Enantioselective, Thiourea-Catalyzed Asymmetric Intermolecular Addition of Indoles to Cyclic *N*-Acyliminium ions

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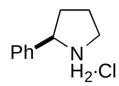
A. General Information

Materials. Commercial reagents were used as received. All solvents were used after being freshly distilled, unless otherwise noted.

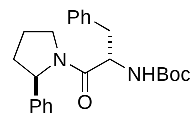
Instrumentation. Proton nuclear magnetic resonance (^{1}H NMR) spectra and carbon nuclear magnetic resonance (^{13}C NMR) were obtained using a Varian Inova (500 MHz) spectrometer. Chiral Super-Fluid Chromatography (SFC) Analysis was performed on a Berger Instrument. Infrared spectra were obtained using a Mattson Galaxy Series FTIR 3000 spectrophotometer.

General Procedures. All reactions were conducted in oven or flame-dried flasks under nitrogen atmosphere. Stainless steel needles and cannulae were used to transfer air and moisture sensitive liguids. Unless otherwise specified, flash chromatography was performed using silica gel 60 (230-400 mesh) from EM Science. The molarities indicated for organolithium reagents were established by titration with BHT and phenanthroline.

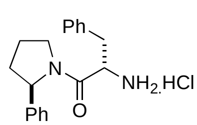
B. Synthesis of Catalyst 5



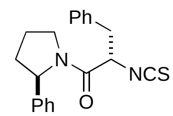
(*R*)-2-phenylpyrrolidine hydrochloride (8). To a flask charged with (*R*)-2-phenyl-*N*-Boc-pyrrolidine (1.51 g, 6.12 mmol), was added CH_2Cl_2 (10 mL) followed by HCl in dioxane (7.6 mL, 30.6 mmol, 4N). The solution was stirred at rt for 3 h at which time it was concentrated *in vacuo* and subjected to high vacuum (<1 Torr) for 2 h to afford the crude salt as a colorless solid which was of sufficient purity for further use in a subsequent step.



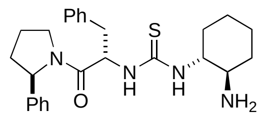
***tert*-butyl (*S*)-1-oxo-3-phenyl-1-((*R*)-2-phenylpyrrolidin-1-yl)propan-2-ylcarbamate (9).** A CH_2Cl_2 (10 mL) solution of HBTU (2.20 g, 5.81 mmol), Boc-(L)-Phenylalanine (1.54 g, 5.81 mmol), and diisopropylamine (3.0 mL, 16.71 mmol) was added to a CH_2Cl_2 solution of (*R*)-2-phenylpyrrolidine hydrochloride **8** (crude product from previous step, assume ~6.2 mmol) in CH_2Cl_2 (10 mL). An additional 10 mLs of CH_2Cl_2 was used to rinse any reagent residue to ensure complete. The resulting heterogeneous mixture was stirred under N_2 at rt for 18 h. The reaction is diluted with Et_2O (200 mL), and washed with 0.5N HCl (2x 100 mL). The acidic aqueous layer is extracted with 100 mL Et_2O, and the combined organics are washed with saturated aqueous $NaHCO_3$ (1x 150 mL) and brine (1x 50 mL). The organics are dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The oily residue was purified on silica gel (8:2 CH_2Cl_2:EtOAc) to yield 2.28 g (95% yield from (*R*)-2-phenyl-*N*-Boc-pyrrolidine) of **9** as a colorless foam: $[\alpha]^{28}_D = +110$ (c = 1.8 g/100 mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$, 1:1 mixture of rotamers) 7.42–7.12 (m, 20H); 6.62–6.59 (m, 2H); 5.43 (d, J = 6.5 Hz, 1H); 5.34 (d, J = 9.5 Hz, 1H); 5.12 (d, J = 8.5 Hz, 1H); 5.08 (d, J = 9.0 Hz, 1H); 4.74 (dd, J = 16.5, 7.5 Hz, 1H); 4.47 (ddd, J = 9.0, 9.0, 4.5 Hz, 1H); 3.87–3.76 (m, 4H); 3.04 (d, J = 7.5 Hz, 2H); 2.76 (dd, J – 16.5, 7.5 Hz, 1H); 2.58 (m, 3 H); 2.07–1.94 (m, 4H); 1.82–1.75 (m, 2H); 1.56–1.47 (m, 1H) 1.50 (s, 9H); 1.36 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$, mixture of rotamers) δ 172.3, 170.5, 155.7, 155.4, 144.1, 142.8, 137.1, 136.9, 129.8, 129.4, 129.2, 128.7, 128.5, 129.3, 127.7, 127.2, 126.8, 126.5, 126.1, 125.6, 79.9, 79.6, 61.9, 60.8, 54.1, 53.3, 47.5, 47.3, 39.8, 39.0, 36.5, 34.1, 28.55, 28.47, 23.3, 22.1, 21.3; IR (film) 2977, 1707, 1636, 1494, 1449, 1365, 1170 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{30}N_2O_3$ (M+H) *m*/*z* 395.2329; found 395.2340.



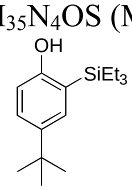
(*S*)-2-amino-3-phenyl-1-((*R*)-2-phenylpyrrolidin-1-yl)propan-1-one hydrochloride (10). To a flask charged with **9** (2.28 g, 5.78 mmol), was added CH_2Cl_2 (10 mL) followed by HCl in dioxane (7.5 mL, 30.0 mmol, 4N). The solution was stirred at rt for 3 h at which time it was concentrated *in vacuo* and subjected to high vacuum (<1 Torr) for 2 h to afford the crude salt **10** as a colorless solid which was of sufficient purity for further use in a subsequent step.



(*S*)-2-isothiocyanato-3-phenyl-1-((*R*)-2-phenylpyrrolidin-1-yl)propan-1-one (11). To a solution of crude **10** (~5.81 mmol) in CH_2Cl_2 (20 mL) at 0 °C was saturated aqueous $NaHCO_3$ (20 mL), and the biphasic mixture was stirred vigorously (~500 rpm) for 10 minutes. The stirring was stopped, and thiophosgene (1.5 mL, 5.8 mmol) was added *via* syringe *to the organic layer*. Immediately, vigorous stirring was restored, and the reaction is removed from the ice bath, and allowed to stir for 30 min at room temperature. The layers were separated and the aqueous layer extracted with CH_2Cl_2 (2 x 50 mL). The combined organics were dried (Na_2SO_4), filtered, concentrated *in vacuo*, and subjected to high vacuum (≤1 Torr) for 10 min, to afford isothiocyanate **11** as an orange oil. The isothiocyanate is sufficiently pure to use in the next step.



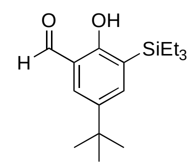
1-((1*R*,2*R*)-2-aminocyclohexyl)-3-((*S*)-1-oxo-3-phenyl-1-((*R*)-2-phenylpyrrolidin-1-yl)propan-2-yl)thiourea (12). In a flame-dried 100 mL round-bottomed flask, (*R,R*)-1,2-cyclohexanediamine (1.00 g, 8.71 mmol) was dissolved in anhydrous CH_2Cl_2 (20 mL). A CH_2Cl_2 solution of isothiocyanate **11** (5 mL, 1.2 M) was added dropwise to this solution *via* syringe over 5 minutes. Additional (3 x 2 mL) portions of CH_2Cl_2 were used to effect quantitative transfer of **11**. The solution is maintained at room temperature for 3 h, and poured into water (10 mL). The layers are separated and the organic layer dried (Na_2SO_4), concentrated *in vacuo*, and the resulting residue purified by silica gel chromatography (10:1 CH_2Cl_2:MeOH–10:1:0.1 CH_2Cl_2:MeOH:NH_4OH) to yield 1.83 g (70 % from **8**) of **12** as a tan foam. ^{1}H NMR (500 MHz, $CDCl_3$, 1:1.6 mix of rotamers) 7.42–7.30 (m, 18H); 7.26–7.20 (m, 4.8H); 7.13–7.08 (m, 2.6H); 6.61 (br s, 1.6H); 6.60 (br s, 1H); 6.01 (d, *J* = 6.5 Hz, 1H); 5.49 (dd, *J* = 9.0, 5.5 Hz, 1.6H); 5.19 (d, *J* = 7.0 Hz, 1.6); 5.19–5.16 (m, 1H); 4.13 (br s, 1.6H); 3.83–3.78 (m, 2.6H); 3.28 (d, *J* = 5.5 Hz, 1H); 3.25 (d, *J* = 5.5 Hz, 1H); 3.16 (dd, *J* = 12.5, 10.0 Hz, 1.6H); 2.72 (dd, *J* = 14.5, 10.0 Hz, 1H); 2.68–2.60 (m, 1.6H); 2.59–2.42 (m, 2.6H); 2.20–19.0 (m, 18H); 1.89–1.59 (18H); 1.58–1.46 (m, 1.6H); 1.32–1.20 (m, 3.2 H); 1.20–1.02 (m, 4H); ^{13}C NMR (125 MHz; $CDCl_3$, mixture of rotamers) δ 182.8, 182.5, 143.8, 142.2, 136.6, 136.0, 129.5, 129.1, 128.9, 128.5, 128.3, 128.0, 127.4, 127.2, 126.8, 126.3, 126.0, 125.4, 61.9, 61.0, 57.5 (br), 47.6, 46.9, 39.3 (br), 38.0 (br), 36.1, 34.8 (br), 34.2 (br), 33.9, 32.0, 24.8, 24.69, 24.67 (br), 22.1 (br), 21.8; IR (film) 3308, 3062, 2929, 2855, 1626, 1543, 1449 cm $^{-1}$; HRMS (ESI) calcd for $C_{26}H_{35}N_4OS$ (M+H) *m/z* 451.2526; found 451.2548.



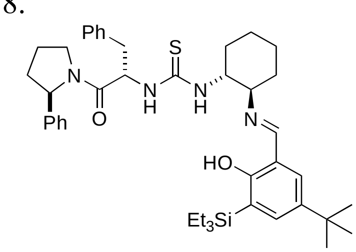
4-*tert*-butyl-2-(triethylsilyl)phenol (13). To a flask charged with 2-bromo-4-*tert*-butylphenol (4.36 g, 19.03 mmol) was added imidazole (1.94 g, 28.54 mmol); 4-dimethylaminopyridine (0.23 g, 1.90 mmol) followed by chlorotriethylsilane (3.8 mL, 22.80 mmol). The heterogeneous mixture was stirred vigorously for 3 h at rt, then diluted with Et_2O (50 mL) and the mixture partitioned between Et_2O (100 mL) and sat. aq. NH_4Cl (100 mL). The layers were separated and the aqueous layer was extracted with Et_2O (2x100 mL). The combined organic fractions were washed with brine (100 mL), dried ($MgSO_4$)

and concentrated *in vacuo* to provide a colorless oil which was of sufficient purity for the following step.

To a flame-dried flask under N_2 charged with the silyl-protected phenol (generated in the previous step ~19 mmol), was added THF (200 mL) and resulting solution was cooled to –78 °C. A hexanes solution of *n*-BuLi (9.8 mL, 24.4 mmol, 2.5 M) was added via syringe over 5 minutes. The solution was maintained at –78 °C for 1 h and then quenched by pouring into saturated aqueous NH_4Cl. The resulting mixture was partitioned between Et_2O (100 mL) and sat. aq. NH_4Cl (100 mL). The layers were separated and the aqueous layer was extracted with Et_2O (2x100 mL). The combined organic fractions were washed with brine (100 mL), dried (Na_2SO_4) and concentrated *in vacuo*. The resulting residue was purified *via* silica gel chromatography (10:1 hexanes:EtOAc) to yield 5.02 g (99% yield) of **13** as a colorless oil: ^{1}H NMR (500 MHz, $CDCl_3$) 7.36 (d, *J* = 3.0 Hz, 1H); 7.23 (dd, *J* = 8.0, 2.0 Hz, 1H); 6.61 (d, *J* = 9.0 Hz, 1H); 1.30 (s, 9H); 0.98 (t, *J* = 8.0 Hz, 9H); 0.86 (q, *J* = 8.0 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.2, 142.5, 133.0, 127.3, 121.5, 113.9, 34.0, 31.6, 7.57, 3.64; IR (film) 3539, 2875, 1593, 1488, 1386, 1264, 1071, 733 cm^{-1}; ; HRMS (CI) calcd for $C_{16}H_{32}NOSi$ (M+NH_4) *m/z* 282.2253; found 282.2239.



5-*tert*-butyl-2-hydroxy-3-(triethylsilyl)benzaldehyde (14).[1] A 3-neck, 500 mL flask with an attached condenser was charged with **13** (5.02 g, 19.0 mmol); paraformaldehyde (1.44 g, 48.0 mmol) and $MgCl_2$ (3.65 g, 38.4 mmol). THF (200 mL) was added, followed by Et_3N (5.35 mL, 38.4 mmol). The resulting yellow slurry was heated at reflux for 20 h. The mixture was then cooled to rt, and 10% HCl (aqueous, 200 mL) was added, followed by Et_2O (200 mL). The layers were separated and the remaining organic layer was washed with an additional portion of 10% aqueous HCl (100 mL), then washed with sat. aqueous $NaHCO_3$ (3x100 mL), brine (100 mL) , dried (Na_2SO_4) and concentrated *in vacuo*. The residue was purified on silica gel (10:1 hexanes:EtOAc) to yield 4.57 g (86% yield) of **14** as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) 11.17 (s, 1H); 9.87 (s, 1H); 7.65 (d, *J* = 2.5 Hz, 1H); 7.48 (d, *J* = 2.5 Hz, 1H); 1.33 (s, 9H); 0.97 (t, *J* = 8.0 Hz, 9H); 0.88 (q, *J* = 8.0 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.3, 164.8, 142.1, 141.6, 131.1, 125.6, 119.1, 34.3, 31.5, 7.72, 3.53; IR (film) 2955, 2875, 2833, 2732, 1658, 1606, 1583, 1401, 1262, 1006, 869 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{28}O_2Si$ (M+H) *m/z* 293.1931; found 293.1938.

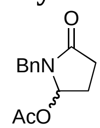


1-((1*R*,2*R*)-2-((*E*)-5-*tert*-butyl-2-hydroxy-3-(triethylsilyl)benzylideneamino)cyclohexyl)-3-((*S*)-1-oxo-3-phenyl-1-((*R*)-2-phenylpyrrolidin-1-yl)propan-2-yl)thiourea (5). A flame-dried 100 mL flask was charged with **12** (0.98 g, 2.18 mmol), aldehyde **14** (0.64 g, 2.18 mmol), and freshly flame-dried Na_2SO_4 (2.0 g, 14.2 mmol). The flask was sealed with a rubber septum, placed under N_2, and CH_2Cl_2 (10 mL) was added. The mixture was stirred at rt for 18 h, concentrated *in vacuo* and purified with a

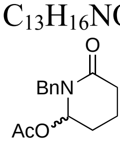
[1] Procedure adapted from T. V. Hansen, L. Skattebøl, *Tetrahedron Lett.* **2005**, *46*, 3829–3830.

PLUG of acitivity III basic alumina[2] (3:1 hexanes:EtOAc) to yield 1.47 g (93% yield) of **5** as a yellow crystalline solid.[3] ^{1}H NMR (500 MHz, $CDCl_3$, mixture of rotamers only diagnostic peaks reported) 12.90 (br s, 1H); 12.72 (br s, 1H); 8.27 (br s, 1H); 8.21 (br s, 0.6 H); 7.44–7.40 (m, 1.6H); 7.35–7.09 (m, 18H); 7.03–6.92 (m, 1.6H); 6.52 (d, *J* = 6.0 Hz, 1H); 6.00 (br s, 0.6H); 5.57 (br s, 0.6H); 5.34 (br s, 1H); 5.03 (d, *J* = 6.0 Hz, 1H); 3.80–3.60 (m, 3H); 2.95–2.80 (m, 3H); 2.58–2.50 (m, 1H); 2.40–2.30 (m, 1H); 2.30–2.24 (m, 0.6H); 1.85–1.50 (m, 12.6H); 1.40–1.10 (m, 14H), 1.30 (s, H); 1.00–0.72 (m, 30H); ^{13}C NMR (125 MHz, $CDCl_3$, mixture of rotamers, only prominent peaks reported) δ 197.1, 165.6, 164.6, 164.0, 163.7, 143.4, 141.3, 140.2, 140.1, 136.4, 136.1, 130.9, 129.6, 129.5, 129.3, 128.9, 128.3, 128.2, 127.9, 127.3, 126.8, 126.6, 126.2, 125.9, 125.5, 123.7, 118.9, 117.0, 61.8, 60.9, 47.4, 46.9, 39.2, 35.9, 33.9, 31.6, 31.4, 31.3, 22.4, 21.77.64, 7.59, 7.46, 3.4, 3.3; IR (film) 3269, 2953, 2873, 1629, 1449, 1263, 733 cm^{-1}; LRMS (ESI) calcd for $C_{43}H_{60}N_4O_2SSi$ (M+H) *m/z* 725.4; found 725.9.

C. Preparation of *N*-acyliminium ion precursors



1-benzyl-5-oxopyrrolidin-2-yl acetate (6a). To a flame-dried 50 mL round bottom was added 1-benzyl-5-hydroxypyrrolidin-2-one (**1**)[4] (1.03 g, 5.37 mmol), 4-(dimethylamino)pyridine (0.06 g, 0.54 mmol) and a stir bar. The flask was sealed with a rubber septum and placed under N_2 atmosphere. CH_2Cl_2 (20 mL) was added followed by Et_3N (0.97 mL, 6.98 mmol) and acetic anhydride (0.56 mL, 5.90 mmol). The solution was maintained at rt for 1 h, at which time it was concentrated *in vacuo* and purified through a short column of activity III alumina to yield 1.24 g (99% yield) of **6a** as a colorless oil. This oil was unstable neat at rt, but could be stored indefinitely as a solution in TBME at –30 °C (solution was stable at rt for several hours, which was long enough for set up of addition reactions). ^{1}H NMR (500 MHz, $CDCl_3$) 7.34–7.29 (m, 2H); 7.23–7.28 (m, 3H); 6.13 (d, *J* = 6.0 Hz, 1H); 4.72 (d, *J* = 15.0 Hz, 1H); 4.20 (d, *J* = 15.5 Hz, 1H); 2.64 (ddd, *J* = 18.0, 10.0, 10.0 Hz, 1H); 2.41 (ddd, *J* = 17.0, 10.0, 2.0 Hz, 1H); 2.32–2.22 (m, 1H); 2.02–1.98 (m, 1H); 1.91 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.5, 170.5, 136.3, 128.6, 128.3, 127.7, 84.2, 44.6, 28.3, 26.0, 20.9; IR (film) 2933, 1742, 1716, 1444, 1419, 1237, 1173 cm^{-1}; HRMS (ESI) calcd for $C_{13}H_{16}NO_3$ (M+H) *m/z* 234.1125; found 234.1121.



1-benzyl-6-oxopiperidin-2-yl acetate (6b). The product was prepared from 1-benzyl-6-hydroxypiperidin-2-one on 5.74 mmol scale in a similar manner to that described for the preparation of **6a**. This procedure yielded 1.41 g (99% yield) of **6b** as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) 7.32–7.28 (m, 2H); 7.27–7.22 (m, 3H); 6.10–6.06 (m, 1H); 4.97 (d, *J* = 15.0 Hz, 1H); 4.19 (d, *J* = 15.0 Hz, 1H); 2.68–2.59 (m, 1H); 2.49–2.40 (m, 1H); 2.11–2.00 (m, 1H); 2.00–1.94 (m, 1H); 1.92 (s, 3H); 1.86–1.76 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.5, 170.2, 137.1, 128.5, 128.2, 127.4, 80.6,

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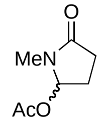
[2] Activity III basic alumina is prepared by the addition of 6% by weight water to Brockman Alumina Acitivty I.

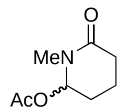
[3] It is possible to observe small amounts of aldehyde **14** in the final ^{1}H NMR of the catalyst **5**. These result from both minor hydrolysis of the final catalyst in the ^{1}H NMR solvent, and/or from measurement error in conducting the final experiment in the catalyst synthesis. This contaminant does not have any deleterious effect on the catalytic reaction.

[4] J. C. Hubert, J. B. P. A. Wunberg, W. N. Speckamp, *Tetrahedron* **1975**, *31*, 1437–1441.

48.1, 32.2, 28.3, 20.9, 16.2; IR (film) 2957, 1736, 1664, 1452, 1234, 1180, 962 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{17}NO_3$ (M+H) *m/z* 248.1281; found 248.1282.

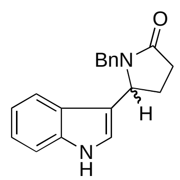


1-methyl-5-oxopyrrolidin-2-yl acetate (6c). The product was prepared from 5-hydroxy-1-methylpyrrolidin-2-one (0.60 g, 5.2 mmol) as described for **6a**. The product **6c** was extremely unstable but could be quickly purified on activity III alumina (8:2:0.5 CH_2Cl_2:EtOAc:Et_3N). The product **6c** could not be detected by thin layer silica gel chromatography, as it decomposed on the plate. It was imperative to use **6c** immediately upon production as prolonged storage resulted in decomposition. The product was consistent with previously reported data.[5]



1-methyl-6-oxopiperidin-2-yl acetate (6d). The product was prepared from 6-hydroxy-1-methylpiperidin-2-one on 2.76 mmol scale in a similar manner to that described for the preparation of **6a**. This procedure yielded 0.11 g (23% yield) of **6d** as a colorless oil. The product **6d** decomposed during thin layer chromatography. It was imperative to use **6d** immediately upon production as prolonged storage resulted in decomposition. ^{1}H NMR (500 MHz, $CDCl_3$) 6.07–6.05 (m, 1H); 2.92 (s, 3H); 2.58–2.49 (m, 1H); 2.39–2.31 (m, 1H); 2.08 (s, 3H); 2.02–1.91 (m, 3H); 1.80–1.74 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.5, 170.7, 83.2, 33.4, 32.3, 28.4, 21.3, 16.7; IR (film) 2956, 1735, 1664, 1399, 1374, 1228, 1050, 982 cm^{-1}.

D. General Procedure for the synthesis of racemic 5-(1*H*-indol-3-yl)pyrrolidin-2-ones



A 1-dram glass vial was charged with indole (9 mg, 0.08 mmol), 1,3-bis(3,5-bis(trifluoromethyl)phenyl)thiourea[6] (3 mg, 8 μmol), and hydroxy-lactam **1** (15 mg, 0.08 mmol). The vial is sealed with a teflon cap and CH_2Cl_2 (0.4 mL) is added and the solution cooled to –78 °C. A CH_2Cl_2 solution of TMSCl (0.10 mL, 0.15 mmol, 1.6 M) is added and the solution is allowed to warm to 0 °C and maintained at this temperature for 18 h. Saturated aqueous $NaHCO_3$ (0.2 mL) is added and the layers separated. The organic layer is loaded directly onto a silica gel column (8:2 CH_2Cl_2:EtOAc) and purified to give 18 mg (80 % yield) of racemic product **2a**. See enantioenriched procedure for spectral data.

E. General Procedures for the preparation of enantioenriched 5-(indol-3-yl)pyrrolidin-2-ones (2).

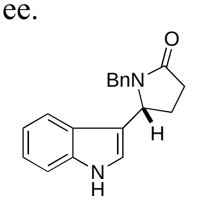
Method A. A 10 mL, flame-dried round bottom flask was charged with indole (0.040 g, 0.343 mmol), catalyst **5** (0.012 g, 0.017 mmol) and sealed with a rubber septum. The flask was flushed with N_2 and anhydrous TBME (1.6 mL) was added. The resulting yellow solution was cooled to –78 °C, and a

[5] Y. Nagao, W.-M. Dai, M. Ochiai, M. Shiro, *Tetrahedron* **1990**, *46*, 6361–6380.
[6] Achiral thiourea catalyst: M. Kotke, P. R. Schreiner, *Tetrahedron* **2006**, *62*, 434–439.

TBME solution of acetoxy-lactam **6** (0.65 mL, 0.34 mmol, 0.53 M) was added. Next, TBME solutions of TMSCl (0.43 mL, 0.69 mmol, 1.6 M) and H_2O[7] (0.20 mL, 0.03 mmol, 0.14 M) were added sequentially and the solution was warmed to –30 °C and stirred for 24 h. Next, the heterogeneous solution is quenched by the addition of an EtOH solution of NaOEt (0.2 mL, 21 wt%), followed by immediate addition of water (1 mL). The mixture is allowed to warm to rt and is diluted with EtOAc until all solid dissolves (amount depends on particular product ~5–10 mL). The layers are separated and the organic layer is dried (Na_2SO_4) and concentrated *in vacuo*. See individual products for purification procedures.

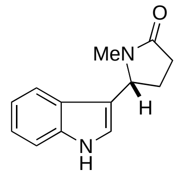
Method B. A 10 mL, flame-dried round bottom flask was charged with indole (0.040 g, 0.343 mmol), catalyst **5** (0.012 g, 0.017 mmol) and sealed with a rubber septum. The flask was flushed with N_2 and anhydrous TBME (1.6 mL) was added. The resulting yellow solution was cooled to –78 °C, and a TBME solution of acetoxy-lactam **6** (0.65 mL, 0.34 mmol, 0.53 M) was added. Next, a TBME solution of BCl_3 (created by adding 0.2 mL of 1.0 M BCl_3 in hexanes into 5 mL of TBME) (0.86 mL, 0.034 mmol, 0.04 M) was added and the solution was warmed to –30 °C and stirred for 48–96 h. Next, the heterogeneous solution is quenched by the addition of an EtOH solution of NaOEt (0.2 mL, 21 wt%), followed by immediate addition of water (1 mL). The mixture is allowed to warm to rt and is diluted with EtOAc until all solid dissolves (amount depends on particular product ~5–10 mL). The layers are separated and the organic layer is dried (Na_2SO_4) and concentrated *in vacuo*.

A note on purification by the trituration procedure: We have observed that trituration from Et_2O leads to further enantiomeric enrichment. The product remaining in the mother-liquor is consequently enriched in minor diastereomer. We hypothesize that the catalyst interacts selectively with the minor enantiomer, further stabilizing it in solution. Independent experiments were conducted where alumina-purified product **2a** (93%ee) was subjected to 10 mol% catalyst, triturated from Et_2O (3x5 mL) and the remaining product was analyzed to be 98% ee.

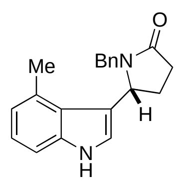


(*R*)-1-benzyl-5-(1*H*-indol-3-yl)pyrrolidin-2-one (2a). The product was prepared by Method A and was scaled to 3.15 mmol. The crude product (93%ee) was purified by trituration from Et_2O (3x10 mL) to yield **2a** (0.854 g, 90% yield) as a colorless crystalline solid. This material was determined to be 99% ee by chiral SFC analysis (ChiralPak AS-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 3.79 min, t_r(major) = 5.80 min. $[\alpha]^{29}_D$ = –49° (*c* = 1.3 g/100mL, MeOH); ^{1}H NMR (500 MHz, d_6-DMSO) 11.12 (s, 1H); 7.42 (d, *J* = 8.5 Hz, 1H); 7.37 (d, *J* = 7.5 Hz, 1H); 7.32–7.26 (m, 4H), 7.14 (dd, *J* = 7.5, 1.0 Hz, 1H); 7.08 (d, *J* = 7.0 Hz, 2H); 7.00 (dd, *J* = 8.0, 1.0 Hz, 1H); 4.83–4.78 (m, 2H); 3.51 (d, *J* = 15 Hz, 1H); 2.60–2.48 (m, 2H, DMSO overlaps); 2.46–2.38 (m, 1H); 2.17–2.08 (m, 1H); ^{13}C NMR (125 MHz, d_6=DMSO) δ 173.9, 137.1, 136.9, 128.3, 127.5, 127.0, 125.1, 124.2, 121.3, 118.9, 118.2, 113.4, 111.9, 54.2, 43.2, 30.1, 26.5; IR (film) 3225, 2925, 1660, 1444, 1264 cm $^{-1}$; HRMS (ESI) calcd for $C_{19}H_{19}N_2O$ (M+H) *m/z* 291.1497; found 291.1498.

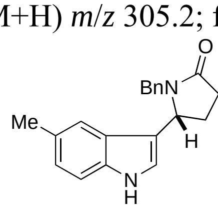
[7] The solubility of water in TBME is 1.5 g in 100 mL. *The Merck Index*, 12th Edition (Eds: S. Budavari et al), MERCK & Co, Whitehouse Station, NJ, **1996**, p 1032.



(*R*)-5-(1*H*-indol-3-yl)-1-methylpyrrolidin-2-one (2b). The product was prepared by Method A on 0.21 mmol scale. Note that acetoxy-lactam **6c** is extremely unstable and must be used immediately upon isolation.[8] The crude product (85%ee) was purified by trituration from 1:1 Et_2O:hexanes (1x10 mL) to yield **2b** (0.042 g, 93% yield) as a colorless crystalline solid. This material was determined to be 86% ee by chiral SFC analysis (ChiralPak AD-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 6.71 min, t_r(major) = 7.23 min. $[\alpha]^{29}_D$ = –21° (*c* = 0.8 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.28 (br s, 1H); 7.54 (dd, *J* = 8.0, 1.0 Hz, 1H); 7.41 (ddd, *J* = 8.5, 1.0, 1.0 Hz, 1H); 7.26 (s, 1H); 7.23 (dd, *J* = 7.0, 7.0, 1.0, 1H); 7.15–7.11 (m, 2H); 4.87 (t, *J* = 8.0 Hz, 1H); 2.71 (s, 3H); 2.68–2.60 (m, 1H); 2.56–2.42 (m, 2H); 2.24–2.16 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.2, 136.9, 125.2, 122.6, 122.5, 119.9, 118.8, 115.1, 111.7, 57.9, 30.7, 28.0, 26.9; IR (film) 3259, 2927, 1659, 1457, 1267, 1109, 744 cm^{-1}; HRMS (ESI) calcd for $C_{13}H_{15}N_2O$ (M+H) *m/z* 215.1179; found 215.1185.



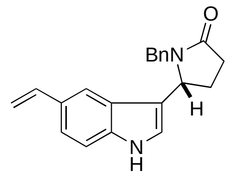
(*R*)-1-benzyl-5-(4-methyl-1*H*-indol-3-yl)pyrrolidin-2-one (2c). The product was prepared by Method A on 0.34 mmol scale. The crude product was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2c** (0.090 g, 86% yield) as a colorless crystalline solid. This material was determined to be 95% ee by chiral SFC analysis (Pirkle S,S-Whelk, 4.0 mL/min, 220 nm, 15% MeOH, t_r(minor) = 6.68 min, t_r(major) = 8.36 min. $[\alpha]^{28}_D$ = –6° (*c* = 0.9 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.75 (br s, 1H); 7.37–7.29 (m, 4H); 7.19 (d, *J* = 5.0 Hz, 2H); 7.15 (dd, *J* = 7.5, 7.5 Hz, 1H); 7.00 (d, *J* = 2.5 Hz, 1H); 6.88 (d, *J* = 6.5 Hz, 1H); 5.25 (d, *J* = 15.0 Hz, 1H); 5.21 (d, *J* = 6.0 Hz, 1H); 3.92 (d, *J* = 14.5 Hz, 1H); 2.70–2.60 (m, 1H); 2.52–2.43 (m, 2H); 2.47 (s, 3H); 2.08–2.00 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.7, 137.4, 137.0, 130.3, 128.9, 128.6, 127.8, 125.0, 122.7, 121.7, 121.0, 117.2, 109.7, 55.2, 44.8, 29.6, 29.3, 20.9; IR (film) 3281, 2943, 1666, 1441, 1417, 750 cm^{-1}; LRMS (ESI) calcd for $C_{20}H_{21}N_2O$ (M+H) *m/z* 305.2; found 305.4.



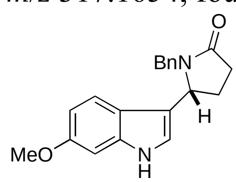
(*R*)-1-benzyl-5-(5-methyl-1*H*-indol-3-yl)pyrrolidin-2-one (2d). The product was prepared by Method A on 0.34 mmol scale. The crude product was purified by trituration from Et_2O (3x2 mL) to yield **2d** (0.082 g, 79% yield) as a colorless crystalline solid. This material was determined to be 91% ee by chiral SFC analysis (ChiralPak AS-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 3.48 min, t_r(major) = 5.01 min. $[\alpha]^{29}_D$ = –74° (*c* = 0.4 g/100mL, MeOH); ^{1}H NMR (500 MHz, d_6-DMSO) 10.98

[8] Use of a mixture of hydroxylactam and acetoxy-lactam (resulting from hydrolysis of **6c**) gave diminished yield and ee (80% yield, 78% ee).

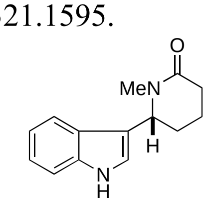
(s, 1H); 7.30–7.26 (m, 3H); 7.24 (d, *J* = 7.0 Hz, 1H); 7.19 (d, *J* = 2.5 Hz, 1H); 7.09 (br s, 1H); 7.05 (d, *J* = 6.5 Hz, 2H); 6.93 (dd, *J* = 8.5, 1.5 Hz, 1H); 4.78 (d, *J* = 15 Hz, 1H); 4.76 (t, 7.0 Hz, 1H); 3.48 (d, *J* = 15Hz, 1H); 2.60–2.42 (m, 2H); 2.40–2.31 (m, 1H); 2.34 (s, 3H); 2.13–2.07 (m, 1H); ^{13}C NMR (125 MHz, d_6-DMSO) δ 173.9, 137.1, 135.2, 128.3, 127.5, 127.3, 126.9, 125.4, 124.3, 123.0, 117.8, 112.8, 111.6, 54.2, 43.2, 30.2, 26.4, 21.3; IR (film) 3215, 2921, 1661, 1435 cm^{-1}; LRMS (ESI) calcd for $C_{20}H_{21}N_2O$ (M+H) *m/z* 305.2; found 305.4.



(*R*)-1-benzyl-5-(5-vinyl-1*H*-indol-3-yl)pyrrolidin-2-one (2e). The product was prepared by Method A on 0.34 mmol scale. The crude product was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2e** (0.090 g, 86% yield) as a colorless crystalline solid. This material was determined to be 90% ee by chiral SFC analysis (ChiralPak AS-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 3.72 min, t_r(major) = 4.99 min. $[\alpha]^{28}_D$ = –71° (*c* = 1.1 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.43 (br s, 1H); 7.41–7.35 (m, 3H); 7.28–7.23 (m, 3H); 7.10 (dd, *J* = 8.0, 2.5 Hz, 2H); 7.00 (d, *J* = 2.0 Hz, 1H); 6.79 (dd, *J* = 17.5, 10.5 Hz, 1H); 5.67 (d, *J* = 17.5 Hz, 1H); 5.17 (d, *J* = 11.0 Hz, 1H); 5.09 (d, *J* = 15.0 Hz, 1H); 4.78 (dd, *J* = 8.0, 6.5 Hz, 1H); 3.60 (d, *J* = 14.5 Hz, 1H); 2.75–2.69 (m, 1H); 2.58 (ddd, *J* = 17.5, 9.5, 7.5 Hz, 1H); 2.43–2.37 (m, 1H); 2.26–2.18 (m, 1H); ^{13}C NMR (125 MHz) δ 175.0, 137.5, 136.8, 136.7, 130.0, 128.43, 128.36, 127.3, 125.6, 123.3, 120.6, 117.4, 115.3, 111.7, 111.6, 54.6, 44.2, 30.8, 26.8; IR (film) 3276, 3031, 2925, 1669, 1441, 1411, 1264, 1161 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{21}N_2O$ (M+H) *m/z* 317.1654; found 317.1653.

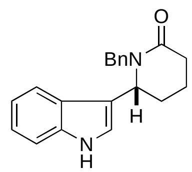


(*R*)-1-benzyl-5-(6-methoxy-1*H*-indol-3-yl)pyrrolidin-2-one (2f). The product was prepared by Method A on 0.34 mmol scale. The crude product (80%ee) was purified by trituration from 9:1 Et_2O:CH_2Cl_2 (3x2 mL) to yield **2f** (0.087 g, 80% yield) as a colorless crystalline solid. This material was determined to be 98% ee by chiral SFC analysis (ChiralPak AS-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 4.19 min, t_r(major) = 6.16 min. $[\alpha]^{29}_D$ = –69° (*c* = 0.5 g/100mL, MeOH); ^{1}H NMR (500 MHz, d_6-DMSO) 10.91 (br s, 1H); 7.34–7.30 (m, 2H); 7.27 (d, *J* = 7.5 Hz, 1H); 7.24 (d, *J* = 8.5 Hz, 1H); 7.14 (d, *J* = 2.5 Hz, 1H); 7.09 (d, *J* = 7.0 Hz, 2H); 6.93 (d, *J* = 2.0 Hz, 1H); 6.68 (dd, *J* = 9.0, 2.5, Hz, 1H); 4.80 (d, *J* = 15.5 Hz, 1H); 4.77 (t, *J* = 7.5 Hz, 1H);[9] 3.80 (s, 3H); 3.52 (d, *J* = 15.0 Hz, 1H); 2.60–2.38 (m, 2H); 2.44–2.36 (m, 1H); 2.15–2.06 (m, 1H); ^{13}C NMR (125 MHz, d_6-DMSO) δ 173.9, 155.7, 137.7, 137.1, 128.4, 127.5, 127.0, 122.9, 119.3, 118.8, 113.4, 109.2, 94.9, 55.1, 54.3, 43.2, 30.1, 26.5; IR (film) 3280, 2950, 1682, 1454, 1294, 1159, 702 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{21}N_2O_2$ (M+H) *m/z* 321.1603; found 321.1595.

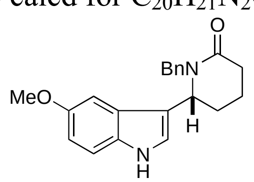


[9] Part of peak overlapping with another resonance.

(*R*)-6-(1*H*-indol-3-yl)-1-methylpiperidin-2-one (2g). The product was prepared by Method A on 0.29 mmol scale. The crude product was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2g** (0.040 g, 60% yield) as a colorless crystalline solid. This material was determined to be 92% ee by chiral SFC analysis (ChiralPak AD-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 3.29 min, t_r(major) = 3.89 min. $[\alpha]^{28}_D$ = +4° (*c* = 0.7 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.34 (br s, 1H); 7.54 (d, *J* = 7.5 Hz, 1H); 7.41 (d, *J* = 8.0 Hz, 1H); 7.26 (s, 1H); 7.23 (ddd, *J* = 8.5, 8.5, 1.5 Hz, 1H); 7.14 (dd, *J* = 7.5, 7.5 Hz, 1H); 7.00 (d, *J* = 3.0 Hz, 1H); 4.89 (t, *J* = 5.0 Hz, 1H); 2.93 (s, 3H); 2.59–2.45 (m, 2H); 2.20–2.09 (m, 2H); 1.87–1.76 (m, 1H); 1.74–1.64 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.8, 136.8, 125.4, 122.4, 122.2, 119.8, 118.5, 116.3, 111.6, 57.0, 34.0, 32.3, 30.2, 18.1; IR (film) 3257, 2947, 1614, 1397, 1246, 742 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{17}N_2O$ (M+H) *m/z* 229.1335; found 229.1335.



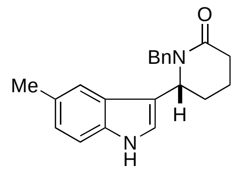
(*R*)-1-benzyl-6-(1*H*-indol-3-yl)piperidin-2-one (2h). The product was prepared by Method A on 0.34 mmol scale. The crude product was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2h** (0.073 g, 70% yield) as a colorless crystalline solid. This material was determined to be 93% ee by chiral SFC analysis (ChiralPak AS-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 3.75 min, t_r(major) = 5.36 min. $[\alpha]^{29}_D$ = –54° (*c* = 0.9 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.54 (br s, 1H); 7.48 (d, *J* = 7.5 Hz, 1H); 7.42 (d, *J* = 8.0 Hz, 1H); 7.32–7.21 (m, 4H); 7.18 (d, *J* = 8.5 Hz, 2H); 7.13 (dd, *J* = 8.0, 1.0 Hz, 1H); 7.00 (d, *J* = 2.0 Hz, 1H); 5.63 (d, *J* = 15.0 Hz, 1H); 4.87 (t, *J* = 5.5 Hz 1H); 3.65 (d, *J* = 15.0 Hz, 1H); 2.70–2.55 (m, 2H); 2.15–2.10 (m, 1H); 2.09–1.98 (m, 1H); 1.90–1.80 (m, 1H); 1.75–1.66 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.0, 138.0, 137.1, 128.7, 128.2, 127.4, 125.8, 122.7, 122.7, 120.0, 118.9, 116.3, 111.8, 53.7, 47.9, 32.6, 30.4, 18.0; IR (film) 3234, 2949, 1620, 1453, 1414, 732 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{21}N_2O$ (M+H) *m/z* 305.1654; found 305.1644.



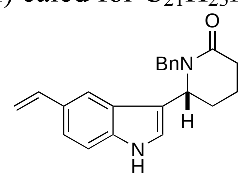
(*R*)-1-benzyl-6-(5-methoxy-1*H*-indol-3-yl)piperidin-2-one (2i). The product was prepared by Method A on 0.34 mmol scale. The crude product (crude ee = 91%)[10] was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2i** (0.099 g, 86% yield) as a colorless crystalline solid. This material was determined to be 90% ee by chiral SFC analysis (ChiralPak AS-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 3.47 min, t_r(major) = 4.85 min. $[\alpha]^{29}_D$ = –56° (*c* = 0.6 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.53 (br s, 1H); 7.32–7.24 (m, 4H); 7.18 (dd, *J* = 7.0, 1.0 Hz, 2H); 6.97 (d, *J* = 2.5 Hz, 1H); 6.88 (dd, *J* = 9.0, 2.5 Hz, 1H); 6.86 (d, *J* = 2.5 Hz, 1H); 5.61 (d, *J* = 15.0 Hz, 1H); 4.83 (t, *J* = 9.5 Hz, 1H); 3.82 (s, 3H); 3.67 (d, *J* = 14.5 Hz, 1H); 2.67–2.58 (m, 2H); 2.15–1.99 (m, 2H); 1.92–1.82 (m, 1H); 1.74–1.66 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.9, 154.3, 138.0, 132.2, 128.7, 128.2, 127.4, 126.2, 123.4, 115.9, 112.8, 112.6, 100.7, 56.1, 53.6, 47.9, 32.6, 30.1, 18.0; IR

[10] This product is readily racemized on silica gel and even undergoes some loss of ee with quick purification on activity III basic alumina.

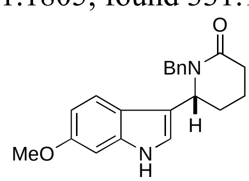
(film) 3271, 2946, 1625, 1485, 1213, 1170, 800 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{23}N_2O_2$ (M+H) *m/z* 335.1760; found 335.1766.



(*R*)-1-benzyl-6-(5-methyl-1*H*-indol-3-yl)piperidin-2-one (2j). The product was prepared by Method A on 0.34 mmol scale. The crude product was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2j** (0.101 g, 93% yield) as a colorless crystalline solid. This material was determined to be 94% ee by chiral SFC analysis (Pirkle S,S-Whelk, 4.0 mL/min, 220 nm, 15% MeOH, t_r(minor) = 7.95 min, t_r(major) = 10.54 min. $[\alpha]^{29}_D$ = –54° (*c* = 1.1 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.36 (br s, 1H); 7.32–7.22 (m, 5H); 7.18 (dd, *J* = 1.5, 8.5 Hz, 2H); 7.06 (dd, *J* = 8.0, 1.0 Hz, 1H); 6.96 (d, *J* = 2.0 Hz, 1H); 5.62 (d, *J* = 14.5 Hz, 1H); 4.84 (t, *J* = 5.0 Hz, 1H); 3.66 (d, *J* = 15.0 Hz, 1H); 2.69–2.56 (m, 2H); 2.44 (s, 3H); 2.18–2.09 (m, 1H); 2.06–1.99 (m, 1H); 1.90–1.81 (m, 1H); 1.66–1.72 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.0, 138.1, 135.4, 129.3, 128.7, 128.2, 127.4, 126.0, 124.3, 122.8, 118.5, 115.8, 111.5, 53.7, 47.9, 32.6, 30.2, 21.8, 18.0; IR (film) 3244, 2946, 2916, 1621, 1451, 1414, 1264 cm^{-1}; LRMS (ESI) calcd for $C_{21}H_{23}N_2O$ (M+H) *m/z* 319.2; found 319.4.

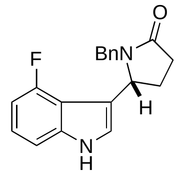


(*R*)-1-benzyl-6-(5-vinyl-1*H*-indol-3-yl)piperidin-2-one (2k). The product was prepared by Method A on 0.34 mmol scale. The crude product was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2k** (0.101 g, 93% yield) as a colorless foam. This material was determined to be 91% ee by chiral SFC analysis (ChiralPak AS-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 3.62 min, t_r(major) = 4.83 min. $[\alpha]^{29}_D$ = –49° (*c* = 0.9 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.49 (br s, 1H); 7.42 (s, 1H); 7.39–7.36 (m, 2H); 7.32–7.22 (m, 3H); 7.17 (dd, *J* = 8.5, 1.5 Hz, 2H); 6.98 (d, *J* = 2.0 Hz, 1H); 6.80 (dd, *J* = 17.0, 11.0 Hz, 1H); 5.68 (dd, *J* = 17.5, 1.0 Hz, 1H); 5.62 (d, *J* = 14.5, 1H); 5.16 (dd, *J* = 11.0, 1.0 Hz, 1H); 4.87 (t, *J* = 4.5 Hz, 1H); 3.66 (d, *J* = 15.0 Hz, 1H); 2.70–2.56 (m, 2H); 2.16–2.10 (m, 1H); 2.09–2.00 (m, 1H); 1.90–1.82 (m, 1H); 1.76–1.66 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.6, 137.7, 137.5, 136.6, 129.9, 128.5, 128.1, 128.0, 127.2, 125.7, 122.9, 120.7, 117.0, 116.5, 111.6, 53.3, 47.7, 32.3, 30.0, 17.7; IR (film) 3249, 2947, 1617, 1475, 1354, 1264 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{23}N_2O$ (M+H) *m/z* 331.1805; found 331.1805.

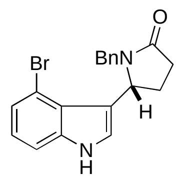


(*R*)-1-benzyl-6-(6-methoxy-1*H*-indol-3-yl)piperidin-2-one (2l). The product was prepared by Method A on 0.34 mmol scale. The crude product (crude ee = 92%)[10] was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2l** (0.087 g, 76% yield) as a colorless foam. This material was determined to be 88% ee by chiral SFC analysis (ChiralPak AS, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 4.66 min, t_r(major) = 6.54 min. $[\alpha]^{29}_D$ = –45° (*c* = 0.5 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.43 (br s, 1H); 7.32 (d, *J* = 9.0 Hz, 1H); 7.32–7.22 (m, 3H);[9] 7.17 (d, *J* = 7.0 Hz, 1H); 6.89 (dd, *J* = 11.5, 2.0 Hz, 1H); 6.79 (dd, *J* = 8.5, 2.0 Hz, 1H); 5.60 (d, *J* = 15.0 Hz, 1H); 4.81 (t, *J* =

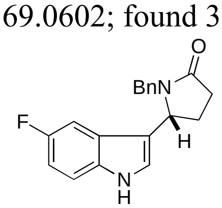
5.0 Hz, 1H); 3.84 (s, 3H); 3.65 (d, *J* = 15.0 Hz, 1H); 2.67–2.54 (m, 2H); 2.12–1.98 (m, 2H); 1.92–1.82 (m, 1H); 1.72–1.68 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.7, 156.7, 137.8, 137.7, 128.5, 128.0, 127.1, 121.2, 119.9, 119.2, 116.0, 109.8, 94.9, 55.6, 53.5, 47.6, 32.4, 30.2, 17.8; IR (film) 3290, 2949, 1617, 1455, 1414, 1161 cm^{-1}; HRMS (ESI) calcd for $C_{21}H_{23}N_2O_2$ (M+H) *m*/*z* 335.1754; found 335.1759.



(*R*)-1-benzyl-5-(4-fluoro-1*H*-indol-3-yl)pyrrolidin-2-one (2m). The product was prepared by Method B on 0.34 mmol scale. The crude product (96%ee) was purified by trituration from Et_2O (2x2 mL) to yield **2m** (0.066 g, 57% yield) as a colorless crystalline solid. This material was determined to be 99% ee by chiral SFC analysis (ChiralPak AS-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 3.85 min, t_r(major) = 5.33 min. $[\alpha]^{29}_D$ = –39° (*c* = 0.7 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.85 (br s, 1H); 7.27–7.17 (m, 4H); 7.14–7.10 (m, 3H); 7.69 (d, *J* = 2.0 Hz, 1H); 6.77 (dd, *J* = 11.0, 7.5 Hz, 1H); 5.07 (d, *J* = 14.5 Hz, 1H); 4.92 (t, *J* = 7.0 Hz, 1H); 3.72 (d, *J* = 15.0, 1H); 2.70 (ddd, *J* = 16.5, 10.5, 6.0 Hz, 1H); 2.53 (ddd, *J* = 16.5, 10.0, 7.0 Hz, 1H); 2.47–2.39 (m, 1H); 2.22–2.16 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$)[11] δ 175.6, 157.8, 155.8, 140.1, 140.0, 137.1, 128.7, 128.5, 127.5, 123.4, 123.3, 123.0, 114.9, 114.8, 114.7, 114.5, 107.9, 107.9, 105.5, 105.4, 55.3, 44.6, 30.5, 27.6; IR (film) 3221, 2926, 1661, 1449, 1420 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{18}FN_2O$ (M+H) *m*/*z* 309.1403; found 309.1397.



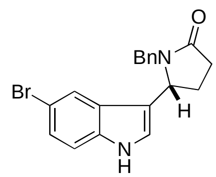
(*R*)-1-benzyl-5-(4-bromo-1*H*-indol-3-yl)pyrrolidin-2-one (2n). The product was prepared by Method B on 0.34 mmol scale. The crude product was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2n** (0.059 g, 47% yield) as a colorless foam. This material was determined to be 97% ee by chiral SFC analysis (Pirkle (*S*,*S*)-Whelk, 4.0 mL/min, 220 nm, 15% MeOH, t_r(minor) = 7.22 min, t_r(major) = 9.39 min. $[\alpha]^{29}_D$ = +10° (*c* = 0.5 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 9.35 (br s, 1H); 7.37 (d, *J* = 8.5 Hz, 1H); 7.31–7.24 (m, 3H); 7.19 (d, *J* = 7.0 Hz, 2H); 7.05 (d, *J* = 8.0 Hz, 1H);[9] 7.03 (d, *J* = 8.0 Hz, 1H);[9] 6.99 (d, *J* = 3.0 Hz, 1H); 5.64 (d, *J* = 7.0 Hz, 1H); 5.15 (d, *J* = 14.5 Hz, 1H); 3.93 (d, *J* = 14.5 Hz, 1H); 2.62–2.54 (m, 1H); 2.53–2.41 (m, 2H); 2.12–2.04 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.9, 138.3, 136.6, 128.6, 128.1, 127.5, 124.6, 124.0, 123.1, 122.2, 116.4, 113.3, 110.9, 54.4, 44.7, 29.2, 29.0; IR (film) 3243, 2925, 1667, 1446, 1336, 1186 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{18}BrN_2O$ (M+H) *m*/*z* 369.0602; found 369.0611.



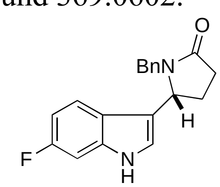
(*R*)-1-benzyl-5-(5-fluoro-1*H*-indol-3-yl)pyrrolidin-2-one (2o). The product was prepared by Method B on 0.34 mmol scale. The crude product was purified on activity III basic alumina (8:2

[11] Note extra ^{13}C resonances due to C–F coupling.

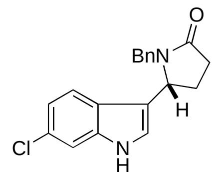
CH_2Cl_2:EtOAc) to yield **2o** (0.058 g, 87% yield) as a colorless foam. This material was determined to be 94% ee by chiral SFC analysis (ChiralPak AS-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 3.25 min, t_r(major) = 4.69 min. $[\alpha]^{29}_D$ = –69° (*c* = 1.0 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.89 (br s, 1H); 7.33 (dd, *J* = 9.0, 4.5 Hz, 1H); 7.28–7.23 (m, 3H); 7.10–7.06 (m, 3H); 6.97 (ddd, *J* = 9.0, 9.0, 2.0 Hz, 1H); 5.07 (d, *J* = 14.5 Hz, 1H); 4.74 (dd, *J* = 8.0, 7.0 Hz, 1H); 3.59 (d, *J* = 15.0 Hz, 1H); 2.75–2.67 (m, 1H); 2.59 (ddd, *J* = 18.0, 10.0, 8.5 Hz, 1H); 2.44–2.35 (m, 1H); 2.24–2.15 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$)[11] δ 175.1, 158.7, 156.8, 136.6, 133.5, 133.3, 128.5, 128.3, 127.4, 125.6, 125.5, 124.7, 124.6, 114.8, 112.4, 112.4, 112.3, 111.0, 110.8, 103.9, 103.7, 54.7, 44.2, 30.8, 26.7; IR (film) 3270, 1675, 1484, 1445, 1419, 702 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{18}FN_2O$ (M+H) *m/z* 309.1403; found 309.1405.



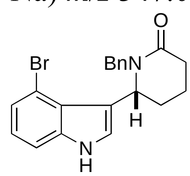
(*R*)-1-benzyl-5-(5-bromo-1*H*-indol-3-yl)pyrrolidin-2-one (2p). The product was prepared by Method B on 0.34 mmol scale. The crude product was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2p** (0.102 g, 80% yield) as a colorless foam. This material was determined to be 93% ee by chiral SFC analysis (ChiralPak AS-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 5.71 min, t_r(major) = 7.41 min. $[\alpha]^{28}_D$ = –51° (*c* = 1.0 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 9.01 (br s, 1H); 7.53 (dd, *J* = 1.5, 1.0 Hz, 1H); 7.29 (d, *J* = 1.0 Hz, 1H); 7.28–7.22 (m, 3H); 7.09–7.04 (m, 2H); 7.03 (s, 1H); 5.06 (d, *J* = 14.5 Hz, 1H); 4.74 (t, *J* = 6.5 Hz, 1H); 3.59 (d, *J* = 15.0 Hz, 1H); 2.75–2.68 (m, 1H); 2.58 (ddd, *J* = 17.5, 10.0, 8.0 Hz, 1H); 2.43–2.33 (m, 1H); 2.23–2.12 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.1, 136.5, 135.4, 128.5, 128.3, 127.4, 127.0, 125.4, 124.0, 121.3, 114.3, 113.1, 113.1, 54.6, 44.3, 30.7, 26.7; IR (film) 3281, 1665, 1453, 1418, 731 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{18}BrN_2O$ (M+H) *m/z* 369.0602; found 369.0602.



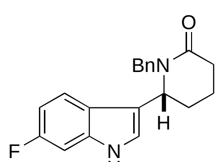
(*R*)-1-benzyl-5-(6-fluoro-1*H*-indol-3-yl)pyrrolidin-2-one (2q). The product was prepared by Method B on 0.34 mmol scale. The crude product was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2q** (0.080 g, 75% yield) as a colorless foam. This material was determined to be 96% ee by chiral SFC analysis (ChiralPak AS-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 3.28 min, t_r(major) = 4.67 min. $[\alpha]^{27}_D$ = –84° (*c* = 1.1 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.57 (br s, 1H); 7.36 (dd, *J* = 9.0, 5.0 Hz, 1H); 7.28–7.22 (m, 3H); 7.12–7.08 (m, 3H); 7.01 (d, *J* = 2.5 Hz, 1H); 6.87 (ddd, *J* = 9.5, 9.5, 2.0 Hz , 1H); 5.07 (d, *J* = 14.5 Hz, 1H); 4.75 (dd, *J* = 8.5, 6.5 Hz, 1H); 3.57 (d, *J* = 15.0 Hz, 1H); 2.75–2.66 (m, 1H); 2.59 (ddd, *J* = 17.5, 9.5, 8.0 Hz, 1H); 2.44–2.36 (m, 1H); 2.23–2.14 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$)[11] δ 175.0, 161.1, 159.2, 137.0, 136.9, 136.7, 128.4, 128.3, 127.4, 123.2, 123.1, 121.8, 119.7, 119.6, 115.1, 108.9, 108.7, 98.1, 97.9, 54.7, 44.2, 30.8, 26.7; IR (film) 3242, 2925, 1661, 1446, 1143, 701 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{17}FN_2NaO$ (M+Na) *m/z* 331.1217; found 331.1223.



(*R*)-1-benzyl-5-(6-chloro-1*H*-indol-3-yl)pyrrolidin-2-one (2r). The product was prepared by Method B on 0.34 mmol scale. The crude product was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2r** (0.090 g, 81% yield) as a colorless foam. This material was determined to be 96% ee by chiral SFC analysis (ChiralPak AS-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 4.67 min, t_r(major) = 6.43 min. $[\alpha]^{29}_D$ = –57° (*c* = 0.5 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.69 (br s, 1H); 7.45 (d, *J* = 1.5 Hz, 1H); 7.39 (d, *J* = 7.0 Hz, 1H); 7.32–7.26 (m, 3H); 7.13–7.10 (m, 3H); 7.06 (d, *J* = 2.0 Hz, 1H); 5.11 (d, *J* = 12.5 Hz, 1H); 4.79 (t, *J* = 5.5 Hz, 1H); 3.59 (d, *J* = 12.5 Hz, 1H); 2.78–2.71 (m, 1H); 2.63 (ddd, *J* = 14.5, 8.0, 6.5 Hz, 1H); 2.48–2.40 (m, 1H); 2.25–2.18 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.1, 137.3, 136.6, 128.6, 128.5, 128.3, 127.4, 123.9, 123.5, 120.7, 119.7, 115.1, 111.6, 54.6, 44.2, 30.8, 26.8; IR (film) 3245, 1669, 1459, 1419, 1258, 804, 703 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{17}ClN_2NaO$ (M+Na) *m/z* 347.0922; found 347.0929.

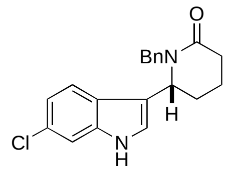


(*R*)-1-benzyl-6-(4-bromo-1*H*-indol-3-yl)piperidin-2-one (2s). The product was prepared by Method B on 0.34 mmol scale. The crude product was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2s** (0.016 g, 12% yield) as a colorless foam. This material was determined to be 85% ee by chiral SFC analysis (Pirkle (*S*,*S*)-Whelk, 4.0 mL/min, 220 nm, 15% MeOH, t_r(minor) = 5.86 min, t_r(major) = 6.56 min. $[\alpha]^{29}_D$ = –21° (*c* = 0.5 g/100mL, MeOH); ^{1}H NMR (500 MHz, d_6-DMSO) 11.6 (br s, 1H); 7.52 (d, *J* = 7.5 Hz, 1H); 7.42–7.38 (m, 2H); 7.35–7.31 (m, 2H); 7.28–7.22 (m, 3H); 7.09 (t, *J* = 8.0 Hz, 1H); 5.59–5.51 (m, 1H); 5.43 (d, *J* = 15.5 Hz, 1H); 3.69 (d, *J* = 15.5 Hz, 1H); 2.60–4.46 (m, 2H); 2.16–2.06 (m, 2H); 1.86–1.78 (m, 1H); 1.69–1.60 (m, 1H); ^{13}C NMR (125 MHz, d_6-DMSO) δ 169.2, 138.4, 137.9, 128.4, 127.3, 126.9, 125.4, 123.7, 123.1, 122.3, 114.8, 112.2, 111.5, 52.3, 47.5, 31.6, 30.3, 15.7; IR (film) 3247, 2954, 1617, 1413, 1334, 1178, 734 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{20}BrN_2O$ (M+H) *m/z* 383.0754; found 383.0753.

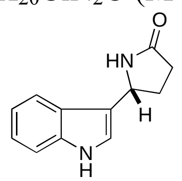


(*R*)-1-benzyl-6-(6-fluoro-1*H*-indol-3-yl)piperidin-2-one (2t). The product was prepared by Method B on 0.34 mmol scale. The crude product was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2t** (0.016 g, 12% yield) as a colorless foam. This material was determined to be 96% ee by chiral SFC analysis (Pirkle (*S*,*S*)-Whelk, 4.0 mL/min, 220 nm, 15% MeOH, t_r(minor) = 6.43 min, t_r(major) = 8.44 min. $[\alpha]^{32}_D$ = –52° (*c* = 0.5 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.91 (br s, 1H); 7.35 (dd, *J* = 8.5, 5.0 Hz, 1H); 7.31–7.22 (m, 3H); 7.16 (d, *J* = 8.5 Hz, 2H); 7.09 (dd, *J* = 9.5, 2.5 Hz, 1H); 6.97 (d, *J* = 2.0 Hz, 1H); 6.87 (ddd, *J* = 10.0, 10.0, 2.5 Hz, 1H); 5.59 (d, *J* = 15.0, 1H); 4.83 (t, *J* = 5.0 Hz, 1H); 3.66 (d, *J* = 15.0 Hz, 1H); 2.70–2.56 (m, 2H); 2.12–2.00 (m, 2H); 1.91–

1.81 (m, 1H); 1.75–1.67 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$)[11] δ 170.8, 161.0, 159.1, 137.5, 136.9, 136.8, 128.5, 127.9, 127.2, 122.81, 122.79, 122.1, 119.3, 119.2, 115.9, 108.6, 108.4, 98.0, 97.8, 53.4, 47.7, 32.3, 30.1, 17.7; IR (film) 3262, 2951, 1624, 1452, 1143, 953 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{19}FN_2NaO$ (M+Na) *m/z* 345.1374; found 345.1379.



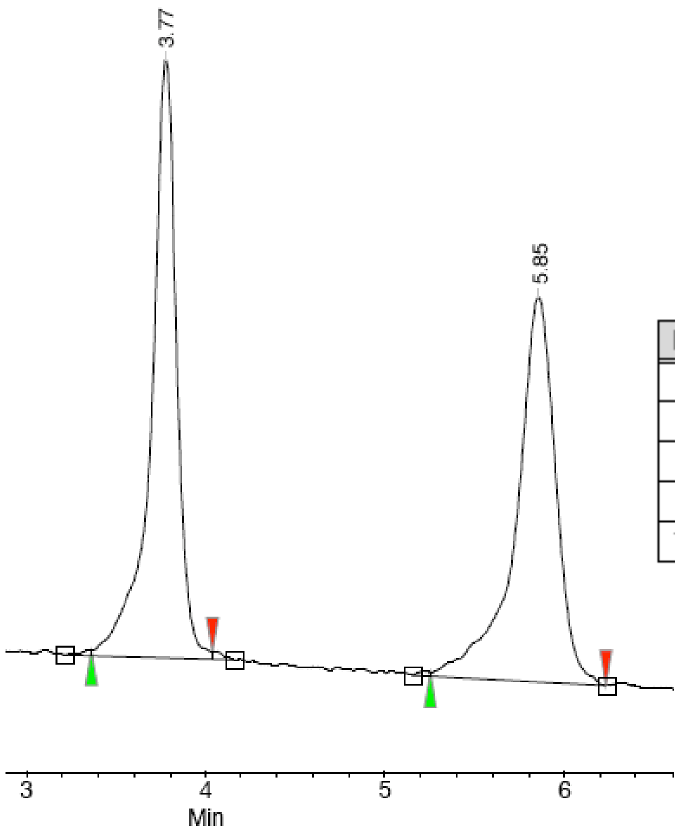
(*R*)-1-benzyl-6-(6-chloro-1*H*-indol-3-yl)piperidin-2-one (2u). The product was prepared by Method B on 0.34 mmol scale. The crude product was purified on activity III basic alumina (8:2 CH_2Cl_2:EtOAc) to yield **2u** (0.046 g, 40% yield) as a colorless foam. This material was determined to be 92% ee by chiral SFC analysis (ChiralPak AS-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 4.27 min, t_r(major) = 5.77 min. $[\alpha]^{29}_D$ = –35° (*c* = 0.7 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.90 (br s, 1H); 7.40 (d, *J* = 1.5 Hz, 1H); 7.35 (d, *J* = 8.5 Hz, 1H); 7.32–7.49 (m, 3H); 7.15 (d, *J* = 8.5 Hz, 2H); 7.07 (dd, *J* = 8.5, 2.0 Hz, 1H); 6.98 (d, *J* = 2.5 Hz, 1H); 5.60 (d, *J* = 15.0 Hz, 1H); 4.83 (t, *J* = 5.0 Hz, 1H); 3.64 (d, *J* = 14.5 Hz, 1H); 2.69–2.57 (m, 2H); 2.09–2.00 (m, 2H); 1.89–1.82 (m, 1H); 1.75–1.68 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.8, 137.5, 137.2, 128.5, 128.3, 127.9, 127.3, 124.1, 123.1, 120.5, 119.4, 116.0, 111.6, 53.3, 47.7, 32.3, 30.2, 17.7; IR (film) 3245, 2948, 1620, 1451, 1413, 1264 cm^{-1}; HRMS (ESI) calcd for $C_{20}H_{20}ClN_2O$ (M+H) *m/z* 339.1259; found 339.1274.

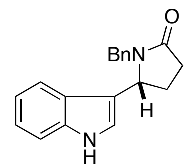


(*R*)-5-(1*H*-indol-3-yl)pyrrolidin-2-one (15). A 3-neck reaction vessel fitted with a liquid NH_3 condenser was charged with **2a** (38.5 mg, 0.13 mmol, 94%ee) under a positive flow of N_2 and THF (1 mL) was added. The solution and condenser were cooled to –78 °C. In a separate 3-neck flask with an attached bubbler was cooled to –78 °C and NH_3 (~50 mL) was condensed directly from the tank into this flask. The NH_3 (~10 mL) was then redistilled from the 3-neck flask *via* cannula into the reaction vessel containing **2a**. Sodium metal (45 mg, 2.0 mmol) was added to the reaction to create a dark blue solution. After 10 min, isoprene (~1 mL) was added until the dark blue color dissipated, giving a yellow solution. Solid NH_4Cl was added until a colorless mixture was produced. The mixture was allowed to warm slowly to rt, and was partitioned between H_2O (10 mL) and EtOAc (20 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3x10 mL). The combined organic layers were dried (Na_2SO_4), concentrated *in vacuo*, and the residue purified on activity III basic alumina to provide 22 mg (85% yield) of **15** as a colorless solid. This material was determined to be 94% ee by chiral SFC analysis (ChiralPak AD-H, 4.0 mL/min, 220 nm, 20% MeOH, t_r(minor) = 5.81 min, t_r(major) = 7.71 min. $[\alpha]^{29}_D$ = +29° (*c* = 0.5 g/100mL, MeOH); ^{1}H NMR (500 MHz, $CDCl_3$) 8.46 (br s, 1H); 7.60 (d, *J* = 8.0 Hz, 1H); 7.39 (ddd, *J* = 8.0, 1.0, 1.0 Hz, 1H); 7.22 (ddd, *J* = 7.5, 7.5, 1.5 Hz, 1H); 7.13 (ddd, *J* = 8.0, 8.0, 1.0 Hz, 1H); 7.10 (d, *J* = 2.5 Hz, 1H); 6.29 (br s, 1H); 5.07 (t, *J* = 5.5 Hz, 1H); 2.60–2.42 (m, 3H); 2.30–2.20 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 178.3, 136.9, 125.1, 122.5, 121.1, 119.8, 118.8, 117.1, 111.6, 51.5, 30.5, 29.5; IR (film) 3404, 3267, 1683, 1457, 1264 cm^{-1}; HRMS (ESI) calcd for $C_{12}H_{12}N_2O$ (M+H) *m/z* 201.1022; found 201.1021.

F. Chiral SFC Data

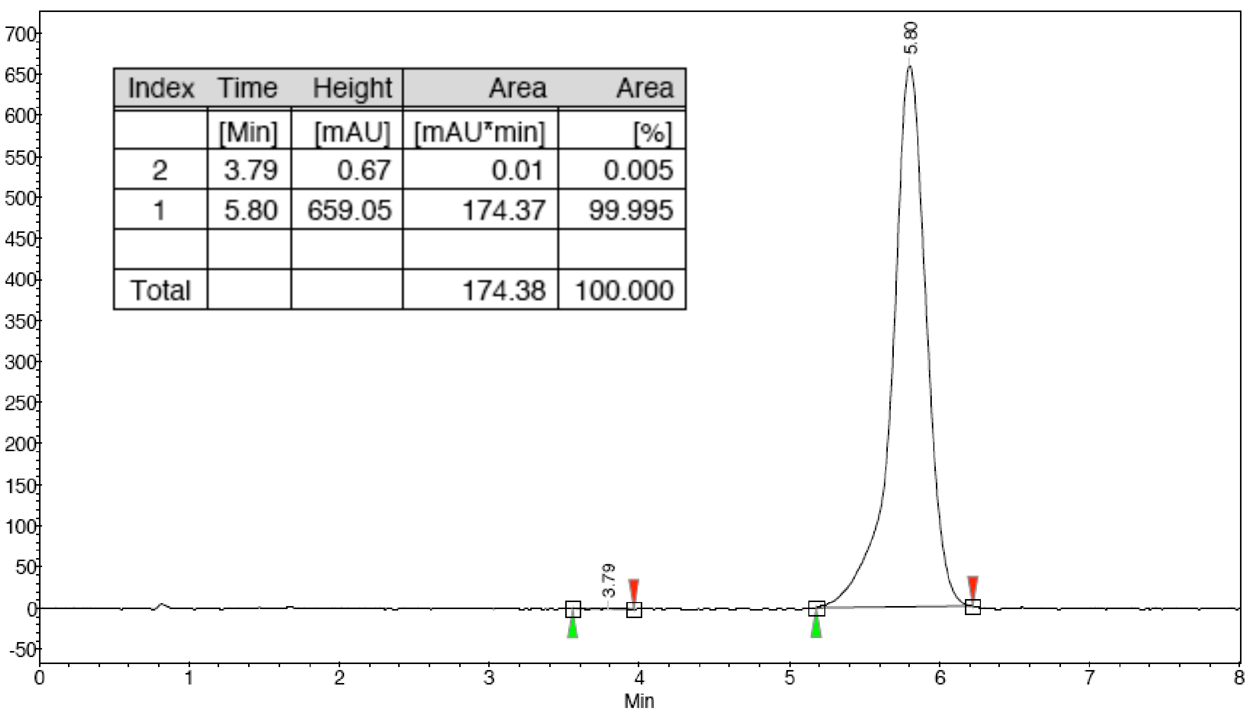
Racemate: ChiralPak AS-H, 20% MeOH, 220 nm





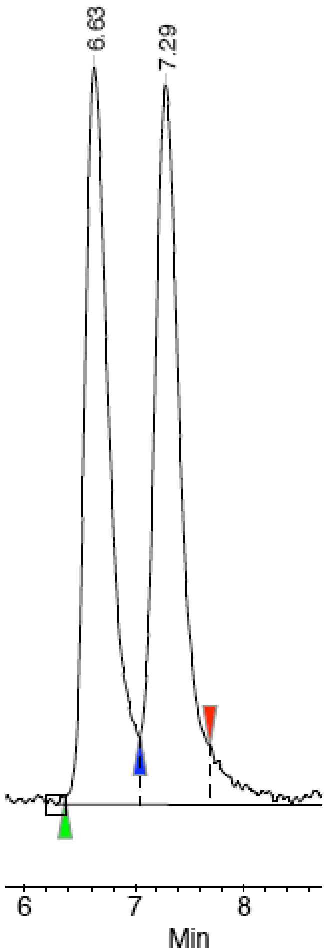
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	3.77	85.37	14.35	50.231
2	5.85	55.01	14.21	49.769
Total			28.56	100.000

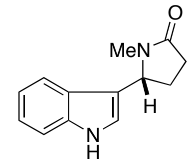
99% ee



Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
2	3.79	0.67	0.01	0.005
1	5.80	659.05	174.37	99.995
Total			174.38	100.000

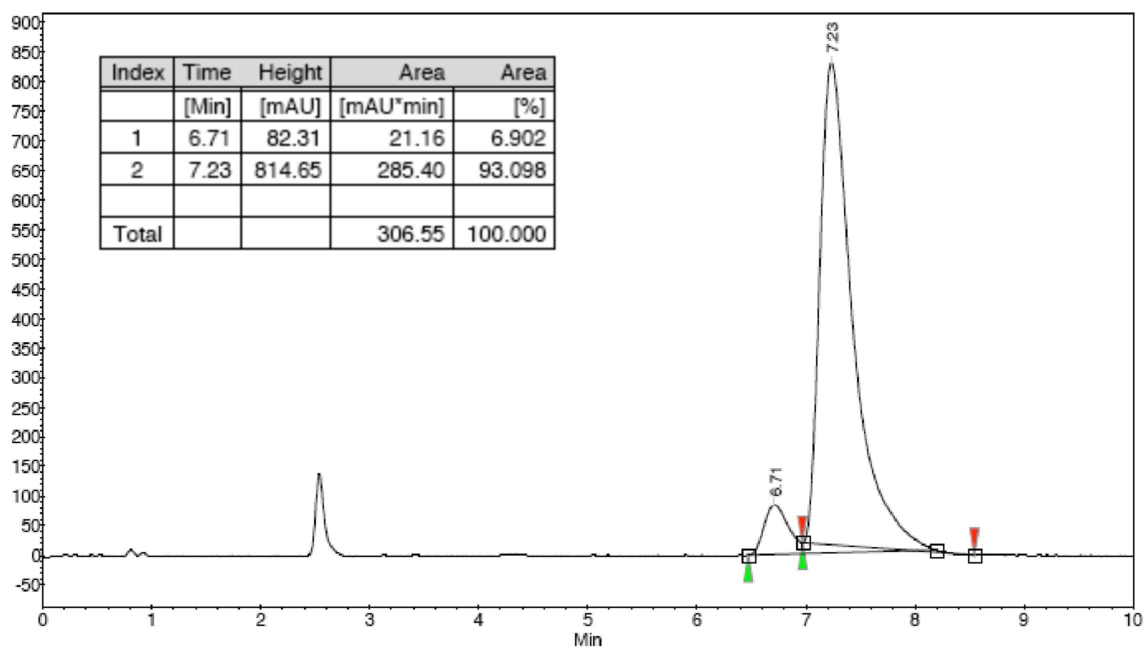
Racemate: ChiralPak AD-H, 10 MeOH/CO_2, 220 nm





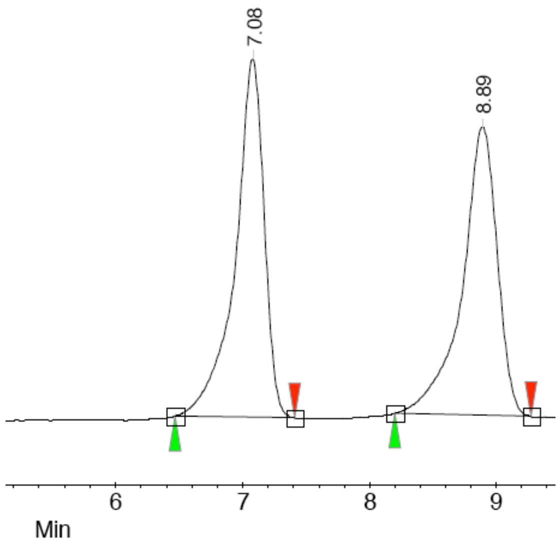
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	0.86	25.12	2.40	3.853
2	6.63	111.73	29.50	47.377
3	7.29	108.94	30.37	48.771
Total			62.28	100.000

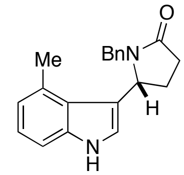
86% ee



Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	6.71	82.31	21.16	6.902
2	7.23	814.65	285.40	93.098
Total			306.55	100.000

Racemate: Pirkle (*S*,*S*)-Whelk, 15% MeOH, 220 nm

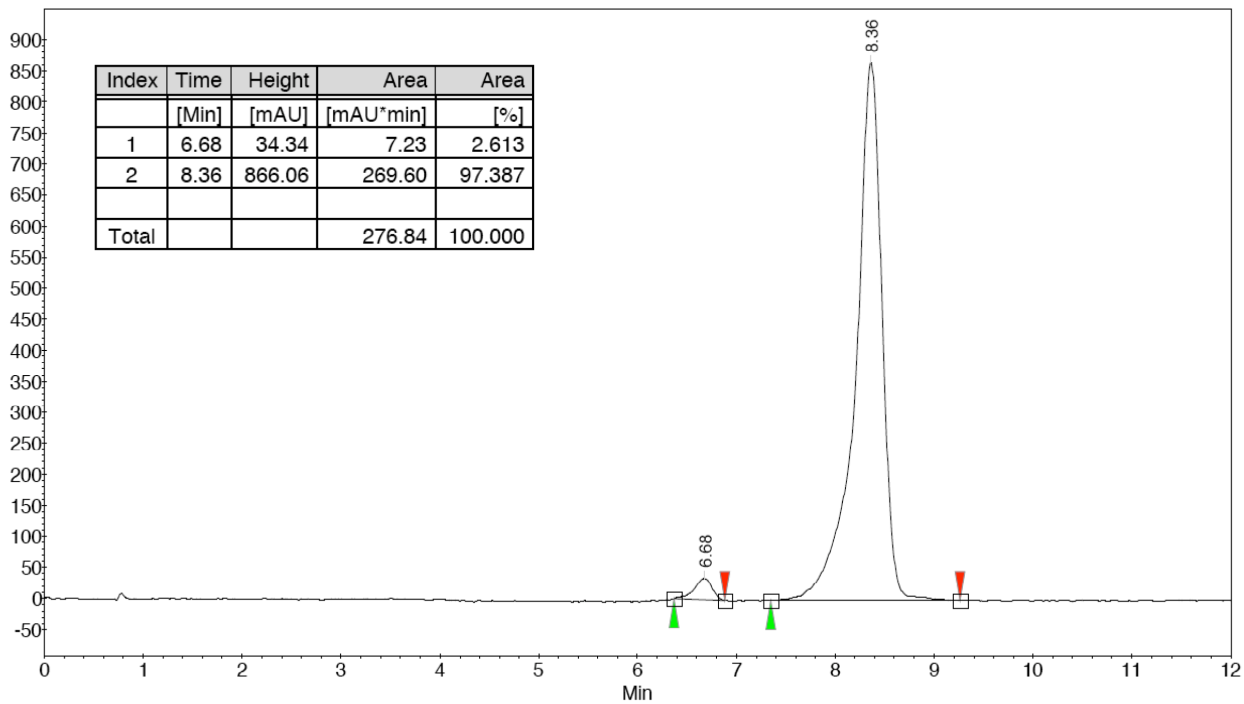




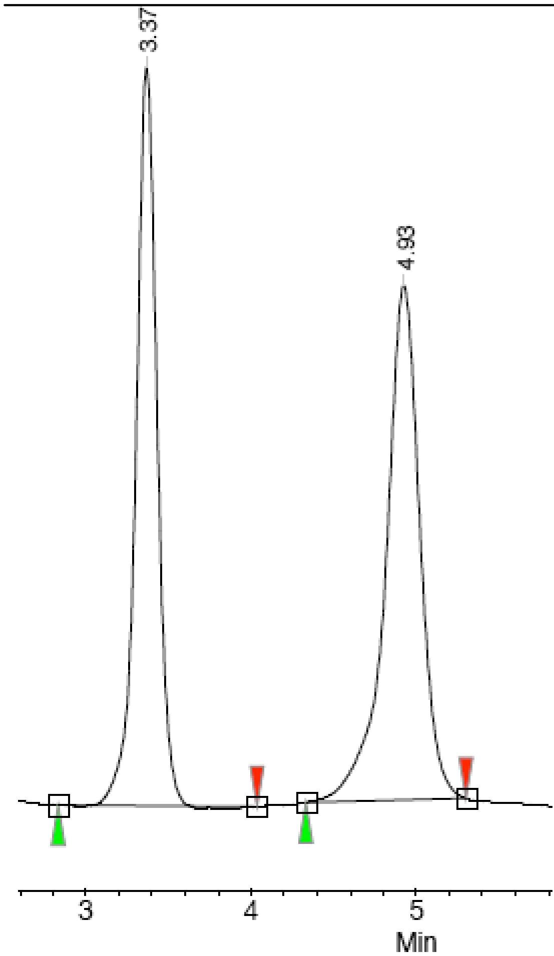
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	7.08	842.85	223.32	50.637
2	8.89	679.70	217.71	49.363
Total			441.03	100.000

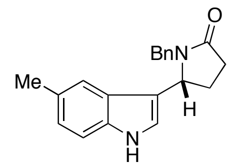
95% ee

Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	6.68	34.34	7.23	2.613
2	8.36	866.06	269.60	97.387
Total			276.84	100.000



Racemate: ChiralPak AS-H, 20% MeOH, 220 nm

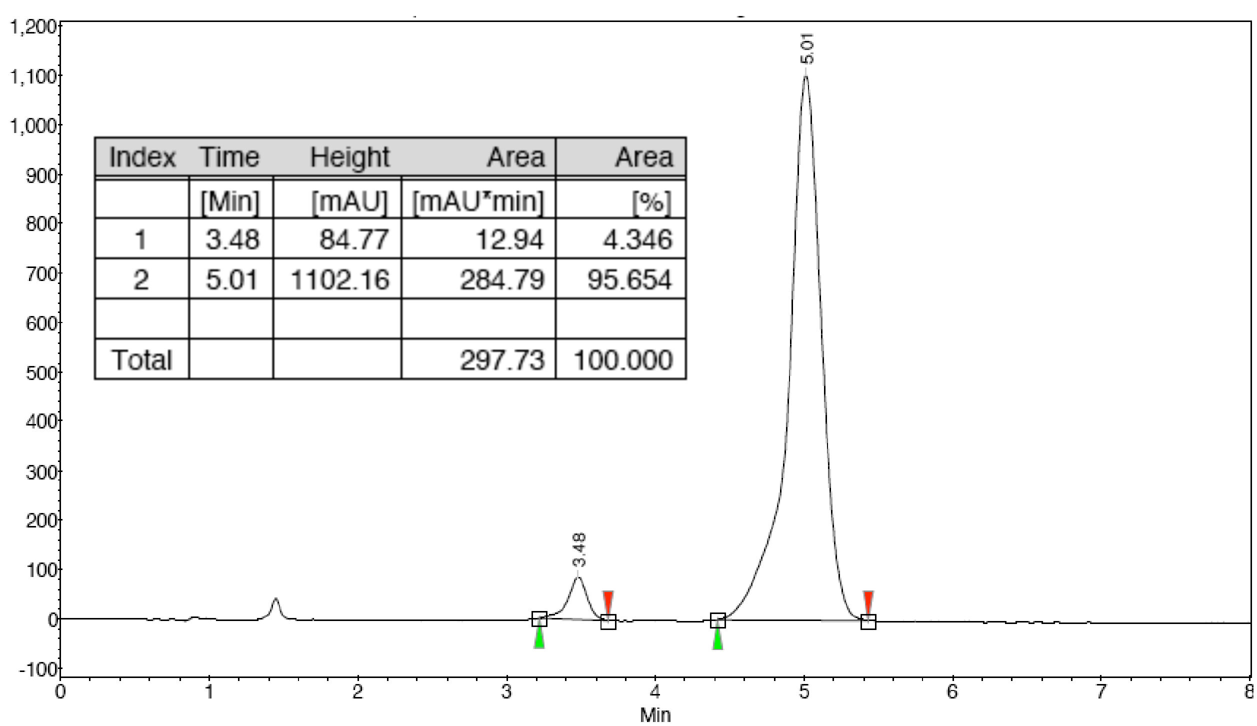




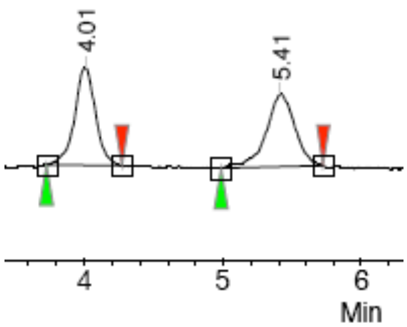
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
2	3.37	392.72	57.75	46.643
1	4.93	273.79	66.06	53.357
Total			123.80	100.000

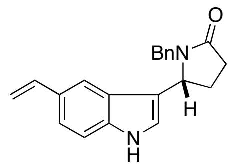
91% ee

Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	3.48	84.77	12.94	4.346
2	5.01	1102.16	284.79	95.654
Total			297.73	100.000



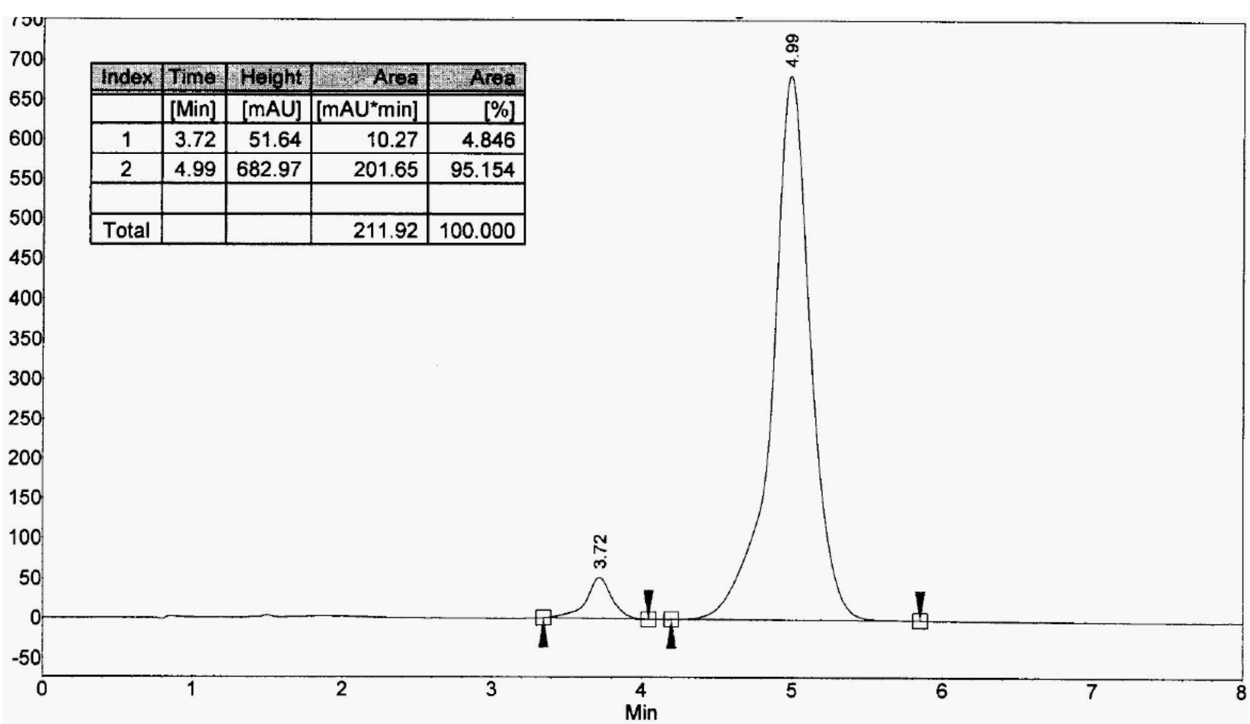
Racemate: ChiralPak AS-H, 20 % MeOH, 220 nm





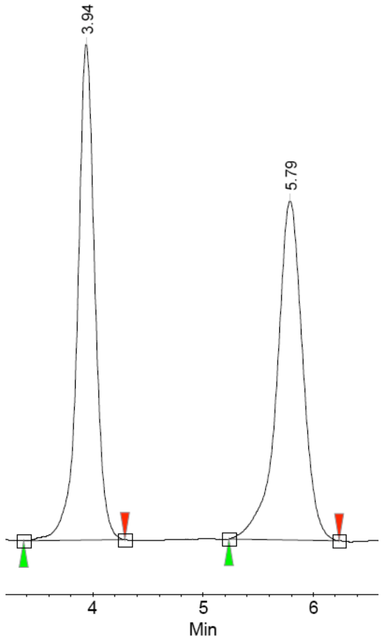
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	4.01	63.08	11.03	49.768
2	5.41	46.46	11.14	50.232
Total			22.17	100.000

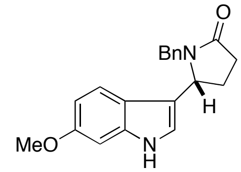
90% ee



Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	3.72	51.64	10.27	4.846
2	4.99	682.97	201.65	95.154
Total			211.92	100.000

Racemate: ChiralPak, AS-H, 20% MeOH/CO_2, 220 nm

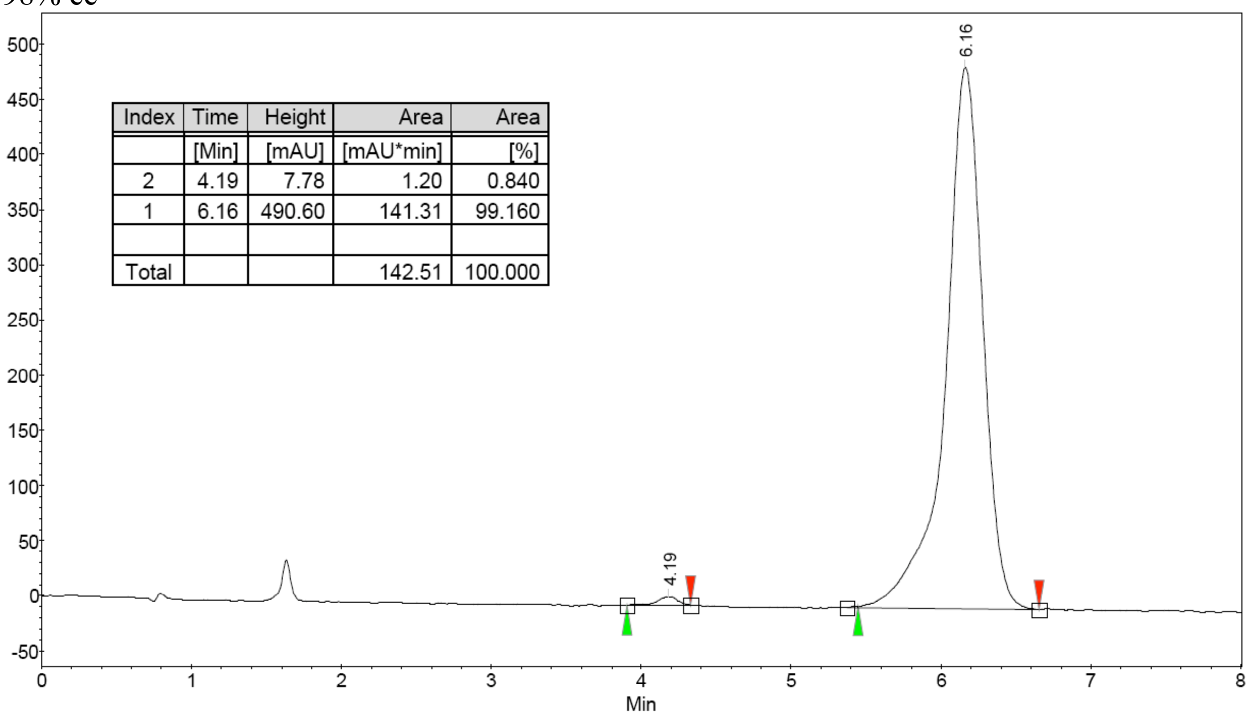




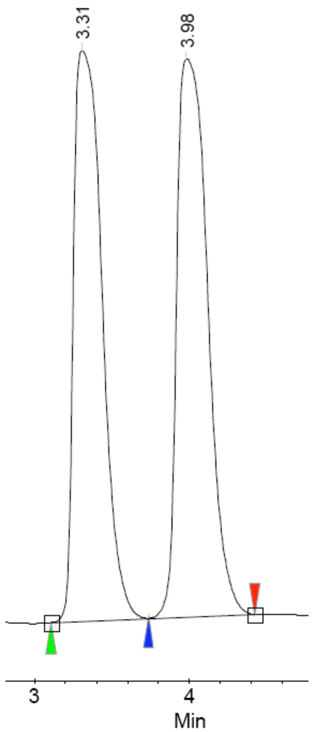
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	3.94	715.40	129.63	49.403
2	5.79	489.48	132.76	50.597
Total			262.39	100.000

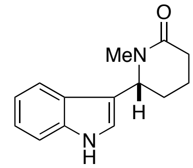
98% ee

Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
2	4.19	7.78	1.20	0.840
1	6.16	490.60	141.31	99.160
Total			142.51	100.000



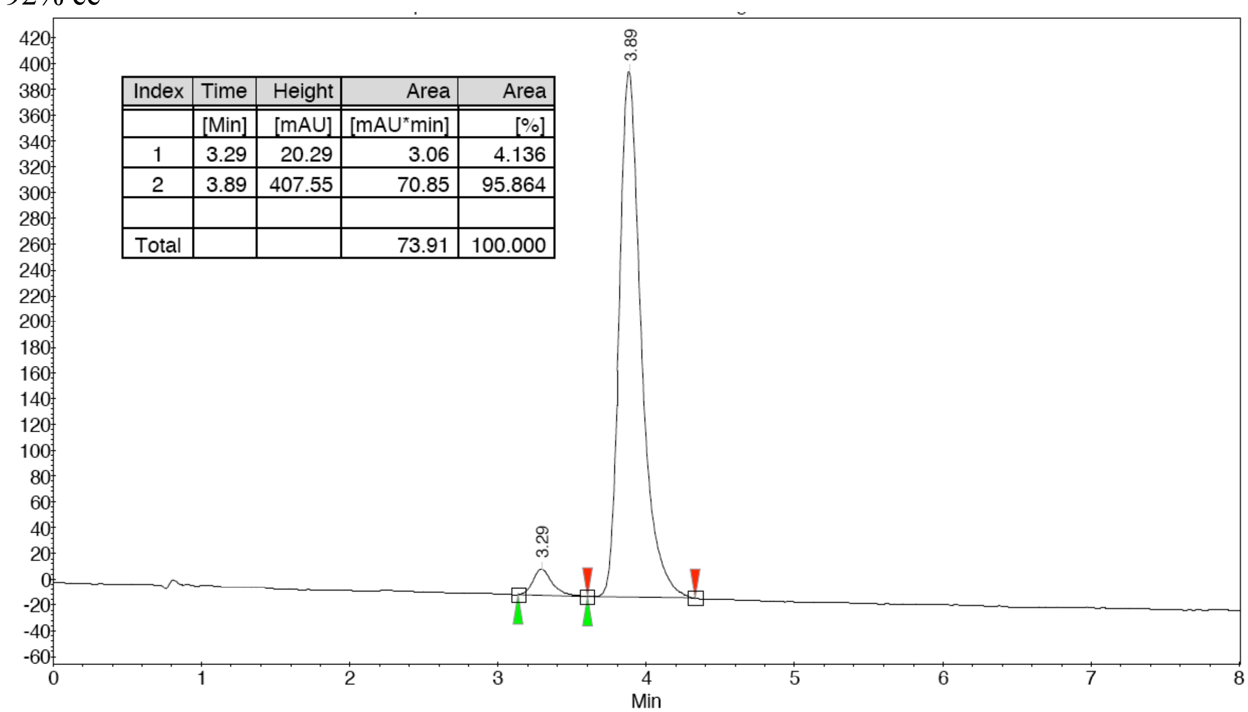
Racemate: ChiralPak AD-H, 20% MeOH/CO_2, 220 nm





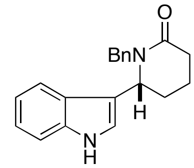
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	3.31	2282.13	482.11	48.472
2	3.98	2232.25	512.50	51.528
Total			994.61	100.000

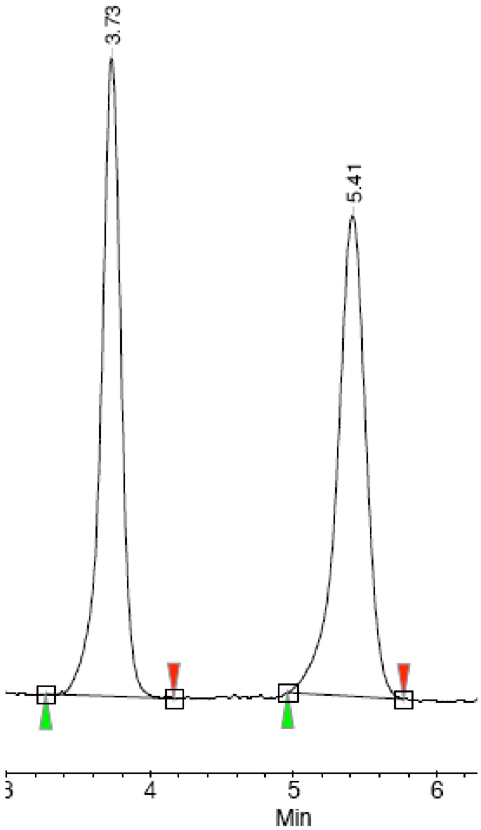
92% ee



Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	3.29	20.29	3.06	4.136
2	3.89	407.55	70.85	95.864
Total			73.91	100.000

Racemate: ChiralPak AS-H, 20% MeOH/CO_2, 220 nm

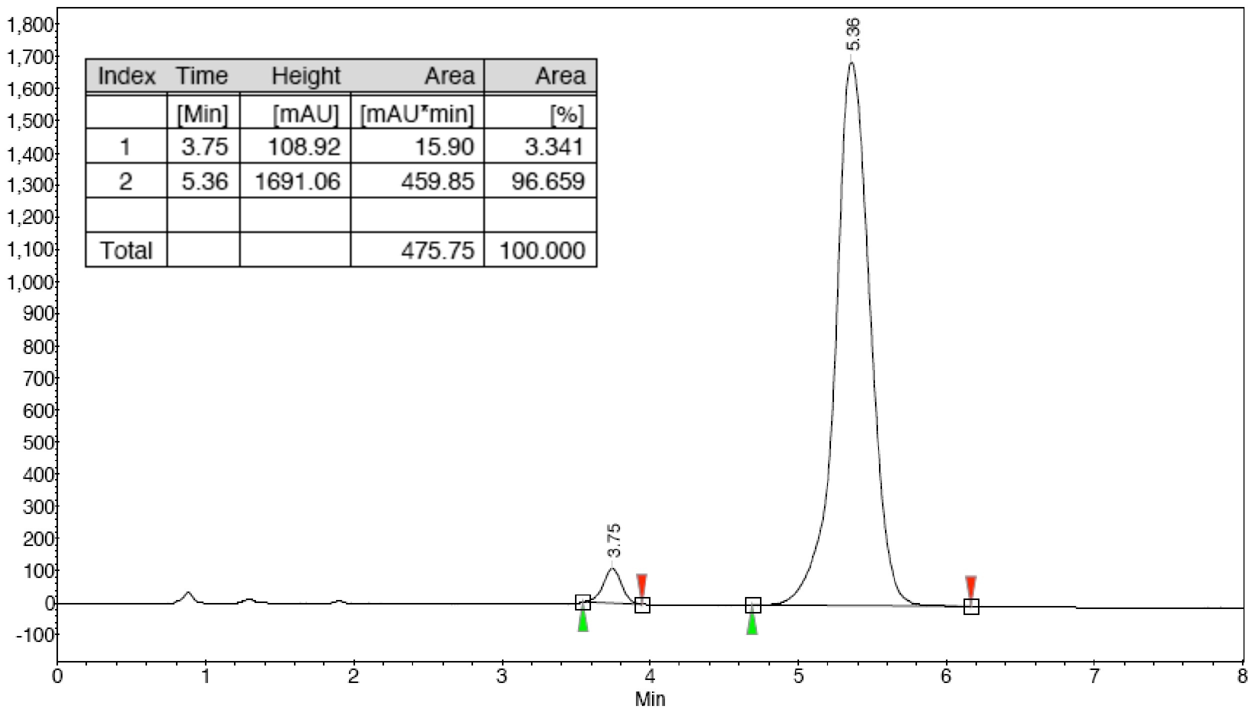




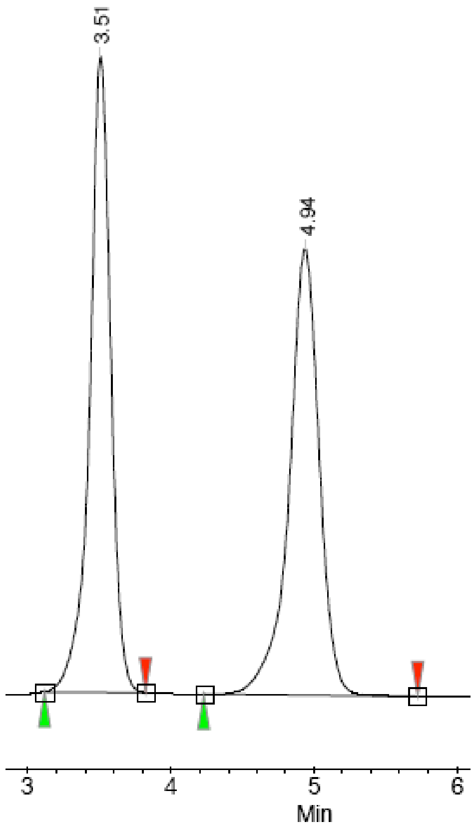
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
2	3.73	188.41	30.57	48.016
1	5.41	141.82	33.09	51.984
Total			63.66	100.000

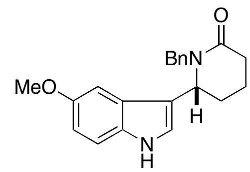
93% ee

Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	3.75	108.92	15.90	3.341
2	5.36	1691.06	459.85	96.659
Total			475.75	100.000



Racemate: ChiralPak AS-H, 20% MeOH/CO_2, 220 nm

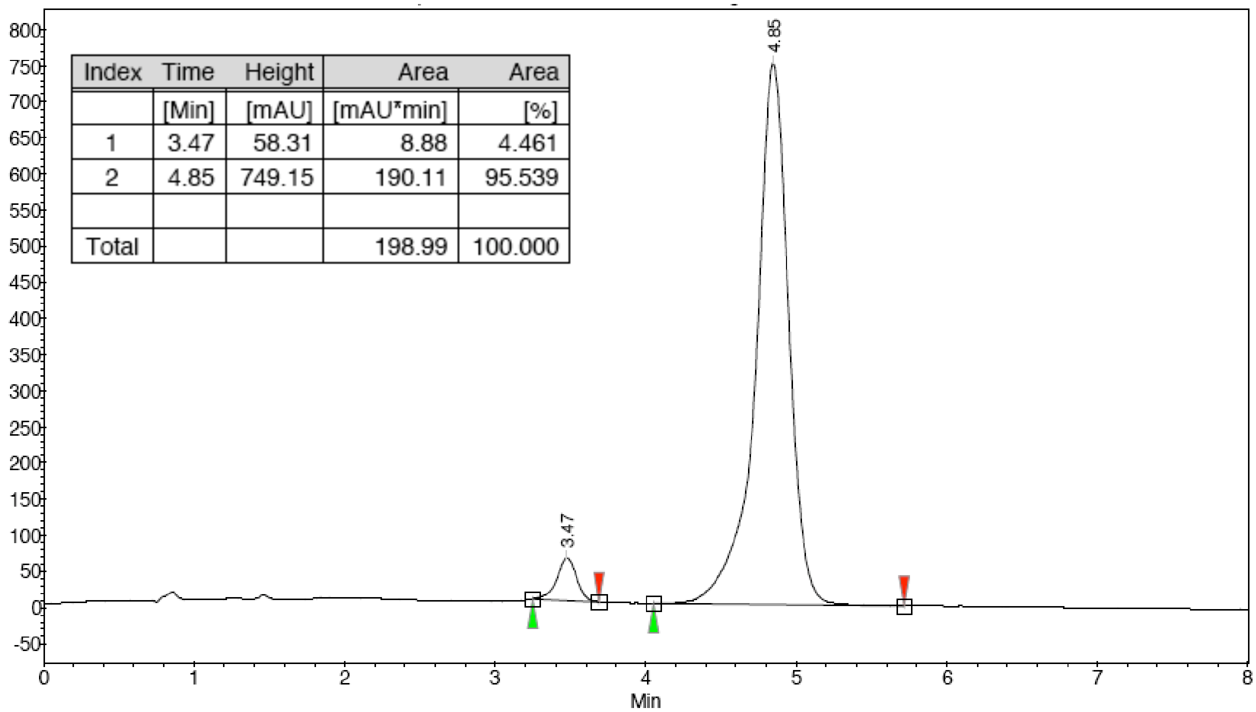




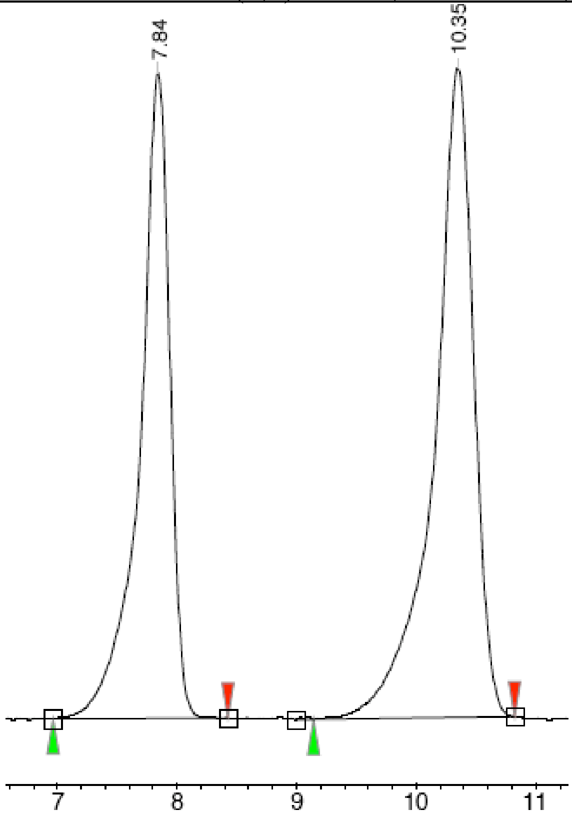
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	3.51	556.19	94.56	49.667
2	4.94	390.92	95.83	50.333
Total			190.39	100.000

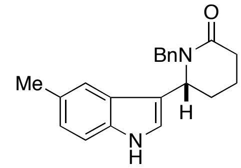
90%ee

Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	3.47	58.31	8.88	4.461
2	4.85	749.15	190.11	95.539
Total			198.99	100.000



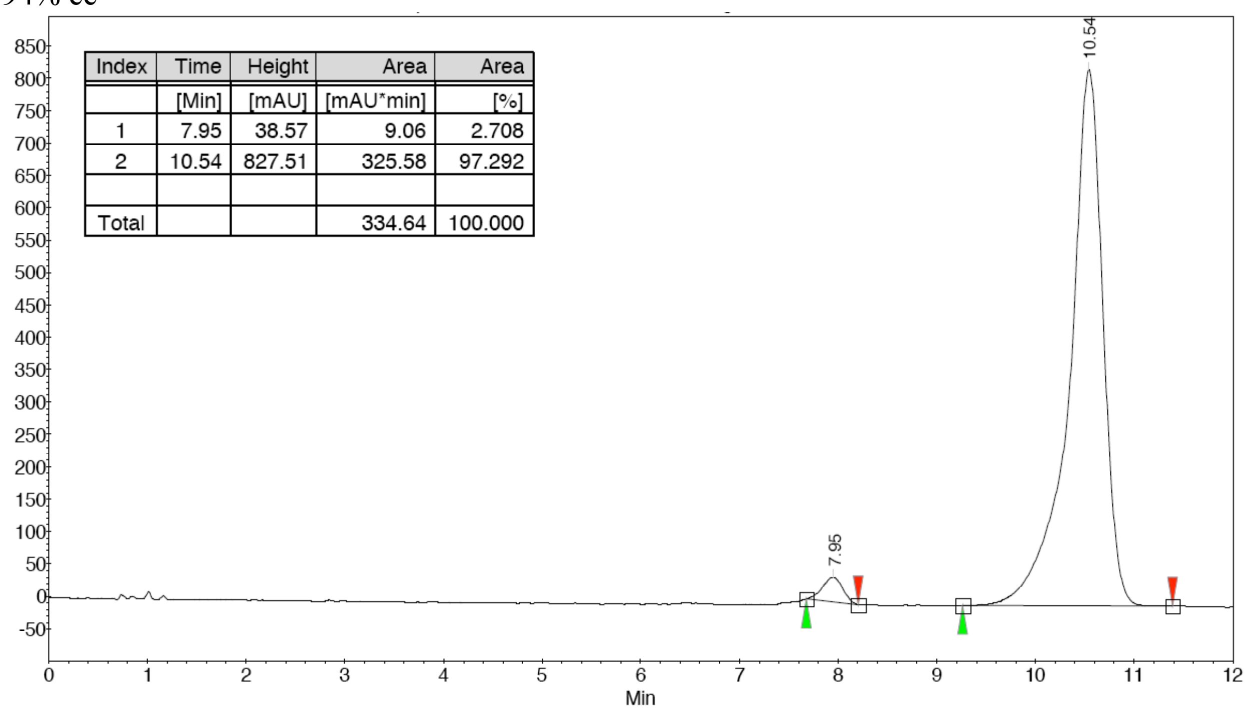
Racemate: Pirkle (*S*,*S*)-Whelk, 15% MeOH, 220 nm





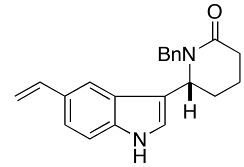
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
2	7.84	437.16	126.63	43.027
1	10.35	439.77	167.67	56.973
Total			294.30	100.000

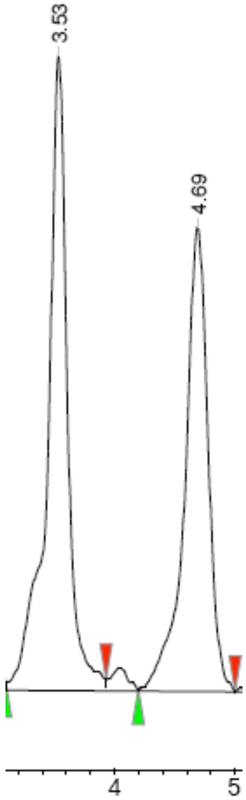
94% ee



Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	7.95	38.57	9.06	2.708
2	10.54	827.51	325.58	97.292
Total			334.64	100.000

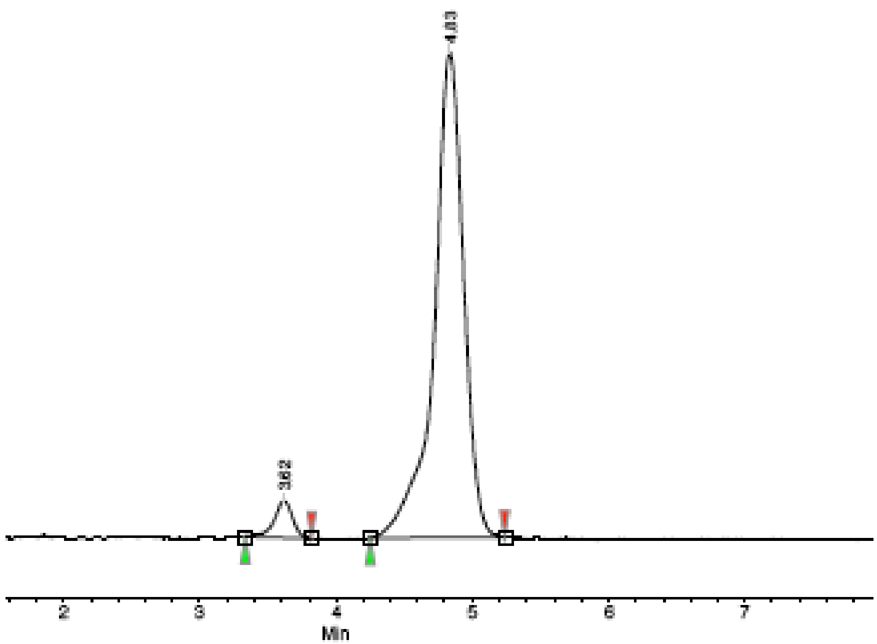
Racemate: ChiralPak AS-H, 20% MeOH/CO_2, 220 nm





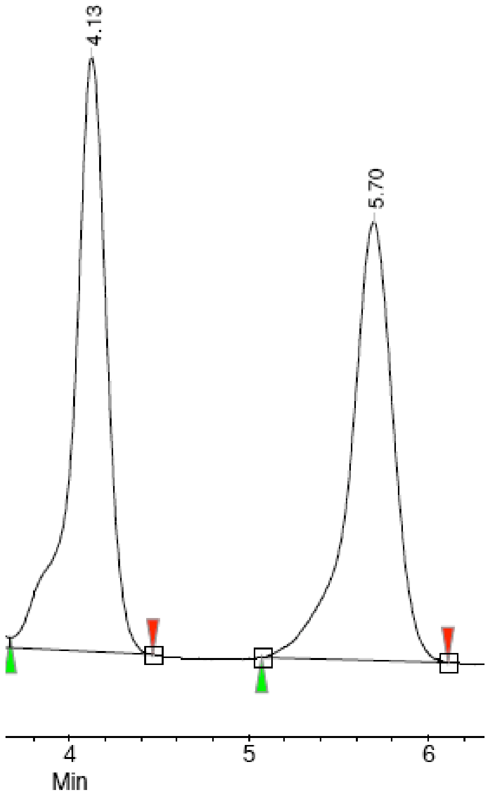
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	3.53	127.17	24.48	53.892
2	4.69	93.13	20.95	46.108
Total			45.43	100.000

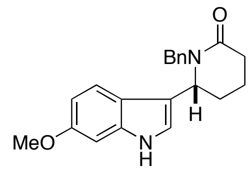
91% ee



Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	3.62	42.37	6.64	4.536
2	4.83	559.83	139.78	95.464
Total			146.42	100.000

Racemate: ChiralPak AS, 20%MeOH/CO_2 220 nm

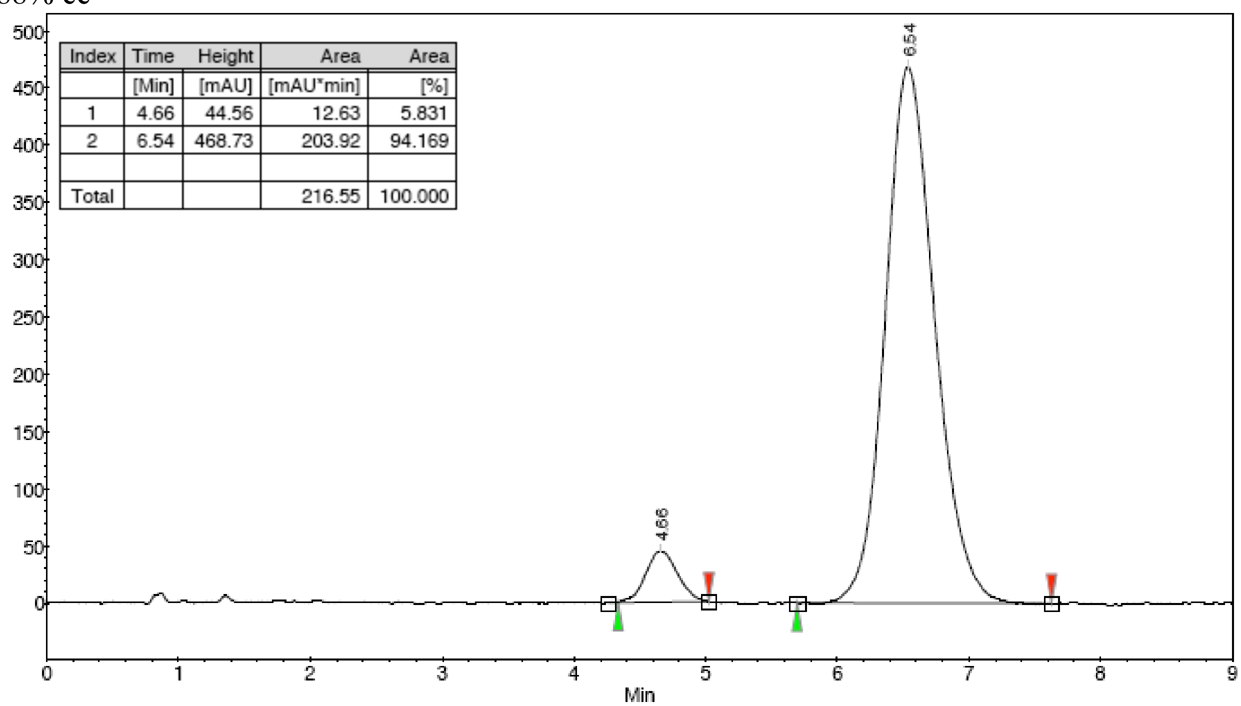




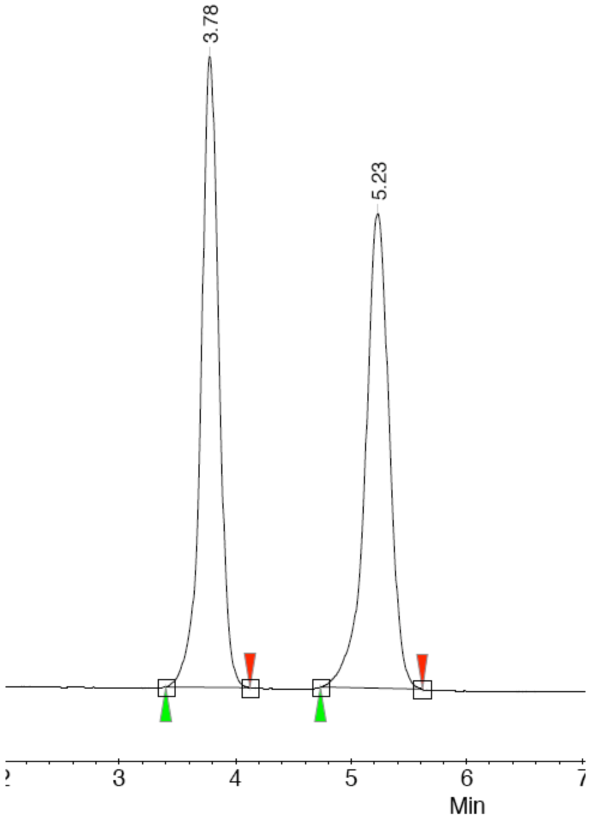
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	4.13	275.72	60.36	51.180
2	5.70	203.58	57.57	48.820
Total			117.93	100.000

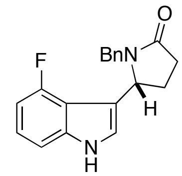
88% ee

Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	4.66	44.56	12.63	5.831
2	6.54	468.73	203.92	94.169
Total			216.55	100.000



Racemate: ChiralPak AS-H, 20%MeOH/CO_2 220 nm

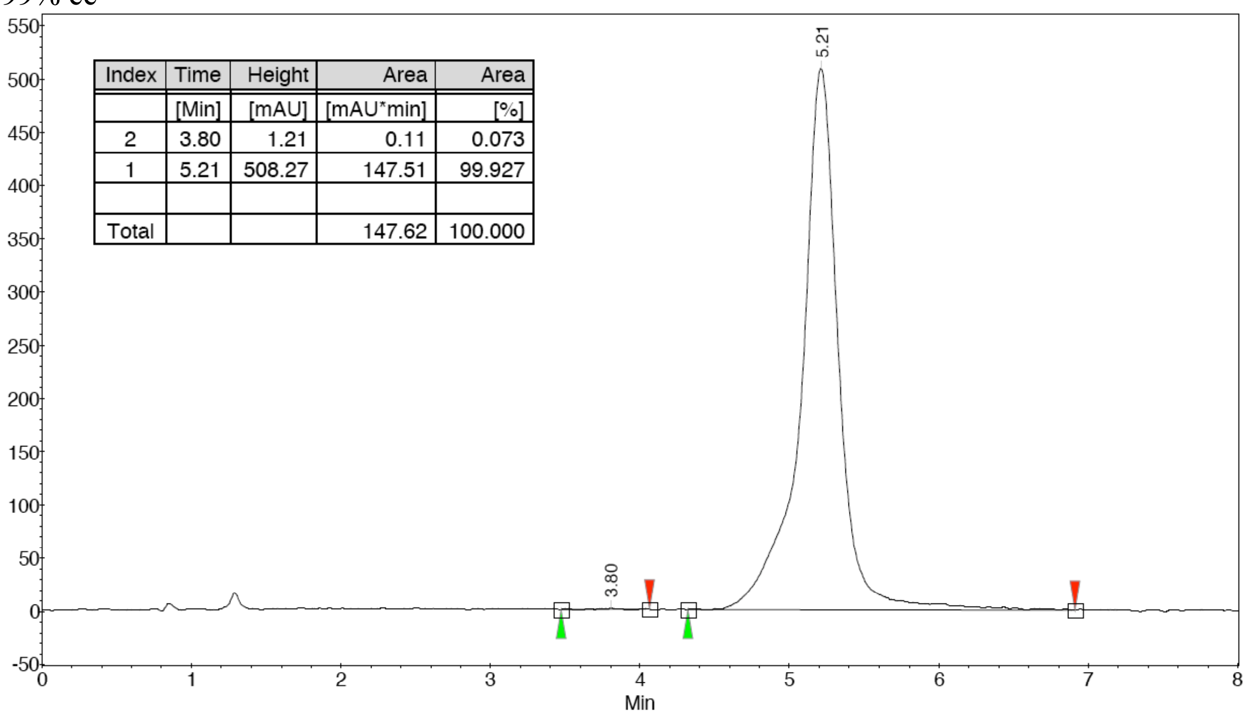




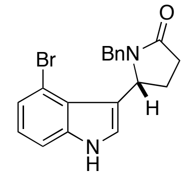
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	3.78	1126.95	202.03	49.857
2	5.23	847.52	203.20	50.143
Total			405.23	100.000

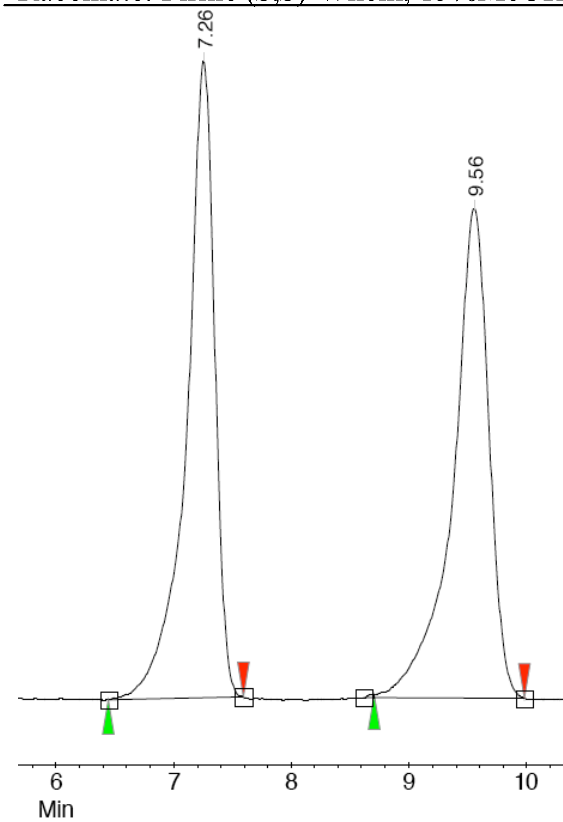
99% ee

Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
2	3.80	1.21	0.11	0.073
1	5.21	508.27	147.51	99.927
Total			147.62	100.000



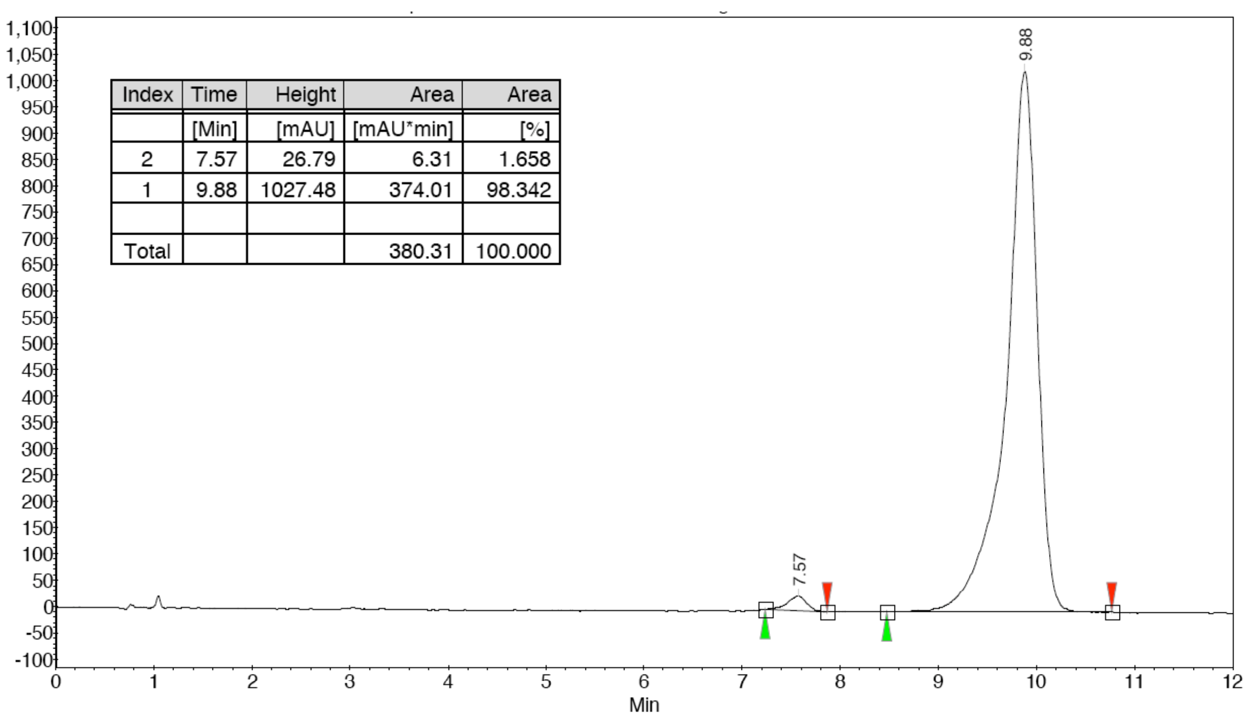
Racemate: Pirkle (*S*,*S*)-Whelk, 15%MeOH/CO_2 220 nm





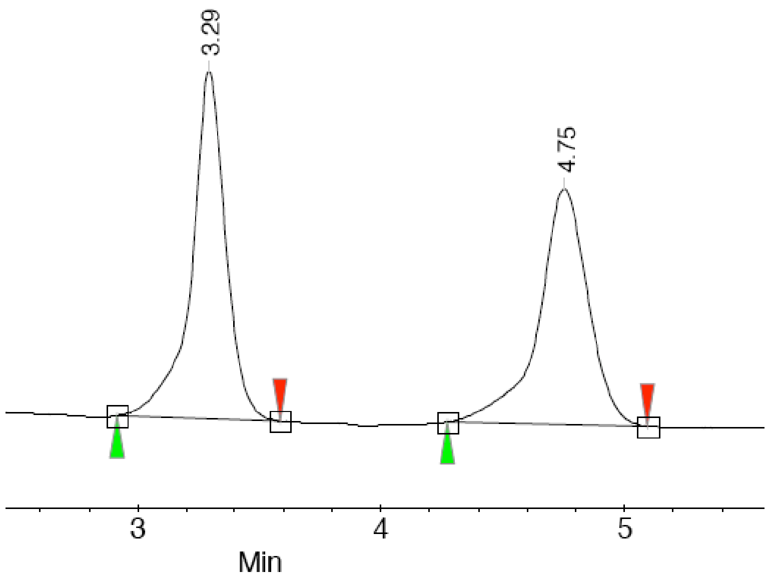
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	7.26	670.68	184.82	50.117
2	9.56	515.95	183.95	49.883
Total			368.77	100.000

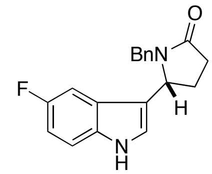
97% ee



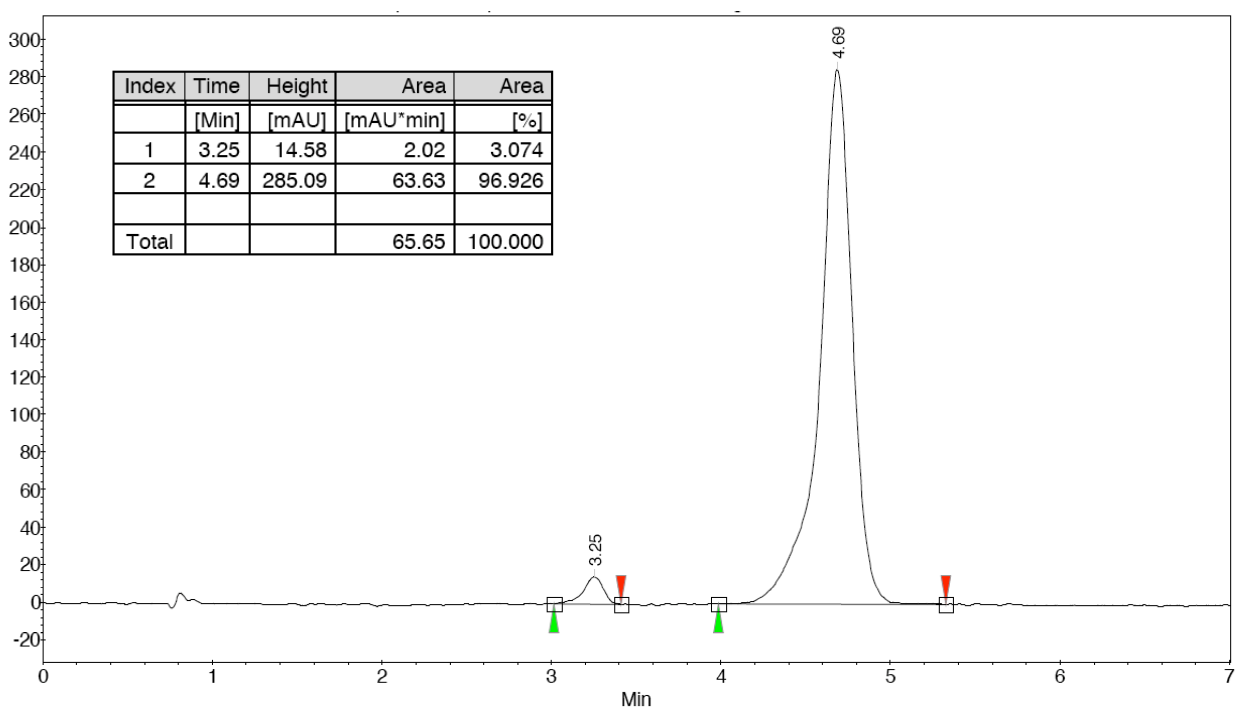
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
2	7.57	26.79	6.31	1.658
1	9.88	1027.48	374.01	98.342
Total			380.31	100.000

Racemate: ChiralPak AS-H, 20%MeOH/CO_2 220 nm



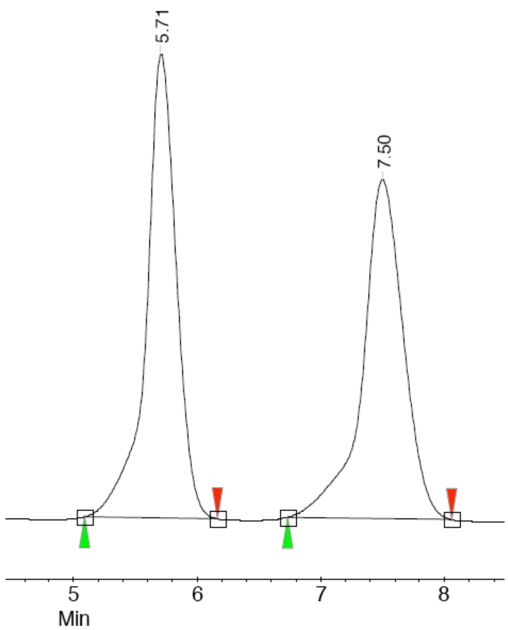


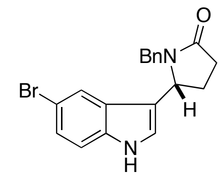
94% ee



Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	3.25	14.58	2.02	3.074
2	4.69	285.09	63.63	96.926
Total			65.65	100.000

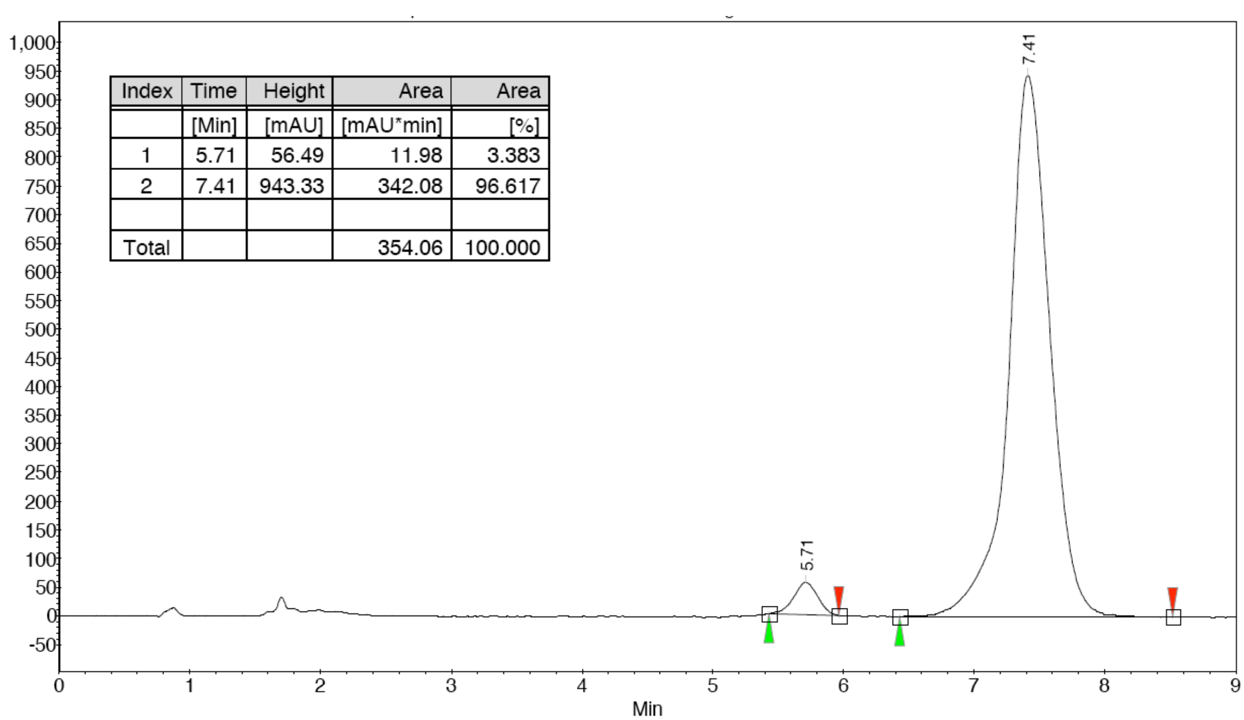
Racemate: ChiralPak AS-H, 20%MeOH/CO_2 220 nm





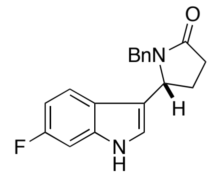
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	5.71	1195.10	337.53	50.195
2	7.50	872.85	334.91	49.805
Total			672.44	100.000

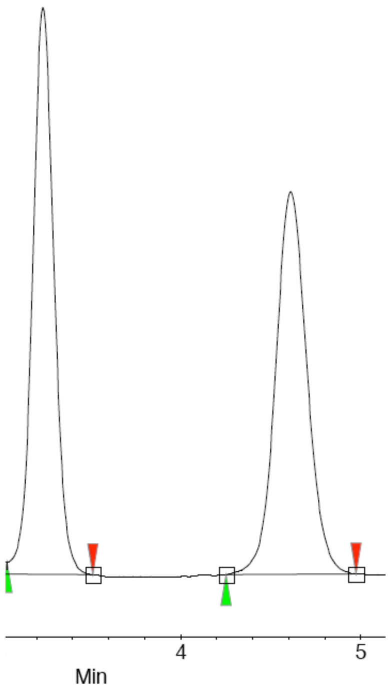
93% ee



Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	5.71	56.49	11.98	3.383
2	7.41	943.33	342.08	96.617
Total			354.06	100.000

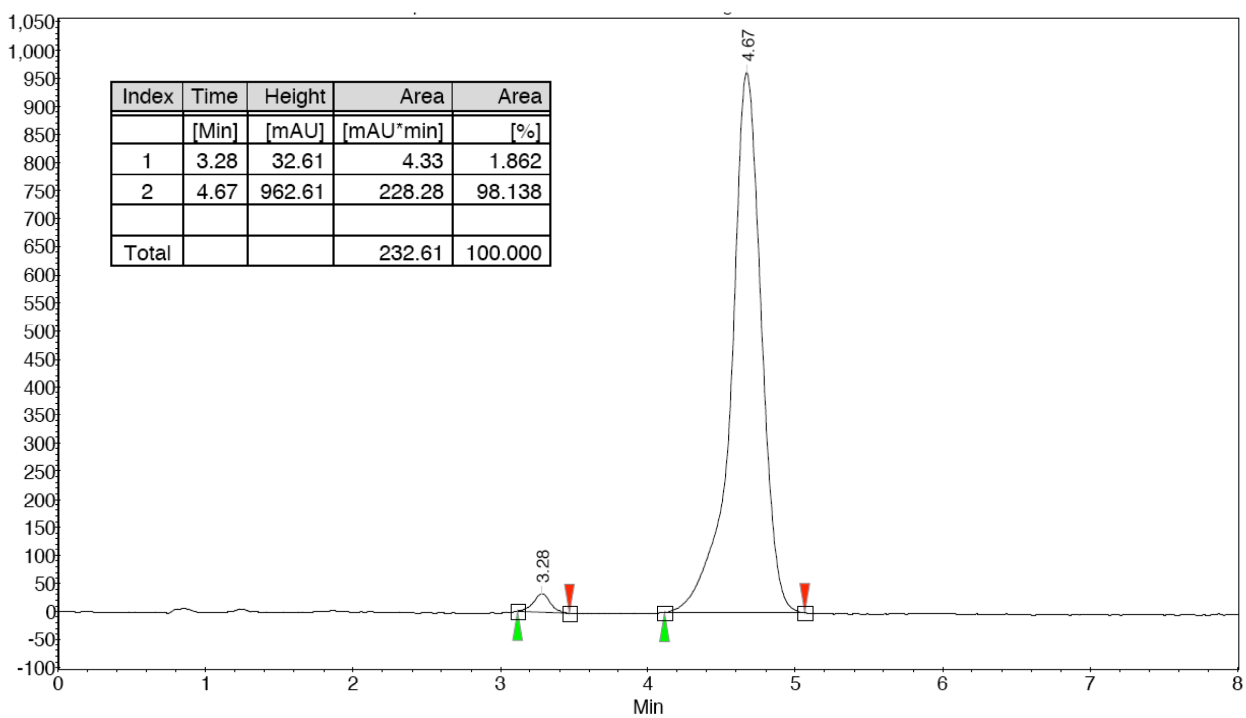
Racemate: ChiralPak AS-H, 20%MeOH/CO_2 220 nm





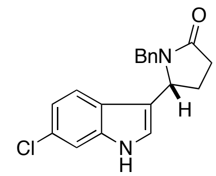
Time	End	RT Offset	Quantity	Height	Area	Area
[Min]	[Min]	[Min]	[% Area]	[μV]	[μV.Min]	[%]
3.23	3.51	0.00	50.48	515.2	71.5	50.478
4.61	4.97	0.00	49.52	347.8	70.2	49.522
			100.00	863.1	141.7	100.000

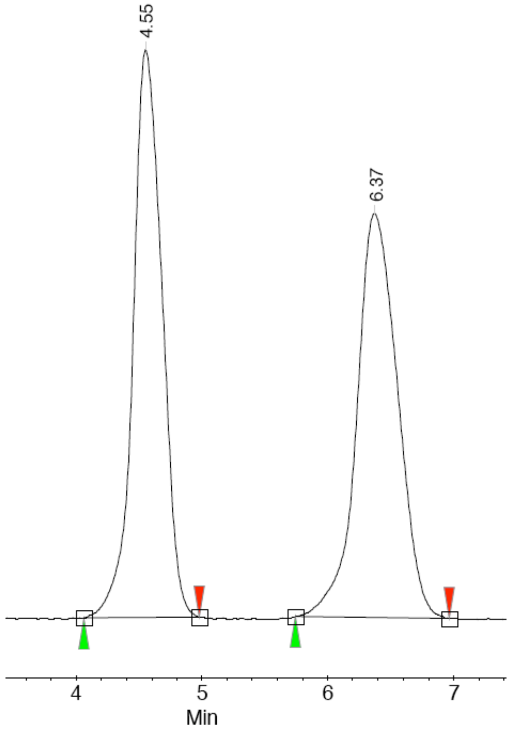
96% ee



Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	3.28	32.61	4.33	1.862
2	4.67	962.61	228.28	98.138
Total			232.61	100.000

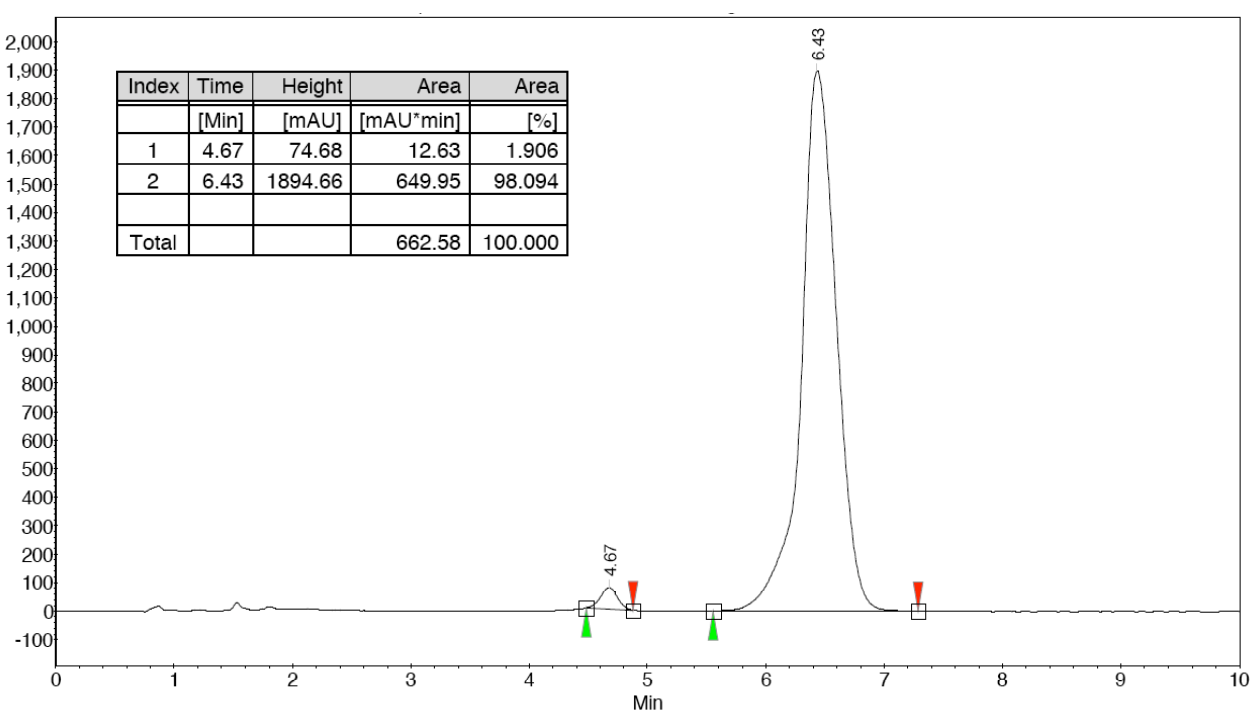
Racemate: ChiralPak AS-H, 20%MeOH/CO_2 220 nm





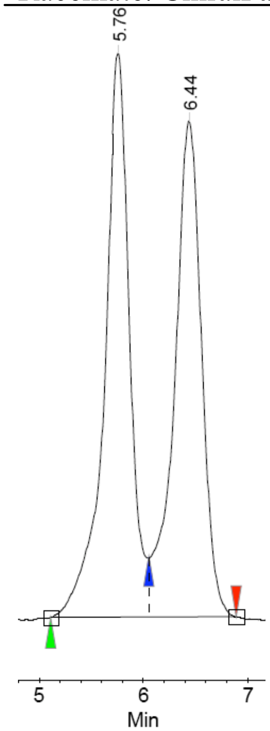
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	4.55	549.55	147.39	50.064
2	6.37	391.19	147.01	49.936
Total			294.41	100.000

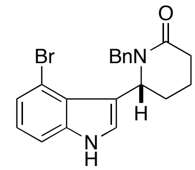
96% ee



Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	4.67	74.68	12.63	1.906
2	6.43	1894.66	649.95	98.094
Total			662.58	100.000

Racemate: ChiralPak AS-H, 20%MeOH/CO_2 220 nm

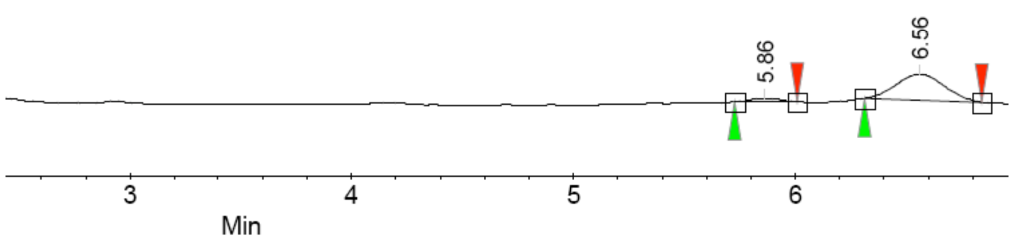




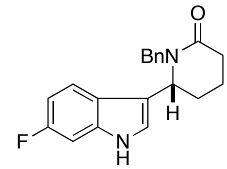
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	5.76	257.49	72.51	51.483
2	6.44	226.50	68.33	48.517
Total			140.85	100.000

85%ee

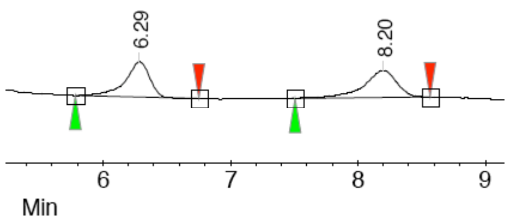
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
2	5.86	10.46	1.63	7.644
1	6.56	78.67	19.71	92.356
Total			21.34	100.000



Racemate: Pirkle (*S*,*S*)-Whelk, 15%MeOH/CO_2 220 nm

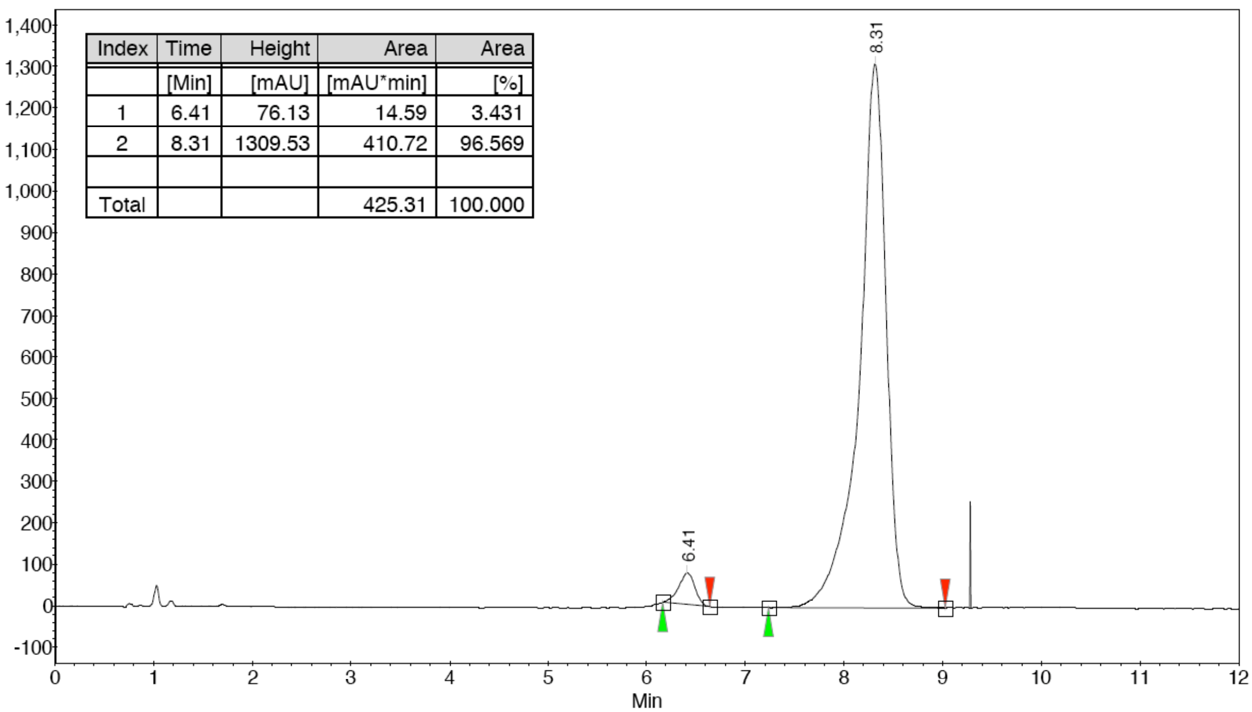


Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
2	6.29	85.06	19.04	49.103
1	8.20	65.71	19.73	50.897
Total			38.77	100.000

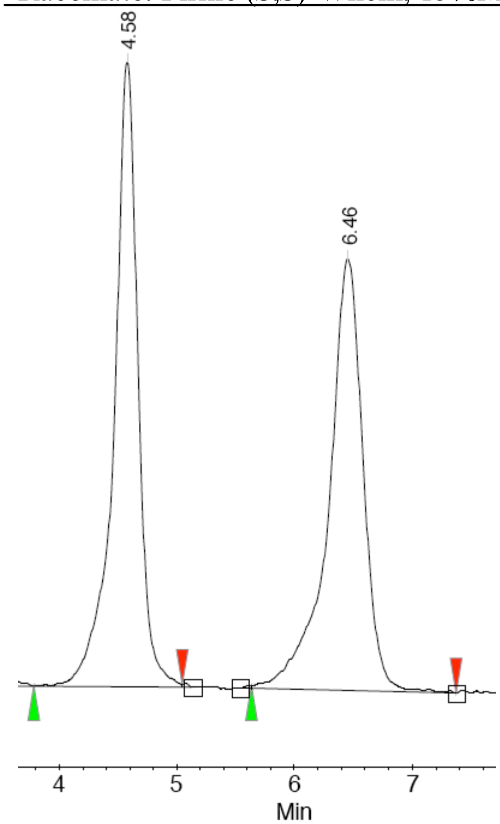


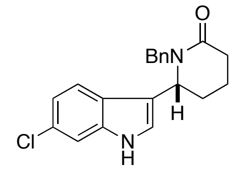
96% ee

Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	6.41	76.13	14.59	3.431
2	8.31	1309.53	410.72	96.569
Total			425.31	100.000



Racemate: Pirkle (*S*,*S*)-Whelk, 15%MeOH/CO_2 220 nm

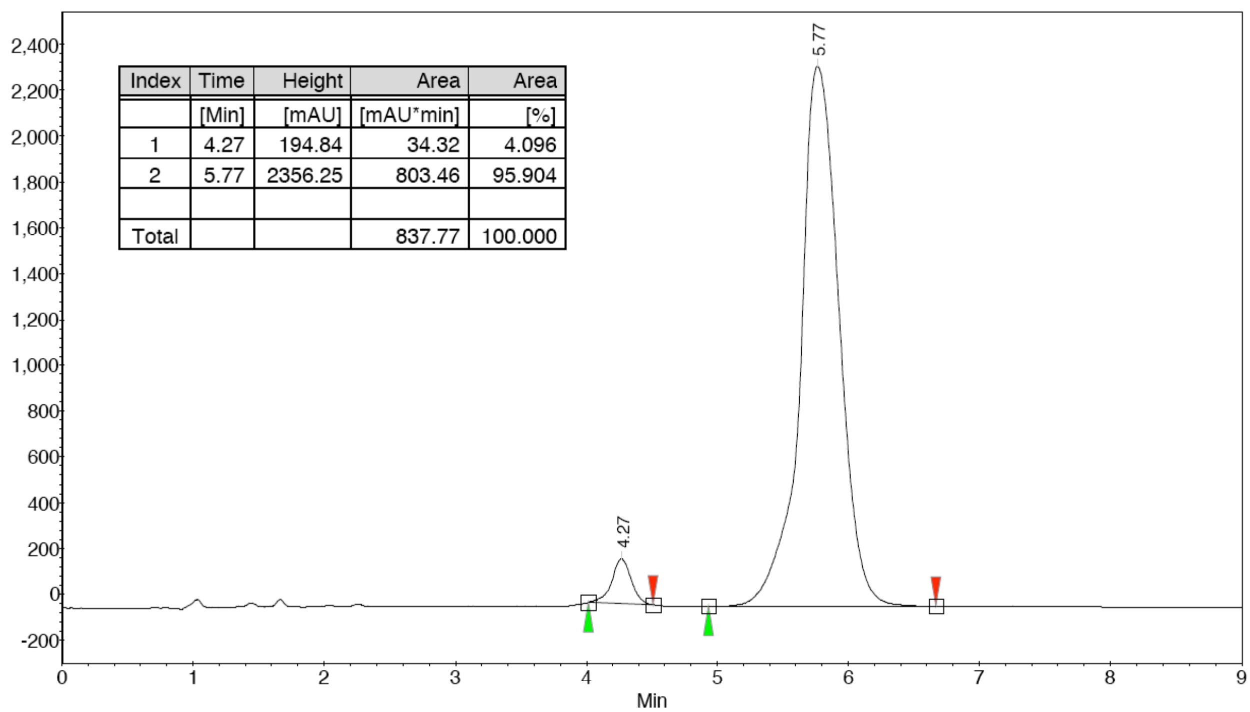




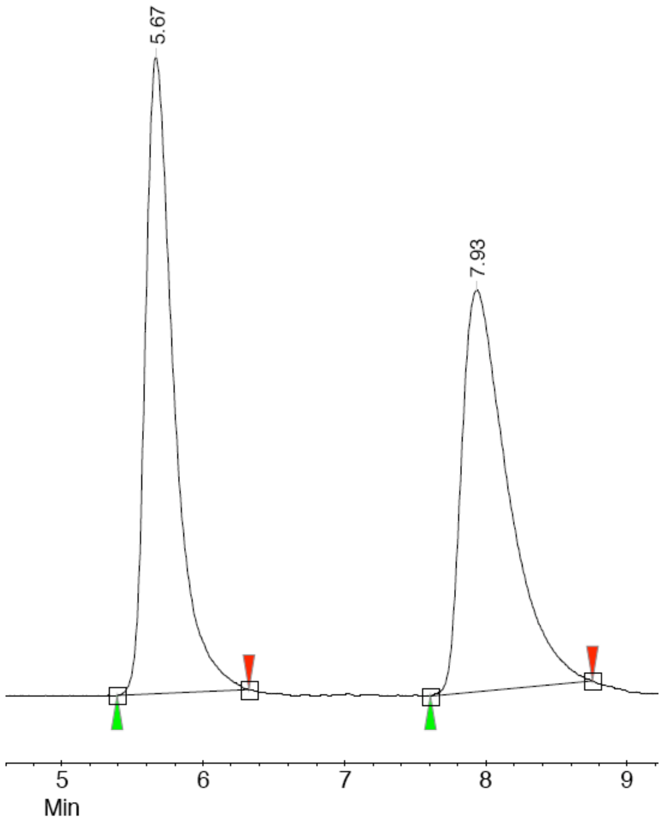
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	4.58	237.05	56.28	50.147
2	6.46	164.10	55.95	49.853
Total			112.22	100.000

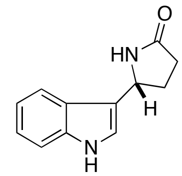
92% ee

Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	4.27	194.84	34.32	4.096
2	5.77	2356.25	803.46	95.904
Total			837.77	100.000



Racemate: ChiralPak AD-H, 20%MeOH/CO_2 220 nm

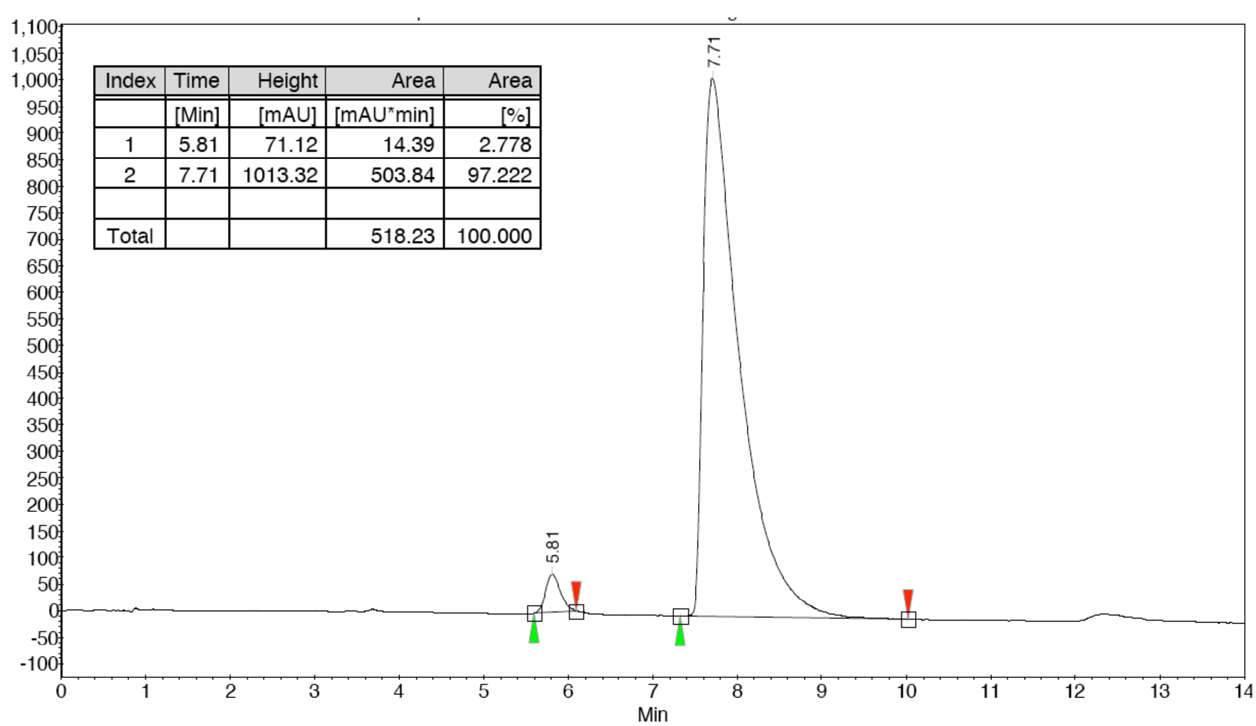




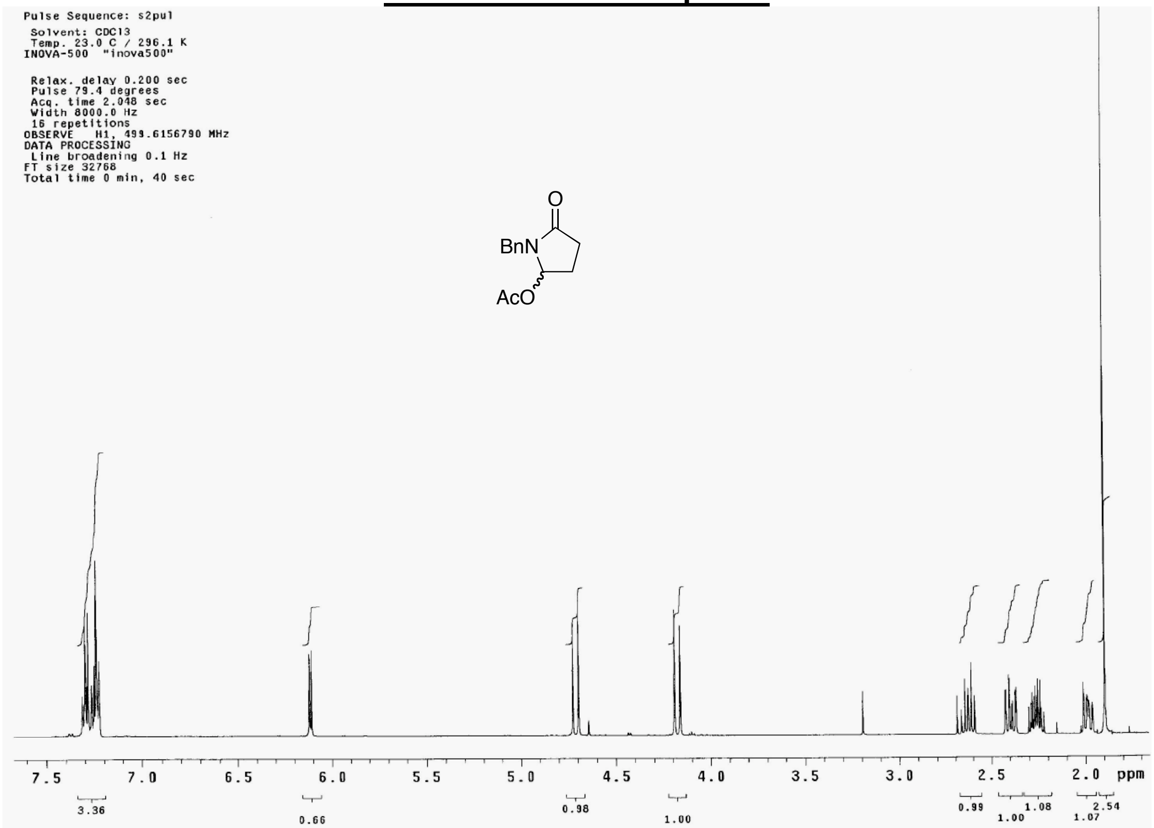
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	5.67	525.08	123.88	49.595
2	7.93	331.23	125.90	50.405
Total			249.78	100.000

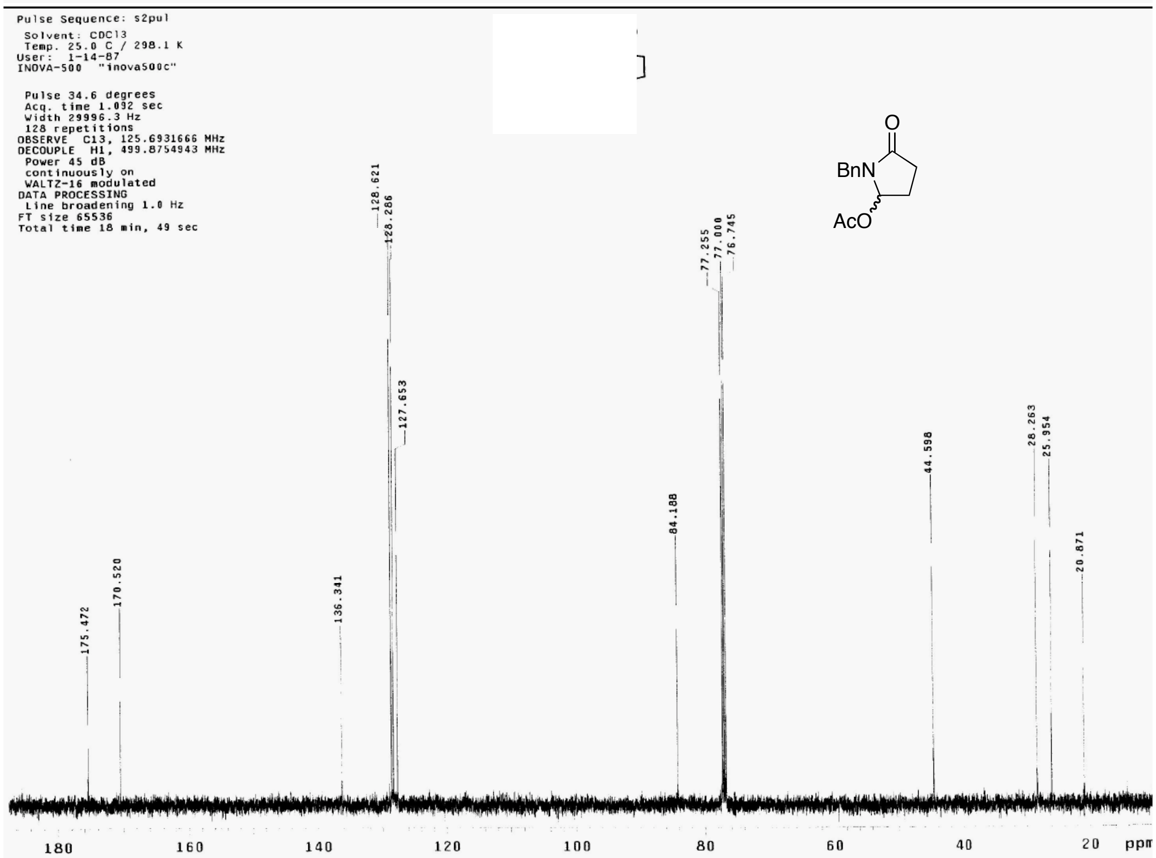
94% ee

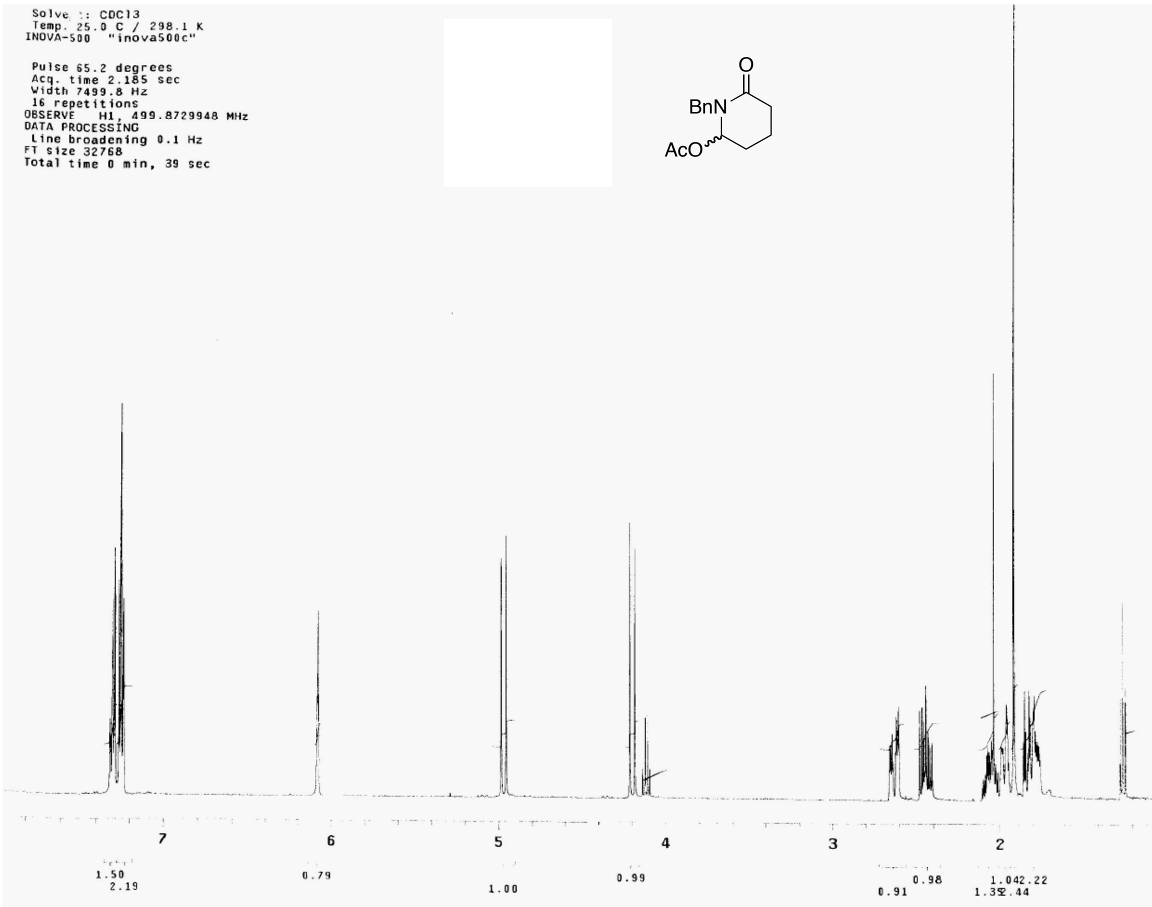
Index	Time	Height	Area	Area
	[Min]	[mAU]	[mAU*min]	[%]
1	5.81	71.12	14.39	2.778
2	7.71	1013.32	503.84	97.222
Total			518.23	100.000

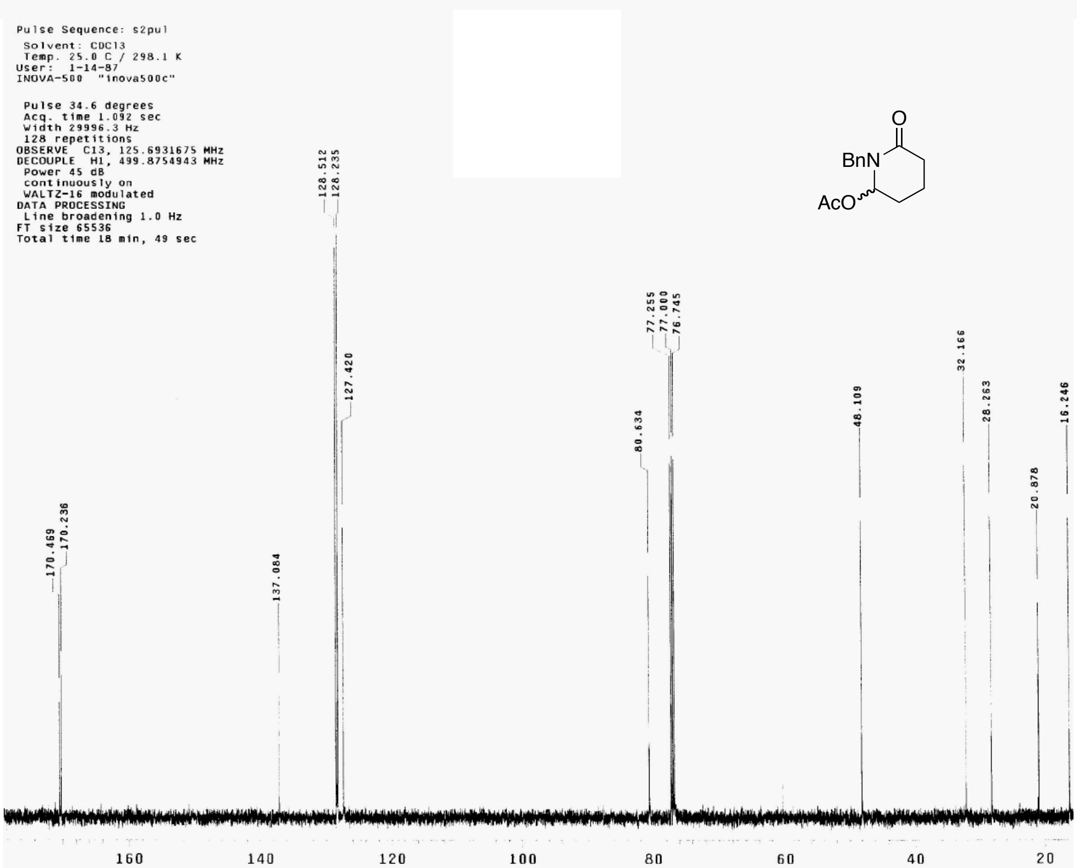


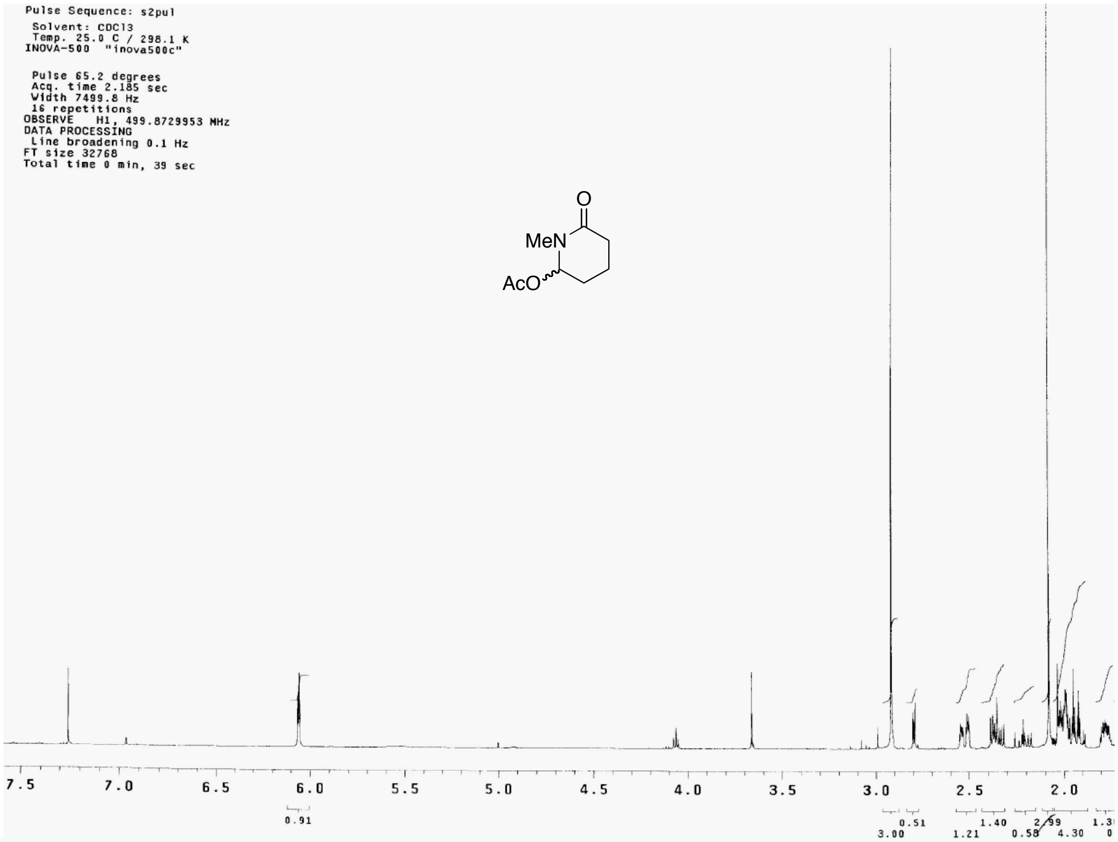
G. ^{1}H and ^{13}C NMR Spectra

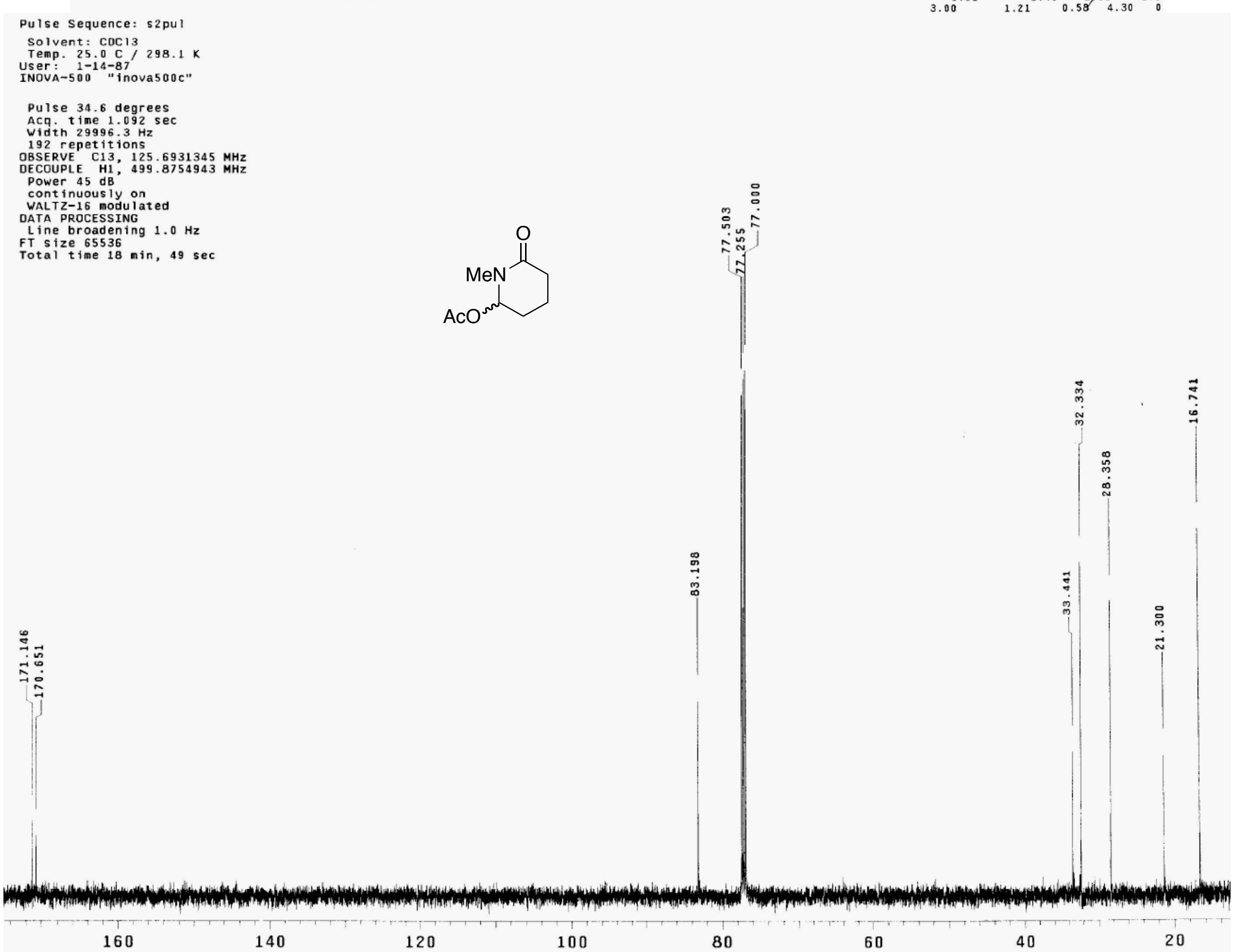


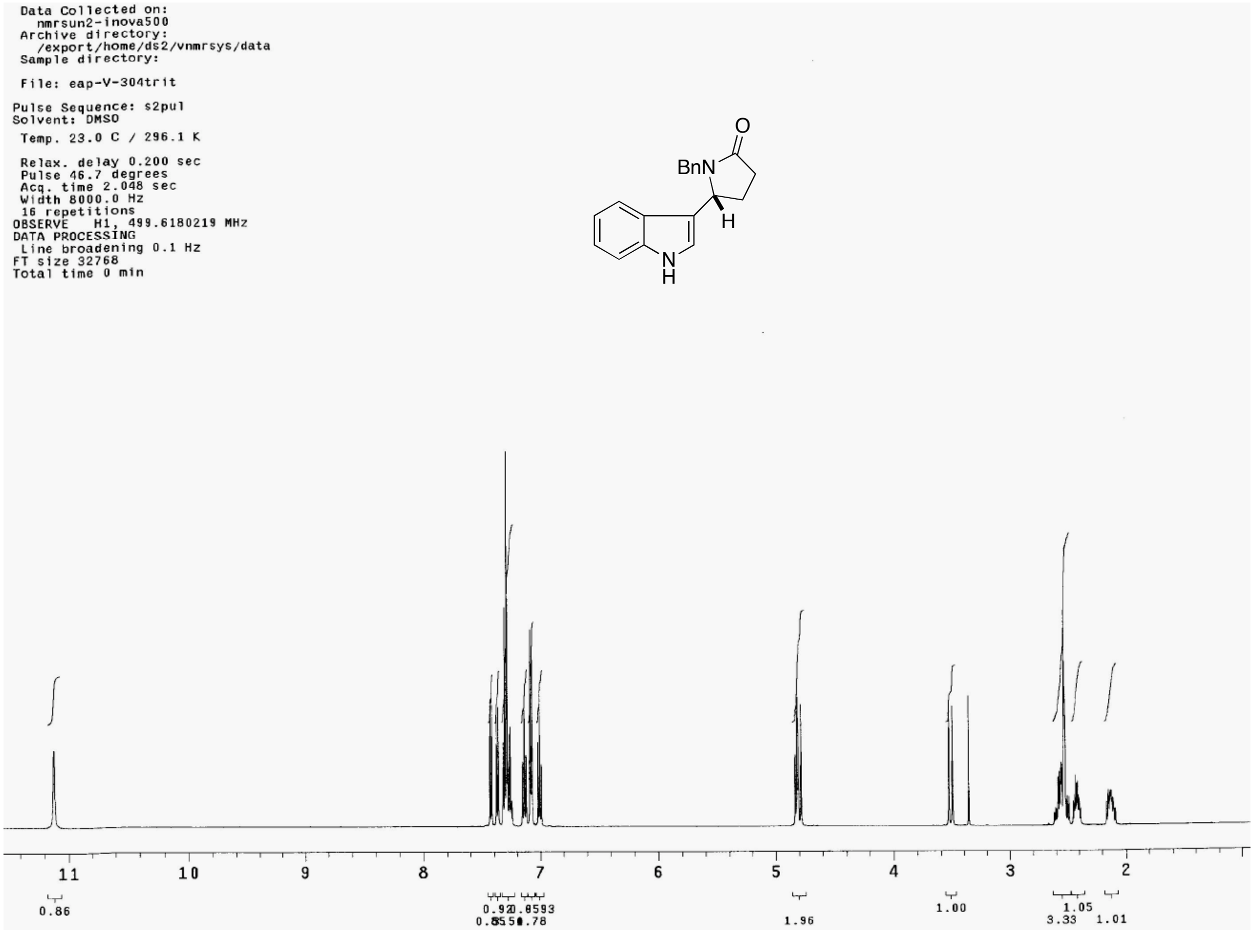


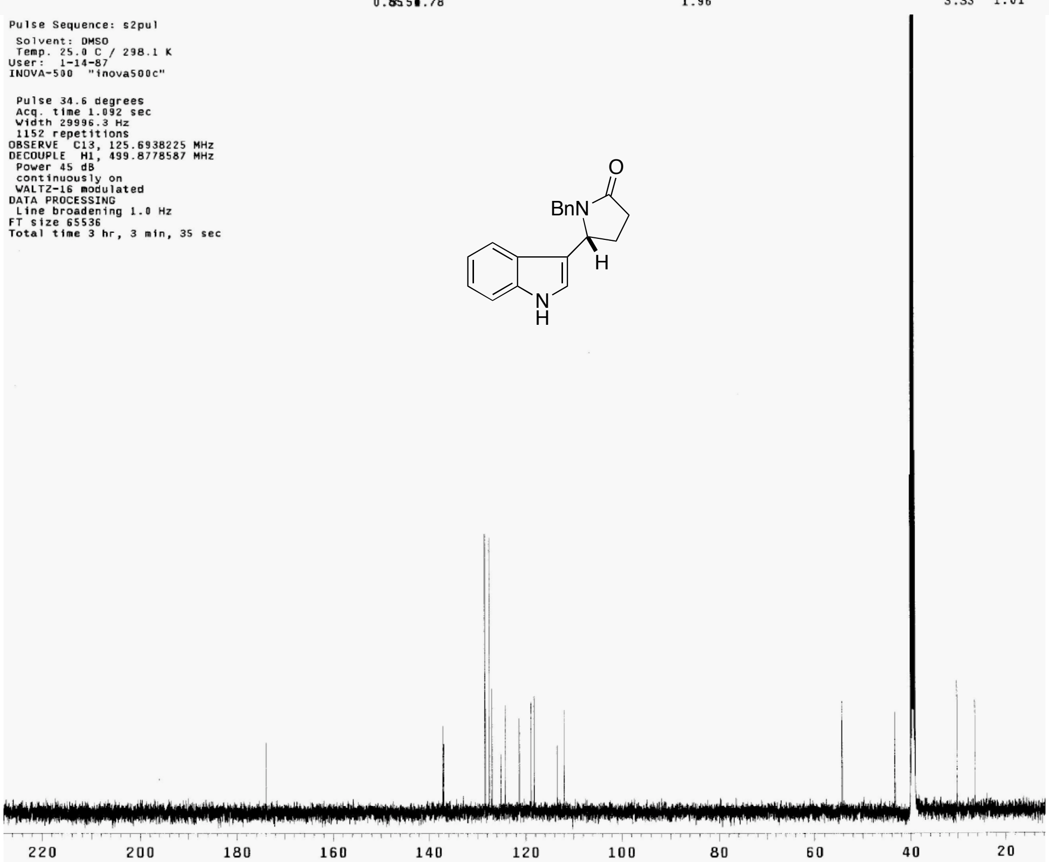

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
BnN
O
AcO
7
6
5
4
3
2
1.50
2.19
0.79
1.00
0.99
0.91
0.98
1.04
2.22
2.44

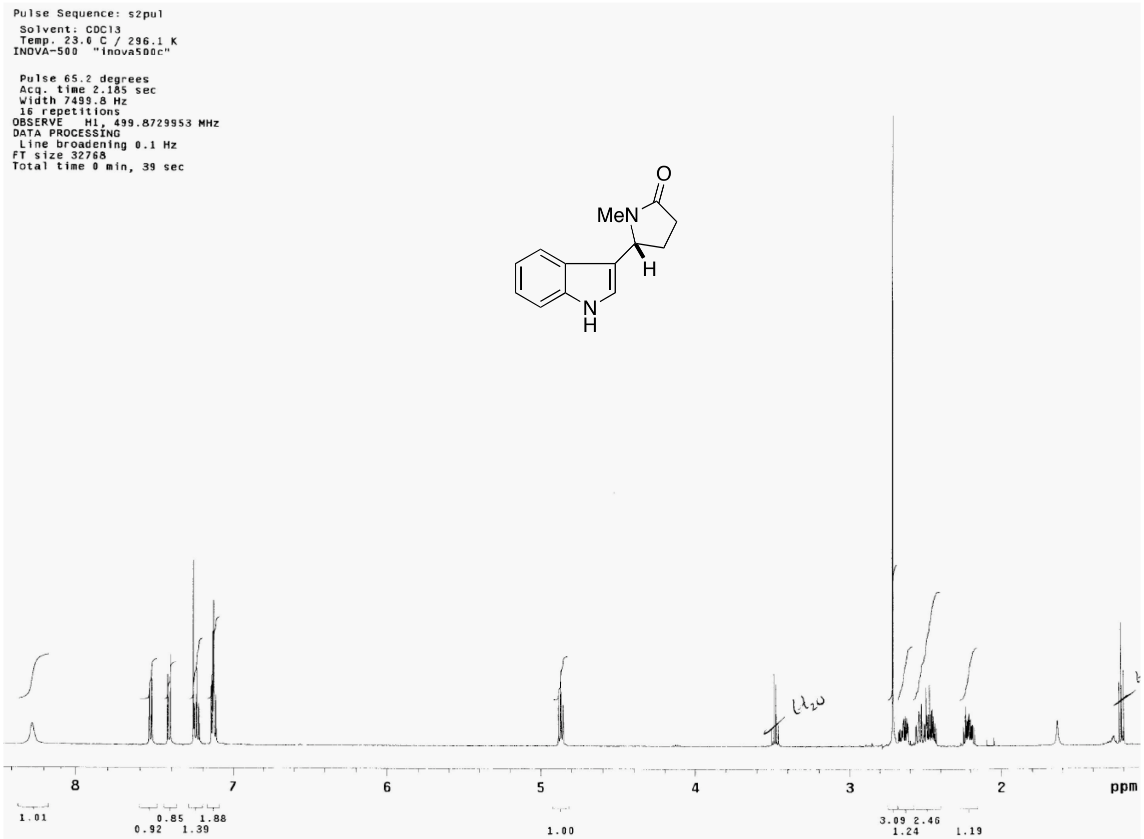

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
128 repetitions
OBSERVE C13, 125.6931675 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
BnN
O
AcO
170.469
170.236
137.084
128.512
128.235
127.420
80.634
77.255
77.000
76.745
48.109
32.166
28.263
20.878
16.246
160
140
120
100
80
60
40
20

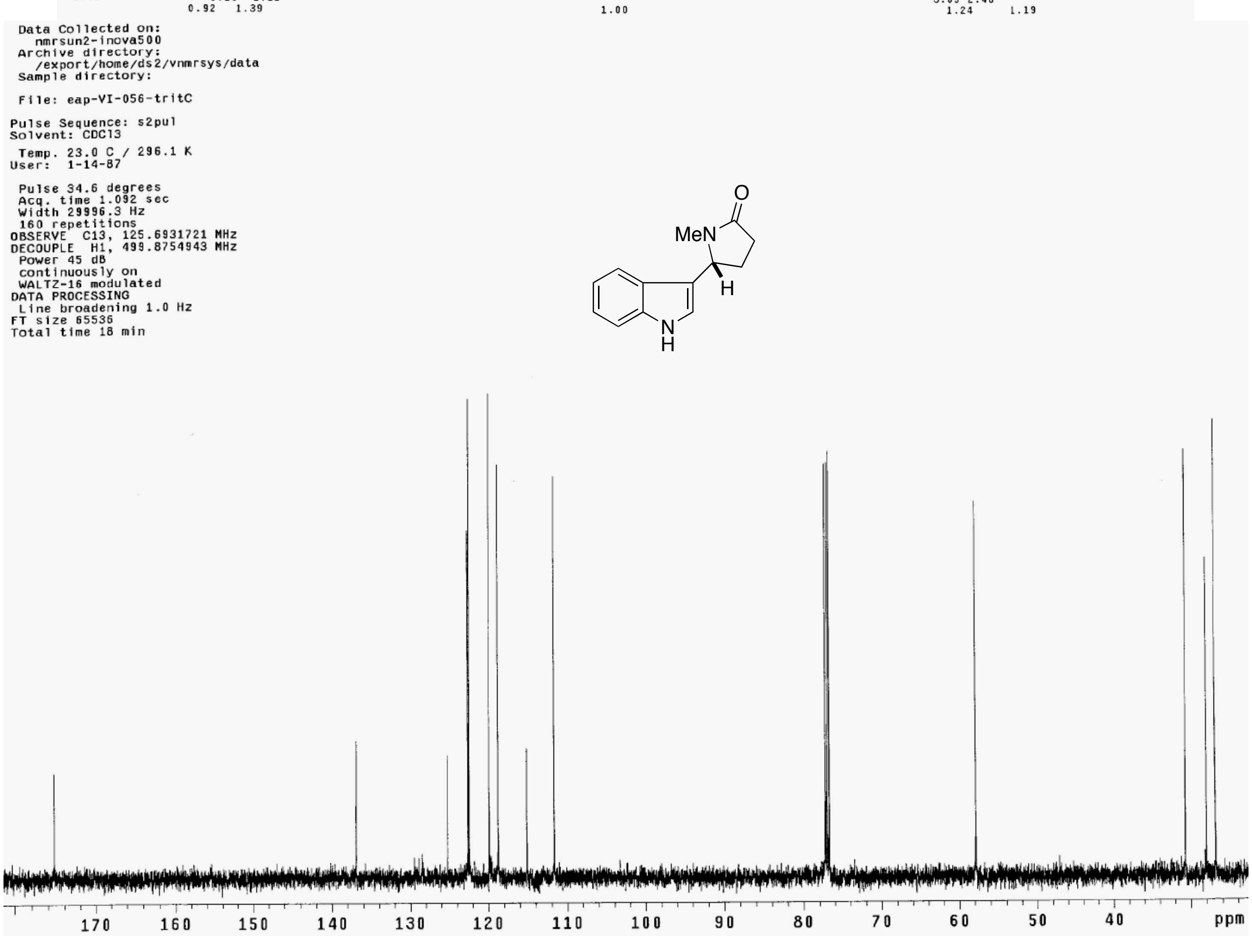

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729953 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
O
MeN
AcO
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0
0.91 3.00 0.51 1.21 1.40 0.58 2.99 4.30

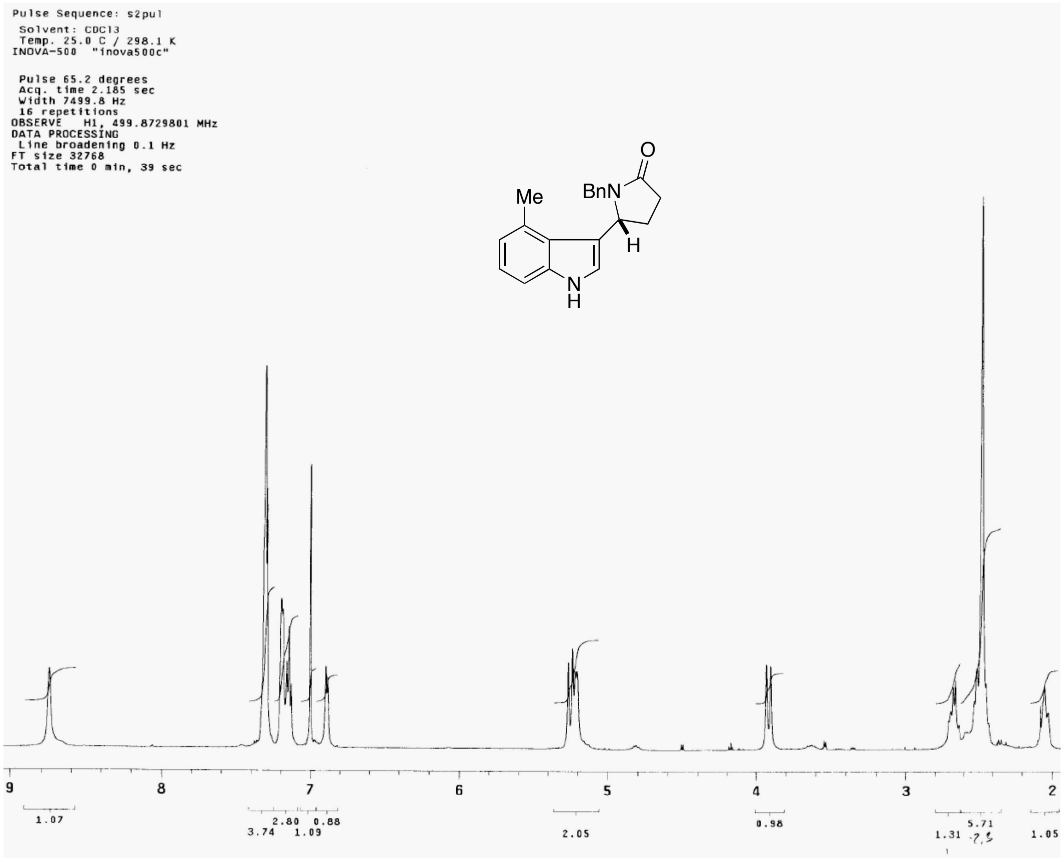

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
192 repetitions
OBSERVE C13, 125.6931345 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
O
MeN
AcO
171.146
170.651
83.198
77.503
77.255
77.000
33.441
32.334
28.358
21.300
16.741
160 140 120 100 80 60 40 20

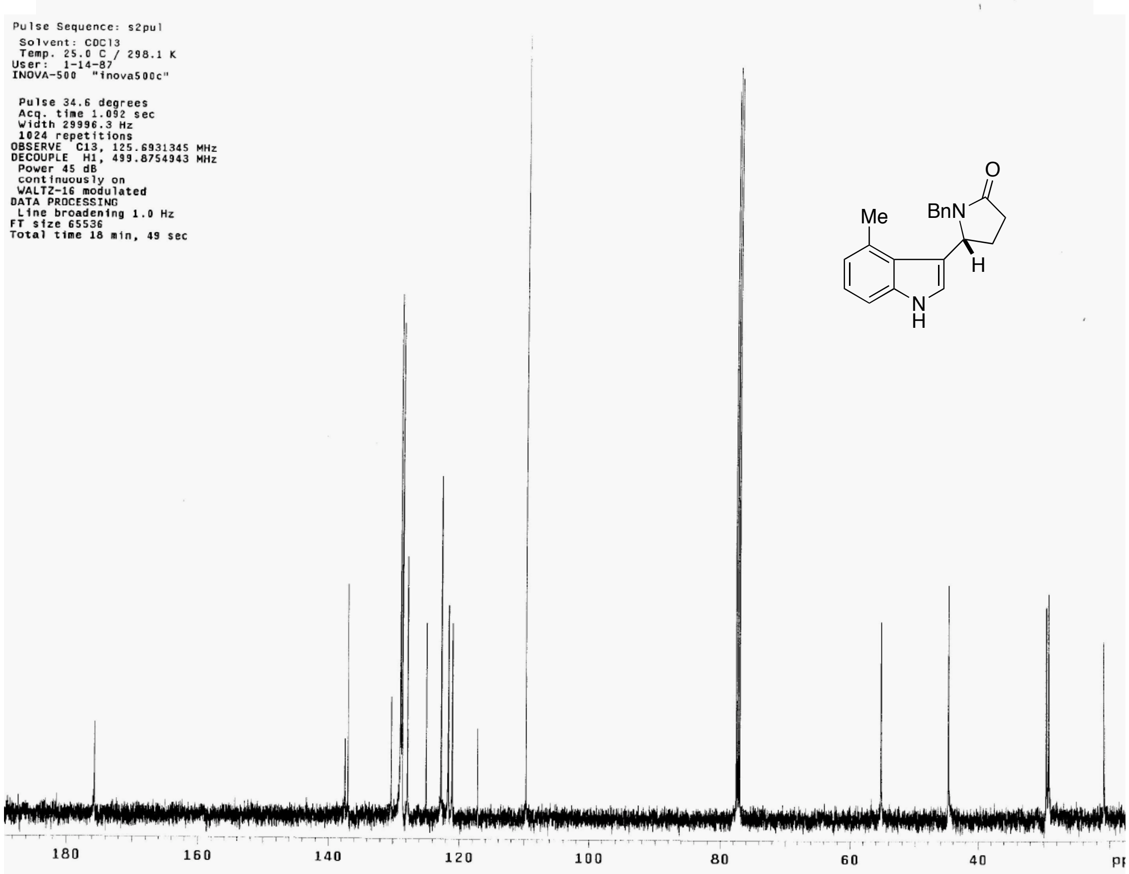

Data Collected on:
nmrsun2-inova500
Archive directory:
/export/home/ds2/vnmrsys/data
Sample directory:
File: eap-V-304trit
Pulse Sequence: s2pul
Solvent: DMSO
Temp. 23.0 C / 296.1 K
Relax. delay 0.200 sec
Pulse 46.7 degrees
Acq. time 2.048 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.6180219 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min
BnN
O
H
N
H
11
10
9
8
7
6
5
4
3
2
0.86
1.96
1.00
1.05
3.33
1.01

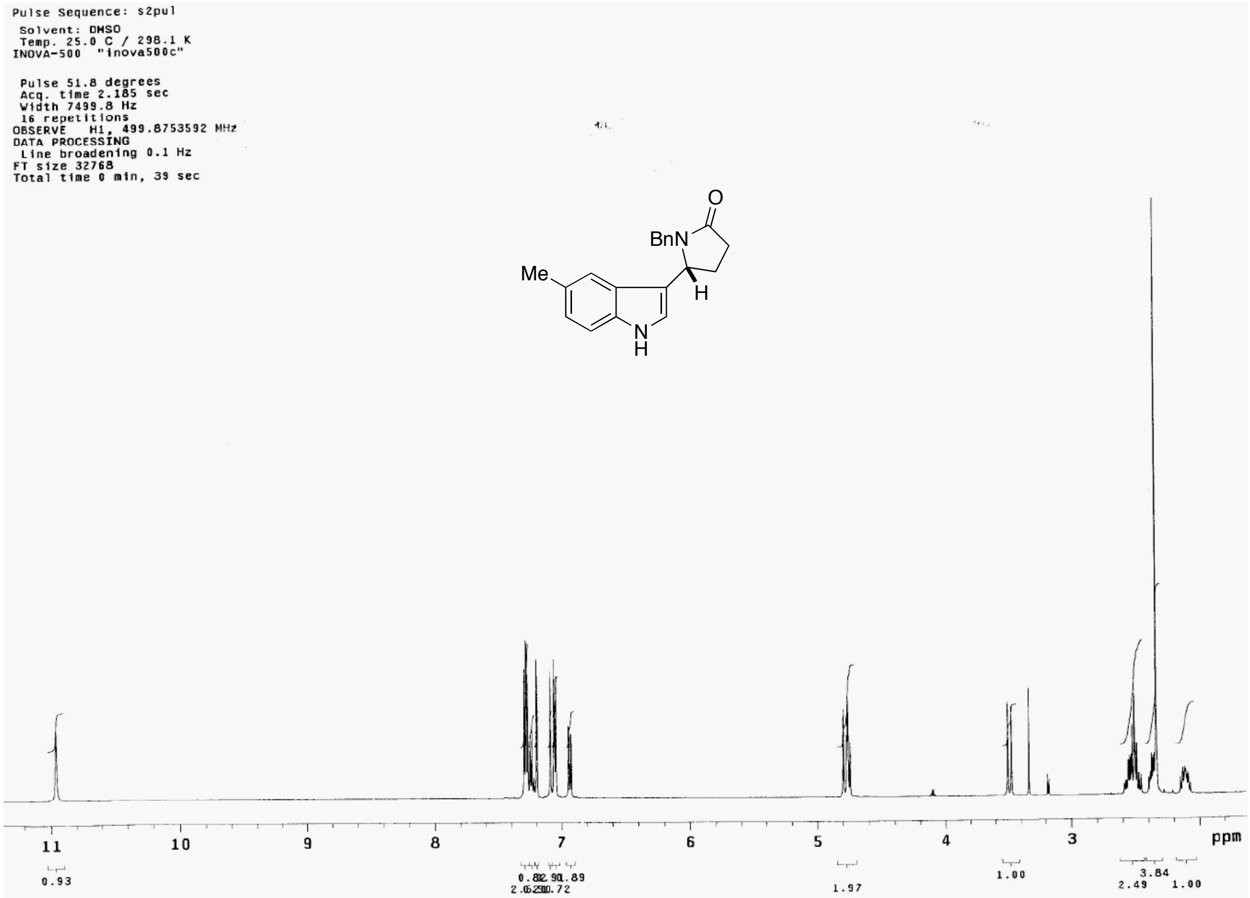

Pulse Sequence: s2pul
Solvent: DMSO
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
1152 repetitions
OBSERVE C13, 125.6938225 MHz
DECOUPLE H1, 499.8778587 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 3 hr, 3 min, 35 sec
BnN
O
H
N
H
220
200
180
160
140
120
100
80
60
40
20

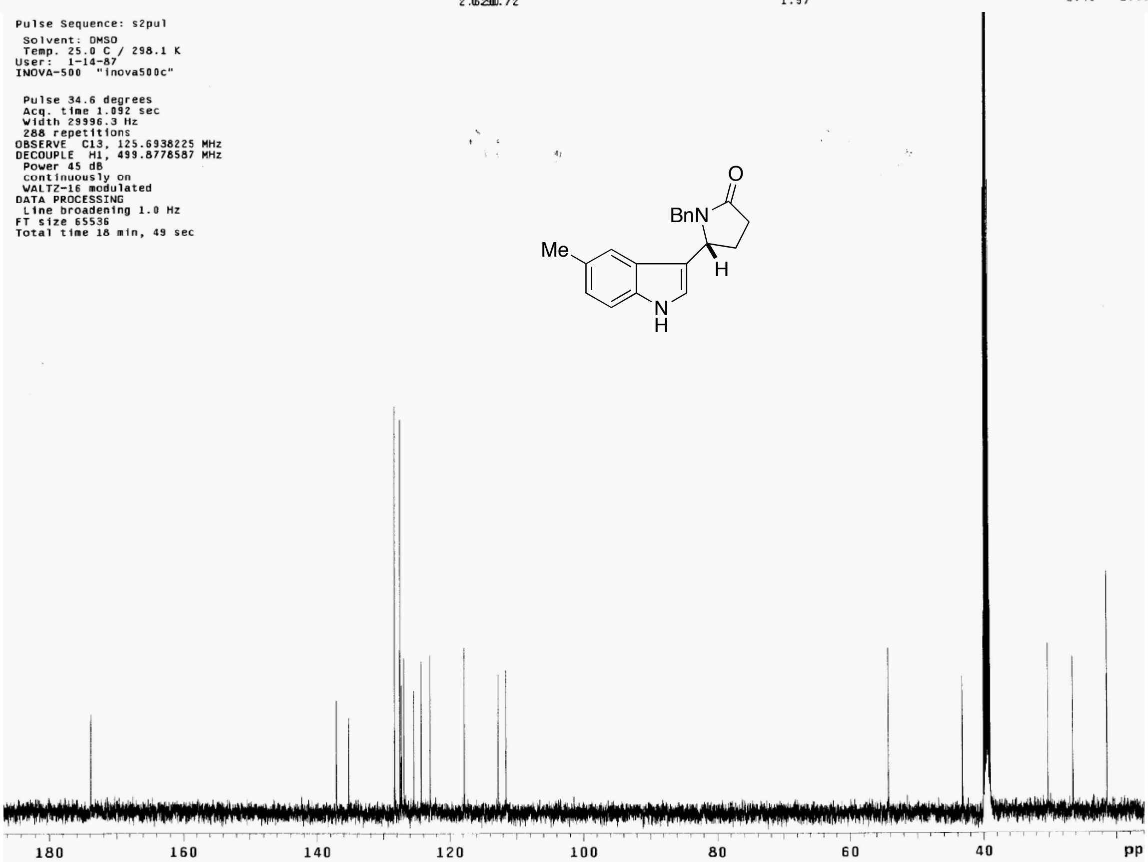

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 23.0 C / 296.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729953 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
MeN
O
H
N
H
ppm

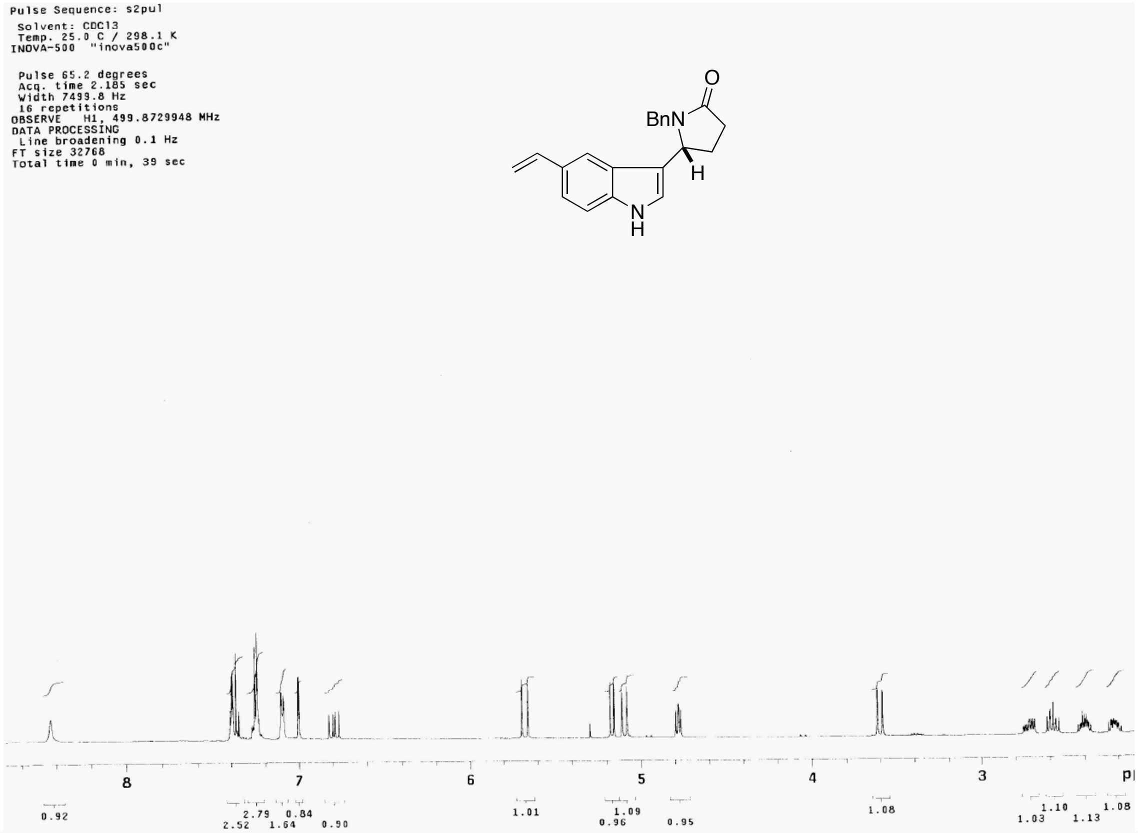

Data Collected on:
nmrsun2-inova500
Archive directory:
/export/home/ds2/vnmrsys/data
Sample directory:
File: eap-VI-056-tritC
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 23.0 C / 296.1 K
User: 1-14-87
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
160 repetitions
OBSERVE C13, 125.6931721 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min
MeN
O
H
N
H
ppm

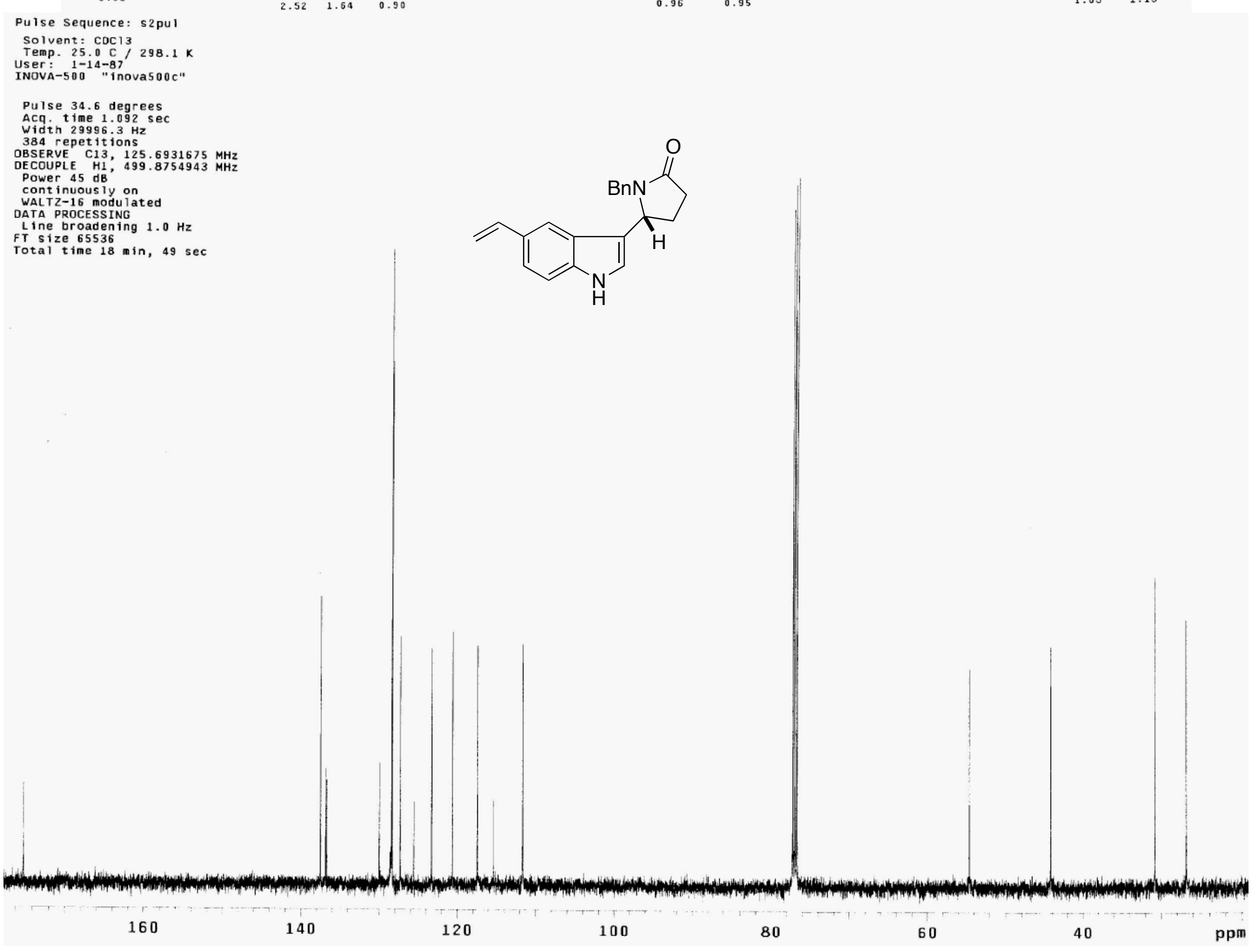

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729801 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
Me
BnN
O
H
N
H
9
8
7
6
5
4
3
2
1.07
2.80 0.88
3.74 1.09
2.05
0.98
5.71
1.31
1.05

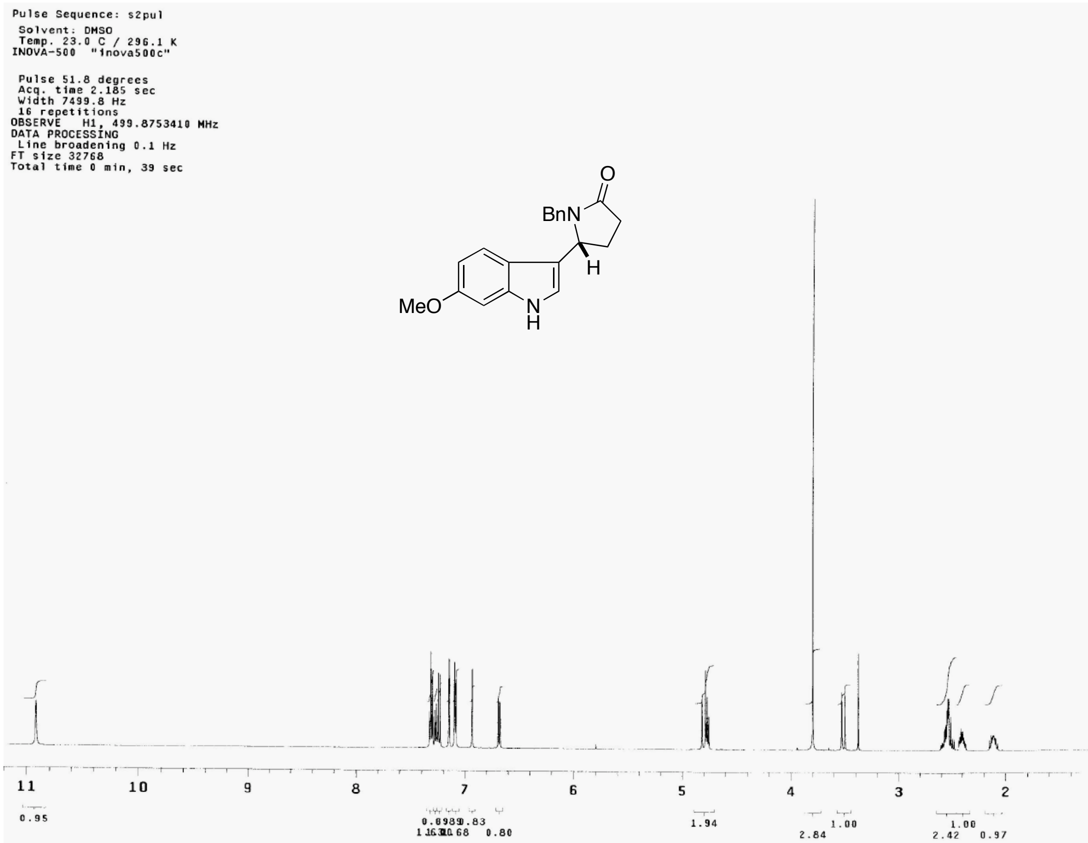

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
1024 repetitions
OBSERVE C13, 125.6931345 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
Me
BnN
O
H
N
H
180
160
140
120
100
80
60
40

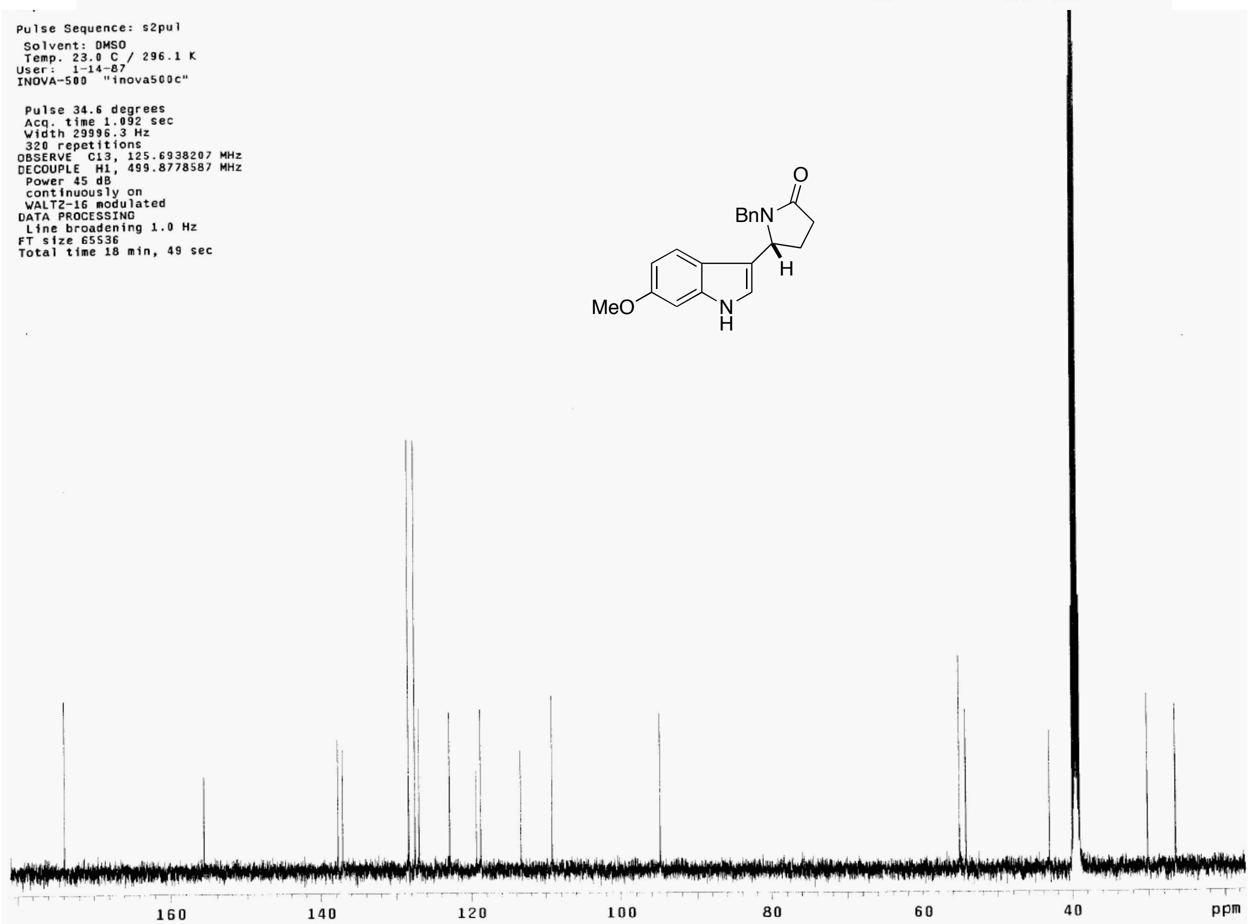

Pulse Sequence: s2pul
Solvent: DMSO
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 51.8 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8753592 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
BnN
O
Me
H
N
H
ppm

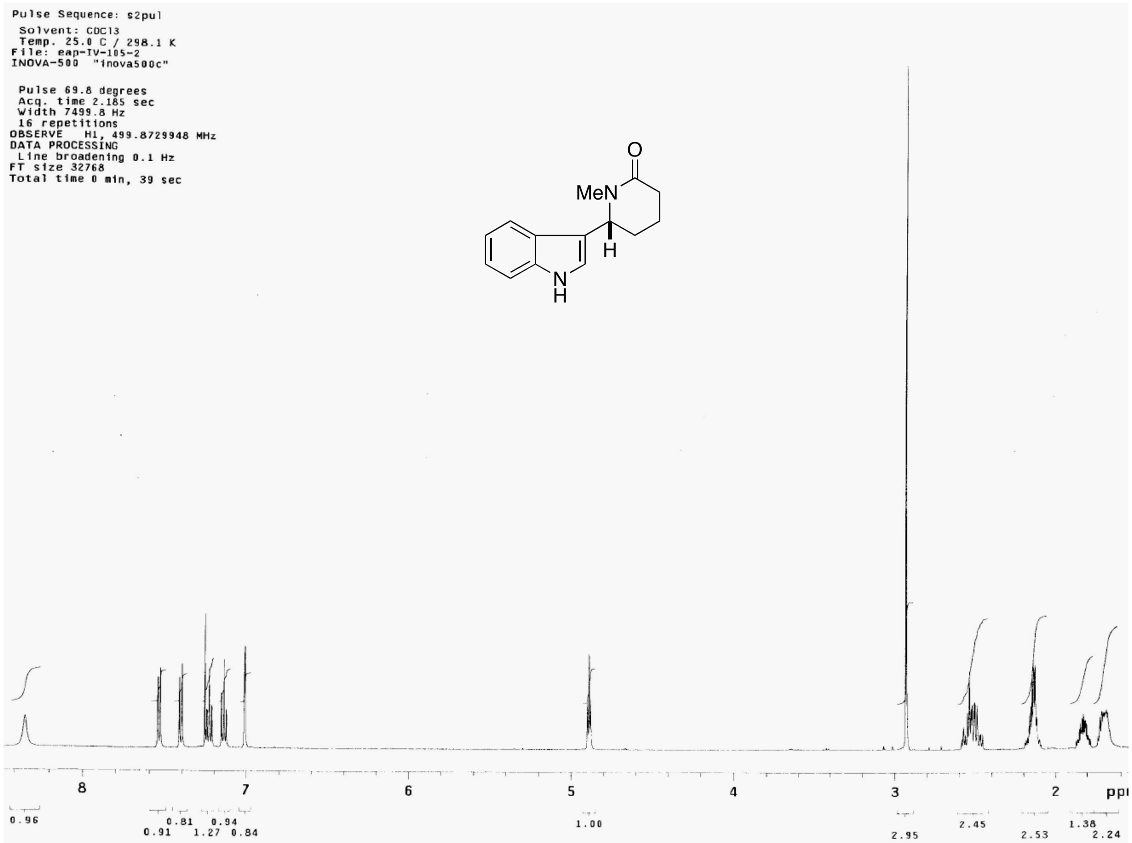

Pulse Sequence: s2pul
Solvent: DMSO
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
288 repetitions
OBSERVE C13, 125.6938225 MHz
DECOUPLE H1, 499.8778587 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
BnN
O
Me
H
N
H

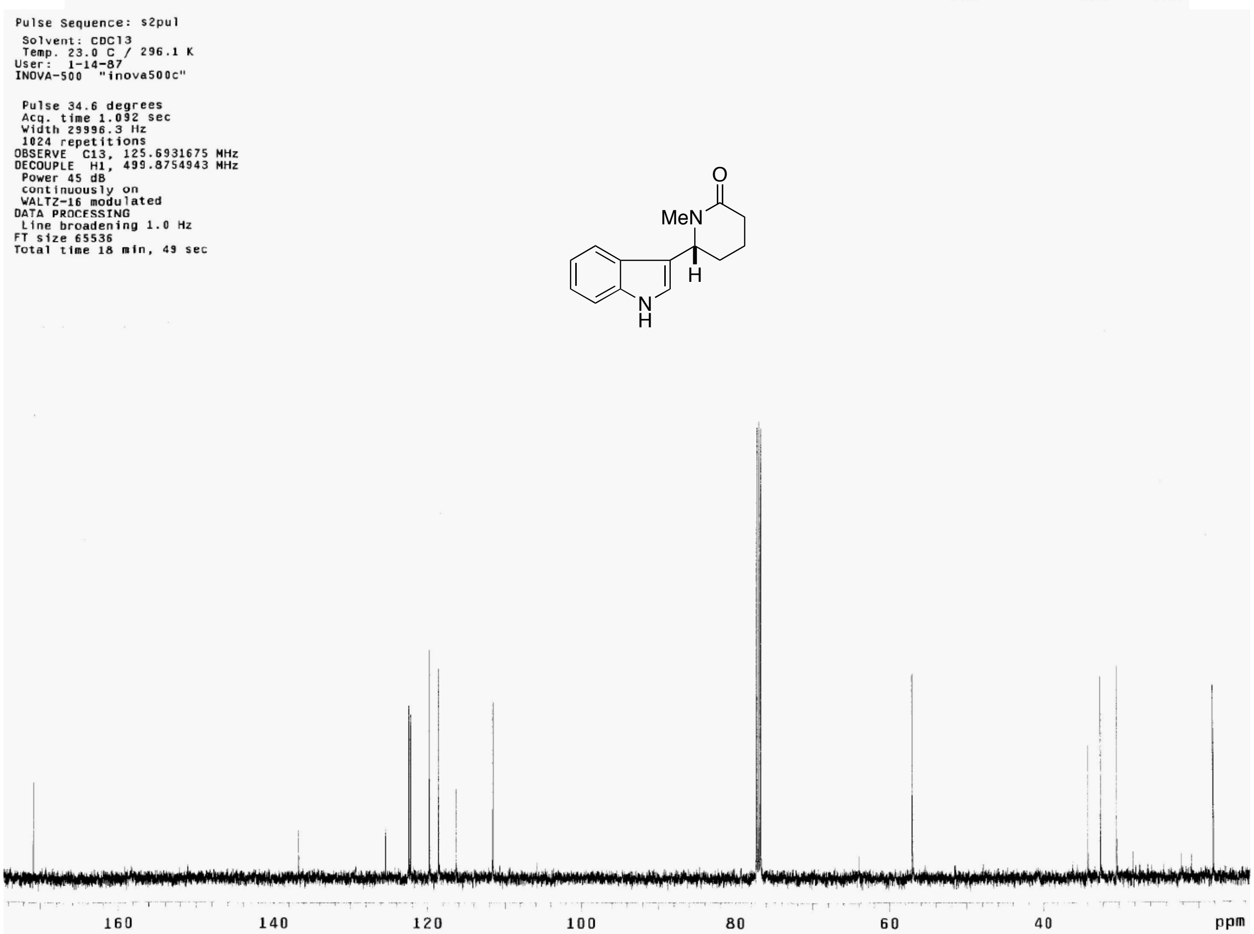

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
O
BnN
H
N
H
8
7
6
5
4
3
0.92
2.79 0.84
2.52 1.64 0.90
1.01
1.09
0.96
0.95
1.08
1.10 1.08
1.03 1.13

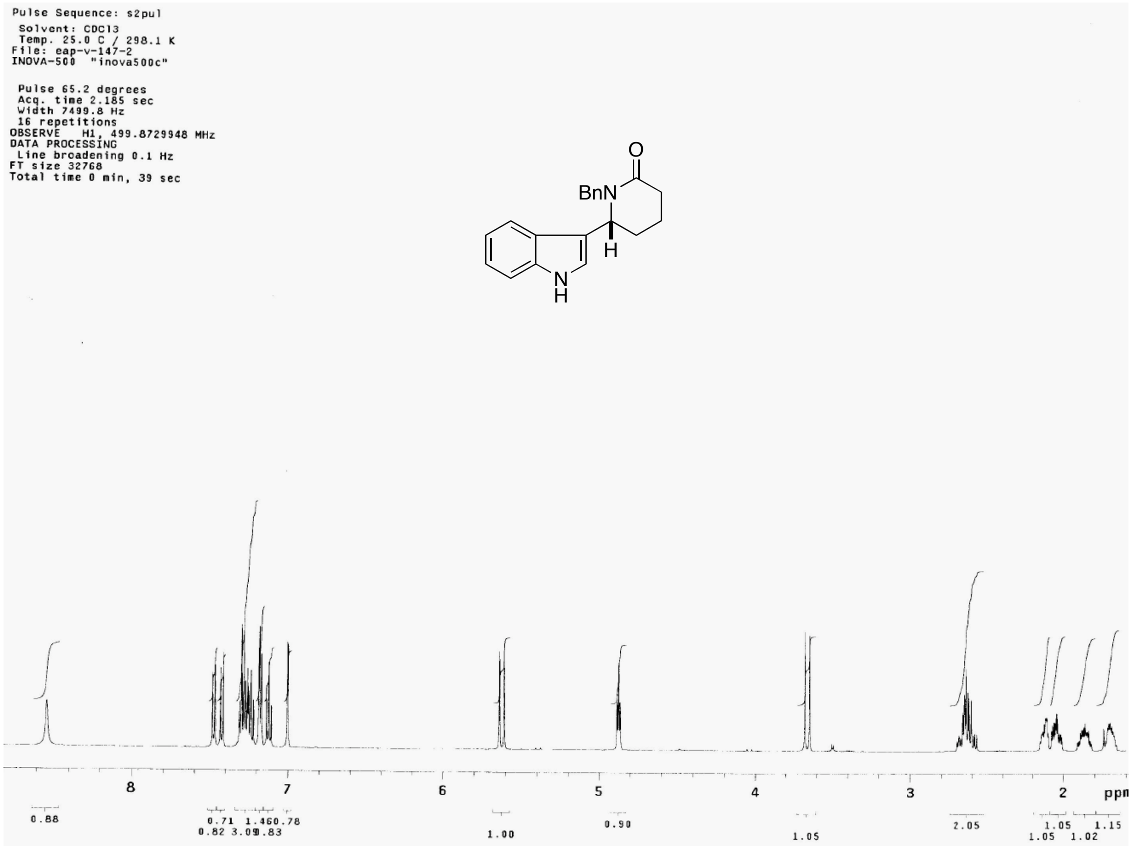

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
384 repetitions
OBSERVE C13, 125.6931675 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
O
BnN
H
N
H
160
140
120
100
80
60
40
ppm

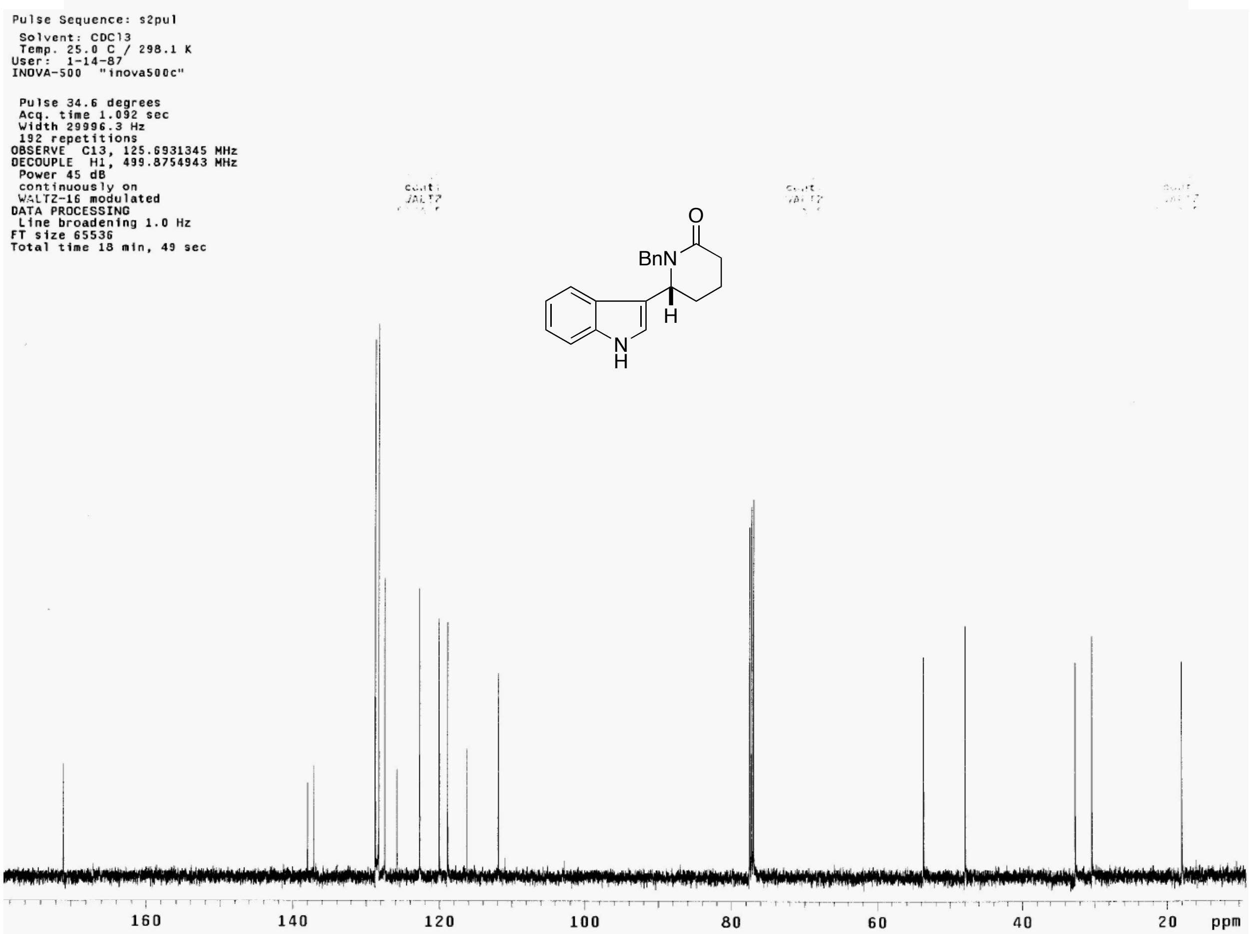

Pulse Sequence: s2pul
Solvent: DMSO
Temp. 23.0 C / 296.1 K
INOVA-500 "inova500c"
Pulse 51.8 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8753410 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
O
BnN
H
MeO
N
H
11 10 9 8 7 6 5 4 3 2
0.95
1.94
2.84
1.00
1.00
2.42
0.97
0.80

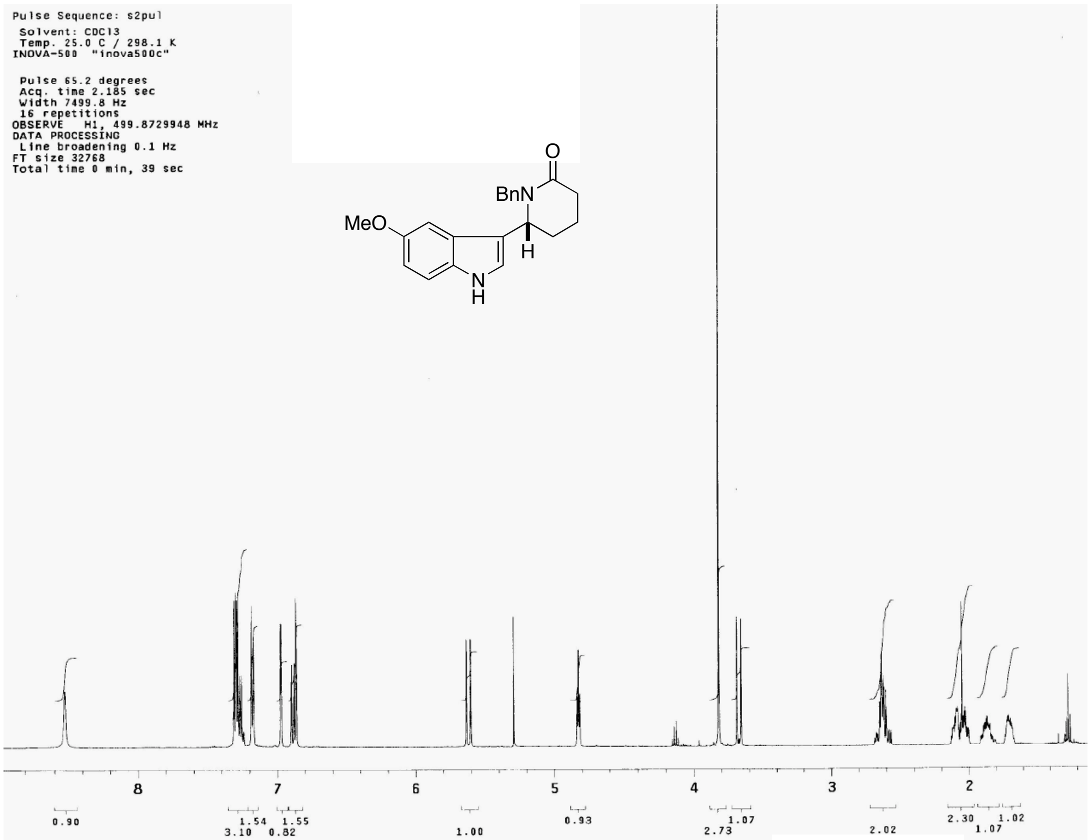

Pulse Sequence: s2pul
Solvent: DMSO
Temp. 23.0 C / 296.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
320 repetitions
OBSERVE C13, 125.6938207 MHz
DECOUPLE H1, 499.8778587 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
O
BnN
H
MeO
N
H
160 140 120 100 80 60 40 ppm

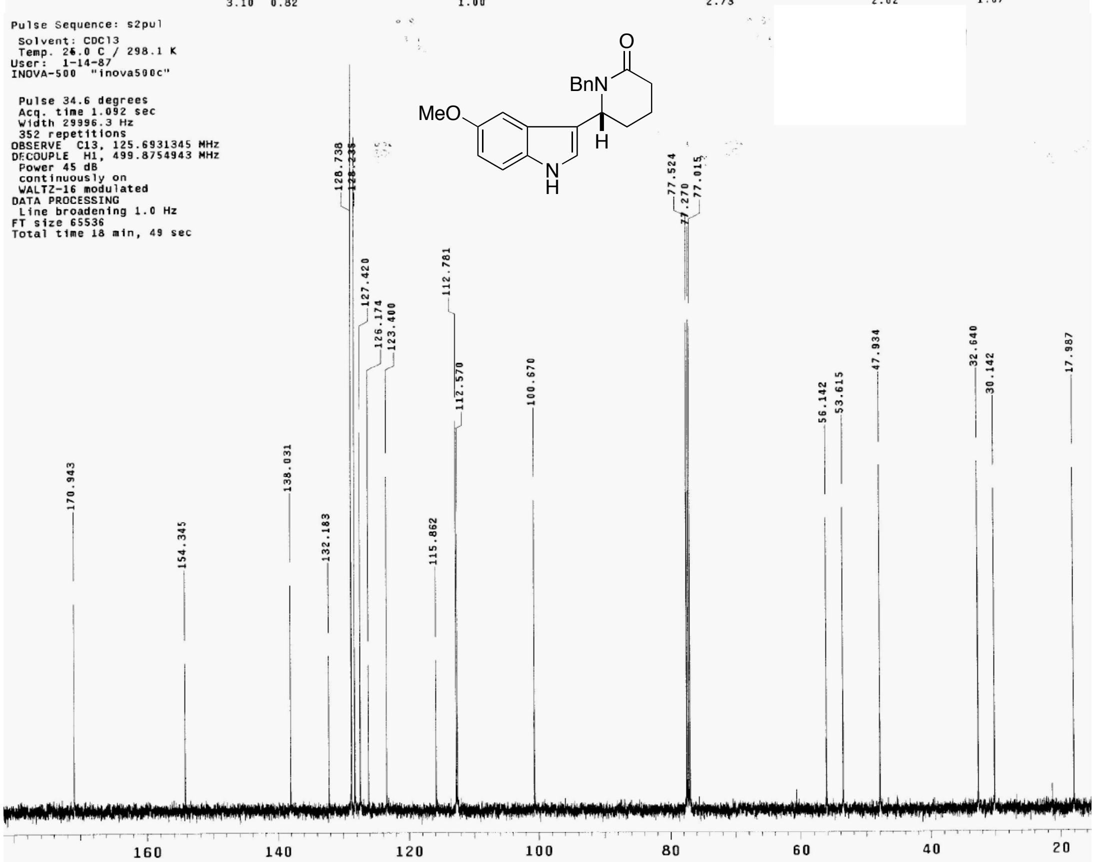

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: eap-IV-105-2
INOVA-500 "inova500c"
Pulse 69.8 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
O
MeN
H
N
H
8
7
6
5
4
3
2
ppm
0.96
0.81 0.94
0.91 1.27 0.84
1.00
2.95
2.45
2.53
1.38
2.24

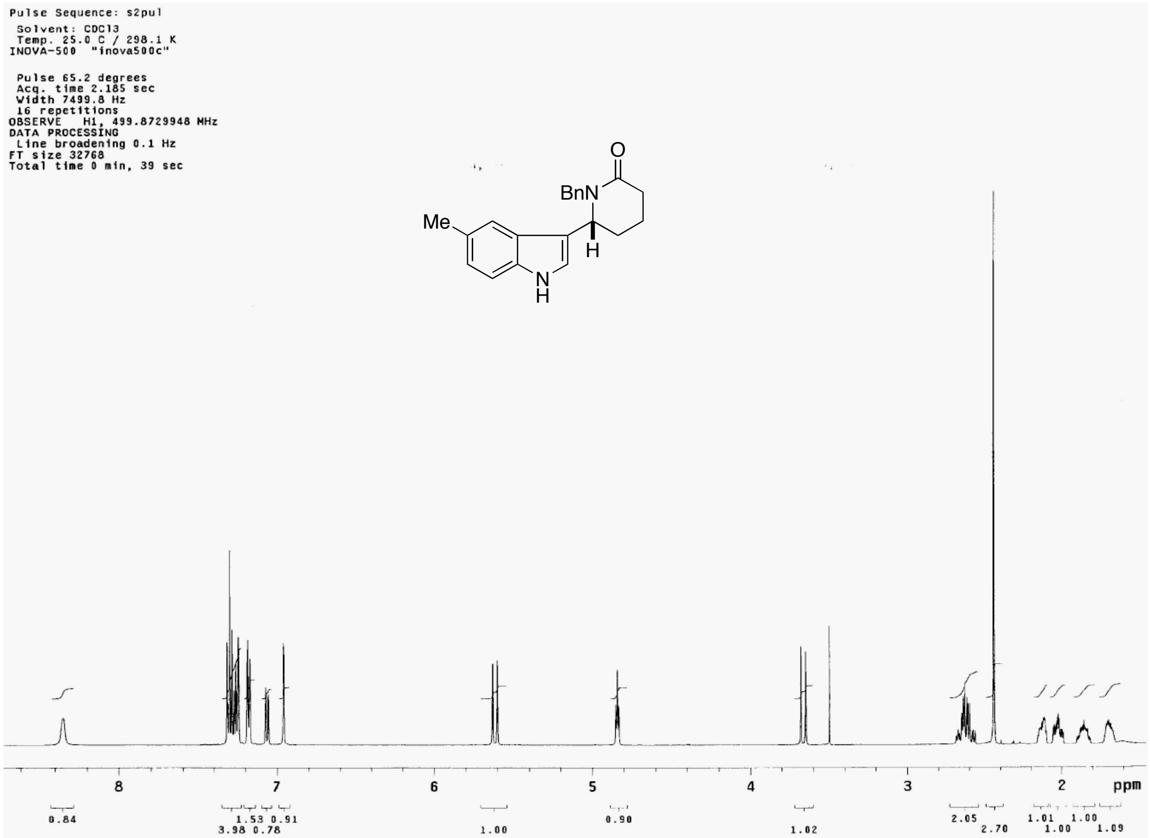

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 23.0 C / 296.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
1024 repetitions
OBSERVE C13, 125.6931675 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
O
MeN
H
N
H
160
140
120
100
80
60
40
ppm

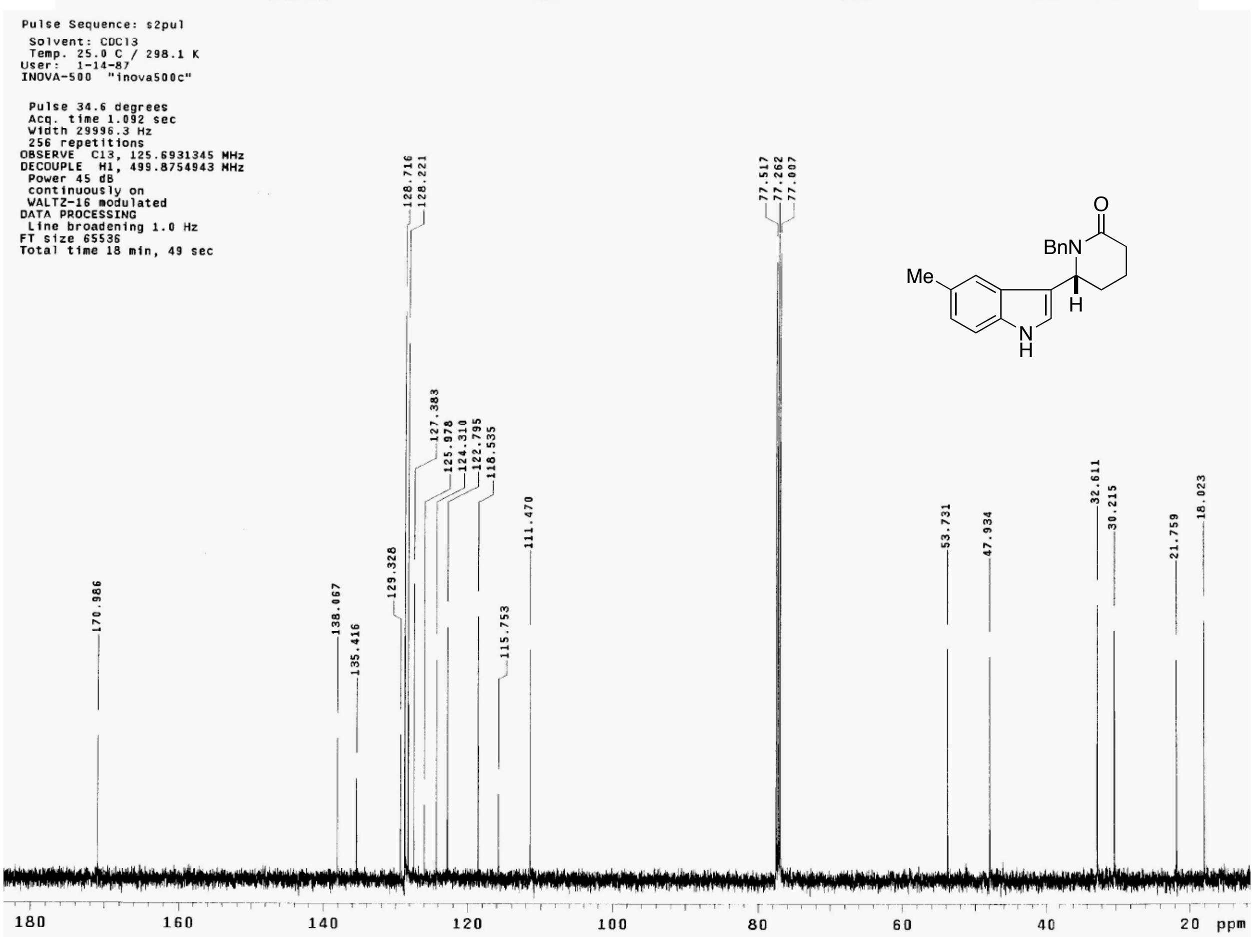

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: eap-v-147-2
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
O
BnN
H
N
H
8
7
6
5
4
3
2
ppm
0.88
0.71 1.46 0.78
0.82 3.09 0.83
1.00
0.90
1.05
2.05
1.05 1.15
1.05 1.02

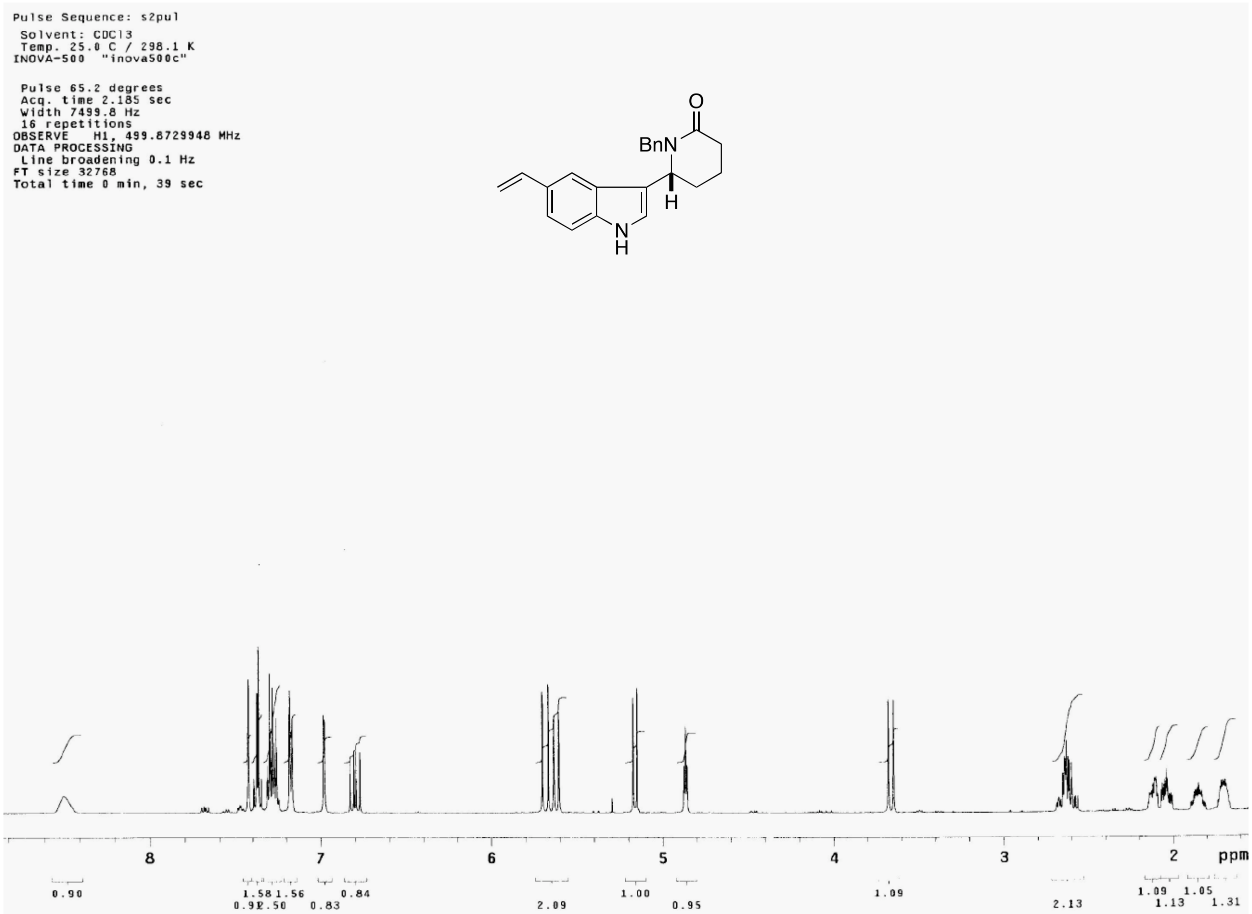

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
192 repetitions
OBSERVE C13, 125.6931345 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
O
BnN
H
N
H
160
140
120
100
80
60
40
20
ppm

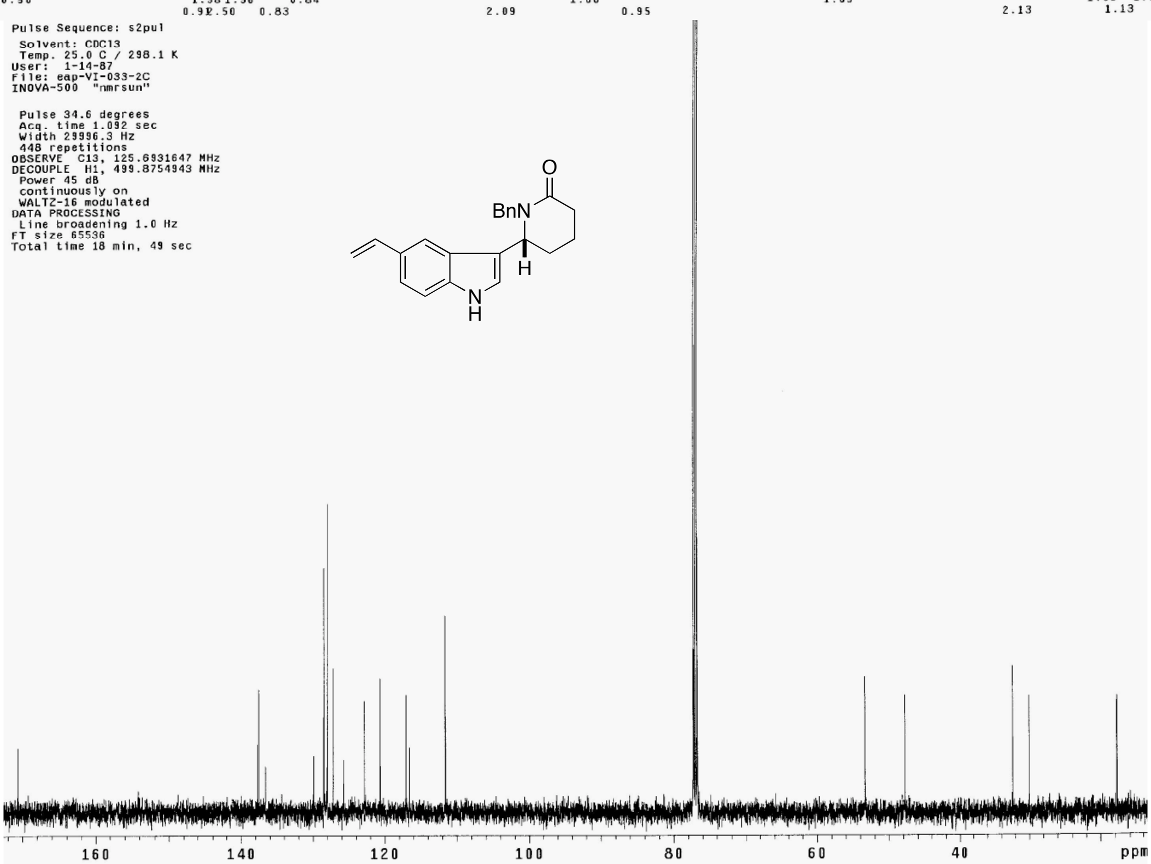

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
O
BnN
MeO
H
N
H
8
7
6
5
4
3
2
0.90
1.54
1.55
3.10
0.82
1.00
0.93
2.73
1.07
2.02
2.30
1.07
1.02

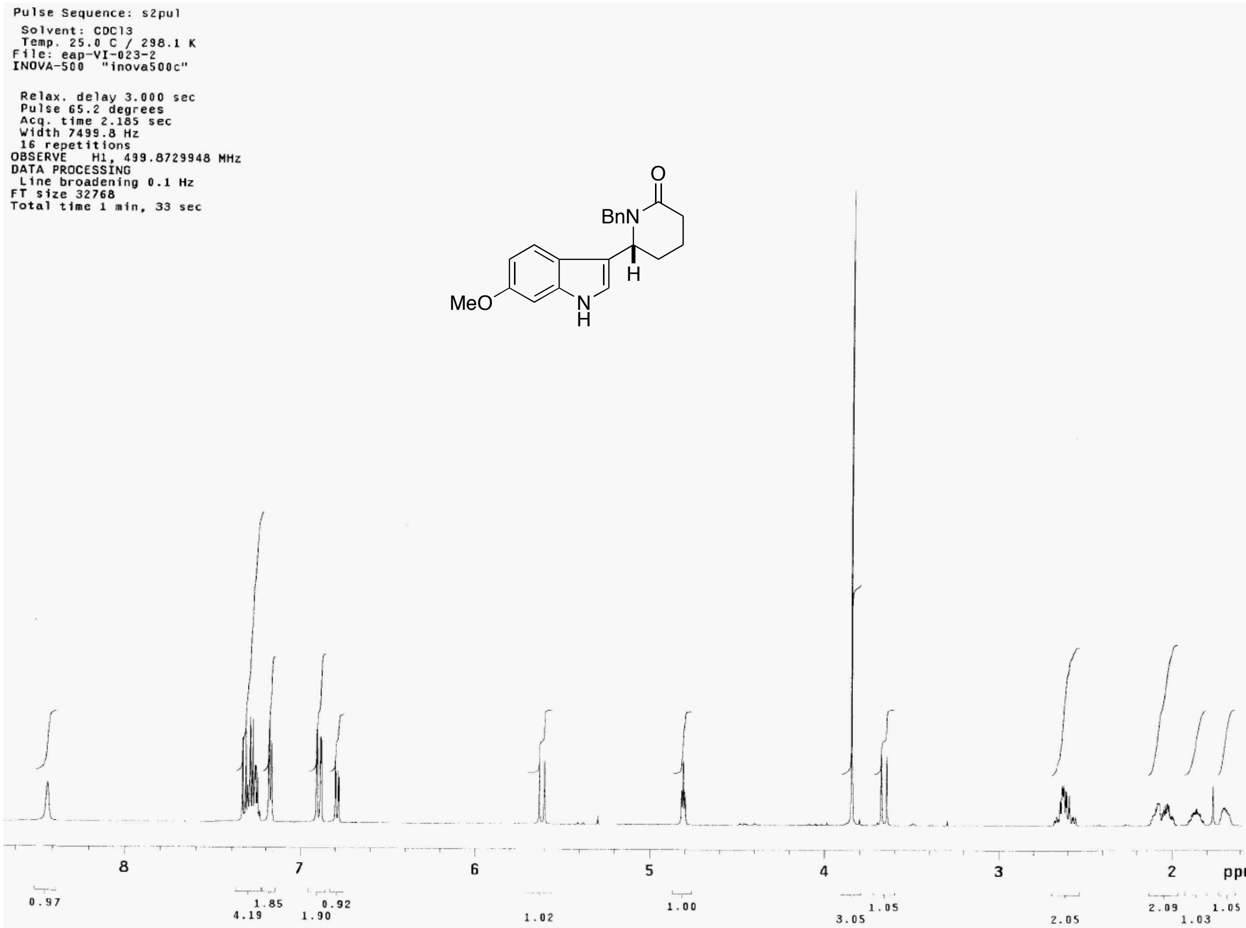

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
352 repetitions
OBSERVE C13, 125.6931345 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
O
BnN
MeO
H
N
H
170.943
154.345
138.031
132.183
128.738
127.420
126.174
123.400
115.862
112.781
112.570
100.670
77.524
77.270
77.015
56.142
53.615
47.934
32.640
30.142
17.987
160
140
120
100
80
60
40
20

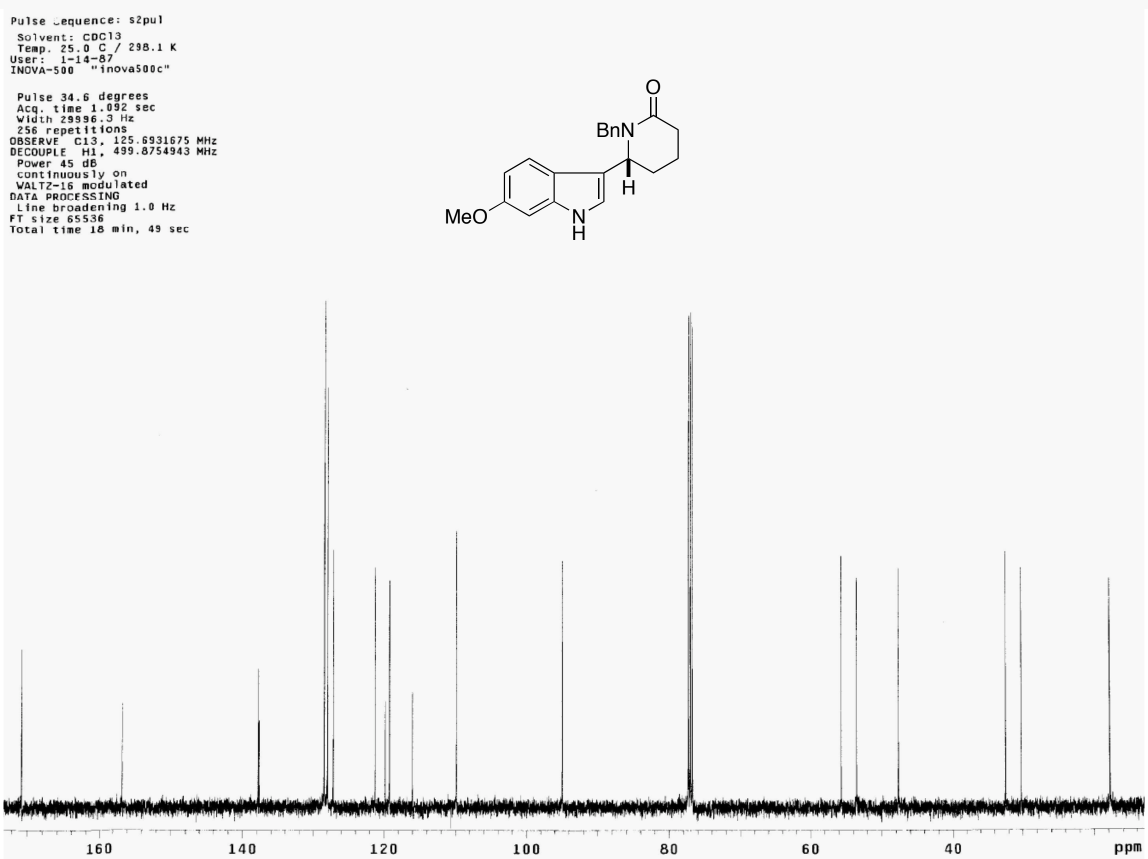

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
O
BnN
Me
H
N
H
8
7
6
5
4
3
2
ppm
0.84
1.53 0.91
3.98 0.78
1.00
0.90
1.02
2.05
2.70
1.01 1.00
1.00 1.09

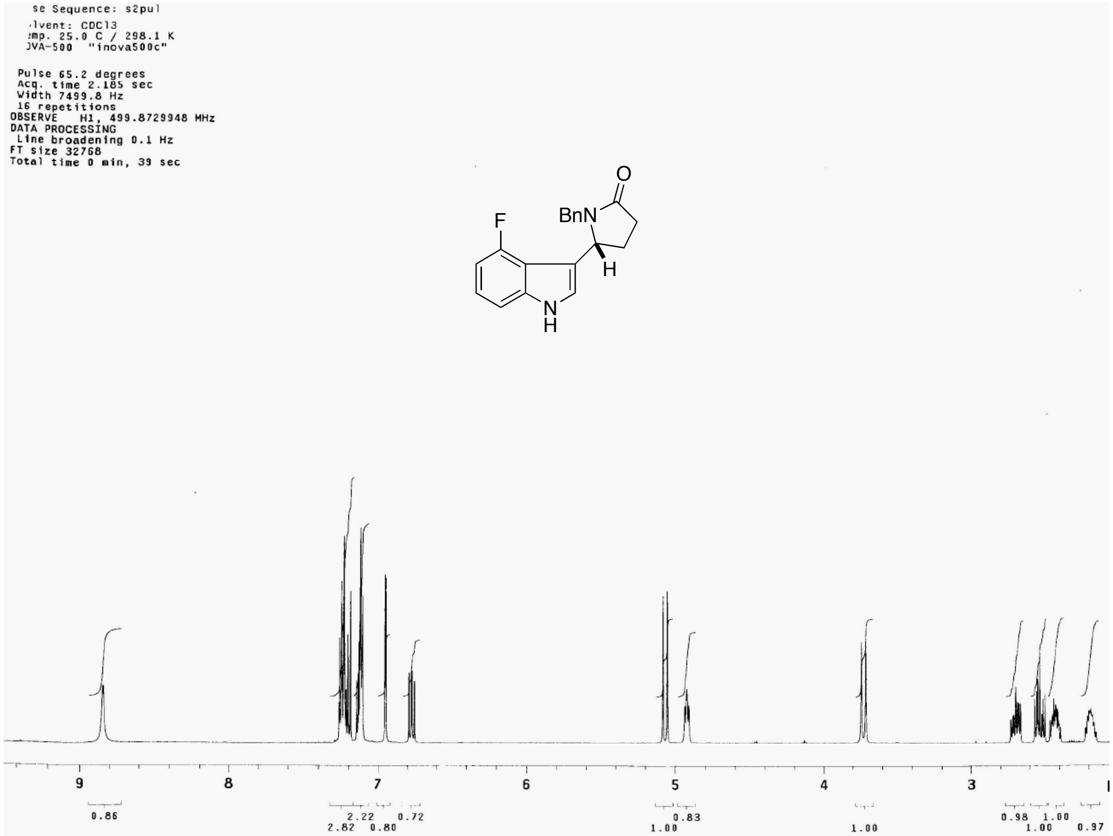

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
256 repetitions
OBSERVE C13, 125.6931345 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
170.986
138.067
135.416
129.328
128.716
128.221
127.383
125.978
124.310
122.795
118.535
115.753
111.470
77.517
77.262
77.007
53.731
47.934
32.611
30.215
21.759
18.023
O
BnN
Me
H
N
H
180
160
140
120
100
80
60
40
20
ppm

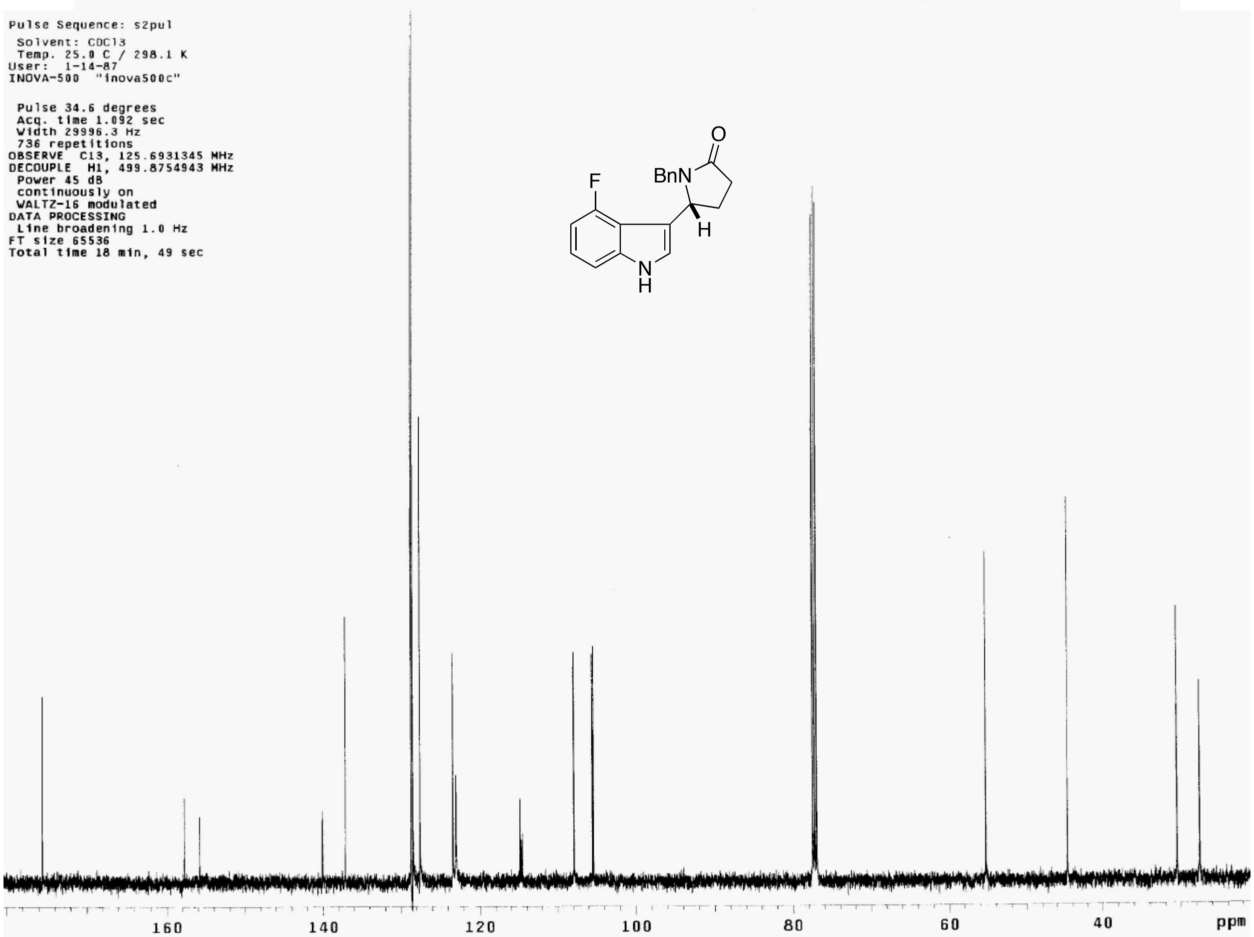

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
BnN
O
H
N
H
ppm

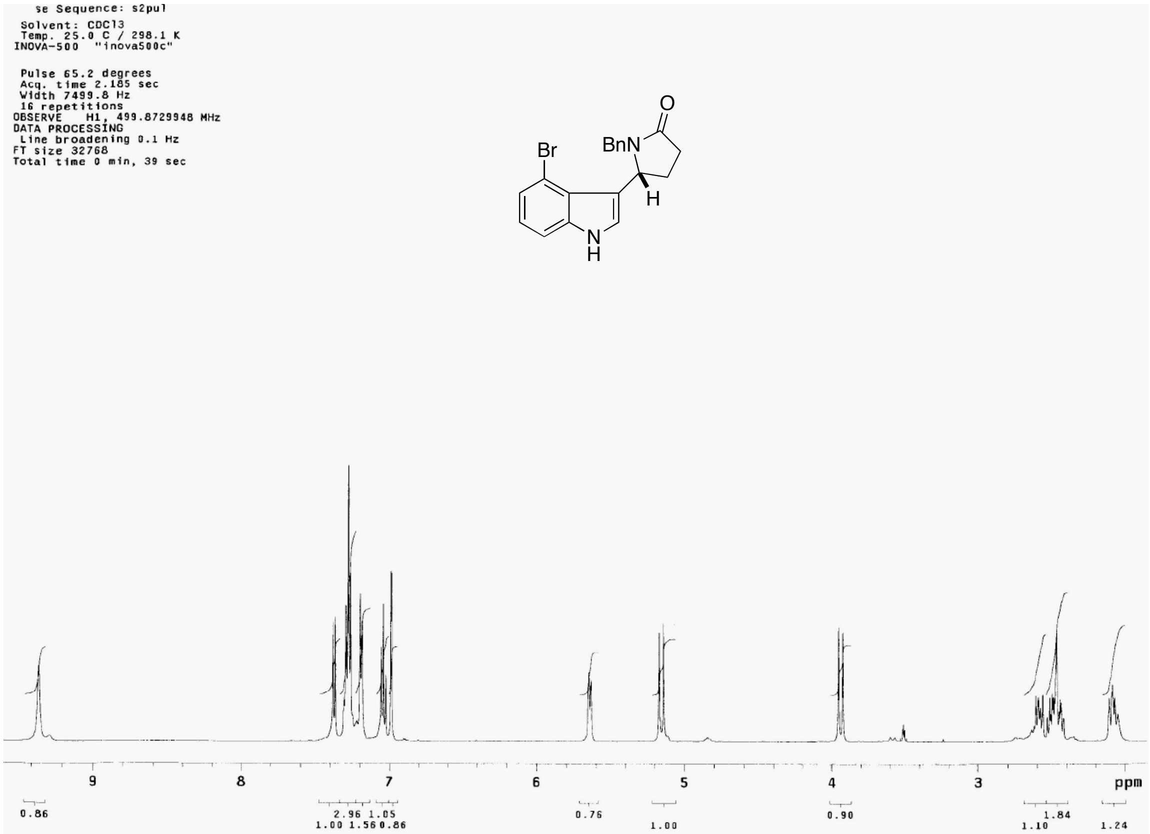

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: eap-VI-033-2C
INOVA-500 "nmrsun"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
448 repetitions
OBSERVE C13, 125.6931647 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
BnN
O
H
N
H
ppm

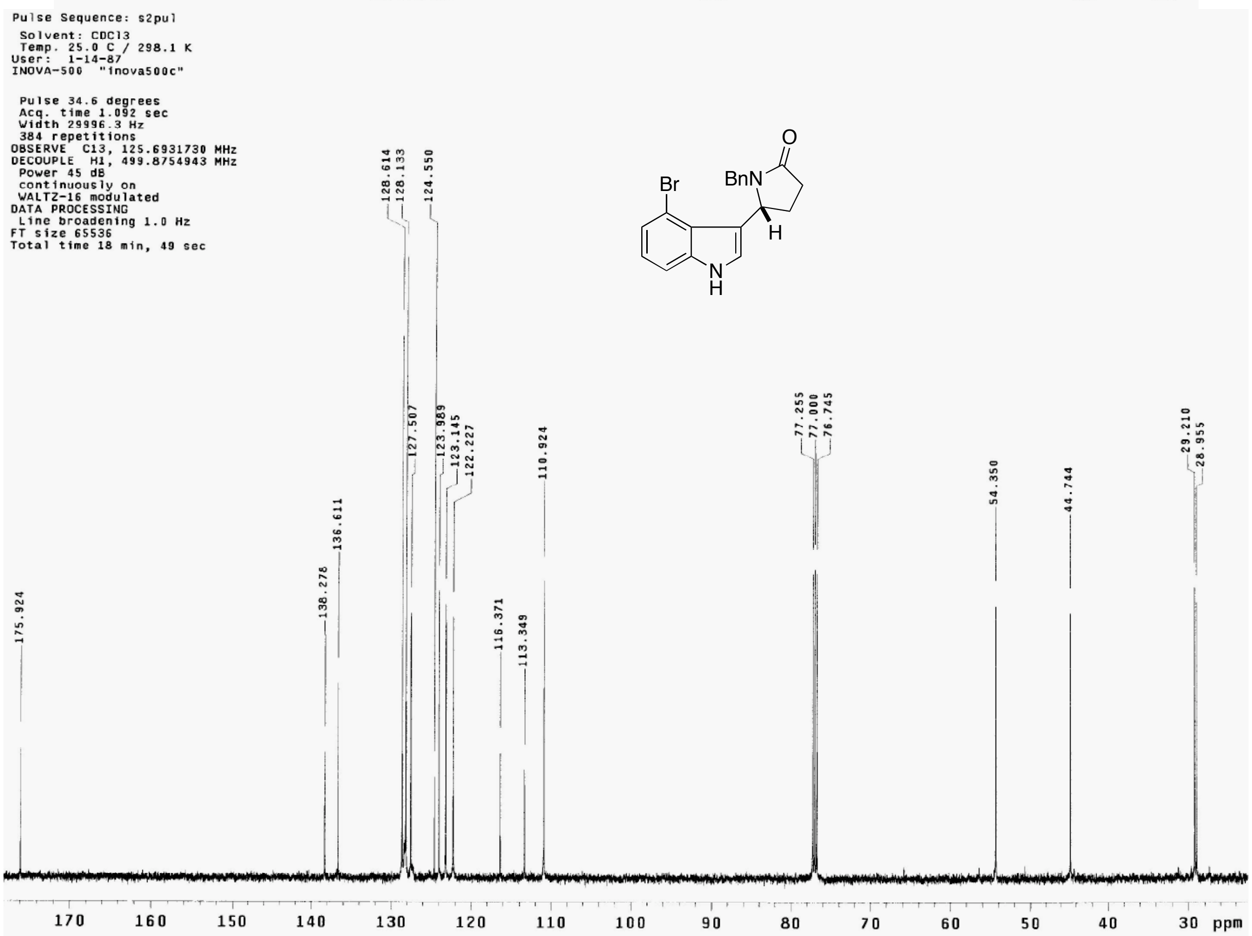

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: eap-VI-023-2
INOVA-500 "inova500c"
Relax. delay 3.000 sec
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 1 min, 33 sec
O
BnN
H
MeO
N
H
ppm

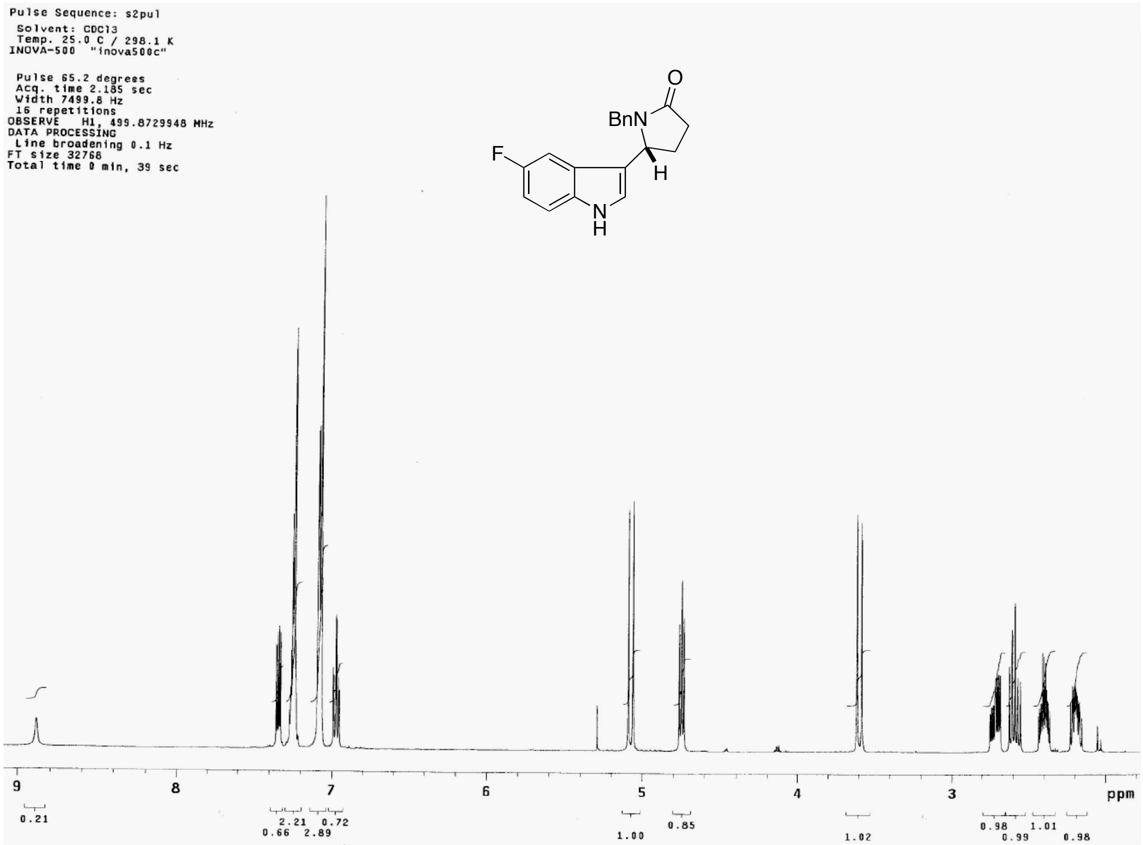

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
256 repetitions
OBSERVE C13, 125.6931675 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
O
BnN
H
MeO
N
H
ppm

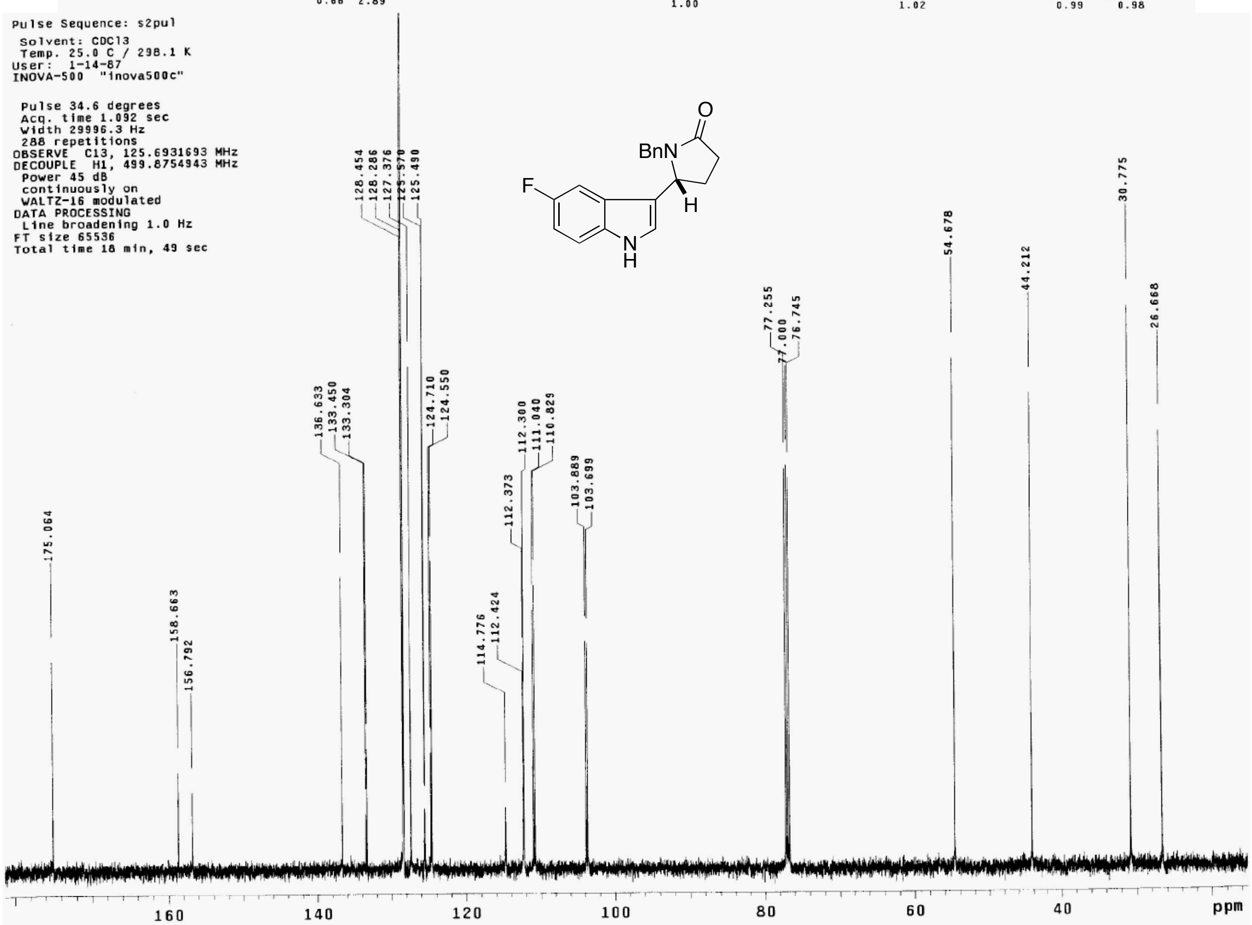

Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
F
BnN
O
H
N
H

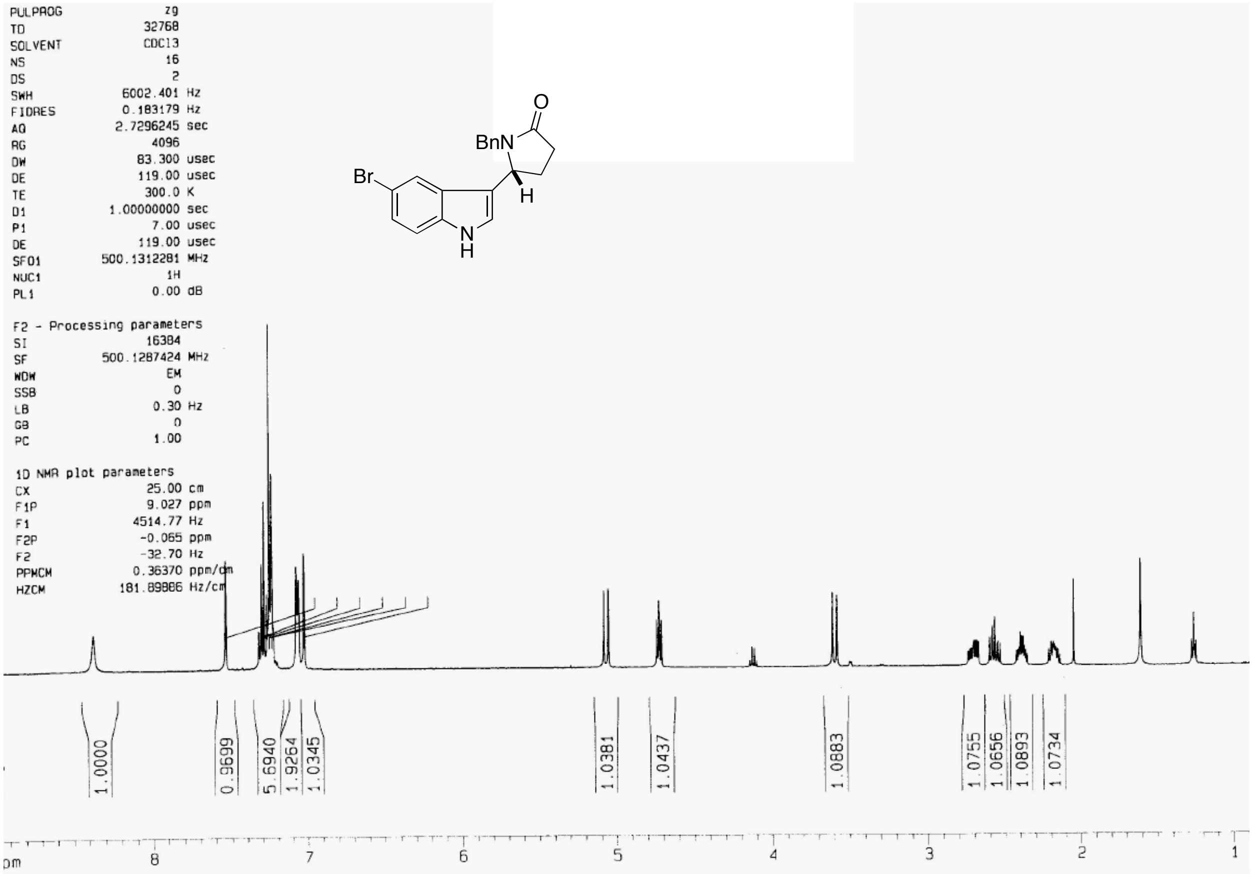

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
736 repetitions
OBSERVE C13, 125.6931345 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
F
BnN
O
H
N
H
ppm

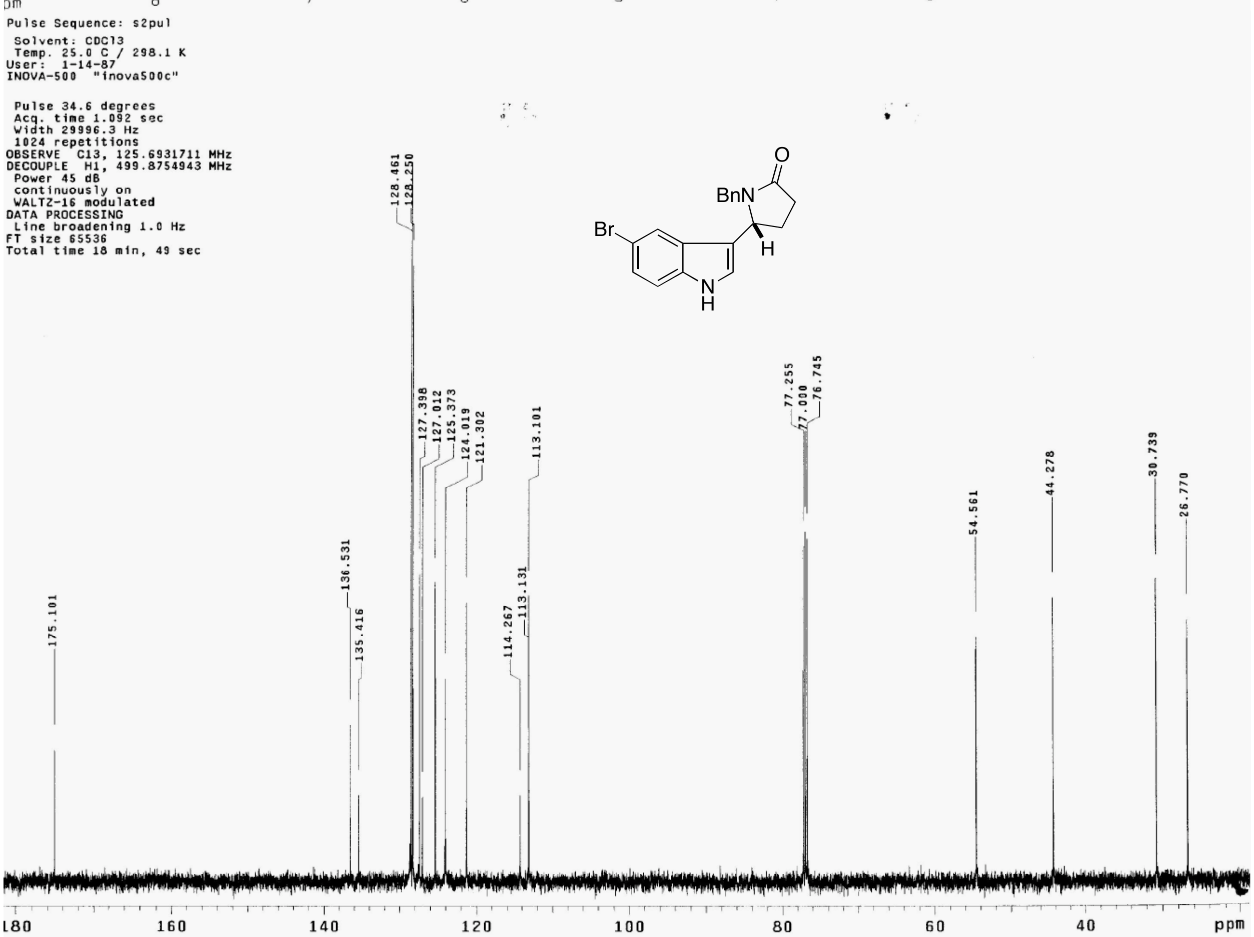

se Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
Br
BnN
O
H
N
H
9
8
7
6
5
4
3
ppm
0.86
2.96 1.05
1.00 1.56 0.86
0.76
1.00
0.90
1.84
1.10
1.24

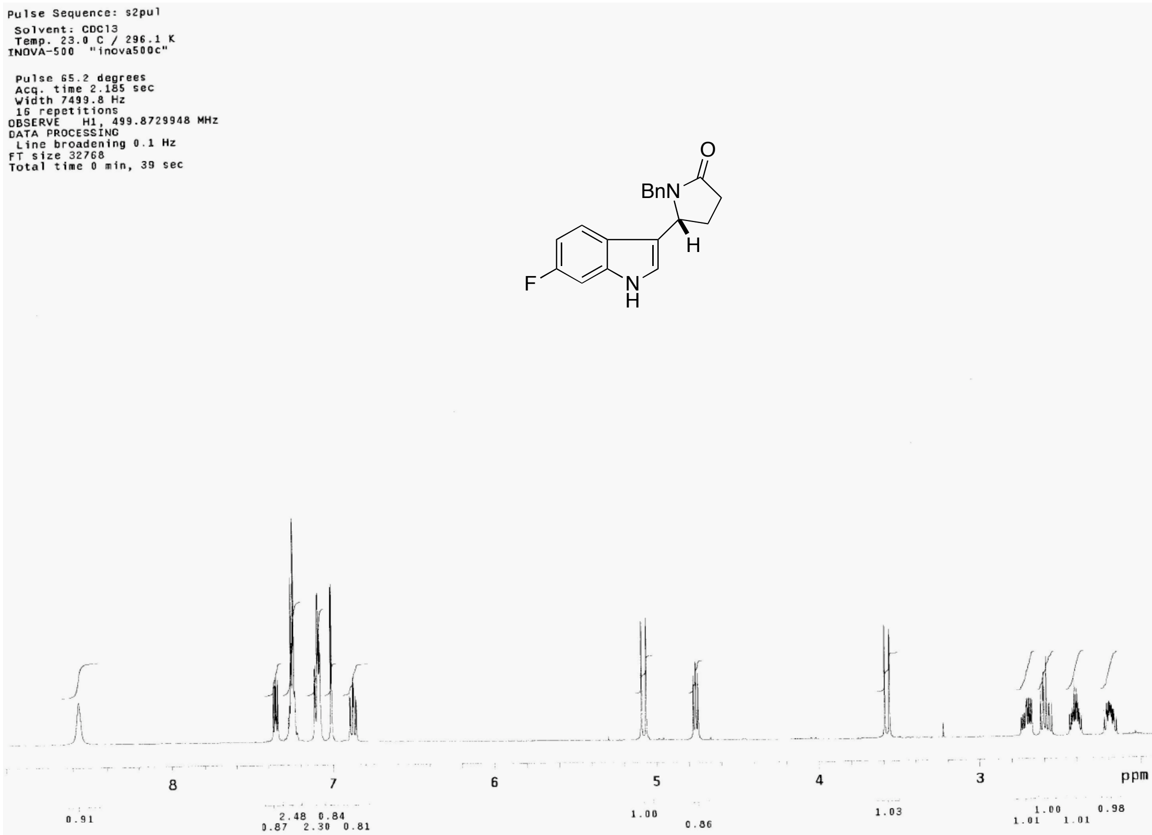

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
384 repetitions
OBSERVE C13, 125.6931730 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
Br
BnN
O
H
N
H
175.924
138.276
136.611
128.614
128.133
127.507
124.550
123.989
123.145
122.227
116.371
113.349
110.924
77.255
77.000
76.745
54.350
44.744
29.210
28.955
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

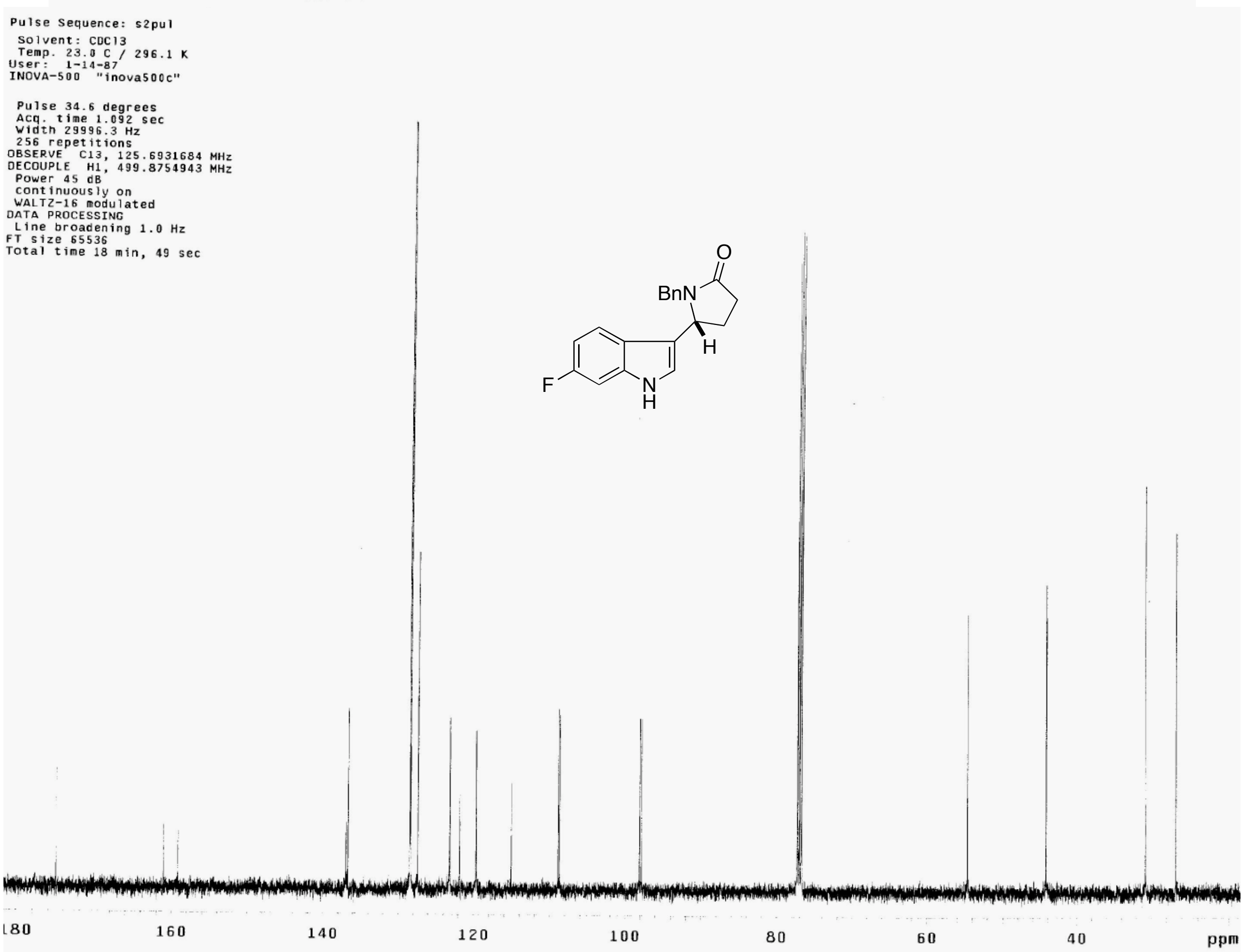

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
O
BnN
F
H
N
H
9
8
7
6
5
4
3
ppm
0.21
2.21
0.72
0.66
2.89
1.00
0.85
1.02
0.98
1.01
0.99
0.98

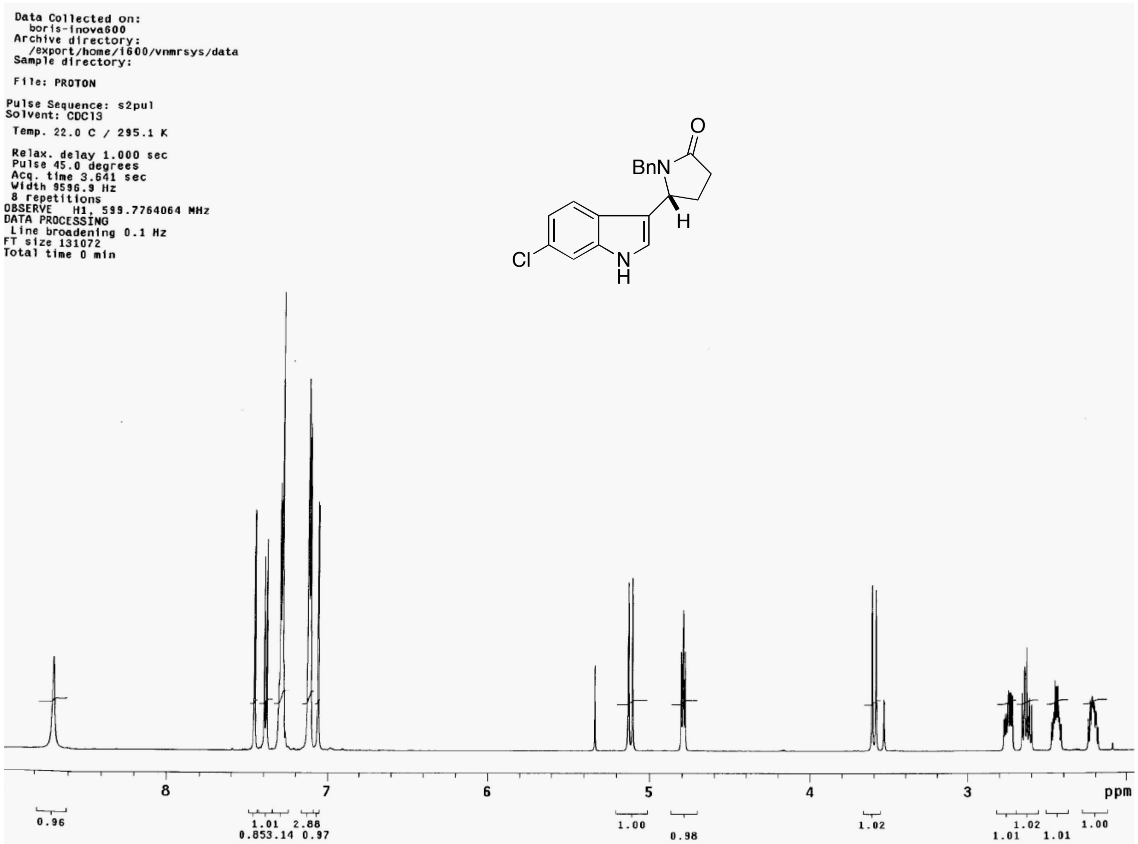

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
288 repetitions
OBSERVE C13, 125.6931693 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
O
BnN
F
H
N
H
175.064
158.663
156.792
136.633
133.450
133.304
128.454
128.286
127.376
125.570
125.490
124.710
124.550
114.776
112.424
112.373
112.300
111.040
110.829
103.889
103.699
77.255
77.000
76.745
54.678
44.212
30.775
26.668
160
140
120
100
80
60
40
ppm

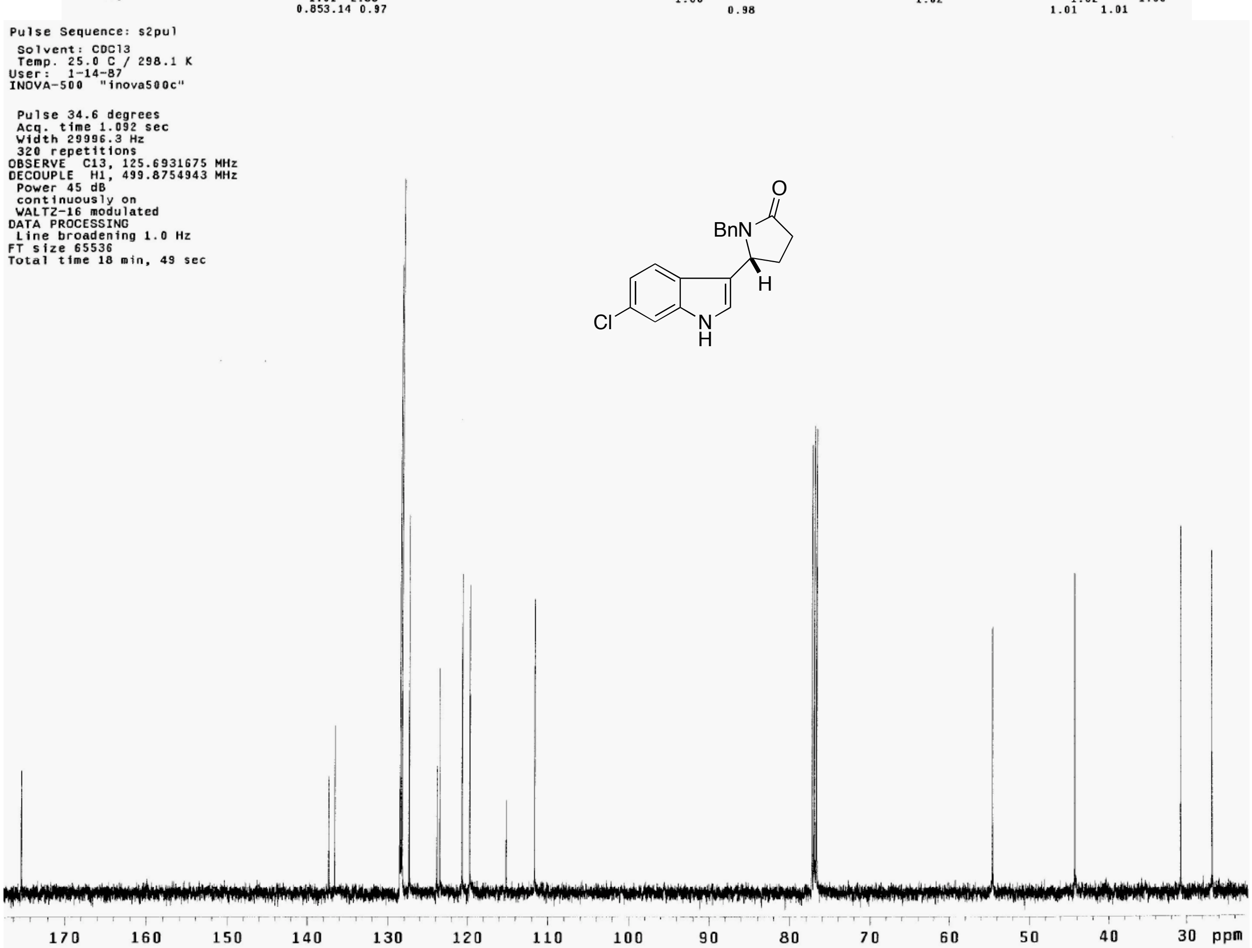

PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6002.401 Hz
FIDRES 0.183179 Hz
AQ 2.7296245 sec
RG 4096
DW 83.300 usec
DE 119.00 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.00 usec
DE 119.00 usec
SFO1 500.1312281 MHz
NUC1 1H
PL1 0.00 dB
F2 - Processing parameters
SI 16384
SF 500.1287424 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
F1P 9.027 ppm
F1 4514.77 Hz
F2P -0.065 ppm
F2 -32.70 Hz
PPMCM 0.36370 ppm/cm
HZCM 181.89886 Hz/cm
BnN
O
Br
H
N
H
1.0000
0.9699
5.6940
1.9264
1.0345
1.0381
1.0437
1.0883
1.0755
1.0656
1.0893
1.0734
ppm
8
7
6
5
4
3
2
1

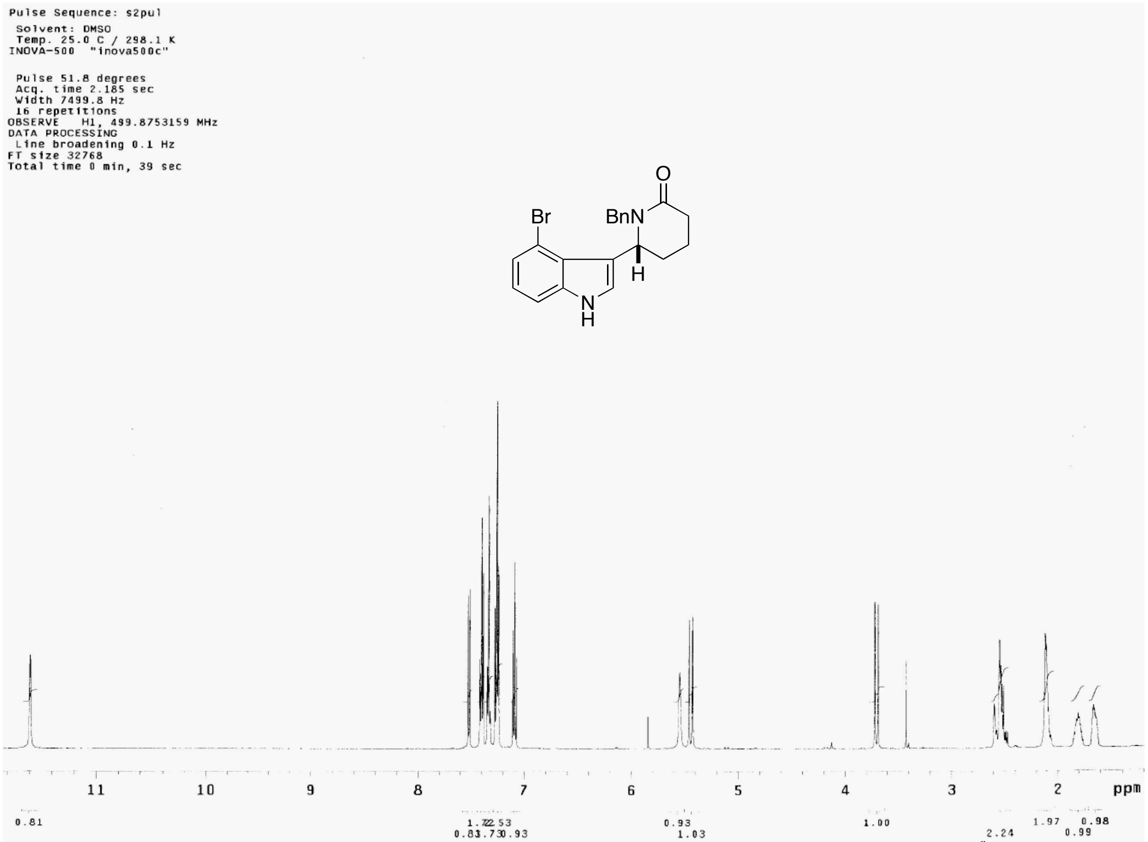

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
1024 repetitions
OBSERVE C13, 125.6931711 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
BnN
O
Br
H
N
H
175.101
136.531
135.416
128.461
128.250
127.398
127.012
125.373
124.019
121.302
114.267
113.131
113.101
77.255
77.000
76.745
54.561
44.278
30.739
26.770
180
160
140
120
100
80
60
40
ppm

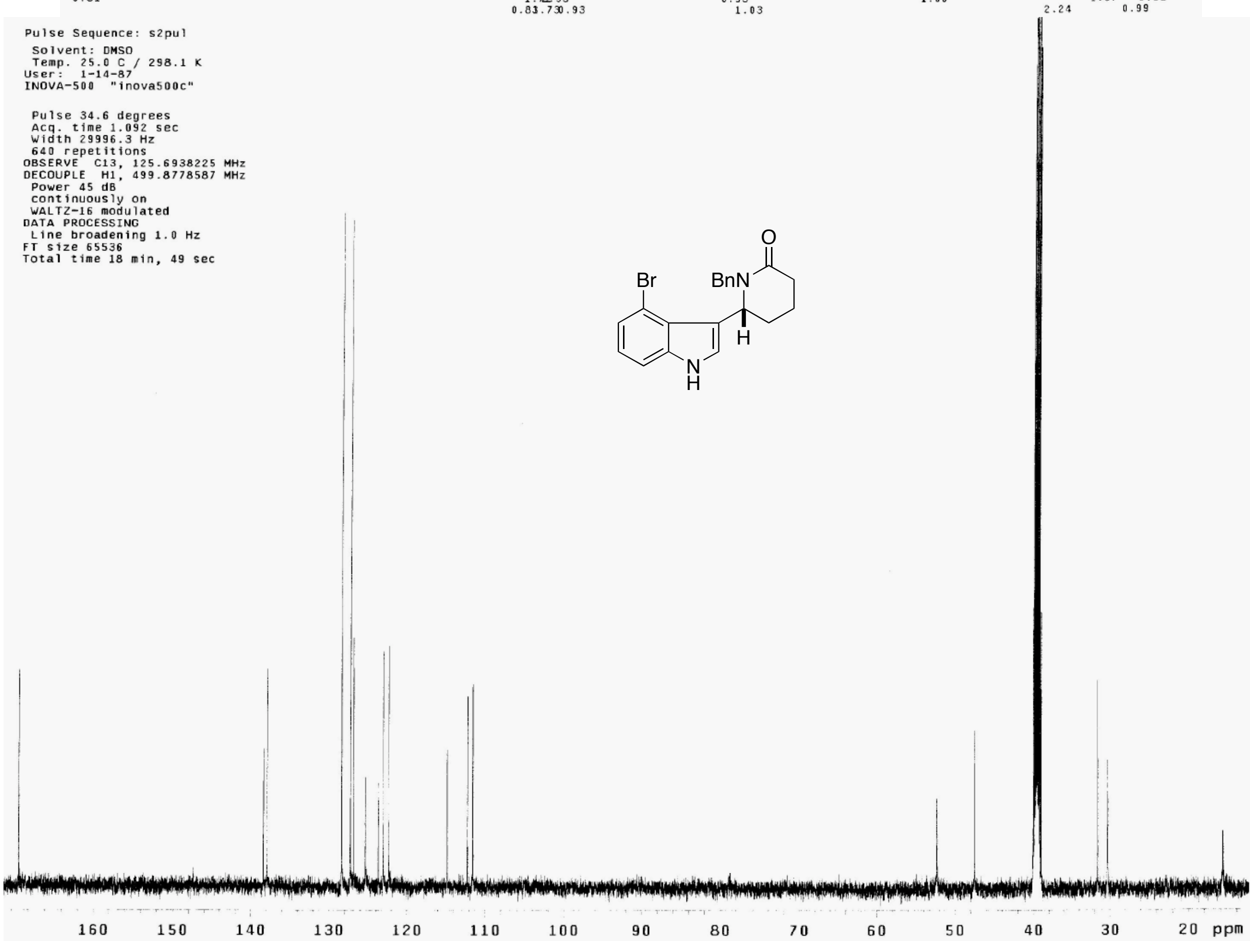

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 23.0 C / 296.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
BnN
O
H
F
N
H
8
7
6
5
4
3
ppm
0.91
2.48 0.84
0.87 2.30 0.81
1.00
0.86
1.03
1.00 0.98
1.01 1.01

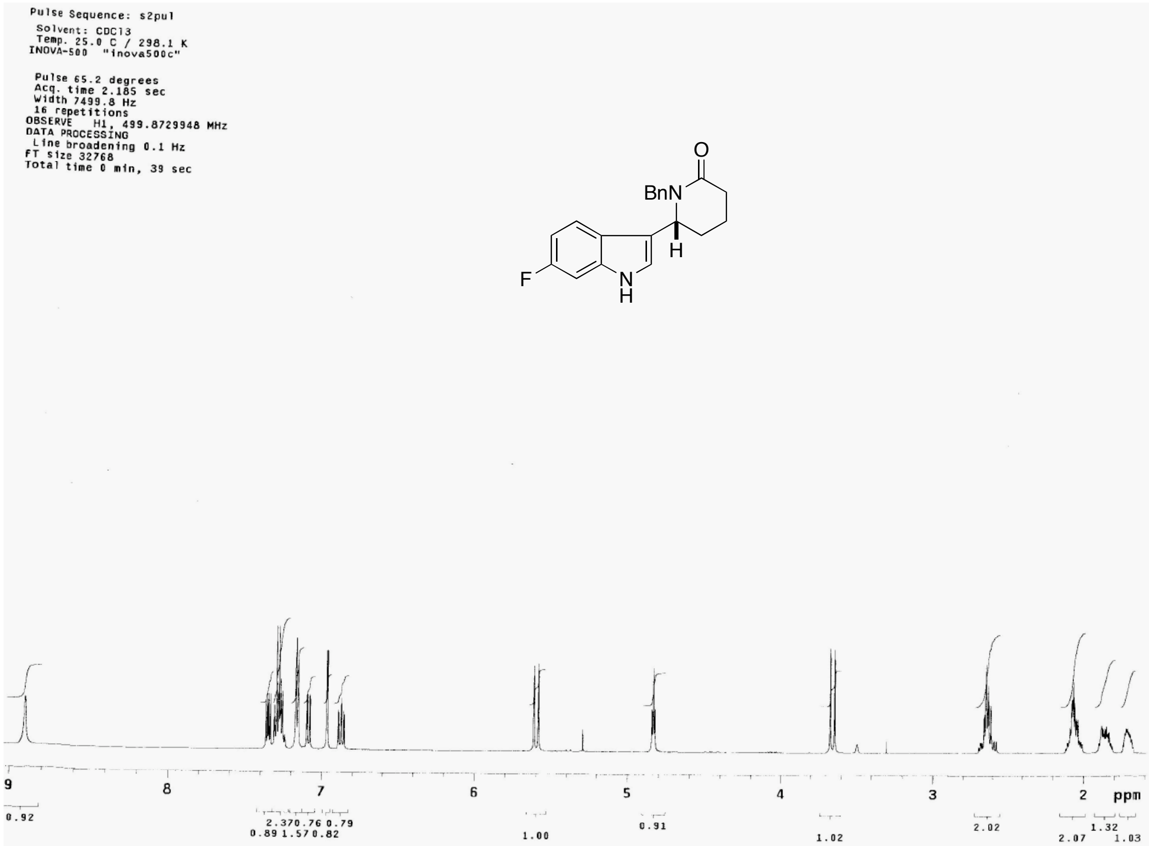

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 23.0 C / 296.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
256 repetitions
OBSERVE C13, 125.6931684 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
BnN
O
H
F
N
H
180
160
140
120
100
80
60
40
ppm

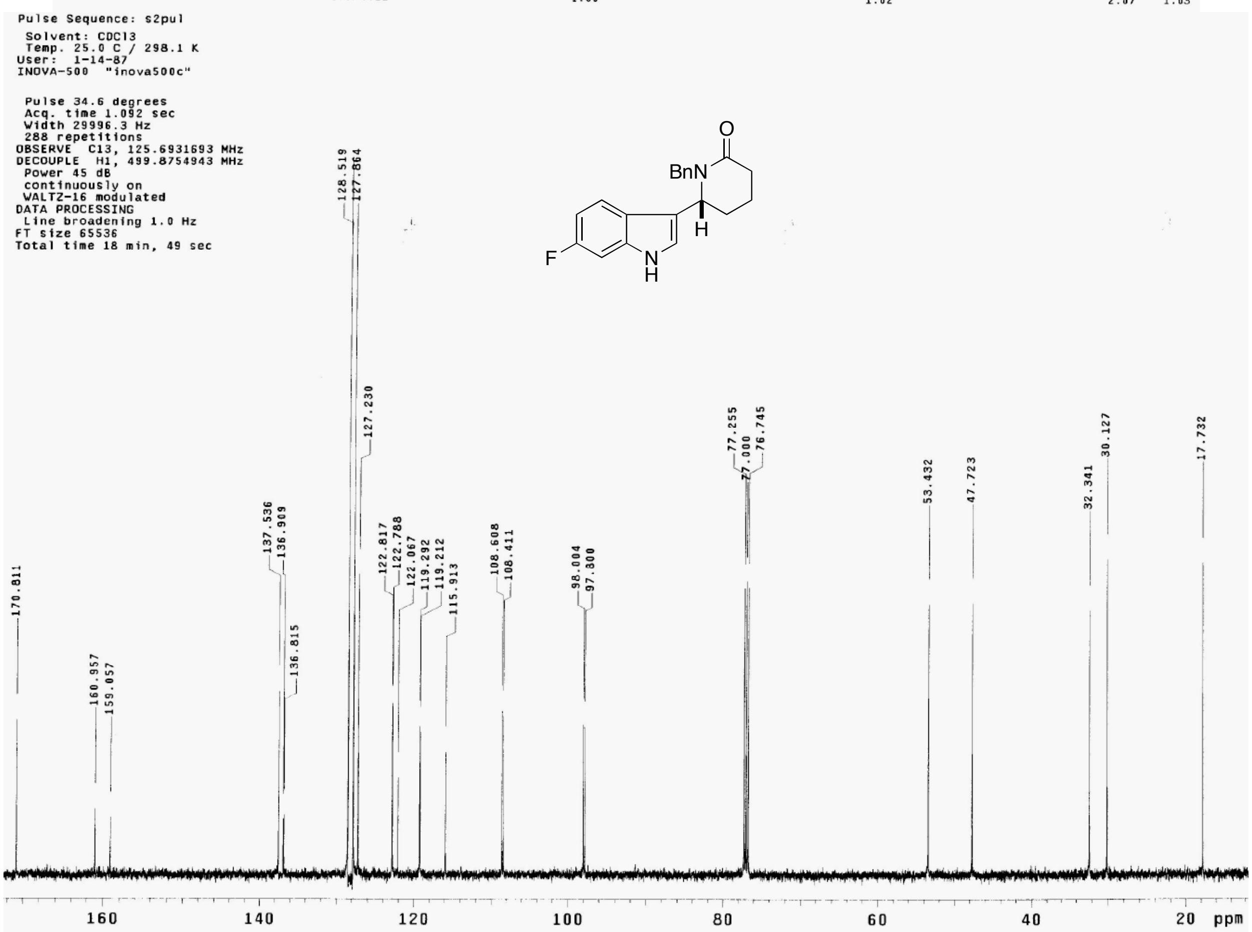


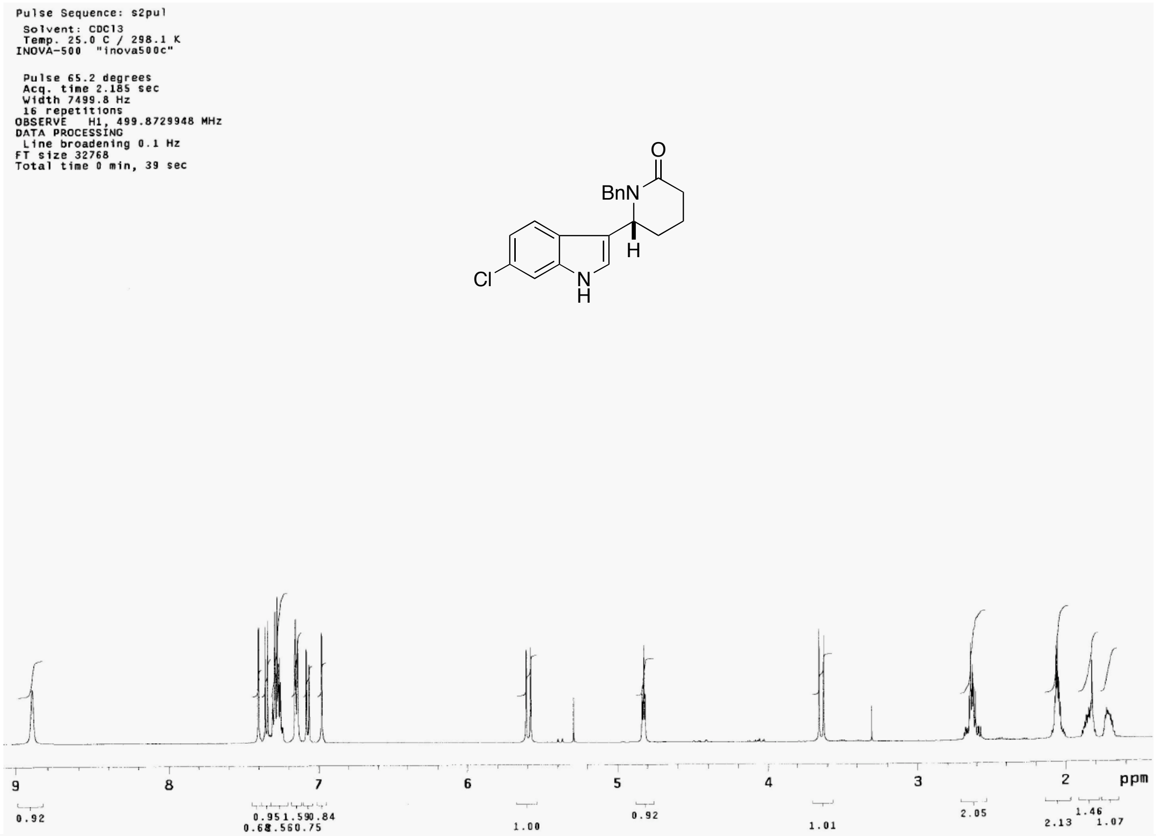


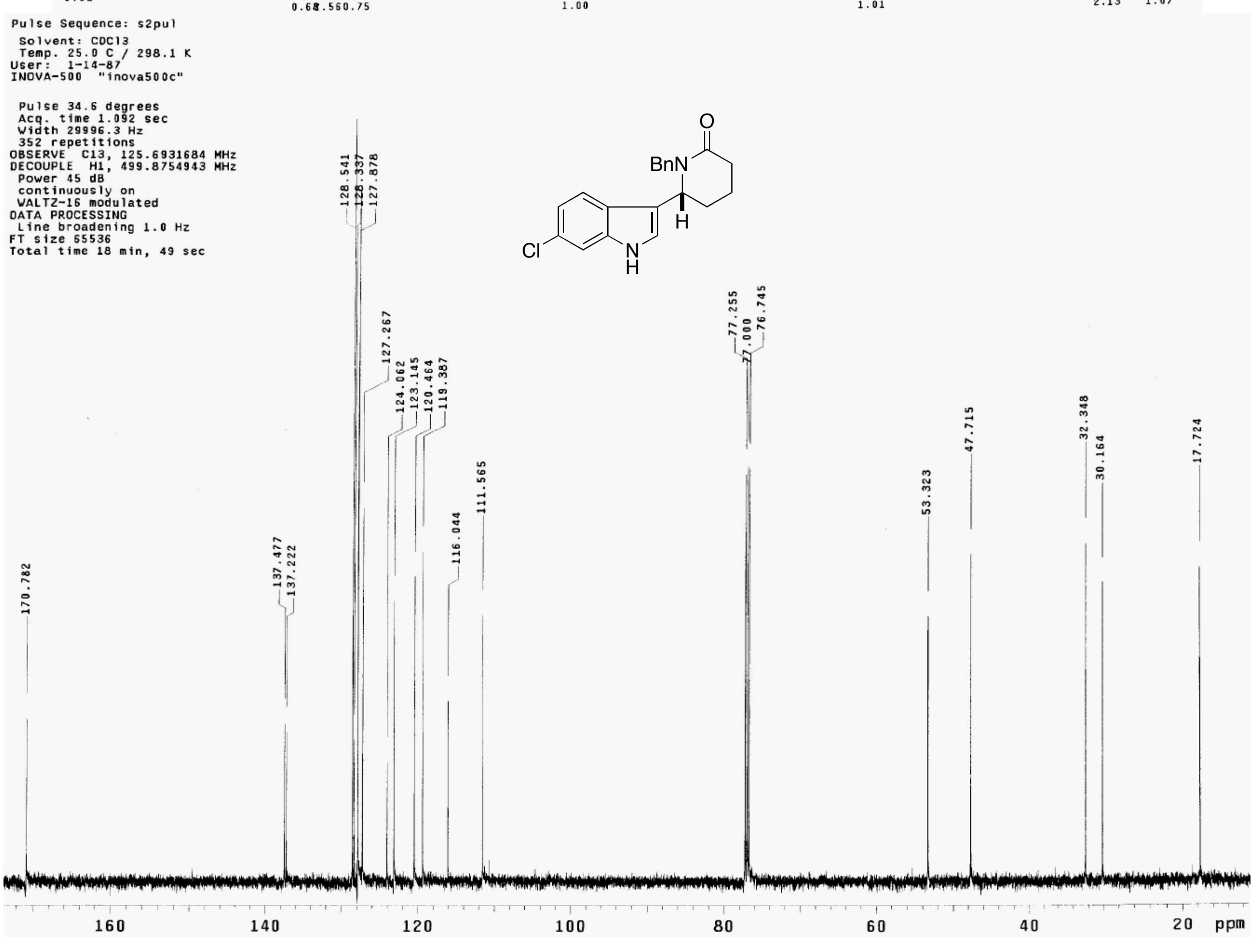

Pulse Sequence: s2pul
Solvent: DMSO
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 51.8 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8753159 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
O
Br
BnN
H
N
H
11
10
9
8
7
6
5
4
3
2
ppm

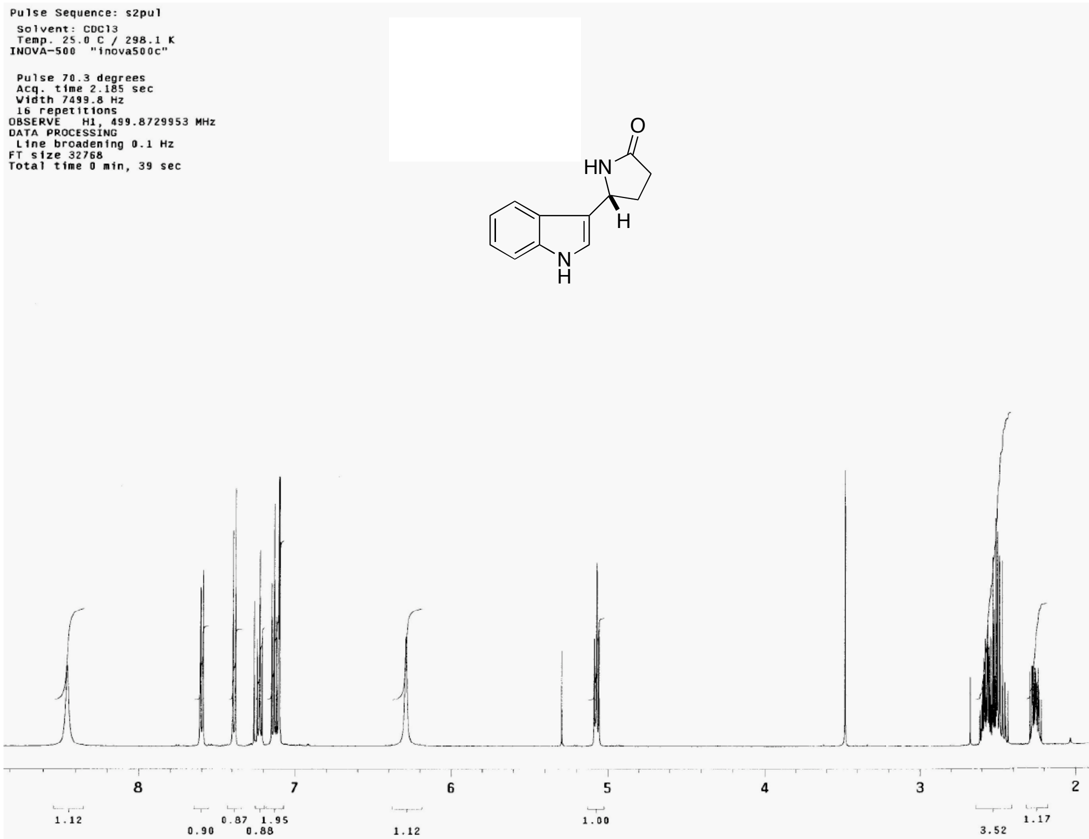

Pulse Sequence: s2pul
Solvent: DMSO
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
640 repetitions
OBSERVE C13, 125.6938225 MHz
DECOUPLE H1, 499.8778587 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
O
Br
BnN
H
N
H
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

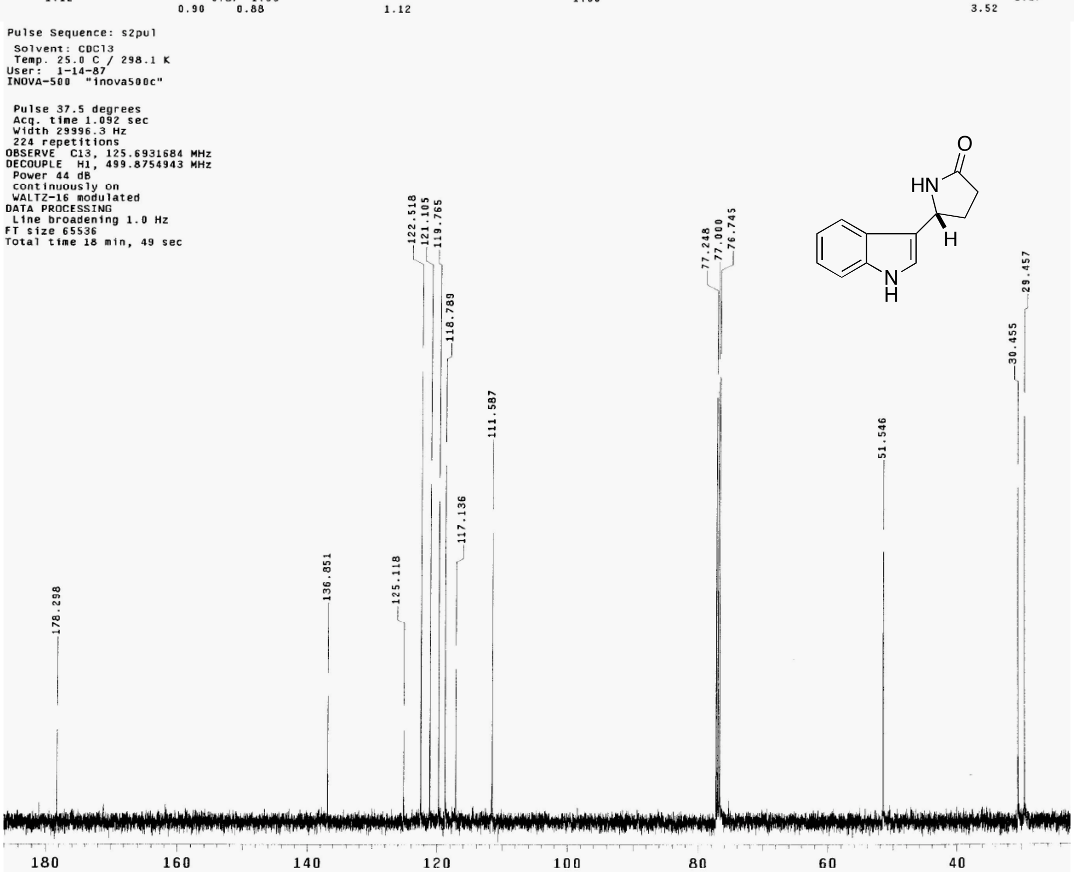

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
BnN
O
H
F
N
H
9 8 7 6 5 4 3 2 ppm
0.92
2.37 0.76 0.79
0.89 1.57 0.82
1.00
0.91
1.02
2.02
2.07 1.32 1.03

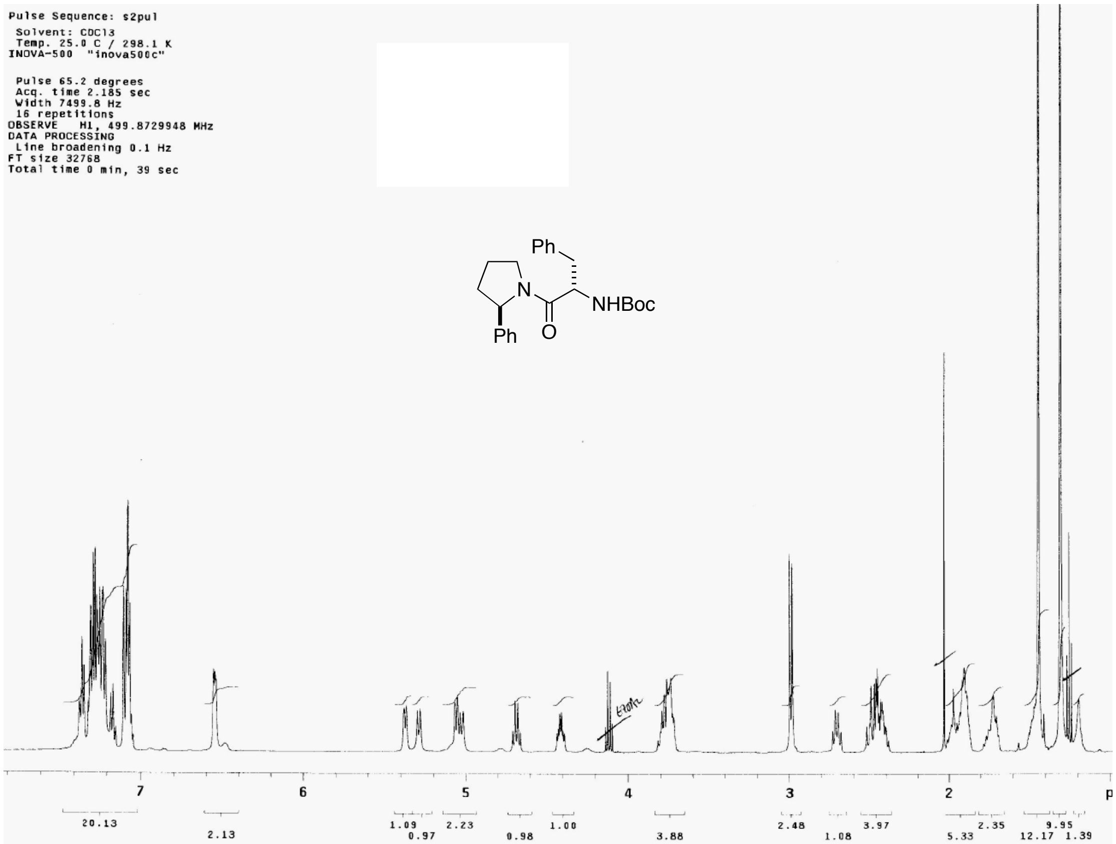

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
288 repetitions
OBSERVE C13, 125.6931693 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
BnN
O
H
F
N
H
170.811
160.957
159.057
137.536
136.909
136.815
128.519
127.864
127.230
122.817
122.788
122.067
119.292
119.212
115.913
108.608
108.411
98.004
97.800
77.255
77.000
76.745
53.432
47.723
32.341
30.127
17.732
160 140 120 100 80 60 40 20 ppm

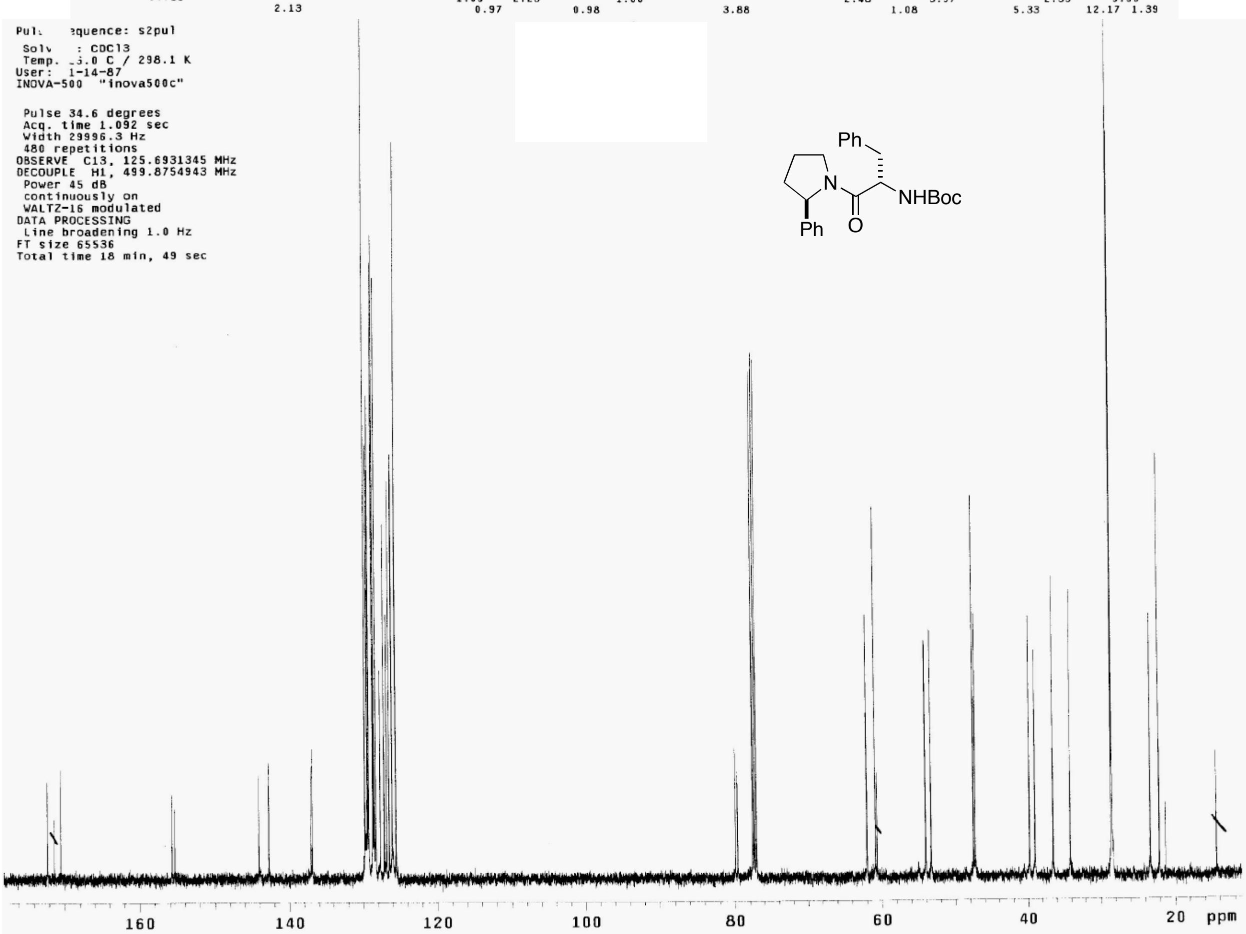

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
O
BnN
H
Cl
N
H
ppm
0.92
1.00
0.92
1.01
2.05
2.13
1.46
1.07

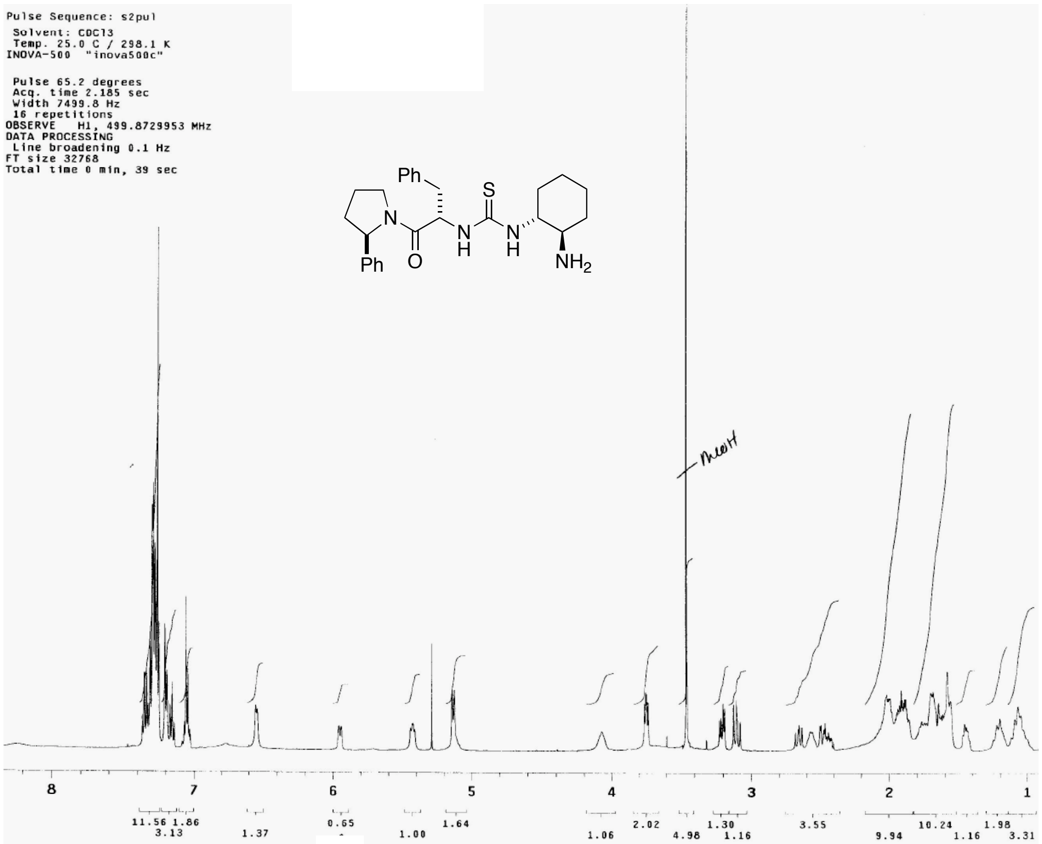

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
352 repetitions
OBSERVE C13, 125.6931684 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
O
BnN
H
Cl
N
H
170.782
137.477
137.222
128.541
128.337
127.878
127.267
124.062
123.145
120.464
119.387
116.044
111.565
77.255
77.000
76.745
53.323
47.715
32.348
30.164
17.724
ppm

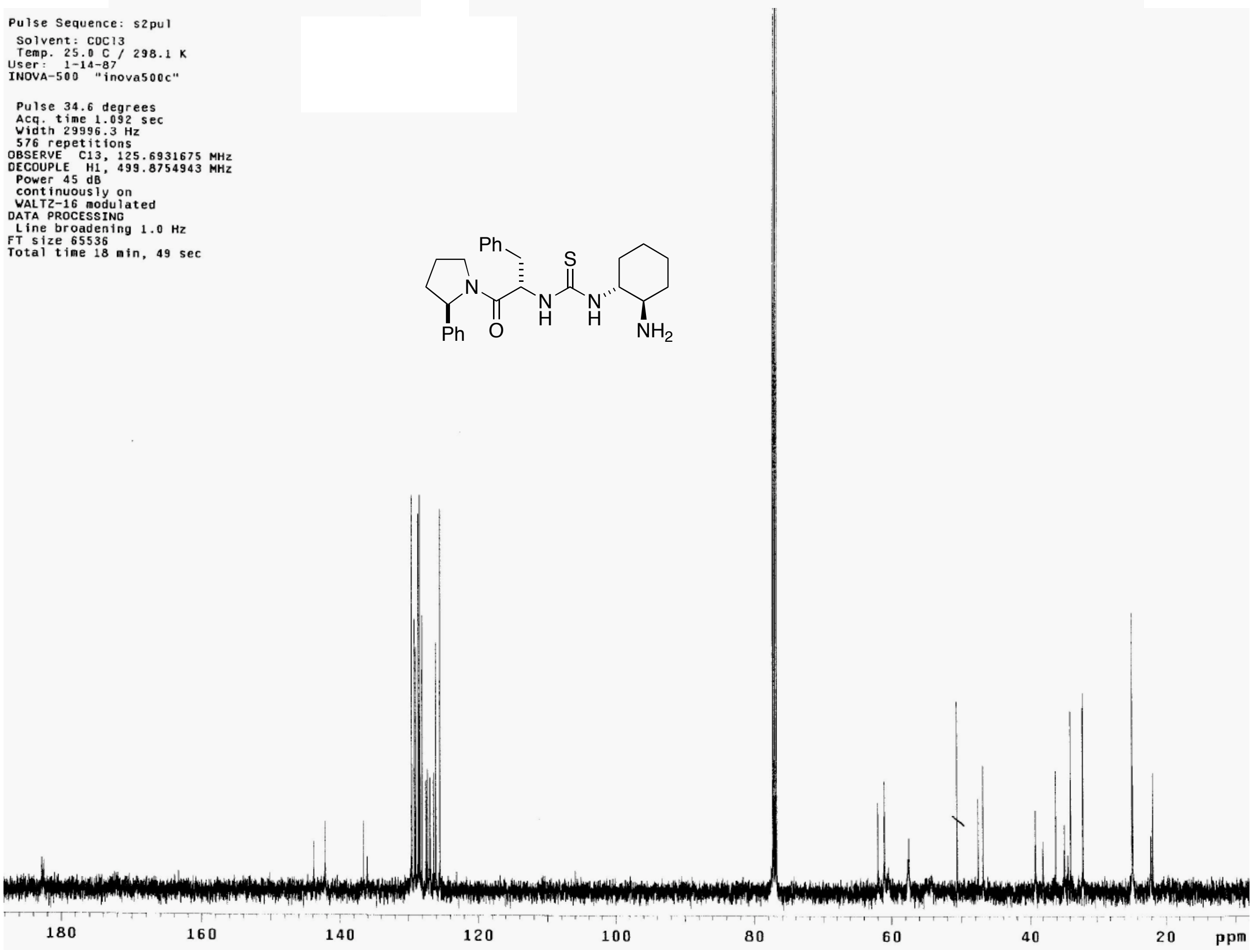

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 70.3 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729953 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
HN
O
H
N
H
8 7 6 5 4 3 2
1.12 0.90 0.87 0.88 1.95 1.12 1.00 3.52 1.17

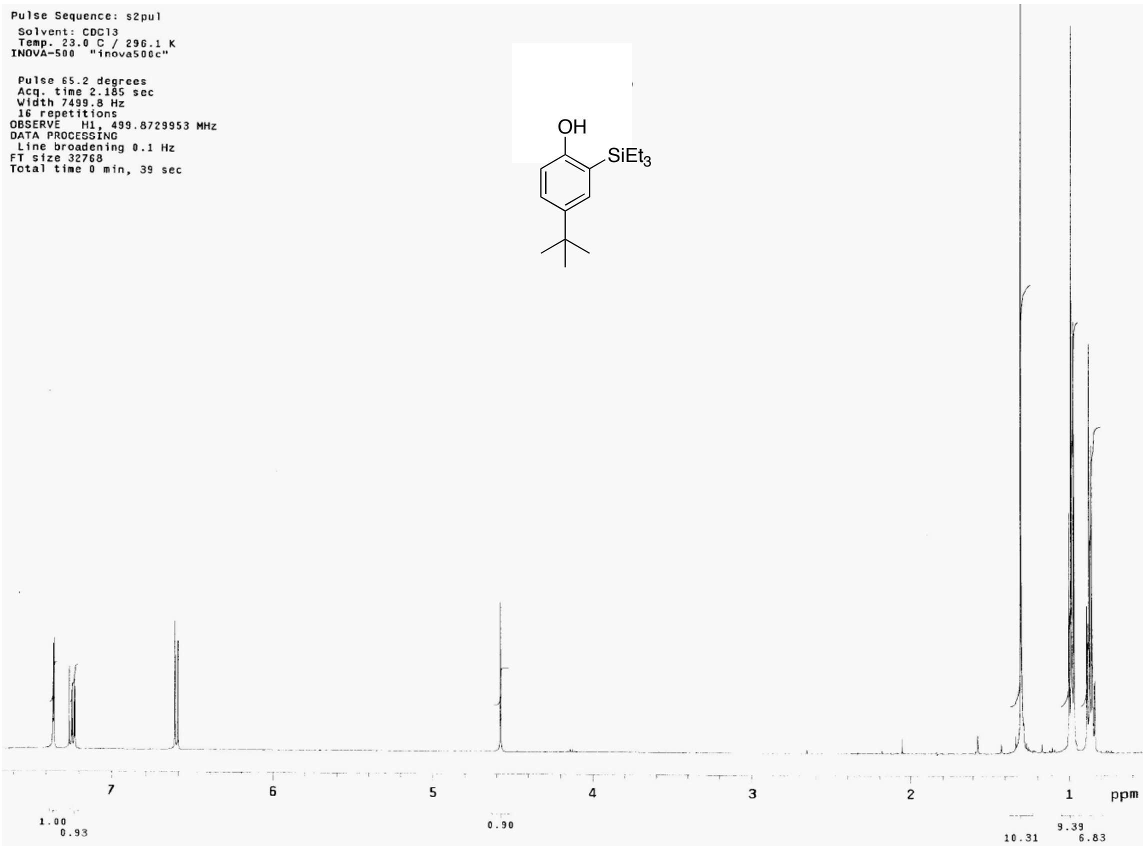

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 37.5 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
224 repetitions
OBSERVE C13, 125.6931684 MHz
DECOUPLE H1, 499.8754943 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
HN
O
H
N
H
178.298
136.851
125.118
122.518
121.105
119.765
118.789
117.136
111.587
77.248
77.000
76.745
51.546
30.455
29.457
180 160 140 120 100 80 60 40

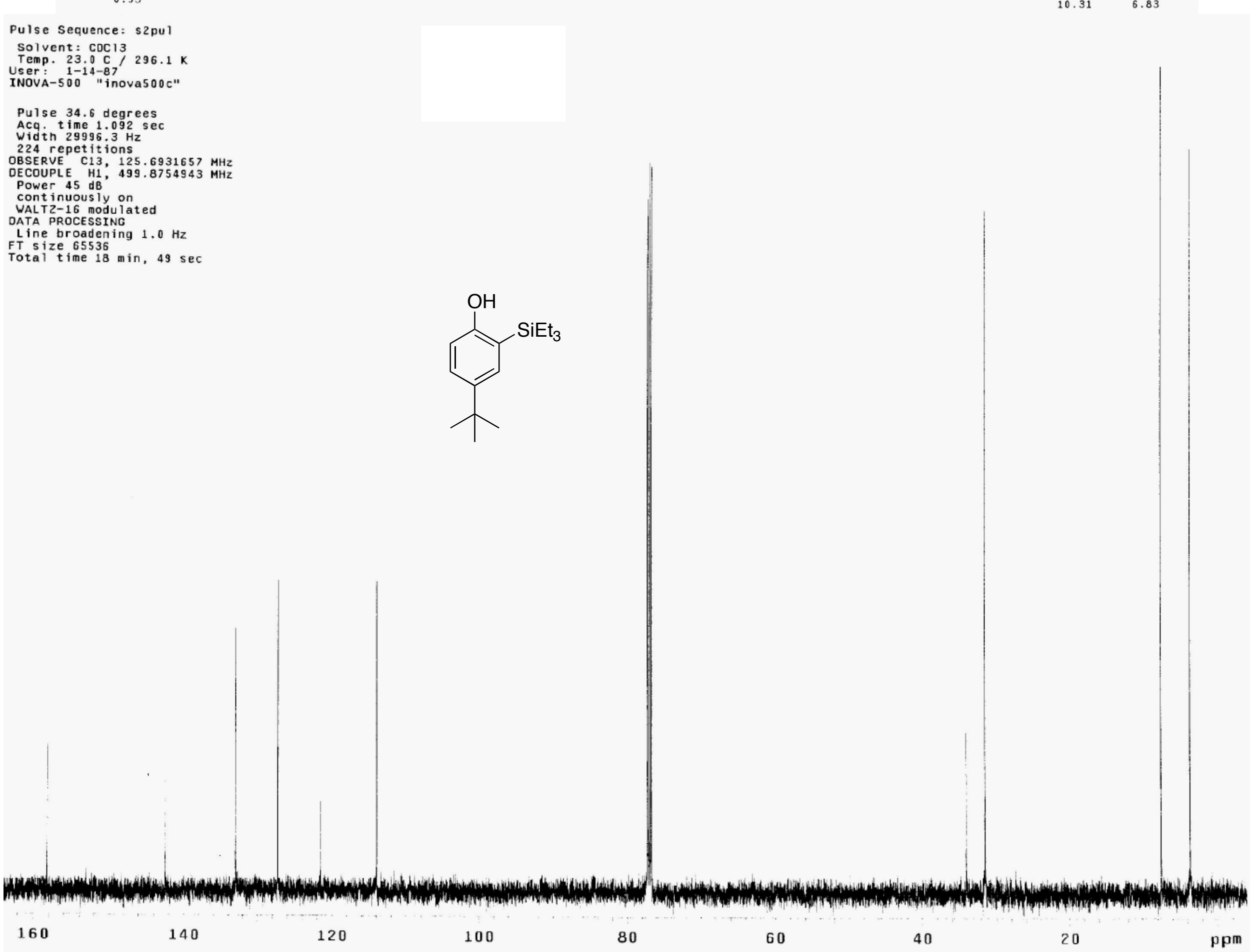

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
Ph
N
NHBoc
Ph
O
7
6
5
4
3
2
20.13
2.13
1.09
0.97
2.23
0.98
1.00
3.88
2.48
1.08
3.97
5.33
2.35
12.17
9.95
1.39

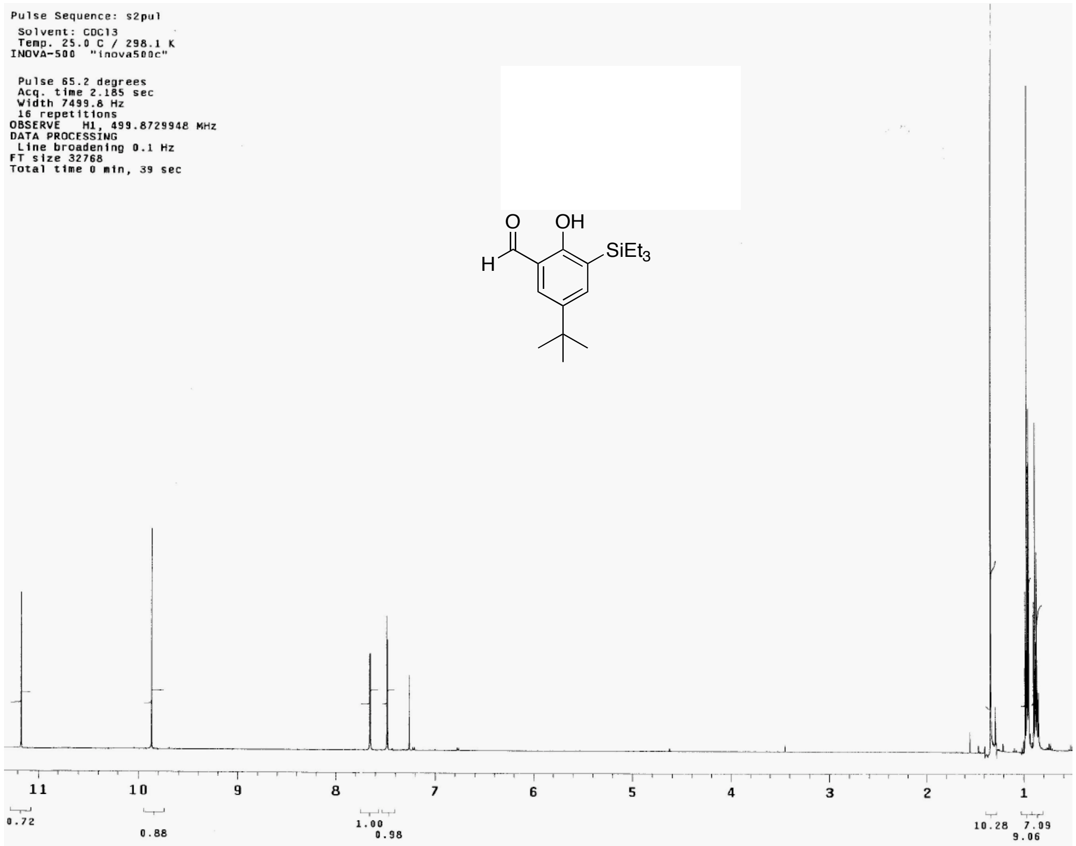

Solvent: CDCl3
Temp. 298.1 K
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
480 repetitions
OBSERVE C13, 125.6931345 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
Ph
N
NHBoc
Ph
O
160
140
120
100
80
60
40
20
ppm

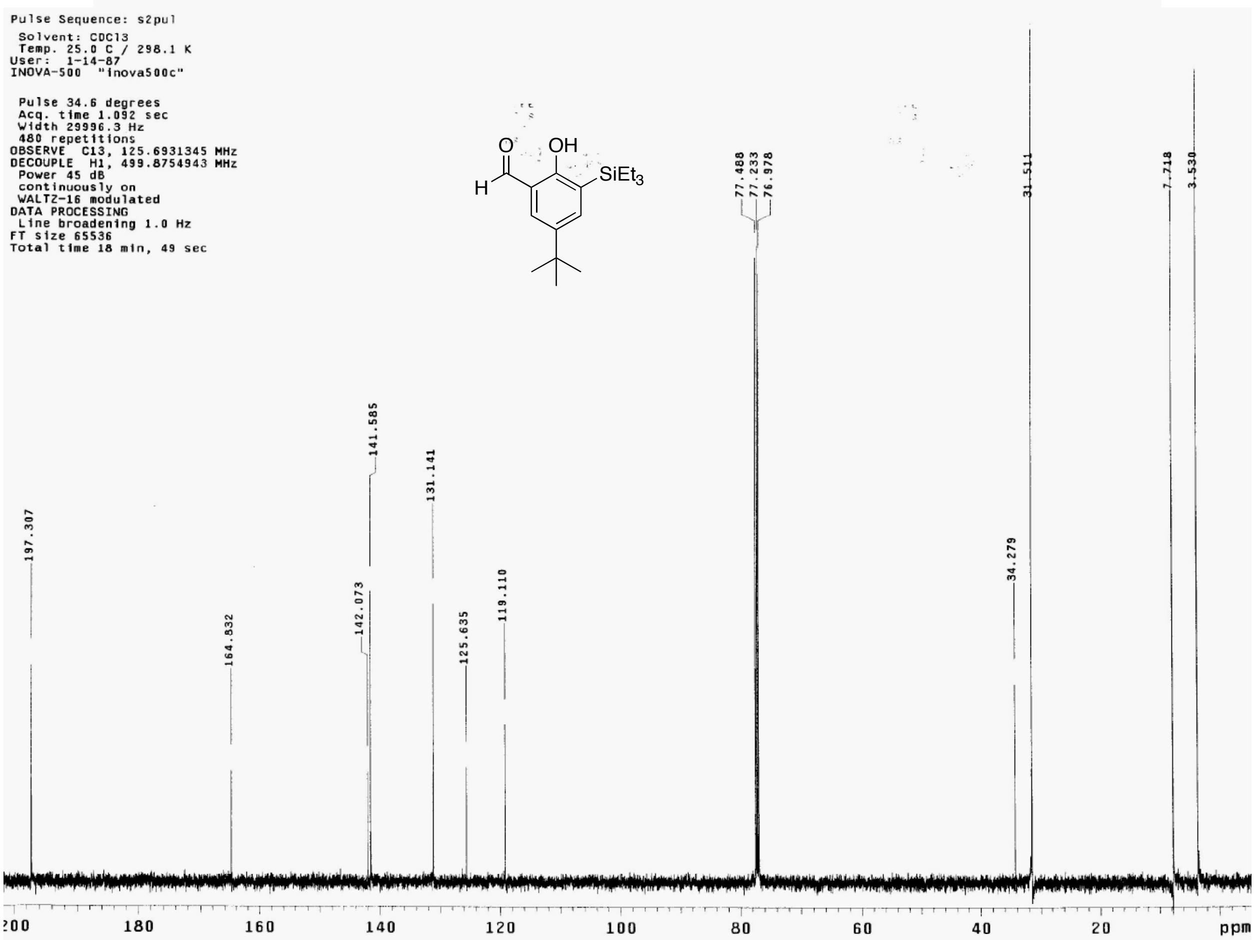

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729953 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
MeOH
8
7
6
5
4
3
2
1
11.56 1.86
3.13
1.37
0.65
1.00
1.64
1.06
2.02
4.98
1.30
1.16
3.55
9.94
10.24
1.16
1.98
3.31

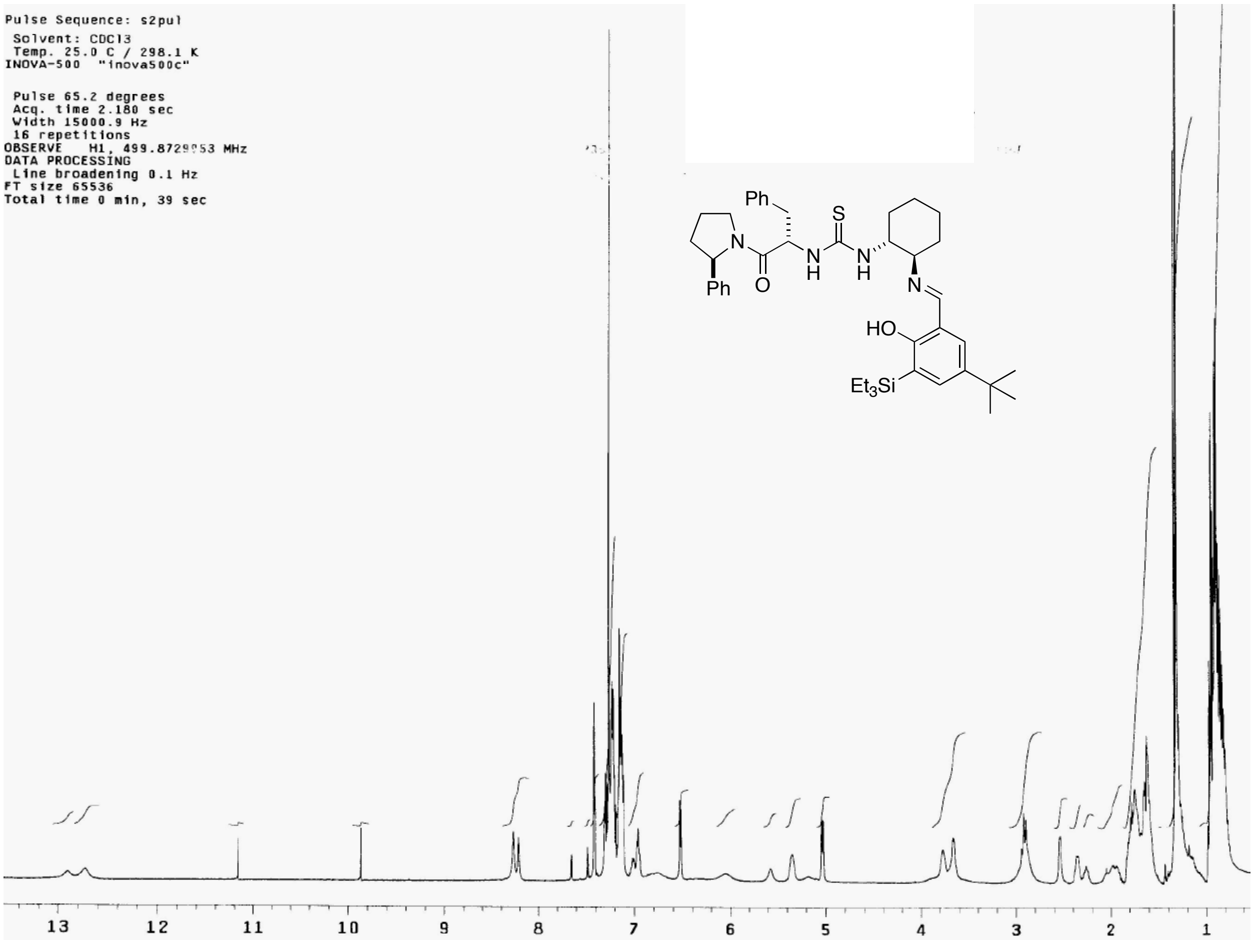

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
576 repetitions
OBSERVE C13, 125.6931675 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
180
160
140
120
100
80
60
40
20
ppm

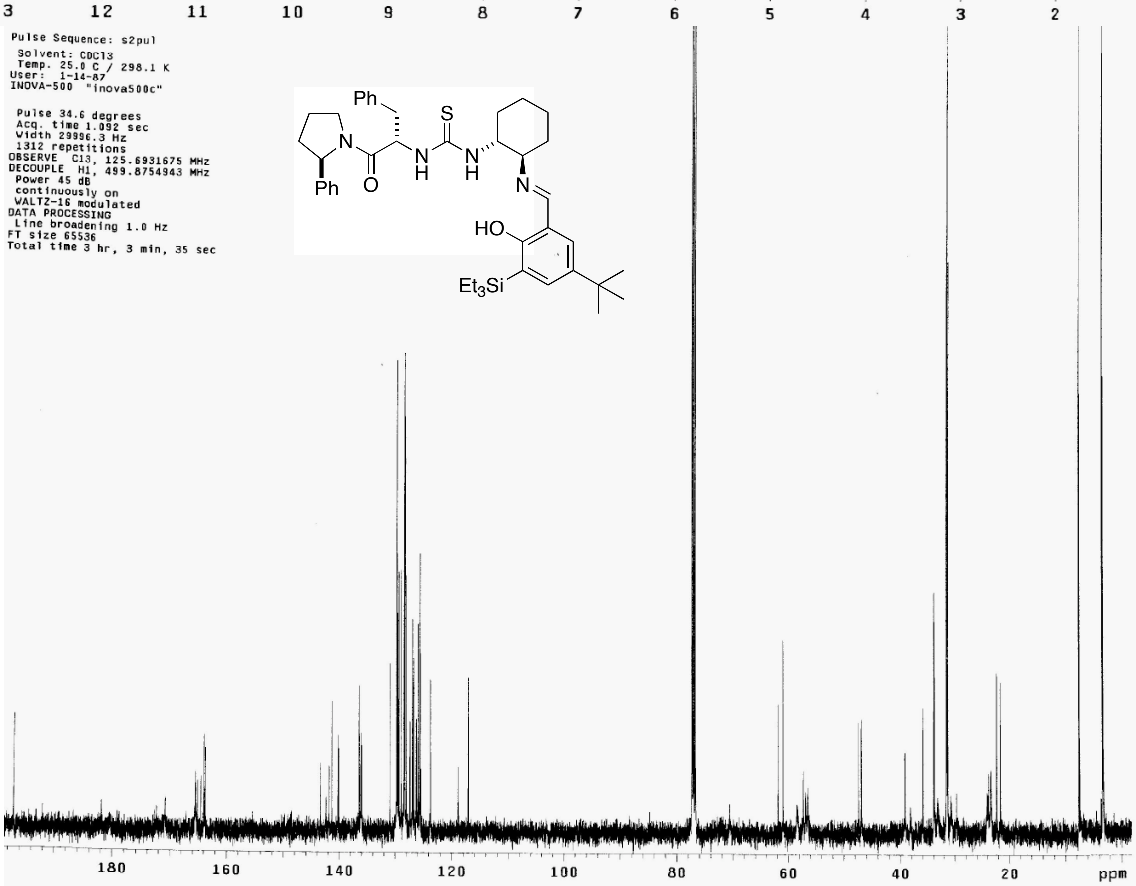

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 23.0 C / 296.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729953 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
OH
SiEt3
7 6 5 4 3 2 1 ppm
1.00
0.93
0.90
10.31
9.39
6.83


Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 23.0 C / 296.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
224 repetitions
OBSERVE C13, 125.6931657 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
OH
SiEt3
160 140 120 100 80 60 40 20 ppm

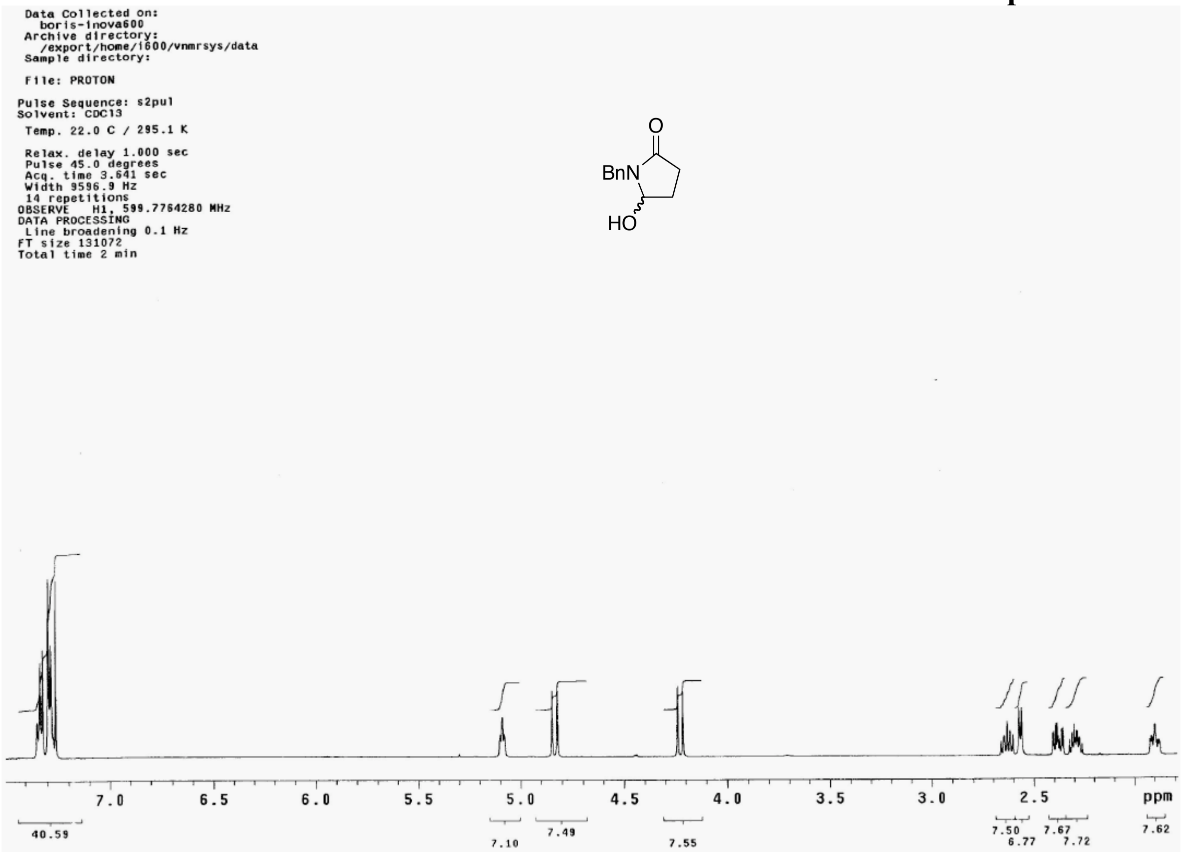

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
16 repetitions
OBSERVE H1, 499.8729948 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 39 sec
O
OH
H
SiEt3
11 10 9 8 7 6 5 4 3 2 1
0.72
0.88
1.00
0.98
10.28
7.09
9.06

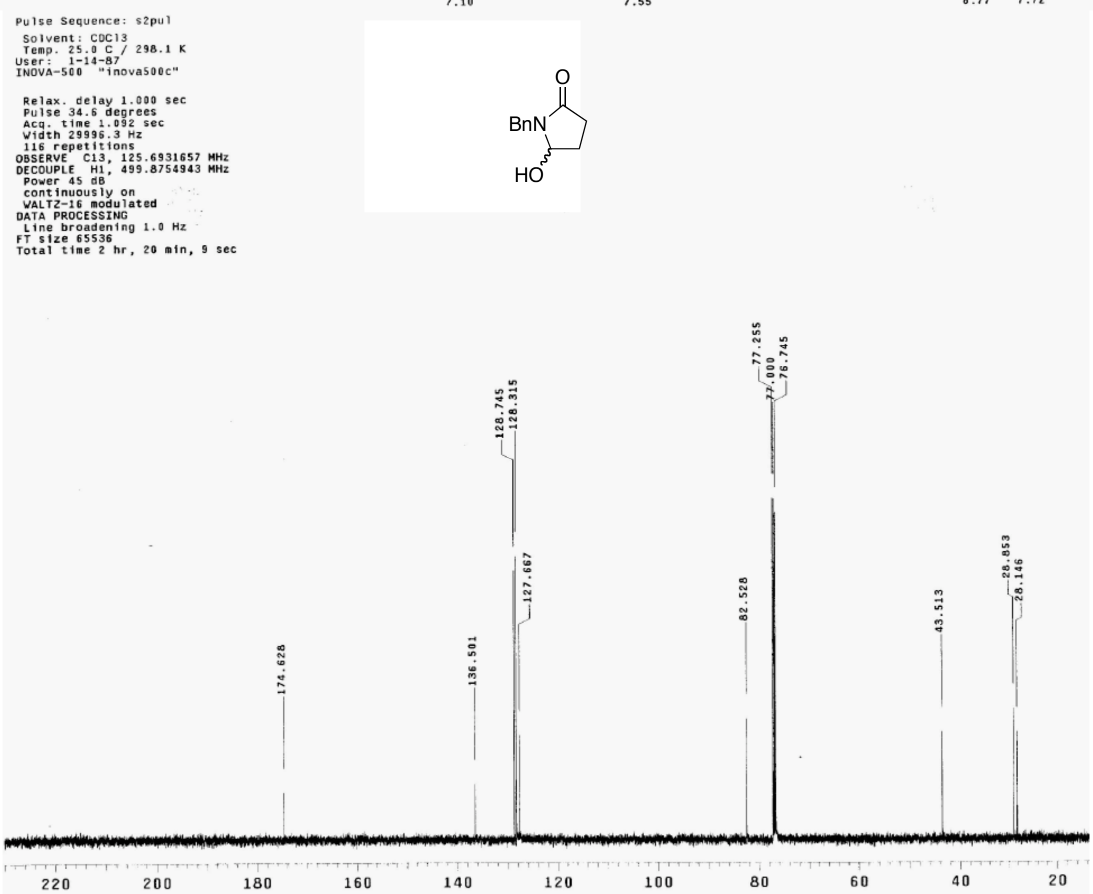

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
480 repetitions
OBSERVE C13, 125.6931345 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 min, 49 sec
O
OH
H
SiEt3
77.488
77.233
76.978
31.511
7.718
3.530
141.585
131.141
197.307
142.073
164.832
125.635
119.110
34.279
200 180 160 140 120 100 80 60 40 20 ppm

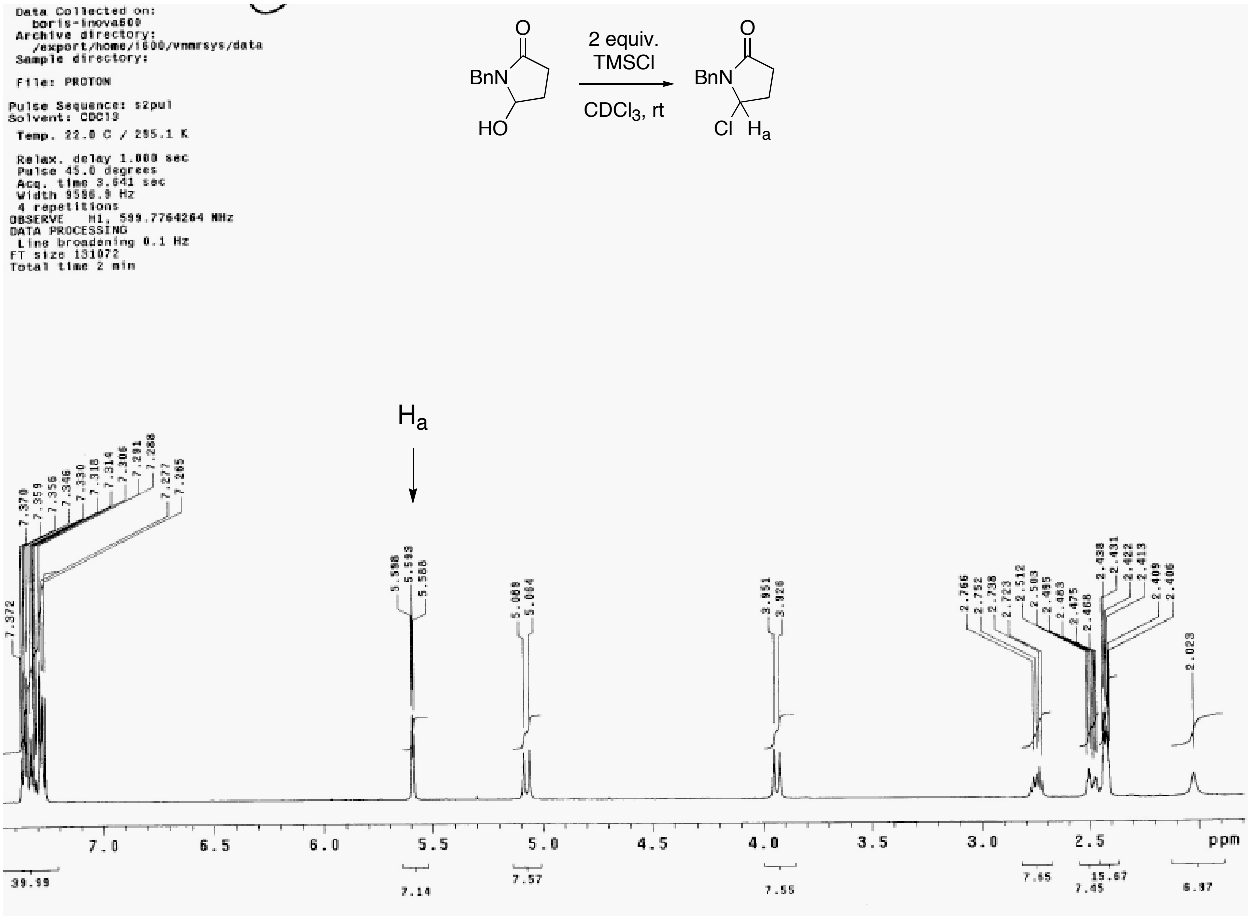

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.180 sec
Width 15000.9 Hz
16 repetitions
OBSERVE H1, 499.8729953 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 0 min, 39 sec
Ph
S
N
N
N
H
H
O
Ph
N
HO
Et3Si
13
12
11
10
9
8
7
6
5
4
3
2
1

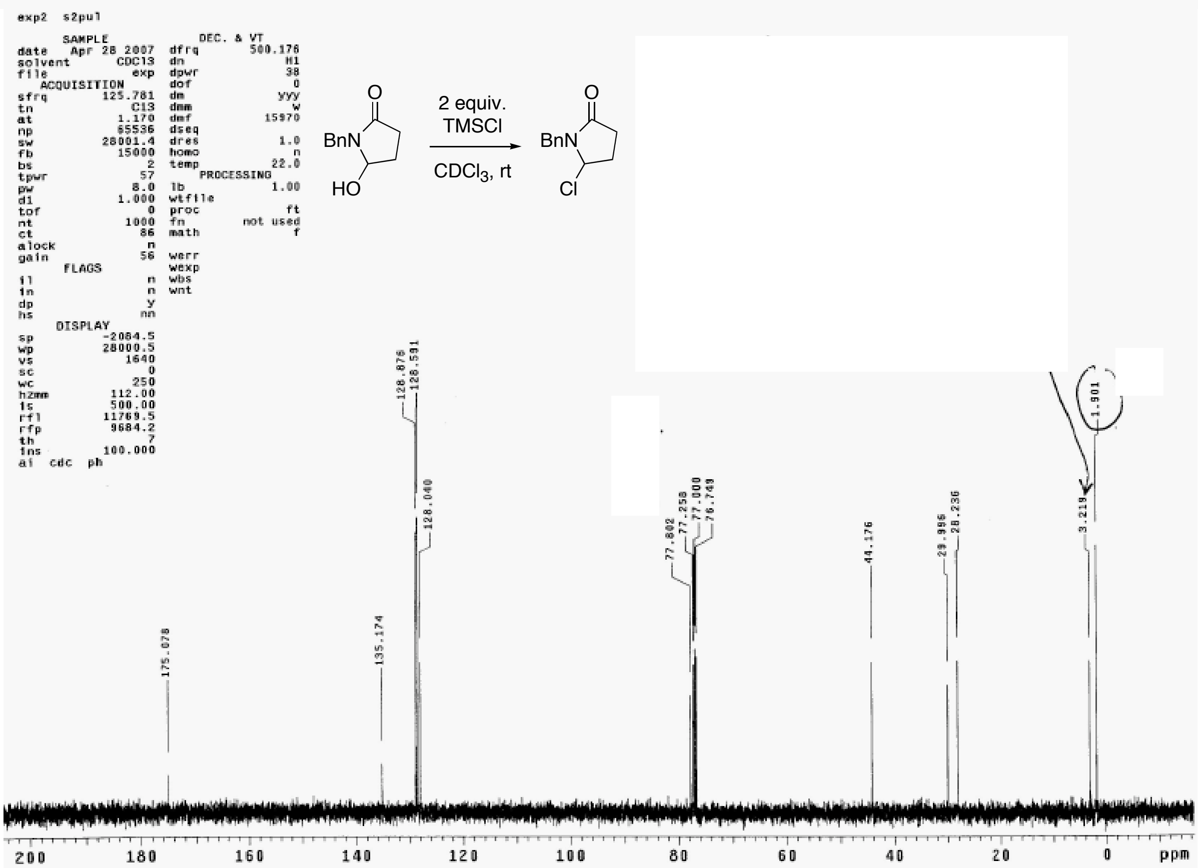

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "inova500c"
Pulse 34.6 degrees
Acq. time 1.092 sec
Width 29996.3 Hz
1312 repetitions
OBSERVE C13, 125.6931675 MHz
DECOUPLE H1, 499.8754943 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 3 hr, 3 min, 35 sec
Ph
S
N
N
N
H
H
O
Ph
N
HO
Et3Si
180
160
140
120
100
80
60
40
20
ppm

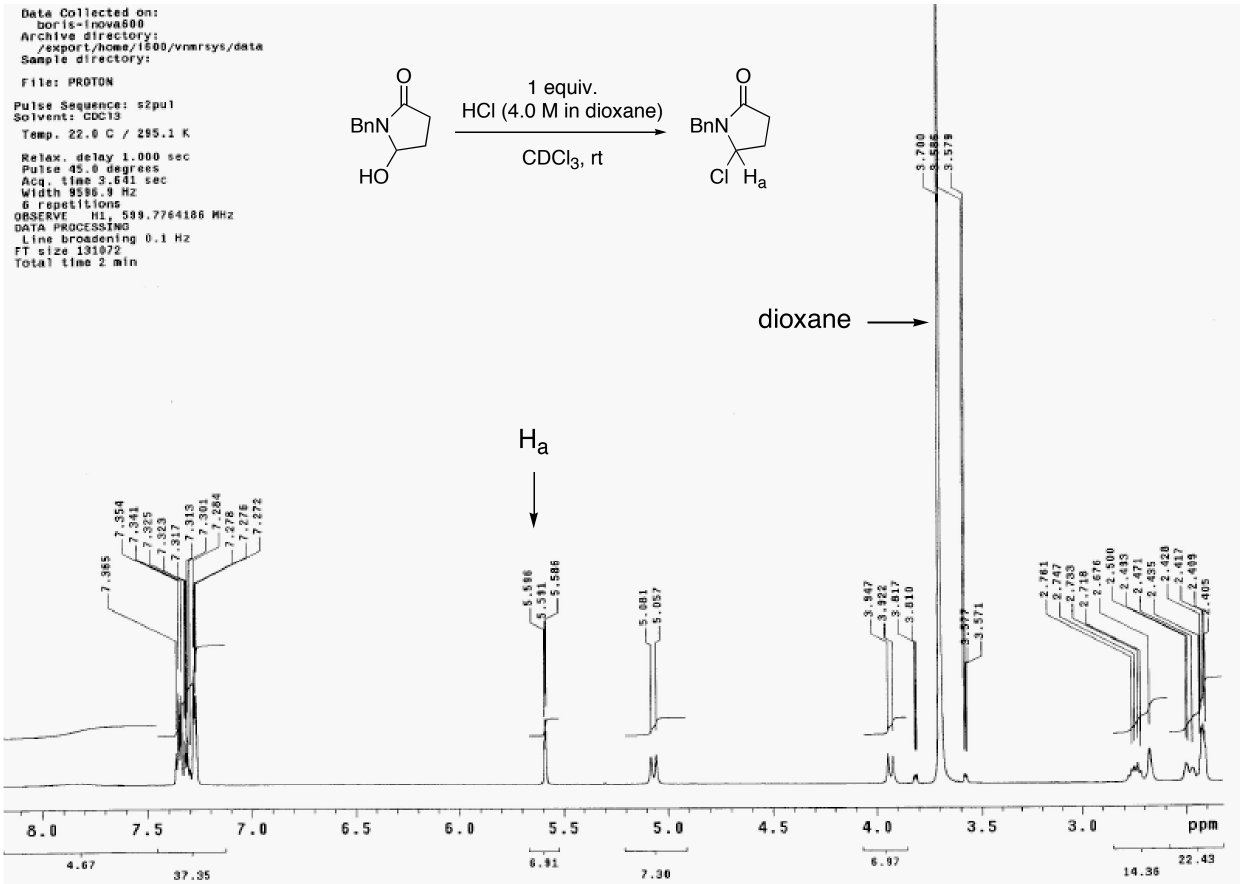
H. ^{1}H and ^{13}C NMR of chlorolactam intermediate 7 and related compounds

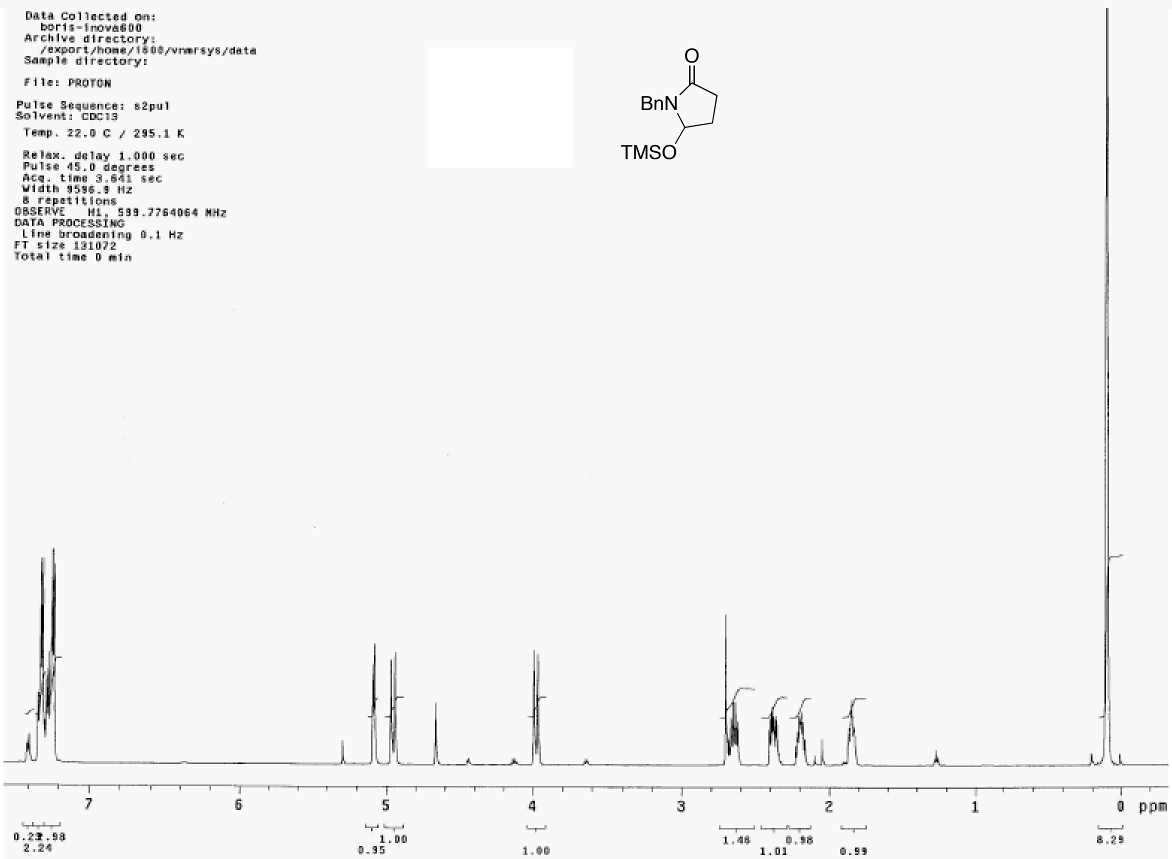


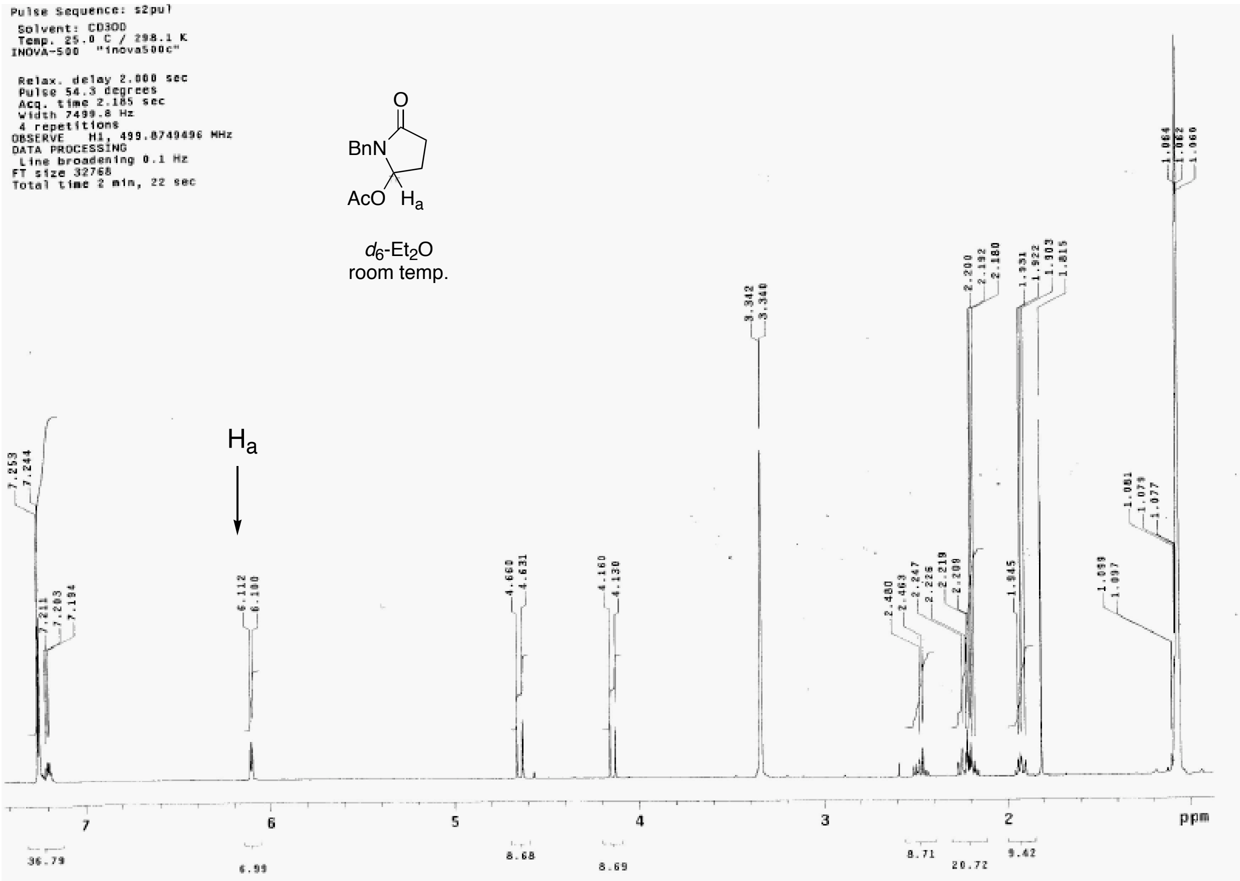


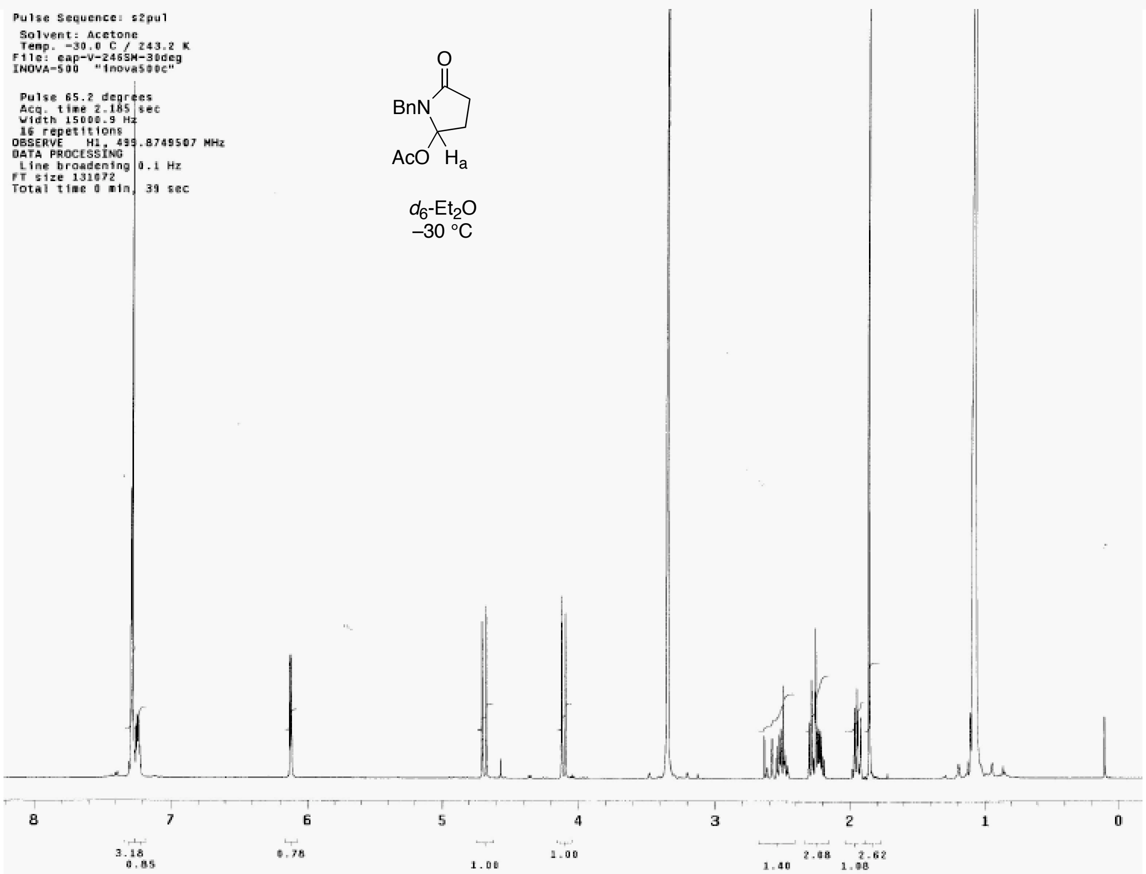

Data Collected on:
boris-inova600
Archive directory:
/export/home/i600/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 22.0 C / 295.1 K
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.641 sec
Width 9596.9 Hz
4 repetitions
OBSERVE H1, 599.7764264 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 131072
Total time 2 min
BnN
HO
O
2 equiv.
TMSCl
CDCl3, rt
BnN
Cl
Ha
O
Ha
ppm

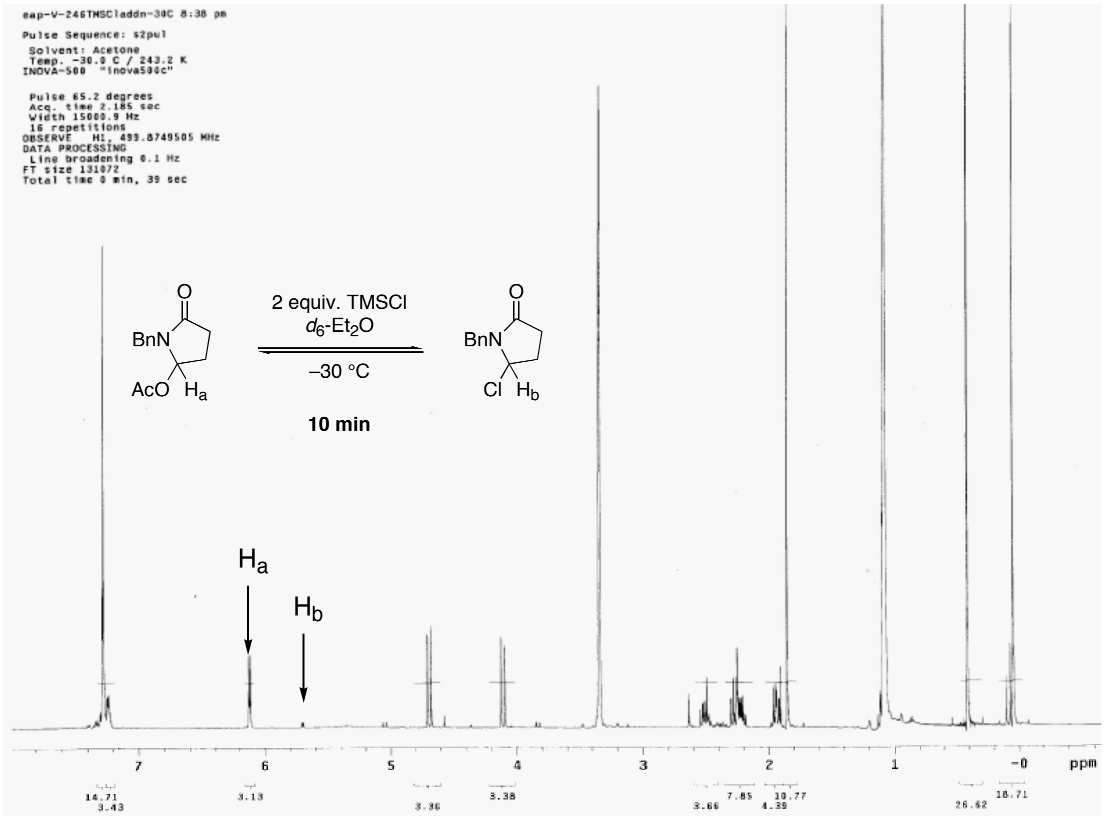

exp2 s2pul
SAMPLE
date Apr 28 2007
solvent CDCl3
file exp
ACQUISITION
DEC. & VT
PROCESSING
FLAGS
DISPLAY
BnN
HO
O
2 equiv.
TMSCl
CDCl3, rt
BnN
Cl
O
ppm

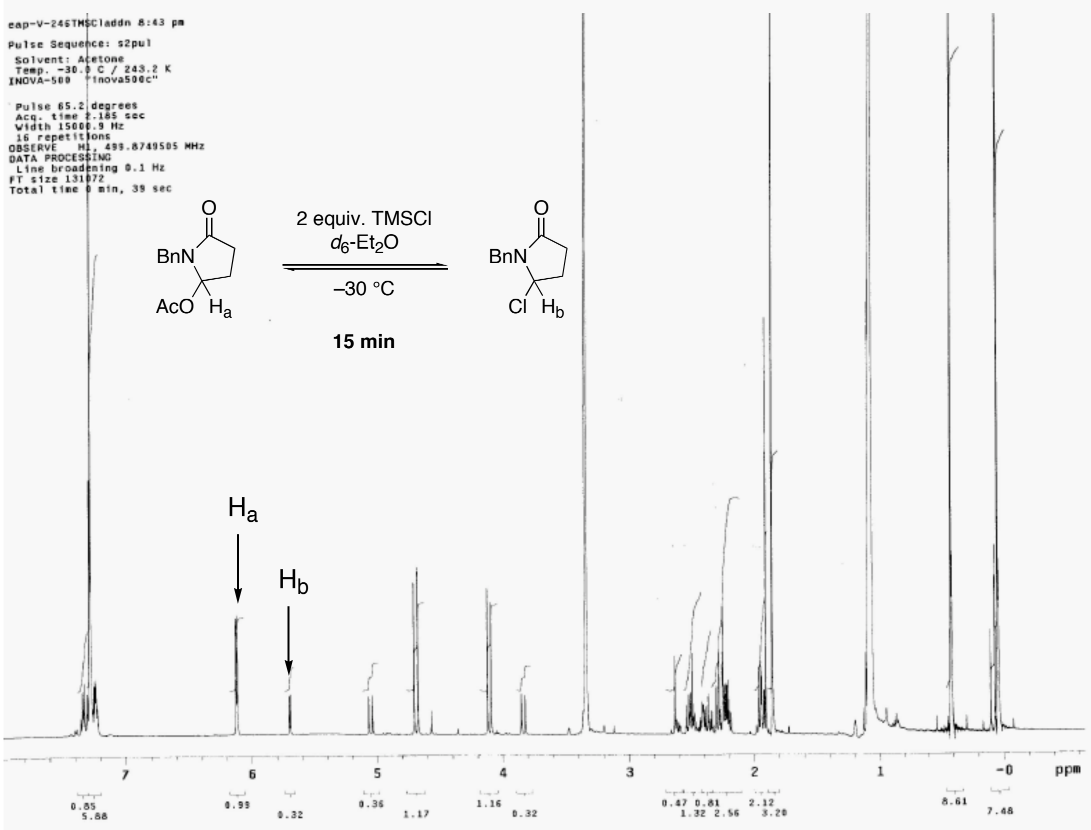

Data Collected on:
boris-inova600
Archive directory:
/export/home/i600/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 22.0 C / 295.1 K
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.641 sec
Width 9596.9 Hz
6 repetitions
OBSERVE H1, 599.7764186 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 131072
Total time 2 min
1 equiv.
HCl (4.0 M in dioxane)
CDCl3, rt
BnN
HO
Cl Ha
dioxane
Ha
ppm

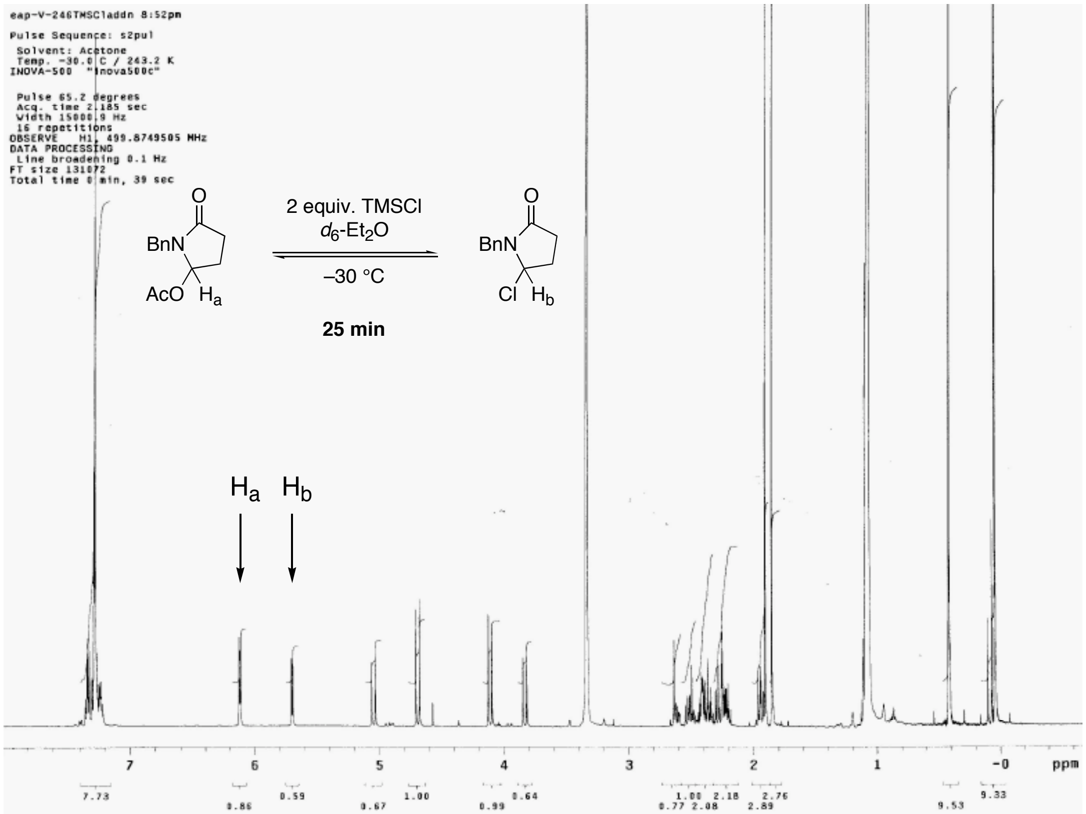

Data Collected on:
boris-inova600
Archive directory:
/export/home/i600/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 22.0 C / 295.1 K
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.641 sec
Width 9596.9 Hz
8 repetitions
OBSERVE H1, 599.7764064 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 131072
Total time 0 min
BnN
TMSO
ppm


Pulse Sequence: s2pul
Solvent: CD3OD
Temp. 25.0 C / 298.1 K
INOVA-500 "inova500c"
Relax. delay 2.000 sec
Pulse 54.3 degrees
Acq. time 2.185 sec
Width 7499.8 Hz
4 repetitions
OBSERVE H1, 499.8749496 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 2 min, 22 sec
BnN
O
AcO
Ha
d6-Et2O
room temp.
Ha
ppm


Pulse Sequence: s2pul
Solvent: Acetone
Temp. -30.0 C / 243.2 K
File: eap-V-246SM-30deg
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 15000.9 Hz
16 repetitions
OBSERVE H1, 499.8749507 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 131072
Total time 0 min, 39 sec
BnN
O
AcO
Ha
d6-Et2O
–30 °C


eap-V-246TMSCladdn-30C 8:38 pm
Pulse Sequence: s2pul
Solvent: Acetone
Temp. -30.0 C / 243.2 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 15000.9 Hz
16 repetitions
OBSERVE H1, 499.8749505 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 131072
Total time 0 min, 39 sec
2 equiv. TMSCl
d6-Et2O
–30 °C
10 min
BnN
AcO Ha
Cl Hb
Ha
Hb
ppm


eap-V-246TMSCladdn 8:43 pm
Pulse Sequence: s2pul
Solvent: Acetone
Temp. -30.0 C / 243.2 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 15000.9 Hz
16 repetitions
OBSERVE H1, 499.8749505 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 131072
Total time 0 min, 39 sec
2 equiv. TMSCl
d6-Et2O
–30 °C
15 min
BnN
AcO Ha
Cl Hb
Ha
Hb
ppm


eap-V-246TMSCladdn 8:52pm
Pulse Sequence: s2pul
Solvent: Acetone
Temp. -30.0 C / 243.2 K
INOVA-500 "inova500c"
Pulse 65.2 degrees
Acq. time 2.185 sec
Width 15000.9 Hz
16 repetitions
OBSERVE H1, 499.8749505 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 131072
Total time 0 min, 39 sec
BnN
AcO Ha
2 equiv. TMSCl
d6-Et2O
–30 °C
25 min
BnN
Cl Hb
Ha
Hb
ppm