Desymmetrization of *meso*-2,5-Diallylpyrrolidinyl Ureas via Asymmetric Pd-Catalyzed Carboamination Reactions. Stereocontrolled Synthesis of Bicyclic Ureas.

Nicholas R. Babij, and John P. Wolfe*

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

Supporting Information

Experimental procedures and characterization data for new compounds in Tables 1–2, Schemes 2–3, and Equation 1.

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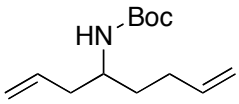
General: All reactions were carried out under a nitrogen atmosphere in flame-dried glassware unless otherwise noted. Tris(dibenzylidene)acetone dipalladium, tri(2-furyl)phosphine, and (*S*)-Siphos-PE were purchased from Strem Chemical Co. and used without purification. Tricyclohexylphosphonium tetrafluoroborate was purchased from Acros Chemical Co. and used without further purification. 2-Di-*tert*-butylphosphino-3,4,5,6-tetramethyl-2',4',6'-triisopropyl-1,1'-biphenyl was purchased from Sigma-Aldrich and used without further purification. All other reagents were obtained from commercial sources and were used as obtained unless otherwise noted. NaO*t*Bu and CuCl were stored in the glove box and removed prior to use. BF_3OEt_2 and $POCl_3$ were purified by distillation under N_2 prior to use. (*Z*)-1-bromobutene[1] was prepared according to a slight modification of a literature procedure; the preparation was conducted at rt instead of using microwave heating. (*Z*)-1-bromohexene,[2] and (*E*)-1-bromohexene[2] were prepared according to published procedures. Toluene, THF, diethyl ether and dichloromethane

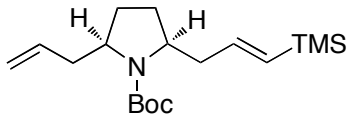
were purified using a GlassContour solvent purification system. Yields refer to isolated yields of compounds estimated to be ≥95% pure as determined by ^{1}H NMR analysis unless otherwise noted. The yields reported in the supporting information describe the result of a single experiment, whereas yields reported in Tables 1–2, Scheme 2, and Equation 1 are average yields of two or more experiments. Thus, the yields reported in the supporting information may differ from those shown in Tables 1–2, Scheme 2, and Equation 1. Structural and stereochemical assignments were made on the basis of 2-D COSY, and NOESY experiments. Ratios of diastereomers were determined by ^{1}H NMR analysis. The reported optical rotation values refer to measurements taken of the isolated mixtures of diastereomers upon which chemical yields were based. Ratios of enantiomers were determined by HPLC analysis. Although diastereomers were not easily separable by chromatography, for most examples (with the exception of **8i** and **8j**) it was possible to separate small amounts of the pure (>20:1 dr) major diastereomer for chiral HPLC analysis.

Preparation and Characterization of *meso*-*N*-Aryl-2,5-Diallylpyrrolidine-1-Carboxamide Substrates



(±)-*tert*-Butyl octa-1,7-dien-4-ylcarbamate (S1). The title compound was prepared by modifying a procedure published by Veenstra.[3] A flame-dried flask was cooled under a stream of N_2, charged with dichloromethane (60 mL) and cooled to 0 ^{O}C. Pent-4-enal (2.96 mL, 30 mmol), allyltrimethylsilane (4.77 mL, 30 mmol) and *tert*-butyl carbamate (3.5 g, 30 mmol) were added to the flask and the resulting solution was stirred for 15 min at 0 ^{O}C. Distilled BF_3OEt_2 (2.3 mL, 18 mmol) was added and the reaction mixture was stirred for 30 min at 0 ^{O}C. The mixture was gradually warmed to rt and stirred for 30 min. The reaction was then quenched with saturated aqueous $NaHCO_3$ (20 mL) and stirred for 5 min at rt. The mixture was transferred to a separatory funnel and the layers were separated. The organic layer was washed with saturated aqueous $NaHCO_3$ (20 mL) and then the combined aqueous layers were extracted with dichloromethane (15 mL). The combined organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel to afford 3.8 g (56%) of the title compound as a clear colorless oil. This compound was found to exist as a mixture of rotamers as judged by ^{1}H and ^{13}C NMR analysis; data are for the mixture. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.84–5.73 (m, 2 H), 5.10–4.95

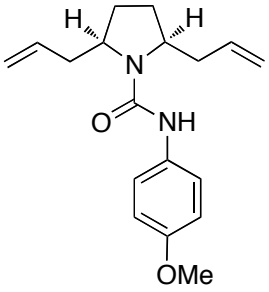
(m, 4 H), 4.33 (d, br, *J* = 7.5 Hz, 1 H), 3.66 (d, br, *J* = 4.5 Hz, 1 H), 2.26–2.07 (m, 4 H), 1.60–1.55 (m, 1 H), 1.48–1.40 (m, 1 H), 1.43 (s, 9 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 155.5, 138.0, 134.4, 117.7, 114.9, 79.0, 49.6, 39.5, 33.9, 30.2, 28.4; IR (film) 3337,1684 cm^{-1}. MS (ESI) 248.1621 (248.1621 calcd for $C_{13}H_{23}NO_2$, M + Na^+).



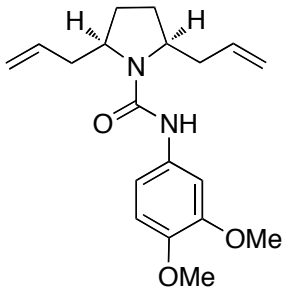
(±)-(*E*,2*R,5*S**)-*tert*-Butyl 2-allyl-5-[3-(trimethylsilyl)allyl]pyrrolidine-1-carboxylate (S2).** A flame-dried Schlenk flask was cooled under a stream of N_2 and charged with $Pd_2(dba)_3$ (81 mg, 0.089 mmol), tri(2-furyl)phosphine (82 mg, 0.36 mmol) and NaO*t*Bu (853 mg, 8.9 mmol). The flask was purged with N_2, then a solution of **S1** (1.0 g, 4.4 mmol) in freshly distilled xylenes (22.2 mL) was added via syringe and the resulting mixture was stirred at rt for 5 min. (*E*)-(2-bromovinyl)trimethylsilane (1.36 mL, 8.9 mmol) was added and the flask was heated to 137 °C and stirred overnight (ca. 14 h). The mixture was cooled to room temperature and saturated aqueous NH_4Cl (10 mL) and ethyl acetate (10 mL) were added. The layers were separated, the organic layer was filtered through a plug of silica gel, and the silica gel was washed with ethyl acetate (20 mL). The filtrate was dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel to afford 1.11 g (77%) of the title compound as a dark red-brown oil. This compound was found to exist as a mixture of rotamers as judged by ^{1}H and ^{13}C NMR analysis; data are for the mixture. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.98–5.92 (m, 1 H), 5.78–5.70 (m, 1 H), 5.68 (d, *J* = 18.5 Hz, 1 H), 5.06–5.01 (m, 2 H), 3.92–3.68 (m, 2 H), 2.64–2.41 (m, 2 H), 2.34 (dt, *J* = 8.0, 13.0 Hz, 1 H), 2.09 (dt, *J* = 8.0, 13.0 Hz, 1 H), 1.87–1.82 (m, 2 H), 1.68–1.64 (m, 2 H), 1.46 (s, 9 H), 0.03 (s, 9 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.7, 143.2, 135.4, 132.9, 116.8, 79.0, 58.0, 57.9, 42.1, 42.0, 40.0, 39.8, 28.5, –1.2; IR (film) 1692 cm^{-1}. MS (ESI) 346.2174 (346.2173 calcd for $C_{18}H_{33}NO_2Si$, M + Na^+).

General Procedure for Synthesis of *meso*-*N*-Aryl-2,5-Diallylpyrrolidine-1-Carboxamide Substrates 7. A round-bottom flask equipped with a stirbar was charged with **S2** (1.0 equiv) and dichloromethane (0.2 M). Trifluoroacetic acid (1.0 M) was added to the flask and the mixture was heated to reflux and stirred overnight. The solution was cooled to rt, diluted with water, basified with NH_4OH to pH > 12, and transferred to a separatory funnel. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3 x 10 mL). The combined organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude

product was dissolved in dichloromethane (0.2 M) and the appropriate isocyanate (1.1 equiv) was added. The reaction mixture was stirred at rt until starting material had been completely consumed as judged by TLC analysis (ca. 1 h). The crude reaction mixture was concentrated *in vacuo*, and purified by flash chromatography on silica gel.

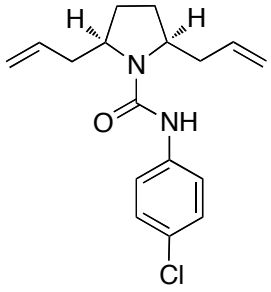


(2*S*,5*R*)-2,5-Diallyl-*N*-(4-methoxyphenyl)pyrrolidine-1-carboxamide (7a). The title compound was prepared from **S2** (2.13 g, 6.6 mmol) and 4-methoxyphenyl isocyanate (940 µL, 7.3 mmol) in two steps via the general procedure described above. This procedure afforded 1.2 g (61%) of the title compound as a white solid: mp = 63–65 °C. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.25 (d, *J* = 8.4 Hz, 2 H), 6.82 (d, *J* = 9.1 Hz, 2 H), 6.33 (s, 1H), 5.91–5.85 (m, 2 H), 5.20–5.15 (m, 4 H), 3.99–3.96 (m, 2 H), 3.77 (s, 3 H), 2.55 (dt, *J* = 14.0, 7.0 Hz, 2 H), 2.24 (dt, *J* = 7.0, 14.0 Hz, 2 H), 2.02–1.97 (m, 2 H), 1.78–1.74 (m, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 155.5, 155.2, 135.2, 132.3, 121.4, 118.0, 114.1, 58.8, 55.5, 40.2, 29.5; IR (film) 3311, 1635 cm^{-1}. MS (ESI) 301.1917 (301.1911 calcd for $C_{18}H_{24}N_2O_2$, M + H$^+$).

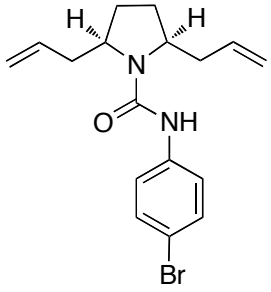


(2*S*,5*R*)-2,5-Diallyl-*N*-(3,4-dimethoxyphenyl)pyrrolidine-1-carboxamide (7b). The title compound was prepared from **S2** (965 mg, 2.98 mmol) and 3,4-dimethoxyphenyl isocyanate (488 µL, 3.3 mmol) in two steps via the general procedure described above. This procedure afforded 542 mg (55%) of the title compound as a tan solid: mp = 112–114 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27 (d, *J* = 2.1 Hz, 1 H), 6.77 (d, *J* = 8.4 Hz, 1 H), 6.62 (dd, *J* = 2.8, 8.4 Hz, 1 H), 6.36 (s, 1H), 5.91–5.86 (m, 2 H), 5.21–5.16 (m, 4 H), 4.01–3.97 (m, 2 H), 3.88 (s, 3 H), 3.84 (s, 3 H), 2.57 (dt, *J* = 6.3,13.3 Hz, 2 H), 2.25 (dt, *J* = 7.7, 13.3 Hz, 2 H), 2.03–1.99 (m, 2 H),

1.79–1.75 (m, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 155.1, 149.1, 144.8, 135.2, 133.0, 118.1, 111.4, 110.9, 104.7, 58.7, 56.2, 55.9, 40.2, 29.5; IR (film) 3327, 1635 cm^{-1}. MS (ESI) 331.2018 (331.2016 calcd for $C_{19}H_{26}N_2O_3$, M + H^+).

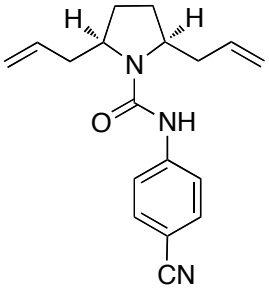


(2*S*,5*R*)-2,5-Diallyl-*N*-(4-chlorophenyl)pyrrolidine-1-carboxamide (7c). The title compound was prepared from **S2** (1.05 g, 3.2 mmol) and 4-chlorophenyl isocyanate (541 mg, 3.5 mmol) in two steps via the general procedure described above. This procedure afforded 574 mg (58%) of the title compound as a white solid: mp = 91–93 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 (d, *J* = 9.0 Hz, 2 H), 7.21 (d, *J* = 9.0 Hz, 2 H), 6.51 (s, 1H), 5.91–5.85 (m, 2 H), 5.22–5.16 (m, 4 H), 4.00–3.95 (m, 2 H), 2.55 (dt, *J* = 14.0, 6.5 Hz, 2 H), 2.25 (dt, *J* = 14.0, 7.5 Hz, 2 H), 2.03–1.97 (m, 2 H), 1.79–1.74 (m, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.5, 137.9, 135.1, 128.7, 127.4, 120.3, 118.3, 58.9, 40.1, 29.6; IR (film) 3318, 1640 cm^{-1}. MS (ESI) 327.1242 (327.1235 calcd for $C_{17}H_{21}ClN_2O$, M + Na^+).

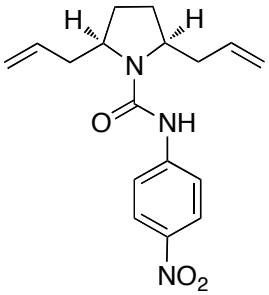


(2*S*,5*R*)-2,5-Diallyl-*N*-(4-bromophenyl)pyrrolidine-1-carboxamide (7d). The title compound was prepared from **S2** (1.2 g, 3.7 mmol) and 4-bromophenyl isocyanate (806 mg, 4.1 mmol) in two steps via the general procedure described above. This procedure afforded 827 mg (64%) of the title compound as a off-white solid: mp = 101–104 °C. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.37 (d, *J* = 7.7 Hz, 2 H), 7.25 (d, *J* = 7.7 Hz, 2 H), 6.49 (s, 1H), 5.91–5.85 (m, 2 H), 5.21–5.17 (m, 4 H), 3.99–3.97 (m, 2 H), 2.55 (dt, *J* = 6.3, 14.0 Hz, 2 H), 2.25 (dt, *J* = 7.0, 14.0 Hz, 2 H), 2.03–1.99 (m, 2 H), 1.80–1.77 (m, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 154.5, 138.4, 135.1, 131.7, 120.7,

118.3, 115.0, 58.9, 40.1, 29.6; IR (film) 3316, 1635 cm^{-1}. MS (ESI) 349.0912 (349.0910 calcd for $C_{17}H_{21}BrN_2O$, M + H^+).



(2*S*,5*R*)-2,5-Diallyl-*N*-(4-cyanophenyl)pyrrolidine-1-carboxamide (7e). The title compound was prepared from **S2** (1.12 g, 3.46 mmol) and 4-cyanophenyl isocyanate (549 mg, 3.81 mmol) in two steps via the general procedure described above. This procedure afforded 613 mg (60%) of the title compound as a off-white solid: mp = 76–79 °C. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.7 Hz, 2 H), 7.25 (d, *J* = 7.7 Hz, 2 H), 6.49 (s, 1H), 5.92–5.86 (m, 2 H), 5.21–5.17 (m, 4 H), 4.02–3.96 (m, 2 H), 2.55 (dt, *J* = 6.3, 14.0 Hz, 2 H), 2.25 (dt, *J* = 7.0, 14.0 Hz, 2 H), 2.03–1.99 (m, 2 H), 1.80–1.77 (m, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 153.9, 143.5, 135.0, 133.1, 119.2, 118.6, 118.6, 105.1, 59.1, 39.9, 29.6; IR (film) 3365, 1652 cm^{-1}. MS (ESI) 296.1756 (296.1757 calcd for $C_{18}H_{21}N_3O$, M + H^+).



(2*S*,5*R*)-2,5-Diallyl-*N*-(4-nitrophenyl)pyrrolidine-1-carboxamide (7f). The title compound was prepared from **S2** (660 mg, 2.04 mmol) and 4-nitrophenyl isocyanate (368 mg, 2.24 mmol) in two steps via the general procedure described above. This procedure afforded 366 mg (57%) of the title compound as a pale-yellow solid: mp = 96–97 °C. ^{1}H NMR (700 MHz, $CDCl_3$) δ 8.15 (d, *J* = 9.1 Hz, 2 H), 7.50 (d, *J* = 9.1 Hz, 2 H), 6.93 (s, 1H), 5.94–5.88 (m, 2 H), 5.25–5.21 (m, 4 H), 4.04–4.01 (m, 2 H), 2.56 (dt, *J* = 7.0, 13.3 Hz, 2 H), 2.29 (dt, *J* = 7.0, 14.0 Hz, 2 H), 2.07–2.03 (m, 2 H), 1.83–1.79 (m, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 153.7, 145.5, 142.2, 135.0, 125.1,

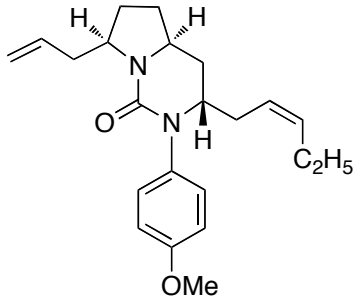
118.8, 117.8, 59.2, 39.9, 29.7; IR (film) 3331, 1652 cm^{-1}. MS (ESI) 316.1656 (316.1656 calcd for $C_{17}H_{21}N_3O_3$, M + H^+).

Preparation and Characterization of Bicyclic Urea Products

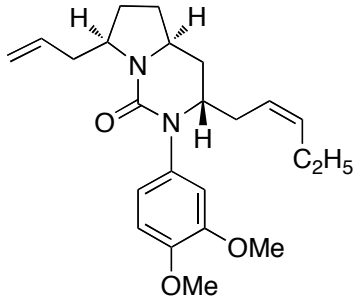
General Procedure for Synthesis of Racemic Bicyclic Ureas (for HPLC assays). A flame-dried Schlenk tube was cooled under vacuum and charged with the appropriate *meso*-*N*-aryl-2,5-diallylpyrrolidine-1-carboxamide substrate (1.0 equiv), $Pd_2(dba)_3$ (0.02 equiv), $PCy_3{\cdot}HBF_4$ (0.08 equiv), and NaO*t*Bu (1.5 equiv). The flask was evacuated and purged with N_2. Toluene (0.2 M) was added via syringe and the resulting mixture was stirred at rt for 2 min. The appropriate aryl or alkenyl bromide (1.5 equiv) was added and the tube was heated to 100 °C and stirred for 2 h. The mixture was cooled to room temperature and saturated aqueous NH_4Cl (5 mL/mmol substrate) and ethyl acetate (5 mL/mmol substrate) were added. The layers were separated, the organic layer was filtered through a plug of silica gel, and the silica gel was washed with ethyl acetate (5 mL/mmol substrate). The filtrate was dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel.

General Procedure for Synthesis of Enantiomerically-Enriched Bicyclic Ureas

A flame-dried Schlenk tube was cooled under vacuum and charged with the appropriate *meso*-*N*-aryl-2,5-diallylpyrrolidine-1-carboxamide substrate (1.0 equiv), $Pd_2(dba)_3$ (0.02 equiv), (*S*)-Siphos-PE (0.08 equiv), and NaO*t*Bu or NaOMe (1.5 equiv). The flask was evacuated and purged with N_2. Toluene (0.2 M) was added via syringe and the resulting mixture was stirred at rt for 2 min. The appropriate aryl or alkenyl bromide (1.5 equiv) was added and the tube was heated to 100 °C. The solution was stirred for 2 h or until the starting material was completely consumed as judged by TLC analysis. The mixture was cooled to room temperature and saturated aqueous NH_4Cl (5 mL/mmol substrate) and ethyl acetate (5 mL/mmol substrate) were added. 6 M HCl was used instead of NH_4Cl to remove aniline side products if column chromatography could not separate the desired product from aniline side products. The layers were separated, the organic layer was filtered through a plug of silica gel, and the silica gel was washed with ethyl acetate (5 mL/mmol substrate). The filtrate was dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel.

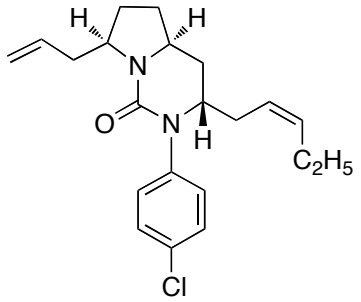


(+)-(Z,3*S*,4a*S*,7*R*)-7-Allyl-2-(4-methoxyphenyl)-3-(pent-2-en-1-yl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8a). The general procedure was employed for the coupling of **7a** (60 mg, 0.2 mmol) and (*Z*)-1-bromobut-1-ene (150 µL, 0.3 mmol, 2.0 M solution in toluene), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 48 mg (68%) of the title compound as a brown oil and as a 7:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ +9.5 (*c* 4.3, CH_2Cl_2). Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.14 (d, *J* = 8.4 Hz, 2 H), 6.88 (d, *J* = 8.4 Hz, 2 H), 5.77–5.71 (m, 1 H), 5.43 (dt, *J* = 7.0, 11.2 Hz, 1 H), 5.12–5.08 (m, 1 H), 5.03 (d, *J* = 17.5 Hz, 1 H), 5.00 (d, *J* = 9.8 Hz, 1 H), 3.99 (dt, *J* = 2.1, 9.1 Hz, 1 H), 3.82–3.78 (m, 1 H), 3.79 (s, 3 H), 3.65 (ddt, *J* = 2.1, 4.9, 11.2 Hz, 1 H), 2.80 (dd, *J* = 5.6, 13.3 Hz, 1 H), 2.29–2.27 (m, 1 H), 2.18–2.15 (m, 2 H), 2.08 (dt, *J* = 8.4, 13.3 Hz, 1 H) 1.99–1.88 (m, 4 H), 1.83 (dd, *J* = 6.3, 12.6 Hz, 1 H) 1.68–1.60 (m, 2 H), 0.89 (t, *J* = 7.7 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 157.6, 154.2, 135.8, 135.1, 134.6, 129.3, 124.2, 116.8, 114.1, 58.3, 57.3, 55.4, 52.7, 37.8, 31.3, 31.0, 30.9, 27.8, 20.7, 14.0; IR (film) 1642 cm^{-1}. MS (ESI) 355.2382 (355.2380 calcd for $C_{22}H_{30}N_2O_2$, M + H^+). The enantiopurity was determined to be 86:14 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 2.5% IPA/Hexanes, 0.75 mL/min, λ 245 nm, RT= 44.2 and 49.1 min).



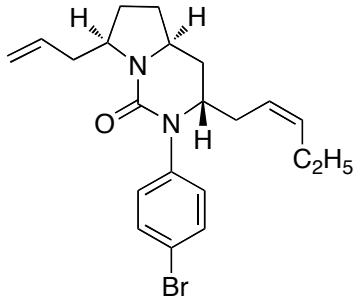
(+)-(*Z*,3*S*,4a*S*,7*R*)-7-Allyl-2-(3,4-dimethoxyphenyl)-3-(pent-2-en-1-yl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8b). The general procedure was employed for the coupling of **7b** (66 mg, 0.2 mmol) and (*Z*)-1-bromobut-1-ene (150 µL, 0.3 mmol, 2.0 M solution in toluene), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 30 mg (39%) of the title compound as a brown oil and as a 7:1 mixture

of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ +7.0 (*c* 2.9, CH_2Cl_2). Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 6.83 (d, *J* = 8.4 Hz, 1 H), 6.78–6.77 (m, 2 H), 5.77–5.71 (m, 1 H), 5.44 (dt, *J* = 7.0, 10.5 Hz, 1 H) 5.13–5.09 (m, 1 H), 5.03 (d, *J* = 16.8 Hz, 1 H), 5.00 (d, *J* = 10.5 Hz, 1 H), 4.01 (dt, *J* = 2.8, 9.1 Hz, 1 H), 3.86 (s, 6 H), 3.85–3.81 (m, 1 H), 3.66 (ddt, *J* = 2.1, 5.6, 11.2 Hz, 1 H), 2.80 (dd, *J* = 5.6, 12.6 Hz, 1 H), 2.30–2.28 (m, 1 H), 2.18–2.15 (m, 2 H), 2.07 (dt, *J* = 8.4, 13.3 Hz, 1 H) 2.00–1.96 (m, 1 H), 1.93–1.87 (m, 3 H), 1.83 (dd, *J* = 7.0, 12.6 Hz, 1 H) 1.69–1.63 (m, 2 H), 0.89 (t, *J* = 7.7 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 154.1, 148.9, 147.2, 135.8, 135.4, 134.7, 124.2, 120.1, 116.8, 112.3, 111.1, 58.5, 57.3, 56.0, 55.9, 52.7, 37.8, 31.3, 31.1, 30.9, 27.7, 20.7, 14.1; IR (film) 1641 cm^{-1}. MS (ESI) 385.2486 (385.2486 calcd for $C_{23}H_{32}N_2O_3$, M + H^+). The enantiopurity was determined to be 82:18 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 205 nm, RT= 20.4 and 23.5 min).

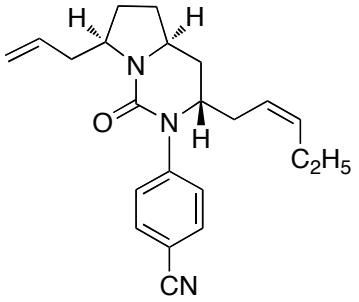


(–)-(Z,3*S*,4a*S*,7*R*)-7-Allyl-2-(4-chlorophenyl)-3-(pent-2-en-1-yl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8c). The general procedure was employed for the coupling of **7c** (305 mg, 1.0 mmol) and (*Z*)-1-bromobut-1-ene (750 µL, 1.5 mmol, 2.0 M solution in toluene), using a catalyst composed of Pd_2dba_3 (18.3 mg, 0.02 mmol), and (*S*)-Siphos-PE (40.4 mg, 0.08 mmol). This procedure afforded 288 mg (80%) of the title compound as a yellow oil: $[\alpha]^{23}_D$ –14.3 (*c* 5.3, CH_2Cl_2). ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.4 Hz, 2 H), 7.19 (d, *J* = 8.4 Hz, 2 H), 5.77–5.71 (m, 1 H), 5.45 (dt, *J* = 7.0, 10.5 Hz, 1 H), 5.12–5.08 (m, 1 H), 5.04 (d, *J* = 16.8 Hz, 1 H), 5.01 (d, *J* = 10.5 Hz, 1 H), 4.01 (dt, *J* = 2.8, 9.1 Hz, 1 H), 3.90 (dt, *J* = 4.2, 10.5 Hz, 1 H), 3.66 (ddt, *J* = 2.1, 4.9, 11.2 Hz, 1 H), 2.78 (dd, *J* = 5.6, 12.6 Hz, 1 H), 2.23–2.15 (m, 3 H), 2.07 (dt, *J* = 9.1, 13.3 Hz, 1 H) 1.99 (dt, *J* = 6.3, 11.2 Hz, 1 H), 1.93–1.88 (m, 3 H), 1.84 (dd, *J* = 6.3, 12.6 Hz, 1 H) 1.69–1.64 (m, 2 H), 0.90 (t, *J* = 7.7 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 153.7, 140.8, 135.6, 134.9, 131.2, 129.3, 128.9, 123.8, 117.0, 58.0, 57.4, 52.8, 37.7, 31.3, 31.0, 30.9, 27.8, 20.7, 14.0; IR (film) 1643 cm^{-1}. MS (ESI) 359.1887 (359.1885 calcd for $C_{21}H_{27}ClN_2O$, M +

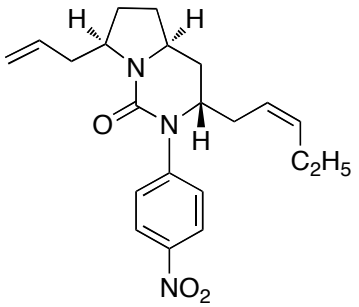
H^+). The enantiopurity was determined to be 95:5 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 190 nm, RT= 13.4 and 18.1 min).



(–)-(*Z*,3*S*,4a*S*,7*R*)-7-Allyl-2-(4-bromophenyl)-3-(pent-2-en-1-yl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8d). The general procedure was employed for the coupling of **7d** (70 mg, 0.2 mmol) and (*Z*)-1-bromobut-1-ene (150 µL, 0.3 mmol, 2.0 M solution in toluene), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 15 mg (18%) of the title compound as a brown oil and as a 18:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ –21.1 (*c* 0.5, CH_2Cl_2). This material also contained ca. 20% of an unidentified side product. Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.4 Hz, 2 H), 7.14 (d, *J* = 8.4 Hz, 2 H), 5.77–5.71 (m, 1 H), 5.45 (dt, *J* = 7.0, 11.2 Hz, 1 H), 5.12–5.08 (m, 1 H), 5.04 (d, *J* = 17.5 Hz, 1 H), 5.01 (d, *J* = 10.5 Hz, 1 H), 4.01 (dt, *J* = 2.8, 8.4 Hz, 1 H), 3.90 (dt, *J* = 4.9, 10.5 Hz, 1 H), 3.66 (ddt, *J* = 2.8, 5.6, 11.2 Hz, 1 H), 2.78 (dd, *J* = 5.6, 12.6 Hz, 1 H), 2.23–2.15 (m, 3 H), 2.07 (dt, *J* = 8.4, 13.3 Hz, 1 H) 1.99 (dt, *J* = 5.6, 11.9 Hz, 1 H), 1.95–1.88 (m, 3 H), 1.84 (dd, *J* = 6.3, 12.6 Hz, 1 H) 1.69–1.63 (m, 2 H), 0.90 (t, *J* = 7.7 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 153.6, 141.4, 135.6, 134.9, 131.9, 129.7, 123.8, 119.2, 117.0, 57.9, 57.4, 52.7, 37.7, 31.3, 31.0, 30.8, 27.7, 20.7, 14.0; IR (film) 1645 cm^{-1}. MS (ESI) 403.1379 (403.1380 calcd for $C_{21}H_{27}BrN_2O$, M + H^+). The enantiopurity was determined to be 95:5 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 205 nm, RT= 14.5 and 20.0 min).

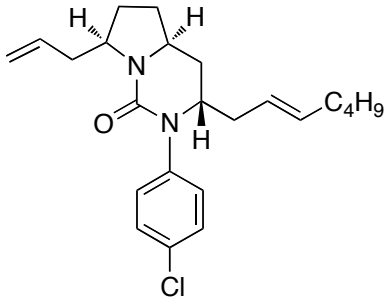


(–)-4-[(*Z*,3*S*,4a*S*,7*R*)-7-Allyl-1-oxo-3-(pent-2-en-1-yl)hexahydropyrrolo[1,2-*c*]pyrimidin-2(1*H*)-yl]benzonitrile (8e). The general procedure was employed for the coupling of **7e** (59 mg, 0.2 mmol) and (*Z*)-1-bromobut-1-ene (150 µL, 0.3 mmol, 2.0 M solution in toluene), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 29 mg (41%) of the title compound as a brown oil and as a 17:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ –71.0 (*c* 2.9, CH_2Cl_2). This material also contained ca. 5% of 4-aminobenzonitrile. Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.62 (d, *J* = 9.1 Hz, 2 H), 7.39 (d, *J* = 9.1 Hz, 2 H), 5.76–5.71 (m, 1 H), 5.45 (dt, *J* = 7.0, 11.2 Hz, 1 H), 5.12–5.08 (m, 1 H), 5.04 (d, *J* = 16.1 Hz, 1 H), 5.02 (d, *J* = 9.1 Hz, 1 H), 4.07 (dt, *J* = 4.9, 10.5 Hz, 1 H), 4.02 (dt, *J* = 2.1, 8.4, Hz 1 H), 3.68 (ddt, *J* = 2.1, 5.6, 11.2 Hz, 1 H), 2.76 (dd, *J* = 6.3, 12.6 Hz, 1 H), 2.23 (d, *J* = 12.6 Hz, 1 H), 2.20–2.14 (m, 2 H), 2.06 (dt, *J* = 8.4, 13.3 Hz, 1 H), 2.03–2.00 (m, 1 H), 1.97–1.85 (m, 4 H), 1.71–1.63 (m, 2 H), 0.89 (t, *J* = 7.7 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 153.1, 146.6, 135.3, 135.2, 132.6, 127.8, 123.4, 118.9, 117.2, 108.5, 57.7, 57.4, 52.7, 37.4, 31.4, 31.0, 30.8, 27.7, 20.7, 14.0; IR (film) 1648 cm^{-1}. MS (ESI) 350.2227 (350.2227 calcd for $C_{22}H_{27}N_3O$, M + H^+). The enantiopurity was determined to be 94:6 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 205 nm, RT= 33.2 and 42.9 min).



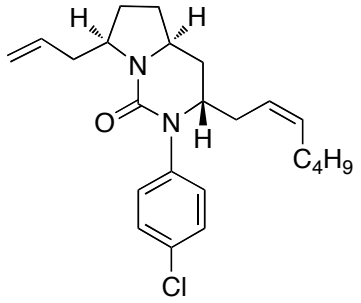
(–)-(*Z*,3*S*,4a*S*,7*R*)-7-Allyl-2-(4-nitrophenyl)-3-(pent-2-en-1-yl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8f). A modification of the general procedure was employed for the coupling of **7f** (63 mg, 0.2 mmol) and (*Z*)-1-bromobut-1-ene (150 µL, 0.3 mmol, 2.0 M solution in toluene), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8

mg, 0.016 mmol). In contrast to the general procedure, this reaction was run overnight (16 h) at 120 °C. This procedure afforded 18 mg (24%) of the title compound as a yellow oil and as a 20:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ –281.3 (*c* 1.1, CH_2Cl_2). This material also contained ca. 8% of unreacted starting material and ca. 3% of a bicyclic urea side product lacking the butenyl group (tentatively assigned as 7-allyl-3-methyl-2-(4-nitrophenyl)-4a,5,6,7-tetrahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one). Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 8.21 (d, *J* = 9.1 Hz, 2 H), 7.46 (d, *J* = 9.1 Hz, 2 H), 5.77–5.71 (m, 1 H), 5.46 (dt, *J* = 7.0, 11.2 Hz, 1 H), 5.12 (ddt, *J* = 2.1, 8.4, 17.5 Hz, 1 H), 5.07–5.03 (m, 2 H), 4.15 (dt, *J* = 4.,9 9.8 Hz, 1 H), 4.05 (dt, *J* = 2.8, 9.1 Hz, 1 H), 3.70 (ddt, *J* = 2.8, 5.6, 11.2 Hz, 1 H), 2.78 (dd, *J* = 5.6, 13.3 Hz, 1 H), 2.25 (d, *J* = 13.3 Hz, 1 H), 2.23–2.16 (m, 2 H), 2.10–2.02 (m, 2 H) 1.97–1.87 (m, 4 H), 1.73–1.66 (m, 2 H), 0.90 (t, *J* = 7.7 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 153.0, 148.5, 144.4, 135.3, 135.3, 127.2, 124.1, 123.3, 117.3, 57.8, 57.4, 52.7, 37.3, 31.5, 31.0, 30.8, 27.7, 20.8, 14.0; IR (film) 1649 cm^{-1}. MS (ESI) 370.2126 (370.2125 calcd for $C_{21}H_{27}N_3O_3$, M + H^+). The enantiopurity was determined to be 96:4 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 1.5 mL/min, λ 310 nm, RT= 19.1 and 26.2 min).

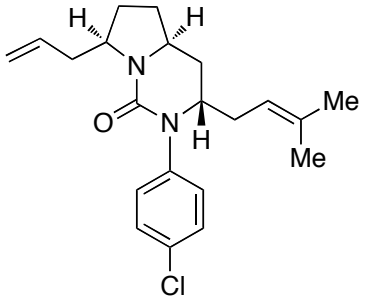


(–)-(*E*,3*S*,4a*S*,7*R*)-7-Allyl-2-(4-chlorophenyl)-3-(hept-2-en-1-yl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8g). The general procedure was employed for the coupling of **7c** (61 mg, 0.2 mmol) and (*E*)-1-bromohex-1-ene (49 mg, 0.3 mmol), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). 6 M HCl was used in the workup to remove 4-chloroaniline side product. This procedure afforded 44 mg (57%) of the title compound as a yellow oil: $[\alpha]^{23}_D$ –30.3 (*c* 1.9, CH_2Cl_2). This material also contained ca. 15% of a regioisomeric bicyclic urea product generated from the coupling of **7c** and 2-bromohex-1-ene (tentatively assigned as (3*S*,4a*S*,7*R*)-7-allyl-2-(4-chlorophenyl)-3-(2-methylenehexyl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one). Data are for the major isomer. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 (d, *J* = 9.0 Hz, 2 H), 7.17 (d, *J* = 9.0 Hz, 2 H), 5.78–5.70 (m, 1 H), 5.41–5.36 (m, 1 H), 5.17–5.10 (m, 1 H), 5.05–5.00 (m, 2 H), 4.00 (dt, *J* = 2.5, 8.5 Hz, 1 H),

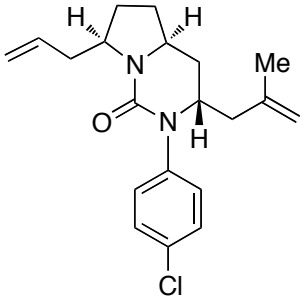
3.92–3.87 (m, 1 H), 3.65 (ddt, *J* = 2.5, 5.5, 11.0 Hz, 1 H), 2.79 (dd, *J* = 6.0, 13.5 Hz, 1 H), 2.28–2.23 (m, 2 H), 2.10–1.98 (m, 3 H), 1.95–1.87 (m, 3 H), 1.84 (dd, *J* = 6.5, 12.5 Hz, 1 H) 1.69–1.62 (m, 2 H), 1.29–1.26 (m, 4 H), 0.87 (t, *J* = 7.0 Hz, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 153.7, 140.9, 135.6, 134.6, 131.2, 129.4, 128.8, 125.1, 116.9, 57.9, 57.4, 52.6, 37.7, 36.9, 32.2, 31.4, 30.8, 27.8, 22.1, 13.9 (one carbon signal is absent due to incidental equivalence); IR (film) 1643 cm^{-1}. MS (ESI) 387.2207 (387.2198 calcd for $C_{23}H_{31}ClN_2O$, M + H^+). The enantiopurity was determined to be 95:5 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 1.5% IPA/Hexanes, 1.5 mL/min, λ 205 nm, RT= 20.0 and 37.5 min).



(–)-(*Z*,3*S*,4a*S*,7*R*)-7-Allyl-2-(4-chlorophenyl)-3-(hept-2-en-1-yl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8h). The general procedure was employed for the coupling of **7c** (61 mg, 0.2 mmol) and (*Z*)-1-bromohex-1-ene (49 mg, 0.3 mmol), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). 6 M HCl was used in the workup to remove 4-chloroaniline side product. This procedure afforded 47 mg (61%) of the title compound as a yellow brown oil: $[\alpha]^{23}_D$ –14.8 (*c* 3.5, CH_2Cl_2). Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.31 (d, *J* = 9.1 Hz, 2 H), 7.19 (d, *J* = 8.4 Hz, 2 H), 5.77–5.71 (m, 1 H), 5.45 (dt, *J* = 7.0, 11.2 Hz, 1 H), 5.15–5.11 (m, 1 H), 5.05–5.00 (m, 2 H), 4.01 (dt, *J* = 2.8, 8.4 Hz, 1 H), 3.91–3.88 (m, 1 H), 3.66 (ddt, *J* = 2.1, 4.9, 11.2 Hz, 1 H), 2.78 (dd, *J* = 8.4, 13.3 Hz, 1 H), 2.24–2.14 (m, 3 H), 2.07 (dt, *J* = 8.4, 14.0 Hz, 1 H), 2.00–1.97 (m, 1 H) 1.95–1.83 (m, 4 H), 1.70–1.62 (m, 2 H), 1.25–1.24 (m, 4 H), 0.86 (t, *J* = 7.0 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 153.7, 140.8, 135.6, 133.4, 131.3, 129.4, 128.9, 124.4, 117.0, 58.1, 57.4, 52.8, 37.7, 31.6, 31.3, 31.0, 30.8, 27.8, 27.2, 22.3, 13.9; IR (film) 1642 cm^{-1}. MS (ESI) 387.2203 (387.2198 calcd for $C_{23}H_{31}ClN_2O$, M + H^+). The enantiopurity was determined to be 95:5 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 1.5% IPA/Hexanes, 1.5 mL/min, λ 254 nm, RT= 20.9 and 36.2 min).

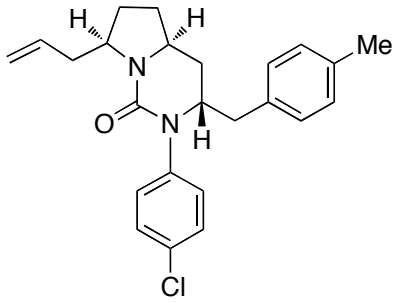


(–)-(3*S*,4a*S*,7*R*)-7-Allyl-2-(4-chlorophenyl)-3-(3-methylbut-2-en-1-yl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8i). The general procedure was employed for the coupling of **7c** (61 mg, 0.2 mmol) and 1-bromo-2-methyl-1-propene (31 µL, 0.3 mmol), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 37 mg (52%) of the title compound as a yellow oil and as a 10:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ –37.9 (*c* 2.2, CH_2Cl_2). Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.30 (d, *J* = 7.7 Hz, 2 H), 7.18 (d, *J* = 9.1 Hz, 2 H), 5.74 (dddd, *J* = 6.3, 7.7, 10.5, 16.8 Hz, 1 H), 5.04 (dd, *J* = 2.1, 16.8 Hz, 1 H), 5.01 (d, *J* = 10.5 Hz, 1 H), 4.89 (dt, *J* = 1.4, 7.0 Hz, 1 H), 4.00 (dt, *J* = 2.8, 9.1 Hz, 1 H), 3.88–3.85 (m, 1 H), 3.66 (ddt, *J* = 2.8, 5.6, 11.2 Hz, 1 H), 2.79 (dd, *J* = 6.3, 12.6 Hz, 1 H), 2.21–2.16 (m, 2 H), 2.09–2.04 (m, 2 H), 2.02–1.99 (m, 1 H), 1.95–1.89 (m, 1 H), 1.84 (dd, *J* = 7.0, 12.6 Hz, 1 H) 1.68–1.62 (m, 2 H), 1.64 (s, 3 H), 1.47 (s, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 153.7, 140.9, 135.6, 134.9, 131.1, 129.3, 128.8, 119.6, 117.0, 58.3, 57.4, 52.8, 37.7, 32.2, 31.1, 30.9, 27.7, 25.7, 17.9; IR (film) 1643 cm^{-1}. MS (ESI) 359.1895 (359.1885 calcd for $C_{21}H_{27}ClN_2O$, M + H^+). The enantiopurity was determined to be 94:6 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 254 nm, RT= 13.8 and 24.0 min).



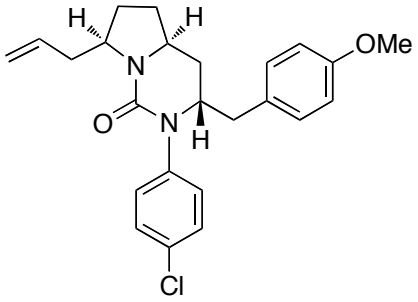
(–)-(3*S*,4a*S*,7*R*)-7-Allyl-2-(4-chlorophenyl)-3-(2-methylallyl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8j). The general procedure was employed for the coupling of **7c** (61 mg, 0.2 mmol) and 2-bromopropene (89 µL, 1.0 mmol), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 39 mg

(56%) of the title compound as a yellow oil and as a 20:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ –33.5 (*c* 2.9, CH_2Cl_2). Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.4 Hz, 2 H), 7.19 (d, *J* = 9.1 Hz, 2 H), 5.77–5.71 (m, 1 H), 5.06–5.01 (m, 2 H), 4.79 (s, 1 H), 4.66 (s, 1 H), 4.09–4.06 (m, 1 H), 4.00 (dt, *J* = 2.8, 8.4 Hz, 1 H), 3.66 (ddt, *J* = 2.8, 4.9, 11.2 Hz, 1 H), 2.79 (dd, *J* = 6.3, 12.6 Hz, 1 H), 2.27–2.23 (m, 2 H), 2.12 (dd, *J* = 11.2, 14.0 Hz, 1 H) 2.07 (dt, *J* = 8.4, 13.3 Hz, 1 H), 2.03–1.99 (m, 1 H), 1.95–1.89 (m, 1 H), 1.85 (dd, *J* = 7.0, 12.6 Hz, 1 H) 1.68–1.64 (m, 2 H), 1.55 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 153.7, 141.5, 140.7, 135.6, 131.2, 129.3, 128.9, 117.0, 113.9, 57.5, 55.8, 52.6, 41.8, 37.7, 30.8, 30.5, 27.8, 22.0; IR (film) 1641 cm^{-1}. MS (ESI) 345.1735 (345.1728 calcd for $C_{20}H_{25}ClN_2O$, M + H$^+$). The enantiopurity was determined to be 88:12 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 3% IPA/Hexanes, 0.75 mL/min, λ 254 nm, RT= 22.8 and 28.4 min).

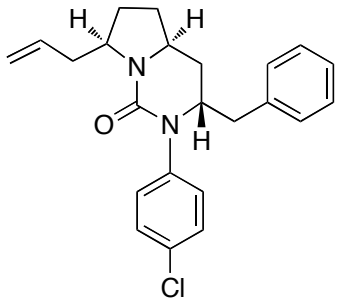


(–)-(3*S*,4a*S*,7*R*)-7-Allyl-2-(4-chlorophenyl)-3-(4-methylbenzyl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8k). The general procedure was employed for the coupling of **7c** (61 mg, 0.2 mmol) and 4-bromotoluene (37 μL, 0.3 mmol), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 66 mg (83%) of the title compound as a pale yellow oil and as a 8:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ –125.6 (*c* 3.1, CH_2Cl_2). Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.4 Hz, 2 H), 7.26 (d, *J* = 8.4 Hz, 2 H), 7.06 (d, *J* = 8.4 Hz, 2 H), 6.86 (d, *J* = 7.7 Hz, 2 H), 5.77–5.71 (m, 1 H), 5.04 (d, *J* = 16.8 Hz, 1 H), 5.01 (d, *J* = 10.5 Hz, 1 H), 4.10 (dt, *J* = 4.9 11.2 Hz, 1 H), 4.03 (dt, *J* = 2.8, 8.4, Hz 1 H), 3.76 (ddt, *J* = 2.8, 5.6, 11.2 Hz, 1 H), 2.90 (dd, *J* = 2.8, 13.3 Hz, 1 H), 2.81–2.78 (m, 1 H), 2.53 (dd, *J* = 11.2, 14.0 Hz, 1 H), 2.30 (s, 3 H), 2.09–2.04 (m, 2 H), 2.00 (dt, *J* = 5.6, 11.9 Hz, 1 H), 1.96–1.91 (m, 1 H), 1.85 (dd, *J* = 5.6, 12.6 Hz, 1 H) 1.64–1.59 (m, 1 H), 1.56 (dt, *J* = 6.3, 12.6 Hz, 1 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 153.6, 140.8, 136.2, 135.6, 134.5, 131.3, 129.3, 129.3, 129.0, 128.9, 117.0, 59.8, 57.5, 52.6, 39.2, 37.6, 30.8, 30.1, 27.8, 21.0; IR (film) 1642 cm^{-1}. MS (ESI) 395.1887 (395.1885 calcd for $C_{24}H_{27}ClN_2O$, M + H$^+$). The enantiopurity was determined to be 92:8 er by

chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 254 nm, RT= 17.3 and 19.4 min).

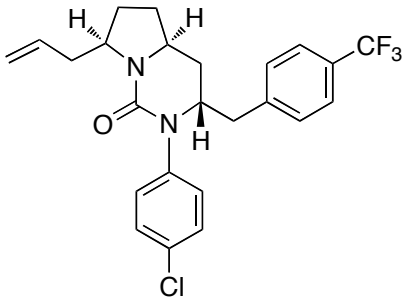


(–)-(3*S*,4a*S*,7*R*)-7-Allyl-2-(4-chlorophenyl)-3-(4-methoxybenzyl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8l). The general procedure was employed for the coupling of **7c** (61 mg, 0.2 mmol) and 4-bromoanisole (38 μL, 0.3 mmol), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 58 mg (70%) of the title compound as a pale yellow oil and as a 8:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ –169.4 (*c* 2.2, CH_2Cl_2). Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.35 (d, *J* = 8.4 Hz, 2 H), 7.25 (d, *J* = 8.4 Hz, 2 H), 6.89 (d, *J* = 9.1 Hz, 2 H), 6.79 (d, *J* = 8.4 Hz, 2 H), 5.77–5.71 (m, 1 H), 5.04 (d, *J* = 17.5 Hz, 1 H), 5.01 (d, *J* = 10.5 Hz, 1 H), 4.08 (dt, *J* = 4.2, 11.2 Hz, 1 H), 4.03 (dt, *J* = 2.1, 8.4 Hz, 1 H), 3.77 (s, 3 H), 3.77–3.72 (m, 1 H), 2.87 (dd, *J* = 3.5. 14.0 Hz, 1 H), 2.80 (dd, *J* = 6.3, 14.0 Hz, 1 H), 2.51 (dd, *J* = 11.2, 13.3 Hz, 1 H), 2.09–2.04 (m, 2 H), 2.00 (dt, *J* = 5.6, 11.9 Hz, 1 H), 1.96–1.91 (m, 1 H), 1.85 (dd, *J* = 7.0, 12.6 Hz, 1 H) 1.65–1.54 (m, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 158.3, 153.6, 140.8, 135.6, 131.3, 130.0, 129.6, 129.3, 129.0, 117.0, 114.0, 59.9, 57.5, 55.2, 52.6, 38.7, 37.6, 30.8, 30.1, 27.8; IR (film) 1642 cm^{-1}. MS (ESI) 411.1834 (411.1834 calcd for $C_{24}H_{27}ClN_2O_2$, M + H^+). The enantiopurity was determined to be 92:8 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 3% IPA/Hexanes, 0.75 mL/min, λ 204 nm, RT= 49.3 and 55.7 min).



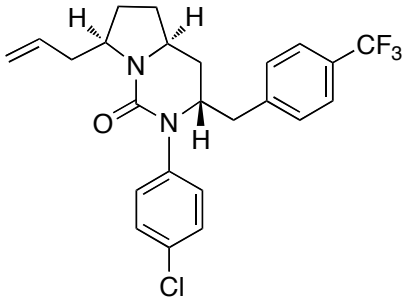
(–)-(3*S*,4a*S*,7*R*)-7-Allyl-3-benzyl-2-(4-chlorophenyl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8m). The general procedure was employed for the coupling of **7c** (61 mg, 0.2 mmol)

and bromobenzene (32 μL, 0.3 mmol), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 63 mg (83%) of the title compound as a pale brown foam oil and as a 7:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ –61.2 (*c* 5.6, CH_2Cl_2). Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.36 (d, *J* = 9.1 Hz, 2 H), 7.27–7.25 (m, 4 H), 7.21–7.20 (m, 1 H), 6.98 (d, *J* = 7.0 Hz, 2 H), 5.77–5.71 (m, 1 H), 5.05–5.00 (m, 2 H), 4.14 (dt, *J* = 4.9, 11.2 Hz, 1 H), 4.03 (dt, *J* = 2.1, 8.4 Hz, 1 H), 3.77 (ddt, *J* = 2.1, 4.9, 11.2 Hz, 1 H), 2.94 (dd, *J* = 3.5, 14.0 Hz, 1 H), 2.80 (dd, *J* = 6.3, 13.3 Hz, 1 H), 2.57 (dd, *J* = 11.2, 14.0 Hz, 1 H), 2.10–2.04 (m, 2 H), 2.03–2.00 (m, 1 H), 1.97–1.91 (m, 1 H), 1.86 (dd, *J* = 6.3, 12.6 Hz, 1 H) 1.66–1.54 (m, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 153.6, 140.8, 137.6, 135.6, 131.4, 129.3, 129.1, 129.0, 128.6, 126.6, 117.0, 59.7, 57.5, 52.7, 39.6, 37.6, 30.8, 30.2, 27.8; IR (film) 1642 cm^{-1}. MS (ESI) 381.1736 (381.1728 calcd for $C_{23}H_{25}ClN_2O$, M + H^+). The enantiopurity was determined to be 90:10 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 245 nm, RT= 21.1 and 24.2 min).

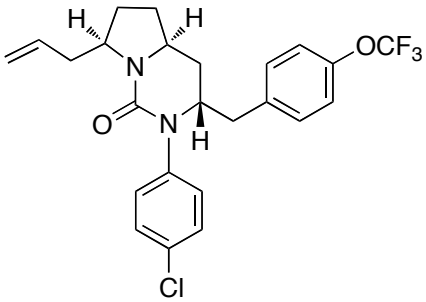


(–)-(3*S*,4a*S*,7*R*)-7-Allyl-2-(4-chlorophenyl)-3-[4-(trifluoromethyl)benzyl]hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8n). The general procedure was employed for the coupling of **7c** (61 mg, 0.2 mmol) and 4-bromobenzotriflouride (42 μL, 0.3 mmol), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 66 mg (74%) of the title compound as a pale yellow oil and as a 5:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ –46.1 (*c* 6.0, CH_2Cl_2). Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.51 (d, *J* = 7.7 Hz, 2 H), 7.36 (d, *J* = 9.1 Hz, 2 H), 7.24 (d, *J* = 9.1 Hz, 2 H), 7.09 (d, *J* = 7.7 Hz, 2 H), 5.77–5.71 (m, 1 H), 5.05–5.01 (m, 2 H), 4.16 (dt, *J* = 4.2, 11.2 Hz, 1 H), 4.03 (dt, *J* = 2.1, 9.1 Hz, 1 H), 3.76 (ddt, *J* = 2.1, 5.6, 11.9 Hz, 1 H), 3.00 (dd, *J* = 3.5, 14.0 Hz, 1 H), 2.79 (dd, *J* = 5.6, 13.3 Hz, 1 H), 2.66 (dd, *J* = 11.2, 13.3 Hz, 1 H), 2.09–2.00 (m, 3 H), 1.98–1.93 (m, 1 H), 1.87 (dd, *J* = 6.3, 11.9 Hz, 1 H) 1.66–1.56 (m, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 153.5, 141.7, 140.6, 135.5, 131.6, 129.4, 129.3, 129.2 (q, *J* = 37 Hz), 129.1, 125.6 (q, 3.3 Hz), 124.0 (q, *J* = 270 Hz), 117.1,

59.5, 57.6, 52.6, 39.6, 37.6, 30.8, 30.3, 27.8; IR (film) 1642 cm^{-1}. MS (ESI) 449.1600 (449.1602 calcd for $C_{24}H_{24}ClF_3N_2O$, M + H^+). The enantiopurity was determined to be 85:15 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 205 nm, RT= 19.9 and 27.5 min).

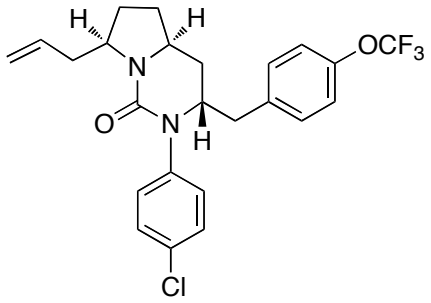


(–)-(3*S*,4a*S*,7*R*)-7-Allyl-2-(4-chlorophenyl)-3-[4-(trifluoromethyl)benzyl]hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8n). A modified general procedure was employed for the coupling of **7c** (61 mg, 0.2 mmol) and 4-bromobenzotriflouride (42 µL, 0.3 mmol) using NaOMe (16.2 mg, 0.3 mmol) as base and a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 54 mg (60%) of the title compound as a pale yellow oil and as a 10:1 mixture of diastereomers as determined by ^1H NMR analysis: $[\alpha]^{23}_D$ –51.1 (*c* 2.3, CH_2Cl_2). The enantiopurity was determined to be 90:10 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 205 nm, RT= 19.4 and 26.7 min). Spectroscopic data were identical to those provided above.

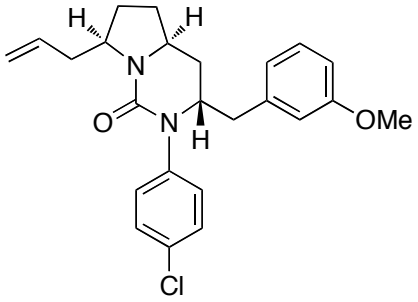


(–)-(3*S*,4a*S*,7*R*)-7-Allyl-3-benzyl-2-(4-chlorophenyl)-3-[4-(trifluoromethoxy)benzyl]hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8o). The general procedure was employed for the coupling of **7c** (61 mg, 0.2 mmol) and 1-bromo-4-(trifluoromethoxy)benzene (45 µL, 0.3 mmol), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 63 mg (68%) of the title compound as a pale yellow oil and as a 8:1 mixture of diastereomers as determined by

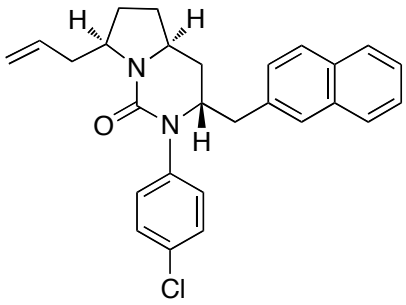
^{1}H NMR analysis: $[\alpha]^{23}_D$ –48.7 (*c* 5.7, CH_2Cl_2). Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.35 (d, *J* = 9.1 Hz, 2 H), 7.24 (d, *J* = 9.1 Hz, 2 H), 7.10 (d, *J* = 7.7 Hz, 2 H), 6.99 (d, *J* = 8.4 Hz, 2 H), 5.77–5.71 (m, 1 H), 5.05–5.01 (m, 2 H), 4.13 (dt, *J* = 4.2, 11.2 Hz, 1 H), 4.03 (dt, *J* = 2.8, 8.4 Hz, 1 H), 3.75 (ddt, *J* = 2.8, 4.9, 11.2 Hz, 1 H), 2.93 (dd, *J* = 4.2, 14.0 Hz, 1 H), 2.79 (dd, *J* = 6.3, 13.3 Hz, 1 H), 2.60 (dd, *J* = 10.5, 14.0 Hz, 1 H), 2.10–2.00 (m, 3 H), 1.98–1.92 (m, 1 H), 1.86 (dd, *J* = 6.3, 12.6 Hz, 1 H) 1.67–1.57 (m, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 153.5, 147.9, 140.7, 136.7, 135.5, 131.5, 130.3, 129.3, 129.1, 121.2, 117.0, 59.6, 57.5, 52.6, 39.1, 37.6, 30.8, 30.3, 27.8 (the CF_3 carbon signal could not be determined due to the appearance of carbon signals from the minor diastereomer in the CF_3 region of the spectrum); IR (film) 1642 cm^{-1}. MS (ESI) 465.1557 (465.1551 calcd for $C_{24}H_{24}ClF_3N_2O_2$, M + H^+). The enantiopurity was determined to be 88:12 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 245 nm, RT= 17.1 and 19.8 min).



(–)-(3*S*,4a*S*,7*R*)-7-Allyl-3-benzyl-2-(4-chlorophenyl)-3-[4-(trifluoromethoxy)benzyl]hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8o). A modified general procedure was employed for the coupling of **7c** (61 mg, 0.2 mmol) and 1-bromo-4-(trifluoromethoxy)benzene (45 µL, 0.3 mmol) using NaOMe (16.2 mg, 0.3 mmol) as base and a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 48 mg (52%) of the title compound as a pale yellow oil and as an 17:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ –55.6 (*c* 1.5, CH_2Cl_2). The enantiopurity was determined to be 93:7 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 245 nm, RT= 16.8 and 19.8 min). Spectroscopic data were identical to those provided above.

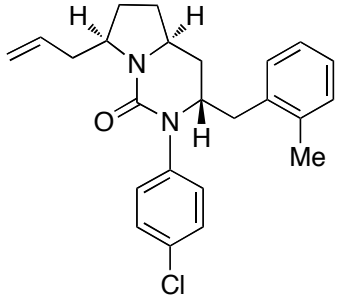


(–)-(3*S*,4a*S*,7*R*)-7-Allyl-2-(4-chlorophenyl)-3-(3-methoxybenzyl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8p). The general procedure was employed for the coupling of **7c** (61 mg, 0.2 mmol) and 3-bromoanisole (38 μL, 0.3 mmol), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 61 mg (74%) of the title compound as a yellow brown solid and as a 5:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ –65.1 (*c* 2.8, CH_2Cl_2). Mp = 132–137 °C. Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.4 Hz, 2 H), 7.25 (d, *J* = 9.1 Hz, 2 H), 7.18 (t, *J* = 8.4 Hz, 1 H), 6.74 (dd, *J* = 2.1, 8.4 Hz, 1 H), 6.57 (d, *J* = 7.0 Hz, 1 H), 6.50 (s, 1 H), 5.77–5.71 (m, 1 H), 5.04 (dd, *J* = 1.4, 16.8 Hz, 1 H), 5.01 (dd, *J* = 1.4, 9.8 Hz, 1 H), 4.14 (dt, *J* = 4.2, 11.2 Hz, 1 H), 4.03 (dt, *J* = 2.1, 8.4 Hz, 1 H), 3.77 (s, 3 H), 3.78–3.73 (m, 1 H), 2.91 (dd, *J* = 3.5, 13.3 Hz, 1 H), 2.80 (dd, *J* = 5.6, 13.3 Hz, 1 H), 2.54 (dd, *J* = 11.2, 14.0 Hz, 1 H), 2.10–2.04 (m, 2 H), 2.03–1.99 (m, 1 H), 1.96–1.91 (m, 1 H), 1.85 (dd, *J* = 6.3, 12.6 Hz, 1 H), 1.65–1.56 (m, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 159.7, 153.6, 140.7, 139.2, 135.6, 131.4, 129.6, 129.3, 129.0, 121.4, 117.0, 115.3, 111.3, 59.6, 57.5, 55.2, 52.7, 39.7, 37.6, 30.8, 30.3, 27.8; IR (film) 1642 cm^{-1}. MS (ESI) 411.1841 (411.1834 calcd for $C_{24}H_{27}ClN_2O_2$, M + H$^+$). The enantiopurity was determined to be 87:13 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 248 nm, RT= 27.2 and 30.6 min).



(–)-(3*S*,4a*S*,7*R*)-7-Allyl-2-(4-chlorophenyl)-3-(napthalen-2-ylmethyl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8q). The general procedure was employed for the coupling of **7c** (61 mg, 0.2 mmol) and 2-bromonapthanlene (62 mg, 0.3 mmol), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded

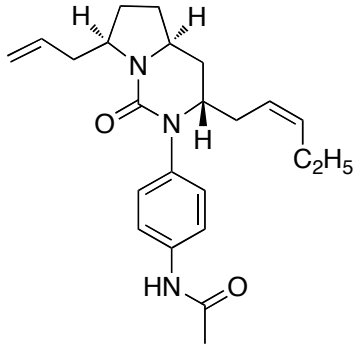
66 mg (77%) of the title compound as a white solid and as a 7:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ –77.9 (*c* 4.6, CH_2Cl_2). Data are for the major isomer. Mp = 63–65 °C. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.7 Hz, 1 H), 7.75 (t, *J* = 7.7 Hz, 2 H), 7.47–7.44 (m, 3 H), 7.38 (d, *J* = 9.1 Hz, 2 H), 7.31 (d, *J* = 8.4 Hz, 2 H), 7.07 (dd, *J* = 0.7, 8.4 Hz, 1 H), 5.78–5.72 (m, 1 H), 5.04 (d, *J* = 16.8 Hz, 1 H), 5.02 (d, *J* = 9.8 Hz, 1 H), 4.25 (dt, *J* = 4.2, 11.2 Hz, 1 H), 4.06 (dt, *J* = 2.1, 9.8 Hz, 1 H), 3.84 (ddt, *J* = 2.1, 4.9, 11.2 Hz, 1 H), 3.11 (dd, *J* = 3.5, 14.0 Hz, 1 H), 2.81 (dd, *J* = 5.6, 13.3 Hz, 1 H), 2.74 (dd, *J* = 11.9, 14.0, Hz, 1 H), 2.11–2.05 (m, 2 H), 2.04–1.94 (m, 2 H), 1.86 (dd, *J* = 7.0, 12.6 Hz, 1 H), 1.65–1.56 (m, 2 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 153.6, 140.8, 135.6, 135.1, 133.4, 132.2, 131.4, 130.4, 129.4, 129.1, 128.4, 127.7, 127.3, 127.1, 126.3, 125.7, 117.0, 59.6, 57.5, 52.7, 37.6, 30.8, 31.0, 30.2, 27.8; IR (film) 1646 cm^{-1}. MS (ESI) 431.1886 (431.1885 calcd for $C_{27}H_{27}ClN_2O$, M + H^+). The enantiopurity was determined to be 88:12 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 215 nm, RT= 24.4and 28.2 min).



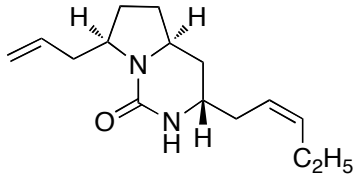
(–)-(3*S*,4a*S*,7*R*)-7-Allyl-2-(4-chlorophenyl)-3-(2-methylbenzyl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (8r). The general procedure was employed for the coupling of **7c** (61 mg, 0.2 mmol) and 2-bromotoluene (36 µL, 0.3 mmol), using a catalyst composed of Pd_2dba_3 (3.7 mg, 0.004 mmol), and (*S*)-Siphos-PE (8 mg, 0.016 mmol). This procedure afforded 65 mg (82%) of the title compound as a pale brown oil and as a 5:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ –30.1 (*c* 5.7, CH_2Cl_2). Data are for the major isomer. ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.36 (d, *J* = 9.1 Hz, 2 H), 7.26 (d, *J* = 8.4 Hz, 2 H), 7.11–7.08 (m, 3 H), 6.93–6.92 (m, 1 H), 5.78–5.71 (m, 1 H), 5.04 (dd, *J* = 1.4, 17.5 Hz, 1 H), 5.01 (dd, *J* = 1.4, 10.5 Hz, 1 H), 4.09 (dt, *J* = 4.2, 12.6 Hz, 1 H), 4.04 (dt, *J* = 2.8, 9.1 Hz, 1 H), 3.86 (ddt, *J* = 2.8, 5.6, 11.2 Hz, 1 H), 2.93 (dd, *J* = 3.5, 14.0 Hz, 1 H), 2.80 (dd, *J* = 5.6, 12.6 Hz, 1 H), 2.62 (dd, *J* = 11.2, 14.0 Hz, 1 H), 2.11–2.02 (m, 3 H), 2.01 (s, 3 H), 1.99–1.94 (m, 1 H), 1.87 (dd, *J* = 6.3, 12.6 Hz, 1 H), 1.65 (ddd, *J* = 6.3, 11.2, 17.5 Hz, 1 H), 1.58 (dt, *J* = 5.6, 12.6 Hz, 1 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 153.6, 140.7, 136.3, 135.7, 135.6, 131.5, 130.6, 130.3, 129.6, 129.0, 126.8, 126.0, 117.0, 58.4, 57.5, 52.9, 37.7, 36.8, 30.8, 30.1, 27.8, 19.2; IR (film) 1642 cm^{-1}. MS (ESI)

395.1885 (395.1885 calcd for $C_{24}H_{27}ClN_2O$, M + H^+). The enantiopurity was determined to be 71:29 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 215 nm, RT= 20.1 and 24.3 min).

Deprotection of Bicyclic Urea Product 8c

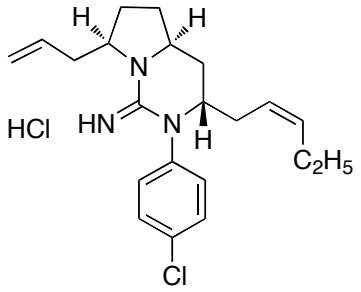


(–)-(*Z*,3*S*,4a*S*,7*R*)-*N*-{4-[7-Allyl-1-oxo-3-(pent-2-en-1-yl)hexahydropyrrolo[1,2-*c*]pyrimidin-2(1*H*)-yl]phenyl}acetamide (S3). A flame-dried screwtop-flask was cooled under vacuum and charged with $Pd_2(dba)_3$ (5.2 mg, 0.006 mmol), 2-di-*tert*-butylphosphino-3,4,5,6-tetramethyl-2',4',6'-triisopropyl-1,1'-biphenyl (13.7 mg, 0.03 mmol), K_3PO_4 (182 mg, 0.86 mmol) and acetamide (50.8 mg, 0.86 mmol). The flask was evacuated and backfilled with N_2, and then a solution of **8c** (206 mg, 0.57 mmol) in *tert*-butanol (3 mL) was added via syringe. The flask was sealed, heated to 110 °C and stirred overnight (14 h). The mixture was cooled to room temperature and the mixture was filtered through a plug of celite, eluted with EtOAc (10 mL), and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel to afford 191 mg (88%) of the title compound as a foamy brown solid: mp = 38–42 °C. $[\alpha]^{23}_D$ –25.2 (*c* 5.3, CH_2Cl_2). ^{1}H NMR (700 MHz, $CDCl_3$) δ 8.93 (s, 1 H), 7.21 (d, *J* = 8.4 Hz, 2 H), 6.98 (d, *J* = 8.4 Hz, 2 H), 5.77–5.72 (m, 1 H), 5.49–5.40 (m, 1 H), 5.07–5.03 (m, 3 H), 4.03 (dt, *J* = 2.8, 9.1 Hz, 1 H), 3.77 (dt, *J* = 4.2, 10.5 Hz, 1 H), 3.67 (ddt, *J* = 2.8, 4.9, 8.4 Hz, 1 H), 2.80 (dd, *J* = 4.9, 12.6 Hz, 1 H), 2.25–2.08 (m, 4 H), 2.05 (s, 3 H) 2.02–1.98 (m, 1 H), 1.95–1.85 (m, 4 H), 1.70–1.64 (m, 2 H), 0.88 (t, *J* = 7.7 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 168.8, 154.5, 137.3, 136.6, 135.5, 134.8, 128.3, 124.0, 121.3, 117.1, 58.5, 57.3, 52.8, 37.8, 31.2, 31.0, 30.8, 27.7, 24.0, 20.8, 14.1; IR (film) 3263, 1687, 1624 cm^{-1}. MS (ESI) 382.2493 (382.2489 calcd for $C_{23}H_{31}N_3O_2$, M + H^+).



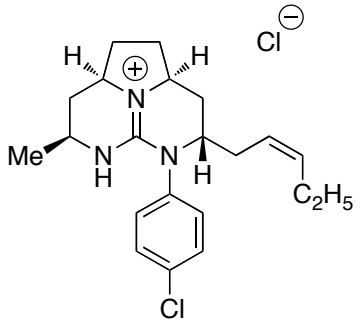
(–)-(*Z*,3*S*,4a*S*,7*R*)-7-Allyl-3-(pent-2-en-1-yl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (9). A Schlenk tube was charged with a stirbar, **S3** (39 mg, 0.1 mmol) and CH_3CN (1 mL). A solution of ceric ammonium nitrate (164 mg, 0.3 mmol) in H_2O (1 mL) was added to the reaction flask and the mixture was stirred at rt for 5 min. The mixture was then heated at 50 °C for 15 min before being cooled to rt, at which time EtOAc (5 mL) was added. The mixture was transferred to a separatory funnel and the layers were separated. The organic layer was washed with saturated aqueous Na_2SO_3 (5 mL), saturated aqueous $NaHCO_3$ (5 mL), and brine (5 mL). The organic layer was dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography (2% MeOH in CH_2Cl_2) on silica gel to afford 19 mg (77%) of the title compound as a yellow brown solid: $[\alpha]^{23}_D$ –63.2 (*c* 0.5, CH_2Cl_2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.80–5.72 (m, 1 H), 5.57–5.51 (m, 1 H), 5.30–5.25 (m, 1 H), 5.06–5.02 (m, 2 H), 4.73 (s, 1 H), 4.00 (dt, *J* = 3.0, 8.5 Hz, 1 H), 3.49 (ddt, *J* = 3.0, 5.5, 11.5 Hz, 1 H), 3.46–3.41 (m, 1 H), 2.72 (d, *J* = 14.5 Hz, 1 H), 2.26–2.20 (m, 1 H), 2.13–1.93 (m, 6 H), 1.88–1.77 (m, 2 H), 1.61–1.52 (m, 2 H), 0.96 (t, *J* = 7.5 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 155.1, 135.6, 135.2, 124.0, 117.0, 56.2, 52.9, 50.0, 38.1, 35.7, 32.3, 30.7, 27.4, 20.8, 14.1; IR (film) 3207, 1652 cm^{-1}. MS (ESI) 249.1963 (249.1961 calcd for $C_{15}H_{24}N_2O$, M + H^+).

Conversion of Bicyclic Urea Product 8c to Tricyclic Guanidine 12



(–)-(*Z*,3*S*,4a*S*,7*R*)-7-Allyl-2-(4-chlorophenyl)-3-(pent-2-en-1-yl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-imine hydrochloride (10). A flame-dried flask was cooled under a stream of N_2 and charged with **8c** (177 mg, 0.49 mmol) and toluene (5 mL). Freshly distilled $POCl_3$ (2.5 mL, 27 mmol) was added and the mixture was stirred at rt until the starting material had been consumed as judged by ESI^+ MS analysis (ca. 3 hr). The reaction mixture was cooled to rt and concentrated *in vacuo*. The crude product was dissolved in acetonitrile (5 mL) and a solution of

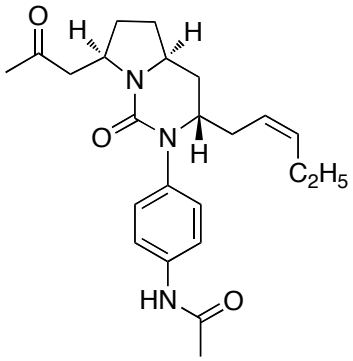
ammonia (20 mL, 2 M in ethanol) was added. The mixture was stirred at rt until the starting material had been consumed as judged by ESI^+ MS analysis (ca. 1 hr). The reaction mixture was concentrated and dissolved in methylene chloride (5 mL). Water (5 mL) was added and the mixture was transferred to a separatory funnel. The layers were separated and the organic layer was washed with saturated aqueous NaCl (3 x 10 mL). The combined aqueous layers were extracted with methylene chloride (3 x 10 mL). The combined organics layers were dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel to afford 146 mg (75%) of the title compound as a pale white-yellow foam: $[\alpha]^{23}_D$ –45.5(*c* 1.1, CH_2Cl_2). ^1H NMR (700 MHz, $CDCl_3$) δ 7.48 (d, *J* = 7.7 Hz, 1 H), 7.40 (d, *J* = 7.7 Hz, 1 H), 7.29 (d, *J* = 7.0 Hz, 1 H), 7.14 (d, *J* = 8.4 Hz, 1 H), 5.96–5.90 (m, 1 H), 5.45 (dt, *J* = 7.0, 10.5 Hz, 1 H), 5.00–4.93 (m, 4 H), 3.75–3.72 (m, 1 H), 3.62–3.58 (m, 1 H), 2.67 (d, *J* = 13.3 Hz, 1 H), 2.25 (dd, *J* = 2.1, 14.0 Hz, 1 H), 2.18–2.16 (m, 1 H) 2.12–2.06 (m, 3 H), 2.03–1.93 (m, 2 H), 1.81–1.68 (m, 4 H), 0.82 (t, *J* = 7.7 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 151.3, 136.6, 136.1, 135.8, 134.5, 131.5, 131.4, 130.8, 128.9, 121.7, 118.3, 59.5, 59.1, 53.1, 36.2, 30.8, 30.4, 29.6, 28.1, 20.8, 13.9; IR (film) 3457, 3275, 1636 cm^{-1}. MS (ESI) 358.2048 (358.2045 calcd for $C_{21}H_{29}ClN_3$, M^+).



(–)-(*Z*,2a*S*,4*S*,7*S*,8a*R*)-5-(4-Chlorophenyl)-7-methyl-4-(pent-2-en-1-yl)-1,2,2a,3,4,5,6,7,8,8a-decahydro-2a^1,5,6-triazaacenaphthylen-2a^1-ium chloride (12). A test tube was charged with **10** (39.4 mg, 0.1 mmol), $PdCl_2$ (3.5 mg, 0.02 mmol), and CuCl (14.8 mg, 0.15 mmol). The tube was capped with a rubber septum, was briefly flushed with oxygen and then an oxygen-filled balloon attached to a needle (via an adaptor) was connected to the tube through the septum. A solution of THF and H_2O (7:1, 1.0 mL) was added to the test tube and the mixture was stirred at rt until the starting material had been consumed as judged by ESI^+ MS analysis (ca. 4 hr). Methanol (1 mL) and $NaCNBH_3$ (62.8 mg, 1.0 mmol) was added and the mixture was heated to 50 ^oC until the starting material had been consumed as judged by ESI^+ MS analysis (ca. 3 hr). The reaction mixture was cooled to rt and concentrated *in vacuo*. The crude product was dissolved in methylene chloride (20 mL), the mixture was transferred to a separatory funnel and

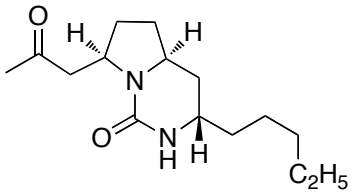
2 M HCl (10 mL) was added. The layers were separated and the organic layer was washed with NH_4OH (10 mL) to potentially remove any excess copper. The layers were separated and the organic layer was washed with 2 M HCl (10 mL). The organics layer was dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel to afford 31 mg (79%) of the title compound as a pale white-tan oil and as a 5:1 mixture of diastereomers as determined by ^{1}H NMR analysis: $[\alpha]^{23}_D$ –38.1 (*c* 0.6, CH_2Cl_2). Data are for the major isomer. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.5 Hz, 2 H), 7.34 (d, *J* = 8.5 Hz, 2 H), 5.57–5.52 (m, 1 H), 5.10–5.05 (m, 1 H), 4.64 (s, 1 H), 3.99–3.90 (m, 2 H), 3.77–3.73 (m, 1 H), 3.69–3.65 (m, 1 H), 2.46–2.32 (m, 5 H), 2.28 (dt, *J* = 3.5, 13.0 Hz, 1 H), 1.99–1.93 (m, 3 H), 1.88–1.80 (m, 2 H), 1.51–1.44 (m, 1 H), 1.22 (d, *J* = 6.5 Hz, 3 H), 0.93 (t, *J* = 7.5 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 148.6, 136.6, 136.5, 135.2, 132.0–129.0 (br, 2 C), 121.9, 59.8, 57.7, 52.1, 47.4, 34.8, 30.0, 29.9, 29.6, 29.4, 20.9, 20.6, 14.0; IR (film) 3276, 1607 cm^{-1}. MS (ESI) 358.2047 (358.2045 calcd for $C_{21}H_{29}ClN_3$, M^+).

Conversion of Bicyclic Urea Product 8c to 9-*epi*-Batzelladine K 16



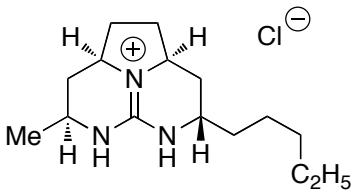
(–)-(*Z*,3*S*,4a*S*,7*R*)-*N*-{4-[1-Oxo-7-(2-oxopropyl)-3-(pent-2-en-1-yl)hexahydropyrrolo[1,2-*c*]pyrimidin-2(1*H*)-yl]phenyl}acetamide (13). A test tube was charged with **S3** (300 mg, 0.79 mmol), $PdCl_2$ (28 mg, 0.16 mmol), and CuCl (117 mg, 1.18 mmol). The tube was capped with a rubber septum, was briefly flushed with oxygen and then an oxygen-filled balloon attached to a needle (via an adaptor) was connected to the tube through the septum. A solution of DMF and H_2O (7:1, 8.0 mL) was added to the test tube and the mixture was stirred at rt until the starting material had been consumed as judged by ESI^+ MS analysis (ca. 4 hr). EtOAc (20 mL) and brine (20 mL) was added and the mixture was transferred to a separatory funnel. The layers were separated and the organic layer was washed with NH_4OH (5 mL) to potentially remove any excess copper. The combined aqueous layers were than extracted with EtOAc (20 mL). The organics layer was dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*.

The crude material was purified by flash chromatography on silica gel to afford 230 mg (74%) of the title compound as a pale yellow-pink solid: mp = 68–72 °C. $[\alpha]^{23}_{D}$ –38.8 (*c* 0.8, CH_2Cl_2). ^{1}H NMR (700 MHz, $CDCl_3$) δ 8.66 (s, 1 H), 7.25 (d, *J* = 8.4 Hz, 2 H), 7.01 (d, *J* = 9.1 Hz, 2 H), 5.44–5.41 (m, 1 H), 5.06–5.03 (m, 1 H), 4.37–4.34 (m, 1 H), 3.80–3.77 (m, 1 H), 3.66 (ddd, *J* = 2.8, 4.9, 11.2 Hz, 1 H), 3.44 (dd, *J* = 2.8, 9.8 Hz, 1 H), 2.31 (dd, *J* = 9.8, 16.8 Hz, 1 H), 2.26–2.13 (m, 3 H), 2.10 (s, 3 H), 2.06 (s, 3 H), 2.09–2.03 (m, 2 H), 1.91–1.86 (m, 2 H), 1.77 (dd, *J* = 7.0, 13.3 Hz, 1 H), 1.65–1.58 (m, 2 H), 0.88 (t, *J* = 7.0 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 207.5, 168.7, 154.2, 137.2, 136.7, 134.9, 128.3, 123.8, 121.1, 58.5, 53.7, 52.8, 47.6, 31.1, 30.8, 30.2, 29.4, 24.0, 20.8, 14.0 (one carbon signal is absent due to incidental equivalence); IR (film) 3261, 1711, 1687, 1621 cm^{-1}. MS (ESI) 398.2439 (398.2438 calcd for $C_{23}H_{31}N_3O_3$, M + H$^+$).



(–)-(3*S*,4a*S*,7*R*)-7-(2-Oxopropyl)-3-pentylhexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (14). A flame-dried flask was cooled under vacuum and charged with **13** (100 mg, 0.25 mmol) and Pd/C (10 mg). The flask was capped with a rubber septum, was briefly flushed with hydrogen and then a hydrogen-filled balloon attached to a needle (via an adaptor) was connected to the flask through the septum. Methanol (2.5 mL) was added to the flask and the mixture was stirred at rt until the starting material had been consumed as judged by ESI$^+$ MS analysis (ca. 45 min). The crude product was then filtered through a plug of celite to remove the Pd/C and washed with methanol (5 mL). The crude material was concentrated *in vacuo* and carried on to the next step without further purification. The crude product was dissolved in CH_3CN (10 mL) and transferred to a round-bottom flask charged with a stirbar. A solution of ceric ammonium nitrate (123 mg, 0.75 mmol) in H_2O (30 mL) was added to the reaction flask and the mixture was stirred at rt for 5 min. The mixture was then heated at 50 °C for 4 hr before being cooled to rt, at which time EtOAc (25 mL) was added. The mixture was transferred to a separatory funnel and the layers were separated. The organic layer was washed with saturated aqueous Na_2SO_3 (15 mL), saturated aqueous $NaHCO_3$ (15 mL), and brine (15 mL). The organic layer was dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel to afford 34 mg (51%) of the title compound as a white solid: mp = 84–88 °C. $[\alpha]^{23}_{D}$ –11.7 (*c* 2.5, CH_2Cl_2). ^{1}H NMR (700 MHz, $CDCl_3$) δ 4.79 (s, 1 H), 4.30–4.28 (m, 1 H), 3.48–3.45 (m, 1 H), 3.43–3.39 (m, 2 H), 2.29 (dd, *J* = 9.8, 16.8 Hz, 1 H),

2.10 (s, 3 H), 2.03–1.98 (m, 2 H), 1.95–1.94 (m, 1 H), 1.72 (dd, *J* = 7.7, 12.6 Hz, 1 H), 1.54–1.46 (m, 3 H), 1.38–1.25 (m, 7 H), 0.88 (t, *J* = 7.0 Hz, 3 H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 207.7, 155.0, 52.8, 52.8, 50.0, 47.6, 37.8, 32.9, 31.6, 30.6, 30.3, 29.0, 25.5, 22.6, 14.0; IR (film) 3207, 1709, 1649 cm^{-1}. MS (ESI) 267.2065 (267.2067 calcd for $C_{15}H_{26}N_2O_2$, M + H$^+$).

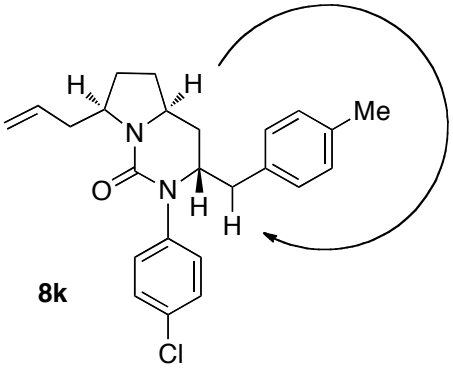


(–)-9-*epi*-Batzelladine K (16). A flame-dried flask was cooled under vacuum and charged with **14** (25 mg, 0.09 mmol) and dichloromethane (0.9 mL). 2,6-di-*tert*-butylpyridine (203 µL, 0.94 mmol) and MeOTf (103 µL, 0.94 mmol) were added and the mixture was stirred at rt until the starting material had been consumed as judged by ESI$^+$ MS analysis (ca. 1 hr). The solvent was then removed in a hood by blowing a constant stream of N_2 over the stirring mixture. The solution was then poured in diethyl ether (20 ml) and washed with 1 M NaOH (10 mL) and brine (10 mL). The organic layer was dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was used without further purification. The crude *O*-methylisourea was dissolved in methanol (2 mL) and transferred to a thick walled glass vial at which time ammonium chloride (10.1mg, 0.19 mmol) was added to this solution. Anhydrous ammonia was bubbled through this solution for ~15 min before the reaction vessel was sealed and heated to 60 ^{o}C overnight (14 hr). The reaction was cooled to rt and concentrated *in vacuo.* The crude guanidine product **15** was used without further purification. Crude product **15** was dissolved in methanol (3 mL), $NaCNBH_3$ (59 mg, 0.94 mmol) was added and the mixture was heated to 50 ^{o}C until the starting material had been consumed as judged by ESI$^+$ MS analysis (ca. 12 hr). The reaction mixture was cooled to rt and concentrated *in vacuo*. The crude product was dissolved in methylene chloride (20 mL), the mixture was transferred to a separatory funnel and washed with 2 M HCl (2 x 10 mL) and brine (1 x 10 mL). The organic layer was dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was determined to be a 3:1 mixture of diastereomers by ^{1}H NMR analysis. The crude material was purified by flash chromatography on silica gel to afford 13 mg (48%) of the title compound as a pale yellow oil. The following data is for the pure isolated major diastereomer. $[\alpha]^{23}_D$ –43.8 (*c* 0.5, CH_2Cl_2). ^{1}H NMR (700 MHz, $CDCl_3$) δ 3.80–3.73 (m, 2 H), 3.58–3.53 (m, 1 H), 3.52–3.49 (m, 1 H), 2.26–2.21 (m, 3 H), 2.19 (dd, *J* = 4.2, 13.3 Hz, 1 H), 1.73–1.64 (m, 2 H), 1.60–1.56 (m, 2 H), 1.52–1.47 (m, 1 H), 1.44–1.27 (m, 7 H), 1.27 (d, *J* = 6.3 Hz, 3 H), 0.93 (t, *J* = 7.0 Hz, 3 H);

^{13}C NMR (175 MHz, $CDCl_3$) δ 149.4, 56.3, 51.6, 48.4, 45.8, 36.2, 35.5, 31.5, 31.2, 30.5, 30.2, 25.5, 22.4, 20.5, 14.0; ^{1}H NMR (700 MHz, CD_3OD) δ 7.56 (d, *J* = 7.5 Hz, 2 H), 7.34 (d, *J* = 8.5 Hz, 2 H), 5.57–5.52 (m, 1 H), 5.10–5.05 (m, 1 H), 4.64 (s, 1 H), 3.99–3.90 (m, 2 H), 3.77–3.73 (m, 1 H), 3.69–3.65 (m, 1 H), 2.46–2.32 (m, 5 H), 2.28 (dt, *J* = 3.5, 13.0 Hz, 1 H), 1.99–1.93 (m, 3 H), 1.88–1.80 (m, 2 H), 1.51–1.44 (m, 1 H), 1.22 (d, *J* = 6.5 Hz, 3 H), 0.93 (t, *J* = 7.5 Hz, 3 H); ^{13}C NMR (175 MHz, CD_3OD) δ 150.4, 57.5, 53.5, 50.2, 47.3, 36.8, 36.1, 32.7, 31.9, 31.3, 30.7, 26.8, 23.6, 20.8 14.3; IR (film) 3284, 3202, 1637 cm^{-1}. MS (ESI) 250.2278 (250.2278 calcd for $C_{15}H_{28}N_3$, M^+).

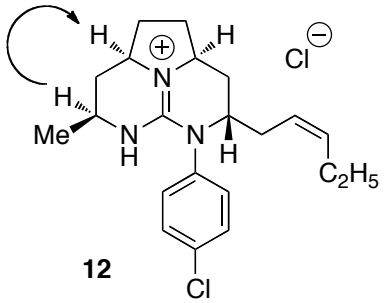
Assignment of Stereochemistry

The relative stereochemistry of compound **8k** was assigned on the basis of observed ^{1}H NMR nOe experiments. Significant nOe relationships are shown below. The stereochemistry of all other bicyclic urea products was assigned based on analogy to **8k**.

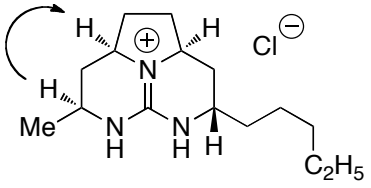


8k

The relative stereochemistry of compounds **12** and **16** were assigned on the basis of observed ^{1}H NMR nOe experiments. Significant nOe relationships are shown below.



12

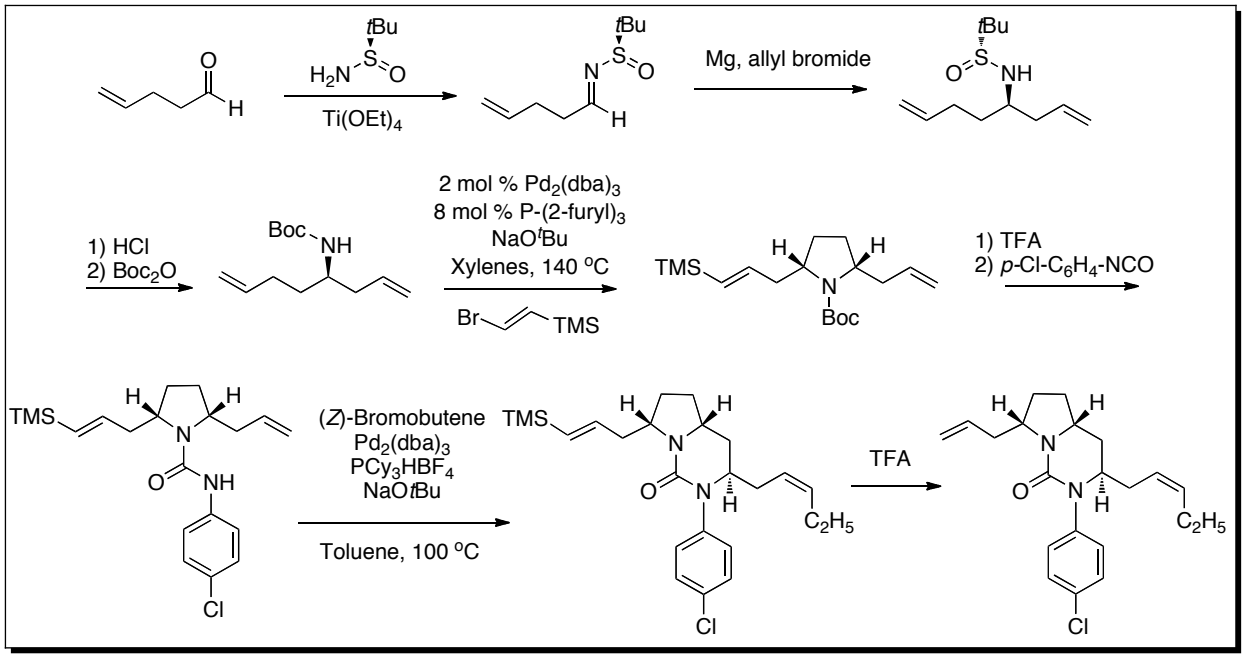


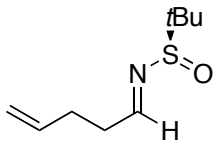
9-*epi*-Batzelladine K (**16**)

The absolute stereochemistry of the urea products was assigned via the synthesis of compound ***ent*-8c** from pent-4-enal via the route illustrated below in Scheme S1. The optical rotation of product ***ent*-8c** prepared via this route was opposite that of the product **8c** generated in the Pd-catalyzed carboamination reaction between **7c** and Z-bromobutene. In addition, analysis of

product ***ent*-8c** by chiral HPLC indicated that ***ent*-8c** was the enantiomer of product **8c** formed in the catalytic reaction.

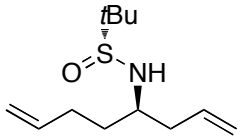
Scheme S1



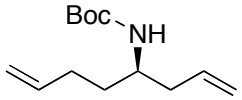


(–)-(R_S)-2-Methyl-*N*-(pent-4-en-1-ylidene)propane-2-sulfinamide (S4). This compound was prepared according to the procedure reported by Ellman.[4] A flame-dried flask was cooled under a stream of N_2 and charged with pent-4-enal (1.38 mL, 14 mmol) and THF (40 mL). Titanium ethoxide (4.2 mL, 20 mmol) was added and the reaction mixture was stirred at rt for 5 min. (*R*)-*tert*-butanesulfinamide (1.21 g, 10 mmol) was added in one portion and the mixture was stirred overnight (ca. 14 h) at rt. The reaction mixture was poured into brine (40 mL) and stirred for 10 min. Ethyl acetate (20 mL) was added, the mixture was filtered through celite and the celite was washed with ethyl acetate (50 mL). The mixture was transferred to a separatory funnel, brine (20 mL) was added, and the layers were separated. The aqueous phase was extracted with ethyl acetate (2 x 30 mL). The combined organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel to afford 1.38 g (74%) of the title compound as a colorless oil. Spectroscopic properties are identical to those previously reported.[5] ^{1}H NMR (500 MHz, $CDCl_3$)

δ 8.08 (t, *J* = 4.5 Hz, 1 H), 5.84 (ddt, *J* = 4.5, 10.0, 17.0 Hz, 1 H), 5.08 (dd, *J* = 1.5, 17.0 Hz, 1 H), 5.02 (dd, *J* = 1.5, 10.0 Hz, 1 H), 2.63 (td, *J* = 4.0, 7.5 Hz, 2 H), 2.40 (q, *J* = 7.0 Hz, 2 H), 1.19 (s, 9 H).

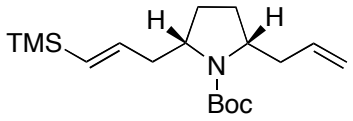


(R_S, 4*R*)-2-Methyl-*N*-(octa-1,7-dien-4-yl)propane-2-sulfinamide (S5). A flame-dried flask was cooled under a stream of N_2 and charged with freshly ground magnesium turnings (720 mg, 4 equiv). The magnesium was suspended in ether (14.8 mL, 1 M), cooled to 0 °C in an ice/water bath and allyl bromide (1.28 mL, 14.8 mmol) was added dropwise. After addition, the ice bath was removed, and the reaction mixture was stirred at rt for 30 min. Stirring was stopped and the solution was filtered through glass wool prior to addition to **S4**. A flame-dried flask was cooled under a stream of N_2 and charged with **S4** (1.38 g, 7.4 mmol) and THF (37 mL, 0.2 M). The sulfinyl imine solution was cooled to 0 °C in an ice/water bath before the filtered Grignard reagent solution was added dropwise. The reaction mixture was stirred at 0 °C until the starting material had been completely consumed as judged by TLC analysis (1 h). Water was then added dropwise until precipitation of magnesium salts occurred and the resulting solution was decanted into a separate flask. The solution was dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. Analysis of the crude product by ^{1}H NMR indicated that a 10:1 mixture of diastereomers had formed. The crude material was purified by flash chromatography on silica gel to afford 1.02 g (60%) of the title compound as a 10:1 mixture of diastereomers as a clear colorless oil. Data are for the major isomer. ^{1}H NMR (500 MHz, $CDCl_3$) d 5.83–5.74 (m, 2 H), 5.18–4.97 (m, 4 H), 3.36–3.32 (m, 1 H), 3.21 (d, *J* = 6.5 Hz, 1 H), 2.45–2.40 (m, 1 H), 2.37–2.32 (m, 1 H), 2.18–2.08 (m, 2 H), 1.62–1.58 (m, 2 H), 1.21 (s, 9 H).

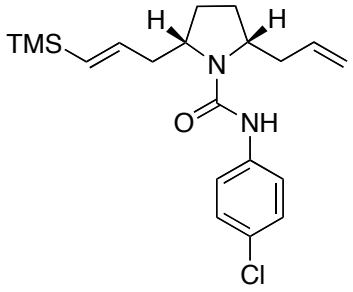


(*R*)-*tert*-Butyl octa-1,7-dien-4-ylcarbamate (S1). A flame-dried flask was cooled under a stream of N_2 and charged with **S5** (1.02 g, 4.4 mmol) and methanol (22 mL). A solution of anhydrous hydrochloric acid (4.4 mL, 17.7 mmol, 4 M in dioxane) was added and the mixture was stirred at rt for 1 h, at which time TLC analysis indicated that the starting material had been

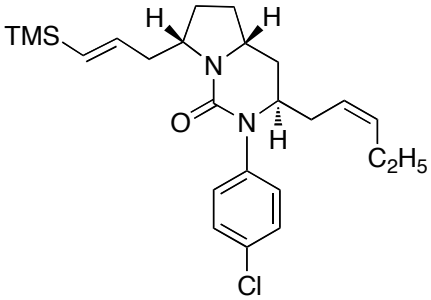
completely consumed. The reaction mixture was diluted with water (10 mL) and CH_2Cl_2 (10 mL), basified with NH_4OH to pH > 12, and transferred to a separatory funnel. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3 x 5 mL). The combined organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude product was dissolved in THF (44 mL, 0.1 M), solid di-*tert*-butyldicarbonate (1.2 g, 5.3 mmol) was added and the reaction mixture was stirred at rt for 3 h. 1 M NaOH (5 mL) was added and the resulting biphasic mixture was stirred for 1 h at rt. The mixture was transferred to a separatory funnel, the layers were separated and the aqueous layer was extracted with ethyl acetate (3 x 20 mL). The combined organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel to afford 941 mg (94%) of the title compound as a clear colorless oil. The spectroscopic properties of this compound were identical to that of compound **(±)-S1** described above.



(*E*,2*R*,5*S*)-*tert*-Butyl 2-allyl-5-[3-(trimethylsilyl)allyl]pyrrolidine-1-carboxylate (S2). A flame-dried Schlenk flask was cooled under a stream of N_2 and charged with $Pd_2(dba)_3$ (77 mg, 0.084 mmol), tri(2-furyl)phosphine (77 mg, 0.33 mmol) and NaO*t*Bu (802 mg, 8.4 mmol). The flask was purged with N_2, then a solution of **(*R*)-S1** (941 mg, 4.2 mmol) in freshly distilled xylenes (21 mL) was added via syringe and the resulting mixture was stirred at rt for 2 min. (*E*)-(2-bromovinyl)trimethylsilane (1.28 mL, 8.4 mmol) was added and the flask was heated to 140 °C and stirred for 3 h. The mixture was cooled to room temperature and saturated aqueous NH_4Cl (10 mL) and ethyl acetate (10 mL) were added. The layers were separated, the organic layer was filtered through a plug of silica gel, and the silica gel was washed with ethyl acetate (20 mL). The filtrate was dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel to afford 647 g (48%) of the title compound as a dark brown oil. The spectroscopic properties of this compound were identical to that of compound **(±)-S2** described above.

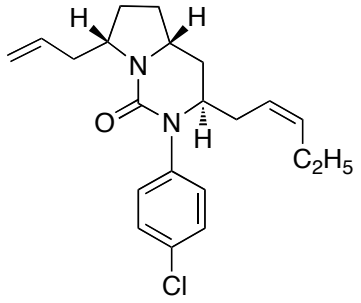


(*E*,2*R*,5*S*)-2-Allyl-*N*-(4-chlorophenyl)-5-[3-(trimethylsilyl)allyl]pyrrolidine-1-carboxamide (S6). A round-bottom flask equipped with a stirbar was charged with **(*E*,2*R*,5*S*)-S2** (647 mg, 2.0 mmol) and dichloromethane (20 mL, 0.1 M). Trifluoroacetic acid (2.0 mL, 1.0 M) was added to the flask and the mixture was stirred for 20 min at rt. The solution was diluted with water, basified with NH_4OH to pH > 12, and transferred to a separatory funnel. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3 x 5 mL). The combined organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude product was dissolved in dichloromethane (20 mL, 0.1 M) and 4-chlorophenyl isocyanate (369 mg, 1.2 equiv) was added. The reaction mixture was stirred at rt for 1 h until starting material had been completely consumed as judged by TLC analysis. The crude reaction mixture was concentrated *in vacuo*, and purified by flash chromatography on silica gel to afford 244 mg (32%) of the title compound as a orange brown oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 (d, *J* = 9.0 Hz, 2 H), 7.22 (d, *J* = 9.0 Hz, 2 H), 6.41 (s, 1 H), 6.04 (dt, *J* = 7.0, 18.5 Hz, 1 H), 5.93–5.84 (m, 1 H), 5.82 (d, *J* = 18.5 Hz, 1 H), 5.22–5.17 (m, 2 H), 4.02–3.95 (m, 2 H), 2.61–2.52 (m, 2 H), 2.35 (dt, *J* = 7.0, 13.5 Hz, 1 H), 2.24 (dt, *J* = 7.5, 14.0 Hz, 1 H), 2.02–1.96 (m, 2 H), 1.80–1.74 (m, 2 H), 0.05 (s, 9 H).



(*E*,*Z*,3*R*,4a*R*,7*S*)-2-(4-Chlorophenyl)-3-(pent-2-en-1-yl)-7-[3-(trimethylsilyl)allyl]hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (S7). A flame-dried Schlenk tube was cooled under vacuum and charged with $Pd_2(dba)_3$ (3.1 mg, 0.003 mmol), $PCy_3{\cdot}HBF_4$ (5.0 mg, 0.014 mmol) and NaO*t*Bu (25 mg, 0.26 mmol). The flask was evacuated and purged with N_2. A solution of **S6** (65 mg, 0.17 mmol) in toluene (0.85 mL) was added via syringe and

the resulting mixture was stirred at rt for 2 min. (*Z*)-1-bromobut-1-ene (130 µL, 0.26 mmol, 2.0 M solution in toluene) was added and the tube was heated to 100 °C and stirred until the starting material was completely consumed as judged by TLC analysis (1 h). The mixture was cooled to room temperature and saturated aqueous NH_4Cl (1 mL) and ethyl acetate (1 mL) were added. The layers were separated, the organic layer was filtered through a plug of silica gel, and the silica gel was washed with ethyl acetate (1 mL). The filtrate was dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel to afford 53 mg (71%) of the title compound as a yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 7.31 (d, *J* = 9.0 Hz, 2 H), 7.19 (d, *J* = 9.0 Hz, 2 H), 5.94 (ddd, *J* = 6.0, 7.5, 18.5 Hz, 1 H), 5.68 (d, *J* = 18.5 Hz, 1 H), 5.47–5.42 (m, 1 H), 5.12–5.07 (m, 1 H), 4.03 (dt, *J* = 2.5, 8.5 Hz, 1 H), 3.90 (dt, *J* = 4.5, 9.5 Hz, 1 H), 3.66 (ddt, *J* = 2.5, 5.0, 11.5 Hz, 1 H), 2.73 (dd, *J* = 5.5, 12.5 Hz, 1 H), 2.27–2.16 (m, 4 H), 2.01–1.89 (m, 4 H), 1.84–1.81 (m, 1 H), 1.69–1.61 (m, 2 H), 0.90 (t, *J* = 7.5 Hz, 3 H), 0.03 (s, 9 H).



(+)-(*Z*,3*R*,4a*R*,7*S*)-7-Allyl-2-(4-chlorophenyl)-3-(pent-2-en-1-yl)hexahydropyrrolo[1,2-*c*]pyrimidin-1(2*H*)-one (*ent*-8c). A Schlenk tube was charged with **S7** (53 mg, 0.12 mmol) and CH_2Cl_2 (1.2 mL). TFA (0.6 mL) was added and the reaction mixture was stirred overnight at 40 °C. The reaction mixture was then cooled to rt, diluted with water (1 mL), and basified with NH_4OH to pH > 12. The reaction mixture was transferred to a separatory funnel and the layers were separated. The organic layer was dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude material was purified by flash chromatography on silica gel. This procedure afforded 29 mg (67%) of the title compound as a yellow oil: $[\alpha]^{23}_D$ +17.7 (*c* 2.9, CH_2Cl_2). The spectroscopic properties of this compound were identical to that of compound **8c**. The enantiopurity was determined to be 10:90 er by chiral HPLC analysis (chiralcel ADH, 25 cm x 4.6 mm, 5% IPA/Hexanes, 0.75 mL/min, λ 190 nm, RT= 13.4 and 17.8 min).

References

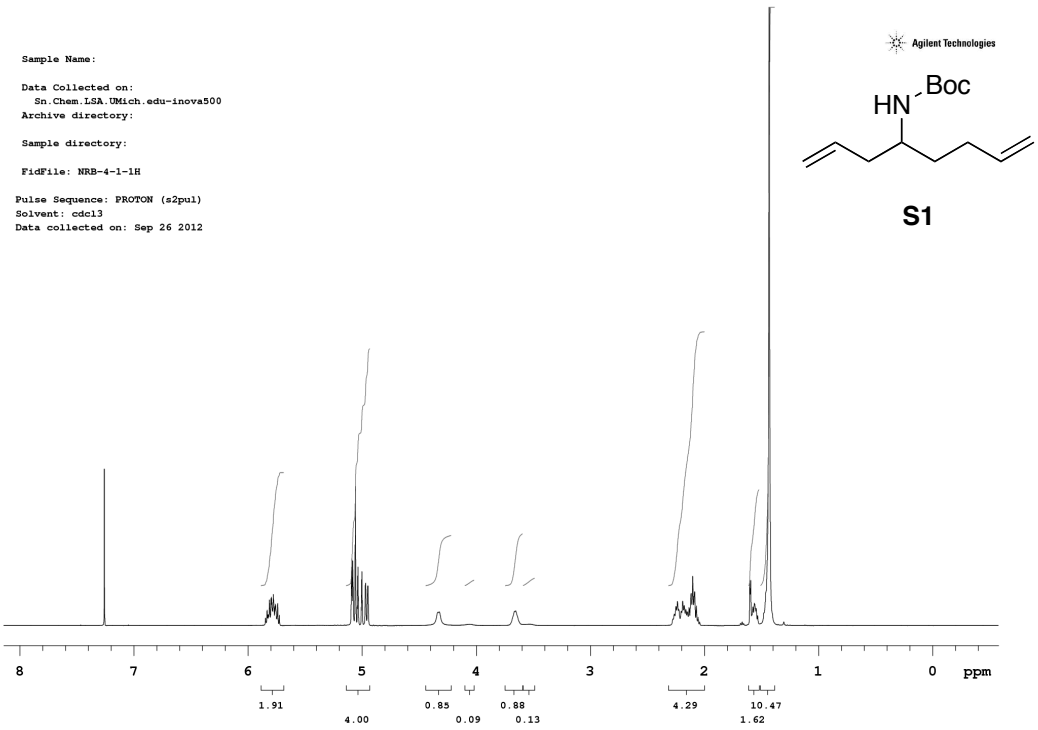
[1] H. Harada, R. K. Thalji, R. G. Bergman, J. A. Ellman, *J. Org. Chem.* **2008**, *73*, 6772–6779.

[2] S. Hanessian, A. Tehim, P. Chen, *J. Org. Chem.* **1993**, *58*, 7768–7781.

[3] S. J. Veenstra, P. Schmid, *Tetrahedron Lett.* **1997**, *38*, 997–1000.

[4] G. Liu, D. A. Cogan, T. D. Owens, T. P. Tang, J. A. Ellman, *J. Org. Chem.* **1999**, *64*, 1278–1284.

[5] N. R. Babij, J. P. Wolfe, *Angew. Chem.* **2012**, *124*, 4204–4206; *Angew. Chem. Int. Ed*. **2012**, *51*, 4128–4130.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Agilent Technologies
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Boc
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5
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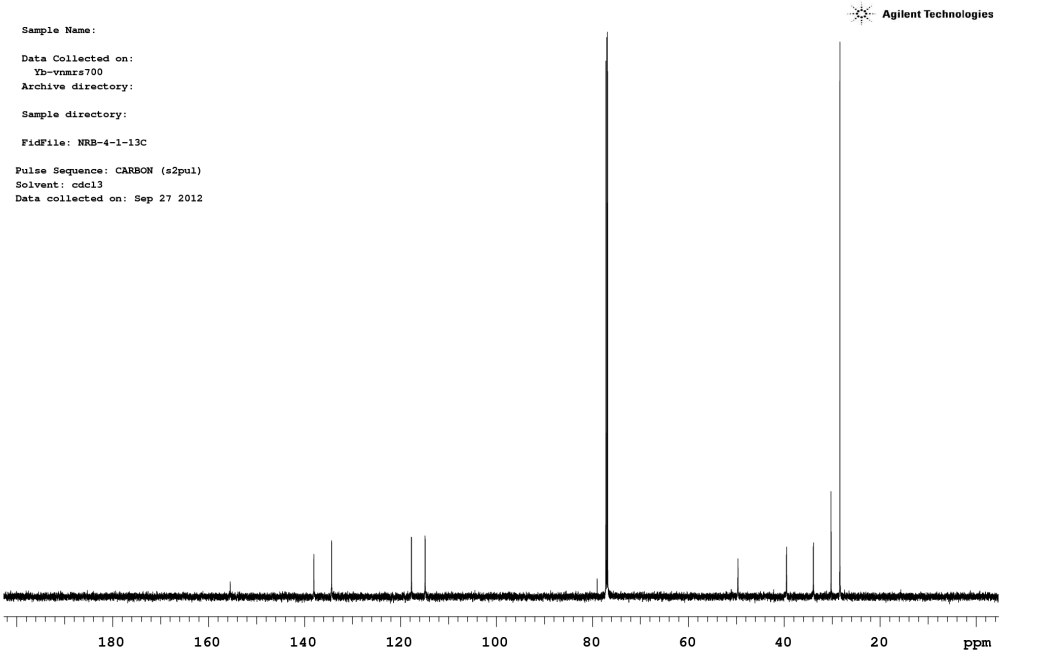
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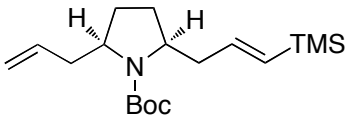
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S2

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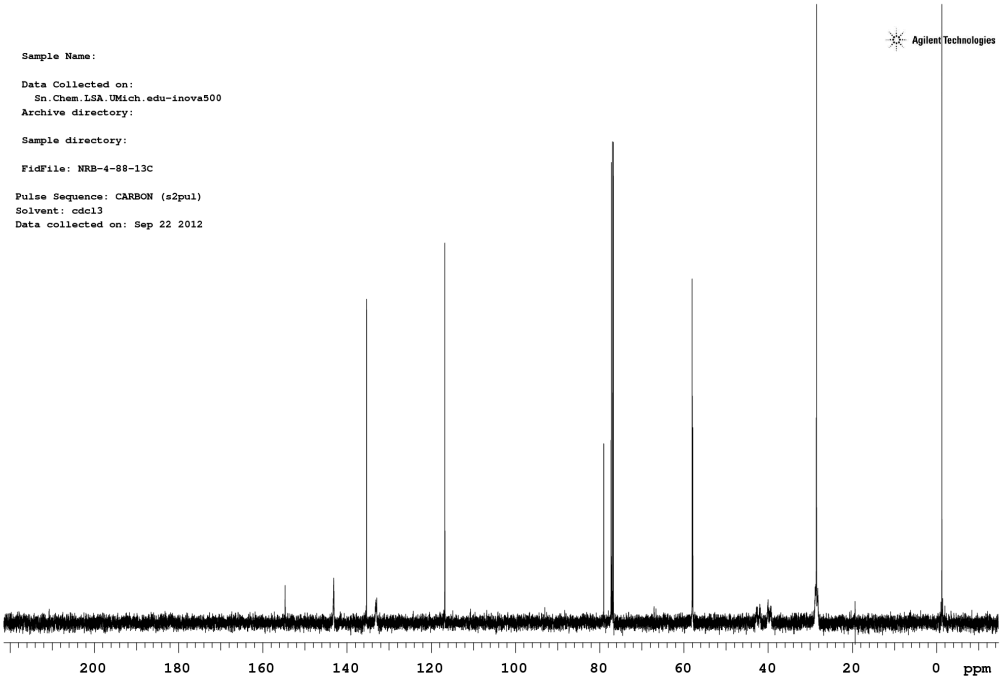
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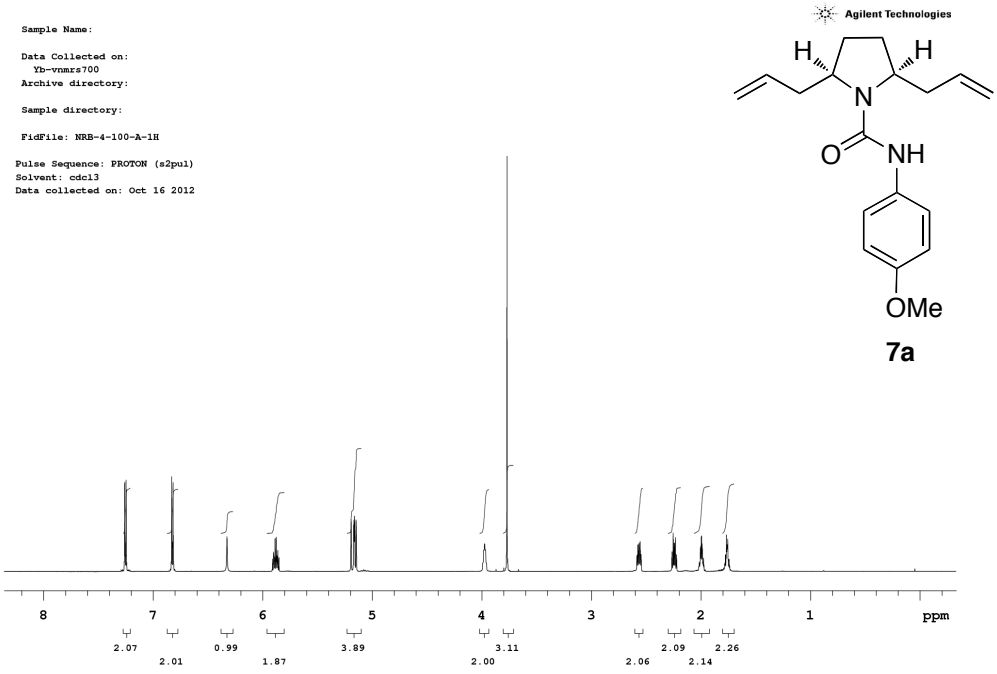
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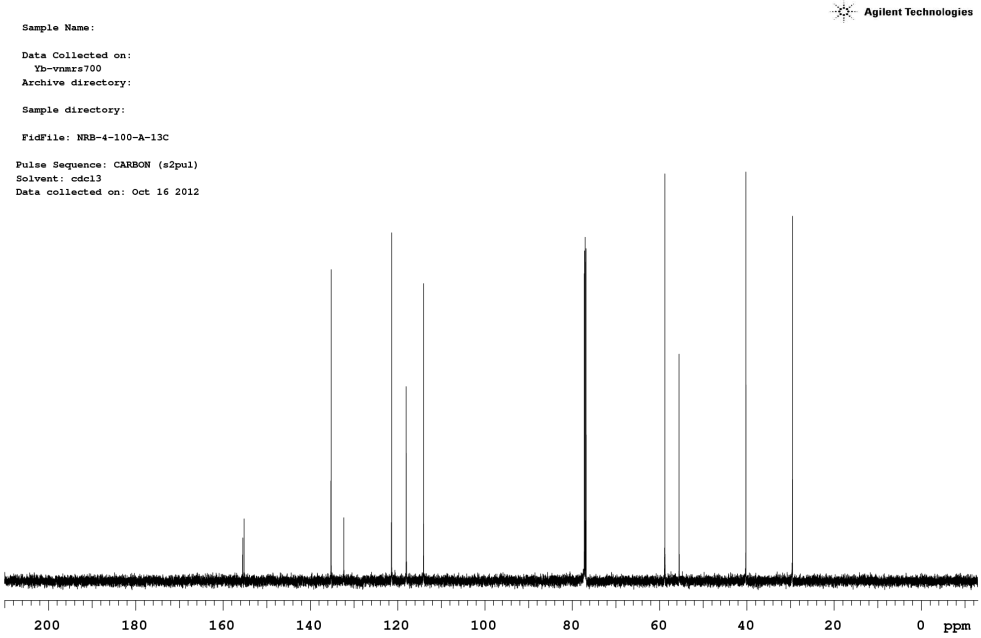
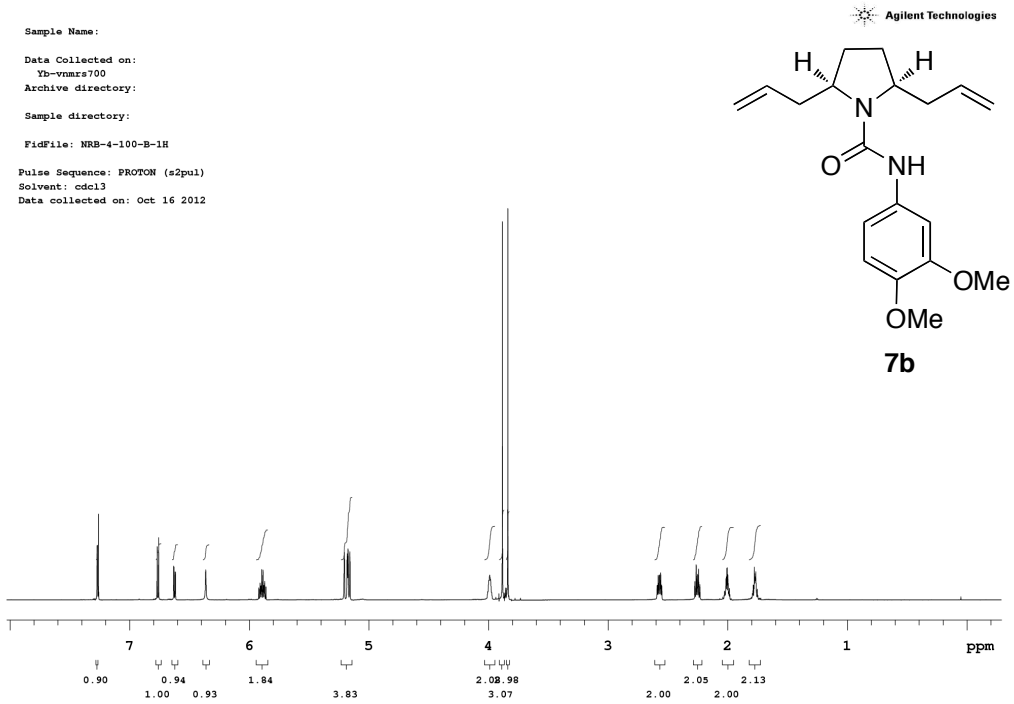
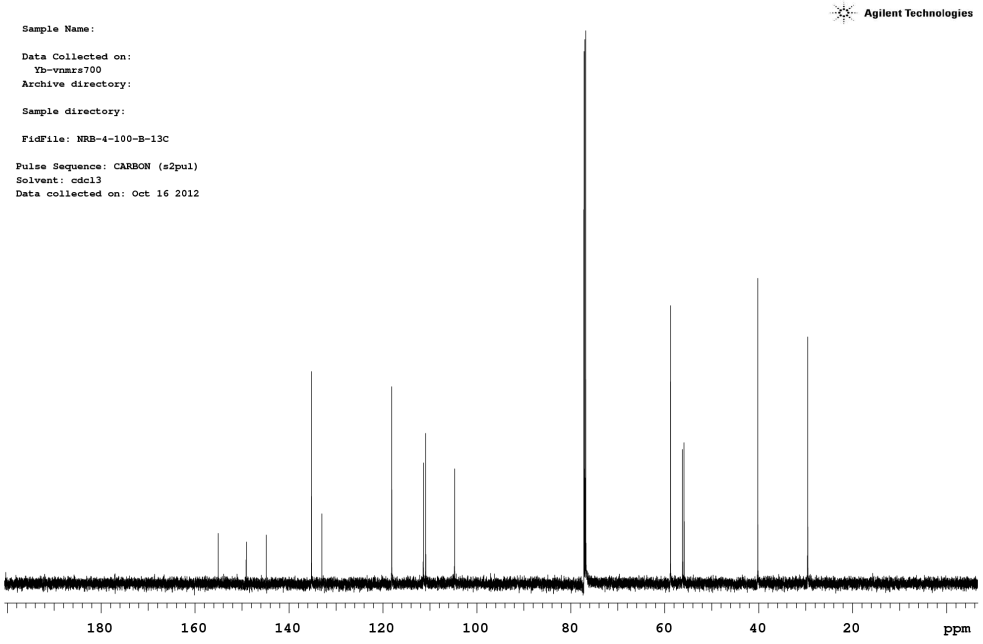
0.99
1.87
3.89
3.11
2.00
2.06
2.09
2.14
2.26


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-100-A-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 16 2012
200
180
160
140
120
100
80
60
40
20
0
ppm


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-100-B-1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 16 2012
H
H
N
O
NH
OMe
OMe
7
6
5
4
3
2
1
ppm
0.90
0.94
1.00
0.93
1.84
3.83
2.02
8.98
3.07
2.00
2.05
2.00
2.13

7b


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-100-B-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 16 2012
180
160
140
120
100
80
60
40
20
ppm

Agilent Technologies

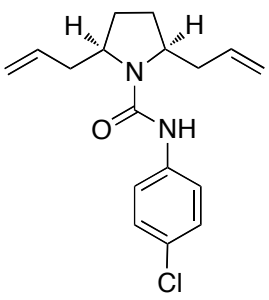
Sample Name:

Data Collected on:
Yb-vnmrs700
Archive directory:

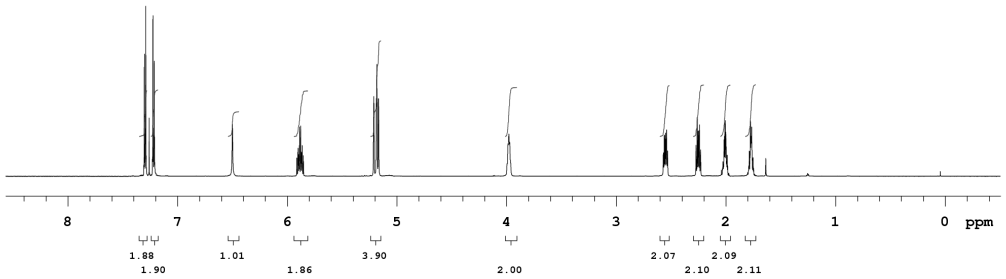
Sample directory:

FidFile: NRB-4-135-sm

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 21 2012



7c



Agilent Technologies

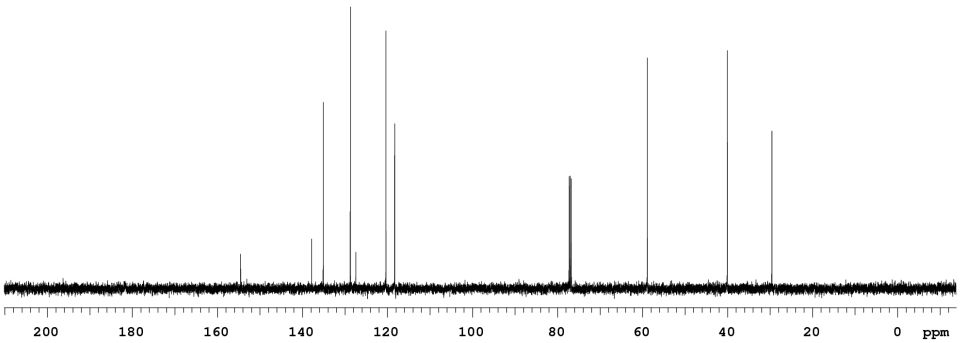
Sample Name:

Data Collected on:
Sn.Chem.LSA.UMich.edu-inova500
Archive directory:

Sample directory:

FidFile: NRB-4-79-sm-13C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 12 2012



Agilent Technologies

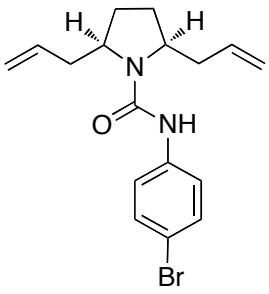
Sample Name:

Data Collected on:
Yb-vnmrs700
Archive directory:

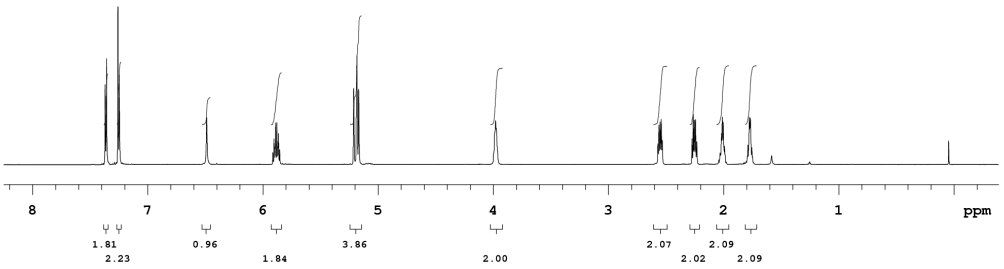
Sample directory:

FidFile: NRB-4-100-C

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 16 2012



7d



Agilent Technologies

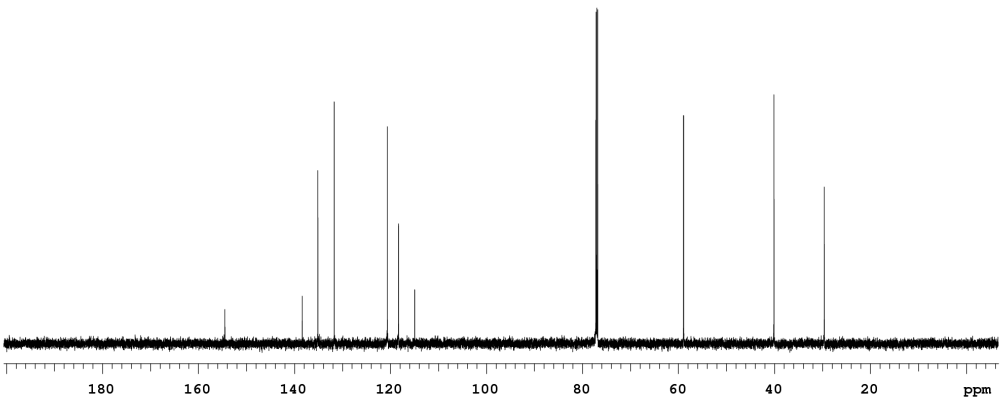
Sample Name:

Data Collected on:
Yb-vnmrs700
Archive directory:

Sample directory:

FidFile: NRB-4-100-C-13C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 16 2012



Agilent Technologies

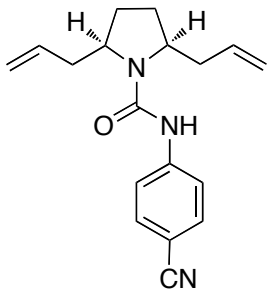
Sample Name:

Data Collected on:
Yb-vnmrs700
Archive directory:

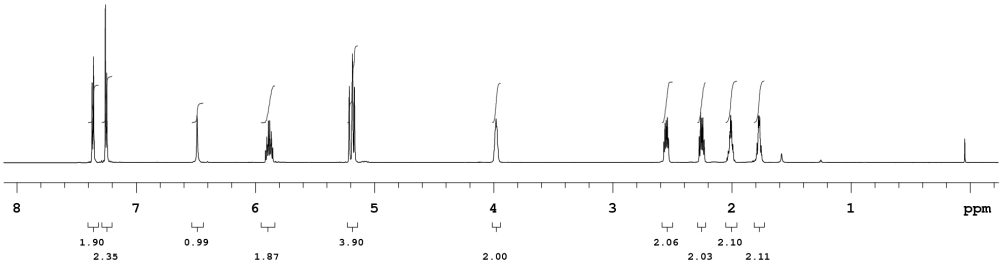
Sample directory:

FidFile: NRB-4-100-D-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 16 2012



7e



Agilent Technologies

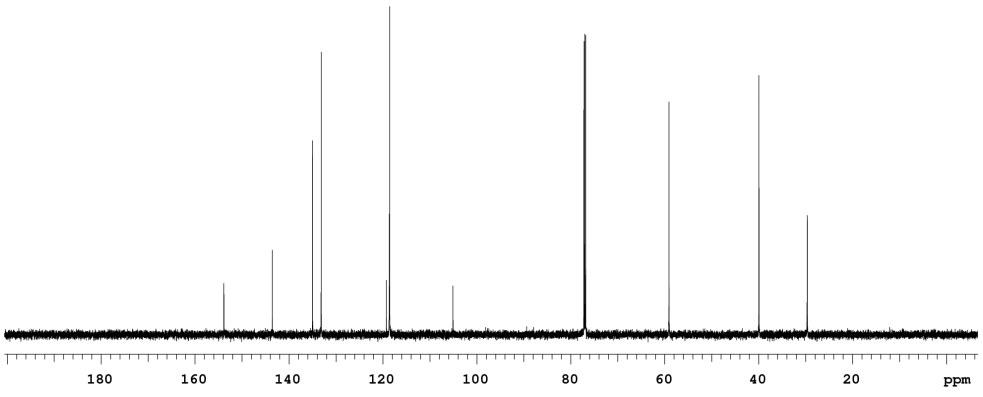
Sample Name:

Data Collected on:
Yb-vnmrs700
Archive directory:

Sample directory:

FidFile: NRB-4-100-D-13C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 16 2012



Agilent Technologies

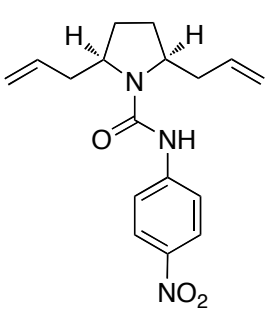
Sample Name:

Data Collected on:
Yb-vnmrs700
Archive directory:

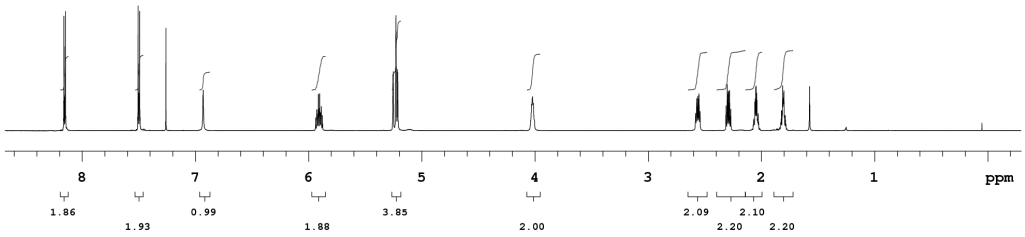
Sample directory:

FidFile: NRB-4-100-E-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 16 2012



7f



Agilent Technologies

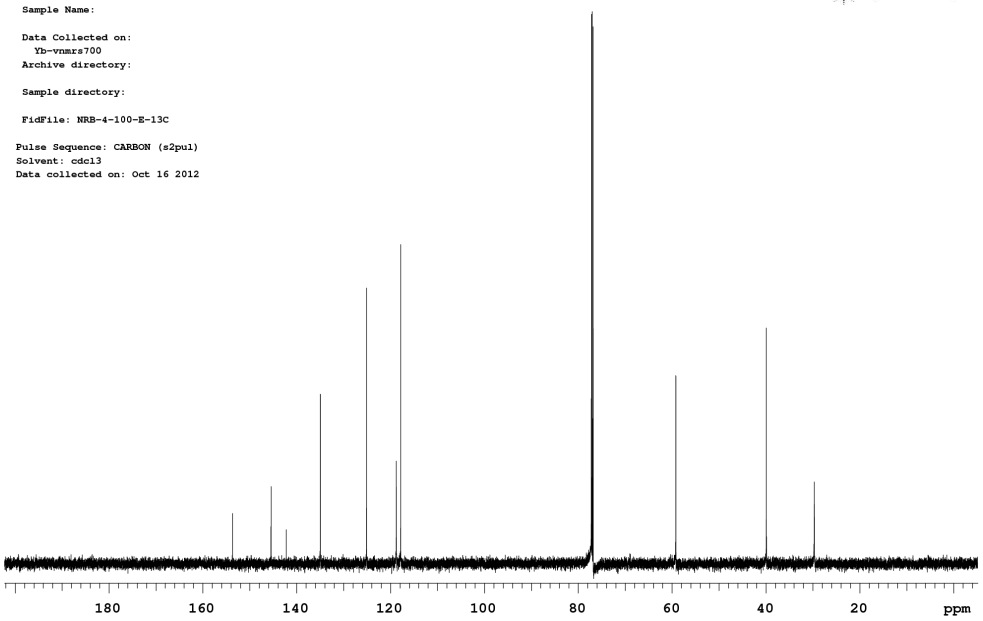
Sample Name:

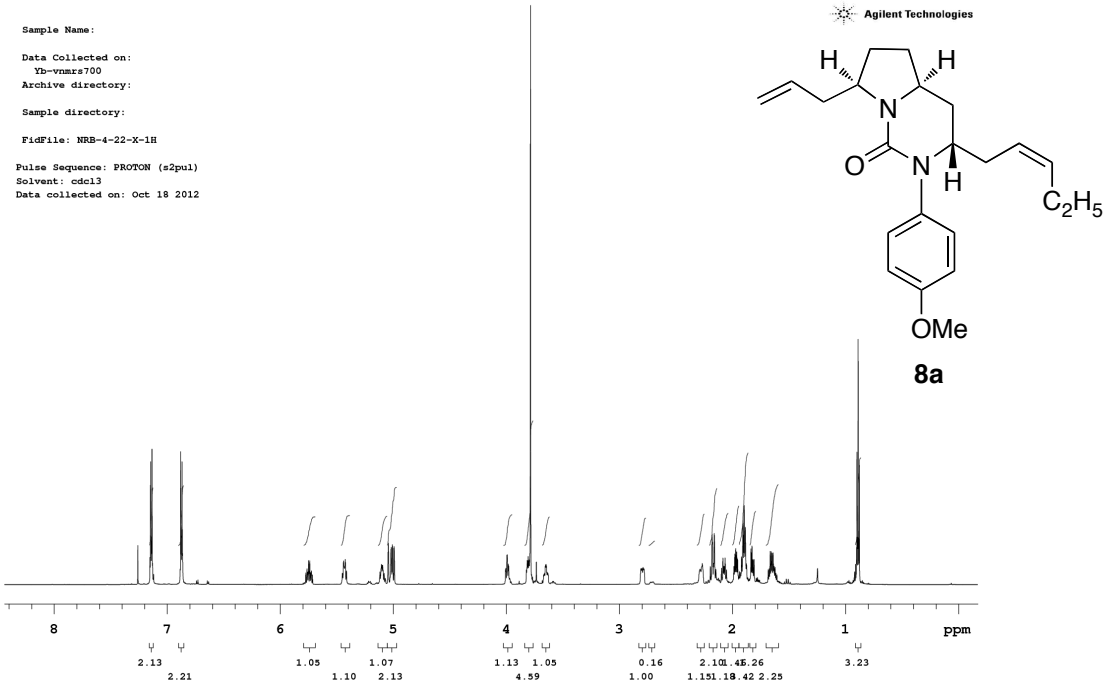
Data Collected on:
Yb-vnmrs700
Archive directory:

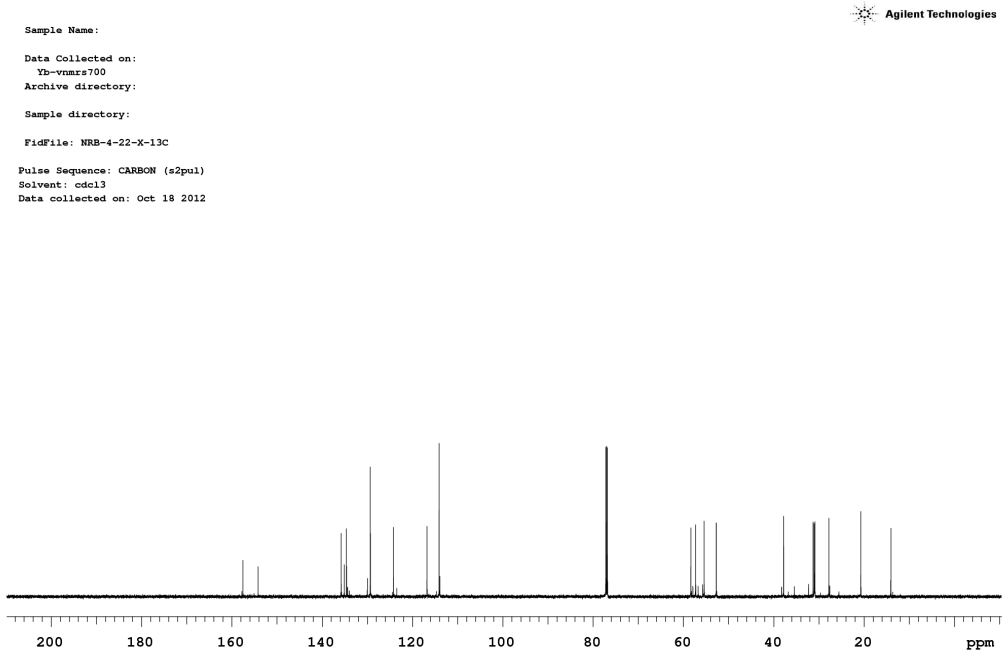
Sample directory:

FidFile: NRB-4-100-E-13C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 16 2012




Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-22-X-1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 18 2012
H
H
N
O
N
H
C_2H_5
OMe
8a
8
7
6
5
4
3
2
1
ppm
2.13
2.21
1.05
1.10
1.07
2.13
1.13
1.05
4.59
0.16
1.00
2.10
1.15
1.18
1.42
5.26
2.25
3.23


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-22-X-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 18 2012
200
180
160
140
120
100
80
60
40
20
ppm

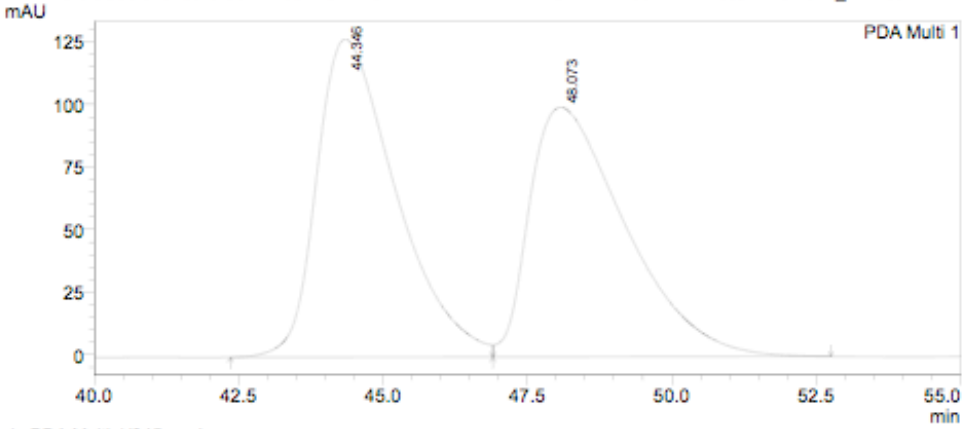
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-11-PMP-Z-BUTENE-100-ADH-2.5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-11-PMP-Z-BUTENE-100-ADH-2.5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-11-PMP-Z-BUTENE-100-ADH-2.5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/24/2012 1:59:11 PM
Data Processed	: 10/24/2012 2:54:56 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-11-PMP-Z-BUTENE-100-ADH-2.5%IPA-0.75ml_min.lcd

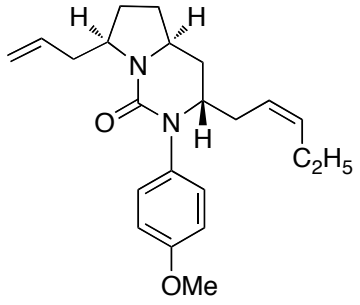


1 PDA Multi 1/245nm 4nm

PeakTable

PDA Ch1 245nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	44.346	12121126	126849	51.297
2	48.073	11508366	99562	48.703
Total		23629492	226411	100.000



8a

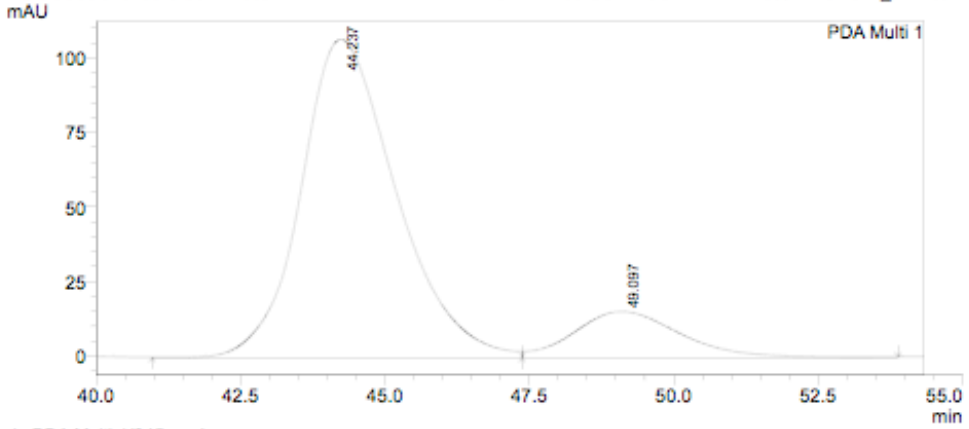
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-11-PMP-Z-BUTENE-90-SSIPHOSPE-ADH-2.5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-11-PMP-Z-BUTENE-90-SSIPHOSP-ADH-2.5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-11-PMP-Z-BUTENE-90-SSIPHOSPE-ADH-2.5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 5/2/2012 6:06:42 PM
Data Processed	: 5/2/2012 7:02:07 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-11-PMP-Z-BUTENE-90-SSIPHOSPE-ADH-2.5%IPA-0.75ml_min.lcd

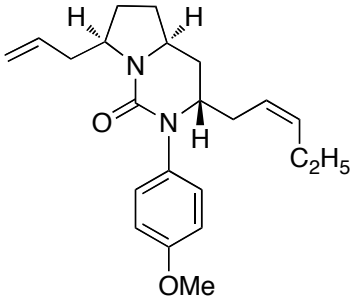


1 PDA Multi 1/245nm 4nm

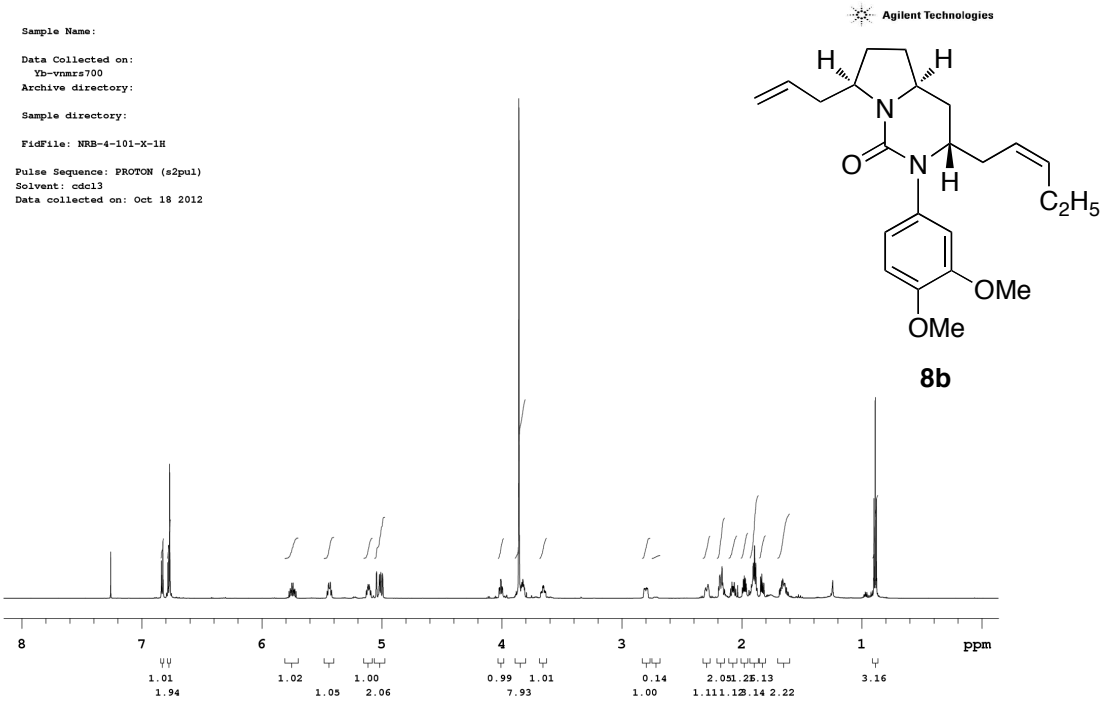
PeakTable

PDA Ch1 245nm 4nm

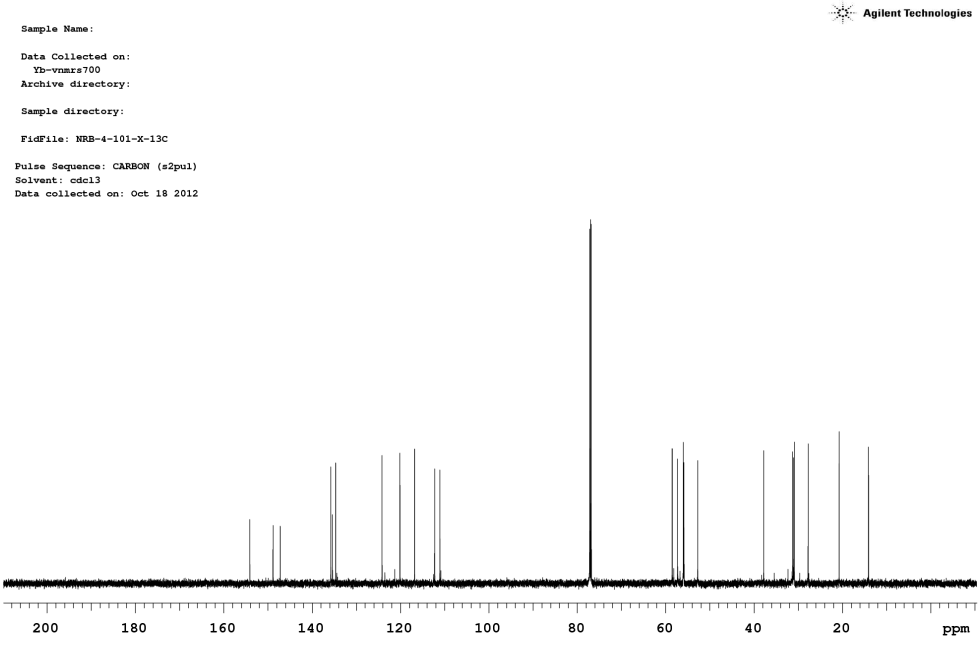
Peak#	Ret. Time	Area	Height	Area %
1	44.237	12187752	106584	86.224
2	49.097	1947258	15346	13.776
Total		14135011	121931	100.000



8a


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-101-X-1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 18 2012
H
H
N
O
N
H
C2H5
OMe
OMe
8
7
6
5
4
3
2
1
ppm
1.01
1.94
1.02
1.05
1.00
2.06
0.99
1.01
7.93
0.14
1.00
2.05
1.11
1.12
1.25
3.14
5.13
2.22
3.16

8b


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-101-X-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 18 2012
200
180
160
140
120
100
80
60
40
20
ppm

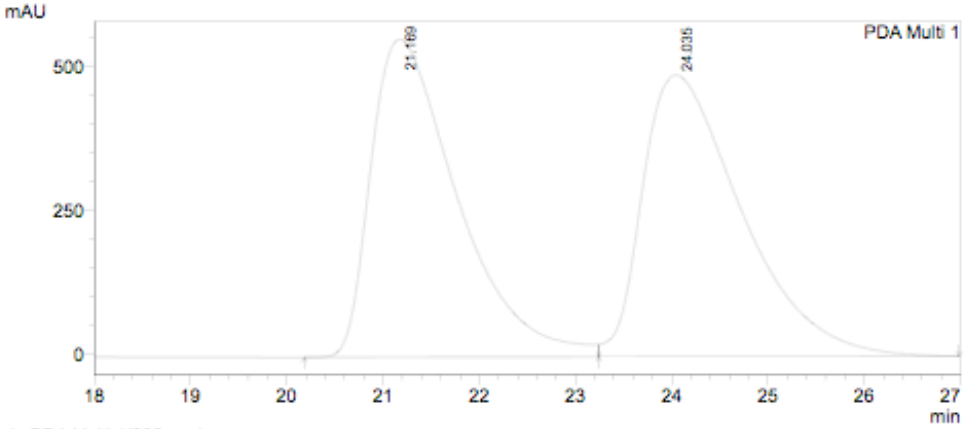
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-101-34PMP-Z-BUTENE-100-PCY3-ADH-5%IPA-0.75ml.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-101-3,4PMP-Z-Butene-100C-ADH-5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-101-34PMP-Z-BUTENE-100-PCY3-ADH-5%IPA-0.75ml.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/17/2012 3:45:42 PM
Data Processed	: 10/17/2012 4:18:20 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-101-34PMP-Z-BUTENE-100-PCY3-ADH-5%IPA-0.75ml.lcd

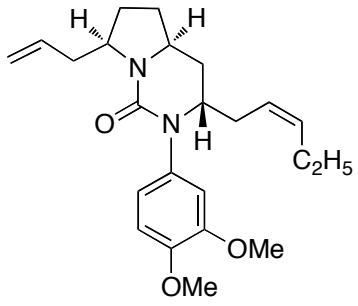


1 PDA Multi 1/205nm 4nm

PeakTable

PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	21.169	34368966	550494	49.582
2	24.035	34947914	487647	50.418
Total		69316880	1038141	100.000



8b

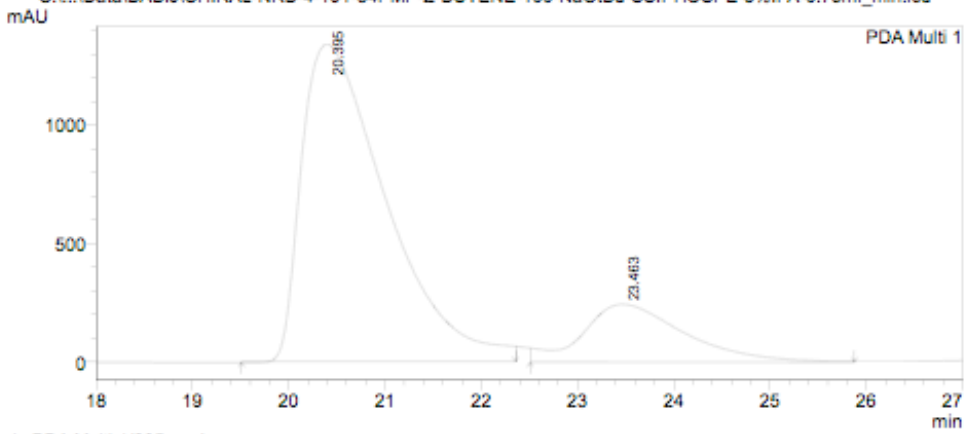
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-101-34PMP-Z-BUTENE-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-101-34PMP-Z-BUTENE-100-NaOtBu-SSIPHOSPE-5%IPA-0.75
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-101-34PMP-Z-BUTENE-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/17/2012 9:55:54 PM
Data Processed	: 10/17/2012 10:28:13 PM

<Chromatogram>

C:\...\Data\BABIJ\CHIRAL-NRB-4-101-34PMP-Z-BUTENE-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

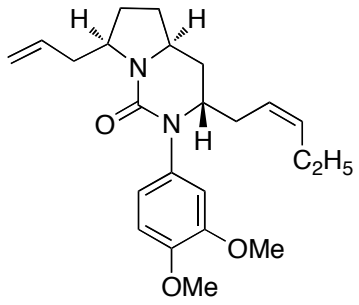


1 PDA Multi 1/205nm 4nm

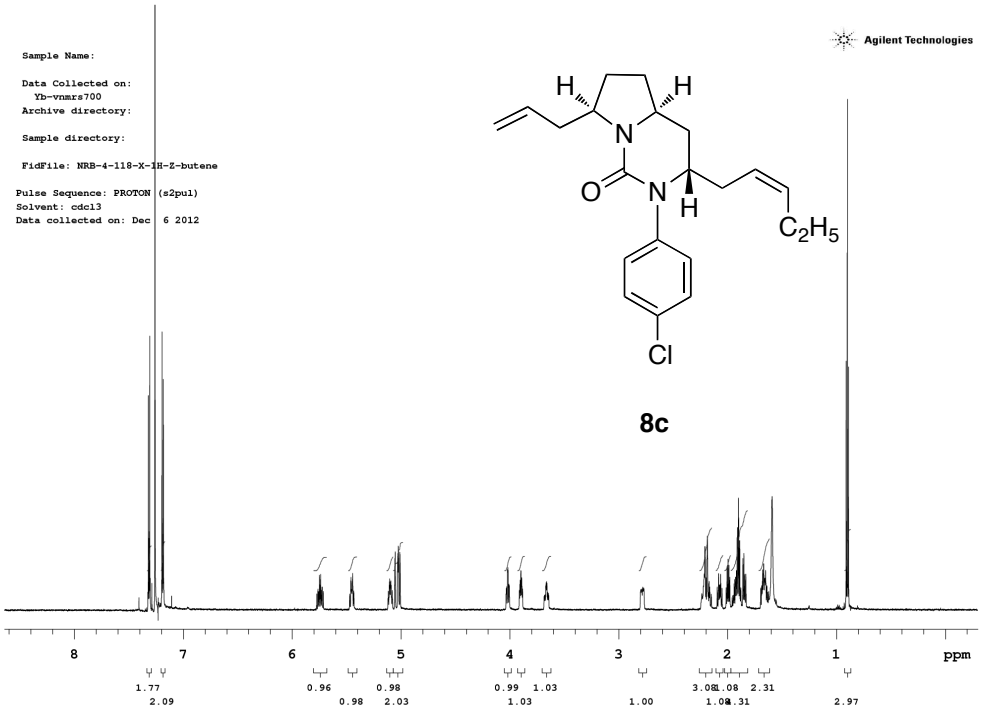
PeakTable

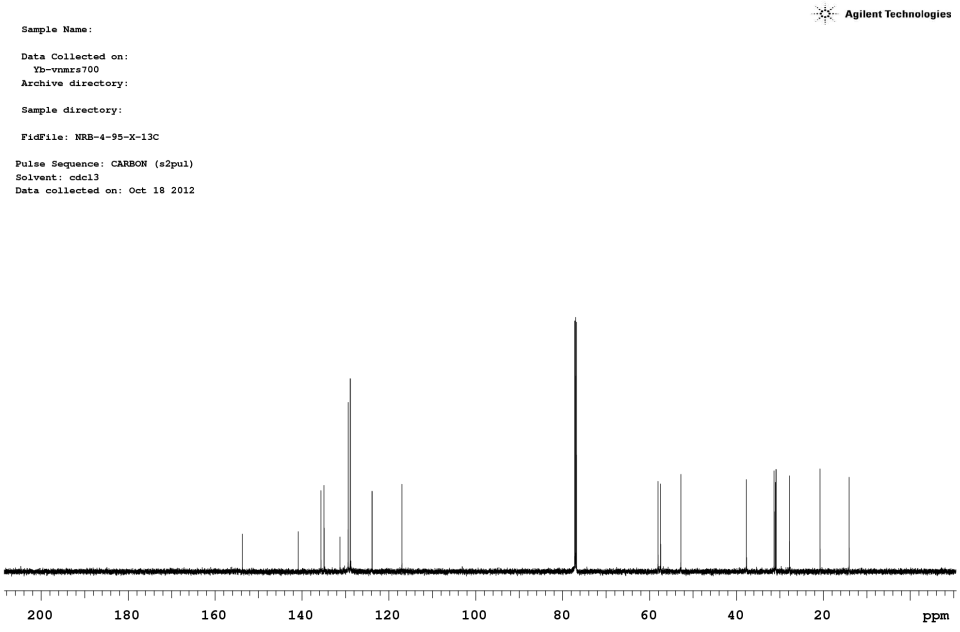
PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.395	81981175	1343363	81.873	84.618
2	23.463	18150993	244195	18.127	15.382
Total		100132168	1587557	100.000	100.000



8b


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-118-X-1H-Z-butene
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 6 2012
H
H
N
O
N
H
C2H5
Cl
8c
8
7
6
5
4
3
2
1
ppm
1.77
2.09
0.96
0.98
0.98
2.03
0.99
1.03
1.03
1.00
3.08
1.08
2.31
1.08
2.31
2.97


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-95-X-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 18 2012
200
180
160
140
120
100
80
60
40
20
ppm

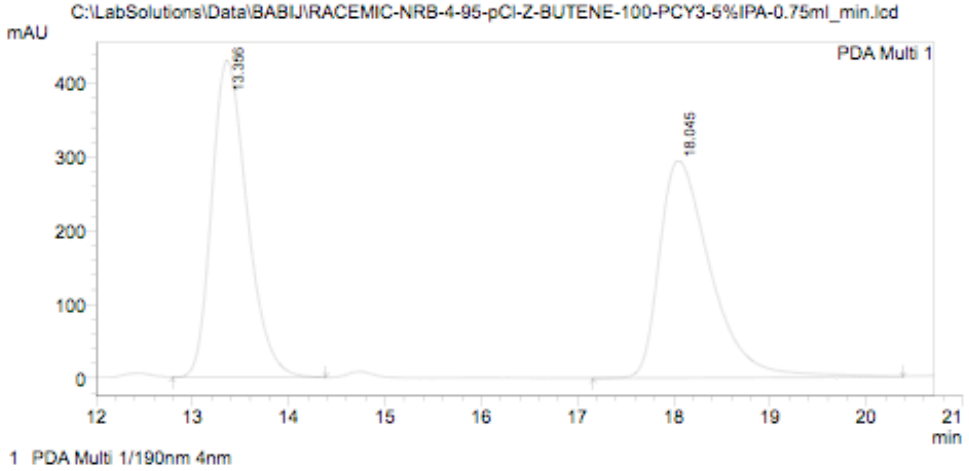
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-95-pCl-Z-BUTENE-100-PCY3-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-95-pCl-Z-BUTENE-100-PCY3-5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-95-pCl-Z-BUTENE-100-PCY3-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/10/2012 3:40:20 PM
Data Processed	: 10/10/2012 4:01:04 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-95-pCl-Z-BUTENE-100-PCY3-5%IPA-0.75ml_min.lcd

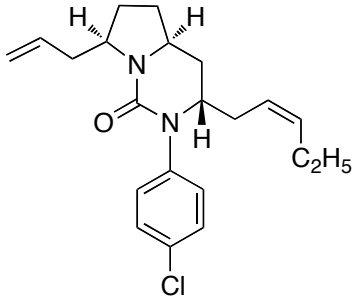


1 PDA Multi 1/190nm 4nm

PeakTable

PDA Ch1 190nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	13.356	10978832	430437	50.161
2	18.045	10908139	295015	49.839
Total		21886971	725452	100.000



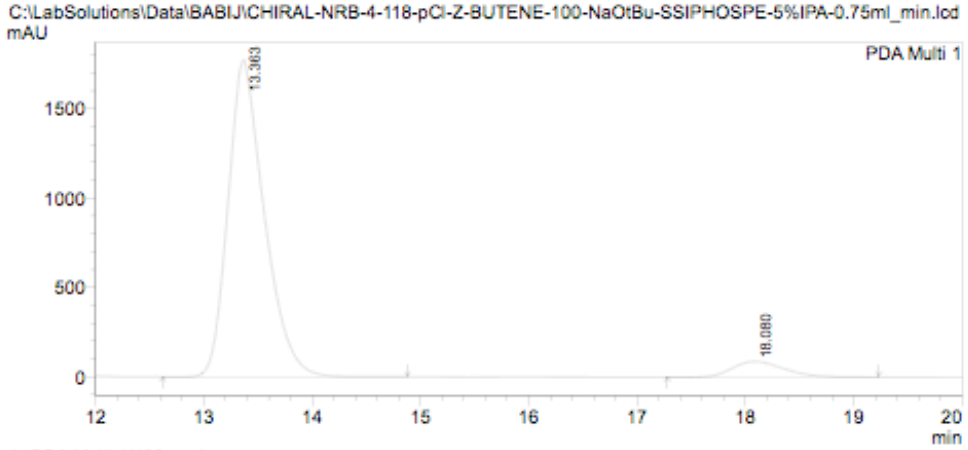
8c

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

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-118-pCl-Z-BUTENE-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-118-pCl-Z-BUTENE-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-118-pCl-Z-BUTENE-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/30/2012 12:32:03 PM
Data Processed	: 11/30/2012 1:30:42 PM

<Chromatogram>

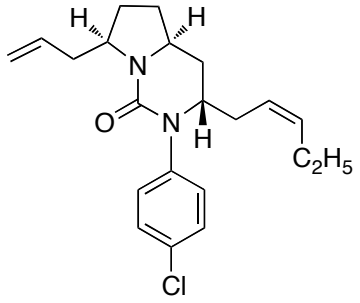


1 PDA Multi 1/190nm 4nm

PeakTable

PDA Ch1 190nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	13.363	40981053	1771633	94.402
2	18.080	2430175	83915	5.598
Total		43411228	1855548	100.000



8c

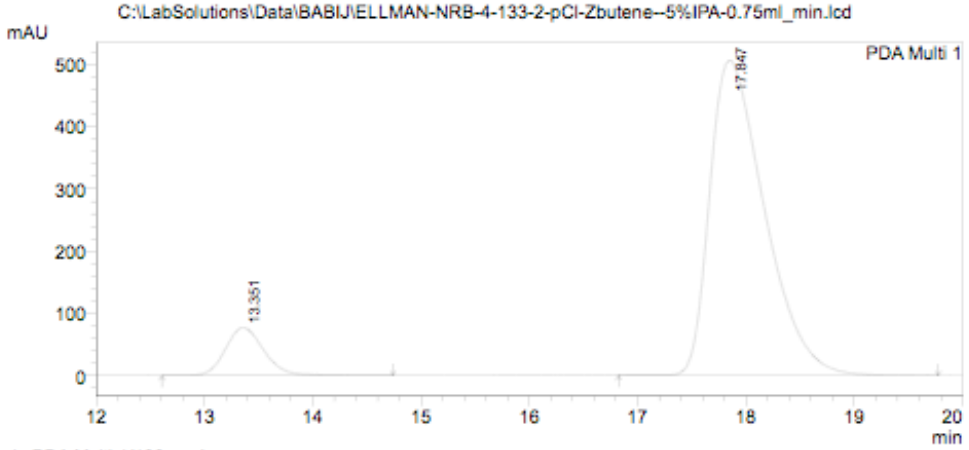
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\ELLMAN-NRB-4-133-2-pCl-Zbutene--5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: ELLMAN-NRB-4-133-2-pCl-Zbutene--5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: ELLMAN-NRB-4-133-2-pCl-Zbutene--5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 12/18/2012 11:59:32 AM
Data Processed	: 12/18/2012 12:39:48 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\ELLMAN-NRB-4-133-2-pCl-Zbutene--5%IPA-0.75ml_min.lcd

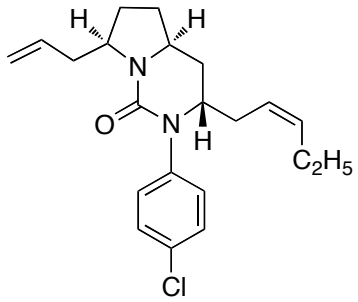


1 PDA Multi 1/190nm 4nm

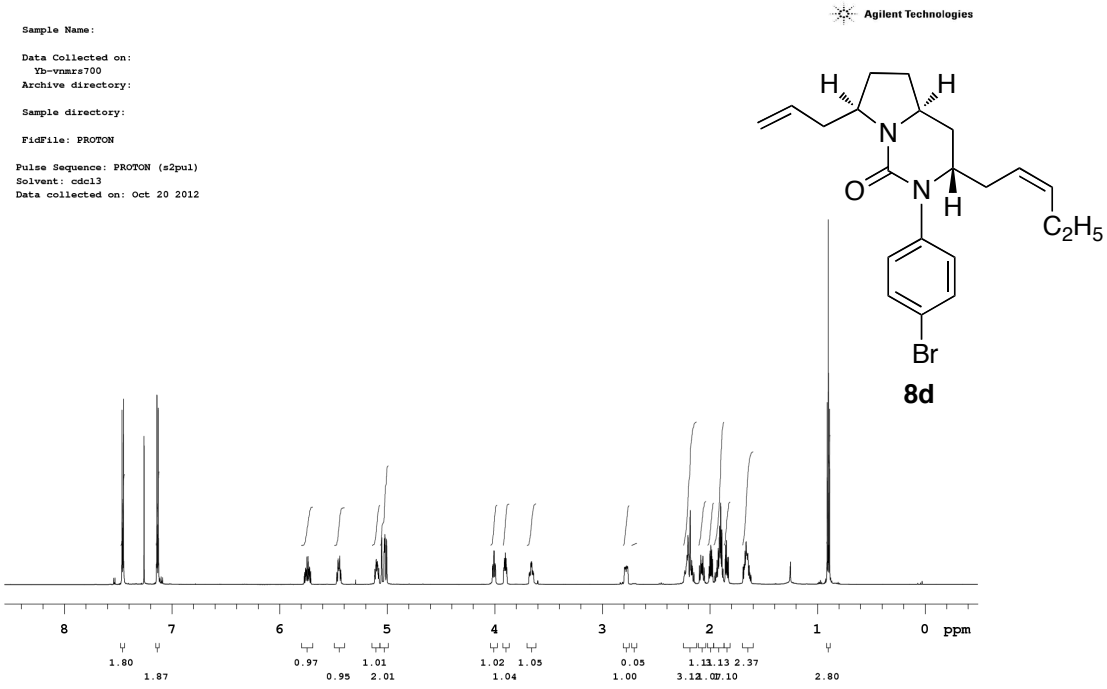
PeakTable

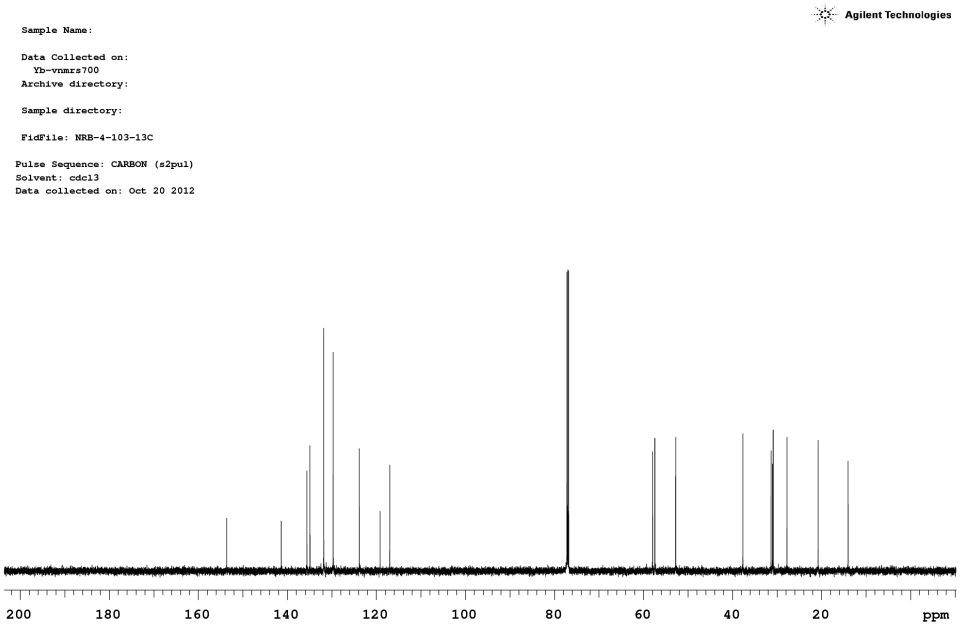
PDA Ch1 190nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	13.351	1900239	77384	9.636
2	17.847	17820385	506100	90.364
Total		19720624	583483	100.000



***ent*-8c**


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2012
H
H
N
O
N
H
C2H5
Br
8d
8
7
6
5
4
3
2
1
0
ppm
1.80
1.87
0.97
0.95
1.01
2.01
1.02
1.05
1.04
0.05
1.00
1.13
2.37
2.80


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-103-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2012
200
180
160
140
120
100
80
60
40
20
ppm

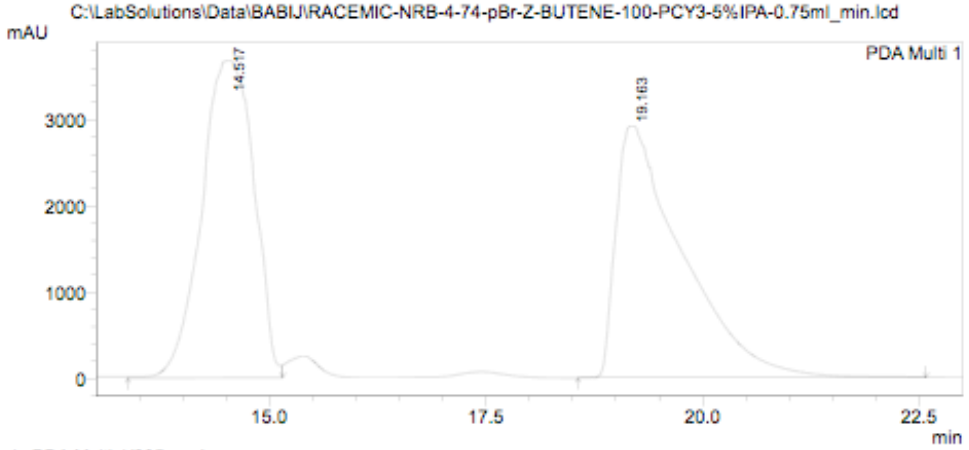
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-74-pBr-Z-BUTENE-100-PCY3-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-74-pBr-Z-BUTENE-100-PCY3-5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-74-pBr-Z-BUTENE-100-PCY3-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 8/28/2012 6:58:36 PM
Data Processed	: 8/28/2012 7:25:17 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-74-pBr-Z-BUTENE-100-PCY3-5%IPA-0.75ml_min.lcd

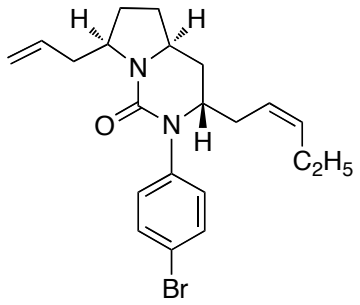


1 PDA Multi 1/205nm 4nm

PeakTable

PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	14.517	154799302	3681642	49.793
2	19.163	156088664	2926638	50.207
Total		310887966	6608280	100.000



8d

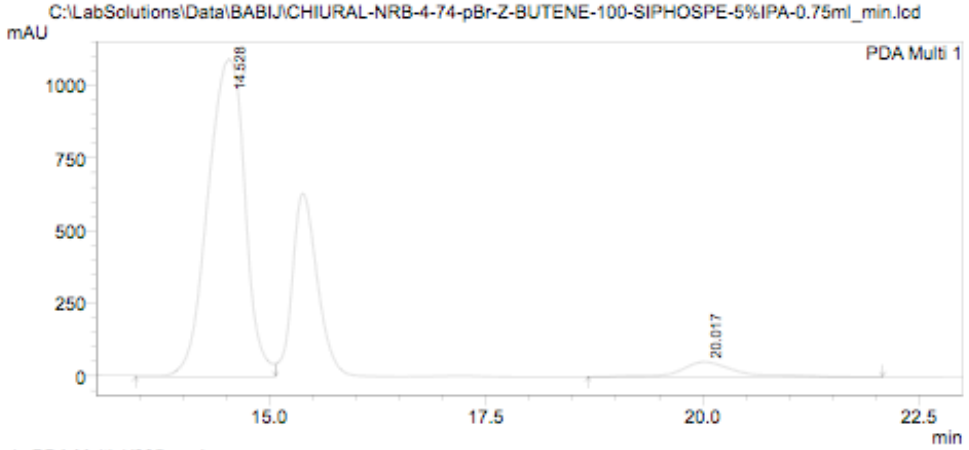
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIURAL-NRB-4-74-pBr-Z-BUTENE-100-SIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIURAL-NRB-4-74-pBr-Z-BUTENE-100-SIPHOSPE-5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIURAL-NRB-4-74-pBr-Z-BUTENE-100-SIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 8/28/2012 7:28:02 PM
Data Processed	: 10/19/2012 7:38:06 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\CHIURAL-NRB-4-74-pBr-Z-BUTENE-100-SIPHOSPE-5%IPA-0.75ml_min.lcd

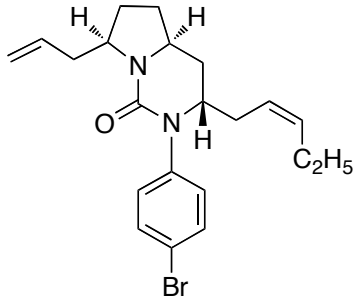


1 PDA Multi 1/205nm 4nm

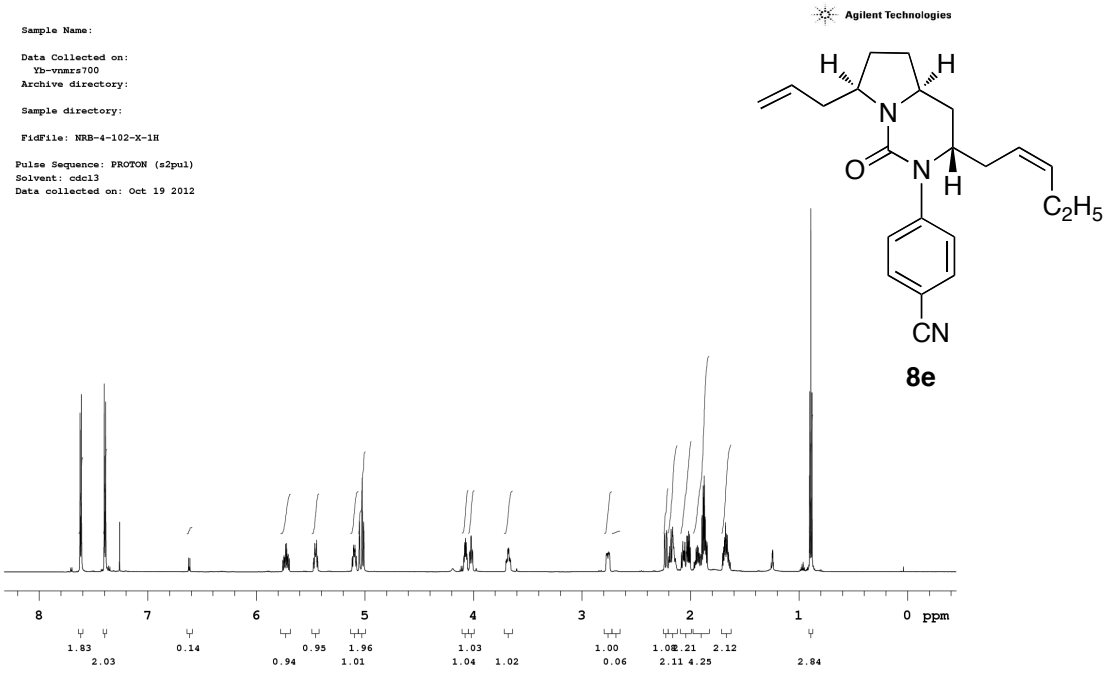
PeakTable

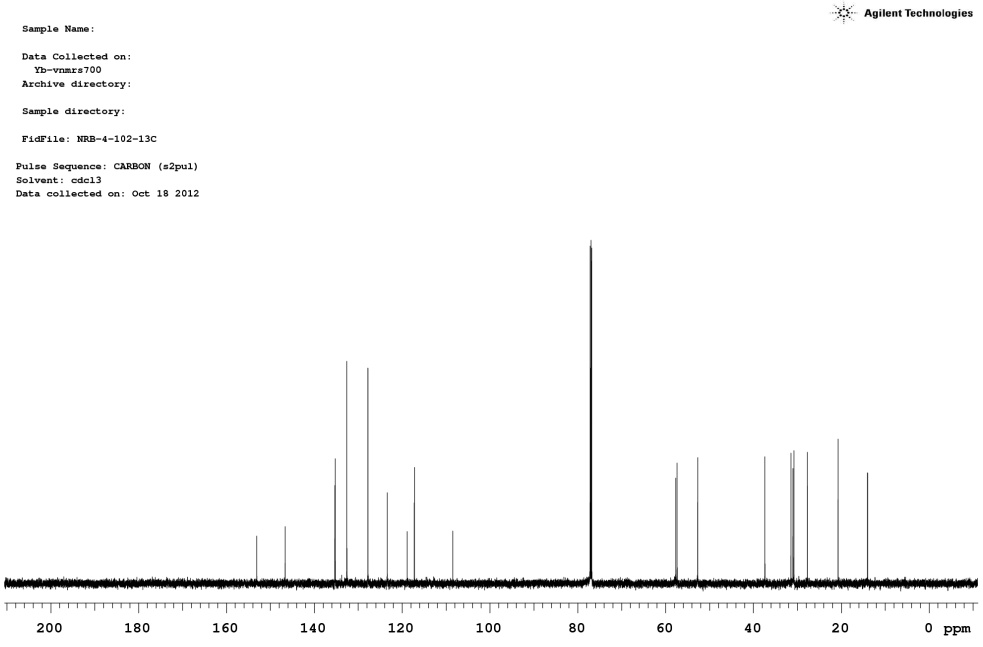
PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	14.528	32443013	1090281	94.464
2	20.017	1901139	49794	5.536
Total		34344152	1140076	100.000



8d


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-102-X-1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 19 2012
H
H
N
O
N
H
C_2H_5
CN
8e
8 7 6 5 4 3 2 1 0 ppm
1.83 2.03 0.14 0.94 0.95 1.01 1.96 1.04 1.03 1.02 1.00 0.06 1.08 2.11 2.21 4.25 2.12 2.84


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-102-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 18 2012
200 180 160 140 120 100 80 60 40 20 0 ppm

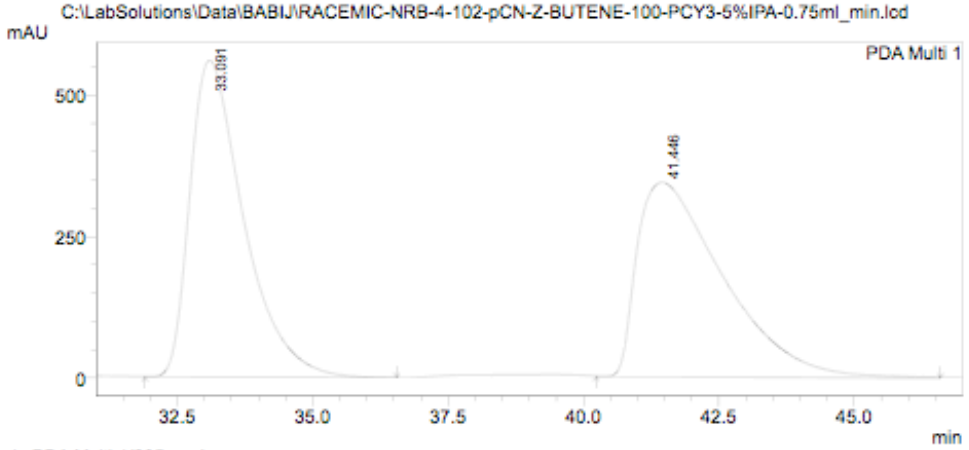
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-102-pCN-Z-BUTENE-100-PCY3-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-102-pCN-Z-BUTENE-100-PCY3-5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-102-pCN-Z-BUTENE-100-PCY3-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/17/2012 4:27:14 PM
Data Processed	: 10/17/2012 5:22:38 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-102-pCN-Z-BUTENE-100-PCY3-5%IPA-0.75ml_min.lcd

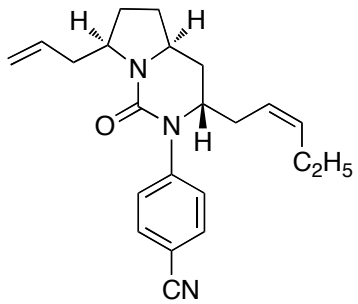


1 PDA Multi 1/205nm 4nm

PeakTable

PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	33.091	38677775	559423	50.478
2	41.446	37945343	343252	49.522
Total		76623118	902676	100.000



8e

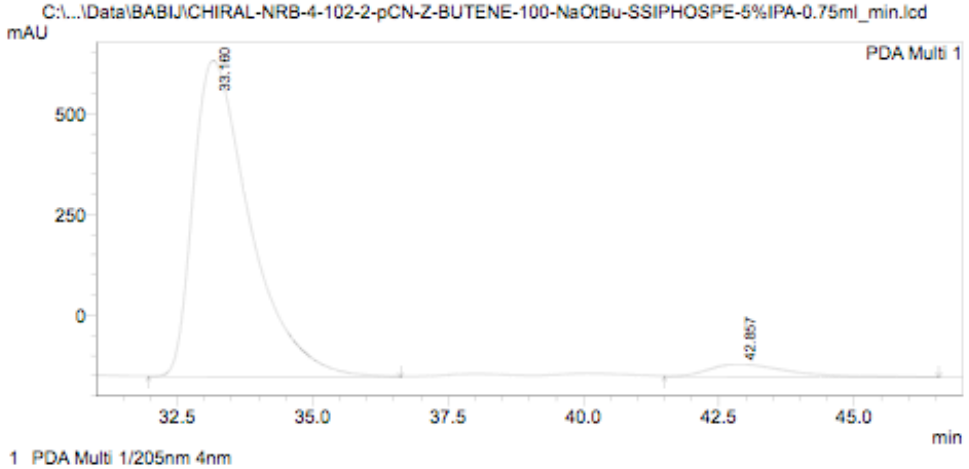
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-102-2-pCN-Z-BUTENE-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-102-2-pCN-Z-BUTENE-100-NaOtBu-SSIPHOSPE-5%IPA-0.75
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-102-2-pCN-Z-BUTENE-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/23/2012 6:10:26 PM
Data Processed	: 10/23/2012 7:08:32 PM

<Chromatogram>

C:\...\Data\BABIJ\CHIRAL-NRB-4-102-2-pCN-Z-BUTENE-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

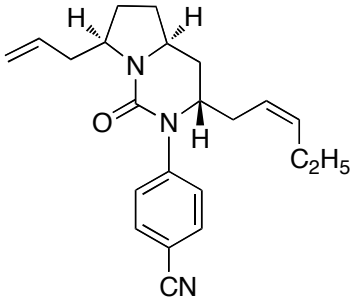


1 PDA Multi 1/205nm 4nm

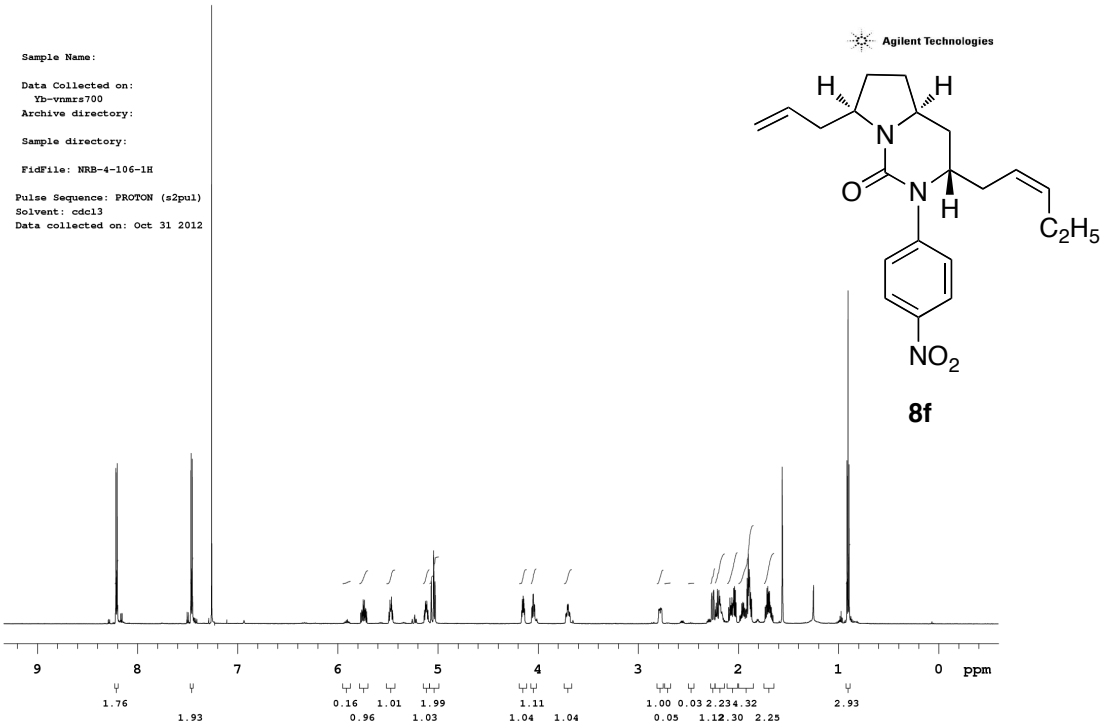
PeakTable

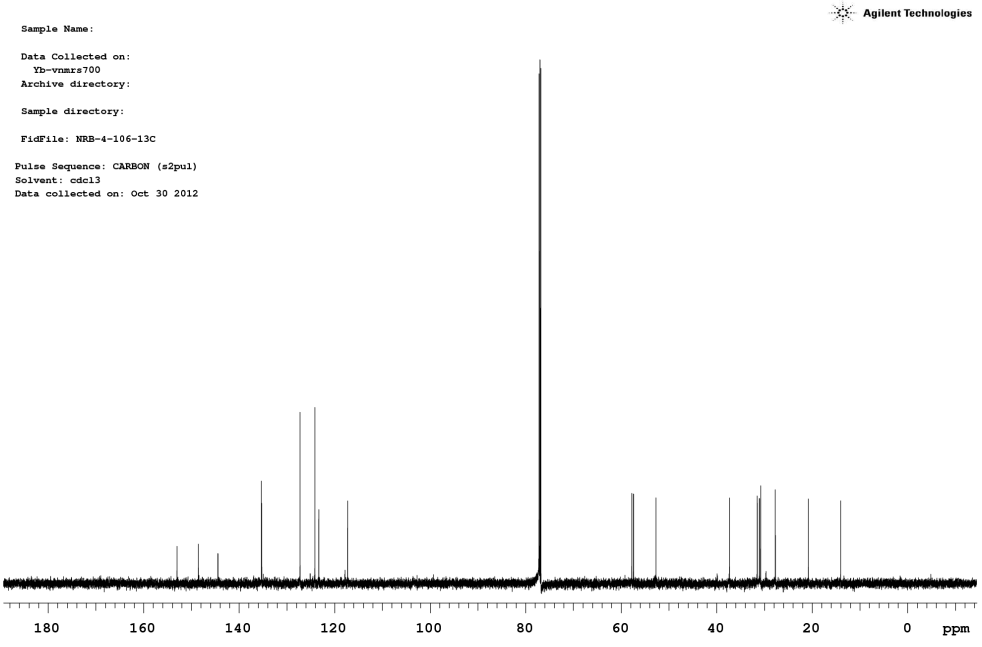
PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	33.160	57259672	784638	94.609
2	42.857	3262654	31536	5.391
Total		60522326	816174	100.000



8e


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-106-1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 31 2012
H
H
N
O
N
H
C2H5
NO2
8f
9 8 7 6 5 4 3 2 1 0 ppm
1.76 1.93 0.16 0.96 1.01 1.03 1.99 1.04 1.11 1.04 1.00 0.05 0.03 1.12 2.23 2.30 4.32 2.25 2.93


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-106-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 30 2012
180 160 140 120 100 80 60 40 20 0 ppm

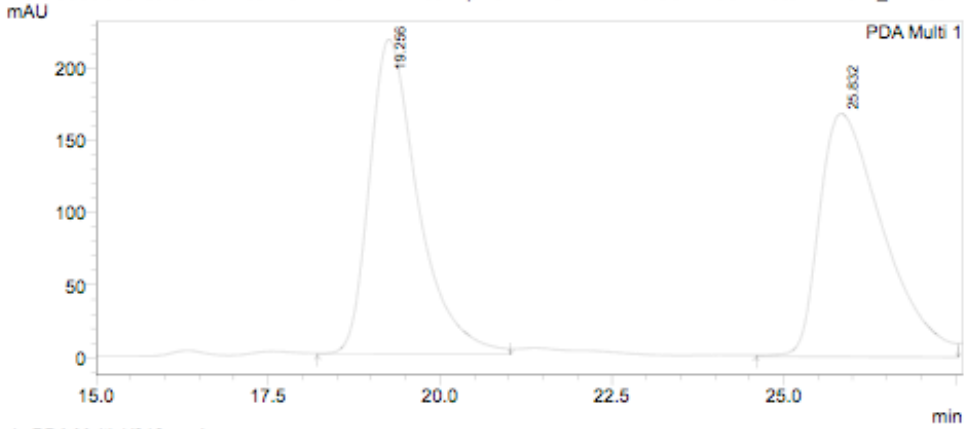
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-106-2-pNO2-Z-BUTENE-120-SSIPHOSPE-5%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-106-2-pNO2-Z-BUTENE-120-SSIPHOSPE-5%IPA-1.5ml
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-106-2-pNO2-Z-BUTENE-120-SSIPHOSPE-5%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/30/2012 4:06:25 PM
Data Processed	: 10/30/2012 4:40:18 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-106-2-pNO2-Z-BUTENE-120-SSIPHOSPE-5%IPA-1.5ml_min.lcd

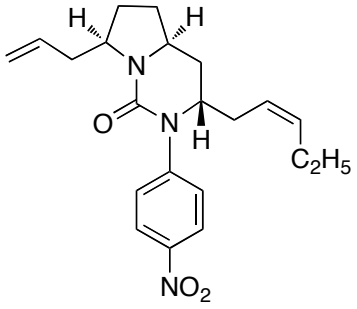


1 PDA Multi 1/310nm 4nm

PeakTable

PDA Ch1 310nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	19.256	10775717	217860	49.820
2	25.832	10853467	168010	50.180
Total		21629184	385871	100.000



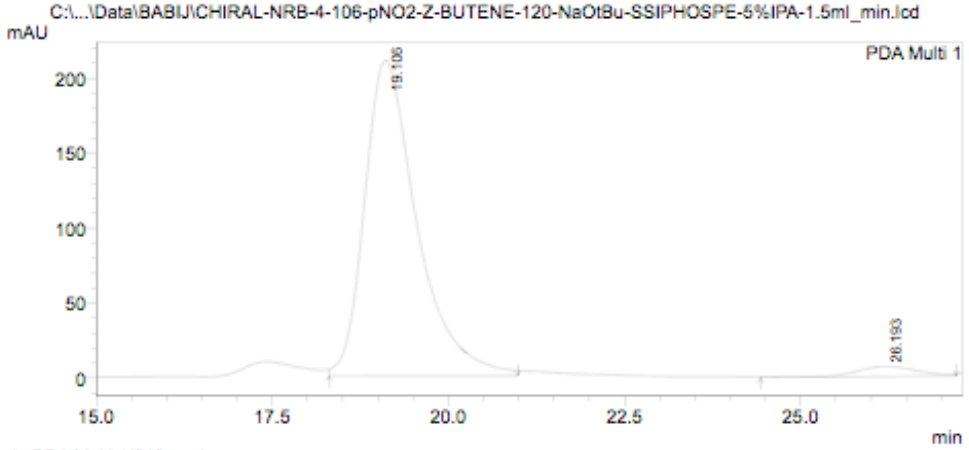
8f

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

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-106-pNO2-Z-BUTENE-120-NaOtBu-SSIPHOSPE-5%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-106-pNO2-Z-BUTENE-120-NaOtBu-SSIPHOSPE-5%IPA-1.5ml
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-106-pNO2-Z-BUTENE-120-NaOtBu-SSIPHOSPE-5%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/30/2012 2:49:31 PM
Data Processed	: 10/30/2012 3:23:55 PM

<Chromatogram>

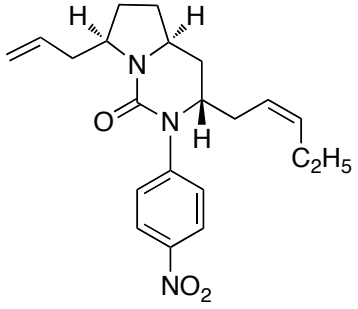


1 PDA Multi 1/310nm 4nm

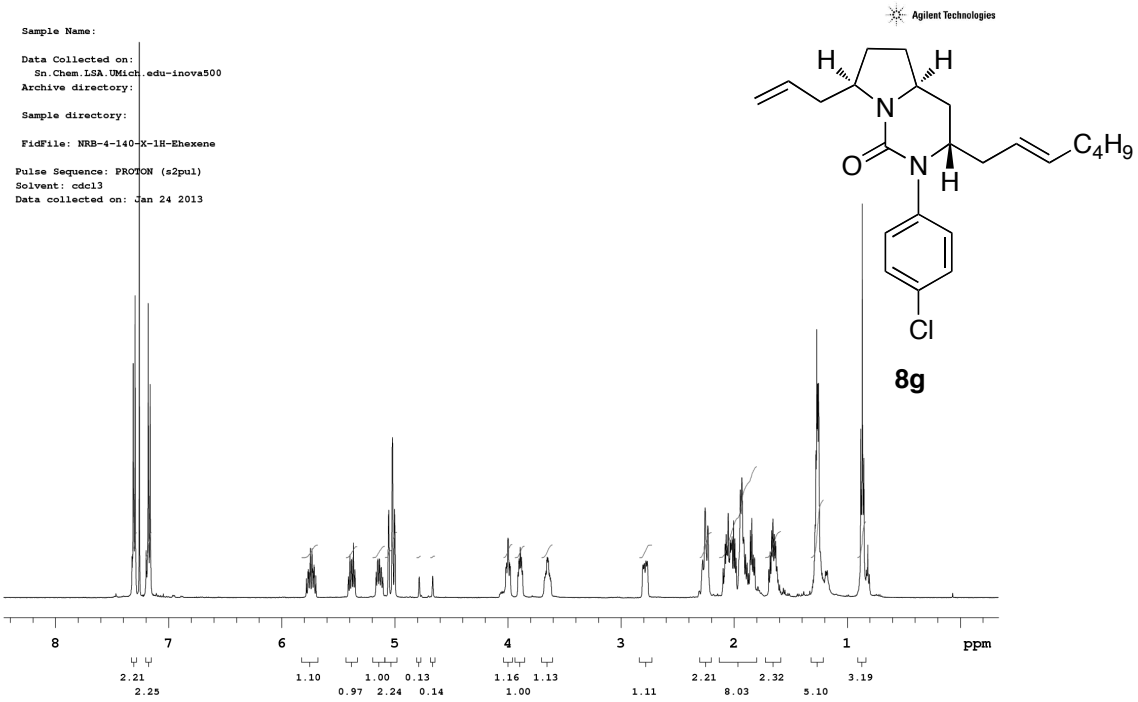
PeakTable

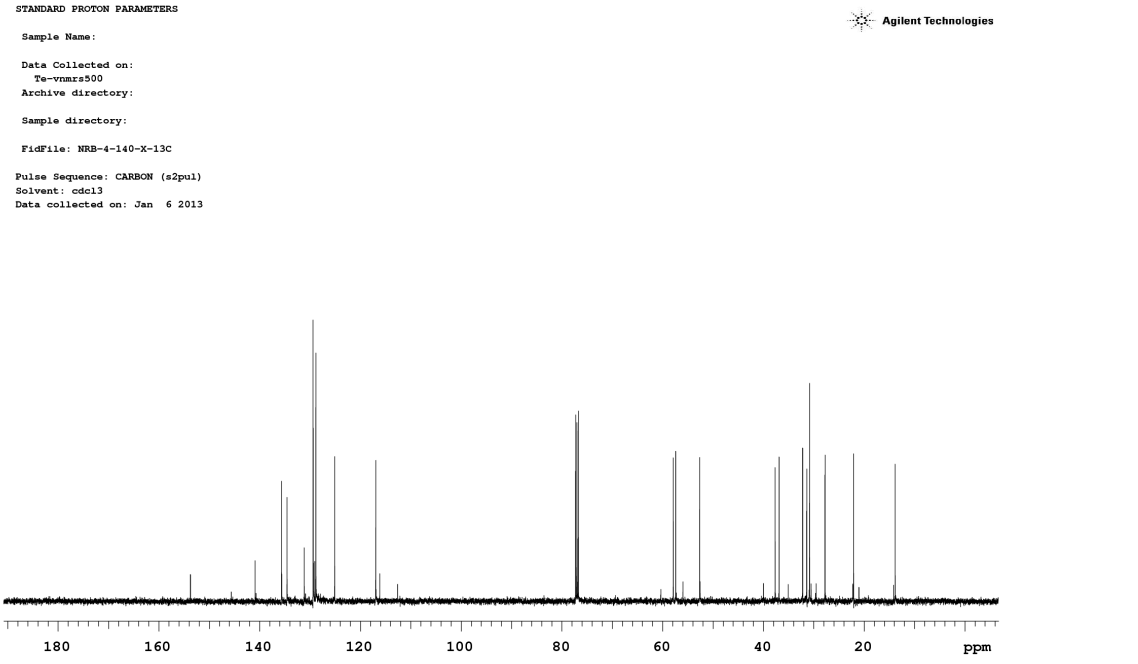
PDA Ch1 310nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	19.106	10898189	210578	96.308
2	26.193	417817	6715	3.692
Total		11316006	217293	100.000



8f


Agilent Technologies
Sample Name:
Data Collected on:
Sn.Chem.LSA.UMich.edu-inova500
Archive directory:
Sample directory:
FidFile: NRB-4-140-X-1H-Ehexene
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 24 2013
H
H
N
O
N
H
C_4H_9
Cl
8g
8
7
6
5
4
3
2
1
ppm
2.21
2.25
1.10
1.00
0.97
0.13
2.24
0.14
1.16
1.00
1.13
1.11
2.21
8.03
2.32
5.10
3.19


STANDARD PROTON PARAMETERS
Agilent Technologies
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: NRB-4-140-X-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 6 2013
180
160
140
120
100
80
60
40
20
ppm

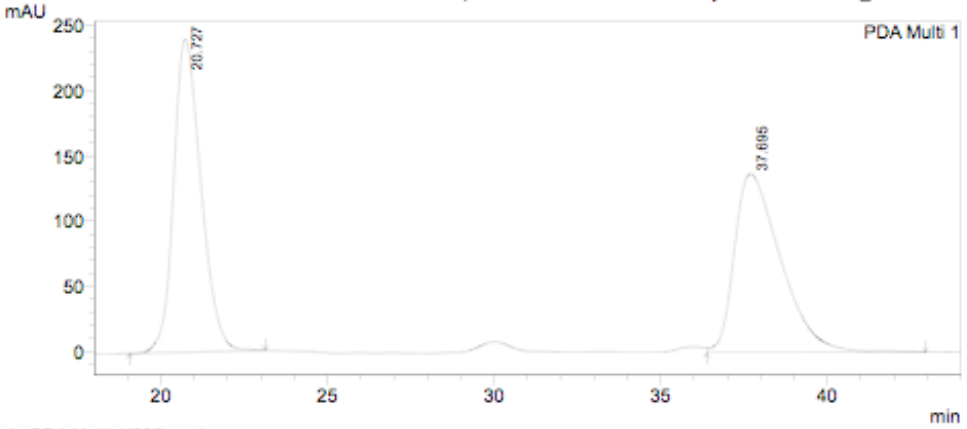
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-140-pCl-Ehexene-100-NaOtBu-PCy3-1.5%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-140-pCl-Ehexene-100-NaOtBu-PCy3-1.5%IPA-1.5ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-140-pCl-Ehexene-100-NaOtBu-PCy3-1.5%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/4/2013 4:14:52 PM
Data Processed	: 1/4/2013 4:58:59 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-140-pCl-Ehexene-100-NaOtBu-PCy3-1.5%IPA-1.5ml_min.lcd

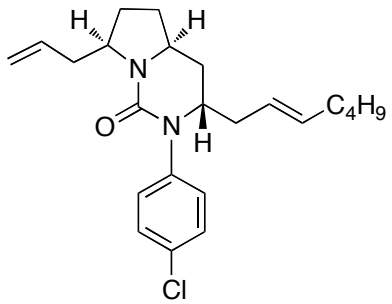


1 PDA Multi 1/205nm 4nm

PeakTable

PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	20.727	13698615	239789	50.749
2	37.695	13294427	136862	49.251
Total		26993042	376651	100.000



8g

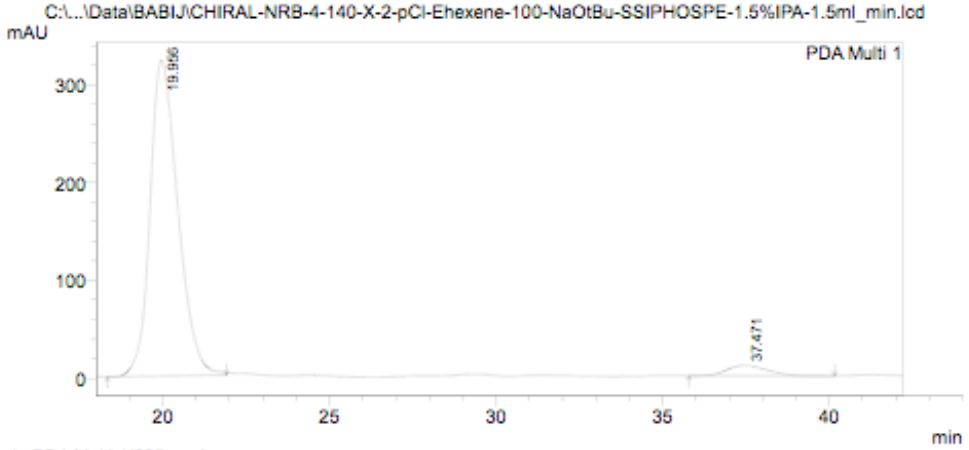
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-140-X-2-pCl-Ehexene-100-NaOtBu-SSIPHOSPE-1.5%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-140-X-2-pCl-Ehexene-100-NaOtBu-SSIPHOSPE-1.5%IPA-1
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-140-X-2-pCl-Ehexene-100-NaOtBu-SSIPHOSPE-1.5%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/4/2013 4:59:58 PM
Data Processed	: 1/4/2013 5:42:12 PM

<Chromatogram>

C:\...\Data\BABIJ\CHIRAL-NRB-4-140-X-2-pCl-Ehexene-100-NaOtBu-SSIPHOSPE-1.5%IPA-1.5ml_min.lcd

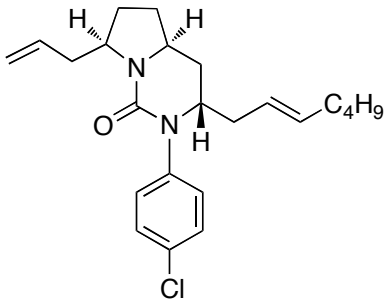


1 PDA Multi 1/205nm 4nm

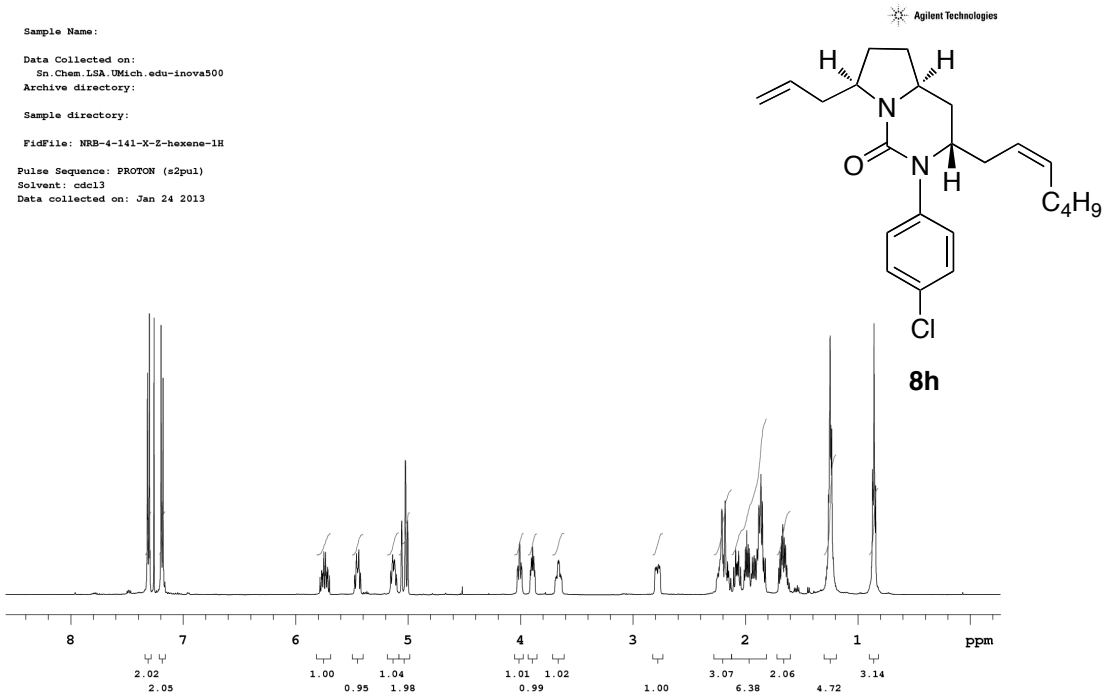
PeakTable

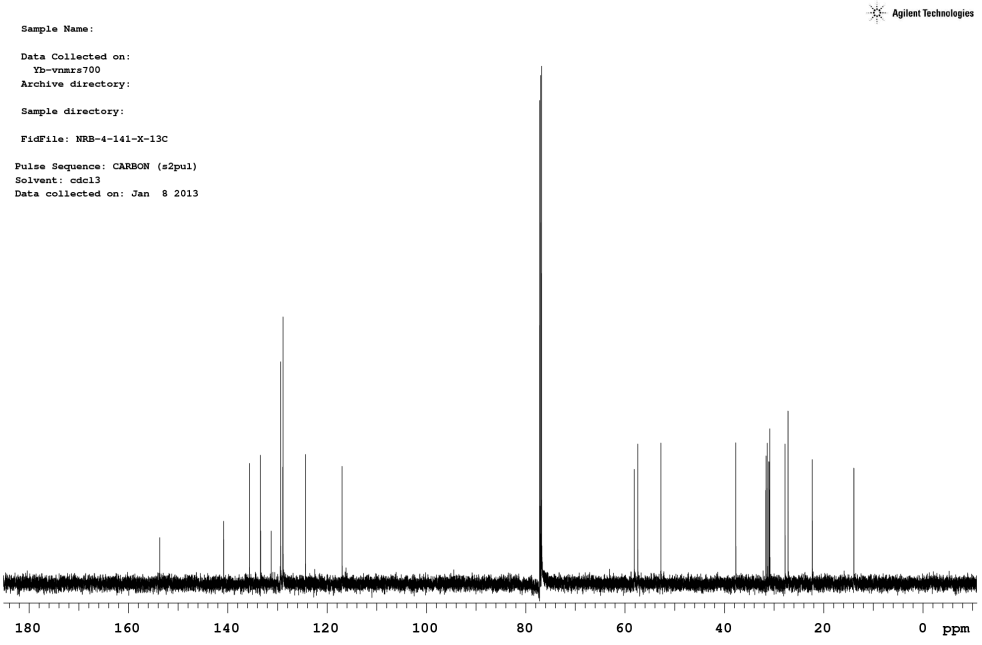
PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	19.956	18822079	322958	95.508
2	37.471	885292	10642	4.492
Total		19707371	333600	100.000



8g


Agilent Technologies
Sample Name:
Data Collected on:
Sn.Chem.LSA.UMich.edu-inova500
Archive directory:
Sample directory:
FidFile: NRB-4-141-X-Z-hexene-1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 24 2013
H
H
N
O
N
H
C4H9
Cl
8h
8
7
6
5
4
3
2
1
ppm
2.02
2.05
1.00
0.95
1.04
1.98
1.01
1.02
0.99
1.00
3.07
6.38
2.06
4.72
3.14


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-141-X-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 8 2013
180
160
140
120
100
80
60
40
20
0
ppm

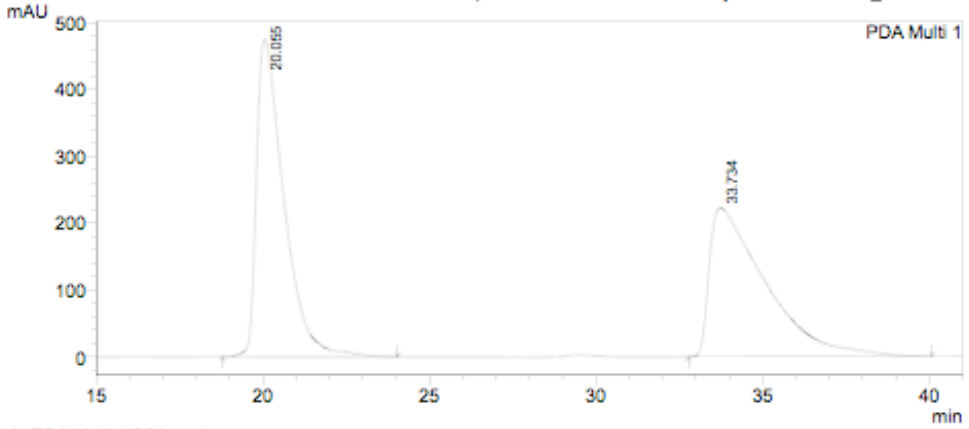
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-141-R-pCl-Zhexene-100-NaOtBu-PCy3-1.5%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-141-R-pCl-Zhexene-100-NaOtBu-PCy3-1.5%IPA-1.5ml_m
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-141-R-pCl-Zhexene-100-NaOtBu-PCy3-1.5%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/8/2013 12:32:09 PM
Data Processed	: 1/8/2013 1:30:14 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-141-R-pCl-Zhexene-100-NaOtBu-PCy3-1.5%IPA-1.5ml_min.lcd

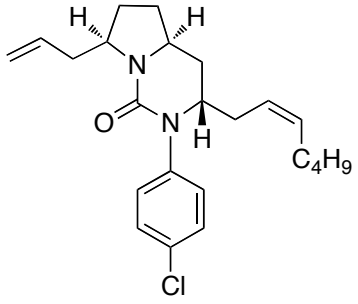


1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	20.055	27108175	474557	50.964
2	33.734	26082514	222410	49.036
Total		53190689	696967	100.000



8h

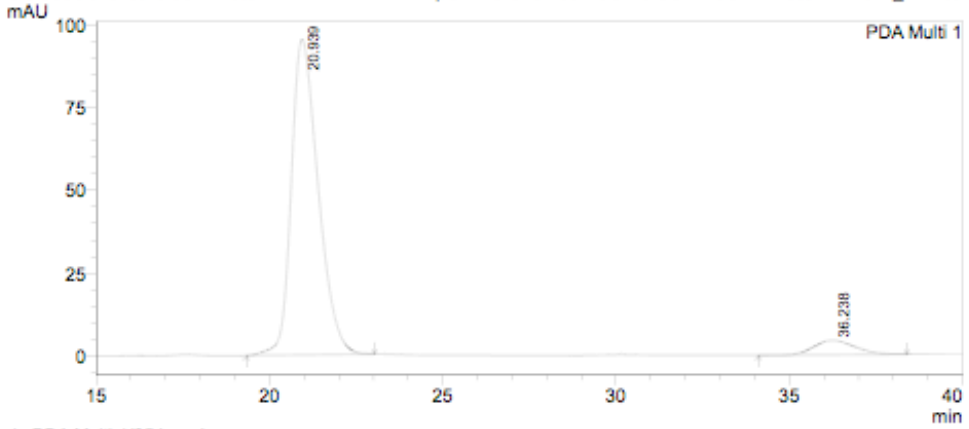
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-141-X-pCl-Zhexene-100-NaOtBu-SSIPHOSPE-1.5%IPA-1.5ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-141-X-pCl-Zhexene-100-NaOtBu-SSIPHOSPE-1.5%IPA-1.5
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-141-X-pCl-Zhexene-100-NaOtBu-SSIPHOSPE-1.5%IPA-1.5ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 1/7/2013 5:02:46 PM
Data Processed	: 1/7/2013 5:47:55 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-141-X-pCl-Zhexene-100-NaOtBu-SSIPHOSPE-1.5%IPA-1.5ml_min.lcd

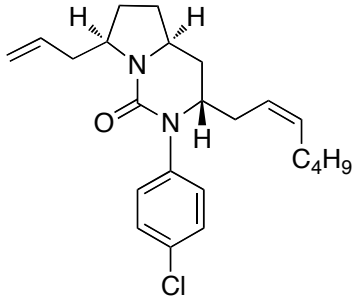


1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	20.939	5164727	95174	93.561
2	36.238	355461	4348	6.439
Total		5520188	99522	100.000



8h

Agilent Technologies

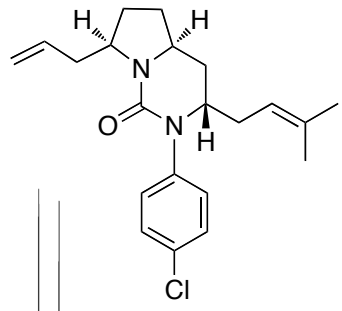
Sample Name:

Data Collected on:
Yb-vnmrs700
Archive directory:

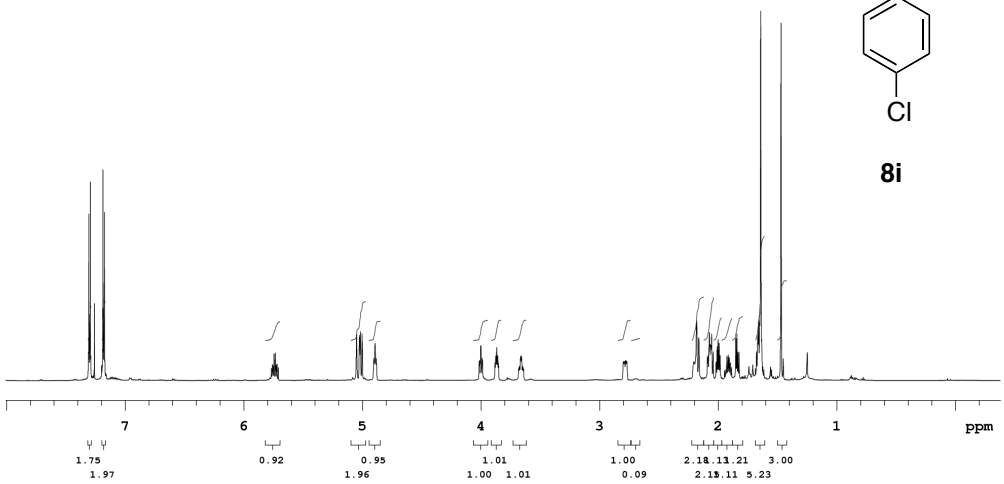
Sample directory:

FidFile: NRB-4-134-X-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 19 2012



8i



Agilent Technologies

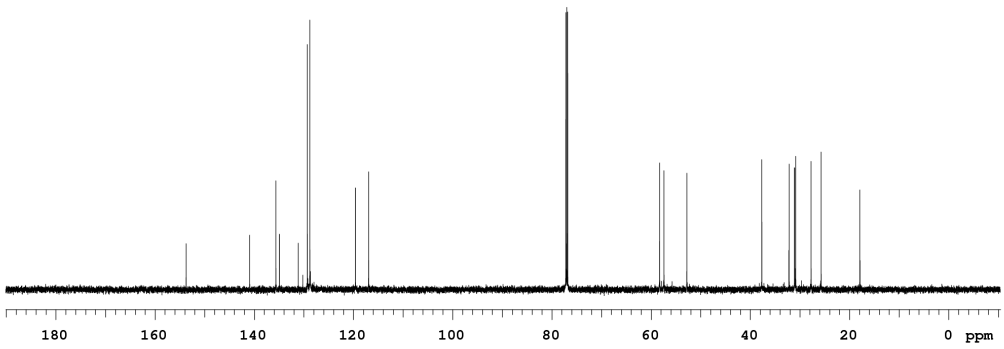
Sample Name:

Data Collected on:
Yb-vnmrs700
Archive directory:

Sample directory:

FidFile: NRB-4-134-X-13C

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Dec 19 2012



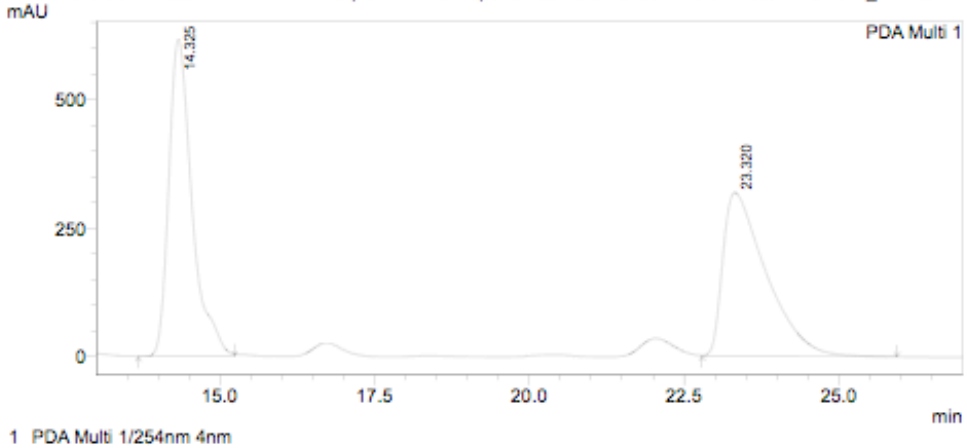
==== Shimadzu LCsolution Analysis Report ====

C:\...\Data\BABIJ\CHIRAL-NRB-4-123-pCl-1Br2MePropene-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-123-pCl-1Br2MePropene-100-NaOtBu-SSIPHOSPE-5%IPA-0
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-123-pCl-1Br2MePropene-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 12/11/2012 6:02:30 PM
Data Processed	: 12/11/2012 6:43:19 PM

<Chromatogram>

C:\...\Data\BABIJ\CHIRAL-NRB-4-123-pCl-1Br2MePropene-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

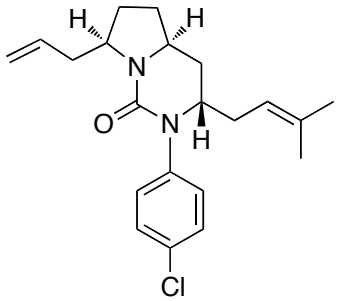


1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	14.325	16086664	615989	50.923
2	23.320	15503816	317951	49.077
Total		31590479	933941	100.000



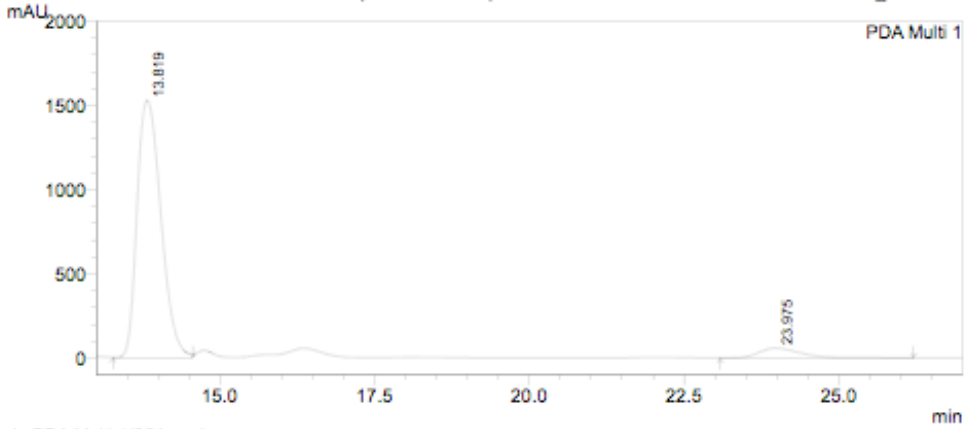
==== Shimadzu LCsolution Analysis Report ====

C:\...\Data\BABIJ\CHIRAL-NRB-4-134-X-pCl-1Br2MePropene-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-134-X-pCl-1Br2MePropene-100-NaOtBu-SSIPHOSPE-5%IPA
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-134-X-pCl-1Br2MePropene-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 12/19/2012 3:20:33 PM
Data Processed	: 12/19/2012 3:51:53 PM

<Chromatogram>

C:\...\Data\BABIJ\CHIRAL-NRB-4-134-X-pCl-1Br2MePropene-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

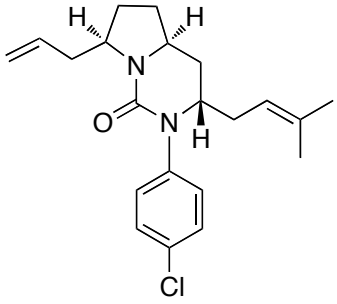


1 PDA Multi 1/254nm 4nm

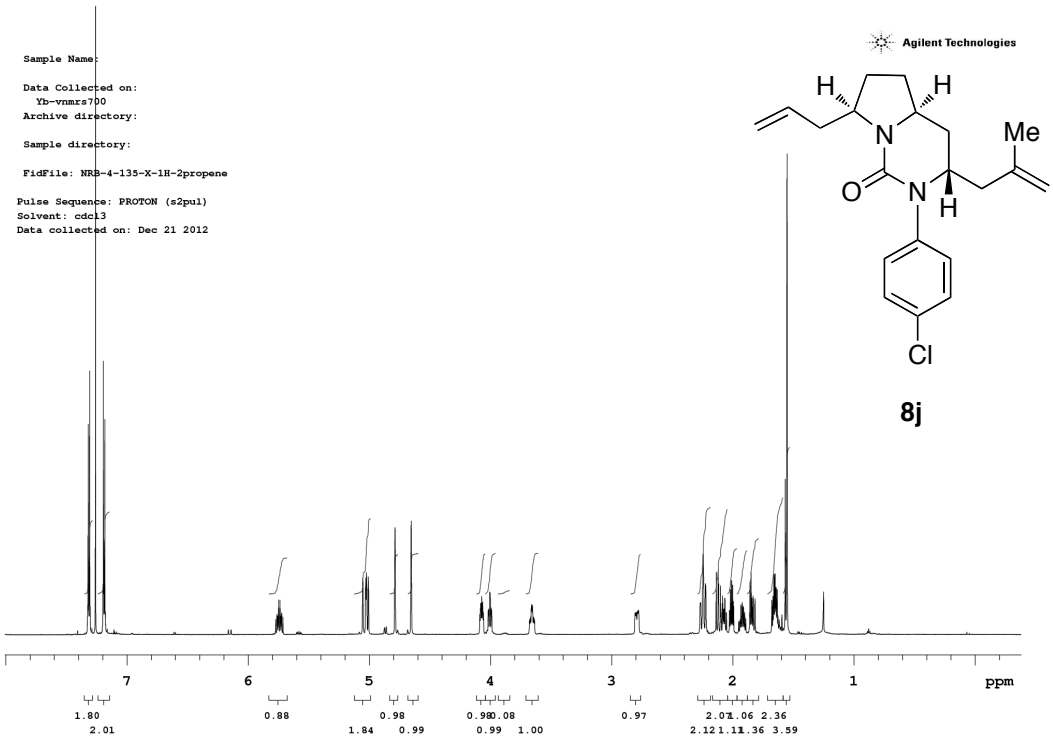
PeakTable

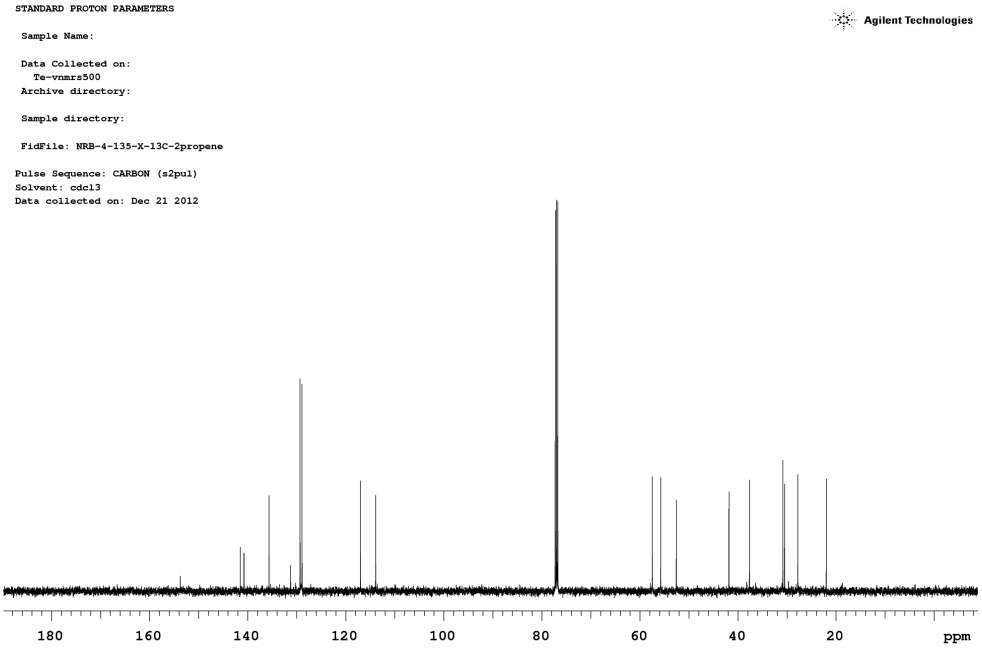
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	13.819	40943767	1527198	93.771
2	23.975	2719612	58406	6.229
Total		43663379	1585604	100.000



8i


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-135-X-1H-2propene
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 21 2012
H
H
N
Me
O
N
H
Cl
8j
7
6
5
4
3
2
1
ppm
1.80
2.01
0.88
0.98
1.84
0.99
0.98
0.99
0.08
1.00
0.97
2.07
2.12
1.06
1.11
2.36
3.59


STANDARD PROTON PARAMETERS
Agilent Technologies
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: NRB-4-135-X-13C-2propene
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Dec 21 2012
180
160
140
120
100
80
60
40
20
ppm

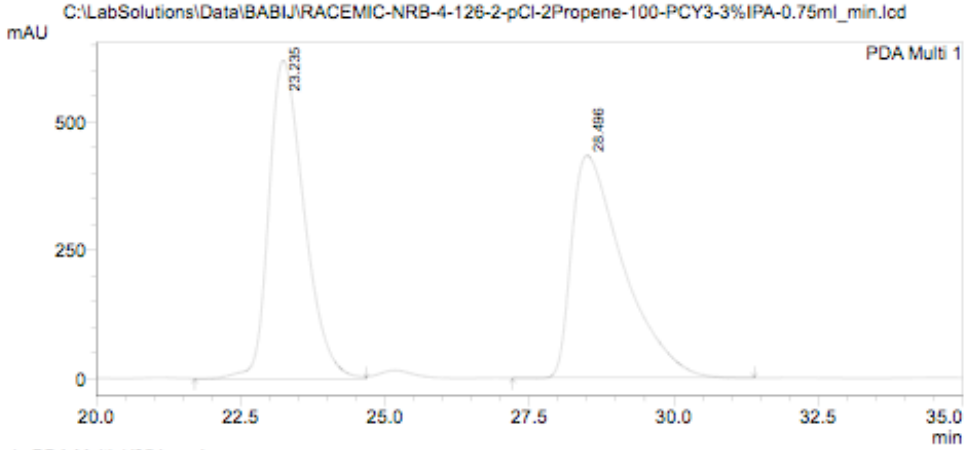
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-126-2-pCl-2Propene-100-PCY3-3%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-126-2-pCl-2Propene-100-PCY3-3%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-126-2-pCl-2Propene-100-PCY3-3%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 12/20/2012 1:52:01 PM
Data Processed	: 12/20/2012 2:31:47 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-126-2-pCl-2Propene-100-PCY3-3%IPA-0.75ml_min.lcd

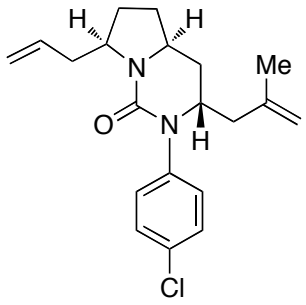


1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	23.235	26432880	621250	49.297
2	28.496	27186656	434267	50.703
Total		53619536	1055517	100.000



8j

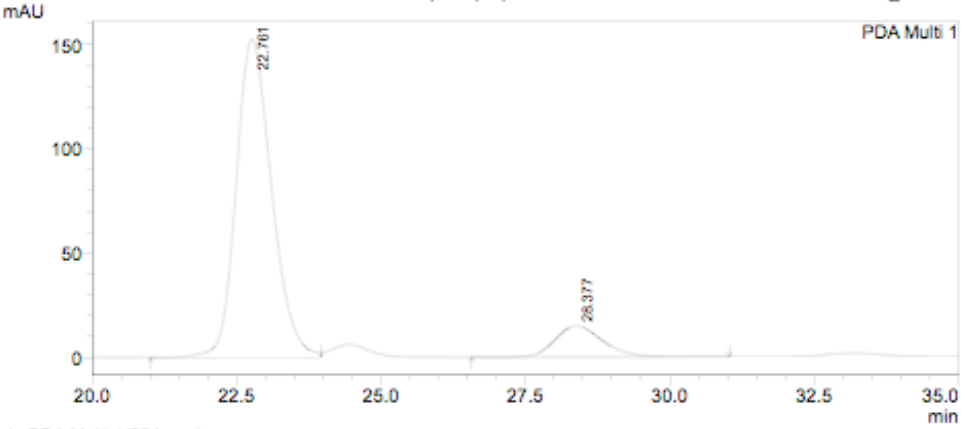
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-135-2-pCl-2propene-100-NaOtBu-SSIPHOSPE-3%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-135-2-pCl-2prop-100-NaOtBu-SSIPHOSPE-3%IPA-0.75ml
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-135-2-pCl-2propene-100-NaOtBu-SSIPHOSPE-3%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 12/20/2012 5:22:09 PM
Data Processed	: 12/20/2012 6:22:39 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-135-2-pCl-2propene-100-NaOtBu-SSIPHOSPE-3%IPA-0.75ml_min.lcd

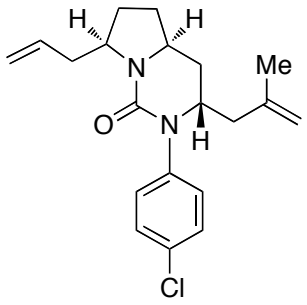


1 PDA Multi 1/254nm 4nm

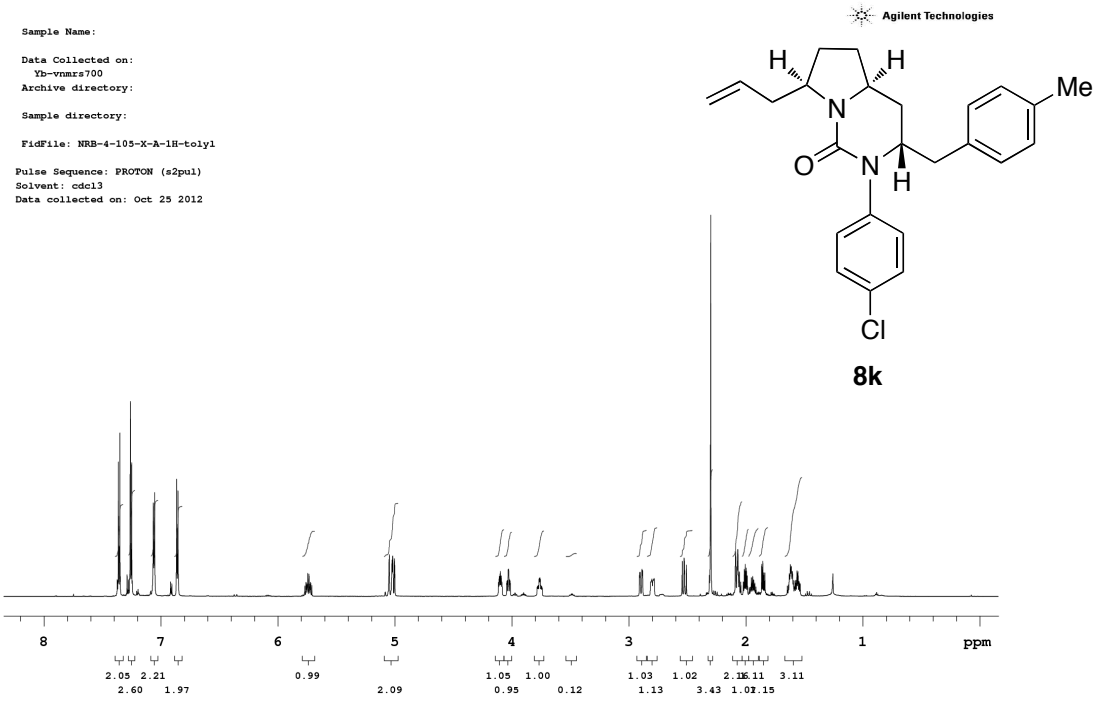
PeakTable

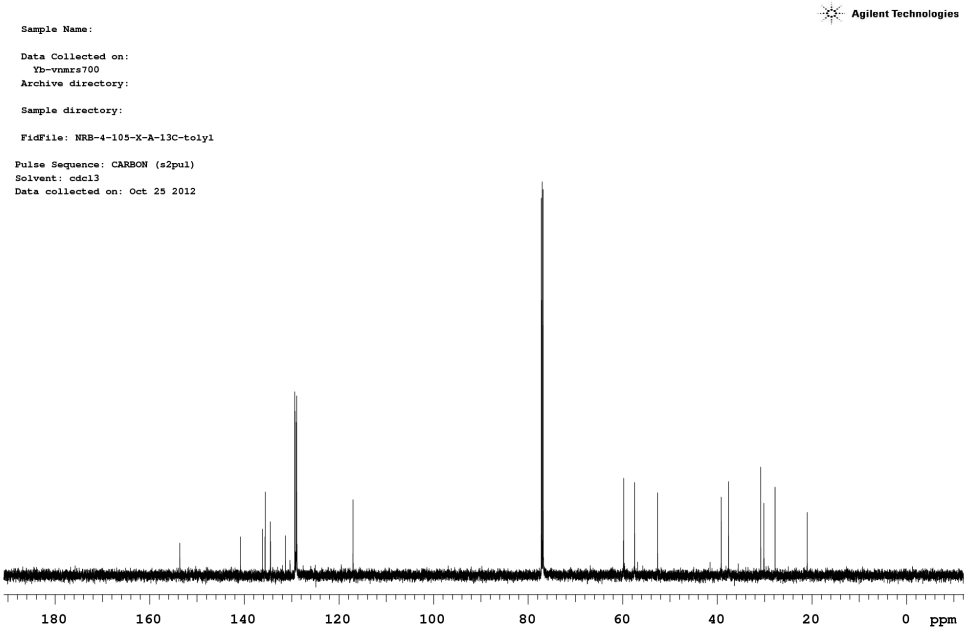
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	22.761	6535743	152637	88.459
2	28.377	852687	14946	11.541
Total		7388431	167583	100.000



8j


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-105-X-A-1H-tolyl
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 25 2012
H
H
N
O
N
H
Me
Cl
8k
8
7
6
5
4
3
2
1
ppm
2.05
2.21
2.60
1.97
0.99
2.09
1.05
1.00
0.95
0.12
1.03
1.02
1.13
3.43
3.11
3.11
1.07
1.15


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-105-X-A-13C-tolyl
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 25 2012
180
160
140
120
100
80
60
40
20
0
ppm

Sample Name:

Data Collected on:
Yb-vnmrs700
Archive directory:

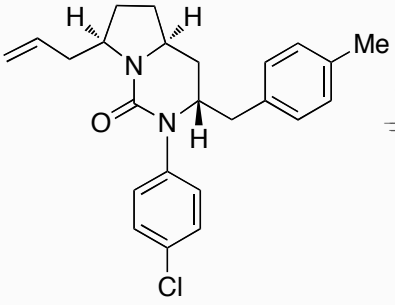
Sample directory:

FidFile: NRB-4-120-COSY-4tol

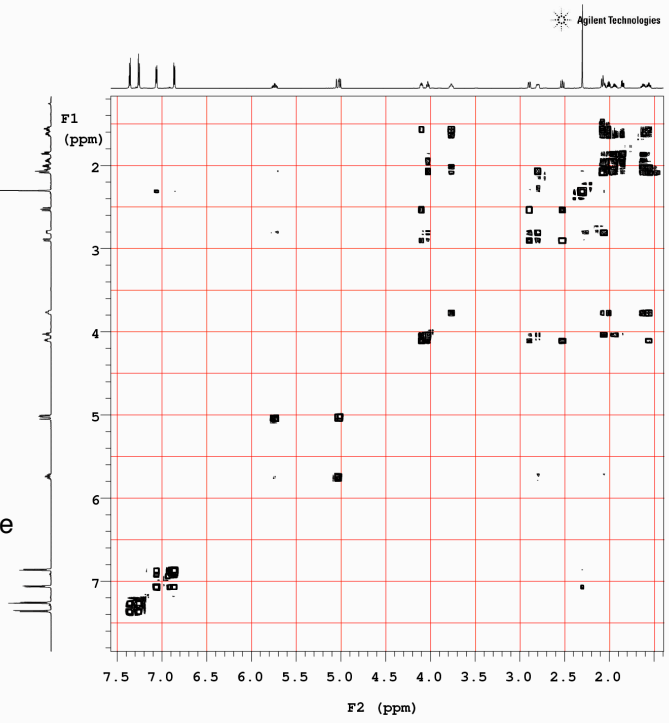
Pulse Sequence: gCOSY
Solvent: cdcl3
Data collected on: Dec 10 2012

Operator: nbabij

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 11160.7 Hz
2D Width 11160.7 Hz
2 repetitions
128 increments
OBSERVE H1, 699.7567660 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Sq. sine bell 0.011 sec
FT size 4096 x 4096
Total time 5 min 40 sec



8k



Sample Name:

Data Collected on:
Yb-vnmrs700
Archive directory:

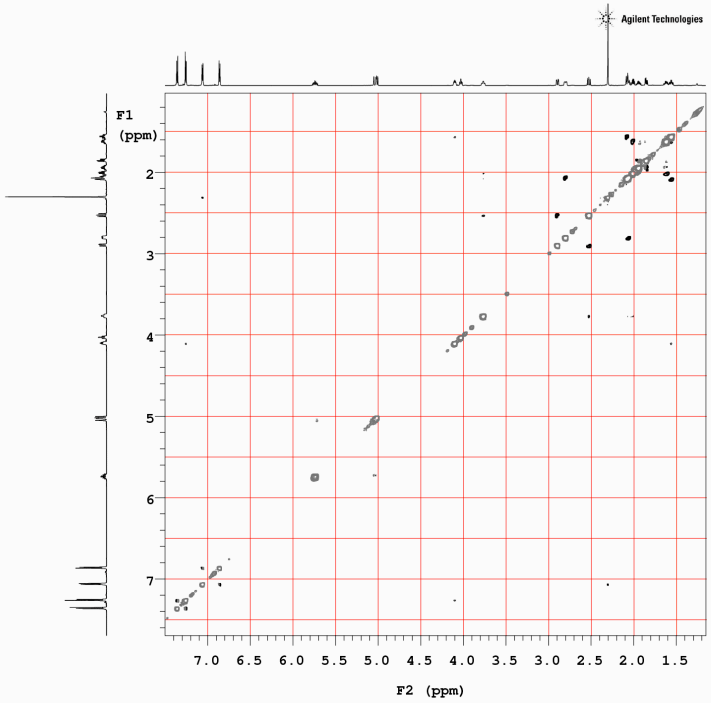
Sample directory:

FidFile: NRB-4-120-NOESY-4tol

Pulse Sequence: NOESY
Solvent: cdcl3
Data collected on: Dec 10 2012

Operator: nbabij

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 5868.5 Hz
2D Width 5868.5 Hz
4 repetitions
2 x 128 increments
OBSERVE H1, 699.7567660 MHz
DATA PROCESSING
Gauss apodization 0.069 sec
F1 DATA PROCESSING
Gauss apodization 0.020 sec
FT size 2048 x 2048
Total time 24 min



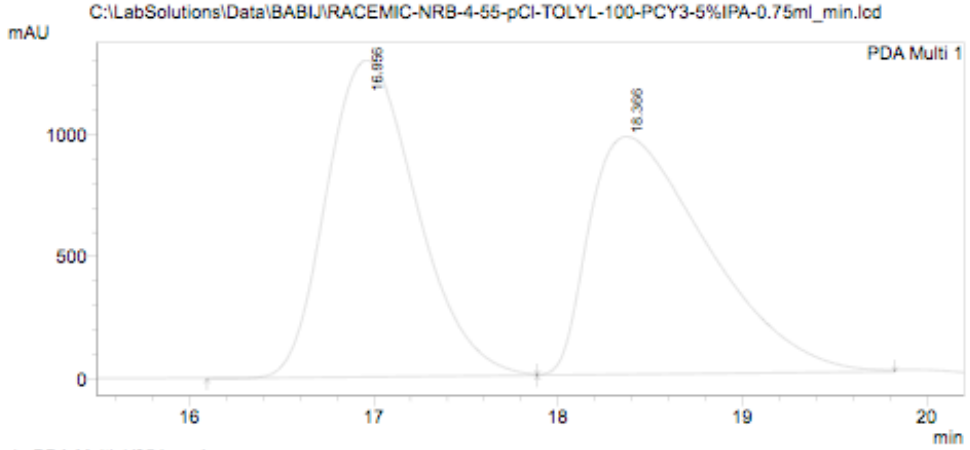
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-55-pCl-TOLYL-100-PCY3-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-55-pCl-TOLYL-100-PCY3-5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-55-pCl-TOLYL-100-PCY3-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 8/8/2012 2:44:19 PM
Data Processed	: 8/8/2012 3:10:15 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-55-pCl-TOLYL-100-PCY3-5%IPA-0.75ml_min.lcd

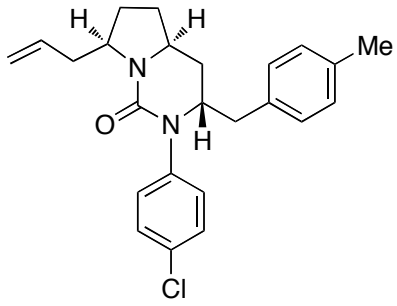


1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	16.956	44268953	1299090	50.371
2	18.366	43617224	974723	49.629
Total		87886176	2273813	100.000



8k

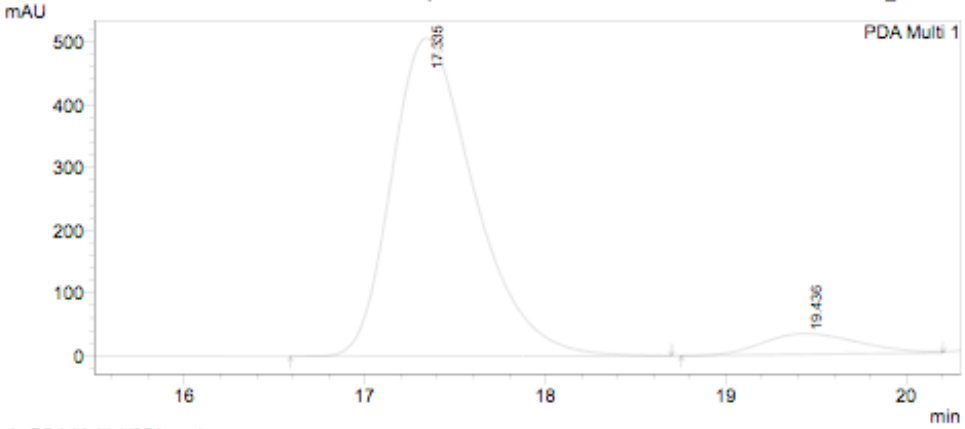
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-92-2-pCl-4-TOLYL-100-NAOtBU-SSIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-92-2-pCl-TOLYL-100-NAOtBU-SSIPHOSPE-5%IPA-0.75ml_m
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-92-2-pCl-4-TOLYL-100-NAOtBU-SSIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/28/2012 4:49:24 PM
Data Processed	: 9/28/2012 5:13:28 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-92-2-pCl-4-TOLYL-100-NAOtBU-SSIPHOSPE-5%IPA-0.75ml_min.lcd

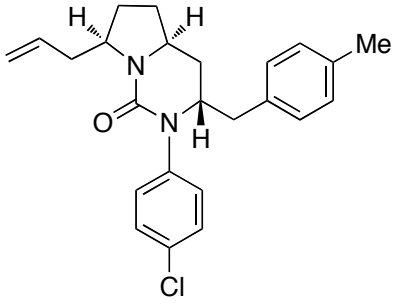


1 PDA Multi 1/254nm 4nm

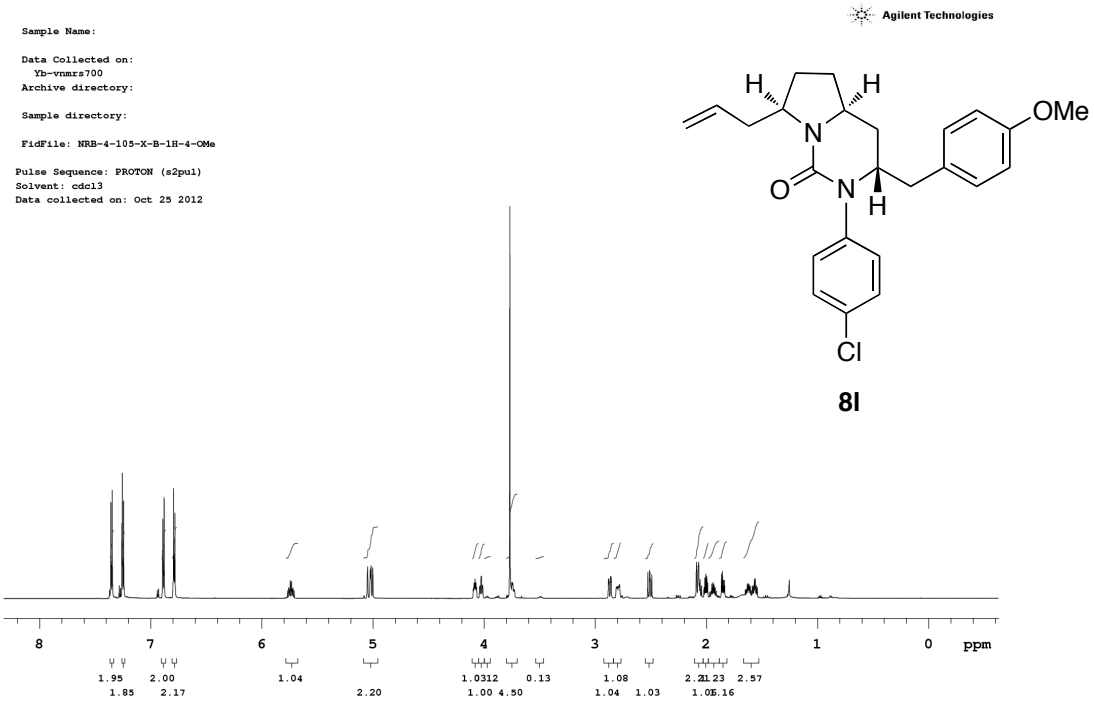
PeakTable

PDA Ch1 254nm 4nm

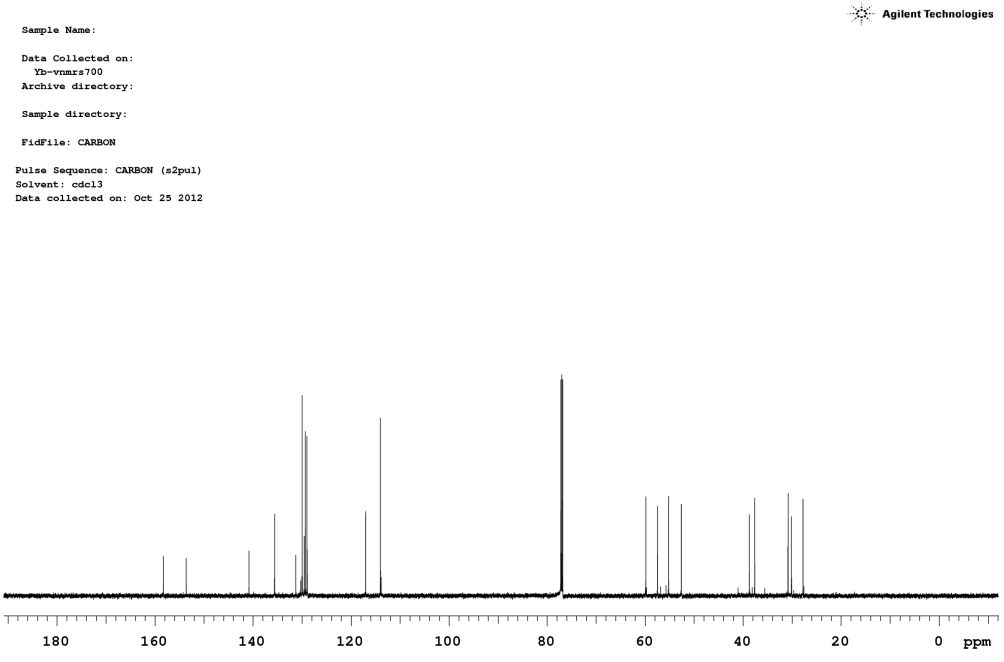
Peak#	Ret. Time	Area	Height	Area %
1	17.335	16104566	506093	92.981
2	19.436	1215674	32944	7.019
Total		17320240	539037	100.000



8k


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-105-X-B-1H-4-OMe
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 25 2012
H
H
N
O
N
H
OMe
Cl
8
7
6
5
4
3
2
1
0
ppm
1.95
2.00
1.04
1.03
3.12
0.13
1.08
2.21
1.23
2.57
1.85
2.17
2.20
1.00
4.50
1.04
1.03
1.05
5.16

8l


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 25 2012
180
160
140
120
100
80
60
40
20
0
ppm

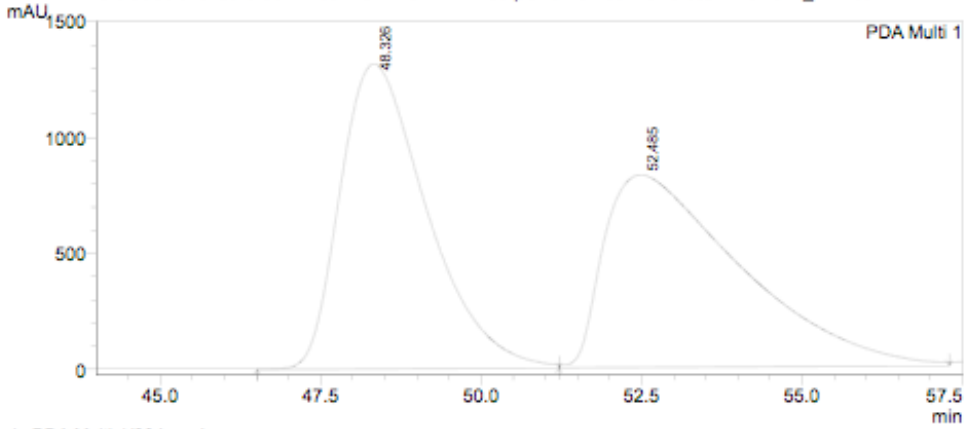
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-86-pCl-OMe-100-PCY3-3%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-86-pCl-OMe-100-PCY3-3%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-86-pCl-OMe-100-PCY3-3%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/29/2012 6:05:51 PM
Data Processed	: 11/29/2012 7:24:45 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-86-pCl-OMe-100-PCY3-3%IPA-0.75ml_min.lcd

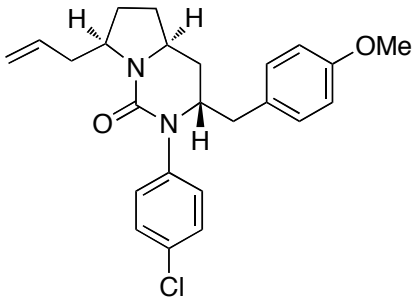


1 PDA Multi 1/204nm 4nm

PeakTable

PDA Ch1 204nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	48.326	123683130	1316190	50.164
2	52.485	122874255	830445	49.836
Total		246557385	2146635	100.000



8l

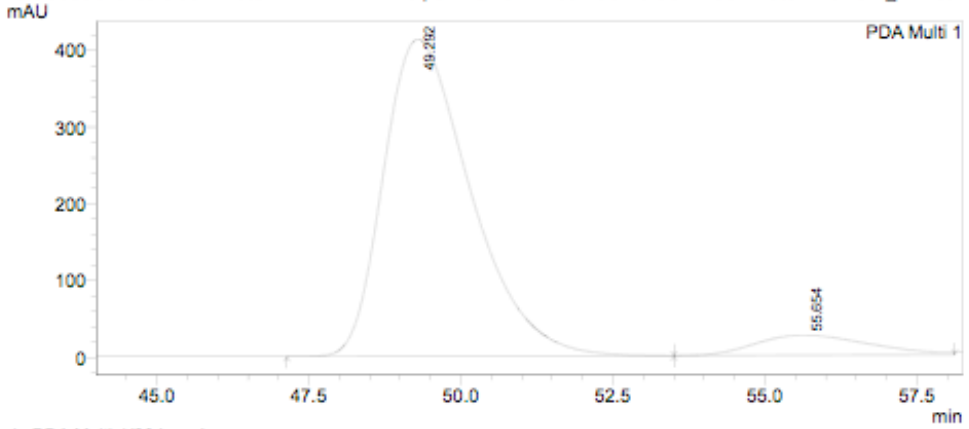
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-116-pCl-4-OMe-21-100-NaOtBu-SSIPHOSPE-3%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-116-pCl-4-OMe-21-100-NaOtBu-SSIPHOSPE-3%IPA-0.75ml
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-116-pCl-4-OMe-21-100-NaOtBu-SSIPHOSPE-3%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/29/2012 4:44:16 PM
Data Processed	: 11/29/2012 6:04:21 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-116-pCl-4-OMe-21-100-NaOtBu-SSIPHOSPE-3%IPA-0.75ml_min.lcd

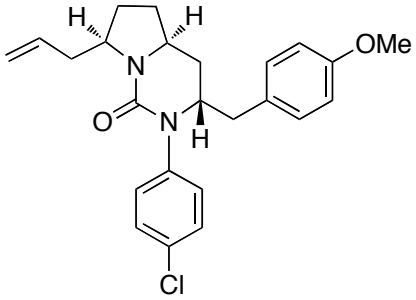


1 PDA Multi 1/204nm 4nm

PeakTable

PDA Ch1 204nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	49.292	41066585	412959	92.385
2	55.654	3384865	26148	7.615
Total		44451450	439107	100.000



8l

Agilent Technologies

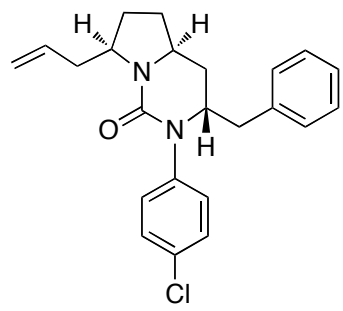
Sample Name:

Data Collected on:
Yb-vnmrs700
Archive directory:

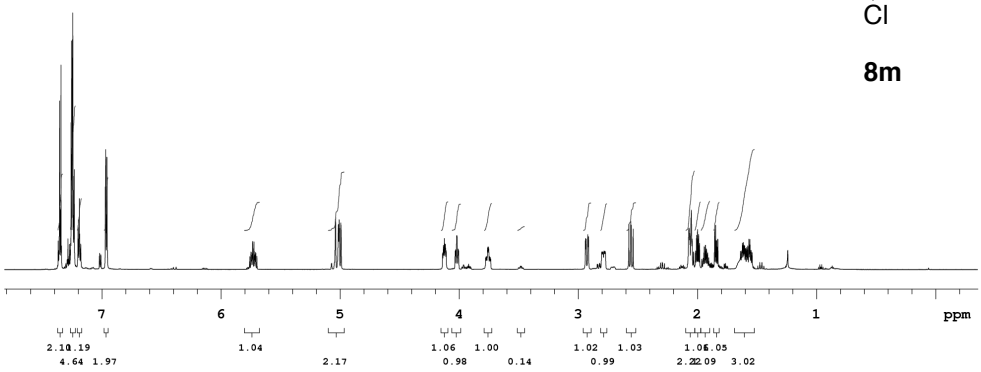
Sample directory:

FidFile: NRB-4-105-E-X-1H-Ph

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 25 2012



8m



Agilent Technologies

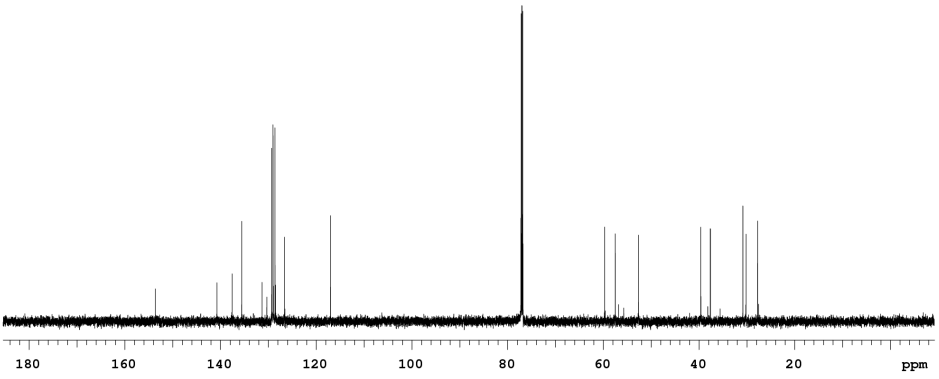
Sample Name:

Data Collected on:
Yb-vnmrs700
Archive directory:

Sample directory:

FidFile: NRB-4-105-E-X-13C-Ph

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 25 2012



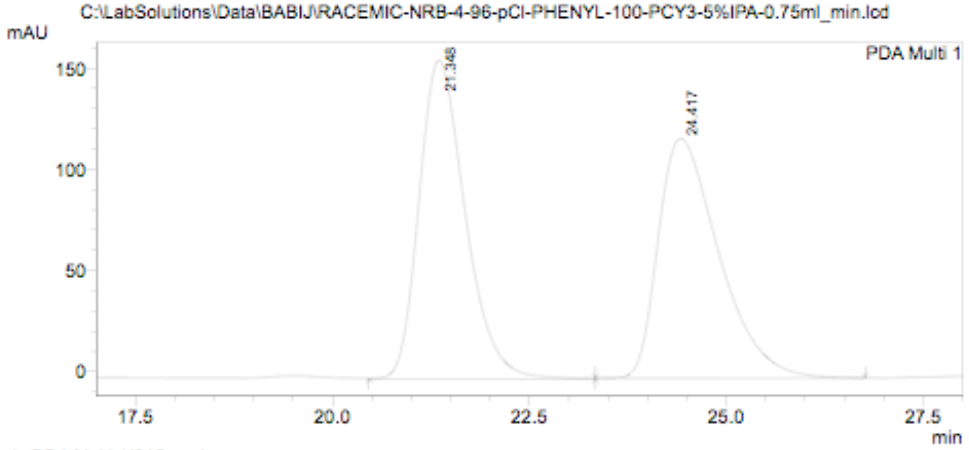
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-96-pCl-PHENYL-100-PCY3-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-96-pCl-PHENYL-100-PCY3-5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-96-pCl-PHENYL-100-PCY3-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/10/2012 2:52:59 PM
Data Processed	: 10/10/2012 3:24:59 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-96-pCl-PHENYL-100-PCY3-5%IPA-0.75ml_min.lcd

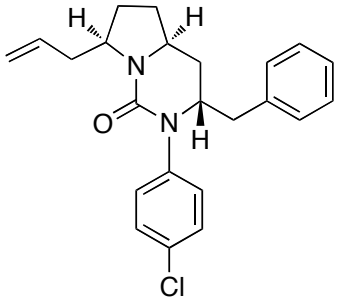


1 PDA Multi 1/245nm 4nm

PeakTable

PDA Ch1 245nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	21.348	6370242	157244	50.083
2	24.417	6349172	118480	49.917
Total		12719414	275724	100.000



8m

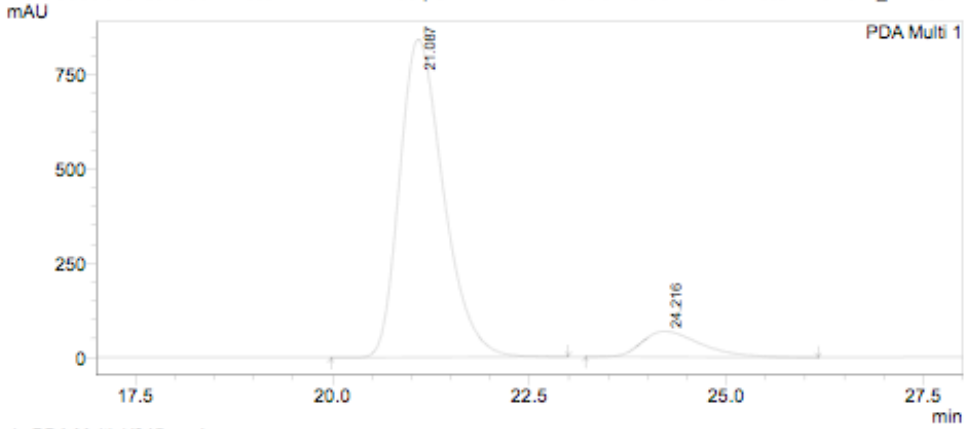
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-96-pCl-PHENYL-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-96-pCl-PHENYL-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_mi
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-96-pCl-PHENYL-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/10/2012 3:37:24 PM
Data Processed	: 10/10/2012 4:11:56 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-96-pCl-PHENYL-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

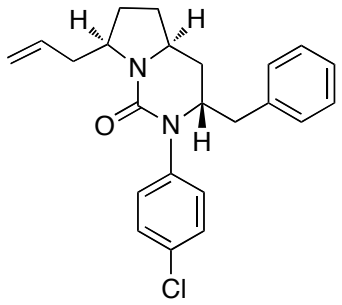


1 PDA Multi 1/245nm 4nm

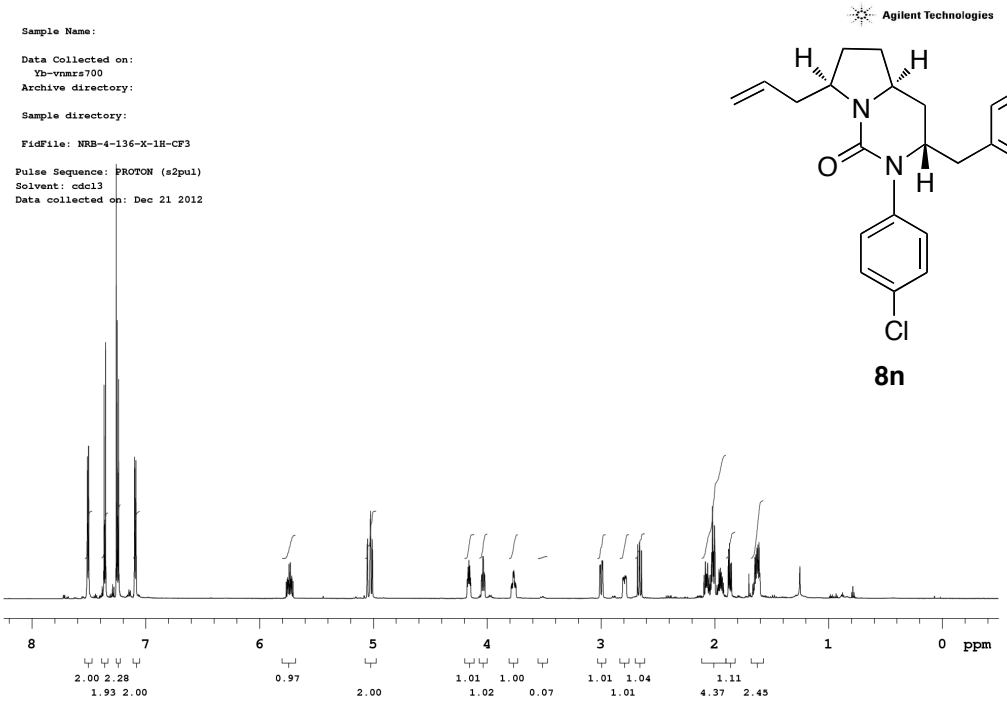
PeakTable

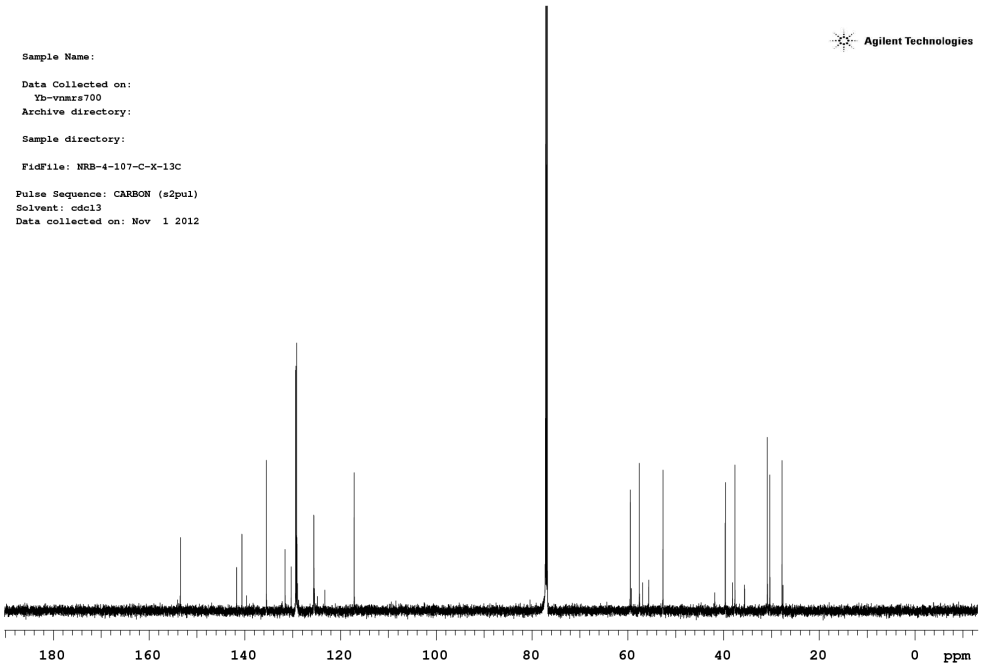
PDA Ch1 245nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	21.087	32587442	843023	90.230
2	24.216	3528682	67790	9.770
Total		36116124	910813	100.000



8m


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-136-X-1H-CF3
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 21 2012
H
H
N
O
N
H
CF3
Cl
8n
8
7
6
5
4
3
2
1
0
ppm
2.00 2.28
1.93 2.00
0.97
2.00
1.01
1.02
1.00
0.07
1.01
1.01
1.04
1.11
4.37
2.45


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-107-C-X-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Nov 1 2012
180
160
140
120
100
80
60
40
20
0
ppm

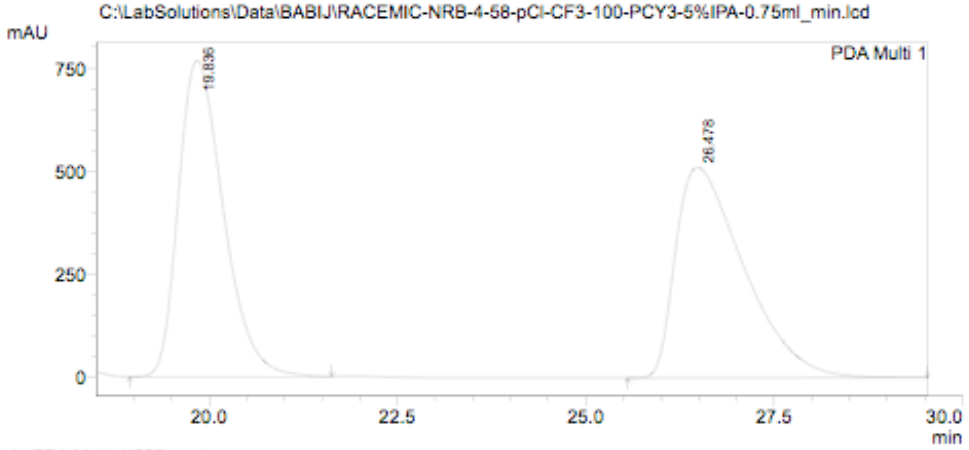
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-58-pCl-CF3-100-PCY3-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-58-pCl-CF3-100-PCY3-5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-58-pCl-CF3-100-PCY3-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 8/10/2012 1:42:35 PM
Data Processed	: 8/10/2012 2:12:09 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-58-pCl-CF3-100-PCY3-5%IPA-0.75ml_min.lcd

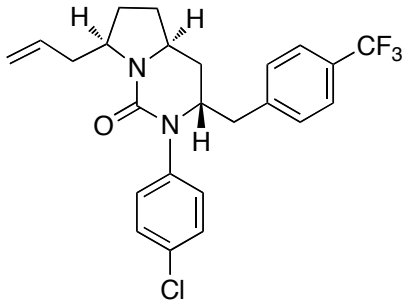


1 PDA Multi 1/205nm 4nm

PeakTable

PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	19.836	31756926	766828	49.635
2	26.478	32223518	510700	50.365
Total		63980444	1277528	100.000



8n

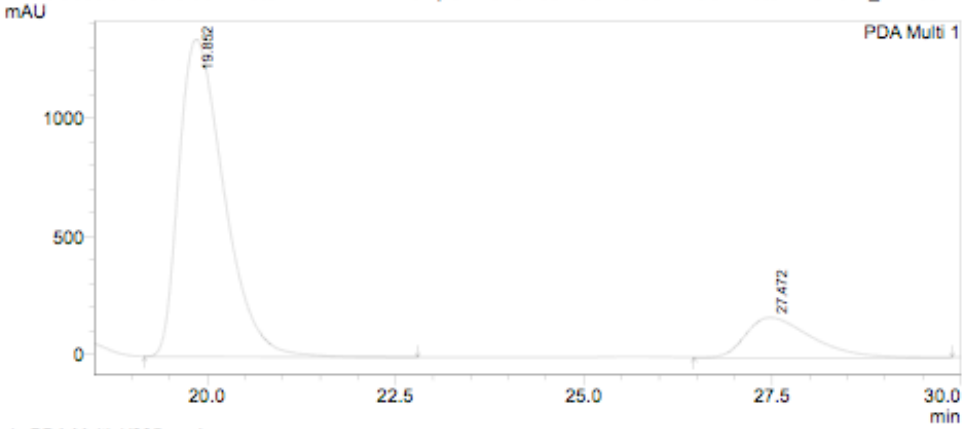
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-107-pCl-4-CF3-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-107-pCl-4-CF3-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_m
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-107-pCl-4-CF3-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 11/1/2012 1:56:29 PM
Data Processed	: 11/1/2012 2:29:27 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-107-pCl-4-CF3-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

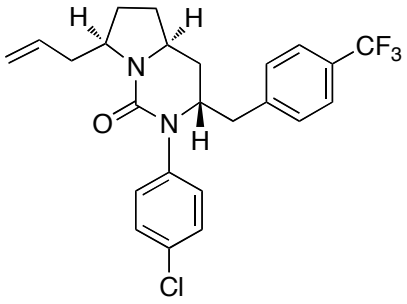


1 PDA Multi 1/205nm 4nm

PeakTable

PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	19.852	55981453	1343838	84.851
2	27.472	9994947	167907	15.149
Total		65976400	1511745	100.000



8n (with NaO*t*Bu)

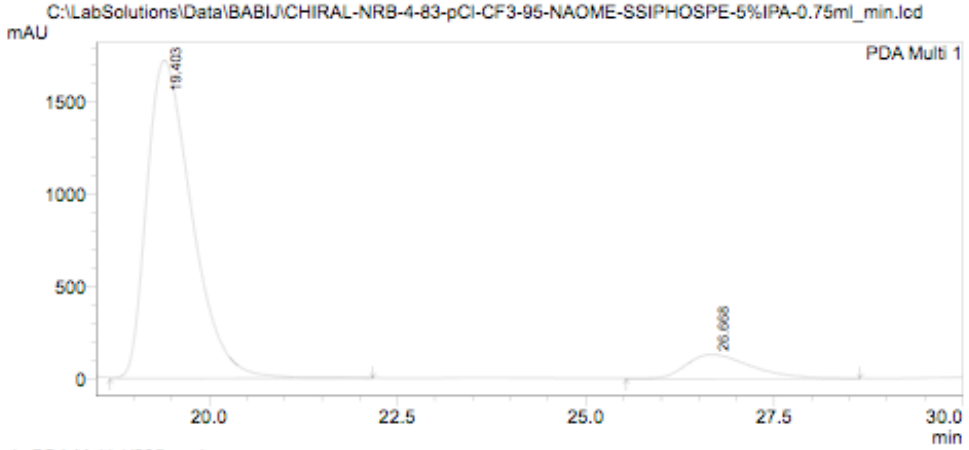
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-83-pCl-CF3-95-NAOME-SSIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-83-pCl-CF3-95-NAOME-SSIPHOSPE-5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-83-pCl-CF3-95-NAOME-SSIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/13/2012 7:30:51 PM
Data Processed	: 9/13/2012 8:02:40 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-83-pCl-CF3-95-NAOME-SSIPHOSPE-5%IPA-0.75ml_min.lcd

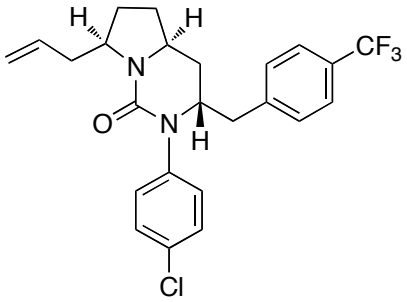


1 PDA Multi 1/205nm 4nm

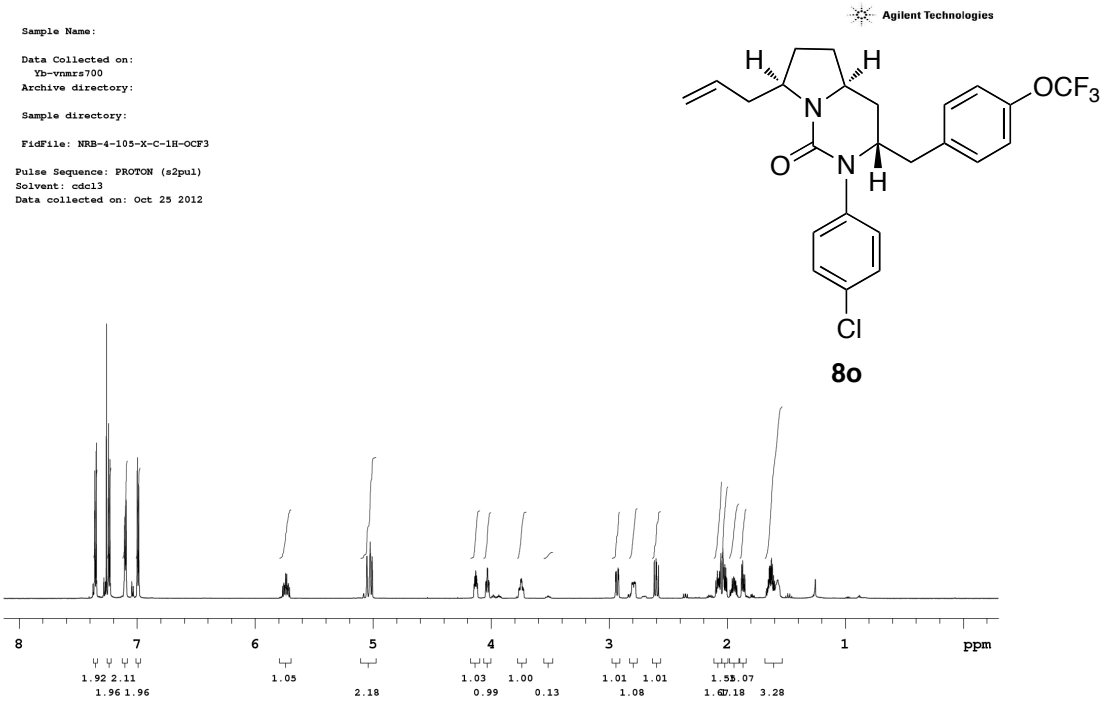
PeakTable

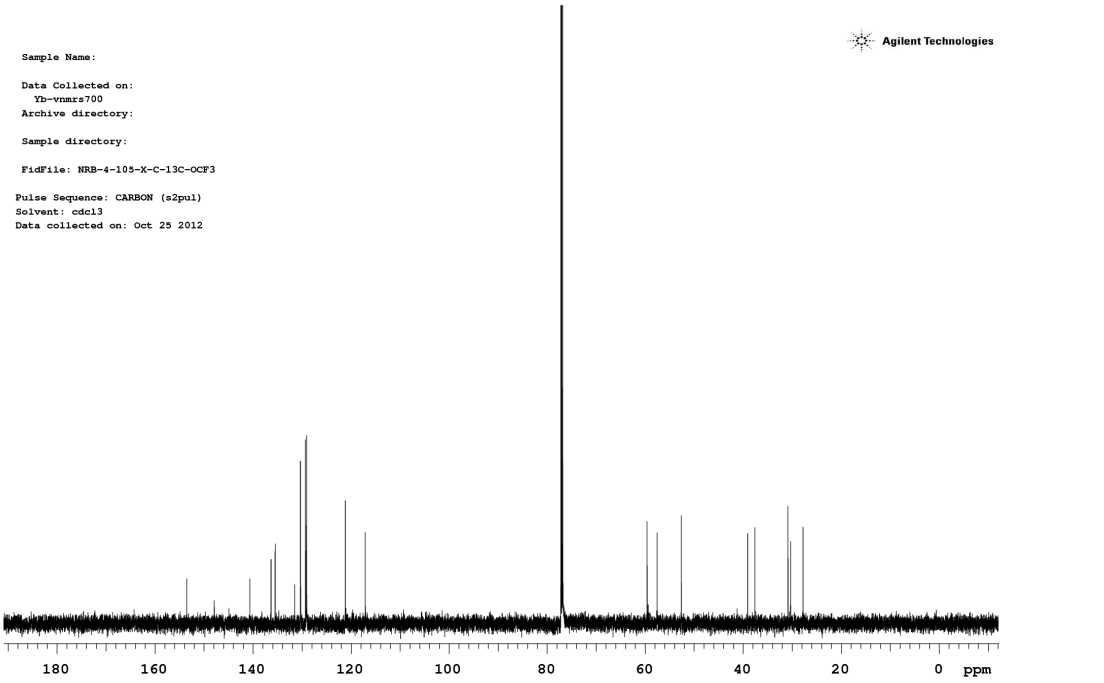
PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	19.403	71981301	1731362	90.036
2	26.668	7966346	131922	9.964
Total		79947647	1863284	100.000



8n (with NaOMe)


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-105-X-C-1H-OCF3
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 25 2012
H
H
N
O
N
H
OCF3
Cl
8o
8
7
6
5
4
3
2
1
ppm
1.92 2.11
1.96 1.96
1.05
2.18
1.03
0.99
1.00
0.13
1.01
1.08
1.01
1.55
1.67
5.07
7.18
3.28


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-105-X-C-13C-OCF3
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 25 2012
180
160
140
120
100
80
60
40
20
0
ppm

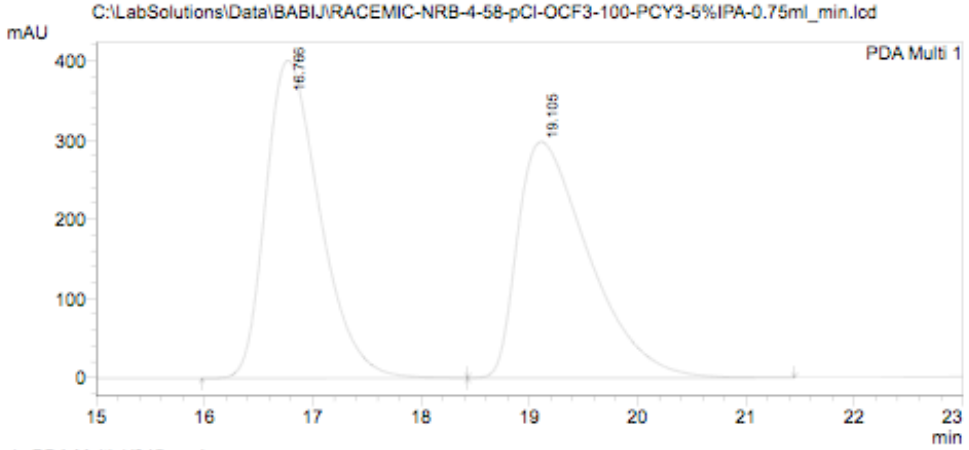
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-58-pCl-OCF3-100-PCY3-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-58-pCl-OCF3-100-PCY3-5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-58-pCl-OCF3-100-PCY3-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 8/10/2012 2:14:53 PM
Data Processed	: 8/10/2012 2:40:44 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-58-pCl-OCF3-100-PCY3-5%IPA-0.75ml_min.lcd

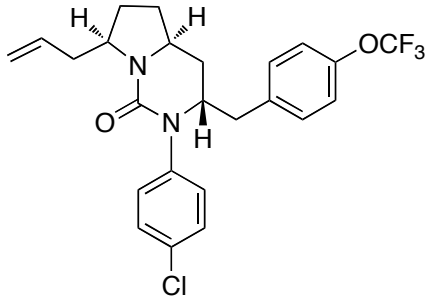


1 PDA Multi 1/245nm 4nm

PeakTable

PDA Ch1 245nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	16.766	13936711	401672	50.472
2	19.105	13675829	298353	49.528
Total		27612540	700025	100.000



8o

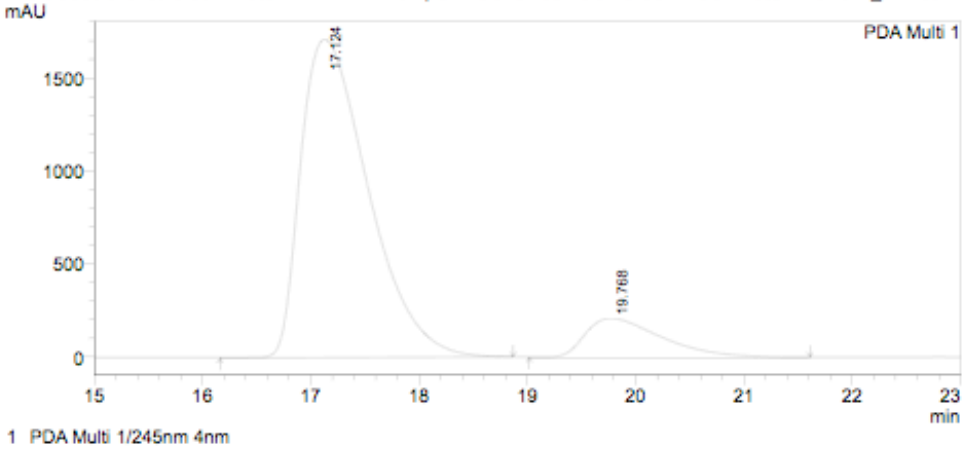
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-94-pCl-4-OCF3-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-94-pCl-4-OCF3-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_mi
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-94-pCl-4-OCF3-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/4/2012 10:46:14 AM
Data Processed	: 10/4/2012 11:09:47 AM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-94-pCl-4-OCF3-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

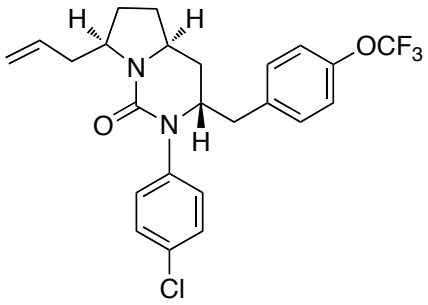


1 PDA Multi 1/245nm 4nm

PeakTable

PDA Ch1 245nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	17.124	74360169	1711279	87.935
2	19.768	10202360	209358	12.065
Total		84562529	1920637	100.000



8o (with NaO*t*Bu)

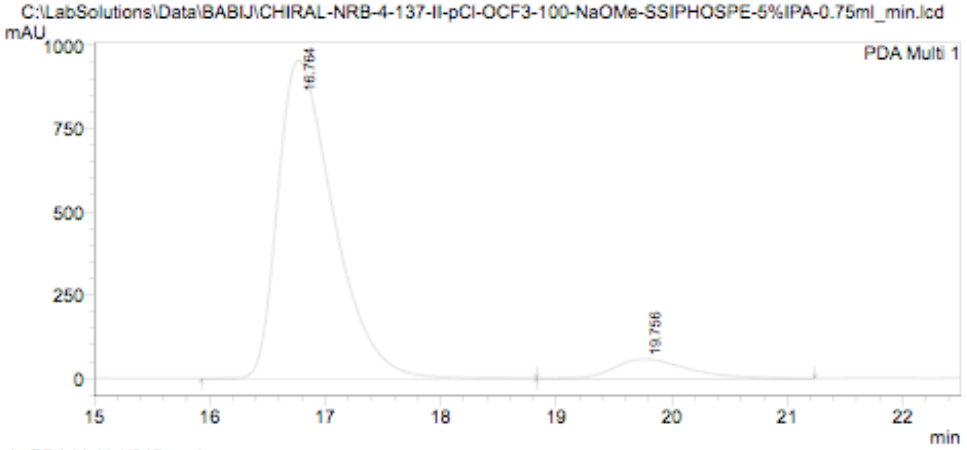
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-137-II-pCl-OCF3-100-NaOMe-SSIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-137-I-pCl-OCF3-100-NaOMe-SSIPHOSPE-5%IPA-0.75ml_mi
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-137-II-pCl-OCF3-100-NaOMe-SSIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 12/23/2012 5:45:35 PM
Data Processed	: 12/23/2012 6:13:19 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-137-II-pCl-OCF3-100-NaOMe-SSIPHOSPE-5%IPA-0.75ml_min.lcd

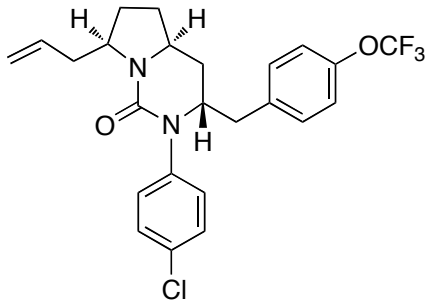


1 PDA Multi 1/245nm 4nm

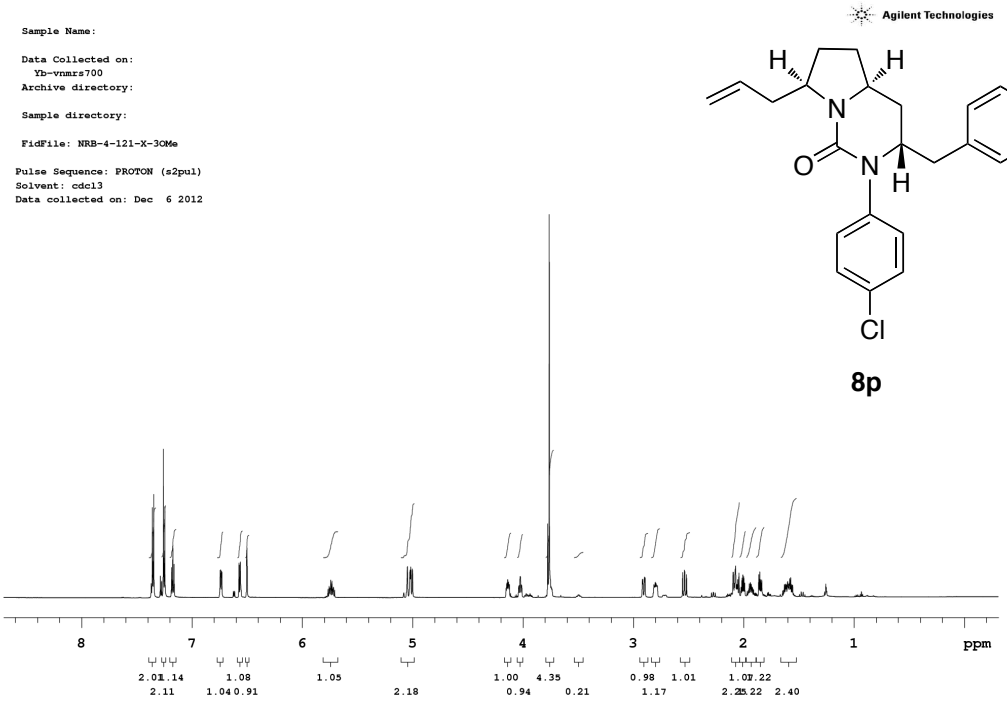
PeakTable

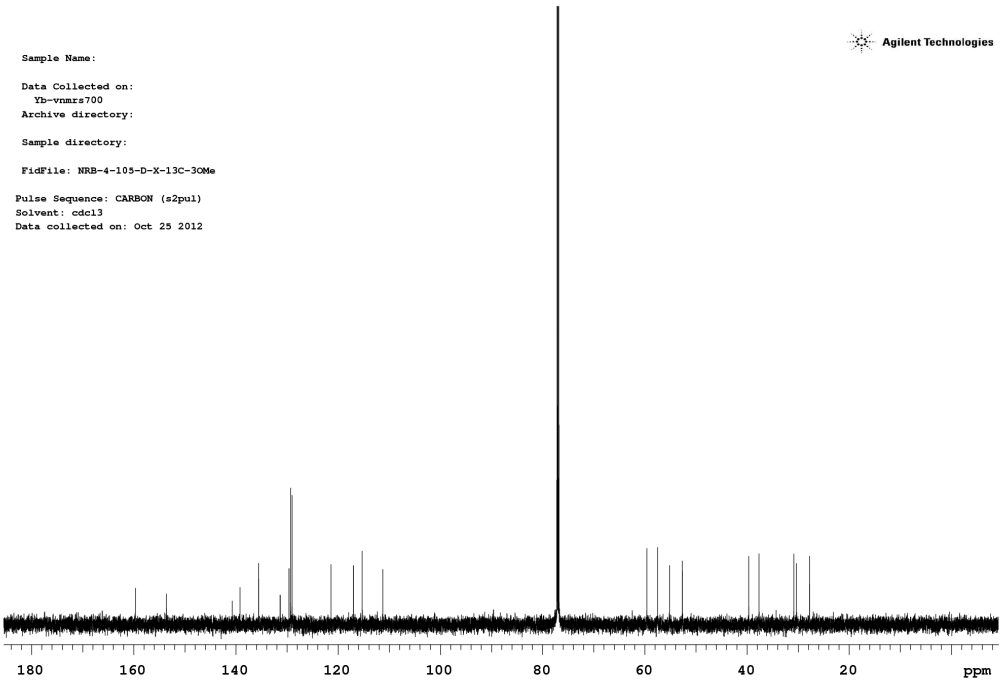
PDA Ch1 245nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	16.764	31880825	954387	92.755
2	19.756	2490104	57122	7.245
Total		34370929	1011509	100.000



8o (with NaOMe)


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-121-X-3OMe
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 6 2012
H
H
N
O
N
H
OMe
Cl
8p
8
7
6
5
4
3
2
1
ppm
2.01 1.14 1.08 1.05 1.00 4.35 0.98 1.01 1.07 7.22
2.11 1.04 0.91 2.18 0.94 0.21 1.17 2.25 5.22 2.40


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-105-D-X-13C-3OMe
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 25 2012
180
160
140
120
100
80
60
40
20
ppm

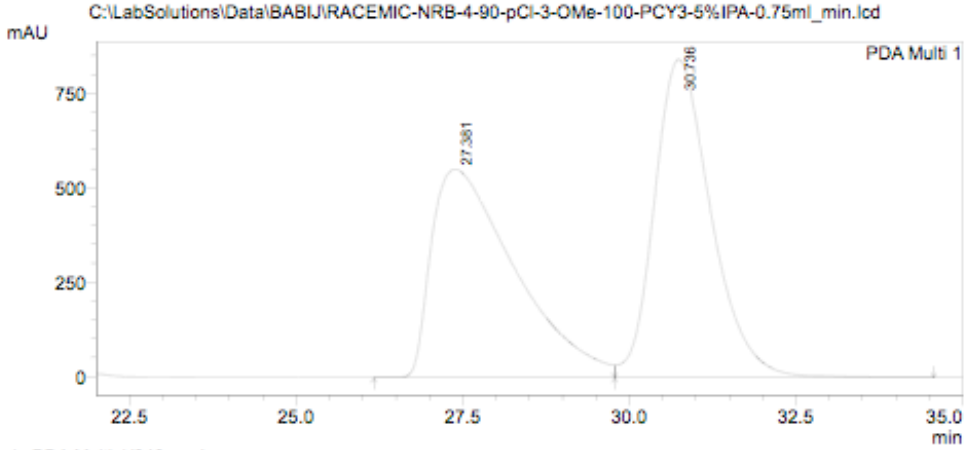
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-90-pCl-3-OMe-100-PCY3-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-90-pCl-3-OMe-100-PCY3-5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-90-pCl-3-OMe-100-PCY3-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/25/2012 7:29:03 PM
Data Processed	: 9/25/2012 8:05:08 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-90-pCl-3-OMe-100-PCY3-5%IPA-0.75ml_min.lcd

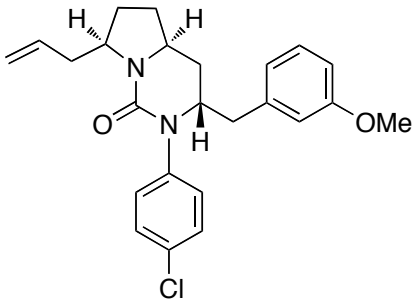


1 PDA Multi 1/248nm 4nm

PeakTable

PDA Ch1 248nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	27.381	48187230	552654	49.026
2	30.736	50102213	843446	50.974
Total		98289443	1396100	100.000



8p

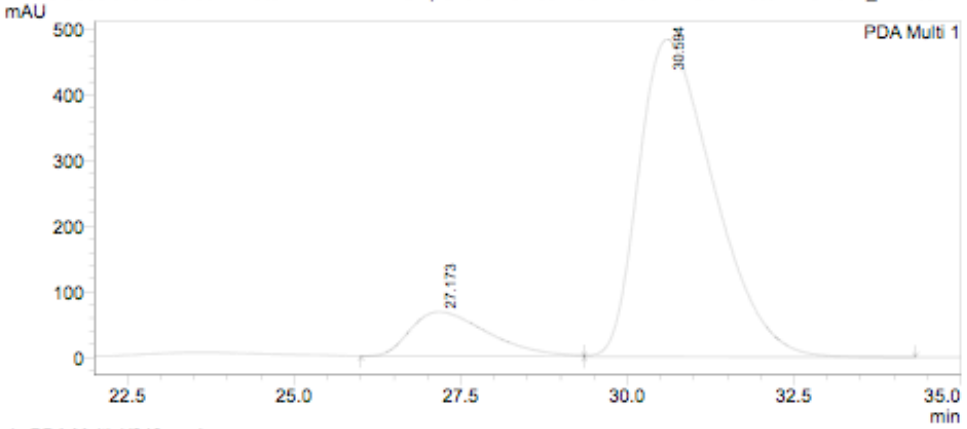
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-94-pCl-3-OMe-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-94-pCl-3-OMe-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-94-pCl-3-OMe-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/4/2012 3:33:10 PM
Data Processed	: 10/4/2012 4:08:43 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-94-pCl-3-OMe-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

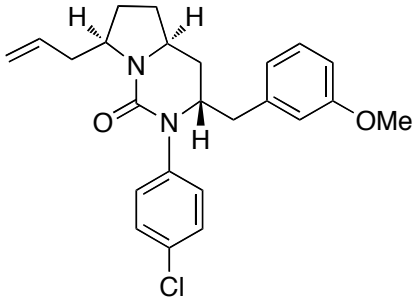


1 PDA Multi 1/248nm 4nm

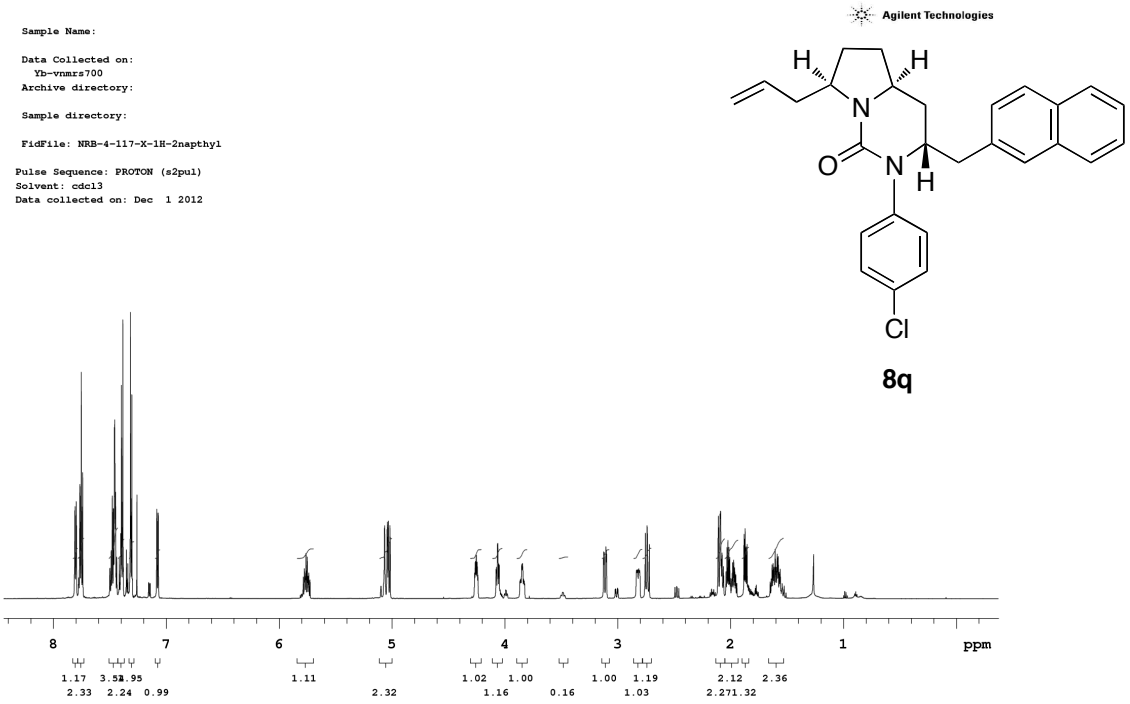
PeakTable

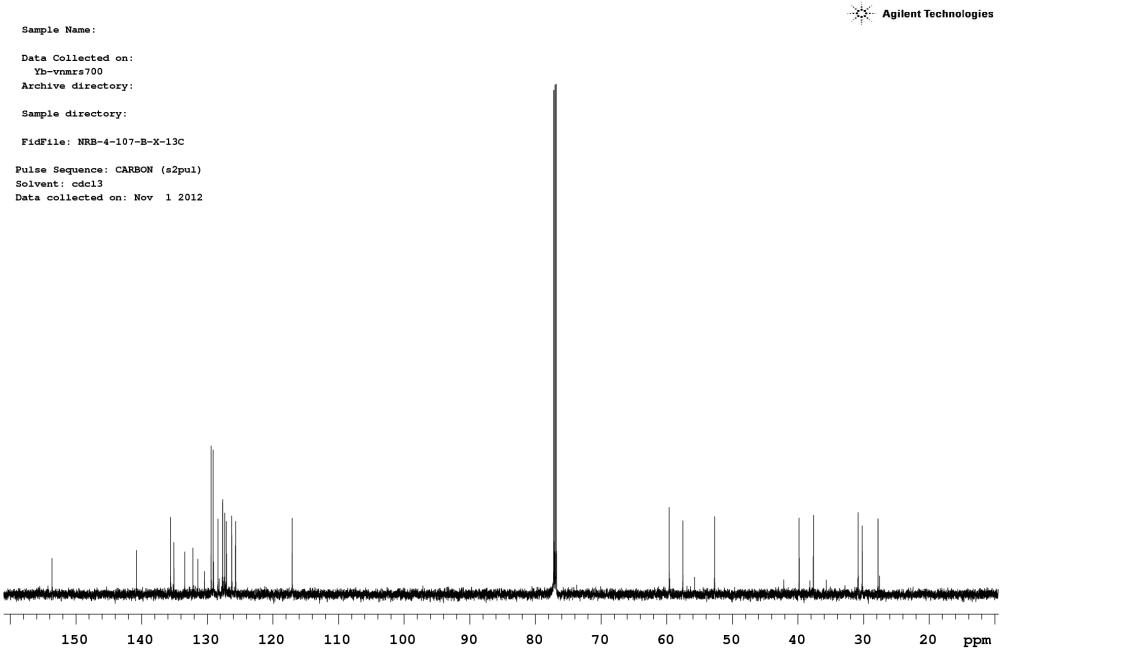
PDA Ch1 248nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	27.173	5497076	67634	12.683
2	30.594	37845830	483921	87.317
Total		43342907	551556	100.000



8p


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-117-X-1H-2napthyl
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 1 2012
H
H
N
O
N
H
Cl
8q
8
7
6
5
4
3
2
1
ppm
1.17 3.53 1.95 1.11 1.02 1.00 1.00 1.19 2.12 2.36
2.33 2.24 0.99 2.32 1.16 0.16 1.03 2.27 1.32


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-107-B-X-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Nov 1 2012
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

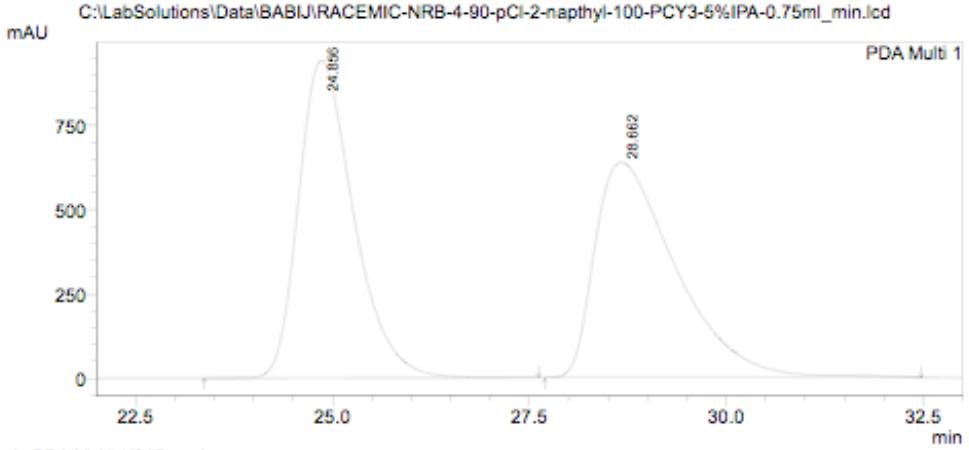
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-90-pCl-2-napthyl-100-PCY3-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-90-pCl-2-napthyl-100-PCY3-5%IPA-0.75ml_min.lcd
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-90-pCl-2-napthyl-100-PCY3-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/26/2012 1:51:54 PM
Data Processed	: 9/26/2012 2:25:28 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-90-pCl-2-napthyl-100-PCY3-5%IPA-0.75ml_min.lcd

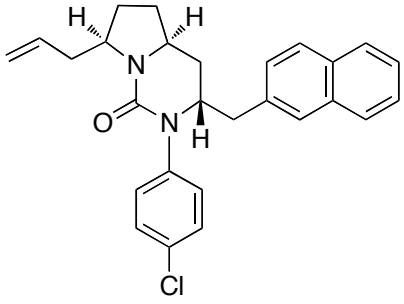


1 PDA Multi 1/215nm 4nm

PeakTable

PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	24.856	45755831	941390	49.764
2	28.662	46190576	639545	50.236
Total		91946407	1580935	100.000



8q

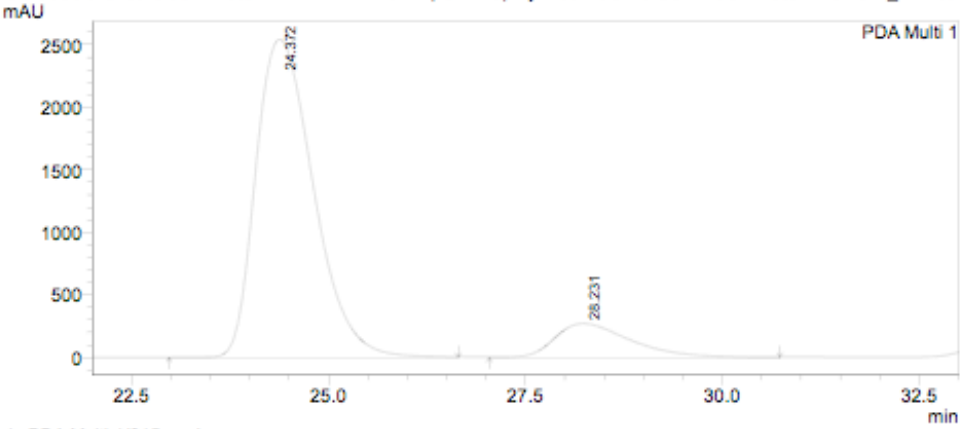
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-107-2-pCl-2-napthyl-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-107-2-pCl-2-napth-100-SSIPHOSPE-5%IPA-0.75ml_
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-107-2-pCl-2-napthyl-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/31/2012 5:21:45 PM
Data Processed	: 10/31/2012 6:05:47 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-107-2-pCl-2-napthyl-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

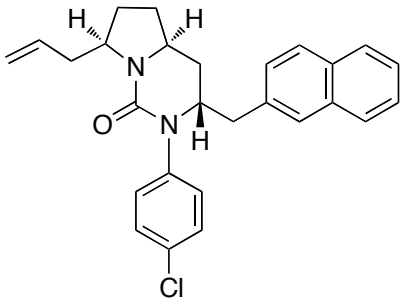


1 PDA Multi 1/215nm 4nm

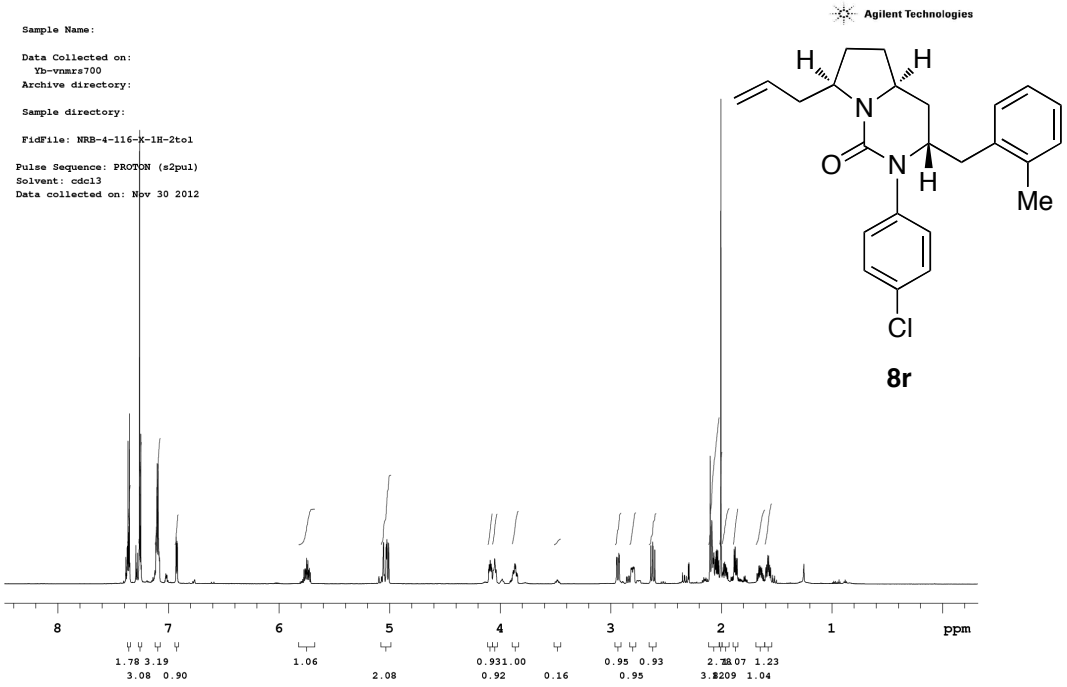
PeakTable

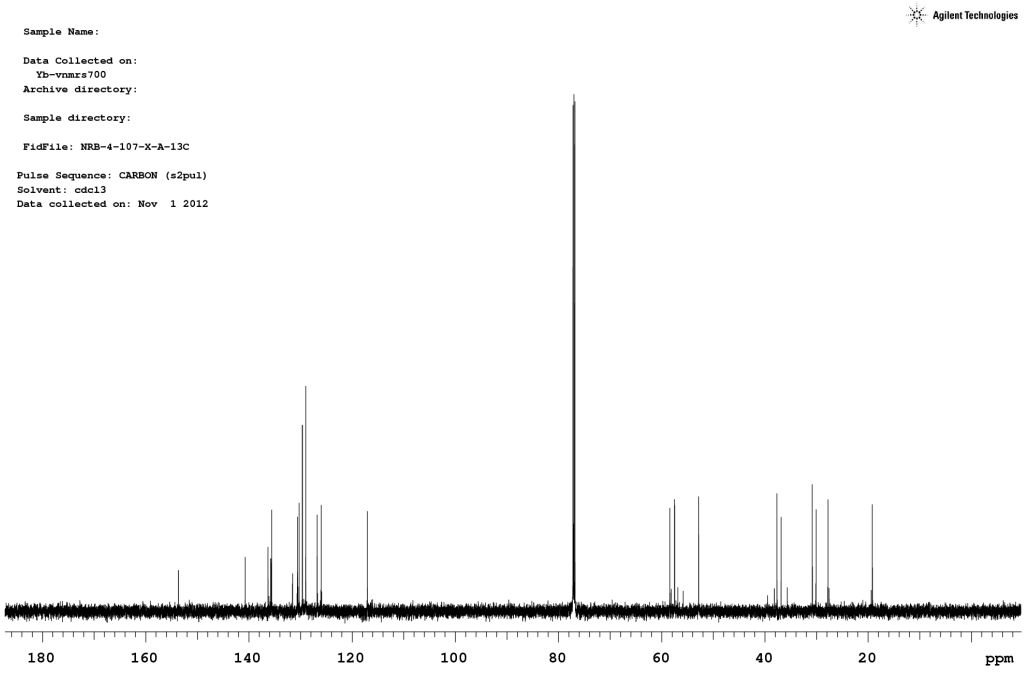
PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	24.372	129657052	2544179	87.682
2	28.231	18215094	271035	12.318
Total		147872146	2815214	100.000



8q


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-116-X-1H-2tol
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Nov 30 2012
H
H
N
O
N
H
Me
Cl
8r
8
7
6
5
4
3
2
1
ppm
1.78 3.19
3.08 0.90
1.06
2.08
0.93 1.00
0.92 0.16
0.95 0.93
0.95
1.23
1.04


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-107-X-A-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Nov 1 2012
180
160
140
120
100
80
60
40
20
ppm

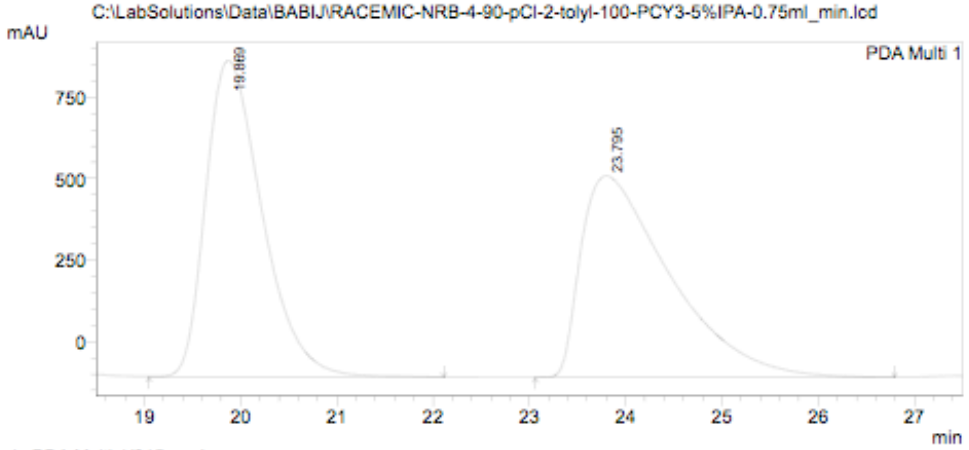
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-90-pCl-2-tolyl-100-PCY3-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: RACEMIC-NRB-4-90-pCl-2-tolyl-100-PCY3-5%IPA-0.75ml_min
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: RACEMIC-NRB-4-90-pCl-2-tolyl-100-PCY3-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 9/26/2012 11:30:07 AM
Data Processed	: 9/26/2012 12:10:21 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\RACEMIC-NRB-4-90-pCl-2-tolyl-100-PCY3-5%IPA-0.75ml_min.lcd

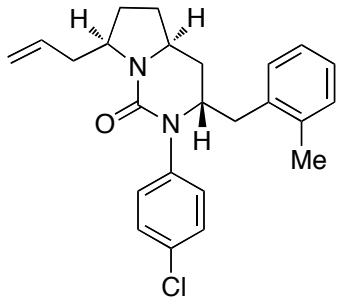


1 PDA Multi 1/215nm 4nm

PeakTable

PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	19.869	39671328	972024	49.912
2	23.795	39810829	618614	50.088
Total		79482157	1590638	100.000



8r

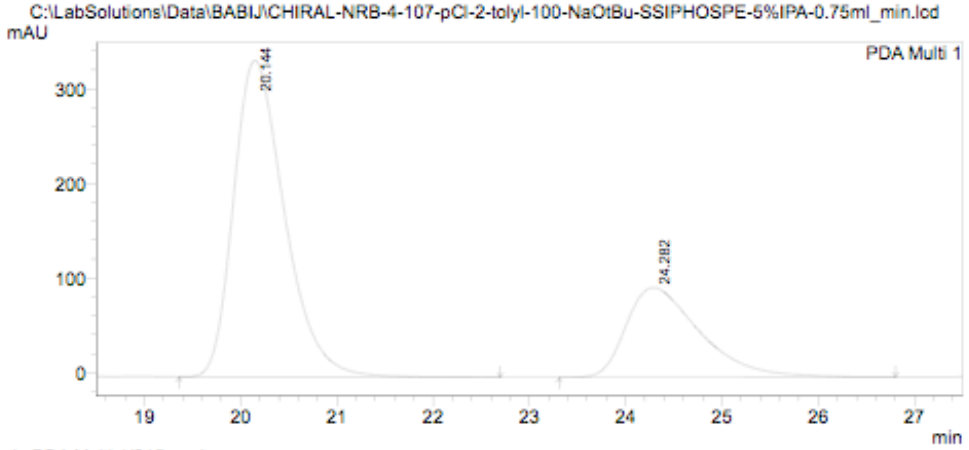
==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-107-pCl-2-tolyl-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

Acquired by	: Admin
Sample Name	: CHIRAL-NRB-4-107-pCl-2-tolyl-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_
Sample ID	: <SAMPLE>
Tray#	: 1
Vail #	: 1
Injection Volume	: 1 uL
Data File Name	: CHIRAL-NRB-4-107-pCl-2-tolyl-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd
Method File Name	: Cyclic Urea Method.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 10/31/2012 3:36:37 PM
Data Processed	: 10/31/2012 4:33:50 PM

<Chromatogram>

C:\LabSolutions\Data\BABIJ\CHIRAL-NRB-4-107-pCl-2-tolyl-100-NaOtBu-SSIPHOSPE-5%IPA-0.75ml_min.lcd

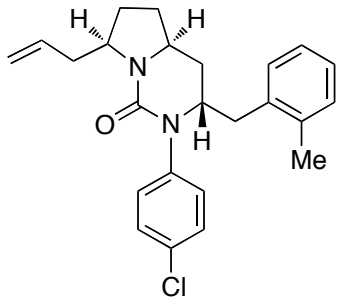


1 PDA Multi 1/215nm 4nm

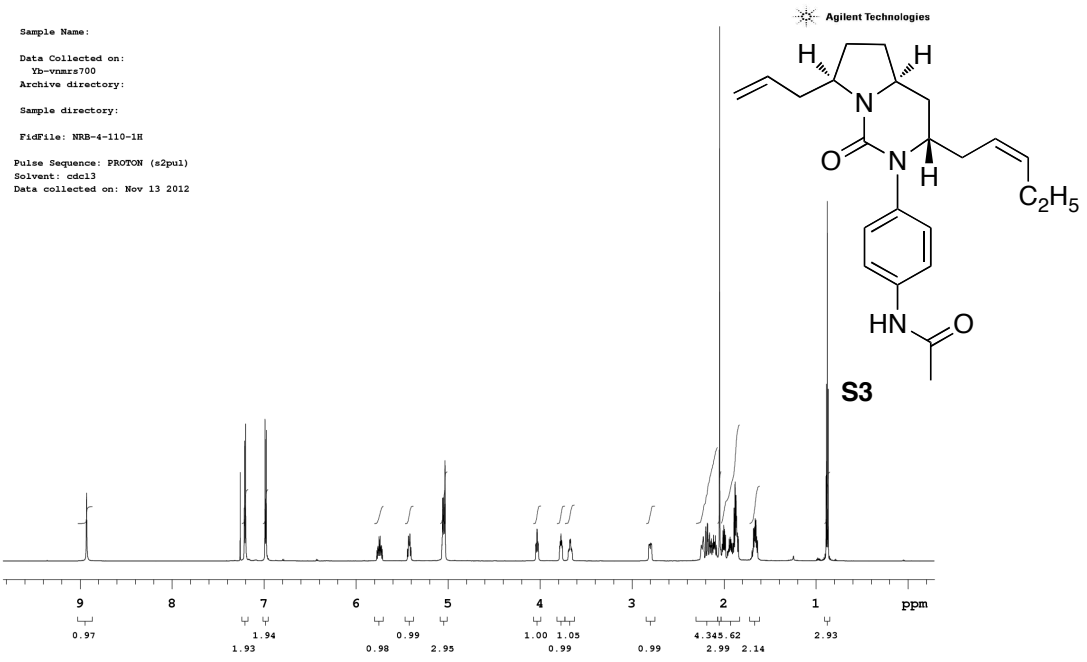
PeakTable

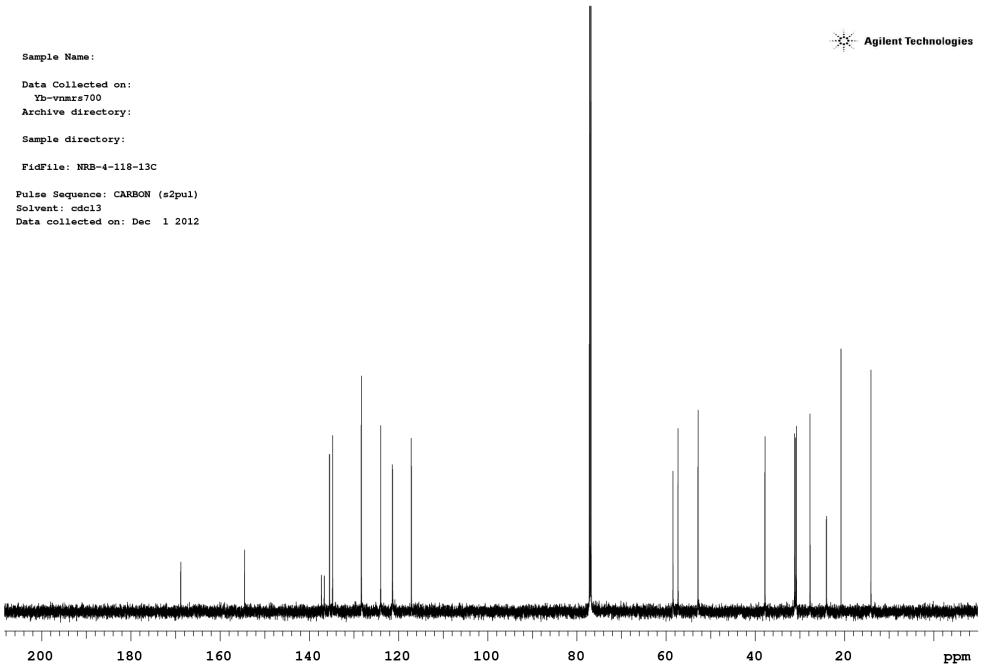
PDA Ch1 215nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	20.144	12340532	334865	70.853
2	24.282	5076517	94929	29.147
Total		17417049	429794	100.000



8r


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-110-1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Nov 13 2012
H
H
N
O
N
H
C_2H_5
HN
O
S3
9
8
7
6
5
4
3
2
1
ppm
0.97
1.94
1.93
0.99
0.98
2.95
1.00
1.05
0.99
0.99
4.34
5.62
2.99
2.14
2.93


Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-118-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Dec 1 2012
200
180
160
140
120
100
80
60
40
20
ppm

Agilent Technologies

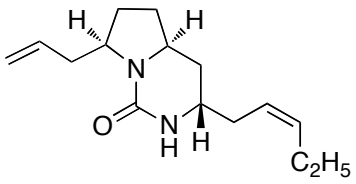
Sample Name:

Data Collected on:
Sn.Chem.LSA.UMich.edu-inova500
Archive directory:

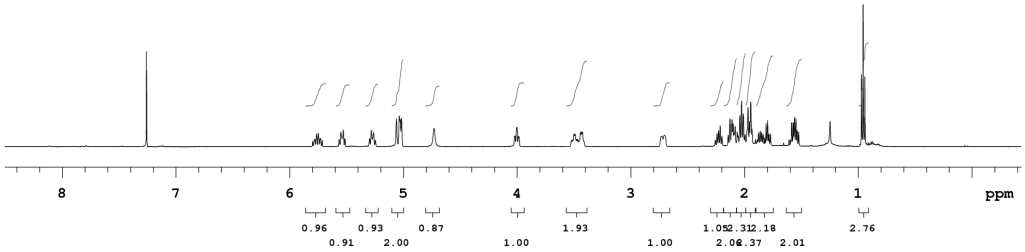
Sample directory:

FidFile: NRB-4-122-1H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Dec 13 2012



9



Agilent Technologies

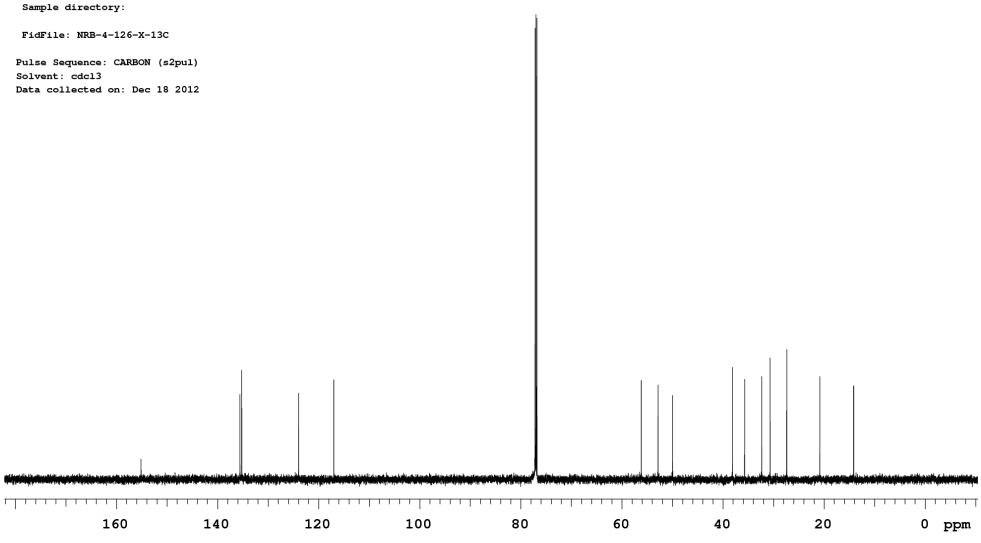
Sample Name:

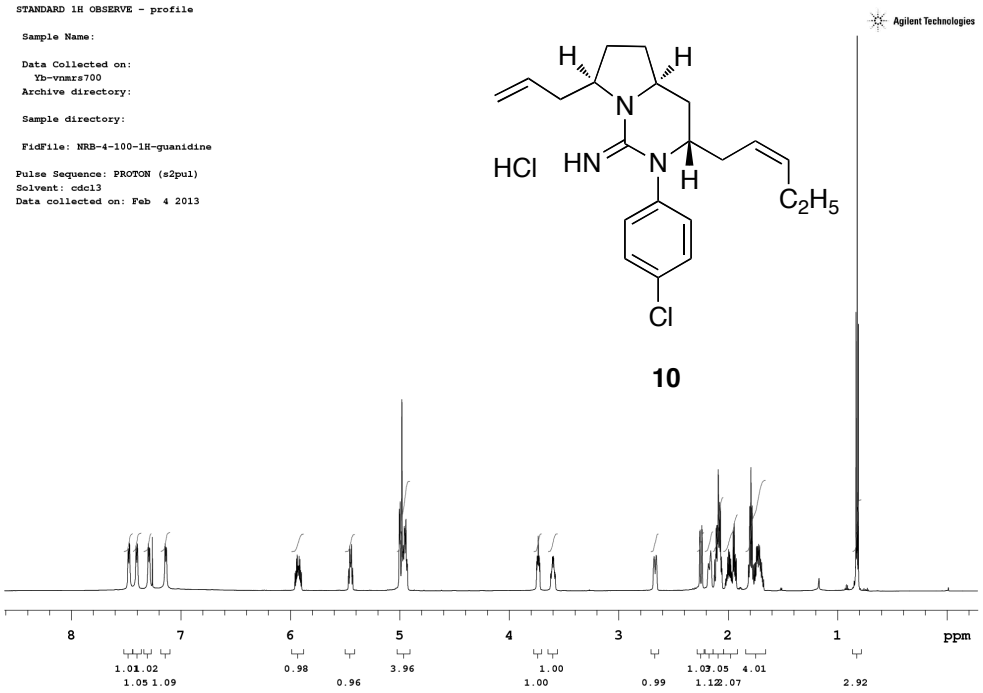
Data Collected on:
Yb-vnmrs700
Archive directory:

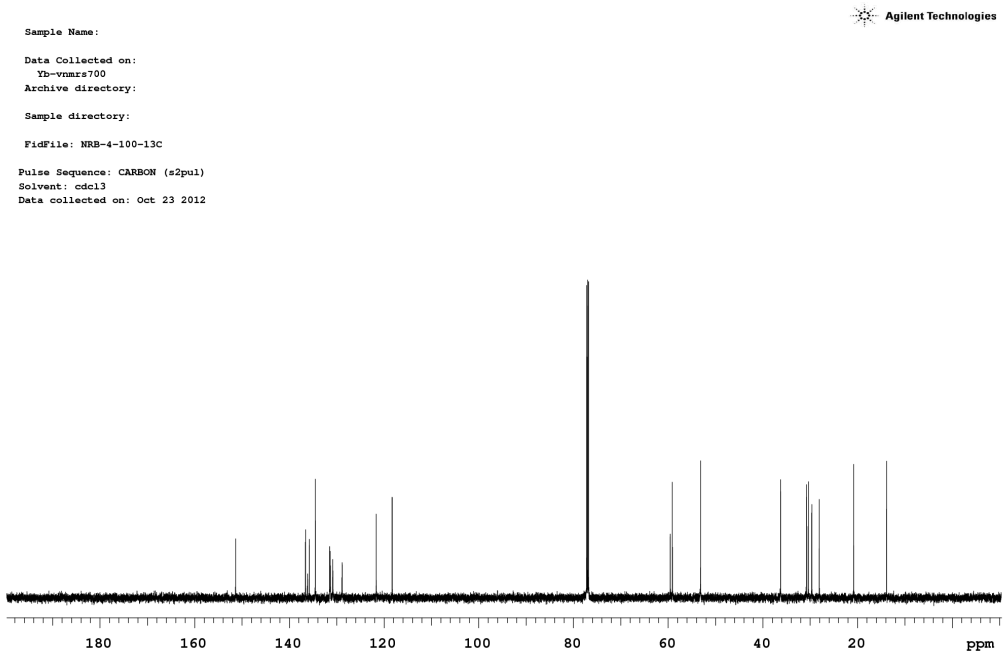
Sample directory:

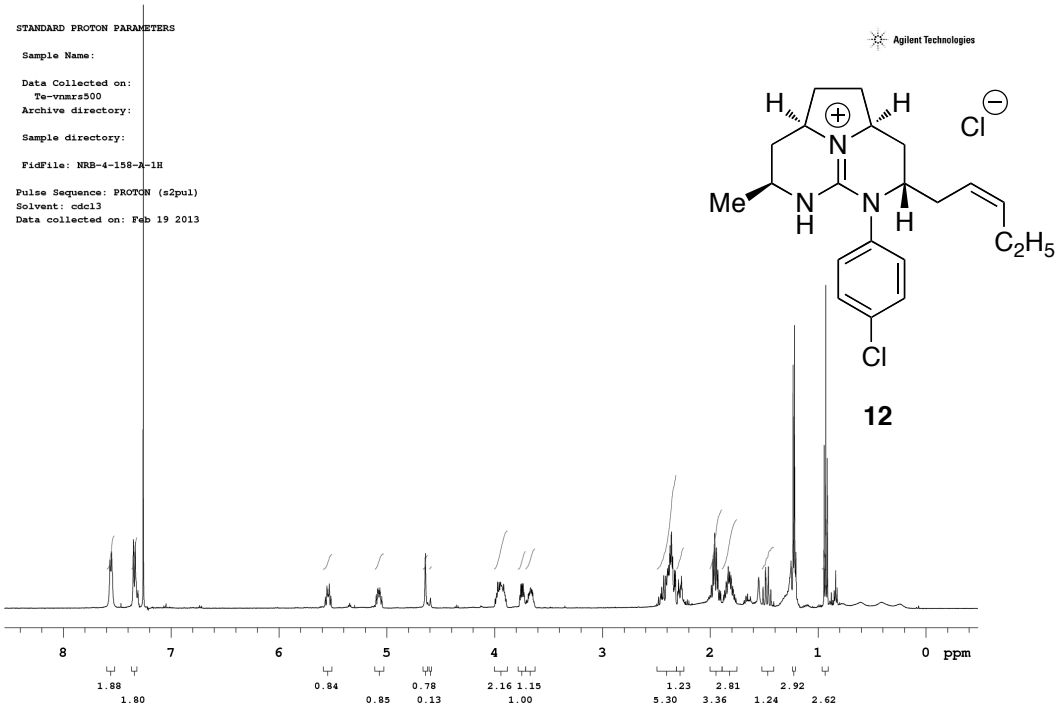
FidFile: NRB-4-126-X-13C

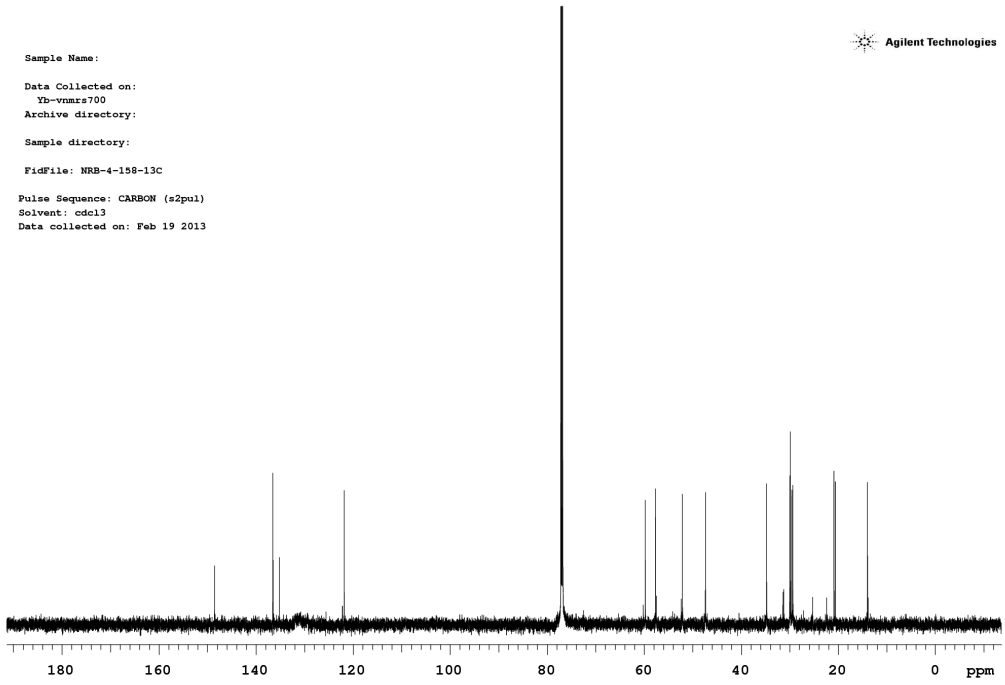
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Dec 18 2012

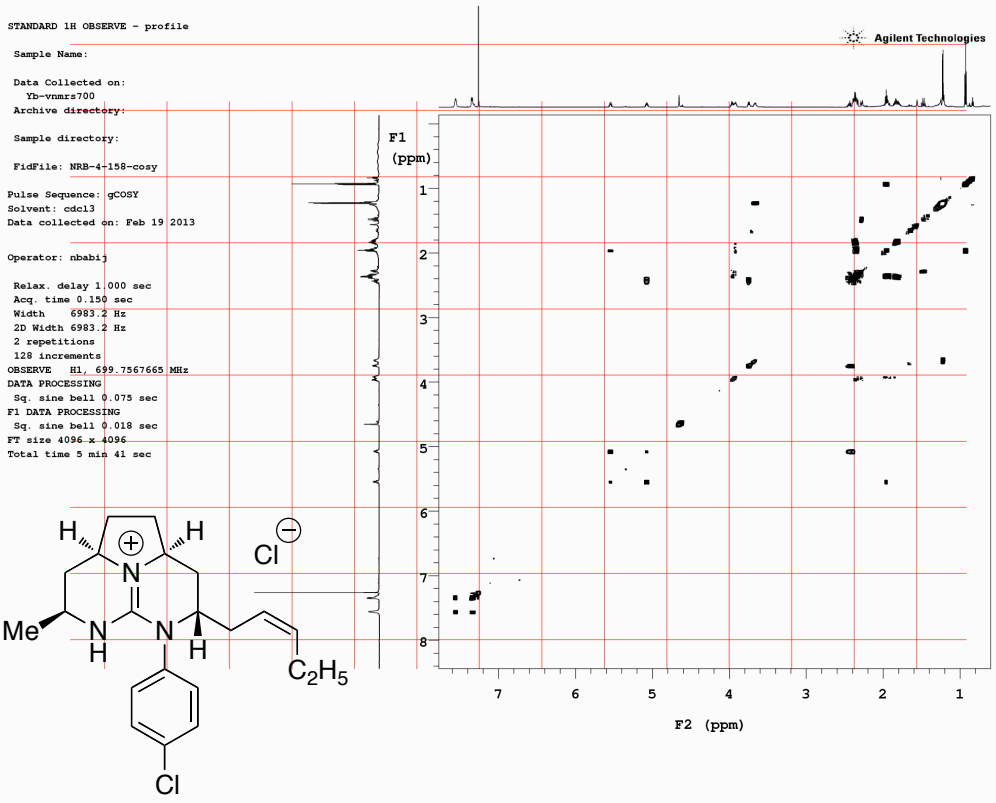



STANDARD 1H OBSERVE - profile
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-100-1H-guanidine
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 4 2013
Agilent Technologies
H
H
N
HCl
HN
N
H
C2H5
Cl
10
8
7
6
5
4
3
2
1
ppm
1.01 1.02
1.05 1.09
0.98
0.96
3.96
1.00
1.00
0.99
1.03 3.05 4.01
1.12 2.07
2.92

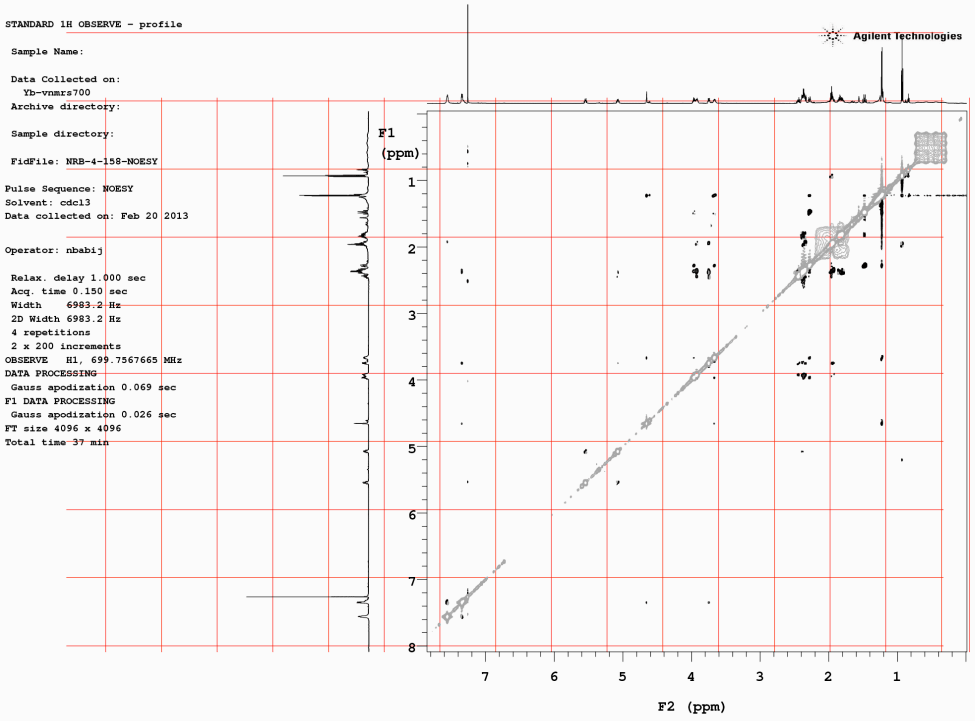

Agilent Technologies
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-100-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 23 2012
180
160
140
120
100
80
60
40
20
ppm

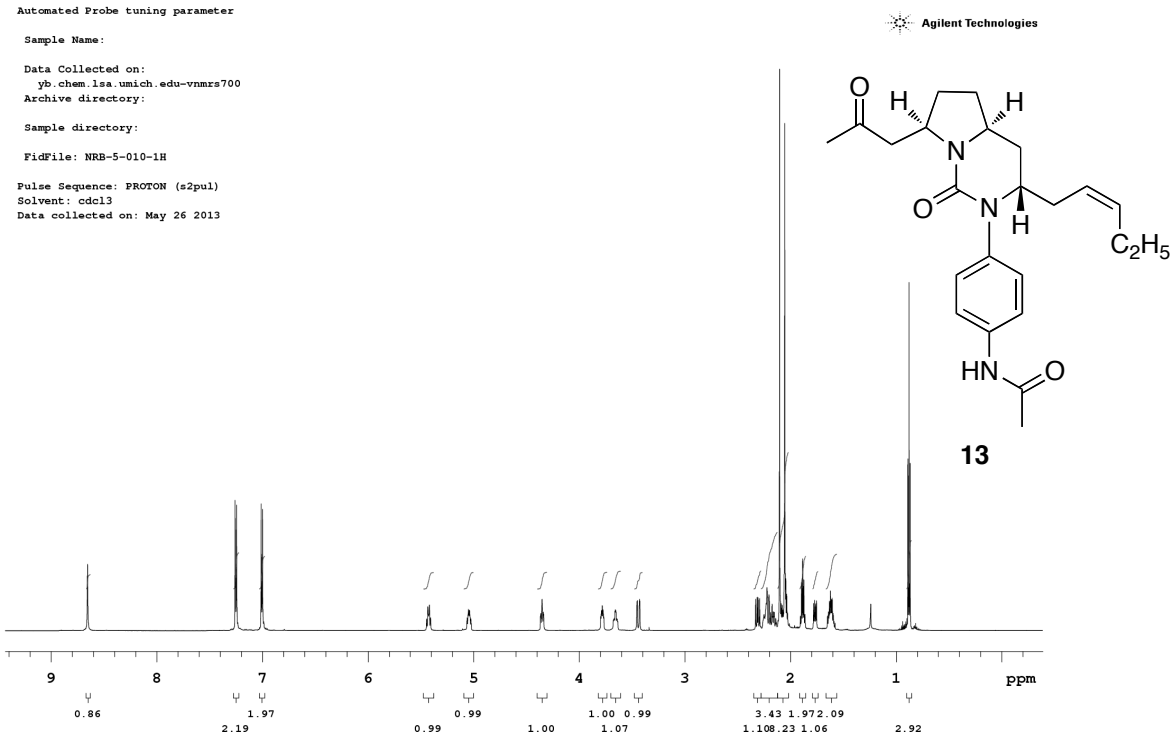

STANDARD PROTON PARAMETERS
Sample Name:
Data Collected on:
Te-vnmrs500
Archive directory:
Sample directory:
FidFile: NRB-4-158-A-1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Feb 19 2013
Agilent Technologies
H
H
N
Cl
Me
N
H
N
H
C_2H_5
Cl
12
8
7
6
5
4
3
2
1
0 ppm
1.88
1.80
0.84
0.85
0.78
0.13
2.16
1.15
1.00
1.23
5.30
2.81
3.36
2.92
1.24
2.62

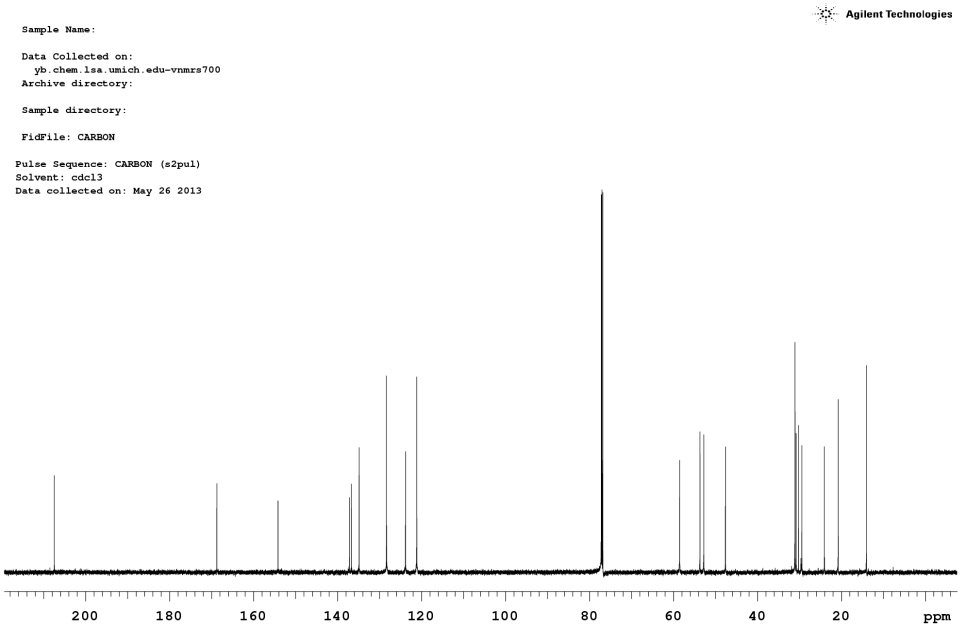

Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-158-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 19 2013
Agilent Technologies
180
160
140
120
100
80
60
40
20
0 ppm

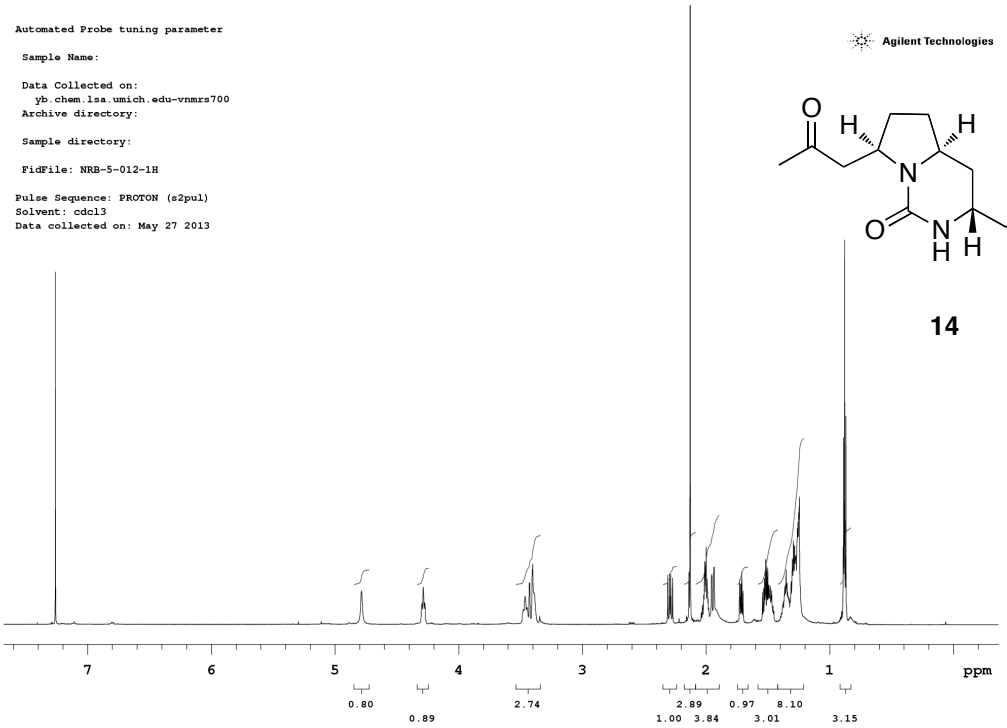

STANDARD 1H OBSERVE - profile
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-158-cosy
Pulse Sequence: gCOSY
Solvent: cdcl3
Data collected on: Feb 19 2013
Operator: nbabij
Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 6983.2 Hz
2D Width 6983.2 Hz
2 repetitions
128 increments
OBSERVE H1, 699.7567665 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Sq. sine bell 0.018 sec
FT size 4096 x 4096
Total time 5 min 41 sec
Agilent Technologies
F1 (ppm)
F2 (ppm)
H
H
N
Cl
Me
N
H
N
H
C_2H_5
Cl

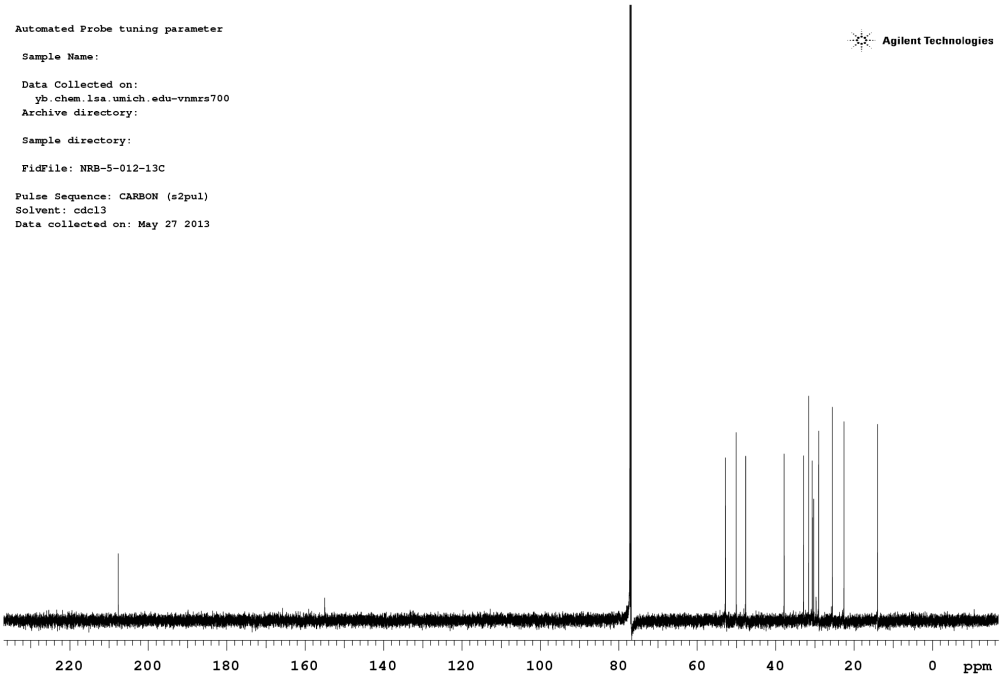
12

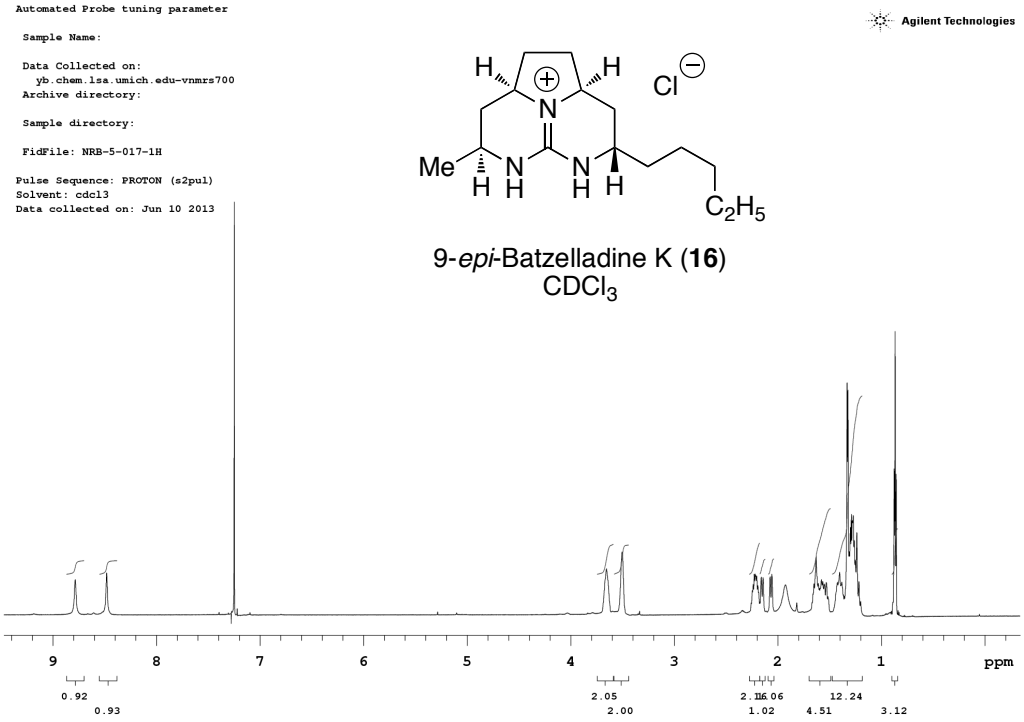

STANDARD 1H OBSERVE - profile
Sample Name:
Data Collected on:
Yb-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-4-158-NOESY
Pulse Sequence: NOESY
Solvent: cdcl3
Data collected on: Feb 20 2013
Operator: nbabij
Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 6983.2 Hz
2D Width 6983.2 Hz
4 repetitions
2 x 200 increments
OBSERVE H1, 699.7567665 MHz
DATA PROCESSING
Gauss apodization 0.069 sec
F1 DATA PROCESSING
Gauss apodization 0.026 sec
FT size 4096 x 4096
Total time 37 min
Agilent Technologies
F1 (ppm)
F2 (ppm)

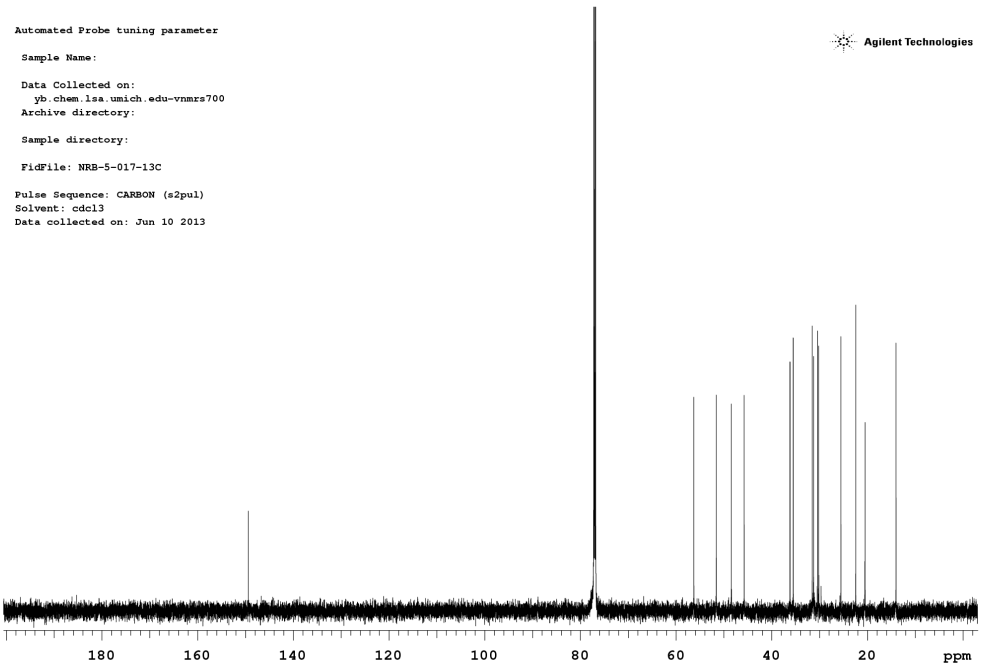

Automated Probe tuning parameter
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-5-010-1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 26 2013
Agilent Technologies
O
H
H
N
O
N
H
C_2H_5
HN
O
13
9
8
7
6
5
4
3
2
1
ppm
0.86
1.97
2.19
0.99
0.99
1.00
1.00
0.99
1.07
3.43
1.97
2.09
1.10
8.23
1.06
2.92

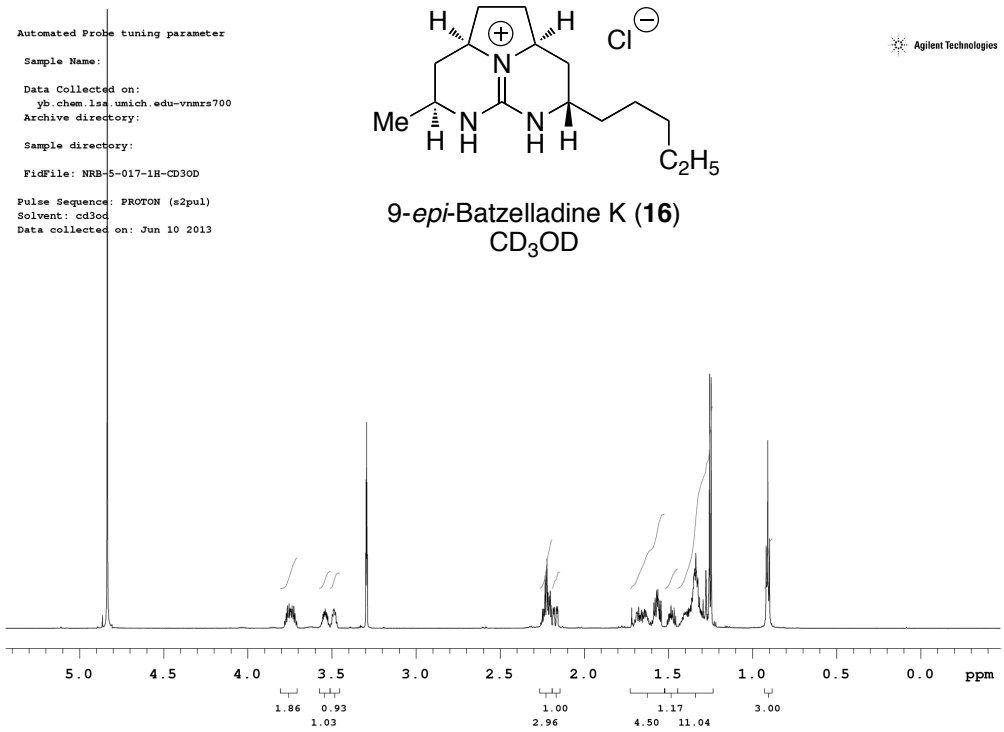

Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 26 2013
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
ppm

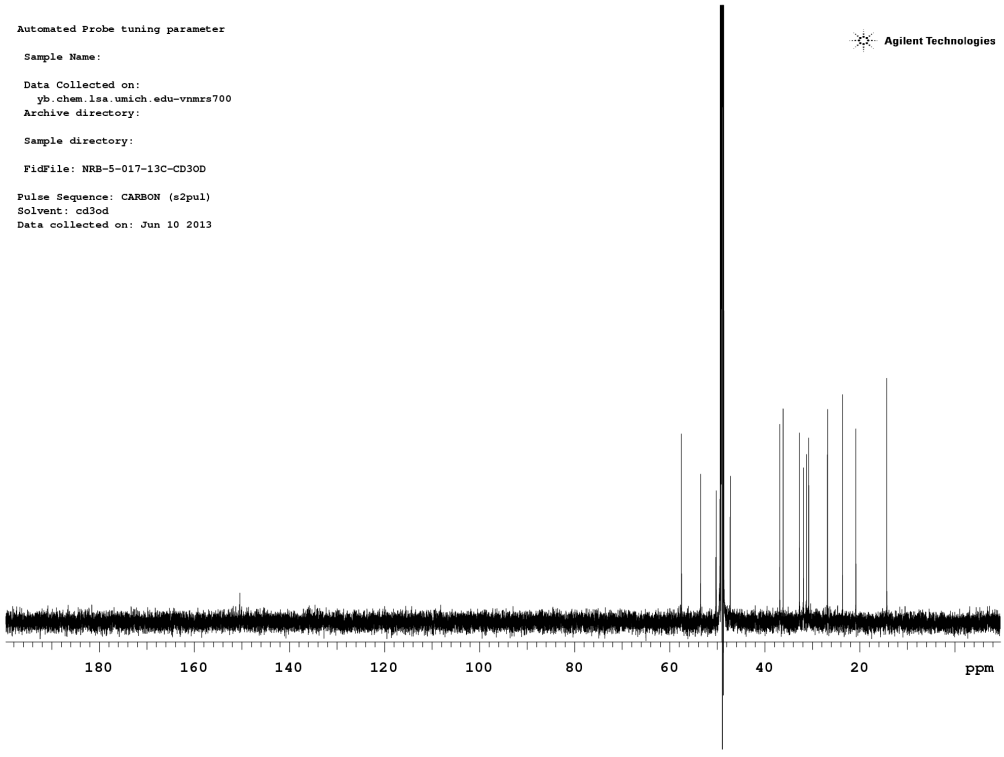

Automated Probe tuning parameter
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-5-012-1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 27 2013
Agilent Technologies
O
H
H
N
O
N
H
H
C2H5
14
7
6
5
4
3
2
1
ppm
0.80
0.89
2.74
2.89
1.00
3.84
0.97
3.01
8.10
3.15


Automated Probe tuning parameter
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-5-012-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 27 2013
Agilent Technologies
220
200
180
160
140
120
100
80
60
40
20
0
ppm

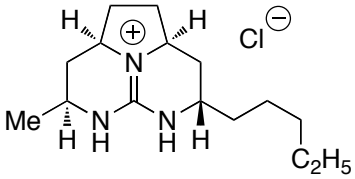

Automated Probe tuning parameter
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-5-017-1H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 10 2013
Agilent Technologies
H
H
Cl
N
Me
N
H
H
N
H
H
C_2H_5
9-*epi*-Batzelladine K (**16**)
$CDCl_3$
9
8
7
6
5
4
3
2
1
ppm
0.92
0.93
2.05
2.00
2.11
6.06
1.02
4.51
12.24
3.12

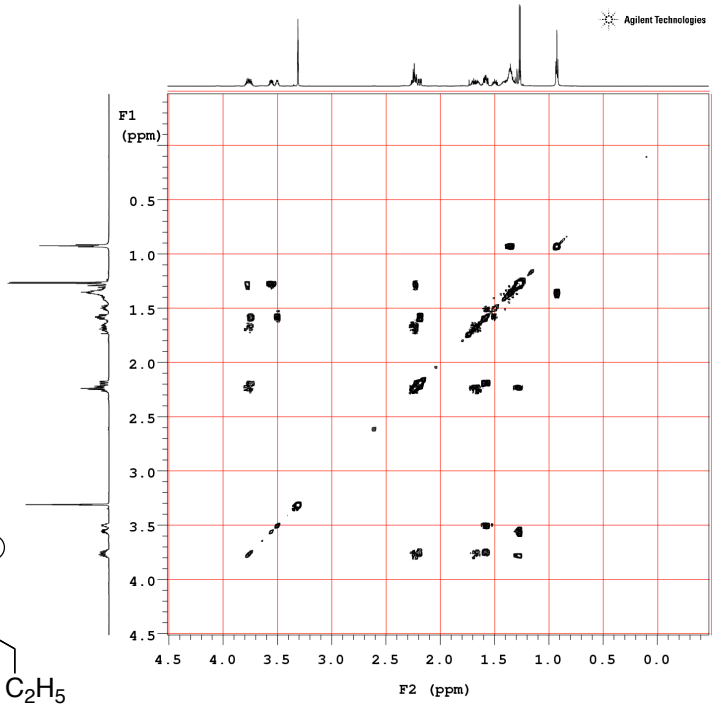

Automated Probe tuning parameter
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-5-017-13C
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 10 2013
Agilent Technologies
180
160
140
120
100
80
60
40
20
ppm


Automated Probe tuning parameter
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-5-017-1H-CD3OD
Pulse Sequence: PROTON (s2pul)
Solvent: cd3od
Data collected on: Jun 10 2013
Agilent Technologies
Me
Cl
C_2H_5
9-*epi*-Batzelladine K (**16**)
CD_3OD
5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.86 0.93 1.03 1.00 2.96 1.17 4.50 11.04 3.00


Automated Probe tuning parameter
Sample Name:
Data Collected on:
yb.chem.lsa.umich.edu-vnmrs700
Archive directory:
Sample directory:
FidFile: NRB-5-017-13C-CD3OD
Pulse Sequence: CARBON (s2pul)
Solvent: cd3od
Data collected on: Jun 10 2013
Agilent Technologies
180 160 140 120 100 80 60 40 20 ppm

```
Automated Probe tuning parameter

 Sample Name:

 Data Collected on:
   yb.chem.lsa.umich.edu-vnmrs700
 Archive directory:

 Sample directory:

 FidFile: NRB-5-017-cosy

Pulse Sequence: gCOSY
Solvent: cd3od
Data collected on: Jun 10 2013

 Temp. 25.0 C / 298.1 K
Operator: nbabij

 Relax. delay 1.000 sec
 Acq. time 0.150 sec
 Width    3491.6 Hz
 2D Width 3491.6 Hz
 2 repetitions
 128 increments
OBSERVE   H1, 699.7595207 MHz
DATA PROCESSING
 Sq. sine bell 0.075 sec
F1 DATA PROCESSING
 Sq. sine bell 0.037 sec
FT size 2048 x 2048
Total time 5 min 44 sec
```





9-*epi*-Batzelladine K (**16**)
CD_3OD

```
Proton Spectrum

 Sample Name:

 Data Collected on:
   yb.chem.lsa.umich.edu-vnmrs700
 Archive directory:

 Sample directory:

 FidFile: NRB-5-017-noesy

Pulse Sequence: NOESY
Solvent: cd3od
Data collected on: Jun 10 2013

 Temp. 25.0 C / 298.1 K
Operator: nbabij

 Relax. delay 1.000 sec
 Acq. time 0.150 sec
 Width    11160.7 Hz
 2D Width 11160.7 Hz
 4 repetitions
 2 x 200 increments
OBSERVE   H1, 699.7595207 MHz
DATA PROCESSING
 Gauss apodization 0.069 sec
F1 DATA PROCESSING
 Gauss apodization 0.017 sec
FT size 4096 x 4096
Total time 37 min
```