Supporting Information:

Nickel-Catalyzed Suzuki–Miyaura Coupling of Aliphatic Amides

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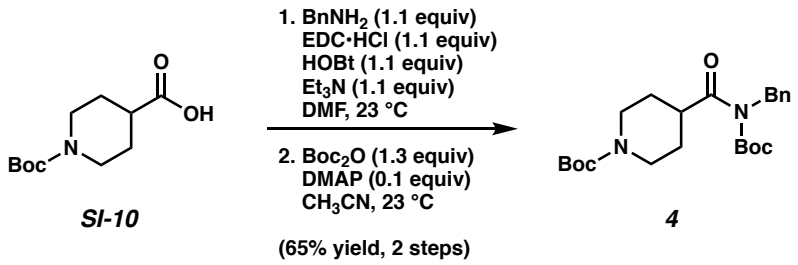
Materials and Methods. Unless stated otherwise, reactions were conducted in flame-dried glassware under an atmosphere of nitrogen or argon and commercially obtained reagents were used as received. Non-commercially available substrates were synthesized following protocols specified in Section A in the Experimental Procedures. Prior to use, toluene was purified by distillation and taken through five freeze-pump-thaw cycles, and phenylhydrazine (**46**) was passed over a plug of basic alumina. Benzylamine was obtained from Sigma–Aldrich. Boronate esters **5**, **SI-1**, **SI-2**, **SI-3**, **SI-4**, **SI-5**, **SI-6**, **37**, **SI-7**, **SI-8**, **SI-9**, and **44** and carboxylic acids **SI-10**, **SI-11**, **SI-12**, **SI-13**, **SI-14**, **SI-15** were obtained from Combi-Blocks. Boronate ester **SI-16**[1] was prepared according to literature procedures. $Ni(cod)_2$, SIPr (**7**), terpyridine (**8**), ICy•HBF_4 (**9**), and Benz-ICy•HCl (**10**) were obtained from Strem Chemicals. K_3PO_4 was obtained from Acros. Reaction temperatures were controlled using an IKAmag temperature modulator, and unless stated otherwise, reactions were performed at room temperature (approximately 23 °C). Thin-layer chromatography (TLC) was conducted with EMD gel 60 F254 pre-coated plates (0.25 mm for analytical chromatography and 0.50 mm for preparative chromatography) and visualized using a combination of UV, anisaldehyde, iodine, and potassium permanganate staining techniques. Silicycle Siliaflash P60 (particle size 0.040–0.063 mm) was used for flash column chromatography. ^{1}H NMR spectra were recorded on Bruker spectrometers (at 300, 400 and 500 MHz) and are reported relative to residual solvent signals. Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz), integration. Data for ^{13}C NMR are reported in terms of chemical shift (at 75 and 125 MHz). IR spectra were recorded on a Perkin-Elmer UATR Two FT-IR spectrometer and are reported in terms of frequency absorption (cm^{-1}). DART-MS spectra were collected on a Thermo Exactive Plus MSD (Thermo Scientific) equipped with an ID-CUBE ion source and a Vapur Interface (IonSense Inc.). Both the source and MSD were controlled by Excalibur software v. 3.0. The analyte was spotted onto OpenSpot sampling cards (IonSense Inc.) using $CHCl_3$ as the solvent. Ionization was accomplished using UHP He plasma with no additional ionization agents. The mass calibration was carried out using Pierce LTQ Velos ESI (+) and (–) Ion calibration solutions (Thermo Fisher Scientific). Determination of enantiopurity was carried out using either a Mettler Toledo SFC (supercritical fluid chromatography) or Agilent HPLC using a Daicel ChiralPak OJ-H column. Optical rotations were measured with a Rudolph Autopol III Automatic Polarimeter.

Experimental Procedures

A. Syntheses of Amide Substrates

Representative Procedure for the synthesis of amide substrates from Tables S1 and S2 and Figures 2, 3, 5, and 6 (synthesis of amide 4 is used as an example).



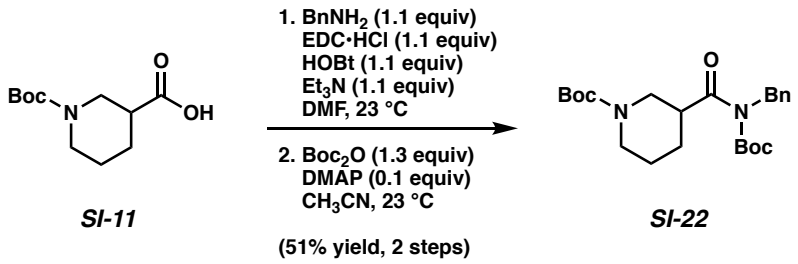
To a mixture of carboxylic acid **SI-10** (3.00 g, 13.1 mmol, 1.0 equiv), EDC•HCl (2.76 g, 14.4 mmol, 1.1 equiv), HOBt (1.94 g, 14.4 mmol, 1.1 equiv), triethylamine (1.99 mL, 14.4 mmol, 1.1 equiv) and DMF (131 mL, 0.1 M) was added benzylamine (1.57 mL, 14.4 mmol, 1.1 equiv). The resulting mixture was stirred at 23 °C for 16 h, and then diluted with deionized water (250 mL) and transferred to a separatory funnel with EtOAc (150 mL) and brine (50 mL). The aqueous layer was extracted with EtOAc (3 x 150 mL), then the organic layers were combined and washed with deionized water (3 x 125 mL), dried over Na_2SO_4, and evaporated under reduced pressure. The resulting crude solid material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (148 mg, 1.21 mmol, 0.1 equiv) followed by acetonitrile (60.0 mL, 0.2 M). Boc_2O (3.43 g, 15.7 mmol, 1.3 equiv) was added in one portion and the reaction vessel was flushed with N_2, then the reaction mixture was allowed to stir at 23 °C for 16 h. The reaction was quenched by addition of saturated aqueous $NaHCO_3$ (200 mL), transferred to a separatory funnel with EtOAc (200 mL) and H_2O (200 mL), and extracted with EtOAc (3 x 100 mL). The organic layers were combined, dried over Na_2SO_4, and evaporated under reduced pressure. The resulting crude residue was purified by flash chromatography (9:1 Hexanes:EtOAc) to yield amide **4** (3.59 g, 65% yield, over two steps) as white solid. Amide **4**: mp: 83–85 ºC; R_f 0.39 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.31–7.26 (m, 2H), 7.24–7.18 (m, 3H), 4.86 (s, 2H), 4.12 (br s, 2H), 3.59 (tt, *J* = 11.2, 3.6, 1H), 2.88–2.70 (m, 2H), 1.91–1.79 (m, 2H), 1.65 (qd, *J* = 12.2, 4.0, 2H), 1.45

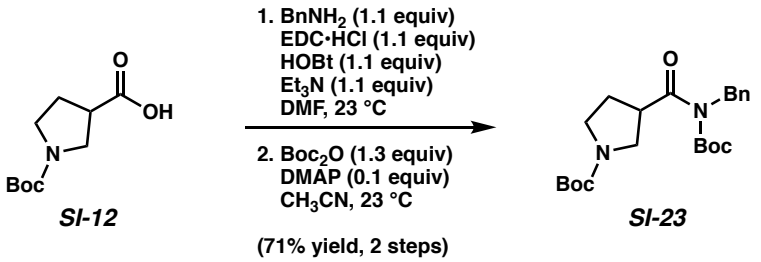
(s, 9H), 1.40 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 178.2, 154.8, 153.1, 138.4, 128.5, 127.5, 127.2, 83.5, 79.6, 47.8, 43.8, 43.0, 29.0, 28.6, 28.0; IR (film): 2976, 2932, 2861, 1731, 1689 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{23}H_{35}N_2O_5$, 419.25405; found 419.25413.

Note: Supporting information for the syntheses of amides shown in Figures 4 and 5 have previously been reported: **SI-17**,[2] **SI-18**,[2] **SI-19**,[2] **SI-20**,[2] **SI-21**,[2] **38**,[3] **41**,[4] **rac-41**,[4] and **SI-25**.[4] Syntheses for the remaining substrates shown in Figures 3, 4, 6, and 7 are as follows:

Any modifications of the conditions shown in the representative procedure above are specified in the following schemes.

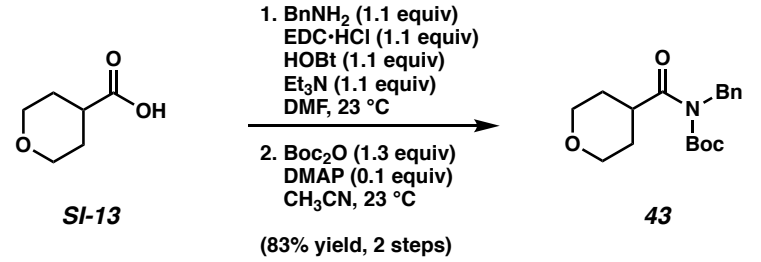


Amide SI-22. Purification by flash chromatography (9:1 Hexanes:EtOAc) generated amide **SI-22** (51% yield, over two steps) as a white solid. Amide **SI-22**: mp: 73–75 ºC; R_f 0.43 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.31–7.26 (m, 2H), 7.24–7.19 (m, 3H), 4.92–4.78 (m, 2H), 4.26–3.94 (m, 2H), 3.51 (tt, *J* = 10.6, 3.6, 1H), 2.99 (dd, *J* = 12.5, 11.0, 1H), 2.75 (br s, 1H), 2.10 (br s, 1H), 1.74–1.68 (m, 1H), 1.62–1.48 (m, 2H), 1.45 (s, 9H), 1.40 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$, 15 of 17 observed): δ 177.2, 154.8, 152.9, 138.3, 128.5, 127.5, 127.3, 83.6, 79.7, 47.7, 43.5, 28.7, 28.6, 28.0, 24.7; IR (film): 2977, 2935, 2862, 1732, 1687 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{23}H_{35}N_2O_5$, 419.25405; found 419.25304.

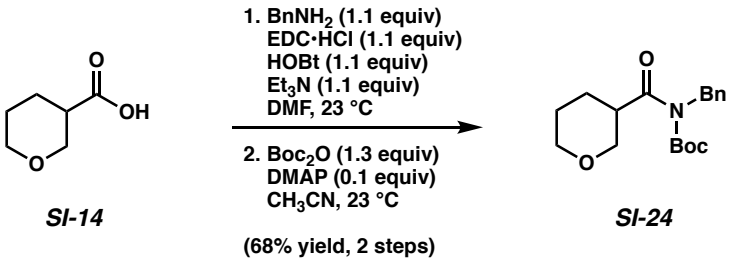


Amide SI-23. Purification by flash chromatography (9:1 Hexanes:EtOAc) generated amide **SI-23** (71% yield, over two steps) as a colorless oil. Amide **SI-23**: R_f 0.47 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32–7.27 (m, 2H), 7.25–7.19 (m, 3H), 4.88 (s, 2H), 4.08 (quint, J = 7.1, 1H), 3.67 (br s, 1H), 3.56 (dd, J = 10.8, 6.3, 1H), 3.53–3.45 (m, 1H), 3.43–3.33 (m, 1H), 2.15 (br s, 2H), 1.46 (s, 9H), 1.42 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$, 15 of 16 observed): δ 176.2, 154.6, 153.2, 138.3, 128.5, 127.6, 127.4, 83.8, 79.4, 49.3, 48.0, 45.6, 29.4, 28.7, 28.1; IR (film): 2979, 2887, 1731, 1693, 1366, 1143 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{22}H_{33}N_2O_5$, 405.23840; found 405.23794.

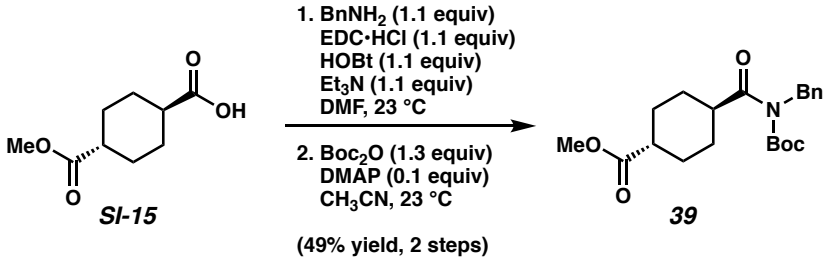
*Note: ^{1}H and ^{13}C NMR spectra of amide **SI-23** were obtained at 57 °C.*



Amide 43. Purification by flash chromatography (14:1 Hexanes:EtOAc) generated amide **43** (83% yield, over two steps) as a white solid. Amide **43**: mp: 52–54 ºC; R_f 0.59 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32–7.27 (m, 2H), 7.25–7.19 (m, 3H), 4.87 (s, 2H), 4.03–3.97 (m, 2H), 3.74–3.65 (m, 1H), 3.48 (td, J = 11.5, 2.4, 2H), 1.91–1.75 (m, 4H), 1.40 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 178.1, 153.1, 138.4, 128.5, 127.5, 127.3, 83.4, 67.5, 47.8, 42.2, 29.7, 28.0; IR (film): 2962, 2842, 1728, 1688, 1366, 1143 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{18}H_{26}NO_4$, 320.18563; found 320.18538.

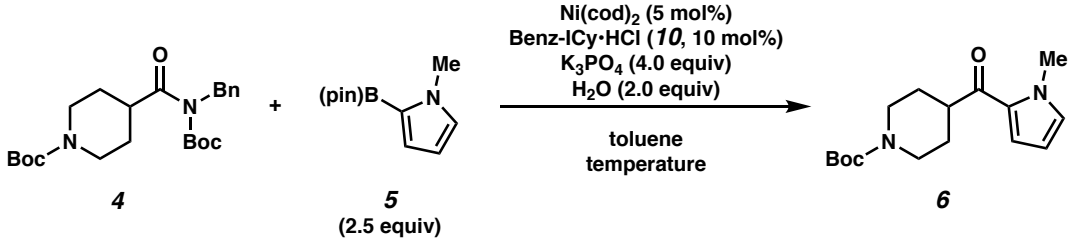


Amide SI-24. Purification by flash chromatography (14:1 Hexanes:EtOAc) generated amide **SI-24** (68% yield, over two steps) as a white solid. Amide **SI-24**: mp: 49–50 ºC; R_f 0.38 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.31–7.26 (m, 2H), 7.25–7.18 (m, 3H), 4.87 (d, J = 14.9, 1H), 4.81 (d, J = 14.9, 1H), 4.10–4.01 (m, 1H), 3.95–3.87 (m, 1H), 3.72–3.63 (m, 1H), 3.55 (t, J = 10.4, 1H), 3.44 (td, J = 10.8, 3.4, 1H), 2.13–2.04 (m, 1H), 1.81–1.63 (m, 3H), 1.42 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 176.7, 153.0, 138.3, 128.5, 127.6, 127.3, 83.7, 70.1, 68.4, 47.7, 44.0, 28.0, 27.3, 25.3; IR (film): 2977, 2847, 1732, 1685, 1371, 1146 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{18}H_{26}NO_4$, 320.18563; found 320.18577.



Amide 39. Purification by flash chromatography (9:1 Hexanes:EtOAc) generated amide **39** (49% yield, over two steps) as a white solid. Amide **39**: mp: 65–67 ºC; R_f 0.49 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.31–7.26 (m, 2H), 7.24–7.20 (m, 3H), 4.85 (s, 2H), 3.67 (s, 3H), 3.43–3.36 (m, 1H), 2.37–2.0 (m, 1H), 2.10–1.96 (m, 4H), 1.58–1.46 (m, 4H), 1.41 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 179.1, 176.2, 153.1, 138.5, 128.5, 127.6, 127.2, 83.4, 51.7, 47.8, 44.1, 42.8, 29.0, 28.4, 28.0; IR (film): 2977, 2946, 2865, 1728, 1689 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{21}H_{30}NO_5$, 376.21185; found 376.21140.

B. Initial Survey of Ligands and Relevant Control Experiments

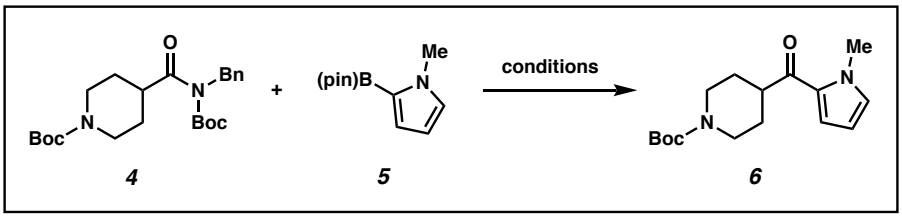


4 ***5*** (2.5 equiv) ***6***

Representative Procedure for Suzuki–Miyaura Reactions from Table S1 (coupling of amide 4 and *N*-methylpyrrole-2-boronic acid pinacol ester (5) is used as an example). A 1-dram vial was charged with anhydrous powdered K_3PO_4 (170 mg, 0.800 mmol, 4.0 equiv) and a magnetic stir bar. The vial and contents were flame-dried under reduced pressure, then allowed to cool under N_2. Amide substrate **4** (83.8 mg, 0.200 mmol, 1.0 equiv), *N*-methylpyrrole-2-boronic acid pinacol ester (**5**) (104 mg, 0.500 mmol, 2.5 equiv), and hexamethylbenzene (9.6 mg, 0.59 mmol, 0.30 equiv) were added. The vial was flushed with N_2, then water (7.2 μL, 0.400 mmol, 2.0 equiv), which had been sparged with N_2 for 10 min, was added. The vial was taken into a glove box and charged with $Ni(cod)_2$ (2.8 mg, 0.010 mmol, 5 mol%) and Benz-ICy•HCl (**10**, 6.4 mg, 0.020 mmol, 10 mol%). Subsequently, toluene (0.20 mL, 1.0 M) was added. The vial was sealed with a Teflon-lined screw cap, removed from the glove box, and stirred vigorously (800 rpm) at 120 °C for 16 h. After cooling to 23 °C, the mixture was diluted with hexanes (0.5 mL) and filtered over a plug of silica gel (10 mL of EtOAc eluent). The volatiles were removed under reduced pressure, and the yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard.

Any modifications of the conditions shown in the representative procedure above are specified below in Table S1.

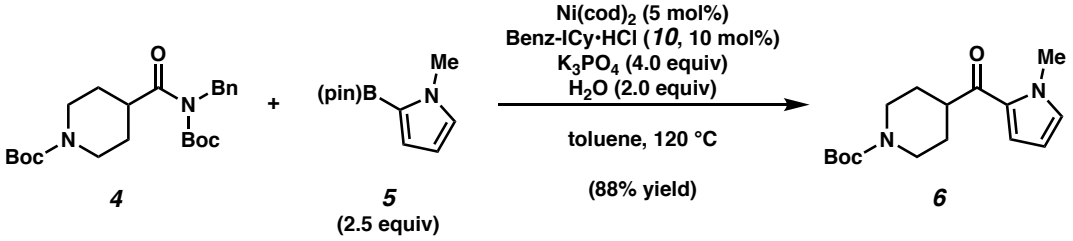
Table S1. Initial Survey of Ligands and Relevant Control Experiments[a]



Reaction Conditions	*Experimental Results* 4	6
5 (2.5 equiv), K_3PO_4 (4.0 equiv), H_2O (2.0 equiv) $Ni(cod)_2$ (5 mol%), SIPr (***7***,10 mol%), toluene (1.0 M), 50 °C, 16 h	100%	0%
5 (2.5 equiv), K_3PO_4 (4.0 equiv), H_2O (2.0 equiv) $Ni(cod)_2$ (5 mol%), SIPr (***7***,10 mol%), toluene (1.0 M), 120 °C, 16 h	52%[b]	0%
5 (2.5 equiv), K_3PO_4 (4.0 equiv), H_2O (2.0 equiv) $Ni(cod)_2$ (5 mol%), terpyridine (***8***,10 mol%), toluene (1.0 M), 120 °C, 16 h	50%[b]	0%
5 (2.5 equiv), K_3PO_4 (4.0 equiv), H_2O (2.0 equiv) $Ni(cod)_2$ (5 mol%), ICy·HBF_4 (***9***,10 mol%), toluene (1.0 M), 120 °C, 16 h	0%	95%
5 (2.5 equiv), K_3PO_4 (4.0 equiv), H_2O (2.0 equiv) $Ni(cod)_2$ (5 mol%), Benz-ICy·HCl (***10***,10 mol%), toluene (1.0 M), 120 °C, 16 h	0%	95%
Control Experiments:		
5 (2.5 equiv), K_3PO_4 (4.0 equiv), H_2O (2.0 equiv) toluene (1.0 M), 120 °C, 16 h	25%[b]	0%
5 (2.5 equiv), K_3PO_4 (4.0 equiv), H_2O (2.0 equiv) Benz-ICy·HCl (***10***,10 mol%), toluene (1.0 M), 120 °C, 16 h	25%[b]	0%
5 (2.5 equiv), K_3PO_4 (4.0 equiv), H_2O (2.0 equiv) $Ni(cod)_2$ (5 mol%), toluene (1.0 M), 120 °C, 16 h	5%[b]	0%

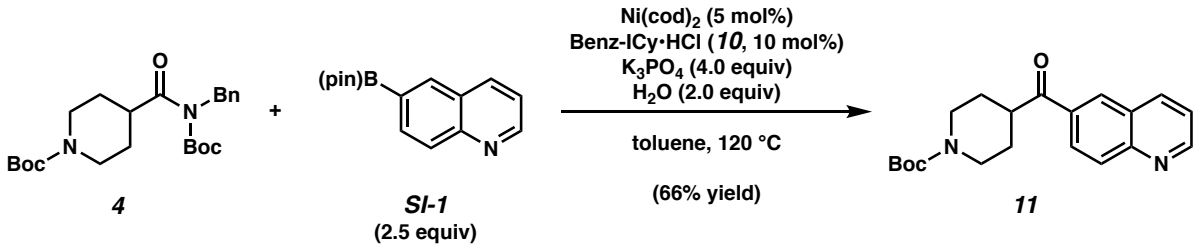
[a] Yields were determined by ^1H NMR analysis using hexamethylbenzene as an internal standard.
[b] Substantial amounts of the corresponding Boc-cleavage product (des-Boc amide starting material) were observed due to the elevated reaction temperature.

C. Scope of Methodology

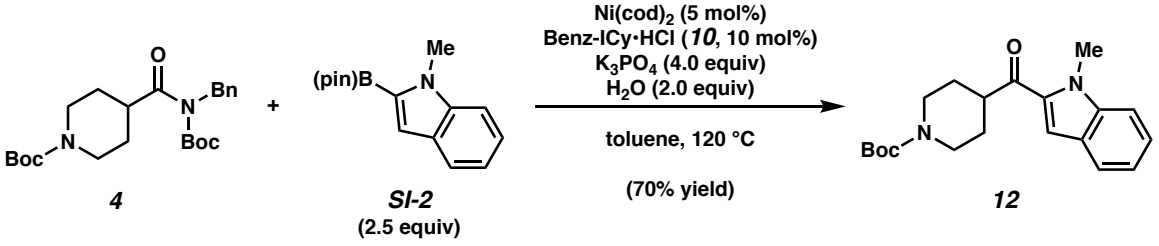


Representative Procedure (coupling of amide 4 and *N*-methylpyrrole-2-boronic acid pinacol ester (5) is used as an example). Ketone 6. A 1-dram vial was charged with anhydrous powdered K_3PO_4 (170 mg, 0.800 mmol, 4.0 equiv) and a magnetic stir bar. The vial and contents were flame-dried under reduced pressure, then allowed to cool under N_2. Amide substrate **4** (83.8 mg, 0.200 mmol, 1.0 equiv) and *N*-methylpyrrole-2-boronic acid pinacol ester (**5**) (104 mg, 0.500 mmol, 2.5 equiv) were added. The vial was flushed with N_2, then water (7.2 μL, 0.400 mmol, 2.0 equiv), which had been sparged with N_2 for 10 min, was added. The vial was taken into a glove box and charged with $Ni(cod)_2$ (2.8 mg, 0.010 mmol, 5 mol%) and Benz-ICy•HCl (**10**, 6.4 mg, 0.020 mmol, 10 mol%). Subsequently, toluene (0.20 mL, 1.0 M) was added. The vial was sealed with a Teflon-lined screw cap, removed from the glove box, and stirred vigorously (800 rpm) at 120 °C for 16 h. After cooling to 23 °C, the mixture was diluted with hexanes (0.5 mL) and filtered over a plug of silica gel (10 mL of EtOAc eluent). The volatiles were removed under reduced pressure, and the crude residue was purified by flash chromatography (19:1 Hexanes:EtOAc → 14:1 Hexanes:EtOAc → 9:1 Hexanes:EtOAc) to yield ketone product **6** (88% yield, average of two experiments) as a white solid. Ketone **6**: mp: 77–80 ºC; R_f 0.18 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.00–6.95 (m, 1H), 6.85–6.80 (m, 1H), 6.16–6.11 (m, 1H), 4.18 (br s, 2H), 3.93 (s, 3H), 3.20–3.10 (m, 1H), 2.93–2.70 (m, 2H), 1.85–1.66 (m, 4H), 1.46 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.1, 154.9, 131.6, 129.8, 118.9, 108.1, 79.7, 44.8, 43.6, 38.0, 29.1, 28.6; IR (film): 2929, 2859, 1686, 1646, 1408, 1168 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{16}H_{25}N_2O_3$, 293.18597; found 293.18535.

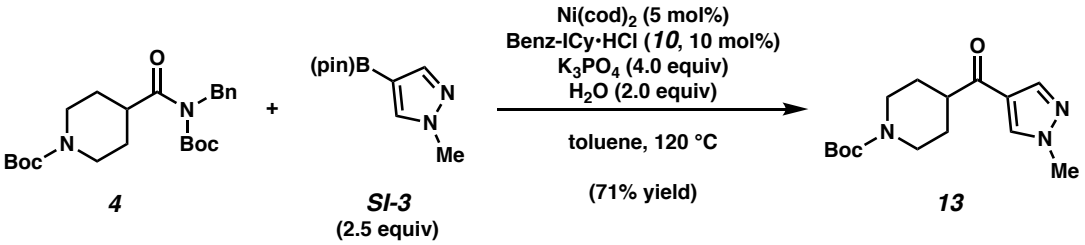
Any modifications of the conditions shown in the representative procedure above are specified in the following schemes, which depict all of the results shown in Figures 3, 4, 5, and 6.



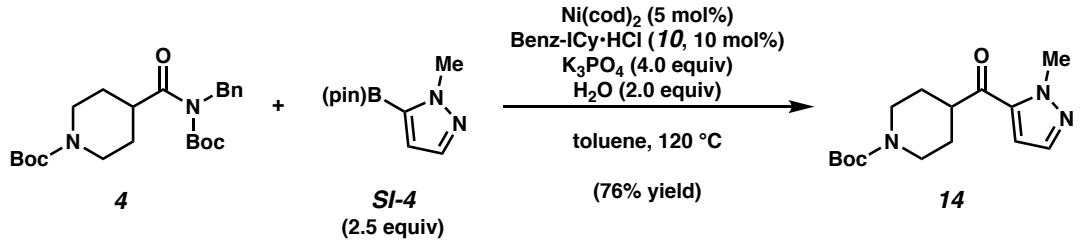
Ketone 11. Purification by flash chromatography (1:1 Hexanes:EtOAc → 1:2 Hexanes:EtOAc) generated ketone **11** (66% yield, average of two experiments) as a clear oil. Ketone **11**: R_f 0.33 (1:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.02 (dd, *J* = 4.2, 1.7, 1H), 8.44 (d, *J* = 1.9, 1H), 8.29 (dd, *J* = 8.3, 1.3, 1H), 8.23 (dd, *J* = 8.8, 1.9, 1H), 8.18 (d, *J* = 8.8, 1H), 7.50 (dd, *J* = 8.3, 4.2, 1H), 4.20 (br s, 2H), 3.56 (tt, *J* = 11.1, 3.7, 1H), 3.04–2.85 (m, 2H), 1.98–1.84 (m, 2H), 1.82–1.74 (m, 2H), 1.47 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$, 14 of 16 observed): δ 201.6, 154.8, 152.8, 150.2, 137.7, 133.8, 130.5, 129.6, 128.0, 127.7, 122.2, 79.9, 43.9, 28.6; IR (film): 2972, 2859, 1676, 1423, 1366, 1161 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{20}H_{25}N_2O_3$, 341.18597; found 341.18465.



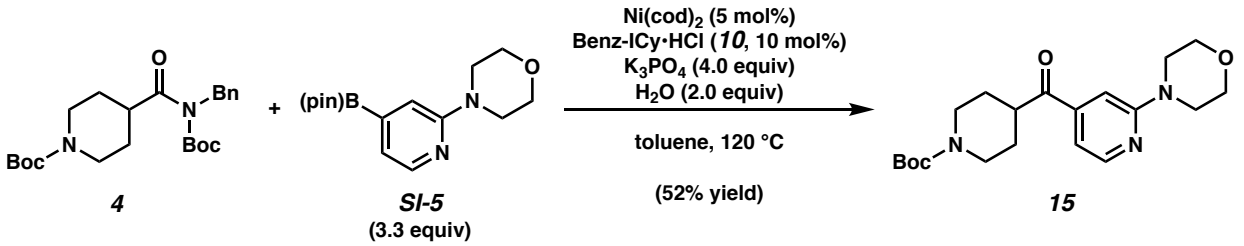
Ketone 12. Purification by flash chromatography (19:1 Hexanes:EtOAc → 14:1 Hexanes:EtOAc → 9:1 Hexanes:EtOAc) generated ketone **12** (70% yield, average of two experiments) as a white solid. Ketone **12**: R_f 0.25 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[4]



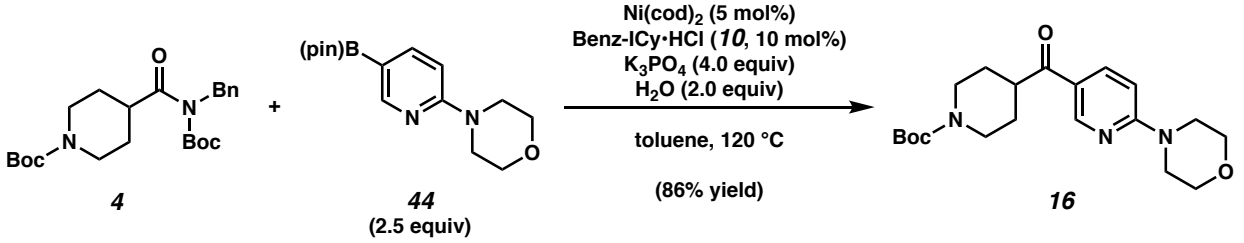
Ketone 13. Purification by flash chromatography (49:1 PhH:CH_3CN → 19:1 PhH:CH_3CN → 1:1 Hexanes:EtOAc → 1:3 Hexanes:EtOAc) generated ketone **13** (71% yield, average of two experiments) as a white solid. Ketone **13**: mp: 99–101 ºC; R_f 0.24 (1:3 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.89 (s, 1H), 7.88 (s, 1H), 4.15 (br s, 2H), 3.94 (s, 3H), 3.27 (tt, J = 11.1, 3.9, 1H), 2.93–2.73 (m, 2H), 1.93–1.63 (m, 4H), 1.46 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$, 10 of 11 observed): δ 196.4, 154.8, 140.4, 132.8, 123.0, 79.8, 46.3, 39.6, 28.6, 28.4; IR (film): 2977, 2937, 2859, 1671, 1540, 1168 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{15}H_{24}N_3O_3$, 294.18122; found 294.18073.



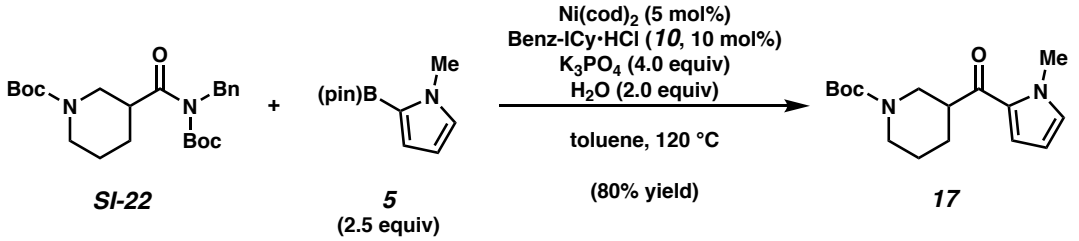
Ketone 14. Purification by flash chromatography (4:1 Hexanes:EtOAc → 3:1 Hexanes:EtOAc) generated ketone **14** (76% yield, average of two experiments) as a clear oil. Ketone **14**: R_f 0.42 (2:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.48 (d, J = 2.1, 1H), 6.84 (d, J = 2.1, 1H), 4.16 (s, 5H), 3.12 (tt, J = 11.3, 3.7, 1H), 2.93–2.75 (m, 2H), 1.89–1.76 (m, 2H), 1.75–1.66 (m, 2H), 1.46 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$, 9 of 11 observed): δ 193.5, 154.8, 137.8, 137.6, 111.2, 79.9, 46.3, 40.6, 28.6; IR (film): 2955, 2860, 1677, 1423, 1366, 1321, 1169 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{15}H_{24}N_3O_3$, 294.18122; found 294.18035.



Ketone 15. Purification by flash chromatography (2:1 Hexanes:EtOAc) generated ketone **15** (52% yield, average of two experiments) as a yellow oil. Ketone **15**: R_f 0.31 (2:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.33 (dd, J = 5.1, 0.8, 1H), 7.03 (s, 1H), 7.00 (dd, J = 5.2, 1.2, 1H), 4.13 (br s, 2H), 3.85–8.80 (m, 4H), 3.59–3.54 (m, 4H), 3.27 (tt, J = 11.1, 3.6, 1H), 2.97–2.80 (m, 2H), 1.91–1.77 (m, 2H), 1.70–1.60 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$, 13 of 14 observed): δ 202.5, 160.5, 154.8, 149.3, 144.1, 111.1, 104.7, 79.9, 66.8, 45.6, 44.2, 28.6, 28.2; IR (film): 2969, 2854, 1688, 1426, 1241, 1166 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{20}H_{30}N_3O_4$, 376.22308; found 376.22152.

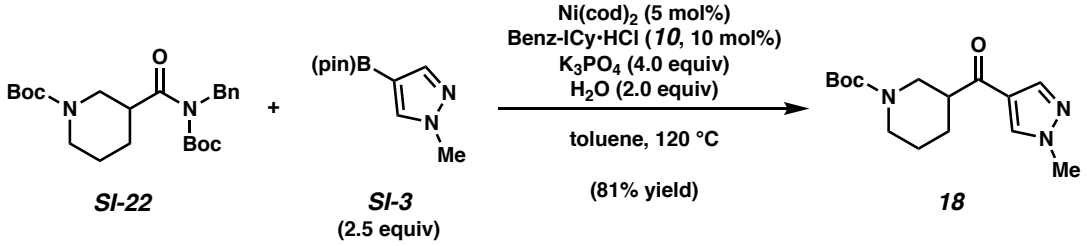


Ketone 16. Purification by flash chromatography (5:1 Hexanes:EtOAc → 9:1 CH_2Cl_2:MeOH) generated ketone **16** (86% yield, average of two experiments) as a white solid. Ketone **16**: mp: 131–133 ºC; R_f 0.52 (1:3 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.82–8.72 (m, 1H), 3.26 (dd, J = 9.1, 2.5, 1H), 6.69–6.58 (m, 1H), 4.17 (br s, 2H), 3.86–3.77 (m, 4H), 3.73–3.64 (m, 4H), 3.27 (tt, J = 11.1, 3.8, 1H), 2.99–2.71 (m, 2H), 1.94–1.64 (m, 4H), 1.46 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$, 12 of 14 observed): δ 199.4, 160.9, 154.9, 150.4, 137.9, 121.5, 105.9, 79.8, 66.7, 45.0, 43.3, 28.6; IR (film): 2969, 2857, 1686, 1593, 1418, 1216, 1168 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{20}H_{30}N_3O_4$, 376.22308; found 376.22247.



Ketone 17. Purification by flash chromatography (19:1 Hexanes:EtOAc → 14:1 Hexanes:EtOAc → 9:1 Hexanes:EtOAc) generated ketone **17** (80% yield, average of two experiments) as a white solid. Ketone **17**: mp: 86–88 ºC; R_f 0.19 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.08–7.02 (m, 1H), 6.82 (br s, 1H), 6.17–6.12 (m, 1H), 4.40–4.00 (m, 2H), 3.93 (s, 3H), 3.22–3.09 (m, 1H), 2.99–2.78 (m, 1H), 2.76–2.61 (m, 1H), 2.02–1.92 (m, 1H), 1.78–1.69 (m, 2H), 1.57–1.50 (m, 1H), 1.47 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.0, 154.9, 131.7, 129.9, 119.5, 108.3, 79.7, 47.8, 47.1, 45.3, 44.0, 37.9, 28.6, 28.4, 24.8; IR (film): 2937, 2862, 1690, 1645, 1408 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{16}H_{25}N_2O_3$, 293.18597; found 293.18458.

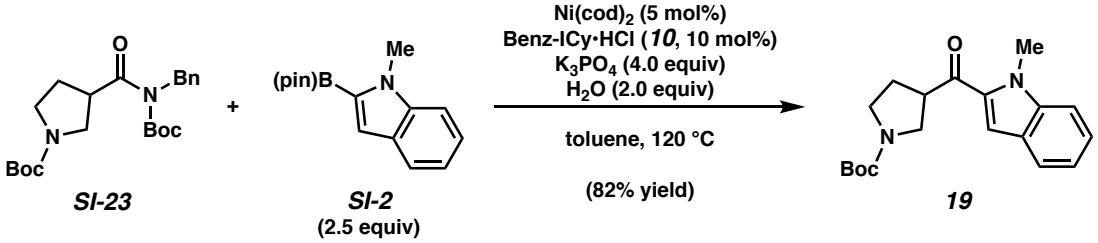
*Note: Ketone **17** was obtained as a mixture of conformers. These data represent empirically observed chemical shifts from the ^{13}C NMR spectrum.*



Ketone 18. Purification by flash chromatography (9:1 Hexanes:EtOAc → 5:1 Hexanes:EtOAc → 2:1 Hexanes:EtOAc → 1:1 Hexanes:EtOAc) generated ketone **18** (81% yield, average of two experiments) as a white solid. Ketone **18**: mp: 96–97 ºC; R_f 0.19 (1:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.93 (s, 1H), 7.91 (br s, 1H), 4.40–4.01 (m, 2H), 3.94 (s, 3H), 3.05–2.65 (m, 3H), 2.03–1.95 (m, 1H), 1.79–1.64 (m, 2H), 1.55–1.43 (m, 10H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 195.3, 154.8, 140.6, 132.8, 123.1, 79.9, 46.7, 45.0, 43.9, 39.5, 28.6, 27.8, 24.8; IR

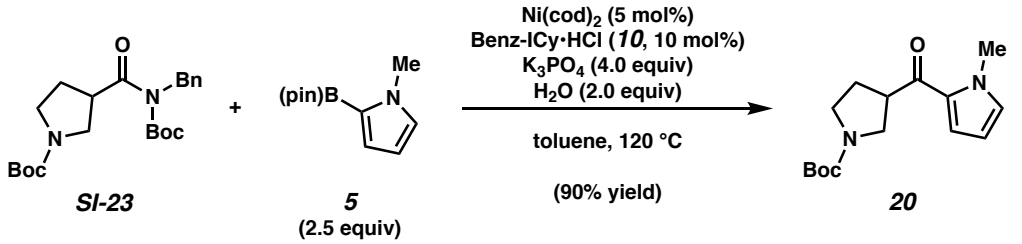
(film): 2939, 2862, 1683, 1663, 1540, 1148 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{15}H_{24}N_3O_3$, 294.18122; found 294.17877.

*Note: Ketone **18** was obtained as a mixture of conformers. These data represent empirically observed chemical shifts from the ^{13}C NMR spectrum.*



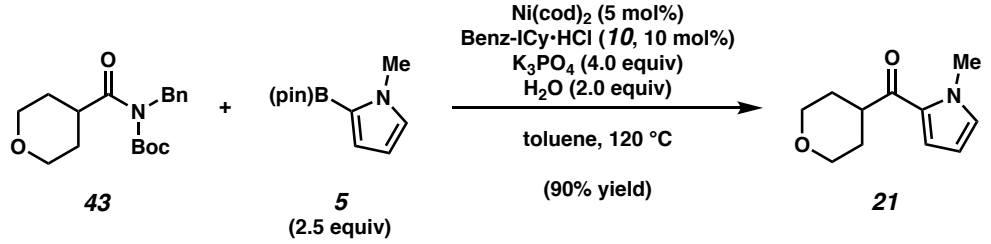
Ketone 19. Purification by flash chromatography (4:1 Hexanes:EtOAc) generated ketone **19** (82% yield, average of two experiments) as a clear oil. Ketone **19**: R_f 0.26 (4:1 Hexanes:EtOAc); ^1H NMR (500 MHz, $CDCl_3$): δ 7.70 (br s, 1H), 7.39 (br s, 2H), 7.33 (br s, 1H), 7.17 (br s, 1H), 4.08 (s, 3H), 4.04–3.91 (m, 1H), 3.82–3.65 (m, 1H), 3.65–3.39 (m, 3H), 2.35–2.12 (m, 2H), 1.47 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.1, 192.9, 154.5, 140.5, 134.2, 126.4, 125.9, 123.1, 121.1, 112.0, 110.6, 79.5, 49.0, 48.9, 47.3, 46.3, 45.8, 45.6, 32.4, 29.7, 29.4, 28.6; IR (film): 2974, 2882, 1688, 1658, 1393, 1166, 1118 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{19}H_{25}N_2O_3$, 329.18597; found 329.18463.

*Note: Ketone **19** was obtained as a mixture of conformers. These data represent empirically observed chemical shifts from the ^{13}C NMR spectrum.*

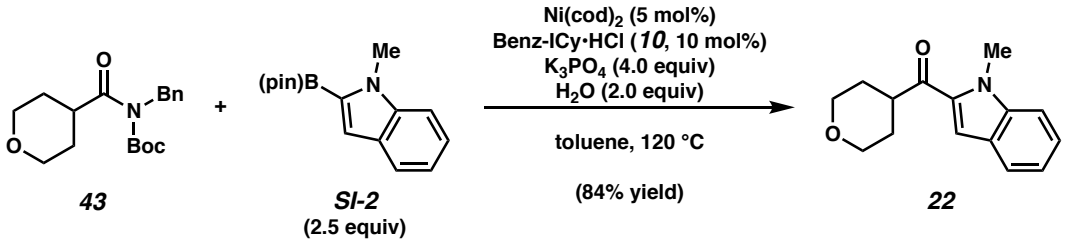


Ketone 20. Purification by flash chromatography (4:1 Hexanes:EtOAc) generated ketone **20** (90% yield, average of two experiments) as a clear oil. Ketone **20**: R_f 0.18 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.97 (br s, 1H), 6.83 (br s, 1H), 6.14 (br s, 1H), 3.93 (s, 3H), 3.83–3.44 (m, 4H), 3.38 (br s, 1H), 2.28–2.12 (m, 1H), 2.08 (br s, 1H), 1.45 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 189.9, 189.7, 154.5, 131.9, 130.2, 119.6, 108.4, 79.4, 49.0, 48.9, 46.4, 45.9, 45.6, 45.5, 37.9, 29.5, 29.4, 28.6; IR (film): 2977, 2882, 1686, 1643, 1401, 1366, 1118 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{15}H_{23}N_2O_3$, 279.17032; found 279.17976.

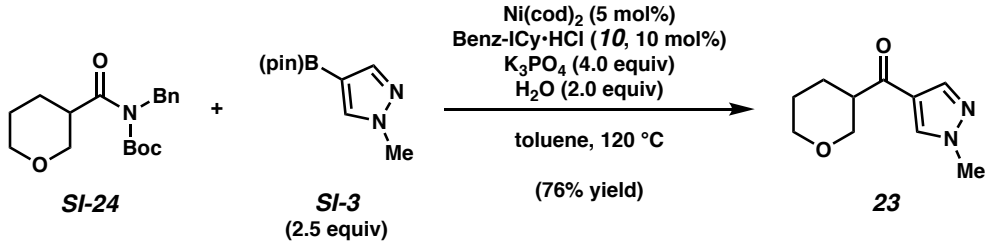
*Note: Ketone **20** was obtained as a mixture of conformers. These data represent empirically observed chemical shifts from the ^{13}C NMR spectrum.*



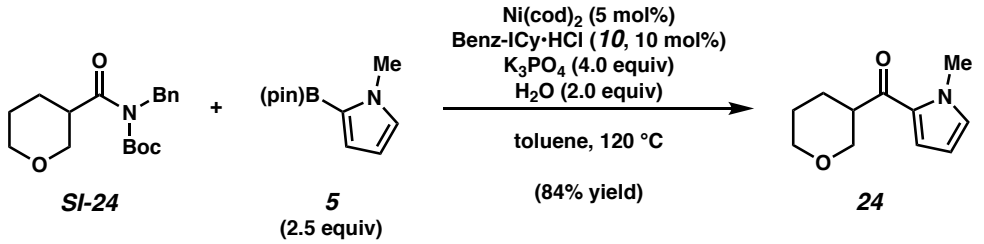
Ketone 21. Purification by flash chromatography (5:1 Hexanes:EtOAc) generated ketone **21** (90% yield, average of two experiments) as a white solid. Ketone **21**: mp: 72–74 ºC; R_f 0.21 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.00–6.95 (m, 1H), 6.82 (s, 1H), 6.15–6.10 (m, 1H), 4.09–4.00 (m, 2H), 3.94 (s, 3H), 3.51 (t, J = 11.8, 2H), 3.26 (tt, J = 11.5, 3.8, 1H), 1.91 (qd, J = 12.4, 4.3, 2H), 1.70 (d, J = 13.4, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.9, 131.6, 129.8, 118.9, 108.1, 67.6, 43.8, 38.0, 29.7; IR (film): 2952, 2847, 1642, 1408, 1306, 1094 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{11}H_{16}NO_2$, 194.11756; found 194.11707.



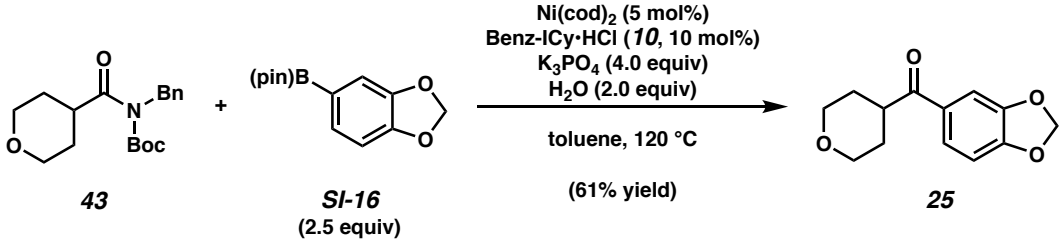
Ketone 22. Purification by flash chromatography (5:1 Hexanes:EtOAc) generated ketone **22** (84% yield, average of two experiments) as a white solid. Ketone **22**: mp: 63–66 ºC; R_f 0.35 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.70 (d, *J* = 8.1, 1H), 7.39 (d, *J* = 3.6, 2H), 7.33 (s, 1H), 7.19–7.14 (m, 1H), 4.12–4.09 (m, 1H), 4.07 (s, 4H), 3.57 (t, *J* = 11.7, 2H), 3.48 (tt, *J* = 11.5, 3.6, 1H), 1.96 (qd, *J* = 12.4, 4.2, 2H), 1.81 (d, *J* = 13.2, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 196.0, 140.4, 133.8, 126.1, 125.9, 123.0, 120.9, 111.1, 110.6, 67.5, 44.7, 32.4, 29.8; IR (film): 2954, 2844, 1656, 1511, 1386, 1118 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{15}H_{18}NO_2$, 244.13321; found 244.13264.



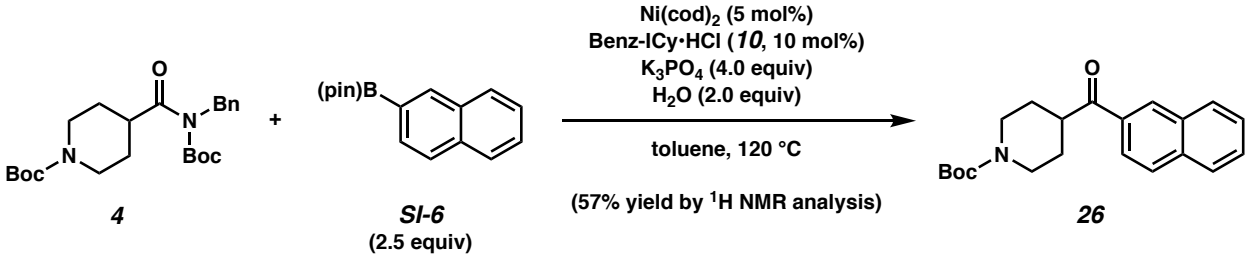
Ketone 23. Purification by flash chromatography (1:2 Hexanes:EtOAc) generated ketone **23** (76% yield, average of two experiments) as a yellow solid. Ketone **23**: mp: 83–84 ºC; R_f 0.25 (1:2 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.89 (s, 1H), 7.87 (s, 1H), 4.04 (d, J = 11.1, 1H), 3.96–3.86 (m, 4H), 3.50 (t, *J* = 10.9, 1H), 3.43–3.34 (m, 1H), 3.20–3.11 (m, 1H), 1.97 (d, *J* = 12.7, 1H), 1.86–1.73 (m, 1H), 1.73–1.63 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 195.2, 140.4, 132.8, 123.3, 69.8, 68.2, 47.2, 39.5, 26.6, 25.2; IR (film): 2947, 2852, 1656, 1541, 1401, 1188, 1080 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{10}H_{15}N_2O_2$, 165.11280; found 165.11223.



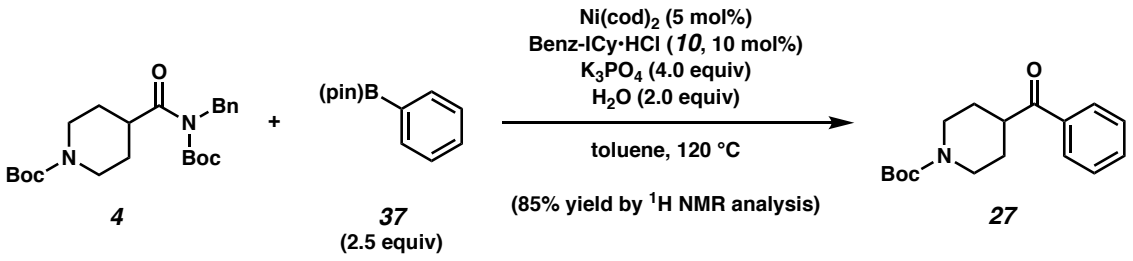
Ketone 24. Purification by flash chromatography (4:1 Hexanes:EtOAc) generated ketone **24** (84% yield, average of two experiments) as a clear oil. Ketone **24**: R_f 0.30 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.04–7.00 (m, 1H), 6.81 (s, 1H), 6.15–6.10 (m, 1H), 4.09–4.02 (m, 1H), 3.98–3.92 (m, 1H), 3.91 (s, 3H), 3.52 (t, *J* = 10.9, 1H), 3.44–3.33 (m, 2H), 2.00–1.93 (m, 1H), 1.84 (qd, *J* = 12.1, 4.3, 1H), 1.78–1.65 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 191.8, 131.7, 130.1, 119.5, 108.2, 70.6, 68.3, 45.7, 37.9, 27.1, 25.4; IR (film): 2947, 2849, 1638, 1406, 1201, 1065 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{11}H_{16}NO_2$, 194.11756; found 194.11699.



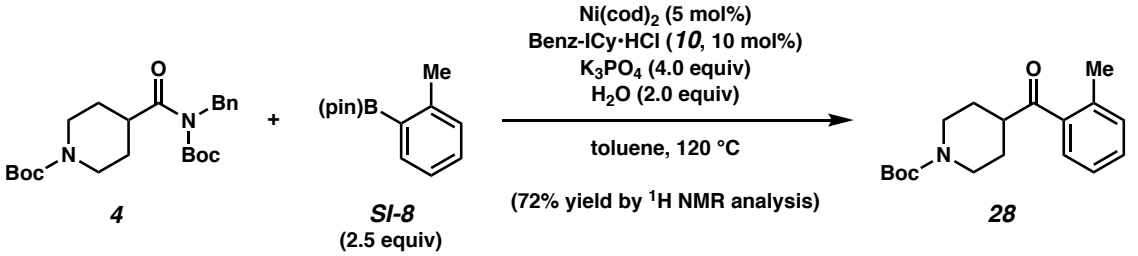
Ketone 25. Purification by flash chromatography (30:15:1 Hexanes:EtOAc:TEA) generated ketone **25** (61% yield, average of two experiments) as a white solid. Ketone **25**: mp: 97–98 ºC; R_f 0.35 (2:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.55 (dd, *J* = 8.2, 1.8, 1H), 7.42 (d, *J* = 1.8, 1H), 6.86 (d, *J* = 8.2, 1H), 6.04 (s, 2H), 4.05 (ddd, *J* = 11.4, 4.0, 2.4, 2H), 3.54 (td, *J* = 11.7, 2.2, 2H), 3.40 (tt, *J* = 11.2, 3.8, 1H), 1.92–1.83 (m, 2H), 1.77–1.72 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 200.0, 151.9, 148.5, 130.7, 124.5, 108.3, 108.1, 102.0, 67.5, 42.6, 29.4; IR (film): 2955, 2847, 1670, 1440, 1258, 1241, 1114 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{13}H_{15}O_4$, 235.09649; found 235.09592.



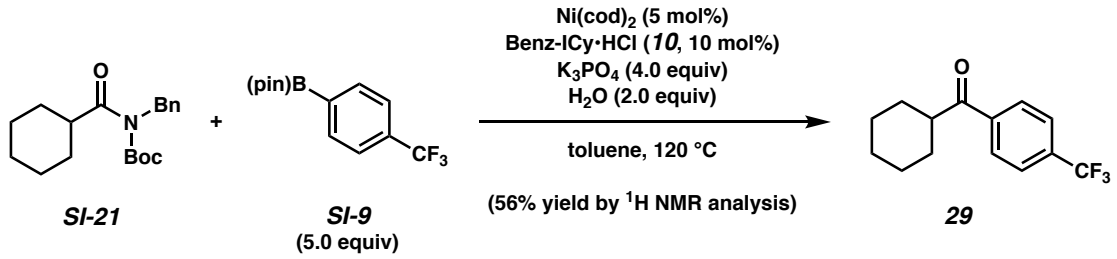
Ketone 26. ^{1}H NMR analysis of the crude reaction mixture indicated a 57% yield of ketone **26** relative to hexamethylbenzene internal standard. Purification by preparative thin-layer chromatography (4:1 Hexanes:EtOAc) provided an analytical sample of ketone **26** as a white amorphous solid. Ketone **26**: R_f 0.29 (4:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.45 (s, 1H), 8.02–7.96 (m, 2H), 7.93–7.86 (m, 2H), 7.63–7.54 (m, 2H), 4.20 (br s, 2H), 3.58 (tt, J = 11.1, 3.7, 1H), 3.04–2.87 (m, 2H), 1.97–1.84 (m, 2H), 1.82–1.71 (m, 2H), 1.48 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 202.2, 154.9, 135.7, 133.3, 132.7, 129.8, 129.7, 128.8, 128.7, 127.9, 127.0, 124.3, 79.8, 43.7, 43.3, 28.7, 28.6; IR (film): 3060, 2975, 2930, 2858, 1682 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{21}H_{26}NO_3$, 340.19072; found 340.19041.



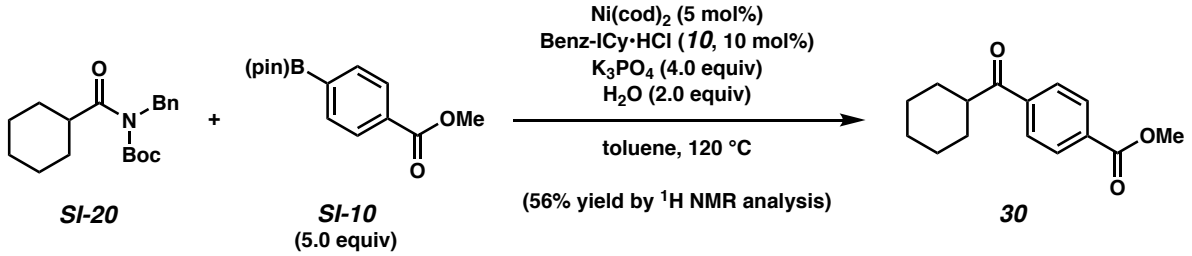
Ketone 27. ^{1}H NMR analysis of the crude reaction mixture indicated an 85% yield of ketone **27** relative to hexamethylbenzene internal standard. Purification by preparative thin-layer chromatography (3:1 Hexanes:EtOAc) provided an analytical sample of ketone **27** as a white amorphous solid. Ketone **27**: R_f 0.21 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[5]



Ketone 28. ^{1}H NMR analysis of the crude reaction mixture indicated a 72% yield of ketone **28** relative to hexamethylbenzene internal standard. Purification by preparative thin-layer chromatography (4:1 Hexanes:EtOAc) provided an analytical sample of ketone **28** as a clear oil. Ketone **28**: R_f 0.42 (3:1 Hexanes:EtOAc). Spectral data match those previously reported.[4]

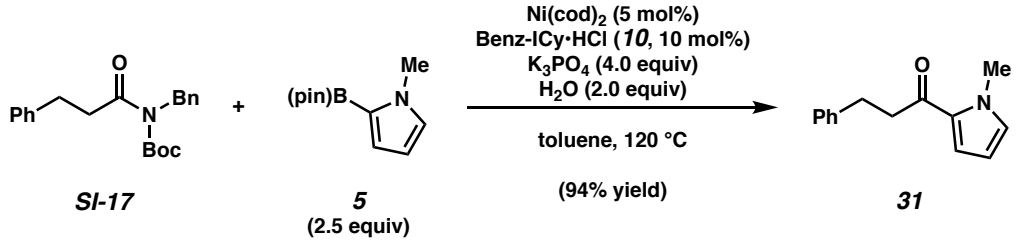


Ketone 29. ^{1}H NMR analysis of the crude reaction mixture indicated a 56% yield of ketone **29** relative to hexamethylbenzene internal standard. Purification by preparative thin-layer chromatography (9:1 Hexanes:EtOAc) provided an analytical sample ketone **29** as a white solid. Ketone **29**: R_f 0.56 (9:1 Hexanes:EtOAc). Spectral data match those previously reported.[6]

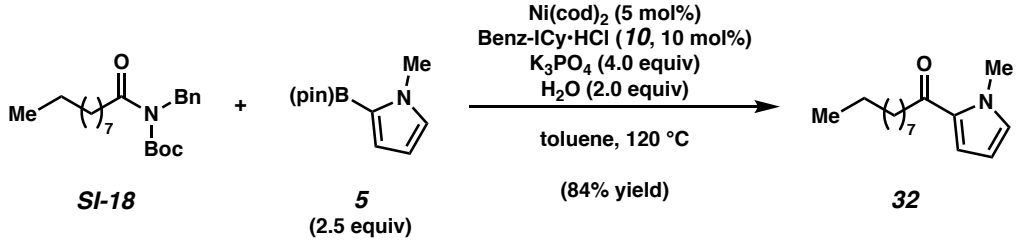


Ketone 30. ^{1}H NMR analysis of the crude reaction mixture indicated a 38% yield of ketone **30** relative to hexamethylbenzene internal standard. Purification by preparative thin-layer

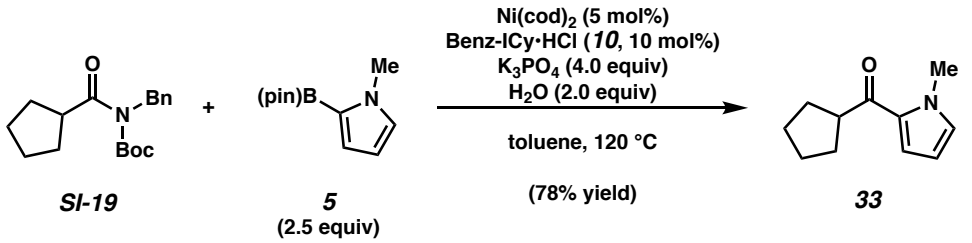
chromatography (9:1 Hexanes:EtOAc) provided an analytical sample of ketone **30** as a white solid. Ketone **30**: R_f 0.39 (9:1 Hexanes:EtOAc). Spectral data match those previously reported.[7]



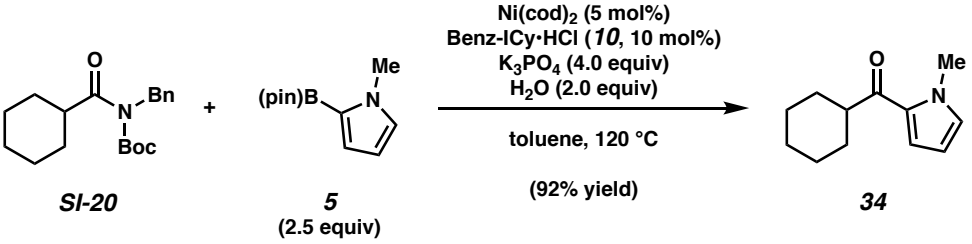
Ketone 31. Purification by flash chromatography (19:1 Hexanes:EtOAc → 14:1 Hexanes:EtOAc → 9:1 Hexanes:EtOAc) generated ketone **31** (94% yield, average of two experiments) as a clear oil. Ketone **31**: R_f 0.43 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[8]



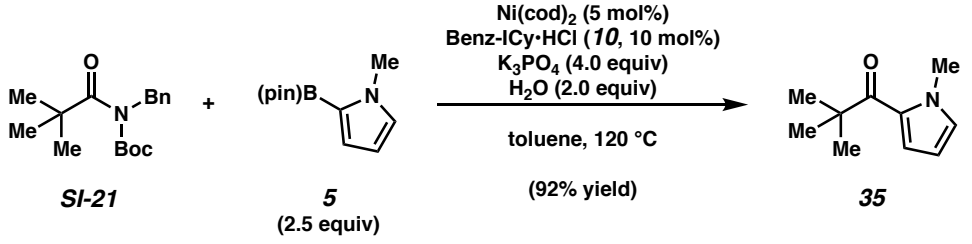
Ketone 32. Purification by flash chromatography (24:1 Hexanes:EtOAc) generated ketone **32** (84% yield, average of two experiments) as a clear oil. Ketone **32**: R_f 0.52 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.95 (dd, J = 4.1, 1.7, 1H), 6.80–6.77 (m, 1H), 6.11 (dd, J = 4.1, 2.5, 1H), 3.94 (s, 3H), 2.77–2.73 (m, 2H), 1.69 (quint, J = 7.5, 2H), 1.39–1.20 (m, 12H), 0.88 (t, J = 7.1, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.0, 131.0, 130.9, 119.0, 107.9, 39.3, 37.9, 32.0, 29.7, 29.633, 29.627, 29.5, 25.5, 22.8, 14.3; IR (film): 2955, 2923, 2853, 1649, 1528 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{15}H_{26}NO$, 236.20089; found 236.20080.



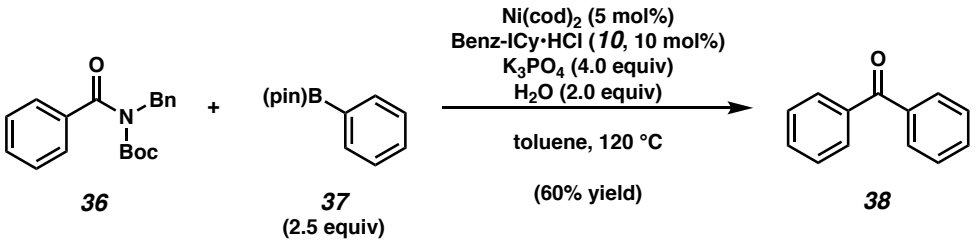
Ketone 33. Purification by flash chromatography (24:1 Hexanes:EtOAc → 19:1 Hexanes:EtOAc) generated ketone **33** (78% yield, average of two experiments) as a clear oil. Ketone **33**: R_f 0.50 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[9]



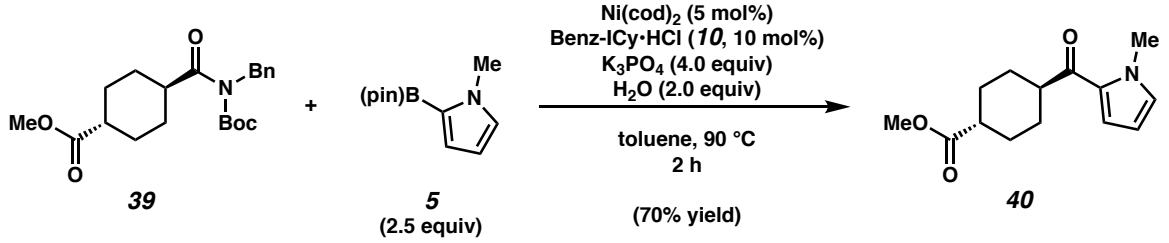
Ketone 34. Purification by flash chromatography (14:1 Hexanes:EtOAc) generated ketone **34** (92% yield, average of two experiments) as a clear oil. Ketone **34**: R_f 0.28 (14:1 Hexanes:EtOAc). Spectral data match those previously reported.[10]



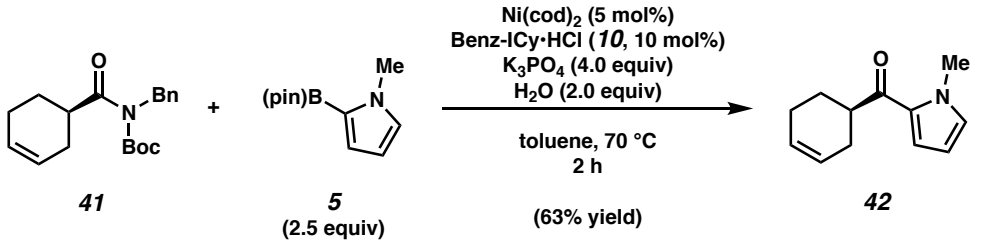
Ketone 35. Purification by flash chromatography (19:1 Hexanes:EtOAc) generated ketone **35** (92% yield, average of two experiments) as a clear oil. Ketone **35**: R_f 0.66 (4:1 Hexanes:EtOAc). Spectral data match those previously reported.[11]



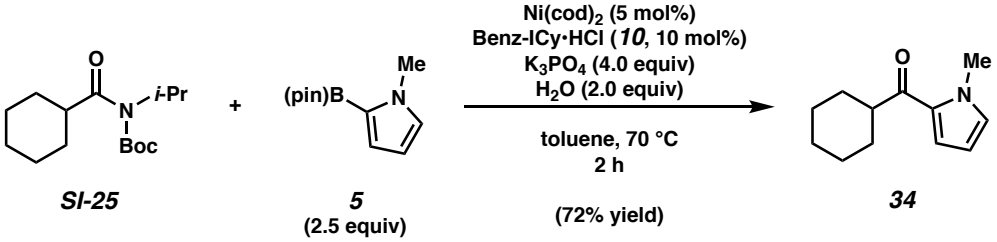
Ketone 38. Purification by thin-layer chromatography (5:1 Hexanes:EtOAc) generated ketone **38** (the reported yield was based on ^{1}H NMR analysis using hexamethylbenzene as an external standard) as a white solid. Ketone **38**: R_f 0.56 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[3]



Ketone 40. Purification by flash chromatography (49:1 $CHCl_3$:CH_3CN) generated ketone **40** (the reported yield was based on ^{1}H NMR analysis using hexamethylbenzene as an external standard) as a white solid. Ketone **40**: R_f 0.48 (19:1 $CHCl_3$:CH_3CN); ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.97 (dd, J = 4.1, 1.7, 1H), 6.83–6.80 (m, 1H), 6.13 (dd, J = 4.1, 2.5, 1H), 3.93 (s, 3H), 3.68 (s, 3H), 3.06–2.99 (m, 1H), 2.38–2.30 (m, 1H), 2.14–2.05 (m, 2H), 1.98–1.88 (m, 2H), 1.63–1.49 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 194.3, 176.3, 131.5, 130.0, 118.9, 108.0, 51.7, 45.9, 42.7, 37.9, 29.0, 28.5; IR (film): 2942, 2862, 1730, 1645, 1408, 1251 cm^{-1}; HRMS-APCI (*m/z*) [M + H]$^+$ calcd for $C_{14}H_{20}NO_3$, 250.14377; found 250.14273.



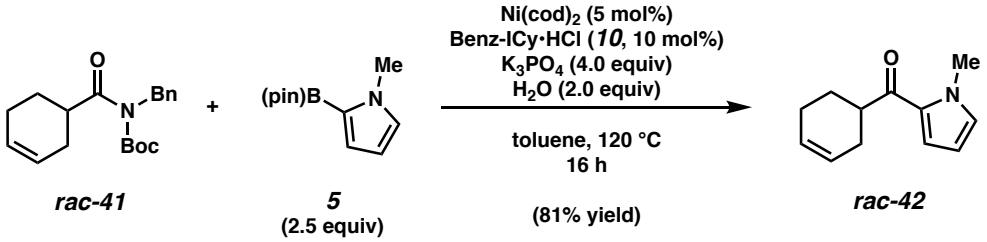
Ketone 42. Purification by flash chromatography (19:1 Hexanes:EtOAc → 14:1 Hexanes:EtOAc) generated ketone **42** (63% yield, average of two experiments) as a clear oil. Ketone **42**: R_f 0.46 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.99 (dd, J = 4.1, 1.6, 1H), 6.83–6.80 (m, 1H), 6.13 (dd, J = 4.1, 2.4, 1H), 5.79–5.70 (m, 2H), 3.95 (s, 3H), 3.32–3.25 (m, 1H), 2.39–2.30 (m, 1H), 2.20–2.11 (m, 3H), 1.96–1.90 (m, 1H), 1.79–1.69 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 194.8, 131.3, 130.3, 126.6, 126.2, 119.0, 108.0, 42.7, 38.0, 28.6, 26.4, 25.2; IR (film): 3107, 3023, 2931, 2838, 1643, 1527 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{12}H_{16}NO$, 190.12264; found 190.12245. $[\alpha]^{20.7}_D$ –6.20 ° (*c* = 1.00, $CHCl_3$).



Ketone 34. Purification by column chromatography (49:1 Hexanes:EtOAc) generated ketone **34** (the reported yield was based on ^{1}H NMR analysis using hexamethylbenzene as an external standard) as a clear oil. Ketone **34**: R_f 0.28 (14:1 Hexanes:EtOAc). Spectral data match those previously reported.10

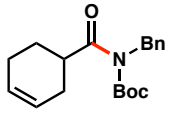
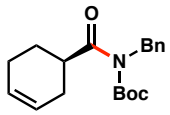
D. Verification of Enantiopurity

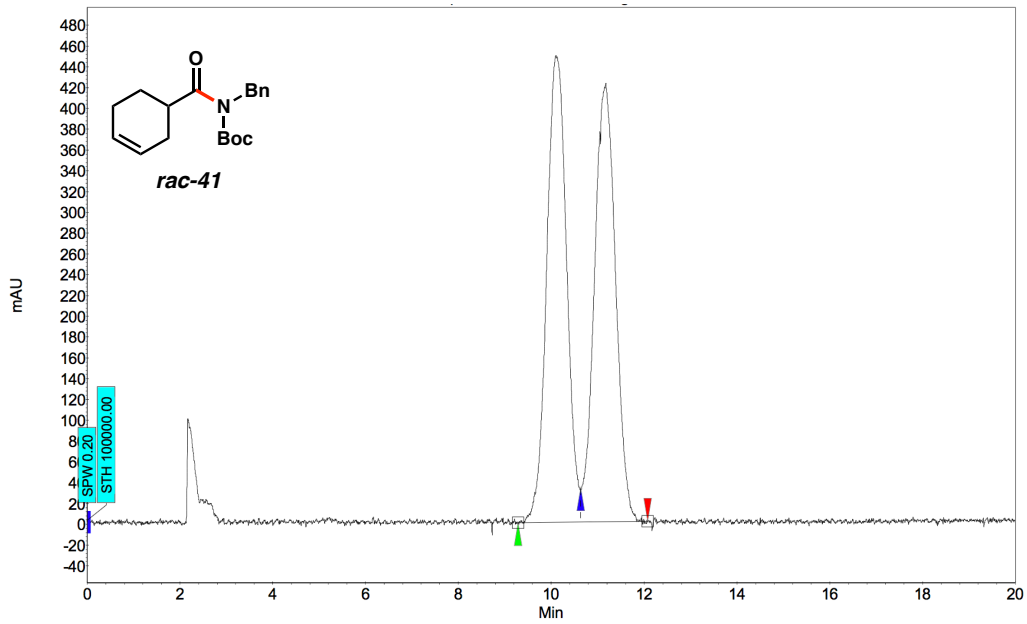
a) Synthesis of Racemic Ketone



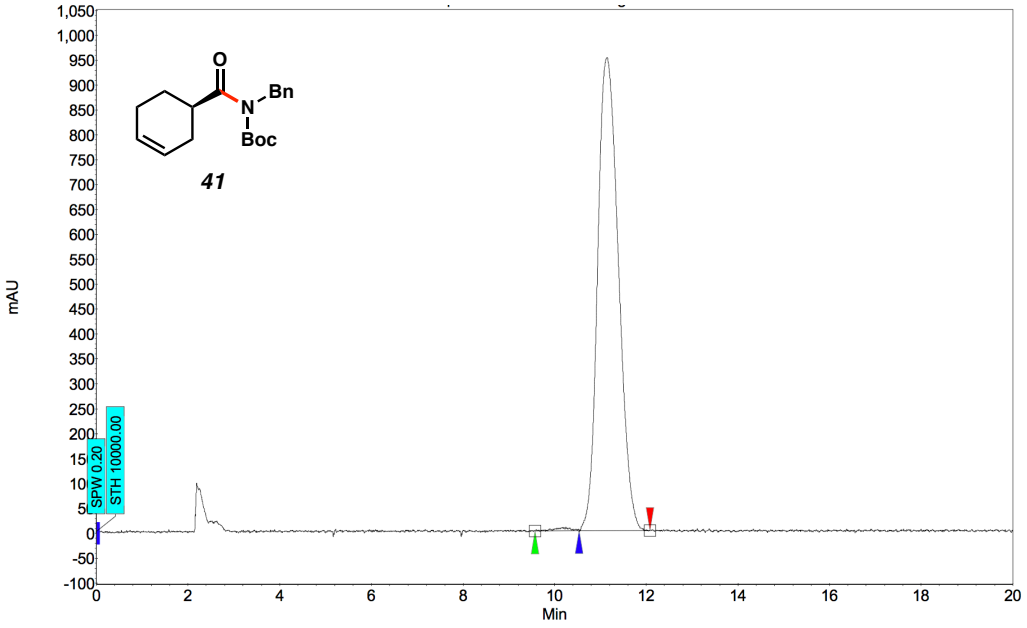
Ketone rac-42. Purification by flash chromatography (19:1 Hexanes:EtOAc → 14:1 Hexanes:EtOAc) generated ketone **rac-42** (81% yield, average of two experiments) as a clear oil. Spectral data match those previously reported (see S23).

b) Chiral SFC Assays

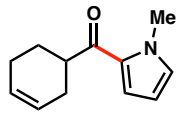
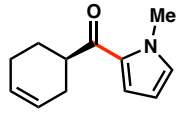
Compound	Method Column/Temp.	Solvent	Method Flow Rate	Retention Times (min)	Enantiomeric Ratio (er)
 *rac-41*	Daicel ChiralPak OJ-H/35 °C	1% isopropanol in CO_2	1 mL/min	9.29/10.63	50:50
 *41*	Daicel ChiralPak OJ-H/35 °C	1% isopropanol in CO_2	1 mL/min	9.57/10.54	99:1

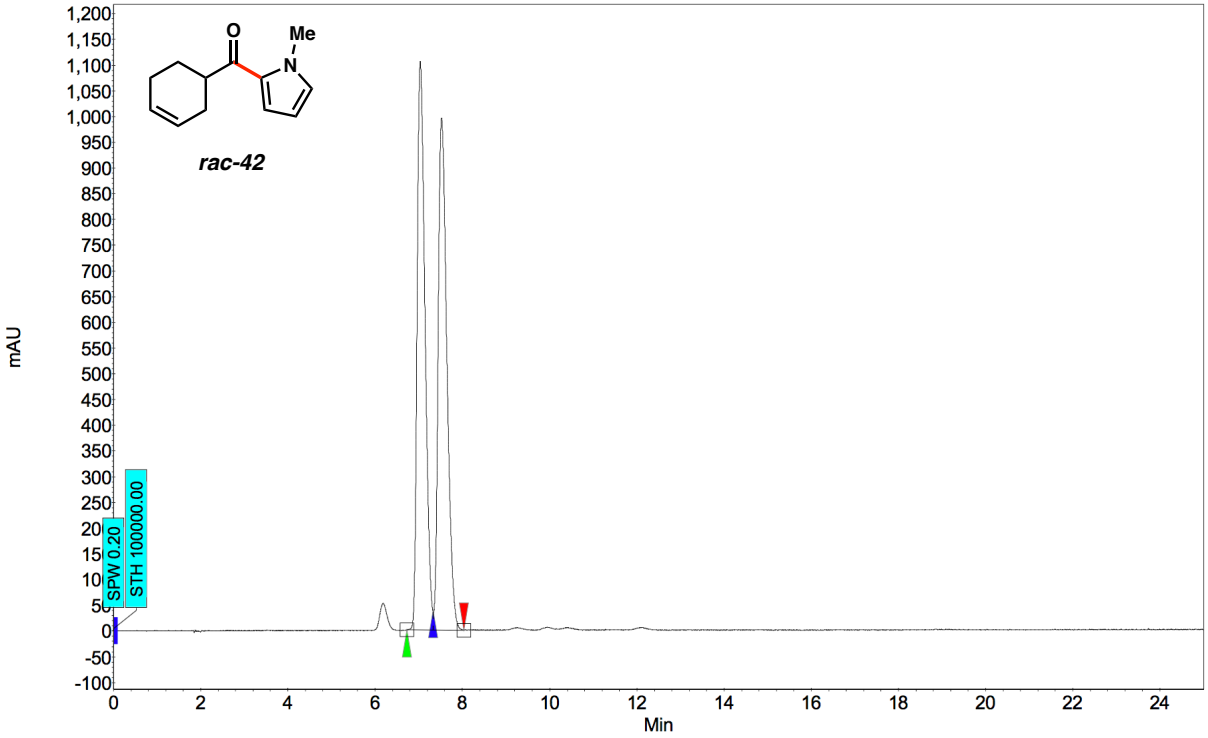


Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	9.29	10.10	10.63	0.00	50.11	449.1	223.3	50.106
2	UNKNOWN	10.63	11.18	12.08	0.00	49.89	422.0	222.4	49.894
Total						100.00	871.2	445.7	100.000

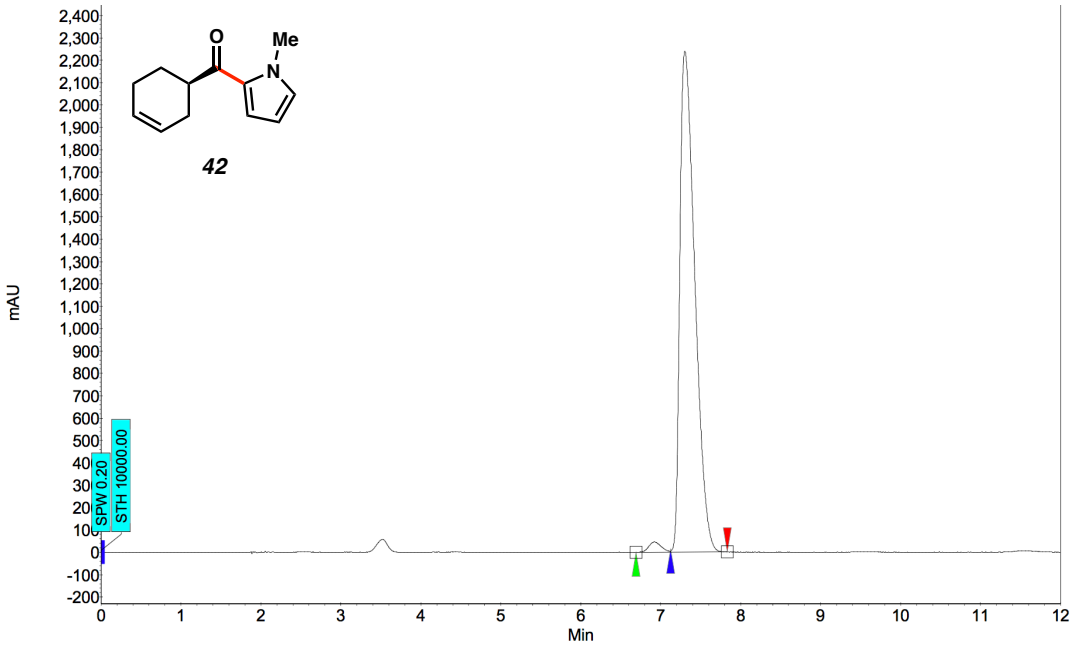


Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	9.57	10.18	10.54	0.00	0.56	7.1	2.8	0.556
2	UNKNOWN	10.54	11.15	12.08	0.00	99.44	950.0	498.7	99.444
Total						100.00	957.2	501.5	100.000

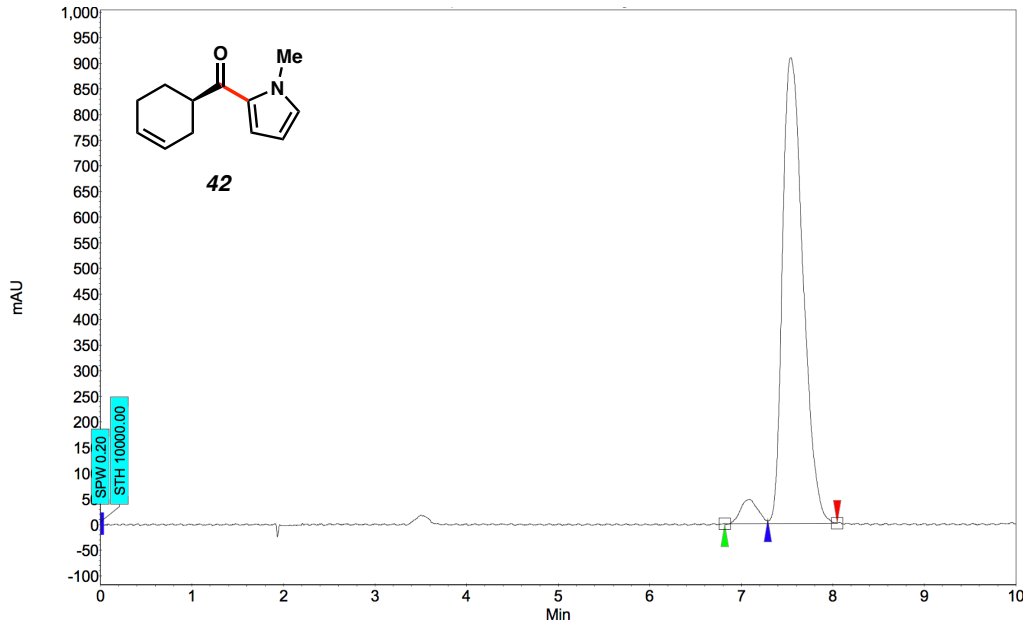
Compound	Method Column/Temp.	Solvent	Method Flow Rate	Retention Times (min)	Enantiomeric Ratio (er)
 ***rac-42***	Daicel ChiralPak OJ-H/35 °C	5% isopropanol in CO_2	2 mL/min	6.72/7.33	50:50
 ***42***	Daicel ChiralPak OJ-H/35 °C	5% isopropanol in CO_2	2 mL/min	6.69/7.12 6.82/7.29	99:2 96:4



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	6.72	7.03	7.33	0.00	50.11	1104.5	231.5	50.113
2	UNKNOWN	7.33	7.52	8.04	0.00	49.89	994.7	230.5	49.887
Total						100.00	2099.2	462.0	100.000

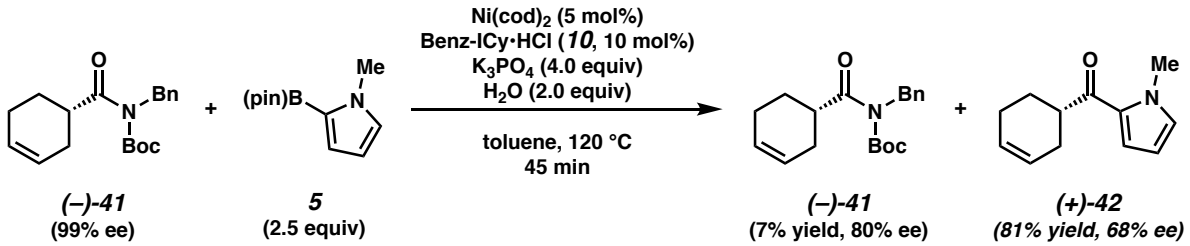


Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	6.69	6.92	7.12	0.00	1.66	46.1	8.2	1.656
2	UNKNOWN	7.12	7.30	7.83	0.00	98.34	2239.4	485.6	98.344
Total						100.00	2285.5	493.8	100.000



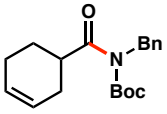
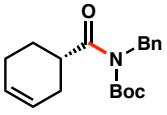
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	6.82	7.08	7.29	0.00	4.17	47.1	10.2	4.168
2	UNKNOWN	7.29	7.54	8.05	0.00	95.83	908.7	235.6	95.832
Total						100.00	955.8	245.9	100.000

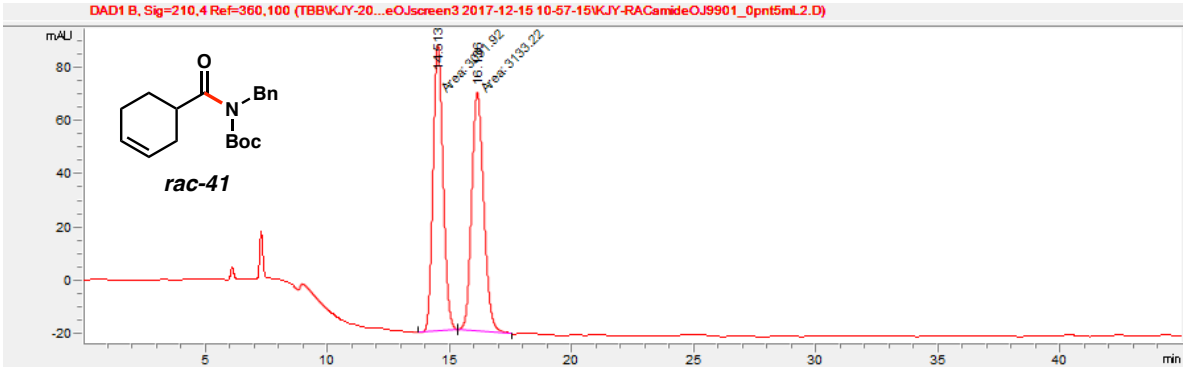
E. Erosion of Stereochemistry Control Experiments



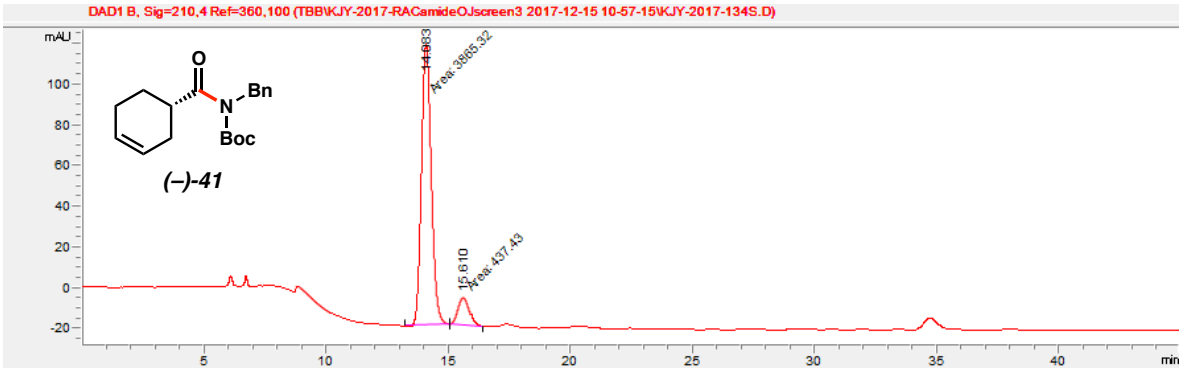
Amide 41 & Ketone 42. Purification by flash chromatography (Hexanes → 49:1 Hexanes:EtOAc → 24:1 Hexanes:EtOAc → 16:1 Hexanes:EtOAc) afforded recovered amide substrate **41** in 80% ee and ketone **42** in 68% ee (the reported yield was based on ^{1}H NMR analysis using hexamethylbenzene as an external standard) as clear oils. Spectral data match those previously reported (see S23).

b) Chiral HPLC Assays

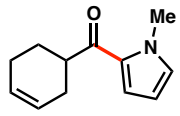
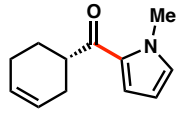
Compound	Method Column/Temp.	Solvent	Method Flow Rate	Retention Times (min)	Enantiomeric Ratio (er)
 *rac-41*	Daicel ChiralPak OJ-H/23 °C	1% isopropanol in hexanes	1 mL/min	14.51/16.14	50:50
 *(–)-41*	Daicel ChiralPak OJ-H/23 °C	1% isopropanol in hexanes	1 mL/min	14.08/15.61	90:10

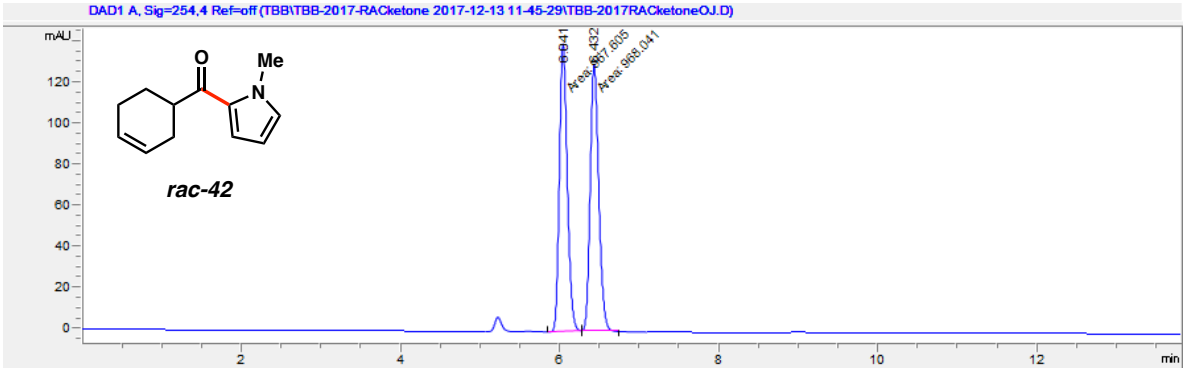


#	Time	Area	Height	Width	Area%	Symmetry
1	14.513	3091.9	108.2	0.4765	49.668	0.859
2	16.136	3133.2	90.2	0.5787	50.332	0.827

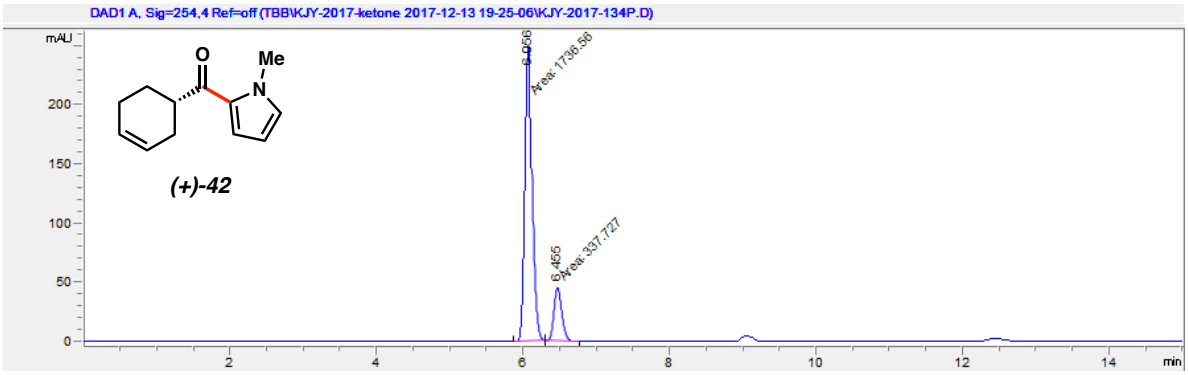


#	Time	Area	Height	Width	Area%	Symmetry
1	14.083	3865.3	138.1	0.4666	89.834	0.841
2	15.61	437.4	13.6	0.5372	10.166	0.848

Compound	Method Column/Temp.	Solvent	Method Flow Rate	Retention Times (min)	Enantiomeric Ratio (er)
 *rac-42*	Daicel ChiralPak OJ-H/23 °C	10% isopropanol in hexanes	1 mL/min	6.04/6.43	50:50
 *(+)-42*	Daicel ChiralPak OJ-H/23 °C	10% isopropanol in hexanes	1 mL/min	6.05/6.46	84:16

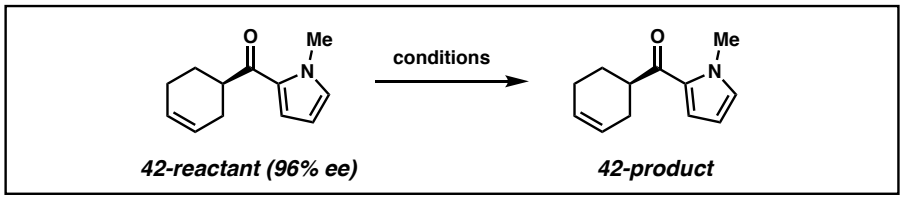


#	Time	Area	Height	Width	Area%	Symmetry
1	6.041	967.6	140.3	0.1149	49.989	0.799
2	6.432	968	129.5	0.1246	50.011	0.805



#	Time	Area	Height	Width	Area%	Symmetry
1	6.056	1736.6	249.8	0.1159	83.718	0.788
2	6.455	337.7	45.3	0.1243	16.282	0.807

Table S2. Evaluation of Impact of Reaction Components on Erosion of α-Stereocenter[a]

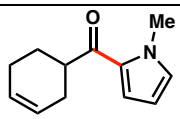
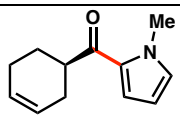
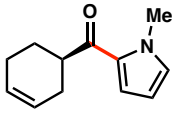


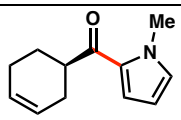
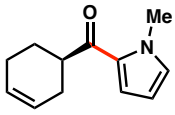
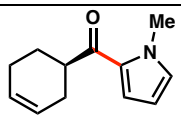
42-reactant (96% ee) *42-product*

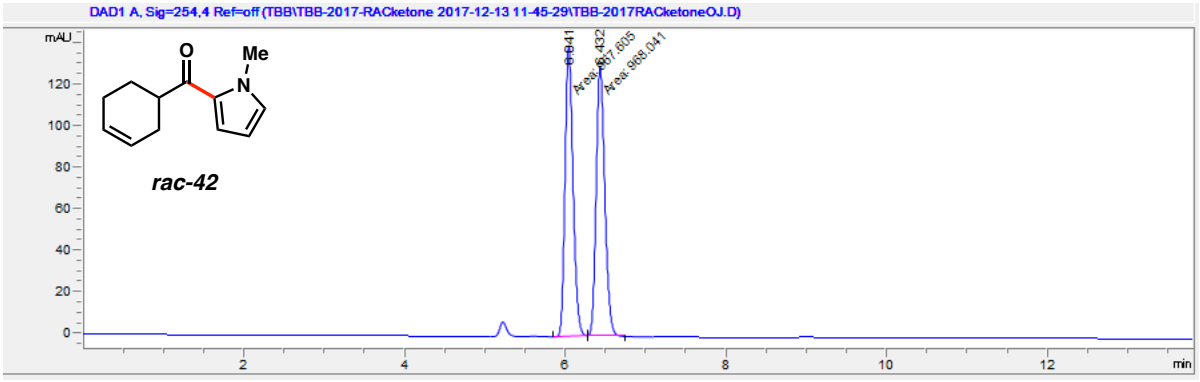
Entry	*Control Experiment Conditions*	*Experimental Results ee of 42-product*
1	K_3PO_4 (4.0 equiv), H_2O (2.0 equiv) toluene (1.0 M), 120 °C, 4 h	88%
2	$Ni(cod)_2$ (5 mol%) toluene (1.0 M), 120 °C, 16 h	92%
3	Benz-ICy·HCl (*10*, 10 mol%) toluene (1.0 M), 120 °C, 4 h	96%
4	$Ni(cod)_2$ (5 mol%), Benz-ICy·HCl (*10*,10 mol%), NaOtBu (9 mol%) toluene (1.0 M), 120 °C, 4 h	51%[a]
5	Benz-ICy·HCl (*10*,10 mol%), NaOtBu (9 mol%) toluene (1.0 M), 120 °C, 4 h	0%[b]

[a] $Ni(cod)_2$, Benz-ICy·HCl, and NaOtBu were stirred for 1 h in toluene at 23 °C to generate active catalyst prior to addition to ketone substrate. [b] Benz-ICy·HCl and NaOtBu were stirred for 1 h in toluene at 23 °C to generate free NHC prior to addition to ketone substrate.

b) Chiral HPLC Assays

Compound	Control Experiment Entry	Method Column/ Temp.	Solvent	Method Flow Rate	Retention Times (min)	Enantiomeric Ratio (er)
 *rac-42*	-	Daicel ChiralPak OJ-H/23 °C	10% isopropanol in hexanes	1 mL/min	6.041/6.432	50:50
 *42*	1	Daicel ChiralPak OJ-H/23 °C	10% isopropanol in hexanes	1 mL/min	6.063/6.455	6:94
 *42*	2	Daicel ChiralPak OJ-H/23 °C	10% isopropanol in hexanes	1 mL/min	6.064/6.456	4:96

 ***42***	3	Daicel ChiralPak OJ-H/23 °C	10% isopropanol in hexanes	1 mL/min	6.073/6.464	2:98
 ***42***	4	Daicel ChiralPak OJ-H/23 °C	10% isopropanol in hexanes	1 mL/min	6.074/6.466	24:76
 ***42***	5	Daicel ChiralPak OJ-H/23 °C	10% isopropanol in hexanes	1 mL/min	6.092/6.488	50:50



#	Time	Area	Height	Width	Area%	Symmetry
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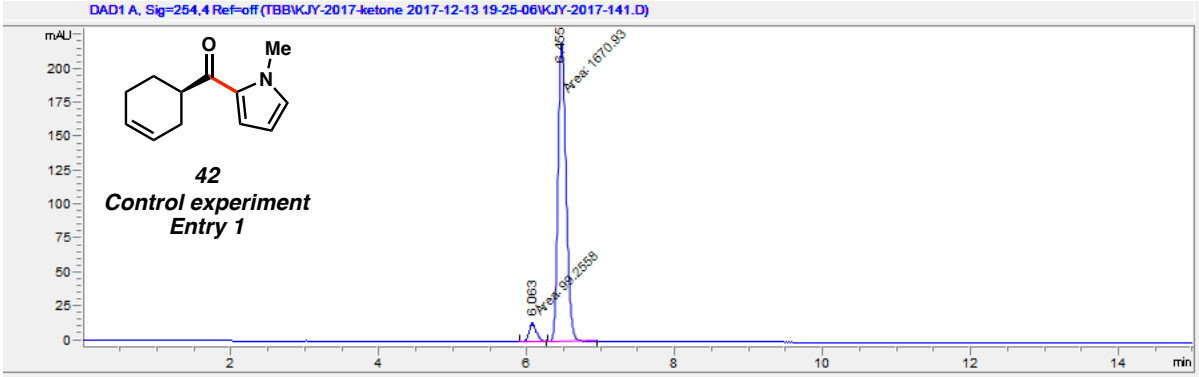
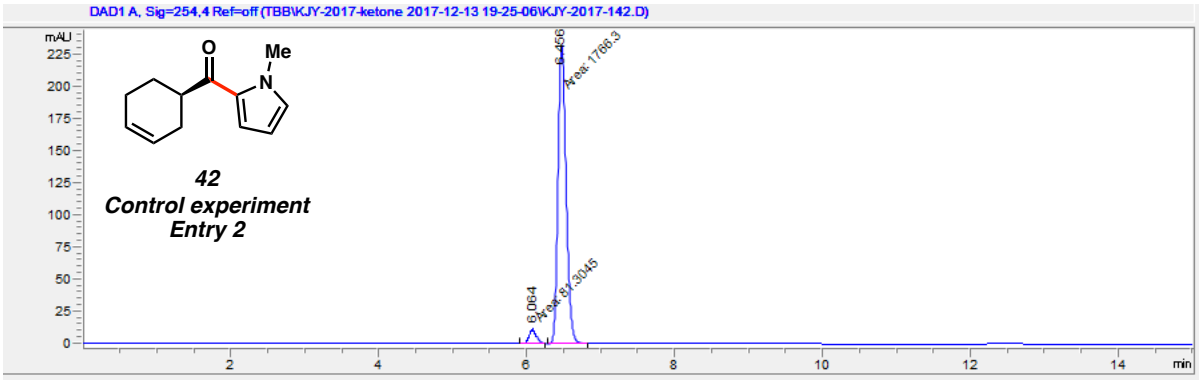
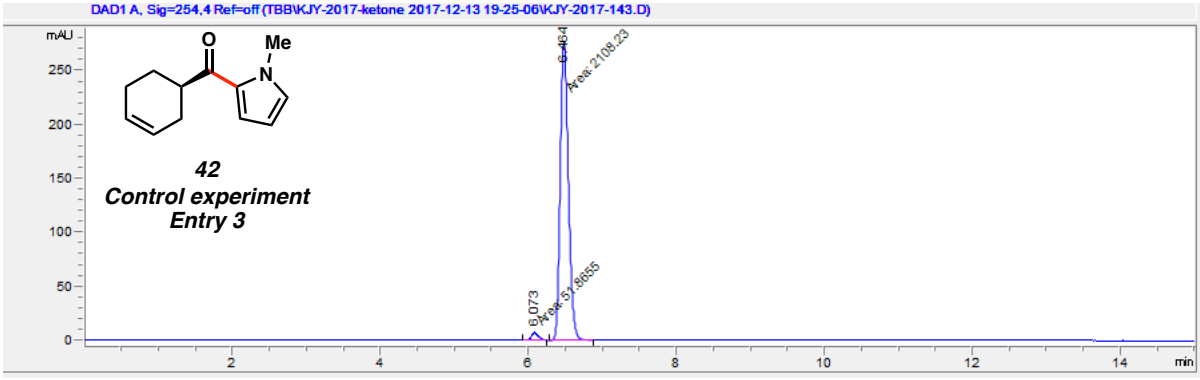
2	6.432	968	129.5	0.1246	50.011	0.805



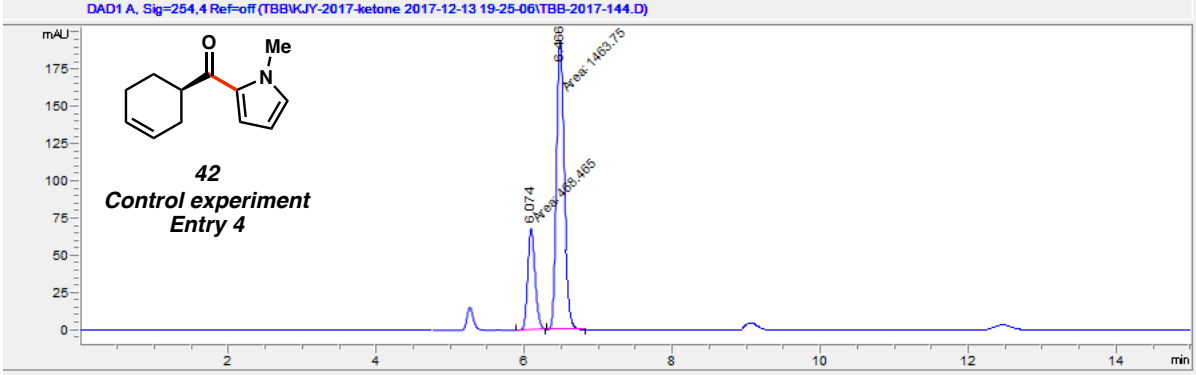
#	Time	Area	Height	Width	Area%	Symmetry
1	6.063	99.3	14.1	0.1172	5.607	0.819
2	6.455	1670.9	219.9	0.1267	94.393	0.799



#	Time	Area	Height	Width	Area%	Symmetry
1	6.064	81.3	11.6	0.1168	4.401	0.798
2	6.456	1766.3	232.5	0.1266	95.599	0.798

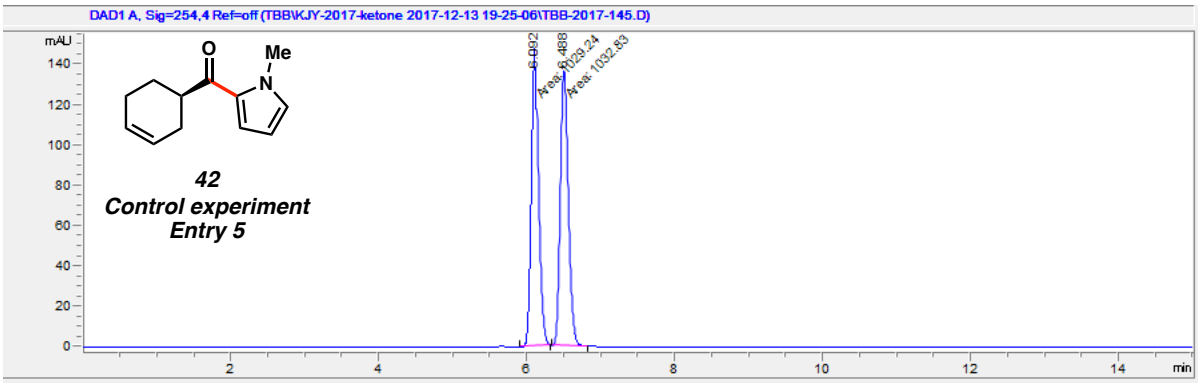


#	Time	Area	Height	Width	Area%	Symmetry
1	6.073	51.9	7.6	0.1144	2.401	0.839
2	6.464	2108.2	276.1	0.1273	97.599	0.796



42
Control experiment
Entry 4

#	Time	Area	Height	Width	Area%	Symmetry
1	6.074	468.5	67.7	0.1154	24.245	0.808
2	6.466	1463.8	193.6	0.126	75.755	0.799

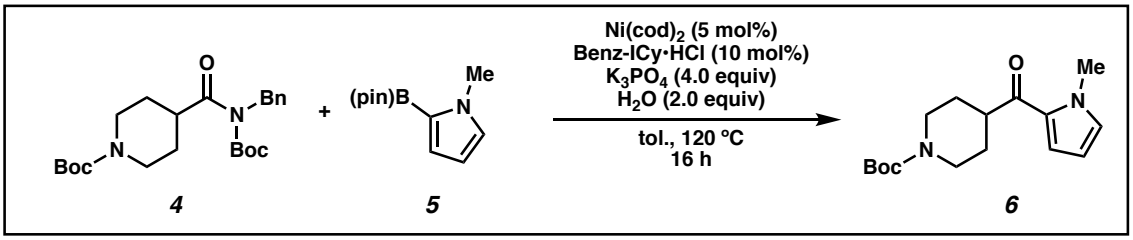


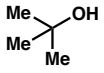
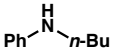
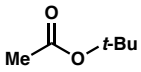
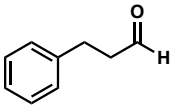
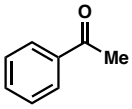
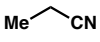
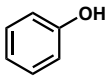
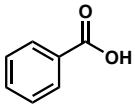
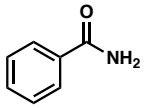
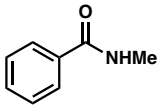
42
Control experiment
Entry 5

#	Time	Area	Height	Width	Area%	Symmetry
1	6.092	1029.2	147.7	0.1161	49.913	0.796
2	6.488	1032.8	136.8	0.1259	50.087	0.801

F. Robustness Screen

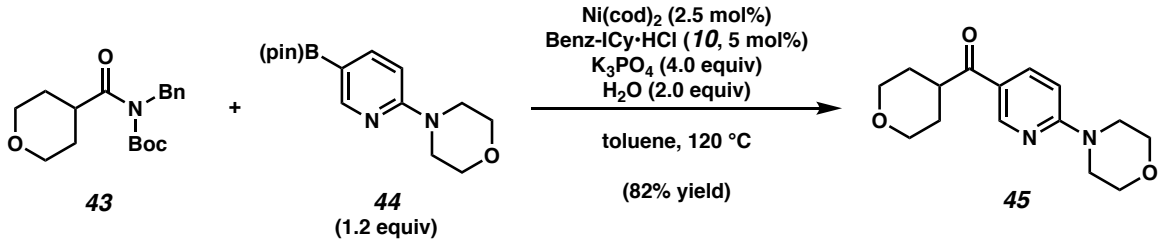
Table S2. Evaluation of Functional Group Compatibility in the Suzuki Reaction[a]



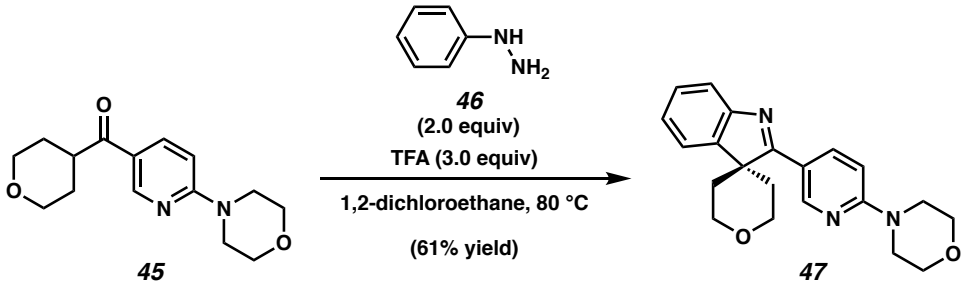
Entry	Additive	Yield of *6* (%)	Additive Remaining (%)	SM Remaining (%)
1	None	95	N.D.	0
2		70	N.D.[b]	0
3		58	73	0
4		66	N.D.[b]	0
5		0	8	46
6		67	73	0
7		0	N.D.[b]	0
8		0	42	0
9		68	0	0
10		0	30	0
11		66	66	0
12		71	4	0
13		26	N.D.[b]	0

[a] Conditions: $Ni(cod)_2$ (5 mol%), Benz-ICy·HCl (10 mol%), substrate (1.0 equiv), PhB(pin) (2.5 equiv), K_3PO_4 (4.0 equiv), toluene (1.0 M), H_2O (2.0 equiv), and additive (1.0 equiv) at 120 °C for 16 h. Yields of coupled product, remaining additive, and remaining starting material were determined by ^1H NMR analysis using hexamethylbenzene as an internal standard. [b] Not determined due to low boiling point.

G. Gram Scale Suzuki–Miyaura Reaction and Subsequent Fischer Indolization



Ketone 45. A 20 mL scintillation vial was charged with anhydrous powdered K_3PO_4 (2.66 g, 12.5 mmol, 4.0 equiv) and a magnetic stir bar. The vial and contents were flame-dried under reduced pressure, then allowed to cool under N_2. Amide substrate **43** (1.00 g, 3.14 mmol, 1.0 equiv) and 2-morpholinopyridine-5-boronic acid pinacol ester (**44**) (1.09 g, 3.76 mmol, 1.2 equiv) were added. The vial was flushed with N_2, then water (113 μL, 6.27 mmol, 2.0 equiv), which had been sparged with N_2 for 10 min, was added. The vial was taken into a glove box and charged with $Ni(cod)_2$ (21.6 mg, 0.0784 mmol, 2.5 mol%) and Benz-ICy•HCl (**10**, 50.0 mg, 0.157 mmol, 5 mol%). Subsequently, toluene (3.14 mL, 1.0 M) was added. The vial was sealed with a Teflon-lined screw cap, removed from the glove box, and stirred vigorously (800 rpm) at 120 °C for 16 h. After cooling to 23 °C, the mixture was diluted with hexanes (7 mL) and filtered over a plug of silica gel (100 mL of EtOAc eluent). The volatiles were removed under reduced pressure, and the crude residue was purified by flash chromatography (3:1 Hexanes:EtOAc → 19:1 CH_2Cl_2:MeOH) to yield ketone product **45** (707 mg, 82% yield) as an off-white solid. Ketone **45**: mp: 122–124 ºC; R_f 0.36 (4:1 PhH:CH_3CN); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.79 (d, J = 2.2, 1H), 8.06 (dd, J = 9.1, 2.4, 1H), 6.63 (d, J = 9.1, 1H), 4.09–4.02 (m, 2H), 3.84–3.78 (m, 4H), 3.71–3.65 (m, 4H), 3.54 (td, J = 11.7, 2.2, 2H), 3.37 (tt, J = 11.2, 3.8, 1H), 1.96–1.84 (m, 2H), 1.79–1.71 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 199.2, 160.7, 150.4, 137.9, 121.5, 105.9, 67.5, 66.7, 45.0, 42.4, 29.3; IR (film): 2955, 2920, 2850, 1663, 1596 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{15}H_{21}N_2O_3$, 277.15467; found 277.15256.



Indolenine 47. A 20 mL scintillation vial was charged with ketone **45** (707 mg, 2.56 mmol, 1.0 equiv) and a magnetic stir bar. Subsequently, 1,2-dichloroethane (12.0 mL, 0.21 M), phenylhydrazine **46** (503 µL, 5.12 mmol, 2.0 equiv), and TFA (588 µL, 7.69 mmol, 3.0 equiv) were added. The vial was sealed with a Teflon-lined screw cap and stirred at 80 °C for 16 h. After cooling to 23 °C, the volatiles were removed under reduced pressure, and the crude residue was purified by flash chromatography (3:1 Hexanes:EtOAc → 1:1 Hexanes:EtOAc → 100% EtOAc) to yield indolenine **47** (546 mg, 61% yield) as a tan solid. Indolenine **47**: mp: 186–189 ºC; R_f 0.26 (4:1 PhH:CH_3CN); ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.11 (d, J = 2.2, 1H), 8.49 (dd, J = 9.1, 2.5, 1H), 7.92 (d, J = 7.4, 1H), 7.69 (d, J = 7.3, 1H), 7.41 (td, J = 7.6, 1.1, 1H), 7.22 (td, J = 7.5, 1.1, 1H), 6.73 (d, J = 9.1, 1H), 4.23–4.08 (m, 4H), 3.87–3.81 (m, 4H), 3.70–3.64 (m, 4H), 2.77–2.67 (m, 2H), 1.36 (d, J = 14.1, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 179.2, 159.5, 154.1, 148.9, 145.9, 138.2, 128.3, 124.8, 123.6, 121.2, 118.4, 106.4, 66.8, 64.0, 54.5, 45.2, 31.6; IR (film): 2960, 2921, 2858, 1596, 1499 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{21}H_{24}N_3O_2$, 350.18630; found 350.18529.

References

[1] Fier, P. S.; Luo, J.; Hartwig, J. F. *J. Am. Chem. Soc.* **2013**, *135*, 2552–2559.

[2] Hie, L.; Baker, E. L.; Anthony, S. M.; Desrosiers, J. –N.; Senanayake, C.; Garg, N. K. *Angew. Chem. Int. Ed.* **2016**, *55*, 15129–15132.

[3] Weires, N. A.; Baker, E. L.; Garg, N. K. *Nat. Chem*. **2016**, *8*, 75–79.

[4] Dander, J. E.; Baker, E. L.; Garg, N. K. *Chem. Sci*. **2017**, *8*, 6433–6438.

[5] Allwood, D. M.; Blakemore, D. C.; Ley, S. V. *Org. Lett.* **2014**, *16*, 3064–3067.

[6] Takemiya, A.; Hartwig, J. F. *J. Am. Chem. Soc*. **2006**, *128*, 14800–14801.

[7] Bechara, W. S.; Pelletier, G.; Charette, A. B. *Nat. Chem*. **2012**, *4*, 228–234.

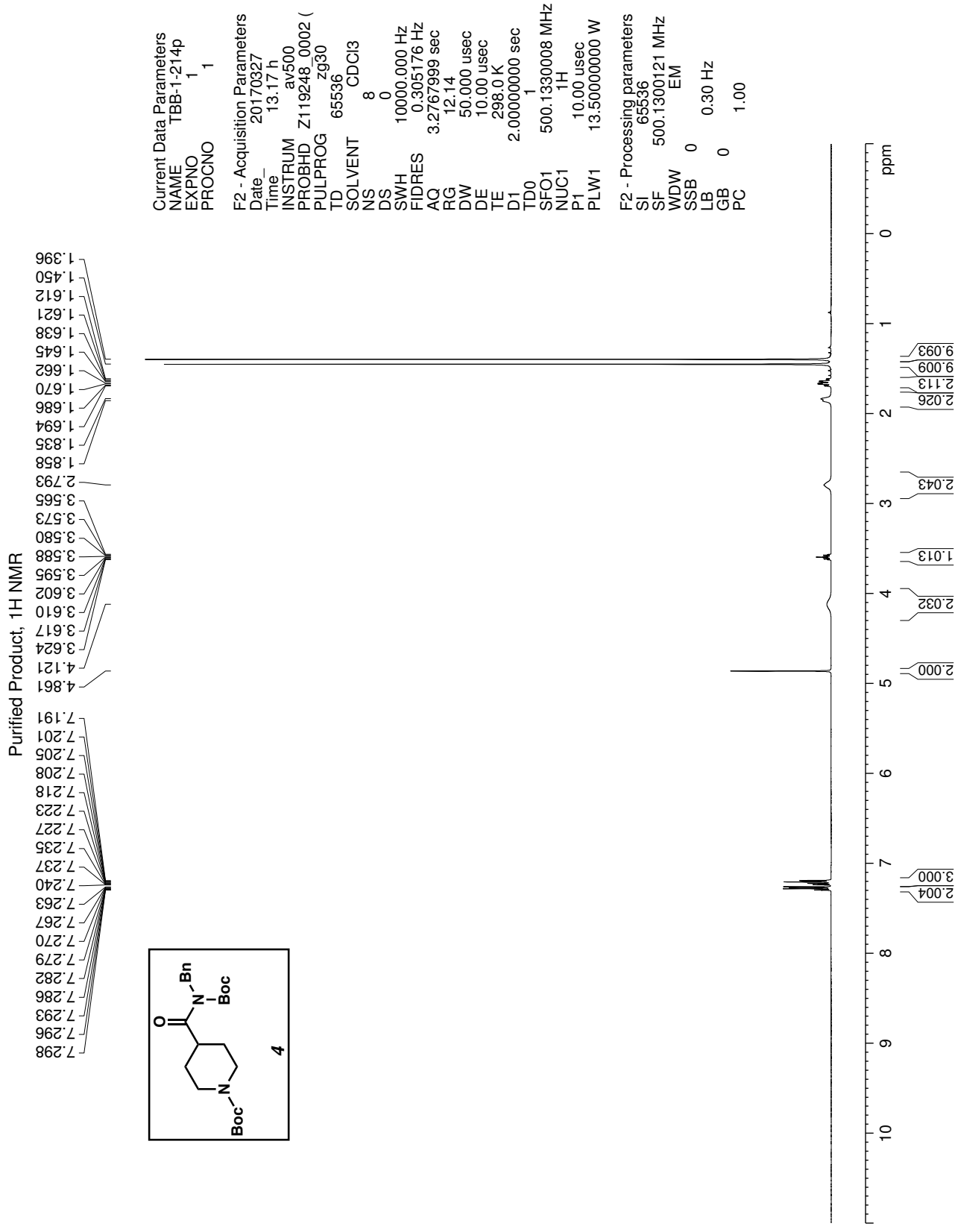
[8] Wakeham, R. J.; Taylor, J. E.; Bull, S. D.; Morris, J. A.; Williams, J. M. J. *Org. Lett.* **2013**, *15*, 702–705.

[9] Clerici, F.; Gelmi, M. L.; Rossi, L. M. *Synthesis* **1987**, 1025–1027.

[10] Barbero, M.; Cadamuro, S.; Degani, I.; Fochi, R.; Gatti, A.; Regondi, V. *J. Org. Chem.* **1988**, *53*, 2245–2250.

[11] Taylor, J. E.; Jones, M. D.; Williams, J. M. J.; Bull, S. D. *Org. Lett.* **2010**, *12*, 5740–5743.

^{1}H NMR Spectra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FIDRES 0.305176 Hz
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DW 50.000 usec
DE 10.00 usec
TE 298.0 K
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TD0 1
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P1 10.00 usec
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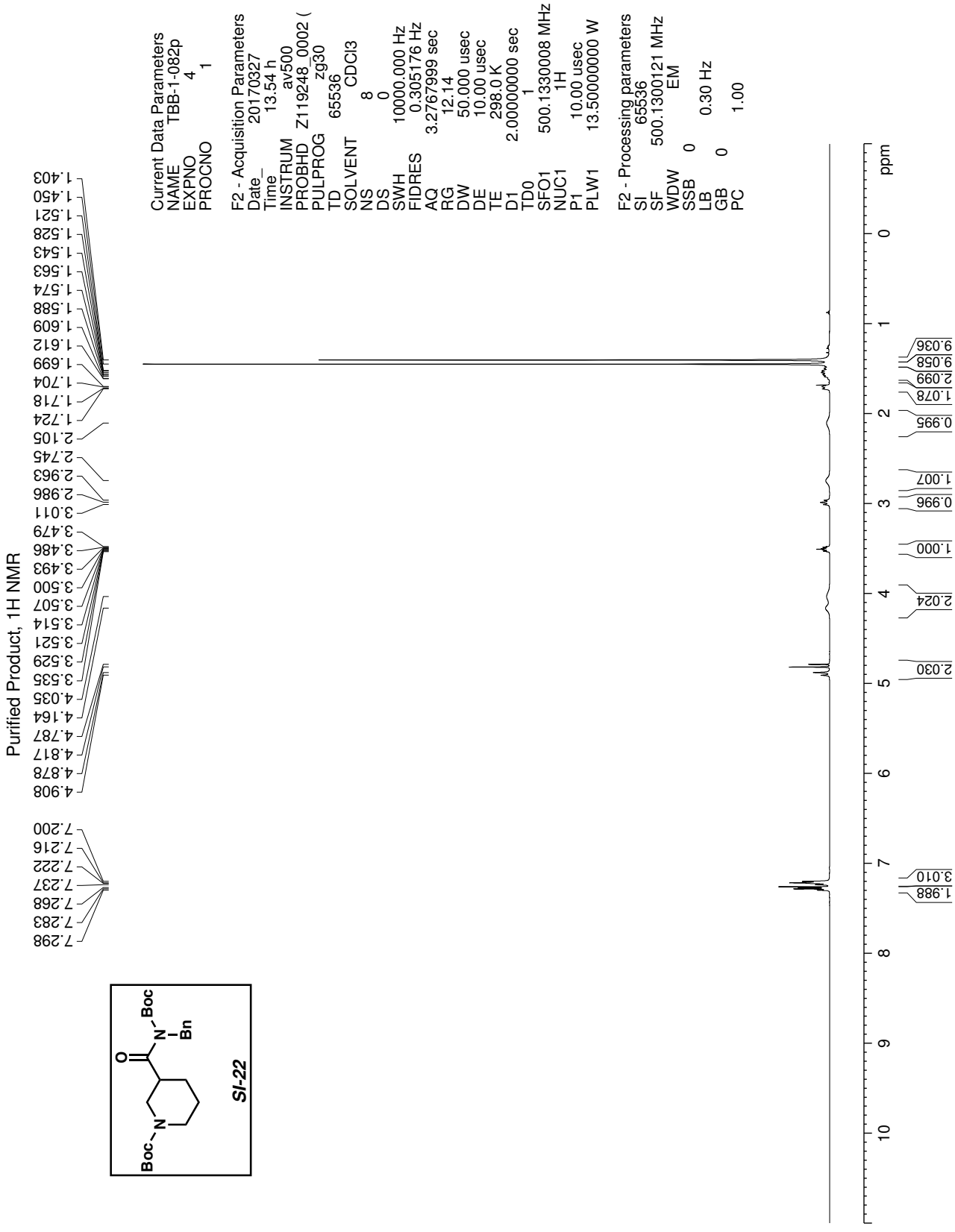
ppm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TD0 1
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NUC1 1H
P1 10.00 usec
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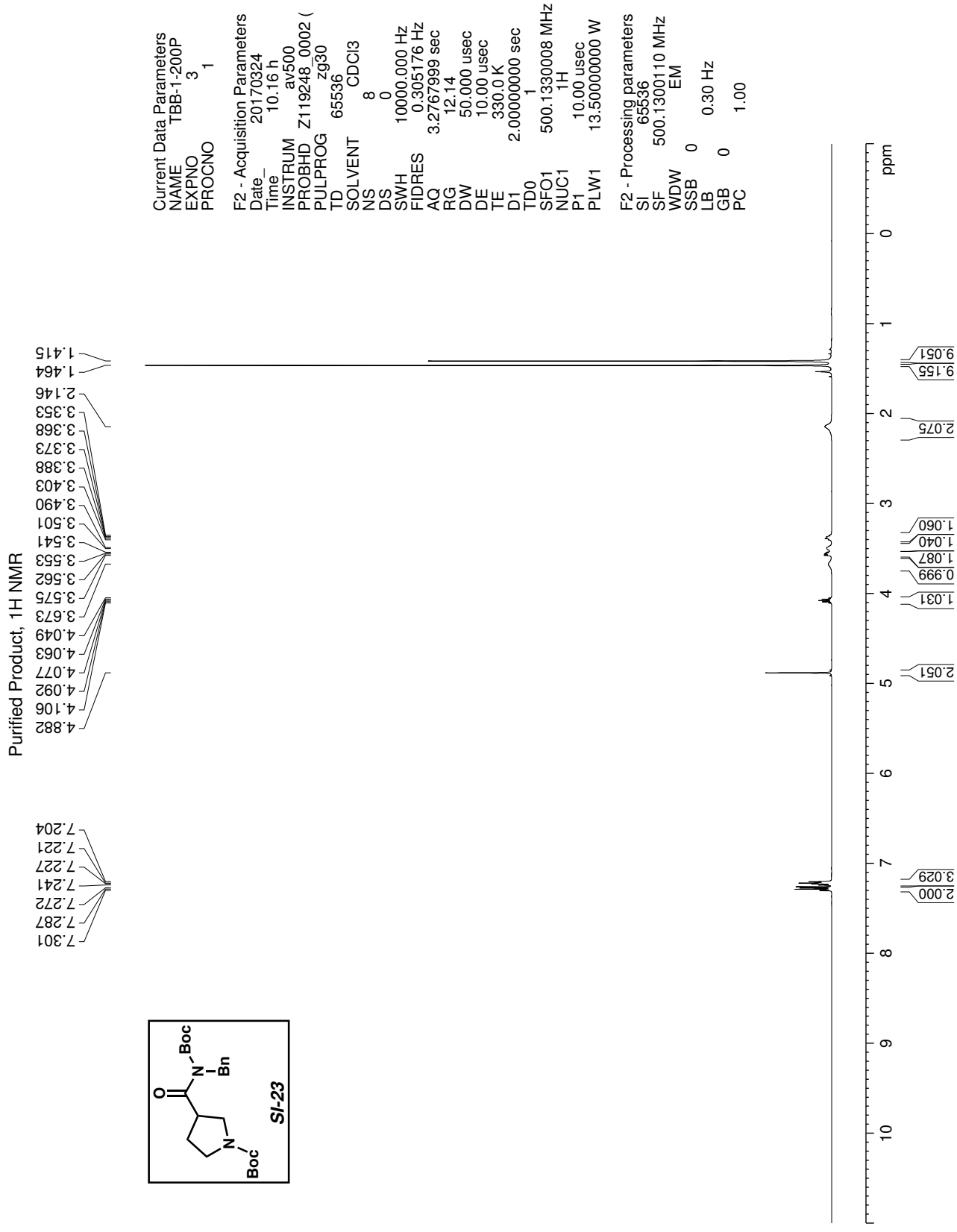
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Bn
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Boc
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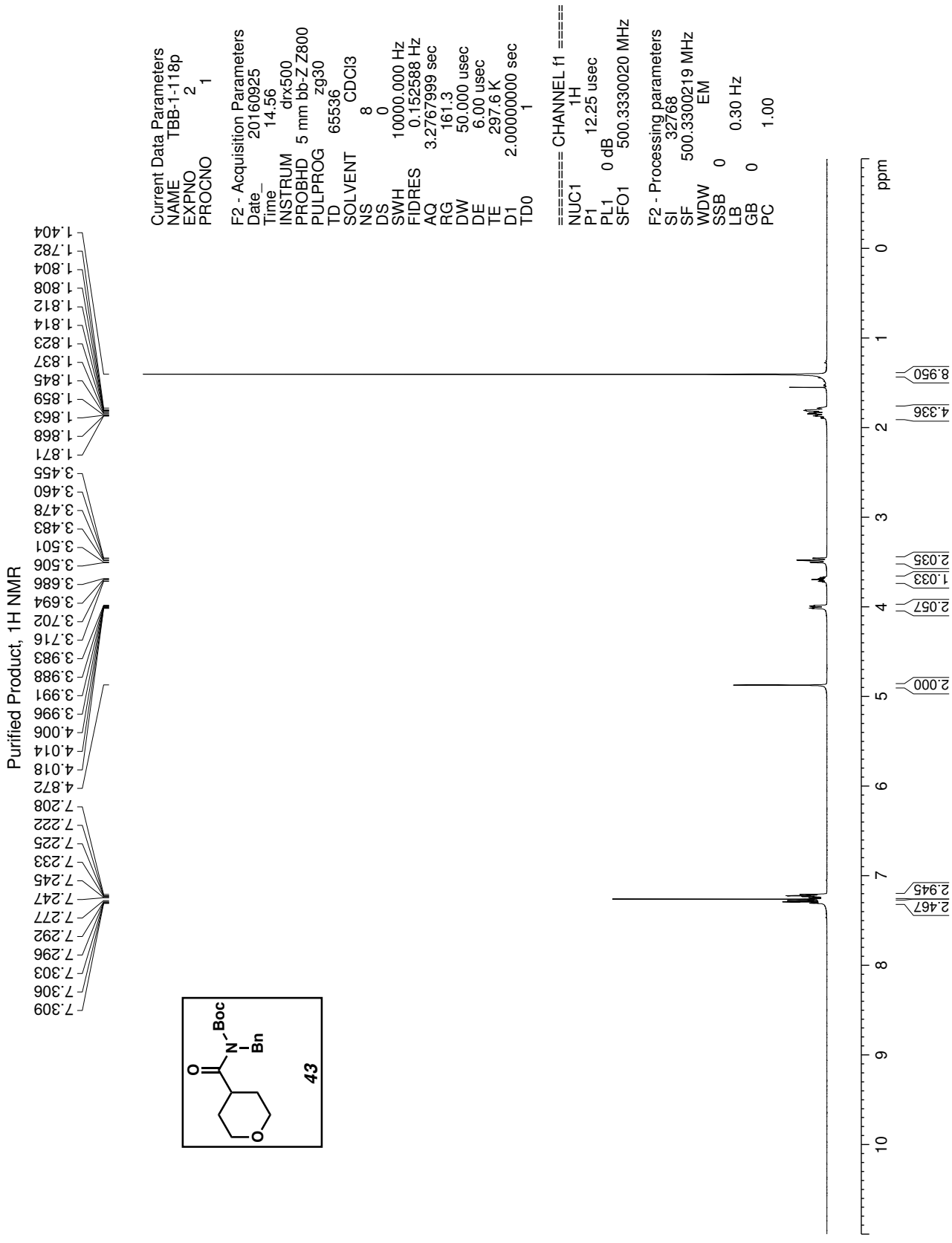
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7.247
7.245
7.233
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4.006
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3.991
3.988
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Bn
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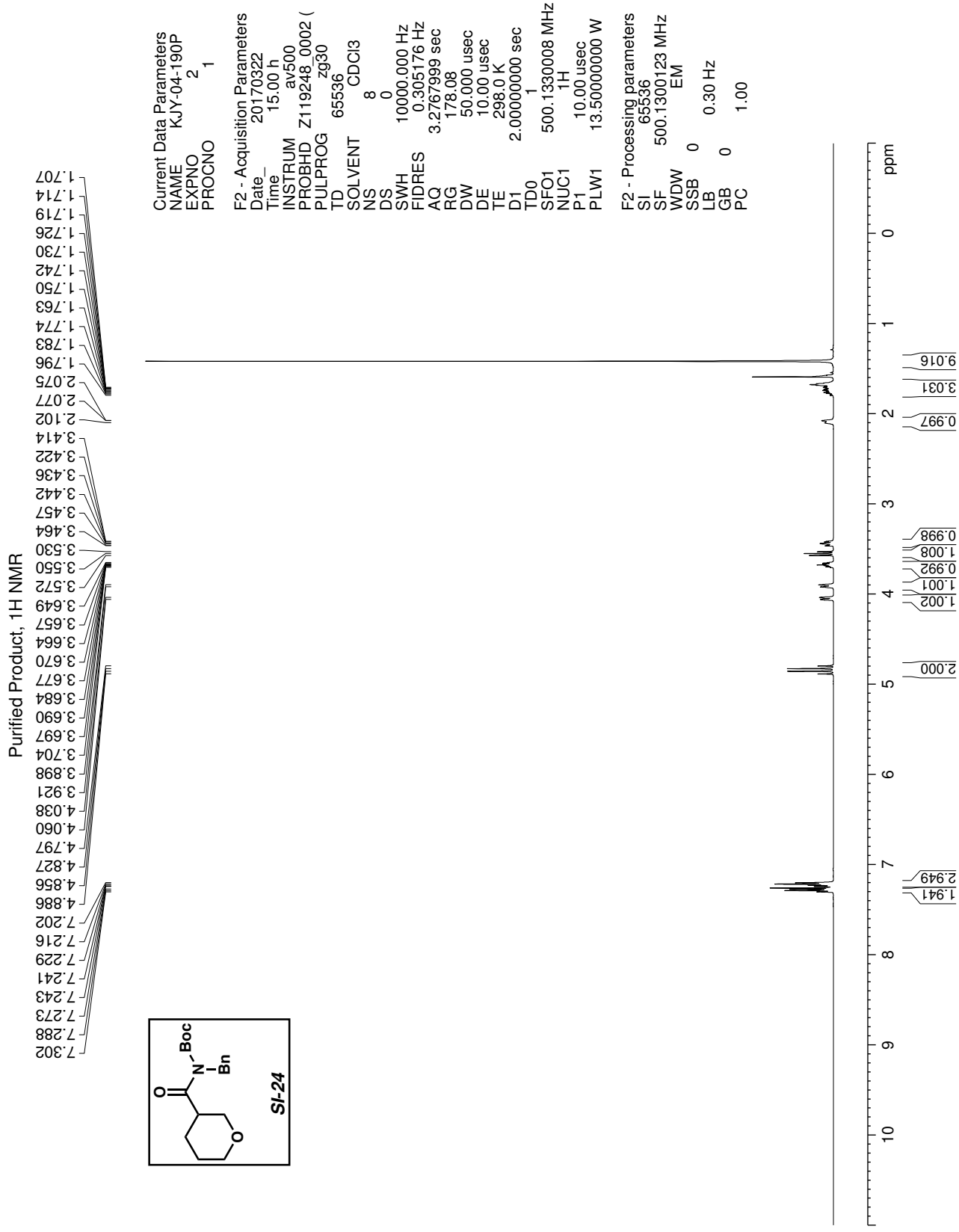
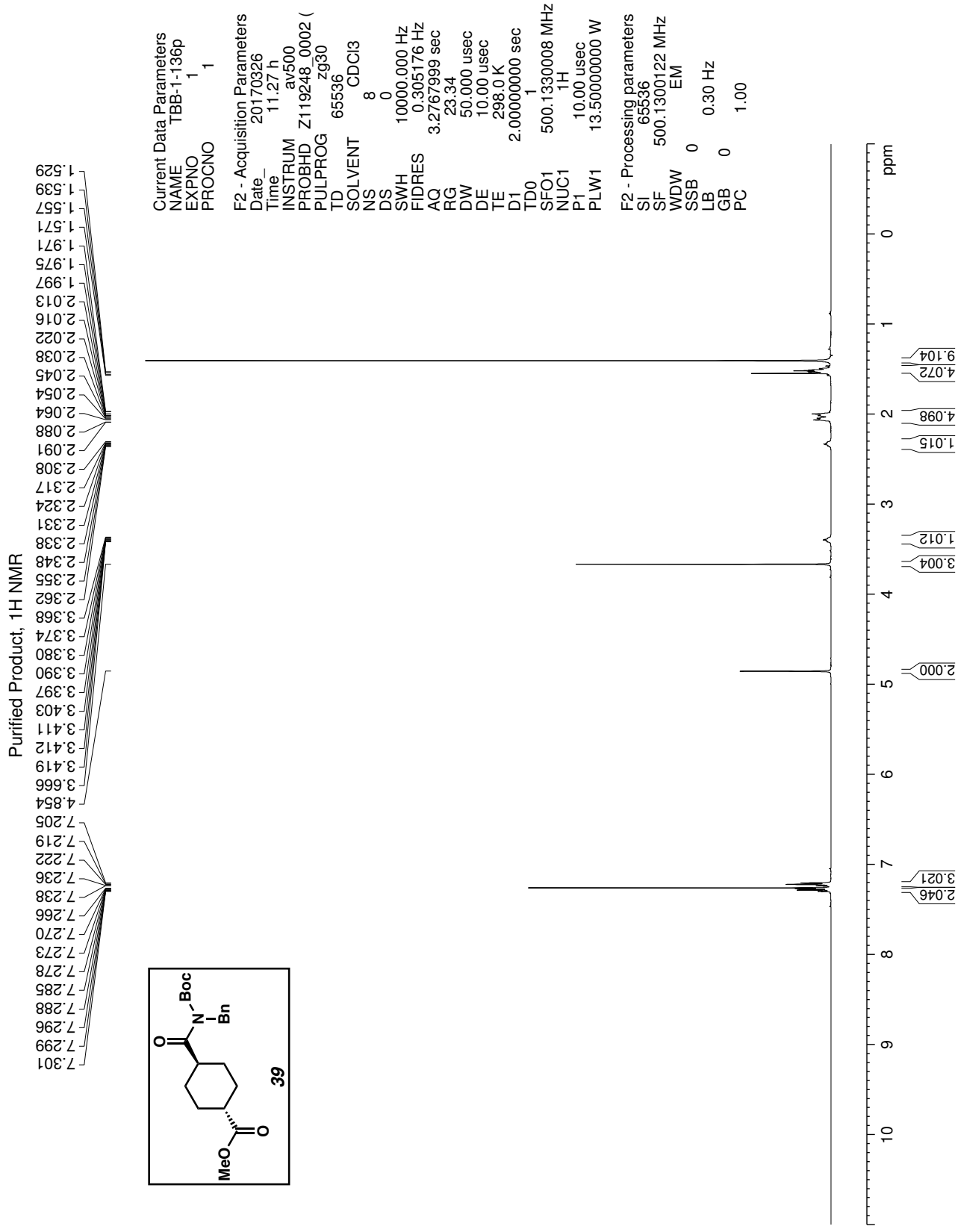
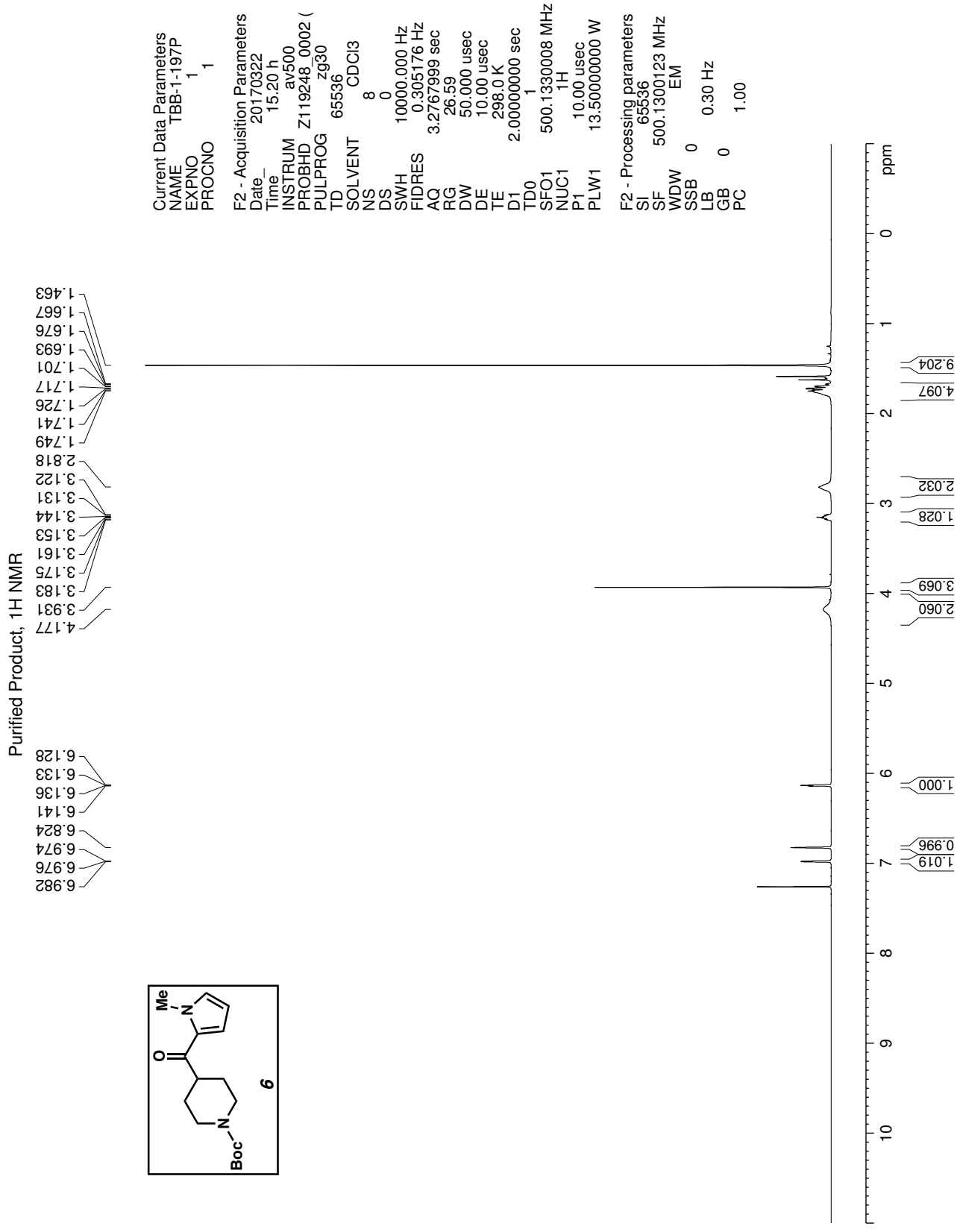
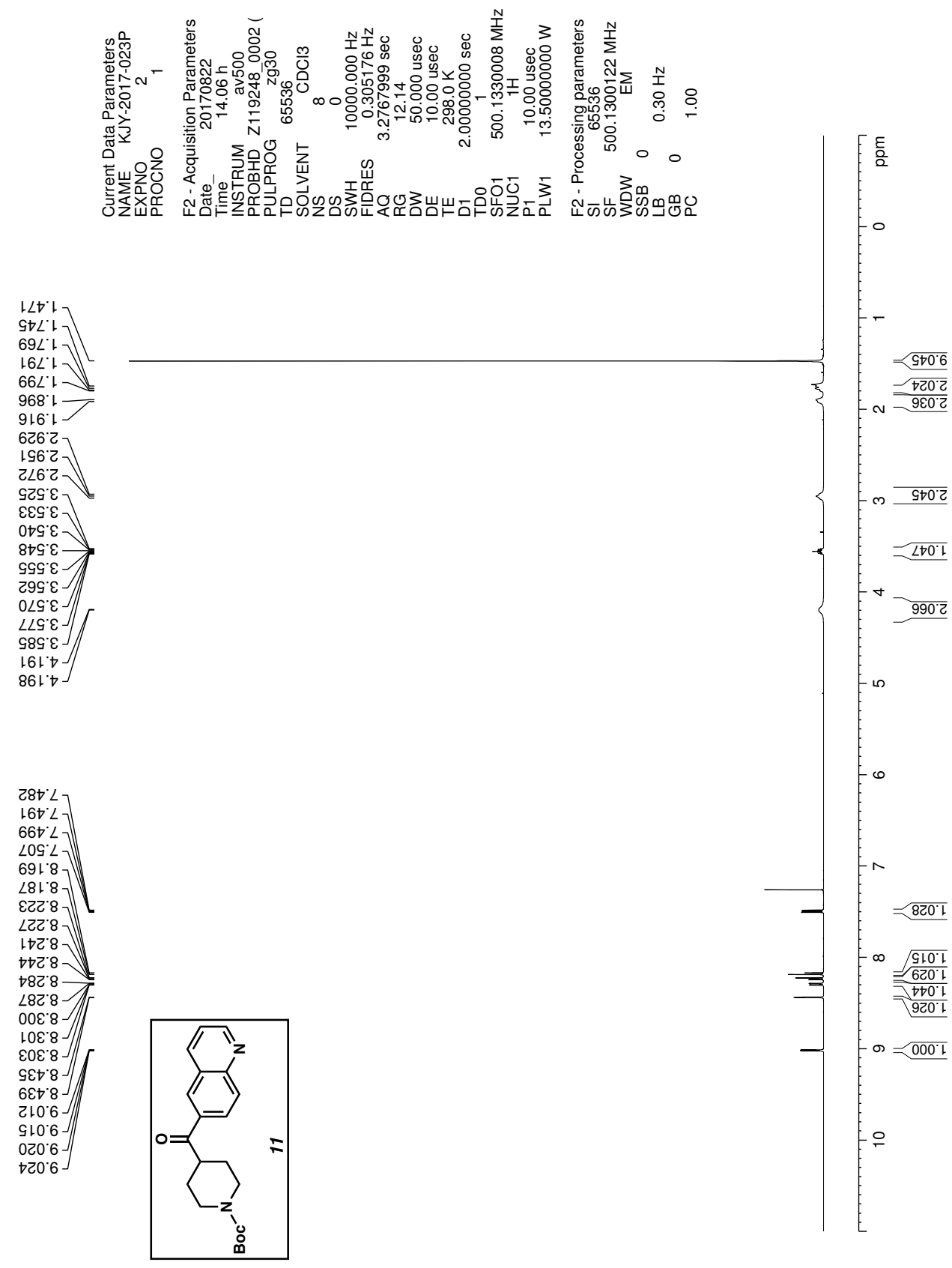
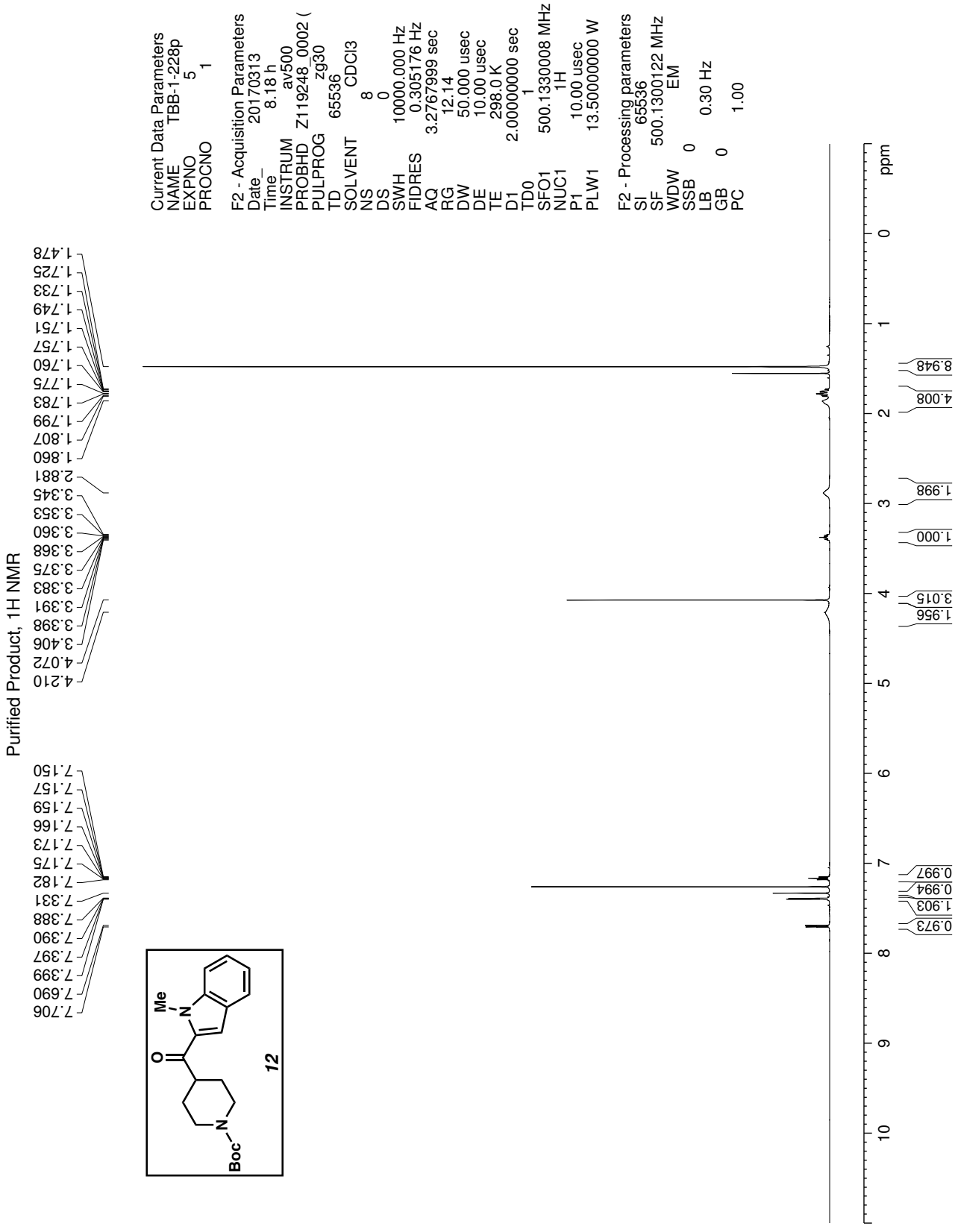
O
43

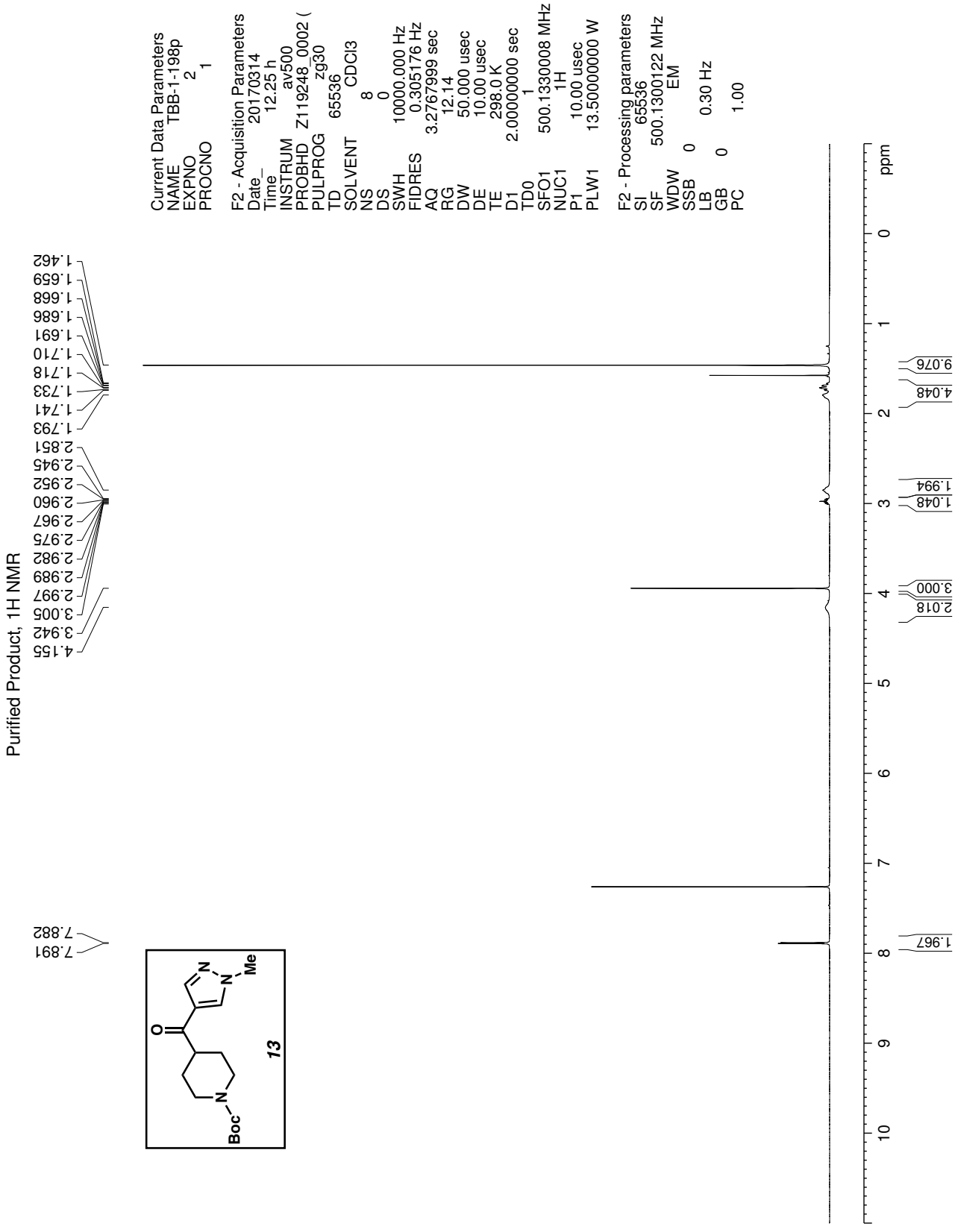

Purified Product, 1H NMR
Current Data Parameters
NAME KJY-04-190P
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170322
Time_ 15.00 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 178.08
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Boc
N
Bn
O
O
SI-24
ppm

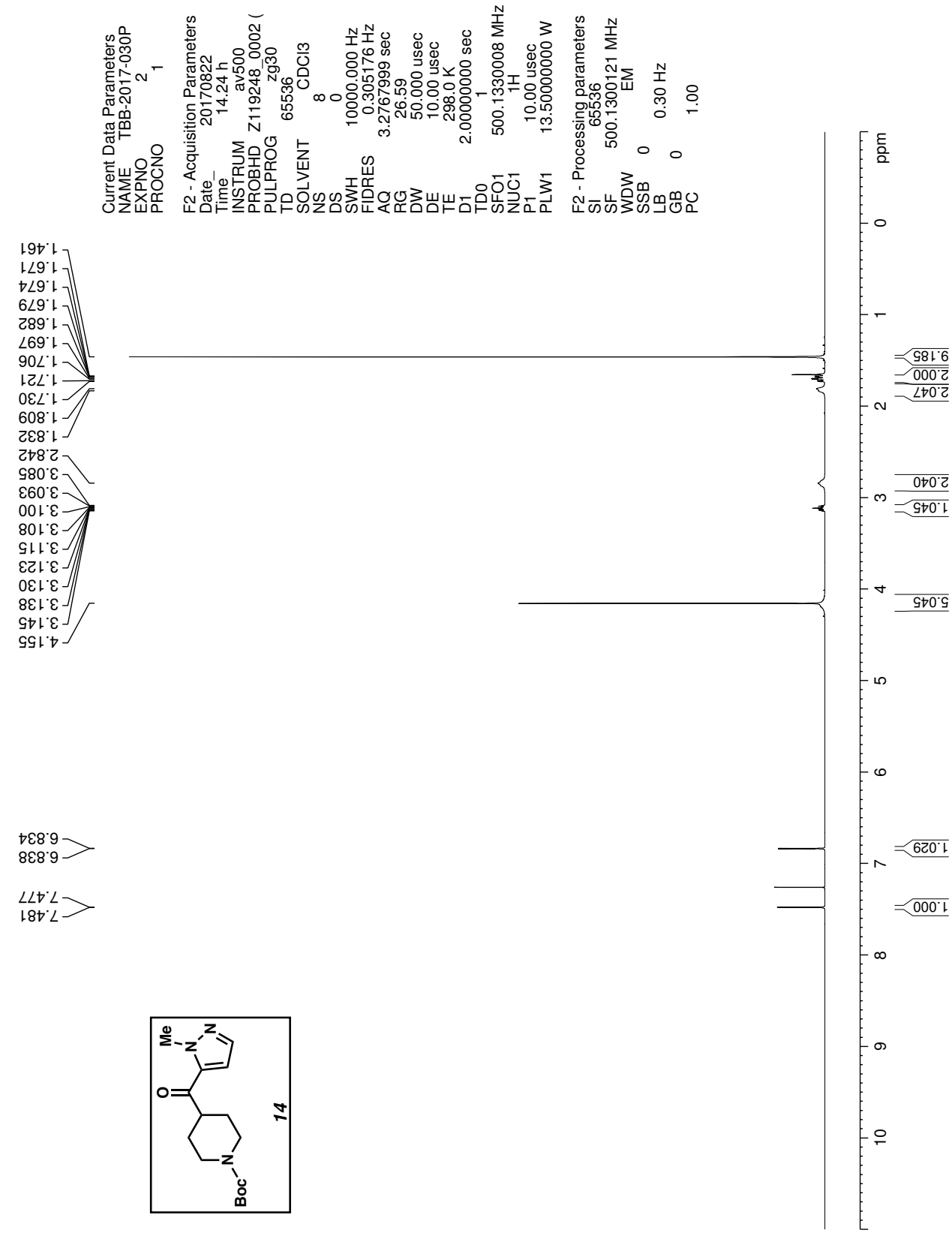

Purified Product, 1H NMR
Current Data Parameters
NAME TBB-1-136p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170326
Time_ 11.27 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 23.34
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Boc
N
Bn
O
MeO
O
39
ppm
9.104
4.072
4.098
1.015
1.012
3.004
2.000
2.046
3.021

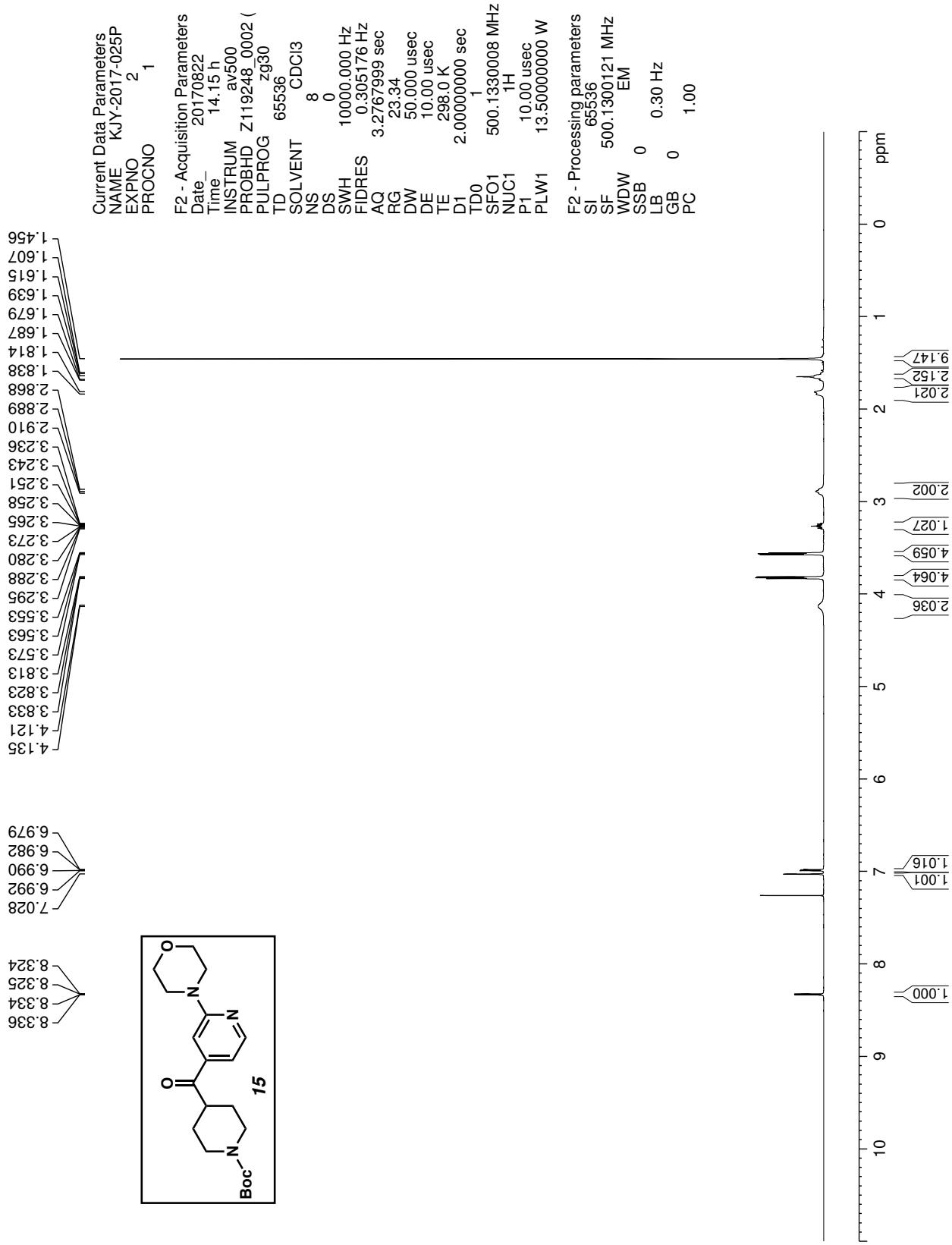

Purified Product, 1H NMR
Current Data Parameters
NAME TBB-1-197P
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170322
Time_ 15.20 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 26.59
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
N
Boc
6
ppm

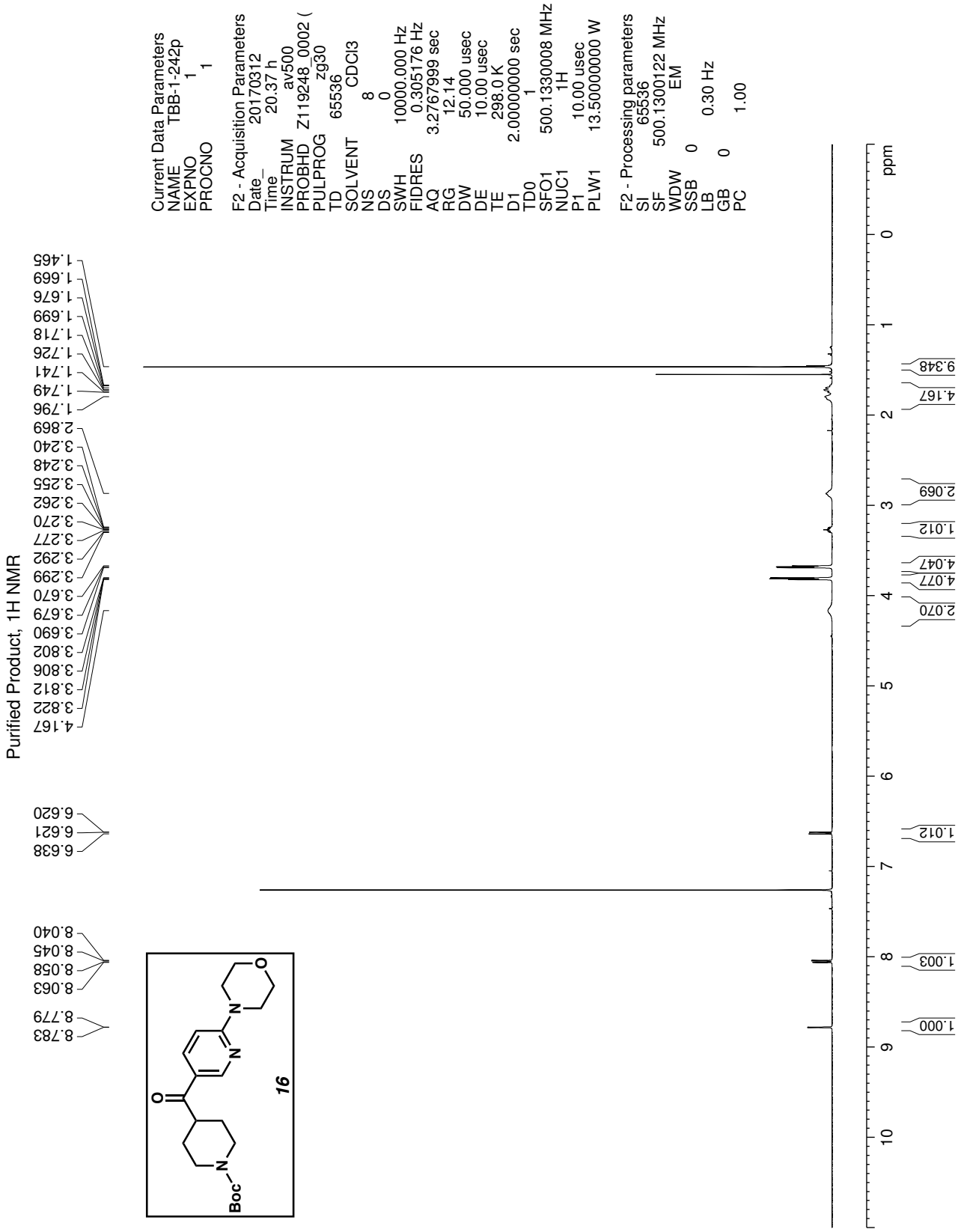

Current Data Parameters
NAME KJY-2017-023P
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170822
Time 14.06 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.024
9.020
9.015
9.012
8.439
8.435
8.303
8.301
8.300
8.287
8.284
8.244
8.241
8.227
8.223
8.187
8.169
7.507
7.499
7.491
7.482
4.198
4.191
3.585
3.577
3.570
3.562
3.555
3.548
3.540
3.533
3.525
2.972
2.951
2.929
1.916
1.896
1.799
1.791
1.769
1.745
1.471
O
N
Boc
11
ppm
1.000
1.026
1.044
1.029
1.015
1.028
2.066
1.047
2.045
2.036
2.024
9.045

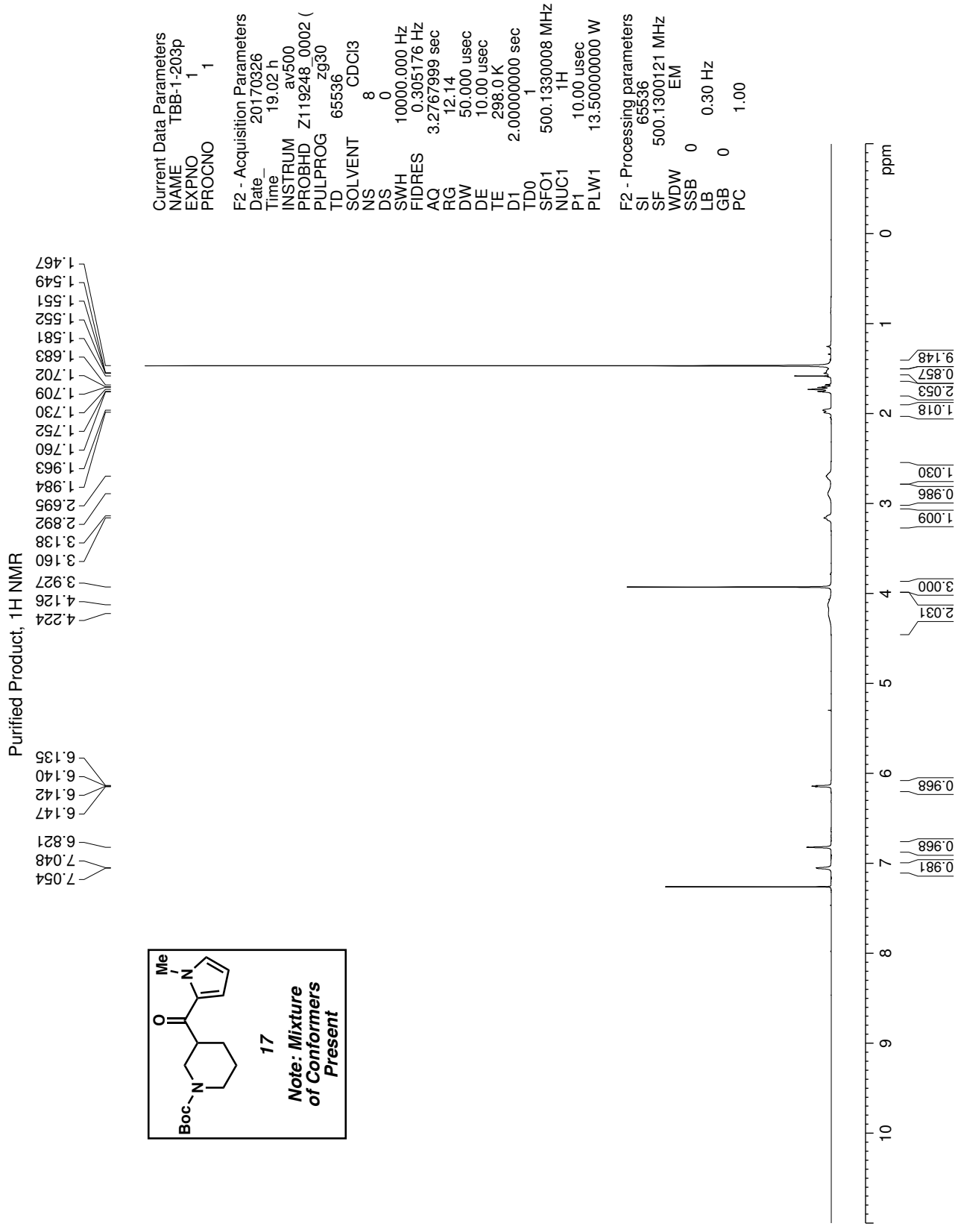

Purified Product, 1H NMR
Current Data Parameters
NAME TBB-1-228p
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time_ 8.18 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
N
Boc
12
ppm

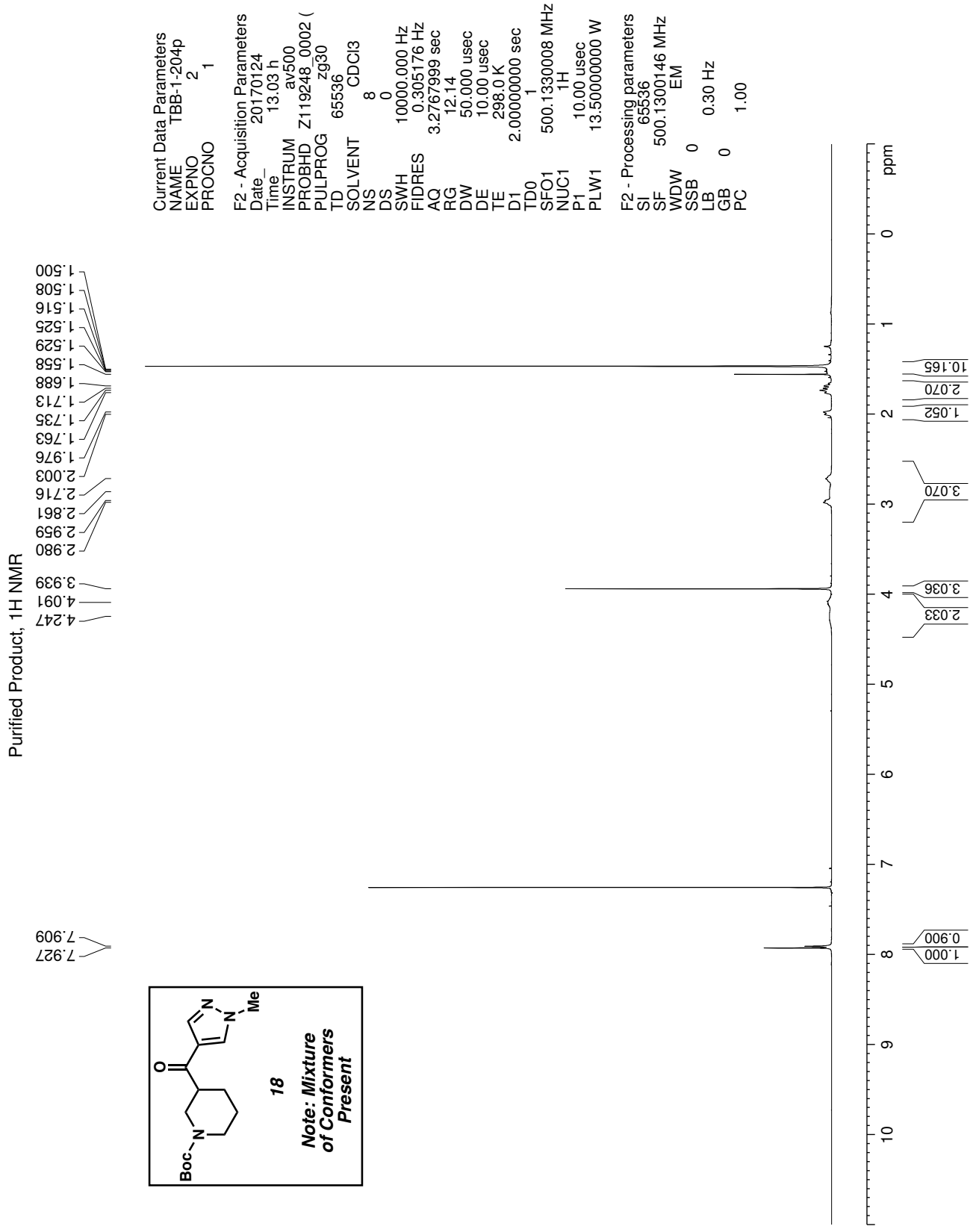

Purified Product, 1H NMR
Current Data Parameters
NAME TBB-1-198p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170314
Time_ 12.25 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.891
7.882
4.155
3.942
3.005
2.997
2.989
2.982
2.975
2.967
2.960
2.952
2.945
2.851
1.793
1.741
1.733
1.718
1.710
1.691
1.686
1.668
1.659
1.462
9.076
4.048
1.048
1.994
3.000
2.018
1.967
ppm
Me
N
N
O
N
Boc
13

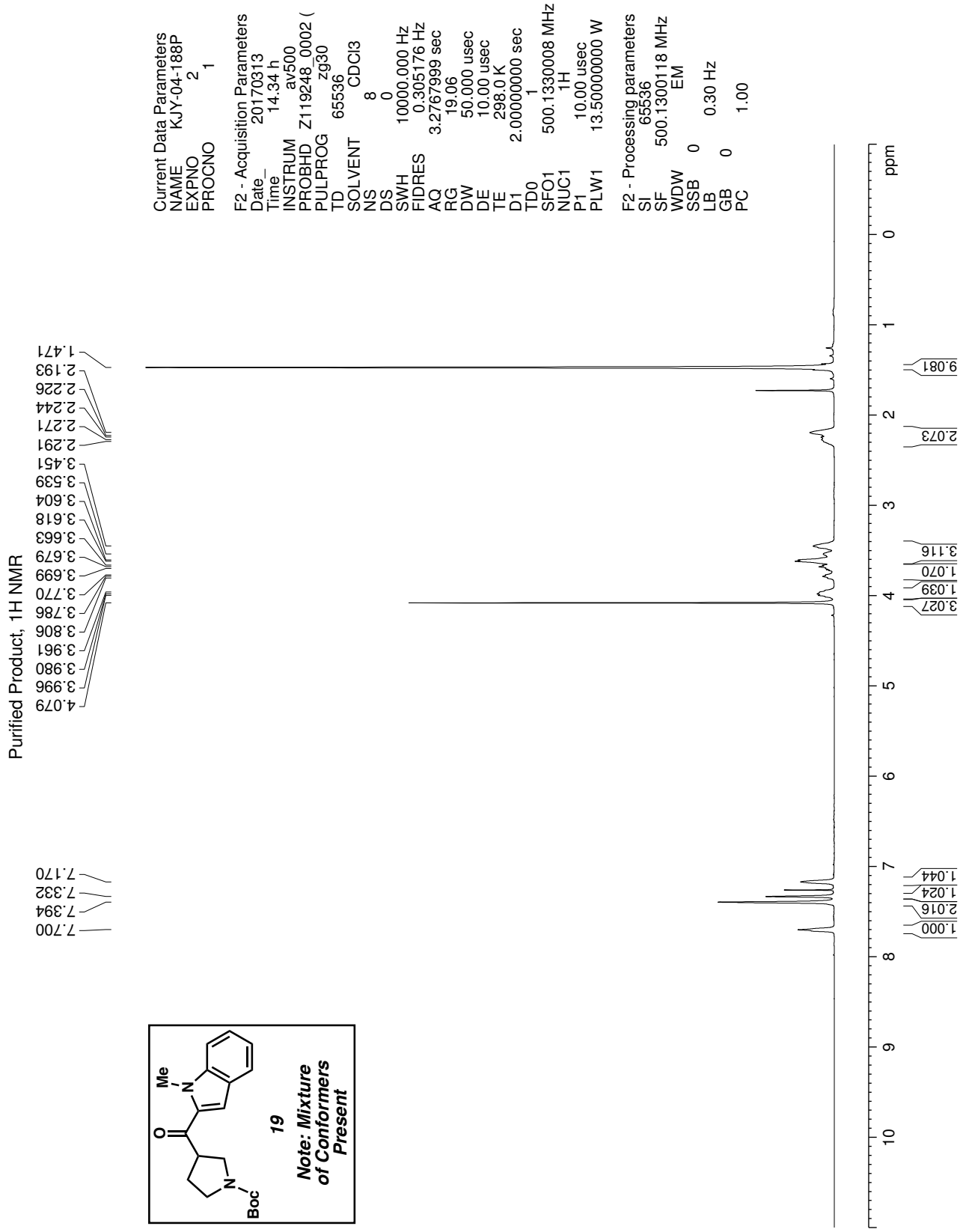

Current Data Parameters
NAME TBB-2017-030P
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170822
Time 14.24 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 26.59
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
O
Boc
14
ppm

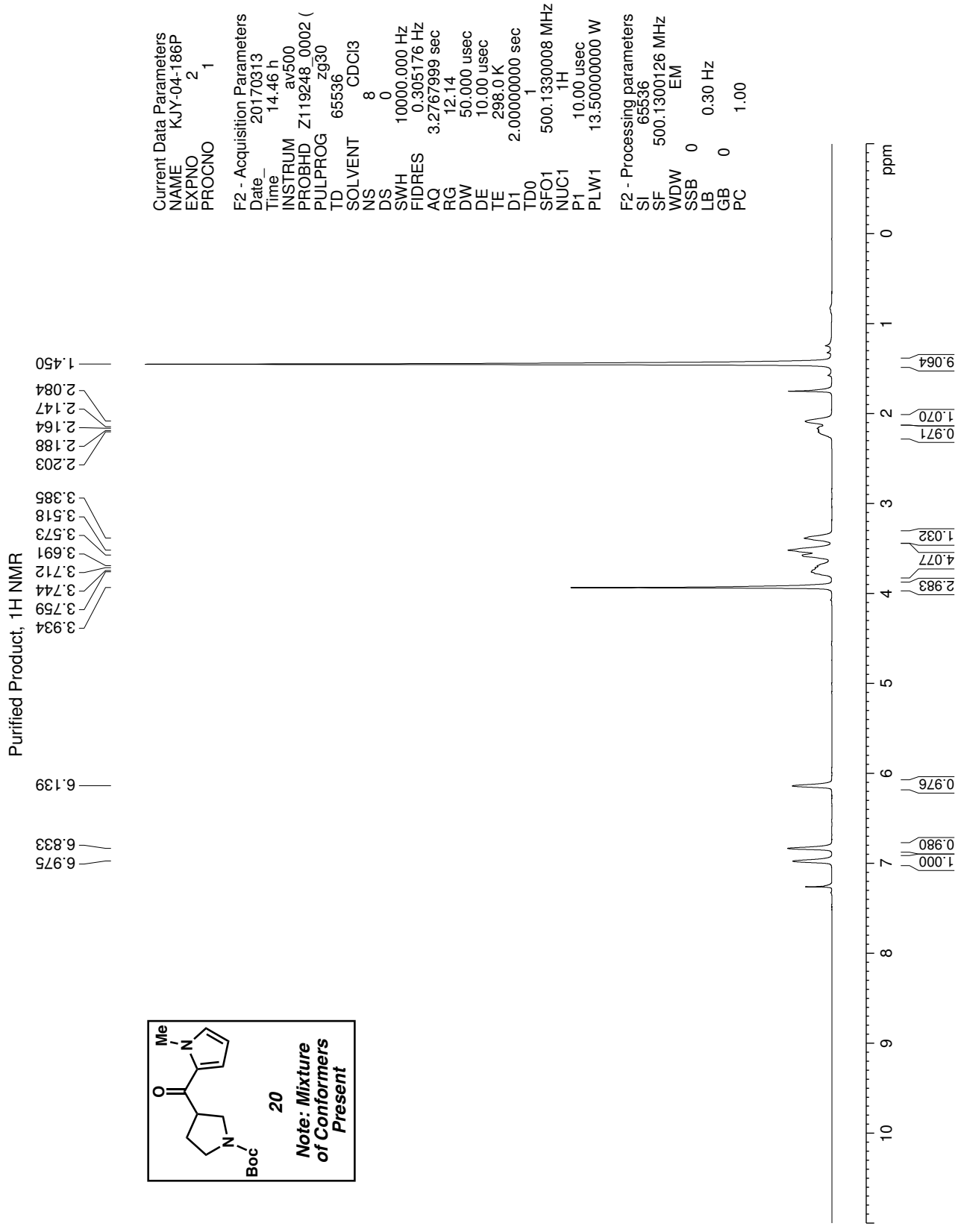

Current Data Parameters
NAME KJY-2017-025P
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170822
Time 14.15 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 23.34
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.336
8.325
8.324
7.028
6.992
6.990
6.982
6.979
4.135
4.121
3.833
3.823
3.813
3.573
3.563
3.553
3.295
3.288
3.280
3.273
3.265
3.258
3.251
3.243
3.236
2.910
2.889
2.868
1.838
1.814
1.687
1.679
1.639
1.615
1.607
1.456
1.000
1.001
1.016
2.036
4.059
4.064
1.027
2.002
2.021
2.152
9.147
Boc
15
ppm

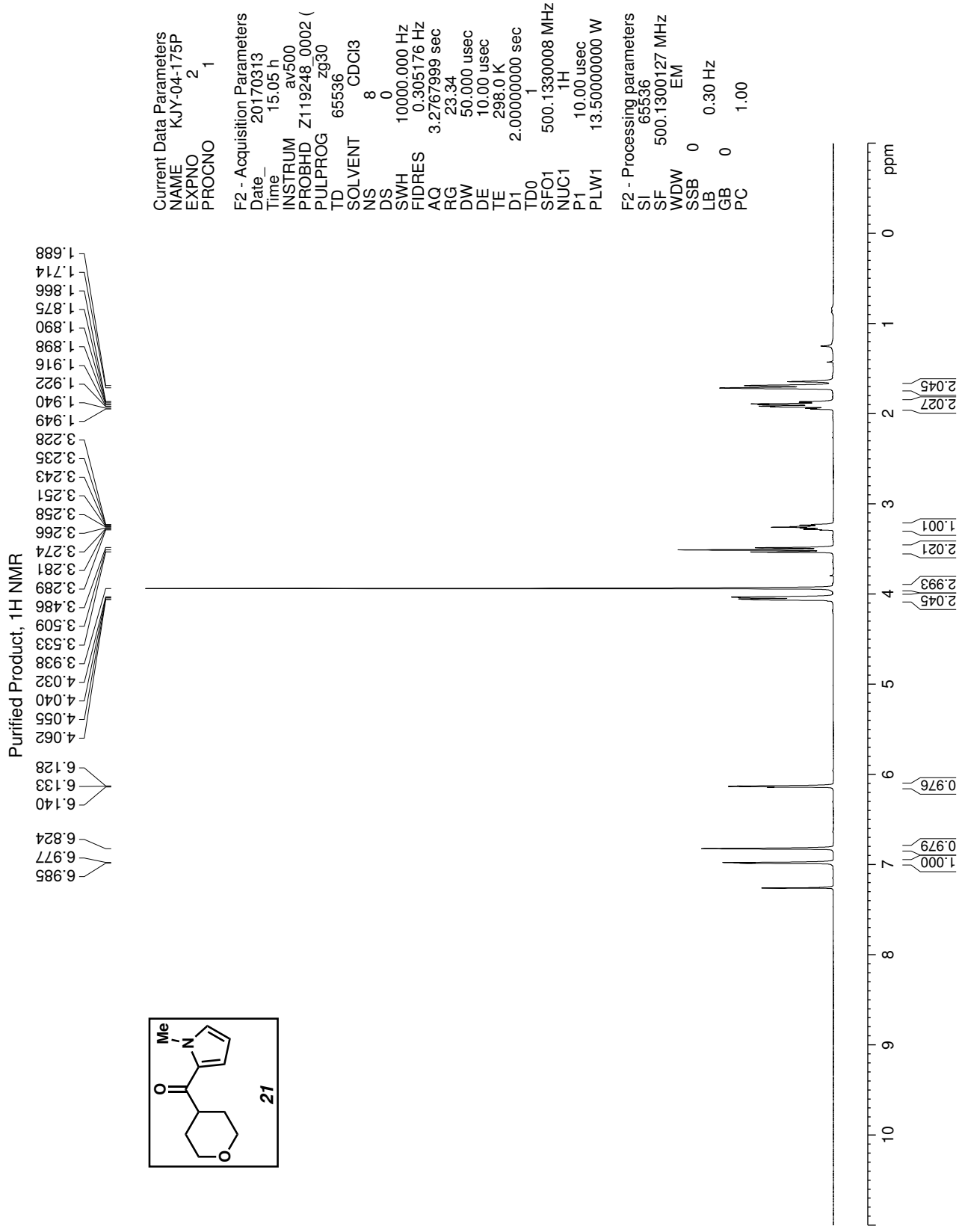

Purified Product, 1H NMR
Current Data Parameters
NAME TBB-1-242p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170312
Time_ 20.37 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
16
Boc
N
O
1.465
1.669
1.676
1.699
1.718
1.726
1.741
1.749
1.796
2.869
3.240
3.248
3.255
3.262
3.270
3.277
3.292
3.299
3.670
3.679
3.690
3.802
3.806
3.812
3.822
4.167
6.620
6.621
6.638
8.040
8.045
8.058
8.063
8.779
8.783
9.348
4.167
2.069
1.012
4.047
4.077
2.070
1.012
1.003
1.000

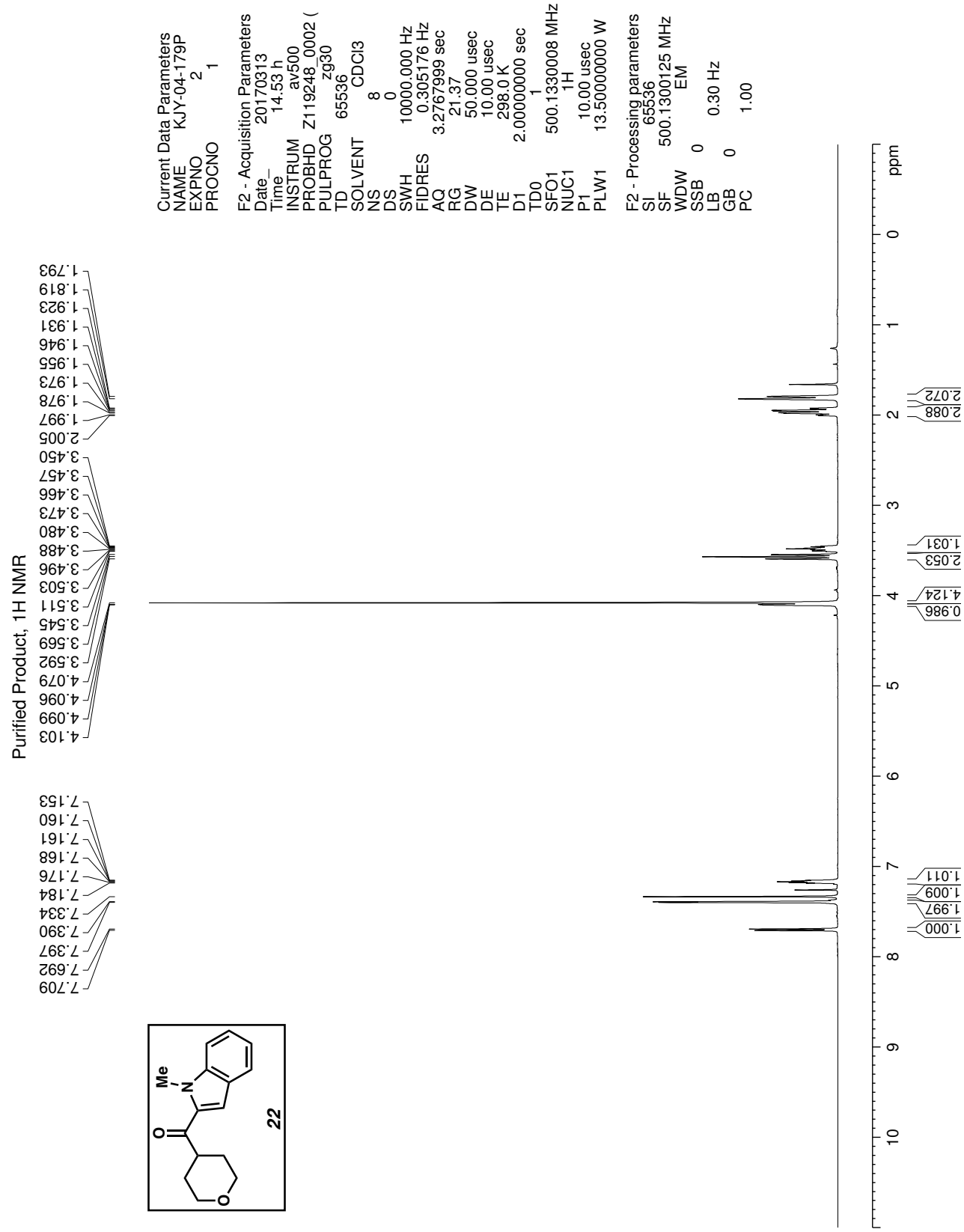

Purified Product, 1H NMR
Current Data Parameters
NAME TBB-1-203p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170326
Time_ 19.02 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
Boc
N
17
Note: Mixture of Conformers Present
ppm

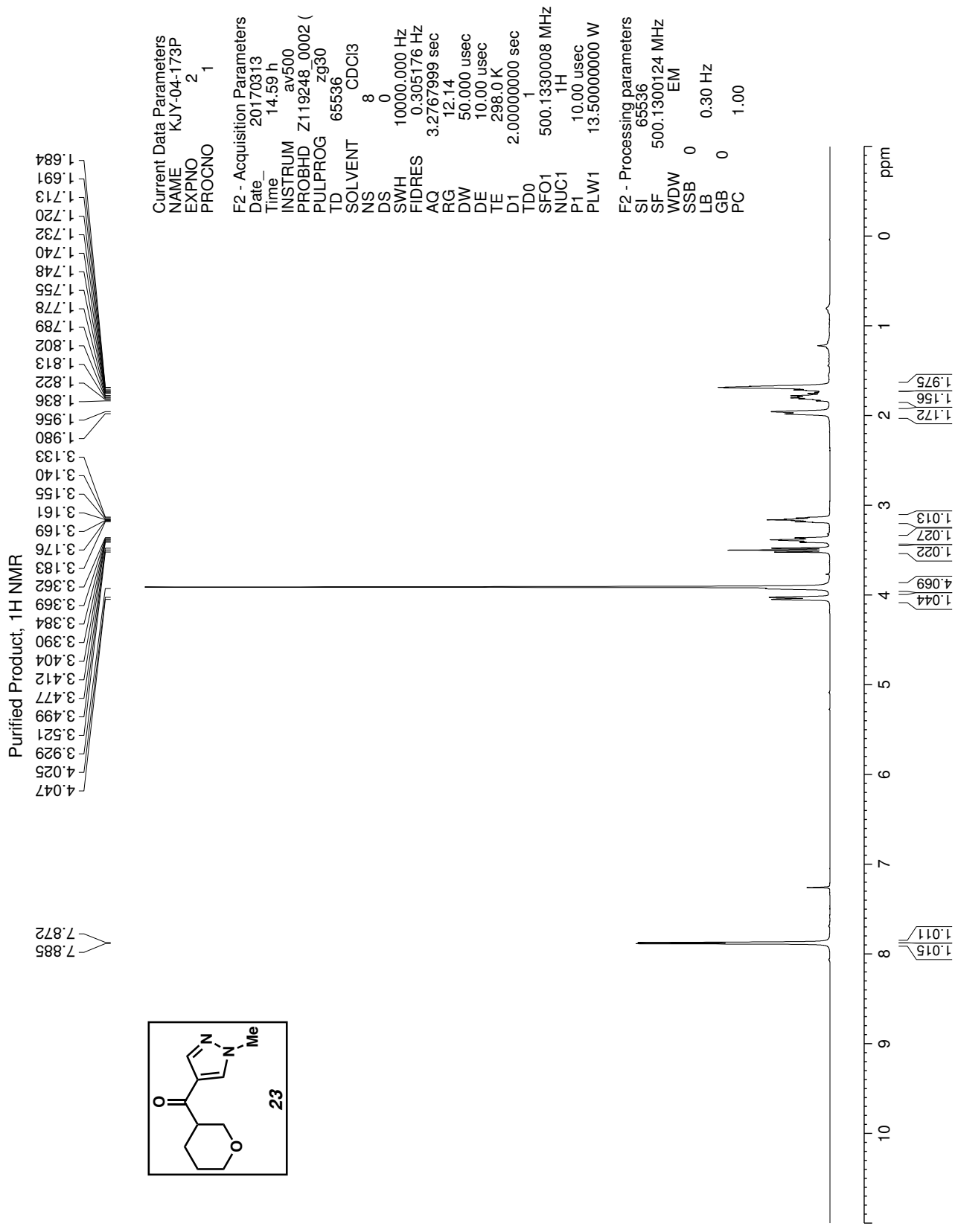

Purified Product, 1H NMR
Current Data Parameters
NAME TBB-1-204p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170124
Time_ 13.03 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300146 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.927
7.909
4.247
4.091
3.939
2.980
2.959
2.861
2.716
2.003
1.976
1.763
1.735
1.713
1.688
1.558
1.529
1.525
1.516
1.508
1.500
1.000
0.900
2.033
3.036
3.070
1.052
2.070
10.165
ppm
Boc
N
O
N
N
Me
18
Note: Mixture of Conformers Present

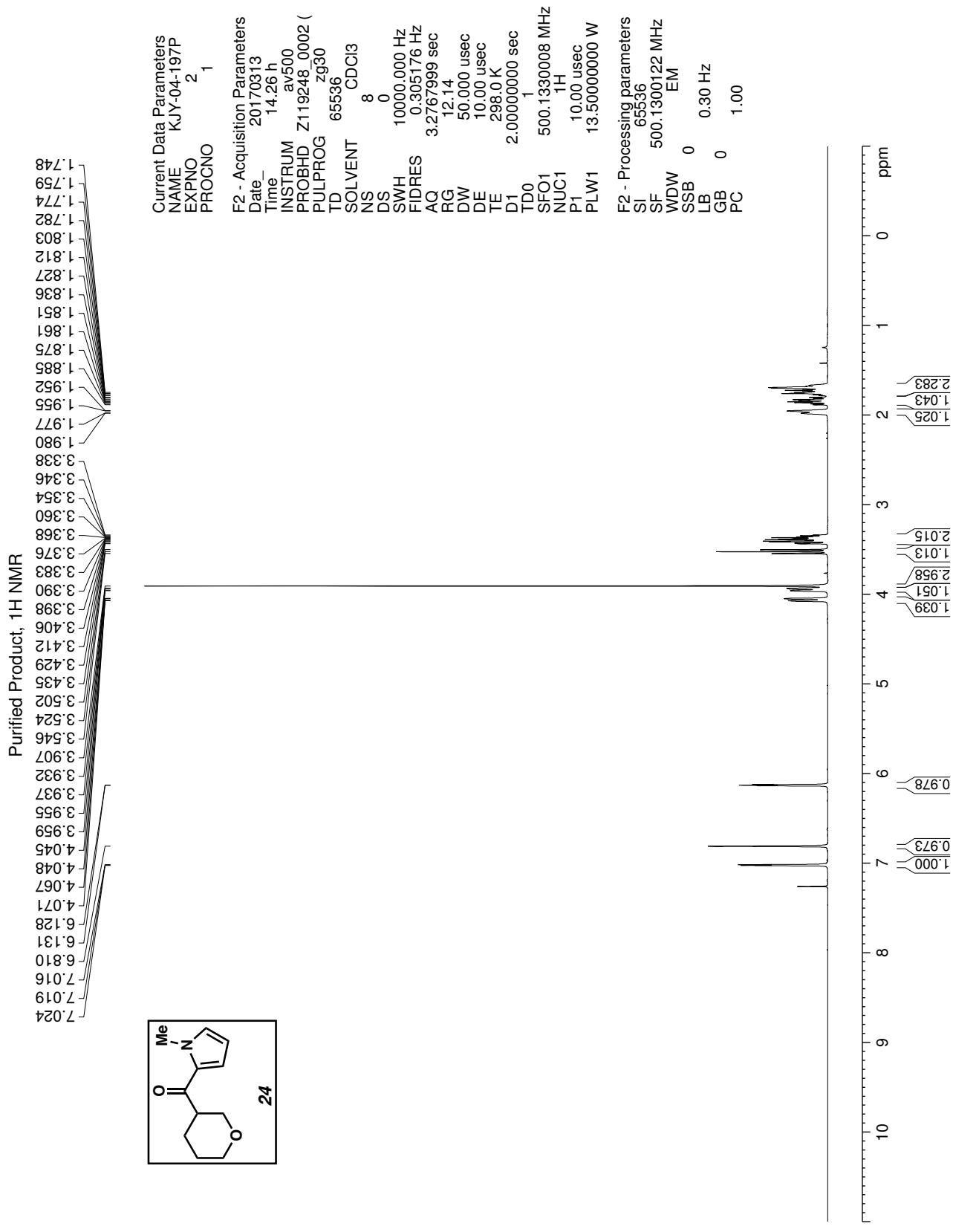

Purified Product, 1H NMR
Current Data Parameters
NAME KJY-04-188P
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time_ 14.34 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 19.06
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300118 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
N
Boc
19
Note: Mixture of Conformers Present
ppm

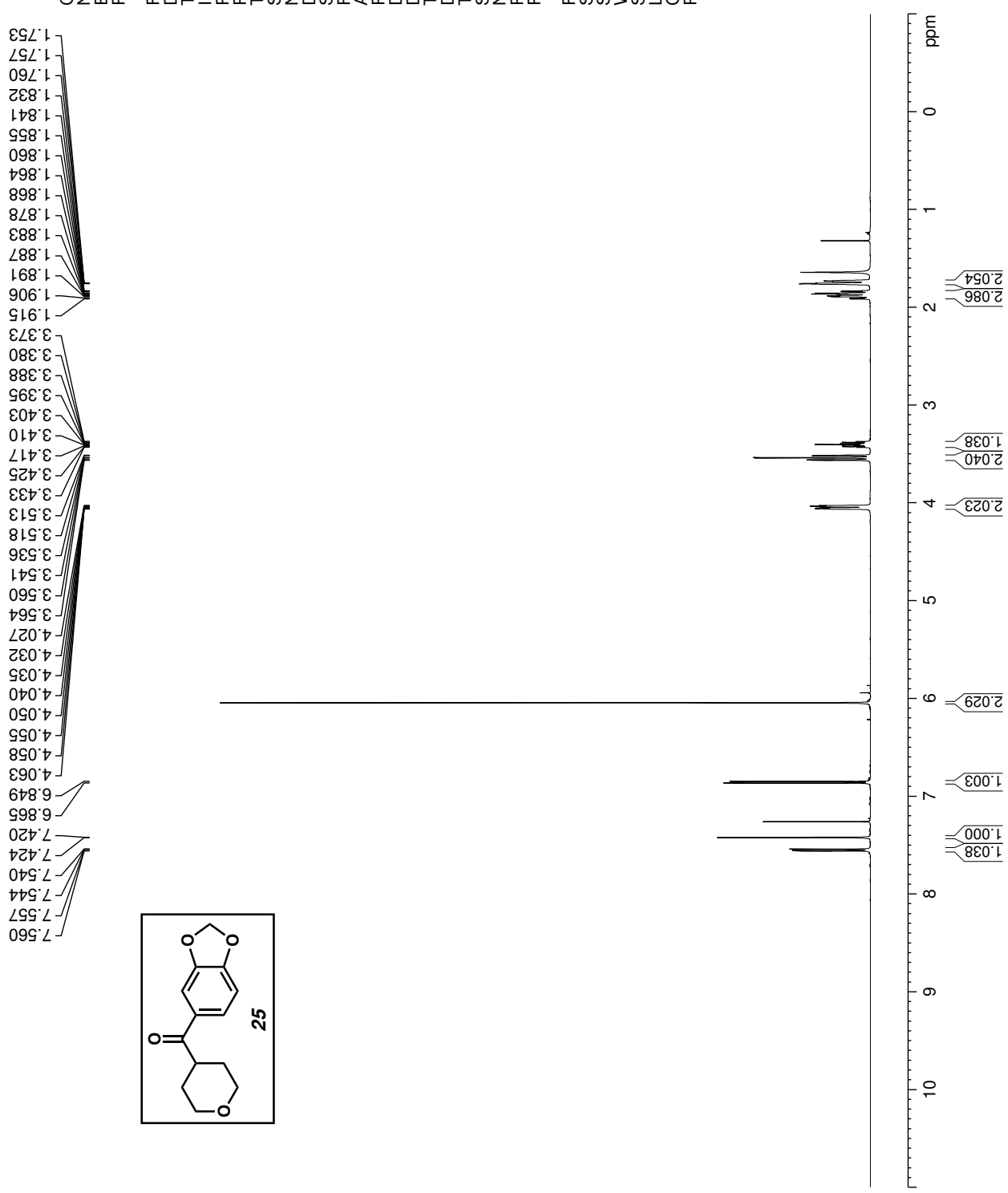

Purified Product, 1H NMR
Current Data Parameters
NAME KJY-04-186P
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time_ 14.46 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300126 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.450
2.084
2.147
2.164
2.188
2.203
3.385
3.518
3.573
3.691
3.712
3.744
3.759
3.934
6.139
6.833
6.975
9.064
1.070
0.971
1.032
4.077
2.983
0.976
0.980
1.000
ppm
Me
N
O
N
Boc
20
Note: Mixture of Conformers Present

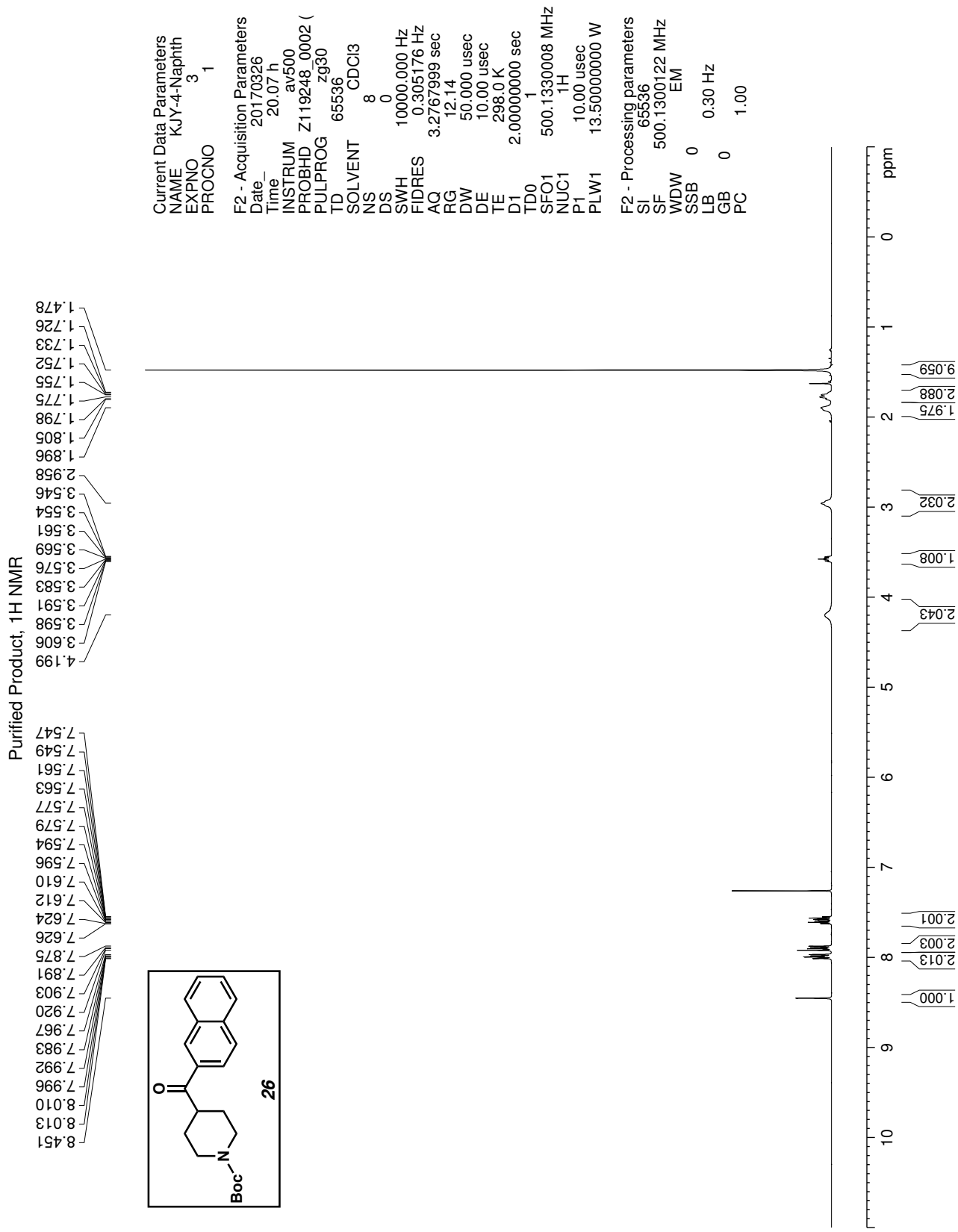

Purified Product, 1H NMR
Current Data Parameters
NAME KJY-04-175P
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time_ 15.05 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 23.34
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300127 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
O
21
ppm

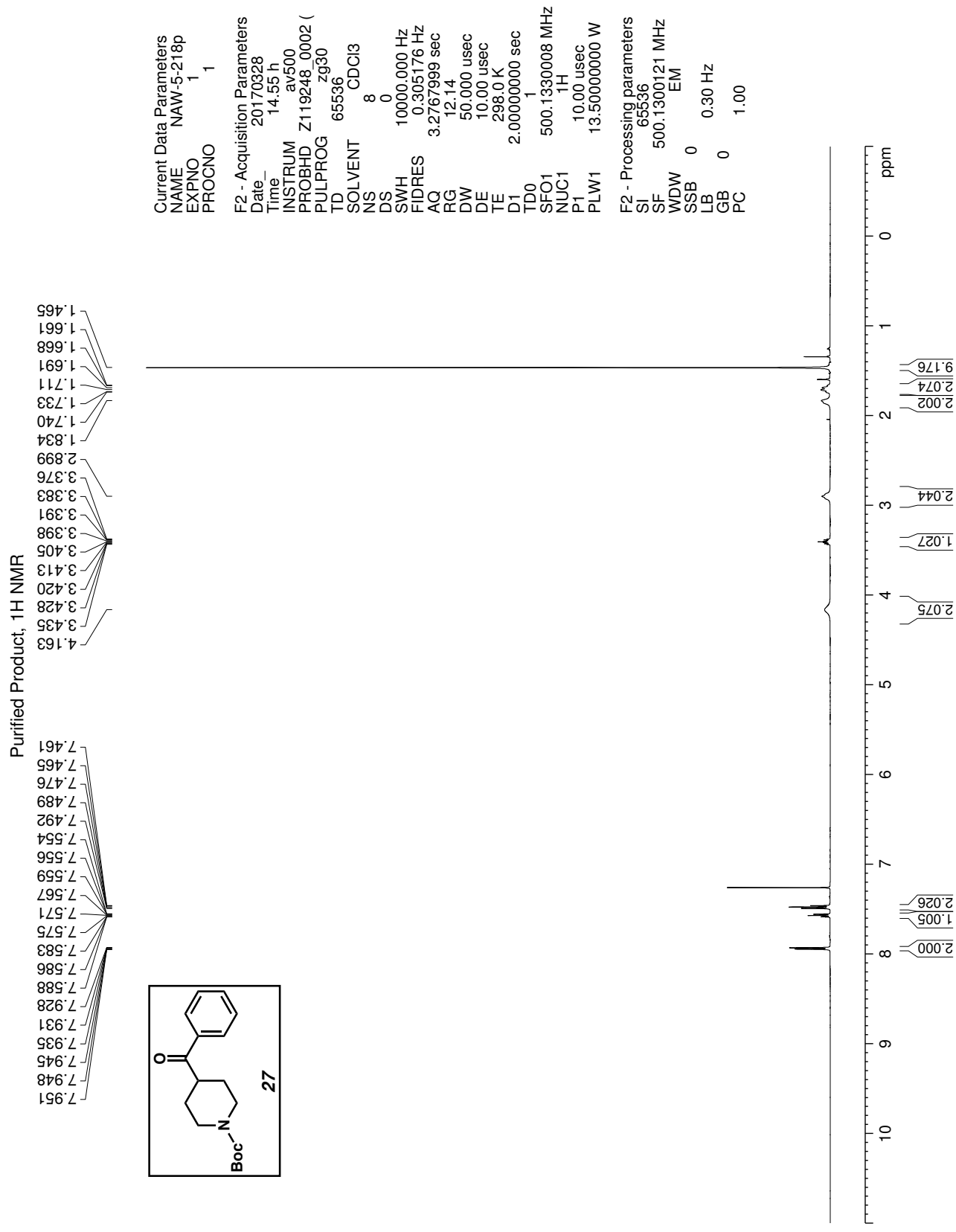

Purified Product, 1H NMR
Current Data Parameters
NAME KJY-04-179P
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time 14.53 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 21.37
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
O
22
ppm


Purified Product, 1H NMR
Current Data Parameters
NAME KJY-04-173P
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time_ 14.59 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300124 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
23


Purified Product, 1H NMR
Current Data Parameters
NAME KJY-04-197P
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time_ 14.26 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
O
24
ppm


Current Data Parameters
NAME KJY-04-255P
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170615
Time 13.08 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 21.37
DW 50.000 usec
DE 10.000 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
25
ppm
2.086
2.054
1.038
2.040
2.023
2.029
1.003
1.038
1.000


Purified Product, 1H NMR
Current Data Parameters
NAME KJY-4-Naphth
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170326
Time_ 20.07 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
Boc
O
N
26


Purified Product, 1H NMR
Current Data Parameters
NAME NAW-5-218p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170328
Time_ 14.55 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
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SF 500.1300121 MHz
WDW EM
SSB 0
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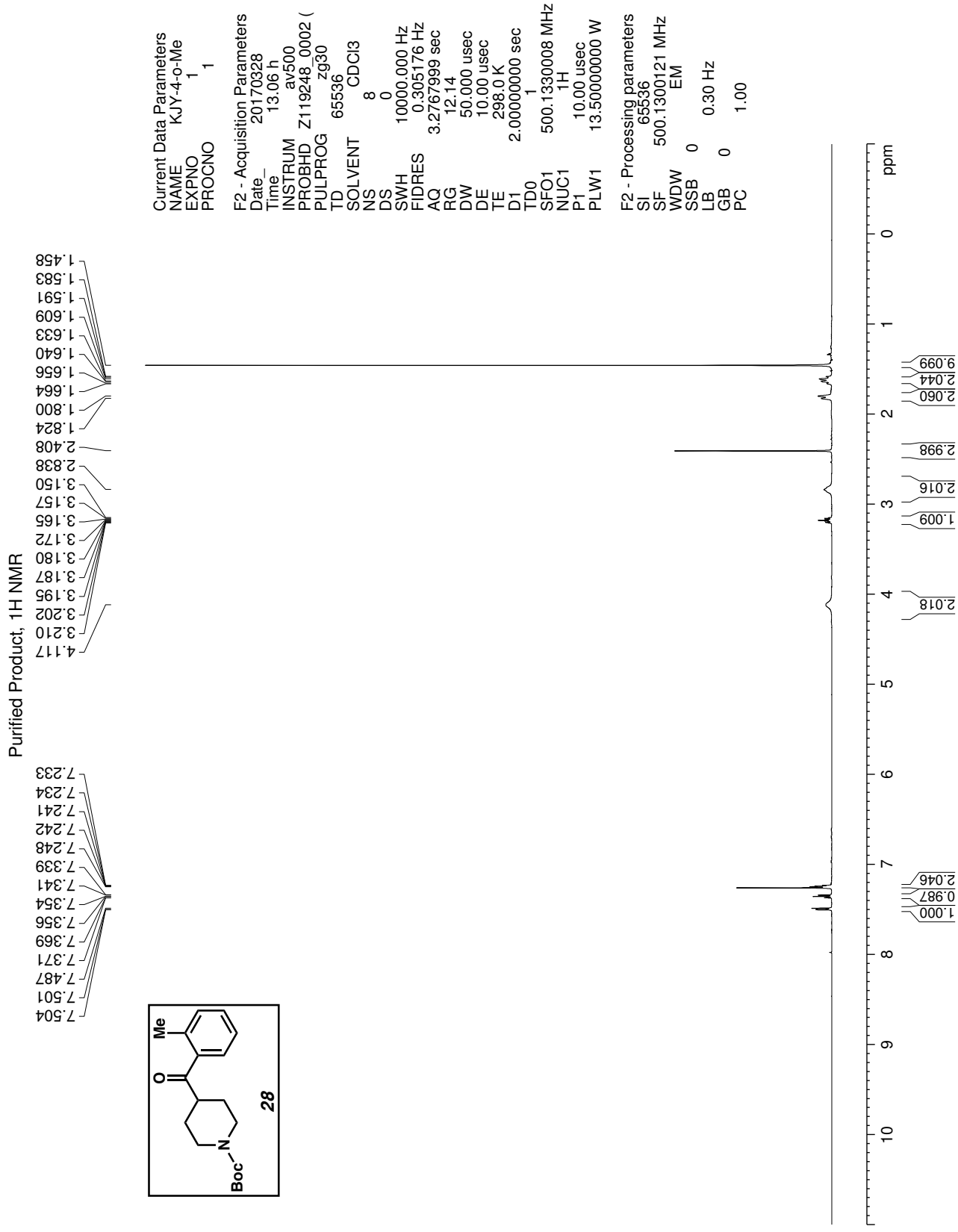
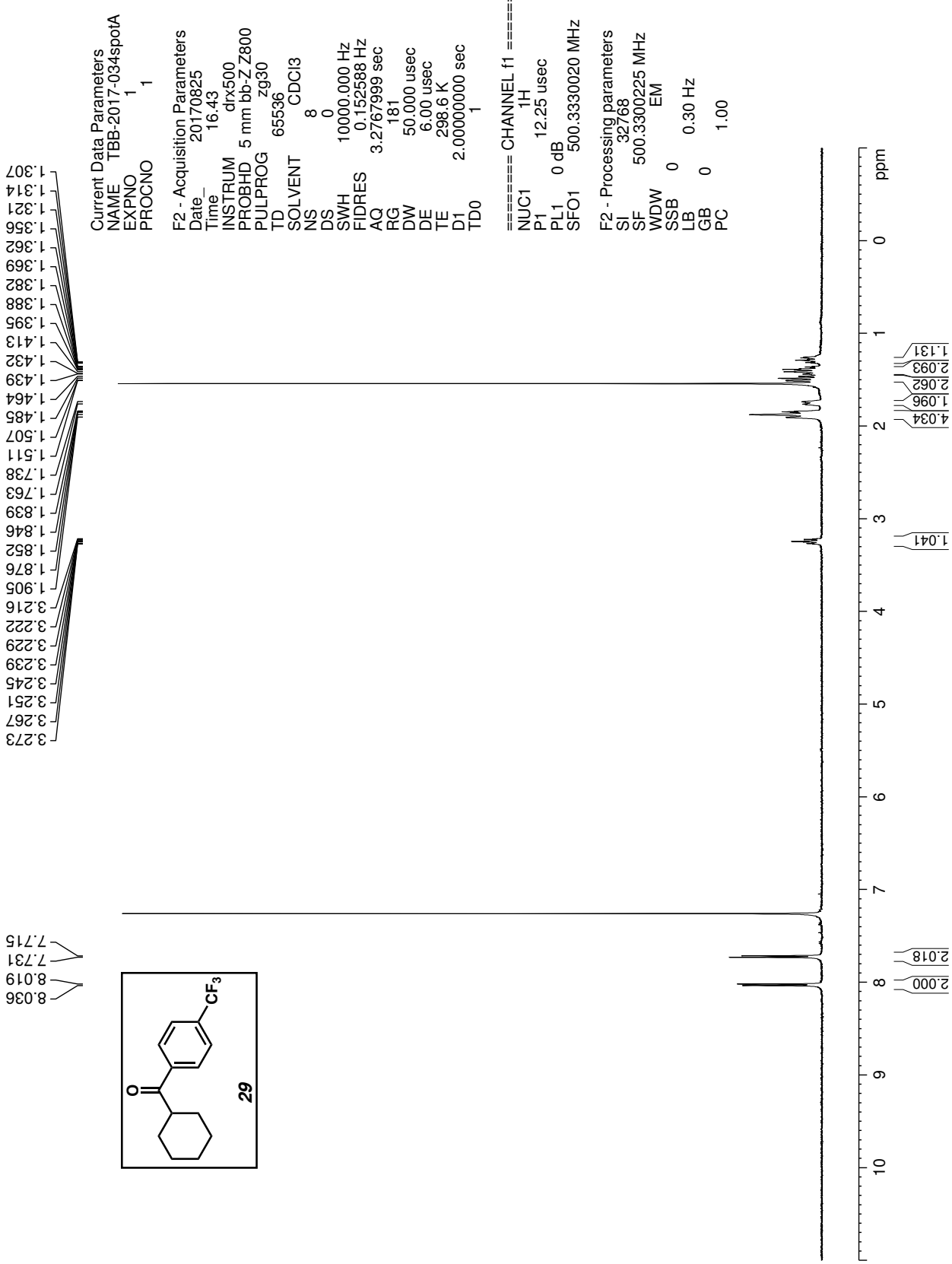
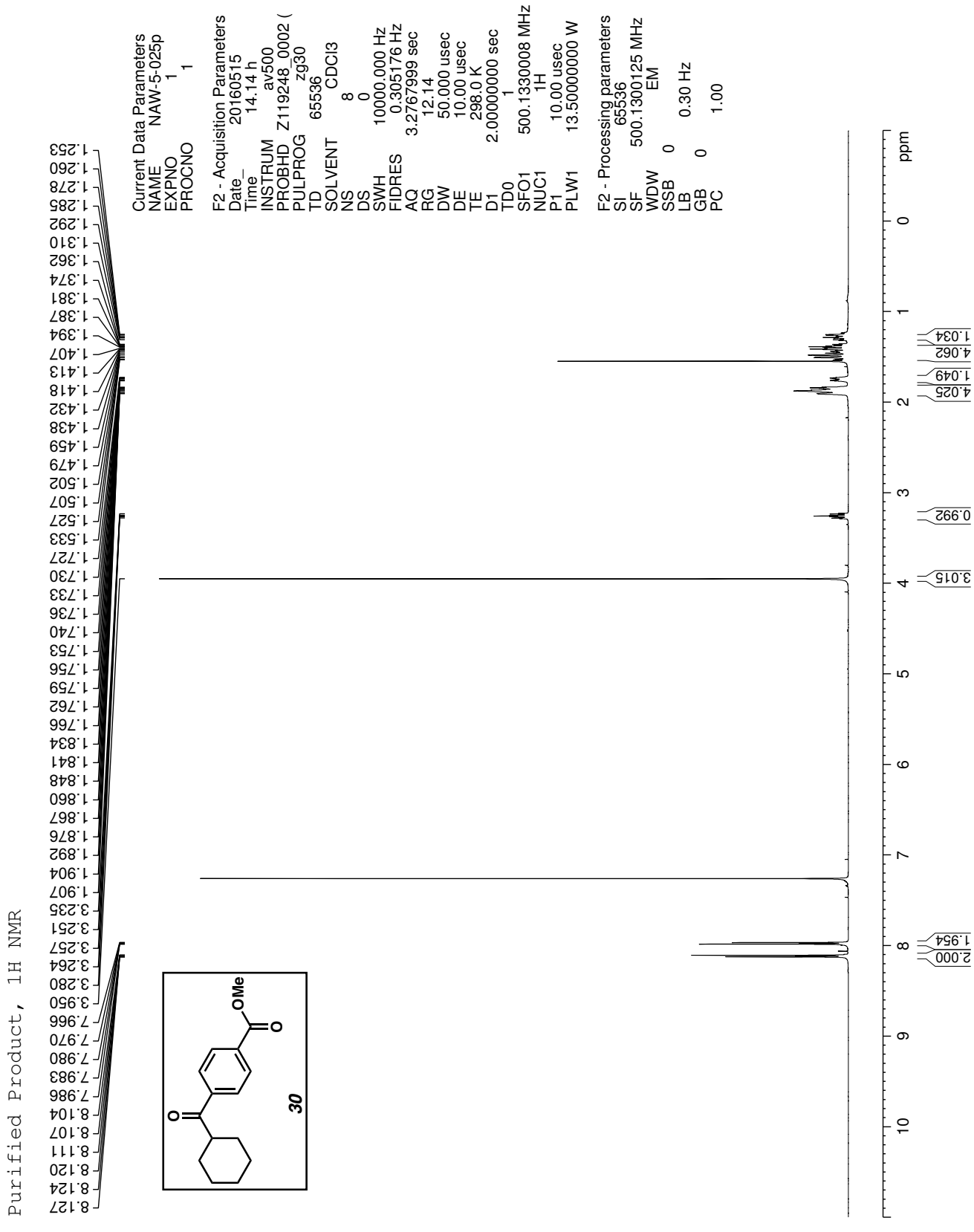
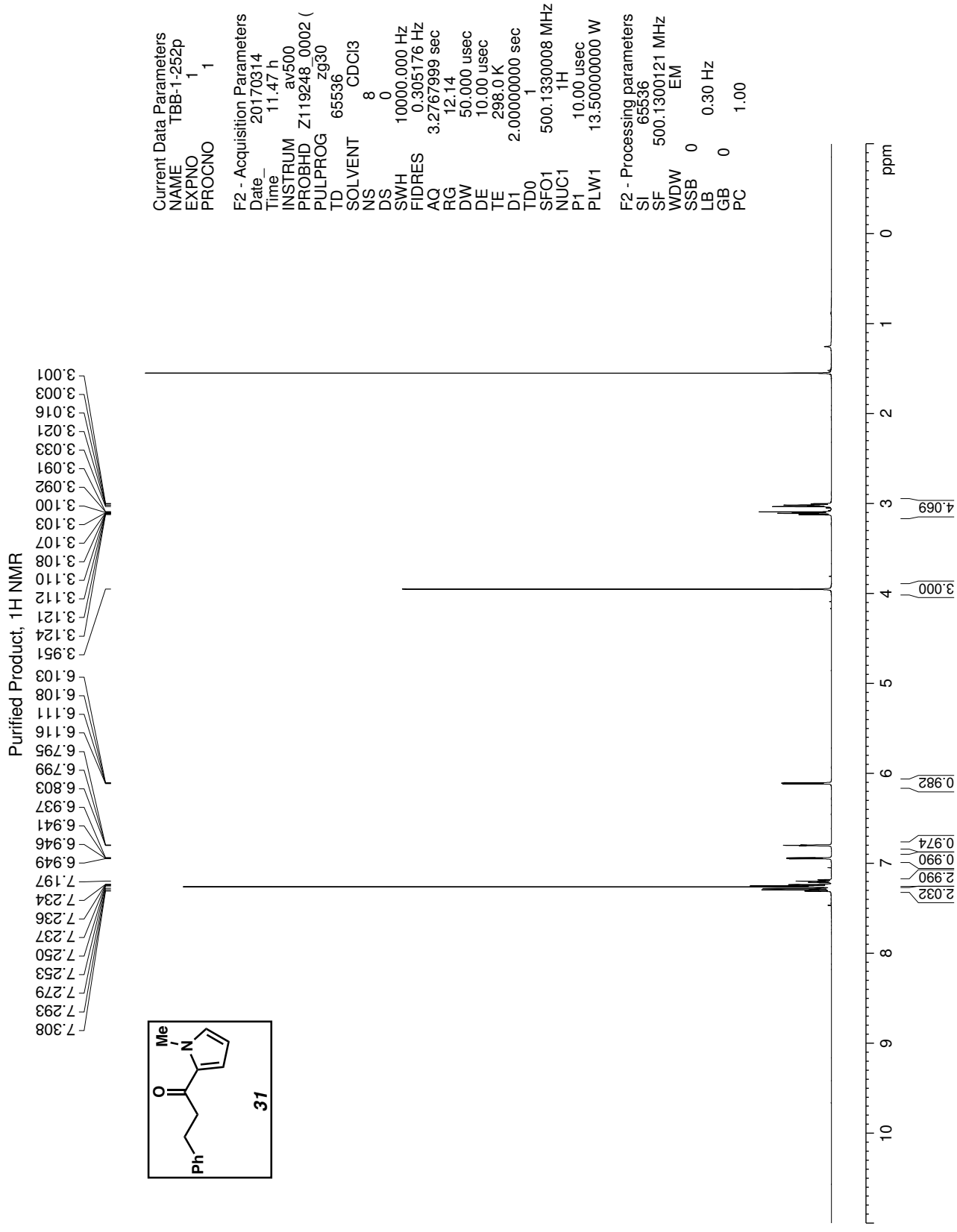
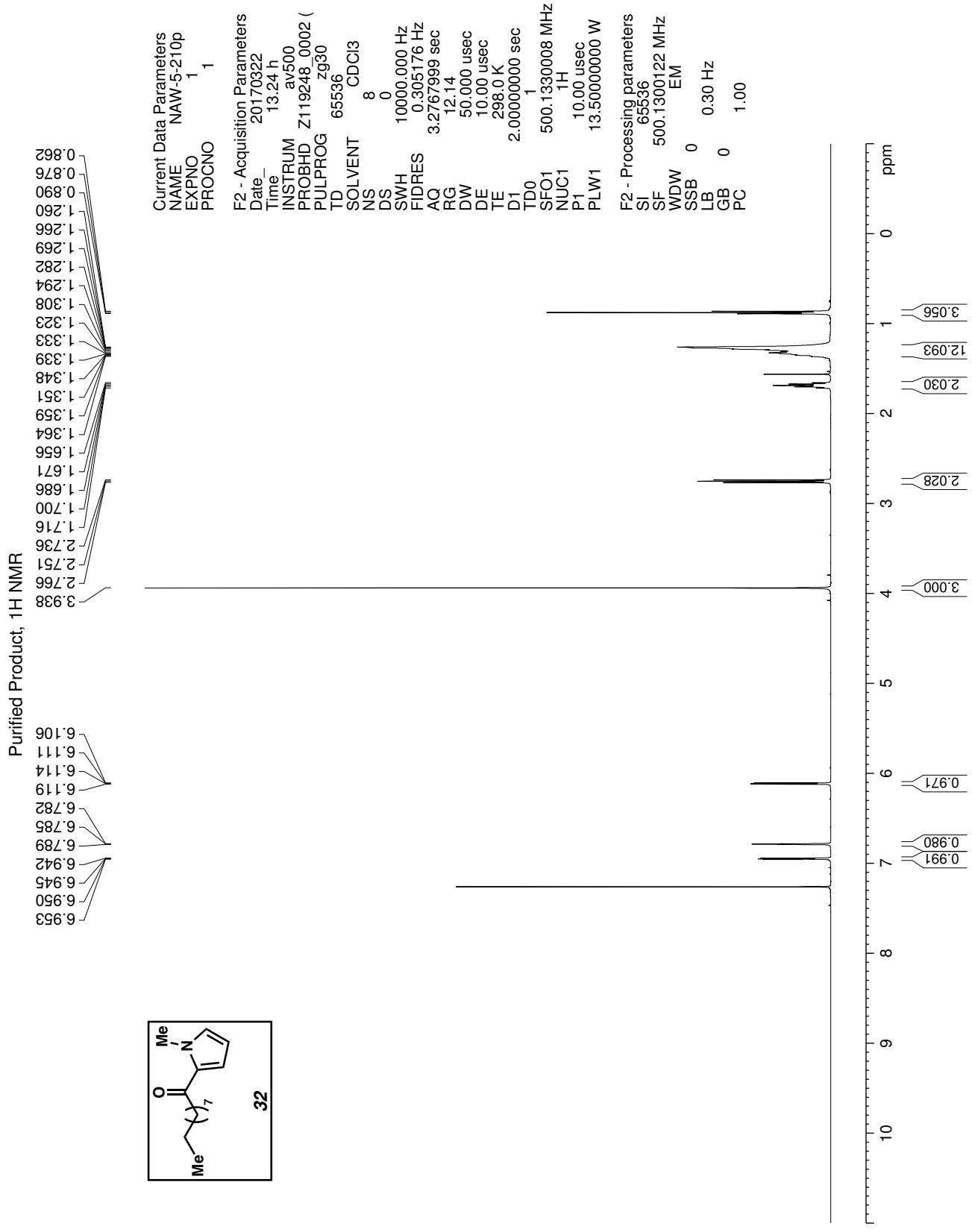
GB 0
PC 1.00
O
N
Boc
27
ppm

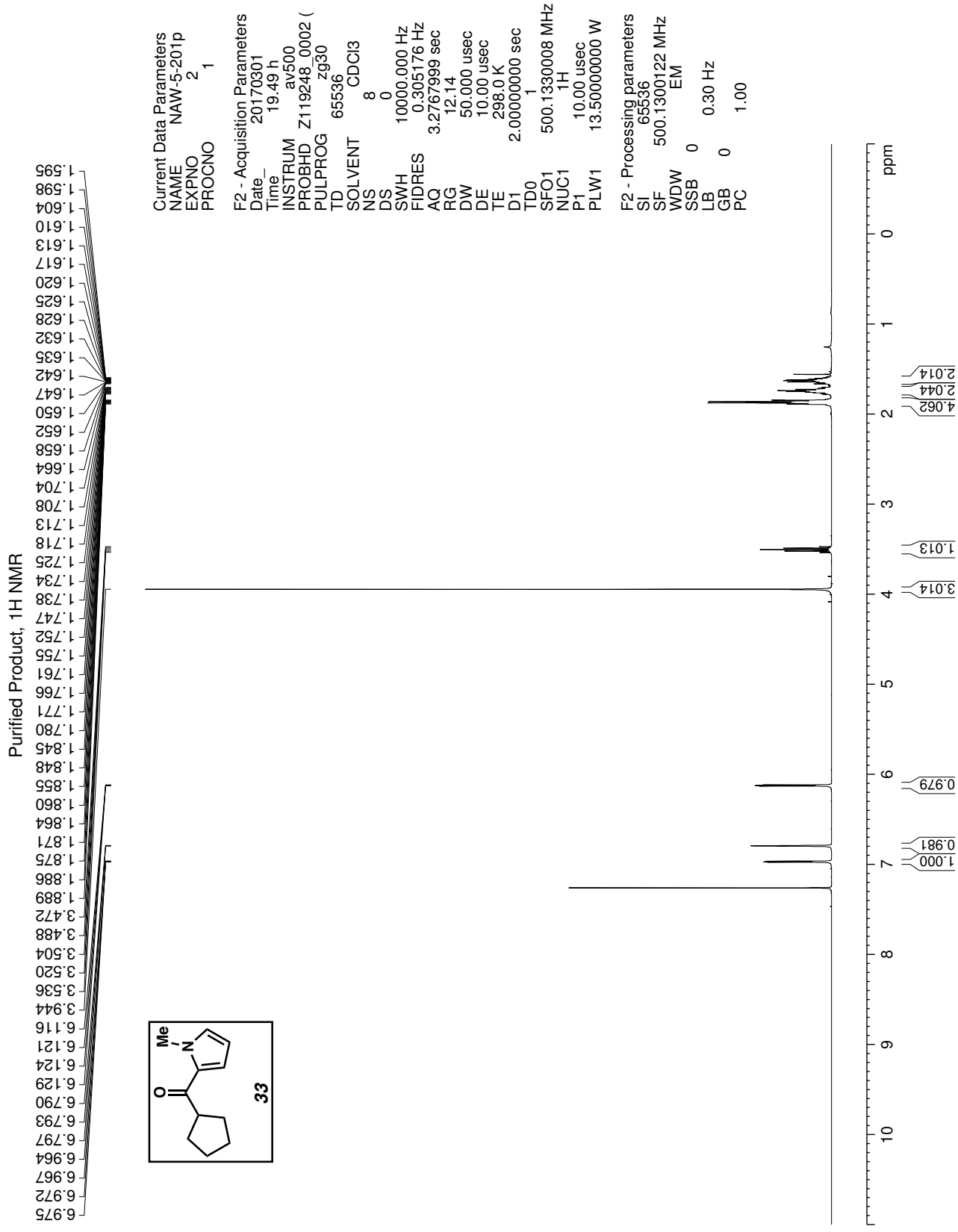

Purified Product, 1H NMR
Current Data Parameters
NAME KJY-4-o-Me
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170328
Time_ 13.06 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
O
N
Boc
28
ppm

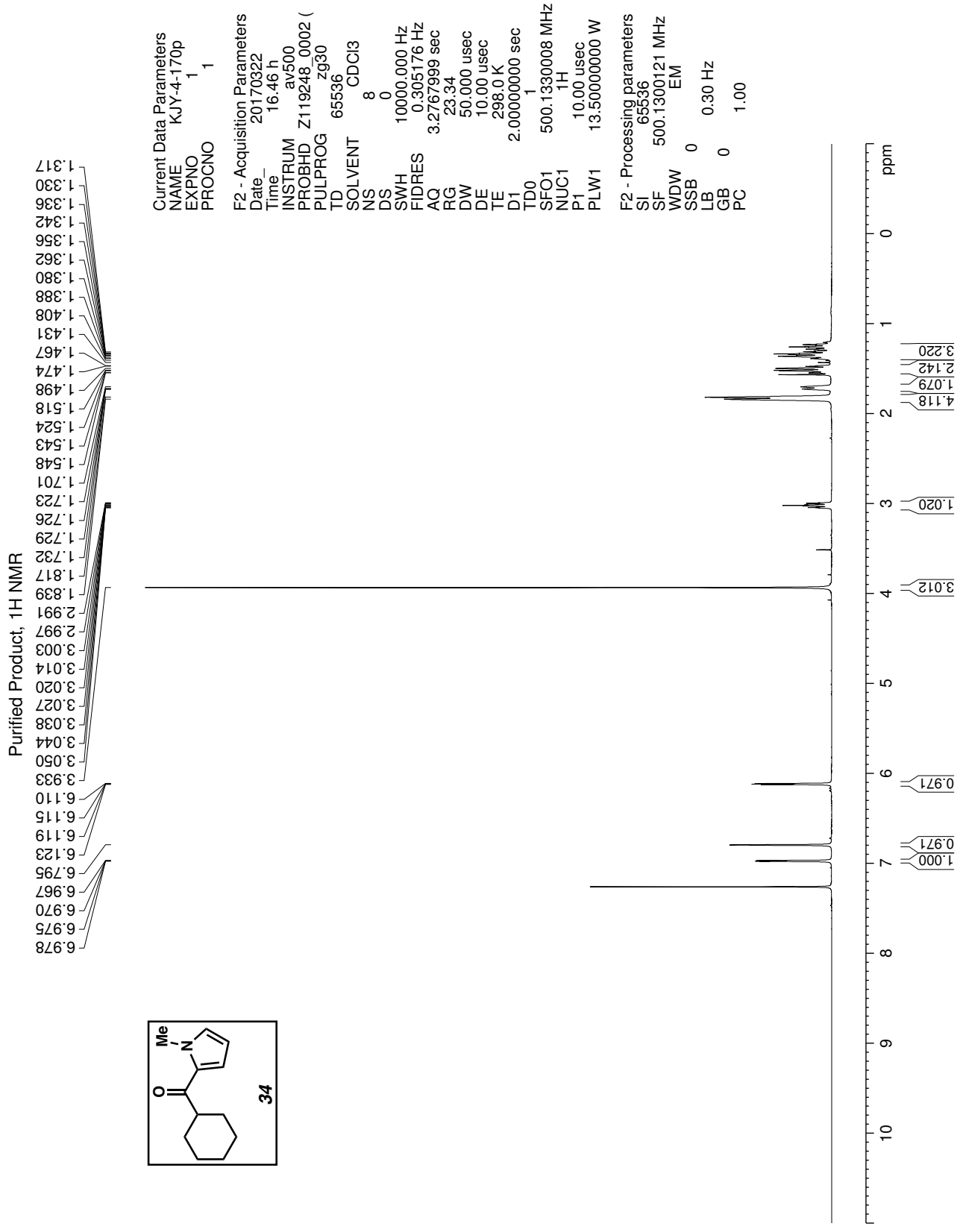

Current Data Parameters
NAME TBB-2017-034spotA
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170825
Time 16.43
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 298.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.25 usec
PL1 0 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300225 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.307
1.314
1.321
1.356
1.362
1.369
1.382
1.388
1.395
1.413
1.432
1.439
1.464
1.485
1.507
1.511
1.738
1.763
1.839
1.846
1.852
1.876
1.905
3.216
3.222
3.229
3.239
3.245
3.251
3.267
3.273
7.715
7.731
8.019
8.036
2.000
2.018
1.041
4.034
1.096
2.062
2.093
1.131
ppm
O
CF3
29

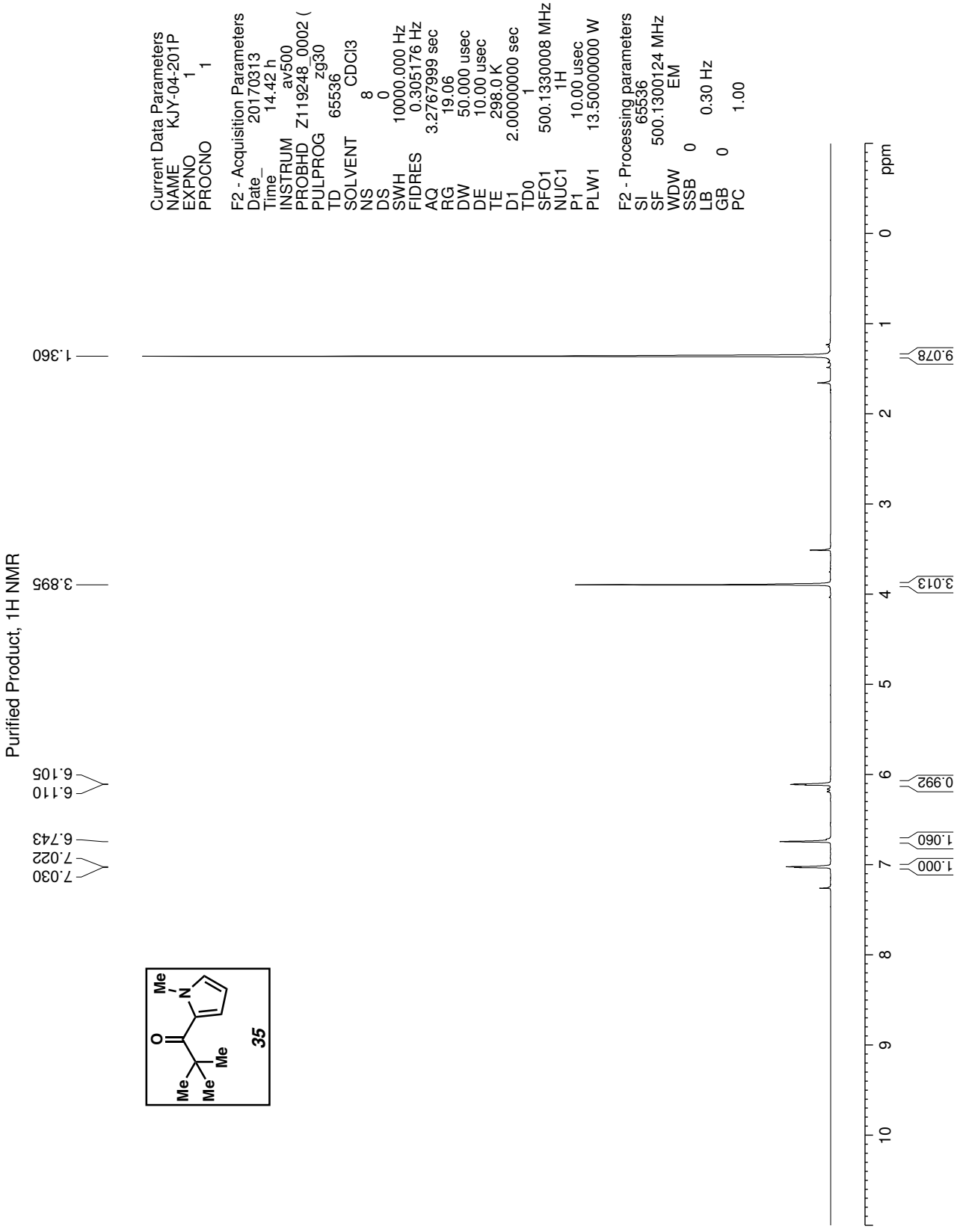

Purified Product, 1H NMR
Current Data Parameters
NAME NAW-5-025p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160515
Time 14.14 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMe
O
O
30
ppm
2.000
1.954
3.015
0.992
4.025
1.049
4.062
1.034

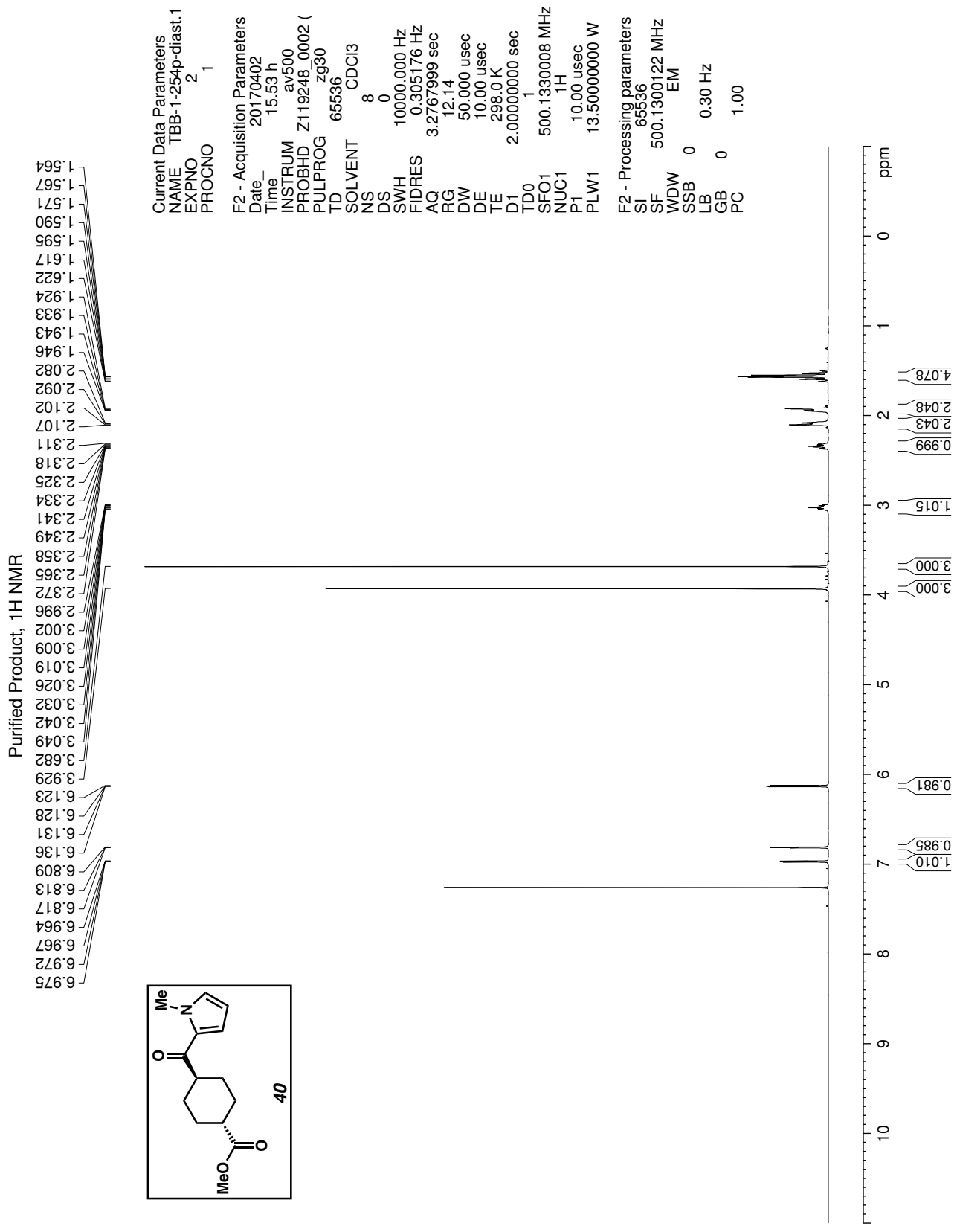

Purified Product, 1H NMR
Current Data Parameters
NAME TBB-1-252p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170314
Time_ 11.47 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.308
7.293
7.279
7.253
7.250
7.237
7.236
7.234
7.197
6.949
6.946
6.941
6.937
6.803
6.799
6.795
6.116
6.111
6.108
6.103
3.951
3.124
3.121
3.112
3.110
3.108
3.107
3.103
3.100
3.092
3.091
3.033
3.021
3.016
3.003
3.001
Me
N
O
Ph
31
4.069
3.000
0.982
0.974
0.990
2.990
2.032
ppm

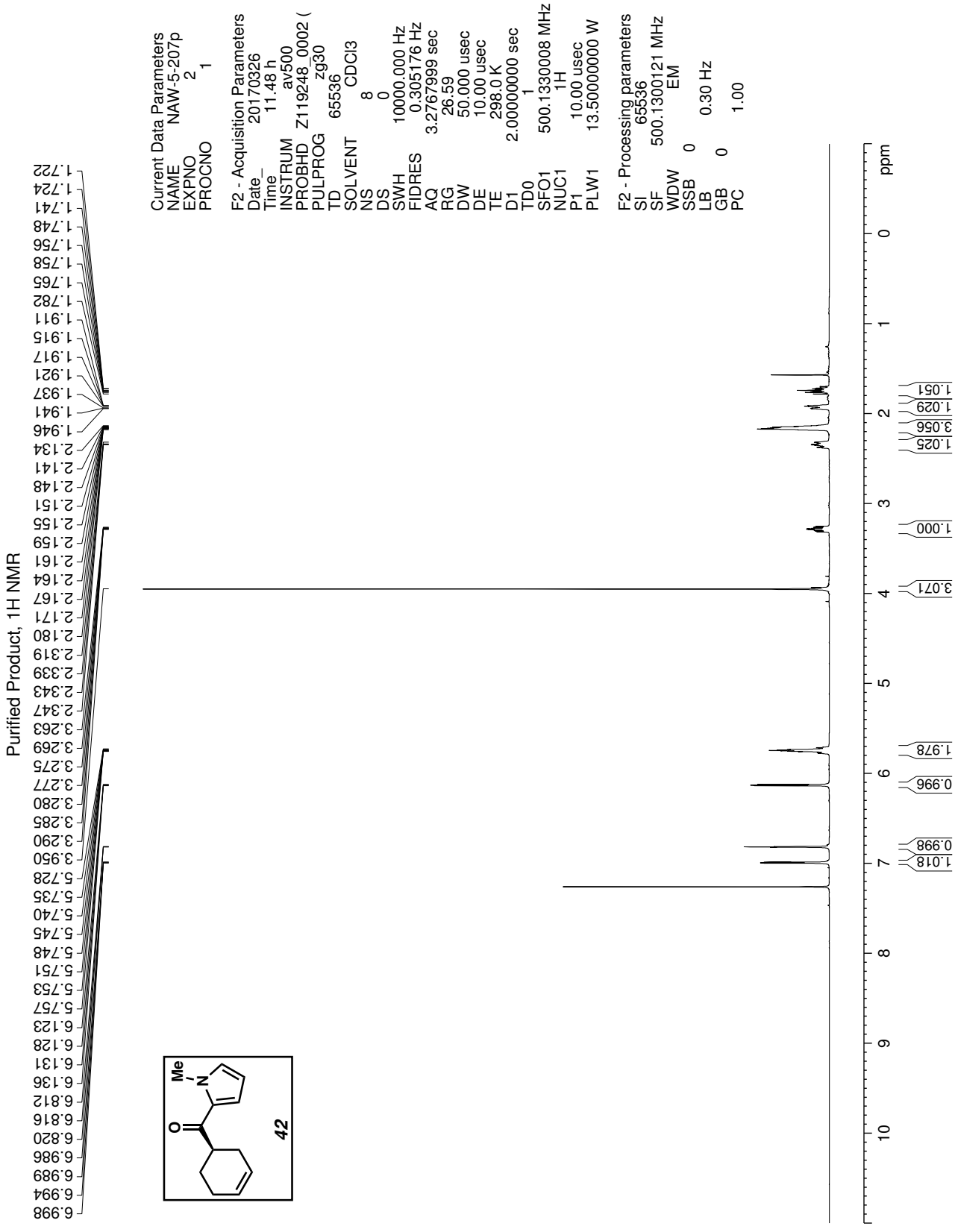

Purified Product, 1H NMR
Current Data Parameters
NAME NAW-5-210p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170322
Time 13.24 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.862
0.876
0.890
1.260
1.266
1.269
1.282
1.294
1.308
1.323
1.333
1.339
1.348
1.351
1.359
1.364
1.656
1.671
1.686
1.700
1.716
2.736
2.751
2.766
3.938
6.106
6.111
6.114
6.119
6.782
6.785
6.789
6.942
6.945
6.950
6.953
Me
N
O
7
Me
32
ppm
0
1
2
3
4
5
6
7
8
9
10
3.056
12.093
2.030
2.028
3.000
0.971
0.980
0.991

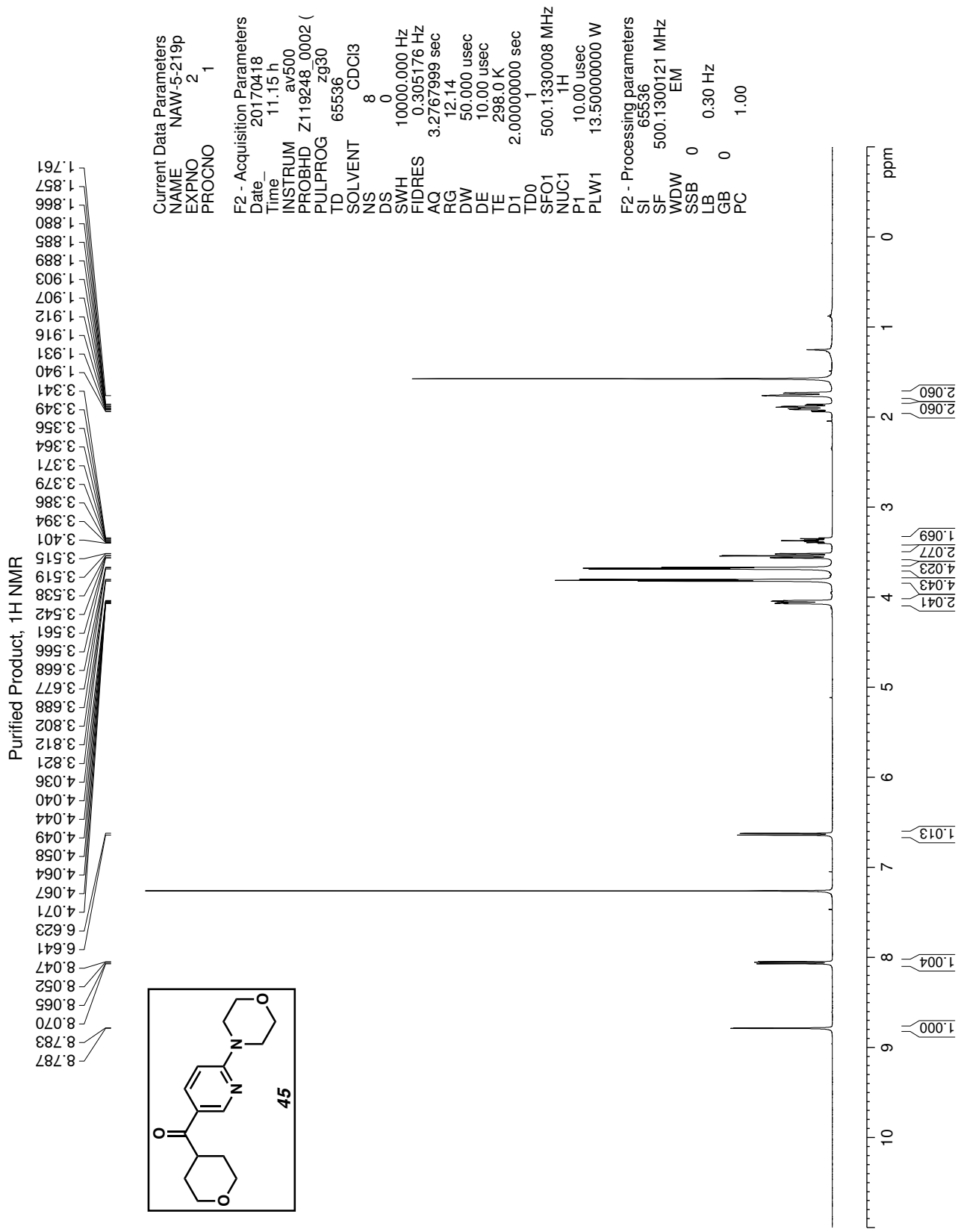

Purified Product, 1H NMR
Current Data Parameters
NAME NAW-5-201p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170301
Time_ 19.49 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
33
ppm
4.062
2.044
2.014
1.013
3.014
0.979
1.000
0.981

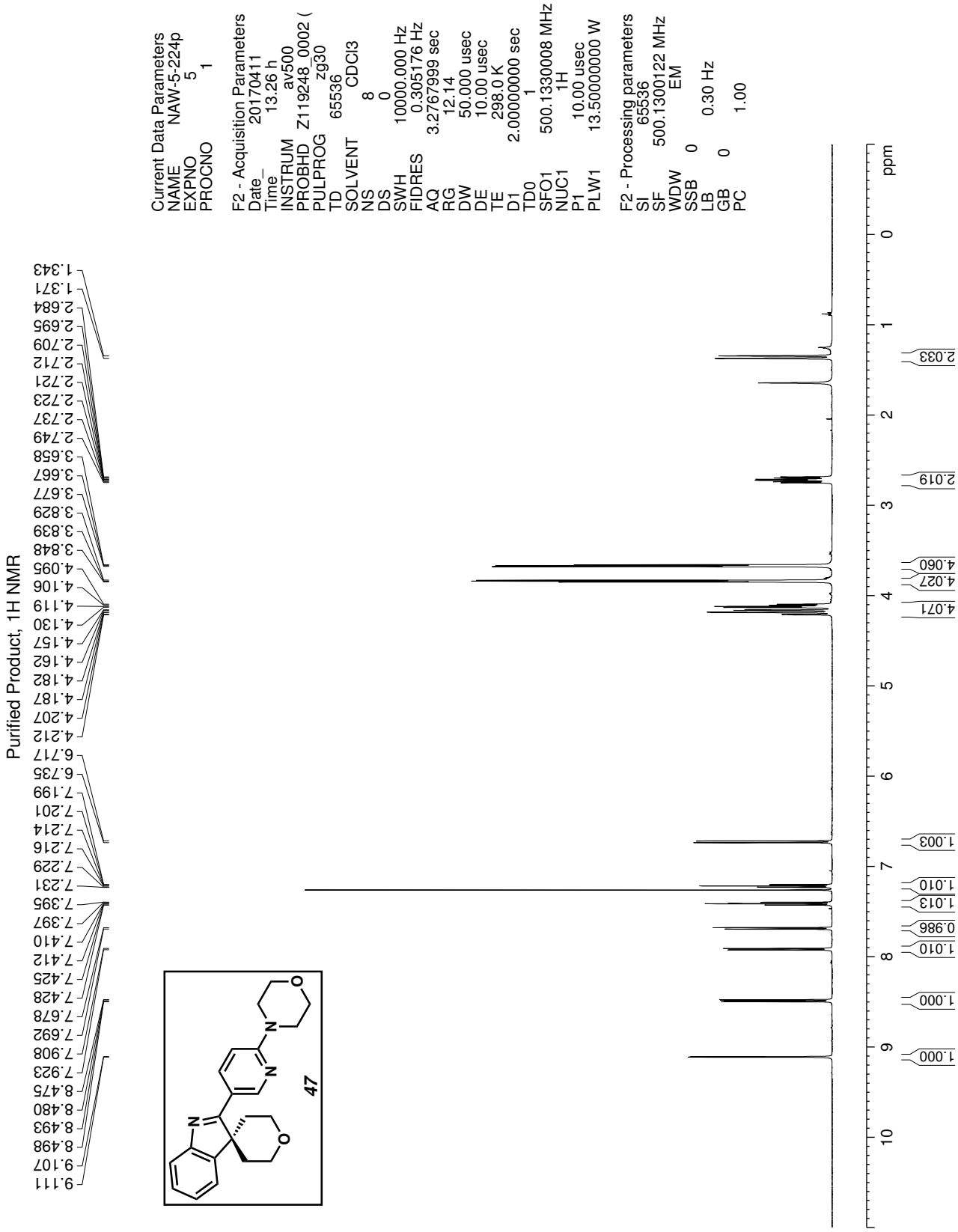

Purified Product, 1H NMR
Current Data Parameters
NAME KJY-4-170p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170322
Time_ 16.46 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 23.34
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
34
ppm
1.000
0.971
0.971
6.978
6.975
6.970
6.967
6.795
6.123
6.119
6.115
6.110
3.933
3.050
3.044
3.038
3.027
3.020
3.014
3.003
2.997
2.991
1.839
1.817
1.732
1.729
1.726
1.723
1.701
1.548
1.543
1.524
1.518
1.498
1.474
1.467
1.431
1.408
1.388
1.380
1.362
1.356
1.342
1.336
1.330
1.317
0.971
3.012
1.020
4.118
1.079
2.142
3.220


Purified Product, 1H NMR
Current Data Parameters
NAME KJY-04-201P
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time 14.42 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 19.06
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300124 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.030
7.022
6.743
6.110
6.105
3.895
1.360
1.000
1.060
0.992
3.013
9.078
ppm
35

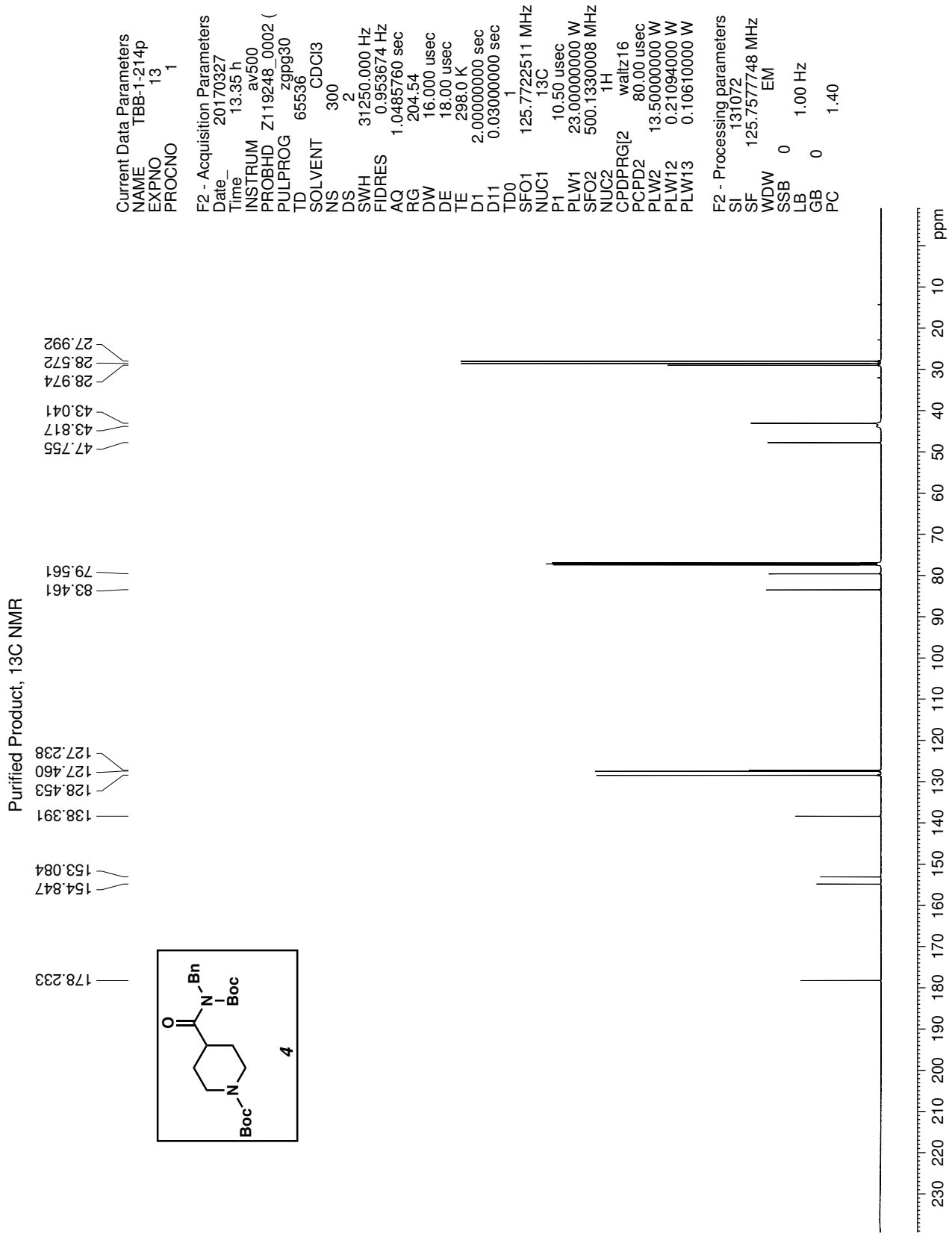

Purified Product, 1H NMR
Current Data Parameters
NAME TBB-1-254p-diast.1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170402
Time_ 15.53 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
O
MeO
O
40
ppm
4.078
2.048
2.043
0.999
1.015
3.000
3.000
0.981
1.010
0.985

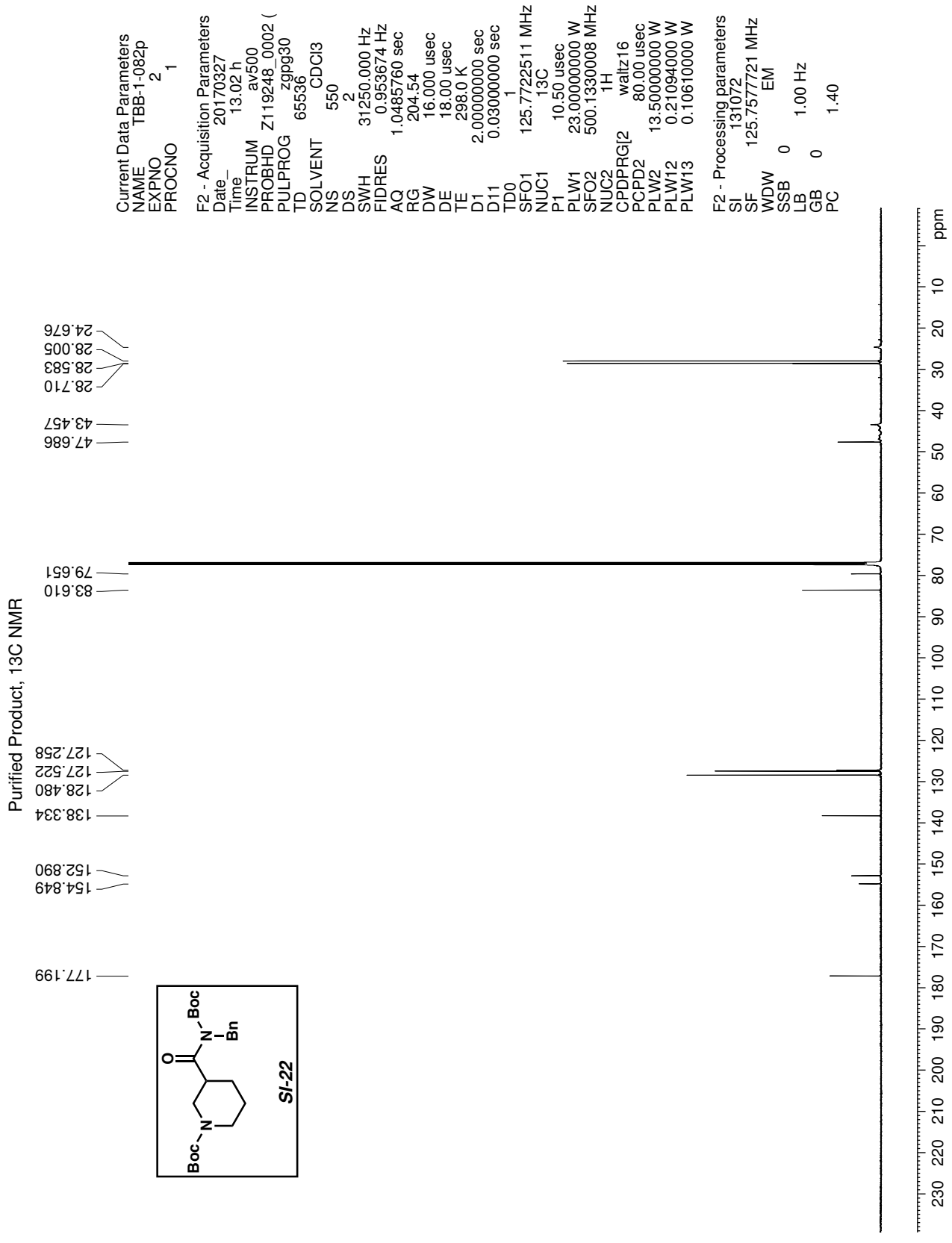

Purified Product, 1H NMR
Current Data Parameters
NAME NAW-5-207p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170326
Time_ 11.48 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 26.59
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
N
O
42
ppm
1.051
1.029
3.056
1.025
1.000
3.071
1.978
0.996
0.998
1.018

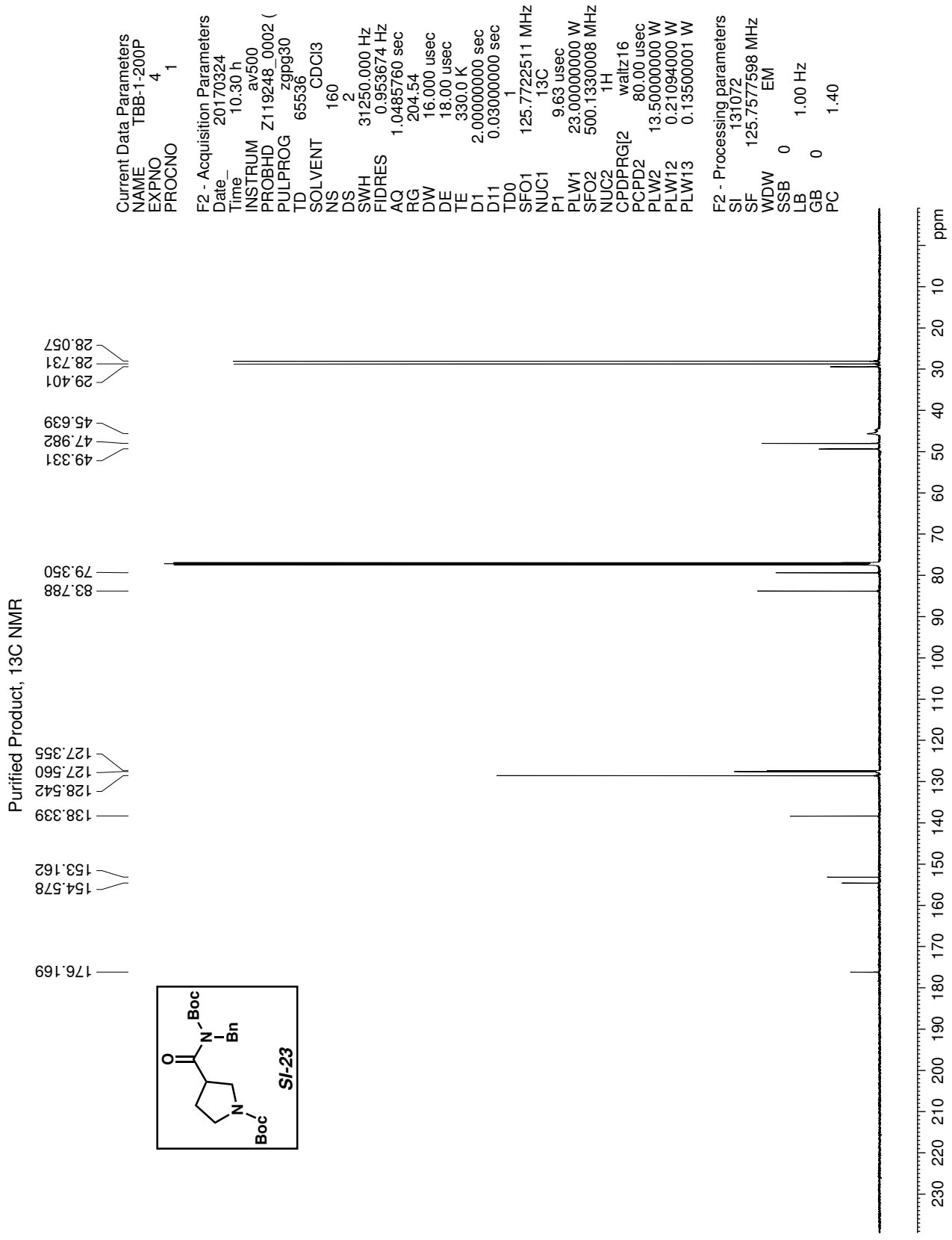

Purified Product, 1H NMR
Current Data Parameters
NAME NAW-5-219p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170418
Time_ 11.15 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
45
ppm

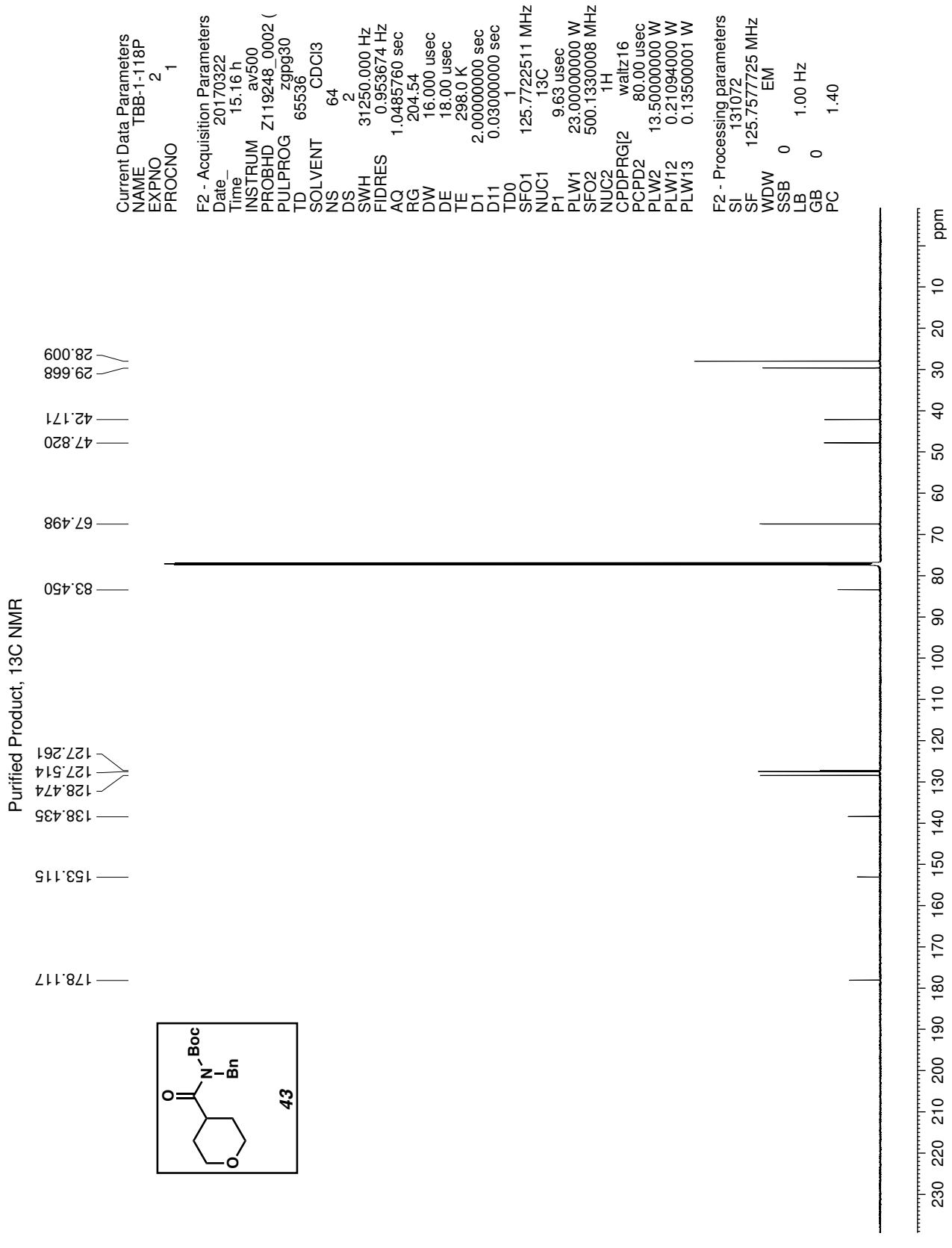

Purified Product, 1H NMR
Current Data Parameters
NAME NAW-5-224p
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170411
Time_ 13.26 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
47
ppm

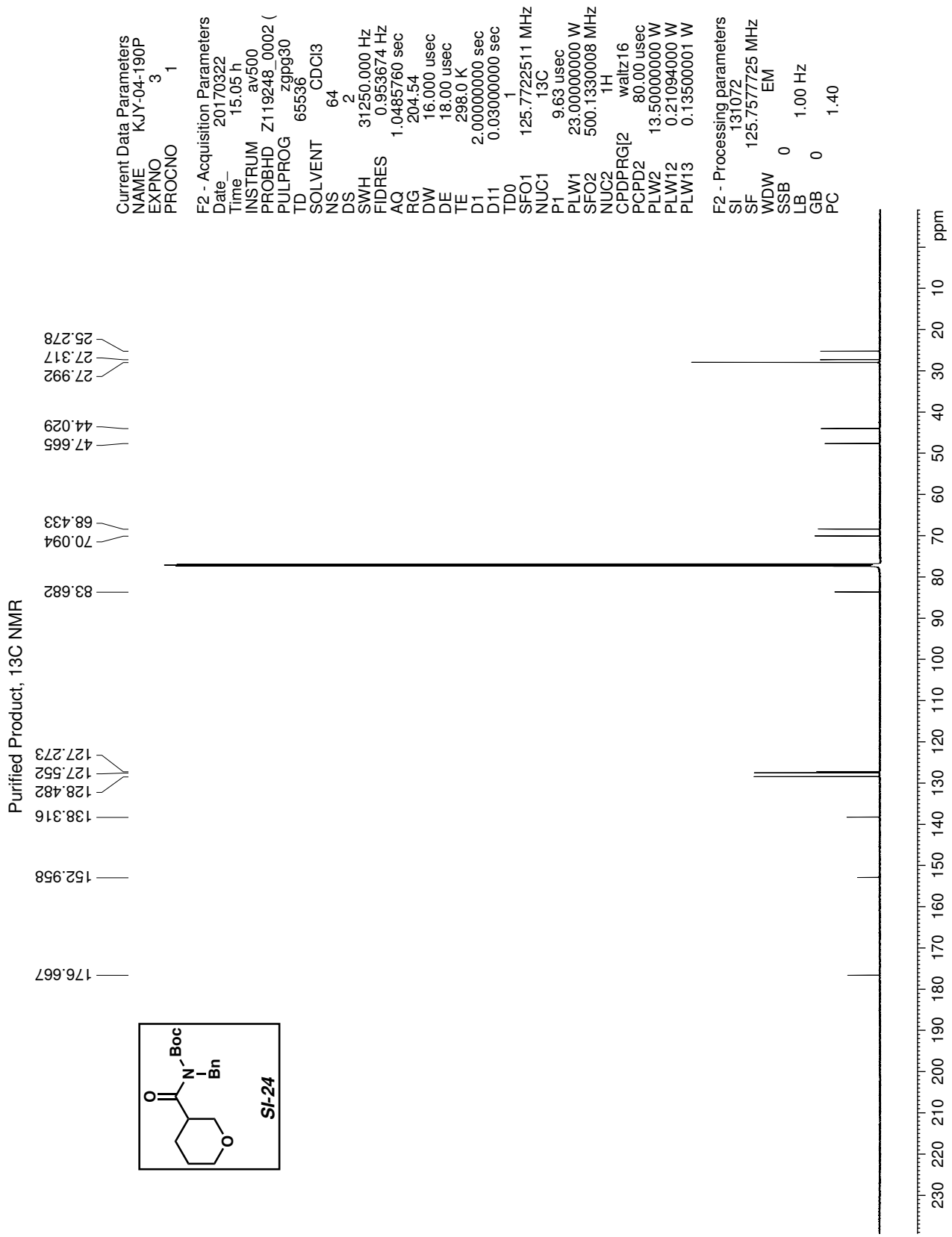
^{13}C NMR Spectra

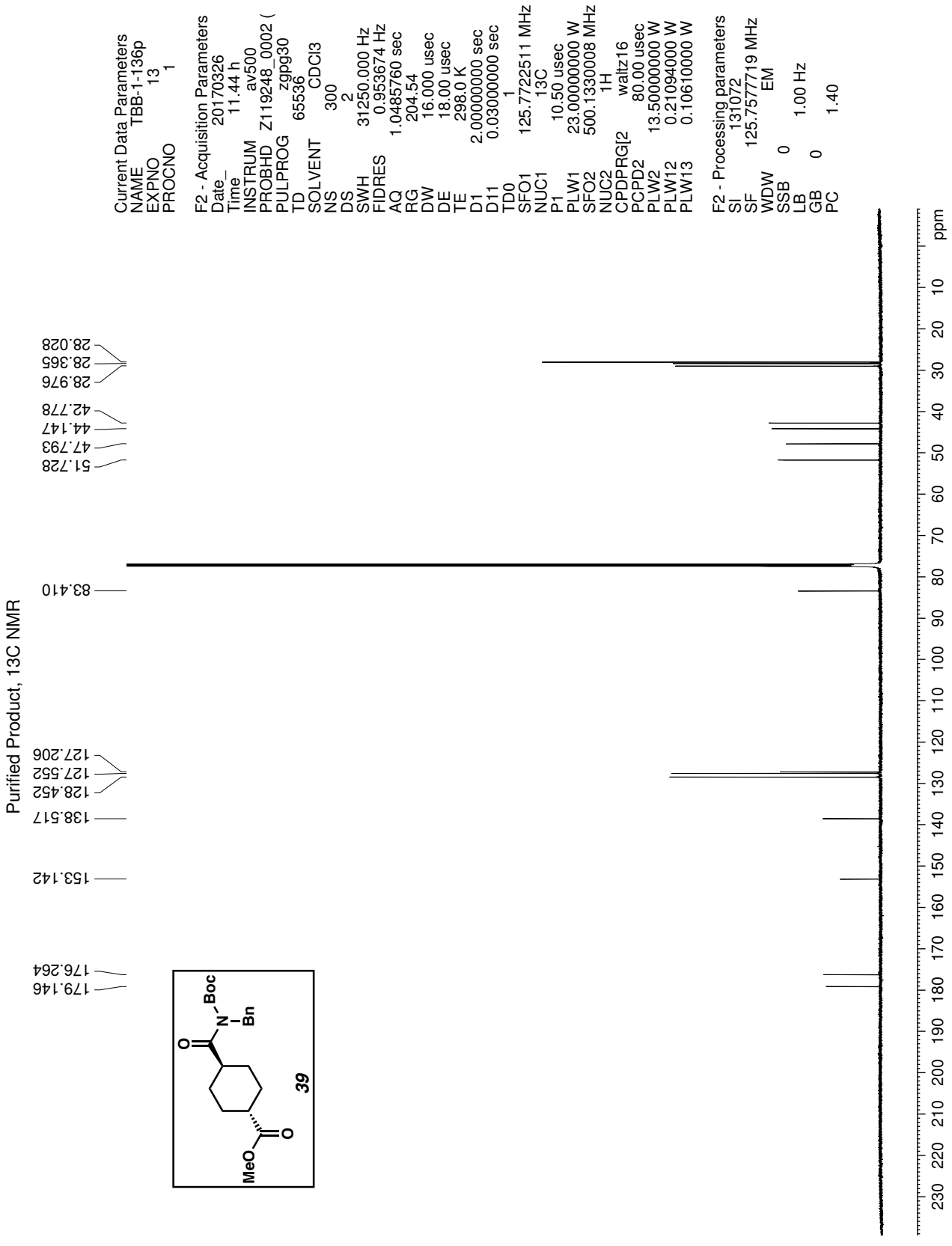

Purified Product, 13C NMR
178.233
154.847
153.084
138.391
128.453
127.460
127.238
83.461
79.561
47.755
43.817
43.041
28.974
28.572
27.992
Boc
N
Bn
O
4
Current Data Parameters
NAME TBB-1-214p
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170327
Time 13.35 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577748 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

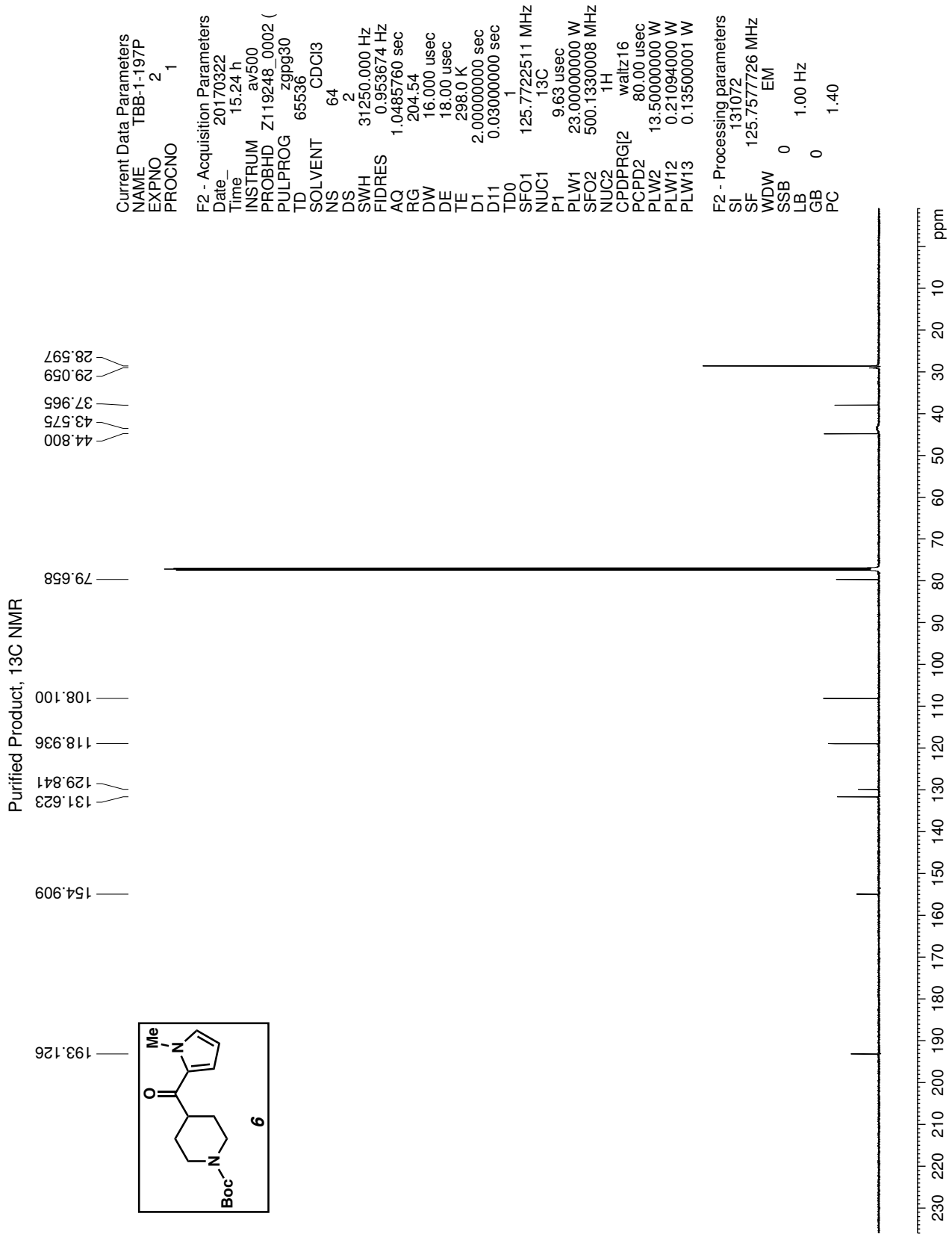

Purified Product, 13C NMR
177.199
154.849
152.890
138.334
128.480
127.522
127.258
83.610
79.651
47.686
43.457
28.710
28.583
28.005
24.676
Boc
N
O
N
Boc
Bn
SI-22
Current Data Parameters
NAME TBB-1-082p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170327
Time 13.02 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 550
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577721 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

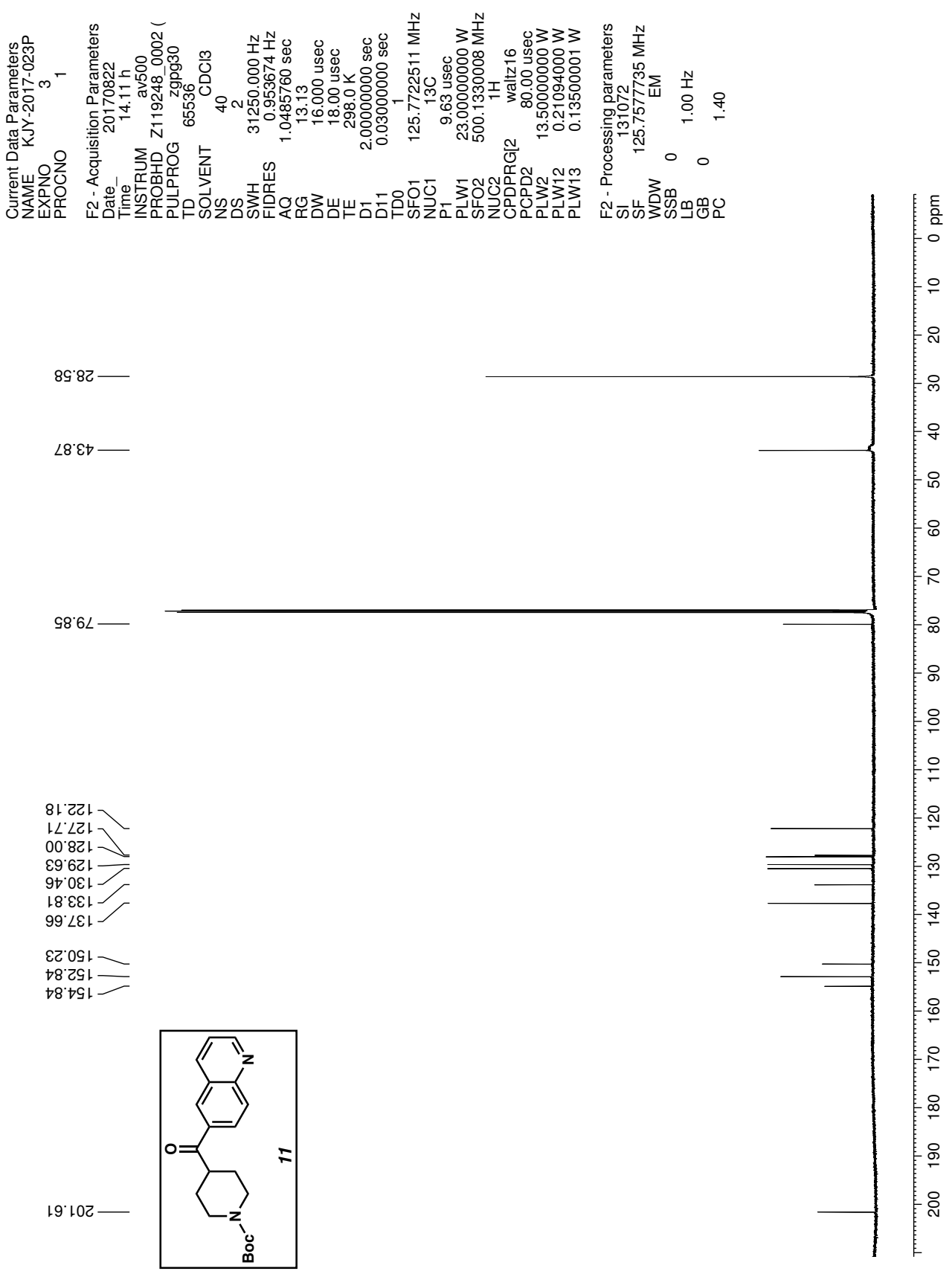

Purified Product, 13C NMR
Current Data Parameters
NAME TBB-1-200P
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170324
Time 10.30 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 160
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 330.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577598 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
176.169
154.578
153.162
138.339
128.542
127.560
127.355
83.788
79.350
49.331
47.982
45.639
29.401
28.731
28.057
Boc
N
Bn
O
N
Boc
SI-23
ppm

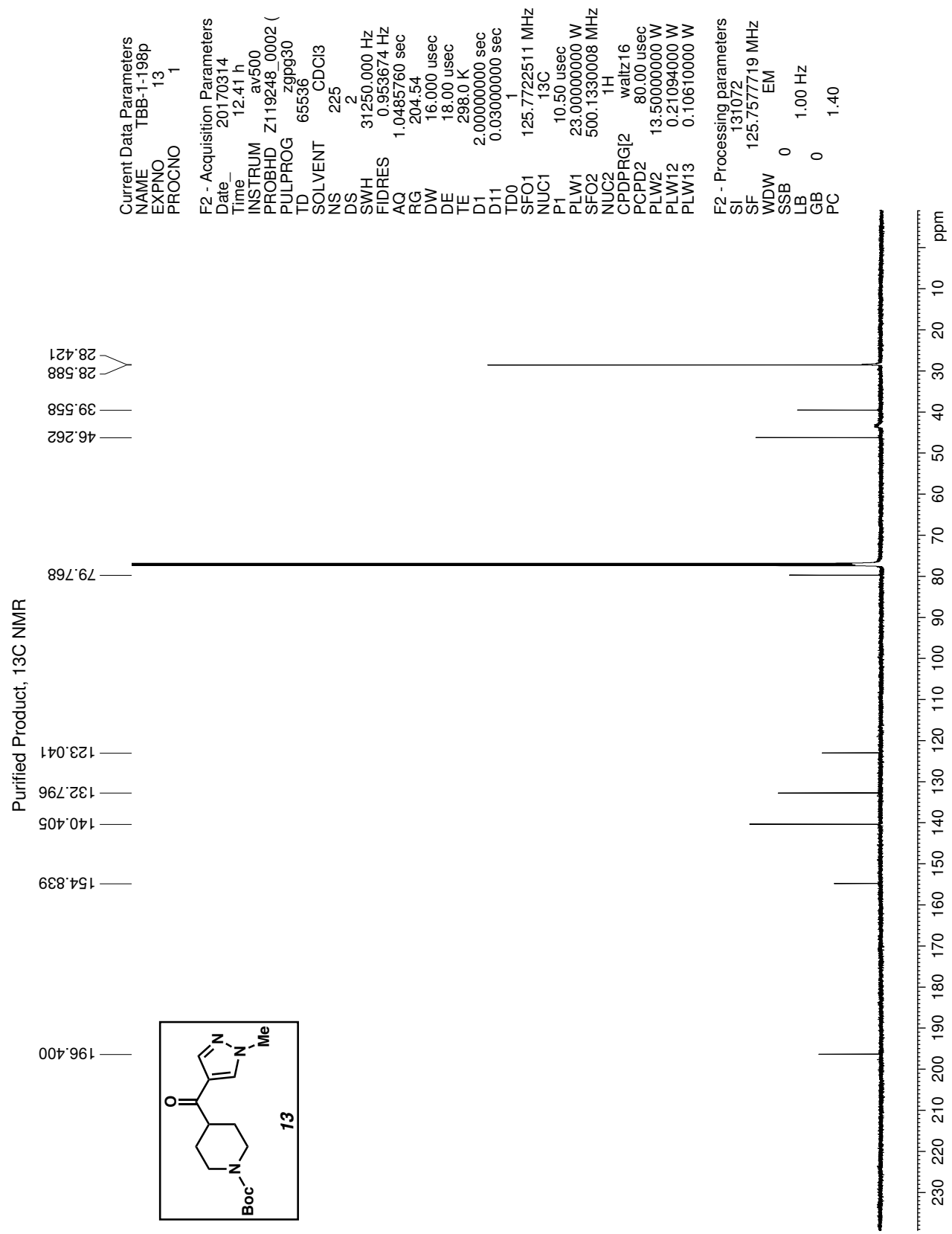

Purified Product, 13C NMR
Current Data Parameters
NAME TBB-1-118P
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170322
Time 15.16 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577725 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
178.117
153.115
138.435
128.474
127.514
127.261
83.450
67.498
47.820
42.171
29.668
28.009
Boc
N
Bn
O
O
43
ppm

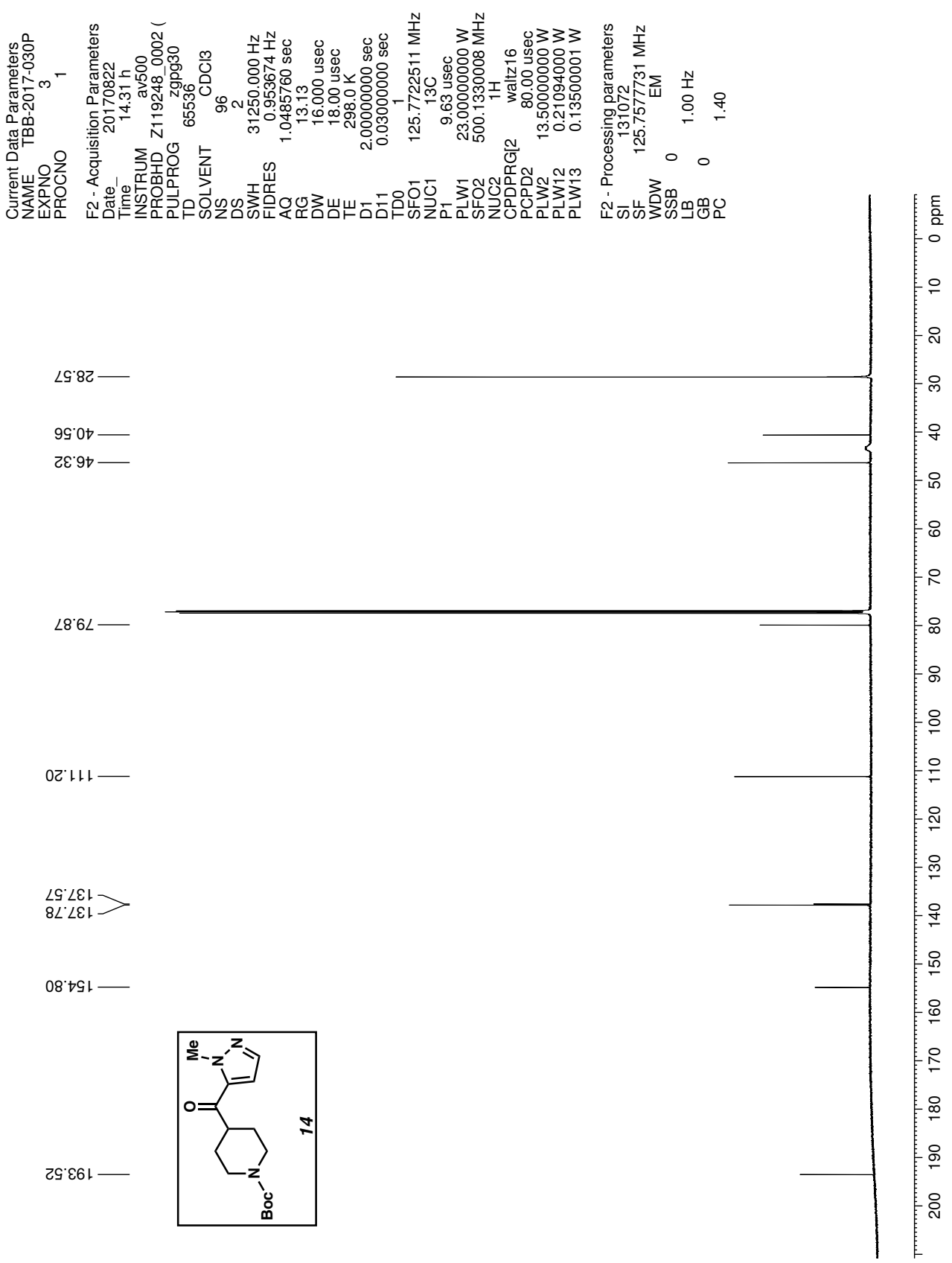

Purified Product, 13C NMR
SI-24
Boc
N
Bn
O
O
176.667
152.958
138.316
128.482
127.552
127.273
83.682
70.094
68.433
47.665
44.029
27.992
27.317
25.278
Current Data Parameters
NAME KJY-04-190P
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170322
Time 15.05 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577725 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm

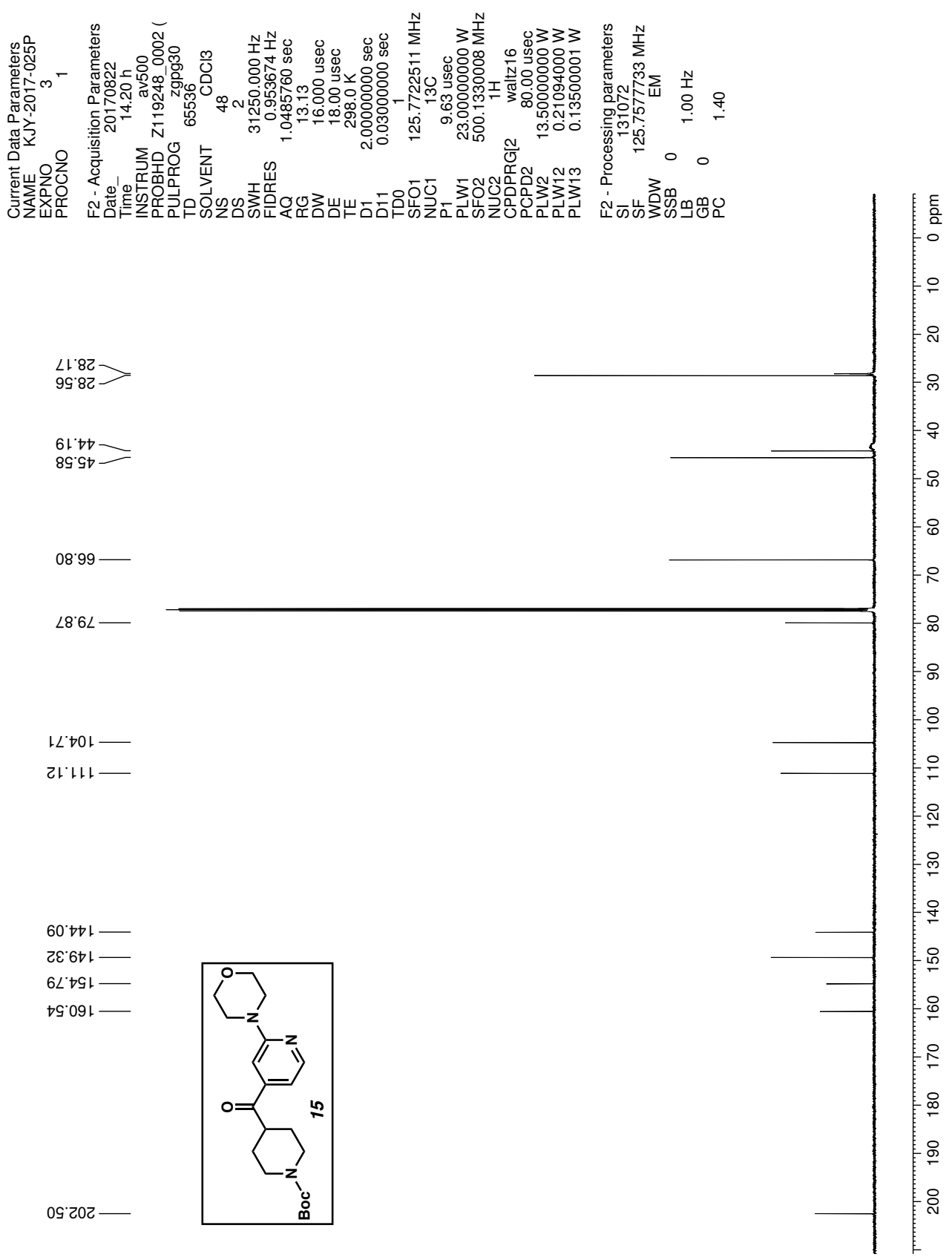

Purified Product, 13C NMR
Current Data Parameters
NAME TBB-1-136p
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170326
Time 11.44 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577719 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
179.146
176.264
153.142
138.517
128.452
127.552
127.206
83.410
51.728
47.793
44.147
42.778
28.976
28.365
28.028
Boc
N
Bn
O
MeO
O
39
ppm

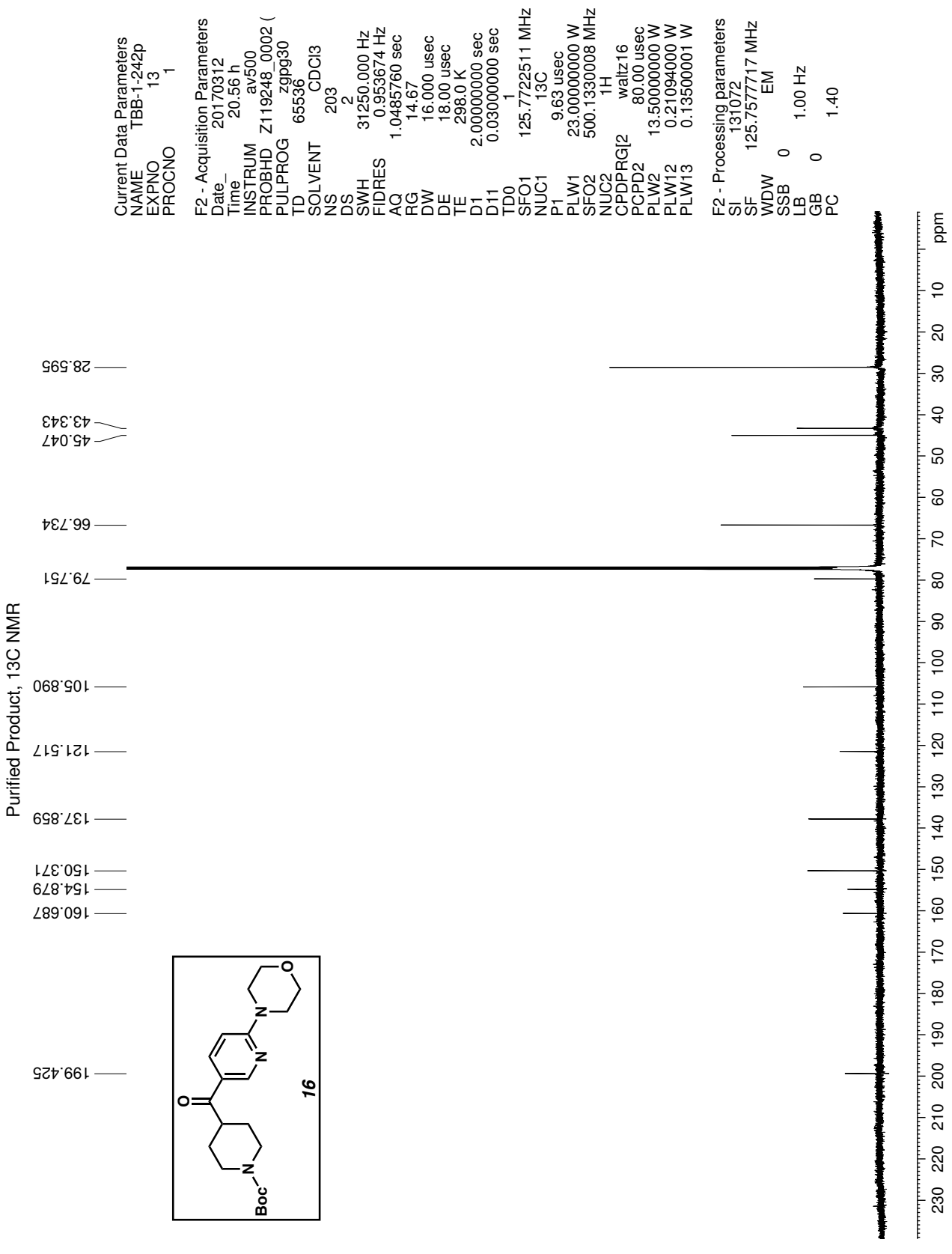

Purified Product, 13C NMR
193.126
154.909
131.623
129.841
118.936
108.100
79.658
44.800
43.575
37.965
29.059
28.597
Me
N
O
N
Boc
6
Current Data Parameters
NAME TBB-1-197P
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170322
Time 15.24 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577726 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm

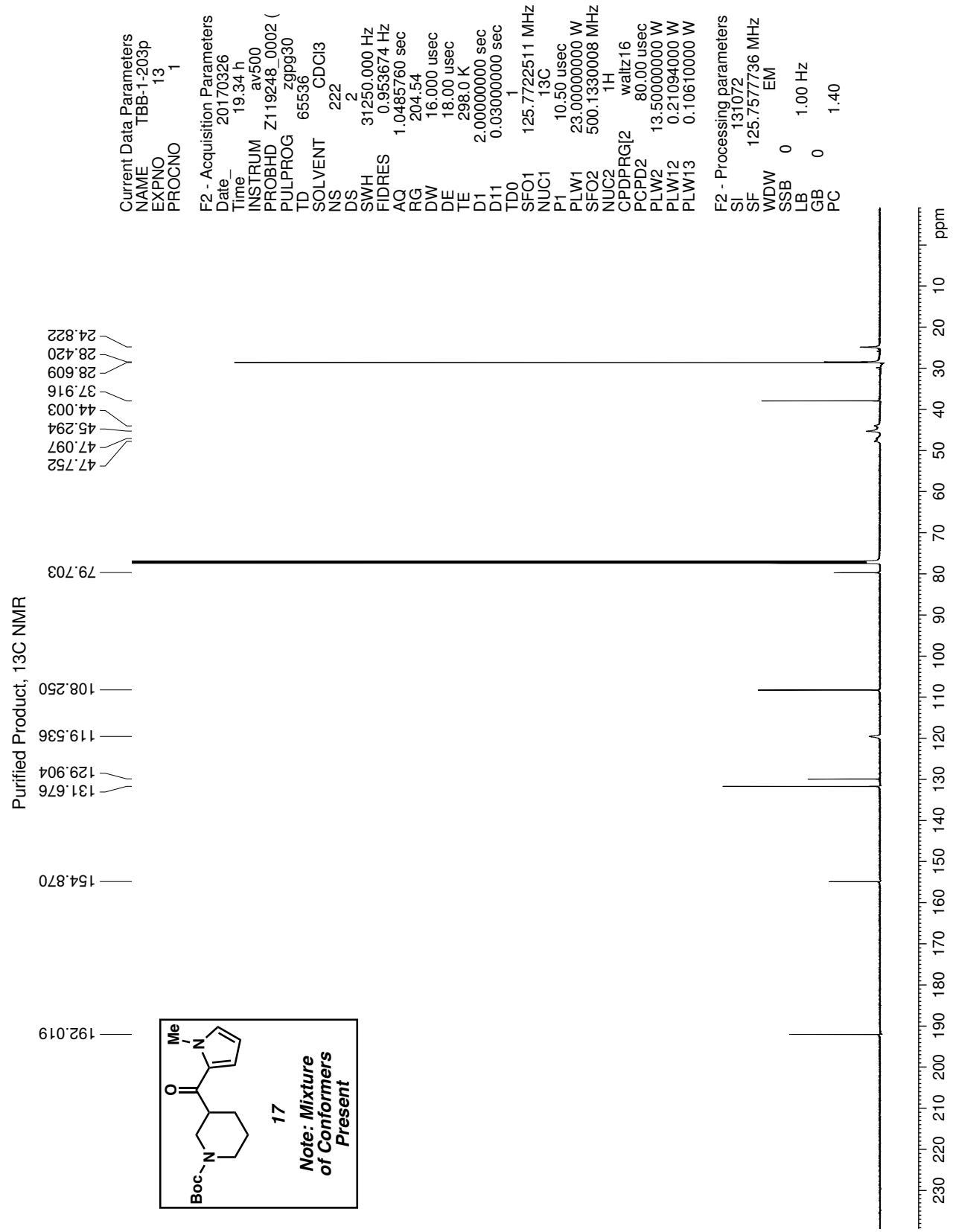

Current Data Parameters
NAME KJY-2017-023P
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170822
Time 14.11 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 40
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 13.13
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577735 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
201.61
154.84
152.84
150.23
137.66
133.81
130.46
129.63
128.00
127.71
122.18
79.85
43.87
28.58
O
N
Boc
11
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

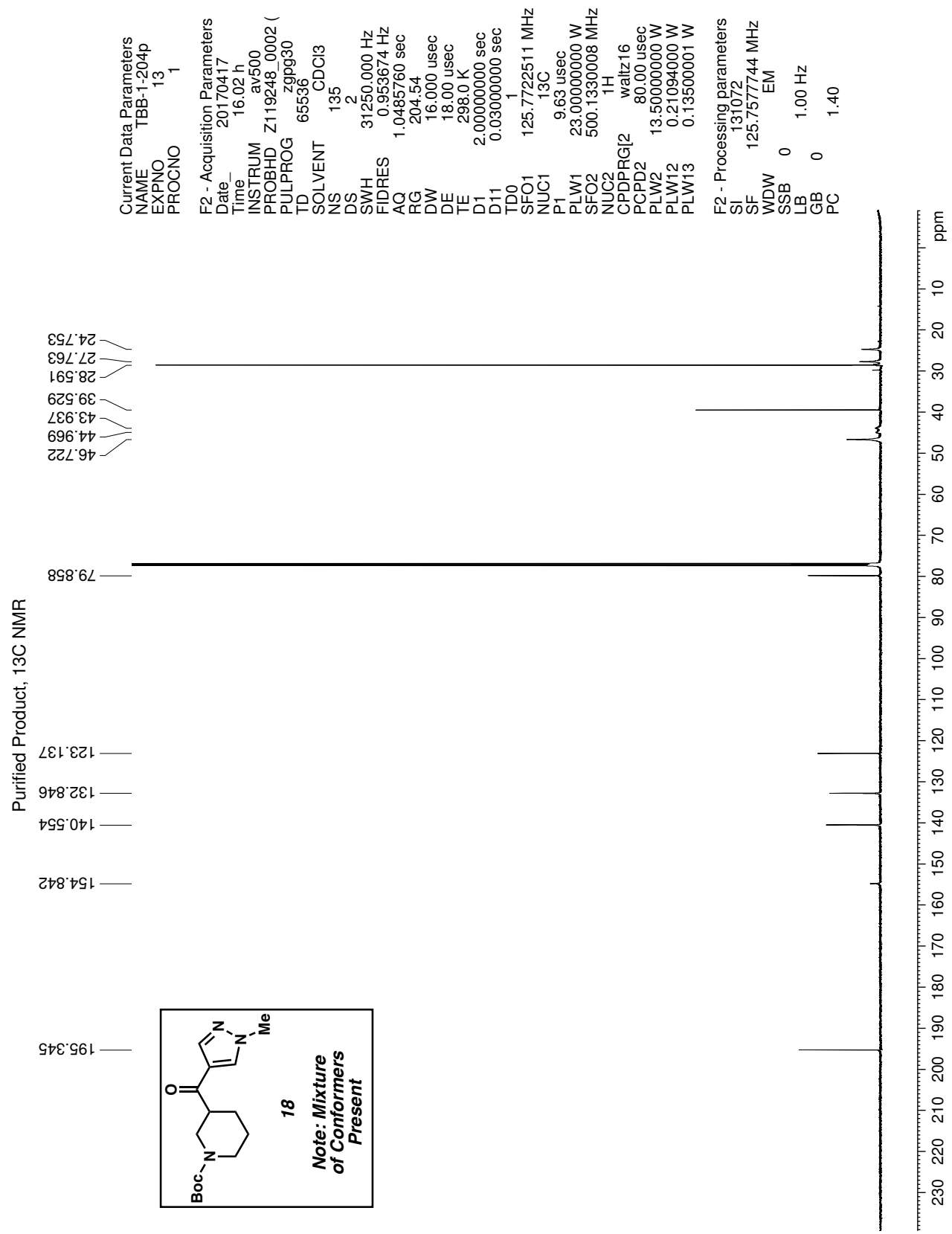

Purified Product, 13C NMR
Current Data Parameters
NAME TBB-1-198p
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170314
Time 12.41 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 225
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577719 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
196.400
154.839
140.405
132.796
123.041
79.768
46.262
39.558
28.588
28.421
ppm
13

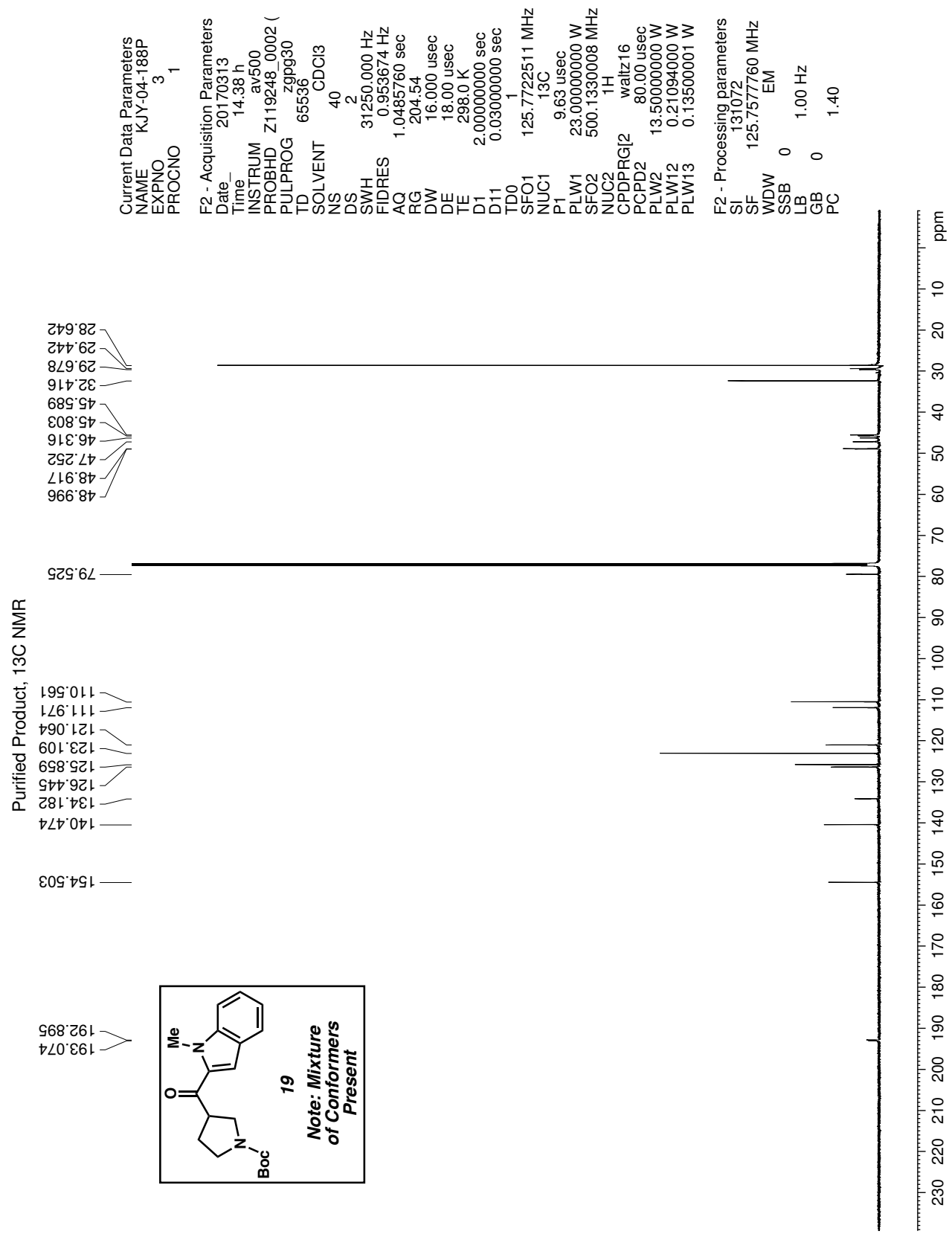

Current Data Parameters
NAME TBB-2017-030P
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170822
Time 14.31 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 96
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 13.13
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577731 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
193.52
154.80
137.78
137.57
111.20
79.87
46.32
40.56
28.57
Me
O
Boc
14
0 ppm

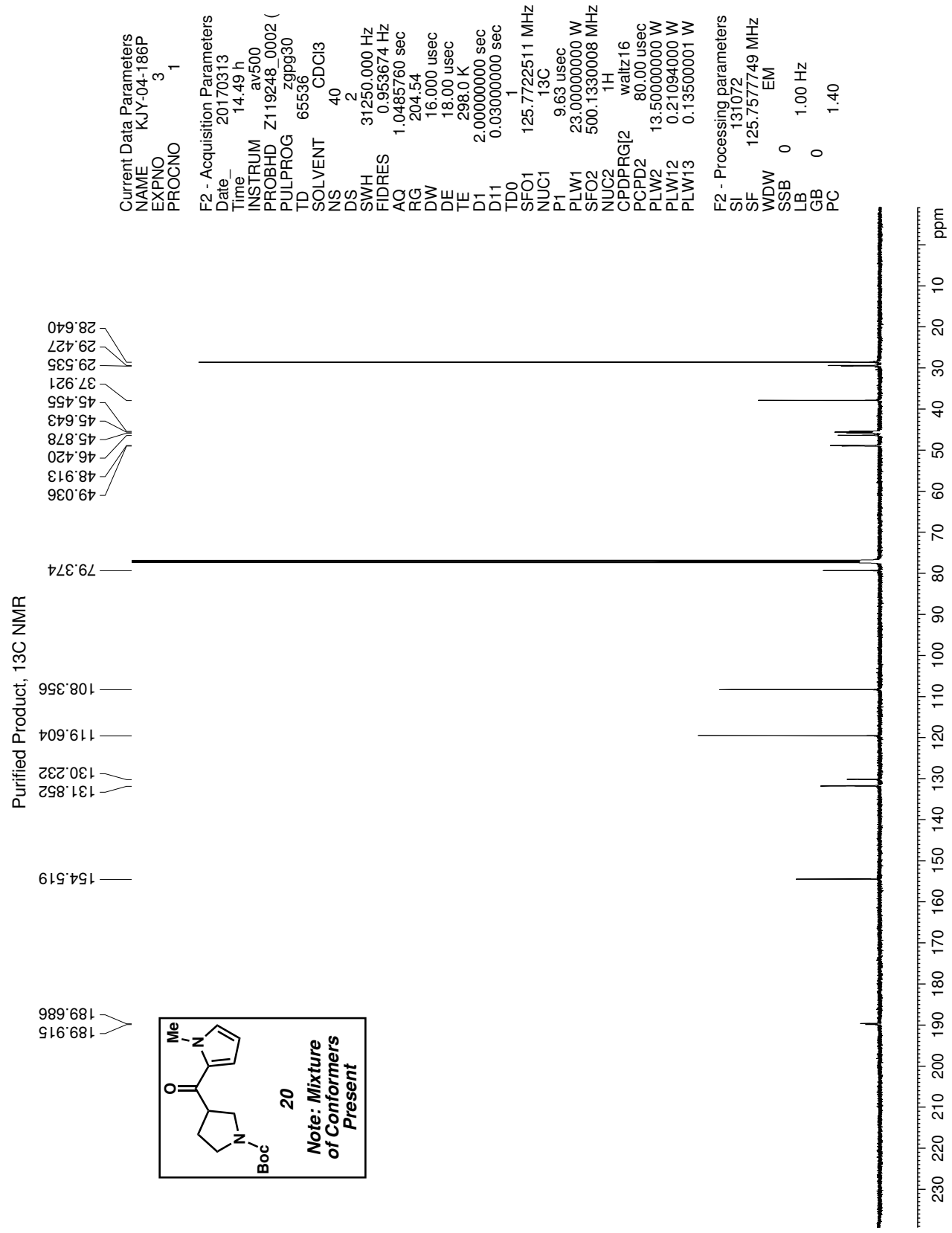

Current Data Parameters
NAME KJY-2017-025P
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170822
Time 14.20 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 48
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 13.13
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577733 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
28.17
28.56
44.19
45.58
66.80
79.87
104.71
111.12
144.09
149.32
154.79
160.54
202.50
O
N
N
O
N
Boc
15
0 ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200

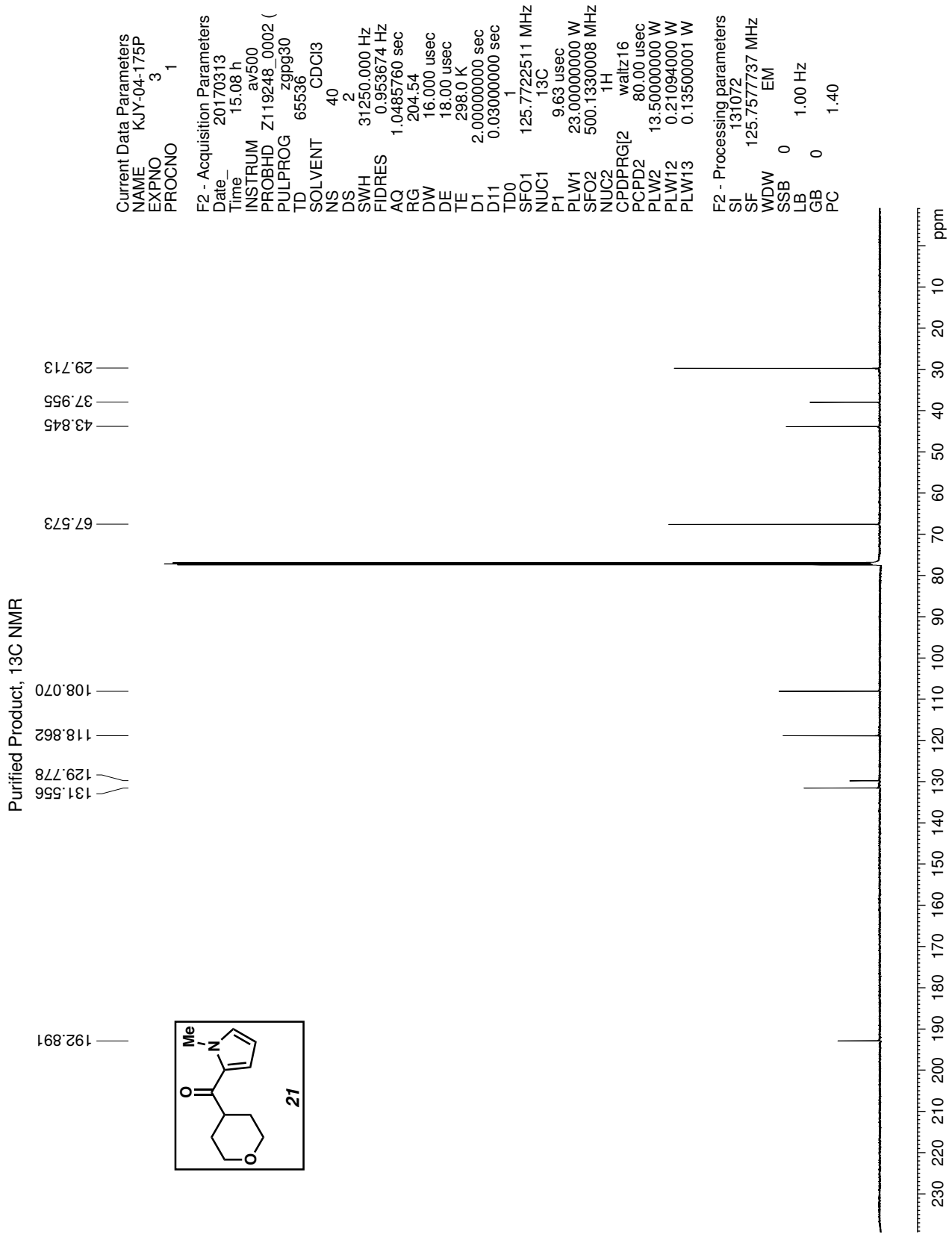

Purified Product, 13C NMR
Current Data Parameters
NAME TBB-1-242p
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170312
Time 20.56 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 203
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 14.67
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577717 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
199.425
160.687
154.879
150.371
137.859
121.517
105.890
79.751
66.734
45.047
43.343
28.595
O
N
N
O
N
Boc
16
ppm

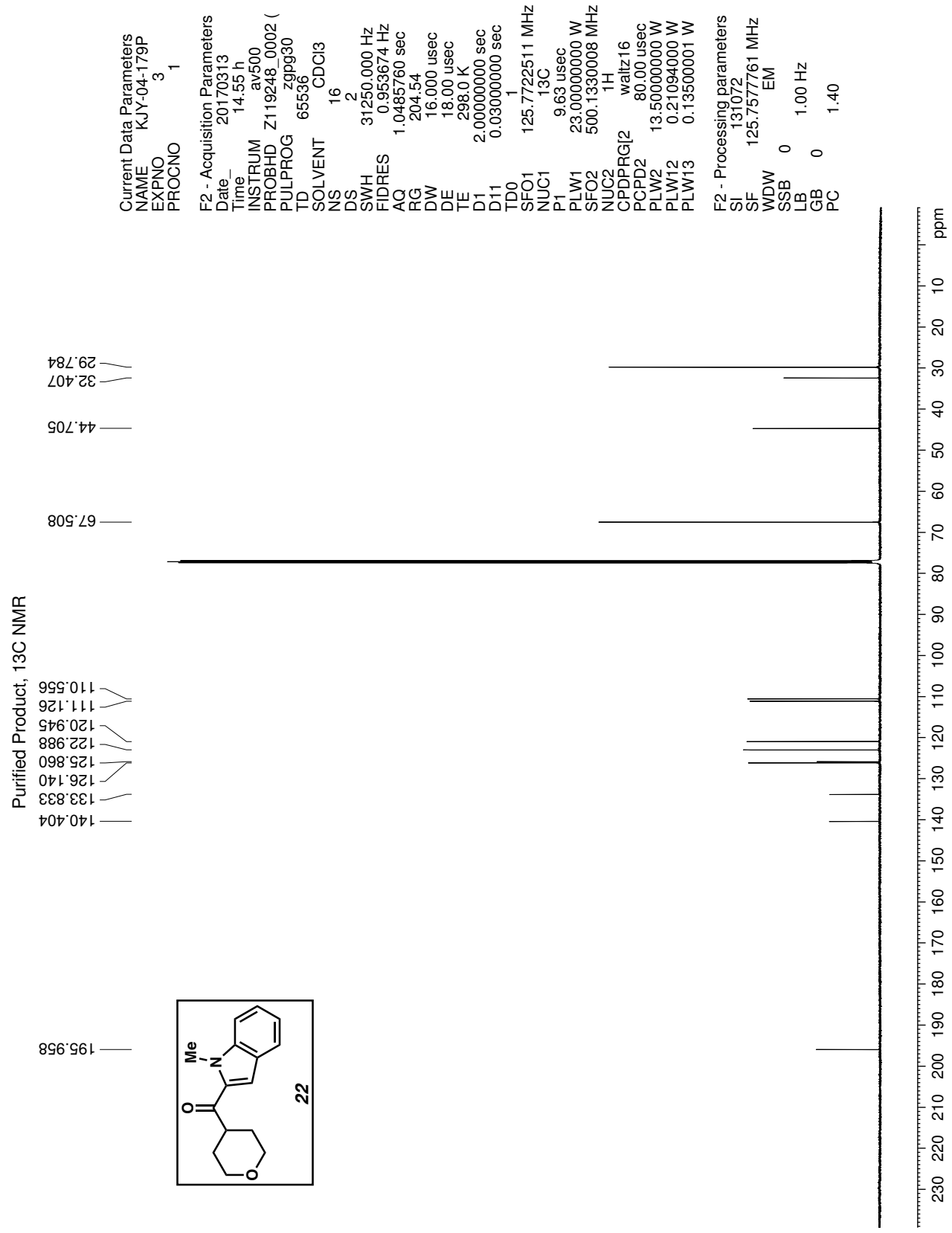

Purified Product, 13C NMR
Current Data Parameters
NAME TBB-1-203p
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170326
Time 19.34 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 222
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
192.019
154.870
131.676
129.904
119.536
108.250
79.703
47.752
47.097
45.294
44.003
37.916
28.609
28.420
24.822
Me
N
O
Boc
N
17
Note: Mixture of Conformers Present
ppm

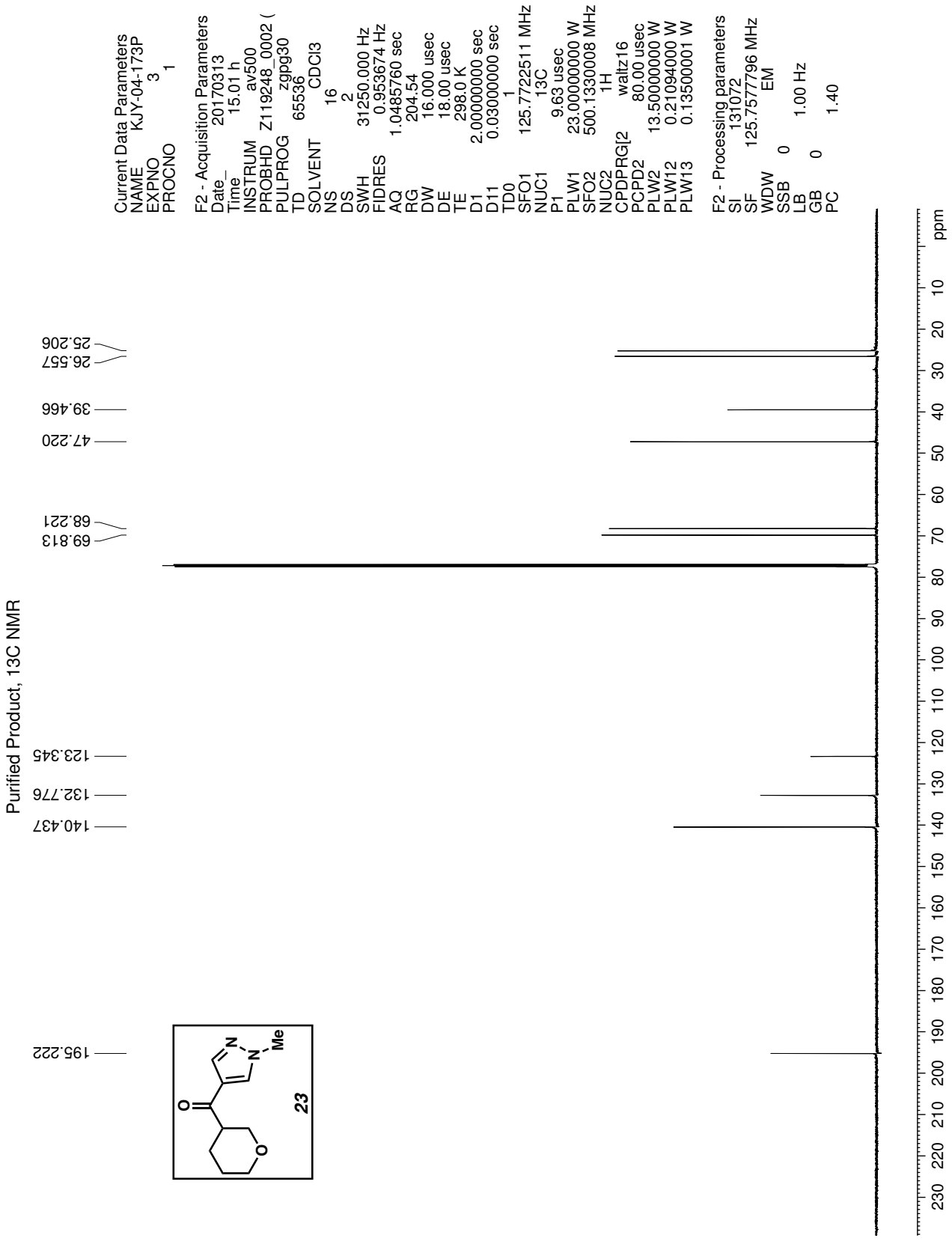

Purified Product, 13C NMR
Current Data Parameters
NAME TBB-1-204p
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170417
Time 16.02 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 135
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577744 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
195.345
154.842
140.554
132.846
123.137
79.858
46.722
44.969
43.937
39.529
28.591
27.763
24.753
Boc
N
O
N
N
Me
18
Note: Mixture of Conformers Present
ppm

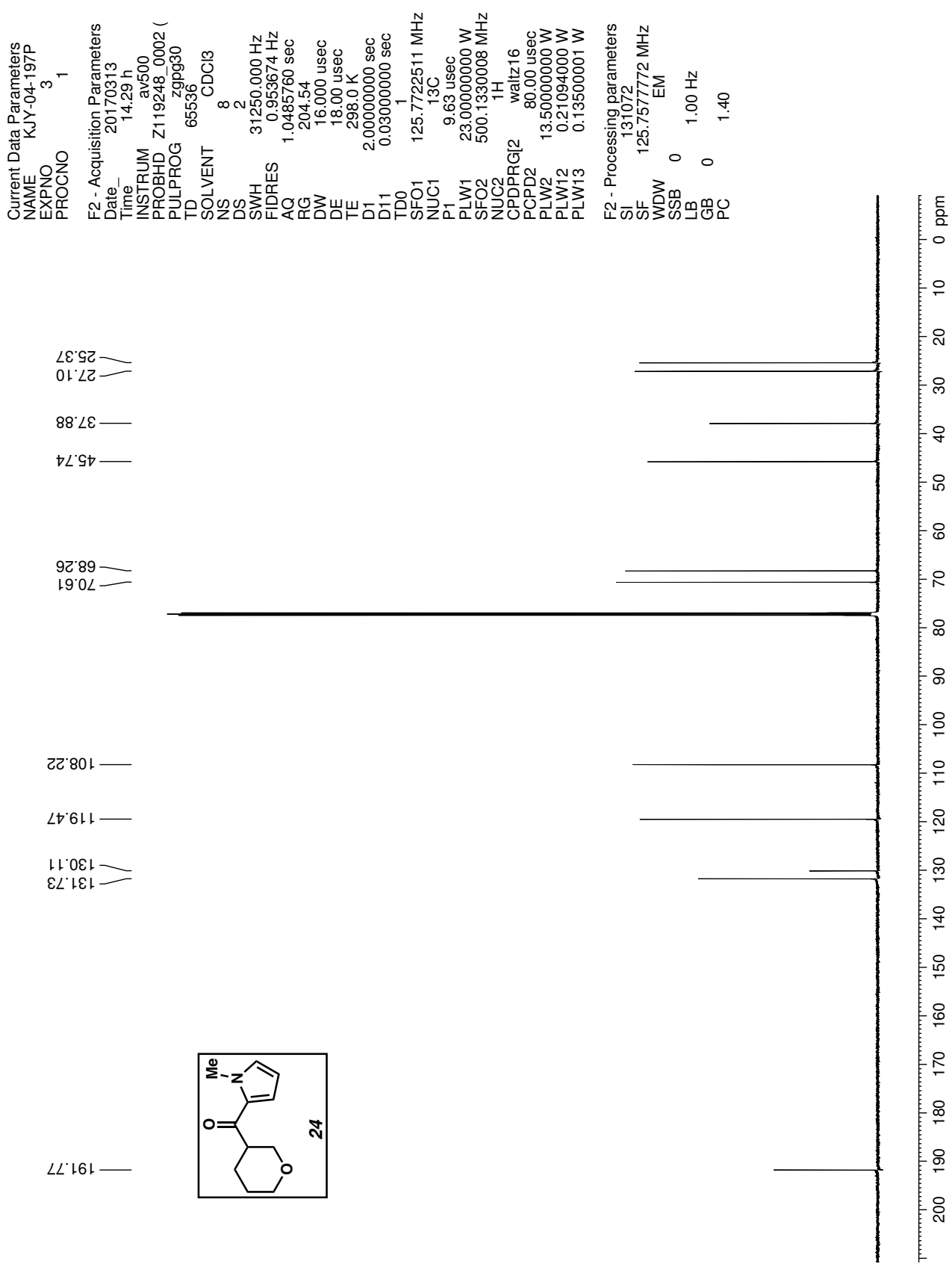

Purified Product, 13C NMR
Current Data Parameters
NAME KJY-04-188P
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time 14.38 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 40
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577760 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
193.074
192.895
154.503
140.474
134.182
126.445
125.859
123.109
121.064
111.971
110.561
79.525
48.996
48.917
47.252
46.316
45.803
45.589
32.416
29.678
29.442
28.642
Me
N
O
N
Boc
19
Note: Mixture of Conformers Present
ppm

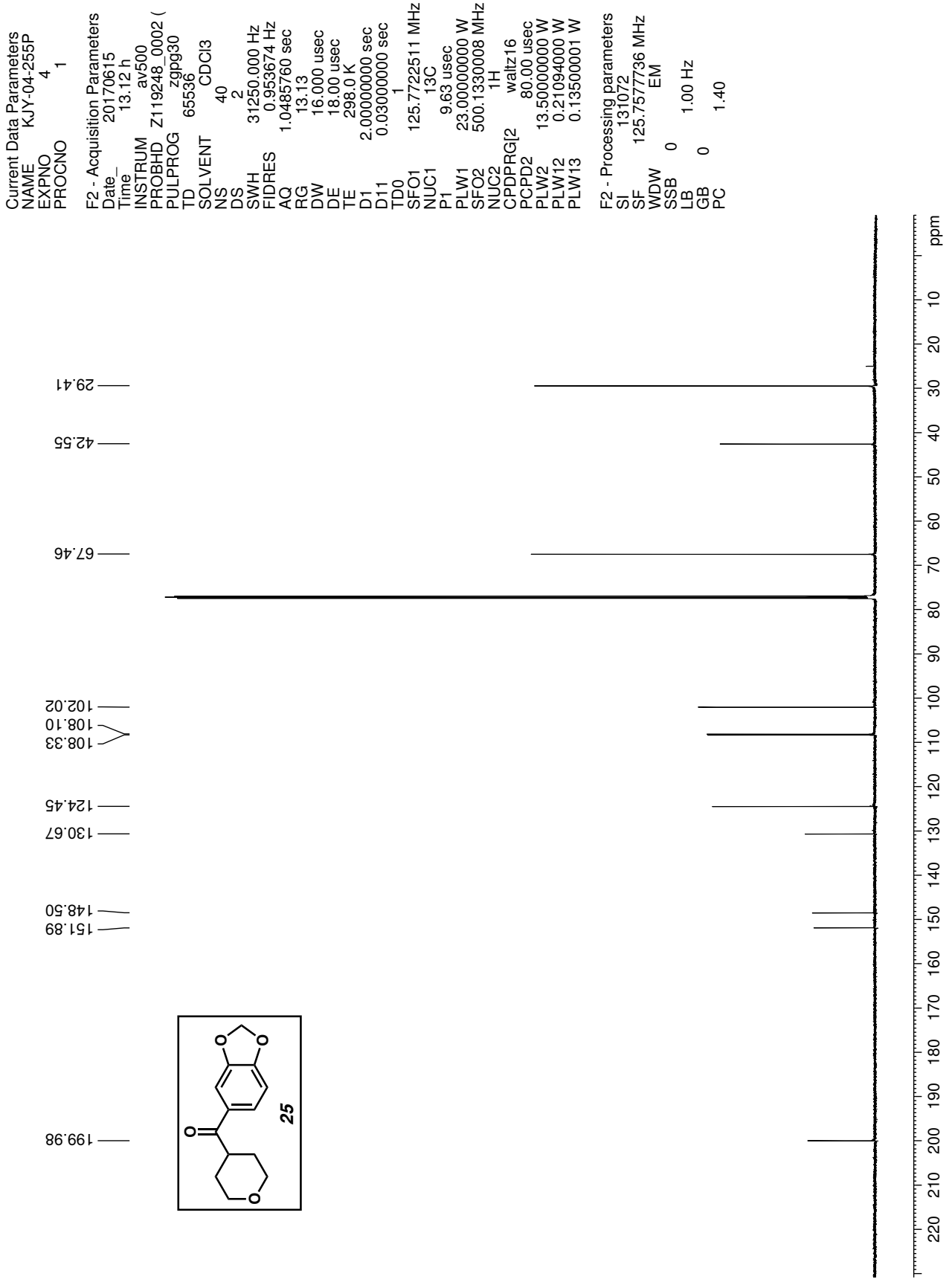

Purified Product, 13C NMR
189.915
189.686
154.519
131.852
130.232
119.604
108.356
79.374
49.036
48.913
46.420
45.878
45.643
45.455
37.921
29.535
29.427
28.640
Me
N
O
N
Boc
20
Note: Mixture of Conformers Present
Current Data Parameters
NAME KJY-04-186P
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time 14.49 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 40
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577749 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

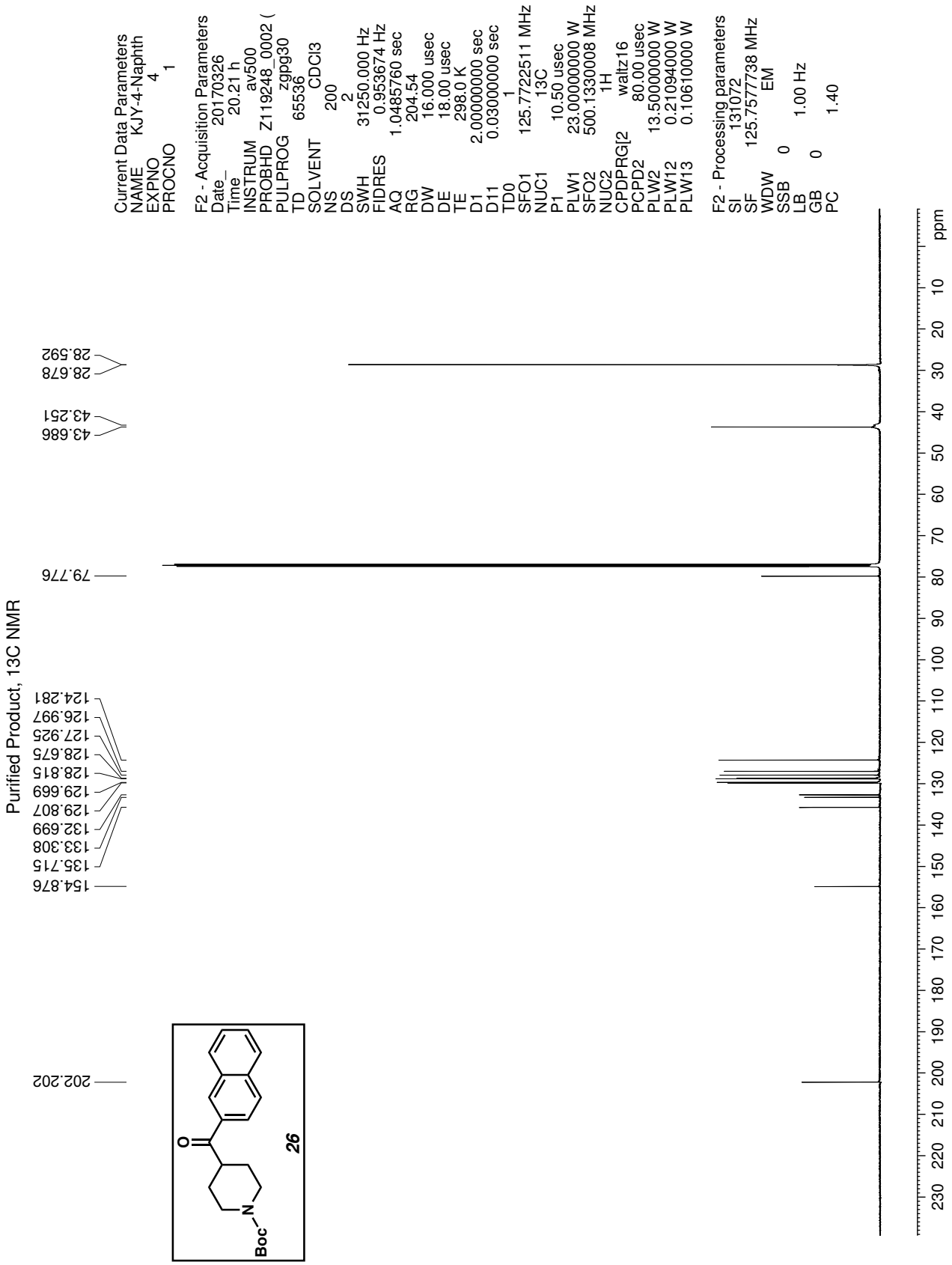

Purified Product, 13C NMR
Current Data Parameters
NAME KJY-04-175P
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time 15.08 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 40
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577737 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
192.891
131.556
129.778
118.862
108.070
67.573
43.845
37.955
29.713
Me
N
O
O
21
ppm

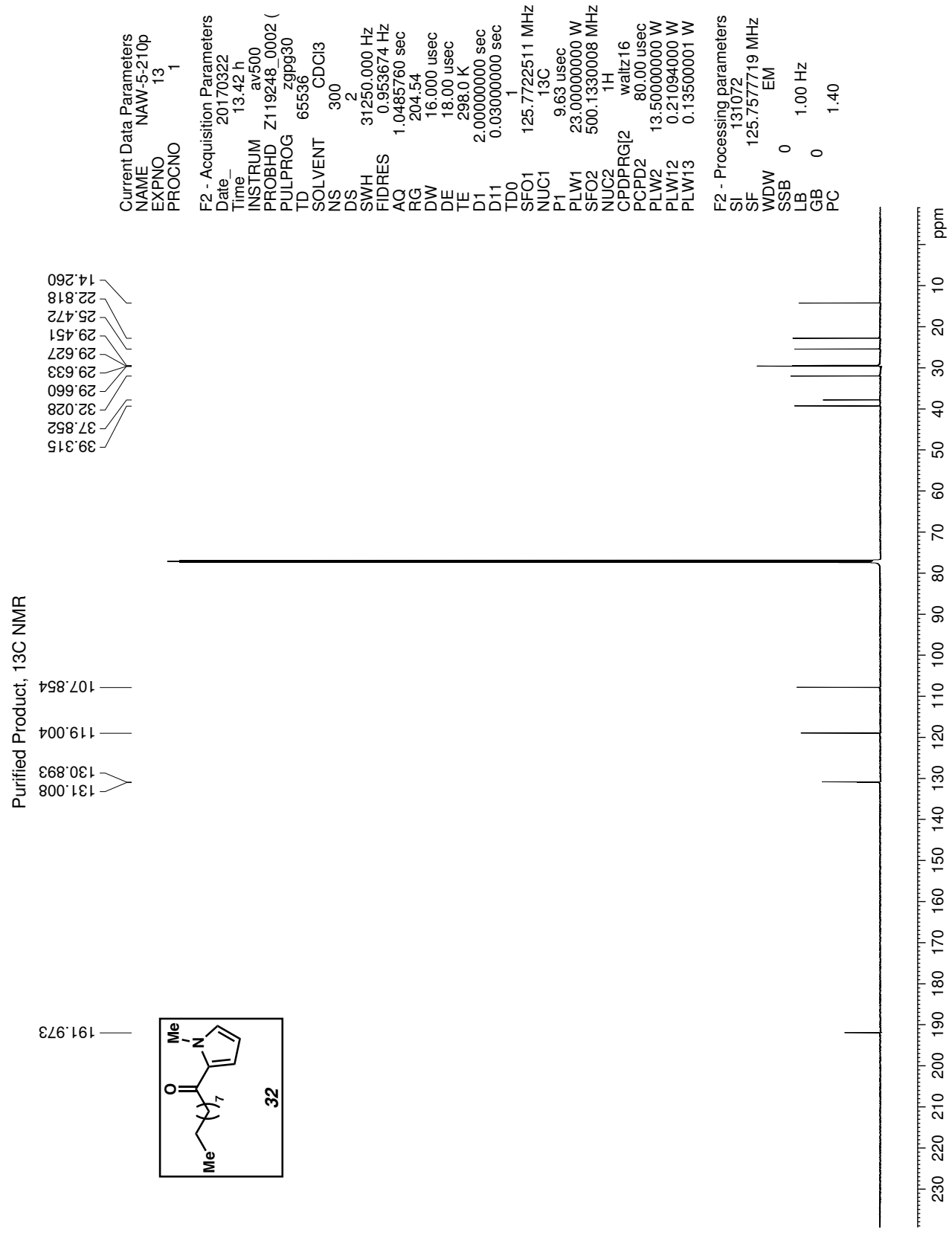

Purified Product, 13C NMR
Current Data Parameters
NAME KJY-04-179P
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time 14.55 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577761 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
195.958
140.404
133.833
126.140
125.860
122.988
120.945
111.126
110.556
67.508
44.705
32.407
29.784
Me
N
O
O
22
ppm

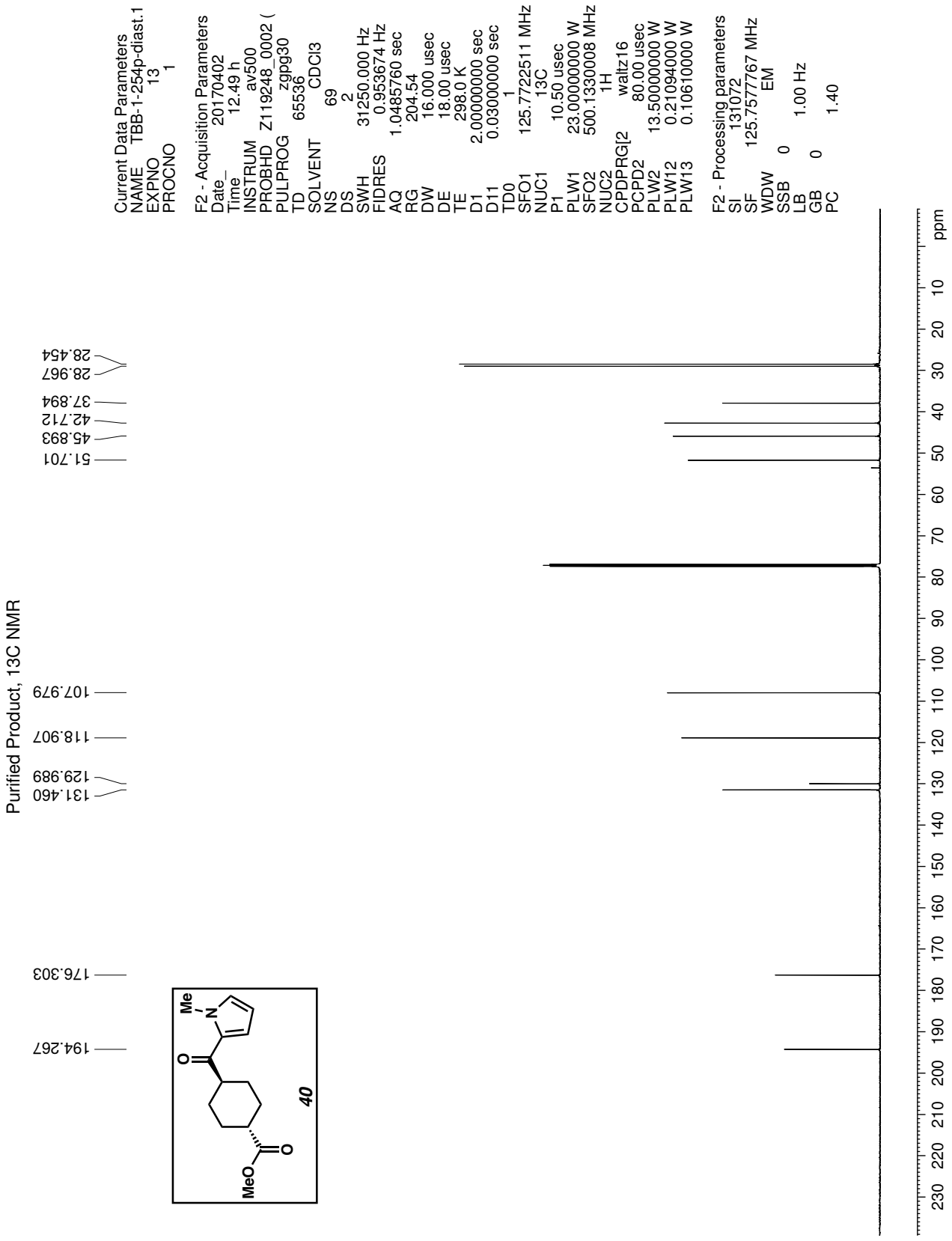

Purified Product, 13C NMR
Current Data Parameters
NAME KJY-04-173P
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time 15.01 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577796 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
195.222
140.437
132.776
123.345
69.813
68.221
47.220
39.466
26.557
25.206
N
N
Me
O
O
23
ppm

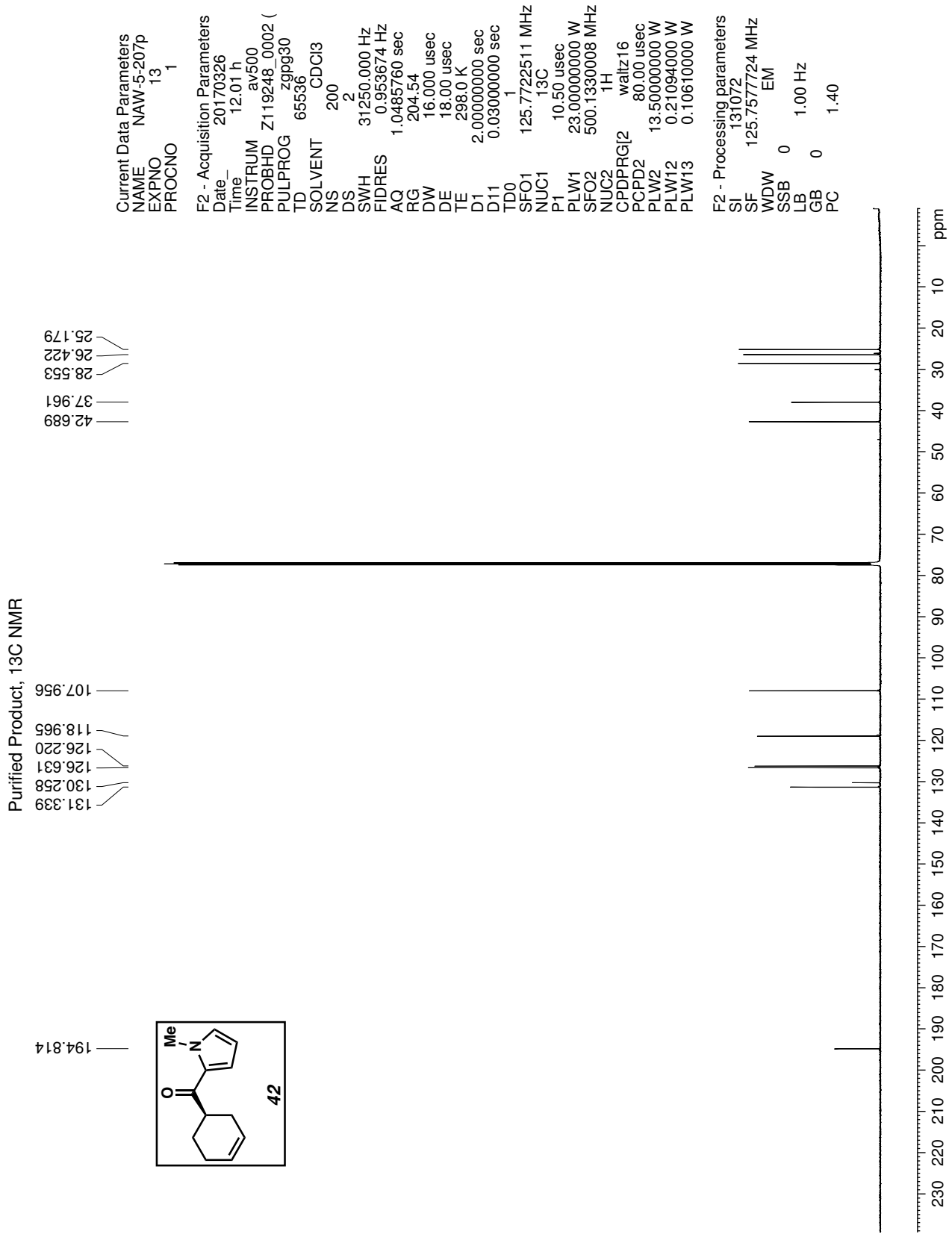

Current Data Parameters
NAME KJY-04-197P
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170313
Time 14.29 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577772 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
191.77
131.73
130.11
119.47
108.22
70.61
68.26
45.74
37.88
27.10
25.37
Me
N
O
O
24
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

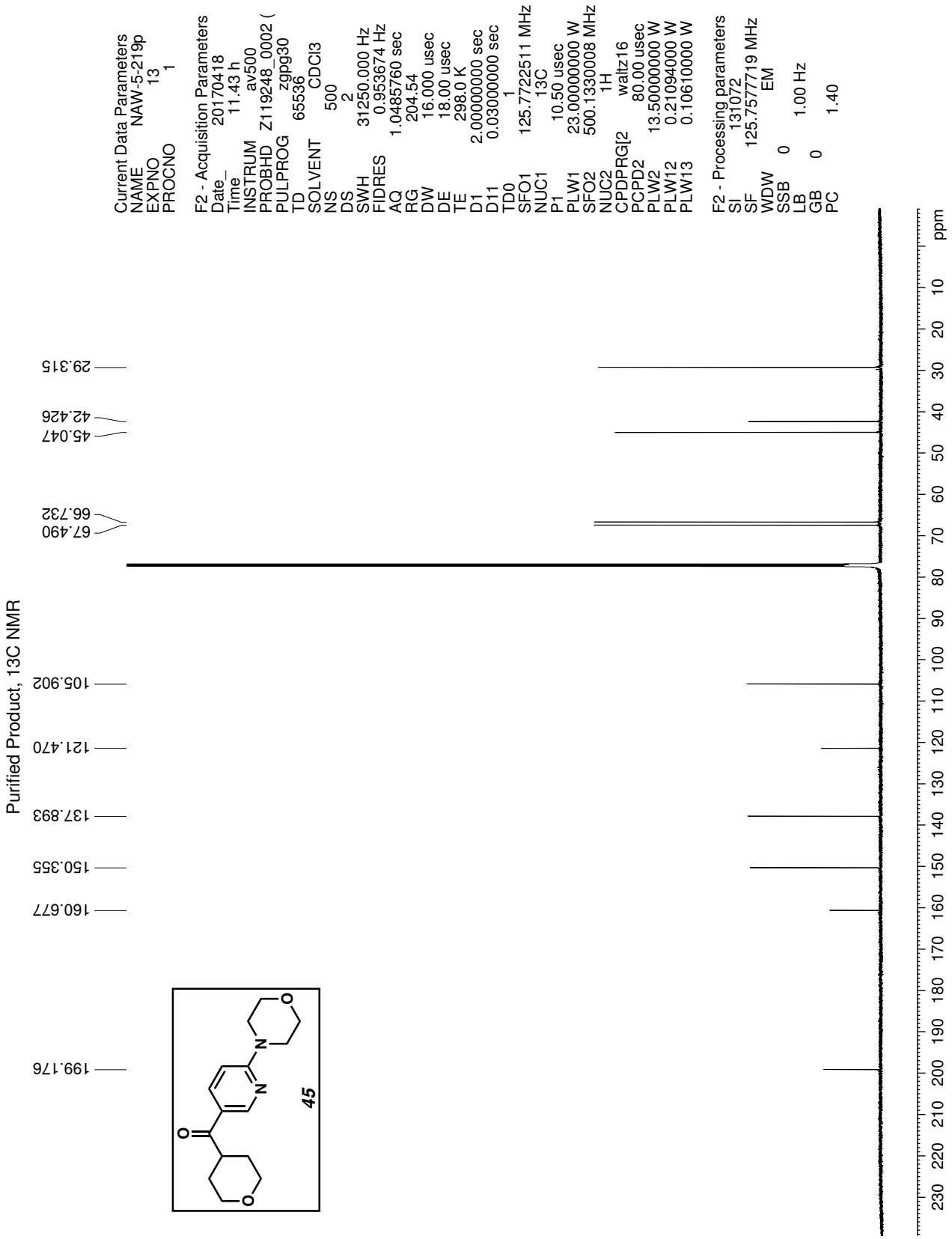

Current Data Parameters
NAME KJY-04-255P
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170615
Time 13.12 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 40
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 13.13
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
29.41
42.55
67.46
102.02
108.10
108.33
124.45
130.67
148.50
151.89
199.98
25
ppm

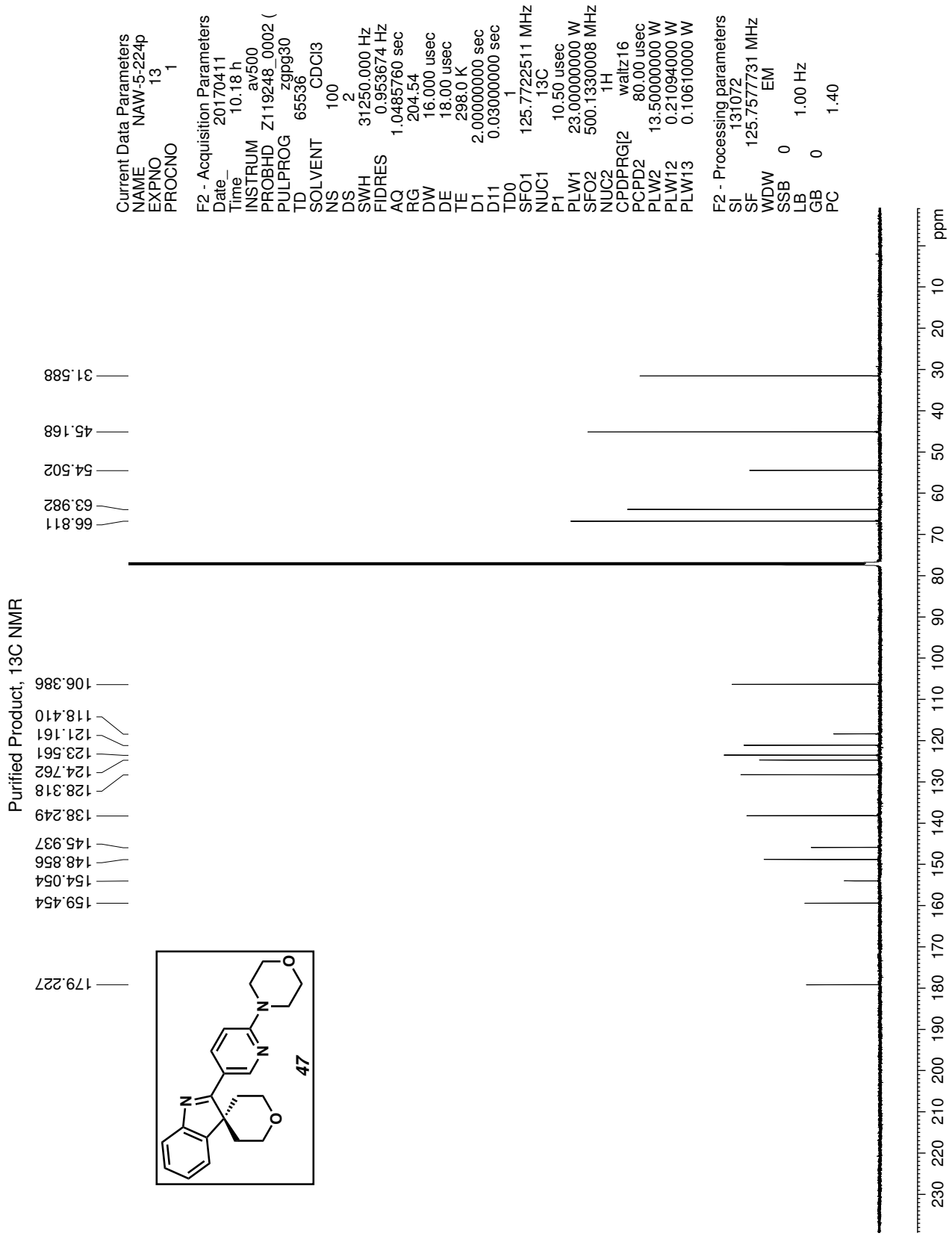

Purified Product, 13C NMR
Current Data Parameters
NAME KJY-4-Naphth
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170326
Time 20.21 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577738 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
202.202
154.876
135.715
133.308
132.699
129.807
129.669
128.815
128.675
127.925
126.997
124.281
79.776
43.686
43.251
28.678
28.592
O
N
Boc
26
ppm


Purified Product, 13C NMR
Current Data Parameters
NAME NAW-5-210p
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170322
Time 13.42 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 9.63 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.13500001 W
F2 - Processing parameters
SI 131072
SF 125.7577719 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
191.973
131.008
130.893
119.004
107.854
39.315
37.852
32.028
29.660
29.633
29.627
29.451
25.472
22.818
14.260
Me
N
O
Me
7
32
ppm


Purified Product, 13C NMR
Current Data Parameters
NAME TBB-1-254p-diast.1
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170402
Time 12.49 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 69
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577767 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
194.267
176.303
131.460
129.989
118.907
107.979
51.701
45.893
42.712
37.894
28.967
28.454
Me
N
O
MeO
O
O
40
ppm


Purified Product, 13C NMR
Current Data Parameters
NAME NAW-5-207p
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170326
Time 12.01 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577724 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
194.814
131.339
130.258
126.631
126.220
118.965
107.956
42.689
37.961
28.553
26.422
25.179
Me
N
O
42
ppm


Purified Product, 13C NMR
Current Data Parameters
NAME NAW-5-219p
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170418
Time 11.43 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 500
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577719 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
29.315
42.426
45.047
66.732
67.490
105.902
121.470
137.893
150.355
160.677
199.176
45
ppm


Purified Product, 13C NMR
179.227
159.454
154.054
148.856
145.937
138.249
128.318
124.762
123.561
121.161
118.410
106.386
66.811
63.982
54.502
45.168
31.588
47
Current Data Parameters
NAME NAW-5-224p
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170411
Time 10.18 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577731 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm