Enantioselective Synthesis of Tetrahydroquinolines, Tetrahydroquinoxalines, and Tetrahydroisoquinolines via Pd-Catalyzed Alkene Carboamination Reactions

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Supporting Information

Experimental procedures and characterization data for new compounds.

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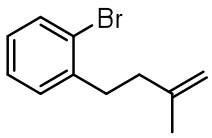
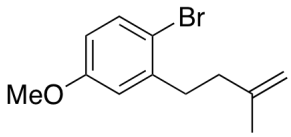
General: Reactions were carried out under nitrogen in flame-dried glassware. Tris(dibenzylideneacetone)dipalladium and (*S*)-Siphos-PE were purchased from Strem Chemical Co. and used without further purification. 2-Allylbenzonitrile was prepared

according to a slight modification of a literature procedure (BuMgCl was used in place of BuMgBr).[1] (*Z*)-1-bromobut-1-ene[2] and 3-(2-Bromophenyl)-*N*-methoxy-*N*-methylpropanamide[3] were synthesized according to published procedures. 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 $\geq$95% pure as judged by ^{1}H NMR or GC analysis. The yields reported in the supporting information describe the result of a single experiment, whereas yields reported in Tables 1–4 and equations 1–3 are average yields of two or more experiments. Thus, the yields reported in the supporting information may differ from those shown in Tables 1–4 and equations 1–3.

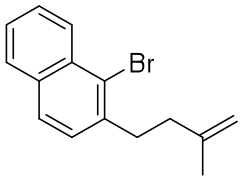


1-Bromo-2-(3-methylbut-3-en-1-yl)benzene (S1a): A flame-dried round bottom flask equipped with a stir bar was cooled under a stream of nitrogen. 2-bromobenzyl bromide (1.25 g, 5.0 mmol), and THF (5 mL) were added to the flask and the resulting solution was cooled to 0 °C. 2-methylallylmagnesium chloride (20 mL, 10 mmol, 0.5 M solution in THF) was slowly added and the resulting mixture was moved into an oil bath and heated to 40 °C for 1.5 h. The mixture was then cooled to rt and quenched with 4 mL of 2M H_2SO_4. Water (5 mL) and ether (15 mL) were added and the mixture was transferred to a separatory funnel. The layers were separated, the aqueous layer was extracted with diethyl ether (3x15 mL), and the organic layers were then combined, dried over anhydrous sodium sulfate, and concentrated in vacuo. The product was purified via flash chromatography on silica gel using hexanes as the eluent to afford 1.06 g (95%) of the title compound as a clear oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.54 (d, J = 7.8 Hz, 1 H), 7.25–7.21 (m, 1 H), 7.08–7.02 (m, 1 H), 4.79 (s, 1 H), 4.76 (s, 1 H), 2.91–2.86 (m, 2 H), 2.32 (t, J = 8.6 Hz, 2 H), 2.81 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ

145.1; 141.5; 132.9; 130.36; 127.7; 127.5; 124.6; 110.7; 38.1; 34.9; 22.7; IR (film) 2933, 1648, 1439 cm^{-1}**.**



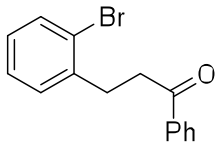
1-Bromo-4-methoxy-2-(3-methylbut-3-en-1-yl)benzene (S1b): The conversion of 2-bromo-5-methoxybenzyl bromide (1.40 g, 5.0 mmol) was accomplished using a procedure analogous to that described above for the preparation of **S1b** except using 2.5 equiv of 2-methylallylmagnesium chloride. This procedure afforded 1.15 g (90%) of the title compound as a clear oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.7 Hz, 1H), 6.77 (d, *J* = 3.1 Hz, 1 H), 6.62 (dd, *J* = 3.1, 8.7 Hz, 1 H), 4.77 (s, 1 H), 4.75 (s, 1 H), 3.78 (s, 3 H), 2.83–2.78 (m, 2 H), 2.29 (t, *J* = 8.5 Hz, 2 H), 1.79 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 159.1, 145.3, 142.6, 133.4, 116.1, 115.0, 113.2, 110.6, 55.6, 38.0, 35.1, 22.7; IR (film) 2933, 1571, 1471 cm^{-1}.



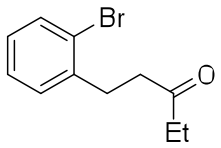
1-Bromo-2-(3-methylbut-3-en-1-yl)naphthalene (S1c): The conversion of 1-bromo-2-(bromomethyl)naphthalene (0.60 g, 2.0 mmol) was accomplished using a procedure analogous to that described above for the preparation of **S1a** except using 2.5 equiv of 2-methylallylmagnesium chloride and a reaction temperature of 45 °C instead of 40 °C for the heated segment of the reaction. This procedure afforded 1.15 g (80%) of the title compound as a clear oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 8.32 (d, *J* = 8.5 Hz, 1 H), 7.80 (d, *J* = 8.0 Hz, 1 H), 7.74 (d, *J* = 8.3 Hz, 1 H), 7.58 (t, *J* = 7.0 Hz, 1 H), 7.48 (t, *J* = 8.0 Hz, 1 H), 7.36 (d, *J* = 8.3 Hz, 1 H), 4.78 (d, *J* = 5.1 Hz, 2 H), 3.12 (t, *J* = 8.8 Hz, 2 H), 2.39 (t, *J*

= 8.9 Hz, 2 H), 1.85 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 145.4, 139.8, 133.4, 132.8, 128.19, 128.18, 127.7, 127.5, 127.4, 126.0, 123.8, 110.7, 38.3, 36.2, 22.8; IR (film) 2916, 1603, 1494 cm^{-1}.

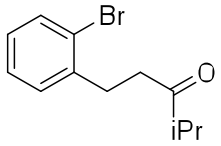
General procedure A: addition of a Grignard reagent to 3-(2-bromophenyl)-*N*-methoxy-*N*-methylpropanamide. A flame dried round bottom flask equipped with a stirbar was cooled under a stream of nitrogen and charged with 3-(2-bromophenyl)-N-methoxy-N-methylpropanamide[3] (1.0 equiv) and diethyl ether (0.40 M) was added to the flask and it was cooled to 0 °C. The appropriate Grignard reagent (1.5 equiv) was added dropwise and the reaction mixture was allowed to slowly warm to rt and stir for 5 hours. The reaction mixture was then cooled to 0 °C and quenched with saturated ammonium chloride (1mL/mmol substrate) and then water (5mL/mmol substrate) was added. This mixture was transferred to a separatory funnel and extracted with EtOAc. The combined organic layers were dried with sodium sulfate and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel.



3-(2-Bromophenyl)-1-phenylpropan-1-one (S2a).[4] General procedure A was used for the reaction of 3-(2-bromophenyl)-*N*-methoxy-*N*-methylpropanamide (2.72 g, 10 mmol), with phenylmagnesium bromide (1 M THF, 15 mL). This procedure afforded 1.92 g (66%) of the title compound as a clear oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.1 Hz, 2 H), 7.57–7.52 (m, 2H), 7.45 (t, *J* = 7.8 Hz, 2 H), 7.31 (d, *J* = 6.9 Hz, 1 H), 7.24 (t, *J* = 8.1 Hz, 1 H), 7.07 (t, *J* = 7.3 Hz, 1 H), 2.82 (t, *J* = 7.3 Hz, 2 H), 2.45 (t, *J* = 7.8 Hz, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 199.0, 140.6, 136.8, 133.2, 132.9, 130.9, 128.7, 128.1, 128.0, 127.7, 124.4, 38.7, 30.1.



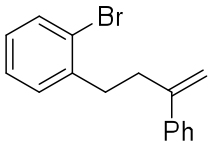
1-(2-Bromophenyl)pentan-3-one (S2b). General procedure A was used for the reaction of 3-(2-bromophenyl)-*N*-methoxy-*N*-methylpropanamide (1.10 g, 4.04 mmol), with ethylmagnesium bromide (3 M THF, 2.02 mL). This procedure afforded 775 mg (80%) of the title compound as a clear oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.47 (d, *J* = 7.8 Hz, 1 H), 7.21–7.16 (m, 2 H), 7.01 (t, *J* = 8.0 Hz, 1 H), 2.97 (t, *J* = 7.7 Hz, 2 H), 2.70 (t, *J* = 7.8 Hz, 2 H), 2.48 (q, *J* = 7.3 Hz, 2 H), 1.01 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 210.0, 140.3, 132.7, 130.5, 127.8, 127.5, 124.1, 41.9, 35.9, 30.3, 7.7; IR (film) 2973, 2936, 1711 cm^{-1}.



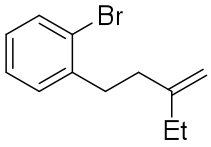
1-(2-Bromophenyl)-4-methylpentan-3-one (S2c). General procedure A was used for the reaction of 3-(2-bromophenyl)-*N*-methoxy-*N*-methylpropanamide (2.72 g, 10 mmol), with isopropylmagnesium chloride (2 M THF, 7.50 mL). This procedure afforded 625 mg (25%) of the title compound as a clear oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.0 Hz, 1 H), 7.24–7.19 (m, 2 H), 7.05 (t, *J* = 8.2 Hz, 1 H), 3.00 (t, *J* = 7.5 Hz, 2 H), 2.77 (t, *J* = 7.7 Hz, 2 H), 2.57 (sept, *J* = 7.0 Hz, 1 H), 1.06 (d, *J* = 7.0 Hz, 6 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 213.4, 140.6, 132.8, 130.7, 127.9, 127.5, 124.2, 41.0, 40.0, 30.5, 18.1; IR (film) 2967, 2932, 1708 cm^{-1}.

General procedure B: Wittig methylenation of ketones. A flame dried round bottom flask equipped with a stirbar was cooled under a stream of nitrogen and charged with methyltriphenylphosphonium bromide (1.4 equiv) and THF (0.15 M). The resulting solution was cooled to 0 °C then potassium *tert*-butoxide (1.4 equiv) added in one portion and the mixture was allowed to stir at 0 °C for 45 min. To this mixture was slowly

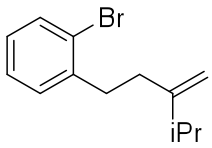
added a solution of the appropriate ketone (1.0 equiv) in THF (0.65 M). The reaction mixture was then allowed to slowly warm to rtand stir for 16 hours. The mixture was concentrated in vacuo, diluted with hexanes, and then filtered through celite. The filtrate was then concentrated in vacuo and the crude product was purified by flash chromatography on silica gel.



1-Bromo-2-(3-phenylbut-3-en-1-yl)benzene (S1d).[4] General procedure B was used for the reaction of methyltriphenylphosphonium bromide (3.31 g, 9.28 mmol), with 3-(2-bromophenyl)-1-phenylpropan-1-one (1.92 g, 6.63 mmol). This procedure afforded 1.06 g (56%) of the title compound as a clear oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.0 Hz, 1 H), 7.46 (d, *J* = 8.2 Hz, 2 H), 7.34 (t, *J* = 6.3 Hz, 2 H), 7.30–7.13 (m, 3H), 7.04 (t, *J* = 8.0 Hz, 1 H), 5.31 (s, 1 H), 5.08 (s, 1 H), 2.91–2.83 (m, 2 H), 2.82–2.76 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.6, 141.2, 140.9, 132.8, 130.6, 128.4, 127.7, 127.5, 127.4, 126.2, 124.4, 112.9, 35.5, 35.4.

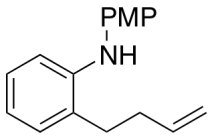


1-Bromo-2-(3-methylenepentyl)benzene (S1e). General procedure B was used for the reaction of methyltriphenylphosphonium bromide (620 mg, 1.73 mmol), with 1-(2-bromophenyl)pentan-3-one (300 mg, 1.24 mmol). This procedure afforded 163 mg (55%) of the title compound as a clear oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.8 Hz, 1 H), 7.23 (d, *J* = 4.7 Hz, 2 H), 7.08–7.04 (m, 1 H), 4.80 (s, 2 H), 2.89 (t, *J* = 8.2 Hz, 2 H), 2.35 (t, *J* = 8.6 Hz, 2 H), 2.13 (q, *J* = 7.4 Hz, 2 H), 1.08 (t, *J* = 7.4 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 150.7, 141.6, 132.8, 130.3, 127.6, 127.4, 124.4, 108.3, 36.4, 35.1, 29.0, 12.4; IR (film) 3024, 2920, 1494 cm^{-1}.

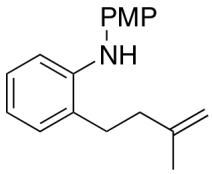


1-Bromo-2-(4-methyl-3-methylenepentyl)benzene (S1f). General procedure B was used for the reaction of methyltriphenylphosphonium bromide (1.19 g, 3.34 mmol), with 1-(2-bromophenyl)-4-methylpentan-3-one (610 mg, 2.40 mmol). This procedure afforded 230 mg (38%) of the title compound as a clear oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.55 (d, *J* = 7.8 Hz, 1 H), 7.25 (d, *J* = 4.4 Hz, 2 H), 7.09–7.05 (m, 1 H), 4.86 (s, 1 H), 4.81 (s, 1 H), 2.89 (t, *J* = 8.2 Hz, 2 H), 2.37–2.31 (m, 3 H), 1.10 (d, *J* = 7.0 Hz, 6 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 155.2, 141.7, 132.8, 130.3, 127.6, 127.4, 124.4, 107.0, 35.4, 34.5, 34.0, 21.9; IR (film) 2959, 2927, 1470 cm^{-1}.

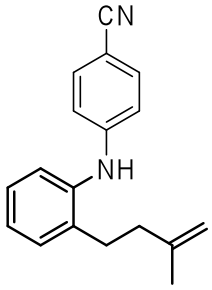
General procedure C: synthesis of 2-(3-methylbut-3-en-1-yl)aniline substrates. A flame dried Schlenk flask equipped with a stir bar was cooled under a stream of nitrogen and charged with $Pd_2(dba)_3$ (0.75 mol %), XPhos (2.25 mol %), 1-bromo-2-(3-methylbut-3-en-1-yl)benzene (1.0 equiv), the appropriate aniline derivative (1.2 equiv), and NaO^{t}Bu (1.5 equiv). The flask was then purged with nitrogen, and toluene (0.5 M) was added. The resulting mixture was heated to 105 °C with stirring until the starting material had been consumed as judged by TLC, GC, or ^{1}H NMR analysis of an aliquot removed from the reaction mixture (ca. 12 h). The reaction mixture was then cooled to rt, saturated aqueous ammonium chloride (6 mL/mmol substrate) was added, and the mixture was transferred to a separatory funnel. The mixture was extracted with ethyl acetate (3 x 20 mL) then the organic layers were combined, dried over anhydrous sodium sulfate, filtered, and concentrated in vacuo. The crude product was then purified by flash chromatography on silica gel using a hexanes/Et_2O mixture as the eluent.



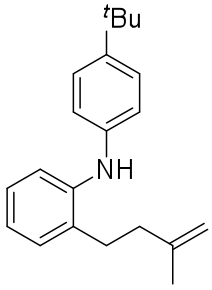
2-(But-3-en-1-yl)-*N*-(4-methoxyphenyl)aniline (1a). General Procedure C was employed for the coupling of 1-bromo-2-(but-3-en-1-yl)benzene (211 mg, 1.0mmol) and *p*-anisidine (147 mg, 1.2 mmol). This procedure afforded 190 mg (75%) of the title compound as a yellow oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.18 (d, *J* = 7.7 Hz, 1 H), 7.09 (t, *J* = 7.6 Hz, 1 H), 7.03 (d, *J* = 8.0 Hz, 1 H), 6.98 (d, *J* = 8.7 Hz, 2 H), 6.89–6.83 (m, 3H), 5.95–5.89 (m, 1H), 5.18 (s, br, 1 H), 5.09 (app. d, *J* = 17.0 Hz, 1 H), 4.86 (app. d, *J* = 10.2 Hz, 1 H), 4.09 (s, 3 H), 2.69 (t, *J* = 7.5 Hz, 2 H), 2.42 (q, *J* = 6.6 Hz, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 155.0, 142.9, 138.2, 136.9, 129.8, 129.7, 127.0, 121.7, 120.6, 116.8, 115.3, 114.8, 55.7, 33.5, 30.9; IR (film) 3402, 2924, 1508 cm^{-1}; MS (ESI+) 254.1543 (254.1539 calcd for $C_{17}H_{19}NO$, M + H^+).



***N*-(4-Methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (1b).** General Procedure C was employed for the coupling of 1-bromo-2-(3-methylbut-3-en-1-yl)benzene (1.125 g, 5.0 mmol) and *p*-anisidine (738 mg, 6.0 mmol). This procedure afforded 1.09 g (82%) of the title compound as a yellow oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.28 (d, *J* = 7.7 Hz, 1 H), 7.18 (t, *J* = 8.7 Hz, 1 H), 7.14 (d, *J* = 8.4 Hz, 1 H), 7.06 (d, *J* = 8.4 Hz, 2 H), 6.98–6.92 (m, 3H), 5.40 (s, br, 1 H), 4.88 (s, 1 H), 4.86 (s, 1 H), 3.86 (s, 3 H), 2.82 (t, *J* = 8.4 Hz, 2 H), 2.45 (t, *J* = 8.4 Hz, 2 H), 1.88 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 155.0, 145.6, 142.8, 137.0, 130.2, 129.7, 127.0, 121.5, 120.7, 117.0, 114.8, 110.5, 55.6, 37.4, 29.9, 22.8; IR (film) 3398, 2933, 1507 cm^{-1}; MS (ESI+) 268.1704 (268.1696 calcd for $C_{18}H_{21}NO$, M + H^+).

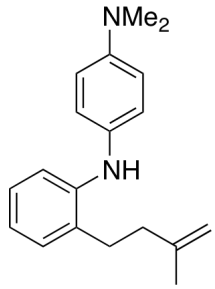


4-{[2-(3-Methylbut-3-en-1-yl)phenyl]amino}benzonitrile (1c). General Procedure C was employed for the coupling of 1-bromo-2-(3-methylbut-3-en-1-yl)benzene (225 mg, 1.0 mmol) and *p*-cyanoaniline (142 mg, 1.20 mmol) except using a catalyst loading of 1 mol % $Pd_2(dba)_3$. This procedure afforded 223 mg (28%) of the title compound as a yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 11.9 Hz, 2 H), 7.30 (d, *J* = 11.2 Hz, 1 H), 7.28–7.16 (m, 3 H), 6.75 (d, *J* = 12.6 Hz, 2 H), 5.89 (s, br, 1 H), 4.74 (s, 1 H), 4.65 (s, 1 H), 2.73 (t, *J* = 10.5 Hz, 2 H), 2.26 (t, *J* = 11.9 Hz, 2 H), 1.72 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 149.9, 145.2, 137.8, 137.4, 133.8, 130.5, 127.3, 126.1, 125.3, 120.3, 114.2, 110.9, 100.5, 38.4, 30.1, 22.7; IR (film) 3338, 2927, 2213, 1513 cm^{-1}; MS (ESI+) 263.1546 (263.1543 calcd for $C_{18}H_{18}N_2$, M + H^+).

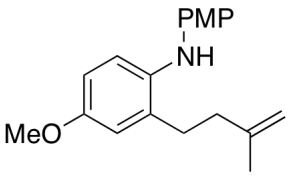


***N*-[4-(*tert*-Butyl)phenyl]-2-(3-methylbut-3-en-1-yl)aniline (1d).** General Procedure C was employed for the coupling of 1-bromo-2-(3-methylbut-3-en-1-yl)benzene (225 mg, 1.0 mmol) and *p-tert*-butylaniline (0.19 mL, 1.2 mmol). This procedure afforded 210 mg (72%) of the title compound as a yellow oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.41 (d, *J* = 9.1 Hz, 2 H), 7.37 (d, *J* = 7.7 Hz, 1 H), 7.32 (d, *J* = 7.7 Hz, 1 H), 7.25 (t, *J* = 7.7 Hz, 1 H), 7.09–7.03 (m, 3 H), 5.52 (s, br, 1 H), 4.91 (s, 1 H), 4.88 (s, 1 H), 2.87 (t, *J* = 8.4 Hz, 2 H), 2.46 (t, *J* = 9.1 Hz, 2 H), 1.90 (s, 3 H), 1.45 (s, 9 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ

145.6, 143.5, 141.7, 141.5, 132.1, 129.9, 127.0, 126.2, 121.9, 119.2, 117.6, 110.7, 37.8, 34.2, 31.7, 30.1, 22.8; IR (film) 3398, 2961, 1514 cm^{-1}; MS (ESI+) 294.2228 (294.2216 calcd for $C_{21}H_{27}N$, M + H^+).

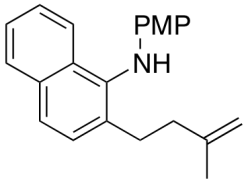


***N*^{1},*N*^{1}-Dimethyl-*N*^{4}-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (1e).** General Procedure C was employed for the coupling of 1-bromo-2-(3-methylbut-3-en-1-yl)benzene (1.10 g, 4.88 mmol) and 4-(dimethylamino)aniline (798 mg, 5.86 mmol). This procedure afforded 1.16 g (85%) of the title compound as a yellow oil. ^{1}H NMR (700 MHz, C_6D_6) δ 7.15–7.13 (m, 1 H), 7.10 (d, *J* = 9.7 Hz, 1 H), 7.07 (t, *J* = 7.8 Hz, 1 H), 6.98 (d, *J* = 8.9 Hz, 2 H), 6.86 (t, *J* = 7.3 Hz, 1 H), 6.61 (d, *J* = 8.9 Hz, 2 H), 5.06 (s, 1 H), 4.78 (s, 2 H), 2.61 (t, *J* = 7.8 Hz, 2 H), 2.56 (s, 6 H), 2.29 (t, *J* = 8.5 Hz, 2 H), 1.62 (s, 3 H); ^{13}C NMR (175 MHz, C_6D_6) δ 147.5, 145.6, 144.5, 134.0, 129.8, 129.4, 127.3, 123.4, 120.2, 116.4, 114.5, 110.8, 41.0, 37.5, 30.2, 22.7; IR (film) 3410, 2934, 1516 cm^{-1}; MS (ESI+) 281.2018 (281.2012 calcd for $C_{19}H_{24}N_2$, M + H^+).

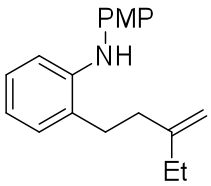


4-Methoxy-N-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (1f). General Procedure C was employed for the coupling of 1-bromo-4-methoxy-2-(3-methylbut-3-en-1-yl)benzene (420 mg, 1.65 mmol) and *p*-anisidine (243 mg, 1.98 mmol) and a reaction time of 3 h. This procedure afforded 421 mg (86%) of the title compound as an orange

oil. ^{1}H NMR (700 MHz, C_6D_6) δ 7.05 (d, *J* = 8.7 Hz, 1 H), 6.88 (d, *J* = 2.7 Hz, 1 H), 6.77 (d, *J* = 8.9 Hz, 2 H), 6.68–6.62 (m, 3 H), 4.77–4.74 (m, 2 H), 4.66 (s, br, 1 H), 3.40 (s, 3 H), 3.36 (s, 3 H), 2.64 (t, *J* = 8.9 Hz, 2 H), 2.24 (t, *J* = 8.3 Hz, 2 H), 1.59 (s, 3 H); ^{13}C NMR (175 MHz, C_6D_6) δ 156.3, 153.7, 145.1, 140.3, 137.0, 135.0, 124.4, 117.3, 115.6, 114.8, 111.9, 110.4, 54.8, 54.7, 38.0, 30.2, 22.2; IR (film) 3379, 2933, 1509 cm^{-1}; MS (ESI+) 297.1726 (297.1723 calcd for $C_{19}H_{23}NO_2$, M + H$^+$).

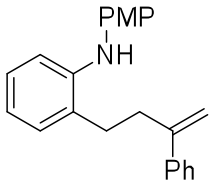


***N*-(4-Methoxyphenyl)-2-(3-methylbut-3-en-1-yl)naphthalen-1-amine (1g).** General Procedure C was employed for the coupling of 1-bromo-2-(3-methylbut-3-en-1-yl)naphthalene (380 mg, 1.38 mmol) and *p*-anisidine (204 mg, 1.65 mmol) except using a catalyst composed of $Pd_2(dba)_3$ (25 mg, 0.0276 mmol, 2.0 mol %) and JohnPhos (16.5 mg, 0.0552 mmol, 4.0 mol %). This procedure afforded 308 mg (70%) of the title compound as a yellow solid, mp 80–84 °C. ^{1}H NMR (700 MHz, C_6D_6) δ 8.06 (d, *J* = 8.2 Hz, 1 H), 7.71 (d, *J* = 7.2 Hz, 1 H), 7.59 (d, *J* = 8.3 Hz, 1 H), 7.28–7.23 (m, 3 H), 6.66 (d, *J* = 8.9 Hz, 2 H), 6.36 (d, *J* = 8.9 Hz, 2 H), 4.93 (s, 1H), 4.73 (d, *J* = 15.2 Hz, 2 H), 3.30 (s, 3 H), 2.80 (t, *J* = 8.8 Hz, 2 H), 2.24 (t, *J* = 8.2 Hz, 2 H), 1.60 (s, 3 H); ^{13}C NMR (175 MHz, C_6D_6) δ 153.4, 145.2, 142.1, 136.7, 135.9, 134.2, 132.5, 128.6, 128.5, 128.4, 126.4, 125.7, 124.6, 115.4, 115.2, 111.0, 55.2, 39.1, 31.1, 22.5; IR (film) 3382, 2959, 1505 cm^{-1}; MS (ESI+) 317.1771 (317.1771 calcd for $C_{22}H_{23}NO$, M + H$^+$).

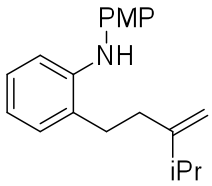


***N*-(4-Methoxyphenyl)-2-(3-methylenepentyl)aniline (1h).** General Procedure C was

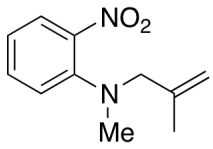
employed for the coupling of 1-bromo-2-(3-methylenepentyl)benzene (160 mg, 0.67 mmol) and *p*-anisidine (99 mg, 0.80 mmol) except using a catalyst composed of $Pd_2(dba)_3$ (9.2 mg, 0.010 mmol, 1.5 mol %) and XPhos (12.7 mg, 0.0268 mmol, 4.0 mol %). This procedure afforded 170 mg (90%) of the title compound as a pale yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.17 (d, *J* = 7.3 Hz, 1 H), 7.09 (t, *J* = 7.8 Hz, 1 H), 7.04 (d, *J* = 7.8 Hz, 1 H), 6.98 (d, *J* = 9.2 Hz, 2 H), 6.90–6.83 (m, 3H), 5.30 (s, br, 1 H), 4.8 (s, 2 H), 3.80 (s, 3 H), 2.73 (t, *J* = 7.8 Hz, 2 H), 2.39 (t, *J* = 8.3 Hz, 2 H), 2.10 (q, *J* = 7.3 Hz, 2 H), 1.06 (t, *J* = 7.3 Hz, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.0, 151.2, 142.8, 136.9, 130.2, 129.6, 126.9, 121.3, 120.6, 116.7, 114.8, 108.2, 55.7, 35.9, 30.2, 29.1, 12.4; IR (film) 3392, 2960, 1508 cm^{-1}; MS (ESI+) 282.1849 (282.1852 calcd for $C_{19}H_{23}NO$, M + H^+).



***N*-(4-Methoxyphenyl)-2-(3-phenylbut-3-en-1-yl)aniline (1i).** General Procedure C was employed for the coupling of 1-bromo-2-(3-phenylbut-3-en-1-yl)benzene (1.06 g, 3.70 mmol) and *p*-anisidine (547 mg, 4.44 mmol). This procedure afforded 1.18 g (95%) of the title compound as a pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J* = 7.6 Hz, 2 H), 7.42–7.32 (m, 3 H), 7.24–7.08 (m, 3 H), 6.98–6.85 (m, 5 H), 5.38 (s, 1 H), 5.20 (s, br, 1 H), 5.14 (s, 1H), 3.83 (s, 3 H), 2.92 (t, *J* = 8.8 Hz, 2 H), 2.80 (t, *J* = 9.0 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.8, 147.7, 147.6, 142.6, 140.7, 136.9, 130.1, 129.8, 128.5, 127.6, 127.0, 126.1, 121.1, 120.7, 117.2, 114.7, 113.0, 55.6, 35.3, 30.5; IR (film) 3406, 2938, 1508 cm^{-1}; MS (ESI+) 330.1852 (330.1852 calcd for $C_{23}H_{23}NO$, M + H^+).

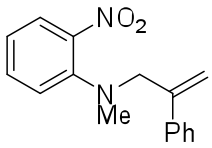


***N*-(4-Methoxyphenyl)-2-(4-methyl-3-methylenepentyl)aniline (1j).** General Procedure C was employed for the coupling of 1-bromo-2-(4-methyl-3-methylenepentyl)benzene (150 mg, 0.59 mmol) and *p*-anisidine (88 mg, 0.71 mmol) except using a catalyst composed of $Pd_2(dba)_3$ (11 mg, 0.0118 mmol, 2.0 mol %) and XPhos (11 mg, 0.0236 mmol, 4.0 mol %). This procedure afforded 170 mg (97%) of the title compound as a pale yellow oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.16 (d, *J* = 7.3 Hz, 1 H), 7.08 (t, *J* = 8.0 Hz, 1 H), 7.03 (d, *J* = 7.8 Hz, 1 H), 6.98 (d, *J* = 8.7 Hz, 2 H), 6.87–6.83 (m, 3H), 5.29 (s, br, 1 H), 4.82 (s, 1 H), 4.77 (s, 1 H), 3.79 (s, 3 H), 2.72 (t, *J* = 8.0 Hz, 2 H), 2.36 (t, *J* = 8.5 Hz, 2 H), 2.28 (sept, *J* = 7.0 Hz, 1 H), 1.04 (d, *J* = 6.8 Hz, 6 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 155.7, 155.0, 142.8, 136.8, 130.2, 129.7, 126.9, 121.7, 120.5, 116.6, 114.7, 106.9, 55.7, 34.1, 33.9, 30.4, 21.9 ; IR (film) 3389, 2958, 1508 cm^{-1}; MS (ESI+) 296.2007 (296.2009 calcd for $C_{20}H_{25}NO$, M + H^+).

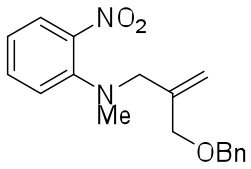


***N*-Methyl-*N*-(2-methylallyl)-2-nitroaniline (S3a).** A flame dried flask equipped with a stir bar was cooled under nitrogen and charged with 1-Fluoro-2-nitrobenzene (2.10 mL, 20 mmol) and anhydrous DMF (20 mL). Methylamine (20 mL, 40 mmol, 2.0 M in THF) was added and the resulting mixture was heated to 50 °C for 12 hours. The reaction mixture was then cooled to rt, and excess methylamine and THF were evaporated in vacuo to afford crude *N*-methyl-2-nitroaniline, which was dissolved in DMF (15 mL) and added slowly to a flame dried flask containing a suspension of NaH (880 mg, 22 mmol, 60% in mineral oil) in DMF (10 mL) that had been cooled to 0 °C. The resulting mixture was stirred for 30 minutes at 0 °C then 3-bromo-2-methylpropene (3.05 mL, 30 mmol),

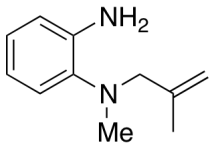
was added slowly. The ice bath was removed, the reaction flask was placed in an oil bath (rt) and then was heated to 100 °C for 6 h. The mixture was then allowed to cool to room temperature and saturated aqueous NH_4Cl (40 mL) and EtOAc (80 mL) were added. The mixture was transferred to a separatory funnel, the layers were separated, and the organic layer was separated and washed with brine (2 x 10 mL). The organic layer was then dried over anhydrous sodium sulfate, filtered and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel using hexanes/Et_2O as the eluant to afford 2.25 g (51%) of the title compound as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.2 Hz, 1 H), 7.34 (t, *J* = 7.8 Hz, 1 H), 7.01 (d, *J* = 8.4 Hz, 1 H), 6.81 (t, *J* = 7.6 Hz, 1 H), 4.91 (s, 1 H), 4.84 (s, 1 H), 3.71 (s, 2 H), 2.76 (s, 3 H), 1.69 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.8, 140.6, 140.1, 133.1, 126.6, 119.8, 118.5, 113.1, 60.6, 40.2, 20.2; IR (film) 2914, 1604, 1512 cm^{-1}.



***N*-Methyl-2-nitro-*N*-(2-phenylallyl)aniline (S3b).** Following the above procedure, 1-Fluoro-2-nitrobenzene (0.97 mL, 9.25 mmol) and methylamine (9.25 mL, 18.5 mmol, 2.0 M in THF) were reacted to form the crude *N*-methyl-2-nitroaniline. This crude product was added dropwise to a flame dried round bottom flask containing a suspension of NaH (60% in mineral oil 406 mg, 10.15 mmol) in DMF at 0 °C. Neat (3-bromoprop-1-en-2-yl)benzene[5] (2.73 g, 13.85 mmol) was added to this mixture. This procedure afforded 1.64 g (66%) of the title compound as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.4 Hz, 1 H), 7.38–7.26 (m, 6 H), 6.95 (d, *J* = 8.6 Hz, 1 H), 6.82 (t, *J* = 7.4 Hz, 1 H), 5.51 (s, 1 H), 5.22 (s, 1 H), 4.22 (s, 2 H), 2.84 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.4, 142.5, 139.6, 138.9, 132.9, 128.3, 127.9, 126.3, 126.0, 119.5, 118.2, 114.3, 58.0, 39.9; IR (film) 2931, 1604, 1511 cm^{-1}.

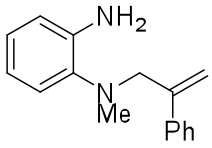


***N*-{2-[(Benzyloxy)methyl]allyl}-N-methyl-2-nitroaniline (S3c).** Following the above procedure, 1-Fluoro-2-nitrobenzene (0.84 mL, 8.00 mmol) and methylamine (8 mL, 16 mmol, 2.0 M in THF) were reacted to form the crude *N*-methyl-2-nitroaniline. This crude product was added dropwise to a flame dried round bottom flask containing a suspension of NaH (60% in mineral oil, 352 mg, 8.80 mmol) in DMF at 0 °C. Neat ({[(2-(chloromethyl)allyl]oxy}methyl)benzene[6] (2.35 g, 12.0 mmol) was added to this mixture. This procedure afforded 810 mg (33%) of the title compound as a yellow oil containing a slight unknown impurity. This material was used without further purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.6 Hz, 1 H), 7.40–7.23 (m, 6 H), 7.05 (d, *J* = 8.4 Hz, 1 H), 6.82 (t, *J* = 7.2 Hz, 1 H), 5.24 (s, 1 H), 5.12 (s, 1 H), 4.46 (s, 2 H), 3.98 (s, 2 H), 3.89 (s, 2 H), 2,76 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.6, 145.6, 141.1, 138.1, 133.0, 128.4, 127.7, 127.6, 126.4, 119.8, 118.5, 115.1, 72.4, 71.3, 56.7, 40.4; IR (film) 2916, 2848, 1511 cm^{-1}.

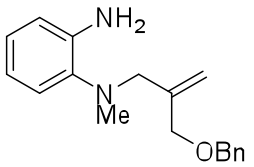


***N*^{1}-Methyl-*N*^{1}-(2-methylallyl)benzene-1,2-diamine (S4a).** A flame dried flask equipped with a stir bar was cooled under nitrogen and charged with zinc dust (8.56 g, 130.9 mmol) and anhydrous ethanol (75 mL). The resulting suspension was vigorously stirred, glacial acetic acid (7.50 mL, 131 mmol) was added, and the mixture was cooled to 0 °C. A solution of *N*-methyl-*N*-(2-methylallyl)-2-nitroaniline (1.80 g, 8.7 mmol) in anhydrous ethanol (15 mL) was added, the ice bath was removed, and the mixture was stirred vigorously at room temperature for 2 h. The mixture was then filtered through celite and the filtrate was evaporated in vacuo. The resulting material was dissolved in EtOAc (50 mL), transferred to a separatory funnel, and washed with saturated aqueous $NaHCO_3$

(100 mL). The layers were separated and the aqueous layer was washed with EtOAc (2 x 50 mL). The combined organic layers were then washed with brine (30 mL), dried over anhydrous sodium sulfate, and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel using hexanes/Et_2O as the eluant to afford 1.12 g (73%) of the title compound as a red oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.02 (d, *J* = 7.8 Hz, 1 H), 6.91 (t, *J* = 7.8 Hz, 1 H), 6.76–6.71 (m, 2 H), 5.03 (s, 1 H), 4.90 (s, 1 H), 4.00 (s, br, 2 H), 3.35 (s, 2 H), 2.58 (s, 3 H), 1.78 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 143.2, 141.8, 140.4, 124.4, 120.5, 118.6, 115.3, 112.5, 62.6, 40.5, 20.6; IR (film) 3441, 2970, 1607, 1499 cm^{-1}.

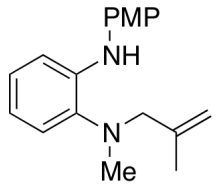


***N*^{1}-methyl-*N*^{1}-(2-phenylallyl)benzene-1,2-diamine (S4b).** Following the above procedure, zinc dust (5.99 g, 91.7 mmol), acetic acid (5.30 mL, 91.7 mmol), and *N*-methyl-2-nitro-*N*-(2-phenylallyl)aniline (1.64 g, 6.11 mmol) were reacted to afford 802 mg (55%) of the title compound as a red oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 7.6 Hz, 2 H), 7.35–7.25 (m, 3 H), 7.08 (d, *J* = 7.8 Hz, 1 H), 6.93 (t, *J* = 7.6 Hz, 1 H), 6.75 (t, *J* = 7.8 Hz, 1 H), 6.70 (d, *J* = 7.8 Hz, 1 H), 5.46 (s, 1 H), 5.39 (s, 1 H), 3.88 (s, 2 H), 3.78 (s, br, 2 H), 2.60 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.6, 142.1, 140.1, 139.9, 128.2, 127.6, 126.5, 124.6, 120.9, 118.3, 115.1, 114.6, 60.4, 40.6; IR (film) 3439, 3347, 1606, 1499 cm^{-1}.

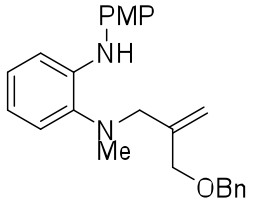


***N*^{1}-{2-[(Benzyloxy)methyl]allyl}-*N*^{1}-methylbenzene-1,2-diamine (S4c).** Following the above procedure, zinc dust (2.55 g, 39 mmol), acetic acid (2.25 mL, 39 mmol), and *N*-

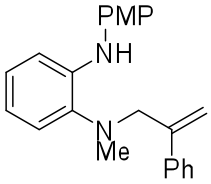
{2-[(benzyloxy)methyl]allyl}-*N*-methyl-2-nitroaniline (810 mg, 2.60 mmol) were reacted to afford 372 mg (50%) of the title compound as a red oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.36–7.32 (m, 4 H), 7.30–7.27 (m, 1 H), 7.01 (d, *J* = 7.7 Hz, 1 H), 6.89 (t, *J* = 7.5 Hz, 1 H), 6.70 (t, *J* = 7.7 Hz, 1 H), 6.67 (d, *J* = 7.8 Hz, 1 H), 5.26 (s, 1 H), 5.22 (s, 1 H), 4.50 (s, 2 H), 4.09 (s, 2 H), 4.03 (s, br, 2 H), 3.49 (s, 2 H), 2.59 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 143.4, 141.8, 139.6, 138.2, 128.4, 127.8, 127.7, 124.4, 120.3, 118.1, 115.2, 115.1, 72.0, 71.5, 58.8, 40.4; IR (film) 2920,1604, 1494 cm^{-1}.



***N*^{1}-(4-Methoxyphenyl)-*N*^{2}-methyl-*N*^{2}-(2-methylallyl)benzene-1,2-diamine (3a).** General Procedure C was employed for the coupling of 4-bromoanisole (0.80 mL, 6.35 mmol) and *N*^{1}-methyl-*N*^{1}-(2-methylallyl)benzene-1,2-diamine (1.12 g, 6.35 mmol), except using a catalyst composed of $Pd_2(dba)_3$ (116 mg, 0.127 mmol, 2.0 mol %) and JohnPhos (75 mg, 0.250 mmol, 4.0 mol %) and a reaction temperature of 85 °C. This procedure afforded 1.49 g (79%) of the title compound as a red oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.16–7.10 (m, 4 H), 6.98 (t, *J* = 7.3 Hz, 1 H), 6.89 (d, *J* = 7.5 Hz, 2 H), 6.81 (t, *J* = 7.3 Hz, 1 H), 6.57 (s, br, 1 H), 5.07 (s, 1 H), 4.95 (s, 1 H), 3.83 (s, 3 H), 3.39 (s, 2 H), 2.65 (s, 3 H), 1.81 (s, 3 H); ^{13}C NMR (175 MHz $CDCl_3$) δ 155.1, 143.1, 141.2, 140.2, 136.2, 124.5, 122.0, 120.7, 118.9, 114.8, 113.0, 112.7, 63.2, 55.7, 40.8, 20.7; IR (film) 3355, 2933, 1510 cm^{-1}; MS (ESI+) 283.1807 (283.1805 calcd for $C_{18}H_{22}N_2O$, M + H^{+}).

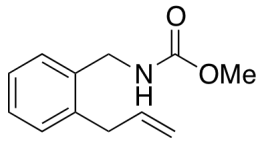


***N*^{1}-{2-[(Benzyloxy)methyl]allyl}-*N*^{2}-(4-methoxyphenyl)-*N*^{1}-methylbenzene-1,2-diamine (3b):** General Procedure C was employed for the coupling of 4-bromoanisole (0.165 mL, 1.32 mmol) and *N*^{1}-{2-[(benzyloxy)methyl]allyl}-*N*^{1}-methylbenzene-1,2-diamine (372 mg, 1.32 mmol), except using a catalyst composed of $Pd_2(dba)_3$ (26 mg, 0.0264 mmol, 2.0 mol %) and JohnPhos (16 mg, 0.528 mmol, 4.0 mol %) and a reaction temperature of 85 °C. This procedure afforded 380 mg (75%) of the title compound as a red oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.32–7.26 (m, 4 H), 7.13 (d, *J* = 7.7 Hz, 1 H), 7.09 (d, *J* = 8.0 Hz, 1 H), 7.06 (d, *J* = 8.7 Hz, 2 H), 6.96 (t, *J* = 7.8 Hz, 1 H), 6.84 (d, *J* = 8.9 Hz, 2 H), 6.78 (t, *J* = 7.7 Hz, 1 H), 6.73 (s, br, 1 H), 5.29 (s, 1 H), 4.44 (s, 2 H), 4.09 (s, 2 H), 3.80 (s, 3 H), 3.53 (s, 2 H), 2.65 (s, 3 H); ^{13}C NMR (175 MHz $CDCl_3$) δ 155.0, 143.2, 140.4, 140.2, 138.1, 135.9, 129.4, 127.8, 127.6, 124.4, 122.5, 120.5, 118.4, 115.5, 114.5, 112.7, 71.7, 71.4, 59.4, 55.6, 40.7; IR (film) 3338, 2946, 1509 cm^{-1}; MS (ESI+) 389.2226 (389.2224 calcd for $C_{25}H_{28}N_2O_2$, M + H$^+$).



***N*^{1}-(4-Methoxyphenyl)-*N*^{2}-methyl-*N*^{2}-(2-phenylallyl)benzene-1,2-diamine (3c).** General Procedure C was employed for the coupling of 4-bromoanisole (0.41 mL, 3.3 mmol) and *N*^{1}-methyl-*N*^{1}-(2-phenylallyl)benzene-1,2-diamine (790 mg, 3.3 mmol), except using a catalyst composed of $Pd_2(dba)_3$ (60 mg, 0.066 mmol, 2.0 mol %) and JohnPhos (40 mg, 0.132 mmol, 4.0 mol %) and a reaction temperature of 85 °C. This procedure afforded 750 mg (66%) of the title compound as a red oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57–7.52 (m, 2 H), 7.46–7.40 (m, 3 H), 7.30 (d, *J* = 7.8 Hz, 1 H), 7.24 (d, *J* =

8.0 Hz, 1 H), 7.12 (t, *J* = 7.4 Hz, 1 H), 7.03 (d, *J* = 8.8 Hz, 2 H), 6.98–6.89 (m, 3 H), 6.41 (s, br, 1 H) 5.56 (s, 1 H), 5.48 (s, 1 H), 4.03 (s, 2 H), 3.89 (s, 3 H), 2.73 (s, 3 H); ^{13}C NMR (100 MHz $CDCl_3$) δ 154.8, 145.8, 140.5, 140.4, 139.9, 135.6, 128.3, 127.5, 126.5, 124.8, 121.8, 121.3, 118.4, 115.2, 114.4, 112.2, 61.0, 55.4, 41.0; IR (film) 3347, 2947, 1510 cm^{-1}; MS (ESI+) 239.1537 (239.1543 calcd for $C_{23}H_{24}N_2O$, M + H^+).

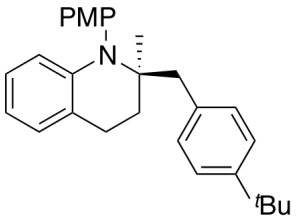


Methyl (2-allylbenzyl)carbamate (5). A flame dried round bottom flask equipped with a stir bar was cooled under a stream of nitrogen and charged with ether (20 mL) and $LiAlH_4$ (39 mL, 39 mmol, 1.0 M in ether). The mixture was cooled to 0 °C, stirred for five min, then a solution of 2-allylbenzonitrile[1] (2.80 g, 19.5 mmol) in ether (15 mL) was added slowly dropwise. The reaction mixture was stirred for 1.5 h at 0 °C and then was slowly quenched with 1.5 mL H_2O, 1.5 mL 15% NaOH and 3.0 mL H_2O. The resulting mixture was stirred at rt for 20 min, then the salts were filtered off through a fritted funnel. The filtrate was dried over anhydrous $MgSO_4$ and concentrated in vacuo to afford 2-allylbenzylamine, which was used without further purification.

The crude 2-allylbenzylamine product from above was dissolved in dichloromethane (60 mL) and added to a flame dried round bottom flask equipped with a stir bar. Solid K_2CO_3 (2.95 g, 21.3 mmol) was added to the flask and the resulting mixture was cooled to 0 °C. Methyl chloroformate (1.0 equiv., 1.5 mL) was then slowly added, and the resulting mixture was warmed to room temperature and stirred for 16 h. The reaction mixture was filtered, and the filtrate was concentrated in vacuo. The crude product was purified by flash chromatography on silica gel using hexanes/Et_2O as the eluent to afford 3.18 g (79%) as a clear oil. ^{1}H NMR (500 MHz, d8-toluene, 90 °C) δ 7.07 (d, *J* = 6.6 Hz, 1 H), 7.02–6.93 (m, 3 H), 5.82–7.72 (m, 1 H), 4.91 (d, *J* = 10.0 Hz, 1 H); 4.85 (d, *J* = 16.9 Hz, 1 H), 4.58 (s, br, 1 H), 4.16 (d, *J* = 5.9 Hz, 2 H), 3.42 (s, 3 H), 3.18 (d, *J* = 6.1 Hz, 2 H); ^{13}C NMR (125 MHz, d8-toluene, 90 °C) δ 157.7, 139.2, 138.5, 138.2, 131.2, 130.0,

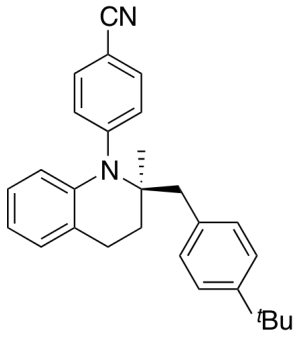
128.8, 127.9, 116.7, 52.6, 44.1, 38.0; IR (film) 3326, 2949, 1702, 1527 cm^{-1}; MS (CI+) 206.1175 (206.1176 calcd for $C_{12}H_{15}NO_2$, M + H^+)

General Procedure D: Asymmetric Pd-catalyzed carboamination reactions. A flame dried Schlenk flask equipped with a stir bar was cooled under a stream of nitrogen and charged with $Pd_2(dba)_3$ (2 mol %), (S)-Siphos-PE (6 mol %), the aryl or alkenyl halide (1.0–2.0 equiv.), NaO^{t}Bu (1.3–2.0 equiv.), and the amino alkene substrate. The flask was purged with nitrogen, and toluene (0.1 M) was added (xylenes was used as solvent in cases where reactions were heated over 110 °C). The resulting mixture was heated to 80–125 °C with stirring for 2–15 hrs. The reaction mixture was then cooled to rt, saturated aqueous ammonium chloride (6 mL/mmol) was added, and the mixture was transferred to a separatory funnel. The mixture was extracted with ethyl acetate and the combined organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated in vacuo. The crude product was then purified by flash chromatography on silica gel using hexanes/Et_2O as the eluant.



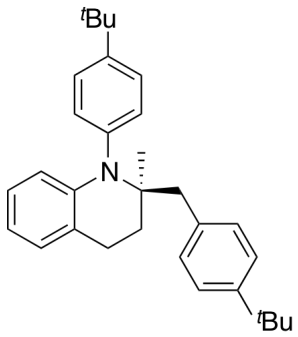
(*R*)-(+)-2-[4-(*tert*-Butyl)benzyl]-1-(4-methoxyphenyl)-2-methyl-1,2,3,4-tetrahydroquinoline (2b). General Procedure D was employed for the coupling of *N*-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (27 mg, 0.10 mmol) and 1-bromo-4-*tert*-butylbenzene (43 mg, 0.20 mmol) using NaO^{t}Bu (19 mg, 0.20 mmol) as the base and a reaction temperature of 110 °C for 14 h. This procedure afforded 35 mg (86%) of the title compound as a white solid, mp 47–50 °C. This material was judged to be 92:8 er by chiral HPLC analysis (Chiracel ADH, 25 cm x 4.6 mm, 0.5% IPA/Hexanes, 1 mL/min, λ 254 nm, RT= 6.1 and 7.3 min). $[\alpha]^{23}_D$ +50.6 (*c* 3.33, CH_2Cl_2); ^{1}H NMR (500

MHz, $CDCl_3$) δ 7.29 (d, *J* = 7.8 Hz, 2 H), 7.13–7.05 (m, 4 H), 7.04–6.91 (m, 3 H), 6.87 (t, *J* = 7.6 Hz, 1 H), 6.62 (t, *J* = 7.1 Hz, 1 H), 6.04 (d, *J* = 8.3 Hz, 1 H), 3.87 (s, 3 H), 3.11 (ddd, *J* = 5.6, 9.6, 15.9 Hz, 1 H), 2.95–2.82 (m, 3 H), 1.96 (dt, *J* = 5.9, 12.3 Hz, 1 H), 1.82–1.76 (m, 1 H), 1.33 (s, 9 H), 1.11 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.2, 149.0, 146.4, 136.2, 135.2, 133.4, 130.5, 129.1, 126.5, 124.8, 120.9, 116.0, 115.1, 114.8, 114.4, 57.4, 55.4, 44.6, 34.4, 32.3, 31.4, 25.9, 24.5 (an extra peak at 114.4 is present due to apparent slow bond rotation); IR (film) 2961, 1603, 1507 cm^{-1}; MS (ESI+) 400.2632 (400.2635 calcd for $C_{28}H_{33}NO$, M + H^+).

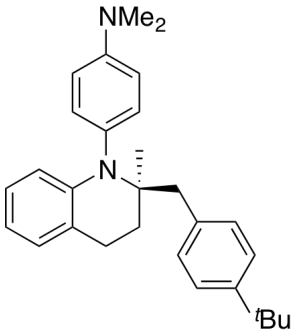


(*R*)-(+)-4-{2-[4-(*tert*-Butyl)benzyl]-2-methyl-3,4-dihydroquinolin-1(2*H*)-yl}benzonitrile (2c). General Procedure D was employed for the coupling of 4-{[2-(3-methylbut-3-en-1-yl)phenyl]amino}benzonitrile (26 mg, 0.10 mmol,) and 1-bromo-4-*tert*-butylbenzene (43 mg, 0.20 mmol) using NaO^tBu (19 mg, 0.20 mmol) as the base and a reaction temperature 110 °C for 14 h. This procedure afforded 20 mg (51%) of the title compound as an orange oil. This material was judged to be 62:38 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 1% IPA/Hexanes, 1 mL/min, λ 254 nm, RT= 5.6 and 8.0 min). $[\alpha]^{23}_D$ +50.3 (*c* 0.68, CH_2Cl_2); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.7 Hz, 2 H), 7.28 (d, *J* = 8.2 Hz, 2 H), 7.11 (d, *J* = 7.5 Hz, 1 H), 7.02 (d, *J* = 8.2 Hz, 2 H), 6.91 (t, *J* = 8.0 Hz, 1 H), 6.88–6.78 (m, 2 H), 6.73 (t, *J* = 7.3 Hz, 1 H), 6.15 (d, *J* = 8.2 Hz, 1 H), 3.09 (ddd, *J* = 6.3, 11.2, 17.3 Hz, 1 H), 2.93–2.86 (m, 2 H), 2.66 (d, *J* = 13.1 Hz, 1 H), 1.95 (ddd, *J* = 4.2, 6.3, 13.3 Hz, 1 H), 1.84 (ddd, *J* = 6.0, 11.2, 13.2 Hz, 1 H), 1.31 (s, 9 H), 1.05 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 150.0, 149.7, 145.0, 134.8, 133.1, 132.3, 130.7, 129.7, 126.8, 125.1,123.1, 119.1, 118.6, 118.5, 109.0, 58.2,

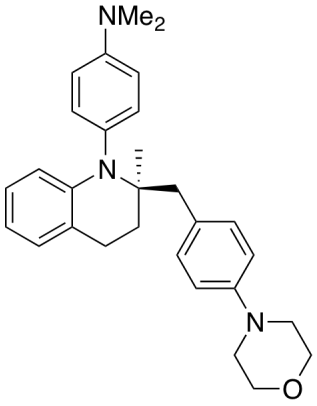
43.4, 34.6, 33.7, 31.6, 26.7, 24.2; IR (film) 2962, 1596, 1500 cm^{-1}; MS (ESI+) 395.2472 (395.2482 calcd for $C_{28}H_{30}N_2$, M + H^+).



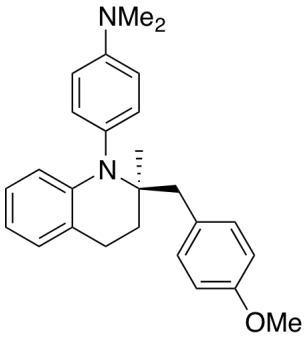
(*R*)-(+)-2-[4-(*tert*-Butyl)benzyl]-1-[4-(tert-butyl)phenyl]-2-methyl-1,2,3,4-tetrahydroquinoline (2d). General Procedure D was employed for the coupling of *N*-[4-(*tert*-butyl)phenyl]-2-(3-methylbut-3-en-1-yl)aniline (29 mg, 0.10 mmol) and 1-bromo-4-*tert*-butylbenzene (43 mg, 0.20 mmol) using NaO^{t}Bu (19 mg, 0.20 mmol) as the base and a reaction temperature of 110 °C for 14 h. This procedure afforded 40 mg (93%) of the title compound as a pale yellow solid, mp 59–63 °C. This material was judged to be 87:13 er by chiral HPLC analysis (LuxAmylose, 25 cm x 4.6 mm, 1% IPA/Hexanes, 0.3 mL/min, λ 254nm, RT= 12.0 and 12.5 min). $[\alpha]^{23}_D$ +40.3 (*c* 2.30, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.1 Hz, 2 H), 7.29 (d, *J* = 8.3 Hz, 2 H), 7.12–6.98 (m, 5 H), 6.87 (t, *J* = 7.3 Hz, 1 H), 6.62 (t, *J* = 7.1 Hz, 1 H), 6.04 (d, *J* = 8.3 Hz, 1 H), 3.12 (ddt, *J* = 5.6, 11.1, 15.5 Hz, 1 H), 2.94–2.81 (m, 3 H), 1.99–1.92 (m, 1 H), 1.80 (ddd, *J* = 5.7, 9.6, 13.2 Hz, 1 H), 1.39 (s, 9 H), 1.33 (s, 9 H), 1.11 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.7, 149.1, 146.4, 140.9, 135.4, 132.0, 130.7, 129.3, 126.6, 126.4, 124.9, 121.0, 116.2, 115.5, 57.5, 44.8, 34.8, 34.6, 32.5, 31.7, 31.6, 26.1, 24.7; IR (film) 2962, 1600, and 1507 cm^{-1}; MS (ESI+) 426.3174 (426.3155 calcd for $C_{31}H_{39}N$, M + H^+).



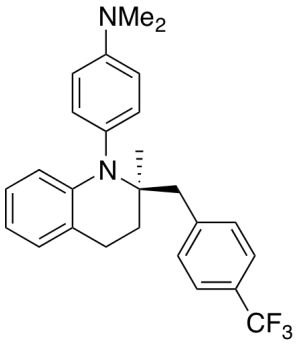
(*R*)-(+)-4-{2-[4-(*tert*-Butyl)benzyl]-2-methyl-3,4-dihydroquinolin-1(2*H*)-yl)-*N*,*N*-dimethylaniline (2e). General Procedure D was employed for the coupling of N^1,N^1-dimethyl-N^4-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 1-bromo-4-*tert*-butylbenzene (85 mg, 0.40 mmol), using NaO^{t}Bu (38 mg, 0.40 mmol,) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 80 mg (95%) of the title compound as a white solid, mp 144–147 °C. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ADH, 25 cm x 4.6 mm, 1.2% IPA/Hexanes, 2 mL/min, λ 254 nm, RT = 3.6 and 5.0 min). $[\alpha]^{23}_{D}$ +31.8 (*c* 1.75, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.1 Hz, 2 H), 7.15–7.01 (m, 5 H), 6.90 (t, *J* = 7.1 Hz, 1 H), 6.85–6.78 (m, 2 H), 6.63 (t, *J* = 7.1 Hz, 1 H), 6.12 (d, *J* = 8.3 Hz, 1 H), 3.20–3.09 (m, 1 H), 3.08–2.94 (m, 7 H), 2.92–2.83 (m, 2 H), 1.98 (m, 1 H), 1.82 (ddd, *J* = 5.3, 9.6, 13.0 Hz, 1 H), 1.36 (s, 9 H), 1.15 (s, 3 H); ^{13}C NMR (125 MHz $CDCl_3$) δ 149.3, 149.0, 147.0, 135.5, 133.2, 132.3, 130.6, 129.1, 126.6, 124.9, 120.8, 115.7, 115.0, 113.4, 113.1, 57.6, 44.9, 40.8, 34.5, 32.4, 31.6, 26.0, 24.7 (an extra peak at 113.1 is present due to apparent slow bond rotation); IR (film) 2961, 1609, 1516 cm^{-1}; MS (ESI+) 413.2955 (413.2951 calcd for $C_{29}H_{36}N_2$, M + H$^+$).



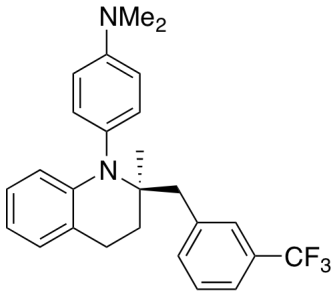
(*R*)-(+)-*N*,*N*-Dimethyl-4-[2-methyl-2-(4-morpholinobenzyl)-3,4-dihydroquinolin-1(2*H*)-yl]aniline (2f). General Procedure D was employed for the coupling of N^1,N^1-dimethyl-N^4-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 4-(4-bromophenyl)morpholine (97 mg, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 77 mg (87%) of the title compound as a yellow solid, mp 158–161 °C. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ADH, 25 cm x 4.6 mm, 1% IPA/Hexanes, 2 mL/min, λ 254 nm, RT = 14.6 and 20.1 min). $[\alpha]^{23}_{D}$ +24.7 (*c* 1.10, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.13–7.03 (m, 5 H), 6.92–6.77 (m, 5 H), 6.61 (t, *J* = 7.2 Hz, 1 H), 6.10 (d, *J* = 8.3 Hz, 1 H), 3.92–3.86 (m, 4 H), 3.20–3.14 (m, 4 H), 3.10–3.04 (m, 1 H), 3.02 (s, 6 H), 2.92–2.79 (m, 3 H), 1.95 (m, 1 H), 1.78 (ddd, *J* = 5.5, 9.5, 13.2 Hz, 1 H), 1.12 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.7, 149.1, 147.0, 133.2, 133.1, 132.2, 131.6, 130.1, 129.1, 126.6, 120.8, 115.7, 115.3, 115.0, 113.3, 113.0, 67.1, 57.6, 49.6, 44.5, 40.8, 32.3, 25.9, 24.7 (extra peaks at 133.1 and 113.0 are present due to apparent slow bond rotation); IR (film) 2963, 1609, 1514 cm^{-1}; MS (ESI+) 442.285 (442.28 calcd for $C_{29}H_{35}N_3O$, M + H$^+$).



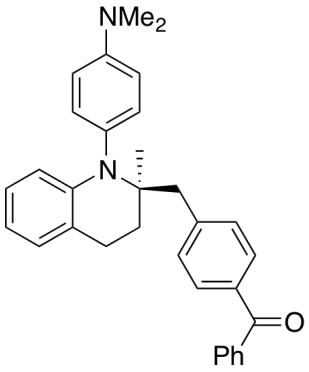
(*R*)-(+)-4-[2-(4-Methoxybenzyl)-2-methyl-3,4-dihydroquinolin-1(2*H*)-yl]-*N,N*-dimethylaniline (2g). General Procedure D was employed for the coupling of N^1,N^1-dimethyl-N^4-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 4-bromoanisole (75 mg, 0.40 mmol), using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 68 mg (88%) of the title compound as a yellow solid, mp 101–105 °C. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ADH, 25 cm x 4.6 mm, 1% IPA/Hexanes, 1 mL/min, λ 254 nm, RT= 10.2 and 15.1 min). $[\alpha]^{23}_{D}$ +30.6 (*c* 2.10, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.13–7.01 (m, 5 H), 6.90 (t, *J* = 7.3 Hz, 1 H), 6.85–6.77 (m, 4 H), 6.62 (t, *J* = 7.4 Hz, 1 H), 6.10 (d, *J* = 8.1 Hz, 1 H), 3.82 (s, 3 H), 3.14–3.05 (m, 1 H), 3.02 (s, 6 H), 2.92–2.80 (m, 3 H), 1.94 (dt, *J* = 6.0, 12.5 Hz, 1 H), 1.79 (ddd, *J* = 5.5, 9.4, 13.3 Hz, 1 H), 1.11 (s, 3 H); ^{13}C NMR (125 MHz $CDCl_3$) δ 158.1, 149.2, 146.9, 133.2, 133.0, 132.3, 131.8, 130.7, 129.1, 126.6, 120.8, 115.8, 115.1, 113.4, 113.3, 113.0, 57.5, 55.3, 44.5, 40.8, 32.3, 25.8, 24.6 (extra peaks at 133.0 and 113.0 are present due to apparent slow bond rotation); IR (film) 2962, 1609, 1512 cm^{-1}; MS (ESI+) 387.2432 (387.2431 calcd for $C_{26}H_{30}N_2O$, M + H$^+$).



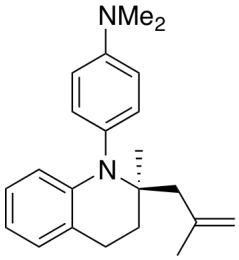
(*R*)-(+)-*N,N*-Dimethyl-4-{2-methyl-2-[4-(trifluoromethyl)benzyl]-3,4-dihydroquinolin-1(2*H*)-yl}aniline (2h). General Procedure D was employed for the coupling of N^1,N^1-dimethyl-N^4-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 4-bromobenzotrifluoride (90.0 mg, 0.40 mmol), using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 75.6 mg (89%) of the title compound as a white solid, mp 127-130 °C. This material was judged to be 94:6 er by chiral HPLC analysis (Chiracel ADH, 25 cm x 4.6 mm, 1% IPA/Hexanes, 1 mL/min, λ 254 nm, RT= 6.1 and 8.9 min). $[\alpha]^{23}_{D}$ +31.5 (*c* 1.43, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.0 Hz, 2 H), 7.30 (d, *J* = 8.0 Hz, 2 H), 7.14–6.97 (m, 3 H), 6.91 (t, *J* = 7.7 Hz, 1 H), 6.86–6.76 (m, 2 H), 6.65 (t, *J* = 7.1 Hz, 1 H), 6.14 (d, *J* = 8.2 Hz, 1 H), 3.15–2.85 (m, 10 H), 1.95 (dt, *J* = 6.0, 12.4 Hz, 1 H), 1.79 (ddd, *J* = 5.6, 9.5, 13.1, Hz, 1 H), 1.13 (s, 3 H); ^{13}C NMR (125 MHz $CDCl_3$) δ 149.4, 146.8, 143.0, 133.0, 132.2, 131.2, 129.2, 128.6 (q, *J* = 32 Hz), 126.8, 124.6 (q, *J* = 270 Hz), 124.9 (q, *J* = 3 Hz), 120.8, 116.2, 115.6, 113.4, 113.0, 57.3, 45.2, 40.6, 32.4, 25.7, 24.4 (an extra peak at 113.0 is present due to apparent slow bond rotation); IR (film) 2971, 1610, 1517 cm^{-1}; MS (ESI+) 425.2202 (425.2199 calcd for $C_{26}H_{27}F_3N_2$, M + H$^+$).



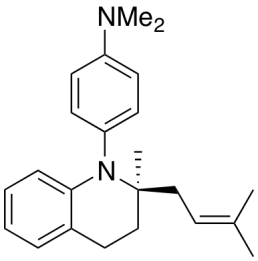
(*R*)-(+)-*N*,*N*-Dimethyl-4-{2-methyl-2-[3-(trifluoromethyl)benzyl]-3,4-dihydroquinolin-1(2*H*)-yl}aniline (2i). General Procedure D was employed for the coupling of N^1,N^1-dimethyl-N^4-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 3-bromobenzotrifluoride (90 mg, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 80 mg (94%) of the title compound as a brown solid, mp 91–94 °C. This material was judged to be 92:8 er by chiral HPLC analysis (Chiracel ADH, 25 cm x 4.6 mm, 1 % IPA/Hexanes, 1 mL/min, λ 254 nm, RT= 4.2 and 5.4 min). $[\alpha]^{23}_{D}$ +22.5 (*c* 1.35, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 (d, *J* = 7.3 Hz, 1 H), 7.46–7.35 (m, 3 H), 7.12–6.93 (m, 3 H), 6.91 (t, *J* = 7.5 Hz, 1 H), 6.85–6.75 (m, 2 H), 6.65 (t, *J* = 7.3 Hz, 1 H), 6.15 (d, *J* = 8.3 Hz, 1 H), 3.15–3.02 (m, 7 H), 3.00–2.97 (m, 2 H), 3.93–3.85 (m, 1 H), 1.95 (dt, *J* = 5.9, 13.9 Hz, 1 H), 1.82 (ddd, *J* = 5.50, 9.40, 13.30 Hz, 1 H), 1.12 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.4, 146.8, 139.7, 134.3, 133.0, 132.9, 132.3, 130.4 (q, *J* = 31 Hz), 129.2, 128.5, 127.5 (q, *J* = 4 Hz), 126.8, 124.4 (q, *J* = 270 Hz), 123.2 (q, *J*= 4 Hz), 120.8, 116.3, 115.7, 113.4, 112.9, 57.3, 45.3, 40.8, 32.6, 25.8, 24.6 (extra peaks at 132.9 and 112.9 are present due to apparent slow bond rotation); IR (film) 2971, 1609, 1518 cm^{-1}; MS (ESI+) 425.22 (425.2199 calcd for $C_{26}H_{27}F_3N_2$, M + H$^+$).



(*R*)-(+)-[4-({1-[4-(Dimethylamino)phenyl]-2-methyl-1,2,3,4-tetrahydroquinolin-2-yl}methyl)phenyl](phenyl)methanone (2j). General Procedure D was employed for the coupling of N^1,N^1-dimethyl-N^4-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 4-bromobenzophenone (104 mg, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 76 mg (82%, 93% pure) of the title compound as a yellow solid, mp 156–159 °C. This material was judged to be 96:4 er by chiral HPLC analysis (Chiracel ADH, 25 cm x 4.6 mm, 5 % IPA/Hexanes, 1 mL/min, λ 254 nm, RT= 11.6 and 25.8 min). $[\alpha]^{23}_{D}$ +32.7 (*c* 1.10, CH_2Cl_2); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.83 (d, *J* = 7.5 Hz, 2 H), 7.76 (d, *J* = 8.0 Hz, 2 H), 7.61 (t, *J* = 7.4 Hz, 1 H), 7.51 (t, *J* = 7.6 Hz, 2 H), 7.30 (d, *J* = 8.0 Hz, 2 H), 7.10–6.98 (m, 3 H), 6.91 (t, *J* = 7.7 Hz, 1 H), 6.79 (dd, *J* = 8.5, 19.0 Hz, 2 H), 6.63 (t, *J* = 7.1 Hz, 1 H), 6.13 (d, *J* = 8.3 Hz, 1 H), 3.15–3.08 (m, 1 H), 3.06–3.00 (m, 7 H), 2.99 (d, *J* = 12.8 Hz, 1 H), 2.89 (dt, *J* = 5.9, 16.6 Hz, 1 H), 1.98 (dt, *J* = 5.9, 12.4 Hz, 1 H), 1.83 (ddd, *J* = 5.5, 9.6, 13.2 Hz, 1 H), 1.15 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 196.4, 149.2, 146.6, 143.8, 137.8, 135.4, 132.8, 132.2, 132.0, 130.7, 130.0, 129.8, 129.0, 128.2, 126.6, 120.6, 116.0, 115.3, 113.2, 112.8, 57.4, 45.3, 40.6, 32.5, 25.9, 24.4 (2 extra peaks in the arene region are present due to apparent slow bond rotation); IR (film) 2927, 1656, 1517 cm^{-1}; MS (ESI+) 461.2583 (461.2587 calcd for $C_{32}H_{32}N_2O$, M + H$^+$).

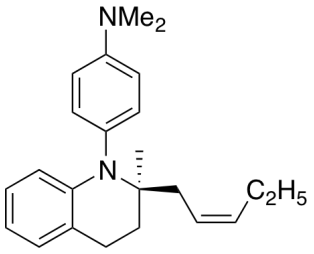


(*R*)-(+)-*N,N*-Dimethyl-4-[2-methyl-2-(2-methylallyl)-3,4-dihydroquinolin-1(2*H*)-yl]aniline (2k). General Procedure D was employed for the coupling of N^1,N^1-dimethyl-N^4-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 2-bromopropene (48 mg 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 55 mg (86%) of the title compound as an off-white solid, mp 76–79 °C. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ODH, 25 cm x 4.6 mm, 1% IPA/Hexanes, 0.2 mL/min, λ 254nm, RT = 13.4 and 15.2 min). $[\alpha]^{23}_{D}$ +37.7 (*c* 1.81, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.12–7.00 (m, 3 H), 6.85 (t, *J* = 7.1 Hz, 1 H), 6.79 (d, *J* = 8.8 Hz, 2 H), 6.58 (t, *J* = 7.3 Hz, 1 H), 6.04 (d, *J* = 8.3 Hz, 1 H), 4.92 (s, 1 H), 4.78 (s, 1 H), 3.06–2.98 (m, 7H), 2.85 (dt, *J* = 5.9, 16.5 Hz, 1 H), 2.50 (d, *J* = 13.1 Hz, 1 H), 2.29 (d, *J* = 13.0 Hz, 1 H), 2.07 (dt, *J* = 5.9, 13.1 Hz, 1 H), 1.93 (ddd, *J*= 5.5, 9.6, 13.2 Hz, 1 H), 1.80 (s, 3 H), 1.21 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.3, 146.8, 142.7, 133.1, 132.3, 129.1, 126.6, 120.8, 115.7, 115.5, 114.9, 113.3, 113.0, 57.2, 46.9, 40.8, 33.0, 27.1, 25.5, 24.7 (an extra peak at 113.0 is present due to apparent slow bond rotation); IR (film) 2969, 1609, 1517 cm^{-1}; MS (ESI+) 321.2329 (321.2325 calcd for $C_{22}H_{28}N_2$, M + H$^+$).



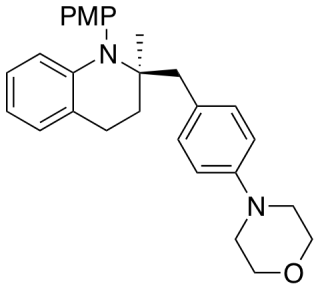
(*R*)-(+)-*N,N*-Dimethyl-4-[2-methyl-2-(3-methylbut-2-en-1-yl)-3,4-dihydroquinolin-1(2*H*)-yl]aniline (2l). General Procedure D was employed for the coupling of N^1,N^1-

dimethyl-*N*^{4}-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 1-bromo-2-methyl-1-propene (54 mg 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 66 mg (98%) of the title compound as an orange oil. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 0.9% IPA/Hexanes, 0.1 mL/min, λ 254 nm, RT = 26.1 and 28.2 min). $[\alpha]^{23}_{D}$ +53.4 (*c* 2.35 CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.12–6.96 (m, 3 H), 6.83 (t, *J* = 7.1 Hz, 1 H), 6.77 (d, *J* = 8.3Hz, 2 H), 6.55 (t, *J* = 7.1 Hz, 1 H), 6.02 (d, *J* = 8.3 Hz, 1 H), 5.19 (t, *J* = 7.3 Hz, 1 H), 3.01 (s, 3 H), 2.88–2.82 (m, 1 H), 2.32 (dd, *J*= 7.2, 14.4 Hz, 1 H), 2.20 (dd, *J* = 7.5, 14.4 Hz, 1 H), 2.00 (dt, *J* = 6.2, 12.9 Hz, 1 H), 1.79 (ddd, *J*= 5.9, 8.1, 13.5 Hz, 1 H), 1.72 (s, 3 H), 1.56 (s, 3 H), 1.22 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.3, 147.2, 133.6, 133.0, 132.3, 129.1, 126.6, 121.0, 120.3, 115.5, 114.8, 113.3, 57.6, 40.8, 37.9, 32.8, 26.3, 25.9, 24.7, 18.3; IR (film) 2967, 1608, 1516 cm^{-1}; MS (ESI+) 335.2136 (335.2482 calcd for $C_{23}H_{30}N_2$, M + H$^+$).

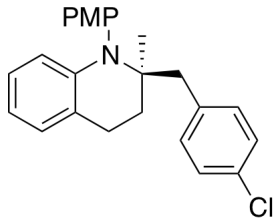


(*Z,R*)-(+)-*N,N*-Dimethyl-4-[2-methyl-2-(pent-2-en-1-yl)-3,4-dihydroquinolin-1(2*H*)-yl]aniline (2m). General Procedure D was employed for the coupling of *N*^{1},*N*^{1}-dimethyl-*N*^{4}-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and (Z)-1-bromo-1-butene (54 mg, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 61 mg (91%) of the title compound as an off-white solid, mp 122–125 °C. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 0.8% IPA/Hexanes, 0.1 mL/min, λ 254 nm, RT = 30.0 and 33.3 min). $[\alpha]^{23}_{D}$ +59.3 (*c* 1.50, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.16–6.93 (m, 3 H), 6.88 (t, *J* = 7.3 Hz, 1 H), 6.81 (d, *J* = 8.8 Hz, 2 H), 6.59 (t, *J* = 7.1 Hz, 1 H), 6.09 (d, *J* = 8.3 Hz, 1 H), 5.55–5.38 (m, 2

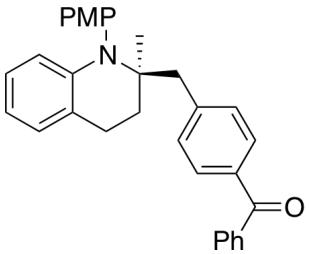
H), 3.04 (s, 6 H), 2.95–2.85 (m, 2 H), 2.45 (dd, *J* = 7.3, 14.4 Hz, 1 H), 2.30 (dd, *J* = 7.2, 14.4 Hz, 1 H), 2.12–1.92 (m, 3 H), 1.86 (ddd, *J* = 5.7, 8.4, 13.6 Hz, 1 H), 1.18 (s, 3 H), 0.98 (t, *J* = 7.6 Hz, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.3, 147.1, 134.0, 133.0, 132.2, 129.1, 126.6, 124.5, 120.9, 115.6, 114.8, 113.2, 57.2, 40.8, 36.9, 32.8, 26.0, 24.6, 21.0, 14.3; IR (film) 2964, 1609, 1517 cm^{-1}; MS (ESI+) 335.2495 (335.2482 calcd for $C_{23}H_{30}N_2$, M + H^+).



(*R*)-(+)-4-(4-{[1-(4-Methoxyphenyl)-2-methyl-1,2,3,4-tetrahydroquinolin-2-yl]methyl}phenyl)morpholine (2n). General Procedure D was employed for the coupling of *N*-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (54 mg, 0.20 mmol) and 4-(4-bromophenyl)morpholine (97 mg, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 90 °C for 14 h. This procedure afforded 70 mg (82%) of the title compound as a white solid, mp 62–65 °C. This material was judged to be 94:6 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 5% IPA/Hexanes, 1 mL/min, λ 254 nm, RT = 8.8 and 11.1 min). $[\alpha]^{23}_D$ +56.1 (*c* 1.11, CH_2Cl_2); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.14–7.05 (m, 5 H), 7.02–6.95 (m, 2 H), 6.88 (t, *J* = 7.2 Hz, 1 H), 6.84 (d, *J* = 8.3 Hz, 2 H), 6.63 (t, *J* = 7.2 Hz, 1 H), 6.05 (d, *J* = 8.4 Hz, 1 H), 3.92–3.84 (m, 7 H), 3.18–3.12 (m, 4H), 3.09 (ddd, *J* = 5.7, 9.6, 15.9 Hz, 1 H), 2.89–2.81 (m, 2 H), 2.78 (d, *J* = 13.2 Hz, 1 H), 1.94 (dt, *J* = 6.0, 12.5 Hz, 1 H), 1.78 (ddd, *J* = 5.5, 9.6, 13.2 Hz, 1 H), 1.09 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 158.2, 149.7, 146.6, 136.2, 133.6, 133.5, 131.6, 129.8, 129.2, 126.6, 121.0, 116.1, 115.3, 115.2, 114.9, 114.5, 67.1, 57.5, 55.5, 49.5, 44.4, 32.3, 25.8, 24.6 (extra peaks at 133.5 and 114.5 are present due to apparent slow bond rotation); IR (film) 2928, 1606, 1505 cm^{-1}; MS (ESI+) 429.2521 (429.2537 calcd for $C_{28}H_{32}N_2O_2$, M + H^+).

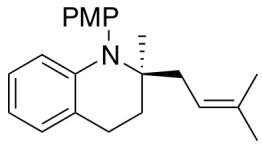


(*R*)-(+)-2-(4-Chlorobenzyl)-1-(4-methoxyphenyl)-2-methyl-1,2,3,4-tetrahydroquinoline (2o). General Procedure D was employed for the coupling of *N*-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (54 mg, 0.20 mmol) and 4-bromochlorobenzene (77 mg, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 90 °C for 14 h. This procedure afforded 62 mg (82%) of the title compound as a viscous oil. This material was judged to be 89:11 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 0.4% IPA/Hexanes, 1.1 mL/min, λ 254 nm, RT = 8.9 and 10.5 min). $[\alpha]^{23}_{D}$ +56.2 (*c* 0.97, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25 (d, *J* = 8.6 Hz, 2 H), 7.15–6.91 (m, 7 H), 6.89 (t, *J* = 7.3 Hz, 1 H), 6.65 (t, *J* = 7.3 Hz, 1 H), 6.08 (d, *J* = 8.3 Hz, 1 H), 3.87 (s, 3 H), 3.04 (ddd, *J* = 5.8, 9.6, 15.9 Hz, 1 H), 2.87–2.79 (m, 3 H), 1.90 (dt, *J* = 5.9, 13.4 Hz, 1 H), 1.77 (ddd, *J* = 5.6, 9.6, 13.3 Hz, 1 H), 1.08 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.3, 146.4, 136.9, 136.3, 133.4, 132.3, 132.2, 129.3, 128.2, 126.7, 121.0, 116.5, 115.7, 115.1, 114.5, 57.3, 55.6, 44.6, 32.5, 25.8, 24.5 (an extra peak at 114.5 is present due to apparent slow bond rotation); IR (film) 2928, 1604, 1505 cm^{-1}; MS (ESI+) 378.1620 (378.1619 calcd for $C_{24}H_{24}ClNO$, M + H$^+$).



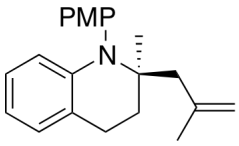
(*R*)-(+)-(4-{[1-(4-Methoxyphenyl)-2-methyl-1,2,3,4-tetrahydroquinolin-2-yl]methyl}phenyl)(phenyl)methanone (2p). General Procedure D was employed for the coupling of *N*-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (54 mg, 0.20

mmol) and 4-bromobenzophenone (104 mg, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 90 °C for 14 h. This procedure afforded 74 mg (83%) of the title compound as a white solid, mp 58–61 °C. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 8% IPA/Hexanes, 1 mL/min, λ 254 nm, RT = 10.4 and 16.5 min). $[\alpha]^{23}_{D}$ +44.7 (*c* 0.90, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 (d, *J* = 7.1 Hz, 2 H), 7.76 (d, *J* = 8.3 Hz, 2 H), 7.60 (t, *J* = 7.3 Hz, 1 H), 7.49 (t, *J* = 7.8 Hz, 2 H), 7.28 (d, *J* = 8.1 Hz, 2 H), 7.15–6.91 (m, 5 H), 6.89 (t, *J* = 7.1 Hz, 1 H), 6.65 (t, *J* = 6.4 Hz, 1 H), 6.08 (d, *J* = 8.1 Hz, 1 H), 3.87 (s, 3 H), 3.10 (ddd, *J*= 5.8, 9.7, 16.1 Hz, 1 H), 3.02–2.93 (m, 2 H), 2.87 (dt, *J* = 5.8, 16.7 Hz, 1 H), 1.95 (dt, *J* = 5.9, 13.2 Hz, 1 H), 1.82 (ddd, *J* = 5.6, 9.7, 13.2 Hz, 1 H), 1.13 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.5, 158.3, 146.4, 143.7, 137.9, 136.3, 135.7, 133.4, 132.4, 130.8, 130.1, 130.0, 129.3, 128.4, 126.8, 121.0, 116.6, 115.8, 115.1, 114.5, 57.5, 55.5, 45.3, 32.6, 26.1, 24.5 (an extra peak at 114.5 is present due to apparent slow bond rotation); IR (film) 2928, 1656, 1603, 1505 cm^{-1}; MS (ESI+) 448.2269 (448.2271 calcd for $C_{31}H_{29}NO_2$, M + H$^+$).

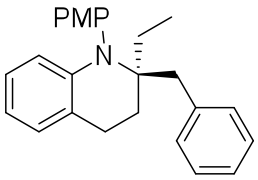


(*R*)-(+)-1-(4-Methoxyphenyl)-2-methyl-2-(3-methylbut-2-en-1-yl)-1,2,3,4-tetrahydroquinoline (2q). General Procedure D was employed for the coupling of *N*-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (54 mg, 0.20 mmol) and 1-Bromo-2-methyl-1-propene (41 µL, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 90 °C for 14 h. This procedure afforded 63 mg (98%) of the title compound as an orange oil. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 0.8% IPA/Hexanes, 0.2 mL/min, λ 254 nm, RT = 22.3 and 24.4 min). $[\alpha]^{23}_{D}$ +58.0 (*c* 1.11, CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.23–7.08 (m, 2 H), 7.07 (d, *J* = 7.3 Hz, 1 H), 6.99 (d, *J* = 9.1 Hz, 2 H), 6.87 (t, *J* = 7.3 Hz, 1 H), 6.61 (t, *J* = 7.4 Hz, 1 H), 6.02 (d, *J* = 8.3 Hz, 1 H), 5.23 (app. t, *J* = 7.1 Hz, 1 H), 3.88 (s, 3 H), 2.95–2.85 (m, 2 H), 2.34 (dd, *J* = 7.2, 14.3 Hz, 1 H), 2.22 (dd, *J* = 7.5,

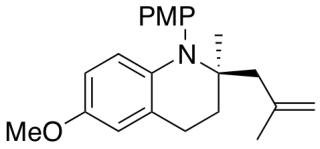
14.3 Hz, 1 H), 2.05 (dt, *J* = 6.4, 13.0 Hz, 1 H), 1.89–1.81 (m, 1 H), 1.77 (s, 3 H), 1.60 (s, 3H), 1.13 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.3, 146.8, 136.2, 133.8, 133.4, 129.2, 126.6, 121.1, 120.0, 115.9, 114.9, 114.7, 57.5, 55.5, 37.9, 32.8, 26.3, 25.9, 24.6, 18.3; IR (film) 2926, 1599, 1507 cm^{-1}; MS (ESI+) 322.2170 (322.2165 calcd for $C_{22}H_{27}NO$, $M+H^+$).



(*R*)-(+)-1-(4-Methoxyphenyl)-2-methyl-2-(2-methylallyl)-1,2,3,4-tetrahydroquinoline (2r). General Procedure D was employed for the coupling of *N*-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (54 mg, 0.20 mmol) and 2-Bromopropene (35 µL, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 90 °C for 14 h. This procedure afforded 52 mg (85%) of the title compound as an orange oil. This material was judged to be 96:4 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 0.8% IPA/Hexanes, 0.2 mL/min, λ 254 nm, RT = 22.9 and 23.9 min). $[\alpha]^{23}_D$ +49.8 (*c* 1.04, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22–7.00 (m, 3 H), 6.98 (d, *J* = 8.2 Hz, 2 H), 6.85 (t, *J* = 7.4 Hz, 1 H), 6.61 (t, *J* = 7.0 Hz, 1 H), 6.00 (d, *J* = 8.2 Hz, 1 H), 4.95 (s, 1 H), 4.77 (s, 1 H), 3.87 (s, 3 H), 3.02 (ddd, *J* = 5.5, 9.2, 15.7 Hz, 1 H), 2.85 (dt, *J* = 6.0, 16.8 Hz, 1 H), 2.48 (d, *J* = 13.1 Hz, 1 H), 2.27 (d, *J* = 13.2 Hz, 1 H), 2.07 (dt, *J* = 5.8, 12.3, Hz, 1 H), 1.93 (ddd, *J*= 5.7, 9.6, 14.0 Hz, 1 H), 1.81 (s, 3 H), 1.19 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.3, 146.4, 142.4, 136.2, 133.5, 129.2, 126.6, 121.0, 116.1, 115.7, 115.1, 114.8, 57.1, 55.5, 46.8, 32.9, 27.0, 25.4, 24.6; IR (film) 2928, 1599, and 1506 cm^{-1}; MS (ESI+) 308.2008 (308.2009 calcd for $C_{21}H_{25}NO$, $M + H^+$).

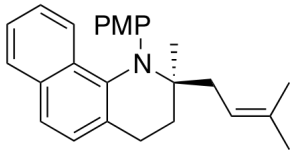


(*R*)-(+)-2-Benzyl-2-ethyl-1-(4-methoxyphenyl)-1,2,3,4-tetrahydroquinoline (2s). General Procedure D was employed for the coupling of *N*-(4-methoxyphenyl)-2-(3-methylenepentyl)aniline (28 mg, 0.10 mmol) and bromobenzene (21 µL, 0.20 mmol) using NaO^{t}Bu (19 mg, 0.20 mmol) as the base and a reaction temperature of 110 °C for 12 h. This procedure afforded 31 mg (86%) of the title compound as a viscous white oil. This material was judged to be 75:25 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 1.00% IPA/Hexanes, 0.75 mL/min, λ 254 nm, RT = 6.1 and 7.5 min). $[\alpha]^{23}_{D}$ +13.03 (*c* 1.35, CH_2Cl_2); ^{1}H NMR (700 MHz, C_6D_6) δ 7.26 (t, *J*= 7.2 Hz, 2 H), 7.22 (t, *J* = 7.2 Hz, 1 H), 7.18–7.09 (m, 3 H), 7.06 (d, *J* = 7.3 Hz, 1 H), 6.93–6.71 (m, 4 H), 6.62 (t, *J* = 7.3 Hz, 1 H), 6.11 (d, *J* = 8.3 Hz, 1 H), 3.82 (s, 3 H), 3.16 (ddd, *J* = 5.9, 11.5, 17.0 Hz, 1 H), 2.95 (d, *J* = 13.5 Hz, 1 H), 2.90–2.81 (m, 2 H), 1.95–1.83 (m, 2H), 1.54–1.47 (m, 1 H), 1.37 dq, *J* = 7.2, 14.5 Hz, 1 H) 0.88 (t, *J* = 7.3 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 157.8, 147.3, 138.5, 136.9, 133.1, 130.9, 128.9, 127.9, 126.4, 126.1, 122.1, 117.0, 116.4, 114.4, 60.7, 55.4, 42.2, 29.2, 27.8, 24.2, 8.5; IR (film) 2927, 1599, and 1507 cm^{-1}; MS (ESI+) 358.2163 (358.2165 calcd for $C_{25}H_{27}NO$, M + H^+).



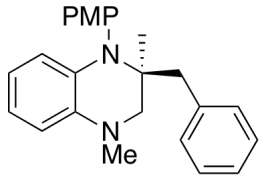
(*R*)-(+)-6-Methoxy-1-(4-methoxyphenyl)-2-methyl-2-(2-methylallyl)-1,2,3,4-tetrahydroquinoline (2t). General Procedure D was employed for the coupling of 4-methoxy-*N*-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (60 mg, 0.20 mmol) and 2-bromopropene (35 µL, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 90 °C for 14 h. This procedure afforded 55 mg (82%) of the title compound as an orange oil. This material was judged to be 96:4 er by chiral HPLC

analysis (lux-amylose, 15 cm x 4.6 mm, 2.5% IPA/Hexanes, 0.2 mL/min, λ 254 nm, RT = 29.7 and 32.9). $[\alpha]^{23}_{D}$ +68.3 (*c* 1.13, CH_2Cl_2); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.16–7.03 (m, 2 H), 6.93 (d, *J* = 7.7 Hz, 2 H), 6.65 (d, *J* = 2.9 Hz, 1 H), 6.47 (dd, *J* = 3.0, 8.9 Hz, 1 H), 5.98 (d, *J* = 9.1 Hz, 1 H), 4.92 (s, 1 H), 4.76 (s, 1 H), 3.84 (s, 3 H), 3.72 (s, 3 H), 2.98 (ddd, *J* = 5.8, 9.6, 16.0 Hz, 1 H), 2.83 (dt, *J* = 5.9,16.8 Hz, 1 H), 2.40 (d, *J* = 13.1 Hz, 1 H), 2.25 (d, *J* = 13.1 Hz, 1 H), 2.01 (dt, *J* = 6.0, 13.2 Hz, 1 H), 1.90 (ddd, *J* = 5.6, 9.6, 13.1 Hz, 1 H), 1.80 (s, 3 H), 1.14 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 157.9, 150.9, 142.5, 140.8, 137.2, 133.3, 122.2, 116.7, 115.3, 114.4, 114.2, 112.5, 56.7, 55.7, 55.4, 46.4, 32.9, 26.6, 25.3, 24.7; IR (film) 2933, 1493 cm^{-1}; MS (ESI+) 337.2032 (337.2036 calcd for $C_{22}H_{27}NO_2$, M^+).

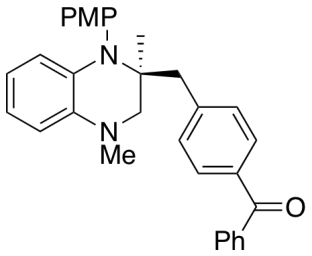


(*R*)-(+)-1-(4-Methoxyphenyl)-2-methyl-2-(3-methylbut-2-en-1-yl)-1,2,3,4-tetrahydrobenzo[h]quinoline (2u). General Procedure D was employed for the coupling of *N*-(4-Methoxyphenyl)-2-(3-methylbut-3-en-1-yl)naphthalen-1-amine (63 mg, 0.20 mmol) and 1-Bromo-2-methyl-1-propene (41 µL, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 95 °C for 18 h. This procedure afforded 47 mg (63%) of the title compound as a clear oil. This material was judged to be 93:7 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 0.8% IPA/Hexanes, 0.150 mL/min, λ 254 nm, RT = 38.1 and 40.9 min). $[\alpha]^{23}_{D}$ +281.2 (*c* 1.67, CH_2Cl_2); ^{1}H NMR (500 MHz, C_6D_6) δ 8.34 (d, *J* = 8.5 Hz, 1 H), 7.56 (d, *J* = 8.0 Hz, 1 H), 7.41 (d, *J* = 8.3 Hz, 1 H), 7.18-7.00 (m, 3 H), 6.95 (d, *J* = 8.2 Hz, 2 H), 6.51 (d, *J* = 8.3 Hz, 2 H), 5.48-5.43 (m, 1 H), 3.14 (s, 3 H), 2.85-2.76 (m, 2H), 2.44 (dd, *J* = 7.0, 14.6 Hz, 1 H), 2.12 (dd, *J* = 7.6, 14.6 Hz, 1 H), 1.78 (dt, *J* = 9.0, 13.5 Hz, 1 H), 1.66 (s, 3 H), 1.52-1.44 (m, 1H), 1.35 (s, 3 H), 1.07 (s, 3 H); ^{13}C NMR (125 MHz, C_6D_6) δ 157.2, 143.2, 142.4, 134.9, 133.3, 131.6, 131.3, 128.0, 126.4, 125.8, 125.7, 125.4, 123.8, 121.9, 114.1, 58.9, 55.0, 37.7, 28.7, 27.4, 26.6, 25.9, 18.4 (one peak missing from arene region due to

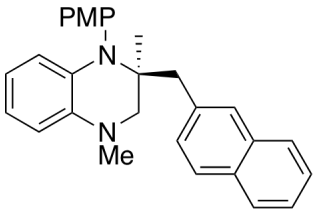
apparent overlap); IR (film) 2926, 1502, and 1390 cm^{-1}; MS (ESI+) 372.2326 (372.2322 calcd for $C_{26}H_{29}NO$, M + H^+).



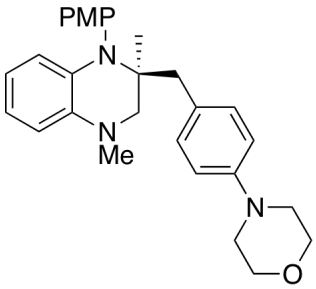
(*S*)-(–)-2-Benzyl-1-(4-methoxyphenyl)-2,4-dimethyl-1,2,3,4-tetrahydroquinoxaline (4a). General Procedure D was employed for the coupling of *N*^{1}-(4-methoxyphenyl)-*N*^{2}-methyl-*N*^{2}-(2-methylallyl)benzene-1,2-diamine (59 mg, 0.20 mmol) and bromobenzene (42 µL, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 110 °C for 14 h. This procedure afforded 57 mg (79%) of the title compound as an orange oil. This material was judged to be 97:3 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 1% IPA/Hexanes, 1mL/min, λ 254 nm, RT = 5.1 and 7.9 min). $[\alpha]^{23}_{D}$ –28.9 (*c* 1.19, CH_2Cl_2); ^{1}H NMR (700 MHz, C_6D_6) δ 7.15 (d, *J* = 7.5 Hz, 2 H), 7.11 (d, *J* = 6.6 Hz, 1 H), 7.08 (d, *J* = 7.5 Hz, 2 H), 6.96 (d, *J* = 8.5 Hz, 2 H), 6.86 (t, *J* = 7.5 Hz, 1 H), 6.81–6.75 (m, 2 H), 6.74 (t, *J* = 7.8 Hz, 1 H), 6.70 (d, *J* = 8.0 Hz, 1 H), 6.40 (d, *J* = 8.0 Hz, 1 H), 3.32 (s, 3 H), 3.22 (d, *J* = 12.6 Hz, 1 H), 2.83 (d, *J* = 12.6 Hz, 1 H), 2.70 (d, *J* = 10.9 Hz, 1 H), 2.65 (d, *J* = 10.9 Hz, 1 H), 2.62 (s, 3 H), 0.90 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 158.7, 139.1, 137.0, 136.5, 136.5, 133.5, 131.2, 128.4, 128.3, 126.5, 119.2, 118.4, 115.5, 115.0, 111.9, 58.0, 57.5, 54.9, 44.1, 38.9, 23.8 (an extra peak at 136.5 is present due to apparent slow bond rotation); IR (film) 2928, 1503 cm^{-1}; MS (ESI+) 359.2118 (359.2118 calcd for $C_{24}H_{26}N_2O$, M + H^+).



(*S*)-(+)-(4-{[1-(4-Methoxyphenyl)-2,4-dimethyl-1,2,3,4-tetrahydroquinoxalin-2-yl]methyl}phenyl)(phenyl)methanone (4b). General Procedure D was employed for the coupling of N^1-(4-methoxyphenyl)-N^2-methyl-N^2-(2-methylallyl)benzene-1,2-diamine (59 mg, 0.2 mmol) and 4-bromobenzophenone (104 mg, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 110 °C for 14 h. This procedure afforded 72 mg (78%) of the title compound as a light yellow solid, mp 63–66 °C. This material was judged to be 96:4 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 3% IPA/Hexanes, 1 mL/min, λ 254 nm, RT = 18.2 and 25.9 min), $[\alpha]^{23}_{D}$ +18.7 (*c* 0.90, CH_2Cl_2); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.0 Hz, 2 H), 7.75 (d, *J* = 8.0 Hz, 2 H), 7.15 (d, *J* = 8.5 Hz, 1 H), 7.08 (t, *J* = 7.6 Hz, 2 H), 7.02 (d, *J* = 8.0 Hz, 2 H), 6.96 (d, *J* = 8.9 Hz, 2 H), 6.87(t, *J* = 7.5 Hz, 1 H), 6.80 (d, *J* = 8.2 Hz, 2 H), 6.75 (t, *J* = 8.0 Hz, 1 H), 6.71 (d, *J* = 8.0 Hz, 1 H), 6.40 (d, *J* = 8.0 Hz, 1 H), 3.34 (s, 3 H), 3.22 (d, *J* = 12.4 Hz, 1 H), 2.81 (d, *J* = 12.3 Hz, 1 H), 2.65 (d, *J* = 11.1 Hz, 1 H), 2.61–2.56 (m, 4 H), 0.85 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 195.5, 158.8, 138.6, 136.8, 136.4, 136.3, 136.2, 133.4, 132.0, 131.0, 130.2, 130.2, 128.4, 119.3, 118.6, 115.6, 115.0, 112.0, 57.9, 57.6, 55.0, 43.9, 38.9, 23.8 (one aromatic carbon signal is missing due to incidental equivalence); IR (film) 2972, 1656 1504 cm^{-1}; MS (ESI+) 463.2371 (463.2380 calcd for $C_{31}H_{30}N_2O_2$, M + H$^+$).

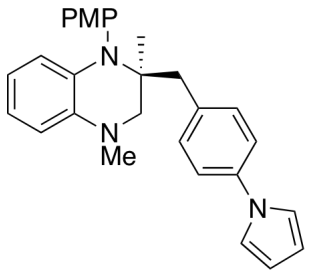


(*S*)-(+)-1-(4-Methoxyphenyl)-2,4-dimethyl-2-(naphthalen-2-ylmethyl)-1,2,3,4-tetrahydroquinoxaline (4c). General Procedure D was employed for the coupling of *N*^{1}-(4-methoxyphenyl)-*N*^{2}-methyl-*N*^{2}-(2-methylallyl)benzene-1,2-diamine (59 mg, 0.2 mmol) and 2-bromonaphthalene (83 mg, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 110 °C for 14 h. This procedure afforded 67 mg (82%) of the title compound as a light yellow solid, mp 62–65 °C. This material was judged to be 93:7 er by chiral HPLC analysis (lux-amylose, 15 cm x 4.6 mm, 3% IPA/Hexanes, 0.25 mL/min, λ 254 nm, RT = 27.9 and 30.2 min), $[\alpha]^{23}_{D}$ +8.56 (*c* 1.39, CH_2Cl_2); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.70–7.64 (m, 2 H), 7.59 (t, *J* = 8.2 Hz, 1 H), 7.52 (s, 1 H), 7.33–7.26 (m, 2 H), 7.22 (t, *J* = 8.4 Hz, 1 H), 7.08–6.98 (m, 2 H), 6.90 (t, *J* = 7.7 Hz, 1 H), 6.84–6.74 (m, 4 H), 6.44 (d, *J* = 8.0 Hz, 1 H), 3.40 (d, *J* = 12.8 Hz, 1 H), 3.35 (s, 3 H), 2.96 (d, *J* = 12.8 Hz, 1 H), 2.71–2.62 (m, 5 H), 0.93 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 158.7, 137.0, 136.6, 136.5, 136.4, 134.1, 133.7, 133.5, 132.8, 129.8, 129.7, 128.0, 127.7, 126.2, 125.7, 119.3, 118.4, 115.4, 115.0, 112.0, 57.9, 57.8, 55.0, 44.1, 38.9, 23.9; IR (film) 2969, 1504 cm^{-1}; MS (ESI+) 409.2268 (409.2274 calcd for $C_{28}H_{28}N_2O$, M + H^{+}).



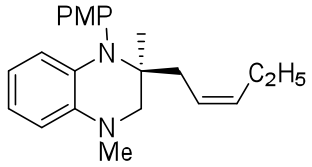
(*S*)-(+)-4-(4-{[1-(4-Methoxyphenyl)-2,4-dimethyl-1,2,3,4-tetrahydroquinoxalin-2-yl]methyl}phenyl)morpholine (4d). General Procedure D was employed for the

coupling of N^1-(4-methoxyphenyl)-N^2-methyl-N^2-(2-methylallyl)benzene-1,2-diamine (59 mg, 0.2 mmol) and 4-(4-bromophenyl)morpholine (97 mg, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 110 °C for 14 h. This procedure afforded 73 mg (82%) of the title compound as a white solid, mp 72–75 °C. This material was judged to be 96:4 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 2% IPA/Hexanes, 1 mL/min, λ 254 nm, RT = 16.3 and 17.8 min), $[\alpha]^{23}_D$ +21.6 (*c* 0.99, CH_2Cl_2); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.08 (d, *J* = 8.4 Hz, 2 H), 7.02 (d, *J* = 7.8 Hz, 2 H), 6.88 (t, *J* =7.2 Hz, 1 H), 6.83–6.72 (m, 4 H), 6.67 (d, *J* = 8.3 Hz, 2 H), 6.43 (d, *J* = 7.0 Hz, 1 H), 3.58 (t, *J* = 4.6 Hz, 4 H), 3.33 (s, 3 H), 3.25 (d, *J* = 12.9 Hz, 1 H), 2.87 (d, *J* = 12.9 Hz, 1 H), 2.83 (d, *J* = 10.8 Hz, 1 H), 2.79 (d, *J* = 4.7 Hz, 4 H), 2.72 (d, *J* = 10.9 Hz, 1 H), 2.70 (s, 3 H), 0.99 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 158.7, 150.3, 137.1, 136.6, 136.5, 133.5, 131.8, 130.0, 128.2, 119.2, 118.3, 115.7, 115.4, 114.9, 111.9, 67.0, 58.1, 57.7, 54.9, 49.6, 43.3, 39.0, 23.8 (an extra peak appears at 136.5 is present due to apparent slow bond rotation); IR (film) 2957, 1504 cm^{-1}; MS (ESI+) 444.2645 (444.2646 calcd for $C_{28}H_{33}N_3O_2$, M + H$^+$).

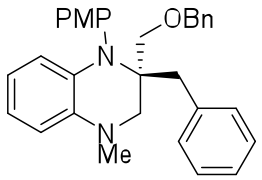


(*S*)-(+)-2-[4-(1*H*-pyrrol-1-yl)benzyl]-1-(4-methoxyphenyl)-2,4-dimethyl-1,2,3,4-tetrahydroquinoxaline (4e). General Procedure BDwas employed for the coupling of N^1-(4-methoxyphenyl)-N^2-methyl-N^2-(2-methylallyl)benzene-1,2-diamine (59 mg, 0.2 mmol) and 1-(4-iodophenyl)pyrrole (108 mg, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 120 °C in xylenes for 14 h. This procedure afforded 60 mg (70%) of the title compound as an white solid, mp 65–68 °C. This material was judged to be 93:7 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 1% IPA/Hexanes, 1 mL/min, λ 254 nm, RT = 16.2 and 22.5 min), $[\alpha]^{23}_D$ +22.7

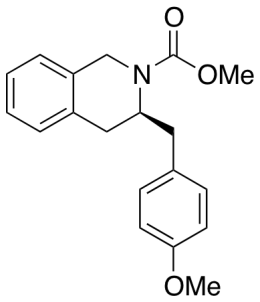
(*c* 0.88, CH_2Cl_2); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.03–6.94 (m, 8 H), 6.89 (t, *J* = 7.3 Hz, 1 H), 6.81 (d, *J* = 8.4 Hz, 2 H), 6.76 (d, *J* = 7.7 Hz, 1 H), 6.74 (d, *J* = 7.8 Hz, 1 H), 6.45 (t, *J* = 1.9 Hz, 2 H), 6.42 (d, *J* = 7.8 Hz, 1 H), 3.34 (s, 3 H), 3.18 (d, *J* = 12.8 Hz, 1 H), 2.79 (d, *J* = 12.8 Hz, 1 H), 2.69–2.63 (m, 5 H), 0.89 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 158.7, 139.5, 136.9, 136.5, 136.1, 136.0, 135.6, 133.0, 131.6, 119.6, 118.94, 118.93, 118.1, 115.2, 114.6, 111.6, 110.7, 57.9, 57.5, 55.0, 43.3, 39.0, 23.8 (an extra peak at 136.0 is present due to apparent slow bond rotation); IR (film) 2970, 2360, 2339.4, 1519, 1504 cm^{-1}; MS (ESI+) 424.2380 (424.2383 calcd for $C_{28}H_{29}N_3O$, M + H^+).



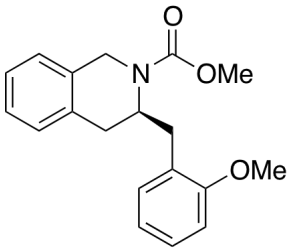
(*S*,*Z*)-(+)-1-(4-methoxyphenyl)-2,4-dimethyl-2-(pent-2-en-1-yl)-1,2,3,4-tetrahydroquinoxaline (4f). General Procedure D was employed for the coupling of *N*^{1}-(4-methoxyphenyl)-*N*^{2}-methyl-*N*^{2}-(2-methylallyl)benzene-1,2-diamine (59 mg, 0.2 mmol) and *Z*-1-bromobutene (54 mg, 0.40 mmol) using NaO^{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 110 °C for 14 h. This procedure afforded 50 mg (75%) of the title compound as a clear oil. This material was judged to be 98:2 er by chiral HPLC analysis (Chriacel ADH, 15 cm x 4.6 mm, 0.50% IPA/Hexanes, 1 mL/min, λ 254 nm, RT = 6.6 and 9.3 min), $[\alpha]^{23}_D$ +21.7 (*c* 2.4, CH_2Cl_2); ^{1}H NMR (500 MHz, C_6D_6) δ 7.04 (d, *J*= 7.3 Hz, 2 H), 6.84 (t, *J* = 7.3 Hz, 1 H), 6.78 (d, *J* = 8.6 Hz, 2 H), 6.72 (t, *J* = 7.7 Hz, 1 H), 6.65 (d, *J* = 7.8 Hz, 1 H), 6.35 (d, *J* = 8.0 Hz, 2 H), 5.50–5.41 (m, 1 H), 5.40–5.32 (m, 1 H), 3.32 (s, 3 H), 2.95 (d, *J* = 10.8 Hz, 1 H), 2.79 (d, *J* = 10.8 Hz, 1 H), 2.70–2.61 (m, 4 H), 2.48 (dd, *J*= 13.7, 7.1 Hz, 1H), 2.05–1.90 (m, 2H), 1.04 (s, 3 H), 0.89 (t, *J*= 7.3 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.7, 137.4, 136.4, 136.3, 134.5, 133.6, 124.8, 119.2, 118.2, 115.0, 114.9 111.8, 59.2, 57.0, 54.9, 39.4, 36.0, 23.9, 21.0, 14.5; IR (film) 2957, 1671, 1504 cm^{-1}; MS (ESI+) 337.2276 (337.2274 calcd for $C_{22}H_{28}N_2O$, M + H^+).



(*S*)-(–)-2-Benzyl-2-[(benzyloxy)methyl]-1-(4-methoxyphenyl)-4-methyl-1,2,3,4-tetrahydroquinoxaline (4g): General Procedure D was employed for the coupling of N^1-{2-[(benzyloxy)methyl]allyl}-N^2-(4-methoxyphenyl)-N^1-methylbenzene-1,2-diamine (39 mg, 0.1 mmol) and bromobenzene (21 µL, 0.20 mmol) using NaO^{t}Bu (19 mg, 0.20 mmol) as the base and a reaction temperature of 125 °C for 12 h. This procedure afforded 37 mg (79%) of the title compound as a viscous white oil. This material was judged to be 96:4 er by chiral HPLC analysis (Chriacel ADH, 15 cm x 4.6 mm, 1.00% IPA/Hexanes, 1 mL/min, λ 254 nm, RT = 7.0 and 10.6 min), $[\alpha]^{23}_{D}$ –19.51 (*c* 1.23, CH_2Cl_2); ^{1}H NMR (700 MHz, C_6D_6) δ 7.31 (t, *J*= 7.5 Hz, 2 H), 7.28–7.18 (m, 7 H), 7.16 (d, *J* = 7.3 Hz, 2 H), 6.98–6.90 (m, 1 H), 6.86 (d, *J* = 8.9 Hz, 2 H), 6.69–6.64 (m, 2H), 6.51 (t, *J*= 7.2 Hz, 1 H), 6.08 (d, *J* = 8.0 Hz, 1 H), 4.29 (d, *J* = 11.8 Hz, 1 H), 4.24 (d, *J* = 11.8 Hz, 1 H), 3.82 (s, 3 H), 3.39 (d, *J* = 9.9 Hz, 1 H), 3.23 (d, *J* = 11.1 Hz, 1 H), 3.21 (d, *J* = 9.7 Hz, 1 H) 3.09 (d, *J* = 13.3 Hz, 1 H), 3.04 (d, *J* = 10.9 Hz, 1 H), 3.00 (d, *J* = 13.1 Hz, 1 H), 2.92 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 158.1, 138.2, 138.1, 136.4, 136.0, 135.8, 133.4, 132.8, 130.9, 128.3, 128.1, 127.5, 127.4, 126.3, 118.2, 117.6, 115.0, 114.6, 114.2, 111.1, 72.9, 72.1, 60.6, 55.4, 54.1, 40.3, 39.2 (extra peaks at 132.8 and 114.2 are present due to apparent slow bond rotation); IR (film) 2923, 2859, 1504 cm^{-1}; MS (ESI+) 465.2536 (465.2537 calcd for $C_{31}H_{32}N_2O_2$, M + H$^+$).

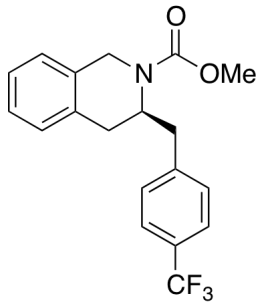


(*S*)-(+)-Methyl 3-(4-methoxybenzyl)-3,4-dihydroisoquinoline-2(1*H*)-carboxylate (6a). General Procedure D was employed for the coupling of (2-allylbenzyl)carbamate (51 mg, 0.25 mmol) and 4-bromoanisole (60 mg, 0.32 mmol) using NaO^{t}Bu (31 mg, 0.32 mmol) as the base and a reaction temperature of 90 °C for 2 h. This procedure afforded 40 mg (51%) of the title compound as a colorless oil. This material was judged to be 93:7 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 5% IPA/Hexanes, 1 mL/min, λ 215 nm, RT = 5.8 and 12.1 min), $[\alpha]^{23}_{D}$ +48.8 (*c* 0.86, CH_2Cl_2); ^{1}H NMR (700 MHz, d8-toluene, 95 °C) δ 7.01–6.95 (m, 2 H), 6.92 (d, *J* = 8.2 Hz, 2 H), 6.88–6.83 (m, 2 H), 6.69 (d, *J* = 8.5 Hz, 1 H), 4.82 (d, *J* = 16.6 Hz, 1 H), 4.73 (s, br, 1 H), 4.34 (d, *J* = 16.7 Hz, 1 H), 3.60 (s, 3 H), 3.49 (s, 3 H), 2.74–2.67 (m, 2 H), 2.67 (d, *J* = 13.6 Hz, 1 H), 2.35 (dd, *J* = 9.0, 13.5, Hz 1 H); ^{13}C NMR (175 MHz, d8-toluene, 95 °C) δ 159.6, 156.4, 134.2, 134.9, 131.6, 130.8, 129.8, 127.3, 126.9, 126.8, 114.9, 55.3, 52.7, 52.4, 44.3, 38.4, 32.5; IR (film) 2952, 1695 cm^{-1}; MS (ESI+) 312.1589 (312.1594 calcd for $C_{19}H_{21}NO_3$, M + H$^+$).



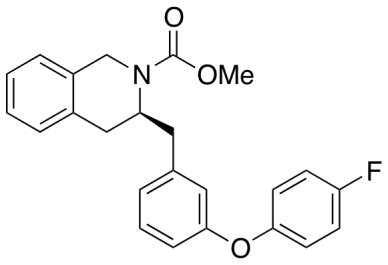
(*S*)-(+)-Methyl 3-(2-methoxybenzyl)-3,4-dihydroisoquinoline-2(1*H*)-carboxylate (6b). General Procedure D was employed for the coupling of (2-allylbenzyl)carbamate (51 mg, 0.25 mmol) and 2-iodoanisole (85 mg, 0.32 mmol) using NaO^{t}Bu (31 mg, 0.32

mmol) as the base and a reaction temperature of 90 °C for 2 h. This procedure afforded 33 mg (42%) of the title compound as a white solid, mp 74-77 °C. This material was judged to be 80:20 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 5% IPA/Hexanes, 1 mL/min, λ 215 nm, RT = 5.7 and 8.2 min), $[\alpha]^{23}_{D}$ +77.0 (*c* 0.67, CH_2Cl_2); ^{1}H NMR (700 MHz, d8-toluene, 95 °C) δ 7.02–6.92 (m, 5 H), 6.88–6.82 (m, 2 H), 6.73 (t, *J* = 7.5 Hz, 1 H), 6.56 (d, *J* = 8.1 Hz, 1 H), 4.88 (s, br, 1 H), 4.80 (d, *J* = 16.8 Hz, 1 H), 4.32 (d, *J* = 16.8 Hz, 1 H), 3.44 (s, 3H), 3.41 (s, 3 H), 2.80–2.72 (m, 2 H), 2.64 (dd, *J* = 8.1, 13.2 Hz, 1 H), 2.46 (d, *J* = 15.9 Hz, 1 H); ^{13}C NMR (175 MHz, d8-toluene, 95 °C) δ 159.1, 156.7, 134.3, 134.2, 131.9, 130.0, 127.2, 126.9, 126.8, 121.3, 111.5, 55.6, 52.4, 51.3, 44.3, 33.7, 33.4 (two aromatic carbon signals are missing due to incidental equivalence); IR (film) 2951, 1698 cm^{-1}; MS (ESI+) 312.1593 (312.1594 calcd for $C_{19}H_{21}NO_3$, M + H^+).

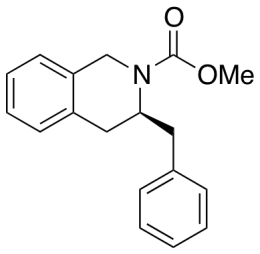


(*S*)-(+)-Methyl 3-[4-(trifluoromethyl)benzyl]-3,4-dihydroisoquinoline-2(1*H*)-carboxylate (6c). General Procedure D was employed for the coupling of (2-allylbenzyl)carbamate (51 mg, 0.25 mmol) and 4-bromobenzotrifluoride (72 mg, 0.32 mmol) using NaO^{t}Bu (31 mg, 0.32 mmol) as the base and a reaction temperature of 90 °C for 2 h. This procedure afforded 63 mg (72%) of the title compound as a colorless oil. This material was judged to be 93:7 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 5% IPA/Hexanes, 1 mL/min, λ 215 nm, RT = 4.4 and 9.3 min), $[\alpha]^{23}_{D}$ +39.5 (*c* 1.24, CH_2Cl_2); ^{1}H NMR (700 MHz, d8-toluene, 95 °C) δ 7.32 (d, *J*= 7.8 Hz, 2 H), 7.03–6.94 (m, 2 H), 6.92 (d, *J* = 7.8 Hz, 2 H), 6.87–6.79 (m, 2 H), 4.67 (d, *J* = 16.6 Hz, 1 H), 4.58 (s, br, 1 H), 4.19 (d, *J* = 16.7 Hz, 1 H), 3.47 (s, 3 H), 2.68–2.62 (m, 2 H), 2.34–2.26 (m, 2 H); ^{13}C NMR (175 MHz, d8-toluene, 95 °C) δ 156.3, 143.8, 133.9, 133.5, 130.3,

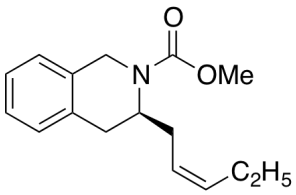
129.7, 127.5, 127.2, 126.8, 125.8, 152.5, 152.3, 44.3, 39.1, 32.6 (two aromatic carbon signals are missing due to incidental equivalence); IR (film) 2954, 1695 cm^{-1}. MS (ESI+) 350.1365 (350.1362 calcd for $C_{19}H_{18}F_3NO_2$, M + H^+).



(*S*)-(+)-Methyl 3-[3-(4-fluorophenoxy)benzyl]-3,4-dihydroisoquinoline-2(1*H*)-carboxylate (6d). General Procedure D was employed for the coupling of (2-allylbenzyl)carbamate (51 mg, 0.25 mmol) and 3-bromo-4'-fluorodiphenyl ether (85 mg, 0.32 mmol) using NaO^tBu (31 mg, 0.32 mmol) as the base and a reaction temperature of 90 °C for 2 h. This procedure afforded 60 mg (61%) of the title compound as colorless oil. This material was judged to be 93:7 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 5% IPA/Hexanes, 1 mL/min, λ 215 nm, RT = 7.7 and 13.3 min), $[\alpha]^{23}_D$ +48.3 (*c* 1.18, CH_2Cl_2); ^{1}H NMR (700 MHz, d8-toluene, 95 °C) δ 7.02–6.93 (m, 3 H), 6.83–6.68 (m, 9 H), 4.68 (d, *J* = 16.8 Hz, 1 H), 4.62 (s, br, 1 H), 4.20 (d, *J* = 16.6 Hz, 1 H), 3.46 (s, 3 H), 2.71–2.62 (m, 2 H), 2.39 (d, *J* = 15.9 Hz, 1 H), 2.31 (dd, *J* = 8.5, 13.5 Hz, 1 H); ^{13}C NMR (175 MHz, d8-toluene, 95 °C) δ 159.8 (d, *J* = 241 Hz), 158.8, 156.3, 154.2, 141.8, 134.0, 133.6, 130.2, 129.7, 127.4, 127.0, 126.8, 124.9, 121.1 (d, *J* = 7 Hz), 120.3, 117.2, 116.8 (d, *J* = 23 Hz), 52.5, 44.3, 39.2, 32.8 (one aliphatic carbon signal is missing due to incidental equivalence); IR (film) 2952, 1695, 1500 cm^{-1}; MS (ESI+) 392.1658 (392.1656 calcd for $C_{24}H_{22}FNO_2$, M + H^+).



(*S*)-(+)-Methyl 3-benzyl-3,4-dihydroisoquinoline-2(1*H*)-carboxylate (6e). General Procedure D was employed for the coupling of (2-allylbenzyl)carbamate (51 mg, 0.25 mmol) and bromobenzene (51 mg, 0.32 mmol) using NaO^{t}Bu (31 mg, 0.32 mmol) as the base and a reaction temperature of 90 °C for 2 h. This procedure afforded 48 mg (68%) of the title compound as a colorless oil. This material was judged to be 94:6 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 5% IPA/Hexanes, 0.5 mL/min, λ 215nm, RT = 9.9 and 18.5 min), $[\alpha]^{23}_{D}$ +63.2 (*c* 1.28, CH_2Cl_2); ^{1}H NMR (700 MHz, d8-toluene, 90 °C) δ 7.10–7.03 (m, 2 H), 7.03–6.95 (m, 5 H), 6.87–6.81 (m, 2 H), 4.72 (d, *J* = 16.7 Hz, 1 H), 4.66 (s, br, 1 H), 4.24 (d, *J* = 16.7 Hz, 1 H), 3.49 (s, 3 H), 2.72 (dd, *J* = 5.8, 13.3 Hz, 1 H), 2.67 (dd, *J* = 5.6, 15.7 Hz, 1 H), 2.41 (d, *J* = 15.7 Hz, 1 H), 2.37 (dd, *J* = 8.9, 13.5 Hz, 1 H); ^{13}C NMR (125 MHz, d8-toluene, 90 °C) δ 156.3, 139.7, 134.1, 133.8, 130.0, 129.7, 129.0, 127.3, 126.9, 126.9, 126.8, 52.6, 52.4, 44.3, 39.3, 32.5; IR (film) 2953 1697 cm^{-1}; MS (ESI+) 282.1491 (282.1489 calcd for $C_{18}H_{19}NO_2$, M + H$^+$).

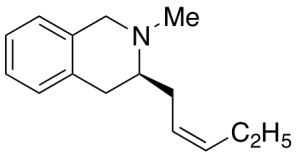


(*Z,S*)-(+)-Methyl 3-[4-(trifluoromethyl)benzyl]-3,4-dihydroisoquinoline-2(1*H*)-carboxylate (6f). General Procedure D was employed for the coupling of (2-allylbenzyl)carbamate (51 mg, 0.25 mmol) and (*Z*)-1-bromo-1-butene (43 mg, 0.32 mmol) using NaO^{t}Bu (31 mg, 0.32 mmol) as the base and a reaction temperature of 90 °C for 2 h. This procedure afforded 36 mg (57%) of the title compound as a colorless oil.

This material was judged to be 93:7 er by chiral HPLC analysis (ODH, 15 cm x 4.6 mm, 1% IPA/Hexanes, 1 mL/min, λ 215 nm, RT = 6.7 and 22.1 min), $[\alpha]^{23}_{D}$ +46.7 (*c* 1.12, CH_2Cl_2); (The mixture was found to exist as a 2.5:1 mixture of rotomers in the nmr, with most of the minor rotomer peaks appearing partially in the major rotomer peaks, however coupling constants given are all for the major rotomer) ^{1}H NMR (700 MHz, d8-toluene, 95 °C) δ 6.98–6.93 (m, 2 H), 6.88–6.84 (m, 2 H), 6.83–6.79 (m, 2 H), 5.38–5.33 (m, 1 H), 5.30–5.25 (m, 1 H), 4.78 (d, *J* = 16.7 Hz, 1 H), 4.48 (s, br, 1 H), 4.19 (d, *J* = 16.7 Hz, 1 H), 3.55 (s, 3 H), 2.78 (dd, *J* = 6.1, 15.6 Hz, 1 H), 2.46 (d, *J* = 15.7 Hz, 1 H), 2.20–2.15 (m, 1 H), 2.00–1.95 (m, 1 H), 1.90-1.84 (m, 0.63 H), 1.80 (quin, *J* = 7.30 Hz, 1.45 H), 0.87 (t, *J* = 7.5 Hz, 0.76 H), 0.80 (t, *J* = 7.5 Hz, 2.22 H); ^{13}C NMR (125 MHz, d8-toluene, 95 °C) δ 156.4, 134.5, 134.1, 133.9, 129.7, 127.2, 126.9, 126.6, 125.7, 52.6, 51.1, 44.1, 33.1, 30.7, 21.2, 14.5; IR (film) 2958, 1699 cm^{-1}; MS (ESI+) 260.1644 (260.1645 calcd for $C_{16}H_{21}NO_2$, M + H^+).

Confirmation of product 6f structure.

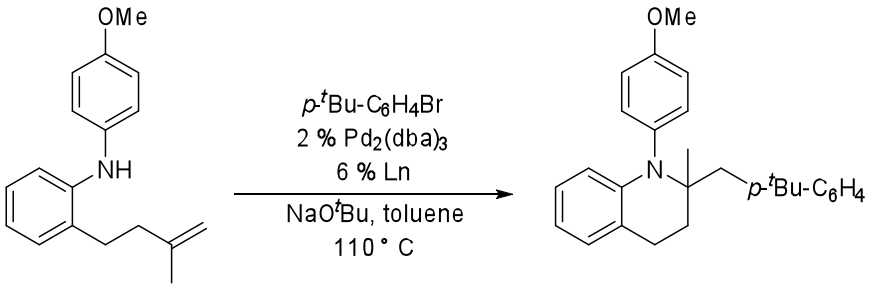
The NMR spectrum of **6f** was complicated due to apparent rotomers. In order to rule out the presence of *E*:*Z* alkene stereoisomers the product was reduced with $LiAlH_4$ to form the analogous *N*-methyl isoquinoline derivative **S4**.

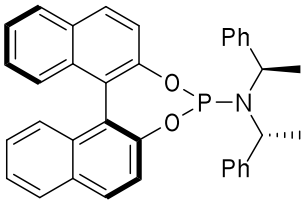


(*Z*,*S*)-(–)-2-methyl-3-(pent-2-en-1-yl)-1,2,3,4-tetrahydroisoquinoline (S4). A flame-dried round-bottom flask equipped with a stirbar was cooled under a stream of nitrogen and charged with **6f** (35 mg, 0.136 mmol) and THF (4 mL). The resulting solution was cooled to 0°C and after five minutes of stirring $LiAlH_4$ (0.27 mL, 0.27 mmol, 1.0M in THF) was added dropwise. The resulting solution was heated to reflux for 1.5 h then cooled to rt and quenched with 0.1 mL H_2O followed by 0.1 mL of a 15% aqueous

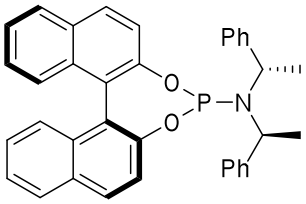
NaOH solution. The mixture was filtered and the solid was washed with ether (2 x 5) mL. The combined organic solutions were dried over anhydrous $MgSO_4$, filtered, and concentrated in vacuo. The crude material was purified by flash chromatography on silica gel using hexanes/EtOAc as the eluent to afford 20 mg (69%) of the product as a clear oil; $[\alpha]^{23}_D$ –24.7 (*c* 1.86, CH_2Cl_2). ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.13–7.09 (m, 2 H), 7.08–7.05 (m, 1 H), 7.04–7.00 (m, 1 H), 5.53–5.48 (m, 1 H), 5.44–5.39 (m, 1 H), 3.82 (d, *J* = 15.5 Hz, 1 H), 3.68 (d, *J* = 15.6 Hz, 1 H), 2.79–2.72 (m, 1 H), 2.69–2.58 (m, 2 H), 2.36 (s, 3 H), 2.32–2.27 (m, 1 H), 2.10–2.04 (m, 1H), 2.03 (quint, *J* = 7.5 Hz, 2 H), 0.97 (t, *J* = 7.5 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 134.3, 134.0, 133.7, 128.8, 126.2, 126.1, 125.6, 125.4, 59.1, 56.4, 41.0, 32.3, 28.3, 20.7, 14.2; IR (film) 2960, 1456 cm^{-1}; MS (ESI+) 216.1747 (216.1747 calcd for $C_{15}H_{21}N$, M + H^+).

Results of a screen of chiral ligands in the reaction of 1b with 1-bromo-4-*tert*-butylbenzene.

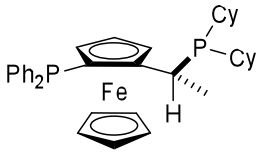




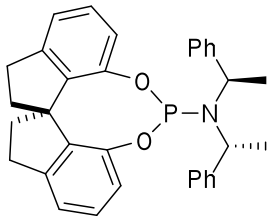
84% yield, 54:46 er



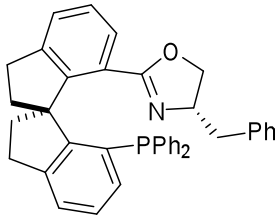
78% yield, 59:41 er



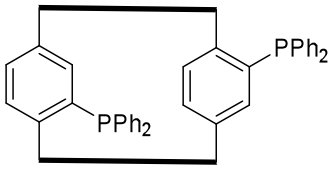
(R)-(S)-Josiphos
25 % yield, 62:38 er



(R)-Siphos-PE
80% yield, 68:32 er



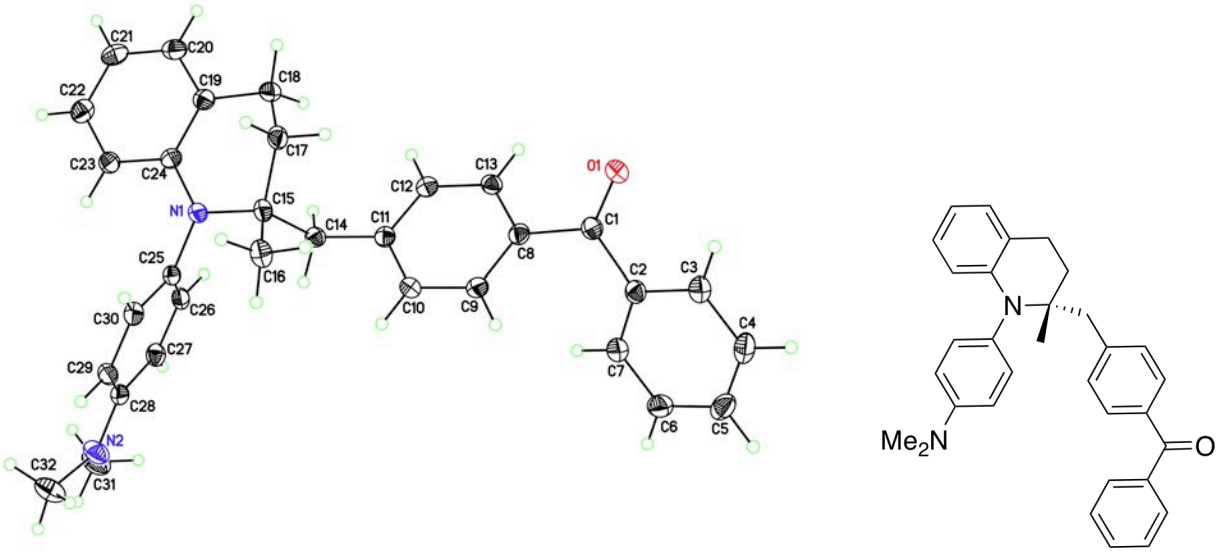
(Ra-S)-Ph-Bn-SIPHOX
65% yield, 83:17 er



(S)-Phanephos
86% yield, 64:38 er

Assignment of Absolute Stereochemistry.

The absolute stereochemistry of product **2j** was established by single crystal x-ray analysis as shown below. The stereochemistry of all other products was assigned based on analogy to **2j**.



References

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(2) H. Arada, R. K. Thalji, R. G. Bergman, J. A. Ellman, *J. Org. Chem*., 2008, **73**, 6772.

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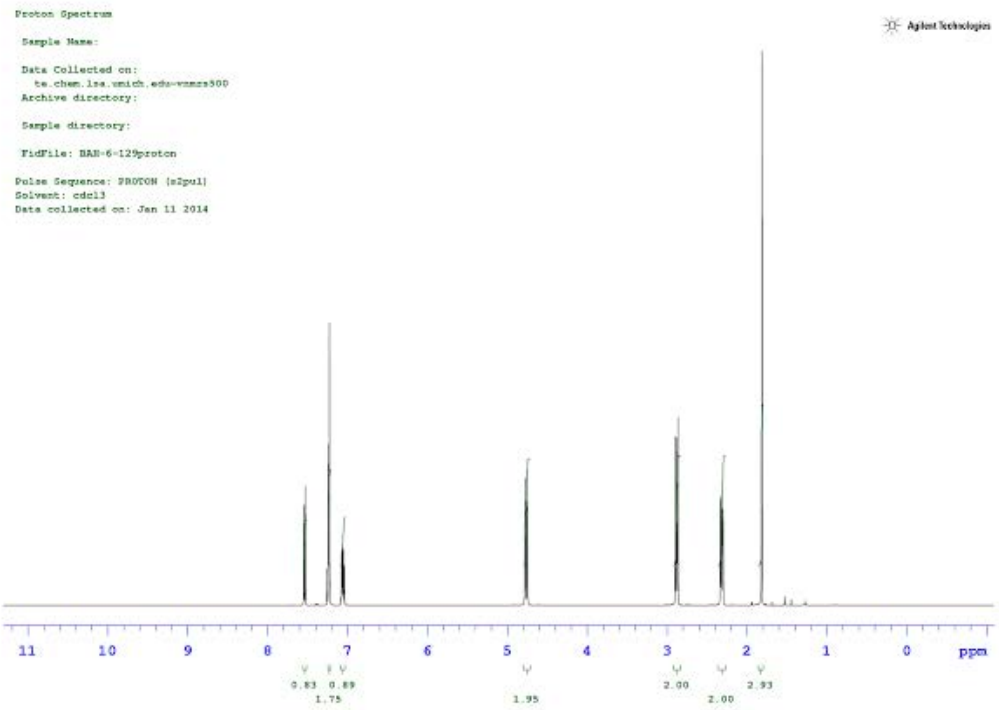
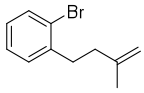
(4) I. D. G. Watson, S. Ritter, F. D. Toste, *J. Am. Chem. Soc*., 2009, **131**, 2056.

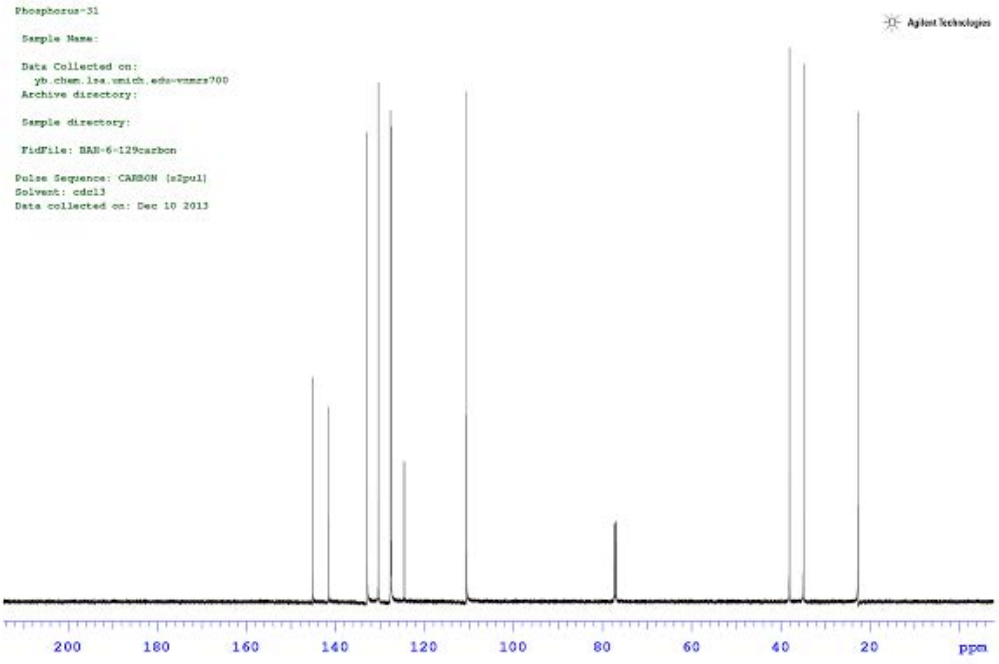
(5) C. B. Tripathi, S. Mukherjee, *Angew. Chem. Int. Ed*., 2013, **52**, 8450.

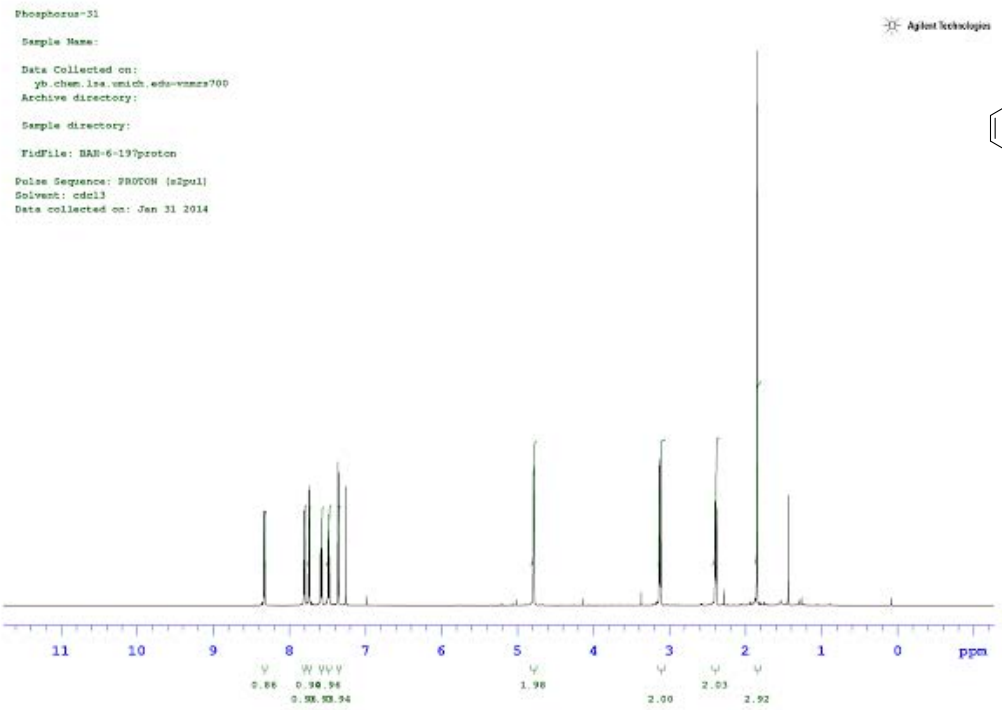
(6) X.-T. Zhou, L. Lu, D. P. Furkert, C. E. Wells, R. G. Carter, *Angew. Chem. Int. Ed*., 2006, **45**, 7622.

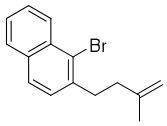

Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-6-129proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 11 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.83 0.89
1.75
1.95
2.00
2.00
2.93


Br

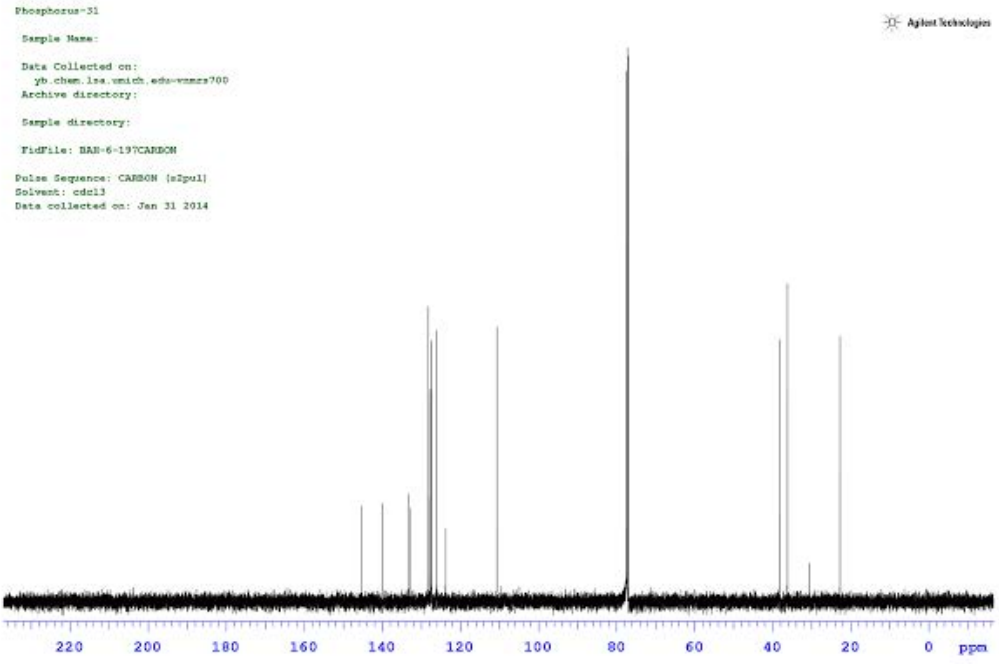
S1a

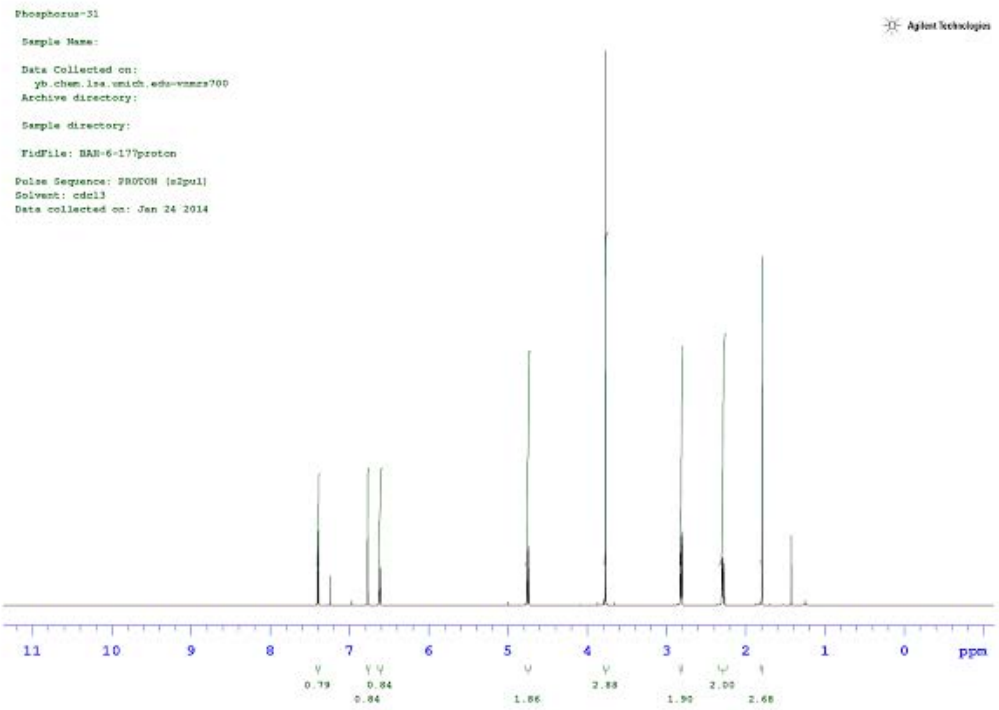

Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-129carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Dec 10 2013
Agilent Technologies
200 180 160 140 120 100 80 60 40 20 ppm


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-6-197proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 31 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.86
0.98 0.96
0.98 0.94
1.98
2.00
2.03
2.92


Br

S1c


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-6-197CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 31 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm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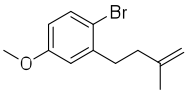
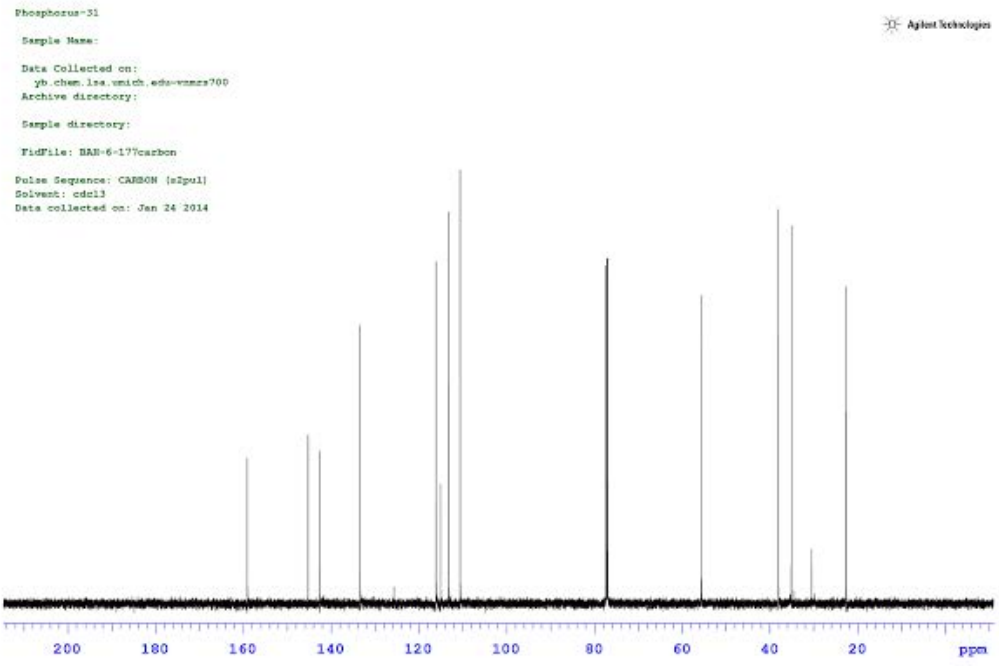
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-177proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 24 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.79
0.84
0.84
1.86
2.88
1.90
2.00
2.68


Br
O

S1b


Phosphorus-31
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Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
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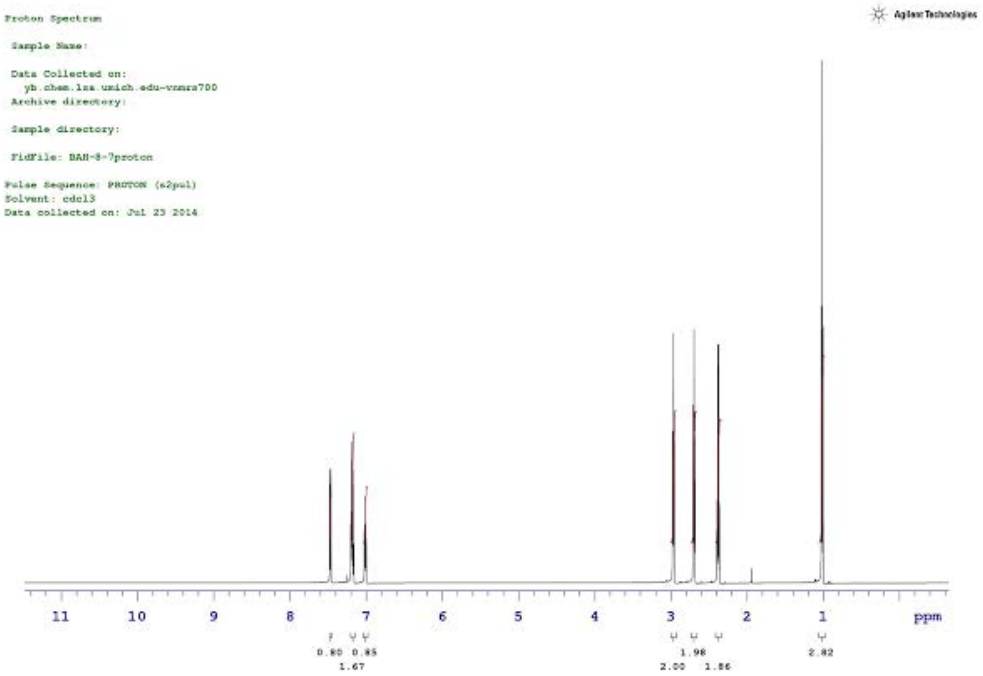
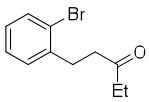
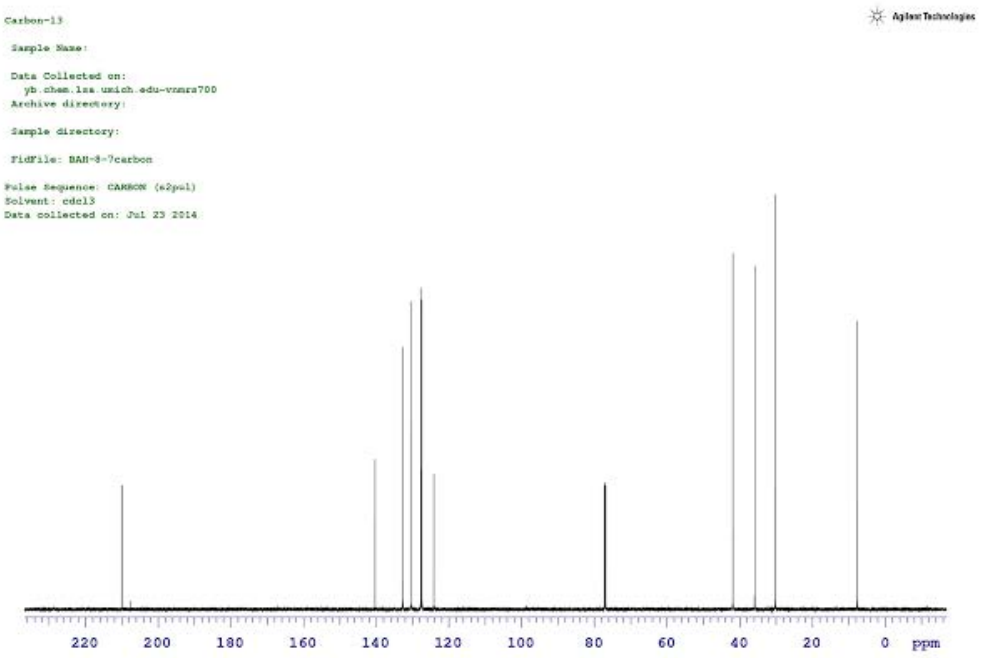
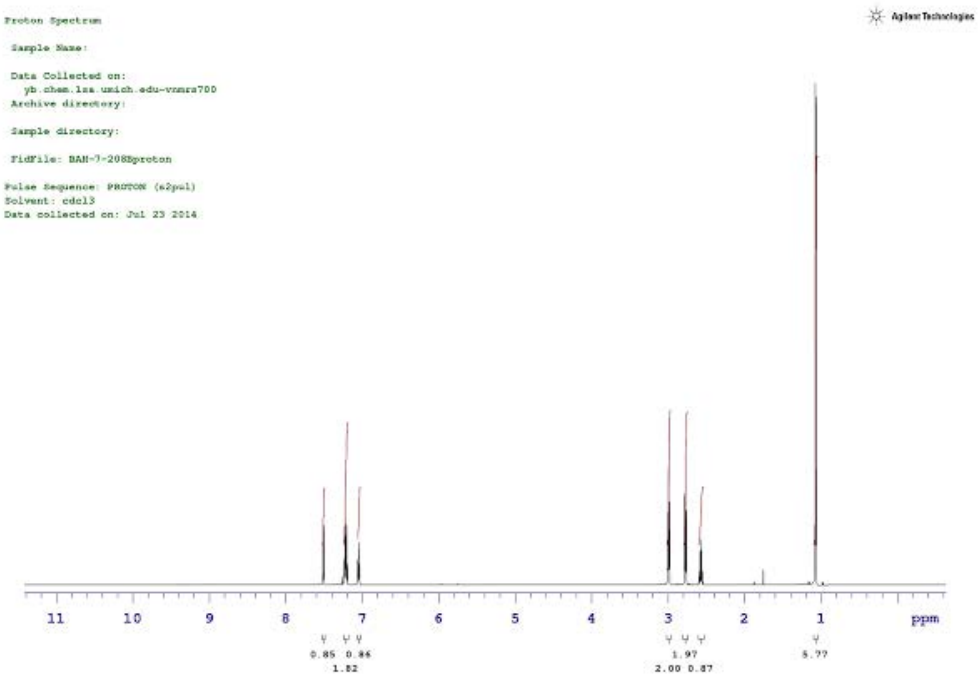
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 24 2014
Agilent Technologies
200 180 160 140 120 100 80 60 40 20 ppm

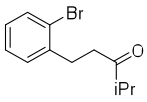

Proton Spectrum
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-8-7proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 23 2014
Agilent Technologies
11
10
9
8
7
6
5
4
3
2
1
ppm
0.80
0.85
1.67
1.98
2.00
1.86
2.82


Br
O
Et

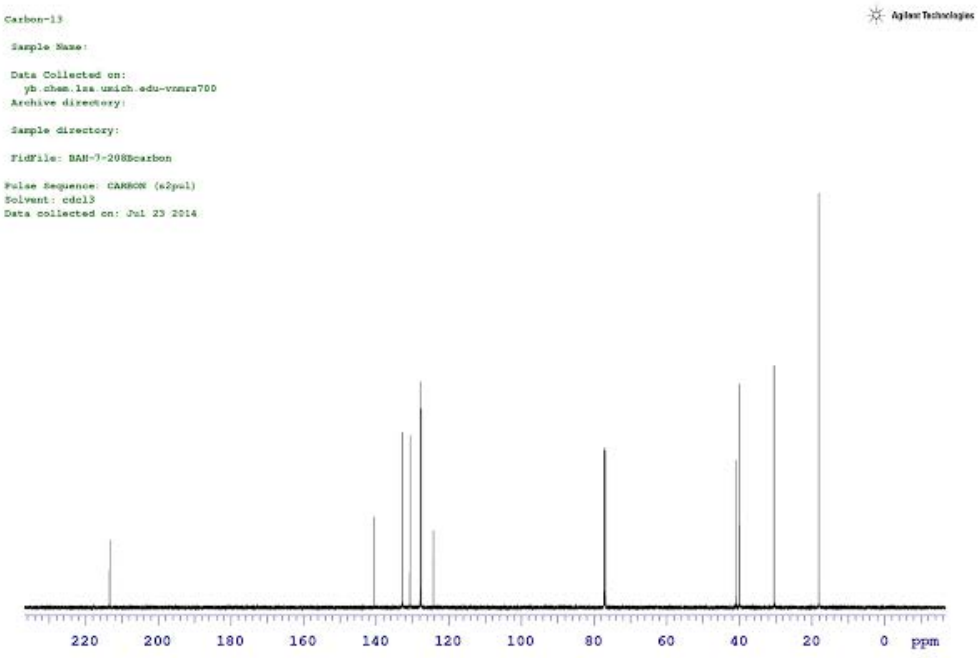
S2b


Carbon-13
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-8-7carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 23 2014
Agilent Technologies
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Proton Spectrum
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-7-208Bproton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 23 2014
Agilent Technologies
11
10
9
8
7
6
5
4
3
2
1
ppm
0.85 0.86
1.82
1.97
2.00 0.87
5.77


Br
O
iPr

S2c


Carbon-13
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Data Collected on:
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Archive directory:
Sample directory:
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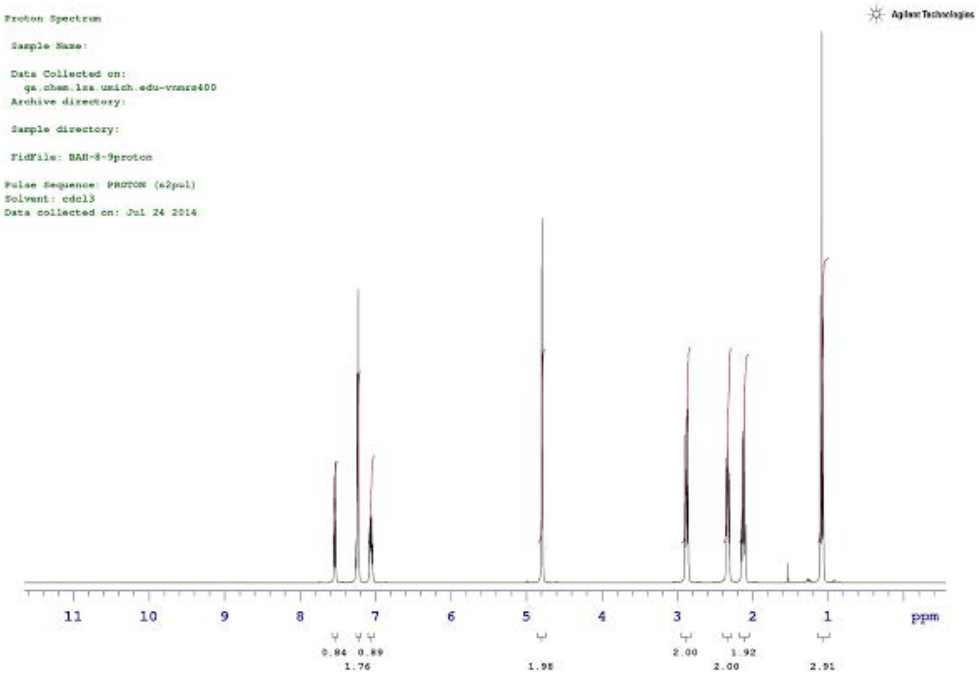
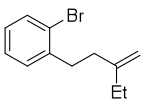
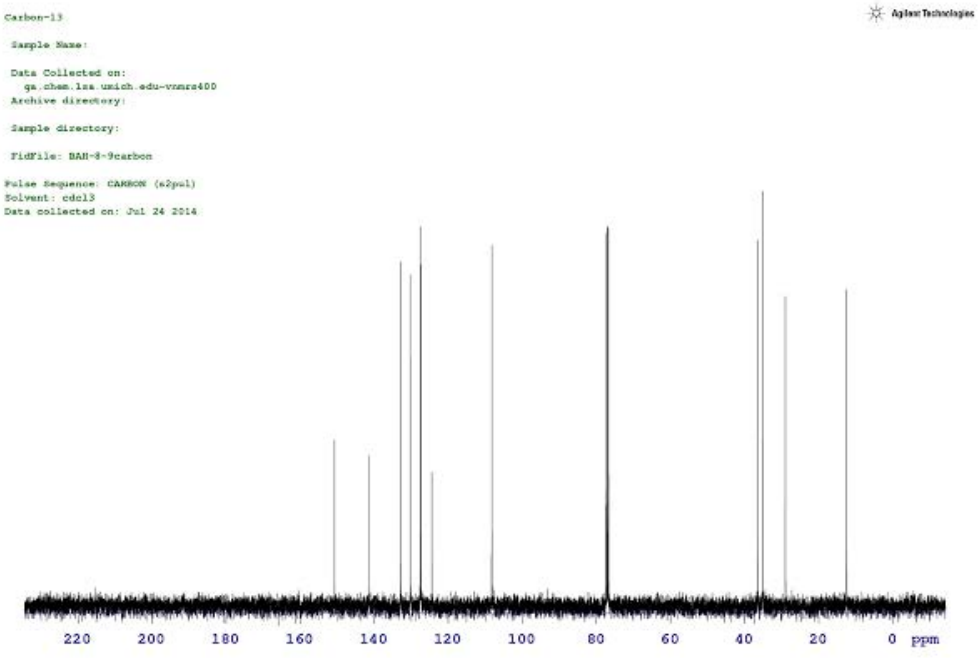
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 23 2014
Agilent Technologies
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Proton Spectrum
Sample Name:
Data Collected on:
qa.chem.lsa.umich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-8-9proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 24 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 ppm
0.84 0.89
1.76
1.98
2.00
1.92
2.00
2.91


Br
Et

S1e


Carbon-13
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Data Collected on:
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Archive directory:
Sample directory:
FidFile: BAH-8-9carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
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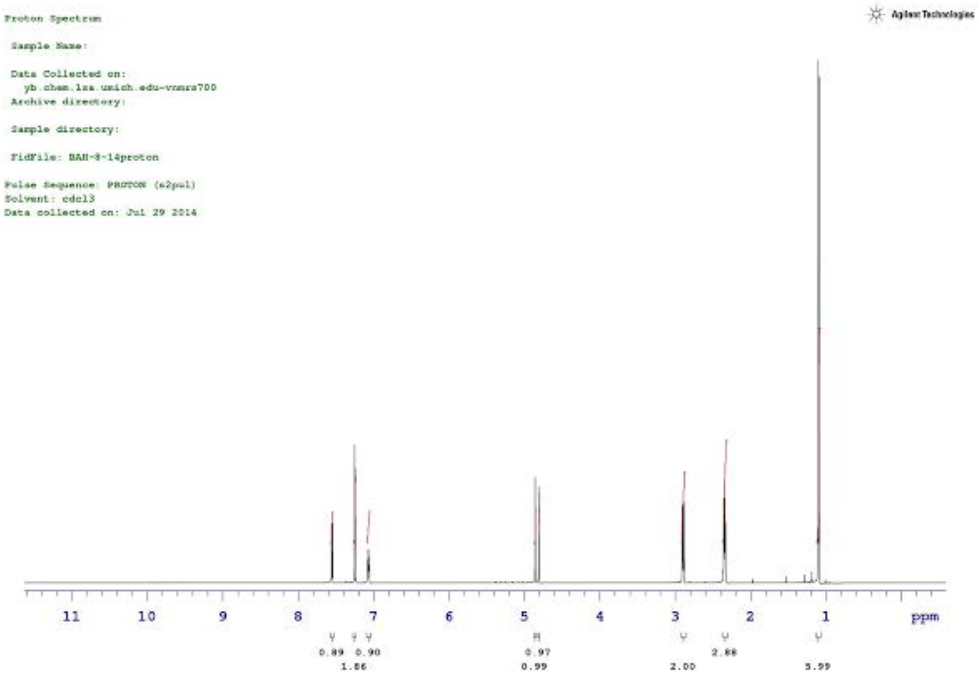
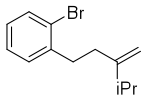
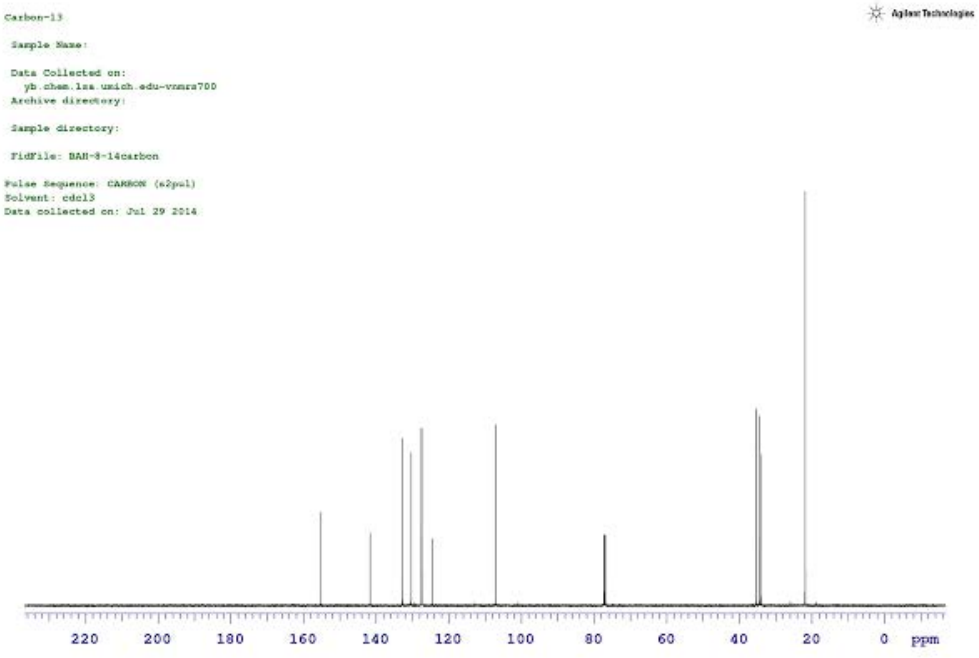
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm


Proton Spectrum
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-8-14proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 29 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 ppm
0.89 0.90
1.86
0.97
0.99
2.00
2.88
5.99


Br
iPr

S1f


Carbon-13
Sample Name:
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yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-8-14carbon
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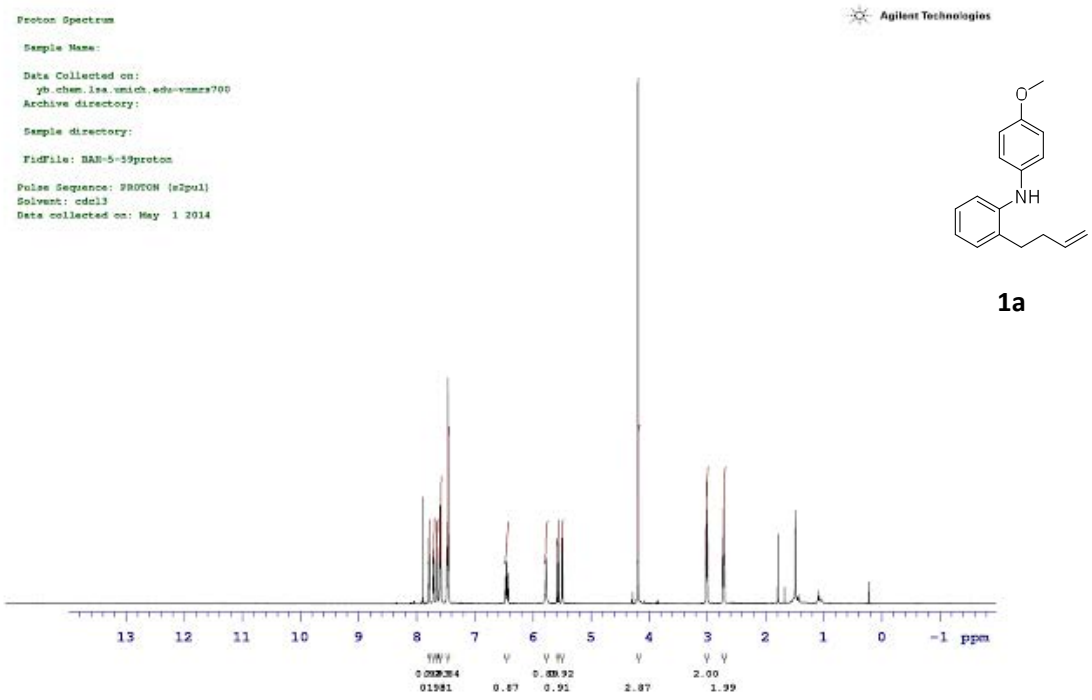
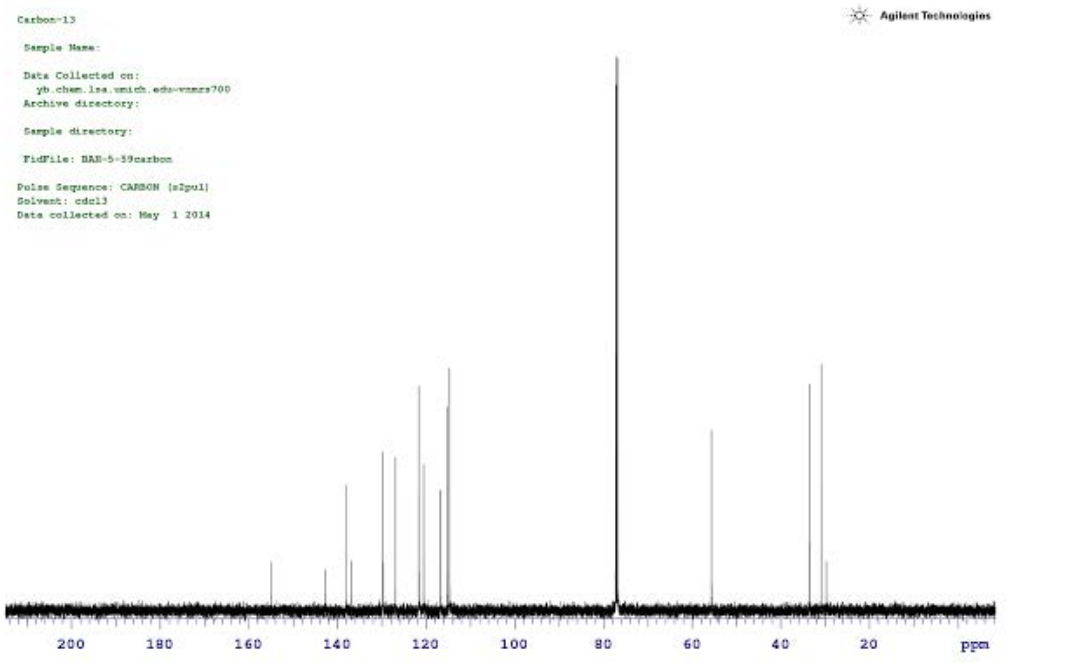
Solvent: cdcl3
Data collected on: Jul 29 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm


Proton Spectrum
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-5-59proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 1 2014
Agilent Technologies
O
NH
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm
0.87
0.91
2.87
2.00
1.99

1a


Carbon-13
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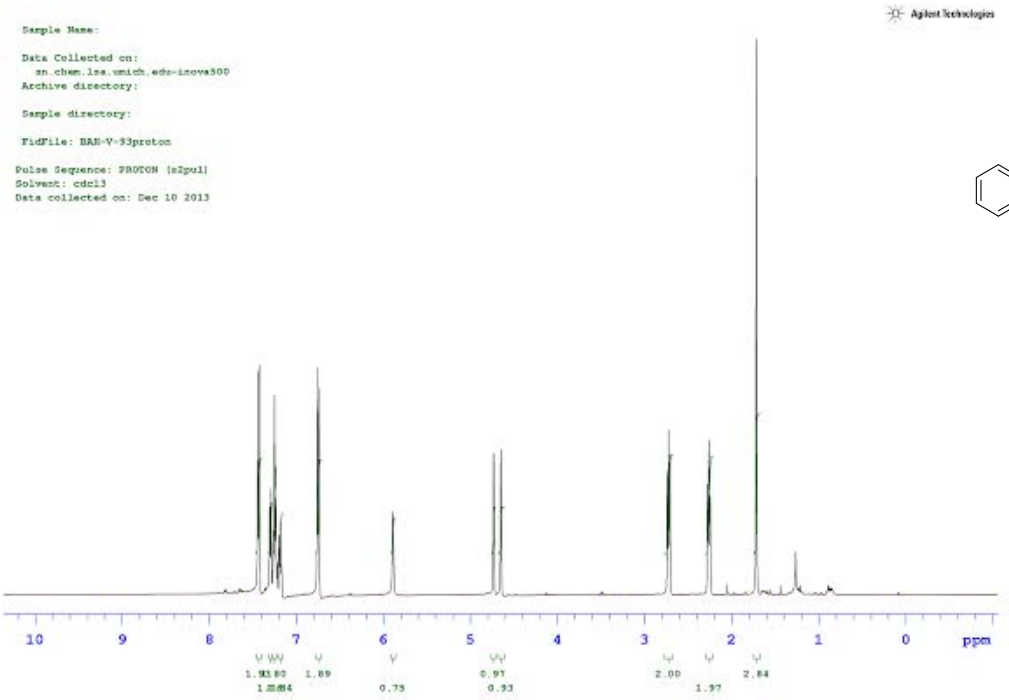
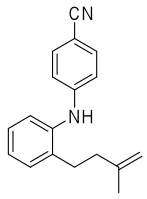
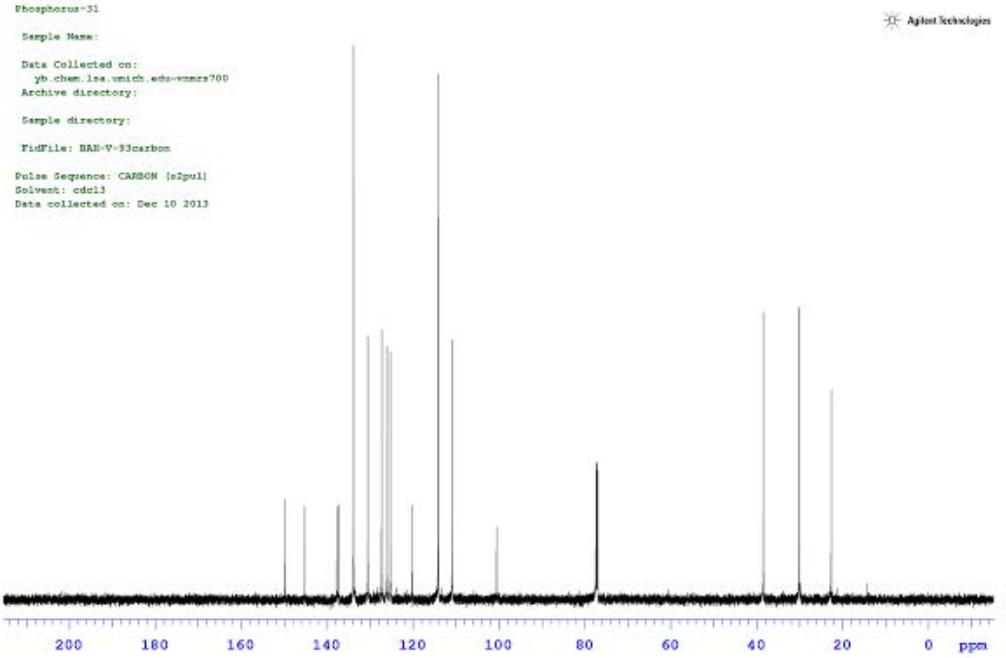
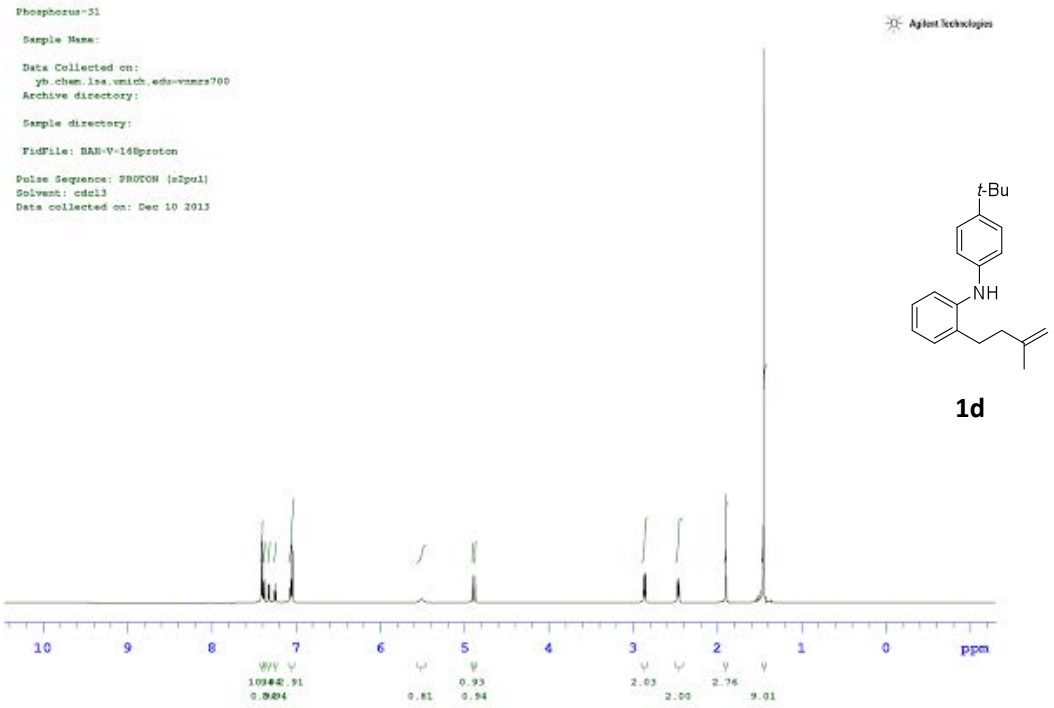
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-5-59carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 1 2014
Agilent Technologies
200 180 160 140 120 100 80 60 40 20 ppm


Agilent Technologies
Sample Name:
Data Collected on:
sn.chem.lsa.umich.edu-inova500
Archive directory:
Sample directory:
FidFile: BAN-V-93proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 10 2013
10
9
8
7
6
5
4
3
2
1
0
ppm
1.93
1.89
0.75
0.97
0.93
2.00
1.97
2.84

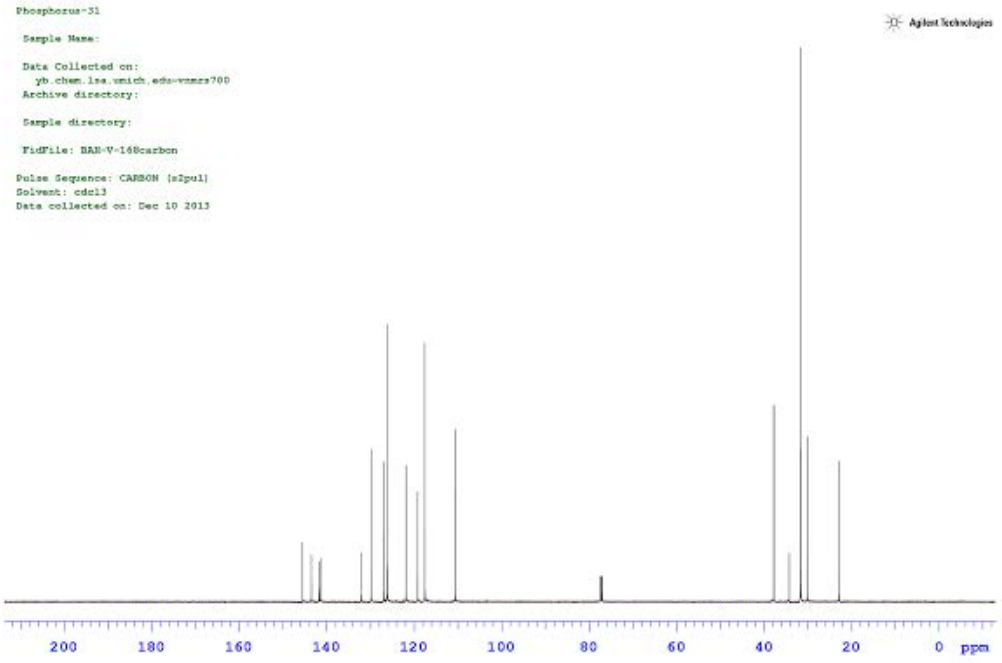

CN
NH

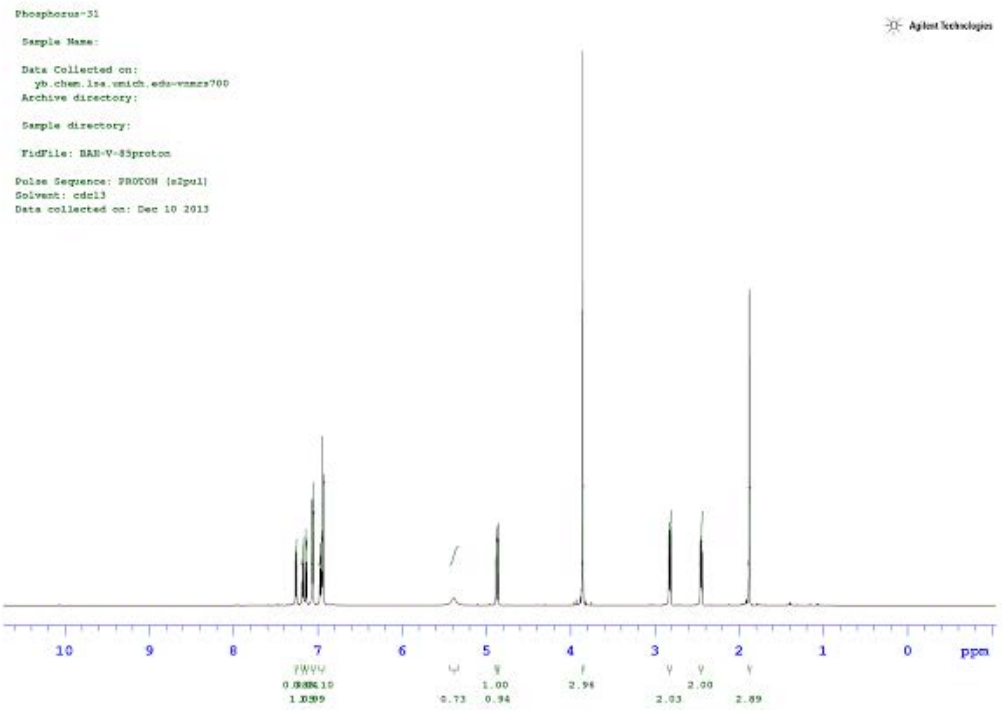
1c

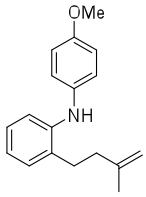

Phosphorus-31
Agilent Technologies
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-V-93carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Dec 10 2013
200
180
160
140
120
100
80
60
40
20
0
ppm


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-V-168proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 10 2013
Agilent Technologies
t-Bu
NH
10 9 8 7 6 5 4 3 2 1 0 ppm
0.81
0.93
0.94
2.03
2.00
2.76
9.01

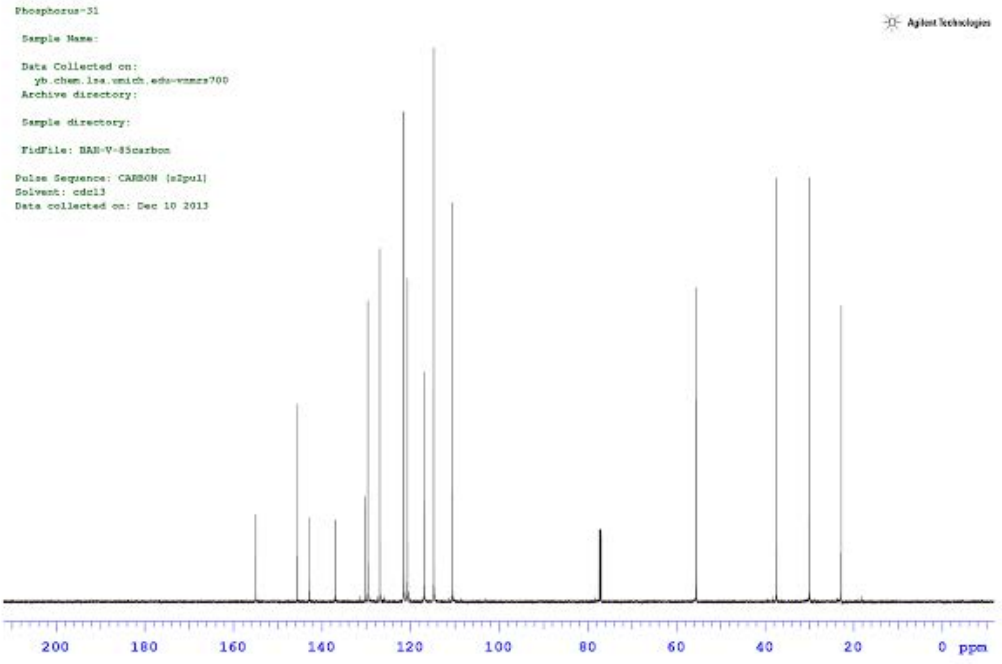
1d

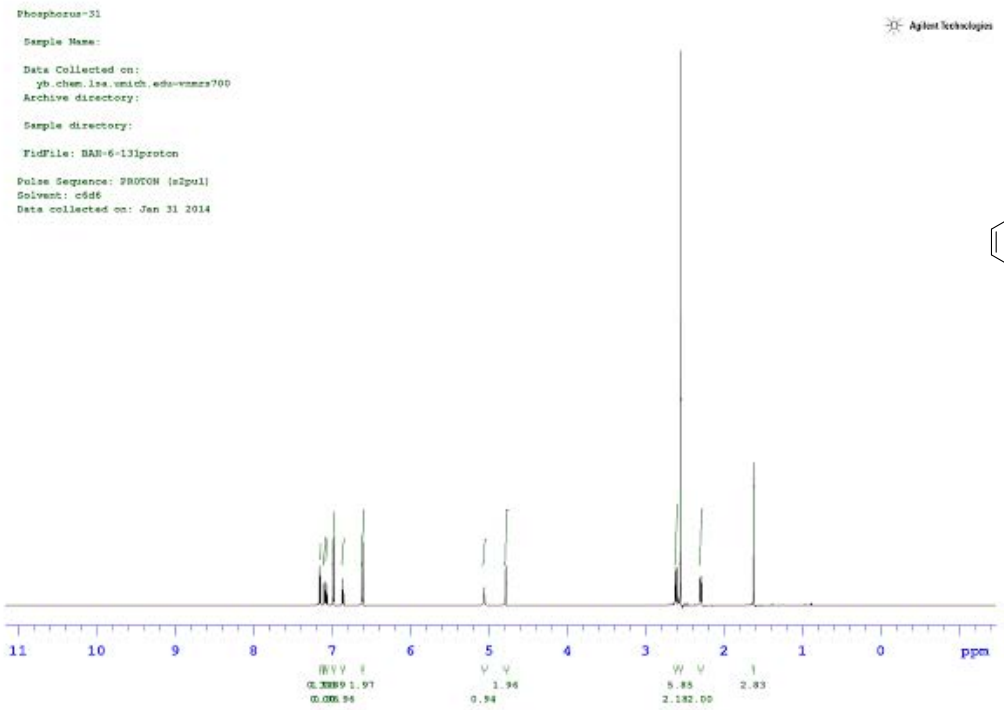

Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-V-168carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Dec 10 2013
Agilent Technologies
200 180 160 140 120 100 80 60 40 20 0 ppm

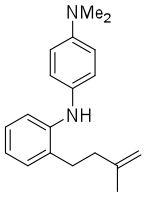

Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-V-85proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 10 2013
Agilent Technologies
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.96
2.00
0.73
0.94
2.03
2.89


OMe
NH

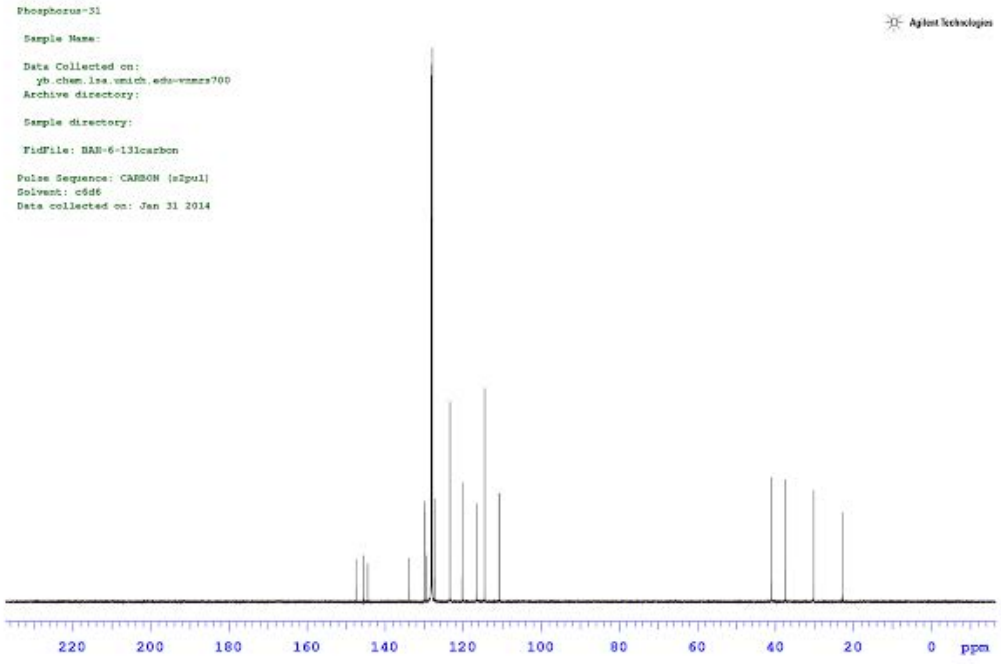
1b

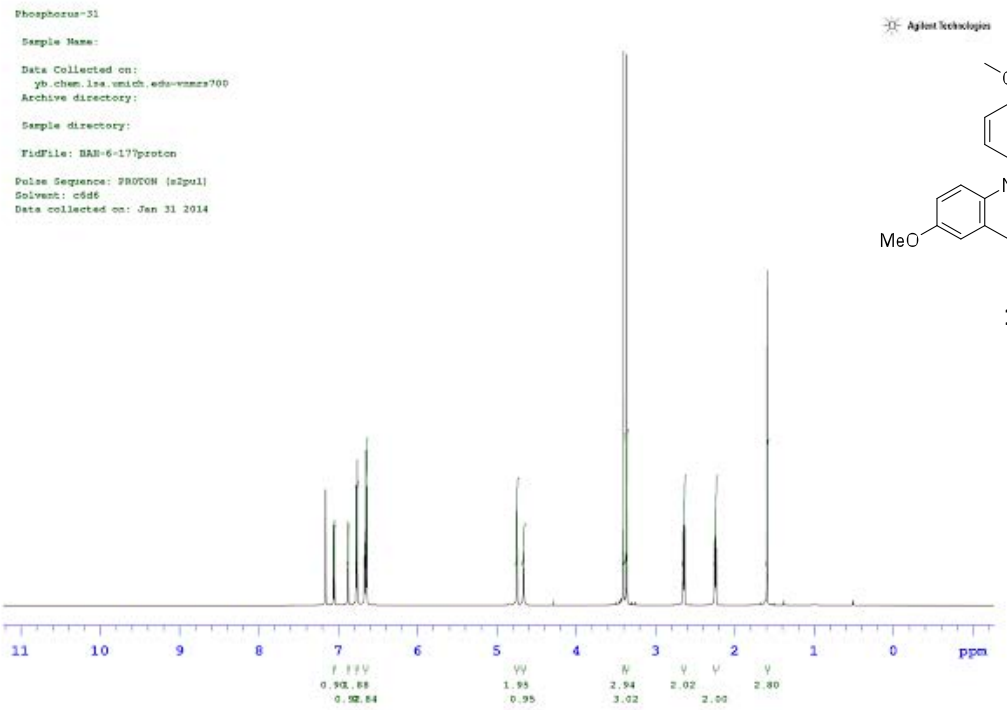

Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-V-85carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Dec 10 2013
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
0
ppm


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-131proton
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Jan 31 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.97
1.96
0.94
5.85
2.83
2.182.00


NMe2
NH

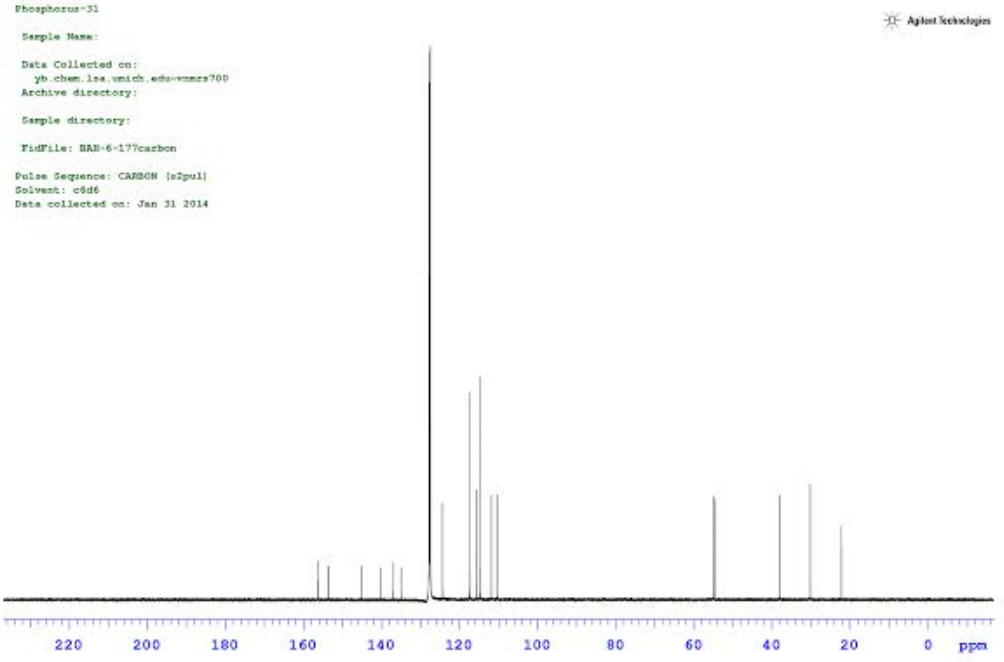
1e

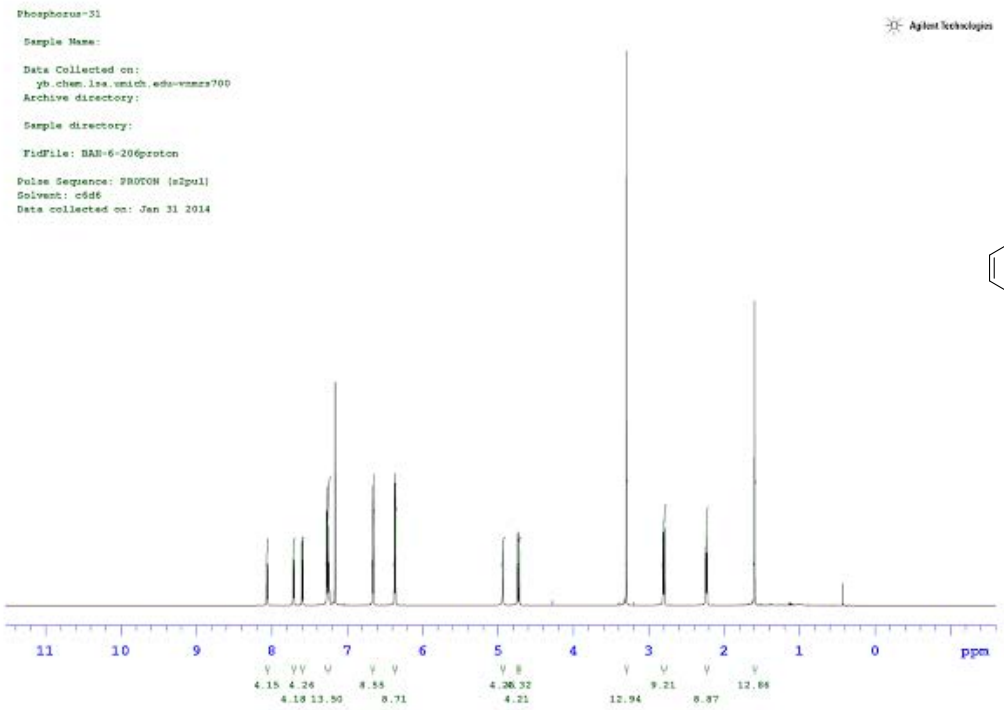

Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-131carbon
Pulse Sequence: CARBON (s2pul)
Solvent: c6d6
Data collected on: Jan 31 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm

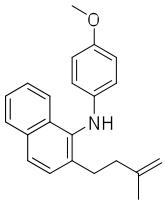

Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-177proton
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Jan 31 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.90 0.88
0.92 0.84
1.95
0.95
2.94
3.02
2.02
2.00
2.80

O
NH
MeO

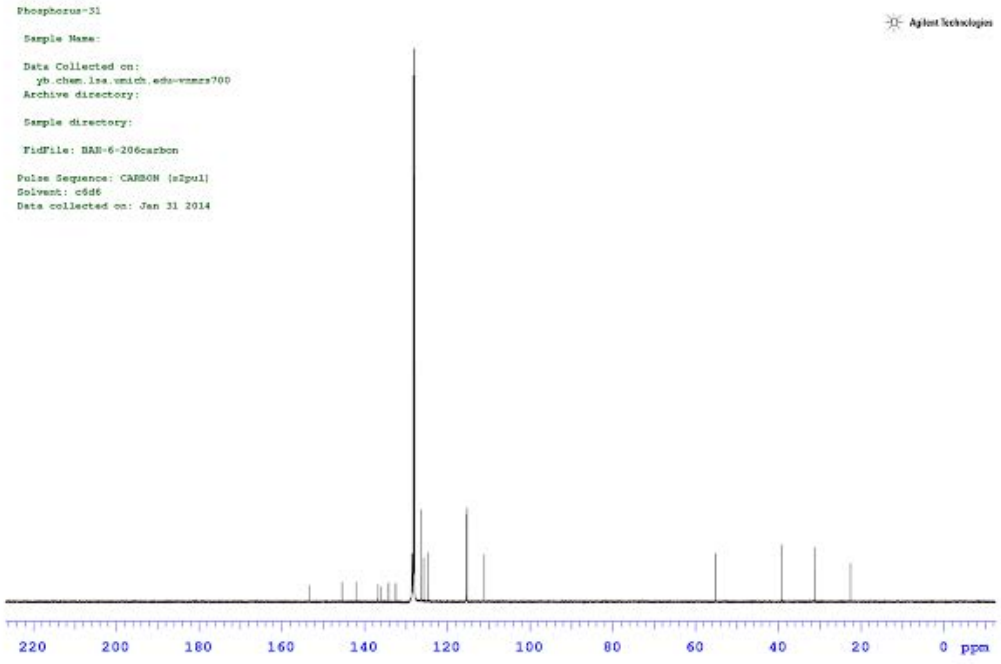
1f

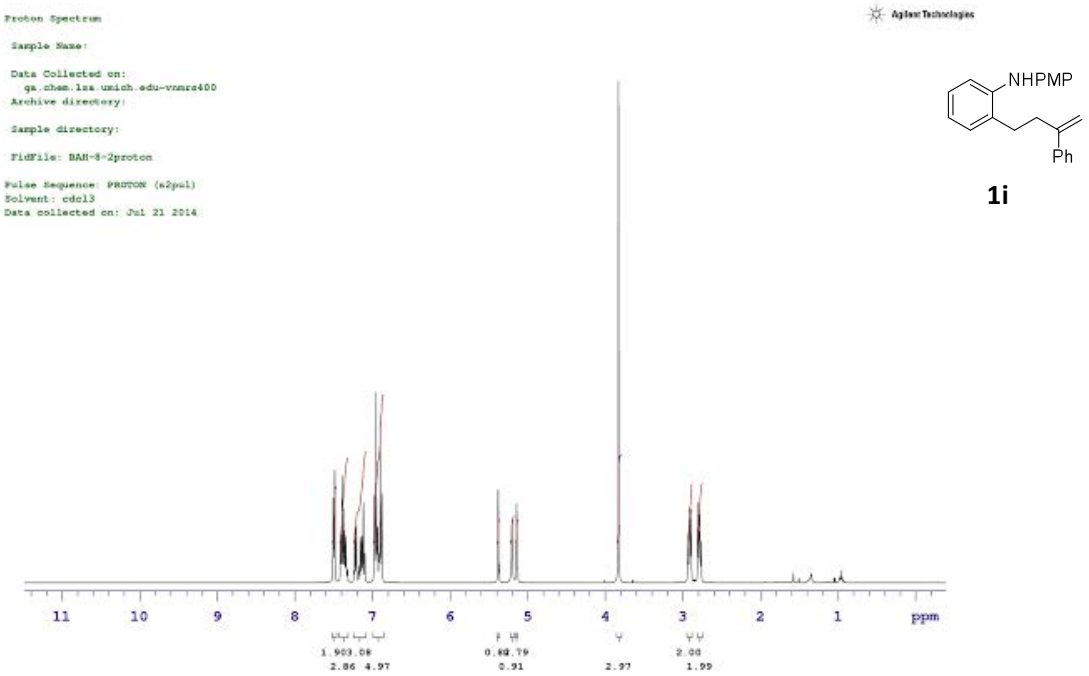

Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-177carbon
Pulse Sequence: CARBON (s2pul)
Solvent: c6d6
Data collected on: Jan 31 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm

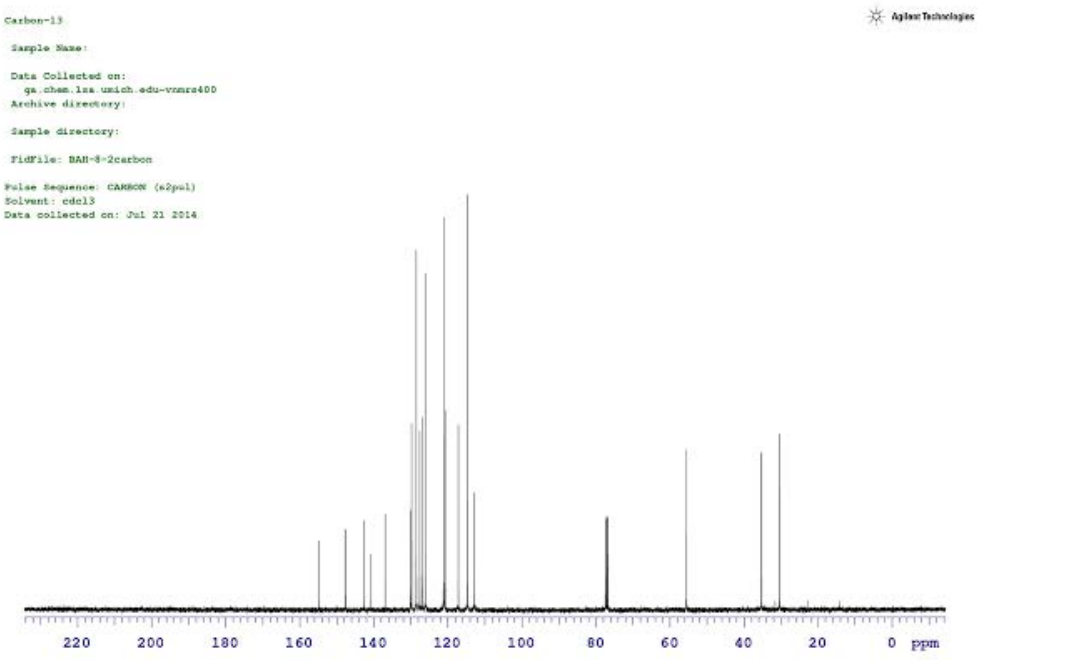

Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-206proton
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Jan 31 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
4.15 4.26 8.55 4.26 32 9.21 12.86
4.18 13.50 8.71 4.21 12.94 8.87

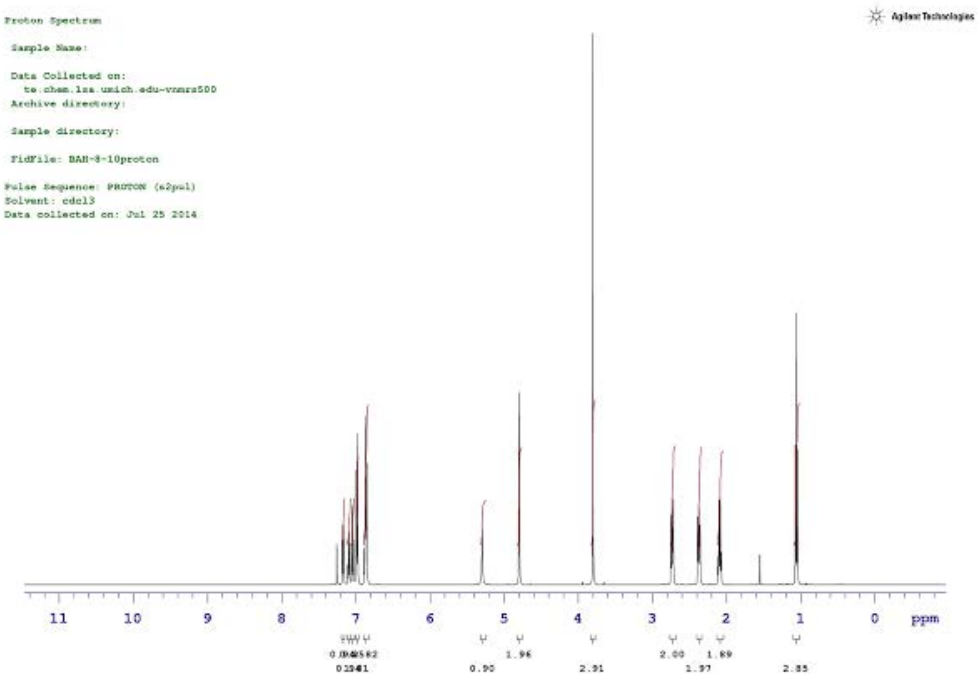

O
NH

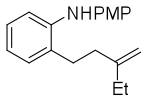
1g


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-206carbon
Pulse Sequence: CARBON (s2pul)
Solvent: c6d6
Data collected on: Jan 31 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm

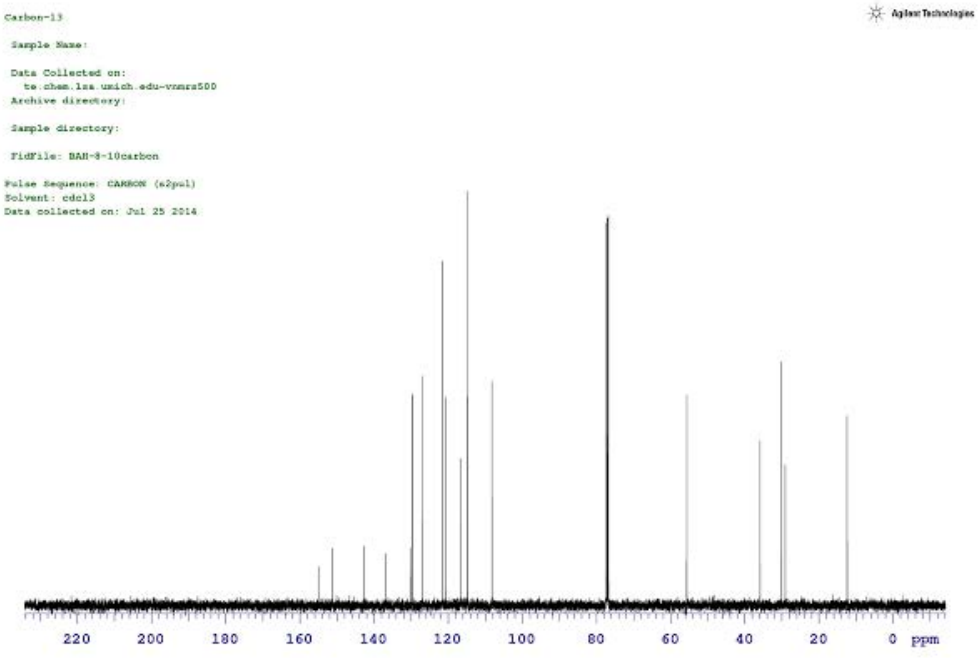

Proton Spectrum
Sample Name:
Data Collected on:
qa.chem.lsa.umich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-8-2proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 21 2014
Agilent Technologies
NHPMP
Ph
1i
11
10
9
8
7
6
5
4
3
2
1
ppm
1.903.08
2.06 4.97
0.89.79
0.91
2.97
2.00
1.99

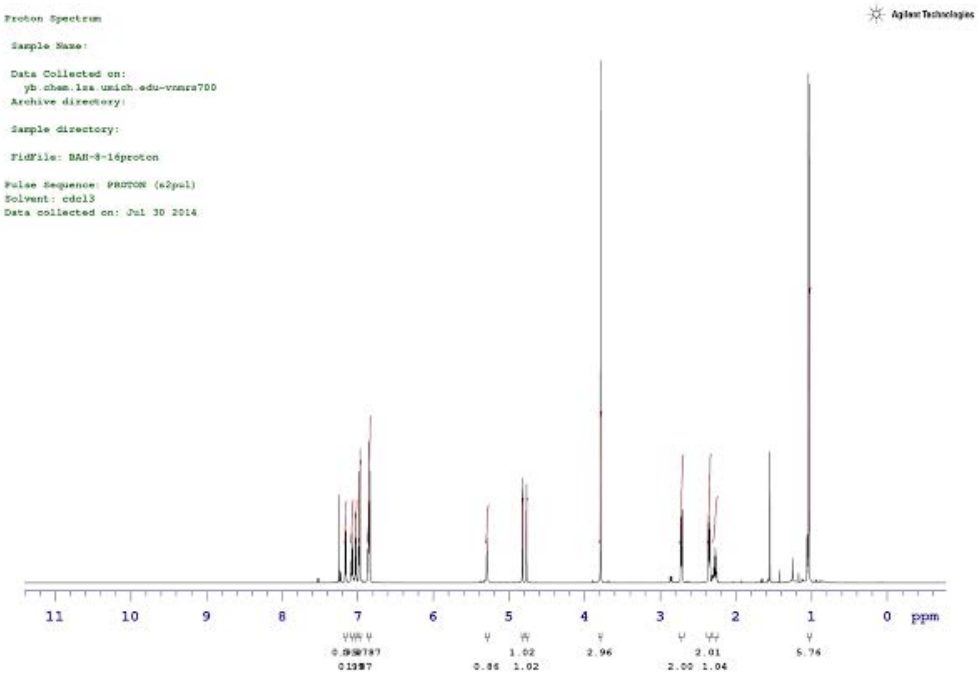

Carbon-13
Sample Name:
Data Collected on:
qa.chem.lsa.umich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-8-2carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 21 2014
Agilent Technologies
220
200
180
160
140
120
100
80
60
40
20
0
ppm

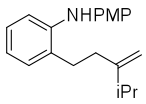

Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAH-8-10proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 25 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.96
2.91
2.00
1.89
1.97
2.85
0.90


NHPMP
Et

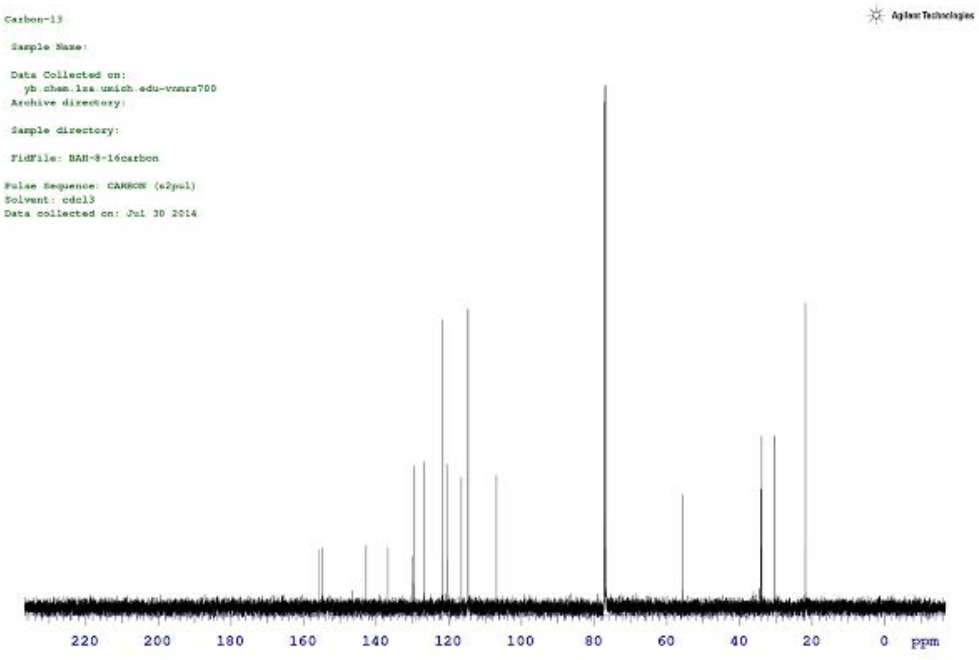
1h

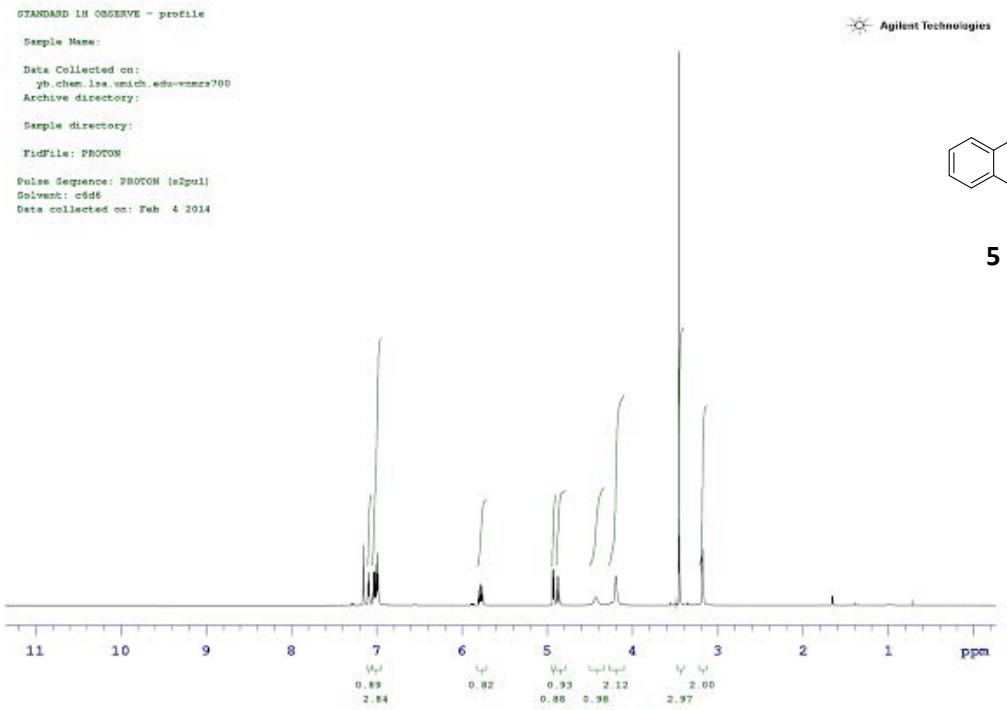

Carbon-13
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAH-8-10carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 25 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm

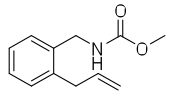

Proton Spectrum
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-8-16proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 30 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.02
2.96
2.01
5.76
0.86 1.02
2.00 1.04


NHPMP
iPr

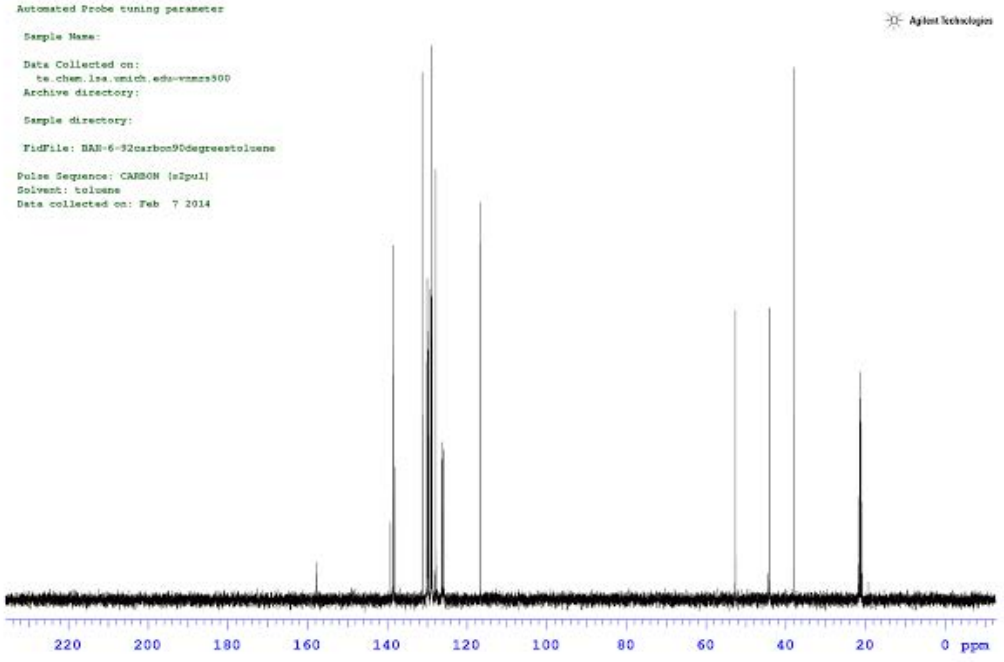
1j

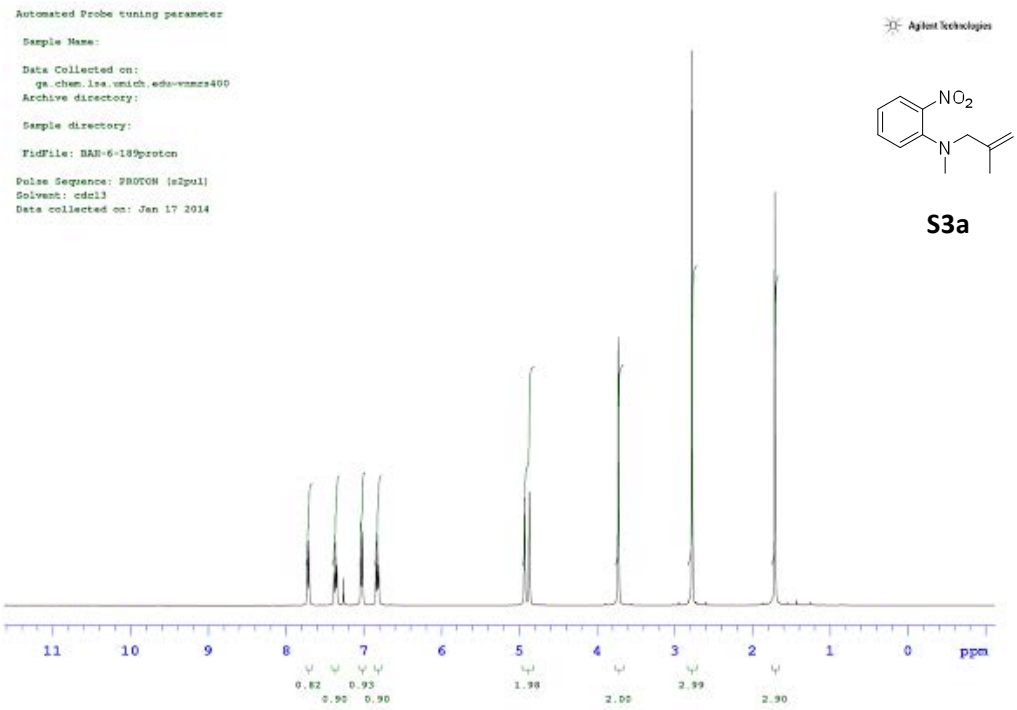

Carbon-13
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-8-16carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 30 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm


STANDARD 1H OBSERVE - profile
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Feb 4 2014
Agilent Technologies
11
10
9
8
7
6
5
4
3
2
1
ppm
0.89
2.84
0.82
0.93
0.88
2.12
0.98
2.00
2.97

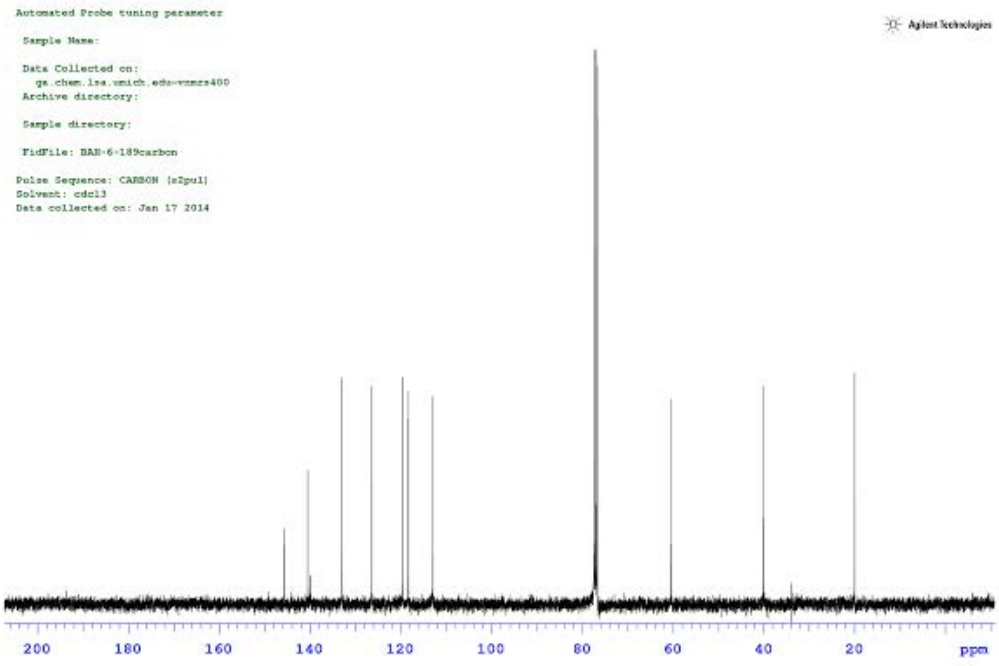


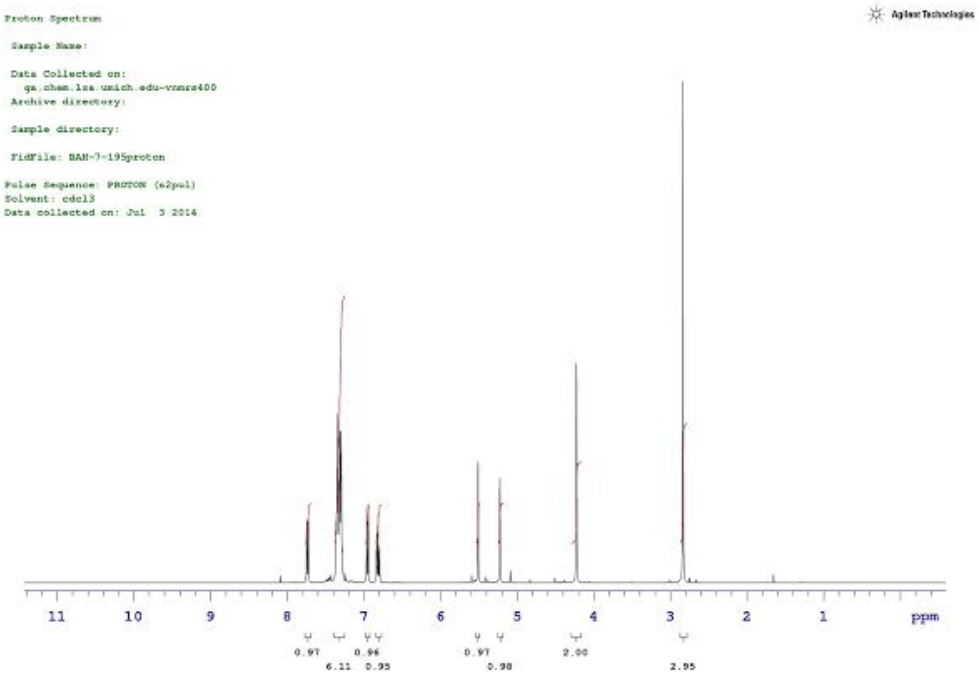
5

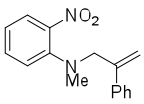

Automated Probe tuning parameter
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAR-6-92carbon90degreestoluene
Pulse Sequence: CARBON (s2pul)
Solvent: toluene
Data collected on: Feb 7 2014
Agilent Technologies
220
200
180
160
140
120
100
80
60
40
20
0 ppm


Automated Probe tuning parameter
Sample Name:
Data Collected on:
ga.chem.lsa.umich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAR-6-189proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 17 2014
Agilent Technologies
NO2
N
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.82 0.93
0.90 0.90
1.98
2.00
2.99
2.90

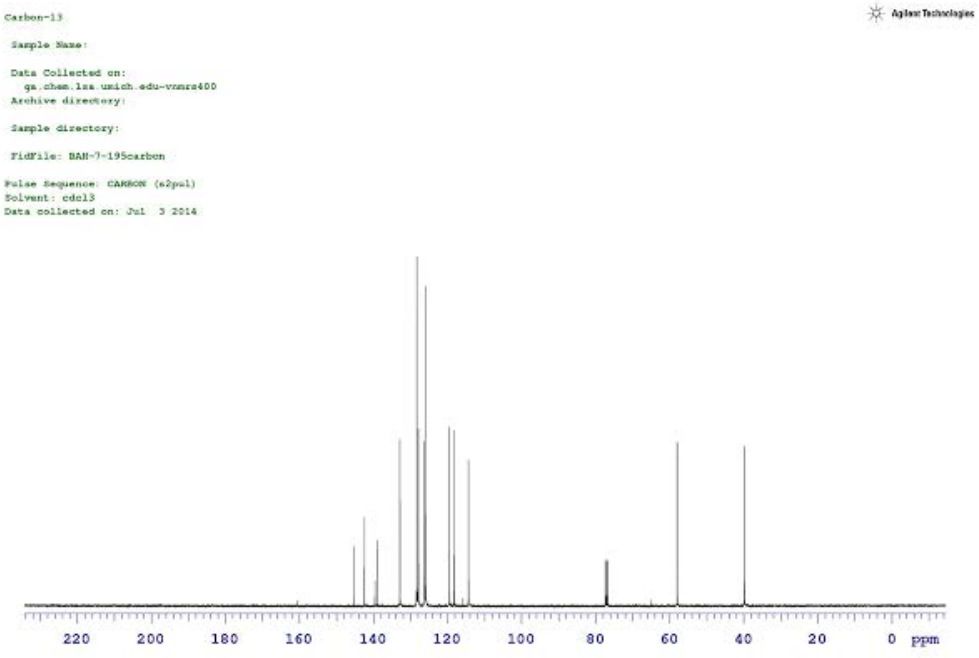
S3a

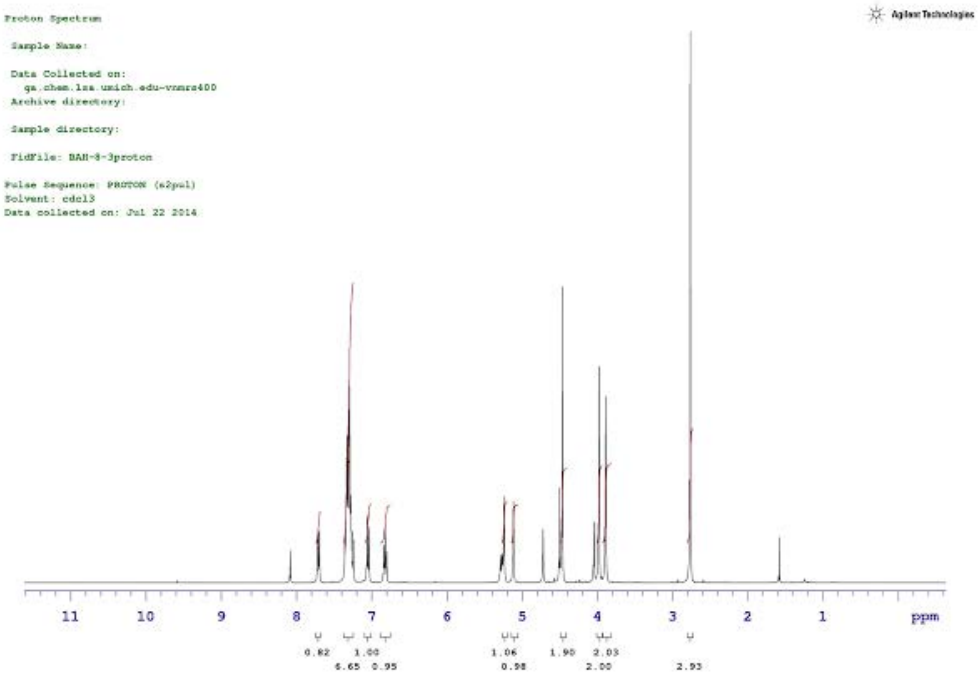

Automated Probe tuning parameter
Sample Name:
Data Collected on:
ga.chem.lsa.umich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAR-6-189carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 17 2014
Agilent Technologies
200 180 160 140 120 100 80 60 40 20 ppm

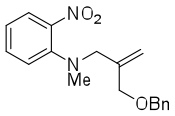

Proton Spectrum
Sample Name:
Data Collected on:
qa.chem.lsa.umich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-7-195proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 3 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 ppm
0.97 0.96 0.97 2.00
6.11 0.95 0.98 2.95


NO2
N
Me
Ph

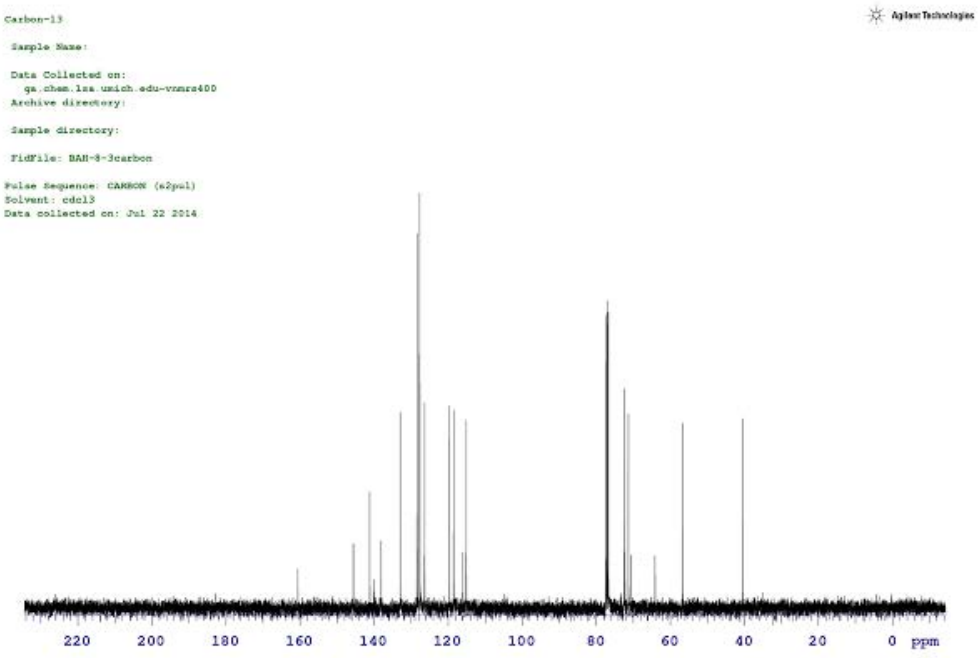
S3b

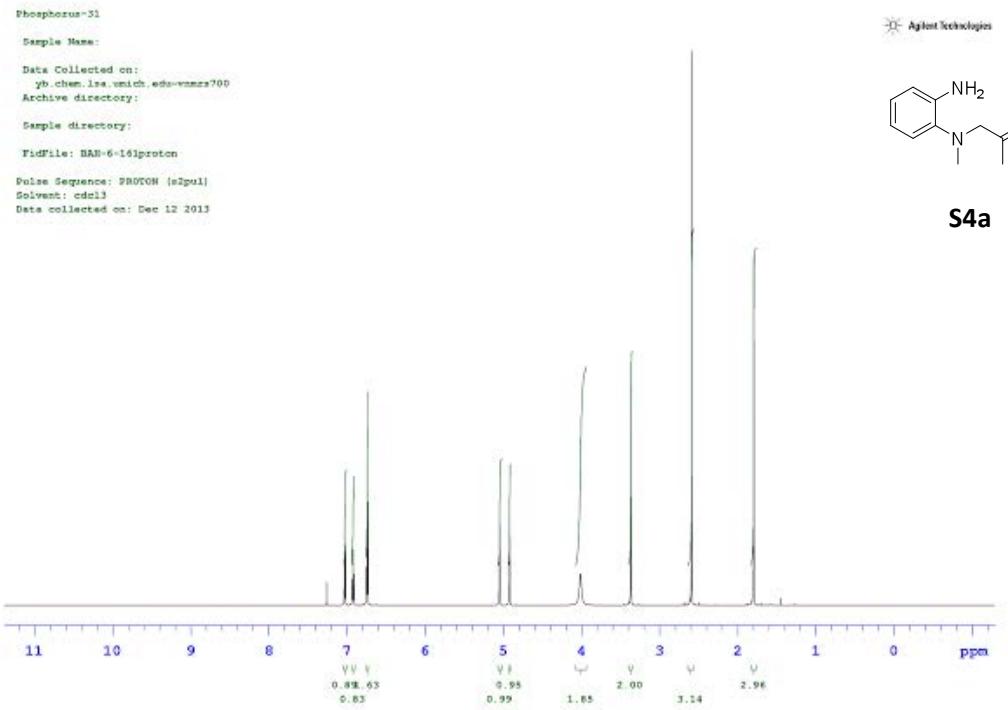

Carbon-13
Sample Name:
Data Collected on:
qa.chem.lsa.umich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-7-195carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 3 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm


Proton Spectrum
Sample Name:
Data Collected on:
qa.chem.lsa.umich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-8-3proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 22 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 ppm
0.82 1.00 1.06 1.90 2.03 2.93
6.65 0.95 0.98 2.00

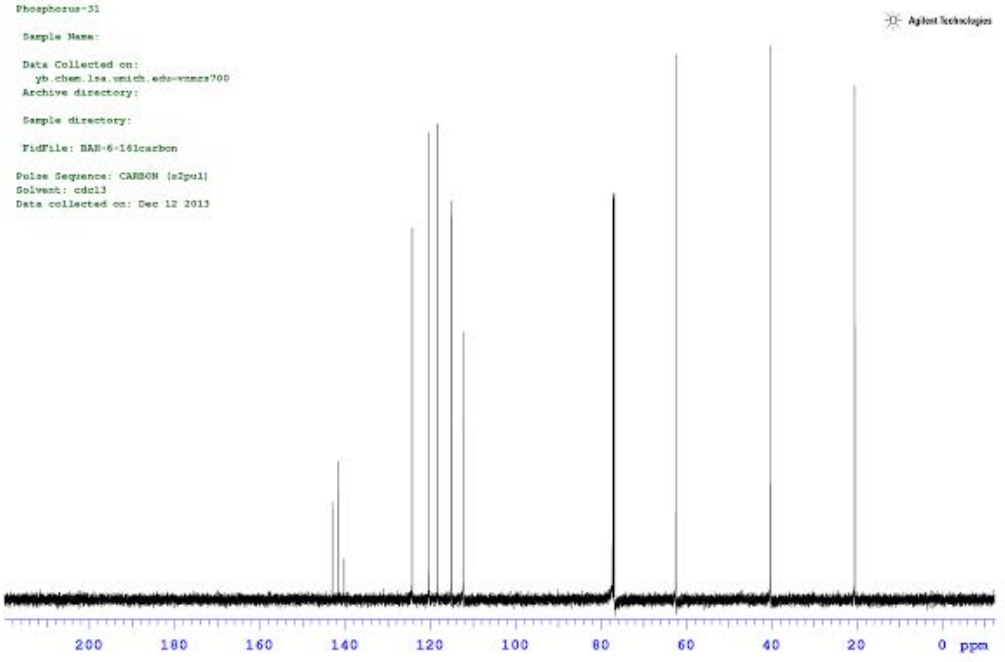

NO2
N
Me
OBn

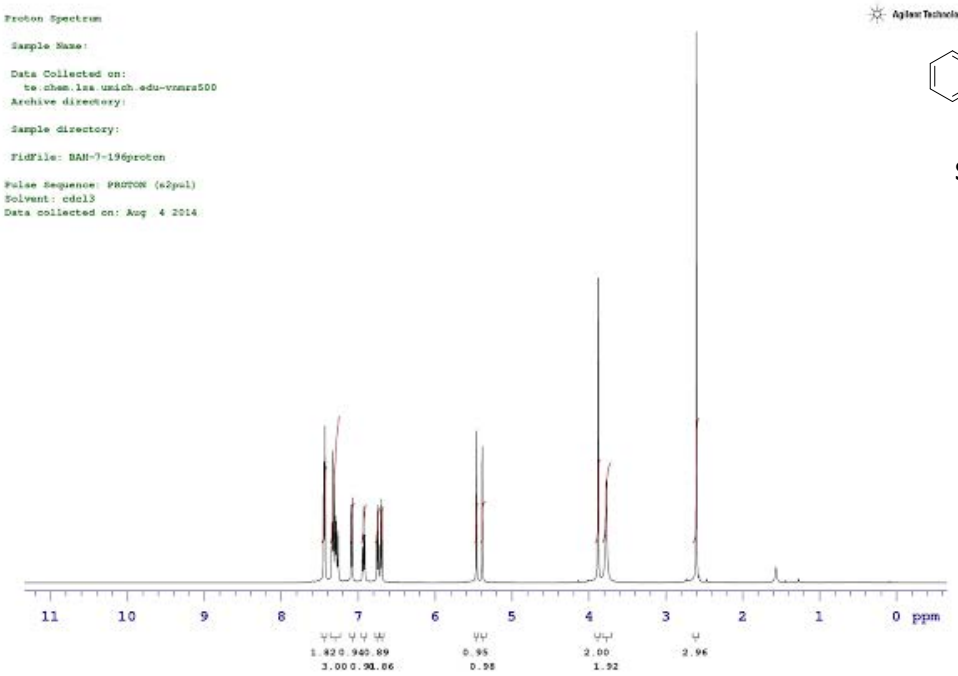
S3c


Carbon-13
Sample Name:
Data Collected on:
qa.chem.lsa.umich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-8-3carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 22 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm

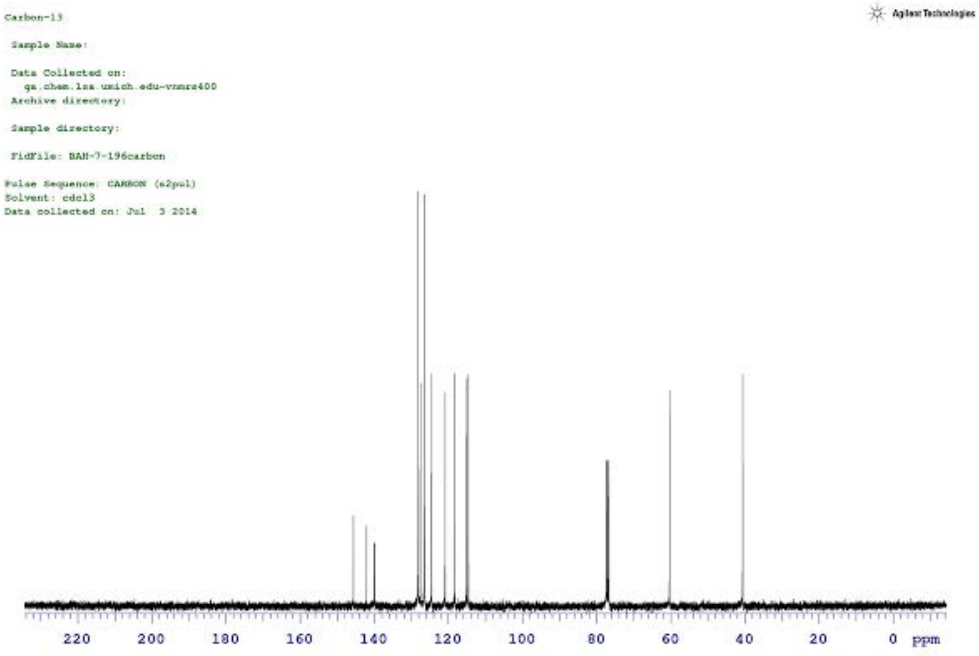

Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-161proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 12 2013
Agilent Technologies
NH2
N
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.83 0.99 1.85 3.14 2.96

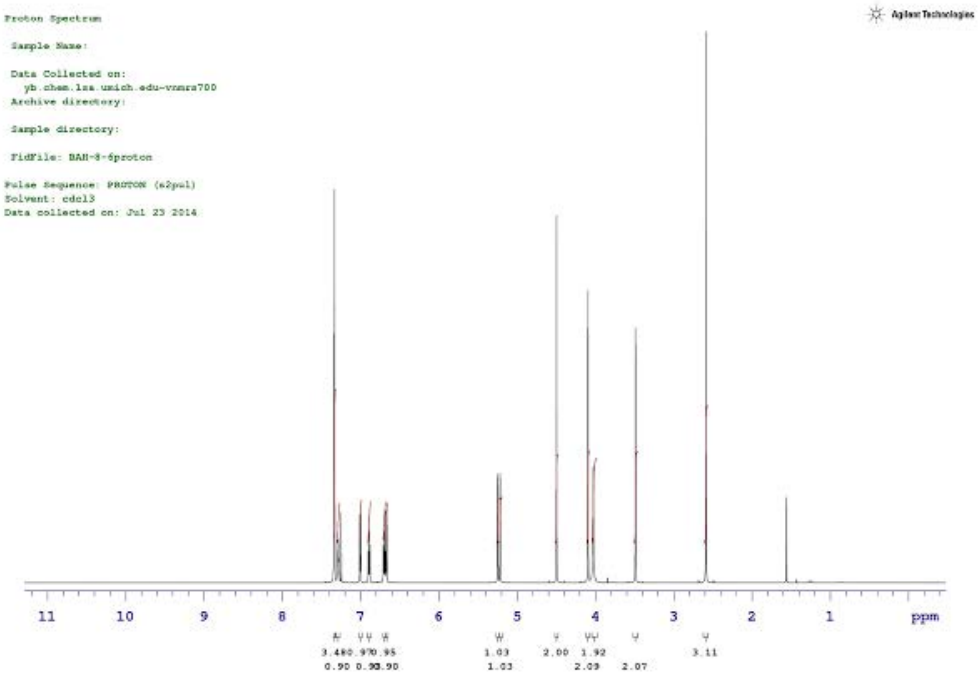
S4a

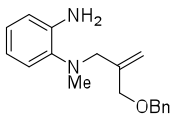

Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-161carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Dec 12 2013
Agilent Technologies
200 180 160 140 120 100 80 60 40 20 0 ppm


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAH-7-196proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Aug 4 2014
Agilent Technologies
NH2
N
Me
Ph
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.82 0.94 3.89 0.95 2.00 2.96
3.00 0.91 1.86 0.98 1.92

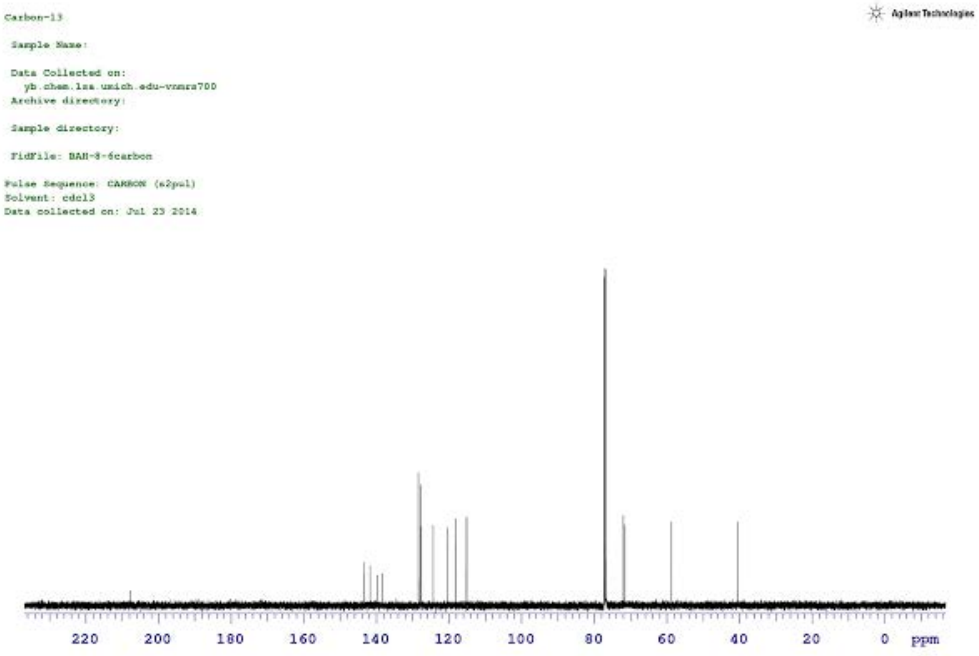
S4b

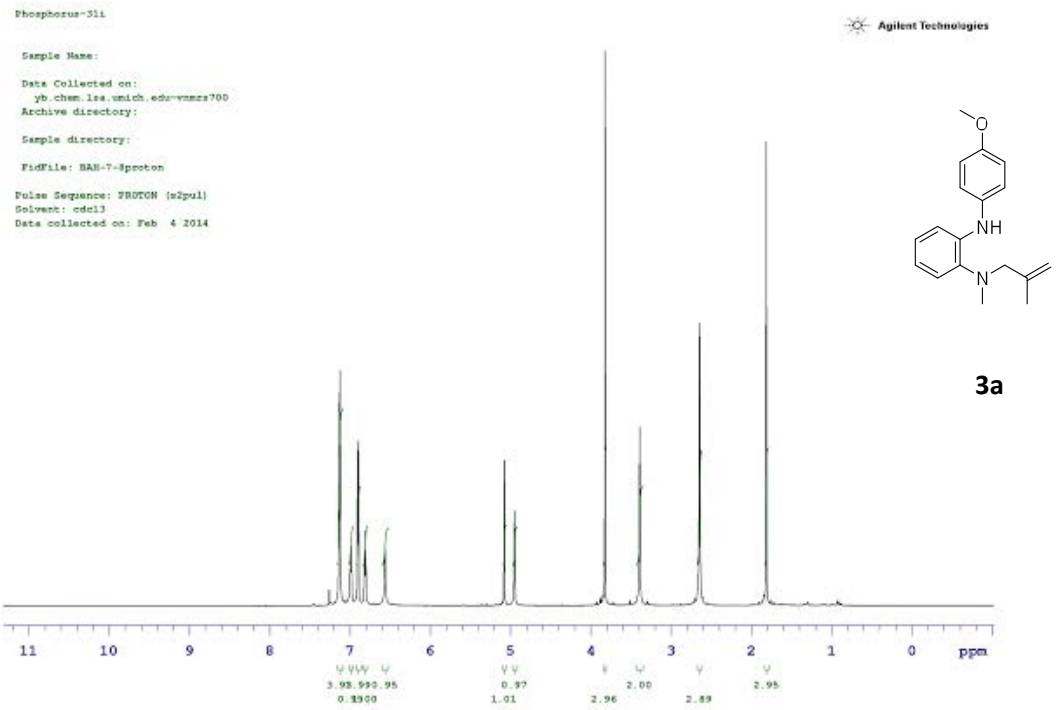

Carbon-13
Sample Name:
Data Collected on:
qa.chem.lsa.umich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-7-196carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 3 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm


Proton Spectrum
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-8-6proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 23 2014
Agilent Technologies
11
10
9
8
7
6
5
4
3
2
1
ppm
3.48
0.97
0.95
0.90
0.93
0.90
1.03
1.03
2.00
1.92
2.09
2.07
3.11

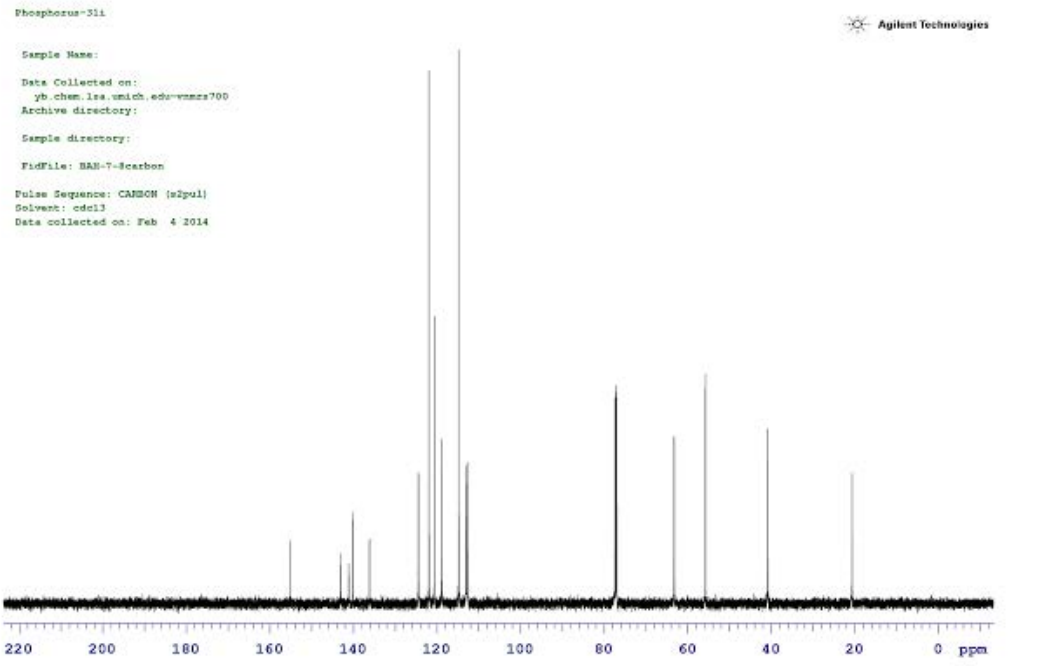

NH2
N
Me
OBn

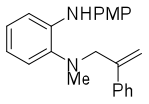
S4c


Carbon-13
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-8-6carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 23 2014
Agilent Technologies
220
200
180
160
140
120
100
80
60
40
20
0
ppm

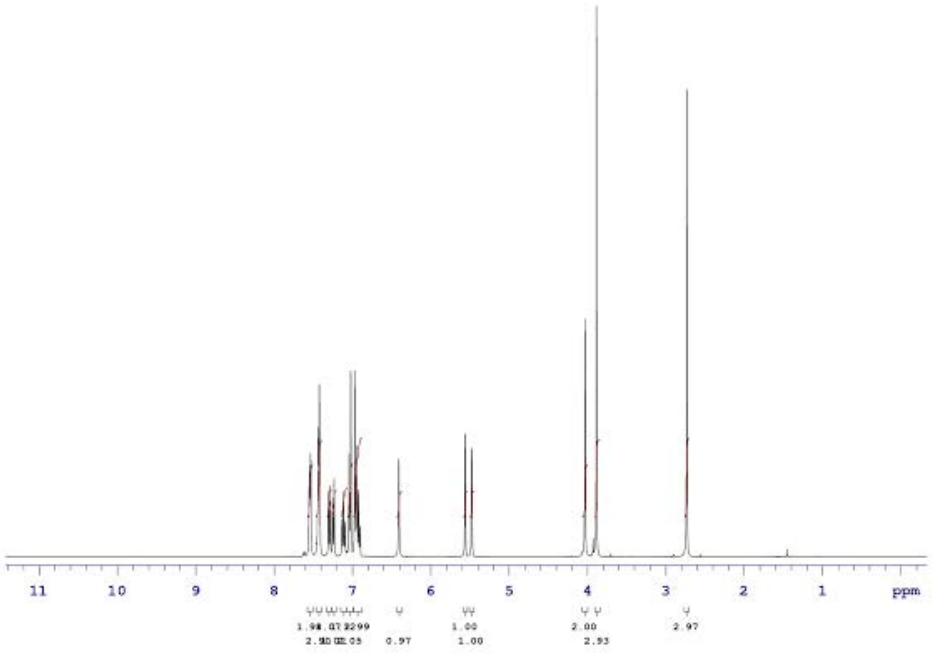

Phosphorus-31i
Agilent Technologies
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-7-8proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 4 2014
O
NH
N
11 10 9 8 7 6 5 4 3 2 1 0 ppm
3.98 3.99 0.95
0.97
2.00
2.95
1.01
2.96
2.89

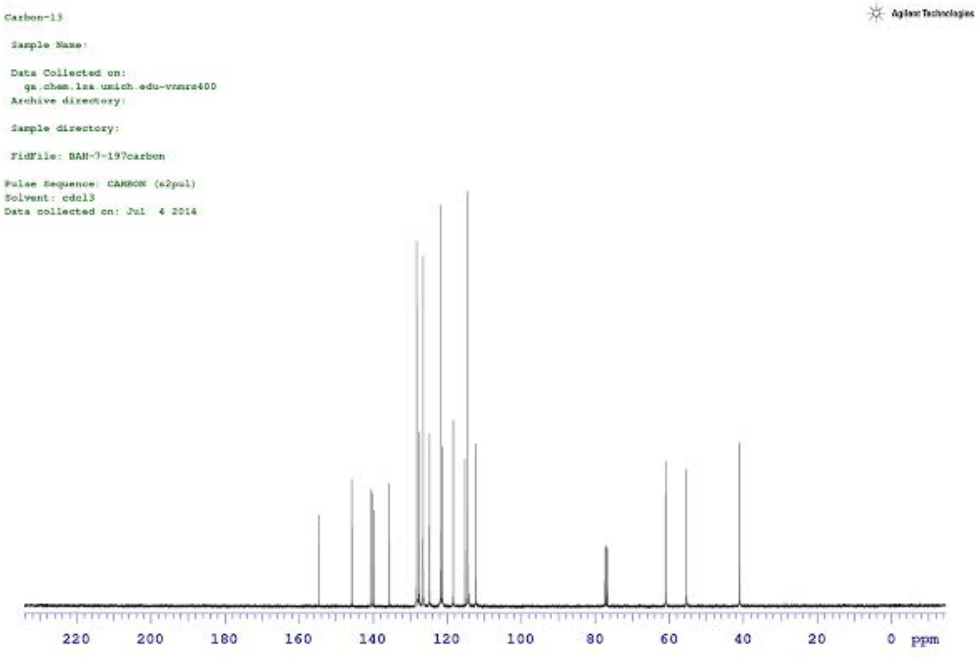
3a

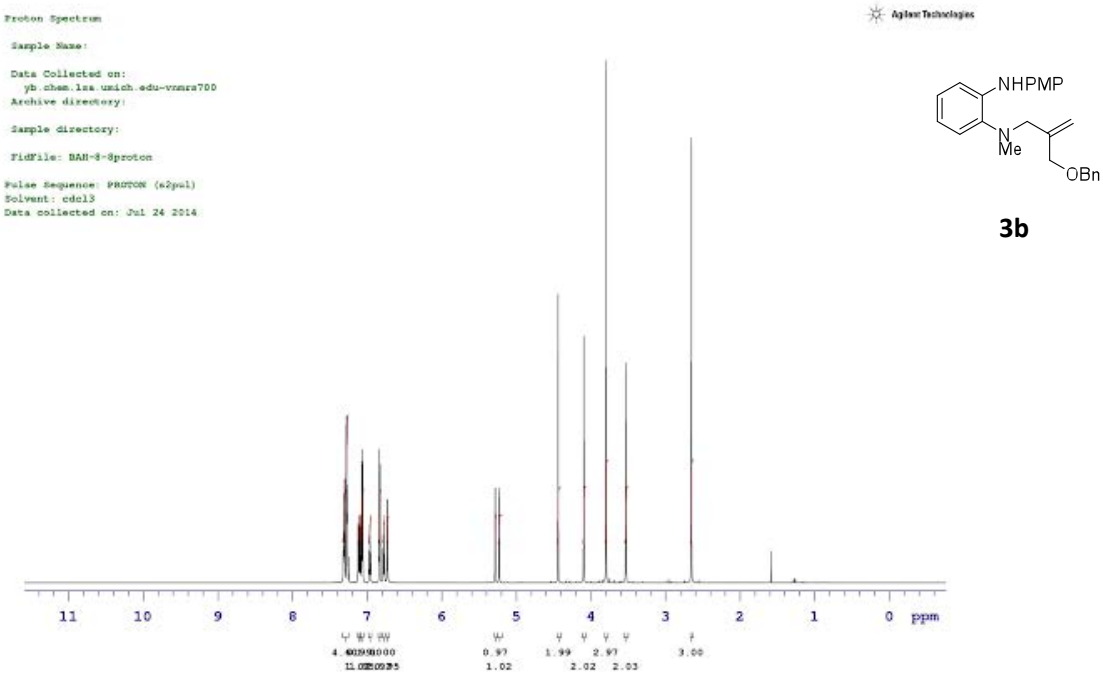

Phosphorus-31i
Agilent Technologies
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-7-8carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 4 2014
220 200 180 160 140 120 100 80 60 40 20 0 ppm

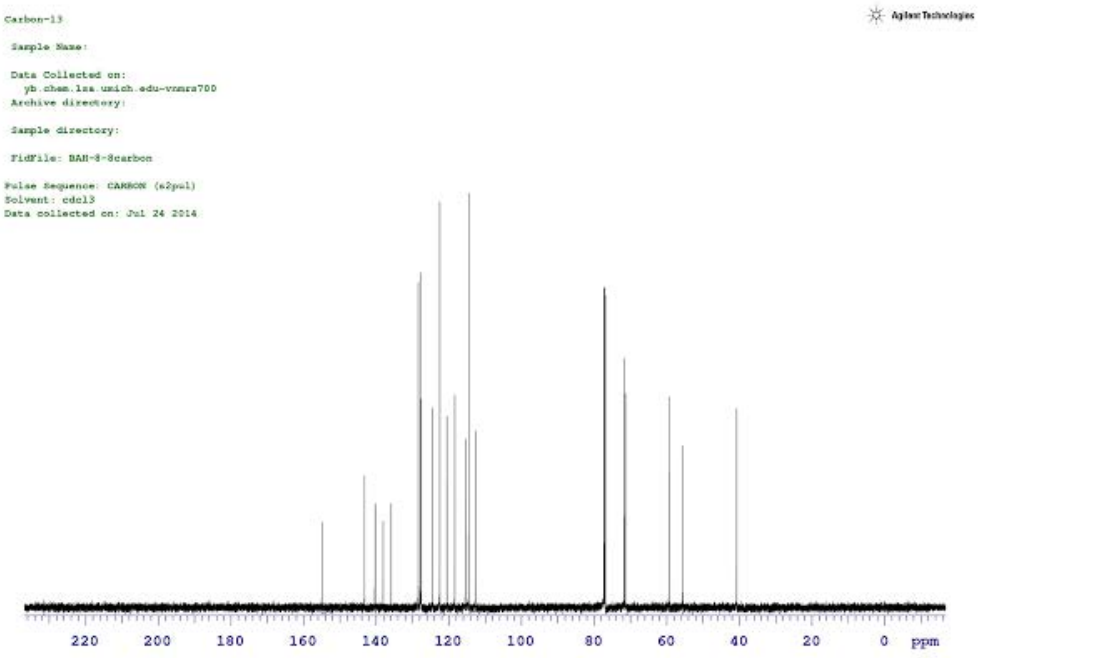

NHPMP
N
Me
Ph

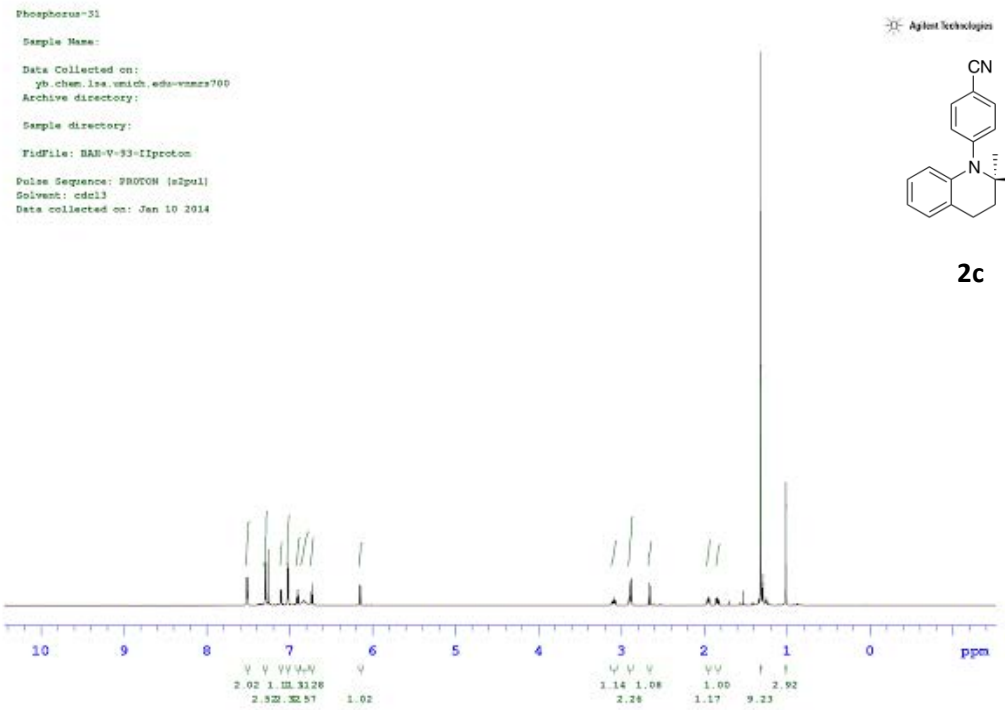
3c


11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.00
2.97
0.97
1.00
2.93


Carbon-13
Sample Name:
Data Collected on:
qa.chem.lsa.umich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAH-7-197carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 4 2014
Agilent Technologies
220
200
180
160
140
120
100
80
60
40
20
0
ppm

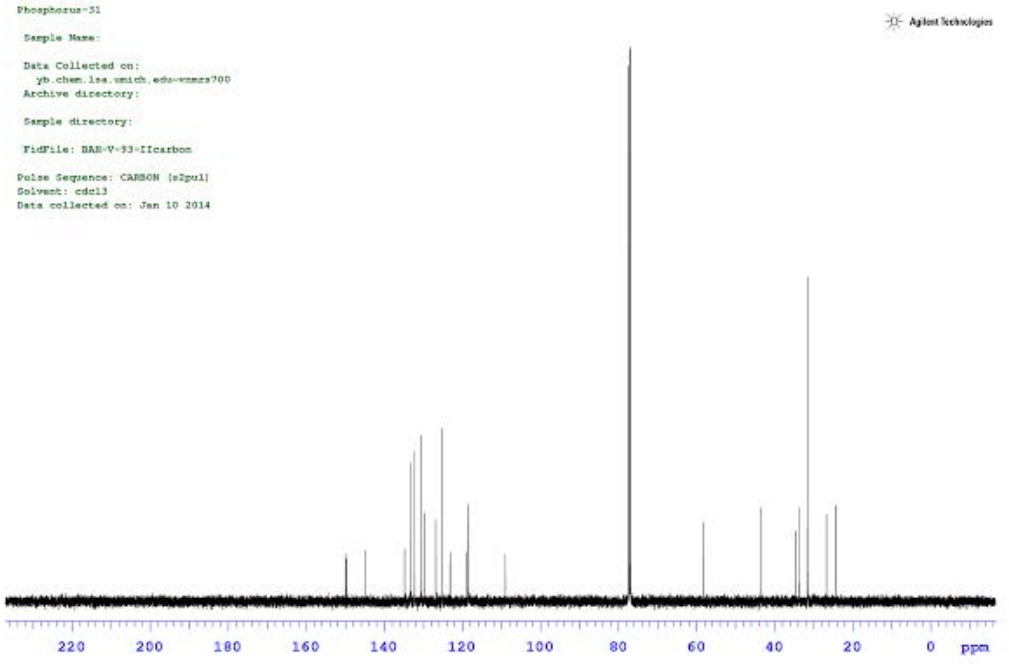

Proton Spectrum
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-8-8proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 24 2014
Agilent Technologies
NHPMP
N
Me
OBn
3b
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.97
1.99
2.97
3.00
1.02
2.02
2.03


Carbon-13
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-8-8carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 24 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-V-93-IIproton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 10 2014
Agilent Technologies
10 9 8 7 6 5 4 3 2 1 0 ppm
2.02 1.13 3.28 1.02
1.14 1.08 1.00 2.92
2.52 2.57 2.26 1.17 9.23

CN
N
tBu

2c


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-V-93-IIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 10 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm

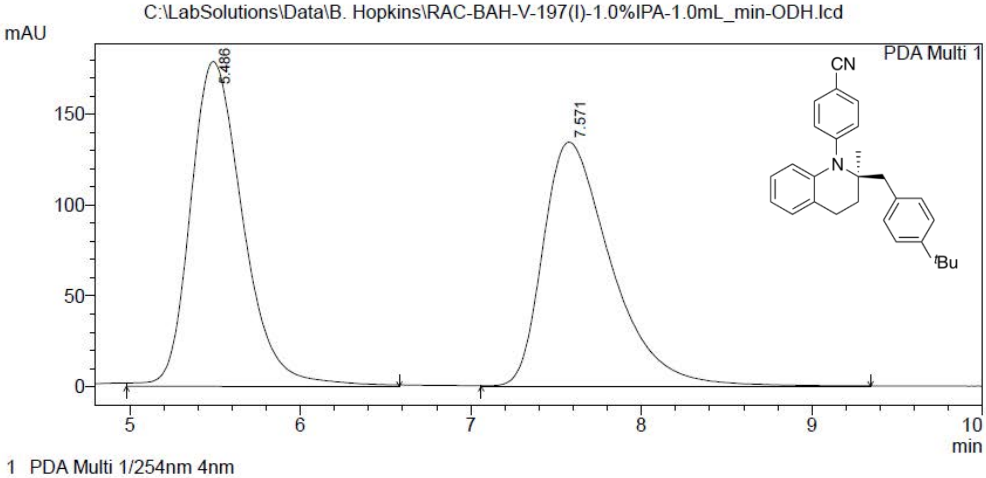
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-V-197(I)-1.0%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-V-197(I)-1.0%IPA-1.0mL_min-ODH
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-V-197(I)-1.0%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/23/2013 9:38:36 AM
Data Processed	: 7/23/2013 10:48:39 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-V-197(I)-1.0%IPA-1.0mL_min-ODH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.486	3847194	178927	50.635	57.087
2	7.571	3750760	134503	49.365	42.913
Total		7597954	313430	100.000	100.000

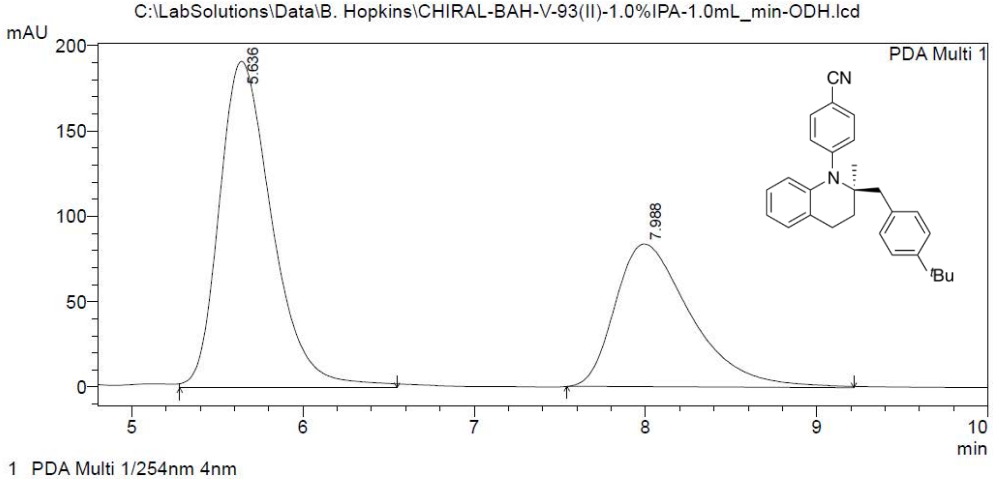
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-V-93(II)-1.0%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-V-93(II)-1.0%IPA-1.0mL_min-ODH
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-V-93(II)-1.0%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/23/2013 9:26:03 AM
Data Processed	: 7/23/2013 9:37:21 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-V-93(II)-1.0%IPA-1.0mL_min-ODH.lcd

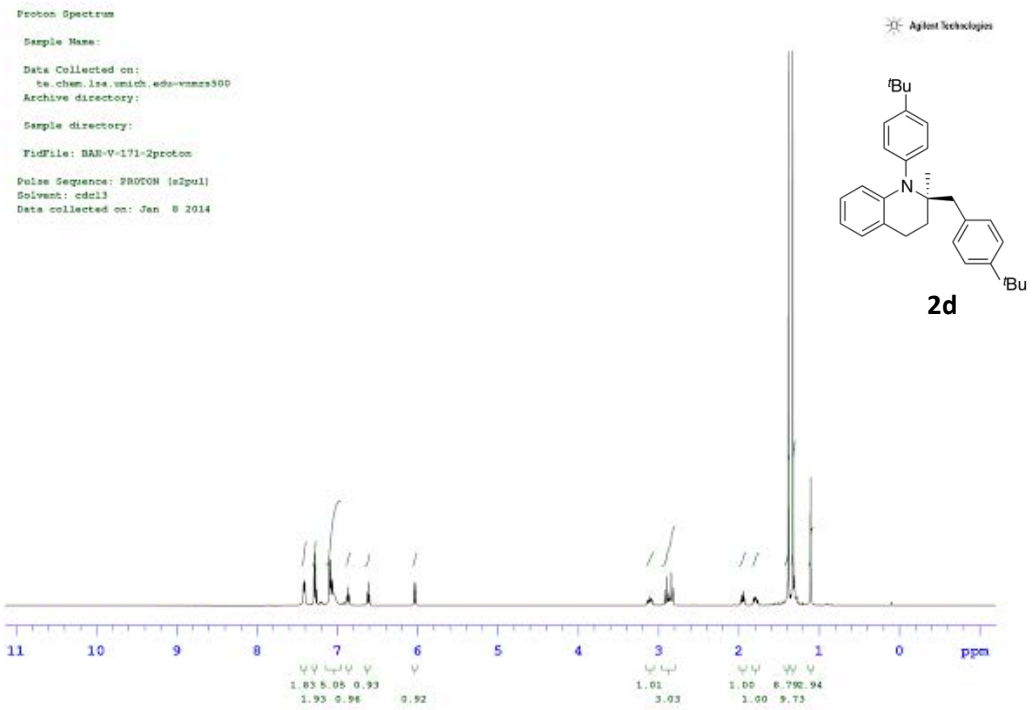


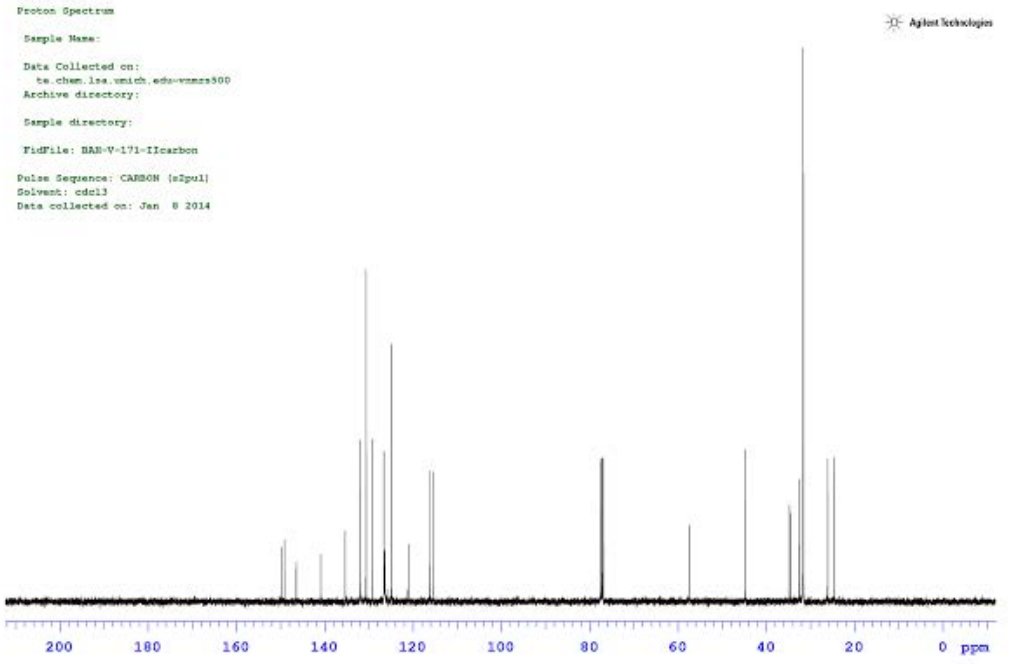
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.636	4103143	191125	61.514	69.617
2	7.988	2567136	83411	38.486	30.383
Total		6670278	274536	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-171-2proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 8 2014
Agilent Technologies
tBu
N
tBu
2d
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.83 5.05 0.93
1.93 0.96 0.92
1.01
3.03
1.00 8.79 2.94
1.00 9.73


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-171-IIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 8 2014
Agilent Technologies
200 180 160 140 120 100 80 60 40 20 0 ppm

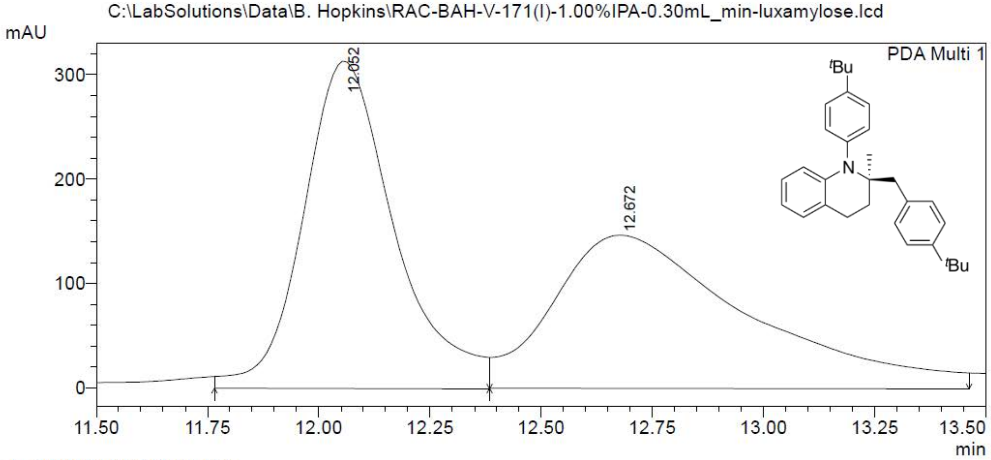
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-V-171(I)-1.00%IPA-0.30mL_min-luxamylose.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-V-171(I)-1.00%IPA-0.30mL_min-luxamylose
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-V-171(I)-1.00%IPA-0.30mL_min-luxamylose.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/4/2013 1:37:22 PM
Data Processed	: 7/4/2013 1:55:13 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-V-171(I)-1.00%IPA-0.30mL_min-luxamylose.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

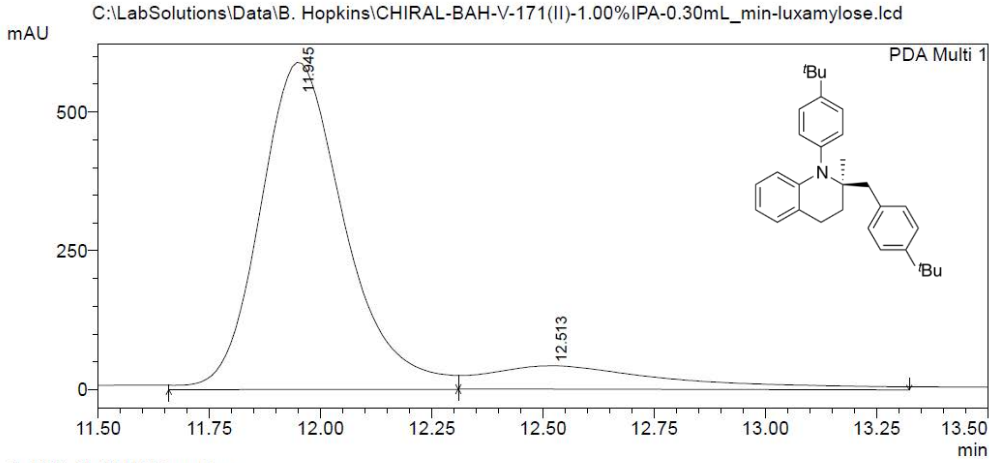
Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.052	4472534	313350	49.934	68.128
2	12.672	4484322	146593	50.066	31.872
Total		8956856	459943	100.000	100.000

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

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-V-171(II)-1.00%IPA-0.30mL_min-luxamylose.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-V-171(II)-1.00%IPA-0.30mL_min-luxamylose
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-V-171(II)-1.00%IPA-0.30mL_min-luxamylose.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/4/2013 2:01:40 PM
Data Processed	: 7/4/2013 2:34:09 PM

<Chromatogram>

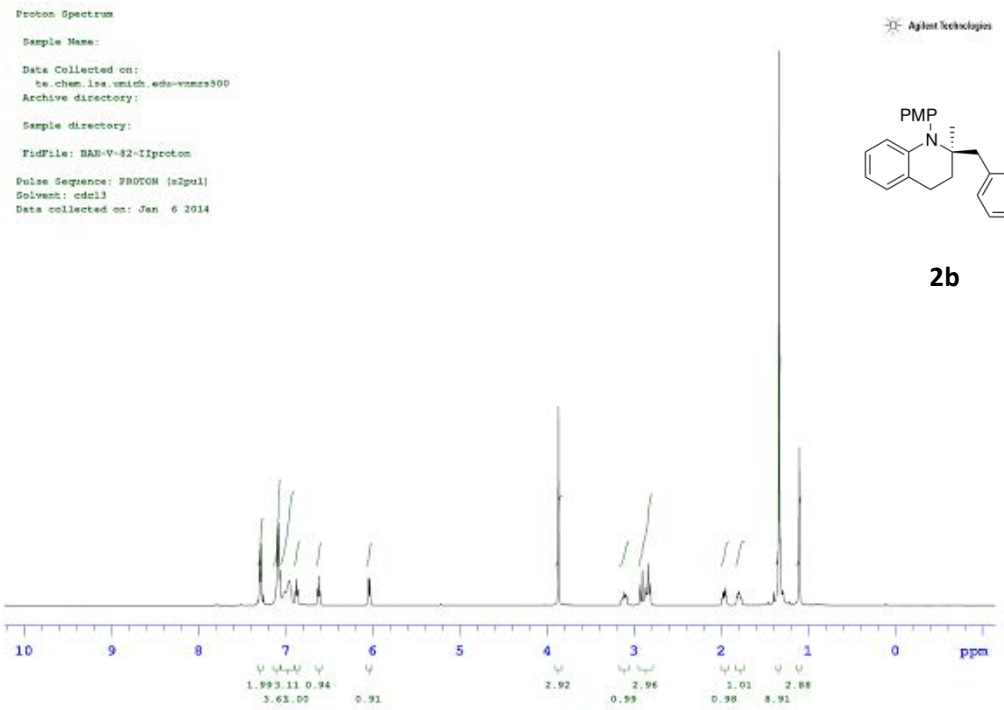


1 PDA Multi 1/254nm 4nm

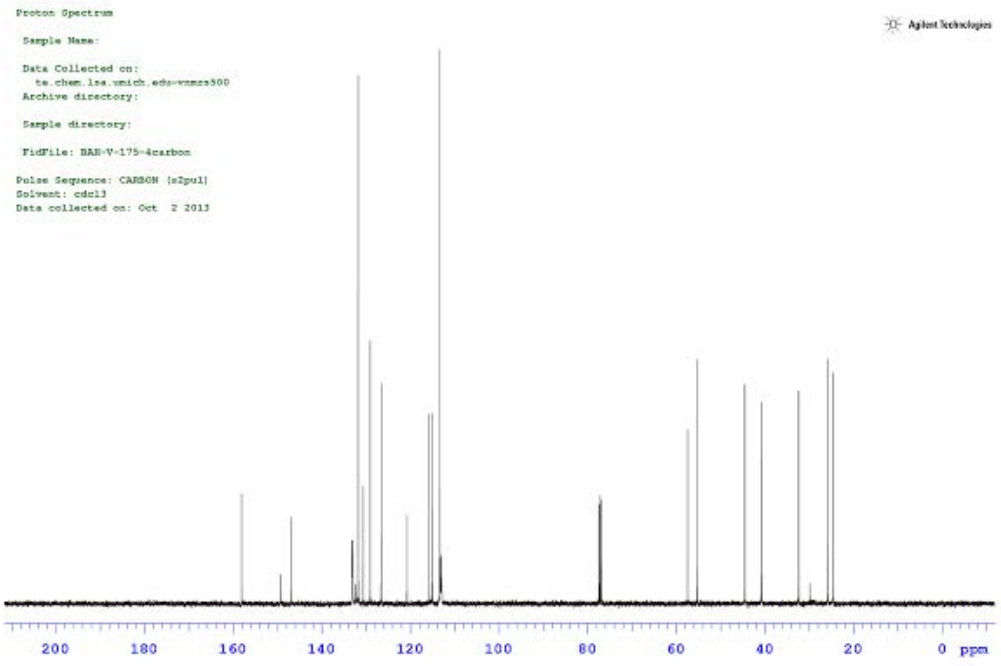
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.945	7822619	589004	86.516	93.366
2	12.513	1219154	41852	13.484	6.634
Total		9041774	630856	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-82-IIproton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 6 2014
Agilent Technologies
PMP
N
tBu
10
9
8
7
6
5
4
3
2
1
0
ppm
1.99
3.11
0.94
3.63
0.91
2.92
2.96
0.99
1.01
0.98
2.88
8.91

2b


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-175-4carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2013
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
0
ppm

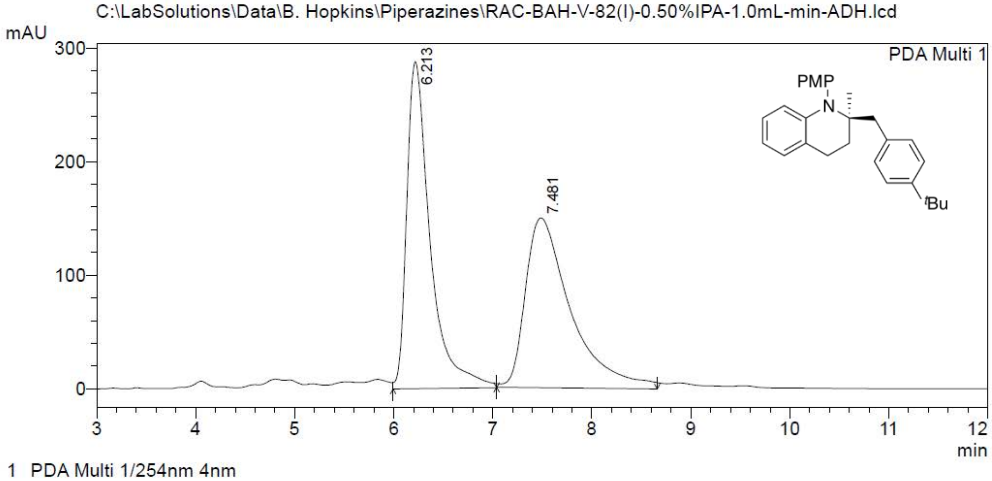
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-V-82(I)-0.50%IPA-1.0mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-V-82(I)-1.0%IPA-1.0mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-V-82(I)-0.50%IPA-1.0mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/7/2014 10:58:24 AM
Data Processed	: 1/7/2014 11:19:41 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-V-82(I)-0.50%IPA-1.0mL-min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.213	4726262	288069	49.830	65.849
2	7.481	4758560	149401	50.170	34.151
Total		9484822	437469	100.000	100.000

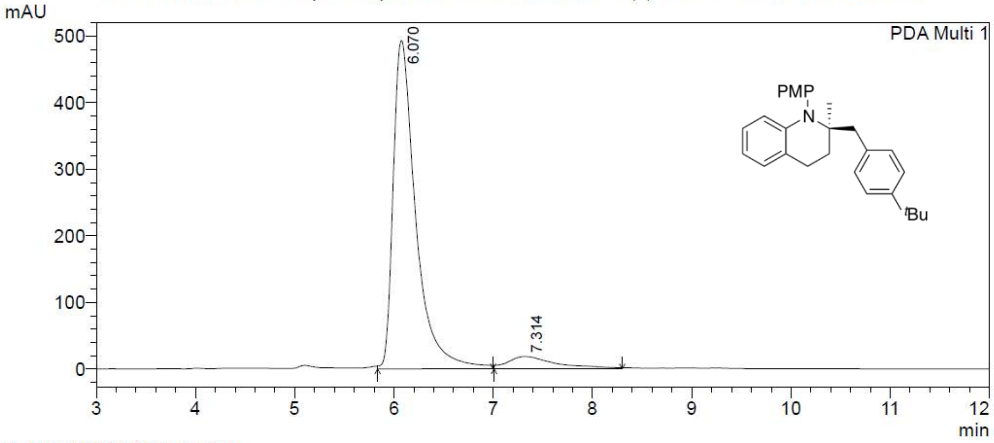
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-V-82(II)-0.50%IPA-1.0mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-V-82(II)-0.50%IPA-1.0mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-V-82(II)-0.50%IPA-1.0mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/7/2014 11:25:39 AM
Data Processed	: 1/7/2014 12:00:39 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-V-82(II)-0.50%IPA-1.0mL-min-ADH.lcd

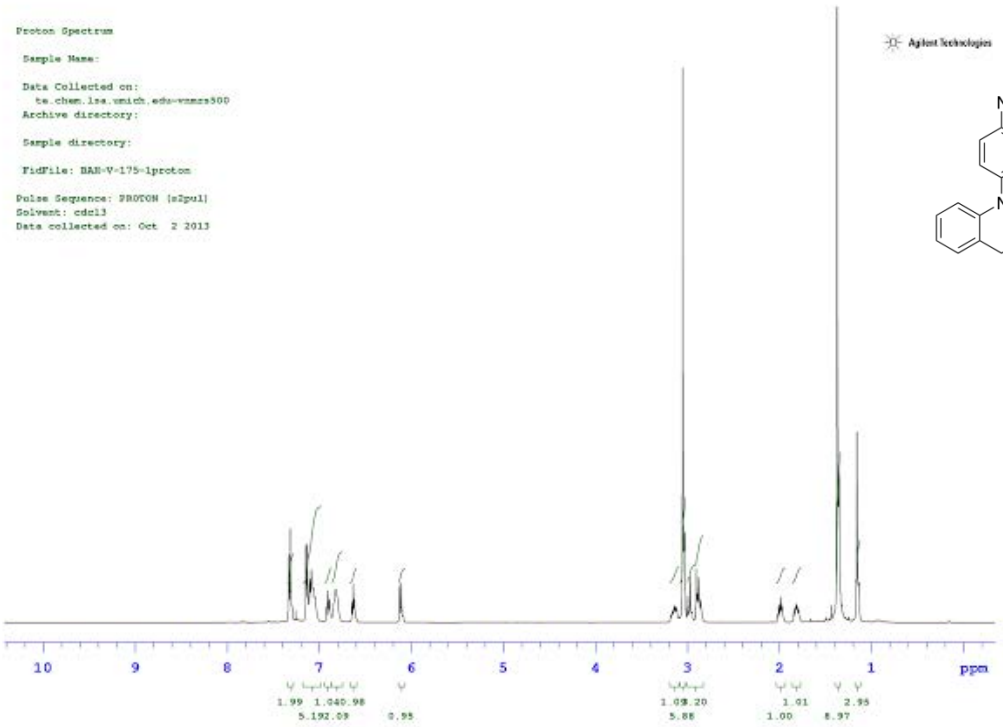


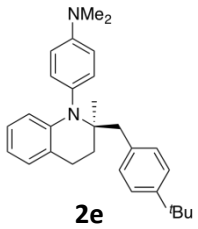
1 PDA Multi 1/254nm 4nm

PeakTable

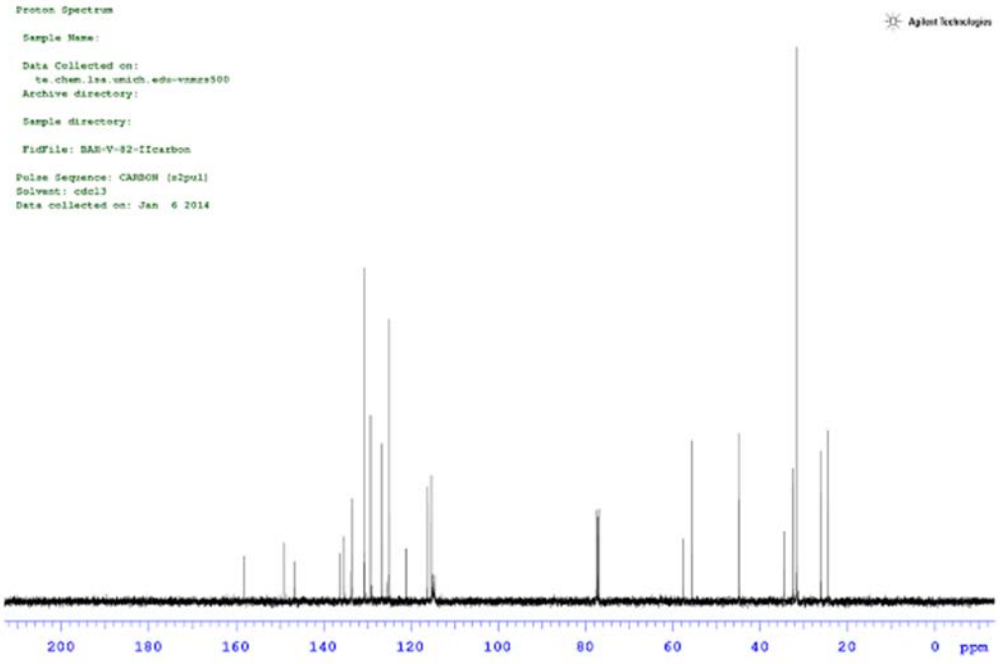
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.070	7785573	492809	92.222	96.307
2	7.314	656637	18898	7.778	3.693
Total		8442211	511707	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-175-1proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2013
Agilent Technologies
10 9 8 7 6 5 4 3 2 1 ppm
1.99 1.040.98 5.192.09 0.95
1.08.20 5.88
1.01 1.00
2.95 8.97


NMe2
N
tBu

2e


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-82-IIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 6 2014
Agilent Technologies
200 180 160 140 120 100 80 60 40 20 0 ppm

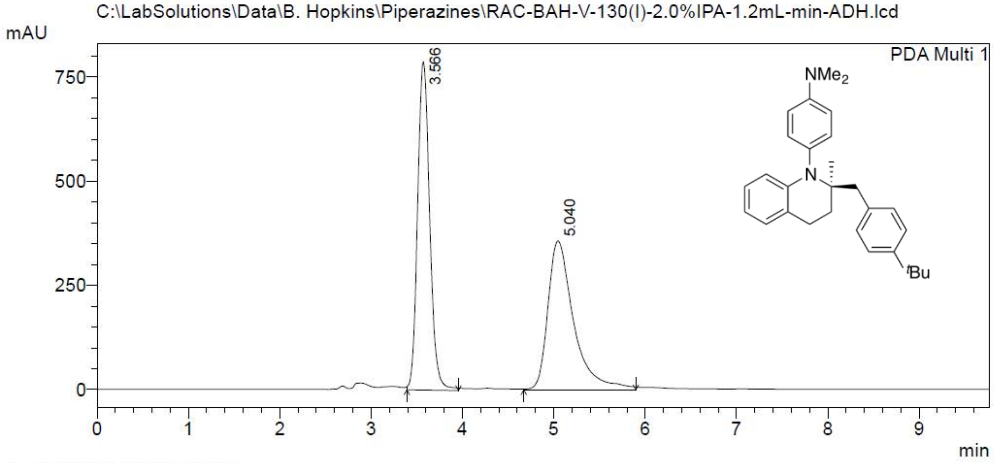
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-V-130(I)-2.0%IPA-1.2mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-V-130(I)-2.0%IPA-1.2mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-V-130(I)-2.0%IPA-1.2mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/7/2014 2:28:35 PM
Data Processed	: 1/7/2014 2:38:23 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-V-130(I)-2.0%IPA-1.2mL-min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	3.566	7275398	788798	50.900	68.798
2	5.040	7018162	357737	49.100	31.202
Total		14293559	1146535	100.000	100.000

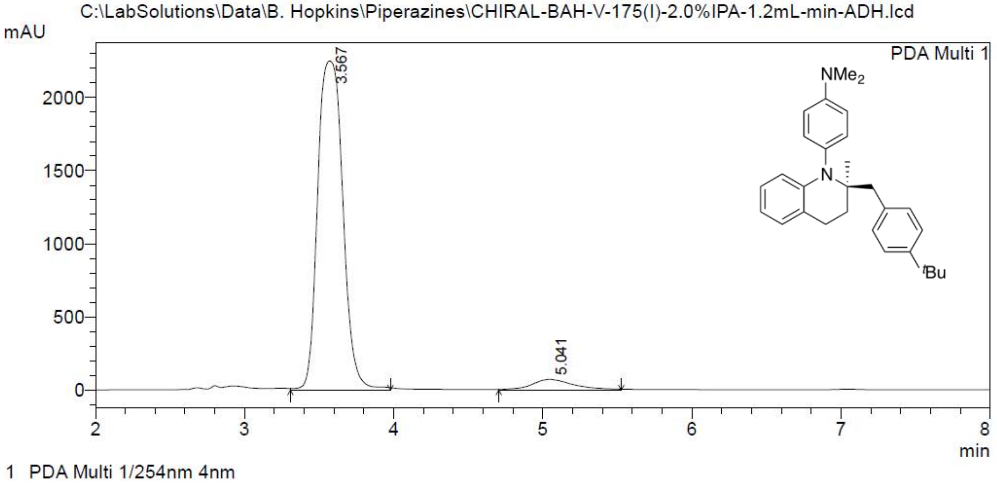
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-V-175(I)-2.0%IPA-1.2mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-V-175(I)-2.0%IPA-1.2mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-V-175(I)-2.0%IPA-1.2mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/7/2014 2:55:49 PM
Data Processed	: 1/7/2014 3:09:31 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-V-175(I)-2.0%IPA-1.2mL-min-ADH.lcd

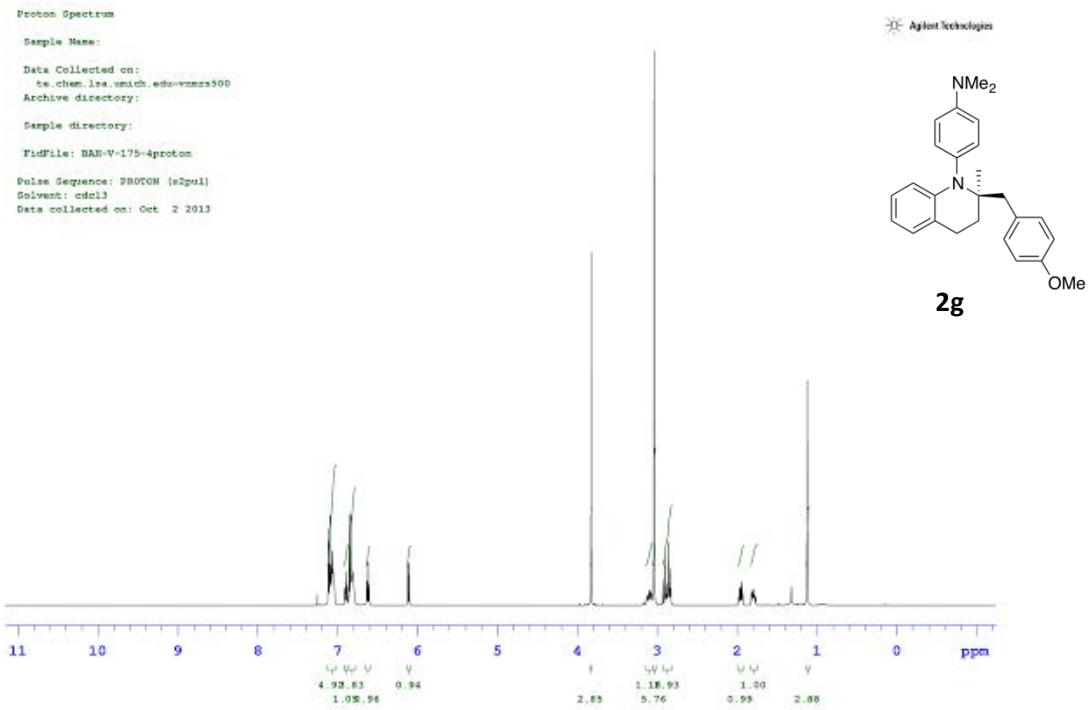


1 PDA Multi 1/254nm 4nm

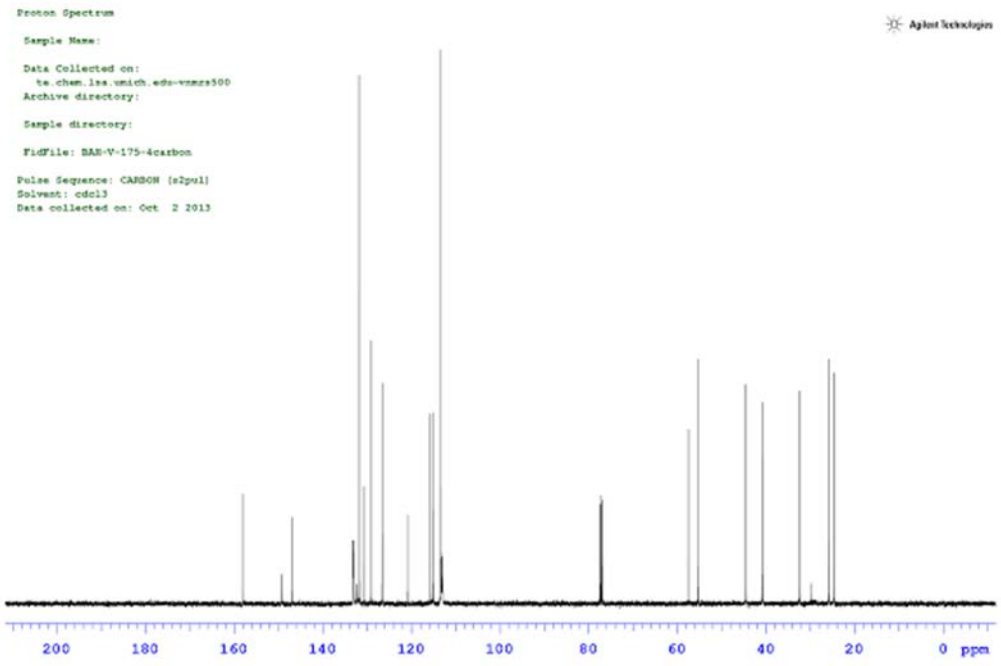
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	3.567	26495662	2250668	94.701	96.851
2	5.041	1482493	73170	5.299	3.149
Total		27978155	2323838	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-175-4proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2013
Agilent Technologies
NMe2
N
OMe
11 10 9 8 7 6 5 4 3 2 1 0 ppm
4.92 3.83 0.94 1.18 3.93 1.00
1.05 5.96 2.85 5.76 0.99 2.88

2g

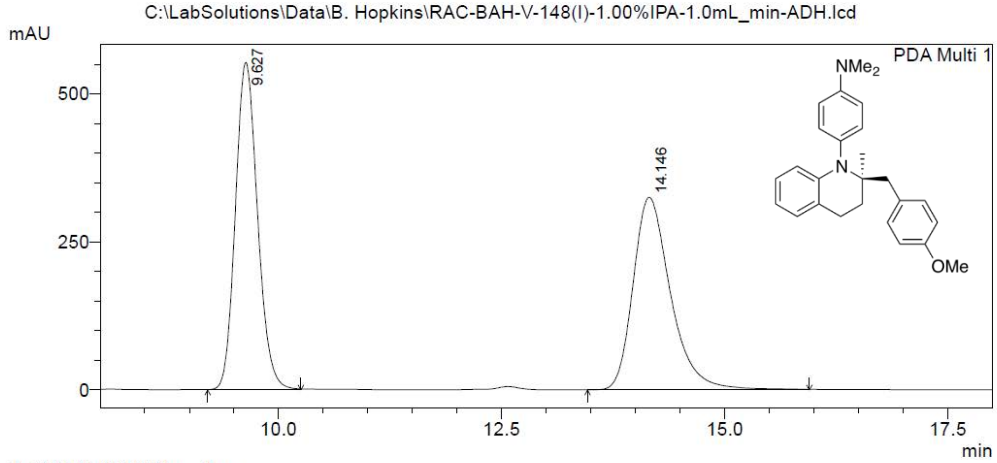

Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-175-4carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2013
Agilent Technologies
200 180 160 140 120 100 80 60 40 20 0 ppm

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

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-V-148(I)-1.00%IPA-1.0mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-V-148(I)-1.00%IPA-1.0mL_min-ADH
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-V-148(I)-1.00%IPA-1.0mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/19/2013 1:55:28 PM
Data Processed	: 6/19/2013 2:26:37 PM

<Chromatogram>



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.627	9572452	553313	50.070	62.964
2	14.146	9545741	325458	49.930	37.036
Total		19118193	878771	100.000	100.000

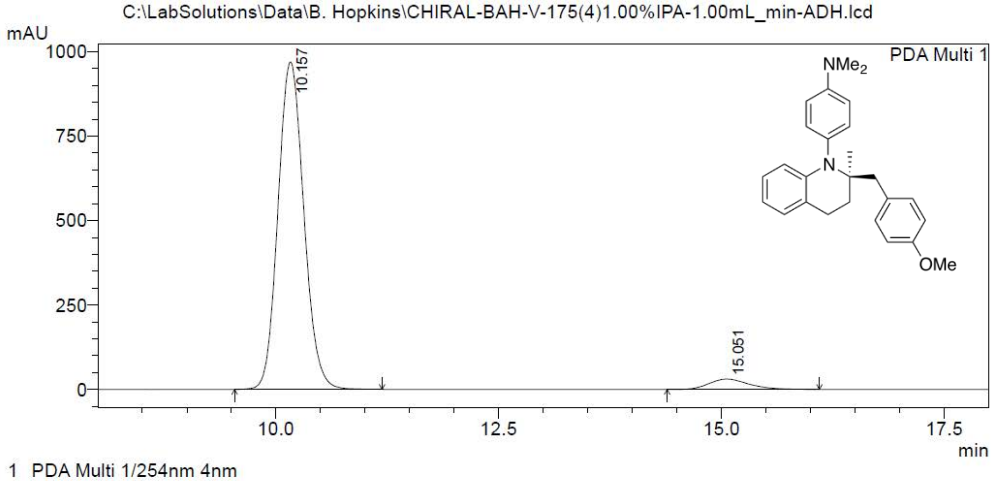
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-V-175(4)1.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-V-175(4)1.00%IPA-1.00mL_min-ADH
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-V-175(4)1.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/9/2013 3:32:48 PM
Data Processed	: 7/9/2013 4:23:53 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-V-175(4)1.00%IPA-1.00mL_min-ADH.lcd

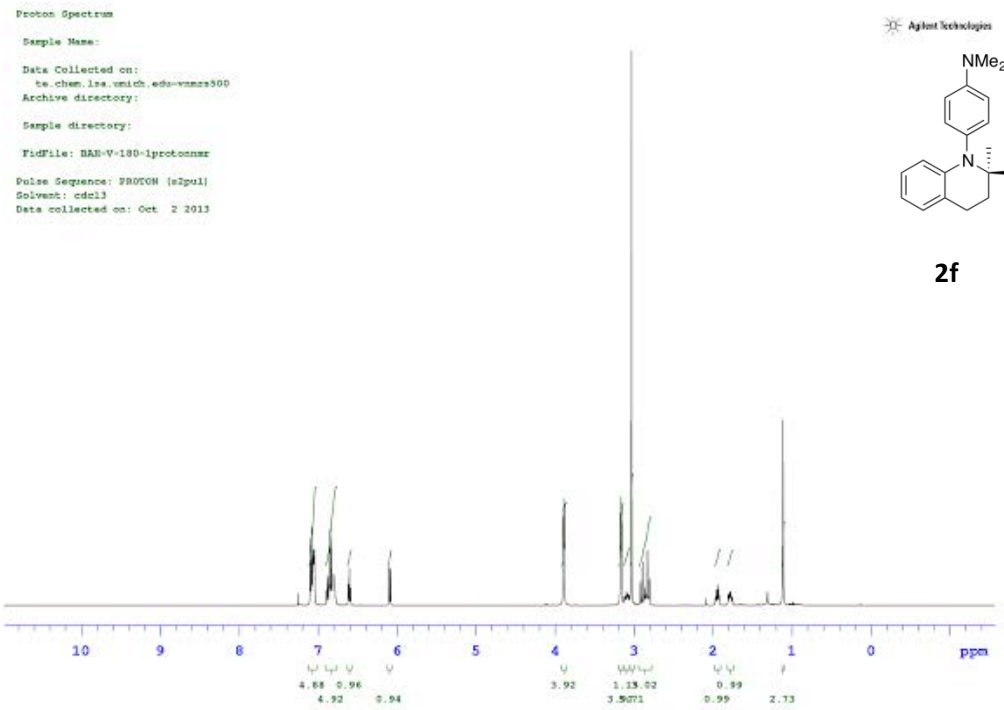


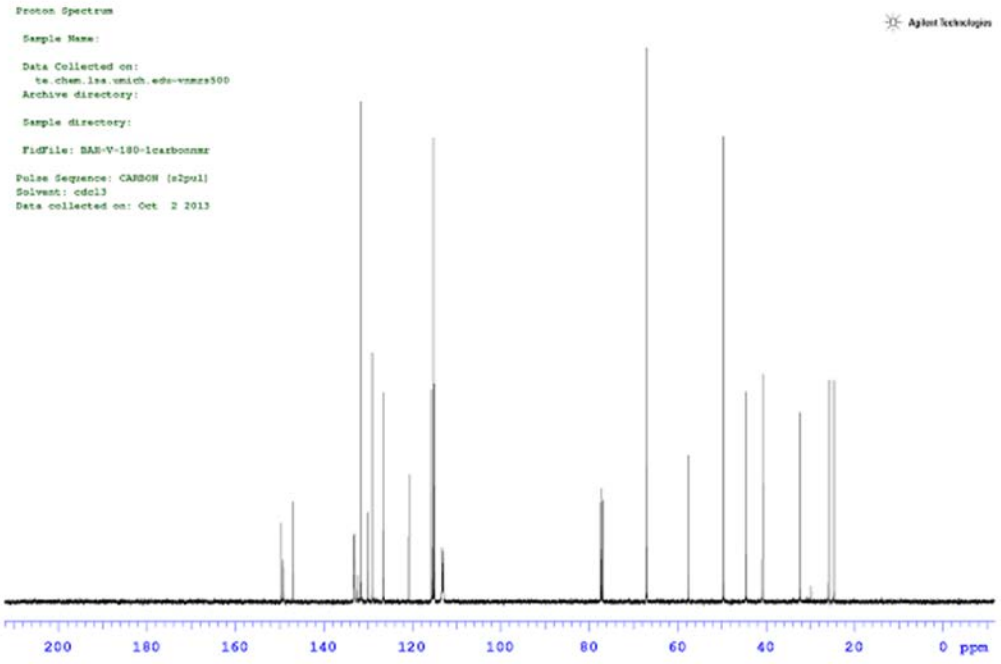
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.157	19581405	969638	95.221	96.912
2	15.051	982727	30893	4.779	3.088
Total		20564132	1000531	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-180-1protonnmr
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2013
Agilent Technologies
NMe2
N
N
O
2f
10
9
8
7
6
5
4
3
2
1
0
ppm
4.68
0.96
3.92
1.13.02
0.89
4.92
0.94
3.9071
0.99
2.73

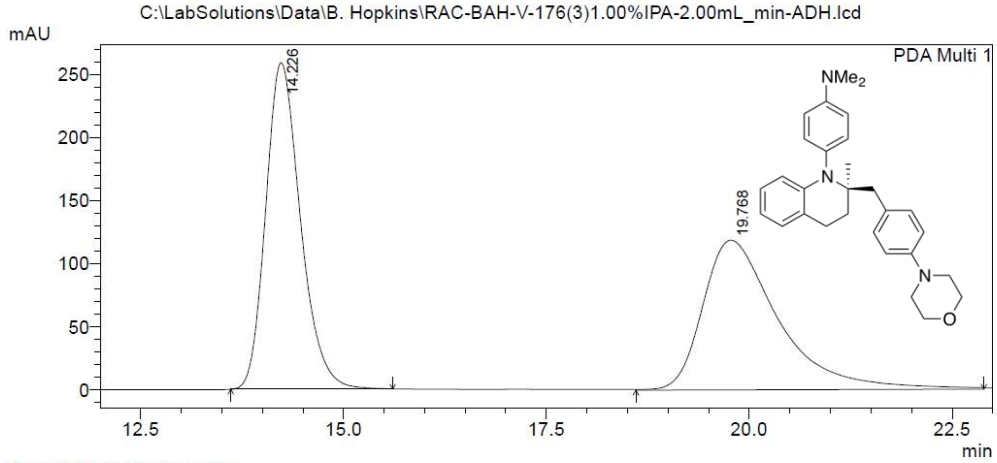

Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-180-1carbonnmr
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2013
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
0 ppm

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

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-V-176(3)1.00%IPA-2.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-V-176(3)1.00%IPA-1.00mL_min-ADH
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-V-176(3)1.00%IPA-2.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/10/2013 1:07:24 PM
Data Processed	: 7/10/2013 1:39:32 PM

<Chromatogram>



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

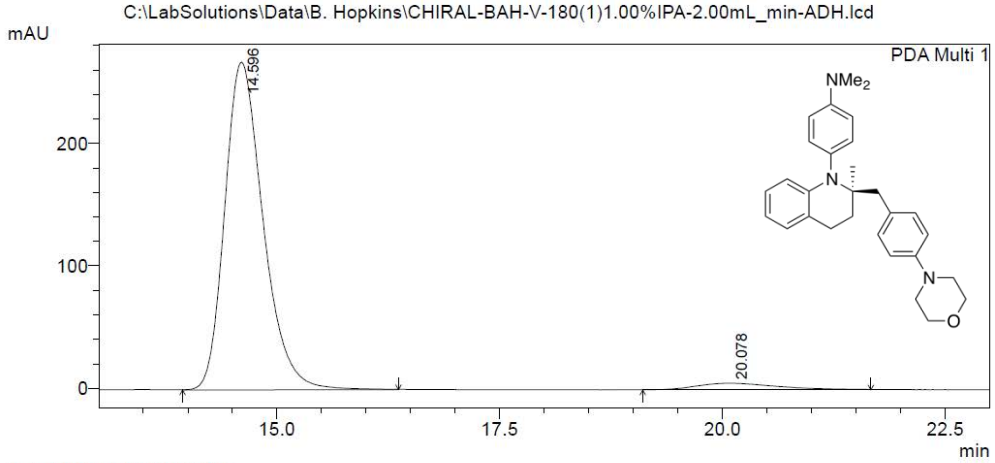
Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.226	7718738	258819	50.029	68.563
2	19.768	7709819	118673	49.971	31.437
Total		15428557	377492	100.000	100.000

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

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-V-180(1)1.00%IPA-2.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-V-180(1)1.00%IPA-2.00mL_min-ADH
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-V-180(1)1.00%IPA-2.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/11/2013 8:16:19 PM
Data Processed	: 7/11/2013 8:41:12 PM

<Chromatogram>

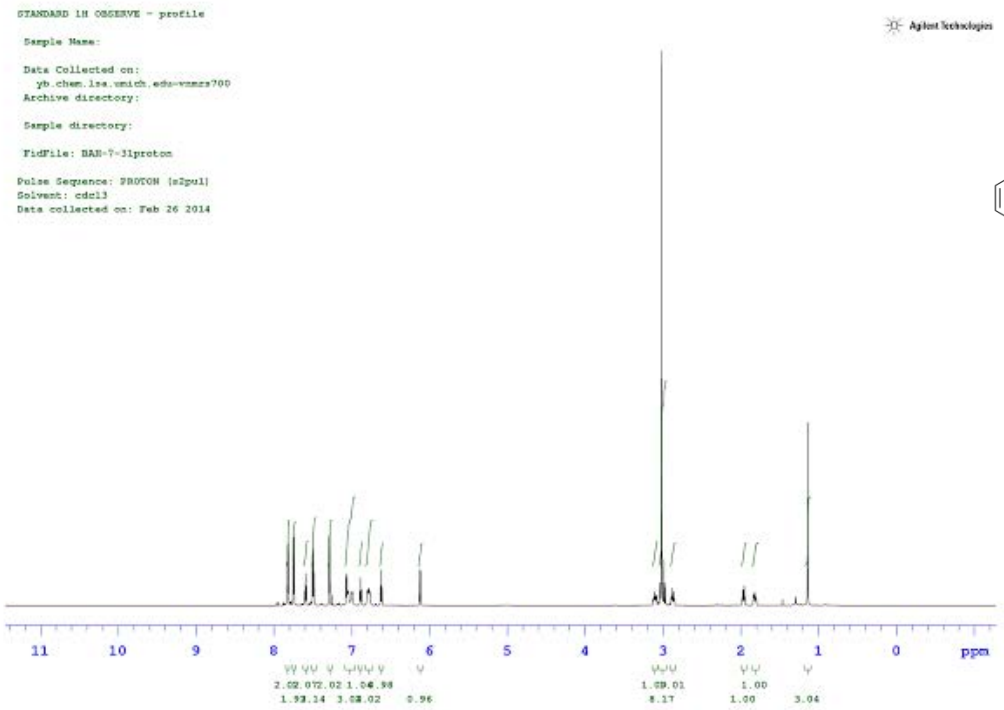


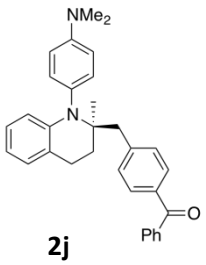
1 PDA Multi 1/254nm 4nm

PeakTable

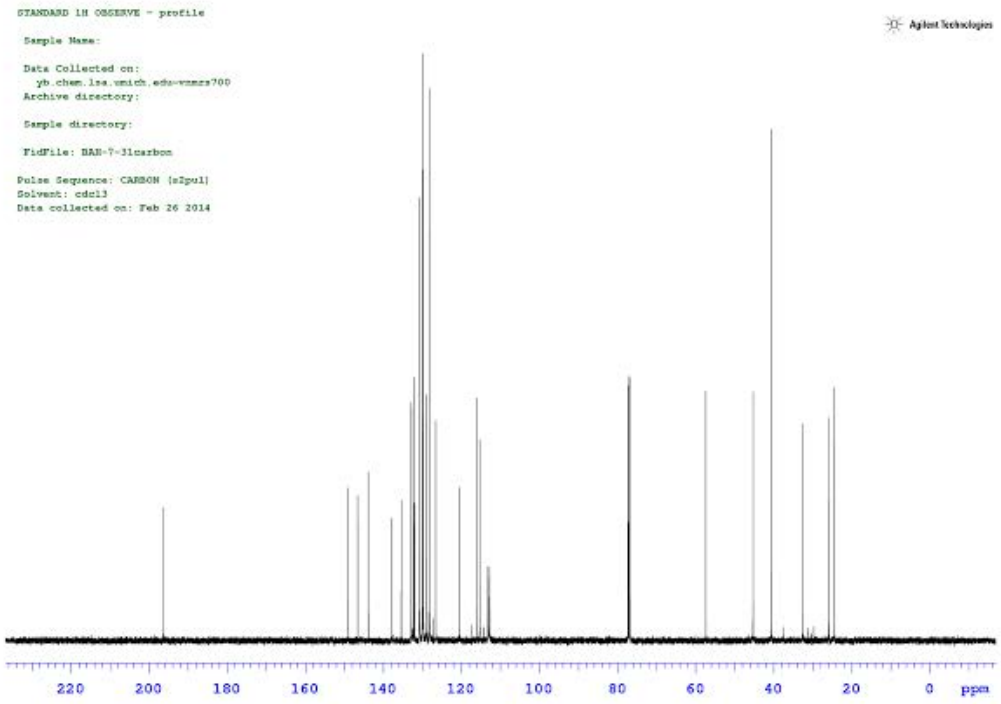
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.596	8029315	267600	95.692	97.980
2	20.078	361478	5518	4.308	2.020
Total		8390793	273118	100.000	100.000


STANDARD 1H OBSERVE - profile
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-7-31proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 26 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.02 2.07 2.02 1.04 0.98
1.93 3.14 3.02 3.02 0.96
1.03 8.01
8.17
1.00
1.00
3.04


NMe2
N
O
Ph

2j


STANDARD 1H OBSERVE - profile
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-7-31carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 26 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm

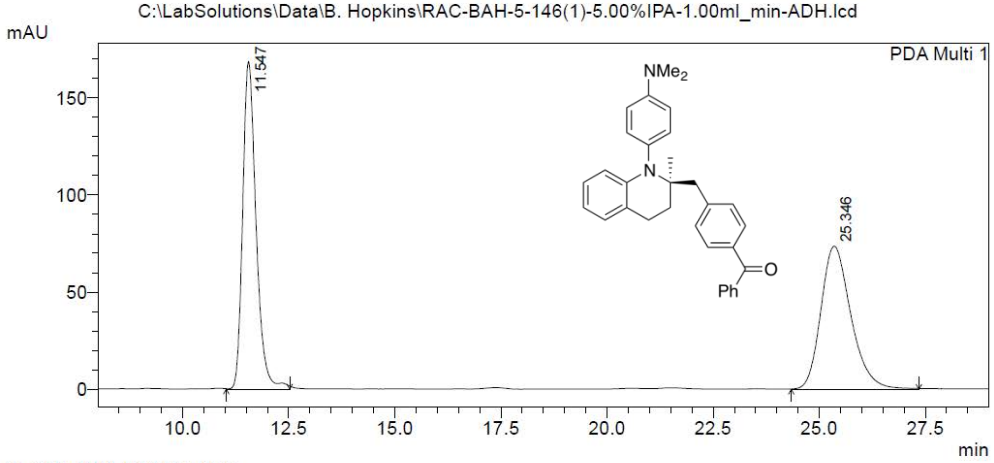
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-5-146(1)-5.00%IPA-1.00ml_min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-5-146(1)-5.00%IPA-1.00ml_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-5-146(1)-5.00%IPA-1.00ml_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/5/2014 4:32:22 PM
Data Processed	: 3/5/2014 5:42:23 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-5-146(1)-5.00%IPA-1.00ml_min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.547	3700277	168662	50.439	69.604
2	25.346	3635825	73655	49.561	30.396
Total		7336102	242317	100.000	100.000

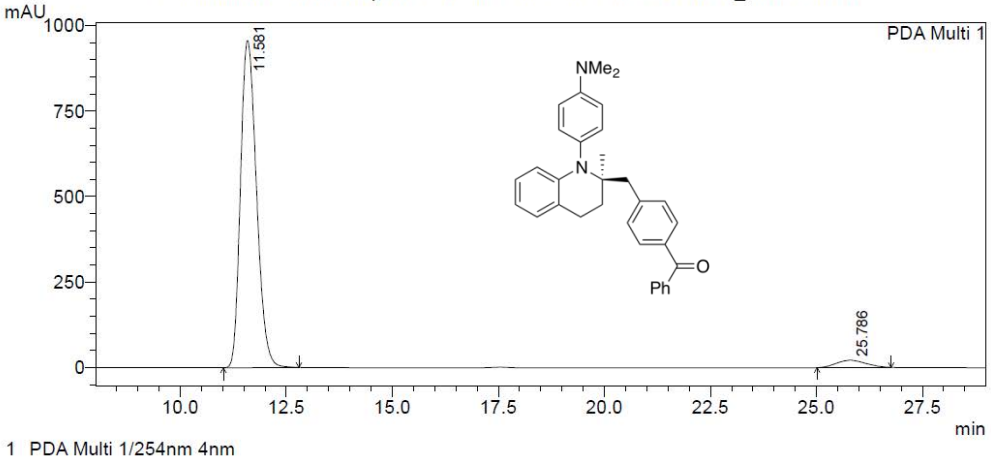
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-7-31-5.00%IPA-1.00ml_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-7-31-5.00%IPA-1.00ml_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-7-31-5.00%IPA-1.00ml_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/6/2014 8:17:12 AM
Data Processed	: 3/6/2014 9:06:37 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-7-31-5.00%IPA-1.00ml_min-ADH.lcd

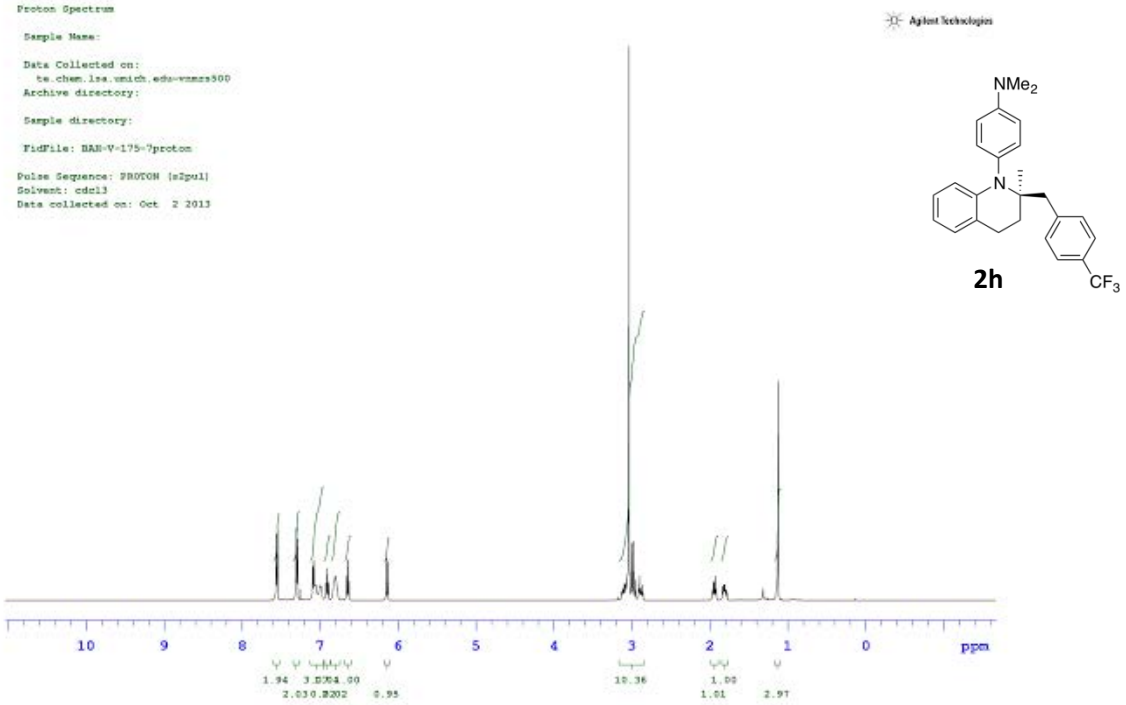


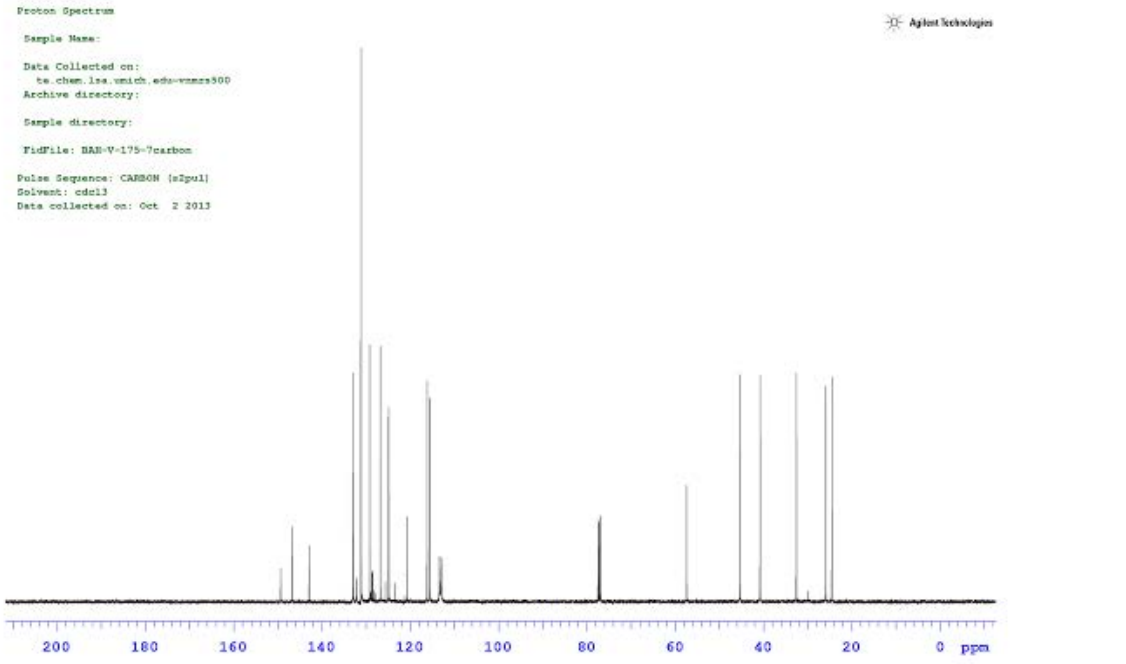
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.581	25116263	957060	95.976	97.815
2	25.786	1053053	21384	4.024	2.185
Total		26169315	978444	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-175-7proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2013
Agilent Technologies
NMe2
N
CF3
2h
10
9
8
7
6
5
4
3
2
1
0
ppm
1.94
3.03
4.00
0.95
10.36
1.00
2.03
0.82
1.01
2.97


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-175-7carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2013
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
0
ppm

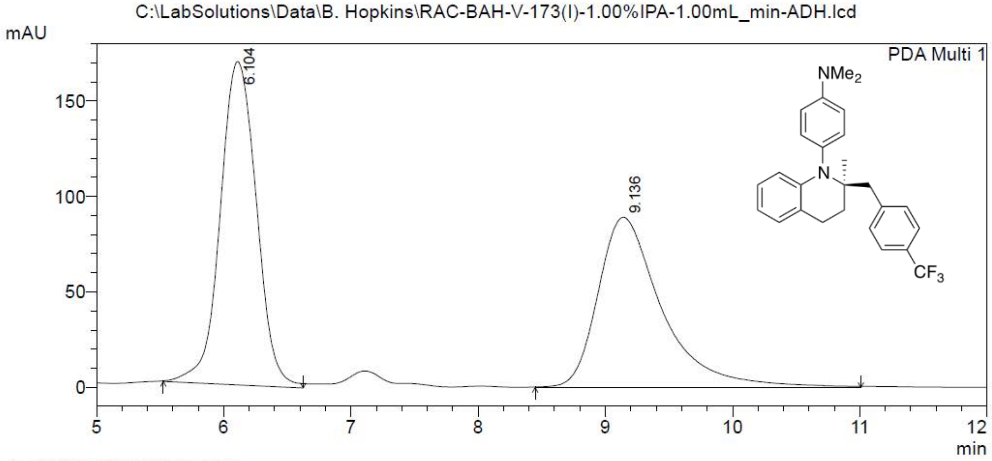
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-V-173(I)-1.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-V-173(I)-1.00%IPA-1.00mL_min-ADH
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-V-173(I)-1.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/5/2013 10:43:49 AM
Data Processed	: 7/5/2013 10:59:32 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-V-173(I)-1.00%IPA-1.00mL_min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.104	3355357	169293	51.728	65.524
2	9.136	3131162	89076	48.272	34.476
Total		6486519	258369	100.000	100.000

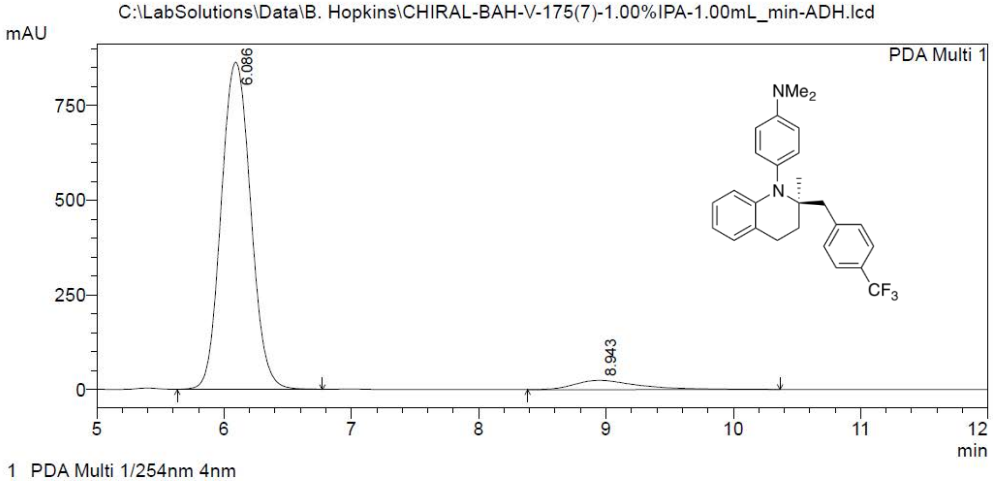
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-V-175(7)-1.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-V-175(7)-1.00%IPA-1.00mL_min-ADH
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-V-175(7)-1.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/8/2013 4:21:15 PM
Data Processed	: 7/8/2013 5:16:21 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-V-175(7)-1.00%IPA-1.00mL_min-ADH.lcd

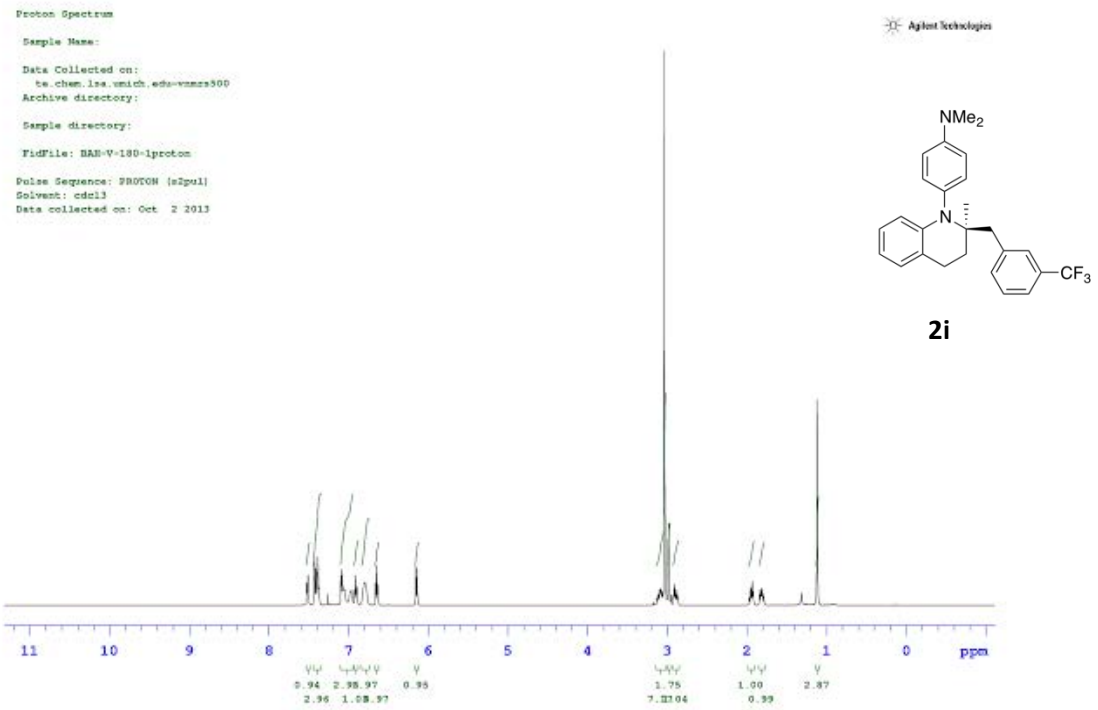


1 PDA Multi 1/254nm 4nm

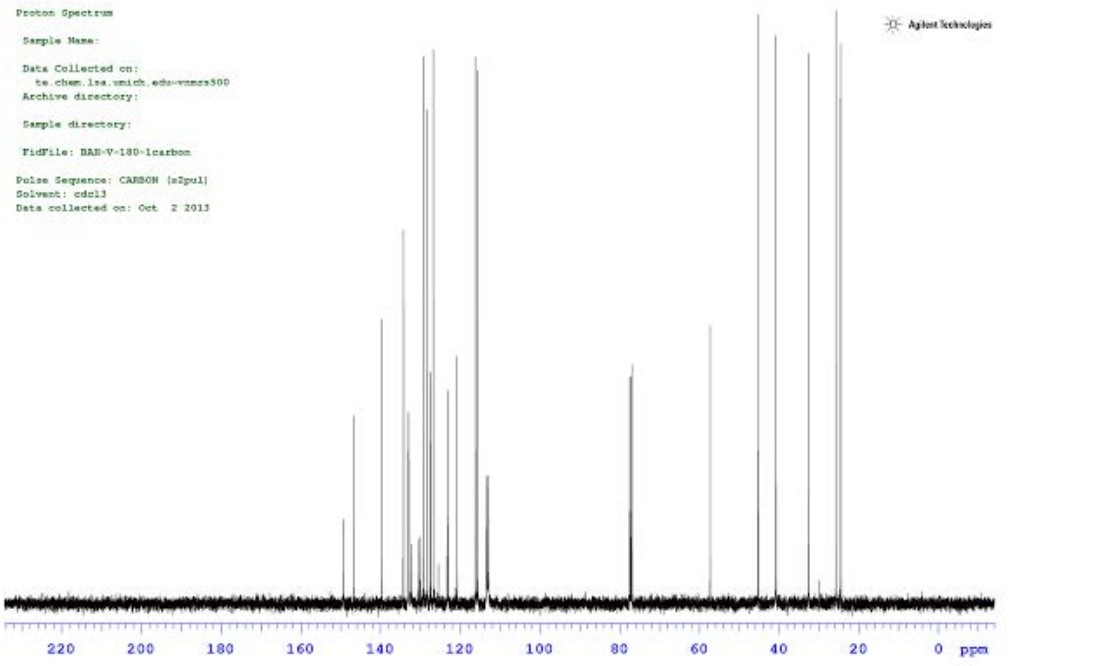
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.086	14005441	865079	94.075	97.253
2	8.943	882164	24434	5.925	2.747
Total		14887605	889513	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-180-1proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2013
Agilent Technologies
NMe2
N
CF3
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.94 2.98.97 0.95
2.96 1.08.97
1.75
7.2104
1.00
0.99
2.87

2i


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-180-1carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 2 2013
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm

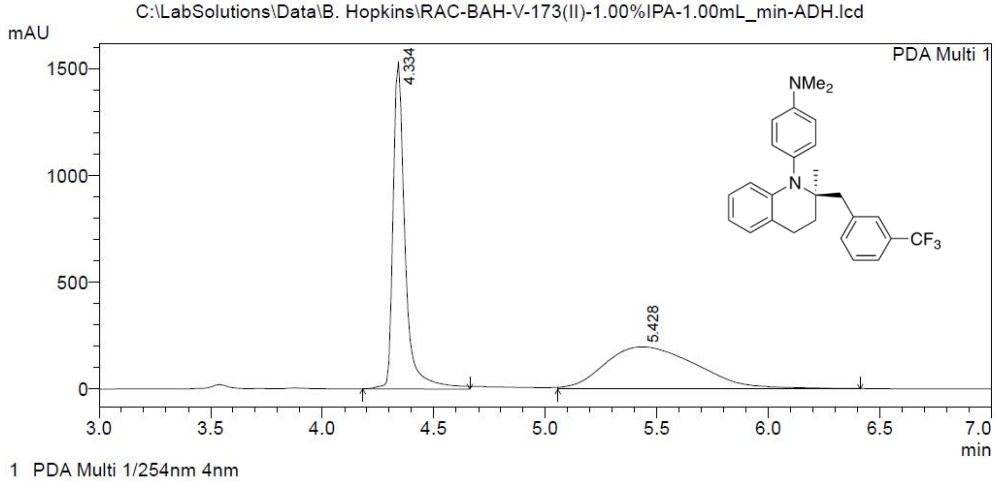
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-V-173(II)-1.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-V-173(II)-1.00%IPA-1.00mL_min-ADH
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-V-173(II)-1.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/5/2013 11:18:13 AM
Data Processed	: 7/5/2013 11:37:51 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-V-173(II)-1.00%IPA-1.00mL_min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

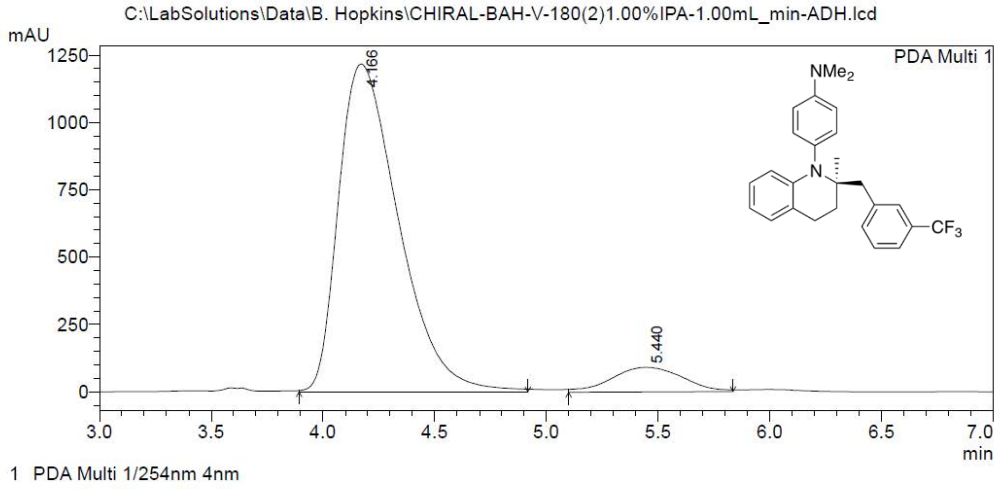
Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.334	5771676	1533578	49.949	88.575
2	5.428	5783466	197806	50.051	11.425
Total		11555142	1731384	100.000	100.000

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

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-V-180(2)1.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-V-180(2)1.00%IPA-1.00mL_min-ADH
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-V-180(2)1.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/11/2013 8:00:07 PM
Data Processed	: 7/11/2013 8:12:14 PM

<Chromatogram>

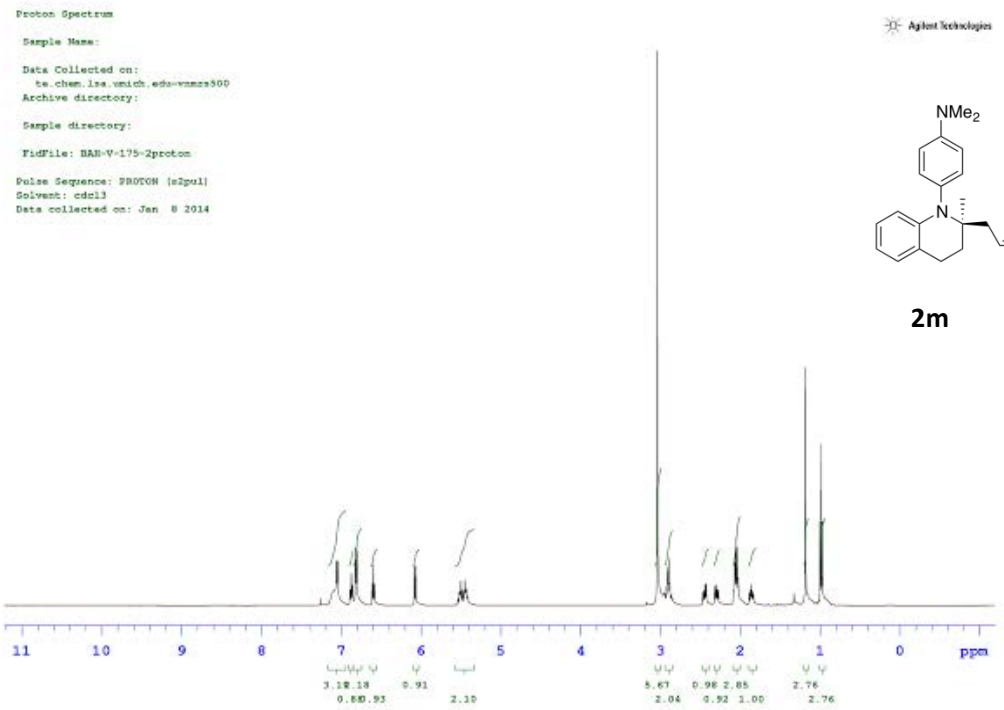


1 PDA Multi 1/254nm 4nm

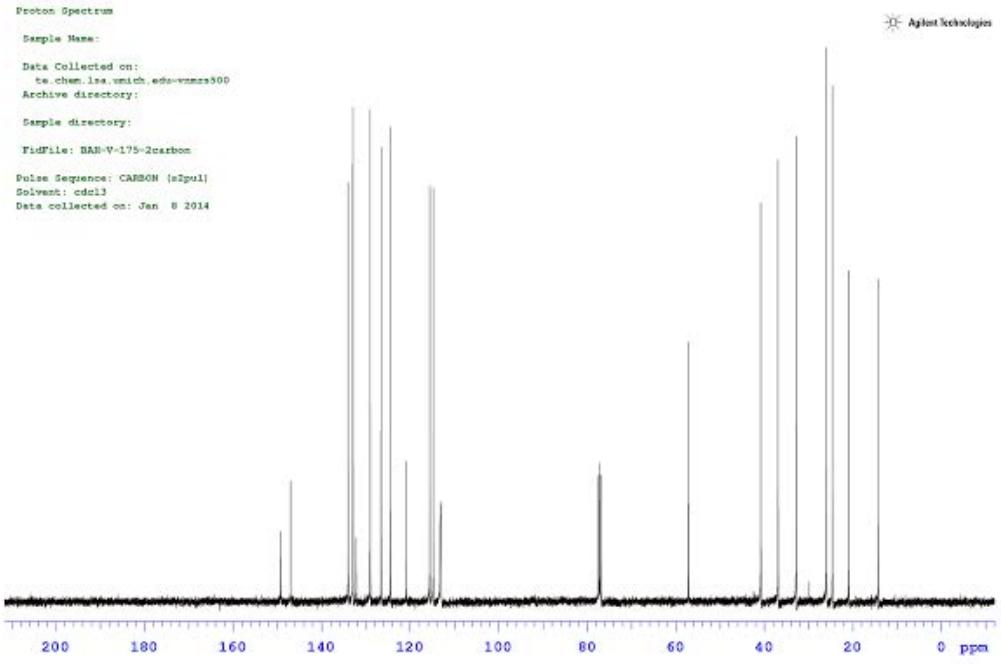
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.166	22771594	1218094	91.847	93.044
2	5.440	2021482	91061	8.153	6.956
Total		24793076	1309155	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-175-2proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 8 2014
Agilent Technologies
NMe2
N
C2H5
11
10
9
8
7
6
5
4
3
2
1
0
ppm
3.18 3.18
0.88 0.93
0.91
2.10
5.67
2.04
0.98 2.85
0.92 1.00
2.76
2.76

2m


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-175-2carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 8 2014
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
0 ppm

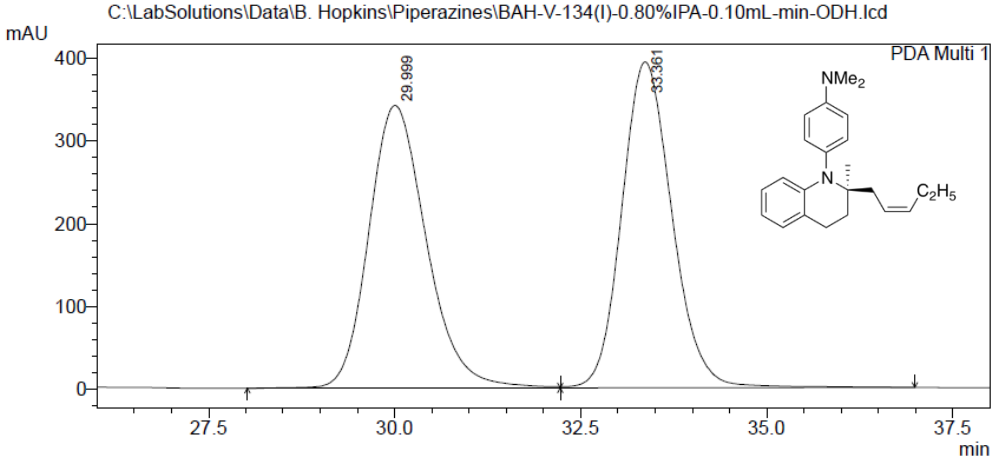
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\BAH-V-134(I)-0.80%IPA-0.10mL-min-ODH.lcd

Acquired by	: Admin
Sample Name	: BAH-V-134(I)-0.80%IPA-0.10mL-min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: BAH-V-134(I)-0.80%IPA-0.10mL-min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/10/2014 11:27:40 AM
Data Processed	: 1/10/2014 12:37:41 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\BAH-V-134(I)-0.80%IPA-0.10mL-min-ODH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.999	18347679	341144	49.807	46.445
2	33.361	18489721	393361	50.193	53.555
Total		36837400	734505	100.000	100.000

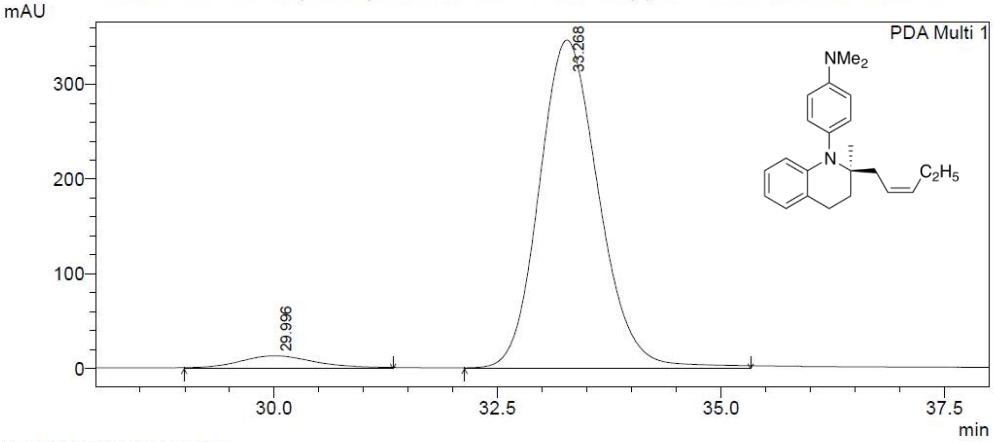
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-V-175(2)-0.80%IPA-0.10mL-min-ODH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-V-175(2)-0.80%IPA-0.10mL-min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-V-175(2)-0.80%IPA-0.10mL-min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/10/2014 1:00:16 PM
Data Processed	: 1/10/2014 1:45:08 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-V-175(2)-0.80%IPA-0.10mL-min-ODH.lcd

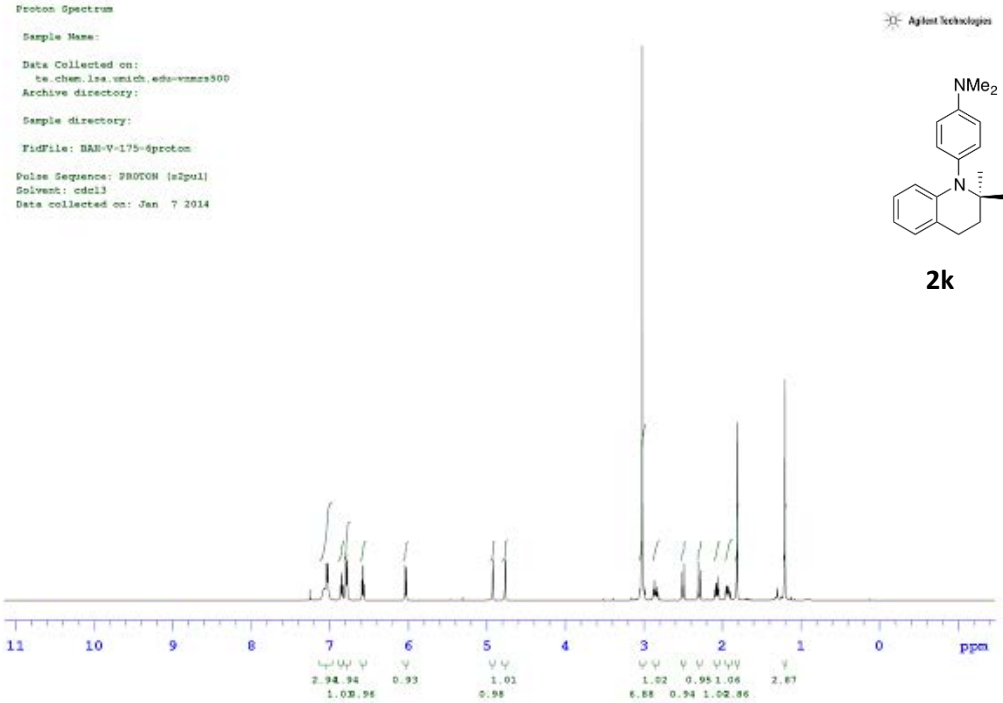


1 PDA Multi 1/254nm 4nm

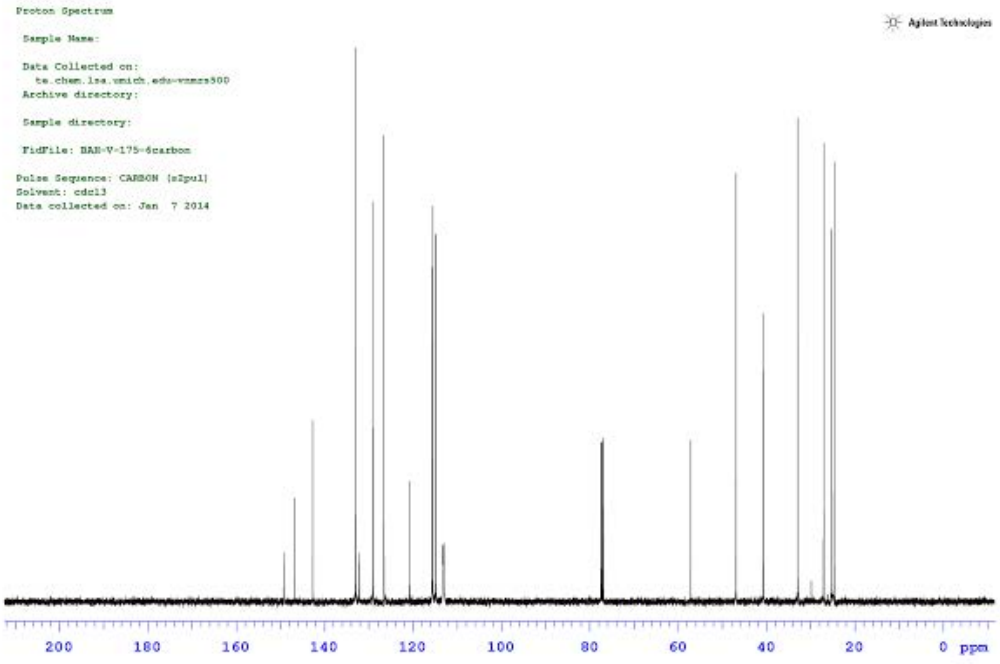
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.996	874522	13856	5.125	3.840
2	33.268	16190761	346963	94.875	96.160
Total		17065283	360819	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-175-6proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 7 2014
Agilent Technologies
NMe2
N
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.94 .94 0.93 1.01 1.02 0.95 1.06 2.87
1.03 .96 0.98 6.88 0.94 1.02 .86

2k


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-175-6carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 7 2014
Agilent Technologies
200 180 160 140 120 100 80 60 40 20 0 ppm

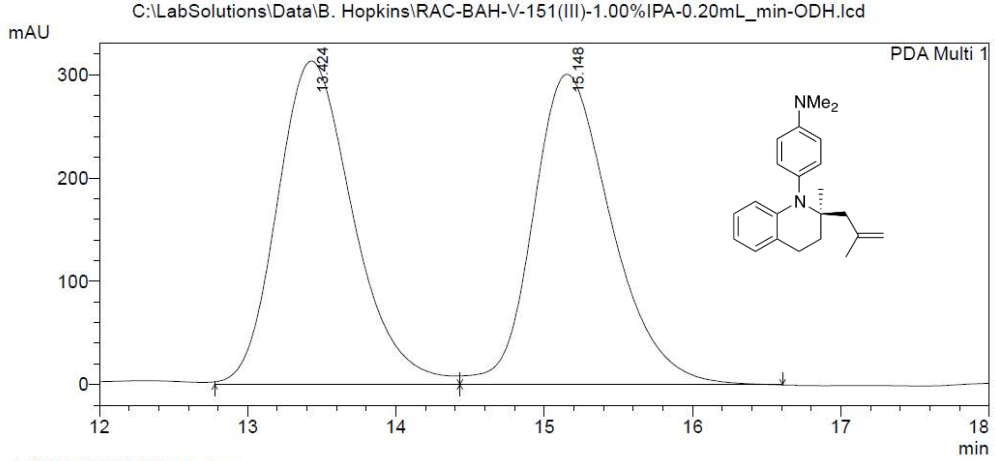
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-V-151(III)-1.00%IPA-0.20mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-V-151(III)-1.00%IPA-0.20mL_min-ODH
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-V-151(III)-1.00%IPA-0.20mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 6/25/2013 7:51:01 AM
Data Processed	: 6/25/2013 8:22:55 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-V-151(III)-1.00%IPA-0.20mL_min-ODH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.424	11170253	313417	50.319	51.046
2	15.148	11028795	300572	49.681	48.954
Total		22199048	613990	100.000	100.000

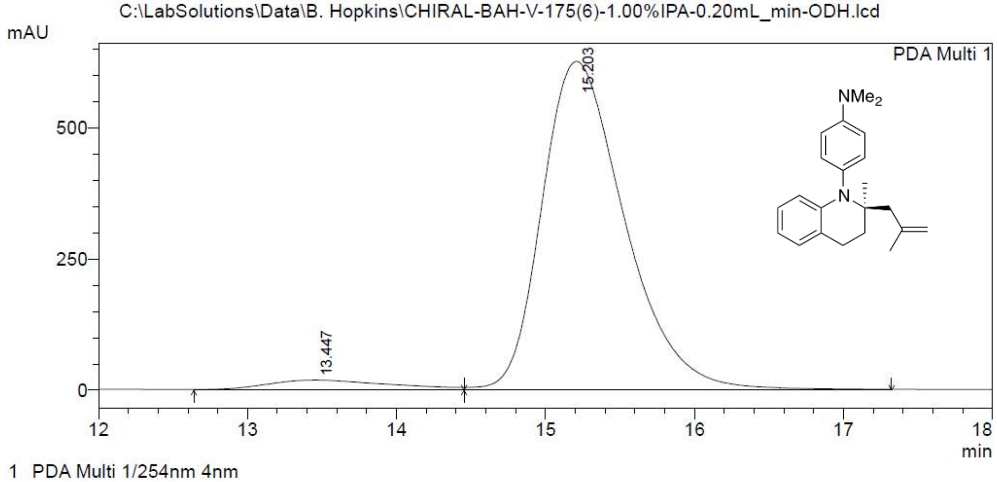
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-V-175(6)-1.00%IPA-0.20mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-V-175(6)-1.00%IPA-0.20mL_min-ODH
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-V-175(6)-1.00%IPA-0.20mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/9/2013 10:08:46 AM
Data Processed	: 7/9/2013 10:46:41 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-V-175(6)-1.00%IPA-0.20mL_min-ODH.lcd

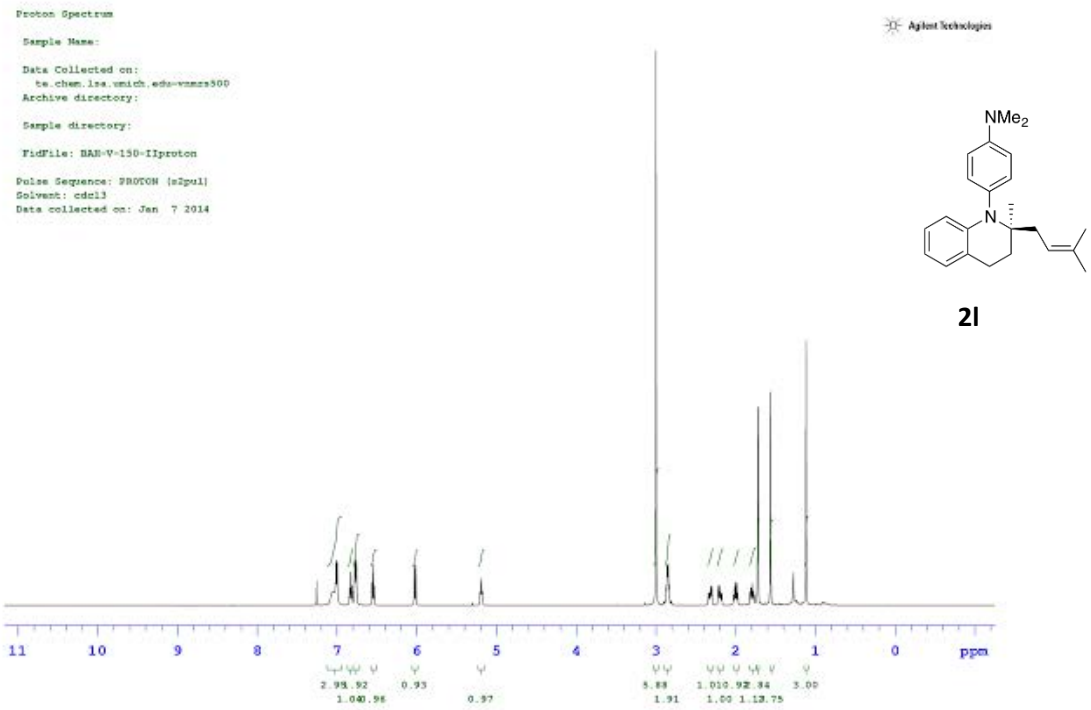


1 PDA Multi 1/254nm 4nm

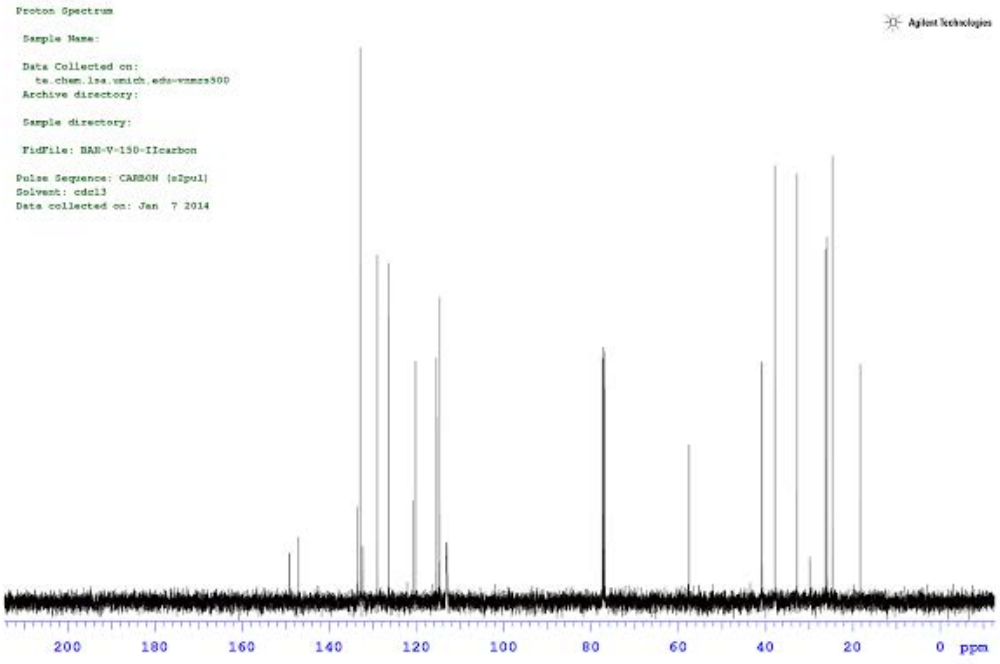
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.447	1140507	19143	4.480	2.964
2	15.203	24314678	626779	95.520	97.036
Total		25455185	645922	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-150-IIproton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 7 2014
Agilent Technologies
NMe2
N
11
10
9
8
7
6
5
4
3
2
1
0
ppm

2l


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-V-150-IIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 7 2014
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
0
ppm

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

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-V-151(II)-0.90%IPA-0.10mL-min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-V-151(II)-0.90%IPA-0.10mL-min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-V-151(II)-0.90%IPA-0.10mL-min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/8/2014 2:39:11 PM
Data Processed	: 1/8/2014 3:16:32 PM

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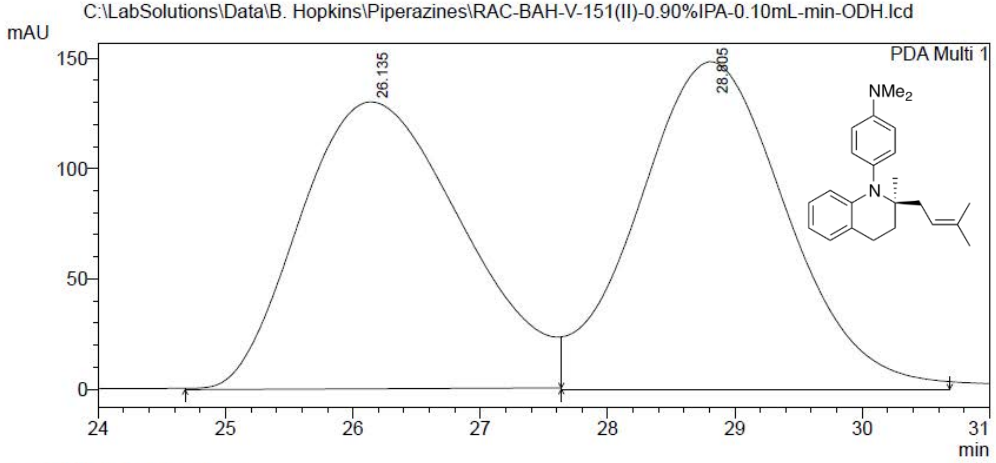
C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-V-151(II)-0.90%IPA-0.10mL-min-ODH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.135	11580726	130031	48.944	46.701
2	28.805	12080286	148401	51.056	53.299
Total		23661011	278432	100.000	100.000

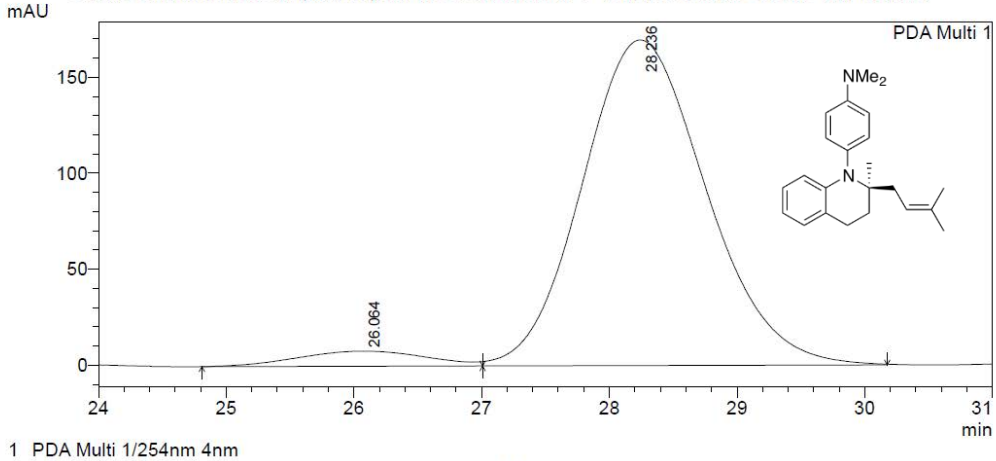
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-V-150(II)-0.90%IPA-0.10mL-min-ODH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-V-150(II)-0.90%IPA-0.10mL-min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-V-150(II)-0.90%IPA-0.10mL-min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/8/2014 1:40:15 PM
Data Processed	: 1/8/2014 2:31:20 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-V-150(II)-0.90%IPA-0.10mL-min-ODH.lcd

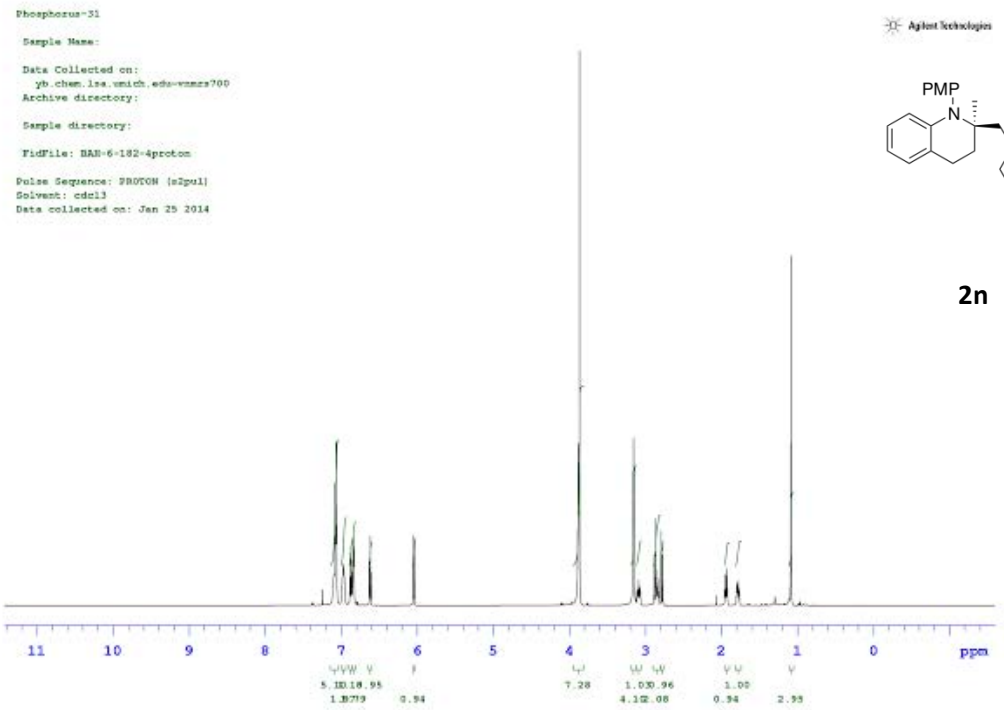


1 PDA Multi 1/254nm 4nm

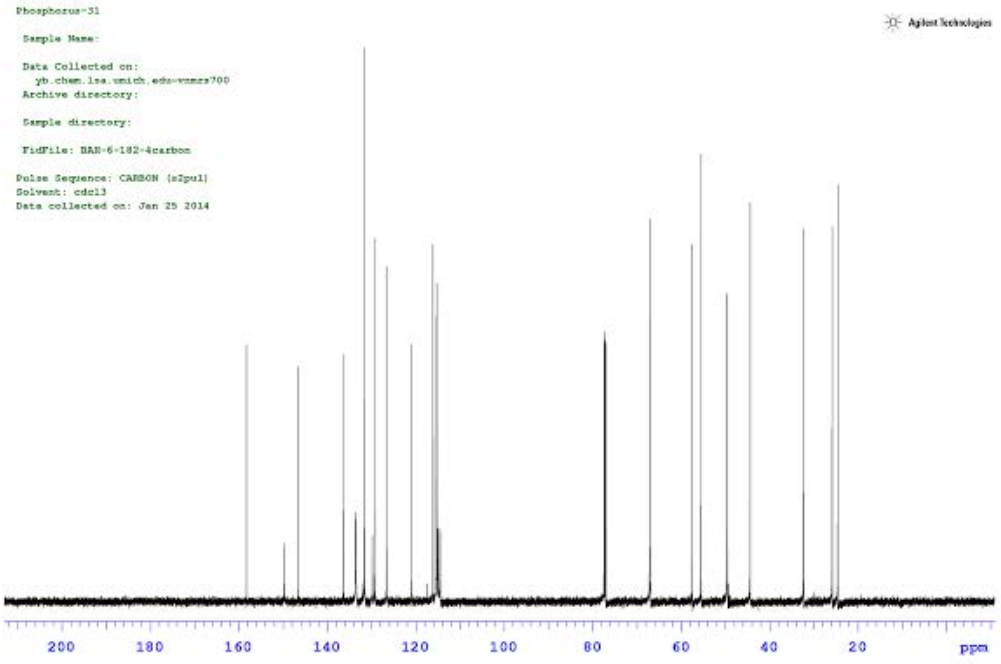
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.064	557639	7913	4.659	4.460
2	28.236	11411454	169485	95.341	95.540
Total		11969093	177398	100.000	100.000


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-182-4proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 25 2014
Agilent Technologies
PMP
N
N
O
11
10
9
8
7
6
5
4
3
2
1
0
ppm
7.28
1.00
0.94
2.95

2n


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-182-4carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 25 2014
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
ppm

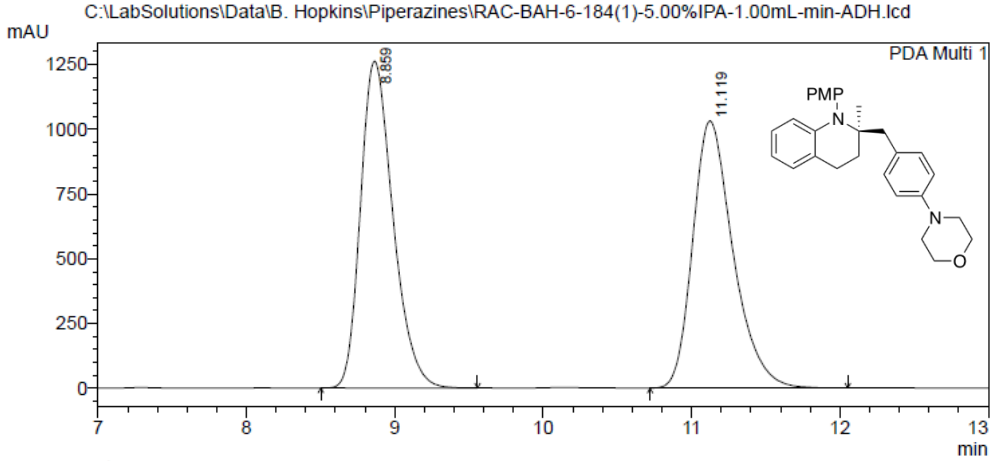
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-6-184(1)-5.00%IPA-1.00mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-184(1)-5.00%IPA-1.00mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-184(1)-5.00%IPA-1.00mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/15/2014 2:01:33 PM
Data Processed	: 1/15/2014 2:39:14 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-6-184(1)-5.00%IPA-1.00mL-min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.859	19256652	1262698	49.675	55.027
2	11.119	19508574	1031994	50.325	44.973
Total		38765226	2294692	100.000	100.000

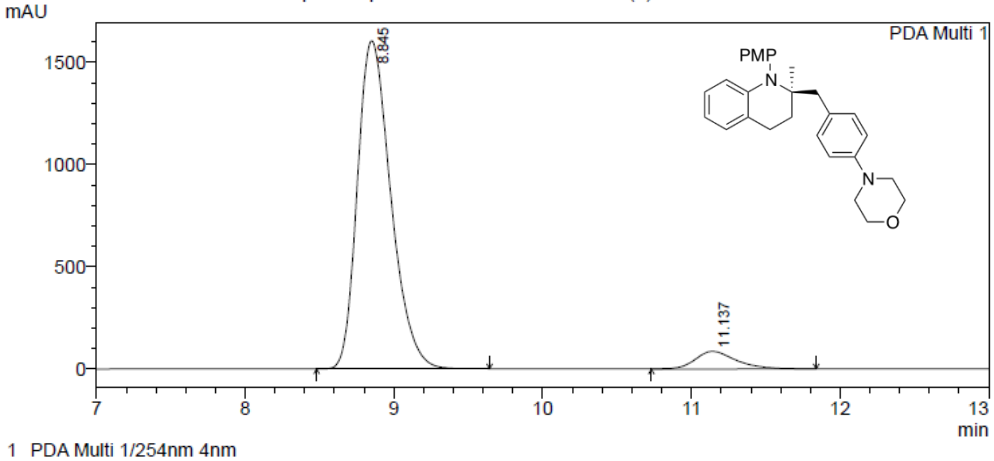
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-6-186(1)-5.00%IPA-1.00mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-6-186(1)-5.00%IPA-1.00mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-6-186(1)-5.00%IPA-1.00mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/15/2014 3:37:40 PM
Data Processed	: 1/15/2014 3:53:10 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-6-186(1)-5.00%IPA-1.00mL-min-ADH.lcd

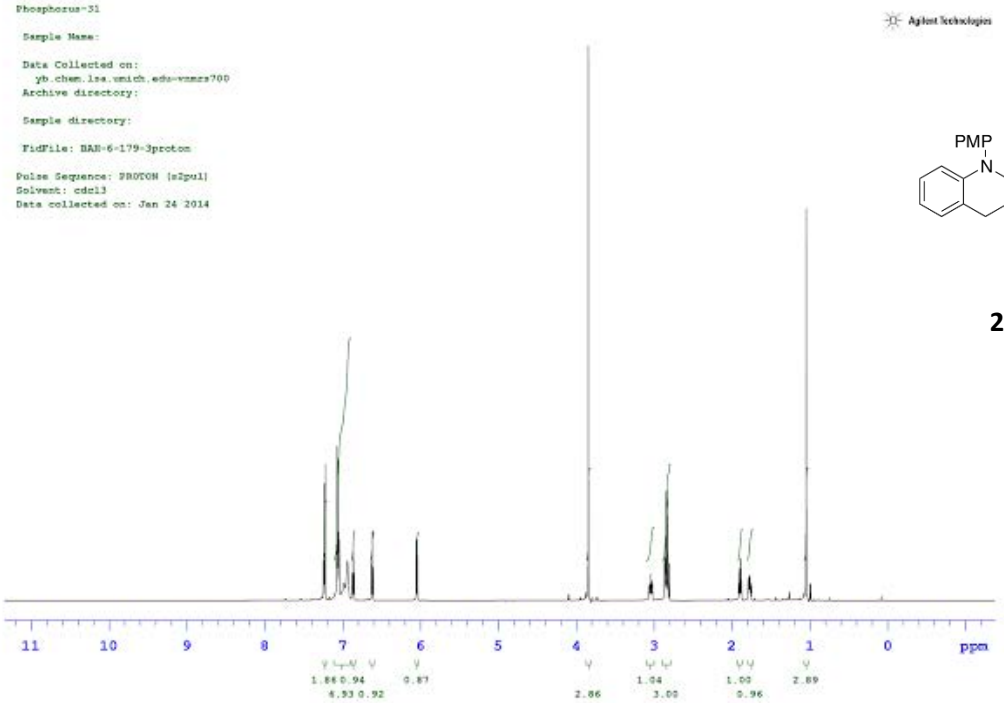


1 PDA Multi 1/254nm 4nm

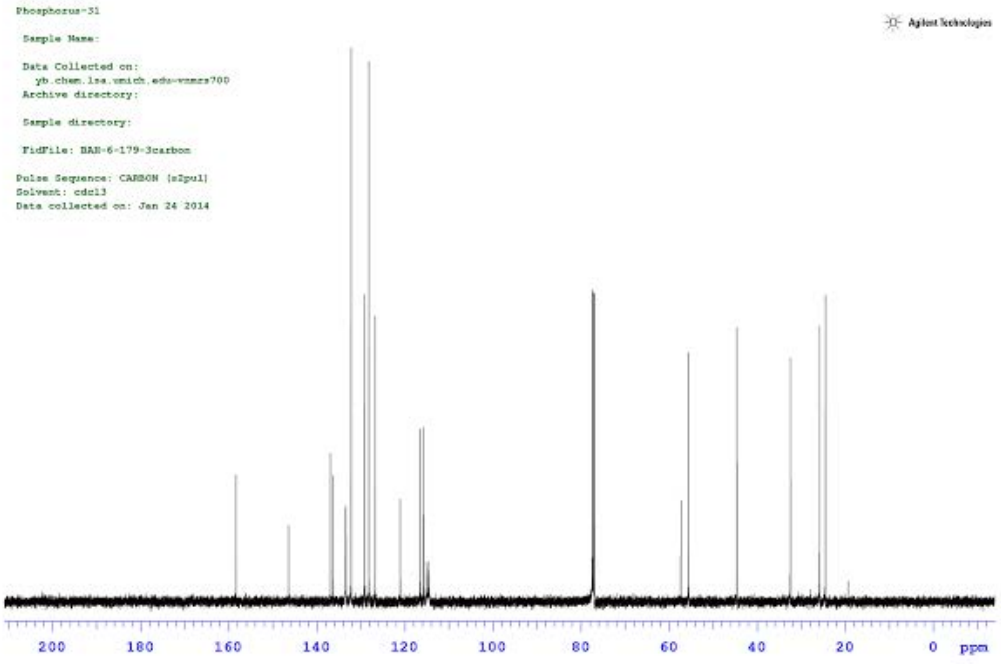
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.845	25086010	1603867	93.832	94.911
2	11.137	1649047	86000	6.168	5.089
Total		26735056	1689867	100.000	100.000


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-6-179-3proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 24 2014
Agilent Technologies
PMP
N
Cl
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.86
0.94
0.87
6.93
0.92
2.86
1.04
3.00
1.00
0.96
2.89

2o

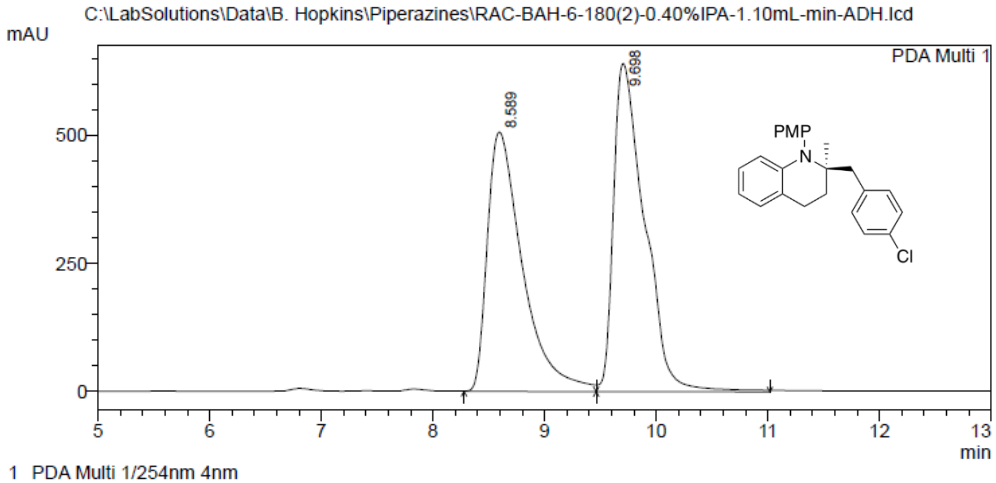

Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-6-179-3carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 24 2014
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
0
ppm

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

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-6-180(2)-0.40%IPA-1.10mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-180(2)-0.40%IPA-1.10mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-180(2)-0.40%IPA-1.10mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/14/2014 5:23:28 PM
Data Processed	: 1/14/2014 5:44:18 PM

<Chromatogram>



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.589	11114931	506846	47.249	44.150
2	9.698	12409202	641165	52.751	55.850
Total		23524132	1148012	100.000	100.000

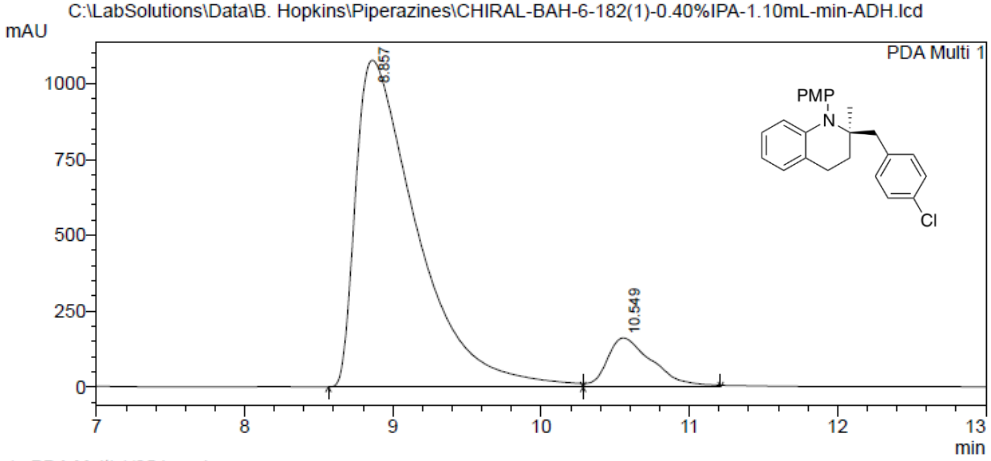
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-6-182(1)-0.40%IPA-1.10mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-6-182(1)-0.40%IPA-1.10mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-6-182(1)-0.40%IPA-1.10mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/14/2014 6:07:27 PM
Data Processed	: 1/14/2014 6:22:02 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-6-182(1)-0.40%IPA-1.10mL-min-ADH.lcd

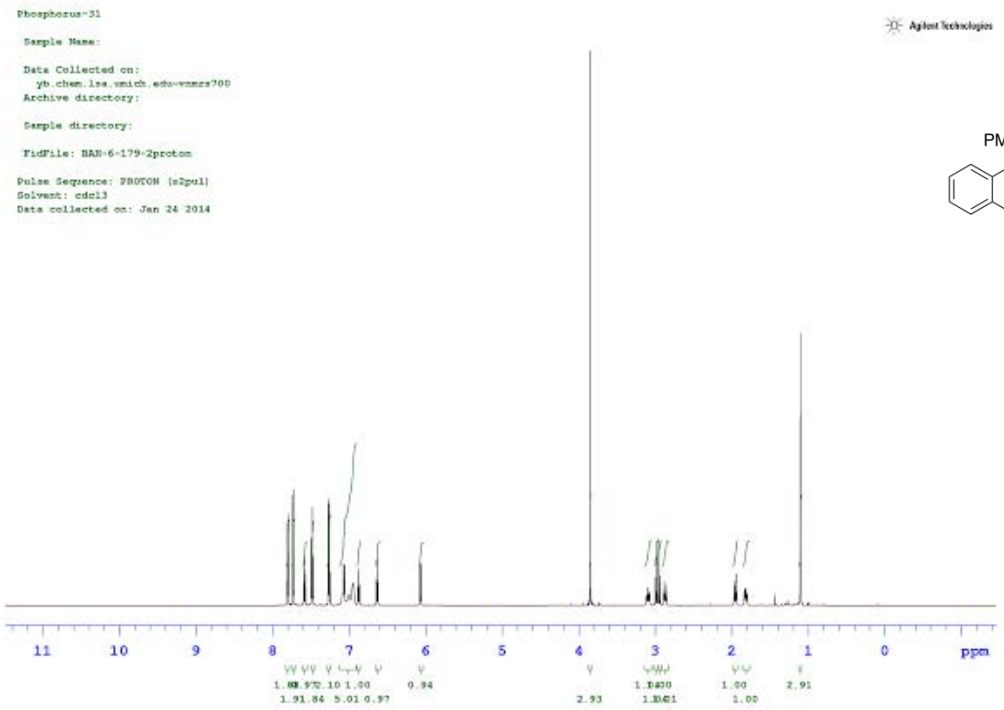


1 PDA Multi 1/254nm 4nm

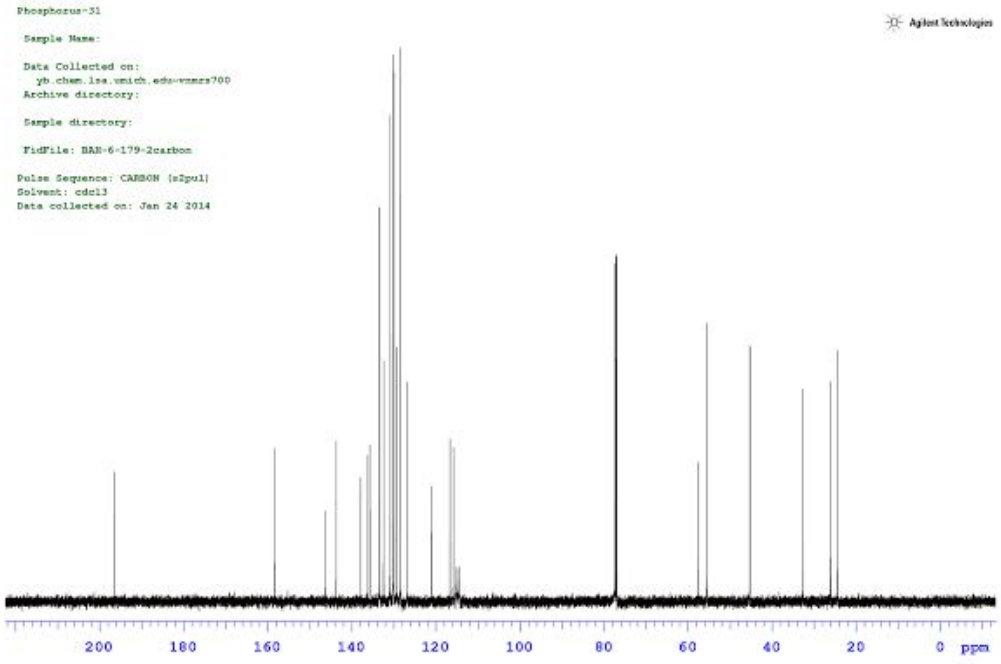
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.857	30934249	1076165	89.951	86.961
2	10.549	3455931	161366	10.049	13.039
Total		34390179	1237530	100.000	100.000


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-6-179-2proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 24 2014
Agilent Technologies
PMP
N
O
Ph
11
10
9
8
7
6
5
4
3
2
1
0
ppm

2p


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-6-179-2carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 24 2014
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
0
ppm

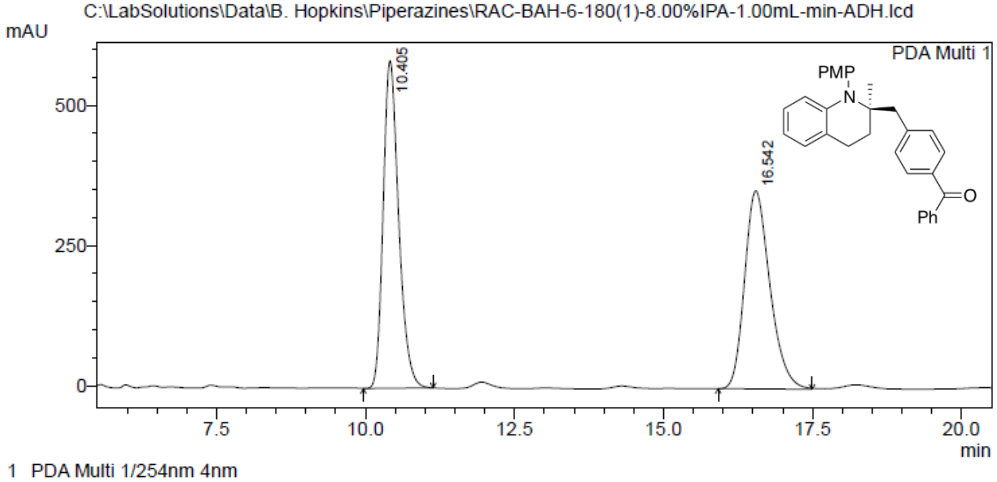
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-6-180(1)-8.00%IPA-1.00mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-180(1)-8.00%IPA-1.00mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-180(1)-8.00%IPA-1.00mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/16/2014 10:04:39 AM
Data Processed	: 1/16/2014 10:27:15 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-6-180(1)-8.00%IPA-1.00mL-min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.405	10574752	584491	50.161	62.330
2	16.542	10506929	353242	49.839	37.670
Total		21081681	937733	100.000	100.000

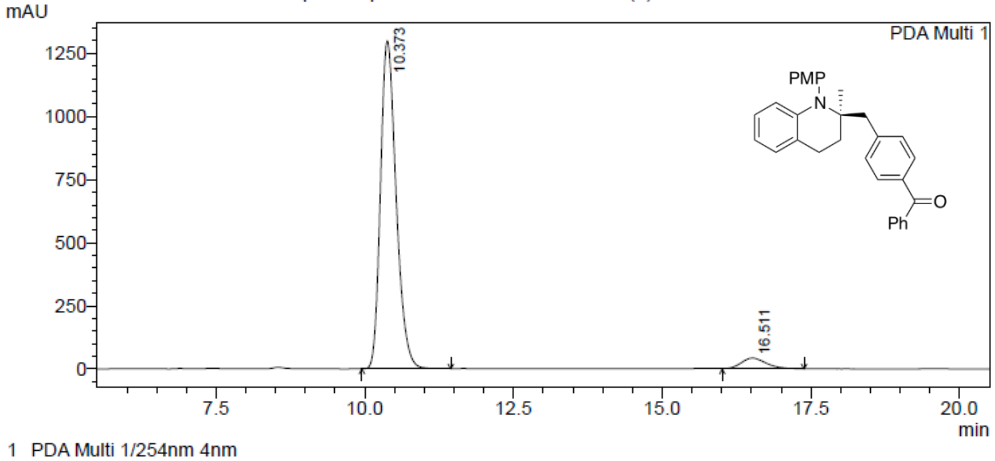
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-6-182(3)-8.00%IPA-1.00mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-6-182(3)-8.00%IPA-1.00mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-6-182(3)-8.00%IPA-1.00mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/16/2014 10:51:31 AM
Data Processed	: 1/16/2014 11:25:02 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-6-182(3)-8.00%IPA-1.00mL-min-ADH.lcd

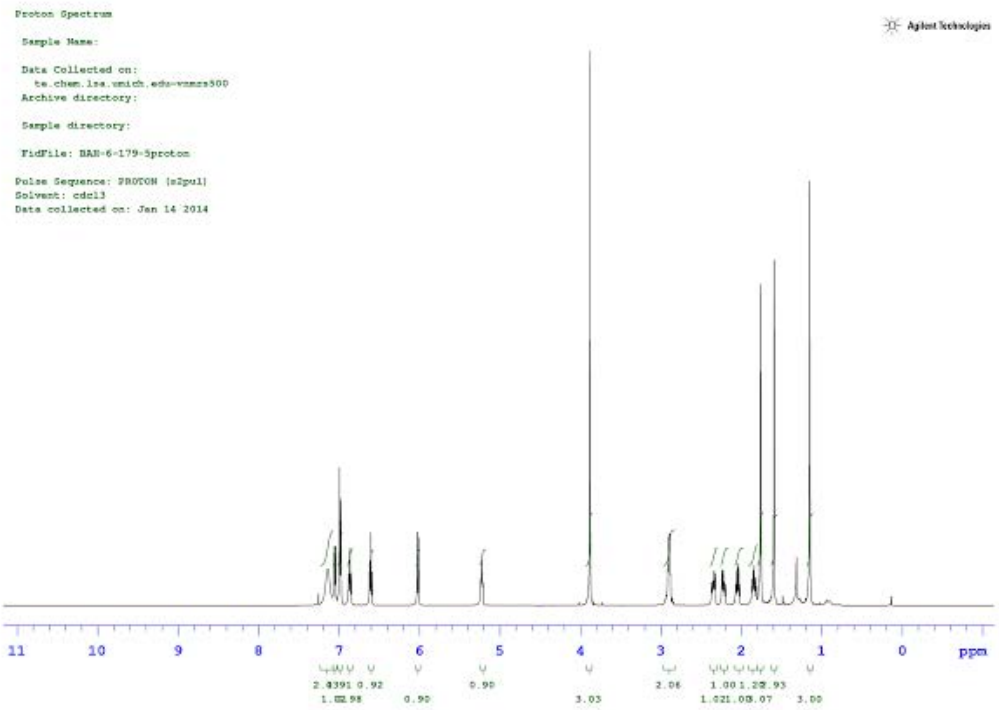


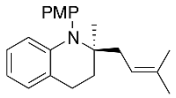
1 PDA Multi 1/254nm 4nm

PeakTable

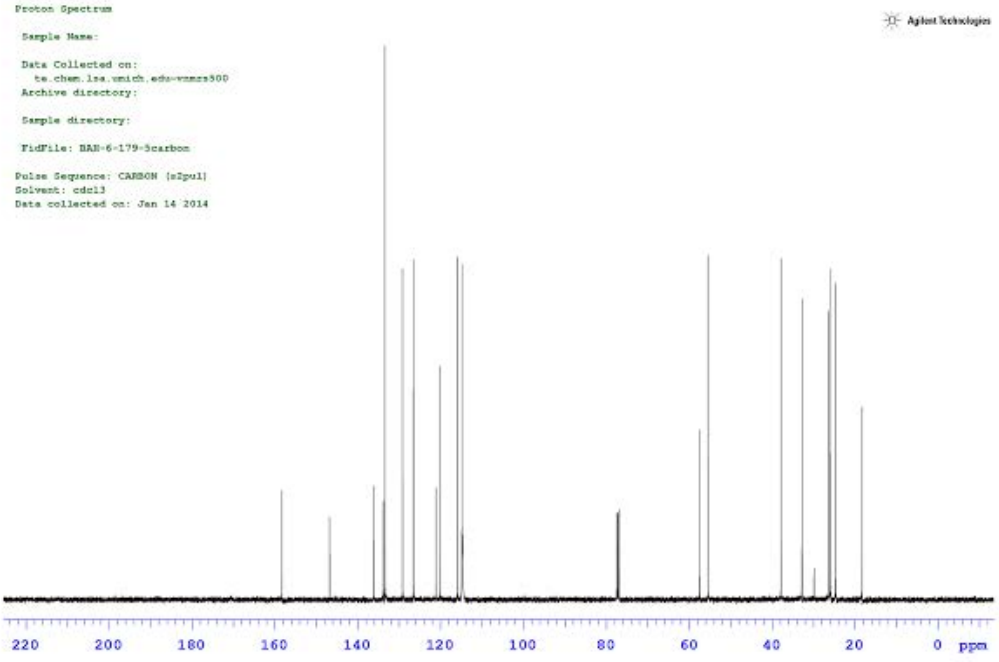
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.373	23846955	1300024	94.917	96.815
2	16.511	1277011	42771	5.083	3.185
Total		25123966	1342795	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAR-6-179-1proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 14 2014
Agilent Technologies
11
10
9
8
7
6
5
4
3
2
1
0
ppm


PMP
N

2q


Proton Spectrum
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAR-6-179-1carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 14 2014
Agilent Technologies
220
200
180
160
140
120
100
80
60
40
20
0
ppm

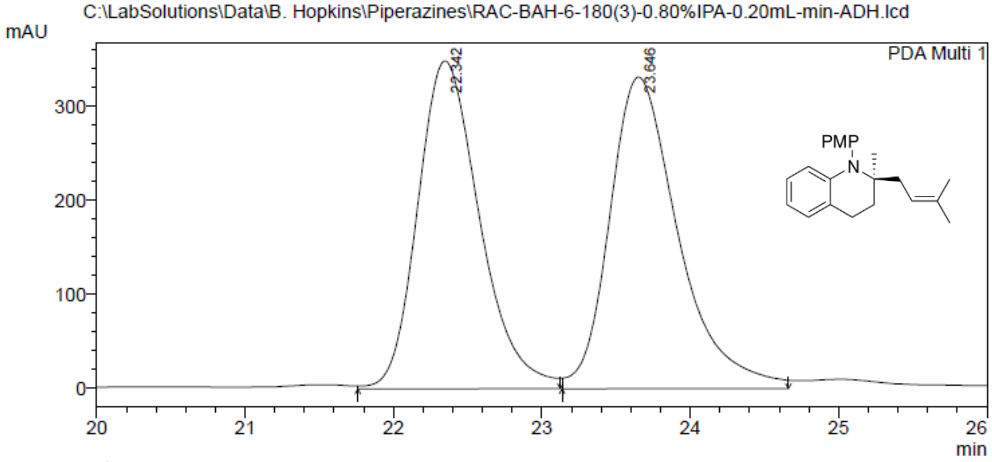
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-6-180(3)-0.80%IPA-0.20mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-180(3)-0.80%IPA-0.20mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-180(3)-0.80%IPA-0.20mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/14/2014 1:01:51 PM
Data Processed	: 1/14/2014 1:32:16 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-6-180(3)-0.80%IPA-0.20mL-min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.342	10124920	348848	48.699	51.279
2	23.646	10665911	331451	51.301	48.721
Total		20790831	680299	100.000	100.000

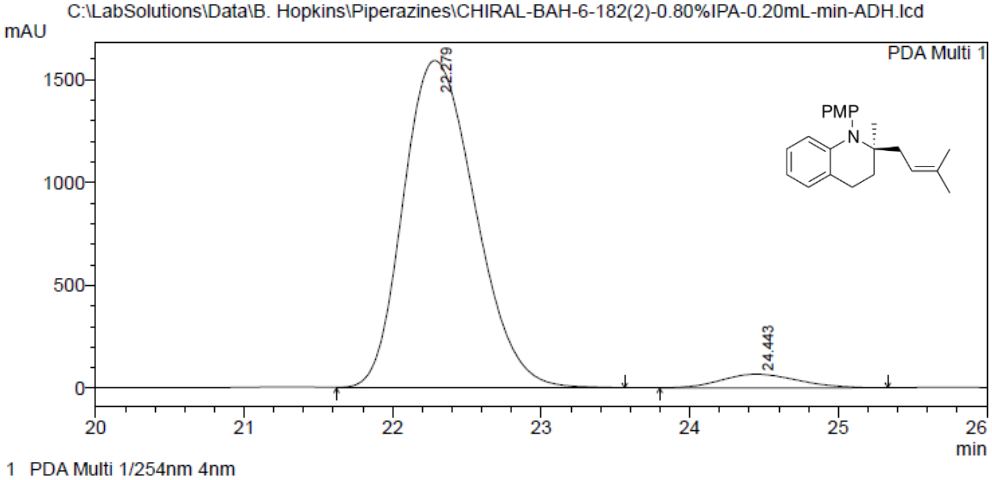
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-6-182(2)-0.80%IPA-0.20mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-6-182(2)-0.80%IPA-0.20mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-6-182(2)-0.80%IPA-0.20mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/14/2014 4:18:32 PM
Data Processed	: 1/14/2014 4:45:54 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-6-182(2)-0.80%IPA-0.20mL-min-ADH.lcd

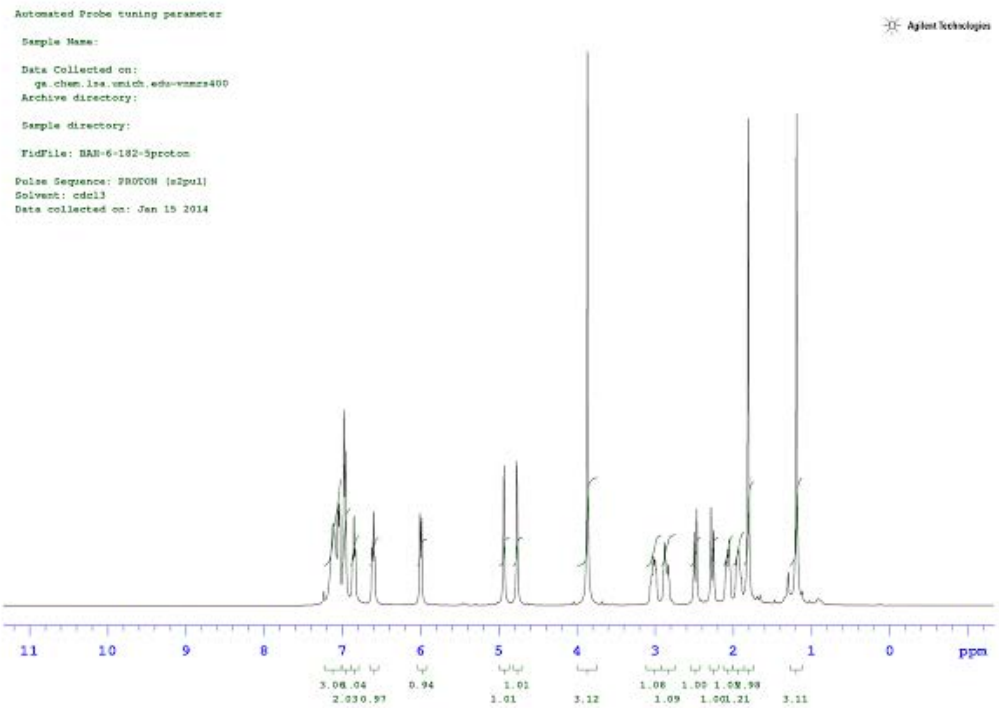


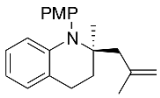
1 PDA Multi 1/254nm 4nm

PeakTable

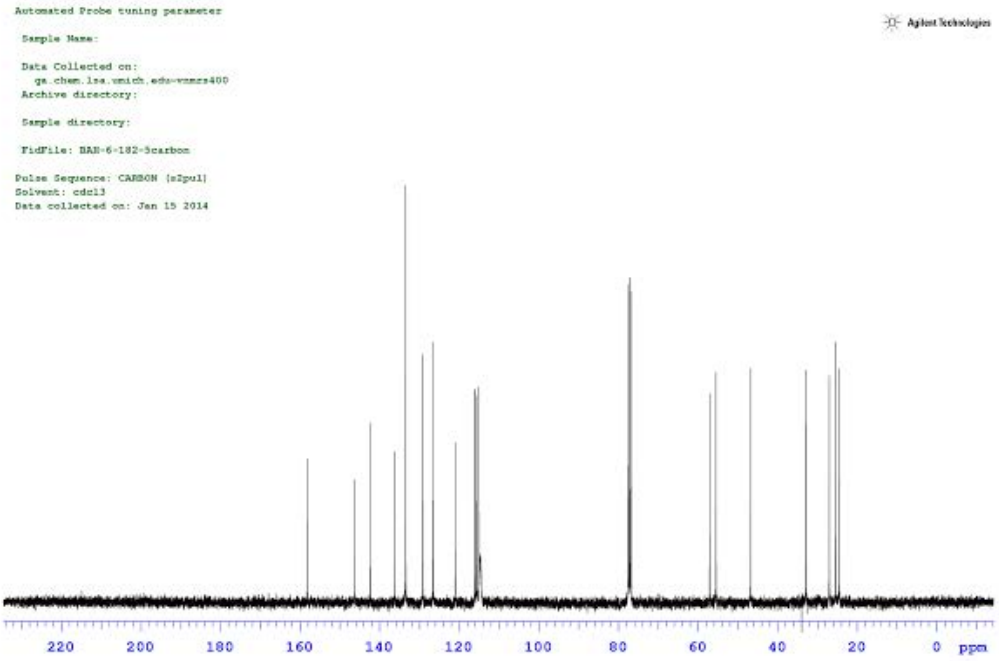
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.279	53696178	1593472	95.689	95.993
2	24.443	2418946	66516	4.311	4.007
Total		56115125	1659988	100.000	100.000


Automated Probe tuning parameter
Sample Name:
Data Collected on:
ga.chem.lsa.umich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAN-6-182-3proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 15 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
3.08 4.04 0.94 1.01 1.08 1.05 1.08 1.98
2.03 0.97 1.01 3.12 1.09 1.00 1.21 3.11


PMP
N

2r


Automated Probe tuning parameter
Sample Name:
Data Collected on:
ga.chem.lsa.umich.edu-vnmrs400
Archive directory:
Sample directory:
FidFile: BAN-6-182-3carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 15 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm

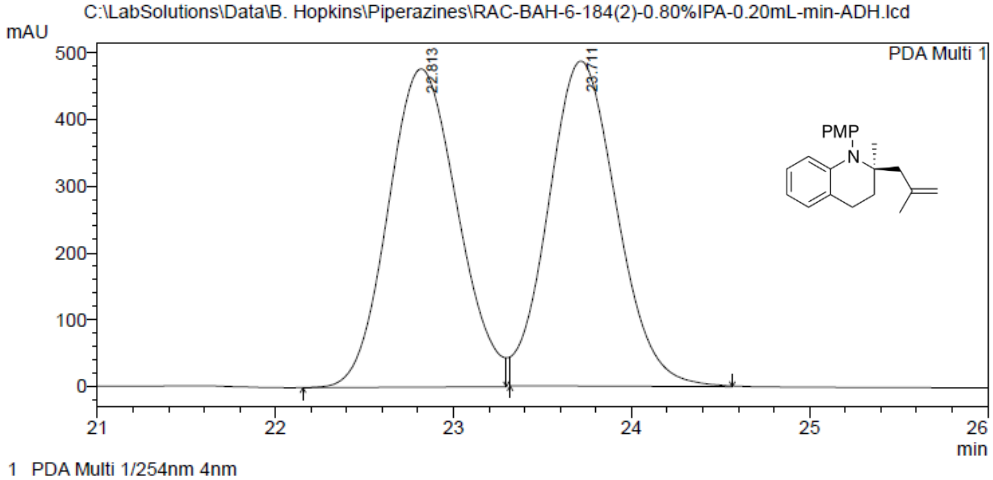
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-6-184(2)-0.80%IPA-0.20mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-184(2)-0.80%IPA-0.20mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-184(2)-0.80%IPA-0.20mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/15/2014 11:23:04 AM
Data Processed	: 1/15/2014 12:04:09 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-6-184(2)-0.80%IPA-0.20mL-min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.813	12964046	477100	49.868	49.487
2	23.711	13032606	487001	50.132	50.513
Total		25996652	964101	100.000	100.000

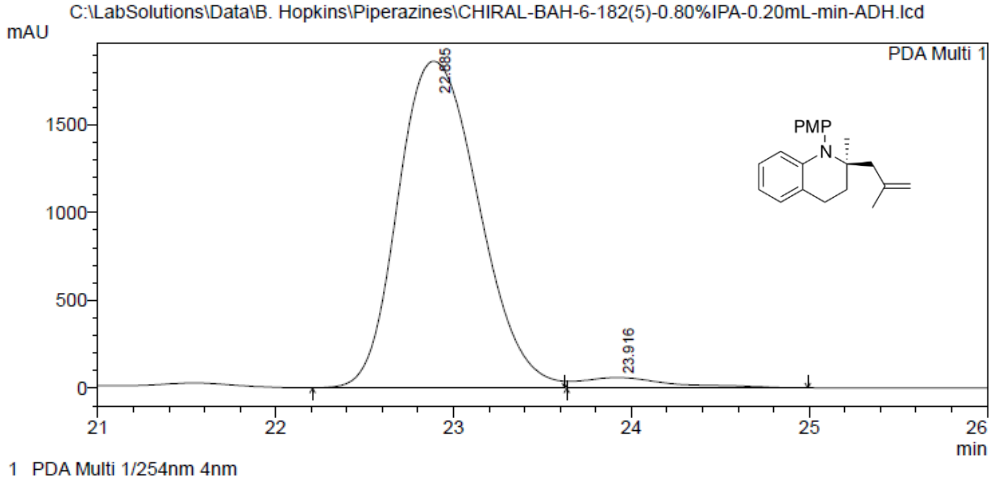
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-6-182(5)-0.80%IPA-0.20mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-6-182(5)-0.80%IPA-0.20mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-6-182(5)-0.80%IPA-0.20mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/15/2014 10:26:04 AM
Data Processed	: 1/15/2014 10:53:36 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-6-182(5)-0.80%IPA-0.20mL-min-ADH.lcd

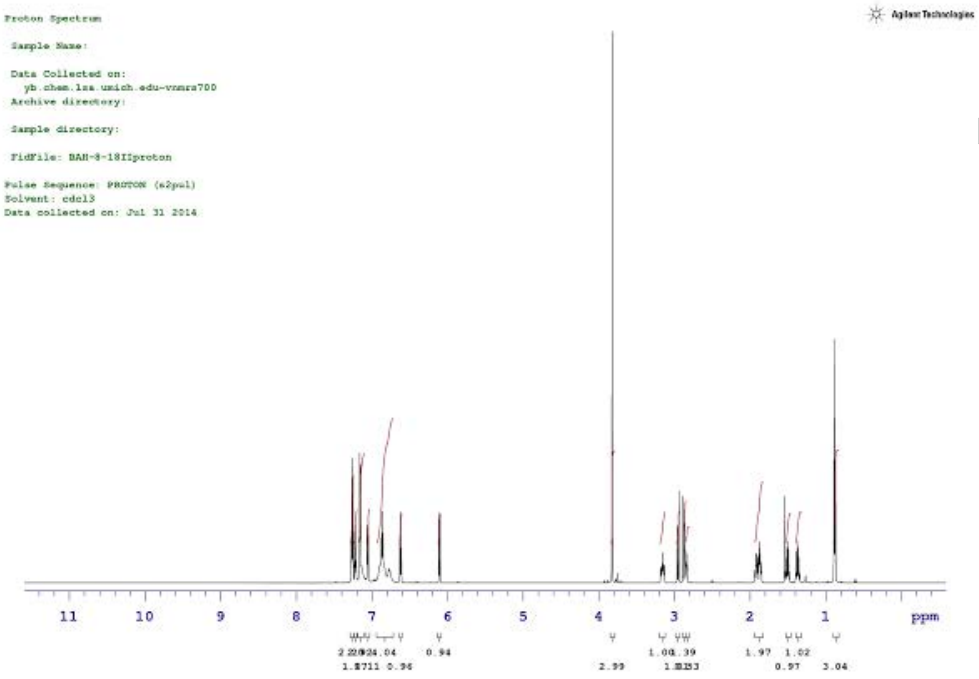


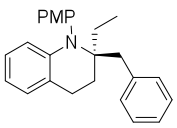
1 PDA Multi 1/254nm 4nm

PeakTable

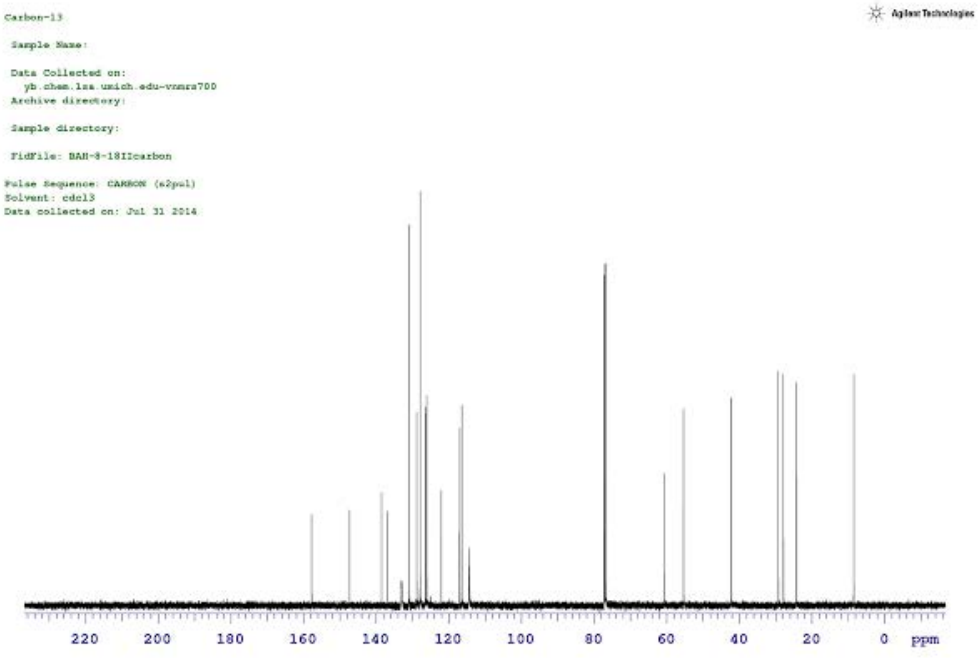
PDA Ch1 254nm 4nm

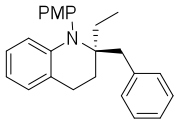
Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.885	58997531	1863335	96.459	96.883
2	23.916	2165565	59945	3.541	3.117
Total		61163096	1923281	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-8-18IIproton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 31 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 ppm


PMP
N

2s


Carbon-13
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-8-18IIcarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 31 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm



2s

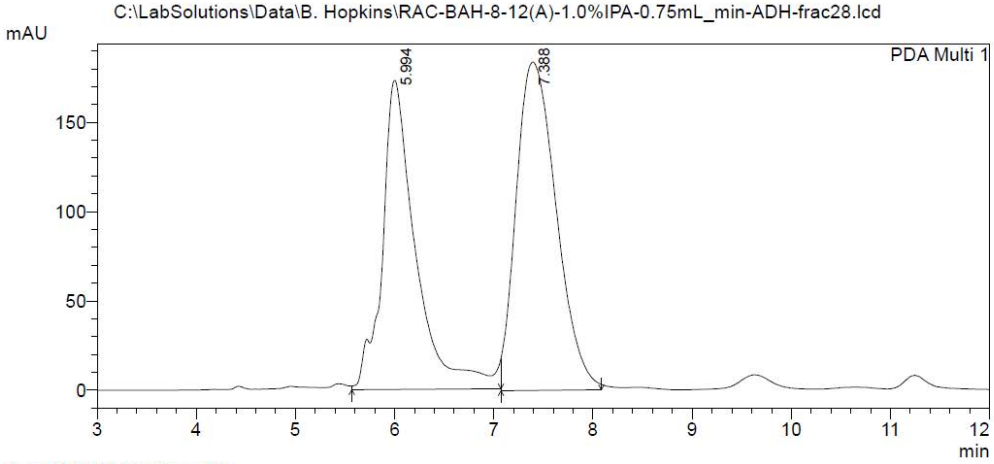
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-8-12(A)-1.0%IPA-0.75mL_min-ADH-frac28.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-8-12(A)-1.0%IPA-0.75mL_min-ADH-frac28
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-8-12(A)-1.0%IPA-0.75mL_min-ADH-frac28.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/30/2014 12:39:14 PM
Data Processed	: 7/30/2014 12:55:50 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-8-12(A)-1.0%IPA-0.75mL_min-ADH-frac28.lcd



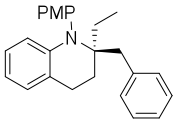
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.994	4085690	173169	44.168	48.469
2	7.388	5164640	184108	55.832	51.531
Total		9250331	357277	100.000	100.000

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

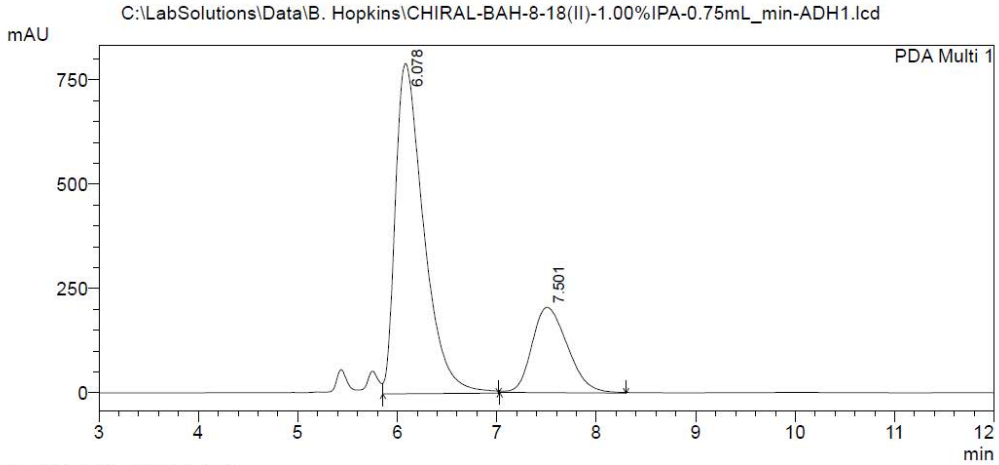


2s

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-8-18(II)-1.00%IPA-0.75mL_min-ADH1.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-8-18(II)-1.00%IPA-0.75mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-8-18(II)-1.00%IPA-0.75mL_min-ADH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/31/2014 11:06:47 PM
Data Processed	: 7/31/2014 11:34:09 PM

<Chromatogram>

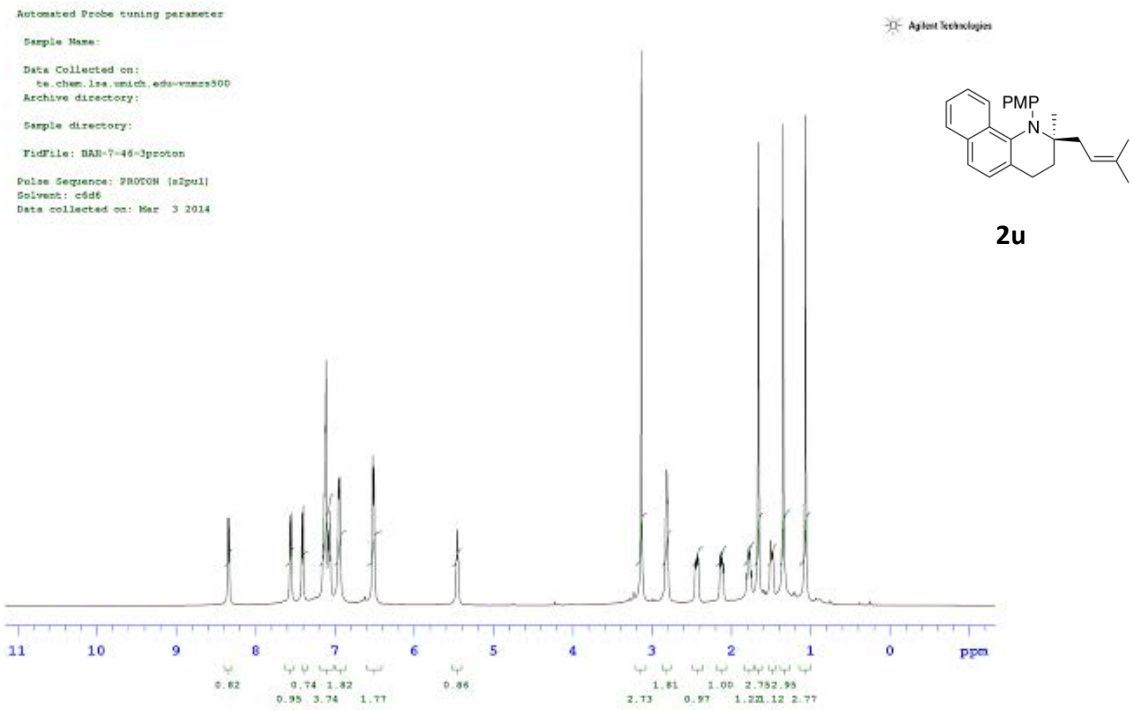


1 PDA Multi 1/254nm 4nm

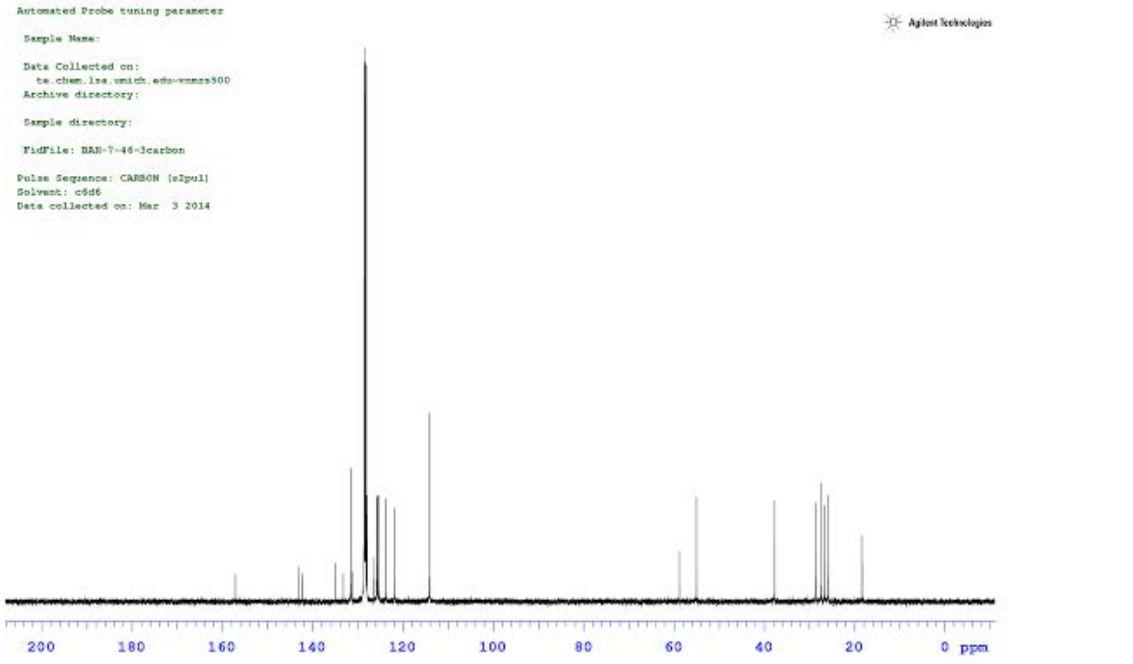
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.078	15663028	791647	75.475	79.490
2	7.501	5089644	204259	24.525	20.510
Total		20752672	995907	100.000	100.000


Automated Probe tuning parameter
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-7-46-3proton
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Mar 3 2014
Agilent Technologies
PMP
N
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.82 0.74 1.82 0.86 1.81 1.00 2.752.95
0.95 3.74 1.77 2.73 0.97 1.221.12 2.77

2u


Automated Probe tuning parameter
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-7-46-3carbon
Pulse Sequence: CARBON (s2pul)
Solvent: c6d6
Data collected on: Mar 3 2014
Agilent Technologies
200 180 160 140 120 100 80 60 40 20 0 ppm

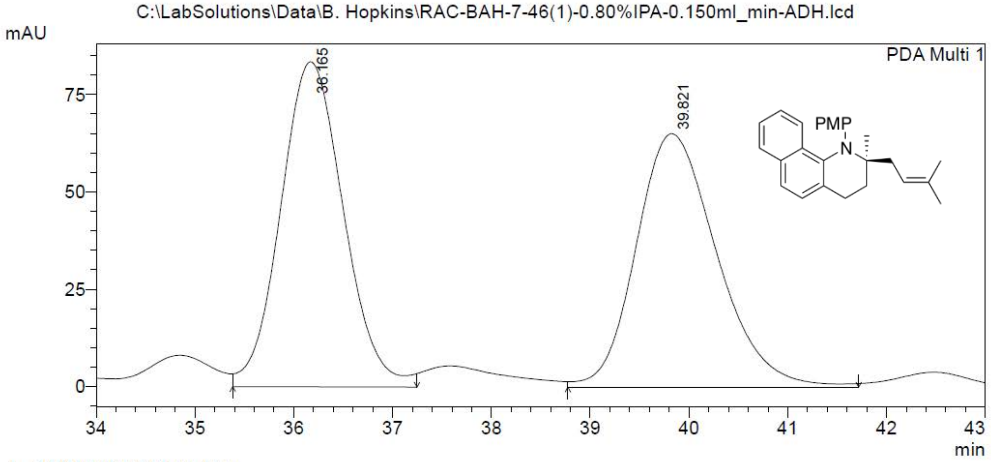
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-7-46(1)-0.80%IPA-0.150ml_min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-7-46(1)-0.80%IPA-0.150ml_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-7-46(1)-0.80%IPA-0.150ml_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/3/2014 11:56:54 AM
Data Processed	: 3/3/2014 1:01:56 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-7-46(1)-0.80%IPA-0.150ml_min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

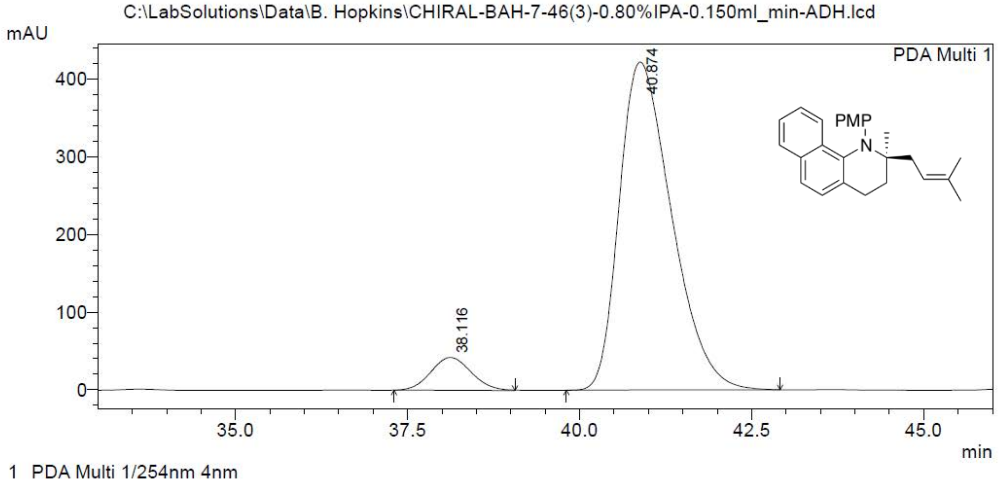
Peak#	Ret. Time	Area	Height	Area %	Height %
1	36.165	3689756	83439	50.002	56.201
2	39.821	3689391	65026	49.998	43.799
Total		7379146	148465	100.000	100.000

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

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-7-46(3)-0.80%IPA-0.150ml_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-7-46(3)-0.80%IPA-0.150ml_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-7-46(3)-0.80%IPA-0.150ml_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/3/2014 10:52:41 AM
Data Processed	: 3/3/2014 11:54:53 AM

<Chromatogram>

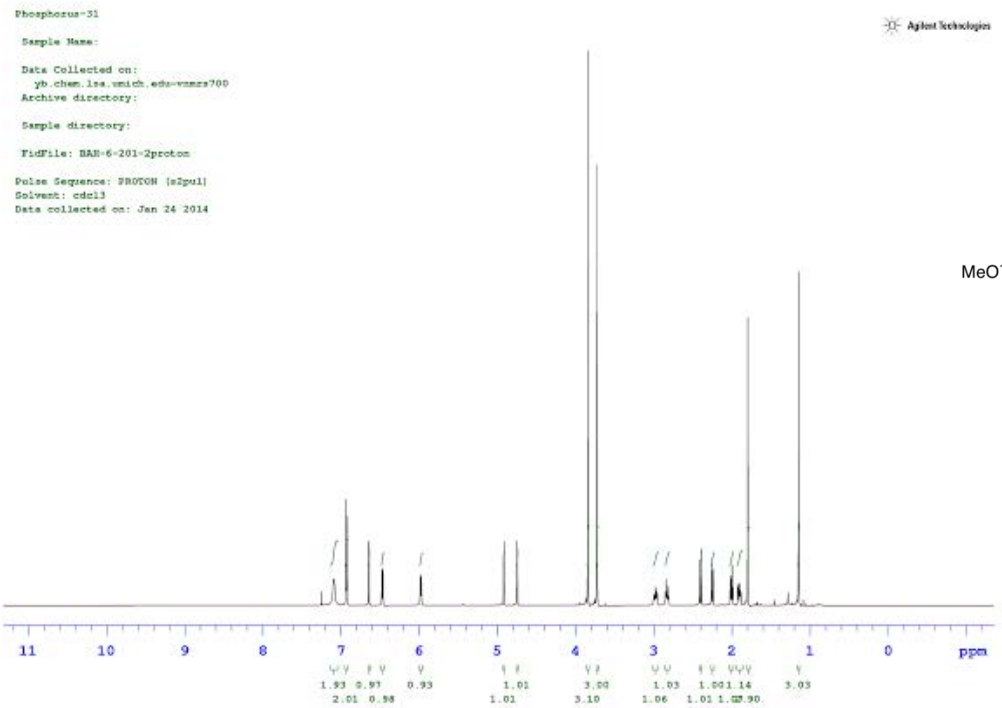


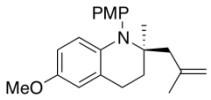
1 PDA Multi 1/254nm 4nm

PeakTable

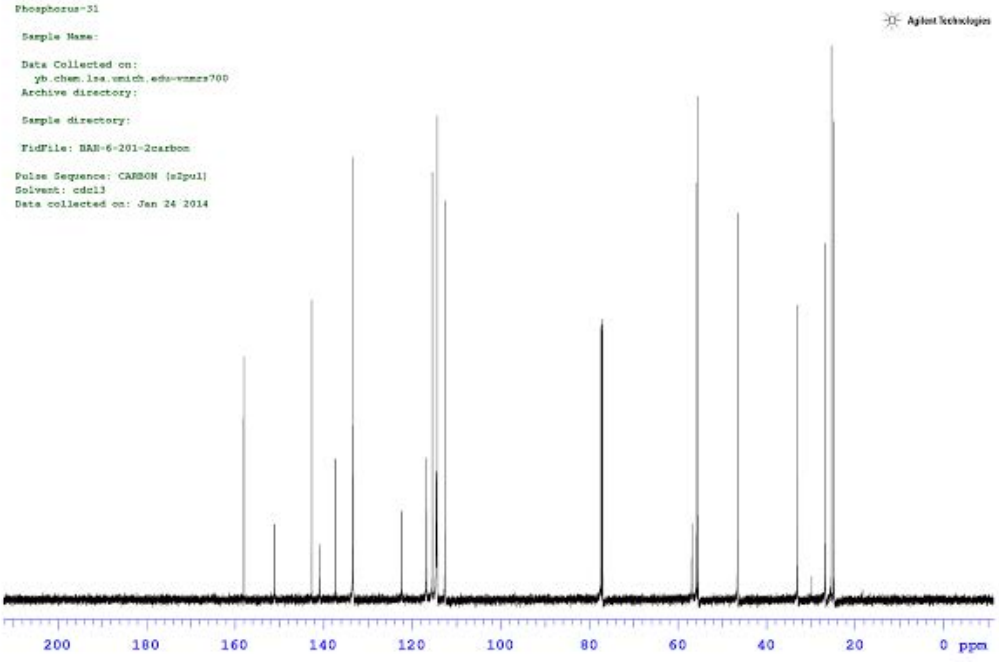
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	38.116	1724903	42239	7.171	9.099
2	40.874	22328018	421977	92.829	90.901
Total		24052921	464216	100.000	100.000


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-6-201-2proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 24 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.93 0.97 0.93 1.01 3.00 1.03 1.00 1.14 3.03
2.01 0.98 1.01 3.10 1.06 1.01 1.02 0.90


PMP
N
MeO

2t


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-6-201-2carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 24 2014
Agilent Technologies
200 180 160 140 120 100 80 60 40 20 0 ppm

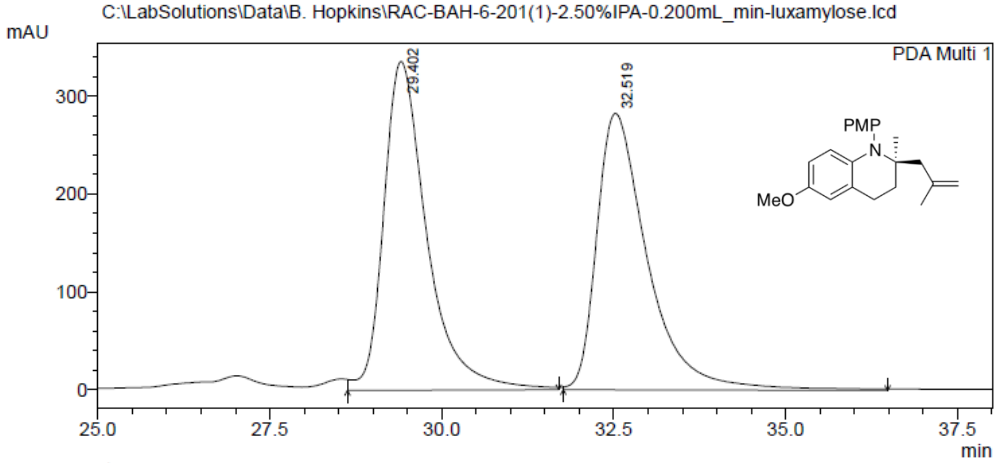
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-201(1)-2.50%IPA-0.200mL_min-luxamylose.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-201(1)-2.50%IPA-0.200mL_min-luxamylose
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-201(1)-2.50%IPA-0.200mL_min-luxamylose.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/3/2014 1:55:36 PM
Data Processed	: 2/3/2014 3:05:38 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-201(1)-2.50%IPA-0.200mL_min-luxamylose.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.402	14676356	335547	50.253	54.331
2	32.519	14528617	282050	49.747	45.669
Total		29204973	617597	100.000	100.000

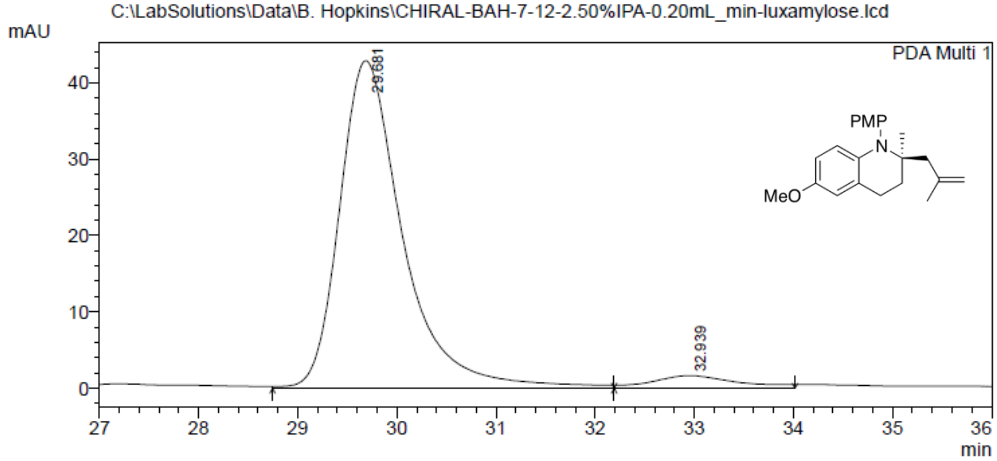
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-7-12-2.50%IPA-0.20mL_min-luxamylose.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-7-12-2.50%IPA-0.20mL_min-luxamylose
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-7-12-2.50%IPA-0.20mL_min-luxamylose.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/5/2014 2:48:02 PM
Data Processed	: 2/5/2014 3:34:27 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-7-12-2.50%IPA-0.20mL_min-luxamylose.lcd

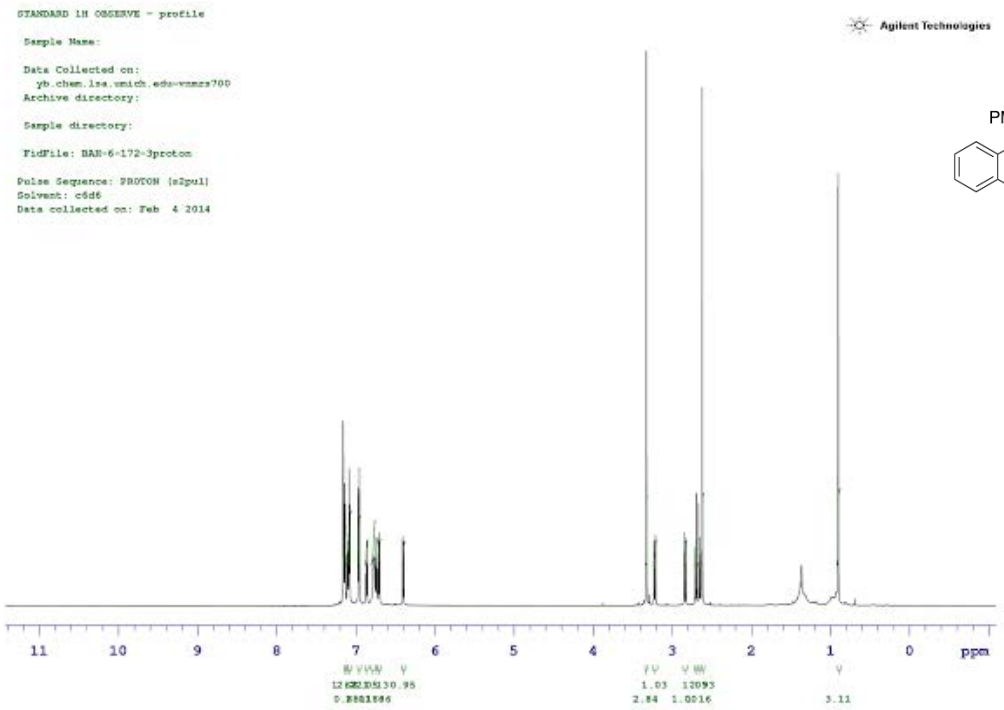


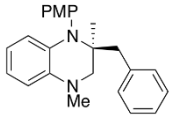
1 PDA Multi 1/254nm 4nm

PeakTable

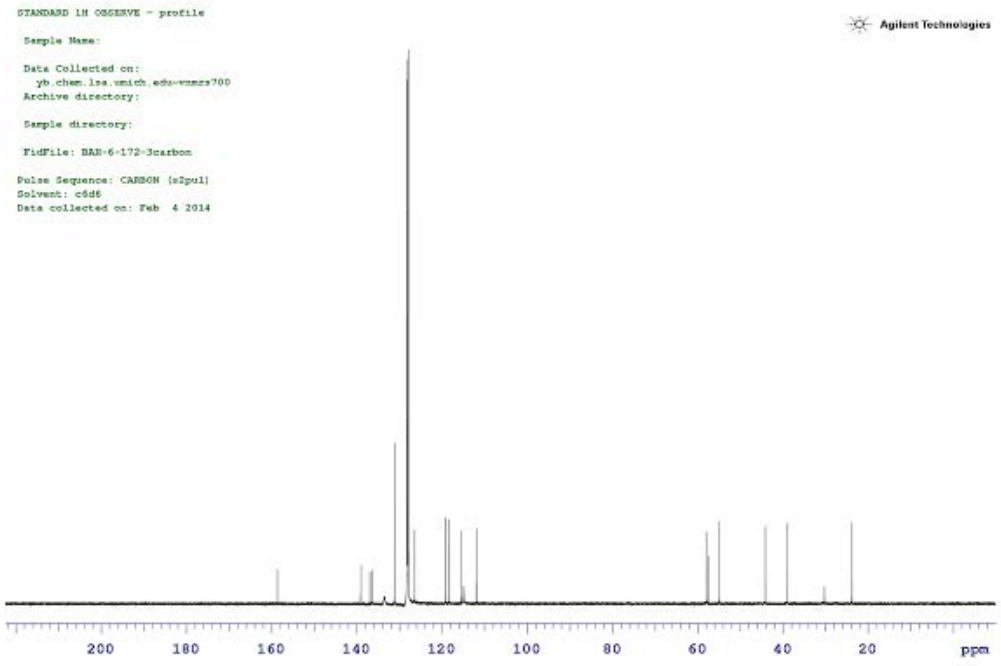
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.681	1908292	42931	95.009	96.295
2	32.939	100237	1652	4.991	3.705
Total		2008530	44584	100.000	100.000


STANDARD 1H OBSERVE - profile
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-6-172-3proton
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Feb 4 2014
Agilent Technologies
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.03
2.84
1.0016
3.11


PMP
N
N
Me

4a


STANDARD 1H OBSERVE - profile
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-6-172-3carbon
Pulse Sequence: CARBON (s2pul)
Solvent: c6d6
Data collected on: Feb 4 2014
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
ppm

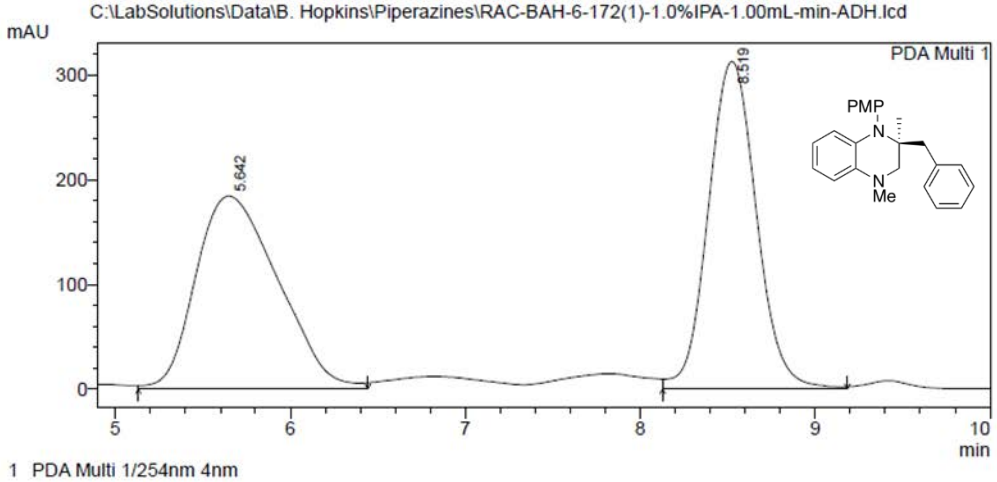
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-6-172(1)-1.0%IPA-1.00mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-172(1)-1.0%IPA-1.00mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-172(1)-1.0%IPA-1.00mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/3/2014 10:42:36 AM
Data Processed	: 1/3/2014 11:03:37 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\RAC-BAH-6-172(1)-1.0%IPA-1.00mL-min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.642	6050836	184529	50.234	37.039
2	8.519	5994384	313674	49.766	62.961
Total		12045221	498203	100.000	100.000

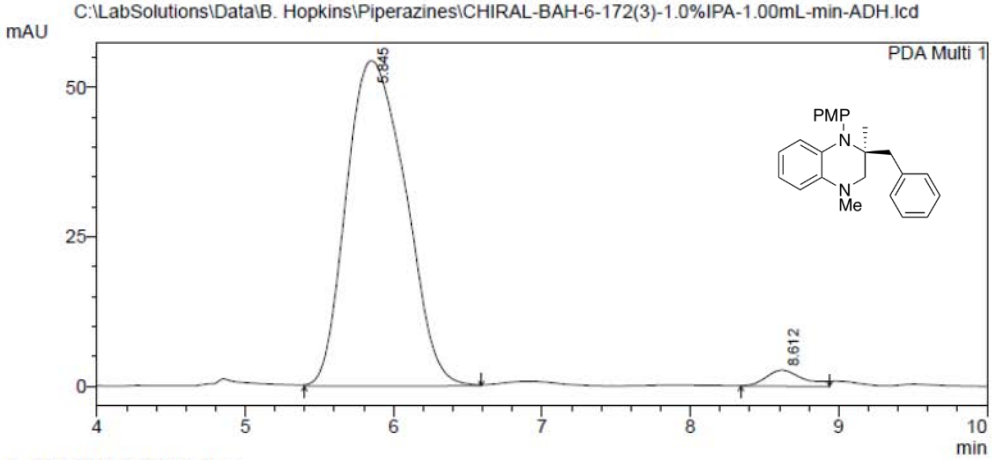
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-6-172(3)-1.0%IPA-1.00mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-6-172(3)-1.0%IPA-1.00mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-6-172(3)-1.0%IPA-1.00mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/3/2014 11:04:35 AM
Data Processed	: 1/3/2014 11:44:24 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\Piperazines\CHIRAL-BAH-6-172(3)-1.0%IPA-1.00mL-min-ADH.lcd

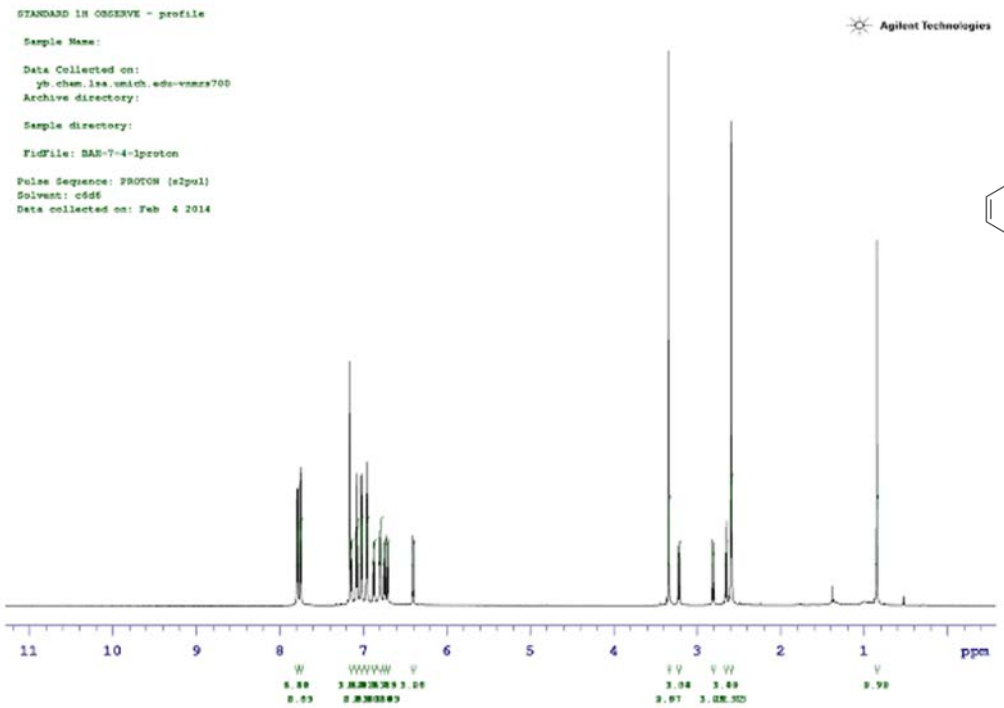


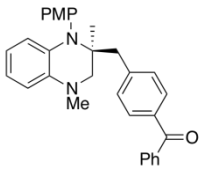
1 PDA Multi 1/254nm 4nm

PeakTable

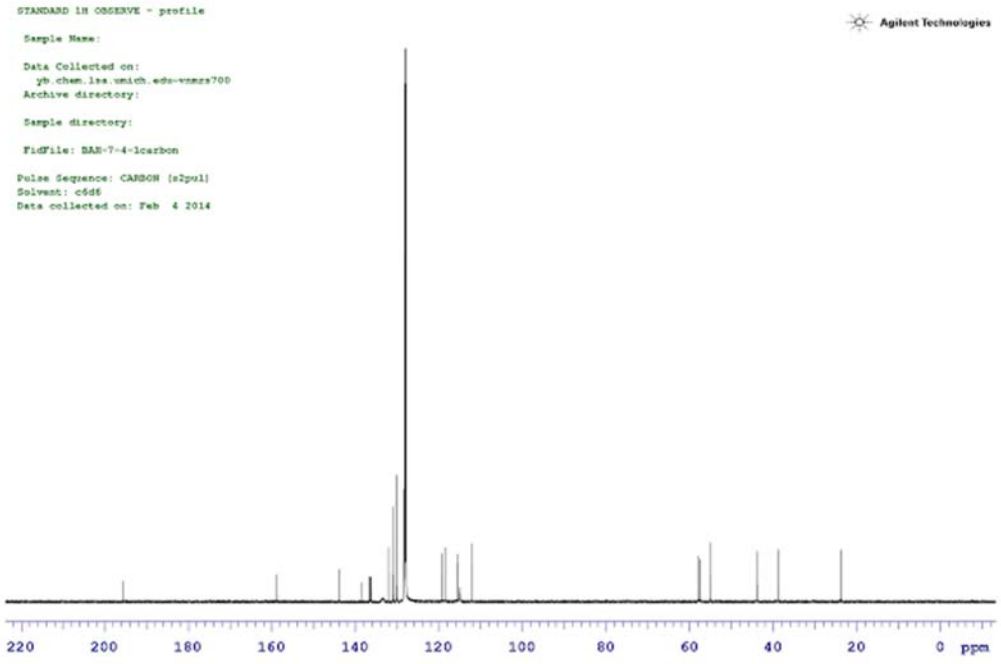
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.845	1513579	54486	96.950	95.315
2	8.612	47623	2678	3.050	4.685
Total		1561202	57165	100.000	100.000


STANDARD 1H OBSERVE - profile
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-7-4-1proton
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Feb 4 2014
Agilent Technologies
11
10
9
8
7
6
5
4
3
2
1
ppm


PMP
N
N
Me
O
Ph

4b


STANDARD 1H OBSERVE - profile
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-7-4-1carbon
Pulse Sequence: CARBON (s2pul)
Solvent: c6d6
Data collected on: Feb 4 2014
Agilent Technologies
220
200
180
160
140
120
100
80
60
40
20
0
ppm

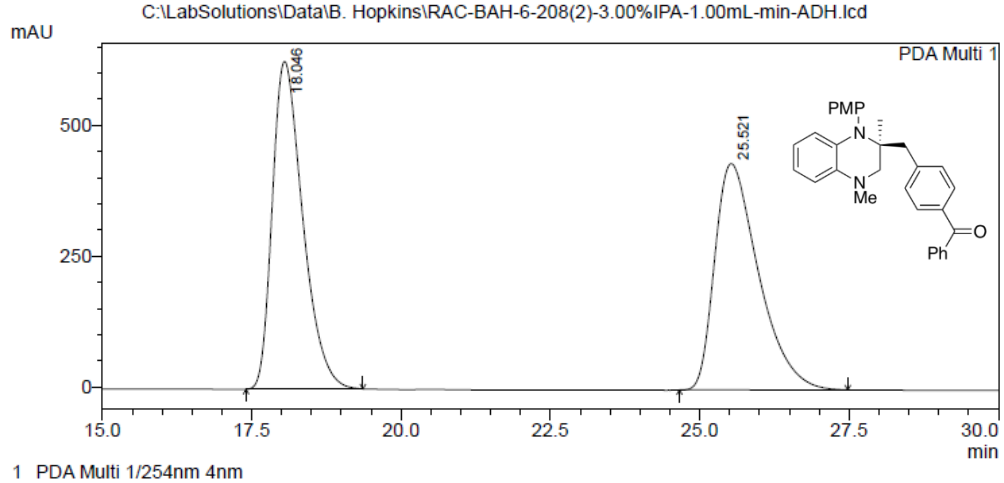
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-208(2)-3.00%IPA-1.00mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-208(2)-3.00%IPA-1.00mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-208(2)-3.00%IPA-1.00mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/29/2014 10:35:25 AM
Data Processed	: 1/29/2014 11:10:54 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-208(2)-3.00%IPA-1.00mL-min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.046	22208539	625945	50.091	59.164
2	25.521	22127776	432046	49.909	40.836
Total		44336314	1057991	100.000	100.000

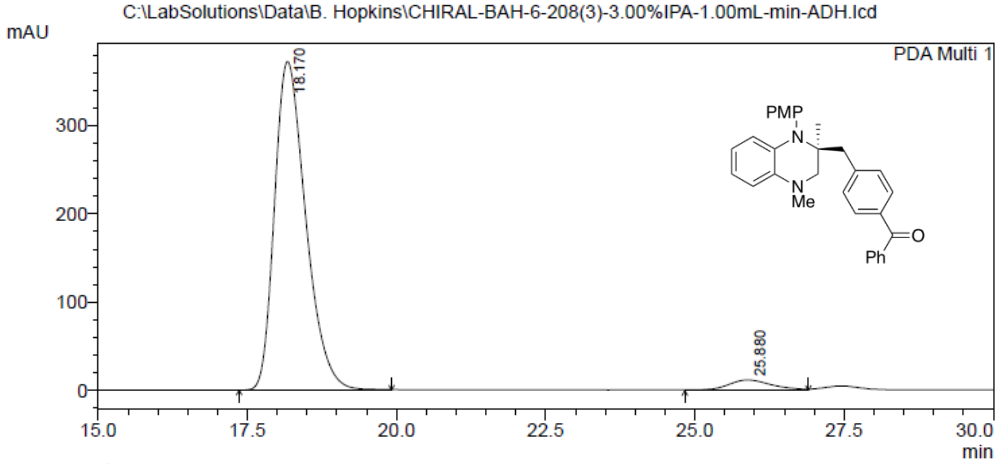
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-6-208(3)-3.00%IPA-1.00mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-6-208(3)-3.00%IPA-1.00mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-6-208(3)-3.00%IPA-1.00mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/28/2014 6:34:23 PM
Data Processed	: 1/28/2014 7:17:08 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-6-208(3)-3.00%IPA-1.00mL-min-ADH.lcd

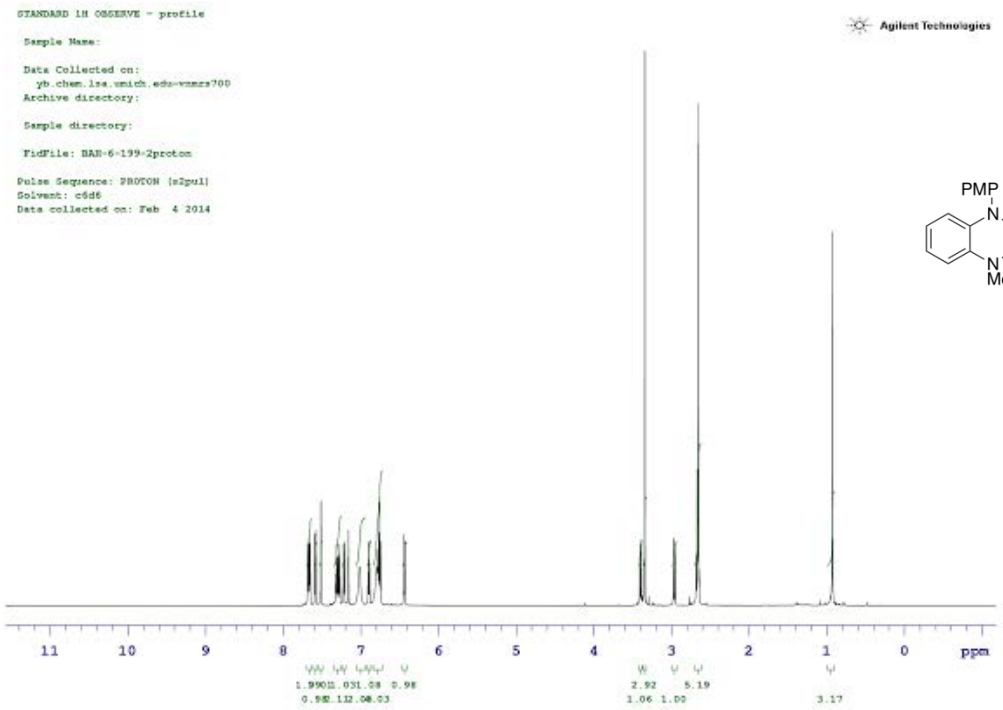


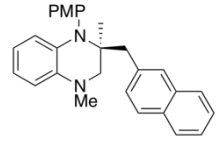
1 PDA Multi 1/254nm 4nm

PeakTable

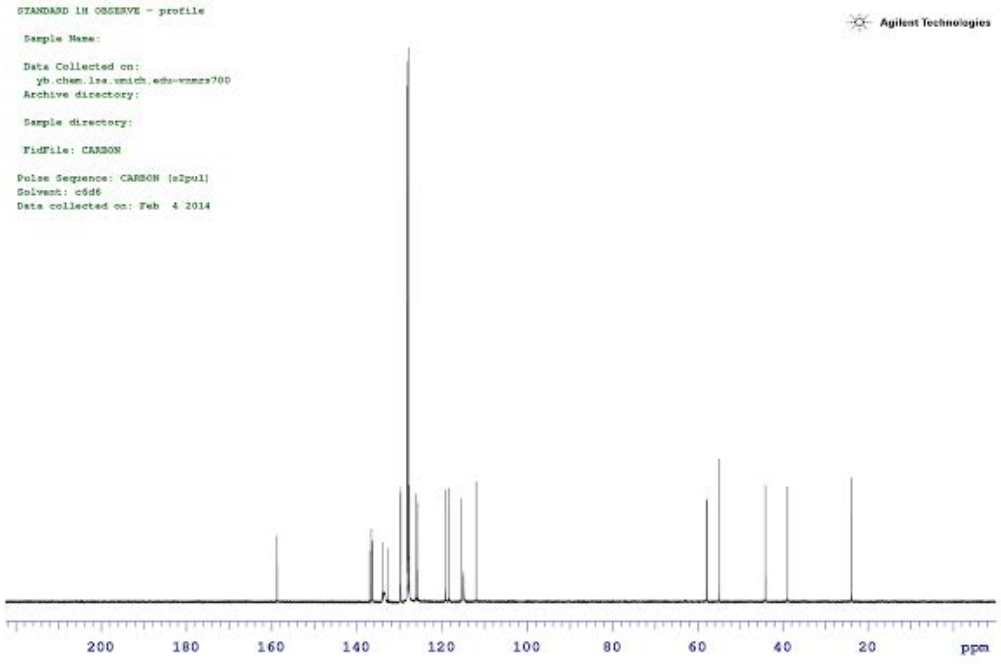
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.170	13571968	372705	95.552	96.911
2	25.880	631853	11878	4.448	3.089
Total		14203821	384583	100.000	100.000


STANDARD 1H OBSERVE - profile
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-6-199-2proton
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Feb 4 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.99 11.03 1.08 0.98
0.98 12.08 3.03
2.92 5.19
1.06 1.00
3.17


PMP
N
N
Me

4c


STANDARD 1H OBSERVE - profile
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: c6d6
Data collected on: Feb 4 2014
Agilent Technologies
200 180 160 140 120 100 80 60 40 20 ppm

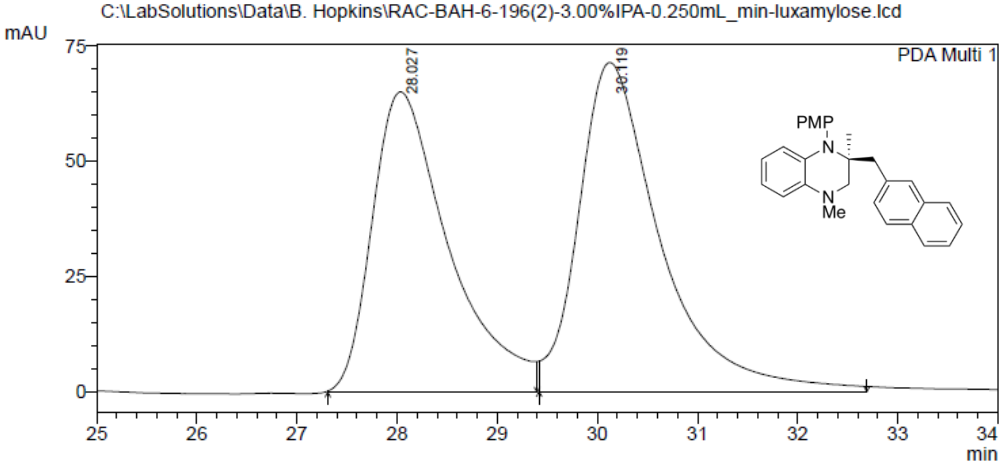
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-196(2)-3.00%IPA-0.250mL_min-luxamylose.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-196(2)-3.00%IPA-0.250mL_min-luxamylose
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-196(2)-3.00%IPA-0.250mL_min-luxamylose.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/31/2014 9:01:50 PM
Data Processed	: 1/31/2014 9:56:24 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-196(2)-3.00%IPA-0.250mL_min-luxamylose.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	28.027	3467850	65168	46.106	47.709
2	30.119	4053656	71426	53.894	52.291
Total		7521506	136594	100.000	100.000

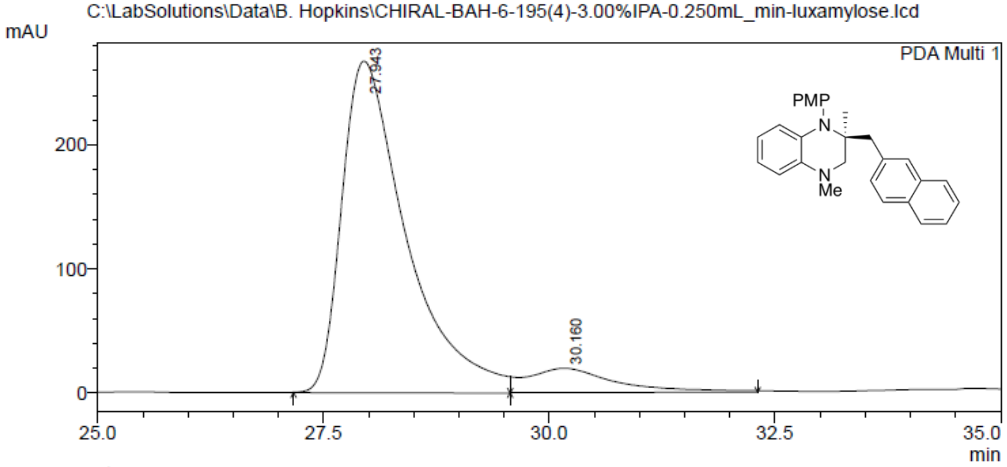
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-6-195(4)-3.00%IPA-0.250mL_min-luxamylose.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-6-195(4)-3.00%IPA-0.250mL_min-luxamylose
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-6-195(4)-3.00%IPA-0.250mL_min-luxamylose.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/31/2014 9:58:24 PM
Data Processed	: 1/31/2014 10:35:08 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-6-195(4)-3.00%IPA-0.250mL_min-luxamylose.lcd

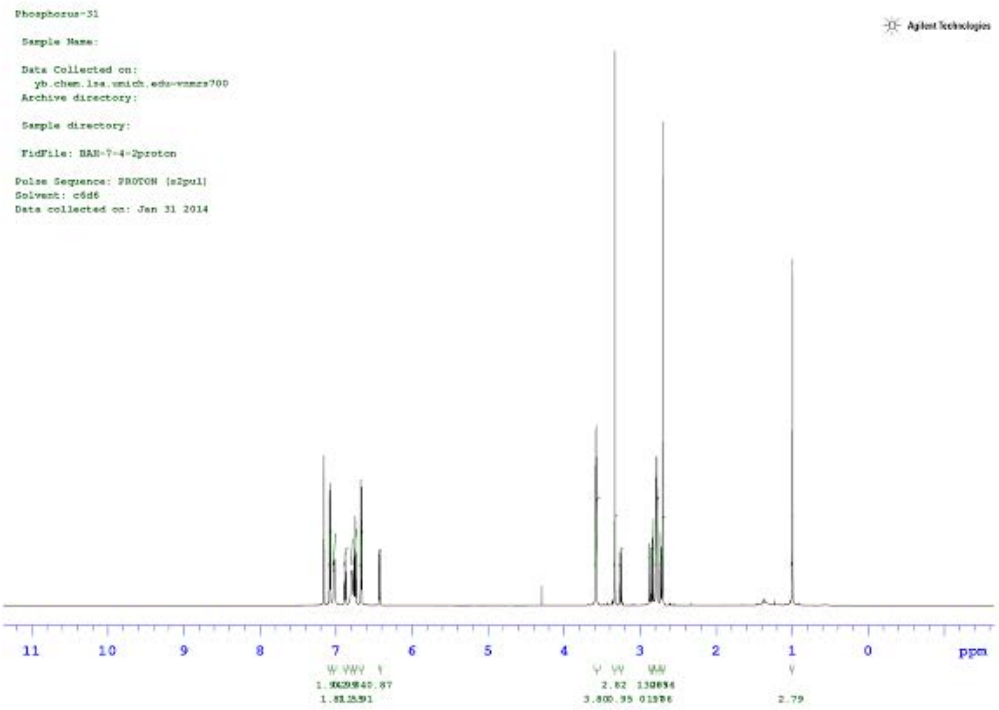


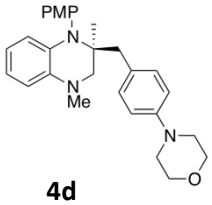
1 PDA Multi 1/254nm 4nm

PeakTable

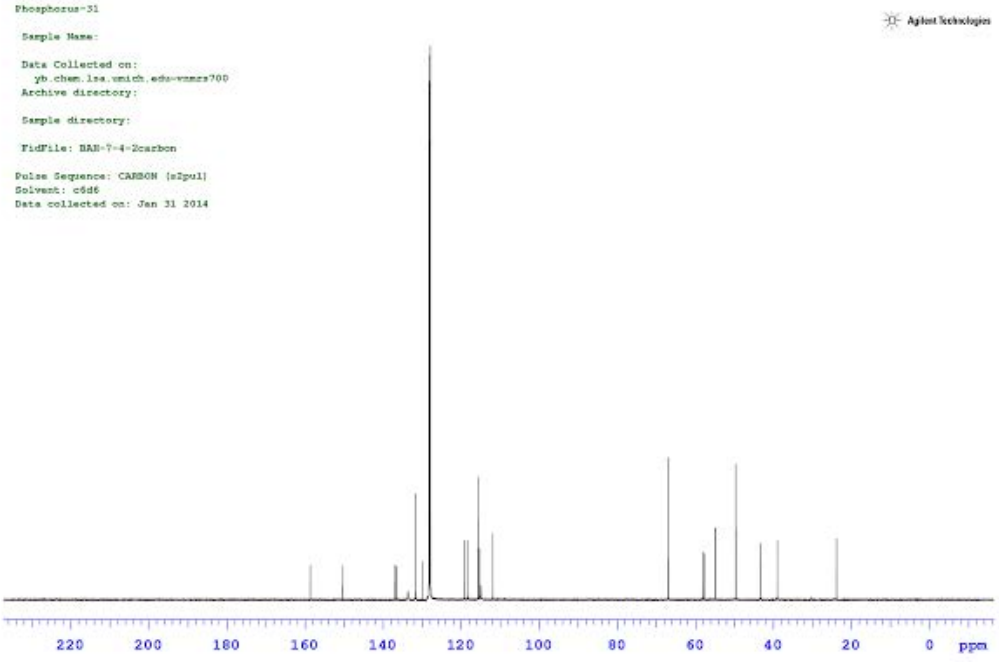
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	27.943	13313508	267455	90.769	93.187
2	30.160	1353989	19553	9.231	6.813
Total		14667497	287008	100.000	100.000


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-7-4-2proton
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Jan 31 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.82
2.79


PMP
N
N
Me
N
O

4d


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-7-4-2carbon
Pulse Sequence: CARBON (s2pul)
Solvent: c6d6
Data collected on: Jan 31 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm

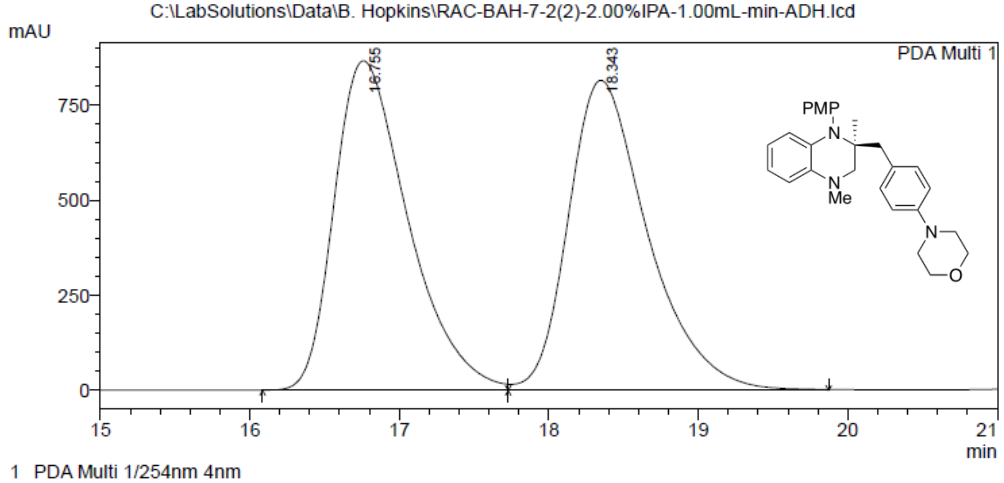
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-7-2(2)-2.00%IPA-1.00mL-min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-7-2(2)-2.00%IPA-1.00mL-min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-7-2(2)-2.00%IPA-1.00mL-min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/29/2014 3:02:32 PM
Data Processed	: 1/29/2014 3:30:05 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-7-2(2)-2.00%IPA-1.00mL-min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.755	29828474	867318	49.657	51.523
2	18.343	30240501	816046	50.343	48.477
Total		60068974	1683364	100.000	100.000

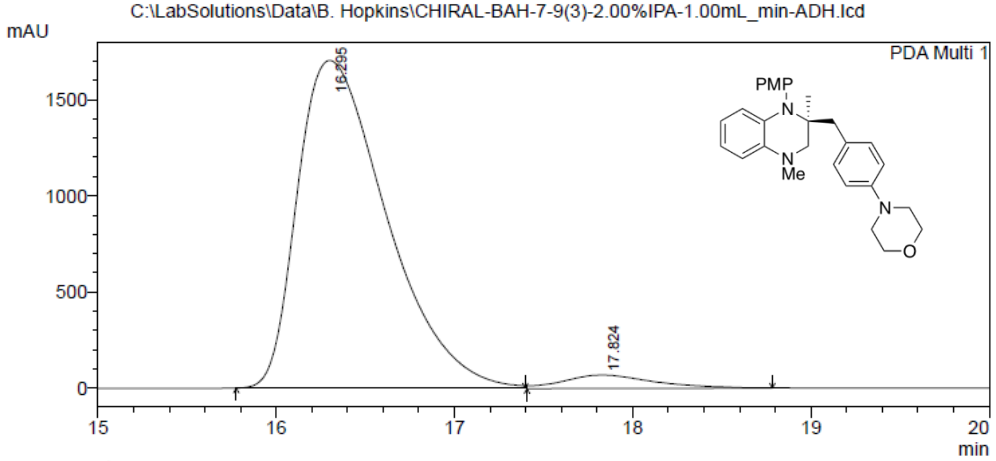
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-7-9(3)-2.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-7-9(3)-2.00%IPA-1.00mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-7-9(3)-2.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/4/2014 12:06:47 PM
Data Processed	: 2/4/2014 12:27:13 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-7-9(3)-2.00%IPA-1.00mL_min-ADH.lcd

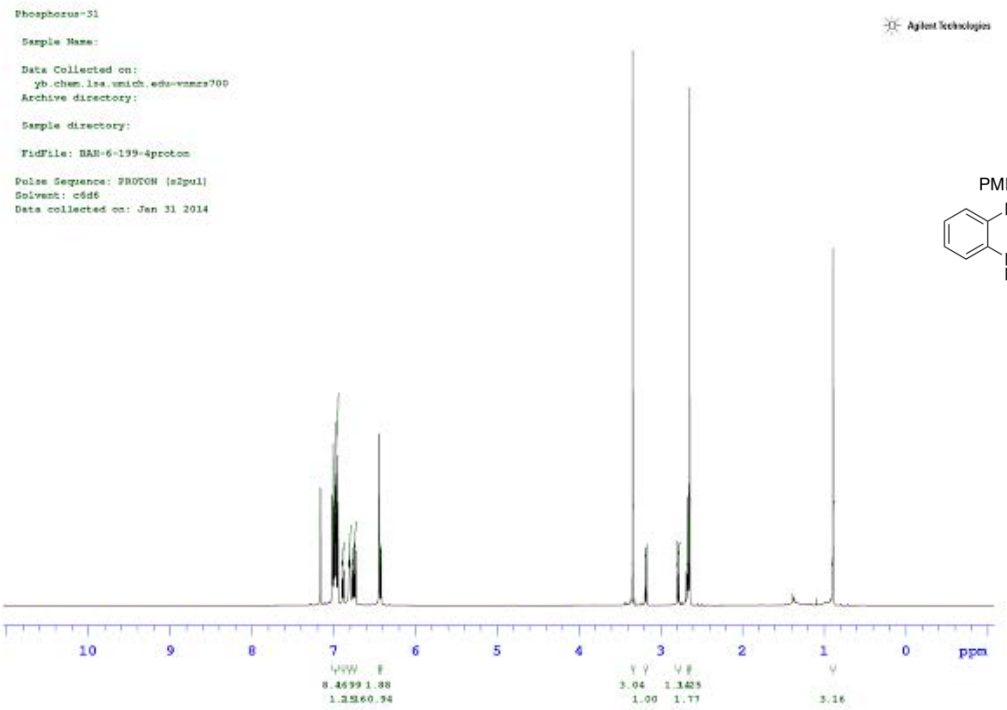


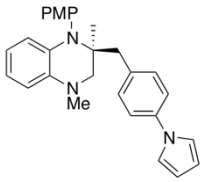
1 PDA Multi 1/254nm 4nm

PeakTable

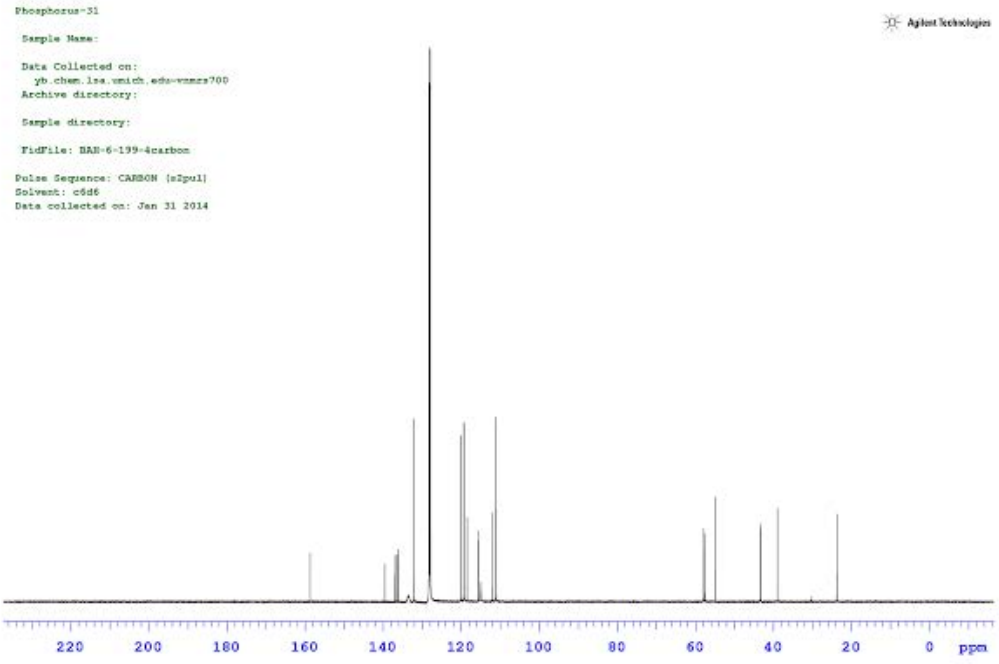
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.295	59174910	1702208	96.009	96.053
2	17.824	2459936	69947	3.991	3.947
Total		61634846	1772156	100.000	100.000


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-199-4proton
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Jan 31 2014
Agilent Technologies
10 9 8 7 6 5 4 3 2 1 0 ppm
8.4699 1.88
1.3560.94
3.04 1.3425
1.00 1.77
3.16


PMP
N
N
Me
N

4e


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-199-4carbon
Pulse Sequence: CARBON (s2pul)
Solvent: c6d6
Data collected on: Jan 31 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm

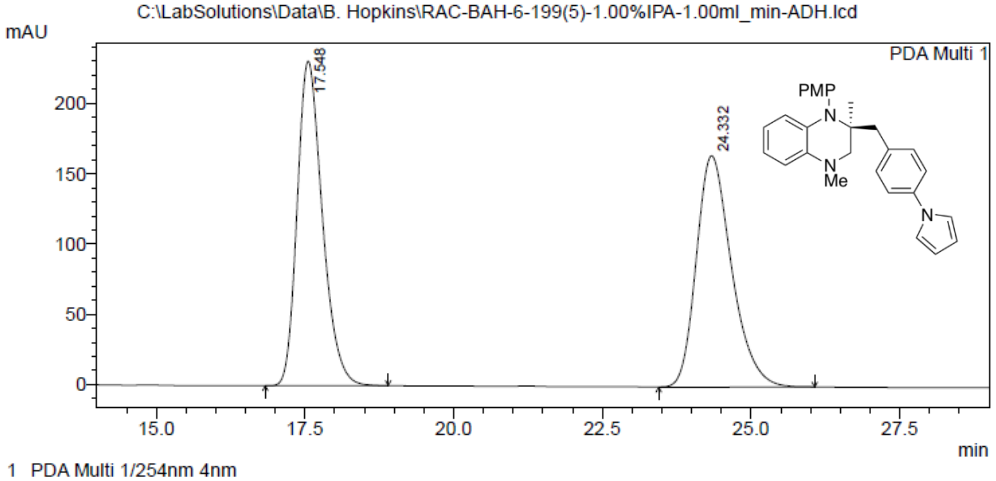
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-199(5)-1.00%IPA-1.00ml_min-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-199(5)-1.00%IPA-1.00ml_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-199(5)-1.00%IPA-1.00ml_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/26/2014 8:46:21 AM
Data Processed	: 2/26/2014 9:36:40 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-199(5)-1.00%IPA-1.00ml_min-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.548	6799607	230777	50.003	58.378
2	24.332	6798670	164539	49.997	41.622
Total		13598278	395316	100.000	100.000

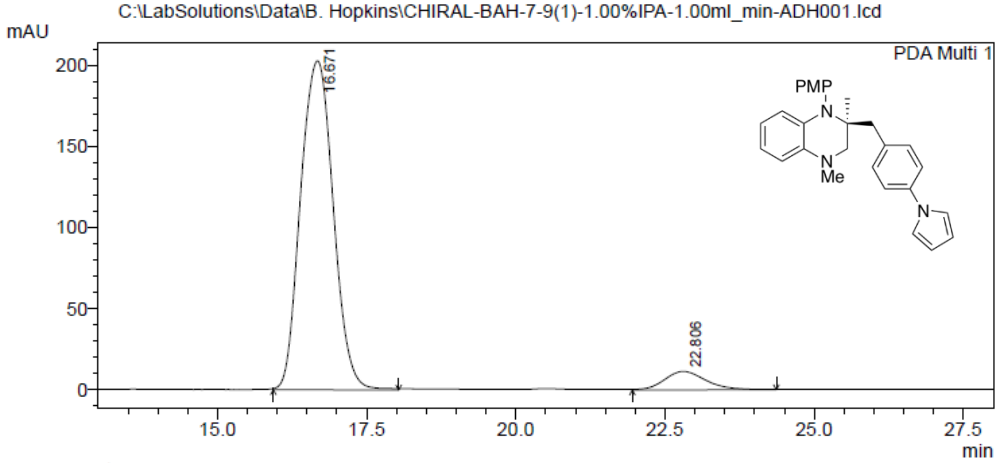
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-7-9(1)-1.00%IPA-1.00ml_min-ADH001.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-7-9(1)-1.00%IPA-1.00ml_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-7-9(1)-1.00%IPA-1.00ml_min-ADH001.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/25/2014 5:00:56 PM
Data Processed	: 2/25/2014 5:38:37 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-7-9(1)-1.00%IPA-1.00ml_min-ADH001.lcd

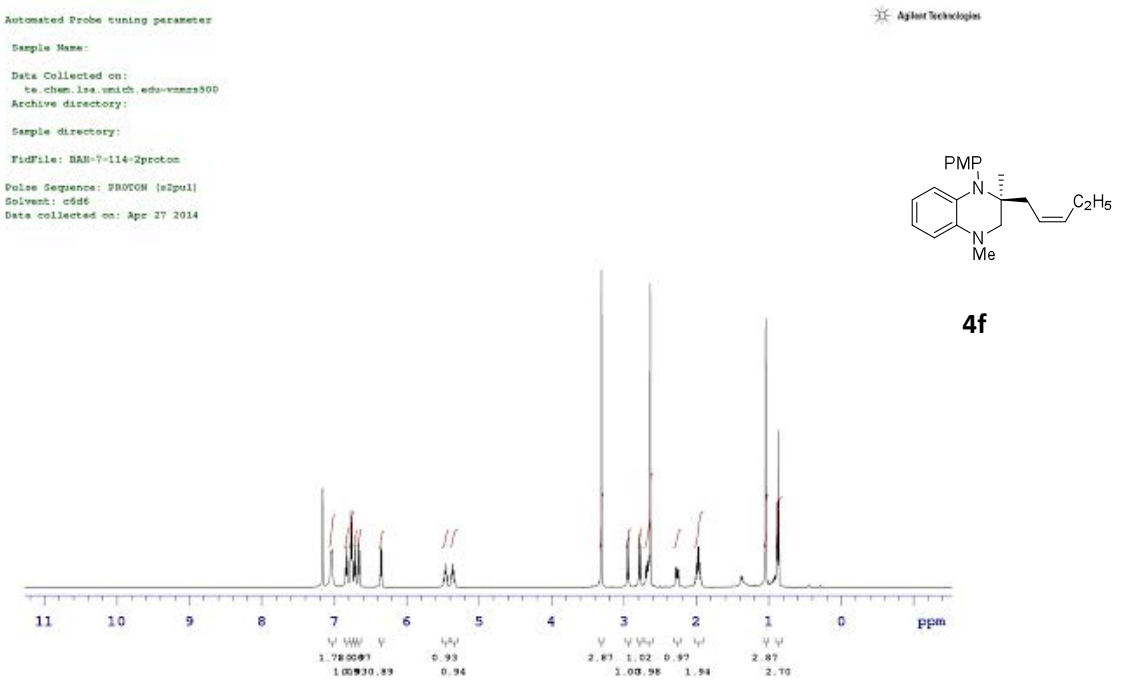


1 PDA Multi 1/254nm 4nm

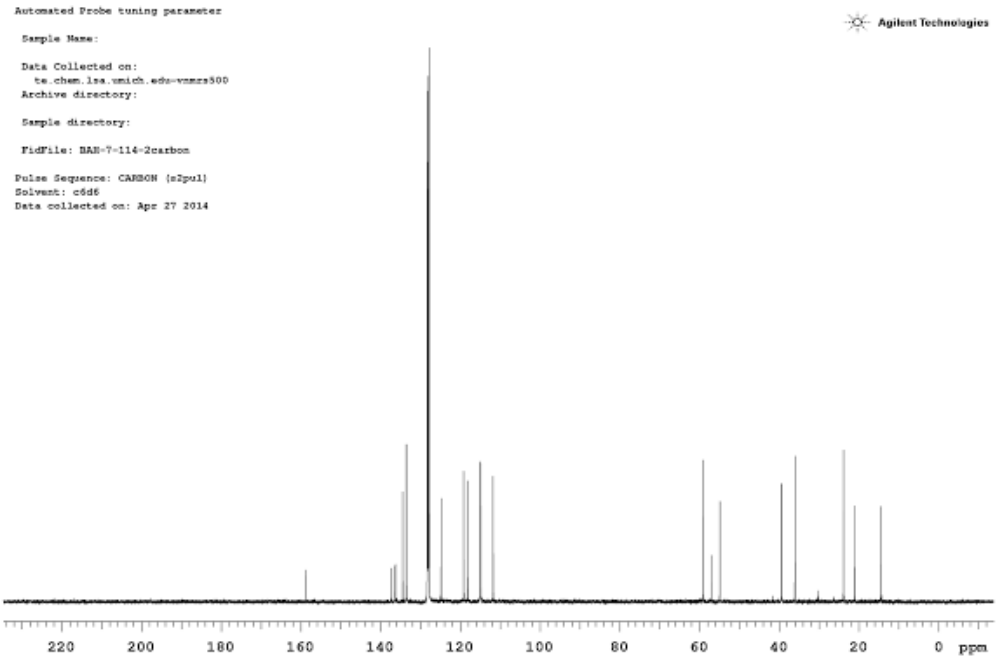
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.671	7768744	202897	93.229	94.658
2	22.806	564197	11451	6.771	5.342
Total		8332942	214348	100.000	100.000


Automated Probe tuning parameter
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-7-114-2proton
Pulse Sequence: PROTON (s2pul)
Solvent: c6d6
Data collected on: Apr 27 2014
Agilent Technologies
PMP
N
Me
C_2H_5
11
10
9
8
7
6
5
4
3
2
1
0
ppm

4f


Automated Probe tuning parameter
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-7-114-2carbon
Pulse Sequence: CARBON (s2pul)
Solvent: c6d6
Data collected on: Apr 27 2014
Agilent Technologies
220
200
180
160
140
120
100
80
60
40
20
0
ppm

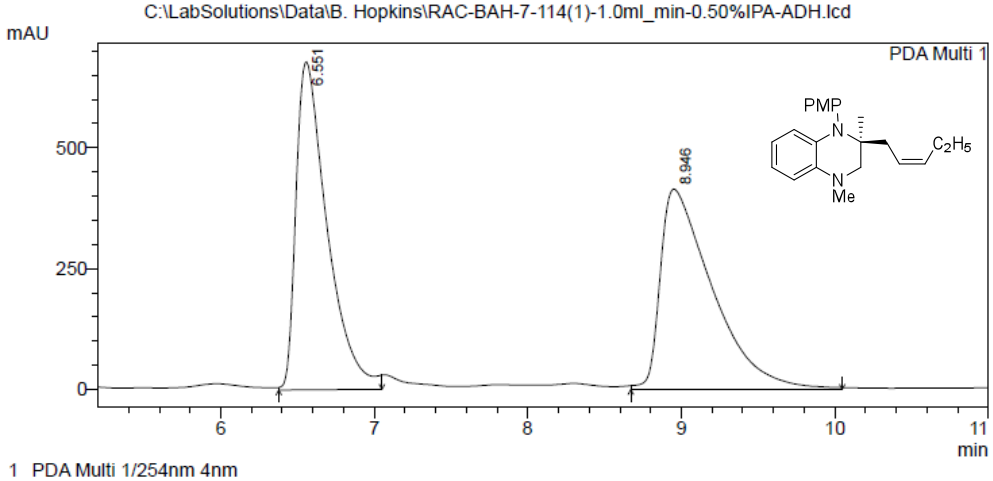
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-7-114(1)-1.0ml_min-0.50%IPA-ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-7-114(1)-1.0ml_min-0.50%IPA-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-7-114(1)-1.0ml_min-0.50%IPA-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/26/2014 11:26:56 AM
Data Processed	: 4/26/2014 11:39:57 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-7-114(1)-1.0ml_min-0.50%IPA-ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.551	9600121	678195	49.520	62.083
2	8.946	9786327	414212	50.480	37.917
Total		19386448	1092407	100.000	100.000

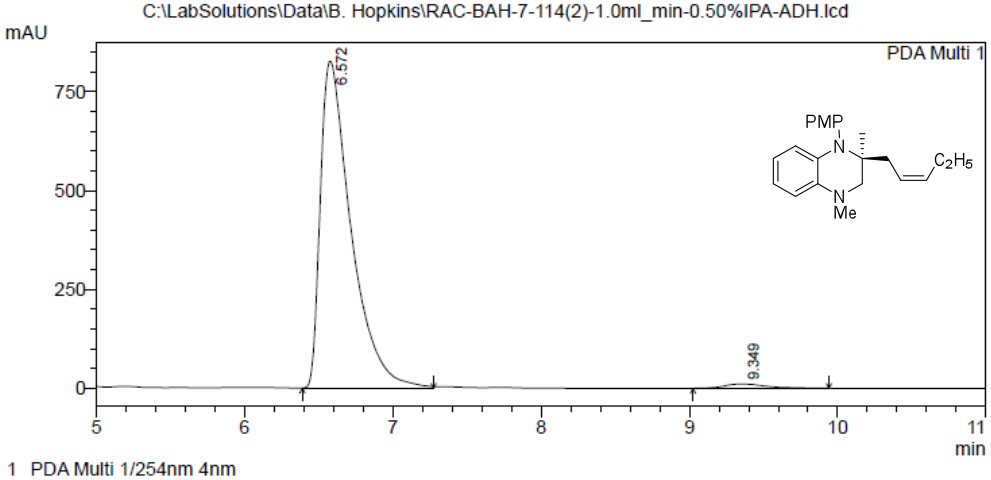
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-7-114(2)-1.0ml_min-0.50%IPA-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-7-114(2)-1.0ml_min-0.50%IPA-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-7-114(2)-1.0ml_min-0.50%IPA-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/26/2014 11:10:53 AM
Data Processed	: 4/26/2014 11:24:11 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-7-114(2)-1.0ml_min-0.50%IPA-ADH.lcd

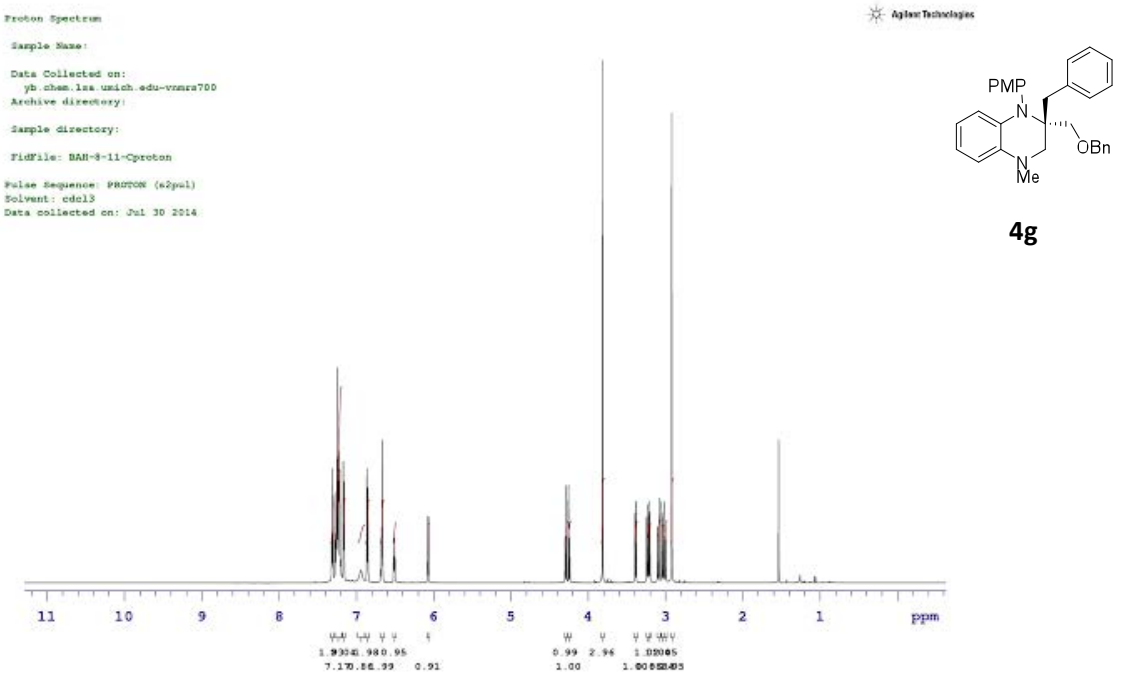


1 PDA Multi 1/254nm 4nm

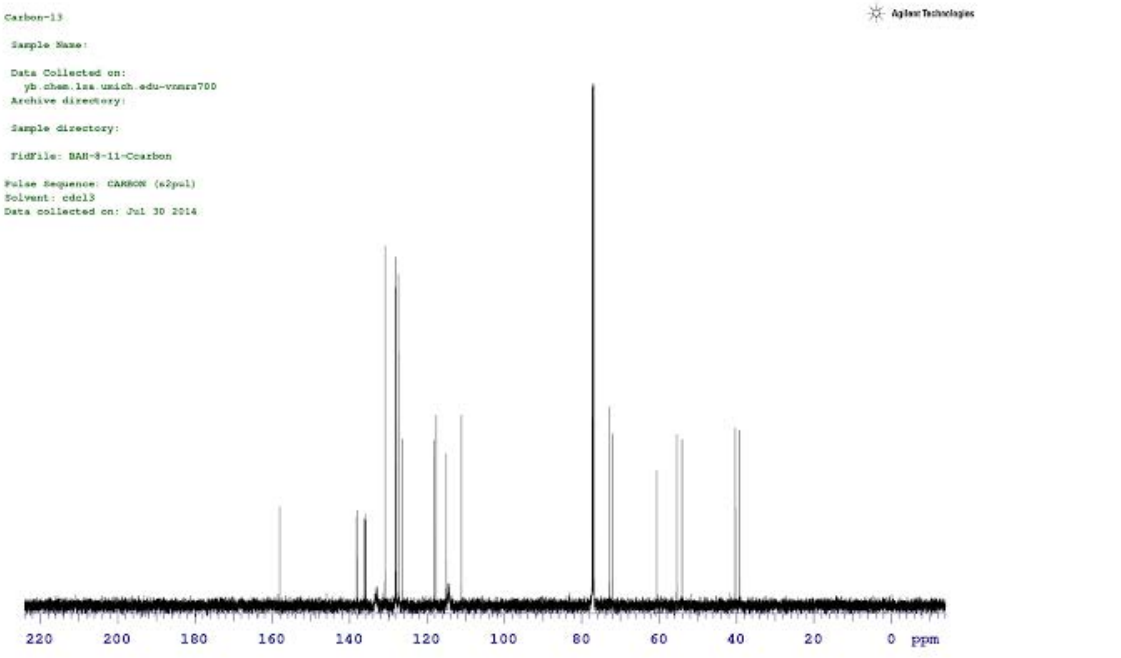
PeakTable

PDA Ch1 254nm 4nm

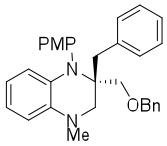
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.572	11920942	826909	98.073	98.629
2	9.349	234186	11490	1.927	1.371
Total		12155128	838399	100.000	100.000


Proton Spectrum
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-8-11-Cproton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 30 2014
Agilent Technologies
PMP
N
OBn
N
Me
11
10
9
8
7
6
5
4
3
2
1
ppm

4g


Carbon-13
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAH-8-11-Ccarbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jul 30 2014
Agilent Technologies
220
200
180
160
140
120
100
80
60
40
20
0
ppm

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



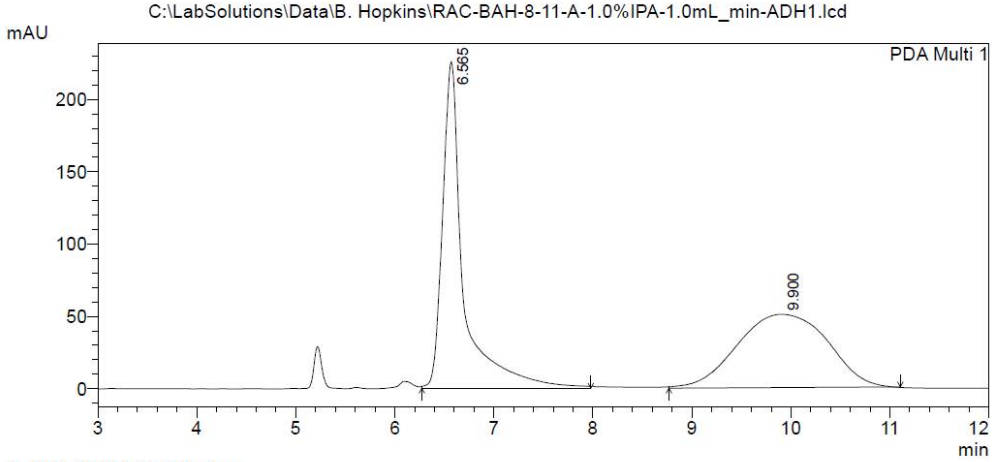
4g

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-8-11-A-1.0%IPA-1.0mL_min-ADH1.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-8-11-A-1.0%IPA-1.0mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-8-11-A-1.0%IPA-1.0mL_min-ADH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/29/2014 10:55:19 AM
Data Processed	: 7/29/2014 11:09:34 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-8-11-A-1.0%IPA-1.0mL_min-ADH1.lcd



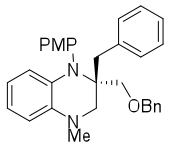
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.565	3322601	225971	50.721	81.685
2	9.900	3228150	50666	49.279	18.315
Total		6550750	276637	100.000	100.000

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



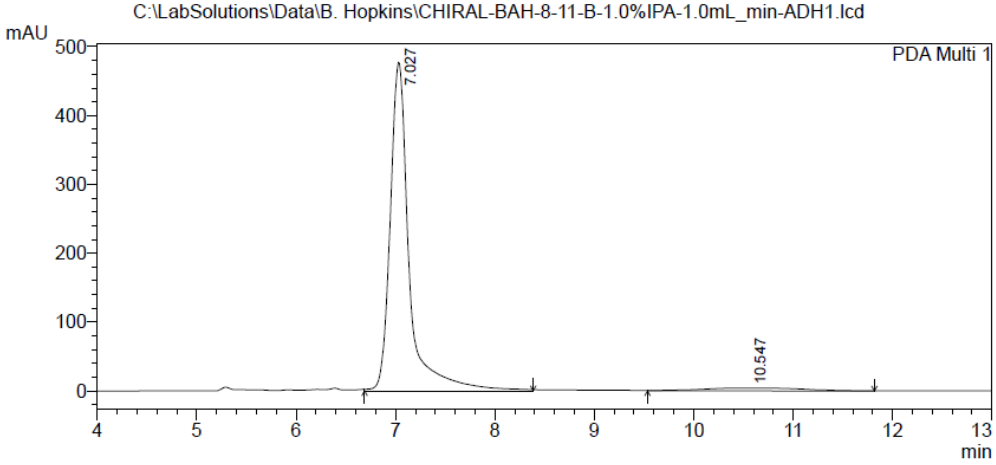
4g

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-8-11-B-1.0%IPA-1.0mL_min-ADH1.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-8-11-B-1.0%IPA-1.0mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-8-11-B-1.0%IPA-1.0mL_min-ADH1.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 7/29/2014 10:38:50 AM
Data Processed	: 7/29/2014 10:54:18 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-8-11-B-1.0%IPA-1.0mL_min-ADH1.lcd

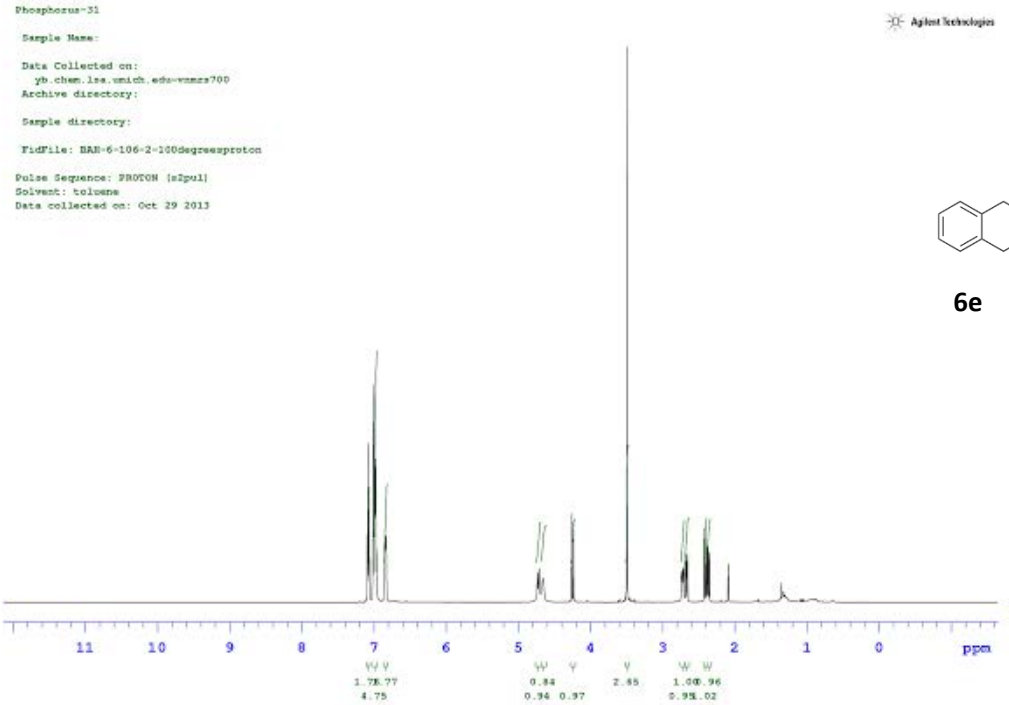


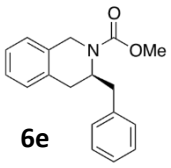
1 PDA Multi 1/254nm 4nm

PeakTable

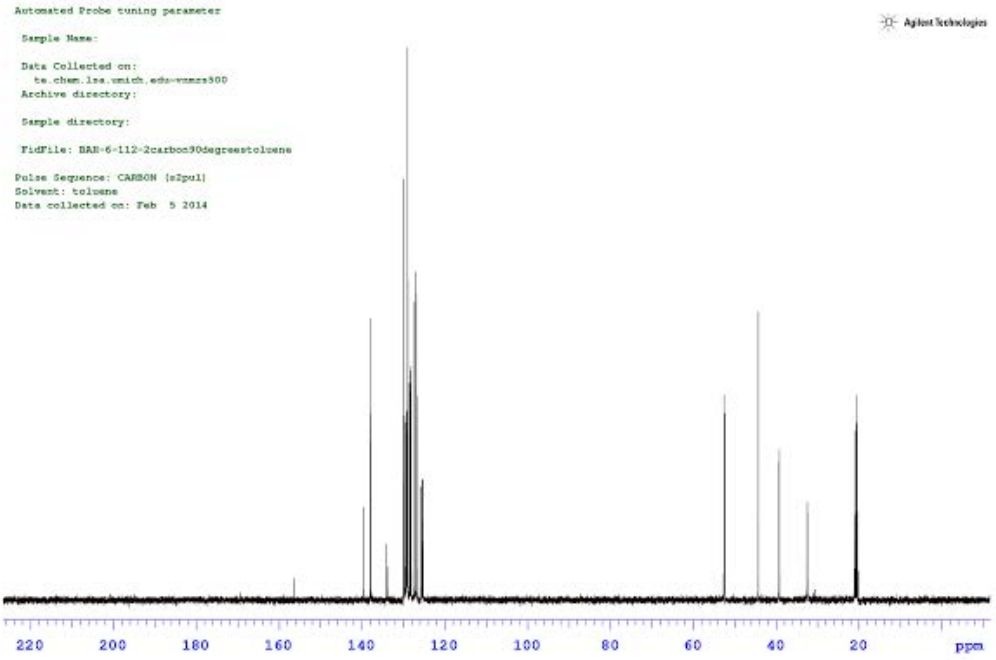
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.027	6365167	477393	95.603	99.146
2	10.547	292782	4111	4.397	0.854
Total		6657949	481503	100.000	100.000


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-6-106-2-100degreesproton
Pulse Sequence: PROTON (s2pul)
Solvent: toluene
Data collected on: Oct 29 2013
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.78 1.77
4.75
0.84
0.94 0.97
2.65
1.00 0.96
0.95 1.02


O
N
OMe

6e


Automated Probe tuning parameter
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAR-6-112-2carbon90degreestoluene
Pulse Sequence: CARBON (s2pul)
Solvent: toluene
Data collected on: Feb 5 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 ppm

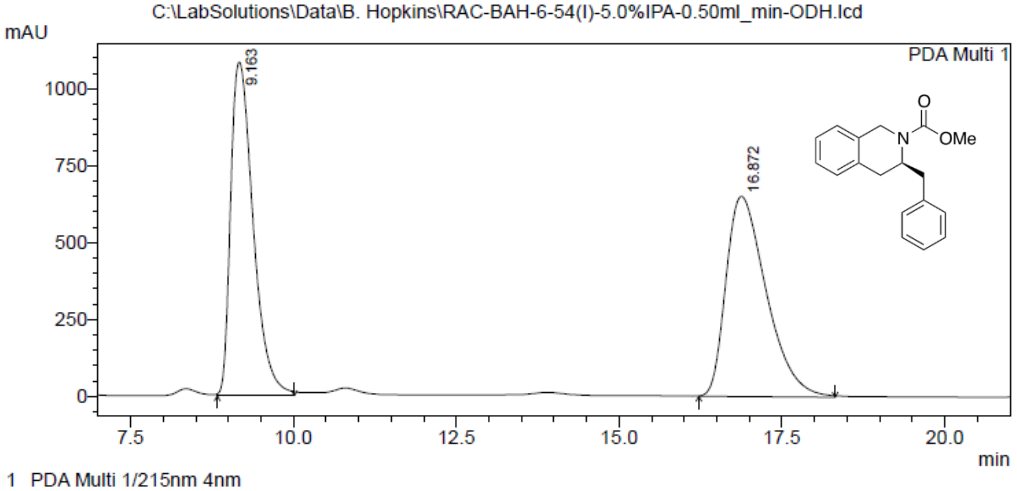
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-54(I)-5.0%IPA-0.50ml_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-54(I)-5.0%IPA-0.50ml_min-ODH
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-54(I)-5.0%IPA-0.50ml_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/16/2013 4:31:42 PM
Data Processed	: 9/16/2013 4:55:39 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-54(I)-5.0%IPA-0.50ml_min-ODH.lcd



1 PDA Multi 1/215nm 4nm

PeakTable

PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.163	26123679	1085130	48.133	62.487
2	16.872	28149981	651453	51.867	37.513
Total		54273660	1736583	100.000	100.000

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

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-6-106(2)-5.0%IPA-0.50mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-6-106(2)-5.0%IPA-0.50mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-6-106(2)-5.0%IPA-0.50mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method-JB.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/29/2013 12:10:26 PM
Data Processed	: 10/29/2013 12:38:41 PM

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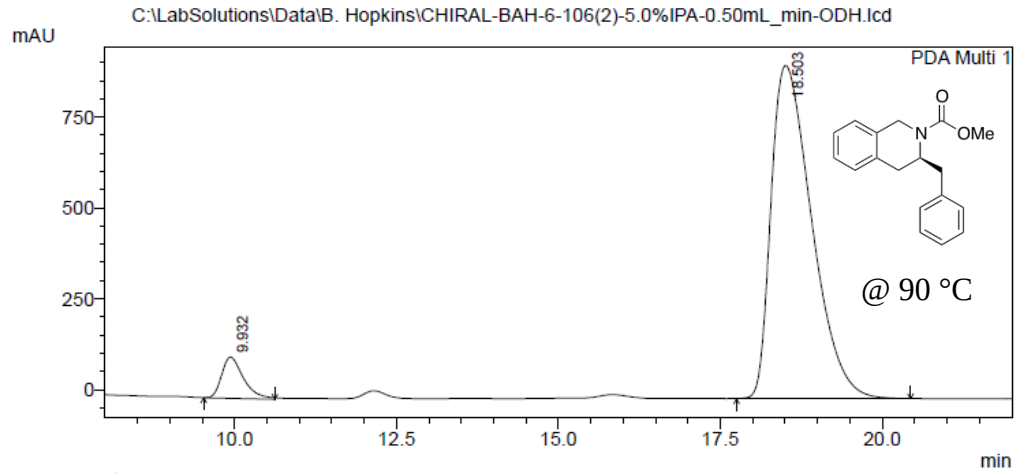


1 PDA Multi 1/215nm 4nm

PeakTable

PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.932	2666869	113604	6.239	11.025
2	18.503	40080519	916776	93.761	88.975
Total		42747387	1030380	100.000	100.000

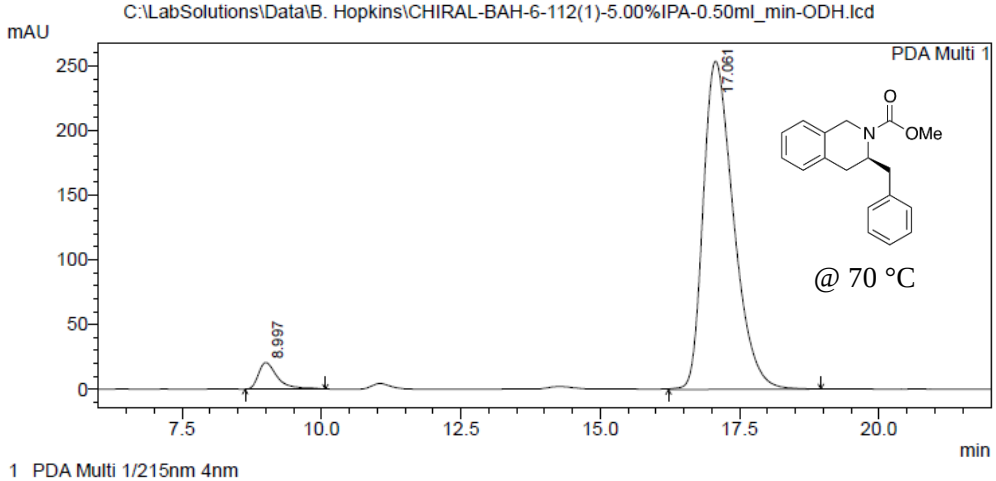
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-6-112(1)-5.00%IPA-0.50ml_min-ODH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-6-112(1)-5.00%IPA-0.50ml_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-6-112(1)-5.00%IPA-0.50ml_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/25/2014 3:22:28 PM
Data Processed	: 2/25/2014 4:09:19 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-6-112(1)-5.00%IPA-0.50ml_min-ODH.lcd

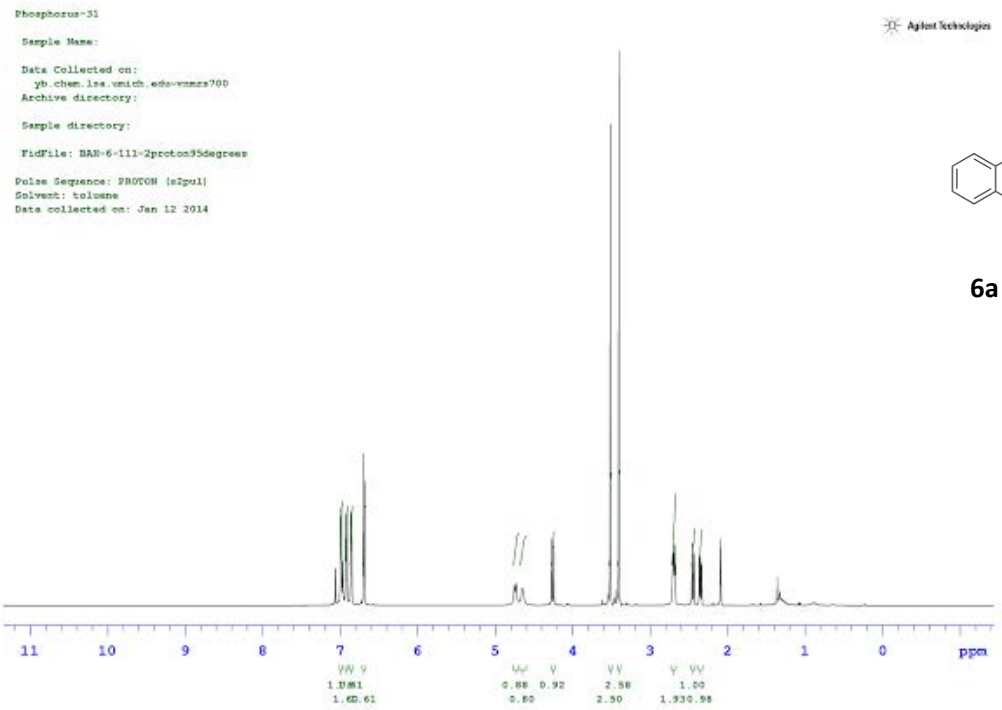


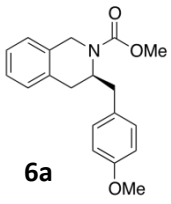
1 PDA Multi 1/215nm 4nm

PeakTable

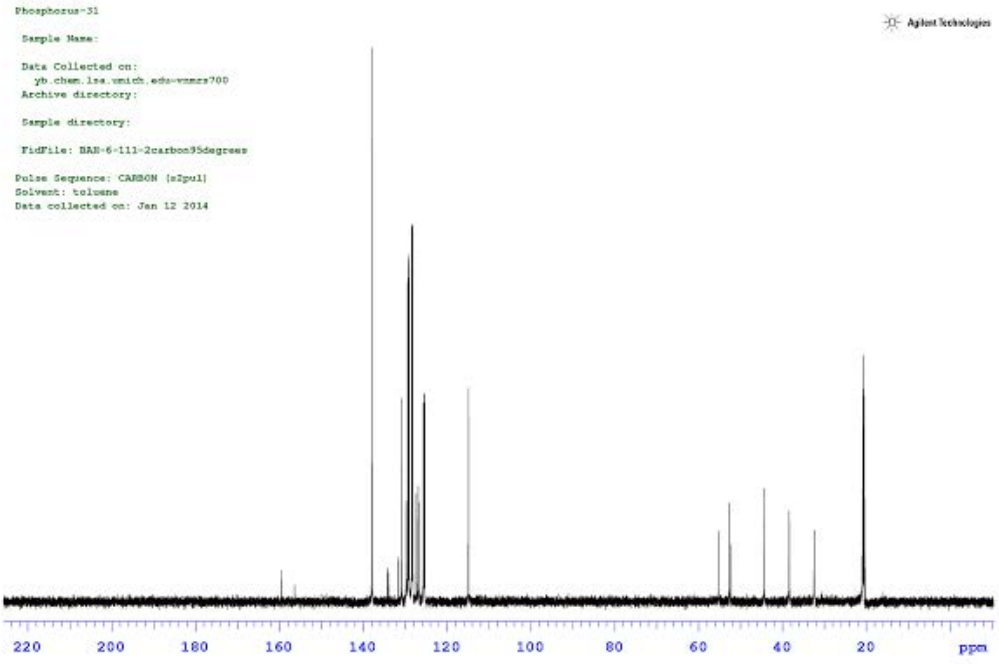
PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.997	497491	20651	4.916	7.524
2	17.061	9621378	253810	95.084	92.476
Total		10118869	274461	100.000	100.000


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-111-2proton95degrees
Pulse Sequence: PROTON (s2pul)
Solvent: toluene
Data collected on: Jan 12 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00 1.01 0.88 0.92 2.58 1.00
1.60 0.61 0.80 2.50 1.93 0.98


O
N
OMe
OMe

6a


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-111-2carbon95degrees
Pulse Sequence: CARBON (s2pul)
Solvent: toluene
Data collected on: Jan 12 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 ppm

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

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-103(I)-5.0%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-103(I)-5.0%IPA-1.0mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-103(I)-5.0%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method-JB.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/21/2013 2:55:46 PM
Data Processed	: 10/21/2013 3:24:12 PM

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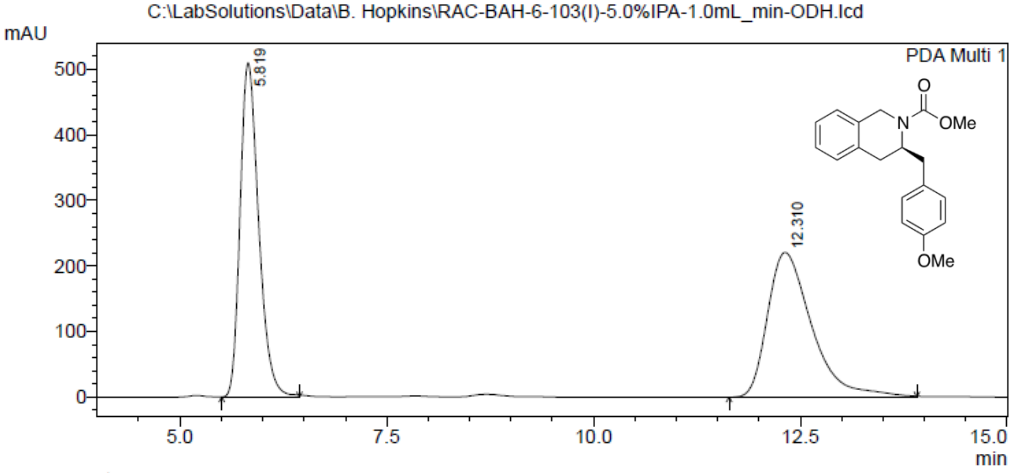
C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-103(I)-5.0%IPA-1.0mL_min-ODH.lcd



1 PDA Multi 1/215nm 4nm

PeakTable

PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.819	7978952	510028	49.290	69.818
2	12.310	8208736	220478	50.710	30.182
Total		16187689	730506	100.000	100.000

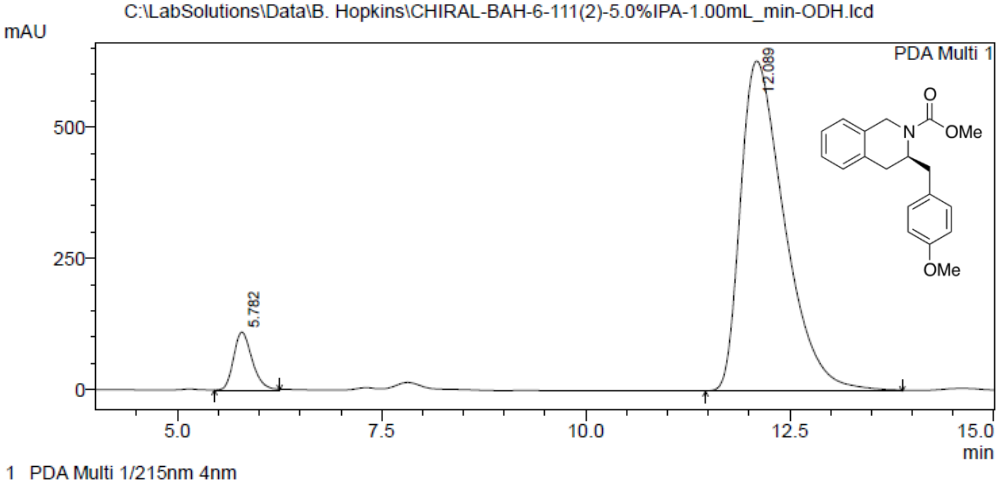
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-6-111(2)-5.0%IPA-1.00mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-6-111(2)-5.0%IPA-1.00mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-6-111(2)-5.0%IPA-1.00mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/1/2013 3:27:51 PM
Data Processed	: 11/1/2013 3:49:14 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-6-111(2)-5.0%IPA-1.00mL_min-ODH.lcd

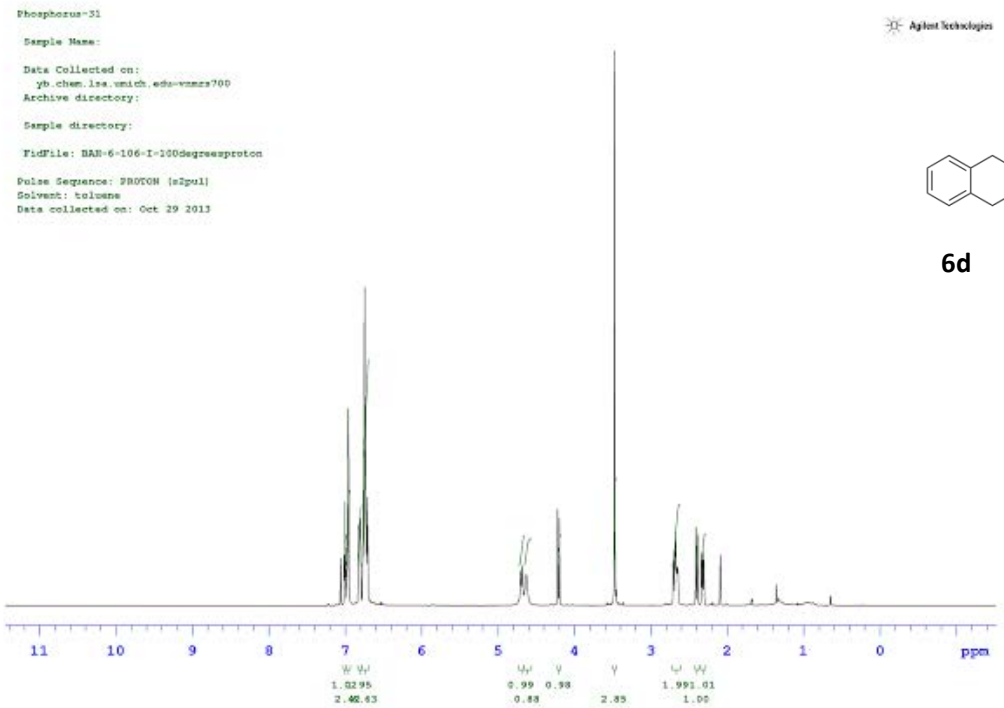


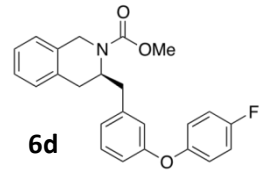
1 PDA Multi 1/215nm 4nm

PeakTable

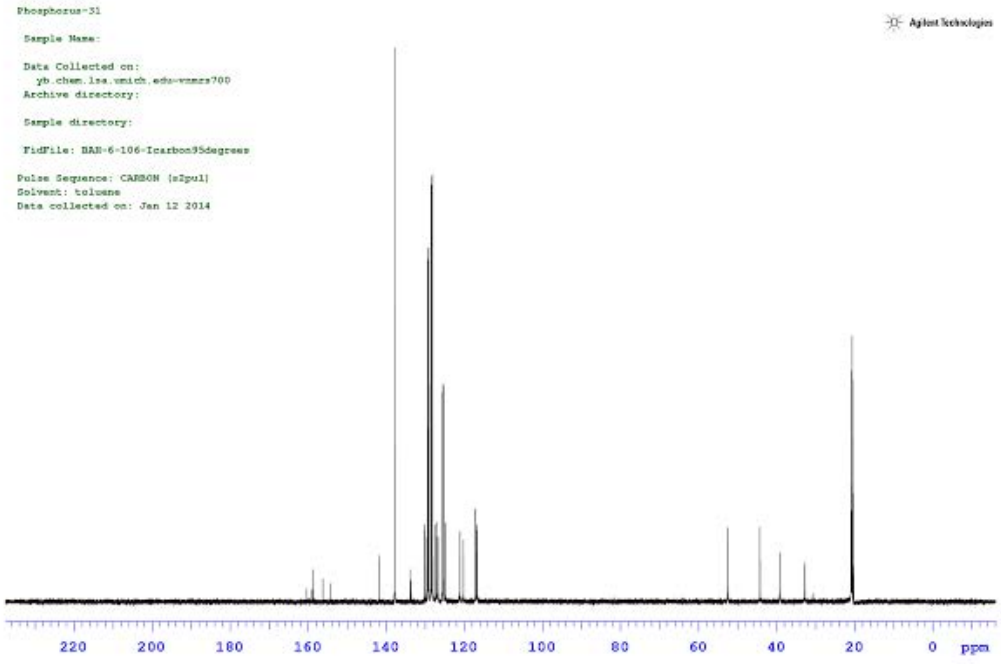
PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.782	1749007	110207	6.882	14.916
2	12.089	23666905	628651	93.118	85.084
Total		25415912	738858	100.000	100.000


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-106-I-100degreesproton
Pulse Sequence: PROTON (s2pul)
Solvent: toluene
Data collected on: Oct 29 2013
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.0295
2.40.63
0.99 0.98
0.88
2.85
1.991.01
1.00


O
N
OMe
F
O

6d


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-106-Icarbon95degrees
Pulse Sequence: CARBON (s2pul)
Solvent: toluene
Data collected on: Jan 12 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm

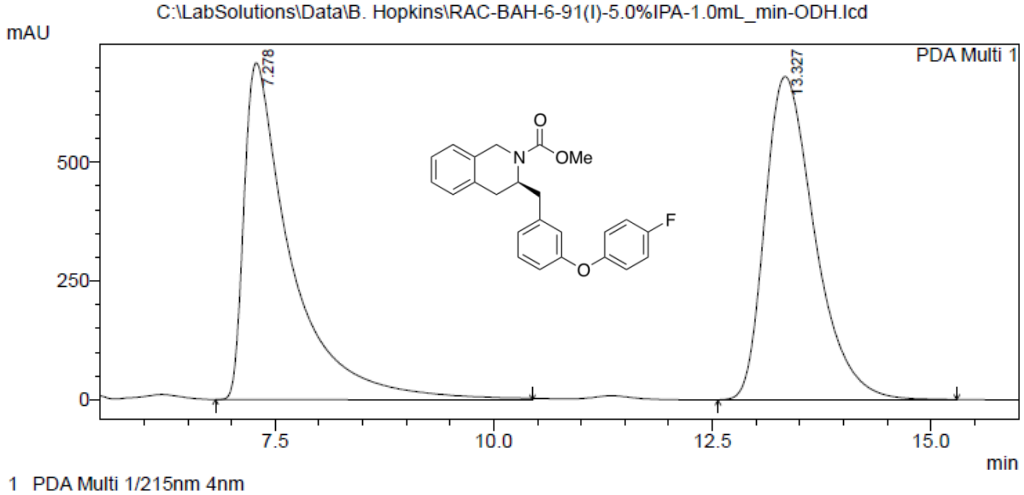
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-91(I)-5.0%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-91(I)-5.0%IPA-1.0mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-91(I)-5.0%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method-JB.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/15/2013 1:33:22 PM
Data Processed	: 10/15/2013 1:55:19 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-91(I)-5.0%IPA-1.0mL_min-ODH.lcd



1 PDA Multi 1/215nm 4nm

PeakTable

PDA Ch1 215nm 4nm

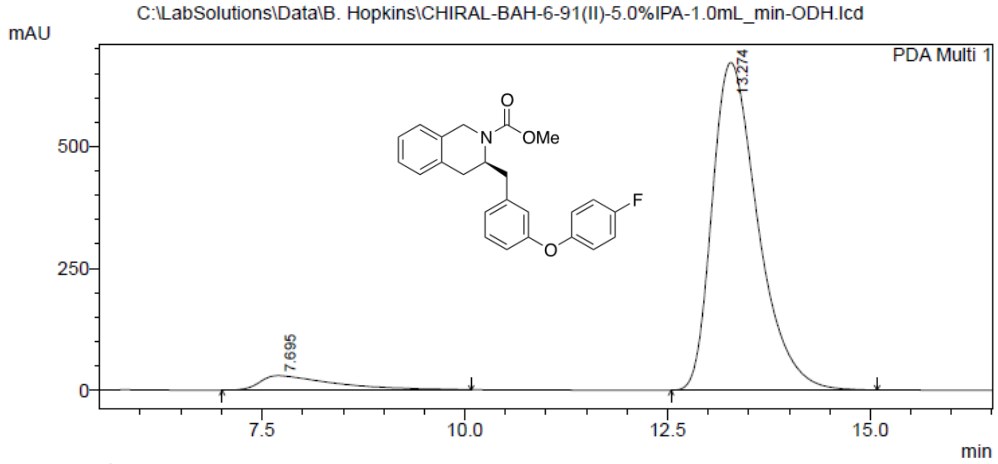
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.278	26660609	708823	49.573	50.999
2	13.327	27119942	681049	50.427	49.001
Total		53780551	1389873	100.000	100.000

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

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-6-91(II)-5.0%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-6-91(II)-5.0%IPA-1.0mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-6-91(II)-5.0%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method-JB.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/15/2013 1:57:19 PM
Data Processed	: 10/15/2013 2:27:52 PM

<Chromatogram>

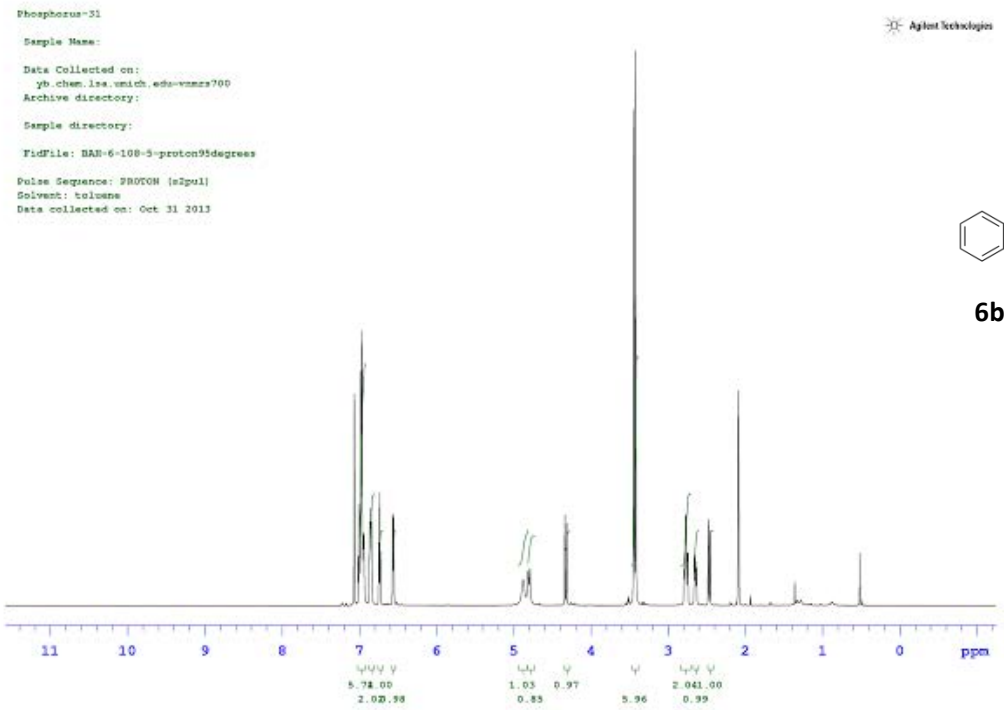


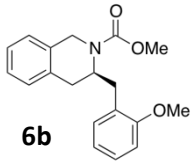
1 PDA Multi 1/215nm 4nm

PeakTable

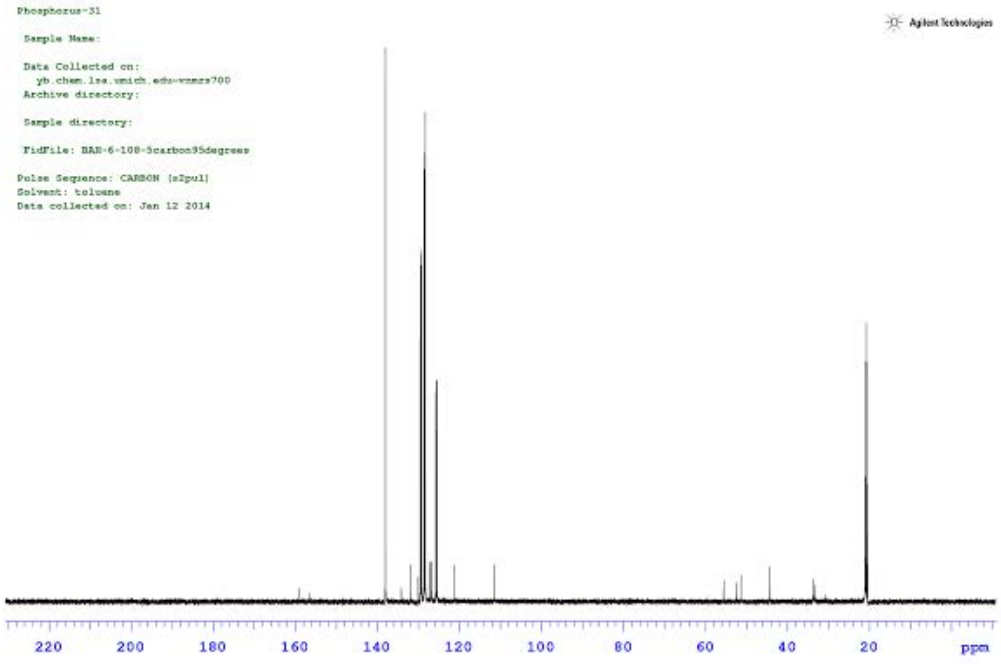
PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.695	1958218	29914	6.888	4.258
2	13.274	26470186	672640	93.112	95.742
Total		28428404	702555	100.000	100.000


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-108-5-proton95degrees
Pulse Sequence: PROTON (s2pul)
Solvent: toluene
Data collected on: Oct 31 2013
Agilent Technologies
11
10
9
8
7
6
5
4
3
2
1
0
ppm
5.71
8.00
2.02
0.98
1.03
0.97
0.85
5.96
2.041.00
0.99


O
N
OMe
OMe

6b


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-108-5carbon95degrees
Pulse Sequence: CARBON (s2pul)
Solvent: toluene
Data collected on: Jan 12 2014
Agilent Technologies
220
200
180
160
140
120
100
80
60
40
20
ppm

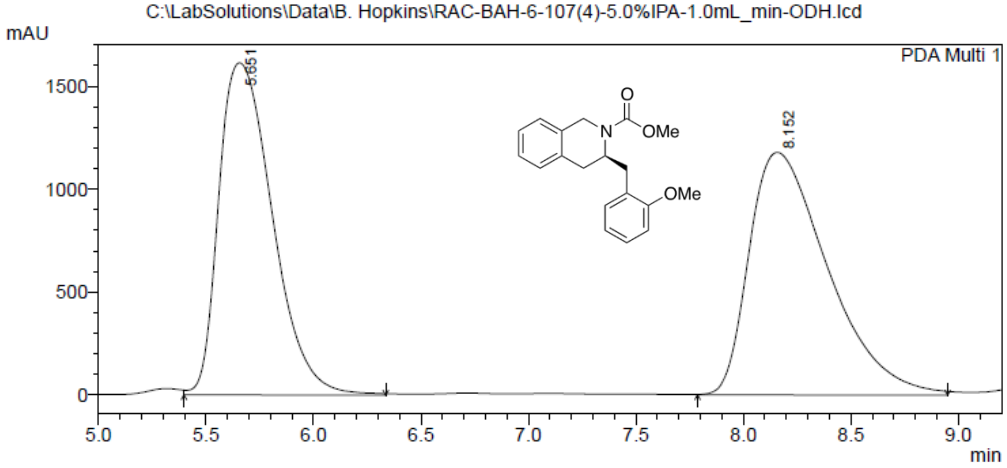
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-107(4)-5.0%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-107(4)-5.0%IPA-1.0mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-107(4)-5.0%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method-JB.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/26/2013 2:44:32 PM
Data Processed	: 10/26/2013 3:06:03 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-107(4)-5.0%IPA-1.0mL_min-ODH.lcd



1 PDA Multi 1/215nm 4nm

PeakTable

PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.651	28040911	1613903	48.263	57.777
2	8.152	30059849	1179440	51.737	42.223
Total		58100760	2793344	100.000	100.000

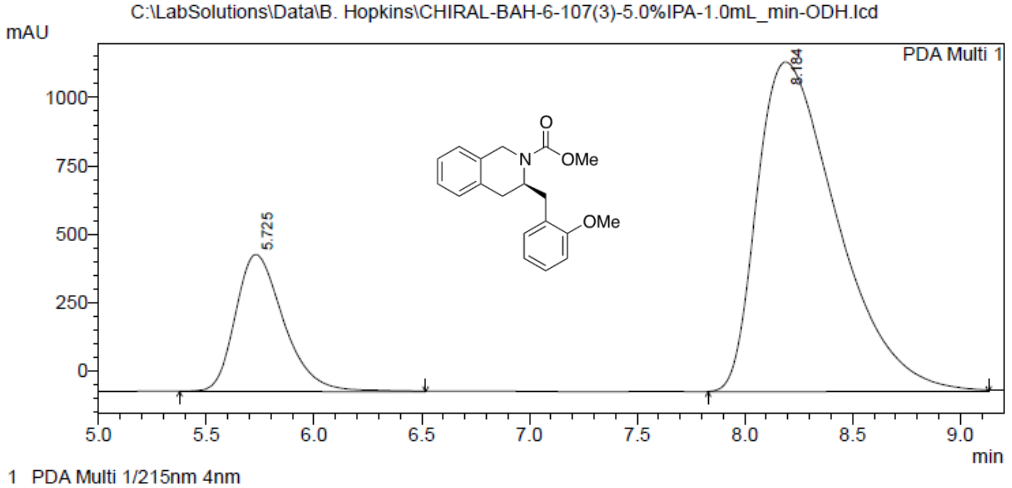
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-6-107(3)-5.0%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-6-107(3)-5.0%IPA-1.0mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-6-107(3)-5.0%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method-JB.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/28/2013 2:22:07 PM
Data Processed	: 10/28/2013 3:32:09 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-6-107(3)-5.0%IPA-1.0mL_min-ODH.lcd

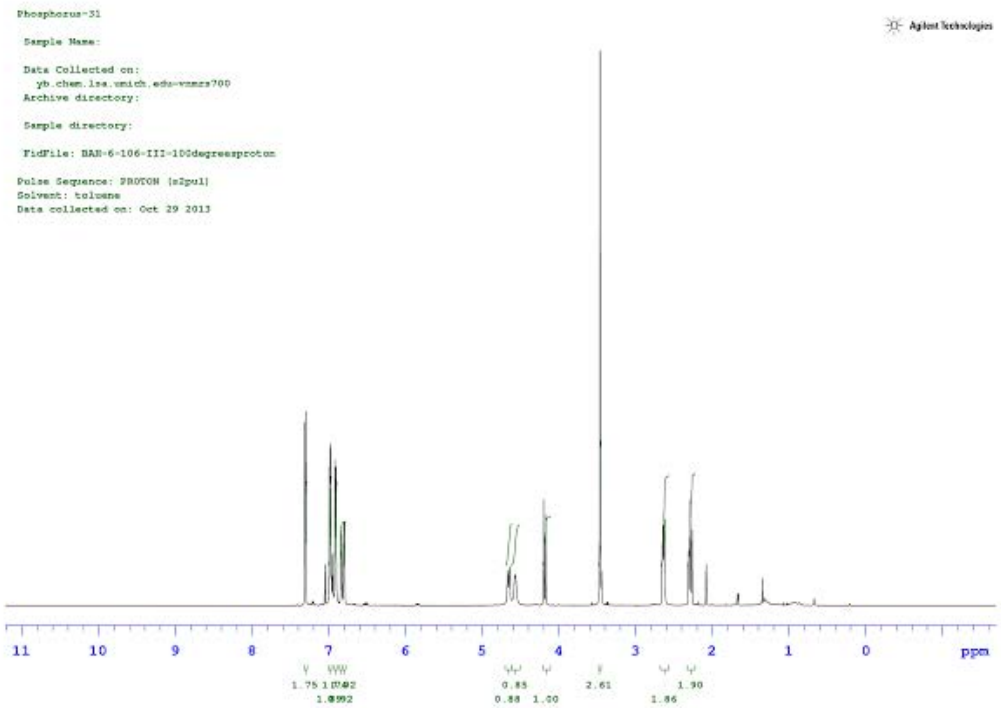


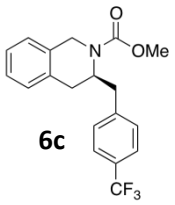
1 PDA Multi 1/215nm 4nm

PeakTable

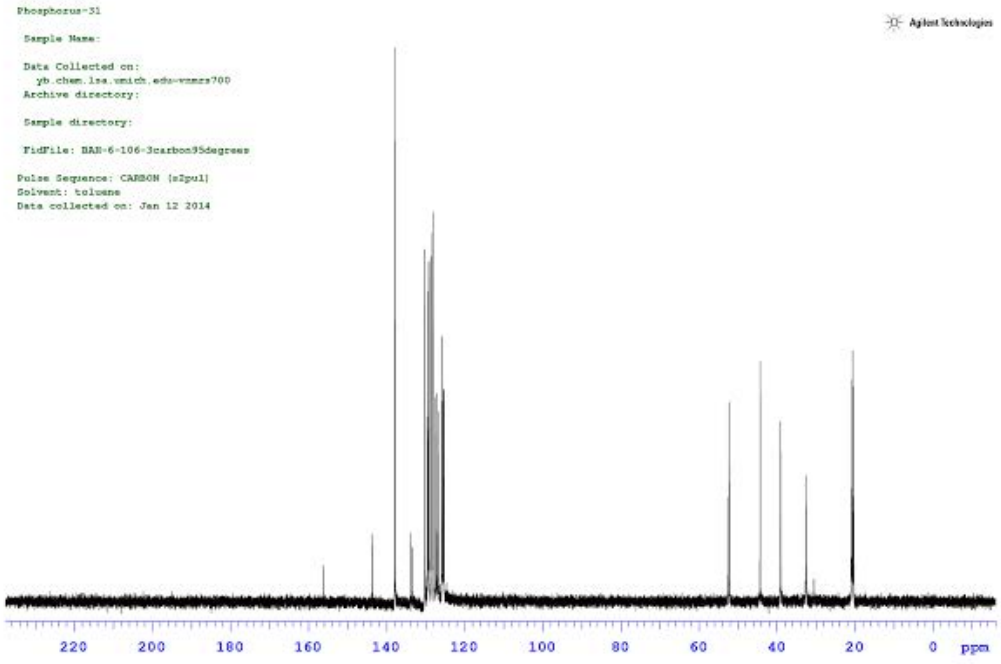
PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.725	7867795	501355	20.097	29.374
2	8.184	31281214	1205431	79.903	70.626
Total		39149009	1706786	100.000	100.000


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-106-III-100degreesproton
Pulse Sequence: PROTON (s2pul)
Solvent: toluene
Data collected on: Oct 29 2013
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.75 1.0792
1.0992
0.85
0.88 1.00
2.61
1.90
1.86


O
N
OMe
CF3

6c

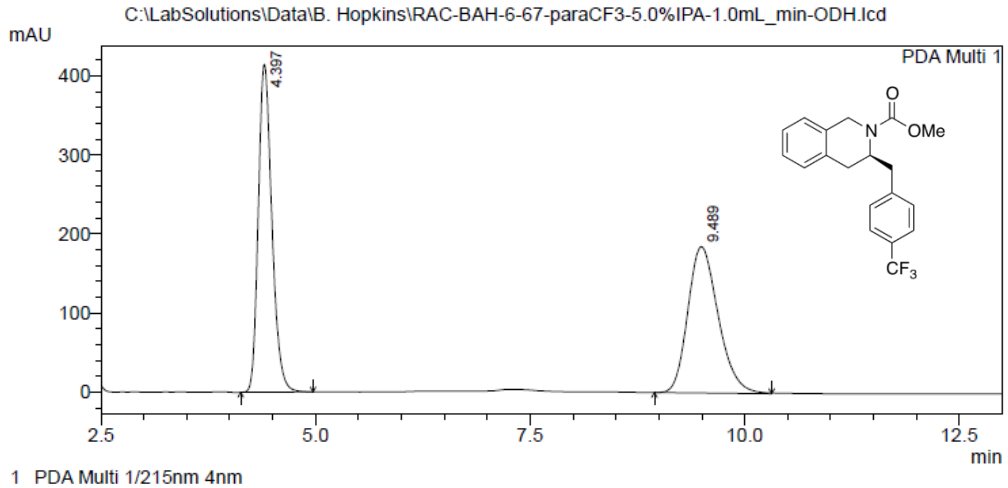

Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-106-3carbon95degrees
Pulse Sequence: CARBON (s2pul)
Solvent: toluene
Data collected on: Jan 12 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm

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

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-67-paraCF3-5.0%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-67-paraCF3-5.0%IPA-1.0mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-67-paraCF3-5.0%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method-JB.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/25/2013 11:21:59 AM
Data Processed	: 10/25/2013 12:31:16 PM

<Chromatogram>



1 PDA Multi 1/215nm 4nm

PeakTable

PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.397	4573026	414768	50.059	69.145
2	9.489	4562247	185083	49.941	30.855
Total		9135272	599851	100.000	100.000

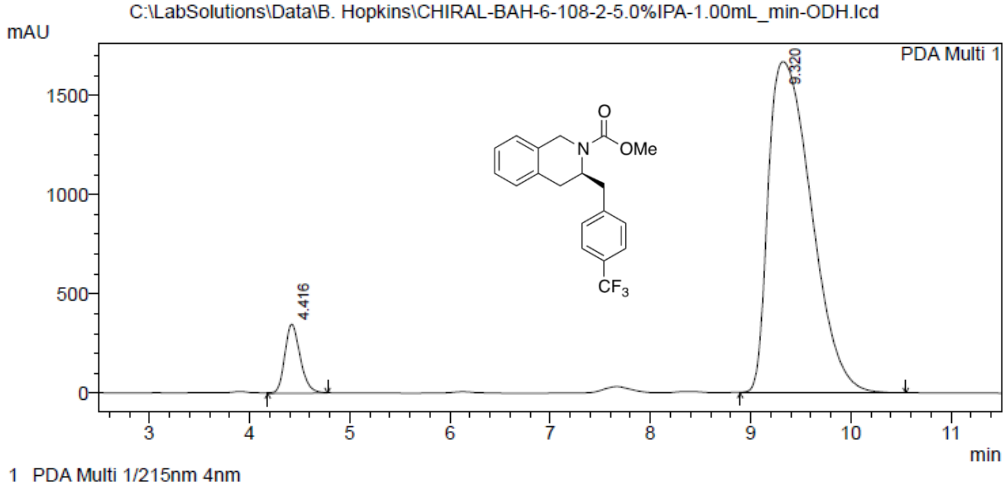
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-6-108-2-5.0%IPA-1.00mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-6-108-2-5.0%IPA-1.00mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-6-108-2-5.0%IPA-1.00mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/30/2013 11:25:48 AM
Data Processed	: 10/30/2013 11:45:19 AM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-6-108-2-5.0%IPA-1.00mL_min-ODH.lcd

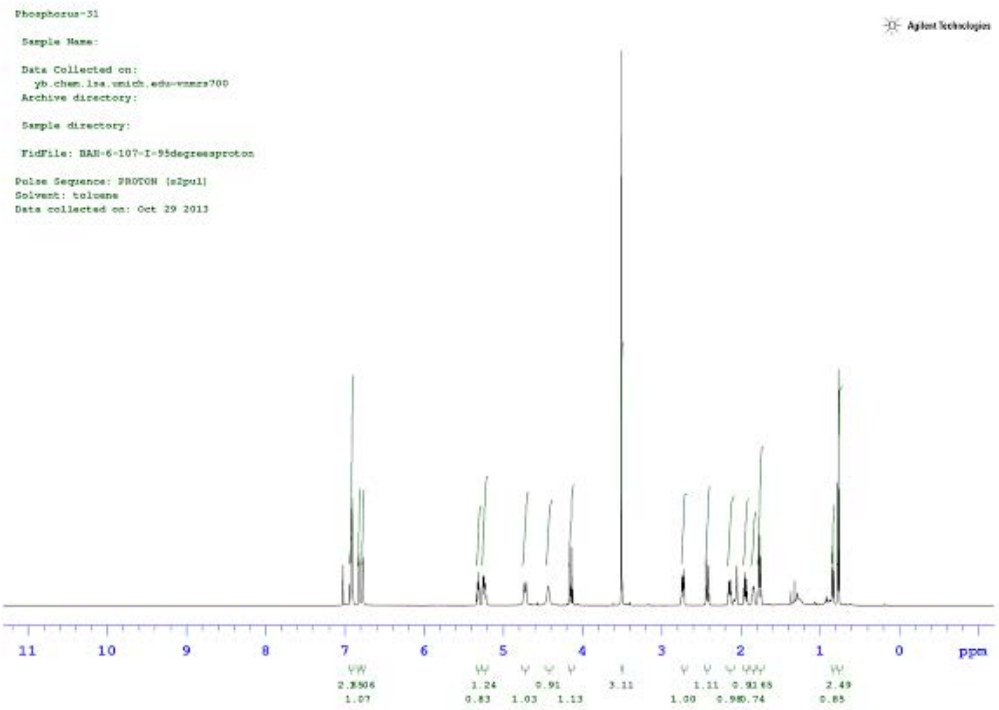


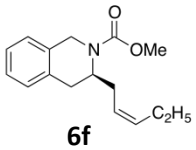
1 PDA Multi 1/215nm 4nm

PeakTable

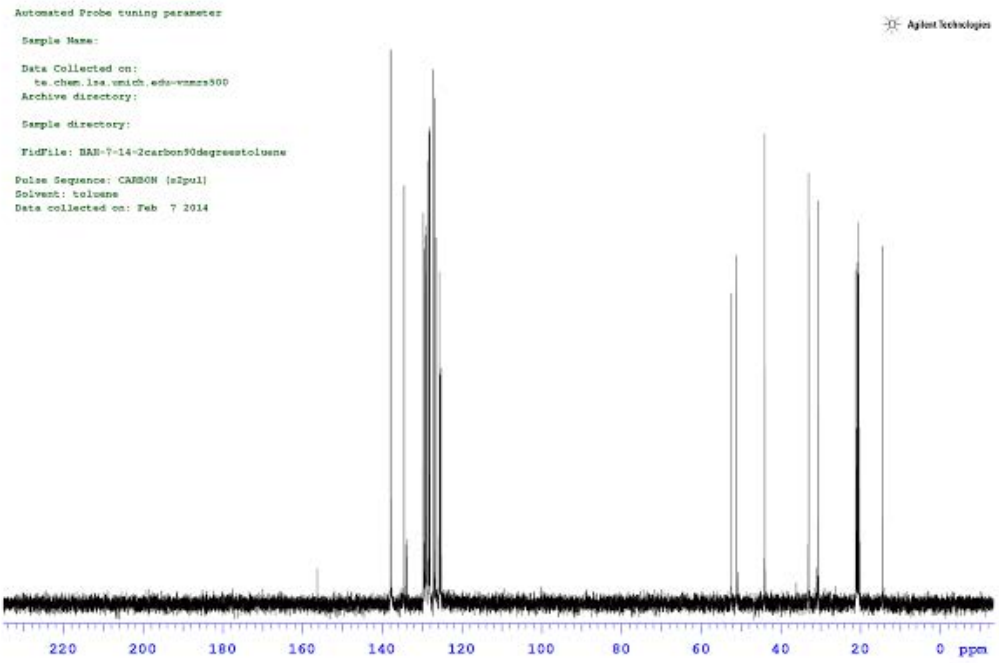
PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.416	3751617	347086	6.982	17.222
2	9.320	49979944	1668275	93.018	82.778
Total		53731561	2015361	100.000	100.000


Phosphorus-31
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAN-6-107-1-95degreesproton
Pulse Sequence: PROTON (s2pul)
Solvent: toluene
Data collected on: Oct 29 2013
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.3506 1.24 0.91 3.11 1.11 0.9165 2.49
1.07 0.83 1.03 1.13 1.00 0.980.74 0.85


O
N
OMe
C2H5

6f


Automated Probe tuning parameter
Sample Name:
Data Collected on:
te.chem.lsa.umich.edu-vnmrs500
Archive directory:
Sample directory:
FidFile: BAN-7-14-2carbon90degreestoluene
Pulse Sequence: CARBON (s2pul)
Solvent: toluene
Data collected on: Feb 7 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm

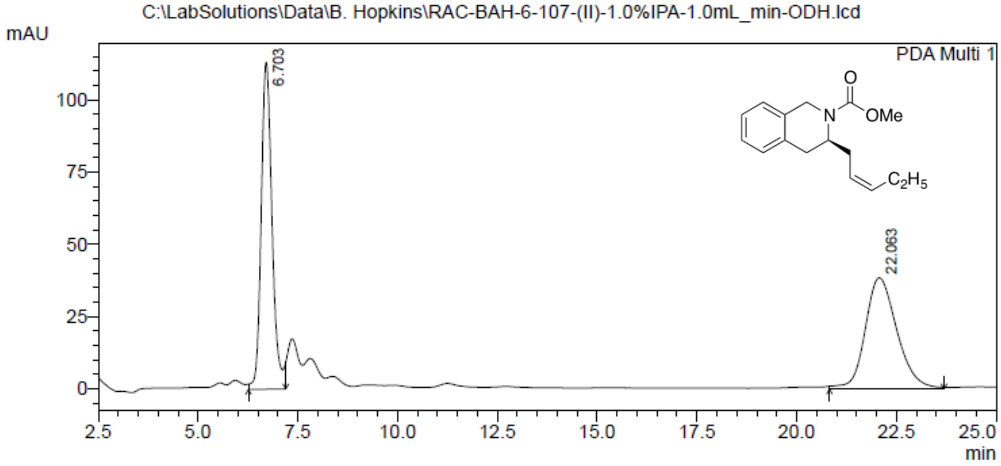
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-107-(II)-1.0%IPA-1.0mL_min-ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-BAH-6-107-(II)-1.0%IPA-1.0mL_min-ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-BAH-6-107-(II)-1.0%IPA-1.0mL_min-ODH.lcd
Method File Name	: Cyclic Urea Method-JB.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/25/2013 2:12:44 PM
Data Processed	: 10/25/2013 3:22:45 PM

<Chromatogram>

C:\LabSolutions\Data\B. Hopkins\RAC-BAH-6-107-(II)-1.0%IPA-1.0mL_min-ODH.lcd



1 PDA Multi 1/215nm 4nm

PeakTable

PDA Ch1 215nm 4nm

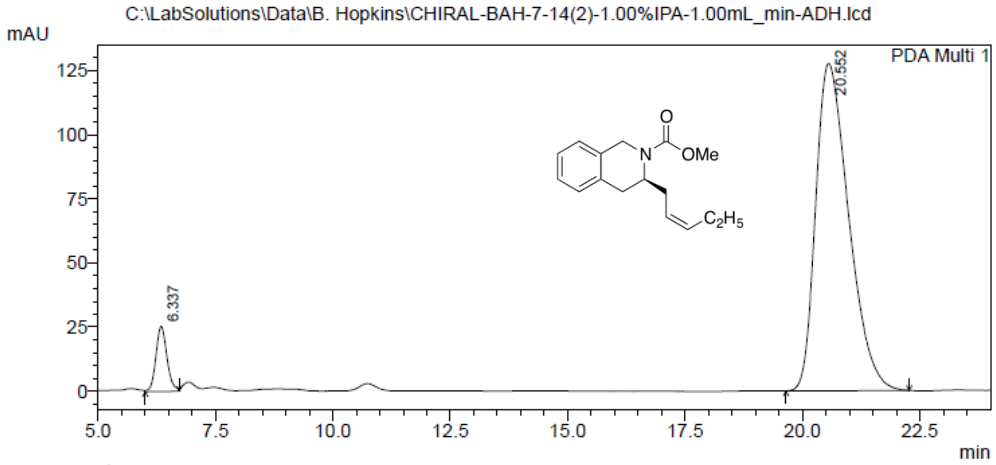
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.703	2147283	113271	50.207	74.646
2	22.063	2129612	38472	49.793	25.354
Total		4276895	151743	100.000	100.000

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

C:\LabSolutions\Data\B. Hopkins\CHIRAL-BAH-7-14(2)-1.00%IPA-1.00mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-BAH-7-14(2)-1.00%IPA-1.00mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-BAH-7-14(2)-1.00%IPA-1.00mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/6/2014 1:31:18 PM
Data Processed	: 2/6/2014 1:58:24 PM

<Chromatogram>

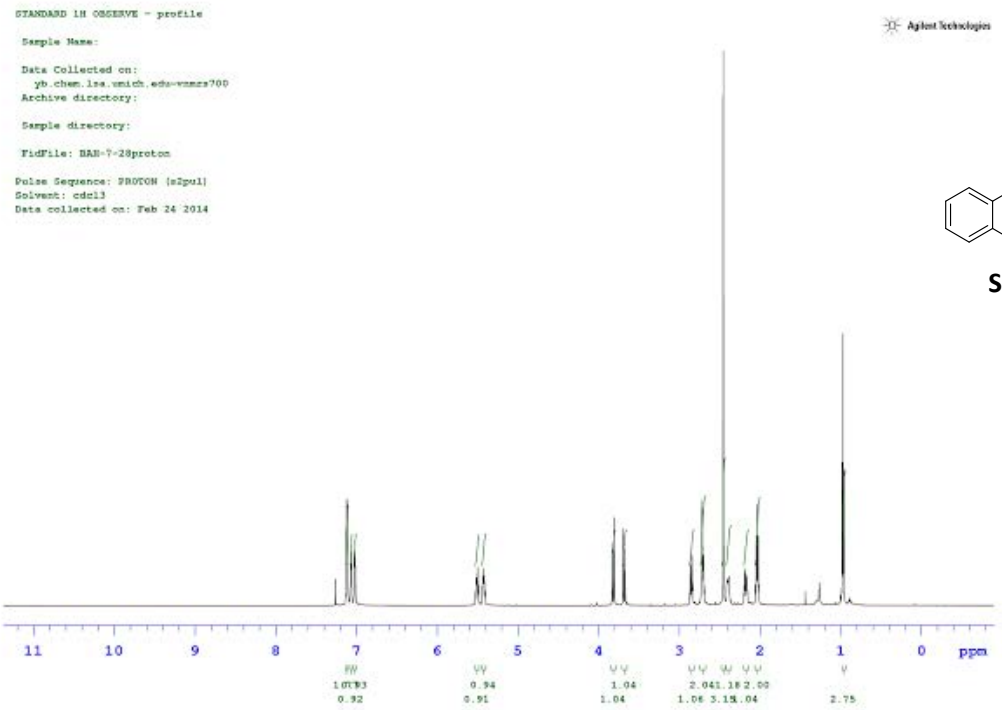


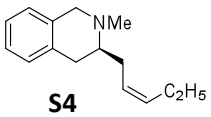
1 PDA Multi 1/215nm 4nm

PeakTable

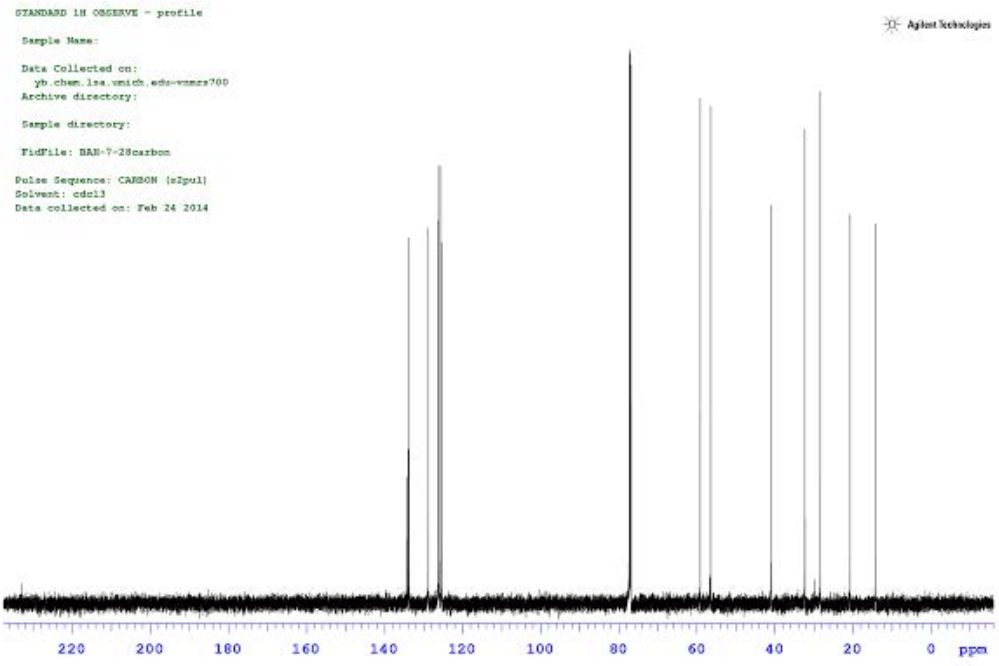
PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.337	420172	25521	6.310	16.654
2	20.552	6238442	127723	93.690	83.346
Total		6658614	153243	100.000	100.000


STANDARD 1H OBSERVE - profile
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-7-28proton
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 24 2014
Agilent Technologies
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00 0.92 0.94 0.91 1.04 1.04 2.04 1.18 2.00 1.06 3.15 1.04 2.75


NMe
C2H5

S4


STANDARD 1H OBSERVE - profile
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: BAR-7-28carbon
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 24 2014
Agilent Technologies
220 200 180 160 140 120 100 80 60 40 20 0 ppm