Indolyne Experimental and Computational Studies: Synthetic Applications and Origins of Selectivities of Nucleophilic Additions

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Supporting Information – Table of Contents

Full reference 35:

Gaussian 09, Revision A.02, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2009.

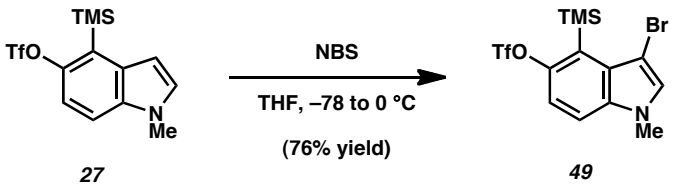
Materials and Methods. Unless stated otherwise, reactions were conducted in flame-dried glassware under an atmosphere of nitrogen using anhydrous solvents (either freshly distilled or passed through activated alumina columns). All commercially obtained reagents were used as received unless otherwise specified. Cesium fluoride (CsF) was obtained from Strem Chemicals. 6-Benzyloxyindole was obtained from Combi-Blocks, Inc. The following reagents were distilled prior to use: chlorotrimethylsilane (TMSCl), trimethylsilyl trifluoromethanesulfonate (TMSOTf), *tert*-butyldimethylsilyl trifluoromethanesulfonate (TBSOTf), and tetramethylethylenediamine (TMEDA); triethylamine (Et_3N) was distilled from calcium hydride; 1,2-dibromoethane was purified by successive aqueous washes (12 M HCl, H_2O, and saturated sodium bicarbonate), dried over $CaCl_2$, and then distilled neat; 1,4-dioxane was degassed (two freeze-pump-thaw cycles), and then distilled over sodium metal. 2-Isopropoxy-4,4,5,5-tetramethyl-1,3,2-dioxaborolane was dried over 3 Å molecular sieves. Diethylamine (Et_2NH) was stirred over KOH for 1 h and then passed over basic Brockman Grade I 58 Å activated alumina prior to use. Aniline was passed over neutral Brockman Grade I 58 Å activated alumina prior to use. DMSO, *n*-pentane, and furan were dried over $MgSO_4$. Reaction temperatures were controlled using an IKAmag temperature modulator, and unless stated otherwise, reactions were performed at room temperature (rt, approximately 23 °C). Thin-layer chromatography (TLC) was conducted with EMD gel 60 F254 pre-coated plates (0.25 mm) and visualized using a combination of UV, anisaldehyde, iodine, vanillin, and potassium permanganate staining. Silicycle Siliaflash P60 (particle size 0.040–0.063 mm) was used for flash column chromatography. ^{1}H NMR and 2D-NOESY spectra were recorded on Bruker spectrometers (at 300 MHz or 500 MHz) and are reported relative to deuterated solvent signals. Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration. ^{13}C NMR spectra were recorded on Bruker Spectrometers (at 75 or 125 MHz). Data for ^{13}C NMR spectra are reported in terms of chemical shift, and when necessary, multiplicity, coupling constant (Hz) and carbon type. IR spectra were recorded on a Perkin-Elmer 100 spectrometer and are reported in terms of frequency of absorption (cm^{-1}). High resolution mass spectra were obtained from the UC Irvine Mass Spectrometry Facility.

Experimental Procedures.

A. Synthesis of a 4,5-indolyne precursor and synthetic applications

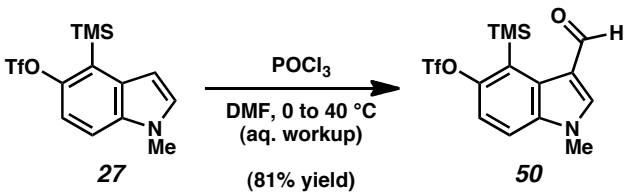
Supporting information for the synthesis of silyltriflate **27** and its synthetic applications has previously been reported in an earlier publication from our laboratory.[1]

B. Stability and functionalization of indolyne precursors



27 *49*

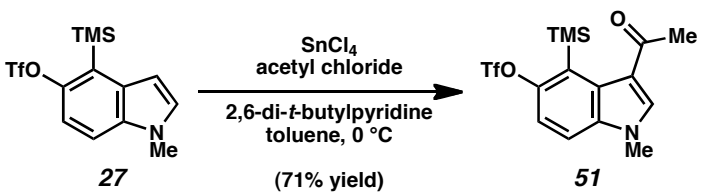
3-Bromo silyltriflate 49. To a solution of silyltriflate **27** (69.1 mg, 0.20 mmol) in THF (2 mL) at –78 °C was added NBS (39.2 mg, 0.22 mmol, 1.1 equiv). After stirring for 5 min, the reaction was allowed to warm to 0 °C over 5 min. The reaction mixture was quenched with aqueous $Na_2S_2O_5$ (5 mL), further diluted with Et_2O (5 mL), and allowed to warm to 23 °C. The layers were separated and the aqueous layer was extracted with Et_2O (2 × 5 mL). The combined organic layers were dried over Na_2SO_4. Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (8:1 Hexanes:EtOAc) to give 3-bromo silyltriflate **49** (64.2 mg, 76% yield) as a white crystalline solid. R_f 0.43 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, C_6D_6): δ 7.04 (d, J = 9.0, 1H), 6.53 (d, J = 9.0, 1H), 6.39 (s, 1H), 2.56 (s, 3H), 0.77 (s, 9H); ^{13}C NMR (125 MHz, C_6D_6): δ 149.0, 134.6, 131.8, 130.7, 125.0, 118.9 (q, J = 319, CF_3), 115.4, 111.9, 89.2, 31.6, 3.4; IR (film): 1415, 1397, 1212, 1131 cm^{-1}; HRMS-ESI (m/z) $[M + H]^+$ calculated for $C_{13}H_{16}BrF_3NO_3SSi$, 429.9756; found, 429.9738.



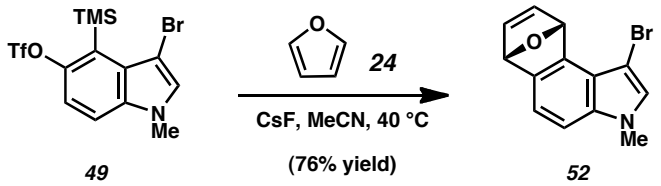
27 *50*

3-Formyl silyltriflate 50: To a vial containing DMF (1.5 mL) at 0 °C was added $POCl_3$ (150 μL, 1.61 mmol, 2.5 equiv). After stirring for 5 min, silyltriflate **27** (223.4 mg, 0.64 mmol) was added as a solution in DMF (1.25 mL), and the reaction was allowed to warm to 23 °C. After stirring for 45 min, the reaction vessel was placed in an aluminum heating block maintained at 40 °C. After stirring for an additional 45 min, the reaction was cooled to 23 °C and saturated

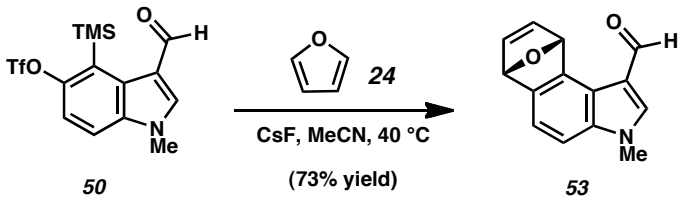
aqueous $NaHCO_3$ (7 mL) was added. The reaction mixture was stirred at 23 °C for 3.3 h, then diluted with Et_2O (15 mL). The layers were separated, and then the aqueous layer was extracted with Et_2O (2 × 15 mL). The combined organic layers were washed with brine (5 mL), then dried over Na_2SO_4. Evaporation under reduced pressure afforded 3-formyl silyltriflate **50** (194.6 mg, 81% yield), which was used in the subsequent step without further purification. R_f 0.42 (1:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.11 (s, 1H), 7.95 (s, 1H), 7.41 (d, J = 9.0, 1H), 7.23 (d, J = 9.0, 1H), 3.89 (s, 3H), 0.51 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 184.9, 150.9, 139.3, 136.3, 130.6, 127.0, 120.4, 118.8 (q, J = 318, CF_3), 116.9, 112.9, 34.2, 2.0; IR (film): 3063, 2952, 1663, 1527, 1414, 1202, 1132 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calculated for $C_{14}H_{16}F_3NO_4SSiNa$, 402.0419; found, 402.0424.



3-Acetyl silyltriflate 51. To a solution of silyltriflate **27** (1 g, 2.85 mmol) and 2,6-di-*t*-butylpyridine (700 μL, 3.24 mmol, 1.1 equiv) in toluene (26 mL) at 0 °C was added sequentially acetyl chloride (370 μL, 5.20 mmol, 1.8 equiv) and a solution of $SnCl_4$ in CH_2Cl_2 (1 M, 5.25 mL, 5.25 mmol, 1.8 equiv). The resulting mixture was stirred for 30 min, poured into a 23 °C solution of saturated aqueous $NaHCO_3$ (25 mL), and further diluted with brine (25 mL). The layers were separated, and the aqueous layer was extracted with EtOAc (3 × 25 mL). The combined organic layers were dried over Na_2SO_4. Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (2:1:1 Hexanes:CH_2Cl_2:Et_2O) to afford 3-acetyl silyltriflate **51** (800 mg, 71% yield) as a white solid. R_f 0.44 (1:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.49 (s, 1H), 7.21 (d, J = 9.0, 1H), 7.13 (d, J = 8.5, 1H), 3.70 (s, 3H), 2.43 (s, 3H), 0.43 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.2, 151.1, 137.6, 135.8, 130.3, 128.8, 120.2, 118.5 (q, J = 318, CF_3), 116.3, 111.9, 33.6, 27.4, 2.0; IR (film): 2956, 1647, 1525, 1406, 1212, 1198 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calculated for $C_{15}H_{18}F_3NO_4SSiNa$, 416.0576; found, 416.0577.

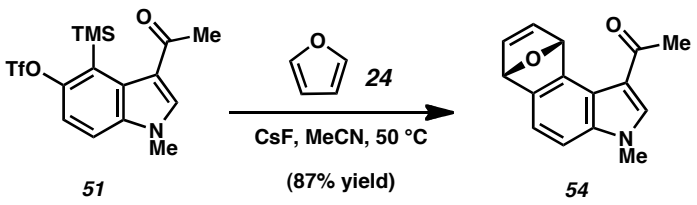


3-Bromo Diels–Alder adduct 52. To a solution of 3-bromo silyltriflate **49** (30.7 mg, 0.071 mmol) and furan (**24**) (26 μL, 0.359 mmol, 5 equiv) in MeCN (0.8 mL) was added CsF (33.9 mg, 0.223 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 40 °C for 90 min. After cooling to 23 °C, the reaction was filtered over silica gel (99:1 EtOAc:Et_3N eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (60:20:20:1 Hexanes:CH_2Cl_2:Et_2O:Et_3N) to give Diels–Alder adduct **52** (15.0 mg, 76% yield) as a white crystalline solid. R_f 0.38 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, C_6D_6): 7.03 (d, J = 8.0, 1H), 6.92 (dd, J = 5.5, 1.5, 1H), 6.77 (dd, J = 5.5, 1.5, 1H), 6.69 (s, 1H), 6.44 (d, J = 8.0, 1H), 6.28 (s, 1H), 5.62 (s, 1H), 2.65 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 144.5, 143.4, 142.0, 141.9, 135.8, 129.6, 122.8, 115.3, 104.7, 86.5, 82.4, 80.6, 33.2; IR (film): 3116, 3017, 2919, 1523, 1290, 1279, 1042 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calculated for $C_{13}H_{10}BrNONa$, 297.9843; found 297.9853.

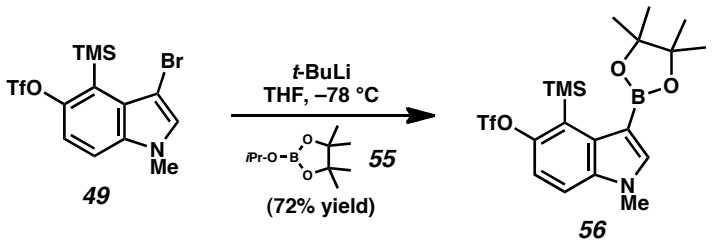


3-Formyl Diels–Alder adduct 53. To a solution of aldehyde **50** (32.2 mg, 0.085 mmol) and furan (**24**) (31 μL, 0.428 mmol, 5 equiv) in MeCN (0.9 mL) was added CsF (42.0 mg, 0.276 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 40 °C for 2 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (1:1 Hexanes:EtOAc) to give adduct **53** (13.9 mg, 73% yield)

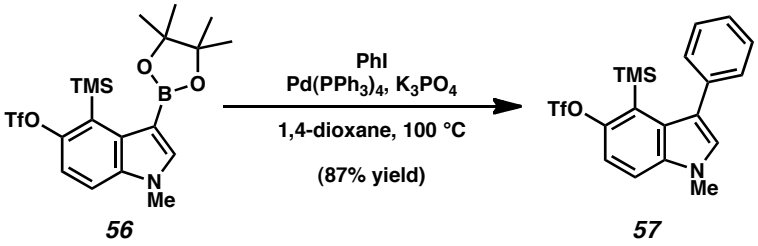
as a white solid. R_f 0.22 (1:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.86 (s, 1H), 7.66 (s, 1H), 7.32 (d, *J* = 7.5, 1H), 7.22 (dd, *J* = 5.4, 1.7, 1H), 7.09 (dd, *J* = 5.4, 1.6, 1H), 6.94 (d, *J* = 7.9, 1H), 6.69 (s, 1H), 5.84 (s, 1H), 3.81 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): (13 of 14 C), δ 183.1, 145.2, 144.3, 143.7, 141.7, 137.2, 119.3, 117.4, 116.3, 105.2, 84.0, 82.7, 33.9; IR (film): 3101, 2800, 1655, 1533 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calculated for $C_{14}H_{11}NO_2Na$, 248.0687; found, 248.0685.



3-Acetyl Diels–Alder adduct 54. To a solution of ketone **51** (31.1 mg, 0.079 mmol) and furan (**24**) (30 μL, 0.414 mmol, 5 equiv) in MeCN (0.8 mL) was added CsF (36.2 mg, 0.238 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50 °C for 130 min. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAC eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (1:1 Hexanes:EtOAc) to give adduct **54** (16.4 mg, 87% yield) as a white solid. R_f 0.20 (1:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.65 (s, 1H), 7.28 (d, *J* = 7.8, 1H), 7.24 (dd, *J* = 5.5, 1.9, 1H), 7.08 (dd, *J* = 5.5, 1.8, 1H), 6.89 (d, *J* = 7.9, 1H), 6.81 (s, 1H), 5.81 (s, 1H), 3.76 (s, 3H), 2.47 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 191.5, 144.9, 144.6, 144.5, 143.6, 137.9, 136.9, 120.6, 116.0, 115.9, 104.9, 84.4, 82.6, 33.7, 26.9; IR (film): 3013, 1635, 1533, 1454, 1374 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calculated for $C_{15}H_{13}NO_2Na$, 262.0844; found, 262.0844.

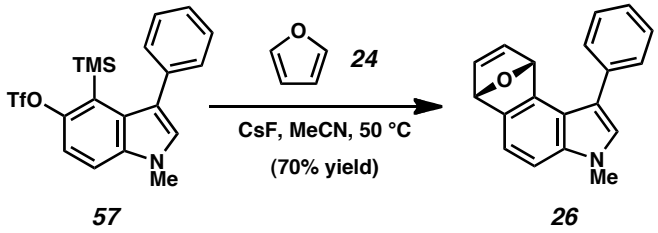


3-Boronic ester silyltriflate 56. To a solution of 3-bromo silyltriflate **49** (72.9 mg, 0.169 mmol) in THF (2 mL) at –78 °C was added *t*-BuLi (550 μL, 0.484 mmol, 2.9 equiv) dropwise over 3 min. After stirring for 20 min, boronic ester **55** (175 μL, 0.856 mmol, 5.1 equiv) was added dropwise over 2 min. The reaction was stirred for 5.5 h, quenched with aqueous NH_4Cl (1 mL), and allowed to warm to 23 °C. The solution was further diluted with H_2O (5 mL) and Et_2O (10 mL). The layers were separated, and the aqueous layer was extracted with Et_2O (2 × 10 mL). The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure. The crude residue was purified by flash chromatography (12:1 Hexanes:EtOAc) to afford boronic ester **56** (58.2 mg, 72% yield) as a white solid. R_f 0.34 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.68 (s, 1H), 7.32 (d, J = 8.9, 1H), 7.13 (d, J = 8.9, 1H), 3.77 (s, 3H), 1.35 (s, 12H), 0.53 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): (12 of 13 C): δ 150.0, 142.3, 137.0, 136.2, 127.8, 118.8 (q, J = 318, CF_3), 115.0, 111.6, 83.3, 33.4, 25.1, 2.1; IR (film): 2987, 1516, 1415, 1217, 1132 cm^{-1}; HRMS-ESI [M + Na]$^+$ calculated for $C_{19}H_{27}BF_3NO_5SSiNa$, 500.1326; found, 500.1331.



3-Phenyl silyltriflate 57. 57 was prepared following the general procedure described by Fürstner for cross-coupling of indolyl 3-boronic esters.[2] A Schlenk tube was charged with boronic ester **56** (61.1 mg, 0.128 mmol) and K_3PO_4 (81.6 mg, 0.384 mmol, 3 equiv), and then brought into a glovebox. To this mixture was added $Pd(PPh_3)_4$ (8.4 mg, 7.3 μmol, 0.057 equiv), iodobenzene

(22 μL, 0.197 mmol, 1.5 equiv), and 1,4-dioxane (1 mL). The vessel was sealed under inert atmosphere, removed from the glovebox, and heated to 80 °C. After 19.5 h the temperature was raised to 100 °C and maintained for 8 h. The reaction was then cooled to 23 °C, diluted with EtOAc (5 mL), and filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (8:1:1 Hexanes:Benzene:Et_2O) to afford cross-coupled product **57** (47.7 mg, 87% yield) as an off-white solid. R_f 0.51 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.47–7.43 (m, 2H), 7.41 (app. t, J = 7.6, 2H), 7.39 (d, J = 9.0, 1H), 7.33-7.29 (m, 1H), 7.17 (d, J = 9.0, 1H), 7.16 (s, 1H), 3.83 (s, 3H), 0.12 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 150.4, 137.4, 135.6, 131.3, 130.9, 129.0, 128.5, 126.4, 125.9, 120.5, 118.6 (q, J = 318, CF_3), 114.7, 111.8, 33.1, 1.8; IR (film): 2948, 1604, 1417, 1405, 1207 cm^{-1}; HRMS-ESI (m/z) $[M + H]^+$ calculated for $C_{19}H_{21}O_3NF_3SSi$, 428.0963; found, 428.0969.



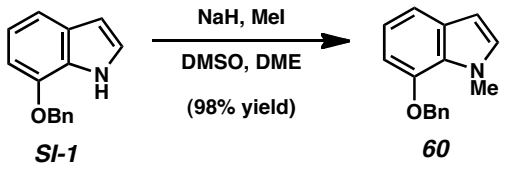
57 *26*

3-Phenyl Diels–Alder adduct 26. To a solution of silyltriflate **57** (35.9 mg, 0.084 mmol) and furan (**24**) (30 μL, 0.414 mmol, 5 equiv) in MeCN (0.85 mL) at 23 °C was added CsF (38.2 mg, 0.251 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50 °C for 6.25 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (6:1 Hexanes:EtOAc) to give adduct **26** (16.1 mg, 70% yield) as a white solid. R_f 0.37 (6:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.52–7.50 (m, 2H), 7.49–7.45 (m, 2H), 7.36–7.32 (m, 1H), 7.29 (d, J = 8.0, 1H), 7.20 (app. t, J = 1.0, 2H), 7.12 (s, 1H), 6.96 (d, J = 8.0, 1H), 6.02 (s, 1H), 6.84 (d, J = 1.0, 1H), 3.78 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 145.2, 142.4, 142.0, 141.2, 136.7, 135.7, 128.52, 128.48, 128.4, 126.1, 122.2, 115.6, 114.7, 104.5, 82.6, 81.9, 33.1; IR (film): 3021, 2922, 1601, 1549, 1456, 1416, 1214

cm^{-1}; HRMS-ESI $[M + Na]^+$ calculated for $C_{19}H_{15}NONa$, 296.1051; found, 296.1056.

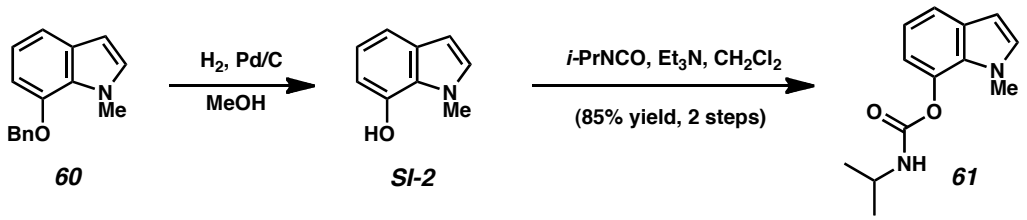
C. Generation and trapping of 5,6- and 6,7-indolynes

Supporting information for the synthesis of *N*-methyl silyltriflates **59** and **68**, and their reactions with various nucleophiles has previously been reported in earlier publications from our laboratory.[1,3]



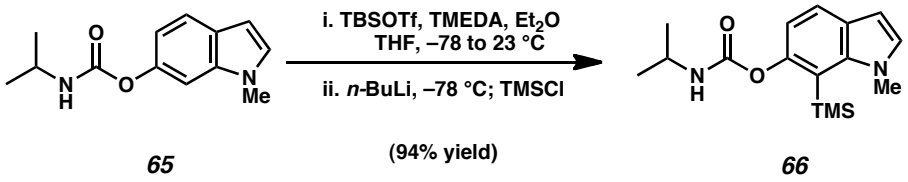
SI-1 ***60***

***N*-Methyl-benzyloxyindole 60.** Benzyloxyindole **60** was prepared following a known procedure, with minor modifications.[4] 95% NaH (0.23 g, 9.0 mmol, 2 equiv) was added to a solution of 7-benzyloxyindole (**SI-1**) (1.0 g, 4.5 mmol) in 1,2-dimethoxyethane (12.4 mL) and DMSO (1.4 mL). The resulting solution was stirred at 23 °C for 40 min, then MeI (0.42 mL, 6.7 mmol, 1.5 equiv) was added dropwise over 1 min. The resulting mixture was stirred for 1.5 h, then quenched with H_2O (4 mL), then brine (4 mL). The biphasic mixture was further diluted with H_2O (20 mL) and EtOAc (20 mL). The layers were separated, and then the aqueous layer was extracted with EtOAc (2 × 20 mL). The combined organic layers were washed with brine (20 mL), then dried over $MgSO_4$. Evaporation under reduced pressure afforded crude **60** (1.04 g, 98% yield) as a red oil, which was used in the subsequent step without further purification. R_f 0.67 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.51 (d, J = 7.3, 2H), 7.44–7.41 (m, 2H), 7.38–7.35 (m, 1H), 7.25 (d, J = 7.9, 1H), 6.99 (app. t, J = 8.2, 1H), 6.94 (d, J = 3.0, 1H), 6.71 (d, J = 7.7, 1H), 6.44 (d, J = 2.9, 1H), 5.20 (s, 2H), 4.06 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 146.9, 137.2, 131.0, 129.8, 128.5, 127.8, 127.4, 126.4, 119.7, 114.0, 103.4, 100.9, 70.3, 36.6; IR (film): 3064, 3030, 2953, 2923, 1575, 1258 cm^{-1}; HRMS-ESI (*m*/*z*) $[M + H]^+$ calcd for $C_{16}H_{16}NO$, 238.1232; found, 238.1236.



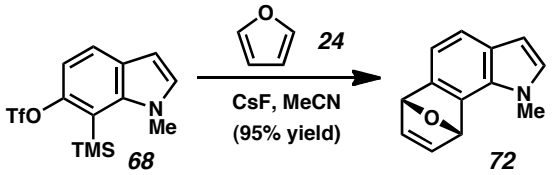
Carbamate 61. *N*-Methyl-benzyloxyindole **60** (0.50 g, 2.1 mmol) was dissolved in MeOH (4.9 mL) and 10% Pd/C (0.11 g, 0.11 mmol, 5.0 mol% Pd) was added.[5] The mixture was placed under an atmosphere of hydrogen (double-balloon), stirred for 11 h at 23 °C, and then filtered over celite (MeOH eluent, 200 mL). Evaporation of the solvent under reduced pressure afforded hydroxyindole **SI-2** (0.30 g, crude) as a grey solid, which was used in the subsequent step without further purification. R_f 0.41 (3:1 Hexanes:EtOAc).

To a solution of crude **SI-2** in CH_2Cl_2 (5 mL) were added sequentially *i*-PrNCO (0.59 mL, 6.0 mmol, 3 equiv) and Et_3N (0.084 mL, 0.6 mmol, 0.3 equiv). The solution was stirred at 23 °C for 9 h, then concentrated to dryness under reduced pressure. Purification by flash chromatography (3:1 Hexanes:EtOAc) provided carbamate **61** (0.41 g, 85% yield, 2 steps). R_f 0.21 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.44 (d, J = 7.9, 1H), 7.02 (app. t, J = 7.7, 1H), 6.93 (d, J = 2.9, 1H), 6.90 (d, J = 7.6, 1H), 6.45 (d, J = 3.0, 1H), 4.96 (s, 1H), 3.98–3.87 (m, 4H), 1.26 (d, J = 6.5, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.1, 136.8, 131.9, 130.3, 128.8, 119.4, 118.4, 115.2, 101.3, 43.5, 35.4, 22.9; IR (film): 3333, 2978, 1696, 1517 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{13}H_{16}N_2O_2Na$, 255.1109; found, 255.1116.



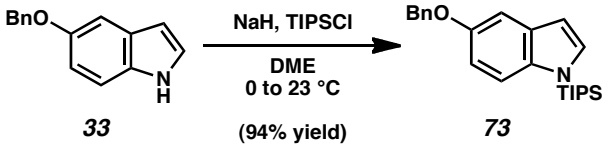
Silyl carbamate 66. To a solution of carbamate **65**[3] (146.4 mg, 0.630 mmol) in 3:1 Et_2O:THF (6.05 mL) at –78 °C was added TMEDA (0.132 mL, 0.882 mmol, 1.40 equiv), followed by a solution of TBSOTf in *n*-pentane (1.30 M, 0.573 mL, 0.756 mmol, 1.2 equiv).[6,7,8] After stirring for 30 min, the white suspension was allowed to warm to 23 °C over 80 min, by which time

TMEDA·TfOH had formed as an oil on the bottom of the flask. TMEDA (0.329 mL, 2.21 mmol, 3.5 equiv) was added, and the mixture was cooled to –78 °C.[9] A solution of *n*-BuLi in hexanes (1.85 M, 1.19 mL, 2.21 mmol, 3.5 equiv) was added dropwise over 5 min.[10] The mixture was stirred at –78 °C for 3 h, then neat TMSCl (0.560 mL, 4.41 mmol, 7.0 equiv) was added dropwise over 5 min. The resulting mixture was stirred at –78 °C for 2 h, quenched with 0.5 M aqueous $NaHSO_4$ (5 mL), and allowed to warm to 23 °C over 1 h with vigorous stirring. The organic layer was separated, and then the aqueous layer was extracted with Et_2O (5 mL). The combined organic layers were washed successively with 0.5 M aqueous $NaHSO_4$ (5 mL) and brine (5 mL), then dried over Na_2SO_4. Evaporation under reduced pressure afforded the crude product, which was purified by flash chromatography (6:1 Hexanes:EtOAc) to afford silylcarbamate **66** (0.180 g, 94% yield). Spectral data match those reported in a previous publication.[3]



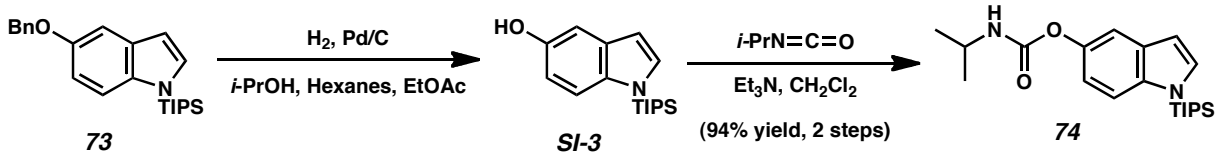
Furan Diels–Alder adduct 72. To a stirred solution of silyltriflate **68**[3] (39.4 mg, 0.1121 mmol) and furan (**24**) (40.8 μL, 0.561 mmol, 5 equiv) in MeCN (1.1 mL) was added CsF (34.1 mg, 0.561 mmol, 2.0 equiv). The solution was stirred at 23 °C for 3 h, then filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (3:1:1 Hexanes:CH_2Cl_2:Et_2O) to provide adduct **72** (21.1 mg, 95% yield). R_f 0.33 (3:1:1 Hexanes:CH_2Cl_2:Et_2O); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.37 (d, *J* = 7.5, 1H), 7.29–7.21 (m, 2H), 7.22 (dd, *J* = 5.5, 1.5, 1H), 7.07 (d, *J* = 3.0, 1H), 6.54 (d, *J* = 3.0, 1H), 6.403–6.400 (m, 1H), 5.97 (d, *J* = 1.0, 1H), 4.03 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 144.8, 144.2, 143.4, 132.1, 131.2, 130.4, 128.9, 117.2, 113.2, 101.6, 83.0, 81.2, 34.9; IR (film): 3089, 3033, 2908, 1304, 1276 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calcd for $C_{13}H_{11}NONa$, 220.0738; found, 220.0737.

D. Indolyne precursors with alternative *N*-substituents



33 *73*

***N*-TIPS benzyloxyindole 73.** To a solution of 5-benzyloxyindole (**33**) (3.00 g, 13.4 mmol) in 1,2-dimethoxyethane (42.0 mL) at 0 °C was added 60% NaH (1.07 g, 26.9 mmol, 2 equiv). The resulting solution was stirred at 0 °C for 20 min, then TIPSCl (4.31 mL, 20.2 mmol, 1.5 equiv) was added dropwise over 5 min. The resulting mixture was removed from the bath and allowed to warm to 23 °C. After stirring for an additional 90 min, the reaction was quenched with brine (60 mL). The biphasic mixture was further diluted with EtOAc (60 mL). The layers were separated, and then the aqueous layer was extracted with EtOAc (2 × 60 mL). The combined organic layers were washed with brine (20 mL), H_2O (20 mL), then dried over $MgSO_4$. Evaporation of the solvent under reduced pressure afforded crude **73**, which was further purified by flash chromatography (3:1 Hexanes:CH_2Cl_2) to afford **73** (4.79 g, 94% yield) as a white solid. R_f 0.73 (3:1 Hexanes:EtOAc); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.60–7.30 (m, 8H), 7.02 (dd, J = 8.7, 2.4, 1H), 6.67 (d, J = 3.0, 1H), 5.18 (s, 2H), 1.75 (septet, J = 7.8, 3H), 1.25 (d, J = 7.5, 18H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.5, 138.0, 136.1, 132.1, 132.0, 128.6, 127.8, 127.7, 114.6, 112.1, 104.7, 103.7, 70.7, 18.2, 12.9; IR (film): 2943, 2864, 1463, 1450, 1158 cm^{-1}; HRMS-ESI (m/z) [M + Na]$^+$ calcd for $C_{24}H_{33}NOSiNa$, 402.2229; found, 402.2217.

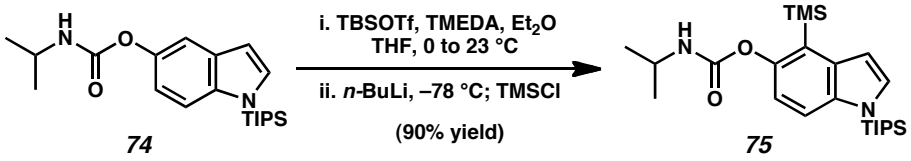


73 *SI-3* *74*

***N*-TIPS carbamate 74.** Carbamate **73** was prepared following the general procedure described by Igarashi.[11] To a solution of *N*-TIPS benzyloxyindole (4.79 g, 12.6 mmol) in 1:1:1 *i*-PrOH:Hexanes:EtOAc (108 mL) was added 5% Pd/C (0.67 g, 0.32 mmol, 2.5 mol% Pd). The

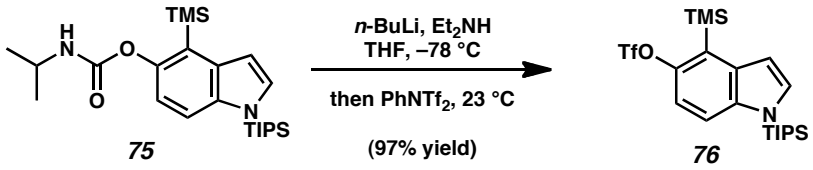
mixture was placed under an atmosphere of hydrogen (double-balloon), stirred for 2.5 h at 23 °C, and then filtered over celite (EtOAc eluent). Evaporation of the solvent under reduced pressure afforded crude **SI-3** as a pink solid, which was used in the subsequent step without further purification. R_f 0.07 (2:1 Hexanes:CH_2Cl_2).

Crude **SI-3** was dissolved in CH_2Cl_2 (63.1 mL), and Et_3N (0.53 mL, 3.79 mmol, 0.3 equiv), followed by *i*-PrNCO (2.66 mL, 27.1 mmol, 2.1 equiv), was added. The solution was stirred at 23 °C for 24 h, then concentrated to dryness under reduced pressure. Purification by flash chromatography (7:3 Hexanes:Et_2O) provided *N*-TIPS carbamate **74** (4.42 g, 94% yield, 2 steps). R_f 0.58 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.45 (d, *J* = 2.0, 1H), 7.43 (d, *J* = 9.0, 1H), 7.25 (s, 1H), 6.91 (dd, *J* = 9.0, 2.0, 1H), 6.58 (d, *J* = 3.5, 1H), 4.87 (d, *J* = 7.0, 1H), 3.91 (m, 1H), 1.68 (septet, *J* = 7.5, 3H), 1.23 (d, *J* = 6.5, 6H), 1.13 (d, *J* = 7.5, 18H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.0, 145.0, 138.7, 132.6, 132.2, 116.0, 114.2, 112.9, 105.3, 43.7, 23.3, 18.4, 13.1; IR (film): 3281, 2955, 2868, 1712 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calcd for $C_{21}H_{34}N_2O_2SiNa$, 397.2287; found, 397.2282.

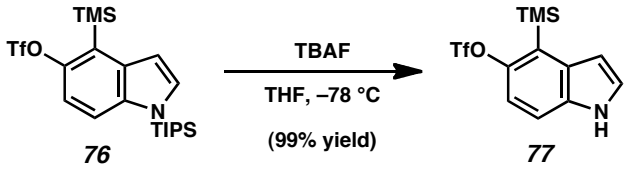


***N*-TIPS silylcarbamates 75.** Silyl carbamate **75** was prepared following the general procedure described by Hoppe and Snieckus for *o*-lithiation of isopropyl carbamates, with minor modifications.[6,7] To a solution of *N*-TIPS carbamate **74** (1.02 g, 2.72 mmol) and TMEDA (0.61 mL, 3.82 mmol, 1.4 equiv) in 3:1 Et_2O:THF (27 mL) at 0 °C was added a solution of TBSOTf in *n*-pentane (1.30 M, 3.21 mL, 3.3 mmol, 1.2 equiv). After stirring for 5 min, the white suspension was allowed to warm to 23 °C over 30 min. TMEDA (1.53 mL, 9.56 mmol, 3.5 equiv) was added, and the mixture was cooled to –78 °C. A solution of *n*-BuLi in hexanes (2.21 M, 4.35 mL, 9.56 mmol, 3.5 equiv) was added dropwise over 8 min. The mixture was stirred at –78 °C for 3 h, then neat TMSCl (2.43 mL, 19.1 mmol, 7 equiv) was added dropwise over 15 min. The resulting mixture was stirred at –78 °C for 1 h, quenched with 1 M $NaHSO_4$ (25 mL), and

allowed to warm to 23 °C over 45 min with vigorous stirring. The organic layer was separated, washed successively with 1 M $NaHSO_4$ (25 mL) and brine (25 mL), then dried over Na_2SO_4. Evaporation under reduced pressure afforded the crude product, which was purified by flash chromatography (15:1:1 Hexanes:Et_2O:CH_2Cl_2) to afford *N*-TIPS silylcarbamate **75** (1.10 g, 90% yield). R_f 0.27 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.49 (d, *J* = 9.0, 1H), 7.29 (d, *J* = 3.0, 1H), 6.88 (d, *J* = 9.0, 1H), 6.79 (d, *J* = 3.5, 1H), 4.82 (d, *J* = 8, 1H), 3.98–3.92 (m, 1H), 1.69 (septet, *J* = 7.5, 3H), 1.24 (d, *J* = 6.5, 6H), 1.15 (d, *J* = 7.5, 18H), 0.44 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.1, 150.0, 138.0, 136.1, 132.1, 121.7, 116.6, 115.6, 106.6, 43.6, 23.3, 18.5, 13.1, 1.5; IR (film): 3400, 2945, 2869, 1742 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{24}H_{42}N_2O_2Si_2Na$, 469.2682; found, 469.2666.

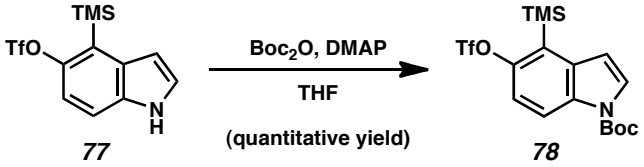


***N*-TIPS silyltriflate 76.** To a solution of silyl carbamate **75** (6.29 g, 14.1 mmol) in THF (142 mL) at –78 °C was added a solution of *n*-BuLi (1.43 M, 12.5 mL, 17.9 mmol, 1.3 equiv) dropwise over 30 min. Et_2NH (1.90 mL, 18.4 mmol, 1.3 equiv) was added and the resulting mixture was stirred at –78 °C for 15 min, then allowed to warm to 23 °C over 15 min. Next, $PhNTf_2$ (7.58 g, 21.2 mmol, 1.5 equiv) was added as a solid. The mixture was allowed to stir for 2 h and then passed over a plug of silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (95:5 Hexanes:Benzene) to provide *N*-TIPS silyltriflate **76** (6.74 g, 97% yield). R_f 0.74 (2:1 Hexanes:Et_2O); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.53 (d, *J* = 9.0, 1H), 7.38 (d, *J* = 3.0, 1H), 7.12 (d, *J* = 9.0, 1H), 6.84 (d, *J* = 3.0, 1H), 1.71 (septet, *J* = 7.5, 3H), 1.17 (d, *J* = 7.5, 18H), 0.53 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 149.2, 138.8, 136.5, 133.3, 123.1, 118.8 (q, *J* = 318, CF_3), 115.8, 113.6, 107.2, 18.2, 13.0, 1.3; IR (film): 2955, 2872, 1411, 1391, 1401 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{21}H_{34}F_3NO_3SSi_2Na$, 516.1648; found, 516.1649.



76 ***77***

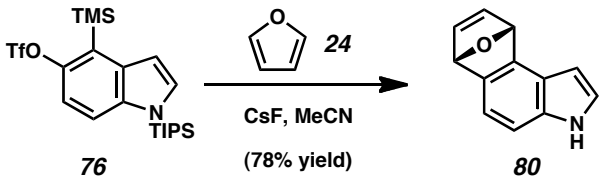
***N*-H silyltriflate 77.** To a solution of *N*-TIPS silyltriflate **76** (0.427 g, 0.865 mmol) in THF (8.9 mL) at –78 °C was added a solution of TBAF in THF (1.0 M, 0.865 mL, 0.865 mmol, 1 equiv) dropwise over 2 min. The solution was stirred for 15 min, then quenched with H_2O (10 mL). After warming to 23 °C, the biphasic mixture was further diluted with Et_2O (10 mL). The layers were separated, and then the aqueous layer was extracted with Et_2O (3 × 10 mL). The combined organic layers were dried over $MgSO_4$. Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (3:1 Hexanes:EtOAc) to provide *N*-H silyltriflate **77** (289 mg, 99% yield). R_f 0.37 (3:1:1 Hexanes:Et_2O:CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.33 (br s, 1H), 7.35 (dd, *J* = 8.9, 0.8, 1H), 7.30 (app. t, *J* = 2.9, 1H), 7.16 (d, *J* = 8.9, 1H) 6.78–6.77 (m, 1H), 0.56 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 148.8, 133.5, 132.5, 126.1, 123.7, 118.7 (q, *J* = 319, CF_3), 114.9, 113.2, 105.0, 1.0; IR (film): 3446, 1396, 1211 cm^{-1}; HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{12}H_{15}F_3NO_3SSi$, 338.0494; found, 338.0490.



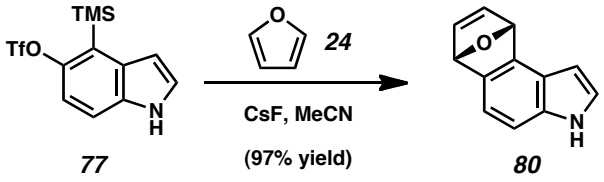
77 ***78***

***N*-Boc silyltriflate 78.** *N*-Boc silyltriflate **78** was prepared following the general procedure described by Gribble for Boc-protection of indoles.[12] DMAP (5.4 mg, 0.044 mmol, 0.1 equiv) was added to a solution of *N*-H silyltriflate **77** (0.150 g, 0.44 mmol) in THF (5 mL). The resulting solution was stirred at 23 °C for 5 min, then Boc_2O (97 mg, 0.44 mmol, 1 equiv) was added. The resulting mixture was stirred for 15 h, then filtered over silica gel (Et_2O eluent). Evaporation under reduced pressure afforded *N*-Boc silyltriflate **78** (0.195 g, quantitative yield). R_f 0.74 (3:2 Hexanes:Et_2O); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.26 (d, *J* = 9.0, 1H), 7.73 (d, *J* = 3.8, 1H), 7.27 (d, *J* = 9.1, 1H), 6.78 (d, *J* = 3.8, 1H), 1.68 (s, 9H), 0.51 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 150.3, 149.2, 135.9, 133.3, 127.5, 124.7, 118.7 (q, *J* = 319, CF_3), 117.4, 116.3,

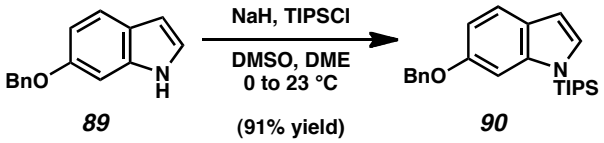
108.5, 84.4, 28.1, 1.1; IR (film): 2982, 1742, 1399, 1210 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{17}H_{22}F_3NO_5SSiNa$, 460.0838; found, 460.0835.



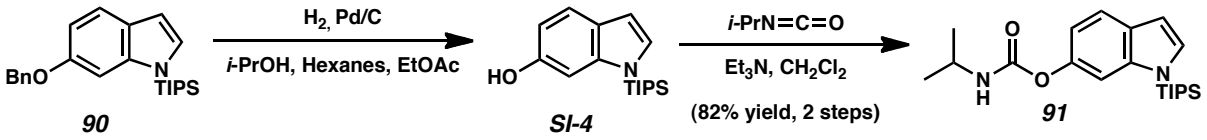
Furan Diels–Alder adduct 80. To a stirred solution of silyltriflate **76** (35.0 mg, 0.0708 mmol) and furan (**24**) (25.7 μL, 0.354 mmol, 5 equiv) in MeCN (1.4 mL) was added CsF (32.3 mg, 0.212 mmol, 3.0 equiv). The solution was stirred at 23 °C for 3.5 h, then filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (3:1:1 Hexanes:CH_2Cl_2:Et_2O) to provide adduct **80** (10.1 mg, 78% yield) as a white solid. R_f 0.27 (3:1:1 Hexanes:CH_2Cl_2:Et_2O); ^1H NMR (500 MHz, $CDCl_3$): δ 8.11 (br s, 1H), 7.24 (dd, *J* = 3.0, 2.5, 1H), 7.21 (d, *J* = 8.0, 1H), 7.16 (app. qd, *J* = 5.5, 2.0, 2H), 7.02 (dd, *J* = 7.5, 0.5, 1H), 6.50–6.49 (m, 1H), 6.04–6.03 (m, 1H), 5.86–5.85 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 145.1, 143.0, 142.0, 141.0, 135.0, 126.2, 123.1, 115.1, 106.2, 99.6, 83.2, 82.0; IR (film): 3293, 3011, 1430 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{12}H_9NONa$, 206.0582; found, 206.0583.



Furan Diels–Alder adduct 80. To a stirred solution of silyltriflate **77** (32.2 mg, 0.0954 mmol) and furan (**24**) (34.7 μL, 0.477 mmol, 5 equiv) in MeCN (1.9 mL) was added CsF (29.0 mg, 0.1908 mmol, 2.0 equiv). The solution was stirred at 23 °C for 2 h, then filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (3:1:1 Hexanes:CH_2Cl_2:Et_2O) to provide adduct **80** (16.9 mg, 97% yield) as a white solid. Spectral data match those reported above (S18**).**

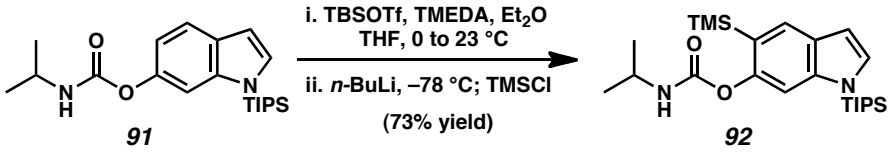


***N*-TIPS benzyloxyindole 90.** To a solution of 6-benzyloxyindole **89** (3.00 g, 13.4 mmol) in 1,2-dimethoxyethane (37.0 mL) and DMSO (4.1 mL) at 0 °C was added 95% NaH (0.97 g, 40.3 mmol, 3 equiv). The resulting solution was stirred at 0 °C for 30 min, then TIPSCl (5.73 mL, 26.8 mmol, 2 equiv) was added dropwise over 5 min. The resulting mixture was stirred at 0 °C for 10 min, and then the reaction was allowed to warm to 23 °C. After 24 h, the mixture was placed in a 0 °C bath and additional NaH (0.50 g, 20.8 mmol, 1.56 equiv), followed by TIPSCl (5.70 mL, 26.6 mmol, 1.99 equiv) were added. The reaction was allowed to warm to 23 °C and stirred for 18.5 h. The reaction was cooled to 0 °C and additional NaH (0.5 g, 20.8 mmol, 1.56 equiv) and TIPSCl (5.7 mL, 26.6 mmol, 1.99 equiv) were added. The reaction was allowed to warm to 23 °C. Following 23 h of stirring at 23 °C, the mixture was cooled to 0 °C and quenched with brine (60 mL). The biphasic mixture was further diluted with EtOAc (60 mL) and H_2O (60 mL). The layers were separated, and then the aqueous layer was extracted with EtOAc (2 × 60 mL). The combined organic layers were washed with washed brine (60 mL), H_2O (60 mL), then dried over $MgSO_4$. Evaporation of the solvent under reduced pressure afforded crude **90**, which was further purified by flash chromatography (100% Hexanes → 3:1 Hexanes:CH_2Cl_2) to afford **90** (4.65 g, 91% yield). R_f 0.74 (3:1 Hexanes:EtOAc); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.50 (d, J = 8.5, 1H), 7.46 (d, J = 7.0, 2H), 7.38 (app. t, J = 8.0, 2H), 7.33–7.30 (m, 1H), 7.13 (d, J = 3.5, 1H), 7.00 (d, J = 2.0, 1H), 6.89 (dd, J = 8.5, 2.0, 1H), 6.54 (d, J = 3.5, 1H), 5.13 (s, 2H), 1.58 (septet, J = 7.5, 3H), 1.09 (d, J = 7.0, 18H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.1, 141.6, 138.0, 130.3, 128.7, 127.8, 127.3, 126.0, 120.8, 110.4, 104.5, 100.0, 71.1, 18.2, 12.9; IR (film): 2948, 2868, 1416, 1207 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calcd for $C_{24}H_{33}NOSiNa$, 402.2229; found, 402.2233.

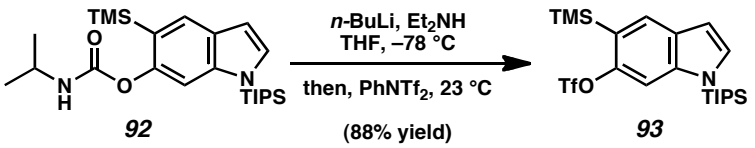


***N*-TIPS carbamate 91.** To a solution of *N*-TIPS benzyloxyindole **90** (4.65 g, 12.2 mmol) in 1:1:1 *i*-PrOH:Hexanes:EtOAc (45 mL) was added 10% Pd/C (1.29 g, 1.22 mmol, 10 mol% Pd). The mixture was placed under an atmosphere of hydrogen (double-balloon), stirred for 25.5 h at 23 °C, at which point was added additional 10% Pd/C (2.07 g, 1.95 mmol, 15 mol% Pd). After stirring for another 48 h at 23 °C was added additional 10% Pd/C (1.16 g, 1.09 mmol, 9 mol% Pd). The reaction was stirred for 5 h at 23 °C, and then filtered over celite (EtOAc eluent). Evaporation of the solvent under reduced pressure afforded crude **SI-4** as a light brown oil, which was used in the subsequent step without further purification. R_f 0.63 (3:1 Hexanes:EtOAc).

Carbamate **91** was prepared following the general procedure described by Igarashi.[11] Crude **SI-4** was dissolved in CH_2Cl_2 (61.0 mL), and Et_3N (0.85 mL, 6.1 mmol, 0.5 equiv), followed by *i*-PrNCO (1.41 mL, 14.4 mmol, 2.0 equiv), was added. The solution was stirred at 23 °C for 4 h, and then additional *i*-PrNCO (1.41 mL, 14.4 mmol, 2.0 equiv) was added. The solution continued to be stirred for 11 h, and then additional *i*-PrNCO (0.70 mL, 7.1 mmol, 0.58 equiv) was added. After stirring at 23 °C for 3 h, the mixture was concentrated to dryness under reduced pressure. Purification by flash chromatography (9:1 Hexanes:EtOAc) provided *N*-TIPS carbamate **91** (3.74 g, 82% yield, 2 steps). R_f 0.63 (2:1 Hexanes:Et_2O); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.56 (d, *J* = 8.5, 1H), 7.24 (d, *J* = 3.5, 1H), 6.91 (dd, *J* = 8.5, 2.5, 1H), 6.60 (dd, *J* = 3.0, 0.5, 1H), 4.82 (d, *J* = 6.0, 1H), 3.95–3.91 (m, 1H), 1.68 (septet, *J* = 7.5, 3H), 1.24 (d, *J* = 6.0, 6H), 1.15 (d, *J* = 7.5, 18H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.4, 146.6, 140.8, 131.8, 129.2, 120.6, 114.5, 107.0, 104.8, 43.5, 23.2, 18.4, 13.0; IR (film): 3324, 2950, 2867, 1735, 1470 cm^{-1}; HRMS-ESI (*m*/*z*) $[M + Na]^+$ calcd for $C_{21}H_{34}N_2O_2SiNa$, 397.2287; found, 397.2286.

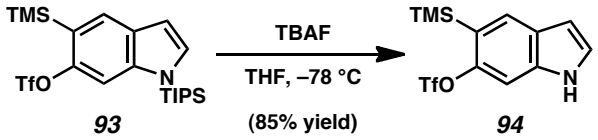


***N*-TIPS silylcarbamate 92.** *N*-TIPS silyl carbamate **92** was prepared following the general procedure described by Hoppe and Snieckus for *o*-lithiation of isopropyl carbamates, with minor modifications.[6,7] To a solution of *N*-TIPS carbamate **91** (0.659 g, 1.76 mmol) and TMEDA (0.394 mL, 2.47 mmol, 1.4 equiv) in 3:1 Et_2O:THF (16.9 mL) at –78 °C was added a solution of TBSOTf in *n*-pentane (1.30 M, 1.61 mL, 2.11 mmol, 1.2 equiv). After stirring for 5 min, the white suspension was allowed to warm to 23 °C over 35 min. TMEDA (0.987 mL, 6.16 mmol, 3.5 equiv) was added, and the mixture was cooled to –78 °C. A solution of *n*-BuLi in hexanes (1.54 M, 4.00 mL, 6.16 mmol, 3.5 equiv) was added dropwise over 20 min. The mixture was stirred at –78 °C for 3 h, then neat TMSCl (1.56 mL, 12.3 mmol, 7 equiv) was added dropwise over 16 min. The resulting mixture was stirred at –78 °C for 1 h, quenched with 1 M $NaHSO_4$ (10 mL), and allowed to warm to 23 °C over 45 min with vigorous stirring. The organic layer was separated, washed successively with 1 M $NaHSO_4$ (10 mL) and brine (10 mL), then dried over $MgSO_4$. Evaporation under reduced pressure afforded the crude product, which was purified by flash chromatography (10:1 Hexanes:Acetone) to afford *N*-TIPS silylcarbamate **92** (0.570 g, 73% yield). R_f 0.67 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.69 (s, 1H), 7.25 (s, 1H), 7.23 (d, *J* = 3.5, 1H), 6.61 (dd, *J* = 3.0, 0.5, 1H), 4.70 (d, *J* = 8.0, 1H), 3.99–3.93 (m, 1H), 1.69 (septet, *J* = 7.5, 3H), 1.24 (d, *J* = 6.5, 6H), 1.15 (d, *J* = 8.0, 18H), 0.32 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.3, 151.1, 142.0, 131.8, 129.4, 126.7, 122.9, 107.9, 104.8, 43.5, 23.3, 18.3, 12.9, –0.3; IR (film): 3331, 2945, 2868, 1704 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{24}H_{42}N_2O_2Si_2Na$, 469.2682; found, 469.2677.

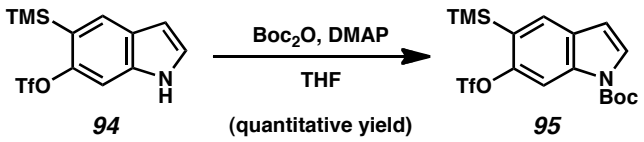


***N*-TIPS silyltriflate 93.** To a solution of *N*-TIPS silylcarbamate **92** (0.468 g, 1.05 mmol) in THF (10.5 mL) at –78 °C were added *n*-BuLi in hexanes (2.91 M, 0.432 mL, 1.26 mmol, 1.2 equiv)

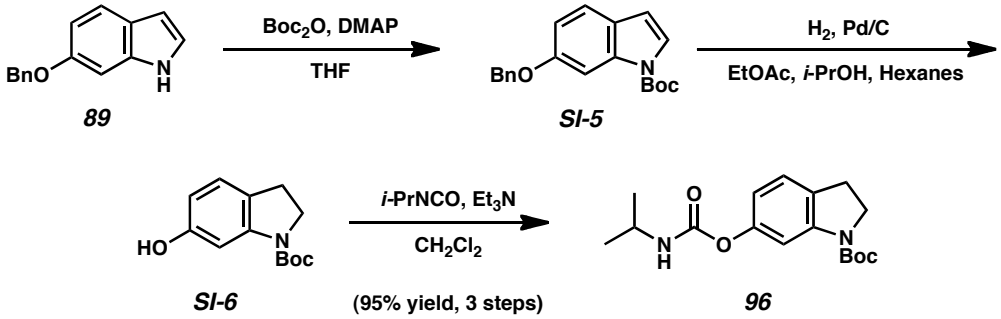
and Et_2NH (0.130 mL, 1.26 mmol, 1.2 equiv). The resulting mixture was stirred at –78 °C for 15 min, then allowed to warm to 23 °C over an additional 30 min. Next, $PhNTf_2$ (0.449 g, 1.26 mmol, 1.2 equiv) was added as a solid in a single portion. After stirring for 1 h, the reaction mixture was passed over a plug of silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (98:2 Hexanes:Benzene) to provide silyltriflate **93** (0.456 g, 88% yield) as a white solid. R_f 0.79 (9:1 Hexanes:Et_2O); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.74 (s, 1H), 7.57 (s, 1H), 7.31 (d, J = 3.0, 1H), 6.65 (d, J = 3.0, 1H), 1.67 (septet, J = 2.5, 3H), 1.15 (d, J = 7.5, 18H), 0.40 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.2, 141.5, 133.4, 130.8, 127.7, 122.5, 118.8 (q, J = 318, CF_3), 105.4, 105.0, 18.1, 12.8, –0.3; IR (film): 2955, 2866, 1417, 1138 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{21}H_{34}F_3NO_3SSi_2Na$, 516.1648; found, 516.1634.



***N*-H silyltriflate 94.** To a solution of *N*-TIPS silyltriflate **93** (0.440 g, 0.891 mmol) in THF (10 mL) at –78 °C was added a solution of TBAF in THF (1.0 M, 0.891 mL, 0.891 mmol, 1 equiv) dropwise. The solution was stirred for 10 min, diluted with Et_2O (5 mL), then quenched with H_2O (5 mL). The biphasic mixture was allowed to warm to 23 °C over 30 min. The layers were separated, and then the aqueous layer was extracted with Et_2O (3 × 10 mL). The combined organic layers were washed with brine (10 mL), then dried over $MgSO_4$. Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (3:1 Hexanes:Et_2O) to provide *N*-H silyltriflate **94** (285 mg, 85% yield). R_f 0.21 (4:1 Hexanes:Et_2O); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.28 (br s, 1H), 7.80 (s, 1H), 7.44 (s, 1H), 7.24 (dd, J = 3.1, 2.5, 1H), 6.59–6.58 (m, 1H), 0.44 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.0, 136.0, 128.0, 127.2, 126.2, 122.5, 118.5 (q, J = 318, CF_3), 102.7, 102.4, –0.5; IR (film): 3447, 2958, 1208 cm^{-1}; HRMS-ESI (*m*/*z*) [M + H]$^+$ calcd for $C_{12}H_{15}F_3NO_3SSi$, 338.0494; found, 338.0490.



***N*-Boc silyltriflate 95.** *N*-Boc silyltriflate **95** was prepared following the general procedure described by Gribble for Boc-protection of indoles.[12] To a stirred solution of *N*-H silyltriflate **94** (0.200 g, 0.59 mmol) in THF (6 mL) were added Boc_2O (0.142 g, 0.65 mmol, 1 equiv) and then DMAP (7.2 mg, 0.059 mmol, 0.1 equiv). The resulting mixture was stirred at 23 °C for 14 h, then additional Boc_2O (70 mg, 0.32 mmol, 0.54 equiv) was added. After stirring at 23 °C for an additional 2 h, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded *N*-Boc silyltriflate **95** (0.261 g, quantitative yield). R_f 0.57 (9:1 Hexanes:Et_2O); ^{1}H NMR (500 MHz, 40 °C, $CDCl_3$): δ 8.20 (s, 1H), 7.692–7.687 (m, 2H), 6.58 (d, J = 3.7, 1H), 1.69 (s, 9H), 0.41 (s, 9H); ^{13}C NMR (125 MHz, 40 °C, $CDCl_3$): δ 152.2, 149.1, 135.5, 129.6, 127.9, 127.7, 125.9, 118.5 (q, J = 318, CF_3), 106.9, 106.7, 84.7, 28.0, –0.8; IR (film): 2983, 1740, 1140 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calcd for $C_{17}H_{22}F_3NO_5SSiNa$, 460.0838; found, 460.0842.

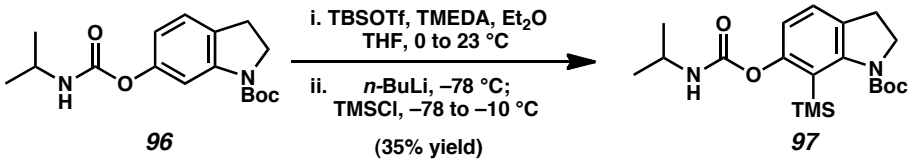


***N*-Boc carbamate 96.** *N*-Boc benzyloxyindole **SI-5** was prepared following the general procedure described by Gribble for Boc-protection of indoles.[12] To a stirred solution of benzyloxyindole **89** (5.00 g, 22.4 mmol) in THF (223 mL) were added Boc_2O (5.38 g, 24.6 mmol, 1.1 equiv) and DMAP (0.274 g, 2.24 mmol, 0.1 equiv). The resulting mixture was stirred at 23 °C for 2 h, then filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure

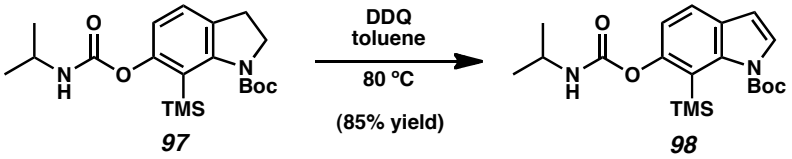
afforded crude **SI-5**, which was used in the subsequent step without further purification. R_f 0.49 (9:1 Hexanes:Et_2O); ^{1}H NMR (500 MHz, 40 °C, $CDCl_3$): δ 7.86 (s, 1H), 7.48–7.46 (m, 3H), 7.42 (d, *J* = 8.5, 1H), 7.39 (app. t, *J* = 7.7, 2H), 7.32 (app. t, *J* = 7.4, 1H), 6.95 (dd, *J* = 8.5, 2.3, 1H), 6.49 (d, *J* = 3.7, 1H), 5.15 (s, 2H), 1.67 (s, 9H); ^{13}C NMR (125 MHz, 40 °C, $CDCl_3$): δ 156.9, 149.7, 137.2, 136.1, 128.3, 127.7, 127.4, 124.6, 124.5, 121.1, 112.7, 106.9, 100.8, 83.3, 70.5, 28.1; IR (film): 2979, 2917, 1729, 1343, 1151 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calcd for $C_{20}H_{21}NO_3Na$, 346.1419; found, 346.1422.

Crude **SI-5** was dissolved in 1:1:1 *i*-PrOH:Hexanes:EtOAc (120 mL), then 10% Pd/C (2.38 g, 2.24 mmol, 10 mol% Pd) was added. The mixture was placed under an atmosphere of hydrogen (double-balloon), stirred for 14 h at 23 °C, and then filtered over celite (EtOAc eluent, 250 mL). Evaporation of the solvent under reduced pressure afforded crude **SI-6** as a light brown oil, which was used in the subsequent step without further purification. R_f 0.35 (4:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, 40 °C, $CDCl_3$): δ 7.36 (br s, 1H), 6.95 (d, *J* = 8.0, 1H), 6.43 (d, *J* = 8.0, 1H), 5.47 (br s, 1H), 3.97 (dd, *J* = 8.5, 8.3, 2H), 2.99 (dd, *J* = 8.7, 8.4, 2H), 1.56 (s, 9H); ^{13}C NMR (125 MHz, 40 °C, $CDCl_3$): δ 155.5, 152.6, 143.6, 124.9, 122.8, 108.9, 102.9, 80.9, 48.4, 28.2, 26.4; IR (film): 3329, 2989, 1657 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calcd for $C_{13}H_{17}NO_3Na$, 258.1106; found, 258.1110.

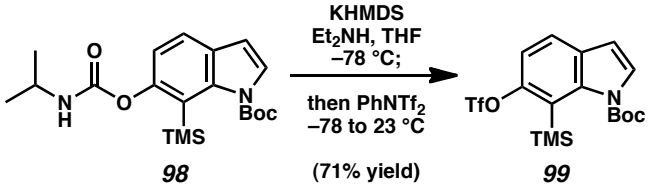
Carbamate **96** was prepared following the general procedure described by Igarashi et al.[11] To a solution of crude **SI-6** in CH_2Cl_2 (224 mL) was added subsequently *i*-PrNCO (3.30 mL, 33.6 mmol, 1.5 equiv) and Et_3N (0.936 mL, 6.72 mmol, 0.3 equiv). The solution was stirred at 23 °C for 18 h, then concentrated to dryness under reduced pressure. Purification by flash chromatography (7:3 Hexanes:EtOAc) provided *N*-Boc carbamate **96** (6.83 g, 95% yield over 3 steps). R_f 0.25 (3:2 Hexanes:Et_2O); ^{1}H NMR (500 MHz, 40 °C, $CDCl_3$): δ 7.54 (br s, 1H), 7.05 (d, *J* = 7.9, 1H), 6.68 (d, *J* = 7.8, 1H), 4.79 (br s, 1H), 3.98 (app. t, *J* = 8.3, 2H), 3.88 (d, *J* = 5.9, 1H), 3.03 (app. t, *J* = 8.6, 2H), 1.55 (s, 9H), 1.22 (d, *J* = 5.4, 6H); ^{13}C NMR (125 MHz, 40 °C, $CDCl_3$): δ 153.8, 152.2, 150.6, 143.4, 127.8, 124.4, 115.1, 108.9, 80.8, 48.2, 43.3, 28.3, 26.7, 22.8; IR (film): 3348, 2977, 1732, 1681 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calcd for $C_{17}H_{24}N_2O_4Na$, 343.1634; found, 343.1632.



***N*-Boc silylcarbamate indoline 97.** *N*-Boc silylcarbamate **97** was prepared following the general procedure described by Hoppe and Snieckus for *o*-lithiation of isopropyl carbamates, with modifications.[6,7] To a solution of *N*-Boc carbamate **96** (3.70 g, 11.5 mmol) and TMEDA (1.32 mL, 16.2 mmol, 1.4 equiv) in 3:1 Et_2O:THF (116 mL) at 0 °C was added a solution of TBSOTf in *n*-pentane (1.30 M, 10.7 mL, 13.9 mmol, 1.2 equiv). After stirring for 5 min, the mixture was allowed to warm to 23 °C over 30 min. TMEDA (3.12 mL, 38.1 mmol, 3.3 equiv) was added, and the mixture was cooled to –78 °C. A solution of *n*-BuLi in hexanes (1.60 M, 23.8 mL, 38.1 mmol, 3.3 equiv) was added dropwise over 45 min. The mixture was stirred at –78 °C for 4 h, then neat TMSCl (10.3 mL, 80.8 mmol, 7 equiv) was added. The resulting mixture was stirred at –78 °C for 1 h, then stirred at –10 °C for 14 h. The reaction mixture was cooled to –78 °C, then quenched with 1 M $NaHSO_4$ (90 mL) and allowed to warm to 23 °C over 1 h with vigorous stirring. The organic layer was separated, washed successively with 1 M $NaHSO_4$ (50 mL) and brine (50 mL), then dried over $MgSO_4$. Evaporation under reduced pressure afforded the crude product, which was purified by flash chromatography (85:15 Hexanes:Acetone) to afford *N*-Boc silylcarbamate **97** (1.58 g, 35% yield). R_f 0.29 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, 40 °C, $CDCl_3$): δ 7.13 (d, *J* = 7.9, 1H), 6.83 (d, *J* = 7.9, 1H), 4.72 (br s, 1H), 4.05 (t, *J* = 7.7, 2H), 3.93 (br s, 1H), 2.91 (t, *J* = 7.7, 2H), 1.52 (s, 9H), 1.24 (d, *J* = 6.5, 6H), 0.30 (s, 9H); ^{13}C NMR (125 MHz, 40 °C, $CDCl_3$): δ 155.1, 155.0, 153.7, 148.8, 129.8, 124.8, 122.5, 118.0, 80.4, 50.1, 43.2, 28.7, 28.3, 22.8, 0.3; IR (film): 3343, 2976, 2245, 1722, 1683 cm^{-1}; HRMS-ESI (*m*/*z*) $[M + Na]^+$ calcd for $C_{20}H_{32}N_2O_4SiNa$, 415.2029; found, 415.2028.

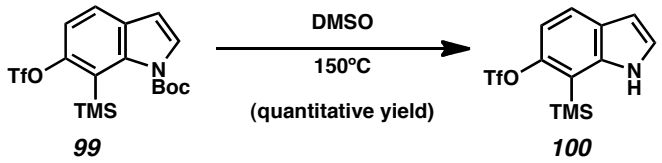


***N*-Boc silyltriflate indole 98.** To a stirred solution of *N*-Boc silyltriflate indoline **97** (1.11 g, 2.83 mmol) in toluene (28 mL) was added DDQ (0.770 g, 3.39 mmol, 1.2 equiv). The reaction vessel was placed in an oil bath maintained at 80 °C for 2.5 h. After cooling to 23 °C, the reaction mixture was filtered over neutral Brockman Grade I 58 Å activated alumina (packed with Hexanes; EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (7:3 Hexanes:Et_2O) to provide **98** (0.933 g, 85% yield) as a white solid. R_f 0.56 (7:3 Hexanes:Et_2O); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.49 (d, J = 8.3, 1H), 7.43 (d, J = 3.8, 1H), 7.05 (d, J = 8.4, 1H), 6.51 (d, J = 3.7, 1H), 4.83 (d, J = 7.9, 1H), 3.98–3.91 (m, 1H), 1.60 (s, 9H), 1.25 (d, J = 6.5, 6H), 0.36 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.1, 154.0, 150.5, 140.8, 128.11, 128.08, 121.9, 119.7, 118.2, 107.8, 83.1, 43.2, 28.1, 22.9, 1.4; IR (film): 2978, 2250, 1737, 1153 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{20}H_{30}N_2O_4SiNa$, 413.1873; found, 413.1873.

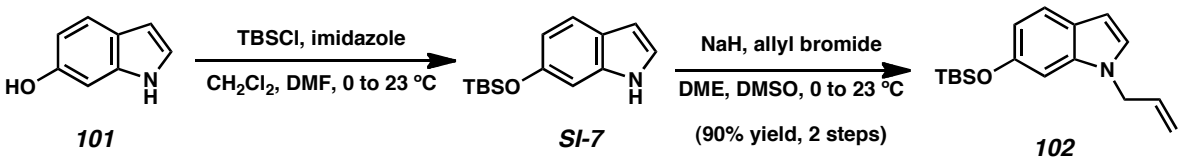


***N*-Boc silyltriflate 99.** To a solution of *N*-Boc silylcarbamate **98** (1.05 g, 2.69 mmol) in THF (27 mL) at –78 °C was added Et_2NH (0.334 mL, 3.23 mmol, 1.2 equiv), followed by a solution of KHMDS in toluene (0.5 M, 6.45 mL, 3.23 mmol, 1.2 equiv) dropwise over 10 min. The reaction was stirred at –78 °C for 20 min, then additional KHMDS in toluene (0.5 M, 1.61 mL, 0.805 mmol, 0.30 equiv), followed by Et_2NH (83 μL, 0.805 mmol, 0.30 equiv), was added. After stirring for 20 min at –78 °C, a solution of $PhNTf_2$ (1.44 g, 4.03 mmol, 1.5 equiv) in THF (8 mL) was added. After stirring for 1 h, the reaction mixture was allowed to warm to 23 °C. The reaction stirred for an additional 14 h at 23 °C, then was passed over a plug of silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further

purified by flash chromatography (85:15 Hexanes:CH_2Cl_2) to provide *N*-Boc silyltriflate **99** (0.830 g, 71% yield). R_f 0.46 (9:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.57–7.54 (m, 2H), 7.17 (d, *J* = 8.5, 1H), 6.56 (d, *J* = 3.8, 1H), 1.62 (s, 9H), 0.43 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.5, 149.9, 140.7, 130.0, 129.6, 122.7, 122.4, 118.5 (q, *J* = 318, CF_3), 116.3, 107.4, 84.0, 28.0, 1.5; IR (film): 2985, 1737, 1199 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{17}H_{22}F_3NO_5SSiNa$, 460.0838; found, 460.0838.



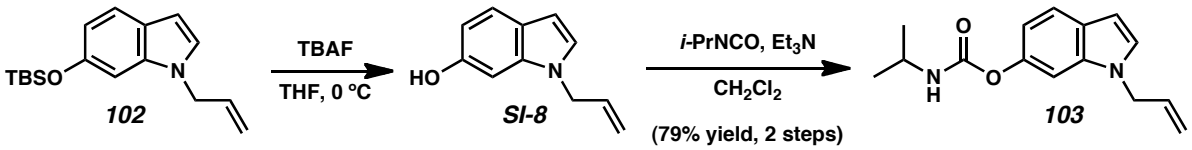
***N*-H silyltriflate 100.** A solution of *N*-Boc silyltriflate **99** (0.234 g, 0.53 mmol) in DMSO (5.3 mL) was stirred at 150 °C for 1 h. After cooling to 23 °C, the reaction mixture was poured into a 1:1 solution of Et_2O:H_2O (15 mL). The layers were separated and the aqueous layer was further diluted with H_2O (10 mL). The aqueous layer was extracted with Et_2O, followed by EtOAc (2 × 20 mL). The combined organic layers were washed successively with H_2O (20 mL) and brine (20 mL), then dried over $MgSO_4$. Evaporation under reduced pressure afforded the crude product, which was purified by flash chromatography (9:1 Hexanes: Et_2O) to afford *N*-H silyltriflate **100** (0.180 g, quantitative yield). R_f 0.29 (6:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.52 (br s, 1H), 7.70 (d, *J* = 8.7, 1H), 7.31 (app. t, *J* = 2.9, 1H), 7.17 (d, *J* = 8.7, 1H), 6.61 (dd, *J* = 3.2, 2.0, 1H), 0.59 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.1, 139.5, 126.3, 126.0, 123.0, 118.6 (q, *J* = 318, CF_3), 113.1, 112.4, 102.5, 0.7; IR (film): 3489, 2957, 1410, 1208 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{12}H_{14}F_3NO_3SSiNa$, 360.0313; found, 360.0312.



***N*-Allyl silylether 102.** To a solution of hydroxyindole **101** (1.60 g, 12.0 mmol) in CH_2Cl_2 (120 mL) and DMF (12 mL) at 0 °C was added imidazole (2.05 g, 30.1 mmol, 2.5 equiv) and TBSCl (2.18 g, 14.4 mmol, 1.2 equiv). After stirring for 10 min at 0 °C, the mixture was warmed to 23 °C and allowed to stir for an additional 80 min. The reaction mixture was quenched with H_2O (80 min), and stirred for 1 min. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3 × 30 mL). The combined organic layers were washed with brine (20 mL), then dried over Na_2SO_4. Evaporation under reduced pressure afforded the crude product, which was purified by flash chromatography (9:1 Hexanes:EtOAc) to afford *N*-H silylether **SI-7** as a white solid, which was used directly in the subsequent step. R_f 0.29 (6:1 Hexanes:EtOAc); **SI-7:** ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.93 (br s, 1H), 7.53 (d, *J* = 8.5, 1H), 7.06 (dd, *J* = 3.1, 2.4, 1H), 6.873–6.870 (m, 1H), 6.79 (dd, *J* = 8.5, 2.1, 1H), 6.53–6.52 (m, 1H), 1.10 (s, 9H), 0.29 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.6, 136.5, 123.3, 122.7, 120.8, 114.4, 102.3, 101.7, 25.8, 18.2, –4.4; IR (film): 3400, 2955, 2929, 2887, 2858, 1621, 1252 cm^{-1}; HRMS-ESI (*m*/*z*) [M + H]$^+$ calcd for $C_{14}H_{22}NOSi$, 248.1471; found, 248.1474.

To a solution of *N*-H silyl ether **SI-7** in 1,2-dimethoxyethane (120 mL) and DMSO (12 mL) at 0 ºC was added 60% NaH (1.36 g, 34.0 mmol, 2.8 equiv). The resulting solution was stirred at 0 °C for 20 min, then allyl bromide (1.5 mL, 17.0 mmol, 1.4 equiv) was added dropwise over 5 min. The mixture was stirred at 0 °C for 10 min, warmed to 23 °C over 90 min, then quenched with H_2O (30 mL). The biphasic mixture was stirred for an additional 10 min. The layers were separated, and then the aqueous layer was extracted with Et_2O (3 × 50 mL). The combined organic layers were washed with H_2O (30 mL), washed with brine (30 mL), then dried over Na_2SO_4. Evaporation of the solvent under reduced pressure afforded the crude product, which was purified by flash chromatography (97:3 Hexanes: Et_2O) to provide *N*-allyl silyl ether **102** (3.10 g, 90% yield over 2 steps) as a colorless oil. R_f 0.64 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.48 (d, *J* = 8.5, 1H), 7.01 (d, *J* = 3.1, 1H), 6.79 (d, *J* = 1.8, 1H), 6.72 (dd, *J* = 8.5, 2.1, 1H), 6.46 (d, *J* = 3.1, 1H), 6.04–5.97 (m, 1H), 5.22 (dd, *J* = 10.2, 1.3, 1H), 5.11 (dd,

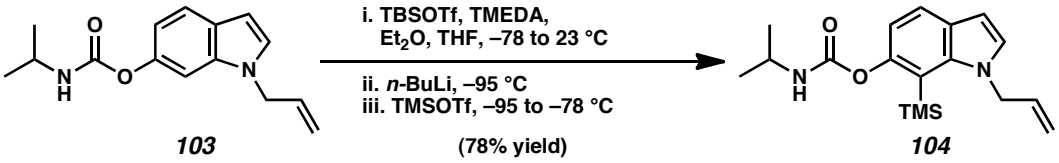
17.06, 1.4, 1H), 4.66 (dd, J = 3.8, 1.2, 2H), 1.05 (s, 9H), 0.24 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.4, 136.8, 133.3, 126.9, 123.4, 121.0, 117.1, 113.9, 101.1, 100.4, 48.8, 25.8, 18.2, –4.4; IR (film): 2955, 2930, 2858, 1620, 1486, 1263 cm^{-1}; HRMS-ESI (m/z) [M + H]$^+$ calcd for $C_{17}H_{26}NOSi$, 288.1784; found, 288.1776.



***N*-Allyl carbamate 103.** To a solution of *N*-allyl silyl ether **102** (4.06 g, 14.12 mmol) in THF (150 mL) at 0 °C was added a solution of TBAF in THF (1.0 M, 15.5 mL, 15.5 mmol, 1.1 equiv). The solution was stirred for 1 h, then quenched with H_2O (100 mL) and diluted with Et_2O (50 mL) allowed to warm to 23 °C over 60 min. The layers were separated, and then the aqueous layer was extracted with Et_2O (3 × 100 mL). The combined organic layers were washed with brine (50 mL), then dried over $MgSO_4$. Evaporation under reduced pressure afforded crude product **SI-8**, which was further purified by flash chromatography (4:1 Hexanes:EtOAc) and then used in the subsequent reaction. R_f 0.10 (9:1 Hexanes:Et_2O); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.46 (d, J = 8.4, 1H), 6.98 (d, J = 3.2, 1H), 6.76 (d, J = 2.2, 1H), 6.67 (dd, J = 8.4, 2.2, 1H), 6.44 (dd, J = 3.2, 0.7, 1H), 6.01–5.93 (m, 1H), 5.21 (dq, J = 10, 1.5, 1H), 5.08 (dq, J = 17.5, 1.5, 1H), 4.64–4.63 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.4, 136.9, 133.2, 126.9, 123.1, 121.4, 117.1, 109.3, 101.3, 95.4, 48.7; IR (film): 3338, 2919, 1624, 1180 cm^{-1}; HRMS-ESI (m/z) [M + H]$^+$ calcd for $C_{11}H_{12}NO$, 174.0919; found, 174.0916.

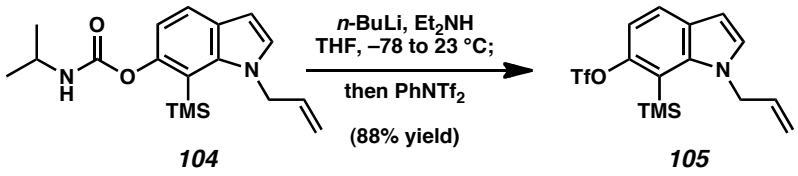
Crude **SI-8** was dissolved in CH_2Cl_2 (150 mL) and *i*-PrNCO (2.08 mL, 21.18 mmol, 1.5 equiv), followed by Et_3N (0.590 mL, 4.24 mmol, 0.3 equiv), was added. The solution was stirred at 23 °C for 16 h, then concentrated to dryness under reduced pressure. Purification by flash chromatography (99:1 DCM:Et_2O) provided *N*-allyl carbamate **103** (2.89 g, 79% yield over 2 steps) as a white solid. R_f 0.29 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.56 (d, J = 8.5, 1H), 7.12 (d, J = 1.3, 1H), 7.08 (d, J = 3.1, 1H), 6.88 (dd, J = 8.5, 1.8, 1H), 6.49 (d, J = 3.0, 1H), 6.02–5.94 (m, 1H), 5.21 (d, J = 10.2, 1H), 5.10 (d, J = 17.1, 1H), 4.87 (br s, 1H), 4.68 (d, J

= 4.5, 2H), 3.95–3.90 (m, 1H), 1.24 (d, J = 6.3, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.3, 146.5, 136.0, 133.1, 128.2, 126.1, 121.0, 117.3, 114.0, 102.7, 101.4, 48.8, 43.3, 22.9; IR (film): 3302, 2975, 1736, 1702, 1239 cm^{-1}; HRMS-ESI (m/z) [M + Na]$^+$ calcd for $C_{15}H_{18}N_2O_2Na$, 281.1266; found, 281.1260.

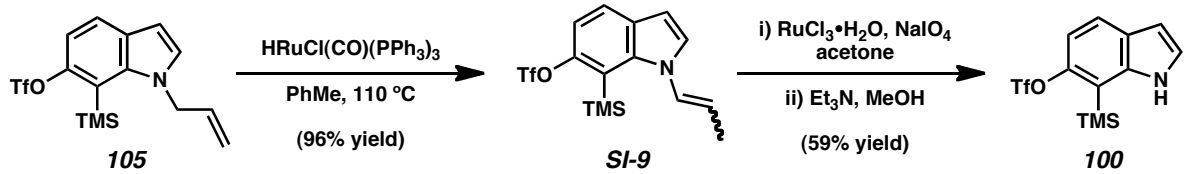


***N*-Allyl silylcarbamate 104.** *N*-allyl silylcarbamate **104** was prepared following the general procedure described by Hoppe and Snieckus for *o*-lithiation of isopropyl carbamates, with modifications.[6,7] To a solution of *N*-allyl carbamate **103** (1.45 g, 5.61 mmol) in 3:1 Et_2O:THF (60 mL) at –78 °C was added TMEDA (1.15 mL, 14.0 mmol, 2.5 equiv), followed by a solution of TBSOTf in *n*-pentane (1.30 M, 8.64 mL, 11.22 mmol, 2.0 equiv) dropwise over 10 minutes. The resulting mixture was stirred at –78 ºC for 5 minutes and allowed to warm to 23 °C over 30 min, by which time TMEDA·TfOH had formed as an oil on the bottom of the flask. TMEDA (1.89 mL, 22.45 mmol, 4 equiv) was added, and the mixture was cooled to –95 °C. A solution of *n*-BuLi in hexanes (1.43 M, 15.7 mL, 22.45 mmol, 4 equiv) was added dropwise over 60 min. The mixture was stirred at –95 °C for 4 h, then neat TMSOTf (7.11 mL, 39.29 mmol, 7.0 equiv) was added dropwise over 30 min. The resulting mixture was stirred at –95 °C for 5 min, stirred at –78 °C for 2 h, then quenched with 1 M aqueous $NaHSO_4$ (50 mL), and allowed to warm to 23 °C over 1 hour with vigorous stirring. The biphasic mixture was further diluted with Et_2O (50 mL). The organic layer was separated, washed successively with 1 M aqueous $NaHSO_4$ (50 mL) and brine (40 mL), then dried over $MgSO_4$. Evaporation under reduced pressure afforded the crude product, which was purified by flash chromatography (9:1 Hexanes:EtOAc) to afford *N*-allyl silylcarbamate **104** (1.45 g, 78% yield). R_f 0.53 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.58 (d, J = 8.4, 1H), 7.07 (d, J = 3.2, 1H), 6.83 (d, J = 8.4, 1H), 6.55 (d, J = 3.3, 1H), 5.88–5.81 (m, 1H), 5.14 (d, J = 10.2, 1H), 4.96 (d, J = 17.1, 1H), 4.79 (d, J = 5.2, 3H), 3.97–3.91 (m, 1H), 1.24 (d, J = 6.5, 6H), 0.46 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.5, 152.5, 141.9, 134.2, 130.1, 127.0, 123.0, 117.3, 115.2, 113.3. 103.6, 52.1, 43.3, 22.9, 2.4; IR (film): 3325,

2972, 1706, 1513, 1200, 1168 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{18}H_{26}N_2O_2SiNa$, 353.1661; found, 353.1659.



***N*-Allyl silyltriflate 105.** To a solution of *N*-allyl silylcarbamate **104** (1.08 g, 3.26 mmol) in THF (40 mL) at –78 °C were added dropwise over 10 min *n*-BuLi in hexanes (1.59 M, 2.4 mL, 3.84 mmol, 1.2 equiv), and then Et_2NH (405 μL, 3.91 mmol, 1.2 equiv). The resulting mixture was stirred at –78 °C for 25 min, then allowed to warm to 23 °C over an additional 30 min. Next, $PhNTf_2$ (1.42 g, 3.90 mmol, 1.2 equiv) was added as a solid in a single portion. After stirring for 1 h, the reaction mixture was passed over a plug of silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (9:1 Hexanes: Et_2O) to provide *N*-allyl silyltriflate **105** (1.00 g, 88% yield). R_f 0.65 (4:1 Hexanes:EtOAc); ^1H NMR (500 MHz, $CDCl_3$): δ 7.63 (d, *J* = 8.5, 1H), 7.18 (d, *J* = 3.2, 1H), 7.02 (d, *J* = 8.5, 1H), 6.60 (d, *J* = 3.2, 1H), 5.86–5.78 (m, 1H), 5.14 (dd, *J* = 10.8, 0.5, 1H), 4.93 (dd, *J* = 17.1, 0.5, 1H), 4.82 (d, *J* = 5.4, 2H), 0.54 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.0, 142.0, 133.3, 131.9, 128.7, 123.5, 118.6 (q, *J* = 319, CF_3), 117.8, 115.5, 113.6, 103.7, 52.3, 2.0; IR (film): 2959, 1408, 1210 cm^{-1}; HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{16}H_{18}F_3NO_3SSi$, 378.0807; found, 378.0800.



***N*-H silyltriflate 100.** To a solution of *N*-allyl silyltriflate **105** (0.210 g, 0.56 mmol) in toluene (5.5 mL) was added $HRuCl(CO)(PPh_3)_3$ (26.6 mg, 27.9 μmol, 0.05 equiv). The flask was topped with a reflux condenser and left open to the air. The flask was placed in a preheated oil bath

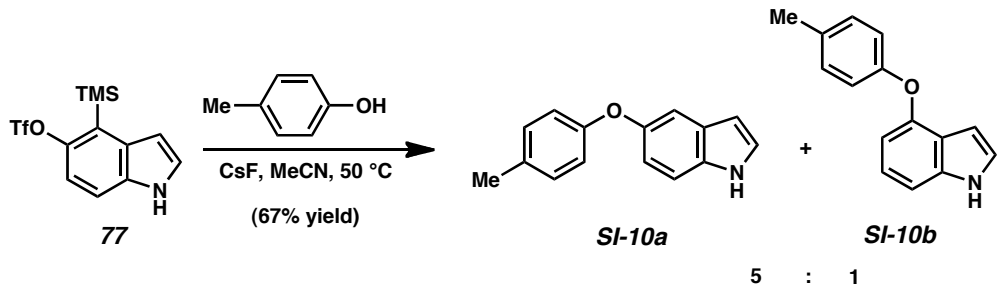
maintained at 120 ºC. After 55 min, the flask was removed from the oil bath. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (9:1 Hexane:Benzene) to provide the isomerized product **SI-9** (202.2 mg, 96% yield) as a mixture of isomers (E:Z = 3:1), which was used in the next step without further purification. R_f = 0.59 (9:1 Hexane:EtOAc).

To a solution of **SI-9** (31.3 mg, 0.083 mmol) in acetone (1.6 mL, filtered over activated, neutral alumina prior to use) were added a 0.035 M aqueous solution of $RuCl_3 \bullet H_2O$ (85 μL, 2.9 μmol, 0.035 equiv) and $NaIO_4$ (73.6 mg, 0.344 mmol, 4.1 equiv, added as 4 equal portions over 5 min). The reaction was stirred for 4 h and then Et_2O (1 mL) was added. After stirring for 1 min, the reaction was further diluted with a solution of saturated aqueous $Na_2S_2O_3$ (1 mL) and stirred for 10 min. The layers were separated, and the aqueous layer was extracted with Et_2O (3 x 2 mL). The combined organic layers were washed with H_2O (2 mL), and then concentrated under reduced pressure. The crude reaction mixture was redissolved in MeOH (1.5 mL), and Et_3N (2.5 μL, 18 μmol, 0.2 equiv) was added. After stirring for 30 min, the reaction was concentrated under reduced pressure to afford crude **100**, which was further purified by flash chromatography (9:1 Hexane:EtOAc) to provide *N*-H silyltriflate **100** (16.4 mg, 59% yield from **SI-9**, 68% based on recovered **SI-9**). Spectral data of **100** match those reported above **(SX)**.

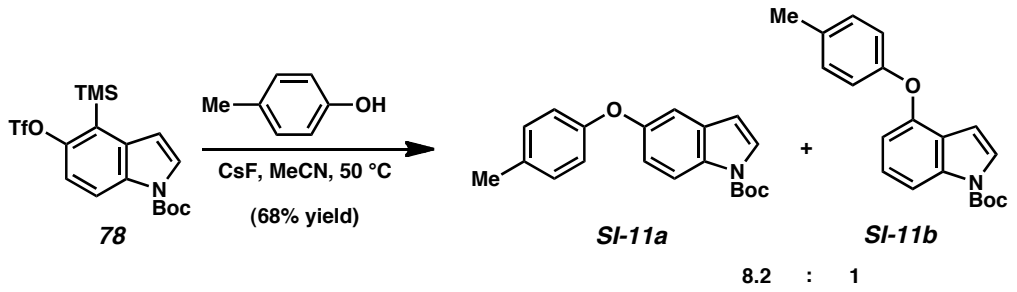
E. Regioselectivity in nucleophilic additions to indolynes

Supporting information for the products of the reactions of the *N*-Me- 4,5-, 5,6-, and 6,7-indolyne precursors with various nucleophiles has previously been reported in an earlier publication from our laboratory.[1,3]

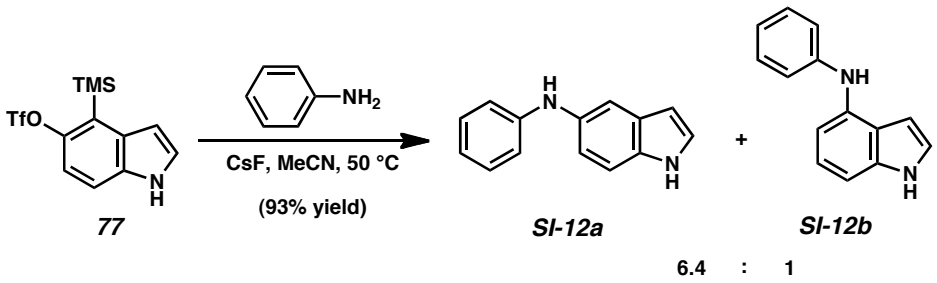
F. Influence of *N*-substituents on the Regioselectivity of indolyne reactions.



***N*-H Ethers SI-10a and SI-10b.** To a stirred solution of *N*-H silyltriflate **77** (40 mg, 0.12 mmol) and *p*-cresol (64 mg, 0.59 mmol, 5 equiv) in MeCN (1.6 mL) was added CsF (54 mg, 0.360 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (65:35 Hexanes:CH_2Cl_2) to provide a mixture of *N*-H ethers **SI-10a** and **SI-10b** (5:1 ratio of **SI-10a**:**SI-10b**, 17.6 mg, 67% yield). An analytical sample of **SI-10a** was obtained by preparative TLC (1:1 Hexanes:CH_2Cl_2). **SI-10a**: R_f 0.31 (1:1 Hexanes:CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.15 (br s, 1H), 7.36 (d, J = 8.7, 1H), 7.27–7.26 (m, 1H), 7.24 (app. t, J = 2.8, 1H), 7.09 (d, J = 8.3, 2H), 6.95 (dd, J = 8.7, 2.3, 1H), 6.90–6.87 (m, 2H), 6.50–6.49 (m, 1H), 2.31 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.8, 150.6, 132.5, 131.4, 129.9, 128.4, 125.2, 117.5, 115.4, 111.7, 110.6, 102.6, 20.5; IR (film): 3420, 3030, 2921, 1504 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{15}H_{13}NONa$, 246.0895; found, 246.0892. **SI-10b**: R_f 0.34 (1:1 Hexanes:CH_2Cl_2).

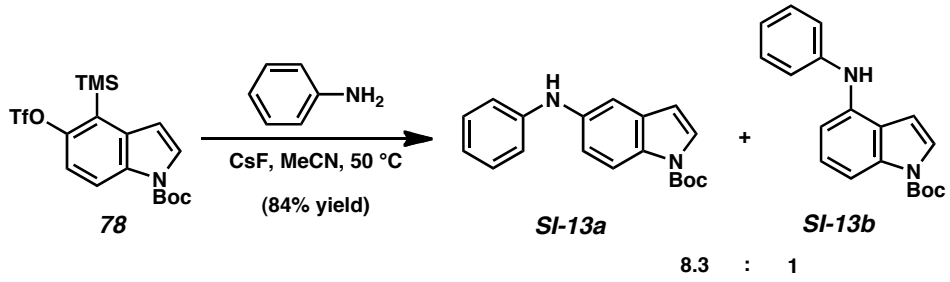


***N*-Boc Ethers SI-11a and SI-11b.** To a stirred solution of *N*-H silyltriflate **78** (20 mg, 0.046 mmol) and *p*-cresol (7.4 mg, 0.068 mmol, 1.5 equiv) in MeCN (1 mL) was added CsF (21 mg, 0.14 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by preparative TLC (1:1 Hexanes:CH_2Cl_2) to provide a mixture of *N*-Boc ethers **SI-11a** and **SI-11b** (8.2:1 ratio of **SI-11a**:**SI-11b**, 9.6 mg, 68% yield). An analytical sample of **SI-11a** was obtained by flash chromatography (85:15 Hexanes:CH_2Cl_2 → 4:1 Hexanes:CH_2Cl_2). **SI-11a**: R_f 0.57 (9:1 Hexanes:Et_2O); ^{1}H NMR (500 MHz, 40 °C, $CDCl_3$): δ 8.09 (d, *J* = 8.9, 1H), 7.59 (d, *J* = 3.7, 1H), 7.15 (d, *J* = 2.4, 1H), 7.11 (d, *J* = 8.3, 2H), 7.02 (dd, *J* = 8.9, 2.4, 1H), 6.92–6.89 (m, 2H), 6.48 (d, *J* = 3.6, 1H), 2.33 (s, 3H), 1.68 (s, 9H); ^{13}C NMR (125 MHz, 40 °C, $CDCl_3$, 15/16 C): δ 156.0, 153.1, 149.5, 132.0, 131.4, 129.9, 126.7, 118.1, 116.3, 115.9, 110.4, 106.9, 83.5, 28.1, 20.4; IR (film): 2979, 1734, 1464 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calcd for $C_{20}H_{21}NO_3Na$, 346.1419; found, 346.1417. **SI-11b**: R_f 0.57 (9:1 Hexanes:Et_2O).



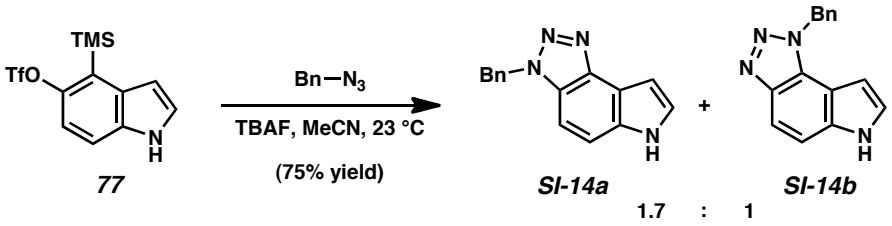
***N*-H Amines SI-12a and SI-12b.** To a stirred solution of *N*-H silyltriflate **77** (20 mg, 0.059 mmol) and aniline (27 μL, 0.30 mmol, 5 equiv) in MeCN (1 mL) was added CsF (27 mg, 0.18 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50

°C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (3:7 Hexanes:CH_2Cl_2) to provide a mixture of *N*-H amines **SI-12a** and **SI-12b** (6.4:1 ratio of **SI-12a**:**SI-12b**, 11.4 mg, 93% yield). An analytical sample of **SI-12a** was obtained by preparative TLC (100% CH_2Cl_2). **SI-12a**: R_f 0.40 (100% CH_2Cl_2); ^{1}H NMR (500 MHz, 40 °C, $CDCl_3$): δ 8.09 (br s, 1H), 7.44 (d, *J* = 1.9, 1H), 7.34 (d, *J* = 8.6, 1H), 7.23–7.20 (m, 3H), 7.04 (dd, *J* = 8.6, 2.1, 1H), 6.95 (d, *J* = 7.7, 2H), 6.82 (app. t, *J* = 7.3, 1H), 6.50–6.49 (m, 1H), 5.64 (br s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 146.1, 135.1, 132.6, 129.1, 128.5, 124.8, 118.9, 118.2, 115.2, 113.0, 111.5, 102.3; IR (film): 3403, 3043, 2918, 2850, 1712, 1598, 1496, 1307 cm^{-1}; HRMS-ESI (*m/z*) [M + H]$^+$ calcd for $C_{14}H_{13}N_2$, 209.1079; found, 209.1080. **SI-12b**: R_f 0.49 (100% CH_2Cl_2).

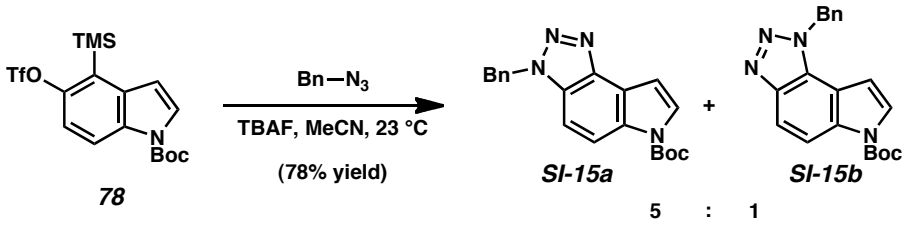


***N*-Boc Amines SI-13a and SI-13b.** To a stirred solution of *N*-Boc silyltriflate **78** (25 mg, 0.057 mmol) and aniline (26 μL, 0.29 mmol, 5 equiv) in MeCN (1 mL) was added CsF (26 mg, 0.17 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (9:1 Hexanes:Et_2O) to provide a mixture of *N*-Boc amines **SI-13a** and **SI-13b** (8.3:1 ratio of **SI-13a**:**SI-13b**, 14.7 mg, 84% yield). An analytical sample of **SI-13a** was obtained by preparative TLC (7:3 Hexanes:Et_2O). **SI-13a**: R_f 0.58 (3:1 Hexanes:Et_2O); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.05 (br s, 1H), 7.58 (s, 1H), 7.31 (d, *J* = 2.0, 1H), 7.26–7.23 (m, 2H), 7.08 (dd, *J* = 9.0, 2.0, 1H), 7.02–7.01 (m, 2H), 6.88 (app. t, *J* = 7.5, 1H), 6.48 (d, *J* = 3.5, 1H), 5.68 (br s, 1H), 1.68 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 149.6, 144.7, 137.9, 131.4, 130.9, 129.2, 126.4, 119.9, 117.9, 116.2, 115.7, 111.2, 106.9, 83.4, 28.1; IR (film): 3393, 2979,

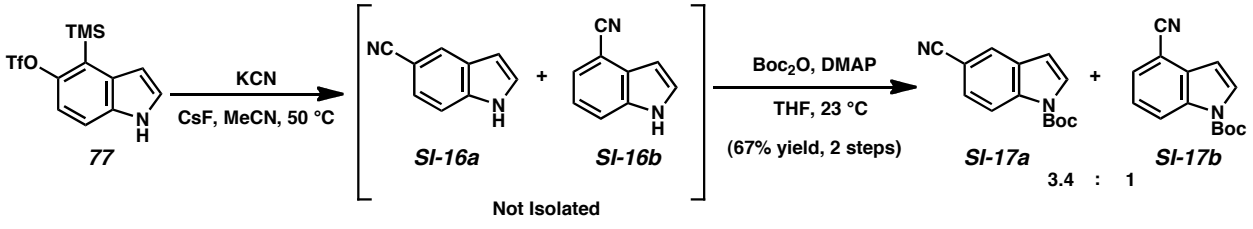
2936, 1730, 1371, 1126 cm^{-1}; HRMS-ESI (*m/z*) $[M + H]^+$ calcd for $C_{19}H_{21}N_2O_2$, 309.1603; found, 309.1596. **SI-13b**: R_f 0.58 (3:1 Hexanes:Et_2O).



***N*-H triazoles SI-14a and SI-14b.** To a stirred solution of *N*-H silyltriflate **77** (20 mg, 0.059 mmol) and benzyl azide[13] (39 mg, 0.30 mmol, 5 equiv) in MeCN (1 mL) at 23 °C was added a solution of TBAF in THF (1.0 M, 120 μL, 0.120 mmol, 2 equiv). The solution was stirred for 10 min, then filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (3:2 Hexanes:EtOAc → 1:1 Hexanes:EtOAc) to provide a mixture of *N*-H triazoles **SI-14a** and **SI-14b** (1.7:1 ratio of **SI-14a**:**SI-14b**, 11 mg, 75% yield). These compounds were characterized as a mixture. R_f 0.34 (1:1 Hexanes:EtOAc); **SI-14a**: ^{1}H NMR (500 MHz, DMSO): δ 11.65 (s, 1H), 7.56 (d, J = 8.5, 1H), 7.453–7.445 (m, 1H), 7.39–7.37 (m, 1H), 7.31–7.20 (m, 5H), 6.85 (s, 1H), 5.93 (s, 2H); **SI-14b**: ^{1}H NMR (500 MHz, DMSO): δ 11.76 (s, 1H), 7.61 (d, J = 9.0, 1H), 7.453–7.445 (m, 1H), 7.39–7.37 (m, 1H), 7.31–7.20 (m, 5H), 6.81 (s, 1H), 6.07 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 142.1, 140.4, 135.5, 135.1, 134.2, 132.1, 129.0, 128.82, 128.79, 128.1, 128.0, 127.8, 127.3, 126.9, 124.1, 123.2, 117.3, 113.5, 113.4, 110.5, 109.9, 103.4, 101.4, 100.8, 52.6, 52.3; IR (film): 3194, 3079, 2920, 1221 cm^{-1}; HRMS-ESI (*m/z*) $[M + Na]^+$ calcd for $C_{15}H_{12}N_4Na$, 271.0960; found, 271.0965.

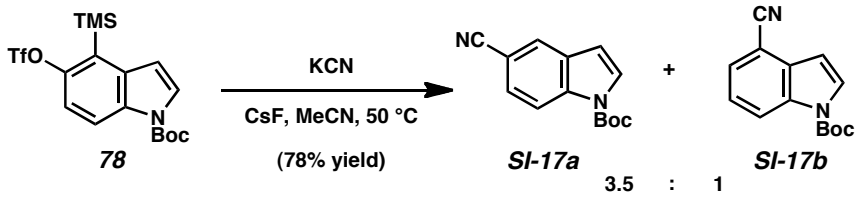


***N*-Boc triazoles SI-15a and SI-15b.** To a stirred solution of *N*-Boc silyltriflate **78** (20 mg, 0.046 mmol) and benzyl azide[13] (30 mg, 0.23 mmol, 5 equiv) in MeCN (1 mL) at 23 °C was added a solution of TBAF in THF (1.0 M, 90 μL, 0.090 mmol, 2 equiv). The solution was stirred for 10 min, then filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (7:3 Hexanes:Et_2O) to provide a mixture of *N*-Boc triazoles **SI-15a** and **SI-15b** (5:1 ratio of **SI-15a**:**SI-15b**, 12.4 mg, 78% yield). These compounds were characterized as a mixture. R_f 0.17 (3:2 Hexanes:Et_2O); **SI-15a**: ^{1}H NMR (500 MHz, 40 °C, $CDCl_3$): δ 8.36 (d, *J* = 9.0, 1H), 7.74 (d, *J* = 3.5, 1H), 7.33–7.26 (m, 5H), 7.21–7.20 (m, 2H), 5.89 (s, 2H), 1.68 (s, 9H); **SI-15b:** ^{1}H NMR (500 MHz, 40 °C, $CDCl_3$): δ 8.29 (d, *J* = 9.5, 1H), 7.94 (d, *J* = 9.5, 1H), 7.62 (d, *J* = 3.5, 1H), 7.33–7.26 (m, 5H), 6.58 (d, *J* = 3.5, 1H), 6.04 (s, 2H), 1.68 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 149.7, 149.5, 143.5, 140.2, 135.5, 135.1, 134.1, 132.0, 130.2, 129.2, 129.1, 128.5, 128.4, 127.6, 127.2, 126.9, 126.7, 126.0, 120.8, 116.6, 115.6, 113.6, 113.1, 105.4, 105.1, 104.1, 84.9, 84.5, 53.0, 52.6, 28.28, 28.25; IR (film): 2981, 1734, 1126 cm^{-1}; HRMS-ESI (*m*/*z*) [M + H]$^+$ calcd for $C_{20}H_{21}N_4O_2$, 349.1664; found, 349.1666.



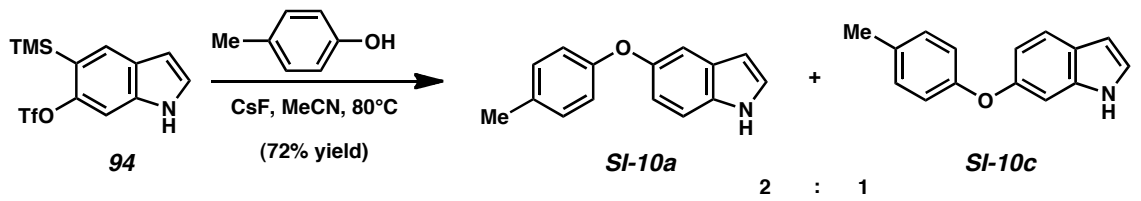
***N*-H cyanoindoles SI-16a and SI-16b.** To a stirred solution of *N*-H silyltriflate **77** (20 mg, 0.059 mmol) and potassium cyanide (5.8 mg, 0.089 mmol, 1.5 equiv) in MeCN (1 mL) was added CsF (27 mg, 0.18 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica

gel (EtOAc eluent). Evaporation under reduced pressure afforded a mixture of crude *N*-H cyanoindoles **SI-16a** and **SI-16b**. This crude mixture was dissolved in THF (1 mL) and treated with DMAP (1 mg, 0.0082 mmol, 0.14 equiv). The mixture was allowed to stir at 23 °C for 5 min, then Boc_2O (13 mg, 0.059 mmol, 1 equiv) was added. The resulting mixture was stirred at 23 °C for an additional 14 h, then filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (95:5 Hexanes:Et_2O) and then preparative TLC (4:1 Hexanes:Et_2O) to provide *N*-Boc cyanoindoles **SI-17a and SI-17b** (5.7 mg and 1.7 mg, respectively; 3.4:1 ratio of **SI-17a:SI-17b**, 67% combined yield). Spectral data of **SI-17a** and **SI-17b** match those reported below (S39).

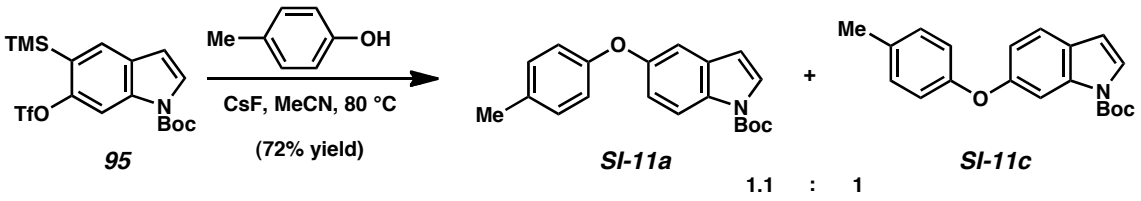


***N*-Boc cyanoindoles SI-17a and SI-17b.** To a stirred solution of *N*-Boc silyltriflate **78** (20 mg, 0.046 mmol) and potassium cyanide (4.5 mg, 0.069 mmol, 1.5 equiv) in MeCN (1 mL) was added CsF (21 mg, 0.14 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by preparative TLC (4:1 Hexanes:Et_2O) to provide *N*-Boc cyanoindoles **SI-17a** and **SI-17b** (6.7 mg and 1.9 mg, respectively; 3.5:1 ratio of **SI-17a**:**SI-17b**, 78% combined yield). **SI-17a:** R_f 0.30 (9:1 Hexanes:Et_2O); ^{1}H NMR (500 MHz, 40 °C, $CDCl_3$): δ 8.25 (d, *J* = 8.3, 1H), 7.90 (d, *J* = 1.0, 1H), 7.70 (d, *J* = 3.6, 1H), 7.56 (dd, *J* = 8.6, 1.5, 1H), 6.63 (d, *J* = 3.7, 1H), 1.68 (s, 9H); ^{13}C NMR (125 MHz, 40 °C, $CDCl_3$): δ 148.9, 137.0, 130.4, 128.0, 127.2, 125.7, 119.7, 115.9, 106.8, 106.0, 84.8, 28.0; IR (film): 2930, 2226, 1741, 1365 cm^{-1}; HRMS-ESI (*m*/*z*) $[M + Na]^+$ calcd for $C_{14}H_{14}N_2O_2Na$, 265.0953; found, 265.0956. **SI-17b:** R_f 0.34 (9:1 Hexanes:Et_2O); ^{1}H NMR (500 MHz, 40 °C, $CDCl_3$): δ 8.40 (d, *J* = 8.4, 1H), 7.74 (d, *J* = 3.7, 1H), 7.54 (d, *J* = 7.5, 1H), 7.35 (app. t, *J* = 8.1, 1H), 6.78 (d, *J* = 3.7, 1H), 1.69 (s, 9H);

^{13}C NMR (125 MHz, 40 °C, $CDCl_3$): δ 148.9, 135.1, 132.2, 128.3, 127.0, 123.9, 119.6, 117.5, 105.3, 103.8, 84.7, 28.0; IR (film): 2980, 2225, 1726, 1120 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{14}H_{14}N_2O_2Na$, 265.0953; found, 265.0948.

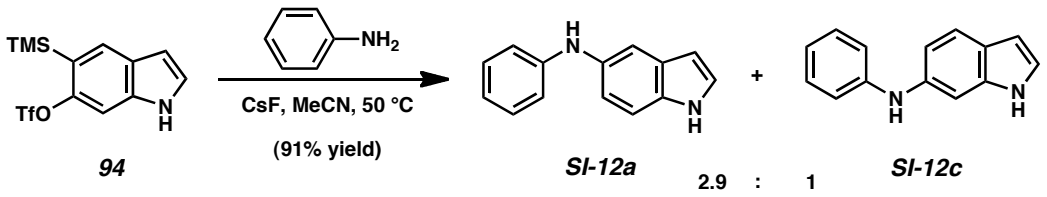


***N*-H ethers SI-10a and SI-10c.** To a stirred solution of *N*-H silyltriflate **94** (20 mg, 0.059 mmol) and *p*-cresol (32 mg, 0.30 mmol, 5 equiv) in MeCN (1 mL) was added CsF (27 mg, 0.18 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 80 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (7:3 Hexanes:CH_2Cl_2) to provide a mixture of *N*-H ethers **SI-10a** and **SI-10c** (2:1 ratio of **SI-10a**:**SI-10c**, 8.9 mg, 72% yield). Spectral data of **SI-10a** and **SI-10c** match those reported above (S34) and below (S45), respectively.

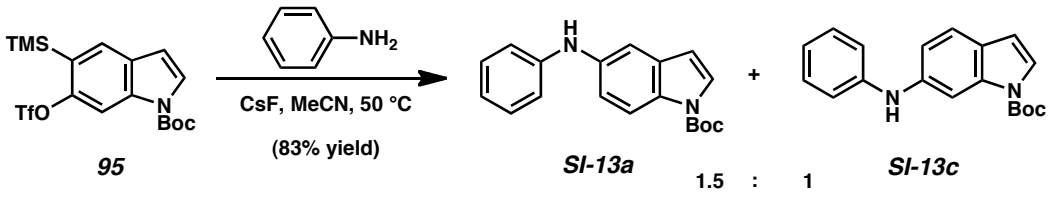


***N*-Boc ethers SI-11a and SI-11c.** To a stirred solution of *N*-Boc silyltriflate **95** (20 mg, 0.046 mmol) and *p*-cresol (7.4 mg, 0.069 mmol, 1.5 equiv) in MeCN (1 mL) was added CsF (21 mg, 0.14 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained 80 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (3:1 Hexanes:CH_2Cl_2) to provide a mixture of *N*-Boc ethers **SI-**

11a and **SI-11c** (1.1:1 ratio of **SI-11a**:**SI-11c**, 10.7 mg, 71% yield). Spectral data of **SI-11a** and **SI-11c** match those reported above (S35) and below (S46), respectively.

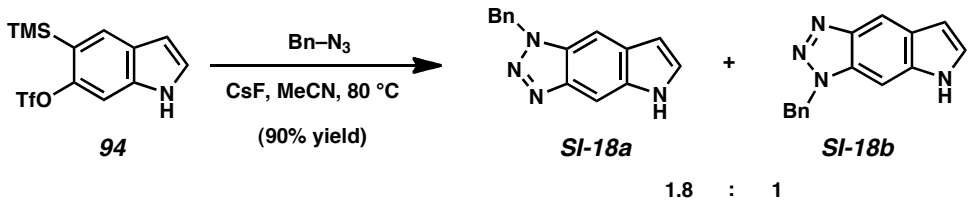


***N*-H amines SI-12a and SI-12c.** To a stirred solution of *N*-H silyltriflate **94** (21.4 mg, 0.0634 mmol) and aniline (24.9 μL, 0.317 mmol, 5 equiv) in MeCN (1.26 mL) was added CsF (28.9 mg, 0.190 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 80 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (100% Benzene) to provide a mixture of *N*-H amines **SI-12a** and **SI-12c** (2.9:1 ratio of **SI-12a**:**SI-12c**, 12.0 mg, 91% yield). Spectral data of **SI-12a** and **SI-12c** match those reported above (S36) and below (S46), respectively.

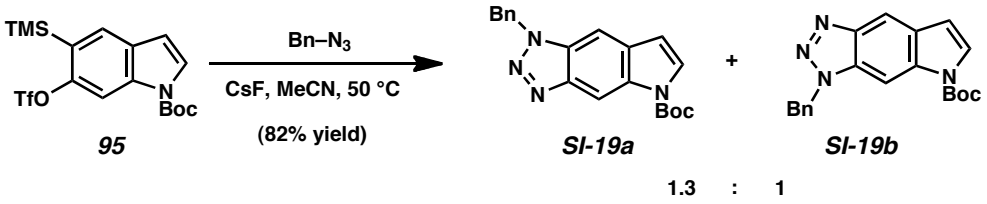


***N*-Boc amines SI-13a and SI-13c.** To a stirred solution of *N*-Boc silyltriflate **95** (25.7 mg, 0.0587 mmol) and aniline (26.8 μL, 0.294 mmol, 5 equiv) in MeCN (1.17 mL) was added CsF (17.8 mg, 0.117 mmol, 2 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50 °C for 15.5 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (1.5:1 Benzene:Hexanes) to provide a mixture of *N*-Boc amines **SI-13a** and **SI-13c** (1.5:1 ratio of **SI-13a**:**SI-13c**, 15.1 mg, 83% yield).

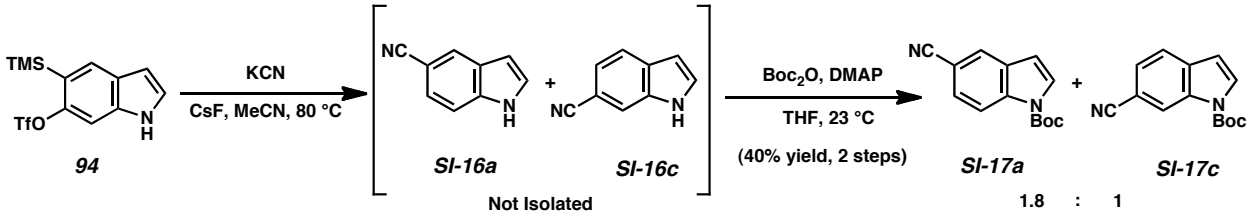
Spectral data of **SI-13a** and **SI-13c** match those reported above (S36) and below (S47), respectively.



***N*-H triazoles SI-18a and SI-18b.** To a stirred solution of *N*-H silyltriflate **94** (20 mg, 0.059 mmol) and benzyl azide[13] (39 mg, 0.30 mmol, 5 equiv) in MeCN (1 mL) was added CsF (27 mg, 0.18 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 80 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (7:3 Hexanes:EtOAc → 1:1 Hexanes:EtOAc) to provide a mixture of *N*-H triazoles **SI-18a** and **SI-18b** (1.8:1 ratio of **SI-18a**:**SI-18b**, 13.2 mg, 90% yield). Analytical samples of **SI-18a** and **SI-18b** were obtained by preparative TLC (4:1 Hexanes:Acetone). **SI-18a:** R_f 0.25 (1:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.65 (br s, 1H), 8.01 (s, 1H), 7.47 (s, 1H), 7.42 (app. t, J = 2.9, 1H), 7.33–7.25 (m, 5H), 6.55 (s, 1H), 5.89 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 144.1, 135.6, 135.2, 131.0, 129.7, 129.3, 128.8, 128.1, 127.4, 101.5, 98.6, 97.6, 52.1. IR (film): 3182, 2919, 1447, 1242 cm^{-1}; HRMS-ESI (*m*/*z*) [M + H]$^+$ calcd for $C_{15}H_{13}N_4$, 249.1140; found, 249.1141. **SI-18b:** R_f 0.33 (1:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.27 (s, 1H), 8.10 (br s, 1H), 7.32–7.25 (m, 6H), 7.15 (s, 1H), 6.67 (s, 1H), 5.86 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 143.1, 137.6, 135.1, 130.6, 128.8, 128.3, 128.1, 127.6, 127.3, 109.6, 102.8, 88.0, 52.0; IR (film): 3204, 2918, 1250 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{15}H_{12}N_4Na$, 271.0960; found, 271.0956.

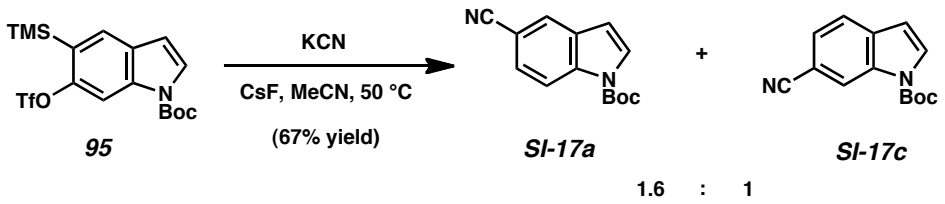


***N*-Boc triazoles SI-19a and SI-19b.** To a stirred solution of *N*-Boc silyltriflate **95** (20 mg, 0.046 mmol) and benzyl azide[13] (30 mg, 0.23 mmol, 5 equiv) in MeCN (1 mL) was added CsF (21 mg, 0.14 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (3:2 Hexanes:Et_2O) to provide a mixture of *N*-Boc triazoles **SI-19a** and **SI-19b** (1.3:1 ratio of **SI-19a**:**SI-19b**, 13 mg, 82% yield). These compounds were characterized as a mixture. R_f 0.29 (7:3 Hexanes:EtOAc); **SI-19a:** ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.76 (br s, 1H), 7.75 (br s, 1H), 7.35 (s, 1H), 7.34–7.25 (m, 5H), 6.56 (d, J = 3.8, 1H), 5.86 (s, 2H), 1.70 (s, 9H); **SI-19b:** ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.19 (br s, 1H), 8.15 (s, 1H), 7.64 (br s, 1H), 7.34–7.25 (m, 5H), 6.66 (d, J = 3.8, 1H), 5.87 (s, 2H), 1.64 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 149.5, 149.4, 145.1, 143.6, 136.0, 135.0, 134.9, 133.5, 132.8, 131.5, 130.2, 130.1, 129.9, 128.84, 128.81, 128.4, 128.2, 128.1, 127.31, 127.30, 110.2, 107.4, 106.7, 104.8, 98.9, 94.4, 84.1, 83.8, 52.1, 51.8, 28.14, 28.08; IR (film): 2979, 2933, 1727, 1143 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{20}H_{20}N_4O_2Na$, 371.1484; found, 371.1473.

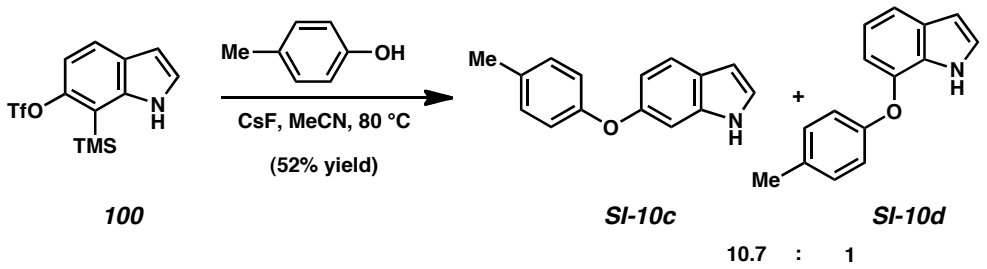


***N*-H cyanoindoles SI-16a and SI-16c.** To a stirred solution of *N*-H silyltriflate **94** (20 mg, 0.059 mmol) and potassium cyanide (14.7 mg, 0.226 mmol, 3.8 equiv) in MeCN (1 mL) was added CsF (27 mg, 0.18 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 80 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica

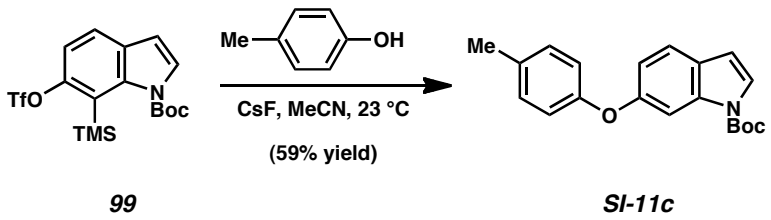
gel (EtOAc eluent). Evaporation under reduced pressure afforded crude *N*-H cyanoindoles **SI-16a** and **SI-16c**. This crude mixture was dissolved in THF (1 mL) and treated with DMAP (1 mg, 0.0081 mmol, 0.14 equiv) and Boc_2O (14.2 mg, 0.065 mmol, 1.1 equiv). The resulting mixture was stirred at 23 °C for an additional 14 h, then filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (95:5 Hexanes:Et_2O → 9:1 Hexanes:Et_2O) to provide a mixture of *N*-Boc cyanoindoles **SI-17a** and **SI-17c** (1.8:1 ratio of **SI-17a:SI-17c**, 4.4 mg, 40% yield). Spectral data of **SI-17a** and **SI-17c** match those reported above (S39) and below (S50), respectively.



***N*-Boc cyanoindoles SI-17a and SI-17c.** To a stirred solution of *N*-Boc silyltriflate **95** (20 mg, 0.046 mmol) and potassium cyanide (38 mg, 0.584 mmol, 12.7 equiv) in MeCN (1 mL) was added CsF (21 mg, 0.14 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50 °C for 24 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (95:5 Hexanes:Et_2O) to provide a mixture of *N*-Boc cyanoindoles **SI-17a** and **SI-17c** (1.6:1 ratio of **SI-17a:SI-17c**, 7.4 mg, 67% yield) as a white solid. Spectral data of **SI-17a** (S39) and **SI-17c** (S50) match those reported above and below, respectively.

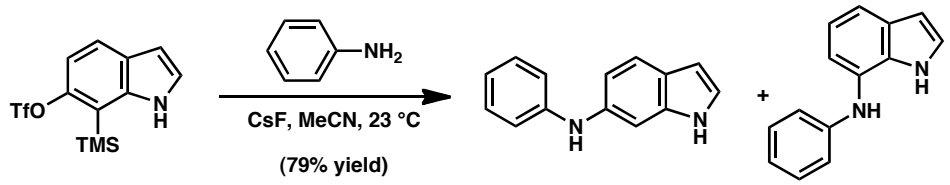


***N*-H ethers SI-10c and SI-10d.** To a stirred solution of *N*-H silyltriflate **100** (37.5 mg, 0.11 mmol) and *p*-cresol (60 mg, 0.56 mmol, 5 equiv) in MeCN (1.2 mL) was added CsF (51 mg, 0.33 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 80 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were purified by flash chromatography (3:1 Hexanes:CH_2Cl_2) to provide *N*-H ether **SI-10c** (11.8 mg) and crude **SI-10d**. Further purification by preparative TLC (3:2 Hexanes:CH_2Cl_2) afforded **SI-10d** (1.1 mg; 10.7:1 ratio of **SI-10c**:**SI-10d**, 52% combined yield). **SI-10c**: R_f 0.12 (85:15 Hexanes:CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.05 (br s, 1H), 7.58 (d, *J* = 8.5, 1H), 7.16 (app. t, *J* = 2.9, 1H), 7.12 (d, *J* = 8.3, 2H), 7.01 (d, *J* = 1.8, 1H), 6.93–6.91 (m, 2H), 6.88 (dd, *J* = 6.4, 2.1, 1H), 6.54–6.53 (m, 1H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.1, 153.3, 136.1, 131.9, 130.0, 124.02, 123.97, 121.3, 118.1, 113.1, 102.5, 101.3, 20.5; IR (film): 3418, 2921, 1506, 1491, 1243, 1219 cm^{-1}; HRMS-ESI (*m/z*) [M + H]$^+$ calcd for $C_{15}H_{14}NO$, 224.1075; found, 224.1075. **SI-10d**: R_f 0.25 (85:15 Hexanes:CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.24 (br s, 1H), 7.40 (d, *J* = 7.9, 1H), 7.18 (app. t, *J* = 2.7, 1H), 7.13 (d, *J* = 8.3, 2H), 7.02 (app. t, *J* = 7.8, 1H), 6.96 (d, *J* = 8.5, 2H), 6.73 (d, *J* = 7.7, 1H), 6.58 (app. t, *J* = 2.8, 1H), 2.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.6, 142.6, 132.6, 130.3, 130.1, 127.7, 124.1, 120.1, 118.0, 115.8, 110.1, 102.9, 20.6; IR (film): 3426, 2924, 2852, 1713, 1505, 1241 cm^{-1}; HRMS-ESI (*m/z*) [M + H]$^+$ calcd for $C_{15}H_{14}NO$, 224.1075; found, 224.1079.



99 ***SI-11c***

***N*-Boc ether SI-11c.** To a stirred solution of *N*-Boc silyltriflate **99** (20 mg, 0.046 mmol) and *p*-cresol (24.7 mg, 0.23 mmol, 5 equiv) in MeCN (1 mL) at 23 °C was added CsF (21 mg, 0.14 mmol, 3 equiv). The solution was stirred for 18 h, then filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (85:15 Hexanes:CH_2Cl_2 → 3:1 Hexanes:CH_2Cl_2) to provide *N*-Boc ether **SI-11c** (8.7 mg, 59% yield) as a light pink solid. R_f 0.31 (4:1 Hexanes:CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.73 (br s, 1H), 7.55 (d, J = 3.5, 1H), 7.47 (d, J = 8.5, 1H), 7.13 (d, J = 8.3, 2H), 6.97–6.93 (m, 3H), 6.53 (dd, J = 3.7, 0.5, 1H), 2.33 (s, 3H), 1.58 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.4, 155.3, 149.6, 135.6, 132.4, 130.1, 126.1, 125.5, 121.3, 118.8, 114.7, 107.0, 105.5, 83.7, 27.9, 20.6; IR (film): 2979, 1735 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{20}H_{21}NO_3Na$, 346.1419; found, 346.1412.

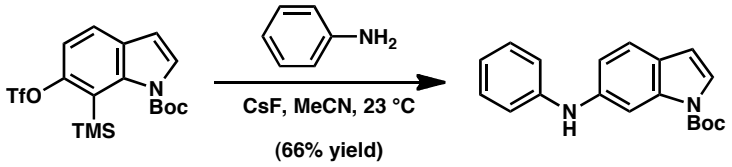


100 ***SI-12c*** ***SI-12d***

13.8 : 1

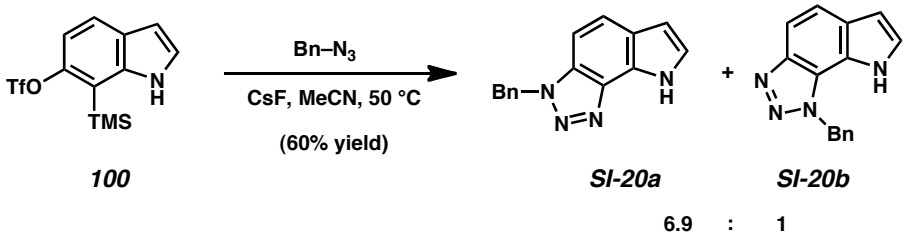
***N*-H amines SI-12c and SI-12d.** To a stirred solution of *N*-H silyltriflate **100** (40 mg, 0.12 mmol) and aniline (54 μL, 0.59 mmol, 5 equiv) in MeCN (1.2 mL) at 23 °C was added CsF (54 mg, 0.36 mmol, 3 equiv). The solution was stirred for 14 h, then filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (2:3 Hexanes:CH_2Cl_2) to provide a mixture of *N*-H amines **SI-12c** and **SI-12d** (13.8:1 ratio of **SI-12c**:**SI-12d**, 19.6 mg, 79% yield). An analytical sample of **SI-12c** was obtained by preparative TLC (3:7 Hexanes:CH_2Cl_2). **SI-12c:** R_f 0.46 (3:7

Hexanes:CH_2Cl_2); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98 (br s, 1H), 7.55 (d, *J* = 9.0, 1H), 7.26–7.22 (m, 2H), 7.178–7.176 (m, 1H), 7.12 (app. t, *J* = 3.0, 1H), 7.04–7.03 (m, 2H), 9.19 (dd, *J* = 8.5, 2.0, 1H), 6.87 (app. t, *J* = 7.5, 1H), 6.51–6.50 (m, 1H), 5.70 (br s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 144.9, 137.9, 136.5, 129.2, 123.5, 123.4, 121.2, 119.7, 116.3, 114.8, 102.5, 101.6; IR (film): 3399, 3048, 1627, 1597, 1494 cm^{-1}; HRMS-ESI (*m/z*) [M + H]$^+$ calcd for $C_{14}H_{13}N_2$, 209.1079; found, 209.1078. **SI-12d:** R_f 0.46.

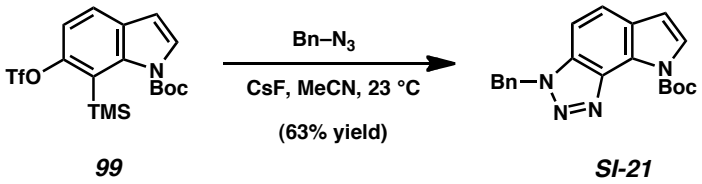


99 ***SI-13c***

***N*-Boc amine SI-13c.** To a stirred solution of *N*-H silyltriflate **99** (40 mg, 0.091 mmol) and aniline (43 μL, 0.46 mmol, 5 equiv) in MeCN (1 mL) at 23 °C was added CsF (42 mg, 0.27 mmol, 3 equiv). The solution was stirred for 14 h, then filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (97:3 Hexanes:Et_2O) to provide *N*-Boc amine **SI-13c** (18.6 mg, 66% yield). R_f 0.17 (95:5 Hexanes:CH_2Cl_2); ^{1}H NMR (500 MHz, 40 °C, $CDCl_3$): δ 7.94 (br s, 1H), 7.50 (d, *J* = 3.7, 1H), 7.43 (d, *J* = 8.4, 1H), 7.26 (app. t, *J* = 7.8, 2H), 7.09 (d, *J* = 7.6, 2H), 7.00 (dd, *J* = 8.3, 2.0, 1H), 6.91 (app. t, *J* = 7.3, 1H), 6.50 (d, *J* = 3.7, 1H), 5.75 (br s, 1H), 1.64 (s, 9H); ^{13}C NMR (125 MHz, 40 °C, $CDCl_3$): δ 149.7, 144.0, 140.3, 136.2, 129.2, 125.2, 124.7, 121.2, 120.4, 117.2, 115.3, 107.1, 105.2, 83.4, 28.1; IR (film): 3391, 2979, 2931, 1727, 1337, 1149 cm^{-1}; HRMS-ESI (*m/z*) [M + Na]$^+$ calcd for $C_{19}H_{20}N_2O_2Na$, 331.1422; found, 331.1423.

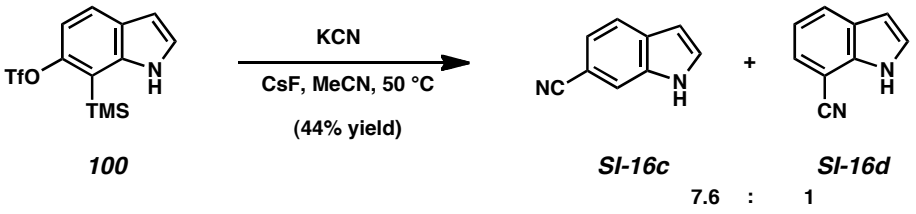


***N*-H triazoles SI-20a and SI-20b.** To a stirred solution of *N*-H silyltriflate **100** (20 mg, 0.059 mmol) and benzyl azide[13] (39 mg, 0.30 mmol, 5 equiv) in MeCN (1 mL) was added CsF (27 mg, 0.18 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (85:15 Hexanes:EtOAc) to provide a mixture of *N*-H triazoles **SI-20a** and **SI-20b** (6.9:1 ratio of **SI-20a**:**SI-20b**, 8.8 mg, 60% yield). An analytical sample of **SI-20a** was obtained by preparative TLC (4:1 Hexanes:Acetone). **SI-20a:** R_f 0.46 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.3 (br s, 1H), 7.68 (d, J = 8.7, 1H), 7.36–7.28 (m, 6H), 7.07 (d, J = 8.7, 1H), 6.72 (app. t, J = 2.3, 1H), 5.91 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 134.9, 134.8, 130.9, 128.8, 128.2, 127.3, 124.4, 123.7, 122.6, 122.3, 104.0, 101.7, 52.4; IR (film): 3181, 3113, 2921, 2851, 1225 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{15}H_{12}N_4Na$, 271.0960; found, 271.0963. **SI-20b:** R_f 0.46 (3:1 Hexanes:EtOAc); 7.75 (d, J = 8.8, 1H), 7.68 (br s, 1H), 7.58 (d, J = 8.9, 1H), 7.46 (m, 3H), 7.35–7.34 (m, 2H), 7.01 (app. t, J = 2.5, 1H), 6.63 (app. t, J = 2.3, 1H), 6.05 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 144.7, 134.9, 129.8, 129.1, 127.0, 126.5, 123.7, 121.9, 118.7, 118.2, 111.8, 104.6, 53.5; IR (film): 3256, 2918, 1235 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{15}H_{12}N_4Na$, 271.0960; found, 271.0963.

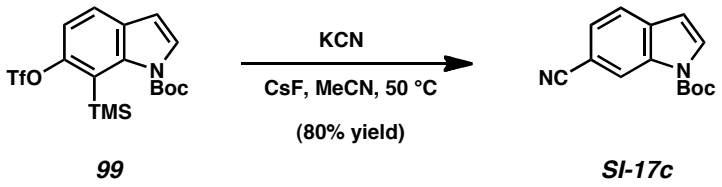


***N*-Boc triazole SI-21.** To a stirred solution of *N*-Boc silyltriflate **99** (20 mg, 0.046 mmol) and benzyl azide[13] (30.4 mg, 0.23 mmol, 5 equiv) in MeCN (1 mL) at 23 °C was added CsF (21 mg,

0.14 mmol, 3 equiv). The solution was stirred for 18 h, then filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (95:5 Benzene:Et_2O) to provide *N*-Boc triazole **SI-21** (10 mg, 63% yield). R_f 0.31 (3:2 Hexanes:Et_2O); ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.68 (d, *J* = 3.6, 1H), 7.58 (d, *J* = 8.6, 1H), 7.31–7.24 (m, 5H), 7.21 (d, *J* = 8.6, 1H), 6.70 (d, *J* = 3.6, 1H), 5.90 (s, 2H), 1.78 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 149.0, 135.9, 134.9, 132.4, 128.8, 128.2, 127.3, 126.4, 125.1, 123.7, 121.7, 107.8, 105.0, 84.8, 52.2, 28.1; IR (film): 2979, 2933, 1760, 1732, 1327, 1150 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{20}H_{20}N_4O_2Na$, 371.1484; found, 371.1478.



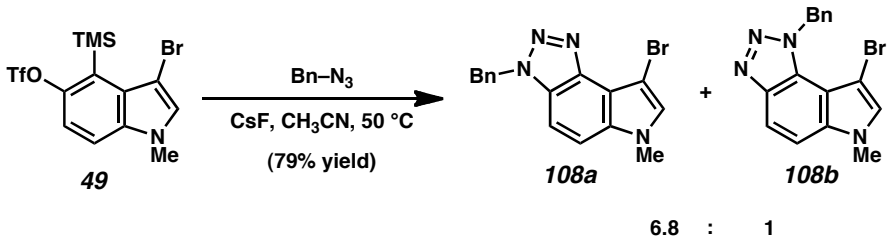
***N*-H cyanoindoles SI-16c and SI-16d.** To a stirred solution of *N*-H silyltriflate **100** (20 mg, 0.060 mmol) and potassium cyanide (49.0 mg, 0.30 mmol, 5 equiv) in MeCN (1 mL) was added CsF (27 mg, 0.18 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude products, which were further purified by flash chromatography (4:1 Hexanes:EtOAc) to provide a mixture of *N*-H cyanoindoles **SI-16c** and **SI-16d** (7.6:1 ratio of **SI-16c**:**SI-16d**, 5.4 mg, 44% yield). An analytical sample of **SI-16c** was obtained by preparative TLC (1:1 Hexanes:Et_2O). **SI-16c:** R_f 0.29 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, 40 °C, $CDCl_3$): δ 8.51 (br s, 1H), 7.76 (s, 1H), 7.70 (d, *J* = 8.2, 1H), 7.43 (app. t, *J* = 2.8, 1H), 7.36 (dd, *J* = 8.2, 1.1, 1H), 6.65–6.64 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 134.5, 131.0, 127.8, 122.7, 121.4, 120.5, 115.8, 104.4, 103.4; IR (film): 3414, 2220, 1348 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_9H_6N_2Na$, 165.0429; found, 165.0430. **SI-16d:** R_f 0.29 (3:1 Hexanes:EtOAc).



99 ***SI-17c***

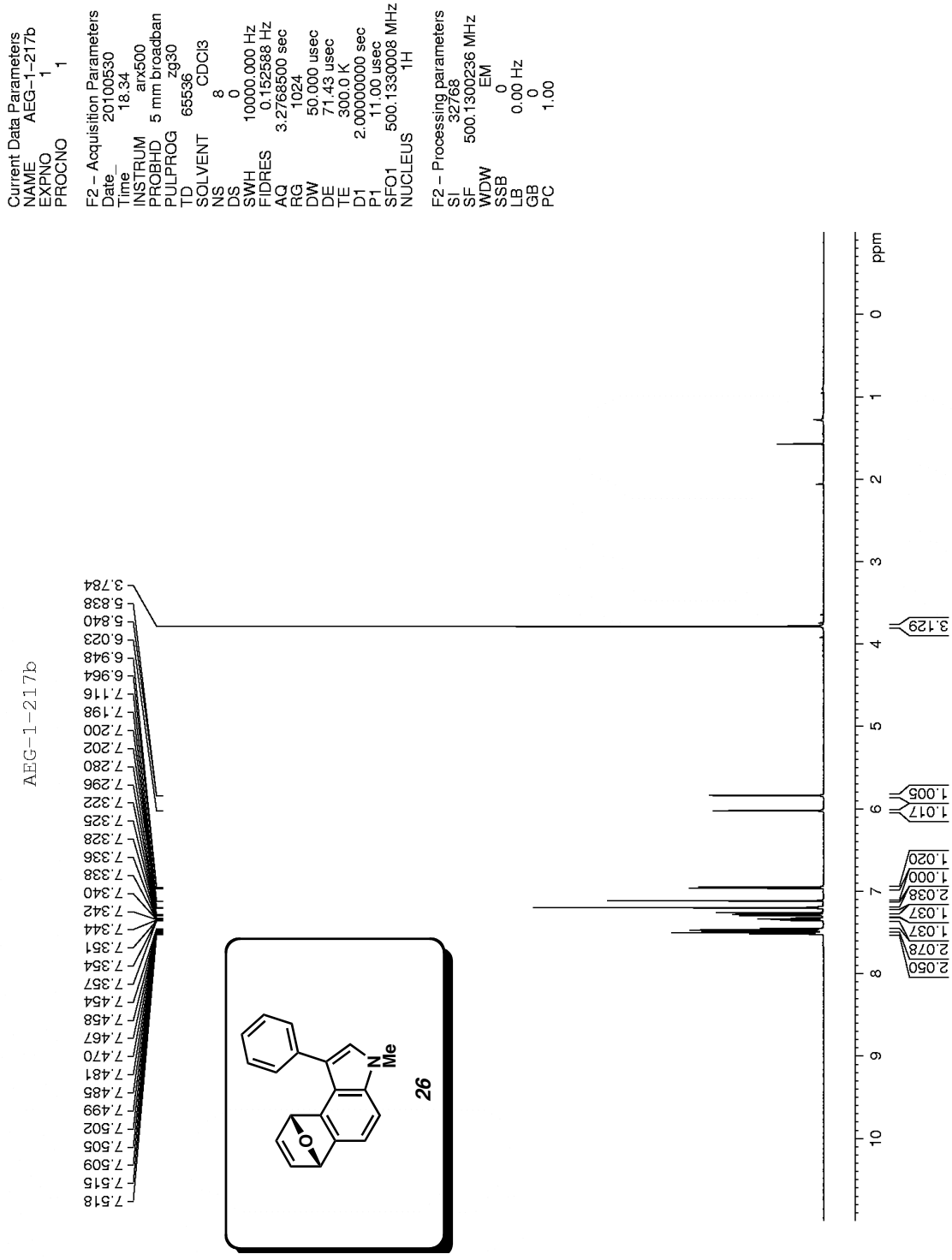
***N*-Boc cyanoindole SI-17c.** To a stirred solution of *N*-Boc silyltriflate **99** (22.0 mg, 0.050 mmol) and potassium cyanide (41.5 mg, 0.25 mmol, 5 equiv) in MeCN (1.0 mL) was added CsF (23.0 mg, 0.15 mmol, 3 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50 °C for 14 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (9:1 Hexanes:Et_2O) to provide cyanoindole **SI-17c** (9.8 mg, 80% yield) as a white solid. R_f 0.26 (9:1 Hexanes:Et_2O); ^{1}H NMR (500 MHz, 40 °C, $CDCl_3$): δ 8.50 (s, 1H), 7.76 (d, J = 3.7, 1H), 7.62 (d, J = 8.1, 1H), 7.47 (dd, J = 8.1, 1.2, 1H), 6.62 (d, J = 3.6, 1H), 1.70 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 148.8, 134.2, 133.7, 129.1, 125.6, 121.5, 119.7, 119.6, 107.2, 107.1, 84.9, 28.0; IR (film): 2981, 2224, 1736, 1342 cm^{-1}; HRMS-ESI (*m*/*z*) [M + Na]$^+$ calcd for $C_{14}H_{14}N_2O_2Na$, 265.0953; found, 265.0948.

G. Influence of a C3 halide substituent on the regioselectivities of 4,5-indolyne reactions.



3-Bromo triazoles 108a and 108b. To a solution of 3-bromo silyltriflate **49** (41.6 mg, 0.097 mmol), and benzyl azide[13] (55.8 mg, 0.420 mmol, 4.3 equiv) in MeCN (1 mL) was added CsF (45.2 mg, 0.300 mmol, 3.1 equiv). The reaction vessel was placed in an aluminum heating block maintained at 50 °C for 7.75 h. After cooling to 23 °C, the reaction mixture was filtered over silica gel (EtOAc eluent). Evaporation under reduced pressure afforded the crude product, which was further purified by flash chromatography (2:1 Hexanes:EtOAc) to give a mixture of 3-bromo triazoles **108a** and **108b** (6.8:1 ratio of **108a**:**108b**, 26.0 mg, 79% yield) as a white solid. These compounds were characterized as a mixture. R_f 0.21 (2:1 Hexanes:EtOAc); **108a:** ^{1}H NMR (500 MHz, CD_2Cl_2): δ 7.16 (d, J = 9.0, 1H), 7.35–7.23 (m, 5H), 7.22 (s, 1H), 7.18 (d, J = 9.0, 1H), 5.89 (s, 2H), 3.85 (s, 3H); **108b:** ^{1}H NMR (500 MHz, CD_2Cl_2): δ 7.85 (d, J = 9.1, 1H), 7.35–7.23 (m, 4H), 7.18 (s, 1H), 7.10–7.06 (m, 2H), 6.55 (s, 2H), 3.87 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 143.0, 139.9, 137.0, 135.8, 135.2, 132.8, 129.4, 129.0, 128.6, 128.4, 128.3, 128.0, 127.8, 127.6, 127.4, 126.7, 117.0, 114.6, 111.6, 110.2, 109.1, 104.3, 87.9, 86.5, 54.8, 52.4, 34.1, 33.9; IR (film): 3028, 2945, 1447, 1307, 1102 cm^{-1}; HRMS-ESI (m/z) [M + Na]$^+$ calculated for $C_{16}H_{13}N_4BrNa$, 363.0221; found, 363.0227. The major isomer was identified by dissolving the mixture of isomers in EtOAc and exposing the mixture to a balloon of H_2 in the presence of Pd/C (10 mol%) and Et_3N (1 equiv) to afford the corresponding debrominated compounds. Spectral data of the debrominated compounds match those previously reported.[1]

^{1}H NMR Spectra: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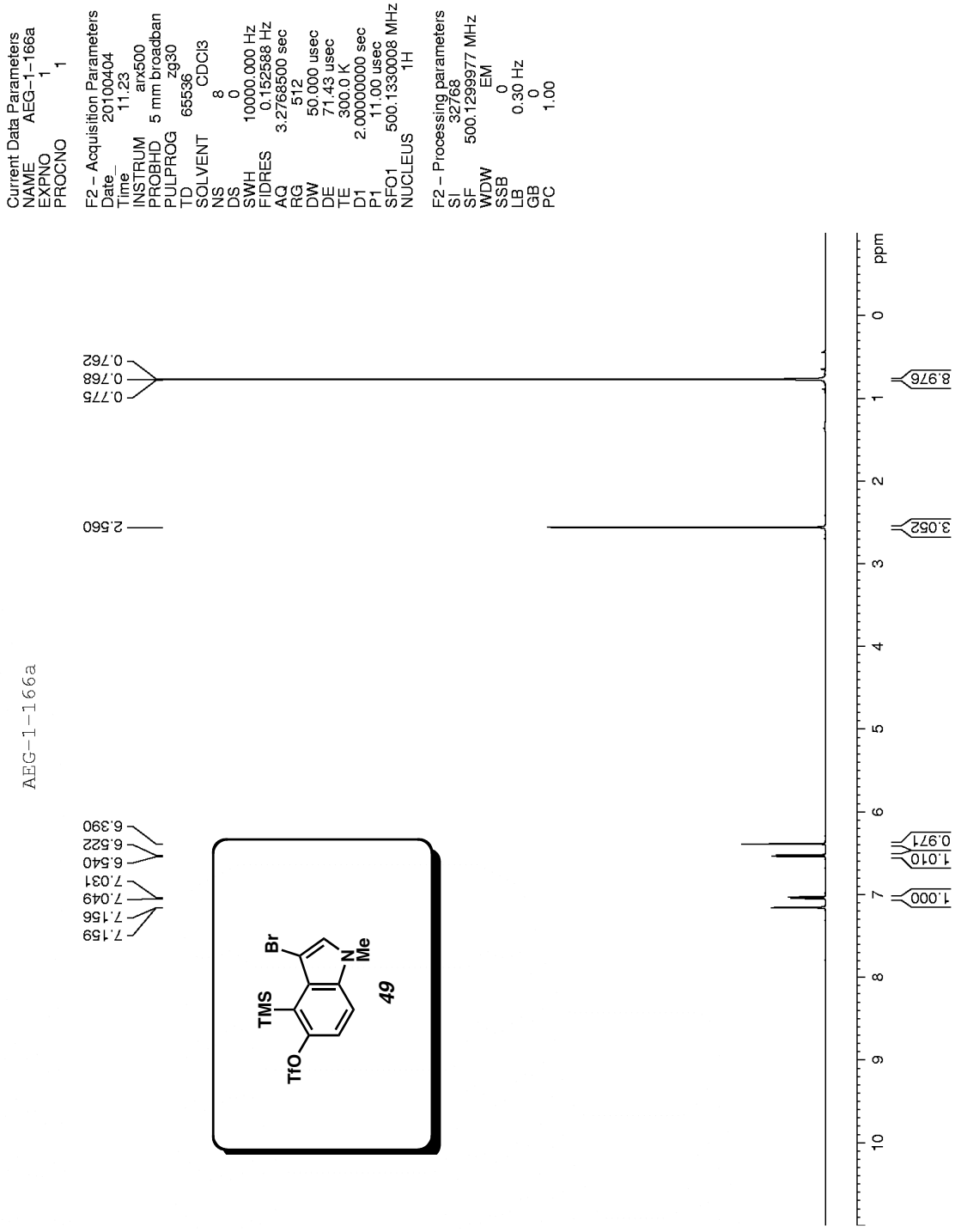
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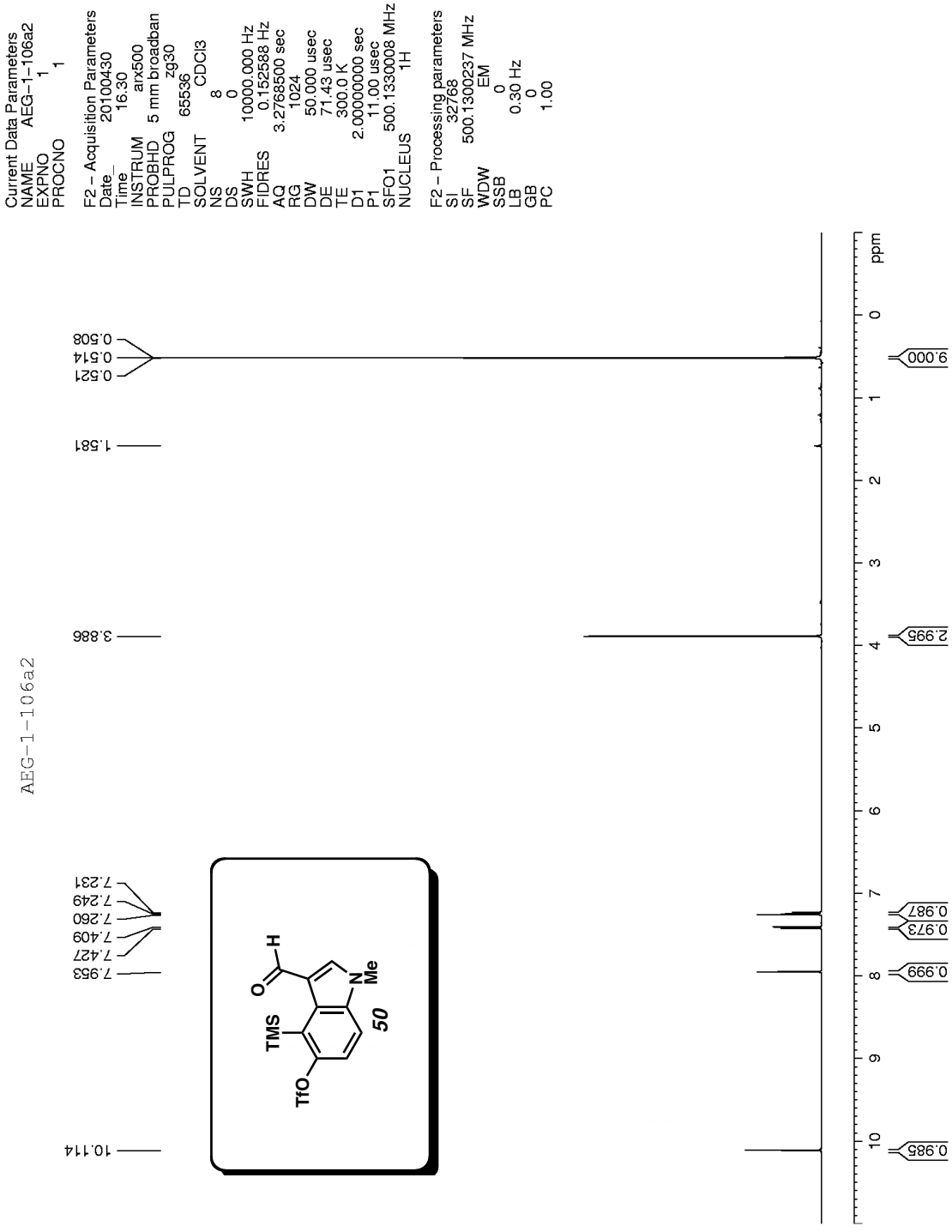
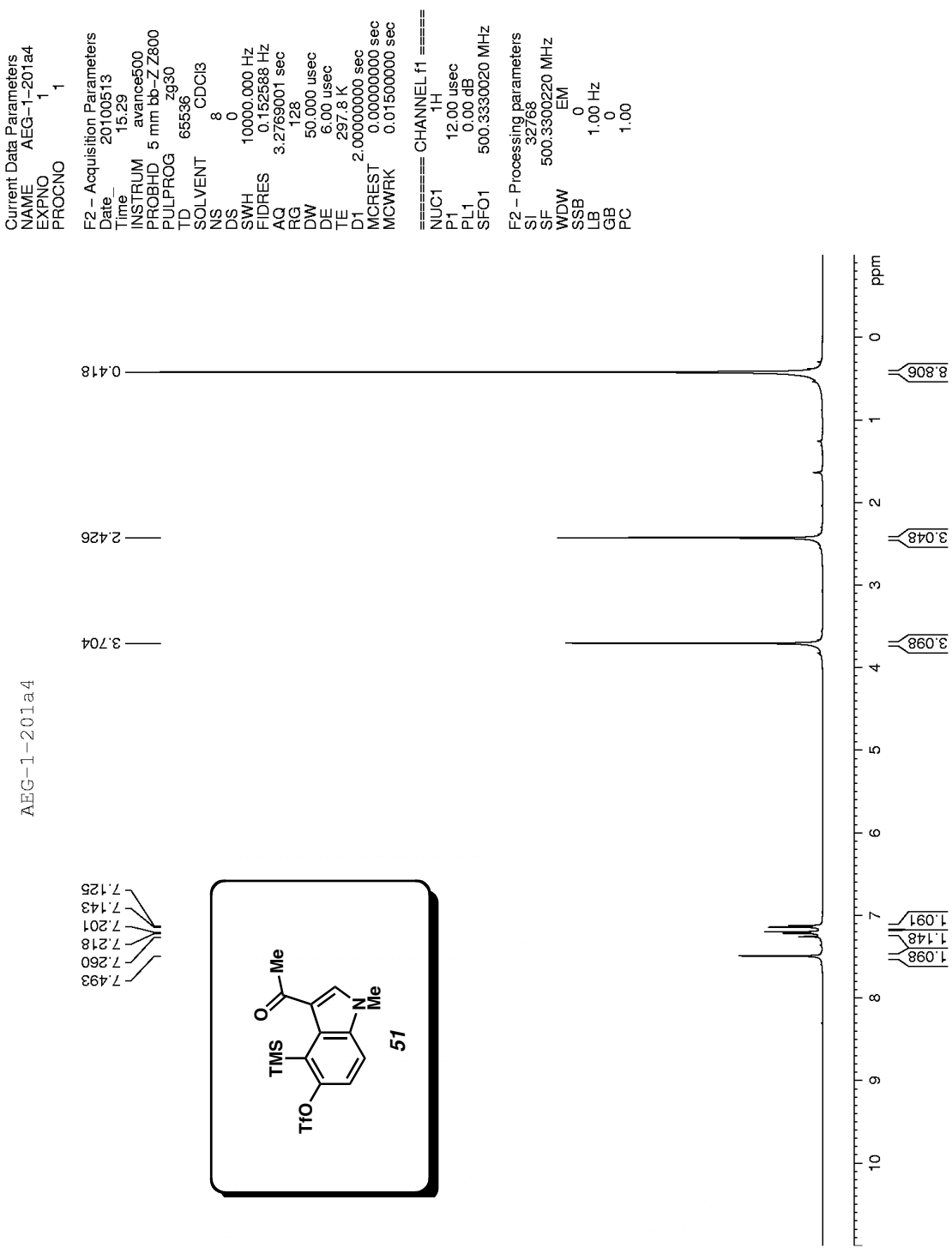
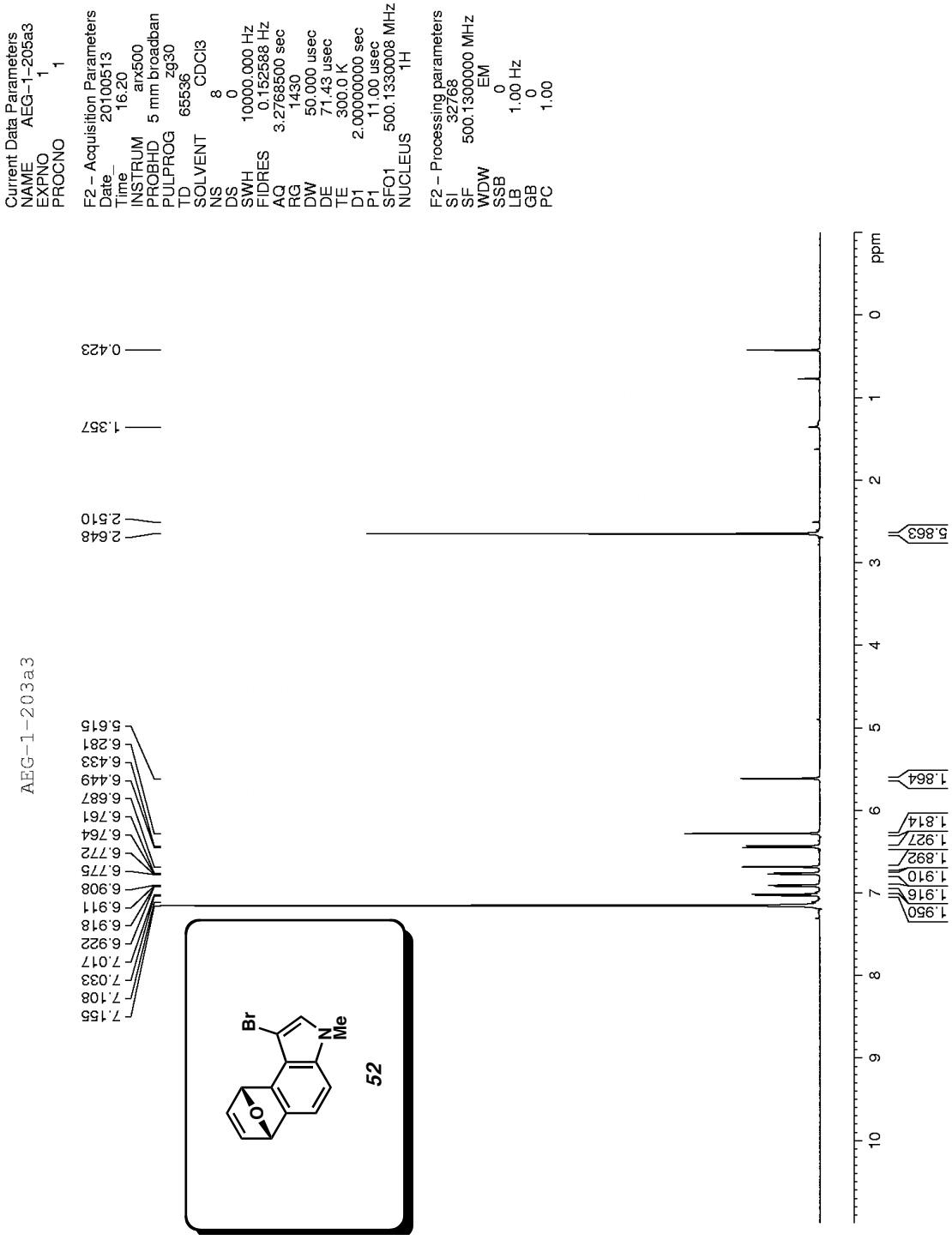
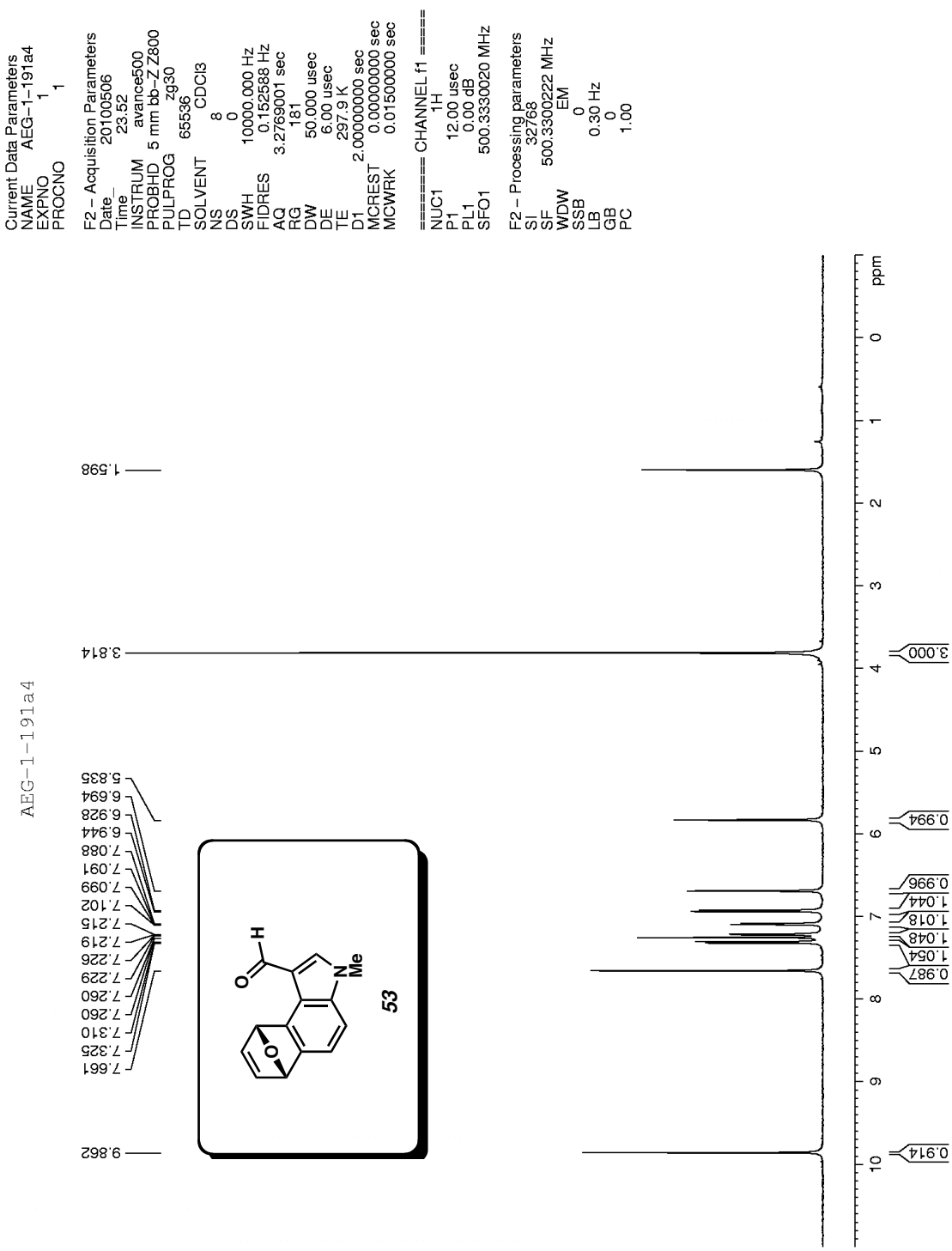
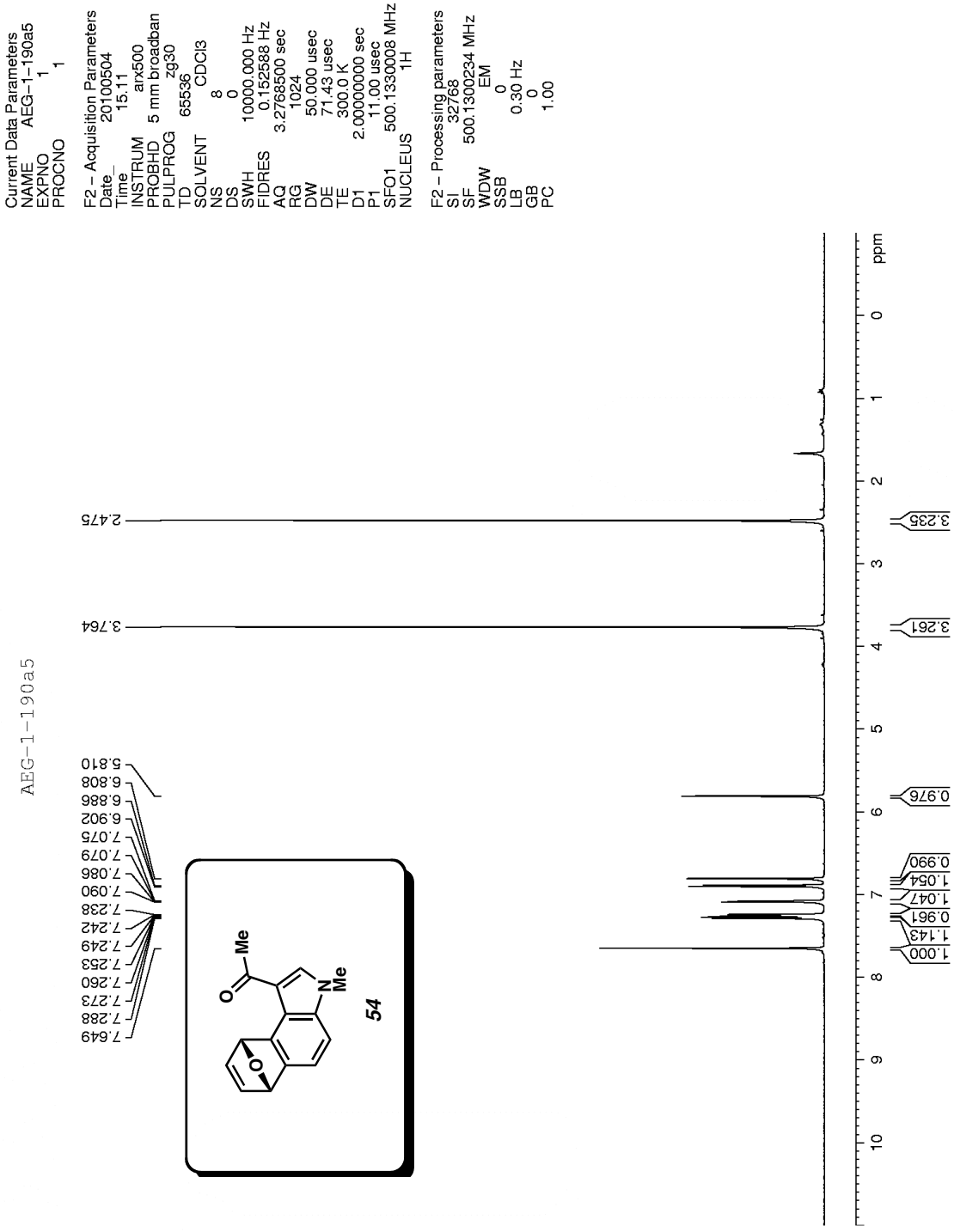
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1299977 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
AEG-1-166a
0.762
0.768
0.775
2.560
6.390
6.522
6.540
7.031
7.049
7.156
7.159
Br
TMS
TfO
N
Me
49
8.976
3.052
1.010
0.971
1.000
ppm

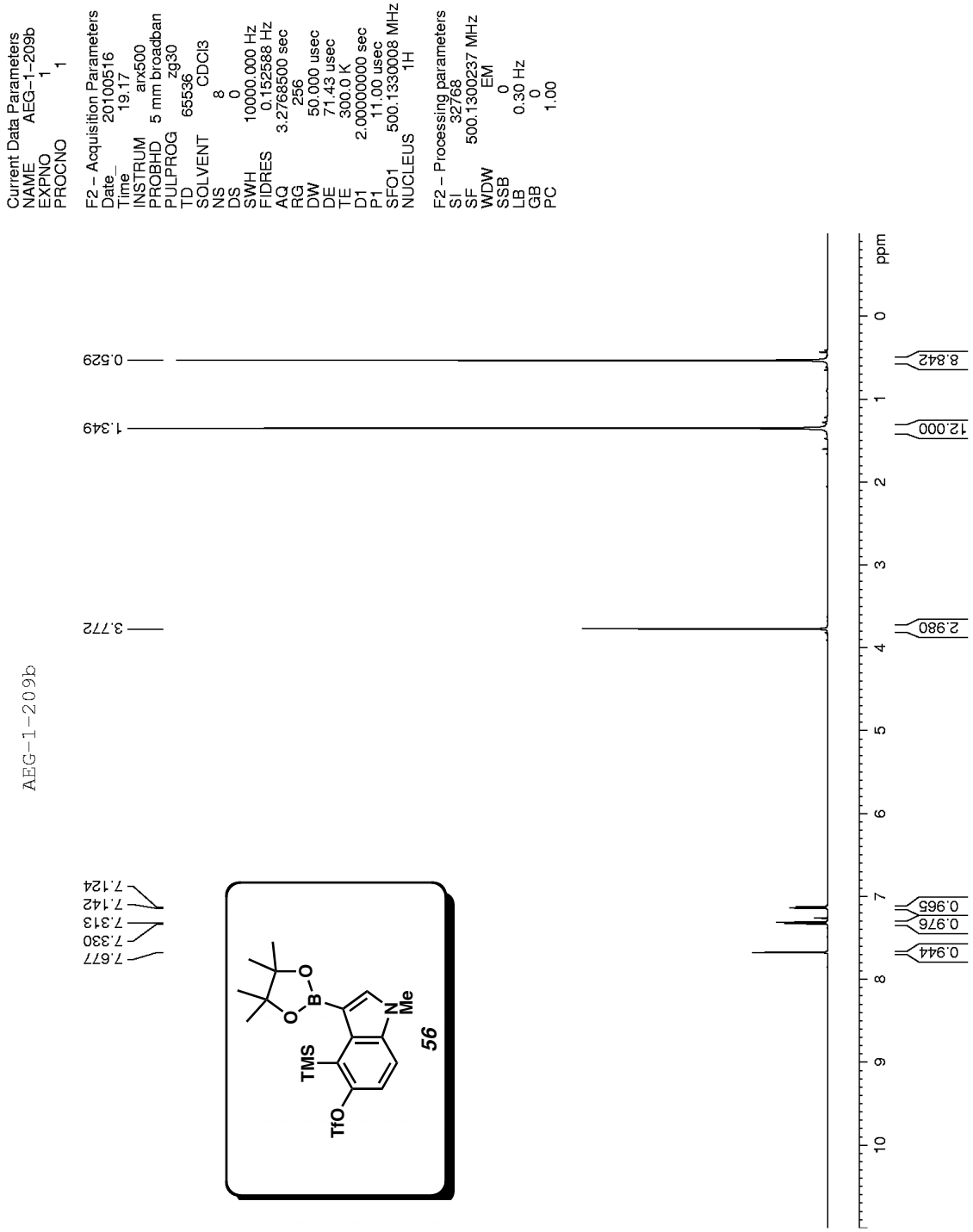

Current Data Parameters
NAME AEG-1-106a2
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date 20100430
Time 16.30
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 1024
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300237 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
AEG-1-106a2
10.114
7.953
7.427
7.409
7.260
7.249
7.231
3.886
1.581
0.521
0.514
0.508
TMS
TfO
O
H
N
Me
50
0.985
0.999
0.973
0.987
2.995
9.000
ppm

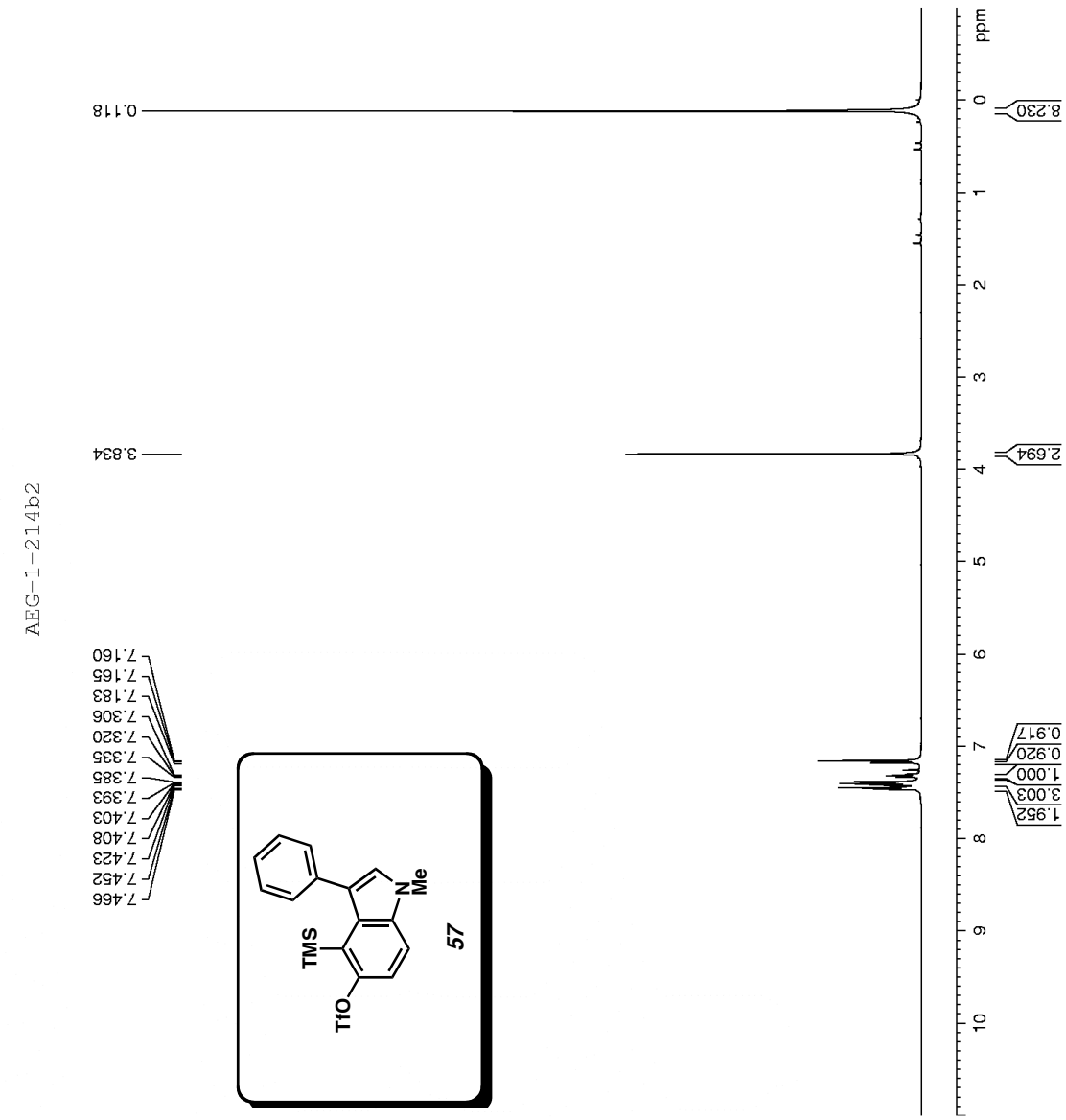

Current Data Parameters
NAME AEG-1-201a4
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date 20100513
Time 15.29
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 128
DW 50.000 usec
DE 6.00 usec
TE 297.8 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
AEG-1-201a4
0.418
2.426
3.704
7.125
7.143
7.201
7.218
7.260
7.493
TMS
TfO
O
Me
N
Me
51
8.806
3.048
3.098
1.091
1.148
1.098
ppm

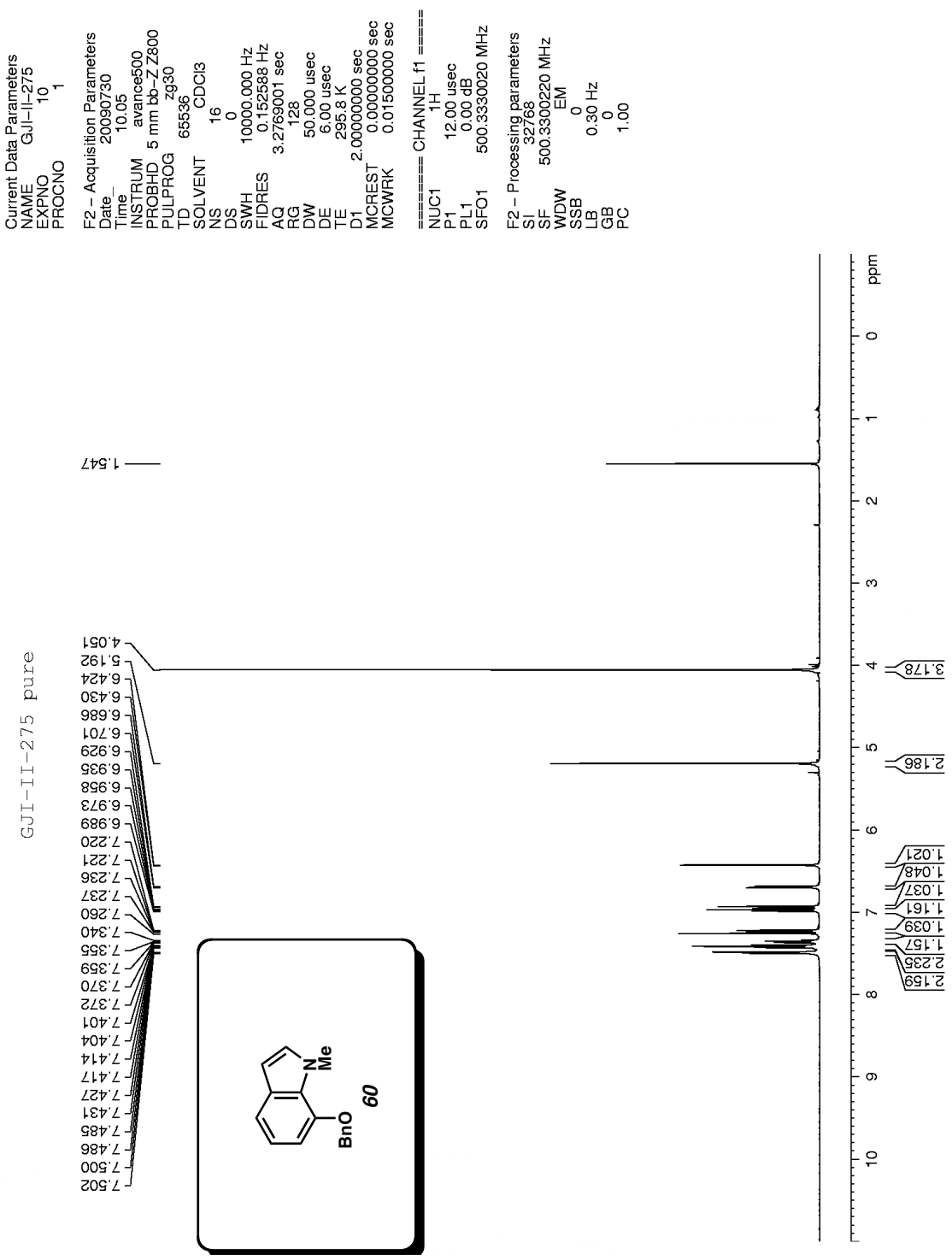

Current Data Parameters
NAME AEG-1-205a3
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date 20100513
Time 16.20
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 1430
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
AEG-1-203a3
0.423
1.357
2.510
2.648
5.615
6.281
6.433
6.449
6.687
6.761
6.764
6.772
6.775
6.908
6.911
6.918
6.922
7.017
7.033
7.108
7.155
5.863
1.864
1.814
1.927
1.892
1.910
1.916
1.950
Br
N
Me
O
52
ppm

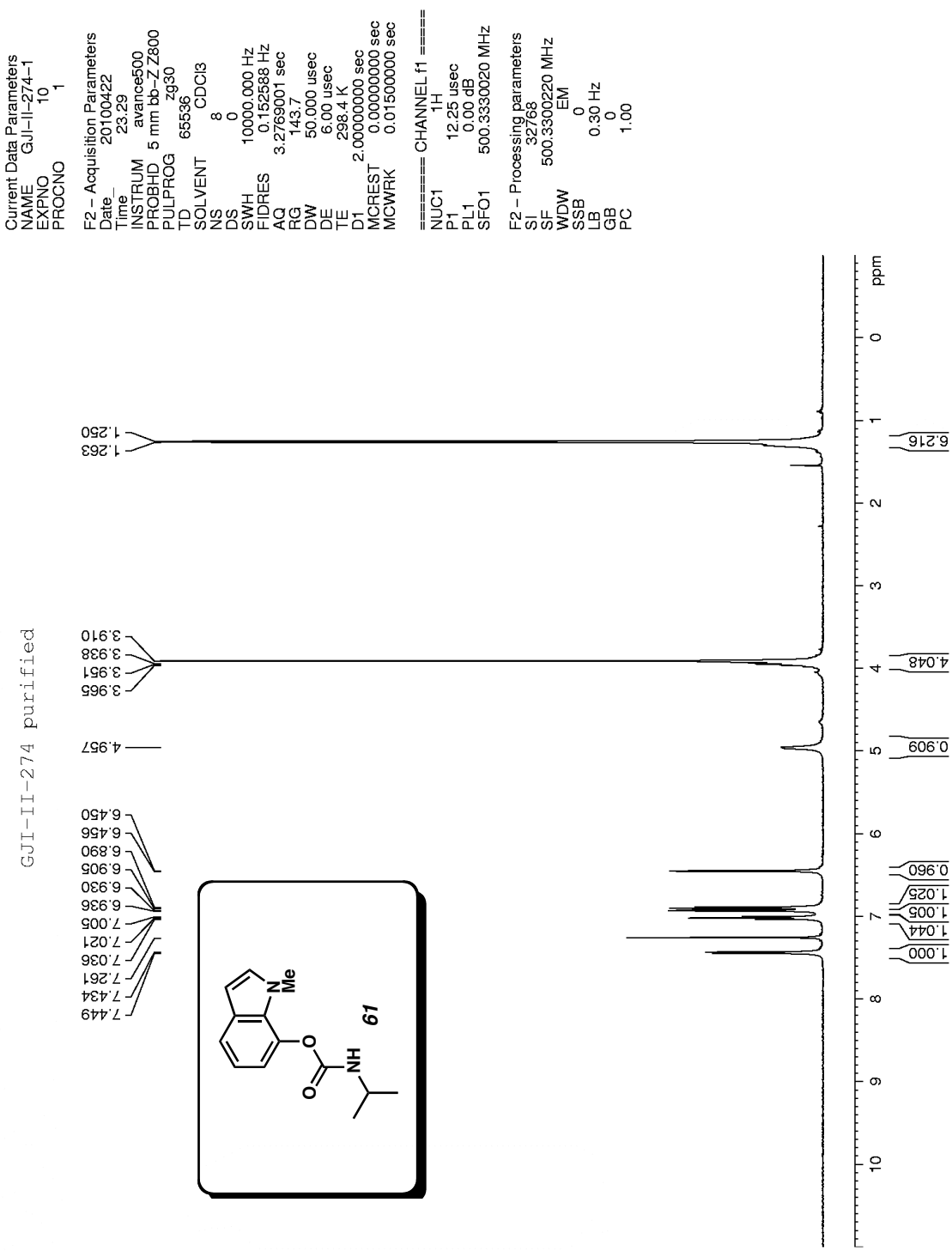

Current Data Parameters
NAME AEG-1-191a4
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date 20100506
Time 23.52
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300222 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
AEG-1-191a4
9.862
7.661
7.325
7.310
7.260
7.260
7.229
7.226
7.219
7.215
7.102
7.099
7.091
7.088
6.944
6.928
6.694
5.835
3.814
1.598
O
H
N
Me
O
53
0.914
0.987
1.054
1.048
1.018
1.044
0.996
0.994
3.000
ppm

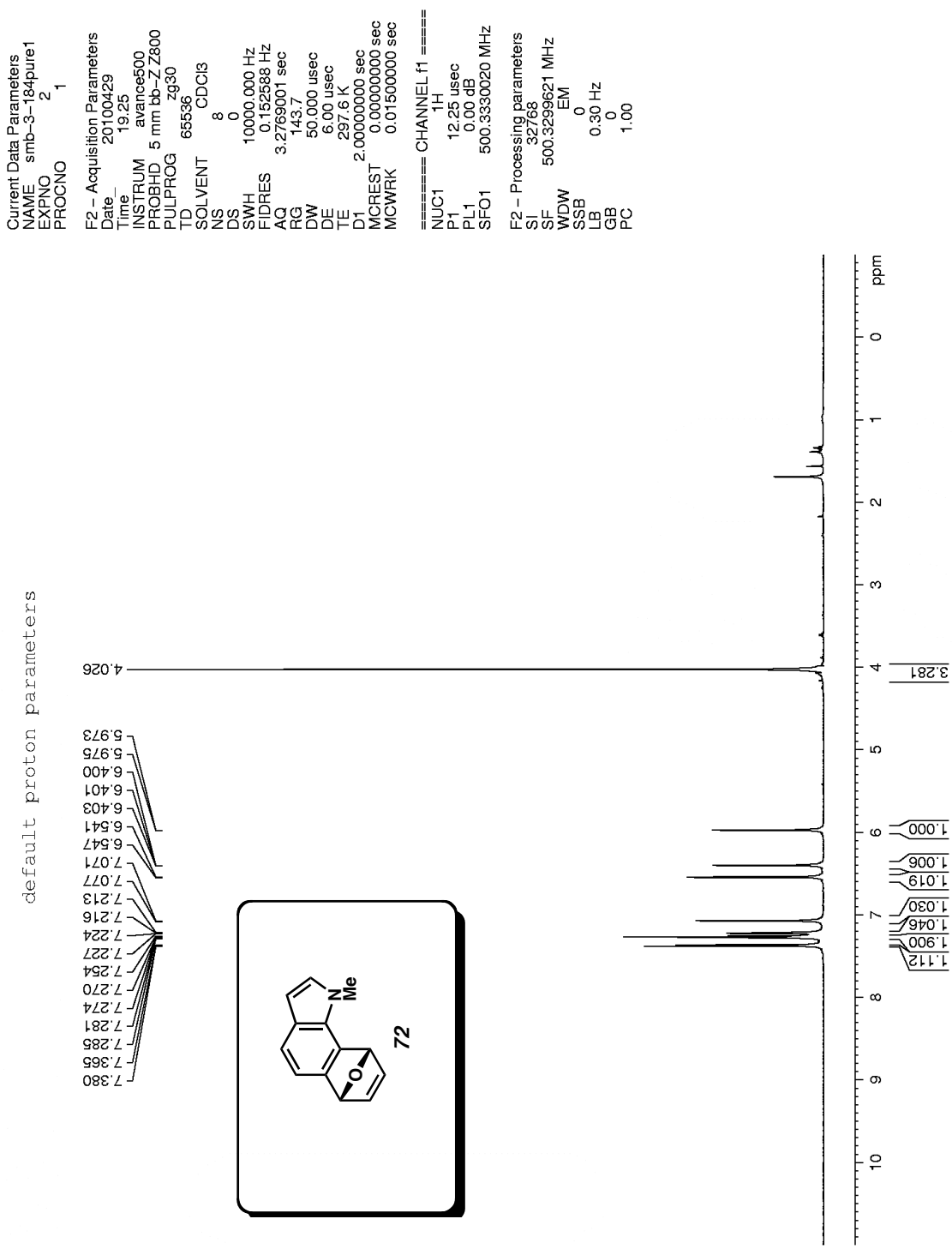

Current Data Parameters
NAME AEG-1-190a5
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date 20100504
Time 15.11
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 1024
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300234 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
AEG-1-190a5
2.475
3.764
5.810
6.808
6.886
6.902
7.075
7.079
7.086
7.090
7.238
7.242
7.249
7.253
7.260
7.273
7.288
7.649
Me
O
N
Me
O
54
3.235
3.261
0.976
0.990
1.054
1.047
0.961
1.143
1.000
ppm

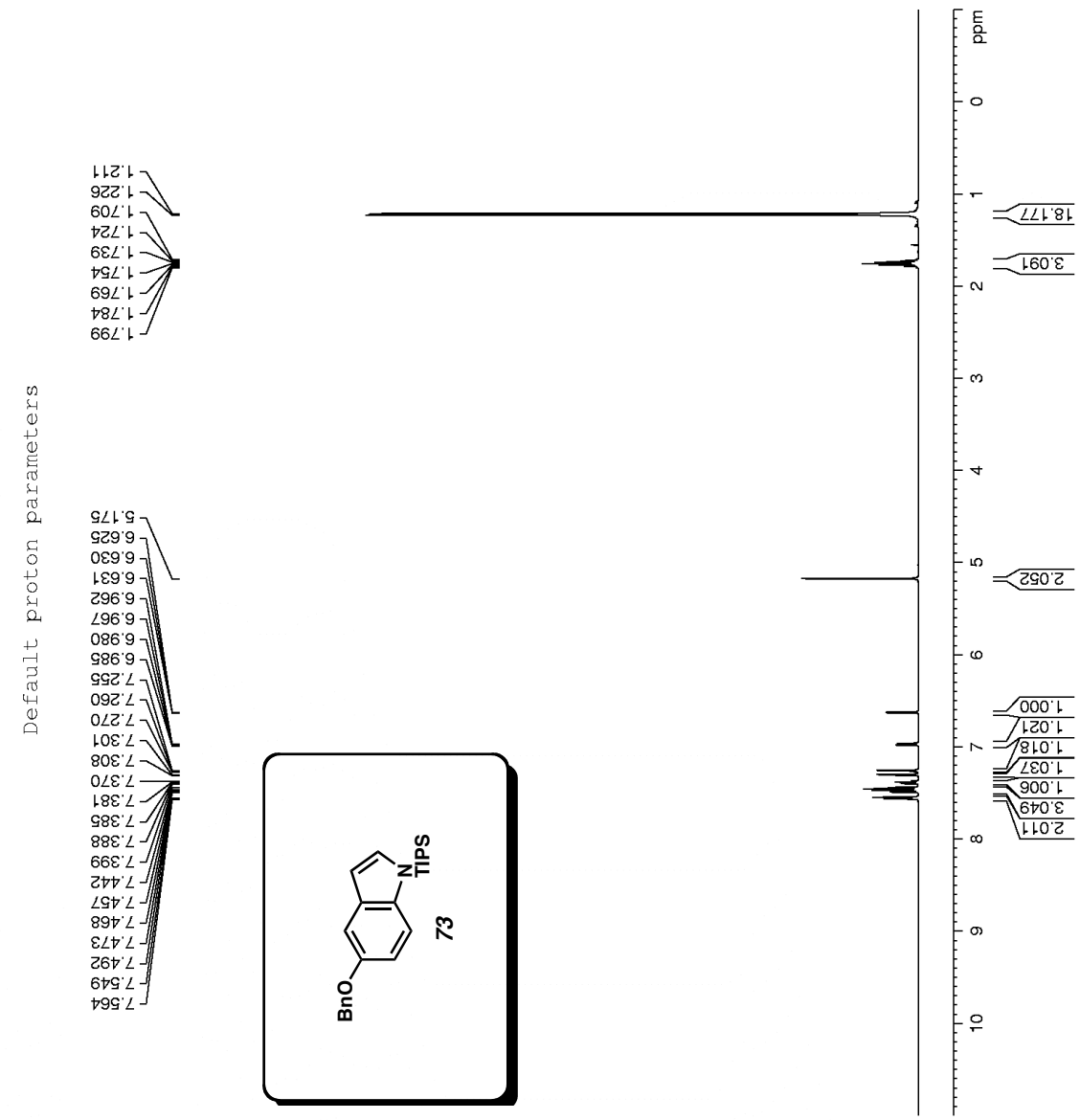

Current Data Parameters
NAME AEG-1-209b
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date 20100516
Time 19.17
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 256
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300237 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
AEG-1-209b
0.529
1.349
3.772
7.677
7.330
7.313
7.142
7.124
TMS
TfO
N
Me
56
8.842
12.000
2.980
0.965
0.976
0.944
ppm

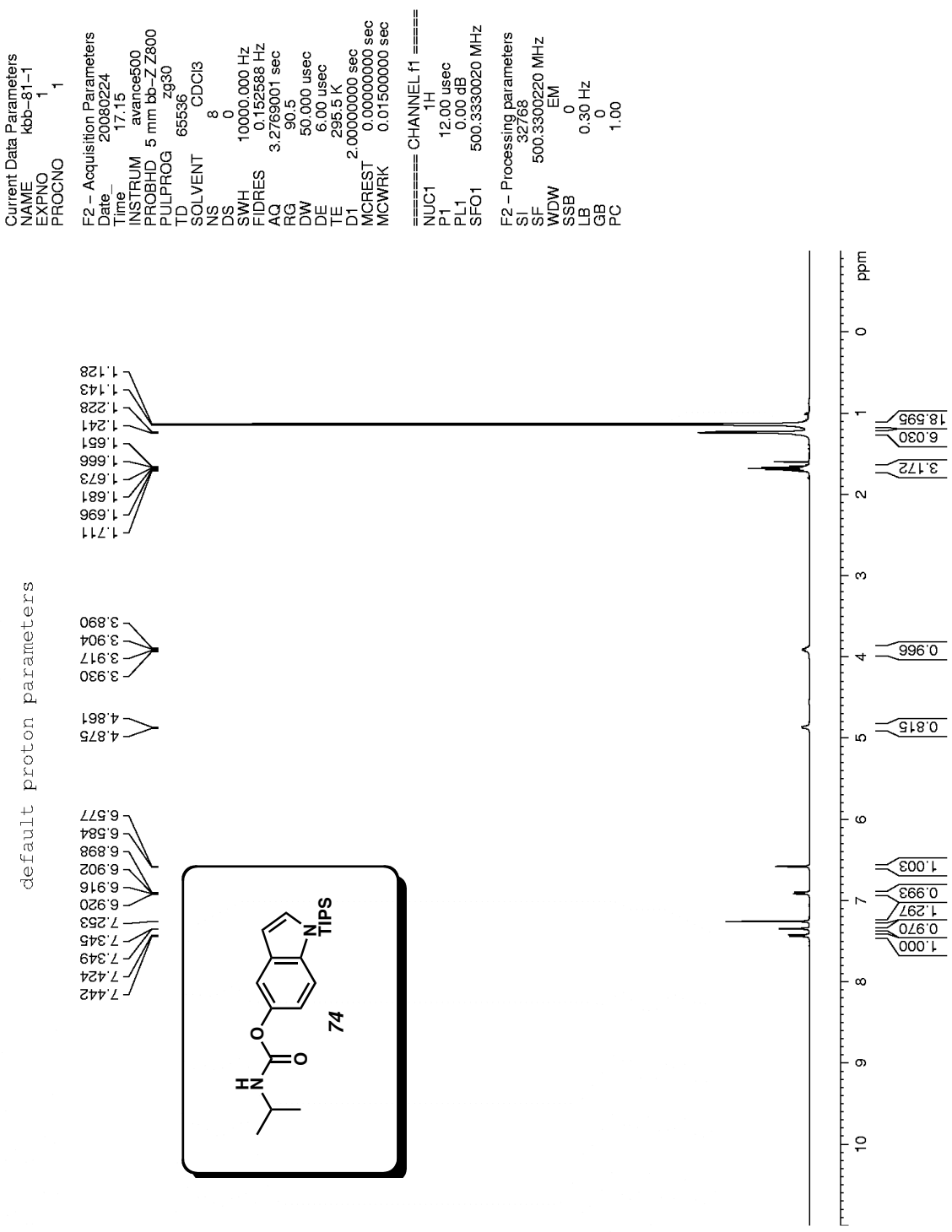

Current Data Parameters
NAME AEG-1-214b2
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date 20100527
Time 15.30
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 90.5
DW 50.000 usec
DE 6.00 usec
TE 297.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
AEG-1-214b2
7.466
7.452
7.423
7.408
7.403
7.393
7.385
7.335
7.320
7.306
7.183
7.165
7.160
3.834
0.118
TMS
TfO
N
Me
57
8.230
2.694
1.952
3.003
1.000
0.920
0.917
ppm

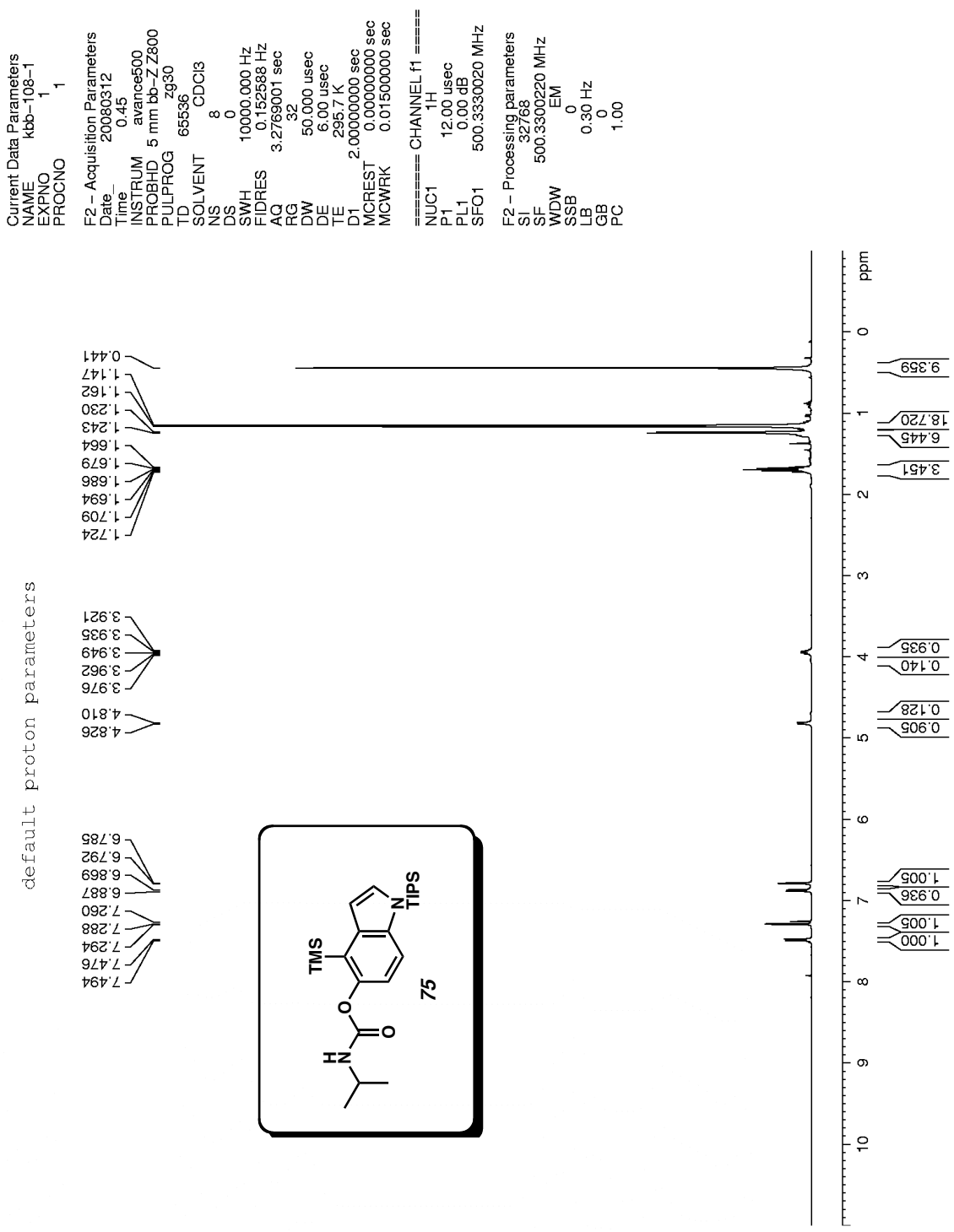

Current Data Parameters
NAME GJI-II-275
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090730
Time 10.05
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 128
DW 50.000 usec
DE 6.00 usec
TE 295.8 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-II-275 pure
1.547
4.051
5.192
6.424
6.430
6.686
6.701
6.929
6.935
6.958
6.973
6.989
7.220
7.221
7.236
7.237
7.260
7.340
7.355
7.359
7.370
7.372
7.401
7.404
7.414
7.417
7.427
7.431
7.485
7.486
7.500
7.502
N
Me
BnO
60
3.178
2.186
2.159
2.235
1.157
1.039
1.161
1.037
1.048
1.021
ppm

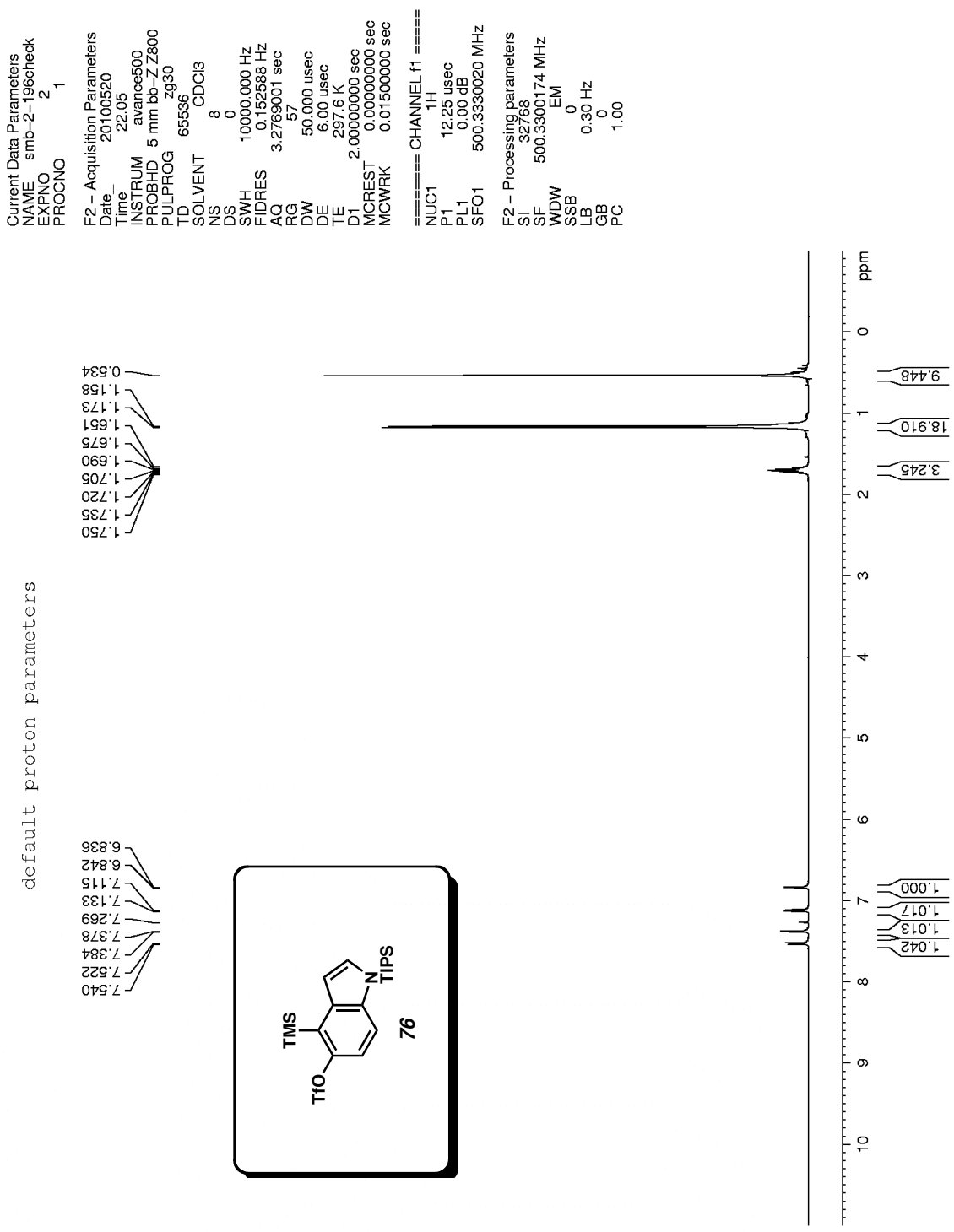

Current Data Parameters
NAME GJI-II-274-1
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100422
Time 23.29
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 143.7
DW 50.000 usec
DE 6.00 usec
TE 298.4 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-II-274 purified
1.263
1.250
3.965
3.938
3.951
3.910
4.957
6.450
6.456
6.890
6.905
6.930
6.936
7.005
7.021
7.036
7.261
7.434
7.449
Me
N
O
O
NH
61
6.216
4.048
0.909
0.960
1.025
1.005
1.044
1.000
ppm

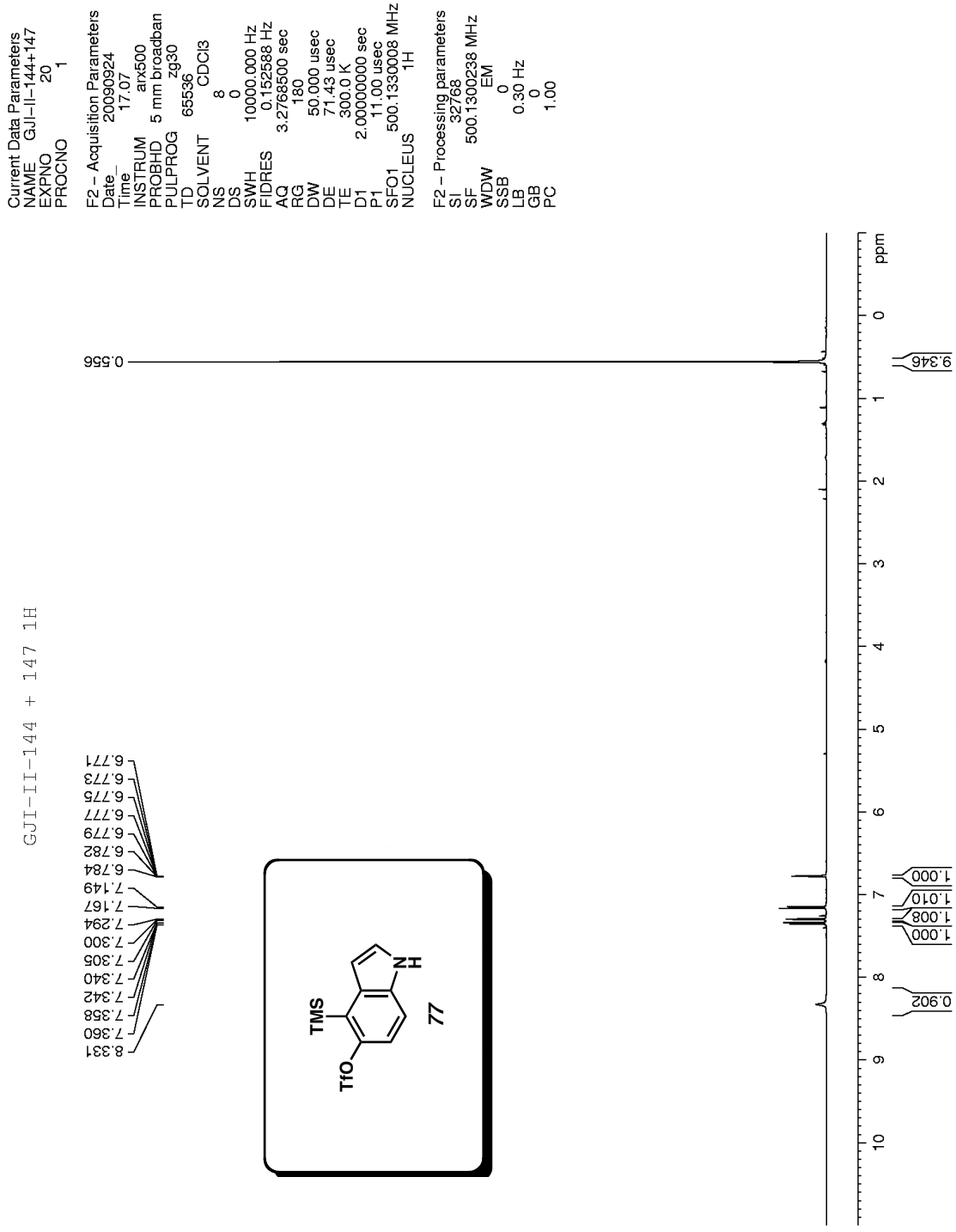

Current Data Parameters
NAME smb-3-184pure1
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date 20100429
Time 19.25
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 143.7
DW 50.000 usec
DE 6.00 usec
TE 297.6 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3299621 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
default proton parameters
4.026
5.973
5.975
6.400
6.401
6.403
6.541
6.547
7.071
7.077
7.213
7.216
7.224
7.227
7.254
7.270
7.274
7.281
7.285
7.365
7.380
N
Me
O
72
3.281
1.000
1.006
1.019
1.030
1.046
1.900
1.112
ppm

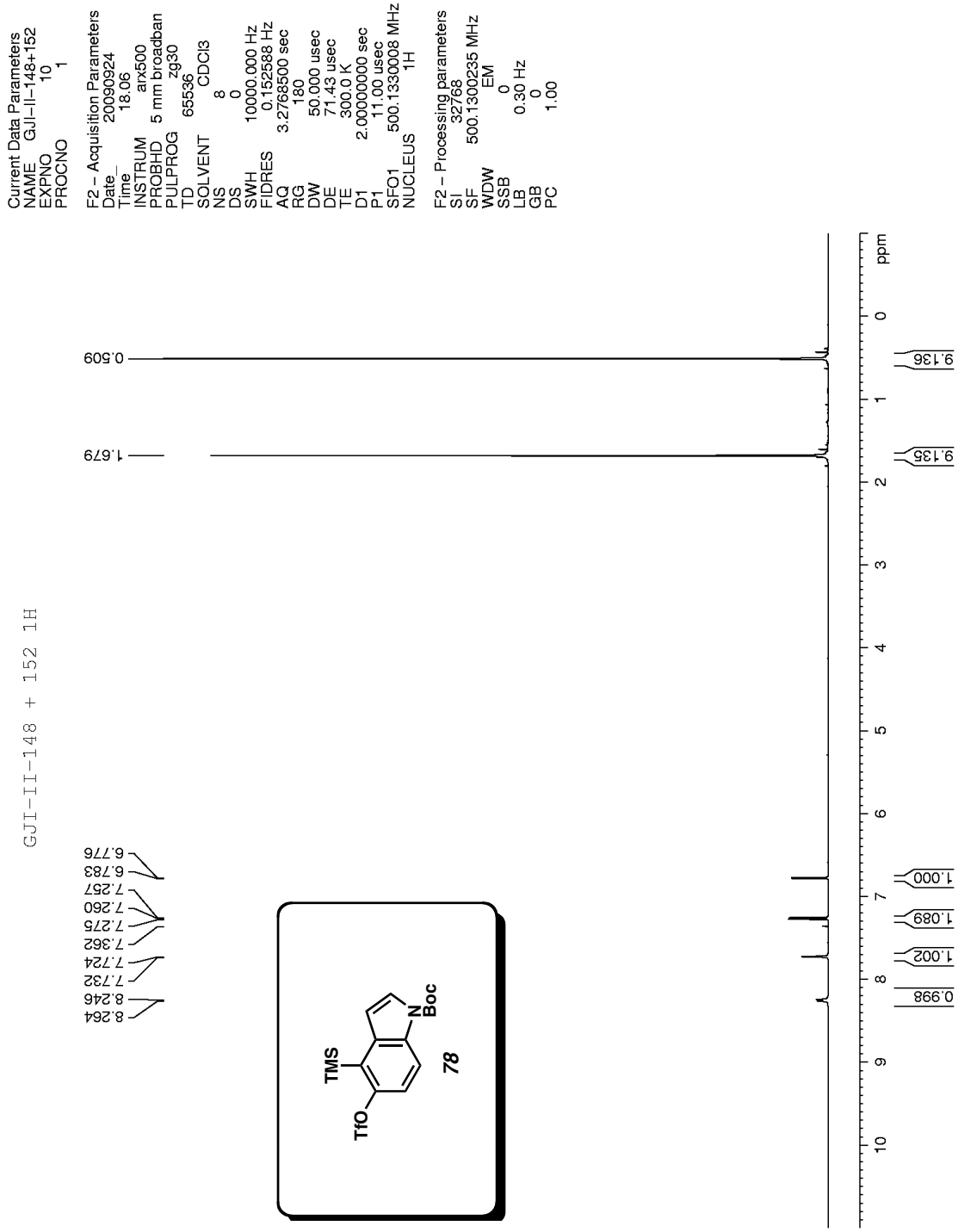

Current Data Parameters
NAME smb-3-188f4
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100502
Time 19.13
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 128
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 12.20 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300186 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Default proton parameters
BnO
N
TIPS
73
ppm
18.177
3.091
2.052
2.011
3.049
1.006
1.037
1.018
1.021
1.000

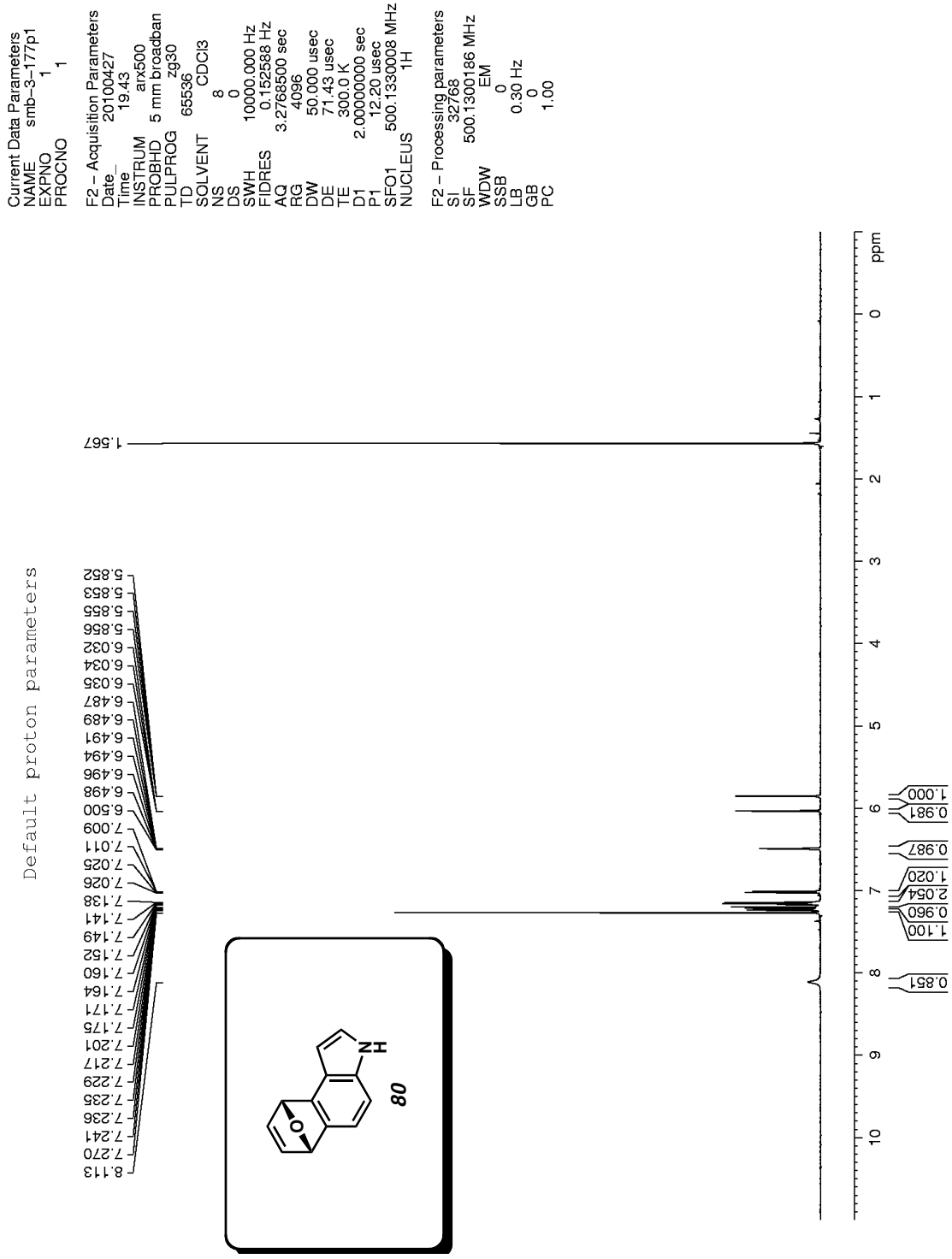

Current Data Parameters
NAME kbb-81-1
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20080224
Time 17.15
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 90.5
DW 50.000 usec
DE 6.00 usec
TE 295.5 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
default proton parameters
7.442
7.424
7.349
7.345
7.253
6.920
6.916
6.902
6.898
6.584
6.577
4.875
4.861
3.930
3.917
3.904
3.890
1.711
1.696
1.681
1.673
1.666
1.651
1.241
1.228
1.143
1.128
TIPS
74
ppm
1.000
0.970
1.297
0.993
1.003
0.815
0.966
3.172
6.030
18.595

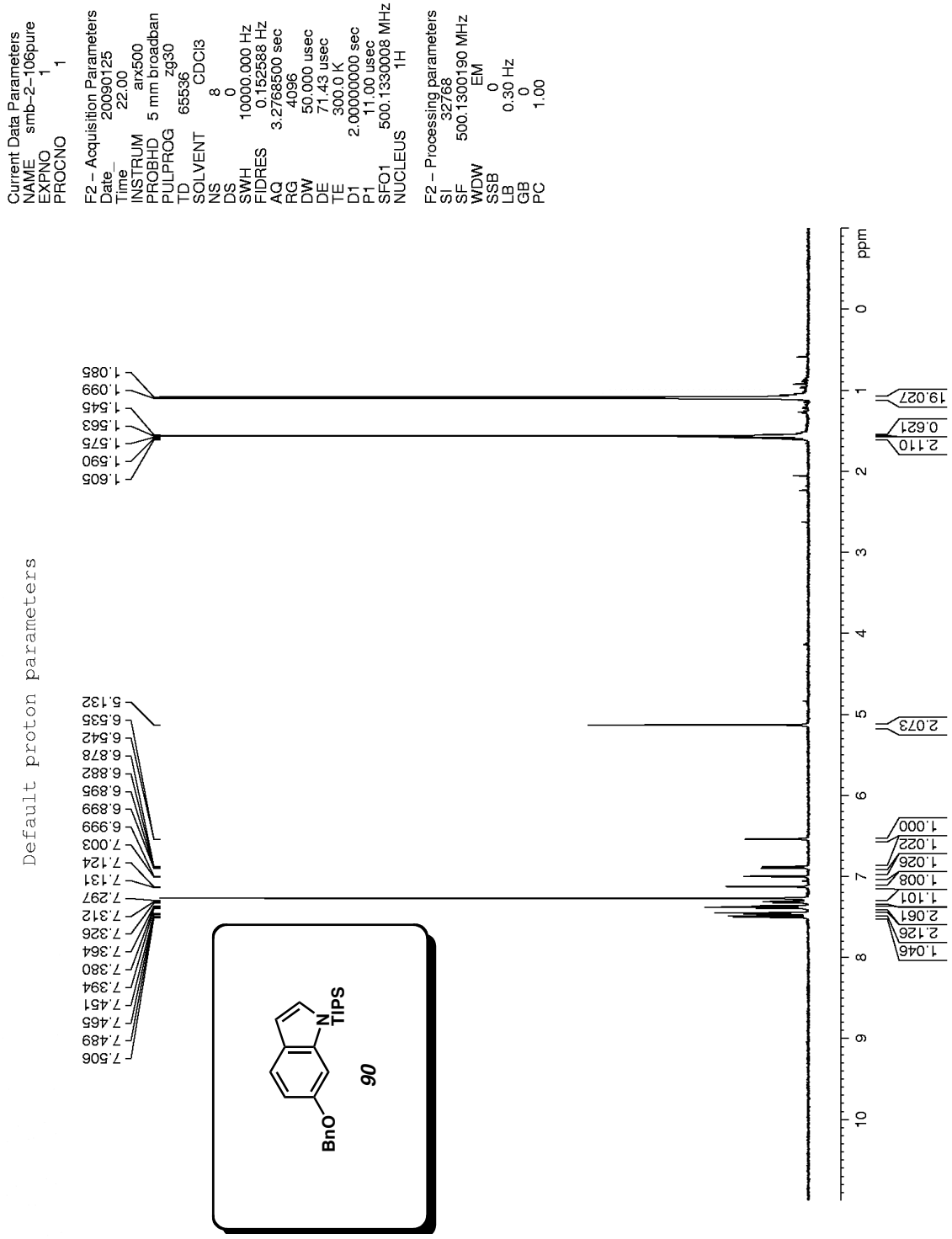

Current Data Parameters
NAME kbb-108-1
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20080312
Time 0.45
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 32
DW 50.000 usec
DE 6.00 usec
TE 295.7 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
default proton parameters
0.441
1.147
1.162
1.230
1.243
1.664
1.679
1.686
1.694
1.709
1.724
3.921
3.935
3.949
3.962
3.976
4.810
4.826
6.785
6.792
6.869
6.887
7.260
7.288
7.294
7.476
7.494
TMS
TIPS
75
9.359
18.720
6.445
3.451
0.935
0.140
0.905
0.128
0.936
1.005
1.005
1.000
ppm

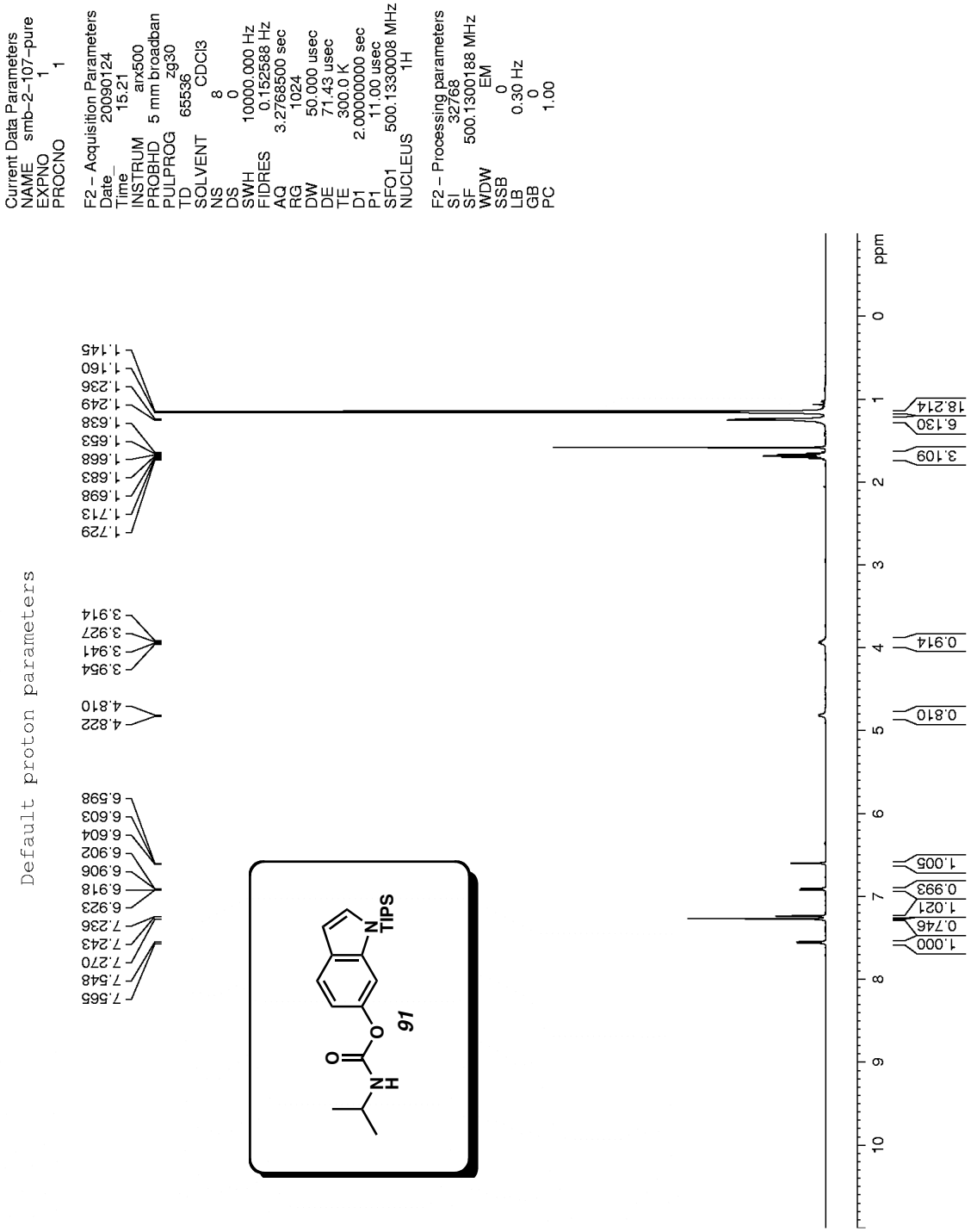

Current Data Parameters
NAME smb-2-196check
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100520
Time 22.05
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 57
DW 50.000 usec
DE 6.00 usec
TE 297.6 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300174 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
default proton parameters
0.534
1.158
1.173
1.651
1.675
1.690
1.705
1.720
1.735
1.750
6.836
6.842
7.115
7.133
7.269
7.378
7.384
7.522
7.540
TMS
TfO
N
TIPS
76
9.448
18.910
3.245
1.042
1.013
1.017
1.000
ppm

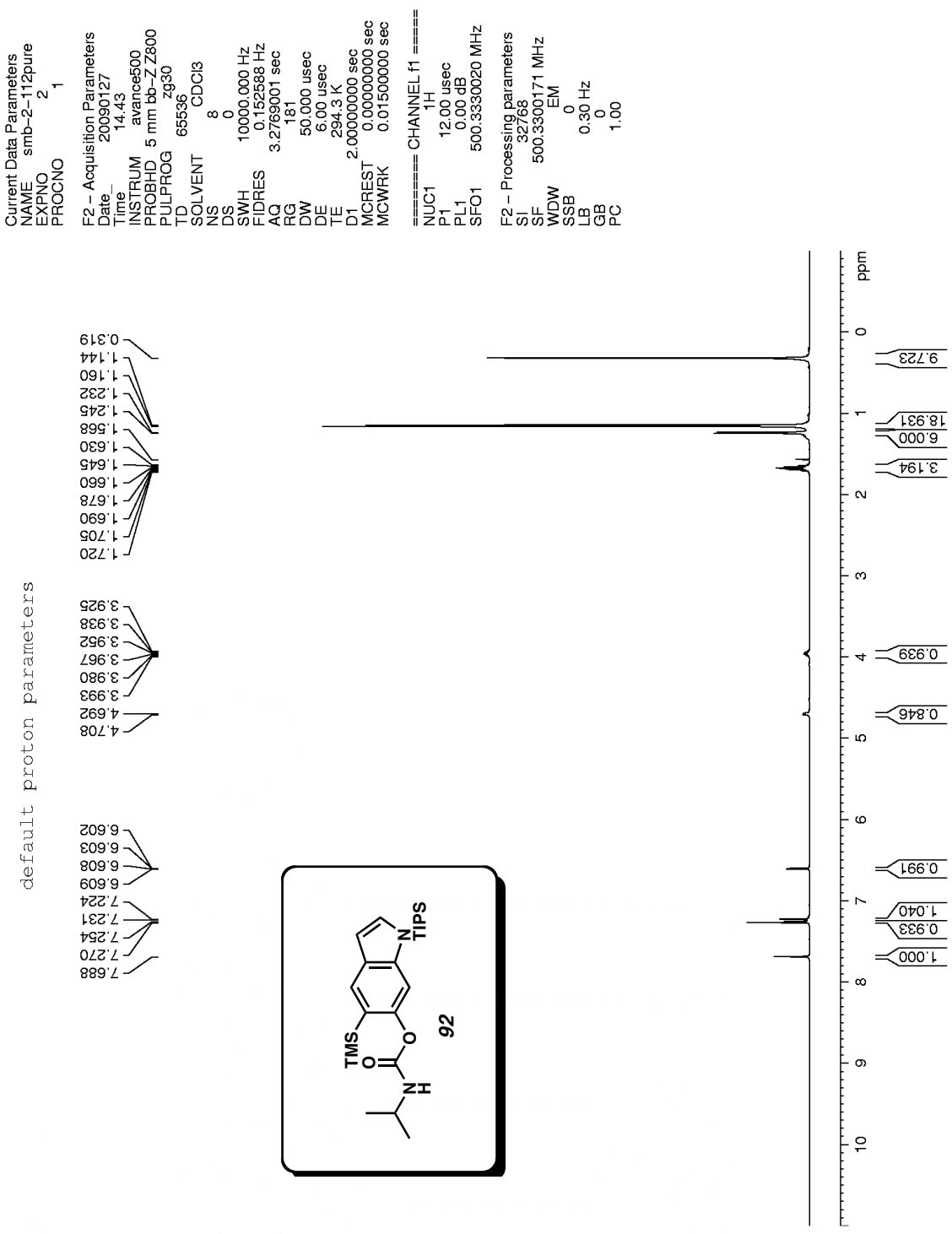

Current Data Parameters
NAME GJI-II-144+147
EXPNO 20
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090924
Time 17.07
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 180
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300238 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-II-144 + 147 1H
8.331
7.360
7.358
7.342
7.340
7.305
7.300
7.294
7.167
7.149
6.784
6.782
6.779
6.777
6.775
6.773
6.771
0.556
TMS
TfO
77
9.346
1.000
1.010
1.008
1.000
0.902
ppm

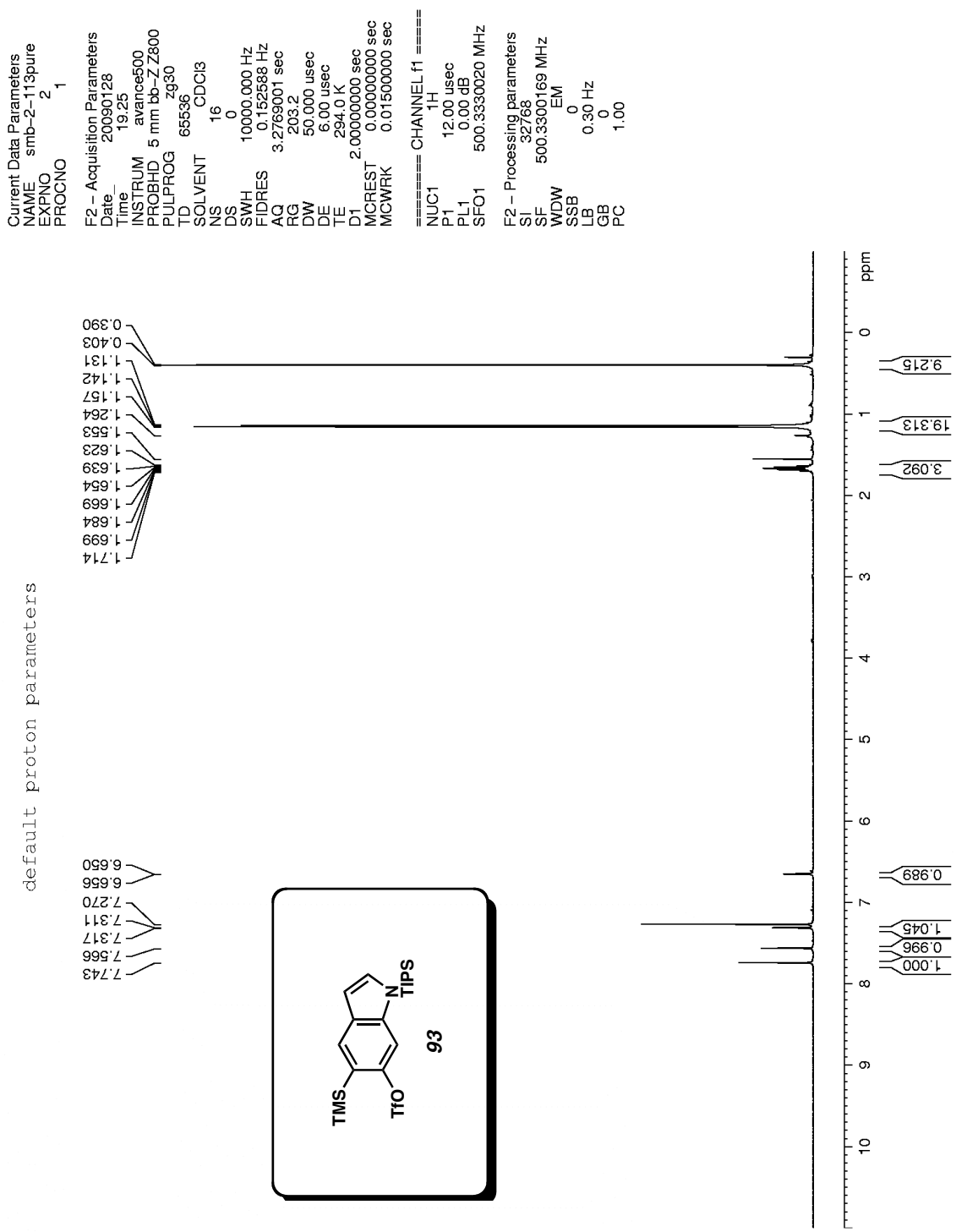

Current Data Parameters
NAME GJI-II-148+152
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date 20090924
Time 18.06
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 180
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300235 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-II-148 + 152 1H
8.264
8.246
7.732
7.724
7.362
7.275
7.260
7.257
6.783
6.776
1.679
0.509
TMS
TfO
N
Boc
78
9.136
9.135
1.000
1.089
1.002
0.998
ppm

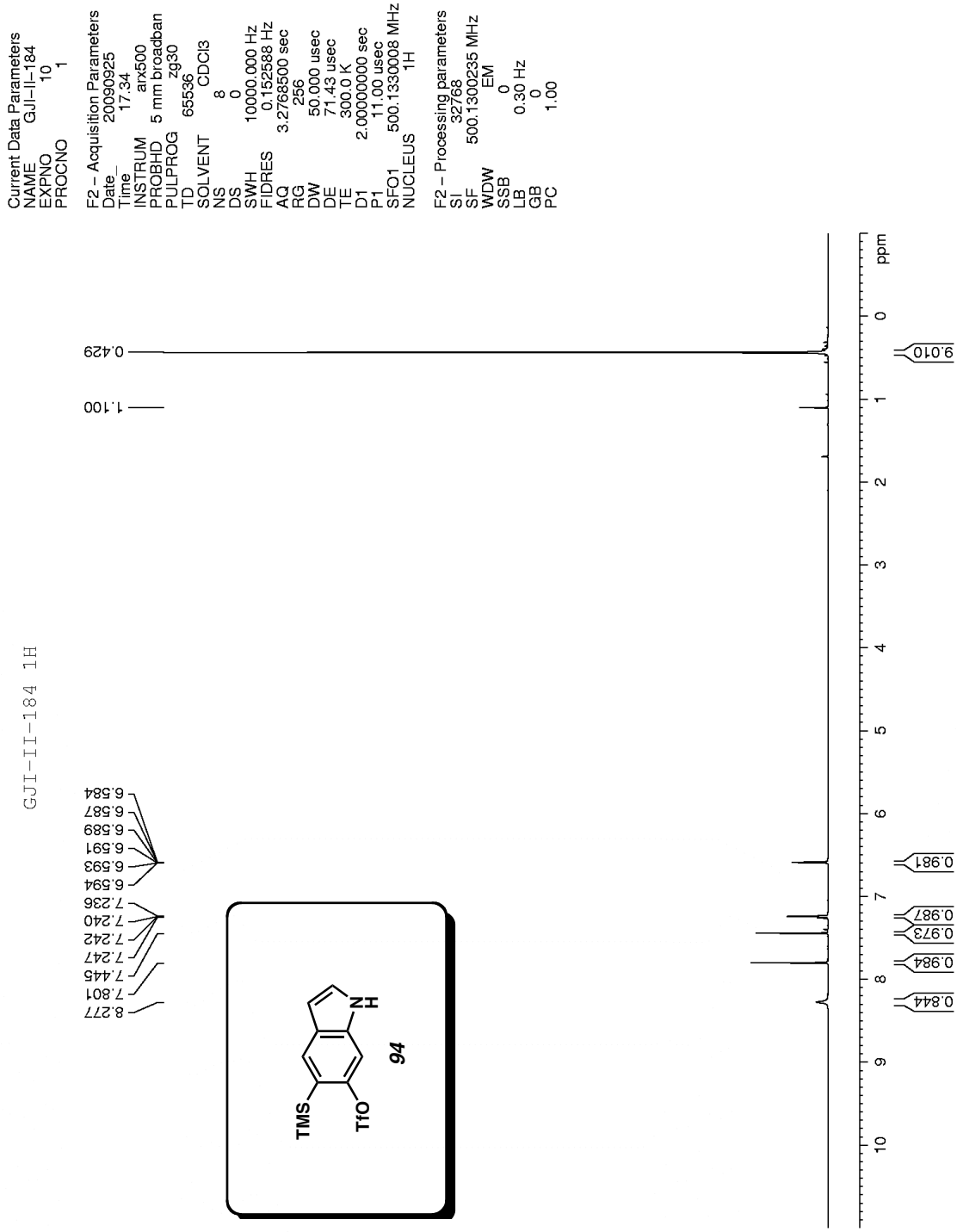

Current Data Parameters
NAME smb-3-177p1
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100427
Time 19.43
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 4096
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 12.20 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300186 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Default proton parameters
1.567
5.852
5.853
5.855
5.856
6.032
6.034
6.035
6.487
6.489
6.491
6.494
6.496
6.498
6.500
7.009
7.011
7.025
7.026
7.138
7.141
7.149
7.152
7.160
7.164
7.171
7.175
7.201
7.217
7.229
7.235
7.236
7.241
7.270
8.113
80
1.000
0.981
0.987
1.020
2.054
0.960
1.100
0.851
ppm

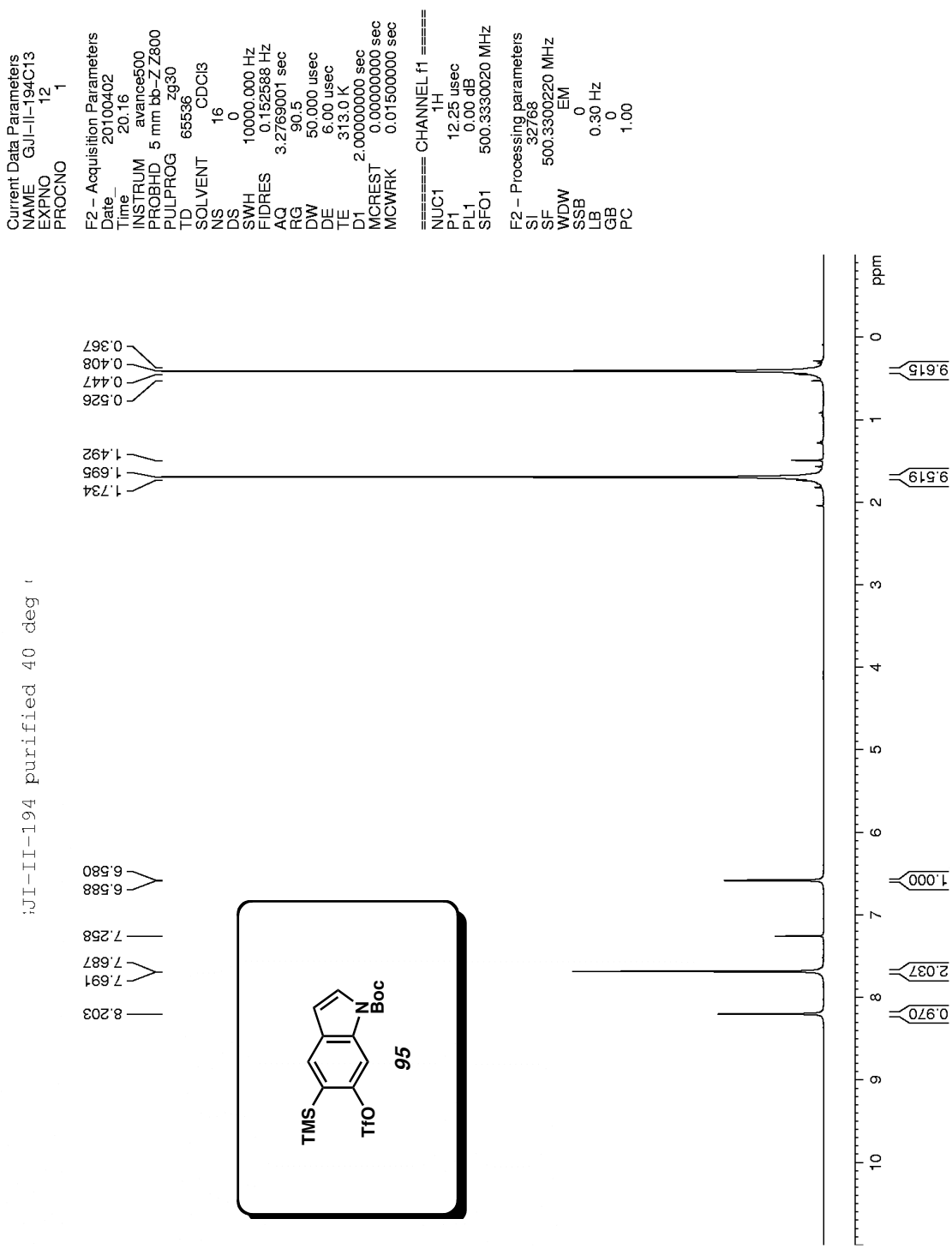

Current Data Parameters
NAME smb-2-106pure
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date 20090125
Time 22.00
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 4096
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300190 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Default proton parameters
1.085
1.099
1.545
1.563
1.575
1.590
1.605
5.132
6.535
6.542
6.878
6.882
6.895
6.899
6.999
7.003
7.124
7.131
7.297
7.312
7.326
7.364
7.380
7.394
7.451
7.465
7.489
7.506
BnO
N
TIPS
90
19.027
0.621
2.110
2.073
1.000
1.022
1.026
1.008
1.101
2.061
2.126
1.046
ppm

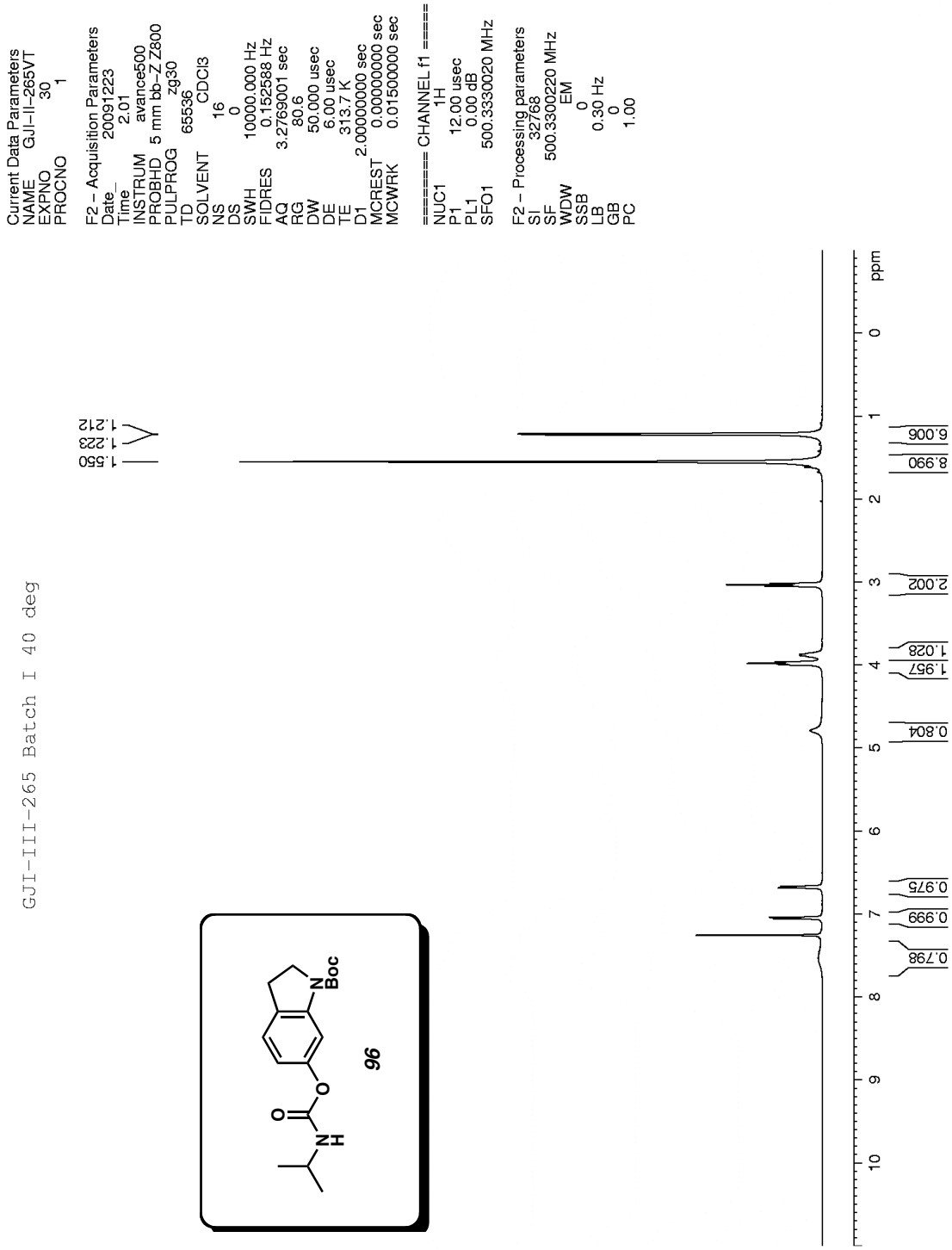

Current Data Parameters
NAME smb-2-107-pure
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090124
Time 15.21
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 1024
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300188 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Default proton parameters
7.565
7.548
7.270
7.243
7.236
6.923
6.918
6.906
6.902
6.604
6.603
6.598
4.822
4.810
3.954
3.941
3.927
3.914
1.729
1.713
1.698
1.683
1.668
1.653
1.638
1.249
1.236
1.160
1.145
TIPS
N
O
O
H
N
91
ppm
1.000
0.746
1.021
0.993
1.005
0.810
0.914
3.109
6.130
18.214

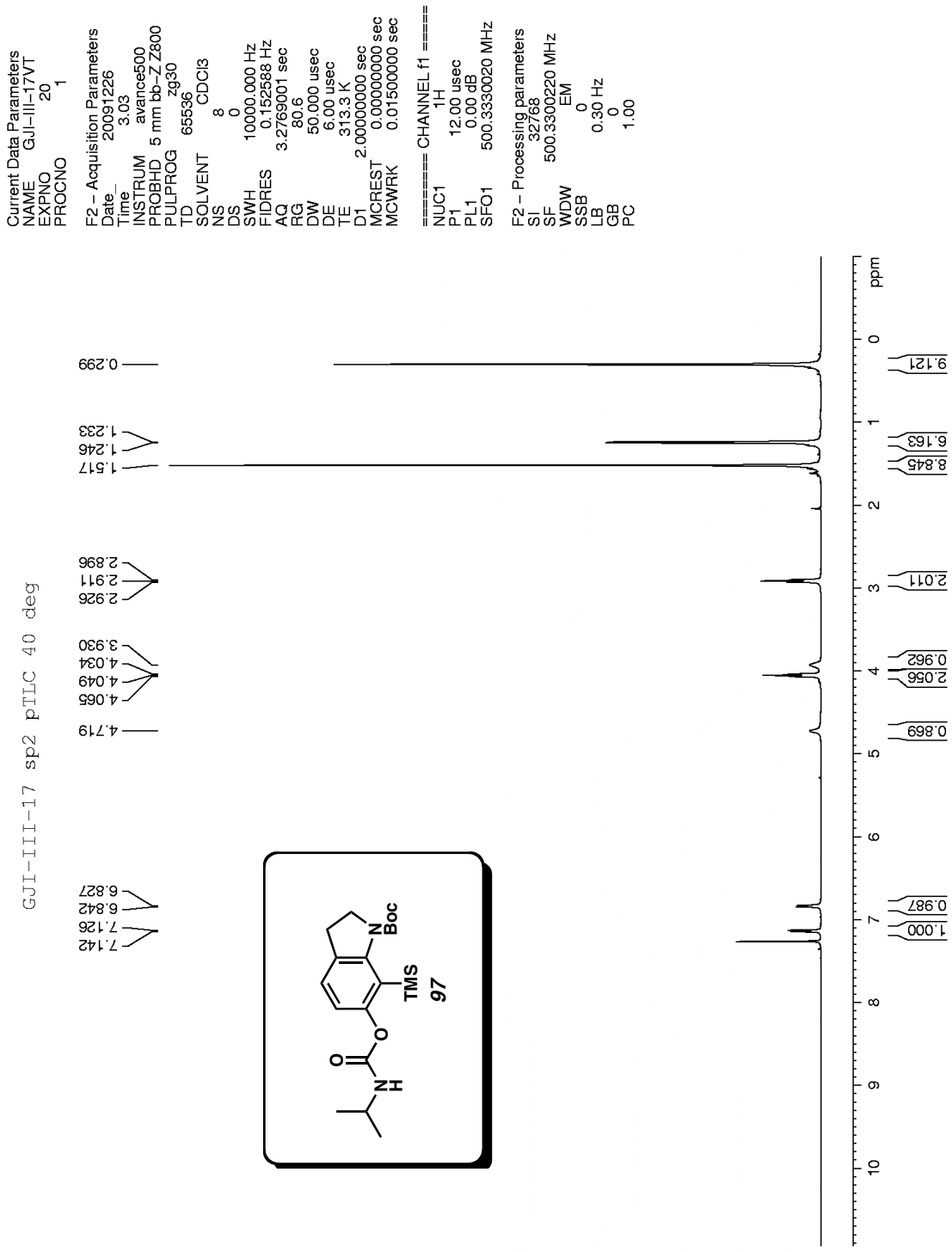

Current Data Parameters
NAME smb-2-112pure
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090127
Time 14.43
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 294.3 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300171 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
default proton parameters
0.319
1.144
1.160
1.232
1.245
1.568
1.630
1.645
1.660
1.678
1.690
1.705
1.720
3.925
3.938
3.952
3.967
3.980
3.993
4.692
4.708
6.602
6.603
6.608
6.609
7.224
7.231
7.254
7.270
7.688
TMS
O
O
N
H
N
TIPS
92
ppm
0
1
2
3
4
5
6
7
8
9
10
9.723
18.931
6.000
3.194
0.939
0.846
0.991
1.040
0.933
1.000

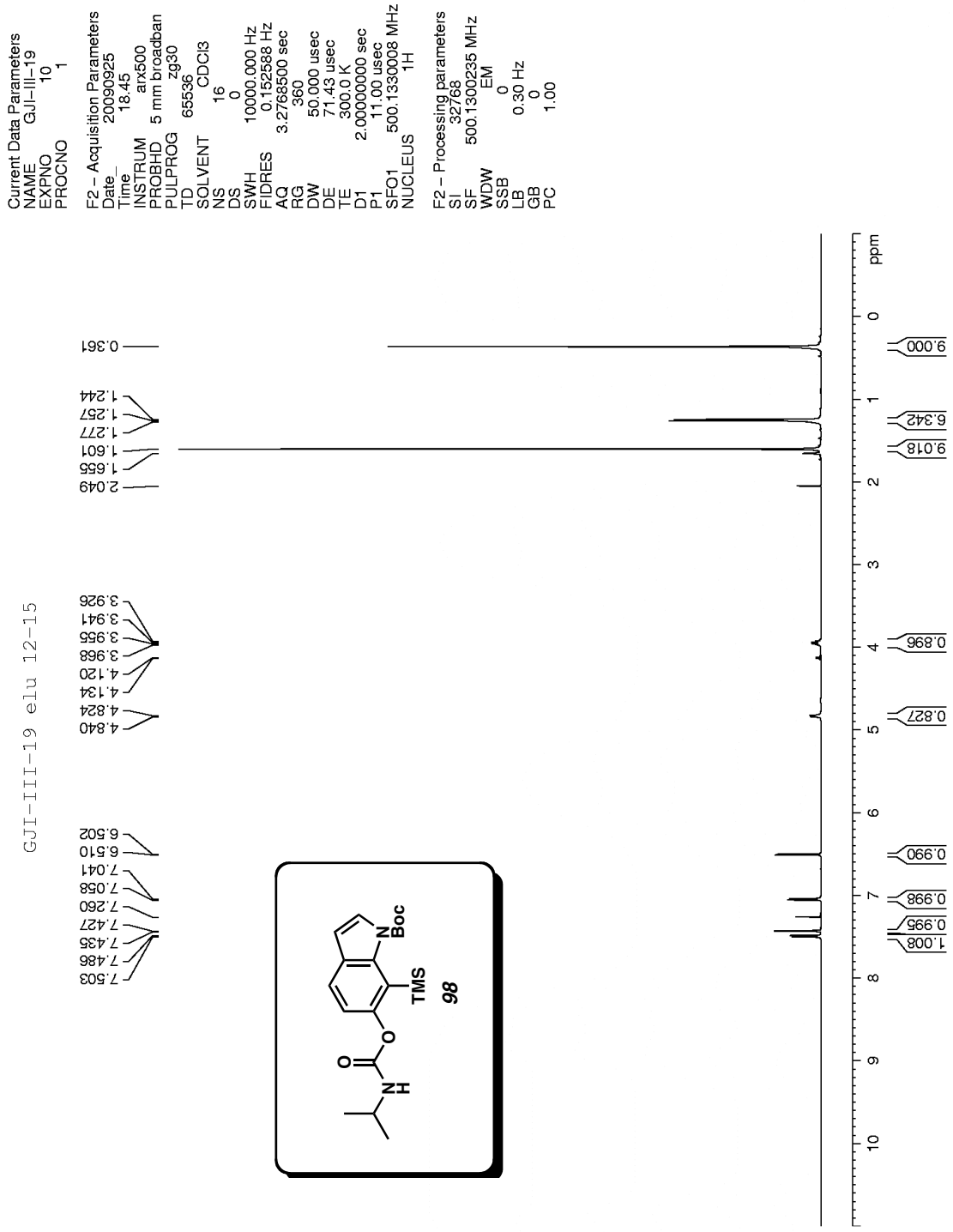

Current Data Parameters
NAME smb-2-113pure
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090128
Time 19.25
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 203.2
DW 50.000 usec
DE 6.00 usec
TE 294.0 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300169 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
default proton parameters
0.390
0.403
1.131
1.142
1.157
1.264
1.553
1.623
1.639
1.654
1.669
1.684
1.699
1.714
6.650
6.656
7.270
7.311
7.317
7.566
7.743
TMS
TfO
N
TIPS
93
ppm
9.215
19.313
3.092
0.989
1.045
0.996
1.000

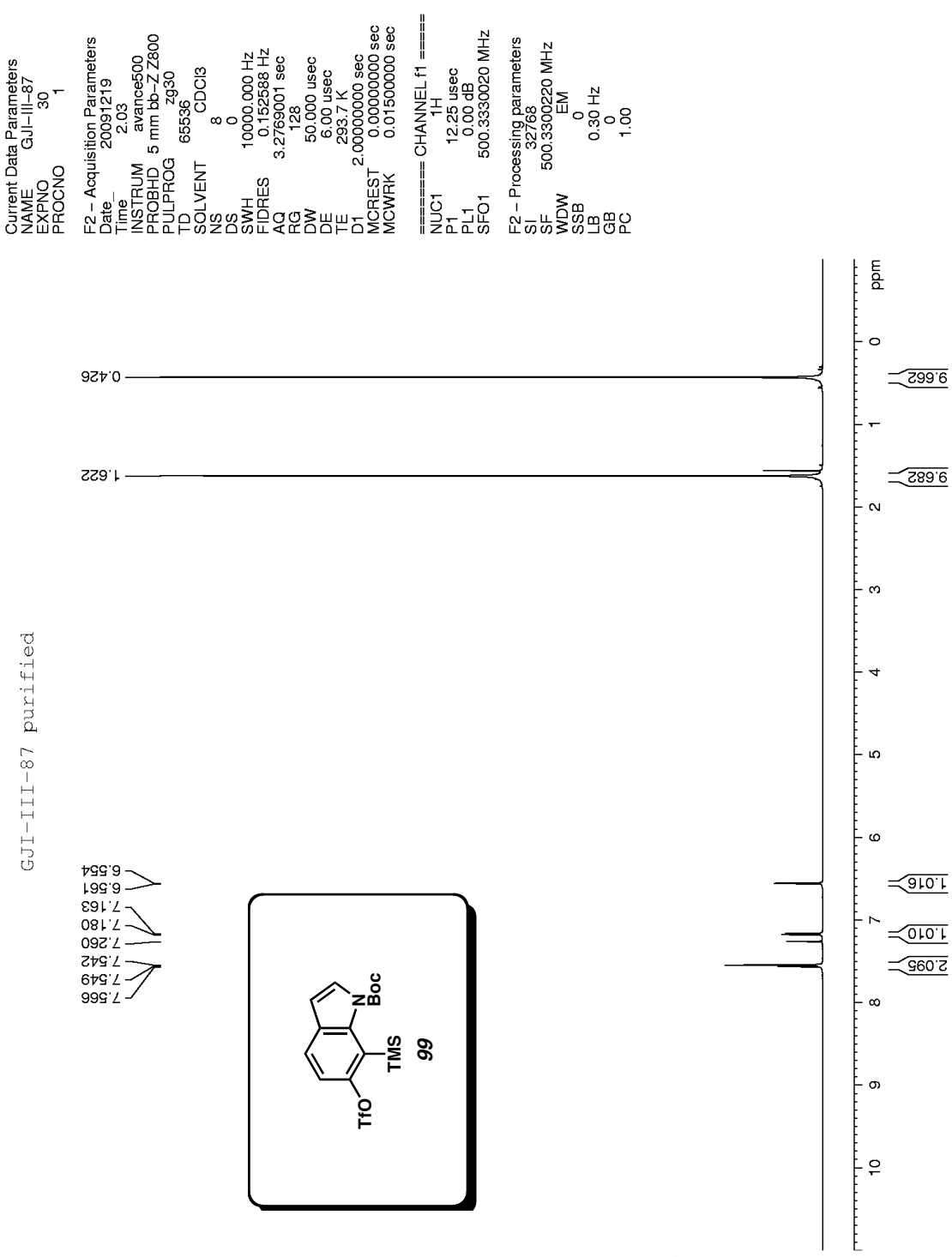

GJI-II-184 1H
Current Data Parameters
NAME GJI-II-184
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090925
Time 17.34
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 256
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300235 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.429
1.100
8.277
7.801
7.445
7.247
7.242
7.240
7.236
6.594
6.593
6.591
6.589
6.587
6.584
TMS
TfO
94
9.010
0.981
0.987
0.973
0.984
0.844
ppm

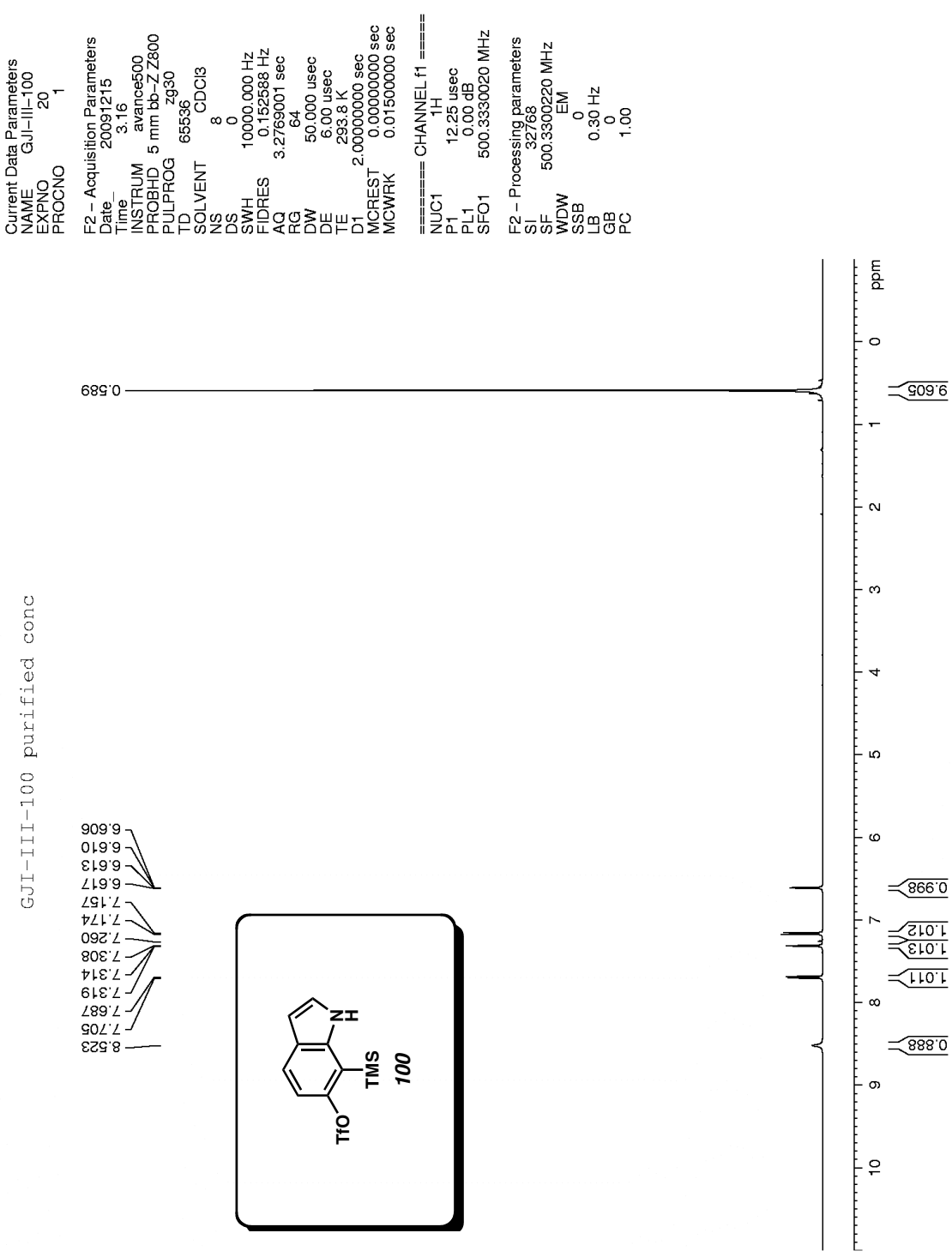

Current Data Parameters
NAME GJI-II-194C13
EXPNO 12
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100402
Time 20.16
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 90.5
DW 50.000 usec
DE 6.00 usec
TE 313.0 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
jJI-II-194 purified 40 deg
8.203
7.691
7.687
7.258
6.588
6.580
1.734
1.695
1.492
0.526
0.447
0.408
0.367
TMS
TfO
N
Boc
95
9.615
9.519
1.000
2.037
0.970
ppm

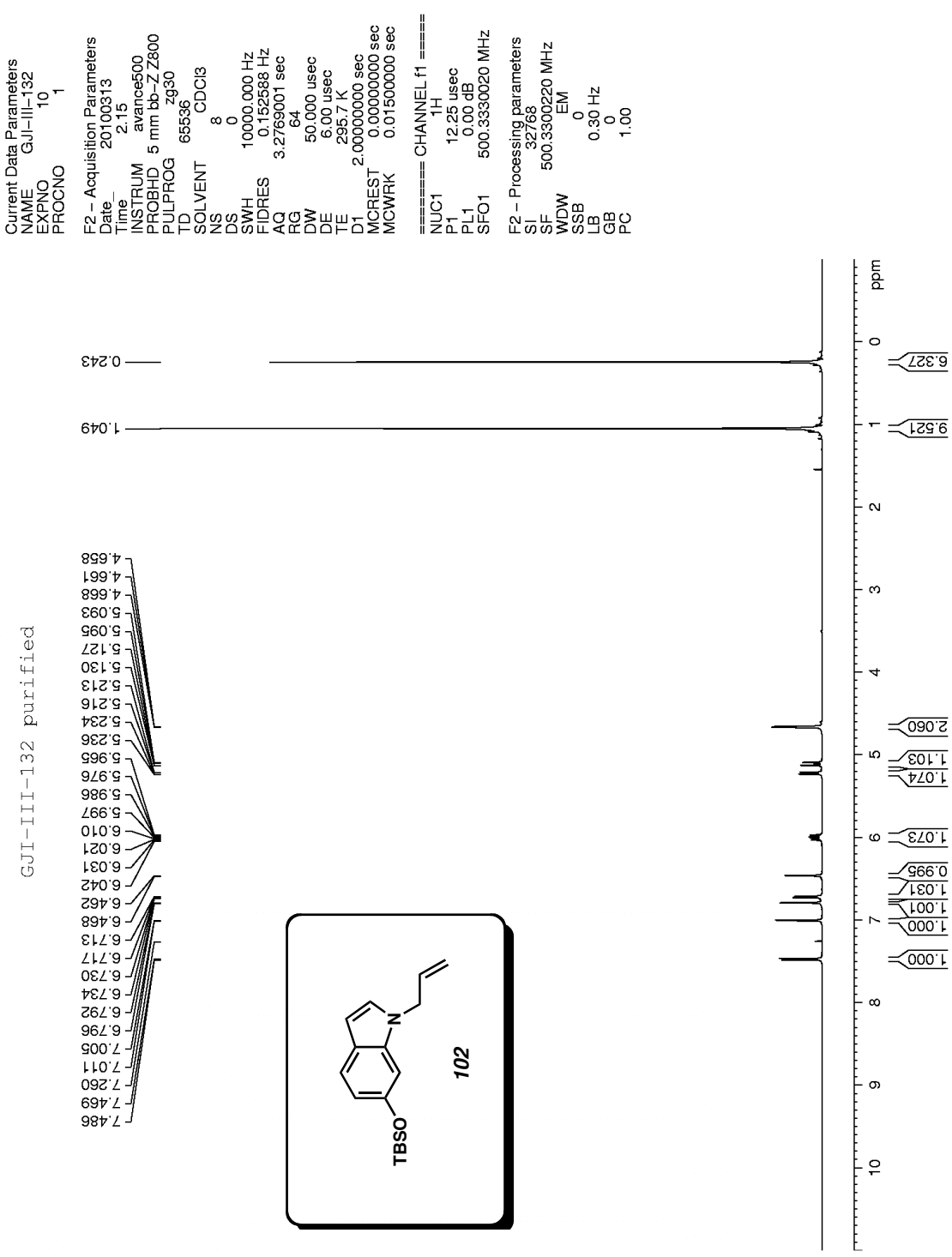

Current Data Parameters
NAME GJI-II-265VT
EXPNO 30
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091223
Time 2.01
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 80.6
DW 50.000 usec
DE 6.00 usec
TE 313.7 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-III-265 Batch I 40 deg
1.550
1.223
1.212
Boc
N
O
O
N
H
96
ppm
0
1
2
3
4
5
6
7
8
9
10
6.006
8.990
2.002
1.028
1.957
0.804
0.975
0.999
0.798

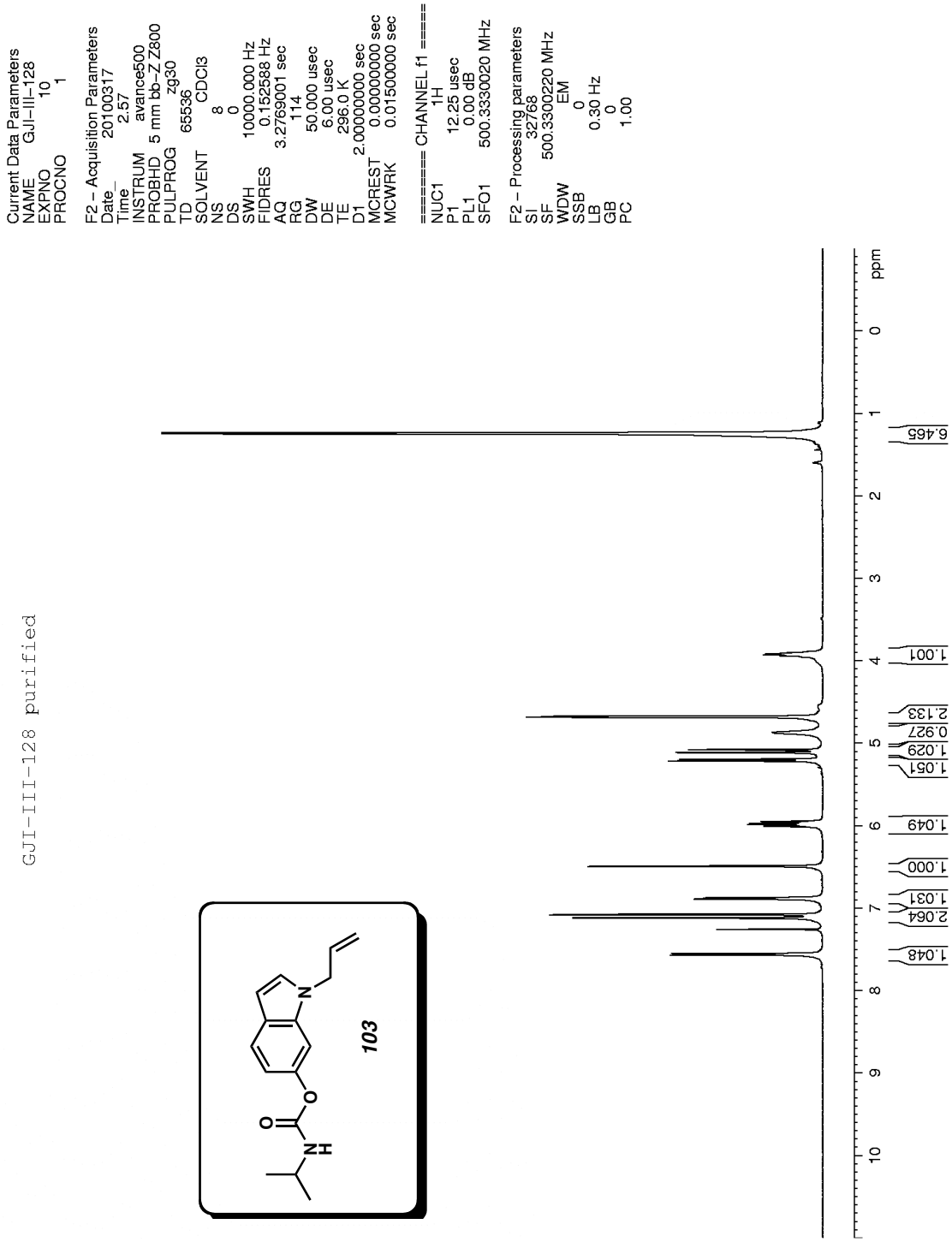

Current Data Parameters
NAME GJI-III-17VT
EXPNO 20
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091226
Time 3.03
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 80.6
DW 50.000 usec
DE 6.00 usec
TE 313.3 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-III-17 sp2 pTLC 40 deg
7.142
7.126
6.842
6.827
4.719
4.065
4.049
4.034
3.930
2.926
2.911
2.896
1.517
1.246
1.233
0.299
N
Boc
TMS
O
O
N
H
97
ppm
0
1
2
3
4
5
6
7
8
9
10
9.121
8.845
6.163
2.011
2.056
0.962
0.869
1.000
0.987

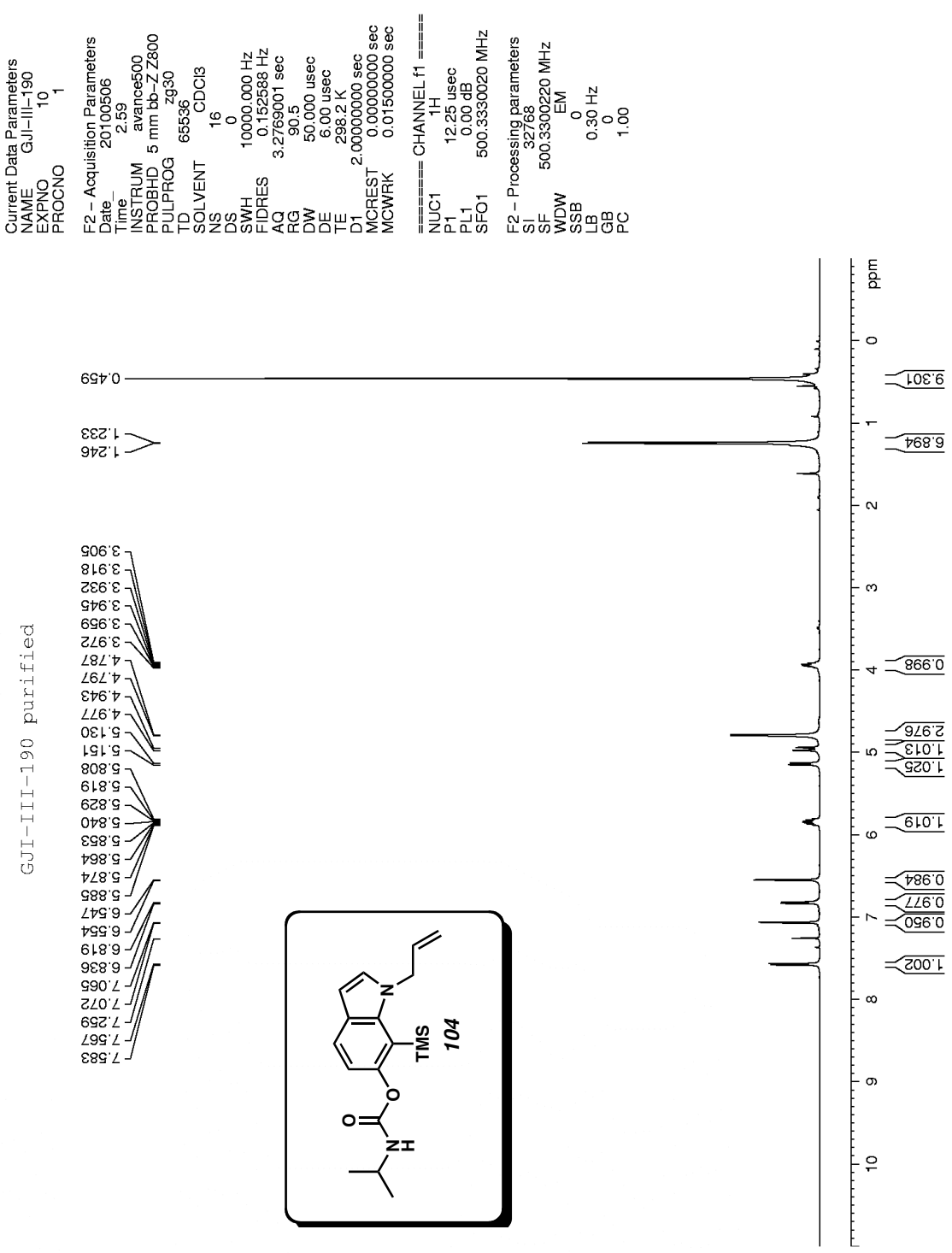

Current Data Parameters
NAME GJI-III-19
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090925
Time_ 18.45
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 360
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300235 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-III-19 elu 12-15
0.361
1.244
1.257
1.277
1.601
1.655
2.049
3.926
3.941
3.955
3.968
4.120
4.134
4.824
4.840
6.502
6.510
7.041
7.058
7.260
7.427
7.435
7.486
7.503
N
Boc
TMS
O
O
N
H
98
9.000
6.342
9.018
0.896
0.827
0.990
0.998
0.995
1.008
ppm

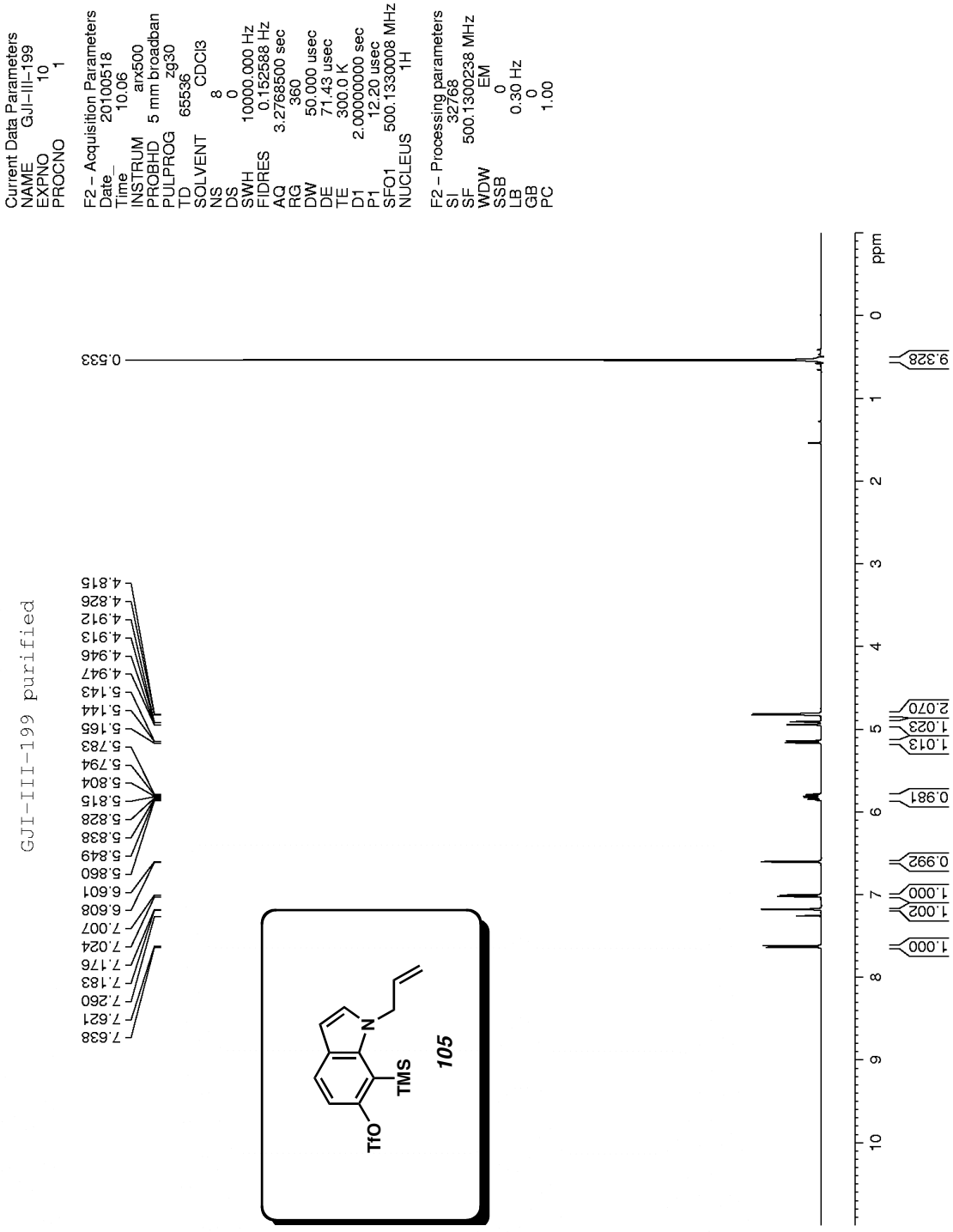

GJI-III-87 purified
Current Data Parameters
NAME GJI-III-87
EXPNO 30
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091219
Time_ 2.03
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 128
DW 50.000 usec
DE 6.00 usec
TE 293.7 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.566
7.549
7.542
7.260
7.180
7.163
6.561
6.554
0.426
1.622
9.662
9.682
1.016
1.010
2.095
ppm
N
Boc
TMS
TfO
99

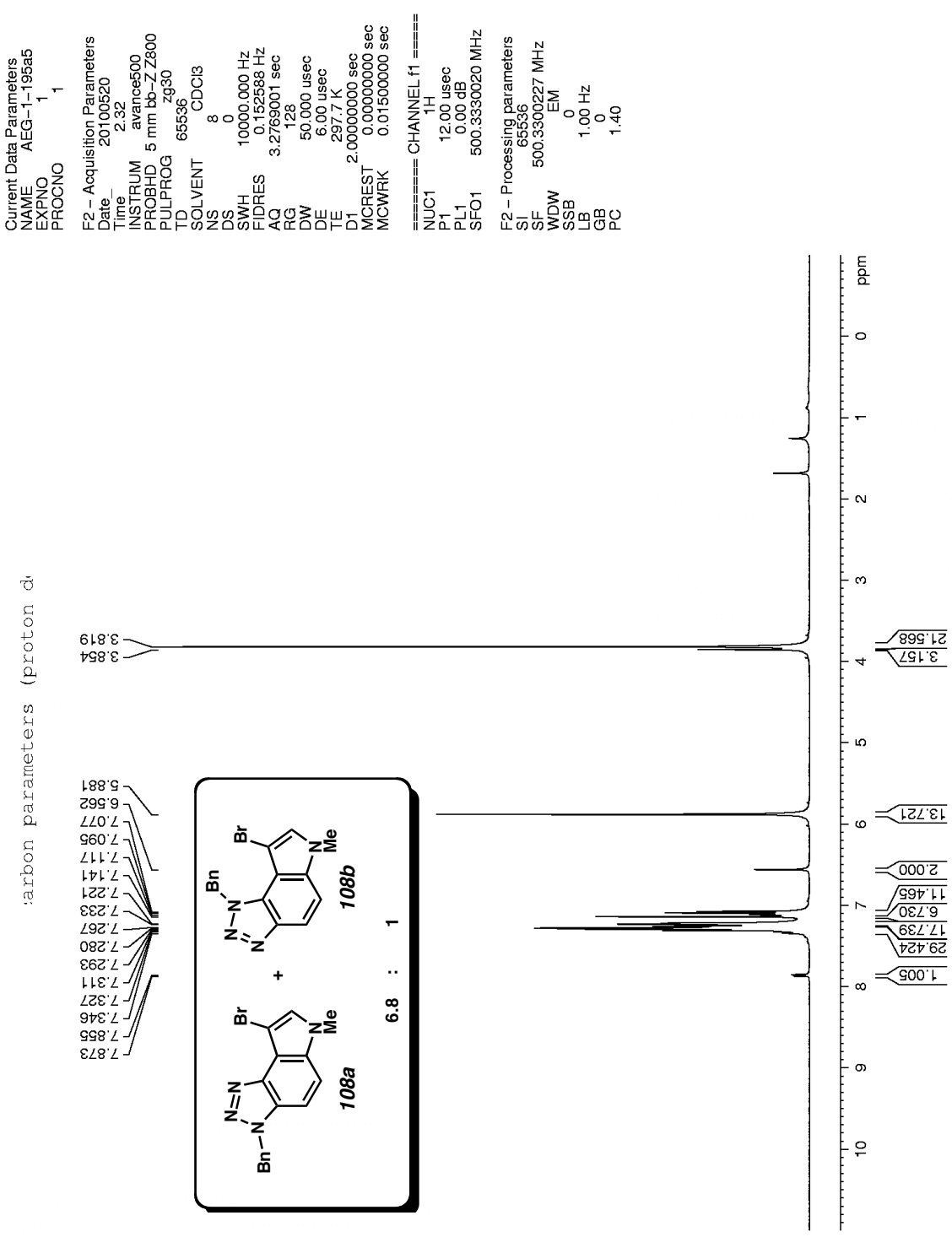

GJI-III-100 purified conc
Current Data Parameters
NAME GJI-III-100
EXPNO 20
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091215
Time 3.16
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 64
DW 50.000 usec
DE 6.00 usec
TE 293.8 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.623
7.705
7.687
7.319
7.314
7.308
7.260
7.174
7.157
6.617
6.613
6.610
6.606
0.589
TfO
TMS
N
H
100
9.605
0.998
1.012
1.013
1.011
0.888
ppm

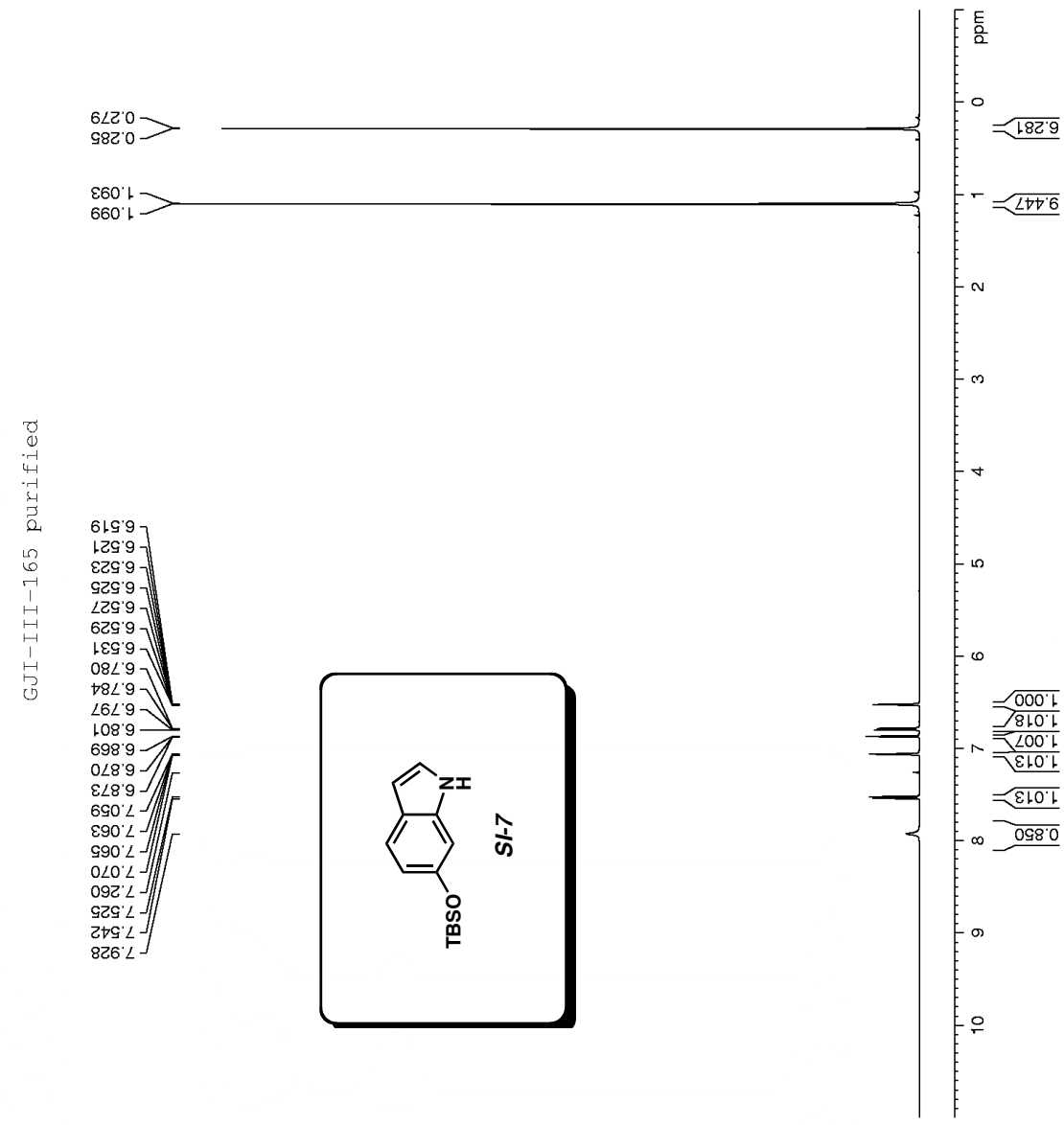

GJI-III-132 purified
Current Data Parameters
NAME GJI-III-132
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100313
Time_ 2.15
INSTRUM avance500
PROBHD 5 mm bb–Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 64
DW 50.000 usec
DE 6.00 usec
TE 295.7 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.243
1.049
4.658
4.661
4.668
5.093
5.095
5.127
5.130
5.213
5.216
5.234
5.236
5.965
5.976
5.986
5.997
6.010
6.021
6.031
6.042
6.462
6.468
6.713
6.717
6.730
6.734
6.792
6.796
7.005
7.011
7.260
7.469
7.486
TBSO
N
102
ppm
6.327
9.521
2.060
1.074
1.103
1.073
0.995
1.031
1.001
1.000
1.000

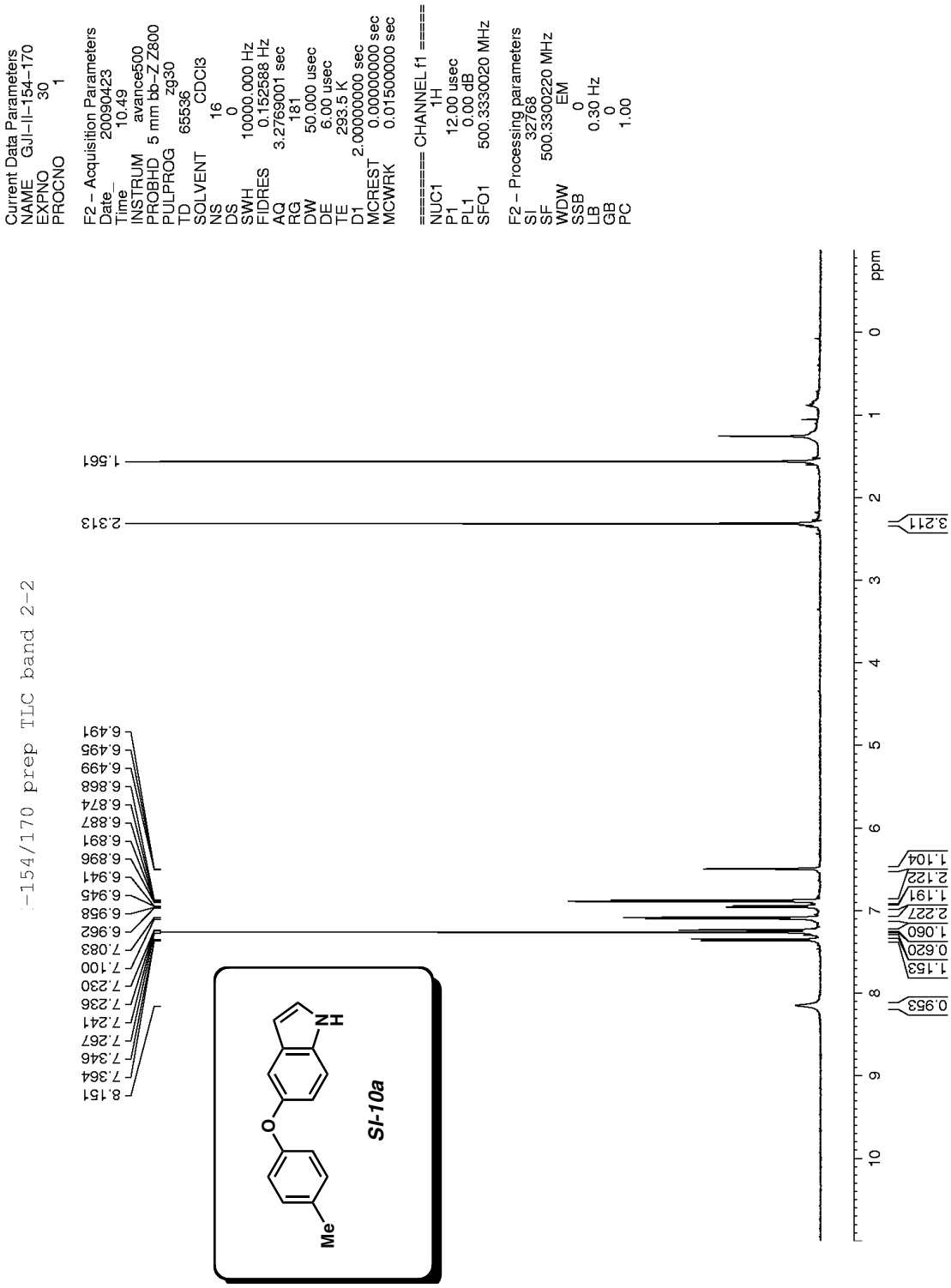

GJI-III-128 purified
Current Data Parameters
NAME GJI-III-128
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100317
Time_ 2.57
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 114
DW 50.000 usec
DE 6.00 usec
TE 296.0 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
103
6.465
1.001
2.133
0.927
1.029
1.051
1.049
1.000
1.031
2.064
1.048
ppm

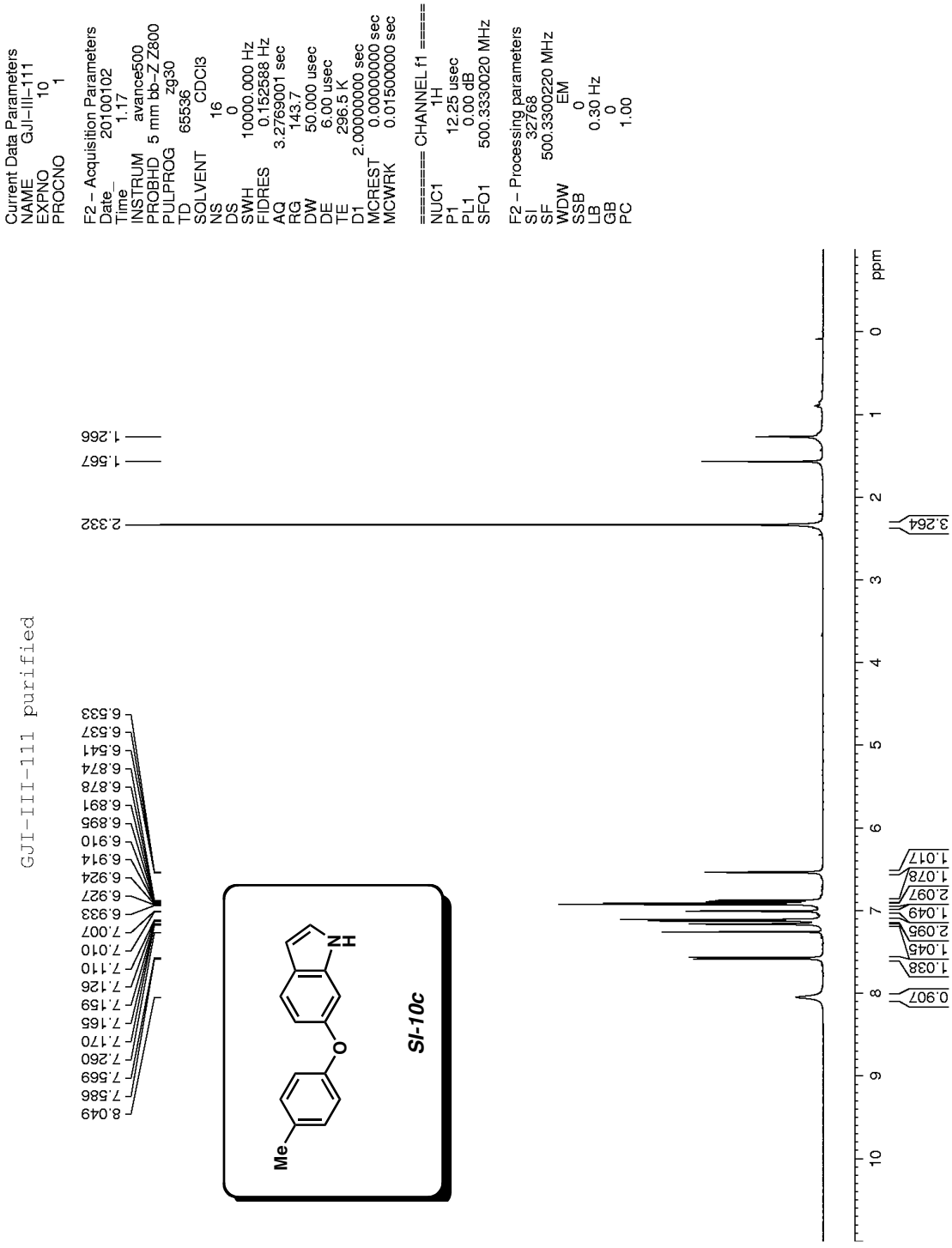

GJI-III-190 purified
Current Data Parameters
NAME GJI-III-190
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100506
Time 2.59
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 90.5
DW 50.000 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.459
1.233
1.246
3.905
3.918
3.932
3.945
3.959
3.972
4.787
4.797
4.943
4.977
5.130
5.151
5.808
5.819
5.829
5.840
5.853
5.864
5.874
5.885
6.547
6.554
6.819
6.836
7.065
7.072
7.259
7.567
7.583
TMS
104
9.301
6.894
0.998
2.976
1.013
1.025
1.019
0.984
0.977
0.950
1.002
ppm

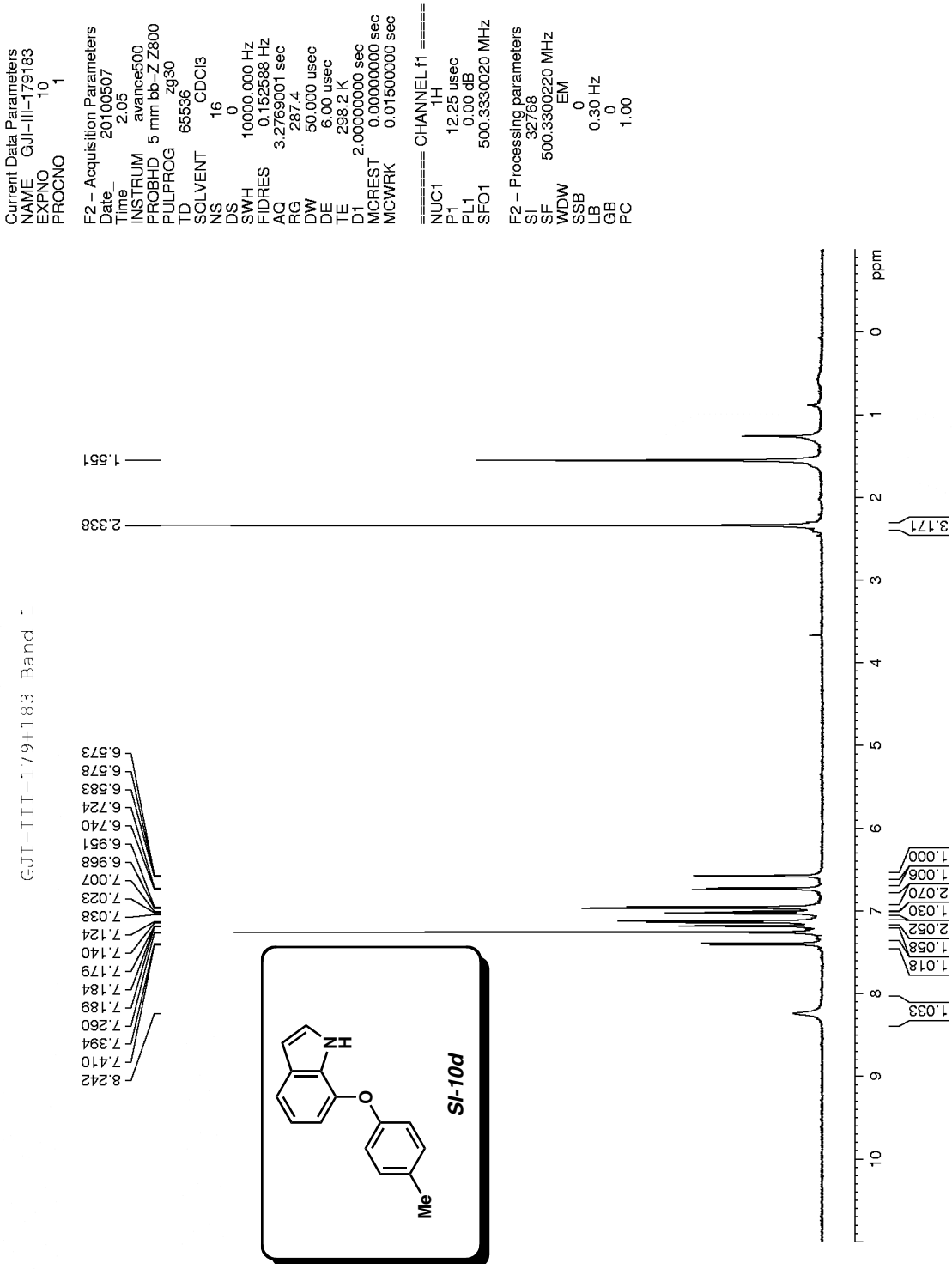

GJI-III-199 purified
Current Data Parameters
NAME GJI-III-199
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100518
Time_ 10.06
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 360
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 12.20 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300238 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.533
4.815
4.826
4.912
4.913
4.946
4.947
5.143
5.144
5.165
5.783
5.794
5.804
5.815
5.828
5.838
5.849
5.860
6.601
6.608
7.007
7.024
7.176
7.183
7.260
7.621
7.638
TfO
TMS
N
105
9.328
2.070
1.023
1.013
0.981
0.992
1.000
1.002
1.000
ppm

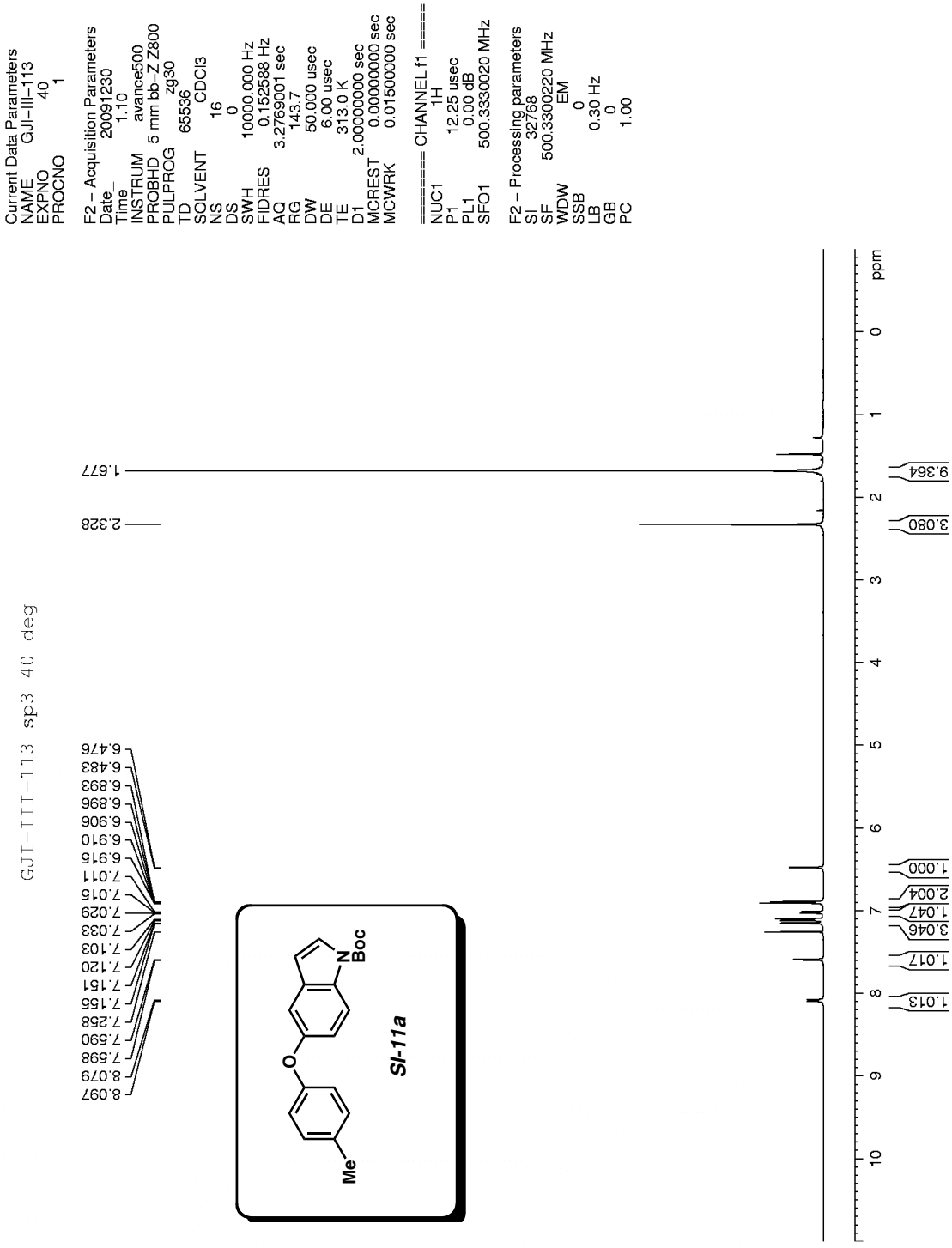

Current Data Parameters
NAME AEG-1-195a5
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100520
Time 2.32
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 128
DW 50.000 usec
DE 6.00 usec
TE 297.7 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 65536
SF 500.3300227 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
carbon parameters (proton d
7.873
7.855
7.346
7.327
7.311
7.293
7.280
7.267
7.233
7.221
7.141
7.117
7.095
7.077
6.562
5.881
3.854
3.819
Br
N
Me
Bn
N
N
N
N
108a
108b
6.8 : 1
3.157
21.568
13.721
2.000
11.465
6.730
17.739
29.424
1.005
ppm

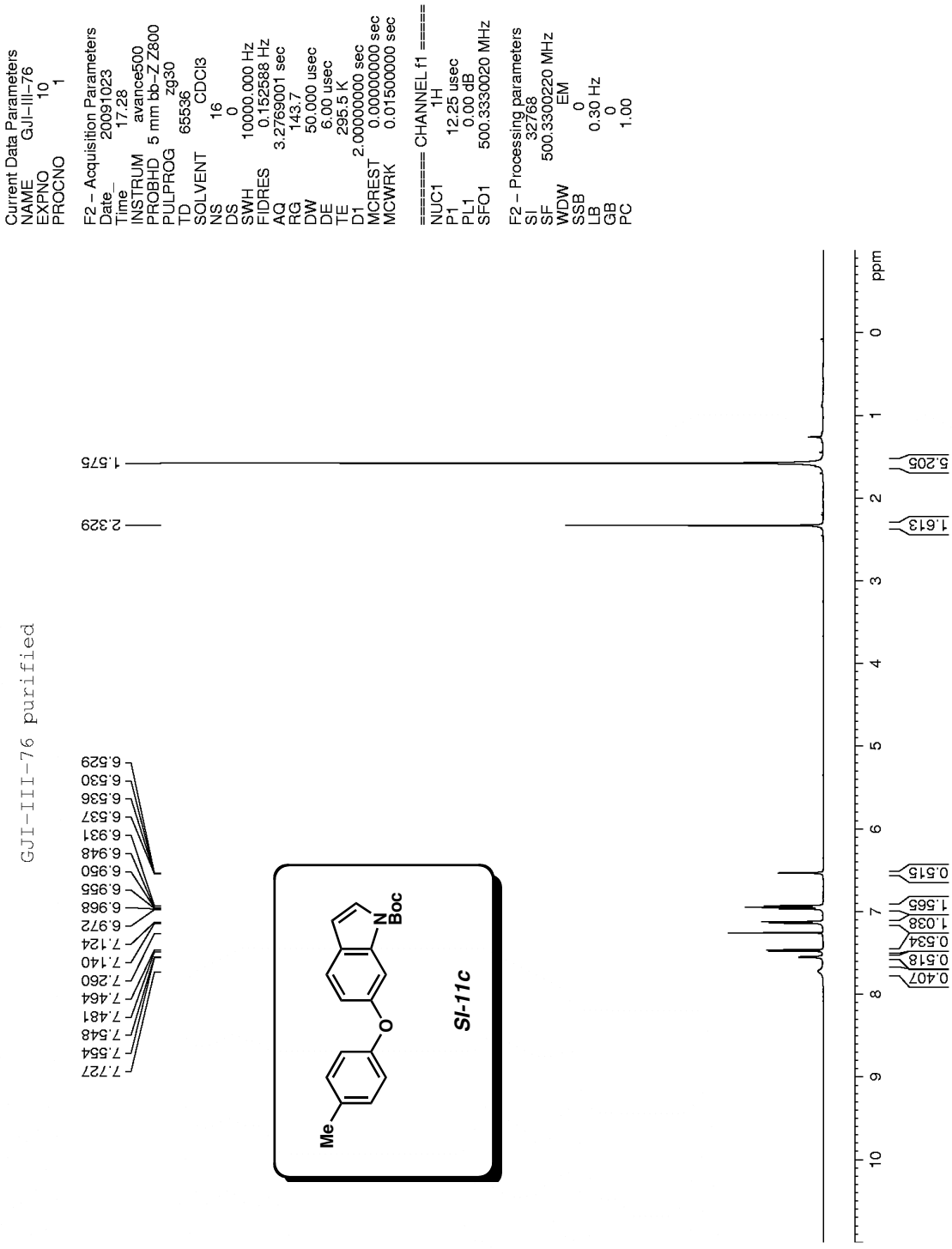

GJI-III-165 purified
Current Data Parameters
NAME GJI-III-165
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100420
Time 20.44
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 128
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 12.20 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300235 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.928
7.542
7.525
7.260
7.070
7.065
7.063
7.059
6.873
6.870
6.869
6.801
6.797
6.784
6.780
6.531
6.529
6.527
6.525
6.523
6.521
6.519
1.099
1.093
0.285
0.279
TBSO
H
N
SI-7
ppm
0
1
2
3
4
5
6
7
8
9
10
6.281
9.447
0.850
1.013
1.013
1.007
1.018
1.000

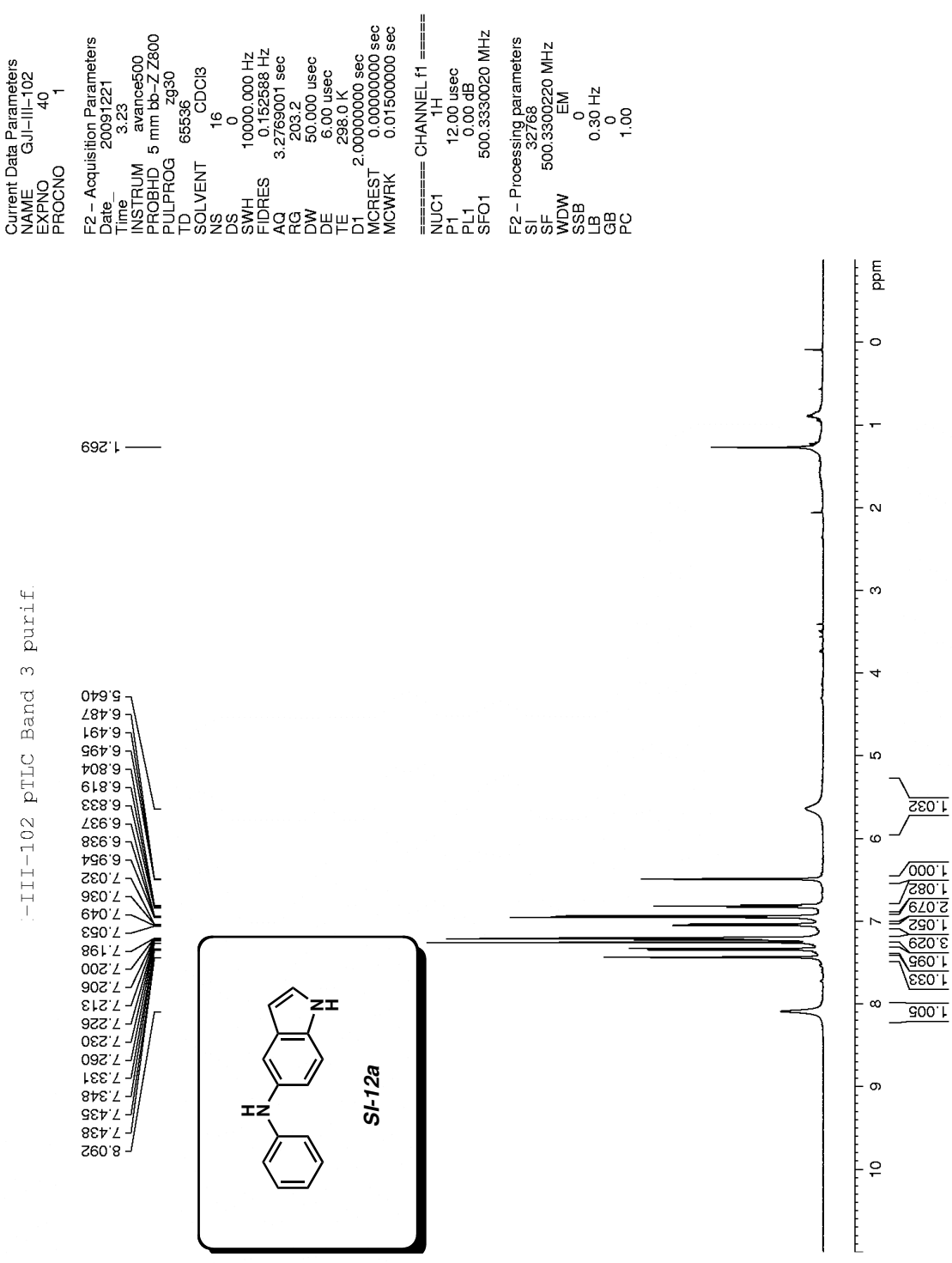

Current Data Parameters
NAME GJI-II-154-170
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090423
Time 10.49
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 293.5 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-154/170 prep TLC band 2-2
8.151
7.364
7.346
7.267
7.241
7.236
7.230
7.100
7.083
6.962
6.958
6.945
6.941
6.896
6.891
6.887
6.874
6.868
6.499
6.495
6.491
2.313
1.561
3.211
1.104
2.122
1.191
2.227
1.060
0.620
1.153
0.953
ppm
Me
O
N
H
SI-10a

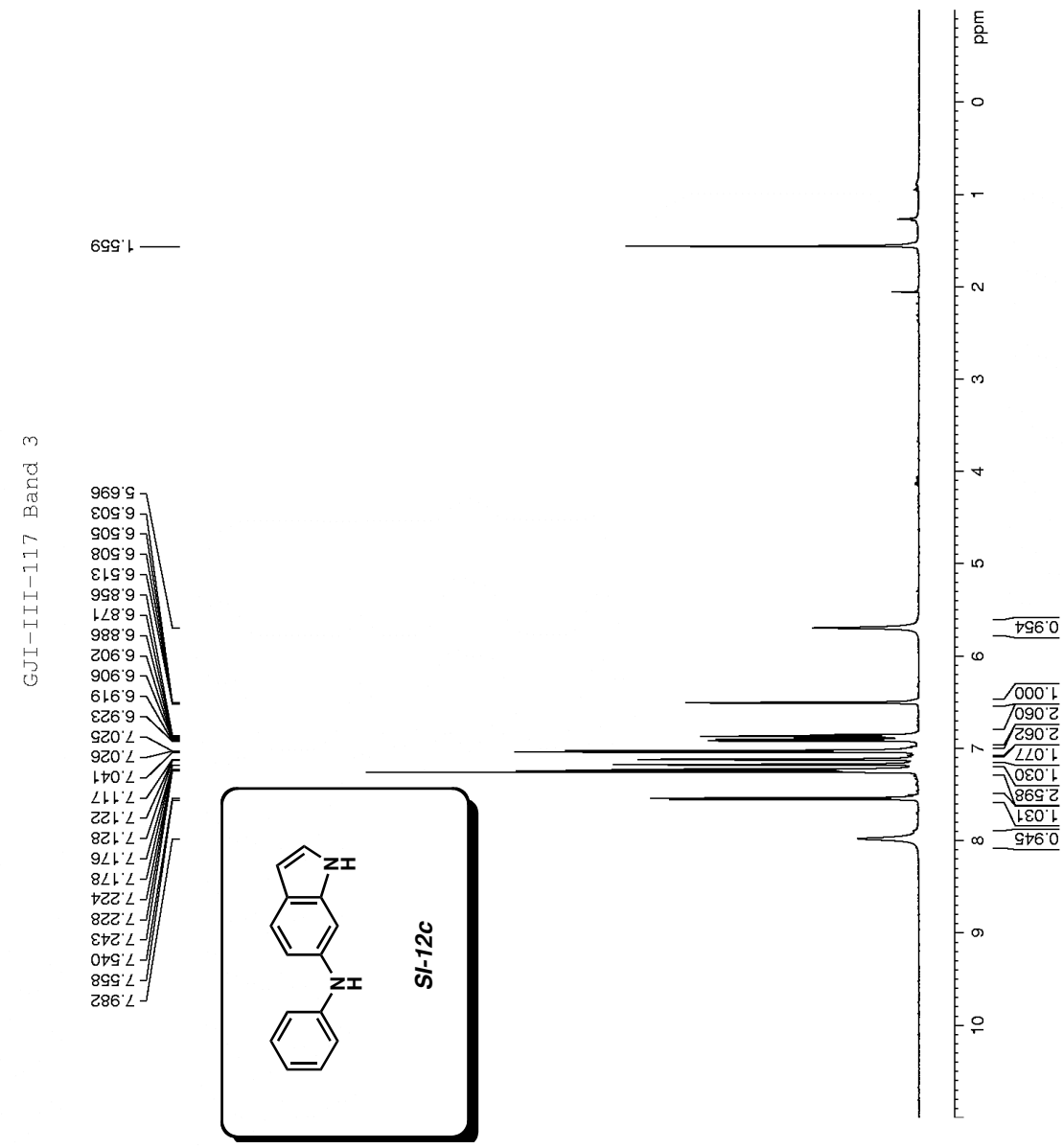

GJI-III-111 purified
Current Data Parameters
NAME GJI-III-111
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100102
Time_ 1.17
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 143.7
DW 50.000 usec
DE 6.00 usec
TE 296.5 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.049
7.586
7.569
7.260
7.170
7.165
7.159
7.126
7.110
7.010
7.007
6.933
6.927
6.924
6.914
6.910
6.895
6.891
6.878
6.874
6.541
6.537
6.533
2.332
1.567
1.266
Me
SI-10c
3.264
1.017
1.078
2.097
1.049
2.095
1.045
1.038
0.907
ppm

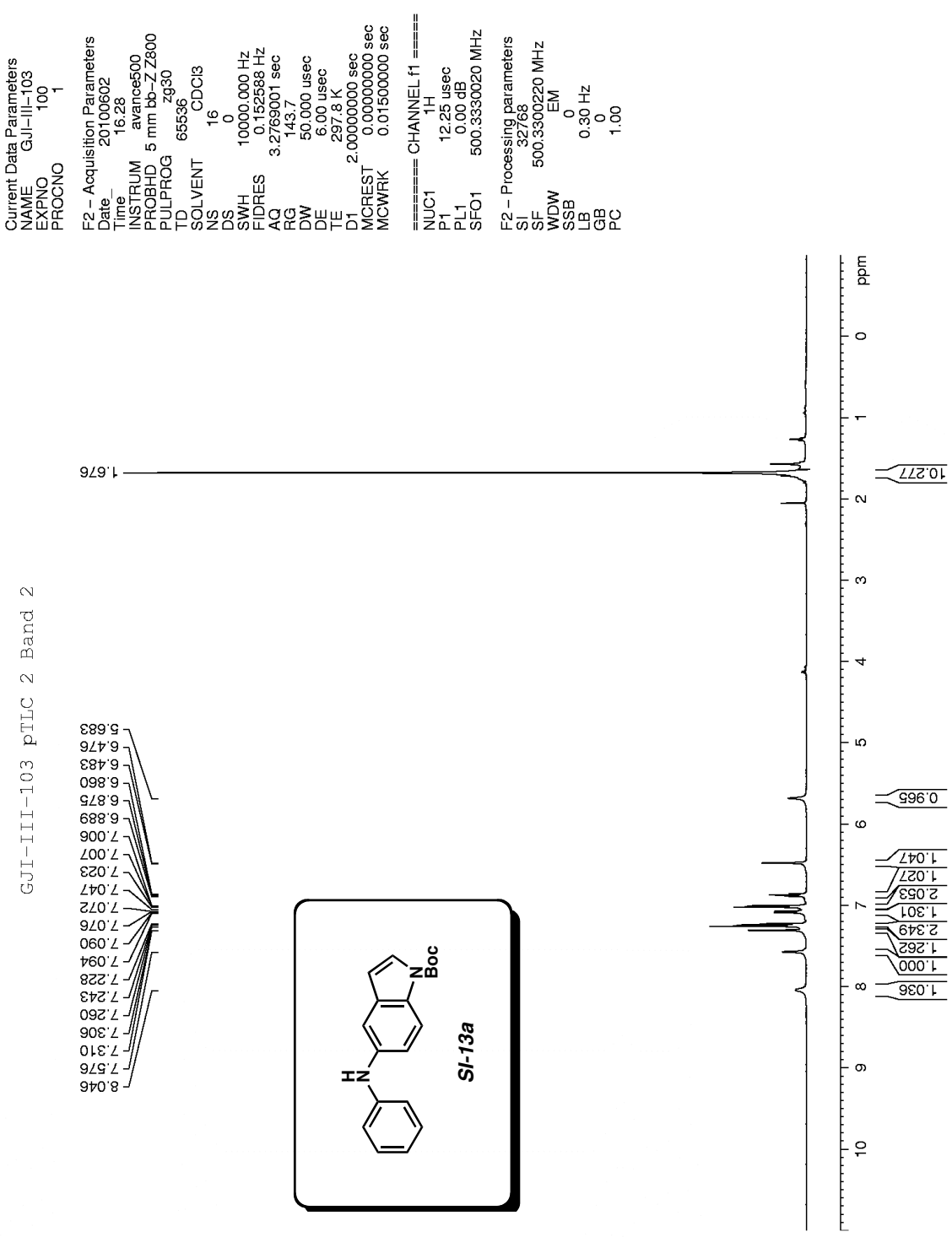

Current Data Parameters
NAME GJI-III-179183
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100507
Time 2.05
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 287.4
DW 50.000 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-III-179+183 Band 1
8.242
7.410
7.394
7.260
7.189
7.184
7.179
7.140
7.124
7.038
7.023
7.007
6.968
6.951
6.740
6.724
6.583
6.578
6.573
2.338
1.551
SI-10d
Me
N
H
O
3.171
1.033
1.018
1.058
2.062
1.030
2.070
1.006
1.000
ppm

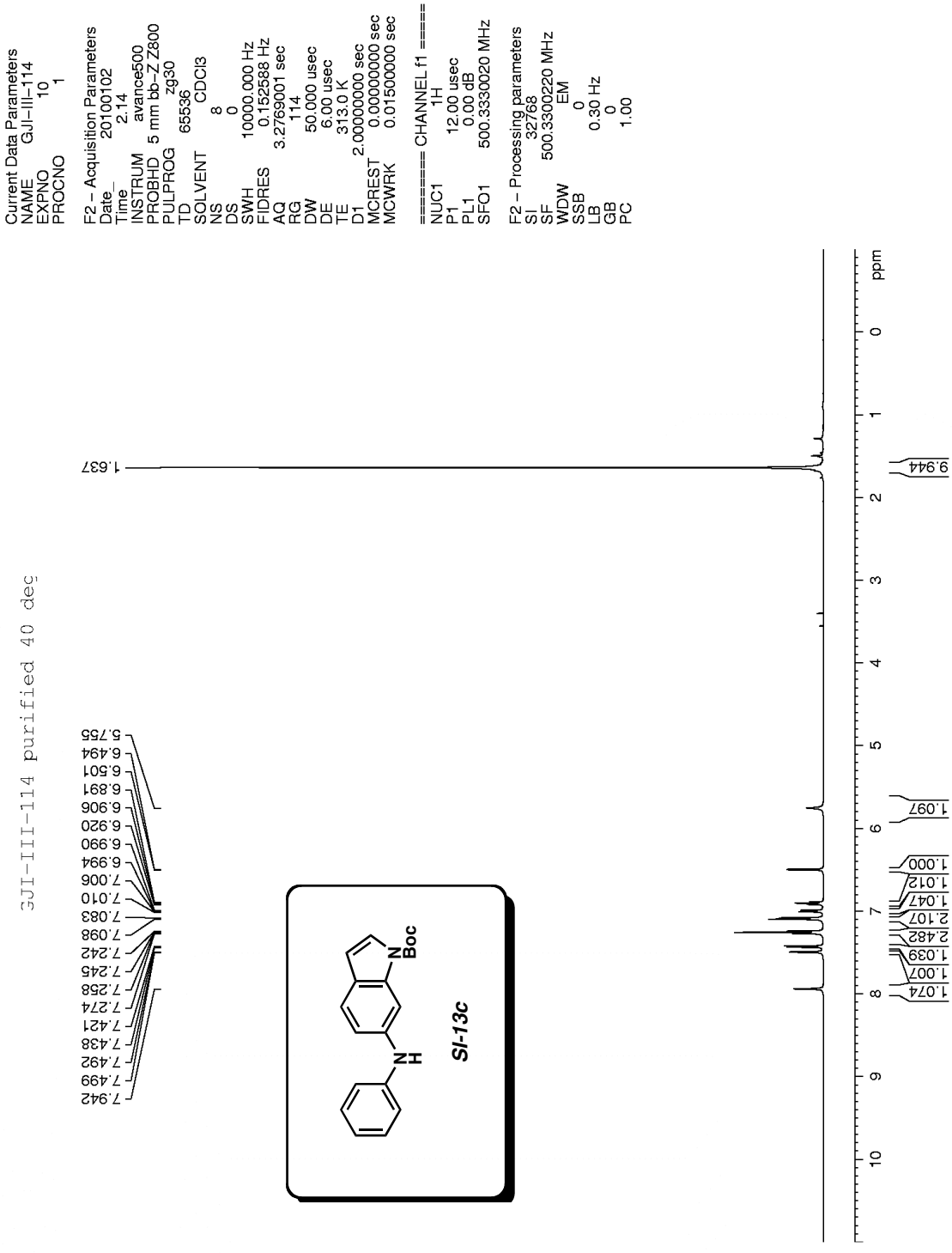

GJI-III-113 sp3 40 deg
Current Data Parameters
NAME GJI-III-113
EXPNO 40
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091230
Time_ 1.10
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 143.7
DW 50.000 usec
DE 6.00 usec
TE 313.0 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.097
8.079
7.598
7.590
7.258
7.155
7.151
7.120
7.103
7.033
7.029
7.015
7.011
6.915
6.910
6.906
6.896
6.893
6.483
6.476
2.328
1.677
N
Boc
O
Me
SI-11a
ppm
1.013
1.017
3.046
1.047
2.004
1.000
3.080
9.364

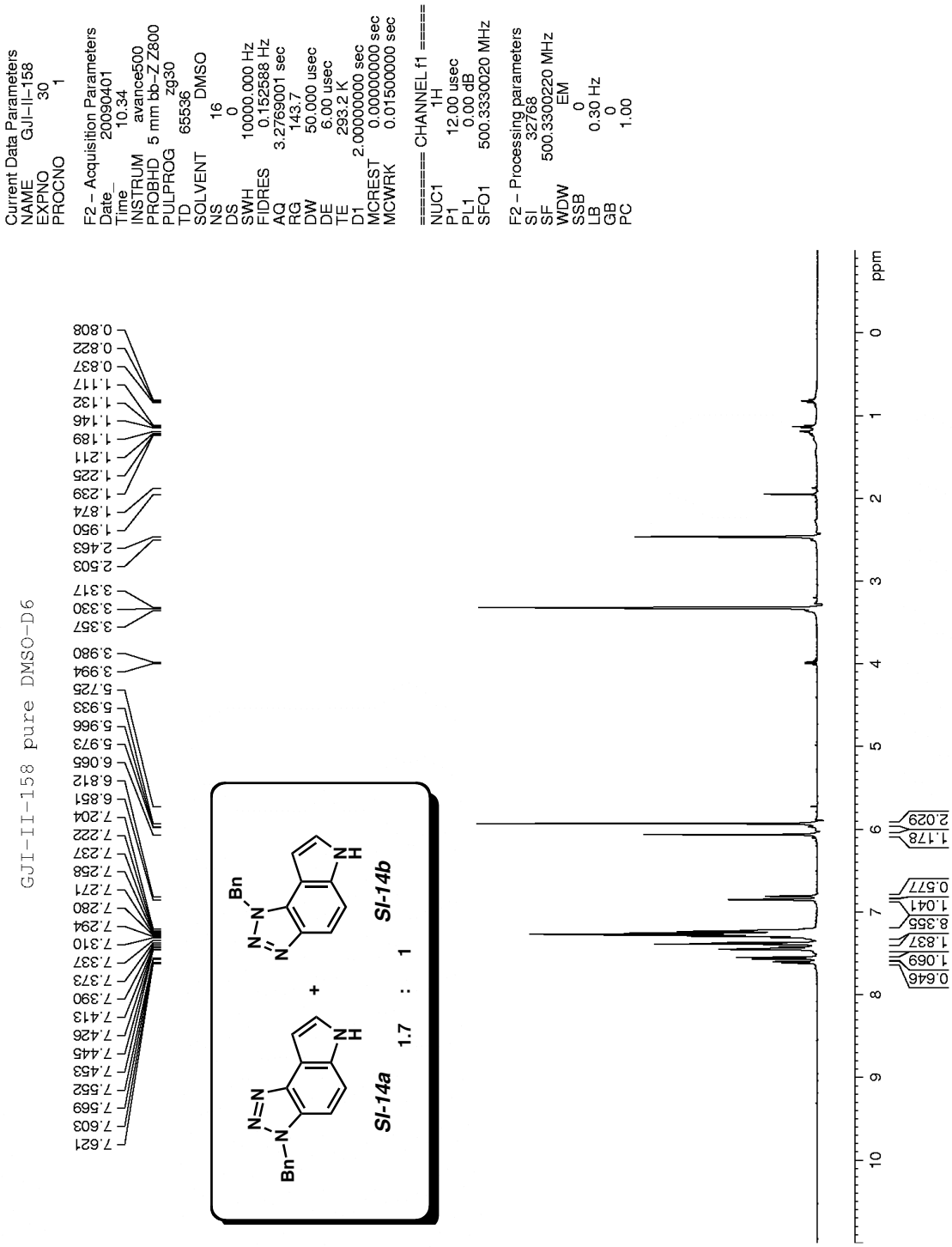

GJI-III-76 purified
Current Data Parameters
NAME GJI-III-76
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091023
Time 17.28
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 143.7
DW 50.000 usec
DE 6.00 usec
TE 295.5 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
6.529
6.530
6.536
6.537
6.931
6.948
6.950
6.955
6.968
6.972
7.124
7.140
7.260
7.464
7.481
7.548
7.554
7.727
2.329
1.575
Me
O
N
Boc
SI-11c
0.515
1.565
1.038
0.534
0.518
0.407
1.613
5.205
ppm

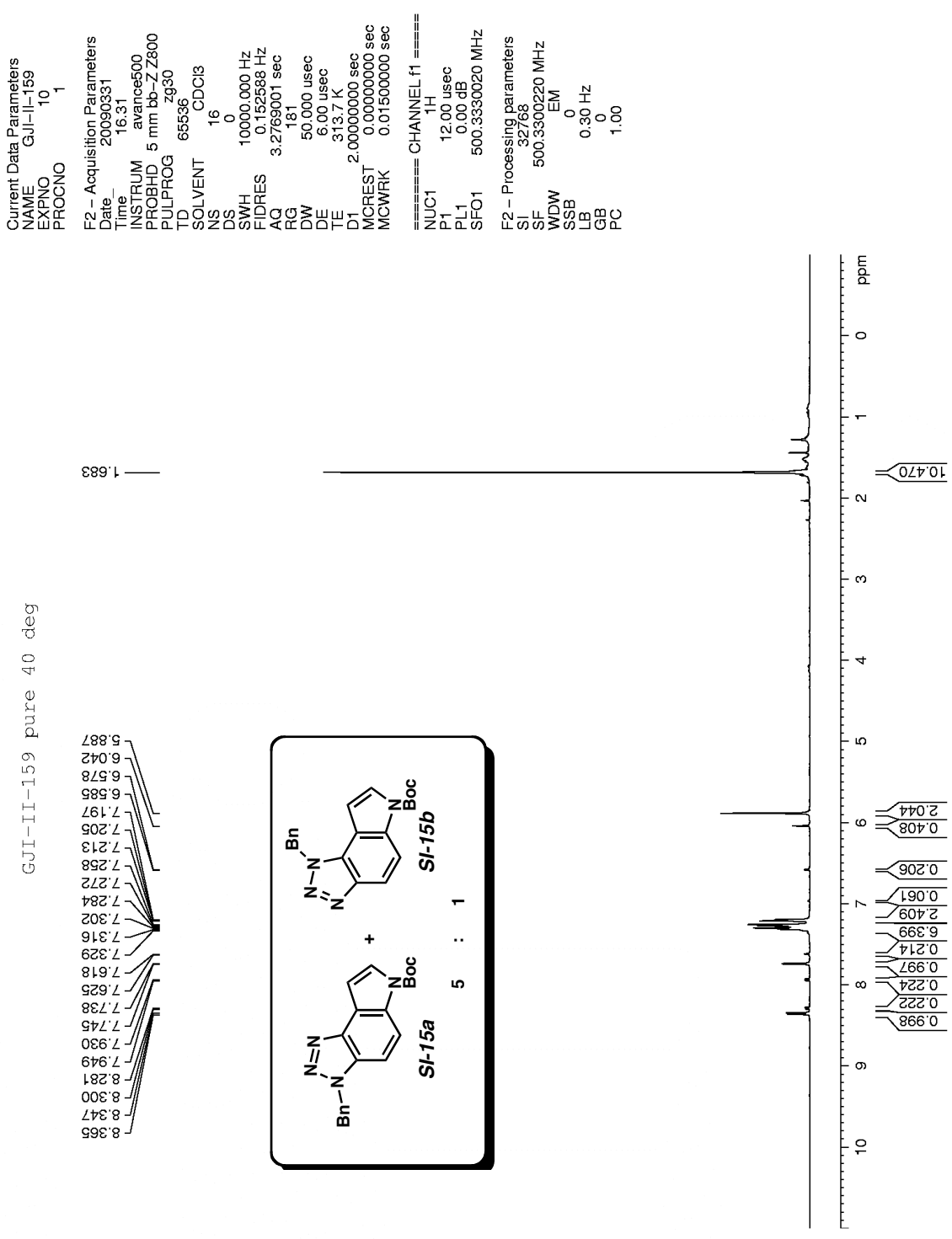

Current Data Parameters
NAME GJI-III-102
EXPNO 40
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091221
Time 3.23
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 203.2
DW 50.000 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-III-102 pTLC Band 3 purif
8.092
7.438
7.435
7.348
7.331
7.260
7.230
7.226
7.213
7.206
7.200
7.198
7.053
7.049
7.036
7.032
6.954
6.938
6.937
6.833
6.819
6.804
6.495
6.491
6.487
5.640
1.269
SI-12a
1.032
1.000
1.082
2.079
1.052
3.029
1.095
1.033
1.005
ppm

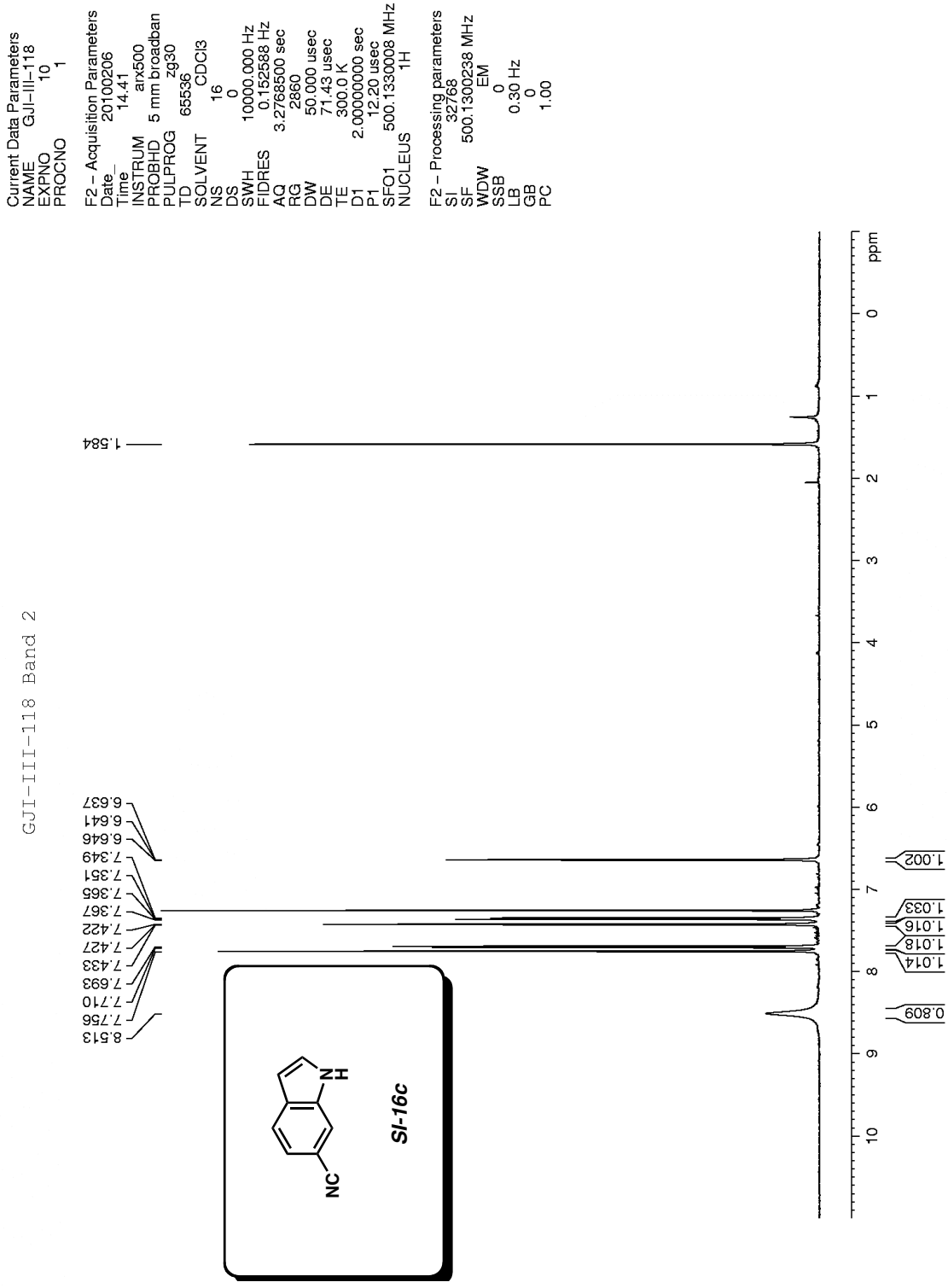

Current Data Parameters
NAME GJI-III-117
EXPNO 30
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100205
Time 14.26
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 1024
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 12.20 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300237 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-III-117 Band 3
7.982
7.558
7.540
7.243
7.228
7.224
7.178
7.176
7.128
7.122
7.117
7.041
7.026
7.025
6.923
6.919
6.906
6.902
6.886
6.871
6.856
6.513
6.508
6.505
6.503
6.696
1.559
SI-12c
0.945
1.031
2.598
1.030
1.077
2.062
2.060
1.000
0.954
ppm

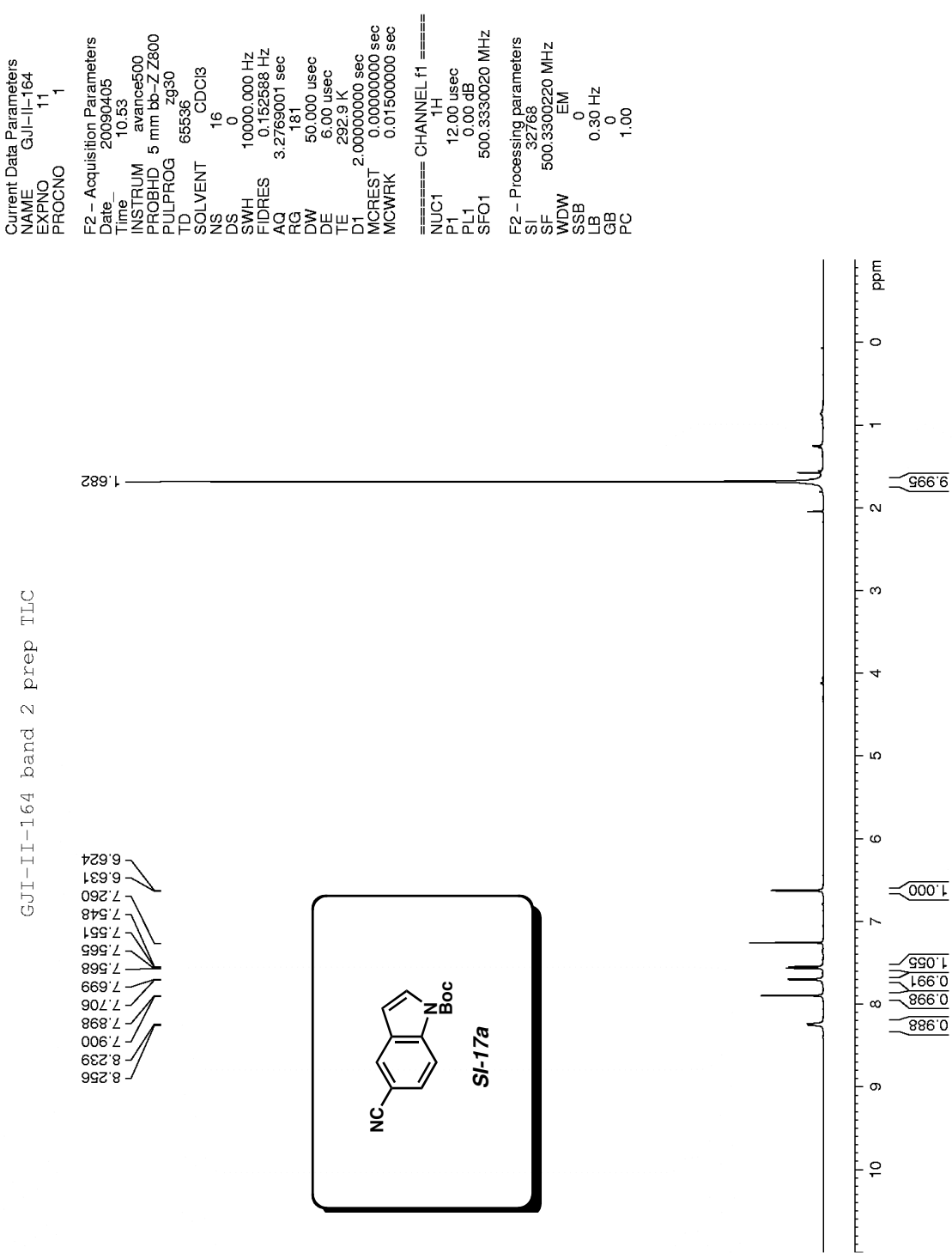

Current Data Parameters
NAME GJI-III-103
EXPNO 100
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100602
Time 16.28
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 143.7
DW 50.000 usec
DE 6.00 usec
TE 297.8 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-III-103 pTLC 2 Band 2
8.046
7.576
7.310
7.306
7.260
7.243
7.228
7.094
7.090
7.076
7.072
7.047
7.023
7.007
7.006
6.889
6.875
6.860
6.483
6.476
5.683
1.676
SI-13a
ppm
10.277
0.965
1.047
1.027
2.053
1.301
2.349
1.262
1.000
1.036

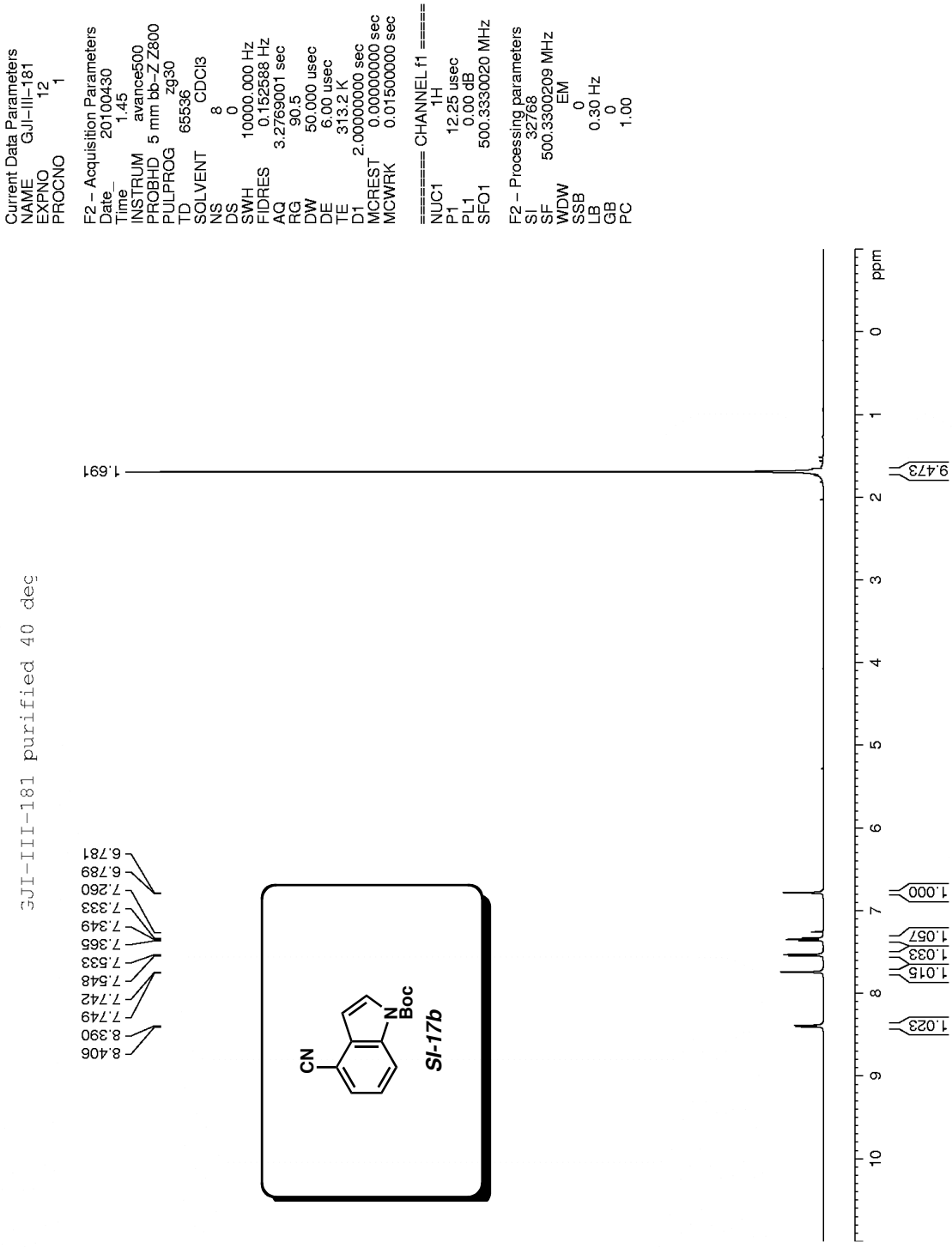

Current Data Parameters
NAME GJI-III-114
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100102
Time_ 2.14
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 114
DW 50.000 usec
DE 6.00 usec
TE 313.0 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-III-114 purified 40 deg
7.942
7.499
7.492
7.438
7.421
7.274
7.258
7.245
7.242
7.098
7.083
7.010
7.006
6.994
6.990
6.920
6.906
6.891
6.501
6.494
5.755
1.637
Boc
N
H
SI-13c
9.944
1.097
1.000
1.012
1.047
2.107
2.482
1.039
1.007
1.074
ppm

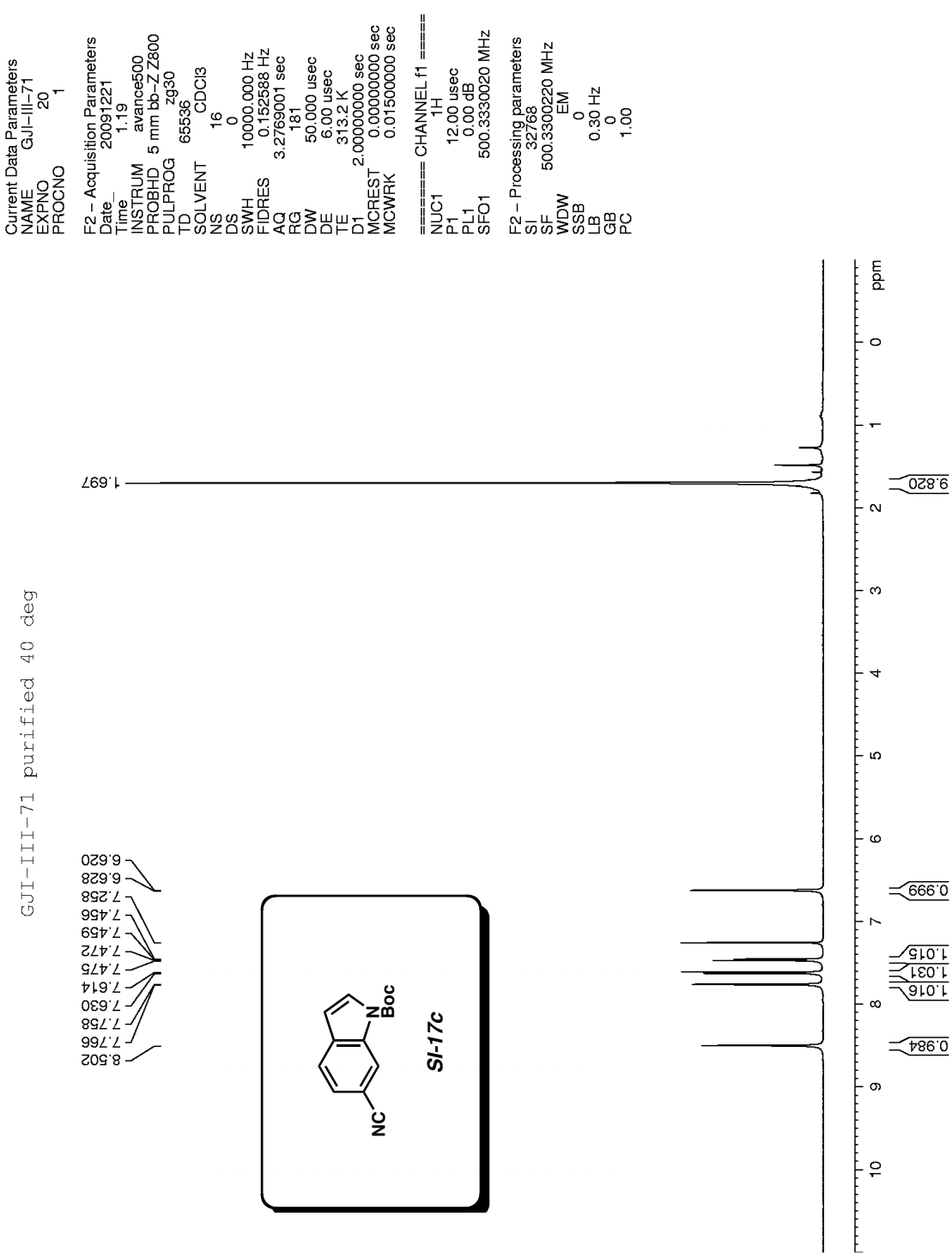

GJI-II-158 pure DMSO-D6
Current Data Parameters
NAME GJI-II-158
EXPNO 30
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090401
Time 10.34
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 143.7
DW 50.000 usec
DE 6.00 usec
TE 293.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Bn
Bn
SI-14a
SI-14b
1.7 : 1
ppm

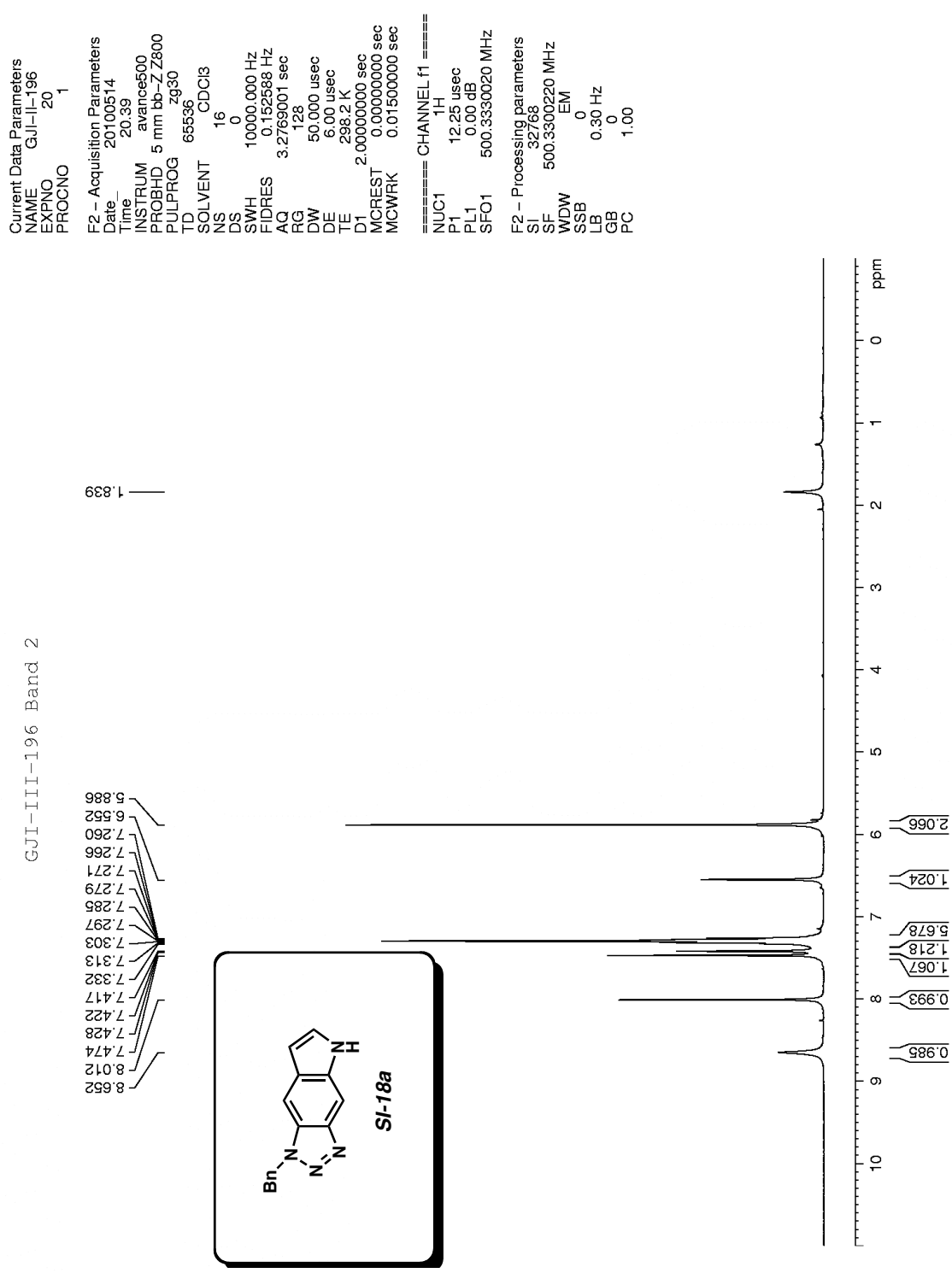

GJI-II-159 pure 40 deg
Current Data Parameters
NAME GJI-II-159
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090331
Time_ 16.31
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 313.7 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.365
8.347
8.300
8.281
7.949
7.930
7.745
7.738
7.625
7.618
7.329
7.316
7.302
7.284
7.272
7.258
7.213
7.205
7.197
6.585
6.578
6.042
5.887
1.683
Bn
N
N
N
Boc
SI-15b
+
N
N
Bn
N
Boc
SI-15a
5 : 1
10.470
2.044
0.408
0.206
0.061
2.409
6.399
0.214
0.997
0.224
0.222
0.998
ppm

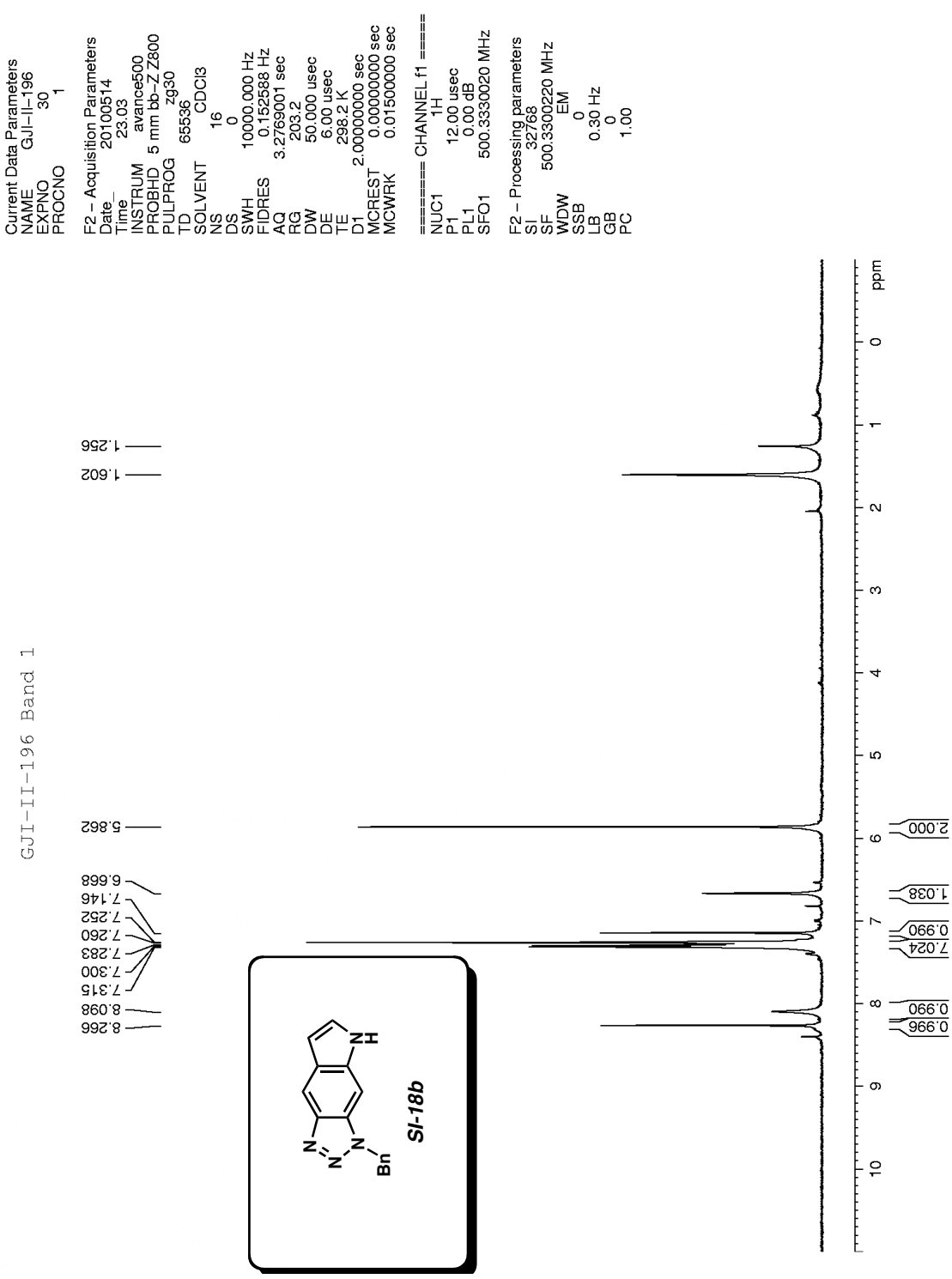

Current Data Parameters
NAME GJI-III-118
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100206
Time 14.41
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 2860
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 12.20 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300238 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-III-118 Band 2
1.584
6.637
6.641
6.646
7.349
7.351
7.365
7.367
7.422
7.427
7.433
7.693
7.710
7.756
8.513
1.002
1.033
1.016
1.018
1.014
0.809
NC
H
N
SI-16c
ppm

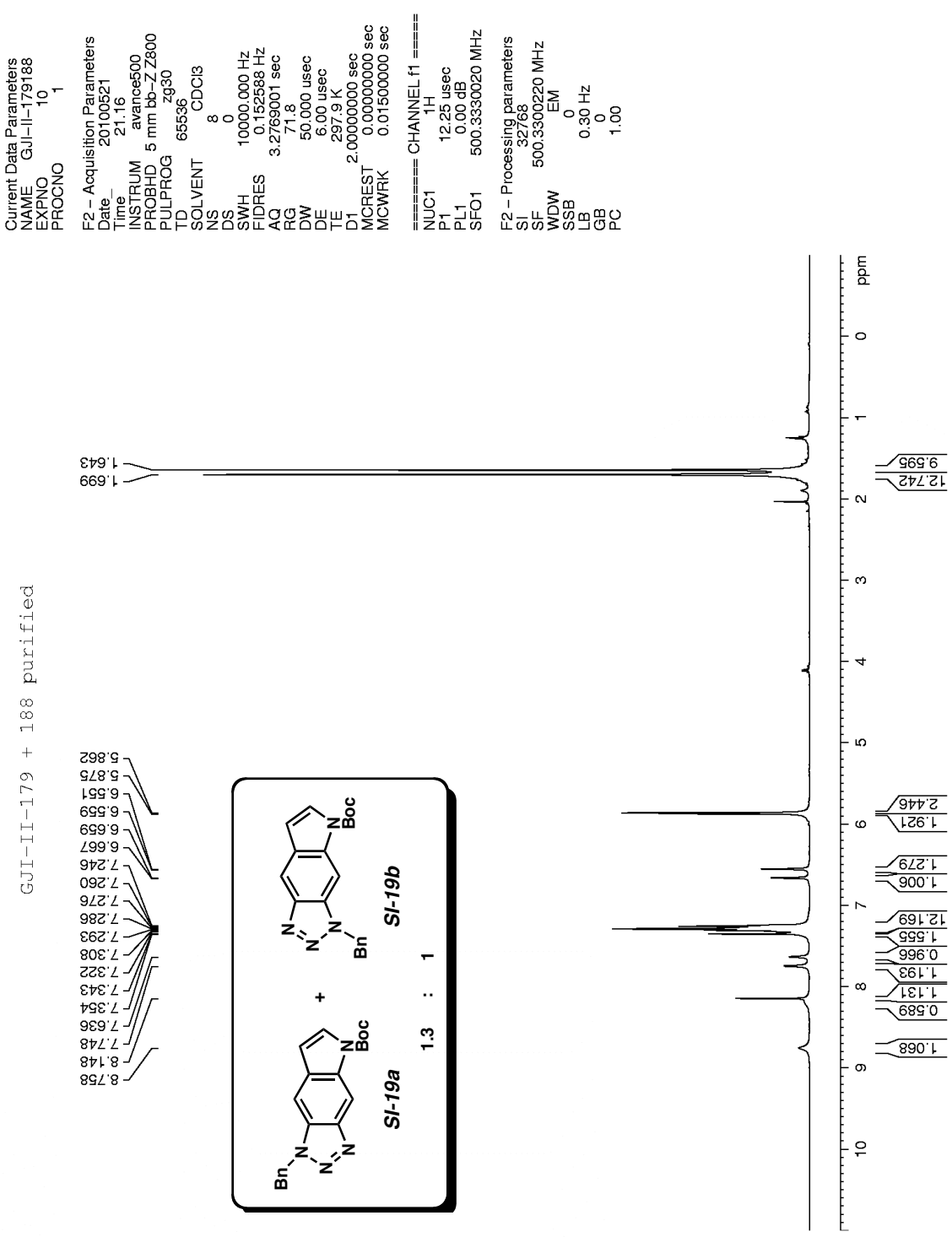

Current Data Parameters
NAME GJI-II-164
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090405
Time_ 10.53
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 292.9 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-II-164 band 2 prep TLC
8.256
8.239
7.900
7.898
7.706
7.699
7.568
7.565
7.551
7.548
7.260
6.631
6.624
1.682
NC
N
Boc
SI-17a
0.988
0.998
0.991
1.055
1.000
9.995
ppm

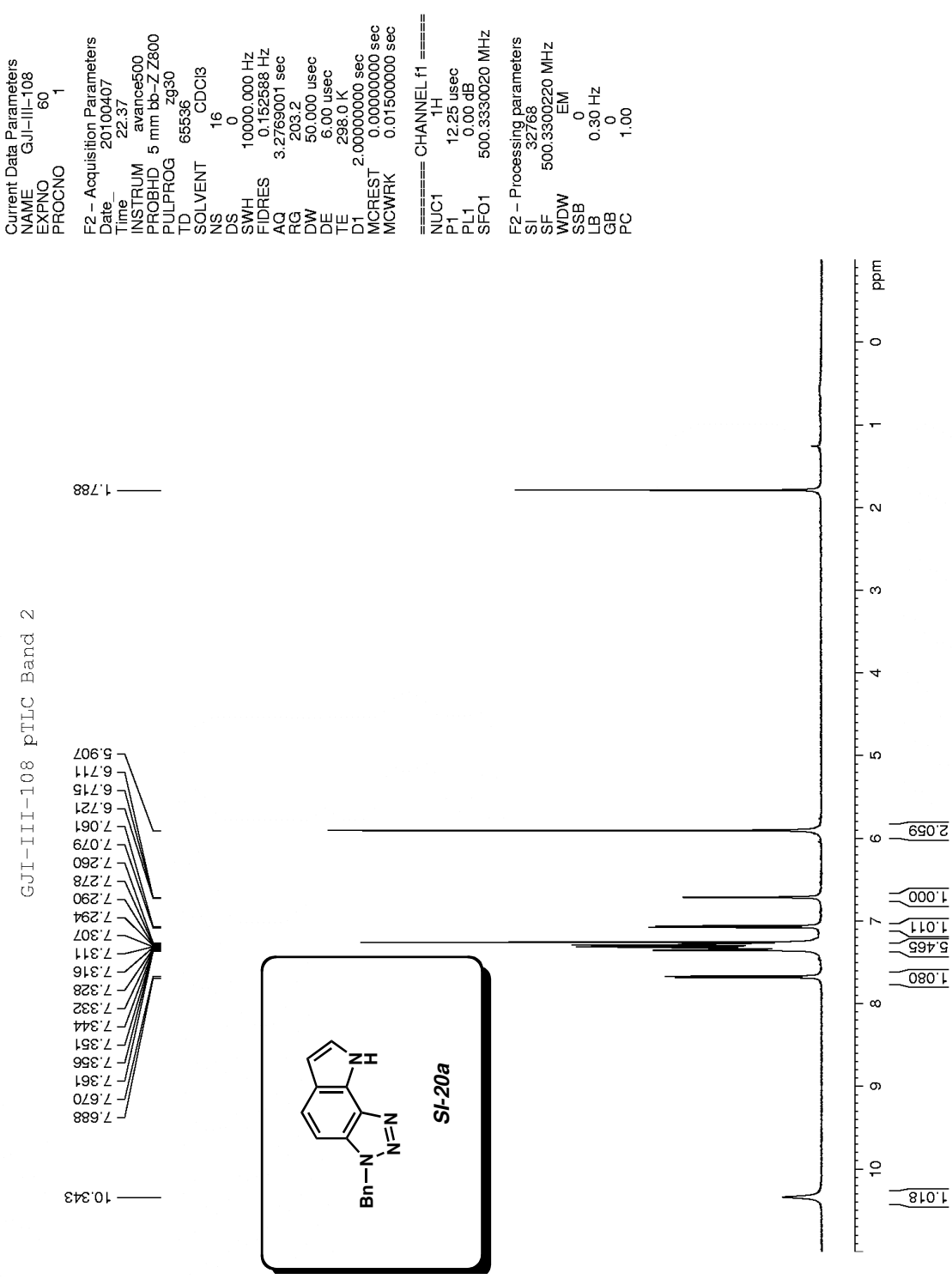

Current Data Parameters
NAME GJI-III-181
EXPNO 12
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100430
Time 1.45
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 90.5
DW 50.000 usec
DE 6.00 usec
TE 313.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300209 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-III-181 purified 40 deg
1.691
6.781
6.789
7.260
7.333
7.349
7.365
7.533
7.548
7.742
7.749
8.390
8.406
CN
N
Boc
SI-17b
9.473
1.000
1.057
1.033
1.015
1.023
ppm

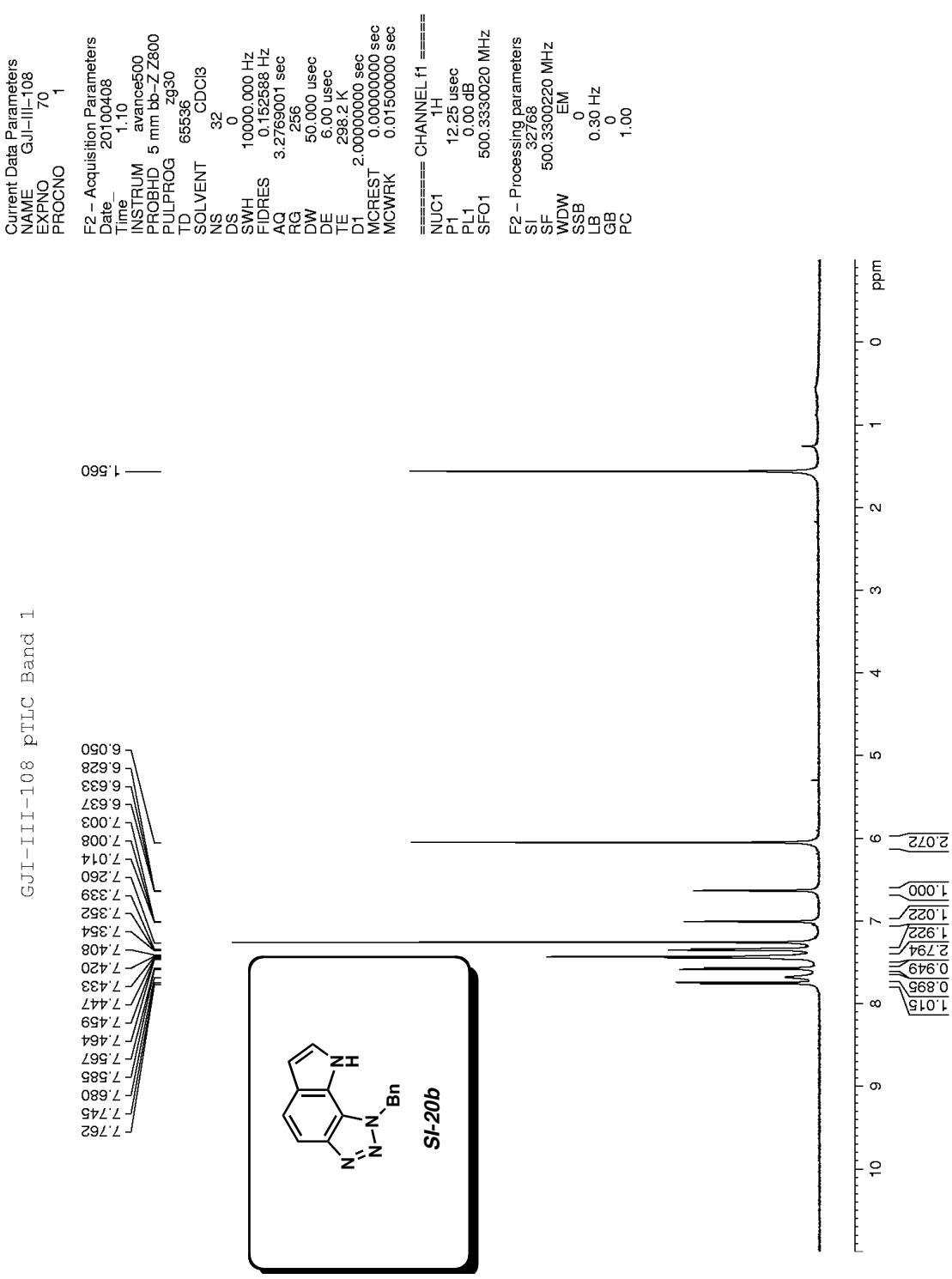

GJI-III-71 purified 40 deg
Current Data Parameters
NAME GJI-III-71
EXPNO 20
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091221
Time_ 1.19
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 313.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.502
7.766
7.758
7.630
7.614
7.475
7.472
7.459
7.456
7.258
6.628
6.620
1.697
9.820
0.999
1.015
1.031
1.016
0.984
N
Boc
NC
SI-17c
ppm

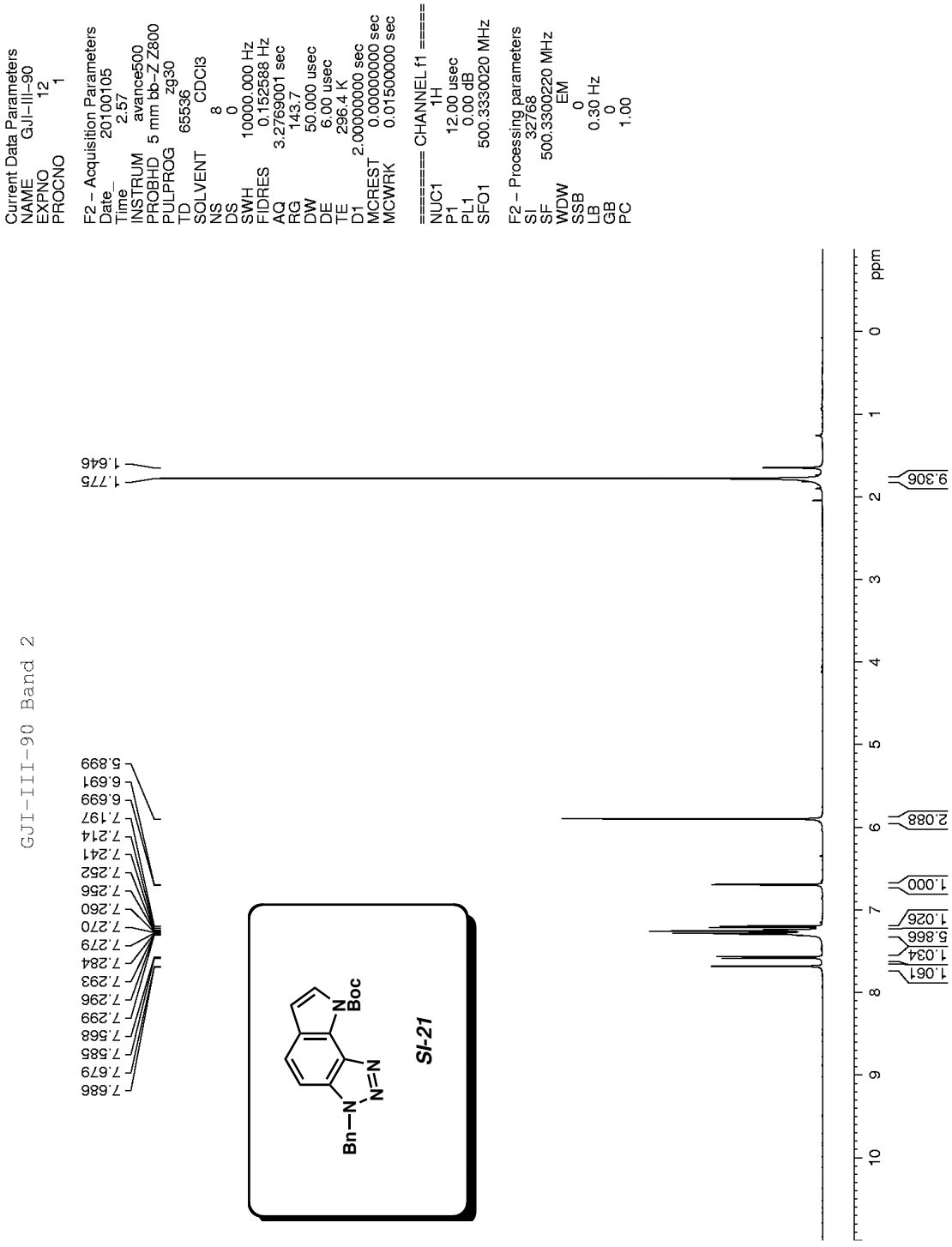

GJI-III-196 Band 2
Current Data Parameters
NAME GJI-II-196
EXPNO 20
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100514
Time 20.39
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 128
DW 50.000 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.839
8.652
8.012
7.474
7.428
7.422
7.417
7.332
7.313
7.303
7.297
7.285
7.279
7.271
7.266
7.260
6.552
5.886
Bn
SI-18a
0.985
0.993
1.067
1.218
5.678
1.024
2.066
ppm


GJI-II-196 Band 1
Current Data Parameters
NAME GJI-II-196
EXPNO 30
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100514
Time 23.03
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 203.2
DW 50.000 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.256
1.602
5.862
6.668
7.146
7.252
7.260
7.283
7.300
7.315
8.098
8.266
2.000
1.038
0.990
7.024
0.990
0.996
ppm
Bn
SI-18b

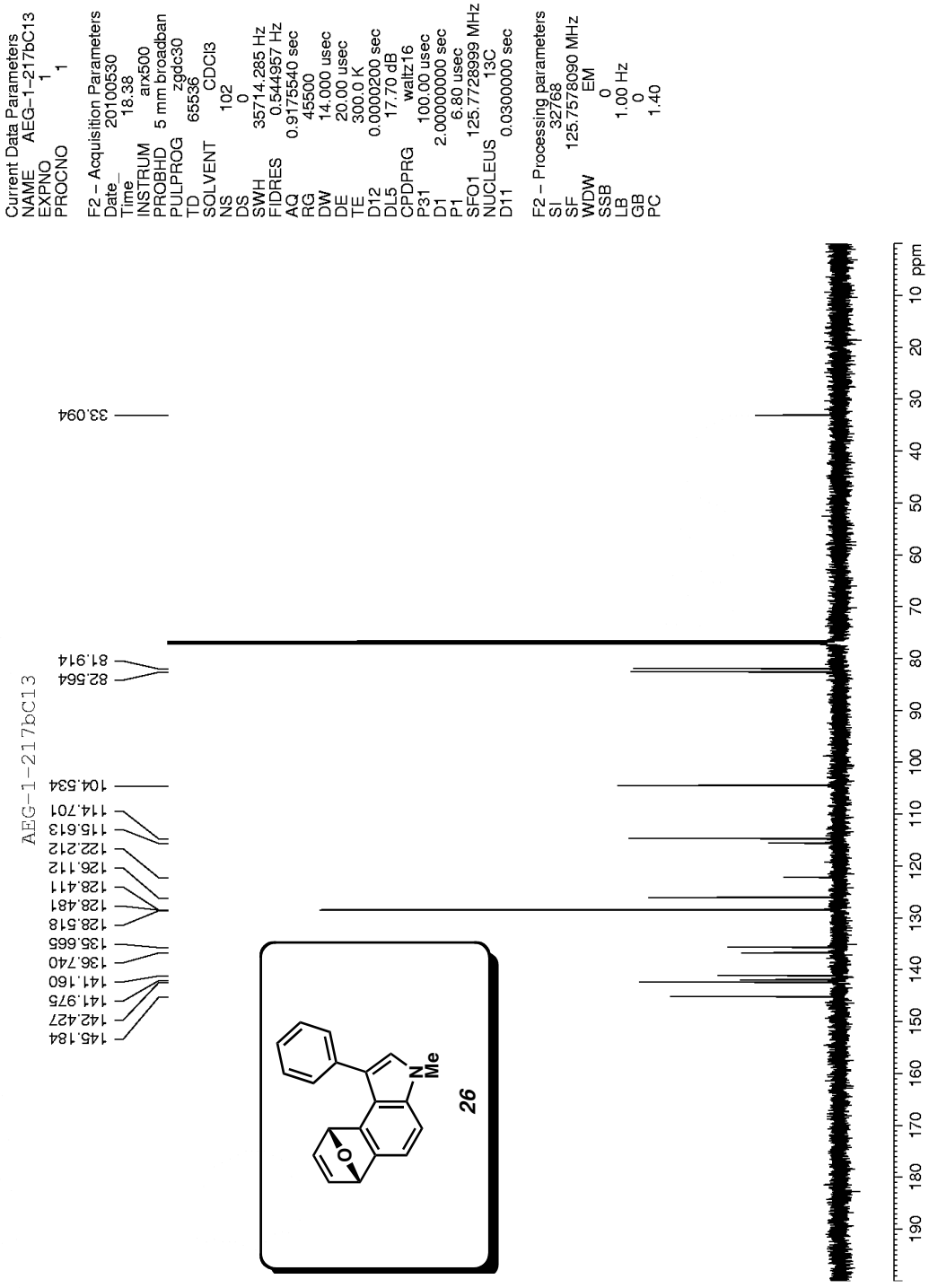

GJI-II-179 + 188 purified
Current Data Parameters
NAME GJI-II-179188
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100521
Time 21.16
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 71.8
DW 50.000 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.758
8.148
7.748
7.636
7.354
7.343
7.322
7.308
7.293
7.286
7.276
7.260
7.246
6.667
6.659
6.559
6.551
5.875
5.862
1.699
1.643
Boc
N
N
N
N
Bn
+
Bn
N
N
N
N
Boc
SI-19a
1.3 : 1
SI-19b
12.742
9.595
2.446
1.921
1.279
1.006
12.169
1.555
0.966
1.193
1.131
0.589
1.068
10
9
8
7
6
5
4
3
2
1
0
ppm

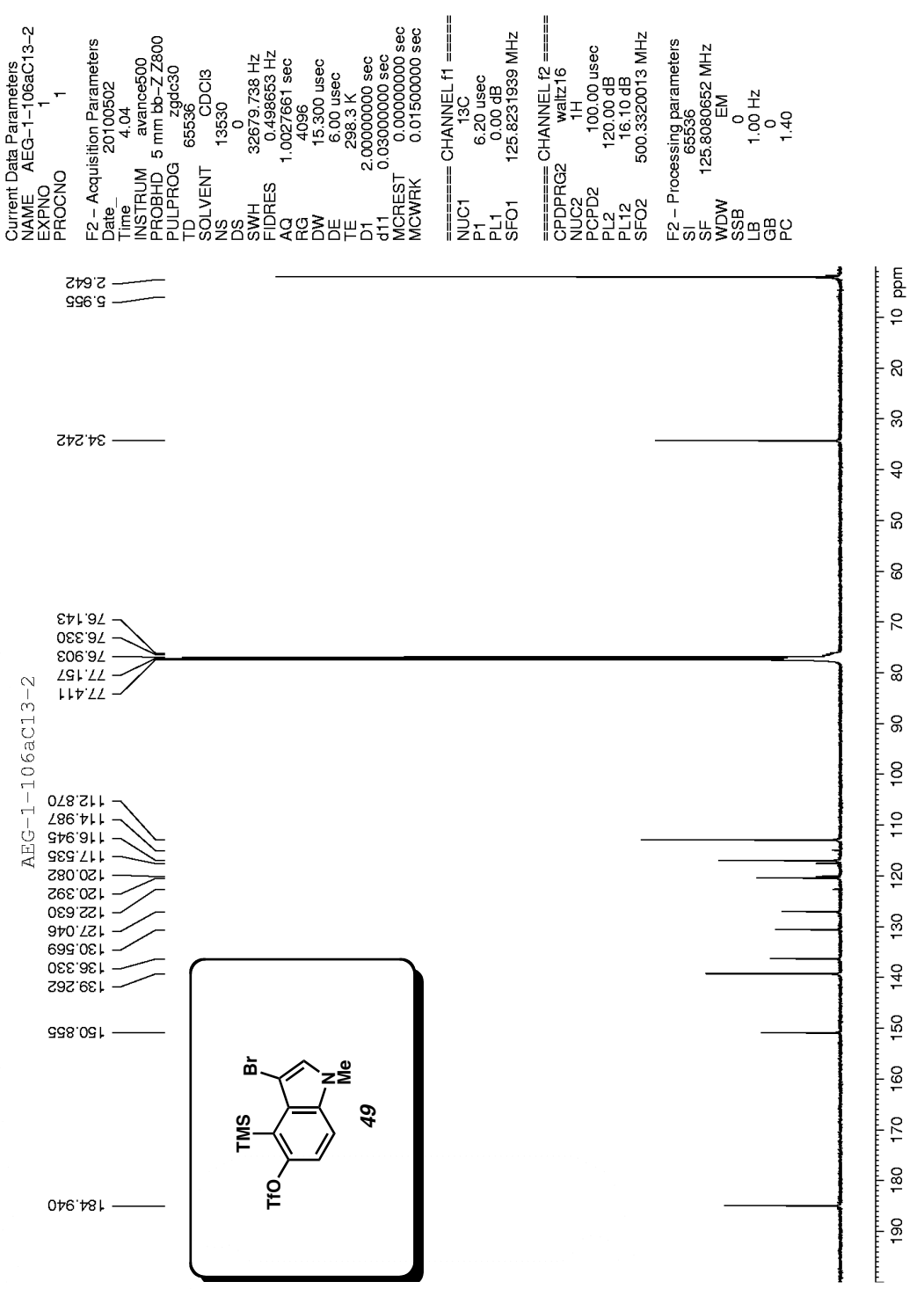

Current Data Parameters
NAME GJI-III-108
EXPNO 60
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100407
Time 22.37
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 203.2
DW 50.000 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-III-108 pTLC Band 2
10.343
7.688
7.670
7.361
7.356
7.351
7.344
7.332
7.328
7.316
7.311
7.307
7.294
7.290
7.278
7.260
7.079
7.061
6.721
6.715
6.711
5.907
1.788
SI-20a
2.059
1.000
1.011
5.465
1.080
1.018
ppm

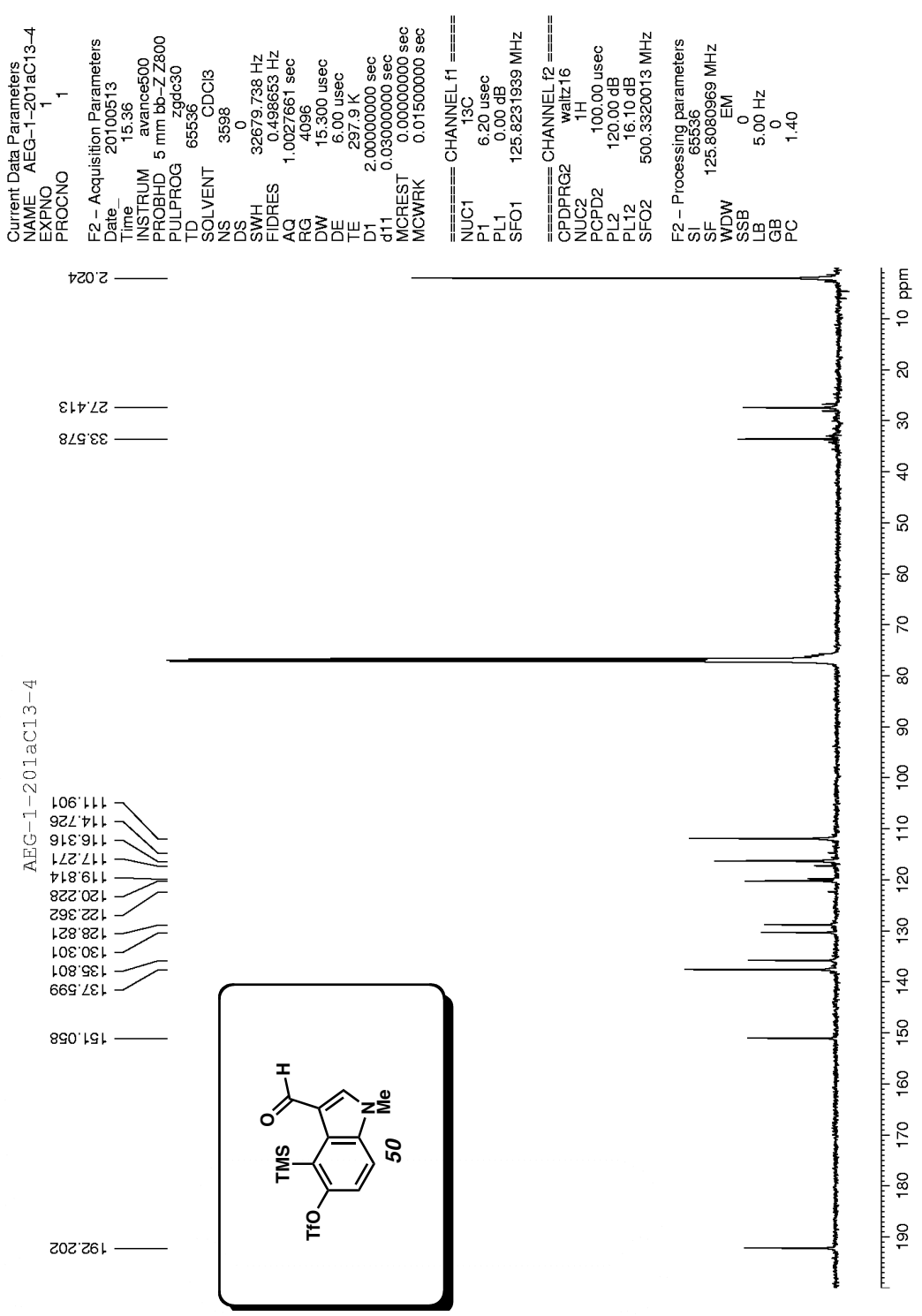

GJI-III-108 pTLC Band 1
Current Data Parameters
NAME GJI-III-108
EXPNO 70
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100408
Time_ 1.10
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 256
DW 50.000 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.560
7.762
7.745
7.680
7.585
7.567
7.464
7.459
7.447
7.433
7.420
7.408
7.354
7.352
7.339
7.260
7.014
7.008
7.003
6.637
6.633
6.628
6.050
Bn
Si-20b
2.072
1.000
1.022
1.922
2.794
0.949
0.895
1.015
ppm

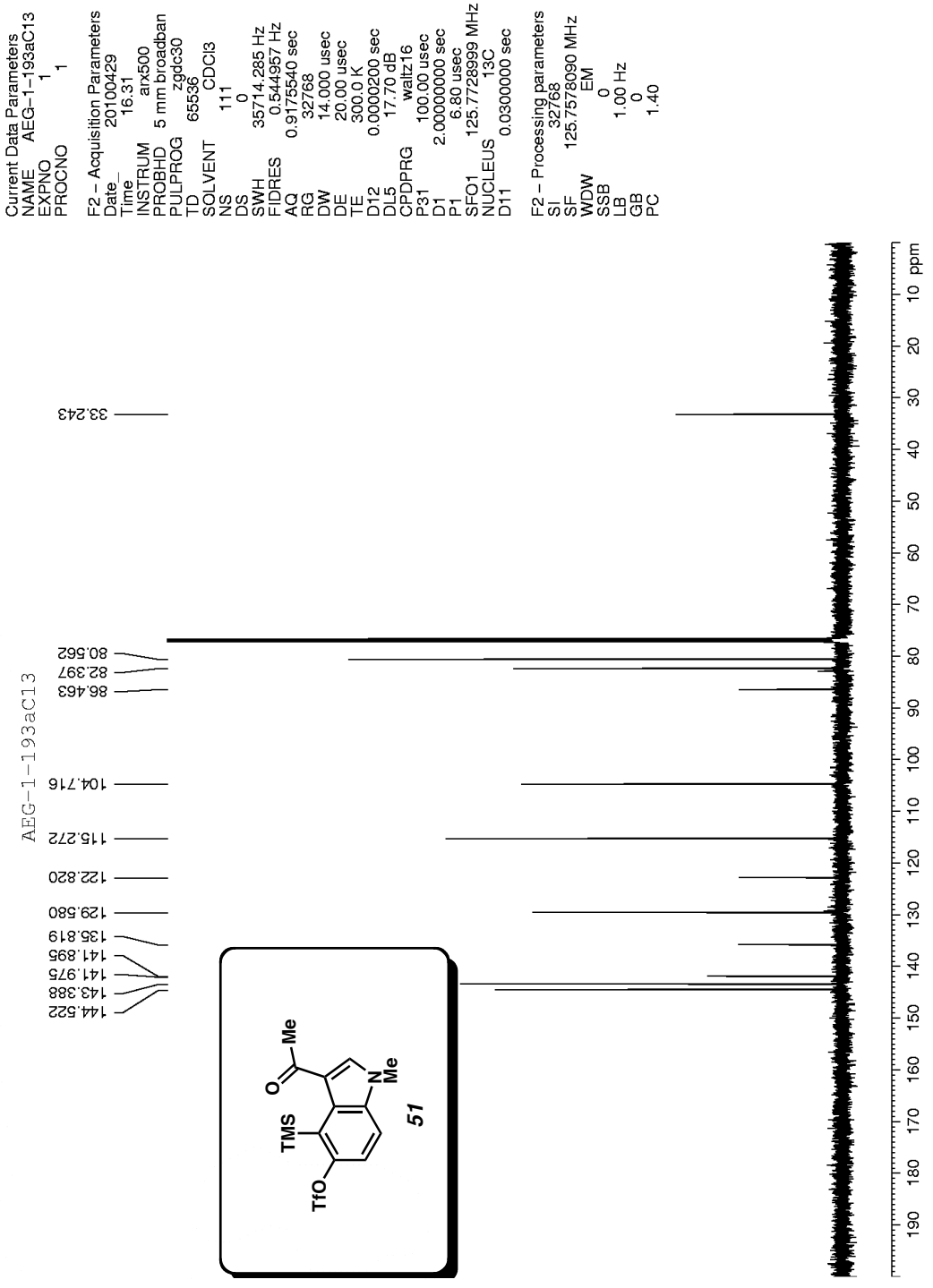

Current Data Parameters
NAME GJI-III-90
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100105
Time 2.57
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 143.7
DW 50.000 usec
DE 6.00 usec
TE 296.4 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-III-90 Band 2
7.686
7.679
7.585
7.568
7.299
7.296
7.293
7.284
7.279
7.270
7.260
7.256
7.252
7.241
7.214
7.197
6.699
6.691
5.899
1.775
1.646
Bn—N
N
N=N
Boc
SI-21
9.306
2.088
1.000
1.026
5.866
1.034
1.061
ppm

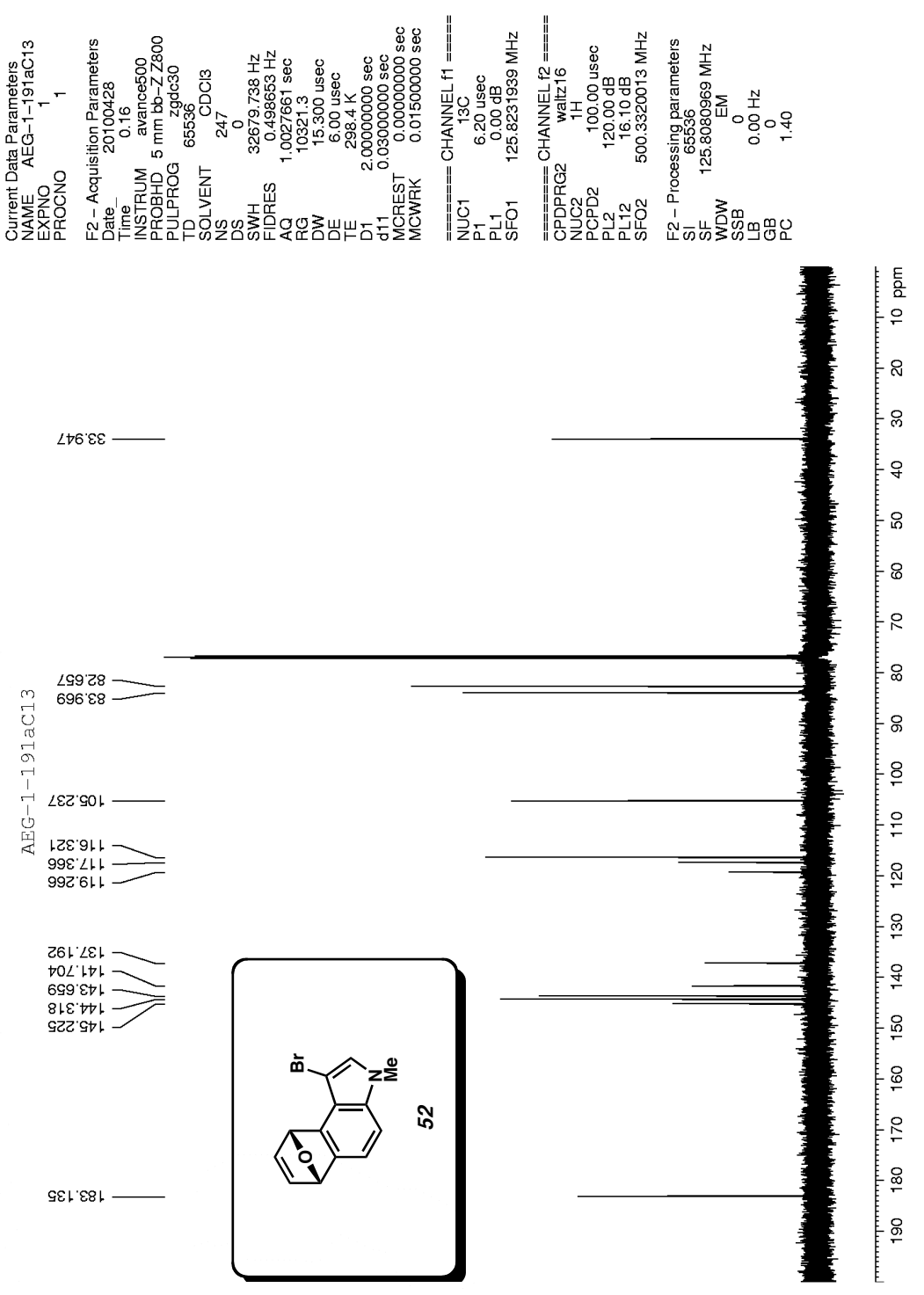
^{13}C NMR Spectra:

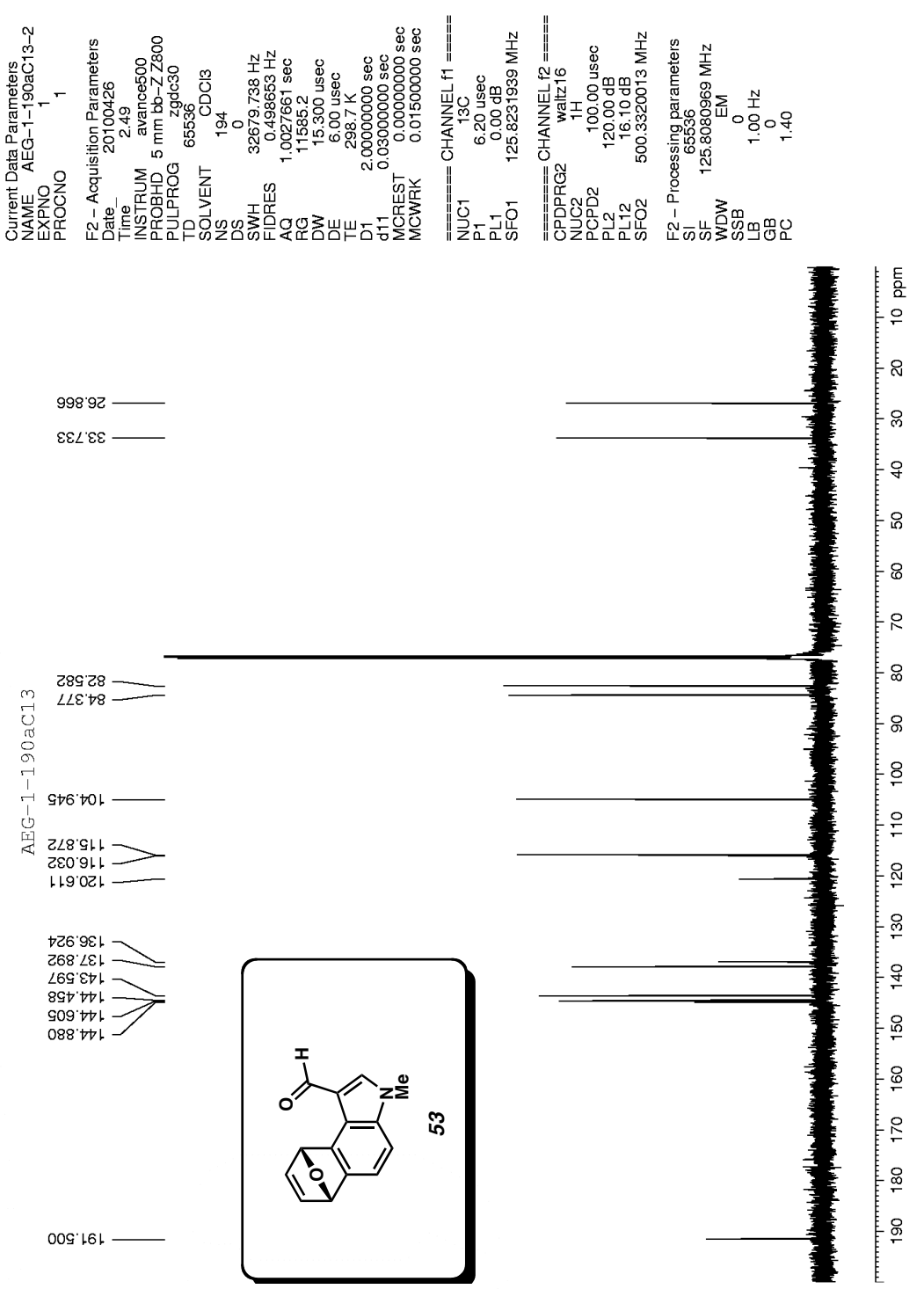

Current Data Parameters
NAME AEG-1-217bC13
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100530
Time_ 18.38
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 102
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 45500
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7578090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
AEG-1-217bC13
33.094
81.914
82.564
104.534
114.701
115.613
122.212
126.112
128.411
128.481
128.518
135.665
136.740
141.160
141.975
142.427
145.184
N
Me
O
26
ppm

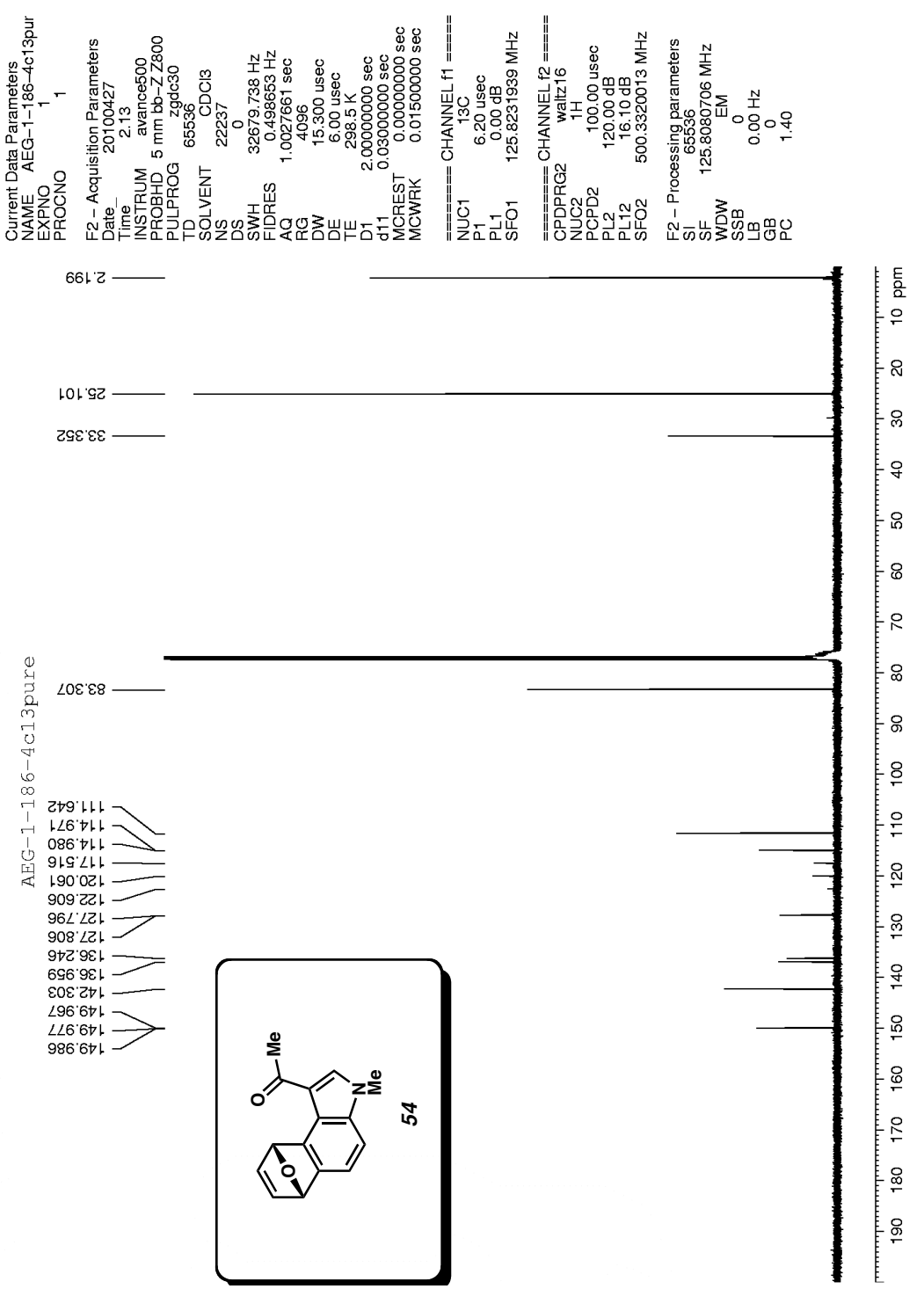

AEG-1-106aC13-2
Current Data Parameters
NAME AEG-1-106aC13-2
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100502
Time 4.04
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 13530
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 298.3 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080652 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
2.642
5.955
34.242
76.143
76.330
76.903
77.157
77.411
112.870
114.987
116.945
117.535
120.082
120.392
122.630
127.046
130.569
136.330
139.262
150.855
184.940
Br
TMS
TfO
N
Me
49
10 ppm

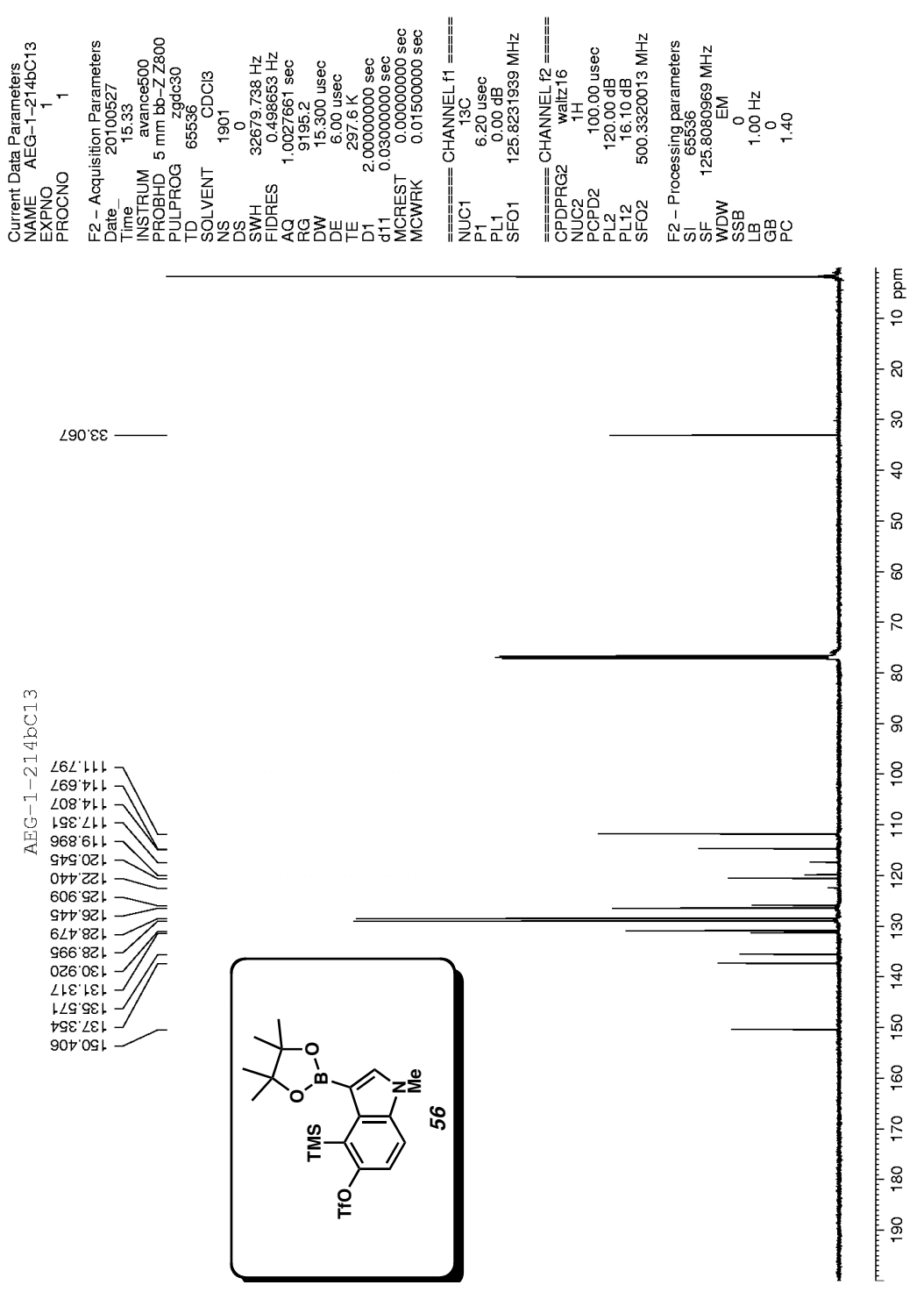

Current Data Parameters
NAME AEG-1-201aC13-4
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date 20100513
Time 15.36
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 3598
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 5.00 Hz
GB 0
PC 1.40
AEG-1-201aC13-4
192.202
151.058
137.599
135.801
130.301
128.821
122.362
120.228
119.814
117.271
116.316
114.726
111.901
33.578
27.413
2.024
TMS
TfO
O
H
N
Me
50
ppm

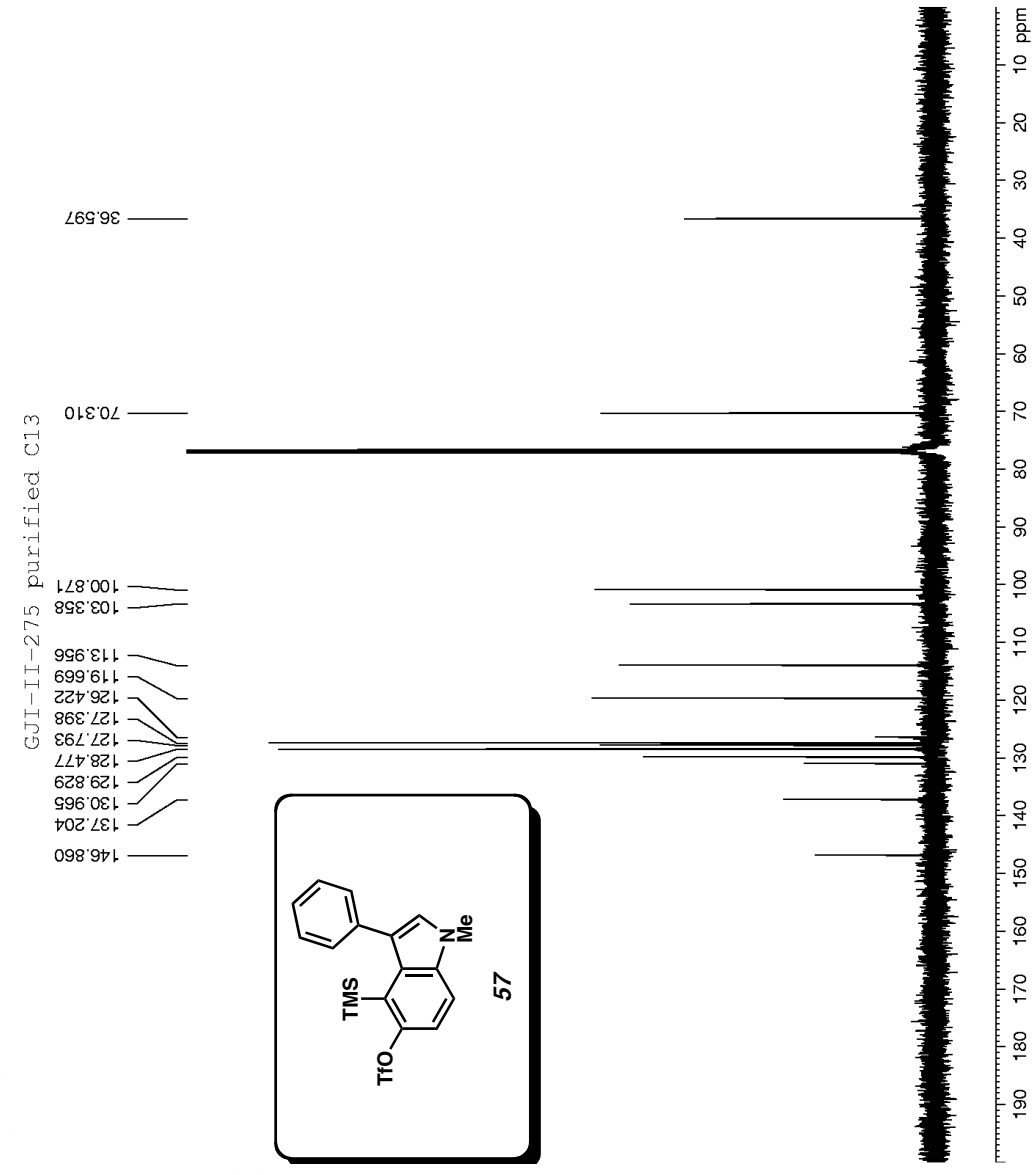

Current Data Parameters
NAME AEG-1-193aC13
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date 20100429
Time 16.31
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 111
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 32768
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7578090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
AEG-1-193aC13
33.243
80.562
82.397
86.463
104.716
115.272
122.820
129.580
135.819
141.895
141.975
143.388
144.522
TMS
TfO
O
Me
N
Me
51
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

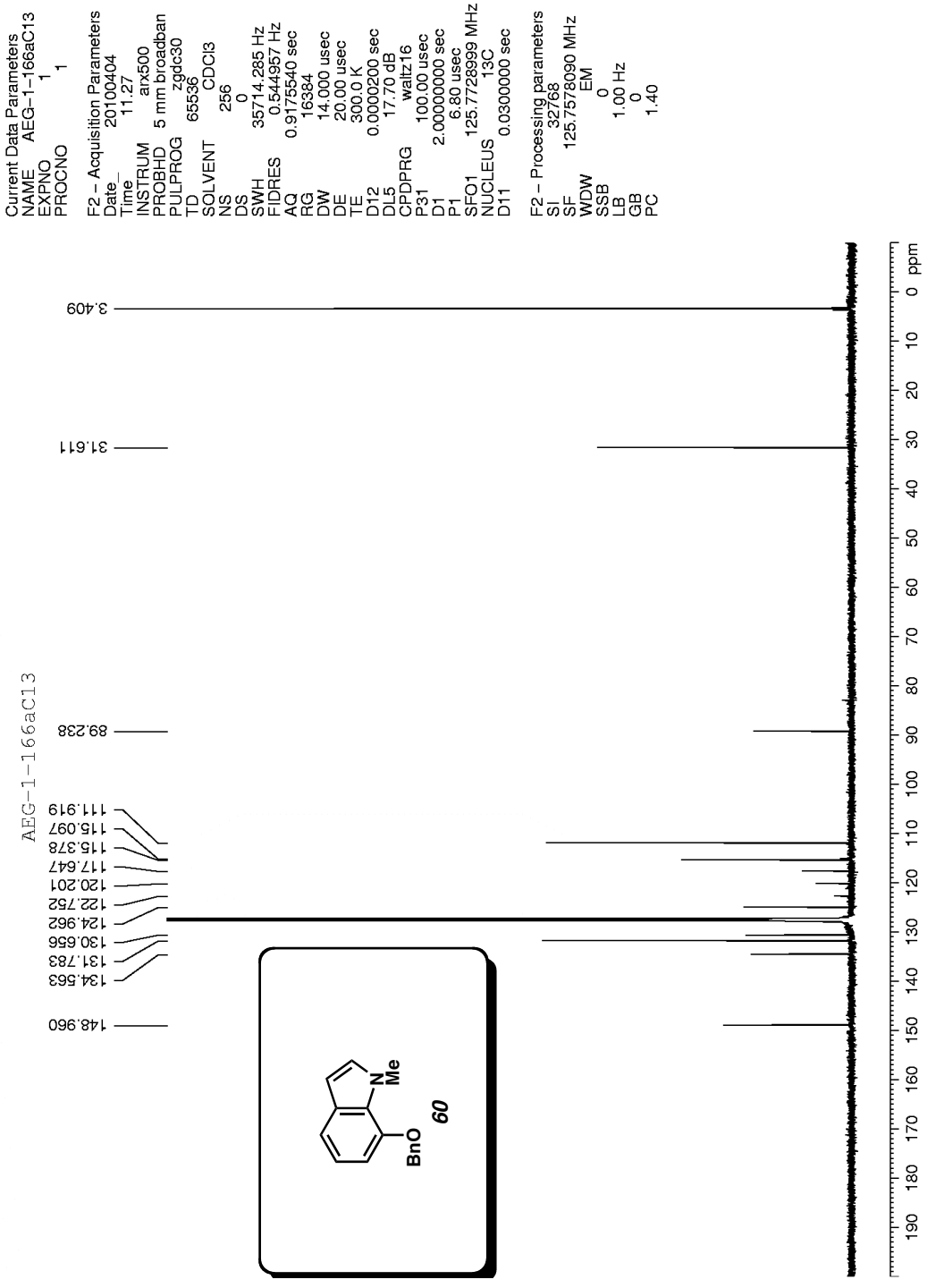

Current Data Parameters
NAME AEG-1-191aC13
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date 20100428
Time 0.16
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 247
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 10321.3
DW 15.300 usec
DE 6.00 usec
TE 298.4 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
AEG-1-191aC13
183.135
145.225
144.318
143.659
141.704
137.192
119.266
117.366
116.321
105.237
83.969
82.657
33.947
Br
N
Me
O
52
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

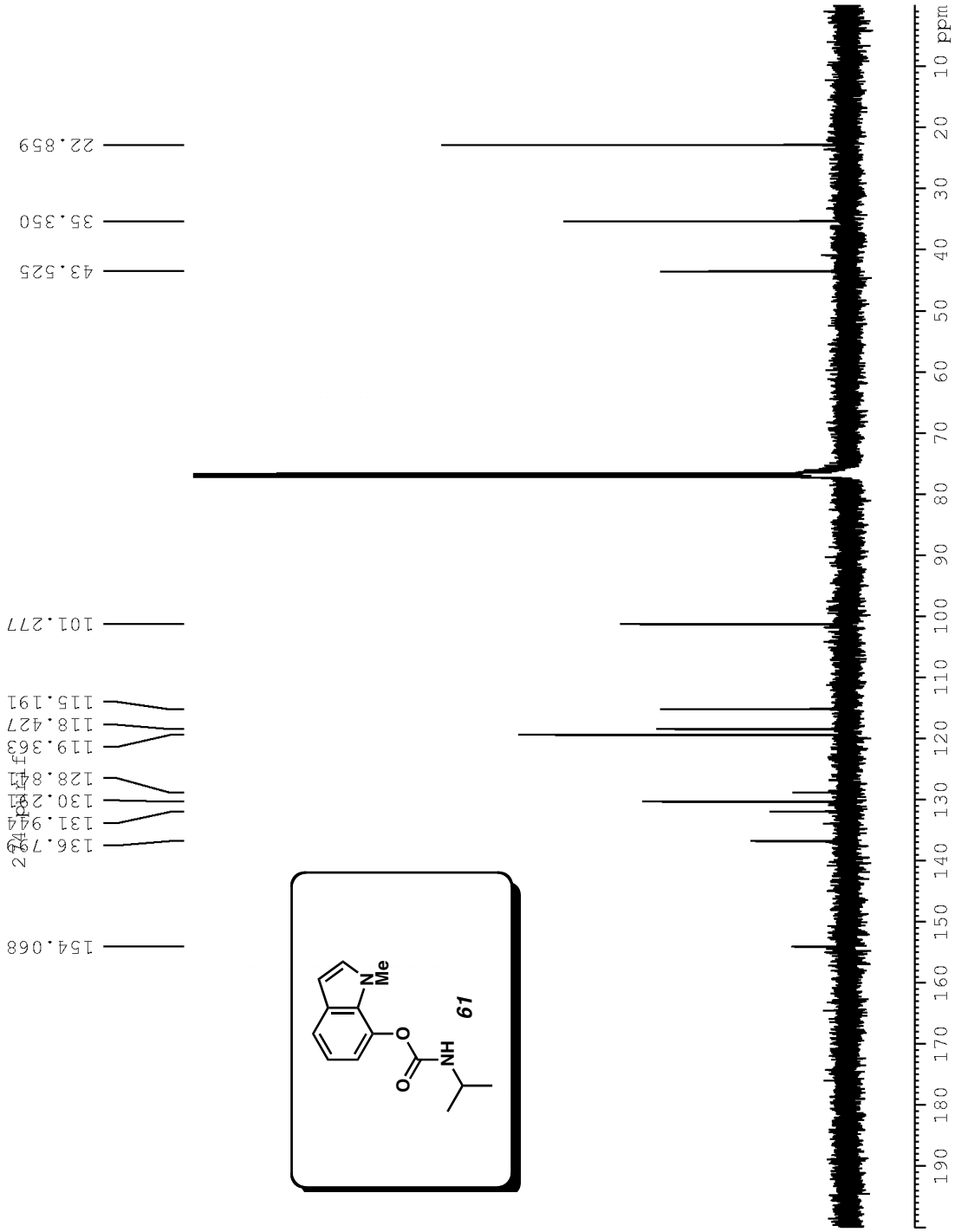

Current Data Parameters
NAME AEG-1-190aC13-2
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100426
Time 2.49
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 194
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 11585.2
DW 15.300 usec
DE 6.00 usec
TE 298.7 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
AEG-1-190aC13
191.500
144.880
144.605
144.458
143.597
137.892
136.924
120.611
116.032
115.872
104.945
84.377
82.582
33.733
26.866
O
H
O
N
Me
53
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

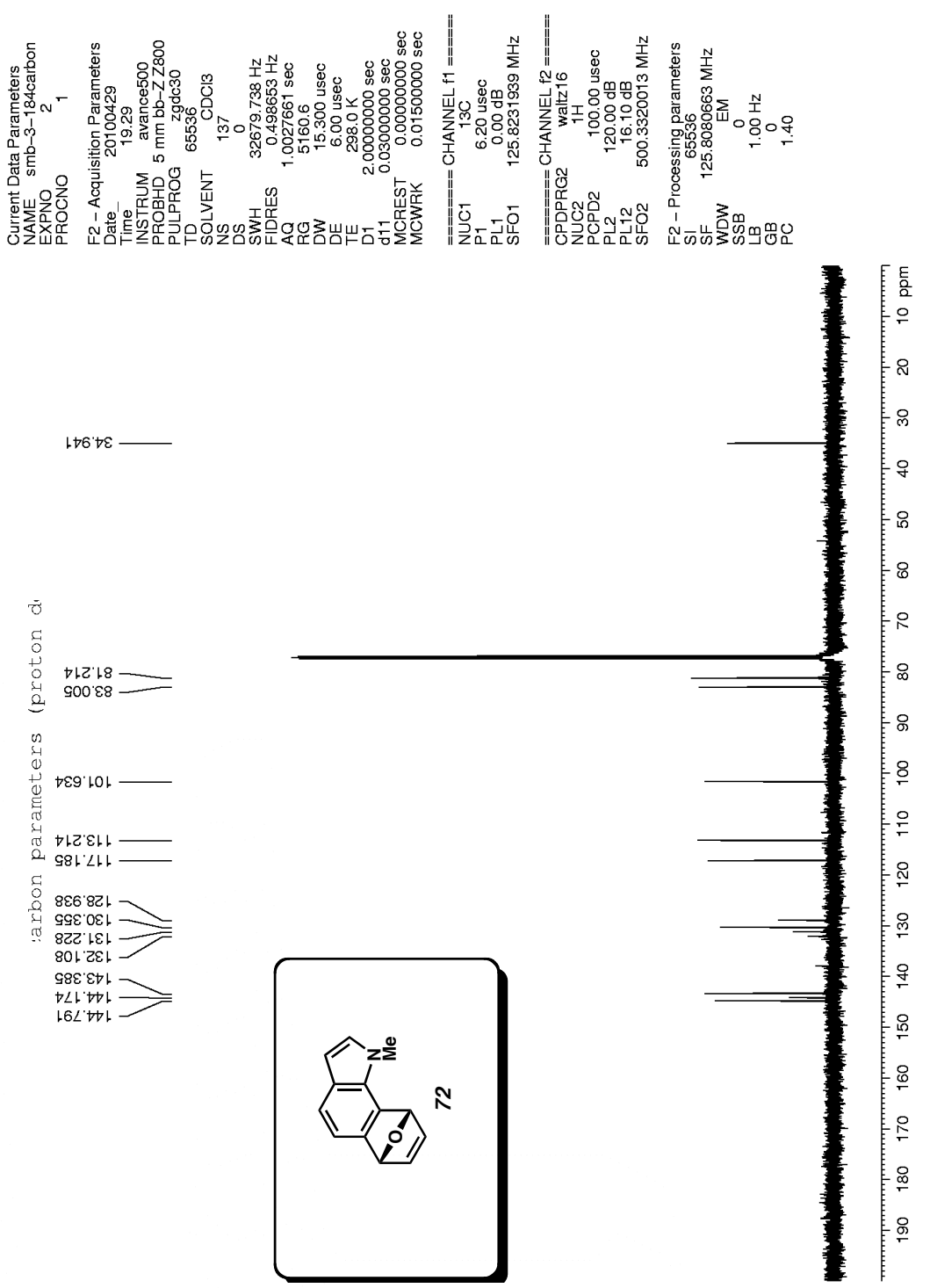

Current Data Parameters
NAME AEG-1-186-4c13pur
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100427
Time 2.13
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 22237
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 298.5 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080706 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
AEG-1-186-4c13pure
2.199
25.101
33.352
83.307
111.642
114.971
114.980
117.516
120.061
122.606
127.796
127.806
136.246
136.959
142.303
149.967
149.977
149.986
Me
O
N
Me
O
54
ppm

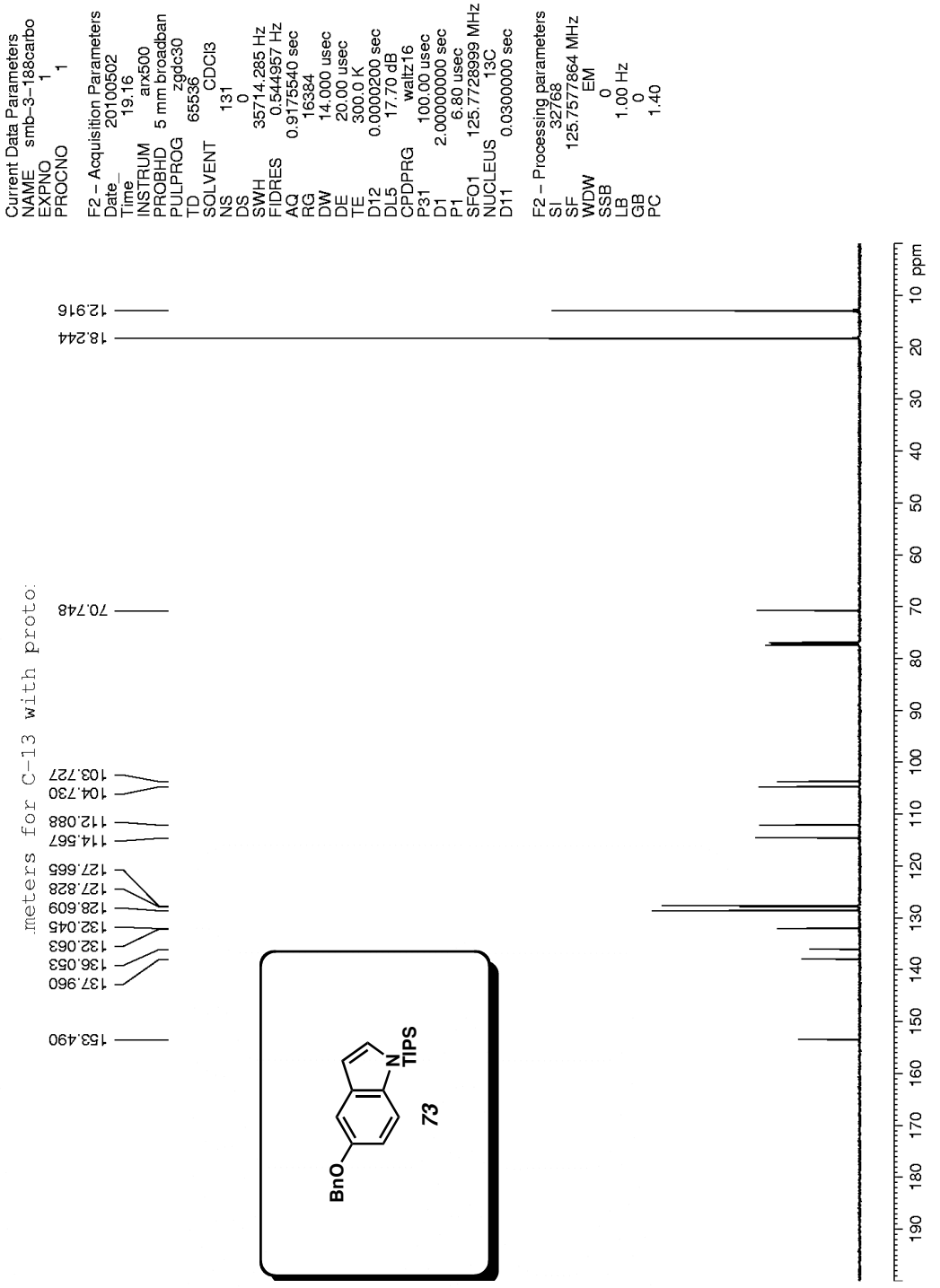

Current Data Parameters
NAME AEG-1-214bC13
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date 20100527
Time 15.33
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 1901
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 9195.2
DW 15.300 usec
DE 6.00 usec
TE 297.6 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
AEG-1-214bC13
33.067
111.797
114.697
114.807
117.351
119.896
120.545
122.440
125.909
126.445
128.479
128.995
130.920
131.317
135.571
137.354
150.406
TMS
TfO
N
Me
56
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

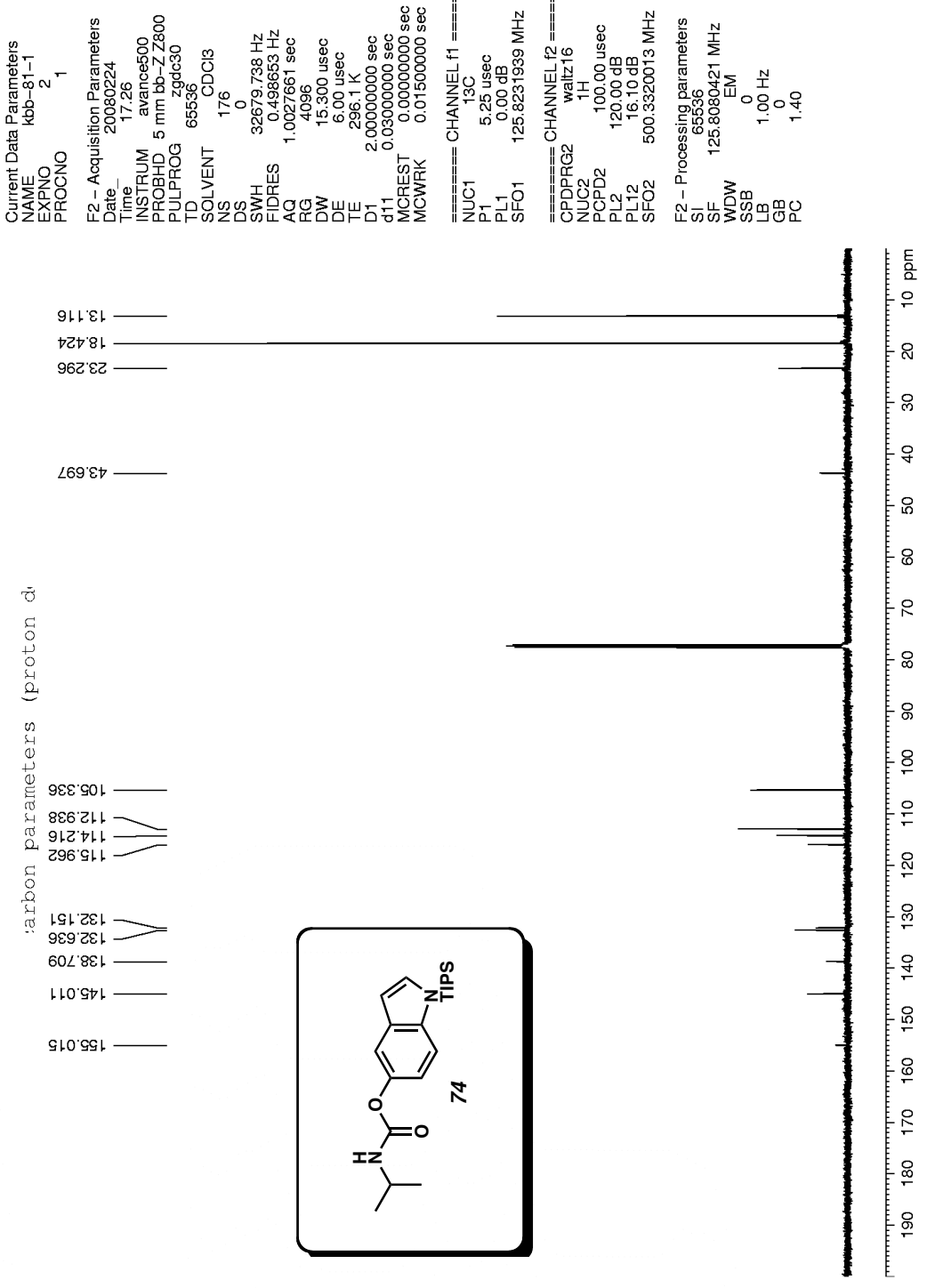

Current Data Parameters
NAME GJI-II-275
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100507
Time 1.44
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 9195.2
DW 15.300 usec
DE 6.00 usec
TE 298.7 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-II-275 purified C13
36.597
70.310
100.871
103.358
113.956
119.669
126.422
127.398
127.793
128.477
129.829
130.965
137.204
146.860
TMS
TfO
N
Me
57
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

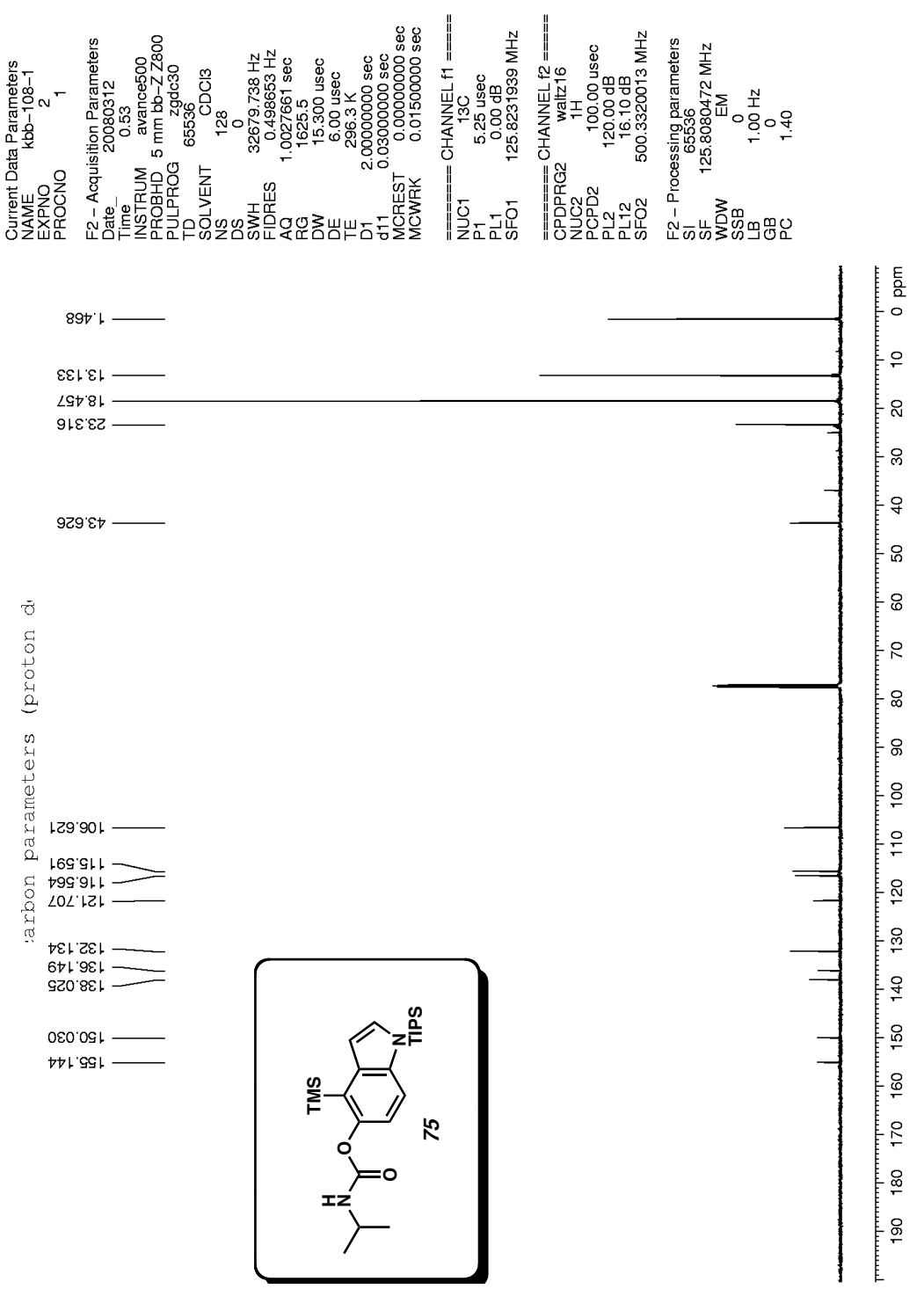

Current Data Parameters
NAME AEG-1-166aC13
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100404
Time 11.27
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 16384
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7578090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
AEG-1-166aC13
3.409
31.611
89.238
111.919
115.097
115.378
117.647
120.201
122.752
124.962
130.656
131.783
134.563
148.960
N
Me
BnO
60
0 ppm

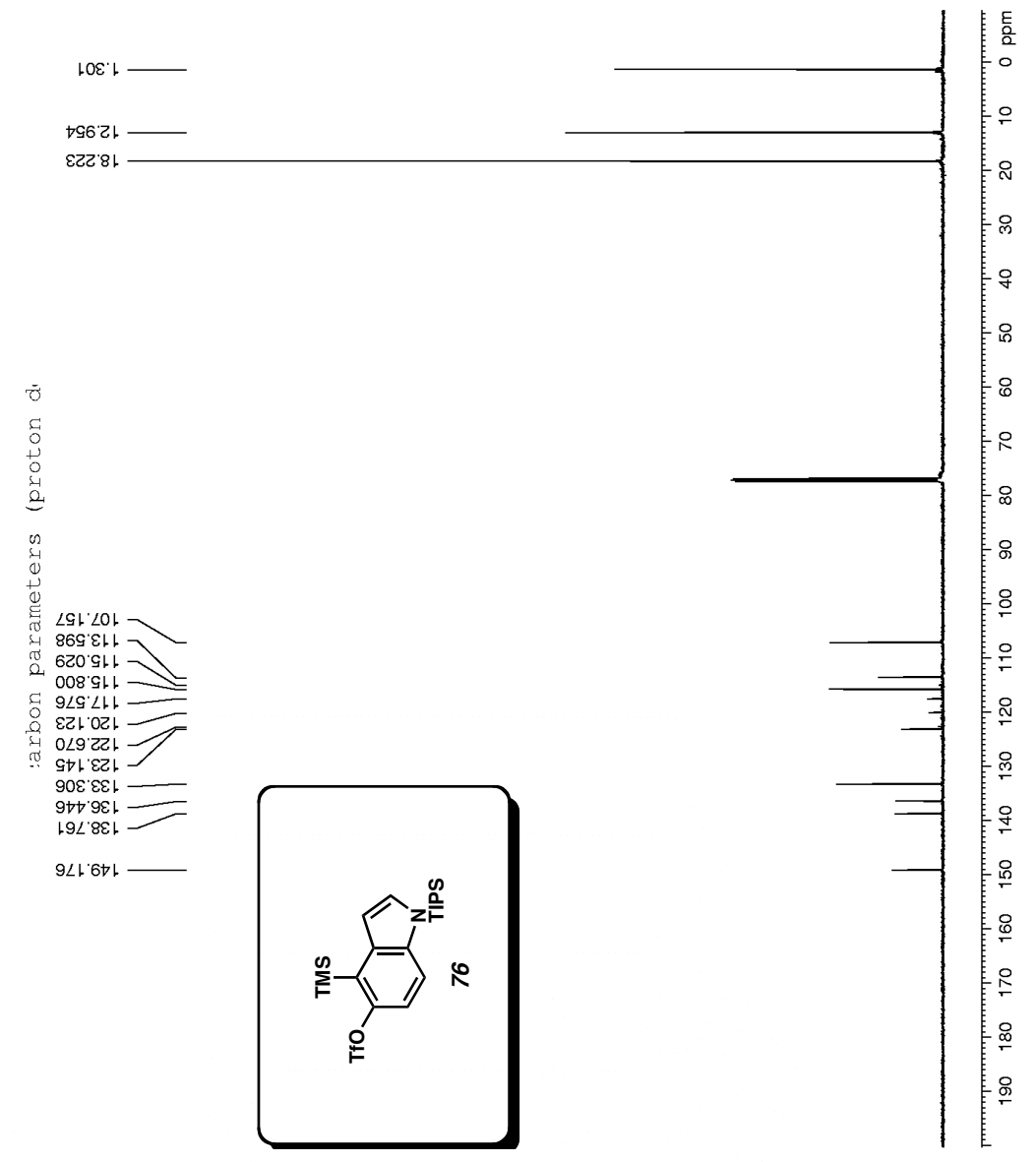

22.859
35.350
43.525
101.277
115.191
118.427
119.363
128.871
130.298
131.944
136.792
154.068
N
Me
O
O
NH
61
ppm

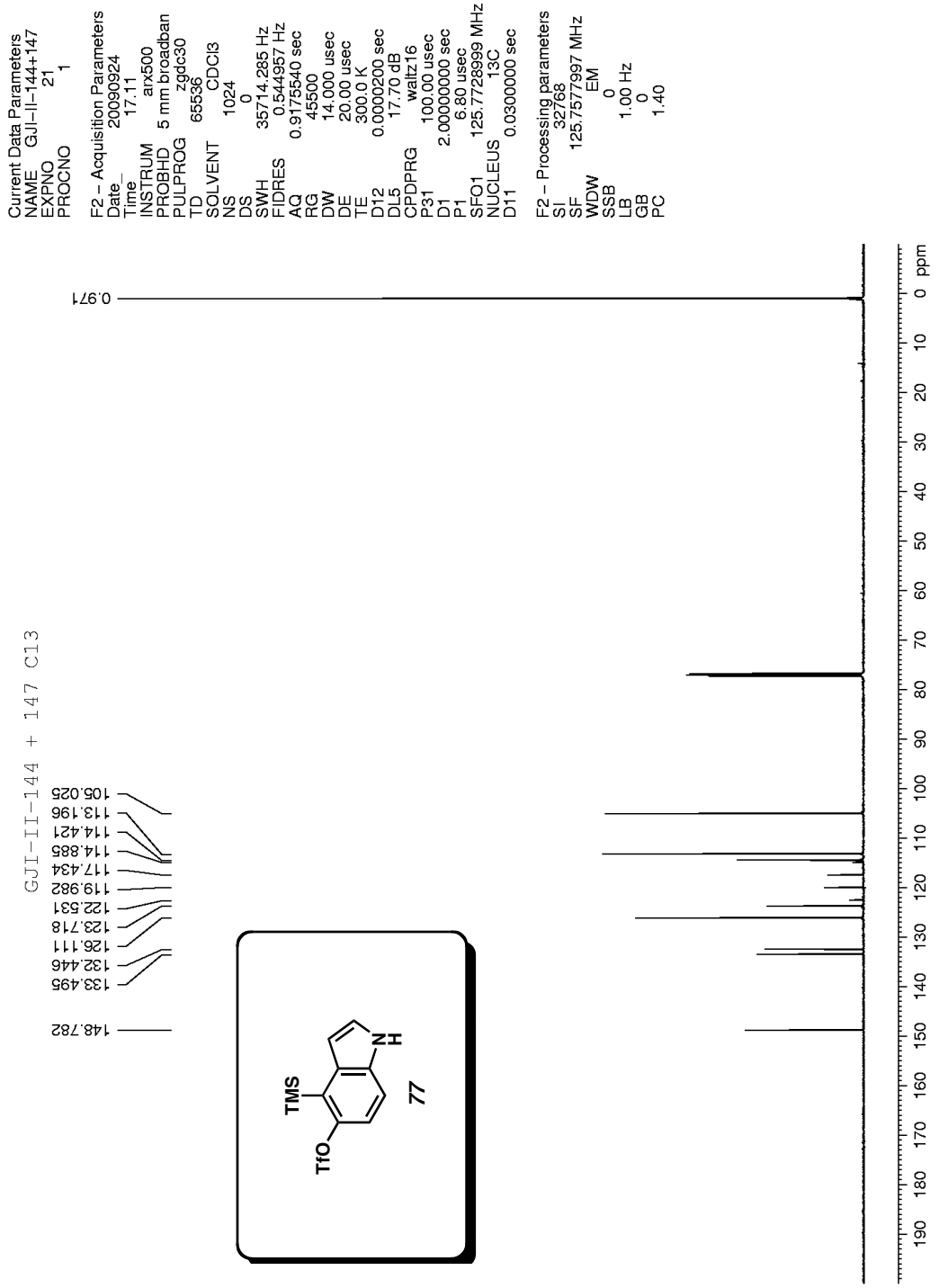

Current Data Parameters
NAME smb-3-184carbon
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100429
Time 19.29
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 137
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 5160.6
DW 15.300 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080663 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
carbon parameters (proton d
144.791
144.174
143.385
132.108
131.228
130.355
128.938
117.185
113.214
101.634
83.005
81.214
34.941
N
Me
O
72
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

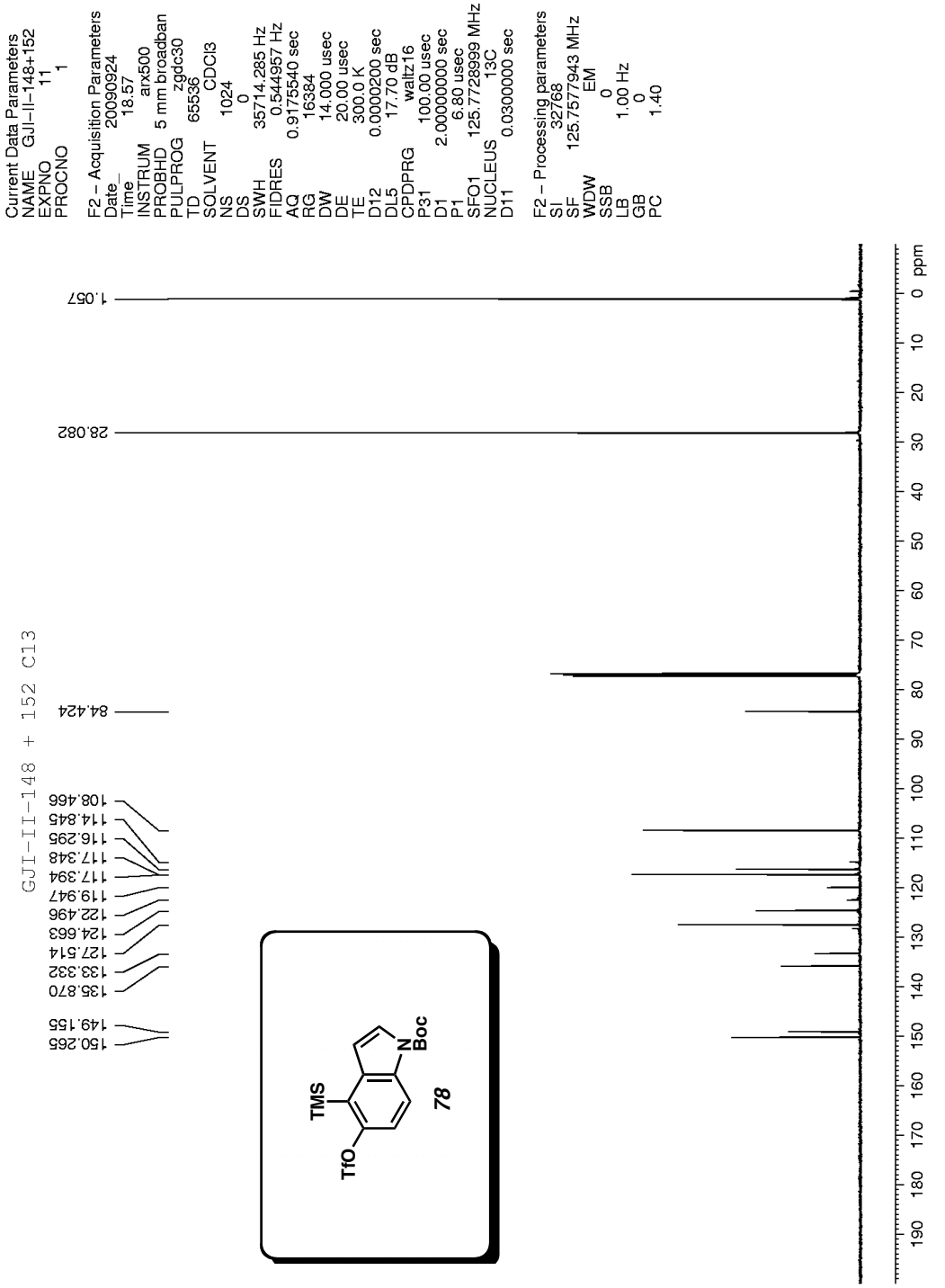

Current Data Parameters
NAME smb-3-188carbo
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100502
Time_ 19.16
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 131
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 16384
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7577864 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
meters for C-13 with proto
153.490
137.960
136.053
132.063
132.045
128.609
127.828
127.665
114.567
112.088
104.730
103.727
70.748
18.244
12.916
BnO
N
TIPS
73
10 ppm

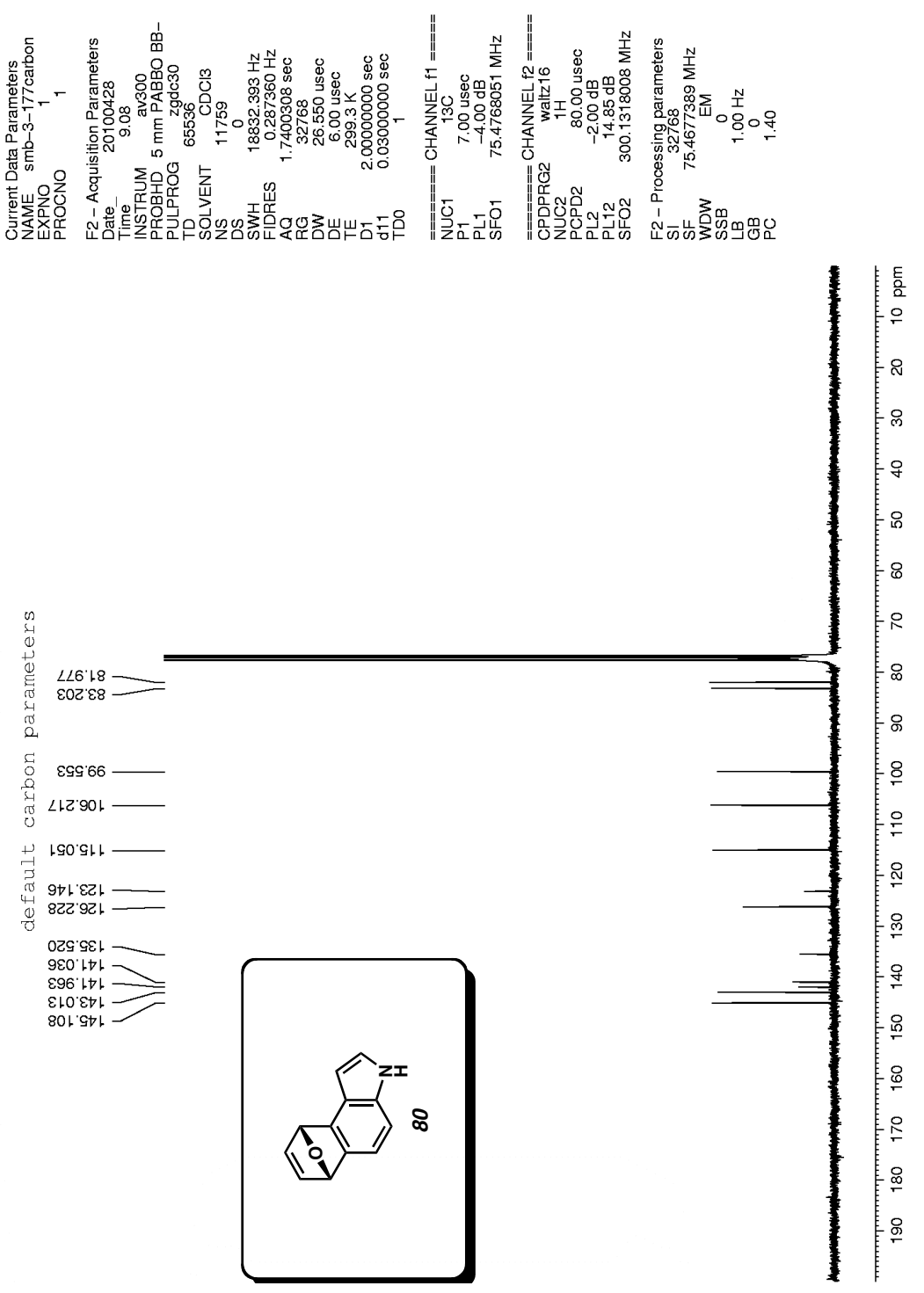

Current Data Parameters
NAME kbb-81-1
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20080224
Time 17.26
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 176
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 296.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080421 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
carbon parameters (proton d
155.015
145.011
138.709
132.636
132.151
115.962
114.216
112.938
105.336
43.697
23.296
18.424
13.116
N
H
O
O
N
TIPS
74
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

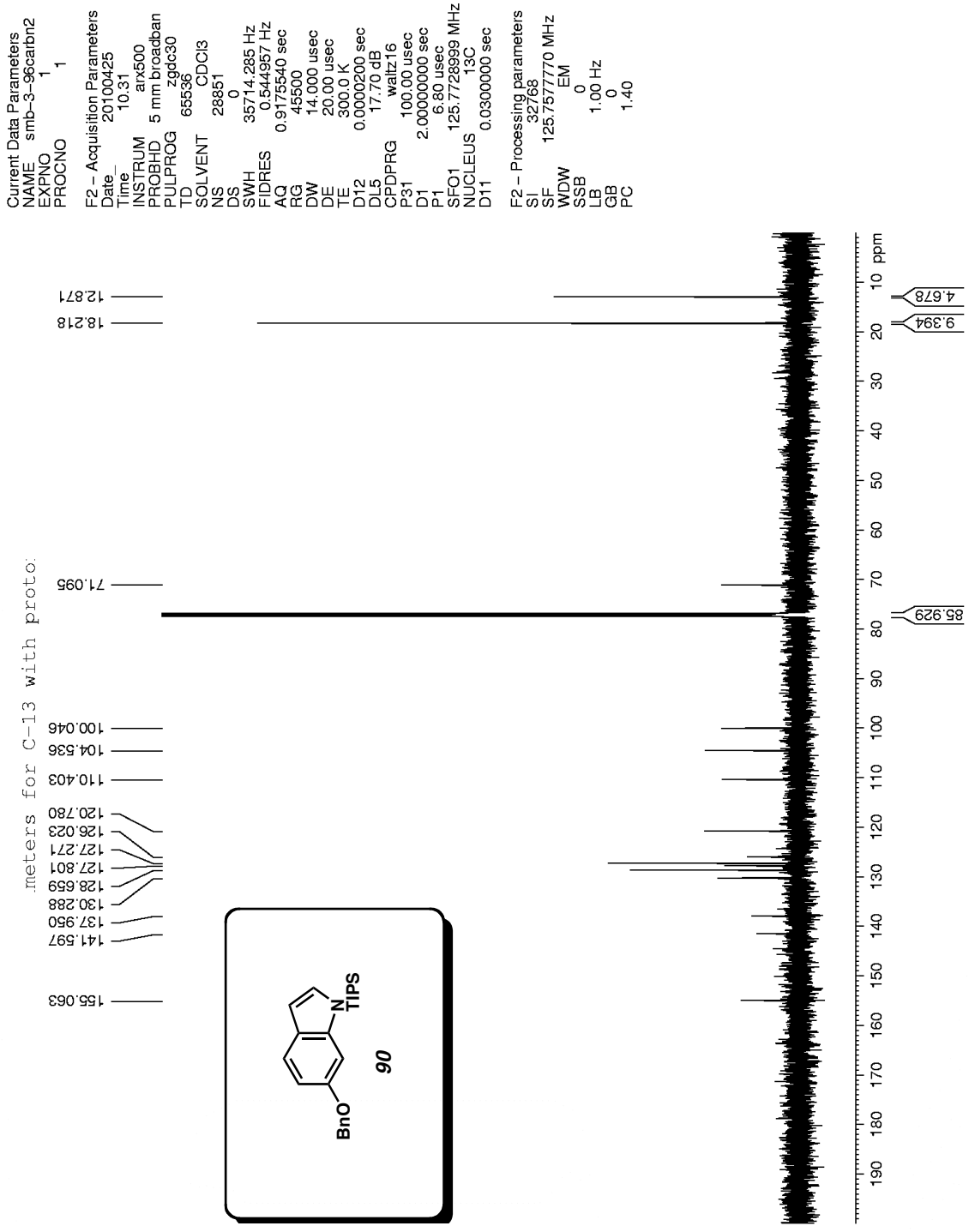

Current Data Parameters
NAME kbb-108-1
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20080312
Time 0.53
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 1625.5
DW 15.300 usec
DE 6.00 usec
TE 296.3 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080472 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
carbon parameters (proton d
1.468
13.133
18.457
23.316
43.626
106.621
115.591
116.564
121.707
132.134
136.149
138.025
150.030
155.144
TMS
TIPS
75
0 ppm

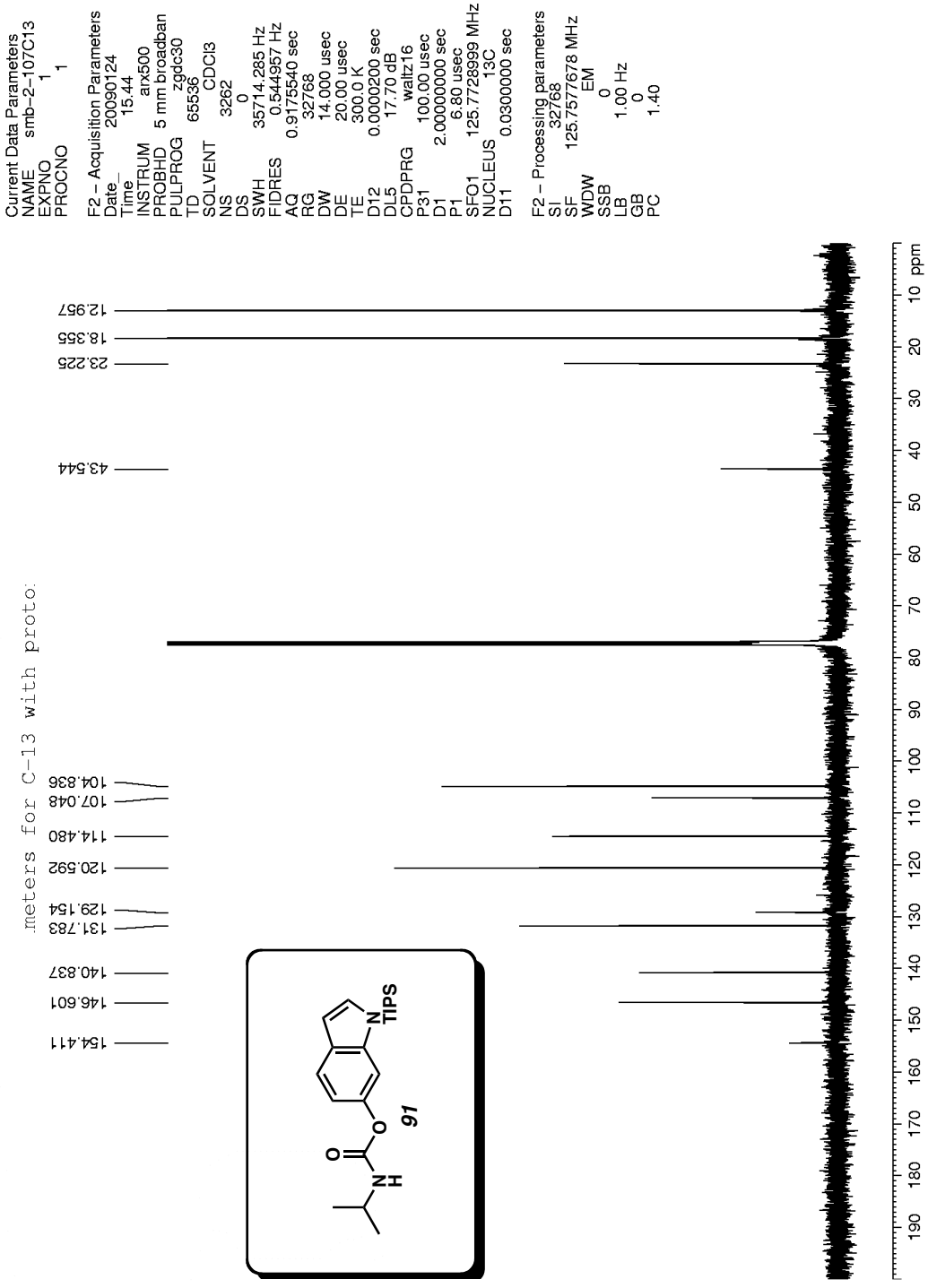

Current Data Parameters
NAME smb-2-196carbon2
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100520
Time 22.08
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 1213
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080636 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
carbon parameters (proton d
149.176
138.761
136.446
133.306
123.145
122.670
120.123
117.576
115.800
115.029
113.598
107.157
18.223
12.954
1.301
TMS
TfO
N
TIPS
76
0 ppm

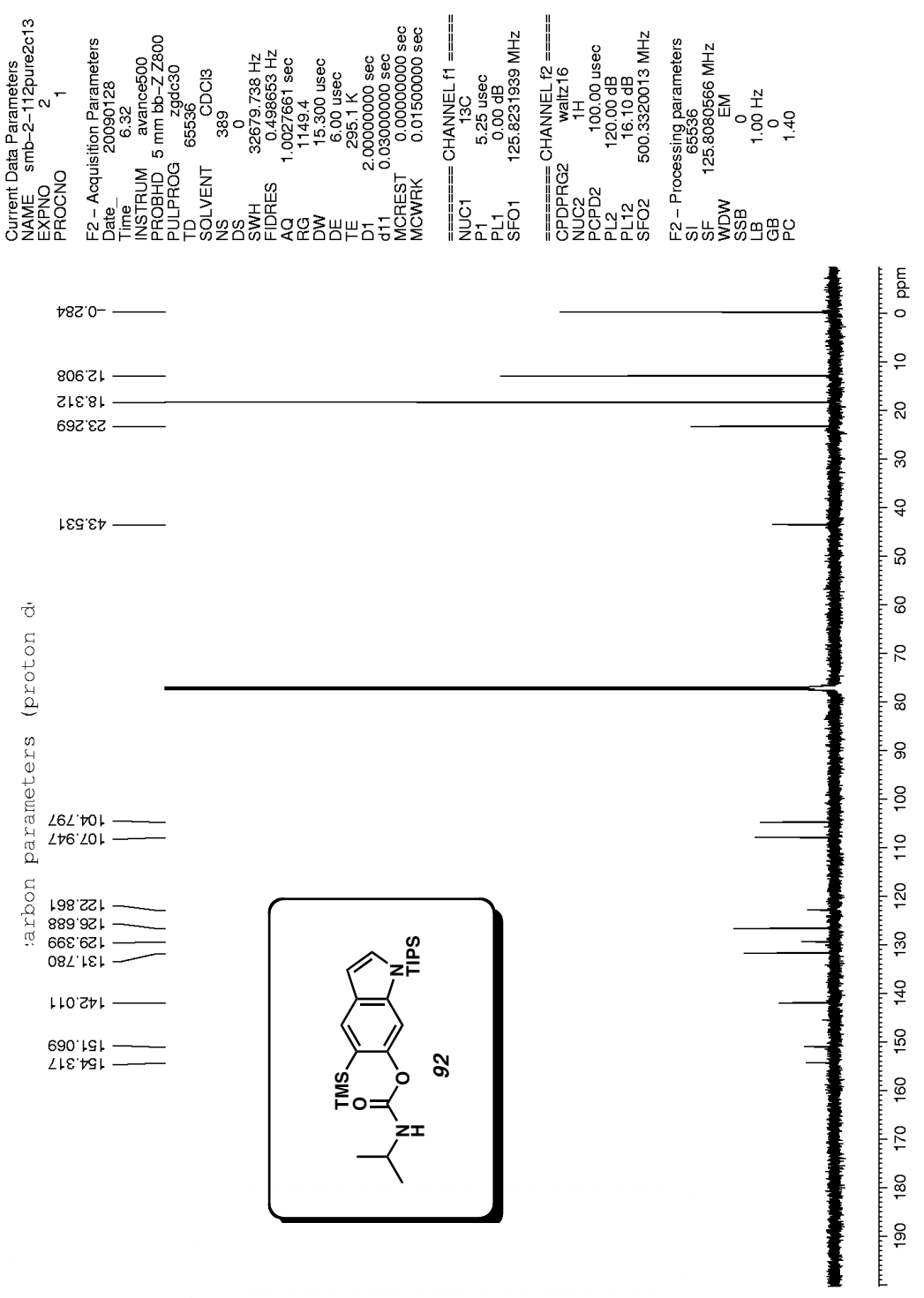

Current Data Parameters
NAME GJI-II-144+147
EXPNO 21
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090924
Time_ 17.11
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 1024
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 45500
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7577997 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-II-144 + 147 C13
148.782
133.495
132.446
126.111
123.718
122.531
119.982
117.434
114.885
114.421
113.196
105.025
0.971
TMS
TfO
77
ppm

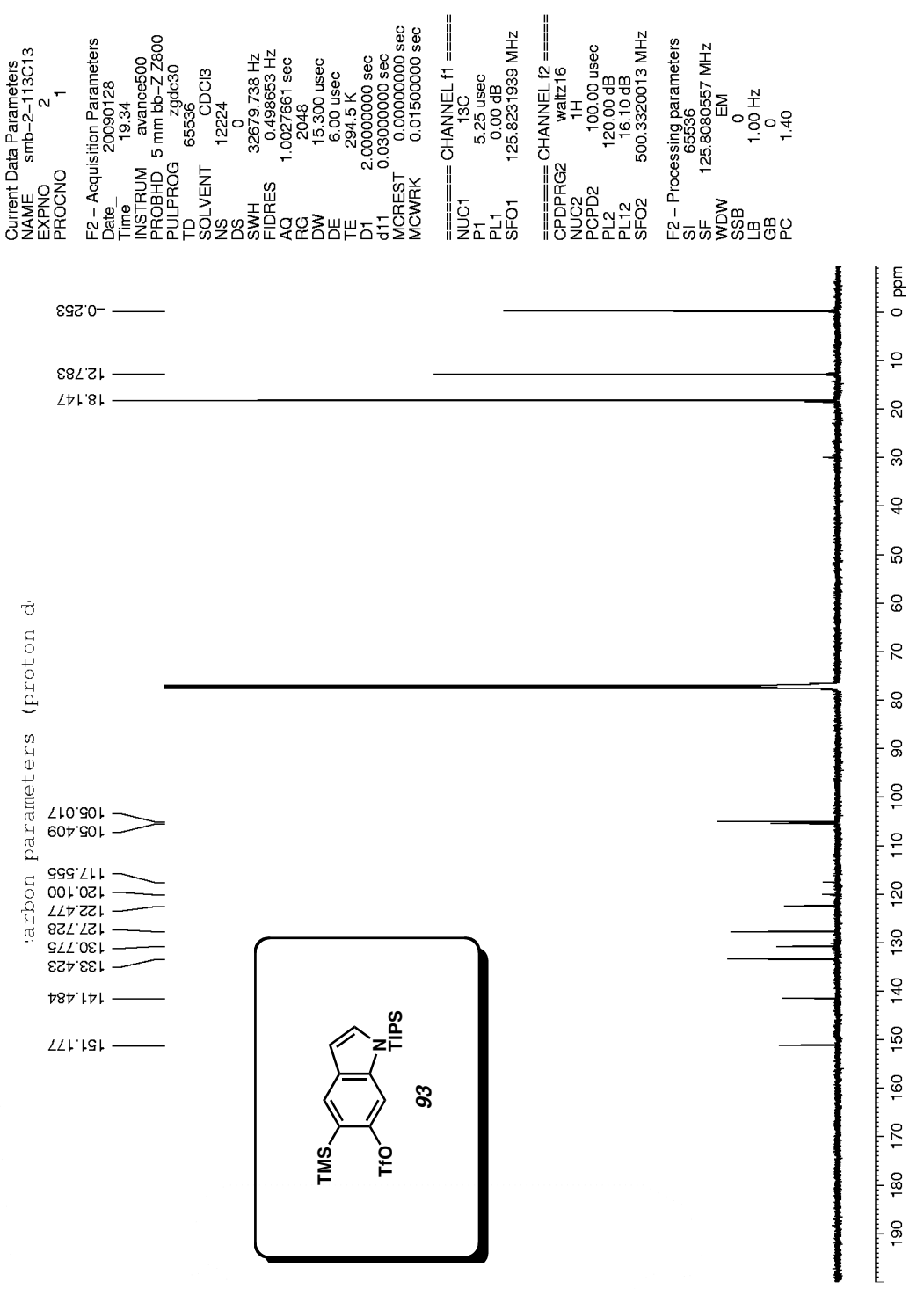

Current Data Parameters
NAME GJI-II-148+152
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090924
Time_ 18.57
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 1024
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 16384
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7577943 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-II-148 + 152 C13
1.057
28.082
84.424
108.466
114.845
116.295
117.348
117.394
119.947
122.496
124.663
127.514
133.332
135.870
149.155
150.265
TMS
TfO
N
Boc
78
ppm

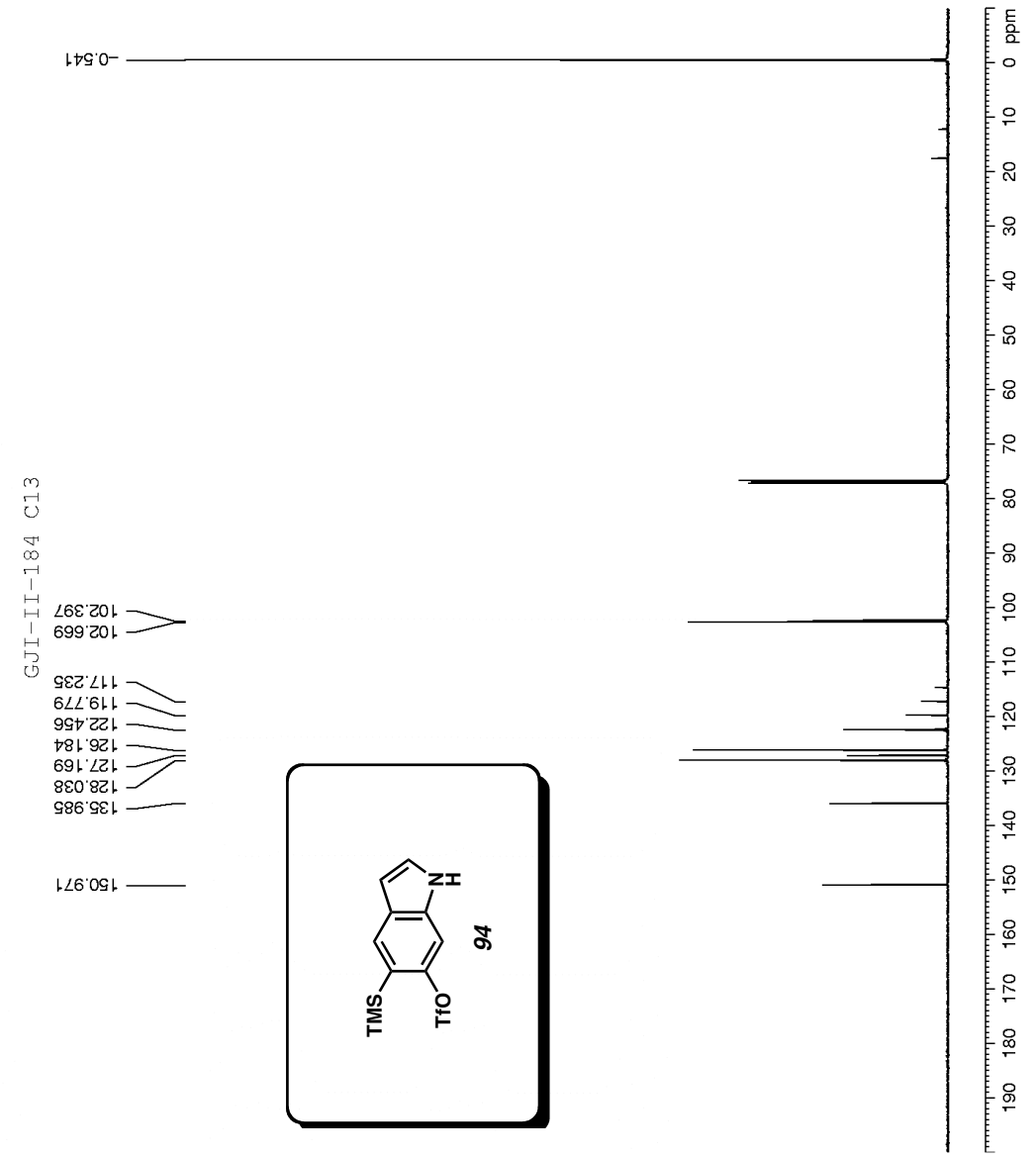

Current Data Parameters
NAME smb-3-177carbon
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100428
Time 9.08
INSTRUM av300
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 11759
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.74400308 sec
RG 32768
DW 26.550 usec
DE 6.00 usec
TE 299.3 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 7.00 usec
PL1 -4.00 dB
SFO1 75.4768051 MHz
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.85 dB
SFO2 300.1318008 MHz
F2 – Processing parameters
SI 32768
SF 75.4677389 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
default carbon parameters
145.108
143.013
141.963
141.036
135.520
126.228
123.146
115.051
106.217
99.553
83.203
81.977
80
NH
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

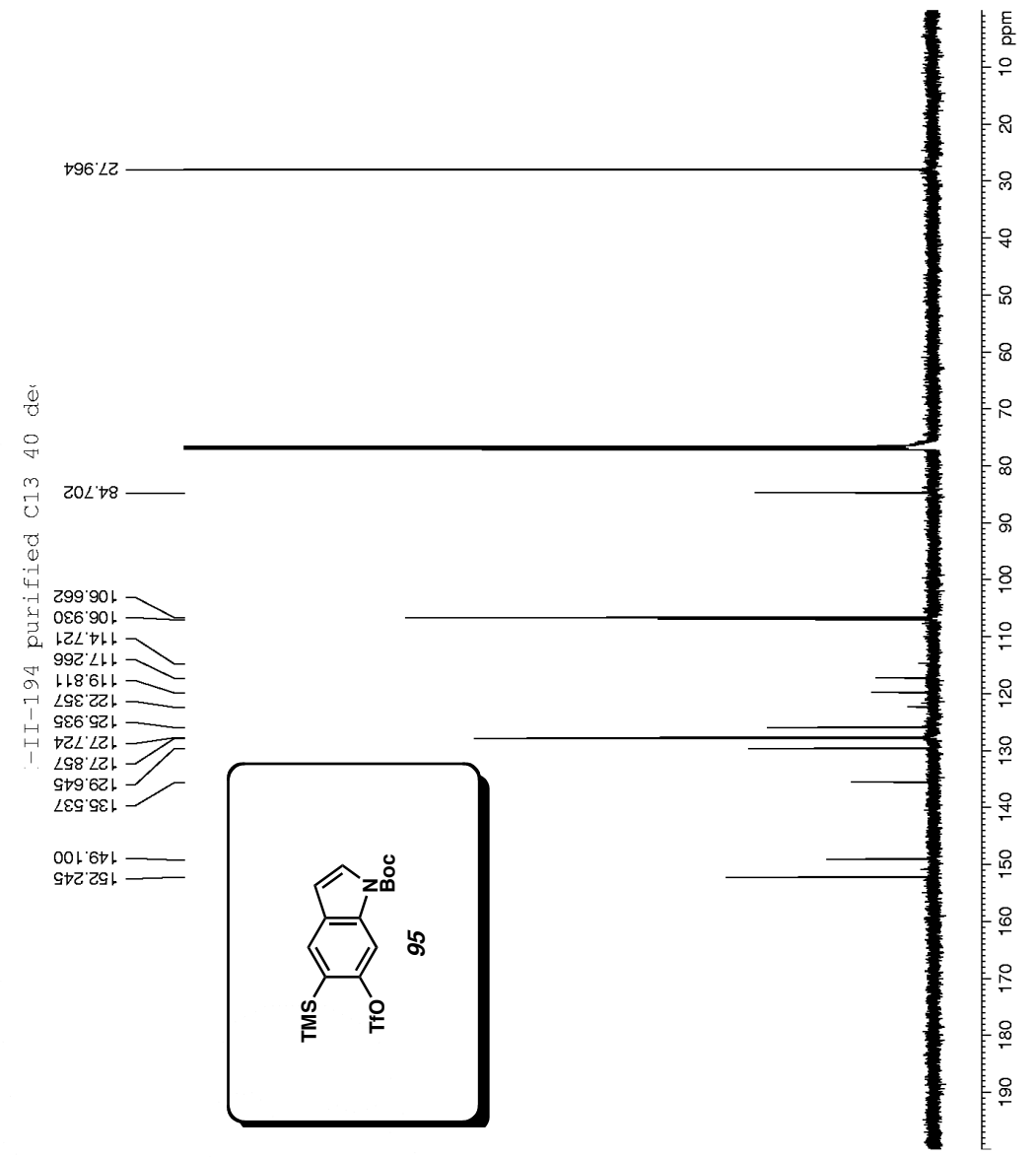

Current Data Parameters
NAME smb-3-96carbn2
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100425
Time 10.31
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 28851
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 45500
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7577770 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
meters for C-13 with proto
155.063
141.597
137.950
130.288
128.659
127.801
127.271
126.023
120.780
110.403
104.536
100.046
71.095
18.218
12.871
BnO
N
TIPS
90
85.929
9.394
4.678
ppm

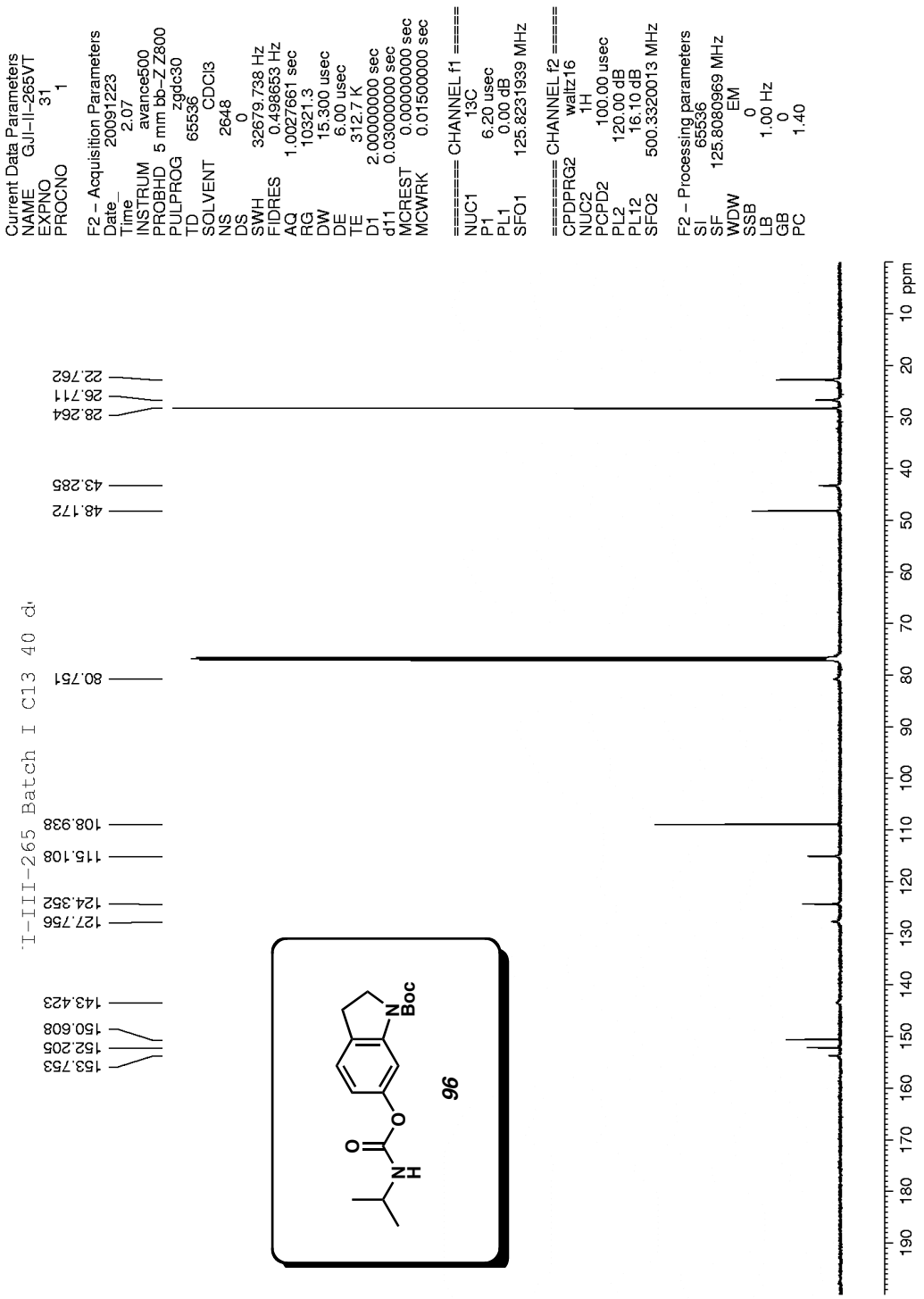

Current Data Parameters
NAME smb-2-107C13
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090124
Time 15.44
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 3262
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 32768
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7577678 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
meters for C-13 with proto
12.957
18.355
23.225
43.544
104.836
107.048
114.480
120.592
129.154
131.783
140.837
146.601
154.411
TIPS
91
10 ppm

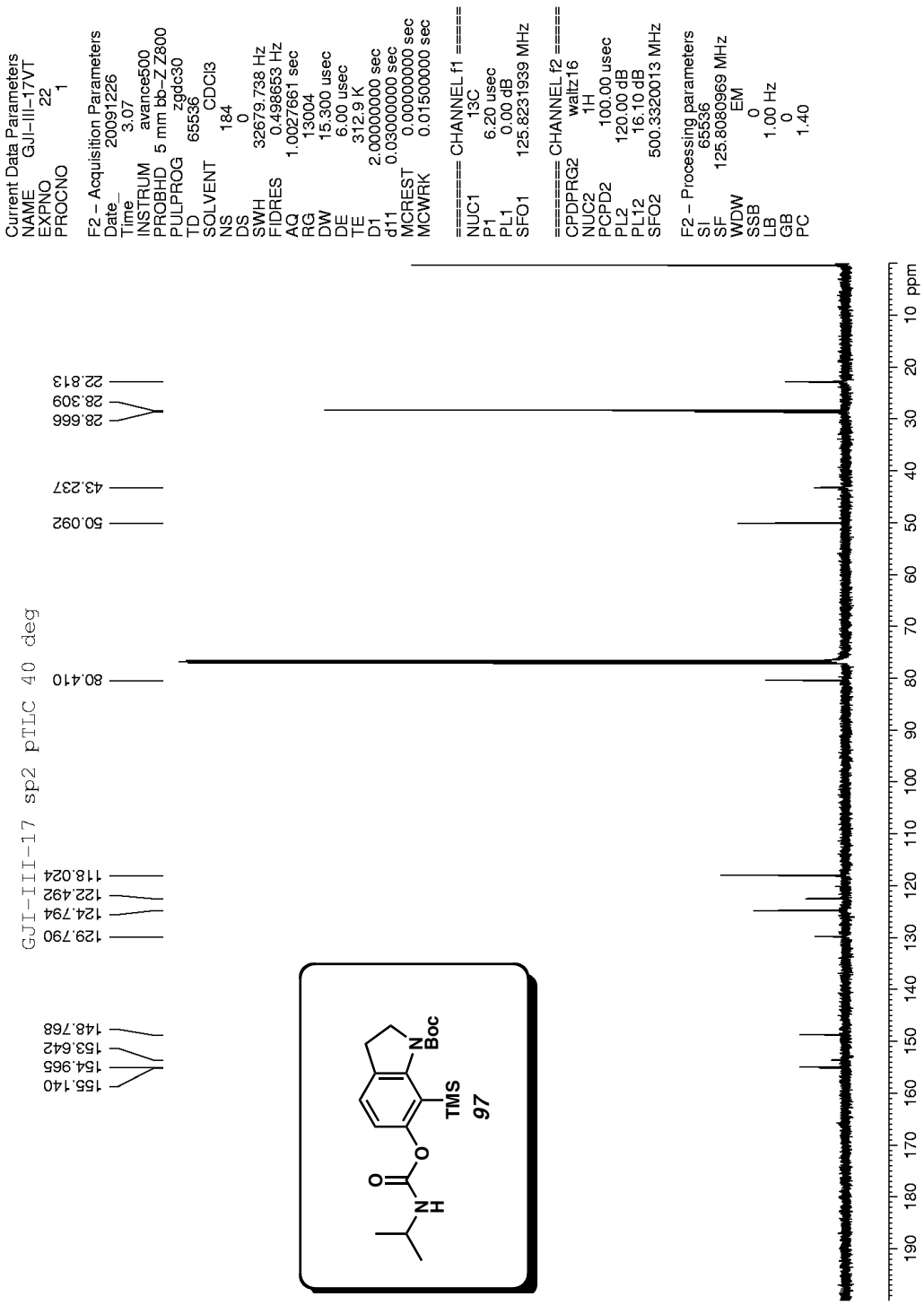

Current Data Parameters
NAME smb-2-112pure2c13
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090128
Time 6.32
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 389
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 1149.4
DW 15.300 usec
DE 6.00 usec
TE 295.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080566 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
carbon parameters (proton d
-0.284
12.908
18.312
23.269
43.531
104.797
107.947
122.861
126.688
129.399
131.780
142.011
151.069
154.317
TMS
TIPS
92
0 ppm

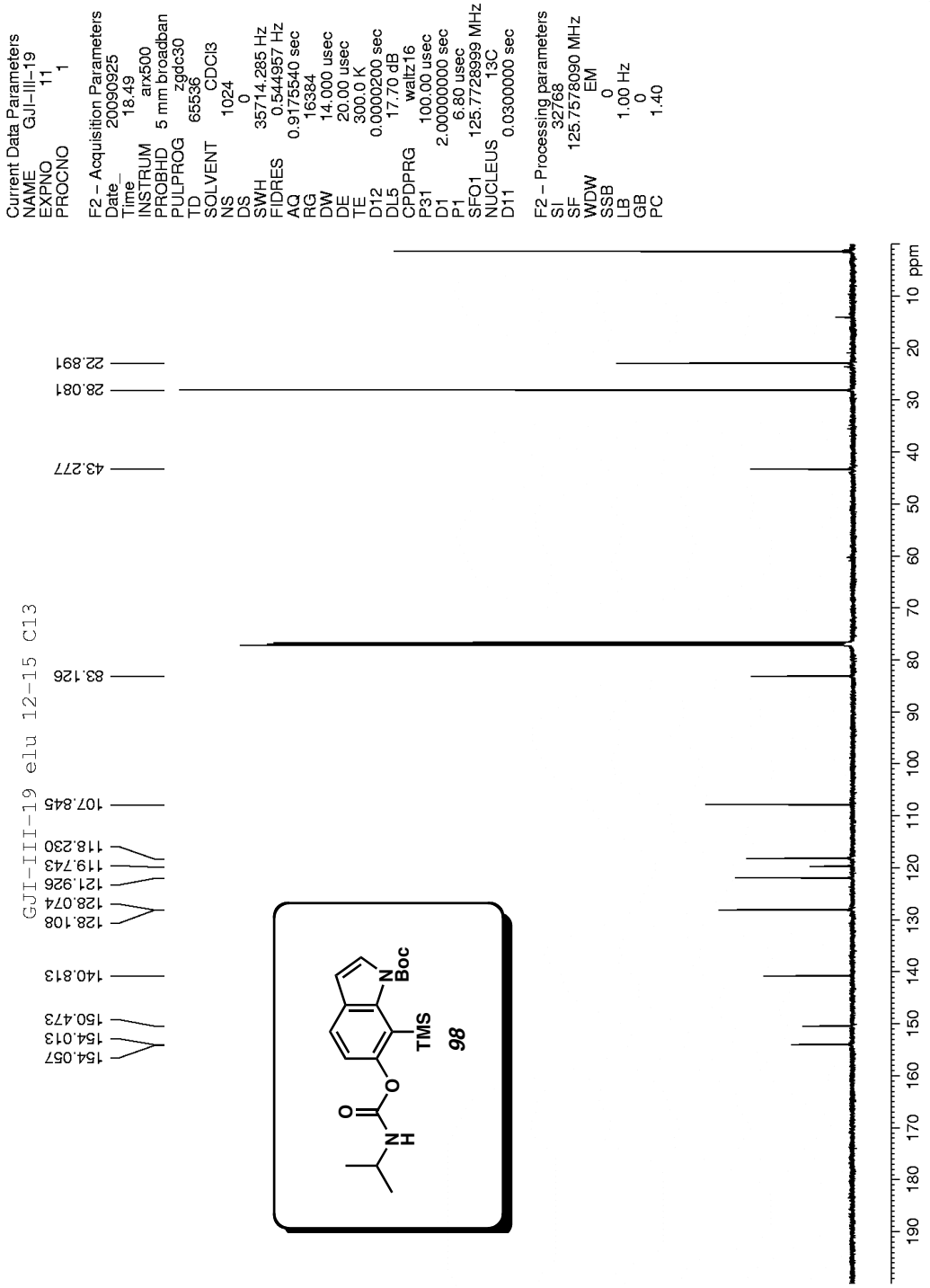

Current Data Parameters
NAME smb-2-113C13
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090128
Time 19.34
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 12224
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 2048
DW 15.300 usec
DE 6.00 usec
TE 294.5 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080557 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
carbon parameters (proton d
151.177
141.484
133.423
130.775
127.728
122.477
120.100
117.555
105.409
105.017
18.147
12.783
-0.253
TMS
TfO
N
TIPS
93
0 ppm

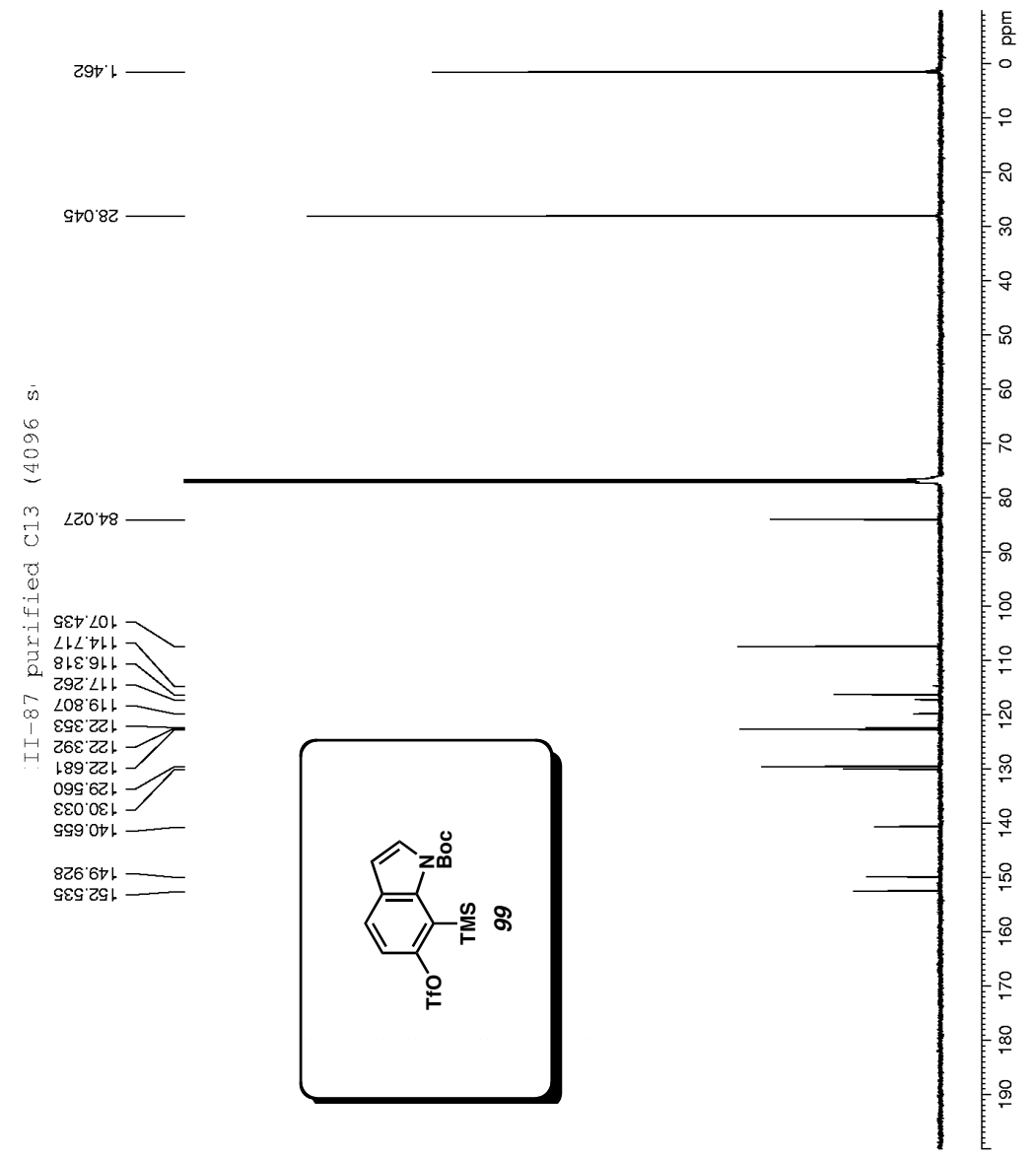

Current Data Parameters
NAME GJI-II-184
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090925
Time 17.38
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 1024
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 45500
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7578090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-II-184 C13
0.541
150.971
135.985
128.038
127.169
126.184
122.456
119.779
117.235
102.669
102.397
TMS
TfO
N
H
94
0 ppm

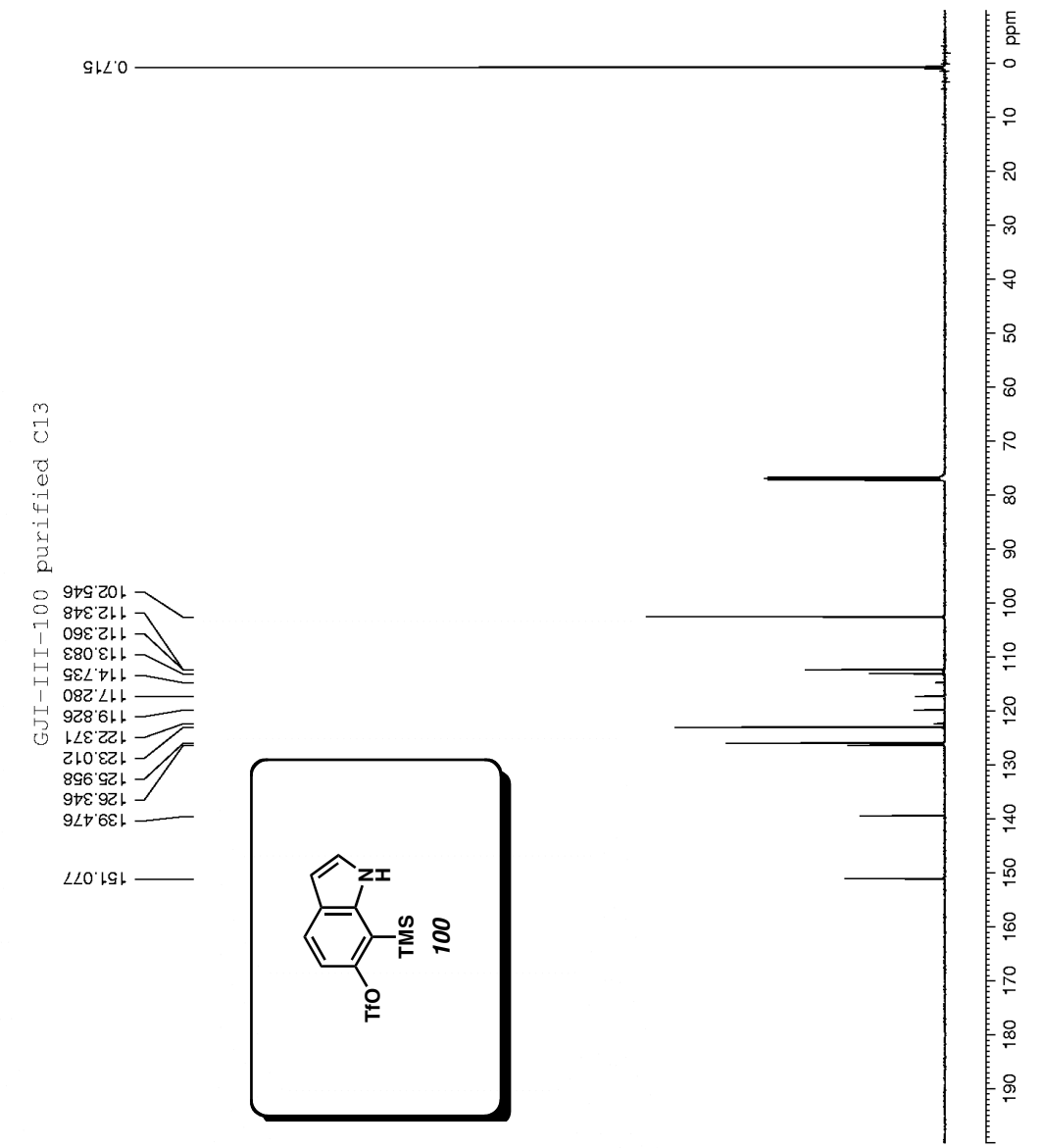

Current Data Parameters
NAME GJI-II-194C13
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100402
Time 20.19
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 1024
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 5792.6
DW 15.300 usec
DE 6.00 usec
TE 313.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
-II-194 purified C13 40 de
152.245
149.100
135.537
129.645
127.857
127.724
125.935
122.357
119.811
117.266
114.721
106.930
106.662
84.702
27.964
TMS
TfO
N
Boc
95
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

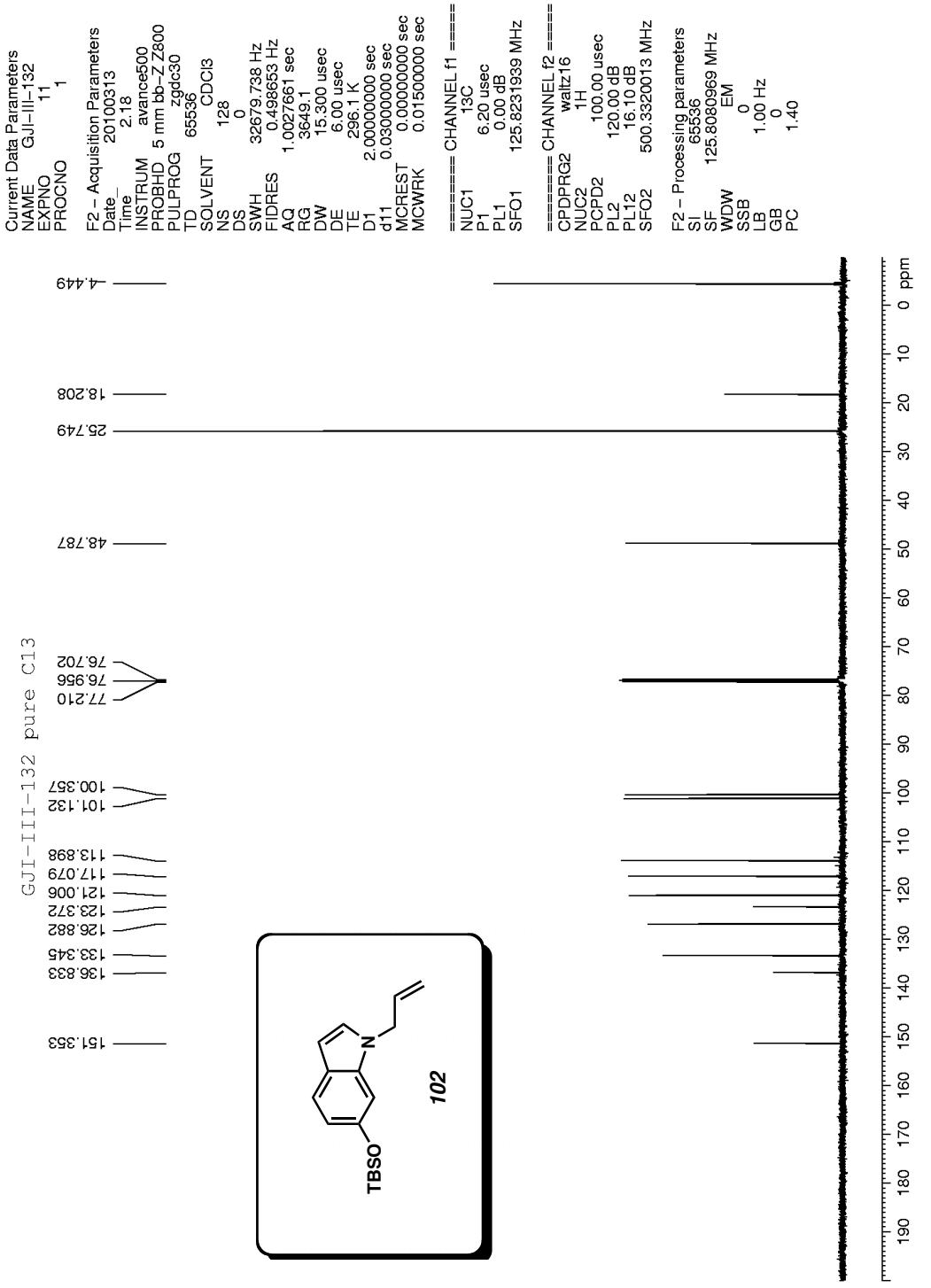

Current Data Parameters
NAME GJI-II-265VT
EXPNO 31
PROCNO 1
F2 – Acquisition Parameters
Date 20091223
Time 2.07
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 2648
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 10321.3
DW 15.300 usec
DE 6.00 usec
TE 312.7 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
I-III-265 Batch I C13 40 d
153.753
152.205
150.608
143.423
127.756
124.352
115.108
108.938
80.751
48.172
43.285
28.264
26.711
22.762
N
Boc
O
O
N
H
96
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

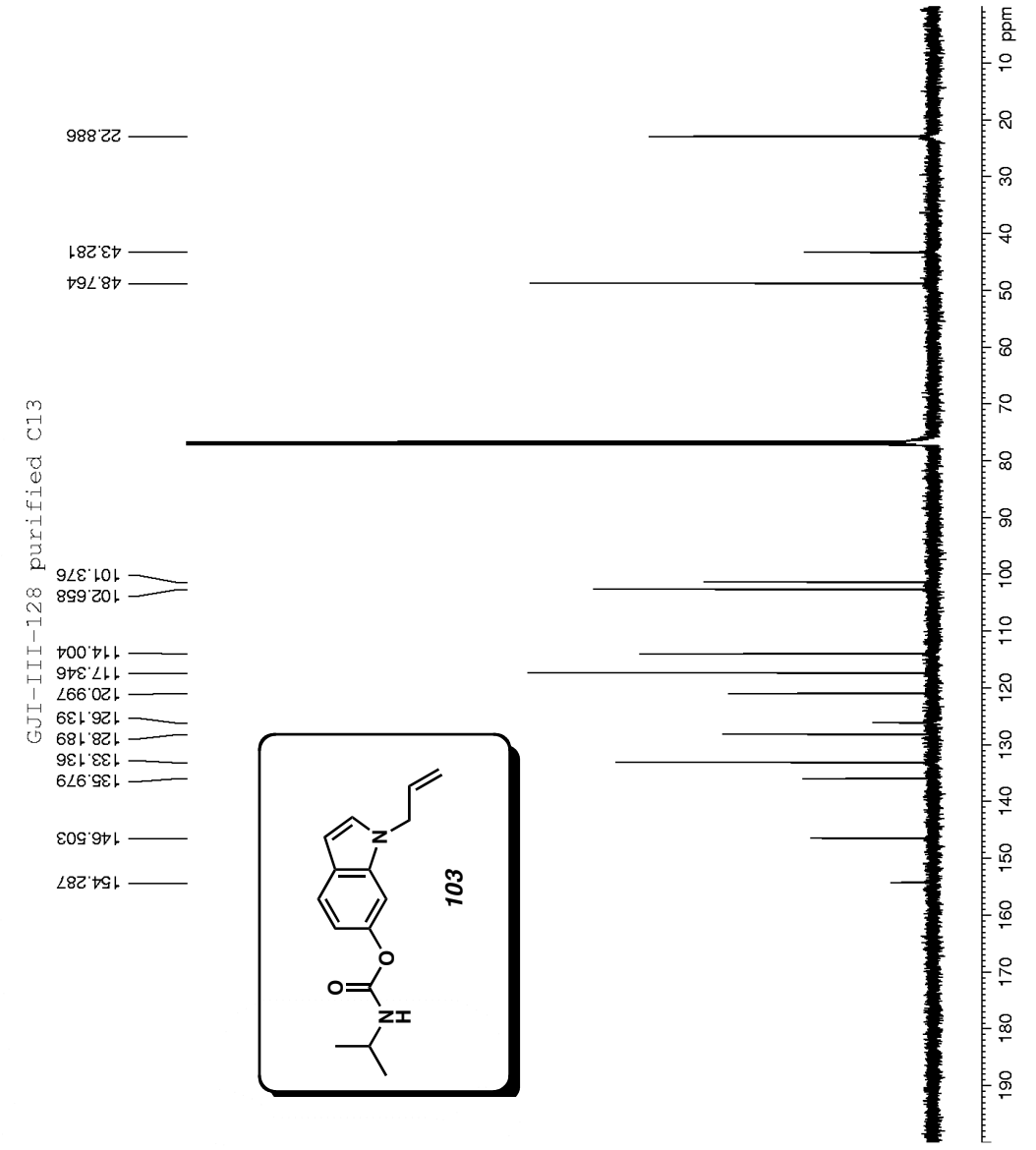

Current Data Parameters
NAME GJI-III-17VT
EXPNO 22
PROCNO 1
F2 – Acquisition Parameters
Date 20091226
Time 3.07
INSTRUM avance500
PROBHD 5 mm bb–Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 184
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 13004
DW 15.300 usec
DE 6.00 usec
TE 312.9 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-III-17 sp2 pTLC 40 deg
155.140
154.965
153.642
148.768
129.790
124.794
122.492
118.024
80.410
50.092
43.237
28.666
28.309
22.813
N
Boc
TMS
O
O
N
H
97
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

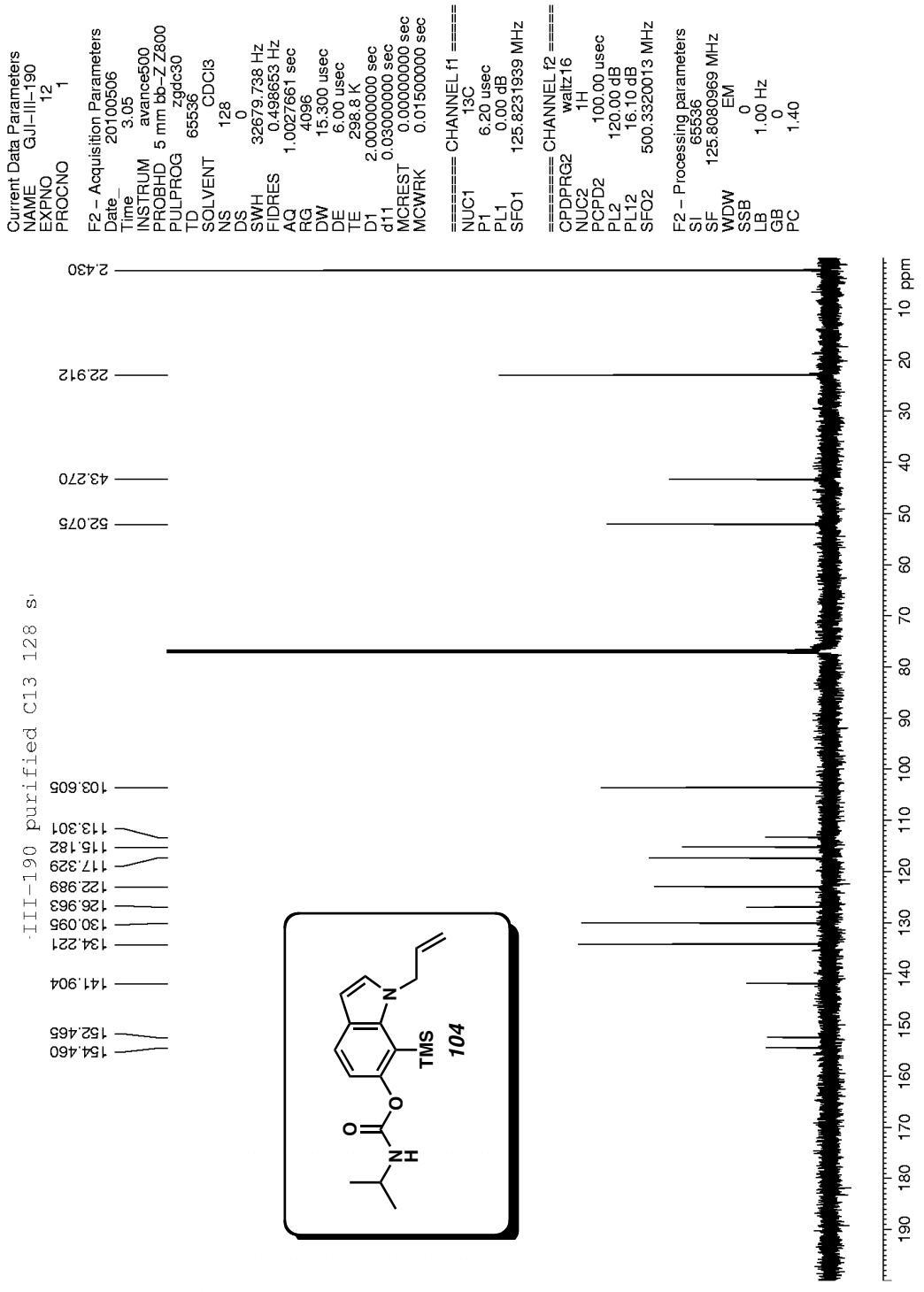

Current Data Parameters
NAME GJI-III-19
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090925
Time 18.49
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 1024
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 16384
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7578090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-III-19 elu 12-15 C13
154.057
154.013
150.473
140.813
128.108
128.074
121.926
119.743
118.230
107.845
83.126
43.277
28.081
22.891
N
Boc
TMS
O
O
N
H
98
10 ppm

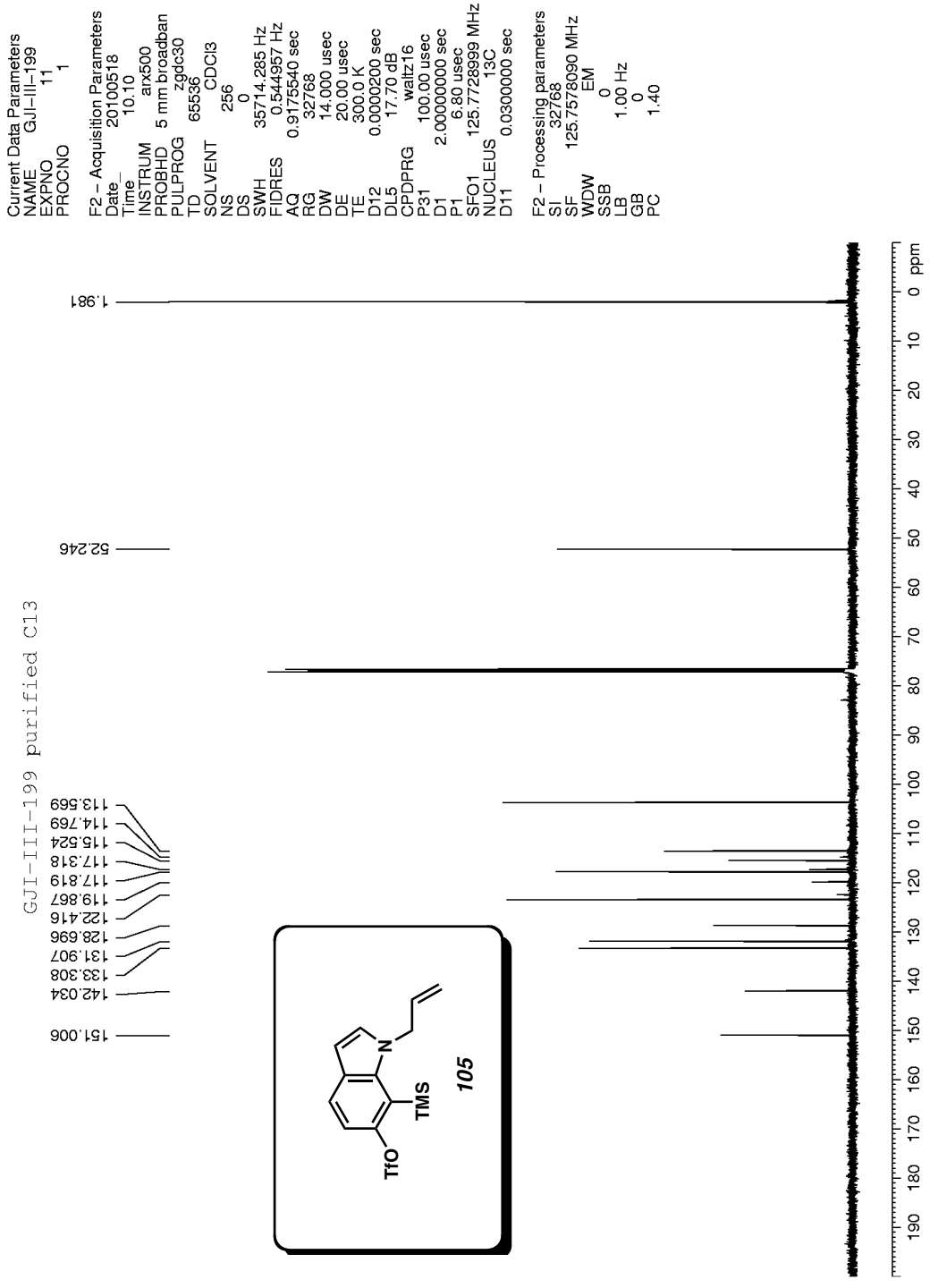

Current Data Parameters
NAME GJI-III-87
EXPNO 31
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091219
Time 5.34
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 4096
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 6502
DW 15.300 usec
DE 6.00 usec
TE 294.6 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
II-87 purified C13 (4096 s
1.462
28.045
84.027
107.435
114.717
116.318
117.262
119.807
122.353
122.392
122.681
129.560
130.033
140.655
149.928
152.535
N
Boc
TMS
TfO
99
0 ppm

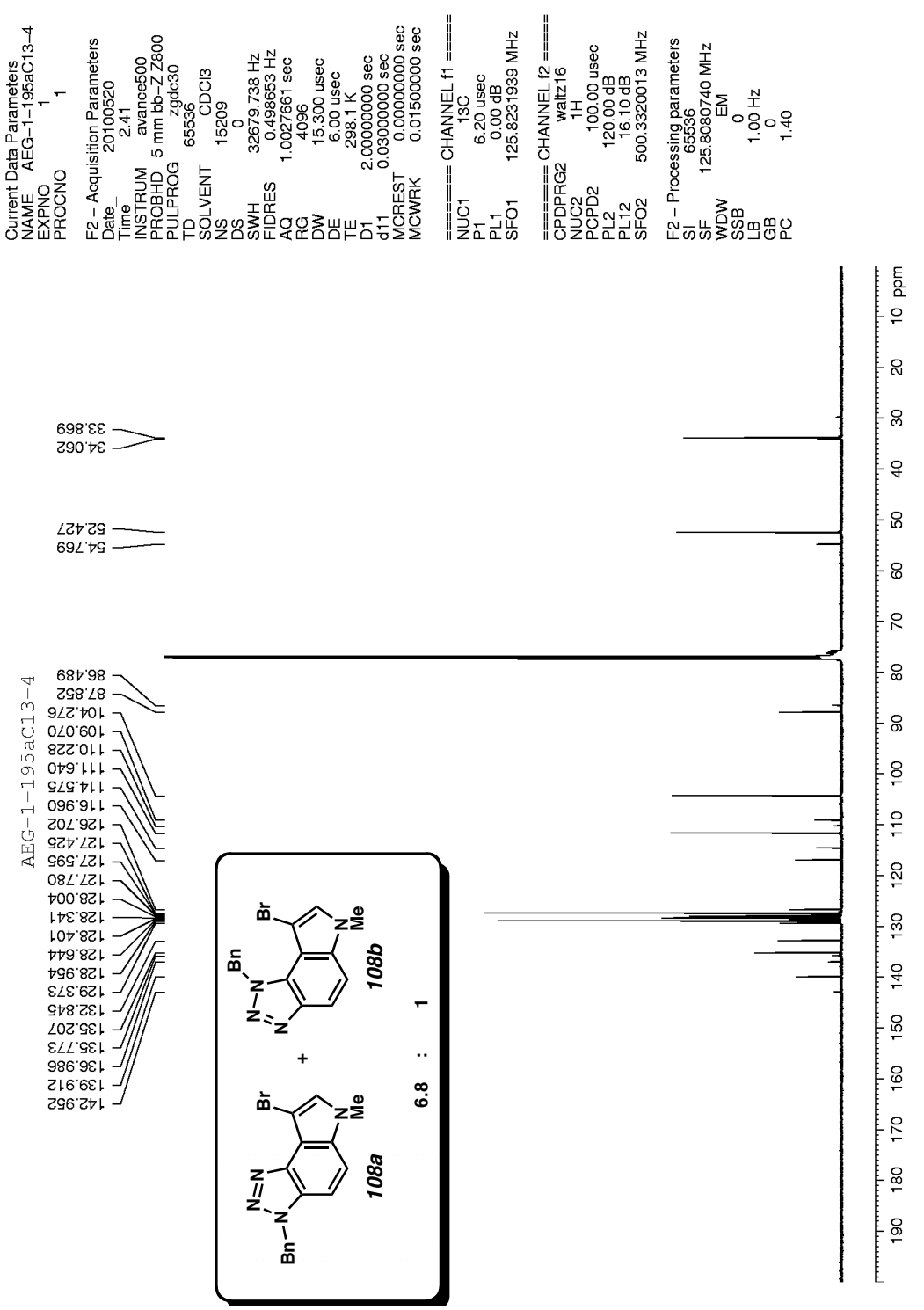

Current Data Parameters
NAME GJI-III-100
EXPNO 21
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091215
Time_ 5.02
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 2048
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 13004
DW 15.300 usec
DE 6.00 usec
TE 294.8 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-III-100 purified C13
0.715
151.077
139.476
126.346
125.958
123.012
122.371
119.826
117.280
114.735
113.083
112.360
112.348
102.546
TfO
TMS
N
H
100
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

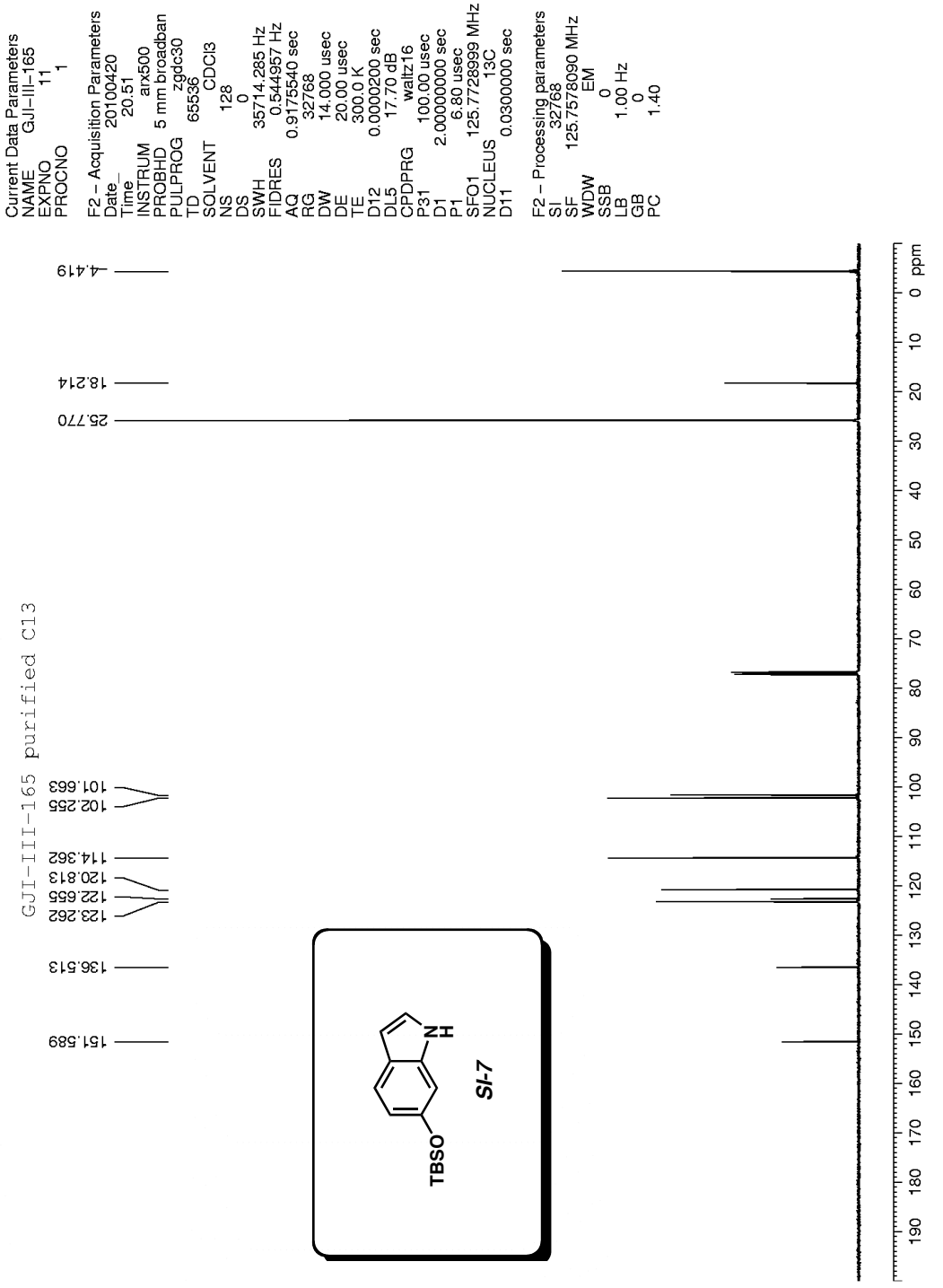

Current Data Parameters
NAME GJI-III-132
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100313
Time 2.18
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3649.1
DW 15.300 usec
DE 6.00 usec
TE 296.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-III-132 pure C13
4.449
18.208
25.749
48.787
76.702
76.956
77.210
100.357
101.132
113.898
117.079
121.006
123.372
126.882
133.345
136.833
151.353
TBSO
N
102
0 ppm

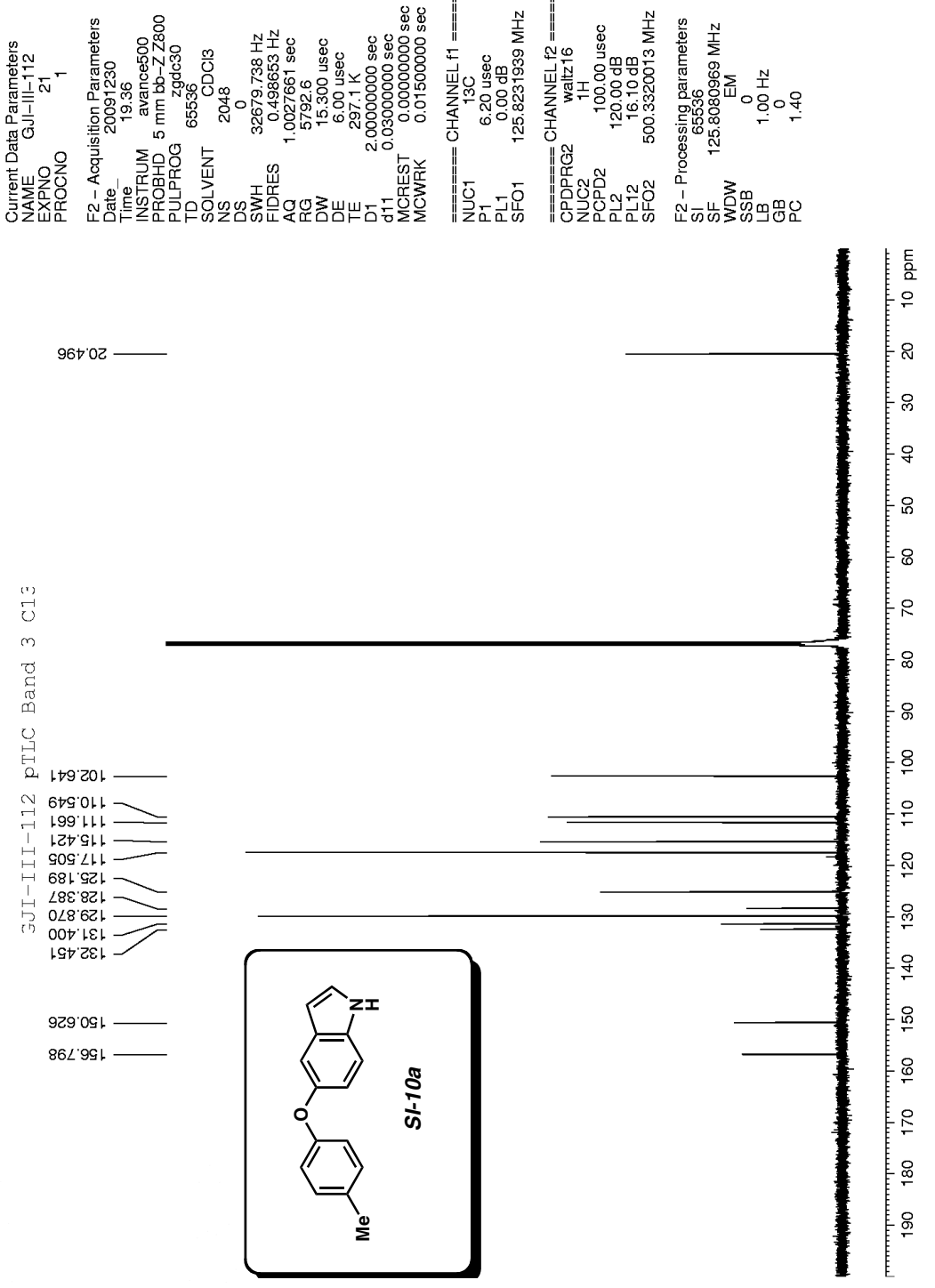

Current Data Parameters
NAME GJI-III-128
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100317
Time 3.01
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 13004
DW 15.300 usec
DE 6.00 usec
TE 296.6 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-III-128 purified C13
22.886
43.281
48.764
101.376
102.658
114.004
117.346
120.997
126.139
128.189
133.136
135.979
146.503
154.287
103
ppm

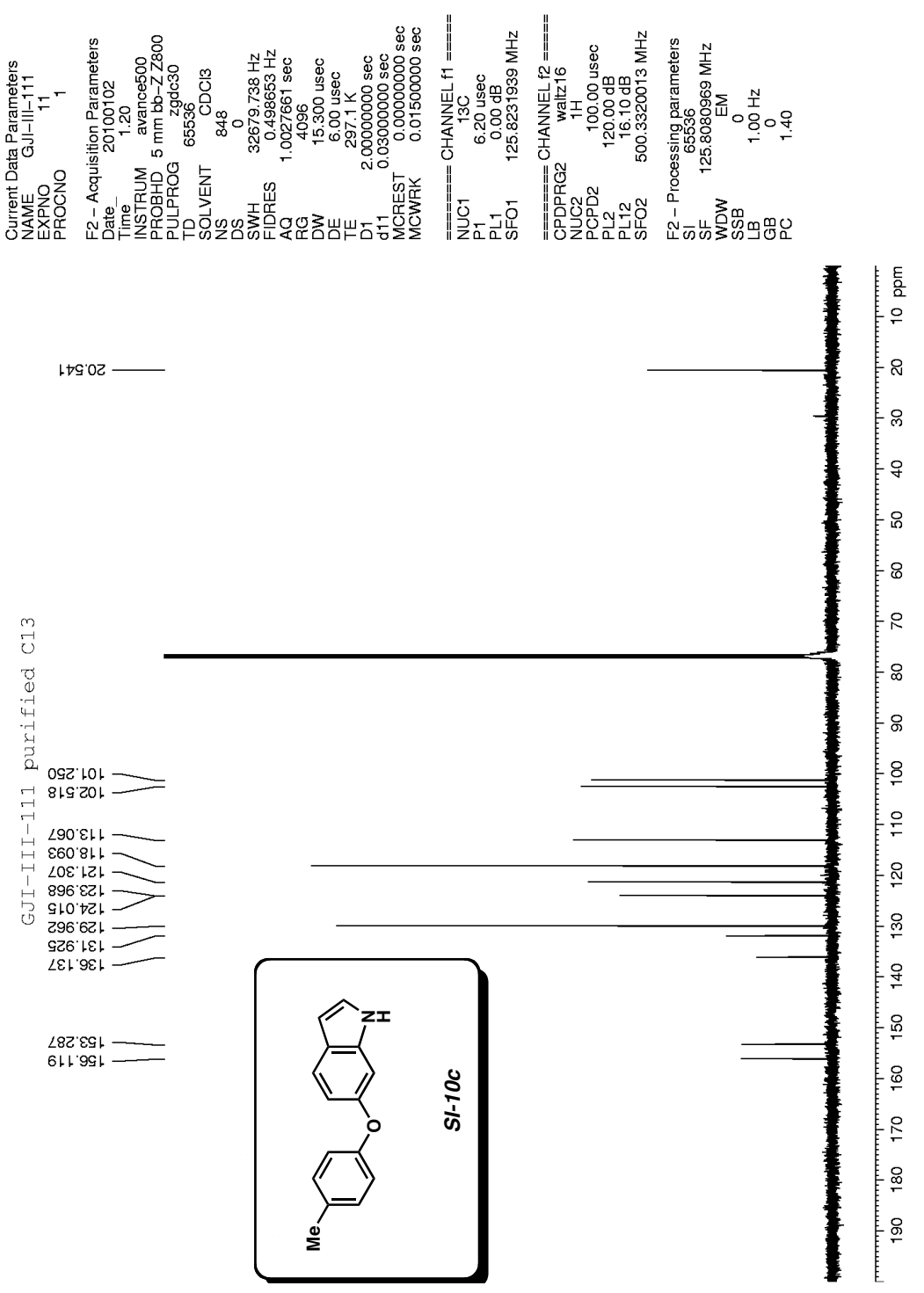

Current Data Parameters
NAME GJI-III-190
EXPNO 12
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100506
Time 3.05
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 298.8 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
III-190 purified C13 128 s
2.430
22.912
43.270
52.075
103.605
113.301
115.182
117.329
122.989
126.963
130.095
134.221
141.904
152.465
154.460
TMS
104
10 ppm

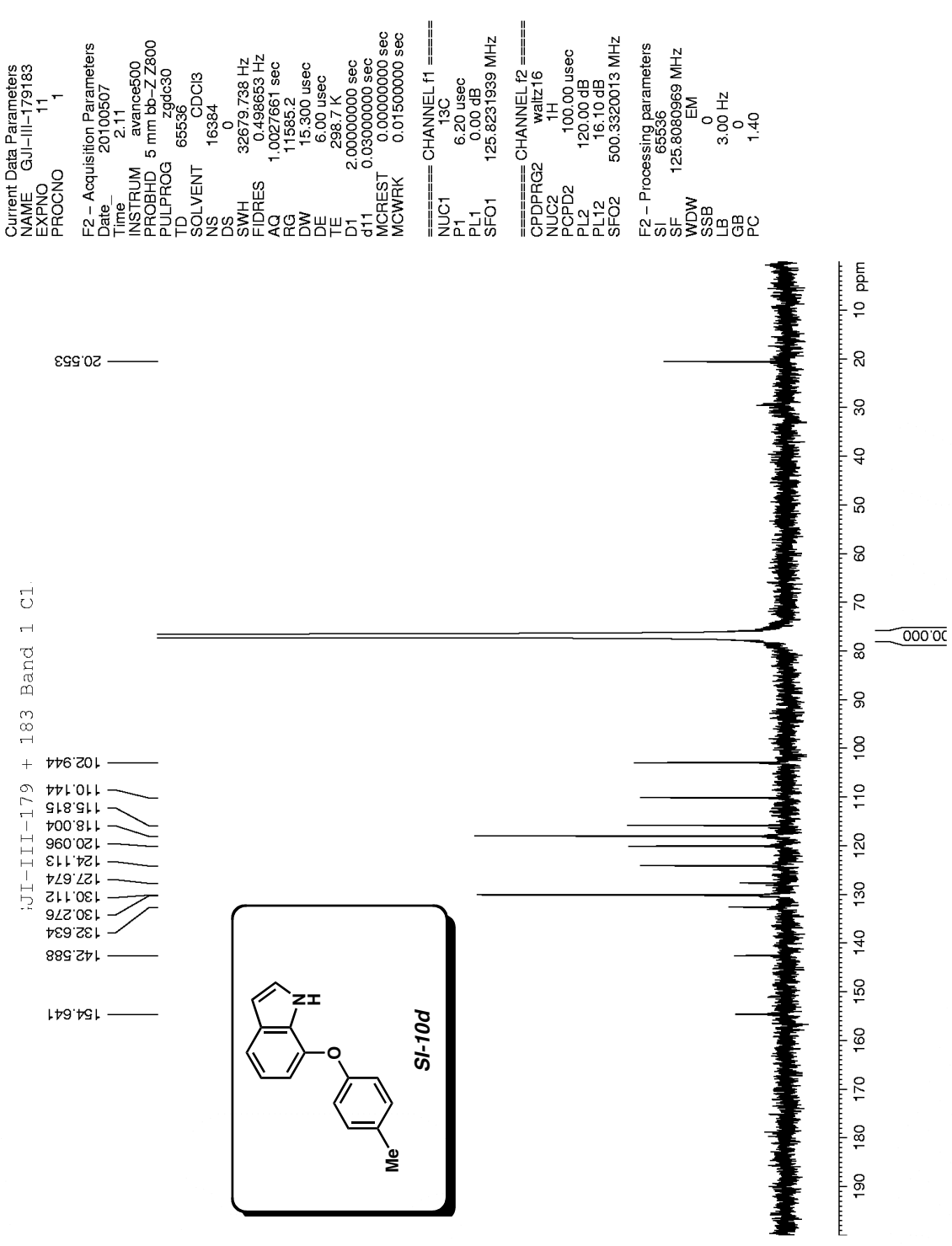

GJI-III-199 purified C13
Current Data Parameters
NAME GJI-III-199
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100518
Time_ 10.10
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 32768
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7578090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1.981
52.246
113.569
114.769
115.524
117.318
117.819
119.867
122.416
128.696
131.907
133.308
142.034
151.006
TfO
TMS
N
105
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

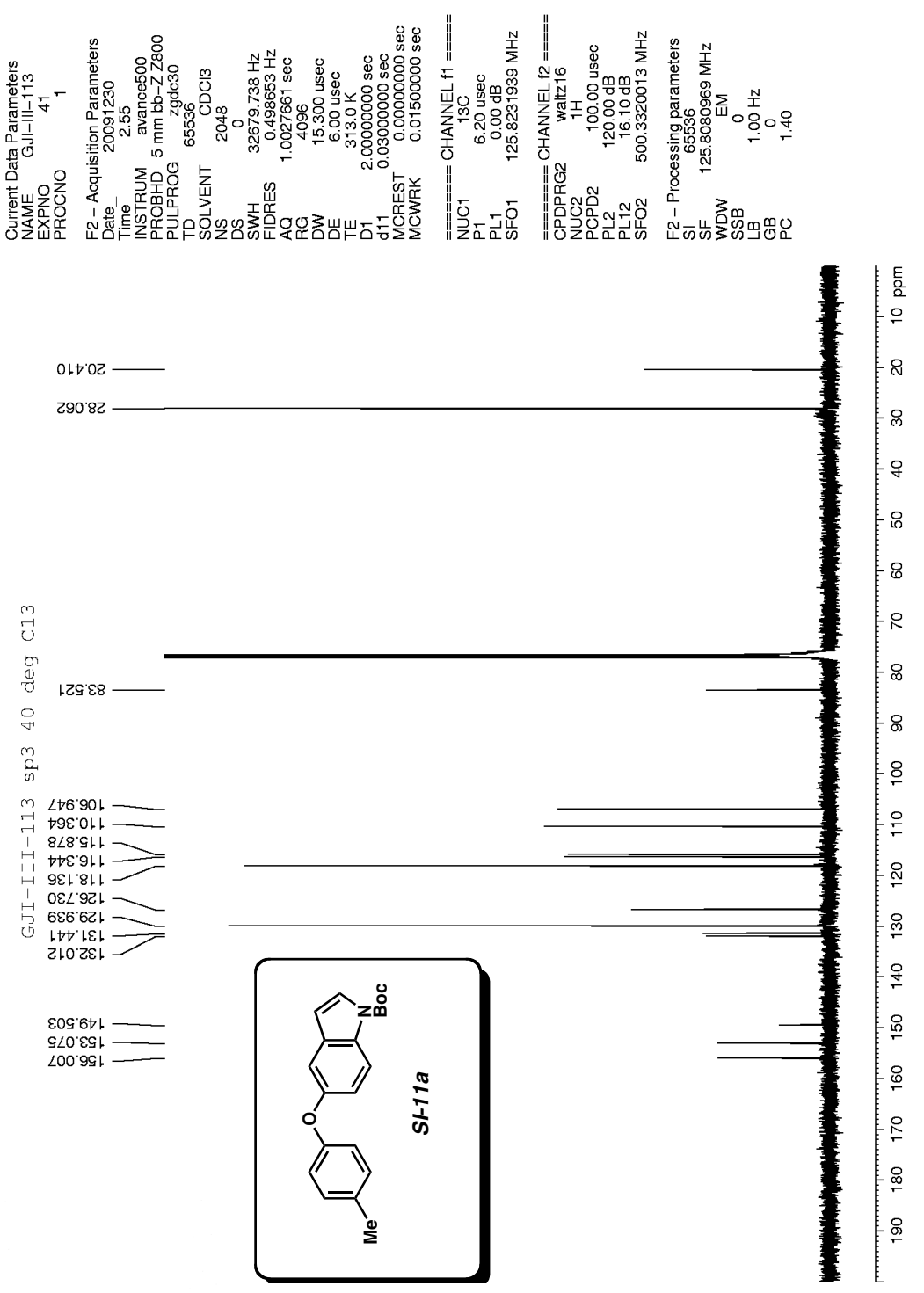

Current Data Parameters
NAME AEG-1-195aC13-4
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100520
Time_ 2.41
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 15209
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 298.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080740 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
AEG-1-195aC13-4
142.952
139.912
136.986
135.773
135.207
132.845
129.373
128.954
128.644
128.401
128.341
128.004
127.780
127.595
127.425
126.702
116.960
114.575
111.640
110.228
109.070
104.276
87.852
86.489
54.769
52.427
34.062
33.869
Bn
N
N
N
Br
Me
108a
+
108b
6.8 : 1
ppm

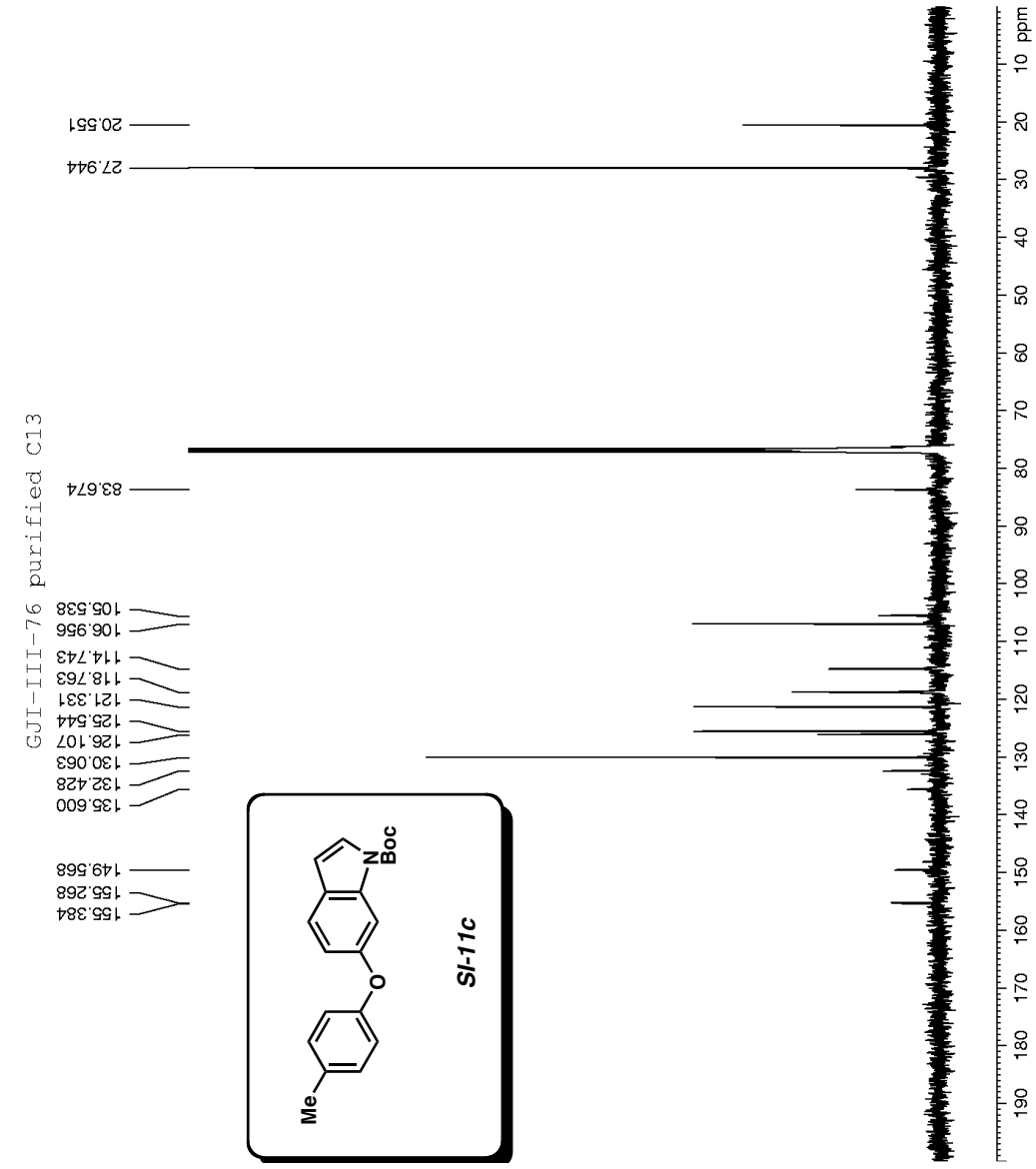

Current Data Parameters
NAME GJI-III-165
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100420
Time 20.51
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 32768
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7578090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-III-165 purified C13
4.419
18.214
25.770
101.663
102.255
114.362
120.813
122.655
123.262
136.513
151.589
TBSO
NH
SI-7
ppm

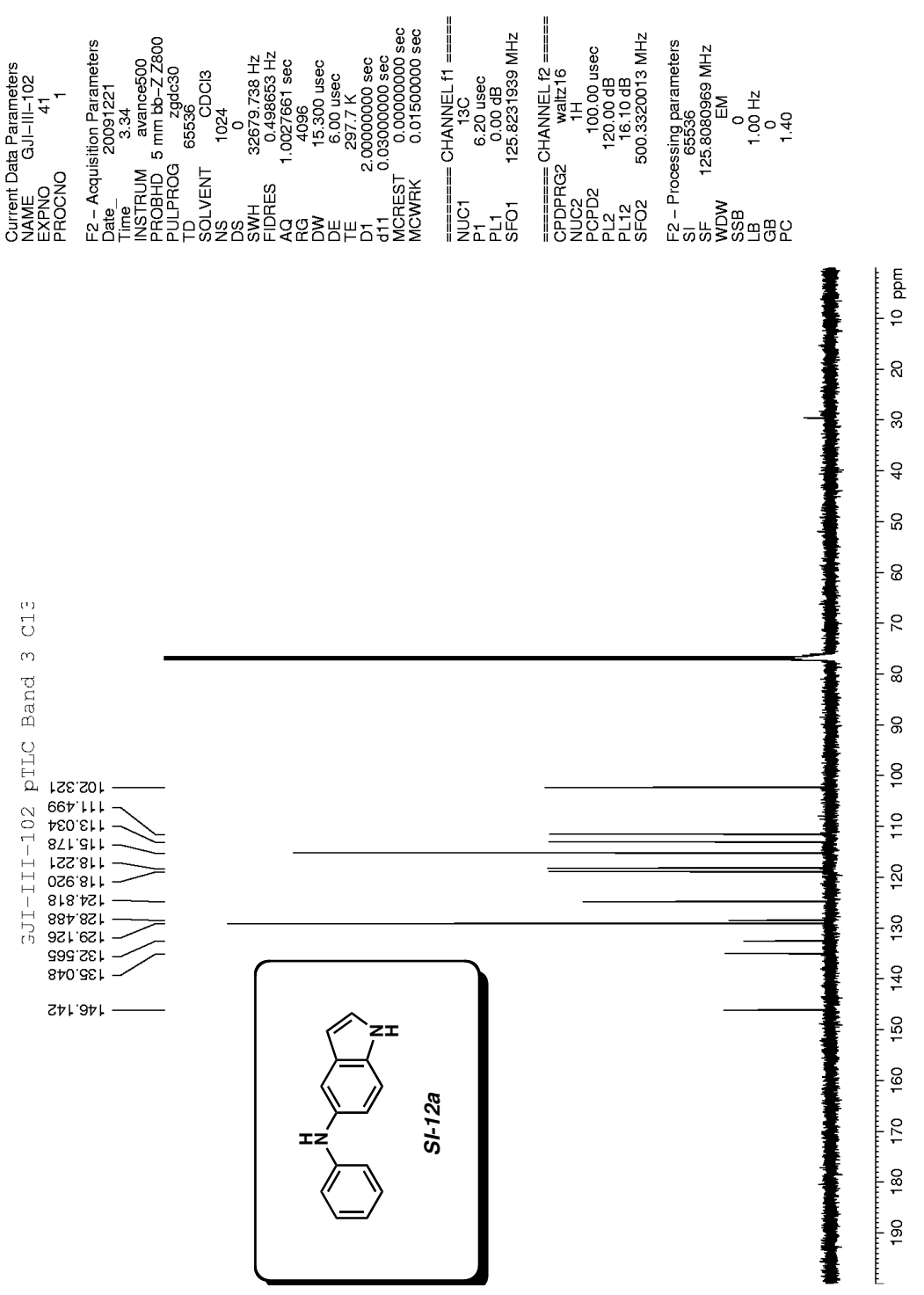

Current Data Parameters
NAME GJI-III-112
EXPNO 21
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091230
Time 19.36
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 2048
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 5792.6
DW 15.300 usec
DE 6.00 usec
TE 297.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-III-112 pTLC Band 3 C13
20.496
102.641
110.549
111.661
115.421
117.505
125.189
128.387
129.870
131.400
132.451
150.626
156.798
N
H
O
Me
SI-10a
10 ppm

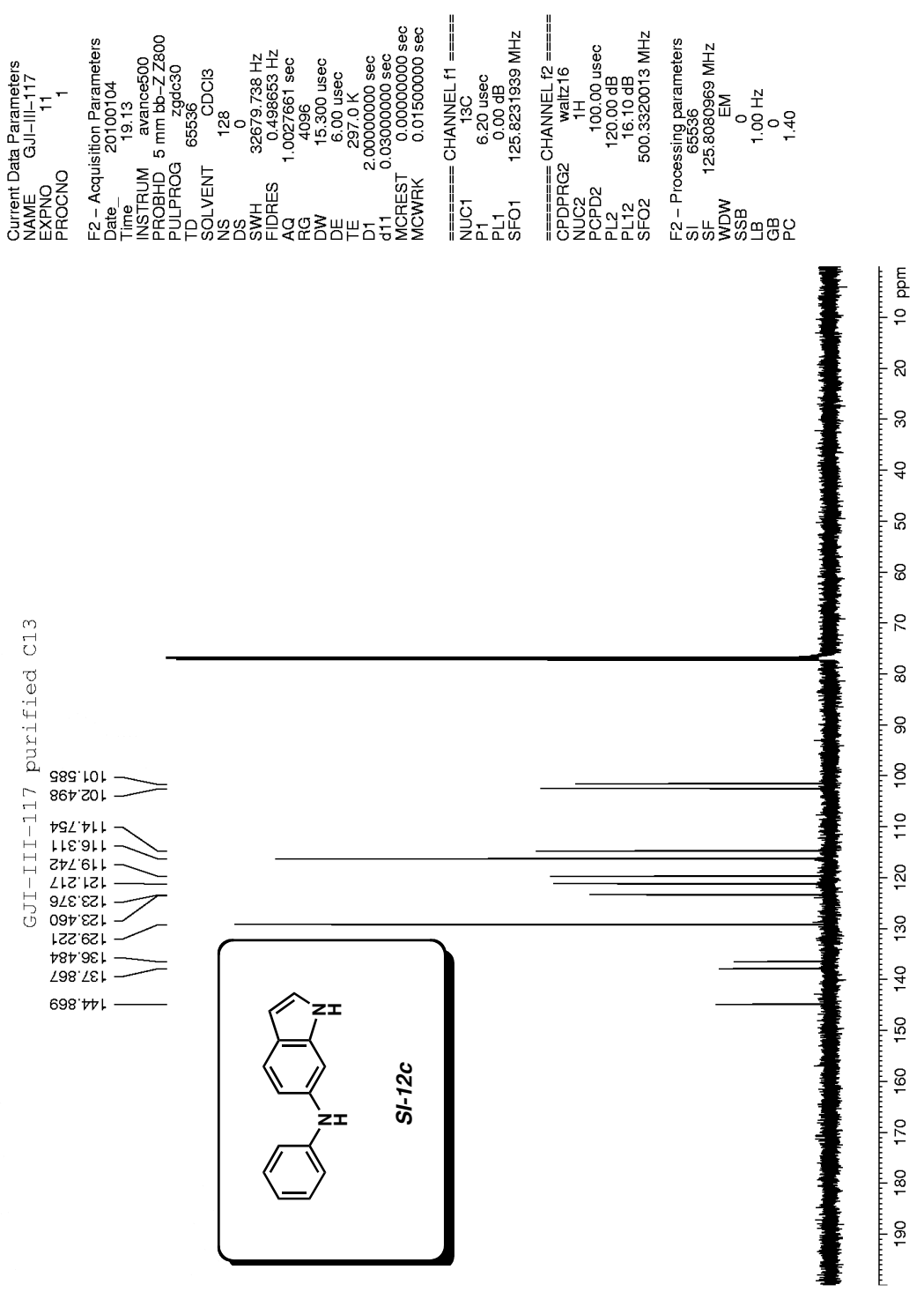

GJI-III-111 purified C13
Current Data Parameters
NAME GJI-III-111
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100102
Time_ 1.20
INSTRUM avance500
PROBHD 5 mm bb–Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 848
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 297.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
20.541
101.250
102.518
113.067
118.093
121.307
123.968
124.015
129.962
131.925
136.137
153.287
156.119
Me
O
N
H
SI-10c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

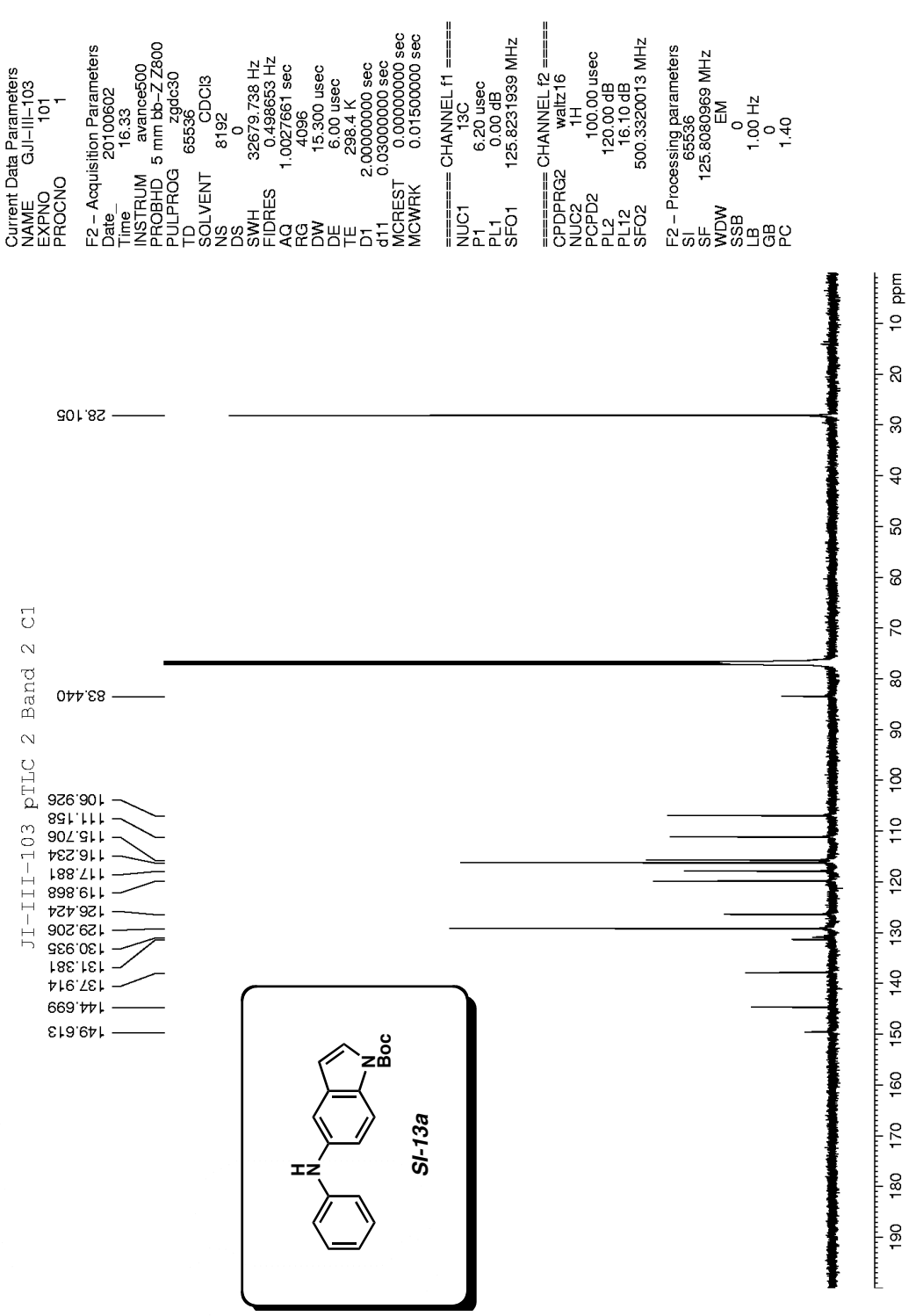

Current Data Parameters
NAME GJI-III-179183
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100507
Time 2.11
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 16384
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 11585.2
DW 15.300 usec
DE 6.00 usec
TE 298.7 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
GJI-III-179 + 183 Band 1 C1
154.641
142.588
132.634
130.276
130.112
127.674
124.113
120.096
118.004
115.815
110.144
102.944
20.553
Me
SI-10d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

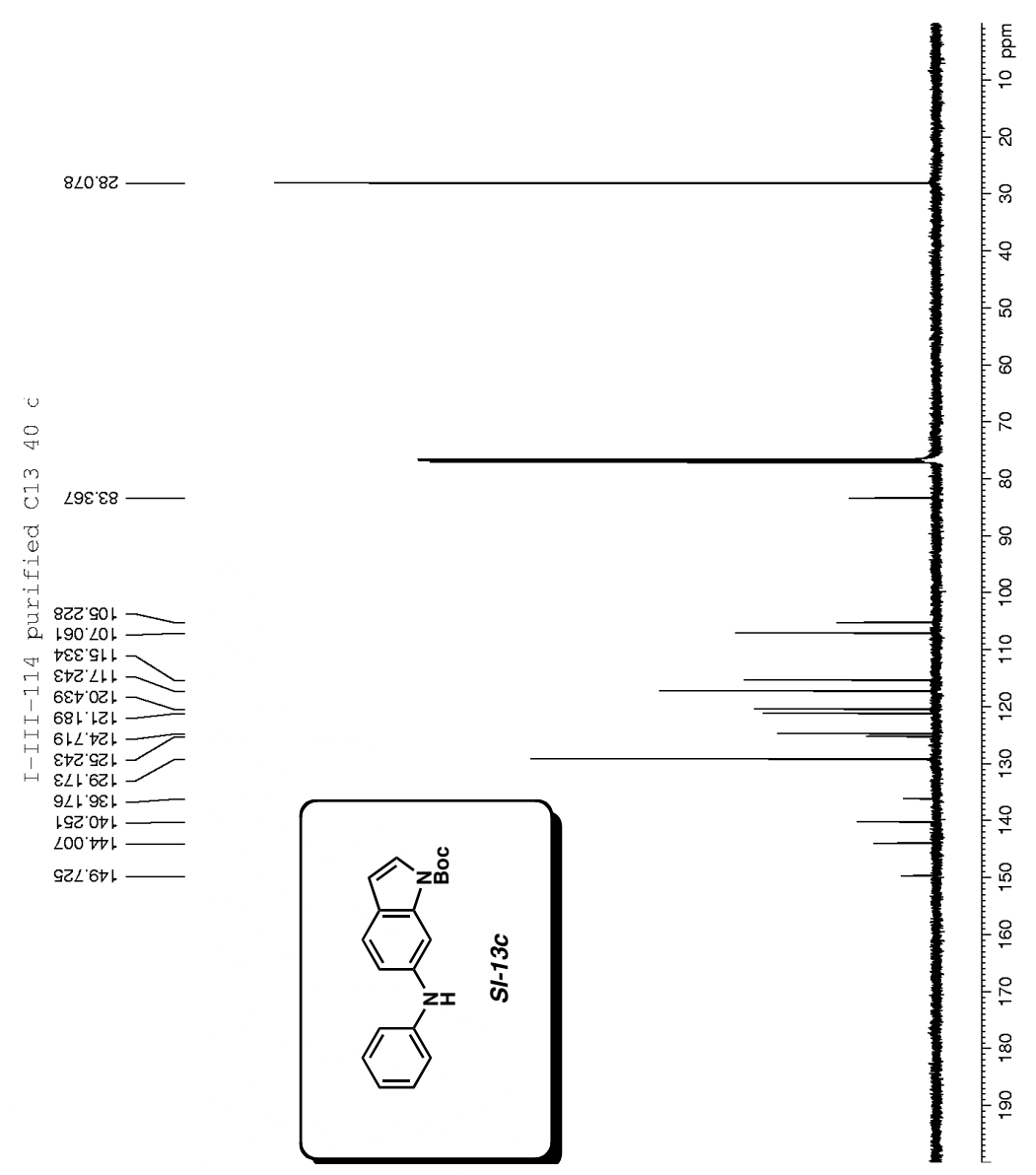

Current Data Parameters
NAME GJI-III-113
EXPNO 41
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091230
Time_ 2.55
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 2048
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 313.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-III-113 sp3 40 deg C13
20.410
28.062
83.521
106.947
110.364
115.878
116.344
118.136
126.730
129.939
131.441
132.012
149.503
153.075
156.007
N
Boc
O
Me
SI-11a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

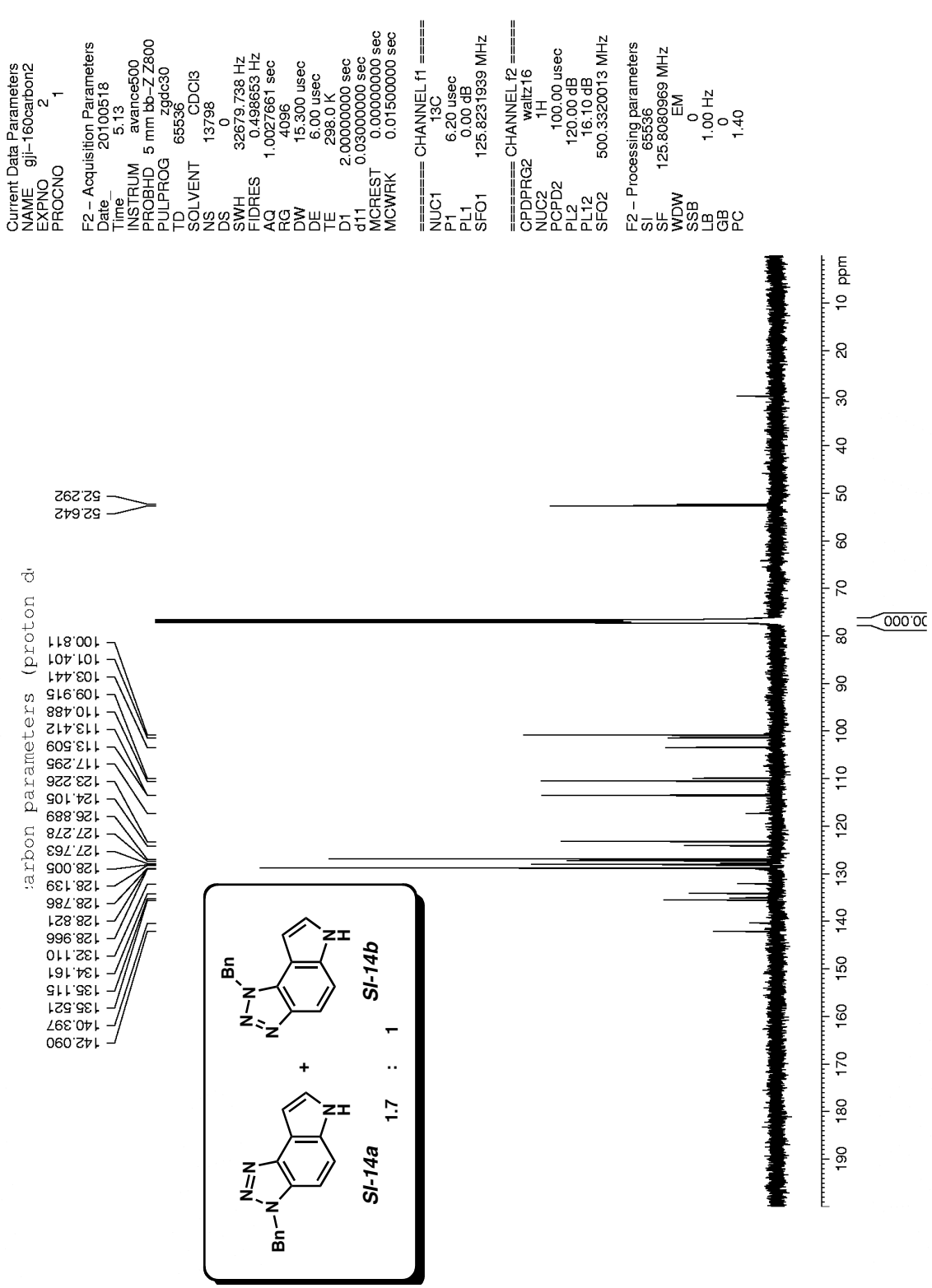

Current Data Parameters
NAME GJI-III-76
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091023
Time_ 18.23
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 1024
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 14596.5
DW 15.300 usec
DE 6.00 usec
TE 296.6 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
GJI-III-76 purified C13
155.384
155.268
149.568
135.600
132.428
130.063
126.107
125.544
121.331
118.763
114.743
106.956
105.538
83.674
27.944
20.551
Me
O
N
Boc
SI-11c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

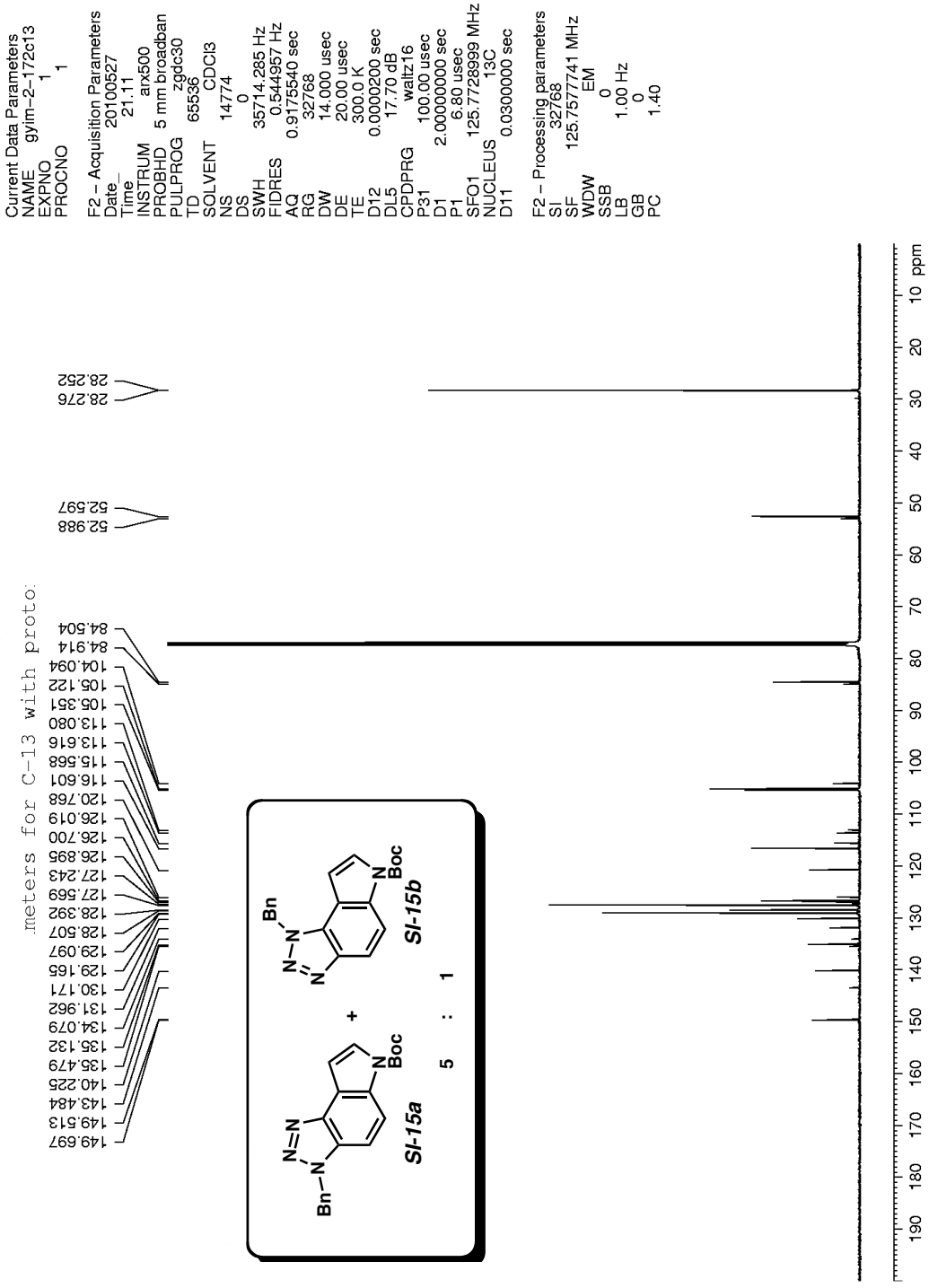

Current Data Parameters
NAME GJI-III-102
EXPNO 41
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091221
Time 3.34
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 1024
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 297.7 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
3JI-III-102 pTLC Band 3 C13
146.142
135.048
132.565
129.126
128.488
124.818
118.920
118.221
115.178
113.034
111.499
102.321
SI-12a
H
N
H
N
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

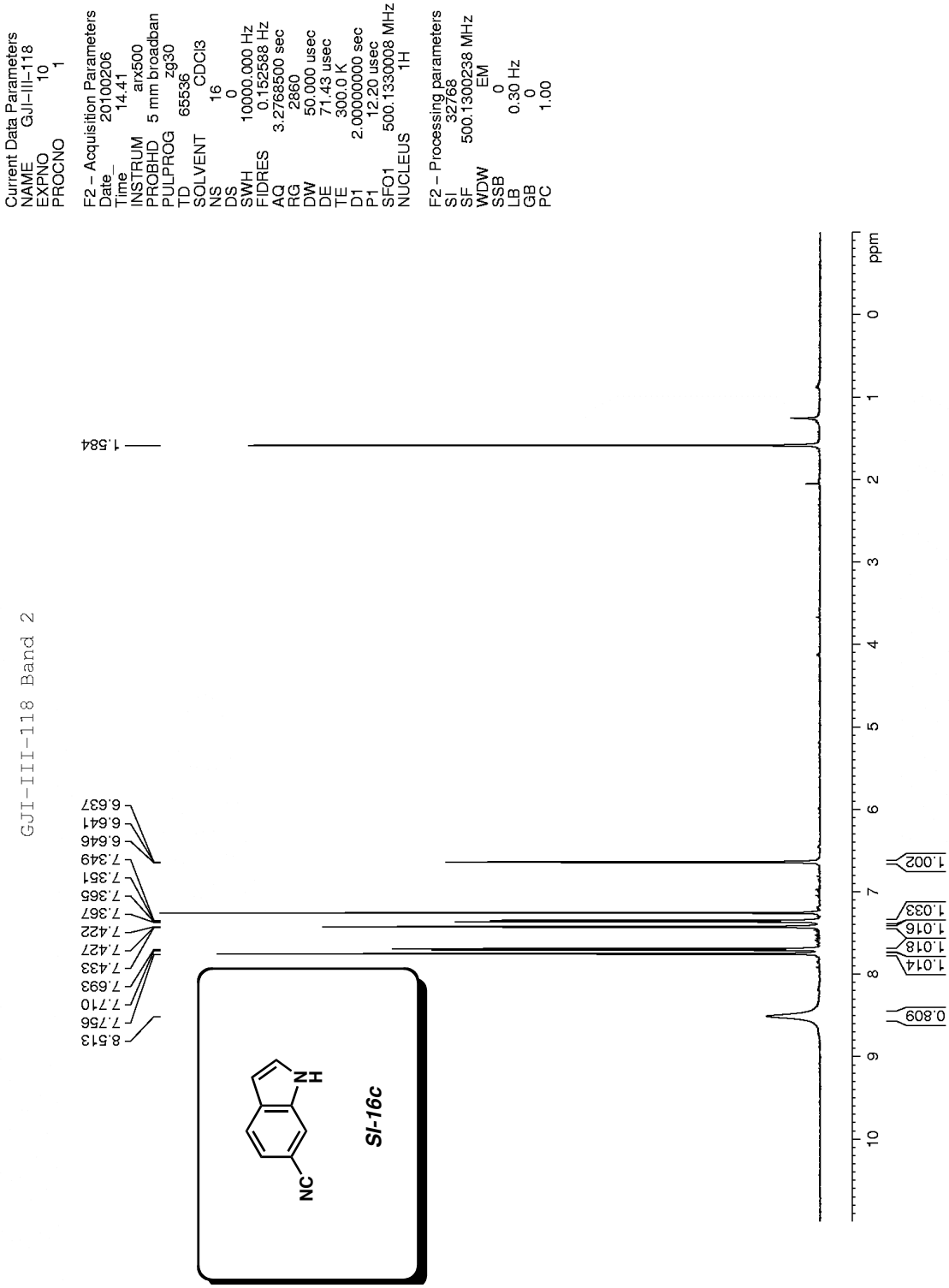

Current Data Parameters
NAME GJI-III-117
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100104
Time 19.13
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-III-117 purified C13
144.869
137.867
136.484
129.221
123.460
123.376
121.217
119.742
116.311
114.754
102.498
101.585
SI-12c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

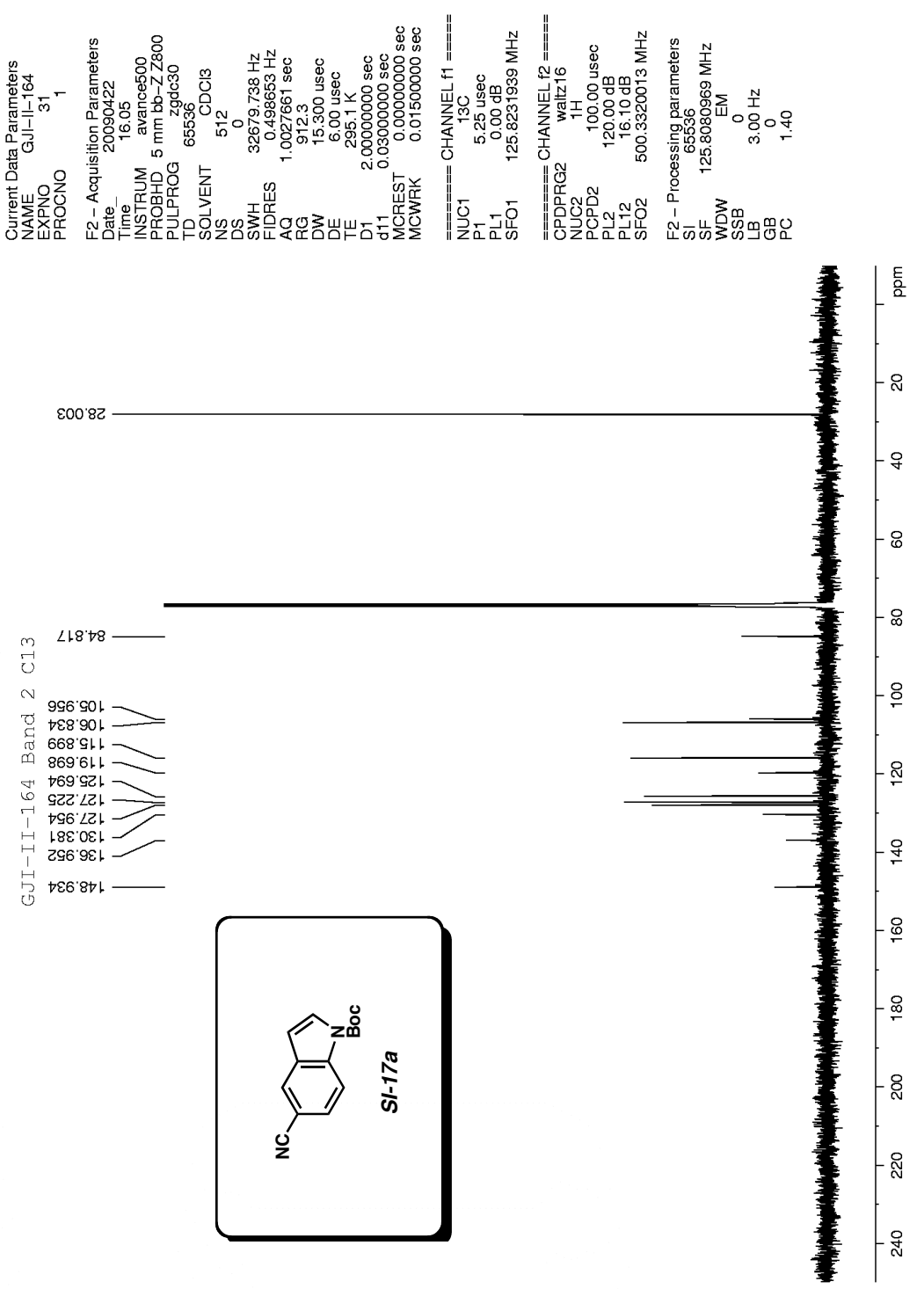

Current Data Parameters
NAME GJI-III-103
EXPNO 101
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100602
Time_ 16.33
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 8192
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 298.4 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
JI-III-103 pTLC 2 Band 2 C1
149.613
144.699
137.914
131.381
130.935
129.206
126.424
119.868
117.881
116.234
115.706
111.158
106.926
83.440
28.105
H
N
N
Boc
SI-13a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

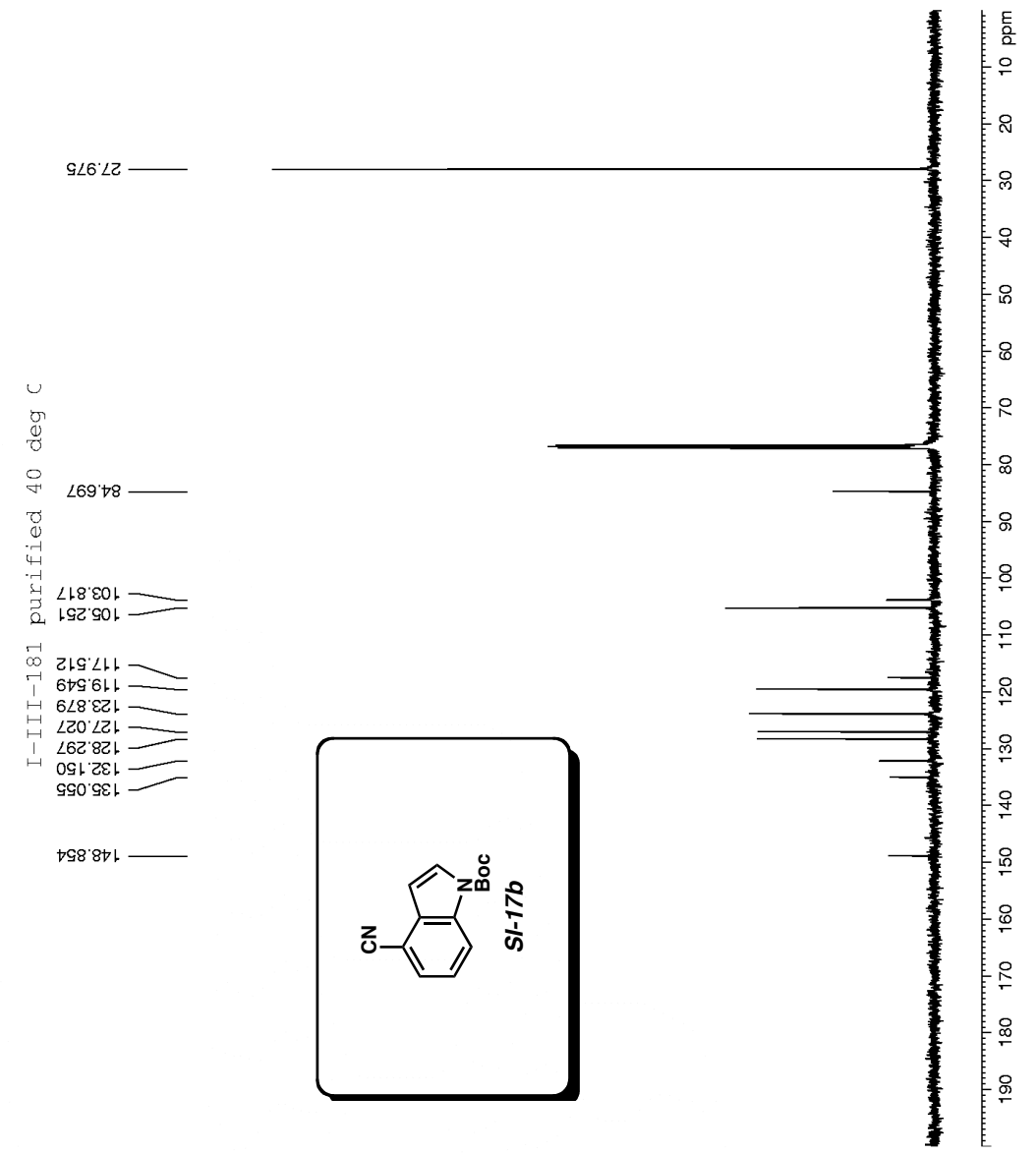

Current Data Parameters
NAME GJI-III-114
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100102
Time_ 2.20
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 8192
DW 15.300 usec
DE 6.00 usec
TE 313.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
I-III-114 purified C13 40 c
149.725
144.007
140.251
136.176
129.173
125.243
124.719
121.189
120.439
117.243
115.334
107.061
105.228
83.367
28.078
N
Boc
N
H
SI-13c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

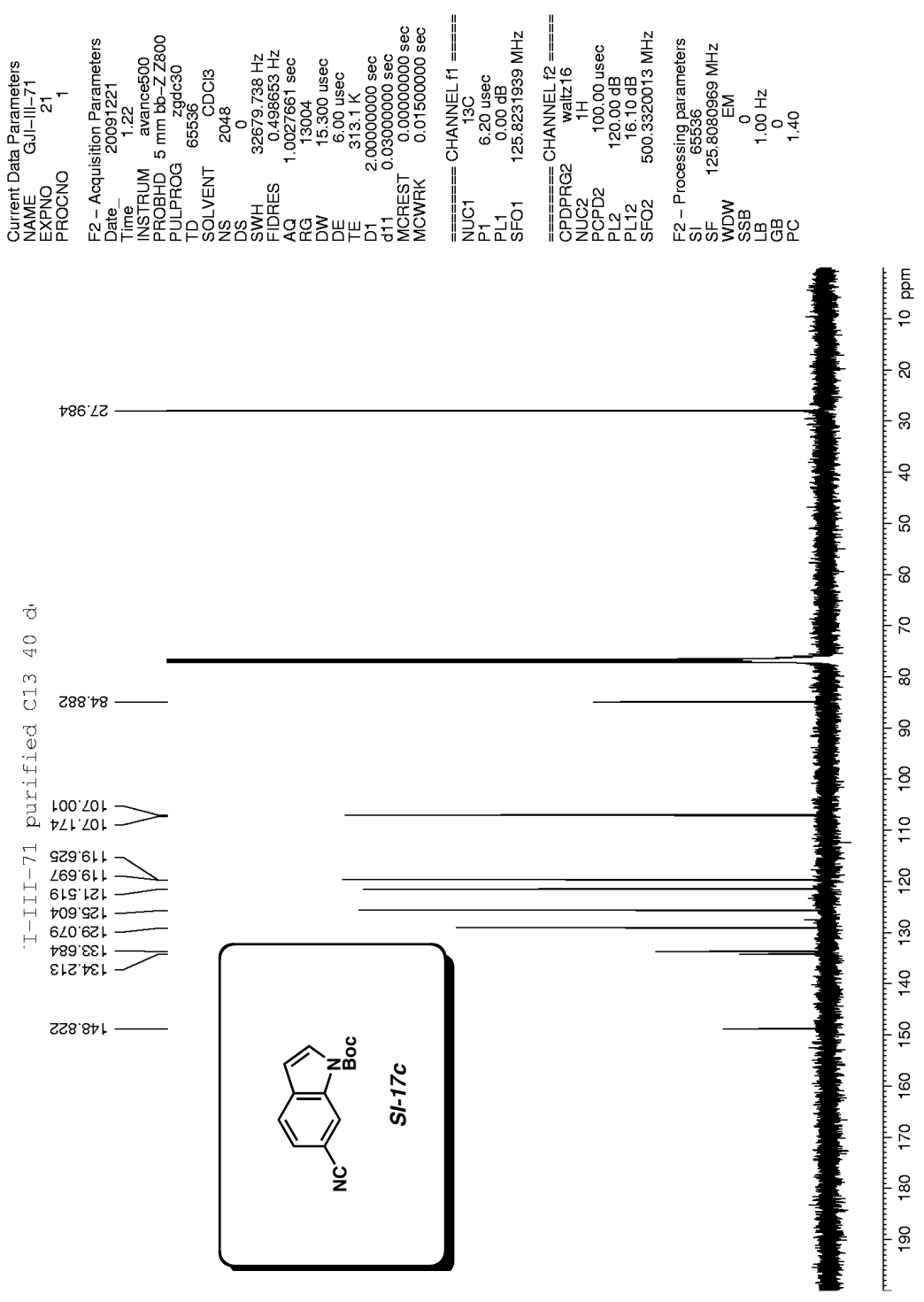

Current Data Parameters
NAME gji-160carbon2
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100518
Time 5.13
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 13798
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
carbon parameters (proton d
142.090
140.397
135.521
135.115
134.161
132.110
128.966
128.821
128.786
128.139
128.005
127.763
127.278
126.889
124.105
123.226
117.295
113.509
113.412
110.488
109.915
103.441
101.401
100.811
52.642
52.292
Bn
N=N
N
Bn
N
H
+
SI-14a
1.7 : 1
SI-14b
10 ppm

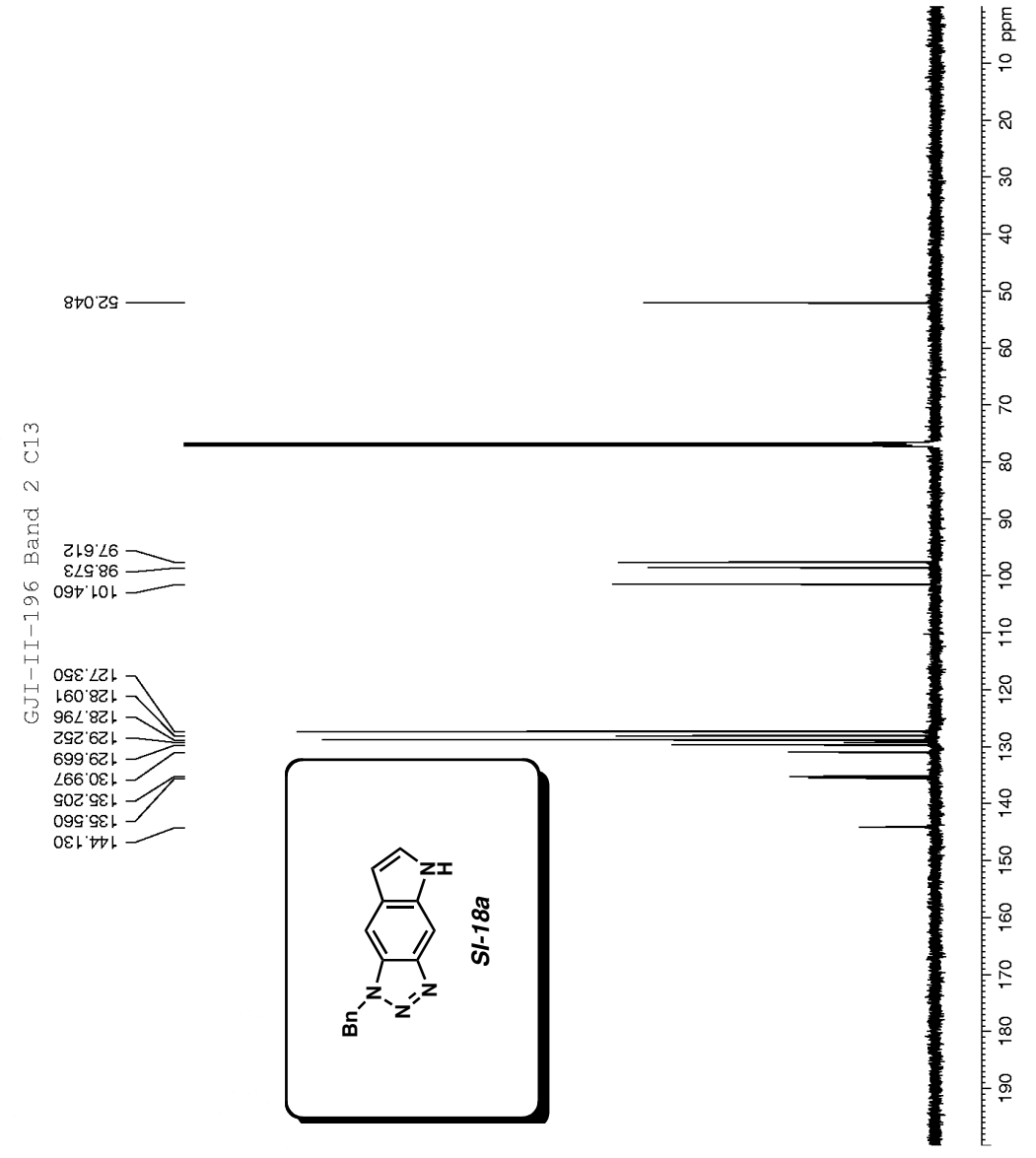

Current Data Parameters
NAME gyim-2-172c13
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100527
Time 21.11
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 14774
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 32768
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7577741 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
meters for C-13 with proto
28.252
28.276
52.597
52.988
84.504
84.914
104.094
105.122
105.351
113.080
113.616
115.568
116.601
120.768
126.019
126.700
126.895
127.243
127.669
128.392
128.507
129.097
129.165
130.171
131.962
134.079
135.132
135.479
140.225
143.484
149.513
149.697
Bn
N=N
N
Boc
SI-15a
+
SI-15b
5 : 1
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

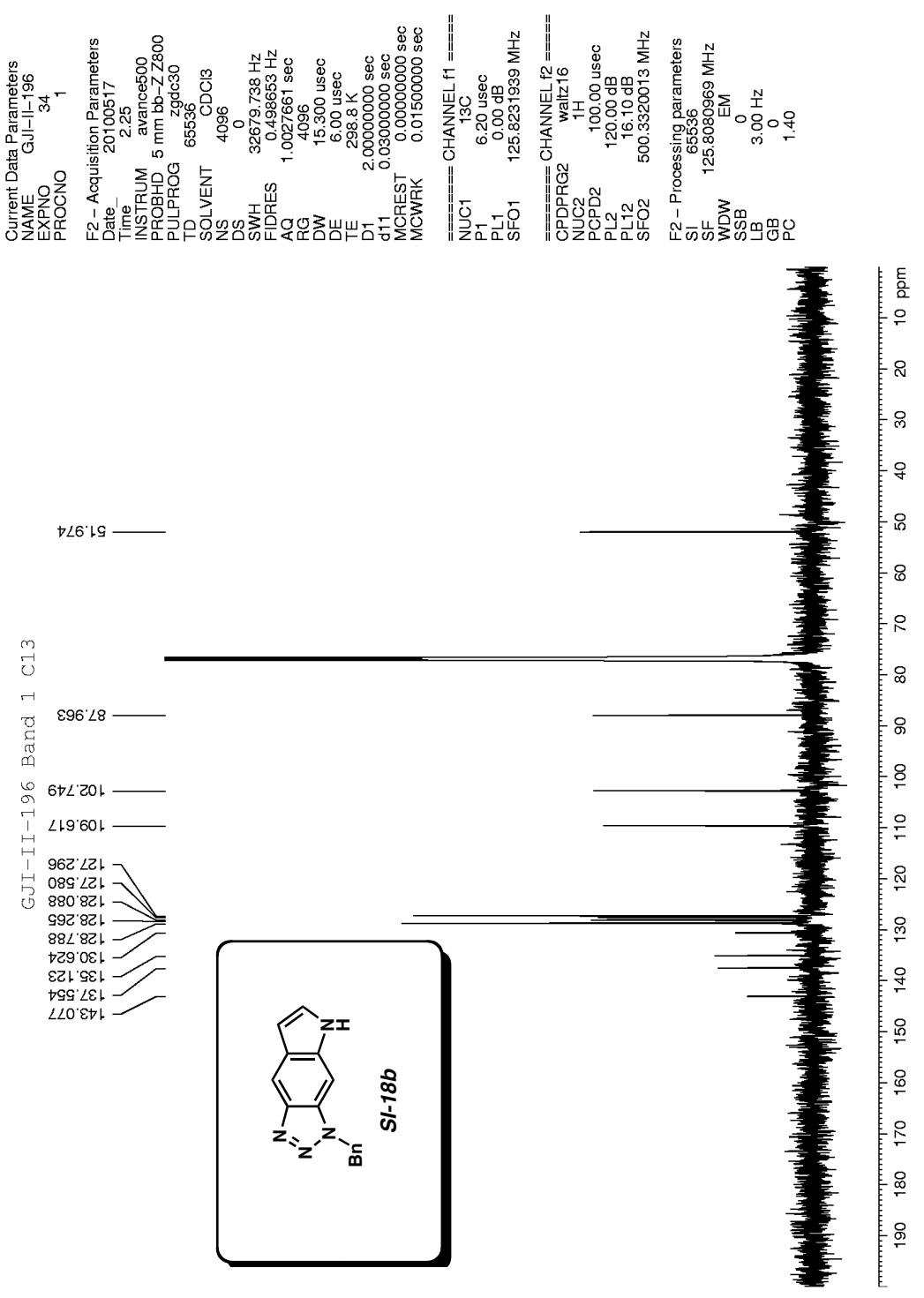

Current Data Parameters
NAME GJI-III-118
EXPNO 10
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100206
Time 14.41
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 2860
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 12.20 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300238 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
GJI-III-118 Band 2
1.584
6.637
6.641
6.646
7.349
7.351
7.365
7.367
7.422
7.427
7.433
7.693
7.710
7.756
8.513
1.002
1.033
1.016
1.018
1.014
0.809
ppm
NC
NH
SI-16c

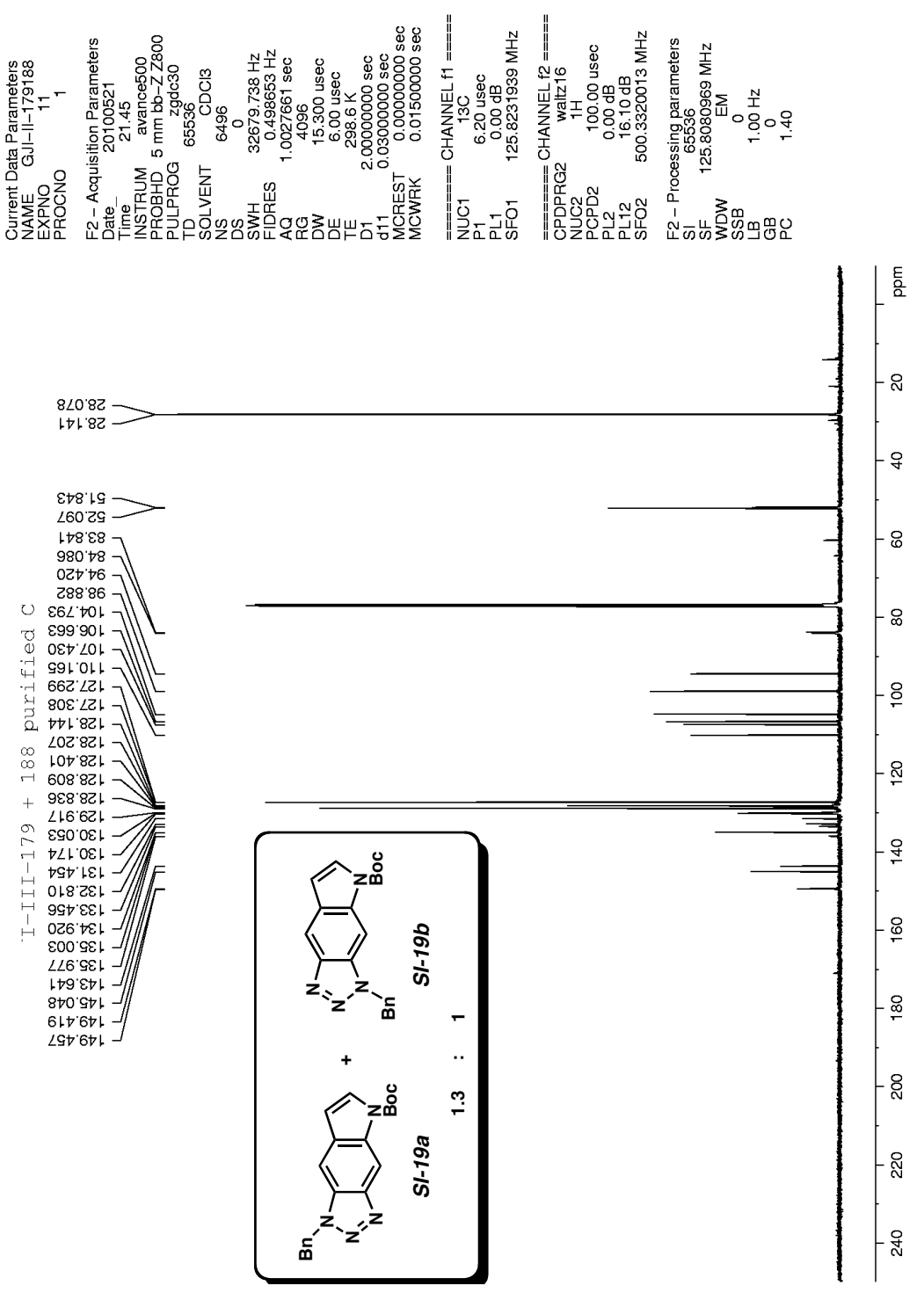

Current Data Parameters
NAME GJI-II-164
EXPNO 31
PROCNO 1
F2 – Acquisition Parameters
Date_ 20090422
Time 16.05
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 912.3
DW 15.300 usec
DE 6.00 usec
TE 295.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
GJI-II-164 Band 2 C13
148.934
136.952
130.381
127.954
127.225
125.694
119.698
115.899
106.834
105.956
84.817
28.003
NC
N
Boc
SI-17a
ppm

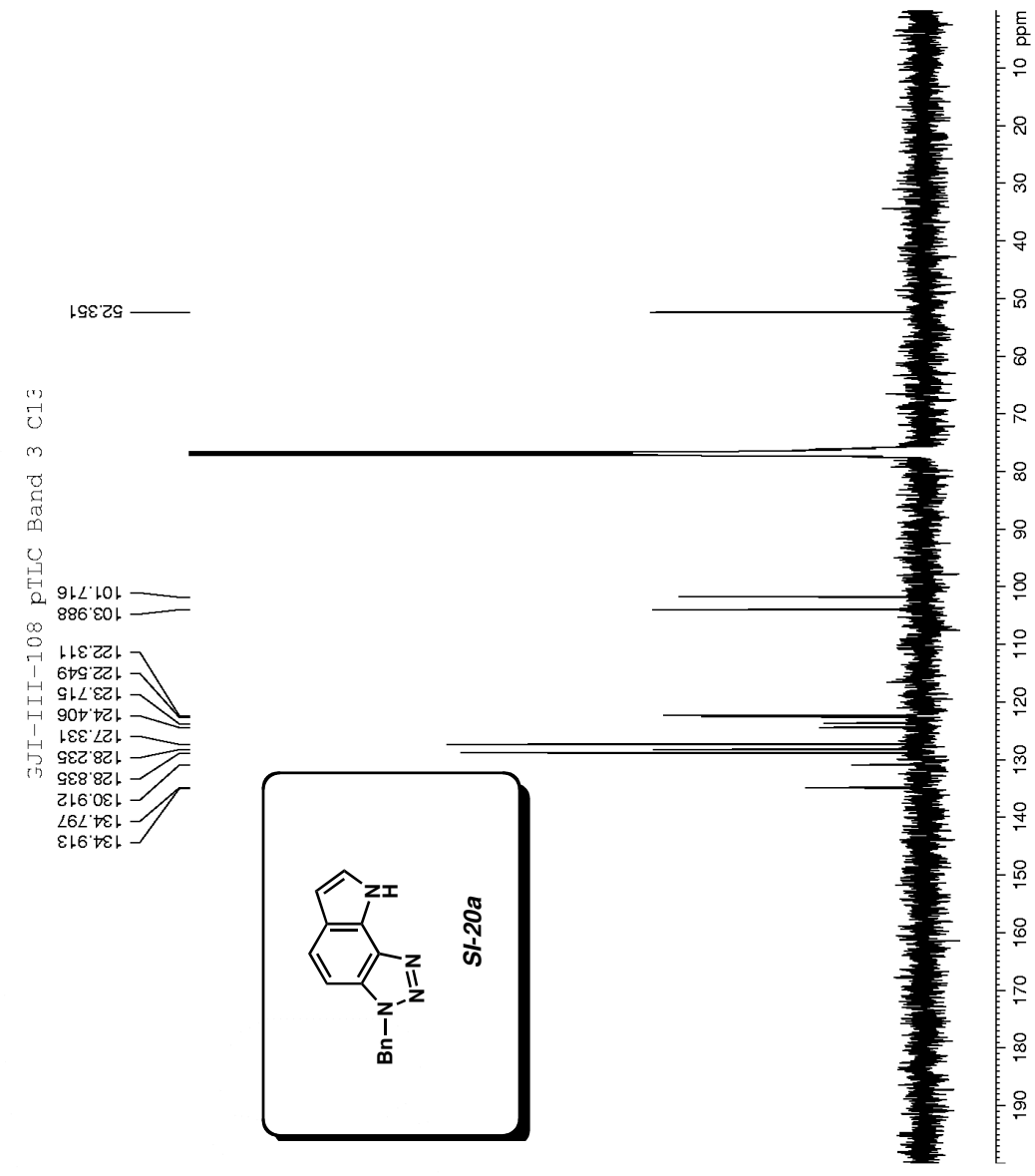

Current Data Parameters
NAME GJI-III-181
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100430
Time 1.49
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 13004
DW 15.300 usec
DE 6.00 usec
TE 312.3 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
I-III-181 purified 40 deg C
148.854
135.055
132.150
128.297
127.027
123.879
119.549
117.512
105.251
103.817
84.697
27.975
CN
N
Boc
SI-17b
ppm

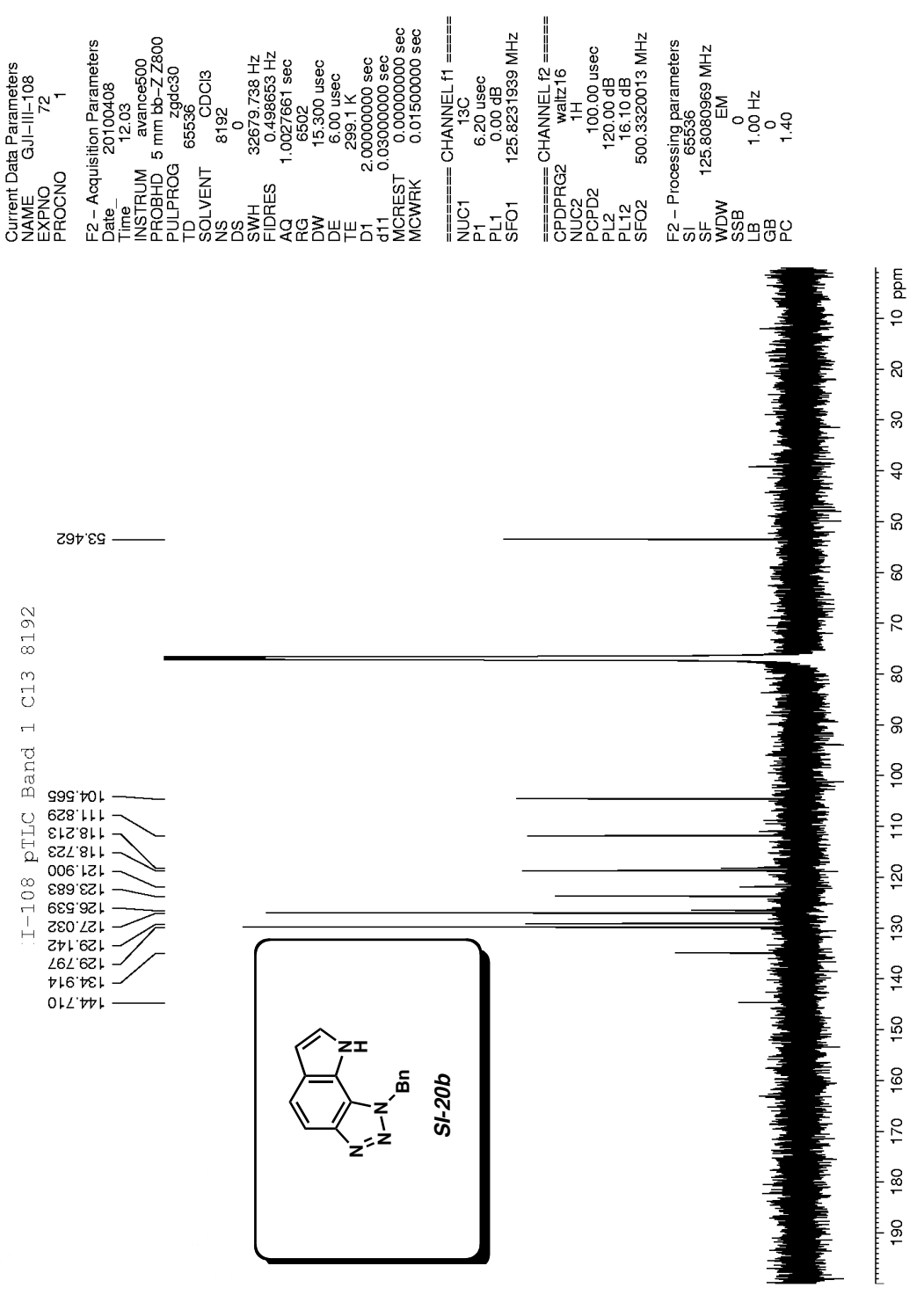

Current Data Parameters
NAME GJI-III-71
EXPNO 21
PROCNO 1
F2 – Acquisition Parameters
Date_ 20091221
Time_ 1.22
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 2048
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 13004
DW 15.300 usec
DE 6.00 usec
TE 313.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
I-III-71 purified C13 40 d
148.822
134.213
133.684
129.079
125.604
121.519
119.697
119.625
107.174
107.001
84.882
27.984
N
Boc
NC
SI-17c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

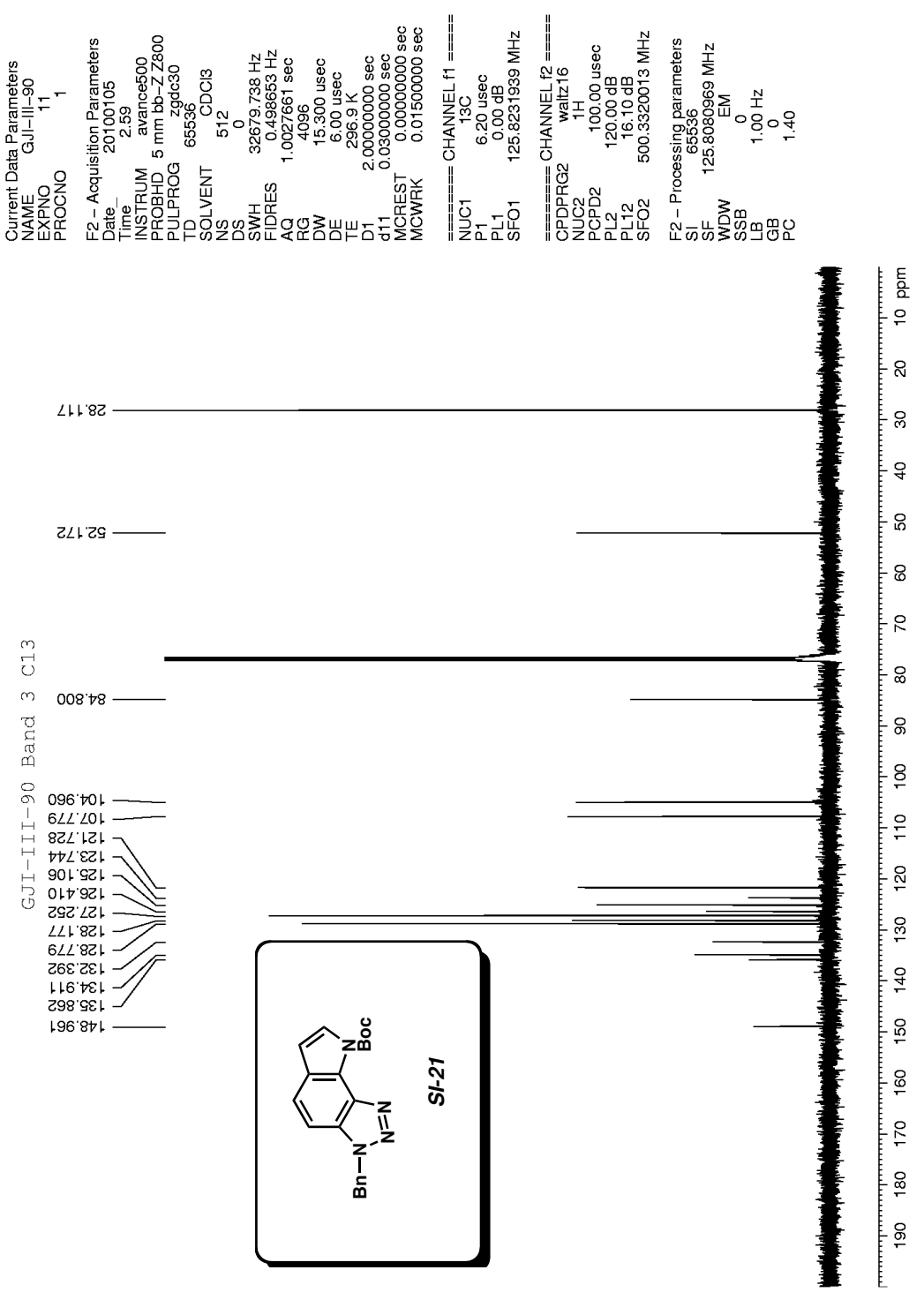

Current Data Parameters
NAME GJI-II-196
EXPNO 21
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100514
Time_ 21.32
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 1024
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3251
DW 15.300 usec
DE 6.00 usec
TE 299.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-II-196 Band 2 C13
52.048
97.612
98.573
101.460
127.350
128.091
128.796
129.252
129.669
130.997
135.205
135.560
144.130
Bn
N
N
N
H
SI-18a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Current Data Parameters
NAME GJI-II-196
EXPNO 34
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100517
Time 2.25
INSTRUM avance500
PROBHD 5 mm bb–Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 4096
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 298.8 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
GJI-II-196 Band 1 C13
51.974
87.963
102.749
109.617
127.296
127.580
128.088
128.265
128.788
130.624
135.123
137.554
143.077
SI-18b
Bn
N
N
N
H
ppm


Current Data Parameters
NAME GJI-II-179188
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100521
Time 21.45
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 6496
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 298.6 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
I-III-179 + 188 purified C
28.078
28.141
51.843
52.097
83.841
84.086
94.420
98.882
104.793
106.663
107.430
110.165
127.299
127.308
128.144
128.207
128.401
128.809
128.836
129.917
130.053
130.174
131.454
132.810
133.456
134.920
135.003
135.977
143.641
145.048
149.419
149.457
Boc
N
Bn
N
N
N
SI-19a
+
SI-19b
1.3 : 1
ppm


Current Data Parameters
NAME GJI-III-108pTLC
EXPNO 41
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100404
Time 19.12
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 1024
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 298.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
GJI-III-108 pTLC Band 3 C13
52.351
101.716
103.988
122.311
122.549
123.715
124.406
127.331
128.235
128.835
130.912
134.797
134.913
Bn
N
N=N
H
SI-20a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Current Data Parameters
NAME GJI-III-108
EXPNO 72
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100408
Time 12.03
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 8192
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 6502
DW 15.300 usec
DE 6.00 usec
TE 299.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
I-108 pTLC Band 1 C13 8192
144.710
134.914
129.797
129.142
127.032
126.539
123.683
121.900
118.723
118.213
111.829
104.565
53.462
N
H
N
N
N
Bn
SI-20b
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Current Data Parameters
NAME GJI-III-90
EXPNO 11
PROCNO 1
F2 – Acquisition Parameters
Date_ 20100105
Time 2.59
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 296.9 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 0.00 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz
F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
GJI-III-90 Band 3 C13
28.117
52.172
84.800
104.960
107.779
121.728
123.744
125.106
126.410
127.252
128.177
128.779
132.392
134.911
135.862
148.961
Boc
N
Bn—N
N=N
SI-21
ppm

References:

[1] Bronner, S. M.; Bahnck, K. B.; Garg, N. K. *Org. Lett.* **2009**, *11*, 1007–1010.

[2] Buchgraber, P.; Domostoj, M. M.; Scheiper, B.; Wirtz, C.; Mynott, R.; Rust, J.; Fürstner, A. *Tetrahedron* **2009**, *65*, 6519–6534.

[3] Cheong, P. H.-Y.; Paton, R. S.; Bronner, S. M.; Im. G.-Y. J.; Garg, N. K.; Houk, K. N. *J. Am. Chem. Soc.* **2010**, *132*, 1267–1269.

[4] a) Stadlwieser, J. F.; Dambaur, M. E. *Helv. Chim. Acta* **2006**, *89*, 936–946. b) Gwaltney, S. L.; Imade, H. M.; Barr, K. J.; Li, Q.; Gehrke, L.; Credo, R. B.; Warner, R. B.; Lee, J. Y.; Kovar, P.; Wang, J.; Nukkala, M. A.; *Bioorg. Med. Chem. Lett.* **2001**, *11*, 871–874.

[5] Gwaltney, S. L.; Imade, H. M.; Barr, K. J.; Li, Q.; Gehrke, L.; Credo, R. B.; Warner, R. B.; Lee, J. Y.; Kovar, P.; Wang, J.; Nukkala, M. A. *Bioorg. Med. Chem. Lett.* **2001**, *11*, 871–874.

[6] Kauch, M.; Hoppe, D. *Can. J. Chem.* **2001**, *79*, 1736–1746.

[7] Kauch, M.; Snieckus, V.; Hoppe, D. *J. Org. Chem.* **2005**, *70*, 7149–7158.

[8] This is an improved procedure from what our group previously reported. See ref. 3.

[9] On larger scales, cooling to –78 °C can cause the carbamate to crash out of the ethereal solution, in which case, small portions of THF should be added to re-solubilize the material prior to the introduction of *n*-BuLi.

[10] The exothermic additions of *n*-BuLi and TMSCl should be executed at such a rate to maintain an internal reaction temperature below –70 °C to avoid C2 lithiation.

[11] Igarashi, Y.; Yanagisawa, E.; Ohshima, T.; Takeda, S.; Aburada, M.; Miyamoto, K. *Chem. Pharm. Bull.* **2007**, *55*, 328–333.

[12] Jiang, J.; Gribble, G. W. *Tetrahedron Lett.* **2002**, *43*, 4115–4117.

[13] Alvarez, S. G.; Alzarez, M. T. *Synthesis* **1997**, 413–414.