Asymmetric Pd-Catalyzed Alkene Carboamination Reactions for the Synthesis of 2-Aminoindane Derivatives.

Derick R. White, Johnathon T. Hutt, and John P. Wolfe*

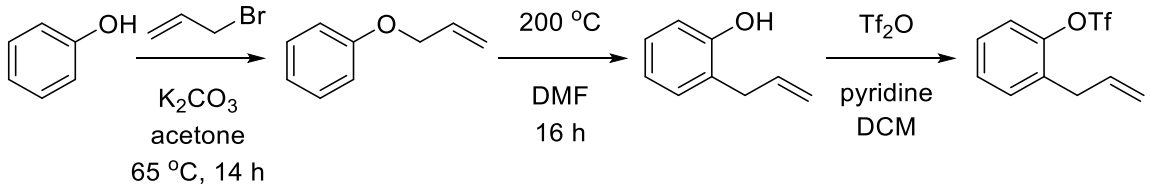
Department of Chemistry, University of Michigan, 920 N. University Avenue, Ann Arbor, Michigan 48109-1055

Supporting Information

General Considerations: All reactions were carried out under a nitrogen atmosphere using oven or flame-dried glassware. All palladium sources and reagents were obtained from commercial sources and used without further purification unless otherwise noted. All amines were distilled from CaH_2 prior to use in reactions. (S)-*t*-Bu-PHOX,[i] 2-allylphenyl trifluoromethanesulfonate (**4a**)[ii], 2-allylnaphthalen-1-yl trifluoromethanesulfonate (**4b**)[iii], and 2-allyl-4-methoxyphenol[iv] were prepared according to published procedures. Toluene and dichloromethane were purified using a GlassContour solvent system. Trifluorotoluene was purified by distillation from P_2O_5. All yields refer to isolated compounds that are estimated to be ≥95% pure as judged by ^{1}H NMR analysis. The yields reported in the supporting information describe the result of a single experiment, whereas yields reported in Table 3 are average yields of two or more experiments. Thus, the yields reported in the supporting information may differ from those shown in Table 3.

Preparation and Characterization of Substrates

General Procedure for the Synthesis of 2-allylphenyl triflate Starting Materials

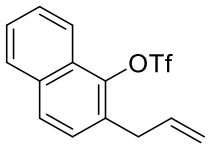


A flame-dried round bottom flask equipped with a stir bar was cooled under a stream of N_2 and charged with the appropriate phenol (1.0 equiv) and K_2CO_3 (2.0 equiv) in acetone (0.2M phenol). Allyl bromide (1.2 equiv) was added and the flask was equipped with a reflux condenser. The flask was then placed in an oil bath and heated to 65 °C for ca 14 h with stirring. The flask was then cooled to rt and the reaction mixture was filtered through a pad of celite. The filtrate was concentrated *in vacuo* and purified by flash chromatography to afford the desired O-allyl phenol product.

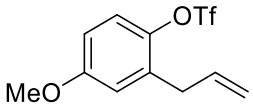
A flame-dried thick-walled glass pressure tube equipped with a stir bar was cooled under a stream of N_2 and charged with the appropriate O-allyl phenol and DMF (1.5 M). The tube was then sealed with a Teflon lined screw cap and placed in an oil bath at 200 °C for 16 h behind a blast shield. The reaction mixture was then cooled to rt, diluted with H_2O (20 mL), and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were washed with brine (60 mL), concentrated *in vacuo*, and purified by flash chromatography to afford the desired 2-allylphenol product.

A flame-dried round bottom flask equipped with a stir bar was cooled under a stream of N_2 and charged with the appropriate 2-allyl phenol (1.0 equiv) and pyridine (2.0 equiv) in DCM (0.5 M). The flask was cooled to 0 °C in an ice bath and Tf_2O (1.2 equiv) was added dropwise at 0 °C. The ice bath was removed and the mixture was warmed to rt and stirred until the starting material had been completely consumed as judged by TLC analysis. The reaction mixture was then filtered through a pad of celite. The filtrate was concentrated *in vacuo* and purified by flash chromatography on silica gel to afford the desired 2-allylphenyl triflate product.

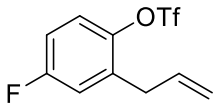
Characterization of Substrates



2-Allylnaphthalen-1-yl trifluoromethanesulfonate (4b). The general procedure was used for the formation of the title compound, starting from 1-naphthol (1.44 g, 10 mmol) and allyl bromide (1.04 mL, 12 mmol). This procedure afforded 1.48 g (47% overall yield) of the title compound as a white solid, mp 49 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (d, J = 8.5 Hz, 1 H) 7.87 (d, J = 8.3 Hz, 1 H) 7.82 (d, J = 8.3 Hz, 1 H) 7.60–7.65 (m, 1 H) 7.52–7.59 (m, 1 H) 7.41 (d, J = 8.5 Hz, 1 H) 5.91– 6.03 (m, 1 H) 5.15–5.22 (m, 2 H) 3.66–3.71 (d, J = 5 Hz, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.3, 134.9, 133.9, 130.6, 128.7, 128, 127.9, 127.8, 127.3, 126.9, 121.5, 118.9 (q, J_{CF} = 320.1 Hz), 117.8, 34.7; IR (film) 1404, 1210, 1136 cm^{-1}. MS (EI) 316.0383 (316.0381 calcd for $C_{14}H_{11}F_3O_3S$, M^+).



2-Allyl-4-methoxyphenyl trifluoromethanesulfonate (4c). The general procedure was used for the formation of the title compound, starting from 4-methoxyphenol (1.24 g, 10 mmol) and allyl bromide (1.73 mL, 20 mmol). This procedure afforded 1.82 g (81% overall yield) of the title compound as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.17 (d, J = 9.0 Hz, 1 H) 6.74–6.84 (m, 2 H) 5.91 (m, 1 H) 5.10–5.20 (m, 2 H) 3.80 (s, 3 H) 3.44 (d, J = 6.6 Hz, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.1, 141.5, 134.6, 134.3, 122.4, 118.8 (q, J_{CF} = 320.3 Hz). 117.8, 116.3, 113, 55.8, 34.4; IR (film) 1205, 1138, 863 cm^{-1}. MS (EI) 296.0341 (296.0330 calcd for $C_{11}H_{11}F_3O_4S$, M^+).



2-Allyl-4-fluorophenyl trifluoromethanesulfonate (4d). The general procedure was used for the formation of the title compound (except the Claisen reaction was conducted for 24 h), starting from 4-fluorophenol (1.12 g, 10 mmol) and allyl bromide (1.04 mL, 12 mmol). This

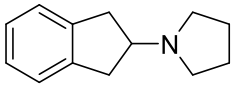
procedure afforded 677 mg (24% overall yield) of the title compound as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.21–7.25 (m, 1 H) 7.04 (dd, J = 8.8, 2.9 Hz, 1 H) 6.94–7.02 (m, 1 H) 5.89 (m, 1 H) 5.21 (d, J = 10 Hz, 1 H) 5.16 (dd, J = 17, 1.3 Hz, 1 H) 3.46 (d, J = 6.6 Hz, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.7 (d, J_{CF} = 247.4 Hz), 135.7 (d, J_{CF} = 7.6 Hz), 133.8, 123.1 (d, J_{CF} = 8.5 Hz), 120, 118.7 (q, J_{CF} = 320.2 Hz), 118.5, 118 (d, J_{CF} = 24.6 Hz), 115 (d, J_{CF} = 23.8 Hz), 34.1; IR (film) 1423, 1212, 1140 cm^{-1}. MS (EI) 284.0143 (284.0130 calcd for $C_{10}H_8F_4O_3S$, M^+).

Preparation and Characterization of Functionalized Indanes

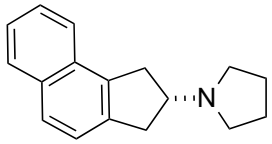
General Procedure for Pd-Catalyzed Alkene Difunctionalization Reactions

A flame-dried Schlenk tube equipped with a stirbar was cooled under a stream of N_2 and charged with $Pd(OAc)_2$ (4 mol %), (*S*)-*t*-Bu-PHOX (10 mol %), the appropriate 2-allylphenyl triflate (0.1 mmol), and lithium *tert*-butoxide (0.14 mmol). The tube was purged with N_2 then toluene (0.5 mL) was added via syringe with stirring. The appropriate amine (0.12 mmol) was then added neat via micro-syringe followed by toluene (0.5 mL) to wash the inside of the Schlenk tube so all reagents were in solution. The resulting mixture was heated to 95 ^{o}C for 3 h at which time the starting material had been completely consumed as judged by ^{1}H NMR analysis of an aliquot removed from the reaction mixture. The mixture was cooled to rt, 1 M aqueous NaOH (2 mL) was added, and the layers were separated. The aqueous layer was extracted with ethyl acetate (3 x 5 mL) then the organic layers were combined, dried over anhydrous Na_2SO_4, filtered, and concentrated *in vacuo*. The crude product was then purified by flash chromatography on silica gel. The fractions containing product were then combined and concentrated *in vacuo* to yield the product•HX salt (which appeared to be generated by protonation by silica gel, unless otherwise noted). The protonated product was then dissolved in dichloromethane (15 mL) and transferred to a separatory funnel containing 1 M NaOH (aq) (10 mL). The aqueous layer was extracted with dichloromethane (3 x 15 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated *in vacuo* to yield the desired 2-aminoindane product.

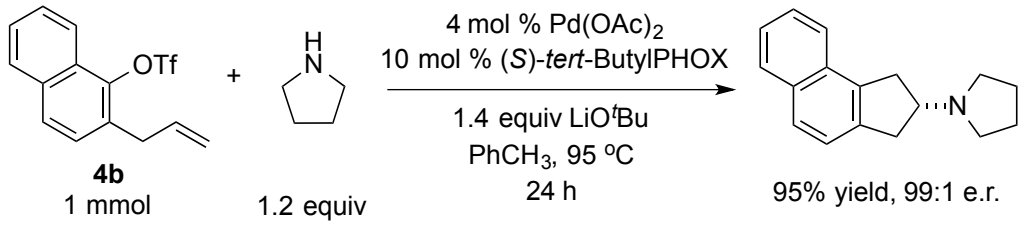
Characterization of Indane Products



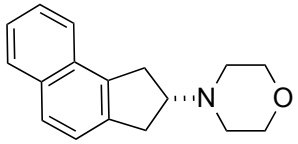
1-(2,3-Dihydro-1*H*-inden-2-yl)pyrrolidine (**7**). The general procedure was used for the coupling of pyrrolidine (19.7 μL, 0.2 mmol) and 2-allylphenyl trifluoromethanesulfonate (53.2 mg, 0.2 mmol), except trifluorotoluene was used as solvent, BrettPhos as ligand, and the reaction was conducted for 16 h. The crude mixture was transferred to a separatory funnel containing 1 M NaOH (aq) (5 mL). The aqueous layer was extracted with dichloromethane (3 x 5 mL). The combined organic layers were dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo* to afford 30 mg (80%) of the title compound as a tan solid, mp 187–189 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.08–7.21 (m, 4 H) 3.38–3.54 (m, 1 H) 3.24–3.13 (m, 4 H) 2.94 (s, br, 4 H) 1.98 (s, br, 4 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.4, 127.0, 124.5, 65.7, 52.6, 37.3, 23.6; IR (film) 2951, 1463 cm^{-1}. MS (ESI) 188.1432 (188.1434 calcd for $C_{13}H_{17}N$, M + H^{+}).



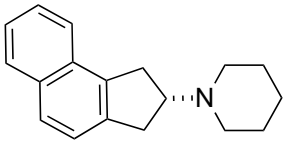
(*R*)-(–)-1-(2,3-Dihydro-1*H*-cyclopenta[*a*]naphthalen-2-yl)pyrrolidine (8a). The general procedure was used for the coupling of pyrrolidine (9.9 μL, 0.12 mmol) and 2-allylnaphthalen-1-yl trifluoromethanesulfonate (32.0 mg, 0.1 mmol). This procedure afforded 23.1 mg (96%) of the title compound as a clear, yellow oil. This material was judged to be >99:1 e.r. by chiral HPLC analysis (Chiralpak ADH, 25 cm x 4.6 mm, 1% IPA/Hexanes (+0.1% diethylamine), 0.5 mL/min, λ 275 nm, R_T = 13.9 min and 27.4 min, $[\alpha]^{23}_{D}$ –29.2 (*c* 2.57, CH_2Cl_2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.1 Hz, 1 H) 7.78 (d, *J* = 8.3 Hz, 1 H) 7.68 (d, *J* = 8.3 Hz, 1 H) 7.46–7.52 (m, 1 H) 7.39–7.46 (m, 1 H) 7.36 (d, *J* = 8.3 Hz, 1 H) 3.50 (dd, *J* = 15.0, 7.5 Hz, 1 H) 3.09–3.36 (m, 4 H) 2.71 (br s, 4 H) 1.89 (br s, 4 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 138.9, 137.3, 132.7, 130.4, 128.6, 127.0, 126.0, 124.9, 124.2, 123.3, 66.2, 53.1, 39.6, 36.8, 23.6; IR (film) 3054, 2930 cm^{-1}. MS (ESI) 238.1593 (238.1590 calcd for $C_{17}H_{19}N$, M + H^{+}).



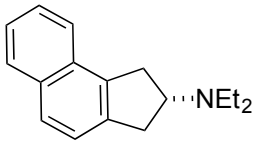
The reaction was also conducted on a larger scale using pyrrolidine (100 μL, 1.2 mmol), 2-allylnaphthalen-1-yl trifluoromethanesulfonate (316 mg, 1 mmol), and 10 mL of toluene. The general procedure was followed except the reaction was heated for 24 h. This procedure afforded 225 mg (95%) of the title compound. This material was judged to be 99:1 e.r. by chiral HPLC analysis.



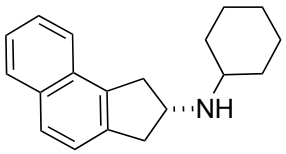
(*R*)-(–)-4-(2,3-Dihydro-1*H*-cyclopenta[*a*]naphthalen-2-yl)morpholine (8b). The general procedure was used for the coupling of morpholine (10.5 μL, 0.12 mmol) and 2-allylnaphthalen-1-yl trifluoromethanesulfonate (32.1 mg, 0.1 mmol) except the product eluted from column chromatography as the free base, so the aqueous NaOH wash was omitted from the post-chromatography procedure. This procedure afforded 23.4 mg (90%) of the title compound as a yellow oil. This material was judged to be 95:5 e.r. by chiral HPLC analysis (Chiralpak ADH, 25 cm x 4.6 mm, 1% IPA/Hexanes (+0.1% diethylamine), 0.5 mL/min, λ 254 nm, R_T = 21.1 min and 24.3 min, $[\alpha]^{23}_D$ –53.1 (*c* 2.12, CH_2Cl_2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.1 Hz, 1 H) 7.77 (d, *J* = 8.1 Hz, 1 H) 7.69 (d, *J* = 8.3 Hz, 1 H) 7.47–7.53 (m, 1 H) 7.40–7.47 (m, 1 H) 7.36 (d, *J* = 8.3 Hz, 1 H) 3.81 (app t, *J* = 4.5 Hz, 4 H) 3.37–3.54 (m, 2 H) 3.06–3.31 (m, 3 H) 2.63 (s, br, 4 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 138.4, 136.8, 132.7, 130.2, 128.6, 127.2, 126.2, 125.0, 124.1, 123.2, 67.1, 66.9, 51.9, 37.6, 34.9; IR (film) 2804, 1116 cm^{-1}. MS (ESI) 254.1543 (254.1539 calcd for $C_{17}H_{19}NO$, M + H^+).



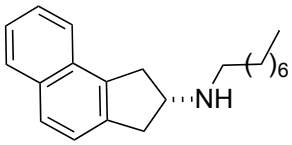
(*R*)-(–)-1-(2,3-Dihydro-1*H*-cyclopenta[*a*]naphthalen-2-yl)piperidine (8c). The general procedure was used for the coupling of piperidine (11.8 μL, 0.12 mmol) and 2-allylnaphthalen-1-yl trifluoromethanesulfonate (31.6 mg, 0.1 mmol). This procedure afforded 23.7 mg (94%) of the title compound as a yellow oil. This material was judged to be 95:5 e.r. by chiral HPLC analysis (Chiralpak ADH, 25 cm x 4.6 mm, 1% IPA/Hexanes (+0.1% diethylamine), 0.5 mL/min, λ 325 nm, R_T = 11.3 min and 12.9 min, $[\alpha]^{23}_D$ –12.7 (*c* 2.93, CH_2Cl_2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.3 Hz, 1 H) 7.78 (d, *J* = 8.1 Hz, 1 H) 7.68 (d, *J* = 8.3 Hz, 1 H) 7.46–7.52 (m, 1 H) 7.40–7.46 (m, 1 H) 7.35 (d, *J* = 8.3 Hz, 1 H) 3.32–3.53 (m, 2 H) 3.05–3.31 (m, 3 H) 2.57 (s, br, 4 H) 1.68 (quint, *J* = 5.6 Hz, 4 H) 1.50 (s, br, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 138.8, 137.2, 132.7, 130.3, 128.6, 127.0, 126.0, 124.9, 124.2, 123.3, 67.1, 52.5, 37.9, 35.1, 26.1, 24.6; IR (film) 3054, 2930 cm^{-1}. MS (ESI) 252.1751 (252.1747 calcd for $C_{18}H_{21}N$, M + H^+).



(*R*)-(–)-*N,N*-Diethyl-2,3-dihydro-1*H*-cyclopenta[*a*]naphthalen-2-amine (8d). The general procedure was used for the coupling of diethylamine (12.4 μL, 0.12 mmol) and 2-allylnaphthalen-1-yl trifluoromethanesulfonate (32 mg, 0.1 mmol). This procedure afforded 11.8 mg (49%) of the title compound as a clear, yellow oil. This material was judged to be 89:11 e.r. by chiral HPLC analysis (Chiralpak ADH, 25 cm x 4.6 mm, 1% IPA/Hexanes (+0.1% diethylamine), 0.5 mL/min, λ 325 nm, R_T = 8.8 min and 10.9 min, $[\alpha]^{23}_D$ –85.9 (*c* 1.41, CH_2Cl_2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.1 Hz, 1 H) 7.78 (d, *J* = 8.1 Hz, 1 H) 7.68 (d, *J* = 8.1 Hz, 1 H) 7.47–7.53 (m, 1 H) 7.39–7.47 (m, 1 H) 7.36 (d, *J* = 8.3 Hz, 1 H) 3.85–3.94 (m, 1 H) 3.48 (dd, *J* = 15.6, 8.1 Hz, 1 H) 3.08–3.29 (m, 3 H) 2.74 (q, *J* = 7.2 Hz, 4 H) 1.13 (t, *J* = 7.2 Hz, 6 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 139, 137.4, 132.7, 130.4, 128.6, 127, 126.1, 124.9, 124.2, 123.3, 62.1, 44.1, 37.9, 35.1, 12.0; IR (film) 2969, 1375 cm^{-1}. MS (ESI) 240.1749 (240.1747 calcd for $C_{17}H_{21}N$, M + H^+).

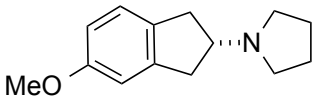


(*R*)-(–)-*N*-Cyclohexyl-2,3-dihydro-1*H*-cyclopenta[*a*]naphthalen-2-amine (8e). The general procedure was used for the coupling of cyclohexylamine (13.7 μL, 0.12 mmol) and 2-allylnaphthalen-1-yl trifluoromethanesulfonate (31.4 mg, 0.1 mmol) except with a reaction time of 4 h. This procedure afforded 18.5 mg (69%) of the title compound as a white solid, mp 64-67 °C. This material was judged to be >99:1 e.r. by chiral HPLC analysis (Chiralpak ADH, 25 cm x 4.6 mm, 1% IPA/Hexanes (+0.1% diethylamine), 0.5 mL/min, λ 254 nm, R_T = 19.1 min and 20.6 min, $[\alpha]^{23}_D$ –26.7 (*c* 2.22, CH_2Cl_2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.1 Hz, 1 H) 7.77 (d, *J* = 8.1 Hz, 1 H) 7.68 (d, *J* = 8.3 Hz, 1 H) 7.45–7.56 (m, 1 H) 7.39–7.45 (m, 1 H) 7.36 (d, *J* = 8.3 Hz, 1 H) 4.00 (quint, *J* = 7.0 Hz, 1 H) 3.57 (dd, *J* = 15.7, 7.5 Hz, 1 H) 3.32–3.43 (m, 1 H) 2.87–3.10 (m, 2 H) 2.62–2.74 (m, 1 H) 1.97 (app. d, *J* = 11 Hz, 2 H) 1.73–1.83 (app. d, *J* = 15 Hz, 2 H) 1.67 (app. d, *J* = 12.7 Hz, 1 H) 1.11–1.38 (m, 6 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 138.708, 137.2, 132.8, 130.6, 128.6, 127.1, 126.1, 124.9, 124.3, 123.5, 56.1, 55.1, 41.5, 38.8, 33.9, 26.3, 25.3; IR (film) 2925, 1448 cm^{-1}. MS (ESI) 266.1902 (266.1903 calcd for $C_{19}H_{23}N$, M + H^+).

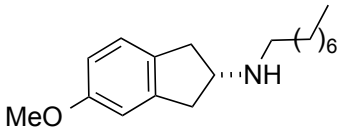


(*R*)-(–)-*N*-Octyl-2,3-dihydro-1*H*-cyclopenta[*a*]naphthalen-2-amine (8f). The general procedure was used for the coupling of *n*-octylamine (19.8 μL, 0.12 mmol) and 2-allylnaphthalen-1-yl trifluoromethanesulfonate (31.9 mg, 0.1 mmol) except with a reaction time of 16 h. This procedure afforded 24.7 mg (83%) of the title compound as an orange oil. This material was judged to be 97:3 e.r. by chiral HPLC analysis (Chiralpak ADH, 25 cm x 4.6 mm, 1% IPA/Hexanes (+0.1% diethylamine), 0.5 mL/min, λ 254 nm, R_T = 22.1 min and 26.0 min, $[\alpha]^{23}_D$ –32.0 (*c* 2.26, CH_2Cl_2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.3 Hz, 1 H) 7.73–7.81 (m, 1 H) 7.68 (d, *J* = 8.1 Hz, 1 H) 7.47–7.53 (m, 1 H) 7.40–7.46 (m, 1 H) 7.36 (d, *J* = 8.3 Hz, 1 H) 3.76–3.89 (m, 1 H) 3.56 (dd, *J* = 15.9, 7.3 Hz, 1 H) 3.37 (dd, *J* = 15.9, 7.3 Hz, 1 H) 3.07 (dd,

J = 15.9, 5.9 Hz, 1 H) 2.96 (dd, *J* = 15.8, 6.0 Hz, 1 H) 2.68–2.80 (m, 2 H) 1.58–1.52 (m, 2 H) 1.23–1.42 (m, 10 H) 0.91–0.88 (m, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 138.8, 137.3, 132.8, 130.6, 128.6, 127.1, 126.1, 124.9, 124.3, 123.5, 59.5, 48.5, 41.2, 38.4, 31.9, 30.5, 29.7, 29.4, 27.7, 22.8, 14.2; IR (film) 2924, 2852 cm^{-1}. MS (ESI) 296.2374 (296.2373 calcd for $C_{21}H_{29}N$, M + H^+).

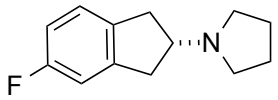


(*R*)-(+)-1-(5-Methoxy-2,3-dihydro-1*H*-inden-2-yl)pyrrolidine (8g). The general procedure was used for the coupling of pyrrolidine (9.9 μL, 0.12 mmol) and 2-allyl-4-methoxyphenyl trifluoromethanesulfonate (29.6 mg, 0.1 mmol). This procedure afforded 12.6 mg (57%) of the title compound as a clear, yellow oil. This material was judged to be 97:3 e.r. by chiral HPLC analysis (Chiralpak ADH, 25 cm x 4.6 mm, 1% IPA/Hexanes (+0.1% diethylamine), 0.5 mL/min, λ 275 nm, R_T = 14.5 min and 17.8 min, $[\alpha]^{23}_D$ +5.5 (*c* 1.91, CH_2Cl_2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.07 (d, *J* = 8.1 Hz, 1 H) 6.75 (s, 1 H) 6.69 (d, *J* = 8.1 Hz, 1 H) 3.77 (s, 3 H) 2.82–3.14 (m, 5 H) 2.63 (s, br, 4 H) 1.85 (s, br, 4 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.8, 143.5, 134.1, 125.0, 112.2, 110.2, 67.2, 55.5, 53.2, 39.3, 38.2, 23.6; IR (film) 2946, 1247 cm^{-1}. MS (ESI) 218.1541 (218.1539 calcd for $C_{14}H_{19}NO$, M + H^+).



(*R*)-(+)-5-Methoxy-*N*-octyl-2,3-dihydro-1*H*-inden-2-ylamine (8h). The general procedure was used for the coupling of *n*-octylamine (19.8 μL, 0.12 mmol) and 2-allyl-4-methoxyphenyl trifluoromethanesulfonate (29.4 mg, 0.1 mmol) except using a reaction time of 4 h This procedure afforded 16.7 mg (61%) of the title compound as a clear, yellow oil. This material was judged to be >99:1 e.r. by chiral HPLC analysis (Chiralpak OJH, 25 cm x 4.6 mm, 1% IPA/Hexanes (+0.1% diethylamine), 0.5 mL/min, λ 275 nm, R_T = 17.4 min and 19.9 min, $[\alpha]^{23}_D$ +5.81 (*c* 1.79, CH_2Cl_2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.08 (d, *J* = 8.1 Hz, 1 H) 6.76 (s, 1 H) 6.70 (d, *J* = 8.3 Hz, 1 H) 3.77 (s, 3 H) 3.65–3.60 (m, 1 H) 3.12 (td, *J* = 16, 7.1 Hz, 2 H) 2.61–2.78 (m, 4 H) 1.54–1.48 (m, 2 H) 1.20–1.38 (m, 11 H) 0.88 (t, *J* = 6.7 Hz, 3 H); ^{13}C NMR (125

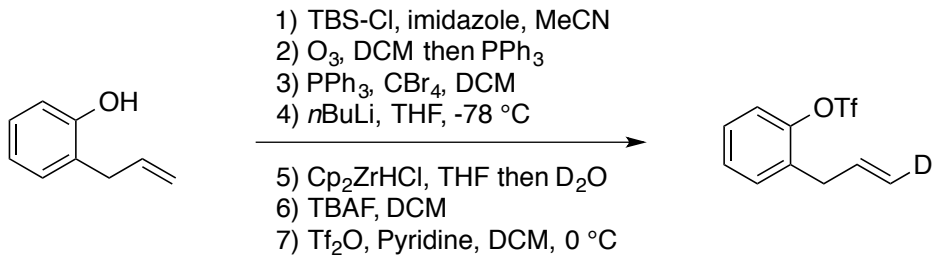
MHz, $CDCl_3$) 158.9, 143.4, 133.9, 125.3, 112.4, 110.4, 60.3, 55.5, 48.5, 40.5, 39.3, 31.9, 30.5, 29.7, 29.4, 27.6, 22.8, 14.2; IR (film) 2925, 1245 cm^{-1}. MS (ESI) 276.2321 (276.2322 calcd for $C_{18}H_{29}NO$, M + H^+).



(*R*)-(+)-1-(5-Fluoro-2,3-dihydro-1*H*-inden-2-yl)pyrrolidine (8i). The general procedure was used for the coupling of pyrrolidine (9.9 μL, 0.12 mmol) and 2-allyl-4-fluorophenyl trifluoromethanesulfonate (29.2 mg, 0.1 mmol). This procedure afforded 16.4 mg (78%) of the title compound as a yellow solid, mp 39-42 °C. This material was judged to be 98:2 e.r. by chiral HPLC analysis (Chiralpak ODH, 15 cm x 4.6 mm, 1% IPA/Hexanes (+0.1% diethylamine), 0.5 mL/min, λ 275 nm, R_T = 5.7 min and 6.9 min, $[\alpha]^{23}_D$ +1.56 (*c* 1.6, CH_2Cl_2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.09 (dd, *J* = 7.9, 5.5 Hz, 1 H) 6.87 (d, *J* = 9.0 Hz, 1 H) 6.76–6.85 (m, 1 H) 2.98–3.17 (m, 3 H) 2.82–2.98 (m, 2 H) 2.62 (s, br, 4 H) 1.78–1.90 (m, 4 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.8, 143.5, 134.1, 125.0, 112.2, 110.2, 67.2, 55.5, 53.2, 39.3, 38.2, 23.6; IR (film) 2960, 1486 cm^{-1}. MS (ESI) 206.1337 (206.1340 calcd for $C_{13}H_{16}FN$, M + H^+).

Aminopalladation Stereochemistry Studies

Deuterated Substrate Synthesis



(*E*)-2-(Allyl-3-*d*)phenyl trifluoromethanesulfonate (*d*-4a). Conditions were based on a procedure reported by de Koning and coworkers.[v] A flame dried round-bottom flask was charged with MeCN (200 mL), 2-allylphenol (4.90 mL, 37.3 mmol), and imidazole (3.05 g, 44.8 mmol). *tert*-Butyldimethylsilyl chloride (6.75 g, 44.8 mmol) was then added to the solution in one portion and the reaction was stirred at rt for 18 h. The reaction mixture was concentrated *in*

vacuo and the resulting material was transferred to a separatory funnel using EtOAc (200 mL). The organic suspension was then washed with water (200 mL) and the aqueous layer was washed with additional portions of EtOAc (2 x 200 mL). The combined organic layers were then washed with brine (300 mL), dried over anhydrous sodium sulfate, filtered and concentrated. The resulting crude product was purified by flash chromatography to obtain 8.75 g (94%) of (2-allylphenoxy)(*tert*-butyl)dimethylsilane as a clear oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17–7.04 (m, 2 H), 6.89 (t, *J* = 7.4 Hz, 1 H), 6.79 (dd, *J* = 8.0, 1.2 Hz, 1 H), 5.98 (ddt, *J* = 18.9, 9.4, 6.6 Hz, 1 H), 5.11–4.97 (m, 2 H), 3.37 (d, *J* = 6.5 Hz, 2 H), 1.02 (s, 9 H), 0.23 (s, 6 H). Spectroscopic data were consistent with those previously reported.[v]

A flame dried round-bottom flask was charged with (2-allylphenoxy)(*tert*-butyl)dimethylsilane (3.00 g, 12.1 mmol), and dichloromethane (173 mL). The solution was then cooled to –78 °C and ozone was bubbled through the reaction mixture until a blue color persisted (ca. 30 min). The mixture was then sparged with nitrogen and PPh_3 6.33 g (24.2 mmol, 2 equiv) was added. The solution was warmed to rt and stirred for 12 h. The mixture was then concentrated *in vacuo* and the crude product was triturated with hexanes, filtered and concentrated. The crude product was then purified by flash chromatography to obtain 2.56 g (85%) of 2-{2-[(*tert*-butyldimethylsilyl)oxy]phenyl}acetaldehyde as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.69 (t, *J* = 2.2 Hz, 1 H), 7.19 (td, *J* = 7.8, 1.8 Hz, 1 H), 7.15 (dd, *J* = 7.5, 1.8 Hz, 1 H), 6.95 (td, *J* = 7.4, 1.2 Hz, 1 H), 6.87 (dd, *J* = 8.0, 1.2 Hz, 1 H), 3.63 (d, *J* = 2.2 Hz, 2 H), 0.99 (s, 9 H), 0.25 (s, 6 H). Spectroscopic data were consistent with those previously reported.[v]

Conditions were based on a procedure reported by Corey and coworkers.[vi] A flame dried round-bottom flask was charged with PPh_3 (2.32 g, 37.1 mmol), CBr_4 (6.14 g, 18.5 mmol) and dichloromethane (46 mL). The mixture was then cooled to 0 °C and 2-{2-[(*tert*-butyldimethylsilyl)oxy]phenyl}acetaldehyde (2.32 g, 9.26 mmol) was added slowly via syringe. The mixture was stirred for 40 min at 0 °C and then filtered through a plug of silica using dichloromethane as the eluent. The resulting solution was concentrated and the crude product was then purified by flash chromatography to obtain 3.63 g (97%) of *tert*-butyl[2-(3,3-dibromoallyl)phenoxy]dimethylsilane as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14

(ddd, J = 15.5, 7.5, 1.8 Hz, 2 H), 6.91 (td, J = 7.5, 1.2 Hz, 1 H), 6.81 (dd, J = 8.0, 1.2 Hz, 1 H), 6.58 (t, J = 7.1 Hz, 1 H), 3.40 (d, J = 7.1 Hz, 2 H), 1.02 (s, 9 H), 0.26 (s, 6 H).

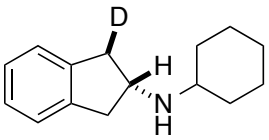
Conditions were based on a procedure reported by Corey and coworkers.[vi] A flame dried round-bottom flask was charged with *tert*-butyl[2-(3,3-dibromoallyl)phenoxy]dimethylsilane (3.62 g, 8.86 mmol) and THF (58 mL). The mixture was then cooled to –78 °C and *n*BuLi (2.5 M in hexanes, 7.27 mL, 18.18 mmol) was added slowly. The resulting mixture was stirred at –78 °C for 1 h, then was warmed to rt and stirred for an additional 1 h. The mixture was then poured into water (150 mL) and the aqueous layer was extracted with hexanes (3 x 150 mL). The combined organic layers were washed with brine, dried over anhydrous sodium sulfate, filtered and concentrated. The crude product was purified by flash chromatography and the resulting product was further purified by kuglerohr distillation to obtain 1.13 g (52%) of *tert*-butyldimethyl[2-(prop-2-yn-1-yl)phenoxy]silane as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, J = 7.7 Hz, 1 H), 7.12 (t, J = 7.0 Hz, 1 H), 6.95 (t, J = 7.5 Hz, 1 H), 6.78 (d, J = 8.1 Hz, 1 H), 3.56 (d, J = 2.7 Hz, 2 H), 2.16 (t, J = 2.7 Hz, 1 H), 1.02 (s, 9 H), 0.24 (s, 6 H).

Conditions were based on a procedure reported by Chemler and coworkers.[vii] A flame dried round-bottom flask was charged with bis(cyclopentadienyl)zirconium chloride hydride (704 mg, 2.73 mmol) followed by a solution of *tert*-butyldimethyl[2-(prop-2-yn-1-yl)phenoxy]silane (538 mg, 2.18 mmol) in THF (1.10 mL). The resulting mixture was stirred at rt for 2.5 h at which time D_2O (58 μL, 3.2 mmol) was added via syringe. The reaction mixture was stirred at rt for an additional 18 h then was diluted with 1:1 Et_2O/hexanes (20 mL), dried over anhydrous sodium sulfate, filtered and concentrated. The crude product was purified by flash chromatography to obtain 507 mg (93%) of (*E*)-(2-(allyl-3-*d*)phenoxy)(*tert*-butyl)dimethylsilane as a colorless oil. This material contained ca 12% of an inseparable side product that resulted from over reduction of the alkyne to the alkane. Data are for the major product. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17–7.04 (m, 2 H), 6.89 (td, J = 7.5, 1.2 Hz, 1 H), 6.79 (dd, J = 8.0, 1.2 Hz, 1 H), 6.04–5.91 (m, 1 H), 5.03 (dt, J = 17.0, 1.7 Hz, 1 H), 3.37 (dd, J = 6.5, 1.6 Hz, 2 H), 1.01 (s, 9 H), 0.23 (s, 6 H).

A flame dried round-bottom flask was charged with (*E*)-[2-(allyl-3-*d*)phenoxy](*tert*-butyl)dimethylsilane (300 mg, 1.20 mmol), dichloromethane (2.4 mL) and TBAF (1 M in THF, 2.4 mL, 2.4 mmol). The mixture was stirred at rt for 1.5 h then was poured into saturated aqueous ammonium chloride (20 mL) and extracted with Et_2O (3 x 30 mL). The combined

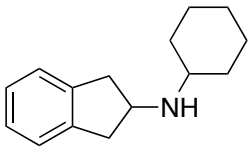
organic layers were then washed with brine (30 mL), dried over anhydrous sodium sulfate, filtered and concentrated. The crude product was then purified by flash chromatography to obtain 116 mg (72%) of (*E*)-2-(allyl-3-*d*)phenol as a colorless oil. This material contained ca 12% of an inseparable side product that resulted from over reduction of the alkyne to the alkane in the previous step. Data are for the major product. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19–7.10 (m, 2 H), 6.90 (td, *J* = 7.4, 1.2 Hz, 1 H), 6.82 (dd, *J* = 8.0, 1.2 Hz, 1 H), 6.04 (dtt, *J* = 17.1, 6.4, 1.5 Hz, 1 H), 5.16 (dt, *J* = 17.1, 1.7 Hz, 1 H), 4.96 (s, br, 1 H), 3.43 (dd, *J* = 6.3, 1.6 Hz, 2 H).

A flame dried round-bottom flask was charged with (*E*)-2-(allyl-3-*d*)phenol (83.4 mg, 0.617 mmol) and dichloromethane (1.23 mL). The reaction mixture was cooled to 0 °C and pyridine was added (100 μL, 1.23 mmol) followed by the dropwise addition of trifluoromethanesulfonic anhydride (125 μL, 0.74 mmol). The reaction mixture was allowed to warm to rt while stirring overnight. The mixture was then diluted with dichloromethane (15 mL), the layers were separated, and the organic layer was washed with water (10 mL), saturated aqueous copper sulfate (10 mL) and brine (10 mL). The organic layer was dried over anhydrous sodium sulfate, filtered and concentrated. The crude product was purified by flash chromatography to obtain 94 mg (56%) of (*E*)-2-(allyl-3-*d*)phenyl trifluoromethanesulfonate as a clear oil. This material contained ca 12% of an inseparable side product that resulted from over reduction of the alkyne to the alkane in a prior step. The desired product contained 90.9% deuterium incorporation as determined by MS analysis. Data are for the major product. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.37–7.23 (m, 5 H), 5.92 (m, 1 H), 5.11 (d, *J* = 17.0 Hz, 1 H), 3.49 (dd, *J* = 6.6, 1.6 Hz, 2 H). ^{13}C NMR (176 MHz, $CDCl_3$) δ 148.1, 134.6, 133.0, 131.6, 128.5, 128.3, 121.5, 118.7 (q, J_{CF} = 320.0 Hz), 117.4 (t, J_{CD} = 24.3 Hz), 34.1; IR (film) 1419, 1211, 1137 cm^{-1}; MS (EI) 267.0278 (267.0287 calcd for $C_{10}H_8DF_3O_3S$, M^+).



(1*R*,2*R*)-*N*-Cyclohexyl-2,3-dihydro-1*H*-inden-1-*d*-2-amine (*d*-7). The general procedure was used for the coupling of cyclohexylamine (13.8 μL, 0.12 mmol) and (*E*)-2-(allyl-3-*d*)phenyl

trifluoromethanesulfonate (26.7 mg, 0.1 mmol) except the reaction was conducted for 16 h. This procedure afforded 6.8 mg (31%) of the title compound as a yellow oil. This product contained 98.1% deuterium incorporation as determined by MS analysis. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.20–7.16 (m, 2 H), 7.16–7.09 (m, 2 H), 3.79 (q, *J* = 7.2 Hz, 1 H), 3.17 (dd, *J* = 15.4, 7.1 Hz, 1 H), 2.75 – 2.67 (m, 2 H), 2.58 (tt, *J* = 10.6, 3.8 Hz, 1 H), 1.97–1.85 (m, 2 H), 1.81–1.70 (m, 2 H), 1.68–1.60 (m, 1 H), 1.51 (s, br, 1 H), 1.32–1.24 (m, 2 H), 1.21–1.06 (m, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 142.0, 126.5, 124.8, 56.6, 55.1, 40.6, 40.3 (t, *J* = 20.0 Hz), 34.0, 26.3, 25.4. MS (ESI) 217.1809 (217.1810 calcd for $C_{15}H_{21}DN$, M + H^+).

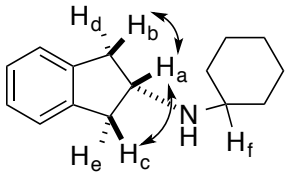


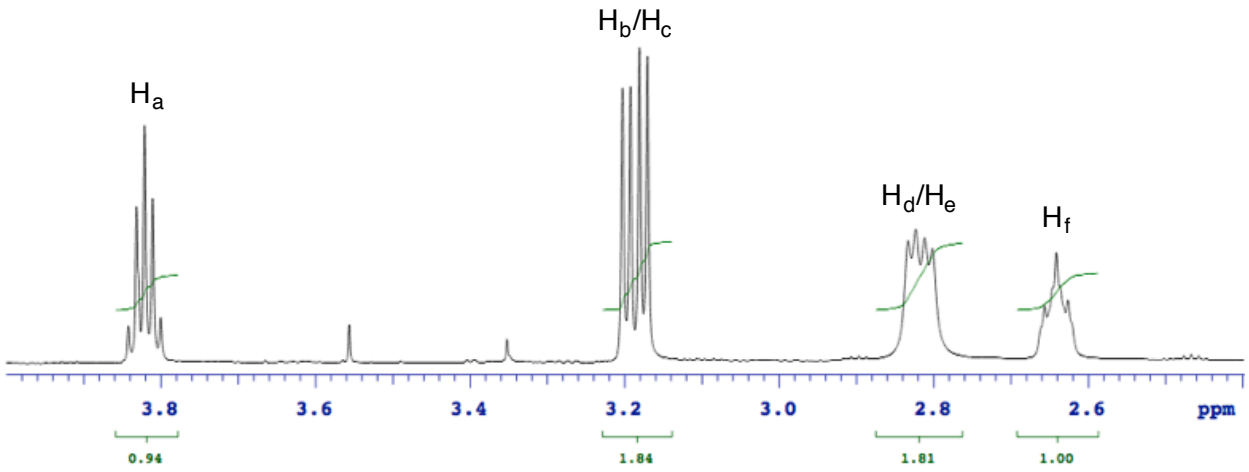
***N*-Cyclohexyl-2,3-dihydro-1*H*-inden-2-amine**. The general procedure was used for the coupling of cyclohexylamine (27.5 μL, 0.24 mmol) and 2-allylphenyl trifluoromethanesulfonate (53 mg, 0.2 mmol) except the scale of the reaction was doubled, BrettPhos was used as the ligand and the reaction was conducted for 16 h. This procedure afforded 23 mg (54%) of the title compound as a yellow oil. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.18 (dd, *J* = 5.3, 3.4 Hz, 2 H), 7.16–7.11 (m, 2 H), 3.82 (pent, *J* = 7.3 Hz, 1 H), 3.19 (dd, *J* = 15.4, 7.2 Hz, 2 H), 2.82 (dd, *J* = 15.7, 7.3 Hz, 2 H), 2.64 (tt, *J* = 10.7, 3.8 Hz, 1 H), 1.96 (dd, *J* = 12.7, 3.9 Hz, 2 H), 1.76 (dp, *J* = 10.7, 3.7 Hz, 2 H), 1.69–1.60 (m, 1 H), 1.36–1.24 (m, 2 H), 1.24–1.13 (m, 3 H); ^{13}C NMR (176 MHz, $CDCl_3$) δ 141.6, 126.6, 124.8, 56.5, 55.3, 40.2, 33.5, 26.2, 25.3; IR (film) 2922, 2849, 1448 cm^{-1}; MS (ESI) 216.1744 (216.1747 calcd for $C_{15}H_{22}N$, M + H^+).

Assignment of stereochemistry for *d*-7

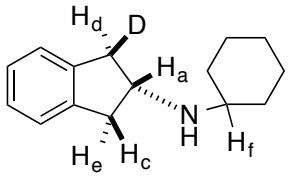
The stereochemical assignment of (+/-)-(1*R*,2*R*)-*N*-cyclohexyl-2,3-dihydro-1*H*-inden-1-*d*-2-amine was determined through comparison with the all proteo analogue *N*-cyclohexyl-2,3-dihydro-1*H*-inden-2-amine. Peak assignments for *N*-cyclohexyl-2,3-dihydro-1*H*-inden-2-amine were made using gCOSY and 1D NOESY experiments.

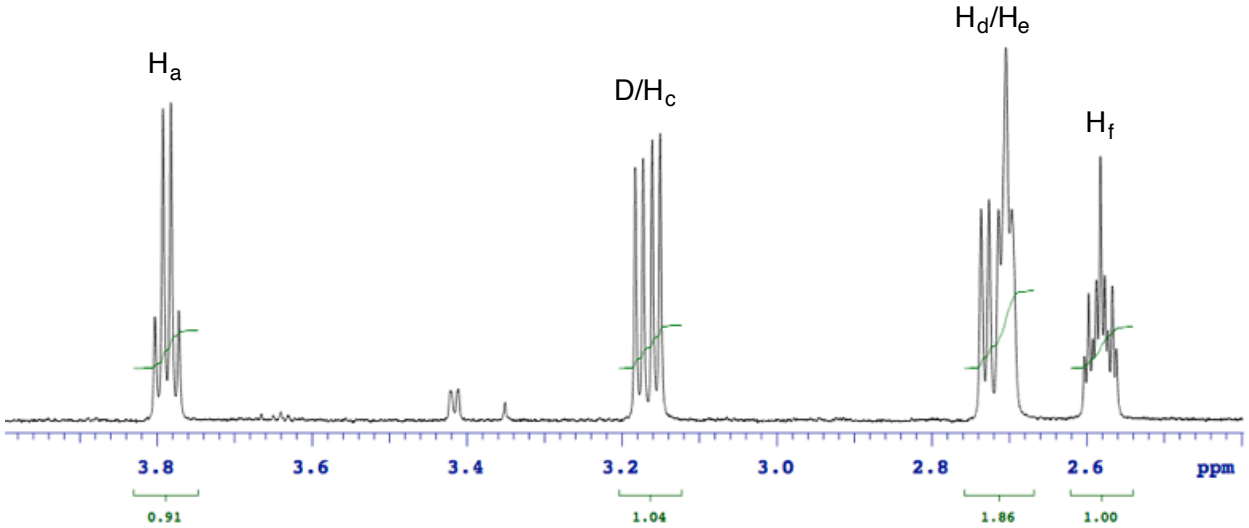
***N*-cyclohexyl-2,3-dihydro-1*H*-inden-2-amine**



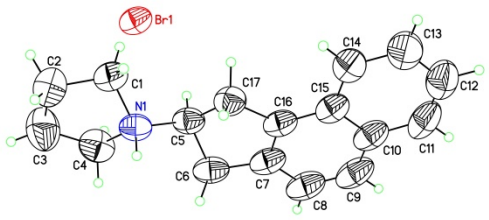


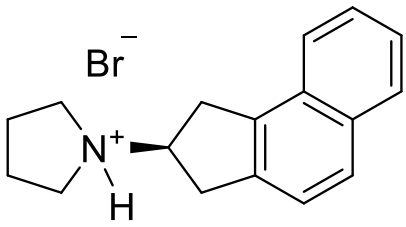
(+/-)-(1*R*,2*R*)-*N*-cyclohexyl-2,3-dihydro-1*H*-inden-1-*d*-2-amine





Absolute stereochemical assignment of 8a Stereochemical assignment of **8a** was accomplished by single-crystal x-ray analysis of **8a·HBr** salt. **8a** was protonated by dropwise addition of hydrobromic acid (48 wt. %, 3 equiv) to a stirring solution of **8a** (40 mg, 0.17 mmol) in 30% diethyl ether/hexanes mixture (0.1 M). The resulting mixture was stirred for 1 h until a yellow solid precipitated from solution. The solid was filtered and washed with hexanes (15 mL). The solid was then dissolved in dichloromethane, dried with Na_2SO_4, filtered, and concentrated to yield a pale-yellow solid. The product was then recrystallized from a saturated toluene solution by slow evaporation. The structure analysis of **8a·HBr** indicated this material had a 1*R* stereochemical configuration.





References

(i) Krout, M. R.; Mohr, J. T.; Stoltz, B. M. *Org. Synth.* **2009**, *86*, 181.

(ii) Ganton, M. D.; Kerr, M. A. *Org. Lett.* **2005**, *7*, 4777.

(iii) Reich, S. H.; Melnick, M; Pino, M. J.; Fuhry, M. A. M.; Trippe, A. J.; Appelt, K.; Davies , J. F., II; Wu, B. -W.; Musick, L. *J. Med. Chem.* **1996**, *39*, 2781.

(iv) Beierlein, J. M.; Karri, N. G.; Anderson, A. C. *J. Med. Chem.* **2010**, *53*, 7327.

(v) Pelly, S. C.; Govender, S.; Fernandes, M. A.; Schmalz, H. G.; Koning; C. D. *J. Org. Chem.* **2007**, *72*, 2857.

(vi) Corey, E. J.; Fuchs, P. L. *Tetrahedron Lett.* **1972**, *13*, 3769.

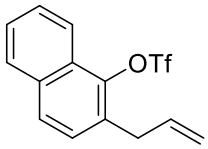
(vii) Sherman, E. M.; Fuller, P. H.; Kasi, D.; Chemler, S. R. *J. Org. Chem.* **2007**, *72*, 3896.

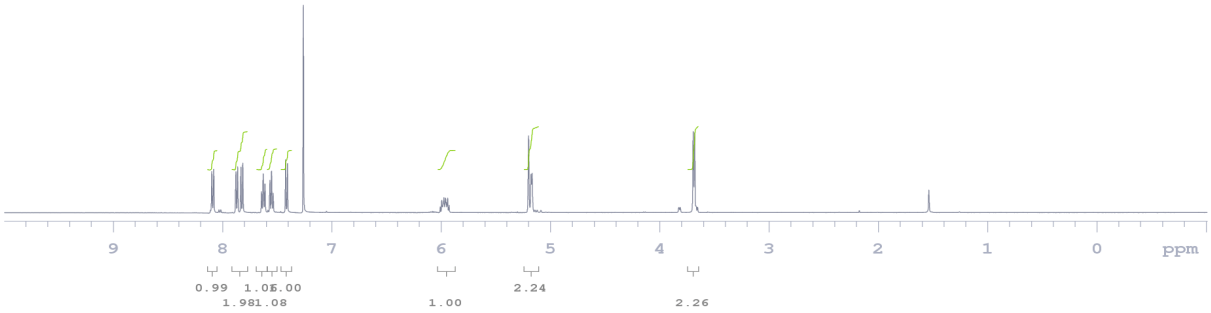
Sample Name
Date collected **2015-03-31**

Pulse sequence **PROTON**
Solvent **cdcl3**

Temperature **25**
Operator **derickrw**

Study owner **derickrw**
Printed from **dy.chem.lsa.umich.edu-vnmrs500**





Data file /misc/i500/derickrw/vnmrsys/data/DW_IV_100_3_c2.fid

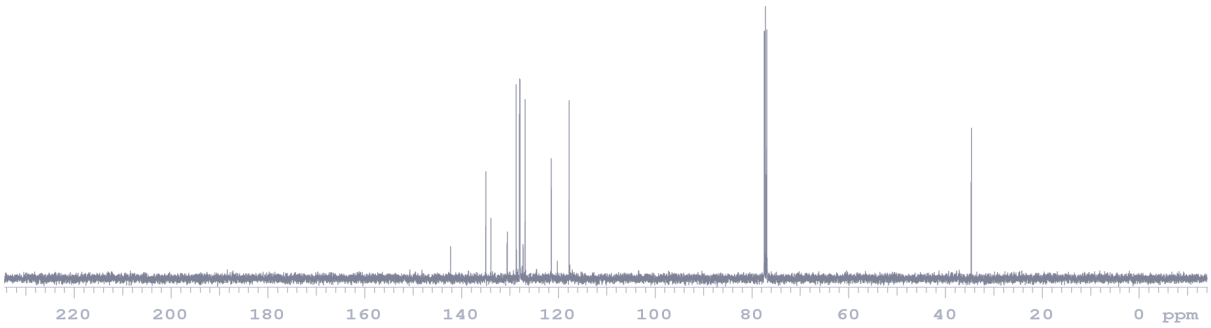
Plot date 2015-05-14

Sample Name
Date collected **2015-05-14**

Pulse sequence **CARBON**
Solvent **cdcl3**

Temperature **24**
Operator **derickrw**

Study owner **derickrw**
Printed from **dy.chem.lsa.umich.edu-vnmrs500**



Data file /misc/Tellurium/derickrw/vnmrsys/data/DW_IV_100_CNMR.fid

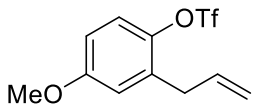
Plot date 2015-05-14

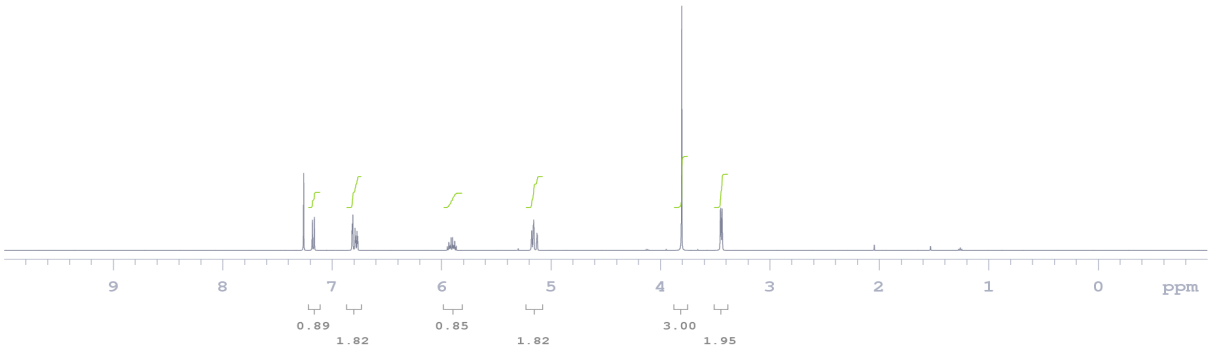
Sample Name
Date collected **2015-03-02**

Pulse sequence **PROTON**
Solvent **cdcl3**

Temperature **25**
Operator **derickrw**

Study owner **derickrw**
Printed from **dy.chem.lsa.umich.edu-vnmrs500**





Data file /misc/i500/derickrw/vnmrsys/data/DW_IV_63_3_c1.fid

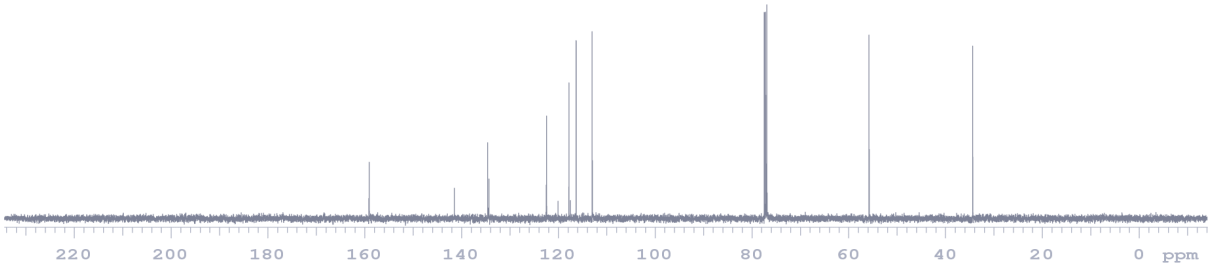
Plot date 2015-05-14

Sample Name
Date collected **2015-05-14**

Pulse sequence **CARBON**
Solvent **cdcl3**

Temperature **24**
Operator **derickrw**

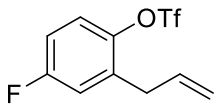
Study owner **derickrw**
Printed from **dy.chem.lsa.umich.edu-vnmrs500**

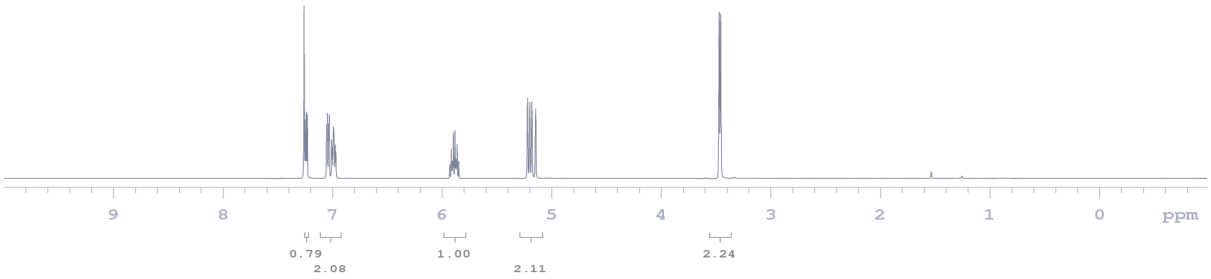


Data file /misc/Tellurium/derickrw/vnmrsys/data/DW_IV_63_CNMR.fid

Plot date 2015-05-14

Sample Name	Pulse sequence **PROTON**	Temperature **25**	Study owner **derickrw**
Date collected **2015-03-23**	Solvent **cdcl3**	Operator **derickrw**	Printed from **dy.chem.lsa.umich.edu-vnmrs500**

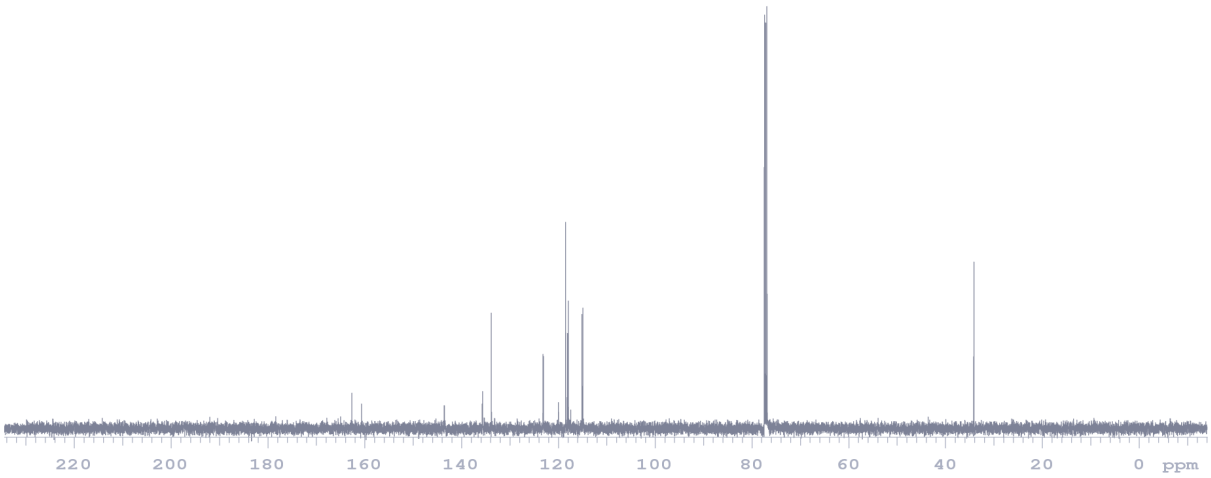




Data file /misc/i500/derickrw/vnmrsys/data/DW_IV_90_3_c1.fid

Plot date 2015-05-14

Sample Name	Pulse sequence **CARBON**	Temperature **24**	Study owner **derickrw**
Date collected **2015-05-14**	Solvent **cdcl3**	Operator **derickrw**	Printed from **dy.chem.lsa.umich.edu-vnmrs500**



Data file /misc/Tellurium/derickrw/vnmrsys/data/DW_IV_90_CNMR.fid

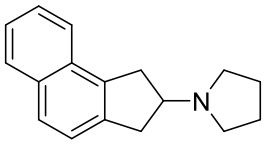
Plot date 2015-05-14

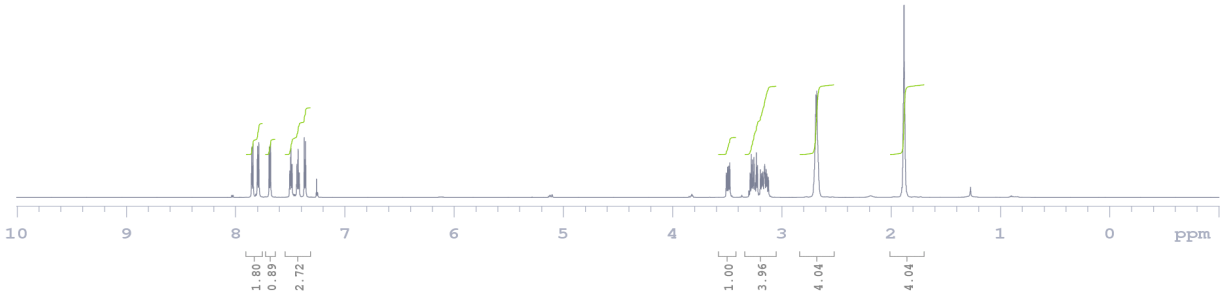
Sample Name
Date collected **2015-04-20**

Pulse sequence **PROTON**
Solvent **cdcl3**

Temperature **23**
Operator **derickrw**

Study owner **derickrw**
Printed from **zn.chem.lsa.umich.edu-vnmrs400**





Data file /misc/Ytterbium/derickrw/vnmrsys/data/DW_IV_139_c1w1.fid

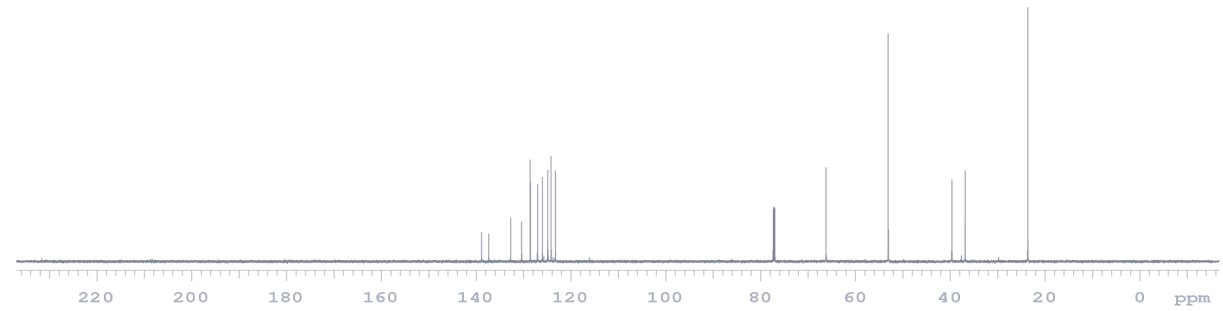
Plot date 2015-04-21

Sample Name
Date collected **2015-04-20**

Pulse sequence **CARBON**
Solvent **cdcl3**

Temperature **23**
Operator **derickrw**

Study owner **derickrw**
Printed from **zn.chem.lsa.umich.edu-vnmrs400**



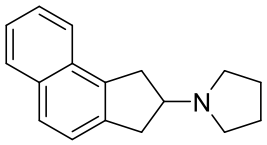
Data file /misc/Ytterbium/derickrw/vnmrsys/data/DW_IV_114_1_CNMR.fid

Plot date 2015-05-04

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

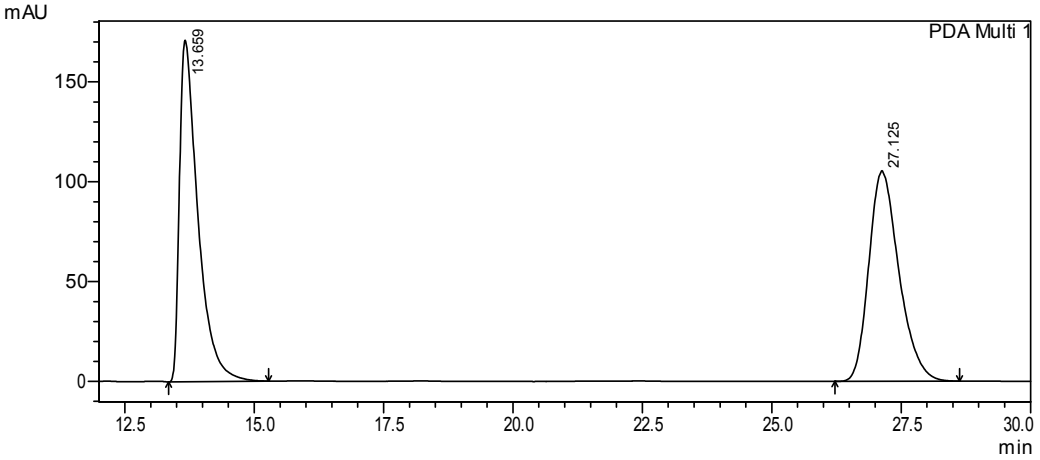
C:\...\Data\DERKPHOS\naphthyltrifate\nitrogen nu\RAC-DW-IV-43(1)-0.5mL_min-1.00%IPA+0.1%DEA-ADH_2.lcd

Acquired by	: Admin
Sample Name	:RAC-DW-IV-43(1)-0.5mL_min-1.00%IPA+0.1%DEA-ADH_2
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-DW-IV-43(1)-0.5mL_min-1.00%IPA+0.1%DEA-ADH_2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2/9/2015 4:08:44 PM
Data Processed	: 2/9/2015 4:43:54 PM



<Chromatogram>

C:\...\Data\DERKPHOS\naphthyltrifate\nitrogen nu\RAC-DW-IV-43(1)-0.5mL_min-1.00%IPA+0.1%DEA-ADH_2.lcd



1 PDA Multi 1/275nm 4nm

PeakTable

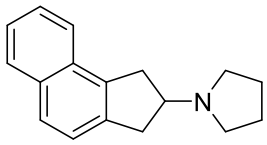
PDA Ch1 275nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.659	4245610	171119	49.853	61.867
2	27.125	4270643	105475	50.147	38.133
Total		8516253	276593	100.000	100.000

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

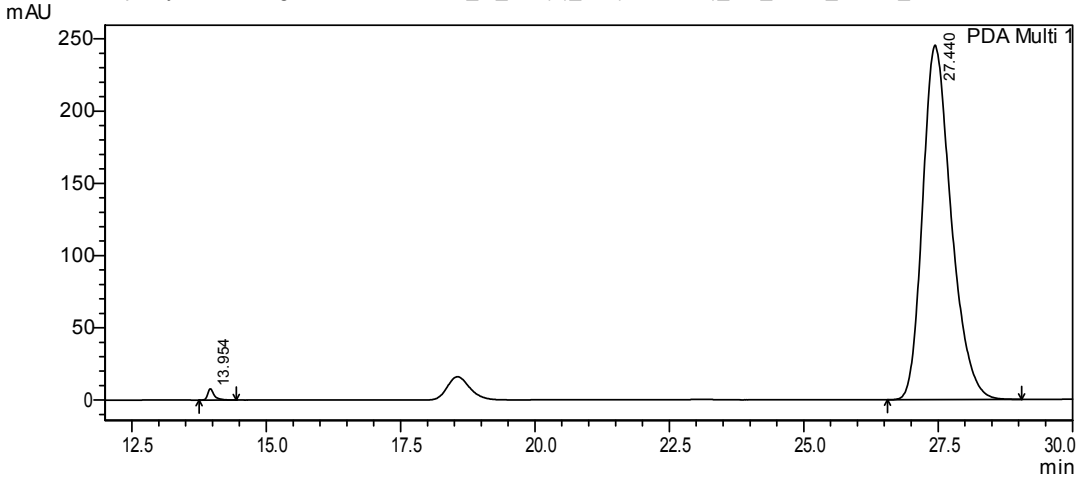
C:\...\DERKPHOS\naphthyltrifate\nitrogen nu\CHIRAL-DW_IV_155(1)_hex(0.1%DEA)_IPA_99to1_0.5mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-DW_IV_155(1)_hex(0.1%DEA)_IPA_99to1_0.5mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-DW_IV_155(1)_hex(0.1%DEA)_IPA_99to1_0.5mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/1/2015 2:30:46 PM
Data Processed	: 5/1/2015 3:01:03 PM



<Chromatogram>

C:\...\naphthyltrifate\nitrogen nu\CHIRAL-DW_IV_155(1)_hex(0.1%DEA)_IPA_99to1_0.5mL_min-ADH.lcd



1 PDA Multi 1/275nm 4nm

PeakTable

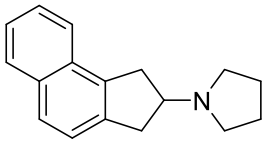
PDA Ch1 275nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.954	67629	7846	0.741	3.098
2	27.440	9059167	245378	99.259	96.902
Total		9126796	253224	100.000	100.000

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

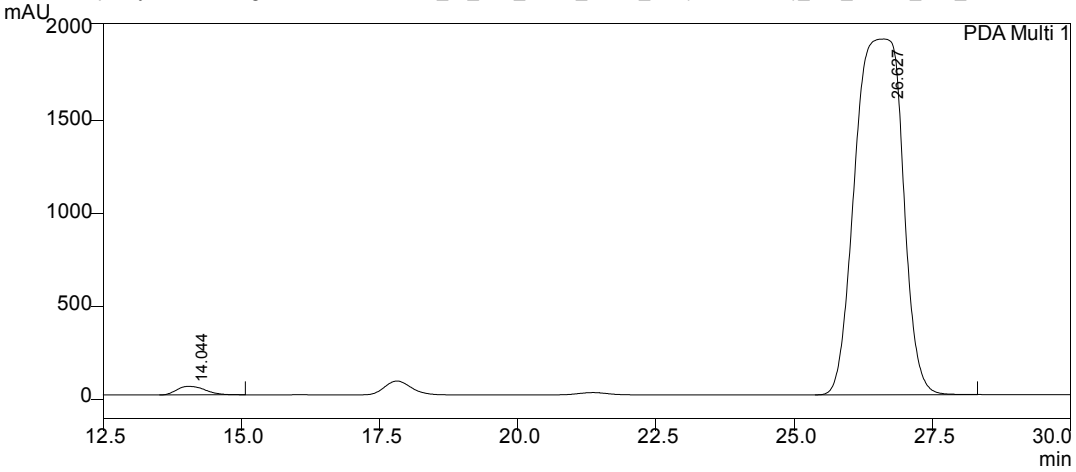
C:\...\naphthyltrifate\nitrogen nu\CHIRAL-DW_IV_185_c1f18_99to1_hex(+0.1%DEA)_IPA_0.5mL_min_ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-DW_IV_185_c1f18_99to1_hex(+0.1%DEA)_IPA_0.5mL_min_ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-DW_IV_185_c1f18_99to1_hex(+0.1%DEA)_IPA_0.5mL_min_ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 8/13/2015 1:50:53 PM
Data Processed	: 8/13/2015 2:40:07 PM



<Chromatogram>

C:\...\naphthyltrifate\nitrogen nu\CHIRAL-DW_IV_185_c1f18_99to1_hex(+0.1%DEA)_IPA_0.5mL_min_ADH.lcd



1 PDA Multi 1/275nm 4nm

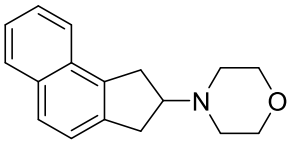
PeakTable

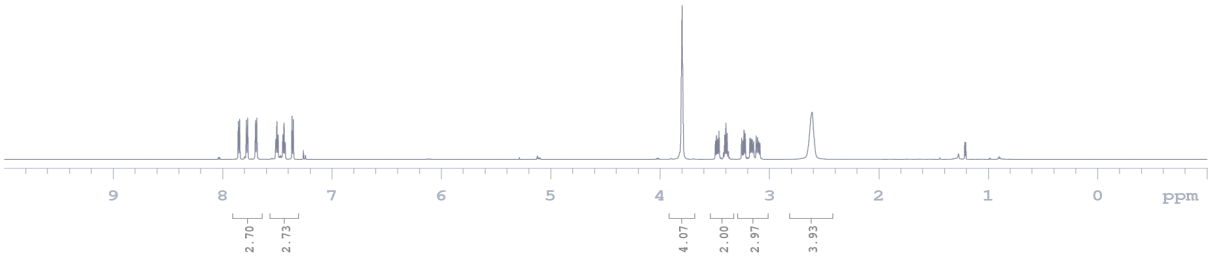
PDA Ch1 275nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.044	1656256	45802	1.426	2.334
2	26.627	114476920	1916818	98.574	97.666
Total		116133175	1962620	100.000	100.000

This HPLC trace of is the 1 mmol scale reaction.

Sample Name	Pulse sequence **PROTON**	Temperature **23**	Study owner **derickrw**
Date collected **2015-04-20**	Solvent **cdcl3**	Operator **derickrw**	Printed from **zn.chem.lsa.umich.edu-vnmrs400**

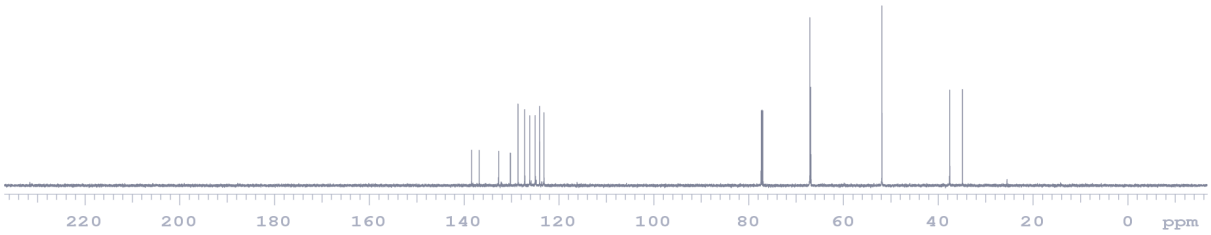




Data file /misc/Ytterbium/derickrw/vnmrsys/data/DW_IV_114_2_HNMR.fid

Plot date 2015-04-21

Sample Name	Pulse sequence **CARBON**	Temperature **23**	Study owner **derickrw**
Date collected **2015-04-20**	Solvent **cdcl3**	Operator **derickrw**	Printed from **zn.chem.lsa.umich.edu-vnmrs400**



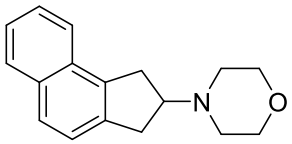
Data file /misc/Ytterbium/derickrw/vnmrsys/data/DW_IV_114_2_CNMR.fid

Plot date 2015-05-04

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

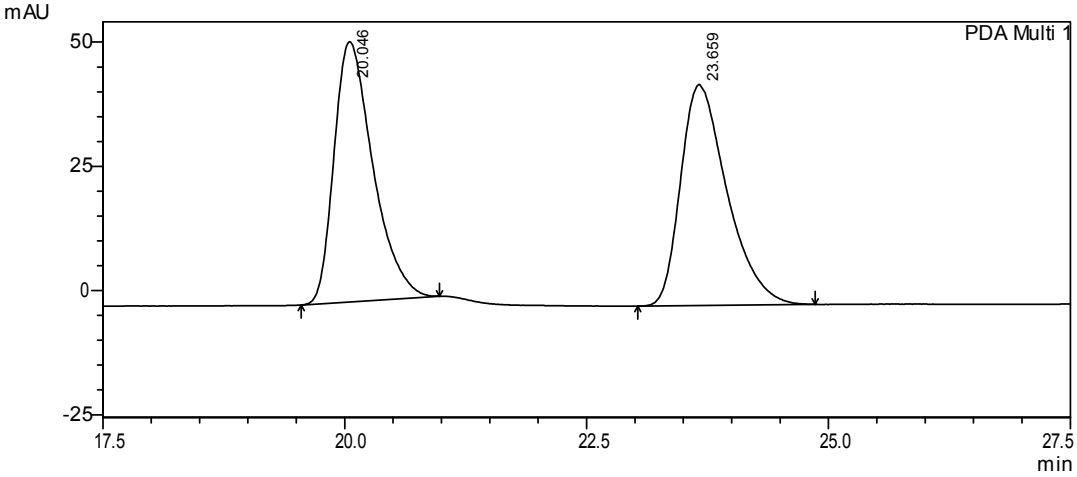
C:\...\naphthyltrifate\nitrogen nu\RAC-DW_IV_1=60(2)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH_4.lcd

Acquired by	: Admin
Sample Name	: RAC-DW_IV_60(2)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH_4
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-DW_IV_1=60(2)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH_4.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/12/2015 1:10:38 PM
Data Processed	: 5/12/2015 1:47:12 PM



<Chromatogram>

C:\...\naphthyltrifate\nitrogen nu\RAC-DW_IV_1=60(2)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH_4.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

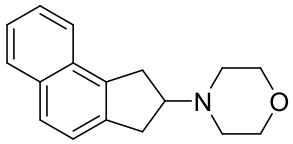
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.046	1466648	52373	49.699	54.065
2	23.659	1484398	44498	50.301	45.935
Total		2951046	96871	100.000	100.000

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

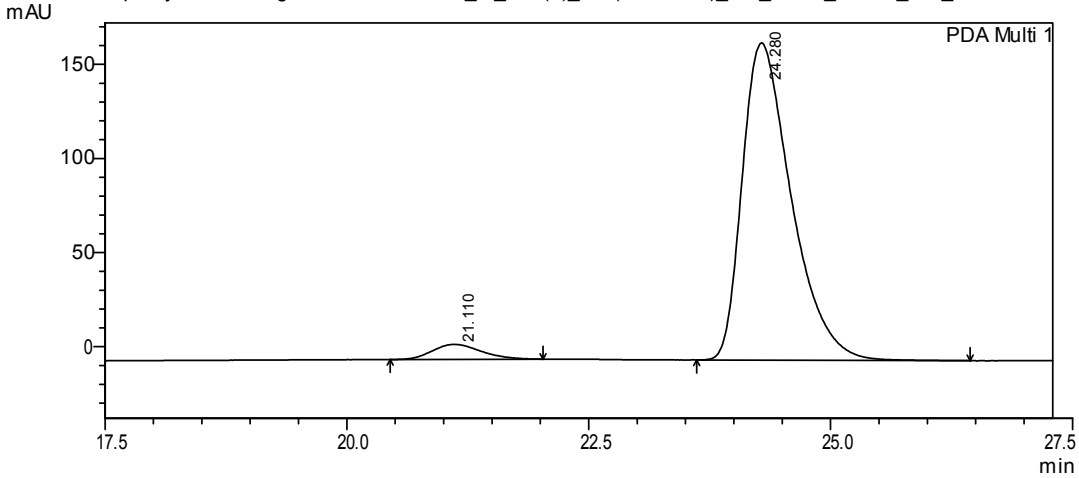
C:\...\DERKPHOS\naphthyltrifate\nitrogen nu\CHIRAL-DW_IV_152(1)_hex(0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-DW_IV_152(1)_hex(0.1%DEA)_IPA_99to1_0.5mL_min_ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-DW_IV_152(1)_hex(0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/30/2015 10:50:46 AM
Data Processed	: 4/30/2015 11:18:06 AM



<Chromatogram>

C:\...\naphthyltrifate\nitrogen nu\CHIRAL-DW_IV_152(1)_hex(0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

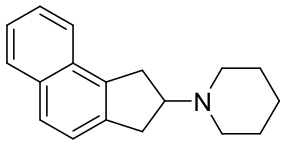
Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.110	279658	7969	4.491	4.514
2	24.280	5947168	168573	95.509	95.486
Total		6226826	176542	100.000	100.000

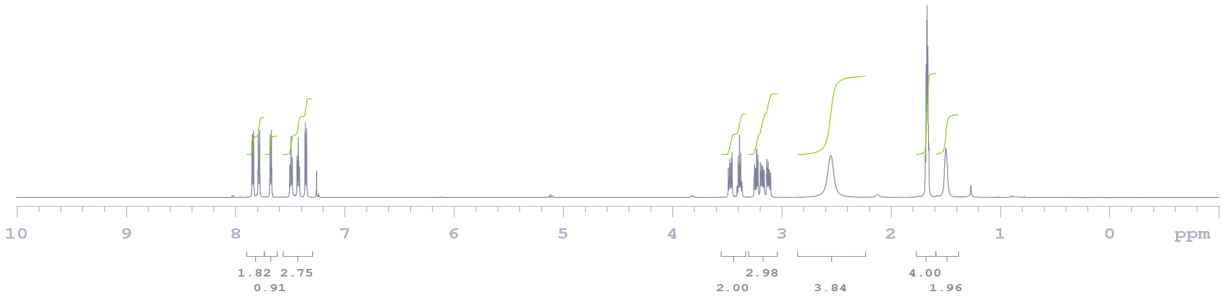
Sample Name
Date collected **2015-04-20**

Pulse sequence **PROTON**
Solvent **cdcl3**

Temperature **23**
Operator **derickrw**

Study owner **derickrw**
Printed from **zn.chem.lsa.umich.edu-vnmrs400**





Data file /misc/Ytterbium/derickrw/vnmrsys/data/DW_IV_130_2_HNMR.fid

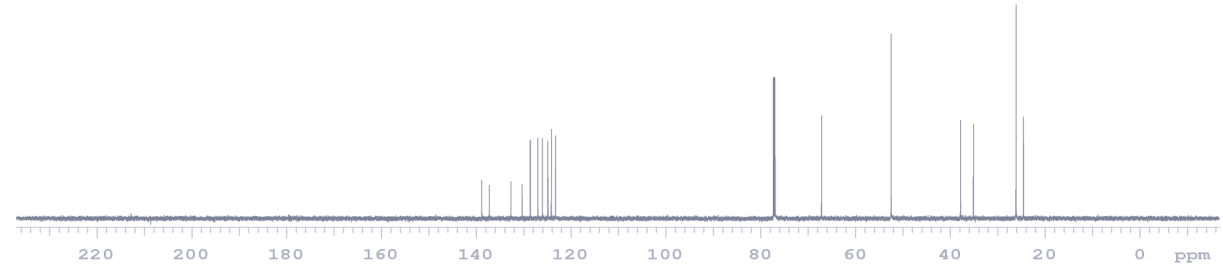
Plot date 2015-04-21

Sample Name
Date collected **2015-04-20**

Pulse sequence **CARBON**
Solvent **cdcl3**

Temperature **23**
Operator **derickrw**

Study owner **derickrw**
Printed from **zn.chem.lsa.umich.edu-vnmrs400**



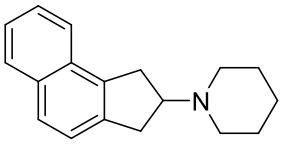
Data file /misc/Ytterbium/derickrw/vnmrsys/data/DW_IV_130_2_CNMR.fid

Plot date 2015-05-04

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

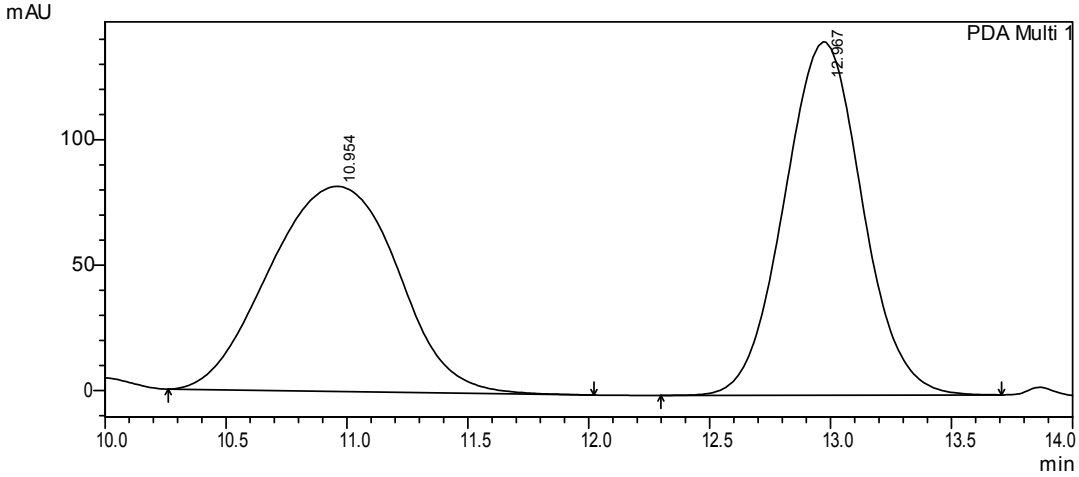
C:\...\DERKPHOS\naphthyltrifate\nitrogen nu\RAC-DW_IV_43(2)_hex(+0.1%DEA)+_IPA_99to1_0.5mL_min_ADH_3.lcd

Acquired by	: Admin
Sample Name	: RAC-DW_IV_43(2)_hex(+0.1%DEA)+_IPA_99to1_0.5mL_min_ADH_3
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-DW_IV_43(2)_hex(+0.1%DEA)+_IPA_99to1_0.5mL_min_ADH_3.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/20/2015 7:53:26 AM
Data Processed	: 4/20/2015 8:09:48 AM



<Chromatogram>

C:\...\naphthyltrifate\nitrogen nu\RAC-DW_IV_43(2)_hex(+0.1%DEA)+_IPA_99to1_0.5mL_min_ADH_3.lcd



1 PDA Multi 1/325nm 4nm

PeakTable

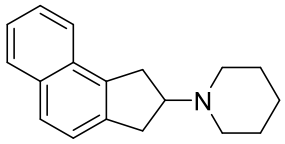
PDA Ch1 325nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.954	3030818	81719	48.901	36.721
2	12.967	3166996	140823	51.099	63.279
Total		6197814	222542	100.000	100.000

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

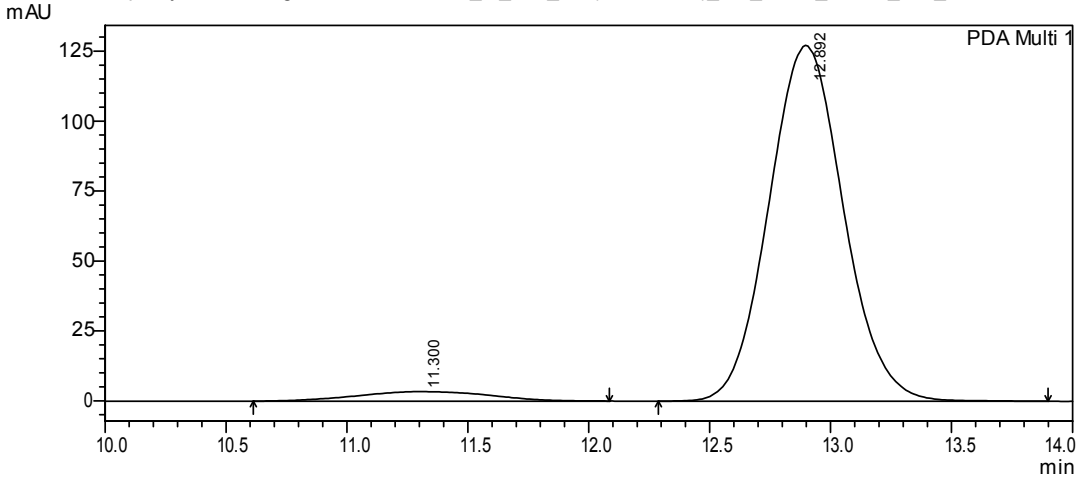
C:\...\DERKPHOS\naphthyltrifate\nitrogen nu\CHIRAL-DW_IV_148_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-DW_IV_148_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-DW_IV_148_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 4/27/2015 9:53:14 AM
Data Processed	: 4/27/2015 10:08:51 AM



<Chromatogram>

C:\...\naphthyltrifate\nitrogen nu\CHIRAL-DW_IV_148_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd



1 PDA Multi 1/325nm 4nm

PeakTable

PDA Ch1 325nm 4nm

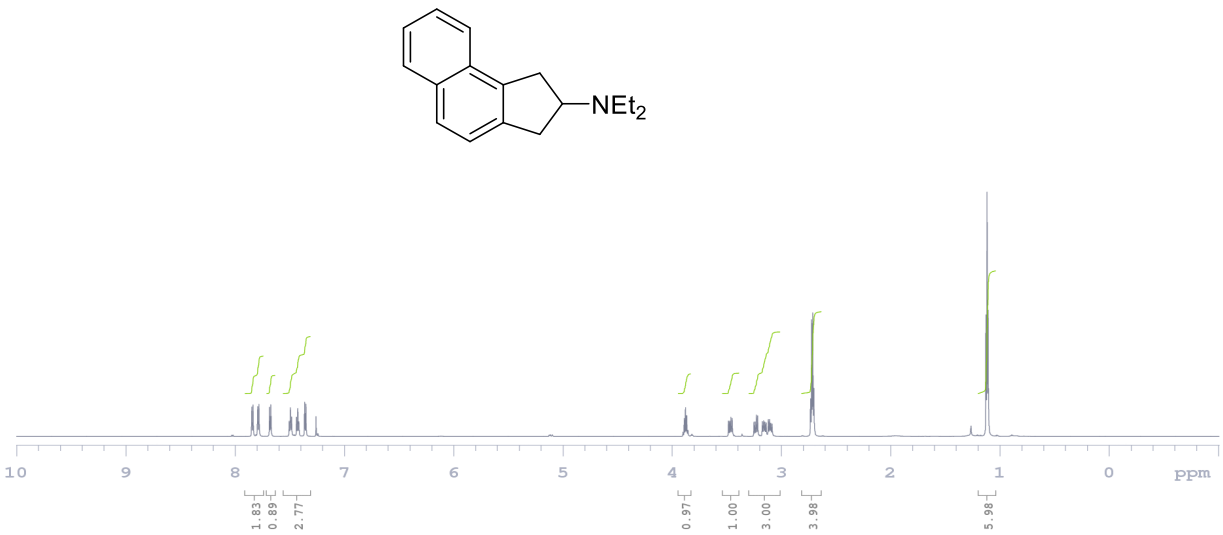
Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.300	129973	3405	4.543	2.607
2	12.892	2730701	127198	95.457	97.393
Total		2860673	130603	100.000	100.000

Sample Name
Date collected **2015-04-20**

Pulse sequence **PROTON**
Solvent **cdcl3**

Temperature **23**
Operator **derickrw**

Study owner **derickrw**
Printed from **zn.chem.lsa.umich.edu-vnmrs400**



Data file /misc/Ytterbium/derickrw/vnmrsys/data/DW_IV_130_1_HNMR.fid

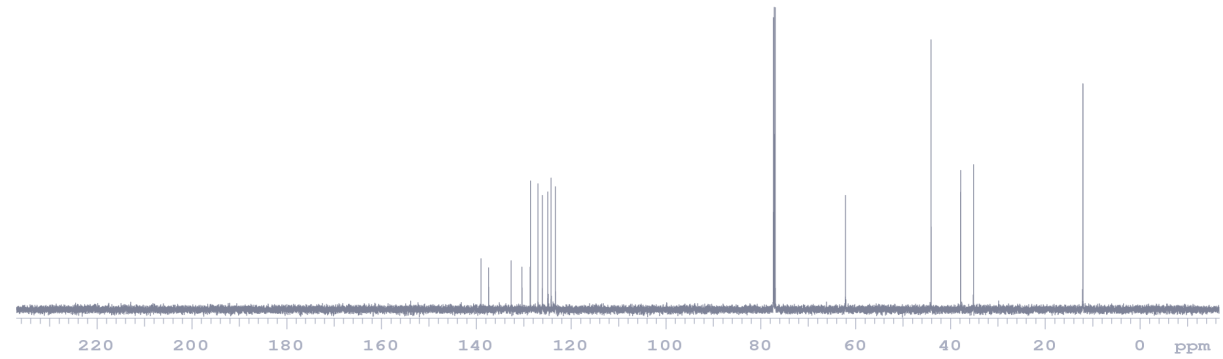
Plot date 2015-04-21

Sample Name
Date collected **2015-04-20**

Pulse sequence **CARBON**
Solvent **cdcl3**

Temperature **23**
Operator **derickrw**

Study owner **derickrw**
Printed from **zn.chem.lsa.umich.edu-vnmrs400**



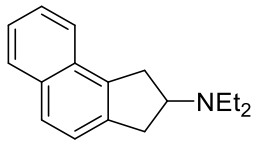
Data file /misc/Ytterbium/derickrw/vnmrsys/data/DW_IV_130_1_CNMR.fid

Plot date 2015-05-04

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

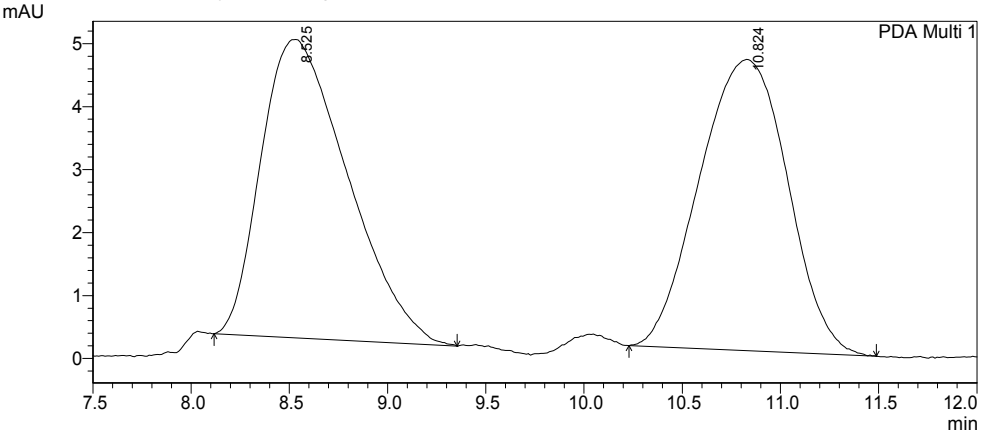
C:\...\DERKPHOS\naphthyltrifate\nitrogen nu\RAC-DW_IV_43(4)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd

Acquired by	: Admin
Sample Name	: RAC-DW_IV_43(4)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-DW_IV_43(4)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/18/2015 3:57:09 PM
Data Processed	: 3/18/2015 4:15:01 PM



<Chromatogram>

C:\...\DERKPHOS\naphthyltrifate\nitrogen nu\RAC-DW_IV_43(4)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd



1 PDA Multi 1/325nm 4nm

PeakTable

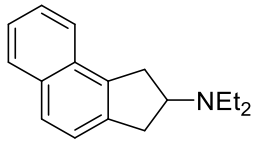
PDA Ch1 325nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.525	145735	4737	50.202	50.612
2	10.824	144560	4623	49.798	49.388
Total		290296	9360	100.000	100.000

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

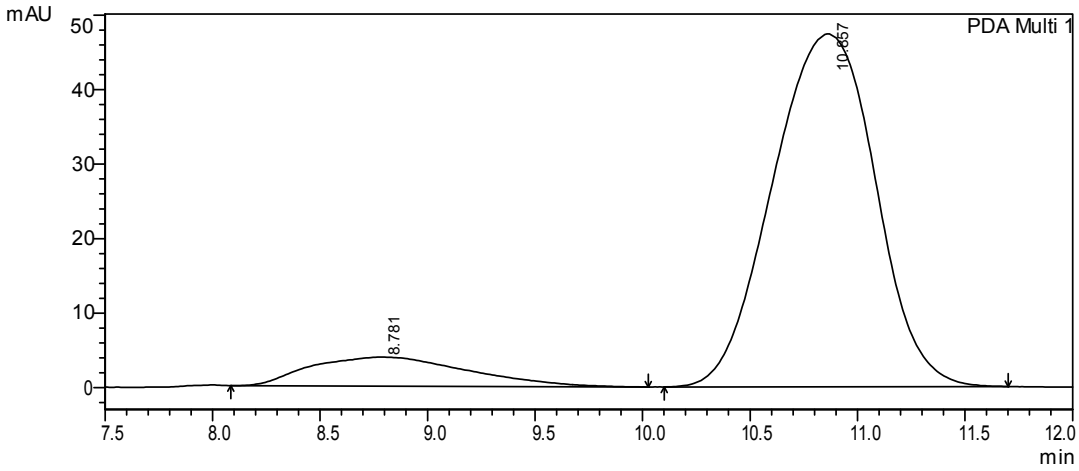
C:\...\naphthyltrifate\nitrogen nu\CHIRAL-DW_IV_159(2)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-DW_IV_159(2)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-DW_IV_159(2)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/5/2015 11:33:38 AM
Data Processed	: 5/5/2015 11:52:50 AM



<Chromatogram>

C:\...\naphthyltrifate\nitrogen nu\CHIRAL-DW_IV_159(2)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd



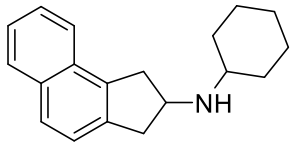
1 PDA Multi 1/325nm 4nm

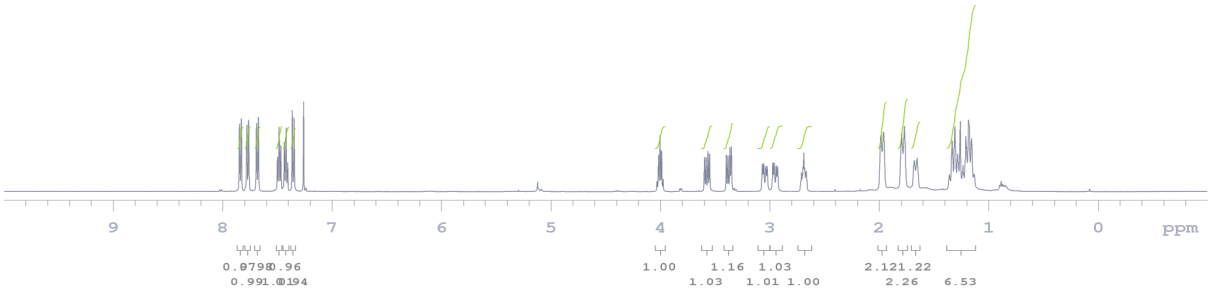
PeakTable

PDA Ch1 325nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.781	199213	3907	11.221	7.621
2	10.857	1576151	47356	88.779	92.379
Total		1775364	51263	100.000	100.000

Sample Name	Pulse sequence **PROTON**	Temperature **25**	Study owner **derickrw**
Date collected **2015-05-07**	Solvent **cdcl3**	Operator **derickrw**	Printed from **kr.chem.lsa.umich.edu-vnmrs500**

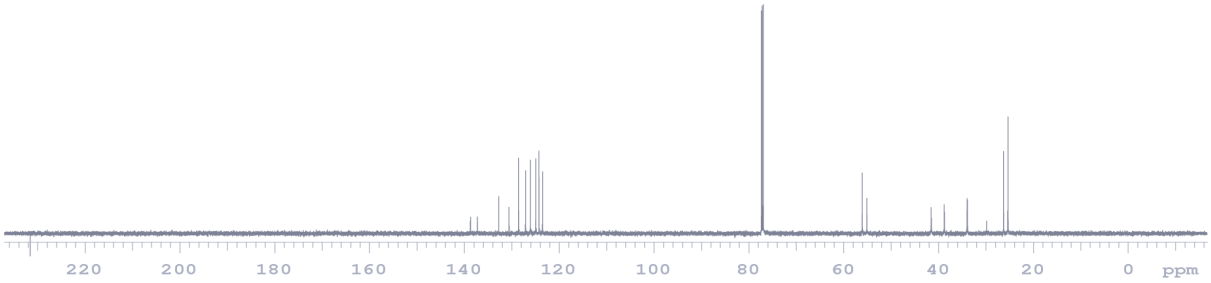




Data file /misc/i500/derickrw/vnmrsys/data/DW_IV_163_1_c1w1.fid

Plot date 2015-05-14

Sample Name	Pulse sequence **CARBON**	Temperature **25**	Study owner **derickrw**
Date collected **2015-05-07**	Solvent **cdcl3**	Operator **derickrw**	Printed from **dy.chem.lsa.umich.edu-vnmrs500**



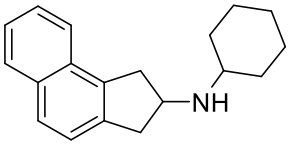
Data file /misc/Ytterbium/derickrw/vnmrsys/data/DW_IV_163_1_CNMR.fid

Plot date 2015-05-14

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

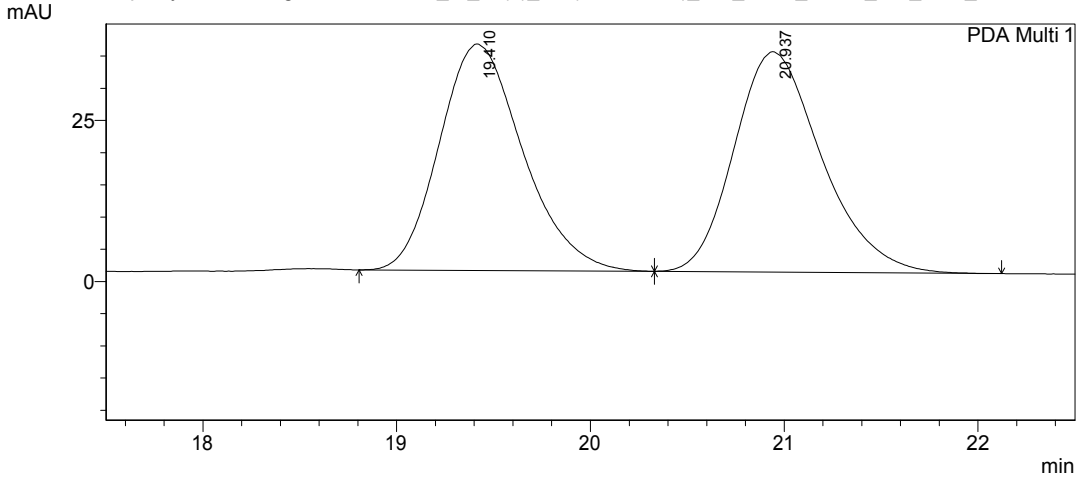
C:\...\DERKPHOS\naphthyltrifate\nitrogen nu\RAC-DW_IV_95(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH_2.lcd

Acquired by	: Admin
Sample Name	: RAC-DW_IV_95(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH_2
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-DW_IV_95(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH_2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/7/2015 12:48:36 PM
Data Processed	: 5/7/2015 1:11:13 PM



<Chromatogram>

C:\...\naphthyltrifate\nitrogen nu\RAC-DW_IV_95(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH_2.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

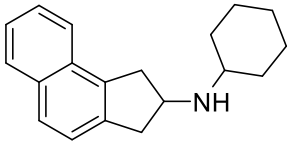
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.410	1051368	35179	49.470	50.695
2	20.937	1073910	34215	50.530	49.305
Total		2125278	69393	100.000	100.000

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

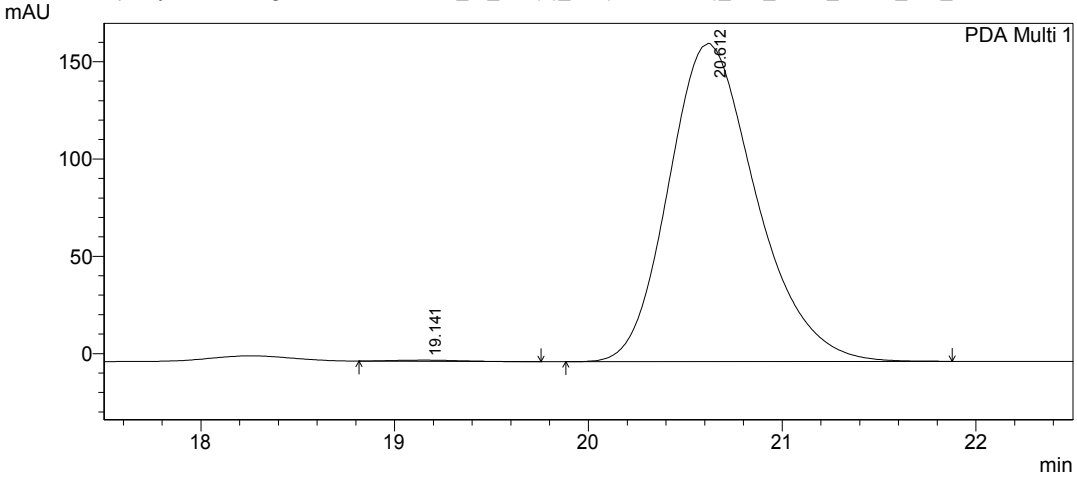
C:\...\naphthyltrifate\nitrogen nu\CHIRAL-DW_IV_163(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-DW_IV_163(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-DW_IV_163(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/6/2015 5:52:09 PM
Data Processed	: 5/6/2015 6:18:26 PM



<Chromatogram>

C:\...\naphthyltrifate\nitrogen nu\CHIRAL-DW_IV_163(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd



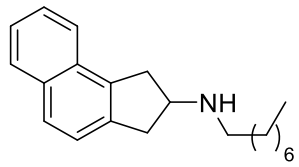
1 PDA Multi 1/254nm 4nm

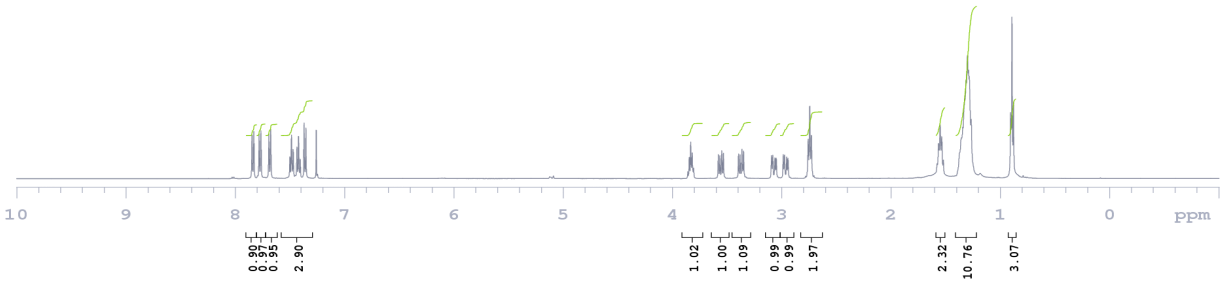
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.141	14672	612	0.285	0.373
2	20.612	5128905	163654	99.715	99.627
Total		5143578	164266	100.000	100.000

Sample Name	Pulse sequence **PROTON**	Temperature **25**	Study owner **derickrw**
Date collected **2015-05-14**	Solvent **cdcl3**	Operator **derickrw**	Printed from **md.chem.lsa.umich.edu-vnmrs400**

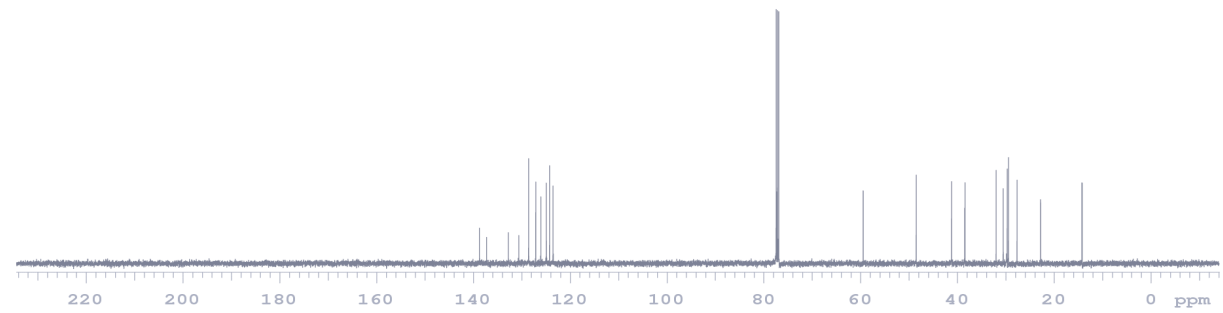




Data file /misc/Tellurium/derickrw/vnmrsys/data/DW_IV_170_1_c1w1_HNMR.fid

Plot date 2015-05-14

Sample Name	Pulse sequence **CARBON**	Temperature **25**	Study owner **derickrw**
Date collected **2015-05-14**	Solvent **cdcl3**	Operator **derickrw**	Printed from **md.chem.lsa.umich.edu-vnmrs400**



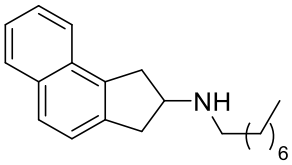
Data file /misc/Tellurium/derickrw/vnmrsys/data/DW_IV_170_1_c1w1_CNMR.fid

Plot date 2015-05-14

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

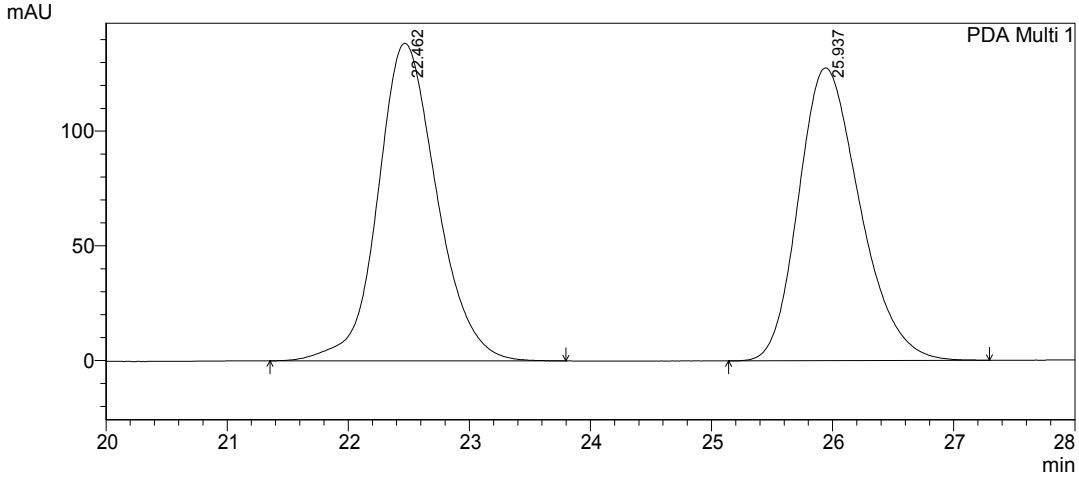
C:\...\DERKPHOS\naphthyltrifate\nitrogen nu\RAC-DW_IV_95(2)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH_2.lcd

Acquired by	: Admin
Sample Name	: RAC-DW_IV_95(2)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH_2
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-DW_IV_95(2)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH_2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/14/2015 3:04:49 PM
Data Processed	: 5/14/2015 3:34:12 PM



<Chromatogram>

C:\...\naphthyltrifate\nitrogen nu\RAC-DW_IV_95(2)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH_2.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

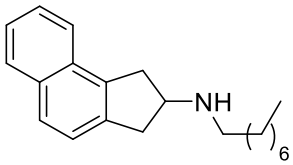
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.462	4690637	138515	51.032	52.033
2	25.937	4500947	127693	48.968	47.967
Total		9191584	266208	100.000	100.000

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

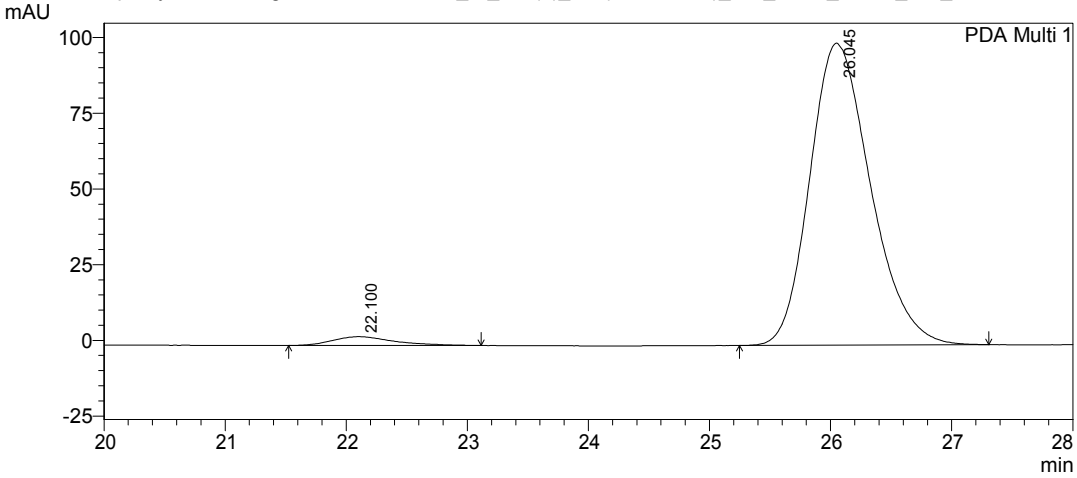
C:\...\naphthyltrifate\nitrogen nu\CHIRAL-DW_IV_170(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-DW_IV_170(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-DW_IV_170(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/14/2015 2:35:24 PM
Data Processed	: 5/14/2015 3:03:44 PM



<Chromatogram>

C:\...\naphthyltrifate\nitrogen nu\CHIRAL-DW_IV_170(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_ADH.lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

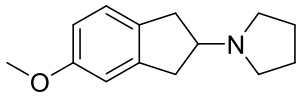
Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.100	101031	2898	2.826	2.824
2	26.045	3474302	99739	97.174	97.176
Total		3575333	102637	100.000	100.000

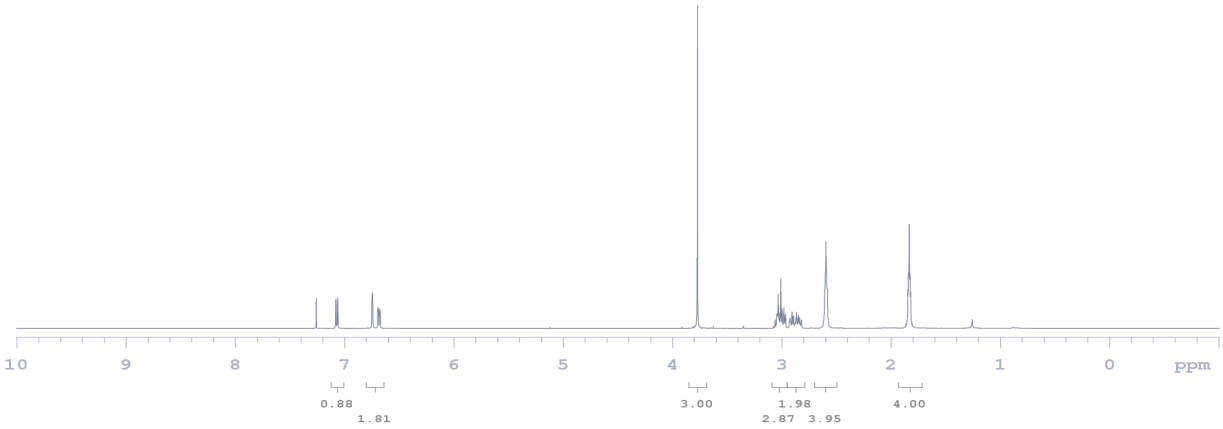
Sample Name
Date collected **2015-04-21**

Pulse sequence **PROTON**
Solvent **cdcl3**

Temperature **25**
Operator **derickrw**

Study owner **derickrw**
Printed from **zn.chem.lsa.umich.edu-vnmrs400**





Data file /misc/Tellurium/derickrw/vnmrsys/data/DW_IV_126_1_HNMR.fid

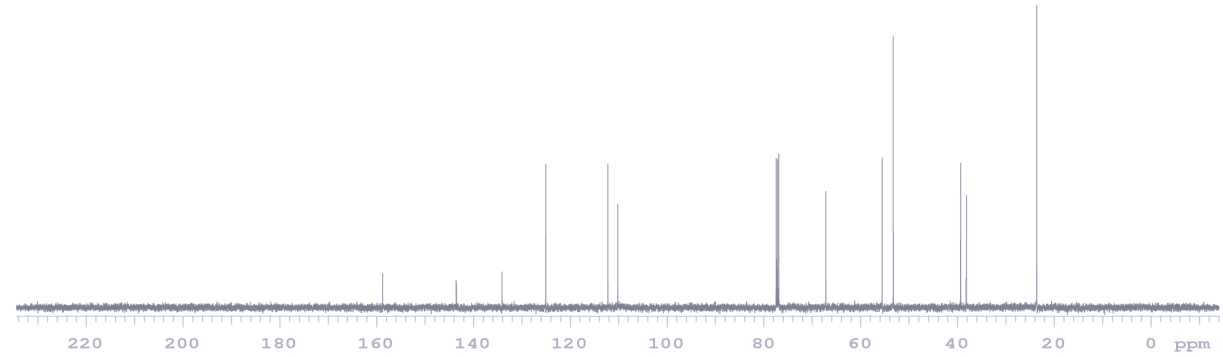
Plot date 2015-04-21

Sample Name
Date collected **2015-04-21**

Pulse sequence **CARBON**
Solvent **cdcl3**

Temperature **25**
Operator **derickrw**

Study owner **derickrw**
Printed from **zn.chem.lsa.umich.edu-vnmrs400**



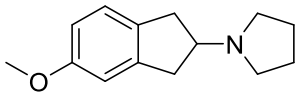
Data file /misc/Tellurium/derickrw/vnmrsys/data/DW_IV_126_1_CNMR.fid

Plot date 2015-05-04

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

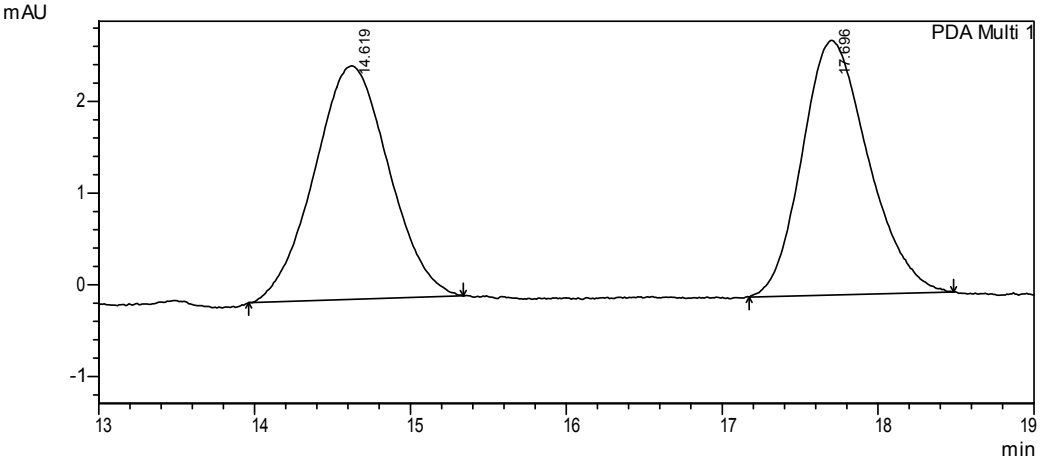
C:\...\Data\DERKPHOS\p-methoxytriflate\nitrogen nu\RAC-DW_IV_69(1)_hex(+0.1%DEA)_IPA_99to1_ADH_4.lcd

Acquired by	: Admin
Sample Name	: RAC-DW_IV_69(1)_hex(+0.1%DEA)_IPA_99to1_ADH_4
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-DW_IV_69(1)_hex(+0.1%DEA)_IPA_99to1_ADH_4.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/16/2015 5:45:09 PM
Data Processed	: 3/16/2015 6:10:32 PM



<Chromatogram>

C:\...\Data\DERKPHOS\p-methoxytriflate\nitrogen nu\RAC-DW_IV_69(1)_hex(+0.1%DEA)_IPA_99to1_ADH_4.lcd



1 PDA Multi 1/275nm 4nm

PeakTable

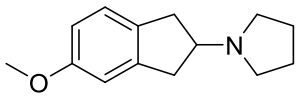
PDA Ch1 275nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.619	84929	2551	50.892	47.888
2	17.696	81953	2776	49.108	52.112
Total		166882	5327	100.000	100.000

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

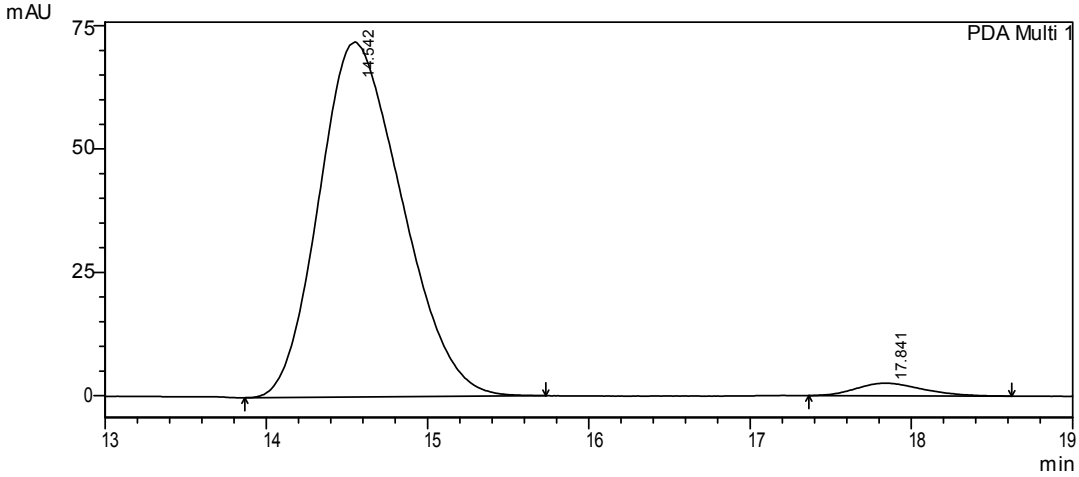
C:\...\p-methoxytriflate\nitrogen nu\CHIRAL-DW_IV_156(2)_hex(0.1%DEA)_IPA_99to1_0.5mL_min-ADH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-DW_IV_156(2)_hex(0.1%DEA)_IPA_99to1_0.5mL_min-ADH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-DW_IV_156(2)_hex(0.1%DEA)_IPA_99to1_0.5mL_min-ADH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/1/2015 5:14:30 PM
Data Processed	: 5/1/2015 5:38:32 PM



<Chromatogram>

C:\...\p-methoxytriflate\nitrogen nu\CHIRAL-DW_IV_156(2)_hex(0.1%DEA)_IPA_99to1_0.5mL_min-ADH.lcd



1 PDA Multi 1/275nm 4nm

PeakTable

PDA Ch1 275nm 4nm

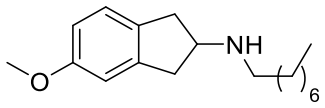
Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.542	2572769	71970	97.235	96.559
2	17.841	73150	2565	2.765	3.441
Total		2645919	74535	100.000	100.000

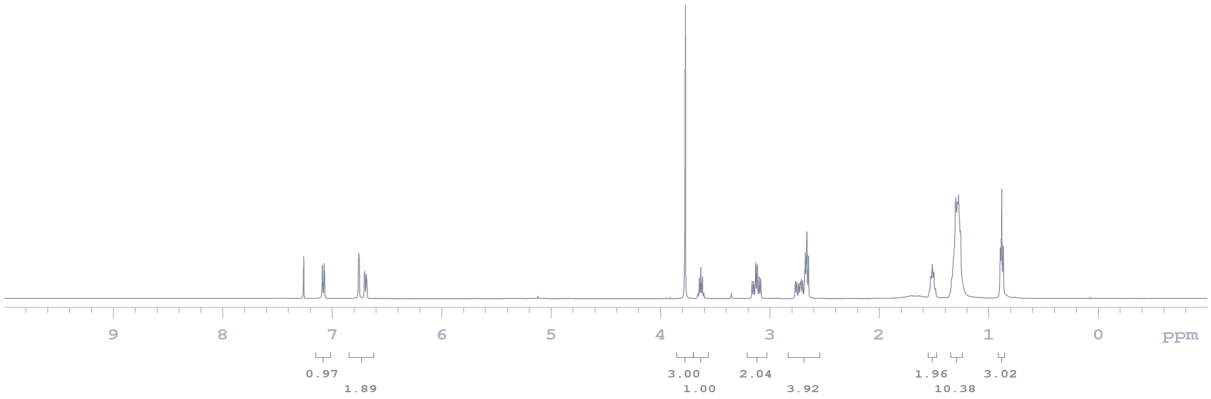
Sample Name
Date collected **2015-05-13**

Pulse sequence **PROTON**
Solvent **cdcl3**

Temperature **25**
Operator **derickrw**

Study owner **derickrw**
Printed from **dy.chem.lsa.umich.edu-vnmrs500**





Data file /misc/i500/derickrw/vnmrsys/data/DW_IV_169_2_c1w1.fid

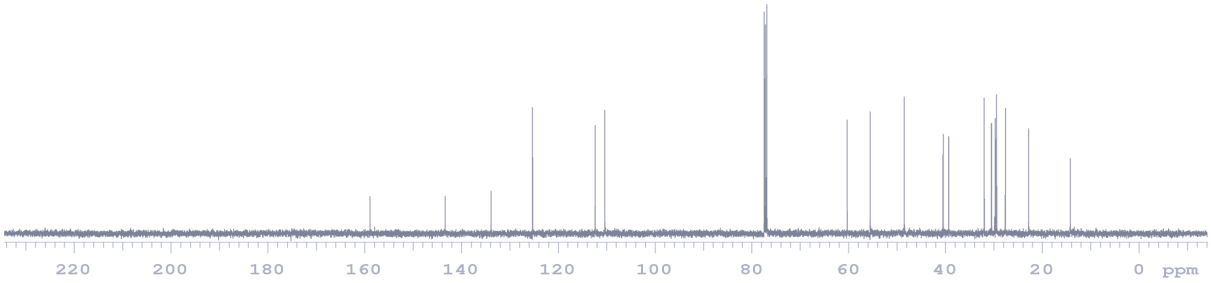
Plot date 2015-05-13

Sample Name
Date collected **2015-05-14**

Pulse sequence **CARBON**
Solvent **cdcl3**

Temperature **25**
Operator **derickrw**

Study owner **derickrw**
Printed from **dy.chem.lsa.umich.edu-vnmrs500**



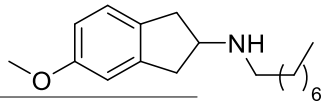
Data file /misc/Tellurium/derickrw/vnmrsys/data/DW_IV_169_1_c1w1_CNMR.fid

Plot date 2015-05-14

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

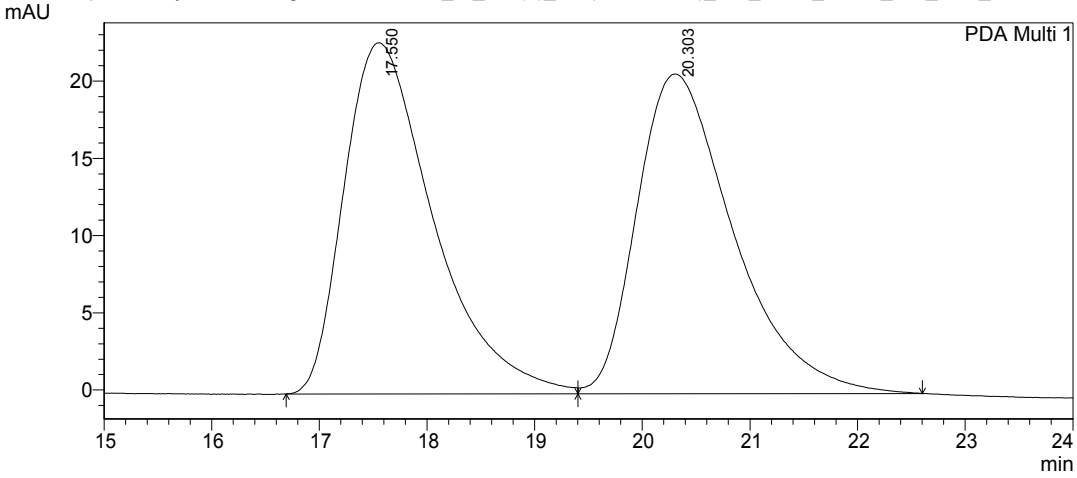
C:\...\p-methoxytriflate\nitrogen nu\RAC-DW_IV_126(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_OJH_2.lcd

Acquired by	: Admin
Sample Name	: RAC-DW_IV_126(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_OJH_2
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-DW_IV_126(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_OJH_2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/13/2015 12:40:06 PM
Data Processed	: 5/13/2015 1:05:09 PM



<Chromatogram>

C:\...\p-methoxytriflate\nitrogen nu\RAC-DW_IV_126(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_OJH_2.lcd



1 PDA Multi 1/275nm 4nm

PeakTable

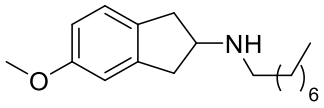
PDA Ch1 275nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.550	1312540	22740	50.409	52.334
2	20.303	1291257	20712	49.591	47.666
Total		2603797	43452	100.000	100.000

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

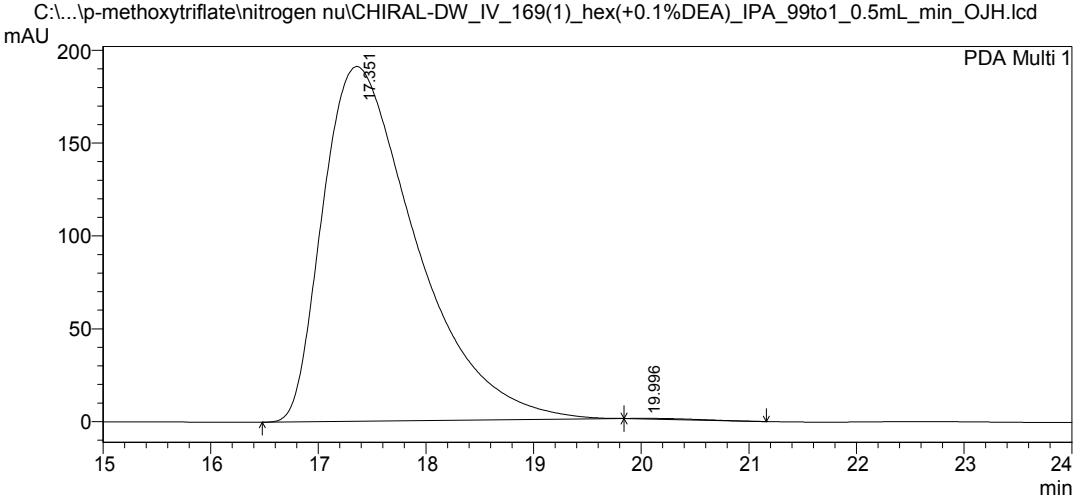
C:\...\p-methoxytriflate\nitrogen nu\CHIRAL-DW_IV_169(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_OJH.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-DW_IV_169(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_OJH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-DW_IV_169(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_OJH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/13/2015 1:06:25 PM
Data Processed	: 5/13/2015 1:33:16 PM



<Chromatogram>

C:\...\p-methoxytriflate\nitrogen nu\CHIRAL-DW_IV_169(1)_hex(+0.1%DEA)_IPA_99to1_0.5mL_min_OJH.lcd



1 PDA Multi 1/275nm 4nm

PeakTable

PDA Ch1 275nm 4nm

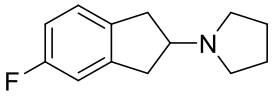
Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.351	11545332	191171	99.839	99.869
2	19.996	18625	251	0.161	0.131
Total		11563956	191423	100.000	100.000

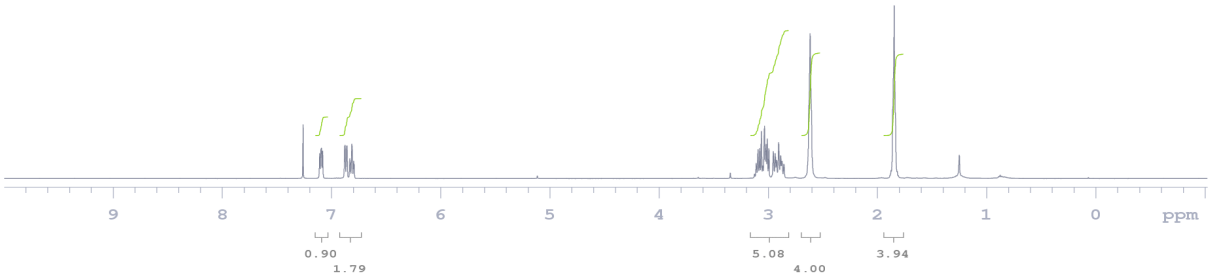
Sample Name
Date collected **2015-05-04**

Pulse sequence **PROTON**
Solvent **cdcl3**

Temperature **25**
Operator **derickrw**

Study owner **derickrw**
Printed from **kr.chem.lsa.umich.edu-vnmrs500**





Data file /misc/i500/derickrw/vnmrsys/data/DW_IV_157_1_c1.fid

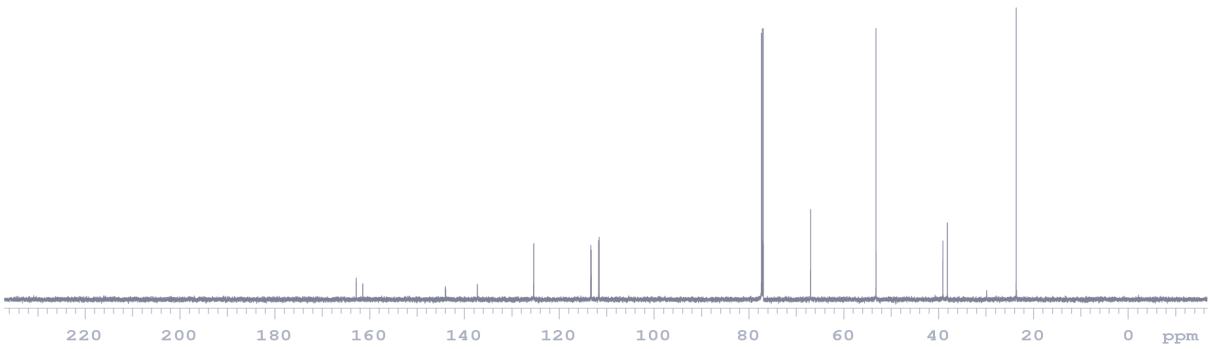
Plot date 2015-05-04

Sample Name
Date collected **2015-05-04**

Pulse sequence **CARBON**
Solvent **cdcl3**

Temperature **25**
Operator **derickrw**

Study owner **derickrw**
Printed from **zn.chem.lsa.umich.edu-vnmrs400**



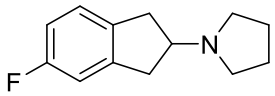
Data file /misc/Ytterbium/derickrw/vnmrsys/data/DW_IV_157_1_CNMR.fid

Plot date 2015-05-04

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

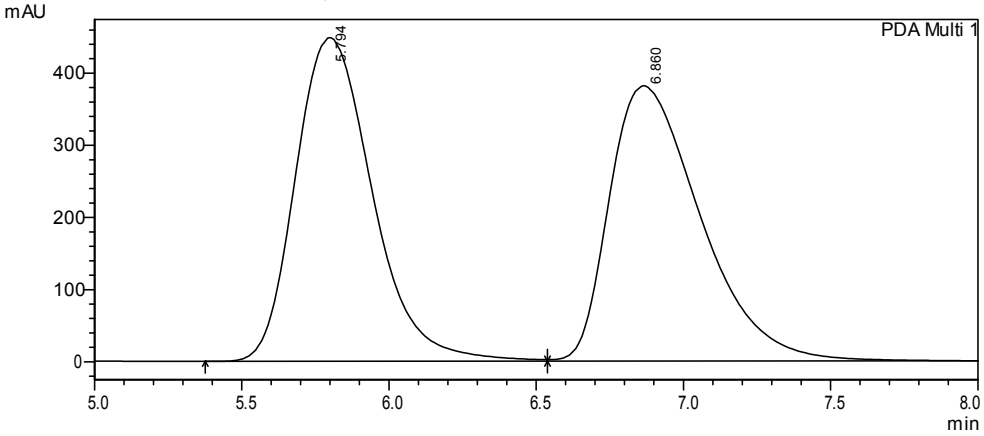
C:\...\DERKPHOS\p-flourotriflate\nitrogen nu\RAC-DW_IV_102(1)_hex(+0.1%DEA)_IPA_99to1_0.5mLmin_ODH.lcd

Acquired by	: Admin
Sample Name	: RAC-DW_IV_102(1)_hex(+0.1%DEA)_IPA_99to1_0.5mLmin_ODH
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RAC-DW_IV_102(1)_hex(+0.1%DEA)_IPA_99to1_0.5mLmin_ODH.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 3/26/2015 1:26:10 PM
Data Processed	: 3/26/2015 1:39:48 PM



<Chromatogram>

C:\...\DERKPHOS\p-flourotriflate\nitrogen nu\RAC-DW_IV_102(1)_hex(+0.1%DEA)_IPA_99to1_0.5mLmin_ODH.lcd



1 PDA Multi 1/275nm 4nm

PeakTable

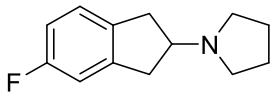
PDA Ch1 275nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.794	8156690	448741	49.796	54.048
2	6.860	8223670	381519	50.204	45.952
Total		16380360	830261	100.000	100.000

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

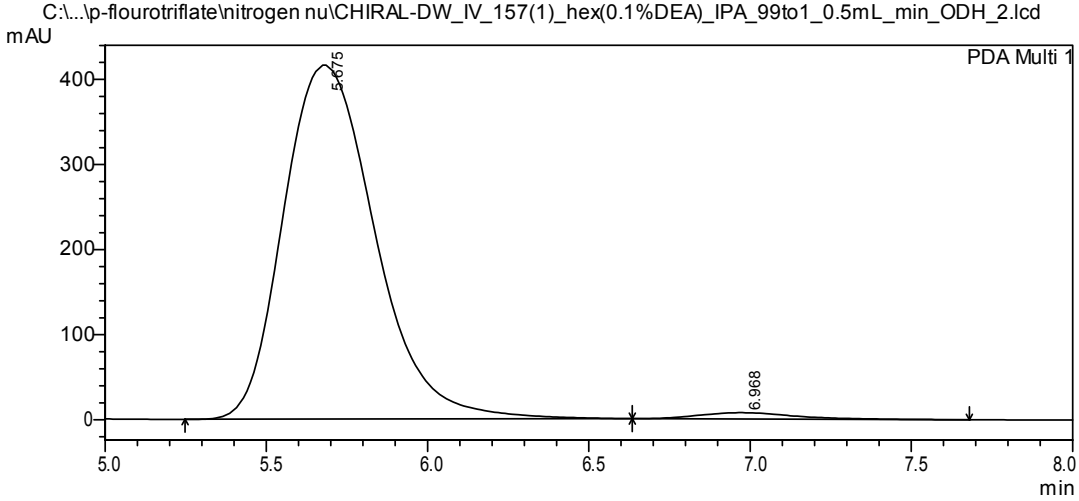
C:\...\p-flourotriflate\nitrogen nu\CHIRAL-DW_IV_157(1)_hex(0.1%DEA)_IPA_99to1_0.5mL_min_ODH_2.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-DW_IV_157(1)_hex(0.1%DEA)_IPA_99to1_0.5mL_min_ODH_2
Sample ID	:
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-DW_IV_157(1)_hex(0.1%DEA)_IPA_99to1_0.5mL_min_ODH_2.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/3/2015 4:06:49 PM
Data Processed	: 5/3/2015 4:21:06 PM



<Chromatogram>

C:\...\p-flourotriflate\nitrogen nu\CHIRAL-DW_IV_157(1)_hex(0.1%DEA)_IPA_99to1_0.5mL_min_ODH_2.lcd



1 PDA Multi 1/275nm 4nm

PeakTable

PDA Ch1 275nm 4nm

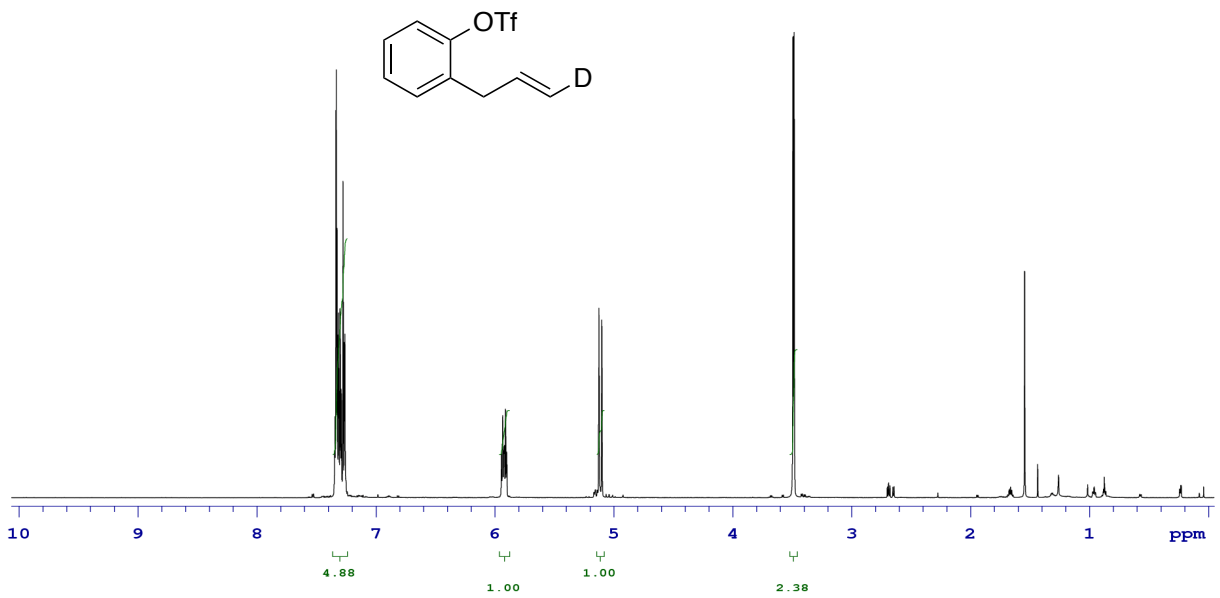
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.675	8224720	416024	98.105	98.219
2	6.968	158875	7544	1.895	1.781
Total		8383595	423569	100.000	100.000

Sample Name
Date collected **2015-07-05**

Pulse sequence **PROTON**
Solvent **cdcl3**

Temperature **25**
Operator **jhutt**

Study owner **jhutt**
Printed from **md.chem.lsa.umich.edu-vnmrs400**



Data file /misc/Ytterbium/jhutt/vnmrsys/data/JTH-III-47-CDCl3.fid

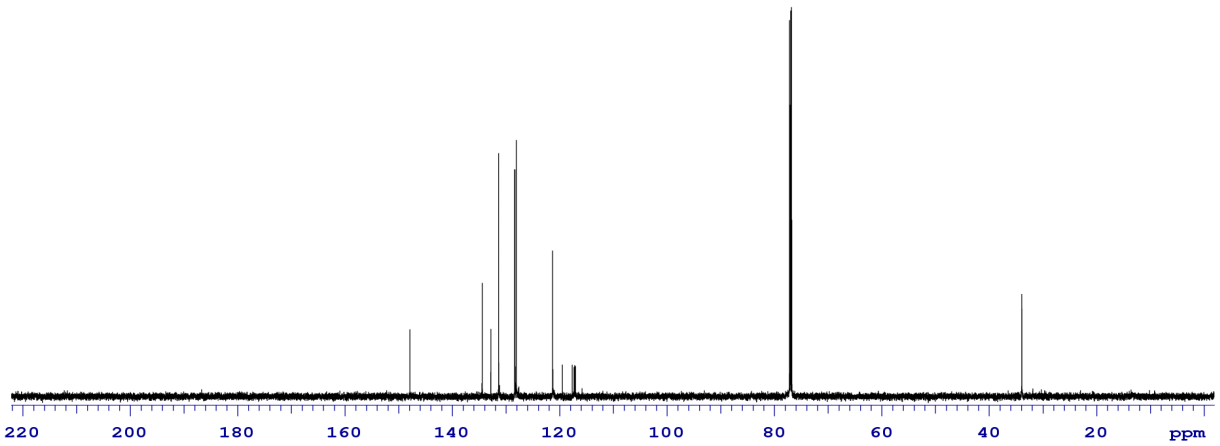
Plot date 2015-07-08

Sample Name
Date collected **2015-07-01**

Pulse sequence **CARBON**
Solvent **cdcl3**

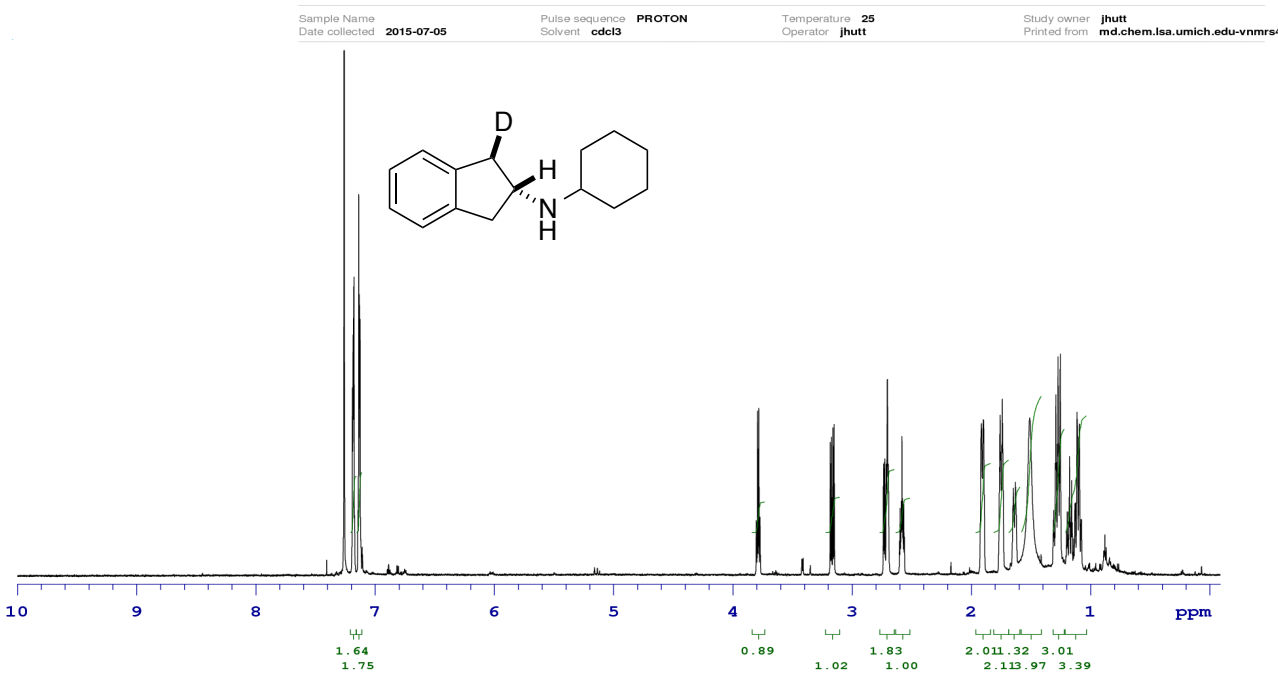
Temperature **25**
Operator **jhutt**

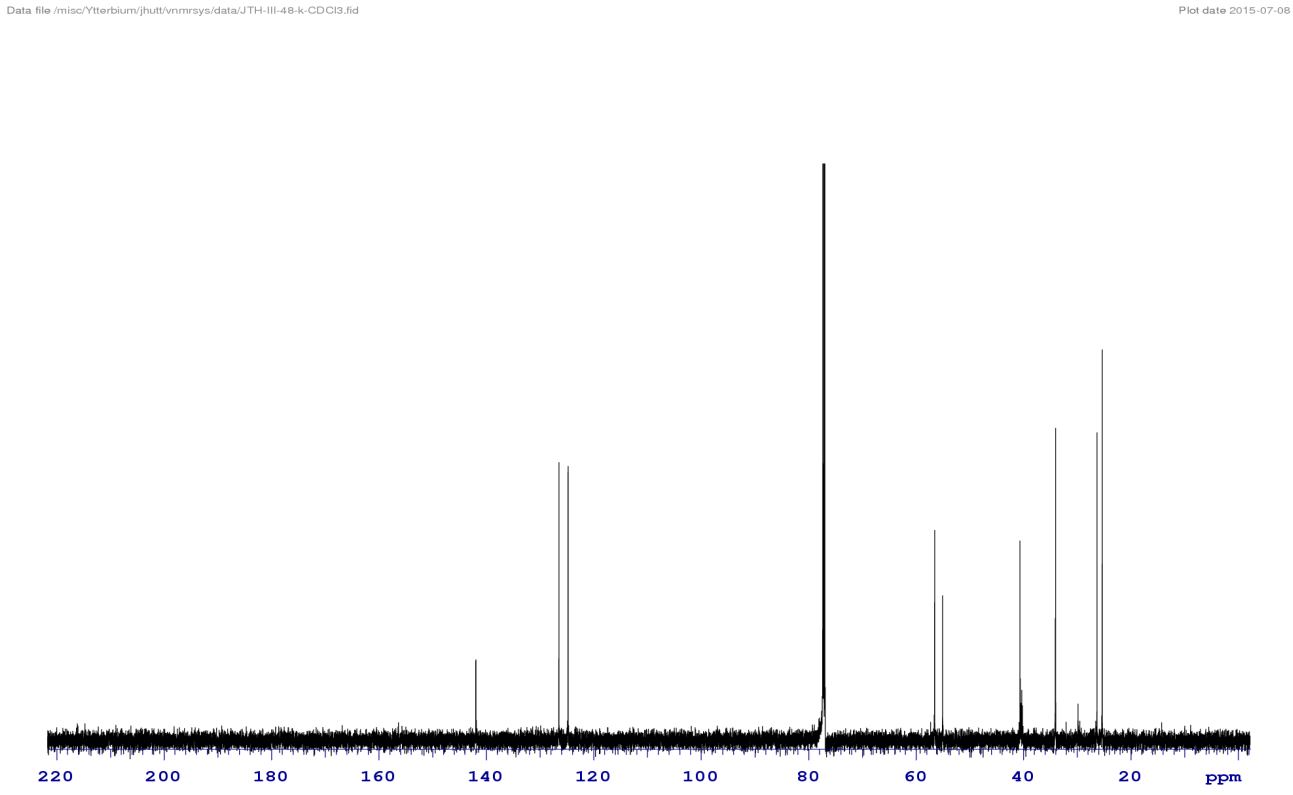
Study owner **jhutt**
Printed from **md.chem.lsa.umich.edu-vnmrs400**



Data file /misc/Ytterbium/jhutt/vnmrsys/data/JTH-III-47-C13-CDCl3.fid

Plot date 2015-07-09


Sample Name
Date collected 2015-07-05
Pulse sequence PROTON
Solvent cdcl3
Temperature 25
Operator jhutt
Study owner jhutt
Printed from md.chem.lsa.umich.edu-vnmrs400
D
H
N
H
10
9
8
7
6
5
4
3
2
1
ppm
1.64
1.75
0.89
1.02
1.83
1.00
2.01
1.32
2.11
3.97
3.01
3.39

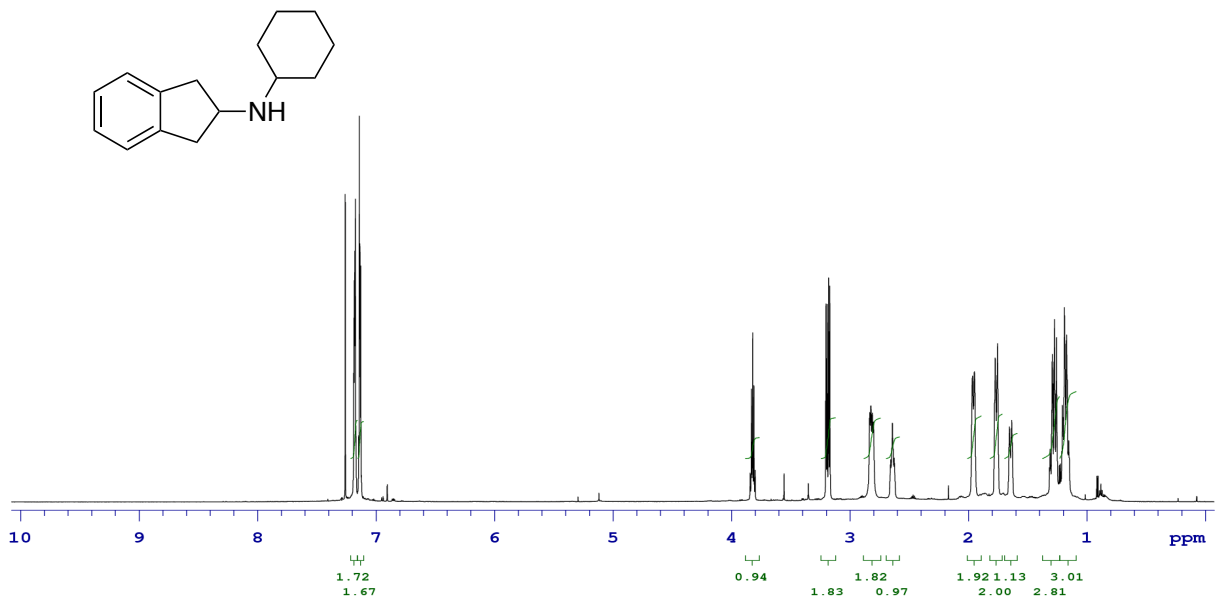

Data file /misc/Ytterbium/jhutt/vnmrsys/data/JTH-III-48-k-CDCl3.fid
Plot date 2015-07-08
220
200
180
160
140
120
100
80
60
40
20
ppm

Sample Name
Date collected **2015-07-06**

Pulse sequence **PROTON**
Solvent **cdcl3**

Temperature **25**
Operator **jhutt**

Study owner **jhutt**
Printed from **md.chem.lsa.umich.edu-vnmrs400**



Data file /misc/Ytterbium/jhutt/vnmrsys/data/DRW-IV-49-CDCl3.fid

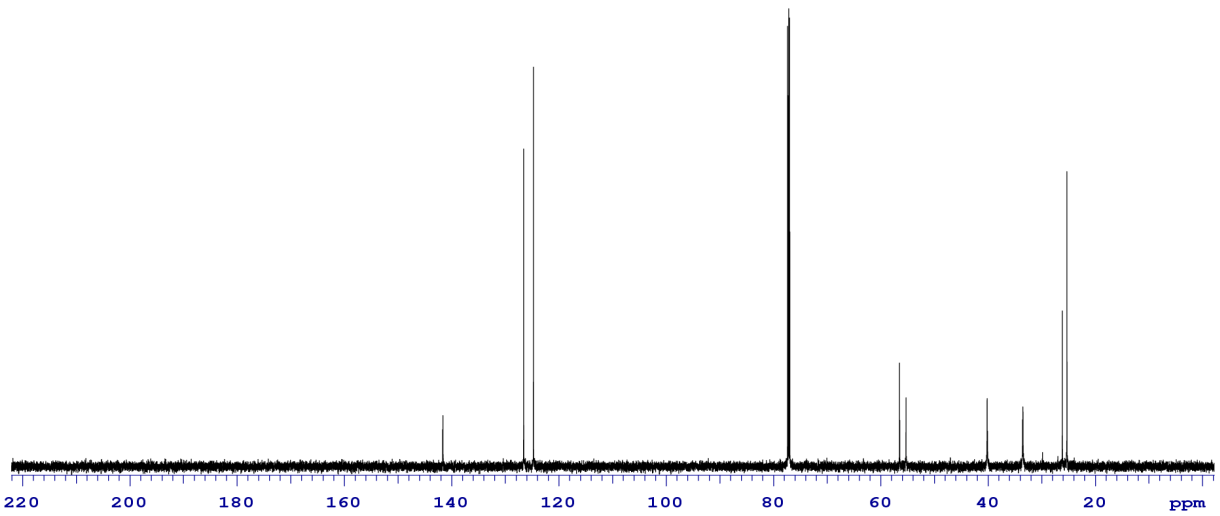
Plot date 2015-07-08

Sample Name
Date collected **2015-07-06**

Pulse sequence **CARBON**
Solvent **cdcl3**

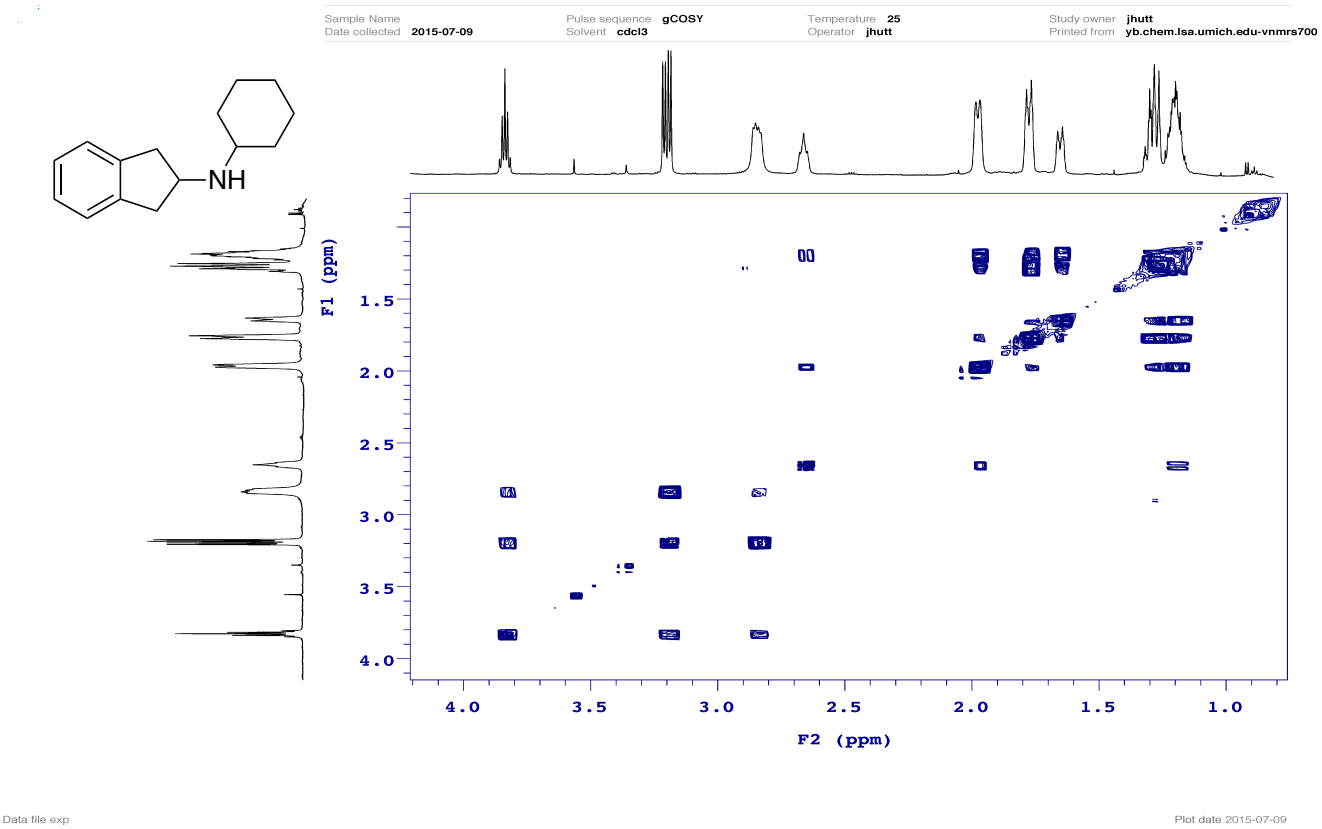
Temperature **25**
Operator **jhutt**

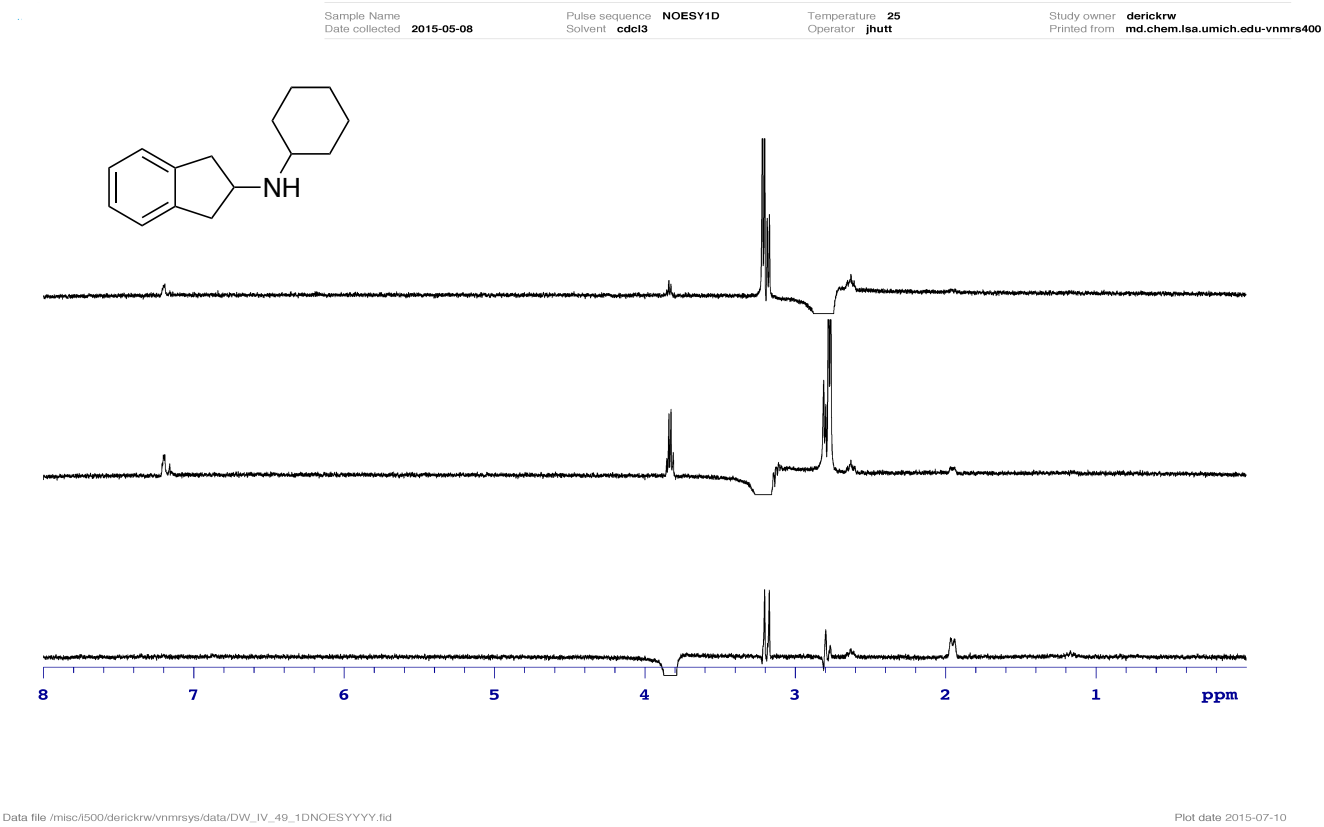
Study owner **jhutt**
Printed from **md.chem.lsa.umich.edu-vnmrs400**



Data file /misc/Ytterbium/jhutt/vnmrsys/data/DRW-IV-49-C13-CDCl3.fid

Plot date 2015-07-09


Sample Name
Date collected 2015-07-09
Pulse sequence gCOSY
Solvent cdcl3
Temperature 25
Operator jhutt
Study owner jhutt
Printed from yb.chem.lsa.umich.edu-vnmrs700
NH
F1 (ppm)
1.5
2.0
2.5
3.0
3.5
4.0
4.0
3.5
3.0
2.5
2.0
1.5
1.0
F2 (ppm)
Data file exp
Plot date 2015-07-09


Sample Name
Date collected 2015-05-08
Pulse sequence NOESY1D
Solvent cdcl3
Temperature 25
Operator jhutt
Study owner derickrw
Printed from md.chem.lsa.umich.edu-vnmrs400
NH
8
7
6
5
4
3
2
1
ppm
Data file /misc/l500/derickrw/vnmrsys/data/DW_IV_49_1DNOESYYYY.fid
Plot date 2015-07-10