Supporting Information – Table of Contents

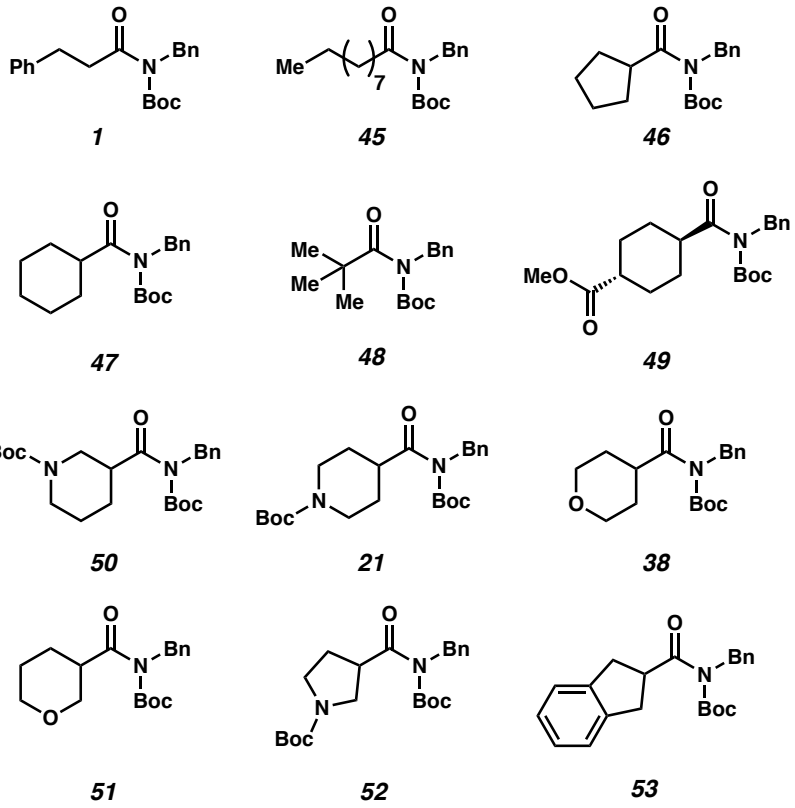
Materials and Methods. Unless stated otherwise, reactions were conducted in flame-dried glassware under an atmosphere of nitrogen or argon and commercially obtained reagents were used as received. Amide substrates were synthesized following protocols specified in Section A in the Experimental Procedures. Alcohol **7** was prepared according to literature procedure.[1] Boronate esters **5**, **59**–**61**, **63**–**65**, **67** and **68** were obtained from Combi-Blocks. Boronate ester **6** was obtained from TCI Chemicals. Boronate ester **39** was obtained from AK Scientific. Boronate esters **62** and **66** were prepared according to literature procedure.[2] $Ni(cod)_2$ and Benz-ICy•HCl (**2**) were obtained from Strem Chemicals. [(TMEDA)Ni(*o*-tolyl)Cl] was prepared according to literature procedure.[3] Ligand A (**71**) was prepared according to literature procedure.[4] Potassium phosphate (K_3PO_4) was obtained from Acros. 1,4-dioxane was obtained from Fisher Scientific and purified by distillation over sodium metal degassed by sparging with N_2 for 1 h. Paraffin wax (mp 53–57 °C ASTM D 87) was obtained from Sigma-Aldrich and used as received). 1,3,5-trimethoxybenzene was obtained from Alfa Aesar and used as received. Reaction temperatures were controlled using an IKAmag temperature modulator, and unless stated otherwise, reactions were performed at room temperature (approximately 23 °C). Thin-layer chromatography (TLC) was conducted with EMD gel 60 F254 pre-coated plates (0.25 mm for analytical chromatography and 0.50 mm for preparative chromatography) and visualized using a combination of UV, anisaldehyde, iodine, and potassium permanganate staining techniques. Silicycle Siliaflash P60 (particle size 0.040–0.063 mm) was used for flash column chromatography. ^{1}H NMR spectra were recorded on Bruker spectrometers (400, 500, and 600 MHz were allowed for our provided spectra) and are reported relative to residual solvent signals. Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz), integration. Data for ^{13}C NMR are reported in terms of chemical shift (at 125 MHz). IR spectra were recorded on a Perkin-Elmer UATR Two FT-IR spectrometer and are reported in terms of frequency absorption (cm^{-1}). DART-MS spectra were collected on a Thermo Exactive Plus MSD (Thermo Scientific) equipped with an ID-CUBE ion source and a Vapur Interface (IonSense Inc.). Both the source and MSD were controlled by Excalibur software v. 3.0. The analyte was spotted onto OpenSpot sampling cards (IonSense Inc.) using $CHCl_3$ or CH_2Cl_2 as the solvent. Ionization was accomplished using UHP He plasma with no additional ionization agents. The mass calibration was carried out using Pierce LTQ Velos ESI (+) and (–) Ion calibration solutions (Thermo Fisher Scientific). Determination of enantiopurity was carried out on a Mettler

Toledo SFC (supercritical fluid chromatography) using Daicel ChiralPak AD–H columnn. Data for SFC are reported in enantiomeric excess (ee). For SFC chromatograms see Section J of Experimental Procedures.

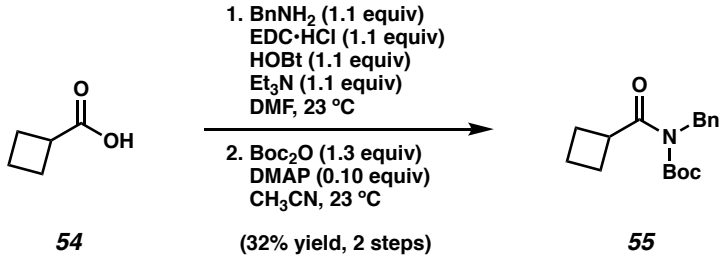
Experimental Procedures

A. Syntheses of Amide Substrates

Supporting information for the syntheses of amides **1**,[5] **45**–**48**,[5] **21**,[6] **38**,[6] **49**–**52**,[6] and **53**[7] have been published and spectral data match those previously reported.



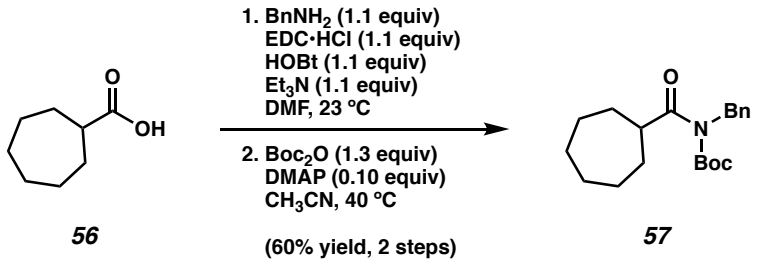
Syntheses for the remaining substrates shown in Figures 4 and 6 are as follows:



To a mixture of carboxylic acid **54** (65.0 mg, 0.650 mmol, 1.00 equiv), EDC•HCl (137 mg, 0.0720 mmol, 1.10 equiv), HOBt (109 g, 0.710 mmol, 1.10 equiv), triethylamine (0.100 mL, 0.710 mmol, 1.10 equiv), and DMF (5.00 mL, 0.130 M) was added benzylamine (78.0 μL, 0.710

mmol, 1.10 equiv). The resulting mixture was stirred at 23 ºC for 17 h, and then diluted with deionized water (5 mL) and transferred to a separatory funnel with brine (5 mL). The aqueous layer was extracted with EtOAc (3 x 10 mL), then the organic layers were combined and washed with deionized water (3 x 10 mL), dried over Na_2SO_4, and evaporated under reduced pressure. The resulting crude material was used in the subsequent step without further purification.

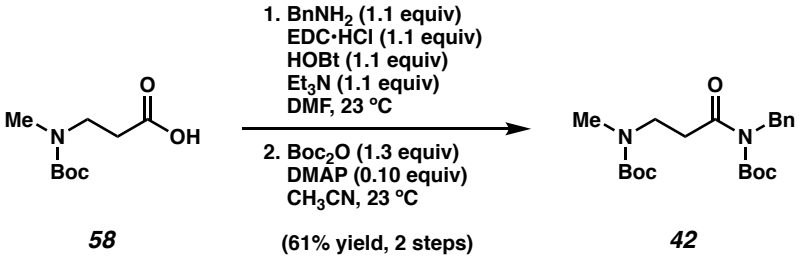
To a flask containing the crude material from the previous step was added DMAP (8.00 mg, 0.0650 mmol, 0.100 equiv) followed by acetonitrile (4.00 mL, 0.160 M). Boc_2O (184 g, 0.850 mmol, 1.30 equiv) was added in one portion and the reaction vessel was flushed with N_2, then the reaction mixture was allowed to stir at 23 ºC for 20 h. The reaction was quenched by addition of saturated aqueous $NaHCO_3$ (5 mL), transferred to a separatory funnel with EtOAc (10 mL) and H_2O (10 mL), and extracted with EtOAc (3 x 10 mL). The organic layers were combined, dried over Na_2SO_4, and evaporated under reduced pressure. The resulting crude residue was purified by flash column chromatography (29:1 Hexanes:EtOAc) to yield amide **55** (60.5 mg, 32% yield, over two steps) as a clear oil. Amide **55**: R_f 0.65 (5:1 Hexanes:EtOAc); ^{1}H NMR (600 MHz, C_6D_6): δ 7.30 (d, J = 7.5, 2H), 7.11 (t, J = 7.5, 2H), 7.03 (t, J = 7.5, 1H), 4.88 (s, 2H), 4.05 (quint, J = 8.3, 1H), 2.54–2.43 (m, 2H), 2.30–2.18 (m, 2H), 1.82–1.67 (m, 2H), 1.17 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 178.0, 152.8, 138.6, 128.4, 127.6, 127.1, 83.0, 47.6, 41.4, 28.0, 25.8, 17.9; IR (film): 2980, 2869, 1732, 1687, 1144, 980 cm^{-1}; HRMS-APCI (*m/z*) [M + H]$^+$ calcd for $C_{17}H_{24}NO_3^+$, 290.17507; found 290.17377.



To a mixture of carboxylic acid **56** (1.00 g, 7.03 mmol, 1.00 equiv), EDC•HCl (1.48 g, 7.74 mmol, 1.10 equiv), HOBt (1.18 g, 7.74 mmol, 1.10 equiv), triethylamine (1.10 mL, 7.74 mmol, 1.10 equiv), and DMF (70 mL, 0.10 M) was added benzylamine (0.850 mL, 7.740 mmol, 1.10 equiv). The resulting mixture was stirred at 23 ºC for 20 h, and then diluted with deionized

water (100 mL) and transferred to a separatory funnel with EtOAc (30 mL) and brine (15 mL). The aqueous layer was extracted with EtOAc (3 x 100 mL), then the organic layers were combined and washed with deionized water (4 x 100 mL), dried over Na_2SO_4, and evaporated under reduced pressure. The resulting crude solid material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (85.9 mg, 0.703 mmol, 0.100 equiv) followed by acetonitrile (35.0 mL, 0.200 M). Boc_2O (1.99 g, 9.14 mmol, 1.30 equiv) was added in one portion and the reaction vessel was flushed with N_2, then the reaction mixture was allowed to stir at 40 ºC for 16 h. The reaction was quenched by addition of saturated aqueous $NaHCO_3$ (100 mL), transferred to a separatory funnel with EtOAc (20 mL) and extracted with EtOAc (3 x 40 mL). The organic layers were combined, dried over Na_2SO_4, and evaporated under reduced pressure. The resulting crude residue was purified by flash chromatography (99:1 Hexanes:EtOAc → 9:1 Hexanes:EtOAc) to yield amide **57** as a white crystalline powder. Hot recrystallization of the purified product from *n*-heptane gave the recrystallized material (1.41 g, 60% yield over two steps) as white crystals. Amide **57**: mp: 57.7–62.8 ºC; R_f 0.57 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32–7.27 (m, 2H), 7.25–7.20 (m, 3H), 4.85 (s, 2H), 3.59 (tt, *J* = 9.7, 3.9, 1H), 1.97–1.87 (m, 2H), 1.81–1.70 (m, 2H), 1.70–1.44 (m, 8H), 1.40 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 180.8, 153.3, 138.7, 128.4, 127.6, 127.1, 83.0, 47.8, 45.8, 31.9, 28.4, 28.0, 26.7; IR (film): 2977, 2928, 2858, 1734, 1694, 1369, 1148 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{20}H_{30}NO_3^+$, 332.22202; found 332.22098.



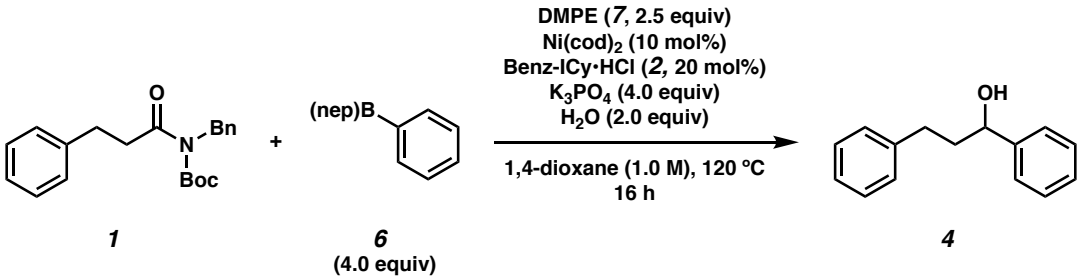
To a mixture of carboxylic acid **58** (500 mg, 2.46 mmol, 1.00 equiv), EDC•HCl (519 mg, 2.71 mmol, 1.10 equiv), HOBt (414 mg, 2.71 mmol, 1.10 equiv), triethylamine (0.380 mL, 2.71

mmol, 1.10 equiv), and DMF (25.0 mL, 0.100 M) was added benzylamine (0.300 mL, 2.71 mmol, 1.10 equiv). The resulting mixture was stirred at 23 ºC for 23 h, and then diluted with deionized water (100 mL) and transferred to a separatory funnel with EtOAc (30 mL) and brine (15 mL). The aqueous layer was extracted with EtOAc (3 x 80 mL), then the organic layers were combined and washed with deionized water (4 x 80 mL), dried over Na_2SO_4, and evaporated under reduced pressure. The resulting crude solid material was used in the subsequent step without further purification.

To a flask containing the crude material from the previous step was added DMAP (28.9 mg, 0.236 mmol, 0.100 equiv) followed by acetonitrile (12.0 mL, 0.200 M). Boc_2O (671 mg, 3.07 mmol, 1.30 equiv) was added in one portion and the reaction vessel was flushed with N_2, then the reaction mixture was allowed to stir at 23 ºC for 17 h. The reaction was quenched by addition of saturated aqueous $NaHCO_3$ (10 mL), transferred to a separatory funnel with EtOAc (20 mL) and extracted with EtOAc (3 x 40 mL). The organic layers were combined, dried over Na_2SO_4, and evaporated under reduced pressure. The resulting crude residue was purified by flash chromatography (19:1 Hexanes:EtOAc → 9:1 Hexanes:EtOAc) to yield amide **42** (0.57 g, 61% yield over two steps) as a clear oil. Amide **42**: R_f 0.38 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33–7.17 (m, 5H), 4.87 (br s, 2H), 3.55 (s, 2H), 3.15 (br s, 2H), 2.86 (br s, 3H), 1.45 (s, 9H), 1.40 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.4, 155.7, 153.1, 138.3, 128.4, 127.6, 127.3, 83.5, 79.6, 79.5, 47.4, 45.6, 45.3, 37.2, 36.6, 35.0, 34.8, 28.6, 28.0; IR (film): 2977, 1734, 1691, 1368, 1145 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{21}H_{33}N_2O_5^+$, 393.23840; found 393.23730.

*Note: **42** was obtained as a mixture of rotamers. These data represent empirically observed chemical shifts from the ^{1}H NMR and ^{13}C NMR spectra.*

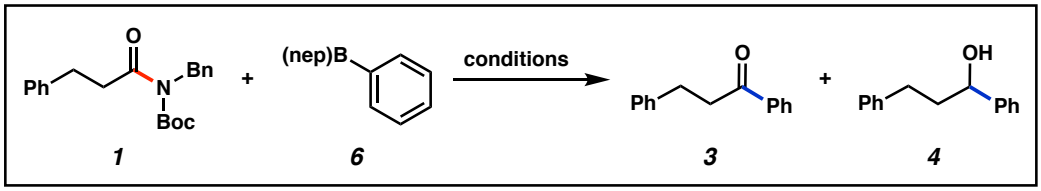
B. Relevant Control Experiments



Representative Procedure for Conversion of Aliphatic Amides to Secondary Alcohols from Figure 3 (amide 1 and boronate ester 6 used as an example). A 1-dram vial was charged with anhydrous powder K_3PO_4 (170 mg, 0.800 mmol, 4.00 equiv) and a magnetic stir bar. The vial and its contents were flame-dried under reduced pressure and allowed to cool under N_2. Amide substrate **1** (67.9 mg, 0.200 mmol, 1.00 equiv), boronate ester nucleophile **6** (152 mg, 0.800 mmol, 4.00 equiv), and DMPE (**7**, 82.6 mg, 0.500 mmol, 2.50 equiv) were added. The vial was flushed with N_2 for 5 min, then water (7.21 μL, 0.400 mmol, 2.00 equiv), which had been sparged with N_2 for 10 min, was added. The vial was taken into a glovebox and charged with $Ni(cod)_2$ (5.50 mg, 0.0200 mmol, 10 mol%) and Benz-ICy•HCl (**2**, 12.8 mg, 0.0400 mmol, 20 mol%). Subsequently, 1,4-dioxane (200 μL, 1.00 M) was added. The vial was sealed with a Teflon-lined screw cap, removed from the glovebox, and stirred vigorously (800 RPM) at 120 ºC for 16 h. After cooling to 23 ºC, the mixture was quenched by the addition of saturated aqueous NH_4Cl (1 mL) and extracted with EtOAc (3 x 2 mL). The combined organic layers were then filtered over a plug of silica gel (3 cm) and Na_2SO_4 (3 cm) using EtOAc (10 mL) as eluent. The volatiles were removed under pressure and the yield of alcohol **4** was determined by ^1H NMR analysis with 1,3,5-trimethoxybenzene as an external standard.

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

Table S1. Relevant Control Experiments



1 *6* *3* *4*

Reaction Conditions	*Experimental Results*[a] *1*	*3*	*4*
6 **(4.0 equiv), DMPE (*7*, 4.0 equiv), K_3PO_4 (4.0 equiv), H_2O (2.0 equiv) 1,4-dioxane (1.0 M), 120 °C, 16 h**	**5%**[b]	**0%**	**0%**
6 **(4.0 equiv), DMPE (*7*, 4.0 equiv), K_3PO_4 (4.0 equiv), H_2O (2.0 equiv) Benz-ICy·HCl (*2*, 20 mol%), 1,4-dioxane (1.0 M), 120 °C, 16 h**	**0%**[b]	**0%**	**0%**
6 **(4.0 equiv), DMPE (*7*, 4.0 equiv), K_3PO_4 (4.0 equiv), H_2O (2.0 equiv) $Ni(cod)_2$ (10 mol%), 1,4-dioxane (1.0 M), 120 °C, 16 h**	**12%**[b]	**0%**	**0%**

[a] **Yields determined by ^{1}H NMR analysis using 1,3,5-trimethoxybenzene as an external standard.**
[b] **Substantial amounts of the corresponding Boc-cleavage product (des-Boc amide starting material) were observed due to the elevated reaction temperature.**

C. General Procedures for Methodology

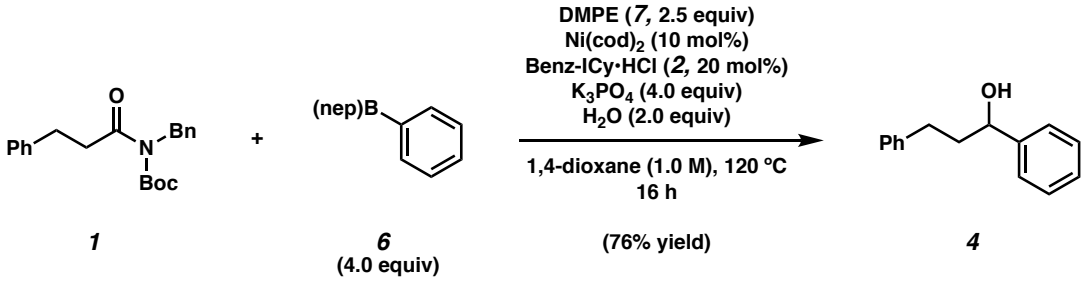
General Procedure A. A 1-dram vial was charged with anhydrous powder K_3PO_4 (170 mg, 8.00 mmol, 4.00 equiv) and a magnetic stir bar. The vial and its contents were flame-dried under reduced pressure and allowed to cool under N_2. Amide substrate (0.200 mmol, 1.00 equiv), boronate ester nucleophile (152 mg, 0.800 mmol, 4.00 equiv), and DMPE (**7**, 82.6 mg, 0.500 mmol, 2.50 equiv) were added. The vial was flushed with N_2 for 5 min, then water (7.21 µL, 0.400 mmol, 2.00 equiv), which had been sparged with N_2 for 10 min, was added. The vial was taken into a glovebox and charged with $Ni(cod)_2$ (5.50 mg, 0.0200 mmol, 10 mol%) and Benz-ICy•HCl (**2**, 12.8 mg, 0.0400 mmol, 20 mol%). Subsequently, 1,4-dioxane (200 µL, 1.00 M) was added. The vial was sealed with a Teflon-lined screw cap, removed from the glovebox, and stirred vigorously (800 RPM) at 120 ºC for 16 h. After cooling to 23 ºC, the mixture was quenched by the addition of saturated aqueous NH_4Cl (1 mL) and extracted with EtOAc (3 x 2 mL). The combined organic layers were then filtered over a plug of silica gel (3 cm) and Na_2SO_4 (3 cm) using EtOAc (10 mL) as eluent and the volatiles were removed under pressure. The crude mixture was adsorbed onto silica gel (450 mg) under reduced pressure and purified by flash column chromatography on silica.

General Procedure B. A 1-dram vial was charged with anhydrous powder K_3PO_4 (170 mg, 8.00 mmol, 4.00 equiv) and a magnetic stir bar. The vial and its contents were flame-dried under reduced pressure and allowed to cool under N_2. Amide substrate (0.200 mmol, 1.00 equiv), boronate ester nucleophile (152 mg, 0.800 mmol, 4.00 equiv), and DMPE (**7**, 82.6 mg, 0.500 mmol, 2.50 equiv) were added. The vial was flushed with N_2 for 5 min, then water (7.21 μL, 0.400 mmol, 2.00 equiv), which had been sparged with N_2 for 10 min, was added. The vial was taken into a glovebox and charged with $Ni(cod)_2$ (5.50 mg, 0.0200 mmol, 10 mol%) and Benz-ICy•HCl (**2**, 12.8 mg, 0.0400 mmol, 20 mol%). Subsequently, 1,4-dioxane (200 μL, 1.00 M) was added. The vial was sealed with a Teflon-lined screw cap, removed from the glovebox, and stirred vigorously (800 RPM) at 120 ºC for 16 h. After cooling to 23 ºC, the mixture was diluted with CH_2Cl_2 (1 mL) and washed with 2 M HCl (3 x 1 mL). The organic layer was then filtered over a plug of silica gel (3 cm) and Na_2SO_4 (3 cm) using EtOAc (10 mL) as eluent and the volatiles were removed under pressure. The crude mixture was adsorbed onto silica gel (450 mg) under reduced pressure and purified by flash column chromatography on silica.

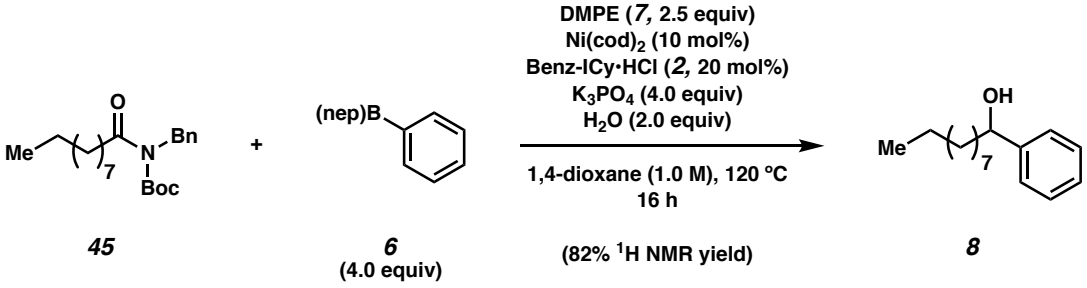
General Procedure C. A 1-dram vial was charged with anhydrous powder K_3PO_4 (170 mg, 8.00 mmol, 4.00 equiv) and a magnetic stir bar. The vial and its contents were flame-dried under reduced pressure and allowed to cool under N_2. Amide substrate (0.200 mmol, 1.00 equiv), boronate ester nucleophile (228 mg, 1.20 mmol, 6.00 equiv), and DMPE (**7**, 82.6 mg, 0.500 mmol, 2.50 equiv) were added. The vial was flushed with N_2 for 5 min, then water (7.21 μL, 0.400 mmol, 2.00 equiv), which had been sparged with N_2 for 10 min, was added. The vial was taken into a glovebox and charged with $Ni(cod)_2$ (11.0 mg, 0.0400 mmol, 20 mol%) and Benz-ICy•HCl (**2**, 25.6 mg, 0.0800 mmol, 40 mol%). Subsequently, 1,4-dioxane (200 μL, 1.00 M) was added. The vial was sealed with a Teflon-lined screw cap, removed from the glovebox, and stirred vigorously (800 RPM) at 120 ºC for 16 h. After cooling to 23 ºC, the mixture was diluted with CH_2Cl_2 (1 mL) and washed with deionized H_2O (3 x 1 mL). The organic layer was then filtered over a plug of silica gel (3 cm) and Na_2SO_4 (3 cm) using EtOAc (10 mL) as eluent and the volatiles were removed under pressure. The crude mixture was adsorbed onto silica gel (450 mg) under reduced pressure and purified by flash column chromatography on silica.

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

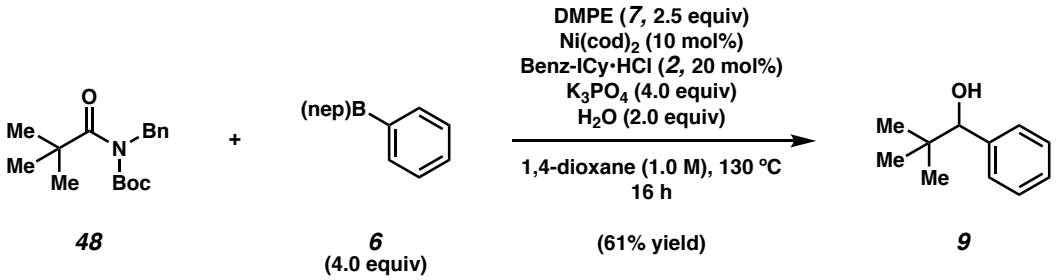
D. Scope of Amide Substrates



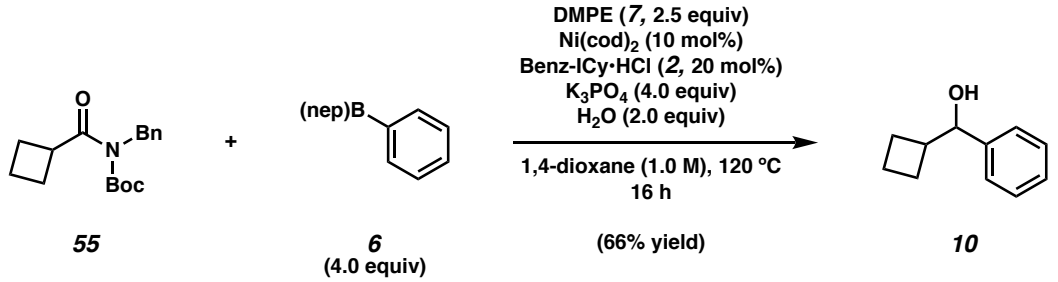
Alcohol 4. Crude alcohol **4** was synthesized following General Procedure A. Purification by flash column chromatography (99:1 Hexanes:EtOAc → 19:1 Hexanes:EtOAc) afforded alcohol **4** (76% yield, average of two experiments) as a white solid. Alcohol **4**: R_f 0.34 (5:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.40–7.34 (m, 4H), 7.32–7.27 (m, 3H), 7.23–7.16 (m, 3H), 4.70 (app t, *J* = 6.5, 1H), 2.76 (ddd, *J* = 13.9, 10.0, 5.8, 1H), 2.68 (ddd, *J* = 13.9, 9.6, 6.4, 1H), 2.21–1.98 (m, 2H), 1.92 (br s, 1H). Spectral data match those previously reported.[8]



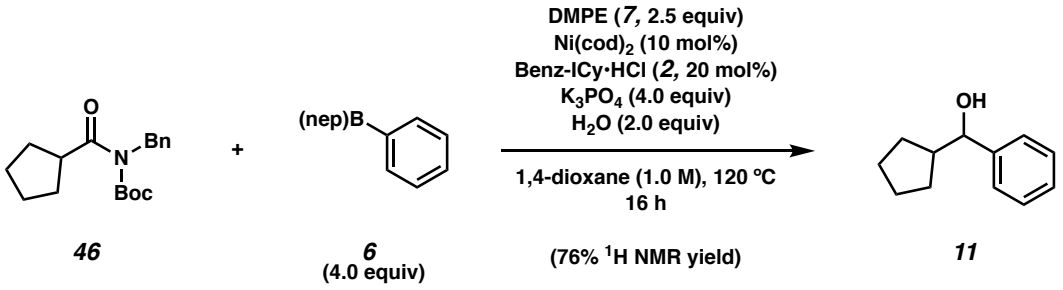
Alcohol 8. Crude alcohol **8** was synthesized following General Procedure A. ^{1}H NMR analysis of the crude reaction mixture indicated an 82% yield of alcohol **8** relative to 1,3,5-trimethoxybenzene external standard. Sequential purification by preparative thin-layer chromatography (3:1 Hexanes:Et_2O, then 4:1 Hexanes:Acetone) provided an analytical sample of alcohol **8** as a clear oil. Alcohol **8**: R_f 0.52 (5:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.41–7.31 (m, 4H), 7.31–7.27 (m, 1H), 4.67 (dd, *J* = 7.5, 5.9, 1H), 1.87–1.75 (m, 2H), 1.75–1.65 (m, 1H), 1.49–1.36 (m, 1H), 1.36–1.16 (m, 13H), 0.87 (t, *J* = 6.8, 3H). Spectral data match those previously reported.[9]



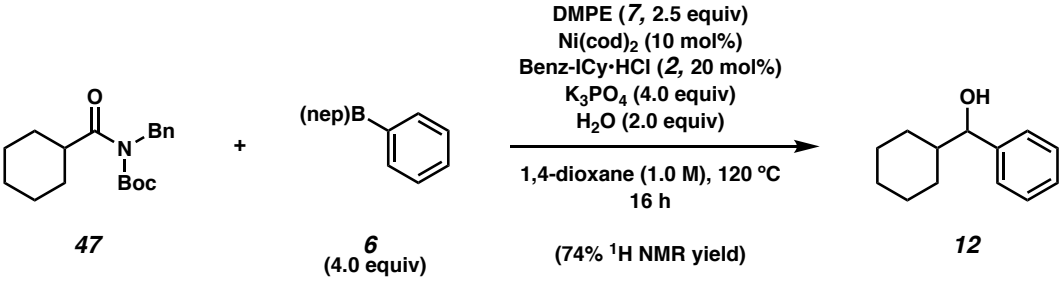
Alcohol 9. Crude alcohol **9** was synthesized following General Procedure A. Purification by flash column chromatography (39:1 Hexanes:Et_2O → 6.5:1 Hexanes:Et_2O) afforded alcohol **9** (61% yield, average of two experiments) as a clear oil. Alcohol **9**: R_f 0.48 (5:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.34–7.28 (m, 4H), 7.29–7.26 (m, 1H), 4.40 (s, 1H), 1.83 (s, 1H), 0.93 (s, 9H). Spectral data match those previously reported.[10]



Alcohol 10. Crude alcohol **10** was synthesized following General Procedure A. Sequential purification by flash column chromatography (98:1:1 Hexanes:CH_2Cl_2:Et_2O → 3:1:1 Hexanes:CH_2Cl_2:Et_2O) followed by preparative thin-layer chromatography (1:1:1 CH_2Cl_2:Et_2O:Hexanes) afforded alcohol **10** (66% yield, average of two experiments) as a clear oil. Alcohol **10**: R_f 0.38 (5:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.37–7.30 (m, 4H), 7.30–7.24 (m, 1H), 4.58 (d, J = 8.0, 1H), 2.69–2.56 (m, 1H), 2.15–1.96 (m, 2H), 1.92–1.75 (m, 5H). Spectral data match those previously reported.[10]

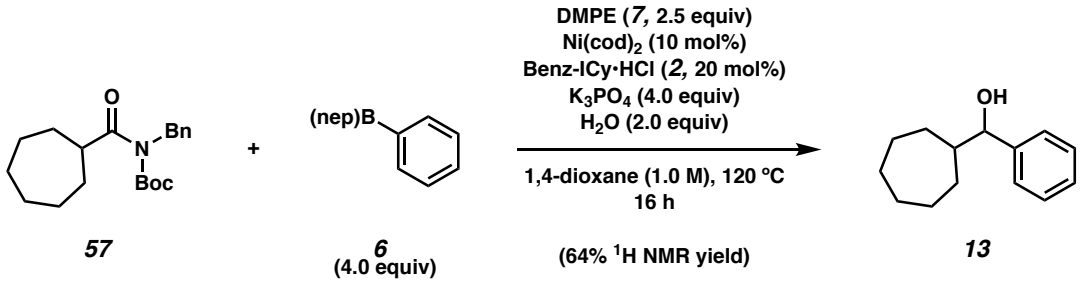


Alcohol 11. Crude alcohol **11** was synthesized following General Procedure A. ^{1}H NMR analysis of the crude reaction mixture indicated a 76% yield of alcohol **11** relative to 1,3,5-trimethoxybenzene external standard. Sequential purification by preparative thin-layer chromatography (5:1 Hexanes:EtOAc, then 5:1:1 Hexanes:CH_2Cl_2:Et_2O) afforded an analytical sample of alcohol **11** as a clear oil. Alcohol **11**: R_f 0.46 (5:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36–7.26 (m, 5H), 7.30–7.27 (m, 1H), 4.41 (d, J = 8.5, 1H), 2.22 (app sext, J = 8.2, 1H), 1.95–1.78 (m, 2H), 1.70–1.63 (m, 1H), 1.54–1.44 (m, 3H), 1.42–1.33 (m, 1H), 1.19–1.10 (m, 1H). Spectral data match those previously reported.[11]



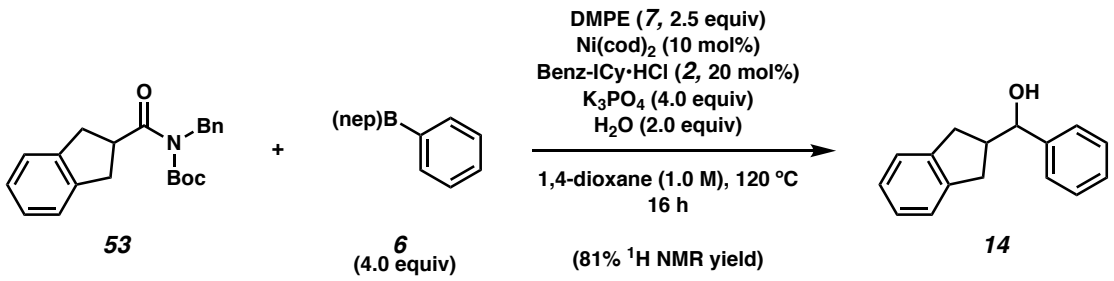
Alcohol 12. Crude alcohol **12** was synthesized following General Procedure A. ^{1}H NMR analysis of the crude reaction mixture indicated a 74% yield of alcohol **12** relative to 1,3,5-trimethoxybenzene external standard. Alcohol **12**: R_f 0.44 (5:1 Hexanes:EtOAc). Spectral data match those previously reported.[12]

Note: The ^{1}H NMR spectrum of the crude material obtained using the reaction conditions above is provided and matches previously reported ^{1}H NMR data.



Alcohol 13. Crude alcohol **13** was synthesized following General Procedure A. ^{1}H NMR analysis of the crude reaction mixture indicated an 64% yield of alcohol **13** relative to 1,3,5-trimethoxybenzene external standard). Preparation of an authentic sample of alcohol **13** from cycloheptyl(phenyl)methanone (see Section G for experimental details) allowed for direct comparison with the ^{1}H NMR spectrum of the crude reaction mixture and full characterization. Alcohol **13**: R_f 0.57 (5:1 Hexanes:EtOAc); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.35–7.23 (m, 5H), 4.47 (d, J = 6.7, 1H), 1.93–1.82 (m, 2H), 1.79 (br s, 1H), 1.72–1.65 (m, 1H), 1.65–1.30 (m, 9H), 1.23–1.09 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 144.0, 128.3, 127.5, 126.8, 79.4, 46.4, 31.2, 29.4, 28.6, 28.5, 26.9, 26.7; IR (film): 3378, 2917, 2852, 1492, 699 cm^{-1}; HRMS-APCI (*m/z*) [M + H]$^+$ calcd for $C_{14}H_{21}O^+$, 205.15869; found 205.15788.

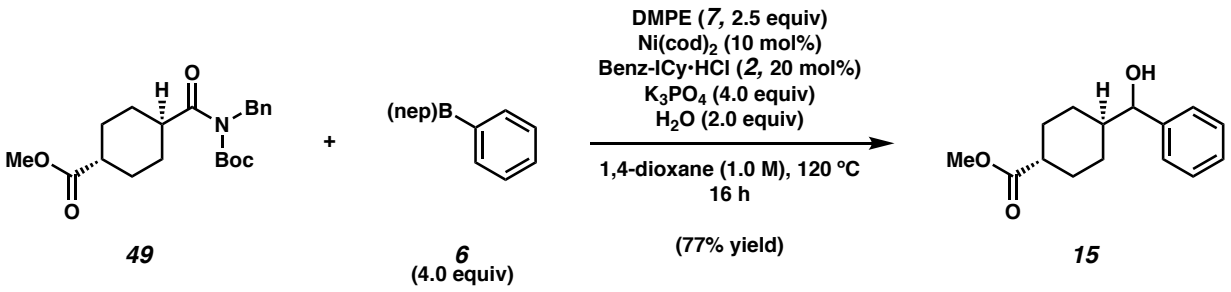
Note: ^{1}H NMR and ^{13}C NMR spectra of the authentic material, as prepared in section G, are provided. A ^{1}H NMR spectrum of the crude material obtained using the reaction conditions above is also provided and matches the ^{1}H NMR spectrum of the authentic material.



Alcohol 14. Crude alcohol **14** was synthesized following General Procedure A. ^{1}H NMR analysis of the crude reaction mixture indicated an 81% yield of alcohol **14** relative to 1,3,5-trimethoxybenzene external standard (average of two experiments). To the crude reaction mixture was added a teflon-coated magnetic stir bar and CH_2Cl_2 (1 mL). The solution was

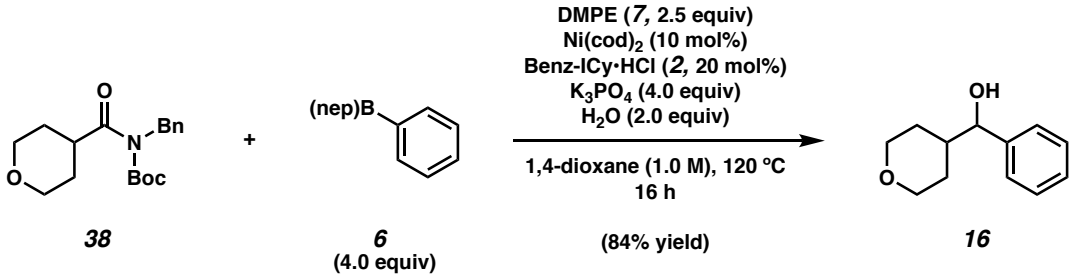
stirred, cooled to 0 °C, TFA (200 μL) was slowly added, and the contents were stirred at 0 °C for 1 h. The volatiles were then removed under reduced pressure to give the crude material, which was purified by flash column chromatography (99:1 Hexanes:EtOAc → 19:1 Hexanes:EtOAc). Treatment of the purified product with a solution (4 mL total volume, 1:1 v/v) of MeOH:2M KOH and stirring the resulting solution at 23 °C for 2 h afforded an analytical sample of alcohol **14** as a white solid. Alcohol **14**: R_f 0.38 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.43–7.29 (m, 5H), 7.23–7.22 (m, 1H), 7.14–7.11 (m, 3H), 4.63 (d, *J* = 8.5, 1H), 3.16 (dd, *J* = 16.0, 8.0, 1H), 3.06 (dd, *J* = 16.0, 8.0, 1H), 2.87 (app sext, *J* = 8.3, 1H), 2.69 (dd, *J* = 16.0, 8.0, 1H), 2.64 (dd, *J* = 16.0, 8.5, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 144.0, 143.2, 142.8, 128.7, 128.0, 126.7, 126.4, 126.3, 124.7, 124.5, 78.5, 47.3, 36.3, 36.1; IR (film): 3556, 3385, 3067, 3028, 2936 cm^{-1}; HRMS-APCI (*m/z*) $[M + NH_4]^+$ calcd for $C_{16}H_{20}NO^+$, 242.15394; found 242.15312.

*Note: **14** was obtained as a mixture of rotamers. These data represent empirically observed chemical shifts from the ^{1}H NMR and ^{13}C NMR spectra.*

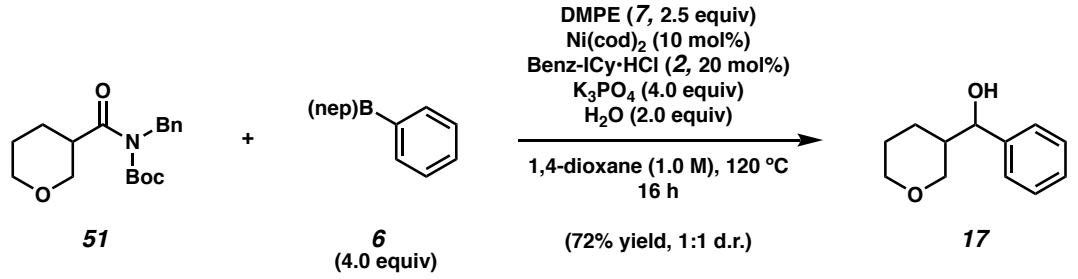


Alcohol 15. Crude alcohol **15** was synthesized following General Procedure A. Purification by flash column chromatography (99:1 Hexanes:Acetone → 9:1 Hexanes:Acetone) afforded a mixture of the trans and cis diastereomers of alcohol **15** (77% yield, 31:1 trans:cis diastereomers, average of two experiments) as a clear oil. Trans diastereomer alcohol **15**: R_f 0.22 (5:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.37–7.24 (m, 5H), 4.35 (d, *J* = 7.2, 1H), 3.63 (s, 3H), 2.20 (tt, *J* = 12.4, 3.6, 1H), 2.13–1.87 (m, 4H), 1.66–1.56 (m, 1H), 1.51–1.28 (m, 3H), 1.13–0.96 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 176.6, 143.5, 128.4, 127.7, 126.7, 79.1, 51.6,

44.2, 43.3, 28.70, 28.68, 28.4, 27.9; IR (film): 3454, 2938, 1731, 1716, 1170 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{15}H_{21}O_3^+$, 249.14852; found 249.14806.



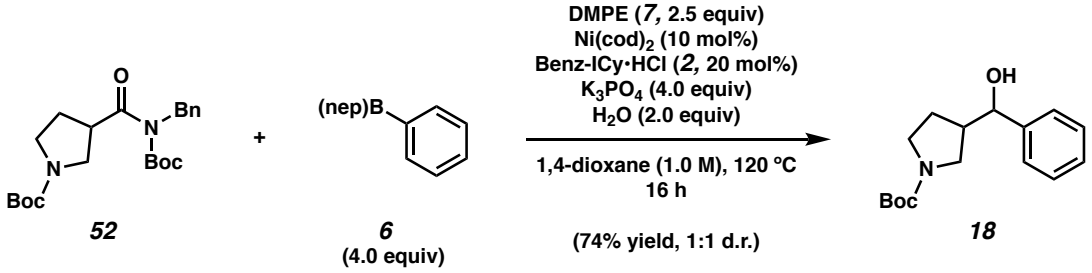
Alcohol 16. Crude alcohol **16** was synthesized following General Procedure B. Purification by flash column chromatography (19:1 Hexanes:EtOAc → 3:1 Hexanes:EtOAc) afforded alcohol **16** (84% yield, average of two experiments) as a white solid. Alcohol **16**: R_f 0.23 (3:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.38–7.26 (m, 5H), 4.32 (d, *J* = 7.7, 1H), 3.98 (app dd, *J* = 11.6, 4.6, 1H), 3.86 (app dd, *J* = 11.6, 4.6, 1H), 3.34 (td, *J* = 11.8, 2.2, 1H), 3.25 (td, *J* = 11.9, 2.3, 1H), 2.21 (br s, 1H), 1.94–1.86 (m, 1H), 1.86–1.72 (m, 1H), 1.50–1.36 (m, 1H), 1.36–1.20 (m, 1H), 1.20–1.07 (m, 1H). Spectral data match those previously reported.[13]



Alcohol 17. Crude alcohol **17** was synthesized following General Procedure B. Purification by flash chromatography (19:1 Hexanes:EtOAc → 1:1 Hexanes:EtOAc) afforded a mixture of diastereomers of alcohol **17** (72% yield, 1:1 d.r., average of two experiments) as a clear oil. Alcohol **17**: R_f 0.27 (3:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.37–7.26 (m, 10H), 4.50 (dd, *J* = 2.8, 7.0, 1H), 4.40 (dd, *J* = 2.9, 8.5, 1H), 4.16 (ddd, *J* = 1.8, 4.0, 11.3, 1H), 3.89–3.71 (m, 2H), 3.58 (ddd, *J* = 1.7, 4.0, 11.3, 1H), 3.49–3.30 (m, 3H), 3.20 (dd, *J* = 11.3, 9.4, 1H),

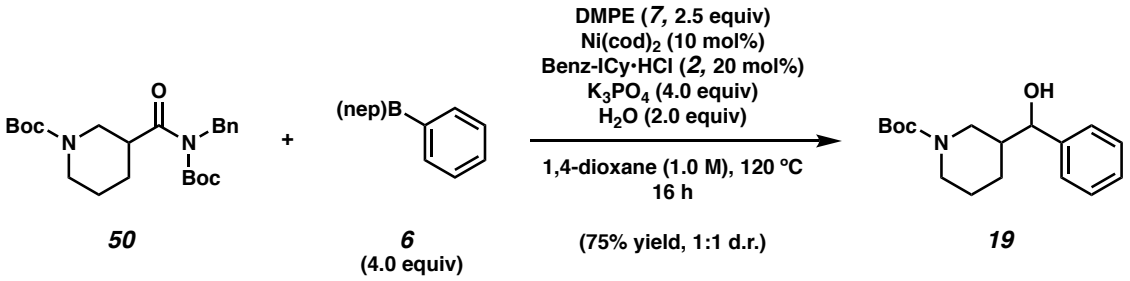
2.13, (d, J = 2.9, 1H), 2.10 (d, J = 3.0, 1H), 2.00–1.86 (m, 3H), 1.73–1.65 (m, 1H), 1.63–1.43 (m, 4H), 1.43–1.35 (m, 1H), 1.20–1.10 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$, 19 of 20 observed): δ 143.1, 143.0, 128.60, 128.56, 128.0, 127.9, 126.7, 126.4, 76.6, 75.9, 70.8, 70.6, 68.6, 68.4, 43.0, 42.9, 26.4, 25.4, 25.3; IR (film): 3401, 2938, 2846, 1453, 1081 cm^{-1}; HRMS-APCI (*m/z*) [M + H]$^+$ calcd for $C_{12}H_{17}O_2^+$, 193.12231; found 193.12228.

*Note: **17** was obtained as a mixture of diastereomers. These data represent empirically observed chemical shifts from the ^{1}H NMR and ^{13}C NMR spectra.*



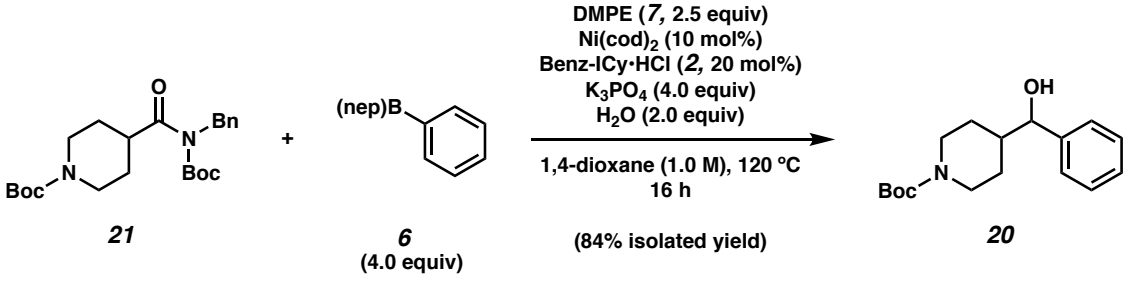
Alcohol 18. Crude alcohol **18** was synthesized following General Procedure B. Purification by flash chromatography (19:1 Hexanes:EtOAc → 1:1 Hexanes:EtOAc) afforded a mixture of diastereomers of alcohol **18** (74% yield, 1:1 d.r., average of two experiments) as a clear oil. Alcohol **18**: R_f 0.27 (9:1 PhH:Acetone). ^{1}H NMR (500 MHz, DMSO-d_6): 7.44–7.08 (m, 5H), 5.45–5.33 (m, 1H), 4.52–4.21 (m, 1H), 3.45–2.83 (m, 4H), 2.47–2.27 (m, 1H), 1.97–1.71 (m, 1H), 1.63–1.30 (m, 10H). Spectral data match those previously reported.[14]

*Note: **18** was obtained as a mixture of rotamers and diastereomers. These data represent empirically observed chemical shifts from the ^{1}H NMR and ^{13}C NMR spectra.*



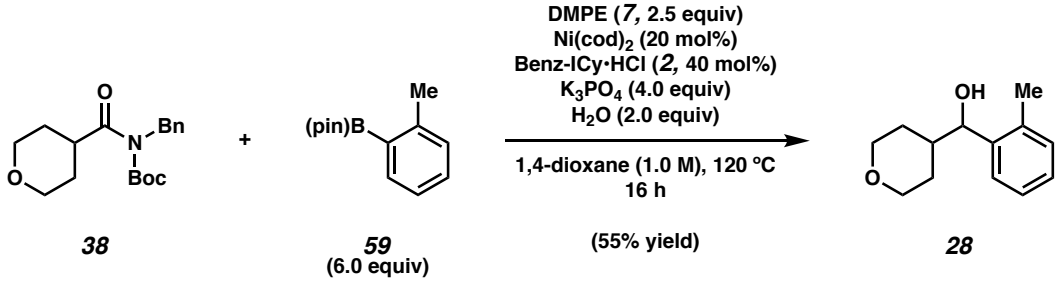
Alcohol 19. Crude alcohol **19** was synthesized following General Procedure B. Purification by flash column chromatography (PhH → 9:1 PhH:Acetone) afforded a mixture of diastereomers of alcohol **19** (75% yield, 1:1 d.r. average of two experiments) as a clear oil. Alcohol **19**: R_f 0.44 (9:1 PhH:Acetone); ^{1}H NMR (500 MHz, $CDCl_3$, 49 of 50 observed): δ 7.81–7.26 (m, 10H), 4.50 (br s, 1H), 4.43 (d, J = 8.5, 1H), 4.16–2.46 (m, 7H), 2.24–1.51 (m, 10H), 1.49–1.31 (m, 18H), 1.22–1.09 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$, 23 of 26 observed): δ 155.4, 155.0, 142.9, 128.57, 128.56, 127.93, 127.91, 126.7, 126.5, 79.7, 79.4, 76.5, 75.9, 46.7, 44.5, 43.1, 43.0, 28.6, 28.5, 27.0, 26.3, 24.8, 24.0; IR (film): 3422, 2975, 2930, 1665, 1424 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{17}H_{26}NO_3^+$, 292.19072; found 292.18998.

*Note: **19** was obtained as a mixture of rotamers and diastereomers. These data represent empirically observed chemical shifts from the ^{1}H NMR and ^{13}C NMR spectra.*

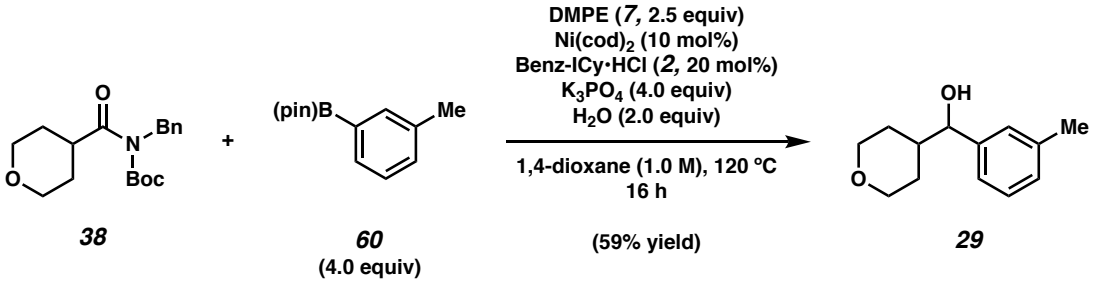


Alcohol 20. Crude alcohol **20** was synthesized following General Procedure B. Purification by flash column chromatography (PhH → 9:1 PhH:Acetone) afforded alcohol **20** (84% yield, average of two experiments) as a clear oil. Alcohol **20**: R_f 0.33 (9:1 PhH:Acetone). ^{1}H NMR (500 MHz, $CDCl_3$): 7.38–7.23 (m, 5H), 4.34 (d, J = 7.5, 1H), 4.29–3.81 (m, 2H), 2.77–2.38 (m, 2H), 2.26 (br s, 1H), 1.98–1.89 (m, 1H), 1.81–1.65 (m, 1H), 1.42 (s, 9H), 1.33–1.17 (m, 2H), 1.11 (app qd, J = 12.5, 4.4, 1H). Spectral data match those previously reported.[13]

E. Scope of Boronate Ester Nucleophiles

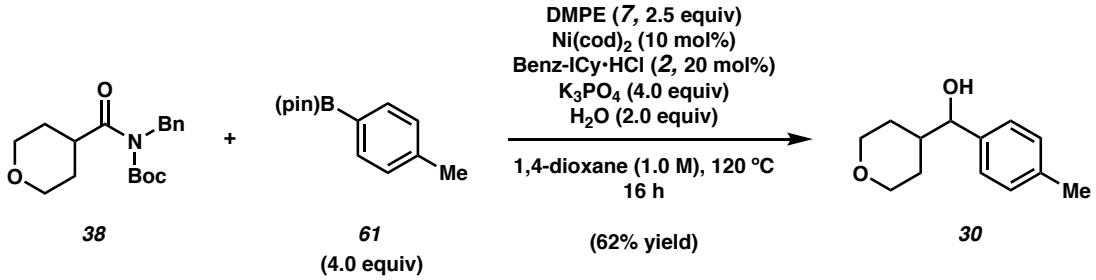


Alcohol 28. Crude alcohol **28** was synthesized following General Procedure B. Purification by flash column chromatography (19:1 Hexanes:EtOAc → 3:1 Hexanes:EtOAc) afforded alcohol **28** (55% yield, average of two experiments) as a crystalline solid. Alcohol **28**: mp: 62–64 ºC; R_f 0.32 (2:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.42 (dd, *J* = 7.7, 1.3, 1H), 7.23 (td, *J* = 7.5, 1.5, 1H), 7.21–7.12 (m, 2H), 4.69 (d, *J* = 7.3, 1H), 4.03 (dd, *J* = 11.2, 4.5, 1H), 3.90 (dd, *J* = 11.5, 4.5, 1H), 3.37 (td, *J* = 9.5, 2.3, 1H), 3.29 (td, *J* = 9.5, 2.3, 1H), 2.35 (s, 3H), 1.96–1.83 (m, 2H), 1.71 (br s, 1H), 1.60–1.48 (m, 1H), 1.42 (qd, *J* = 12.5, 4.6, 1H), 1.22–11.5 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 141.3, 135.3, 130.6, 127.5, 126.39, 126.36, 74.6, 68.1, 67.9, 42.2, 29.4, 29.2, 19.6; IR (film): 3420, 2951, 2847, 1090, 1016 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{13}H_{19}O_2^+$, 207.13796; found 207.13823.

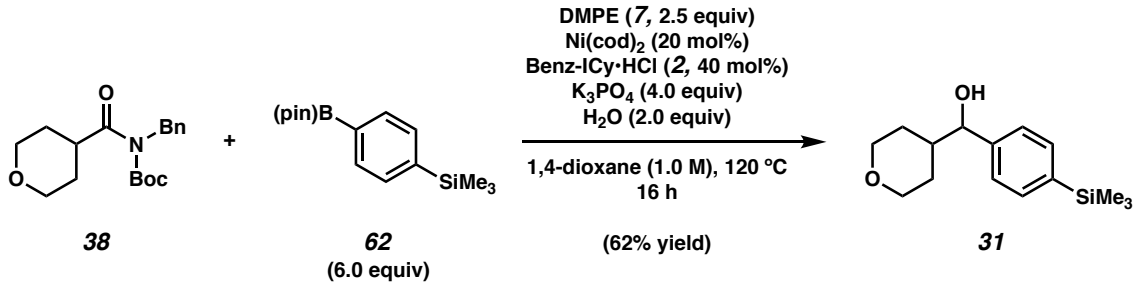


Alcohol 29. Crude alcohol **29** was synthesized following General Procedure B. Purification by flash chromatography (19:1 Hexanes:EtOAc → 3:1 Hexanes:EtOAc) afforded alcohol **29** (59% yield, average of two experiments) as a white solid. Alcohol **29**: mp: 97–99 ºC; R_f 0.36 (2:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.23 (t, *J* = 7.5, 1H), 7.12–7.09 (m, 3H), 4.33 (d, *J* = 7.9, 1H), 4.02 (dd, *J* = 11.4, 4.4, 1H), 3.90 (dd, *J* = 11.4, 4.4, 1H), 3.37 (td, *J* = 11.9, 2.3,

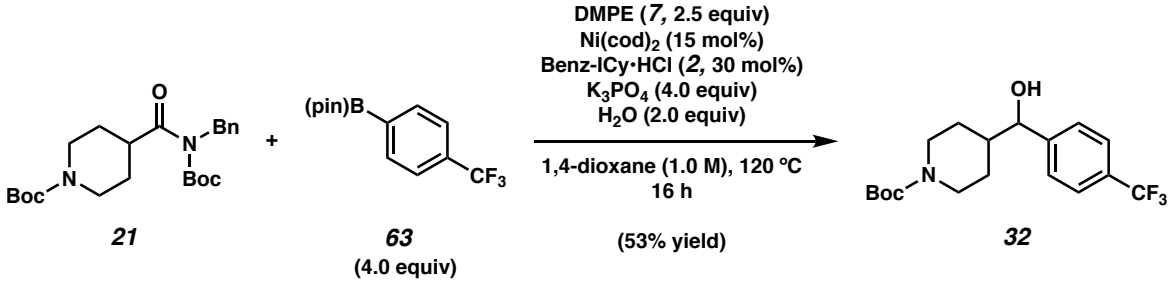
1H), 3.29 (td, *J* = 11.9, 2.3, 1H), 2.36 (s, 3H), 1.93–1.90 (m, 1H), 1.89–1.79 (m, 2H), 1.47 (qd, *J* = 12.2, 4.5, 1H), 1.32 (qd, *J* = 12.2, 4.6, 1H), 1.18–1.15 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 143.0, 138.2, 128.7, 128.4, 127.4, 123.8, 79.1, 68.0, 67.8, 42.5, 29.5, 29.4, 21.6; IR (film): 3409, 2950, 2848, 1135, 1089, 1035 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{13}H_{19}O_2^+$, 207.13796; found 207.13826.



Alcohol 30. Crude alcohol **30** was synthesized following General Procedure B. Purification by flash chromatography (19:1 Hexanes:EtOAc → 3:1 Hexanes:EtOAc) afforded alcohol **30** (62% yield, average of two experiments) as a white solid. Alcohol **30**: mp: 71–74 ºC; R_f 0.25 (2:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.24–7.13 (m, 4H), 4.33 (d, *J* = 7.9, 1H), 4.02 (dd, *J* = 11.8, 4.4, 1H), 3.89 (dd, *J* = 11.2, 4.8, 1H), 3.37 (td, *J* = 11.9, 2.3, 1H), 3.28 (td, *J* = 11.9, 2.3, 1H), 2.34 (s, 3H), 1.94–1.91 (m, 1H), 1.86–1.79 (m, 1H), 1.46 (qd, *J* = 12.3, 4.7, 1H), 1.30 (qd, *J* = 12.3, 4.7, 1H), 1.17–1.13 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 143.0, 137.6, 129.2, 126.7, 78.8, 68.0, 67.8, 42.5, 29.6, 29.3, 21.3; IR (film): 3410, 2950, 2847, 1089, 1033, 1017 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{13}H_{19}O_2^+$, 207.13796; found 207.13823.



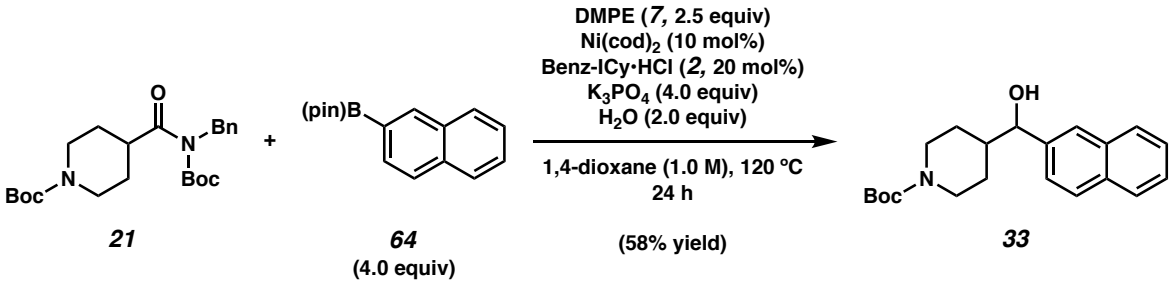
Alcohol 31. Crude alcohol **31** was synthesized following General Procedure B. Purification by flash chromatography (PhH → 9:1 PhH:Acetone) afforded alcohol **31** (62% yield, average of two experiments) as a clear oil. Alcohol **31**: R_f 0.26 (9:1 PhH:Acetone); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.51 (d, *J* = 8.1, 2H), 7.30 (d, *J* = 8.1, 2H), 4.37 (d, *J* = 7.5, 1H), 4.02 (dd, *J* = 11.4, 4.6, 1H), 3.90 (dd, *J* = 11.4, 4.6, 1H), 3.37 (td, *J* = 12.0, 2.3, 1H), 2.29 (td, *J* = 12.0, 2.3, 1H), 1.95–1.89 (m, 1H), 1.89–1.74 (m, 2H), 1.47 (app qd, *J* = 12.6, 4.7, 1H), 1.39–1.29 (m, 1H), 1.22–1.16 (m, 1H), 0.26 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 143.5, 140.2, 133.6, 126.1, 79.0, 68.0, 67.8, 42.4, 29.4, 29.3, –0.98; IR (film): 3409, 2953, 2846, 1247, 831 cm^{-1}; HRMS-APCI (*m/z*) $[M + K]^+$ calcd for $C_{15}H_{24}O_2SiK^+$, 303.11771; found 303.11798.



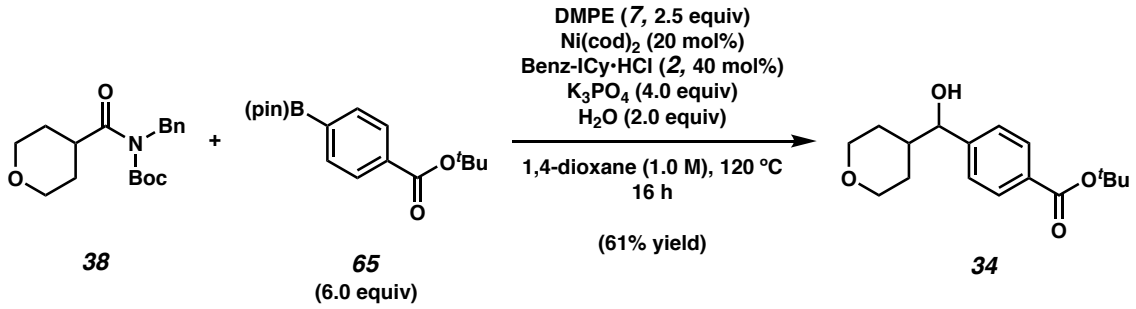
Alcohol 32. Crude alcohol **32** was synthesized following General Procedure B. Purification by flash chromatography (PhH → 9:1 PhH:Acetone) afforded alcohol **32** (53% yield, average of two experiments) as a clear oil. Alcohol **32**: mp: 140–143 ºC; R_f 0.29 (9:1 PhH:Acetone). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.61 (d, *J* = 8.3, 2H), 7.43 (d, *J* = 8.3, 2H), 4.48 (d, *J* = 7.1, 1H), 4.29–3.93 (m, 2H), 2.77–2.45 (m, 2H), 2.01 (br s, 1H), 1.93–1.84 (m, 1H), 1.80–1.69 (m, 1H), 1.44 (s, 9H), 1.34–1.12 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.9, 147.1, 130 (q, *J* = 32), 127.0, 125.4 (q, *J* = 3.6), 125.4, 125.3, 123.1, 79.6, 77.9, 43.7, 28.6, 28.4, 27.9; IR (film): 3418,

2932, 2859, 1666, 1325, 1162, 1125 cm^{-1}; HRMS-APCI (*m/z*) $[M]^+$ calcd for $C_{18}H_{24}F_3NO_3^+$, 359.17028; found 359.17126.

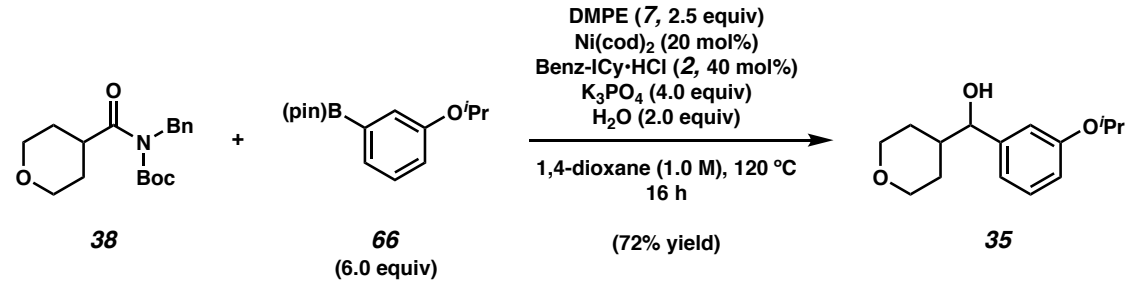
*Note: **32** was obtained as a mixture of rotamers. These data represent empirically observed chemical shifts from the ^1H NMR and ^{13}C NMR spectra.*



Alcohol 33. Crude alcohol **33** was synthesized following General Procedure B. Purification by flash chromatography (PhH → 9:1 PhH:Acetone) afforded alcohol **33** (58% yield, average of two experiments) as a clear oil. Alcohol **33**: R_f 0.39 (9:1 PhH:Acetone); ^1H NMR (500 MHz, $CDCl_3$): δ 7.89–7.80 (m, 3H), 7.73, (s, 1H), 7.53–7.43 (m, 3H), 4.56 (d, *J* = 7.5, 1H), 4.17 (app d, *J* = 13.4, 1H), 4.04 (app d, *J* = 13.4, 1H), 2.68 (td, *J* = 12.9, 2.7, 1H), 2.58 (td, *J* = 12.9, 2.7, 1H), 2.04–1.98 (m, 1H), 1.89–1.82 (m, 1H), 1.43 (s, 9H), 1.36–1.15 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$, 16 of 17 observed): δ 154.9, 140.5, 133.3, 133.2, 128.4, 128.0, 127.8, 126.4, 126.1, 125.7, 124.5, 79.4, 78.8, 43.5, 28.6, 28.4; IR (film): 3418, 2974, 2929, 2856, 1666, 1425, 1162 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{21}H_{28}NO_3^+$, 342.20637; found 342.20615.

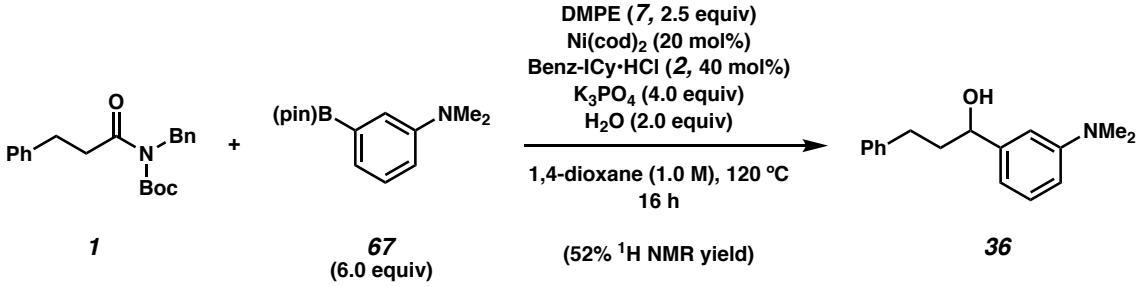


Alcohol 34. Crude alcohol **34** was synthesized following General Procedure B. Purification by flash chromatography (19:1 Hexanes:EtOAc → 3:1 Hexanes:EtOAc) generated alcohol **34** (61% yield, average of two experiments) as a clear oil. Alcohol **34**: R_f 0.18 (2:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00–7.95 (m, 2H), 7.39–7.34 (m, 2H), 4.45 (d, *J* = 7.2, 1H), 4.01 (app dd, *J* = 11.4, 4.2, 1H), 3.90 (app dd, *J* = 11.4, 4.2, 1H), 3.35 (td, *J* = 12.0, 2.0, 1H), 3.27 (td, *J* = 12.0, 2.0, 1H), 1.91 (br s, 1H), 1.88–1.78 (m, 2H), 1.59 (s, 9H), 1.52–1.42 (m, 1H), 1.40–1.31 (m, 1H), 1.18–1.14 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 165.7, 147.5, 131.6, 129.7, 128.5, 126.5, 81.2, 78.4, 68.0, 67.7, 42.6, 29.3, 29.1, 28.3; IR (film): 3417, 2953, 2848, 1710, 1292, 1117 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{17}H_{25}O_4^+$, 293.17474; found 293.17416.

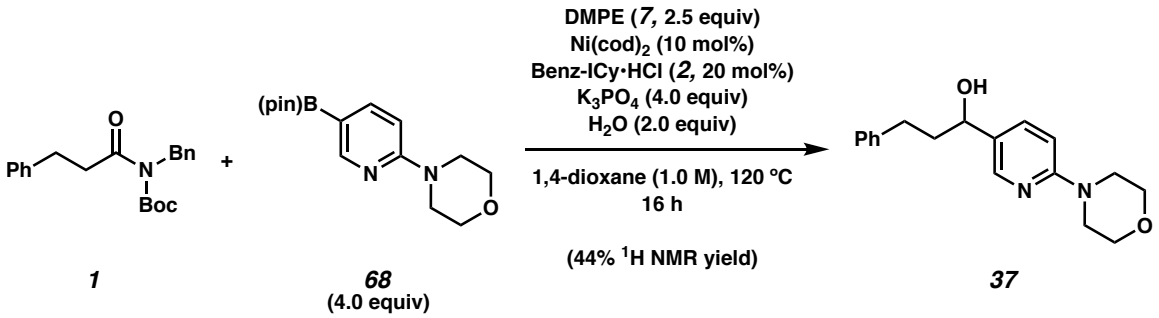


Alcohol 35. Crude alcohol **35** was synthesized following General Procedure B. Purification by flash chromatography (PhH → 9:1 PhH:Acetone) generated alcohol **35** (72% yield, average of two experiments) as a clear oil. Alcohol **35**: R_f 0.31 (9:1 PhH:Acetone); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.24 (t, *J* = 8.1, 1H), 6.87–6.84, (m, 2H), 6.81 (ddd, *J* = 8.1, 2.5, 0.85, 1H), 4.56 (sept, *J* = 6.0, 1H), 4.32 (d, *J* = 7.6, 1H), 4.02 (app dd, *J* = 11.4, 4.2, 1H), 3.90 (app dd, *J* = 11.4, 4.2, 1H), 3.36 (td, *J* = 12.0, 2.2, 1H), 3.28 (td, *J* = 11.8, 2.2, 1H), 1.94–1.87 (m, 1H), 1.87–1.78 (m,

1H), 1.58 (br s, 1H) 1.46 (qd, *J* = 12.3, 4.6, 1H), 1.34 (d, *J* = 6.1), 1.33 (qd, *J* = 12.3, 4.4) (7H total), 1.23–1.15 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.1, 144.7, 129.5, 118.9, 115.0, 114.3, 78.9, 69.9, 68.0, 67.8, 42.5, 29.4, 29.3, 22.2; IR (film): 3406, 2974, 2847, 1599, 1583, 1253, 1116 cm^{-1}; HRMS-APCI (*m/z*) $[M]^+$ calcd for $C_{15}H_{22}O_3^+$, 250.15635; found 250.15623.



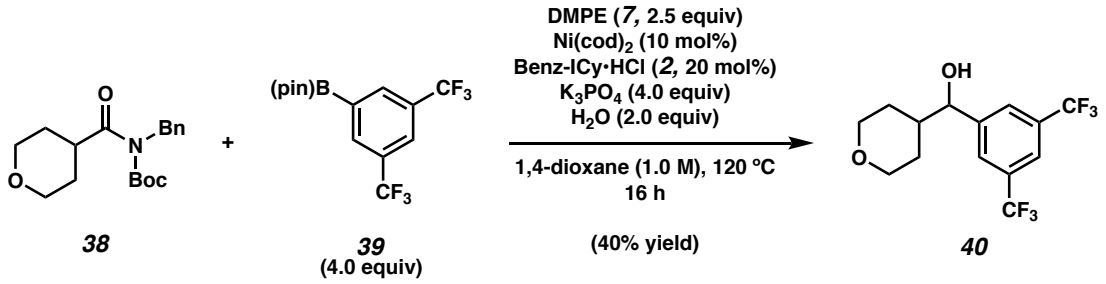
Alcohol 36. Crude alcohol **36** was synthesized following General Procedure C. ^{1}H NMR analysis of the crude reaction mixture indicated an 52% yield of alcohol **36** relative to 1,3,5-trimethoxybenzene external standard (average of two experiments). Purification by preparative thin-layer chromatography (9:1 PhH:Acetone) provided an analytical sample of alcohol **36** as a clear oil. Alcohol **36**: R_f 0.54 (2:1 Hex:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.30–7.26 (m, 2H), 7.25–7.14 (m, 3H), 6.75–6.72 (m, 1H), 6.70 (app d, *J* = 7.5, 1H), 6.66 (ddd, *J* = 8.5, 2.6, 0.8, 1H), 4.62 (ddd, *J* = 8.3, 5.4, 3.3, 1H), 2.96 (s, 6H), 2.77 (ddd, *J* = 14.5, 9.8, 5.8, 1H), 2.69 (ddd, *J* = 14.5, 9.8, 6.4, 1H), 2.20–2.10 (m, 1H), 2.10–1.99 (m, 1H), 1.81 (d, *J* = 3.3, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.0, 145.7, 142.1, 129.4, 128.6, 128.5, 125.9, 114.3, 112.1, 110.1, 74.6, 40.8, 40.4, 32.3; IR (film): 3366, 3025, 2918, 2858, 2801, 1602, 1495, 697 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{17}H_{22}NO^+$, 256.16959; found 256.16915.



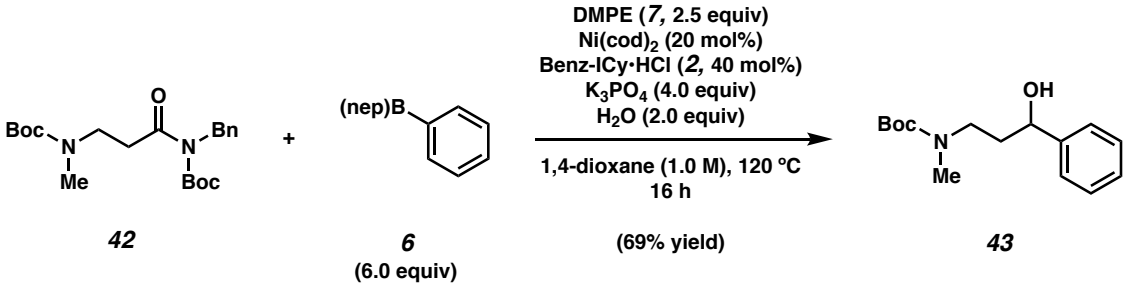
Alcohol 37. Crude alcohol **37** was synthesized following General Procedure C. ^{1}H NMR analysis of the crude reaction mixture indicated a 44% yield of alcohol **37** and 56% yield of the corresponding ketone intermediate relative to 1,3,5-trimethoxybenzene external standard (average of two experiments). Preparation of an authentic sample of alcohol **37** (see Section G for experimental details) allowed for direct comparison with the ^{1}H NMR spectrum of the crude reaction mixture and full characterization. Alcohol **37**: R_f 0.45 (3:1 PhH:Acetone); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.15 (d, J = 2.4, 1H), 7.55 (dd, J = 8.8, 2.4, 1H), 7.31–7.26 (m, 2H), 7.22–7.15 (m, 3H), 6.65 (d, J = 8.8, 1H), 4.65–4.59 (m, 1H), 3.83 (app t, J = 4.8, 4H), 3.50 (app t, J = 4.8, 4H), 2.76–2.58 (m, 2H), 2.20–2.10 (m, 1H), 2.06–1.95 (m, 1H), 1.78–1.68 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.6, 146.3, 141.7, 135.8, 129.5, 128.56, 128.55, 126.1, 107.1, 71.6, 66.9, 45.8, 40.0, 32.2; IR (film): 3391, 3025, 2918, 2855, 1605, 1494, 1245 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{18}H_{23}N_2O_2^+$, 299.17540; found 299.17471.

Note: ^{1}H NMR and ^{13}C NMR spectra of the authentic material, as prepared in section G, are provided. A ^{1}H NMR spectrum of the crude material obtained using the reaction conditions above is also provided and matches the ^{1}H NMR spectrum of the authentic material.

F. Syntheses of Alcohols 40 and 43



Alcohol 40. Crude alcohol **40** was synthesized following General Procedure B. Purification by flash column chromatography (19:1 Hexanes:EtOac → 3:1 Hexanes:EtOAc) afforded alcohol **40** (40% yield, average of two experiments) as a clear oil. Alcohol **40**: R_f 0.45 (2:1 Hexanes:EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.81 (s, 1H), 7.78 (s, 2H), 4.58 (dd, *J* = 6.9, 3.5, 1H), 4.03 (app dd, *J* = 11.6, 4.3, 1H), 3.95 (app dd, *J* = 11.6, 4.2, 1H), 3.36 (td, *J* = 12.1, 2.0), 3.32 (td, *J* = 12.1, 2.0) (2H total), 2.08 (d, *J* = 3.5, 1H), 1.90–1.80 (m, 1H), 1.79–1.72 (m, 1H), 1.49 (qd, 12.3, 4.6), 1.43 (qd, *J* = 12.3, 4.7) (2H total), 1.29–1.19 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 145.6, 131.8 (q, *J* = 33), 126.8, 126.7, 124.5, 122.3, 121.8 (sept, *J* = 3.7), 77.5, 67.8, 67.6, 42.6, 29.1, 28.4; IR (film): 3401, 2956, 2855, 1277, 1128 cm^{-1}; HRMS-APCI (*m/z*) $[M + H]^+$ calcd for $C_{14}H_{15}F_6O_2^+$, 329.09708; found 329.09637.

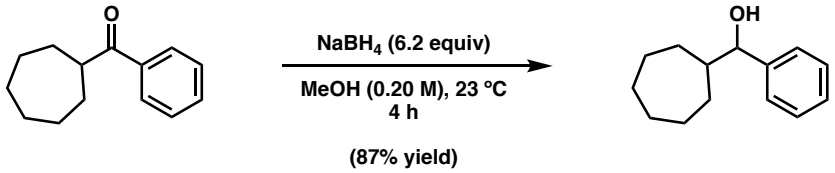


Alcohol 43. Crude alcohol **43** was synthesized following General Procedure B. Purification by flash column chromatography (PhH → 9:1 PhH:Acetone) yield alcohol **43** (69% yield, average of two experiments) as a clear oil. Alcohol **43**: R_f 0.48 (2:1 Hexanes:EtOAc); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42–7.18 (m, 5H), 4.60 (br s, 1H), 4.33 (br s), 3.90 (br s), 3.47 (br s), 3.30–

2.92 (m), 2.50 (br s), (total 3H), 2.87 (s, 3H), 2.00–1.88 (m, 1H), 1.88–1.67 (m, 1H), 1.47 (s, 9H). Spectral data match those previously reported.[15]

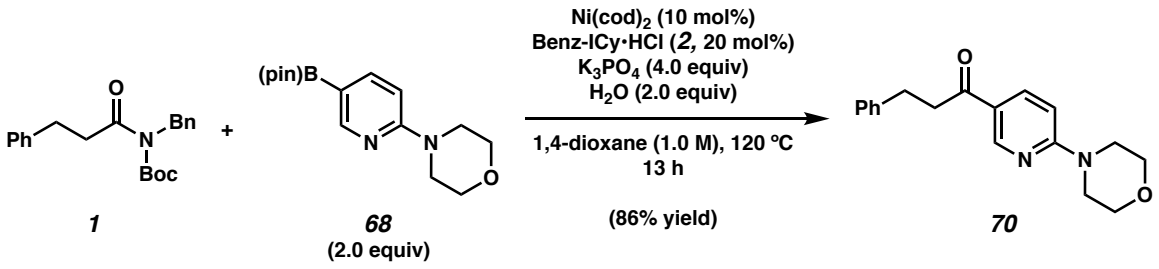
*Note: **43** was obtained as a mixture of rotamers. These data represent empirically observed chemical shifts from the ^{1}H NMR spectrum.*

G. Syntheses of Authentic Samples of Alcohols 13 and 37

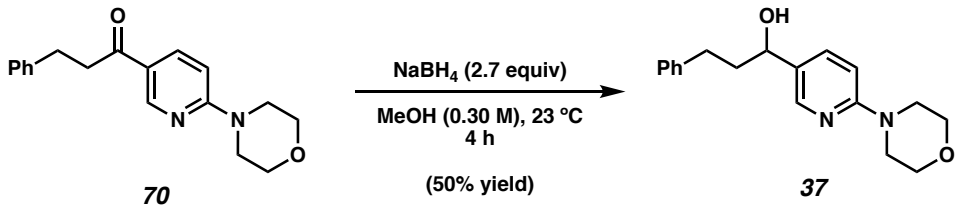


To a flame-dried 1-dram vial equipped with a magnetic stir bar was added the ketone **69** (11.4 mg, 0.0560 mmol, 1.00 equiv) in MeOH. The solvent was then evaporated under reduced pressure. The vial was then capped with a septum cap, and the atmosphere was purged with N_2. To the vial was added MeOH (0.300 mL, 0.190 M) and the reaction was stirred to give a homogeneous solution. $NaBH_4$ (6.80 mg, 0.180 mmol, 3.20 equiv) was added in a single portion and the vial was stirred at 23 °C. After 1 h, $NaBH_4$ (6.40 mg, 0.170 mmol, 3.00 equiv) was added in a single portion and the vial was stirred at 23 °C. After 3 h, the reaction was quenched with H_2O (2 mL). The aqueous layer was extracted with EtOAc (4 x 3 mL), the combined organic layers were dried over anhydrous $MgSO_4$, and the volatiles were removed under reduced pressure. The resulting crude residue was purified by preparative thin-layer chromatography (5:1 Hexanes:EtOAc) to yield alcohol **13** as a clear oil (10.0 mg, 87% yield).

Note: See section D for chemical shifts from the ^{1}H NMR and ^{13}C NMR spectra.



A 1-dram vial was charged with anhydrous powder K_3PO_4 (170 mg, 0.800 mmol, 4.00 equiv) and a magnetic stir bar. The vial and its contents were flame-dried under reduced pressure and allowed to cool under N_2. Amide substrate **1** (67.9 mg, 0.200 mmol, 1.00 equiv) and boronate ester nucleophile **68** (116 mg, 0.400 mmol, 2.00 equiv) were added. The vial was flushed with N_2 for 5 min, then water (7.21 μL, 0.400 mmol, 2.00 equiv), which had been sparged with N_2 for 10 min, was added. The vial was taken into a glovebox and charged with $Ni(cod)_2$ (5.50 mg, 10 mol%) and Benz-ICy•HCl (**2**, 12.8 mg, 20 mol%). Subsequently, 1,4-dioxane (200 μL, 1.00 M) was added. The vial was sealed with a Teflon-lined screw cap, removed from the glovebox, and stirred vigorously (800 RPM) at 120 ºC for 13 h. After cooling to 23 ºC, the mixture was quenched by the addition of saturated aqueous NH_4Cl (1 mL) and extracted with EtOAc (3 x 2 mL). The combined organic layers were then filtered over a plug of silica gel (3 cm) and Na_2SO_4 (3 cm) using EtOAc (10 mL) as eluent. The volatiles were removed under reduced pressure and the resulting crude residue was purified by flash column chromatography (9:1 Hexanes:EtOAc → EtOAc) to yield ketone **70** as a clear oil (10.0 mg, 87% yield). Alcohol **70**: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.78 (d, *J* = 2.3, 1H), 8.05 (dd, *J* = 9.1, 2.3, 1H), 7.32–7.28 (m, 2H), 7.26–7.18 (m, 3H), 6.60 (d, *J* = 9.1, 1H), 3.81 (t, *J* = 5.0, 4H), 3.67 (t, *J* = 5.0, 4H), 3.19 (t, *J* = 7.5, 4H), 3.05 (t, *J* = 7.5, 4H). Spectral data match those previously reported.[16]

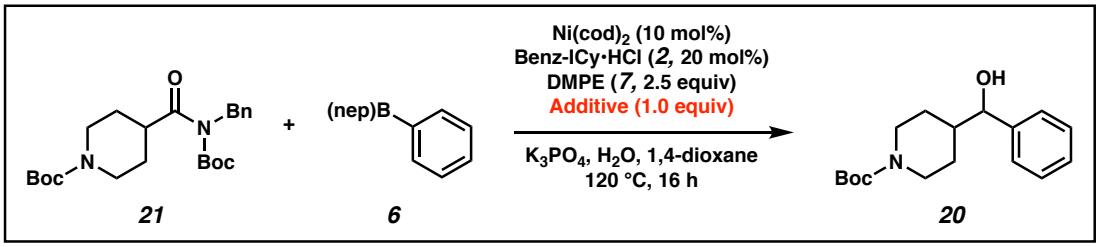


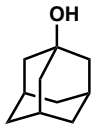
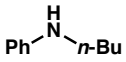
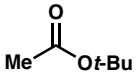
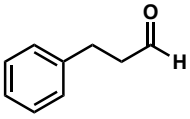
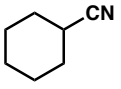
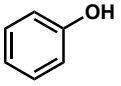
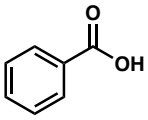
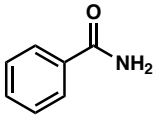
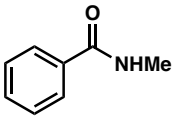
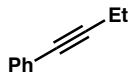
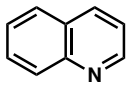
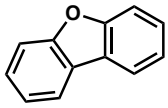
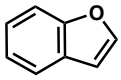
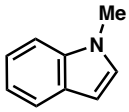
To a flame-dried 1-dram vial equipped with a magnetic stir bar was added $NaBH_4$ (17.0 mg, 0.450 mmol, 2.70 equiv). The ketone **70** (50.0 mg, 0.0170 mmol, 1.00 equiv) was dissolved in MeOH (0.60 mL, 0.30 M) and added to the vial. After 1 h, the reaction was quenched with H_2O (2 mL). The aqueous layer was extracted with EtOAc (4 x 3 mL), the combined organic layers were dried over anhydrous $NaSO_4$, and the volatiles were removed under reduced pressure. The resulting crude residue was purified by preparative TLC (1:1 Hexanes:EtOAc) to yield alcohol **37** as a clear oil (25.0 mg, 50% yield).

Note: See section E for chemical shifts from the ^{1}H NMR and ^{13}C NMR spectra.

H. Robustness Screen

Table S2: Evaluation of Functional Group Compatibility in the Suzuki–Miyaura Coupling and Transfer Hydrogenation Cascade

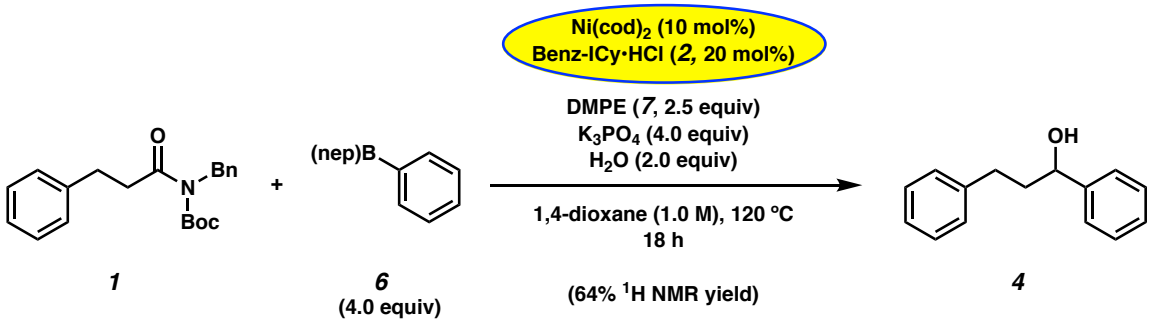


Entry	Additive	Yield of *20* (%)	Additive Remaining (%)	SM Remaining (%)
1	–	81	N.D.	0
2		89	>99	0
3		83	>99	0
4		42	N.D.[b]	0
5		43	0	0
6		0	0	0
7		0	0	0
8		31	0	0
9		13	0	0
10		58	88	0
11		68	0	0
12		22	18	0
13		88	98	0
14		68	94	0
15		25	0	0
16		78	80	0

Conditions: amide *21* (0.20 mmol, 1.00 equiv), PhB(nep) (*6*, 0.80 mmol, 4.00 equiv), DMPE (*7*, 0.50 mmol, 2.50 equiv), additive (0.20 mmol, 1.00 equiv), $Ni(cod)_2$ (0.020 mmol, 10 mol%), Benz-ICy·HCl (*2*, 0.040 mmol, 20 mol%), K_3PO_4 (0.80 mmol, 4.00 equiv), H_2O (0.40 mmol, 2.00 equiv), and 1,4-dioxane (1.0 M) in a sealed vial at 120 °C for 16 h. Yields of coupled product, remaining additive, and remaining starting material were determined by ^1H NMR analysis using 1,3,5-trimethoxybenzene or hexamethylbenzene as an external standard.

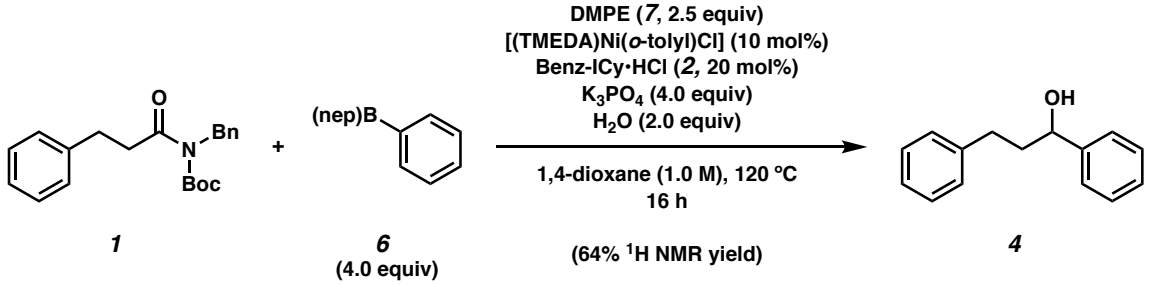
I. Benchtop Variants of Methodology

Procedure A: Employing a paraffin wax encapsulation approach. Note: The supporting information for the preparation of Ni(cod)$_2$/Benz-ICy–paraffin capsules has been previously reported.[17]



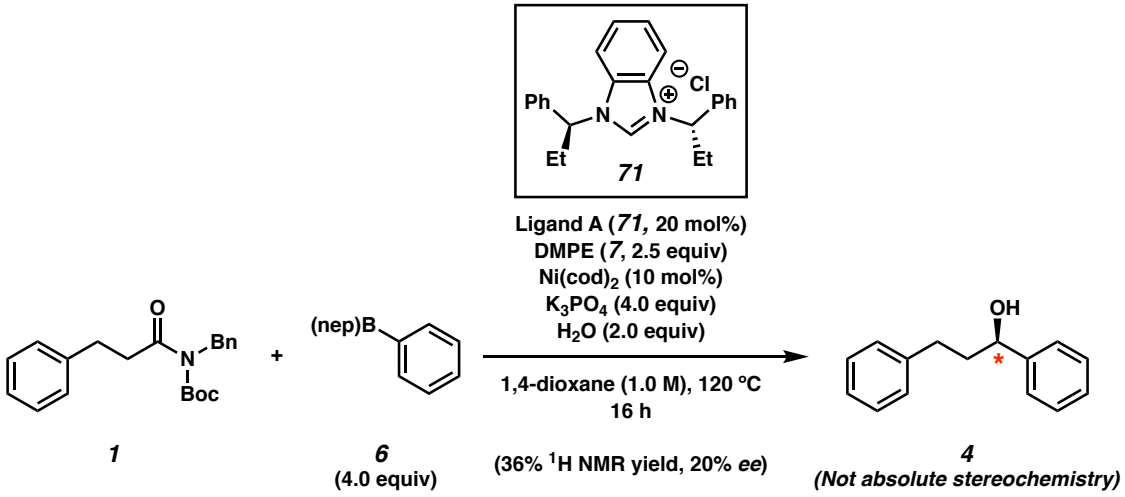
A 2-dram vial was charged with anhydrous powder K_3PO_4 (340 mg, 1.60 mmol, 4.00 equiv) and a magnetic stir bar (egg-shaped 3/8 x 3/16 in). The vial and its contents were flame-dried under reduced pressure and allowed to cool under N_2. The vial was then charged with amide substrate **1** (136 mg, 0.40 mmol, 1.00 equiv), boronate ester nucleophile **6** (304 mg, 1.60 mmol, 4.00 equiv), DMPE (**7**, 165 mg, 1.00 mmol, 2.50 equiv), and a paraffin wax capsule containing Ni(cod)$_2$ (11.0 mg, 0.0400 mmol, 10 mol%) and Benz-ICy•HCl (**2**, 25.5 mg, 0.0800 mmol, 20 mol%) were added. The vial was purged with N_2 and subsequently deionized water (14.0 µL, 0.80 mmol, 2.00 equiv) and 1,4-dioxane (0.400 mL, 1.00 M), which had been sparged with N_2 for 10 min, were added. The vial was capped with a Teflon-lined screw cap under a flow of N_2 and the reaction mixture was stirred vigorously (800 RPM) at 120 ºC for 18 h. After removing the vial from heat, the reaction mixture was transferred to a 100 mL pear-shaped flask containing 2.0 g of silica gel with hexanes (6 mL) and CH_2Cl_2 (6 mL). The mixture was adsorbed onto the silica gel under reduced pressure and filtered over a plug of silica gel (4.0 cm OD x 3.0 cm, 300 mL of hexanes eluent to remove paraffin, then 250 mL of EtOAc eluent). The volatiles were removed under reduced pressure. ^{1}H NMR analysis of the crude reaction mixture indicated a 64% yield of alcohol **4** relative to 1,3,5-trimethoxybenzene external standard (average of two experiments).

Procedure B: Employing an air-stable Ni(II) precatalyst



A 1-dram vial was charged with anhydrous powder K_3PO_4 (170 mg, 0.800 mmol, 4.00 equiv) and a magnetic stir bar. The vial and its contents were flame-dried under reduced pressure and allowed to cool under N_2. Amide substrate **1** (67.9 mg, 0.200 mmol, 1.00 equiv), boronate ester nucleophile **6** (152 mg, 0.800 mmol, 4.00 equiv), DMPE (**7**, 82.6 mg, 0.500 mmol, 2.50 equiv), [(TMEDA)Ni(*o*-tolyl)Cl][3] (6.03 mg, 0.0200 mmol, 10 mol%), and Benz-ICy•HCl (**2**, 12.8 mg, 0.0400 mmol, 20 mol%) were added. The vial was flushed with N_2 for 5 min, then water (7.21 μL, 0.400 mmol, 2.00 equiv) and 1,4-dioxane (200 μL, 1.00 M), which had been sparged with N_2 for 10 min, were added. The vial was capped with a Teflon-lined screw cap under a flow of N_2 and the reaction mixture was stirred vigorously (800 RPM) at 120 ºC for 16 h. After cooling to 23 ºC, the mixture was quenched by the addition of saturated aqueous NH_4Cl (1 mL) and extracted with EtOAc (3 x 2 mL). The combined organic layers were then filtered over a plug of silica gel (3 cm) and Na_2SO_4 (3 cm) using EtOAc (10 mL) as eluent. The volatiles were removed under reduced pressure. ^{1}H NMR analysis of the crude reaction mixture indicated a 64% yield of alcohol **4** relative to 1,3,5-trimethoxybenzene external standard.

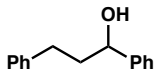
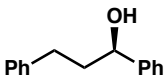
J. Enantioselectivity Experiments

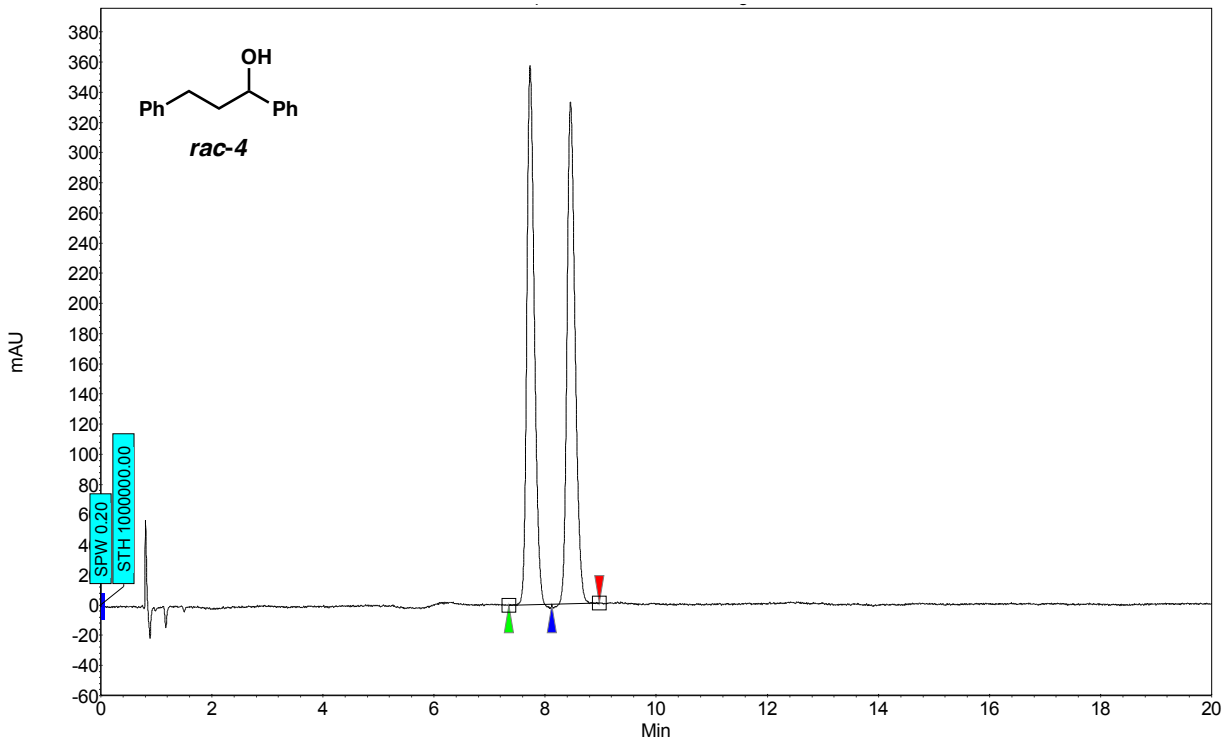


A 1-dram vial was charged with anhydrous powder K_3PO_4 (170 mg, 0.800 mmol, 4.00 equiv) and a magnetic stir bar. The vial and its contents were flame-dried under reduced pressure and allowed to cool under N_2. Amide substrate **1** (67.9 mg, 0.200 mmol, 1.00 equiv), boronate ester nucleophile **6** (152 mg, 0.800 mmol, 4.00 equiv), and DMPE (**7**, 82.6 mg, 0.500 mmol, 2.50 equiv) were added. The vial was flushed with N_2 for 5 min, then water (7.21 μL, 0.400 mmol, 2.00 equiv), which had been sparged with N_2 for 10 min, was added. The vial was taken into a glovebox and charged with $Ni(cod)_2$ (5.50 mg, 0.0200 mmol, 10 mol%) and Ligand A (**71**, 15.6 mg, 0.0400 mmol, 20 mol%). Subsequently, 1,4-dioxane (200 μL, 1.00 M) was added. The vial was sealed with a Teflon-lined screw cap, removed from the glovebox, and stirred vigorously (800 RPM) at 120 ºC for 16 h. After cooling to 23 ºC, the mixture was quenched by the addition of saturated aqueous NH_4Cl (1 mL) and extracted with EtOAc (3 x 2 mL). The combined organic layers were then filtered over a plug of silica gel (3 cm) and Na_2SO_4 (3 cm) using EtOAc (10 mL) as eluent. ^1H NMR analysis of the crude reaction mixture indicated a 36% yield of alcohol **4** relative to 1,3,5-trimethoxybenzene external standard. Purification by preparative thin-layer chromatography (4:1 Hexanes:EtOAc) provided an analytical sample of enantioenriched alcohol **4** as a clear oil. The spectral data match those previously reported in section D of Experimental Procedures for ***rac*-4**.

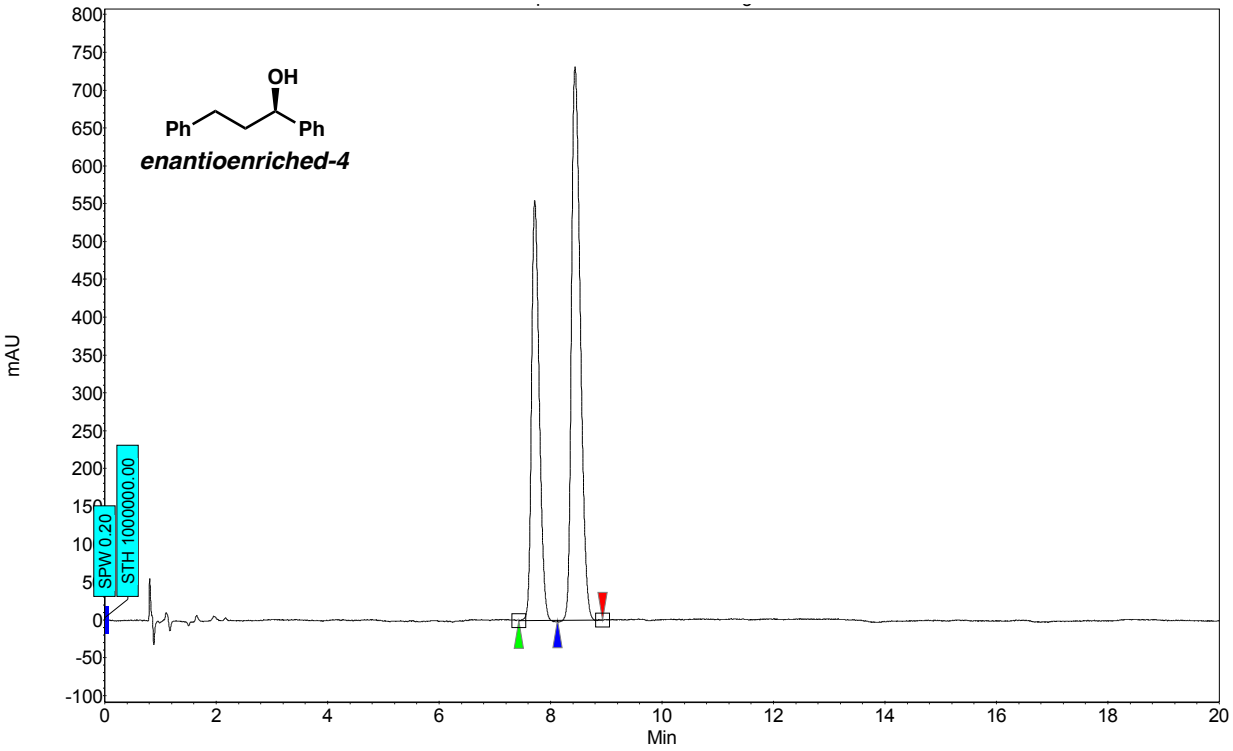
K. Verification of Enantioenrichment.

Chiral SFC Assay

Compound	Method Column/Temp.	Solvent	Method Flow Rate	Retention Times (min)	Enantiomeric Ratio (er)
 ***rac-4***	Daicel ChiralPak AD-H/35 °C	1% isopropanol in CO_2	3.5 mL/min	7.35/8.12	50:50
 ***enantioenriched-4***	Daicel ChiralPak AD-H/35 °C	1% isopropanol in CO_2	3.5 mL/min	7.43/8.12	40:60



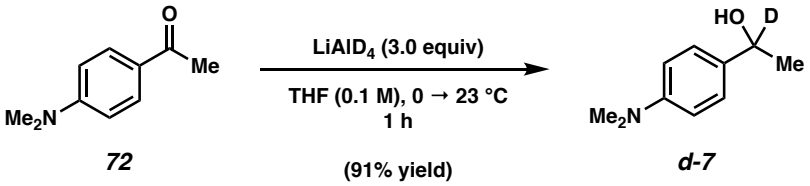
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	7.35	7.73	8.12	0.00	49.91	357.5	55.6	49.913
2	UNKNOWN	8.12	8.46	8.98	0.00	50.09	332.4	55.8	50.087
Total						100.00	689.9	111.4	100.000



Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	7.43	7.72	8.12	0.00	39.72	554.9	88.6	39.725
2	UNKNOWN	8.12	8.44	8.94	0.00	60.28	731.0	134.5	60.275
Total						100.00	1285.9	223.1	100.000

L. Deuterium Incorporation Experiments

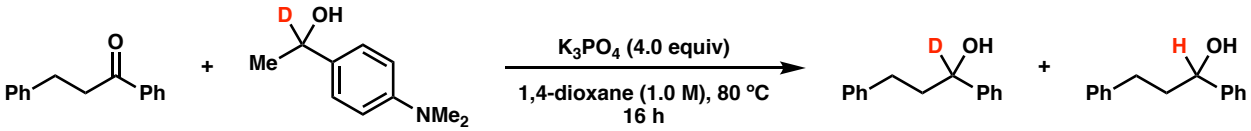
A. Preparation of deuterated reducing agent *d*-DMPE (*d*-7)



72 *d-7*

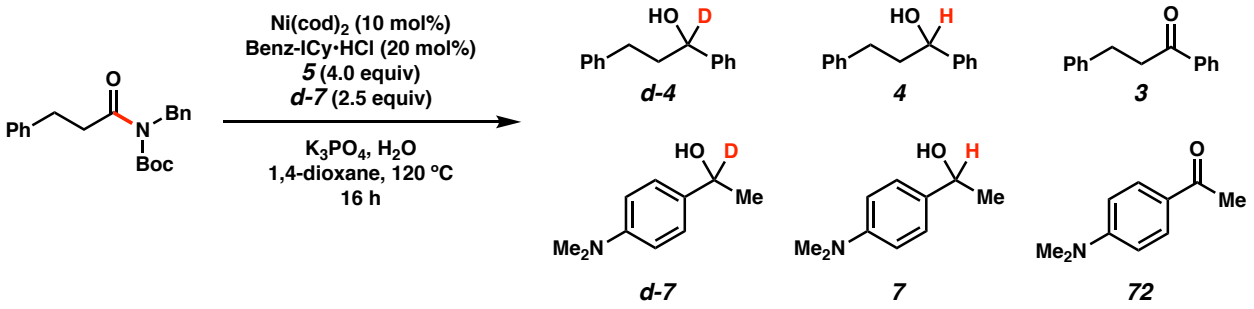
To a flame-dried flask equipped with a magnetic stir bar was added ketone **72** (50.0 mg, 0.307 mmol, 1.00 equiv) and THF (3.0 mL, 0.10 M). The flask was cooled to 0 °C and lithium aluminum deuteride (39 mg, 0.921 mmol. 3.00 equiv) was added in a single portion. The reaction was then warmed to 23 °C and stirred for 1 h. The reaction was cooled to 0 °C and quenched by the sequential addition of MeOH (5 mL), and deionized water (3 mL) and the resulting mixture was transferred to a separatory funnel with CH_2Cl_2 (10 mL) and water (10 mL). The aqueous layer was extracted with CH_2Cl_2 (3 x 10 mL), then the organic layers were combined, dried over Na_2SO_4, and evaporated under reduced pressure. Purification of the crude residue by flash chromatography (4:1 Hexanes:EtOAc) afforded deuterated alcohol ***d*-7** (46 mg, 91% yield) as a white solid. Alcohol **d-7**: R_f 0.33 (3:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.26 (d, J = 9.0, 2H), 6.73 (d, J = 9.0, 2H), 2.94 (s, 3 H), 1.62 (s, 1H), 1.48 (s, 3H).

A. Deuterium incorporation experiments using *d*-DMPE (*d*-7).

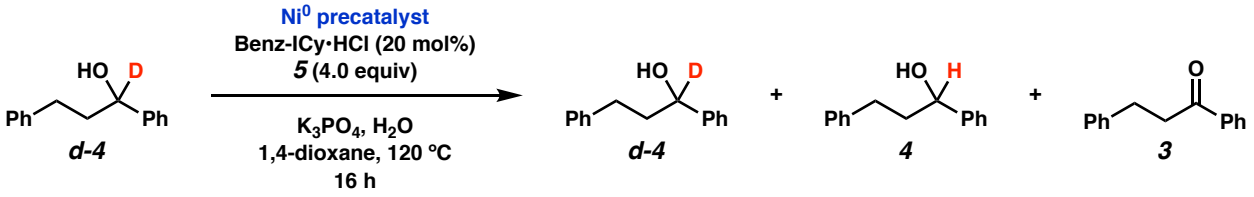


3 *d-7* *(2.5 equiv, >99% D)* *d-4* *4*

Conversion	Yield of *d*-4	Yield of 4
75%	75%	0%



Conversion	Yield *d*-4	Yield *4*	Yield *3*	Yield *d-7*	Yield *7*	Yield *72*
100%	0%	72%	15%	58%	1%	30%



Ni^0 precatalyst	Yield *d*-4	Yield *4*	Yield *3*
None	95%	0%	0%
Ni(cod)$_2$ (10 mol%)	12%	22%	20%

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derivatives and related compounds as potential antidepressant drugs with high affinity for serotonin (5-HT) and norepinephrine (NE) transporters. *J. Med. Chem*. **2003**, *46*, 5512–5532.

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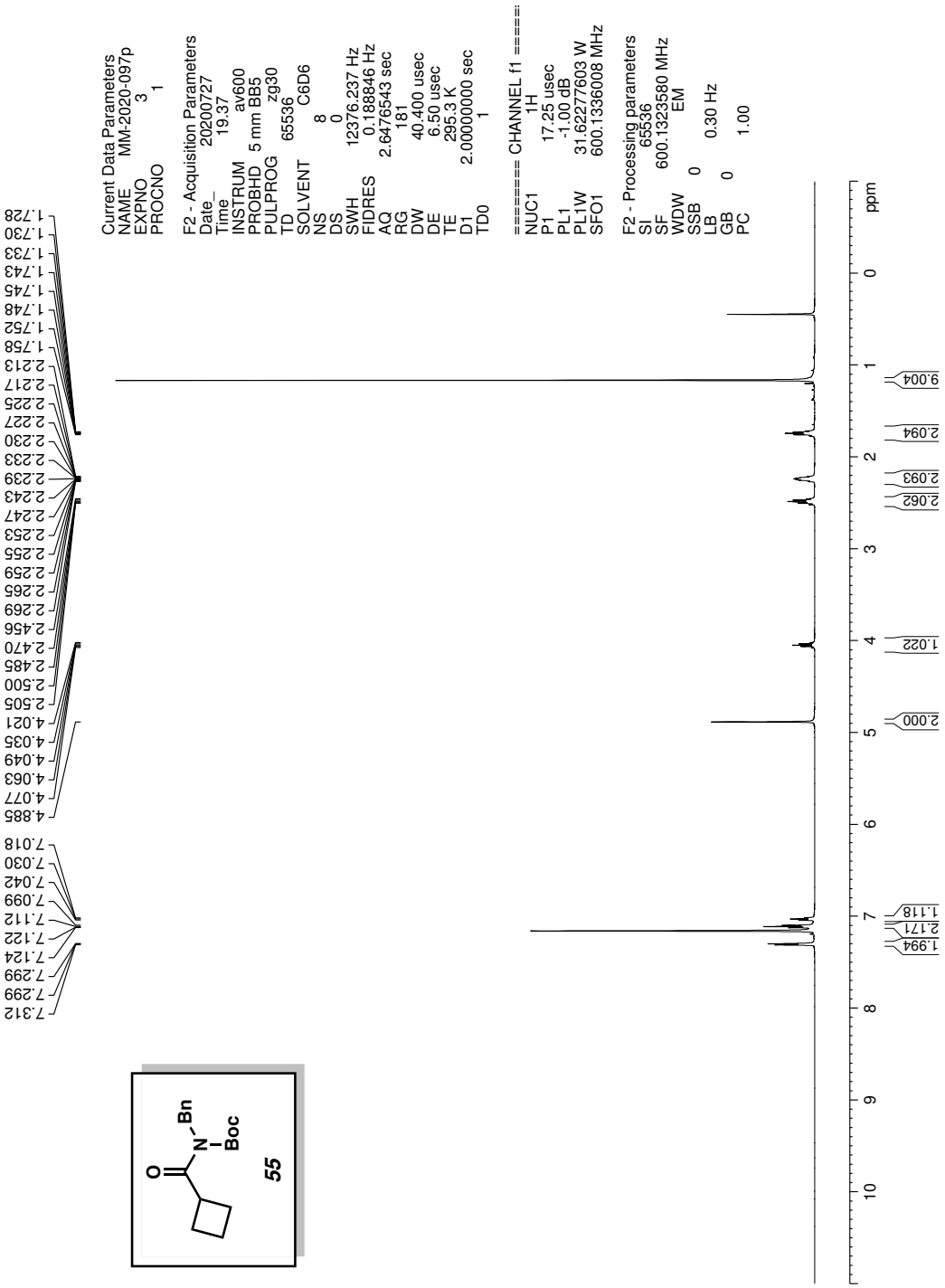
^{1}H NMR Spectra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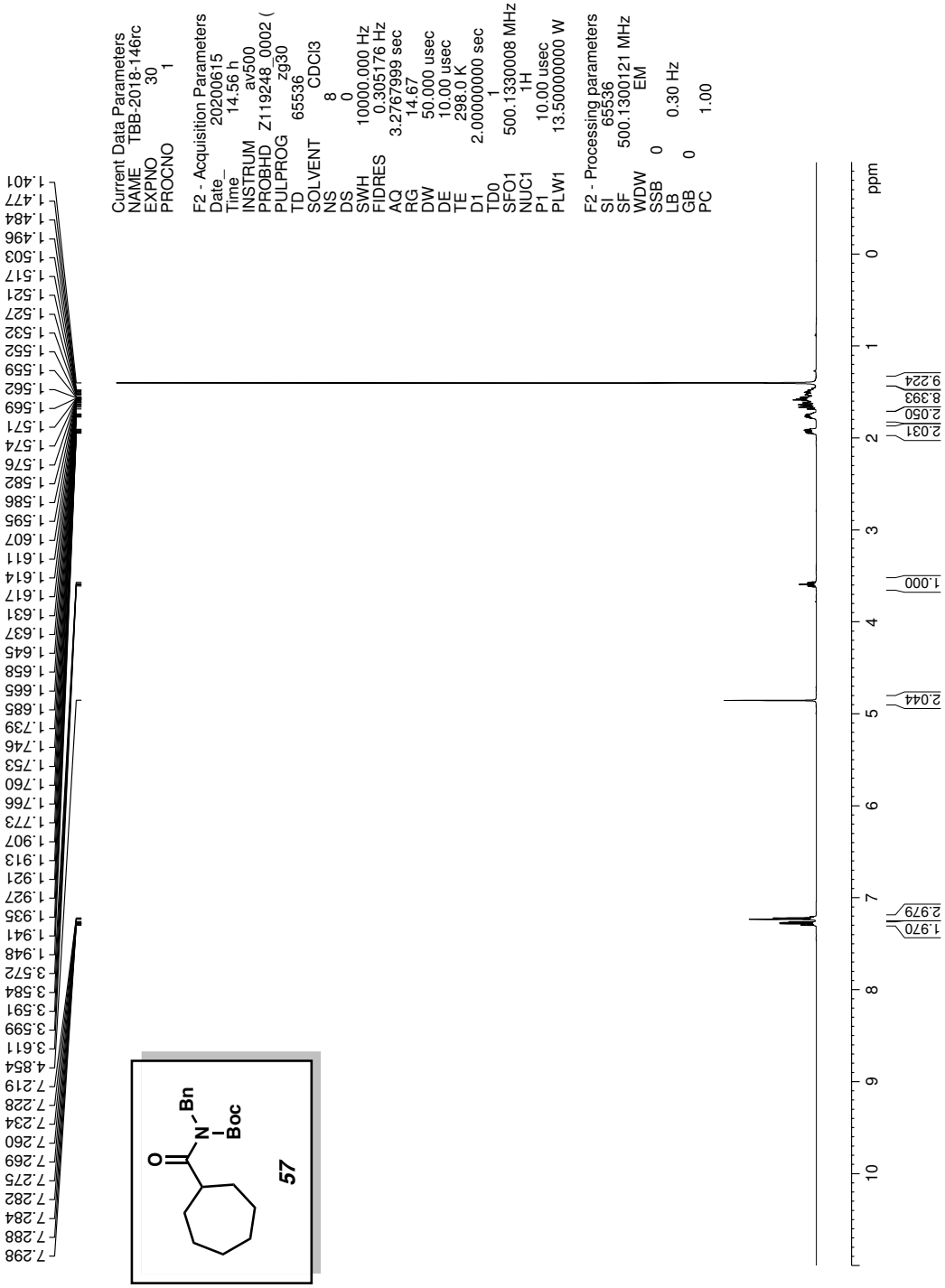
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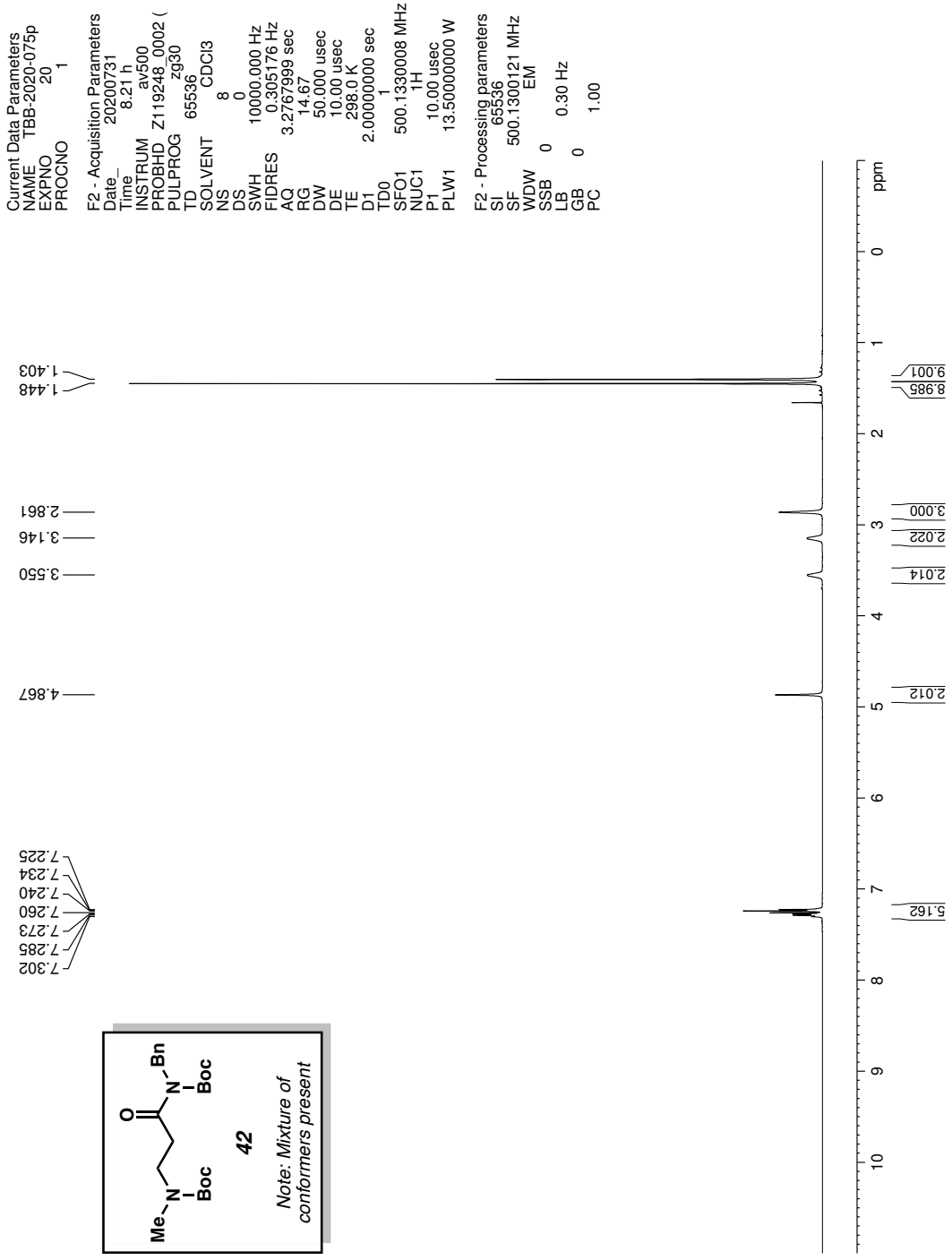
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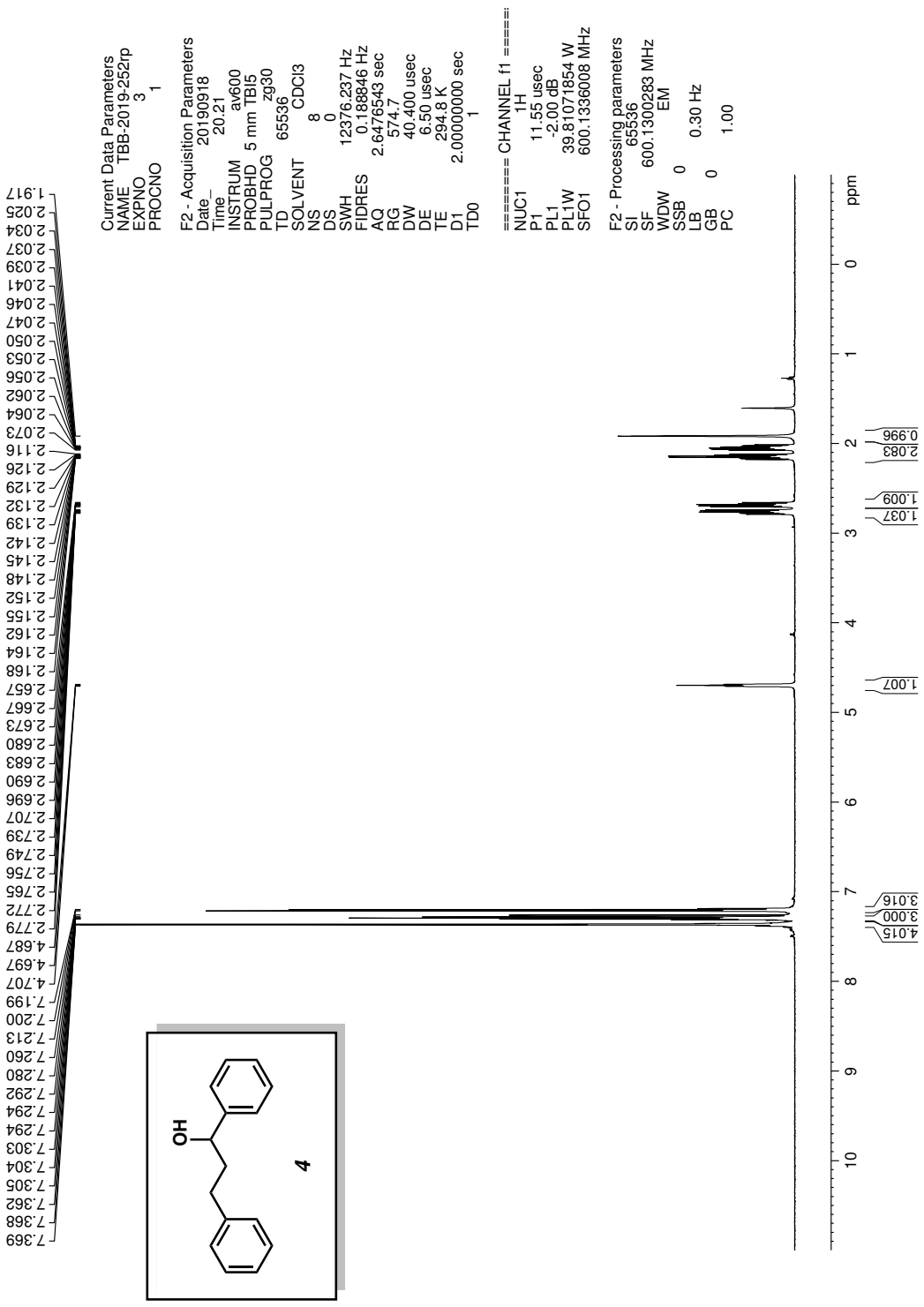
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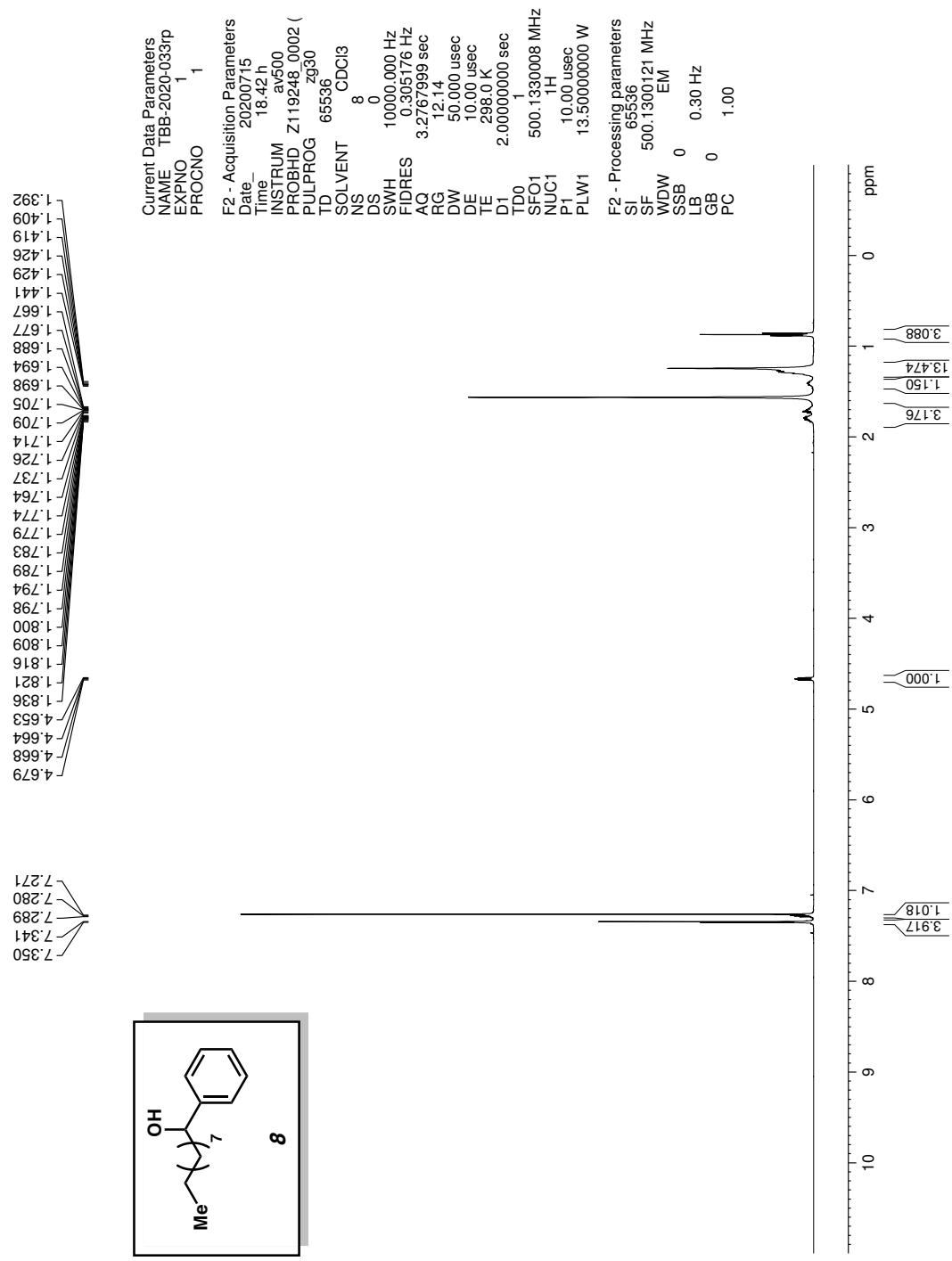
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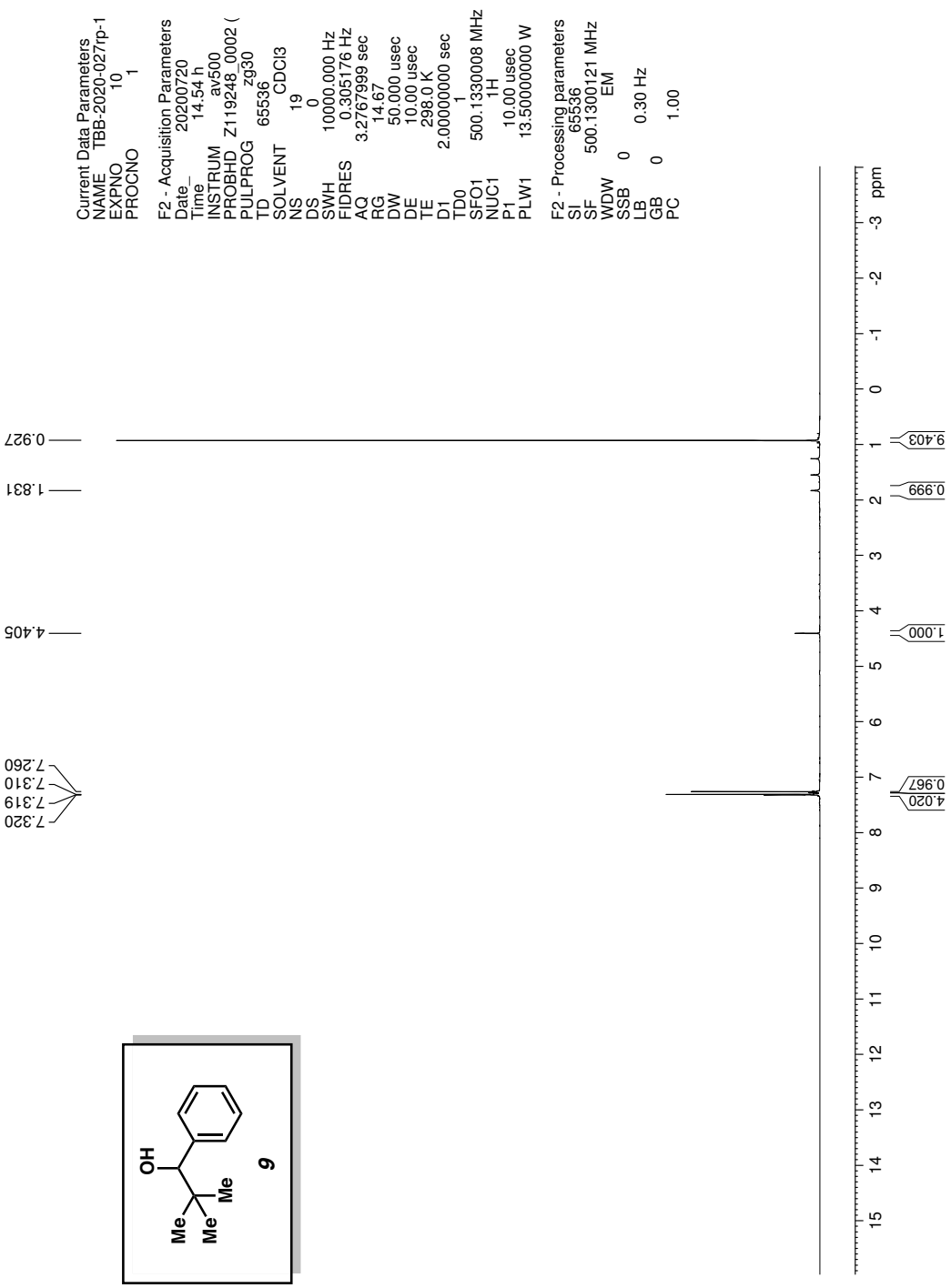
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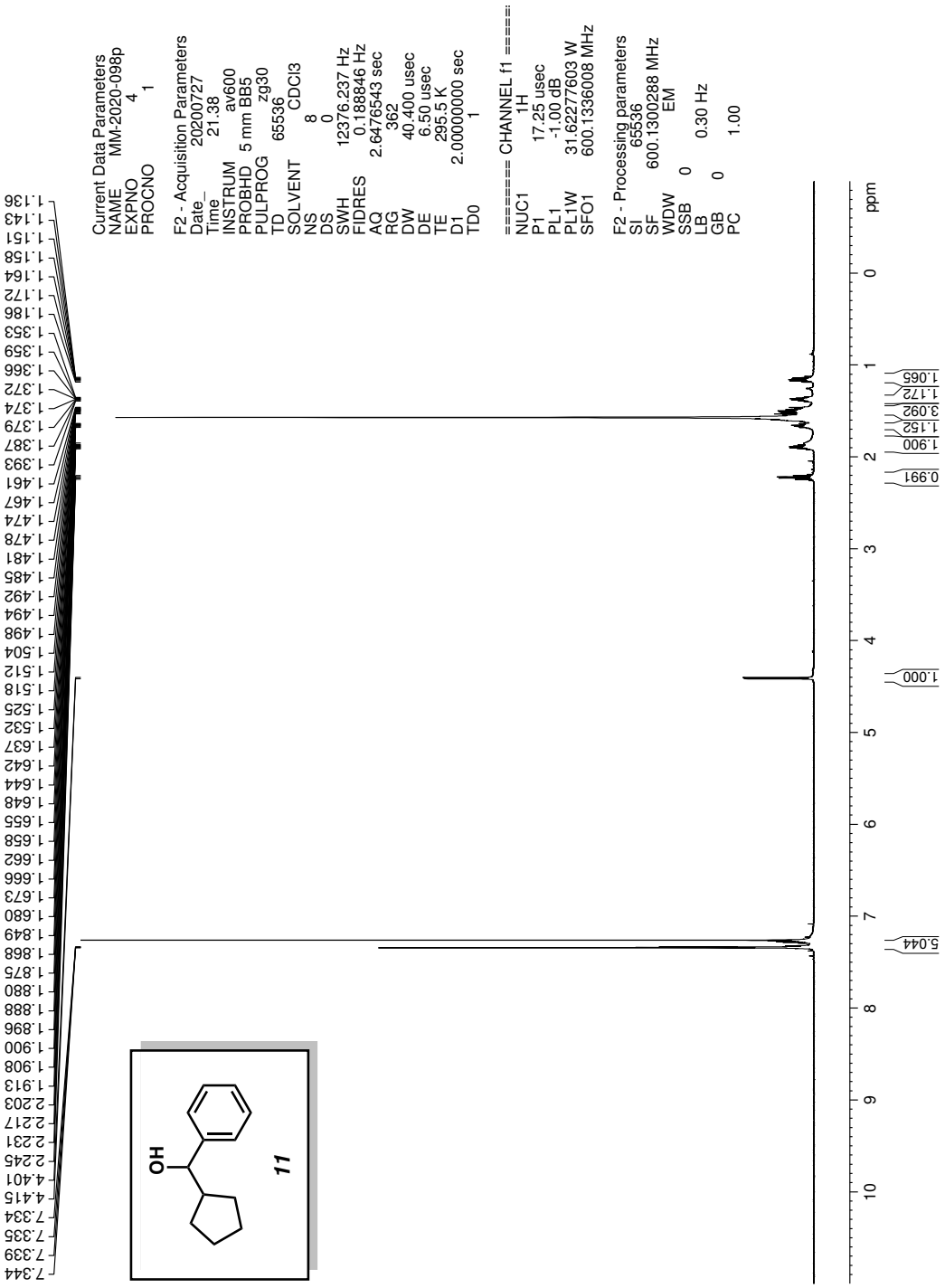
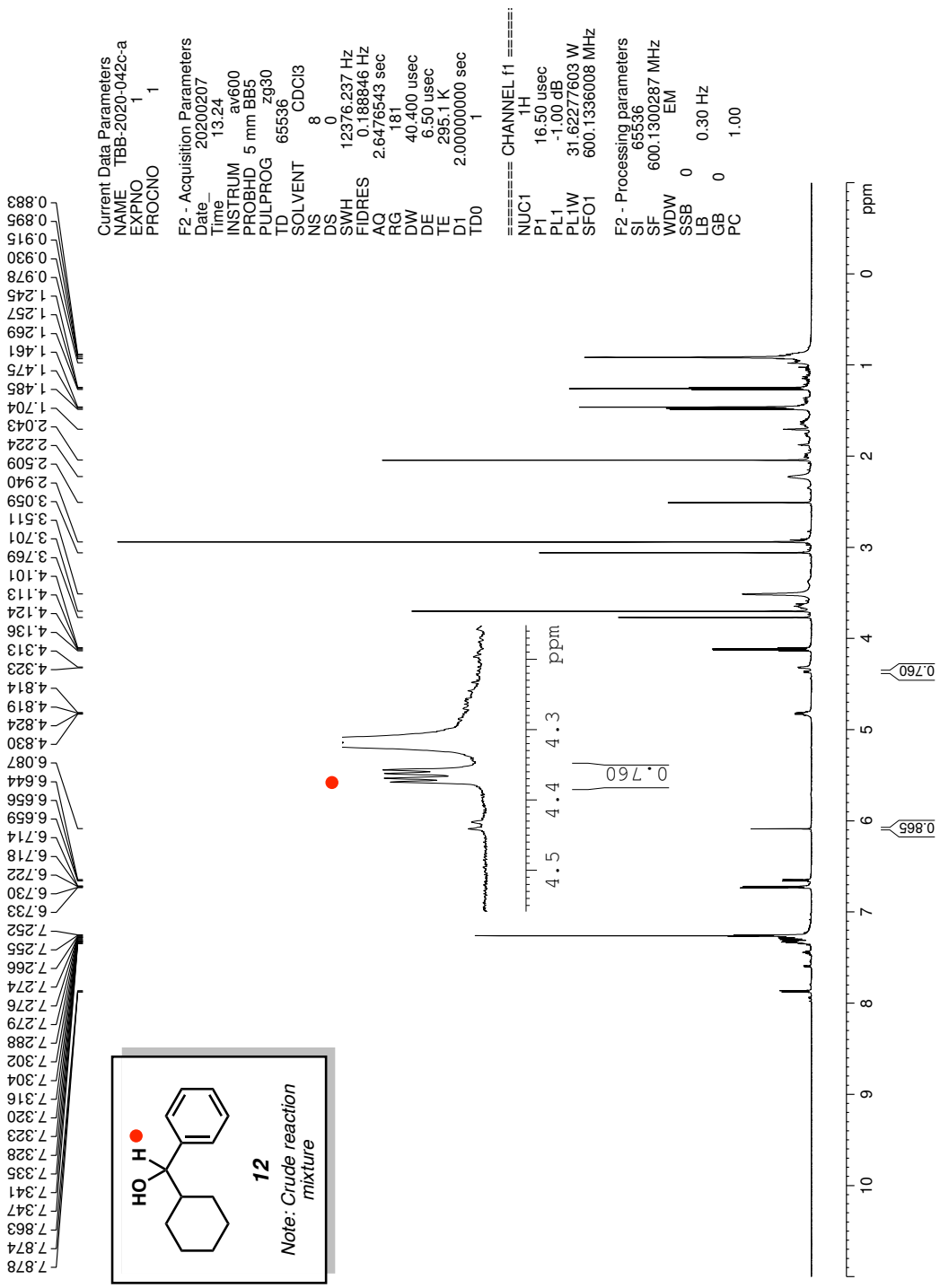
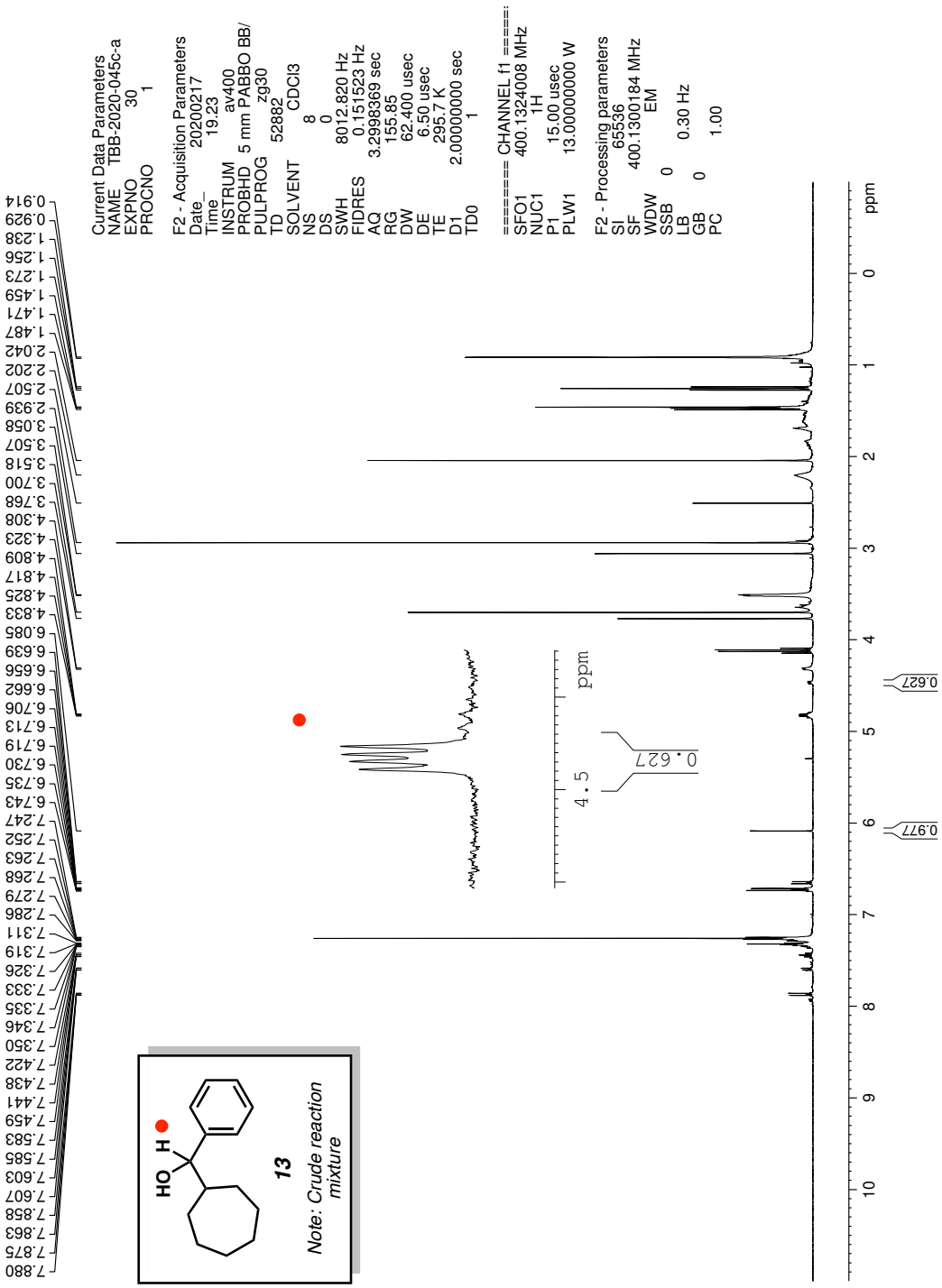
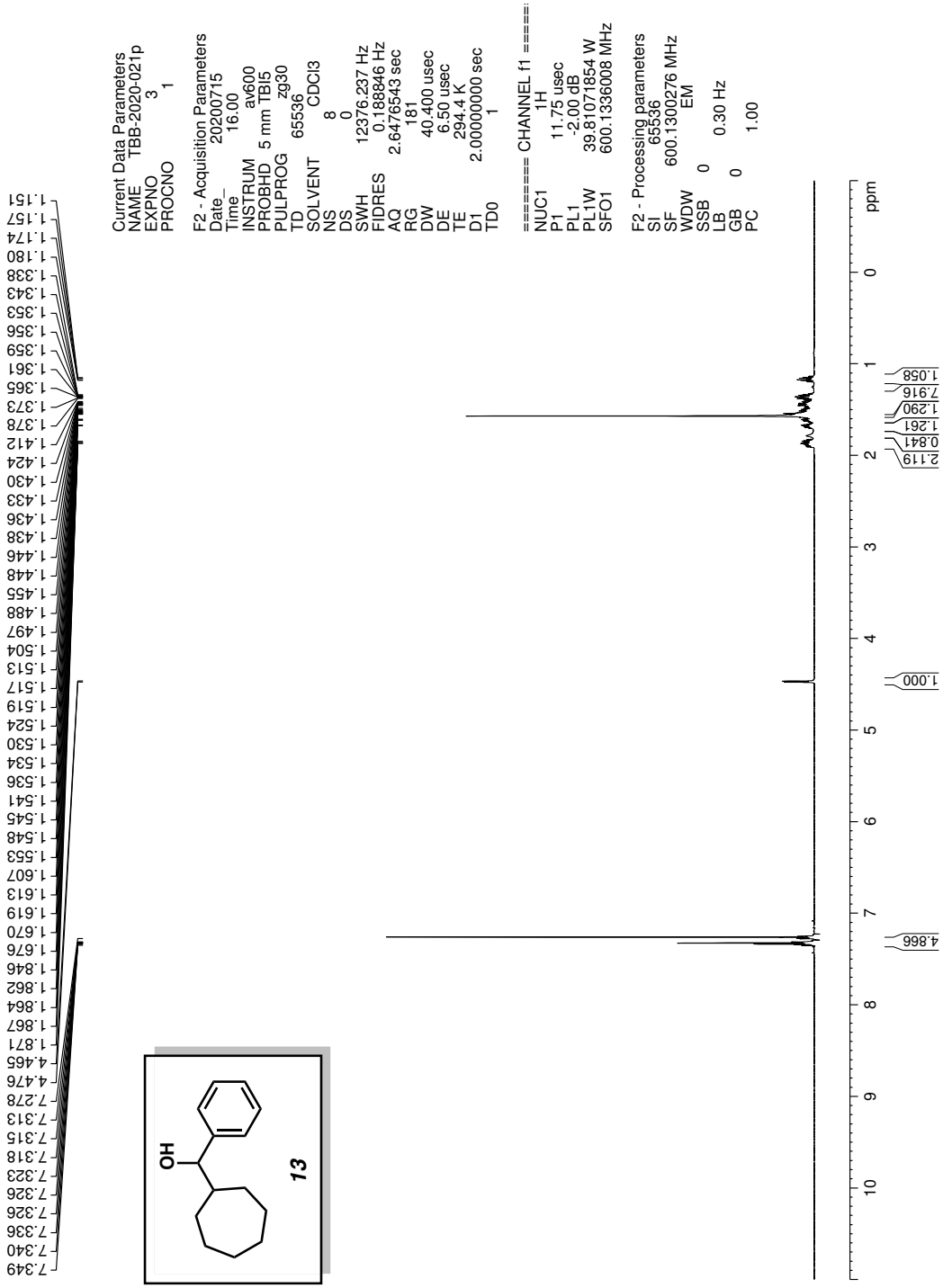
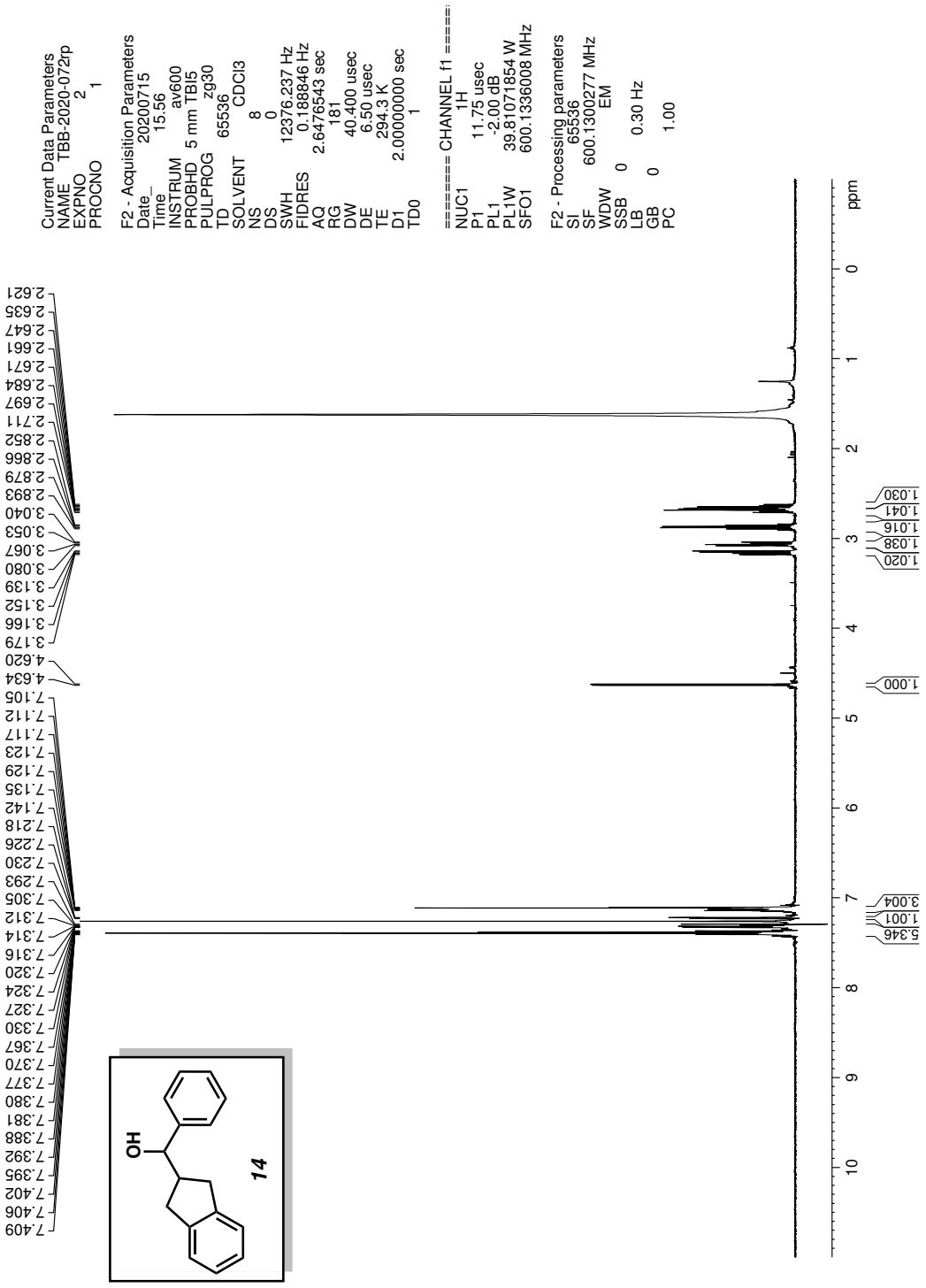
7.319
7.310
7.260
9.403
0.999
1.000
4.020
0.967
OH
Me
Me
Me
9
ppm

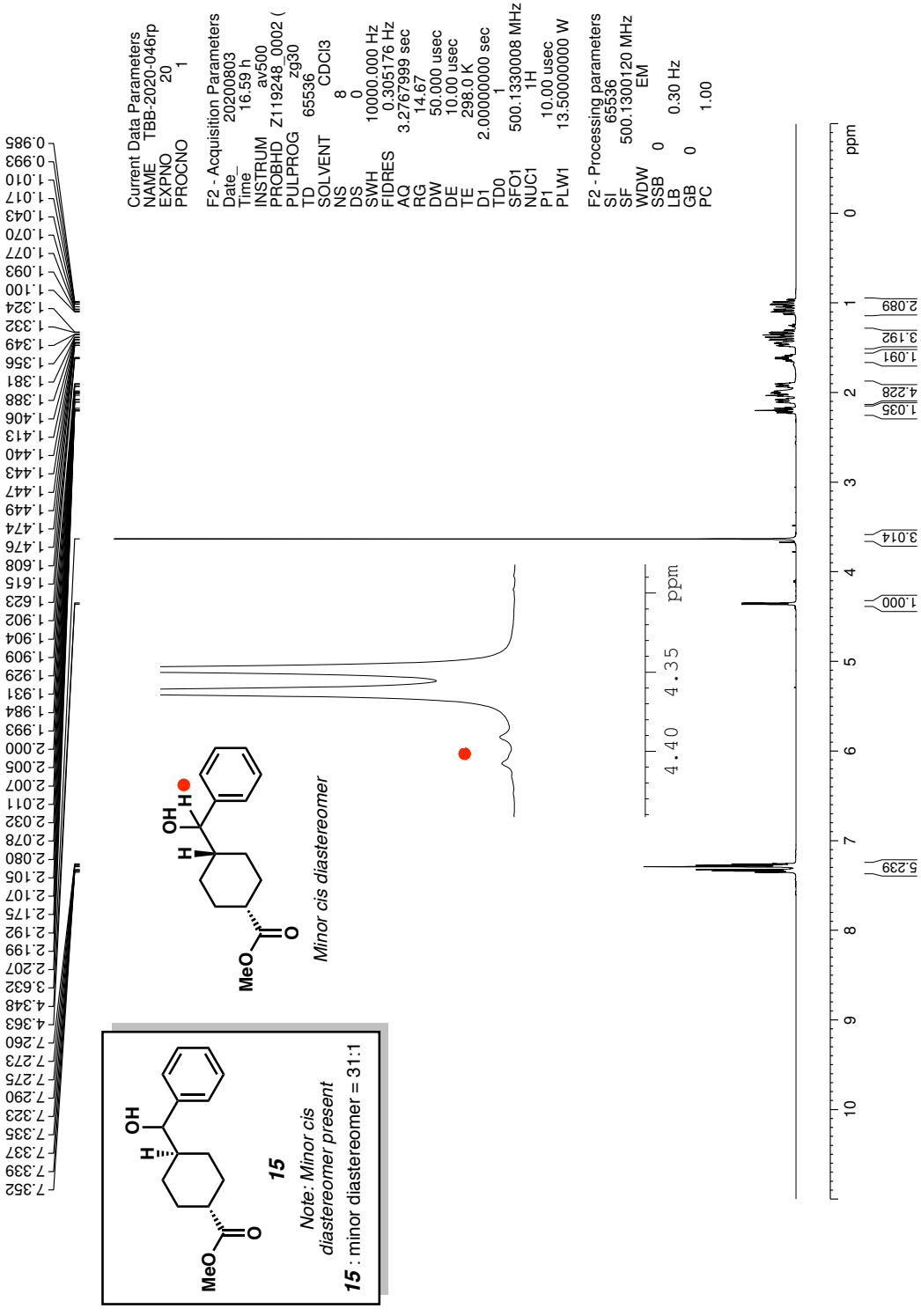

Current Data Parameters
NAME MM-2020-098p
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200727
Time 21.38
INSTRUM av600
PROBHD 5 mm BB5
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6476543 sec
RG 362
DW 40.400 usec
DE 6.50 usec
TE 295.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 17.25 usec
PL1 -1.00 dB
PL1W 31.62277603 W
SFO1 600.1336008 MHz
F2 - Processing parameters
SI 65536
SF 600.1300288 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
11
ppm
5.044
1.000
0.991
1.900
1.152
3.092
1.172
1.065

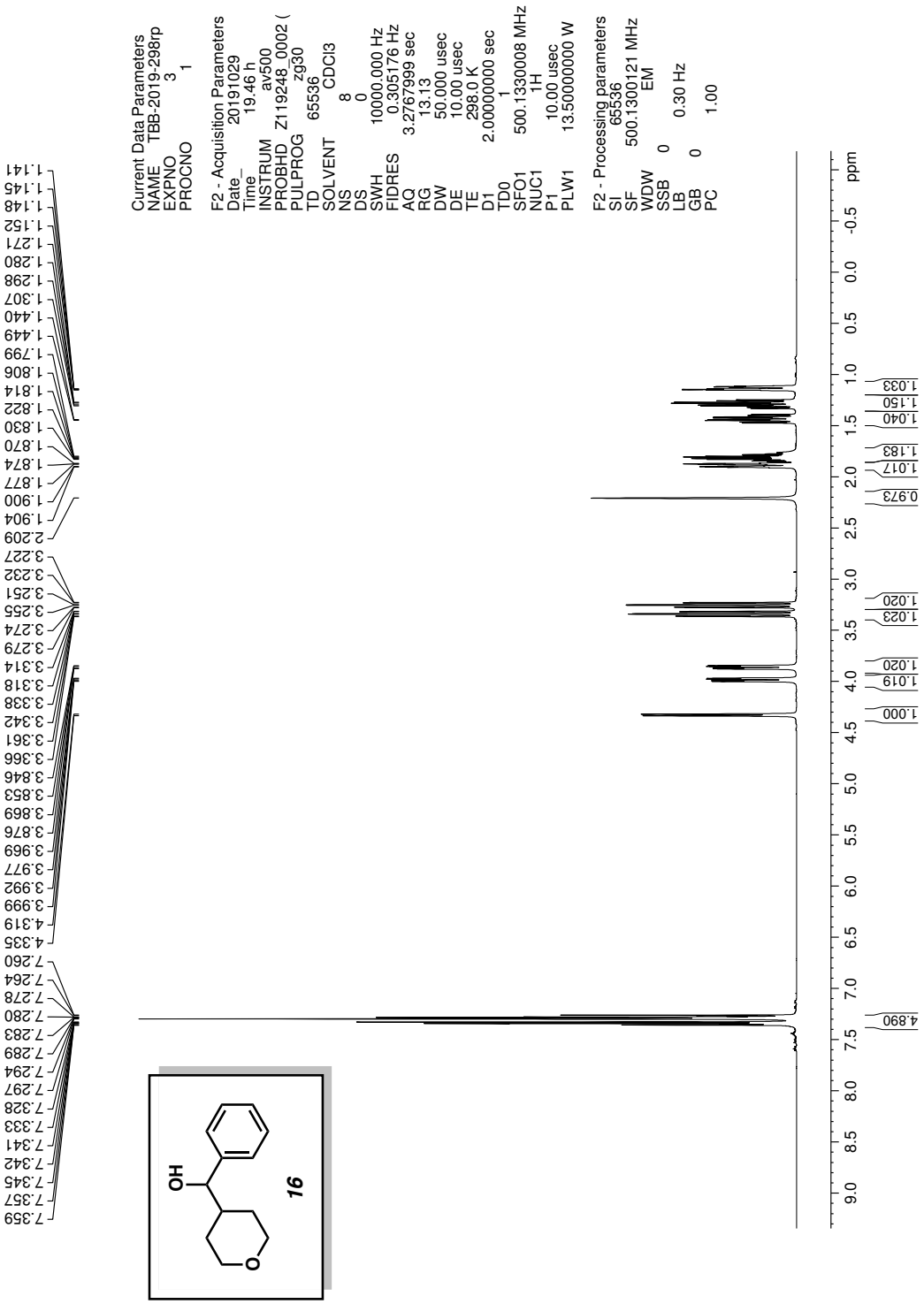

Current Data Parameters
NAME TBB-2020-042c-a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200207
Time 13.24
INSTRUM av600
PROBHD 5 mm BB5
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6476543 sec
RG 181
DW 40.400 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 16.50 usec
PL1 -1.00 dB
PL1W 31.62277603 W
SFO1 600.1336008 MHz
F2 - Processing parameters
SI 65536
SF 600.1300287 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.760
0.865
ppm
12
Note: Crude reaction mixture

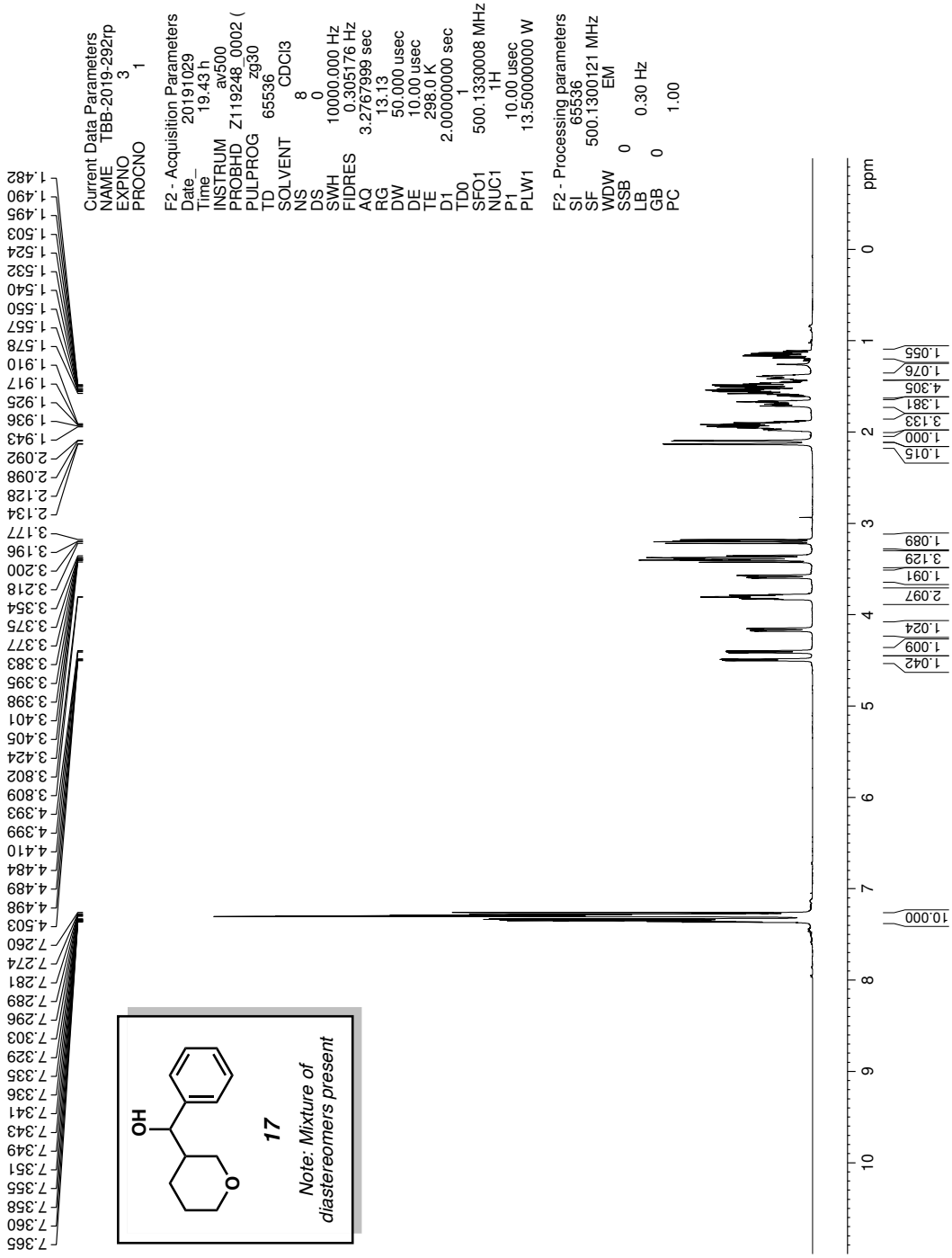

Current Data Parameters
NAME TBB-2020-045c-a
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200217
Time 19.23
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 155.85
DW 62.400 usec
DE 6.50 usec
TE 295.7 K
D1 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
HO H
13
Note: Crude reaction mixture
0.627
4.5
ppm
0.627
0.977
ppm

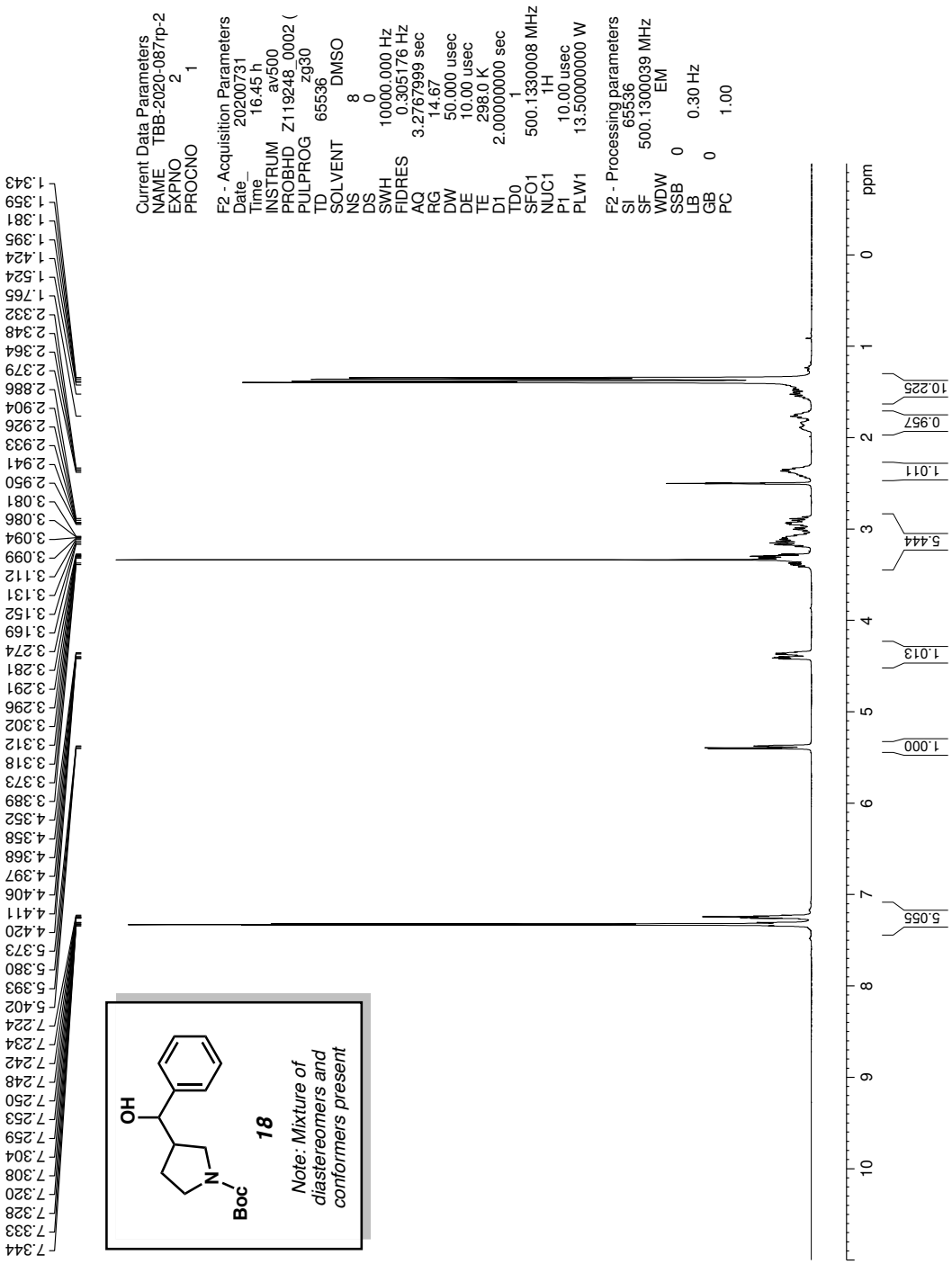

Current Data Parameters
NAME TBB-2020-021p
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200715
Time 16.00
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6476543 sec
RG 181
DW 40.400 usec
DE 6.50 usec
TE 294.4 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.75 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1336008 MHz
F2 - Processing parameters
SI 65536
SF 600.1300276 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
13
ppm
4.866
1.000
2.119
0.841
1.261
1.290
7.916
1.058

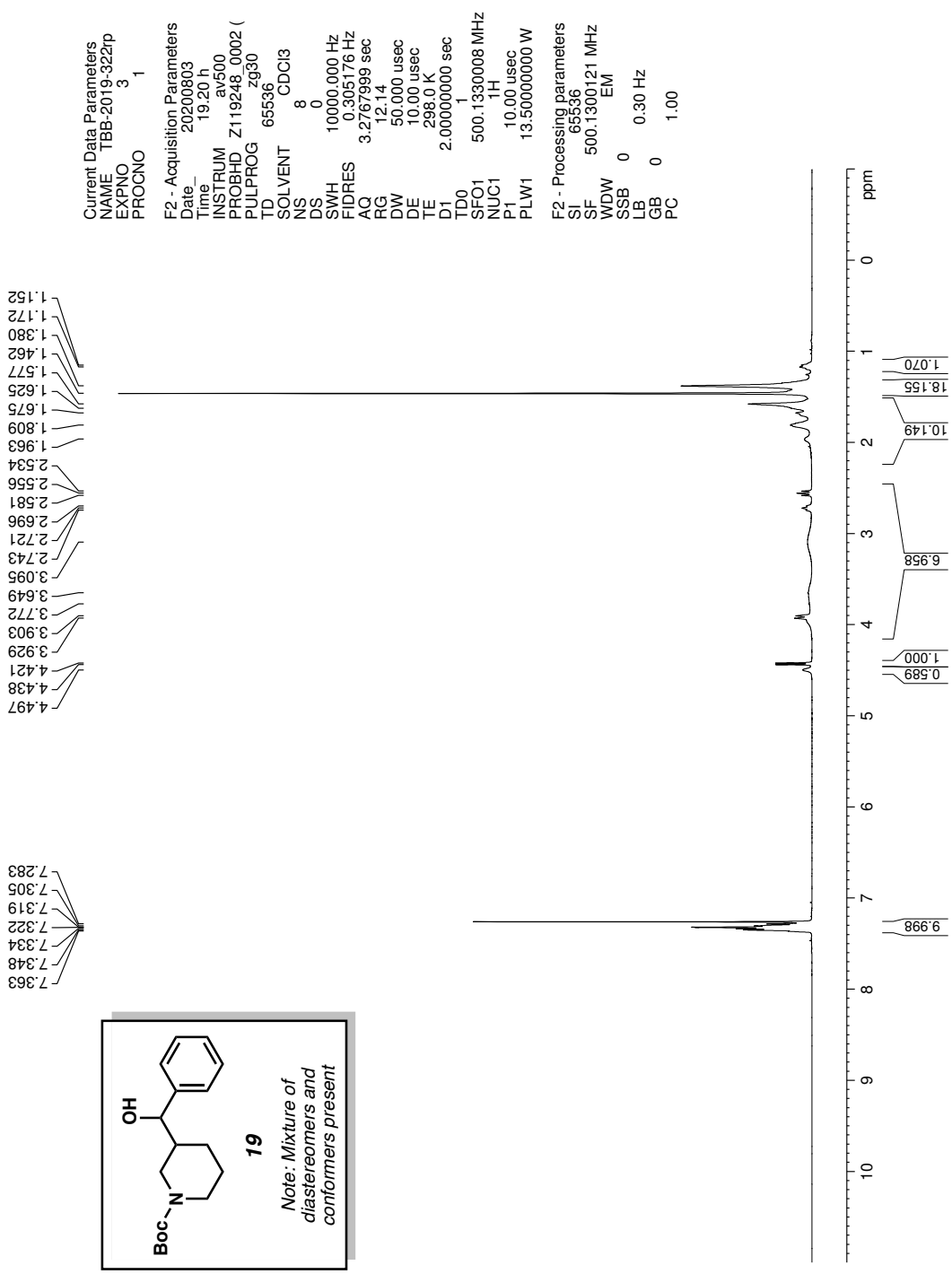

Current Data Parameters
NAME TBB-2020-072rp
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200715
Time 15.56
INSTRUM av600
PROBHD 5 mm TBI5
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6476543 sec
RG 181
DW 40.400 usec
DE 6.50 usec
TE 294.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.75 usec
PL1 -2.00 dB
PL1W 39.81071854 W
SFO1 600.1336008 MHz
F2 - Processing parameters
SI 65536
SF 600.1300277 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
14
ppm
1.030
1.041
1.016
1.038
1.020
1.000
3.004
1.001
5.346

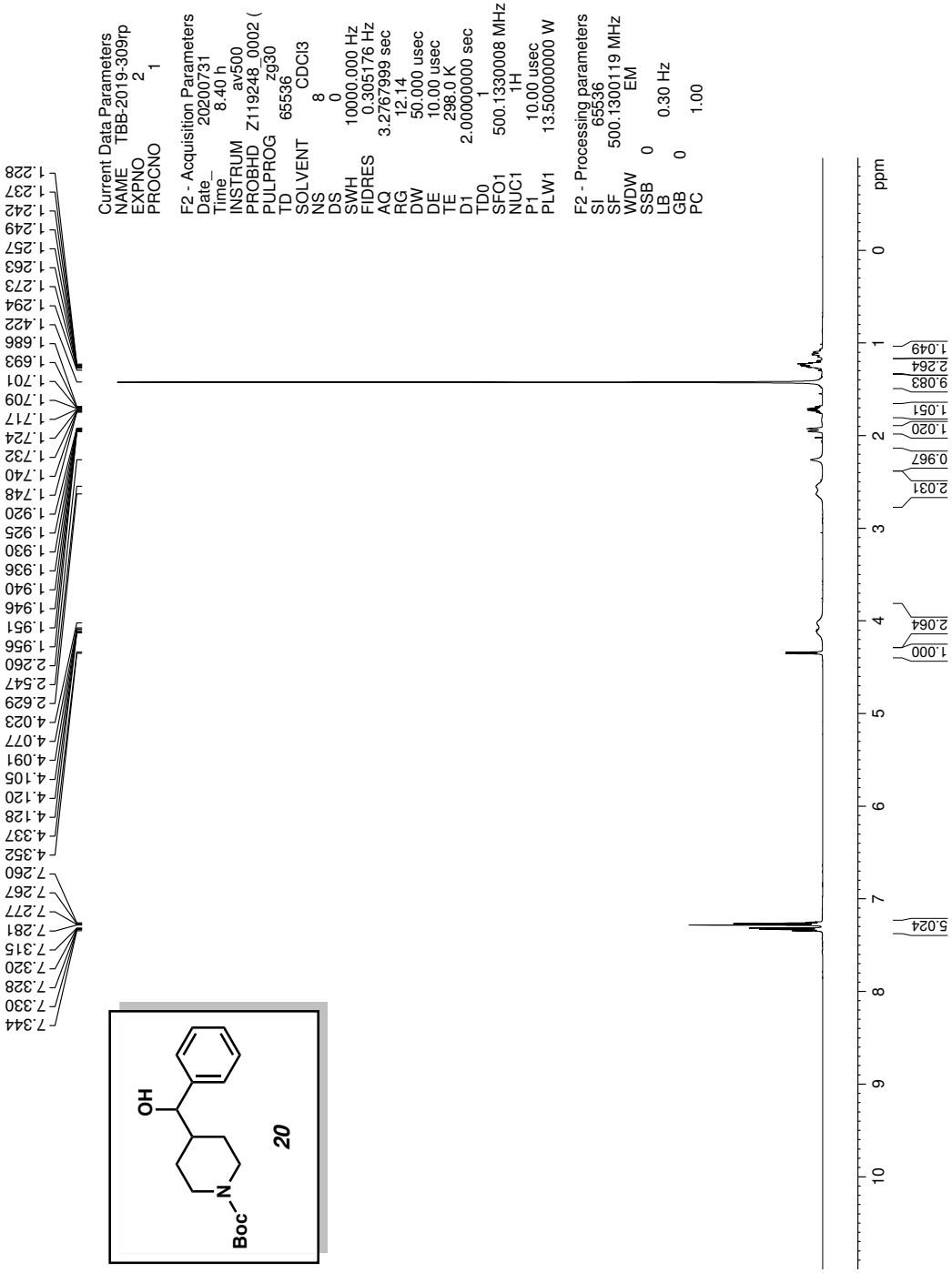

Current Data Parameters
NAME TBB-2020-046rp
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200803
Time 16.59 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 14.67
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
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 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
H
MeO
O
Minor cis diastereomer
4.40
4.35
ppm
5.239
3.014
1.000
2.089
3.192
1.091
4.228
1.035
ppm
15
Note: Minor cis diastereomer present
15 : minor diastereomer = 31:1

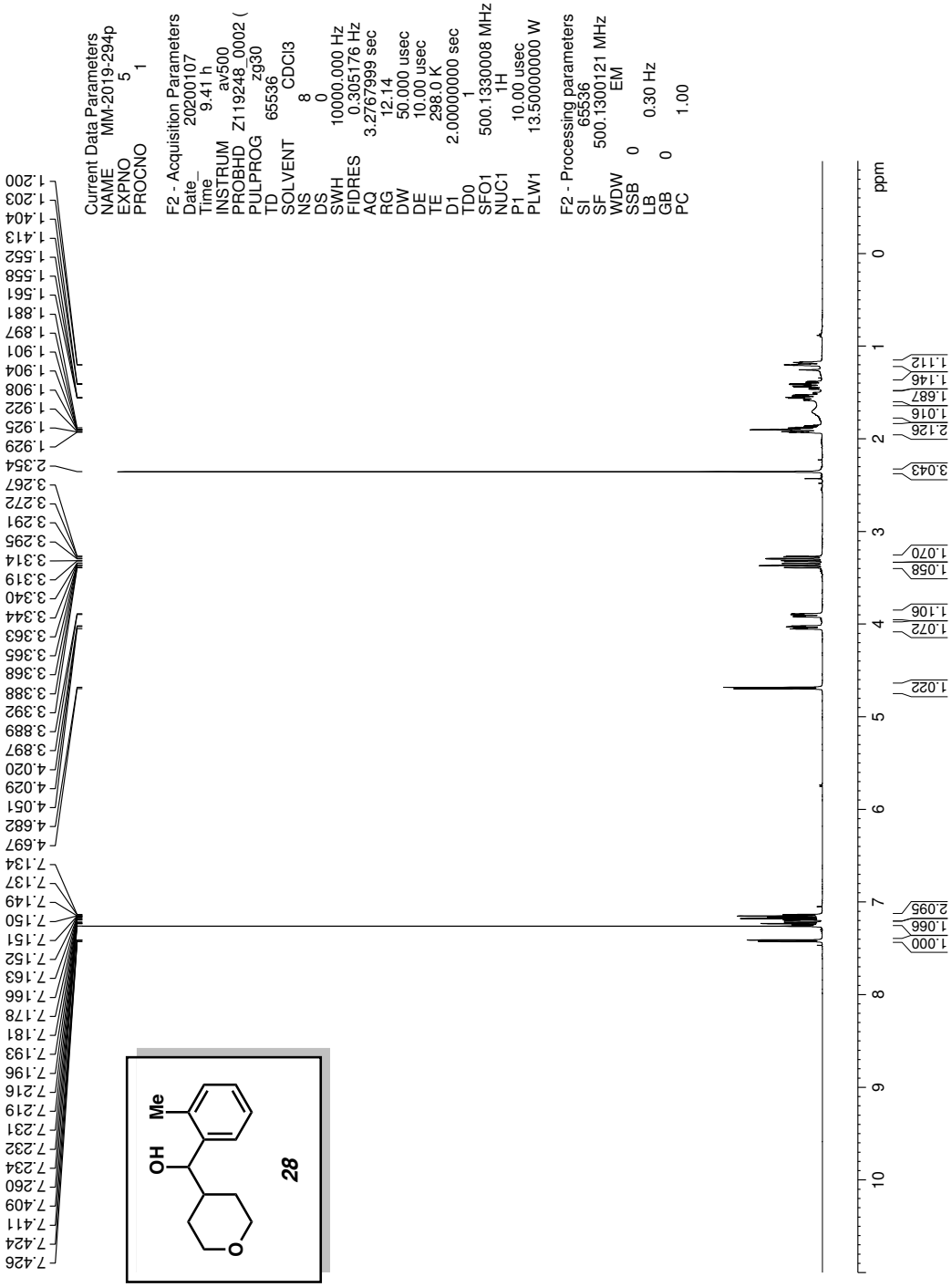

Current Data Parameters
NAME TBB-2019-298rp
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191029
Time 19.46 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.3051176 Hz
AQ 3.2767999 sec
RG 13.13
DW 50.000 usec
DE 10.000 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
O
16
4.890
1.000
1.019
1.020
1.020
1.023
1.020
0.973
1.017
1.183
1.040
1.150
1.033
ppm

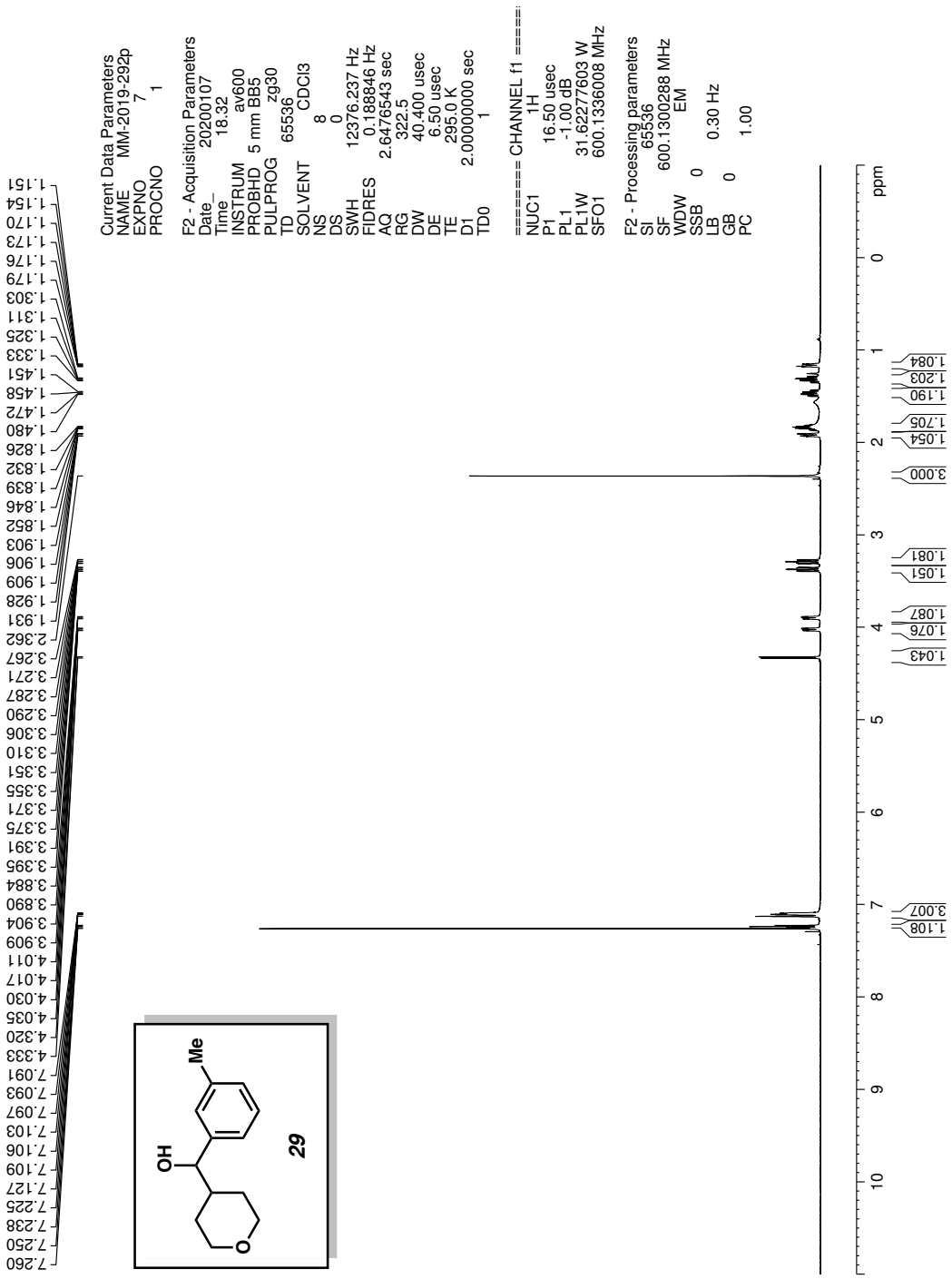

Current Data Parameters
NAME TBB-2019-292rp
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191029
Time 19.43 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 13.13
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
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
OH
O
17
Note: Mixture of diastereomers present
ppm
10.000
1.042
1.009
1.024
2.097
1.091
3.129
1.089
1.015
1.000
3.133
1.381
4.305
1.076
1.055

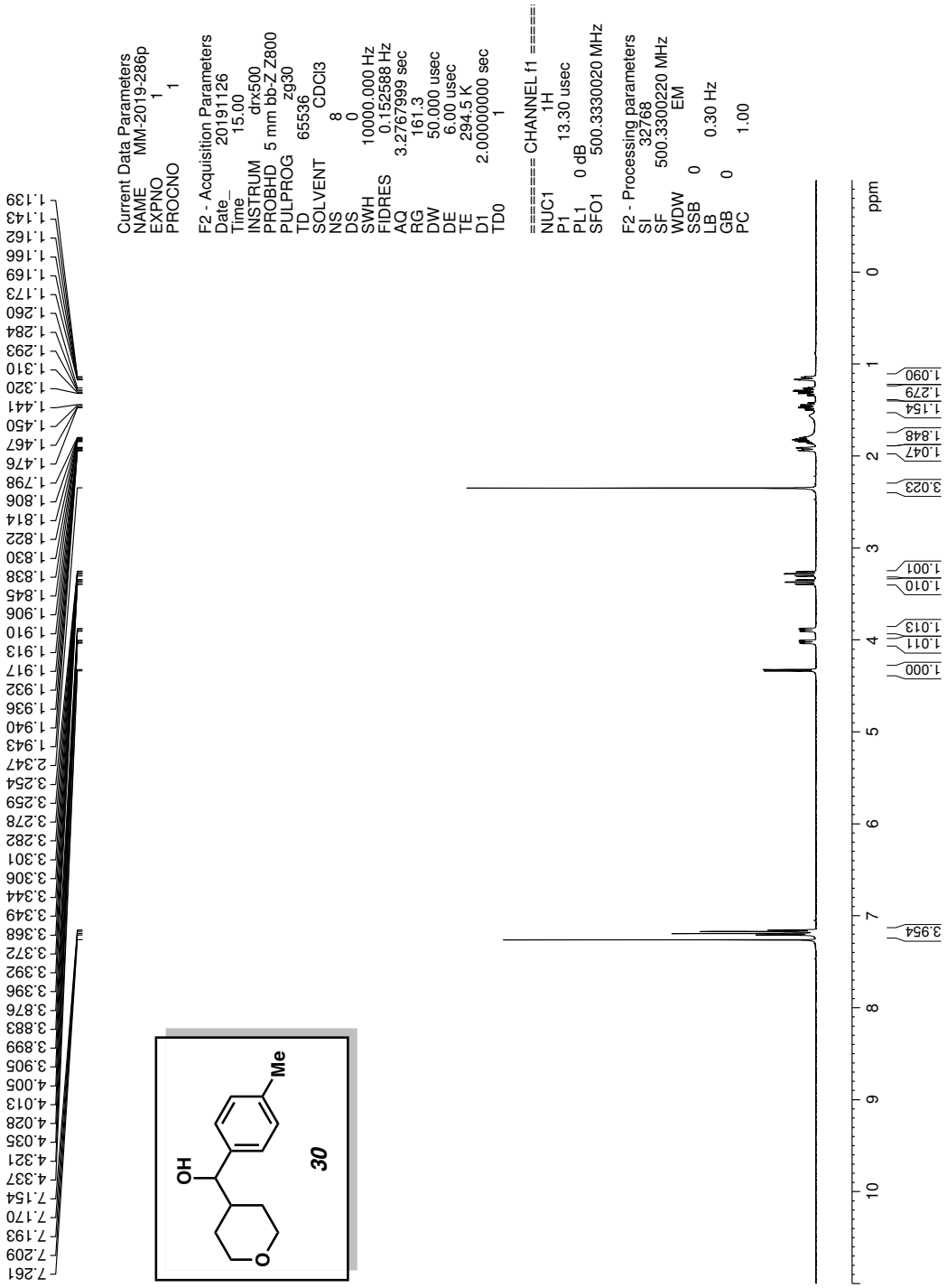

Current Data Parameters
NAME TBB-2020-087rp-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200731
Time_ 16.45 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 14.67
DW 50.000 usec
DE 10.00 usec
TE 298.0 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 500.1300039 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
N
Boc
18
Note: Mixture of diastereomers and conformers present
ppm
5.055
1.000
1.013
5.444
1.011
0.957
10.225

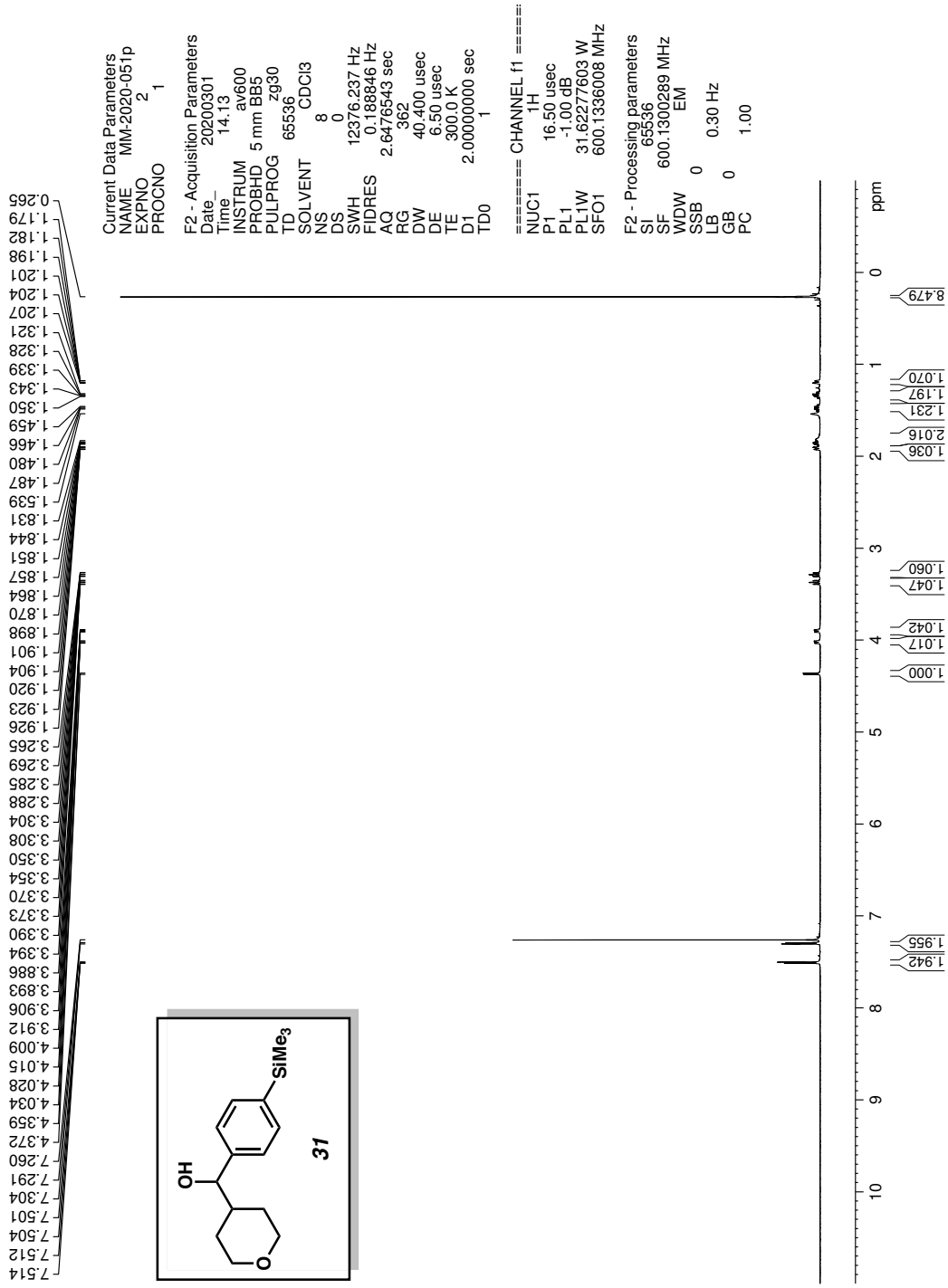

Current Data Parameters
NAME TBB-2019-322rp
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200803
Time 19.20 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.363
7.348
7.334
7.322
7.319
7.305
7.283
4.497
4.438
4.421
3.929
3.903
3.772
3.649
3.095
2.743
2.721
2.696
2.581
2.556
2.534
1.963
1.809
1.675
1.625
1.577
1.462
1.380
1.172
1.152
9.998
1.070
18.155
10.149
6.958
1.000
0.589
ppm
OH
Boc
N
19
Note: Mixture of diastereomers and conformers present

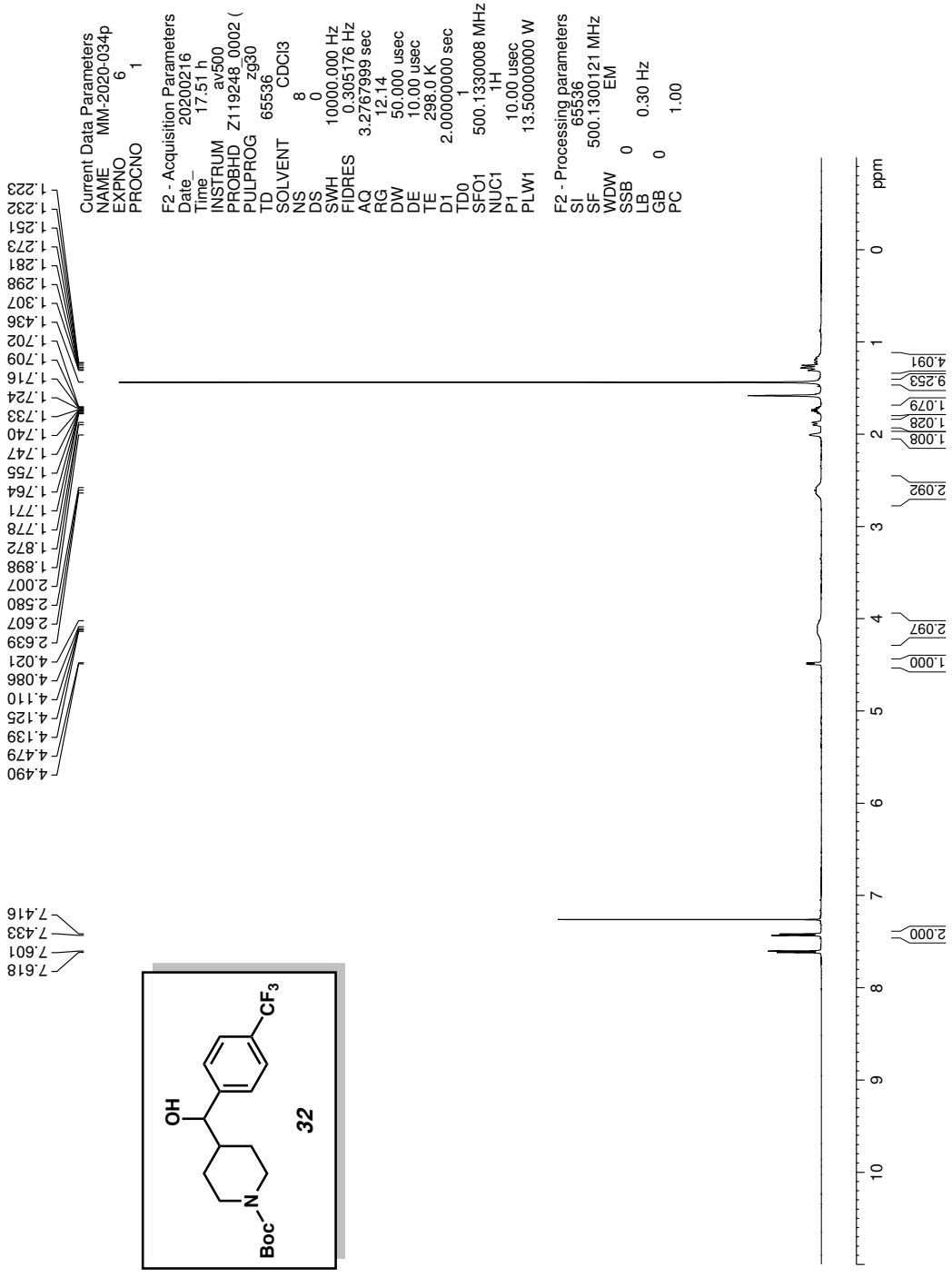

Current Data Parameters
NAME TBB-2019-309rp
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200731
Time 8.40 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 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 500.1300119 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
N
Boc
20
ppm
1.000
2.064
2.031
0.967
1.020
1.051
9.083
2.264
1.049
5.024

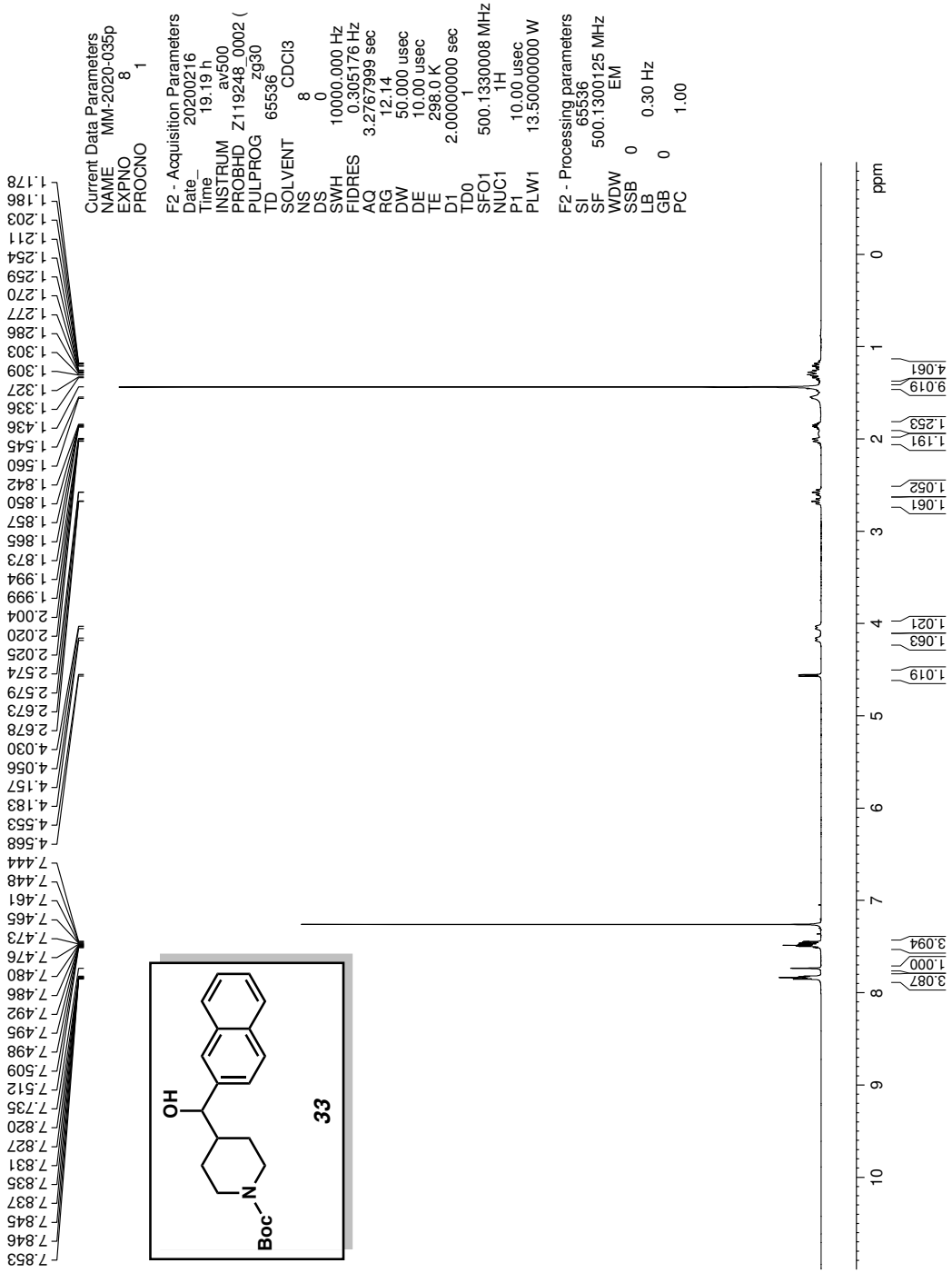

Current Data Parameters
NAME MM-2019-294p
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200107
Time_ 9.41 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 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
OH
Me
O
28
ppm
1.112
1.146
1.687
1.016
2.126
3.043
1.058
1.070
1.072
1.106
1.022
2.095
1.066
1.000

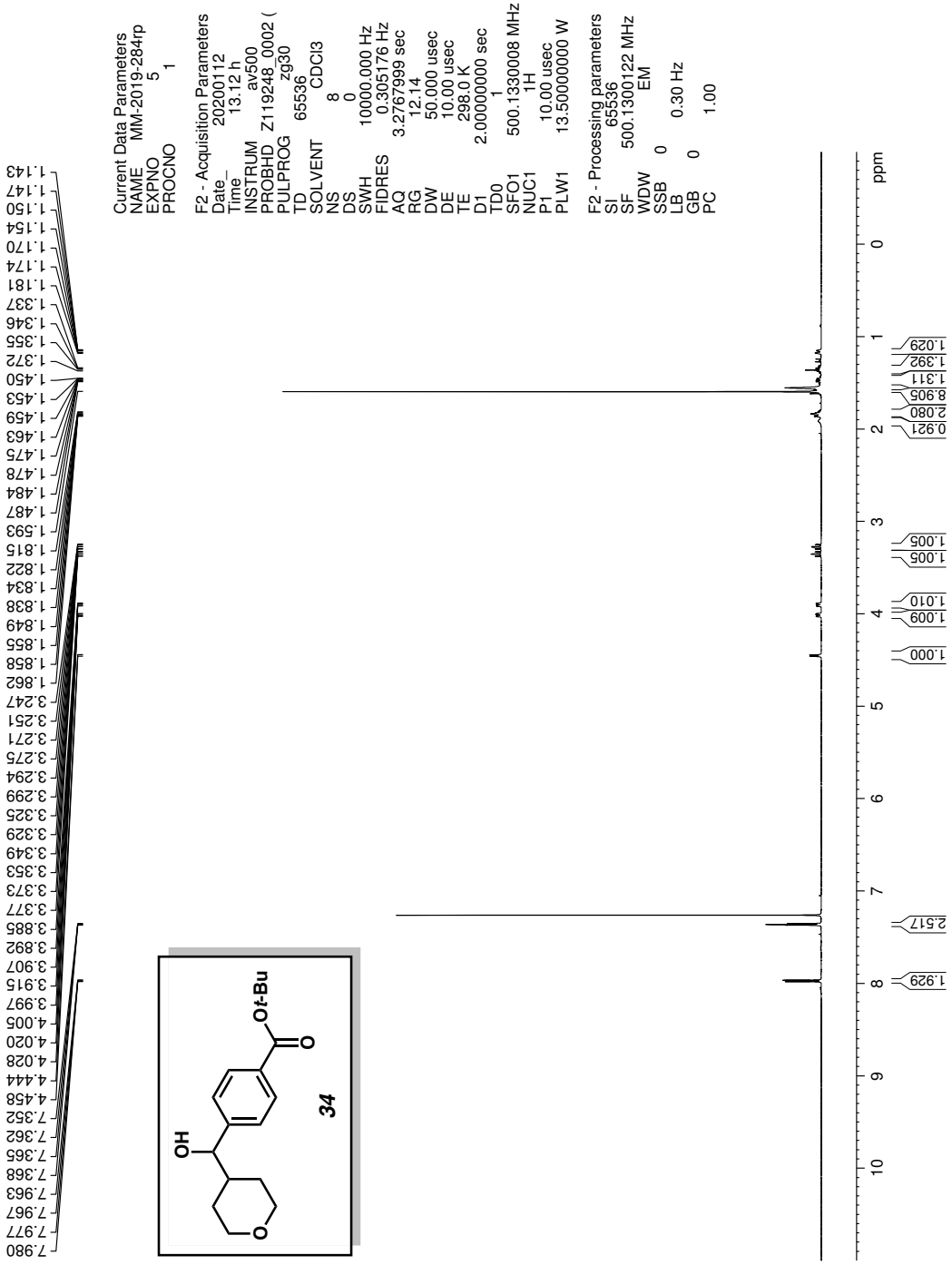

Current Data Parameters
NAME MM-2019-292p
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200107
Time 18.32
INSTRUM av600
PROBHD 5 mm BB5
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.64476543 sec
RG 322.5
DW 40.400 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 16.50 usec
PL1 -1.00 dB
PL1W 31.62277603 W
SFO1 600.1336008 MHz
F2 - Processing parameters
SI 65536
SF 600.1300288 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
Me
O
29
ppm
1.108
3.007
1.043
1.076
1.087
1.051
1.081
3.000
1.054
1.705
1.190
1.203
1.084

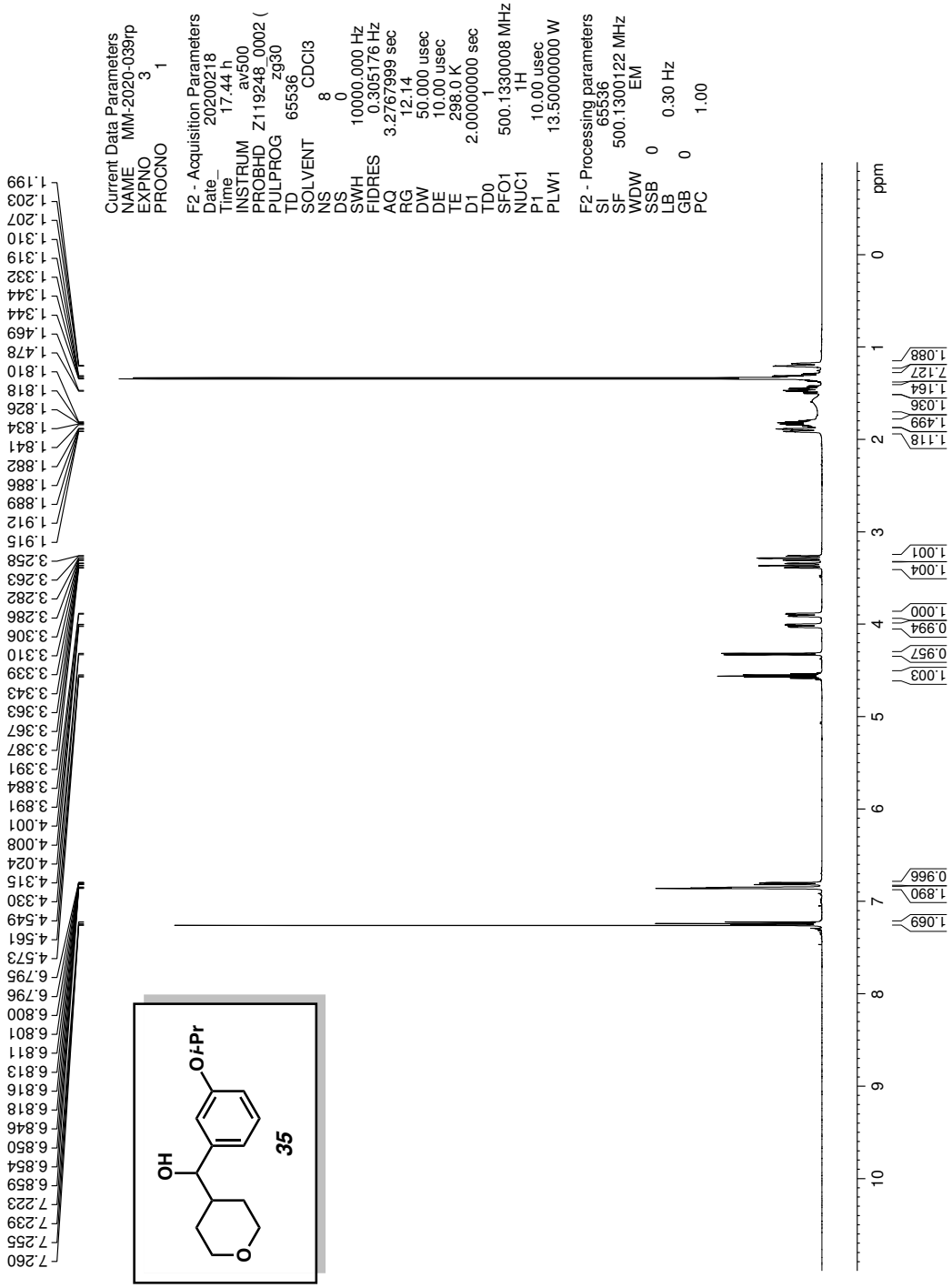

Current Data Parameters
NAME MM-2019-286p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191126
Time_ 15.00
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 161.3
DW 50.000 usec
DE 6.00 usec
TE 294.5 K
D1 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 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
Me
O
30
ppm

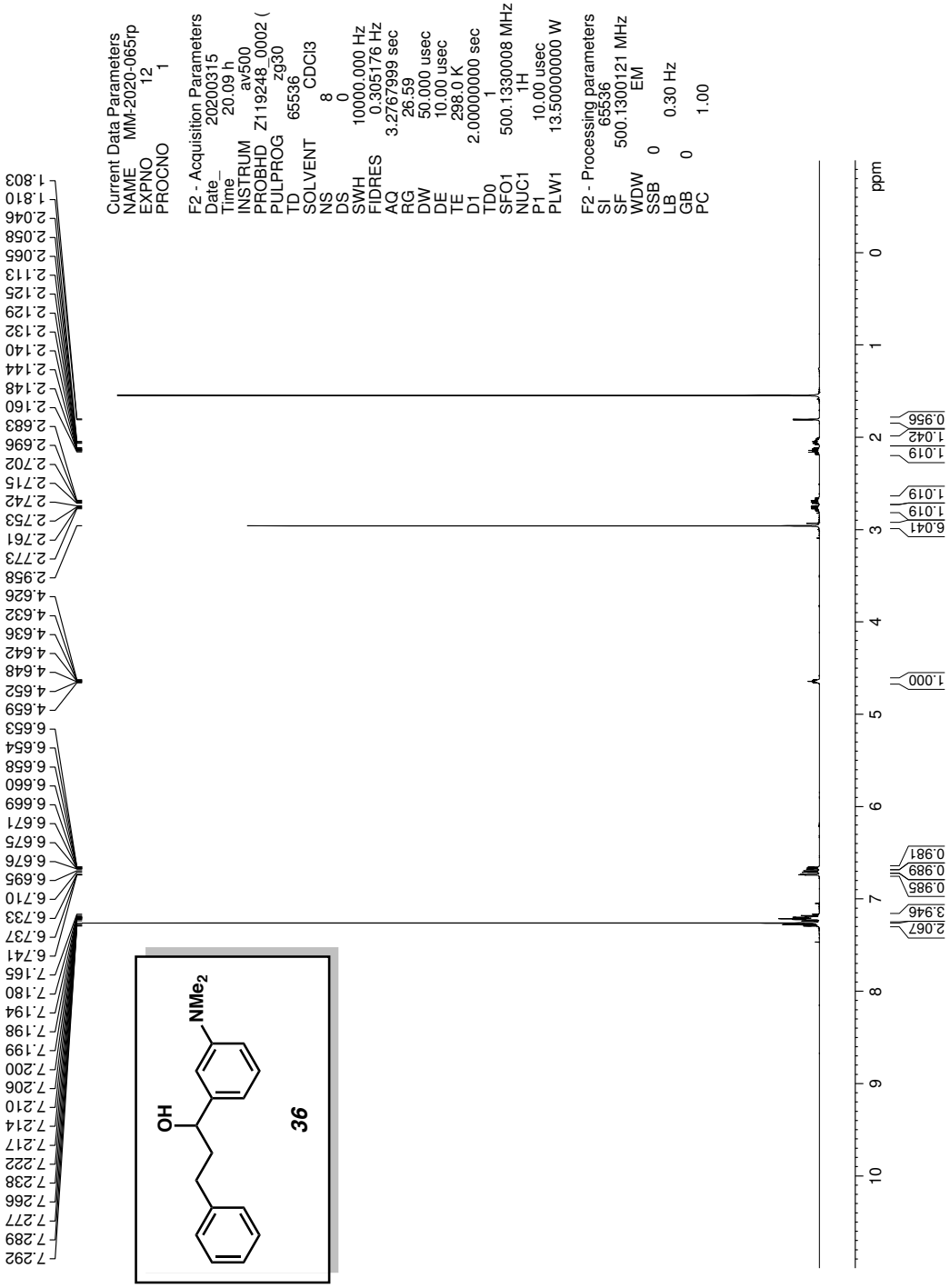

Current Data Parameters
NAME MM-2020-051p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200301
Time 14.13
INSTRUM av600
PROBHD 5 mm BB5
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6476543 sec
RG 362
DW 40.400 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 16.50 usec
PL1 -1.00 dB
PL1W 31.62277603 W
SFO1 600.1336008 MHz
F2 - Processing parameters
SI 65536
SF 600.1300289 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
SiMe3
O
31
ppm
8.479
1.970
1.197
1.231
2.016
1.036
1.060
1.047
1.017
1.042
1.000
1.955
1.942

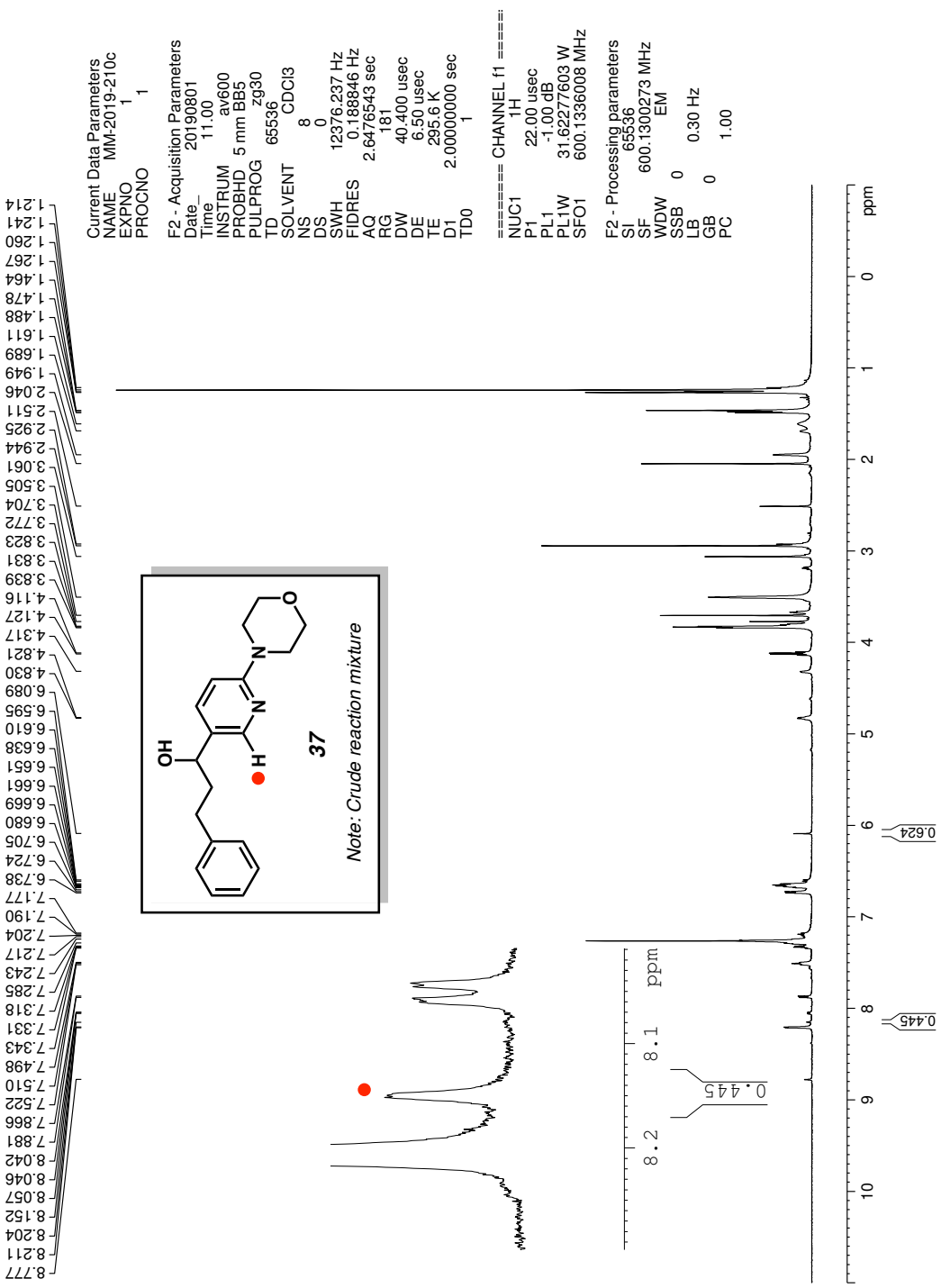

Current Data Parameters
NAME MM-2020-034p
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200216
Time 17.51 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 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
ppm
OH
CF3
N
Boc
32

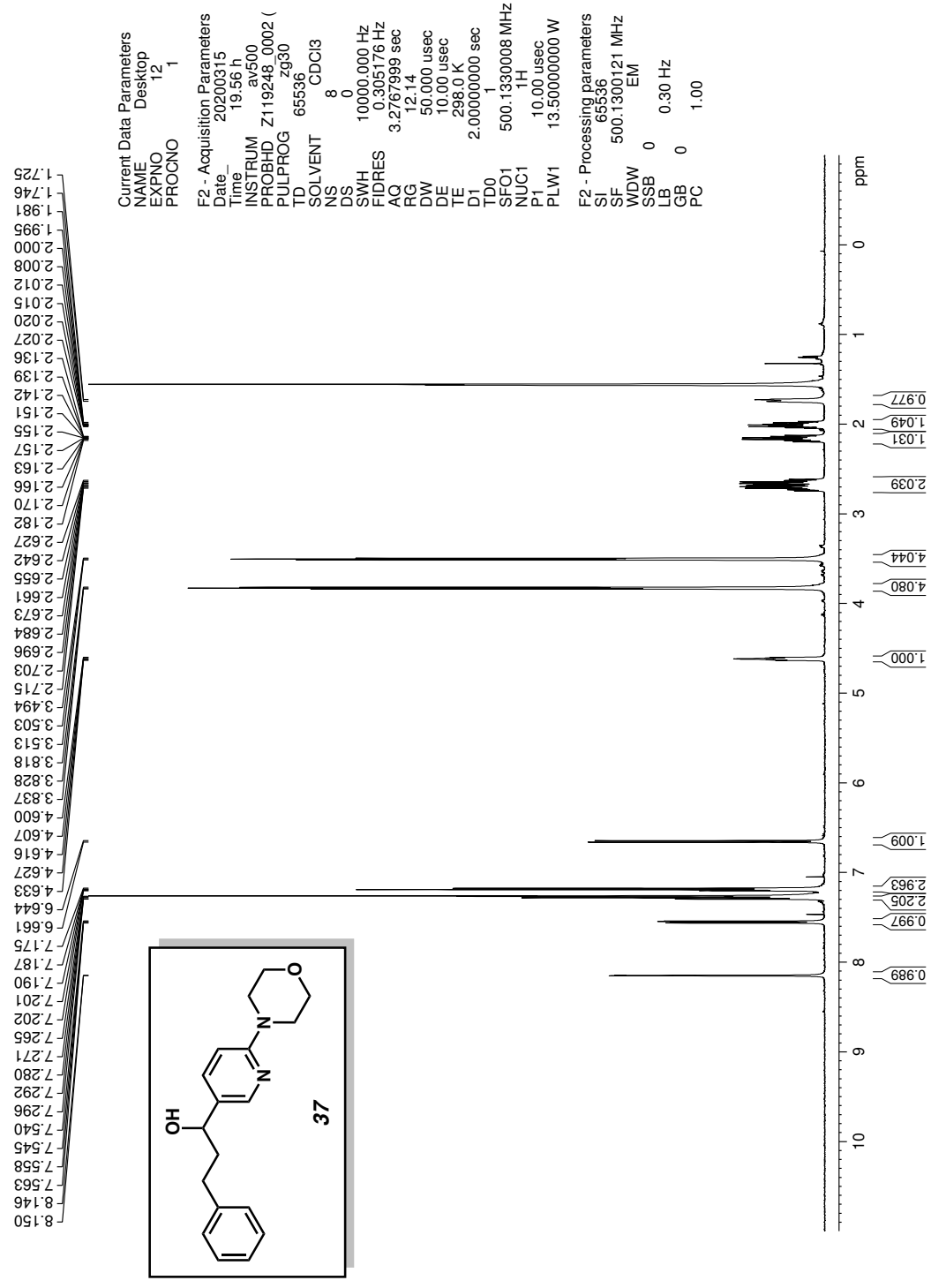

Current Data Parameters
NAME MM-2020-035p
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200216
Time 19.19 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 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
Boc
33
ppm
4.061
9.019
1.191
1.253
1.061
1.052
1.021
1.063
1.019
3.087
1.000
3.094

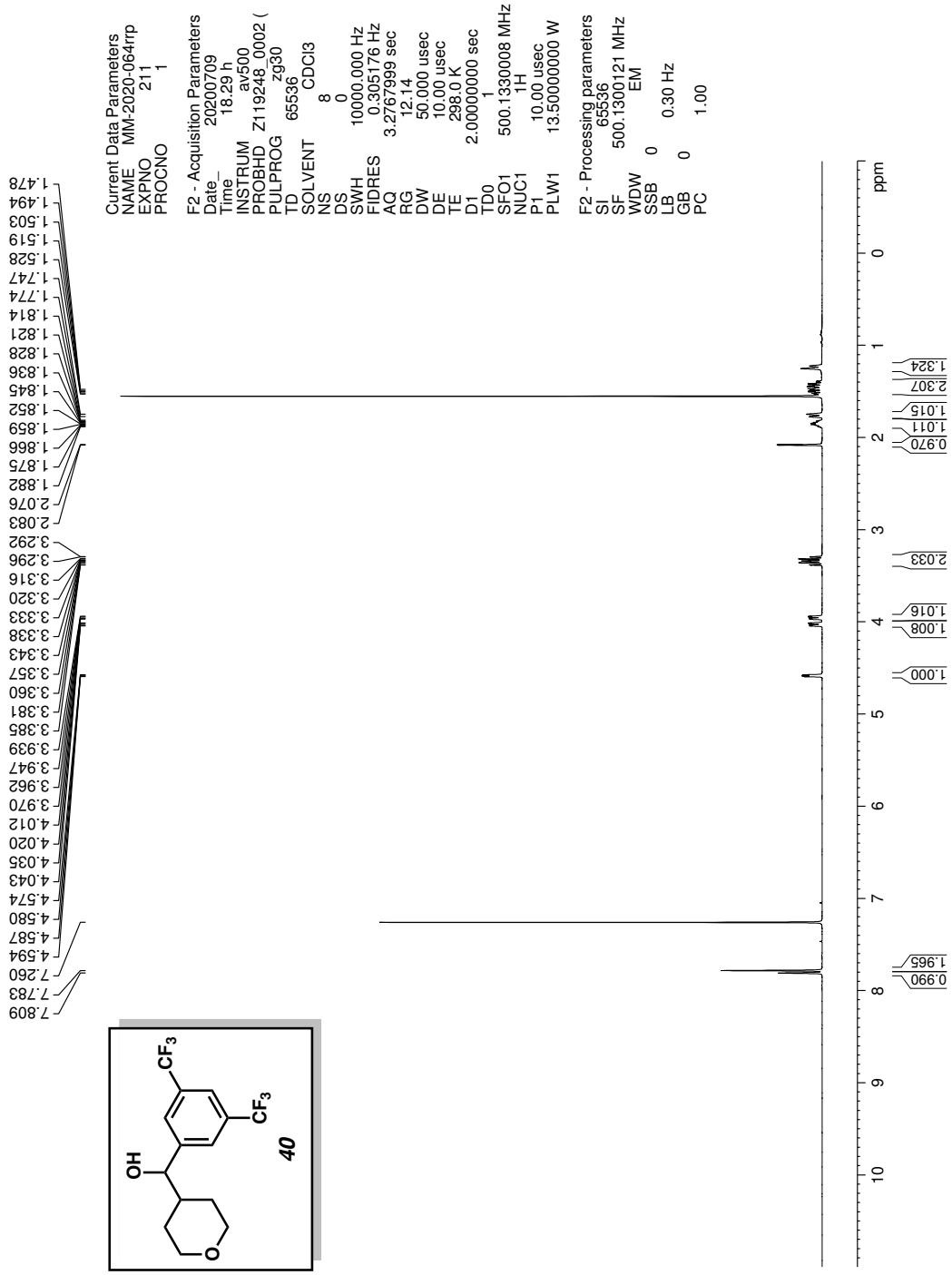

Current Data Parameters
NAME MM-2019-284rp
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200112
Time 13.12 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 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
OH
Ot-Bu
O
O
34
ppm
0.921
2.080
8.905
1.311
1.392
1.029
1.005
1.005
1.009
1.010
1.000
2.517
1.929

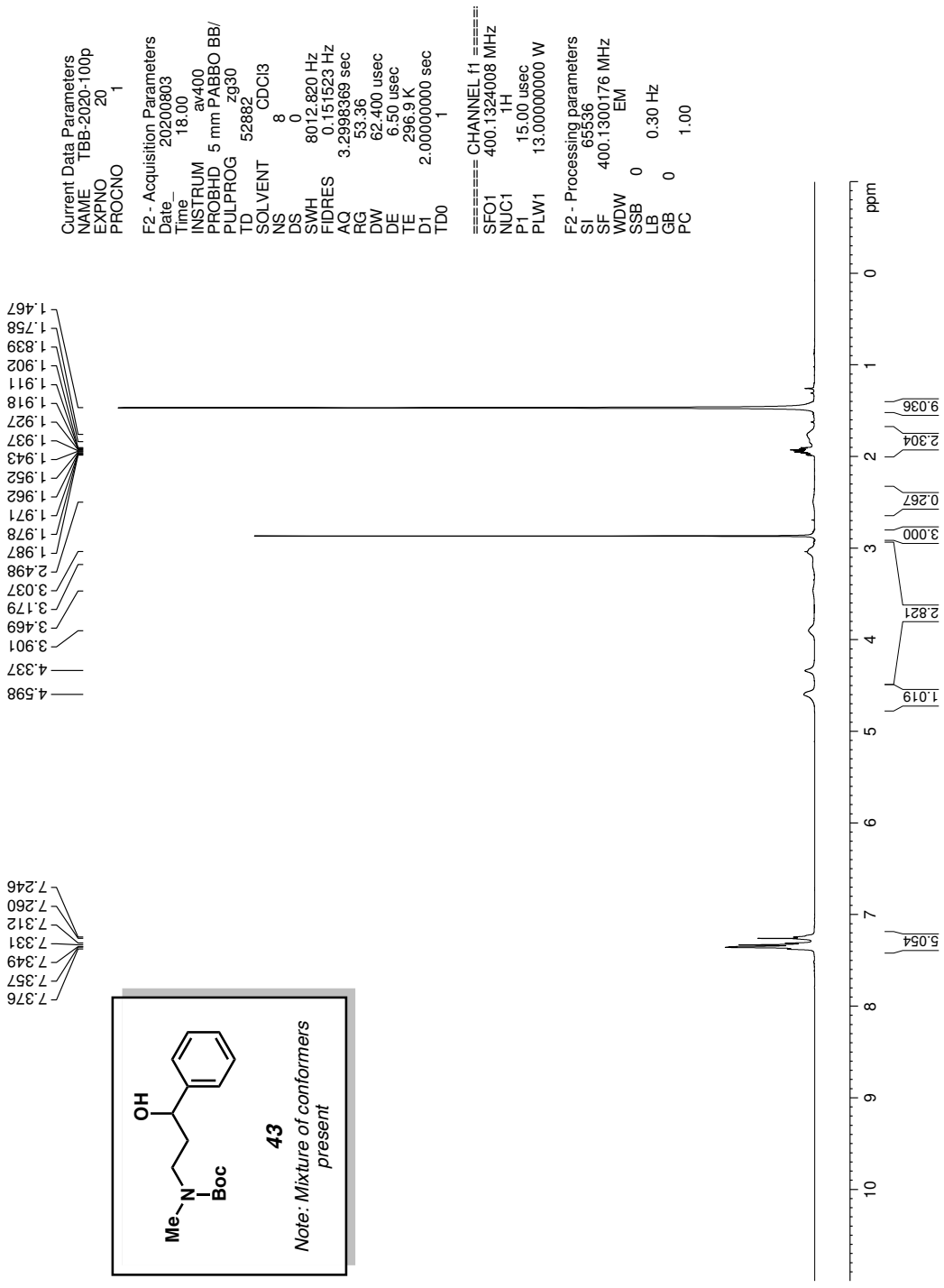

Current Data Parameters
NAME MM-2020-039rp
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200218
Time_ 17.44 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
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 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
O-i-Pr
O
35
ppm

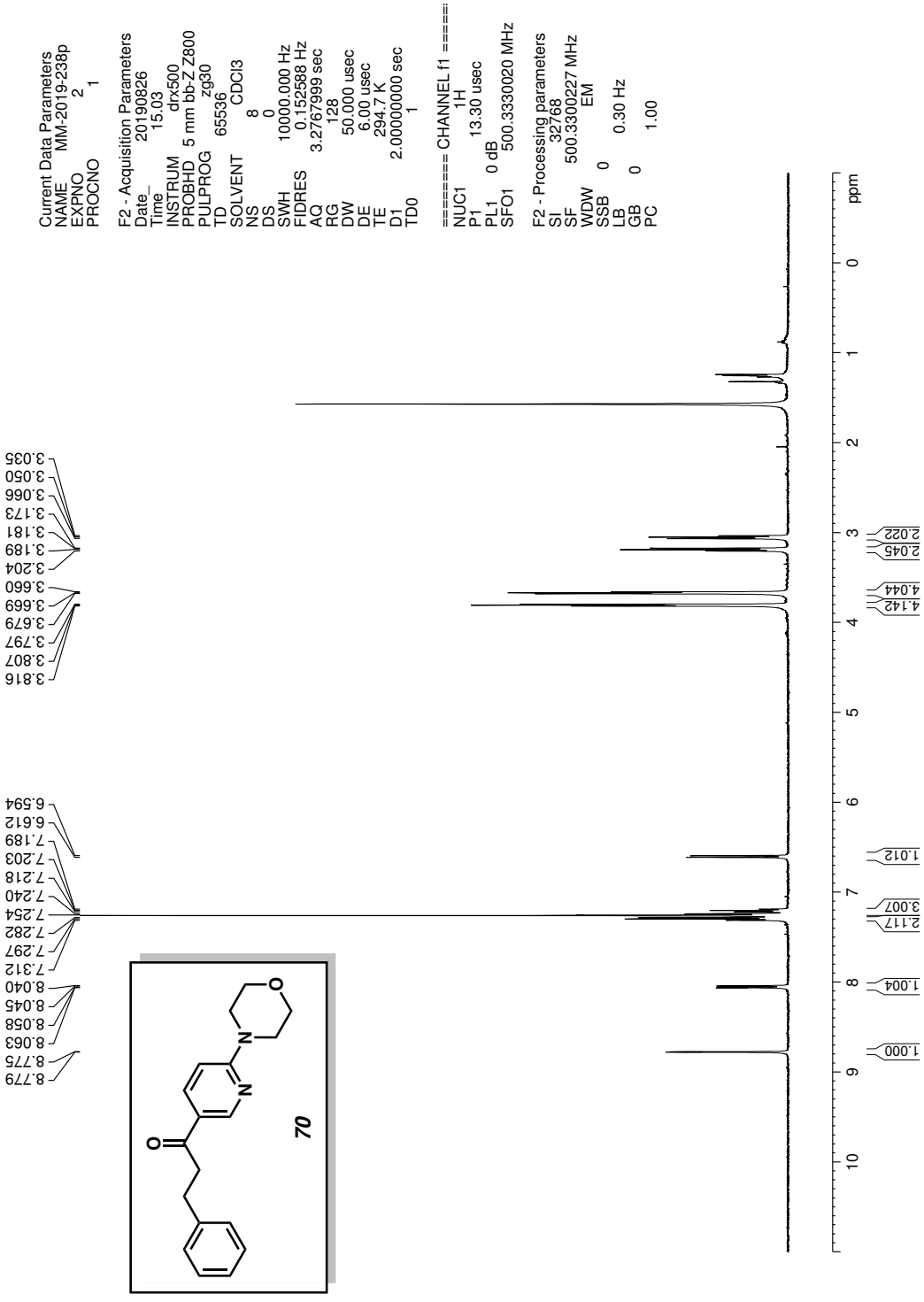

Current Data Parameters
NAME MM-2020-065rp
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200315
Time_ 20.09 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 26.59
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NMe2
OH
36
ppm
2.067
3.946
0.985
0.989
0.981
1.000
6.041
1.019
1.019
1.019
0.956
1.042
1.019

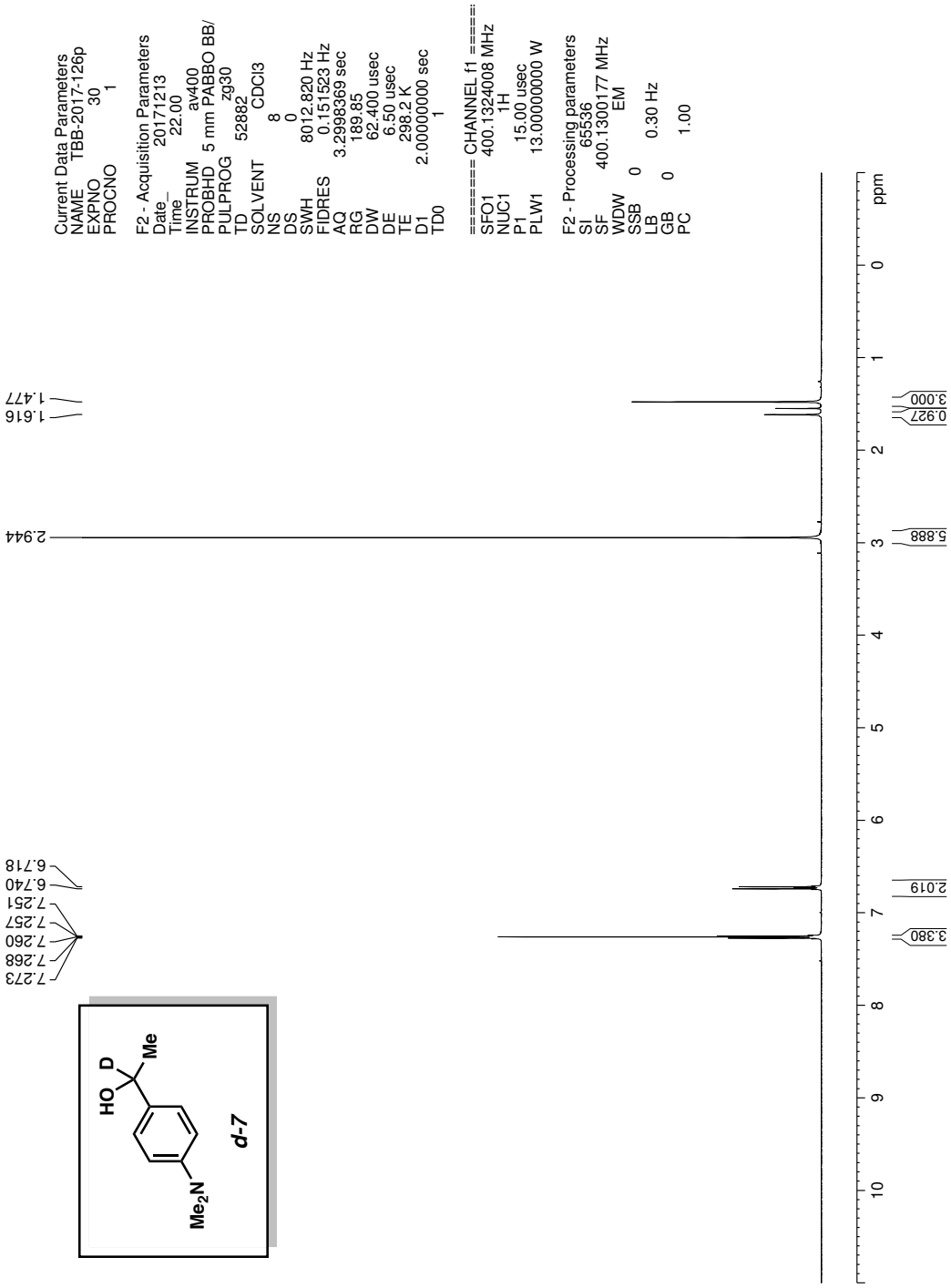

Current Data Parameters
NAME MM-2019-210c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190801
Time 11.00
INSTRUM av600
PROBHD 5 mm BB5
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6476543 sec
RG 181
DW 40.400 usec
DE 6.50 usec
TE 295.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 22.00 usec
PL1 -1.00 dB
PL1W 31.62277603 W
SFO1 600.1336008 MHz
F2 - Processing parameters
SI 65536
SF 600.1300273 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
H
N
N
O
37
Note: Crude reaction mixture
0.624
0.445
0.445
8.2
8.1
ppm
ppm


Current Data Parameters
NAME Desktop
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200315
Time 19.56 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 3.27677999 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
ppm
0.977
1.049
1.031
2.039
4.044
4.080
1.000
1.009
2.963
2.205
0.997
0.989
OH
37

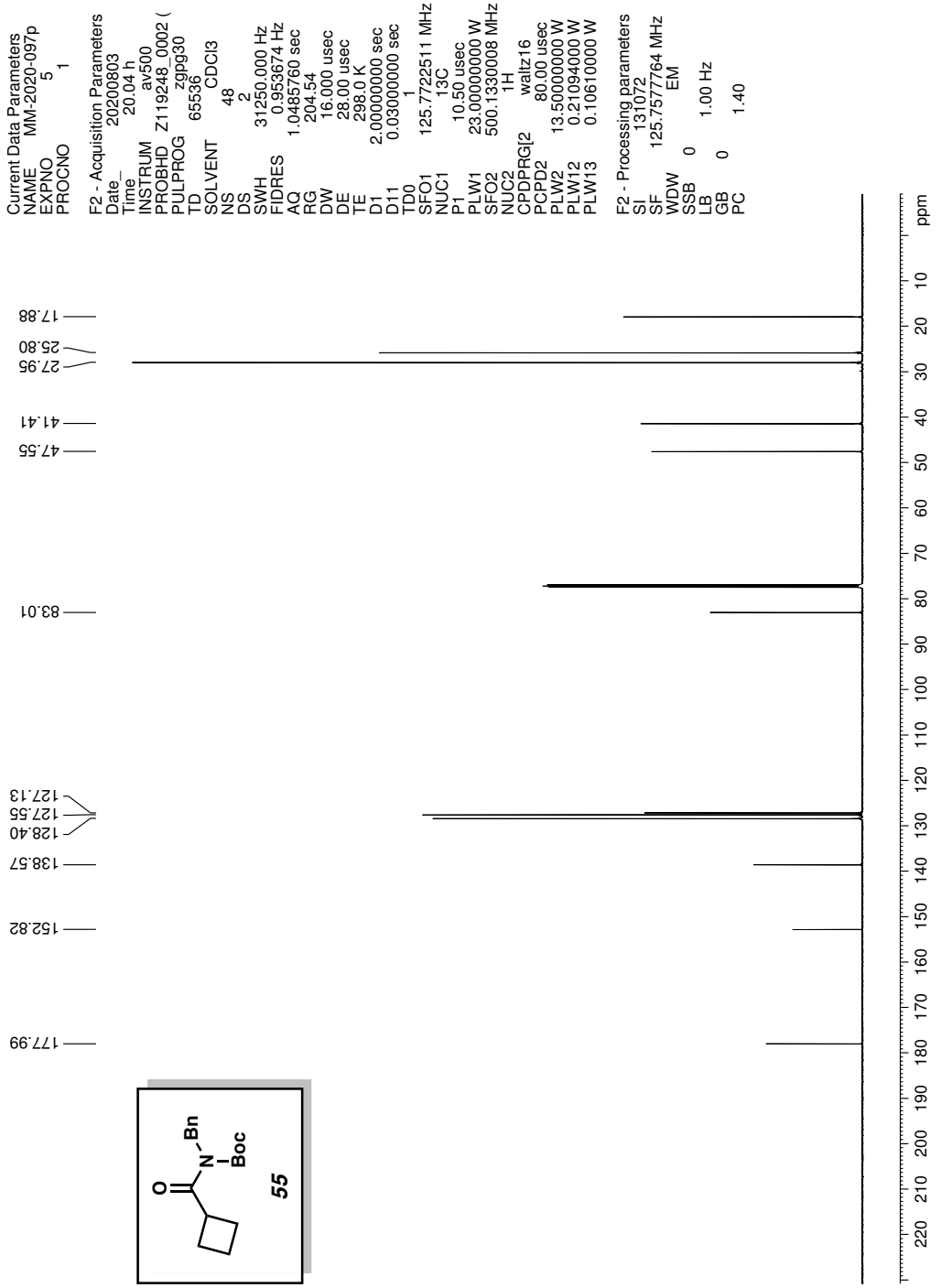

Current Data Parameters
NAME MM-2020-064rrp
EXPNO 211
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200709
Time 18.29 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
SFO1 500.1330008 MHz
NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.809
7.783
7.260
4.594
4.587
4.580
4.574
4.043
4.035
4.020
4.012
3.970
3.962
3.947
3.939
3.385
3.381
3.360
3.357
3.343
3.338
3.333
3.320
3.316
3.296
3.292
2.083
2.076
1.882
1.875
1.866
1.859
1.852
1.845
1.836
1.828
1.821
1.814
1.774
1.747
1.528
1.519
1.503
1.494
1.478
0.990
1.965
1.000
1.008
1.016
2.033
0.970
1.011
1.015
2.307
1.324
ppm
OH
CF3
CF3
O
40

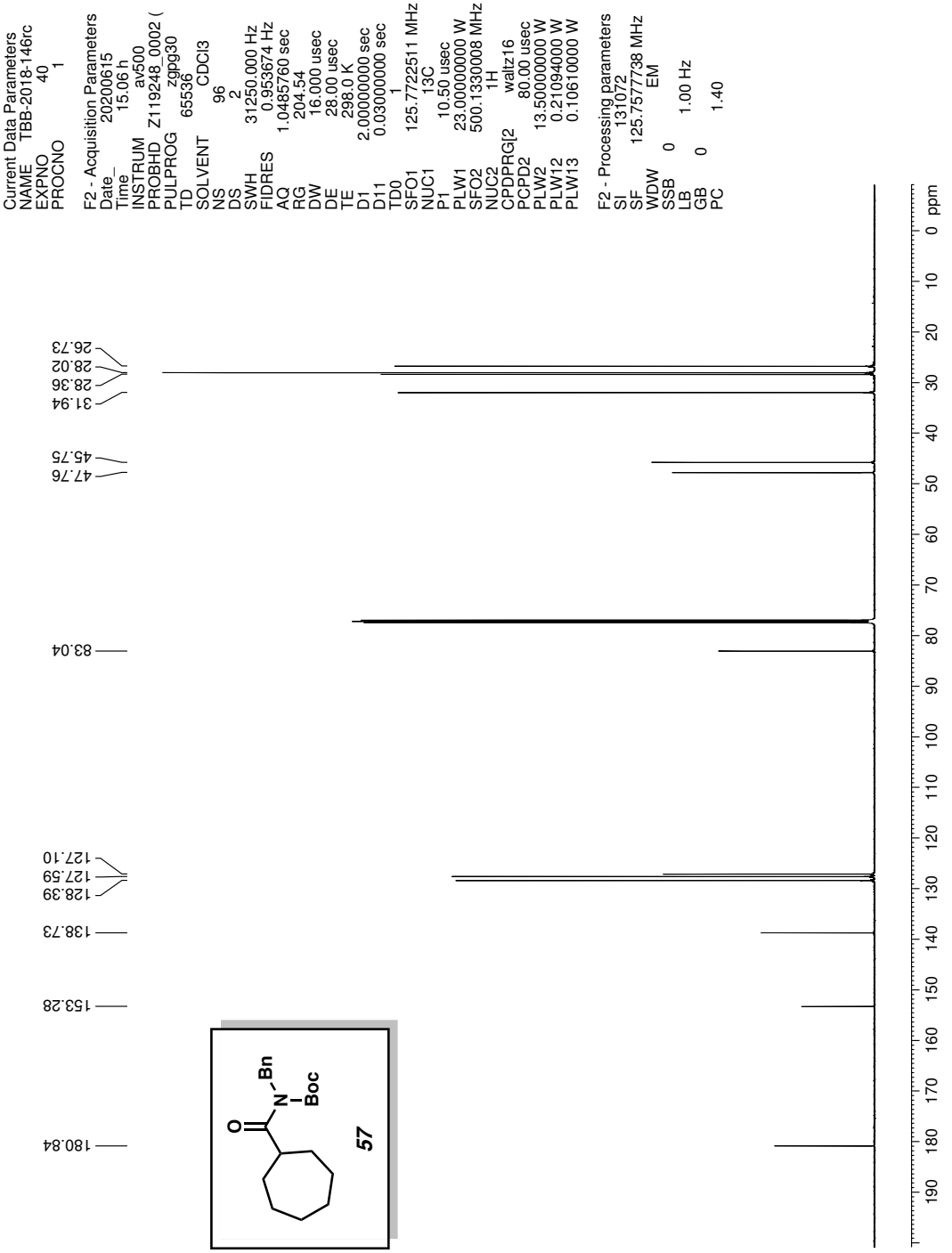

Current Data Parameters
NAME TBB-2020-100p
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200803
Time 18.00
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 53.36
DW 62.400 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 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
OH
Me
N
Boc
43
Note: Mixture of conformers present
ppm

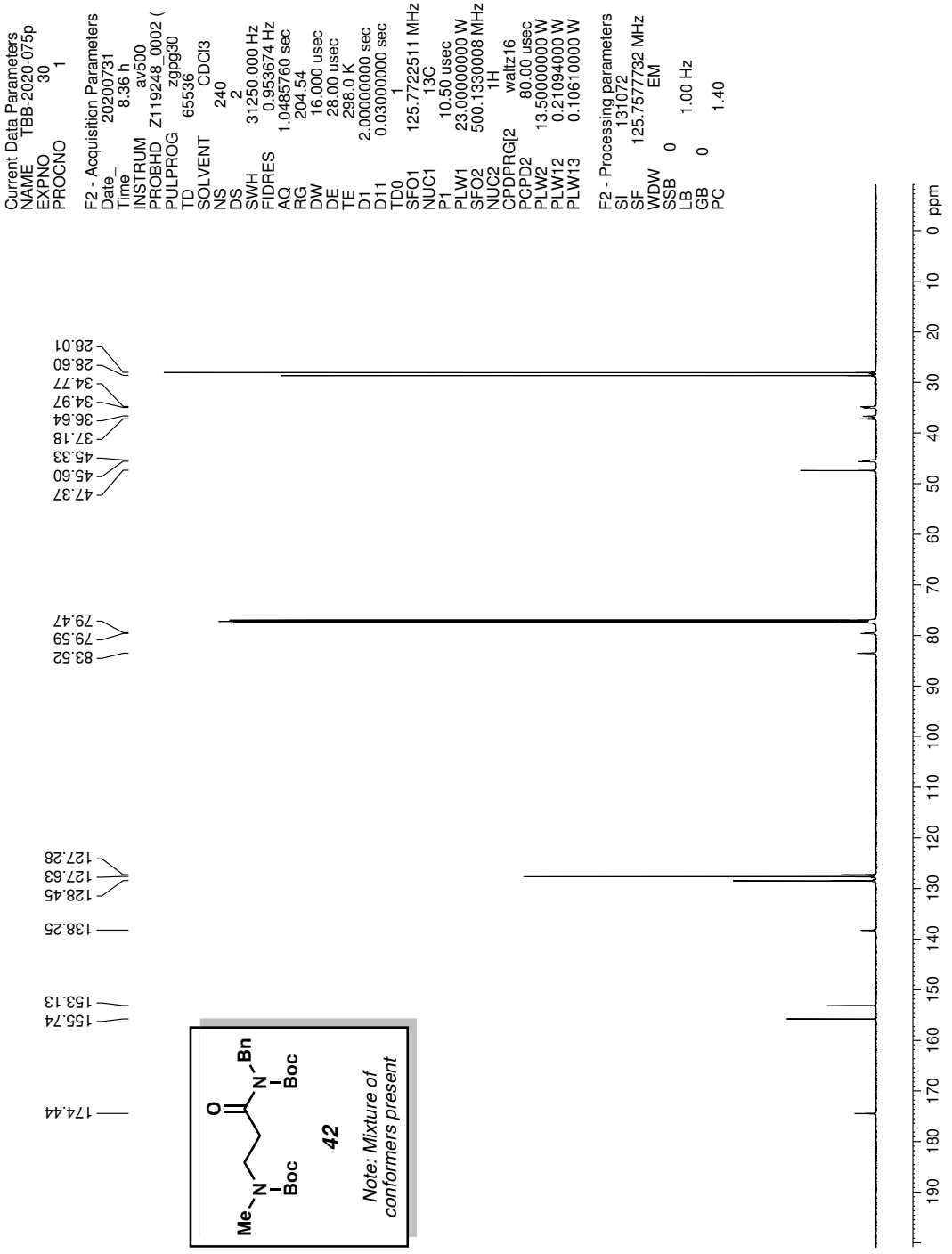

Current Data Parameters
NAME MM-2019-238p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190826
Time 15.03
INSTRUM drx500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 128
DW 50.000 usec
DE 6.00 usec
TE 294.7 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 0 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300227 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.779
8.775
8.063
8.058
8.045
8.040
7.312
7.297
7.282
7.254
7.240
7.218
7.203
7.189
6.612
6.594
3.816
3.807
3.797
3.679
3.669
3.660
3.204
3.189
3.181
3.173
3.066
3.050
3.035
1.000
1.004
2.117
3.007
1.012
4.142
4.044
2.045
2.022
O
N
O
70
ppm

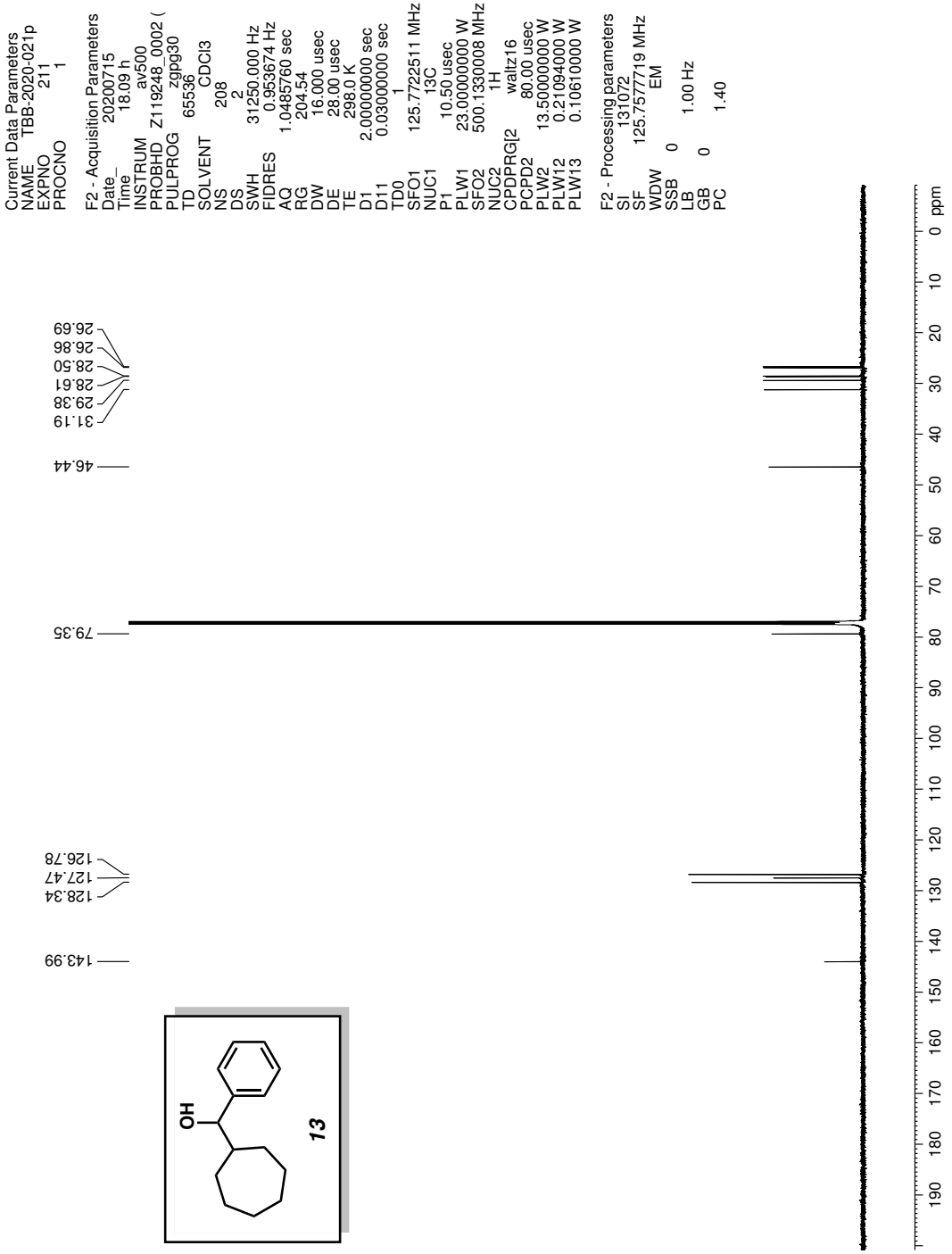

Current Data Parameters
NAME TBB-2017-126p
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171213
Time 22.00
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 52882
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.151523 Hz
AQ 3.2998369 sec
RG 189.85
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 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 400.1300177 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.477
1.616
2.944
6.718
6.740
7.251
7.257
7.260
7.268
7.273
HO D
Me
Me2N
d-7
3.000
0.927
5.888
2.019
3.380
ppm

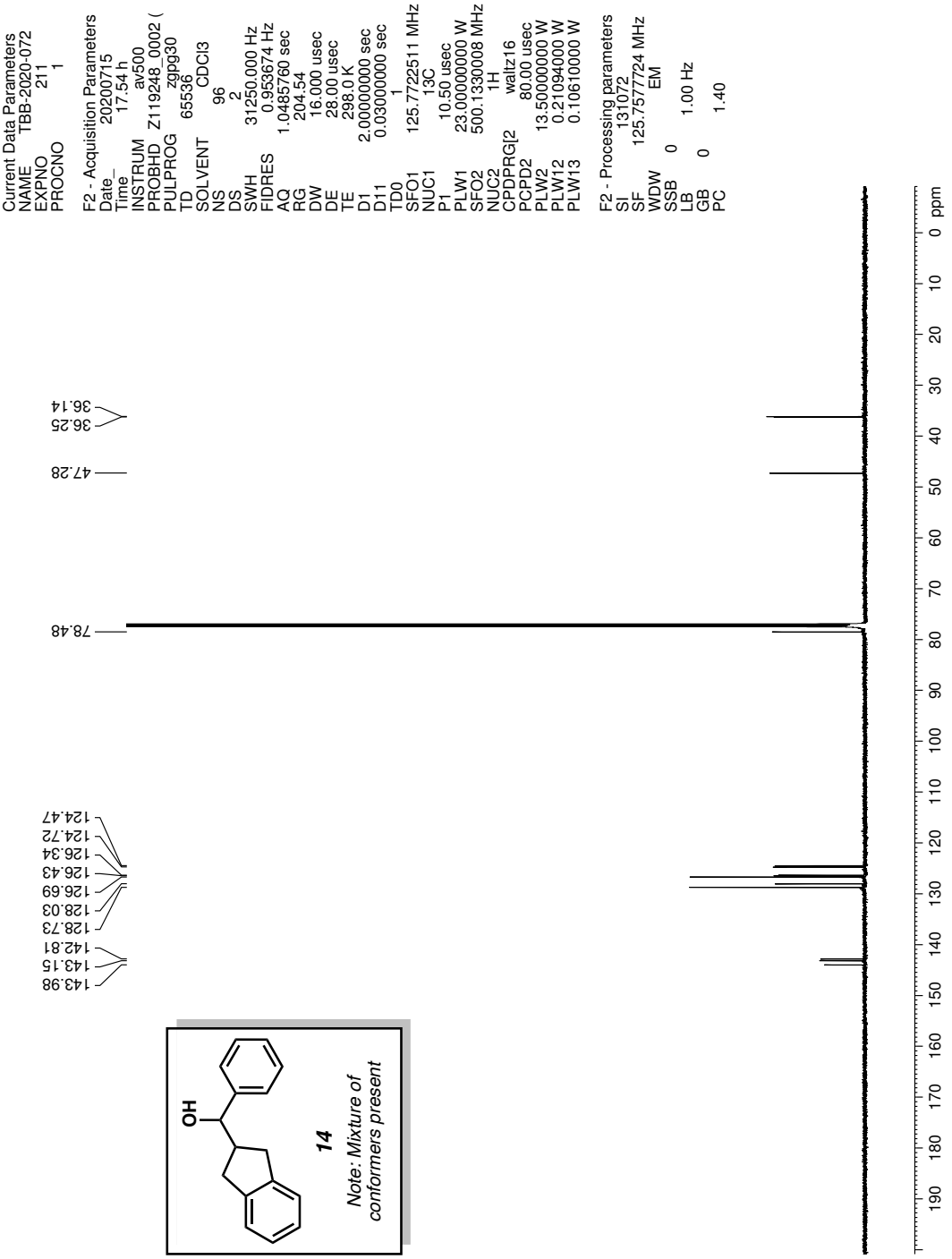
^{13}C NMR Spectra

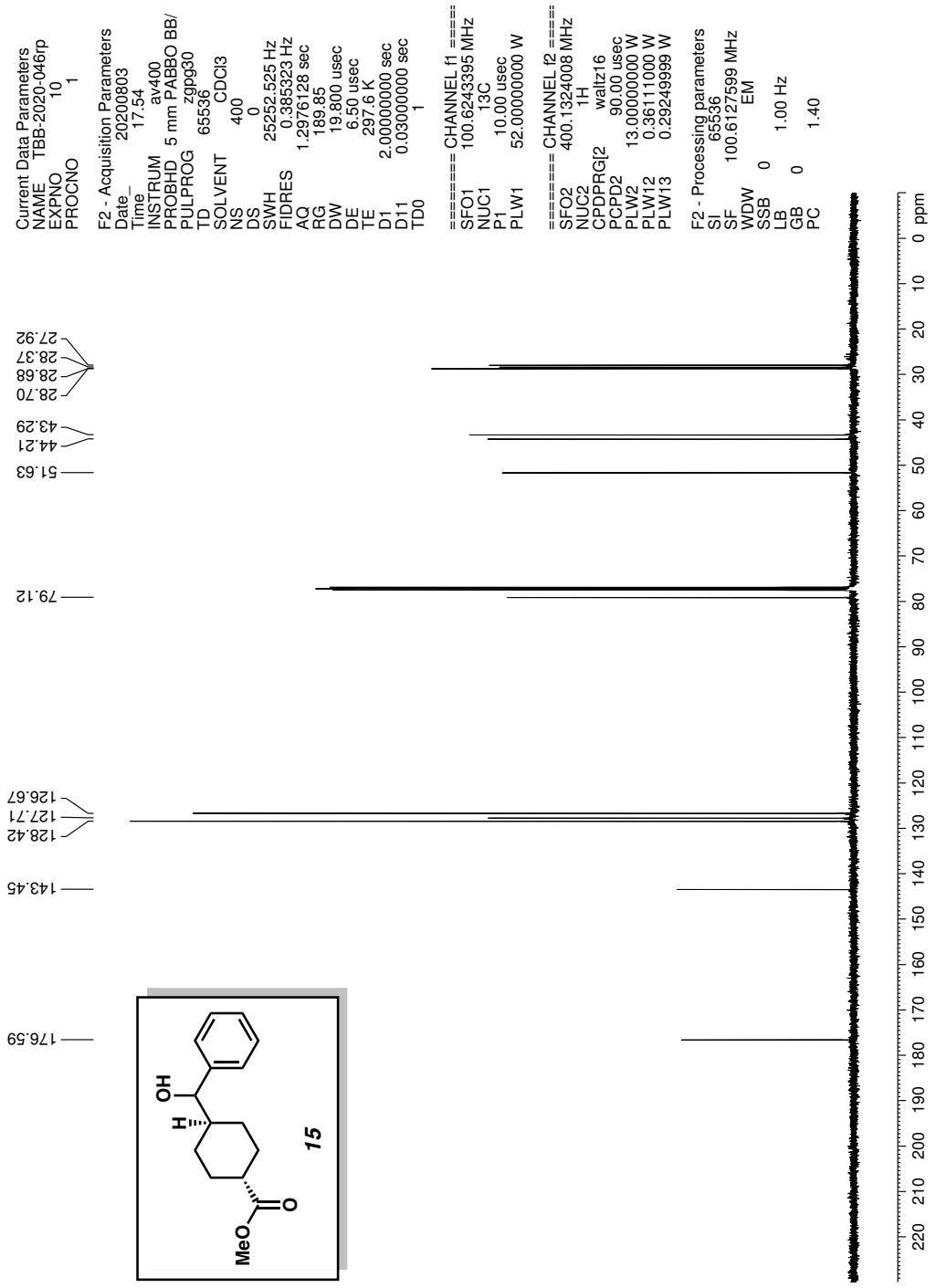

Current Data Parameters
NAME MM-2020-097p
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200803
Time_ 20.04 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 48
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 usec
TE 298.0 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 125.7577764 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
177.99
152.82
138.57
128.40
127.55
127.13
83.01
47.55
41.41
27.95
25.80
17.88
Bn
N
Boc
O
55
ppm

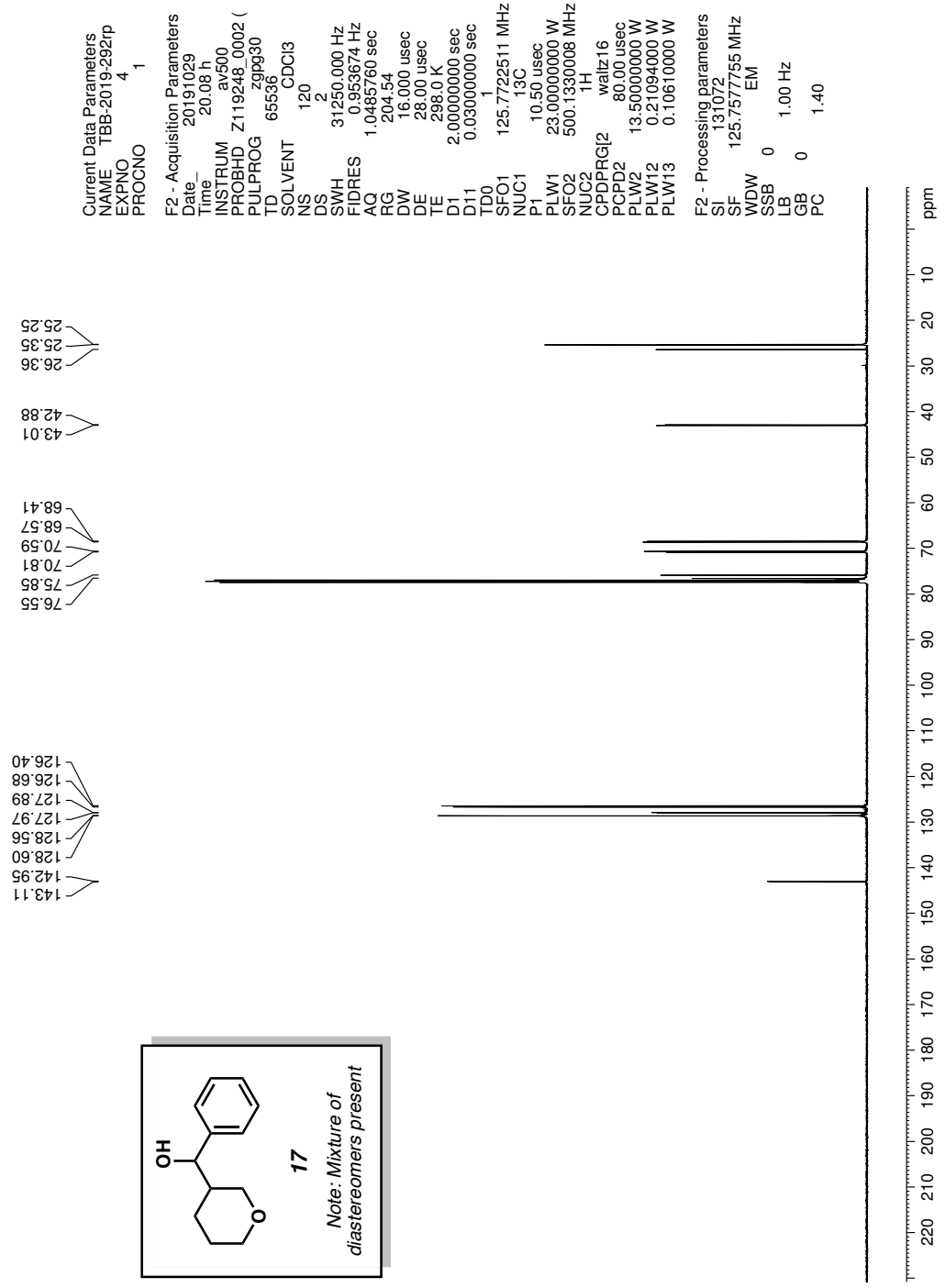

Current Data Parameters
NAME TBB-2018-146rc
EXPNO 40
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200615
Time 15.06 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 96
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 usec
TE 298.0 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
26.73
28.02
28.36
31.94
45.75
47.76
83.04
127.10
127.59
128.39
138.73
153.28
180.84
O
N
Bn
Boc
57
0 ppm

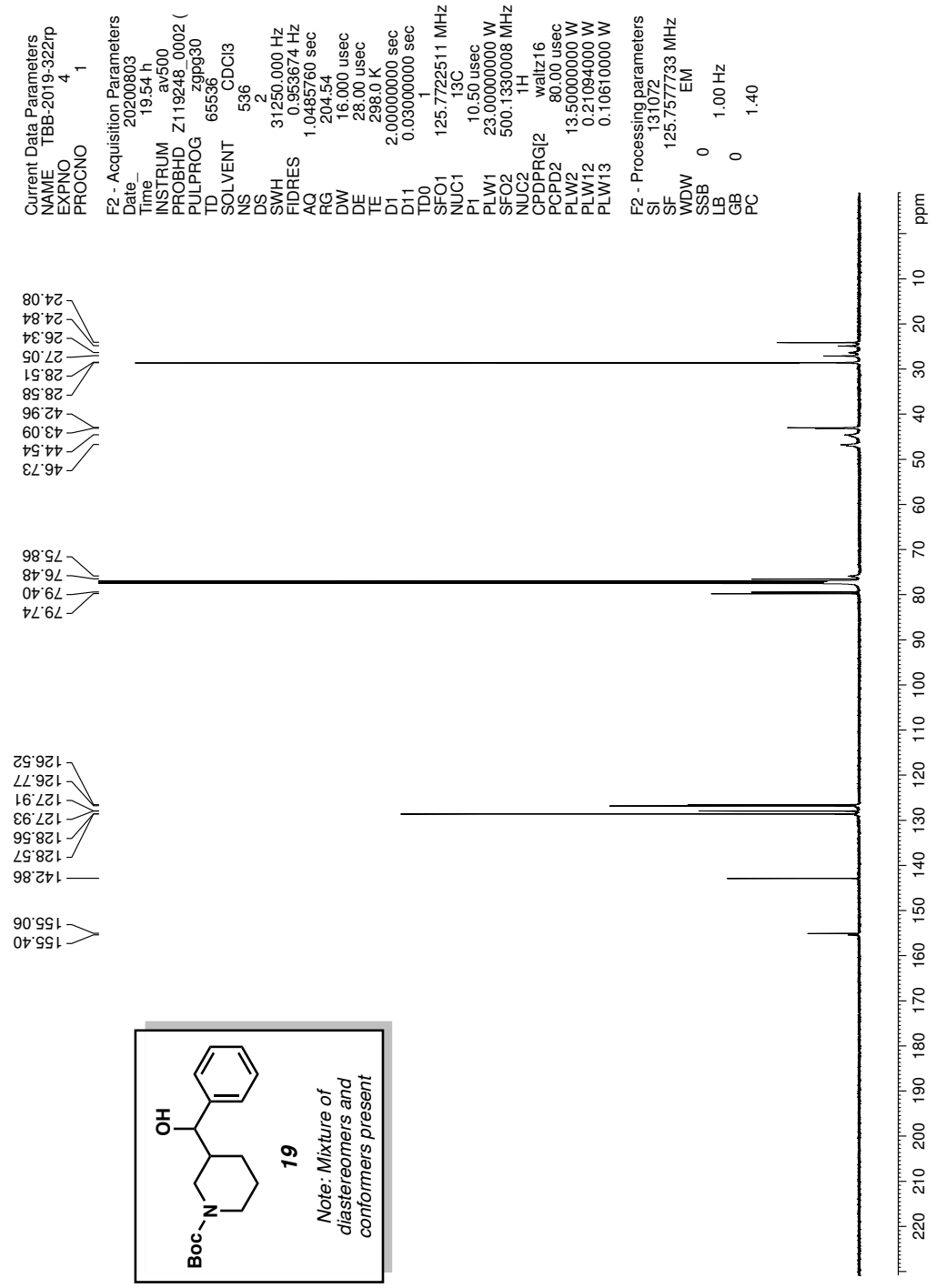

Current Data Parameters
NAME TBB-2020-075p
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200731
Time_ 8.36 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 240
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 usec
TE 298.0 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 125.7577732 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
28.01
28.60
34.77
34.97
36.64
37.18
45.33
45.60
47.37
79.47
79.59
83.52
127.28
127.63
128.45
138.25
153.13
155.74
174.44
Me
N
Boc
O
N
Bn
Boc
42
Note: Mixture of conformers present
0 ppm

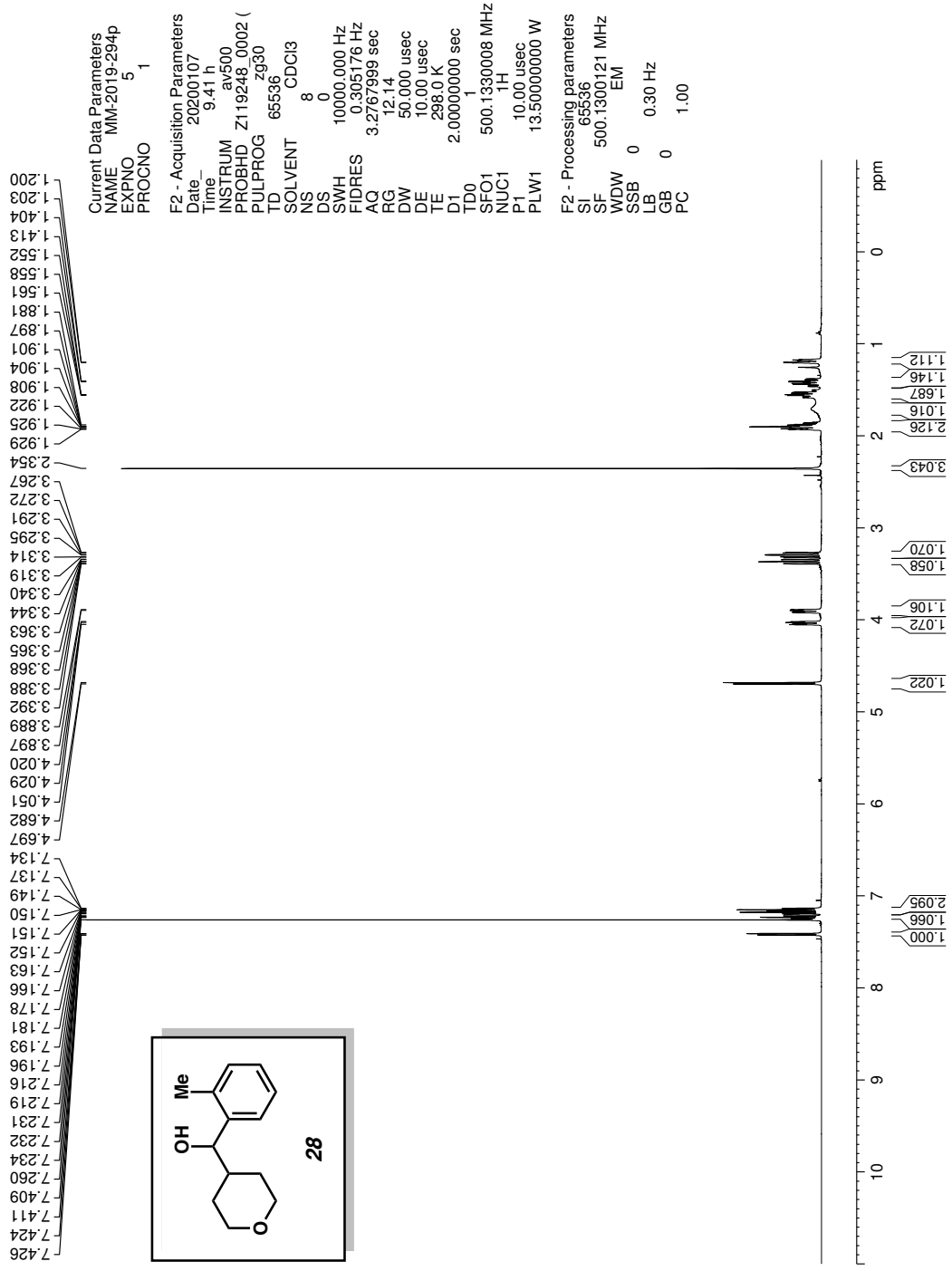

Current Data Parameters
NAME TBB-2020-021p
EXPNO 211
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200715
Time_ 18.09 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 208
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577719 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
143.99
128.34
127.47
126.78
79.35
46.44
31.19
29.38
28.61
28.50
26.86
26.69
OH
13
ppm

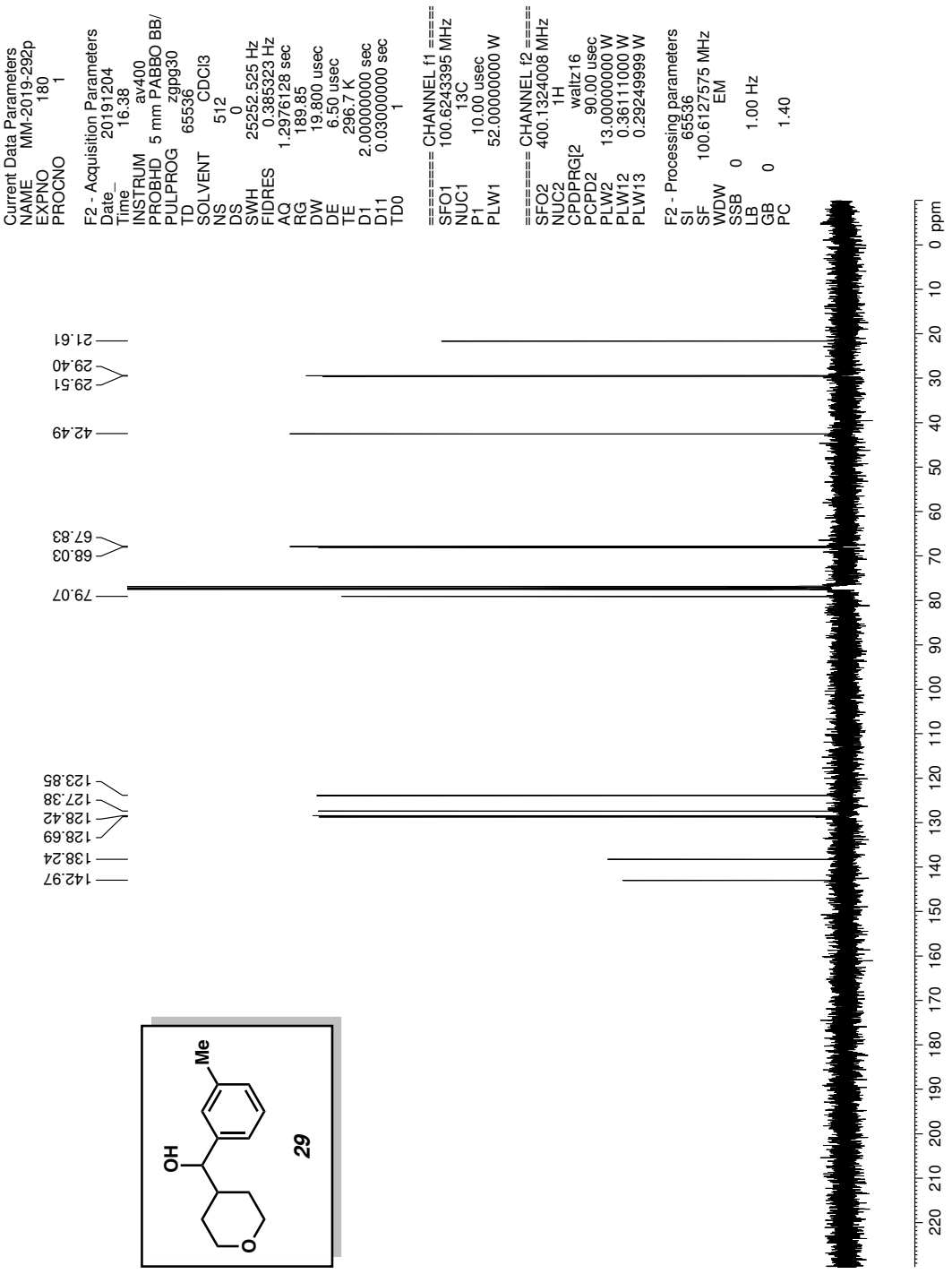

Current Data Parameters
NAME TBB-2020-072
EXPNO 211
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200715
Time_ 17.54 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 96
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577724 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
78.48
47.28
36.25
36.14
143.98
143.15
142.81
128.73
128.03
126.69
126.43
126.34
124.72
124.47
OH
14
Note: Mixture of conformers present
ppm

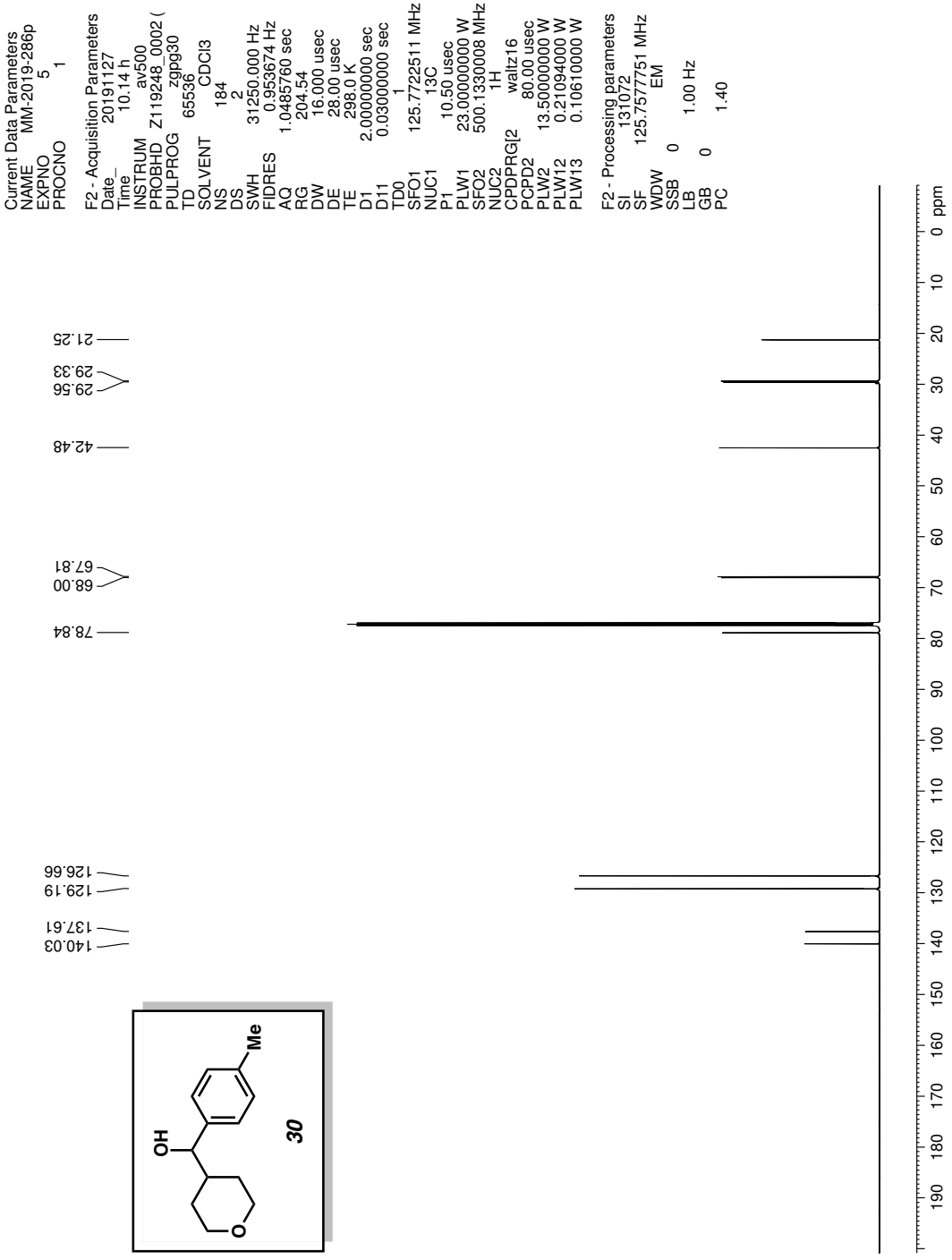

Current Data Parameters
NAME TBB-2020-046rp
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200803
Time 17.54
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976128 sec
RG 189.85
DW 19.800 usec
DE 6.50 usec
TE 297.6 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 100.6127599 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
176.59
143.45
128.42
127.71
126.67
79.12
51.63
44.21
43.29
28.70
28.68
28.37
27.92
OH
H
O
MeO
15
0 ppm

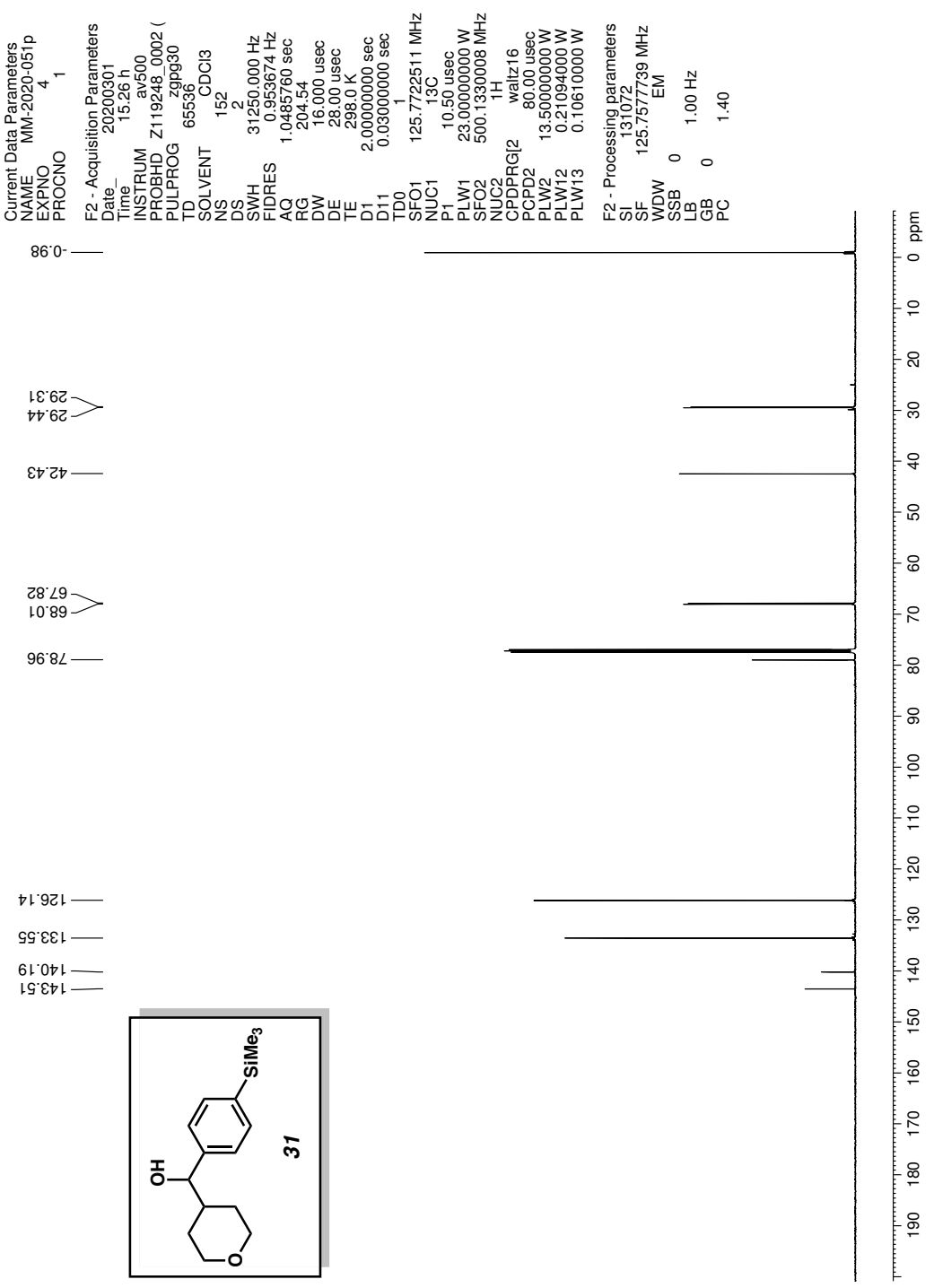

Current Data Parameters
NAME TBB-2019-292rp
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191029
Time 20.08 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 120
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 usec
TE 298.0 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
CPDPRGJ2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577755 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
143.11
142.95
128.60
128.56
127.97
127.89
126.68
126.40
76.55
75.85
70.81
70.59
68.57
68.41
43.01
42.88
26.36
25.35
25.25
OH
O
17
Note: Mixture of diastereomers present
ppm

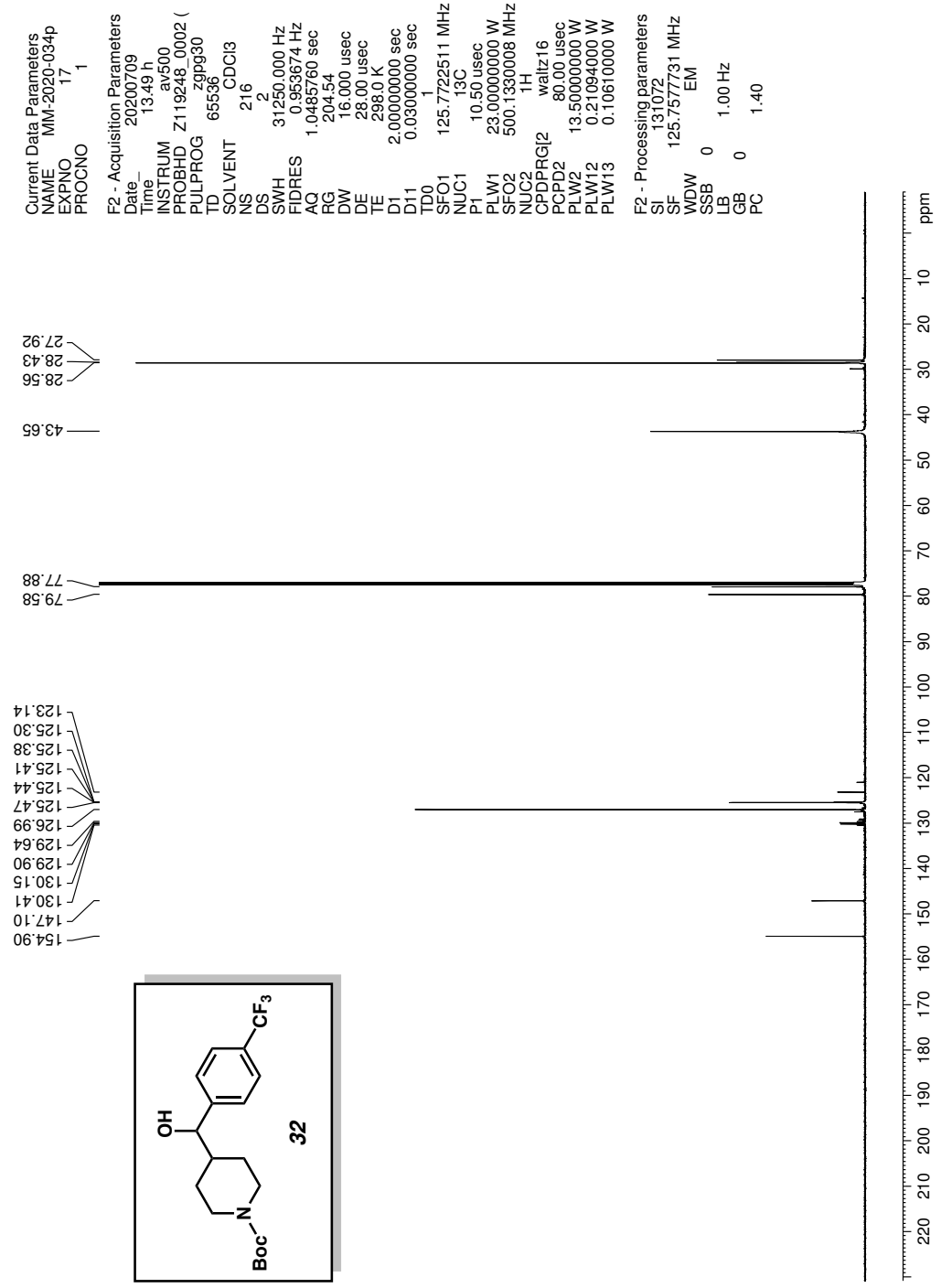

Current Data Parameters
NAME TBB-2019-322rp
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200803
Time_ 19.54 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 536
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 usec
TE 298.0 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 125.7577733 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
155.40
155.06
142.86
128.57
128.56
127.93
127.91
126.77
126.52
79.74
79.40
76.48
75.86
46.73
44.54
43.09
42.96
28.58
28.51
27.05
26.34
24.84
24.08
ppm
OH
Boc
N
19
Note: Mixture of diastereomers and conformers present

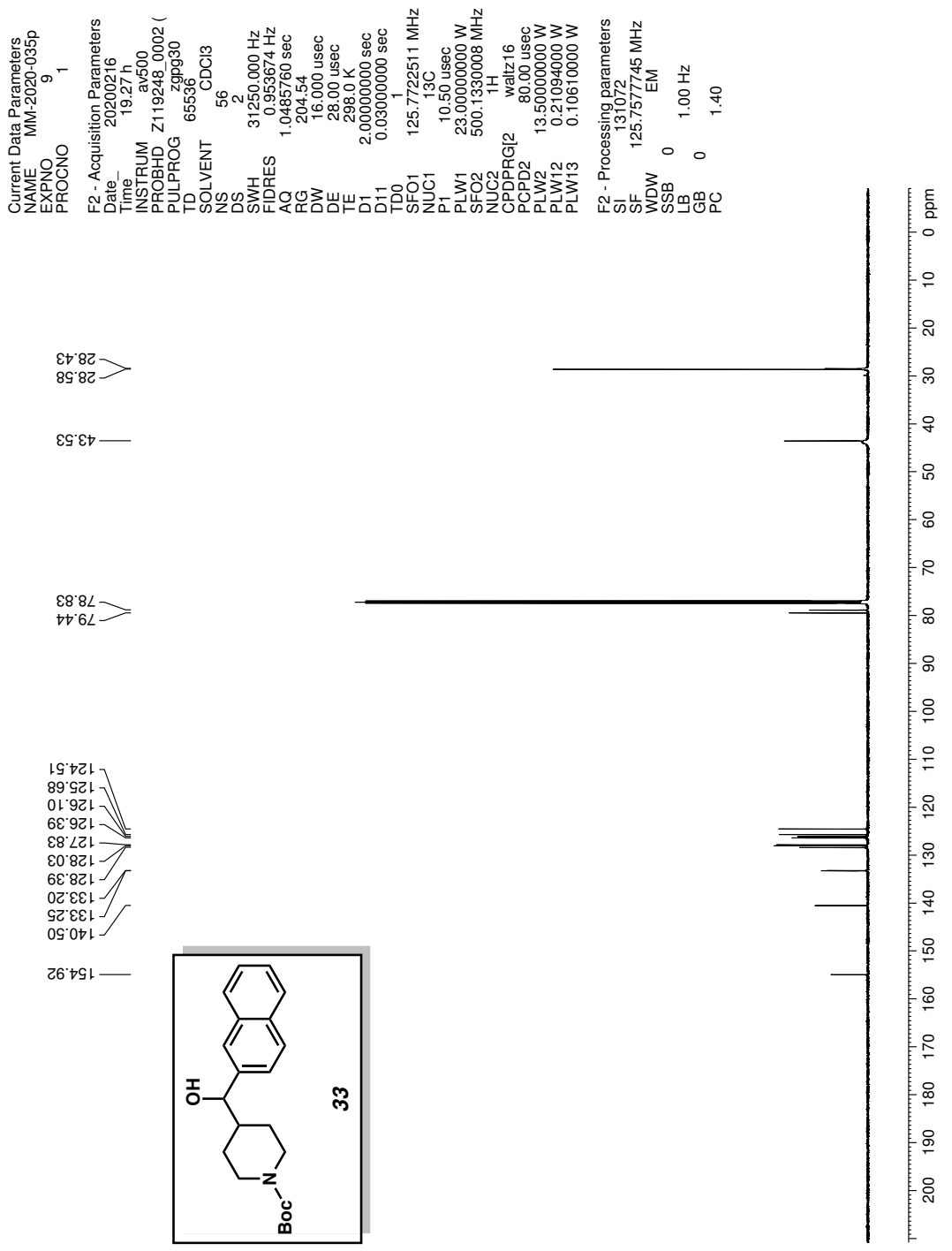

Current Data Parameters
NAME MM-2019-294p
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200107
Time 9.41 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 12.14
DW 50.000 usec
DE 10.00 usec
TE 298.0 K
D1 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
OH
Me
O
28
ppm

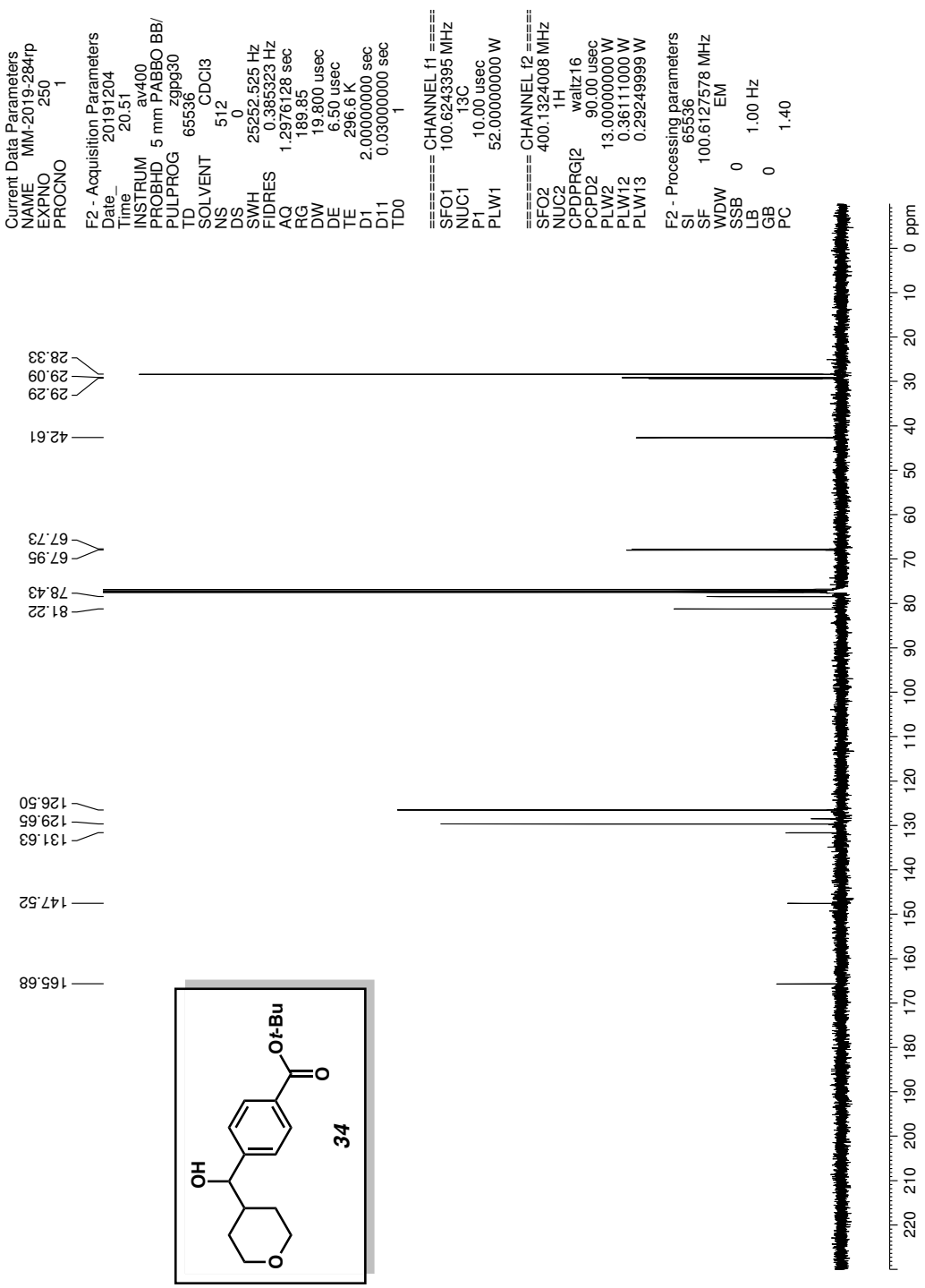

Current Data Parameters
NAME MM-2019-292p
EXPNO 180
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191204
Time 16.38
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976128 sec
RG 189.85
DW 19.800 usec
DE 6.50 usec
TE 296.7 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 100.6127575 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.61
29.40
29.51
42.49
67.83
68.03
79.07
123.85
127.38
128.42
128.69
138.24
142.97
Me
OH
O
29
0 ppm

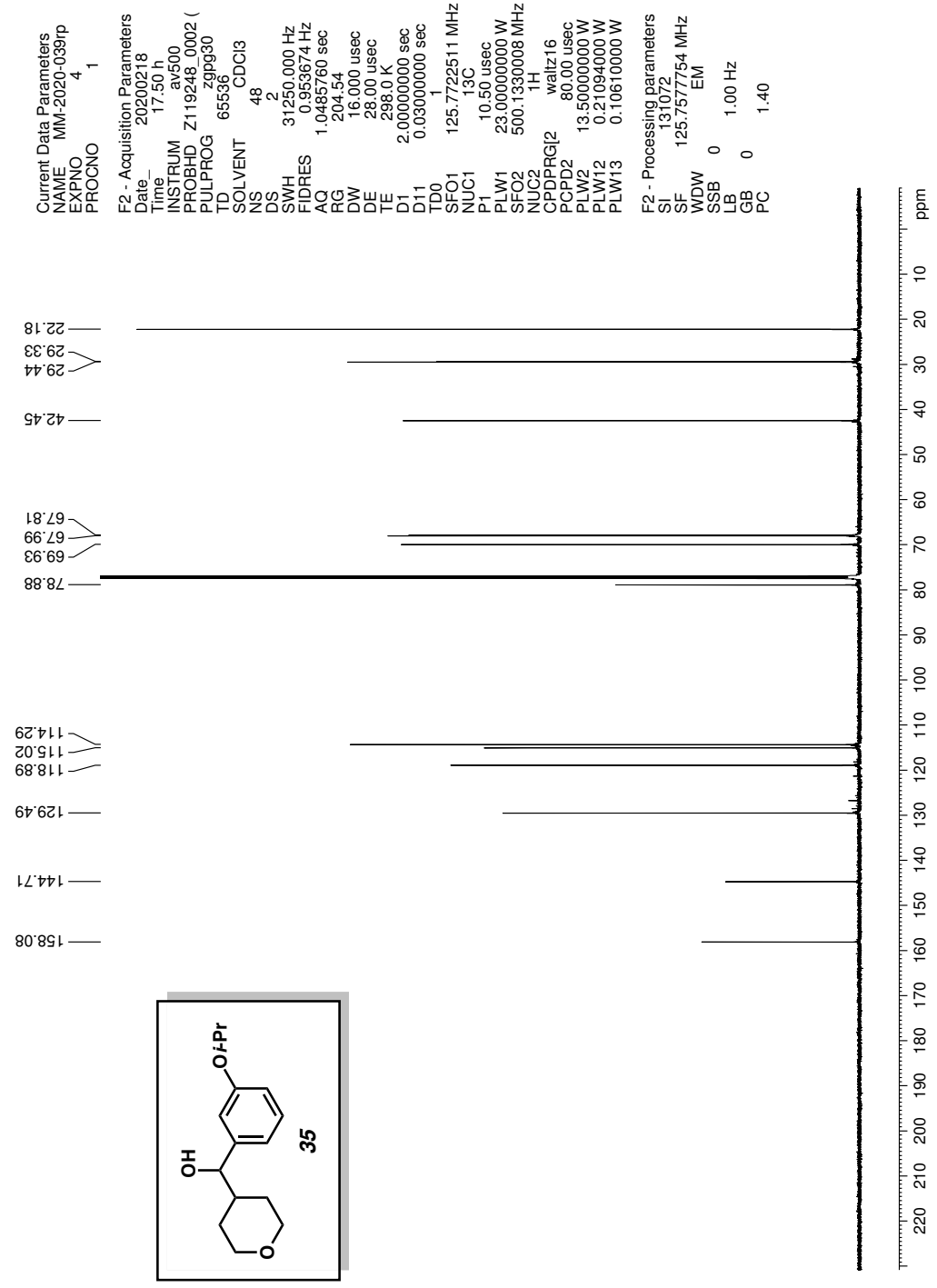

Current Data Parameters
NAME MM-2019-286p
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191127
Time 10.14 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 184
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 usec
TE 298.0 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 125.7577751 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.25
29.56
29.33
42.48
67.81
68.00
78.84
126.66
129.19
137.61
140.03
OH
Me
O
30
0 ppm

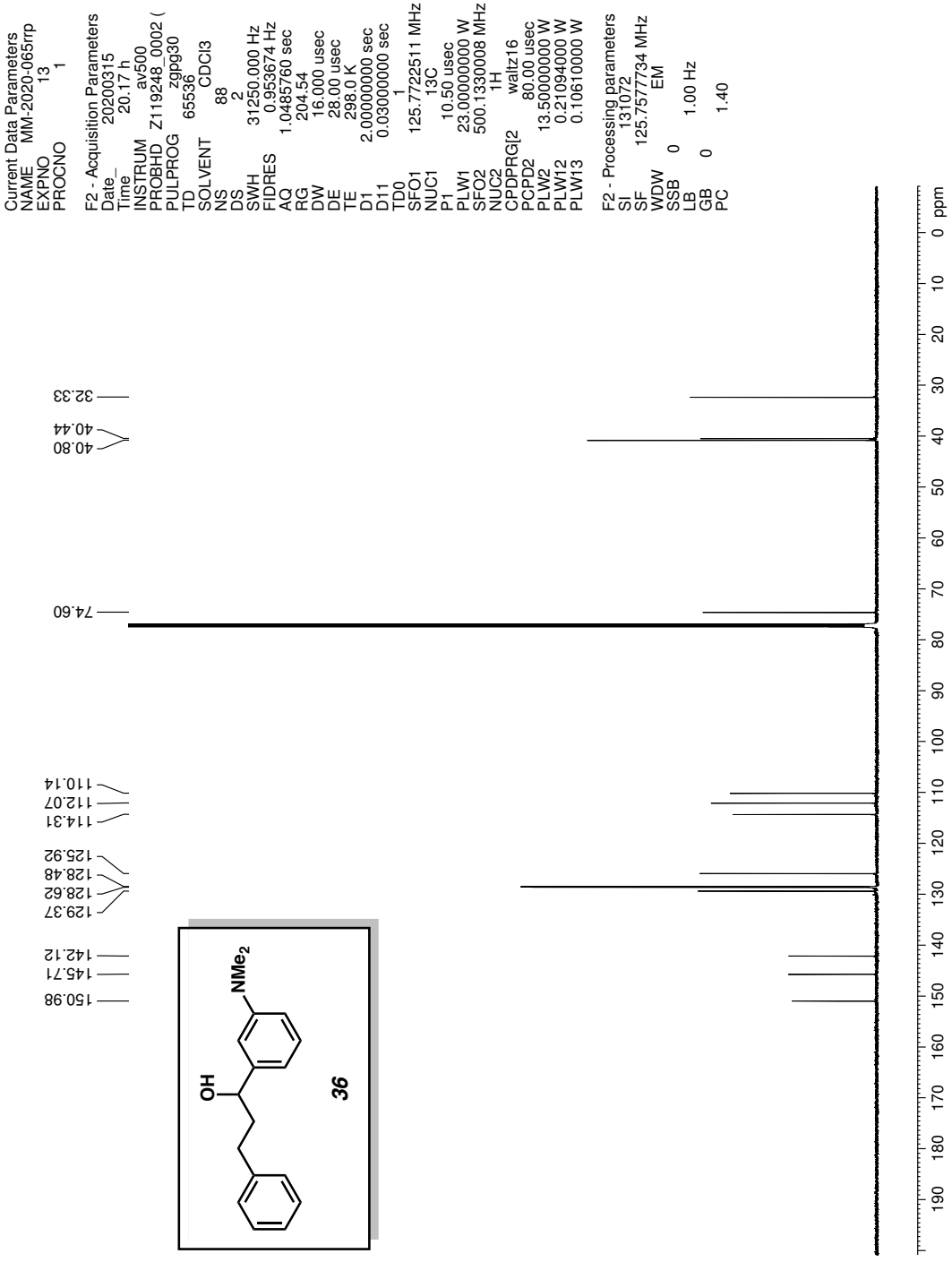

Current Data Parameters
NAME MM-2020-051p
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200301
Time 15.26 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 152
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 usec
TE 298.0 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 125.7577739 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
-0.98
29.31
29.44
42.43
67.82
68.01
78.96
126.14
133.55
140.19
143.51
OH
SiMe3
O
31
ppm

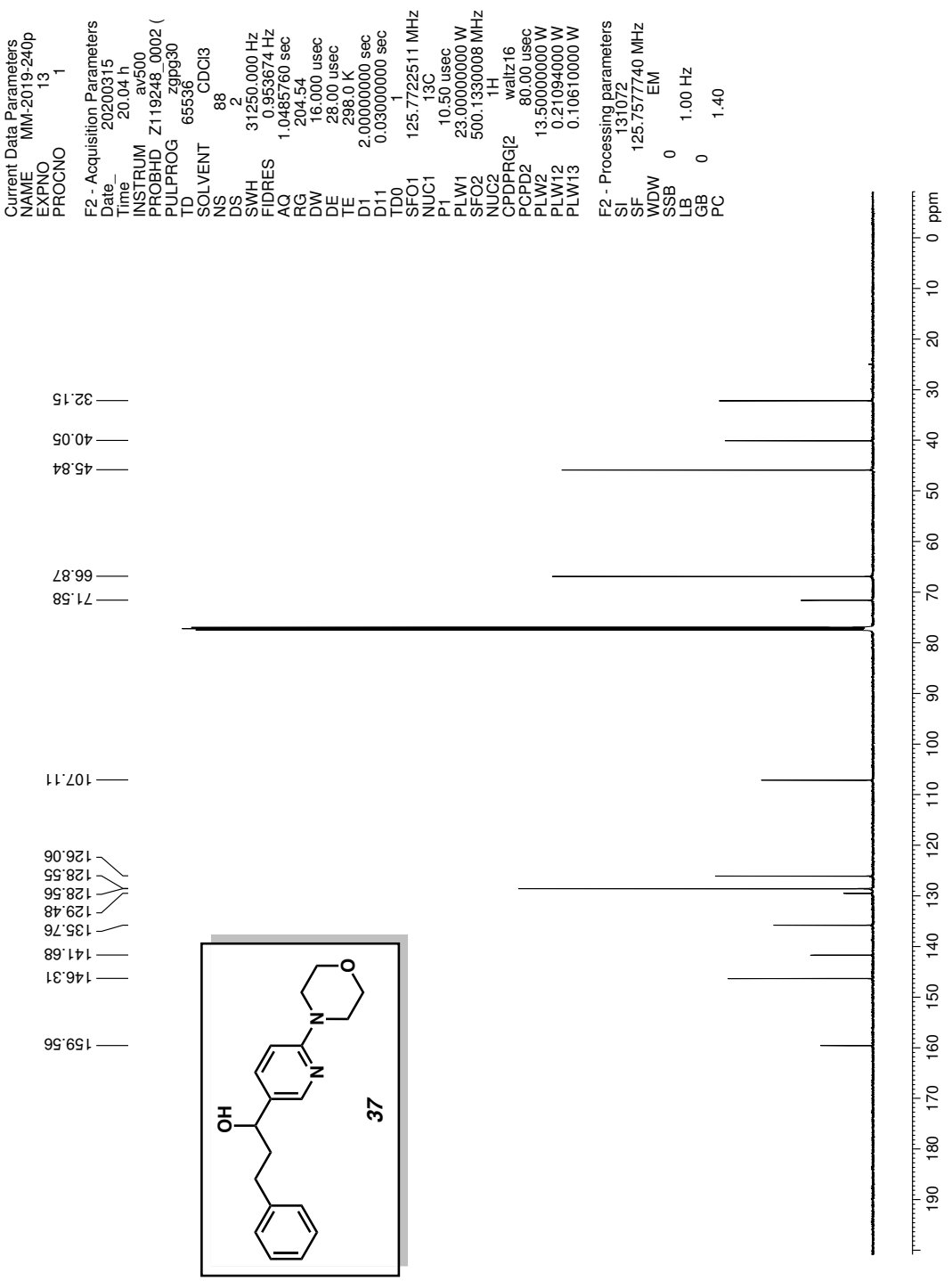

Current Data Parameters
NAME MM-2020-034p
EXPNO 17
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200709
Time_ 13.49 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 216
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
PLW1 23.00000000 W
SFO2 500.1330008 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.50000000 W
PLW12 0.21094000 W
PLW13 0.10610000 W
F2 - Processing parameters
SI 131072
SF 125.7577731 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
27.92
28.43
28.56
43.65
77.88
79.58
123.14
125.30
125.38
125.41
125.44
125.47
126.99
129.64
129.90
130.15
130.41
147.10
154.90
OH
CF3
N
Boc
32
ppm

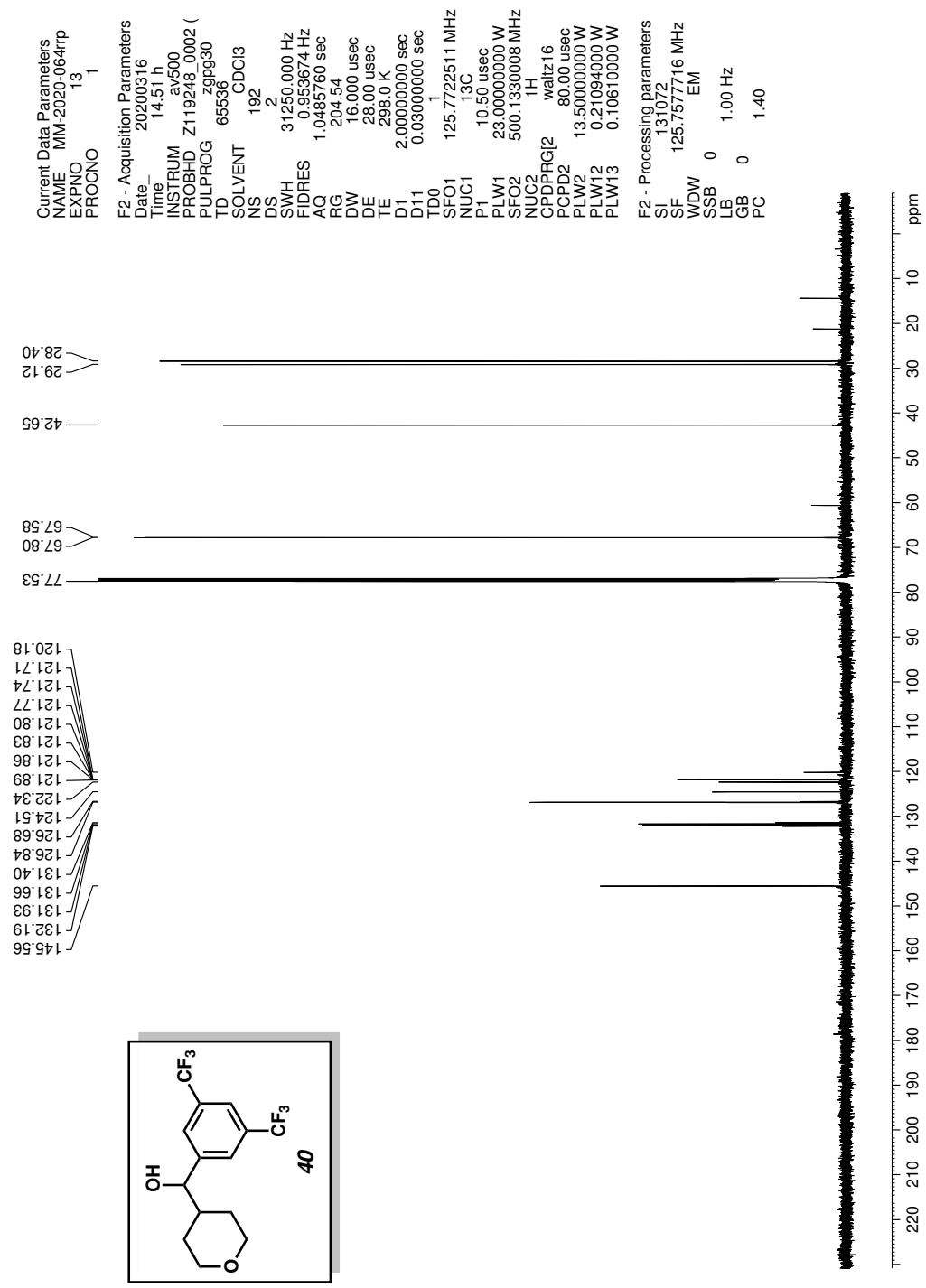

Current Data Parameters
NAME MM-2020-035p
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200216
Time 19.27 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 56
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 usec
TE 298.0 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 125.7577745 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
28.43
28.58
43.53
78.83
79.44
124.51
125.68
126.10
126.39
127.83
128.03
128.39
133.20
133.25
140.50
154.92
OH
N
Boc
33
0 ppm


Current Data Parameters
NAME MM-2019-284rp
EXPNO 250
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191204
Time_ 20.51
INSTRUM av400
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
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 296.6 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 100.6127578 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
28.33
29.09
29.29
42.61
67.73
67.95
78.43
81.22
126.50
129.65
131.63
147.52
165.68
OH
Ot-Bu
O
O
34
0 ppm


Current Data Parameters
NAME MM-2020-039rp
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200218
Time 17.50 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 48
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 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 125.7577754 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
22.18
29.33
29.44
42.45
67.81
67.99
69.93
78.88
114.29
115.02
118.89
129.49
144.71
158.08
ppm
OH
O-i-Pr
O
35


Current Data Parameters
NAME MM-2020-065rrp
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200315
Time 20.17 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 88
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 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
32.33
40.44
40.80
74.60
110.14
112.07
114.31
125.92
128.48
128.62
129.37
142.12
145.71
150.98
NMe2
OH
36
ppm


Current Data Parameters
NAME MM-2019-240p
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200315
Time_ 20.04 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 88
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 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 125.7577740 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
32.15
40.05
45.84
66.87
71.58
107.11
126.06
128.55
128.56
129.48
135.76
141.68
146.31
159.56
OH
N
N
O
37
ppm


Current Data Parameters
NAME MM-2020-064rrp
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200316
Time 14.51 h
INSTRUM av500
PROBHD Z119248_0002 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 192
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 1.0485760 sec
RG 204.54
DW 16.000 usec
DE 28.00 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 125.7577716 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
28.40
29.12
42.65
67.58
67.80
77.53
120.18
121.71
121.74
121.77
121.80
121.83
121.86
121.89
122.34
124.51
126.68
126.84
131.40
131.66
131.93
132.19
145.56
OH
CF3
CF3
O
40
ppm