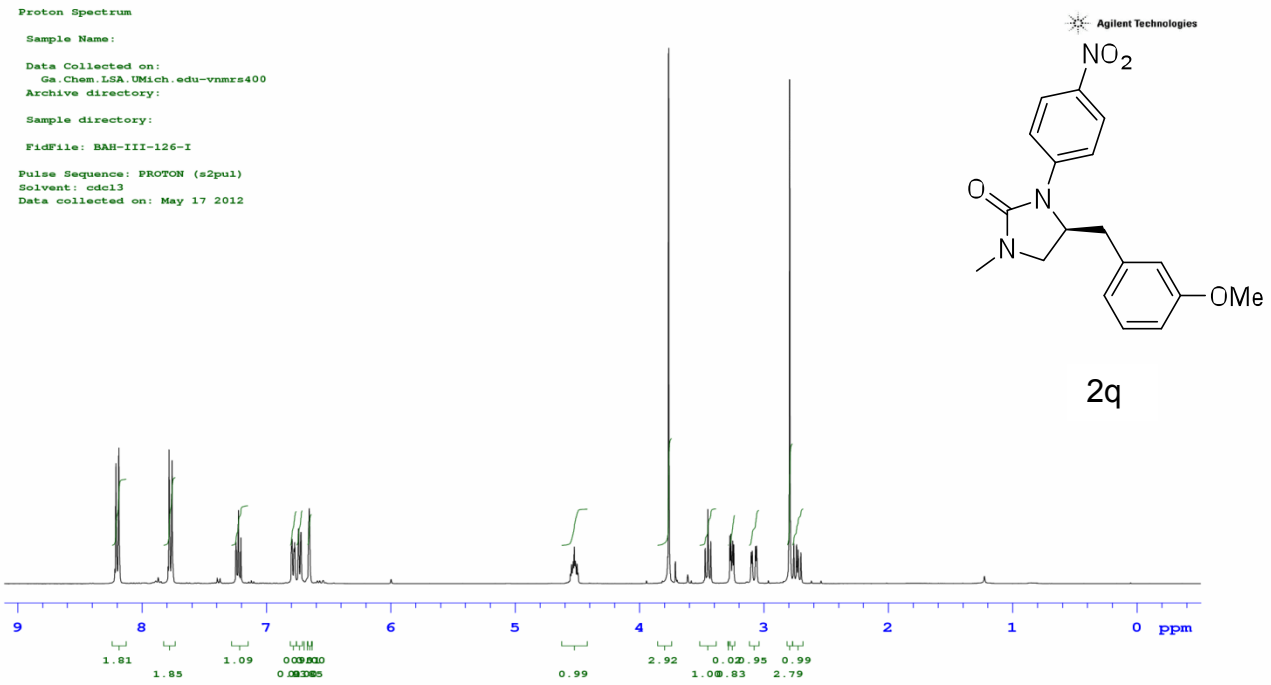


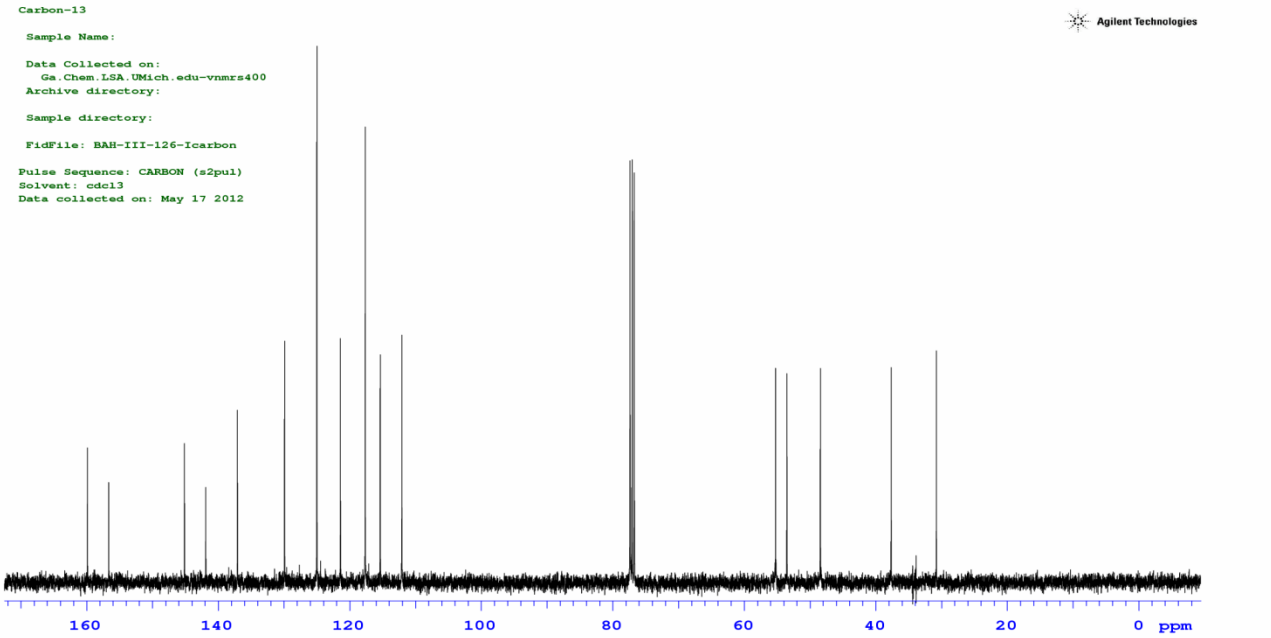
1 PDA Multi 1/195nm 4nm

PeakTable

PDA Ch1 195nm 4nm

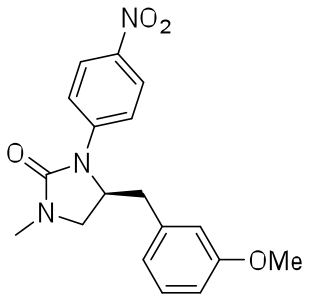
Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.141	36769486	1952058	94.785	97.818
2	25.892	2023005	43552	5.215	2.182
Total		38792491	1995609	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-126-I
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 17 2012
Agilent Technologies
NO2
O
N
N
OMe
2q
9
8
7
6
5
4
3
2
1
0
ppm
1.81
1.85
1.09
0.99
2.92
1.00
0.83
2.79
0.99


Carbon-13
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-126-Icarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 17 2012
Agilent Technologies
160
140
120
100
80
60
40
20
0
ppm

6/12/2012 19:06:14 1 / 1

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



2q

C:\LabSolutions\Data\B. Hopkins\rac-BAH-III-93(IV)-ADH-15%IPA-1.5ml_min1.lcd

Acquired by	: Admin
Sample Name	: rac-BAH-III-93(IV)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: rac-BAH-III-93(IV)-ADH-15%IPA-1.5ml_min1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/16/2012 6:56:16 PM
Data Processed	: 2/16/2012 8:06:18 PM

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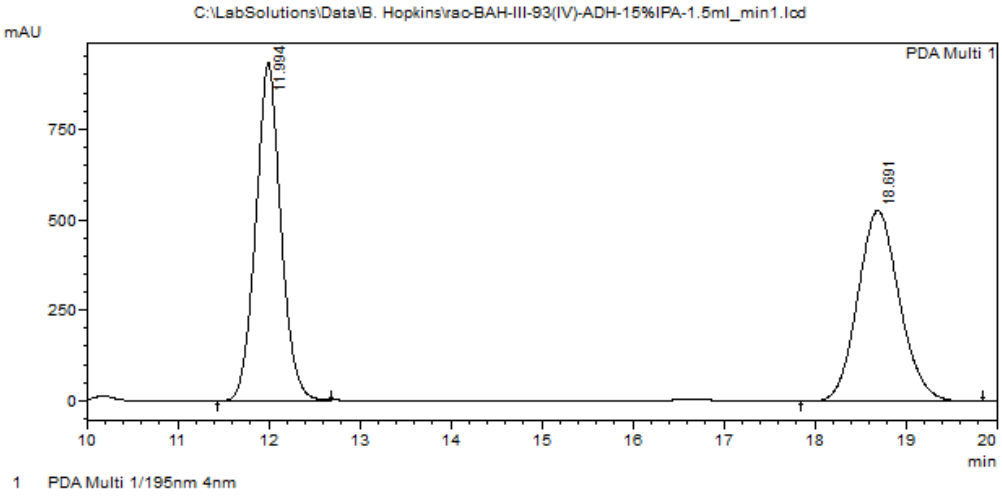


1 PDA Multi 1/195nm 4nm

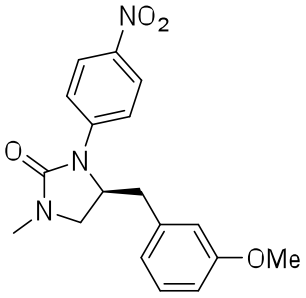
PeakTable

PDA Ch1 195nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.994	17954287	933121	51.641	63.876
2	18.691	16813544	527718	48.359	36.124
Total		34767830	1460839	100.000	100.000

6/12/2012 19:12:36 1 / 1

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



2q

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-126(II-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-126(II)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-126(II-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/23/2012 3:25:19 PM
Data Processed	: 3/23/2012 3:52:33 PM

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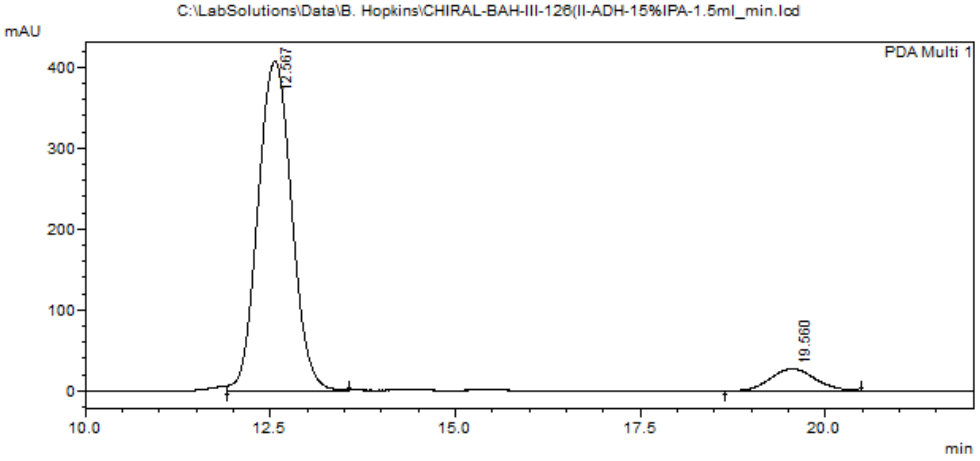
C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-126(II-ADH-15%IPA-1.5ml_min.lcd

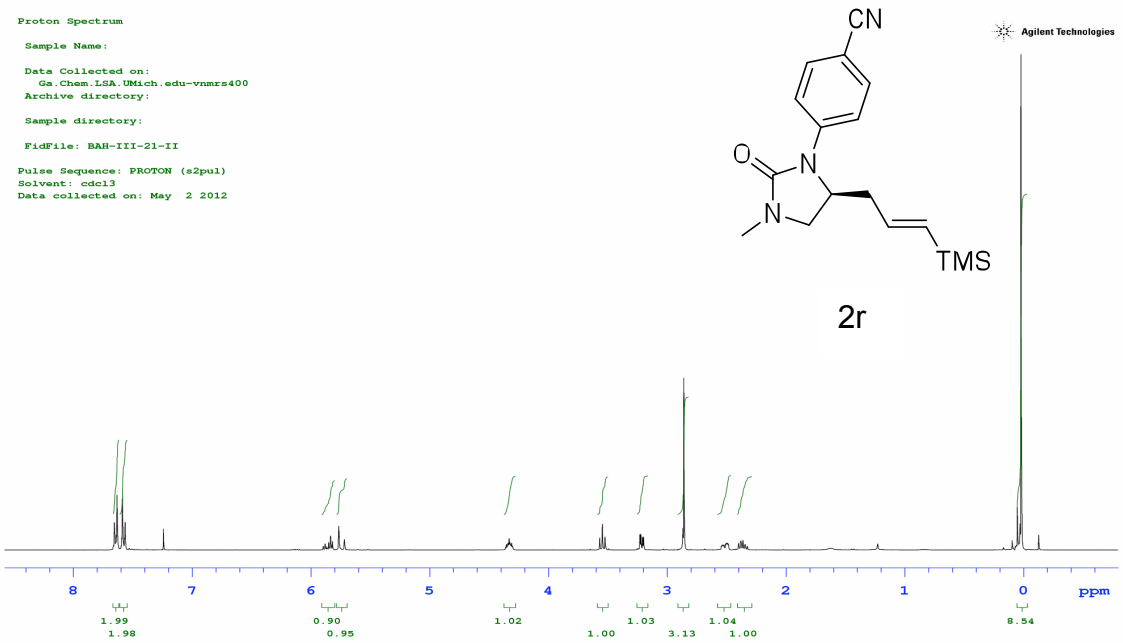


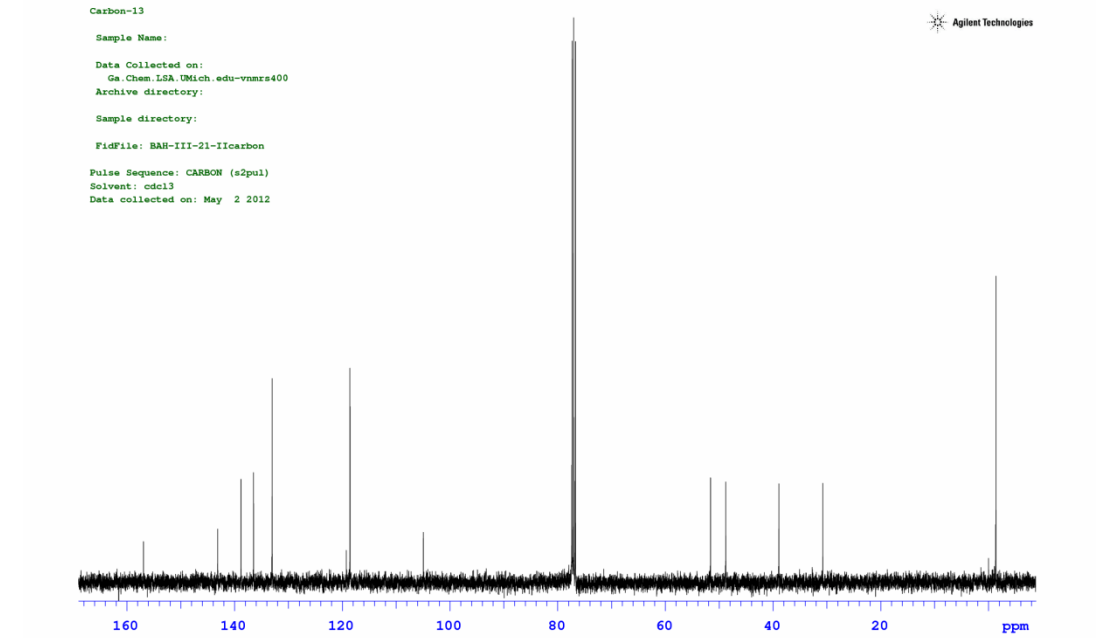
1 PDA Multi 1/195nm 4nm

PeakTable

PDA Ch1 195nm 4nm

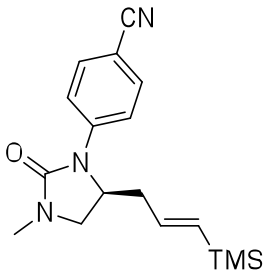
Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.567	12963493	407430	91.720	93.694
2	19.560	1170337	27423	8.280	6.306
Total		14133829	434854	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-21-II
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 2 2012
Agilent Technologies
CN
O
N
N
TMS
2r
8
7
6
5
4
3
2
1
0
ppm
1.99
1.98
0.90
0.95
1.02
1.00
1.03
3.13
1.04
1.00
8.54


Carbon-13
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-21-IIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 2 2012
Agilent Technologies
160
140
120
100
80
60
40
20
ppm

6/12/2012 19:19:43 1 / 1

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



2r

C:\LabSolutions\Data\B. Hopkins\rac-BAH-III-21(I)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: rac-BAH-III-21(I)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: rac-BAH-III-21(I)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/10/2012 3:54:23 PM
Data Processed	: 6/10/2012 4:02:42 PM

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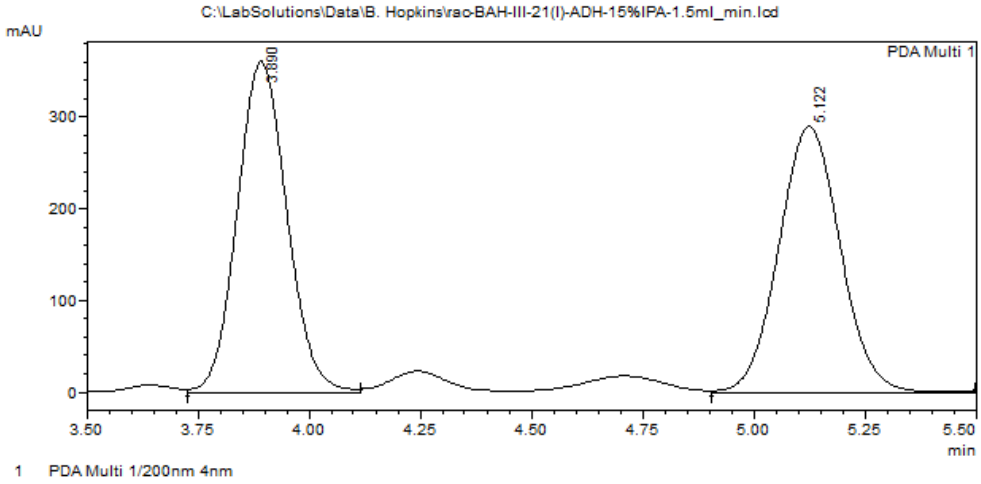


1 PDA Multi 1/200nm 4nm

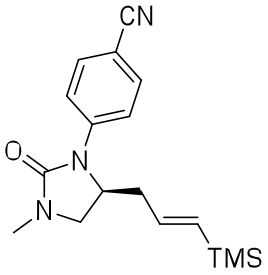
PeakTable

PDA Ch1 200nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	3.890	2795759	361237	50.067	55.453
2	5.122	2788303	290187	49.933	44.547
Total		5584062	651424	100.000	100.000

6/12/2012 19:22:43 1 / 1

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

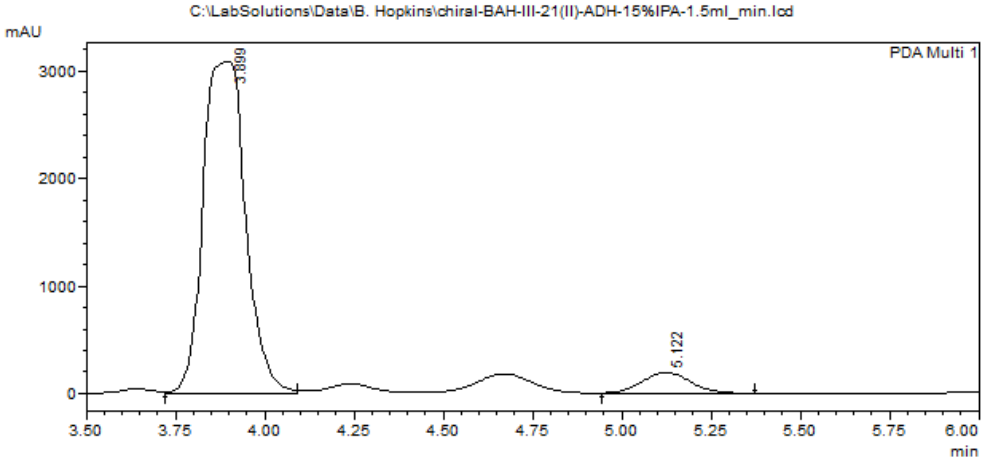


2r

C:\LabSolutions\Data\B. Hopkins\chiral-BAH-III-21(II)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: chiral-BAH-III-21(II)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: chiral-BAH-III-21(II)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/10/2012 4:04:38 PM
Data Processed	: 6/10/2012 4:18:18 PM

<Chromatogram>

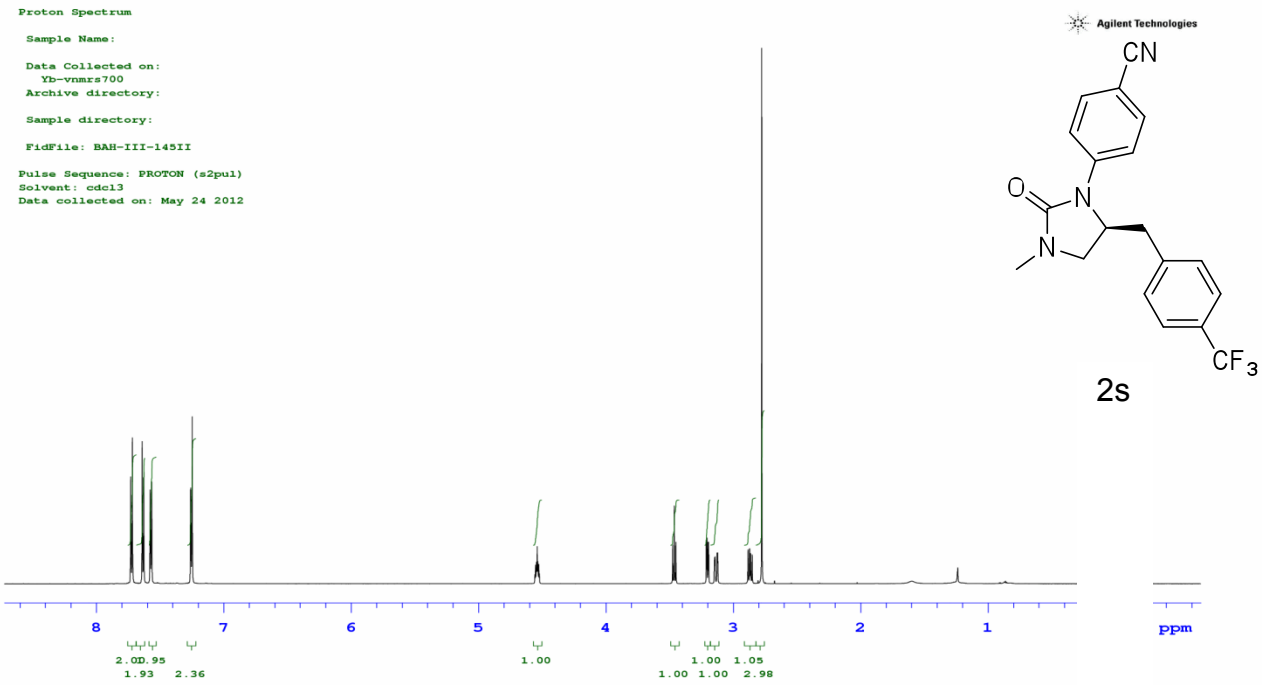


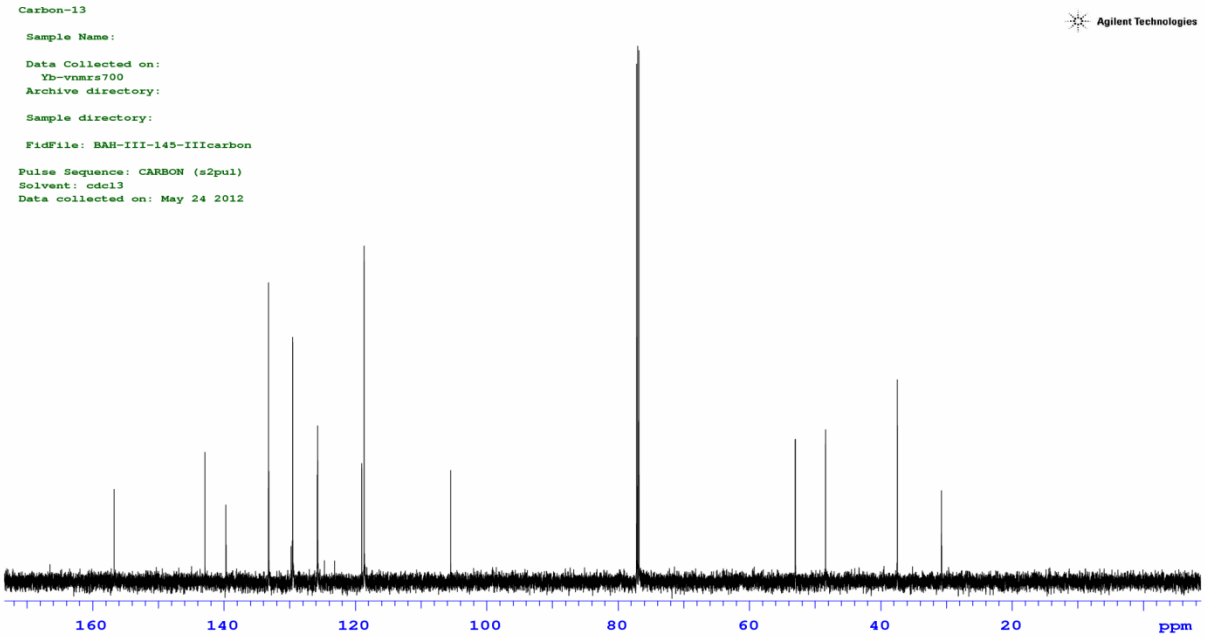
1 PDA Multi 1/200nm 4nm

PeakTable

PDA Ch1 200nm 4nm

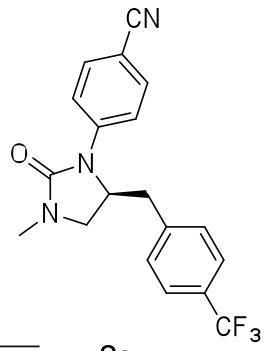
Peak#	Ret. Time	Area	Height	Area %	Height %
1	3.899	25522977	3087157	93.036	93.989
2	5.122	1910444	197454	6.964	6.011
Total		27433421	3284611	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-III-145II
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 24 2012
Agilent Technologies
CN
O
N
N
CF3
2s
8
7
6
5
4
3
2
1
ppm
2.00 0.95
1.93
2.36
1.00
1.00
1.00
1.05
2.98


Carbon-13
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-III-145-IIIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 24 2012
Agilent Technologies
160
140
120
100
80
60
40
20
ppm

6/12/2012 19:27:04 1 / 1

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

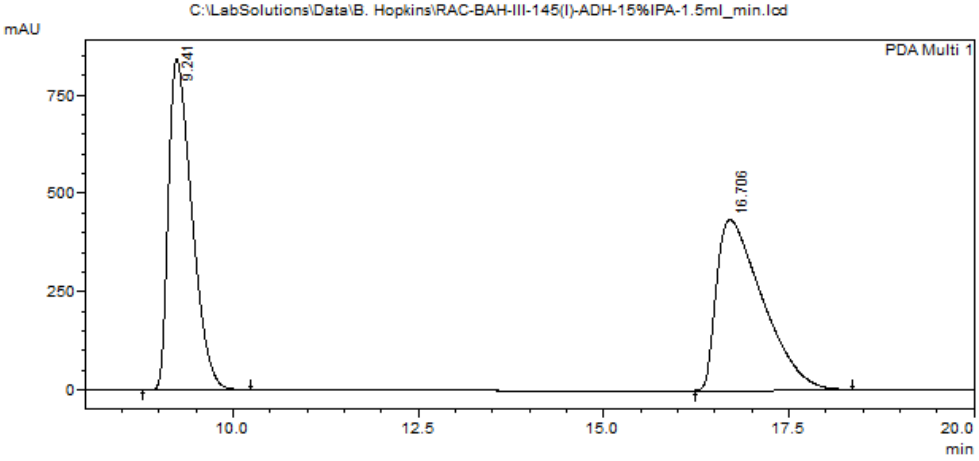


2s

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-145(I)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-145(I)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-145(I)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/4/2012 1:35:10 PM
Data Processed	: 5/4/2012 2:02:04 PM

<Chromatogram>



1 PDA Multi 1/200nm 4nm

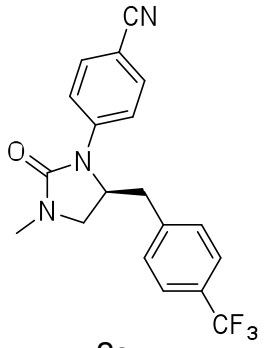
PeakTable

PDA Ch1 200nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.241	18721309	842315	49.835	65.899
2	16.706	18845151	435870	50.165	34.101
Total		37566460	1278185	100.000	100.000

6/12/2012 19:44:26 1 / 1

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



2s

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-145(III)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-145(III)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-145(III)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/4/2012 2:33:09 PM
Data Processed	: 5/4/2012 2:52:53 PM

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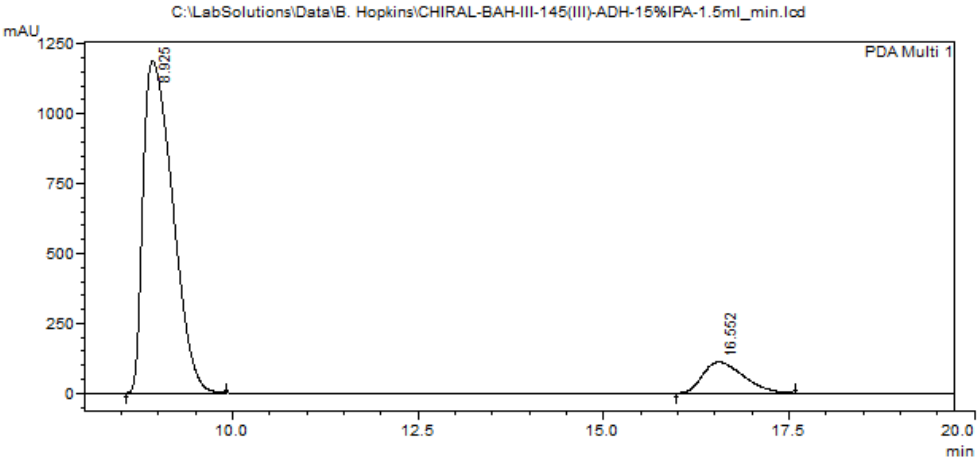


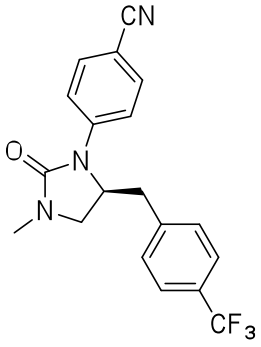
1 P

PeakTable

PDA Ch1 200nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.925	33083004	1188356	88.368	91.425
2	16.552	4354750	111457	11.632	8.575
Total		37437754	1299812	100.000	100.000

6/12/2012 19:47:17 1 / 1



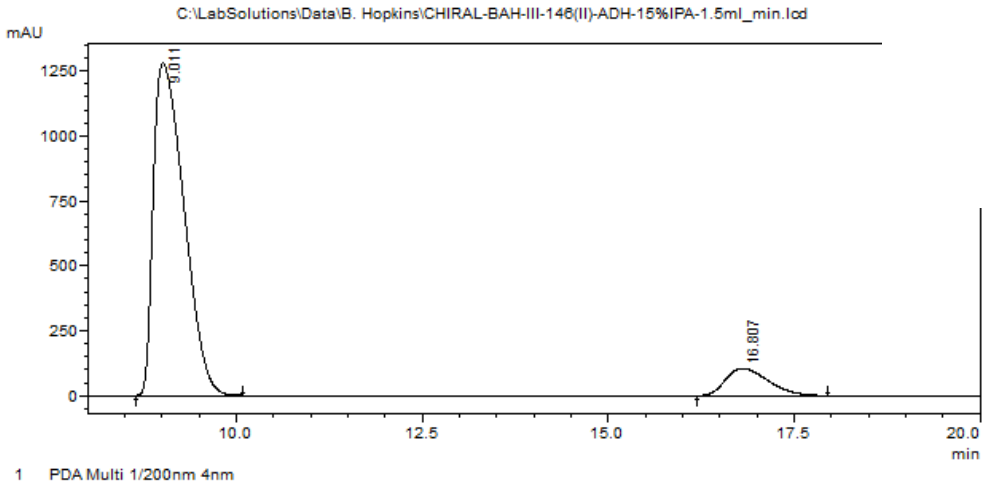
2s (4-iodobenzotrifluoride)

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

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-146(II)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-146(II)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-146(II)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/6/2012 12:05:35 PM
Data Processed	: 5/6/2012 12:27:26 PM

<Chromatogram>

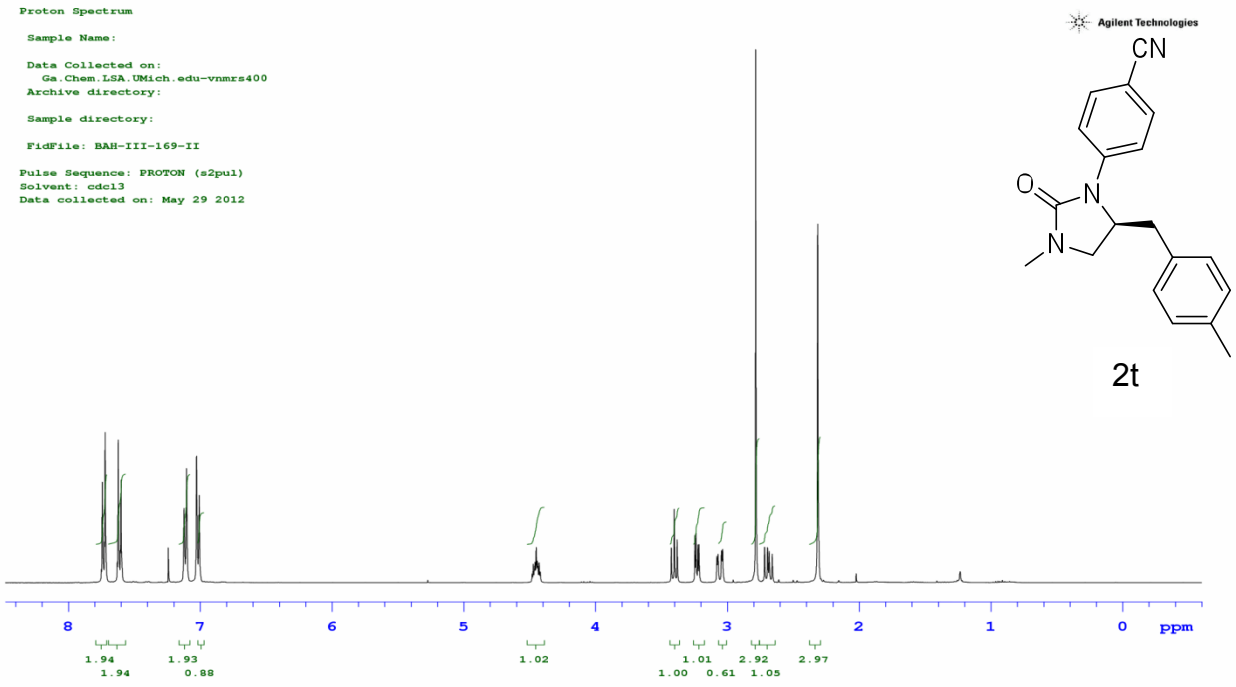


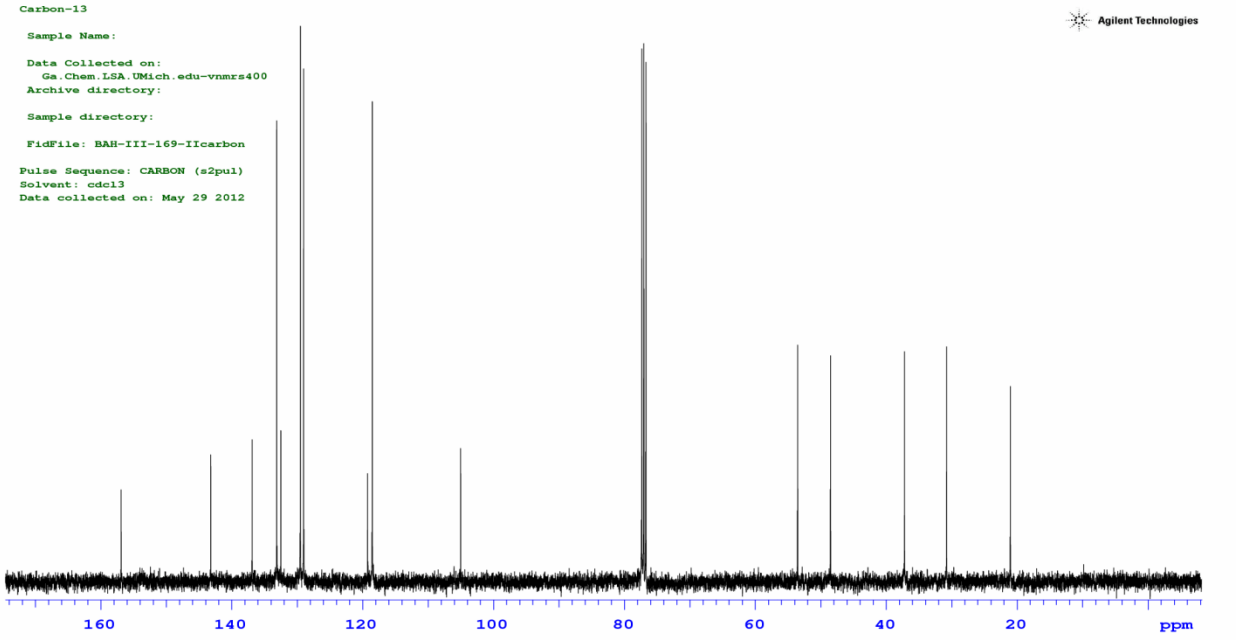
1 PDA Multi 1/200nm 4nm

PeakTable

PDA Ch1 200nm 4nm

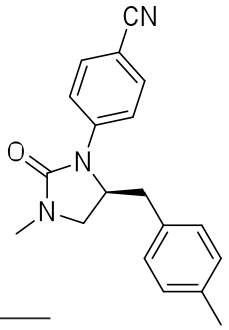
Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.011	36905138	1281521	89.880	92.421
2	16.807	4155151	105094	10.120	7.579
Total		41060288	1386615	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-169-II
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 29 2012
Agilent Technologies
CN
O
N
N
2t
8
7
6
5
4
3
2
1
0
ppm
1.94
1.94
1.93
0.88
1.02
1.00
0.61
1.01
1.05
2.92
2.97


Carbon-13
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-169-IIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 29 2012
Agilent Technologies
160
140
120
100
80
60
40
20
ppm

7/1/2012 17:55:37 1 / 1

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



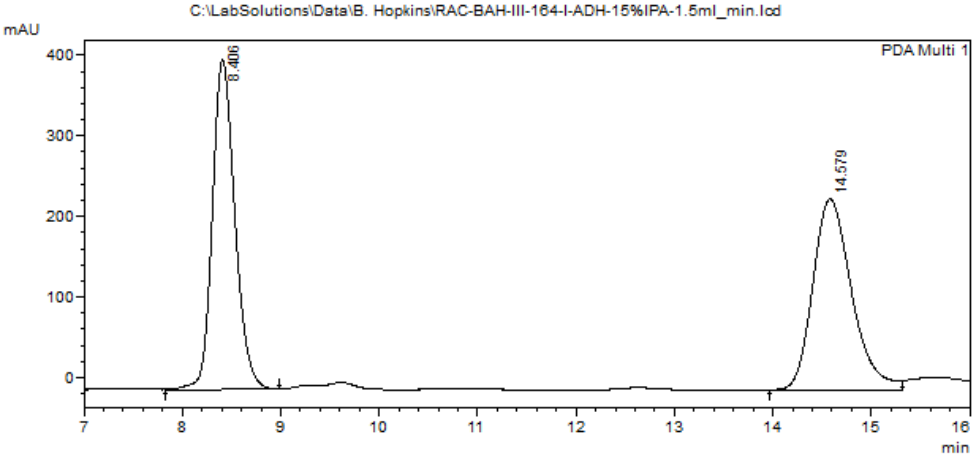
2t

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-164-I-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-164-I-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-164-I-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/19/2012 2:18:36 PM
Data Processed	: 6/19/2012 2:36:22 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-164-I-ADH-15%IPA-1.5ml_min.lcd



1 PDA Multi 1/198nm 4nm

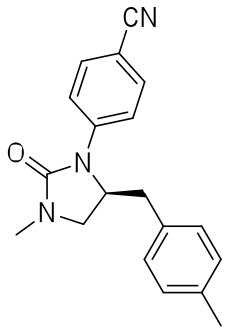
PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.406	6452556	409002	49.490	63.221
2	14.579	6585639	237940	50.510	36.779
Total		13038195	646942	100.000	100.000

7/1/2012 17:57:55 1 / 1

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



2t

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-II-164(II)-ADH-15%IPA-1.50ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-II-164(II)-ADH-15%IPA-1.50ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-II-164(II)-ADH-15%IPA-1.50ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/27/2012 5:07:24 PM
Data Processed	: 6/27/2012 5:27:36 PM

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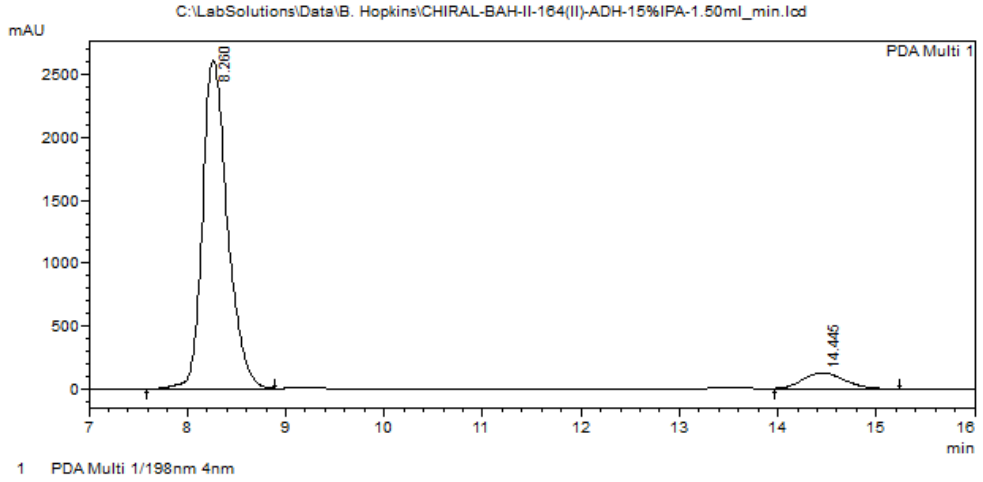


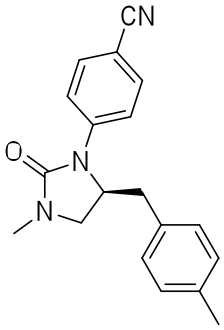
1 PDA Multi 1/198nm 4nm

PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.260	46152171	2612300	92.314	95.337
2	14.445	3842436	127773	7.686	4.663
Total		49994607	2740073	100.000	100.000

7/2/2012 18:50:24 1 / 1



2t (w/ 4-iodotoluene)

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

C:\LabSolutions\Data\B. Hopkins\BAH-II-166-I-2t.lcd

Acquired by	: Admin
Sample Name	: BAH-II-166-I-2t
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: BAH-II-166-I-2t.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/2/2012 6:29:55 PM
Data Processed	: 7/2/2012 6:47:39 PM

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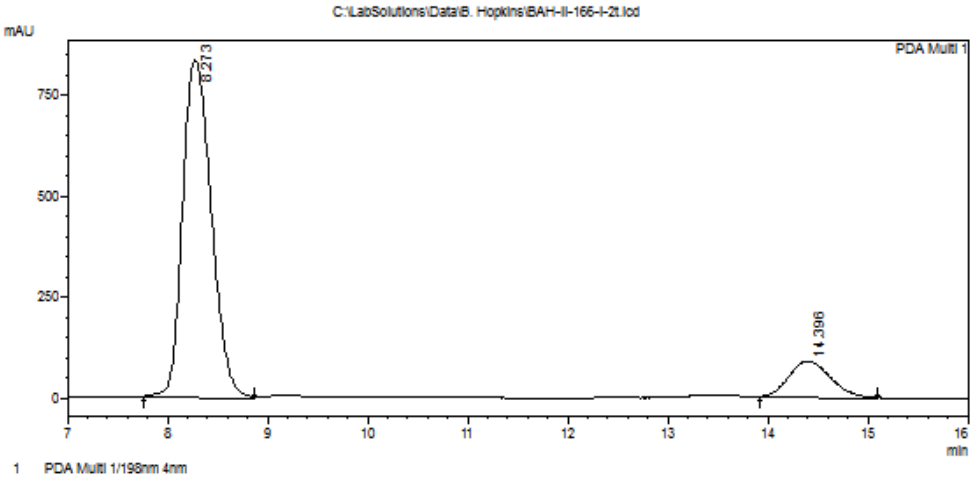
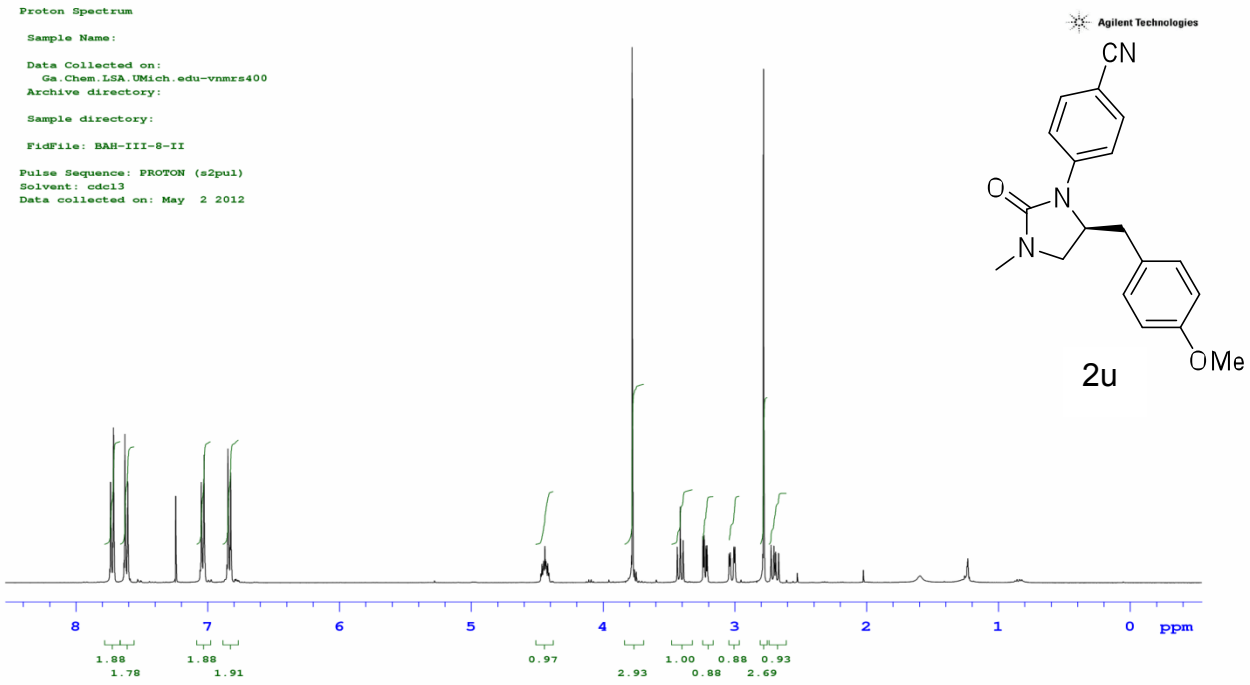
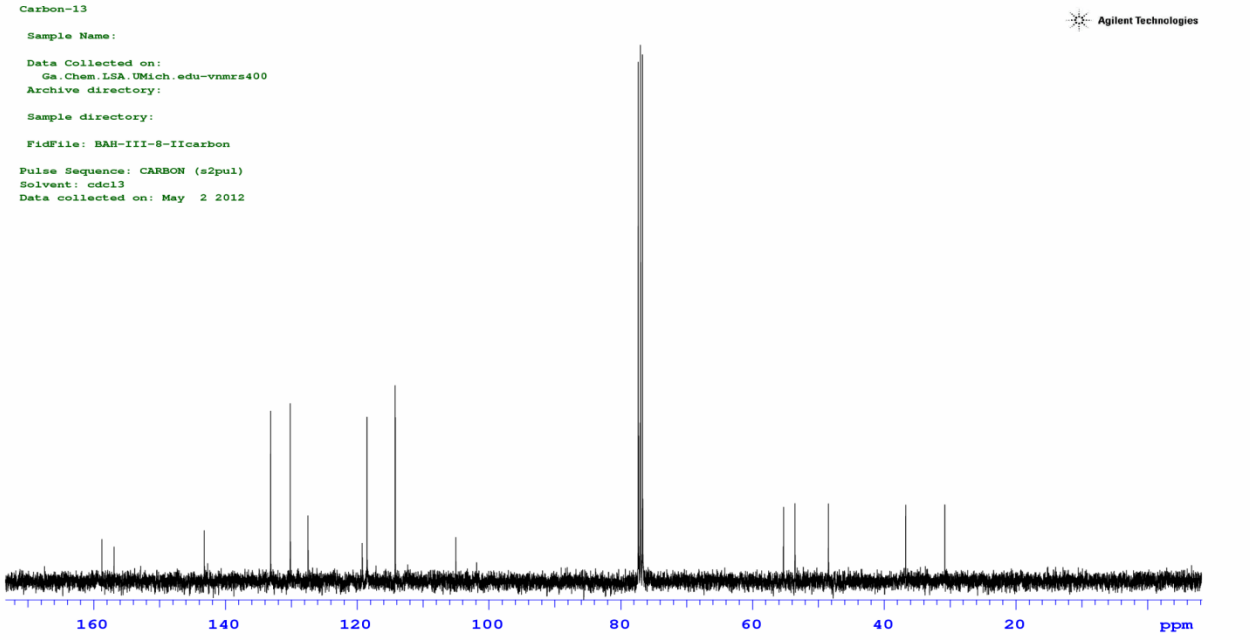


PeakTable

PDA Ch1 198nm 4nm

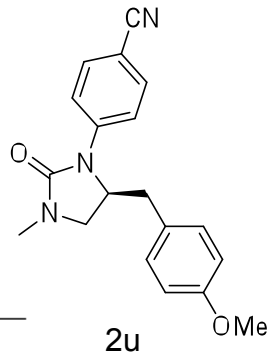
Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.273	16759962	837717	86.506	90.301
2	14.396	2614446	89977	13.494	9.699
Total		19374408	927694	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-8-II
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 2 2012
Agilent Technologies
CN
O
N
N
OMe
2u
8
7
6
5
4
3
2
1
0
ppm
1.88
1.78
1.88
1.91
0.97
2.93
1.00
0.88
0.88
2.69
0.93


Carbon-13
Sample Name:
Data Collected on:
Ga.Chem.LSA.UMich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-III-8-IIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 2 2012
Agilent Technologies
160
140
120
100
80
60
40
20
ppm

7/1/2012 18:36:32 1 / 1

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



2u

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-8(I)-ADH-15%IPA-1.50ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-8(I)-ADH-15%IPA-1.50ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-8(I)-ADH-15%IPA-1.50ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/1/2012 6:03:56 PM
Data Processed	: 7/1/2012 6:31:27 PM

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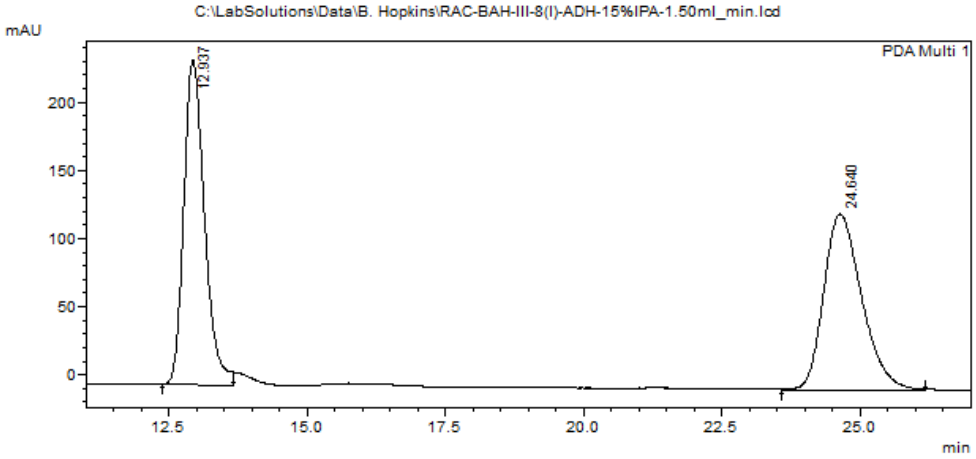


1 PDA Multi 1/198nm 4nm

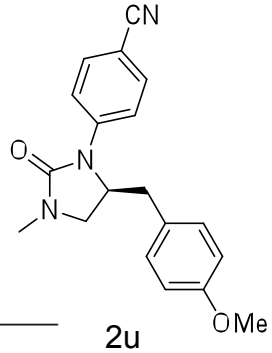
PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.937	6228978	238054	50.256	64.861
2	24.640	6165434	128968	49.744	35.139
Total		12394413	367022	100.000	100.000

7/1/2012 19:06:04 1 / 1

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



2u

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-8(II)-ADH-15%IPA-1.50ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRALBAH-III-8(II)-ADH-15%IPA-1.50ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-8(II)-ADH-15%IPA-1.50ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/1/2012 6:33:11 PM
Data Processed	: 7/1/2012 7:03:56 PM

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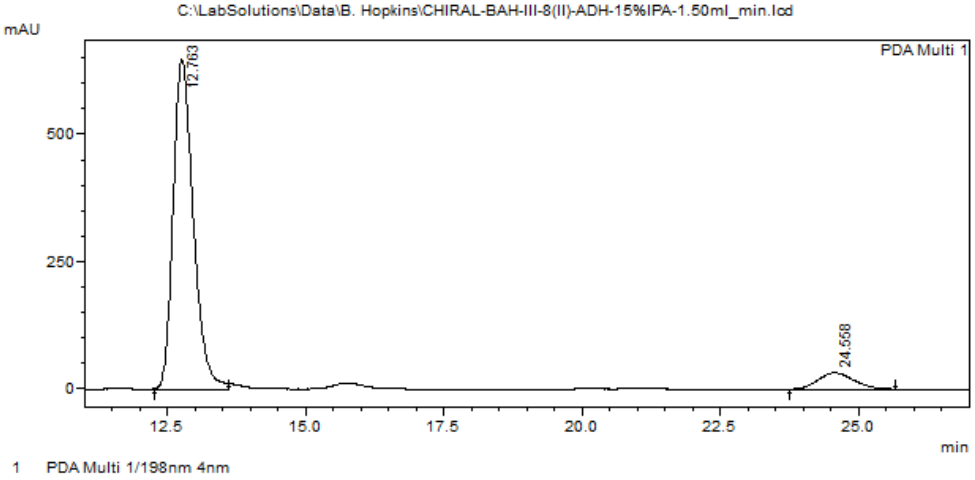
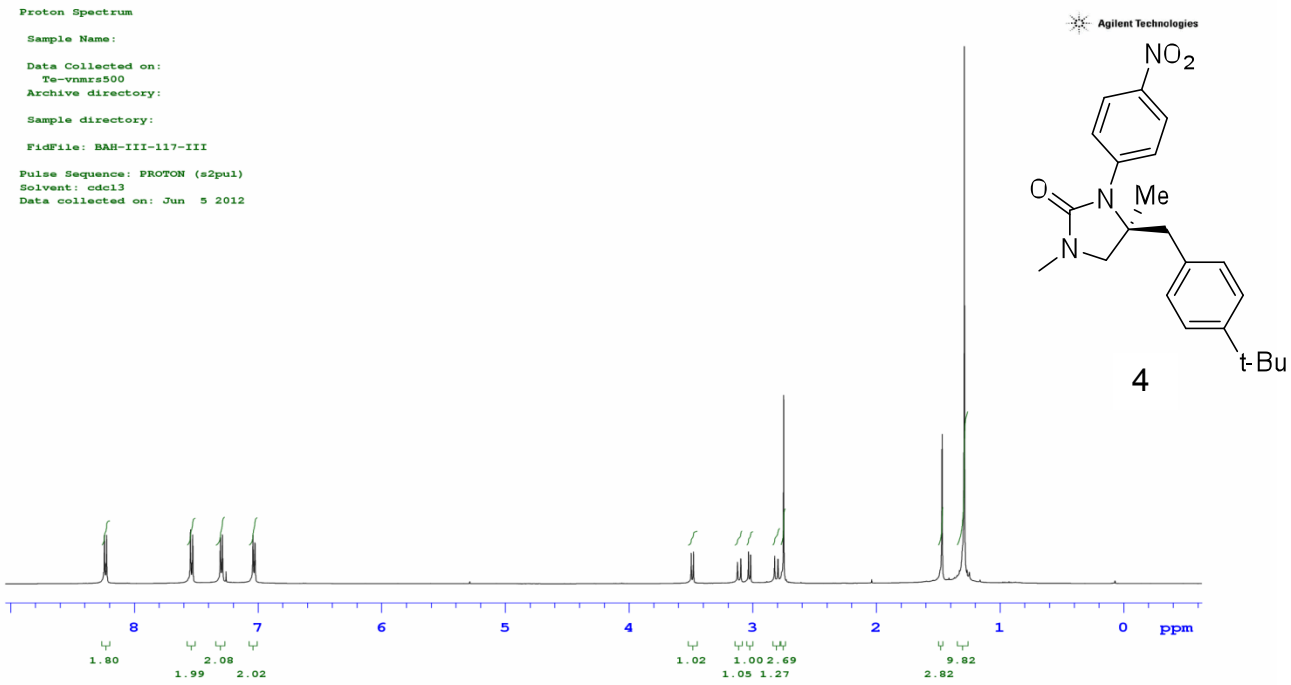


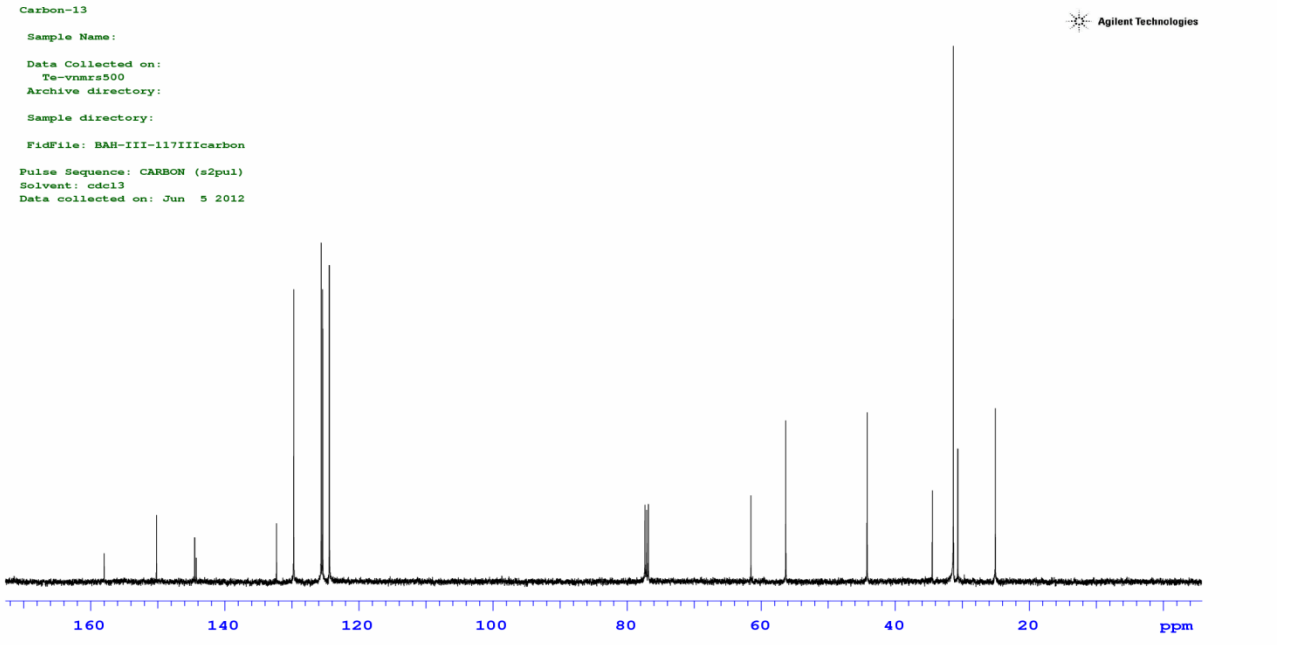
1 PDA Multi 1/198nm 4nm

PeakTable

PDA Ch1 198nm 4nm

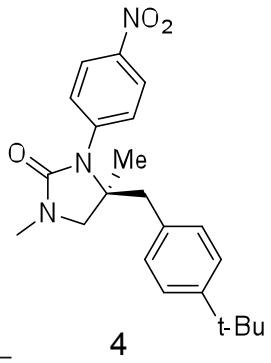
Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.763	16531529	648808	92.070	95.327
2	24.558	1423871	31803	7.930	4.673
Total		17955400	680611	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: BAH-III-117-III
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 5 2012
Agilent Technologies
NO2
O
N
Me
N
t-Bu
4
8
7
6
5
4
3
2
1
0
ppm
1.80
2.08
1.99
2.02
1.02
1.00
2.69
1.05
1.27
9.82
2.82


Carbon-13
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: BAH-III-117IIIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 5 2012
Agilent Technologies
160
140
120
100
80
60
40
20
ppm

6/18/2012 18:24:33 1 / 1

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



4

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-192-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-192-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-192-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/15/2012 4:57:33 PM
Data Processed	: 6/15/2012 5:15:16 PM

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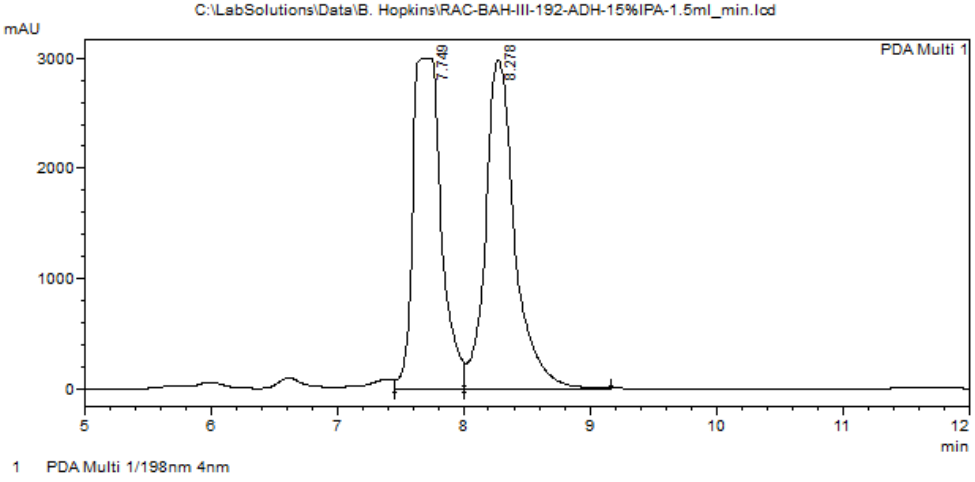


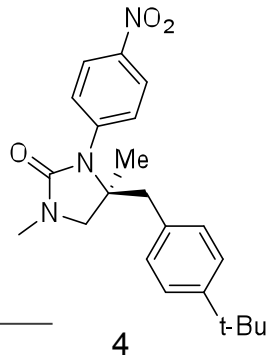
1 PDA Multi 1/198nm 4nm

PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.749	47039979	2993895	49.544	50.052
2	8.278	47905890	2987667	50.456	49.948
Total		94945869	5981562	100.000	100.000

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



4

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-117(III)-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-117(III)-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-117(III)-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/9/2012 4:39:55 PM
Data Processed	: 3/9/2012 5:03:22 PM

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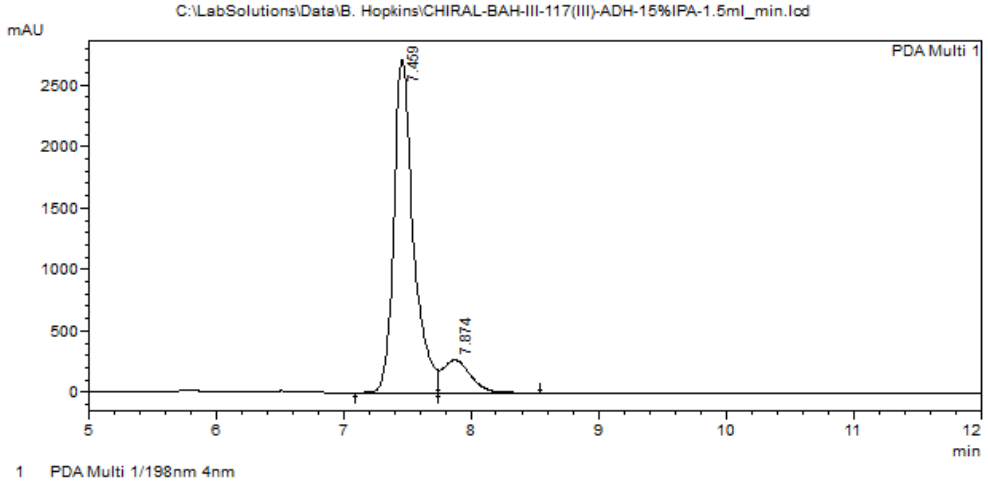
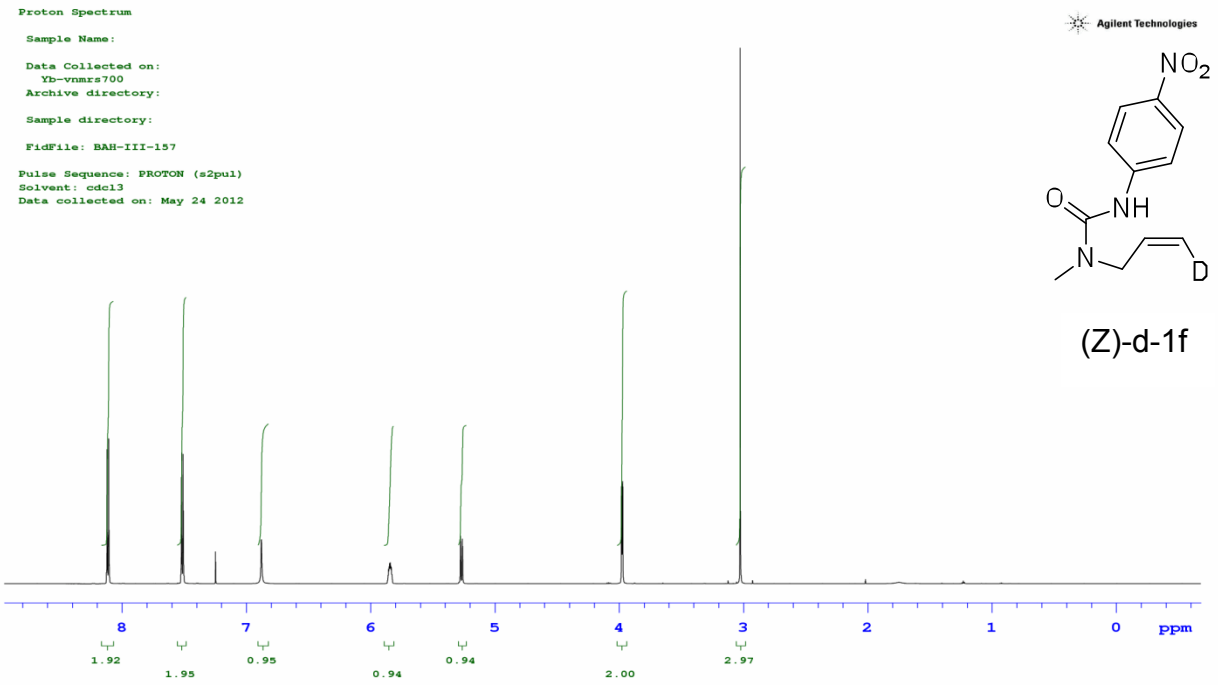


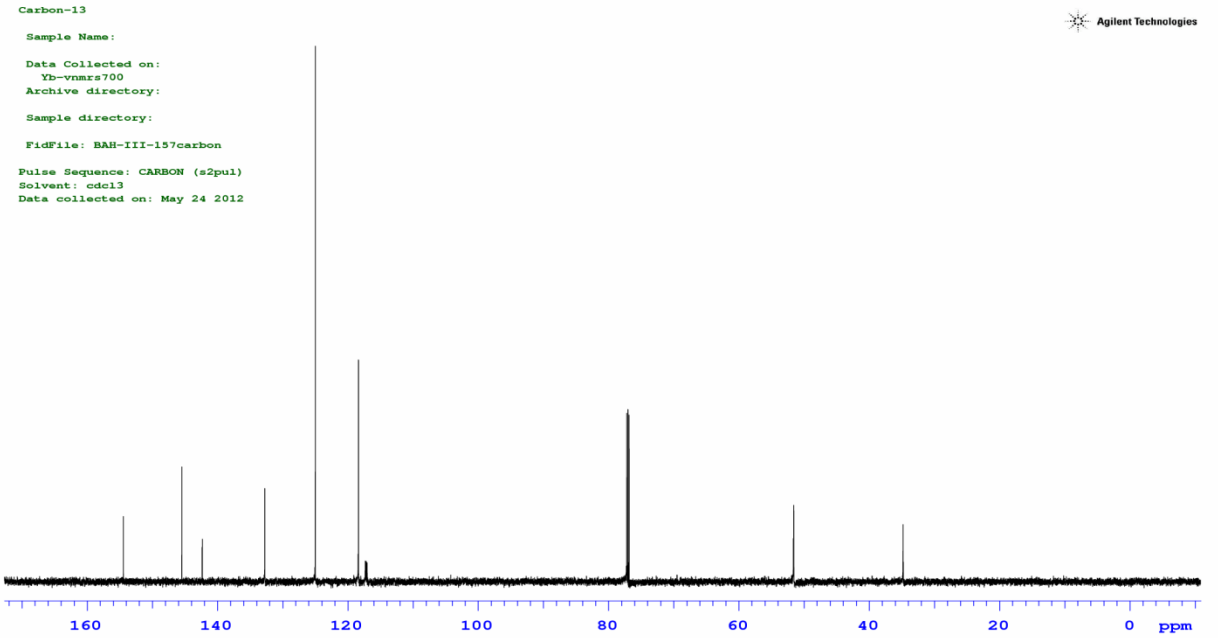
1 PDA Multi 1/198nm 4nm

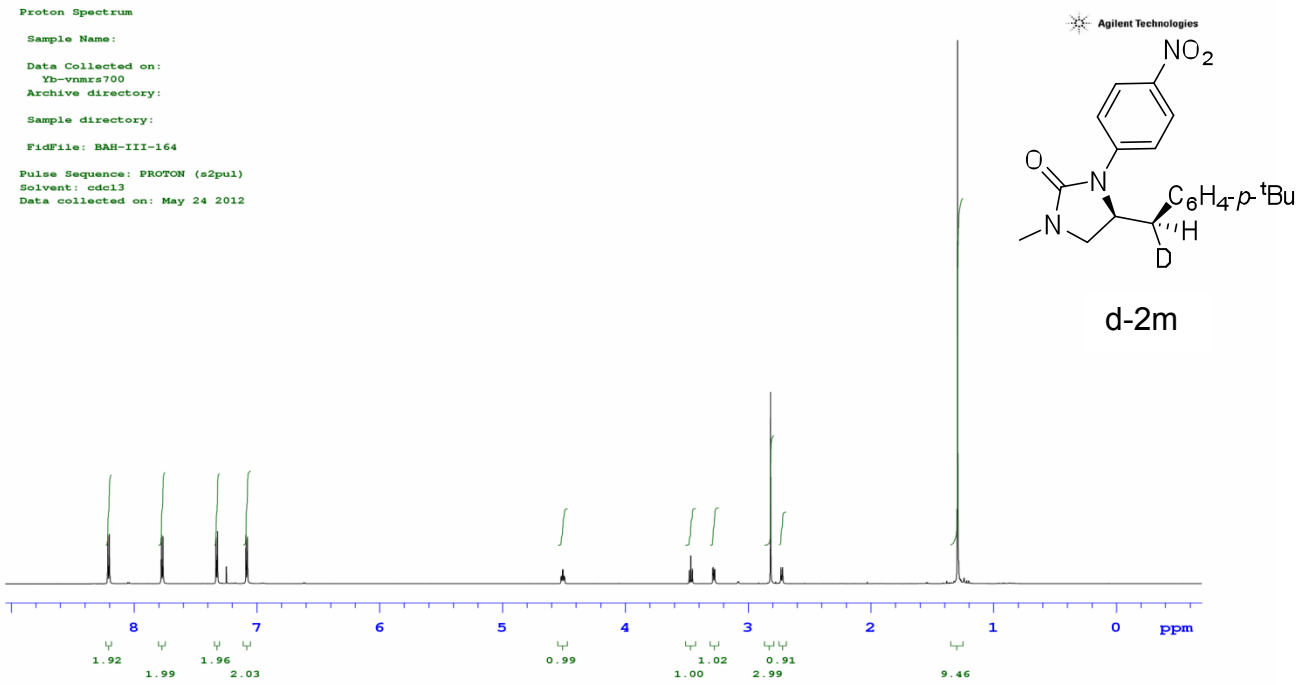
PeakTable

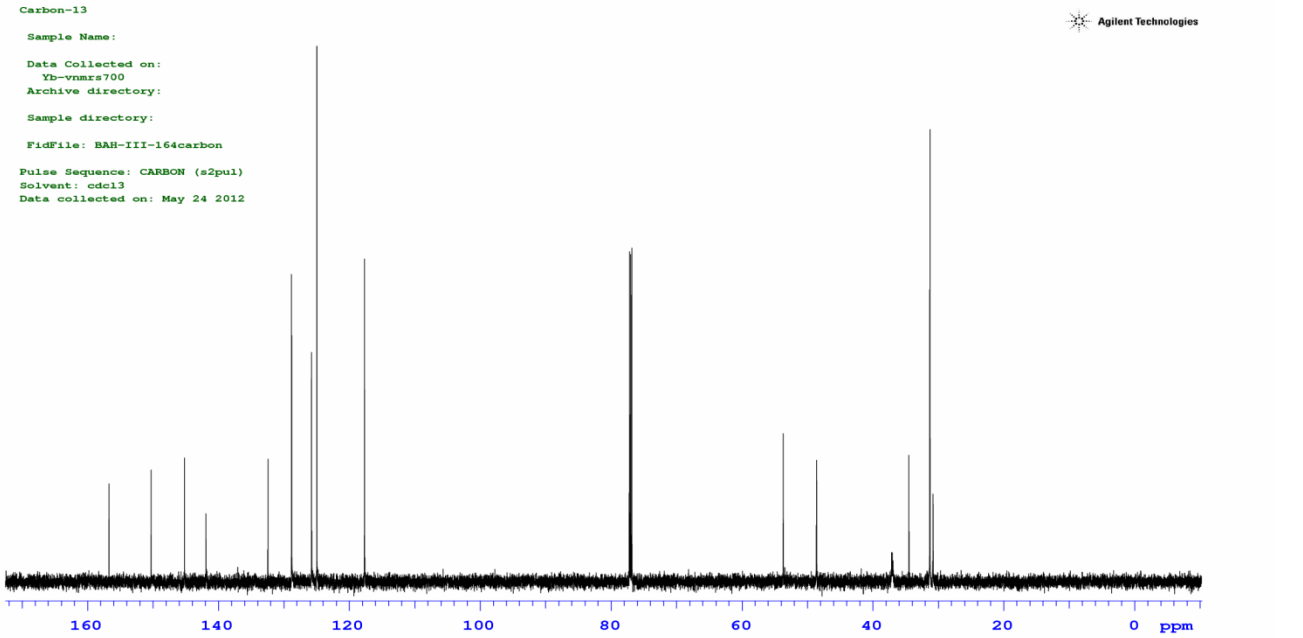
PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.459	28888734	2703157	87.952	91.063
2	7.874	3957164	265284	12.048	8.937
Total		32845898	2968441	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-III-157
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 24 2012
Agilent Technologies
NO2
O
NH
N
D
(Z)-d-1f
8
7
6
5
4
3
2
1
0
ppm
1.92
1.95
0.95
0.94
0.94
2.00
2.97

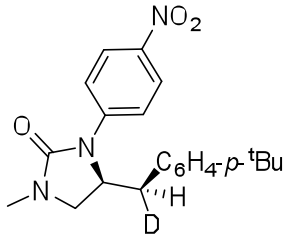

Carbon-13
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-III-157carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 24 2012
Agilent Technologies
160
140
120
100
80
60
40
20
0
ppm


Proton Spectrum
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-III-164
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 24 2012
Agilent Technologies
NO2
O
N
N
C6H4-p-tBu
H
D
d-2m
8
7
6
5
4
3
2
1
0
ppm
1.92
1.99
1.96
2.03
0.99
1.00
1.02
2.99
0.91
9.46


Carbon-13
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-III-164carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 24 2012
Agilent Technologies
160
140
120
100
80
60
40
20
0
ppm

6/18/2012 13:21:30 1 / 1

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

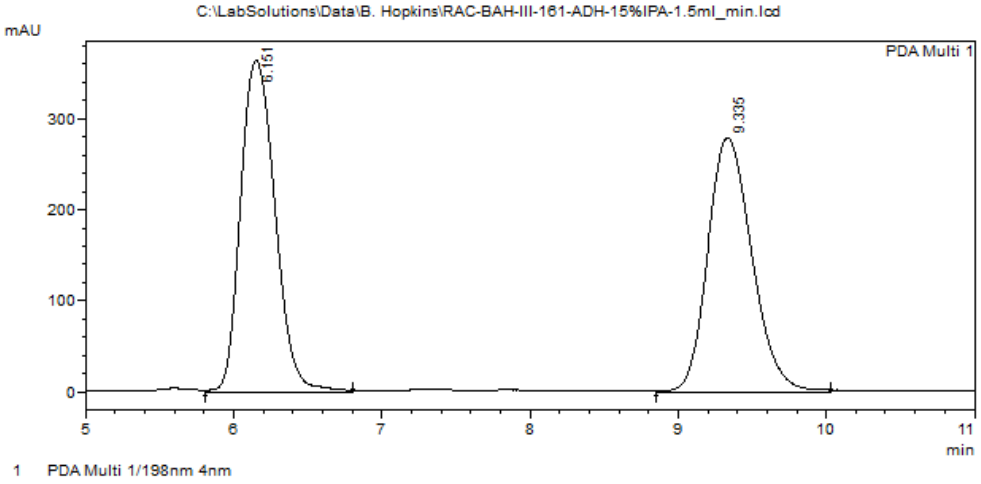


d-2m

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-161-ADH-15%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-161-ADH-15%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-161-ADH-15%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/17/2012 2:13:45 PM
Data Processed	: 5/17/2012 2:40:16 PM

<Chromatogram>



1 PDA Multi 1/198nm 4nm

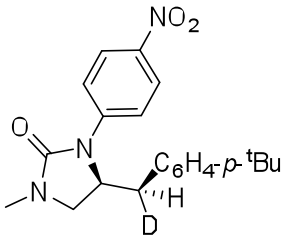
PeakTable

PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.151	5884305	363565	50.295	56.586
2	9.335	5815254	278938	49.705	43.414
Total		11699559	642503	100.000	100.000

7/2/2012 19:03:31 1 / 1

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

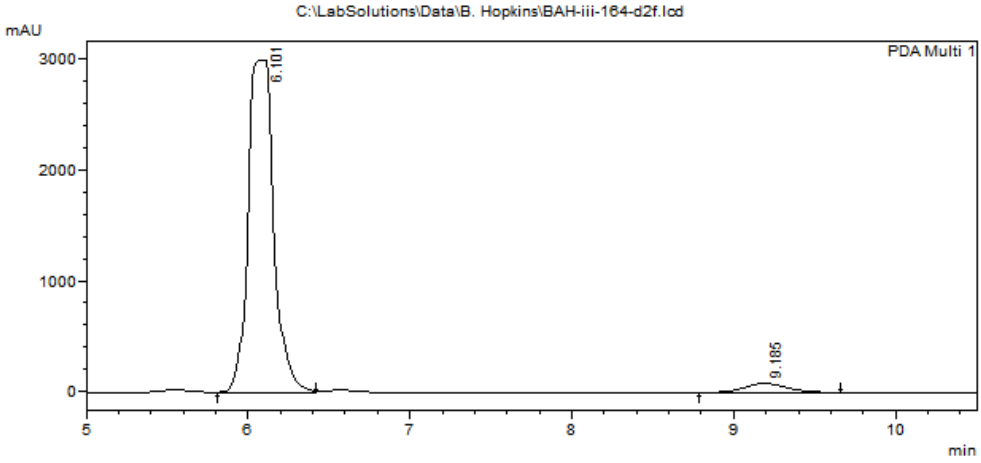


d-2m

C:\LabSolutions\Data\B. Hopkins\BAH-iii-164-d2f.lcd

Acquired by	: Admin
Sample Name	: BAH-iii-164-d2f
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: BAH-iii-164-d2f.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/2/2012 6:48:32 PM
Data Processed	: 7/2/2012 6:59:43 PM

<Chromatogram>

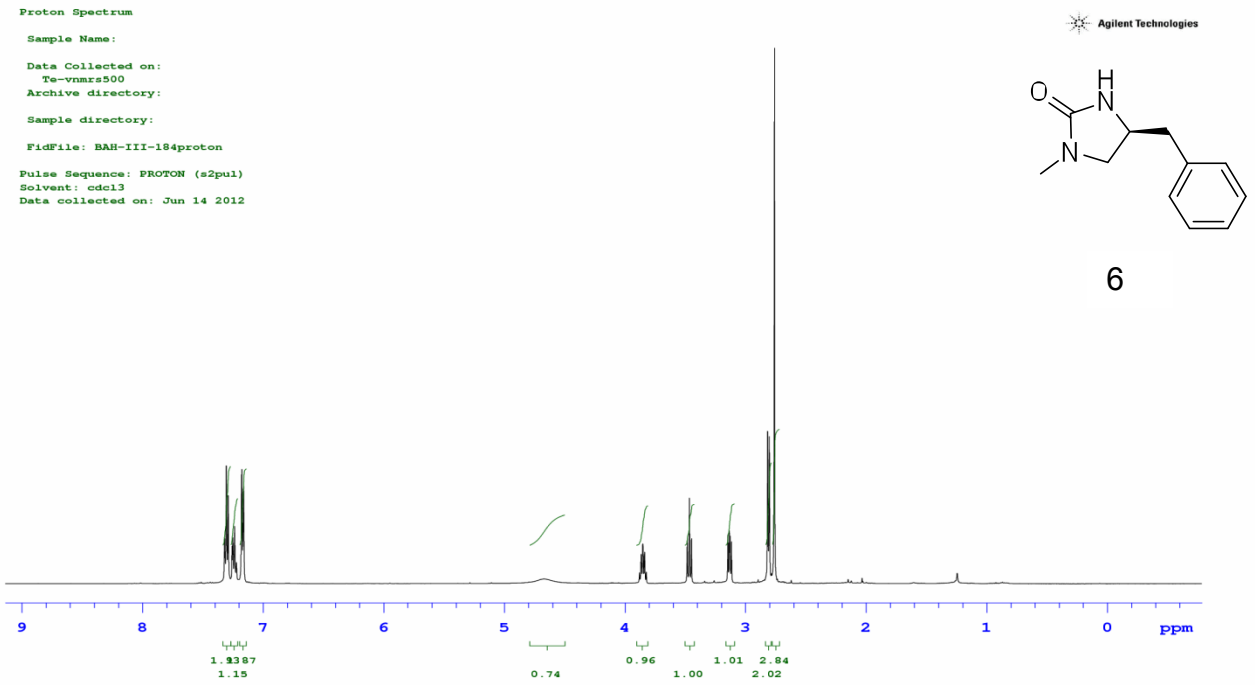


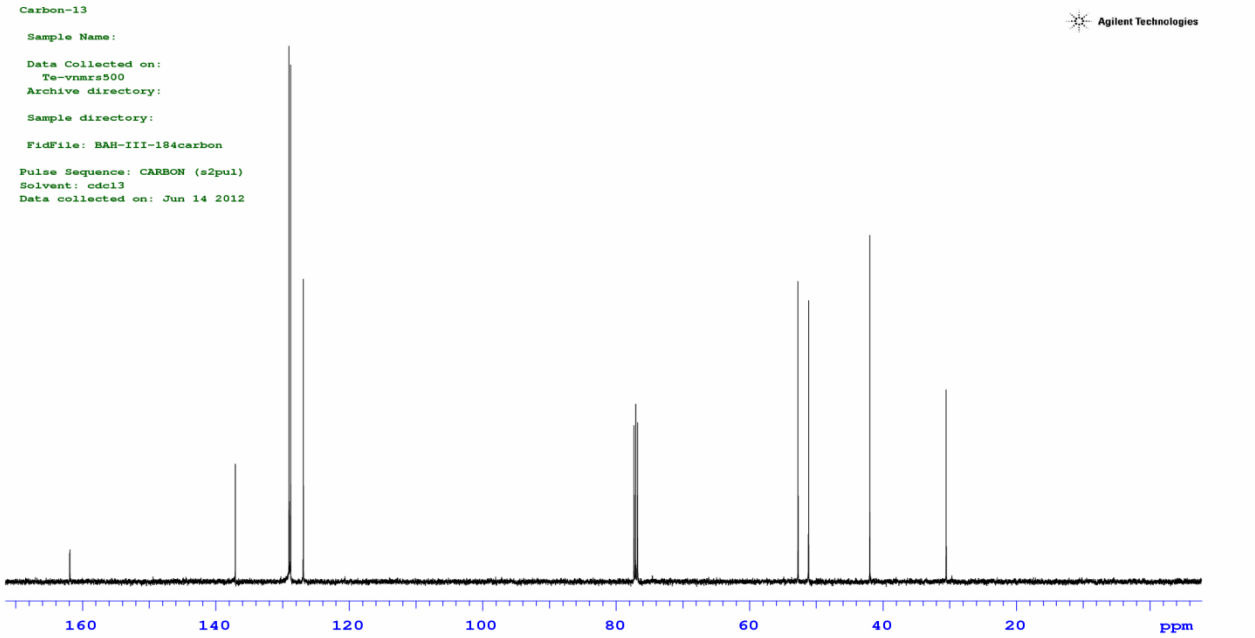
1 PDA Multi 1/198nm 4nm

PeakTable

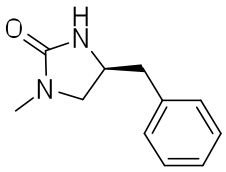
PDA Ch1 198nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.101	33242987	2983724	95.537	97.340
2	9.185	1552846	81526	4.463	2.660
Total		34795833	3065250	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: BAH-III-184proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 14 2012
Agilent Technologies
O
H
N
N
6
9
8
7
6
5
4
3
2
1
0
ppm
1.15
0.74
0.96
1.00
1.01
2.02
2.84


Carbon-13
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: BAH-III-184carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 14 2012
Agilent Technologies
160
140
120
100
80
60
40
20
ppm

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

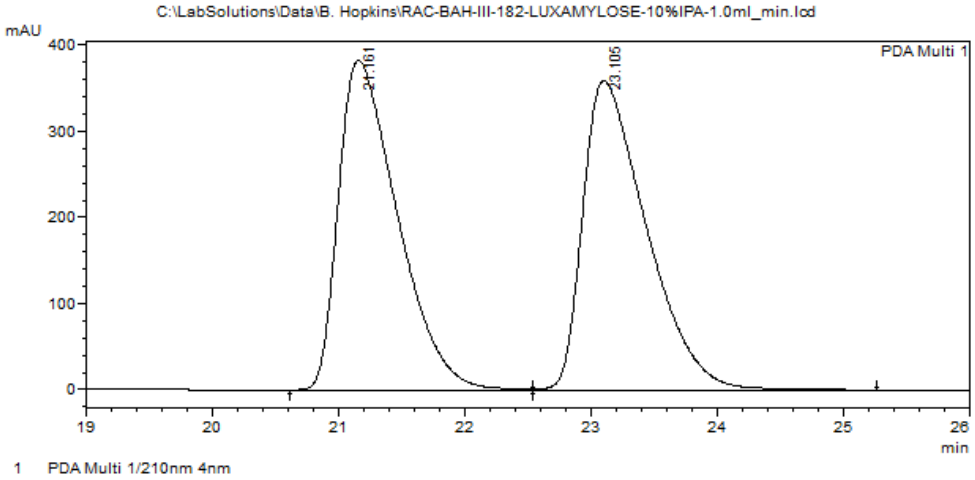


6

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-182-LUXAMYLOSE-10%IPA-1.0ml_min.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-III-182-LUXAMYLOSE-10%IPA-1.0ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-182-LUXAMYLOSE-10%IPA-1.0ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/11/2012 1:48:26 PM
Data Processed	: 6/11/2012 2:36:15 PM

<Chromatogram>



1 PDA Multi 1/210nm 4nm

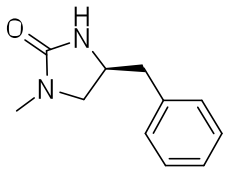
PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.161	12449205	382753	49.721	51.581
2	23.105	12588871	359291	50.279	48.419
Total		25038076	742044	100.000	100.000

6/18/2012 13:39:16 1 / 1

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



6

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-III-184-LUXAMYLOSE-10%IPA-1.0ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-III-184-LUXAMYLOSE-10%IPA-1.0ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-III-184-LUXAMYLOSE-10%IPA-1.0ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/11/2012 3:27:03 PM
Data Processed	: 6/11/2012 3:53:15 PM

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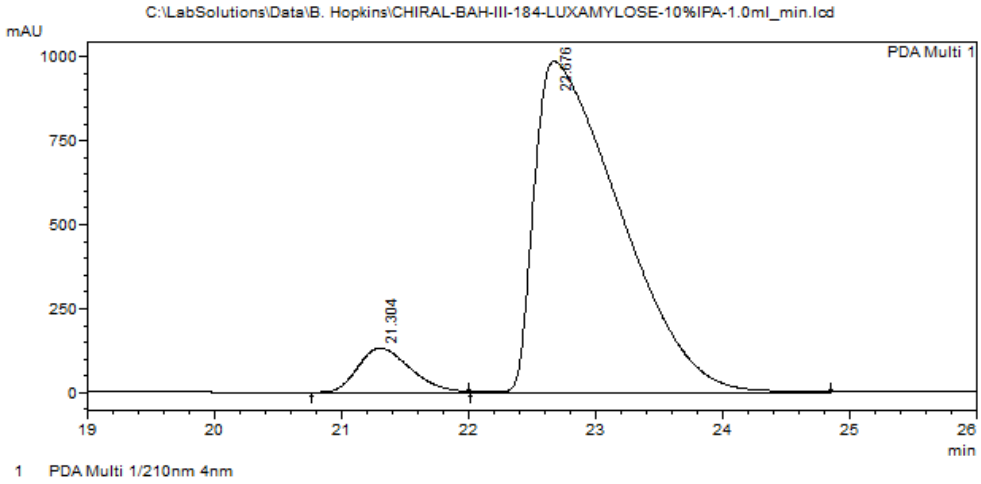

1 PDA Multi 1/210nm 4nm

PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.304	3839790	132706	7.660	11.861
2	22.676	46287854	986107	92.340	88.139
Total		50127644	1118813	100.000	100.000

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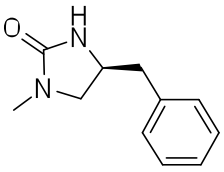
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-183-LUXAMYLOSE-10%IPA-1.0ml_min.lcd

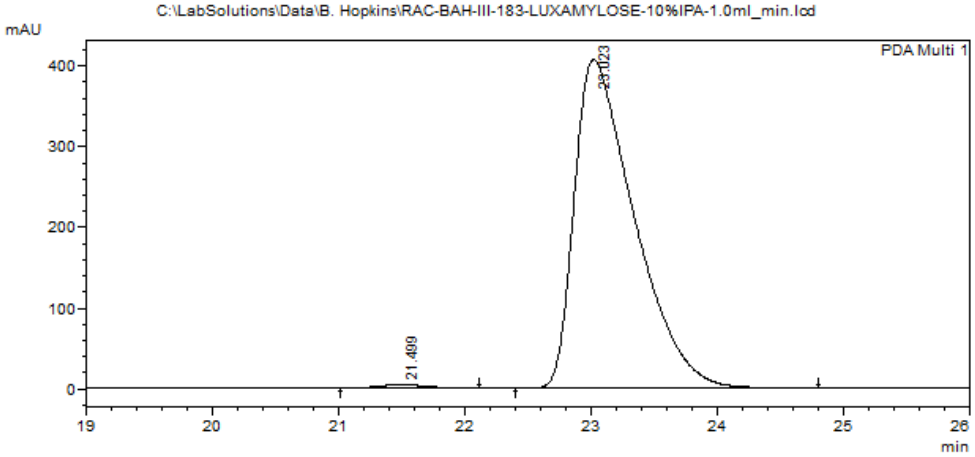
Acquired by	: Admin
Sample Name	: RAC-BAH-III-183-LUXAMYLOSE-10%IPA-1.0ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-III-183-LUXAMYLOSE-10%IPA-1.0ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/11/2012 2:39:30 PM
Data Processed	: 6/11/2012 3:17:28 PM



6 (from L-phenylalanine)

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-III-183-LUXAMYLOSE-10%IPA-1.0ml_min.lcd



1 PDA Multi 1/210nm 4nm

PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.499	120677	4554	0.889	1.106
2	23.023	13458652	407238	99.111	98.894
Total		13579329	411792	100.000	100.000