Ni-Catalyzed Suzuki–Miyaura Cross-Coupling of Aliphatic Amides on the Benchtop

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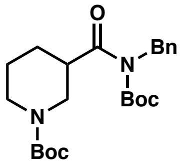
Supporting Information – Table of Contents

Materials and Methods. Unless stated otherwise, reactions were conducted in flame-dried glassware under an atmosphere of nitrogen or argon and commercially obtained reagents were used as received. Boronate esters **5**, **18**, **27**–**30** were obtained from Combi-Blocks. Ni(cod)$_2$ and Benz-ICy•HCl were obtained from Strem Chemicals. Potassium phosphate (K_3PO_4) was obtained from Acros. 1,4-Dioxane was obtained from Fisher Scientific and purified by distillation (over Na^0 and benzophenone) and degassed by sparging with N_2 for 1 h prior to use. Deionized water was degassed by sparging with N_2 for ≥10 min prior to use. 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) 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 absorption frequency (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 $CHCl_3$, $CDCl_3$, 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).

Note: Supporting information for the syntheses of amides **1**, **4**, **20**, **21**[1] and **22**–**24**[2] have been published and spectral data match those previously reported.



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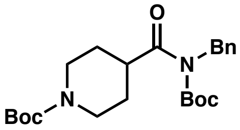

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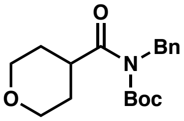

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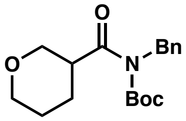

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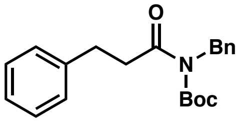

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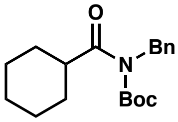

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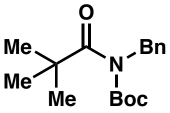

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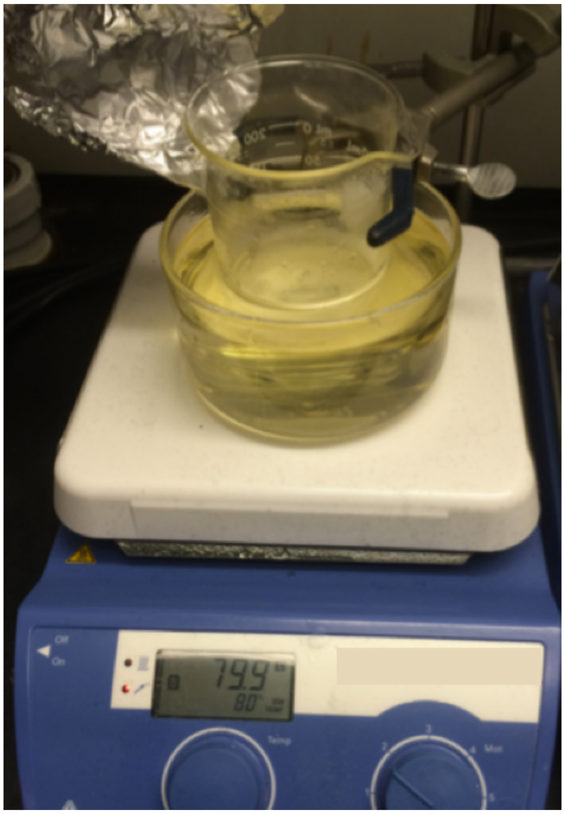
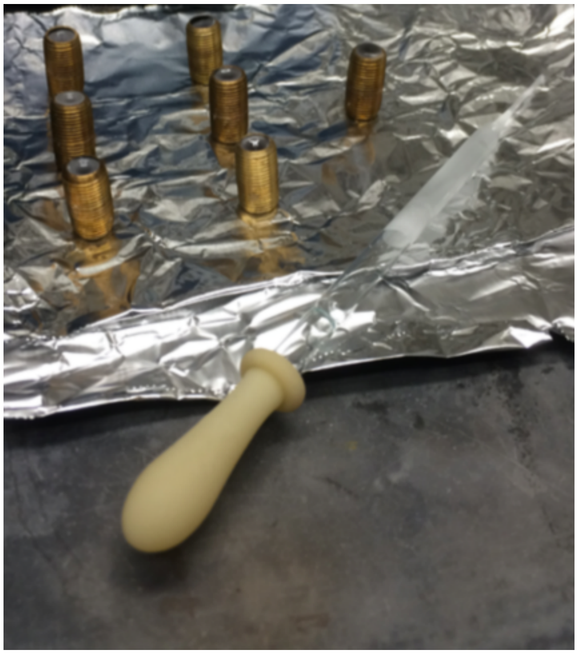
Experimental Procedures

A. Preparation of Paraffin Wax Capsules

Representative Procedure for the preparation of paraffin wax capsules for Sections C and D in the Experimental Procedures. Paraffin wax (mp 53–57 °C ASTM D 87) was melted in a 250 mL beaker suspended in an oil bath maintained at 80 °C.



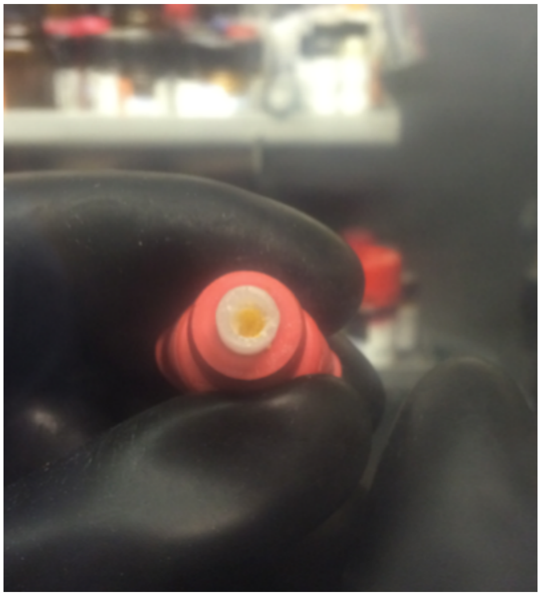
The molten paraffin was then pipetted into a standard brass mold (Brass Nipple, 1/8 in x close) using a 5 3/4 in glass pipette and pipette bulb.



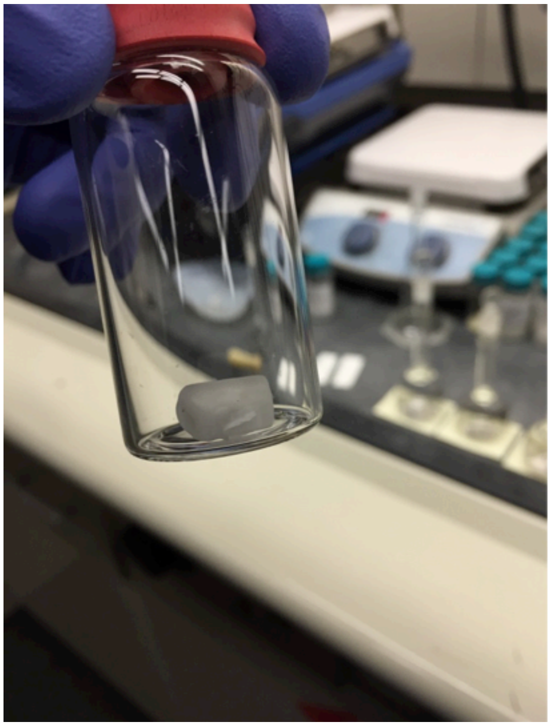
After cooling, the resulting wax cylinder was removed from the brass mold and trimmed to approximately 1 cm in length using a razor blade.

Next, a cavity was bored in the wax cylinder using a standard drill bit (5/32 in, black oxide), taking care not to bore through the entire cylinder.

The resulting hollow and open capsule was brought into a glovebox, inserted into a 14/20 septum for ease of handling, and charged with Ni(cod)$_2$ (5.5 mg, 0.020 mmol, 5 mol%) and Benz-ICy•HCl (12.8 mg, 0.040 mmol, 10 mol%).



After charging the capsule, a warm metal spatula (maintained at approximately 80 °C using a hot plate in the glovebox) was used to melt the top of the capsule closed. Removal from the glovebox and re-dipping in molten wax twice (to ensure a proper seal) gave the desired capsules that were ready for use on the benchtop. The capsules were stored in a freezer maintained at –20 °C under an atmosphere of air until use.

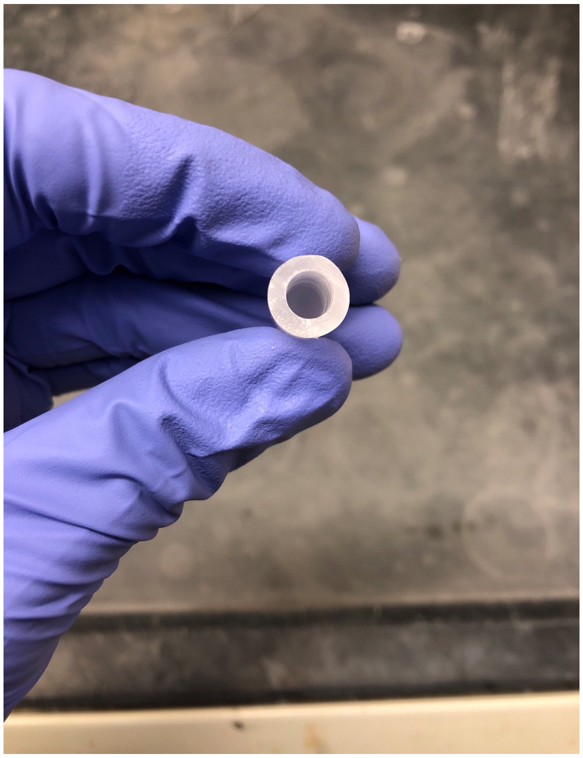


Note: Supporting information for the preparation of similar paraffin capsules has been previously disclosed.[3] Typically, paraffin wax capsules generated in this way were used within 1–2 weeks of being prepared. The stability of paraffin capsules to air and moisture was examined over a period of two months and is detailed in Section C in the Experimental Procedures.

B. Preparation of Paraffin Wax Capsules for Gram Scale Coupling

Representative Procedure for preparation of paraffin wax capsules for Section E in the Experimental Procedures. Paraffin wax (mp 53–57 °C ASTM D 87) was melted in a 250 mL beaker suspended in an oil bath maintained at 80 °C. The molten paraffin (approximately 4 mL) was then pipetted into a standard glass VWR culture tube (12 x 75 mm) using a 5 3/4 in glass pipette and pipette bulb. After cooling, the resulting wax cylinder was removed from the culture tube (by scoring and carefully breaking the glass away from the paraffin) and trimmed to approximately 2.0 cm in length using a razor blade.

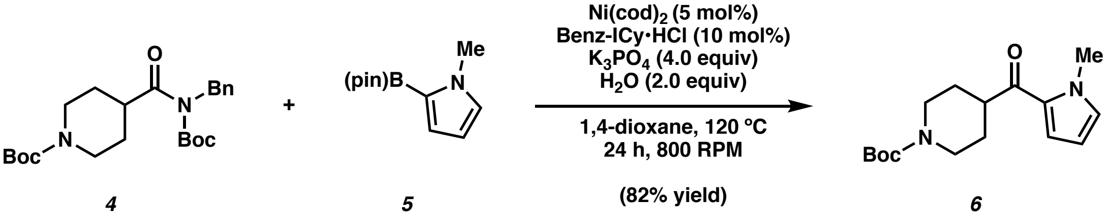
Next, a cavity was bored in the wax cylinder using a standard drill bit (15/64 in, black oxide), taking care not to bore through the entire cylinder.



The resulting hollow and open capsule was brought into a glovebox and charged with $Ni(cod)_2$ (32.9 mg, 0.119 mmol, 5 mol%) and Benz-ICy•HCl (76.2 mg, 0.239 mmol, 10 mol%). After charging the capsule, a warm metal spatula (maintained at approximately 80 °C using a hot plate in the glovebox) was used to melt the top of the capsule closed. Removal from the glovebox and re-dipping in molten wax twice (to ensure a proper seal) gave the desired capsules that were ready for use on the benchtop (Section E in the Experimental Procedures).

Note: Supporting information for the preparation of similar gram-scale paraffin capsules has been previously disclosed.[3]

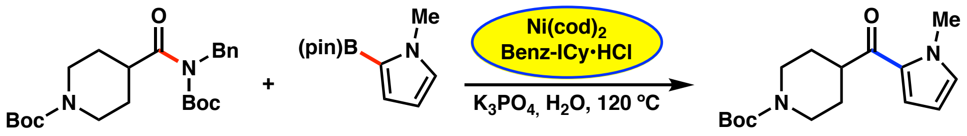
C. Optimization of Methodology



Representative Procedure for Table 1 (coupling of amide 4 and *N*-methylpyrrole-2-boronic acid pinacol ester (5) is used as an example). 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 **4** (167 mg, 0.40 mmol, 1.00 equiv), *N*-methylpyrrole-2-boronic acid pinacol ester (**5**, 414 mg, 2.00 mmol, 5.00 equiv), and a paraffin wax capsule containing $Ni(cod)_2$ (5.50 mg, 0.02 mmol, 0.05 equiv) and Benz-ICy•HCl (12.8 mg, 0.04 mmol, 0.10 equiv) prepared as described in Section A of the Experimental Procedures. The vial was purged with N_2 and subsequently deionized water (14.0 µL, 0.80 mmol, 2.00 equiv) and 1,4-dioxane (0.40 mL, 1.00 M) 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 24 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 and the yield of ketone **6** was determined by ^{1}H NMR analysis with 1,3,5-trimethoxybenzene as an external standard.[2]

Any modifications of the conditions shown in the representative procedure

above are specified below in Table 1.



4 *5* *6*

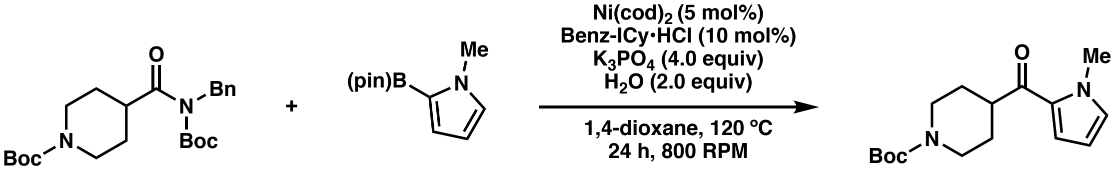
Entry	*Solvent (1.0 M)*	*equiv. 5*	*Time*	*Stir Rate*	*Yield of 6* [a]
1	toluene	2.5	16	400 RPM	28%
2	1,4-dioxane	2.5	16	400 RPM	71%
3	1,4-dioxane	5.0	24	800 RPM	91%
4[b]	1,4-dioxane	2 month stability test			97%

[a] Yields were determined by ^{1}H NMR analysis using 1,3,5-trimethoxybenzene as an external standard and reflect the average of two experiments.

[b] Reaction performed using conditions outlined in Entry 3.

Table 1: Optimization Studies

D. Scope of Methodology

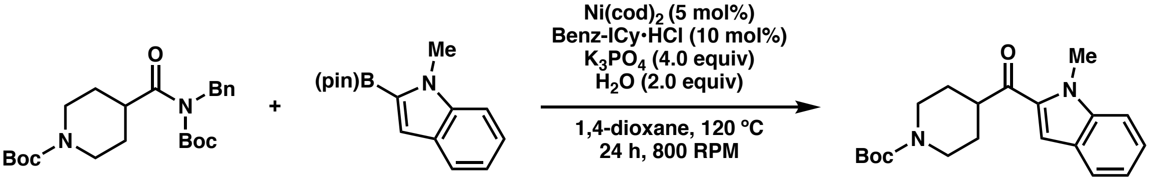


(82% yield)

4 *5* *6*

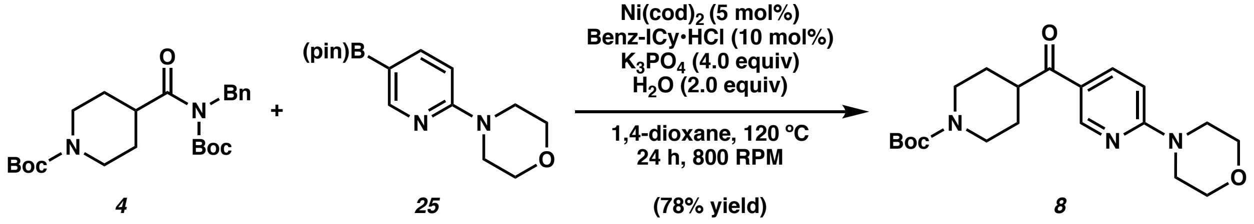
Representative Procedure for Figures 3 and 4 (coupling of amide 4 and *N*-methylpyrrole-2-boronic acid pinacol ester (5) is used as an example). Ketone 6. A 2-dram vial was charged with anhydrous powder K$_3$PO$_4$ (340 mg, 1.60 mmol, 4.00 equiv) and a magnetic stir bar (egg-shaped 3/8 x 3/16 in). The 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 **4** (167 mg, 0.40 mmol, 1.00 equiv), *N*-methylpyrrole-2-boronic acid pinacol ester (414 mg, 2.00 mmol, 5.0 equiv), and a paraffin wax capsule containing Ni(cod)$_2$ (5.50 mg, 0.02 mmol, 0.05 equiv) and Benz-ICy•HCl (12.8 mg, 0.04 mmol, 0.10 equiv) prepared as described in Section A of the Experimental Procedures. The vial was purged with N$_2$ and subsequently deionized water (14.0 µL, 0.8 mmol, 2.00 equiv) and 1,4-dioxane (0.40 mL, 1.00 M) 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 24 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 and the crude residue was purified by flash column chromatography (19:1 Hexanes:EtOAc → 9:1 Hexanes:EtOAc) to yield ketone **6** (82% yield, average of two experiments) as a yellow oil. Ketone **6**: R_f 0.25 (5:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.98 (dd, J = 4.1, 1.7, 1H), 6.83 (t, J = 2.0, 1H), 6.14 (q, 1H), 4.18 (br s, 2H), 3.93 (s, 3H), 3.21–3.10 (m, 1H), 2.82 (br s, 2H), 1.85–1.64 (m, 4H), 1.47 (s, 9H). Spectral data match those previously reported.[2]

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

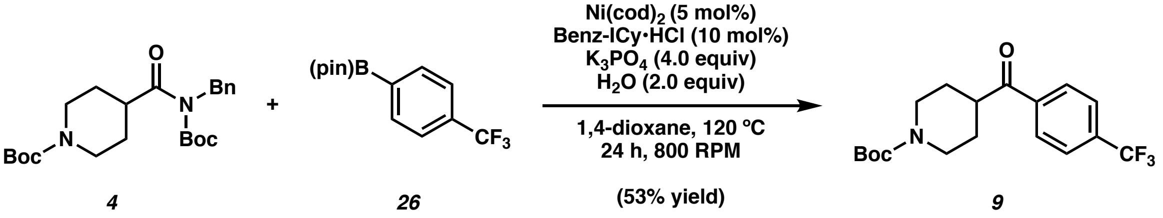


4 *18* (69% yield) *7*

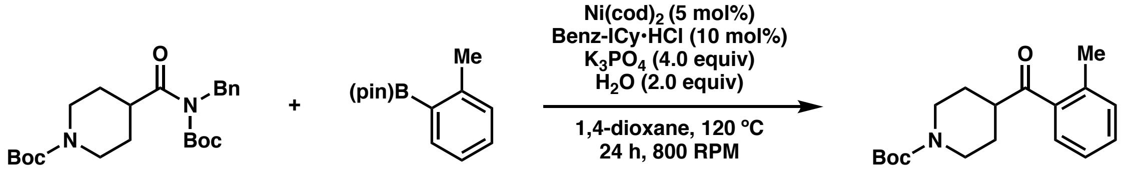
Ketone 7. Purification by flash chromatography (19:1 Hexanes:EtOAc → 9:1 Hexanes:EtOAc) generated ketone **7** (69% yield, average of two experiments) as a white solid. Ketone **7**: R_f 0.33 (5:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.70 (d, J = 7.9, 1H), 7.39 (d, J = 3.9, 2H), 7.33 (s, 1H), 7.20–7.14 (m, 1H) 4.21 (br s, 2H), 4.07 (s, 3H), 3.47–3.32 (m, 1H), 2.88 (br s, 2H), 1.86 (br s, 2H), 1.82–1.70 (m, 2H), 1.48 (s, 9H). Spectral data match those previously reported.[2]



Ketone 8. ^{1}H NMR analysis of the crude reaction mixture indicated a 78% yield of alcohol **8** relative to a 1,3,5-trimethoxybenzene external standard (average of two experiments). Purification by preparative thin-layer chromatography (1:1 Hexanes:EtOAc) provided an analytical sample of ketone **8** as a white solid. Ketone **8**: R_f 0.30 (1:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.78 (d, J = 2.6, 1H), 8.05 (dd, J = 9.1, 2.5, 1H), 6.63 (d, J = 9.1, 1H), 4.16 (br s, 2H), 3.81 (t, J = 5.2, 4H), 3.68 (t, J = 4.7, 4H), 3.32–3.21 (m, 1H), 2.87 (br s, 2H), 1.79 (br s, 2H), 1.76–1.65 (m, 2H), 1.46 (s, 9H). Spectral data match those previously reported.[2]

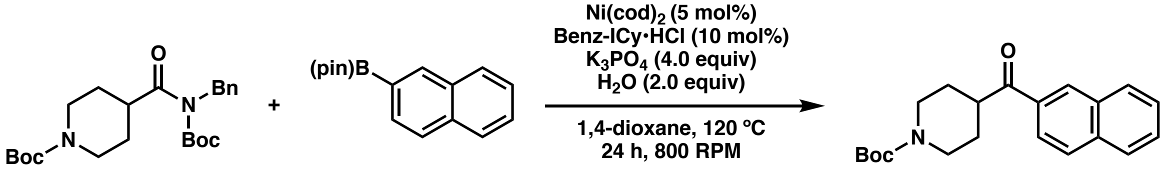


Ketone 9. Purification by flash chromatography (19:1 Hexanes:EtOAc → 9:1 Hexanes:EtOAc) generated ketone **9** (53% yield, average of two experiments) as a white solid. Ketone **9**: R_f 0.33 (5:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.03 (d, J = 8.4, 2H), 7.74 (d, J = 8.4, 2H), 4.16 (br s, 2H), 3.46–3.31 (m, 1H), 2.91 (br s, 2H), 1.85 (d, J = 13.3, 2H), 1.76–1.64 (m, 2H), 1.46 (d, J = 4.0, 9H). Spectral data match those previously reported.[2]



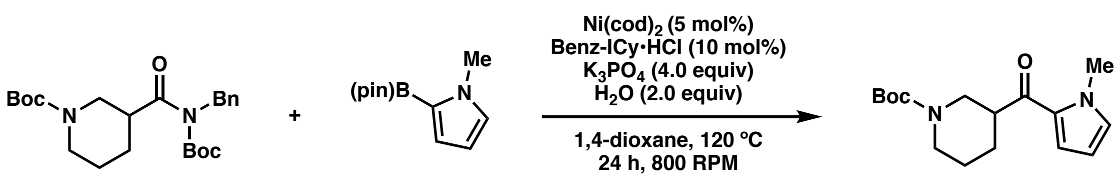
4 27 (74% yield) 10

Ketone 10. Purification by flash chromatography (24:1 Hexanes:EtOAc → 5:1 Hexanes:EtOAc) generated ketone **10** (74% yield, average of two experiments) as a yellow oil. Ketone **10**: R*f* 0.16 (9:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, CDCl$_3$): δ 7.50 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.36 (td, *J* = 7.4, 1.3, 1H), 7.26–7.22 (m, 2H), 4.12 (br s, 2H), 3.18 (tt, *J* = 11.2, 3.6, 1H), 2.84 (br s, 2H), 2.41 (s, 3H), 1.81 (d, *J* = 13.1, 2H), 1.60–1.58 (m, 2H), 1.46 (s, 9H). Spectral data match those previously reported.[2]



4 28 (71% yield) 11

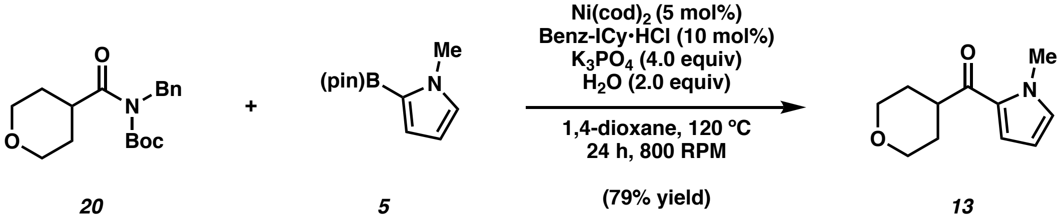
Ketone 11. Purification by flash chromatography (19:1 Hexanes:EtOAc → 14:1 Hexanes:EtOAc) generated ketone **11** (71% yield, average of two experiments) as a white solid. Ketone **11**: R*f* 0.16 (9:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, CDCl$_3$): δ 8.45 (s, 1H), 8.04–7.85 (m, 4H), 7.65–7.53 (m, 2H), 4.20 (br s, 2H), 3.58 (tt, *J* = 11.2, 4.0, 1H), 2.96 (t, *J* = 2.8, 2H), 1.90 (br s, 2H), 1.83–1.69 (m, 2H), 1.48 (s, 9H). Spectral data match those previously reported.[2]



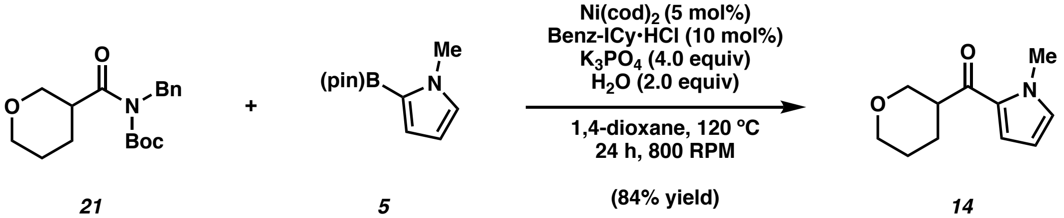
1 5 (92% yield) 12

Ketone 12. Purification by flash chromatography (19:1 Hexanes:EtOAc → 9:1 Hexanes:EtOAc) generated ketone **12** (92% yield, average of two experiments) as a light brown oil. Ketone **12**: R*f*

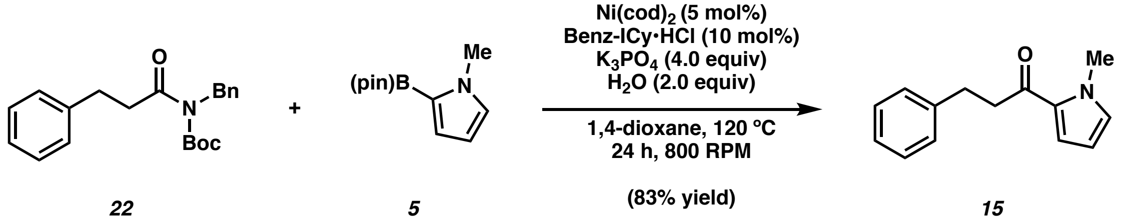
0.26 (5:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.05 (dd, *J* = 4.2, 1.6, 1H), 6.82 (t, *J* = 1.9, 1H), 6.14 (dd, *J* = 4.1, 2.5, 1H), 4.25 (br d, *J* = 13.5, 1H), 4.12 (br d, *J* = 11.7, 1H), 3.93 (br s, 3H), 3.16 (tt, *J* = 11.4, 3.6, 1H), 2.94–2.83 (m, 1H), 2.70 (td, *J* = 12.7, 2.4, 1H), 2.02–1.92 (m, 1H), 1.79–1.68 (m, 2H), 1.61–1.50 (m, 1H), 1.47 (s, 9H). Spectral data match those previously reported.[2]



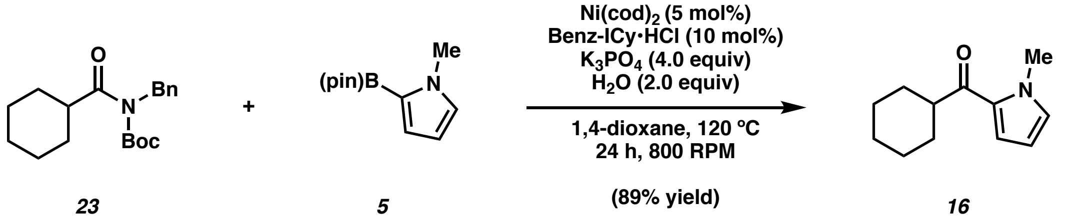
Ketone 13. Purification by sequential preparative thin-layer chromatography (9:1 Hexanes:EtOAc and 5:1 Hexanes:EtOAc) generated ketone **13** (79% yield, average of two experiments) as a white solid. Ketone **13**: R$_f$ 0.17 (5:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.99 (dd, *J* = 4.1, 1.6, 1H), 6.83 (t, *J* = 1.9, 1H), 6.14 (dd, *J* = 4.1, 2.5, 1H), 4.09–4.00 (m, 2H), 3.94 (s, 3H), 3.52 (td, *J* = 11.8, 2.1, 2H), 3.26 (tt, *J* = 11.5, 3.8, 1H), 1.97–1.86 (m, 2H), 1.74–1.67 (m, 2H). Spectral data match those previously reported.[2]



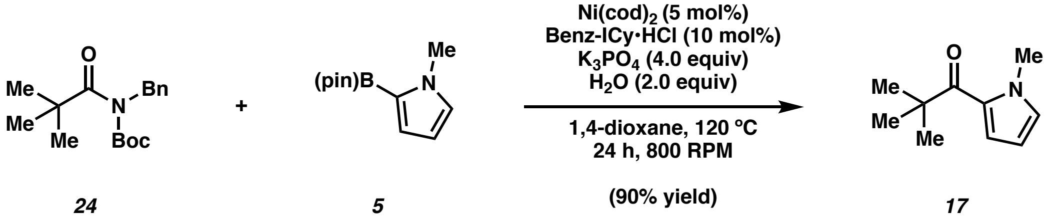
Ketone 14. Purification by flash chromatography (9:1 Hexanes:EtOAc) generated ketone **14** (84% yield, average of two experiments) as a colorless oil. Ketone **14**: R$_f$ 0.22 (5:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.03 (dd, *J* = 4.2, 1.7, 1H), 6.82 (t, *J* = 1.9, 1H), 6.14 (dd, *J* = 4.2, 2.5, 1H), 4.10–4.04 (m, 1H), 3.99–3.93 (m, 1H), 3.92 (s, 3H), 3.53 (t, *J* = 10.8, 1H), 3.46–3.34 (m, 2H), 2.02–1.94 (m, 1H), 1.91–1.80 (m, 1H), 1.80–1.65 (m, 2H). Spectral data match those previously reported.[2]



Ketone 15. Purification by flash chromatography (19:1 Hexanes:EtOAc) generated ketone **15** (83% yield, average of two experiments) as a colorless oil. Ketone **15**: R$_f$ 0.40 (5:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32–7.17 (m, 2H), 7.26–7.23 (m, 2H), 7.22–7.17 (m, 1H), 6.94 (dd, *J* = 4.1, 1.7, 1H), 6.80 (t, *J* = 1.9, 1H), 6.11 (dd, *J* = 4.1, 2.4, 1H), 3.95 (s, 3H), 3.14–3.08 (m, 2H), 3.05–2.99 (m, 2H). Spectral data match those previously reported.[2]

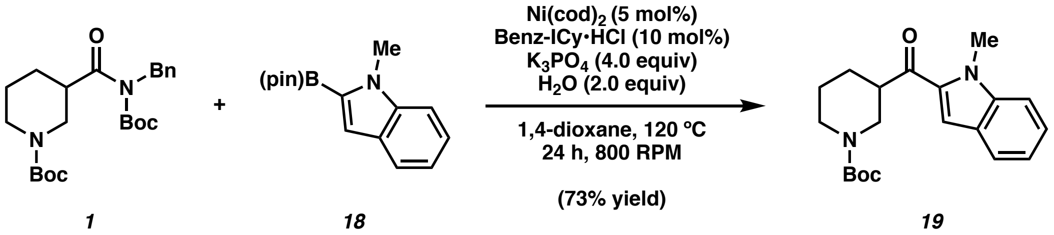


Ketone 16. Purification by flash chromatography (25:4:1 Hexanes:PhH:Et_2O) generated ketone **16** (89% yield, average of two experiments) as a colorless oil. Ketone **16**: R$_f$ 0.55 (5:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.97 (dd, *J* = 4.1, 1.7, 1H), 6.80 (t, *J* = 1.9, 1H), 6.12 (dd, *J* = 4.1, 2.5, 1H), 3.93 (s, 3H), 3.02 (tt, *J* = 11.7, 3.2, 1H), 1.88–1.79 (m, 4H), 1.76–1.67 (m, 1H), 1.56–1.45 (m, 2H), 1.41–1.30 (m, 2H), 1.29–1.19 (m, 1H). Spectral data match those previously reported.[2]



Ketone 17. ^{1}H NMR analysis of the crude reaction mixture indicated a 90% yield of ketone **17** relative to a 1,3,5-trimethoxybenzene external standard. Purification by preparative thin-layer chromatography (49:1 Cyclohexane:EtOAc), eluted twice, provided an analytical sample of ketone **17** as a colorless oil. Ketone **17**: R_f 0.65 (4:1 Hexanes:EtOAc). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.03 (dd, J = 4.1, 1.6, 1H), 6.75 (t, J = 1.9, 1H), 6.11 (dd, J = 4.1, 2.5, 1H), 3.90 (s, 3H), 1.36 (m, 9H). Spectral data match those previously reported.[2]

E. Gram-Scale Benchtop Suzuki–Miyaura Cross-Coupling



Ketone 19. A 20-mL scintillation vial was charged with anhydrous powder K_3PO_4 (2.03 g, 9.56 mmol, 4.00 equiv) and a magnetic stir bar (football shaped, 0.5 x 1.5 cm). The vial and its contents were flame-dried under reduced pressure and allowed to cool under N_2. The vial was charged with amide substrate **1** (1.00 g, 2.39 mmol, 1.00 equiv), boronic ester **18** (3.07 g, 11.9 mmol, 5.00 equiv), and a paraffin wax capsule containing Ni(cod)$_2$ (32.9 mg, 0.119 mmol, 0.050 equiv) and Benz-ICy•HCl (76.2 mg, 0.239 mmol, 0.100 equiv) prepared as described in Section A of the Experimental Procedures. The vial was purged with N_2 and subsequently deionized water (86.1 µL, 4.78 mmol, 2.00 equiv) and 1,4-dioxane (2.39 mL, 1.00 M) 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 24 h. After removing the vial from heat, the reaction mixture was transferred to a 100 mL pear-shaped flask containing 12.0 g of silica gel with hexanes (10 mL) and CH_2Cl_2 (10 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 and the crude residue was purified by flash column chromatography (14:1 Hexanes:Et_2O → 4:1 Hexanes:Et_2O) to yield ketone **19** (73% yield, average of two experiments) as a white amorphous solid. Ketone **19**: R_f 0.25 (5:1 Hexanes:EtOAc); ^1H NMR (500 MHz, $CDCl_3$): δ 7.70 (d, J = 8.1, 1H), 7.44–7.34 (m, 3H), 7.20–7.11 (m, 1H), 4.32 (br s, 1H), 4.24–3.96 (m, 4H), 3.47–3.27 (m, 1H), 2.98 (br s, 1H), 2.74 (br s, 1H), 2.12–2.00 (m, 1H), 1.84–1.68 (m, 2H), 1.67–1.54 (m, 1H), 1.49 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 195.3, 154.9, 140.5, 133.9, 126.3, 125.9, 123.2, 121.0, 111.9, 110.5, 79.9, 47.9, 46.2, 44.9, 44.0, 32.4, 28.64, 28.61, 24.8; IR (film): 2973, 2938, 2861, 1691, 1656, 1614, 1423, 1168, 1146, 970 cm^{-1}; HRMS-APCI (m/z) $[M + H]^+$ calcd for $C_{20}H_{27}N_2O_3$, 343.2016; found 343.2010.

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

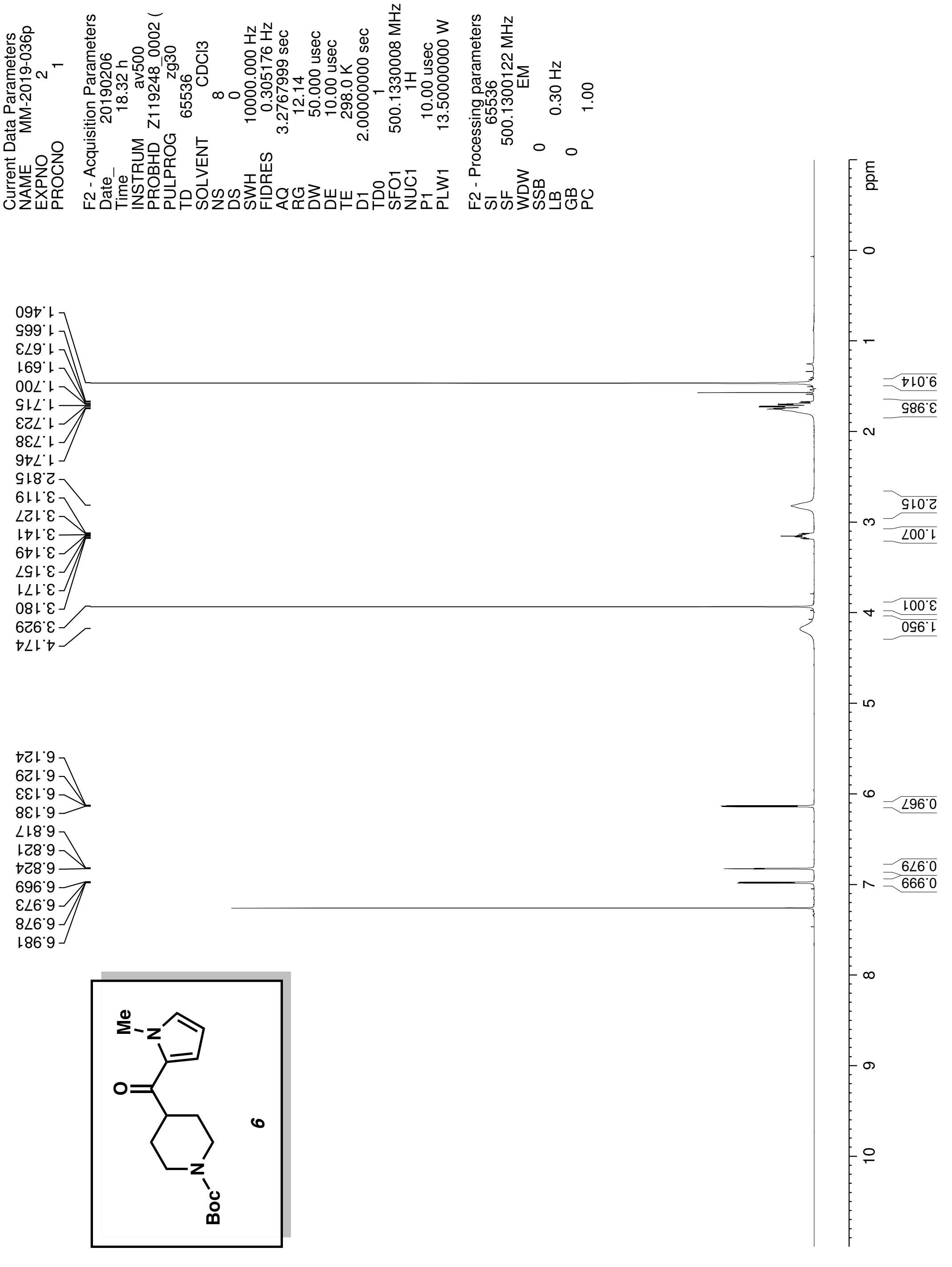
References

[1] Hie, L.; Baker, E. L.; Anthony, S. M.; Desrosiers, J.-N.; Senanayake, C.; Garg, N. K. Nickel-catalyzed esterification of aliphatic amides. *Angew. Chem., Int. Ed.* **2016**, *55*, 15129–15132.

[2] Boit, T. B.; Weires, N. A.; Kim, J.; Garg, N. K. Nickel-catalyzed Suzuki–Miyaura coupling of aliphatic amides. *ACS Catal.* **2018**, *2*, 1003–1008.

[3] Dander, J. E.; Weires, N. A.; Garg, N. K. Benchtop delivery of $Ni(cod)_2$ using paraffin capsules. *Org. Lett.* **2016**, *18*, 3934–3936.

^{1}H NMR Spectra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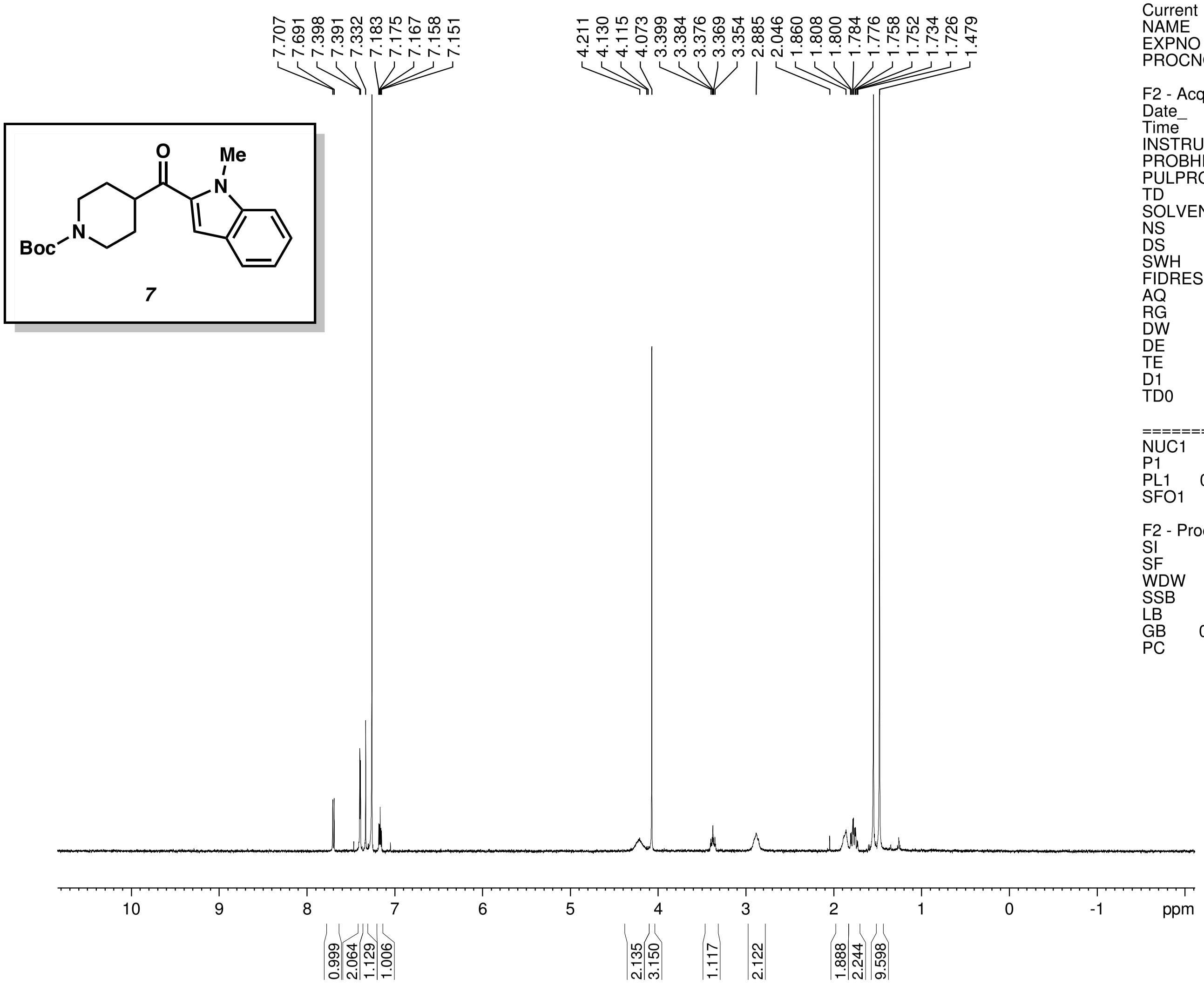
6



7

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FIDRES 0.152588 Hz
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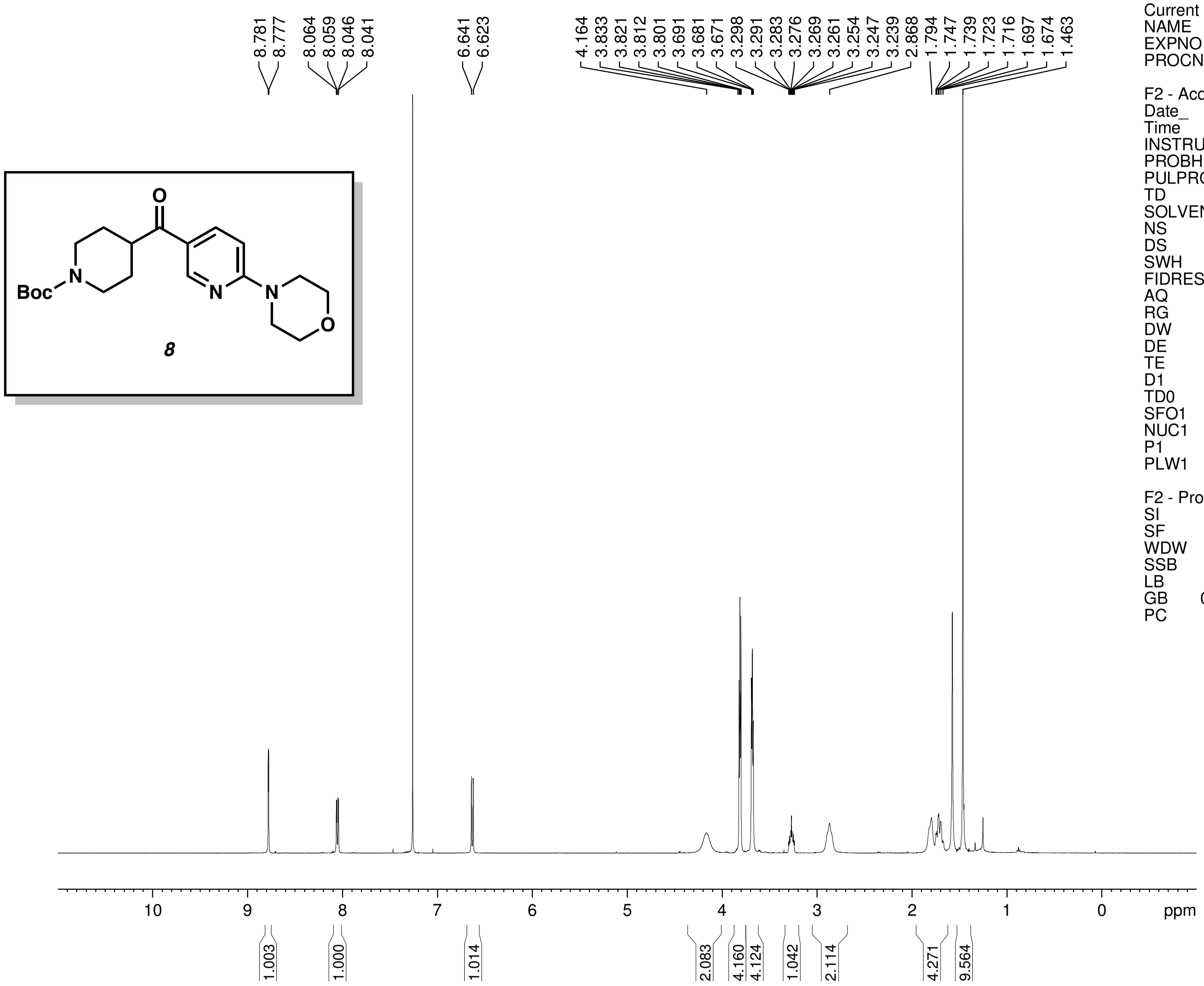

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RG              12.14
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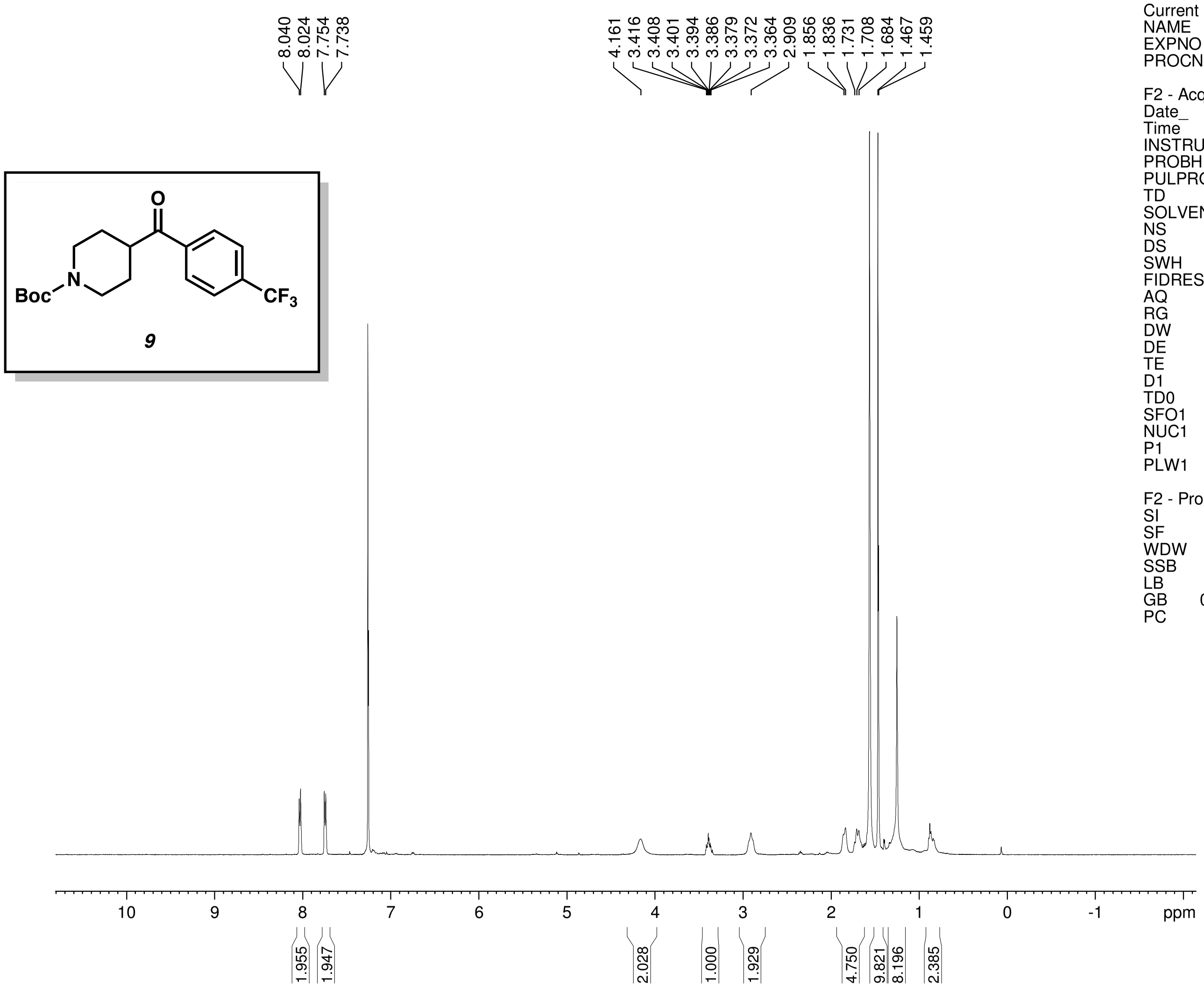
9



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PROCNO 1

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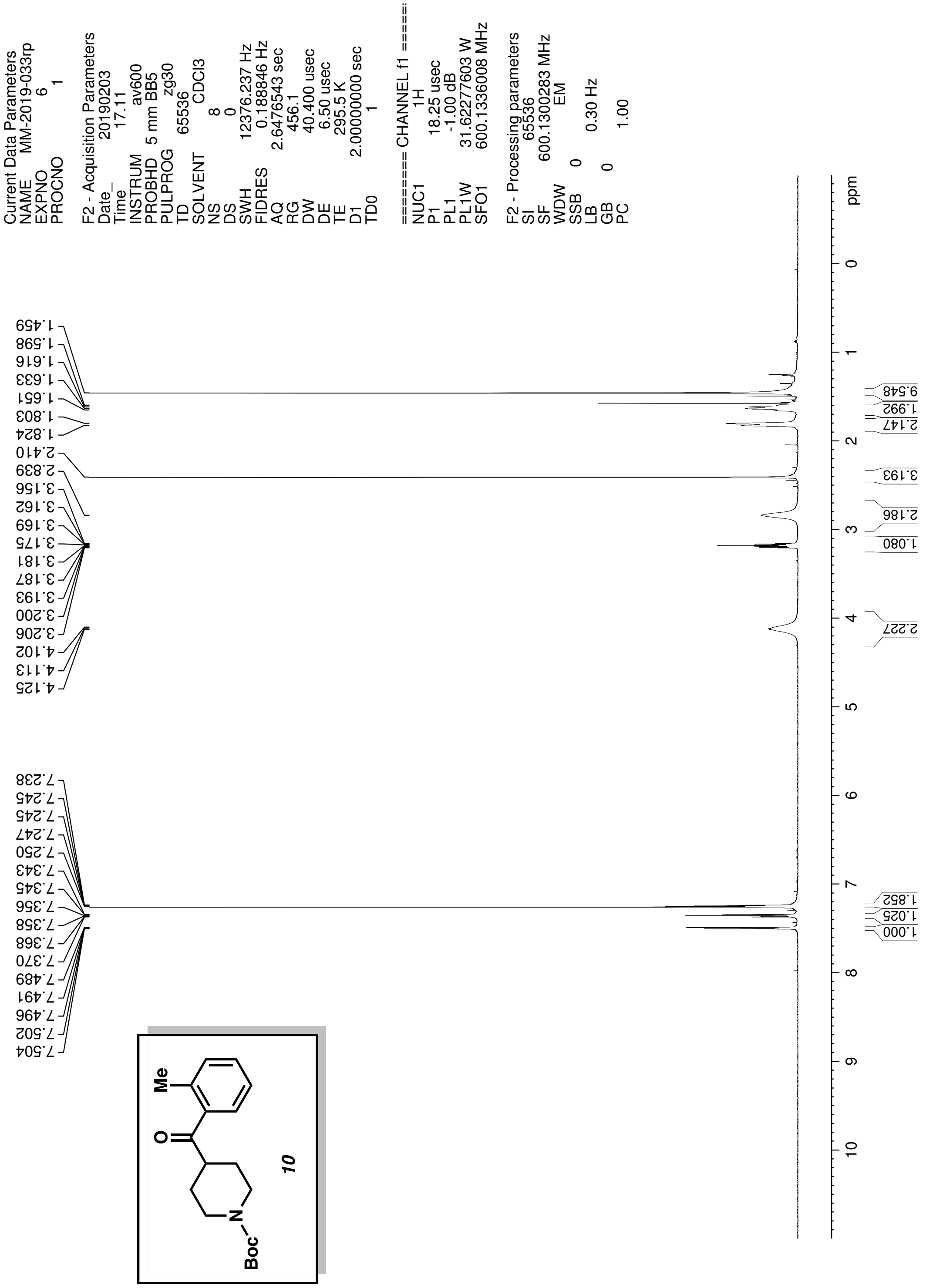
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FIDRES 0.188846 Hz
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D1 2.00000000 sec
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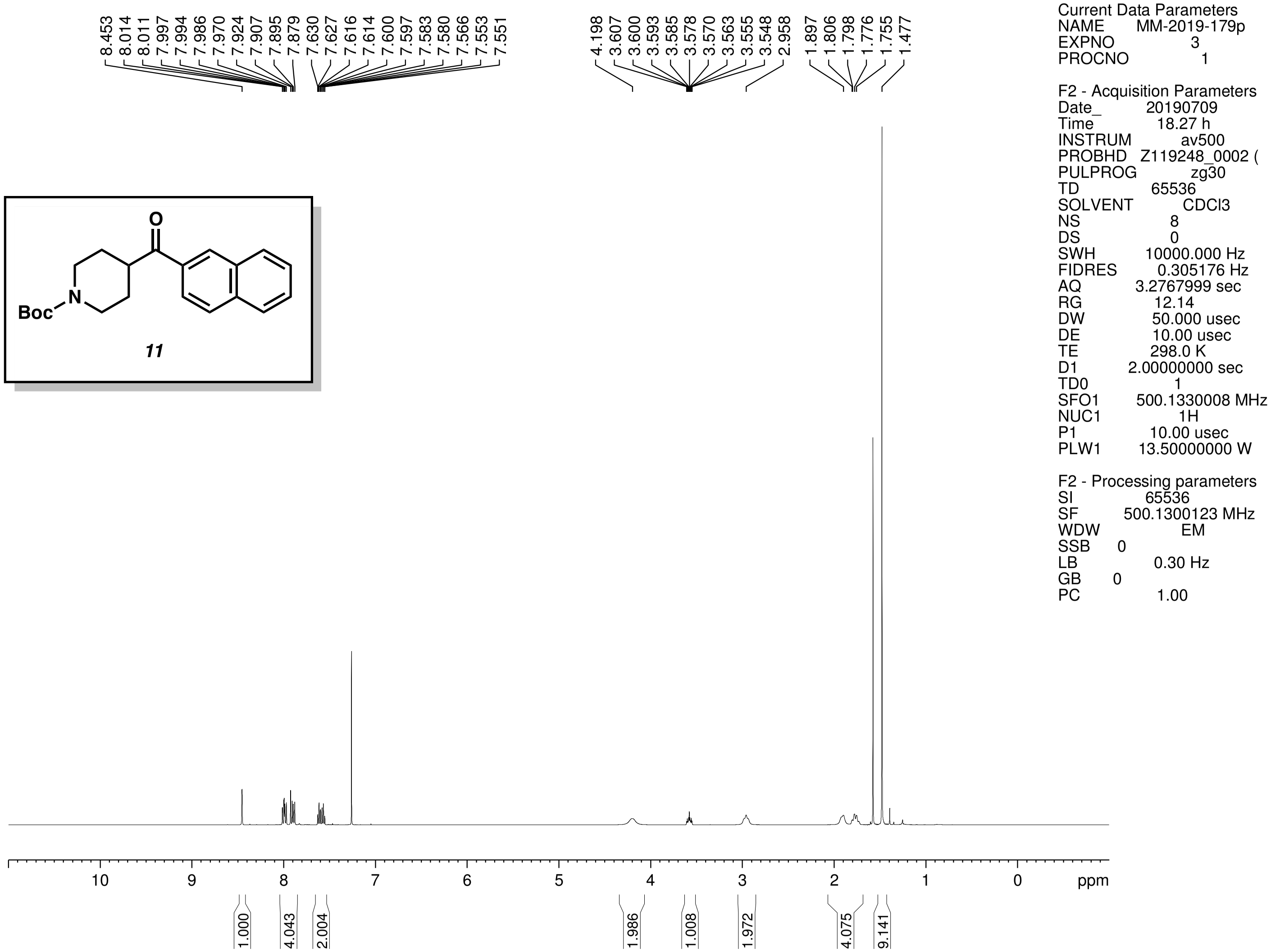
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PROCNO 1
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PULPROG zg30
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SOLVENT CDCl3
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NUC1 1H
P1 10.00 usec
PLW1 13.50000000 W
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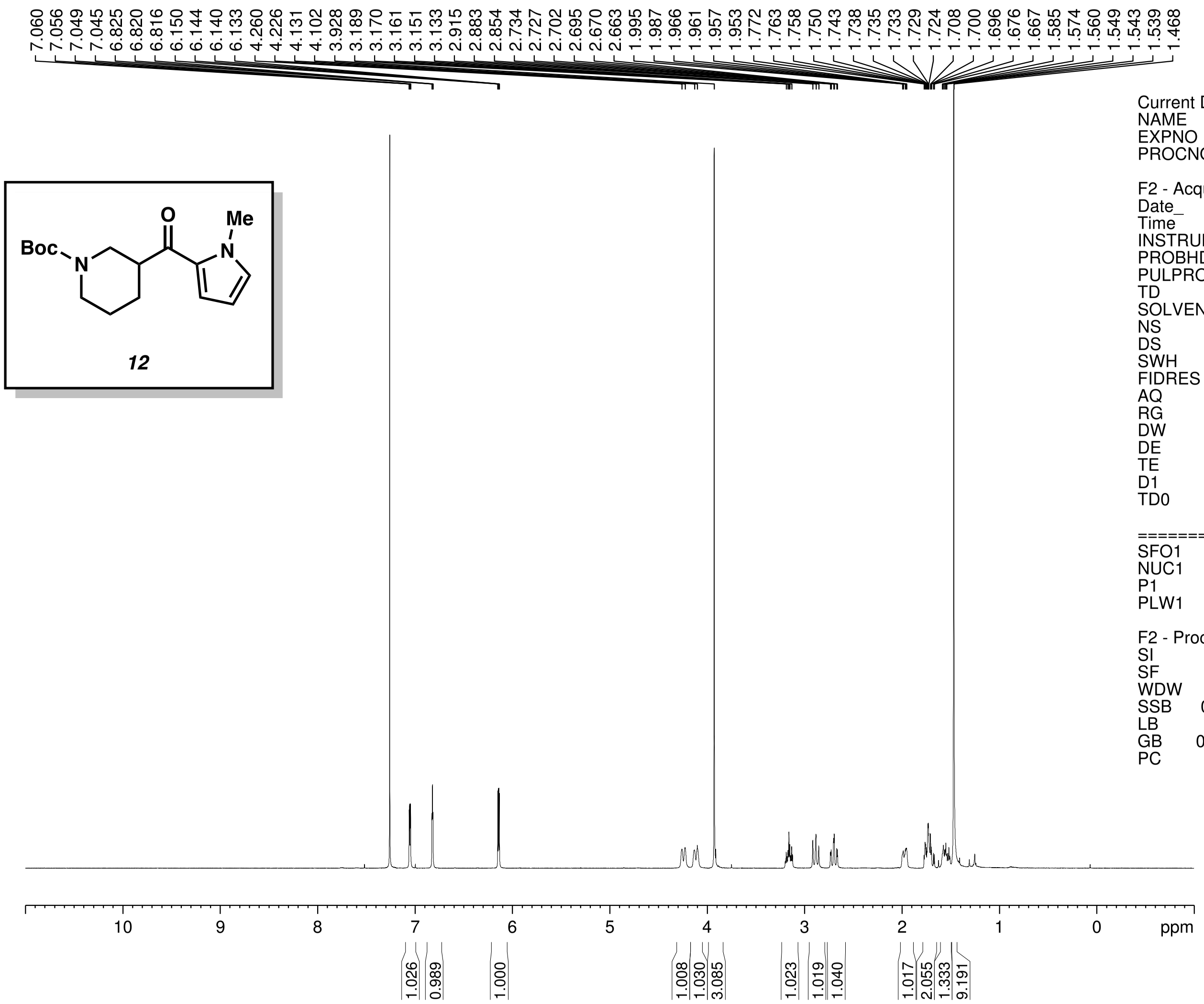

12

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PROCNO 1

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SOLVENT CDCl3
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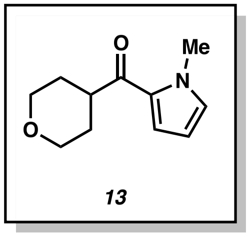
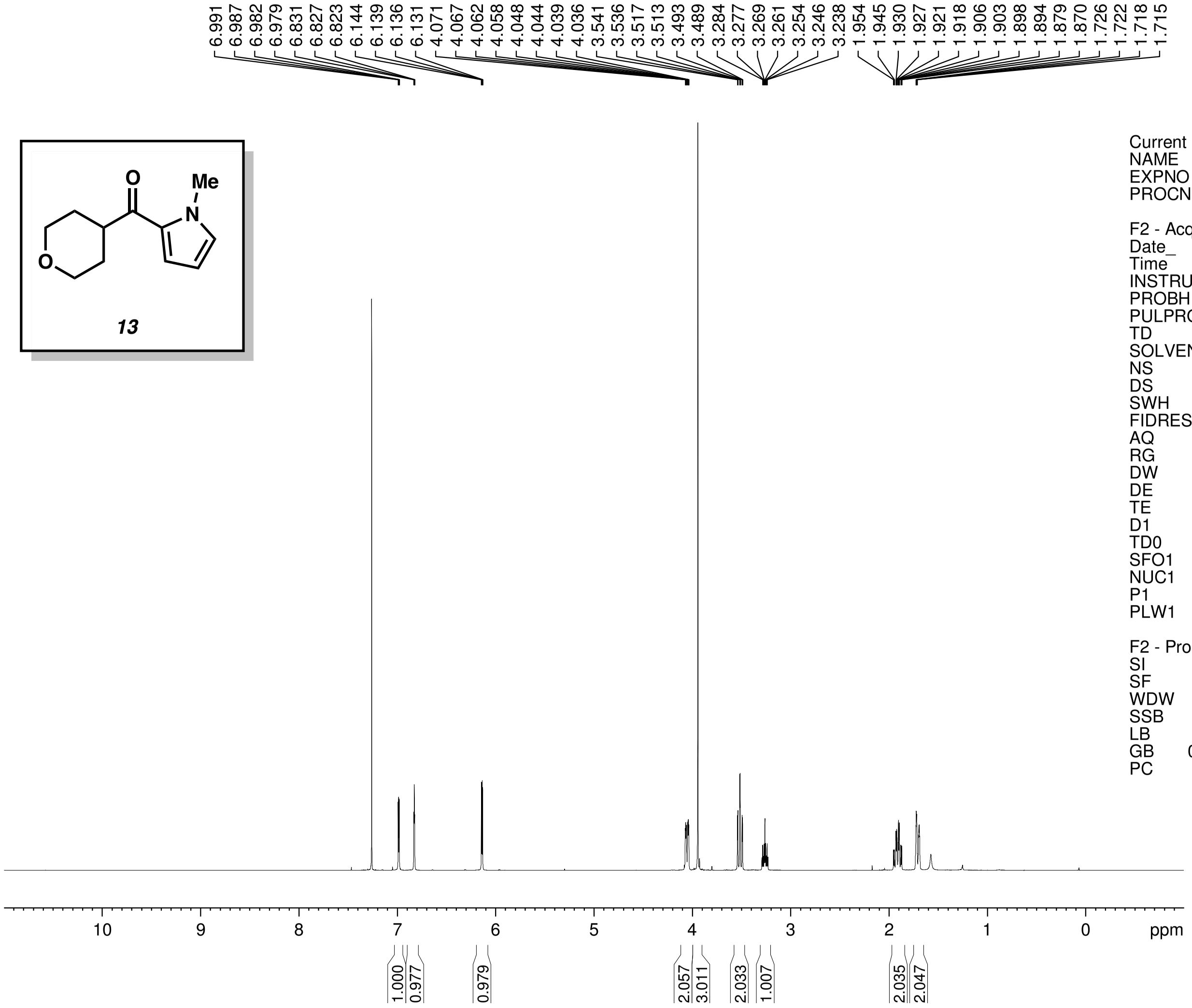


13



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TE	298.0 K
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F2 - Processing parameters

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PROCNO                1

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SOLVENT           CDCl3
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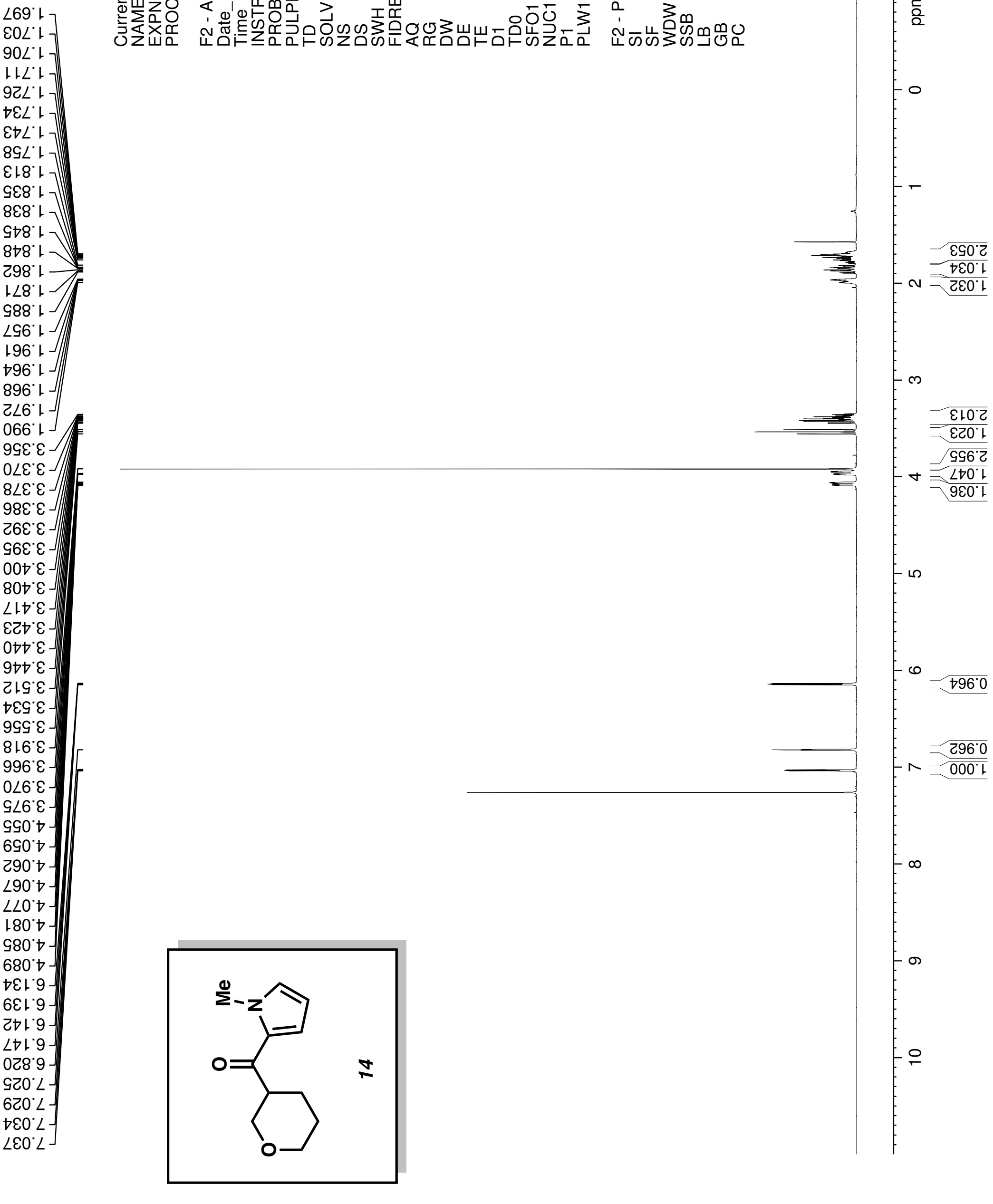

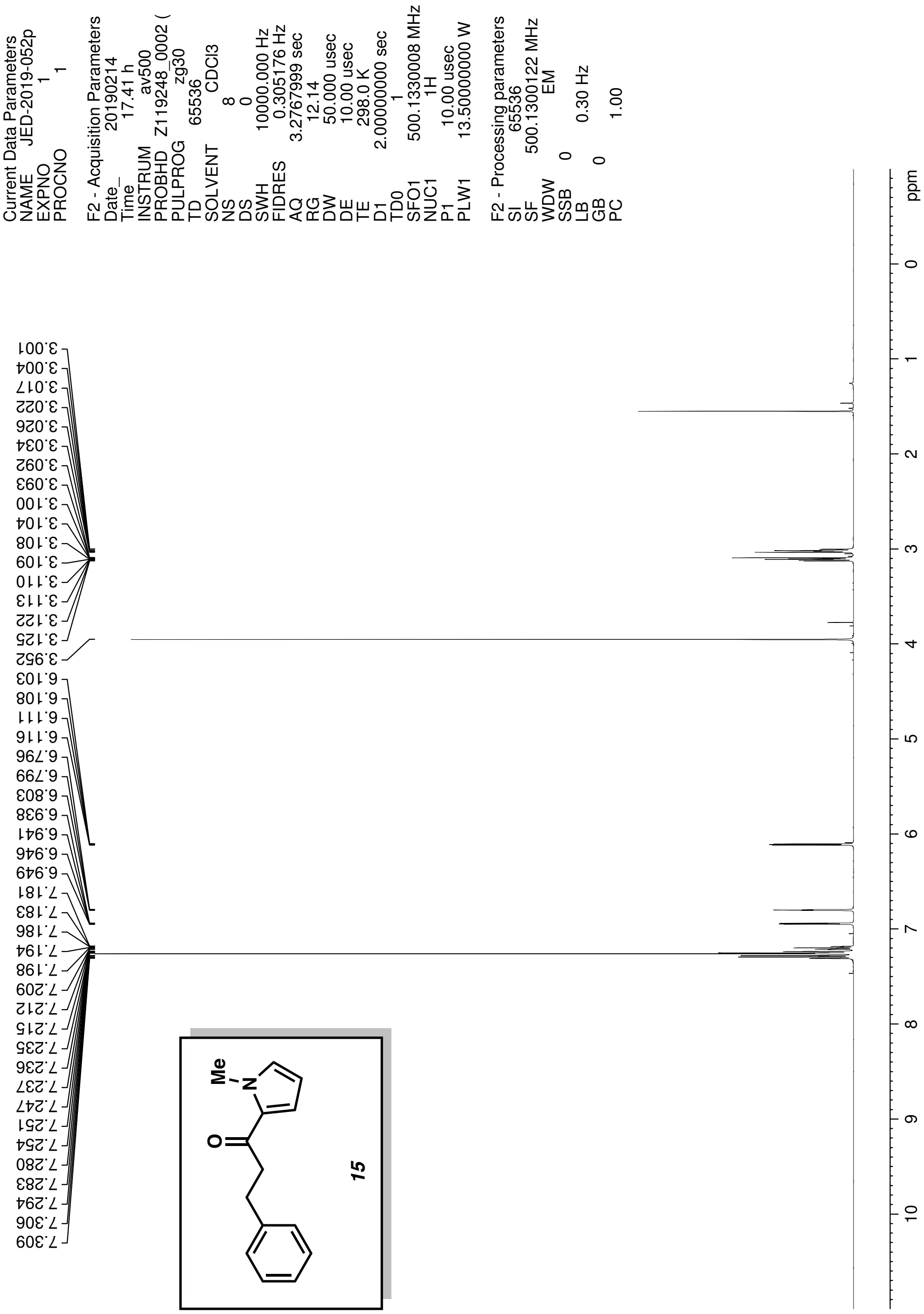

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PROCNO 1

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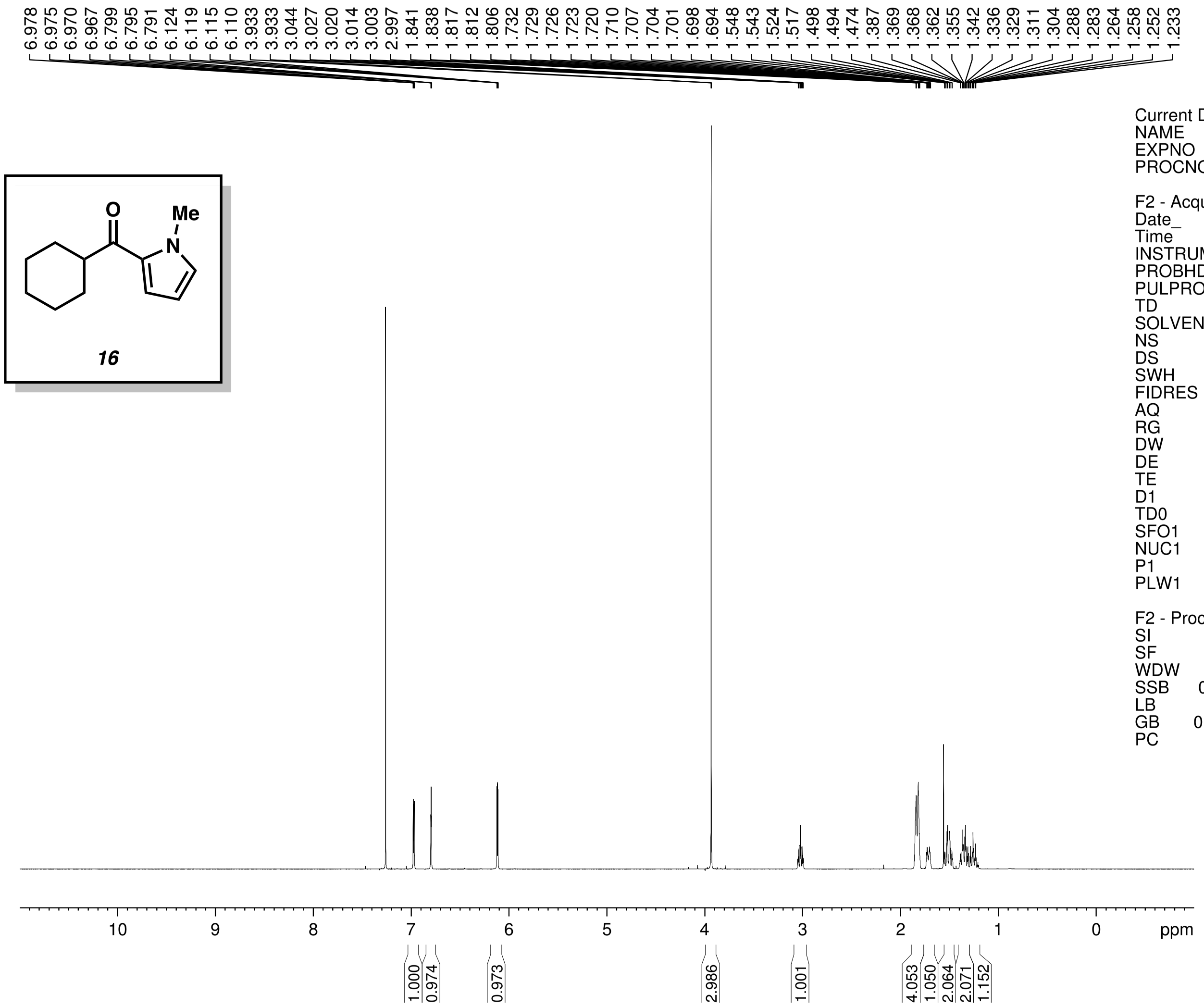

16

Current Data Parameters

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PROCNO	1

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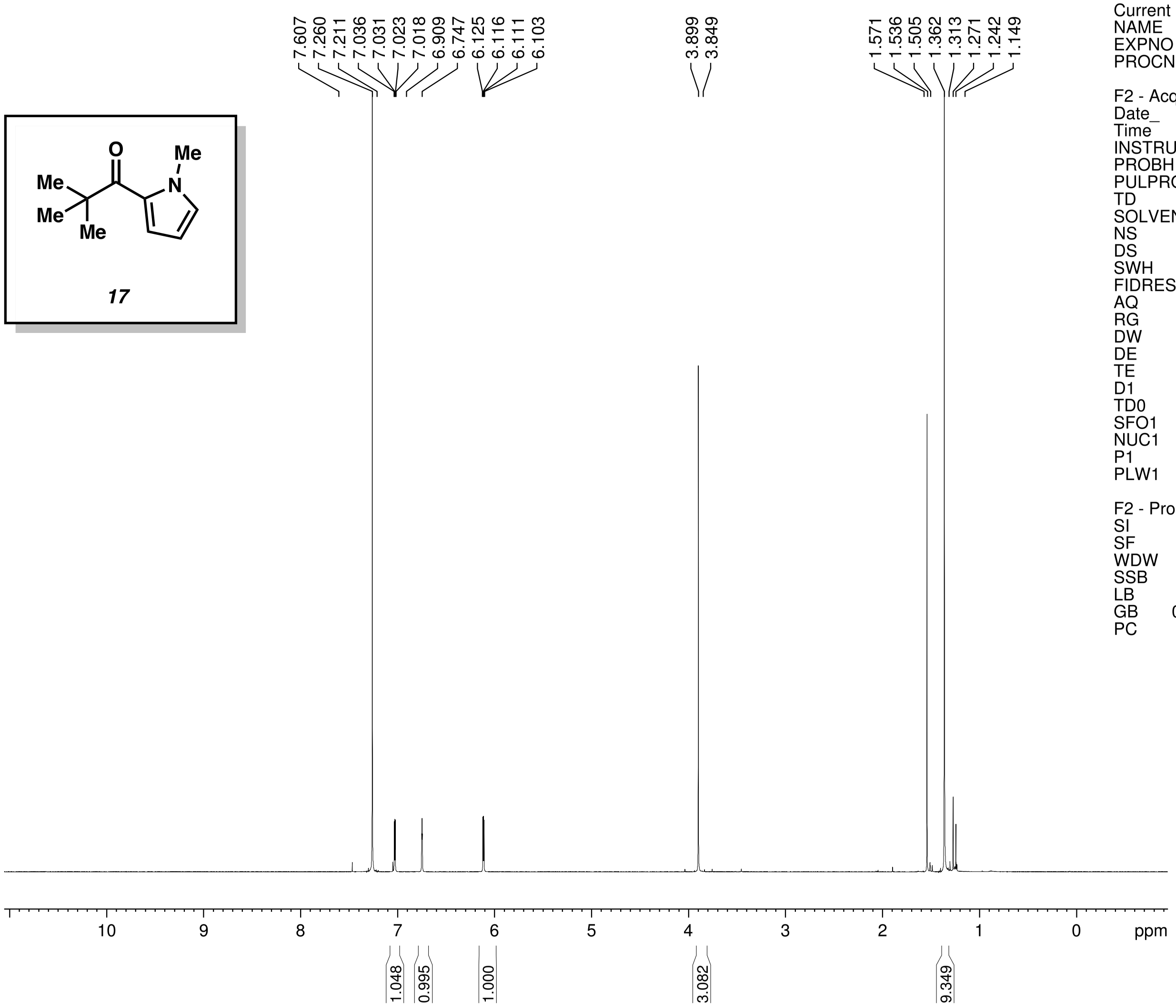
17



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SOLVENT          CDCl3
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TE             298.0 K
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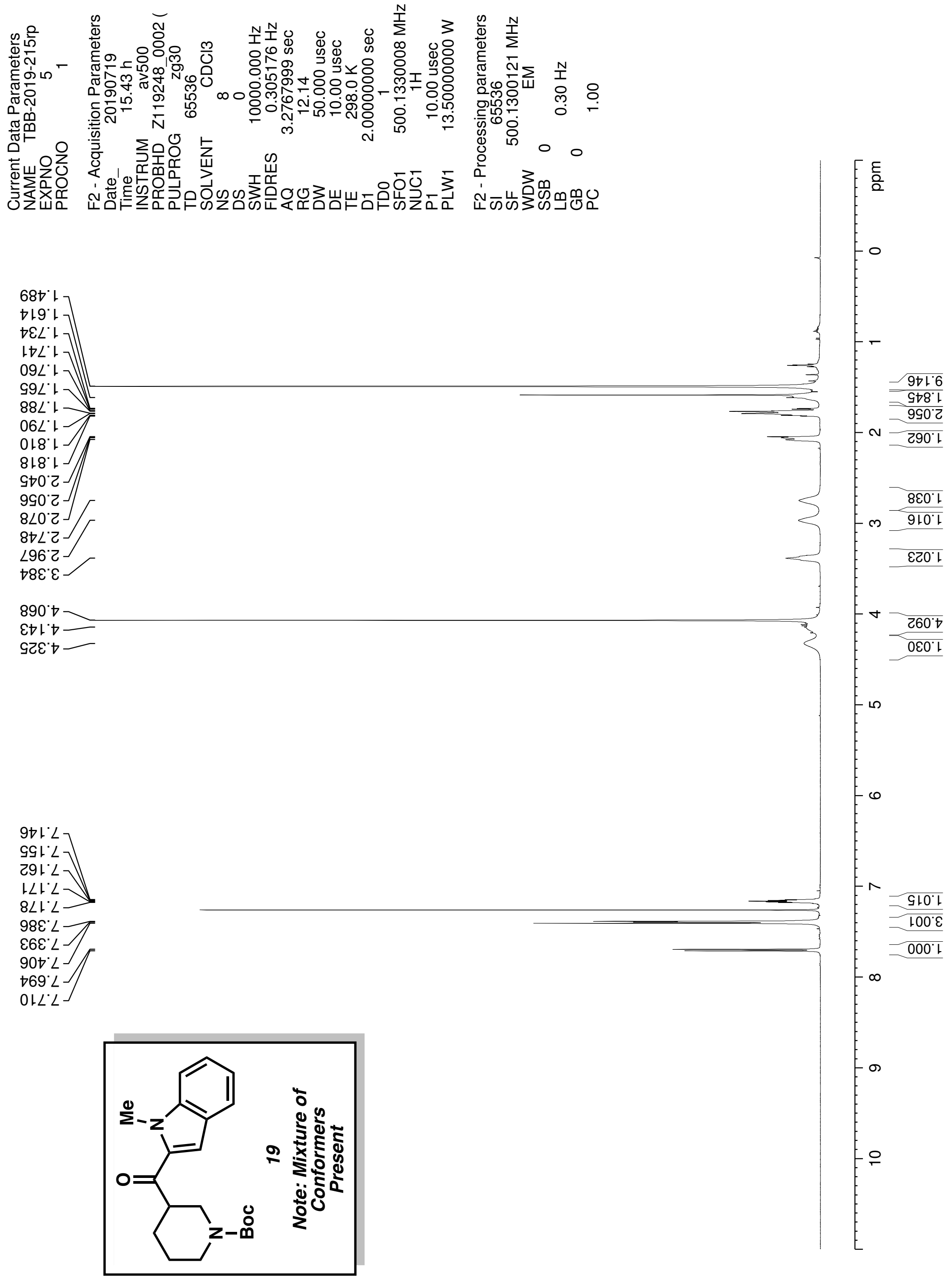

19

Note: Mixture of Conformers Present

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PULPROG         zg30
TD          65536
SOLVENT        CDCl3
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AQ         3.2767999 sec
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P1             10.00 usec
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F2 - Processing parameters
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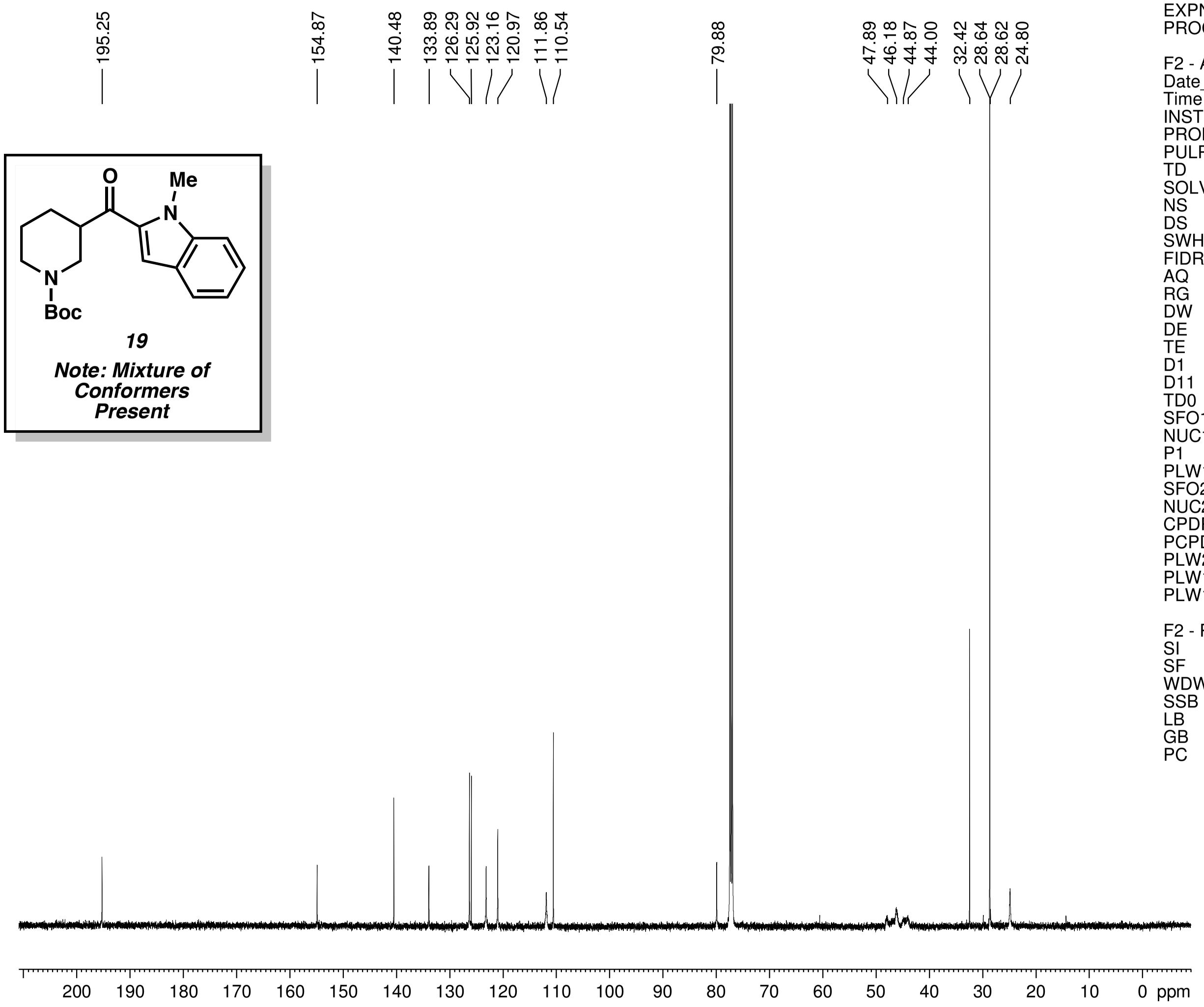
^{13}C NMR Spectra



19

Note: Mixture of Conformers Present

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