A Mild and General Larock Indolization Protocol for the Preparation of Unnatural Tryptophans

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Supporting Information:

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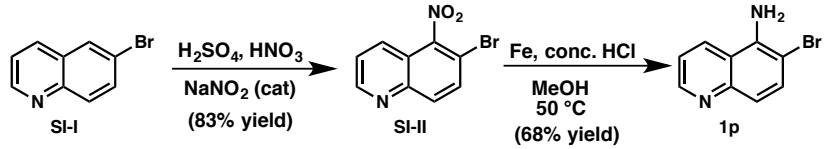
I. General Procedures. Unless otherwise stated, reactions were performed under a nitrogen atmosphere using freshly dried solvents. Tetrahydrofuran (THF), methylene chloride (CH_2Cl_2), acetonitrile (MeCN), dimethylformamide (DMF), and toluene (PhMe) were dried by passing through activated alumina columns. 1,4-Dioxane was dried by passing through activated alumina columns or purchased from Sigma-Aldrich (>99.8%, anhydrous). Triethylamine (Et_3N), diisopropylamine (*i*-Pr_2NH), diisopropylethylamine (*i*-Pr_2NEt), and dicyclohexylmethylamine (Cy_2NMe) were distilled over calcium hydride prior to use. Unless otherwise stated, chemicals and reagents were used as received. All 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, or $KMnO_4$ staining. Flash column chromatography was performed using silica gel (particle size 0.032–0.063) purchased from Silicyle. Optical rotations were measured on a Jasco P-2000 polarimeter using a 100 mm path-length cell at 589 nm. ^1H and ^{13}C NMR spectra were recorded on a Varian 400 MR (at 400 MHz and 101 MHz, respectively), a Varian Inova 500 (at 500 MHz and 126 MHz, respectively), or a Varian Inova 600 (at 600 MHz and 150 MHz, respectively), and are reported relative to internal $CHCl_3$ (^1H, δ = 7.26), MeCN (^1H, δ = 1.94), CH_2Cl_2 (^1H, δ = 5.32) or DMSO (^1H, δ = 2.50), and $CDCl_3$ (^{13}C, δ = 77.0), MeCN (^{13}C, δ = 118.26), CD_2Cl_2 (^{13}C, δ = 54.0) or DMSO (^{13}C, δ = 40.0). Data for ^1H 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}). Preparatory HPLC was performed with an Agilent 1200 Series HPLC utilizing an Agilent XDB-C18 5µm column (30 x 250 mm). Analytical SFC was performed with a Mettler SFC supercritical CO_2 analytical chromatography system with Chiralcel AD-H column (4.6 mm x 25 cm). HRMS were acquired using 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.

II. Preparation of Haloaniline Substrates

Bromoaniline 1p



6-Bromoquinoline (SI-I) was purchased from Combi-Blocks and nitrated using a known procedure[1] to give **SI-II**. **SI-II** (500 mg, 2.0 mmol, 1.0 equiv) was dissolved in MeOH (6 mL). Fe powder (331 mg, 5.9 mmol, 3.0 equiv) and concentrated HCl (2 mL) were added and the reaction was heated to 50 °C for 1 h. Upon cooling, the reaction was basified with NH_4OH to pH 9, filtered through celite, and extracted with EtOAc (2 x 10 mL). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude material was purified by chromatography on silica gel (40% acetone, 60% hexanes) to provide **1p** as a light yellow, amorphous solid (300 mg, 1.3 mmol, 68% yield).

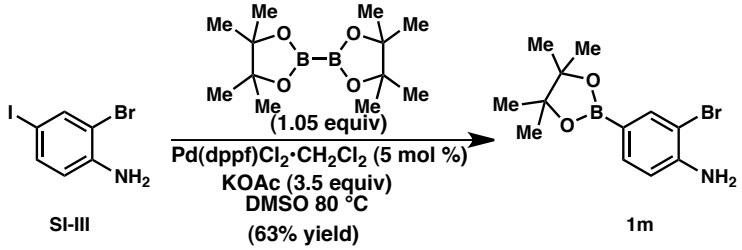
^{1}H NMR: (500 MHz, $CDCl_3$) δ 8.90 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.16 (ddd, *J* = 8.6, 1.5, 0.9 Hz, 1H), 7.72 (d, *J* = 9.0 Hz, 1H), 7.45 (dd, *J* = 9.0, 0.7 Hz, 1H), 7.39 (dd, *J* = 8.6, 4.2 Hz, 1H), 4.68 (s, 2H);

^{13}C NMR: (126 MHz, $CDCl_3$) δ 150.3, 148.1, 139.6, 133.3, 129.4, 120.7, 120.2, 118.7, 104.3;

FTIR: (NaCl, thin film): cm^{-1}; 3423, 3297, 3162, 1635, 1581, 1569, 1457, 1398, 1357, 1323;

HRMS (MM) calc'd $[M+H]^+$ 222.9865, found 222.9862.

Bromoaniline 1m



In a glovebox, an oven-dried 2-dram vial was charged with 2-bromo-4-iodoaniline **SI-III** (500 mg, 1.68 mmol, 1.00 equiv), $Pd(dppf)Cl_2•CH_2Cl_2$ (69 mg, 0.08 mmol, 0.05 equiv), bis(pinacolato)diboron (448 mg, 1.77 mmol, 1.05 equiv), KOAc (557 mg, 5.9 mmol, 3.5 equiv), and DMSO (5 mL). The vial was sealed, removed from the glovebox and heated to 80 °C. After 24 h, the reaction was cooled, filtered through a plug of Celite, and the plug was rinsed with EtOAc. The resulting filtrate was then washed with water (3 x 20 mL), dried over anhydrous Na_2SO_4, filtered, and

[1] Bounaud, P–Y.; Smith, C. R.; Jefferson, E. A. (SGX Pharmaceuticals Inc). Bicyclic Triazoles as Protein Kinase Modulators. US2007/081841, July 24, 2008.

concentrated *in vacuo*. The crude reaction mixture was purified by chromatography on silica gel (10% EtOAc, 90% hexanes) to give white, amorphous solid **1m** (315 mg, 1.06 mmol, 63% yield).

^{1}H NMR: (500 MHz, $CDCl_3$) δ 7.86 (d, *J* = 1.3 Hz, 1H), 7.52 (dd, *J* = 7.9, 1.4 Hz, 1H), 6.72 (d, *J* = 7.9 Hz, 1H), 1.32 (s, 12H);

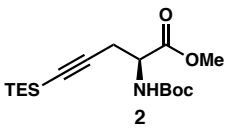
^{13}C NMR: (126 MHz, $CDCl_3$) δ 146.6, 139.2, 135.0, 114.8, 108.8, 83.6, 24.8 (Boron-bearing *ipso*-carbon was not observed);

FTIR: (NaCl, thin film): cm^{-1}; 3477, 3368, 2977, 2930, 1616, 1594, 1385, 1372, 1319, 1143, 1098;

HRMS: (MM) calc'd $[M+H]^+$ 297.0645, found 297.0637.

III. Preparation of Alkyne Substrates

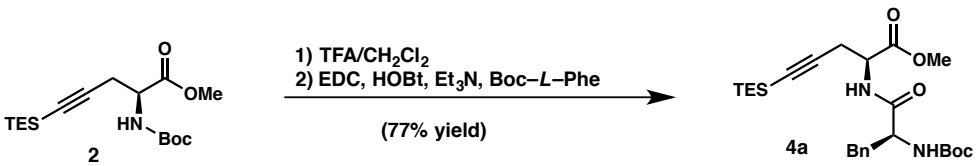
Alkyne 2



2

Alkyne **2** was prepared on decagram scale according to the procedure reported by Baran and coworkers[2] with the following modification: Zn(0)-dust (Aldrich, <10 μm, >98%) was freshly activated by the following procedure: Zinc powder (50 g) was added to a rapidly stirring solution of 2% aqueous HCl and stirred for ~3 minutes. The aqueous solution was decanted and the zinc was washed in rapid succession with EtOH (150 mL), acetone (2 x 150 mL), and anhydrous ether (300 mL). The zinc was rapidly filtered and collected into a 500-mL round bottomed flask that was evacuated to <1 mmHg and dried at 80 °C for a minimum of two hours. The freshly activated zinc is quickly weighed out from this flask prior to the reaction.

Alkyne 4a



To a solution of alkyne **2** (500 mg, 1.5 mmol, 1.00 equiv) in CH_2Cl_2 (15 mL) at 0 °C was added TFA (2.0 mL). The mixture was warmed to room temperature and stirred for 3 hours, after which PhMe (30 mL) was added and the reaction was concentrated. The resultant oil was dissolved in THF (10 mL) and cooled to 0 °C under a positive pressure of N_2. In a separate flask, (*L*)-Boc-Phe-OH (466 mg, 1.8 mmol, 1.2 equiv) was dissolved in THF (24 mL) and cooled to 0 °C. EDC (337 mg, 1.8 mmol, 1.2 equiv), anhydrous HOBt (277 mg, 2.0 mmol, 1.4 equiv), and Et_3N (610 μL, 4.4 mmol, 3.0 equiv) were added sequentially. After stirring for 5 minutes, the alkyne was transferred via cannula. The reaction was warmed to room temperature and stirred for 12 h. The heterogeneous reaction was concentrated and purified by chromatography on silica gel (20% EtOAc, 80% hexanes) to provide alkyne **4a** as a colorless oil (552 mg, 1.13 mmol, 77% yield).

^{1}H NMR: (500 MHz, $CDCl_3$, Major Rotamer) δ 7.28 – 7.22 (m, 2H), 7.19 (dd, *J* = 7.1, 7.1 Hz, 3H), 6.77 (d, *J* = 6.3 Hz, 1H), 5.16 (d, *J* = 5.9 Hz, 1H), 4.67 (d, *J* = 6.5 Hz, 1H), 4.44 (d, *J* = 6.1 Hz, 1H), 3.70 (s, 3H), 3.11 (dd, *J* = 13.9, 6.3 Hz, 1H), 2.98 (dd, *J* = 12.8, 6.6 Hz, 1H), 2.73 (dd, *J* = 17.0, 4.0 Hz, 1H), 2.57 (dd, *J* = 17.1, 5.3 Hz, 1H), 1.35 (s, 9H), 0.98 – 0.87 (m, 9H), 0.57 – 0.46 (m, 6H);

^{13}C NMR: (126 MHz, $CDCl_3$) δ 170.8, 170.4, 155.2, 136.4, 129.2, 128.4, 126.7, 101.2, 85.5, 79.8, 66.9, 55.3, 52.4, 50.8, 38.4, 28.0, 23.5, 7.2, 4.1;

FTIR: (NaCl, thin film): cm^{-1}; 3419, 3335, 2963, 2868, 2179, 1743, 1661, 1518, 1451, 1365;

$[\alpha]_D^{25}$ = +52.7 (*c* = 5.4, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 489.2779, found 489.2793.

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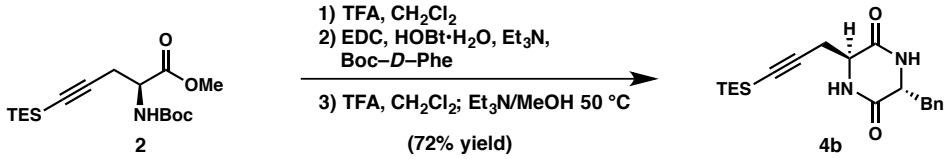
[2] Newhouse, T.; Lewis, C. A.; Baran, P. S. *J. Am. Chem. Soc.* **2009**, *131*, 6360.

Alkyne 4b



Alkyne **2** (1.02 g, 3.0 mmol, 1.00 equiv) was dissolved in a 5:1 mixture of CH_2Cl_2:TFA (20 mL). After one hour, the reaction was concentrated *in vacuo* and dried on high vacuum, then redissolved in 34 mL anhydrous CH_2Cl_2. The solution was cooled to 0 °C under N_2 and EDC•HCl (0.862 g, 4.5 mmol, 1.50 equiv) and HOBt•H_2O (0.680 g, 4.5 mmol, 1.50 equiv) were added sequentially. Et_3N (1.88 mL, 13.5 mmol, 4.5 equiv) was then added dropwise by syringe. The mixture was then stirred for 5 minutes, and Boc–*D*–phenylalanine (1.59 g, 6.0 mmol, 2.0 equiv) was added in a single portion. The reaction was slowly warmed to 23 °C over 2 hours and stirring continued for 20 hours. The reaction was then quenched with 1 N HCl (500 mL) and extracted with EtOAc (3 x 250 mL), then the combined organics washed with saturated aqueous $NaHCO_3$ (500 mL), and aqueous layer back-extracted with EtOAc (200 mL). The combined organic layers were then dried over anhydrous Na_2SO_4, filtered, and concentrated *in vacuo* to afford crude dipeptide as a viscous oil.

The residue was then dissolved in CH_2Cl_2 (50 mL) and trifluoroacetic acid (15 mL) was added dropwise by addition funnel at room temperature over 10 minutes. Stirring was continued for 20 minutes, then the solution was diluted with toluene (100 mL) and the mixture was concentrated *in vacuo* to afford a thick oil. The residue was then redissolved in MeOH (35 mL) and the mixture was cooled to 0 °C. Et_3N (27 mL) was then added dropwise to the stirring solution over 10 minutes by addition funnel. Upon completion of the addition, the cooling bath was removed and the reaction was heated to 50 °C over 16 h. The mixture was cooled to 0 °C to yield a milky solution, which was filtered and washed with cold methanol to provide alkyne **4b** as a colorless solid (771 mg, 72% yield)

^{1}H NMR: (500 MHz, DMSO-d_6) δ 8.35 (d, *J* = 1.6 Hz, 1H), 8.02 (d, *J* = 1.0 Hz, 1H), 7.29 – 7.19 (m, 3H), 7.18 – 7.12 (m, 2H), 4.15 (s, 1H), 3.19 – 3.11 (m, 2H), 2.88 (dd, *J* = 13.5, 4.9 Hz, 1H), 2.63 (dd, *J* = 16.9, 3.2 Hz, 1H), 2.40 (dd, *J* = 17.0, 4.7 Hz, 1H), 0.91 (t, *J* = 7.9 Hz, 9H), 0.50 (q, *J* = 7.9 Hz, 6H);

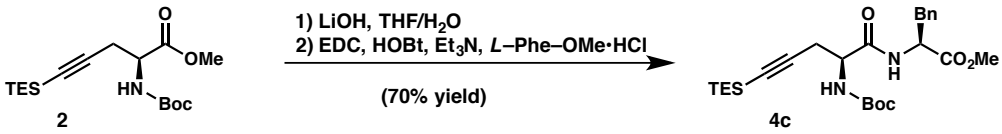
^{13}C NMR: (126 MHz, DMSO-d_6) δ 166.6, 166.0, 135.8, 130.1, 128.0, 126.7, 104.0, 83.9, 55.4, 52.6, 38.4, 24.8, 7.3, 3.9;

FTIR: (NaCl, thin film): cm^{-1}; 2916, 2350, 1732, 1651, 1557, 1455;

$[\alpha]_D^{25}$ = –19.4 (*c* = 0.28, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 357.1993, found 357.1992.

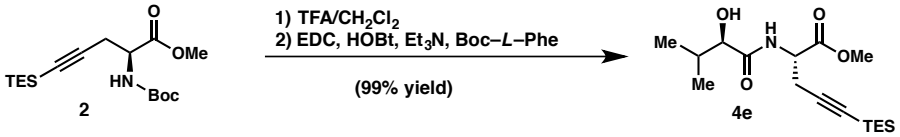
Alkyne 4c



To a solution of alkyne **2** (550 mg, 1.5 mmol, 1.00 equiv) in THF/H_2O (4 mL/2 mL) at 0 °C under a positive pressure of N_2 was added aqueous LiOH (1 M, 1.9 mL, 1.2 equiv). After 1 hour, the reaction was quenched by slow addition of 1 M HCl (3 mL) and Et_2O (6 mL). The layers were separated and the aqueous was extracted with Et_2O (3 x 10 mL). The organics were combined, washed with brine, dried over $MgSO_4$, filtered, and concentrated *in vacuo* to afford a colorless oil. The oil was dissolved in 24 mL THF and cooled to 0 °C under a positive pressure of N_2. EDC (337 mg, 1.8 mmol, 1.2 equiv), anhydrous HOBt (277 mg, 2.0 mmol, 1.4 equiv) and Et_3N (610 µL, 4.4 mmol, 3.0 equiv) were added sequentially. After 5 minutes of stirring, a solution of (*L*)-Phe-OMe•HCl (347 mg, 1.6 mmol, 1.1 equiv) in THF (10 mL) was added via cannula. The reaction was warmed to room temperature and stirred for 12 h. The heterogeneous solution was concentrated and purified by chromatography on silica gel (20% EtOAc, 80% hexanes) to give white, amorphous solid **4c** (500 mg, 1.02 mmol, 70% yield)

^{1}H NMR: (500 MHz, $CDCl_3$, Major Rotamer) δ 7.30 – 7.20 (m, 3H), 7.10 – 7.07 (m, 2H), 6.84 (d, *J* = 4.9 Hz, 1H), 5.25 (s, 1H), 4.81 (ddd, *J* = 7.5, 6.0, 6.0 Hz, 1H), 4.22 (d, *J* = 4.9 Hz, 1H), 3.67 (s, 3H), 3.13 – 3.05 (m, 2H), 2.74 (dd, *J* = 17.1, 6.1 Hz, 1H), 2.65 (dd, *J* = 17.1, 6.5 Hz, 1H), 1.42 (s, 9H), 0.95 (t, *J* = 7.9 Hz, 9H), 0.55 (q, *J* = 7.9 Hz, 6H);
^{13}C NMR: (126 MHz, $CDCl_3$) δ 171.3, 169.9, 155.3, 135.7, 129.1, 128.4, 127.0, 102.5, 85.5, 80.2, 53.4, 53.0, 52.2, 38.0, 28.1, 23.3, 7.4, 4.2;
FTIR: (NaCl, thin film): cm^{-1}; 3319, 2954, 2935, 2874, 2177, 1746, 1689, 1660, 1527, 1498, 1456, 1367, 1274, 1251, 1172, 1048, 1017;
$[\alpha]_D^{25}$ = +39.2 (*c* = 4.29, $CHCl_3$);
HRMS: (MM) calc'd $[M–C_4H_9]^+$ 433.2153, found 433.2138.

Alkyne 4e



To a solution of alkyne **2** (1.00 g, 2.9 mmol, 1.00 equiv) in CH_2Cl_2 (30 mL) at 0 °C was added TFA (4 mL). The mixture was warmed to room temperature and stirred for 3 hours, after which PhMe (100 mL) was added and the reaction concentrated. The resultant oil was dissolved in THF (10 mL) and cooled to 0 °C under a positive pressure of N_2. In a separate flask, (*R*)-2-hydroxy-3-methylbutanoic acid (346 mg, 2.9 mmol, 1.0 equiv) was dissolved in THF (100 mL) and cooled to 0 °C. EDC (674 mg, 3.5 mmol, 1.2 equiv), anhydrous HOBt (554 mg, 4.1 mmol, 1.4 equiv) and ^{i}Pr$_2$NEt (1.5

mL, 8.6 mmol, 3.0 equiv) were added sequentially. After stirring for 5 minutes, the alkyne was added to this solution dropwise via cannula. The reaction was then warmed to room temperature and stirred for 12 h. The heterogeneous reaction was concentrated and purified by chromatography on silica gel (100% EtOAc) to provide alkyne **4e** as a colorless oil (995 mg, 2.9 mmol, 99% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 7.43 (d, *J* = 7.6 Hz, 1H), 4.59 (dt, *J* = 8.2, 5.3 Hz, 1H), 3.89 (dd, *J* = 5.5, 3.1 Hz, 1H), 3.71 (d, *J* = 5.6 Hz, 1H), 3.69 (s, 3H), 2.80 (dd, *J* = 17.2, 5.5 Hz, 1H), 2.73 (dd, *J* = 17.2, 5.2 Hz, 1H), 2.07 (heptd, *J* = 6.9, 3.1 Hz, 1H), 1.01 – 0.93 (m, 12H), 0.82 (d, *J* = 6.9 Hz, 3H), 0.61 – 0.52 (m, 6H);

^{13}C NMR: (126 MHz, CD_3CN) δ 174.2, 171.7, 103.6, 85.8, 76.4, 53.0, 51.4, 32.7, 23.9, 19.5, 15.9, 7.8, 5.0;

FTIR: (NaCl, thin film): cm^{-1}; 3385, 2952, 2863, 2176, 1744, 1653, 1507;

$[\alpha]_D^{25}$ = +89.4 (*c* = 3.40, $CHCl_3$);

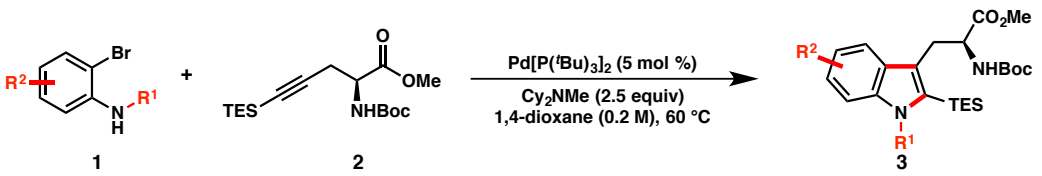
HRMS: (MM) calc'd $[M+H]^+$ 342.2095, found 342.2087.

IV. Optimization of Reaction Parameters

Optimization Procedure – In a glovebox, an oven-dried 1-dram vial was charged with 2-bromoaniline (17.2 mg, 0.1 mmol, 1.0 equiv), alkyne **2** (68.3 mg, 0.2 mmol, 2.0 equiv), base (2.5 equiv), Pd-catalyst (0.05 equiv), and appropriate solvent (0.5 mL). The vial was sealed and heated to the reported temperature while the reaction progress was monitored by UHPLC. Upon consumption of the starting material (2 – 36 h), the vial was cooled and the crude reaction mixture was filtered through a silica plug, thoroughly washed with EtOAc, and concentrated *in vacuo* to provide a crude oil.

The crude residue was dissolved in a standard solution of 2,3,5,6-tetrachloronitrobenzene in DMSO-d_6, and the yield of **3a** was determined by ^{1}H NMR by integration relative to the internal standard. In entry 11 of Table 1, $Pd_2(dba)_3$ and P^{t}Bu$_3$ were prestirred for 1 h before being added to a vial containing the other reagents.

V. Substrate Scope – Characterization Data



Glovebox-Free Procedure: For ease of reaction monitoring, the substrate scope studies were conducted in a nitrogen filled glovebox. However, comparable yields are readily obtained outside of the glovebox utilizing the following protocol – To an oven-dried, 2-dram vial was charged bromoaniline (0.30 mmol, 1.0 equiv), alkyne (0.60 mmol, 2.0 equiv), and $Pd[P(P^tBu)_3]_2$ (0.015 mmol, 0.05 equiv). The vial was evacuated and backfilled with N_2 (3 x) and anhydrous 1,4-dioxane (1.5 mL, 0.2 M) added via syringe. Cy_2NMe (0.75 mmol, 2.5 equiv) was then added via syringe and the vial was submerged in a preheated oil bath at 60 °C and stirred for the indicated time below. Alternatively the vial may be sealed with a Teflon-lined vial cap and subsequently submerged in an oil bath. Upon cooling, the crude mixture was filtered through a plug of silica gel, which was subsequently rinsed with EtOAc. The filtrate was then concentrated *in vacuo* and the substrate purified via column chromatography on silica gel to provide the tryptophan derivative.

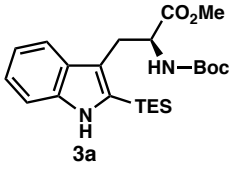
General Procedure I: In a glovebox, a 2-dram vial was charged with bromoaniline (0.3 mmol, 1.0 equiv), alkyne **2** (0.6 mmol, 2.0 equiv), Cy_2NMe (0.75 mmol, 2.5 equiv), $Pd[P(P^tBu)_3]_2$ (0.015 mmol, 0.05 equiv) and anhydrous 1,4-dioxane (1.5 mL, 0.2 M). The vial was sealed and heated to 60 °C until there was complete consumption of starting material (12 – 72 h). In most cases the solution became cloudy as the reaction progressed. Upon cooling, the crude mixture was filtered through a plug of silica, which was subsequently washed with EtOAc. The organics were concentrated and the crude residue was purified by chromatography on silica gel to provide the tryptophan derivative.

General Procedure II: In a glovebox, a 2-dram vial was charged with bromoaniline (0.3 mmol, 1.0 equiv), alkyne **2** (0.6 mmol, 2.0 equiv), Cy_2NMe (0.75 mmol, 2.5 equiv), $Pd[P(P^tBu)_3]_2$ (0.015 mmol, 0.05 equiv) and anhydrous 1,4-dioxane (1.5 mL, 0.2 M). The vial was sealed and heated to 80 °C until there was complete consumption of starting material (12 – 72 h). In most cases, the solution became cloudy as the reaction progressed. Upon cooling, the crude mixture was filtered through a plug of silica, which was subsequently washed with EtOAc. The organics were concentrated and the crude residue was purified by chromatography on silica gel to provide tryptophan derivative.

General Procedure III: In a glovebox, a 2-dram vial was charged with bromoaniline (0.3 mmol, 1.0 equiv), alkyne **2** (0.6 mmol, 2.0 equiv), Cy_2NMe (0.75 mmol, 2.5 equiv), $Pd[P(P^tBu)_3]_2$ (0.015 mmol, 0.05 equiv) and anhydrous 1,4-dioxane (1.5 mL, 0.2 M). The vial was sealed and heated to 80 °C until there was complete consumption of starting material (12 – 72 h). In most cases, the solution became cloudy as the reaction progressed. Upon cooling, the crude

mixture was filtered through a plug of silica, which was subsequently washed with EtOAc. The organics were concentrated and the crude residue was dissolved in 1M TBAF in THF. After 20 minutes, aqueous NH_4Cl was added and the reaction mixture was partitioned in a separatory funnel. The aqueous layer was back-extracted with EtOAc (3 X 15 mL). The organics were then recombined, washed with brine, dried over Na_2SO_4, filtered, and concentrated. The crude residue was purified using silica gel chromatography to provide the tryptophan derivative.

Tryptophan 3a



3a

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (88% hexanes, 12% EtOAc) to afford **3a** as a colorless oil (113.6 mg, 0.26 mmol, 88% yield).

^{1}H NMR: (500 MHz, $CDCl_3$, Major Rotamer) δ 8.04 (s, 1H), 7.57 (d, *J* = 7.9 Hz, 1H), 7.36 (d, *J* = 8.1 Hz, 1H), 7.18 (ddd, *J* = 8.1, 6.9, 1.1 Hz, 1H), 7.09 (t, *J* = 7.4 Hz, 1H), 4.93 (d, *J* = 7.7 Hz, 1H), 4.57 (dd, *J* = 14.4, 7.1 Hz, 1H), 3.63 (s, 3H), 3.36 – 3.18 (m, 2H), 1.36 (s, 9H), 1.05 – 0.98 (m, 9H), 0.97 – 0.89 (m, 6H);

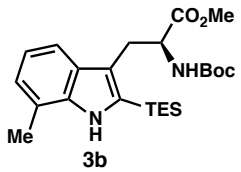
^{13}C NMR: (126 MHz, $CDCl_3$) δ 173.4, 155.1, 138.5, 132.8, 128.6, 122.4, 119.5, 119.3, 118.9, 110.8, 79.6, 54.2, 52.2, 29.3, 28.2, 7.4, 3.7;

FTIR: (NaCl, thin film): cm^{-1}; 3383, 2954, 2911, 2875, 1739, 1700, 1501, 1456, 1367, 1284, 1164;

$[\alpha]_D^{25}$ = +1.4 (*c* = 1.4, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 433.2517, found 433.2519.

Tryptophan 3b



3b

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (88% hexanes, 12% EtOAc) to afford **3b** as a colorless oil (102.9 mg, 0.230 mmol, 77% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 7.75 (d, *J* = 8.4 Hz, 1H), 7.61 (d, *J* = 8.4 Hz, 1H), 7.35 (ddd, *J* = 8.5, 7.1, 1.3 Hz, 1H), 7.27 (ddd, *J* = 7.9, 7.1, 0.9 Hz, 1H), 5.57 (d, *J* = 8.6 Hz, 1H), 4.40 (q, *J* = 7.9 Hz, 1H), 3.54 (s, 3H), 3.37 (dd, *J* = 14.4, 6.7 Hz, 1H), 3.20 (dd, *J* = 14.4, 8.2 Hz, 1H), 2.78 (s, 3H), 1.29 (s, 9H), 1.04 – 0.83 (m, 15H);

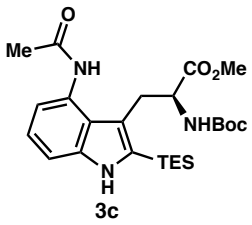
^{13}C NMR: (126 MHz, CD_3CN) δ 173.4, 171.0, 156.1, 138.0, 137.1, 133.7, 131.2, 125.9, 123.4, 120.4, 115.2, 80.0, 55.9, 52.7, 28.8, 28.4, 27.0, 8.6, 6.8;

FTIR: (NaCl, thin film): cm^{-1}; 3396, 2954, 2912, 2874, 1704, 1498, 1366, 1279, 1217, 1163, 1018;

$[\alpha]_D^{25}$ = –5.8 (*c* = 0.40, $CHCl_3$);

HRMS: (MM) calc'd $[M–C_4H_9]^+$ 391.2048, found 391.2038.

Tryptophan 3c



3c

Prepared following *General Procedure II* (12 h). The crude residue was purified by silica gel chromatography (80% hexanes, 20% acetone) to afford **3c** as a white, amorphous solid (114.3 mg, 0.234 mmol, 78% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 9.21 (s, 1H), 8.32 (s, 1H), 7.30 (d, *J* = 8.1 Hz, 1H), 7.07 (t, *J* = 7.7 Hz, 1H), 6.93 (d, *J* = 7.4 Hz, 1H), 5.54 (s, 1H), 4.33 (dd, *J* = 15.7, 7.6 Hz, 1H), 3.59 (s, 3H), 3.36 (dd, *J* = 14.7, 6.1 Hz, 1H), 3.05 – 3.00 (m, 1H), 2.13 (s, 3H), 1.34 – 1.18 (m, 9H), 1.03 – 0.89 (m, 15H);

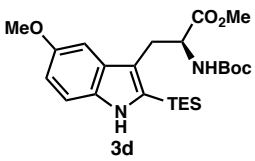
^{13}C NMR: (126 MHz, CD_3CN) δ 174.3, 171.1, 156.2, 141.6, 134.4, 130.5, 124.7, 122.8, 119.8, 118.5, 110.7, 79.9, 57.0, 52.6, 29.3, 28.3, 23.8, 7.7, 4.3;

FTIR: (NaCl, thin film): cm^{-1}; 3313, 2953, 1700, 1672, 1506, 1367, 1168;

$[\alpha]_D^{25}$ = –18.3 (*c* = 1.10, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 490.2732, found 490.2719.

Tryptophan 3d



3d

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (85% hexanes, 15% EtOAc) to afford **3d** as a colorless oil (102.2 mg, 0.220 mmol, 74% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 8.95 (s, 1H), 7.28 (d, *J* = 8.8 Hz, 1H), 7.03 (d, *J* = 1.5 Hz, 1H), 6.77 (dd, *J* = 8.8, 2.4 Hz, 1H), 5.53 (d, *J* = 8.2 Hz, 1H), 4.36 (dd, *J* = 14.6, 8.2 Hz, 1H), 3.82 (s, 3H), 3.60 (s, 3H), 3.26 (dd, *J* = 14.5, 6.1 Hz, 1H), 3.08 (dd, *J* = 14.5, 8.2 Hz, 1H), 1.28 (s, 9H), 1.01 – 0.95 (m, 9H), 0.95 – 0.91 (m, 6H);

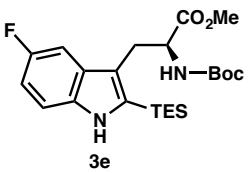
^{13}C NMR: (126 MHz, CD_3CN) δ 174.0, 156.1, 154.7, 135.2, 134.1, 130.0, 120.5, 113.3, 112.6, 101.1, 79.8, 56.3, 56.2, 52.6, 29.8, 28.4, 7.7, 4.2;

FTIR: (NaCl, thin film): cm^{-1}; 3379, 2953, 2874, 1700, 1620, 1506, 1437, 1391, 1366, 1218, 1164;

$[\alpha]_D^{25}$ = +6.3 (*c* = 3.75, $CHCl_3$);

HRMS: (MM) calc'd $[M–C_4H_9]^+$ 407.1997, found 407.1994.

Tryptophan 3e



3e

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (88% hexanes, 12% EtOAc) to afford **3e** as a colorless oil (97.2 mg, 0.216 mmol, 72% yield).

^{1}H NMR: (500 MHz, $CDCl_3$, Major Rotamer) δ 8.01 (s, 1H), 7.26 – 7.22 (m, 1H), 7.21 – 7.14 (m,

1H), 6.91 (ddd, J = 8.9, 8.9, 2.2 Hz, 1H), 4.93 (d, J = 8.2 Hz, 1H), 4.53 (dd, J = 14.7, 7.0 Hz, 1H), 3.65 (s, 3H), 3.30 – 3.14 (m, 2H), 1.35 (s, 9H), 1.05 – 0.97 (m, 9H), 0.96 – 0.88 (m, 6H);

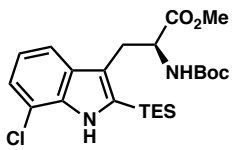
^{13}C NMR: (126 MHz, $CDCl_3$) δ 173.2, 157.7 (d, $J_{C\text{-}F}$ = 234.9 Hz), 155.0, 135.1 (d, $J_{C\text{-}F}$ = 13.4 Hz), 129.0 (d, $J_{C\text{-}F}$ = 9.2 Hz), 119.6 (d, $J_{C\text{-}F}$ = 4.7 Hz), 111.3 (d, $J_{C\text{-}F}$ = 9.8 Hz), 110.8 (d, $J_{C\text{-}F}$ = 26.5 Hz), 103.6 (d, $J_{C\text{-}F}$ = 23.6 Hz), 79.7, 54.2, 52.3, 29.5, 28.2, 7.4, 3.6;

FTIR: (NaCl, thin film): cm^{-1}; 3372, 2956, 2875, 1734, 1718, 1700, 1502, 1437, 1367, 1166, 1073, 1010;

$[\alpha]_D^{25}$ = +3.6 (c = 2.0, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 395.1797, found 395.1804.

Tryptophan 3f



3f

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (80% hexanes, 20% acetone) to afford **3f** as a colorless oil (114.3 mg, 0.245 mmol, 82% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 8.87 (s, 1H), 7.50 (d, J = 8.0 Hz, 1H), 7.17 (dd, J = 7.5, 0.8 Hz, 1H), 7.04 – 7.01 (m, 1H), 5.50 (d, J = 8.1 Hz, 1H), 4.37 (dd, J = 15.1, 7.9 Hz, 1H), 3.56 (s, 3H), 3.29 (dd, J = 14.5, 6.5 Hz, 1H), 3.12 (dd, J = 14.5, 8.0 Hz, 1H), 1.29 (s, 9H), 1.02 – 0.96 (m, 15H);

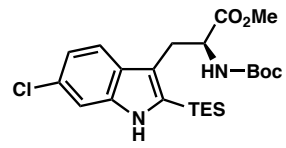
^{13}C NMR: (126 MHz, CD_3CN) δ 173.7, 156.1, 136.6, 135.3, 131.5, 122.6, 122.4, 120.8, 118.6, 116.9, 79.9, 56.2, 52.6, 29.7, 28.4, 7.7, 4.2;

FTIR: (NaCl, thin film): cm^{-1}; 3380, 2954, 2875, 1734, 1718, 1507, 1499, 1366, 1164;

$[\alpha]_D^{25}$ = +6.9 (c = 0.87, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 411.1501, found 411.1504.

Tryptophan 3g



3g

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (88% hexanes, 12% EtOAc) to afford **3g** as a colorless oil (103.9 mg, 0.222 mmol, 74% yield).

^{1}H NMR: (500 MHz, $CDCl_3$, Major Rotamer) δ 7.98 (s, 1H), 7.46 (d, J = 8.5 Hz, 1H), 7.33 (s, 1H), 7.04 (d, J = 8.5 Hz, 1H), 4.92 (d, J = 8.1 Hz, 1H), 4.55 (dd, J = 14.7, 7.1 Hz, 1H), 3.61 (s, 3H), 3.22 (d, J = 6.6 Hz, 2H), 1.35 (s, 9H), 1.04 – 0.97 (m, 9H), 0.94 – 0.88 (m, 6H);

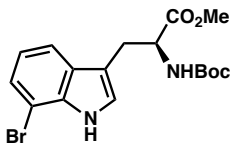
^{13}C NMR: (126 MHz, $CDCl_3$) δ 173.2, 155.0, 138.8, 133.9, 128.4, 127.3, 120.1, 119.7, 110.7, 79.8, 54.2, 52.3, 29.5, 28.2, 7.4, 3.6;

FTIR: (NaCl, thin film): cm^{-1}; 3369, 2954, 2875, 1738, 1699, 1505, 1439, 1392, 1367, 1338, 1163, 1062;

$[\alpha]_D^{25}$ = +7.1 (c = 1.63, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 467.2127, found 467.2129.

Tryptophan 3h



3h

Prepared following *General Procedure III* (36 h). The crude residue was purified by silica gel chromatography (80% hexanes, 20% acetone) to afford **3h** as a colorless oil (61.2 mg, 0.245 mmol, 52% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 9.33 (s, 1H), 7.54 (d, *J* = 7.9 Hz, 1H), 7.34 (d, *J* = 7.2 Hz, 1H), 7.17 (d, *J* = 1.7 Hz, 1H), 7.00 (dd, *J* = 7.8, 7.8 Hz, 1H), 5.51 (d, *J* = 7.4 Hz, 1H), 4.43 (dd, *J* = 13.5, 7.6 Hz, 1H), 3.64 (s, 3H), 3.23 (dd, *J* = 14.7, 5.4 Hz, 1H), 3.10 (dd, *J* = 14.7, 7.7 Hz, 1H), 1.35 (s, 9H);

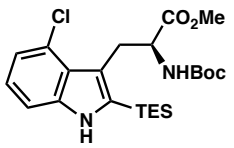
^{13}C NMR: (126 MHz, CD_3CN) δ 173.5, 156.2, 135.6, 130.0, 125.5, 125.0, 121.3, 118.9, 112.5, 105.2, 79.9, 55.3, 52.7, 28.4, 28.3;

FTIR: (NaCl, thin film): cm^{-1}; 3365, 2968, 1738, 1696, 1501, 1434, 1365, 1335;

$[\alpha]_D^{25}$ = +44.0 (*c* = 0.385, $CHCl_3$);

HRMS: (MM) calc'd $[M–C_5H_{10}O_2]^+$ 297.0233, found 297.0229.

Tryptophan 3i



3i

Prepared following *General Procedure II* (12 h, 80 °C). The crude residue was purified by silica gel chromatography (88% hexanes, 12% EtOAc) to afford **3i** as a white, amorphous solid (113.7 mg, 0.243 mmol, 82% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 9.29 (s, 1H), 7.38 (dd, *J* = 7.8, 0.9 Hz, 1H), 7.06 (t, *J* = 7.7 Hz, 1H), 7.02 (dd, *J* = 7.5, 1.2 Hz, 1H), 5.41 (d, *J* = 7.7 Hz, 1H), 4.53 (dd, *J* = 15.2, 8.8 Hz, 1H), 3.61 (s, 3H), 3.55 (dd, *J* = 14.3, 5.7 Hz, 1H), 3.28 – 3.17 (m, 1H), 1.23 (s, 9H), 1.02 – 0.89 (m, 15H);

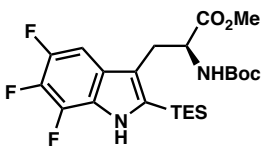
^{13}C NMR: (126 MHz, CD_3CN) δ 173.8, 156.1, 141.7, 136.0, 125.8, 125.7, 123.5, 121.1, 120.4, 111.4, 79.8, 57.2, 52.5, 29.8, 28.3, 7.7, 4.2;

FTIR: (NaCl, thin film): cm^{-1}; 3369, 2954, 2934, 2875, 1721, 1700, 1499, 1456, 1436, 1366, 1167;

$[\alpha]_D^{25}$ = –9.0 (*c* = 4.1, $CHCl_3$);

HRMS: (MM) calc'd $[M–C_4H_9]^+$ 411.1501, found 411.1505.

Tryptophan 3j



3j

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (88% hexanes, 12% EtOAc) to afford **3j** as a colorless oil (109.1 mg, 0.188 mmol, 72% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 9.36 (s, 1H), 7.25 (dd, *J* = 10.0, 7.1 Hz, 1H), 5.58 (d, *J* = 8.4 Hz, 1H), 4.32 (dd, *J* = 14.7, 8.5 Hz, 1H), 3.59 (s, 3H), 3.24 (dd, *J* = 14.7, 6.0 Hz, 1H), 3.04 (dd, *J* = 14.6, 8.7 Hz, 1H), 1.27 (s, 9H), 1.03 – 0.90 (m, 15H);

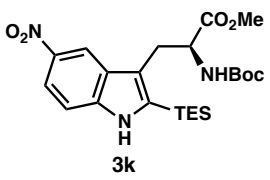
^{13}C NMR: (126 MHz, CD_3CN) δ 173.5, 156.0, 147.0 (dd, $J_{C\text{-}F}$ = 236.4, 11.9 Hz), 139.5 – 137.1 (m), 137.4 (d, $J_{C\text{-}F}$ = 3.6 Hz), 136.2 (ddd, $J_{C\text{-}F}$ = 239.4, 18.9, 12.5 Hz), 125.8 (dd, $J_{C\text{-}F}$ = 9.1, 5.4 Hz), 124.3 (dd, $J_{C\text{-}F}$ = 10.4, 2.1 Hz), 122.5 – 122.1 (m), 101.1 (d, $J_{C\text{-}F}$ = 19.1 Hz), 79.9, 56.2, 52.7, 29.4, 28.3, 7.6, 4.0;

FTIR: (NaCl, thin film): cm^{-1}; 3351, 2956, 2876, 1700, 1514, 1467, 1436, 1367, 1350, 1165;

$[\alpha]_D^{25}$ = +4.2 (*c* = 0.65, $CHCl_3$);

LRMS: (ESI) calc'd $[M–C_4H_9]^+$ 431.5, found 431.2.

Tryptophan 3k



3k

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (85% hexanes, 15% EtOAc) to afford **3k** as a yellow oil (105.0 mg, 0.219 mmol, 73% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 9.66 (s, 1H), 8.30 (d, *J* = 2.0 Hz, 1H), 7.91 (dd, *J* = 8.9, 2.1 Hz, 1H), 7.66 (d, *J* = 8.9 Hz, 1H), 5.59 (d, *J* = 8.4 Hz, 1H), 4.37 (dd, *J* = 15.0, 8.3 Hz, 1H), 3.57 (s, 3H), 3.32 (dd, *J* = 14.6, 6.3 Hz, 1H), 3.15 (dd, *J* = 14.6, 8.4 Hz, 1H), 1.26 (s, 9H), 1.05 – 0.92 (m, 15H);

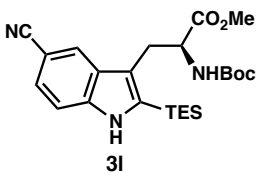
^{13}C NMR: (126 MHz, CD_3CN) δ 173.5, 144.0, 142.1, 138.1, 134.1, 121.9, 119.8, 114.9, 108.6, 79.9, 56.3, 52.7, 29.6, 28.3, 7.6, 4.0;

FTIR: (NaCl, thin film): cm^{-1}; 3380, 2968, 2873, 1736, 1716, 1696, 1508, 1330, 1162, 1065, 1004;

$[\alpha]_D^{25}$ = +7.9 (*c* = 0.75, $CHCl_3$);

LRMS: (ESI) calc'd $[M+H]^+$ 478.3, found 478.3.

Tryptophan 3l



3l

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (80% hexanes, 20% EtOAc) to afford **3l** as a white, amorphous solid (109.0 mg, 0.238 mmol, 79% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 9.48 (s, 1H), 8.03 (s, 1H), 7.51 (d, *J* = 8.5 Hz, 1H), 7.38 (dd, *J* = 8.5, 1.5 Hz, 1H), 5.66 (d, *J* = 8.8 Hz, 1H), 4.35 (td, *J* = 9.0, 5.5 Hz, 1H), 3.62 (s, 3H), 3.31 (dd, *J* = 14.6, 5.4 Hz, 1H), 3.10 (dd, *J* = 14.6, 9.2 Hz, 1H), 1.23 (s, 9H), 1.00 – 0.93 (m, 15H);

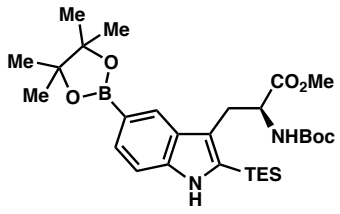
^{13}C NMR: (126 MHz, CD_3CN) δ 173.5, 155.9, 141.4, 136.8, 129.6, 126.0, 125.3, 122.3, 121.8, 113.0, 102.4, 79.8, 56.5, 52.7, 29.6, 28.3, 7.6, 4.0;

FTIR: (NaCl, thin film): cm^{-1}; 3350, 2953, 2878, 2218, 1728, 1696, 1508, 1370, 1167;

$[\alpha]_D^{25}$ = –2.3 (*c* = 2.2, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 458.2470, found 458.2454.

Tryptophan 3m



3m

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (85% hexanes, 15% EtOAc – 80% hexanes, 20% EtOAc) to afford **3m** as a white, amorphous solid (127.0 mg, 0.227 mmol, 76% yield).

^{1}H NMR: (500 MHz, $CDCl_3$, Major Rotamer) δ 8.10 – 7.98 (m, 2H), 7.61 (d, *J* = 8.2 Hz, 1H), 7.33 (d, *J* = 8.2 Hz, 1H), 4.90 (d, *J* = 8.1 Hz, 1H), 4.56 (dd, *J* = 14.4, 6.7 Hz, 1H), 3.75 (s, 3H), 3.31 (qd, *J* = 14.5, 6.5 Hz, 2H), 1.36 (d, *J* = 2.8 Hz, 12H), 1.32 (s, 9H), 1.03 – 0.97 (m, 9H), 0.96 – 0.90 (m, 6H);

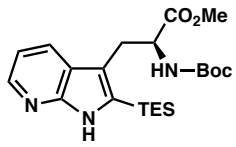
^{13}C NMR: (126 MHz, $CDCl_3$) δ173.1, 155.2, 140.5, 133.1, 128.6, 128.4, 126.7, 120.1, 110.2, 83.4, 79.5, 54.0, 52.3, 28.8, 28.2, 24.9, 7.4, 3.7 (carbon adjacent to boron was not observed);

FTIR: (NaCl, thin film): cm^{-1}; 3379, 2976, 2874, 1741, 1700, 1499, 1351, 1146;

$[\alpha]_D^{25}$ = +15.0 (*c* = 1.0, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 558.3406, found 558.3388.

Tryptophan 3n



3n

Prepared following *General Procedure I* (12 h). The crude residue was purified by silica gel chromatography (98% dichloromethane, 2% methanol) to afford **3n** as a light yellow oil (111.2 mg, 0.256 mmol, 85% yield).

^{1}H NMR: (500 MHz, $CDCl_3$, Major Rotamer) δ 9.76 (d, *J* = 14.4 Hz, 1H), 8.28 (d, *J* = 3.8 Hz, 1H), 7.90 (d, *J* = 7.7 Hz, 1H), 7.03 (dd, *J* = 7.7, 4.8 Hz, 1H), 5.14 (d, *J* = 8.4 Hz, 1H), 4.59 (dd, *J* = 15.0, 7.1 Hz, 1H), 3.59 (s, 3H), 3.25 (d, *J* = 6.8 Hz, 2H), 1.33 (s, 9H), 1.02 – 0.88 (m, 15H);

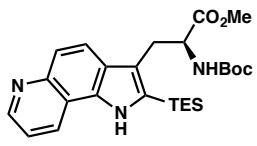
^{13}C NMR: (126 MHz, $CDCl_3$) δ173.3, 155.0, 150.8, 143.3, 134.0, 127.3, 120.9, 118.0, 115.2, 79.7, 54.2, 52.2, 29.8, 28.1, 7.3, 3.6;

FTIR: (NaCl, thin film): cm^{-1}; 3380, 3226, 2953, 1743, 1691, 1582, 1496, 1439, 1367, 1283, 1172;

$[\alpha]_D^{25}$ = +8.7 (*c* = 2.5, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 434.2470, found 434.2490.

Tryptophan 3p



3p

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (98% dichloromethane, 2% methanol) to afford **3p** as a light yellow oil (119.5 mg, 0.249 mmol, 83% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 9.78 (s, 1H), 8.78 (dd, *J* = 4.3, 1.7 Hz, 1H), 8.76

(ddd, *J* = 8.3, 1.5, 0.7 Hz, 1H), 7.86 (d, *J* = 8.9 Hz, 1H), 7.63 (dd, *J* = 8.9, 0.8 Hz, 1H), 7.47 (dd, *J* = 8.3, 4.3 Hz, 1H), 5.60 (d, *J* = 8.3 Hz, 1H), 4.40 (dd, *J* = 15.0, 7.8 Hz, 1H), 3.57 (s, 3H), 3.37 (dd, *J* = 14.5, 6.6 Hz, 1H), 3.22 (dd, *J* = 14.5, 8.0 Hz, 1H), 1.26 (s, 9H), 1.01 (s, 15H);

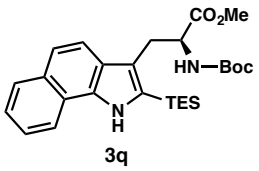
^{13}C NMR: (126 MHz, CD_3CN) δ 173.8, 156.1, 148.6, 147.4, 133.9, 133.3, 130.0, 125.6, 123.3, 123.1, 122.0, 121.2, 117.8, 79.9, 56.6, 52.7, 29.5, 28.3, 7.8, 4.4;

FTIR: (NaCl, thin film): cm^{-1}; 3350, 2953, 2873, 1734, 1717, 1700, 1696, 1570, 1496, 1377, 1164;

$[\alpha]_D^{25}$ = +8.7 (*c* = 1.2, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$484.2626, found 484.2621.

Tryptophan 3q



3q

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (88% hexanes, 12% EtOAc) to afford **3q** as a colorless foam (103.2 mg, 0.213 mmol, 71% yield).

^{1}H NMR: (500 MHz, $CDCl_3$, Major Rotamer) δ 8.67 (s, 1H), 8.05 (d, *J* = 8.1 Hz, 1H), 7.92 (d, *J* = 8.0 Hz, 1H), 7.64 (d, *J* = 8.7 Hz, 1H), 7.53 (ddd, *J* = 8.2, 6.9, 1.3 Hz, 1H), 7.49 (d, *J* = 8.7 Hz, 1H), 7.44 (ddd, *J* = 8.1, 7.0, 1.1 Hz, 1H), 4.97 (d, *J* = 7.9 Hz, 1H), 4.61 (*app* q, *J* = 7.1 Hz, 1H), 3.62 (s, 3H), 3.34 (d, *J* = 6.8 Hz, 2H), 1.35 (s, 9H), 1.08 – 1.02 (m, 9H), 1.02 – 0.97 (m, 6H);

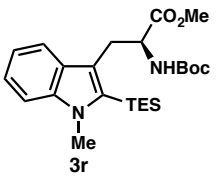
^{13}C NMR: (126 MHz, $CDCl_3$) δ173.3, 155.1, 133.6, 130.7, 130.6, 128.8, 125.4, 124.6, 124.2, 121.4, 121.3, 120.4, 119.4, 118.9, 79.7, 54.4, 52.2, 28.2, 24.7, 7.5, 3.8;

FTIR: (NaCl, thin film): cm^{-1}; 3409, 3350, 2953, 2868, 1743, 1694, 1501, 1392, 1362, 1165;

$[\alpha]_D^{25}$ = +54.8 (*c* = 0.97, $CHCl_3$);

HRMS: (MM) calc'd $[M–C_4H_9]^+$ 427.2048, found 427.2066.

Tryptophan 3r



3r

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (88% hexanes, 12% EtOAc) to afford **3r** as a colorless oil (70.1 mg, 0.156 mmol, 52% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 7.51 (d, *J* = 8.0 Hz, 1H), 7.35 (d, *J* = 8.3 Hz, 1H), 7.20 (ddd, *J* = 8.2, 6.9, 1.1 Hz, 1H), 7.04 (ddd, *J* = 7.9, 7.0, 0.9 Hz, 1H), 5.41 (d, *J* = 7.3 Hz, 1H), 4.33 (dd, *J* = 15.1, 7.5 Hz, 1H), 3.83 (s, 3H), 3.52 (s, 3H), 3.33 (dd, *J* = 14.6, 7.1 Hz, 1H), 3.18 (dd, *J* = 14.5, 7.4 Hz, 1H), 1.31 (s, 9H), 1.03 – 0.95 (m, 15H);

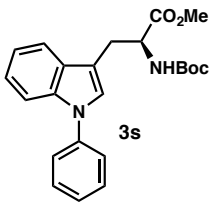
^{13}C NMR: (126 MHz, CD_3CN) δ 173.9, 156.1, 141.0, 135.5, 129.6, 123.1, 121.8, 119.6, 119.5, 110.2, 79.9, 56.4, 52.5, 33.8, 28.9, 28.4, 7.9, 5.2;

FTIR: (NaCl, thin film): cm^{-1}; 3350, 2956, 2876, 1700, 1516, 1465, 1367, 1165;

$[\alpha]_D^{25}$ = +4.9 (*c* = 0.34, $CHCl_3$);

HRMS: (MM) calc'd $[M–C_4H_9]^+$ 391.2048, found 391.2034.

Tryptophan 3s



Prepared following *General Procedure III* (12 h). The crude residue was purified by silica gel chromatography (20% acetone, 80% hexanes) to afford **3s** as a colorless oil (80.2 mg, 0.203 mmol, 68% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 7.63 (d, *J* = 7.8 Hz, 1H), 7.58 – 7.50 (m, 5H), 7.41 – 7.35 (m, 1H), 7.31 (s, 1H), 7.22 (ddd, *J* = 8.3, 7.0, 1.3 Hz, 1H), 7.17 (ddd, *J* = 8.0, 7.0, 1.1 Hz, 1H), 5.58 (d, *J* = 7.8 Hz, 1H), 4.51 (dd, *J* = 13.5, 7.7 Hz, 1H), 3.67 (s, 3H), 3.31 (dd, *J* = 14.7, 5.4 Hz, 1H), 3.18 (dd, *J* = 14.7, 7.6 Hz, 1H), 1.35 (s, 9H);

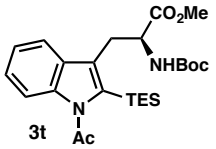
^{13}C NMR: (126 MHz, CD_3CN) δ 173.6, 156.3, 140.4, 136.7, 130.7, 130.0, 127.8, 127.3, 124.8, 123.5, 121.1, 120.0, 113.0, 111.4, 79.9, 55.2, 52.7, 28.4;

FTIR: (NaCl, thin film): cm^{-1}; 3380, 2966, 2930, 1741, 1714, 1501, 1455, 1367;

$[\alpha]_D^{25}$ = +32.1 (*c* = 1.86, $CHCl_3$);

HRMS: (MM) calc'd $[M–C_4H_9]^+$ 339.1339, found 339.1326.

Tryptophan 3t



Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (86% hexanes, 14% EtOAc) to afford **3t** as a colorless oil (107.0 mg, 0.226 mmol, 75% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 7.75 (d, *J* = 8.4 Hz, 1H), 7.61 (d, *J* = 7.8 Hz, 1H), 7.35 (ddd, *J* = 8.5, 7.2, 1.3 Hz, 1H), 7.27 (ddd, *J* = 7.6, 7.6, 0.8 Hz, 1H), 5.57 (d, *J* = 8.3 Hz, 1H), 4.40 (dd, *J* = 15.2, 8.0 Hz, 1H), 3.54 (s, 3H), 3.37 (dd, *J* = 14.4, 6.7 Hz, 1H), 3.20 (dd, *J* = 14.3, 8.2 Hz, 1H), 2.78 (s, 3H), 1.30 (s, 9H), 1.00 – 0.89 (m, 15H);

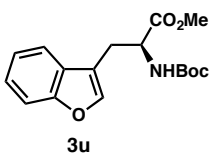
^{13}C NMR: (126 MHz, CD_3CN) δ 173.4, 171.0, 156.1, 138.0, 137.1, 133.7, 131.2, 125.9, 123.4, 120.4, 115.2, 80.0, 55.9, 52.7, 28.8, 28.4, 27.0, 8.6, 6.8;

FTIR: (NaCl, thin film): cm^{-1}; 3373, 2953, 2874, 1746, 1700, 1499, 1435, 1369, 1321, 1223, 1167, 1109;

$[\alpha]_D^{25}$ = +5.0 (*c* = 0.69, $CHCl_3$);

HRMS: (MM) calc'd $[M–C_4H_9]^+$ 419.1997, found 419.1986.

Tryptophan 3u



Prepared following *General Procedure III* (24 h). The crude residue was purified by silica gel chromatography (25% acetone, 75% hexanes) to afford **3u** as a colorless oil (68.9 mg, 0.216 mmol, 72% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 7.60 (d, *J* = 7.2 Hz, 1H), 7.58 (s, 1H), 7.49 (d, *J* = 8.1 Hz, 1H), 7.32 (ddd, *J* = 8.3, 7.3, 1.4 Hz, 1H), 7.27 (td, *J* = 7.5, 1.0 Hz, 1H), 5.63 (d, *J* = 6.5 Hz, 1H), 4.48 (dd, *J* = 13.5, 7.9 Hz, 1H), 3.67 (s, 3H), 3.20 (dd, *J* = 14.8, 5.3 Hz, 1H), 3.07 (dd, *J* = 14.8, 8.0 Hz, 1H), 1.35 (s, 9H);

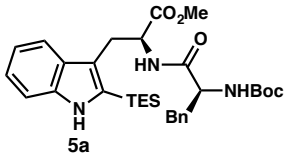
^{13}C NMR: (126 MHz, CD_3CN) δ 173.2, 156.0, 144.1, 128.8, 125.4, 123.6, 120.7, 116.7, 112.2, 80.0, 54.5, 52.8, 28.4, 26.6;

FTIR: (NaCl, thin film): cm^{-1}; 3375, 2977, 2925, 1744, 1716, 1690, 1505, 1455, 1367, 1165;

$[\alpha]_D^{25}$ = +16.8 (*c* = 0.64, $CHCl_3$);

LRMS: (ESI) calc'd $[M–C_4H_9]^+$ 263.2, found 263.2.

Tryptophan 5a



5a

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (80% hexanes, 20% acetone) to afford **5a** as a colorless oil (109.1 mg, 0.188 mmol, 63% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 9.12 (s, 1H), 7.59 (dd, *J* = 7.9, 0.8 Hz, 1H), 7.39 (d, *J* = 8.1 Hz, 1H), 7.29 – 7.19 (m, 3H), 7.16 – 7.05 (m, 3H), 7.04 (ddd, *J* = 9.0, 5.6, 1.8 Hz, 1H), 5.44 (d, *J* = 6.4 Hz, 1H), 4.62 (dd, *J* = 13.1, 6.8 Hz, 1H), 4.24 (ddd, *J* = 8.4, 8.4, 5.7 Hz, 1H), 3.48 (s, 3H), 3.24 (dd, *J* = 14.4, 7.8 Hz, 1H), 3.10 (dd, *J* = 14.4, 7.9 Hz, 1H), 2.85 (dd, *J* = 13.9, 5.4 Hz, 1H), 2.62 (dd, *J* = 13.9, 8.5 Hz, 1H), 1.33 (s, 9H), 1.03 – 0.88 (m, 15H);

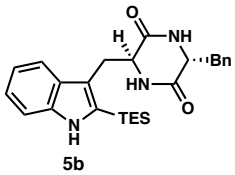
^{13}C NMR: (126 MHz, CD_3CN) δ 173.1, 172.1, 156.2, 139.8, 138.3, 133.3, 130.2, 129.7, 129.1, 127.4, 122.8, 120.4, 119.8, 119.4, 112.0, 79.9, 56.2, 55.4, 52.6, 38.4, 29.7, 28.4, 7.7, 4.2;

FTIR: (NaCl, thin film): cm^{-1}; 3380, 2948, 2878, 1736, 1666, 1506, 1367, 1244, 1165;

$[\alpha]_D^{25}$ = –4.2 (*c* = 1.6, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 580.3201, found 580.3206.

Tryptophan 5b



5b

Prepared following *General Procedure I* (72 h). The crude residue was purified by silica gel chromatography (55% hexanes, 40% EtOAc, 5% methanol) to afford **5b** as a colorless oil (95.2 mg, 0.213 mmol, 71% yield).

^{1}H NMR: (500 MHz, $CDCl_3$) δ 8.15 (s, 1H), 7.39 (d, *J* = 8.2 Hz, 1H), 7.36 – 7.32 (m, 2H), 7.29 (ddd, *J* = 6.3, 5.1, 2.1 Hz, 2H), 7.22 (ddd, *J* = 8.1, 7.0, 1.1 Hz, 1H), 7.18 (dd, *J* = 8.0, 1.2 Hz, 2H), 7.10 (ddd, *J* = 7.9, 7.0, 0.9 Hz, 1H), 6.94 (d, *J* = 2.2 Hz, 1H), 5.64 (s, 1H), 4.24 (ddd, *J* = 5.2, 5.2, 2.5 Hz, 1H), 3.59 (dd, *J* = 14.5, 3.8 Hz, 1H), 3.45 (dd, *J* = 11.5, 3.8 Hz, 1H), 3.14 (d, *J* = 5.1 Hz, 2H), 2.87 (dd, *J* = 14.5, 11.5 Hz, 1H), 1.02 – 0.94 (m, 9H), 0.90 – 0.82 (m, 6H);

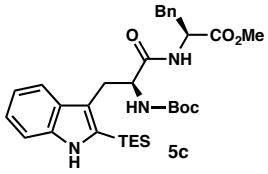
^{13}C NMR: (126 MHz, $CDCl_3$) δ 168.8, 167.0, 138.7, 134.8, 133.9, 129.9, 128.9, 127.6, 127.6, 122.8, 119.7, 118.7, 118.0, 111.1, 56.6, 53.3, 40.2, 30.0, 7.4, 3.7;

FTIR: (NaCl, thin film): cm^{-1}; 3356, 3226, 2958, 2864, 1676, 1451, 1437, 1316;

$[\alpha]_D^{25}$ = +5.6 (*c* = 0.47, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 448.2415, found 448.2426.

Tryptophan 5c



5c

Prepared following *General Procedure I* (36 h). The crude residue was purified by silica gel chromatography (80% hexanes, 20% acetone) to afford **5c** as a colorless oil (108.0 mg, 0.186 mmol, 62% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 9.09 (s, 1H), 7.59 (dd, *J* = 7.9, 0.8 Hz, 1H), 7.40 (d, *J* = 8.1 Hz, 1H), 7.29 – 7.19 (m, 3H), 7.16 – 7.05 (m, 3H), 7.05 – 7.01 (m, 1H), 6.69 (d, *J* = 6.1 Hz, 1H), 5.24 (d, *J* = 6.4 Hz, 1H), 4.62 (q, *J* = 6.8 Hz, 1H), 4.23 (dt, *J* = 8.4, 5.7 Hz, 1H), 3.59 (s, 3H), 3.24 (ddd, *J* = 14.4, 6.5, 4.7 Hz, 1H), 3.08 – 2.91 (m, 3H), 1.25 (s, 9H), 1.03 – 0.88 (m, 15H);

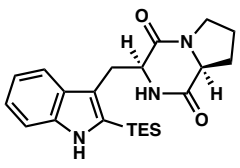
^{13}C NMR: (126 MHz, CD_3CN) δ 172.5, 172.3, 140.0, 137.5, 133.3, 130.3, 129.5, 129.3, 127.7, 122.9, 120.9, 119.7, 119.7, 112.0, 79.9, 57.1, 54.2, 52.7, 38.2, 29.5, 28.3, 7.7, 4.2;

FTIR: (NaCl, thin film): cm^{-1}; 3370, 2953, 2878, 1745, 1666, 1508, 1449, 1370, 1241, 1170;

$[\alpha]_D^{25}$ = +10.0 (*c* = 1.06, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 580.3201, found 580.3206.

Tryptophan 5d



5d

Prepared following *General Procedure I* (72 h). The crude residue was purified by silica gel chromatography (55% hexanes, 40% EtOAc, 5% methanol) to afford **5d** as an amorphous, white solid (98.6 mg, 0.249 mmol, 83% yield).

^{1}H NMR: (500 MHz, $CDCl_3$) δ 8.12 (s, 1H), 7.56 (dd, *J* = 7.9, 0.7 Hz, 1H), 7.40 (ddd, *J* = 8.2, 0.8, 0.8 Hz, 1H), 7.22 (ddd, *J* = 8.2, 7.0, 1.1 Hz, 1H), 7.11 (ddd, *J* = 8.0, 7.0, 0.9 Hz, 1H), 5.59 (s, 1H), 4.42 (dd, *J* = 11.8, 2.4 Hz, 1H), 4.07 (dd, *J* = 11.6, 4.5 Hz, 1H), 3.84 (dd, *J* = 15.0, 3.9 Hz, 1H), 3.75 – 3.66 (m, 1H), 3.65 – 3.54 (m, 1H), 3.00 (dd, *J* = 15.0, 11.8 Hz, 1H), 2.39 – 2.29 (m, 1H), 2.13 – 2.00 (m, 2H), 1.99 – 1.87 (m, 1H), 1.04 – 0.98 (m, 9H), 0.94 – 0.85 (m, 6H);

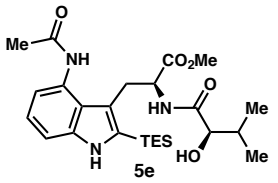
^{13}C NMR: (126 MHz, $CDCl_3$) δ 169.0, 165.7, 138.8, 133.6, 127.9, 123.0, 119.9, 118.8, 118.3, 111.3, 59.2, 54.8, 45.4, 28.4, 27.5, 22.6, 7.4, 3.8;

FTIR: (NaCl, thin film): cm^{-1}; 3365, 2953, 2873, 1671, 1456, 1412, 1303, 1239;

$[\alpha]_D^{25}$ = –34.4 (*c* = 0.82, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 398.2258, found 398.2272.

Tryptophan 5e



Prepared following *General Procedure II* (0.87 mmol scale, 12 h). The crude residue was purified by silica gel chromatography (100% EtOAc) to afford **5e** as a light yellow oil (370.2 mg, 0.756 mmol, 86% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 9.25 (s, 1H), 8.40 (s, 1H), 7.31 (d, *J* = 8.1 Hz, 1H), 7.14 (d, *J* = 5.6 Hz, 1H), 7.08 (t, *J* = 7.8 Hz, 1H), 6.92 (d, *J* = 7.4 Hz, 1H), 4.49 (dt, *J* = 10.3, 6.3 Hz, 1H), 3.61 (s, 3H), 3.62 – 3.58 (m, 1H), 3.55 (d, *J* = 5.9 Hz, 1H), 3.41 (dd, *J* = 14.7, 6.2 Hz, 1H), 3.14 (dd, *J* = 14.7, 10.3 Hz, 1H), 2.16 (s, 3H), 1.88 – 1.76 (m, 1H), 1.07 – 0.94 (m, 15H), 0.82 (d, *J* = 6.9, 3H), 0.66 (d, *J* = 6.8 Hz, 3H);

^{13}C NMR: (126 MHz, CD_3CN) δ 174.4, 173.6, 171.5, 141.7, 134.9, 130.2, 124.9, 122.9, 119.4, 118.9, 110.9, 76.3, 52.6, 32.6, 29.5, 29.3, 23.8, 19.4, 15.6, 7.7, 4.3;

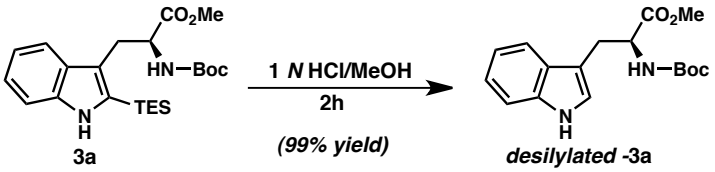
FTIR: (NaCl, thin film): cm^{-1}; 3324, 2956, 2875, 1742, 1657, 1516, 1435, 1369;

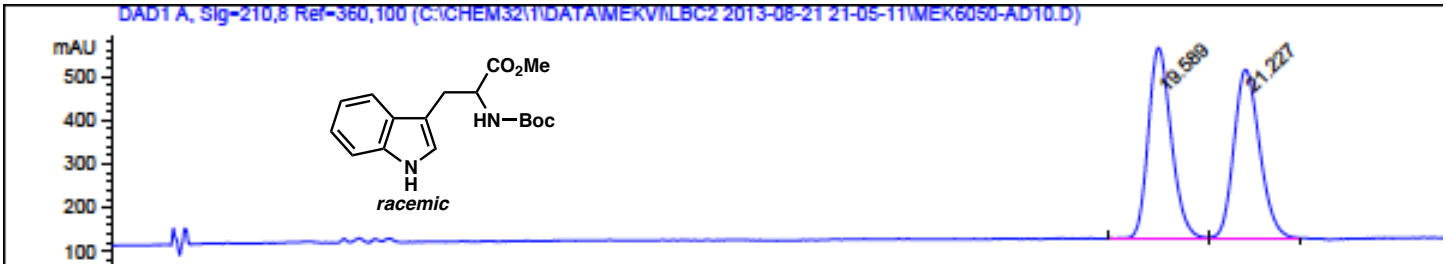
$[\alpha]_D^{25}$ = –1.8 (*c* = 1.3, $CHCl_3$);

HRMS: (MM) calc'd $[M+H]^+$ 490.2732, found 490.2772.

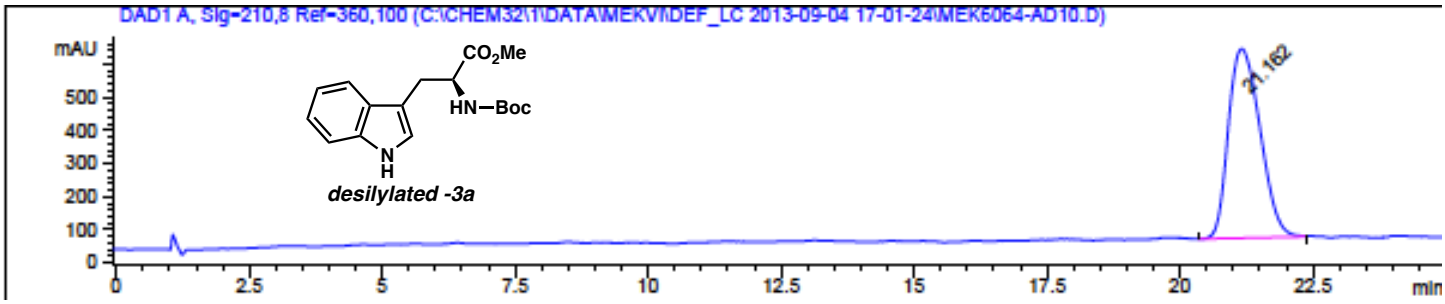
VI. Stability of the Tryptophan Stereocenter

In order to confirm that the tryptophan products were not undergoing deleterious racemization under the reaction conditions, tryptophan **3a** was desilylated with 1 *N* HCl/MeOH and compared to *racemic N*-Boc-tryptophan methyl ester using chiral supercritical fluid chromatography (SFC) (AD-H, 2.5 mL/min, 10% IPA in CO_2, λ = 254 nm): t_R(minor) = 19.6 min, t_R(major) = 21.2 min. We observed no racemization of the tryptophan stereocenter under the reaction conditions. Additionally, Larock indole syntheses using dipeptide-derived alkynes to provide tryptohans **5a** – **5b** show the formation of a single diastereomer of product by crude ^{1}H NMR and LCMS, further supporting the stability of the tryptophan stereocenter under Larock conditions. The low optical rotations exhibited by tryptophans **3a** – **3u** are consistent with literature values of related compounds.[3,4]





Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.589	BV	0.4601	1.31030e4	436.62933	50.0927
2	21.227	VB	0.5243	1.30545e4	386.23413	49.9073

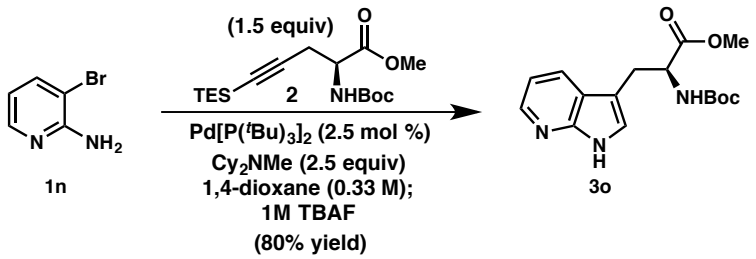


Signal 1: DAD1 A, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.162	VB	0.6657	2.36458e4	570.35565	100.0000

[3] Ma, B.; Banerjee, B.; Litvinov, D. N.; He, L.; Castle, S. L. *J. Am. Chem. Soc.* **2009**, *132*, 1159.
[4] Preciado, S.; Mendive-Tapia, L.; Albericio, F.; Lavilla, R. *J. Org. Chem.* **2013**, *78*, 8129.

VII. Scale-up and Desilylation of Tryptophan 3o

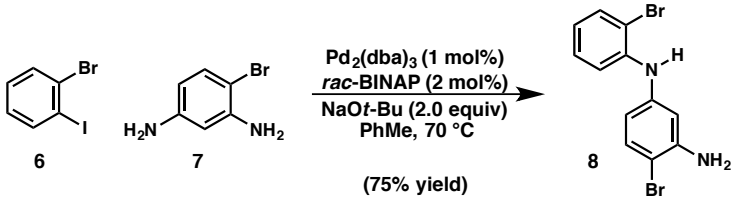


In a glovebox, pyridyl aniline **1n** (865 mg, 5.0 mmol, 1.0 equiv), alkyne **2** (2.56 g, 7.5 mmol, 1.5 equiv), $Pd[P(^tBu)_3]_2$ (64 mg, 0.125 mmol, 0.025 equiv), and Cy_2NMe (2.7 mL, 12.5 mmol, 2.5 equiv) were combined in a 50 mL flask. The solids were dissolved in 15 mL 1,4-dioxane and the solution was heated to an internal temperature of 60 °C for 30 h.[5] Upon cooling, the milky yellow solution was filtered through a silica plug, which was washed thoroughly with EtOAc. The solution was concentrated and then redissolved in 50 mL EtOAc and 1 M TBAF in THF (5 mL). After 20 minutes, aqueous NH_4Cl was added and the reaction mixture was partitioned in a separatory funnel. The aqueous layer was back extracted with EtOAc (3 x 150 mL). The organics were then recombined, washed with brine, dried over Na_2SO_4, filtered and concentrated. The crude residue was purified using silica gel chromatography (60% hexanes, 35% EtOAc, 5% methanol) to afford tryptophan **3o** as a light yellow solid (1.28 g, 80% yield).

^{1}H NMR: (500 MHz, CD_3CN, Major Rotamer) δ 10.06 (s, 1H), 8.23 (dd, *J* = 4.8, 1.5 Hz, 1H), 7.90 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.20 (s, 1H), 7.06 (dd, *J* = 7.9, 4.7 Hz, 1H), 5.64 (d, *J* = 8.3 Hz, 1H), 4.45 (dd, *J* = 5.4, 7.8 Hz, 1H), 3.64 (s, 3H), 3.23 (dd, *J* = 14.7, 5.4 Hz, 1H), 3.11 (dd, *J* = 14.7, 7.5 Hz, 1H), 1.34 (s, 9H);
^{13}C NMR: (126 MHz, CD_3CN) δ 173.6, 156.2, 149.6, 143.8, 127.8, 125.0, 120.7, 116.3, 109.9, 79.9, 55.3, 52.7, 28.4, 28.4;
FTIR: (NaCl, thin film): cm^{-1}; 3365, 2978, 1743, 1698, 1511, 1434, 1362;
$[\alpha]_D^{25}$ = 49.1 (*c* = 1.25, $CHCl_3$);
HRMS: (MM) calc'd $[M+H]^+$ 320.1605, found 320.1594.

[5] For larger scale preparations (>1.00 mmol) maintaining an *internal* temperature ≥ 60 °C is critical for conversion.

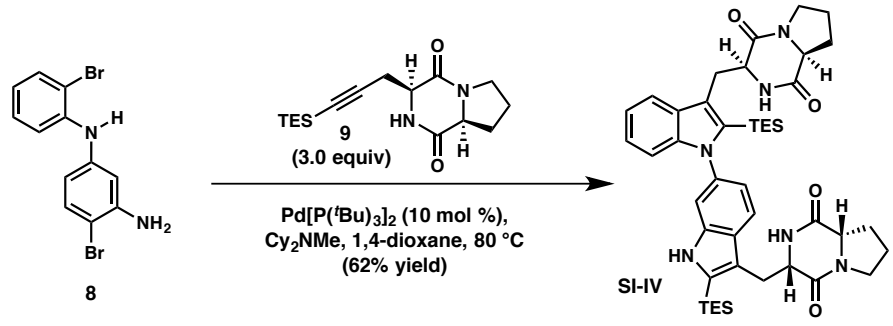
VIII. Total Synthesis of (–)-Aspergilazine A
Preparation of Diarylamine 8



In a glove box, a flame-dried 250 mL flask was charged with iodobromobenzene (**6**) (771 μL, 6.0 mmol, 1.0 equiv), dianiline **7** (1.34 g, 7.2 mmol, 1.2 equiv), $Pd_2(dba)_3$ (54 mg, 0.06 mmol, 0.01 equiv), *rac*-BINAP (75 mg, 0.12 mmol, 0.02 equiv), and NaO^{t}Bu (865 mg, 9.0 mmol, 1.5 equiv). PhMe (60 mL) was added and the reaction flask was sealed and heated to 70 °C for 3.5 hours. Upon cooling, the reaction mixture was filtered through a plug of silica gel, which was flushed with EtOAc. The organics were concentrated and purified by silica gel chromatography (20% acetone, 80% hexanes) to provide the diarylamine **8** as a light yellow oil (1.54 g, 75% yield).

^{1}H NMR: (500 MHz, $CDCl_3$) δ 7.62 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.39 (d, *J* = 8.5 Hz, 1H), 7.34 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.26 (ddd, *J* = 8.2, 7.2, 1.5 Hz, 1H), 6.86 (ddd, *J* = 8.0, 7.2, 1.6 Hz, 1H), 6.59 (d, *J* = 2.5 Hz, 1H), 6.52 (dd, *J* = 8.5, 2.6 Hz, 1H), 6.03 (s, 1H), 4.14 (s, 2H);
^{13}C NMR: (126 MHz, $CDCl_3$) δ 144.6, 141.9, 140.7, 132.9, 132.8, 128.0, 121.2, 116.6, 112.5, 111.1, 106.3, 101.9;
FTIR: (NaCl, thin film): cm^{-1}; 3464, 3380, 1612, 1582, 1511, 1459, 1407, 1330, 1303, 1276;
HRMS: (MM) calc'd $[M+H]^+$ 340.9284, found 340.9264.

Synthesis of Bis-Triethylsilyl-(–)-Aspergilazine A



In a glovebox, a one-dram vial was charged with diarylamine **8** (35 mg, 0.1 mmol, 1.0 equiv), alkyne **9**[6] (94 mg, 0.3 mmol, 3.0 equiv), Cy_2NMe (55 μL, 0.25 mmol, 2.5 equiv), $Pd[P(^tBu)_3]_2$ (5.2 mg, 0.01 mmol, 0.1 equiv) and 1,4-dioxane (500 μL). The vial was sealed and heated to 80 °C for 4 hours. Upon cooling, the reaction mixture was filtered

[6] Kieffer, M. E.; Chuang, K. V.; Reisman, S. E. *J. Am. Chem. Soc.* **2013**, *135*, 5557.

through celite, which was washed with EtOAc (15 mL). The organics were concentrated and the crude reaction mixture was purified by preparative reverse phase HPLC (65–85% acetonitrile in H_2O, 30 mL/min, 20 min) to give the product as a colorless solid (49.5 mg, 62% yield).

^{1}H NMR: (500 MHz, CD_2Cl_2, Major Rotamer) δ 8.41 (d, *J* = 11.0 Hz, 1H), 7.66 (dd, *J* = 8.3, 2.5 Hz, 1H), 7.64 – 7.59 (m, 1H), 7.41 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.17 – 7.05 (m, 3H), 6.97 – 6.89 (m, 1H), 5.65 (s, 1H), 5.50 (s, 1H), 4.55 – 4.46 (m, 1H), 4.47 – 4.41 (m, 1H), 4.15 – 4.04 (m, 2H), 3.91 – 3.80 (m, 2H), 3.71 – 3.61 (m, 2H), 3.60 – 3.53 (m, 2H), 3.11 (ddd, *J* = 14.7, 11.7, 2.9 Hz, 1H), 3.05 (ddd, *J* = 14.9, 11.7, 1.4 Hz, 1H), 2.37 – 2.24 (m, 2H), 2.12 – 1.99 (m, 4H), 1.97 – 1.87 (m, 2H), 1.11 – 1.02 (m, 9H), 1.01 – 0.93 (m, 6H), 0.92 – 0.79 (m, 9H), 0.67 – 0.50 (m, 6H);

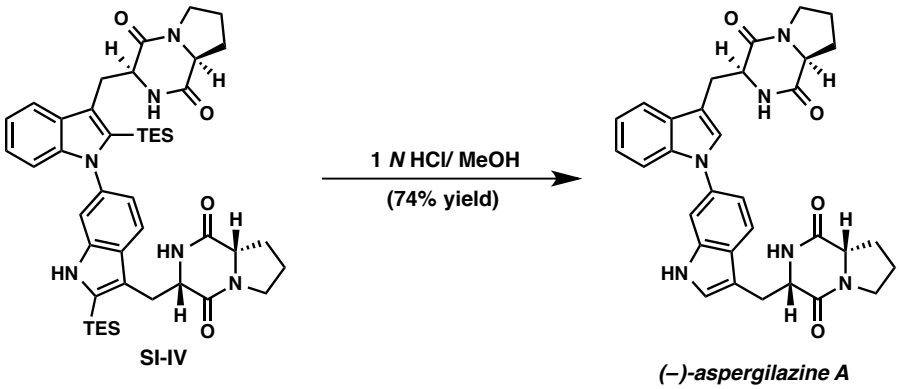
Note: The presence of rotomeric peaks complicates the analysis of the ^{13}C NMR spectra for this compound.
^{13}C NMR: (126 MHz, CD_2Cl_2) δ 169.6, 169.6, 166.0, 165.9, 165.8, 143.1, 143.0, 139.3, 139.3, 137.4, 137.3, 136.7, 136.6, 135.8, 135.8, 128.7, 128.5, 128.4, 128.4, 123.5, 123.5, 121.8, 121.7, 121.6, 121.6, 120.3, 120.3, 119.8, 119.7, 119.2, 118.5, 112.5, 112.4, 111.6, 111.6, 59.8, 59.7, 55.9, 55.8, 55.6, 54.9, 45.9, 45.8, 30.2, 29.0, 28.9, 28.2, 27.6, 23.2, 23.2, 23.1, 7.9, 7.9, 7.8, 4.9, 4.8, 4.3.

^{1}H NMR: (400 MHz, CD_3CN, 25 ºC) δ 9.53 (d, *J* = 6.9 Hz, 1H), 7.70 (dd, *J* = 8.4, 4.3 Hz, 1H), 7.67 – 7.61 (m, 1H), 7.48 (dd, *J* = 12.8, 1.7 Hz, 1H), 7.09 (qd, *J* = 5.9, 4.5, 1.9 Hz, 3H), 6.93 – 6.82 (m, 1H), 5.61 (d, *J* = 11.6 Hz, 1H), 5.50 (d, *J* = 18.5 Hz, 1H), 4.44 (tt, *J* = 10.9, 4.9 Hz, 2H), 4.12 (t, *J* = 7.6 Hz, 2H), 3.86 – 3.70 (m, 2H), 3.64 – 3.52 (m, 2H), 3.46 (dtd, *J* = 10.7, 7.2, 6.6, 3.1 Hz, 2H), 3.13 (ddd, *J* = 15.0, 11.4, 8.5 Hz, 1H), 3.04 (dd, *J* = 14.9, 11.0 Hz, 1H), 2.19 (s, 8H), 2.04 – 1.78 (m, 8H), 0.98 (m, 15H), 0.83 (m, 9H), 0.66 – 0.45 (m, 6H).

The ^{1}H NMR was found to coalesce in deuterated acetonitrile at 60 °C.
^{1}H NMR: (400 MHz, CD_3CN, 60 ºC) δ 9.40 (s, 1H), 7.72 (d, *J* = 8.3 Hz, 1H), 7.70 – 7.63 (m, 1H), 7.48 (s, 1H), 7.22 – 7.04 (m, 3H), 6.91 (s, 1H), 5.58 (s, 1H), 5.47 (s, 1H), 4.45 (t, *J* = 11.4 Hz, 2H), 4.13 (t, *J* = 7.7 Hz, 2H), 3.83 (d, *J* = 14.7 Hz, 1H), 3.76 (dd, *J* = 15.0, 4.2 Hz, 1H), 3.70 – 3.57 (m, 2H), 3.48 (ddd, *J* = 11.6, 8.1, 3.8 Hz, 2H), 3.18 (t, *J* = 13.1 Hz, 1H), 3.09 (dd, *J* = 14.9, 10.8 Hz, 1H), 2.29 – 2.16 (m, 2H), 2.02 – 1.81 (m, 6H), 1.23 – 0.94 (m, 15H), 0.88 (t, *J* = 7.7 Hz, 9H), 0.74 – 0.51 (m, 6H).

FTIR: (NaCl, thin film): cm^{-1}; 3375, 2963, 2859, 1671, 1446, 1414;
$[\alpha]_D^{25}$ = –79.5 (*c* = 0.055, 1:1 DCM:MeOH);
HRMS: (MM) calc'd $[M–SiC_6H_{15}]^+$ 679.3423, found 679.3426.

Synthesis of (–)-Aspergilazine A (10)



Silylated compound **SI-IV** (49.5 mg, 0.06 mmol, 1.0 equiv) was dissolved in 1 *N* HCl in MeOH (10 mL) and allowed to stir for 15 minutes. The reaction was quenched by addition of aqueous $NaHCO_3$ and diluted with EtOAc. The organics were removed *in vacuo* and the aqueous layer was extracted with EtOAc (3 X 20 mL). The organics were combined, dried over Na_2SO_4, filtered, and concentrated. The crude residue was purified by silica gel chromatography (5% MeOH, 95% CH_2Cl_2) to provide (–)-aspergilazine A as a colorless solid (26.0 mg, 74% yield).

Spectroscopic and physical data, including ^1H, ^{13}C NMR in DMSO-*d6,* IR, and MS obtained for (–)-aspergilazine A matched that as reported during isolation by Gu et. al[7] and data obtained by Sperry and co-workers.[8] See below ^1H and ^{13}C NMR data comparison.

The sign of the optical rotation by Gu et. al was subsequently corrected by the authors from (+)- to (–)-aspergilazine A.[9] The initially reported rotation of "$[\alpha]_D^{25}$ = 72.0" was revised to "$[\alpha]_D^{25}$ = –72.0." Studies toward aspergilazine A by Boyd and Sperry[8] confirm the negative sign value (reported rotation, $[\alpha]_D^{25}$ = –70.0). The obtained synthetic material in this report was of slightly greater magnitude $[\alpha]_D^{25}$ = –90.6 (*c* = 0.625, 1:1 CH_2Cl_2:MeOH), but of the same sign.

^{1}H NMR: (500 MHz, DMSO-d_6) δ 11.05 (s, 1H), 7.99 (s, 1H), 7.87 (s, 1H), 7.74 (d, J = 8.4 Hz, 1H), 7.67 (d, J = 7.9 Hz, 1H), 7.47 (d, J = 8.3 Hz, 1H), 7.46 (s, 1H), 7.44 (d, J = 1.9 Hz, 1H), 7.28 (d, J = 2.3 Hz, 1H), 7.16 (t, *J* = 7.6 Hz, 1H), 7.14 (dd, *J* = 8.2, 1.7 Hz, 1H), 7.09 (t, *J* = 7.4 Hz, 1H), 4.40 (t, *J* = 4.8 Hz, 1H), 4.36 (t, *J* = 5.2 Hz, 1H), 4.16 – 4.00 (m, 2H), 3.46 – 3.29 (m, 3H), 3.33 – 3.20 (m, 3H), 3.15 (dd, *J* = 14.9, 5.7 Hz, 2H), 2.05 – 1.89 (m, 2H), 1.82 – 1.51 (m, 4H), 1.47 – 1.31 (m, 2H);

[7] Cai, S.; Kong, X.; Wang, W.; Zhou, H.; Zhu, T.; Li, D.; Gu, Q. *Tetrahedron Lett.* **2012**, *53*, 2615.
[8] Boyd, E. M.; Sperry, J. *Org. Lett.* **2014**, *16*, 5056.
[9] Cai, S.; Kong, X.; Wang, W.; Zhou, H.; Zhu, T.; Li, D.; Gu, Q. *Tetrahedron Lett.* **2014,** *55*, 5404.

^{13}C NMR: (126 MHz, DMSO-d_6) δ 169.6, 169.6, 166.0, 165.9, 136.6, 136.1, 133.5, 129.0, 128.8, 126.6, 126.2, 122.5, 120.2, 119.9, 115.7, 111.5, 110.6, 110.2, 107.2, 59.0, 55.8, 55.7, 45.1, 28.2, 28.2, 26.3, 26.3, 22.4, 22.4;

FTIR: (NaCl, thin film): cm^{-1}; 3365, 3246, 2933, 1666, 1459, 1414;

$[\alpha]_D^{25}$ = –90.6 (*c* = 0.625, 1:1 CH_2Cl_2:MeOH);

HRMS: (MM) calc'd $[M+H]^+$ 565.2558, found 565.2555.

^{1}H NMR: (500 MHz, **CD_2Cl_2**) δ 8.71 (s, 1H), 7.82 – 7.68 (m, 1H), 7.56 (d, *J* = 8.3 Hz, 1H), 7.50 – 7.40 (m, 1H), 7.33 (s, 1H), 7.23 (s, 1H), 7.21 – 7.07 (m, 4H), 6.82 (s, 1H), 5.97 (s, 1H), 4.54 – 4.39 (m, 1H), 4.39 – 4.24 (m, 1H), 4.03 (dt, *J* = 13.2, 7.5 Hz, 2H), 3.74 – 3.46 (m, 4H), 3.40 (tt, *J* = 7.8, 3.2 Hz, 1H), 3.29 (dt, *J* = 10.9, 7.7 Hz, 1H), 3.20 (dd, *J* = 15.1, 8.8 Hz, 1H), 3.00 (dd, *J* = 15.1, 9.9 Hz, 1H), 2.23 (dt, *J* = 9.5, 6.6 Hz, 1H), 2.20 – 2.14 (m, 1H), 1.97 – 1.79 (m, 2H), 1.78 – 1.67 (m, 2H).

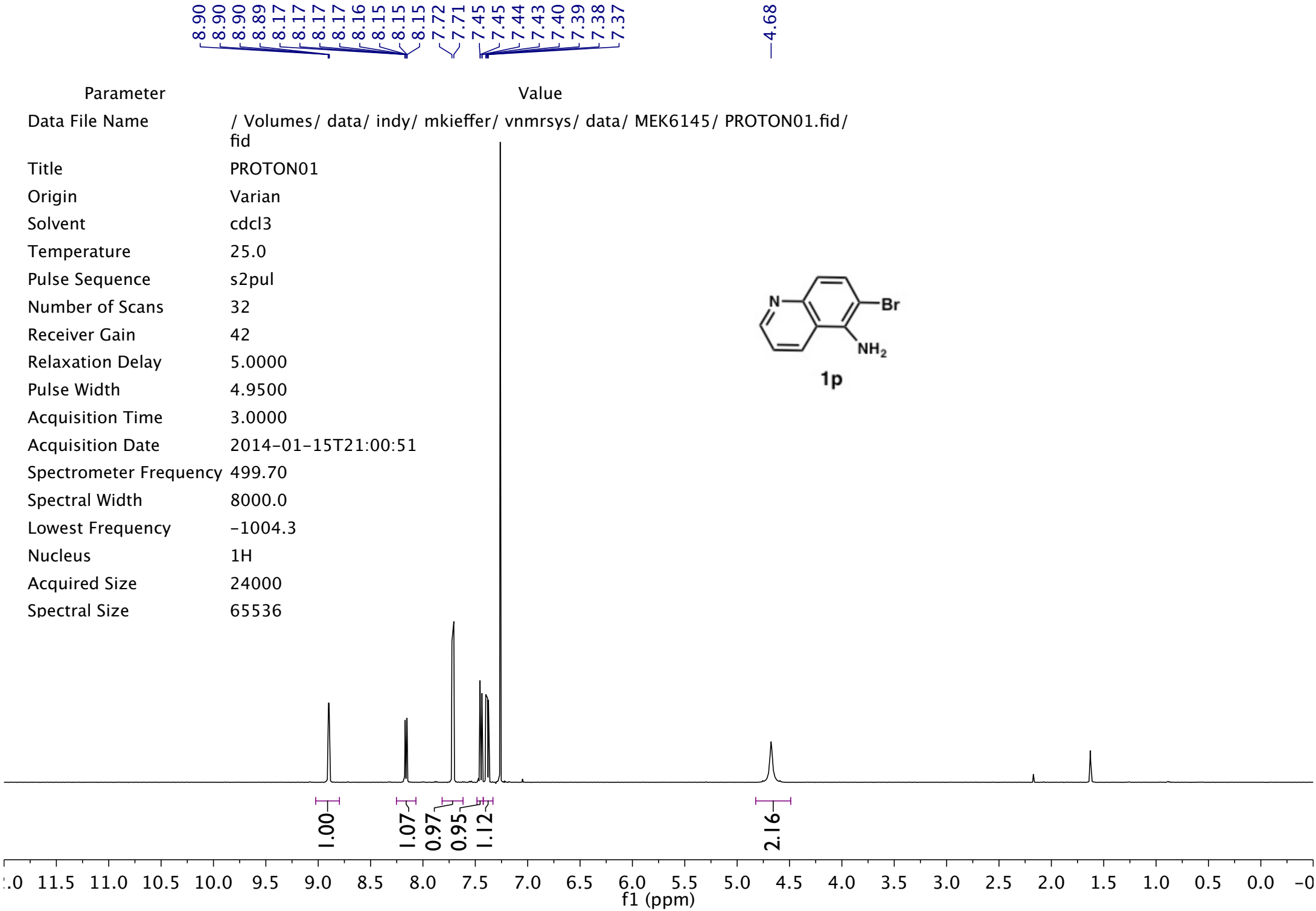
^{13}C NMR: (126 MHz, **CD_2Cl_2**) δ 170.5, 170.0, 165.9, 165.9, 137.3, 137.2, 134.8, 128.9, 128.8, 126.2, 125.5, 123.2, 120.6, 119.8, 119.8, 117.4, 111.4, 111.0, 110.4, 107.9, 59.7, 55.5, 55.2, 45.8, 45.6, 28.8, 28.7, 27.0, 26.8, 23.1, 22.9.

Table S1. Comparison of ^{1}H NMR data for Natural vs. Synthetic (-)-Aspergilazine A

Isolation[7] **(–)-Aspergilazine A** **^{1}H NMR, 600 MHz, DMSO-d_6**	**This Work** **(–)-Aspergilazine A** **^{1}H NMR, 500 MHz, DMSO-d_6**
11.09 (s, 1H)	11.05 (s, 1H)
8.00 (s, 1H)	7.99 (s, 1H)
7.89 (s, 1H)	7.87 (s, 1H)
7.75 (br d, J = 8.4 Hz, 1H)	7.74 (d, J = 8.4 Hz, 1H)
7.68 (br d, J = 7.8 Hz, 1H)	7.67 (d, J = 7.9 Hz, 1H)
7.48 (d, J = 8.2 Hz, 1H)	7.47 (d, J = 8.3 Hz, 1H)
7.47 (s, 1H)	7.46 (s, 1H)
7.45 (d, J = 1.9 Hz, 1H)	7.44 (d, J = 1.9 Hz, 1H)
7.29 (d, J = 1.7 Hz, 1H)	7.28 (d, J = 2.3 Hz, 1H)
7.16 (ddd, J = 7.7, 7.4, 1.0 1H)	7.16 (t, J = 7.6 Hz, 1H)
7.14 (dd, J = 8.3, 1.9, 1H)	7.14 (dd, J = 8.2, 1.7 Hz, 1H)
7.09 (ddd, J = 7.4, 7.4, 0.8 1H)	7.09 (t, J = 7.4 Hz, 1H)
4.41 (dd, J = 4.9, 5.0 Hz, 1H)	4.40 (t, J = 4.8 Hz, 1H)
4.37 (dd, J = 5.0, 5.0 Hz, 1H)	4.36 (t, J = 5.2 Hz, 1H)
4.07 (dd, J = 8.3, 8.3 Hz, 2H)	4.16 – 4.00 (m, 2H)
3.38 (m, 3H)	3.46 – 3.29 (m, 3H)
3.26 (m, 3H)	3.33 – 3.20 (m, 3H)
3.16 (m, 2H)	3.15 (dd, J = 14.9, 5.7 Hz, 2H)
1.98 (m, 2H)	2.05 – 1.89 (m, 2H)
1.65 (m, 4H)	1.82 – 1.51 (m, 4H)
1.37 (m, 2H)	1.47 – 1.31 (m, 2H)

Table S2. Comparison of ^{13}C NMR data for Natural vs. Synthetic (–)-Aspergilazine A

Isolation[7] (–)-Aspergilazine A ^{13}C NMR, 150 MHz, DMSO	This Work (–)-Aspergilazine A ^{13}C NMR, 126 MHz, DMSO	Chemical Shift Difference, Δδ
169.7	169.6	0.1
169.6	169.6	0.0
166.0	166.0	0.0
165.9	165.9	0.0
136.7	136.6	0.1
136.2	136.1	0.1
133.6	133.5	0.1
129.1	129.0	0.1
128.9	128.8	0.1
126.6	126.6	0.0
126.3	126.2	0.1
122.6	122.5	0.1
120.3	120.2	0.1
119.9	119.9	0.0
115.8	115.7	0.1
111.5	111.5	0.0
110.6	110.6	0.0
110.3	110.2	0.1
107.2	107.2	0.0
59.0	59.0	0.0
59.0	59.0	0.0
55.8	55.8	0.0
55.7	55.7	0.0
45.2	45.1	0.1
45.2	45.1	0.1
28.3	28.2	0.1
28.3	28.2	0.1
26.4	26.3	0.1
26.3	26.3	0.0
22.4	22.4	0.0
22.4	22.4	0.0


8.90
8.90
8.90
8.89
8.17
8.17
8.17
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8.15
8.15
8.15
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7.71
7.45
7.45
7.44
7.43
7.40
7.39
7.38
7.37
4.68
Parameter
Value
Data File Name
/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6145/ PROTON01.fid/ fid
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PROTON01
Origin
Varian
Solvent
cdcl3
Temperature
25.0
Pulse Sequence
s2pul
Number of Scans
32
Receiver Gain
42
Relaxation Delay
5.0000
Pulse Width
4.9500
Acquisition Time
3.0000
Acquisition Date
2014-01-15T21:00:51
Spectrometer Frequency
499.70
Spectral Width
8000.0
Lowest Frequency
-1004.3
Nucleus
1H
Acquired Size
24000
Spectral Size
65536
N
Br
NH2
1p
1.00
1.07
0.97
0.95
1.12
2.16
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

Parameter	Value
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Receiver Gain	30
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Acquisition Time	1.0420
Acquisition Date	2014-01-15T21:05:23
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Nucleus	13C
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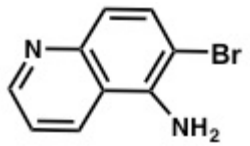
Spectral Size	65536



1p

Parameter	Value
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Solvent	cdcl3
Temperature	25.0
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Receiver Gain	22
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
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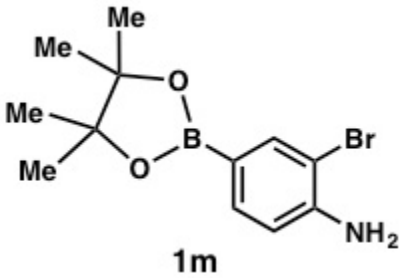

1m

Parameter	Value
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Solvent	cdcl3
Temperature	25.0
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Number of Scans	512
Receiver Gain	30
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Pulse Width	4.2500
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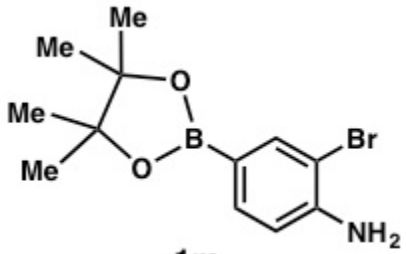
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1901.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



1m

Parameter	Value
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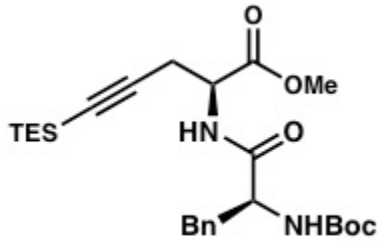
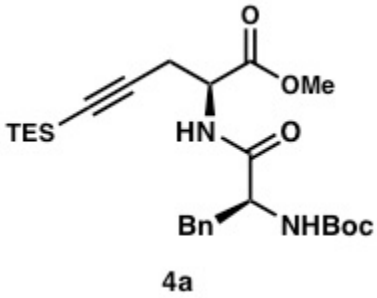
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	0
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2013-09-03T14:37:23
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1003.8
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



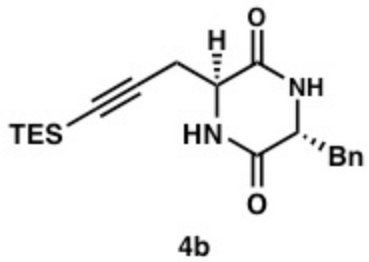
4a

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6031/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	256
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2013-09-03T14:41:55
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1901.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



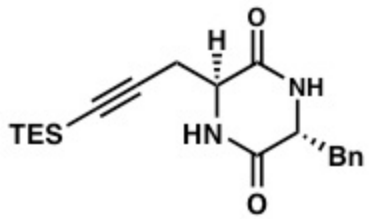
4a

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ kangway/ vnmrsys/ data/ KVC16-065/ PROTON02.fid/ fid
Title	PROTON02
Origin	Varian
Solvent	dmso
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	32
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-02-02T23:06:12
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-996.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536

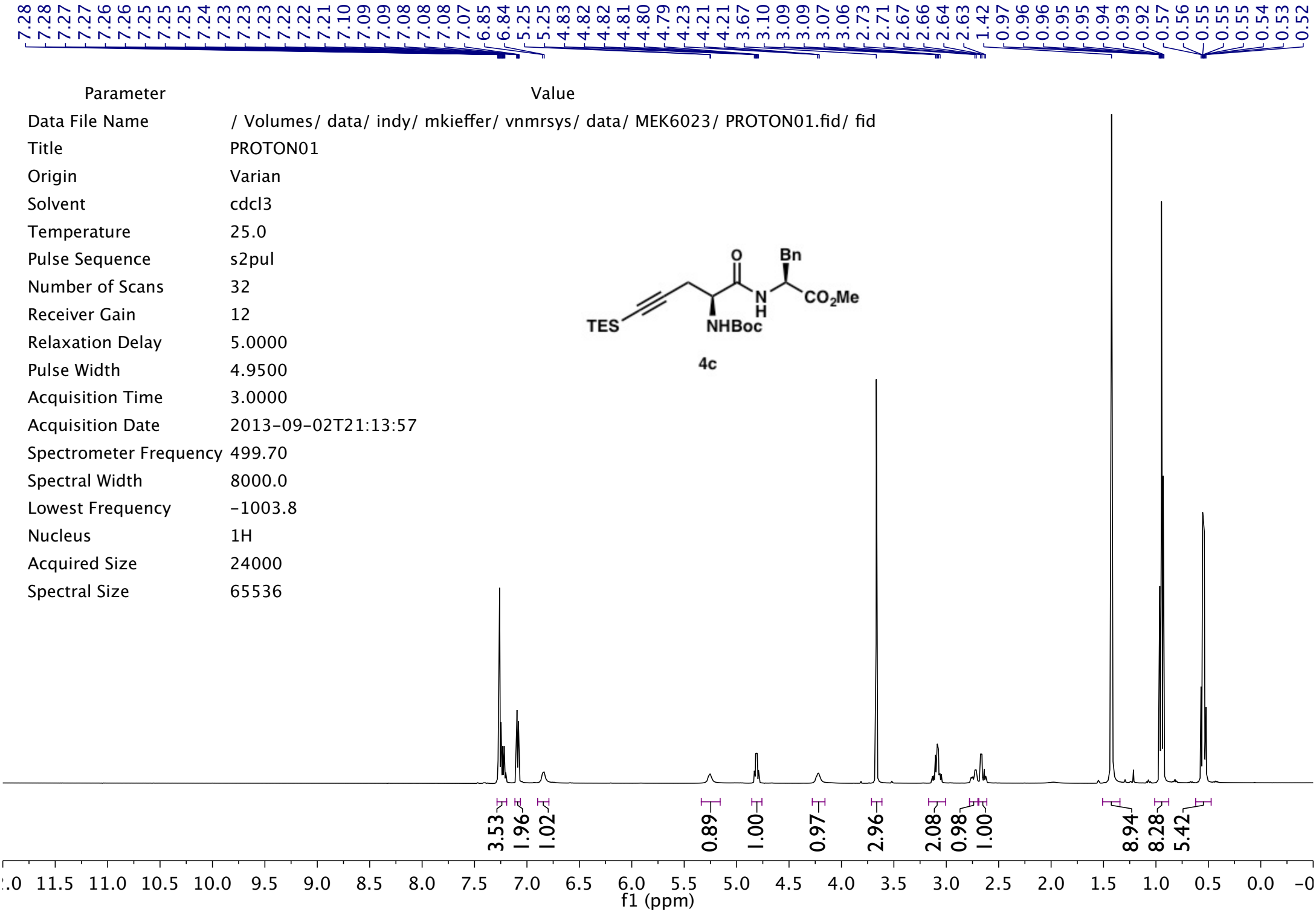


4b

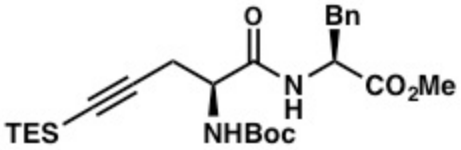
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ kangway/ vnmrsys/ data/ KVC16-065/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	dmso
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	512
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-02-02T23:10:51
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1958.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



4b


7.28 7.28 7.27 7.27 7.26 7.26 7.25 7.25 7.25 7.24 7.23 7.23 7.23 7.22 7.22 7.21 7.10 7.09 7.09 7.08 7.08 7.08 7.07 6.85 6.84 5.25 5.25 4.83 4.82 4.82 4.81 4.80 4.79 4.23 4.21 4.21 3.67 3.10 3.09 3.09 3.07 3.06 2.73 2.71 2.67 2.66 2.64 2.63 1.42 0.97 0.96 0.96 0.95 0.95 0.94 0.93 0.92 0.57 0.56 0.55 0.55 0.55 0.54 0.53 0.52
Parameter
Value
Data File Name / Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6023/ PROTON01.fid/ fid
Title PROTON01
Origin Varian
Solvent cdcl3
Temperature 25.0
Pulse Sequence s2pul
Number of Scans 32
Receiver Gain 12
Relaxation Delay 5.0000
Pulse Width 4.9500
Acquisition Time 3.0000
Acquisition Date 2013-09-02T21:13:57
Spectrometer Frequency 499.70
Spectral Width 8000.0
Lowest Frequency -1003.8
Nucleus 1H
Acquired Size 24000
Spectral Size 65536
O
Bn
N
H
CO2Me
TES
NHBoc
4c
3.53 1.96 1.02 0.89 1.00 0.97 2.96 2.08 0.98 1.00 8.94 8.28 5.42
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

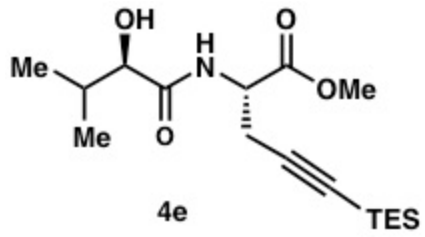
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6023/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	256
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2013-09-02T21:18:28
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1891.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



4c

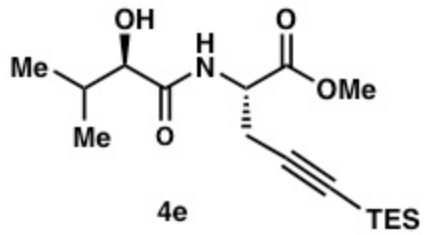
7.44 7.42 4.61 4.60 4.59 4.58 4.58 4.57 3.90 3.90 3.89 3.88 3.72 3.71 3.69 2.83 2.82 2.79 2.78 2.75 2.74 2.72 2.71 2.10 2.09 2.08 2.08 2.07 2.06 2.05 2.05 2.04 2.03 1.95 1.94 1.94 1.93 1.93 0.99 0.98 0.97 0.97 0.96 0.95 0.94 0.83 0.81 0.60 0.59 0.58 0.57 0.57 0.56 0.56 0.55 0.55 0.54 0.53 0.53

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6221/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	16
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-07-11T03:02:01
Spectrometer Frequency	499.69
Spectral Width	8000.0
Lowest Frequency	-1019.8
Nucleus	1H
Acquired Size	24000
Spectral Size	65536

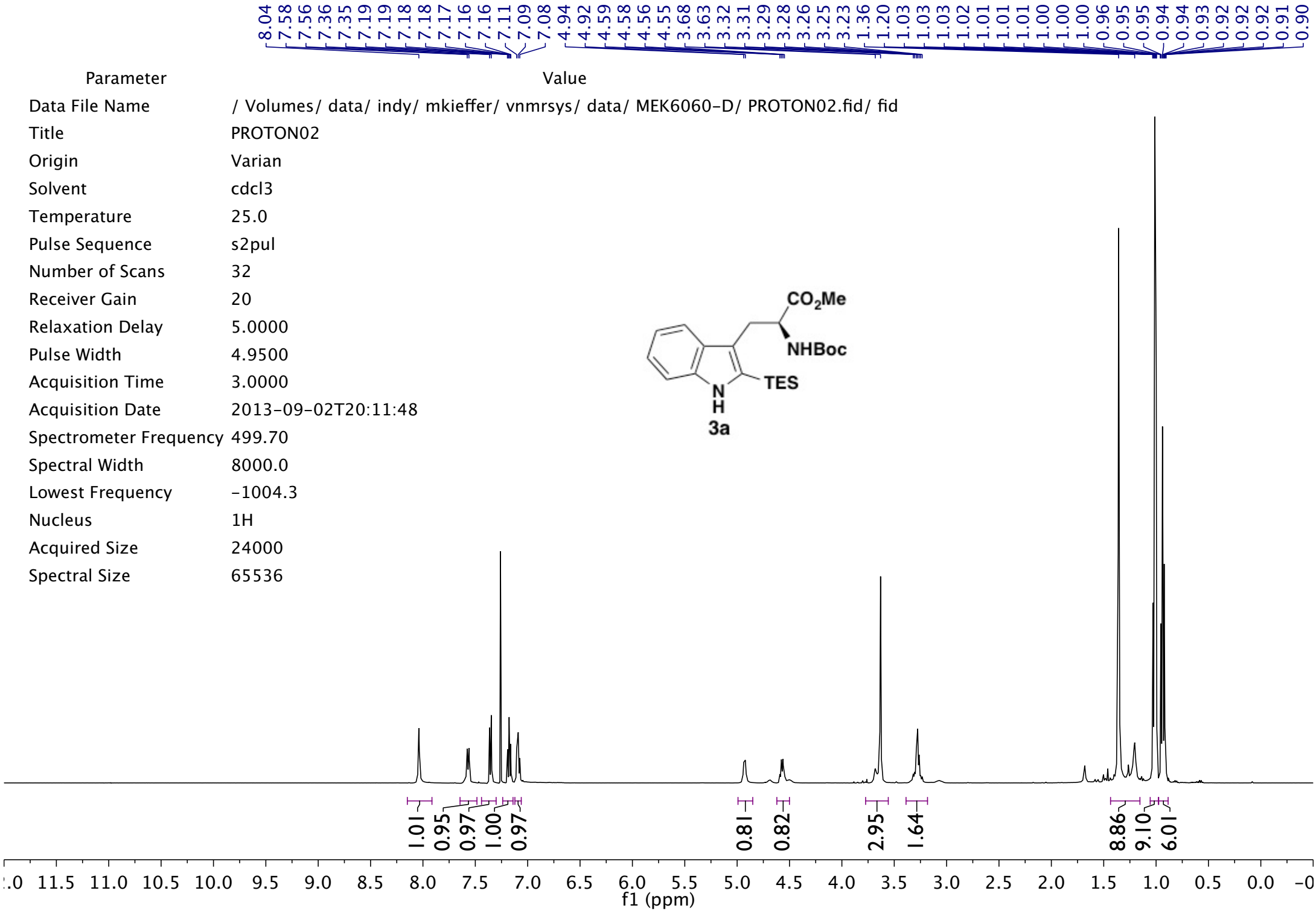


4e

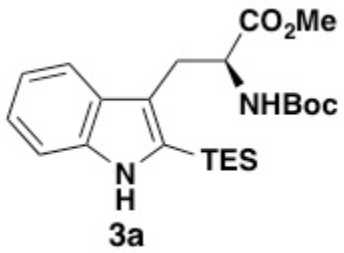
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6221/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	2000
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-07-11T03:06:40
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1781.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



4e


8.04 7.58 7.56 7.36 7.35 7.19 7.19 7.18 7.18 7.17 7.16 7.16 7.11 7.09 7.08
4.94 4.92 4.59 4.58 4.56 4.55 3.68 3.63 3.32 3.31 3.29 3.28 3.26 3.25 3.23 1.36 1.20 1.03 1.03 1.03 1.02 1.01 1.01 1.01 1.00 1.00 1.00 0.96 0.95 0.95 0.94 0.94 0.93 0.92 0.92 0.92 0.91 0.90
Parameter
Value
Data File Name / Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6060-D/ PROTON02.fid/ fid
Title PROTON02
Origin Varian
Solvent cdcl3
Temperature 25.0
Pulse Sequence s2pul
Number of Scans 32
Receiver Gain 20
Relaxation Delay 5.0000
Pulse Width 4.9500
Acquisition Time 3.0000
Acquisition Date 2013-09-02T20:11:48
Spectrometer Frequency 499.70
Spectral Width 8000.0
Lowest Frequency -1004.3
Nucleus 1H
Acquired Size 24000
Spectral Size 65536
CO2Me
NHBoc
TES
N
H
3a
1.01 0.95 0.97 1.00 0.97 0.81 0.82 2.95 1.64 8.86 9.10 6.01
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

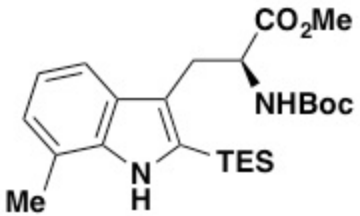
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6060-D/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	256
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2013-09-02T20:16:19
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1887.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



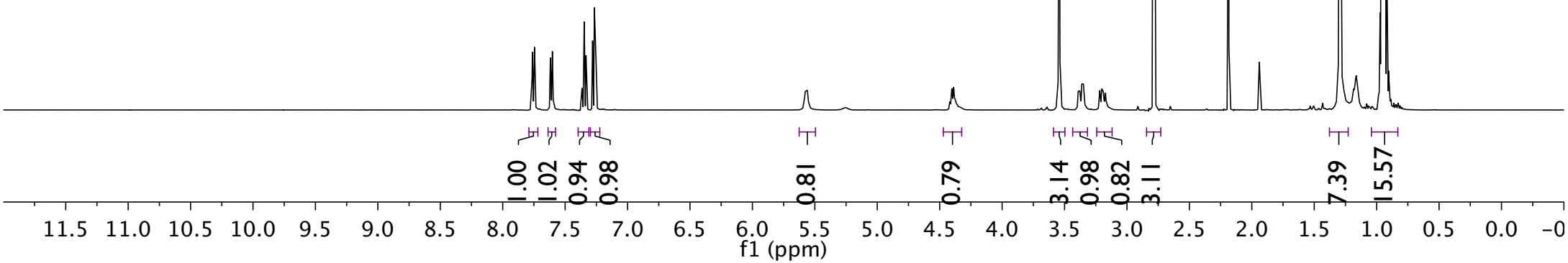
3a

7.76 7.75 7.62 7.62 7.60 7.60 7.36 7.36 7.35 7.35 7.34 7.33 7.33 7.28 7.28 7.27 7.27 7.26 7.25 7.25 5.58 5.56 4.42 4.40 4.39 4.37 3.54 3.39 3.38 3.36 3.35 3.22 3.20 3.19 3.17 2.78 2.78 1.29 1.28 0.98 0.98 0.97 0.97 0.96 0.96 0.95 0.95 0.94 0.94 0.94 0.94 0.93 0.93 0.92 0.92 0.91 0.91 0.91 0.90 0.90 0.90 0.89 0.89

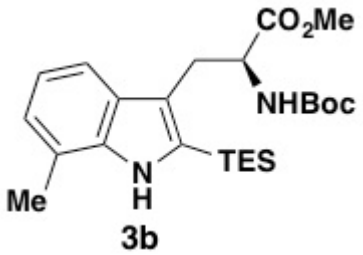
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6140-B/ PROTON02.fid/ fid
Title	PROTON02
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	8
Receiver Gain	16
Relaxation Delay	1.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-01-12T19:04:14
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1016.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



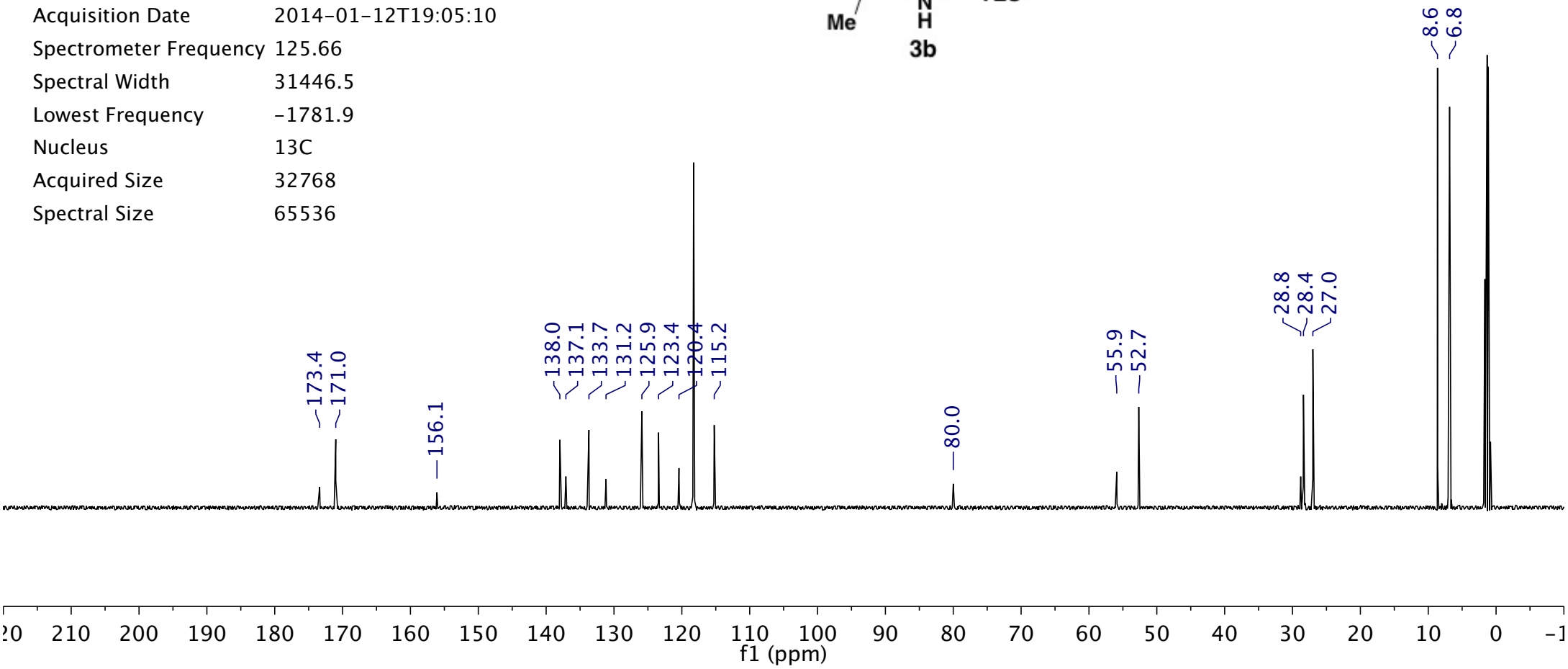
3b



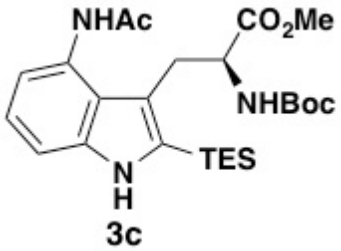
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6140-B/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	512
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-01-12T19:05:10
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1781.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



3b

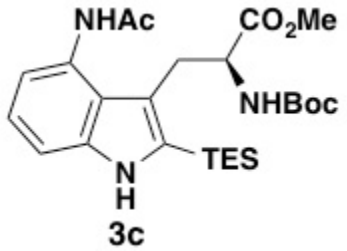


Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6266-A/ PROTON02.fid/ fid
Title	PROTON02
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	24
Relaxation Delay	5.0000
Pulse Width	5.9000
Acquisition Time	3.0000
Acquisition Date	2015-03-01T13:44:05
Spectrometer Frequency	499.68
Spectral Width	8000.0
Lowest Frequency	-1039.5
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



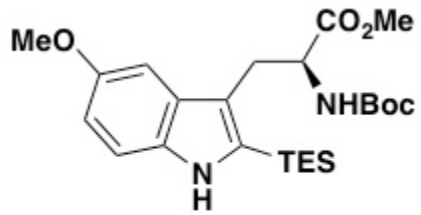
3c

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6266-A/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	2000
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-07-11T05:48:55
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1786.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



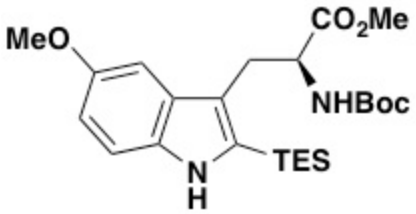
3c

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6150-A/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	22
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-01-31T15:48:34
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1016.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



3d

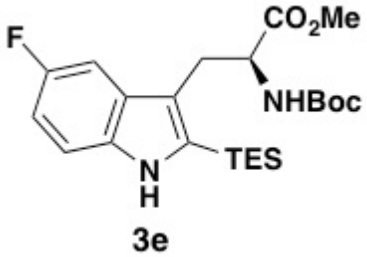
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6150-A/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	1000
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-01-31T15:53:13
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1784.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



3d

8.01 7.25 7.24 7.23 7.20 7.19 7.18 7.18 7.18 7.17 7.17 7.16 6.93 6.93 6.92 6.91 6.90 6.89 4.94 4.92 4.56 4.54 4.53 4.51 3.70 3.69 3.68 3.65 3.63 3.26 3.24 3.23 3.22 3.21 3.20 3.19 3.17 1.35 1.25 1.22 1.21 1.03 1.02 1.02 1.02 1.01 1.00 1.00 1.00 0.99 0.99 0.99 0.98 0.94 0.94 0.93 0.92 0.92 0.91 0.91 0.90 0.90 0.89

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6060-C/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	22
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2013-09-02T20:29:37
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1004.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



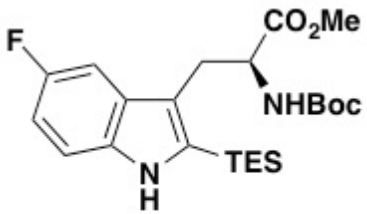
3e

1.00 1.03 0.93 1.04 0.80 0.78 2.82 1.73 8.54 8.39 5.94

.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

f1 (ppm)

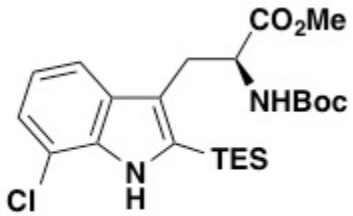
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6060-C/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	512
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2013-09-02T20:34:09
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1884.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



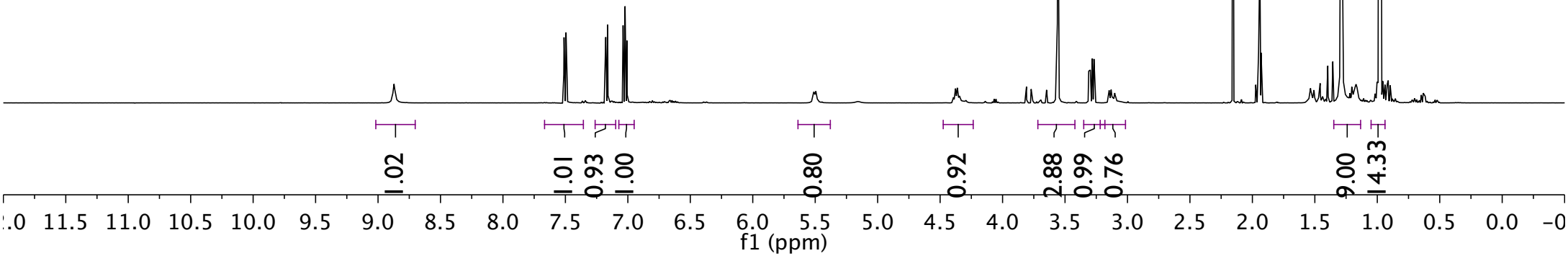
3e

8.87 7.51 7.51 7.51 7.50 7.50 7.49 7.18 7.18 7.16 7.16 7.04 7.02 7.01 5.51 5.49 5.45 4.46 4.39 4.38 4.36 4.35 4.29 3.56 3.31 3.30 3.28 3.27 3.15 3.13 3.12 3.10 1.33 1.29 1.27 1.25 1.22 1.20 1.19 1.17 1.16 1.02 1.02 1.01 1.01 1.01 1.01 1.00 1.00 1.00 0.99 0.98 0.98 0.98 0.97 0.97 0.96 0.96 0.96 0.95 0.95 0.94 0.94

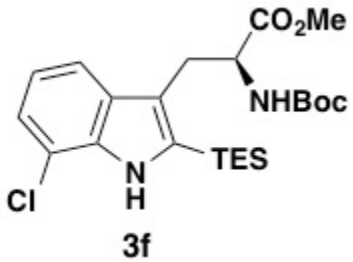
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK-7-chloro/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	26
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-07-24T18:56:24
Spectrometer Frequency	499.69
Spectral Width	8000.0
Lowest Frequency	-1019.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



3f

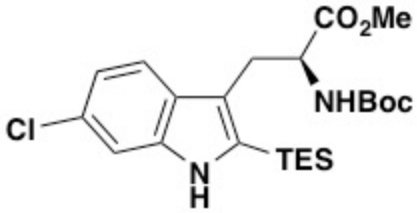


Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK-7-chloro/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	1000
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-07-24T19:00:55
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1785.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



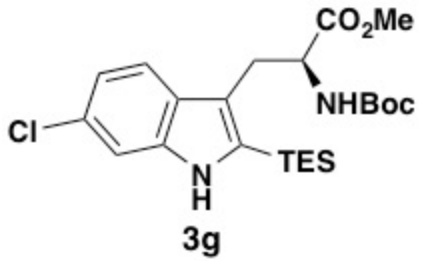
3f

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6060-B/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	24
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2013-09-02T20:56:08
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1004.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



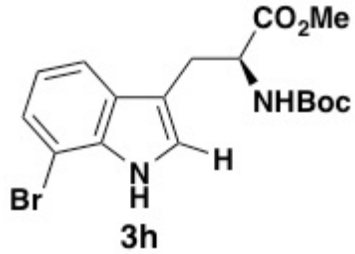
3g

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6060-B/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	256
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2013-09-02T21:00:40
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1884.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



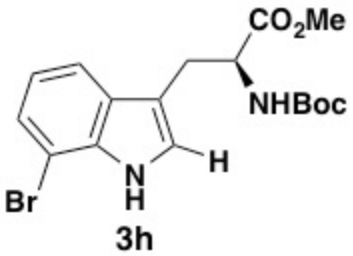
3g

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK-7-bromo-TBAF/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	34
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-09-09T13:44:59
Spectrometer Frequency	499.69
Spectral Width	8000.0
Lowest Frequency	-1019.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



3h

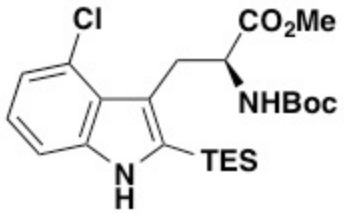
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK-7-bromo-TBAF/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	1500
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-09-09T13:49:30
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1785.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



3h

9.29 7.39 7.38 7.37 7.37 7.07 7.06 7.04 7.03 7.03 7.02 7.01 5.41 5.40 4.55 4.54 4.53 4.53 4.52 4.51 4.50 3.65 3.61 3.57 3.55 3.54 3.53 3.52 3.28 3.27 3.24 3.22 3.21 3.19 1.27 1.23 1.20 1.19 1.00 1.00 1.00 0.99 0.99 0.98 0.97 0.97 0.97 0.96 0.96 0.95 0.95 0.94 0.93 0.93 0.92 0.92 0.91 0.91 0.90 0.90 0.89 0.88

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK-4-chlor/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	28
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2013-11-06T18:42:23
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1016.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



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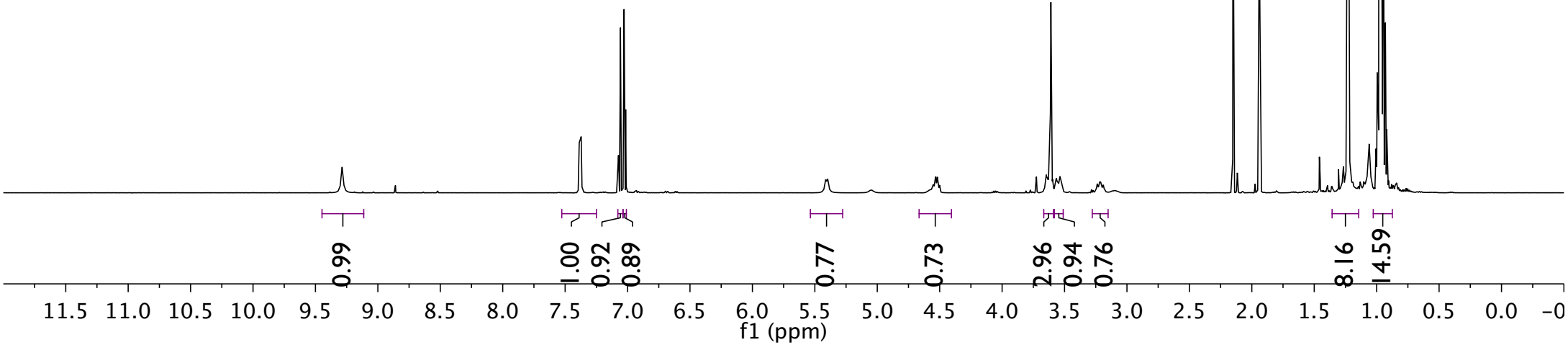
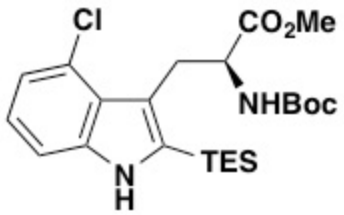
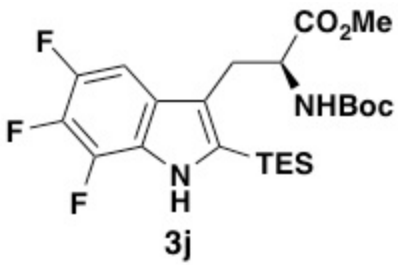


Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK-4-chlor/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	512
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2013-11-06T18:47:01
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1783.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



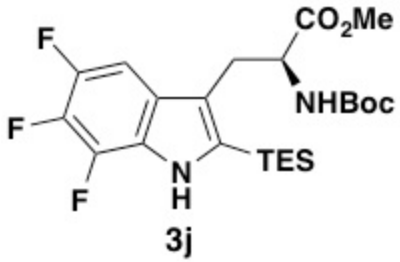
3i

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ kangway/ vnmrsys/ data/ MEK6150-C/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	30
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-02-03T03:25:19
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1016.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



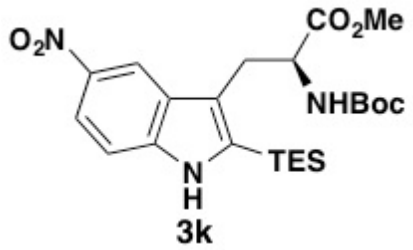
3j

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ kangway/ vnmrsys/ data/ MEK6150-C/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	8000
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-02-03T03:29:58
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1782.8
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



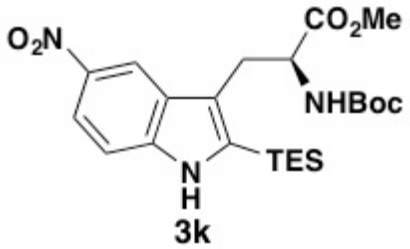
3j

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK-nitro/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	30
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-01-20T17:21:38
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1016.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



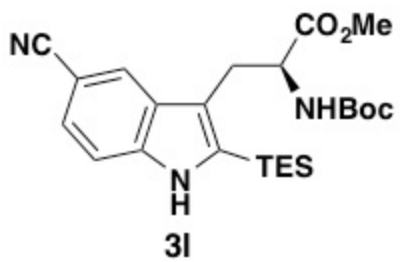
3k

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK-nitro/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	512
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-01-20T17:26:17
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1782.8
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



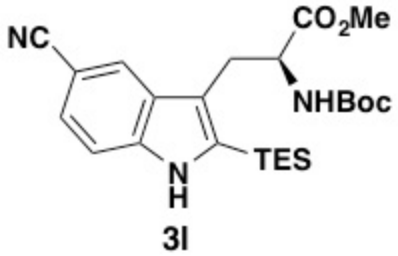
3k

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ kangway/ vnmrsys/ data/ MEK6122/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	32
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-02-03T01:30:52
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1016.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



3l

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ kangway/ vnmrsys/ data/ MEK6122/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	3000
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-02-03T01:35:31
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1783.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



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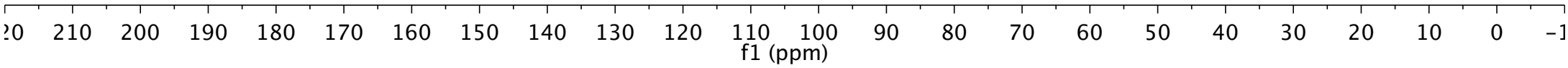
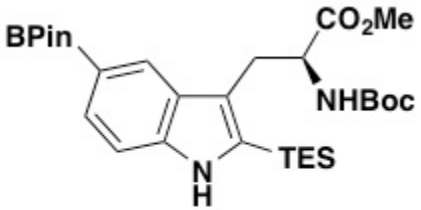
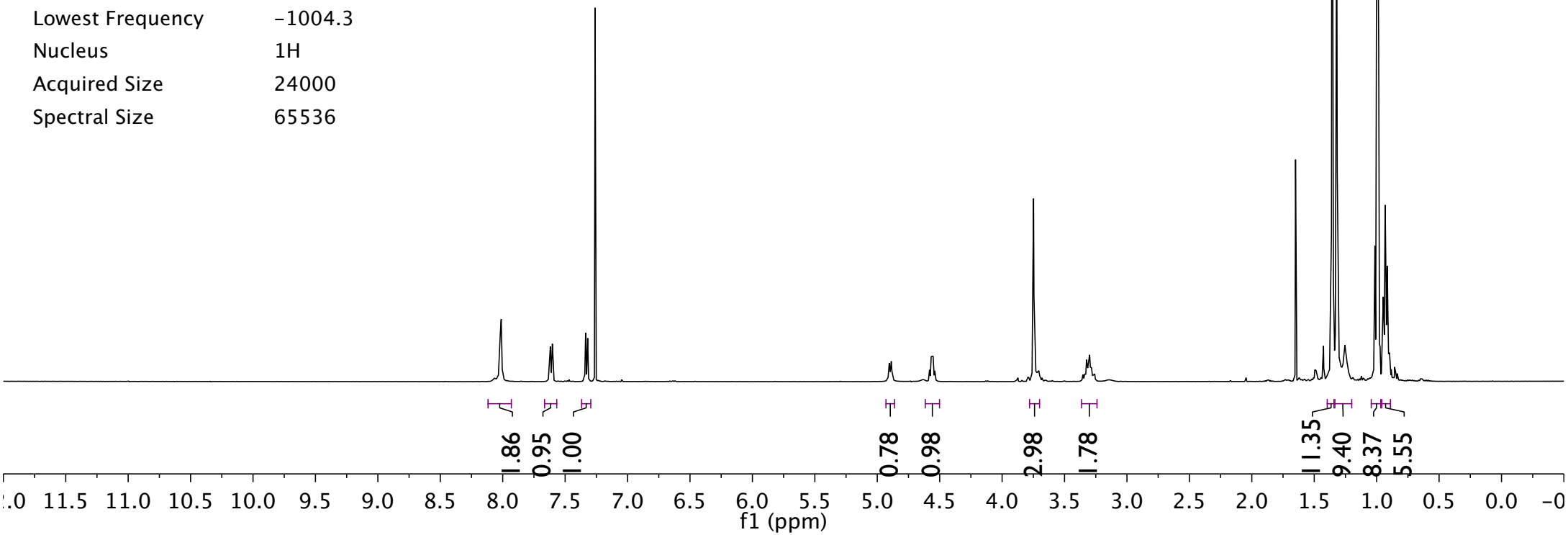


8.10 8.08 8.07 8.06 8.02 8.01 7.98 7.97 7.96 7.96 7.95 7.62 7.60 7.34 7.32 4.90 4.89 4.58 4.57 4.57 4.55 4.54 3.75 3.73 3.71 3.71 3.71 3.35 3.34 3.32 3.31 3.30 3.29 3.27 3.26 1.36 1.35 1.32 1.25 1.24 1.23 1.02 1.01 1.01 1.00 1.00 1.00 0.99 0.99 0.98 0.98 0.97 0.95 0.94 0.93 0.93 0.92 0.92 0.91

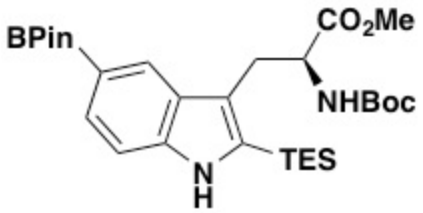
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6131/ PROTON02.fid/ fid
Title	PROTON02
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	26
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2013-12-18T05:59:15
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1004.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



3m

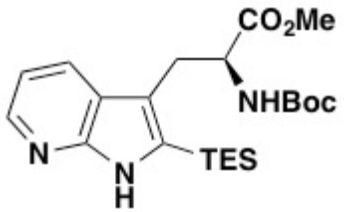


Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6131/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	512
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2013-12-18T06:03:46
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1883.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



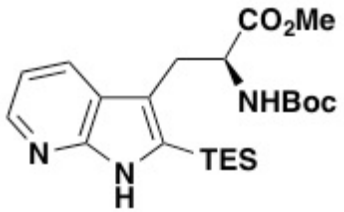
3m

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6060-A/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	16
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2013-09-03T14:11:07
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1004.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



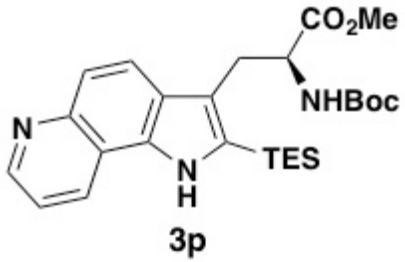
3n

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6060-A/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	512
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2013-09-03T14:15:38
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1887.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



3n

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6146/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	26
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-01-22T14:02:41
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1016.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



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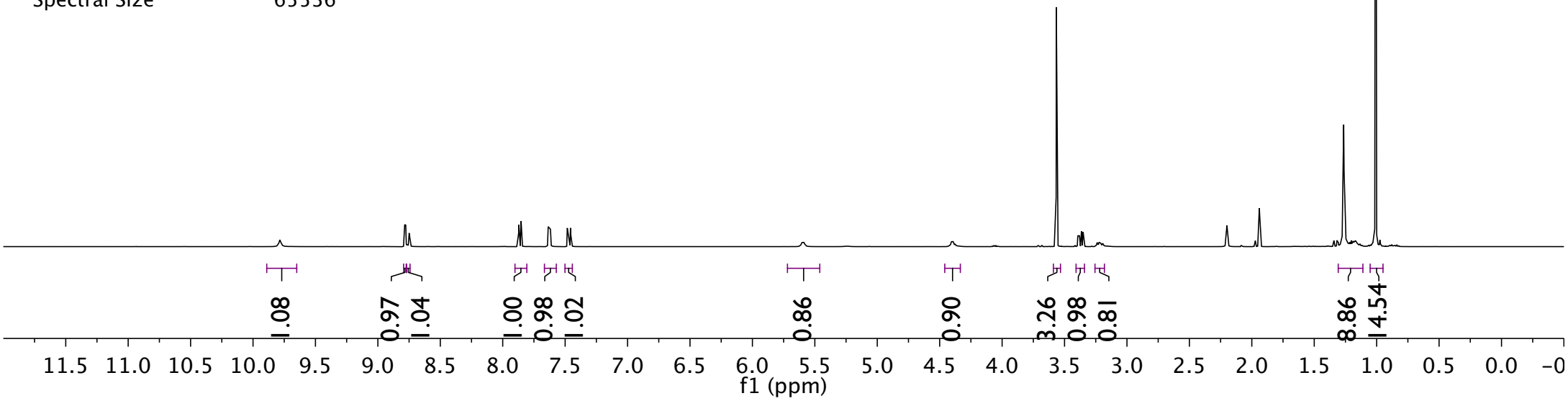
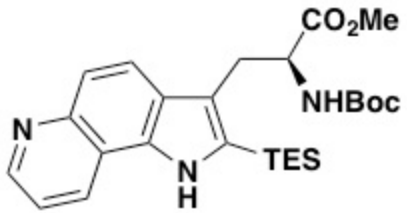


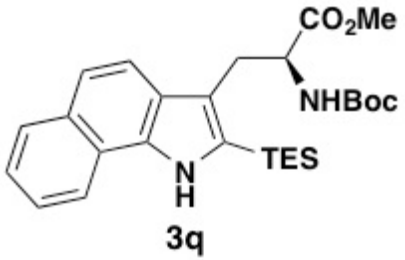
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6146/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	1000
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-01-22T14:07:19
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1783.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



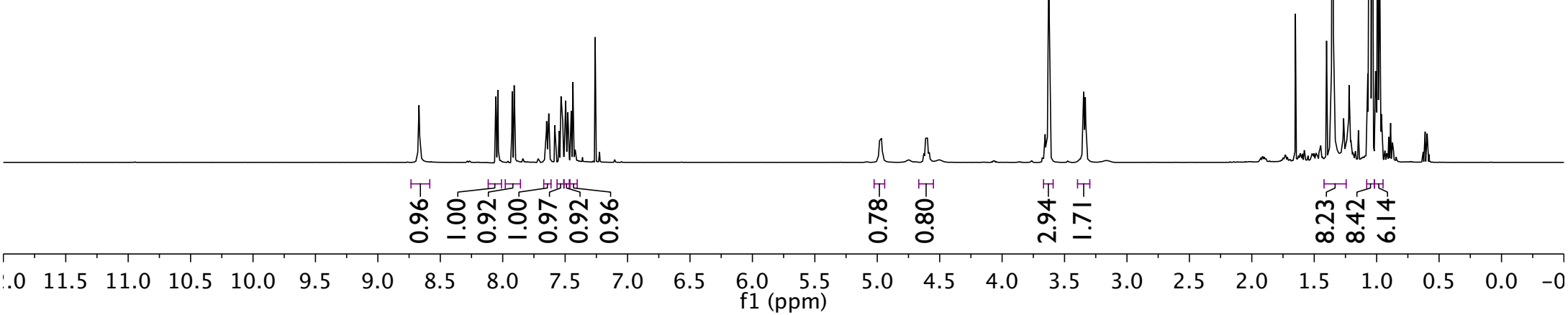
3p

8.67 8.06 8.05 8.04 8.04 7.93 7.92 7.91 7.91 7.65 7.63 7.55 7.55 7.54 7.53 7.53 7.52 7.52 7.52 7.50 7.48 7.45 7.45 7.44 7.44 7.43 7.42 7.42 4.98 4.97 4.61 4.60 3.65 3.62 3.35 3.33 1.40 1.38 1.35 1.26 1.08 1.07 1.07 1.06 1.06 1.05 1.05 1.04 1.04 1.04 1.03 1.02 1.02 1.01 1.00 0.99 0.99 0.98 0.98 0.98 0.98 0.97 0.96 0.96 0.96

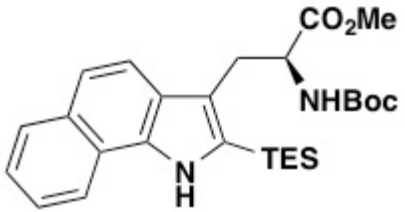
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6067-A/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	22
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2013-09-30T22:32:31
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1004.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



3q

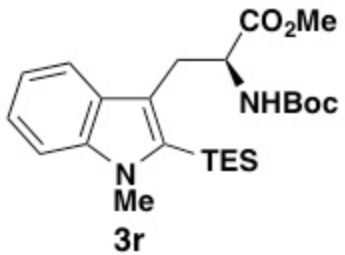


Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6067-A/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	512
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2013-09-30T22:37:02
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1885.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



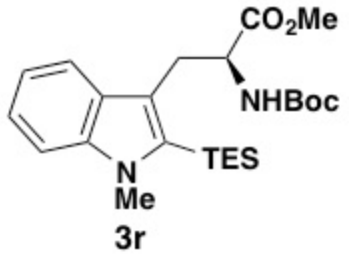
3q

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ kangway/ vnmrsys/ data/ MEK6140-A/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	28
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-02-02T23:36:25
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1016.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



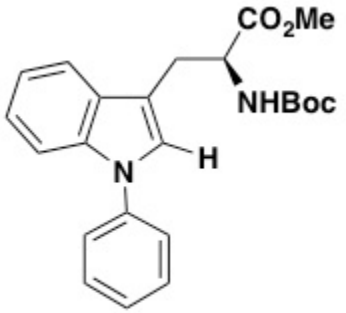
3r

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ kangway/ vnmrsys/ data/ MEK6140-A/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	3000
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-02-02T23:41:03
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1782.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



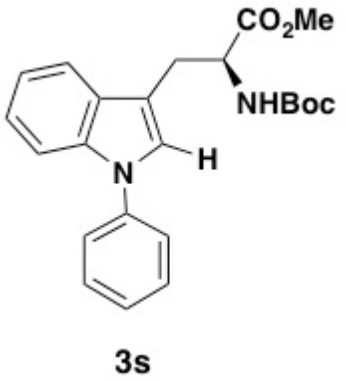
3r

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ mkieffer/ vnmrsys/ data/ MEK-N-PH-TBAF/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	16
Receiver Gain	26
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-08-15T14:39:36
Spectrometer Frequency	499.69
Spectral Width	8000.0
Lowest Frequency	-1019.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



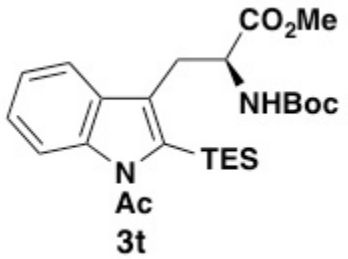
3s

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ mkieffer/ vnmrsys/ data/ MEK-N-PH-TBAF/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	1000
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-08-15T14:42:00
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1785.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



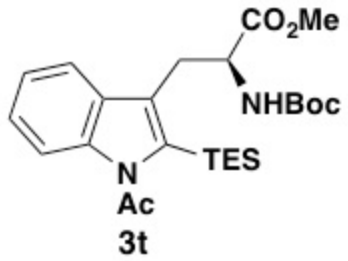
3s

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6140-B/ PROTON02.fid/ fid
Title	PROTON02
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	8
Receiver Gain	16
Relaxation Delay	1.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-01-12T19:04:14
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1016.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536

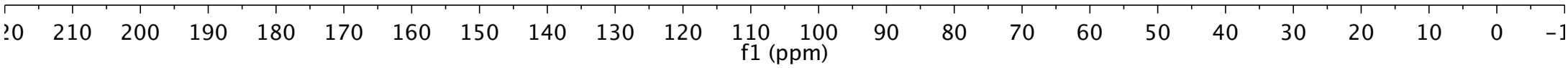


3t

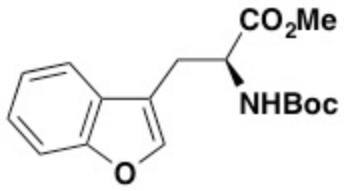
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6140-B/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	512
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-01-12T19:05:10
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1781.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



3t

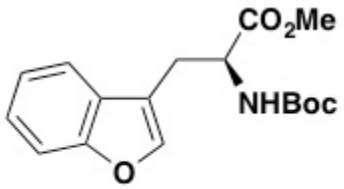


Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK-Phenol-TBAF/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	16
Receiver Gain	30
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-08-15T13:58:50
Spectrometer Frequency	499.69
Spectral Width	8000.0
Lowest Frequency	-1019.8
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



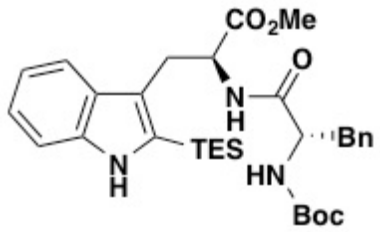
3u

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK-Phenol-TBAF/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	1000
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-08-15T14:01:14
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1786.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



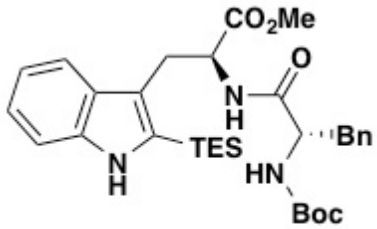
3u

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6062-C-MeCN/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	18
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2013-10-13T20:57:06
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1016.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



5a

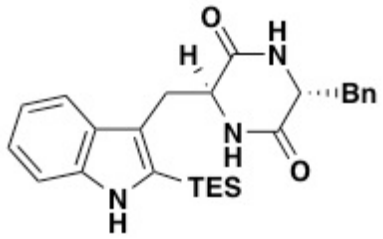
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6062-C-MeCN/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	256
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2013-10-13T21:01:45
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1790.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



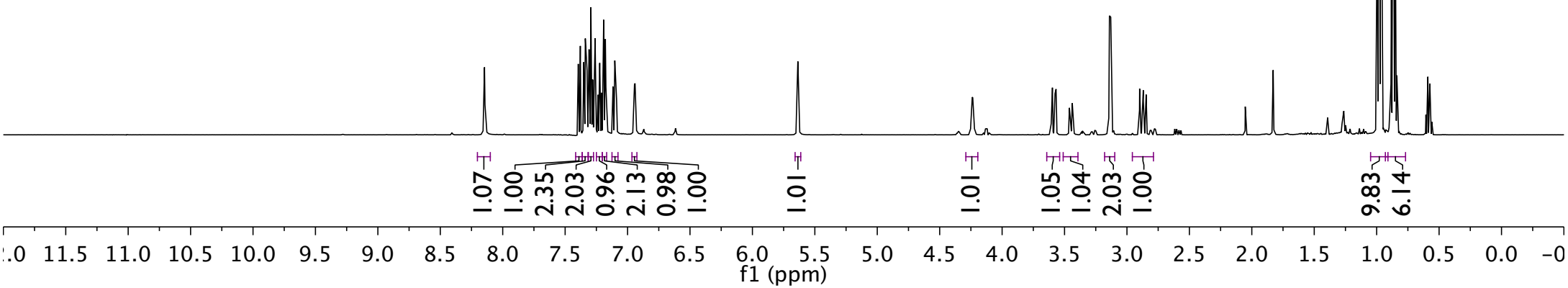
5a

8.15 7.40 7.39 7.38 7.38 7.38 7.35 7.35 7.34 7.34 7.33 7.33 7.32 7.31 7.31 7.30 7.29 7.29 7.28 7.23 7.22 7.19 7.19 7.19 7.18 7.17 7.12 7.12 7.10 7.10 7.10 6.94 6.94 5.64 5.63 3.60 3.58 3.57 3.14 3.13 2.90 2.88 2.87 1.00 0.99 0.99 0.99 0.98 0.98 0.97 0.97 0.96 0.96 0.95 0.88 0.88 0.87 0.87 0.86 0.86 0.85 0.85 0.85 0.84 0.83

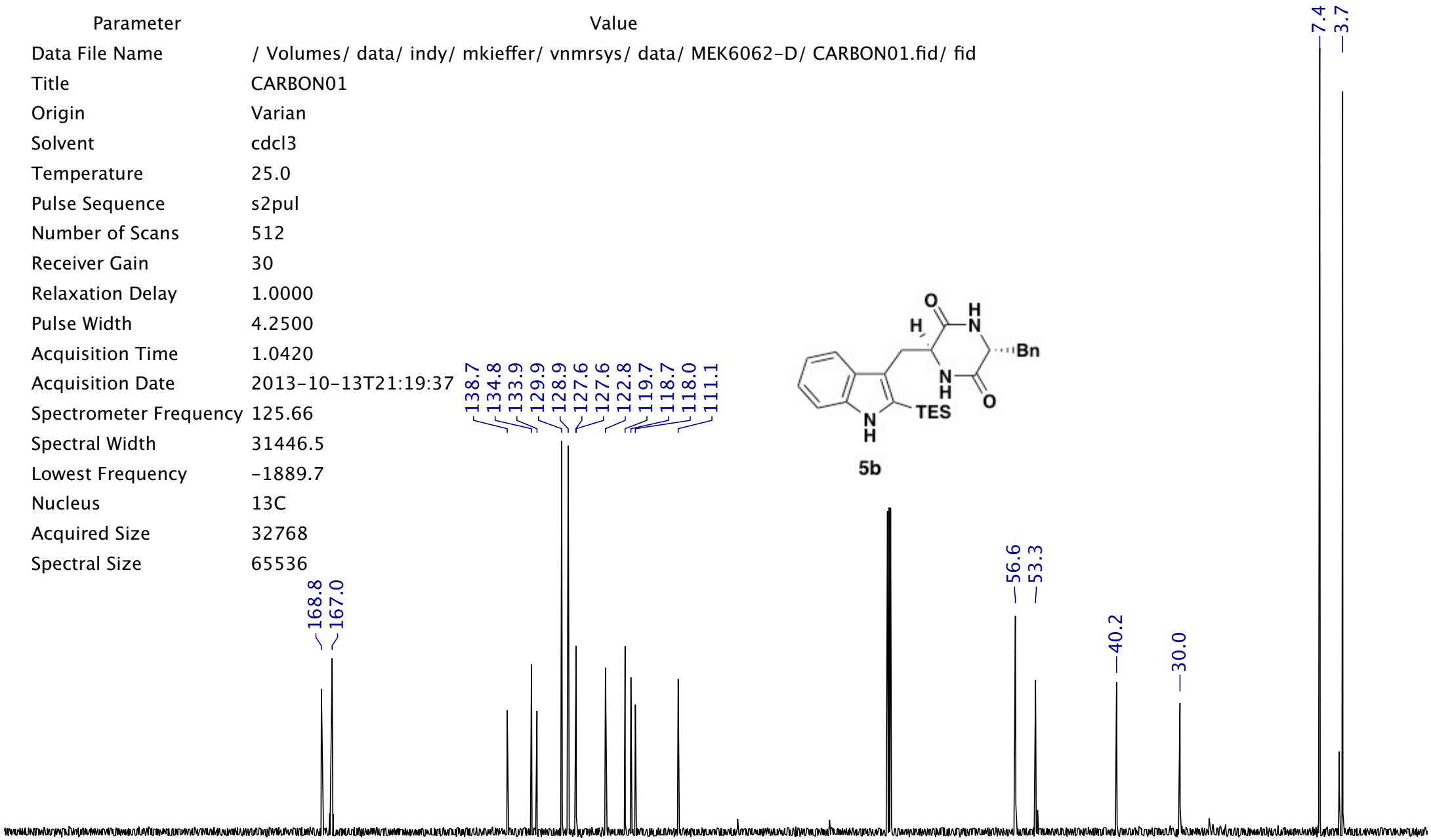
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6062-D/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	22
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2013-10-13T21:15:06
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1004.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



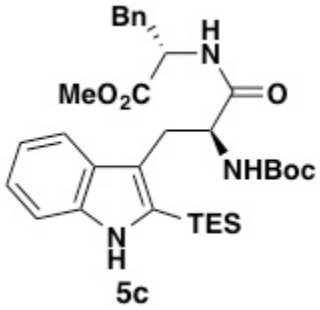
5b



Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6062-D/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	512
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2013-10-13T21:19:37
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1889.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

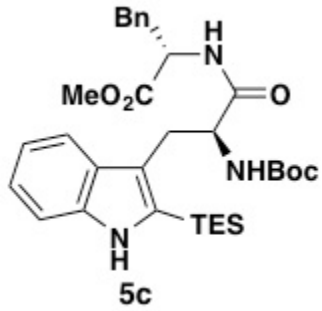


Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6062-A-MeCN/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	22
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2013-10-13T20:28:41
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1016.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



5c

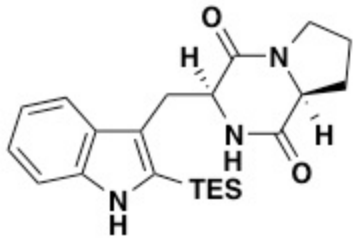
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6062-A-MeCN/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	512
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2013-10-13T20:33:19
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1789.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



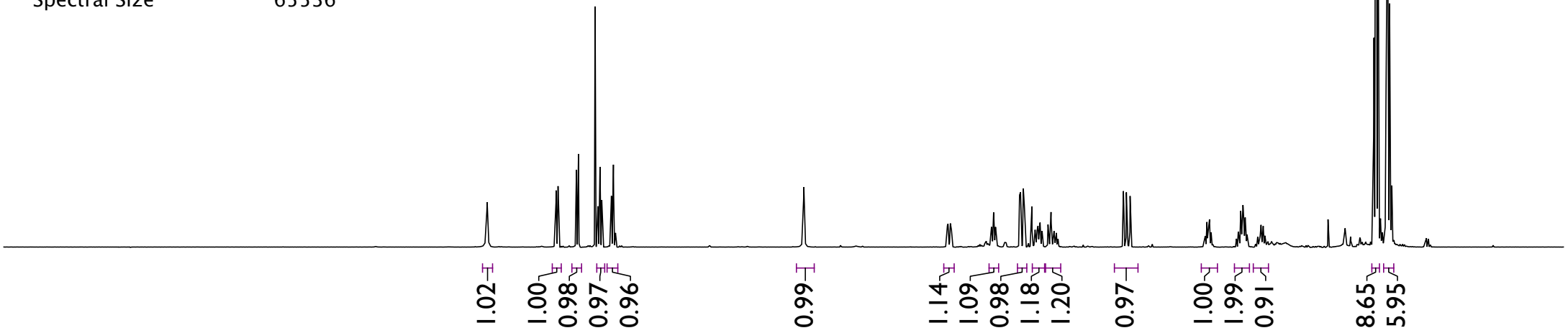
5c

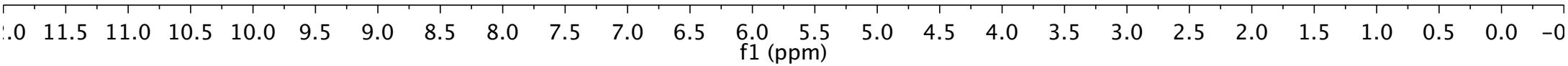
8.12 7.57 7.57 7.56 7.55 7.41 7.41 7.41 7.39 7.39 7.39 7.24 7.23 7.22 7.22 7.22 7.21 7.20 7.13 7.13 7.12 7.11 7.11 7.10 7.10 5.59 4.07 3.86 3.85 3.83 3.82 3.61 3.03 3.00 3.00 2.97 2.09 2.07 2.07 2.07 2.07 1.02 1.02 1.02 1.01 1.01 1.01 1.01 1.00 1.00 1.00 0.99 0.99 0.99 0.98 0.93 0.93 0.91 0.91 0.91 0.90 0.90 0.89 0.88 0.88

Parameter	Value
Data File Name	/ Volumes/ nmrdata/ mkieffer/ vnmrsys/ data/ MEK6062-B/ PROTON02.fid/ fid
Title	PROTON02
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	28
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2013-09-09T22:42:21
Spectrometer Frequency	499.70
Spectral Width	8000.0
Lowest Frequency	-1004.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536

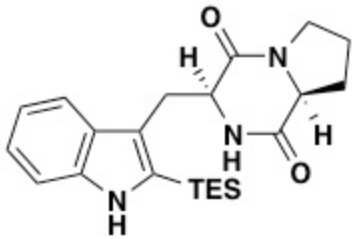


5d



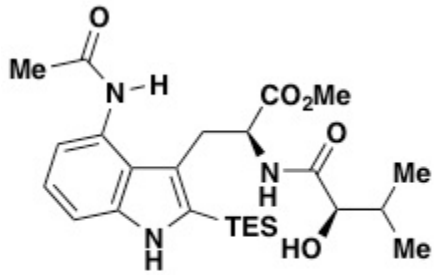


Parameter	Value
Data File Name	/ Volumes/ nmrdata/ mkieffer/ vnmrsys/ data/ MEK6062-B/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	512
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2013-09-09T22:46:52
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1884.7
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



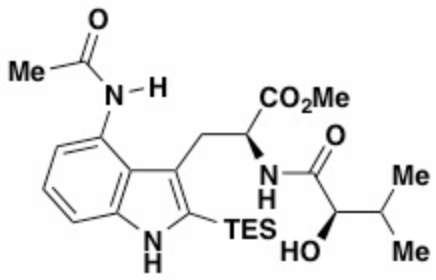
5d

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6247/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	24
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2014-07-11T04:22:38
Spectrometer Frequency	499.69
Spectral Width	8000.0
Lowest Frequency	-1019.3
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



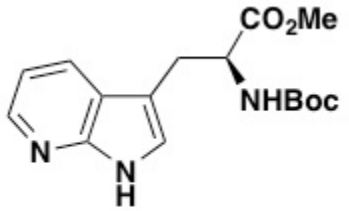
5e

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK6247/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	2000
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2014-07-11T04:27:17
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1786.4
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



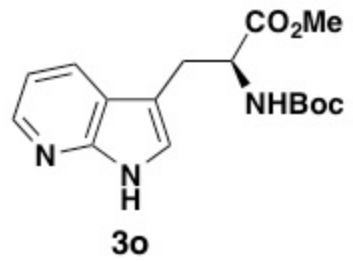
5e

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK7013/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	28
Relaxation Delay	5.0000
Pulse Width	5.9000
Acquisition Time	3.0000
Acquisition Date	2015-02-23T17:05:11
Spectrometer Frequency	499.68
Spectral Width	8000.0
Lowest Frequency	-1039.5
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



3o

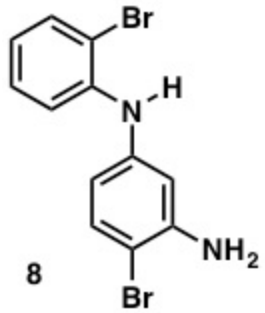
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK7013/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	512
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.5000
Acquisition Time	1.0420
Acquisition Date	2015-02-23T17:09:44
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1790.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



3o

7.63 7.63 7.61 7.61 7.40 7.38 7.35 7.35 7.33 7.33 7.28 7.28 7.26 7.26 7.26 7.26 7.25 7.25 6.87 6.87 6.86 6.86 6.86 6.85 6.84 6.84 6.59 6.59 6.53 6.52 6.51 6.51 6.18 6.17 6.17 6.06 6.05 4.15 4.14

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK7017/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	16
Receiver Gain	16
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2015-01-15T22:20:38
Spectrometer Frequency	499.68
Spectral Width	8000.0
Lowest Frequency	-986.9
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



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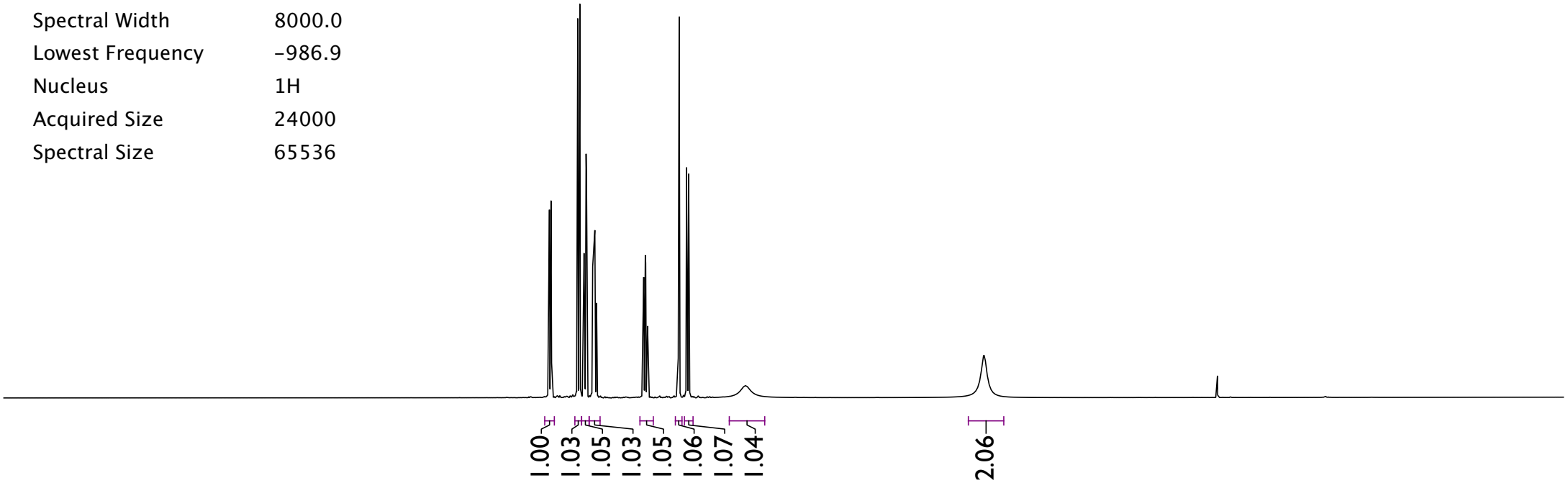
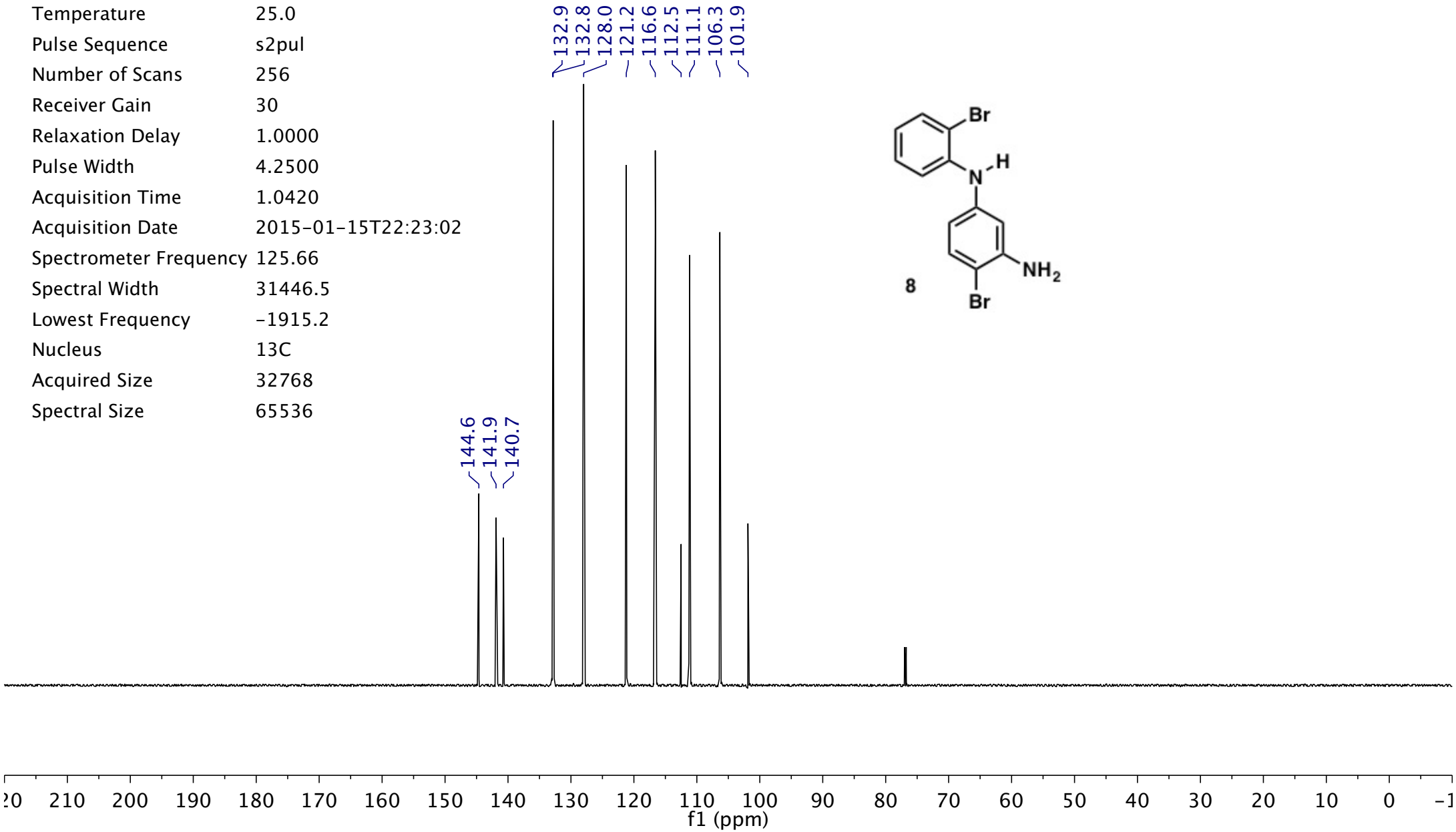
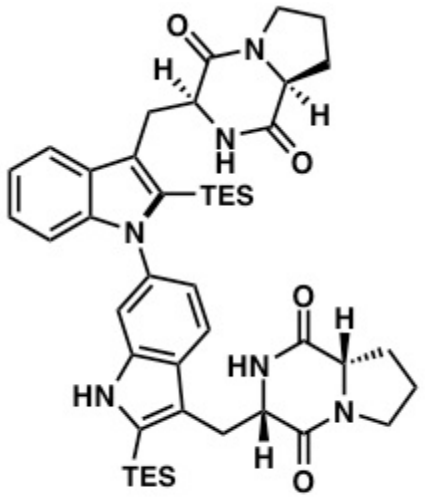


Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK7017/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cdcl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	256
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2015-01-15T22:23:02
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1915.2
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

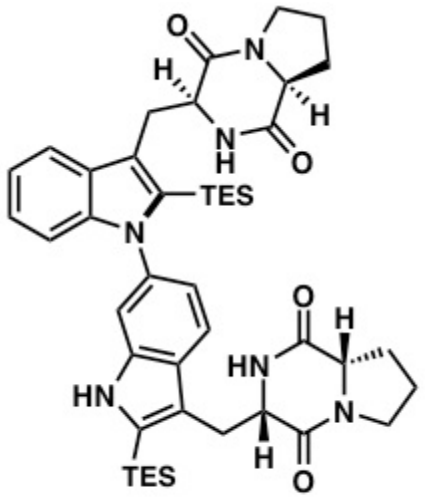


Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK7028-silyated/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd2cl2
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	24
Relaxation Delay	5.0000
Pulse Width	5.9000
Acquisition Time	3.0000
Acquisition Date	2015-02-22T15:25:22
Spectrometer Frequency	499.68
Spectral Width	8000.0
Lowest Frequency	-1032.0
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



SI-IV, CD_2Cl_2

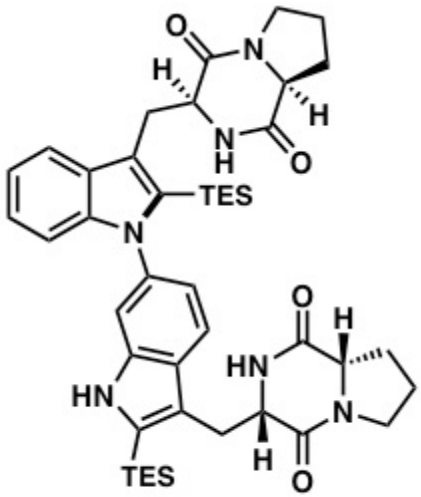
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ MEK7028-silylated/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd2cl2
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	1700
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.5000
Acquisition Time	1.0420
Acquisition Date	2015-02-22T15:49:42
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1832.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



SI-IV, CD_2Cl_2

7.70 7.66 7.64 7.50 7.50 7.47 7.46 7.10 7.09 7.08 7.08 7.07 5.59 5.47 4.43 4.12 4.10 3.76 3.57 3.48 3.47 3.46 2.21 2.20 2.19 1.97 1.96 1.96 1.95 1.95 1.94 1.93 1.93 1.92 1.91 1.91 1.89 1.03 1.02 1.00 1.00 0.99 0.98 0.97 0.96 0.96 0.95 0.94 0.94 0.86 0.84 0.84 0.82 0.82 0.80 0.59 0.58 0.57 0.56 0.55 0.54 0.54 0.53 0.52 0.52

Parameter	Value
Data File Name	/ Volumes/ data/ siena/ mkieffe1/ vnmrsys/ data/ MEK-asp-silyl/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	8
Receiver Gain	30
Relaxation Delay	2.0000
Pulse Width	4.2500
Acquisition Time	2.5559
Acquisition Date	2015-03-01T13:30:55
Spectrometer Frequency	399.80
Spectral Width	6410.3
Lowest Frequency	-815.6
Nucleus	1H
Acquired Size	16384
Spectral Size	65536



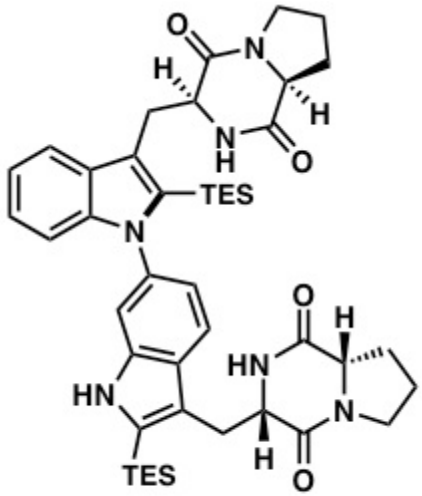
SI-IV, CD_3CN, 25 °C

1.00 1.08 1.06 1.00 3.09 1.02 1.01 1.00 2.19 2.14 2.08 2.15 2.09 0.99 1.00 7.87 8.43 15.26 9.34 6.01

11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0

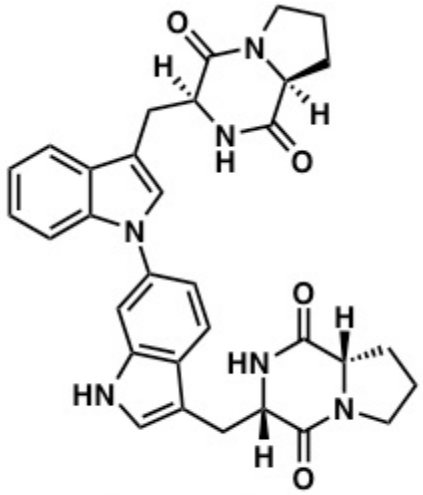
f1 (ppm)

Parameter	Value
Data File Name	/ Volumes/ nmrdata-1/ mkieffe1/ vnmrsys/ data/ MEK-asp-silyl-60degrees/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd3cn
Temperature	61.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	50
Relaxation Delay	5.0000
Pulse Width	4.2500
Acquisition Time	2.5559
Acquisition Date	2015-03-01T13:53:15
Spectrometer Frequency	399.80
Spectral Width	6410.3
Lowest Frequency	-806.4
Nucleus	1H
Acquired Size	16384
Spectral Size	65536



SI-IV, CD_3CN, 60 °C

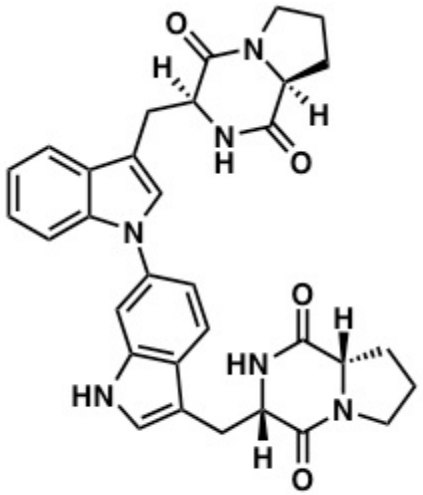
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ aspergilazine_A_DMSO/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	dmso
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	34
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2015-02-19T12:14:22
Spectrometer Frequency	499.68
Spectral Width	8000.0
Lowest Frequency	-1018.9
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



(−)-aspergilazine A (10)
DMSO

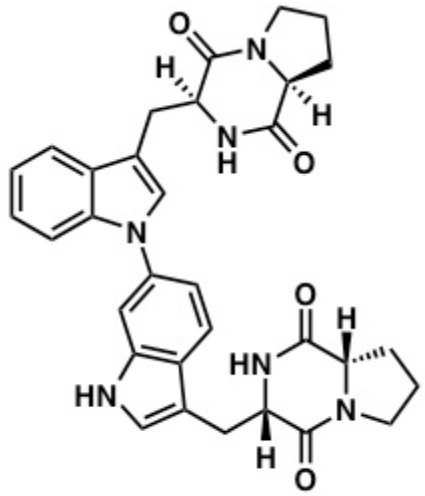
f1 (ppm)

Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ aspergilazine_A_DMSO/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	dmso
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	1500
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2015-02-19T12:18:55
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1957.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

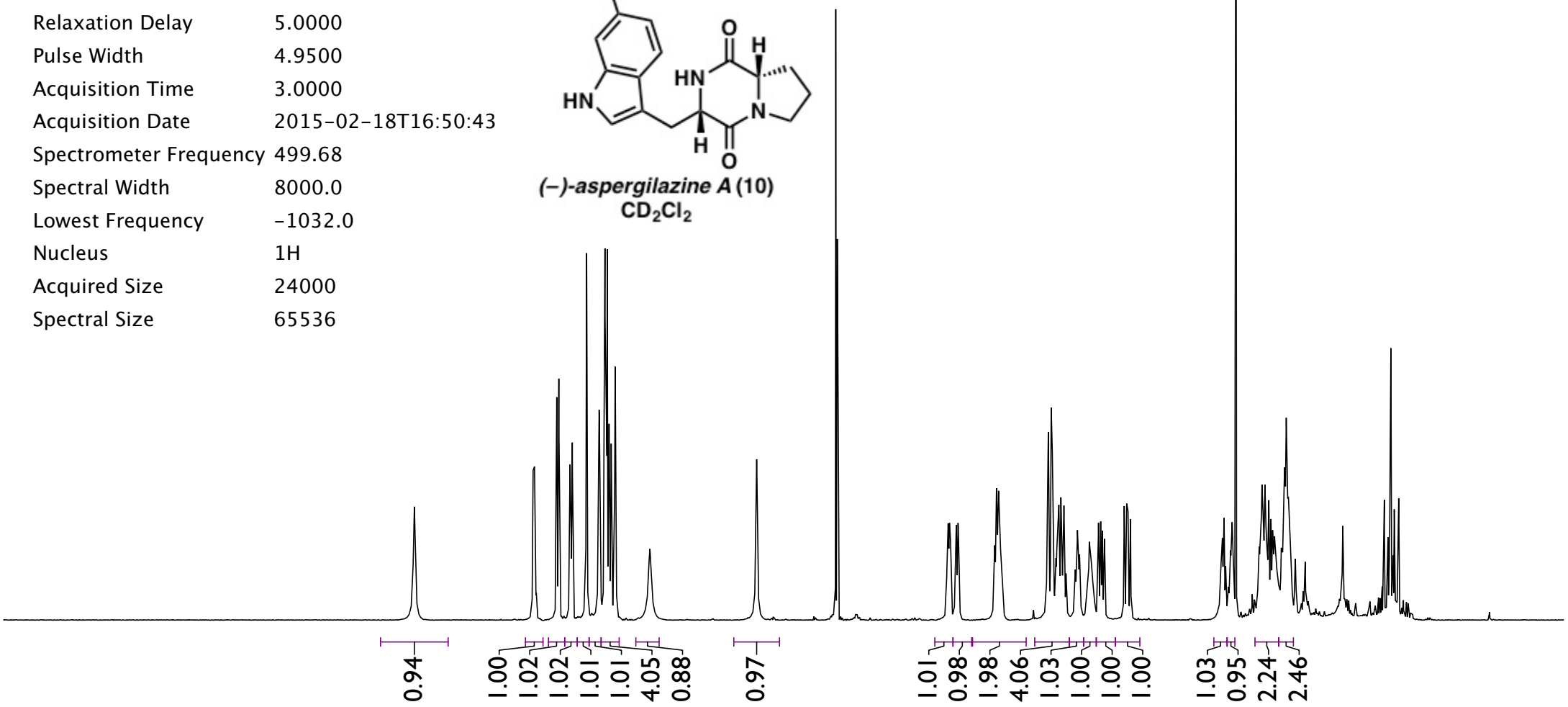


(−)-aspergilazine A (10)
DMSO

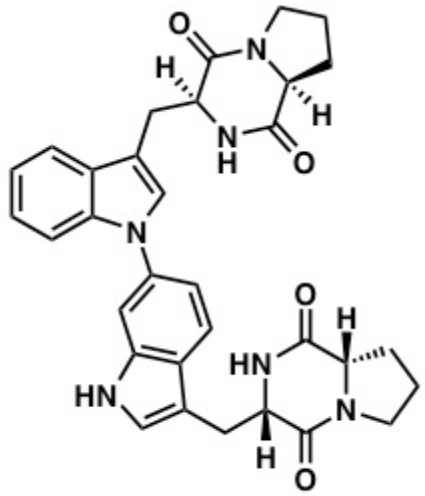
Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ aspergilzine_A_CD2Cl2/ PROTON01.fid/ fid
Title	PROTON01
Origin	Varian
Solvent	cd2cl2
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	32
Receiver Gain	30
Relaxation Delay	5.0000
Pulse Width	4.9500
Acquisition Time	3.0000
Acquisition Date	2015-02-18T16:50:43
Spectrometer Frequency	499.68
Spectral Width	8000.0
Lowest Frequency	-1032.0
Nucleus	1H
Acquired Size	24000
Spectral Size	65536



(−)-aspergilazine A (10)
CD_2Cl_2



Parameter	Value
Data File Name	/ Volumes/ data/ indy/ mkieffer/ vnmrsys/ data/ aspergilzine_A_CD2Cl2/ CARBON01.fid/ fid
Title	CARBON01
Origin	Varian
Solvent	cd2cl2
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	1500
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	4.2500
Acquisition Time	1.0420
Acquisition Date	2015-02-18T16:55:16
Spectrometer Frequency	125.66
Spectral Width	31446.5
Lowest Frequency	-1833.8
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



(−)-aspergilazine A (10)
CD_2Cl_2