Enantioselective Indoline Synthesis by a Conjugate Addition/Asymmetric Protonation/Aza-Prins Cyclization Cascade

Blake E. Daniels[‡], Jane Ni[‡], and Sarah E. Reisman*

The Warren and Katharine Schlinger Laboratory of Chemistry and Chemical Engineering
Division of Chemistry and Chemical Engineering, California Institute of Technology, Pasadena, California 91125
reisman@caltech.edu

Supporting Information 1 (Experimental Procedures):

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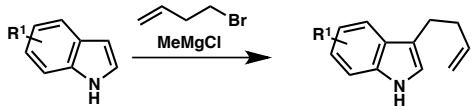
General Considerations. Unless otherwise stated, reactions were performed under a nitrogen atmosphere using freshly dried solvents. Tetrahydrofuran (THF), methylene chloride (CH_2Cl_2), toluene (PhMe), and dimethylformamide (DMF) were dried by passing through activated alumina columns. Dimethylformamide was dried over activated molecular sieves. All other commercially obtained reagents were used as received unless specifically indicated. (*R*)-BINOL was obtained from Alfa Aesar. Reactions were monitored by thin-layer chromatography using EMD/Merck silica gel 60 F254 pre-coated plates (0.25 mm) and were visualized by UV, *p*-anisaldehyde, CAM, or $KMnO_4$ staining. Flash column chromatography was performed either as described by Still et al.[1] using silica gel (particle size 0.032-0.063) purchased from Silicycle, or pre-packaged RediSep®Rf columns on a CombiFlash Rf system (Teledyne ISCO Inc.). Diastereomeric ratios were determined by integration of NMR spectra. Optical rotations were measured on a Jasco P-2000 polarimeter using a 100 mm path-length cell at 589 nm. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Mercury 300 (at 300 MHz and 75 MHz, respectively), a Varian 400 (at 400 MHz and 100 MHz, respectively) or a Varian Inova 500 (at 500 MHz and 125 MHz, respectively), and are reported relative to internal chloroform (^{1}H, δ = 7.26, ^{13}C, δ = 77.0). Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm) (multiplicity, coupling constant (Hz), integration). Multiplicity and qualifier abbreviations are as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad, app = apparent. IR spectra were recorded on a Perkin Elmer Paragon 1000 spectrometer and are reported in frequency of absorption (cm^{-1}). Preparative HPLC was performed with either an Agilent 1100 or 1200 Series HPLC utilizing an Agilent Zorbax RX-SIL 5μm column (9.4 x 250 mm). Analytical SFC was performed with a Mettler SFC supercritical CO_2 analytical chromatography system with Chiralcel AD-H and OJ-H columns (4.6 mm x 25 cm). Melting points were determined using a Büchi B-545 capillary melting point apparatus and the values reported are uncorrected. HRMS were acquired using either an Agilent 6200 Series TOF with an Agilent G1978A Multimode source in electrospray ionization (ESI), atmospheric pressure chemical ionization (APCI) or mixed (MM) ionization mode.

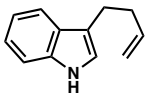
Indole Substrates for Conjugate Addition/Asymmetric Protonation/Prins Cyclization Cascade

General Procedure A: Preparation of 3-(but-3-en-1-yl) N-H indoles



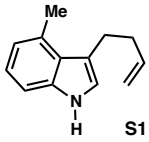
The preparation of 3-(but-3-en-1-yl)-indole derivatives was adapted from Youn et al.[2] To a solution of the indole substrate (1.00 equiv) in benzene was added a solution of MeMgCl (1.06 equiv, 3.0 M solution in THF) at rt. After 10 min, 4-bromobutene (0.86 equiv) was added and the reaction mixture was heated to reflux. After 27 h, the reaction mixture was cooled and quenched with sat. NH_4Cl. The organic phase was separated and the aqueous phase was extracted twice with EtOAc. The organic layer was washed with water and brine, dried over $MgSO_4$, and concentrated in vacuo. The residue was purified by column chromatography on silica gel.

3-(but-3-en-1-yl)-*1H*-indole



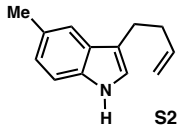
Prepared from indole (1.75 g, 15 mmol) according to General Procedure A to yield 3-(but-3-en-1-yl)-1*H*-indole in 41% yield (1.07 g). Spectral data of 3-(but-3-en-1-yl)-*1H*-indol were found to be in agreement with those reported in the literature.[3]

4-methyl-3-(but-3-en-1-yl)indole (S1)



Prepared from 4-methylindole (2.6 g, 19.8 mmol) according to General Procedure A to yield 4-methyl-3-(but-3-en-1-yl)indole (**S1**) in 27% yield (983 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (s, 1H), 7.19 (d, *J* = 8.1 Hz, 1H), 7.06 (t, *J* = 7.1 Hz, 1H), 6.95 (dt, *J* = 2.2, 1.0 Hz, 1H), 6.84 (dt, *J* = 7.1, 0.9 Hz, 1H), 5.97 (ddt, *J* = 16.8, 10.2, 6.5 Hz, 1H), 5.11 (dq, *J* = 17.1, 1.7 Hz, 1H), 5.02 (ddt, *J* = 10.2, 2.2, 1.2 Hz, 1H), 3.10 – 2.98 (m, 2H), 2.72 (s, 3H), 2.51 – 2.43 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.66, 136.75, 130.97, 125.93, 121.96, 121.29, 120.91, 117.25, 114.72, 108.98, 35.48, 26.82, 20.31; IR (NaCl/thin film) 3409, 1638, 1412, 1341, 1113, 910, 747 cm^{-1}; HRMS (ESI) calc'd for $C_{13}H_{15}N$ $[M^*]^+$ 185.1199, found 185.1200.

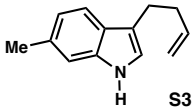
5-methyl-3-(but-3-en-1-yl)indole (S2)



S2

Prepared from 5-methylindole (2g, 15.2 mmol) according to General Procedure A to yield 5-methyl-3-(but-3-en-1-yl)indole (**S2**) in 48% yield (1.34 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82 (s, 1H), 7.42 (dq, *J* = 1.6, 0.8 Hz, 1H), 7.30 – 7.24 (m, 1H), 7.07 – 7.03 (m, 1H), 6.98 (dd, *J* = 2.3, 1.1 Hz, 1H), 5.98 (ddt, *J* = 16.9, 10.2, 6.5 Hz, 1H), 5.13 (dq, *J* = 17.1, 1.7 Hz, 1H), 5.03 (ddt, *J* = 10.2, 2.3, 1.3 Hz, 1H), 2.91 – 2.83 (m, 2H), 2.54 – 2.47 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.87, 134.62, 128.34, 127.73, 123.49, 121.32, 118.58, 115.77, 114.60, 110.71, 34.29, 24.75, 21.55; IR (NaCl/thin film) 3413, 2919, 1640, 1423, 1225, 1091, 995, 911, 792 cm^{-1}; HRMS (ESI) calc'd for $C_{13}H_{15}N$ $[M+H]^+$ 186.1277, found 186.1274.

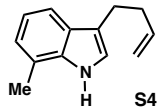
6-methyl-3-(but-3-en-1-yl)indole (S3)



S3

Prepared from 6-methylindole (2.6 g, 19.8 mmol) according to General Procedure A to yield 6-methyl-3-(but-3-en-1-yl)indole **(S3**) in 42% yield (1.54 g). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (s, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.15 (s, 1H), 6.97 (dd, *J* = 8.1, 1.4 Hz, 1H), 6.96 – 6.89 (m, 1H), 5.95 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.10 (dq, *J* = 17.2, 1.7 Hz, 1H), 5.01 (ddt, *J* = 10.2, 2.3, 1.3 Hz, 1H), 2.89 – 2.81 (m, 2H), 2.53 – 2.44 (m, 5H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.88, 136.77, 131.71, 125.40, 120.92, 120.51, 118.60, 116.13, 114.63, 111.04, 34.34, 24.85, 21.74; IR (NaCl/thin film) 3412, 2918, 2100, 1639, 1455, 1339, 1229, 1089, 995, 910, 800 cm^{-1}; HRMS (ESI) calc'd for $C_{13}H_{15}N$ $[M+H]^+$ 186.1277, found 186.1277.

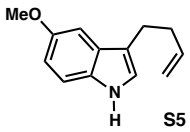
7-methyl-3-(but-3-en-1-yl)indole (S4)



S4

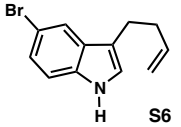
Prepared from 7-methylindole (2.6 g, 19.8 mmol) according to General Procedure A to yield 7-methyl-3-(but-3-en-1-yl)indole (**S4**) in 38% yield (1.39 g). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (s, 1H), 7.49 (d, *J* = 7.8, 1H), 7.10 – 7.04 (m, 1H), 7.04 – 7.00 (m, 2H), 5.96 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.11 (dq, *J* = 17.1, 1.7 Hz, 1H), 5.02 (ddt, *J* = 10.2, 2.3, 1.2 Hz, 1H), 2.91 – 2.85 (m, 2H), 2.55 – 2.45 (m, 5H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.85, 135.89, 127.05, 122.47, 120.90, 120.23, 119.40, 116.81, 116.70, 114.67, 34.35, 24.91, 16.64; IR (NaCl/thin film) 3419, 2919, 1639, 1433, 1342, 1065, 911, 783, 746 cm^{-1}; HRMS (ESI) calc'd for $C_{13}H_{15}N$ $[M+H]^+$ 186.1277, found 186.1277.

5-methoxy-3-(but-3-en-1-yl)indole (S5)



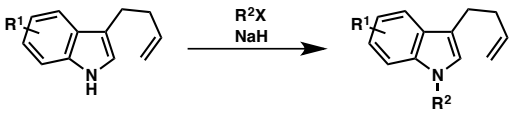
Prepared from 5-methoxyindole (3 g, 20.4 mmol) according to General Procedure A to yield 5-methoxy-3-(but-3-en-1-yl)indole (**S5**) in 44% yield (1.80 g). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (s, 1H), 7.25 (d, *J* = 8.5 Hz, 1H), 7.05 (d, *J* = 2.5 Hz, 1H), 6.98 (dd, *J* = 2.3, 1.1 Hz, 1H), 6.86 (dd, *J* = 8.8, 2.5 Hz, 1H), 5.96 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.10 (dq, *J* = 17.1, 1.7 Hz, 1H), 5.01 (ddt, *J* = 10.2, 2.2, 1.2 Hz, 1H), 3.88 (s, 3H), 2.87 – 2.79 (m, 2H), 2.48 (tdt, *J* = 7.9, 6.5, 1.5 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.83, 138.82, 131.46, 127.89, 122.05, 115.99, 114.70, 112.03, 111.77, 100.84, 55.98, 34.14, 24.76; IR (NaCl/thin film) 3414, 2919, 1584, 1482, 1291, 1213, 1172, 1059, 1028, 914, 794 cm^{-1}; HRMS (ESI) calc'd for $C_{13}H_{15}NO$ $[M+H]^+$ 202.1226, found 202.1223.

5-bromo-3-(but-3-en-1-yl)-indole (S6)



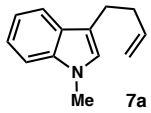
Prepared from 5-bromoindole (4.1 g, 21.1 mmol) according to General Procedure A to yield 5-bromo-3-(but-3-en-1-yl)-indole (**S6**) in 25% yield (1.34 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 (s, 1H), 7.73 (d, *J* = 1.8 Hz, 1H), 7.28 – 7.25 (m, 1H), 7.22 (d, *J* = 8.6 Hz, 1H), 7.00 (d, *J* = 2.4 Hz, 1H), 5.91 (ddt, *J* = 16.8, 10.2, 6.6 Hz, 1H), 5.09 (dq, *J* = 17.1, 1.7 Hz, 1H), 5.01 (ddt, *J* = 10.2, 2.3, 1.3 Hz, 1H), 2.85 – 2.78 (m, 2H), 2.46 (tdt, *J* = 7.8, 6.5, 1.5 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.41, 134.86, 129.32, 124.72, 122.43, 121.57, 116.02, 114.94, 112.47, 112.45, 34.13, 24.52; IR (NaCl/thin film) 3429, 2921, 1639, 1458, 1224, 1093, 995, 912, 792 cm^{-1}; HRMS (MM) calc'd for $C_{12}H_{12}BrN$ $[M+K]^-$ 287.9796, found 287.9786.

General Procedure B: Preparation of 3-(but-3-en-1-yl) N-substituted indoles



The appropriate 3-(but-3-en-1-yl) N-H indole was dissolved in DMF. NaH (2.0 equiv, 60% dispersion in mineral oil) was added at rt, followed by either methyl iodide or allyl bromide or benzyl bromide (2.5 equiv). The reaction was stirred at room temperature until consumption of starting material of observed by TLC. The reaction was diluted with ethyl acetate, and quenched with water (10× volume of DMF). The aqueous layer was extracted 3× with ethyl acetate. The combined organic layers were washed with water, dried ($MgSO_4$), filtered, and concentrated under reduced pressure. The crude residue was purified by column chromatography on silica gel.

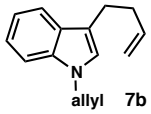
3-(but-3-en-1-yl)-1-methylindole (7a)



7a

Prepared from 3-(but-3-en-1-yl)-1*H*-indole (1.07 g, 6.2 mmol) according to General Procedure B to yield **7a** in 95% yield (1.09 g). Spectral data of **7a** were found to be in agreement with those reported in the literature.[4]

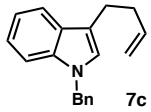
3-(but-3-en-1-yl)-1-allylindole (7b)



7b

Prepared from 3-(but-3-en-1-yl)-1*H*-indole (400 mg, 2.3 mmol) according to General Procedure B to yield **7b** in 73% yield (355 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (dt, *J* = 7.9, 1.0 Hz, 1H), 7.30 (dt, *J* = 8.3, 0.9 Hz, 1H), 7.21 (ddd, *J* = 8.2, 6.9, 1.2 Hz, 1H), 7.12 (ddd, *J* = 8.0, 7.0, 1.0 Hz, 1H), 6.90 (s, 1H), 6.04 – 5.91 (m, 2H), 5.20 (dq, *J* = 10.2, 1.5 Hz, 1H), 5.10 (app ddq, *J* = 17.0, 3.0, 1.6 Hz, 2H), 5.01 (ddt, *J* = 10.2, 2.2, 1.2 Hz, 1H), 4.69 (dt, *J* = 5.4, 1.6 Hz, 2H), 2.91 – 2.84 (m, 2H), 2.49 (dtt, *J* = 9.2, 6.5, 1.4 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.82, 136.42, 133.73, 128.11, 125.03, 121.47, 119.09, 118.69, 117.04, 115.14, 114.64, 109.48, 48.63, 34.46, 24.74; IR (NaCl/thin film) 3075, 2917, 2848, 1640, 1466, 1374, 1326, 1190, 990, 738 cm^{-1}; HRMS (MM) calc'd for $C_{15}H_{17}N$ $[M+H]^+$ 212.1434, found 212.1434.

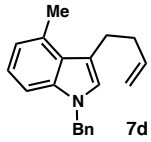
3-(but-3-en-1-yl)-1-benzylindole (7c)



7c

Prepared from 3-(but-3-en-1-yl)-1*H*-indole (1.07 g, 6.2 mmol) according to General Procedure B to yield **7c** in 87% yield (1.42 g). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (dt, *J* = 7.6, 1.0 Hz, 1H), 7.43 – 7.33 (m, 4H), 7.32 – 7.18 (m, 4H), 7.02 (s, 1H), 6.08 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.35 (s, 2H), 5.23 (dq, *J* = 17.2, 1.7 Hz, 1H), 5.14 (ddt, *J* = 10.2, 2.3, 1.3 Hz, 1H), 3.07 – 2.95 (m, 2H), 2.66 – 2.58 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.69, 137.77, 136.60, 128.61, 128.11, 127.39, 126.64, 125.40, 121.59, 119.05, 118.77, 115.31, 114.64, 109.54, 49.69, 34.37, 24.68; IR (NaCl/thin film) 3435, 3061, 2920, 1466, 1356, 1327, 1177, 1014, 911, 798, 738 cm^{-1}; HRMS (MM) calc'd for $C_{19}H_{19}N$ $[M^*]^+$ 261.1512, found 261.1509.

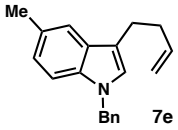
4-methyl-3-(but-3-en-1-yl)-1-benzylindole (7d)



7d

Prepared from 4-methyl-3-(but-3-en-1-yl)indole (**S1**) (871 mg, 5.2 mmol) according to General Procedure B to yield **7d** in 90% yield (1.31 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 – 7.23 (m, 3H), 7.16 – 7.05 (m, 4H), 6.88 (s, 1H), 6.83 (dt, *J* = 6.9, 0.9 Hz, 1H), 5.96 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.26 (s, 2H), 5.10 (dq, *J* = 17.1, 1.6 Hz, 1H), 5.02 (ddt, *J* = 10.2, 2.2, 1.3 Hz, 1H), 3.08 – 3.00 (m, 2H), 2.74 (s, 3H), 2.51 – 2.42 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.62, 137.81, 137.04, 131.15, 128.68, 127.43, 126.68, 126.57, 125.67, 121.64, 120.66, 116.30, 114.72, 107.52, 49.83, 35.61, 26.76, 20.31; IR (NaCl/thin film) 2918, 1496, 1453, 910, 742 cm^{-1}; HRMS (MM) calc'd for $C_{20}H_{21}N$ $[M+H]^+$ 276.1747, found 276.1759.

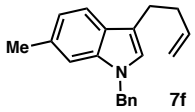
5-methyl-3-(but-3-en-1-yl)-1-benzylindole (7e)



7e

Prepared from 5-methyl-3-(but-3-en-1-yl)indole (**S2**) (963 mg, 5.2 mmol) according to General Procedure B to yield **7e** in 95% yield (1.37 g): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (dt, *J* = 1.7, 0.8 Hz, 1H), 7.31 – 7.23 (m, 3H), 7.14 (d, *J* = 8.3 Hz, 1H), 7.11 – 7.08 (m, *J* = 7.2, 1.4, 0.7 Hz, 2H), 7.00 (dd, *J* = 8.3, 1.6 Hz, 1H), 6.89 (s, 1H), 5.95 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.26 (s, 2H), 5.14 – 5.05 (m, 1H), 5.00 (ddt, *J* = 10.2, 2.3, 1.2 Hz, 1H), 2.89 – 2.81 (m, 2H), 2.51 – 2.45 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.85, 138.01, 135.07, 128.67, 128.36, 128.02, 127.41, 126.66, 125.62, 123.21, 118.79, 114.83, 114.60, 109.32, 49.86, 34.45, 24.74, 21.50; IR (NaCl/thin film) 3434, 2916, 1639, 1487, 1453, 910, 787 cm^{-1}; HRMS (MM) calc'd for $C_{20}H_{21}N$ $[M^*]^+$ 275.1669, found 275.1665.

6-methyl-3-(but-3-en-1-yl)-1-benzylindole (7f)

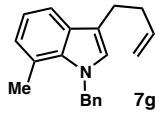


7f

Prepared from 6-methyl-3-(but-3-en-1-yl)indole (**S3**) (963 mg, 5.2 mmol) according to General Procedure B to yield **7f** in 90% yield (1.29 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.0 Hz, 1H), 7.33 – 7.24 (m, 3H), 7.13 – 7.09 (m, 2H), 7.06 (s, 1H), 6.96 (dd, *J* = 8.1, 1.4 Hz, 1H), 6.85 (s, 1H), 5.94 (ddt, *J* = 16.8, 10.2, 6.6 Hz, 1H), 5.26 (s, 2H), 5.09 (dq, *J* = 17.1, 1.7 Hz, 1H), 5.00 (ddt, *J* = 10.2, 2.3, 1.2 Hz, 1H), 2.93 – 2.87 (m, 2H), 2.51 – 2.45 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.84, 138.03, 137.11, 131.46, 128.69, 127.40, 126.66, 126.02, 124.84,

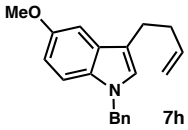
120.60, 118.80, 115.31, 114.61, 109.50, 49.61, 34.48, 24.83, 21.90; IR (NaCl/thin film) 2916, 1622, 1468, 1452, 1325, 1174, 1028, 910, 798 cm^{-1}; HRMS (MM) calc'd for $C_{20}H_{21}N$ $[M+H]^+$ 276.1747, found 276.1755.

7-methyl-3-(but-3-en-1-yl)-1-benzylindole (7g)



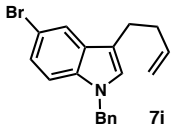
Prepared from 7-methyl-3-(but-3-en-1-yl)indole (**S4**) (963 mg, 5.2 mmol) according to General Procedure B to yield **7g** in 92% yield (1.31 g). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.0 Hz, 1H), 7.31 – 7.20 (m, 3H), 7.02 (dd, *J* = 7.9, 7.1 Hz, 1H), 6.94 – 6.87 (m, 3H), 6.87 (s, 1H), 5.95 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.54 (s, 2H), 5.09 (dq, *J* = 17.1, 1.7 Hz, 1H), 5.00 (ddt, *J* = 10.2, 2.3, 1.2 Hz, 1H), 2.90 – 2.83 (m, 2H), 2.54 – 2.45 (m, 5H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.98, 138.81, 135.37, 129.18, 128.78, 127.46, 127.20, 125.43, 124.55, 121.07, 119.17, 117.13, 115.38, 114.69, 51.97, 34.32, 24.64, 19.56; IR (NaCl/thin film) 3434, 1639, 1495, 1451, 1414, 1366, 1325, 1173, 1077, 911, 781, 743 cm^{-1}; HRMS (MM) calc'd for $C_{20}H_{21}N$ $[M+OH]^+$ 292.1696, found 292.1694.

5-methoxy-3-(but-3-en-1-yl)-1-benzylindole (7h)



Prepared from 5-methoxy-3-(but-3-en-1-yl)indole (**S5**) (1.8 g, 8.9 mmol) according to General Procedure B to yield **7h** in 73% yield (1.90 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 – 7.22 (m, 3H), 7.12 (d, *J* = 8.8 Hz, 1H), 7.10 – 7.07 (m, 2H), 7.05 (d, *J* = 2.3 Hz, 1H), 6.91 (s, 1H), 6.82 (ddd, *J* = 8.8, 2.5, 0.4 Hz, 1H), 5.95 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.25 (s, 2H), 5.09 (ddt, *J* = 17.1, 2.1, 1.6 Hz, 1H), 5.00 (ddt, *J* = 10.2, 2.0, 1.2 Hz, 1H), 3.87 (s, 3H), 2.87 – 2.80 (m, 2H), 2.48 (dtt, *J* = 9.2, 6.5, 1.4 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.67, 138.79, 137.92, 131.96, 128.69, 128.44, 127.46, 126.65, 126.20, 114.83, 114.68, 111.71, 110.39, 101.02, 55.95, 50.04, 34.28, 24.73; IR (NaCl/thin film) 2917, 1487, 1452, 1228, 1044, 909, 790 cm^{-1}; HRMS (MM) calc'd for $C_{20}H_{21}NO$ $[M+H]^+$ 292.1696, found 292.1699.

5-bromo-3-(but-3-en-1-yl)-1-benzylindole (7i)



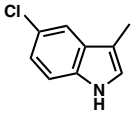
7i

Prepared from 5-bromo-3-(but-3-en-1-yl)indole (**S6**) (1.5 g, 6.0 mmol) according to General Procedure B to yield **7i** in 93% yield (1.9 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.74 (dd, *J* = 2.0, 0.5 Hz, 1H), 7.32 – 7.26 (m, 3H), 7.23 (dd, *J* = 8.7, 1.9 Hz, 1H), 7.10 (dd, *J* = 8.7, 0.5 Hz, 1H), 7.08 – 7.05 (m, 2H), 6.93 (s, 1H), 5.91 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.25 (s, 2H), 5.08 (dq, *J* = 17.1, 1.6 Hz, 1H), 5.01 (ddt, *J* = 10.2, 2.2, 1.2 Hz, 1H), 2.85 – 2.79 (m, 2H), 2.46 (dtt, *J* = 9.2, 6.5, 1.4 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.40, 137.33, 135.27, 129.90, 128.79, 127.68, 126.75, 126.61, 124.43, 121.73, 115.06, 114.95, 112.25, 111.13, 50.04, 34.30, 24.52; IR (NaCl/thin film) 3429, 2920, 1639, 1470, 1453, 1355, 1175, 1053, 912, 789, 732 cm^{-1}; HRMS (ESI) calc'd for $C_{19}H_{18}BrN$ $[M+OH]^+$ 356.0645, found 356.0640.

General Procedure C: Preparation of *N-H* indole substrates

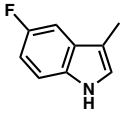
N-H indoles were prepared according to either the method of Jørgensen and co-workers[5] or a Fisher indole synthesis protocol. For the Fisher indole synthesis, the appropriate phenylhydrazine (1.0 equiv) and aldehyde (1.0 equiv) were refluxed in THF along with HCl (1.96 equiv) for 24 hr. Upon cooling to rt, the reaction mixture was diluted with water. The organic phase was separated and the aqueous phase was extracted with EtOAc twice. The organic layer was washed with water and brine, dried over $MgSO_4$, and concentrated in vacuo. The residue was purified by column chromatography on silica gel.

5-chloro-3-methyl-1*H*-indole



Prepared from 2-bromo-4-chloroaniline (3.7 g, 18 mmol) according to the method of Jørgensen and co-workers to yield **5-chloro-3-methyl-1*H*-indole** in 29% yield (345 mg). **5-chloro-3-methyl-1*H*-indole**'s spectral properties were found to be in agreement with those reported in the literature.[6]

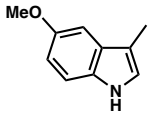
5-fluoro-3-methyl-1*H*-indole



Prepared from 4-fluorophenylhydrazine hydrochloride (1.5 g, 9.2 mmol) and propionaldehyde (538 mg, 9.2 mmol) according to the Fischer indole synthesis protocol to yield **5-fluoro-3-methyl-1*H*-indole** in 18% yield (245

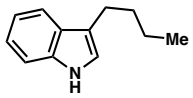
mg). **5-fluoro-3-methyl-1*H*-indole**'s spectral properties were found to be in agreement with those reported in the literature.

5-methoxy-3-methyl-1*H*-indole



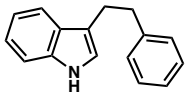
Prepared from 4-methoxyphenylhydrazine hydrochloride (1.2 g, 6.9 mmol) and propionaldehyde (400 mg, 6.9 mmol) according to the Fischer indole synthesis protocol to yield **5-methoxy-3-methyl-1*H*-indole** in 40% yield (450 mg). **5-methoxy-3-methyl-1*H*-indole**'s spectral properties were found to be in agreement with those reported in the literature.[7]

3-butyl-1*H*-indole



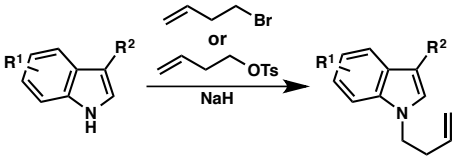
Prepared from phenylhydrazine (3.2 g, 30 mmol) and hexanal (3.0 g, 30 mmol) according to the Fischer indole synthesis protocol to yield **3-butyl-1*H*-indole** in 85% yield (4.4 g). **3-butyl-1*H*-indole**'s spectral properties were found to be in agreement with those reported in the literature.[8]

3-phenethyl-1*H*-indole



Prepared from phenylhydrazine (550 mg, 5.1 mmol) and 4-phenylbutanal (755 mg, 5.1 mmol) according to the Fischer indole synthesis protocol to yield **3-phenethyl-1*H*-indole** in 75% yield (845 mg). **3-phenethyl-1*H*-indole**'s spectral properties were found to be in agreement with those reported in the literature.[9]

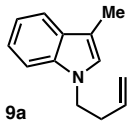
General Procedure D: Preparation of *N*-(but-3-en-1-yl) indole substrates



The indole was dissolved in DMF. NaH (1.2 equiv, 60% dispersion in mineral oil) was added at rt, followed by either 4-bromo-1-butene or *O*-tosyl-but-3-en-1-ol (1.2 equiv). The reaction was stirred at room temperature for 24 h. The reaction was quenched with a 1:1 mixture of water and a saturated sodium bicarbonate solution (10×

volume of DMF). The aqueous layer was extracted 3× with ethyl acetate. The combined organic layers were washed with water, dried ($MgSO_4$), filtered, and concentrated under reduced pressure. The crude residue was purified by column chromatography on silica gel.

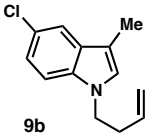
1-(but-3-en-1-yl)-3-methylindole (9a)



9a

Prepared from 3-methylindole (500 mg, 3.8 mmol) according to General Procedure D to yield **9a** in 20% yield (141 mg). **9a**'s spectral properties were found to be in agreement with those reported in the literature.[10]

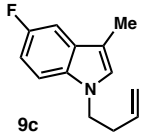
1-(but-3-en-1-yl)-5-chloro-3-methylindole (9b)



9b

Prepared from 5-chloro-3-methylindole (345 mg, 2.1 mmol) according to General Procedure D to yield **9b** in 36% yield (289 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, *J* = 1.9 Hz, 1H), 7.20 (d, *J* = 8.7 Hz, 1H), 7.13 (dd, *J* = 8.7, 2.0 Hz, 1H), 6.88 (s, 1H), 5.75 (ddt, *J* = 17.1, 10.2, 6.8 Hz, 1H), 5.07 (dtd, *J* = 17.1, 1.5, 0.5 Hz, 1H), 5.05 (dtd, *J* = 10.2, 1.7, 0.9 Hz, 1H), 4.09 (t, *J* = 7.2 Hz, 2H), 2.53 (q, *J* = 7.0 Hz, 2H), 2.27 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 134.60, 134.55, 129.78, 126.68, 124.37, 121.55, 118.55, 117.44, 110.15, 109.98, 45.90, 34.64, 9.48; IR (NaCl/thin film) 3431, 1641, 1471, 1354, 916, 834, 783 cm^{-1}; HRMS (ESI) calc'd for $C_{13}H_{14}ClN$ $[M+H]^+$ 220.0888, found 220.0890.

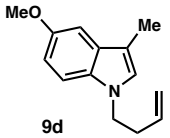
1-(but-3-en-1-yl)-5-fluoro-3-methylindole (9c)



9c

Prepared from 5-fluoro-3-methylindole according (245 mg, 1.6 mmol) to General Procedure D to yield **9c** in 85% yield (283 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.16 (m, 1H), 6.93 (td, *J* = 9.2, 2.6 Hz, 1H), 6.90 (s, 1H), 5.77 (ddt, *J* = 17.1, 10.2, 6.8 Hz, 1H), 5.08 (dq, *J* = 17.1, 1.5 Hz, 1H), 5.05 (m, 1H), 4.10 (t, *J* = 7.2 Hz, 2H), 2.59 – 2.48 (m, 2H), 2.27 (d, *J* = 1.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ, 157.48 (d, *J* = 233.7 Hz), 134.65, 132.81, 128.95 (d, *J* = 9.5 Hz), 126.99, 117.34, 110.14 (d, *J* = 4.8 Hz), 109.73, 109.55 (d, *J* = 16.9 Hz), 103.84 (d, *J* = 22.9 Hz). 45.98, 34.67, 9.55; IR (NaCl/thin film) 3434, 2924, 1488, 1457, 1358, 1199, 906, 850, 785, 619 cm^{-1}; HRMS (MM) calc'd for $C_{13}H_{14}FN$ $[M+H]^+$ 204.1183, found 204.1189.

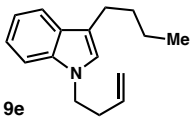
1-(but-3-en-1-yl)-5-methoxy-3-methylindole (9d)



9d

Prepared from 5-methoxy-3-methylindole (450 mg, 2.8 mmol) according to General Procedure D to yield **9d** in 48% yield (290 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (d, *J* = 8.8 Hz, 1H), 7.00 (d, *J* = 2.2 Hz, 1H), 6.88 (dd, *J* = 2.5 Hz, 8.9 Hz, 1H), 6.85 (s, 1H), 5.78 (ddt, *J* = 17.1, 10.2, 6.8 Hz, 1H), 5.12 – 5.02 (m, 2H), 4.08 (t, *J* = 7.2 Hz, 2H), 3.88 (s, 3H), 2.58 – 2.50 (m, 2H), 2.29 (d, *J* = 1.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.57, 134.89, 131.54, 128.95, 126.03, 117.14, 111.56, 109.91, 109.64, 100.87, 55.99, 45.92, 34.77, 9.66; IR (NaCl/thin film) 3421, 2090, 1640, 1490, 1226, 783 cm^{-1}; HRMS (MM) calc'd for $C_{14}H_{17}NO$ $[M+H]^+$ 216.1383, found 216.1386.

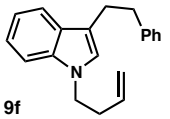
1-(but-3-en-1-yl)-3-butylindole (9e)



9e

Prepared from 3-butylindole (4.4 g, 25.4 mmol) according to General Procedure D to yield **9e** in 29% yield (1.68 g). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (dt, *J* = 7.9, 1.0 Hz, 1H), 7.33 (dt, *J* = 8.2, 1.0 Hz, 1H), 7.23 (ddd, *J* = 8.2, 7.0, 1.2 Hz, 1H), 7.12 (ddd, *J* = 7.9, 6.9, 1.0 Hz, 1H), 6.89 (s, 1H), 5.82 (ddt, *J* = 17.1, 10.2, 6.8 Hz, 1H), 5.15 – 5.06 (m, 2H), 4.15 (t, *J* = 7.3 Hz, 1H), 2.82 – 2.73 (m, 2H), 2.59 (tdd, *J* = 8.2, 6.2, 1.3 Hz, 2H), 1.77 – 1.67 (m, 2H), 1.50 – 1.39 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.27, 134.92, 128.19, 124.84, 121.28, 119.22, 118.45, 117.19, 115.72, 109.22, 45.79, 34.70, 32.56, 24.82, 22.71, 14.06; IR (NaCl/thin film) 3431, 2954, 2927, 1641, 1467, 1373, 1181, 917, 736 cm^{-1}; HRMS (MM) calc'd for $C_{16}H_{21}N$ $[M^*]^+$ 227.1669, found 227.1664.

1-(but-3-en-1-yl)-3-phenethylindole (9f)



9f

Prepared from 3-phenethylindole (845 mg, 3.8 mmol) according to General Procedure D to yield **9f** in 43% yield (446 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (dt, *J* = 7.9, 1.0 Hz, 1H), 7.33 – 7.26 (m, 3H), 7.24 – 7.18 (m, 4H), 7.11 (ddd, *J* = 8.0, 7.0, 1.0 Hz, 1H), 6.81 (s, 1H), 5.76 (ddt, *J* = 17.1, 10.2, 6.8 Hz, 1H), 5.12 – 5.02 (m, 2H), 4.12 (t, *J* = 7.2 Hz, 1H), 3.09 – 2.98 (m, 4H), 2.59 – 2.48 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.46, 136.22, 134.83, 128.51, 128.27, 127.95, 125.79, 125.10, 121.39, 119.04, 118.62, 117.21, 114.69, 109.30, 45.80, 36.69,

34.64, 27.27; IR (NaCl/thin film) 3430, 2102, 1641, 1467, 918, 737, 699 cm^{-1}; HRMS (MM) calc'd for $C_{20}H_{21}N$ $[M+H]^+$ 276.1747, found 276.1747.

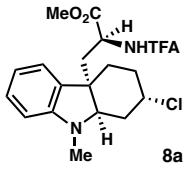
Aza-Prins Cascades

General Procedure E. Conjugate Addition/Asymmetric Protonation/Prins Cyclization Cascade

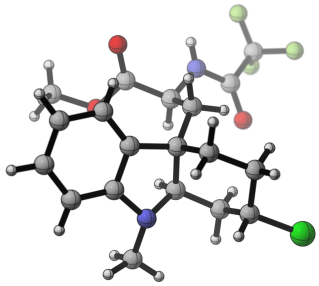
To a flame-dried flask was added indole (0.20 mmol, 1.00 equiv), acrylate (0.24 mmol, 1.20 equiv), and (*R*)-3,3'-dibromo-BINOL (0.04 mmol, 0.20 equiv), and 2,6-dibromophenol (0.20 mmol, 1.00 equiv). The flask was charged with DCM (1.5 mL), followed by addition of TMSCl (0.2 mmol, 1.00 equiv), $ZrCl_4$ (0.32 mmol, 1.60 equiv unless specifically indicated), then stirred at room temperature for 24 h. The reaction was quenched by diluting with 1 mL MeCN and 1 mL 1 M HCl, followed by addition of 5 mL H_2O. The aqueous layer was extracted with ethyl acetate (3 x 5 mL) and the combined organic layers were washed with either saturated $NaHCO_{3(aq)}$ (10 mL). The aqueous layer was back extracted with EtOAc (10 mL) and the combined organic layers were dried (Na_2SO_4), filtered, and concentrated. The crude residue was purified by flash chromatography.

Indoline Products from Conjugate Addition/Asymmetric Protonation/Prins Cyclization Cascade

Indoline 8a

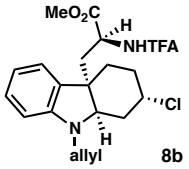


8a



Prepared from 1-methyl-3-(but-3-en-1-yl)-indole (**7a**) (37 mg, 0.2 mmol) and methyl 2-trifluoroacetamidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C to yield a mixture of **8a** and **8a'** in 84% yield (70 mg). The diastereomeric ratio was determined to be 7:1 by ^{1}H NMR analysis of the crude reaction mixture. The enantiomeric excess of the major diastereomer was determined to be 87% by chiral SFC analysis (AD-H, 2.5 mL/min, 7% IPA in CO_2, λ = 254 nm): t_R(major) = 4.9 min; t_R(minor) = 6.0 min. The major diastereomer was separated by flash chromatography (10% ethyl acetate/hexanes). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.14 (td, *J* = 7.7, 1.3 Hz, 1H), 6.95 (dd, *J* = 7.3, 0.8 Hz, 1H), 6.82 (d, *J* = 7.8 Hz, 1H), 6.75 (td, *J* = 7.4, 0.9 Hz, 1H), 6.54 (d, *J* = 7.8 Hz, 1H), 4.55 (td, *J* = 7.6, 5.8 Hz, 1H), 4.28 – 4.21 (m, 1H), 3.50 (s, 3H), 3.45 (t, *J* = 5.1 Hz, 1H), 2.72 (s, 3H), 2.41 (dd, *J* = 14.9, 7.4 Hz, 1H), 2.24 (dd, *J* = 14.9, 5.7 Hz, 1H), 2.13 – 1.99 (m, 2H), 1.91 – 1.76 (m, 4H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.3, 156.5 (q, $J_{C\text{-}F}$ = 37.7 Hz), 150.9, 133.0, 128.5, 121.7, 118.8, 115.5 (q, $J_{C\text{-}F}$ = 287.6 Hz), 108.8, 68.7, 55.8, 52.6, 50.2, 44.5, 37.8, 32.9, 32.8, 32.0, 31.0; IR (NaCl/thin film) 3312, 2954, 2864, 1711, 1607, 1482, 1209, 1178 cm^{-1}; $[\alpha]_D^{25}$ = +55.6 (*c* = 2.06, CH_2Cl_2). HRMS (MM) calc'd for $C_{19}H_{22}ClF_3N_2O_3$ $[M+H]^+$ 419.1344, found 419.1358.

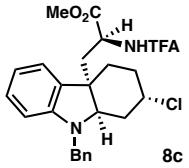
Indoline 8b



8b

Prepared from 1-allyl-3-(but-3-en-1-yl)indole (**7b**) (42 mg, 0.2 mmol) and methyl 2-trifluoroacetamidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C to yield a mixture of **8b** and **8b'** in 70% yield (62 mg). The diastereomeric ratio was determined to be 4:1 by ^{1}H NMR analysis of the crude reaction mixture. The enantiomeric excess of the major diastereomer was determined to be 89% by chiral SFC analysis (OD-H, 2.5 mL/min, 7% IPA in CO_2, λ = 254 nm): t_R(minor) = 5.0 min: t_R(major) = 8.0 min. The major diastereomer was separated by recrystallization (10% ethyl acetate/hexanes). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.11 (td, *J* = 7.7, 1.3 Hz, 1H), 6.94 (dd, *J* = 7.3, 0.8 Hz, 1H), 6.73 (td, *J* = 7.4, 0.9 Hz, 1H), 6.60 (d, *J* = 7.7 Hz, 1H), 6.57 (d, *J* = 7.8 Hz, 1H), 5.87 (dddd, *J* = 17.2, 10.2, 7.0, 4.8 Hz, 1H), 5.31 (ddd, *J* = 17.2, 3.1, 1.6 Hz, 1H), 5.25 (ddd, *J* = 10.2, 2.8, 1.4 Hz, 1H), 4.62 (dd, *J* = 13.8, 7.5 Hz, 1H), 4.23 (qd, *J* = 7.6, 3.7 Hz, 1H), 3.91 (ddt, *J* = 15.9, 4.8, 1.6 Hz, 1H), 3.68 (t, *J* = 4.9 Hz, 1H), 3.64 – 3.56 (m, 1H), 3.47 (s, *J* = 2.1 Hz, 3H), 2.41 (dd, *J* = 14.8, 7.3 Hz, 1H), 2.27 (dd, *J* = 14.8, 6.0 Hz, 1H), 2.11 (dt, *J* = 13.5, 4.1 Hz, 1H), 2.03 – 1.94 (m, 1H), 1.93 – 1.73 (m, 4H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.4, 156.4 (q, $J_{C\text{-}F}$ = 37.8 Hz), 149.6, 133.2, 133.0, 128.4, 121.8, 118.6, 117.9, 115.4 (q, $J_{C\text{-}F}$ = 288.0 Hz), 109.0, 65.8, 55.7, 52.6, 50.1, 48.2, 44.4, 37.33, 32.9, 32.2, 31.0; IR (NaCl/thin film) 3310, 2951, 1711, 1606, 1553, 1479, 1462, 1441, 1209, 1174 cm^{-1}; $[\alpha]_D^{25}$ = +78.1 (*c* = 1.39, CH_2Cl_2). HRMS (MM) calc'd for $C_{21}H_{23}ClF_3N_2O_3$ $[M+H]^+$ 445.1500, found 445.1496.

Indoline 8c

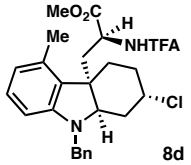


8c

Prepared from 1-benzyl-3-(but-3-en-1-yl)indole (**7c**) (52 mg, 0.2 mmol) and methyl 2-trifluoroacetmidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C to yield a mixture of **8c** and **8c'** in 82% yield (81 mg). The diastereomeric ration was determined to be 5:1 by ^{1}H NMR analysis of the crude reaction mixture. The enantiomeric excess of the major diastereomer was determined to be 86% by chiral SFC analysis (AD-H, 2.5 mL/min, 10% MeOH in CO_2, λ = 254 nm): t_R(major) = 2.5 min; t_R(minor) = 4.6 min. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.28 (m, 5H), 7.07 (td, *J* = 7.7, 1.2 Hz, 1H), 6.96 (dd, *J* = 7.3, 0.9 Hz, 1H), 6.73 (td, *J* = 7.4, 0.8 Hz, 1H), 6.57 (d, *J* = 7.9 Hz, 1H), 6.50 (d, *J* = 7.8 Hz, 1H), 4.64 (dd, *J* = 13.6, 7.5 Hz, 1H), 4.42 (d, *J* = 15.6 Hz, 1H), 4.22 (tt, *J* = 7.4, 3.5 Hz, 1H), 4.16 (d, *J* = 15.5 Hz, 1H), 3.63 (t, *J* = 5.1 Hz, 1H), 3.45 (s, *J* = 2.5 Hz, 3H), 2.44 (dd, *J* =

14.8, 7.3 Hz, 1H), 2.29 (dd, J = 14.8, 5.8 Hz, 1H), 2.15 – 2.07 (m, 1H), 2.04 – 1.78 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.4, 156.5 (q, $J_{C\text{-}F}$ = 37.8 Hz), 150.1, 137.9, 132.9, 128.7, 128.7, 127.5, 127.4, 121.9, 118.6, 115.4 (q, $J_{C\text{-}F}$ = 287.9 Hz) 109.0, 66.6, 55.9, 52.7, 50.2, 50.0, 44.5, 37.2, 33.0, 31.6, 30.9; IR (NaCl/thin film) 3309, 2952, 1717, 1605, 1495, 1479, 1210, 1174, 753 cm^{-1}; $[\alpha]_D^{25}$ = +65.1 (c = 1.70, CH_2Cl_2). HRMS (MM) calc'd for $C_{25}H_{26}ClF_3N_2O_3$ $[M+H]^+$ 495.1657, found 495.1648.

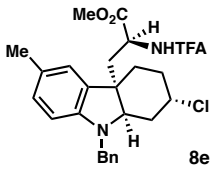
Indoline 8d



8d

Prepared from 1-benzyl-3-(but-3-en-1-yl)-4-methylindole (**7d**) (55 mg, 0.2 mmol) and methyl 2-trifluoroacetamidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C to yield a mixture of **8d** and **8d'** in 90% yield (91 mg). The diastereomeric ratio was determined to be 4:1 by ^{1}H NMR analysis of the crude reaction mixture. The enantiomeric excess of the major diastereomer was determined to be 88% by chiral SFC analysis (OD-H, 2.5 mL/min, 10% EtOH in CO_2, λ = 254 nm): t_R(minor) = 6.7 min; t_R(major) = 7.5 min. The major diastereomer was separated by flash chromatography (5→10% ethyl acetate/hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.27 (m, 5H), 6.96 (t, J = 7.7 Hz, 1H), 6.67 (d, J = 7.6 Hz, 1H), 6.48 (d, J = 7.6 Hz, 1H), 6.37 (d, J = 7.9 Hz, 1H), 4.64 (q, J = 7.0 Hz, 1H), 4.40 (d, J = 15.8 Hz, 1H), 4.21 – 4.12 (m, 2H), 3.60 (t, J = 4.3 Hz, 1H), 3.38 (s, 3H), 2.51 (dd, J = 15.0, 6.6 Hz, 1H), 2.40 (dd, J = 15.0, 7.0 Hz, 1H), 2.29 (s, 3H), 2.21 (dt, J = 14.6, 4.3 Hz, 1H), 2.03 – 1.88 (m, 4H), 1.88 – 1.75 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.3, 156.5 (q, $J_{C\text{-}F}$ = 37.8 Hz), 150.9, 138.0, 133.9, 129.7, 128.7 (×2), 128.3, 127.4 (×2), 127.3, 122.2, 115.4 (q, $J_{C\text{-}F}$ = 287.9 Hz), 107.0, 65.8, 55.5, 52.6, 50.6, 50.4, 46.2, 36.6, 33.1, 31.3, 31.1, 19.1; IR (NaCl/thin film) 3311, 2953, 1711, 1589, 1452, 1212, 1177 cm^{-1}; $[\alpha]_D^{25}$ = +79.4 (c = 0.81, CH_2Cl_2). HRMS (MM) calc'd for $C_{26}H_{28}ClF_3N_2O_3$ $[M+H]^+$ 509.1813, found 509.1806.

Indoline 8e

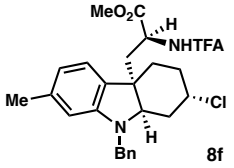


8e

Prepared from 1-benzyl-3-(but-3-en-1-yl)-5-methylindole (**7e**) (55 mg, 0.2 mmol) and methyl 2-trifluoroacetamidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C to yield a mixture of **8e** and **8e'** in quantitative yield (102 mg). The diastereomeric ratio was determined to be 6:1 by ^{1}H NMR analysis of the crude reaction mixture. The enantiomeric excess of the major diastereomer was determined to be 92% by chiral SFC

analysis (OJ-H, 2.5 mL/min, 8% EtOH in CO_2, λ = 254 nm); t_R(minor) = 5.1 min; t_R(major) = 7.0 min. The major diastereomer was separated by flash chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ .39 – 7.27 (m, 5H), 6.87 (ddd, *J* = 7.9, 1.7, 0.7 Hz, 1H), 6.78 (d, *J* = 1.7 Hz, 1H), 6.50 (d, *J* = 7.7 Hz, 1H), 6.39 (d, *J* = 7.9 Hz, 1H), 4.62 (td, *J* = 7.6, 5.5 Hz, 1H), 4.37 (d, *J* = 15.4 Hz, 1H), 4.24 (dt, *J* = 11.3, 3.7 Hz, 1H), 4.10 (d, *J* = 15.4 Hz, 1H), 3.56 (t, *J* = 5.2 Hz, 1H), 3.49 (s, *J* = 2.3 Hz, 3H), 2.44 (dd, *J* = 14.8, 7.5 Hz, 1H), 2.30 – 2.21 (m, 4H), 2.09 – 1.78 (m, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.4, 156.4 (q, J_{C-F} = 37.8 Hz), 147.9, 138.0, 133.0, 128.7, 128.6 (×2), 128.0, 127.5 (×2), 127.3, 122.8, 116.6 (q, J_{C-F} = 287.8 Hz), 109.0, 66.9, 56.1, 52.6, 50.4, 50.1, 44.6, 37.3, 32.8, 31.1, 30.8, 20.7; IR (NaCl/thin film) 3314, 2951, 2868, 1715, 1552, 1490, 1440, 1210, 1177 cm^{-1}; $[\alpha]_D^{25}$ = +55.3 (*c* = 0.85, CH_2Cl_2). HRMS (MM) calc'd for $C_{26}H_{28}ClF_3N_2O_3$ $[M+H]^+$ 509.1813, found 509.1831.

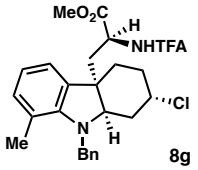
Indoline 8f



8f

Prepared from 1-benzyl-3-(but-3-en-1-yl)-6-methylindole (**7f**) (55 mg, 0.2 mmol) and methyl 2-trifluoroacetamidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C (but with 1.1 equiv $ZrCl_4$) to yield a mixture of **8f** and **8f'** in 74% yield (75 mg). The diastereomeric ratio was determined to be 3:1 by ^{1}H NMR analysis of the crude reaction mixture. The enantiomeric excess of the major diastereomer was determined to be 93% by chiral SFC analysis (AD-H, 2.5 mL/min, 20% IPA in CO_2, λ = 254 nm): t_R(minor) = 1.9 min; t_R(major) = 2.6 min. The major diastereomer was separated by flash chromatography (5→10% ethyl acetate/hexanes). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.27 (m, 5H), 6.84 (d, *J* = 7.4 Hz, 1H), 6.54 (d, *J* = 7.4 Hz, 1H), 6.49 (d, *J* = 7.7 Hz, 1H), 6.33 (s, 1H), 4.60 (td, *J* = 7.7, 5.4 Hz, 1H), 4.38 (d, *J* = 15.6 Hz, 1H), 4.21 (dq, *J* = 11.0, 3.7 Hz, 1H), 4.13 (d, *J* = 15.6 Hz, 1H), 3.58 (t, *J* = 5.3 Hz, 1H), 3.49 (s, *J* = 2.4 Hz, 3H), 2.39 (dd, *J* = 14.8, 7.6 Hz, 1H), 2.27 – 2.21 (m, 4H), 2.06 – 1.77 (m, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.4, 156.5 (q, J_{C-F} = 37.8 Hz), 150.3, 138.6, 138.0, 129.9, 128.7 (×2), 127.4 (×2), 127.3, 121.8, 119.2, 115.4 (q, J_{C-F} = 287.8 Hz), 109.8, 66.7, 56.0, 52.7, 50.1, 50.0, 44.37, 37.6, 33.0, 31.0, 30.8, 21.7; IR (NaCl/thin film) 3312, 2950, 1712, 1612, 1551, 1493, 1452, 1210, 1176 cm^{-1}; $[\alpha]_D^{25}$ = +65.8 (*c* = 0.89, CH_2Cl_2). HRMS (MM) calc'd for $C_{26}H_{28}ClF_3N_2O_3$ $[M+H]^+$ 509.1813, found 509.1823.

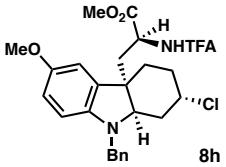
Indoline 8g



8g

Prepared from 1-benzyl-3-(but-3-en-1-yl)-7-methylindole (**7g**) (55 mg, 0.2 mmol) and methyl 2-trifluoroacetamidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C to yield a mixture of **8g** and **8g'** in 89% yield (90 mg). The diastereomeric ratio was determined to be 6:1 by ^{1}H NMR analysis of the crude reaction mixture. The enantiomeric excess of the major diastereomer was determined to be 89% by chiral SFC analysis (AD-H, 2.5 mL/min, 20% IPA in CO_2, λ = 254 nm): t_R(minor) = 1.8 min; t_R(major) = 2.5 min. The major diastereomer was separated by flash chromatography (5→10% ethyl acetate/hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.27 (m, 5H), 6.91 (d, J = 7.5 Hz, 1H), 6.82 (d, J = 7.3 Hz, 1H), 6.70 (t, J = 7.4 Hz, 1H), 6.46 (d, J = 7.8 Hz, 1H), 4.71 (d, J = 16.5 Hz, 1H), 4.62 – 4.49 (m, 2H), 4.19 (td, J = 7.9, 3.8 Hz, 1H), 3.51 (s, 3H), 3.45 (t, J = 4.9 Hz, 1H), 2.35 (s, 3H), 2.24 (d, J = 6.3 Hz, 2H), 2.04 (dt, J = 15.0, 4.4 Hz, 1H), 1.96 – 1.69 (m, 5H).; ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 156.4 (q, $J_{C\text{-}F}$ = 37.7 Hz), 148.0, 139.3, 133.7, 132.3, 128.7 (×2), 127.3, 127.2 (×2), 120.4, 120.0, 119.3, 115.4 (q, $J_{C\text{-}F}$ = 287.8 Hz), 66.4, 55.9, 52.7, 52.3, 50.0, 44.6, 37.9, 33.9, 32.0, 30.8, 19.6; IR (NaCl/thin film) 3314, 2952, 1715, 1558, 1452, 1208, 1176 cm^{-1}; $[\alpha]_D^{25}$ = +57.2 (c = 0.94, CH_2Cl_2). HRMS (MM) calc'd for $C_{26}H_{28}ClF_3N_2O_3$ $[M–H]^-$ 507.1668, found 507.1681.

Indoline 8h

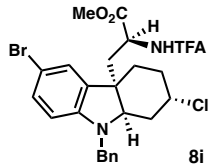


8h

Prepared from 1-benzyl-3-(but-3-en-1-yl)-5-methoxyindole (**7h**) (58 mg, 0.2 mmol) and methyl 2-trifluoroacetamidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C (but with 1.1 equiv $ZrCl_4$) to yield a mixture of **8h** and **8h'** in 93% yield (97 mg). The diastereomeric ratio was determined to be 6:1 by ^{1}H NMR analysis of the crude reaction mixture. The enantiomeric excess of the major diastereomer was determined to be 91% by chiral SFC analysis (AD-H, 2.5 mL/min, 12% IPA in CO_2, λ = 254 nm): t_R(minor) = 4.2 min; t_R(major) = 4.9 min. The major diastereomer was separated by flash chromatography (15→20% ethyl acetate/hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.27 (m, 5H), 6.63 – 6.57 (m, 2H), 6.53 (d, J = 7.7 Hz, 1H), 6.39 (d, J = 8.0 Hz, 1H), 4.60 (dd, J = 13.8, 6.8 Hz, 1H), 4.34 (d, J = 15.4 Hz, 1H), 4.23 (dd, J = 9.8, 6.4 Hz, 1H), 4.07 (d, J = 15.4 Hz, 1H), 3.72 (s, 3H), 3.55 (t, J = 4.6 Hz, 1H), 3.47 (s, 3H), 2.44 (dd, J = 14.9, 7.0 Hz, 1H), 2.28 (dd, J = 14.9, 5.8 Hz, 1H), 2.16 – 2.09 (m, 1H), 1.99 – 1.72 (m, 5H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.3, 156.4 (q, $J_{C\text{-}F}$ = 37.7 Hz), 153.4, 144.2, 138.0, 134.7, 128.6 (×2), 127.5 (×2), 127.3, 115.4 (q, $J_{C\text{-}F}$ = 288.0), 112.7, 109.8, 109.2, 67.0,

55.8, 52.7, 51.2, 50.1, 44.7, 36.9, 33.0, 32.3, 31.1, 29.7; IR (NaCl/thin film) 3315, 2925, 1716, 1555, 1490, 1215, 1176 cm^{-1}; $[\alpha]_D^{25}$ = +40.4 (*c* = 0.96, CH_2Cl_2). HRMS (MM) calc'd for $C_{26}H_{28}ClF_3N_2O_4$ $[M+H]^+$ 525.1762, found 525.1749.

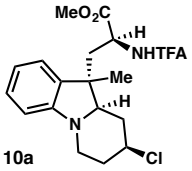
Indoline 8i



8i

Prepared from 1-benzyl-3-(but-3-en-1-yl)-5-bromoindole (**8i**) (68 mg, 0.2 mmol) and methyl 2-trifluoroacetamidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C to yield a mixture of **8i** and **8i'** in 70% yield (80 mg). The diastereomeric ratio was determined to be 5:1 by ^{1}H NMR analysis of the crude reaction mixture. The enantiomeric excess of the major diastereomer was determined to be 85% by chiral SFC analysis (OD-H, 2.5 mL/min, 10% EtOH in CO_2, λ = 254 nm): t_R(minor) = 7.7 min; t_R(major) = 9.5 min. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 – 7.28 (m, 5H), 7.15 (dd, *J* = 8.3, 2.0 Hz, 1H), 7.03 (d, *J* = 2.0 Hz, 1H), 6.68 (d, *J* = 8.1 Hz, 1H), 6.36 (d, *J* = 8.4 Hz, 1H), 4.62 (dd, *J* = 14.6, 6.6 Hz, 1H), 4.38 (d, *J* = 15.6 Hz, 1H), 4.22 – 4.09 (m, 2H), 3.66 (t, *J* = 4.8 Hz, 1H), 3.49 (s, 3H), 2.38 (dd, *J* = 14.9, 6.8 Hz, 1H), 2.28 (dd, *J* = 14.9, 6.2 Hz, 1H), 2.01 – 1.75 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.2, 156.5 (q, J_{C-F} = 38.1 Hz), 149.3, 137.3, 135.5, 131.0, 128.8 (×2), 127.5, 127.4 (×2), 125.0, 115.4 (q, J_{C-F} = 287.9 Hz), 110.4, 110.2, 66.6, 60.4, 55.3, 52.8, 50.2, 49.9, 44.7, 37.1, 33.0, 32.1, 30.9. IR (NaCl/thin film) 3308, 2951, 2864, 1713, 1475, 1210, 1175 cm^{-1}; $[\alpha]_D^{25}$ = +41.4 (*c* = 0.90, CH_2Cl_2). HRMS (MM) calc'd for $C_{25}H_{25}BrClF_3N_2O_3$ $[M+H]^+$ 573.0762, found 573.0745.

Indoline 10a

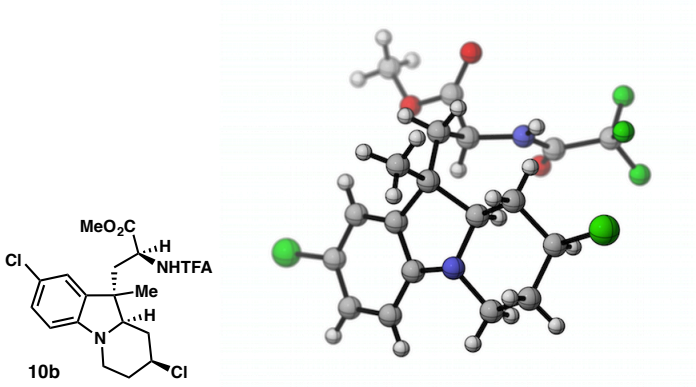


10a

Prepared from 1-(but-3-en-1-yl)-3-methylindole (**9a**) (37 mg) and methyl 2-trifluoroacetamidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C to yield a mixture of **10a** and **10a'** in 93% yield (70 mg). The diastereomeric ratio was determined to be 4:1 by ^{1}H NMR analysis of the crude reaction mixture. The enantiomeric excess of the major diastereomer was determined to be 86% by chiral SFC analysis (OD-H, 2.5 mL/min, 8% EtOH in CO_2, λ = 254 nm): t_R(minor) = 5.3 min; t_R(major) = 6.0 min. The major diastereomer was separated by flash chromatography (12→15% ethyl acetate/hexanes). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.12 (td, *J* = 7.7, 1.2 Hz, 1H), 6.99 (dd, *J* = 7.7, 1.3 Hz, 1H), 6.89 (d, *J* = 7.9 Hz, 1H), 6.74 (td, *J* = 7.4, 0.9 Hz, 1H), 6.50 (d, *J*

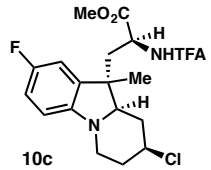
= 7.9 Hz, 1H), 4.58 (td, *J* = 7.8, 4.6 Hz, 1H), 3.98 (tt, *J* = 11.8, 4.0 Hz, 1H), 3.72 (ddd, *J* = 13.3, 4.7, 2.1 Hz, 1H), 3.59 (s, *J* = 3.5 Hz, 3H), 3.16 (dd, *J* = 11.8, 2.7 Hz, 1H), 2.82 (tt, *J* = 18.4, 9.2 Hz, 1H), 2.30 (dd, *J* = 14.9, 4.7 Hz, 1H), 2.21 – 2.07 (m, 3H), 1.89 – 1.77 (m, 1H), 1.72 (q, *J* = 11.9 Hz, 1H), 1.22 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.3, 156.6 (q, $J_{C\text{-}F}$ = 37.7 Hz), 148.8, 134.4, 128.5, 122.7, 118.8, 115.5 (q, $J_{C\text{-}F}$ = 287.7 Hz), 107.3, 70.1, 57.1, 52.7, 50.3, 45.7, 43.9, 40.4, 35.9, 34.6, 21.0; IR (NaCl/thin film) 3314, 2958, 1711, 1606, 1482, 1454, 1211, 1173 cm^{-1}; $[\alpha]_D^{25}$ = +42.0 (*c* = 4.3, CH_2Cl_2). HRMS (MM) calc'd for $C_{19}H_{22}ClF_3N_2O_3$ $[M+H]^+$ 419.1344, found 419.1342.

Indoline 10b



Prepared from 1-(but-3-en-1-yl)-5-chloro-3-methylindole (**9b**) (44 mg) and methyl 2-trifluoroacetamidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C to yield a mixture of **10b** and **10b'** in 56% yield (51 mg). The diastereomeric ratio was determined to be 4:1 by ^{1}H NMR analysis of the crude reaction mixture. The enantiomeric excess of the major diastereomer was determined to be 87% by chiral SFC analysis (AD-H, 2.5 mL/min, 7% IPA in CO_2, λ = 254 nm): t_R(major) = 10.3 min; t_R(minor) = 13.4 min. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.11 (dd, *J* = 8.3, 2.1 Hz, 1H), 6.96 (d, *J* = 2.1 Hz, 1H), 6.71 (d, *J* = 8.2 Hz, 1H), 6.44 (d, *J* = 8.3 Hz, 1H), 4.64 (td, *J* = 7.6, 5.1 Hz, 1H), 4.00 (tt, *J* = 11.8, 4.0 Hz, 1H), 3.71 (ddd, *J* = 13.2, 4.7, 2.2 Hz, 1H), 3.65 (s, 3H), 3.24 (dd, *J* = 11.8, 2.7 Hz, 1H), 2.86 (td, *J* = 12.9, 2.9 Hz, 1H), 2.35 (dd, *J* = 14.9, 5.2 Hz, 1H), 2.21 (ddq, *J* = 12.4, 4.2, 2.2 Hz, 1H), 2.13 (ddq, *J* = 12.2 4.4, 2.1 Hz, 1H), 2.10 (dd, *J* = 15.0, 7.2 Hz, 1H), 1.87 (qd, *J* = 12.5, 4.7 Hz, 1H), 1.75 (q, *J* = 11.9 Hz, 1H), 1.24 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.11, 156.42 (q, $J_{C\text{-}F}$ = 37.9 Hz), 147.47, 136.39, 128.22, 123.41, 123.11, 115.45 (q, $J_{C\text{-}F}$ = 287.8 Hz), 108.09, 69.82, 56.71, 52.92, 50.03, 45.78, 43.96, 40.64, 35.80, 34.48, 21.12; IR (NaCl/thin film) 3403, 2360, 1714, 1646, 1481, 1212, 1171, 811, 726 cm^{-1}; $[\alpha]_D^{25}$ = +22.9 (*c* = 0.14, CH_2Cl_2). HRMS (ESI) calc'd for $C_{19}H_{21}Cl_2F_3N_2O_3$ $[M+Cl]^-$ 487.0575, found 487.0594.

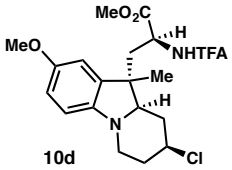
Indoline 10c



10c

Prepared from 1-(but-3-en-1-yl)-5-fluoro-3-methylindole (**9c**) (40 mg) and methyl 2-trifluoroacetamidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C to yield a mixture of **10c** and **10c'** in 82% yield (71 mg). The diastereomeric ratio was determined to be 4:1 by ^{1}H NMR analysis of the crude reaction mixture. The enantiomeric excess of the major diastereomer was determined to be 84% by chiral SFC analysis (AD-H, 2.5 mL/min, 7% IPA in CO_2, λ = 254 nm): t_R(major) = 6.2 min; t_R(minor) = 7.5 min. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.81 (td, *J* = 8.9, 2.6 Hz, 1H), 6.71 (dd, *J* = 8.2, 2.6 Hz, 2H), 6.38 (dd, *J* = 8.5, 4.1 Hz, 1H), 4.58 (td, *J* = 7.8, 4.9 Hz, 1H), 3.94 (tt, *J* = 11.8, 4.1 Hz, 1H), 3.65 (ddd, *J* = 4.7, 2.2, 13.2 Hz, 1H), 3.62 (s, 3H), 3.13 (dd, *J* = 11.8, 2.7 Hz, 1H), 2.78 (td, *J* = 12.9, 2.8 Hz, 1H), 2.31 (dd, *J* = 15.0, 5.0 Hz, 1H), 2.19 – 2.12 (m, 1H), 2.07 (dd, *J* = 14.3, 6.7 Hz, 1H), 2.04-2.10 (m, 1H), 1.84 (qd, *J* = 12.5, 4.7 Hz, 1H), 1.72 (q, *J* = 11.9 Hz, 1H), 1.18 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.17, 156.98 (d, J_{C-F} = 237.1 Hz), 156.45 (q, J_{C-F} = 37.8 Hz), 145.03, 136.24, 116.90 (q, J_{C-F} = 288.86 Hz), 114.36 (d, J_{C-F} = 23.1 Hz), 110.52 (d, J_{C-F} = 24.4 Hz), 107.61 (d, J_{C-F} = 8.3 Hz), 70.19, 56.87, 52.89, 50.10, 45.81, 44.39, 40.30, 35.74, 34.49, 21.16. IR (NaCl/thin film) 3412, 2095, 1708, 1643, 1486, 1212, 1171, 859, 808, 730 cm^{-1}; $[\alpha]_D^{25}$ = +43.3 (*c* = 0.53, CH_2Cl_2). HRMS (MM) calc'd for $C_{19}H_{21}ClF_4N_2O_3$ $[M^*]^+$ 436.1171, found 436.1187.

Indoline 10d

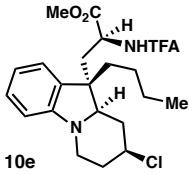


10d

Prepared from 1-(but-3-en-1-yl)-5-methoxy-3-methylindole (**9d**) (43 mg) and methyl 2-trifluoroacetamidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C to yield a mixture of **10d** and **10d'** in 80% yield (72 mg). The diastereomeric ratio was determined to be 3:1 by ^{1}H NMR analysis of the crude reaction mixture. The enantiomeric excess of the major diastereomer was determined to be 81% by chiral SFC analysis (OD-H, 2.5 mL/min, 4% IPA in CO_2, λ = 254 nm): t_R(minor) = 22.4 min; t_R(major) = 24.2 min. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.80 (d, *J* = 7.9 Hz, 1H), 6.67 (dd, *J* = 8.4, 2.6 Hz, 1H), 6.62 (d, *J* = 2.5 Hz, 1H), 6.41 (d, *J* = 8.4 Hz, 1H), 4.51 (td, *J* = 7.7, 4.7 Hz, 1H), 3.94 (tt, *J* = 11.9, 4.1 Hz, 1H), 3.74 (s, 3H), 3.65 (ddd, *J* = 13.3, 4.9, 2.5 Hz, 1H), 3.61 (s, 3H), 3.08 (dd, *J* = 11.7, 2.7 Hz, 1H), 2.77 (td, *J* = 12.9, 2.8 Hz, 1H), 2.29 (dd, *J* = 15.0, 4.8 Hz, 1H), 2.16 – 2.01 (m, 3H), 1.84 (qd, *J* = 12.5, 4.6 Hz, 1H), 1.71 (q, *J* = 11.9 Hz, 1H), 1.19 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ

171.22, 156.45 (q, $J_{C\text{-}F}$ = 37.7 Hz), 153.61, 142.80, 135.94, 116.91 (q, $J_{C\text{-}F}$ = 287.8 Hz), 112.82, 110.15, 107.93, 70.45, 57.19, 55.85, 52.80, 50.36, 45.89, 44.56, 40.35, 35.71, 34.36, 21.13. IR (NaCl/thin film) 3429, 2100, 1644, 1486, 1212, 1182, 730 cm^{-1}; $[\alpha]_D^{25}$ = 57.4 (*c* = 0.54, CH_2Cl_2). HRMS (MM) calc'd for $C_{20}H_{24}ClF_3N_2O_4$ $[M+H]^+$ 449.1449, found 449.1451.

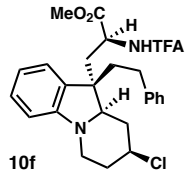
Indoline 10e



10e

Prepared from 1-(but-3-en-1-yl)-3-butylindole (**9e**) (45 mg) and methyl 2-trifluoroacetamidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C to yield a mixture of **10e** and **10e'** in 87% yield (80 mg). The diastereomeric ratio was determined to be 5:1 by ^1H NMR analysis of the crude reaction mixture. The enantiomeric excess of the major diastereomer was determined to be 90% by chiral SFC analysis (AS-H, 2.5 mL/min, 10% IPA in CO_2, λ = 254 nm): t_R(major) = 2.5 min; t_R(minor) = 3.5 min. ^1H NMR (500 MHz, $CDCl_3$) δ 7.12 (td, *J* = 7.7, 1.2 Hz, 1H), 6.94 (dd, *J* = 7.2, 1.2 Hz, 1H), 6.73 (td, *J* = 7.4, 0.9 Hz, 1H), 6.58 (d, *J* = 8.0 Hz, 1H), 6.50 (d, *J* = 7.8 Hz, 1H), 4.53 (td, *J* = 7.8, 4.9 Hz, 1H), 3.95 (tt, *J* = 11.8, 4.1 Hz, 1H), 3.70 (ddd, *J* = 12.9, 4.7, 2.2 Hz, 1H), 3.54 (s, 3H), 3.15 (dd, *J* = 11.8, 2.6 Hz, 1H), 2.75 (td, *J* = 12.8, 2.8 Hz, 1H), 2.40 (dd, *J* = 15.0, 4.9 Hz, 1H), 2.17 (ddq, *J* = 12.9, 4.5, 2.2 Hz, 1H), 2.10 (dd, *J* = 15.1, 7.7 Hz, 1H), 1.87 (qd, *J* = 12.5, 4.7 Hz, 1H), 1.81 (q, *J* = 11.9 Hz, 2H), 1.76 (td, *J* = 13.0, 3.9 Hz, 1H), 1.44 – 1.37 (m, 1H), 1.37 – 1.20 (m, 3H), 1.18 – 1.09 (m, 1H), 0.88 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.33, 156.42 (q, $J_{C\text{-}F}$ = 37.8 Hz), 149.15, 133.09, 128.48, 123.82, 118.73, 116.58 (q, $J_{C\text{-}F}$ = 288.54 Hz), 107.71, 70.74, 57.13, 52.73, 50.15, 48.51, 44.42, 37.15, 35.31, 34.62, 33.17, 26.29, 23.24, 14.01; IR (NaCl/thin film) 3418, 2957, 1713, 1644, 1460, 1210, 1184, 911, 730 cm^{-1}; $[\alpha]_D^{25}$ = +7.85 (*c* = 0.20, CH_2Cl_2). HRMS (MM) calc'd for $C_{22}H_{28}ClF_3N_2O_3$ $[M+OH]^+$ 477.1762, found 477.1765.

Indoline 10f



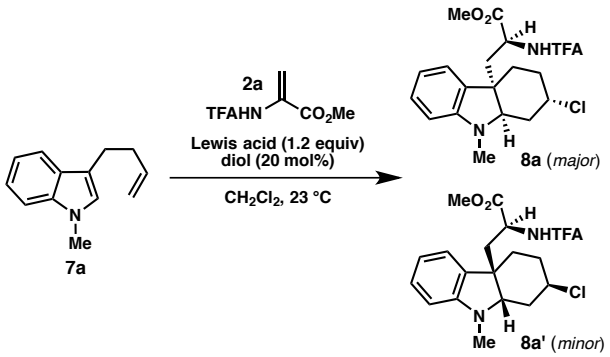
10f

Prepared from 1-(but-3-en-1-yl)-3-phenethylindole (**9a**) (55 mg) and methyl 2-trifluoroacetamidoacrylate (**2a**) (47 mg, 0.24 mmol) using General Procedure C to yield a mixture of **10f** and **10f'** in 83% yield (84 mg). The diastereomeric ratio was determined to be 3:1 by ^1H NMR analysis of the crude reaction mixture. The

enantiomeric excess of the major diastereomer was determined to be 91% by chiral SFC analysis (OB-H, 2.5 mL/min, 8% IPA in CO_2, λ = 254 nm): t_R(minor) = 7.5 min; t_R(major) = 10.1 min. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 – 7.24 (m, 2H), 7.20 – 7.13 (m, 2H), 7.11 – 7.08 (m, 2H), 7.01 (dd, *J* = 7.7, 0.9 Hz, 1H), 6.77 (td, *J* = 7.4, 1.0 Hz, 1H), 6.68 (d, *J* = 8.0 Hz, 1H), 6.54 (d, *J* = 7.8 Hz, 1H), 4.62 (td, *J* = 7.6, 5.4 Hz, 1H), 3.95 (tt, *J* = 11.7, 4.1 Hz, 1H), 3.72 (ddd, *J* = 12.7, 4.7, 2.2 Hz, 1H), 3.55 (s, 3H), 3.21 (dd, *J* = 11.8, 2.4 Hz, 1H), 2.74 (td, *J* = 12.6, 2.7 Hz, 1H), 2.67 (td, *J* = 13.1, 4.8 Hz, 1H), 2.57 (dd, *J* = 15.0, 5.4 Hz, 1H), 2.46 (td, *J* = 13.2, 4.4 Hz, 1H), 2.20 (m, 2H), 2.17 (dd, *J* = 14.9, 7.2 Hz, 1H), 2.06 (td, *J* = 13.4, 4.8 Hz, 1H), 1.90 (qd, *J* = 12.4, 4.6 Hz, 1H), 1.88 (q, 12.0 Hz, 2H), 1.70 (ddd, *J* = 13.8, 12.7, 4.4 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.37, 156.46 (q, $J_{C\text{-}F}$ = 37.8 Hz), 149.57, 141.64, 132.30, 128.64, 128.49, 128.20, 126.05, 123.70, 118.81, 115.44 (q, $J_{C\text{-}F}$ = 287.9 Hz), 107.79, 70.22, 56.99, 52.81, 50.09, 48.57, 44.57, 36.93, 36.32, 35.34, 34.90, 30.50; IR (NaCl/thin film) 3324, 2925, 2357, 1713, 1605, 1557, 1480, 1456, 1211, 1174, 747, 699 cm^{-1}; $[\alpha]_D^{25}$ = +5.17 (*c* = 1.03, CH_2Cl_2). HRMS (MM) calc'd for $C_{25}H_{28}ClF_3N_2O_3$ $[M+H]^+$ 509.1813, found 509.1821.

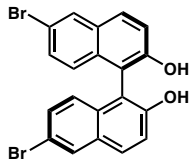
Catalyst Optimization

Table 1. Reaction optimization: Lewis Acid and chiral diol screen.[a]

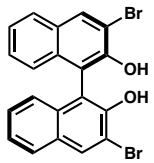


Entry	Lewis Acid	Diol	Yield **8** (%)[b]	dr	ee **8a** (%)[c]
1	$SnCl_4$	(*R*)-BINOL (**L1**)	19	10:1	88
2	$TiCl_4$	(*R*)-BINOL	27	6:1	0
3	$SbCl_5$	(*R*)-BINOL	0	--	--
4	$ZrCl_4$	(*R*)-BINOL	30	9:1	40
5	$Zr(O^tBu)_4$	(*R*)-BINOL	0	--	--
6	$ZrCl_4$	(*R*)-6,6'-dibromo-BINOL (**L2**)	38	9:1	30
7	$ZrCl_4$	(*R*)-3,3'-dibromo-BINOL (**L3**)	40	7:1	76
8	$ZrCl_4$	(*S*)-VANOL (**L4**)	33	6:1	74
9	$ZrCl_4$	(4*R*, 5*R*)-Ph-TADDOL (**L5**)	37	>10:1	66

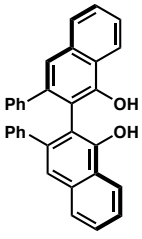
[a] Reactions conducted with 0.20 mmol **7a** and 0.24 mmol **2a**. [b] Combined isolated yield of two diastereomers. [c] The ee of the major diastereomer was determined by SFC using a chiral stationary phase.



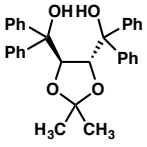
(*R*)-6,6'-dibromo-BINOL



(*R*)-3,3'-dibromo-BINOL



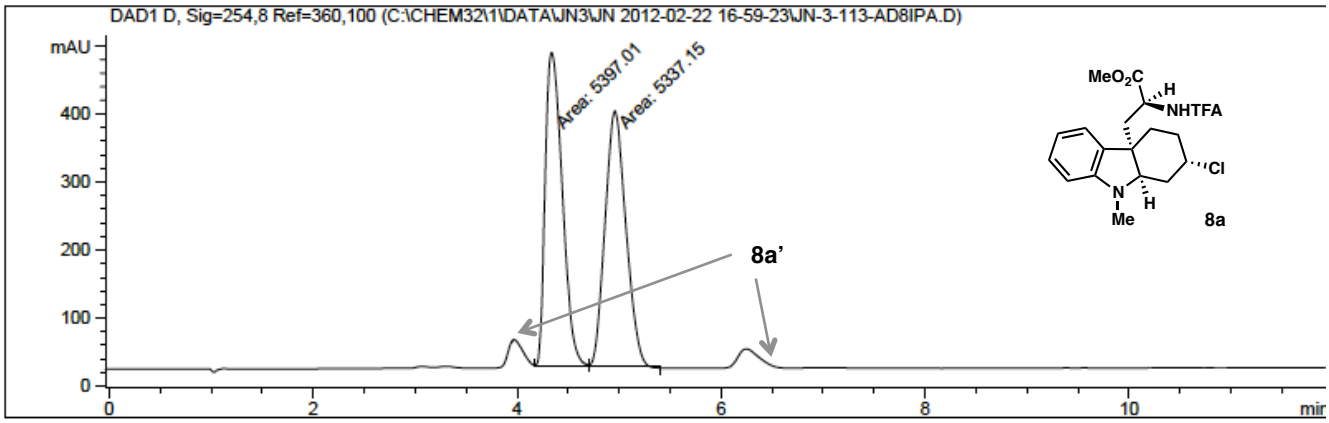
(*S*)-VANOL



(4*R*,5*R*)-TADDOL

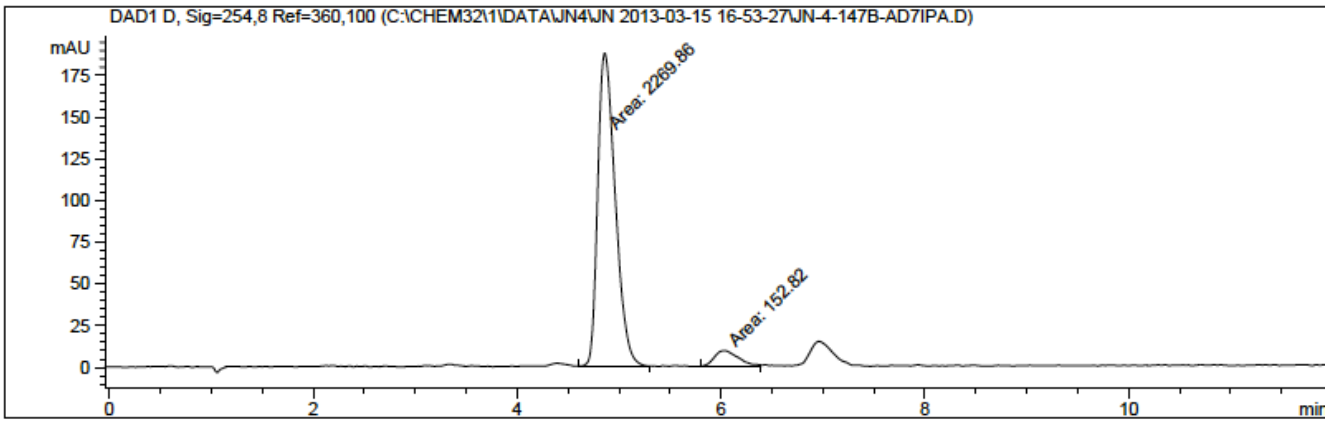
SFC/HPLC traces of racemic and enantioenriched indolines

8a: racemic



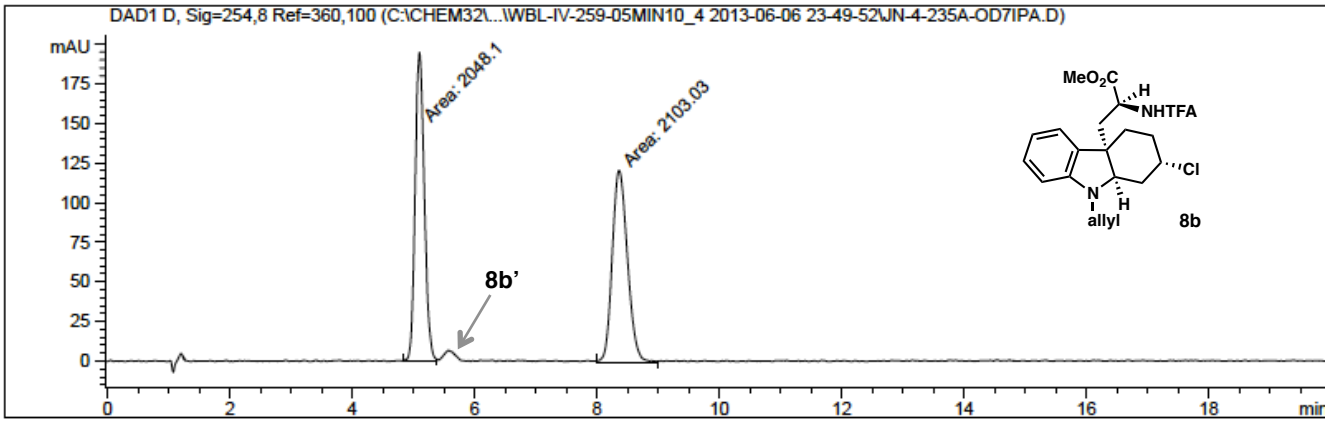
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.338	MM	0.1948	5397.01465	461.86542	50.2788
2	4.959	MM	0.2366	5337.15186	375.89224	49.7212

8a: 87% ee



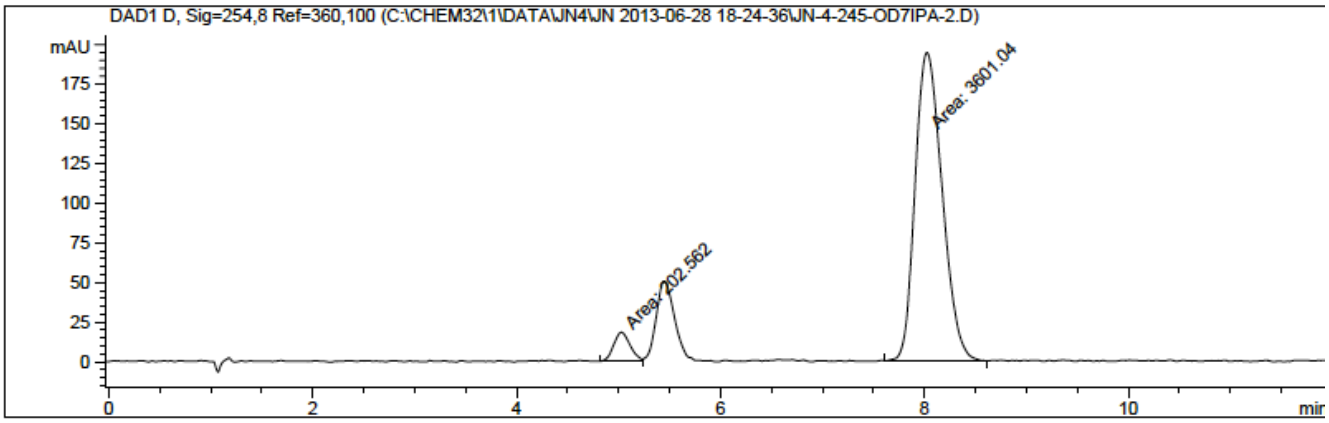
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.860	MM	0.2008	2269.86377	188.42389	93.6921
2	6.036	MM	0.2709	152.81998	9.40274	6.3079

8b: racemic



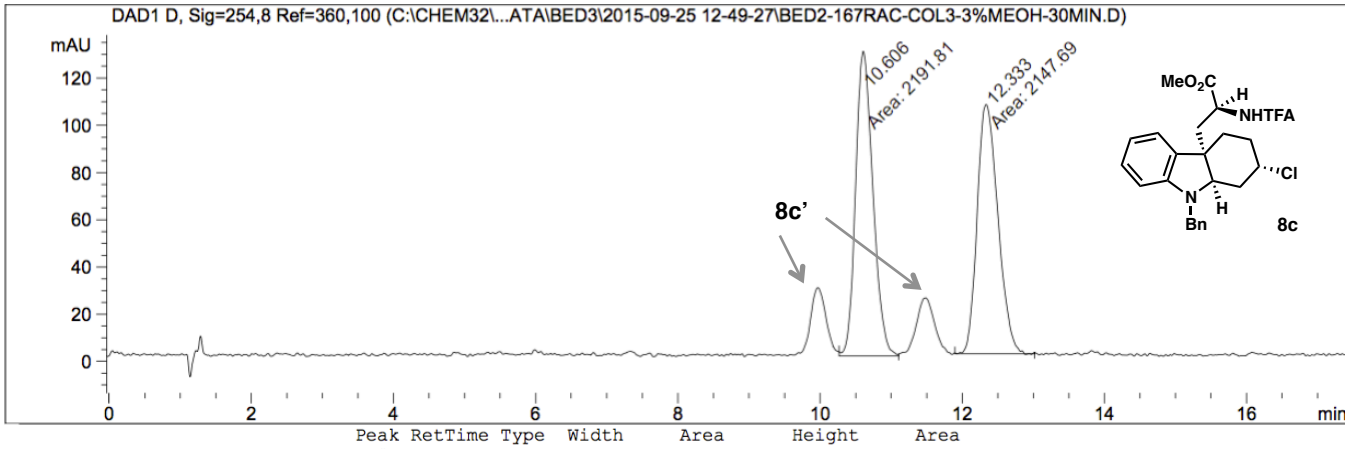
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.099	MM	0.1751	2048.10229	194.90222	49.3384
2	8.364	MM	0.2889	2103.03052	121.33443	50.6616

8b: 89% ee



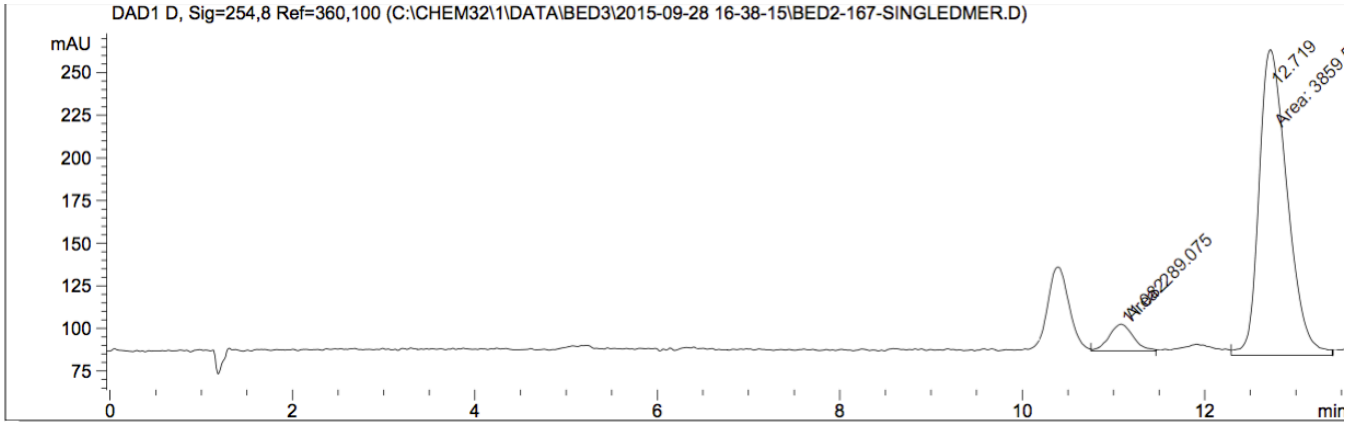
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.028	MM	0.1861	202.56195	18.14186	5.3255
2	8.024	MM	0.3085	3601.03638	194.54140	94.6745

8c: racemic



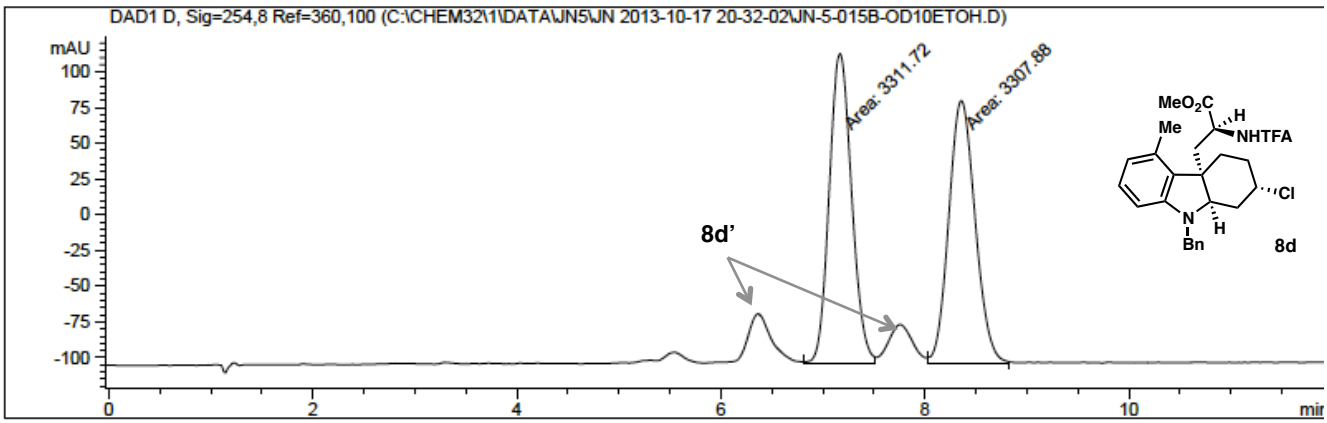
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.606	MM	0.2830	2191.81152	129.09554	50.5084
2	12.333	MM	0.3388	2147.68750	105.65442	49.4916

8c: 86% ee



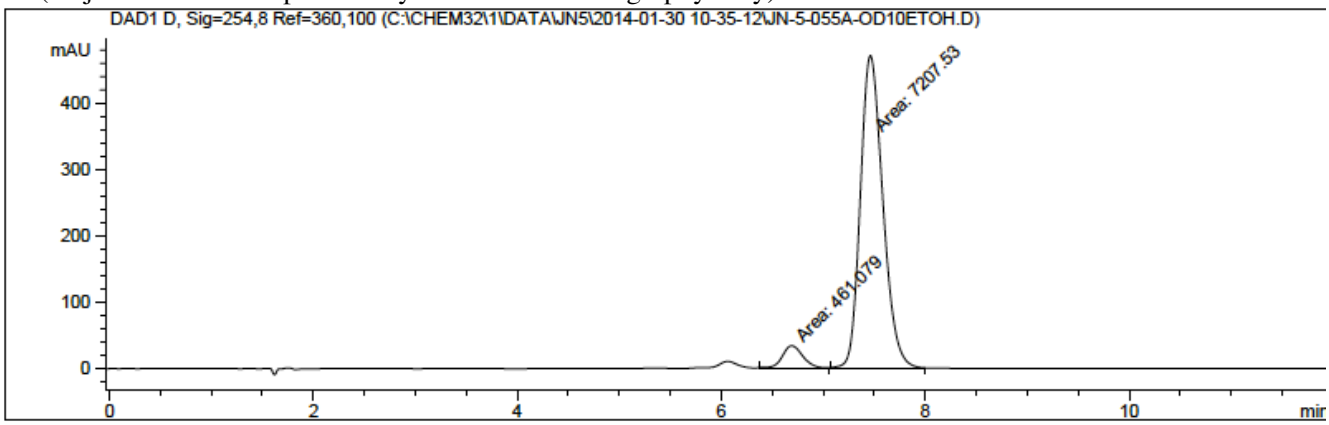
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.082	MM	0.3059	289.07486	15.74899	6.9679
2	12.719	MM	0.3589	3859.57520	179.25320	93.0321

8d: racemic



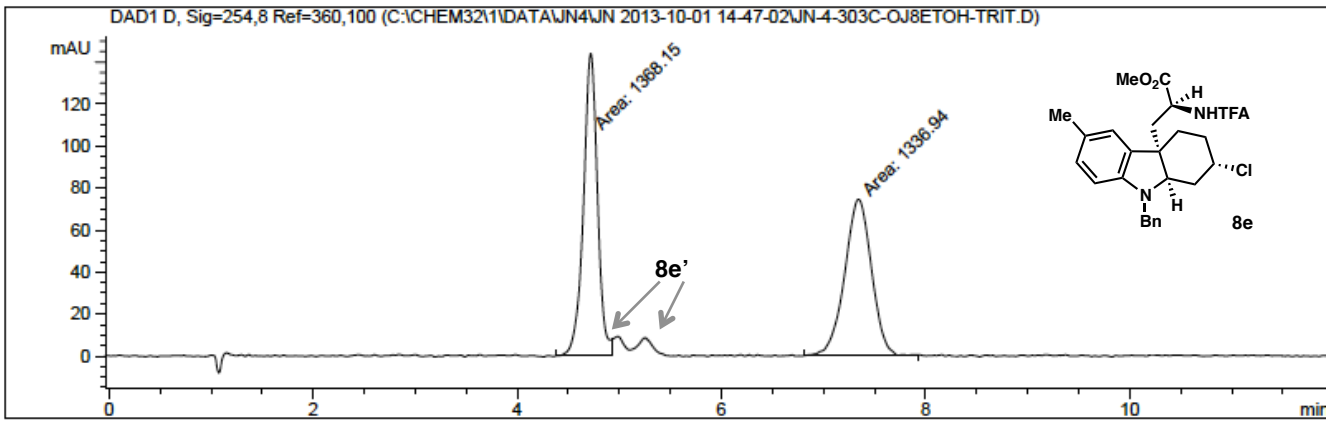
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.167	MM	0.2547	3311.72388	216.69806	50.0291
2	8.356	MM	0.3001	3307.87622	183.70784	49.9709

8d (major diastereomer purified by column chromatography only): 88% ee



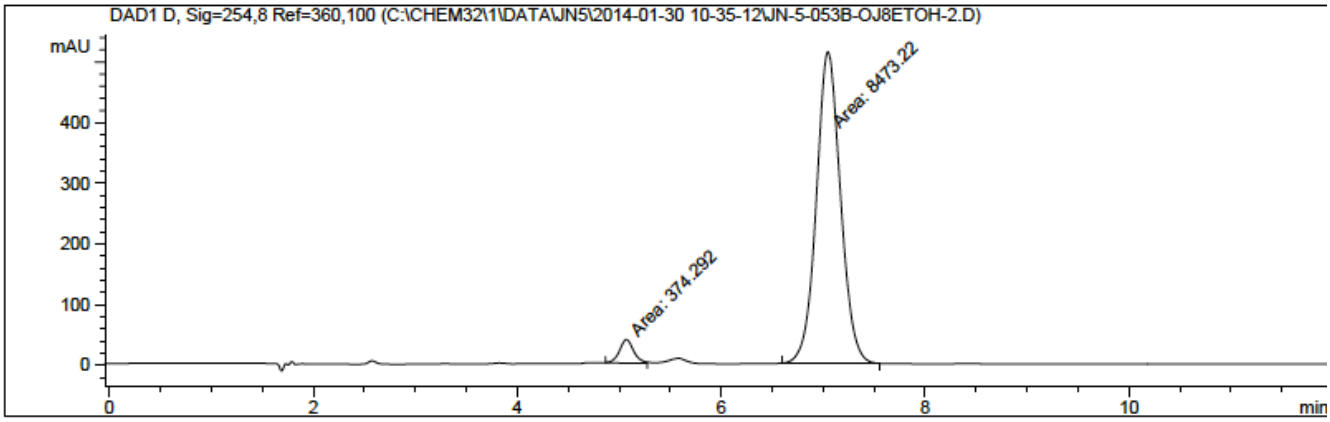
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.689	MM	0.2305	461.07950	33.33388	6.0126
2	7.461	MM	0.2553	7207.52832	470.58481	93.9874

8e: racemic



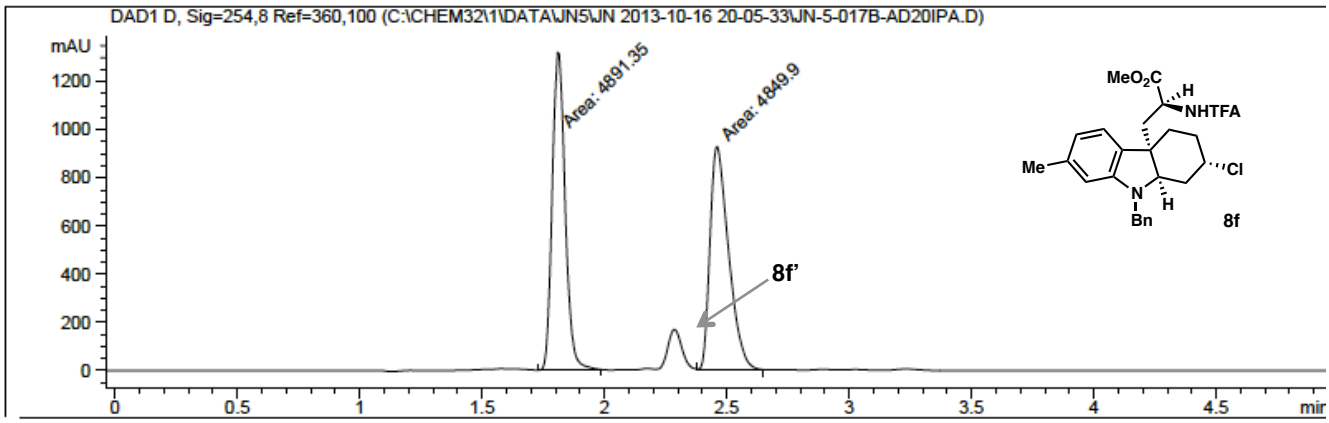
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.720	MM	0.1588	1368.15161	143.62547	50.5769
2	7.343	MM	0.3010	1336.93970	74.03839	49.4231

8e: 92% ee



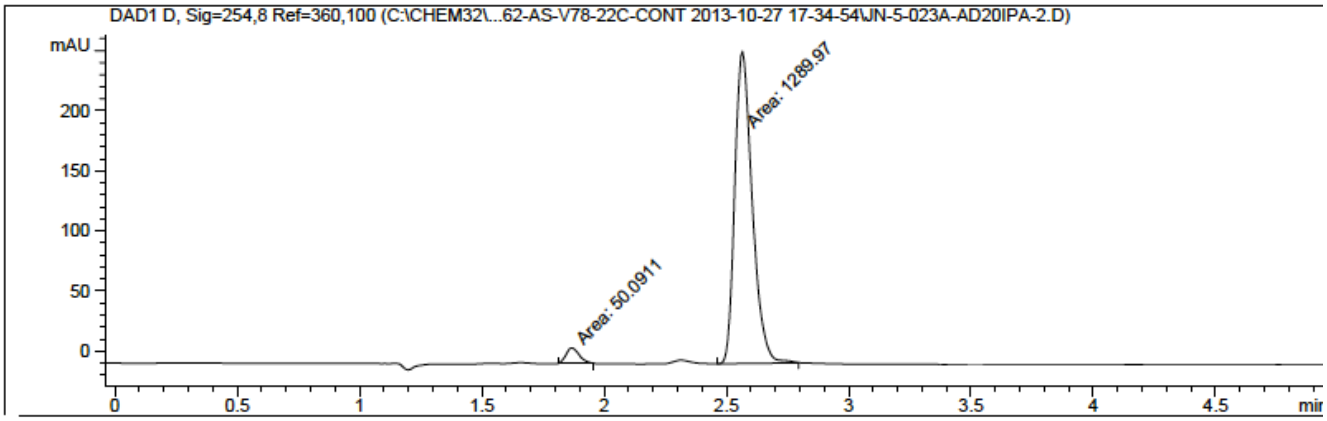
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.071	MM	0.1609	374.29236	38.77222	4.2305
2	7.047	MM	0.2750	8473.22168	513.56281	95.7695

8f: racemic



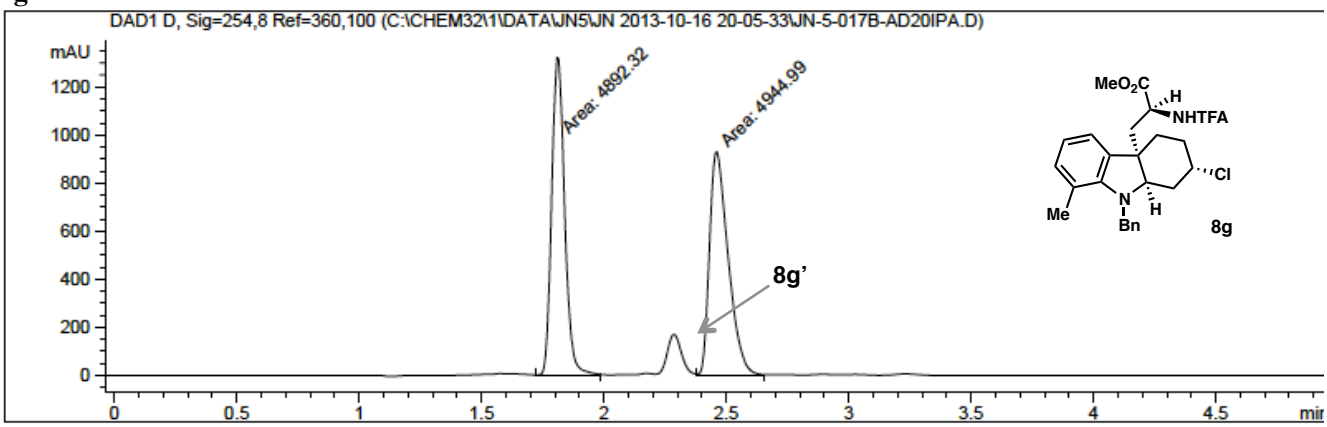
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.812	MM	0.0605	4891.34961	1347.66492	50.2127
2	2.461	MM	0.0868	4849.90088	931.37439	49.7873

8f: 93% ee



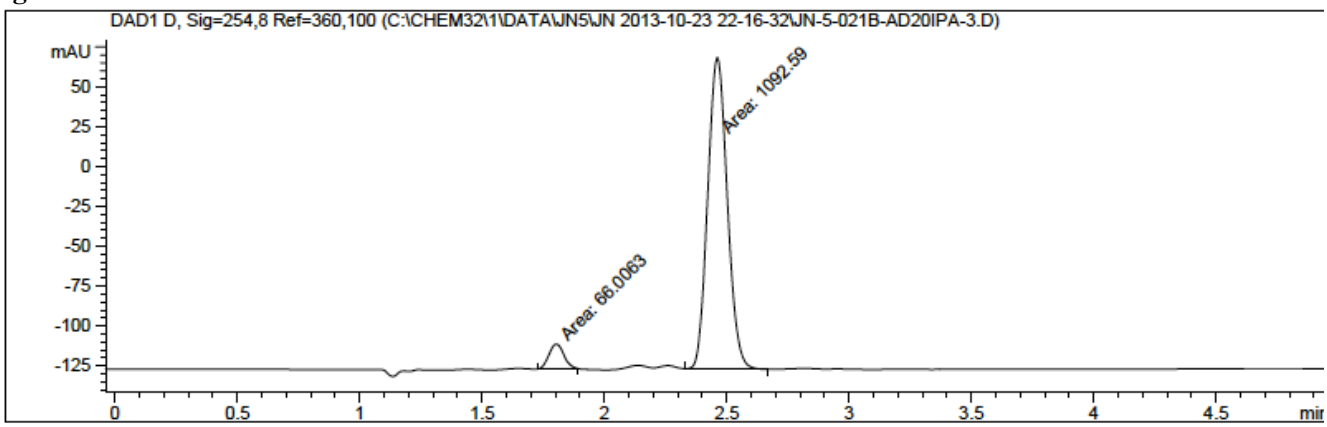
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.869	MM	0.0635	50.09111	13.15408	3.7380
2	2.565	MM	0.0826	1289.97192	260.39725	96.2620

8g: racemic



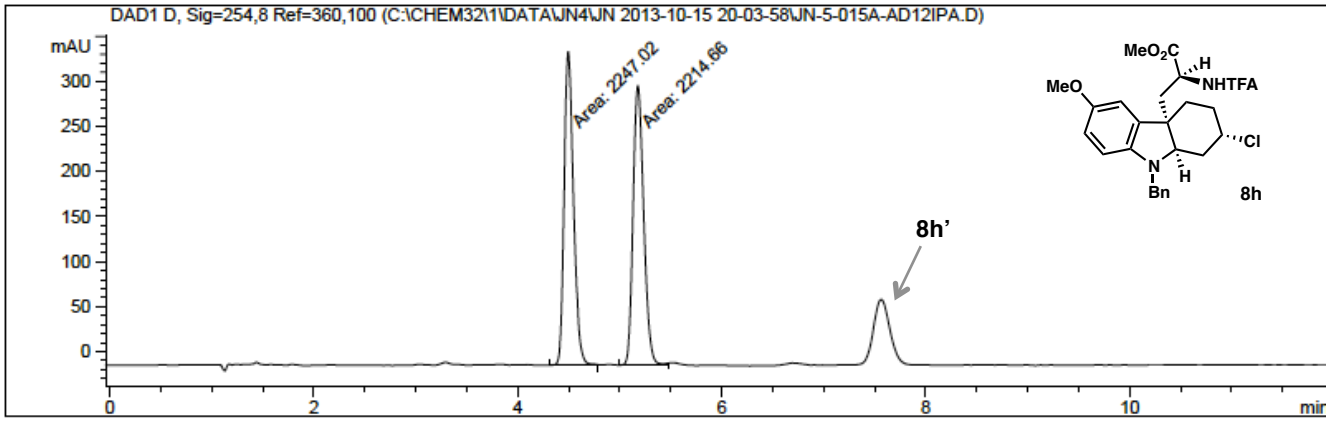
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.812	MM	0.0610	4892.31689	1336.32910	49.7323
2	2.460	MM	0.0883	4944.98926	933.73901	50.2677

8g: 89% ee



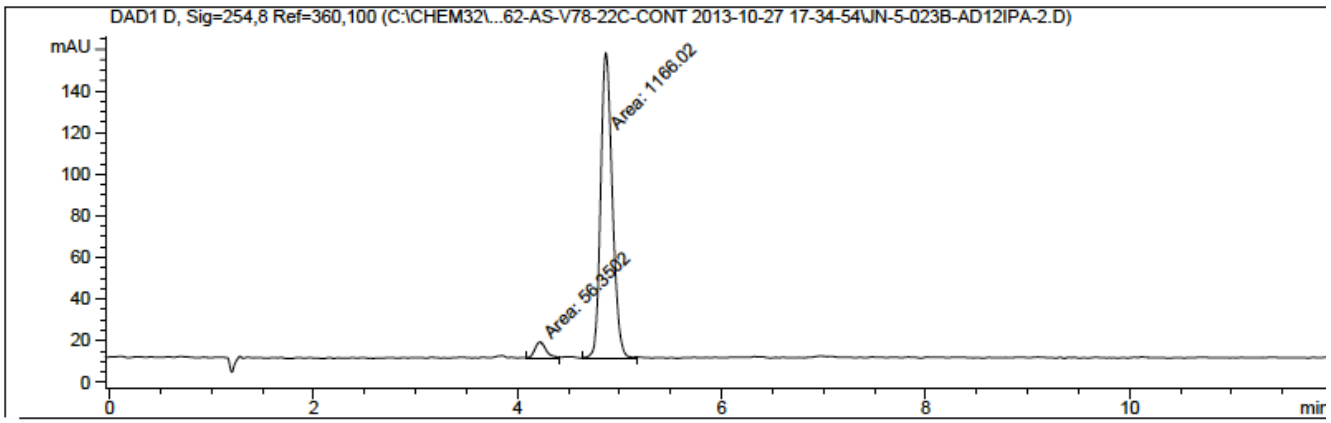
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.804	MM	0.0707	66.00635	15.56793	5.6971
2	2.462	MM	0.0929	1092.58643	196.06543	94.3029

8h: racemic



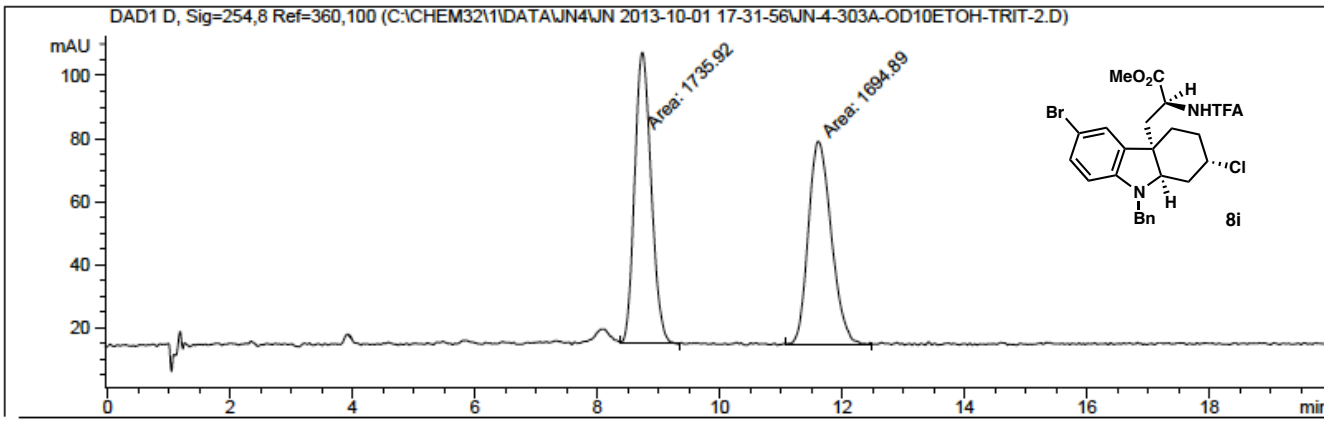
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.496	MM	0.1072	2247.02026	349.30011	50.3626
2	5.178	MM	0.1193	2214.66113	309.27405	49.6374

8h: 91% ee



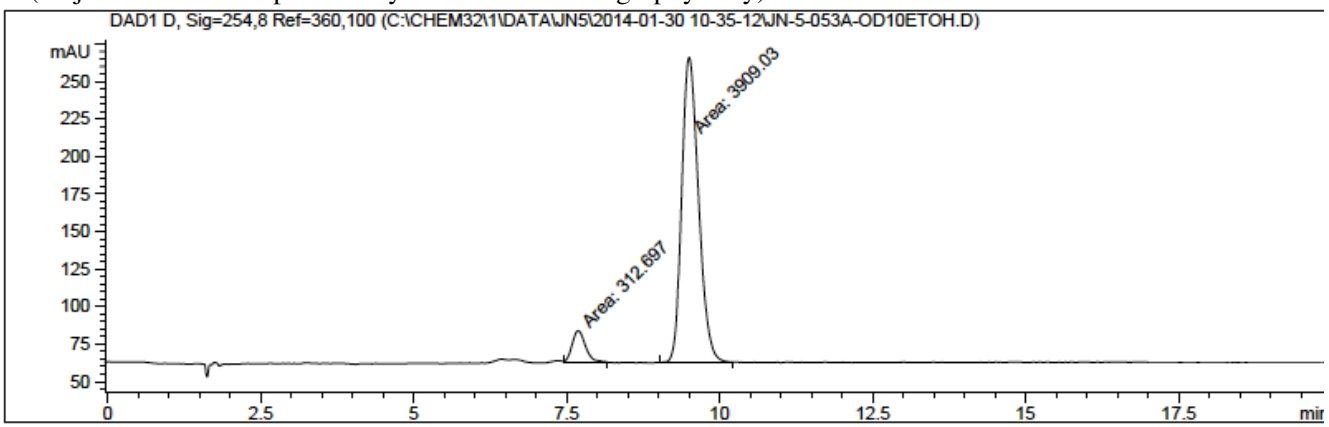
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.219	MM	0.1233	56.35015	7.61557	4.6099
2	4.866	MM	0.1325	1166.02209	146.64792	95.3901

8i: racemic



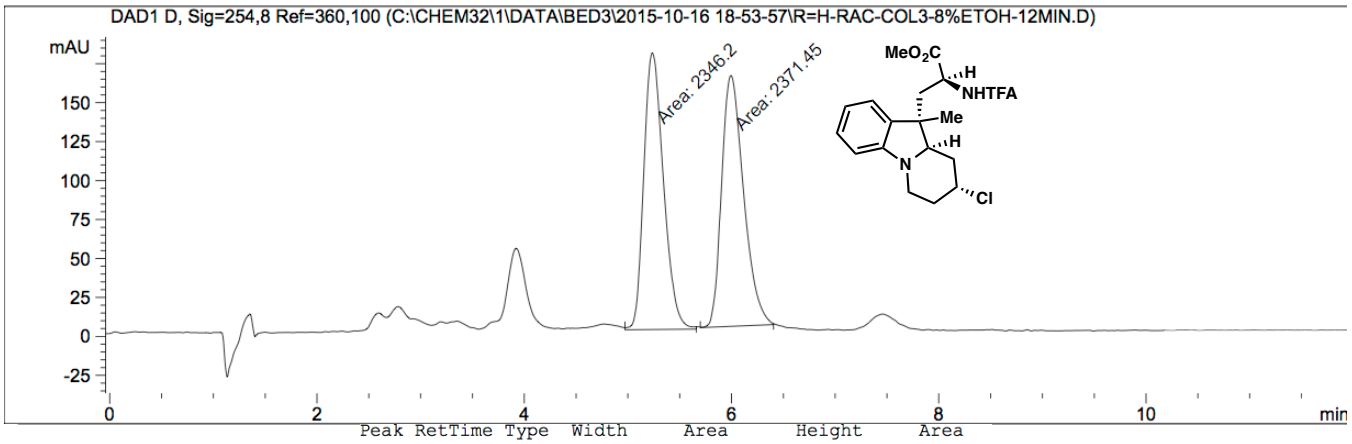
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.739	MM	0.3135	1735.92102	92.27703	50.5979
2	11.611	MM	0.4383	1694.89441	64.44508	49.4021

8i (major diastereomer purified by column chromatography only): 85% ee



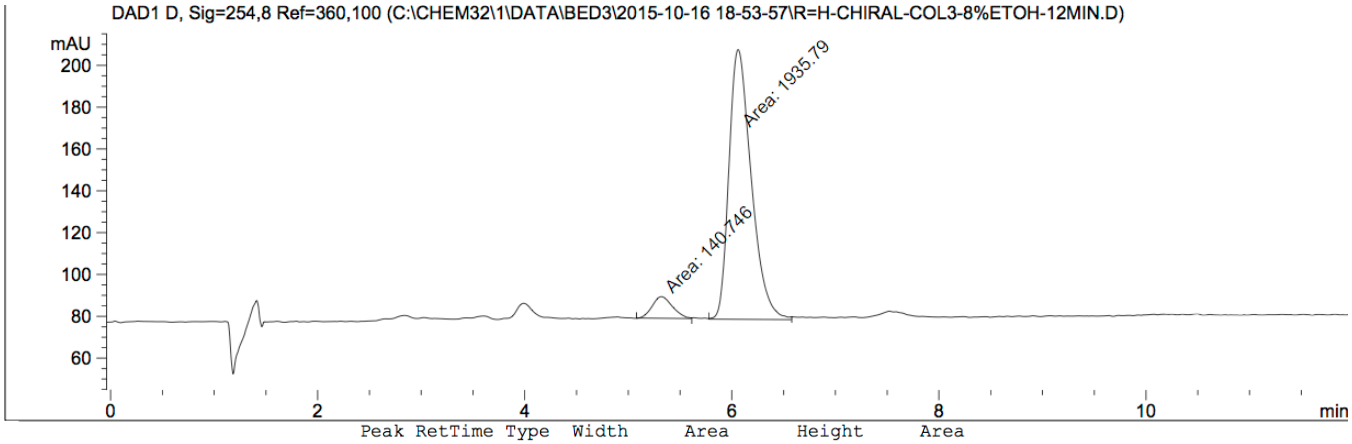
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.690	MM	0.2497	312.69736	20.87528	7.4069
2	9.505	MM	0.3200	3909.02954	203.61522	92.5931

10a: racemic



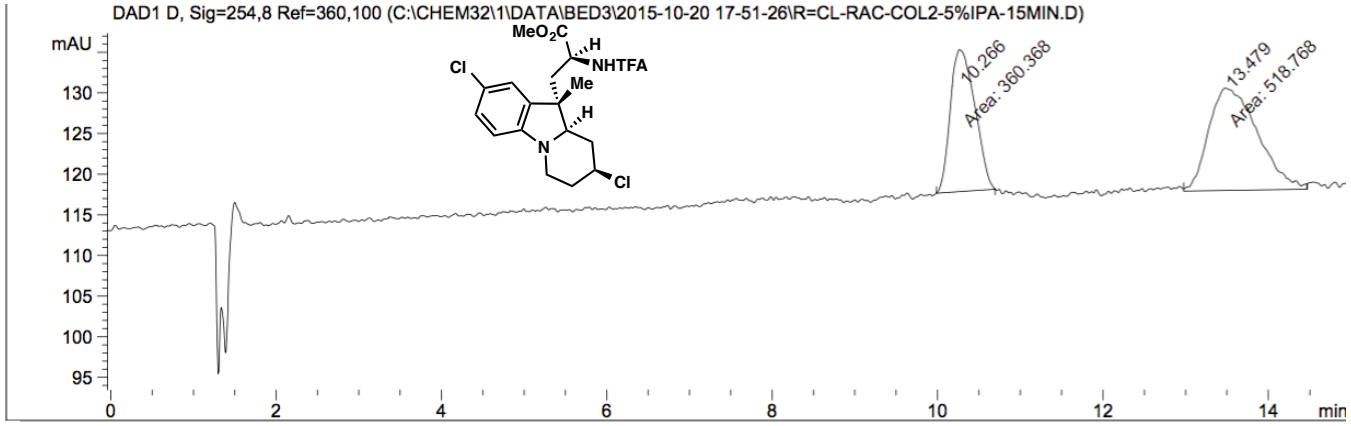
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.236	MM	0.2202	2346.19751	177.59077	49.7324
2	5.994	MM	0.2455	2371.44751	161.00914	50.2676

10a: 86% ee



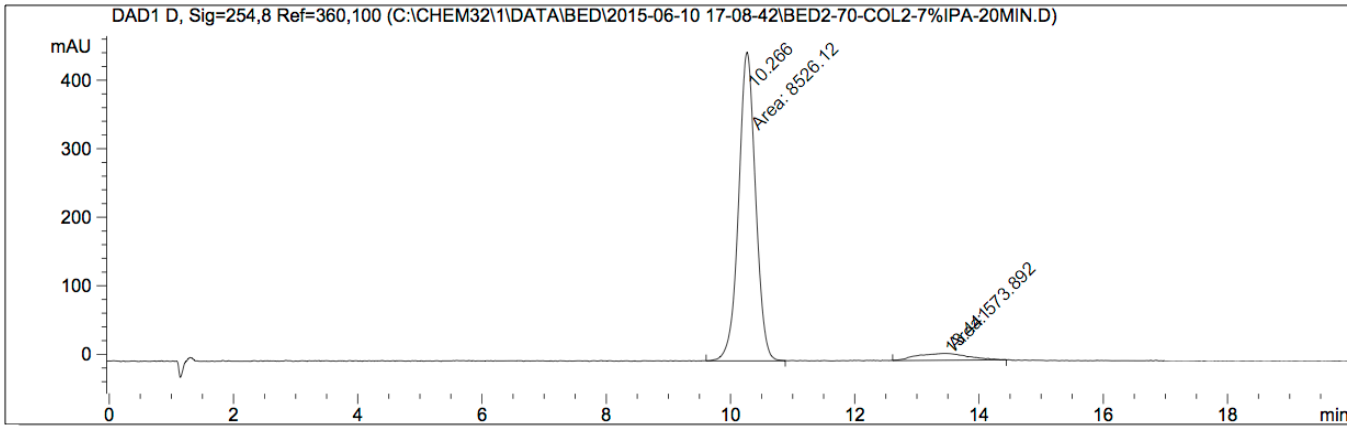
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.317	MM	0.2285	140.74594	10.26775	6.7779
2	6.061	MM	0.2502	1935.79187	128.95279	93.2221

10b: racemic



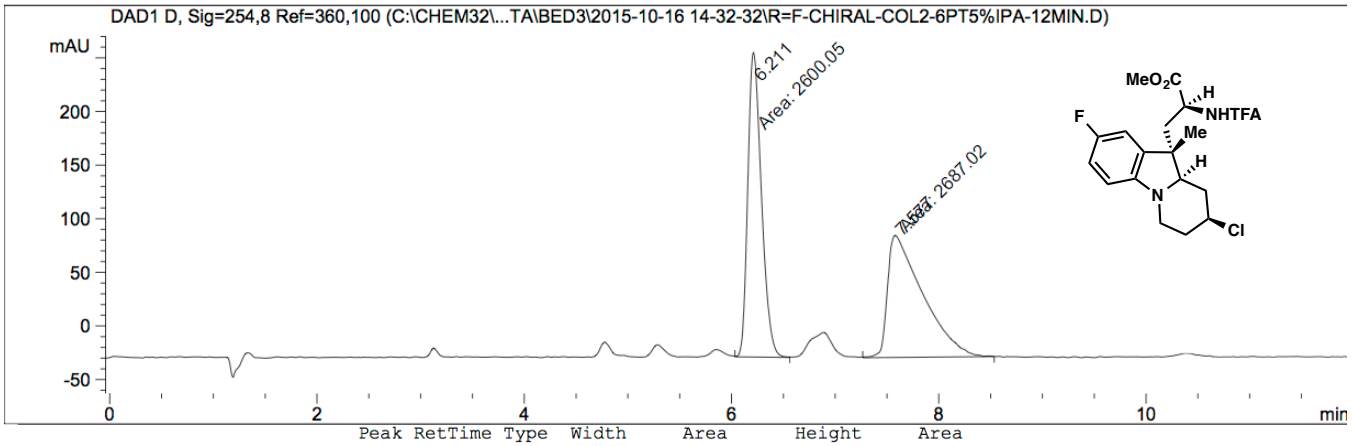
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.266	MM	0.3432	360.36826	17.49969	40.9912
2	13.479	MM	0.6848	518.76770	12.62589	59.0088

10b: 87% ee



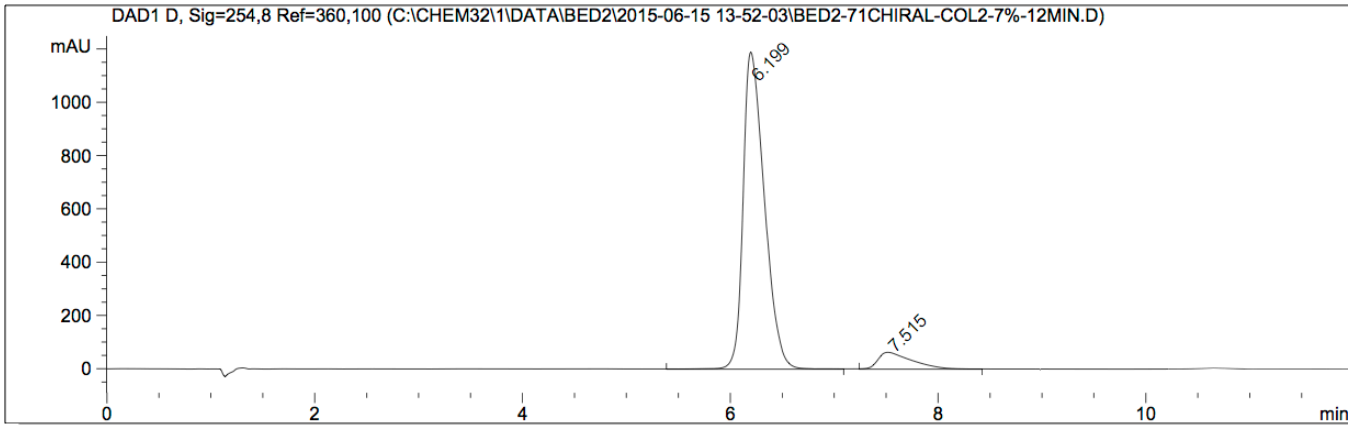
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.266	MM	0.3154	8526.11914	450.50446	93.6935
2	13.441	MM	0.9368	573.89166	10.21014	6.3065

10c: racemic



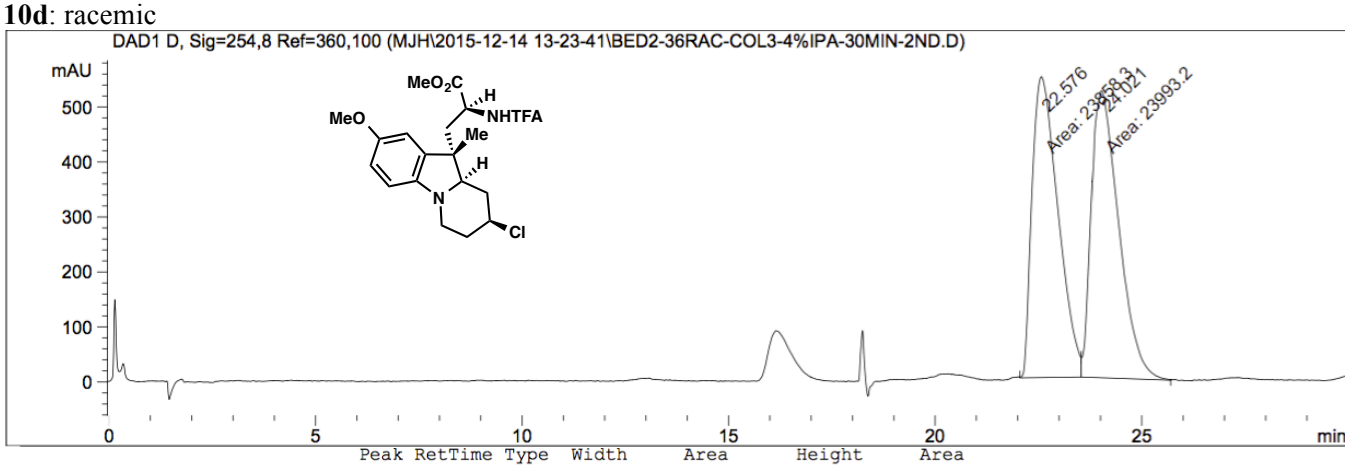
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.211	MM	0.1524	2600.04932	284.43759	49.1775
2	7.577	MM	0.3934	2687.01880	113.83910	50.8225

10c: 84% ee



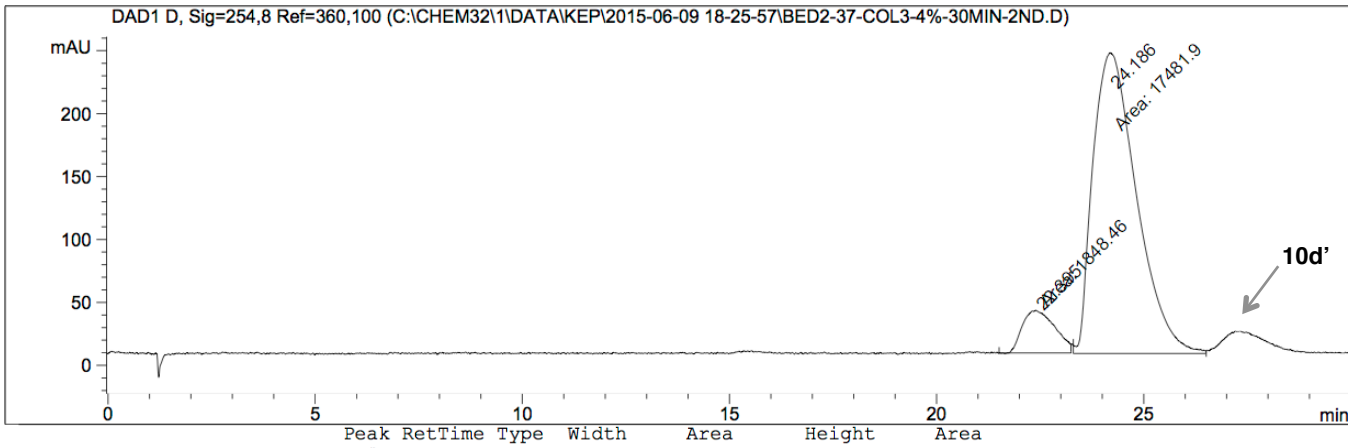
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.199	VB	0.2225	1.69622e4	1188.69360	92.1586
2	7.515	BB	0.3253	1443.23254	63.47738	7.8414

10d: racemic



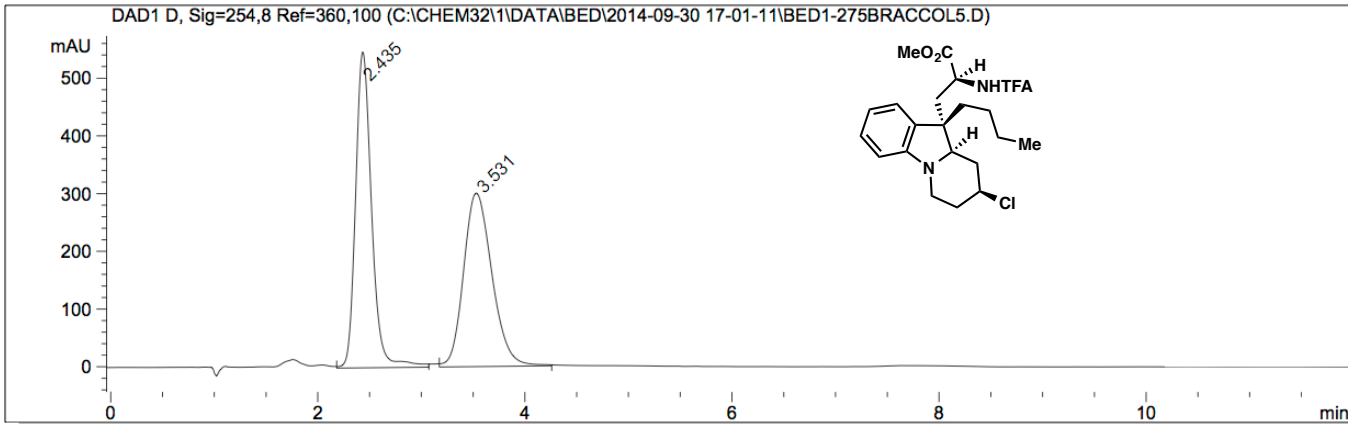
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.576	MM	0.7267	2.38583e4	547.18787	49.8591
2	24.021	MM	0.7659	2.39932e4	522.12537	50.1409

10d: 81% ee



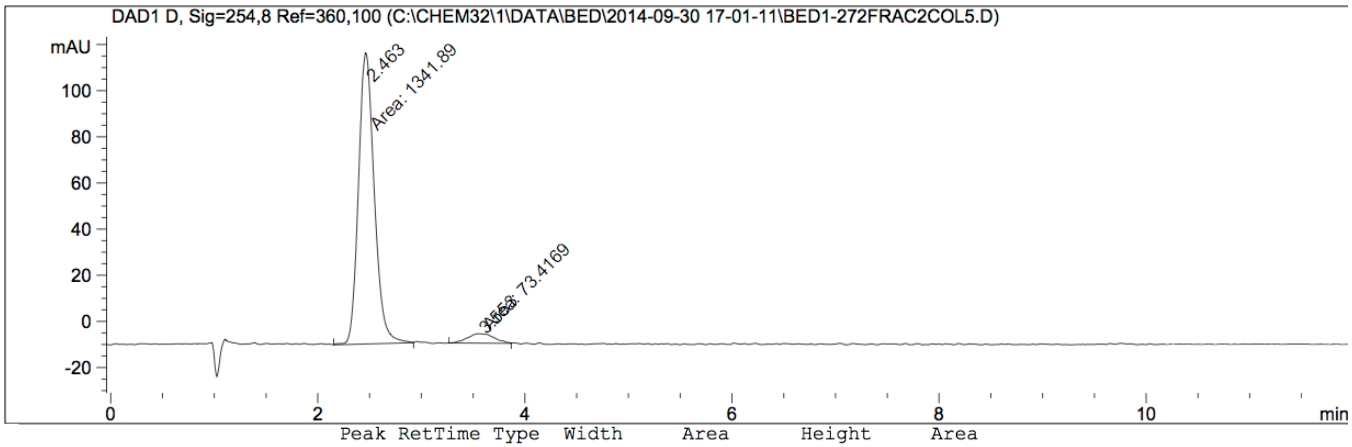
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.395	MM	0.9125	1848.46448	33.76014	9.5625
2	24.186	MM	1.2198	1.74819e4	238.86002	90.4375

10e: racemic



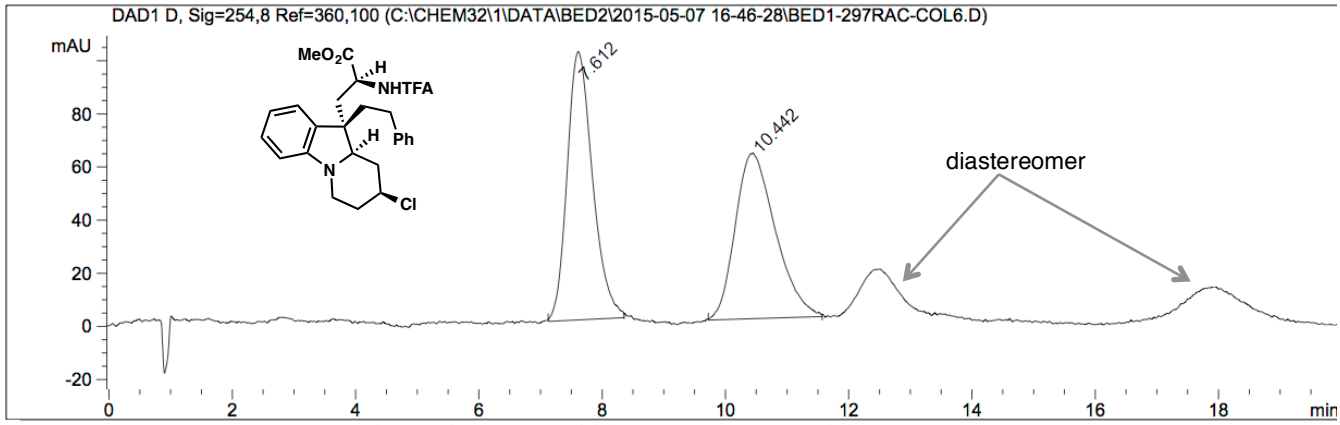
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.435	VB	0.1658	5897.84619	547.18097	50.7050
2	3.531	BB	0.2947	5733.85010	300.42203	49.2950

10e: 90% ee



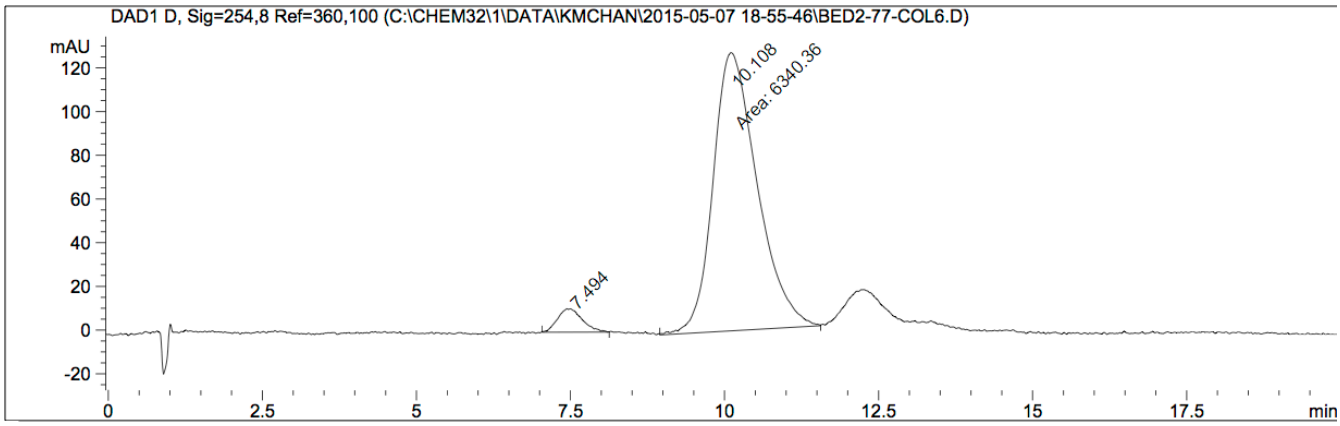
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.463	MM	0.1771	1341.88623	126.29272	94.8126
2	3.553	MM	0.2999	73.41692	4.08012	5.1874

10f: racemic



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.612	BB	0.4109	2899.81396	101.00822	50.7118
2	10.442	BB	0.5397	2818.41040	62.31292	49.2882

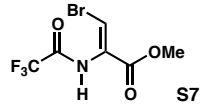
10f: 91% ee



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.494	BB	0.3423	299.89087	10.65778	4.5163
2	10.108	MM	0.8299	6340.36426	127.33179	95.4837

Synthesis of deuterated amidoacrylate 7-d$_1$

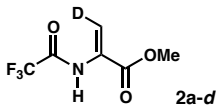
Acrylate **2a** (1.97 g, 10 mmol, 1.0 equiv) was dissolved in 50 mL CH_2Cl_2 and cooled to –78 °C. Molecular bromine (0.51 mL, 10 mmol, 1.0 equiv) was added dropwise, and the reaction was stirred for 10 minutes before moving to an ice bath, where it was stirred for 40 minutes. DABCO (1.1 g, 10 mmol, 1.0 equiv) was added as a solution in 15 mL CH_2Cl_2. The reaction was stirred for 1.5 h, then filtered through celite, and concentrated. The crude mixture was purified by flash chromatography (30% Et_2O/pentane) to provide bromoacrylate **S7** in 74% yield (2.03 g).



S7

S7: ^{1}H NMR (300 MHz, acetone) δ 7.89 (s, 1H), 3.80 (s, 3H); ^{13}C NMR (126 MHz, acetone) δ 161.13, 155.06 (q, J = 37.9 Hz), 130.41, 121.72, 115.86 (q, J = 287.2 Hz), 52.35; IR (NaCl/thin film) 3256, 1733, 1669, 1623, 1506, 1436, 1336, 1232, 1122 cm^{-1}; HRMS (MM) calc'd for $C_6H_5BrF_3NO_3$ $[M+Li]^+$ 280.9551, found 280.0969.

Bromoacrylate **S7** (830 mg, 3 mmol) was dissolved in 6 mL ethyl acetate (not dried), and Pd/$BaSO_4$ (reduced, 29 mg) was added. The reaction was sparged with D_2, then sealed and stirred until the reaction no longer progressed by TLC (approximately four days). The reaction was filtered through celite, concentrated, and purified by flash chromatography (20% Et_2O/pentane) to provide deuterium labeled acrylate **2a-*d*$_1$** in 30% yield (180.5 mg).



2a-*d*$_1$

2a-*d*$_1$: ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.53 (s, 1H), 6.13 (d, J = 1.4 Hz, 1H), 3.92 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.5, 155.1 (q, $J_{C\text{-}F}$ = 38.2 Hz), 129.4, 115.2 (q, $J_{C\text{-}F}$ = 288.3 Hz), 112.1 (t, $J_{C\text{-}D}$ = 26 Hz), 53.47; IR (NaCl/thin film) 3385, 1714, 1539, 1444, 1294, 974, 862, 763, 734 cm^{-1}; HRMS (MM) calc'd for $C_6H_5DF_3N_2O_3$ $[M–H]^–$ 197.0290, found 197.0295.

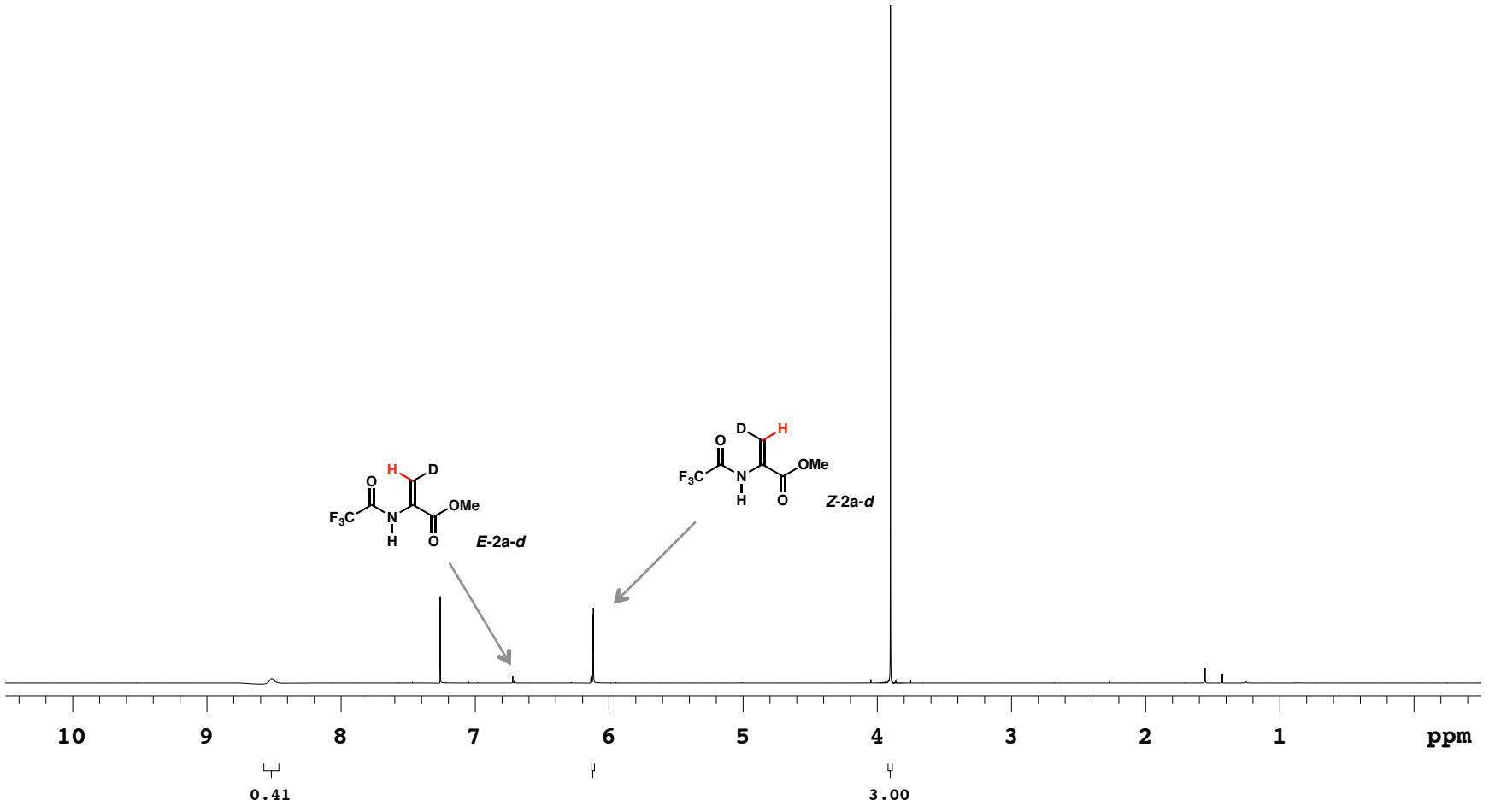
Investigation of the reversibility of the conjugate addition.

To a flame-dried flask was added indole (0.20 mmol, 1.00 equiv), acrylate (0.24 mmol, 1.20 equiv), and (*R*)-3,3'-dibromo-BINOL (0.04 mmol, 0.20 equiv), and 2,6-dibromophenol (0.20 mmol, 1.00 equiv). The flask was charged with DCM (1.5 mL), followed by addition of TMSCl (0.20 mmol, 1.00 equiv), $ZrCl_4$ (0.32 mmol, 1.60 equiv), then stirred at room temperature for 30 min. The reaction was quenched by diluting with 1 mL MeCN and 1 mL 1 M HCl, followed by addition of 5 mL H_2O. The aqueous layer was extracted with ethyl acetate (3 x 5 mL) and the combined organic layers were washed with either saturated $NaHCO_{3(aq)}$ (10 mL). The aqueous layer was back extracted with EtOAc (10 mL) and the combined organic layers were dried (Na_2SO_4), filtered, and concentrated. The ratio of *Z*-**2a**-*d* and *E*-**2a-d** was found to be 1:1 by analysis of the ^{1}H NMR of the crude reaction mixture. The combined yield of product (both diastereomers) was determined to be 44% by ^{1}H NMR by integration of the product signals at 4.55 ppm and 4.63 ppm relative to that of 3,3'-dibromo-BINOL (as an internal standard).

Agilent Technologies

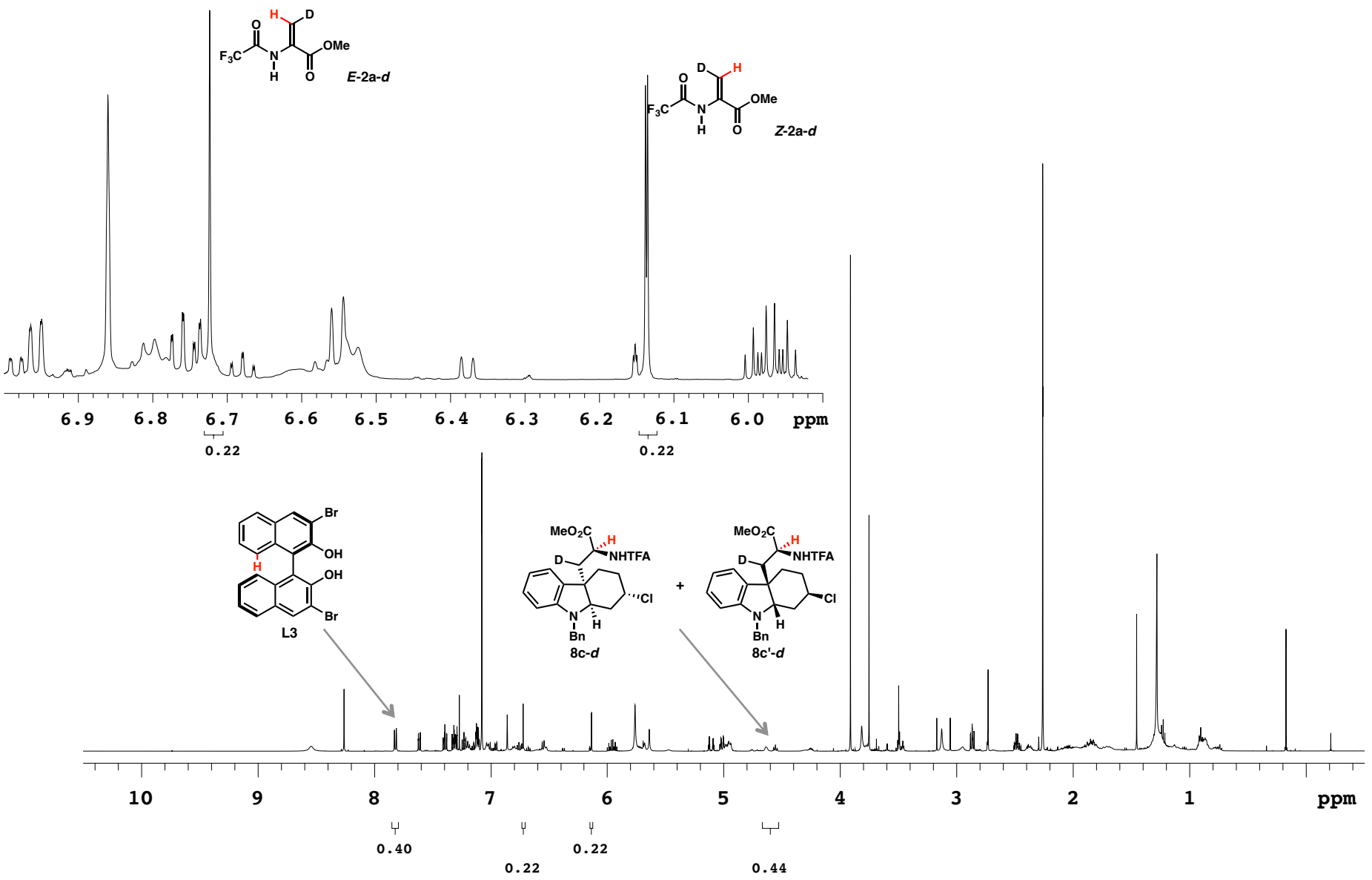
JN-5-117

Sample Name	**JN-5-117**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**jni**
Date collected	**2014-08-05**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



JN-5-121a-crude

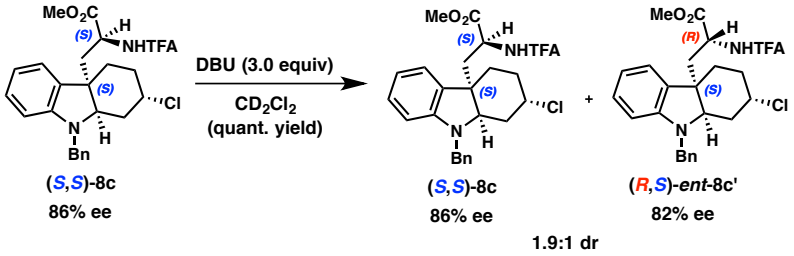
Sample Name	**JN-5-121a-crude**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**jni**
Date collected	**2014-05-11**	Solvent	**cdcl3**	Spectrometer	**indy.caltech.edu-inova500**	Operator	**jni**



Data file /data/indy/jni/vnmrsys/data/JN-5-121a-crude/PROTON01.fid

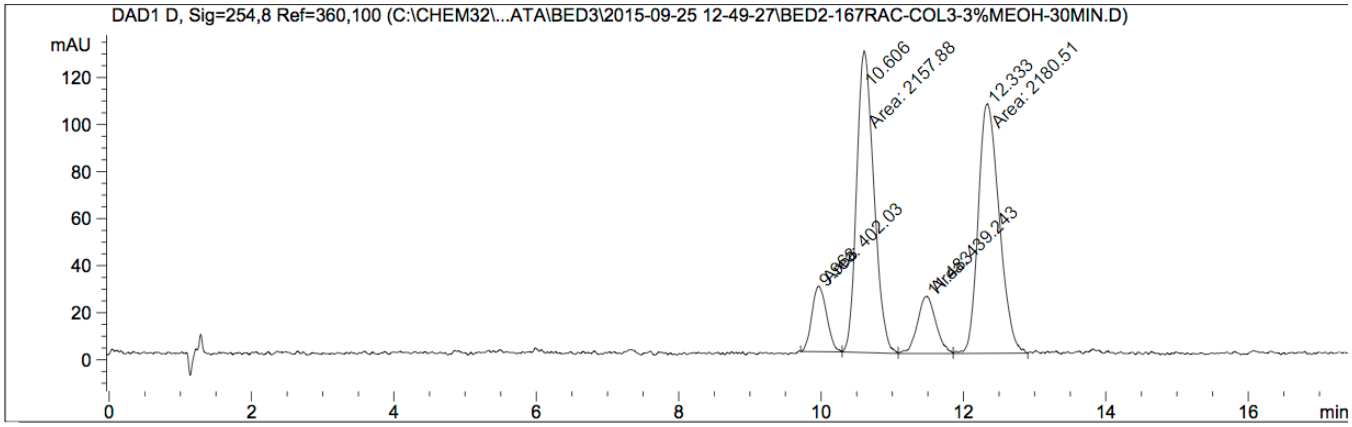
Plot date 2015-11-20

Epimerization Studies



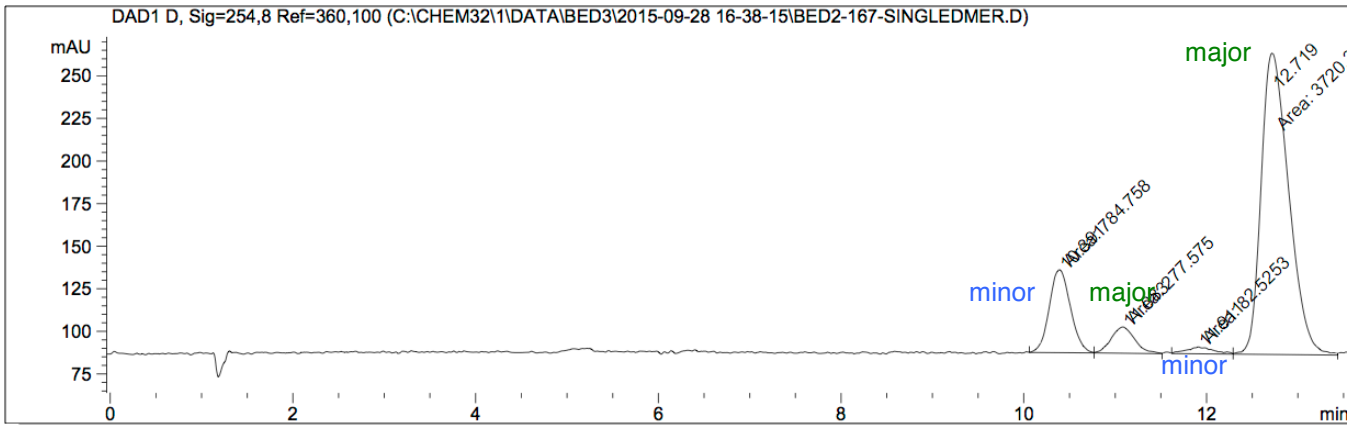
Diastereomerically pure **8c** (1.0 equiv) was dissolved in CD_2Cl_2 and DBU (3.0 equiv) was added. After 24 h, the reaction mixture was concentrated in vacuo. SFC analysis (OD-H, 2.5 mL/min, 3% MeOH in CO_2, λ = 254 nm) showed that the previously diastereomerically pure **8c** was now a 1.8:1 mixture of (*S,S*)-**8c** and (*R,S*)-*ent*-**8c'**, where (*R,S*)-*ent*-**8c'** is the enantiomer of the minor diastereomer originally formed in the Prins reaction.

8c: racemic, mixture of major and minor diastereomers



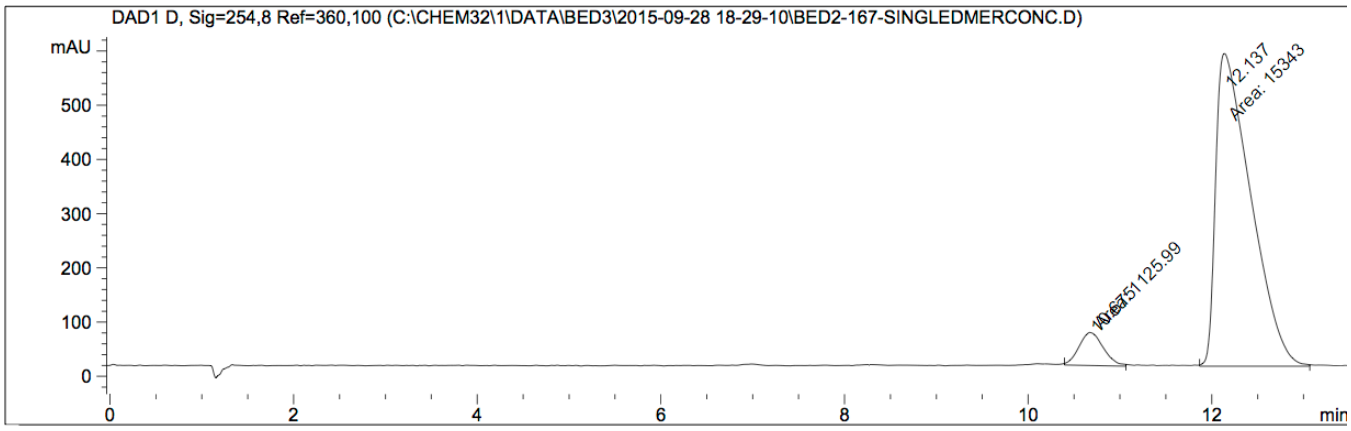
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.968	MM	0.2401	402.03046	27.90723	7.7617
2	10.606	MM	0.2802	2157.87891	128.36487	41.6606
3	11.483	MM	0.3005	439.24347	24.35994	8.4802
4	12.333	MM	0.3424	2180.50879	106.14445	42.0975

8c: enantioenriched, mixture of major and minor diastereomers



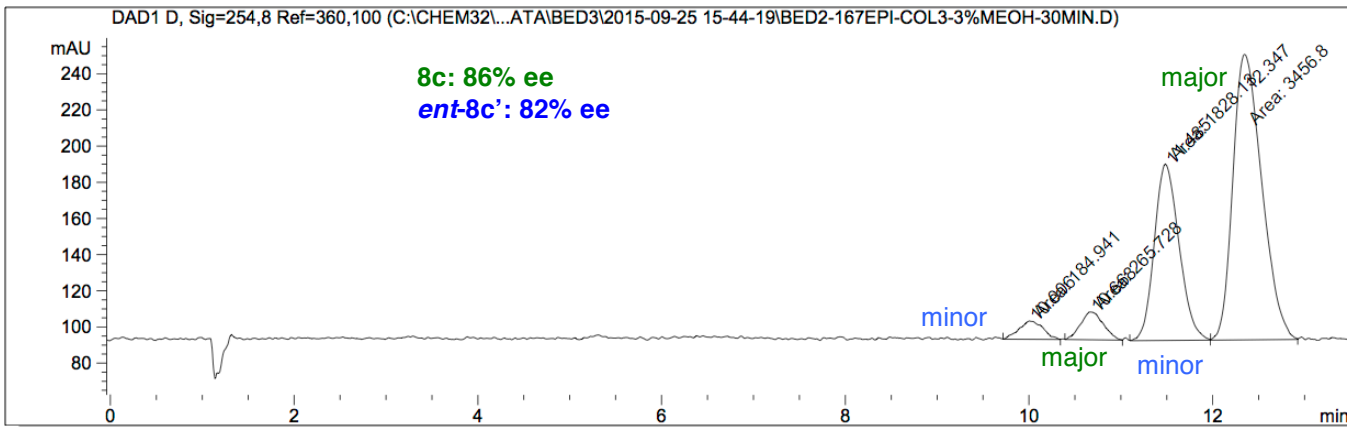
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.391	MM	0.2689	784.75781	48.64483	16.1301
2	11.083	MM	0.2995	277.57465	15.44593	5.7053
3	11.911	MM	0.3468	82.52526	3.96614	1.6962
4	12.719	MM	0.3502	3720.33228	177.07256	76.4684

8c: enantioenriched, major diastereomer, before epimerization



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.675	MM	0.3082	1125.98901	60.88314	6.8370
2	12.137	MM	0.4428	1.53430e4	577.46967	93.1630

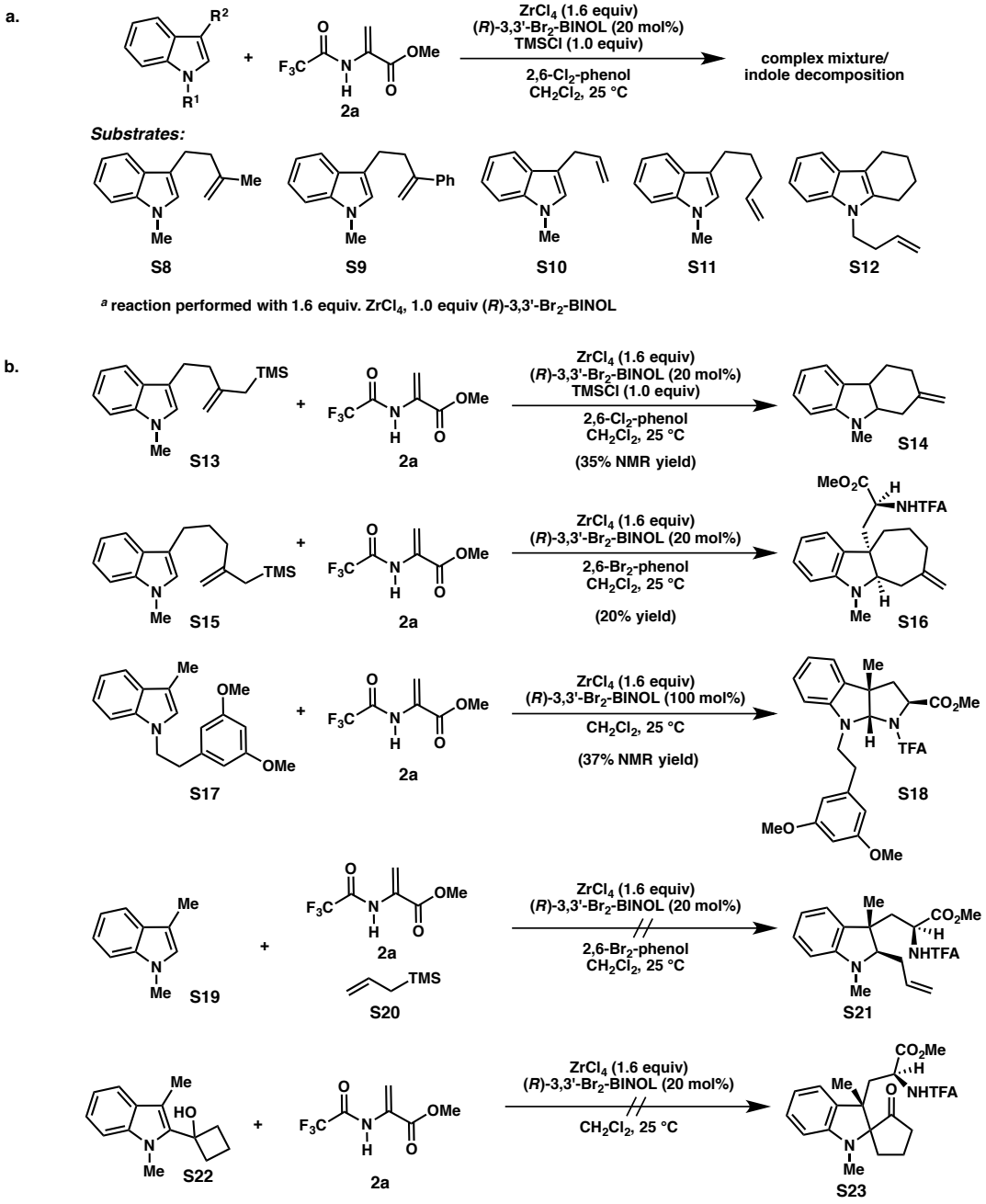
8c and *ent*-**8c'**: after epimerization



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.006	MM	0.3056	184.94051	10.08753	3.2244
2	10.668	MM	0.2887	265.72806	15.34276	4.6330
3	11.485	MM	0.3125	1828.13062	97.48498	31.8734
4	12.347	MM	0.3646	3456.80005	158.02347	60.2692

Selected Unsuccessful Substrates

Scheme S1. Unsuccesful alternative substrates.



Several substrates with alternative tether structures were synthesized. When exposed to the conditions for the conjugate addition/Prins cyclization, many formed complex mixtures of products (Scheme S1). However, some substrates underwent competing reaction mechanisms. For example, the allylsilane moiety of indole **S13** facilitates cyclization such that it occurs at a faster rate than conjugate

addition, and the observed product results from protonation of the indole followed by cyclization. On the other hand, a small amount of the desired product was formed from allylsilane substrate **S15**, likely because cyclization to form the seven-membered ring is slower than the six-membered ring analogue (**S16** vs. **S14**). Friedel–Crafts substrate **S17** failed to undergo cyclization by the aryl ring, forming pyrroloindoline **S18** instead. Intermolecular allylsilane trapping to provide **S21** failed, as did an intramolecular Prins-pinacol rearrangement to afford **S23**.

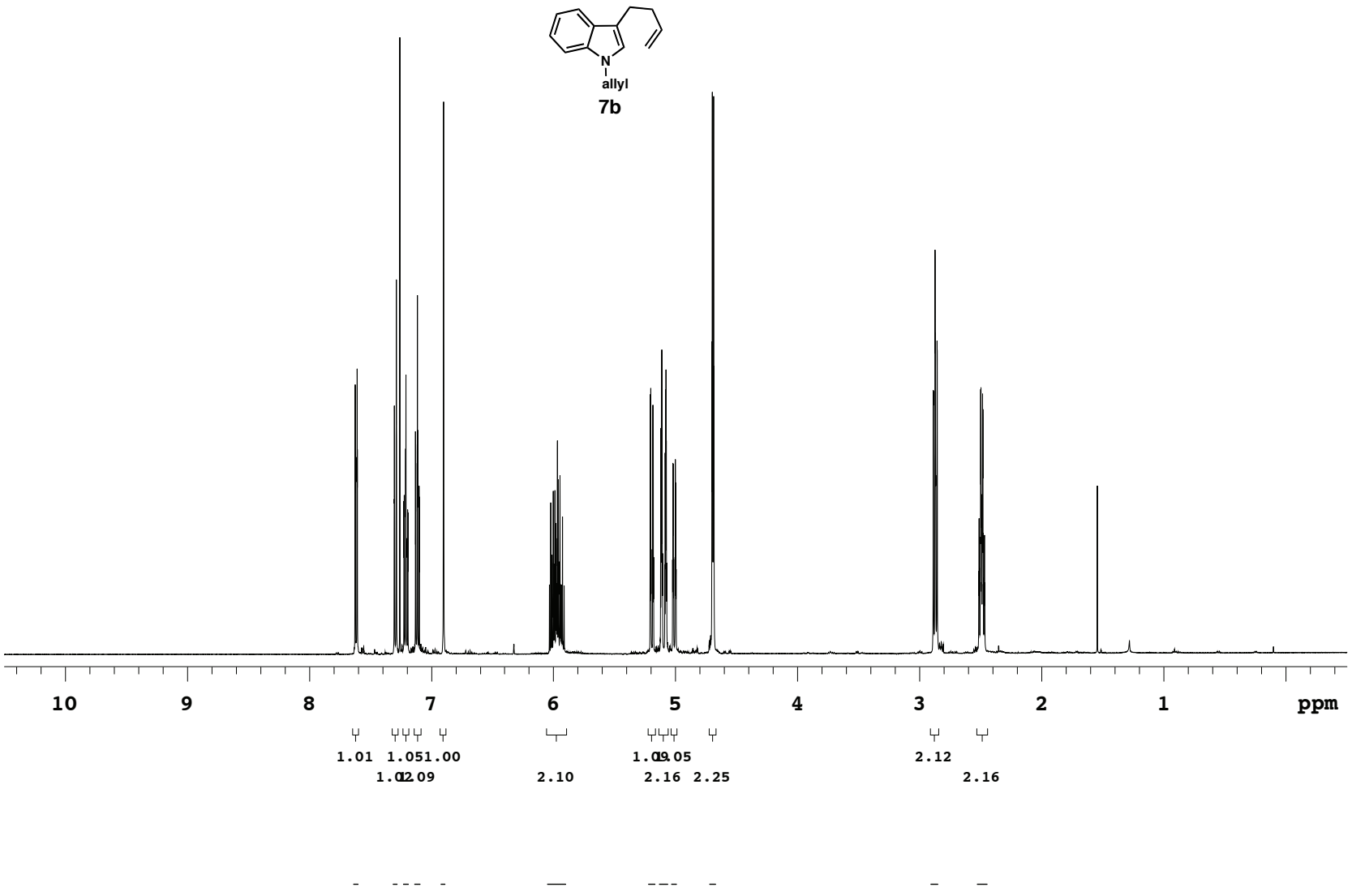
References

(1) Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.
(2) Youn, S.W.; Pastine, S.J.; Sames, D. *Org. Lett.* **2004**, 581.
(3) Liu, C. and Widenhoefer, R. A. *J. Am. Chem. Soc.* **2004**, *126*, 10250.
(4) *Ibid*.
(5) Jørgensen, M. *et. al. Angew. Chem. Int. Ed.* **2008**, *47*, 888.
(6) *Ibid*.
(7) Quancard, J. and Trost, B. M. *J. Am. Chem. Soc.* **2006**, *128*, 6314.
(8) Imm, S. *et. al. Chem Eur. J.* **2010**, *16*, 2705.
(9) Siddiki, S. M. A. H.; Kon, K.; and Shimizu, K. *Chem. Eur. J.* **2013**, *19*, 14416.
(10) Zhang, L. *et. al. Tetrahedron Lett.* **2015**, *56*, 1703.

Agilent Technologies

JN-4-269B-char

Sample Name	**JN-4-269B-char**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-03**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



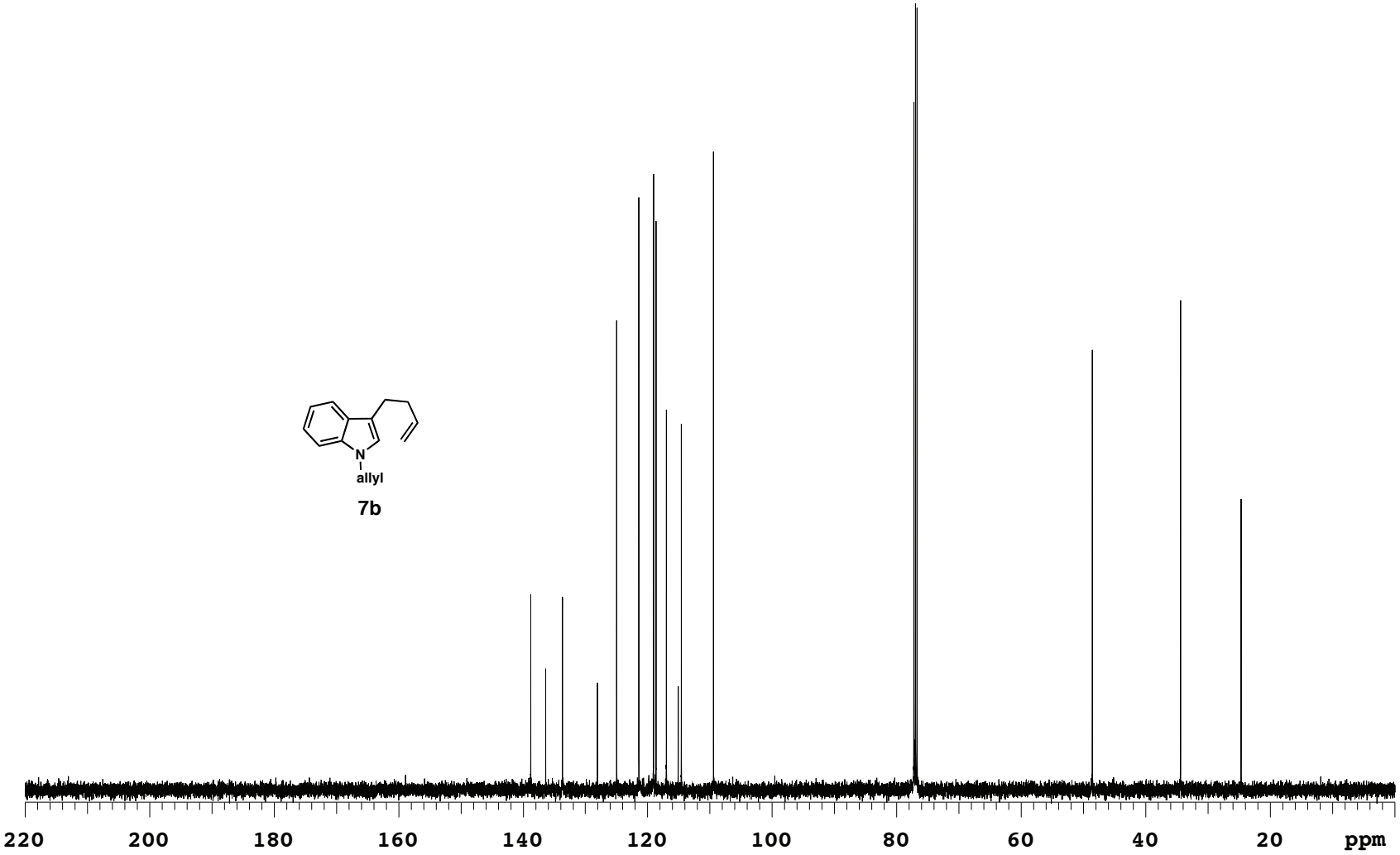
Data file /indy/bdaniels/vnmrsys/data/JN-4-269B-char/PROTON01.fid

Plot date 2015-06-07

Agilent Technologies

JN-4-269B-char

Sample Name	**JN-4-269B-char**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-03**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



Data file /indy/bdaniels/vnmrsys/data/JN-4-269B-char/CARBON01.fid

Plot date 2015-06-07

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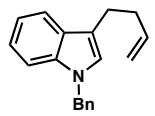
Current Data Parameters
NAME       BED2-86-char
EXPNO                 1
PROCNO                1

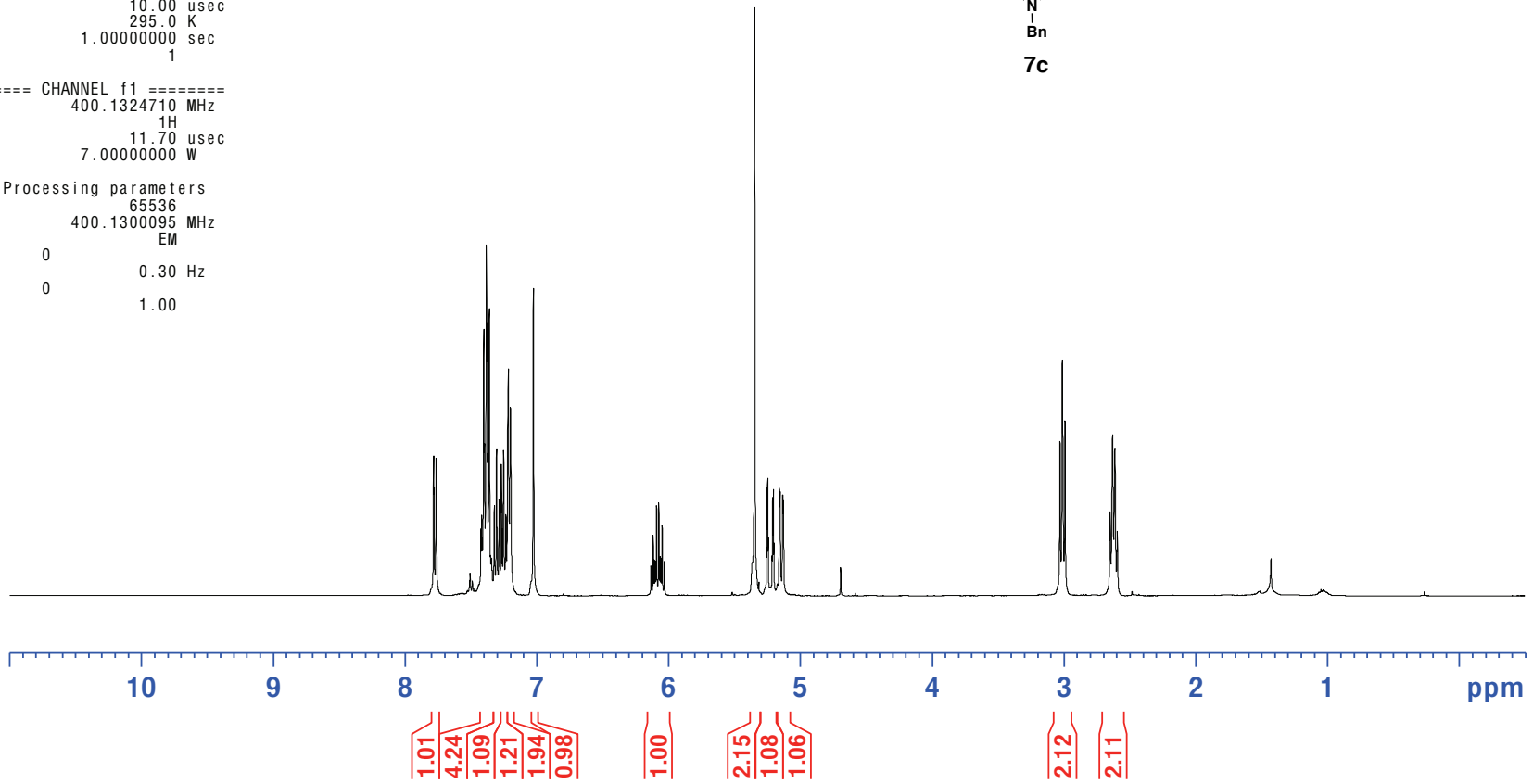
F2 - Acquisition Parameters
Date_          20150608
Time              16.33
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                12.68
DW               62.400 usec
DE               10.00 usec
TE               295.0 K
D1            1.00000000 sec
TD0                   1

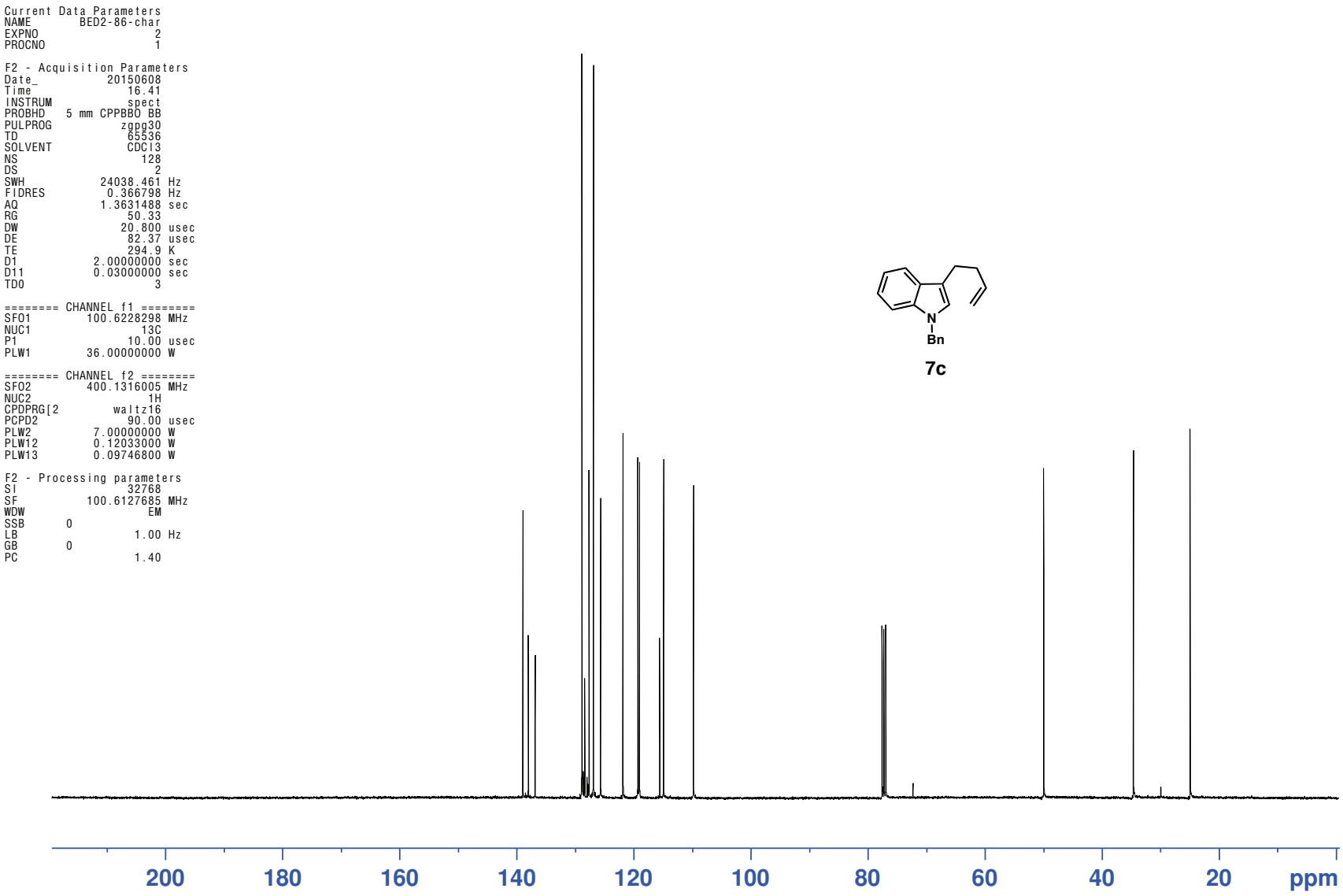
======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                11.70 usec
PLW1          7.00000000 W

F2 - Processing parameters
SI                65536
SF           400.1300095 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



7c

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NAME BED2-86-char
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150608
Time 16.41
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 50.33
DW 20.800 usec
DE 82.37 usec
TE 294.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 3
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 36.00000000 W
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 7.00000000 W
PLW12 0.12033000 W
PLW13 0.09746800 W
F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
Bn
7c
200
180
160
140
120
100
80
60
40
20
ppm

```
Current Data Parameters
NAME      JN-4-301A-char
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150604
Time               15.01
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                197.44
DW                62.400 usec
DE                10.00 usec
TE                295.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 11.70 usec
PLW1          7.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300099 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
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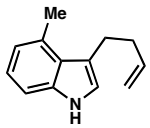
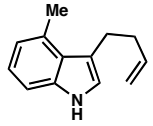


S1

```
Current Data Parameters
NAME      JN-4-301A-char
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150604
Time              15.09
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  384
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                78.69
DW               20.800 usec
DE                82.37 usec
TE                295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   3

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        36.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          7.00000000 W
PLW12         0.12033000 W
PLW13         0.09746800 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```

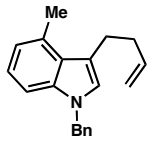


S1

Agilent Technologies

JN-5-013A-char

Sample Name	**JN-5-013A-char**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-03**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



7d

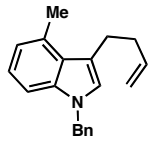
Data file /indy/bdaniels/vnmrsys/data/JN-5-013A-char/PROTON01.fid

Plot date 2015-06-07

Agilent Technologies

JN-5-013A-char

Sample Name	**JN-5-013A-char**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-03**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



7d

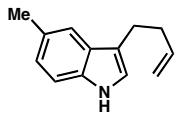
Data file /indy/bdaniels/vnmrsys/data/JN-5-013A-char/CARBON01.fid

Plot date 2015-06-07

Agilent Technologies

BED2-87char

Sample Name	**BED2-87char**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-10**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



S2

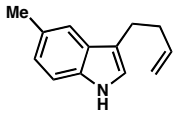
Data file /data/indy/bdaniels/vnmrsys/data/BED2-87char/PROTON01.fid

Plot date 2015-06-11

Agilent Technologies

BED2-87char

Sample Name	**BED2-87char**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-10**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



S2

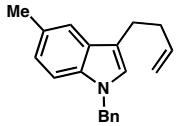
Data file /data/indy/bdaniels/vnmrsys/data/BED2-87char/CARBON01.fid

Plot date 2015-06-11

Agilent Technologies

JN-4-299-char

Sample Name	**JN-4-299-char**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-03**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



7e

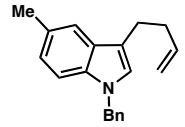
Data file /indy/bdaniels/vnmrsys/data/JN-4-299-char/PROTON01.fid

Plot date 2015-06-07

Agilent Technologies

JN-4-299-char

Sample Name	**JN-4-299-char**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-03**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**

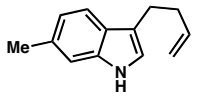


7e

Data file /indy/bdaniels/vnmrsys/data/JN-4-299-char/CARBON01.fid

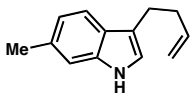
Plot date 2015-06-07

```
Current Data Parameters
NAME      JN-4-301B-char
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150604
Time               15.28
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                 98.88
DW                62.400 usec
DE                10.00 usec
TE                295.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 11.70 usec
PLW1          7.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300098 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



S3

```
Current Data Parameters
NAME      JN-4-301B-char
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150604
Time              15.35
INSTRUM           spect
PROBHD    5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  384
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                 87.75
DW                20.800 usec
DE                 82.37 usec
TE                 295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   3

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        36.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          7.00000000 W
PLW12         0.12033000 W
PLW13         0.09746800 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

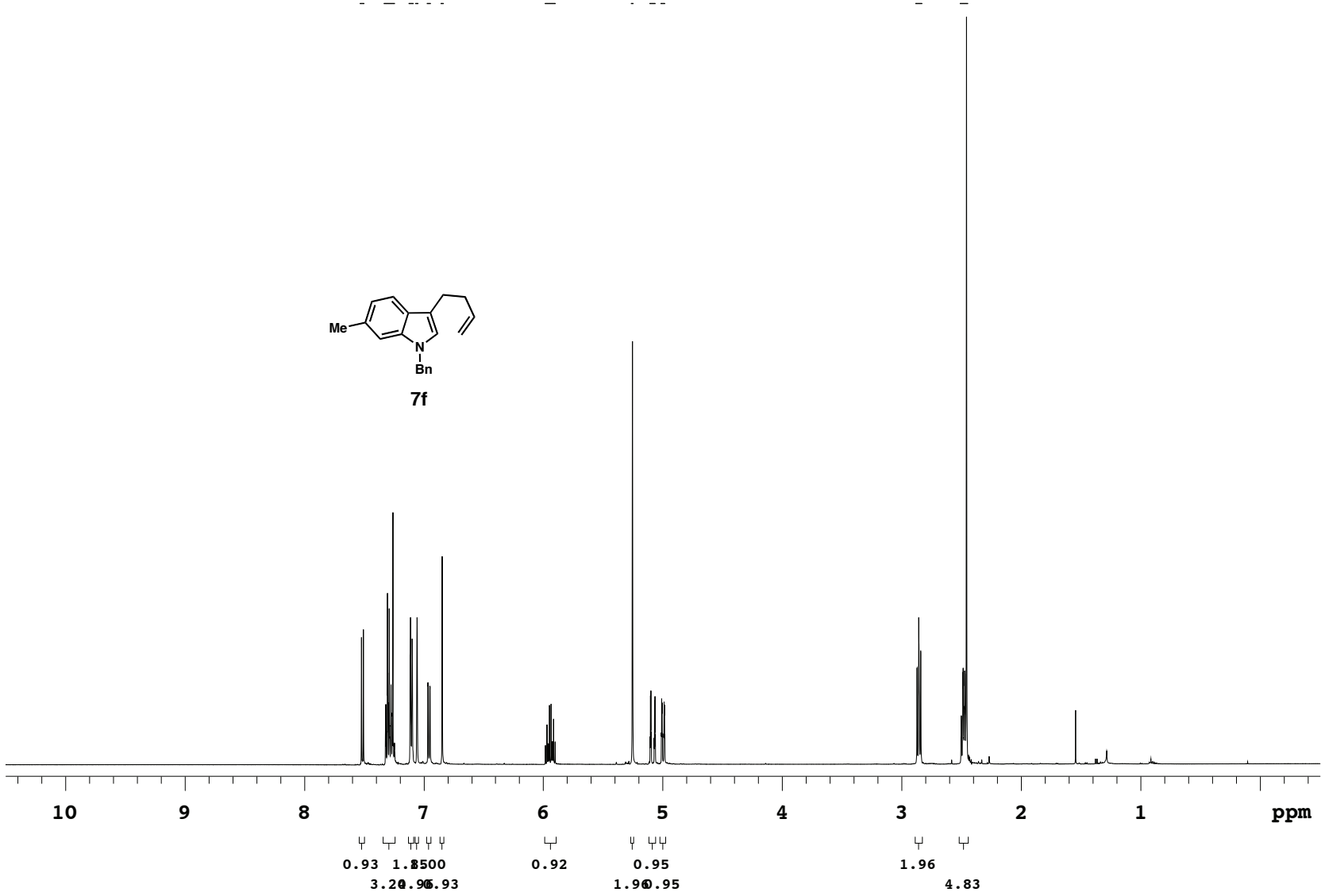


S3

Agilent Technologies

JN-5-013B-char

Sample Name	JN-5-013B-char	Pulse sequence	PROTON	Temperature	25	Study owner	bdaniels
Date collected	2015-06-03	Solvent	cdcl3	Spectrometer	-vnmrs400	Operator	autouser



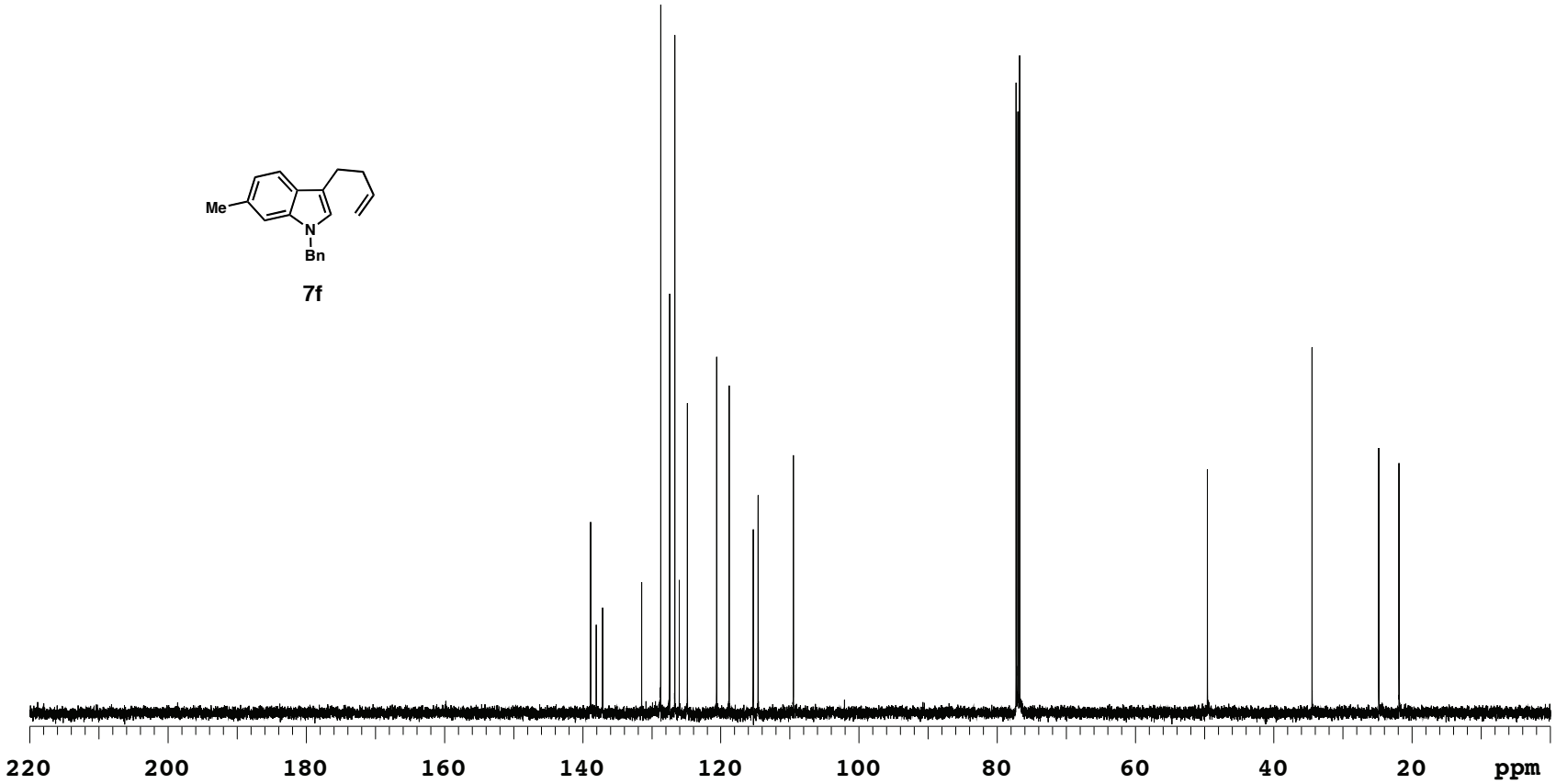
Data file /indy/bdaniels/vnmrsys/data/JN-5-013B-char/PROTON01.fid

Plot date 2015-06-07

Agilent Technologies

JN-5-013B-char

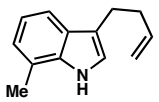
Sample Name	**JN-5-013B-char**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-03**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



Data file /indy/bdaniels/vnmrsys/data/JN-5-013B-char/CARBON01.fid

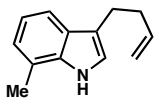
Plot date 2015-06-07

```
Current Data Parameters
NAME      JN-5-011-char
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_           20150604
Time              15.55
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                87.75
DW               62.400 usec
DE               10.00 usec
TE               295.0 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                1H
P1                11.70 usec
PLW1          7.00000000 W

F2 - Processing parameters
SI                65536
SF           400.1300098 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



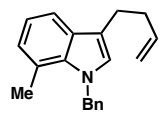
S4

```
Current Data Parameters
NAME      JN-5-011-char
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_           20150604
Time              16.02
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  384
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                87.75
DW               20.800 usec
DE                82.37 usec
TE                294.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   3

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        36.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          7.00000000 W
PLW12         0.12033000 W
PLW13         0.09746800 W

F2 - Processing parameters
SI                32768
SF          100.6127685 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



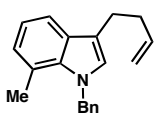
S4

```
Current Data Parameters
NAME      JN-5-013C-char
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150604
Time               16.21
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 98.88
DW                62.400 usec
DE                10.00 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 11.70 usec
PLW1          7.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300099 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



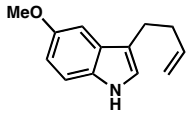
7g

```
Current Data Parameters
NAME      JN-5-013C-char
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150604
Time              16.29
INSTRUM           spect
PROBHD    5 mm CPPBBO BB
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  384
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                78.69
DW               20.800 usec
DE                82.37 usec
TE                295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   3

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        36.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          7.00000000 W
PLW12         0.12033000 W
PLW13         0.09746800 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



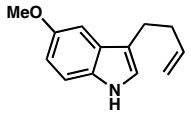
7g

```
Current Data Parameters
NAME      JN-4-285A-char
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150604
Time               14.34
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 98.88
DW                62.400 usec
DE                10.00 usec
TE                294.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 11.70 usec
PLW1          7.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300099 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



S5

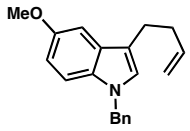
```
Current Data Parameters
NAME      JN-4-285A-char
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150604
Time              14.42
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  384
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                 87.75
DW                20.800 usec
DE                 82.37 usec
TE                 295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   3

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        36.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          7.00000000 W
PLW12         0.12033000 W
PLW13         0.09746800 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



S5

Agilent Technologies

Sample Name	**JN-4-295A-char**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-03**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



7h

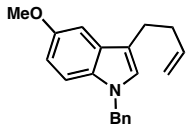
Data file /indy/bdaniels/vnmrsys/data/JN-4-295A-char/PROTON01.fid

Plot date 2015-06-07

Agilent Technologies

JN-4-295A-char

Sample Name	**JN-4-295A-char**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-03**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



7h

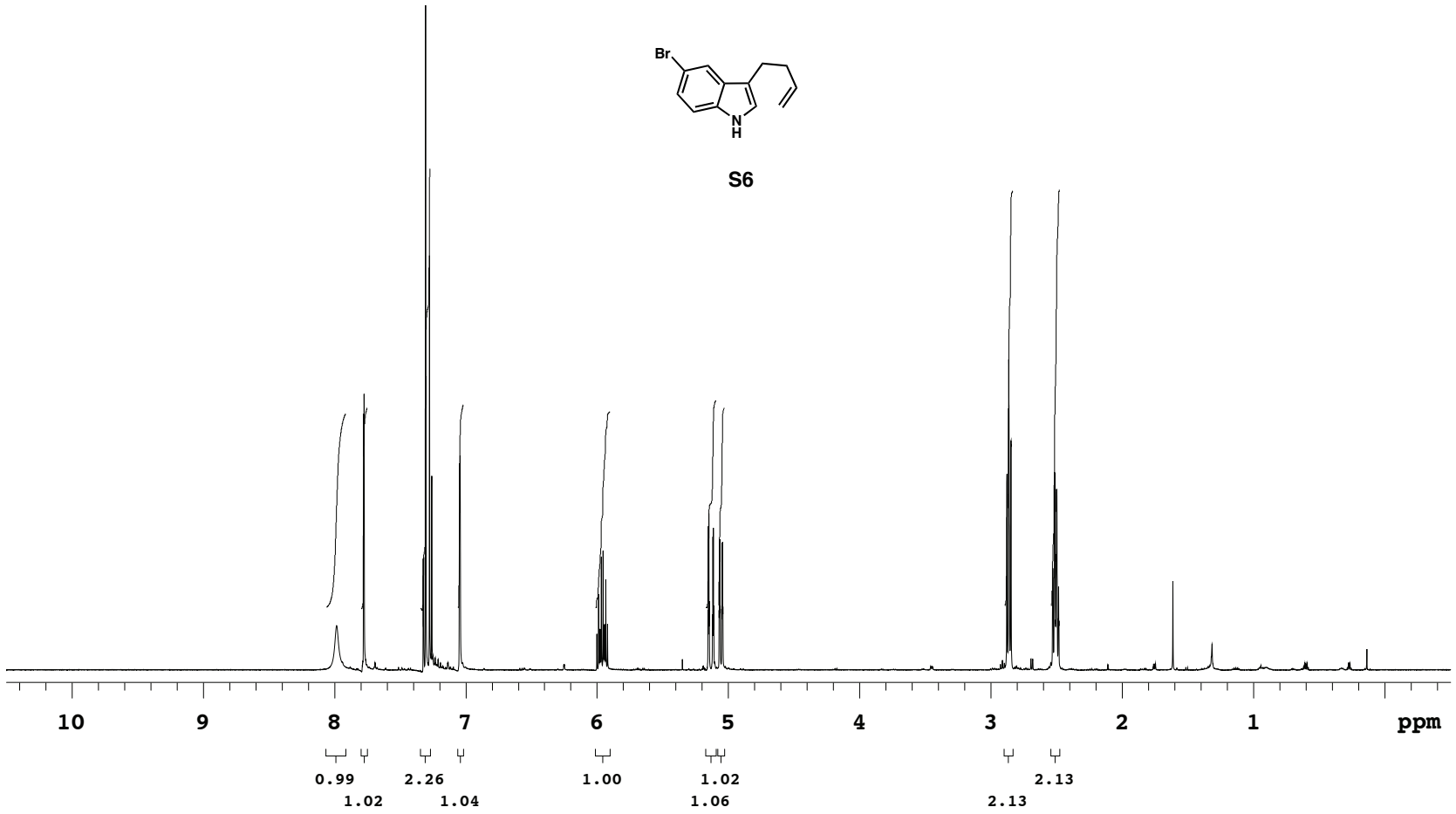
Data file /indy/bdaniels/vnmrsys/data/JN-4-295A-char/CARBON01.fid

Plot date 2015-06-07

Agilent Technologies

BED2-88char

Sample Name	**BED2-88char**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-10**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



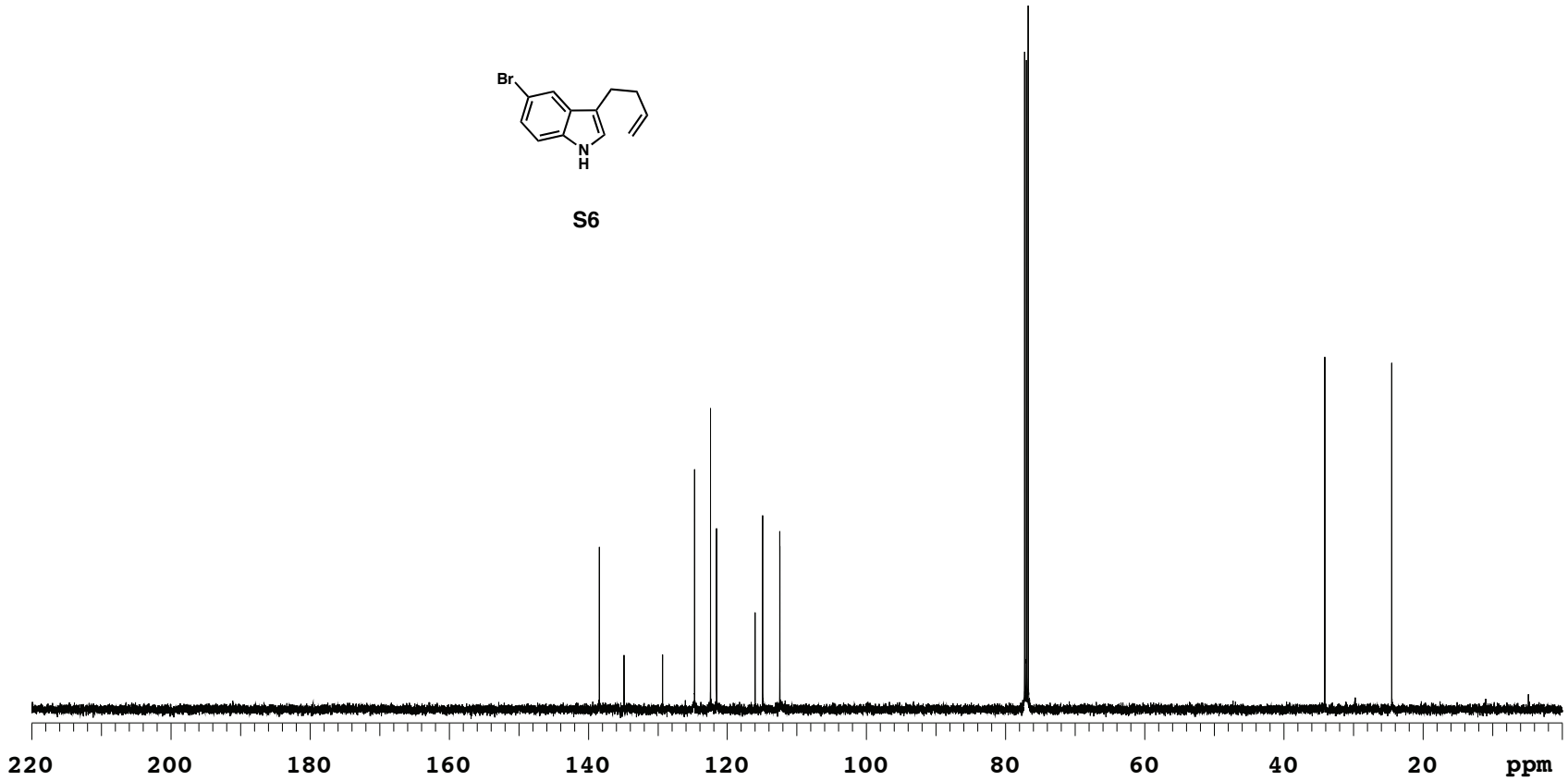
Data file /data/indy/bdaniels/vnmrsys/data/BED2-88char/PROTON01.fid

Plot date 2015-06-11

Agilent Technologies

BED2-88char

Sample Name	**BED2-88char**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-10**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



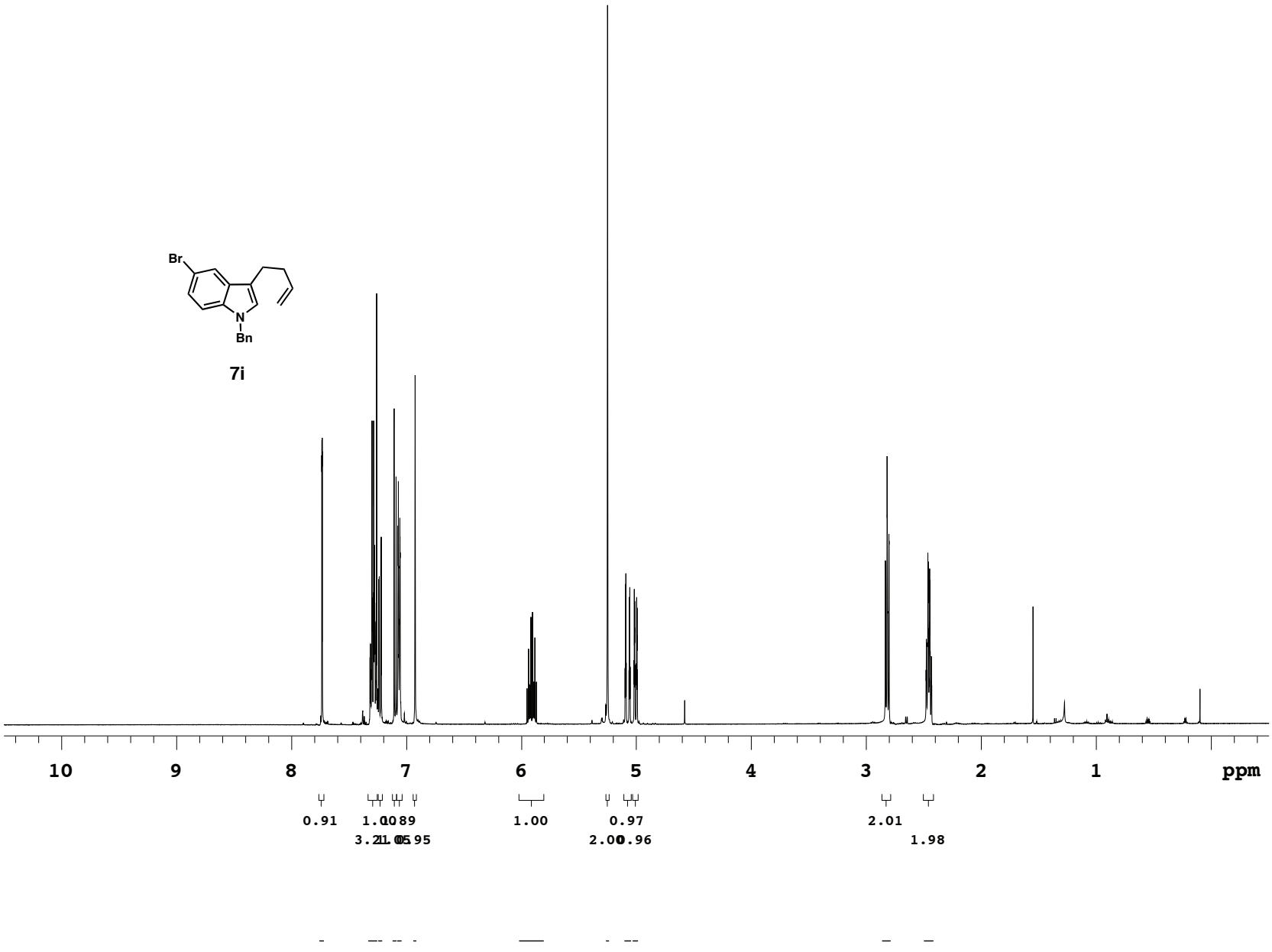
Data file /data/indy/bdaniels/vnmrsys/data/BED2-88char/CARBON01.fid

Plot date 2015-06-11

Agilent Technologies

JN-4-295B-char

Sample Name	**JN-4-295B-char**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-03**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



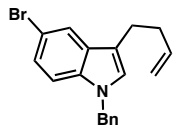
Data file /indy/bdaniels/vnmrsys/data/JN-4-295B-char/PROTON01.fid

Plot date 2015-06-07

Agilent Technologies

JN-4-295B-char

Sample Name	**JN-4-295B-char**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-06-03**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



7i

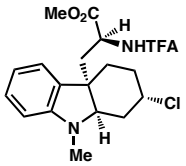
Data file /indy/bdaniels/vnmrsys/data/JN-4-295B-char/CARBON01.fid

Plot date 2015-06-07

Agilent Technologies

JN-5-061a-col2

Sample Name	JN-5-061a-col2	Pulse sequence	PROTON	Temperature	25	Study owner	jni
Date collected	2014-02-26	Solvent	cdcl3	Spectrometer	-vnmrs400	Operator	autouser



8a

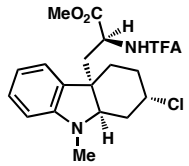
Data file /data/indy/jni/vnmrsys/data/JN-5-061a-col2/PROTON01.fid

Plot date 2015-06-09

Agilent Technologies

JN-5-061a-col2

Sample Name	**JN-5-061a-col2**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**jni**
Date collected	**2014-02-26**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



8a

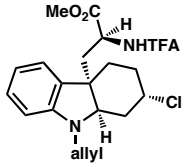
Data file /data/indy/jni/vnmrsys/data/JN-5-061a-col2/CARBON01.fid

Plot date 2015-06-09

Agilent Technologies

JN-4-239b-rxst-Mar6

Sample Name	**JN-4-239b-rxst-Mar6**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**jni**
Date collected	**2014-03-06**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



8b

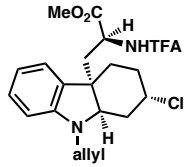
Data file /data/indy/jni/vnmrsys/data/JN-4-239b-rxst-Mar6/PROTON01.fid

Plot date 2015-06-09

Agilent Technologies

JN-4-239b-rxst-Mar6-carbon

Sample Name	JN-4-239b-rxst-Mar6-carbon	Pulse sequence	CARBON	Temperature	25	Study owner	jni
Date collected	2014-03-06	Solvent	cdcl3	Spectrometer	-vnmrs400	Operator	autouser



8a

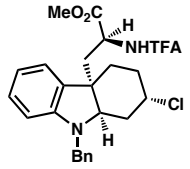
Data file /data/indy/jni/vnmrsys/data/JN-4-239b-rxst-Mar6-carbon/CARBON01.fid

Plot date 2015-06-09

Agilent Technologies

JN-4-275b-col2

Sample Name	**JN-4-275b-col2**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**jni**
Date collected	**2014-09-21**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



8c

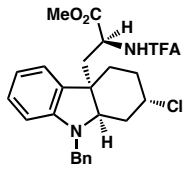
Data file /data/indy/jni/vnmrsys/data/JN-4-275b-col2/PROTON01.fid

Plot date 2015-06-10

Agilent Technologies

JN-4-275b-col2

Sample Name	**JN-4-275b-col2**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**jni**
Date collected	**2014-09-21**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



8c

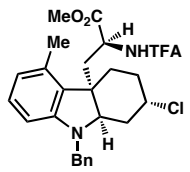
Data file /data/indy/jni/vnmrsys/data/JN-4-275b-col2/CARBON01.fid

Plot date 2015-06-10

Agilent Technologies

JN-5-055a-col2

Sample Name	JN-5-055a-col2	Pulse sequence	PROTON	Temperature	50	Study owner	jni
Date collected	2014-01-22	Solvent	cdcl3	Spectrometer	-vnmrs400	Operator	autouser



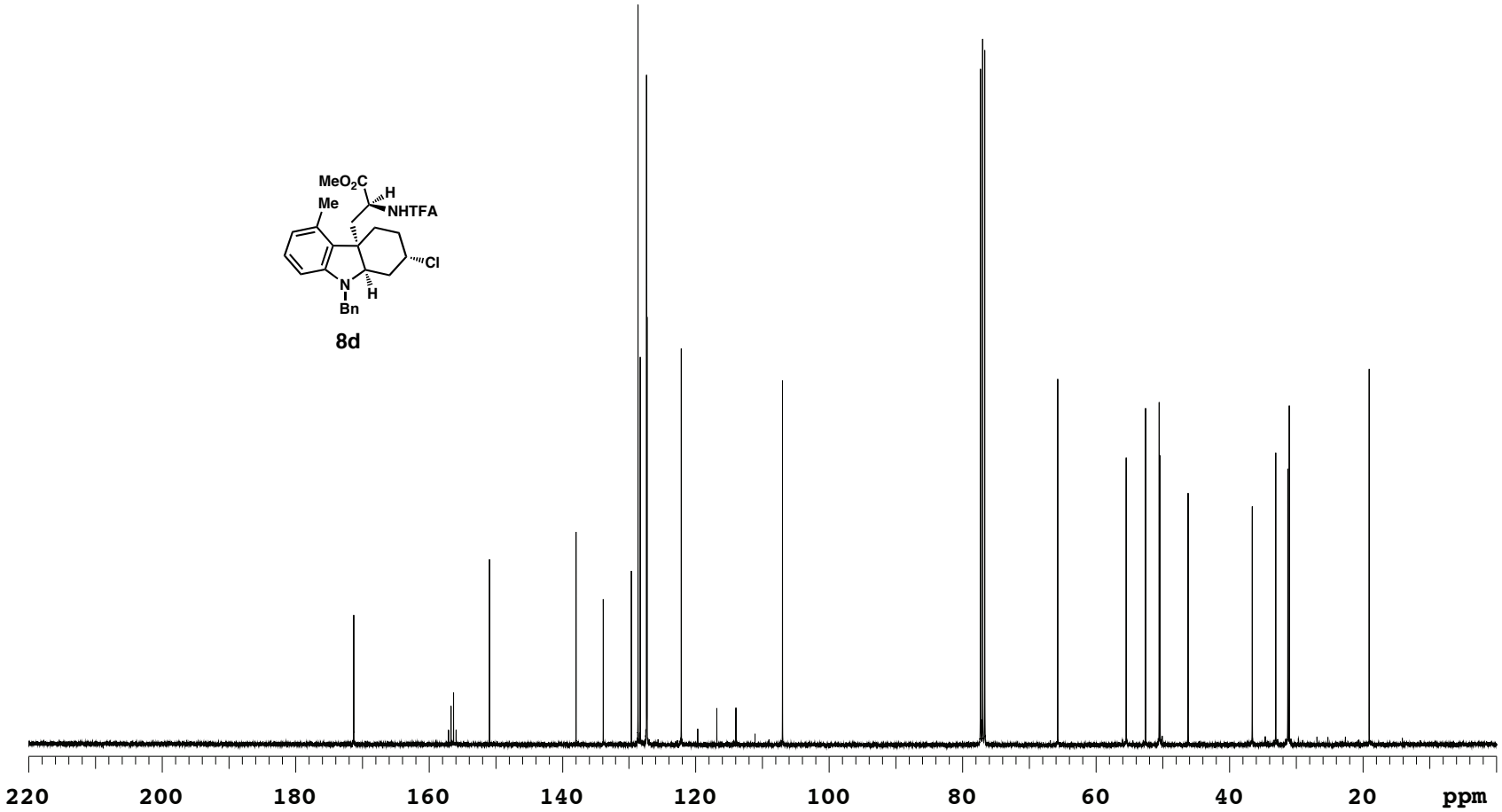
8d

Data file /data/siena/jni/vnmrsys/data/JN-5-055a-col2/PROTON01.fid

Plot date 2015-06-09

Agilent Technologies

Sample Name	JN-5-055a-col2	Pulse sequence	CARBON	Temperature	50	Study owner	jni
Date collected	2014-01-22	Solvent	cdcl3	Spectrometer	-vnmrs400	Operator	autouser



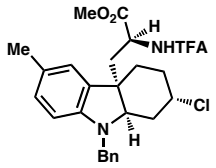
8d

Data file /data/siena/jni/vnmrsys/data/JN-5-055a-col2/CARBON01.fid

Plot date 2015-06-09

Agilent Technologies

Sample Name	**JN-5-053b-col2**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**jni**
Date collected	**2014-01-24**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



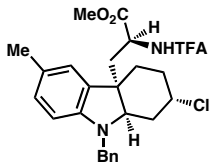
8e

Data file /data/indy/jni/vnmrsys/data/JN-5-053b-col2/PROTON01.fid

Plot date 2015-06-09

Agilent Technologies

Sample Name	**JN-5-053b-col2**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**jni**
Date collected	**2014-01-24**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



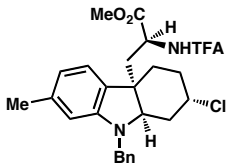
8e

Data file /data/indy/jni/vnmrsys/data/JN-5-053b-col2/CARBON01.fid

Plot date 2015-06-09

Agilent Technologies

Sample Name	JN-5-049a-col2	Pulse sequence	PROTON	Temperature	25	Study owner	jni
Date collected	2014-01-13	Solvent	cdcl3	Spectrometer	-vnmrs400	Operator	autouser



8f

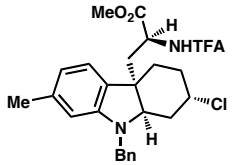
Data file /data/indy/jni/vnmrsys/data/JN-5-049a-col2/PROTON01.fid

Plot date 2015-06-09

Agilent Technologies

JN-5-049a-col2

Sample Name	**JN-5-049a-col2**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**jni**
Date collected	**2014-01-13**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



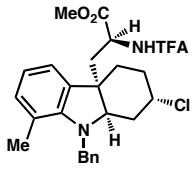
8f

Data file /data/indy/jni/vnmrsys/data/JN-5-049a-col2/CARBON01.fid

Plot date 2015-06-09

Agilent Technologies

Sample Name	JN-5-055b-col2	Pulse sequence	PROTON	Temperature	50	Study owner	jni
Date collected	2014-01-22	Solvent	cdcl3	Spectrometer	-vnmrs400	Operator	autouser



8g

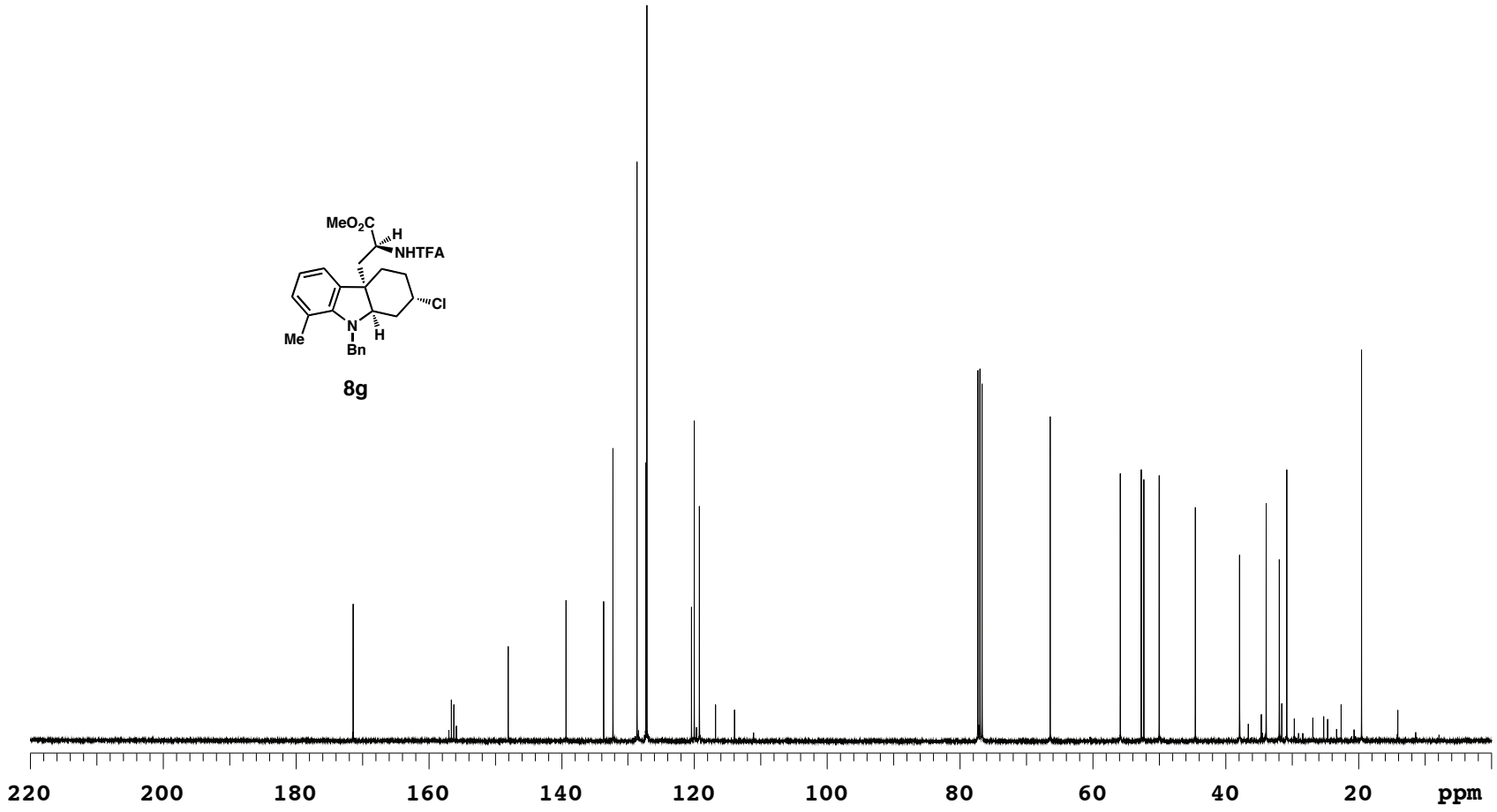
Data file /data/siena/jni/vnmrsys/data/JN-5-055b-col2/PROTON01.fid

Plot date 2015-06-09

Agilent Technologies

JN-5-055b-col2

Sample Name	**JN-5-055b-col2**	Pulse sequence	**CARBON**	Temperature	**50**	Study owner	**jni**
Date collected	**2014-01-22**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



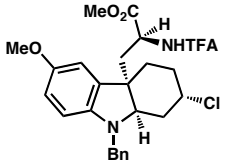
Data file /data/siena/jni/vnmrsys/data/JN-5-055b-col2/CARBON01.fid

Plot date 2015-06-09

Agilent Technologies

JN-5-049b-col2

Sample Name	**JN-5-049b-col2**	Pulse sequence	**PROTON**	Temperature	**65**	Study owner	**jni**
Date collected	**2014-01-15**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



8h

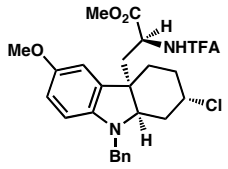
Data file /data/siena/jni/vnmrsys/data/JN-5-049b-col2/PROTON01.fid

Plot date 2015-06-09

Agilent Technologies

JN-5-049b-col2

Sample Name	JN-5-049b-col2	Pulse sequence	CARBON	Temperature	65	Study owner	jni
Date collected	2014-01-15	Solvent	cdcl3	Spectrometer	-vnmrs400	Operator	autouser



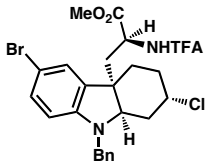
8h

Data file /data/siena/jni/vnmrsys/data/JN-5-049b-col2/CARBON01.fid

Plot date 2015-06-09

Agilent Technologies

Sample Name	JN-5-053a-col2	Pulse sequence	PROTON	Temperature	25	Study owner	jni
Date collected	2014-01-30	Solvent	cdcl3	Spectrometer	-vnmrs400	Operator	autouser



8i

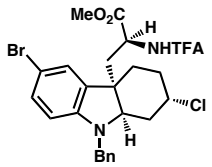
Data file /data/indy/jni/vnmrsys/data/JN-5-053a-col2/PROTON01.fid

Plot date 2015-06-09

Agilent Technologies

JN-5-053a-col2

Sample Name	**JN-5-053a-col2**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**jni**
Date collected	**2014-01-30**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**

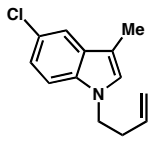


8i

Data file /data/indy/jni/vnmrsys/data/JN-5-053a-col2/CARBON01.fid

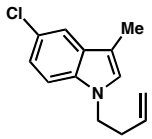
Plot date 2015-06-09

```
Current Data Parameters
NAME       BED2-28char2
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20150217
Time              19.45
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG               197.44
DW               62.400 usec
DE               10.00 usec
TE               298.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                11.70 usec
PLW1          7.00000000 W

F2 - Processing parameters
SI                65536
SF           400.1300400 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



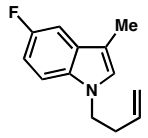
9b

```
Current Data Parameters
NAME       BED2-28char2
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_           20150217
Time              20.01
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  896
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                55.48
DW               20.800 usec
DE                82.37 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   7

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        36.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          7.00000000 W
PLW12         0.12033000 W
PLW13         0.09746800 W

F2 - Processing parameters
SI                32768
SF          100.6127685 MHz
WDW                  no
SSB       0
LB        0 Hz
GB        0
PC                 1.40
```



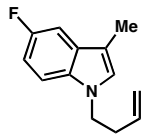
9b

```
Current Data Parameters
NAME         BED2-53char
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150222
Time               20.32
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                197.44
DW                62.400 usec
DE                10.00 usec
TE                298.2 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 11.70 usec
PLW1          7.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300098 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



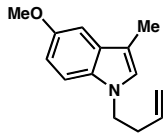
9c

```
Current Data Parameters
NAME         BED2-53char
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150222
Time              20.40
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  896
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                 64.2
DW                20.800 usec
DE                 82.37 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   7

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        36.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          7.00000000 W
PLW12         0.12033000 W
PLW13         0.09746800 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

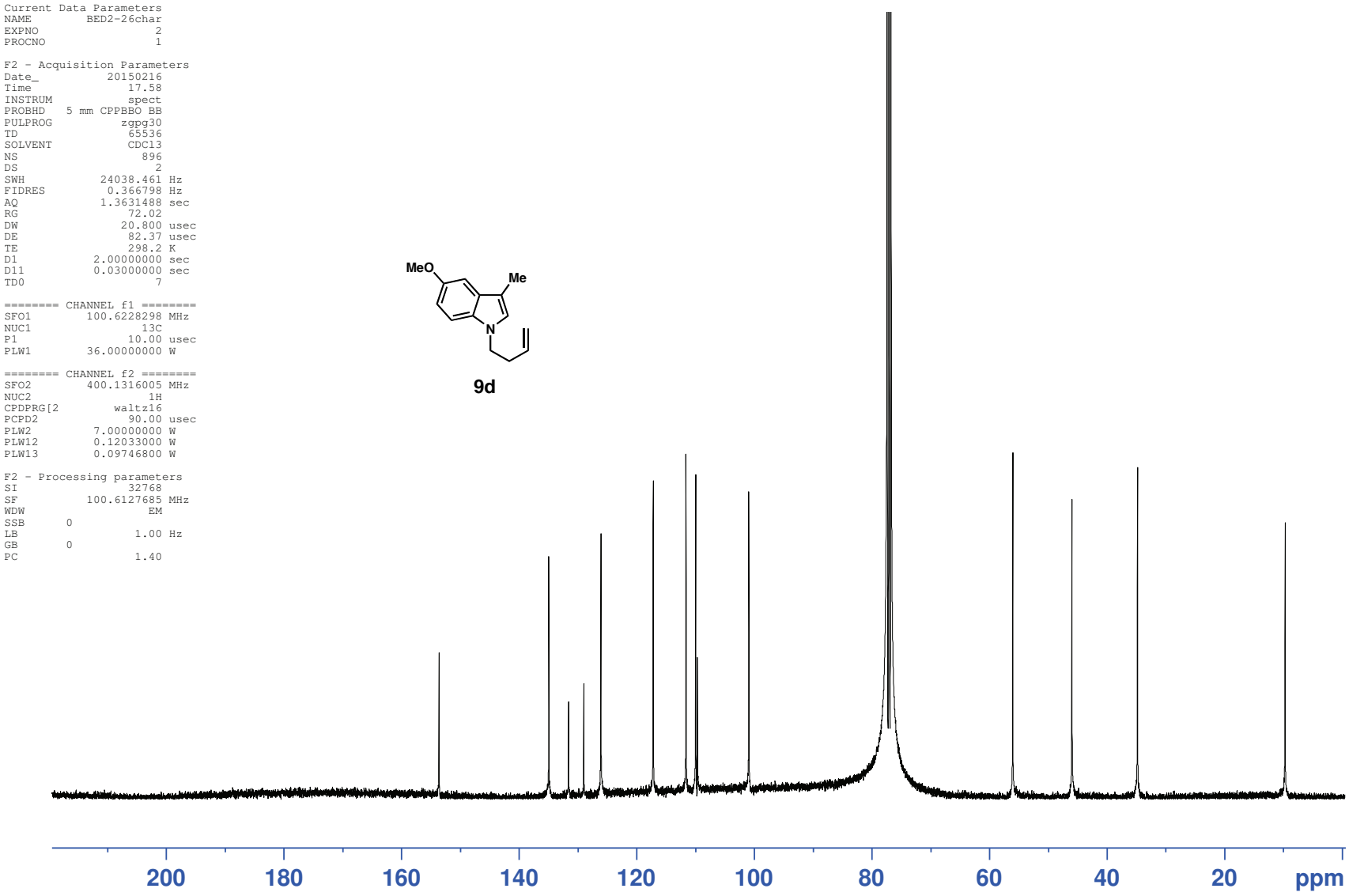


9c

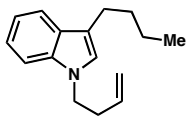
```
Current Data Parameters
NAME         BED2-26char
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150216
Time               17.50
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                156.15
DW                62.400 usec
DE                10.00 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 11.70 usec
PLW1          7.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300097 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



9d

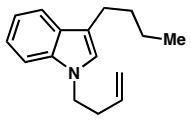

Current Data Parameters
NAME BED2-26char
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150216
Time 17.58
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 896
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 72.02
DW 20.800 usec
DE 82.37 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 7
======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 36.00000000 W
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 7.00000000 W
PLW12 0.12033000 W
PLW13 0.09746800 W
F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
MeO
Me
N
9d
200
180
160
140
120
100
80
60
40
20
ppm

```
Current Data Parameters
NAME       BED1-268char
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20150224
Time              16.07
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                30.29
DW               62.400 usec
DE               10.00 usec
TE               298.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 11.70 usec
PLW1          7.00000000 W

F2 - Processing parameters
SI                65536
SF           400.1300096 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



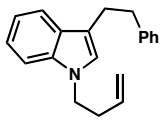
9e

```
Current Data Parameters
NAME       BED1-268char
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20150224
Time              16.15
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                43.71
DW               20.800 usec
DE                82.37 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   7

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        36.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         7.00000000 W
PLW12        0.12033000 W
PLW13        0.09746800 W

F2 - Processing parameters
SI                32768
SF           100.6127685 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



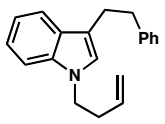
9e

```
Current Data Parameters
NAME       BED1-278char
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20150506
Time              19.13
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG               197.44
DW               62.400 usec
DE                10.00 usec
TE                298.1 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                11.70 usec
PLW1         7.00000000 W

F2 - Processing parameters
SI                65536
SF          400.1300098 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



9f

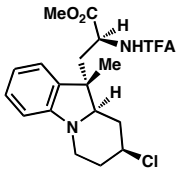
```
Current Data Parameters
NAME       BED1-287char
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_           20150328
Time              18.42
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  640
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                55.48
DW               20.800 usec
DE                82.37 usec
TE                298.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   7

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        36.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         7.00000000 W
PLW12        0.12033000 W
PLW13        0.09746800 W

F2 - Processing parameters
SI                32768
SF          100.6127685 MHz
WDW                  no
SSB       0
LB        0 Hz
GB        0
PC                1.40
```



9f

Agilent Technologies

Sample Name	JN-5-061b-col2	Pulse sequence	PROTON	Temperature	25	Study owner	jni
Date collected	2014-03-05	Solvent	cdcl3	Spectrometer	-vnmrs400	Operator	autouser



10a

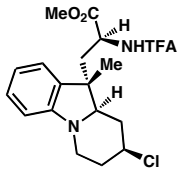
Data file /data/indy/jni/vnmrsys/data/JN-5-061b-col2/PROTON01.fid

Plot date 2015-06-09

Agilent Technologies

JN-5-061b-col2

Sample Name	JN-5-061b-col2	Pulse sequence	CARBON	Temperature	25	Study owner	jni
Date collected	2014-03-05	Solvent	cdcl3	Spectrometer	-vnmrs400	Operator	autouser



10a

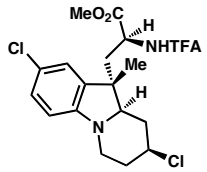
Data file /data/indy/jni/vnmrsys/data/JN-5-061b-col2/CARBON01.fid

Plot date 2015-06-09

Agilent Technologies

BED2-70char

Sample Name	**BED2-70char**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-05-26**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



10b

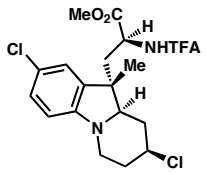
Data file /indy/bdaniels/vnmrsys/data/BED2-70char/PROTON01.fid

Plot date 2015-05-31

Agilent Technologies

BED2-70char

Sample Name	**BED2-70char**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-05-26**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**

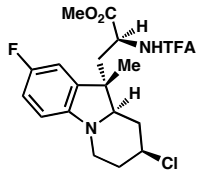


10b

Data file /indy/bdaniels/vnmrsys/data/BED2-70char/CARBON01.fid

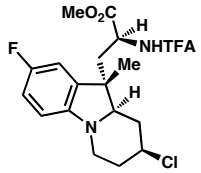
Plot date 2015-05-31

```
Current Data Parameters
NAME         BED2-71char
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150521
Time              21.24
INSTRUM           spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                156.15
DW                62.400 usec
DE                10.00 usec
TE                294.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 11.70 usec
PLW1          7.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300098 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



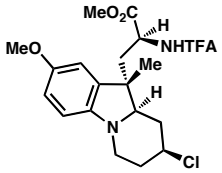
10c

```
Current Data Parameters
NAME         BED2-71char
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150521
Time              23.51
INSTRUM           spect
PROBHD    5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2560
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                72.02
DW               20.800 usec
DE                82.37 usec
TE                294.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1        36.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          7.00000000 W
PLW12         0.12033000 W
PLW13         0.09746800 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.40
```



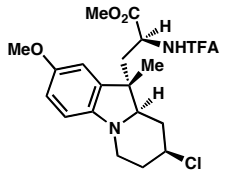
10c

```
Current Data Parameters
NAME         BED2-37char
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150524
Time               22.34
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                156.15
DW                62.400 usec
DE                10.00 usec
TE                294.9 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 11.70 usec
PLW1          7.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300098 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



10d

```
Current Data Parameters
NAME         BED2-37char
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_           20150525
Time              1.01
INSTRUM           spect
PROBHD    5 mm CPPBBO BB
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2560
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG                50.33
DW               20.800 usec
DE                82.37 usec
TE                294.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1        36.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         7.00000000 W
PLW12        0.12033000 W
PLW13        0.09746800 W

F2 - Processing parameters
SI                32768
SF          100.6127721 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

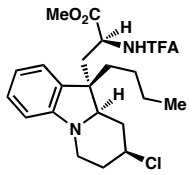


10d

Agilent Technologies

BED1-272majordiastereomer10-2-2014

Sample Name	**BED1-272majordiastereomer10-2-2014**	Pulse Sequence	**PROTON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2014-10-02**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



10e

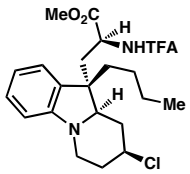
Data file /indy/bdaniels/vnmrsys/data/BED1-272majordiastereomer10-2-2014/PROTON01.fid

Plot date 2015-05-31

Agilent Technologies

BED1-272longercarbon

Sample Name	**BED1-272longercarbon**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2014-10-03**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



10e

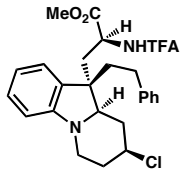
Data file /indy/bdaniels/vnmrsys/data/BED1-272longercarbon/CARBON01.fid

Plot date 2015-05-31

Agilent Technologies

BED2-77charcheckpostfailedcarbon

Sample Name	**BED2-77charcheckpostfailedcarbon**	Pulse Sequence	**PROTON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-05-12**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



10f

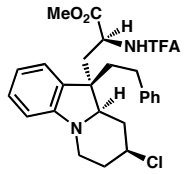
Data file /indy/bdaniels/vnmrsys/data/BED2-77charcheckpostfailedcarbon/PROTON01.fid

Plot date 2015-05-31

Agilent Technologies

BED2-77char

Sample Name	**BED2-77char**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**bdaniels**
Date collected	**2015-05-12**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



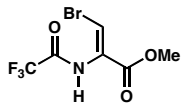
10f

Data file /indy/bdaniels/vnmrsys/data/BED2-77char/CARBON01.fid

Plot date 2015-05-31

Agilent Technologies

Sample Name	**JN-5-085-col**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**jni**
Date collected	**2014-04-14**	Solvent	**acetone**	Spectrometer	**-vnmrs400**	Operator	**autouser**



S7

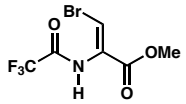
Data file /data/indy/jni/vnmrsys/data/JN-5-085-col/PROTON01.fid

Plot date 2015-06-09

Agilent Technologies

JN-5-085-col2

Sample Name	**JN-5-085-col2**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**jni**
Date collected	**2014-04-14**	Solvent	**acetone**	Spectrometer	**-vnmrs400**	Operator	**autouser**



S7

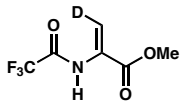
Data file /data/indy/jni/vnmrsys/data/JN-5-085-col2/CARBON01.fid

Plot date 2015-06-09

Agilent Technologies

JN-5-117

Sample Name	**JN-5-117**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**jni**
Date collected	**2014-08-05**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



2a-*d*

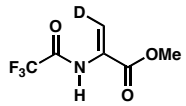
Data file /data/indy/jni/vnmrsys/data/JN-5-117/PROTON01.fid

Plot date 2015-06-09

Agilent Technologies

JN-5-117

Sample Name	**JN-5-117**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**jni**
Date collected	**2014-08-05**	Solvent	**cdcl3**	Spectrometer	**-vnmrs400**	Operator	**autouser**



2a-*d*

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Plot date 2015-06-09