Nickel-Catalyzed Reduction of Secondary and Tertiary Amides

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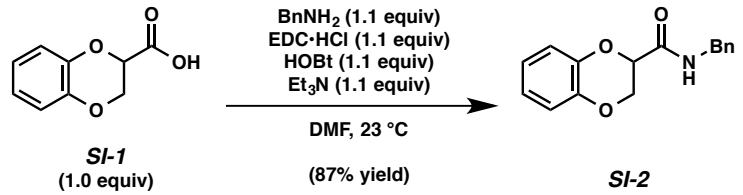
Supporting Information – Table of Contents

Materials and Methods. Unless stated otherwise, reactions were conducted in flame-dried glassware under an atmosphere of nitrogen and commercially obtained reagents were used as received. Non-commercially available substrates were synthesized following protocols specified in Section A of the Experimental Procedures. Prior to use, toluene was purified by distillation and taken through five freeze-pump-thaw cycles. $NiCl_2$(dme) and $LiAlD_4$ was obtained from Strem Chemicals. Hexamethylbenzene and $PhSiH_3$ were obtained from Alfa Aesar and used as received. Carboxylic acids **SI-1**, **SI-3**, **SI-8**, amines **SI-6**, **SI-12**, **rac-SI-6**, **rac-SI-45**, **rac-SI-46**, and lactam **SI-37** were obtained from Combi-Blocks. Carboxylic acid **SI-5** was obtained from Eastman Organic. Lactams **SI-38**, **SI-39**, and $PhSiCl_3$ were obtained from Sigma–Aldrich and used as received. Benzylamine was obtained from Acros Organics and used as received. Reaction temperatures were controlled using an IKAmag temperature modulator, and unless stated otherwise, reactions were performed at elevated temperatures (approximately 115 °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 ^{2}H NMR spectra are reported in terms of chemical shift (at 76 MHz). 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}). High-resolution mass spectra were obtained on Thermo Scientific™ Exactive Mass Spectrometer with DART ID-CUBE. Determination of enantiopurity was carried out on a Mettler Toledo SFC (supercritical fluid chromatography) using a Daicel ChiralPak column.

Experimental Procedures

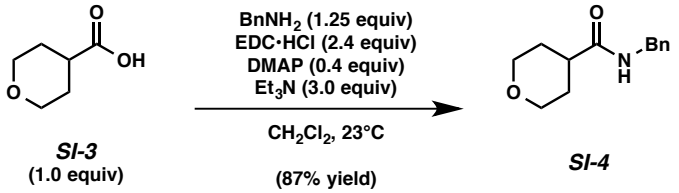
A. Syntheses of Amide Substrates

Representative Procedure A for the synthesis of amide substrates from Figure 2. (SI-2 is used as an example).



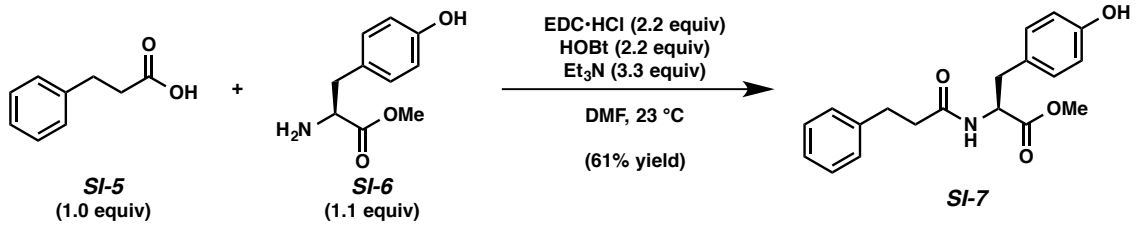
Amide SI-2. To a solution of carboxylic acid **SI-1** (2.00 g, 11.1 mmol, 1.0 equiv), HOBt (1.87 g, 12.2 mmol, 1.1 equiv), and triethylamine (1.70 mL, 12.2 mmol, 1.1 equiv) in DMF (111.0 mL, 0.1 M) at 23 °C was added EDC•HCl (1.89 g, 12.2 mmol, 1.1 equiv) followed by benzylamine (1.3 mL, 12.2 mmol, 1.1 equiv) dropwise over 1 min under a N_2 atmosphere. After stirring at 23 °C for 18 h, the reaction was diluted with deionized water (100 mL) and transferred to a separatory funnel with EtOAc (75 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 x 75 mL). The organic layers were combined and washed with water (3 x 75 mL), dried over Na_2SO_4, and the volatiles were removed under reduced pressure. The resulting crude residue was purified by flash chromatography (10:1 Hexanes:EtOAc → 2:1 Hexanes:EtOAc) to yield amide **SI-2** (2.6 g, 87% yield) as a white solid. Amide **SI-2**: mp: 130–131 ºC; R_f 0.30 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35–7.27 (m, 3H), 7.25–7.23 (m, 2H), 6.94–6.85 (m, 5H), 4.75 (dd, J = 7.3, 2.7, 1H), 4.58 (dd, J = 11.4, 2.7, 1H), 4.52 (d, J = 6.0, 2H), 4.23 (dd, J = 11.4, 7.3, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.3, 143.5, 141.7, 137.6, 128.9, 127.9, 127.8, 122.6, 122.1, 117.8, 117.3, 73.4, 65.6, 43.3; IR (film): 3029, 2924, 2876, 1670, 1493 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{16}H_{16}NO_3$, 270.11247; found 270.11082.

Representative Procedure B for the synthesis of amide substrates from Figure 2. (SI-4 is used as an example).



Amide SI-4. To a solution of carboxylic acid **SI-3** (500.0 mg, 3.84 mmol, 1.0 equiv), EDC•HCl (1.77 g, 9.22 mmol, 2.4 equiv), and DMAP (188.0 mg, 1.54 mmol, 0.4 equiv) in CH_2Cl_2 (12 mL, 0.35 M) was added Et_3N (1.6 mL, 11.52 mmol, 3.0 equiv) followed by benzylamine **(**525.0 µL, 4.80 mmol,1.25 equiv) dropwise over 1 min under a N_2 atmosphere. After stirring at 23 °C for 18 h, the reaction mixture was transferred to a separatory funnel with EtOAc (30 mL) and washed with 1.0 M aqueous HCl (2 x 10 mL), followed by 1.0 M aqueous NaOH (2 x 10 mL), and deionized water (10 mL). The organic layer was dried over Na_2SO_4, and the volatiles were removed under reduced pressure. The resulting crude residue was purified by flash chromatography (1:15 Hexanes:EtOAc) to yield amide **SI-4** (775.5 mg, 87% yield) as a white solid. Amide **SI-4**: mp: 116–117 ºC; R_f 0.46 (1:4 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35–7.33 (m, 2H), 7.30–7.27 (m, 3H), 5.71 (br. s, 1H), 4.46 (d, *J* = 5.7, 2H), 4.04–4.00 (m, 2H), 3.41 (td, *J* = 11.5, 2.8, 2H), 2.40–2.34 (m, 1H), 1.88–1.82 (m, 2H), 1.81–1.77 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 174.1, 138.3, 128.9, 127.9, 127.8, 67.4, 43.7, 42.4, 29.4; IR (film): 3064, 2951, 2842, 1635, 1541 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{13}H_{18}NO_2$, 220.13321; found 220.13214.

Representative Procedure C for the synthesis of amide substrates from Figure 5. (SI-7 is used as an example).

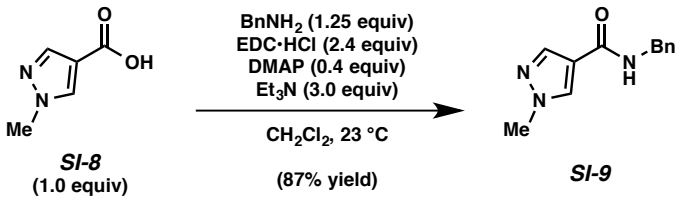


Amide SI-7. To a solution of carboxylic acid **SI-5** (500.0 mg, 3.33 mmol, 1.0 equiv), HOBt (988.9 mg, 7.33 mmol, 2.2 equiv), and EDC•HCl (1.41 g, 7.33 mmol, 2.2 equiv) in DMF (16.6 mL, 0.2 M) at 23 °C was added amine **SI-6** (714.9 mg, 3.66 mmol, 1.1 equiv), followed by

triethylamine (1.50 mL, 10.9 mmol, 3.3 equiv) under a N_2 atmosphere. After stirring at 23 °C for 18 h, the reaction was diluted with deionized water (50 mL) and transferred to a separatory funnel with EtOAc (40 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 x 40 mL). The organic layers were combined and washed with water (3 x 40 mL), dried over Na_2SO_4, and the volatiles were removed under reduced pressure. The resulting crude residue was purified by flash chromatography (3:1 Hexanes:EtOAc → 1:1 Hexanes:EtOAc) to yield amide **SI-7** (662.1 mg, 61% yield) as a white solid. Amide **SI-7**: mp: 89–92 ºC; R_f 0.25 (1:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, CD_2Cl_2): δ 7.29–7.26 (m, 2H), 7.20–7.17 (m, 3H), 6.83–6.82 (m, 2H), 6.71–6.68 (m, 2H), 5.94 (br. d, *J* = 7.8, 1H), 4.79 (dt, *J* = 7.8, 5.9, 1H), 3.69 (s, 3H), 2.99 (dd, *J* = 13.8, 5.6, 1H), 2.93 (dd, *J* = 13.8, 5.6, 1H), 2.91–2.88 (m, 2H), 2.53–2.42 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.1, 171.7, 154.9, 140.8, 130.4, 128.6, 128.4, 127.5, 126.3, 115.5, 53.1, 52.4, 38.2, 37.1, 31.4; IR (film): 3287, 3026, 2951, 1736, 1647 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{19}H_{20}NO_4$, 326.13937, found 326.13868. $[\alpha]^{27.6}_D$ +60.00° (c = 0.10, CH_2Cl_2).

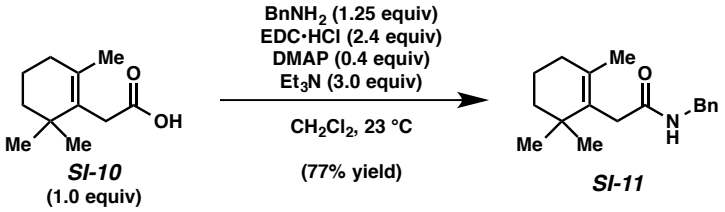
Note: Supplementary information for the syntheses of some amides used in Figures 2, 3, 4, and 5 have previously been reported: **7**,[1] **SI-14**,[2] **SI-15**,[3] **SI-16**,[4] **SI-17**,[5] **SI-18**,[6] **SI-19**,[7] **SI-20**,[2] **SI-21**,[3] **SI-22**,[8] **SI-23**,[1] **SI-24**,[9] **SI-25**,[10] **SI-26**,[10] **SI-27**,[11] **SI-28**,[12] **SI-29**,[13] **SI-30**,[14] **SI-31**,[15] **SI-32**,[16] **SI-33**,[17] **SI-34**,[18] **SI-35**,[19] **SI-36**,[20] **SI-42**,[21] **SI-43**,[22] **SI-44**.[23] Syntheses for the remaining substrates shown in Figures 3 and 6 are as follows:

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

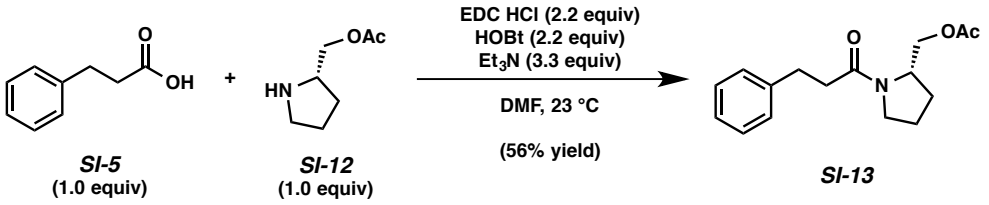


Amide SI-9. Following representative procedure B. Purification by flash chromatography (9:1 EtOAc:$CHCl_3$) yielded amide **SI-9** (810.2 mg, 87% yield) as a white solid. Amide **SI-9**: mp: 157–158 °C; R_f 0.27 (1:4 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.83 (s, 1H), 7.70 (s, 1H), 7.36–7.32 (m, 4H), 7.30–7.27 (m, 1H), 6.06 (s, 1H), 4.59 (d, *J* = 5.6, 2H), 3.91 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 162.4, 138.4, 137.8, 131.9, 128.9, 128.0, 127.7, 118.8, 43.6, 39.4; IR

(film): 3111, 3064, 2941, 1630, 1566 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{12}H_{14}N_3O$, 216.11314; found 216.11201.



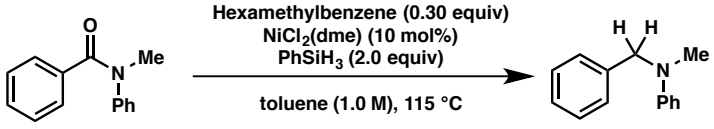
Amide SI-11. Following representative procedure B. Purification by flash chromatography (4:1 Hexanes:EtOAc) yielded amide **SI-11** (230.0 mg, 77% yield) as a white solid. Amide **SI-11**: mp: 78–80 °C; R_f 0.32 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, C_6D_6): δ 7.13–7.09 (m, 4H), 7.05–7.02 (m, 1H), 5.62 (br. s, 1H), 4.33 (d, *J* = 5.9, 2H), 2.99 (s, 2H), 1.69 (t, *J* = 6.4, 2H), 1.42 (s, 3H), 1.41–1.38 (m, 2H), 1.31–1.28 (m, 2H), 0.90 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 171.3, 138.4, 132.9, 132.5, 128.7, 127.5, 127.4, 43.5, 39.4, 36.7, 34.9, 32.6, 28.1, 20.5, 19.1; IR (film): 3299, 2926, 2865, 1640, 1516 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{18}H_{26}NO$, 272.19901; found 272.20089.



Amide SI-13. Following representative procedure C. Purification by flash chromatography (1:1 Hexanes:EtOAc) yielded amide **SI-13** (234.0 mg, 56% yield) as a colorless oil. Amide **SI-13** was observed as a 2.9:1 mixture of rotational isomers in $CDCl_3$. Amide **SI-13**: R_f 0.29 (1:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$, **Major rotational isomer**): δ 7.26–7.23 (m, 2H), 7.20–7.14 (m, 3H), 4.34–4.31 (m, 1H), 4.14 (dd, *J* = 10.7, 3.8, 1H), 4.06 (dd, *J* = 10.7, 6.9, 1H), 3.35–3.31 (m, 1H), 3.28–3.23 (m, 1H), 2.99–2.92 (m, 2H), 2.53 (t, *J* = 7.7, 2H), 2.01 (s, 3H), 1.95–1.77 (m, 4H); ^{1}H NMR (500 MHz, $CDCl_3$, **Minor rotational isomer**): δ 7.26–7.26 (m, 2H), 7.20–7.14 (m, 3H), 3.94 (dd, *J* = 10.7, 5.5, 1H), 3.91–3.86 (m, 1H), 3.76 (dd, *J* = 10.7, 7.4, 1H), 3.51 (dt, *J* = 12.3, 8.5, 1H), 3.43–3.38 (m, 1H), 2.99–2.92 (m, 2H), 2.66 (t, *J* = 7.7, 2H), 1.99 (s, 3H), 1.95–1.77 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$, **Major rotational isomer**): δ 171.4, 170.9, 141.5, 128.6, 128.6, 126.2, 64.0, 55.5, 47.2, 36.9, 31.2, 27.4, 24.1, 21.0; ^{13}C NMR

(100 MHz, $CDCl_3$, **Minor rotational isomer**): δ 171.6, 170.7, 141.4, 128.6, 128.6, 126.2, 64.7, 55.8, 45.7, 36.2, 31.8, 28.7, 21.8, 20.9; IR (film): 3474, 2955, 2878, 1737, 1636 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{16}H_{22}NO_3$, 276.15906; found 276.15942. $[\alpha]^{29.6}_D$ –29.33° (c = 0.10, CH_2Cl_2).

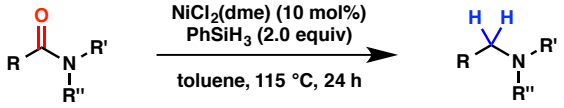
B. Initial Survey of Amide Substrates with Phenylsilane

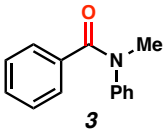
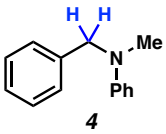
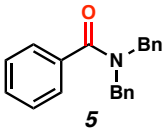
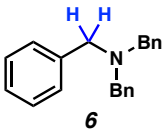
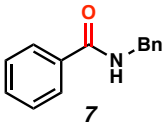
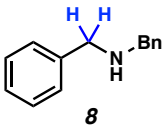
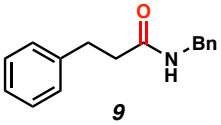
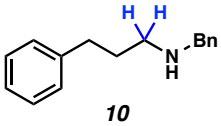


3 ***4***

Representative Procedure for the reduction of amides from Figure 2 and Figure SI-1 (reduction of amide 3 is used as an example). A 1-dram vial was charged with a magnetic stir bar, flame-dried under reduced pressure, and allowed to cool under a N_2 atmosphere. Amide substrate **3** (42.3 mg, 0.200 mmol, 1.0 equiv), $NiCl_2$(dme) (4.4 mg, 0.0200 mmol, 10 mol%), and hexamethylbenzene (9.7 mg, 0.0600 mmol, 0.30 equiv) were added, and the vial was flushed with N_2 for 5 min. $PhSiH_3$ (49.4 μL, 0.400 mmol, 2.0 equiv) was added under a N_2 atmosphere via syringe followed by toluene (200 μL, 1.0 M). The vial was then capped with a Teflon-lined screw cap under a flow of N_2. The reaction mixture was then placed in a pre-heated aluminum block and allowed to stir at 115 °C for 24 h. After cooling to room temperature, the reaction mixture was transferred to a separatory funnel with EtOAc (3 mL) and basified with 1.0 M aqueous NaOH (4 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 x 5 mL). The combined organic layers were washed with saturated aqueous NaCl (5 mL). The volatiles were removed under reduced pressure, and the yield was determined by ^1H NMR analysis with hexamethylbenzene as an internal standard.

Figure SI-1. Initial Survey of Amide Substrates with Phenylsilane[a]

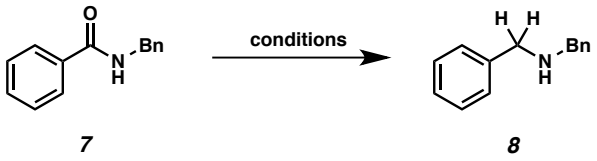


Entry	*Amide*	*Amine*	*Yield*[b]
1	 *3*	 *4*	*86%*
2	 *5*	 *6*	*92%*
3	 *7*	 *8*	*95%*
4	 *9*	 *10*	*93%*

[a] Conditions unless otherwise stated: $NiCl_2$(dme) (10 mol%), substrate (1.0 equiv, 0.2 mmol), $PhSiH_3$ (2.0 equiv), and toluene (1.0 M) at 115 °C for 24 h in a sealed vial. [b] Yields determined by ^1H NMR analysis using hexamethylbenzene as an internal standard.

C. Relevant Control Experiments for the Reduction of Amide 7

Figure SI-2. Relevant Control Experiments in the Reduction of Amide **7**[a]

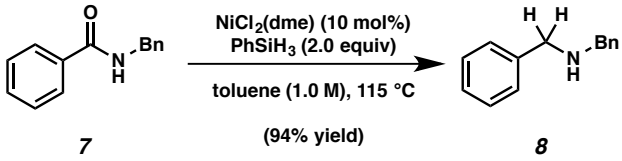


7 *8*

Reaction Conditions	*Experimental Results* *7*	*8*
$NiCl_2$(dme) (10 mol%), $PhSiH_3$ (2.0 equiv), toluene (1.0 M), 115 °C, 24 h	0%	95%
Control Experiments:		
$PhSiH_3$ (2.0 equiv), toluene (1.0 M), 115 °C, 24 h	98%	0%
$NiCl_2$(dme) (10 mol%), toluene (1.0 M), 115 °C, 24 h	100%	0%

[a] Yields were determined by ^1H NMR analysis using hexamethylbenzene as an internal standard.

D. Scope of Methodology

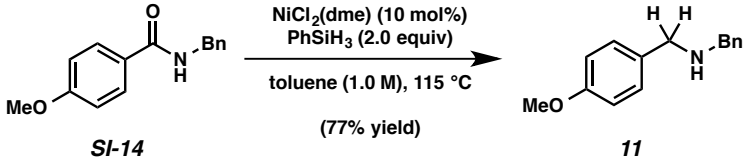


7 *8*

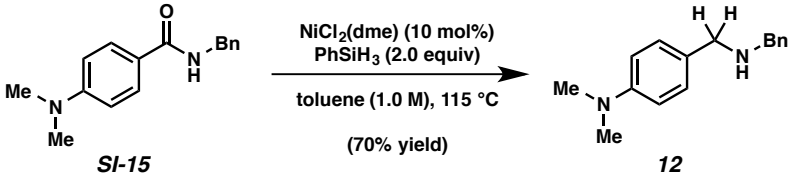
Representative Procedure (reduction of amide 7 is used as an example). Amine 8. A 1-dram vial was charged with a magnetic stir bar, flame-dried under reduced pressure, and allowed to cool under a N_2 atmosphere. Amide substrate **7** (42.3 mg, 0.200 mmol, 1.0 equiv) and $NiCl_2$(dme) (4.4 mg, 0.0200 mmol, 10 mol%) was added, and the vial was flushed with N_2. $PhSiH_3$ (49.4 µL, 0.4000 mmol, 2.0 equiv) was added under a N_2 atmosphere via syringe followed by toluene (200 µL, 1.0 M). The vial was then capped with a Teflon-lined screw cap under a flow of N_2. The reaction mixture was then placed in a pre-heated aluminum block and allowed to stir at 115 °C for 24 h. After cooling to room temperature, the reaction mixture was transferred to a separatory funnel with EtOAc (3 mL) and basified with 1.0 M aqueous NaOH (4 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 x 5 mL). The combined organic layers were washed with saturated aqueous NaCl (5 mL). The volatiles were removed under reduced pressure, and the crude residue was purified by flash chromatography

(1:1 Hexanes:EtOAc) to yield amine **8** (94% yield, average of two experiments) as a colorless oil. Amine **8**: R_f 0.36 (1:1 Hexanes:EtOAc). Spectral data match those previously reported.[24]

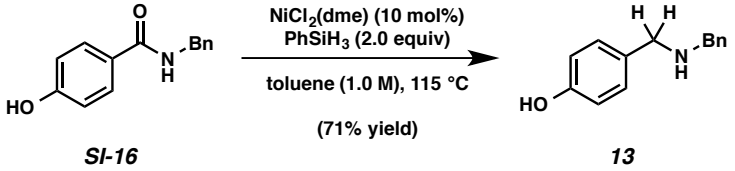
Any modifications of the conditions shown in the representative procedure above are specified in the following schemes, which depict all of the results shown in Figures 2, 3, and 4.



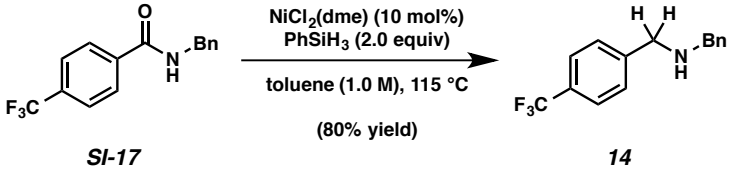
Amine 11. Purification by flash chromatography (3:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **11** (77% yield, average of two experiments) as a colorless oil. Amine **11**: R_f 0.41 (4:1 Hexanes:EtOAc, 2% Et_3N). Spectral data match those previously reported.[25]



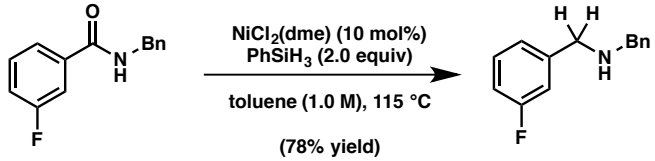
Amine 12. Purification by flash chromatography (1:1 CH_2Cl_2:CH_3CN, 2% Et_3N) yielded amine **12** (70% yield, average of two experiments) as a colorless oil. Amine **12**: R_f 0.50 (1:1 CH_2Cl_2:CH_3CN, 2% Et_3N). Spectral data match those previously reported.[26]



Amine 13. Purification by flash chromatography (EtOAc, 2% Et_3N) yielded amine **13** (71% yield, average of two experiments) as a white solid. Amine **13**: R_f 0.52 (EtOAc, 2% Et_3N). Spectral data match those previously reported.[27]

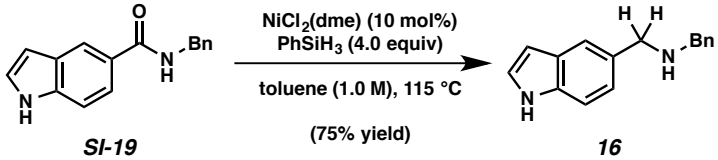


Amine 14. Purification by flash chromatography (1:1 Hexanes:EtOAc) yielded amine **14** (80% yield, average of two experiments) as a colorless oil. Amine **14**: R_f 0.60 (1:1 Hexanes:EtOAc). Spectral data match those previously reported.[28]



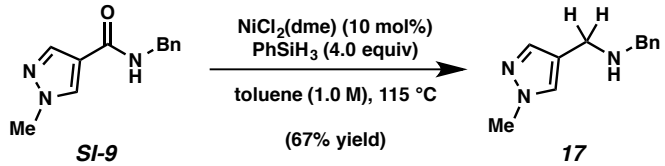
SI-18 ***15***

Amine 15. Purification by flash chromatography (1:1 Hexanes:EtOAc) yielded amine **15** (78% yield, average of two experiments) as a colorless oil. Amine **15**: R_f 0.59 (1:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35 (d, J = 4.4, 4H), 7.31–7.26 (m, 2H), 7.12–7.09 (m, 2H), 6.95 (td, J = 8.6, 2.3, 1H), 3.81 (s, 4H), 1.77 (br. s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 164.0, 162.1, 143.0 (d, $J_{C–F}$ = 6.9), 140.1, 129.8 (d, $J_{C–F}$ = 8.1), 128.3 (d, $J_{C–F}$ = 37.6), 127.1, 123.6 (d, $J_{C–F}$ = 2.9), 114.9 ($J_{C–F}$ = 21.1), 113.8 ($J_{C–F}$ = 21.1), 53.1, 52.6; IR (film): 3064, 3028, 2831, 1615, 1588 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{14}H_{15}FN$, 216.11830; found 216.11803.



SI-19 ***16***

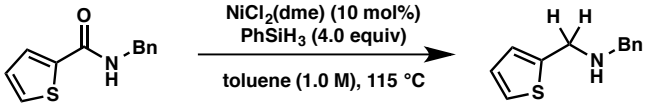
Amine 16. Purification by flash chromatography (4:1 Hexanes:EtOAc, 2% Et_3N → 1:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **16** (75% yield, average of two experiments) as a colorless solid. Amine **16**: mp: 51–54 °C; R_f 0.36 (1:1 Hexanes:EtOAc, 2% Et_3N). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.34 (br. s, 1H), 7.60 (s, 1H), 7.39–7.33 (m, 5H), 7.23–7.26 (m, 1H), 7.21–7.18 (m, 2H), 6.53 (d, J = 3.1, 1H), 3.93 (s, 2H), 3.85 (s, 2H), 2.44 (br. s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 140.0, 135.3, 131.1, 128.5, 128.4, 128.1, 127.1, 124.7, 122.9, 120.5, 111.2, 102.5, 53.5, 52.9; IR (film): 3410, 3026, 2919, 1623, 1452 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{16}H_{17}N$, 237.13863; found 237.13806.



SI-9 ***17***

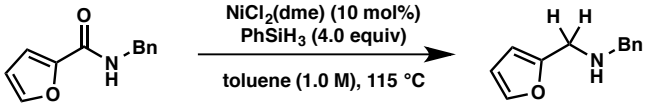
Amine 17. Purification by flash chromatography (EtOAc, 2% Et_3N) yielded amine **17** (67% yield, average of two experiments) as a colorless oil. Amine **17**: R_f 0.26 (EtOAc, 2% Et_3N). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.43 (s, 1H), 7.35–7.32 (m, 4H), 7.31–7.30 (m, 1H), 7.28–7.24 (m, 2H), 3.87 (s, 3H), 3.81 (s, 2H), 3.69 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 140.3, 138.9, 129.1,

128.6, 128.3, 127.1, 120.6, 53.3, 43.3, 39.0; IR (film): 3303, 2936, 2849, 1642, 1453 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{12}H_{16}N_3$, 202.13387; found 202.13355.



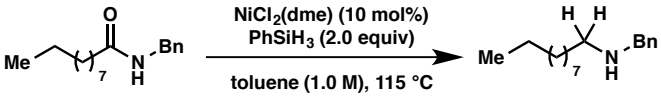
SI-20 ***18***

Amine 18. Purification by flash chromatography (10:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **18** (62% yield, average of two experiments) as a colorless oil. Amine **18**: R_f 0.34 (4:1 Hexanes:EtOAc, 2% Et_3N). Spectral data match those previously reported.[26]



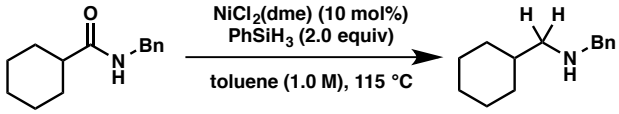
SI-21 ***19***

Amine 19. Purification by flash chromatography (4:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **19** (96% yield, average of two experiments) as a colorless oil. Amine **19**: R_f 0.49 (4:1 Hexanes:EtOAc, 2% Et_3N). Spectral data match those previously reported.[25]



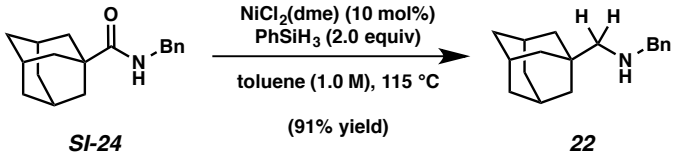
SI-22 ***20***

Amine 20. Purification by flash chromatography (10:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **20** (75% yield, average of two experiments) as a colorless oil. Amine **20**: R_f 0.38 (10:1 Hexanes:EtOAc, 2% Et_3N). Spectral data match those previously reported.[29]



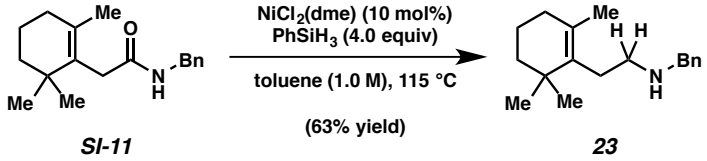
SI-23 ***21***

Amine 21. Purification by flash chromatography (10:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **21** (81% yield, average of two experiments) as a colorless oil. Amine **21**: R_f 0.40 (10:1 Hexanes:EtOAc, 2% Et_3N). Spectral data match those previously reported.[30]



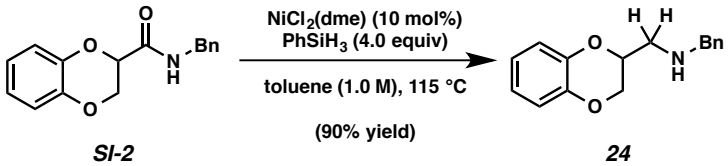
SI-24 ***22***

Amine 22. Purification by flash chromatography (4:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **22** (91% yield, average of two experiments) as a colorless oil. Amine **22**: R_f 0.52 (4:1 Hexanes:EtOAc, 2% Et_3N). Spectral data match those previously reported.[31]



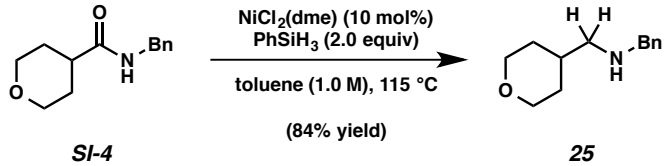
SI-11 ***23***

Amine 23. Purification by preparative thin-layer chromatography (10:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **23** (63% yield, average of two experiments) as a colorless oil. Amine **23**: R_f 0.40 (10:1 Hexanes:EtOAc, 2% Et_3N). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32–7.30 (m, 4H), 7.27–7.21 (m, 1H), 3.82 (s, 2H), 2.67–2.63 (m, 2H), 2.23 (t, J = 8.5, 2H), 1.89 (t, J = 6.4, 2H), 1.59 (s, 3H), 1.57–1.53 (m, 2H), 1.41–1.39 (m, 2H), 0.97 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 140.7, 134.9, 128.5, 128.4, 128.1, 127.0, 54.1, 49.7, 39.9, 34.9, 32.9, 29.8, 28.9, 20.1, 19.6; IR (film): 3026, 2926, 2864, 1873, 1452 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{18}H_{28}N$, 258.22074; found 258.22163.

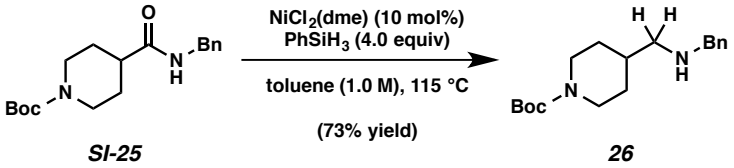


SI-2 ***24***

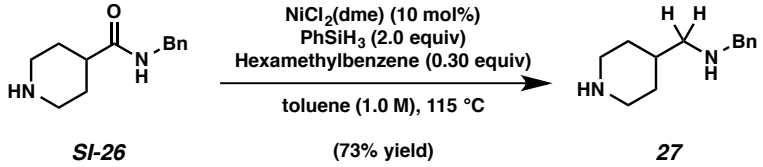
Amine 24. Purification by flash chromatography (10:1 Hexanes:EtOAc, 2% Et_3N → 4:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **24** (90% yield, average of two experiments) as a colorless oil. Amine **24**: R_f 0.38 (4:1 Hexanes:EtOAc, 2% Et_3N). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36–7.35 (m, 4H), 7.30–7.27 (m, 1H), 6.90–6.84 (m, 4H), 4.33–4.30 (m, 1H), 4.28 (dd, J = 11.0, 2.4, 1H), 4.05 (dd, J = 11.0, 7.3, 1H), 3.87 (s, 2H), 2.94 (dd, J = 12.4, 6.6, 1H), 2.88 (dd, J = 12.4, 4.6, 1H), 2.00 (br. s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 143.4, 143.3, 139.9, 128.6, 128.2, 127.2, 121.6, 121.5, 117.4, 117.2, 72.7, 66.7, 54.0, 49.3; IR (film): 3027, 2919, 2831, 1592, 1492 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{16}H_{18}NO_2$, 256.13321; found 256.13258.



Amine 25. Purification by flash chromatography (1:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **25** (84% yield, average of two experiments) as a colorless oil. Amine **25**: R_f 0.54 (1:1 Hexanes:EtOAc, 2% Et_3N). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.43–7.31 (m, 4H), 7.27–7.24 (m, 1H), 3.97–3.94 (m, 2H), 3.78 (s, 2H), 3.37 (ddd, *J* = 11.8, 11.8, 1.9, 2H), 2.52 (d, *J* = 6.6, 2H), 1.79 (br. s, 1H), 1.72 (ttt, *J* = 11.8, 6.9, 3.8, 1H), 1.65 (m, 2H), 1.29 (dddd, *J* = 11.8, 11.8, 11.8, 4.4, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 140.6, 128.5, 128.1, 127.0, 67.9, 55.6, 54.2, 35.6, 31.4; IR (film): 3026, 2916, 2838, 1603, 1453 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{13}H_{20}NO$, 206.15394; found 206.15352.

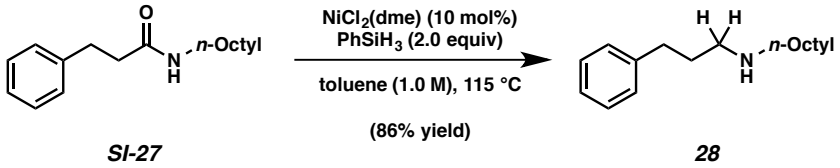


Amine 26. Purification by flash chromatography (4:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **26** (73% yield, average of two experiments) as an amorphous solid. Amine **26**: R_f 0.52 (4:1 Hexanes:EtOAc, 2% Et_3N). ^{1}H NMR (500 MHz, $CDCl_3$, 60 °C): δ 7.32–7.27 (m, 4H), 7.22–7.18 (m, 1H), 4.03 (d, *J* = 12.7, 2H), 3.75 (s, 2H), 2.67 (t, *J* = 12.5, 2H), 2.49 (br. s, 2H) 1.67 (d, *J* = 12.4, 2H), 1.61–1.55 (m, 2H) 1.42 (s, 9H), 1.13–1.06 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$, 60 °C): δ 154.7, 140.5, 128.2, 127.8, 126.7, 78.9, 54.9, 54.0, 43.8, 36.6, 30.3, 28.3; IR (film): 2975, 2921, 2849, 1685, 1452 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{18}H_{29}N_2O_2$, 305.22235; found 305.22153.



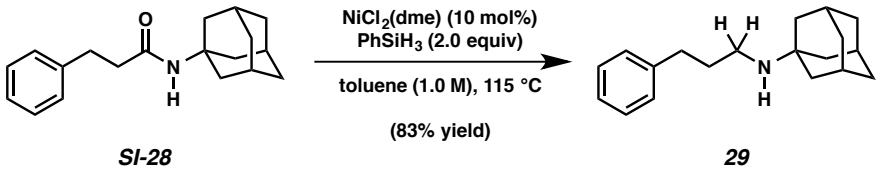
Amine 27. ^{1}H NMR analysis of the crude reaction mixture indicated a 73% yield (average of two experiments) of amine **27** relative to hexamethylbenzene internal standard. Amine **27**: R_f 0.11 (CH_2Cl_2, 2% MeOH). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33–7.29 (m, 4H), 7.25–7.22 (m, 1H), 3.77 (s, 2H), 3.05 (ddd, *J* = 12.1, 2.7, 2.7, 2H), 2.58 (ddd, *J* = 12.1, 12.1, 2.4, 2H), 2.49 (d, *J* =

6.7, 2H), 1.77 (br. s, 1H), 1.72–1.69 (m, 2H), 1.59 (ttt, J = 12.1, 6.8, 2.5, 1H), 1.11 (dddd, J = 12.1, 12.1, 12.1, 3.9, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 140.6, 128.5, 128.2, 126.9, 55.9, 54.3, 46.6, 36.8, 31.9; IR (film): 3308, 3026, 2921, 1633, 1542 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{13}H_{21}N_2$, 205.16993; found 205.16948.



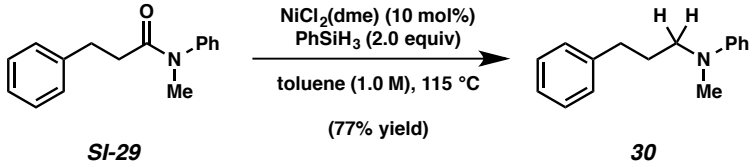
SI-27 ***28***

Amine 28. Purification by preparative thin-layer chromatography (EtOAc, 2% Et_3N) yielded amine **28** (86% yield, average of two experiments) as a colorless oil. Amine **28**: R_f 0.25 (EtOAc, 2% Et_3N). Spectral data match those previously reported.[32]



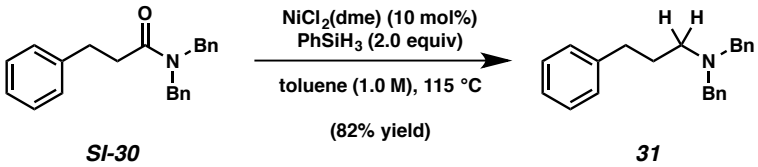
SI-28 ***29***

Amine 29. Purification by preparative thin-layer chromatography (1:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **29** (83% yield, average of two experiments) as a colorless oil. Amine **29**: R_f 0.34 (1:1 Hexanes:EtOAc, 2% Et_3N). Spectral data match those previously reported.[32]



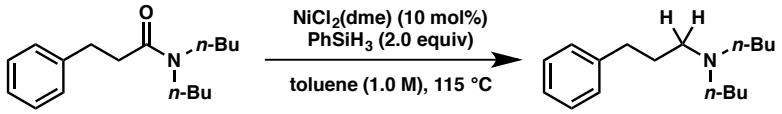
SI-29 ***30***

Amine 30. Purification by flash chromatography (20:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **30** (77% yield, average of two experiments) as a colorless oil. Amine **30**: R_f 0.57 (10:1 Hexanes:EtOAc, 2% Et_3N). Spectral data match those previously reported.[33]



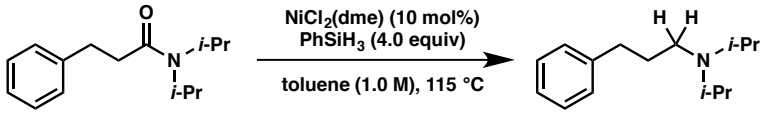
SI-30 ***31***

Amine 31. Purification by flash chromatography (20:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **31** (82% yield, average of two experiments) as a colorless oil. Amine **31**: R_f 0.63 (10:1 Hexanes:EtOAc, 2% Et_3N). Spectral data match those previously reported.[34]



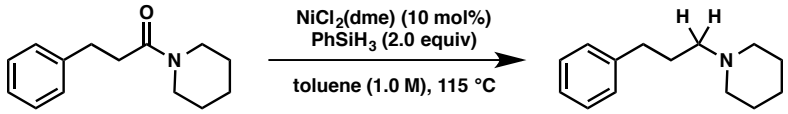
SI-31 ***32***

Amine 32. Purification by flash chromatography (7:3 Hexanes:EtOAc, 2% Et_3N) followed by preparative thin-layer chromatography (7:3 Hexanes:EtOAc, 2% Et_3N) yielded amine **32** (74% yield, average of two experiments) as a colorless oil. Amine **32**: R_f 0.35 (7:3 Hexanes:EtOAc, 2% Et_3N). Spectral data match those previously reported.[35]



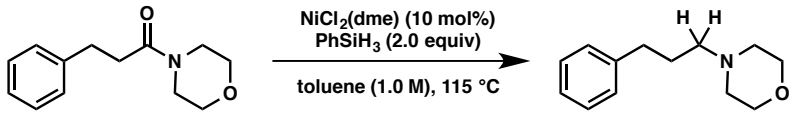
SI-32 ***33***

Amine 33. ^{1}H NMR analysis of the crude reaction mixture indicated a 77% yield (average of two experiments) of amine **33** relative to hexamethylbenzene internal standard. Amine **33**: R_f 0.23 (3:1 Hexanes:EtOAc, 2% Et_3N). Spectral data match those previously reported.[35]



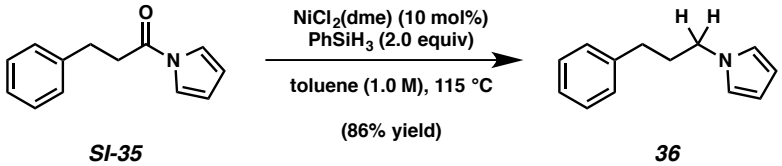
SI-33 ***34***

Amine 34. Purification by preparative thin-layer chromatography (1:4 Hexanes:EtOAc, 2% Et_3N) yielded amine **34** (67% yield, average of two experiments) as a colorless oil. Amine **34**: R_f 0.23 (1:4 Hexanes:EtOAc, 2% Et_3N). Spectral data match those previously reported.[36]



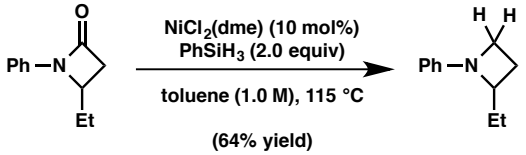
SI-34 ***35***

Amine 35. Purification by preparative thin-layer chromatography (1:4 Hexanes:EtOAc, 2% Et_3N) yielded amine **35** (83% yield, average of two experiments) as a colorless oil. Amine **35**: R_f 0.26 (1:4 Hexanes:EtOAc, 2% Et_3N). Spectral data match those previously reported.[37]



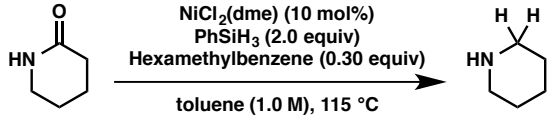
SI-35 ***36***

Amine 36. Purification by flash chromatography (9:1 Hexanes:EtOAc) yielded amine **36** (86% yield, average of two experiments) as a colorless oil. Amine **36**: R_f 0.55 (4:1 Hexanes:EtOAc). Spectral data match those previously reported.[38]



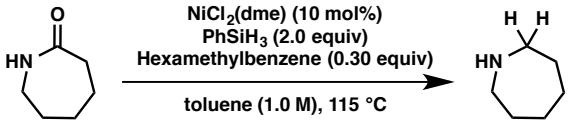
SI-36 ***37***

Amine 37. Purification by flash chromatography (39:1 Pentane:Et_2O) yielded amine **37** (64% yield, average of two experiments) as a colorless oil. Amine **37**: R_f 0.23 (39:1 Pentane:Et_2O). Spectral data match those previously reported.[20]



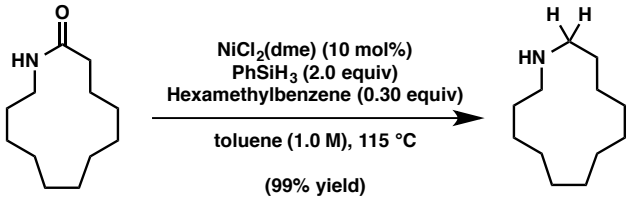
SI-37 ***38***

Amine 38. ^{1}H NMR analysis of the crude reaction mixture indicated a 58% yield (average of two experiments) of amine **38** relative to hexamethylbenzene internal standard. Spectral data match those previously reported.[39]



SI-38 ***39***

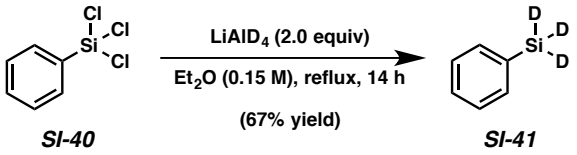
Amine 39. ^{1}H NMR analysis of the crude reaction mixture indicated an 83% yield (average of two experiments) of amine **39** relative to hexamethylbenzene internal standard. Spectral data match those previously reported.[39]



SI-39 ***40***

Amine 40. ^{1}H NMR analysis of the crude reaction mixture indicated a 99% yield (average of two experiments) of amine **40** relative to hexamethylbenzene internal standard. Amine **40**: R_f 0.39 (Hexanes). Spectral data match those previously reported.[40]

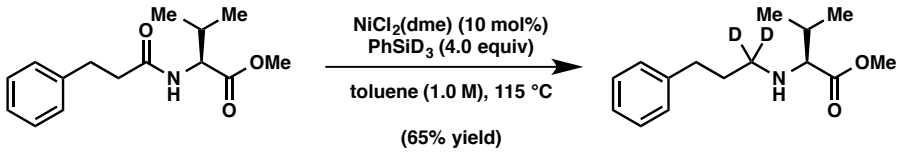
E. Synthesis of $PhSiD_3$



SI-40 ***SI-41***

A round bottom flask equipped with a reflux condenser and a magnetic stir bar was flame-dried under reduced pressure, and then cooled under a N_2 atmosphere. $LiAlD_4$ (1.14 g, 27.21 mmol, 2 equiv) was added, and the flask was flushed with N_2 for 10 min. After cooling to 0 °C, Et_2O (90 mL) was added, followed by $PhSiCl_3$ (**SI-40**) (2.18 mL, 13.61 mmol, 1 equiv) dropwise over 2 min. The resulting grey suspension was refluxed for 14 h. After cooling to room temperature, the resulting suspension was filtered through celite and cooled to 0 °C. The filtrate was then quenched by the dropwise addition of chilled H_2O (50 mL, 0 °C) with vigorous stirring. The reaction mixture was transferred to a separatory funnel and the aqueous layer was extracted with Et_2O (2 x 30 mL). The organic layers were combined, dried over $MgSO_4$, and filtered. Using a rotary evaporator, with a bath cooled to 0 °C, the solution was evaporated at 220 mbar for 2 h until minimal solvent remained. $MgSO_4$ was added and the mixture was filtered through cotton. Using a rotary evaporator, with a bath cooled to 0 °C, the solution was evaporated at 130 mbar for 1 h, and then warmed to 23 °C and evaporated at 20 mbar for 10 min. Desired compound **SI-41** (1.02 g, 67% yield) was obtained as a colorless oil. Spectral data match those previously reported.[41]

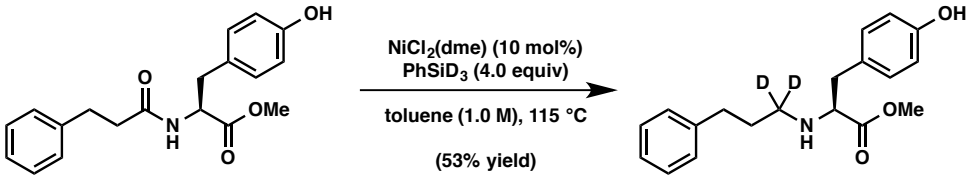
F. Reduction of Amino Acid Derivatives using $PhSiD_3$



SI-42 ***41***

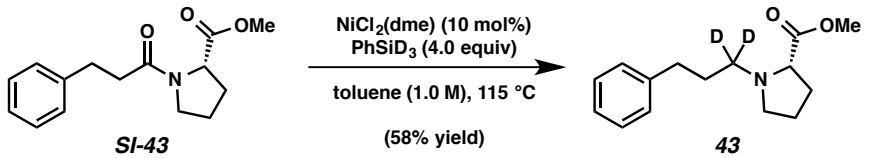
Representative Procedure (reduction of amide SI-42 is used as an example). Amine 41. A 1-dram vial was charged with a magnetic stir bar, flame-dried under reduced pressure, and allowed to cool under a N_2 atmosphere. Amide substrate **SI-42** (26.3 mg, 0.100 mmol, 1.0 equiv) and $NiCl_2$(dme) (2.2 mg, 0.0100 mmol, 10 mol%) were added, and the vial was flushed with N_2 for 5 min. $PhSiD_3$ (49.4 μL, 0.4000 mmol, 4.0 equiv) was added under a N_2 atmosphere via syringe followed by toluene (100 μL, 1.0 M). The vial was then capped with a Teflon-lined screw cap under a flow of N_2. The reaction mixture was then placed in a pre-heated aluminum block and allowed to stir at 115 °C for 24 h. After cooling to room temperature, the reaction mixture was transferred to a separatory funnel with EtOAc (3 mL) and basified with 1.0 M aqueous NaOH (4 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 x 5mL). The combined organic layers were washed with saturated aqueous NaCl (5 mL). The volatiles were removed under reduced pressure, and the crude residue was purified by preparative thin-layer chromatography (4:1 Hexanes:EtOAc) to yield amine **41** (65% yield, average of two experiments) as a colorless oil. Amine **41**: R_f 0.52 (4:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.28–7.26 (m, 2H), 7.18–7.16 (m, 3H), 3.71 (s, 3H), 2.97 (d, J = 6.2, 1H), 2.70–2.59 (m, 2H), 1.88 (dsept, J = 6.8, 6.8, 1H), 1.81–1.71 (m, 2H), 0.95 (d, J = 6.8, 3H), 0.93 (d, J = 6.8, 3H); ^{2}H NMR (76 MHz, $CDCl_3$): δ 2.6, 2.4; ^{13}C NMR (125 MHz, $CDCl_3$): δ 176.0, 142.4, 128.6, 128.4, 125.8, 67.5, 51.5, 47.42 (m), 33.5, 31.8, 31.7, 19.4, 18.9; IR (film): 3336, 3026, 2927, 1732, 1453 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{15}H_{22}D_2NO_2$, 252.19235; found 252.19381; $[\alpha]^{30.4}_D$ –7.33° (c = 0.10, CH_2Cl_2).

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 Figure 5.



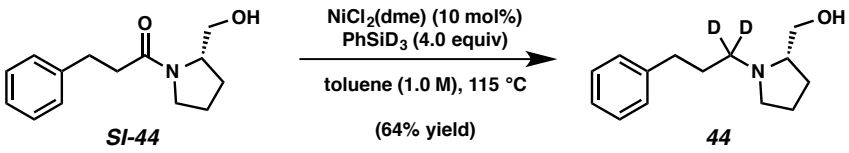
SI-7 ***42***

Amine 42. Purification by preparative thin-layer chromatography (1:1 Hexanes:EtOAc) yielded amine **42** (53% yield, average of two experiments) as a colorless oil. Amine **42**: R_f 0.39 (1:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, CD_2Cl_2): δ 7.25 (t, J = 7.4, 2H), 7.17–7.12 (m, 3H), 7.02 (d, J = 8.4, 2H), 6.72 (d, J = 8.4, 2H), 3.62 (s, 3H), 3.44 (t, J = 6.6, 1H), 2.89–2.82 (m, 2H), 2.60–2.57 (m, 2H), 1.77–1.67 (m, 2H); ^{2}H NMR (76 MHz, CD_2Cl_2): δ 2.59, 2.45; ^{13}C NMR (125 MHz, CD_2Cl_2): (14 of 15 signals observed) δ 175.2, 155.0, 142.6, 130.8, 129.8, 128.8, 128.6, 126.1, 115.5, 63.4, 51.9, 38.9, 35.6, 31.8; IR (film): 3291, 3025, 2927, 1734, 1515 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{19}H_{22}D_2NO_3$, 316.18762; found 316.18708; $[\alpha]\ ^{29.7}_D$ +5.33° (c = 0.10, CH_2Cl_2).



SI-43 ***43***

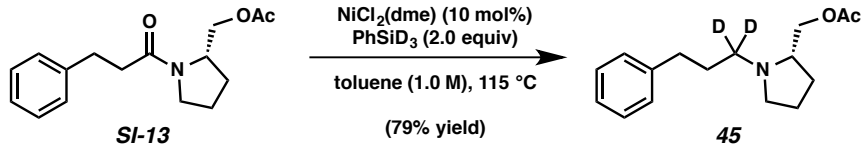
Amine 43. Purification by preparative thin-layer chromatography (1:1 Hexanes:EtOAc) yielded amine **43** (58% yield, average of two experiments) as a colorless oil. Amine **43**: R_f 0.52 (1:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, CD_2Cl_2): δ 7.27–7.25 (m, 2H), 7.20 –7.14 (m, 3H), 3.65 (s, 3H), 3.16–3.09 (m, 2H), 2.68–2.57 (m, 2H), 2.37–2.32 (m, 1H), 2.10–2.01 (m, 1H), 1.91–1.84 (m, 2H), 1.82–1.74 (m, 3H); ^{2}H NMR (76 MHz, $CDCl_3$): δ 2.68, 2.39; ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.9, 142.2, 128.5, 128.4, 125.9, 66.2, 54.0 (m), 53.6, 52.0, 33.8, 30.1, 29.5, 23.3; IR (film): 3059, 2947, 2860, 1731, 1453 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for C_{15}-$H_{20}D_2NO_2$, 250.17587; found 250.17816; $[\alpha]\ ^{30.8}_D$ –57.33° (c = 0.10, CH_2Cl_2).



SI-44 ***44***

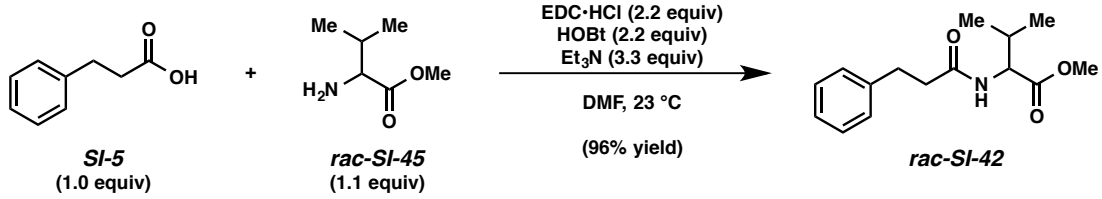
Amine 44. Purification by preparative thin-layer chromatography (EtOAc, 2% Et_3N) yielded amine **44** (64% yield, average of two experiments) as a colorless oil. Amine **44**: R_f 0.45 (EtOAc. 2% Et_3N). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.29–7.26 (m, 2H), 7.19–7.17 (m, 3H), 3.59 (dd, J =

10.6, 3.9, 1H), 3.37 (dd, *J* = 10.6, 2.2, 1H), 3.20–3.16 (m, 1H), 2.73–2.67 (m, 1H), 2.61–2.54 (m, 2H), 2.27–2.21 (m, 1H), 1.90–1.69 (m, 6H); ^{2}H NMR (76 MHz, $CDCl_3$): δ 2.73, 2.29; ^{13}C NMR (125 MHz, $CDCl_3$): δ 142.3, 128.5, 128.4, 125.9, 64.9, 61.9, 54.2, 53.5 (m), 33.7, 30.5, 27.8, 23.8; IR (film): 3362, 3084, 2936, 2800, 1602 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{14}H_{20}D_2NO$, 222.18170; found 222.18214; $[\alpha]^{31.0}_D$ –44.66° (c = 0.10, CH_2Cl_2).



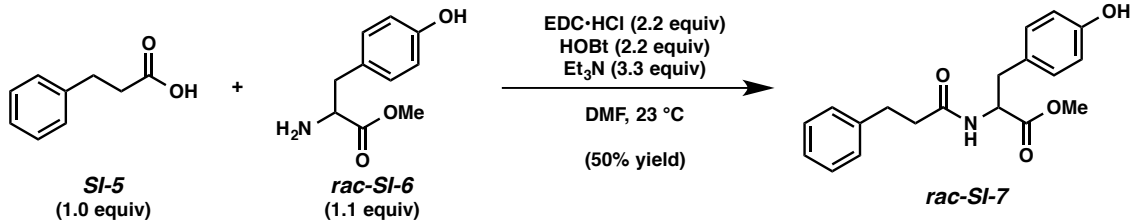
Amine 45. Purification by preparative thin-layer chromatography (1:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **45** (79% yield, average of two experiments) as a colorless oil. Amine **45**: R_f 0.35 (1:1 Hexanes:EtOAc, 2% Et_3N). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.28–7.26 (m, 2H), 7.19–7.16 (m, 3H), 4.04 (dd, *J* = 10.9, 5.1, 1H), 3.92 (dd, *J* = 10.9, 6.1, 1H), 3.15–3.12 (m, 1H), 2.70–2.57 (m, 3H), 2.23–2.18 (m, 1H), 2.01 (s, 3H), 1.92–1.86 (m, 1H), 1.85–1.79 (m, 2H), 1.78–1.71 (m, 2H), 1.62–1.56 (m, 1H); ^{2}H NMR (76 MHz, $CDCl_3$): δ 2.79, 2.33; ^{13}C NMR (125 MHz, $CDCl_3$): δ 171.3, 142.4, 128.5, 128.4, 125.8, 67.4, 62.4, 54.8 (m), 54.4, 33.8, 30.3, 28.4, 23.2, 21.1; IR (film): 3062, 2940, 2785, 1738, 1603 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{16}H_{22}D_2NO_2$, 264.19221; found 264.19271; $[\alpha]^{29.9}_D$ –54.66° (c = 0.10, CH_2Cl_2).

G. Verification of Enantiopuritiy – Racemic Compound Synthesis

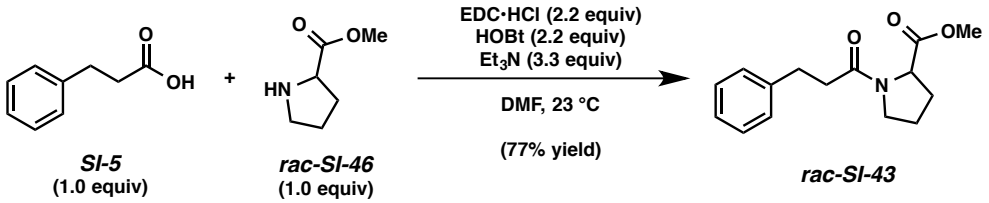


Representative Procedure for the synthesis of racemic amide substrates (synthesis of amide rac-SI-42 is used as an example). Amide rac-SI-42. To a solution of carboxylic acid **SI-5** (300.0 mg, 1.99 mmol, 1.0 equiv), HOBt (594.0 mg, 4.39 mmol, 2.2 equiv), and EDC•HCl (848.0 mg, 4.39 mmol, 2.2 equiv) in DMF (10.0 mL, 0.2 M) at 23 °C was added amine **rac-SI-45** (376.0 mg, 2.19 mmol, 1.1 equiv) followed by triethylamine (0.92 mL, 6.59 mmol, 1.1 equiv) under a N_2 atmosphere. The reaction mixture stirred at 23 °C for 18 h. The reaction was diluted with deionized water (50 mL) and transferred to a separatory funnel with EtOAc (40 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 x 40 mL). The organic

layers were combined and washed with water (3 x 40 mL), dried over Na_2SO_4, and the volatiles were removed under reduced pressure. The resulting crude residue was purified by flash chromatography (3:1 Hexanes:EtOAc) to yield amide **rac-SI-42** (506.0 mg, 96% yield) as an off-white solid.

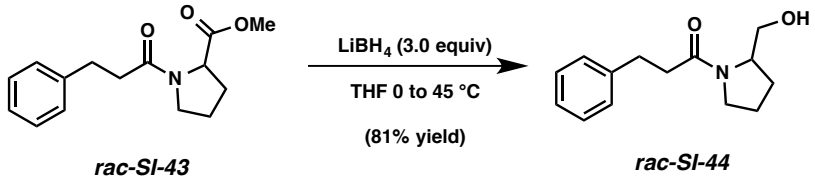


Amide rac-SI-7. Following representative procedure. Purification by flash chromatography (3:1 Hexanes:EtOAc → 1:1 Hexanes:EtOAc) to yield amide **rac-SI-7** (269.3 mg, 50% yield) as a white solid.



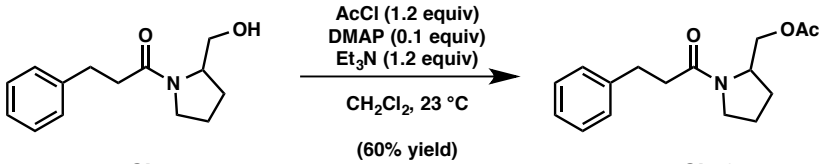
Amide rac-SI-43. Following representative procedure. Purification by flash chromatography (1:1 Hexanes:EtOAc → 1:3 Hexanes:EtOAc) to yield amide **rac-SI-43** (221.1 mg, 77% yield) as a colorless oil.

Synthesis of racemic proline-derived amide substrates



Amide rac-SI-44. To a solution of amide **rac-SI-43** (100.0 mg, 0.383 mmol, 1 equiv) in THF (8.0 mL, 0.05 M) at 0 °C under a N_2 atmosphere, was added $LiBH_4$ (25.0 mg, 1.15 mmol, 3 equiv) in one portion. The vial was then capped with a Teflon-lined screw cap under a flow of N_2 and placed in a pre-heated aluminum block and allowed to stir at 45 °C for 15 h. After cooling to room temperature, deionized H_2O was added (2 mL) and the reaction mixture was transferred to a separatory funnel with CH_2Cl_2 (3 mL) and extracted with CH_2Cl_2 (3 x 3mL). The combined

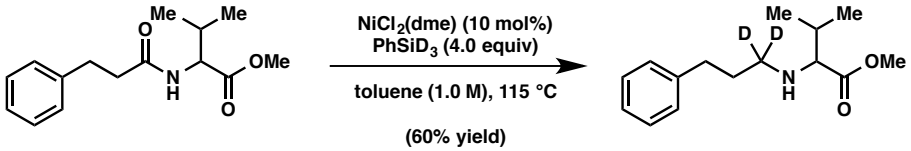
organic layers were washed with saturated aqueous NaCl (5 mL). The volatiles were removed under reduced pressure, and the resulting crude residue was purified by flash chromatography (1:3 Hexanes:EtOAc → EtOAc) to yield amide **rac-SI-44** (72.0 mg, 81% yield) as a colorless oil.



rac-SI-44 ***rac-SI-13***

Amide rac-SI-13. A 1-dram vial was charged with a magnetic stir bar, flame-dried under reduced pressure, and allowed to cool under a N_2 atmosphere. Amide substrate **rac-SI-44** (25.6 mg, 0.110 mmol, 1 equiv) and DMAP (1.3 mg, 0.010 mmol, 0.1 equiv) were added and the vial flushed with N_2 for 5 min. CH_2Cl_2 (2.0 mL, 0.05 M), AcCl (9.4 μL, 0.132 mmol, 1.2 equiv), and Et_3N (18.3 μL, 0.132 mmol, 1.2 equiv) were added under a N_2 atmosphere and the reaction was left to stir for 3 h at 23 °C. NH_4Cl (2 mL) was added and the reaction mixture was transferred to separatory funnel with CH_2Cl_2 (3 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3 x 3 mL). The combined organic layers were washed with saturated aqueous NaCl (5 mL). The volatiles were removed under reduced pressure, and the resulting crude residue was purified by preparative thin-layer chromatography (1:3 Hexanes:EtOAc → EtOAc) to yield amide **rac-SI-13** (16.0 mg, 60% yield) as a colorless oil.

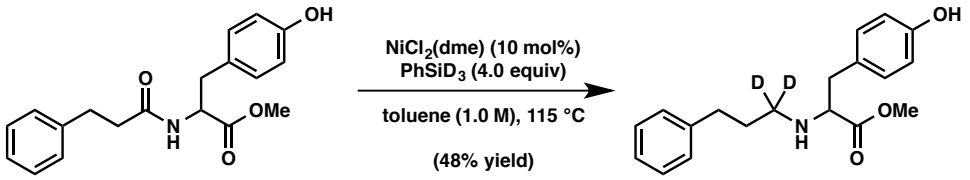
Representative Procedure for the deuterium reduction of racemic amino acid derivatives



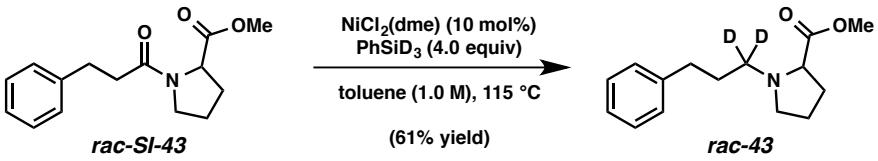
rac-SI-42 ***rac-41***

Representative Procedure (reduction of amide rac-SI-42 is used as an example). Amine rac-41. A 1-dram vial was charged with a magnetic stir bar, flame-dried under reduced pressure, and allowed to cool under a N_2 atmosphere. Amide substrate **rac-SI-42** (26.3 mg, 0.100 mmol, 1.0 equiv) and $NiCl_2$(dme) (2.2 mg, 0.0100 mmol, 10 mol%) was added, and the vial was flushed with N_2. $PhSiD_3$ (49.4 μL, 0.4000 mmol, 4.0 equiv) was added under a N_2 atmosphere via syringe followed by toluene (100 μL, 1.0 M). The vial was then capped with a Teflon-lined screw cap under a flow of N_2. The reaction mixture was then placed in a pre-heated aluminum block and

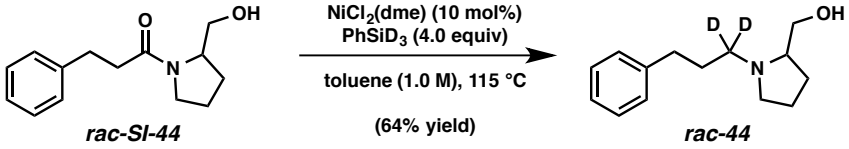
allowed to stir at 115 °C for 24 h. After cooling to room temperature, the reaction mixture was transferred to a separatory funnel with EtOAc (3 mL) and basified with 1.0 M aqueous NaOH (4 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 x 5mL). The combined organics were washed with saturated aqueous NaCl (5 mL). The volatiles were removed under reduced pressure, and the crude residue was purified by preparative thin-layer chromatography (4:1 Hexanes:EtOAc) to yield amine **rac-41** (15.0 mg, 60% yield) as a colorless oil.



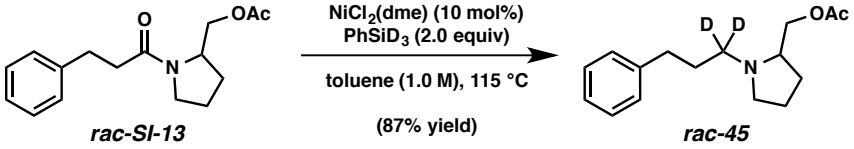
Amine rac-42. Purification by preparative thin-layer chromatography (1:1 Hexanes:EtOAc) yielded amine **rac-42** (15.0 mg, 48% yield) as a colorless oil.



Amine rac-43. Purification by preparative thin-layer chromatography (1:1 Hexanes:EtOAc) yielded amine **rac-43** (15.2 mg, 61% yield) as a colorless oil.



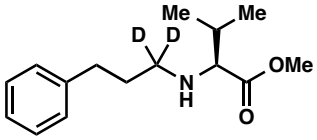
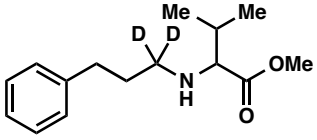
Amine rac-44. Purification by preparative thin-layer chromatography (EtOAc, 2% Et_3N) yielded amine **rac-44** (14.1 mg, 64% yield) as a colorless oil.

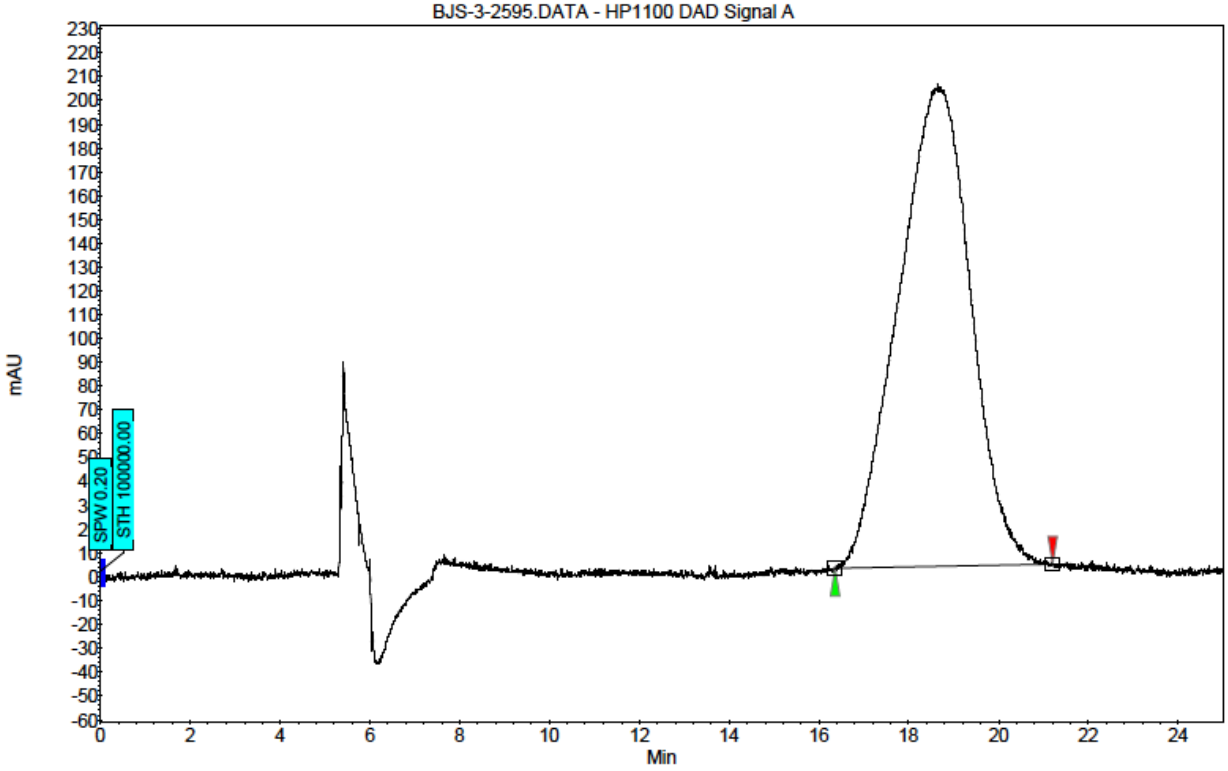


Amine rac-45. Purification by preparative thin-layer chromatography (1:1 Hexanes:EtOAc, 2% Et_3N) yielded amine **rac-45** (22.2 mg, 87% yield) as a colorless oil.

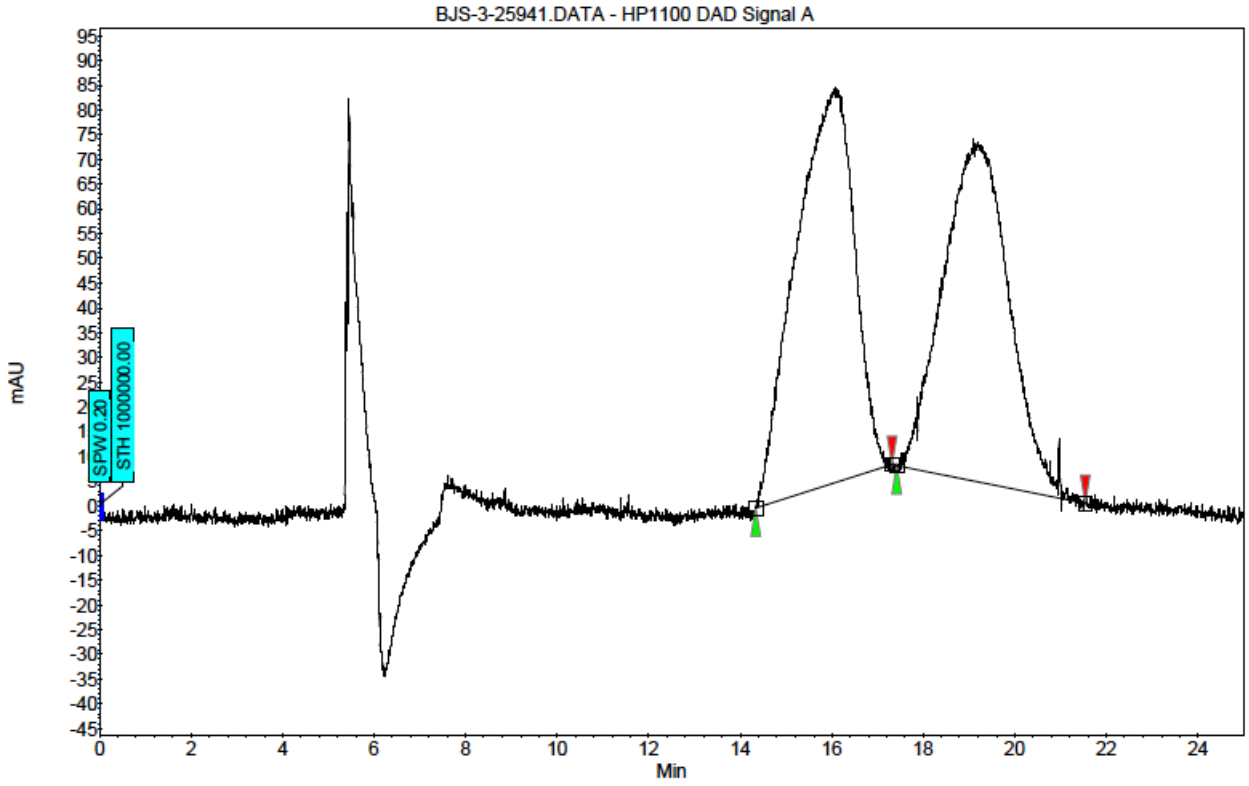
H. Verification of Enantiopuritiy – er and dr determination

The enantiomeric ratio of **41** was determined by chiral SFC analysis.

Compound	Method Column /Temp	Polar Cosolvent	Method Flow Rate	Retention Times	Enantiomeric Ratio (er)
 ***41***	Daicel ChiralPak OB-H / 35 °C	1% iPrOH	0.60 mL/min	16.36/21.20 min	0:100
 ***rac-41***	Daicel ChiralPak OB-H / 35 °C	1% iPrOH	0.60 mL/min	14.34/17.31 min 17.42/21.53 min	51:49

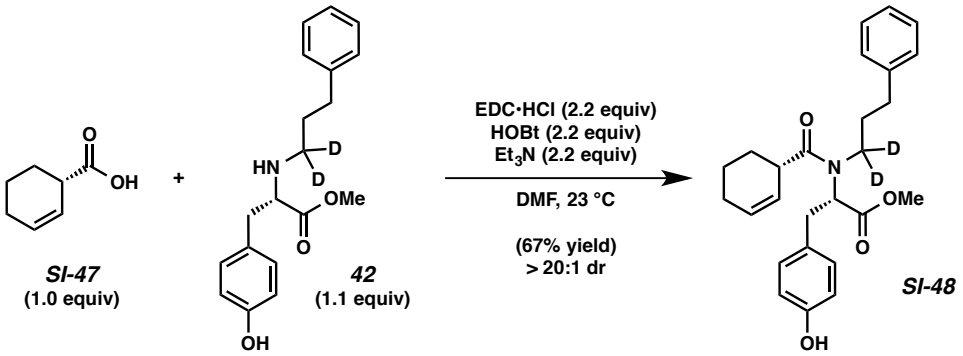


Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	16.36	18.64	21.20	0.00	100.00	202.4	375.6	100.000
Total						100.00	202.4	375.6	100.000



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	14.34	16.07	17.31	0.00	50.91	79.8	118.9	50.910
2	UNKNOWN	17.42	19.10	21.53	0.00	49.09	69.1	114.7	49.090
Total						100.00	148.9	233.6	100.000

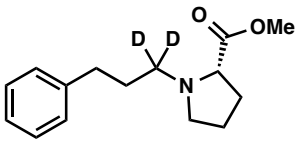
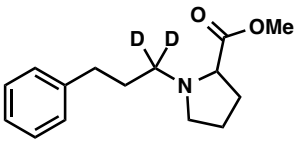
Chiral SFC analysis of **rac-42** did not lead to separation of the enantiomers. The diastereomeric ratio of **42** was then determined by coupling with enantiopure carboxylic acid **SI-47**.

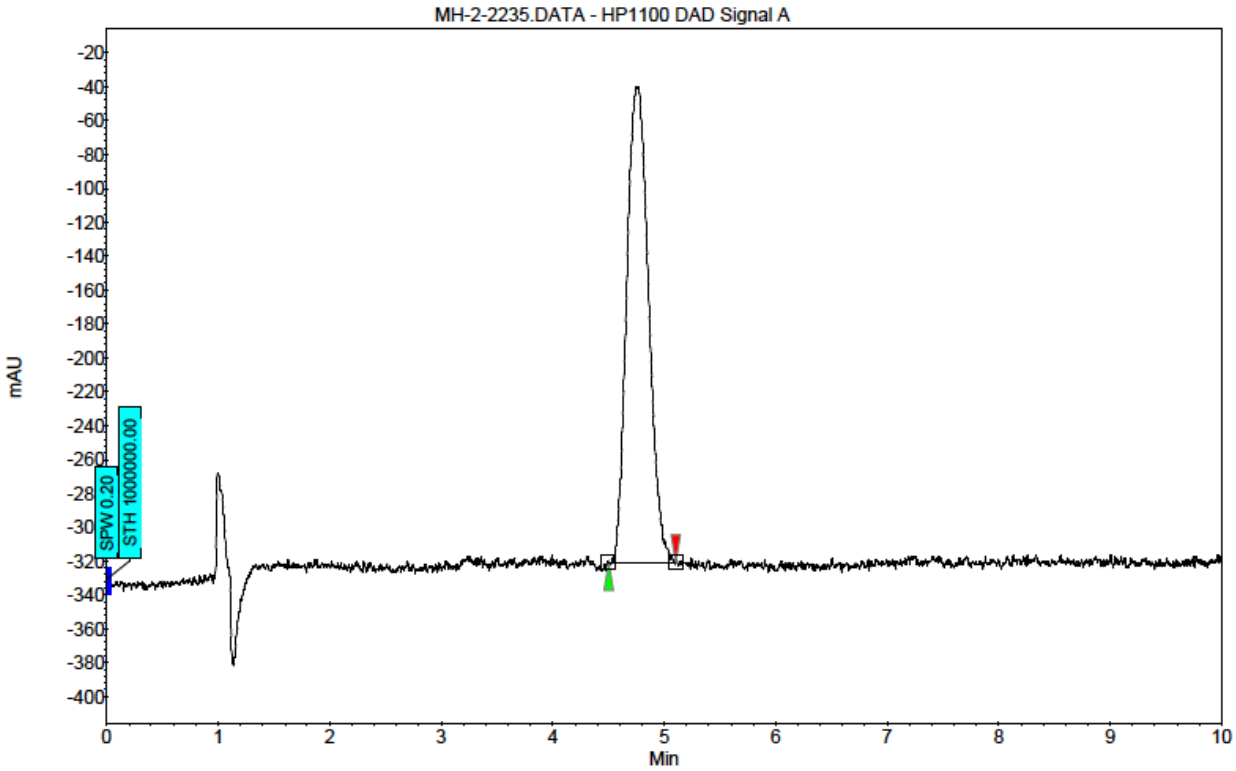


Amide SI-48. To a solution of carboxylic acid **SI-47** (7.3 mg, 0.058 mmol, 1.0 equiv), HOBt (17.3 mg, 0.128 mmol, 2.2 equiv), and EDC•HCl (24.5 mg, 0.128 mmol, 2.2 equiv) in DMF (582 µL, 0.1 M) at 23 °C was added amine **42** (20.2 mg, 0.064 mmol, 1.1 equiv), followed by

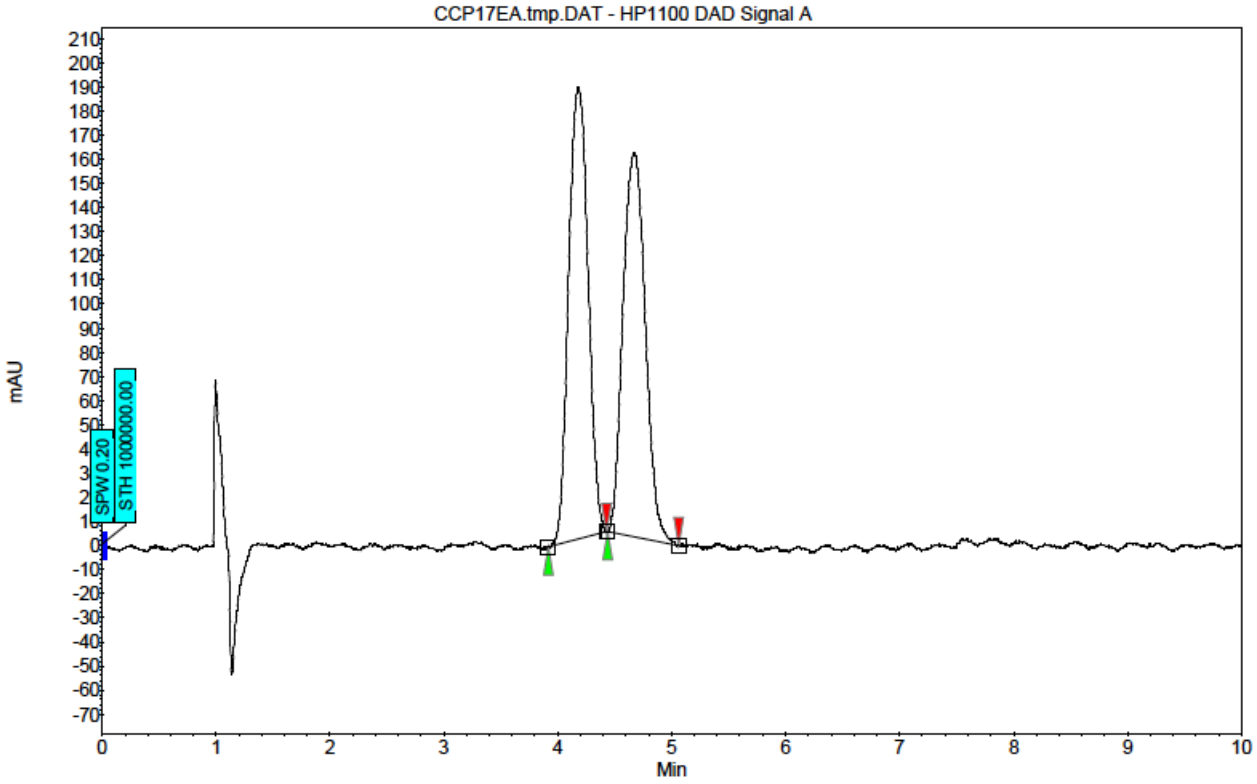
triethylamine (17.8 μL, 0.128 mmol, 2.2 equiv) under a N_2 atmosphere. The reaction mixture stirred at 23 °C for 18 h. The reaction was diluted with deionized water (2 mL) and transferred to a separatory funnel with EtOAc (3 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 x 3 mL). The organic layers were combined and washed with water (3 x 3 mL), dried over Na_2SO_4, and the volatiles were removed under reduced pressure. The resulting crude residue was purified by preparative thin-layer chromatography (3:1 Hexanes:EtOAc) to yield amide **SI-48** (16.6 mg, 67% yield, > 20:1 dr) as a colorless oil. Amide **SI-48**: R_f 0.50 (2:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.27–7.24 (m, 2H), 7.19–7.17 (m, 3H), 7.14–7.12 (m, 2H), 7.00–6.99 (m, 2H), 5.74–5.73 (m, 2H), 3.63 (s, 3H), 3.47 (t, J = 6.9, 1H), 2.92 (d, J = 6.9, 2H), 2.83–2.78 (m, 1H), 2.59 (t, J = 7.7, 2H), 2.40–2.38 (m, 2H), 2.19–2.14 (m, 3H), 1.87–1.81 (m, 1H), 1.80–1.70 (m, 2H); ^{2}H NMR (76 MHz, CD_2Cl_2): δ 2.61, 2.45; ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.2, 174.5, 149.8, 142.1, 134.9, 130.2, 128.5, 128.4, 126.9, 125.9, 125.1, 121.6, 63.1, 51.8, 47.3 (m), 39.5, 39.3, 33.4, 31.5, 27.5, 25.2, 24.5 IR (film): 3331, 3025, 2925, 1752, 1507 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{26}H_{30}D_2NO_4$, 424.24514; found 424.24403; $[\alpha]^{31.3}_D$ –34.00° (c = 0.10, CH_2Cl_2).

The enantiomeric ratio of **43** was determined by chiral SFC analysis.

Compound	Method Column /Temp	Polar Cosolvent	Method Flow Rate	Retention Times	Enantiomeric Ratio (er)
 ***43***	Daicel ChiralPak OB-H / 35 °C	3% iPrOH	3.00 mL/min	4.50/5.10 min	0:100
 ***rac-43***	Daicel ChiralPak OB-H / 35 °C	3% iPrOH	3.00 mL/min	3.92/4.43 min 4.44/5.06 min	50:50

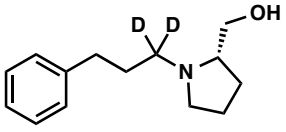
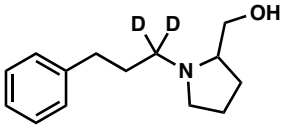


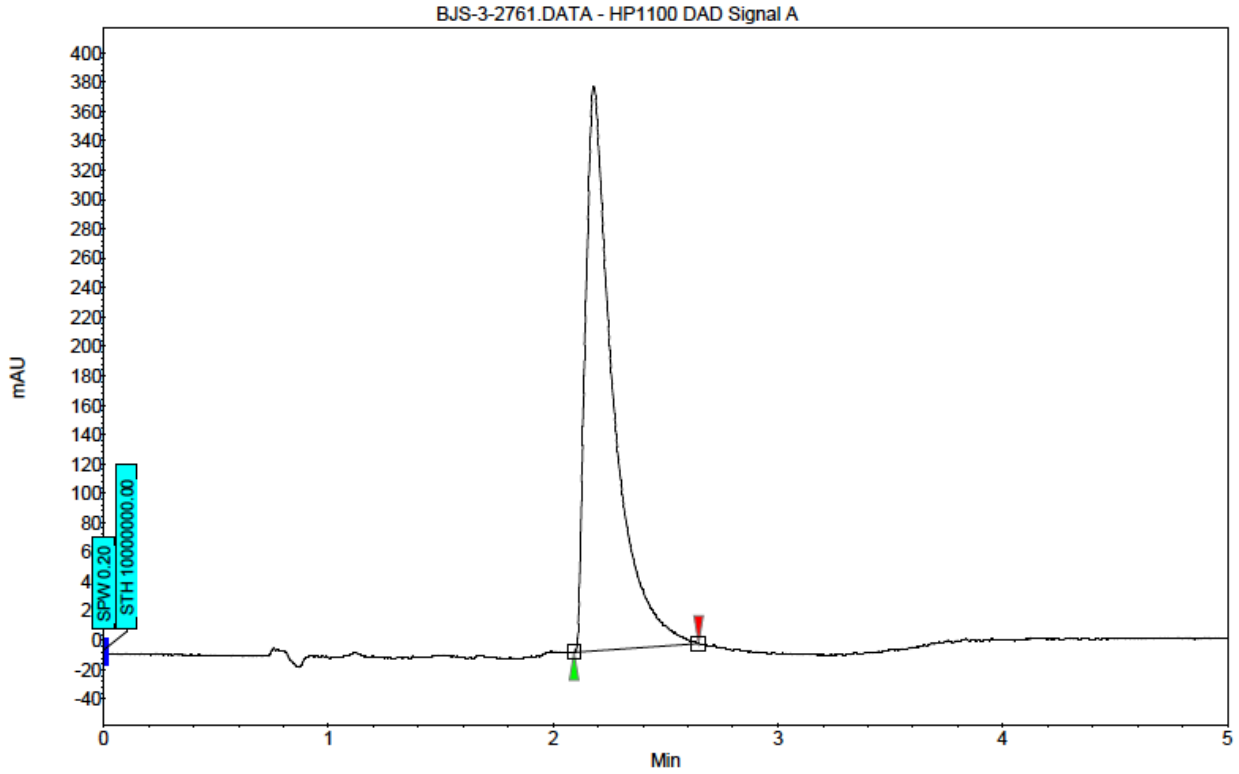
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	4.50	4.75	5.10	0.00	100.00	280.7	63.7	100.000
Total						100.00	280.7	63.7	100.000



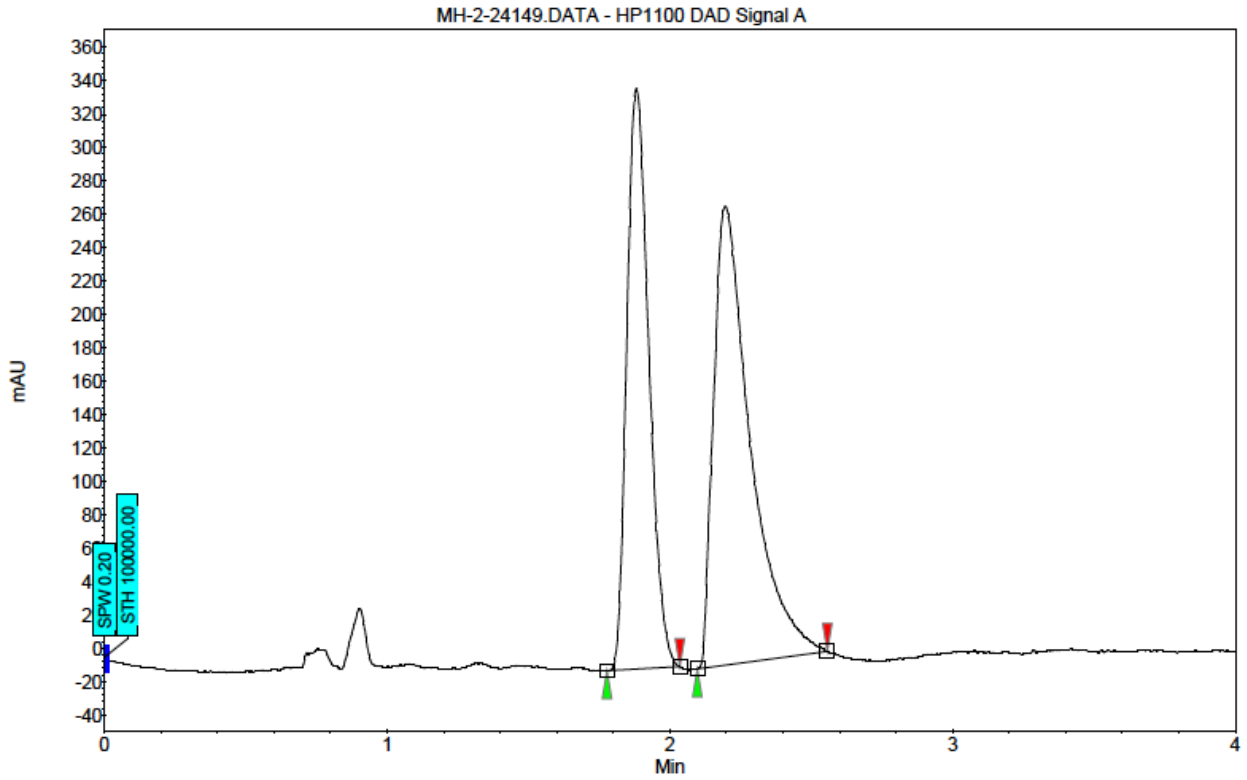
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	3.92	4.18	4.43	0.00	50.60	187.4	37.1	50.602
2	UNKNOWN	4.44	4.67	5.06	0.00	49.40	159.2	36.3	49.398
Total						100.00	346.7	73.4	100.000

The enantiomeric ratio of **44** was determined by chiral SFC analysis.

Compound	Method Column /Temp	Polar Cosolvent	Method Flow Rate	Retention Times	Enantiomeric Ratio (er)
 ***44***	Daicel ChiralPak OD-H / 35 °C	15% MeOH, 2.00 min 10%/min, 25% MeOH	4.00 mL/min	2.09/2.65 min	0:100
 ***rac-44***	Daicel ChiralPak OD-H / 35 °C	15% MeOH, 2.00 min 10%/min, 25% MeOH	4.00 mL/min	1.78/2.03 min 2.10/2.56 min	43:57

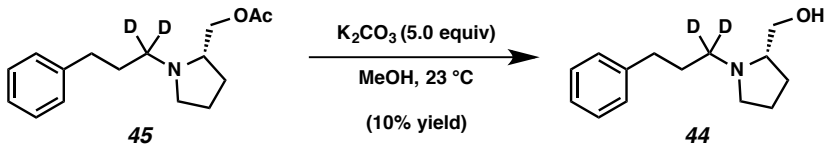


Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	2.09	2.18	2.65	0.00	100.00	384.5	54.9	100.000
Total						100.00	384.5	54.9	100.000



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	1.78	1.88	2.03	0.00	43.15	347.9	31.4	43.149
2	UNKNOWN	2.10	2.20	2.56	0.00	56.85	275.2	41.4	56.851
Total						100.00	623.1	72.8	100.000

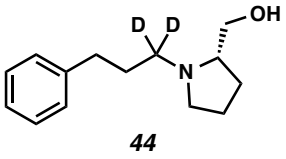
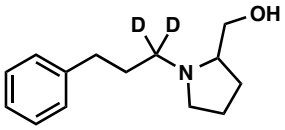
Chiral SFC analysis of **rac-45** did not lead to separation of the enantiomers. For this reason, acetate **45** was converted to alcohol **44** and the enantiomeric ratio was determined by chiral SFC analysis in comparison to **rac-44**.

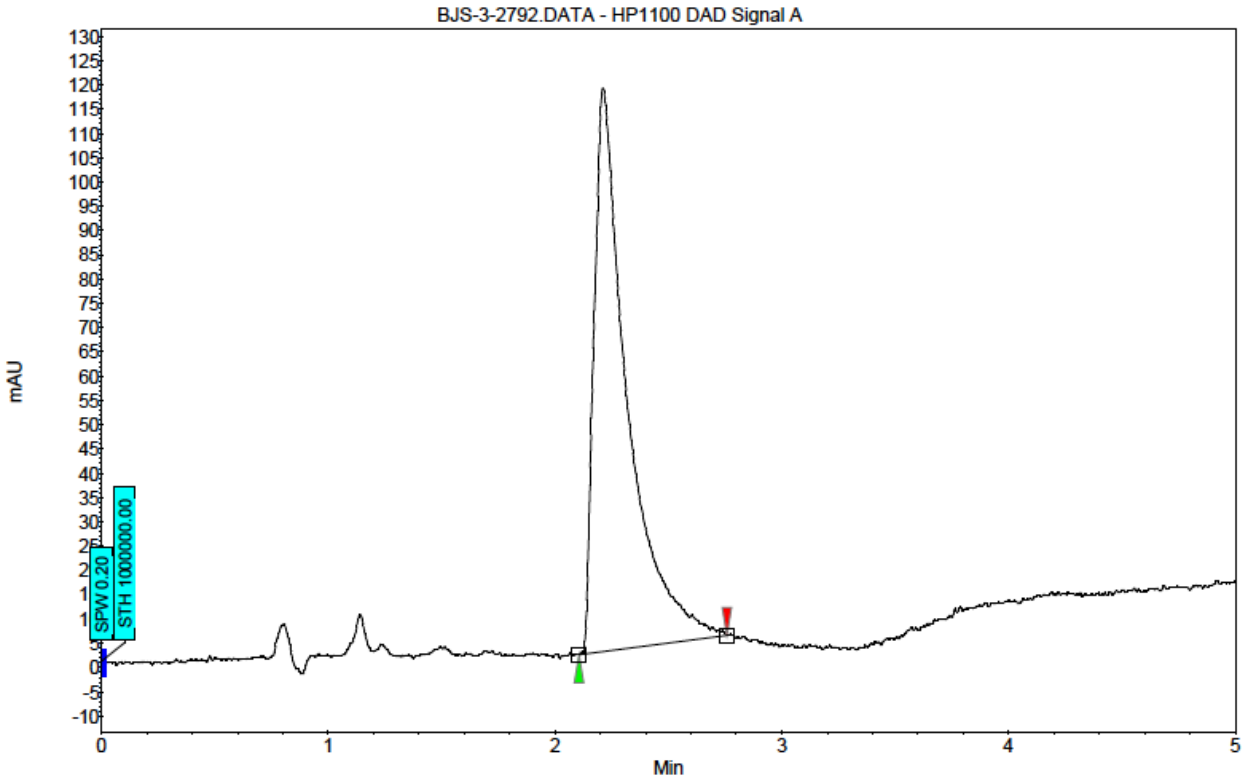


45 ***44***

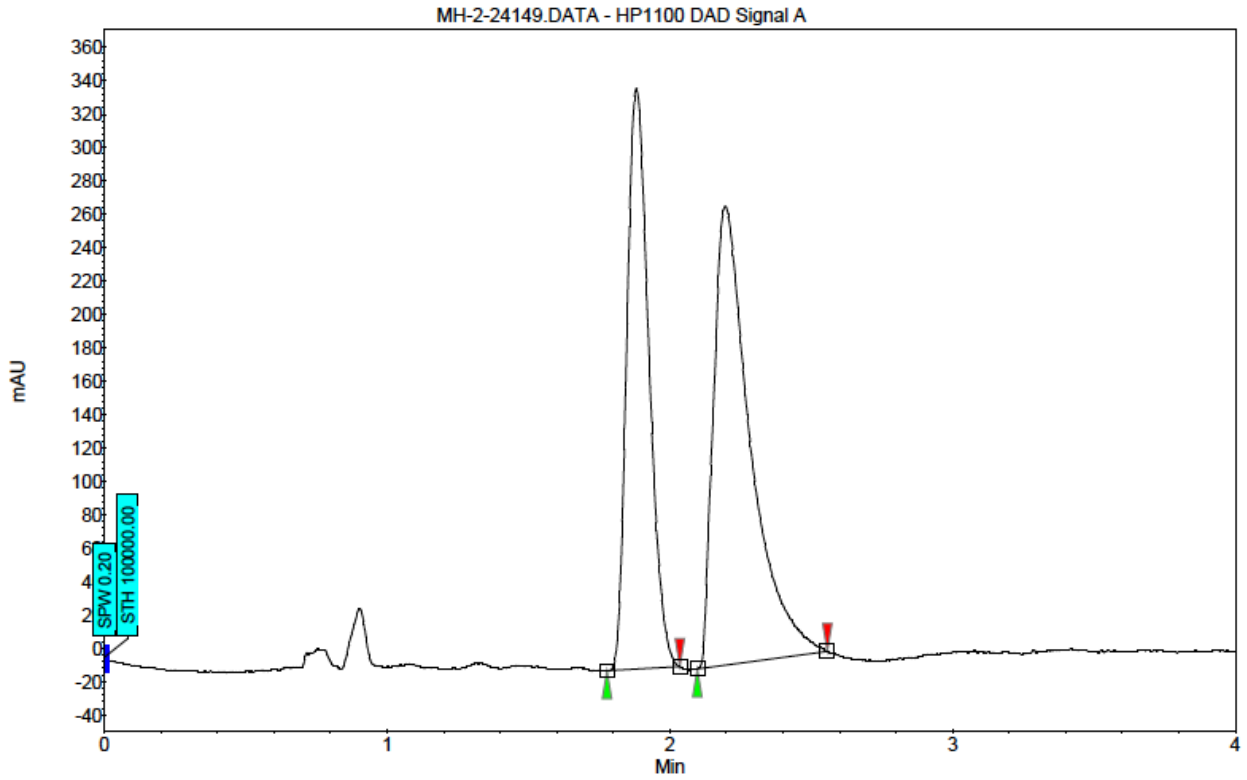
Amine 44. To a solution of amine **45** (20.0 mg, 0.073 mmol, 1 equiv) in MeOH (726 µL, 0.1 M) at 23 °C was added K_2CO_3 (50.2 mg, 0.363 mmol, 5 equiv) in one portion. The reaction was stirred vigorously for 1 h. The reaction mixture was diluted with EtOAc (10 mL) and transferred to a seperatory funnel. Deionized H_2O (10 mL) was added, the layers separated, and the aqueous layer was extracted with EtOAc (3 x 10 mL). The combined organics were washed with

saturated aqueous NaCl (5 mL) and dried over Na_2SO_4. The volatiles were removed under reduced pressure, and the resulting crude residue was purified by preparative thin-layer chromotagraphy (EtOAc, 2% Et_3N) to yield amine **44** (1.6 mg, 10% yield) as a colorless oil.

Compound	Method Column /Temp	Polar Cosolvent	Method Flow Rate	Retention Times	Enantiomeric Ratio (er)
 ***44***	Daicel ChiralPak OD-H / 35 °C	15% MeOH, 2.00 min 10%/min, 25% MeOH	4.00 mL/min	2.10/2.76 min	0:100
 ***rac-44***	Daicel ChiralPak OD-H / 35 °C	15% MeOH, 2.00 min 10%/min, 25% MeOH	4.00 mL/min	1.78/2.03 min 2.10/2.56 min	43:57

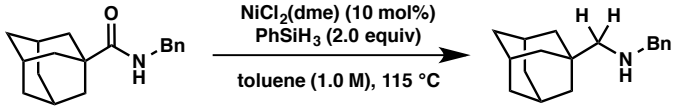


Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	2.10	2.21	2.76	0.00	100.00	116.0	19.7	100.000
Total						100.00	116.0	19.7	100.000



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	1.78	1.88	2.03	0.00	43.15	347.9	31.4	43.149
2	UNKNOWN	2.10	2.20	2.56	0.00	56.85	275.2	41.4	56.851
Total						100.00	623.1	72.8	100.000

I. Procedure for Reduction Performed on 1.0 mmol Scale



SI-24 *22*

Representative procedure for the 1.0 mmol scale reduction of amide SI-24. Amine 22. A 20-scintillation vial was charged with a magnetic stir bar, flame-dried under reduced pressure, and allowed to cool under a N_2 atmosphere. Amide substrate **SI-24** (269.2 mg, 1.000 mmol, 1.0 equiv) and $NiCl_2$(dme) (21.9 mg, 0.1000 mmol, 10 mol %) was added, and the vial was flushed with N_2 for 10 min. $PhSiH_3$ (246.7 µL, 2.000 mmol, 2.0 equiv) was added under a N_2 atmosphere via syringe followed by toluene (1.0 mL, 1.0 M). The vial was then capped with a Teflon-lined screw cap under a flow of N_2. The reaction mixture was then placed in a pre-heated aluminum

block and allowed to stir at 115 °C for 24 h. After cooling to room temperature, the reaction mixture was transferred to a separatory funnel with EtOAc (10 mL) and basified with 1.0 M aqueous NaOH (10 mL). The layers were separated and the aqueous layer was extracted with EtOAc (3 x 30 mL). The combined organic layers were washed with saturated aqueous NaCl (45 mL). The volatiles were removed under reduced pressure, and the crude residue was purified by flash chromatography (4:1 Hexanes:EtOAc, 2% Et_3N) to yield amine **22** (92% yield) as a colorless oil. Amine **22**: R_f 0.52 (4:1 Hexanes:EtOAc, 2% Et_3N). Spectral data match those previously reported.[31]

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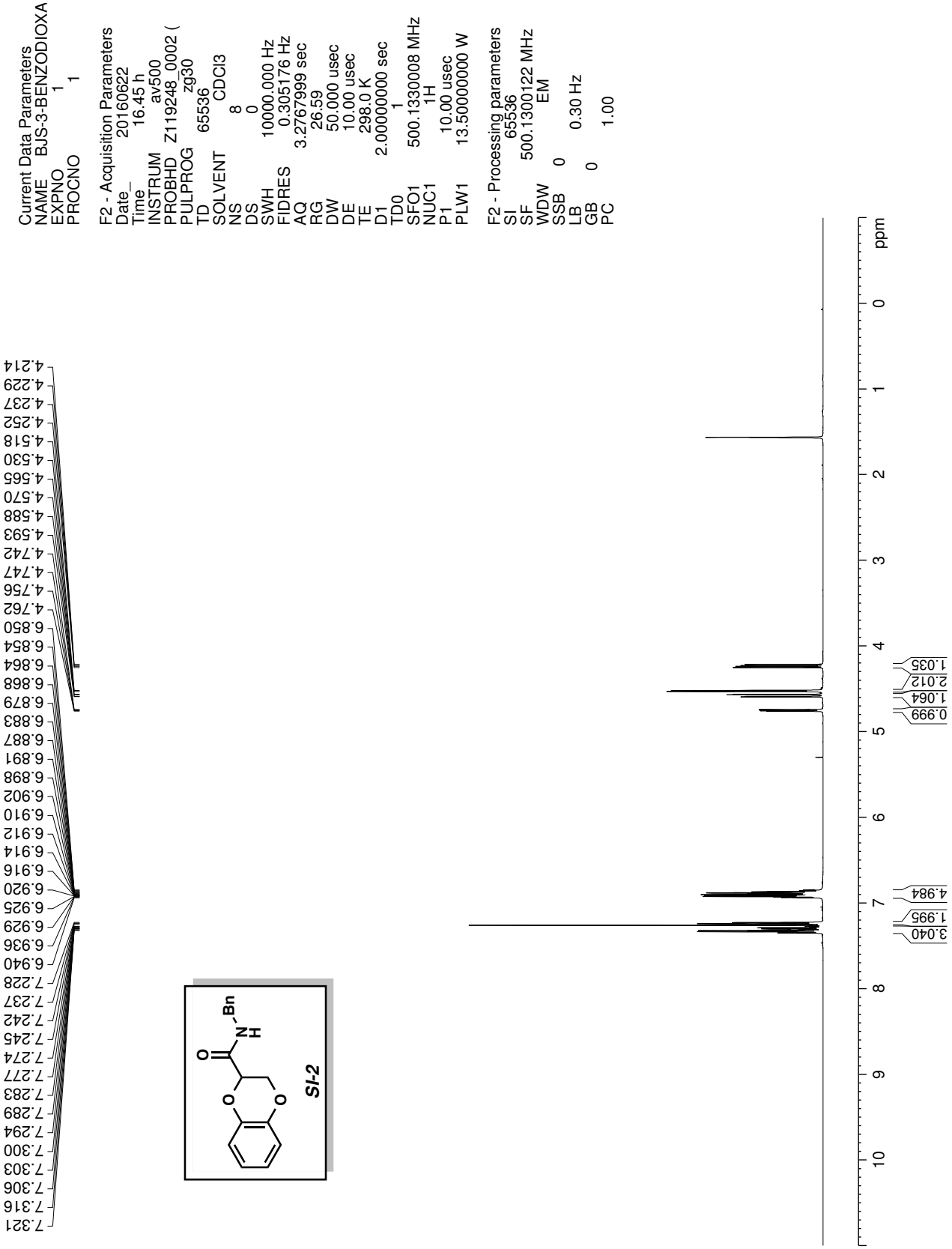
^{1}H NMR Spectra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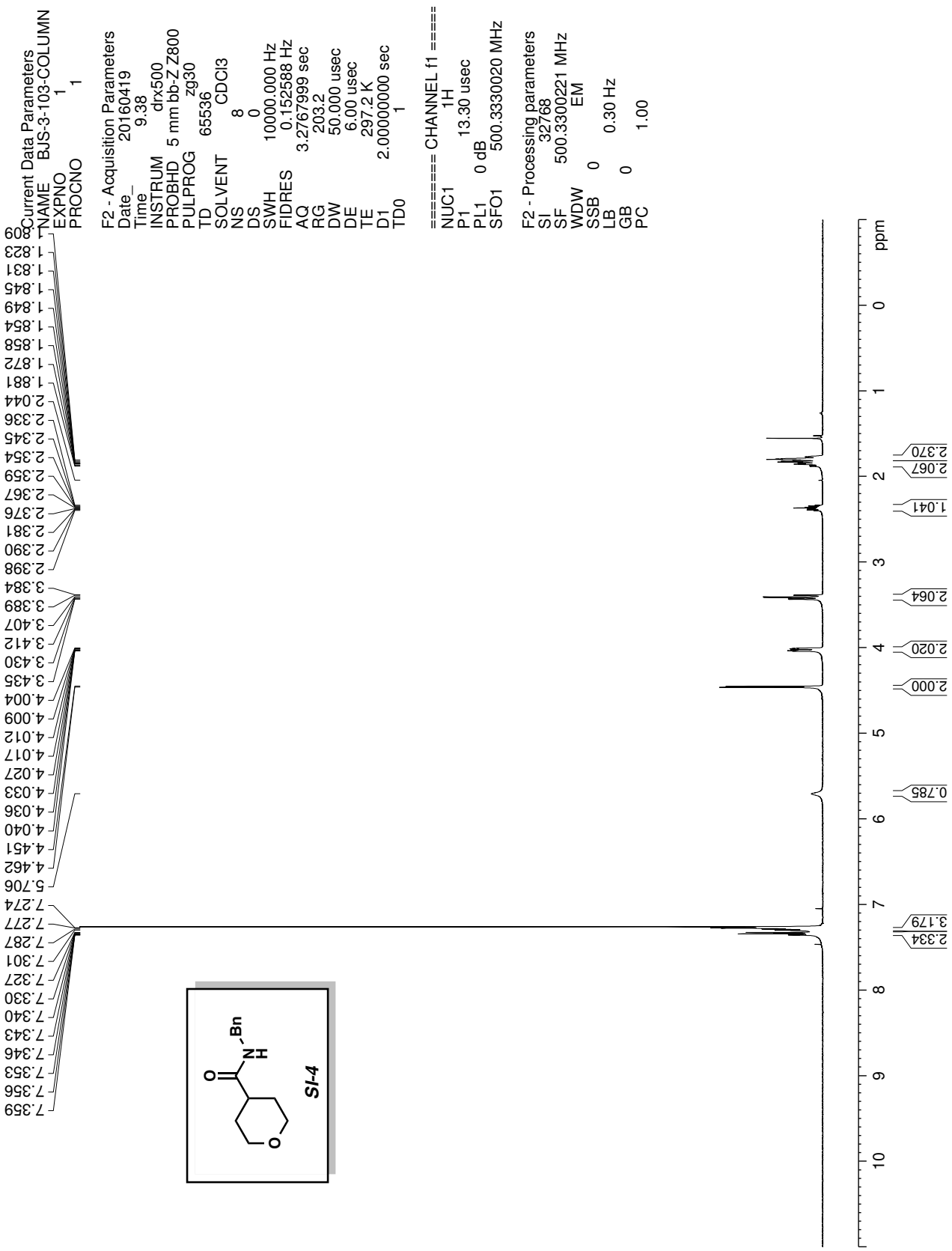
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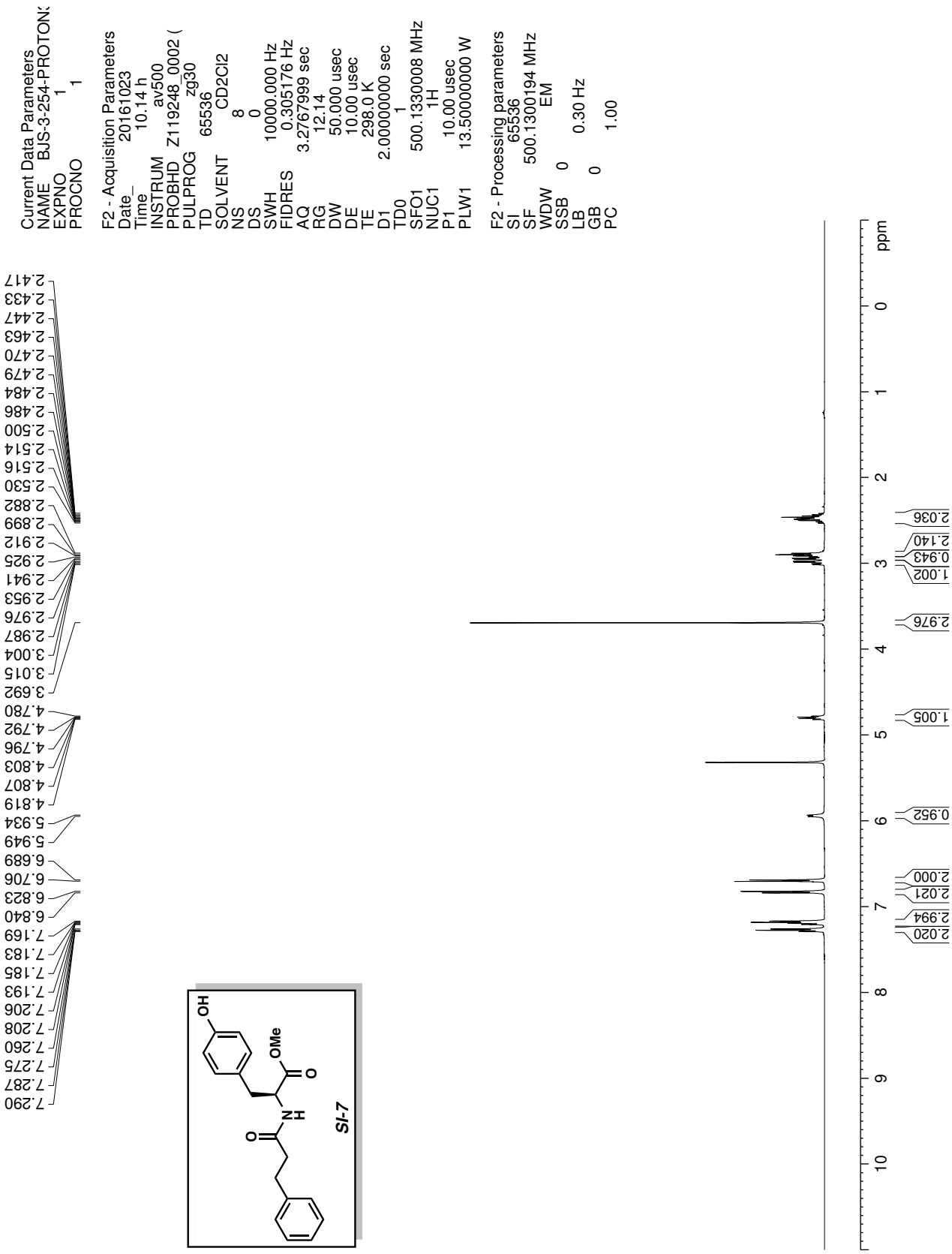
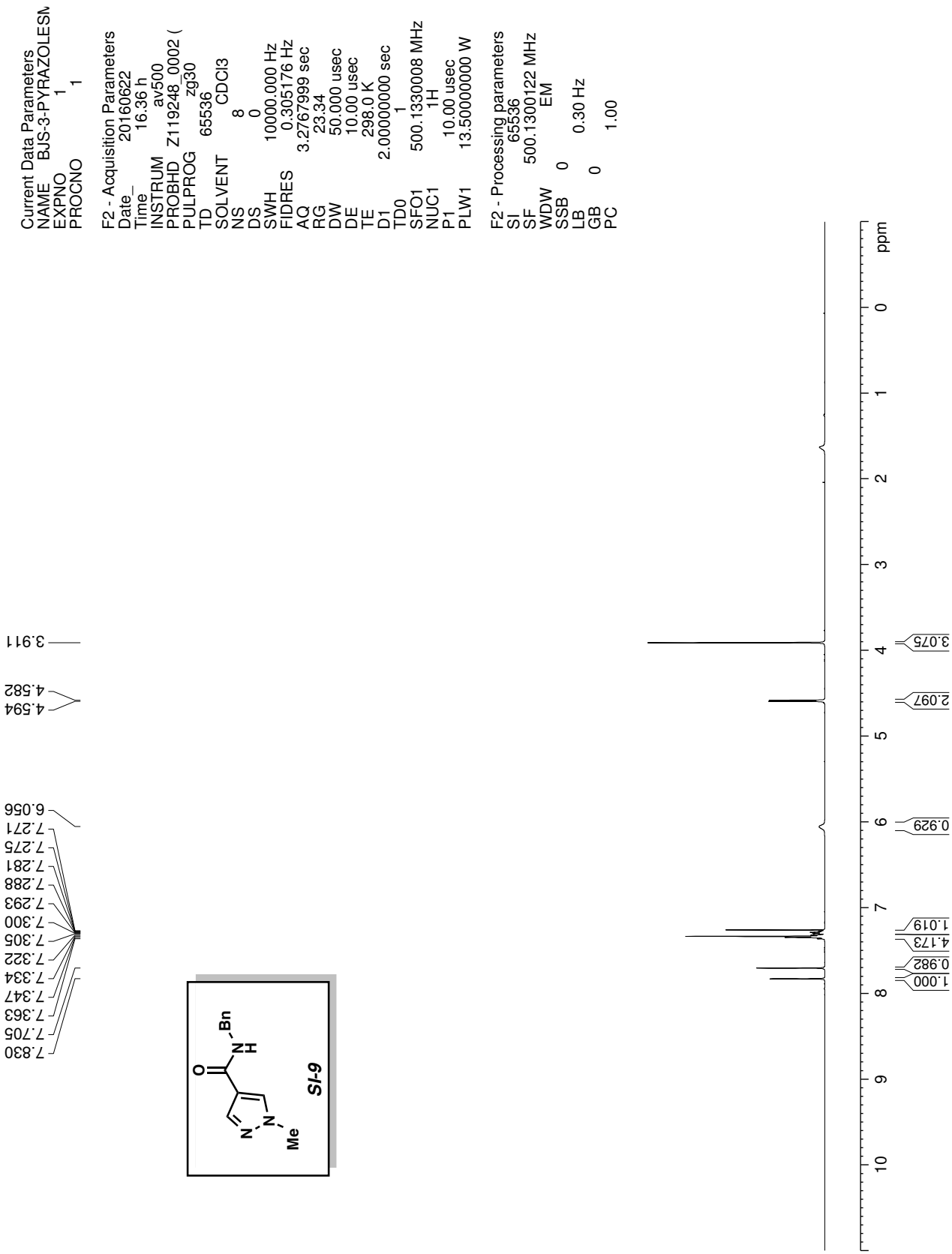
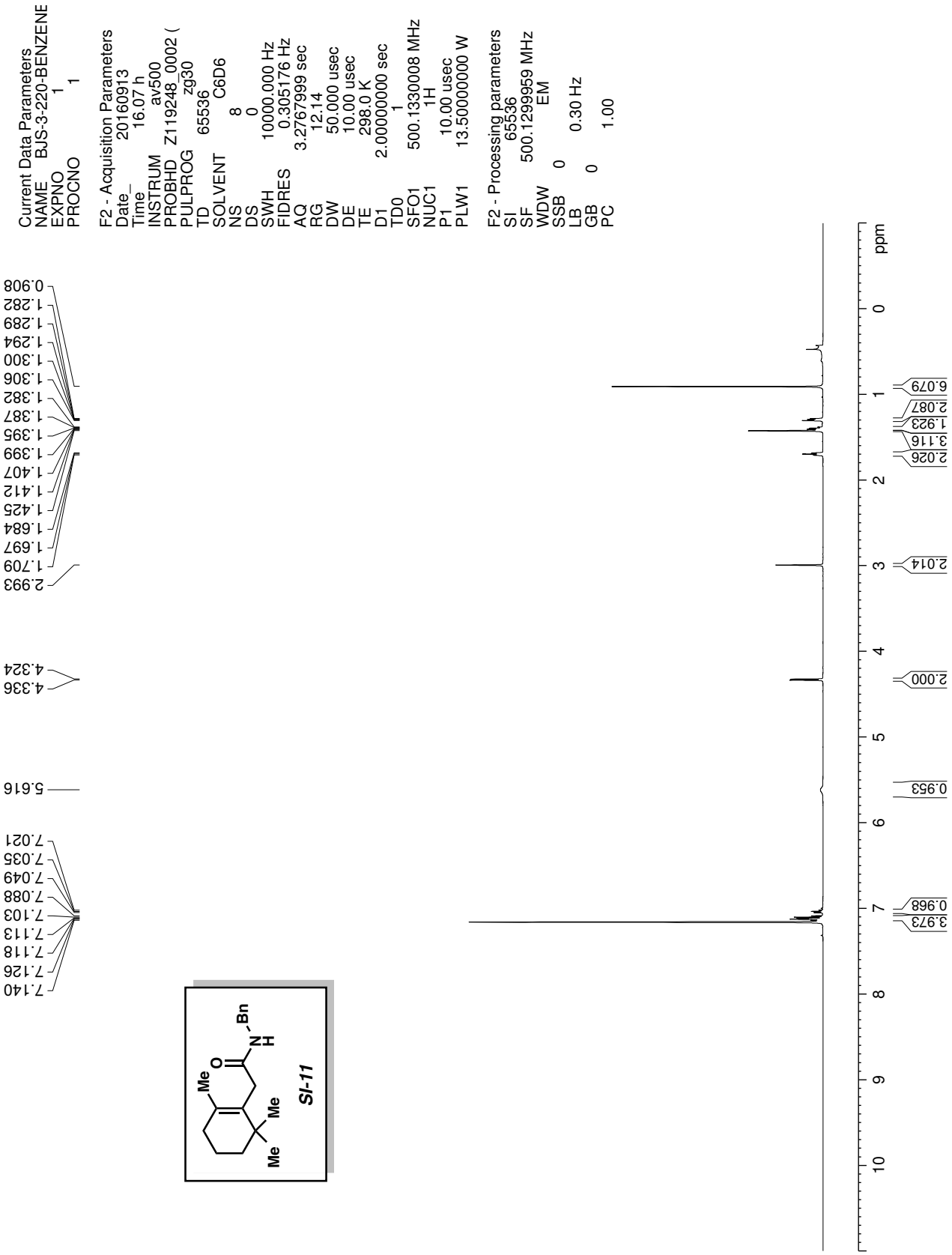
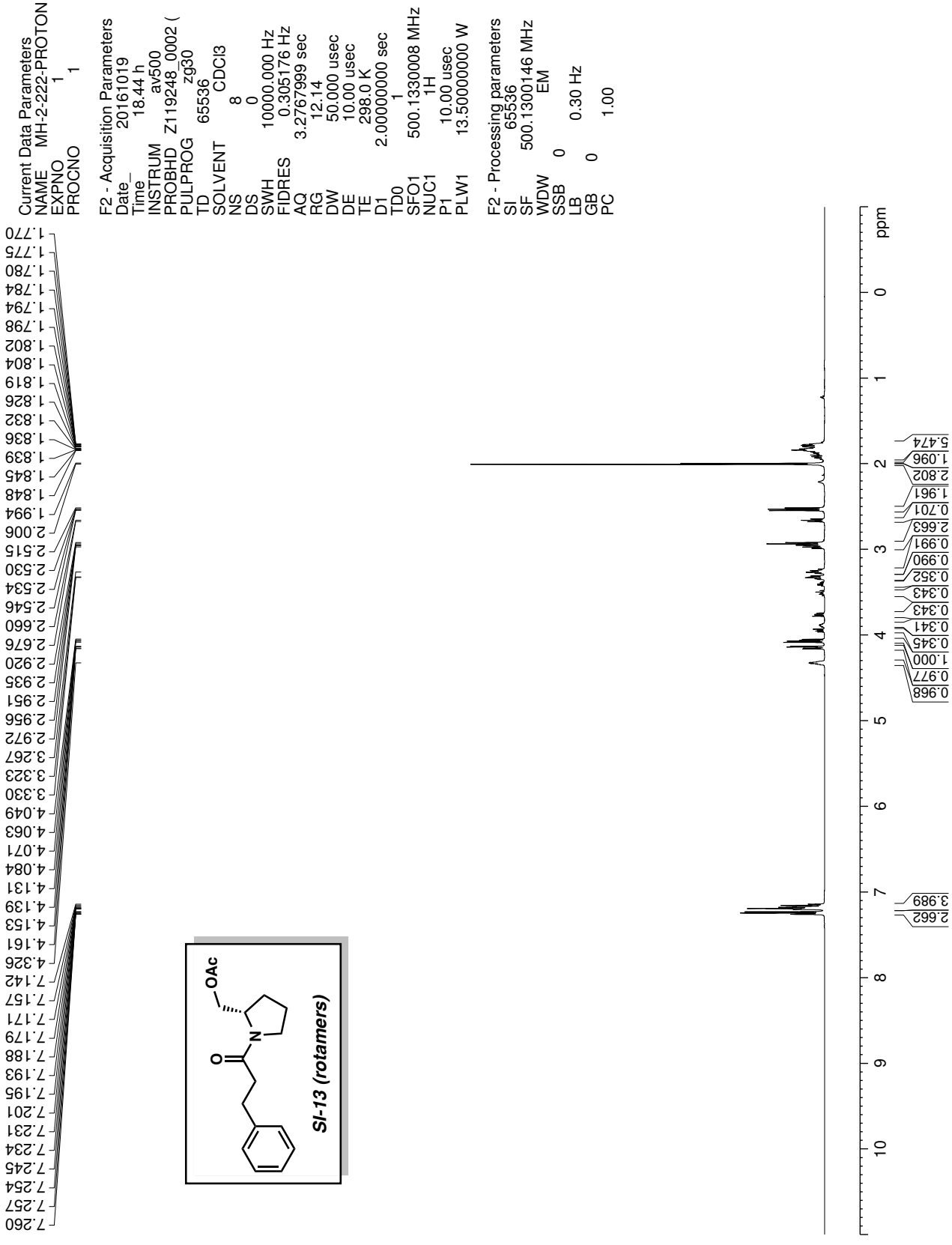
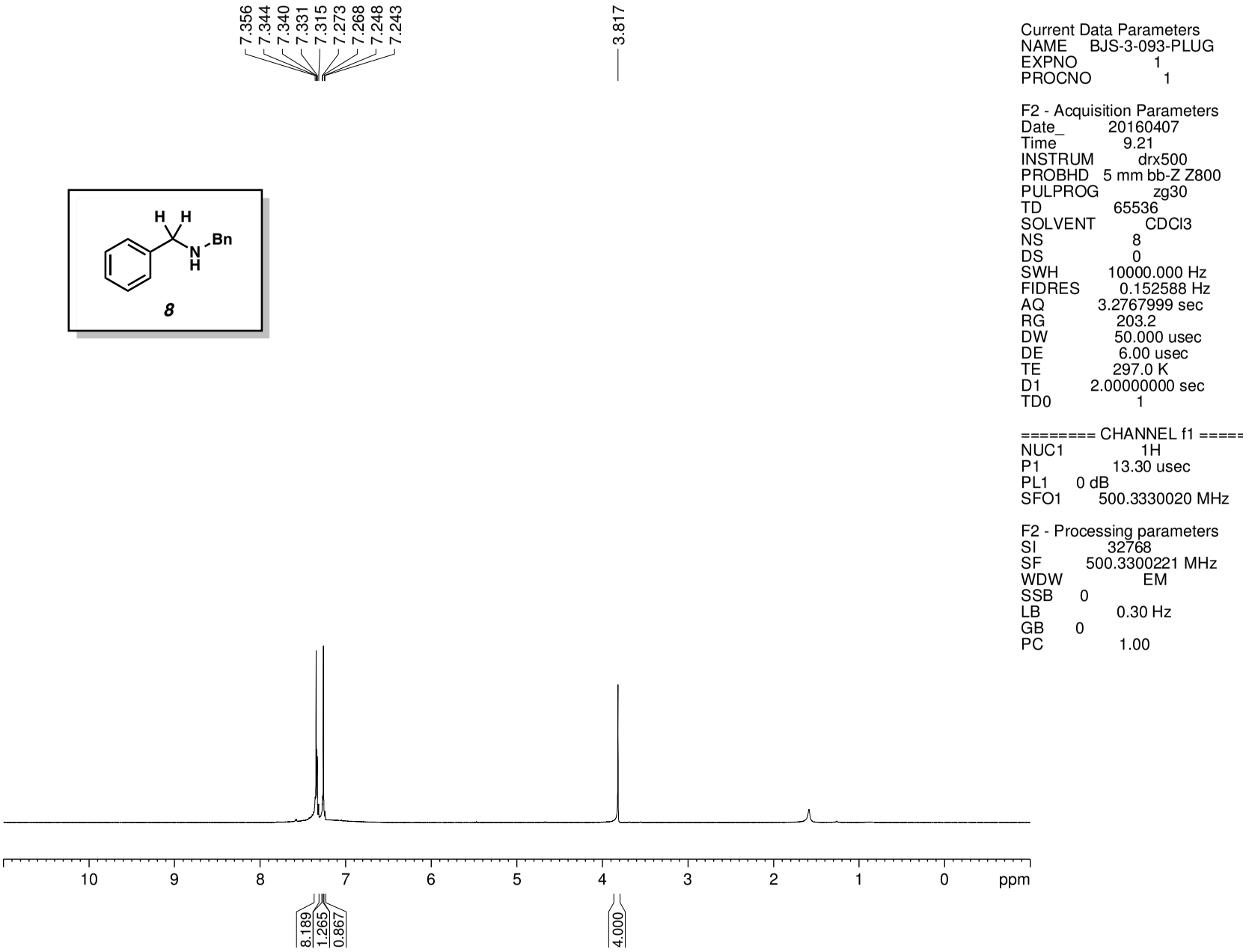
LB 0.30 Hz
GB 0
PC 1.00
7.359
7.356
7.353
7.346
7.343
7.340
7.330
7.327
7.301
7.287
7.277
7.274
5.706
4.462
4.451
4.040
4.036
4.033
4.027
4.017
4.012
4.009
4.004
3.435
3.430
3.412
3.407
3.389
3.384
2.398
2.390
2.381
2.376
2.367
2.359
2.354
2.345
2.336
2.044
1.881
1.872
1.858
1.854
1.849
1.845
1.831
1.823
1.809
O
N
H
Bn
O
SI-4
ppm
2.370
2.067
1.041
2.064
2.020
2.000
0.785
3.179
2.334

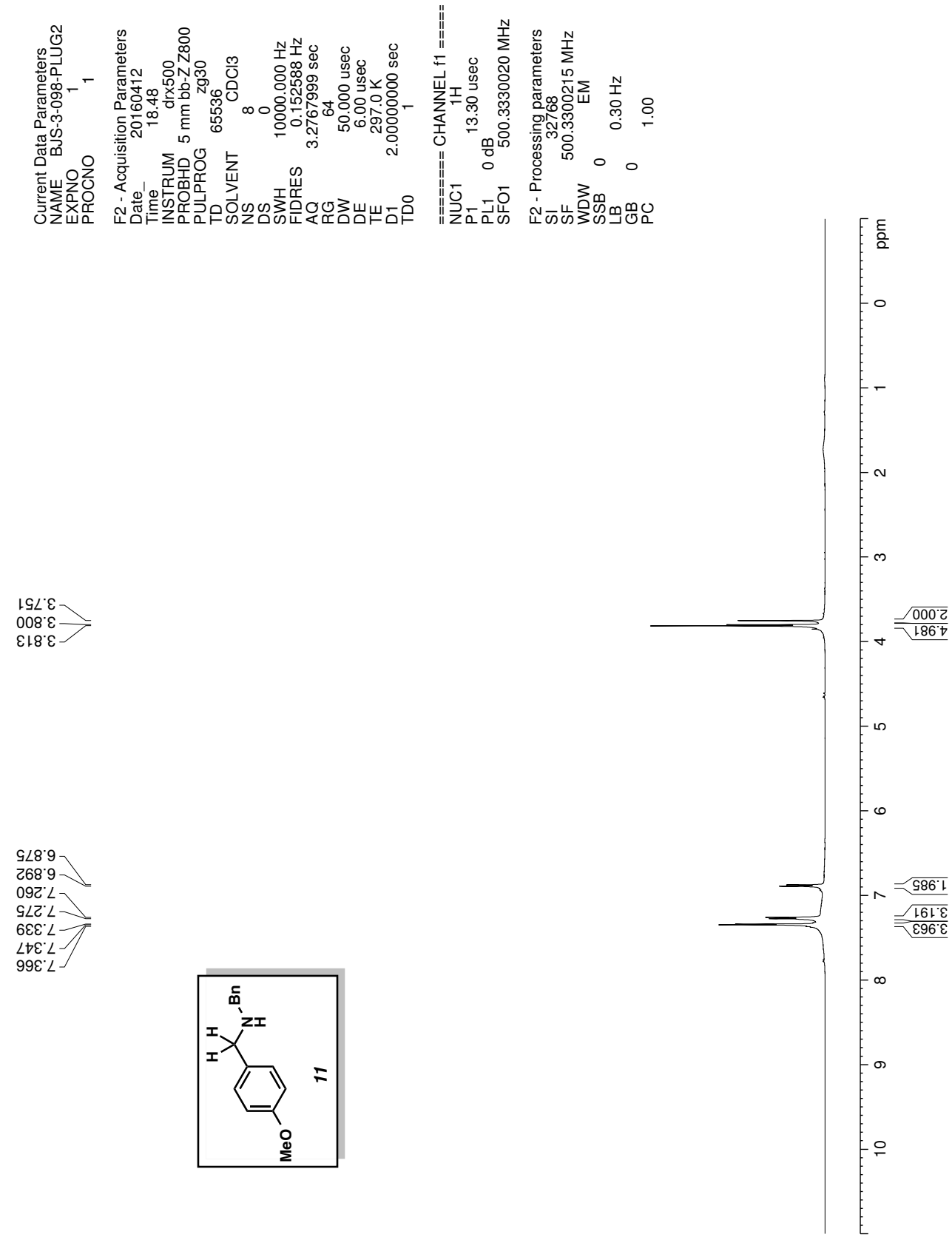

Current Data Parameters
NAME BJS-3-254-PROTON
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161023
Time 10.14 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 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 500.1300194 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
OMe
O
O
N
H
O
SI-7
7.290
7.287
7.275
7.260
7.208
7.206
7.193
7.185
7.183
7.169
6.840
6.823
6.706
6.689
5.949
5.934
4.819
4.807
4.803
4.796
4.792
4.780
3.692
3.015
3.004
2.987
2.976
2.953
2.941
2.925
2.912
2.899
2.882
2.530
2.516
2.514
2.500
2.486
2.484
2.479
2.470
2.463
2.447
2.433
2.417
2.020
2.994
2.021
2.000
0.952
1.005
2.976
1.002
0.943
2.140
2.036
ppm

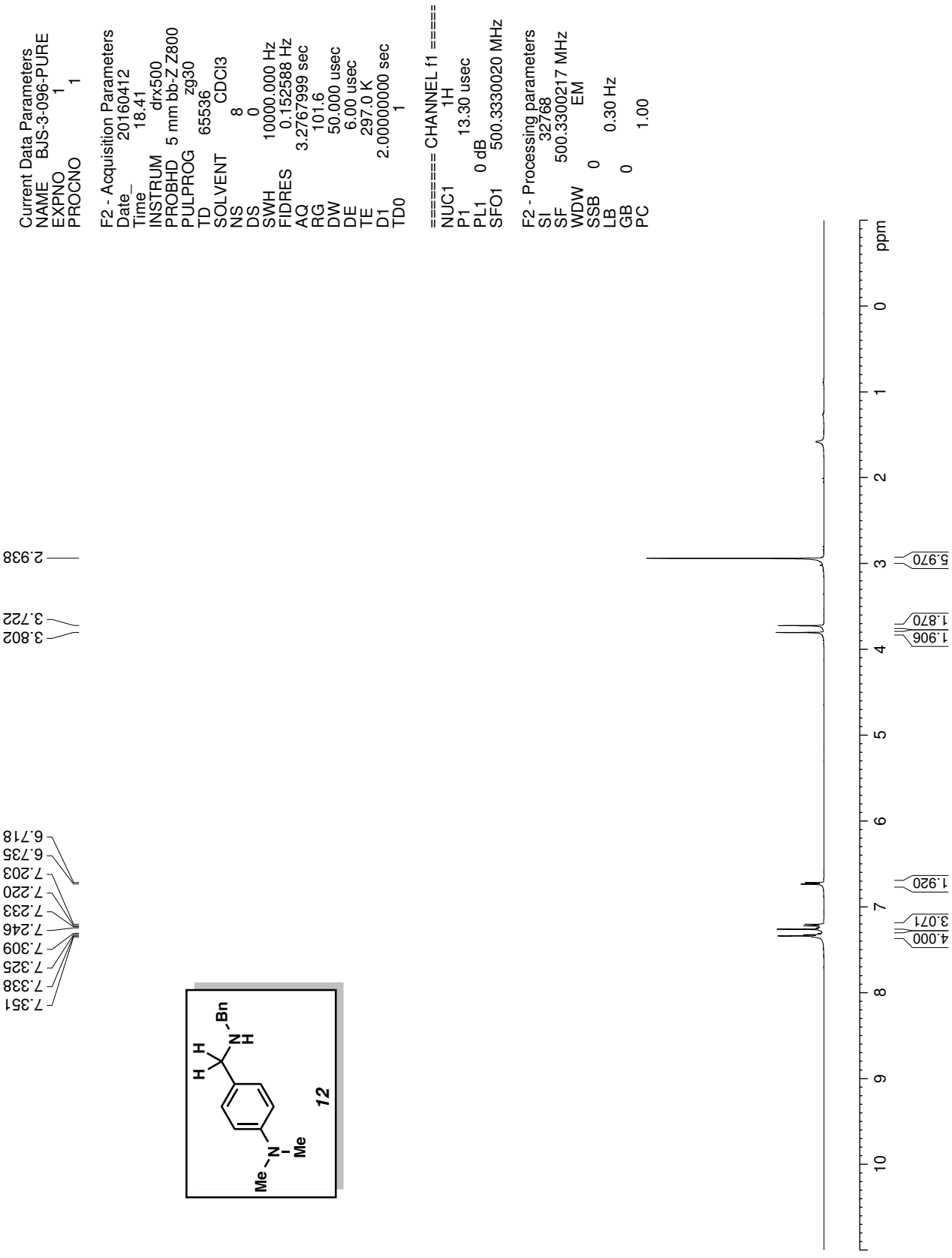

Current Data Parameters
NAME BJS-3-PYRAZOLESM
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160622
Time 16.36 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 23.34
DW 50.000 usec
DE 10.00 usec
TE 298.0 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
3.911
4.594
4.582
6.056
7.271
7.275
7.281
7.288
7.293
7.300
7.305
7.322
7.334
7.347
7.363
7.705
7.830
Bn
N
H
O
N
N
Me
SI-9
3.075
2.097
0.929
1.019
4.173
0.982
1.000
ppm

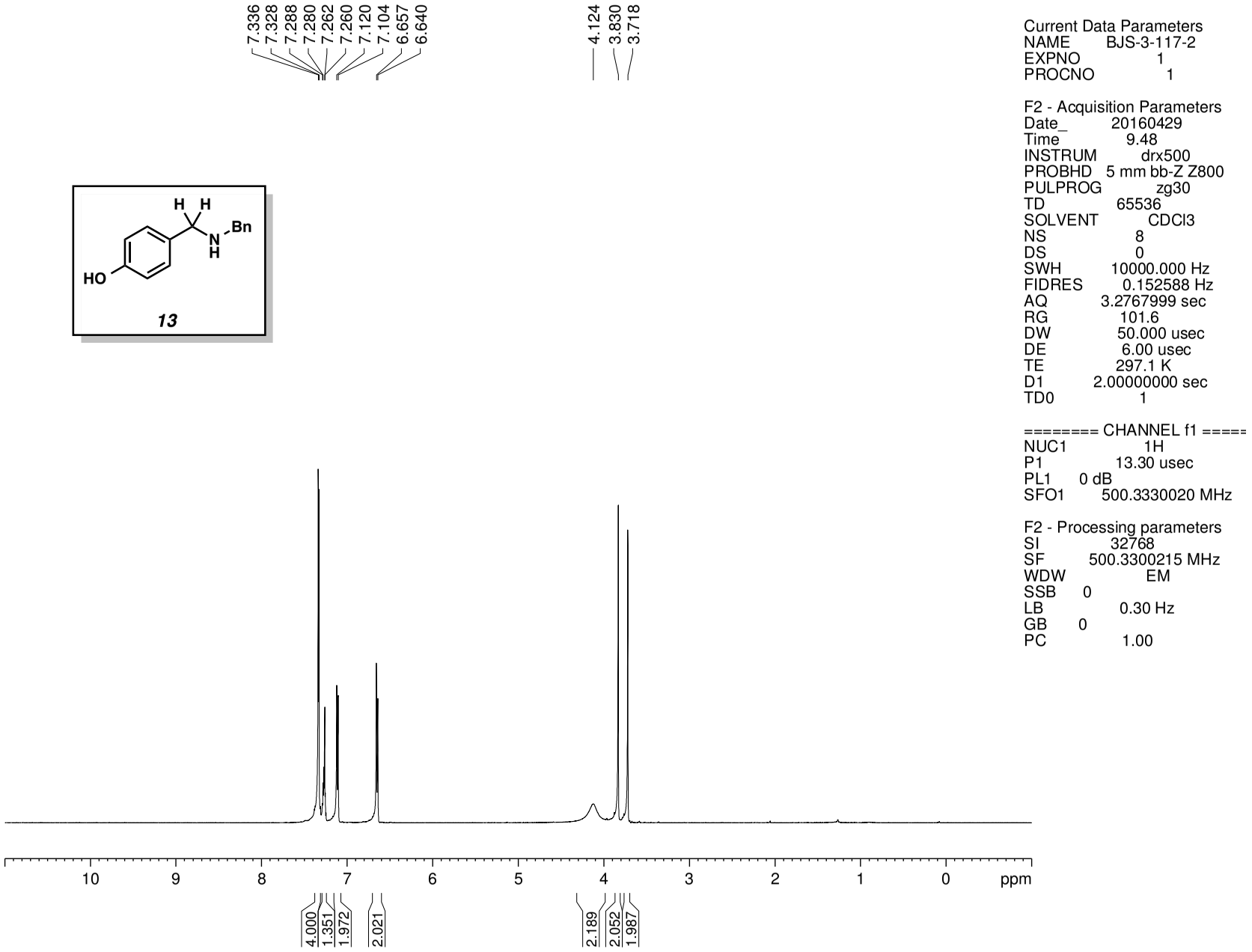

Current Data Parameters
NAME BJS-3-220-BENZENE
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160913
Time 16.07 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 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 500.1299959 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
O
N
H
Bn
Me
Me
SI-11
0.908
1.282
1.289
1.294
1.300
1.306
1.382
1.387
1.395
1.399
1.407
1.412
1.425
1.684
1.697
1.709
2.993
4.324
4.336
5.616
7.021
7.035
7.049
7.088
7.103
7.113
7.118
7.126
7.140
ppm
6.079
2.087
1.923
3.116
2.026
2.014
2.000
0.953
0.968
3.973


Current Data Parameters
NAME MH-2-222-PROTON
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161019
Time_ 18.44 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
OAc
O
N
SI-13 (rotamers)
ppm


7.356
7.344
7.340
7.331
7.315
7.273
7.268
7.248
7.243
3.817
H H
N
H
Bn
8
Current Data Parameters
NAME BJS-3-093-PLUG
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160407
Time 9.21
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 203.2
DW 50.000 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300221 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
8.189
1.265
0.867
4.000


Current Data Parameters
NAME BJS-3-098-PLUG2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160412
Time 18.48
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 64
DW 50.000 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300215 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.751
3.800
3.813
6.875
6.892
7.260
7.275
7.339
7.347
7.366
MeO
11
4.981
2.000
1.985
3.191
3.963
ppm


Current Data Parameters
NAME BJS-3-096-PURE
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160412
Time 18.41
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 101.6
DW 50.000 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300217 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.938
3.722
3.802
6.718
6.735
7.203
7.220
7.233
7.246
7.309
7.325
7.338
7.351
5.970
1.870
1.906
1.920
3.071
4.000
ppm
12

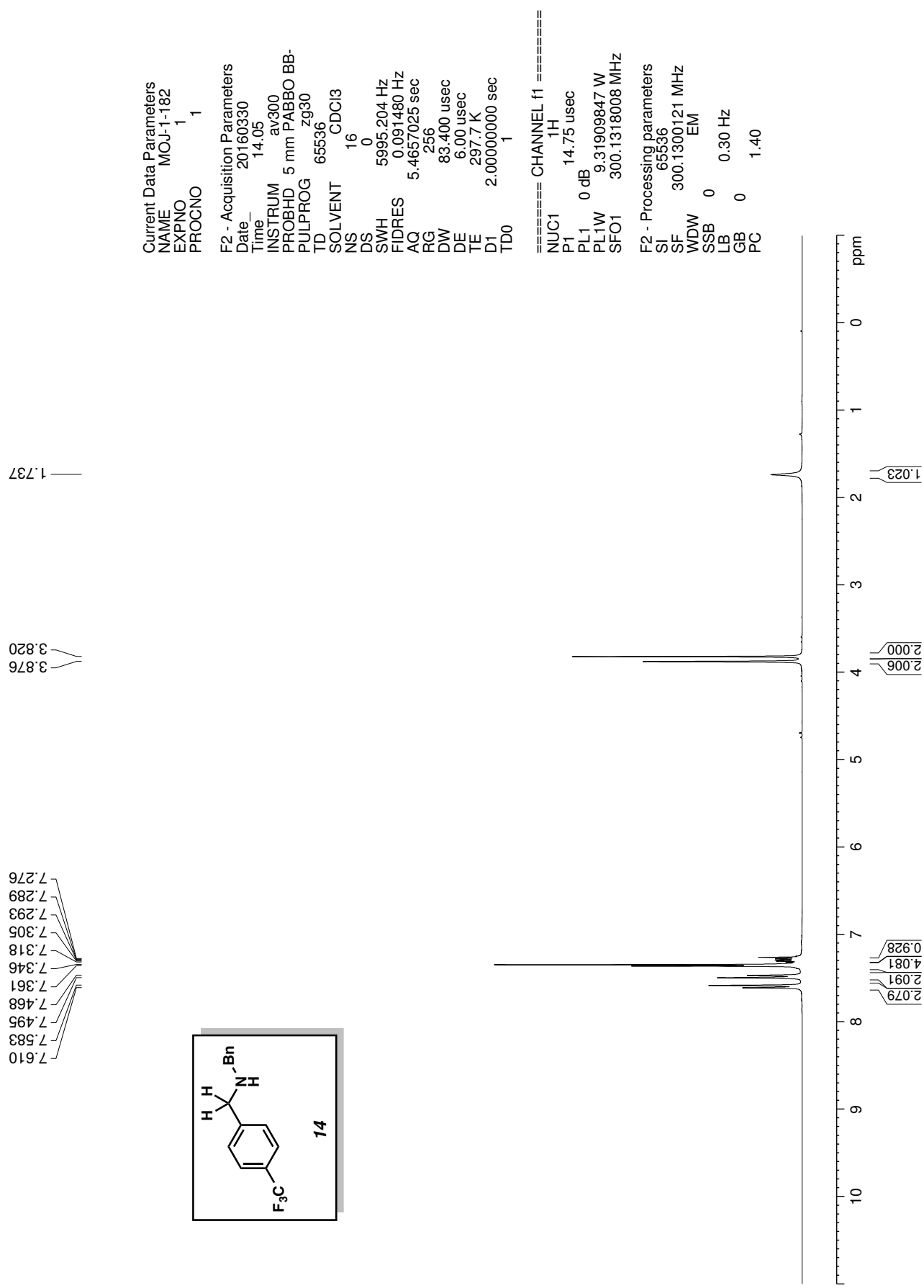


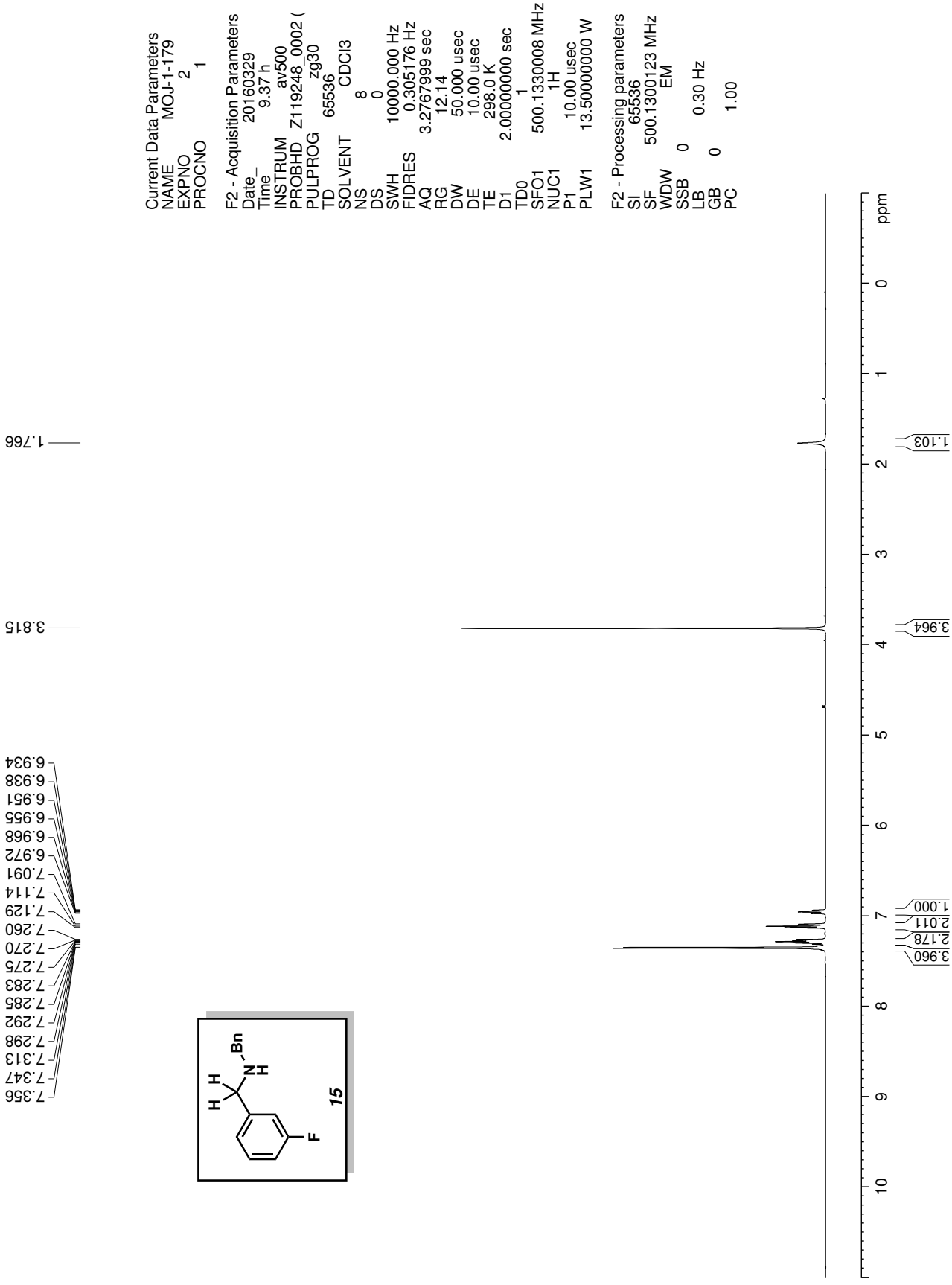
Current Data Parameters
NAME BJS-3-117-2
EXPNO 1
PROCNO 1

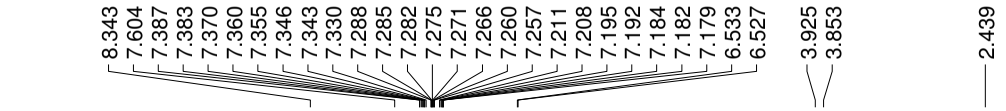
F2 - Acquisition Parameters
Date_ 20160429
Time 9.48
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 101.6
DW 50.000 usec
DE 6.00 usec
TE 297.1 K
D1 2.00000000 sec
TD0 1

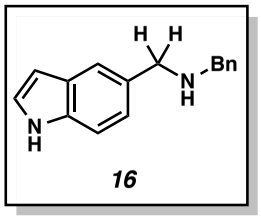
======== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz

F2 - Processing parameters
SI 32768
SF 500.3300215 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


Current Data Parameters
NAME MOJ-1-182
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160330
Time 14.05
INSTRUM av300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.091480 Hz
AQ 5.4657025 sec
RG 256
DW 83.400 usec
DE 6.00 usec
TE 297.7 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.75 usec
PL1 0 dB
PL1W 9.31909847 W
SFO1 300.1318008 MHz
F2 - Processing parameters
SI 65536
SF 300.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
7.610
7.583
7.495
7.468
7.361
7.346
7.318
7.305
7.293
7.289
7.276
3.876
3.820
1.737
2.079
2.091
4.081
0.928
2.000
2.006
1.023
ppm
14


Current Data Parameters
NAME MOJ-1-179
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160329
Time 9.37 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 500.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.766
3.815
7.356
7.347
7.313
7.298
7.292
7.285
7.283
7.275
7.270
7.260
7.129
7.114
7.091
6.972
6.968
6.955
6.951
6.938
6.934
Bn
15
ppm
1.103
3.964
3.960
2.178
2.011
1.000



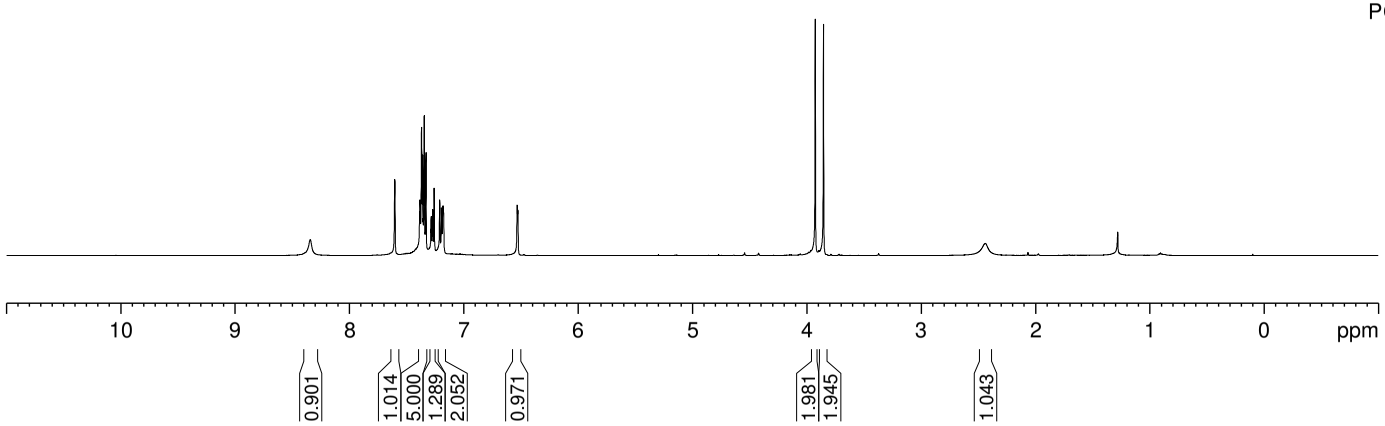


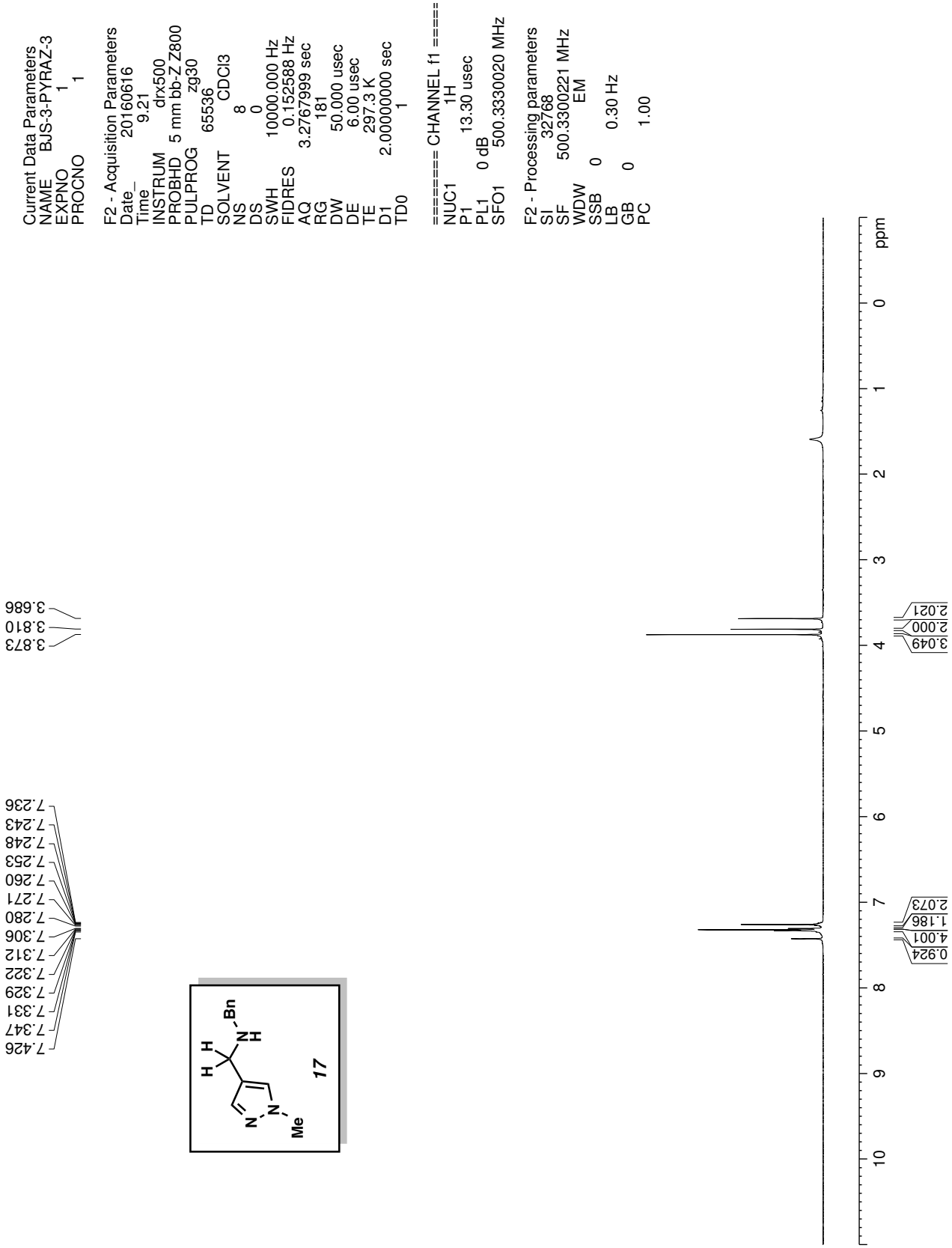
Current Data Parameters
NAME BJS-3-144-PREP3
EXPNO 1
PROCNO 1

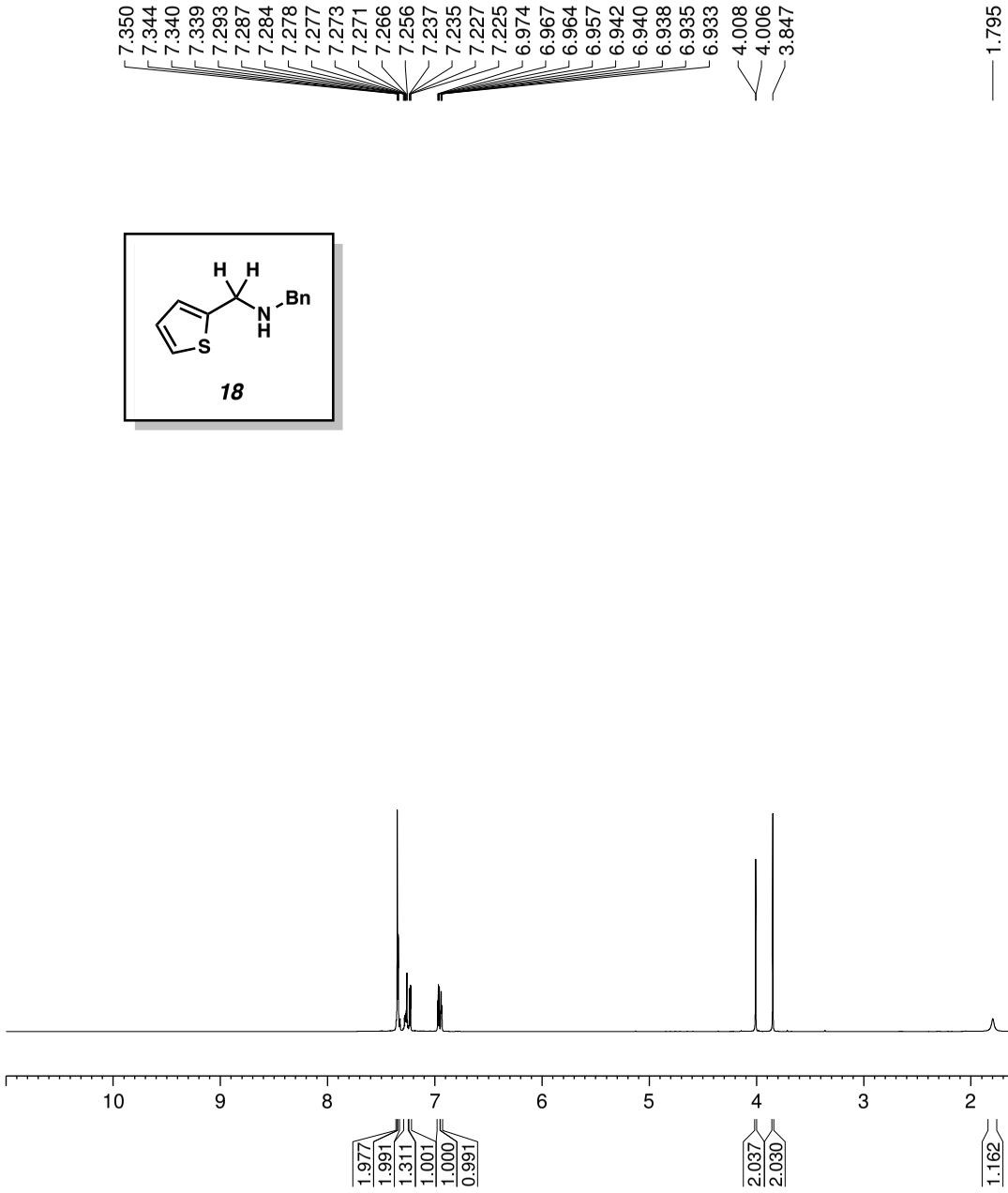
F2 - Acquisition Parameters
Date_ 20160518
Time 9.06
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 101.6
DW 50.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

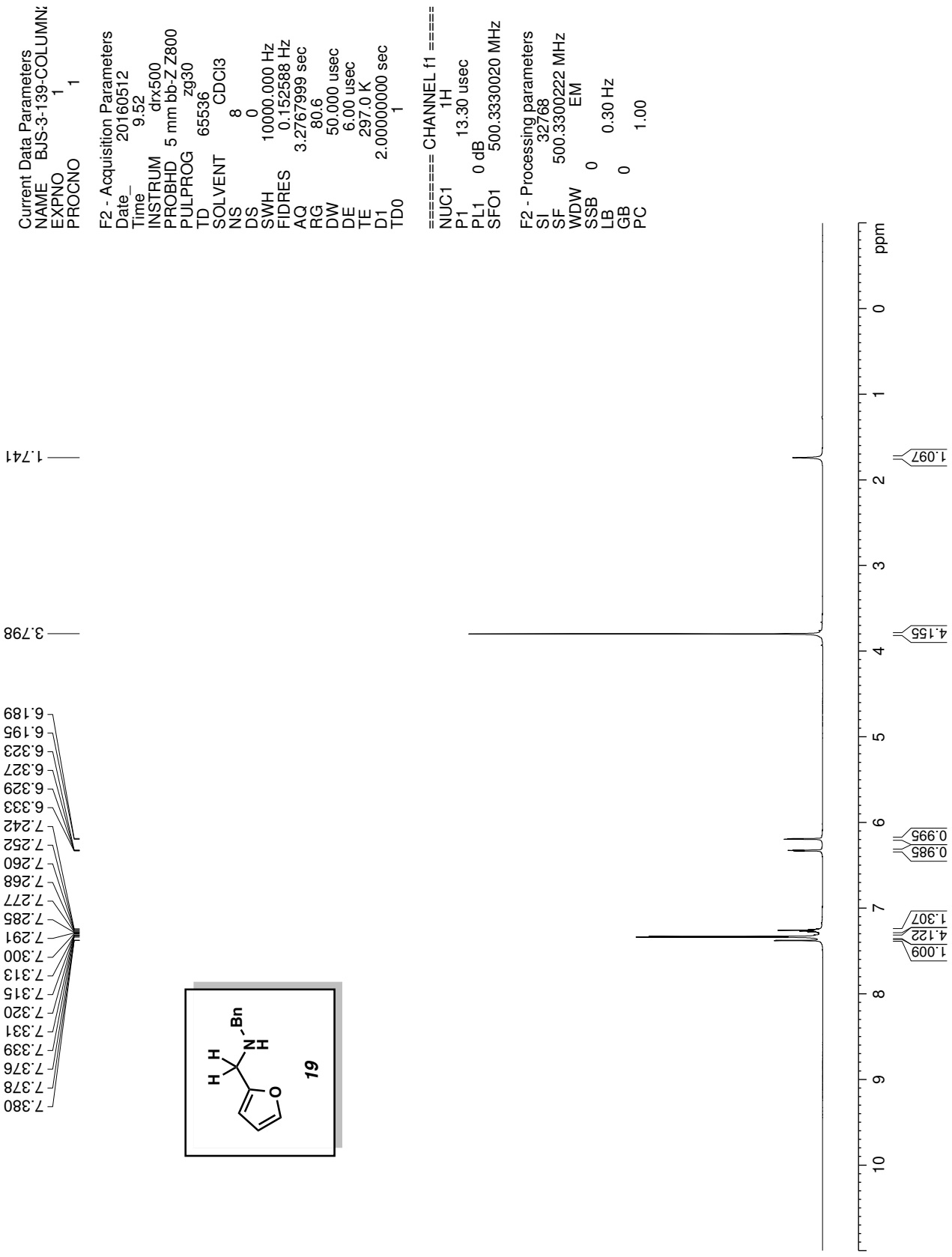
======== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz

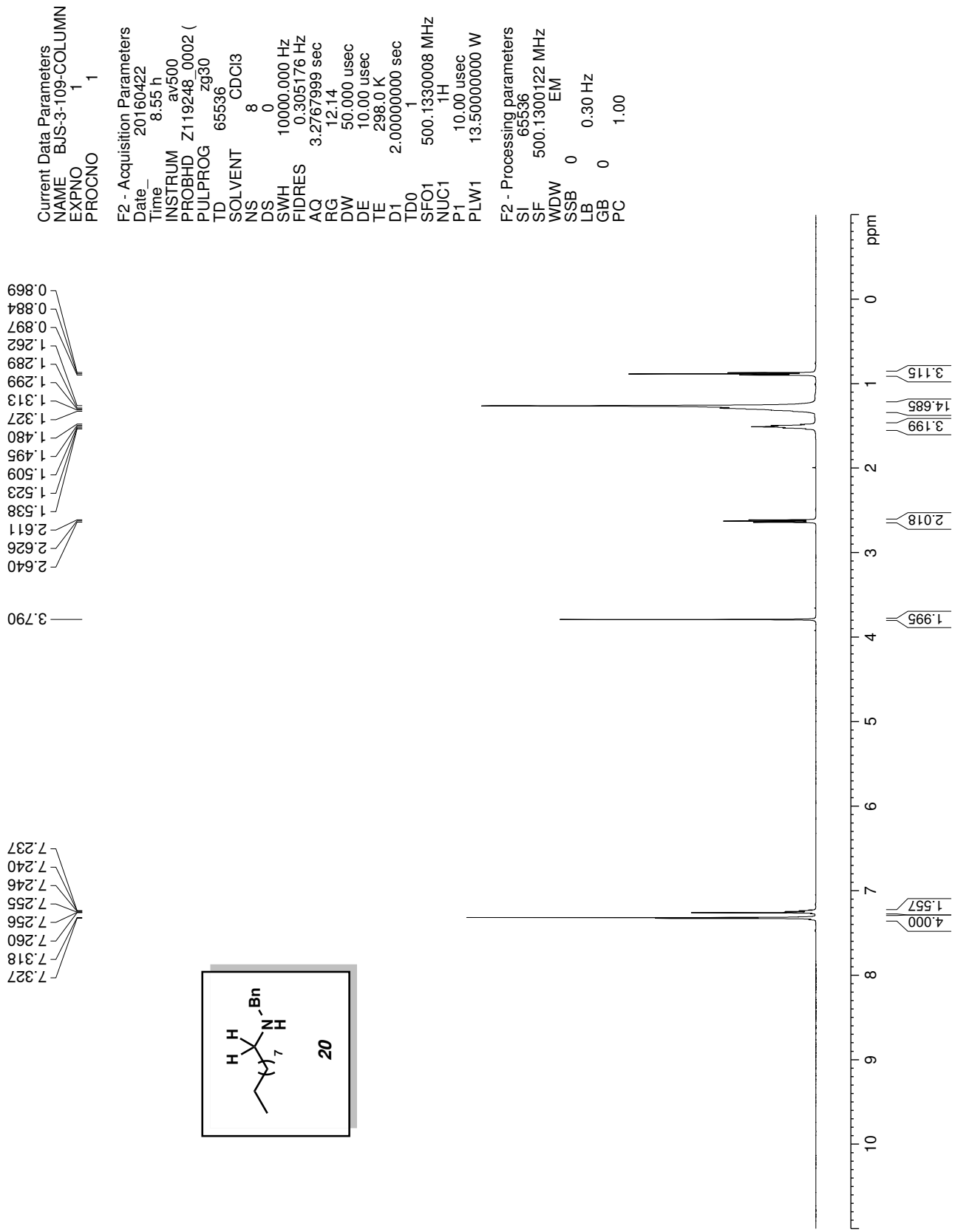
F2 - Processing parameters
SI 32768
SF 500.3300219 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

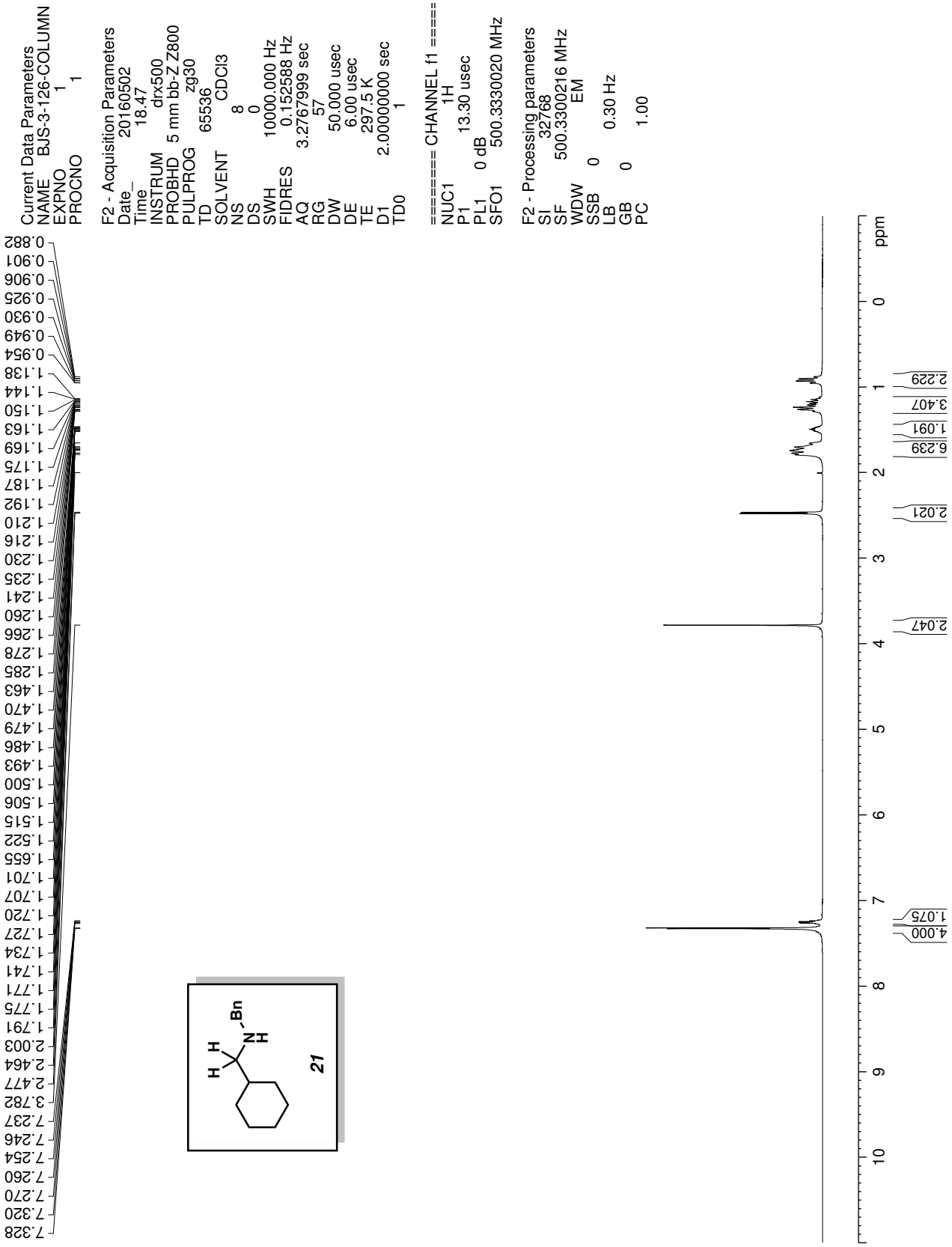


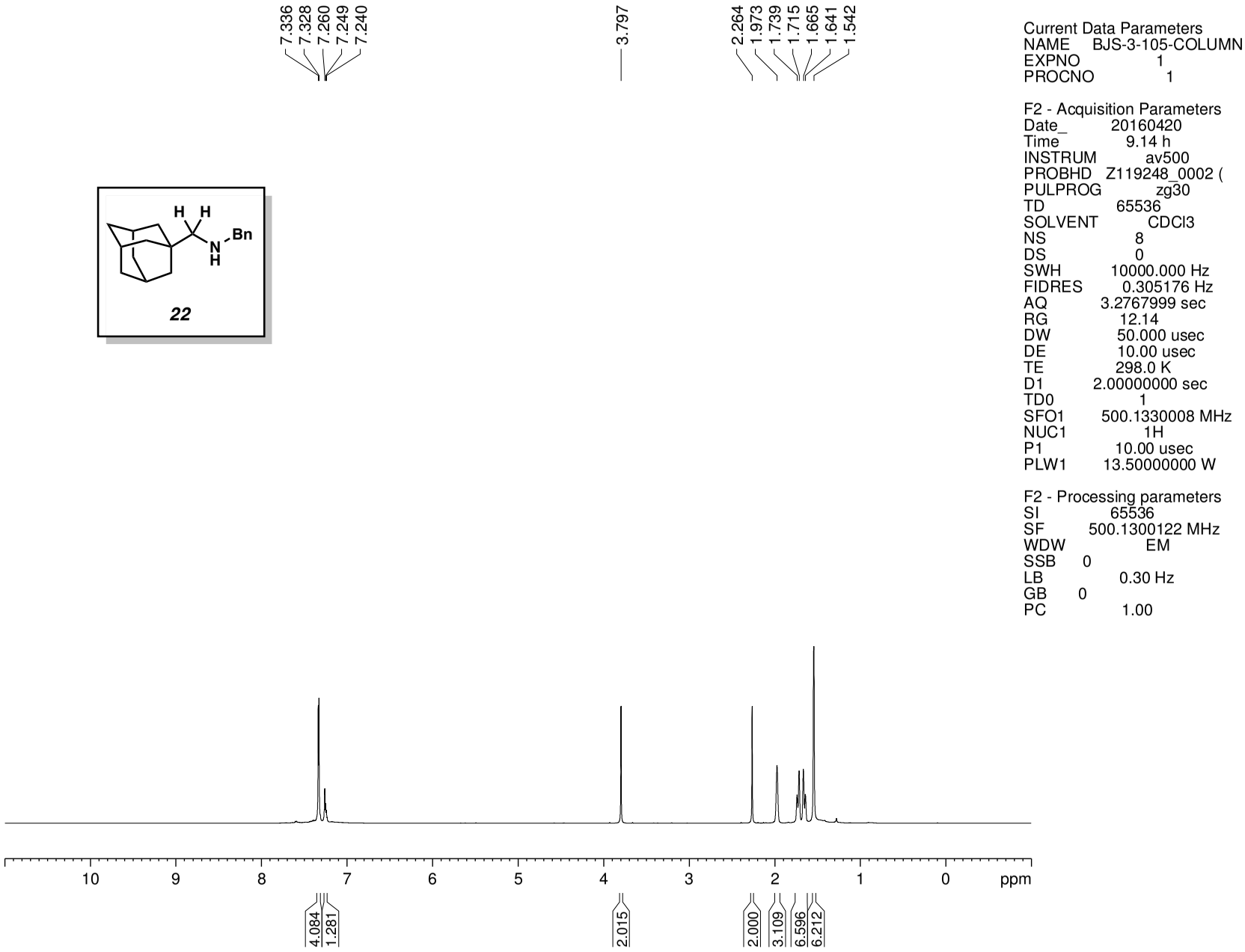

Current Data Parameters
NAME BJS-3-PYRAZ-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160616
Time 9.21
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 297.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300221 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.873
3.810
3.686
7.426
7.347
7.331
7.329
7.322
7.312
7.306
7.280
7.271
7.260
7.253
7.248
7.243
7.236
Bn
H
H
N
N
Me
17
3.049
2.000
2.021
0.924
4.001
1.186
2.073
ppm


7.350
7.344
7.340
7.339
7.293
7.287
7.284
7.278
7.277
7.273
7.271
7.266
7.256
7.237
7.235
7.227
7.225
6.974
6.967
6.964
6.957
6.942
6.940
6.938
6.935
6.933
4.008
4.006
3.847
1.795
H H
N
H
Bn
S
18
Current Data Parameters
NAME BJS-3-141-COLUMN
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160515
Time 9.16 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
10
9
8
7
6
5
4
3
2
1
0
ppm
1.977
1.991
1.311
1.001
1.000
0.991
2.037
2.030
1.162


Current Data Parameters
NAME BJS-3-139-COLUMN2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160512
Time 9.52
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 80.6
DW 50.000 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300222 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.741
3.798
6.189
6.195
6.323
6.327
6.329
6.333
7.242
7.252
7.260
7.268
7.277
7.285
7.291
7.300
7.313
7.315
7.320
7.331
7.339
7.376
7.378
7.380
Bn
N
H
H
O
19
1.097
4.155
0.985
0.995
1.009
4.122
1.307
ppm


Current Data Parameters
NAME BJS-3-109-COLUMN
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160422
Time_ 8.55 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
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.869
0.884
0.897
1.262
1.289
1.299
1.313
1.327
1.480
1.495
1.509
1.523
1.538
2.611
2.626
2.640
3.790
7.237
7.240
7.246
7.255
7.256
7.260
7.318
7.327
H H
N H
Bn
7
20
ppm
0
1
2
3
4
5
6
7
8
9
10
3.115
14.685
3.199
2.018
1.995
1.557
4.000

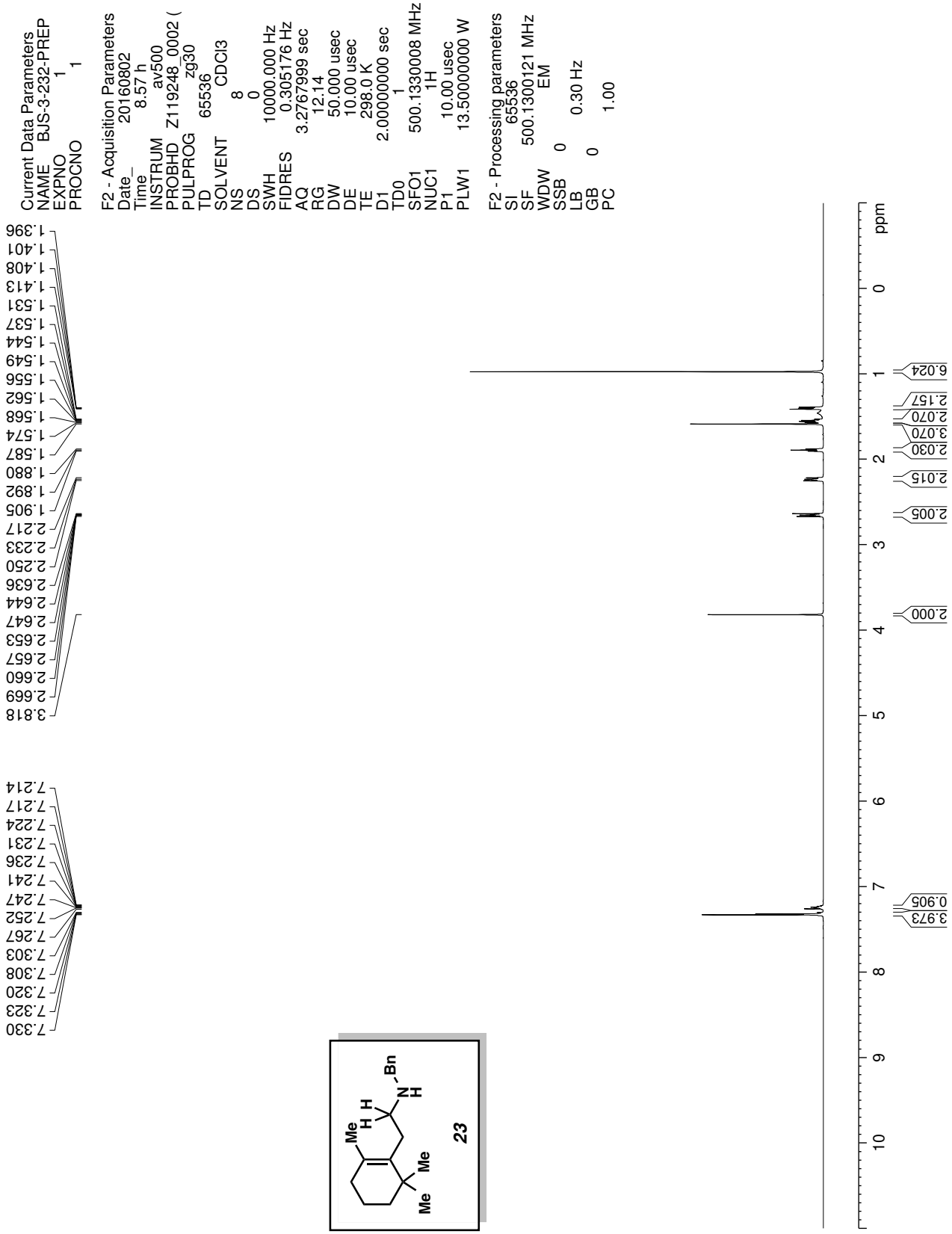

Current Data Parameters
NAME BJS-3-126-COLUMN
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160502
Time 18.47
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 57
DW 50.000 usec
DE 6.00 usec
TE 297.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300216 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Bn
N
H
H
H
21
4.000
1.075
2.047
2.021
6.239
1.091
3.407
2.229
ppm

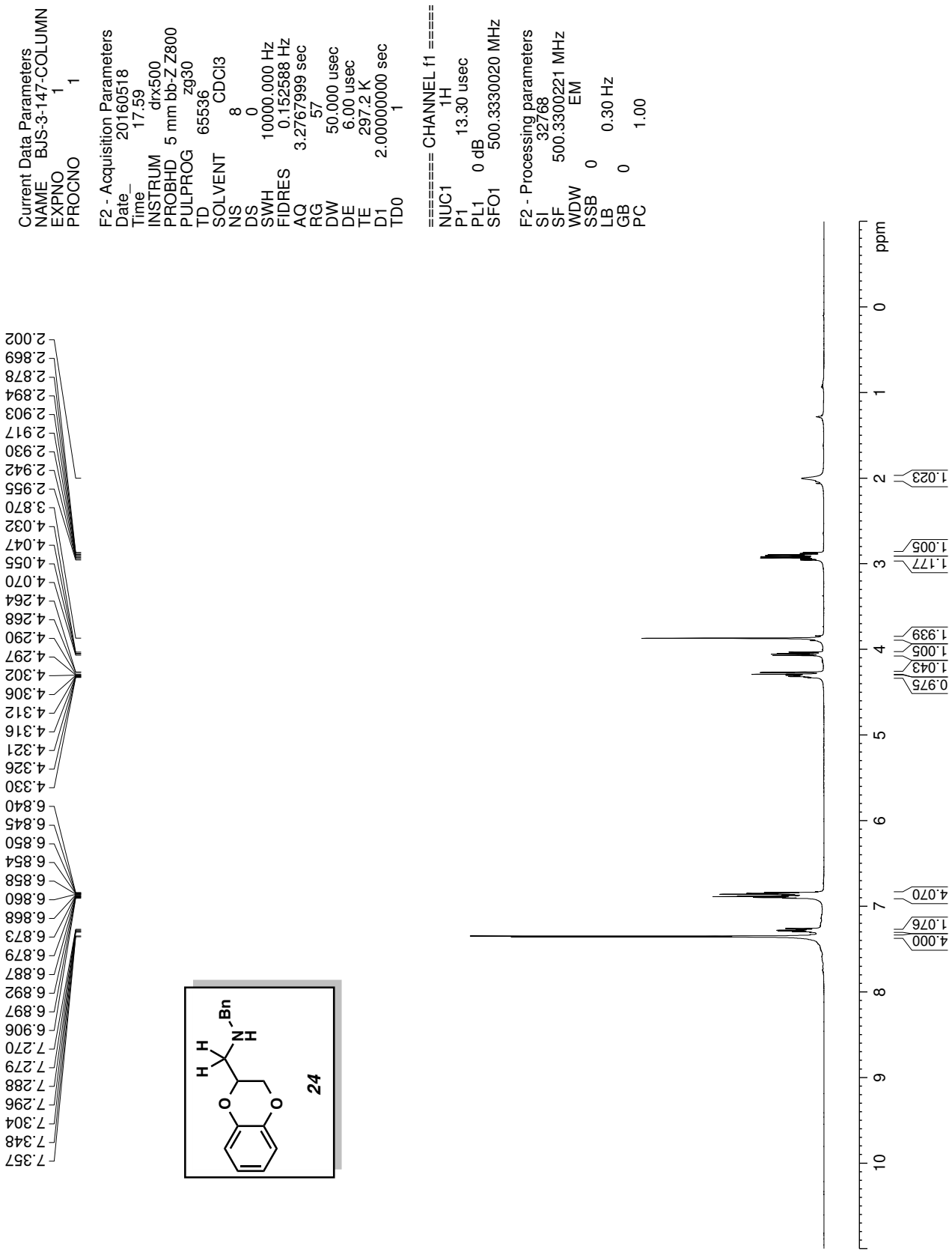


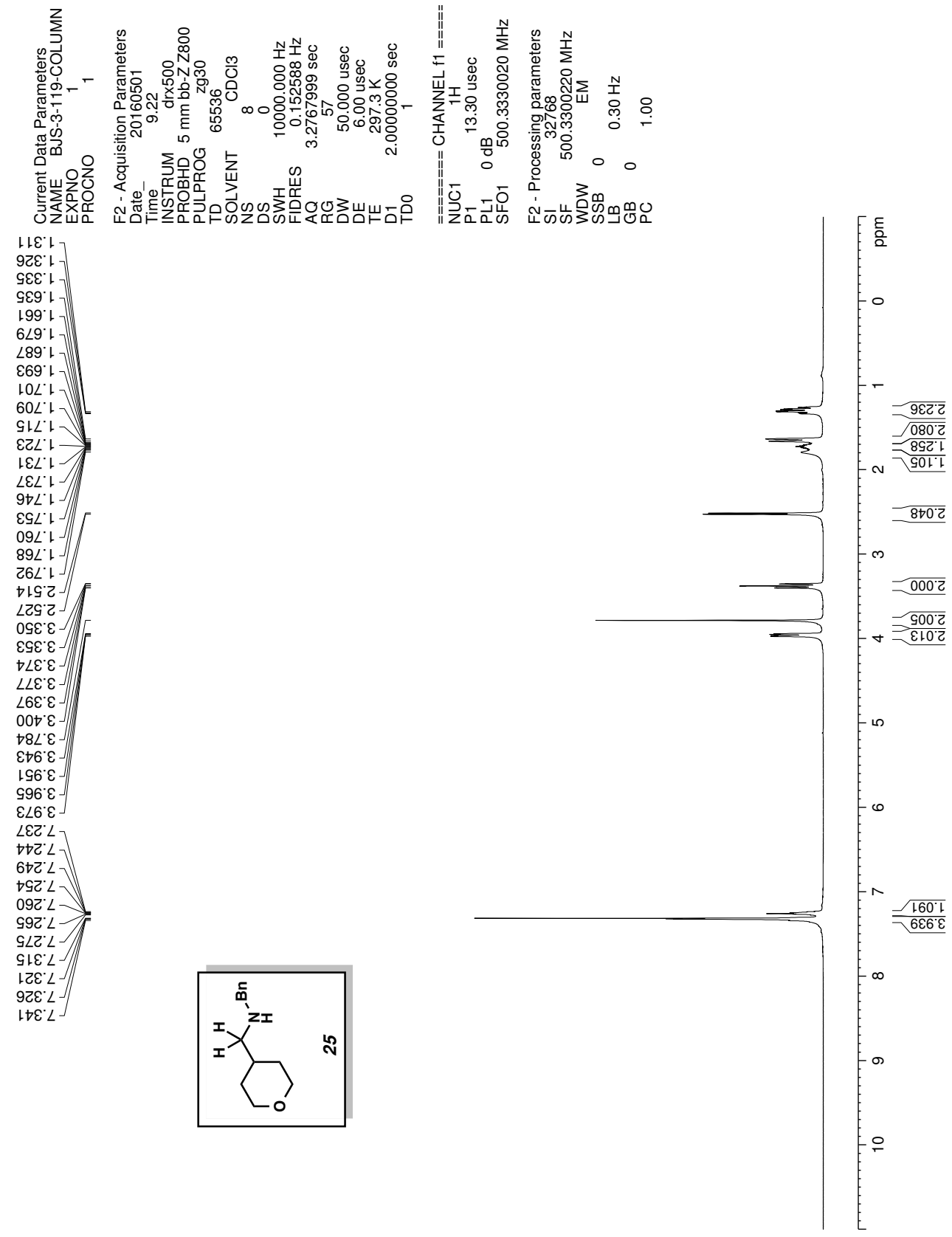
Current Data Parameters
NAME BJS-3-105-COLUMN
EXPNO 1
PROCNO 1

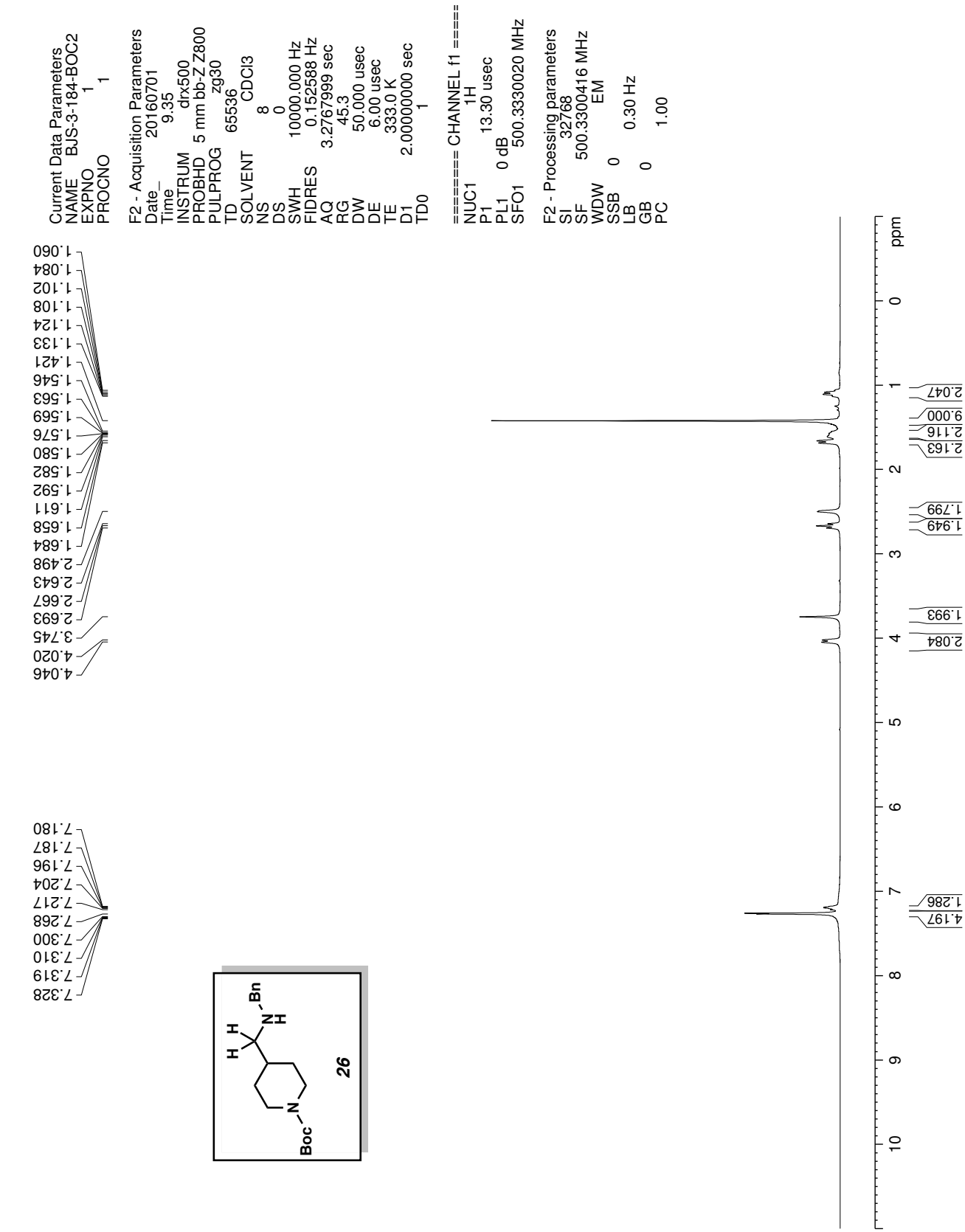
F2 - Acquisition Parameters
Date_ 20160420
Time 9.14 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W

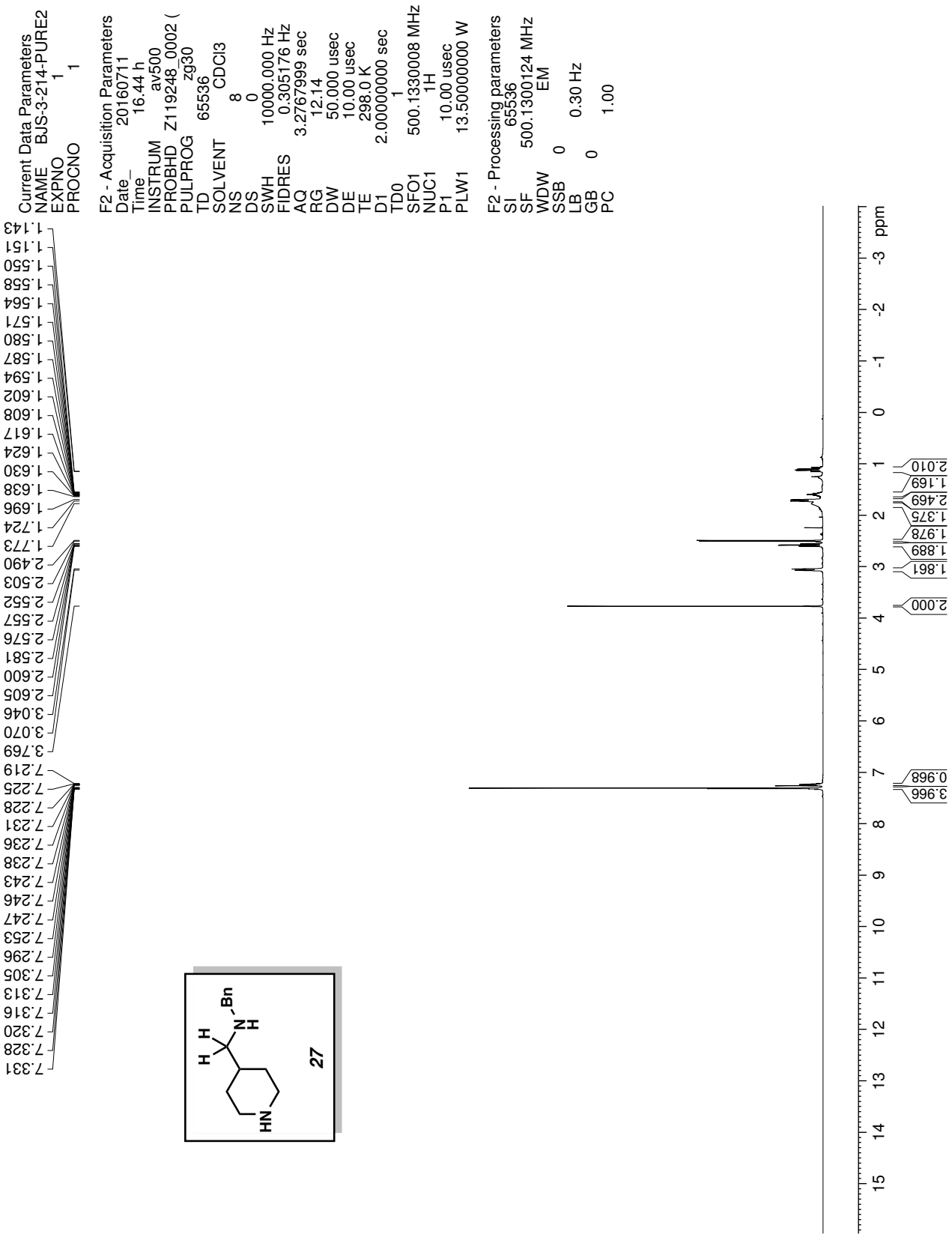
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

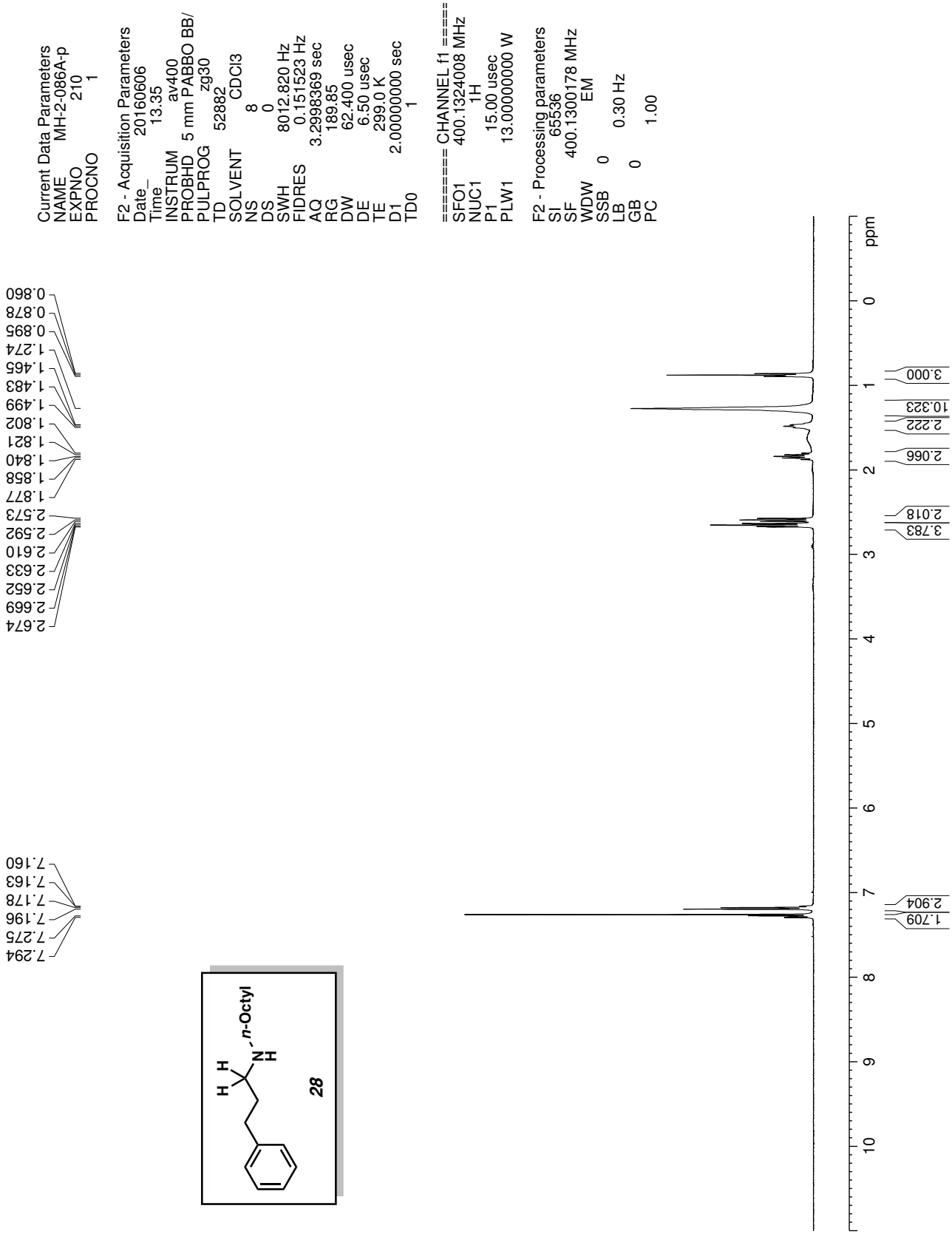

Current Data Parameters
NAME BJS-3-232-PREP
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160802
Time 8.57 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 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
H H
N
H
Bn
Me
Me
23
ppm
6.024
2.157
2.070
3.070
2.030
2.015
2.005
2.000
3.973
0.905

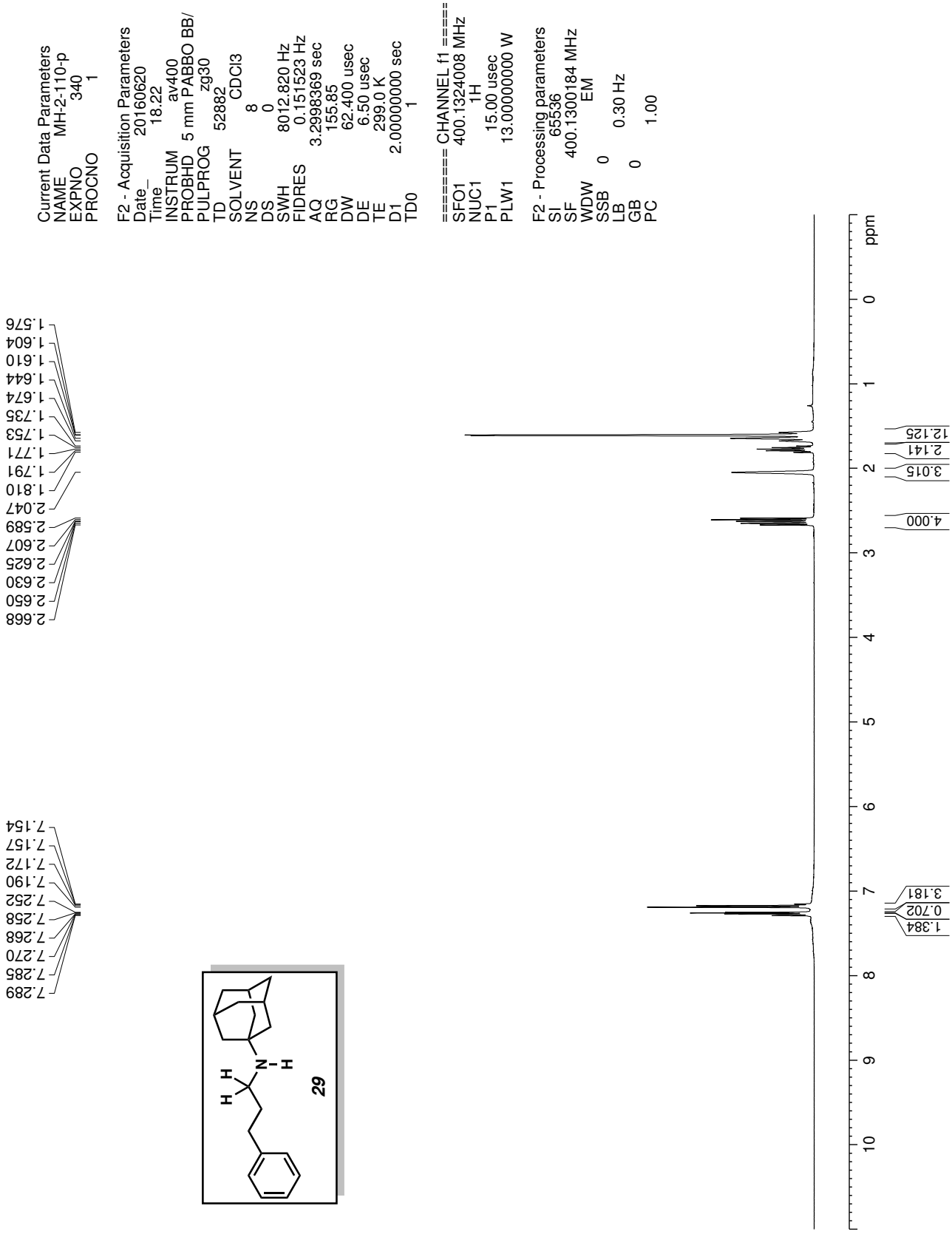

Current Data Parameters
NAME BJS-3-147-COLUMN
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160518
Time 17.59
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 57
DW 50.000 usec
DE 6.00 usec
TE 297.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300221 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
24
ppm

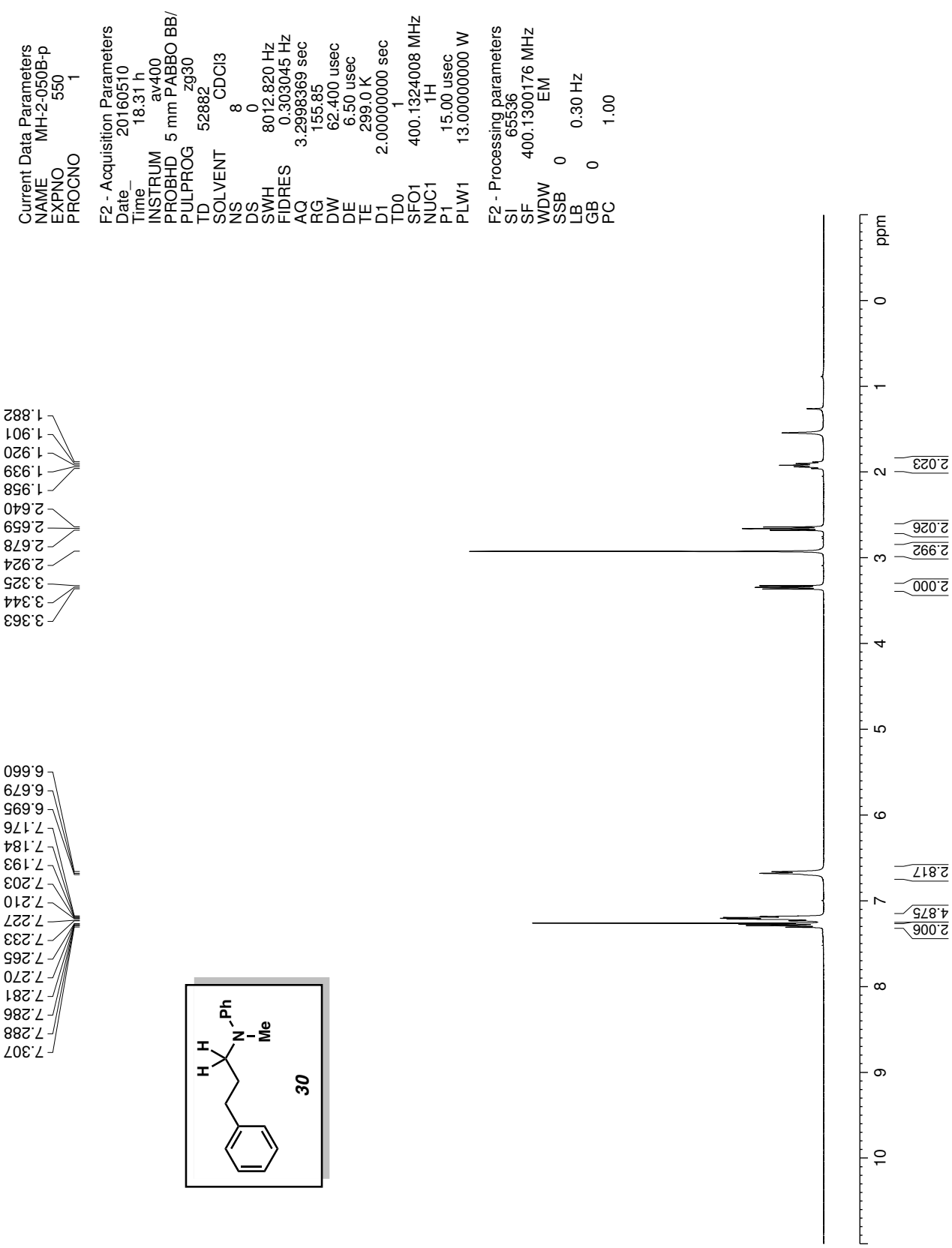

Current Data Parameters
NAME BJS-3-119-COLUMN
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160501
Time 9.22
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 57
DW 50.000 usec
DE 6.00 usec
TE 297.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.311
1.326
1.335
1.635
1.661
1.679
1.687
1.693
1.701
1.709
1.715
1.723
1.731
1.737
1.746
1.753
1.760
1.768
1.792
2.514
2.527
3.350
3.353
3.374
3.377
3.397
3.400
3.784
3.943
3.951
3.965
3.973
7.237
7.244
7.249
7.254
7.260
7.265
7.275
7.315
7.321
7.326
7.341
Bn
N
H
H
H
O
25
ppm
0
1
2
3
4
5
6
7
8
9
10
3.939
1.091
2.013
2.005
2.000
2.048
1.105
1.258
2.080
2.236

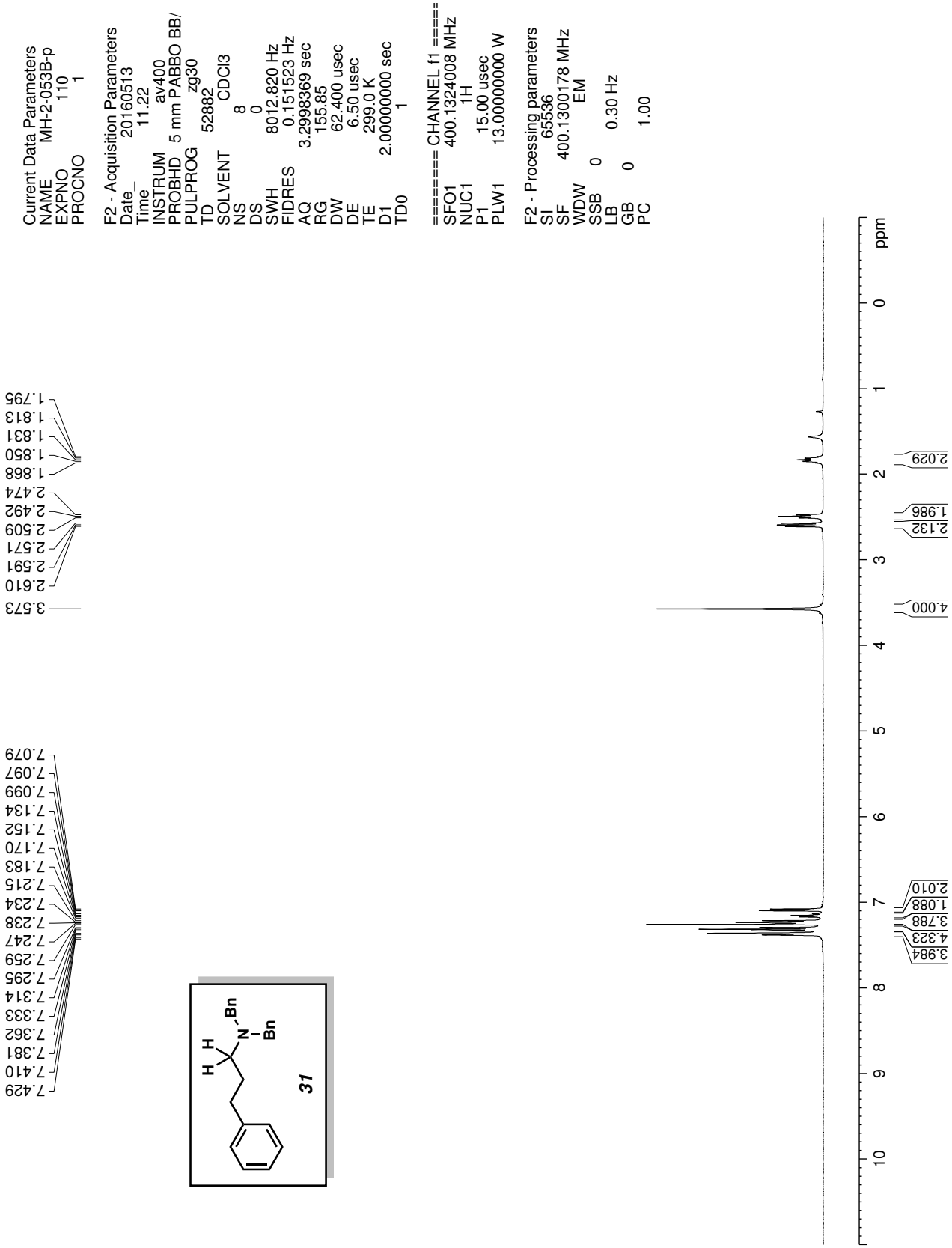

Current Data Parameters
NAME BJS-3-184-BOC2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160701
Time 9.35
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 45.3
DW 50.000 usec
DE 6.00 usec
TE 333.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300416 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.046
4.020
3.745
2.693
2.667
2.643
2.498
1.684
1.658
1.611
1.592
1.582
1.580
1.576
1.569
1.563
1.546
1.421
1.133
1.124
1.108
1.102
1.084
1.060
7.328
7.319
7.310
7.300
7.268
7.217
7.204
7.196
7.187
7.180
Boc
N
H
H
N
Bn
26
4.197
1.286
2.084
1.993
1.949
1.799
2.163
2.116
9.000
2.047
ppm

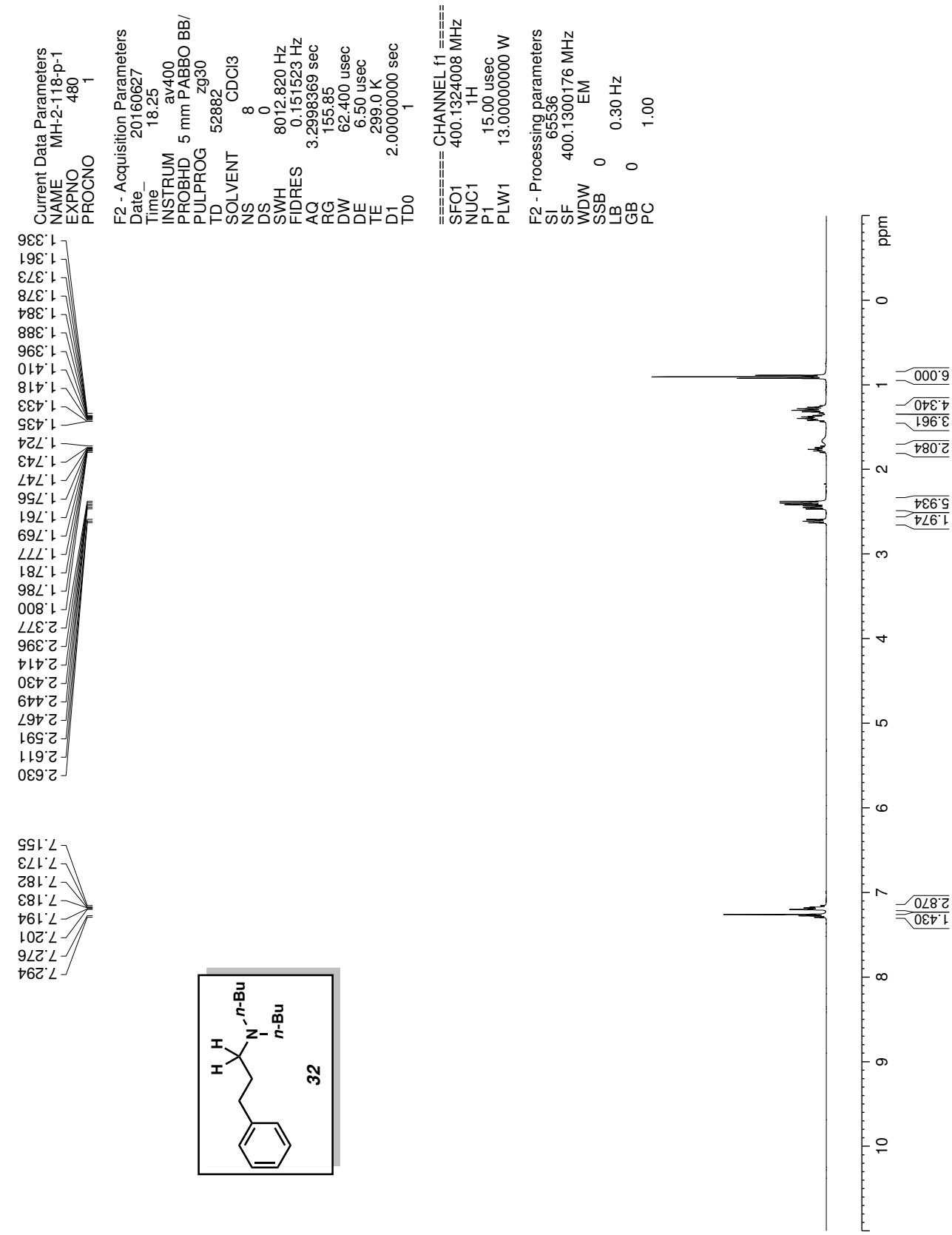

Current Data Parameters
NAME BJS-3-214-PURE2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160711
Time 16.44 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
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
7.331
7.328
7.320
7.316
7.313
7.305
7.296
7.253
7.247
7.246
7.243
7.238
7.236
7.231
7.228
7.225
7.219
3.769
3.070
3.046
2.605
2.600
2.581
2.576
2.557
2.552
2.503
2.490
1.773
1.724
1.696
1.638
1.630
1.624
1.617
1.608
1.602
1.594
1.587
1.580
1.571
1.564
1.558
1.550
1.151
1.143
Bn
N
H
H
H
HN
27
3.966
0.968
2.000
1.861
1.889
1.978
1.375
2.469
1.169
2.010
ppm

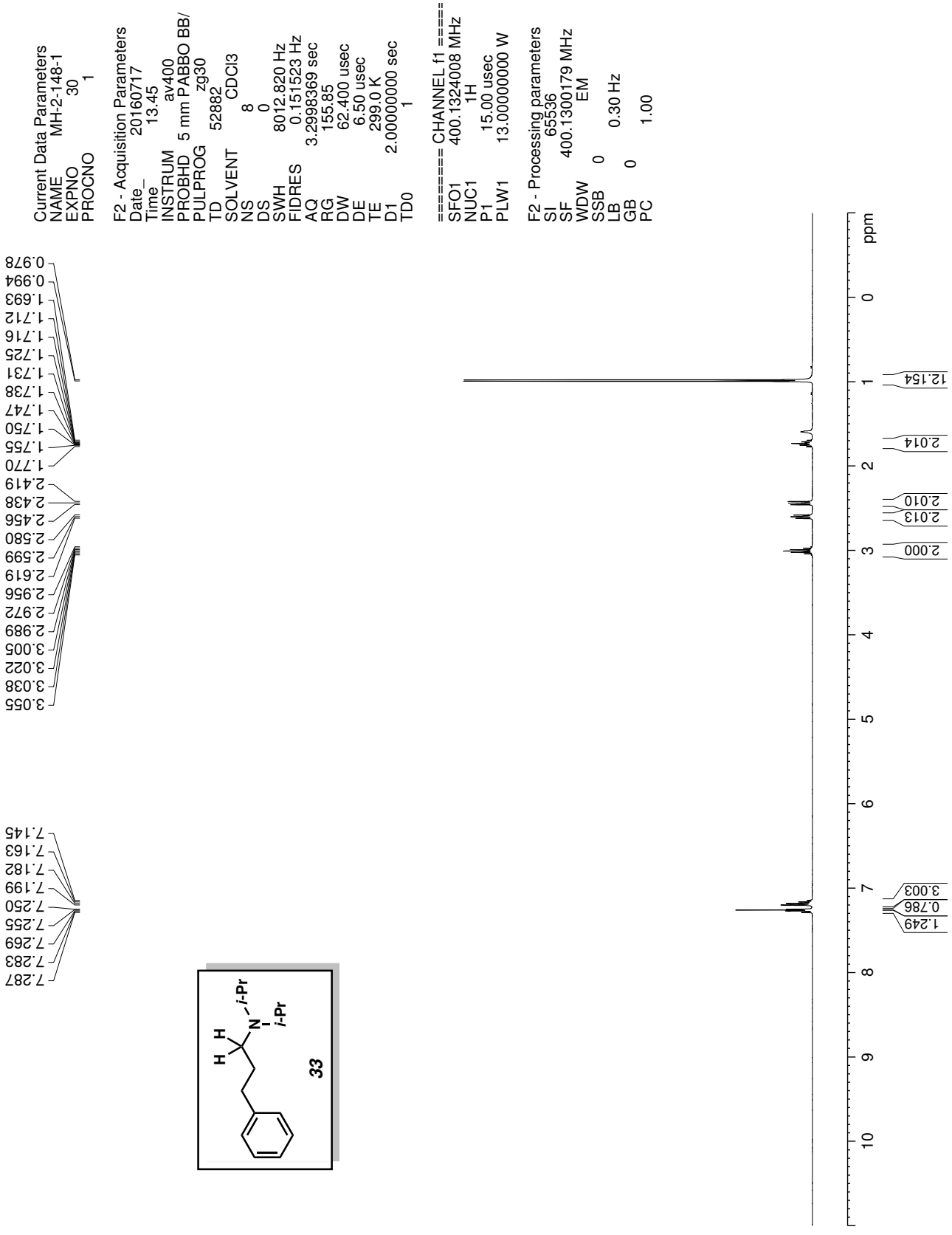

Current Data Parameters
NAME MH-2-086A-p
EXPNO 210
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160606
Time 13.35
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 189.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ======
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300178 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.294
7.275
7.196
7.178
7.163
7.160
2.674
2.669
2.652
2.633
2.610
2.592
2.573
1.877
1.858
1.840
1.821
1.802
1.499
1.483
1.465
1.274
0.895
0.878
0.860
H H
N
H
n-Octyl
28
1.709
2.904
2.018
3.783
2.066
2.222
10.323
3.000
ppm

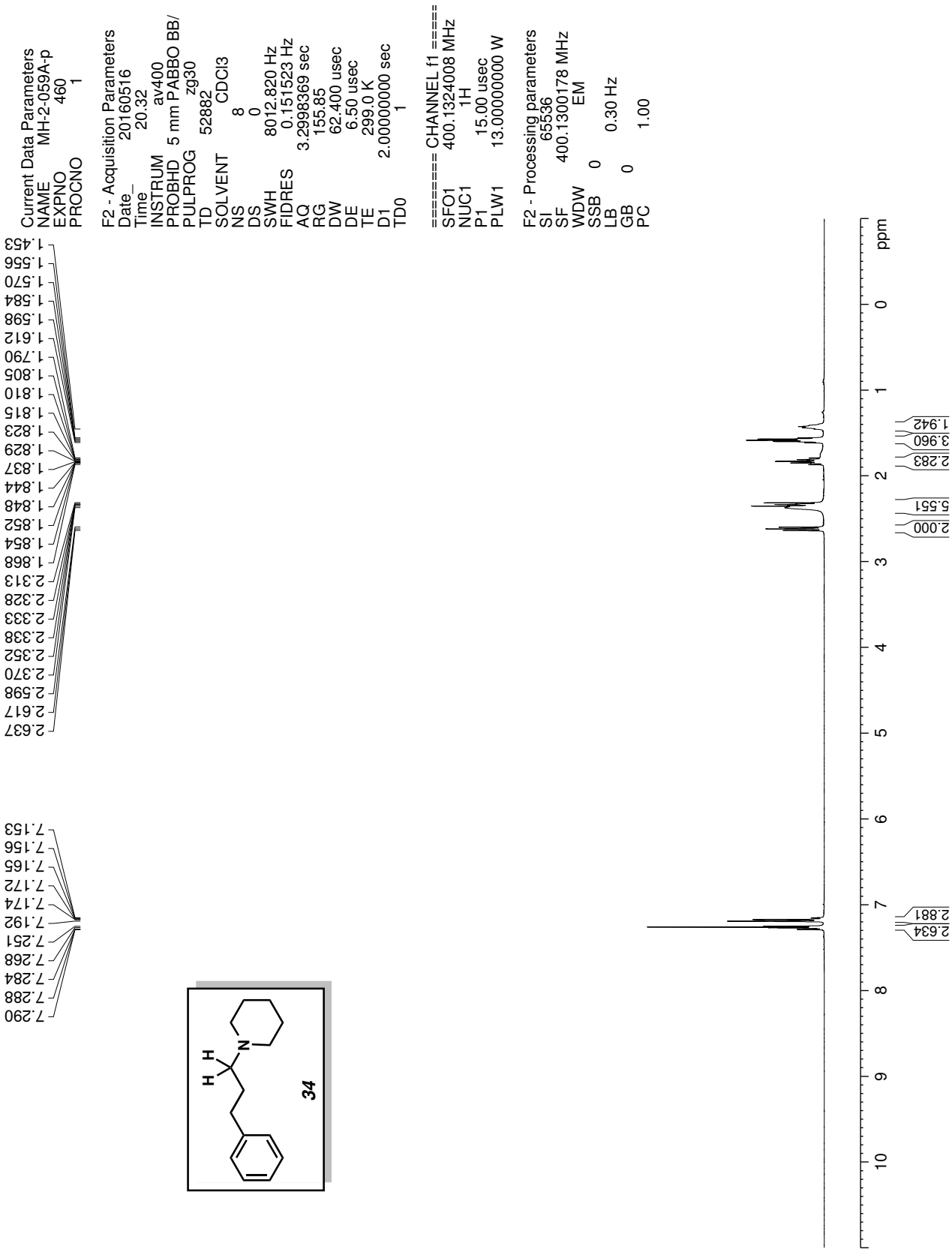

Current Data Parameters
NAME MH-2-110-p
EXPNO 340
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160620
Time 18.22
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 155.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ======
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300184 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.289
7.285
7.270
7.268
7.258
7.252
7.190
7.172
7.157
7.154
2.668
2.650
2.630
2.625
2.607
2.589
2.047
1.810
1.791
1.771
1.753
1.735
1.674
1.644
1.610
1.604
1.576
29
1.384
0.702
3.181
4.000
3.015
2.141
12.125
ppm

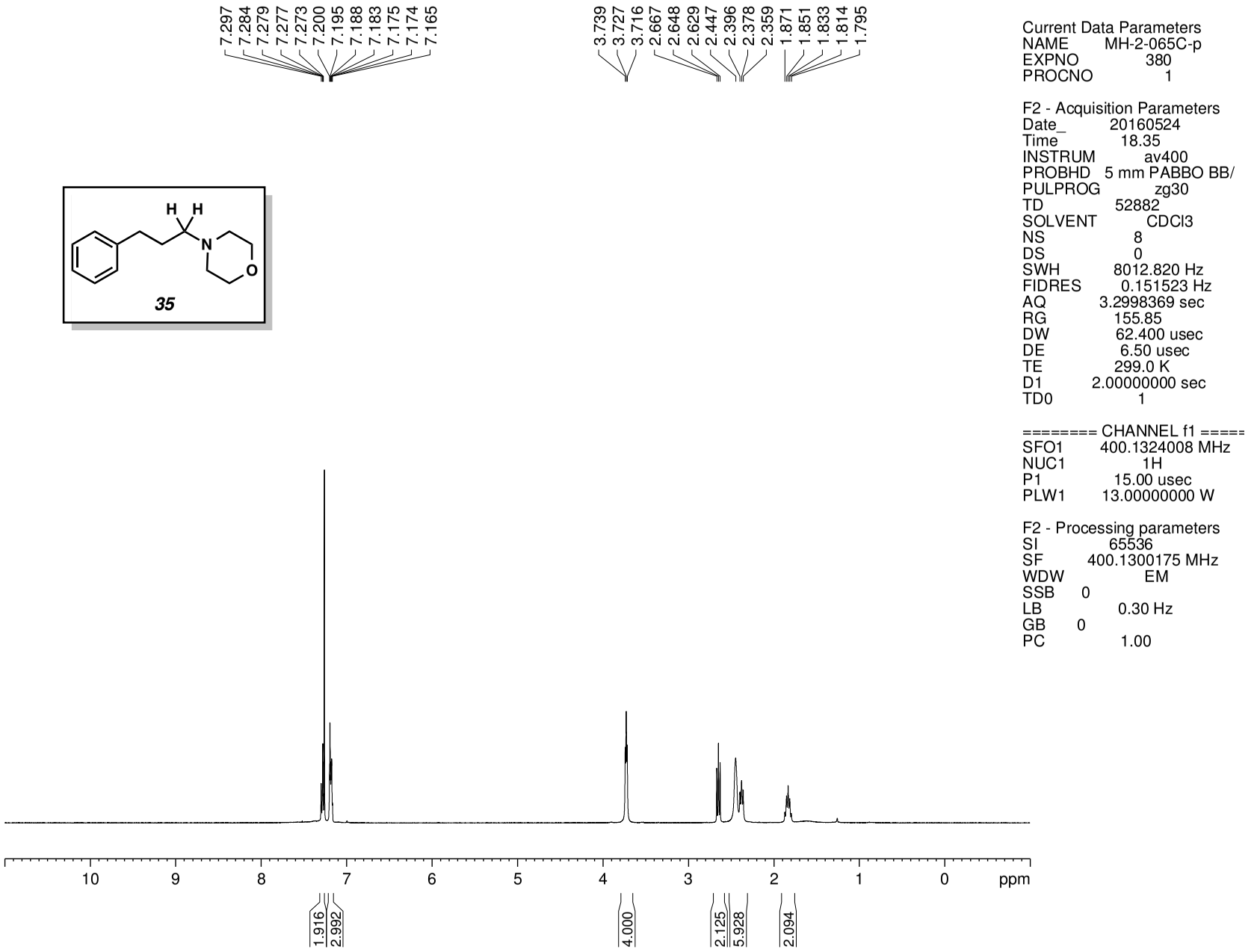

Current Data Parameters
NAME MH-2-050B-p
EXPNO 550
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160510
Time 18.31 h
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.303045 Hz
AQ 3.2998369 sec
RG 155.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300176 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.307
7.288
7.286
7.281
7.270
7.265
7.233
7.227
7.210
7.203
7.193
7.184
7.176
6.695
6.679
6.660
3.363
3.344
3.325
2.924
2.678
2.659
2.640
1.958
1.939
1.920
1.901
1.882
H
H
N
Ph
Me
30
2.023
2.026
2.992
2.000
2.817
4.875
2.006
ppm


Current Data Parameters
NAME MH-2-053B-p
EXPNO 110
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160513
Time 11.22
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 155.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ======
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300178 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.429
7.410
7.381
7.362
7.333
7.314
7.295
7.259
7.247
7.238
7.234
7.215
7.183
7.170
7.152
7.134
7.099
7.097
7.079
3.573
2.610
2.591
2.571
2.509
2.492
2.474
1.868
1.850
1.831
1.813
1.795
H
H
Bn
N
Bn
31
ppm
3.984
4.323
3.788
1.088
2.010
4.000
2.132
1.986
2.029


Current Data Parameters
NAME MH-2-118-p-1
EXPNO 480 1
PROCNO
F2 - Acquisition Parameters
Date_ 20160627
Time 18.25
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 155.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ======
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300176 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.336
1.361
1.373
1.378
1.384
1.388
1.396
1.410
1.418
1.433
1.435
1.724
1.743
1.747
1.756
1.761
1.769
1.777
1.781
1.786
1.800
2.377
2.396
2.414
2.430
2.449
2.467
2.591
2.611
2.630
7.155
7.173
7.182
7.183
7.194
7.201
7.276
7.294
H
H
n-Bu
N
n-Bu
32
ppm
0
1
2
3
4
5
6
7
8
9
10
6.000
4.340
3.961
2.084
1.974
5.934
1.430
2.870


Current Data Parameters
NAME MH-2-148-1
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160717
Time 13.45
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 155.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ======
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.978
0.994
1.693
1.712
1.716
1.725
1.731
1.738
1.747
1.750
1.755
1.770
2.419
2.438
2.456
2.580
2.599
2.619
2.956
2.972
2.989
3.005
3.022
3.038
3.055
7.145
7.163
7.182
7.199
7.250
7.255
7.269
7.283
7.287
H H
N
i-Pr
i-Pr
33
ppm
12.154
2.014
2.010
2.013
2.000
3.003
0.786
1.249


Current Data Parameters
NAME MH-2-059A-p
EXPNO 460
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160516
Time_ 20.32
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 155.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ======
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300178 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.453
1.556
1.570
1.584
1.598
1.612
1.790
1.805
1.810
1.815
1.823
1.829
1.837
1.844
1.848
1.852
1.854
1.868
2.313
2.328
2.333
2.338
2.352
2.370
2.598
2.617
2.637
7.153
7.156
7.165
7.172
7.174
7.192
7.251
7.268
7.284
7.288
7.290
34
2.634
2.881
2.000
5.551
2.283
3.960
1.942
ppm

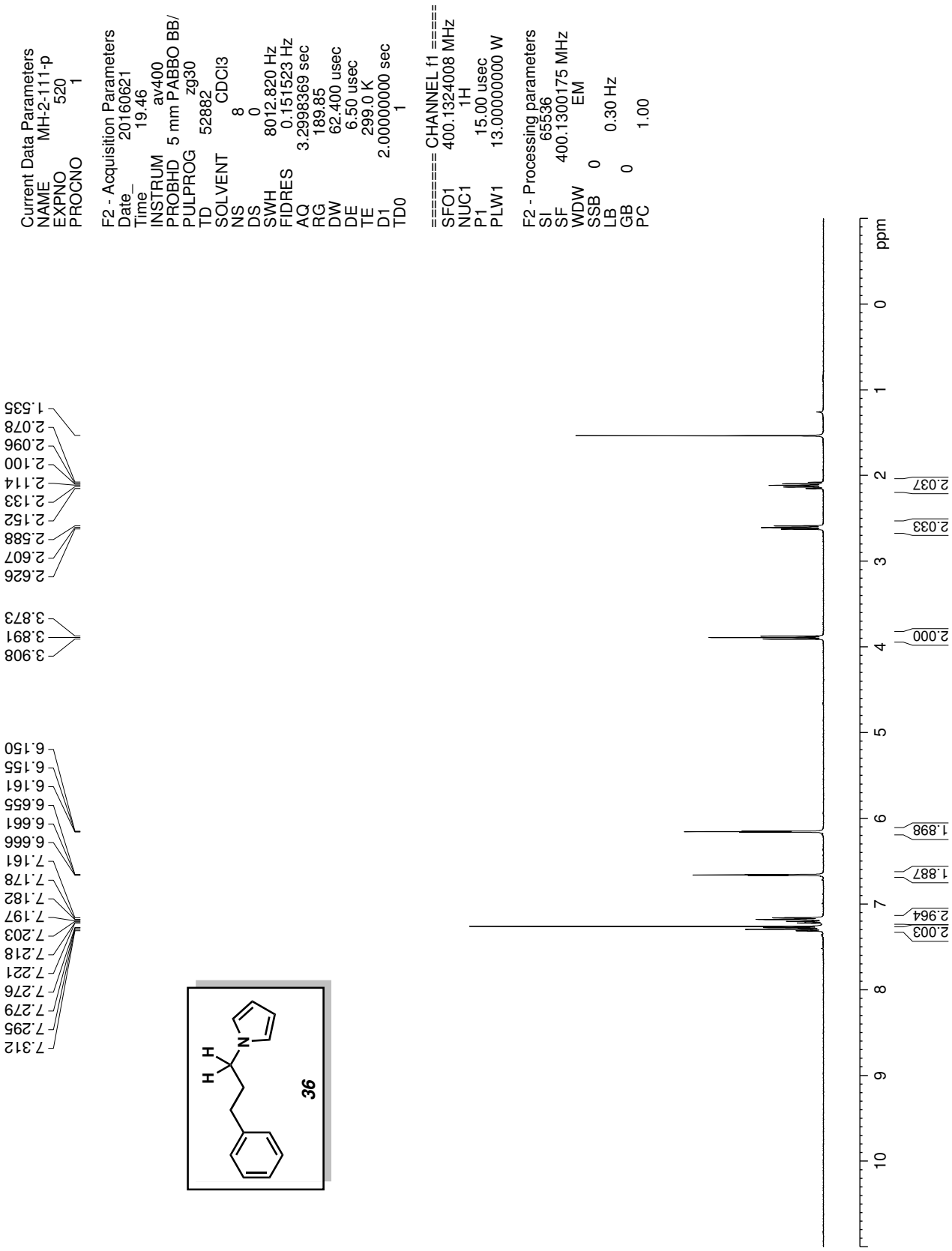


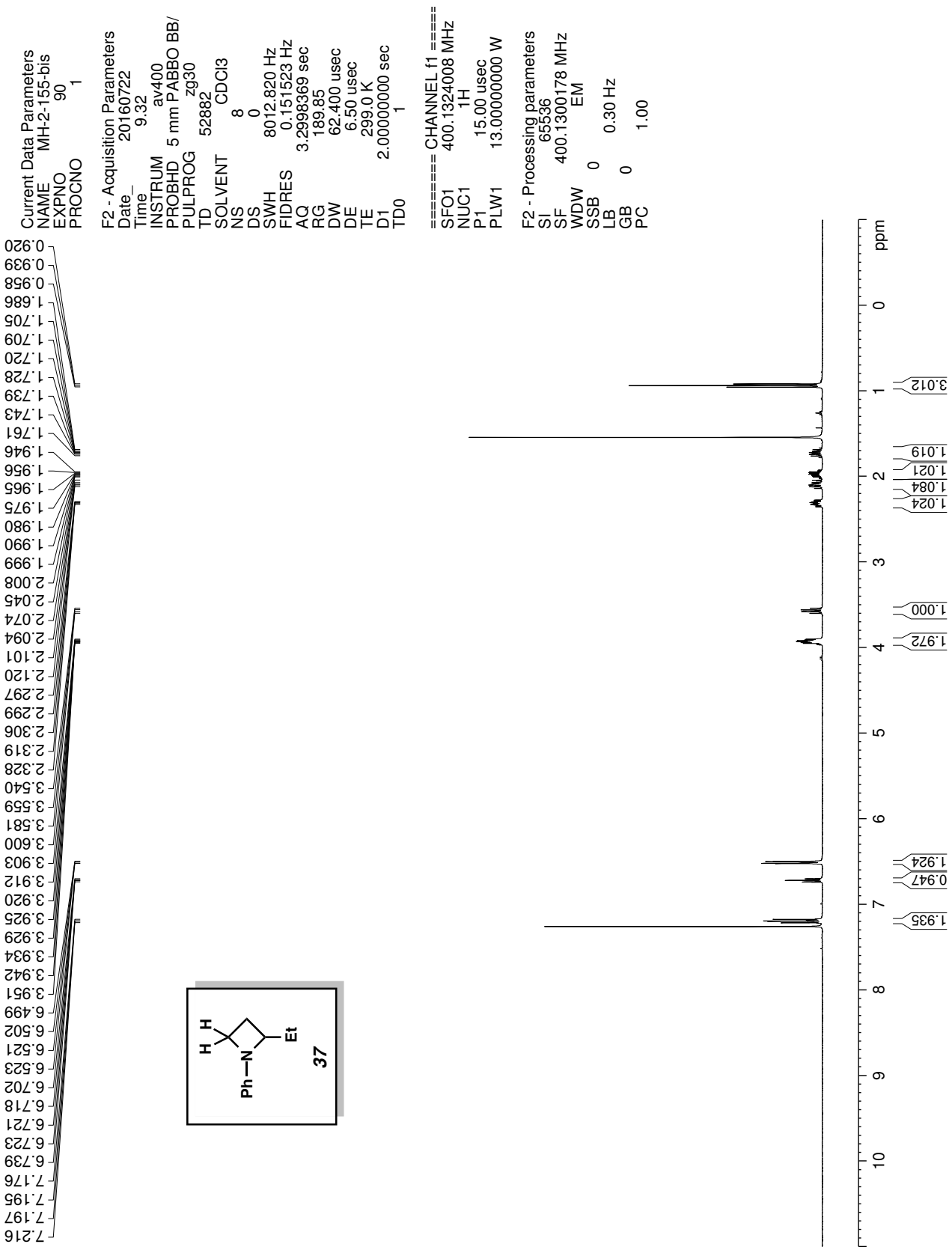
Current Data Parameters
NAME MH-2-065C-p
EXPNO 380
PROCNO 1

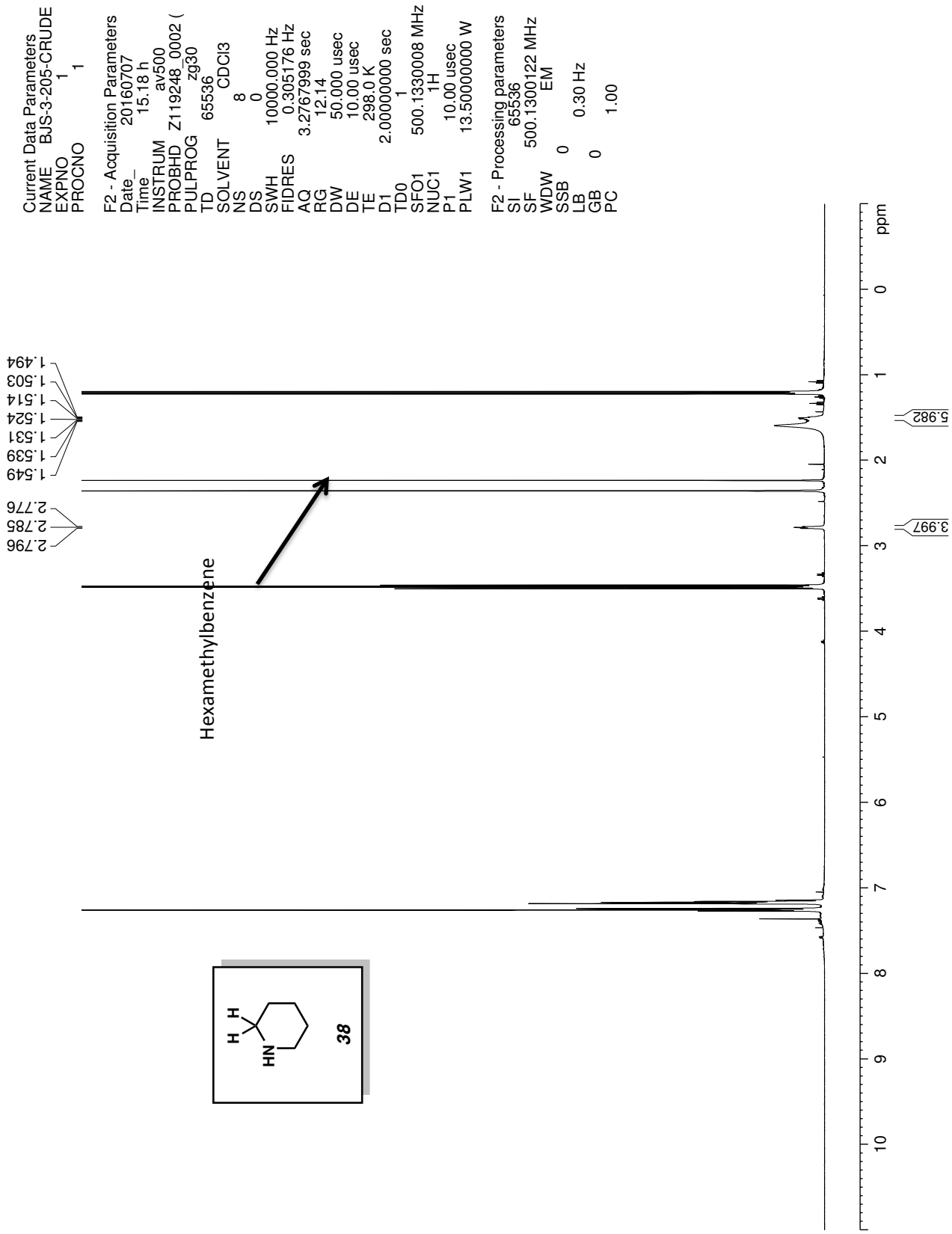
F2 - Acquisition Parameters
Date_ 20160524
Time 18.35
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 155.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1

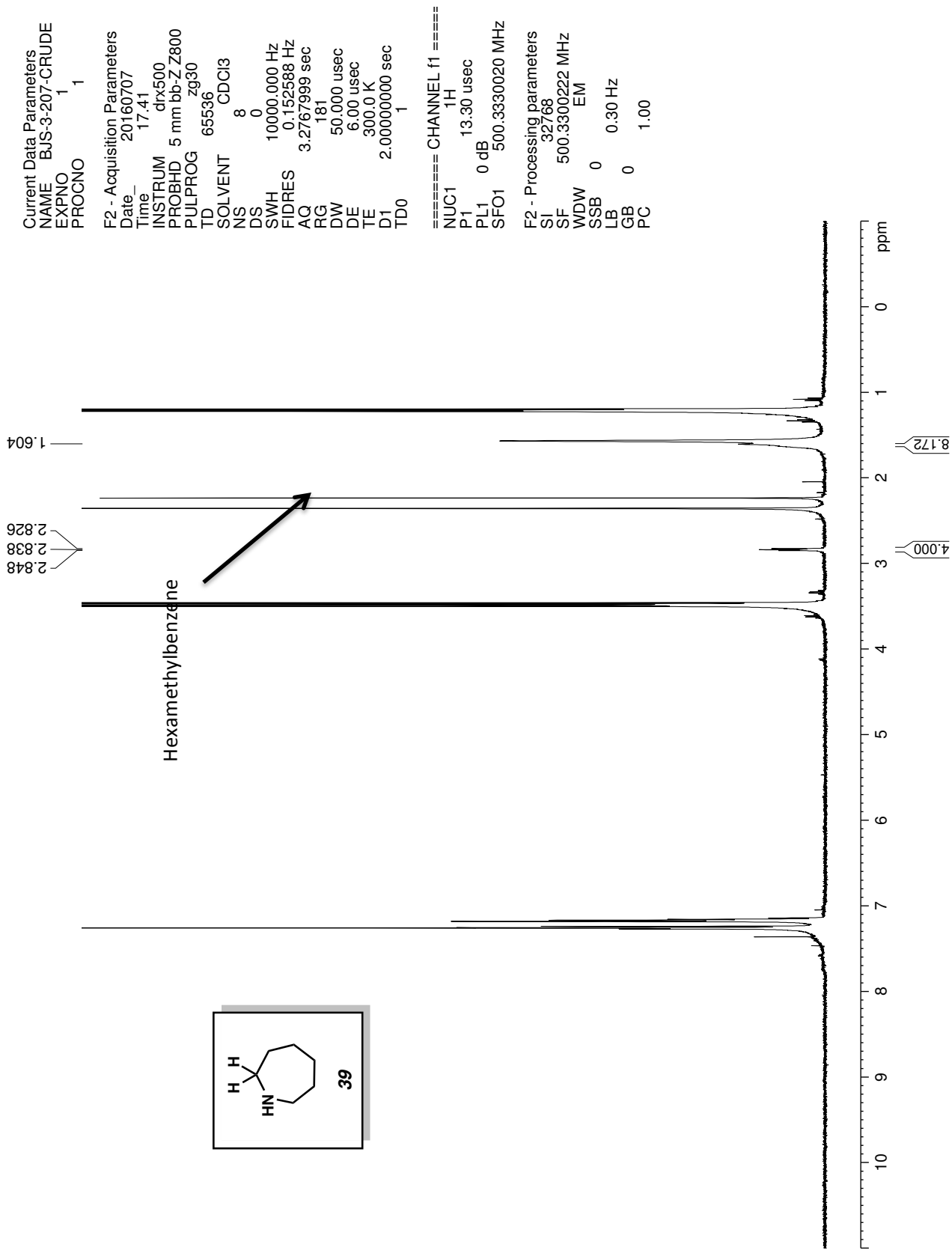
======== CHANNEL f1 =====
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W

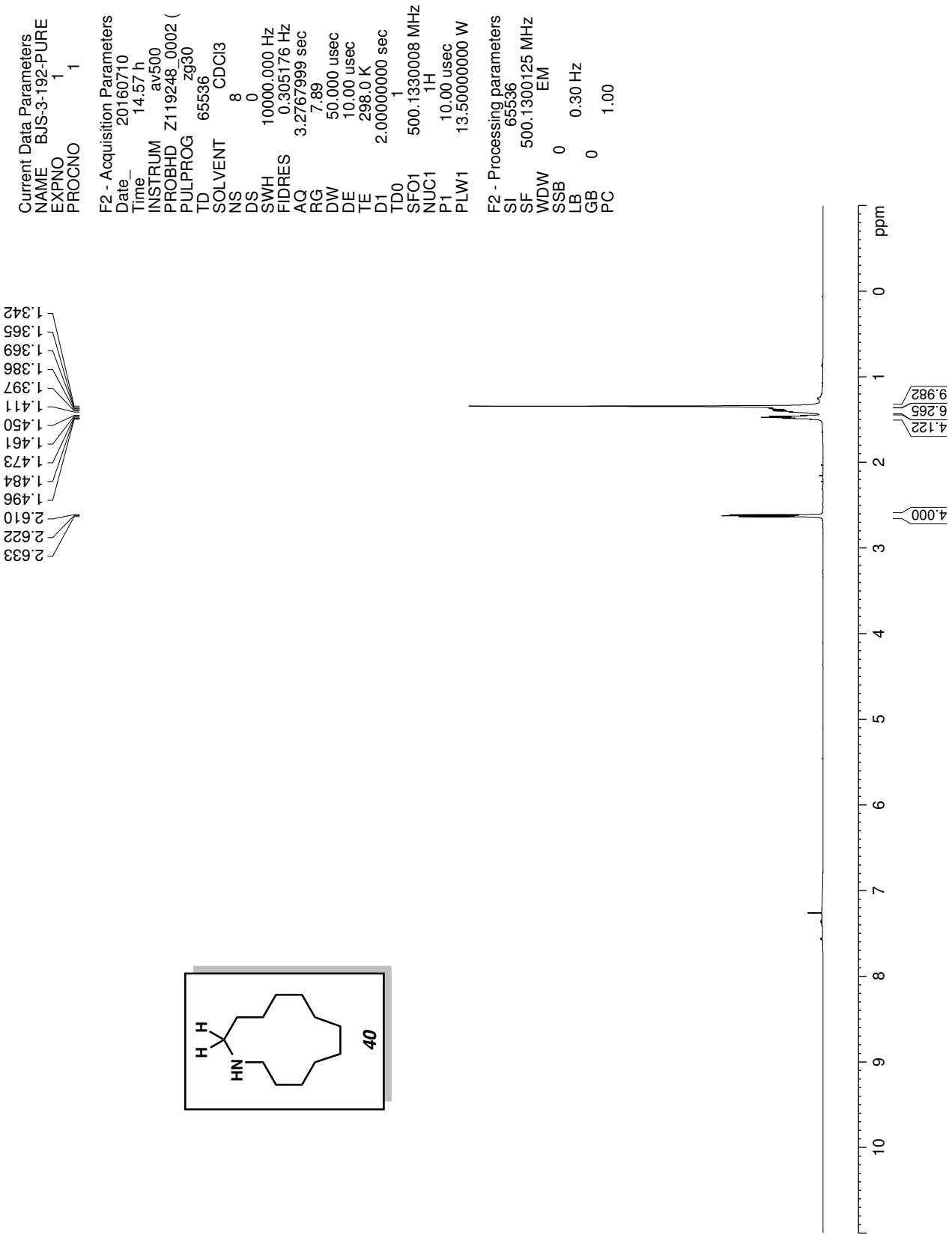
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

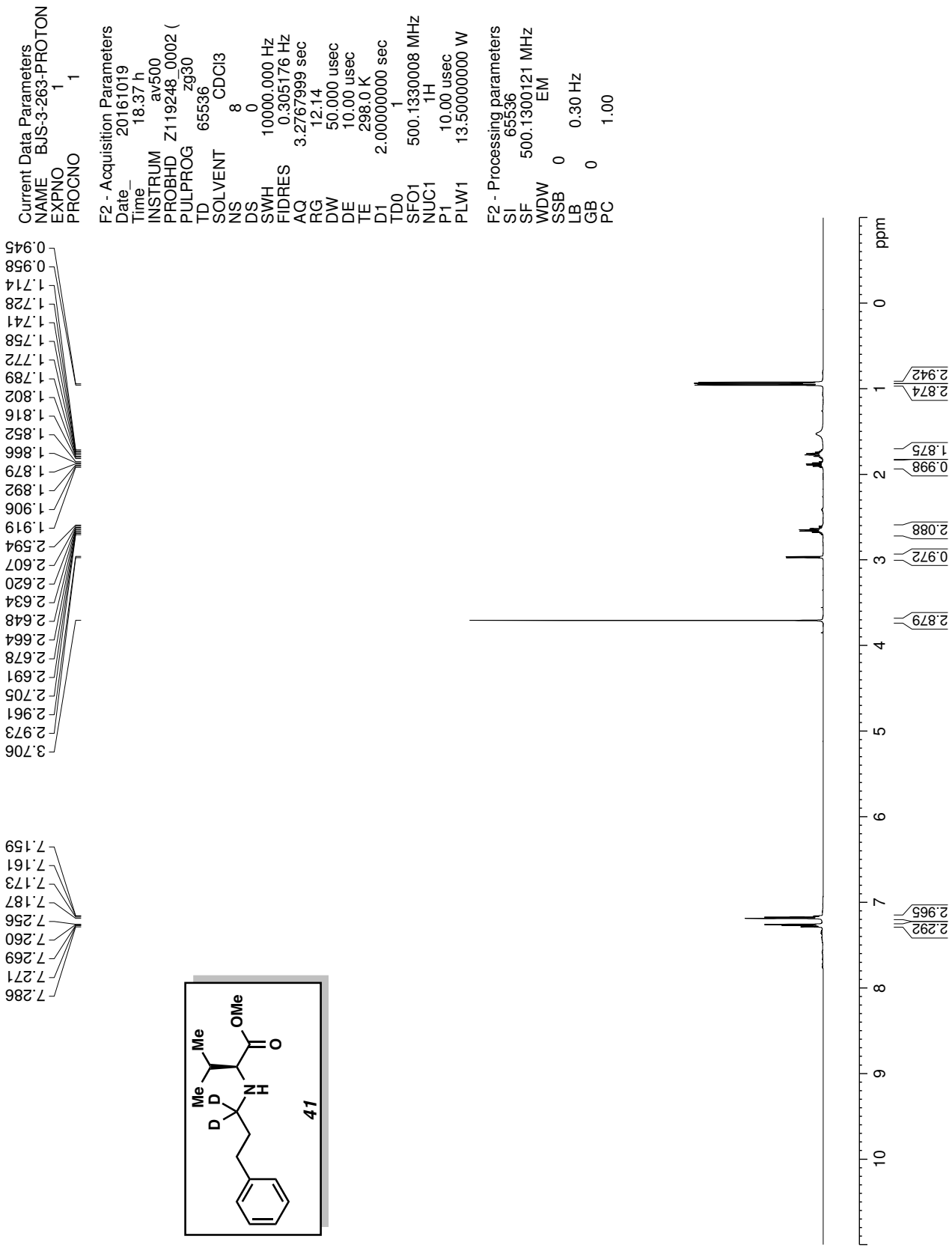

Current Data Parameters
NAME MH-2-111-p
EXPNO 520
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160621
Time 19.46
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 189.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ======
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.312
7.295
7.279
7.276
7.221
7.218
7.203
7.197
7.182
7.178
7.161
6.666
6.661
6.655
6.161
6.155
6.150
3.908
3.891
3.873
2.626
2.607
2.588
2.152
2.133
2.114
2.100
2.096
2.078
1.535
36
2.037
2.033
2.000
1.898
1.887
2.964
2.003
ppm

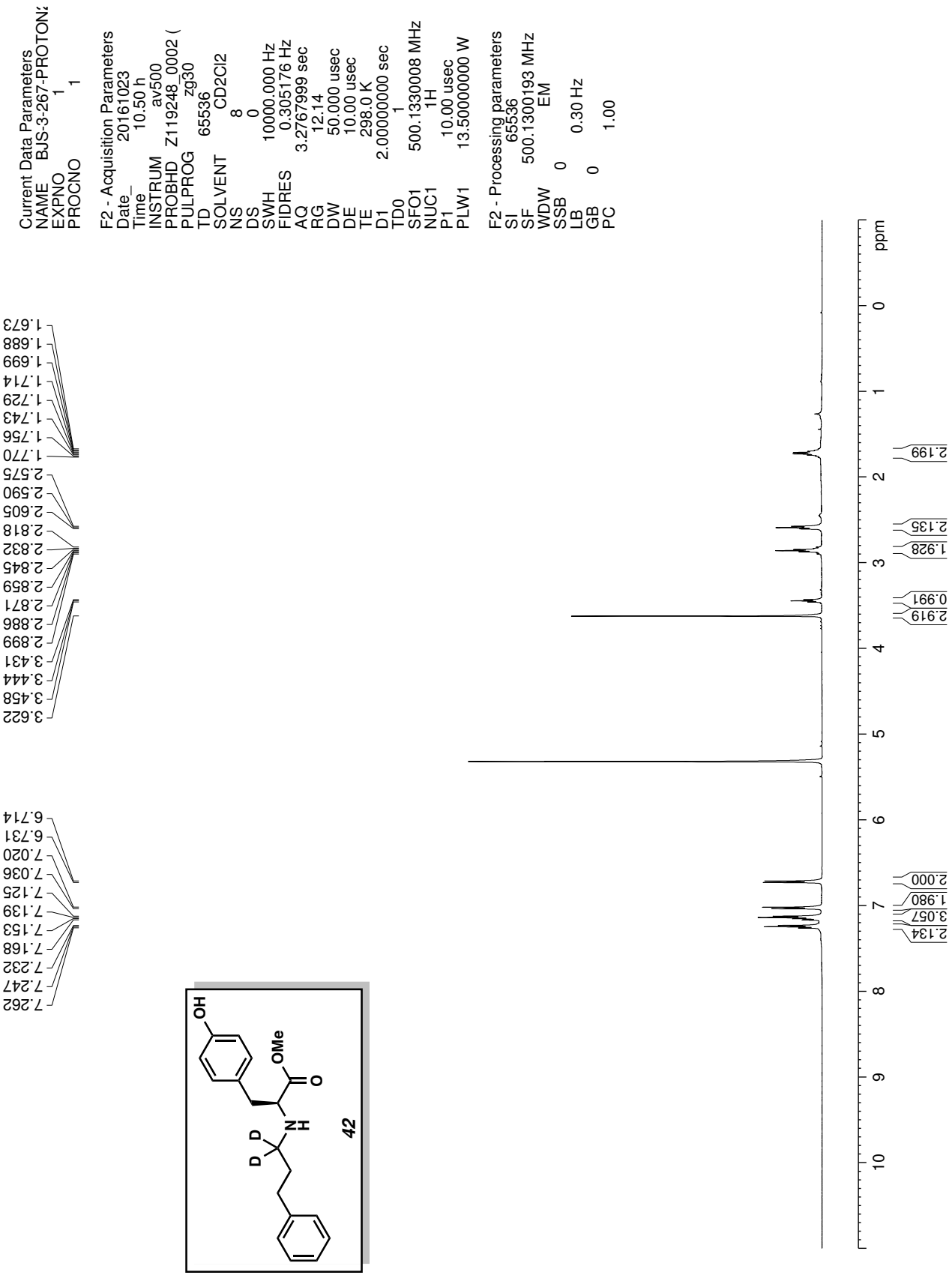

Current Data Parameters
NAME MH-2-155-bis
EXPNO 90 1
PROCNO
F2 - Acquisition Parameters
Date_ 20160722
Time 9.32
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 189.85
DW 62.400 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ======
SFO1 400.1324008 MHz
NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300178 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph—N
H H
Et
37
ppm
3.012
1.019
1.021
1.084
1.024
1.000
1.972
1.935
0.947
1.924

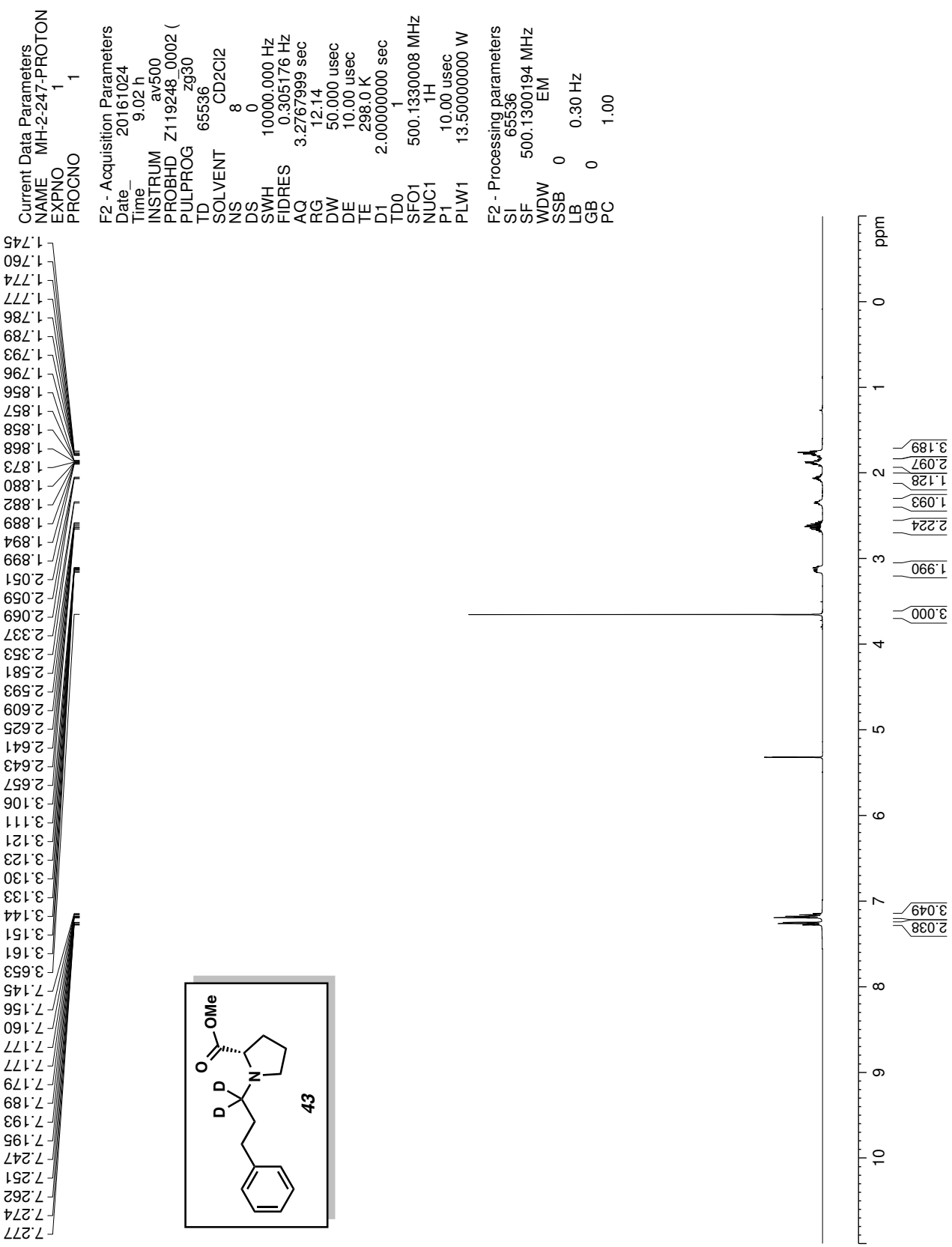

Current Data Parameters
NAME BJS-3-205-CRUDE
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160707
Time 15.18 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
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
1.494
1.503
1.514
1.524
1.531
1.539
1.549
2.776
2.785
2.796
Hexamethylbenzene
5.982
3.997
ppm
38

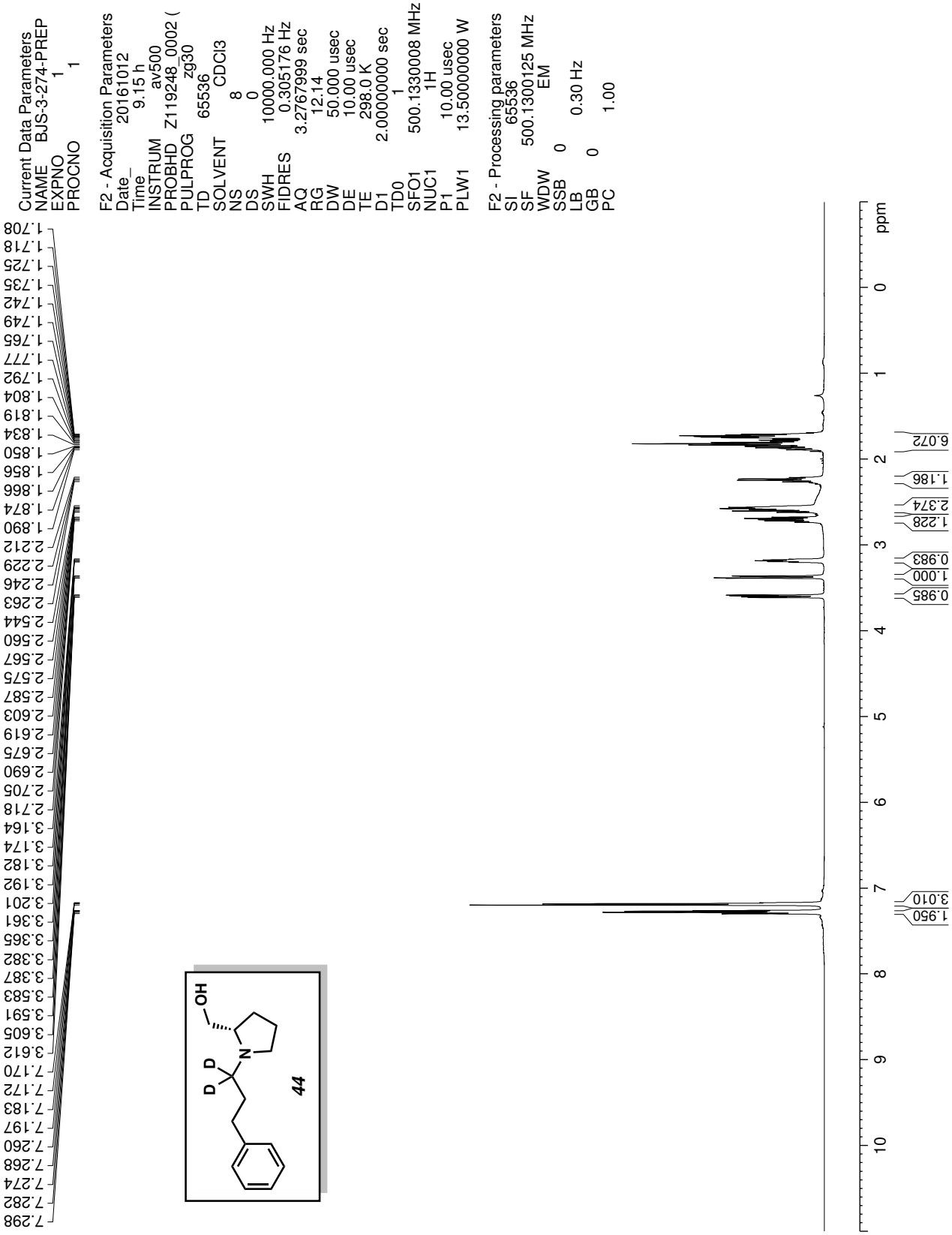

Current Data Parameters
NAME BJS-3-207-CRUDE
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160707
Time 17.41
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300222 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.604
2.848
2.838
2.826
Hexamethylbenzene
8.172
4.000
ppm
39

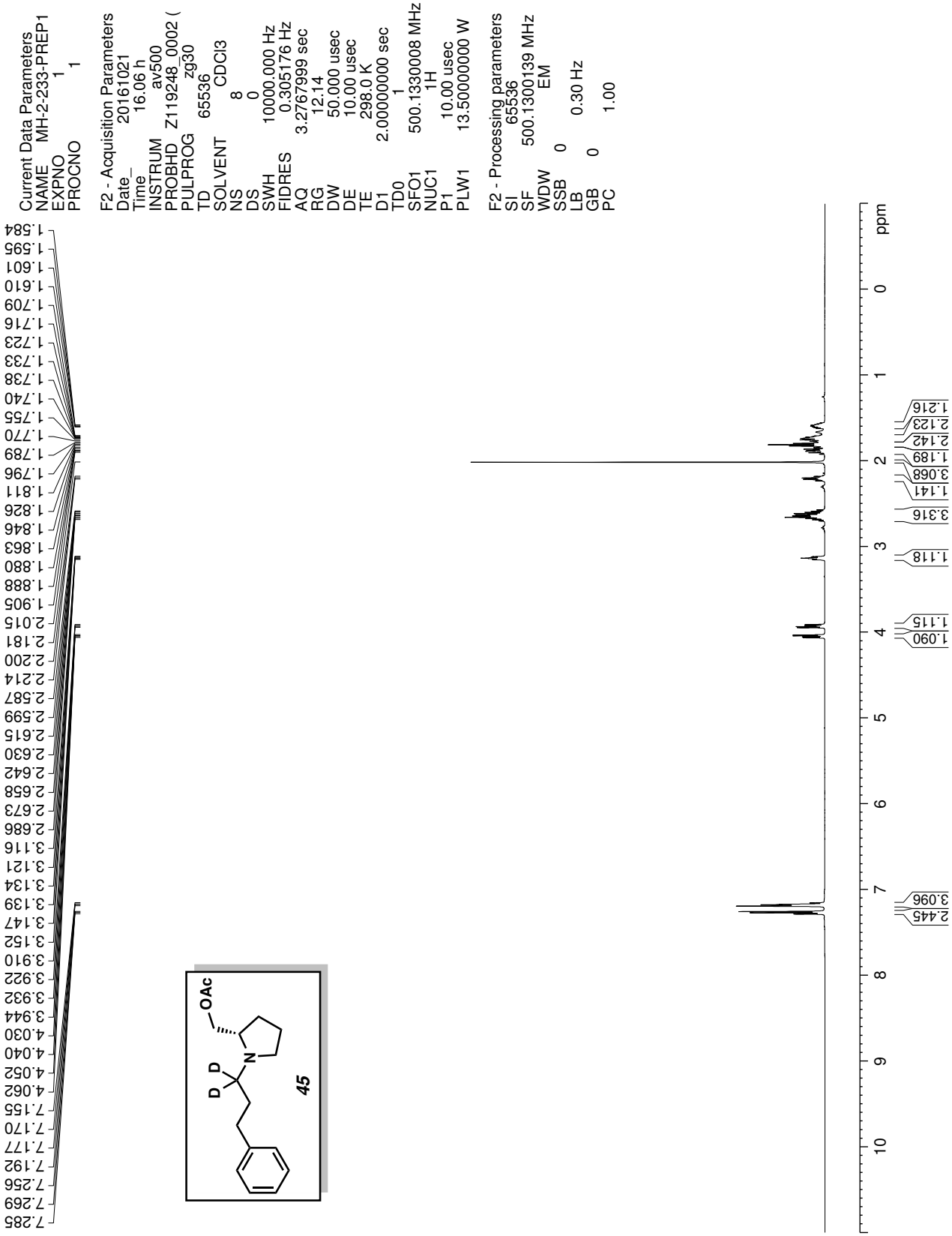

Current Data Parameters
NAME BJS-3-192-PURE
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160710
Time 14.57 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 7.89
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 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
2.633
2.622
2.610
1.496
1.484
1.473
1.461
1.450
1.411
1.397
1.386
1.369
1.365
1.342
4.000
9.982
6.265
4.122
ppm
40

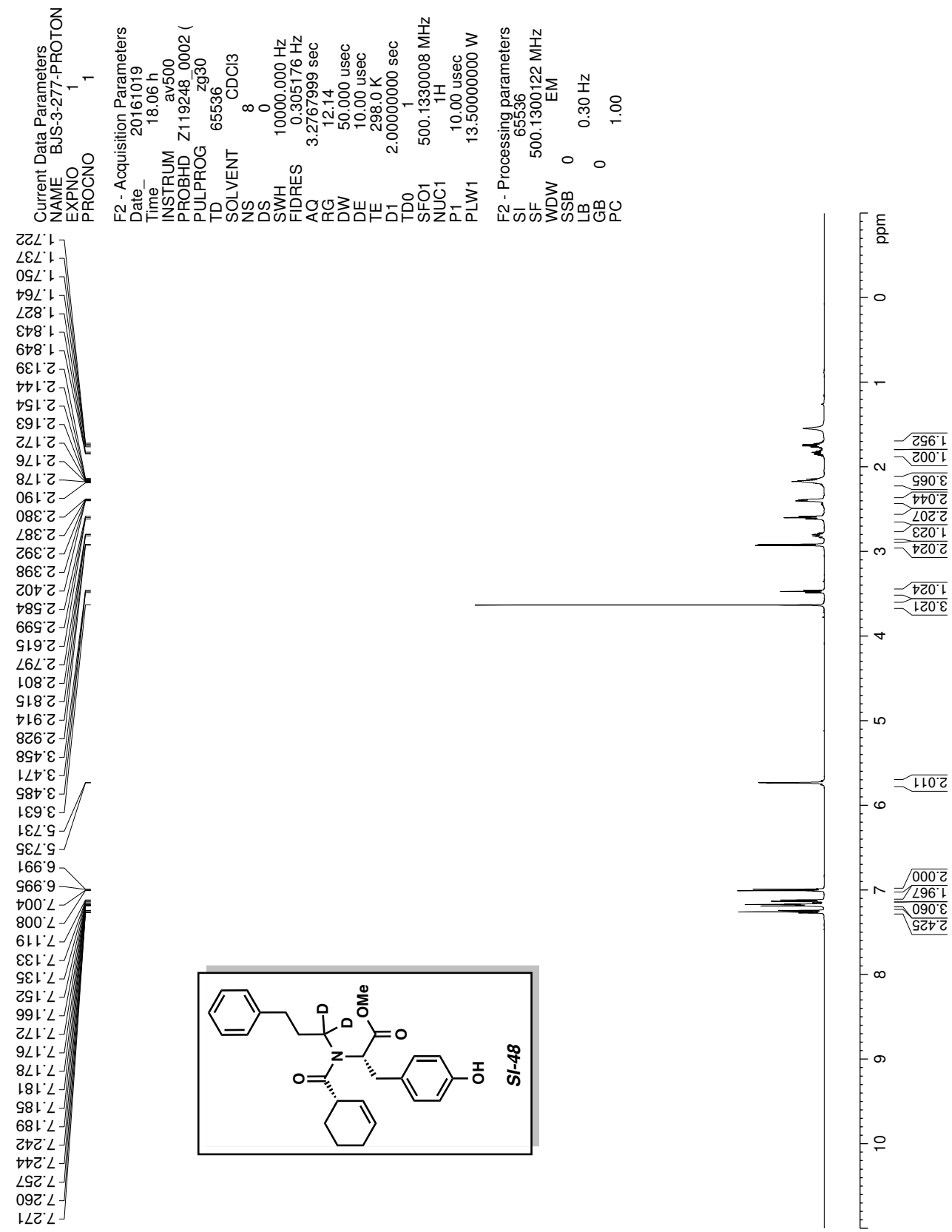

Current Data Parameters
NAME BJS-3-263-PROTON
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161019
Time 18.37 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 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 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
D
D
N
H
Me
OMe
O
41
ppm
2.942
2.874
1.875
0.998
2.088
0.972
2.879
2.965
2.292


Current Data Parameters
NAME BJS-3-267-PROTON
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161023
Time 10.50 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
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 500.1300193 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.262
7.247
7.232
7.168
7.153
7.139
7.125
7.036
7.020
6.731
6.714
3.622
3.458
3.444
3.431
2.899
2.886
2.871
2.859
2.845
2.832
2.818
2.605
2.590
2.575
1.770
1.756
1.743
1.729
1.714
1.699
1.688
1.673
OH
OMe
O
N
H
D
D
42
ppm
2.134
3.057
1.980
2.000
2.919
0.991
1.928
2.135
2.199

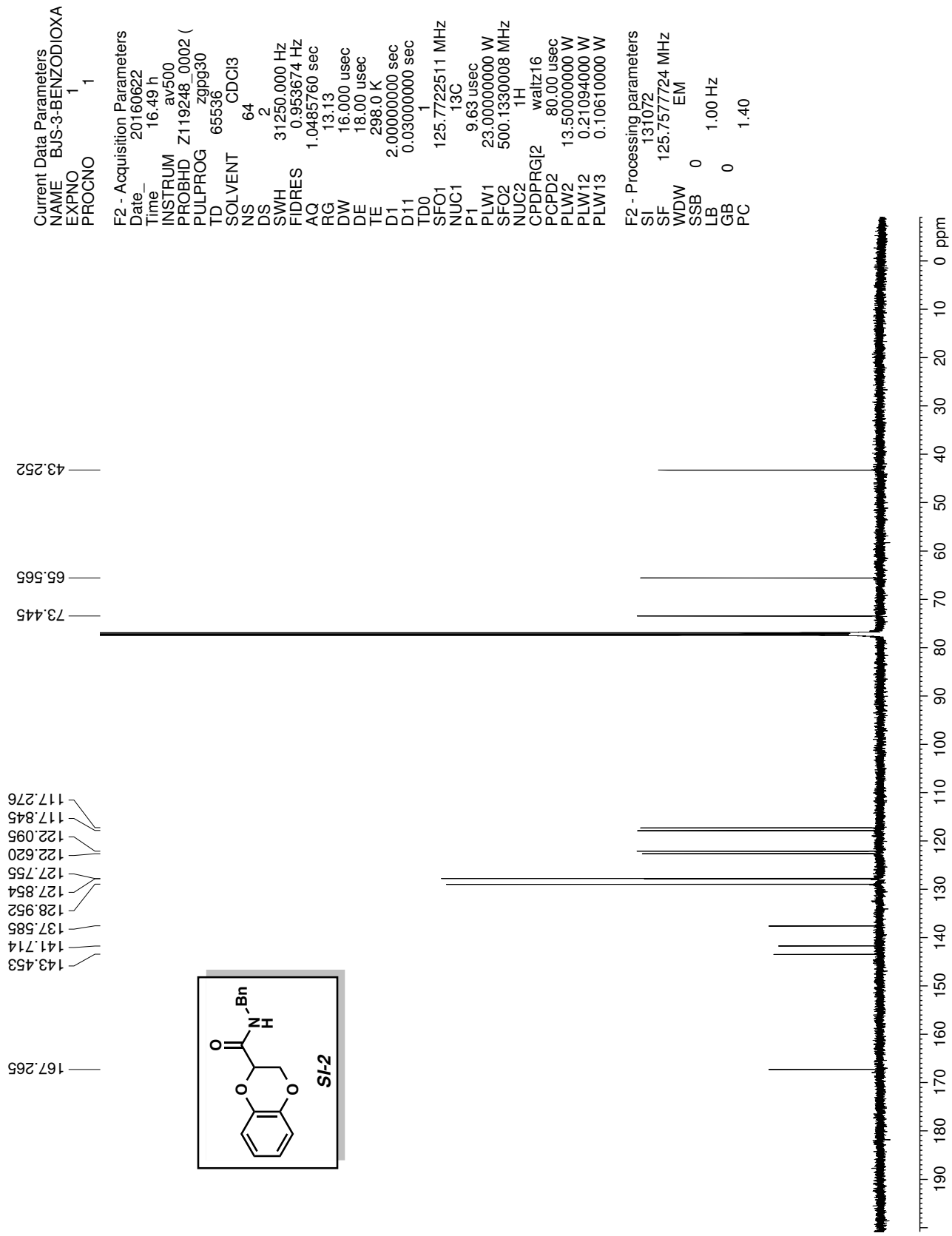

Current Data Parameters
NAME MH-2-247-PROTON
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161024
Time 9.02 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
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 500.1300194 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.745
1.760
1.774
1.777
1.786
1.789
1.793
1.796
1.856
1.857
1.858
1.868
1.873
1.880
1.882
1.889
1.894
1.899
2.051
2.059
2.069
2.337
2.353
2.581
2.593
2.609
2.625
2.641
2.643
2.657
3.106
3.111
3.121
3.123
3.130
3.133
3.144
3.151
3.161
3.653
7.145
7.156
7.160
7.177
7.177
7.179
7.189
7.193
7.195
7.247
7.251
7.262
7.274
7.277
OMe
O
N
D D
43
3.189
2.097
1.128
1.093
2.224
1.990
3.000
2.038
3.049
ppm

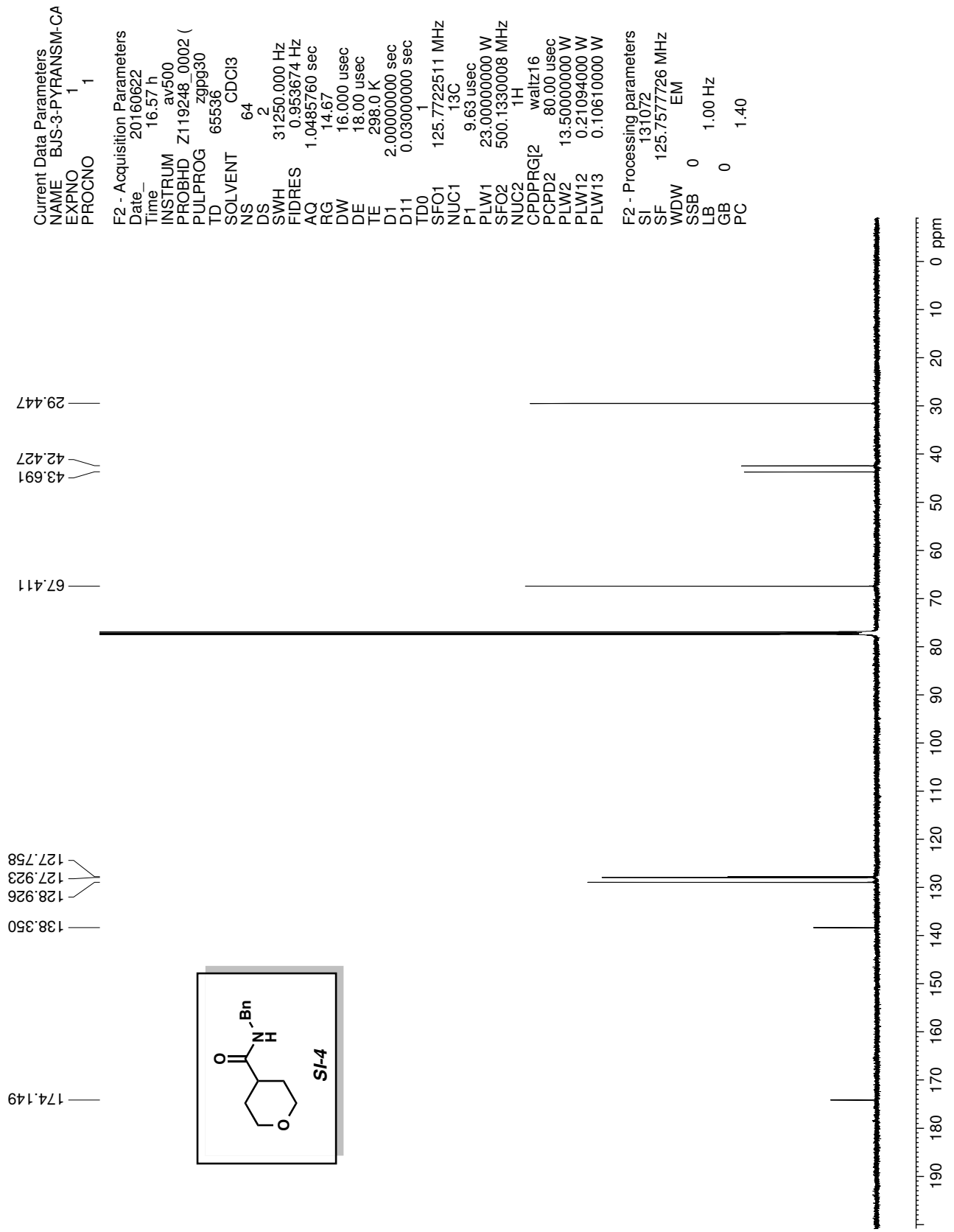

Current Data Parameters
NAME BJS-3-274-PREP
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161012
Time 9.15 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 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 500.1300125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
N
D D
44
ppm
6.072
1.186
2.374
1.228
0.983
1.000
0.985
1.950
3.010

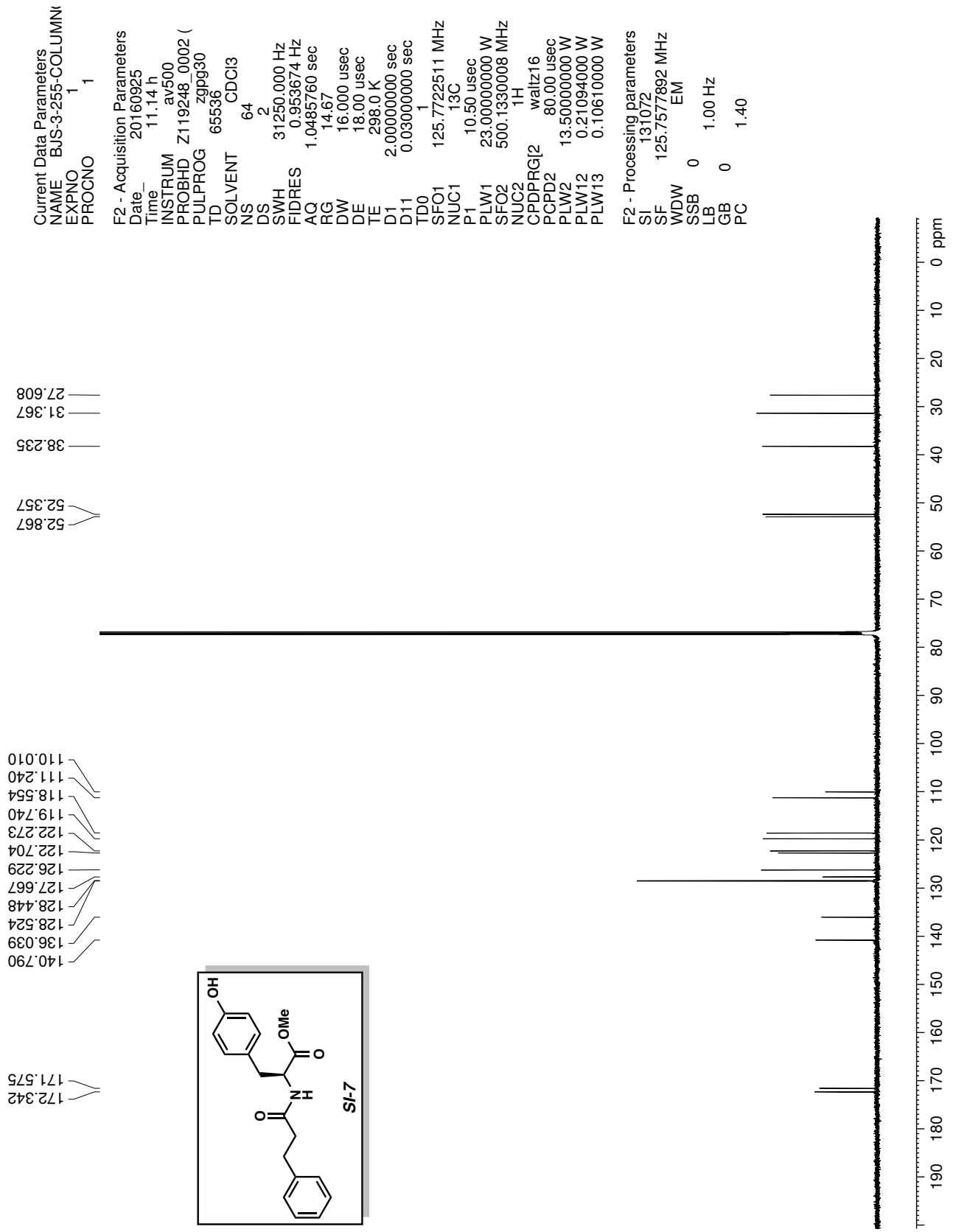

Current Data Parameters
NAME MH-2-233-PREP1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161021
Time 16.06 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
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 500.1300139 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OAc
D D
N
45
ppm
2.445
3.096
1.090
1.115
1.118
3.316
1.141
3.068
1.189
2.142
2.123
1.216

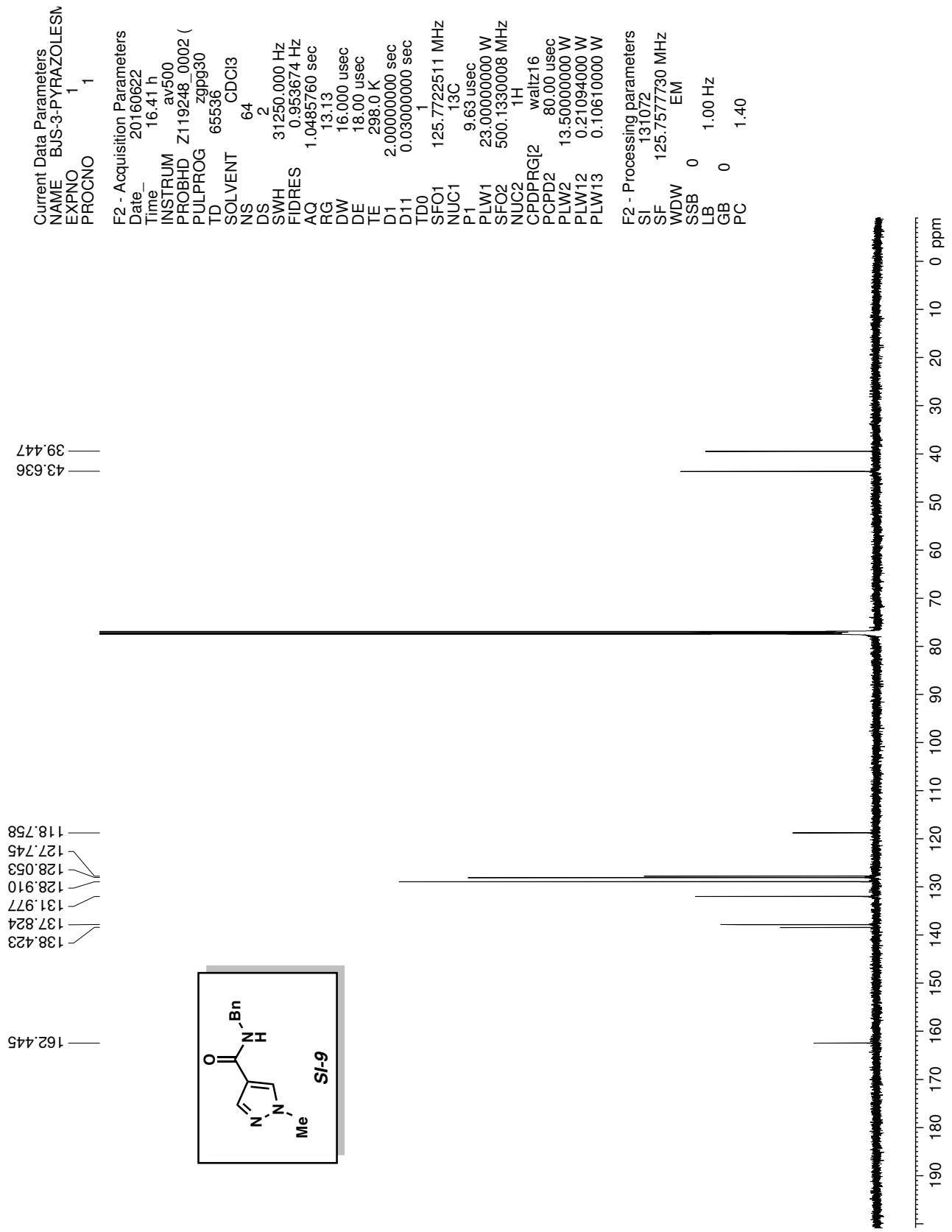

Current Data Parameters
NAME BJS-3-277-PROTON
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161019
Time 18.06 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 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
SI-48
ppm
2.425
3.060
1.967
2.000
2.011
3.021
1.024
2.024
1.023
2.207
2.044
3.065
1.002
1.952

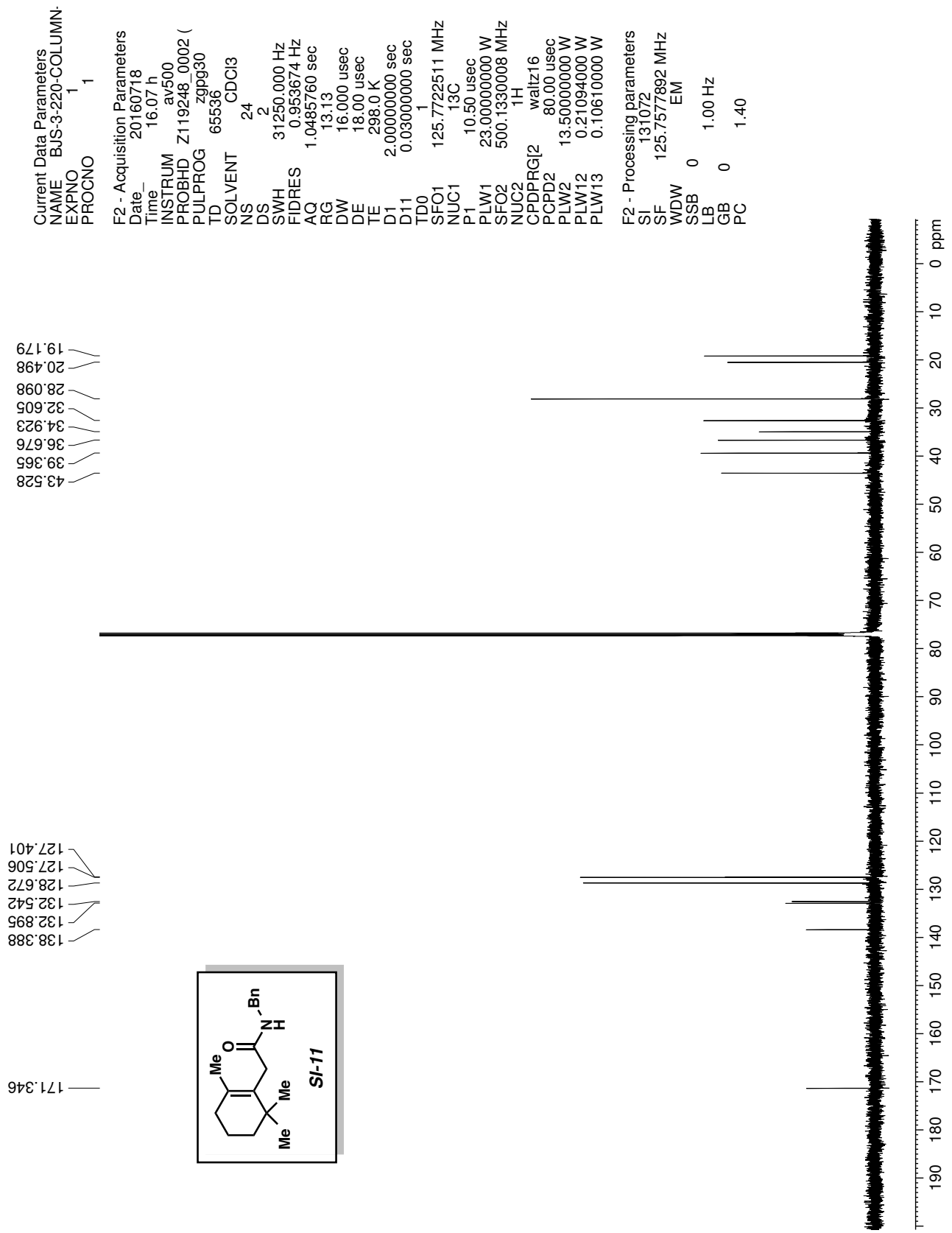
^{13}C NMR Spectra

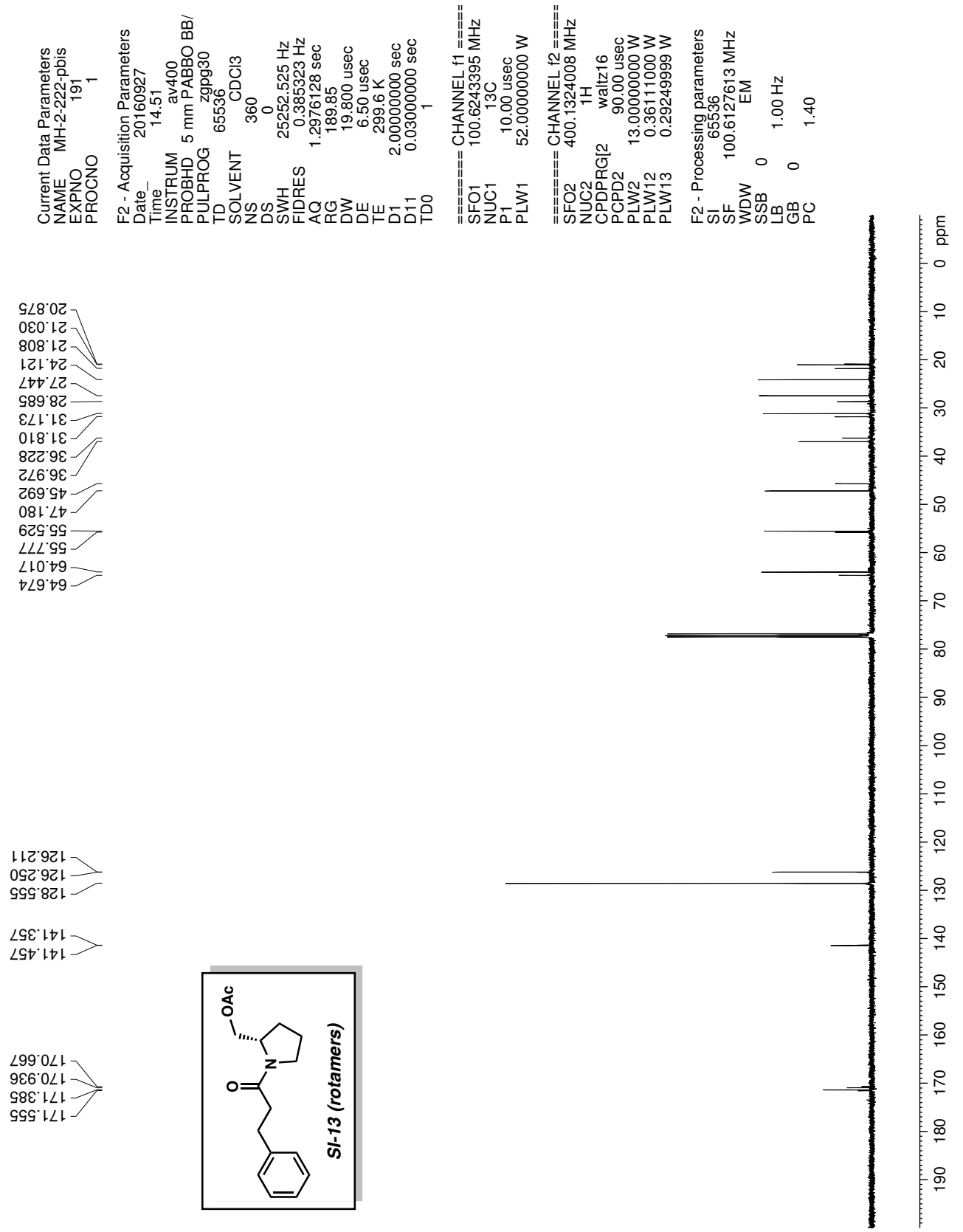

Current Data Parameters
NAME BJS-3-BENZODIOXA
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160622
Time 16.49 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 13.13
DW 16.000 usec
DE 18.00 usec
TE 298.0 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 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577724 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
43.252
65.565
73.445
117.276
117.845
122.095
122.620
127.755
127.854
128.952
137.585
141.714
143.453
167.265
O
N
H
Bn
O
O
SI-2
0 ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190

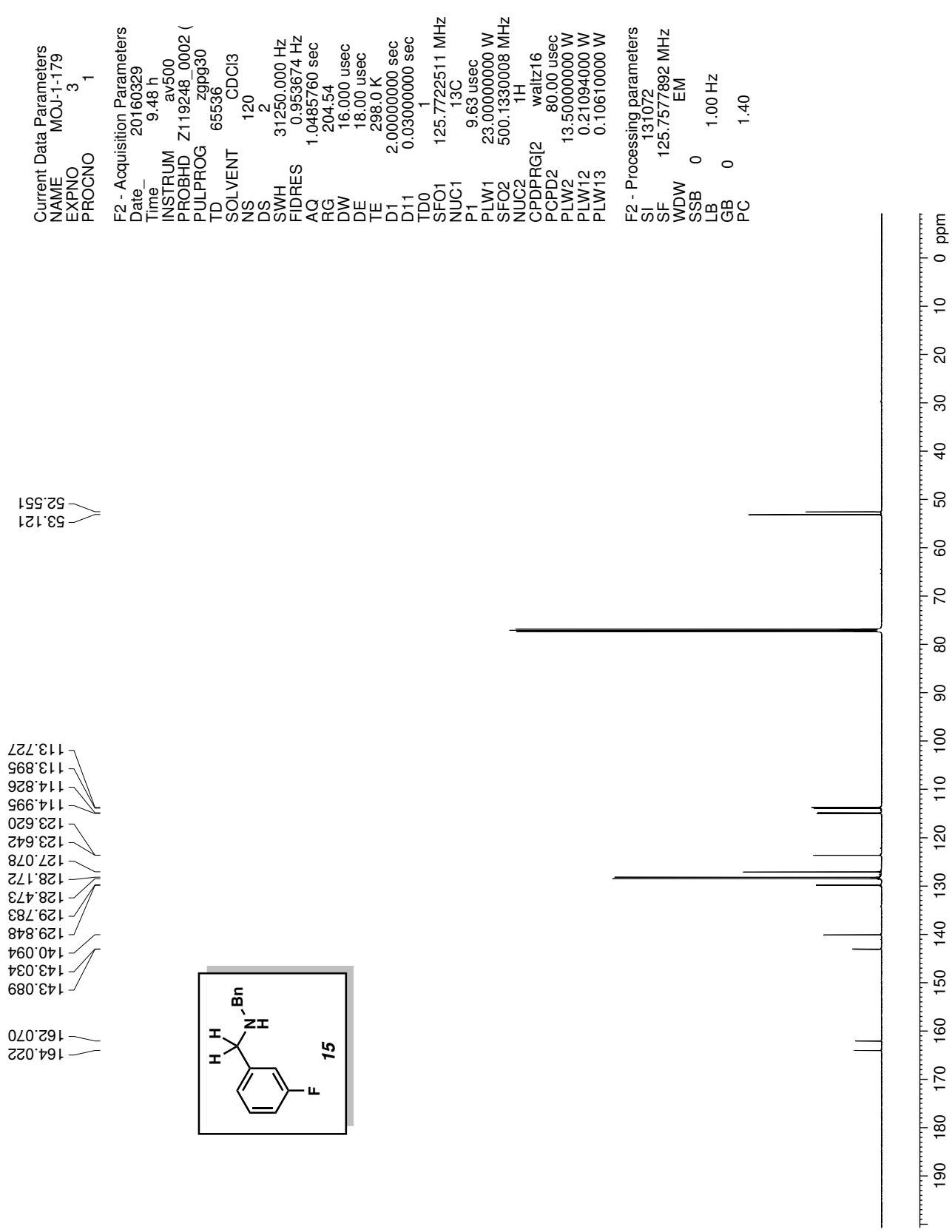

Current Data Parameters
NAME BJS-3-PYRANSM-CA
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160622
Time 16.57 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 14.67
DW 16.000 usec
DE 18.00 usec
TE 298.0 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 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577726 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
174.149
138.350
128.926
127.923
127.758
67.411
43.691
42.427
29.447
SI-4
Bn
N
H
O
O
0 ppm

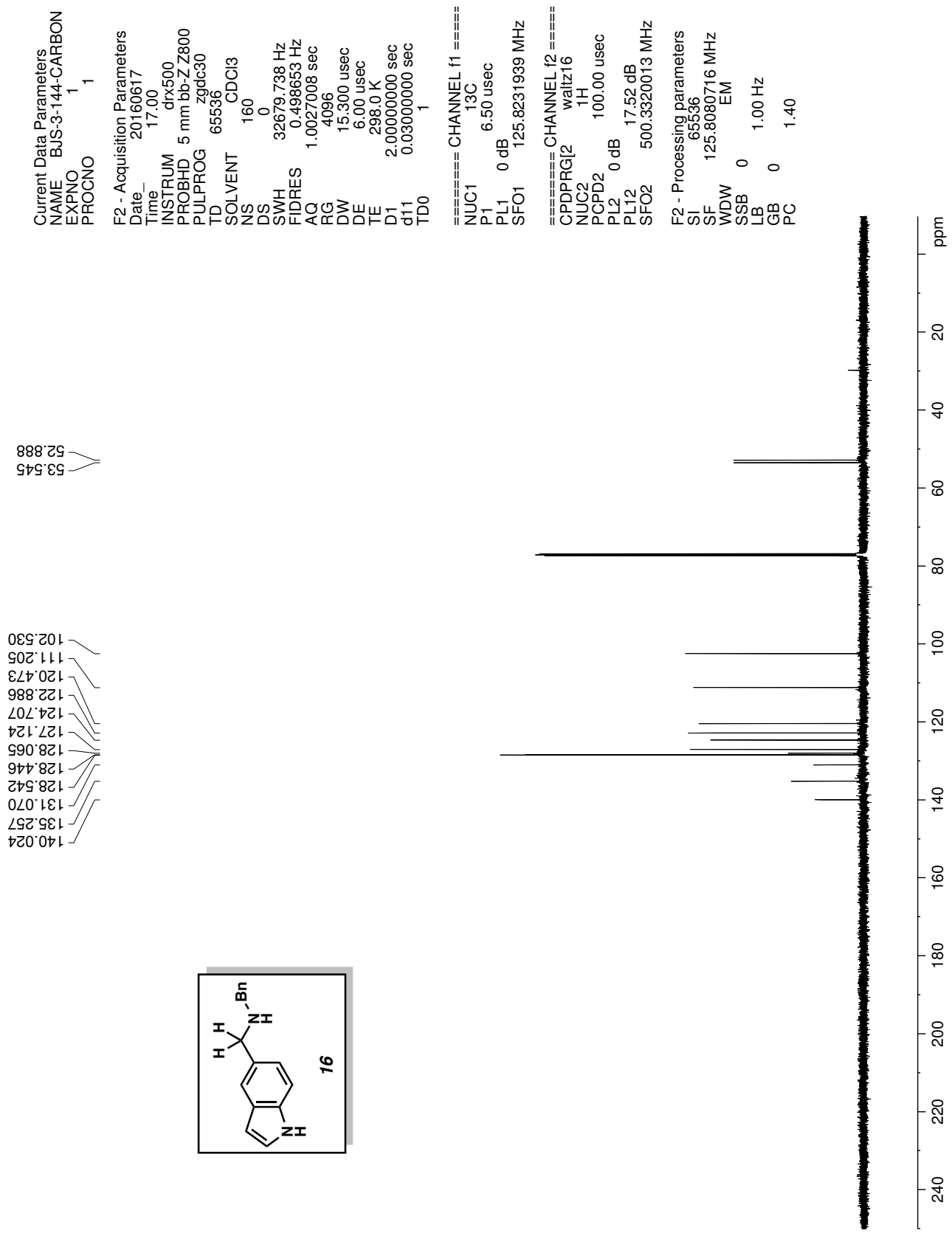

Current Data Parameters
NAME BJS-3-255-COLUMN
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160925
Time 11.14 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 14.67
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 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 125.7577892 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
27.608
31.367
38.235
52.357
52.867
110.010
111.240
118.554
119.740
122.273
122.704
126.229
127.667
128.448
128.524
136.039
140.790
171.575
172.342
OH
OMe
O
N
H
O
SI-7
0 ppm

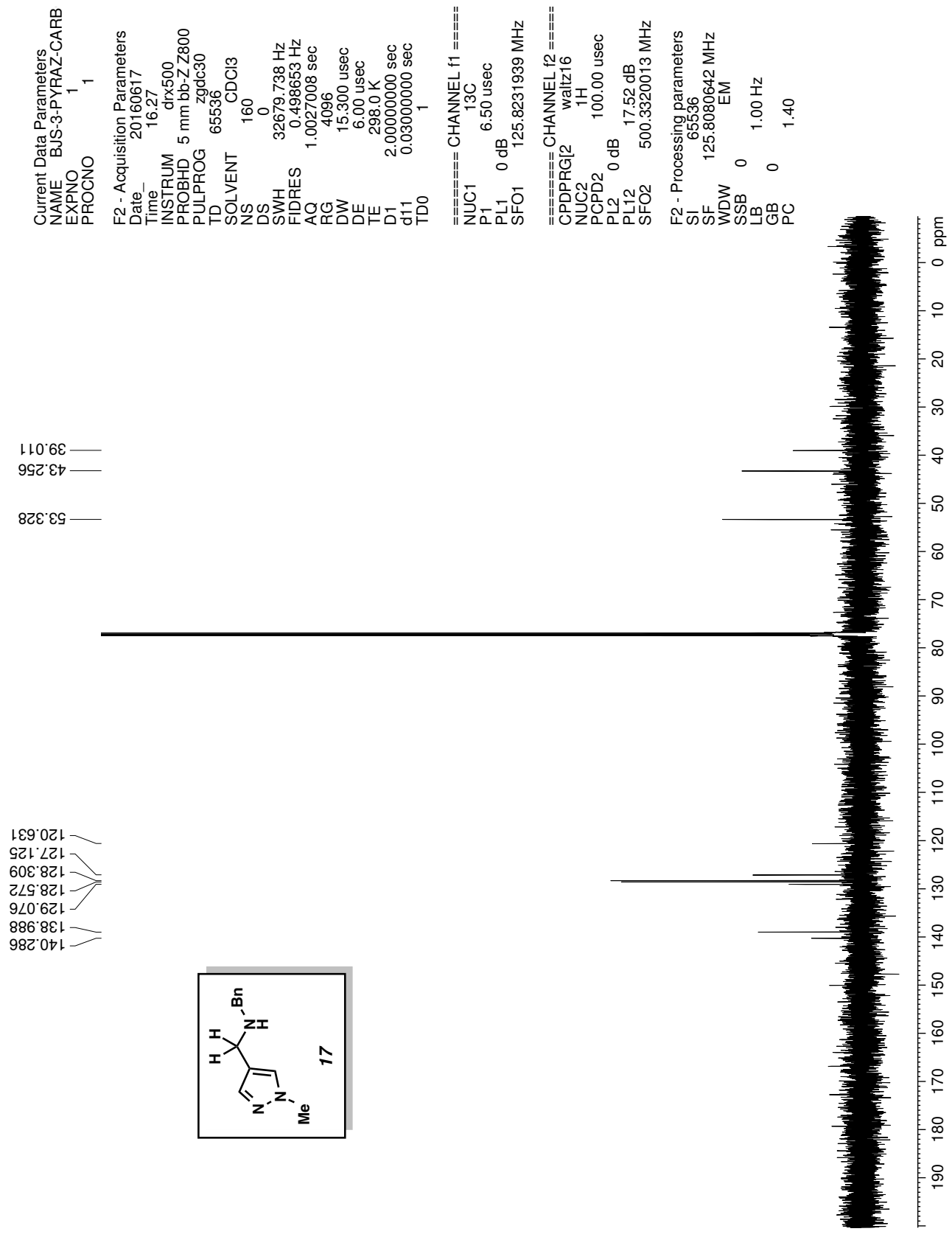

Current Data Parameters
NAME BJS-3-PYRAZOLESM
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160622
Time 16.41 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 13.13
DW 16.000 usec
DE 18.00 usec
TE 298.0 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 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577730 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
39.447
43.636
118.758
127.745
128.053
128.910
131.977
137.824
138.423
162.445
Bn
N
H
O
N
N
Me
SI-9
0 ppm

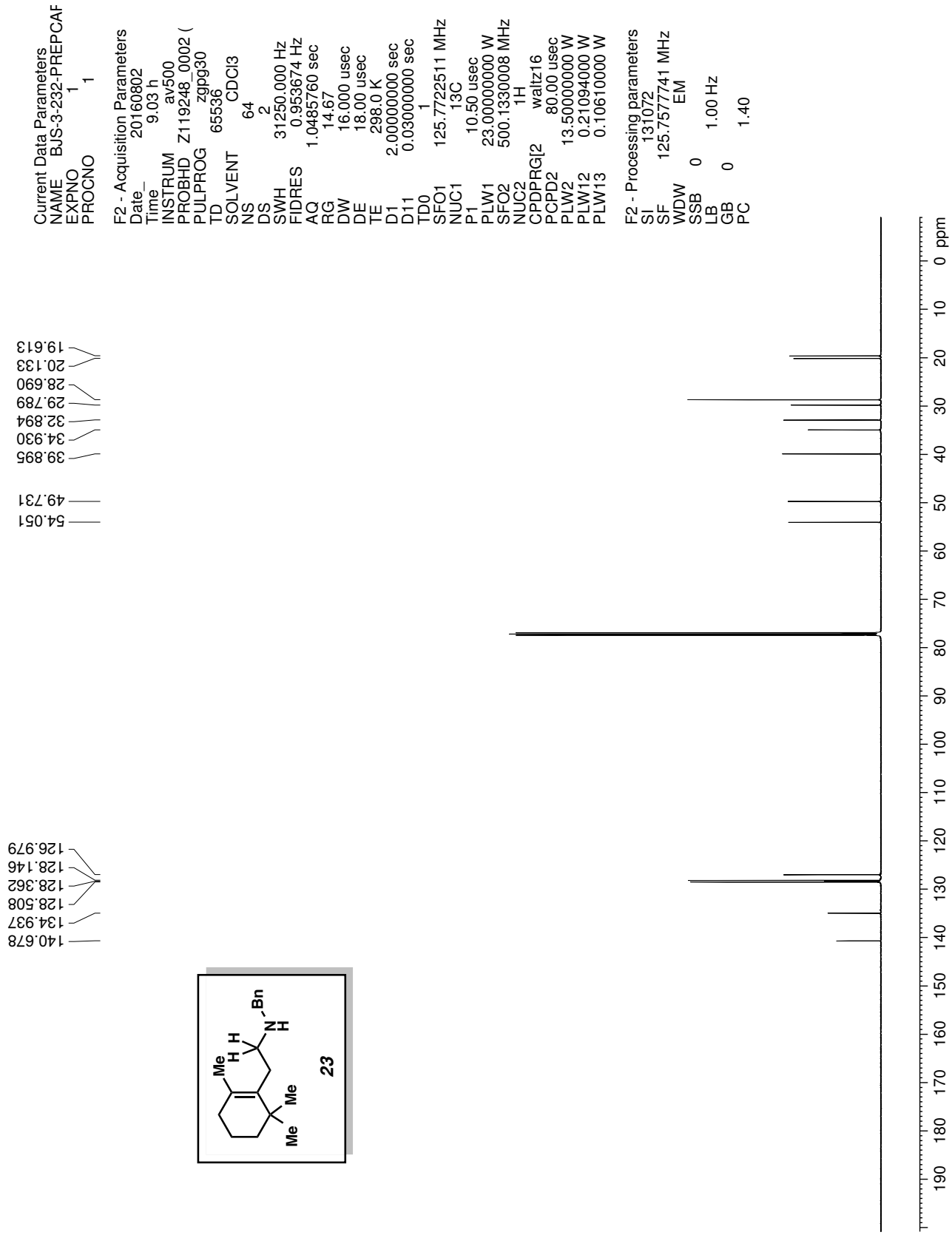

Current Data Parameters
NAME BJS-3-220-COLUMN-
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160718
Time 16.07 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 24
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 13.13
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 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 125.7577892 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
171.346
138.388
132.895
132.542
128.672
127.506
127.401
43.528
39.365
36.676
34.923
32.605
28.098
20.498
19.179
Me
O
N
H
Bn
Me
Me
SI-11
0 ppm

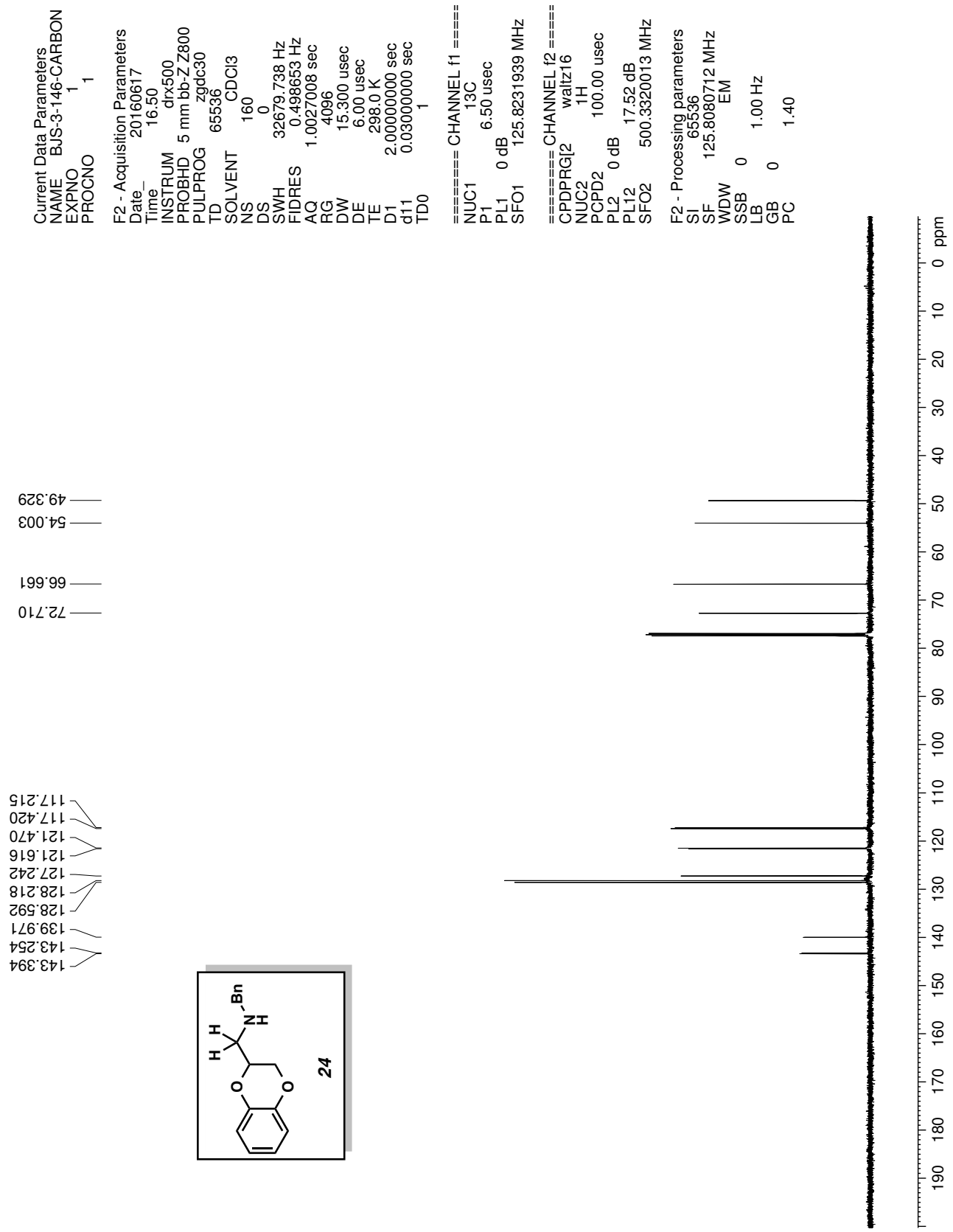

Current Data Parameters
NAME MH-2-222-pbis
EXPNO 191
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160927
Time 14.51
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 360
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976128 sec
RG 189.85
DW 19.800 usec
DE 6.50 usec
TE 299.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =====
SFO1 100.6243395 MHz
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
======== CHANNEL f2 =====
SFO2 400.1324008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.36111000 W
PLW13 0.29249999 W
F2 - Processing parameters
SI 65536
SF 100.6127613 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
20.875
21.030
21.808
24.121
27.447
28.685
31.173
31.810
36.228
36.972
45.692
47.180
55.529
55.777
64.017
64.674
126.211
126.250
128.555
141.357
141.457
170.667
170.936
171.385
171.555
OAc
O
N
SI-13 (rotamers)
ppm

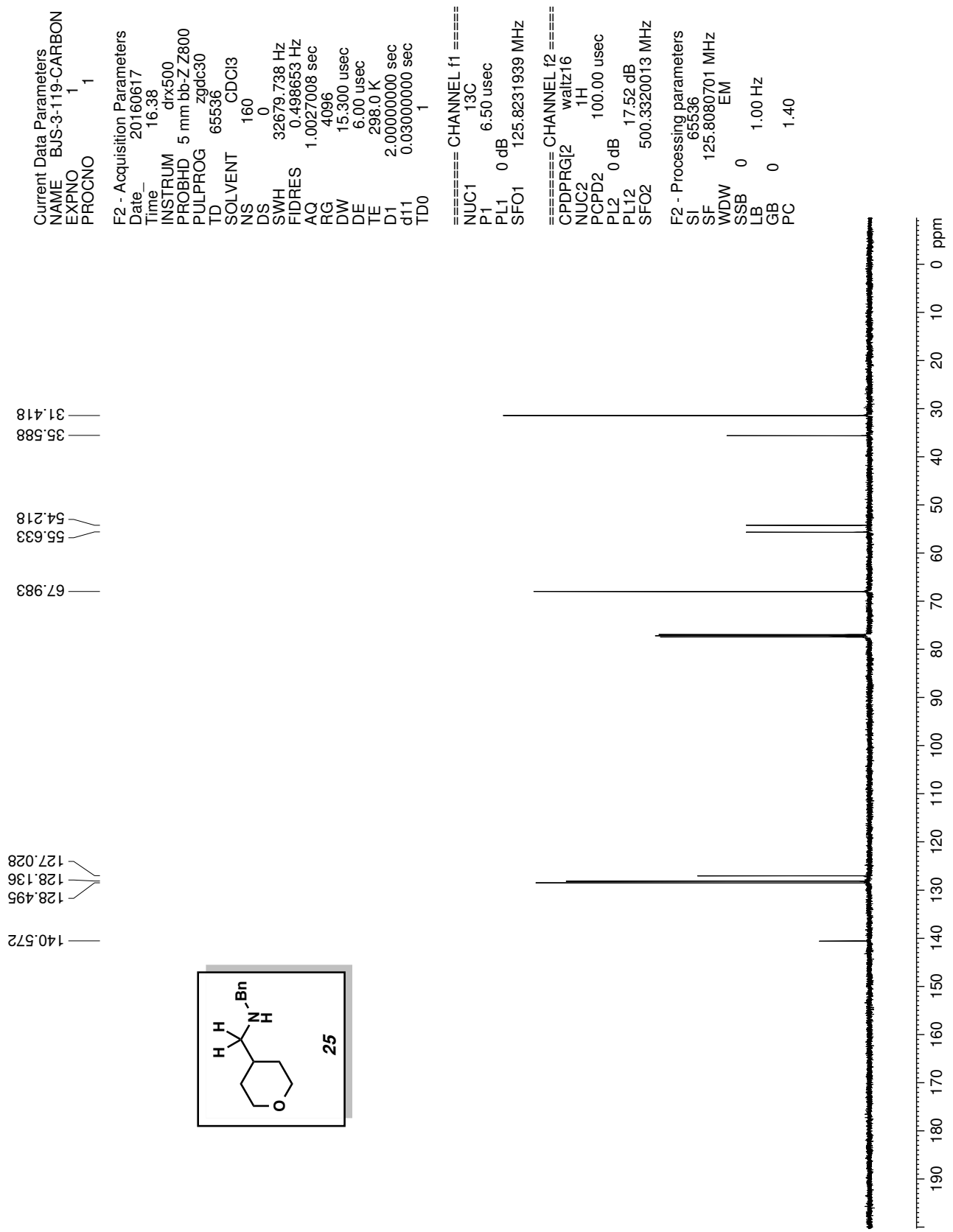

Current Data Parameters
NAME MOJ-1-179
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160329
Time 9.48 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 120
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 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 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577892 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
164.022
162.070
143.089
143.034
140.094
129.848
129.783
128.473
128.172
127.078
123.642
123.620
114.995
114.826
113.895
113.727
53.121
52.551
H
H
N
H
Bn
F
15
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

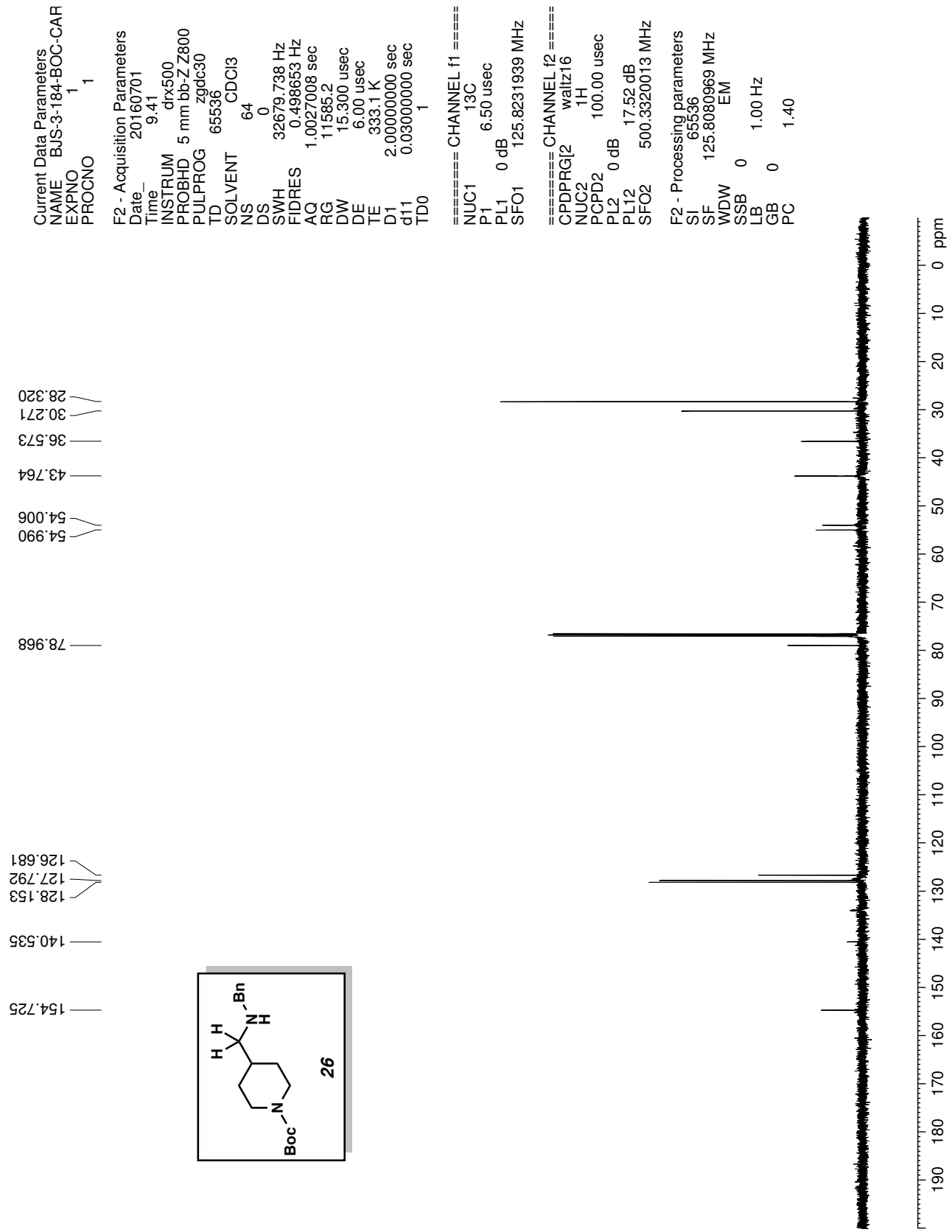

Current Data Parameters
NAME BJS-3-144-CARBON
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160617
Time 17.00
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 160
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027008 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 =====
NUC1 13C
P1 6.50 usec
PL1 0 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG[2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0 dB
PL12 17.52 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080716 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
53.545
52.888
140.024
135.257
131.070
128.542
128.446
128.065
127.124
124.707
122.886
120.473
111.205
102.530
16
ppm

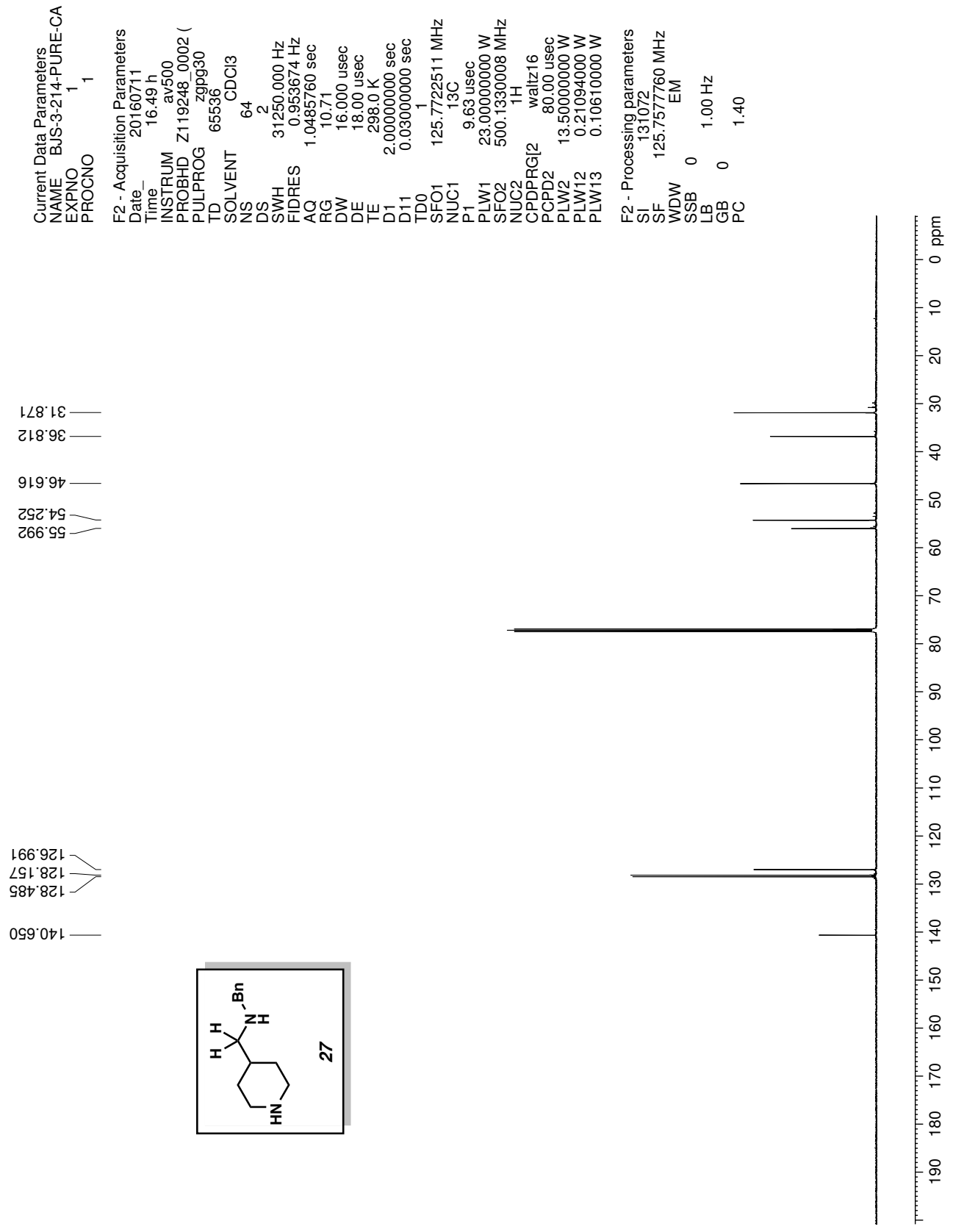

Current Data Parameters
NAME BJS-3-PYRAZ-CARB
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160617
Time 16.27
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 160
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027008 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 =====
NUC1 13C
P1 6.50 usec
PL1 0 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG[2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0 dB
PL12 17.52 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080642 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
140.286
138.988
129.076
128.572
128.309
127.125
120.631
53.328
43.256
39.011
Bn
Me
17
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

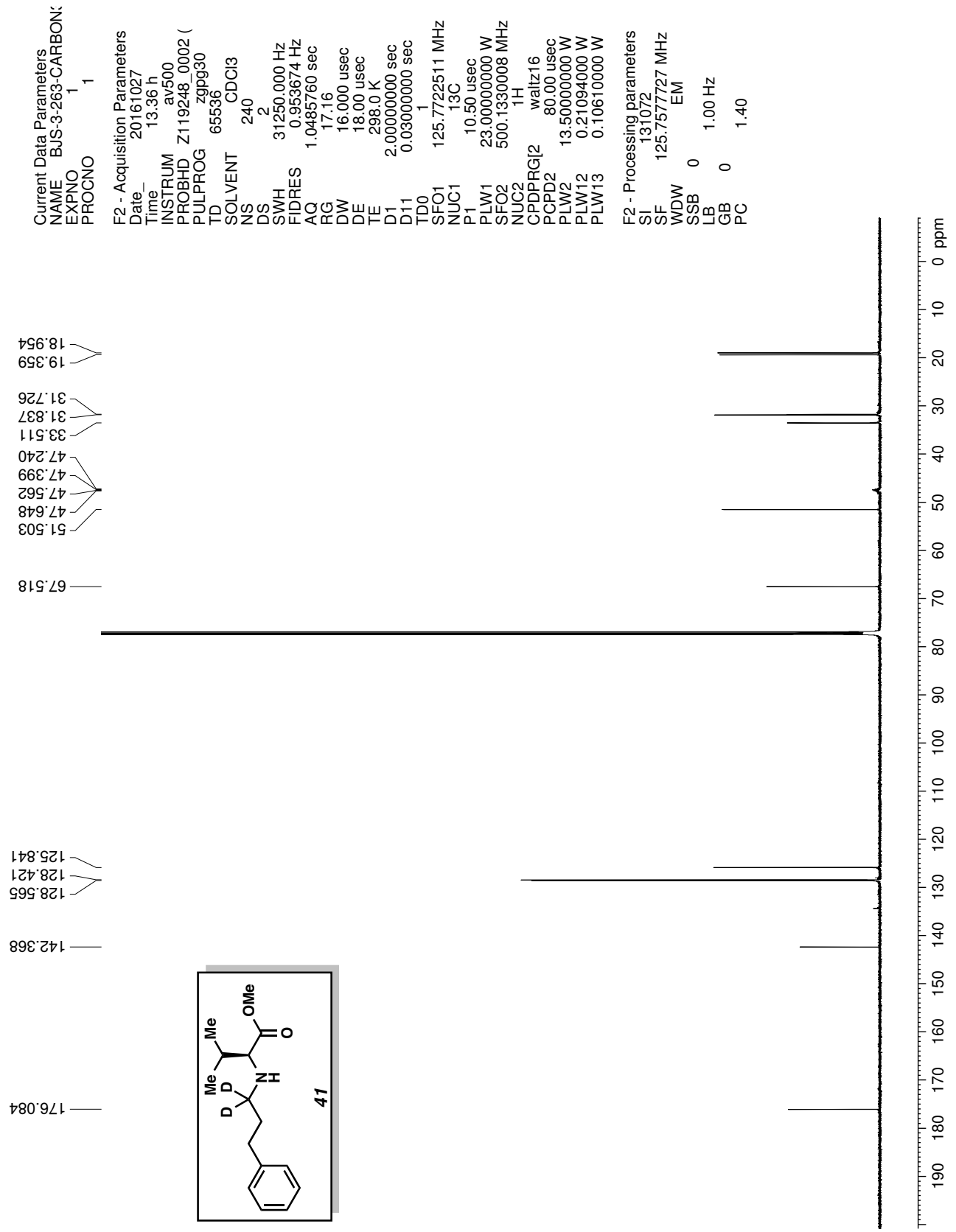

Current Data Parameters
NAME BJS-3-232-PREPCAF
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160802
Time 9.03 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 14.67
DW 16.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 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 125.7577741 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
140.678
134.937
128.508
128.362
128.146
126.979
54.051
49.731
39.895
34.930
32.894
29.789
28.690
20.133
19.613
Me
H
H
N
Bn
Me
Me
23
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

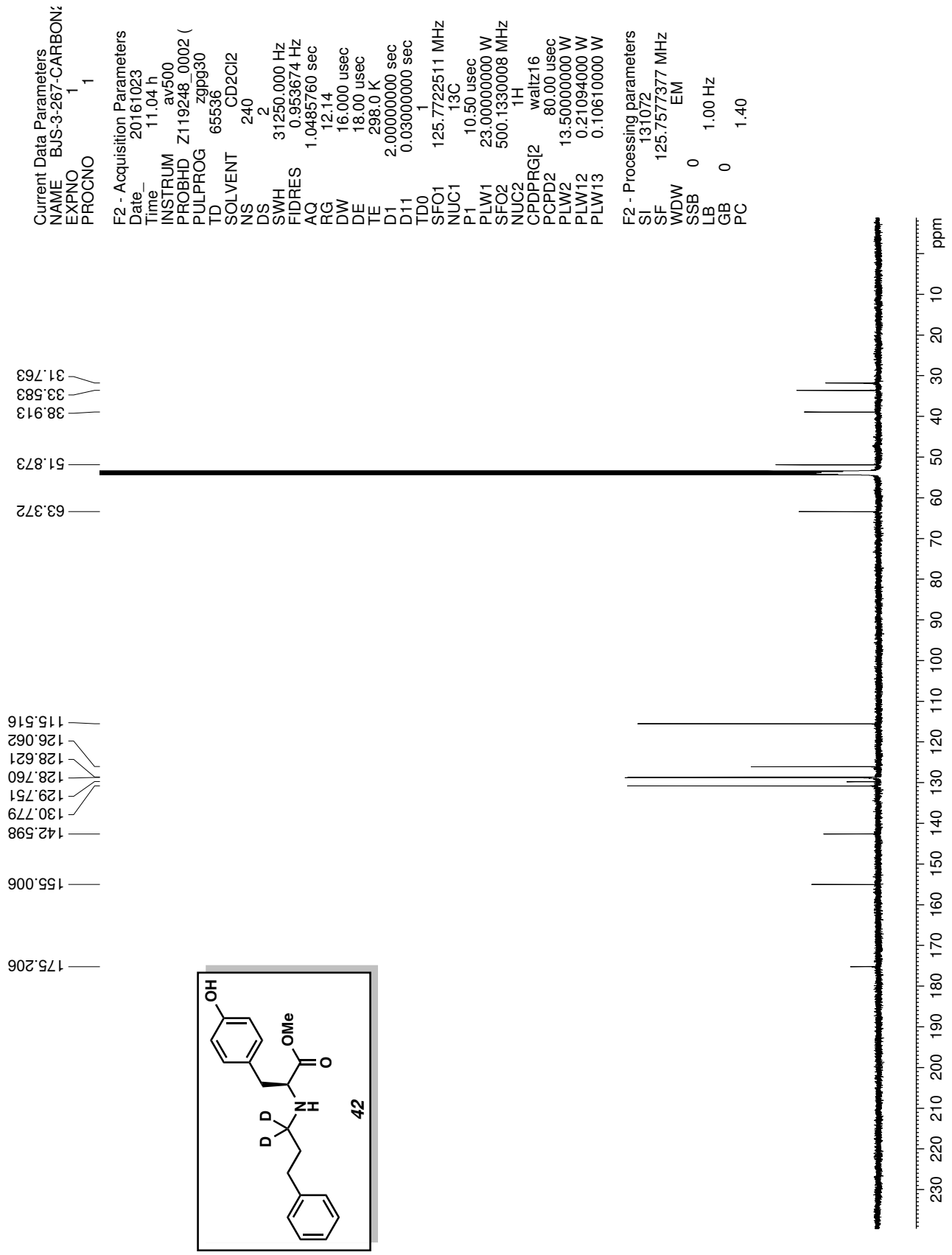

Current Data Parameters
NAME BJS-3-146-CARBON
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160617
Time 16.50
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 160
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027008 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 6.50 usec
PL1 0 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ======
CPDPRG[2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0 dB
PL12 17.52 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080712 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
143.394
143.254
139.971
128.592
128.218
127.242
121.616
121.470
117.420
117.215
72.710
66.661
54.003
49.329
24
0 ppm

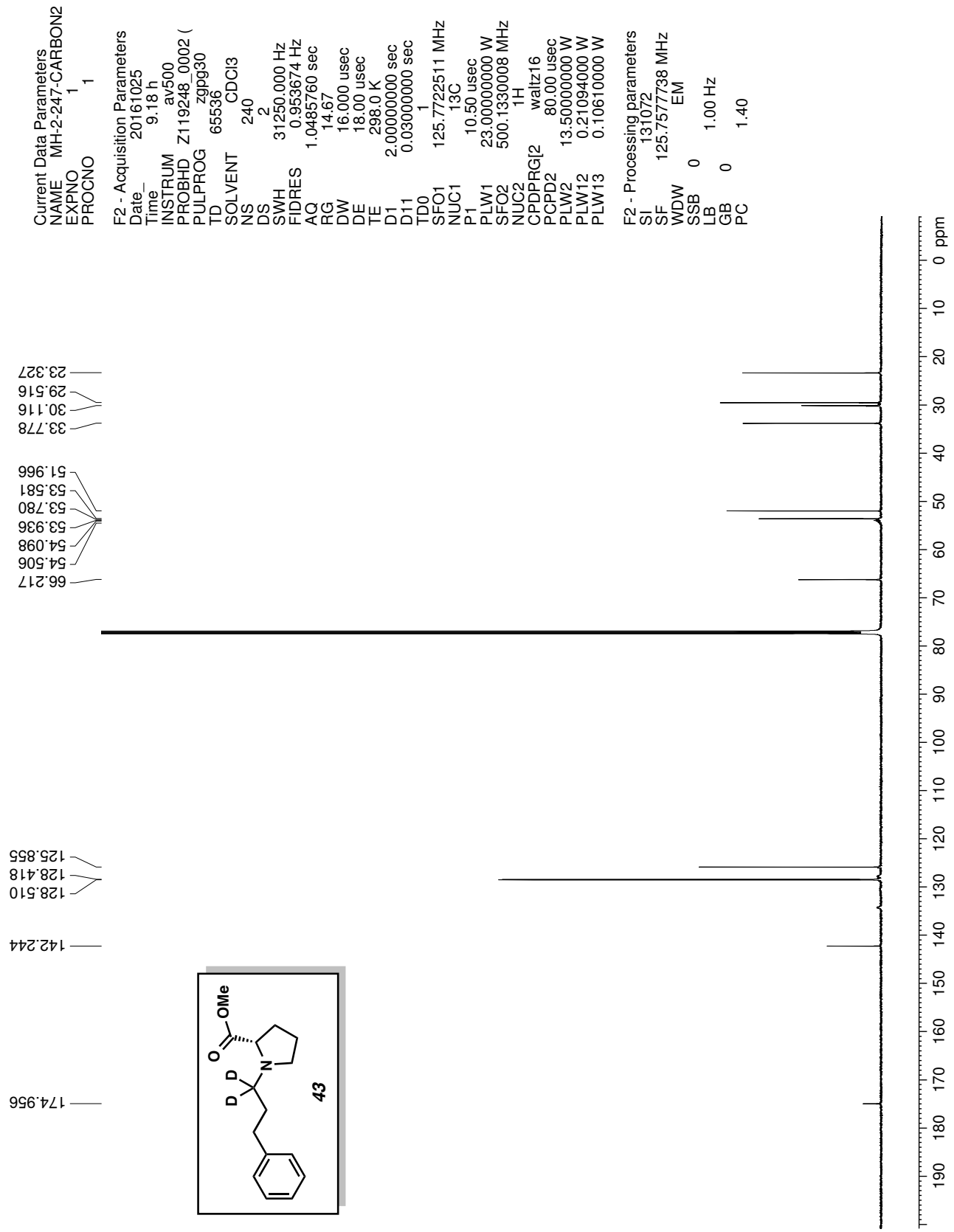

Current Data Parameters
NAME BJS-3-119-CARBON
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160617
Time 16.38
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 160
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027008 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 6.50 usec
PL1 0 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 ======
CPDPRG[2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0 dB
PL12 17.52 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080701 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
31.418
35.588
54.218
55.633
67.983
127.028
128.136
128.495
140.572
H
H
N
H
Bn
O
25
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

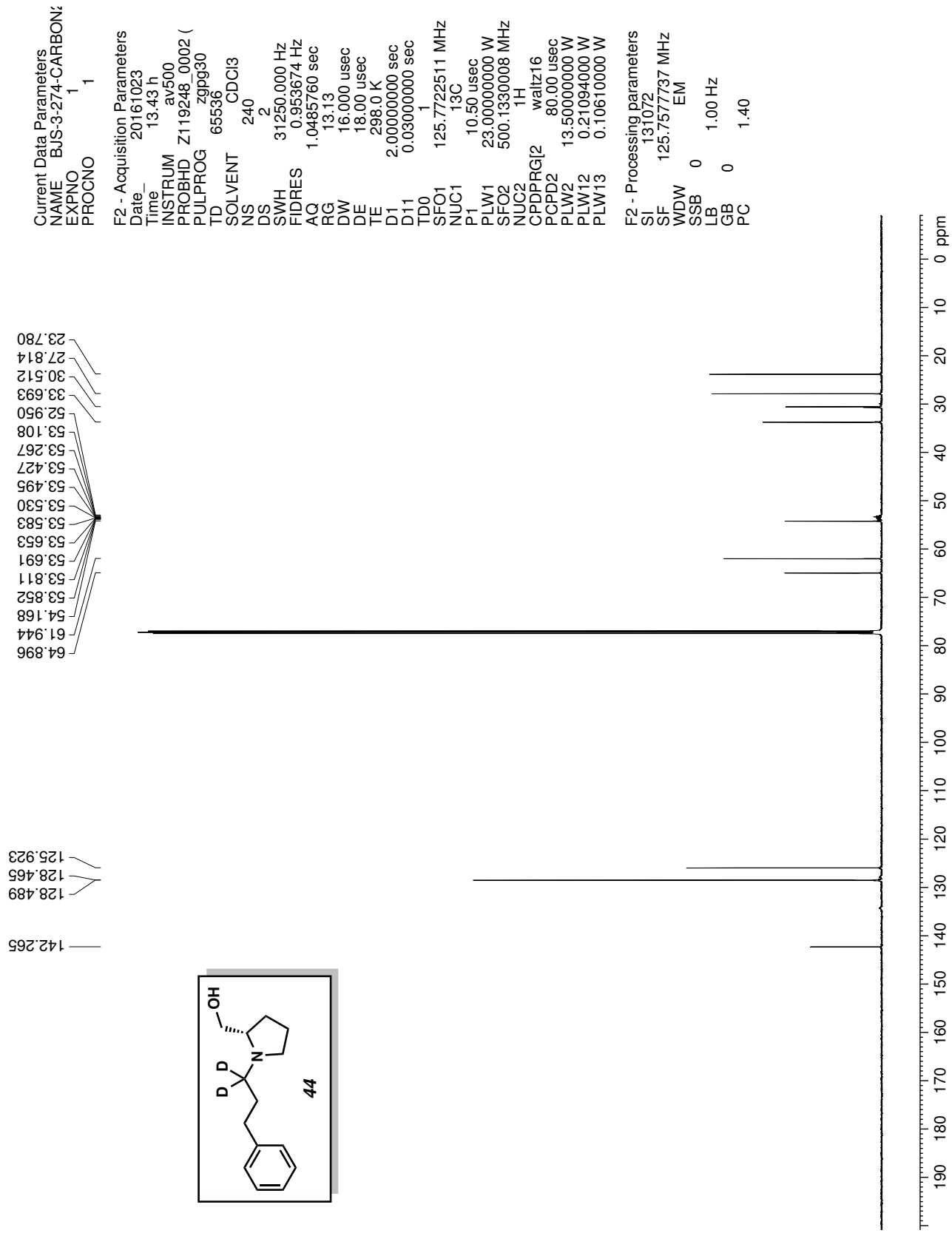

Current Data Parameters
NAME BJS-3-184-BOC-CAR
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160701
Time 9.41
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027008 sec
RG 11585.2
DW 15.300 usec
DE 6.00 usec
TE 333.1 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 =====
NUC1 13C
P1 6.50 usec
PL1 0 dB
SFO1 125.8231939 MHz
======== CHANNEL f2 =====
CPDPRG[2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0 dB
PL12 17.52 dB
SFO2 500.3320013 MHz
F2 - Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
154.725
140.535
128.153
127.792
126.681
78.968
54.990
54.006
43.764
36.573
30.271
28.320
Boc
N
H
H
N
Bn
26
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

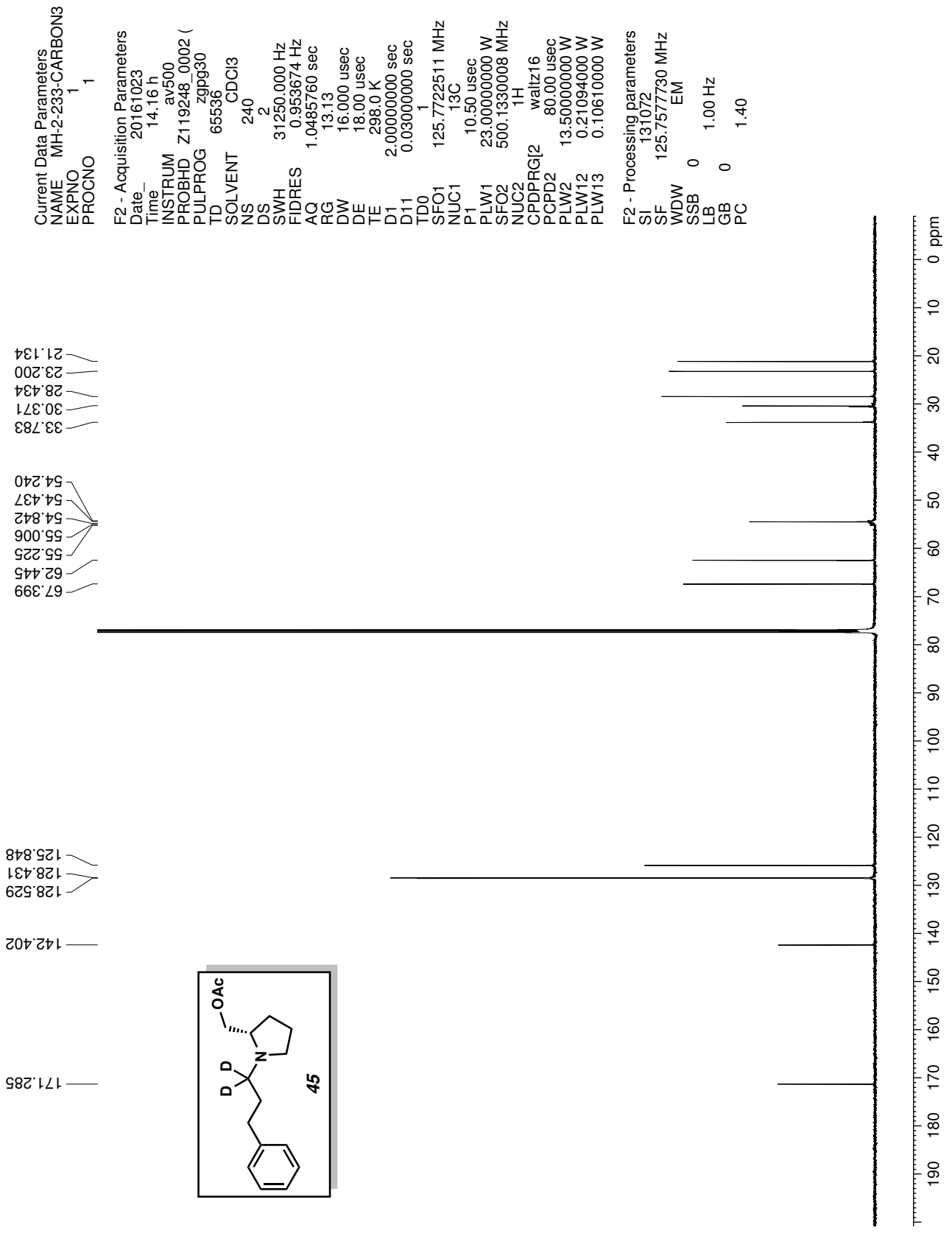

Current Data Parameters
NAME BJS-3-214-PURE-CA
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160711
Time 16.49 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 10.71
DW 16.000 usec
DE 18.00 usec
TE 298.0 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 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577760 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
31.871
36.812
46.616
54.252
55.992
128.485
128.157
126.991
140.650
HN
H
H
N
H
Bn
27
0 ppm

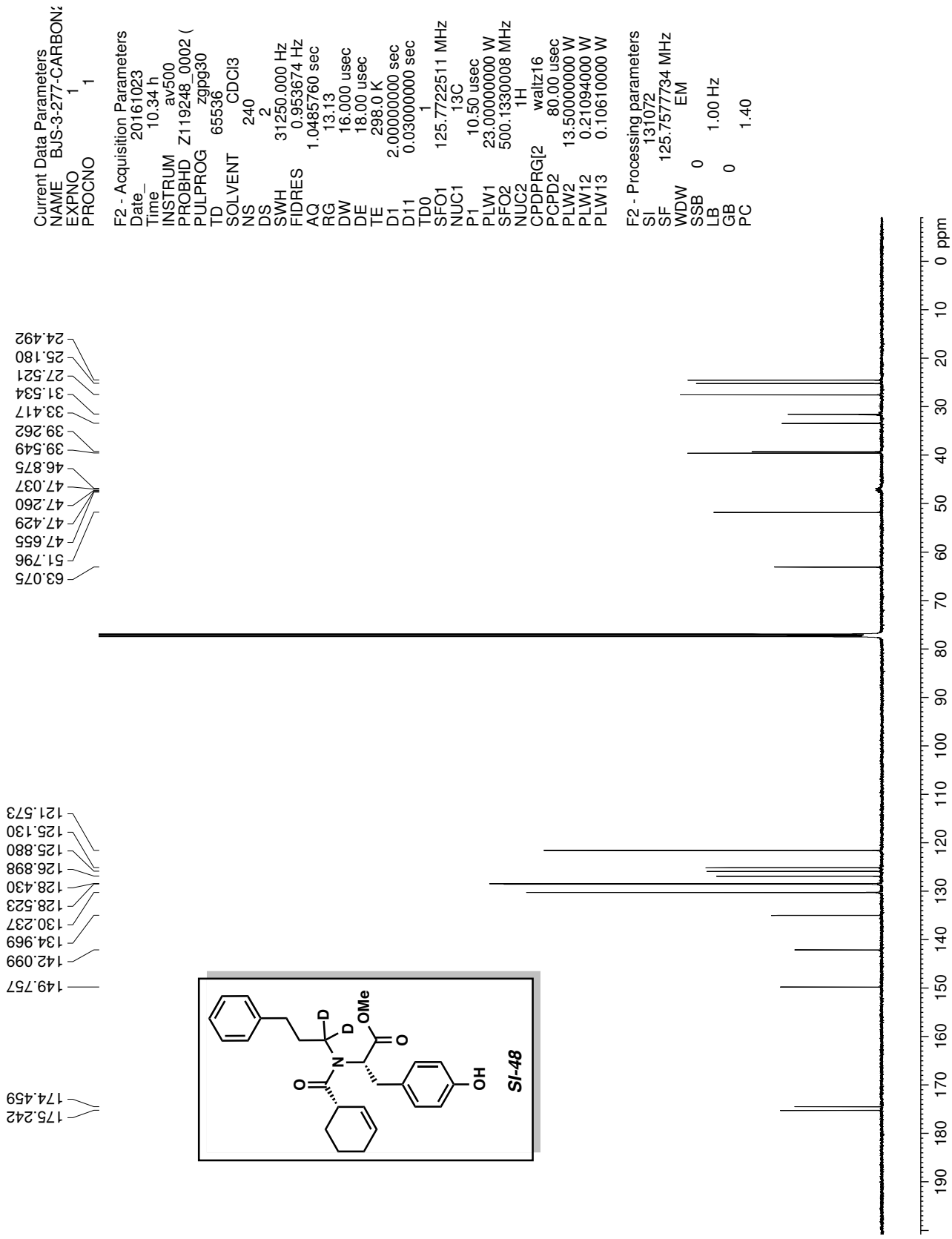

Current Data Parameters
NAME BJS-3-263-CARBON
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161027
Time 13.36 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 240
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 17.16
DW 16.000 usec
DE 18.00 usec
TE 298.0 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 125.7577727 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
176.084
142.368
128.565
128.421
125.841
67.518
51.503
47.648
47.562
47.399
47.240
33.511
31.837
31.726
19.359
18.954
Me
D
D
Me
N
H
O
OMe
41
0 ppm


Current Data Parameters
NAME BJS-3-267-CARBON
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161023
Time 11.04 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CD2Cl2
NS 240
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 12.14
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 125.7577377 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
175.206
155.006
142.598
130.779
129.751
128.760
128.621
126.062
115.516
63.372
51.873
38.913
33.583
31.763
ppm
OH
OMe
O
N
H
D
D
42


Current Data Parameters
NAME MH-2-247-CARBON2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161025
Time 9.18 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 240
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 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
174.956
142.244
128.510
128.418
125.855
66.217
54.506
54.098
53.936
53.780
53.581
51.966
33.778
30.116
29.516
23.327
OMe
O
N
D D
43
0 ppm


Current Data Parameters
NAME BJS-3-274-CARBON
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161023
Time 13.43 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 240
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 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 125.7577737 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
64.896
61.944
54.168
53.852
53.811
53.691
53.653
53.583
53.530
53.495
53.427
53.267
53.108
52.950
33.693
30.512
27.814
23.780
142.265
128.489
128.465
125.923
OH
N
D D
44
0 ppm


Current Data Parameters
NAME MH-2-233-CARBON3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161023
Time 14.16 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 240
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 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 125.7577730 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.134
23.200
28.434
30.371
33.783
54.240
54.437
54.842
55.006
55.225
62.445
67.399
125.848
128.431
128.529
142.402
171.285
OAc
D D
N
45
0 ppm


Current Data Parameters
NAME BJS-3-277-CARBON
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161023
Time 10.34 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 240
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 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 125.7577734 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
175.242
174.459
149.757
142.099
134.969
130.237
128.523
128.430
126.898
125.880
125.130
121.573
63.075
51.796
47.655
47.429
47.260
47.037
46.875
39.549
39.262
33.417
31.534
27.521
25.180
24.492
D
D
OMe
O
N
O
OH
SI-48
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm