

**Nickel-Catalyzed Amination of Aryl Carbamates and Sulfamates
Using an Air-Stable Precatalyst**

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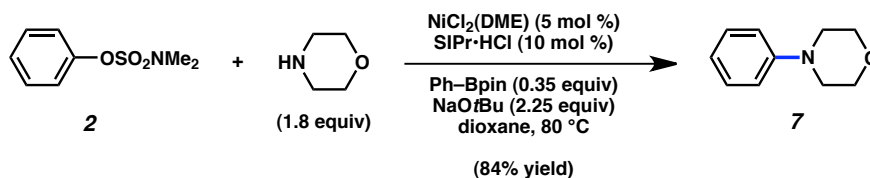
Materials and Methods. Unless stated otherwise, reactions were conducted in flame-dried glassware under an atmosphere of nitrogen using anhydrous solvents (either freshly distilled or passed through activated alumina columns). Unless otherwise stated, commercially obtained reagents were used as received. Amines were purified by filtration over basic Brockman Grade I 58 Å Al₂O₃ (Activity 1), followed by distillation over calcium hydride, prior to use. NiCl₂(DME) was obtained from Strem Chemicals. NaOtBu was obtained from Alfa Aesar. The amines, SIPr•HCl, and Ph–B(pin) were obtained from Sigma Aldrich and Alfa Aesar. Dioxane was purified by distillation over sodium benzophenone ketyl. Reaction temperatures were controlled using an IKAmag temperature modulator, and unless stated otherwise, reactions were performed at room temperature (rt, approximately 23 °C). Thin-layer chromatography (TLC) was conducted with EMD gel 60 F254 pre-coated plates (0.25 mm) and visualized using a combination of UV, anisaldehyde, ceric ammonium molybdate, iodine, vanillin, and potassium permanganate staining. Silicycle Siliaflash P60 (particle size 0.040–0.063 mm) was used for flash column chromatography. ¹H NMR spectra were recorded on Bruker spectrometers (at 300, 400, 500, 600 MHz) and are reported relative to deuterated solvent signals. Data for ¹H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration.

Experimental Procedures.

A. Synthesis of Aryl Carbamate and Sulfamate Substrates

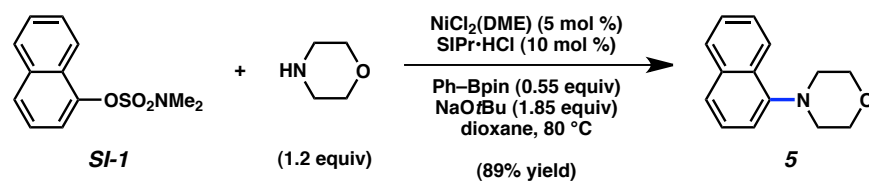
Note: Supporting information for the synthesis of the aryl sulfamates and carbamates shown in Table 1–2 and Figure 2–3 have previously been reported.¹

B. Aminations of Aryl Carbamates and Sulfamates

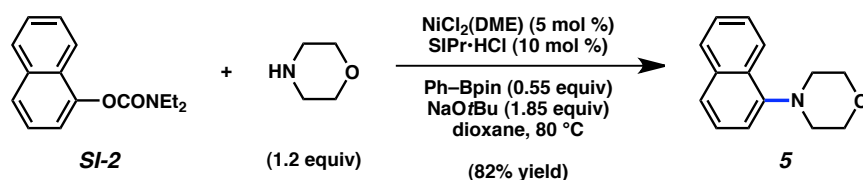


Representative Procedure (coupling of phenylsulfamate 2 is used as an example). 7 (Figure 2). A 4 mL reaction vial was charged with a magnetic stir bar, flame-dried under reduced pressure, and allowed to cool under N₂. The vial was then charged with Ph–B(pin) (35.71 mg, 0.175 mmol, 35 mol%), anhydrous powdered NaOtBu (108.1 mg, 1.125 mmol, 2.25 equiv), NiCl₂(DME) (5.5 mg, 0.025 mmol, 5 mol%), and SIPr•HCl (21.3 mg, 0.05 mmol, 10 mol%). Subsequently, dioxane (2.5 ml), phenylsulfamate 2 (100.6 mg, 0.50 mmol, 1.0 equiv), and morpholine (87.1 μL, 0.9 mmol, 1.8 equiv) were added, sequentially. The resulting heterogenous mixture was stirred for 1 min while purging with N₂, and the vial was sealed with a Teflon-lined screw cap. The mixture was stirred at 23 °C for 1 h, and then at 80 °C for 3 h in a preheated aluminum heating block. After cooling the reaction vessel to 23 °C and concentrating the mixture under reduced pressure, the crude residue was purified by flash chromatography (9:1 Hexanes:EtOAc) to yield aminated product 7 (68.3 mg, 84% yield) as a white solid. R_f 0.28 (9:1 Hexanes:EtOAc). Spectral data match those previously reported.²

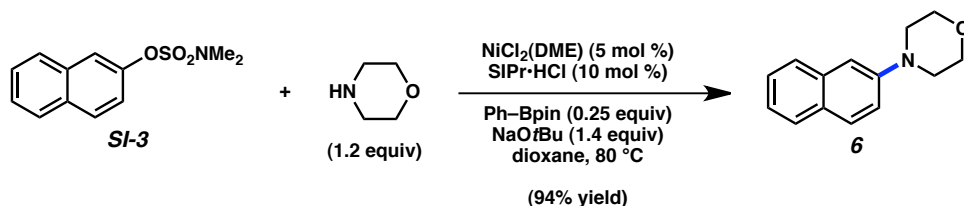
Any modifications of the conditions shown in this representative procedure are specified in the following schemes, which depict all of the results shown in Figures 2–3.



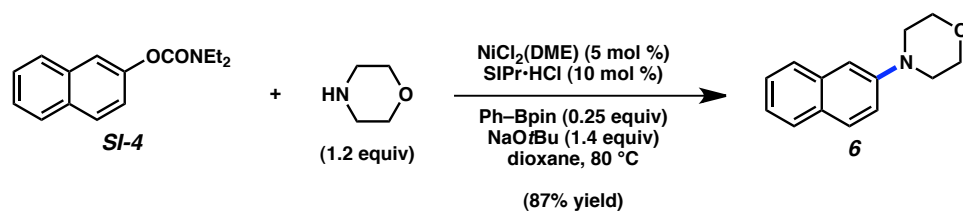
5 (Figure 2). Purification by flash chromatography (9:1 Hexanes:EtOAc) yielded aminated product **5** (89% yield) as a white solid. R_f 0.41 (9:1 Hexanes:EtOAc). Spectral data match those previously reported.³



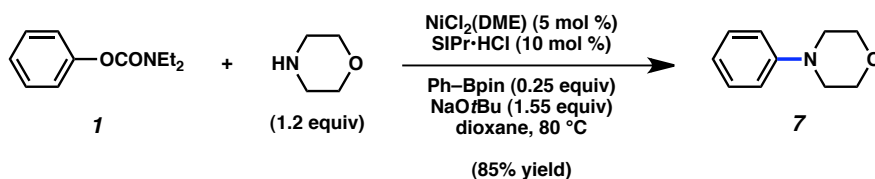
5 (Figure 2). Purification by flash chromatography (9:1 Hexanes:EtOAc) afforded aminated product **5** (82% yield) as a white solid. R_f 0.41 (9:1 Hexanes:EtOAc). Spectral data match those previously reported.³



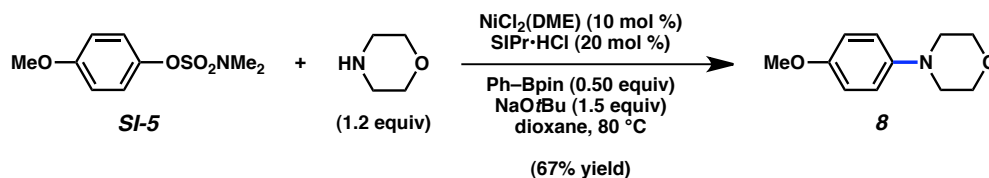
6 (Figure 2). Purification by flash chromatography (9:1 Hexanes:EtOAc) produced aminated product **6** (94% yield) as a white solid. R_f 0.23 (9:1 Hexanes:EtOAc). Spectral data match those previously reported.⁴



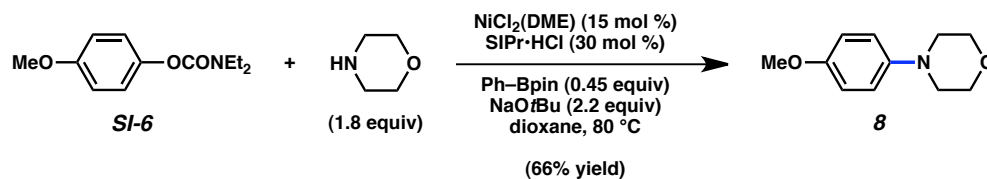
6 (Figure 2). Purification by flash chromatography (9:1 Hexanes:EtOAc) generated aminated product **6** (87% yield) as a white solid. R_f 0.23 (9:1 Hexanes:EtOAc). Spectral data match those previously reported.⁴



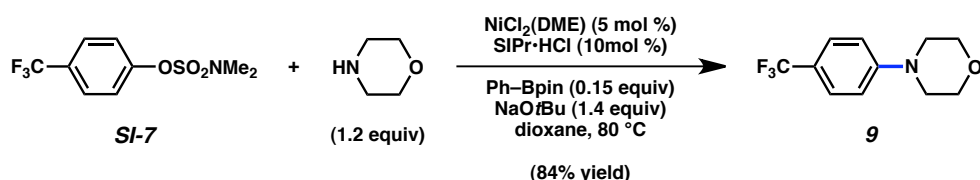
7 (Figure 2). Purification by flash chromatography (9:1 Hexanes:EtOAc) supplied aminated product **7** (85% yield) as a white solid. R_f 0.28 (9:1 Hexanes:EtOAc). Spectral data match those previously reported.²



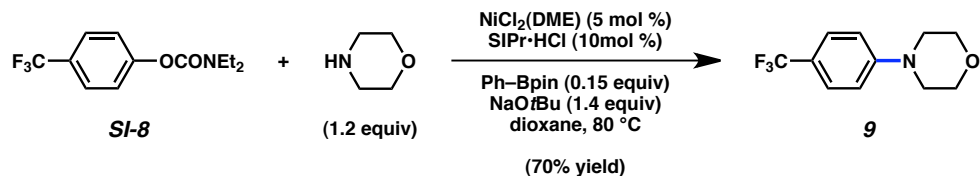
8 (Figure 2). Purification by flash chromatography (10:1:1 Benzene:Et₂O:CH₂Cl₂) afforded aminated product **8** (67% yield) as a white solid. R_f 0.16 (10:1:1 Benzene:Et₂O:CH₂Cl₂). Spectral data match those previously reported.⁵



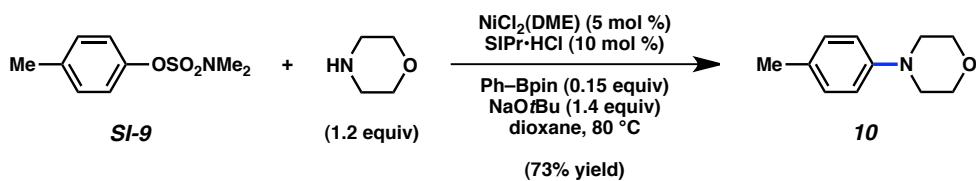
8 (Figure 2). Purification by flash chromatography (10:1:1 Benzene:Et₂O:CH₂Cl₂) afforded aminated product **8** (66% yield) as a white solid. *R_f* 0.16 (10:1:1 Benzene:Et₂O:CH₂Cl₂). Spectral data match those previously reported.⁵



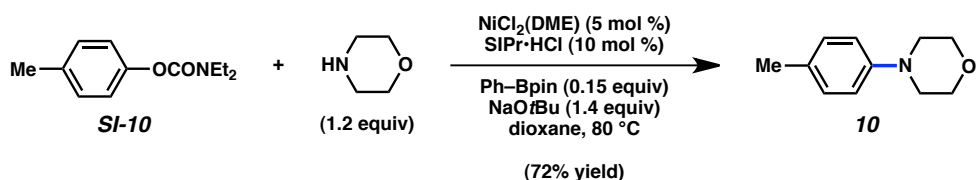
9 (Figure 2). Purification by flash chromatography (30:1 Benzene:Et₂O) generated aminated product **9** (84% yield) as a white solid. *R_f* 0.38 (30:1 Benzene:Et₂O). Spectral data match those previously reported.⁶



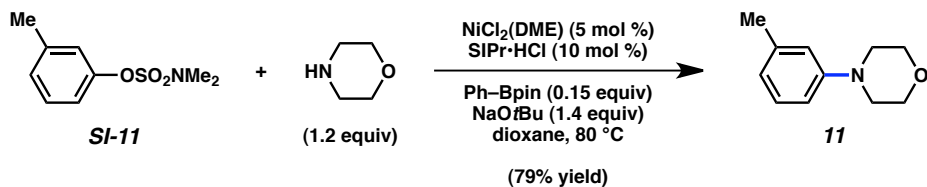
9 (Figure 2). Purification by flash chromatography (30:1 Benzene:Et₂O) produced aminated product **9** (70% yield) as a white solid. *R_f* 0.38 (30:1 Benzene:Et₂O). Spectral data match those previously reported.⁶



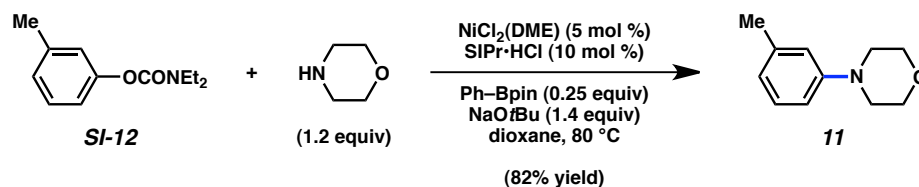
10 (Figure 2). Purification by flash chromatography (19:1 Benzene:Et₂O) afforded aminated product **10** (73% yield) as a white solid. *R_f* 0.29 (19:1 Benzene:Et₂O). Spectral data match those previously reported.⁷



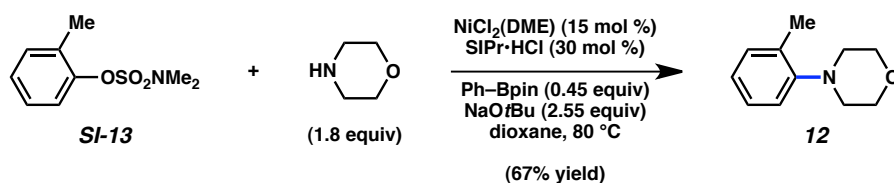
10 (Figure 2). Purification by flash chromatography (19:1 Benzene:Et₂O) yielded aminated product **10** (72% yield) as a white solid. *R_f* 0.29 (19:1 Benzene:Et₂O). Spectral data match those previously reported.⁷



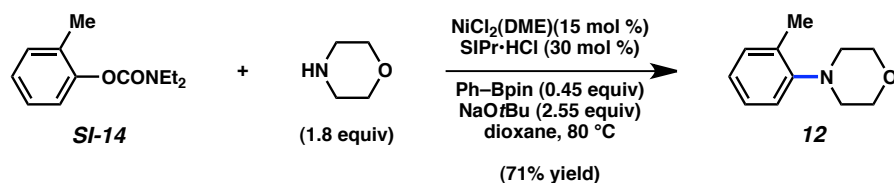
11 (Figure 2). Purification by flash chromatography (19:1 Benzene:Et₂O) generated aminated product **11** (79% yield) as a yellow oil. *R_f* 0.34 (19:1 Benzene:Et₂O). Spectral data match those previously reported.⁷



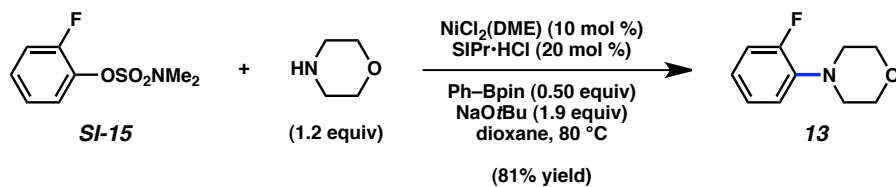
11 (Figure 2). Purification by flash chromatography (19:1 Benzene:Et₂O) afforded aminated product **11** (82% yield) as a yellow oil. *R_f* 0.34 (19:1 Benzene:Et₂O). Spectral data match those previously reported.⁷



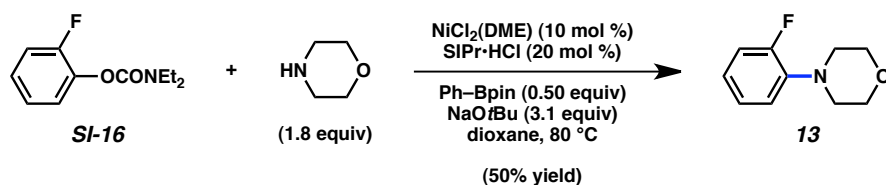
12 (Figure 2). Purification by flash chromatography (20:1 Hexanes:EtOAc) produced aminated product **12** (67% yield) as a yellow oil. *R_f* 0.30 (20:1 Hexanes:EtOAc). Spectral data match those previously reported.⁷



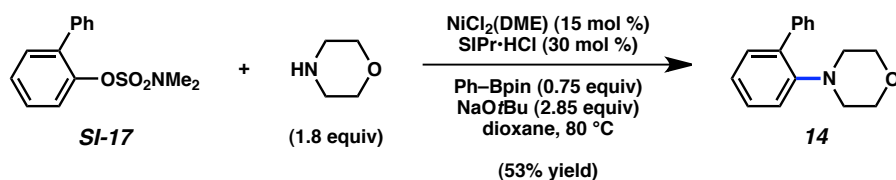
12 (Figure 2). Purification by flash chromatography (20:1 Hexanes:EtOAc) supplied aminated product **12** (71% yield) as a yellow oil. *R_f* 0.30 (20:1 Hexanes:EtOAc). Spectral data match those previously reported.⁷



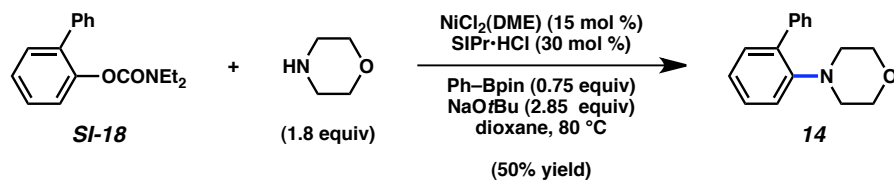
13 (Figure 2). Purification by flash chromatography (60:1:1 Benzene:Et₂O:CH₂Cl₂) afforded aminated product **13** (81% yield) as an off-white solid. *R_f* 0.45 (60:1:1 Benzene:Et₂O:CH₂Cl₂). Spectral data match those previously reported.⁹



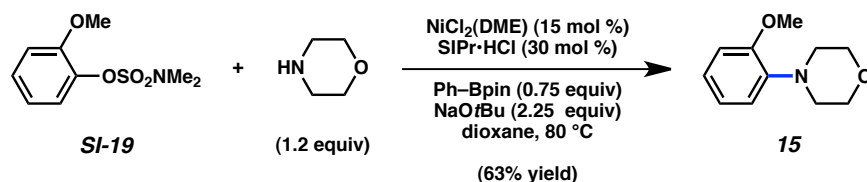
13 (Figure 2). Purification by flash chromatography (60:1:1 Benzene:Et₂O:CH₂Cl₂) supplied aminated product **13** (50% yield) as an off-white solid. *R_f* 0.45 (60:1:1 Benzene:Et₂O:CH₂Cl₂). Spectral data match those previously reported.⁹



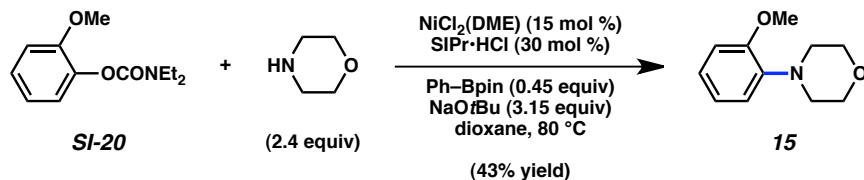
14 (Figure 2). Purification by flash chromatography (100% Benzene) yielded aminated product **14** (53% yield) as a yellow oil. *R_f* 0.50 (100% Benzene). Spectral data match those previously reported.⁸



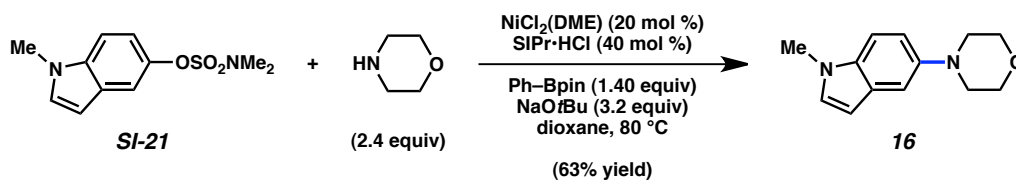
14 (Figure 2). Purification by flash chromatography (100% Benzene) afforded aminated product **14** (50% yield) as a yellow oil. R_f 0.50 (100% Benzene). Spectral data match those previously reported.⁸



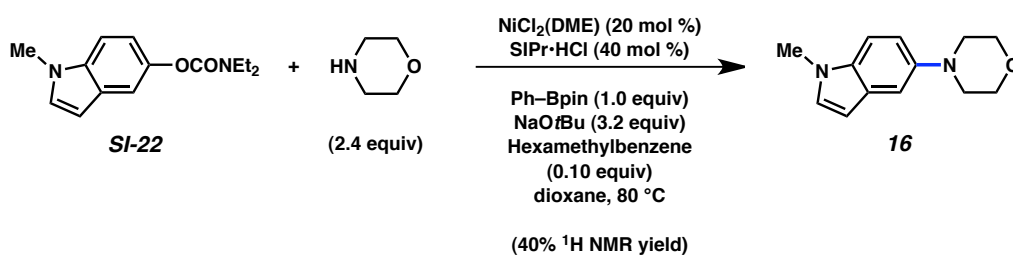
15 (Figure 2). Purification by flash chromatography (20:1 Hexanes:EtOAc) supplied aminated product **15** (63% yield) as a yellow oil. R_f 0.26 (20:1 Hexanes:EtOAc). Spectral data match those previously reported.⁹



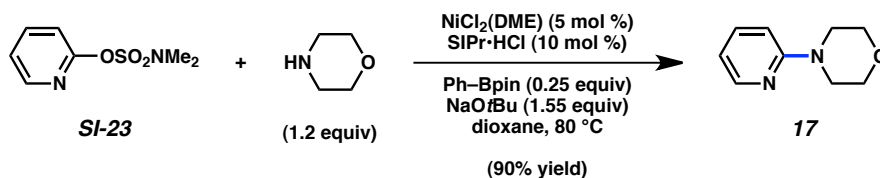
15 (Figure 2). Purification by flash chromatography (9:1 Benzene:Et₂O) generated aminated product **15** (43% yield) as a yellow oil. R_f 0.27 (9:1 Benzene:Et₂O). Spectral data match those previously reported.⁹



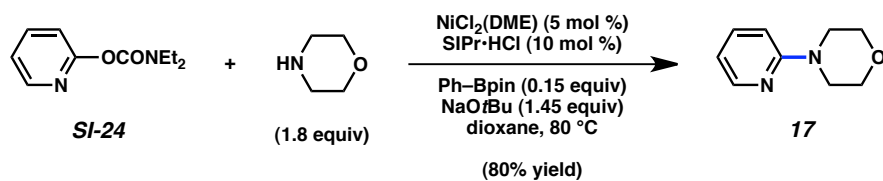
16 (Figure 2). Purification by flash chromatography (6:1:1 Benzene:Et₂O:CH₂Cl₂) yielded aminated product **16** (63% yield) as an off-white solid. *R_f* 0.36 (6:1:1 Benzene:Et₂O:CH₂Cl₂). Spectral data match those previously reported.^{1h}



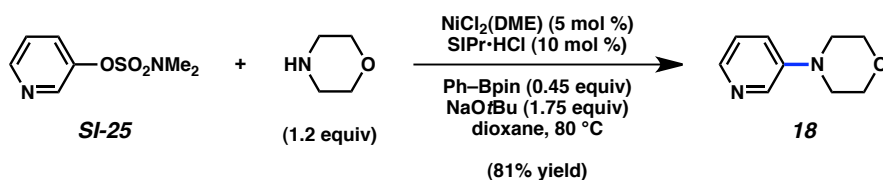
16 (Figure 2). The reaction mixture was filtered over a short plug of silica gel (eluted with EtOAc (10 mL)), then volatiles were removed in in vacuo and evaporated to dryness. The yield was determined by ¹H NMR analysis with Hexamethylbenzene as an internal standard.



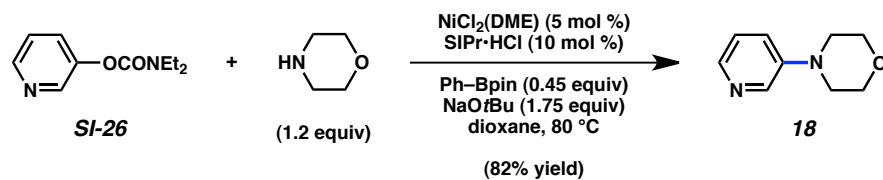
17 (Figure 2). Purification by flash chromatography (2:1 Hexanes:EtOAc) afforded aminated product **17** (90% yield) as a pale yellow oil. *R_f* 0.23 (2:1 Hexanes:EtOAc). Spectral data match those previously reported.¹⁰



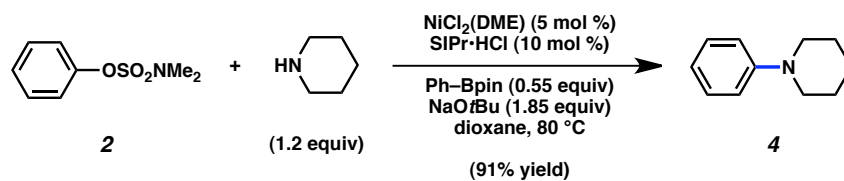
17 (Figure 2). Purification by flash chromatography (2:1 Hexanes:EtOAc) produced aminated product **17** (80% yield) as a pale yellow oil. R_f 0.27 (2:1 Hexanes:EtOAc). Spectral data match those previously reported.¹⁰



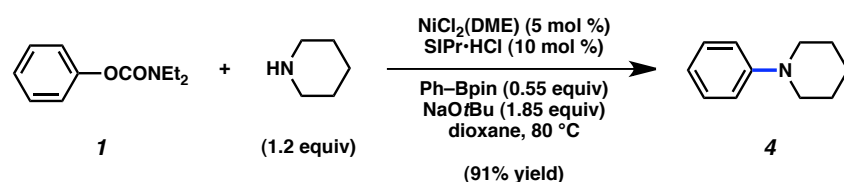
18 (Figure 2). Purification by flash chromatography (100% EtOAc) afforded aminated product **18** (81% yield) as a pale yellow oil. R_f 0.14 (100% EtOAc). Spectral data match those previously reported.¹⁰



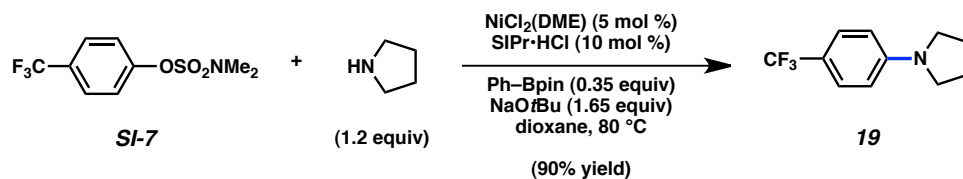
18 (Figure 2). Purification by flash chromatography (100% EtOAc) generated aminated product **18** (82% yield) as a pale yellow oil. R_f 0.14 (100% EtOAc). Spectral data match those previously reported.¹⁰



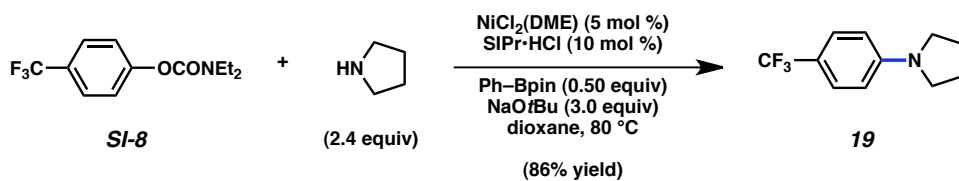
4 (Figure 3). Purification by flash chromatography (50:1 Hexanes:EtOAc) afforded aminated product **4** (91% yield) as a clear oil. R_f 0.39 (50:1 Hexanes:EtOAc). Spectral data match those previously reported.¹⁰



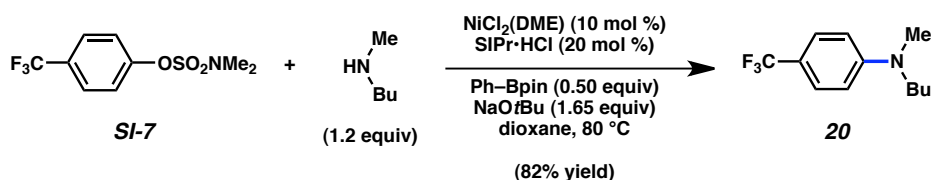
4 (Figure 3). Purification by flash chromatography (50:1 Hexanes:EtOAc) supplied aminated product **4** (91% yield) as a clear oil. R_f 0.39 (50:1 Hexanes:EtOAc). Spectral data match those previously reported.¹¹



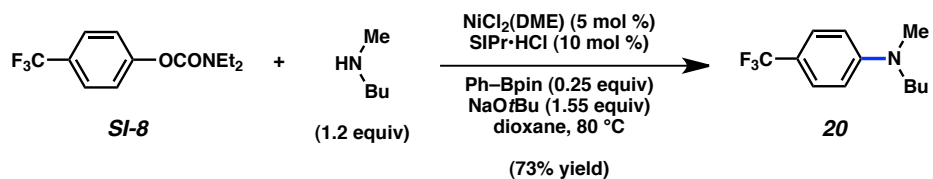
19 (Figure 3). Purification by flash chromatography (50:1 Hexanes:Et₂O) generated aminated product **19** (90% yield) as a white solid. R_f 0.34 (50:1 Hexanes:Et₂O). Spectral data match those previously reported.¹²



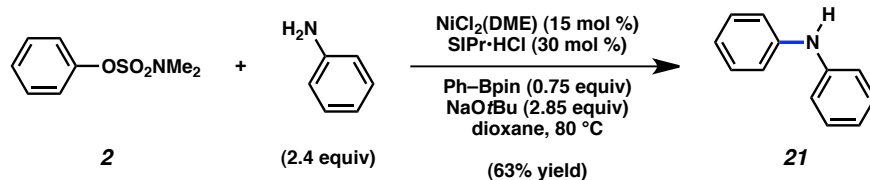
19 (Figure 3). Purification by flash chromatography (50:1 Hexanes:Et₂O) produced aminated product **19** (86% yield) as a white solid. *R_f* 0.34 (50:1 Hexanes:Et₂O). Spectral data match those previously reported.¹¹



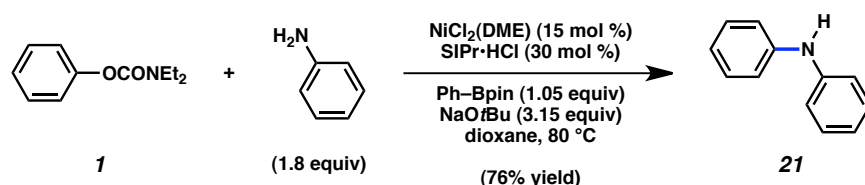
20 (Figure 3). Purification by flash chromatography (90:1 Hexanes:Et₂O) afforded aminated product **20** (82% yield) as a clear oil. *R_f* 0.37 (90:1 Hexanes:Et₂O). Spectral data match those previously reported.^{1h}



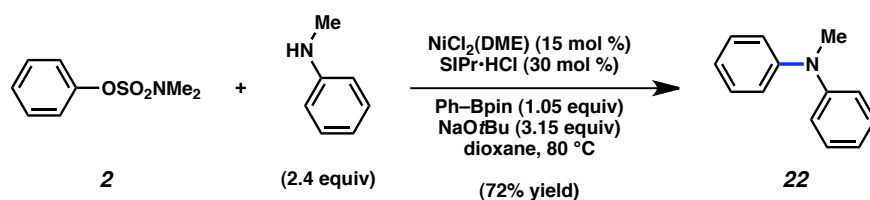
20 (Figure 3). Purification by flash chromatography (90:1 Hexanes:Et₂O) generated aminated product **20** (73% yield) as a clear oil. *R_f* 0.37 (90:1 Hexanes:Et₂O). Spectral data match those previously reported.^{1h}



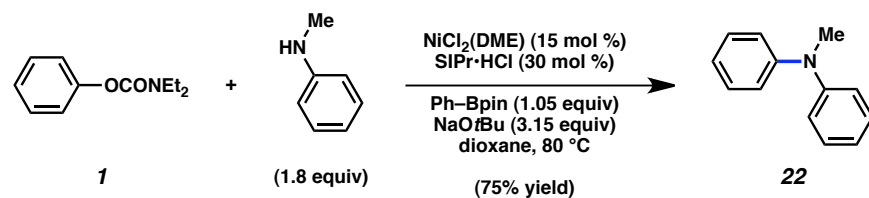
21 (Figure 3). Purification by flash chromatography (4:1 Hexanes: CH_2Cl_2) afforded aminated product **21** (63% yield) as a yellow solid. R_f 0.20 (4:1 Hexanes: CH_2Cl_2). Spectral data match those previously reported.⁷



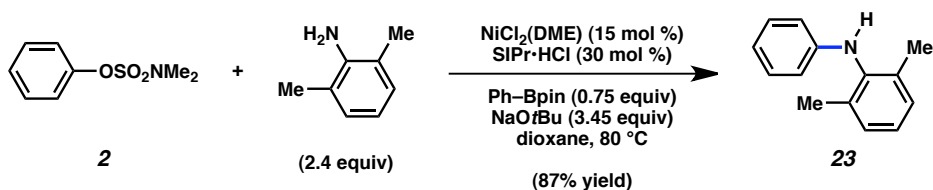
21 (Figure 3). Purification by flash chromatography (4:1 Hexanes: CH_2Cl_2) yielded aminated product **21** (76% yield) as a yellow solid. R_f 0.20 (4:1 Hexanes: CH_2Cl_2). Spectral data match those previously reported.⁷



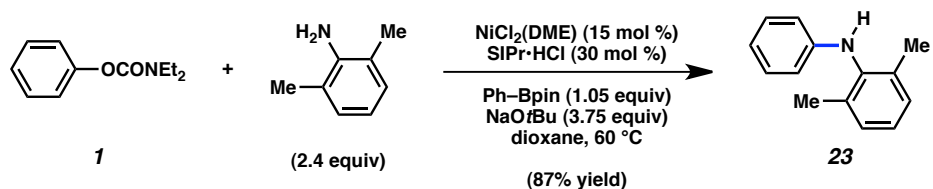
22 (Figure 3). Purification by flash chromatography (100% Hexanes) afforded aminated product **22** (72% yield) as a yellow oil. R_f 0.15 (100% Hexanes). Spectral data match those previously reported.⁷



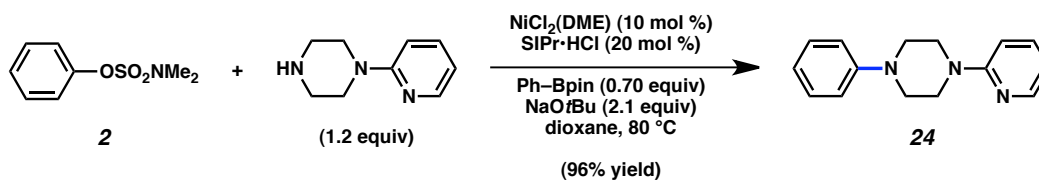
22 (Figure 3). Purification by flash chromatography (100% Hexanes) generated aminated product **22** (75% yield) as a yellow oil. R_f 0.15 (100% Hexanes). Spectral data match those previously reported.⁷



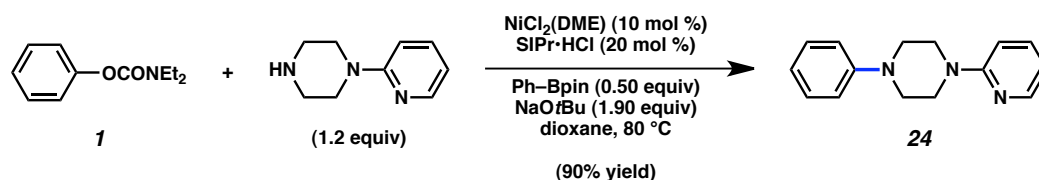
23 (Figure 3). Purification by flash chromatography (20:1 Hexanes:Et₂O) yielded aminated product **23** (87% yield) as a clear oil. R_f 0.45 (40:1 Hexanes:Et₂O). Spectral data match those previously reported.⁷



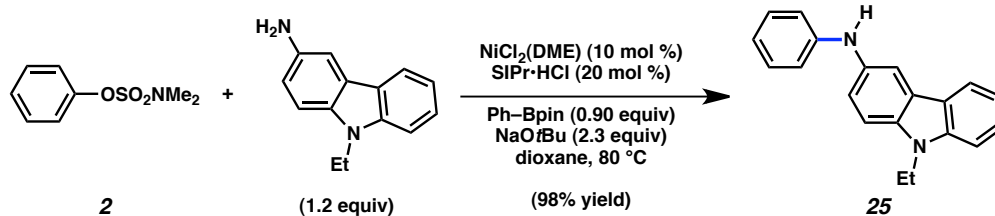
23 (Figure 3). Purification by flash chromatography (20:1 Hexanes:Et₂O) produced aminated product **23** (87% yield) as a clear oil. R_f 0.45 (40:1 Hexanes:Et₂O). Spectral data match those previously reported.⁷



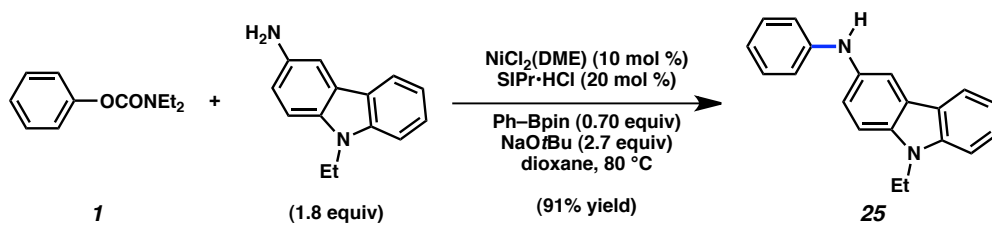
24 (Figure 3). Purification by flash chromatography (8:1 Hexanes:EtOAc) supplied aminated product **24** (96% yield) as a white solid. R_f 0.18 (8:1 Hexanes:EtOAc). Spectral data match those previously reported.¹⁰



24 (Figure 3). Purification by flash chromatography (8:1 Hexanes:EtOAc) afforded aminated product **24** (90% yield) as a white solid. R_f 0.18 (8:1 Hexanes:EtOAc). Spectral data match those previously reported.¹⁰



25 (Figure 3). Purification by flash chromatography (300:150:1 Hexanes:CH₂Cl₂:Et₃N) yielded aminated product **25** (98% yield) as a white solid. R_f 0.19 (300:150:1 Hexanes:CH₂Cl₂:Et₃N). Spectral data match those previously reported.¹¹



25 (Figure 3). Purification by flash chromatography (300:150:1 Hexanes: CH_2Cl_2 : Et_3N) afforded aminated product **25** (91% yield) as a white solid. R_f 0.19 (300:150:1 Hexanes: CH_2Cl_2 : Et_3N). Spectral data match those previously reported.¹¹

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² Barker, T. J.; Jarvo, E. R. *J. Am. Chem. Soc.* **2009**, *131*, 15598–15599.

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⁷ Desmarests, C.; Schneider, R.; Fort, Y. *J. Org. Chem.* **2002**, *67*, 3029–3036.

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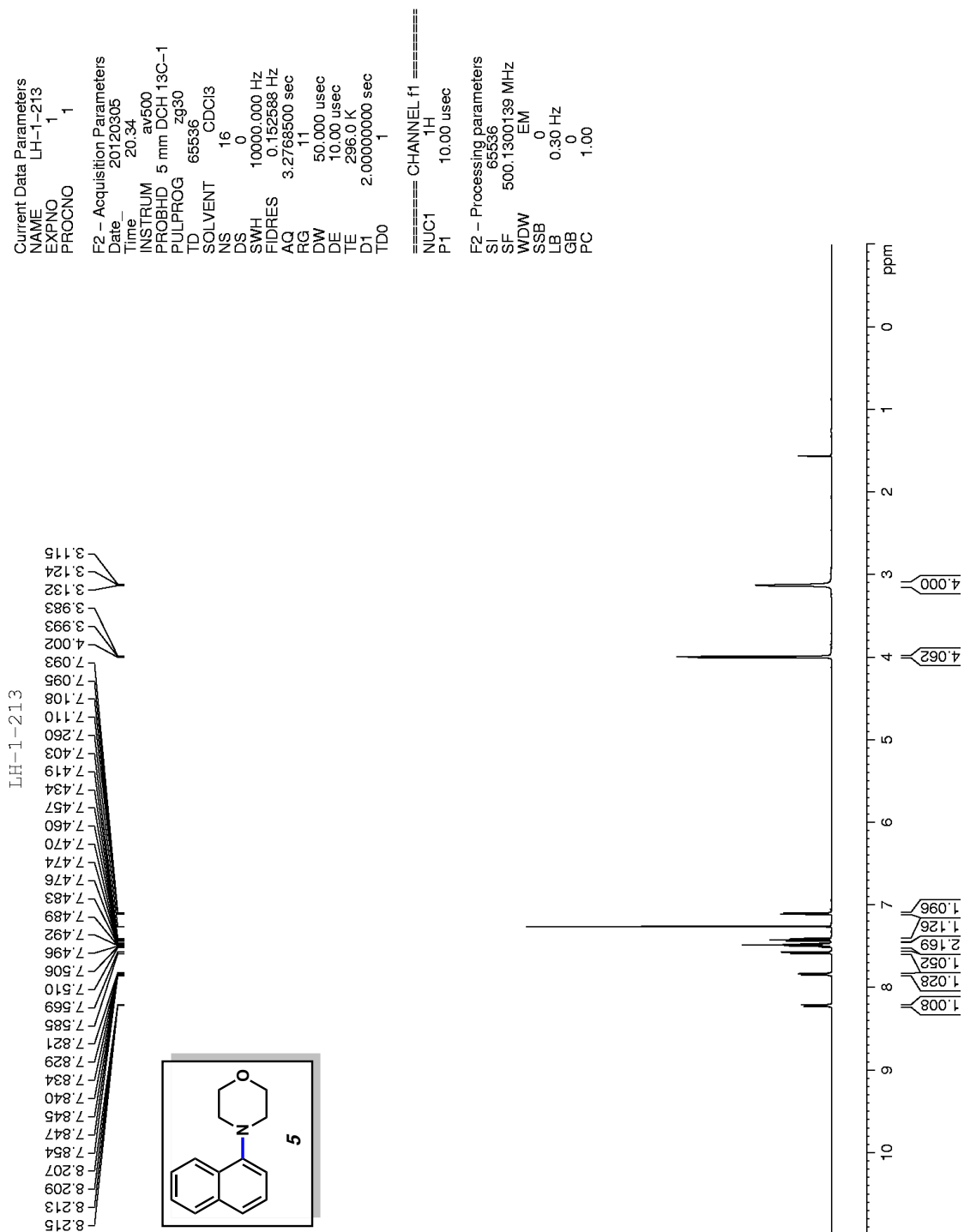
⁹ Fasani, E.; Tilocca, F.; Protti, S.; Merli, D.; Albini, A. *Org. Biomol. Chem.* **2008**, *6*, 4634–4642.

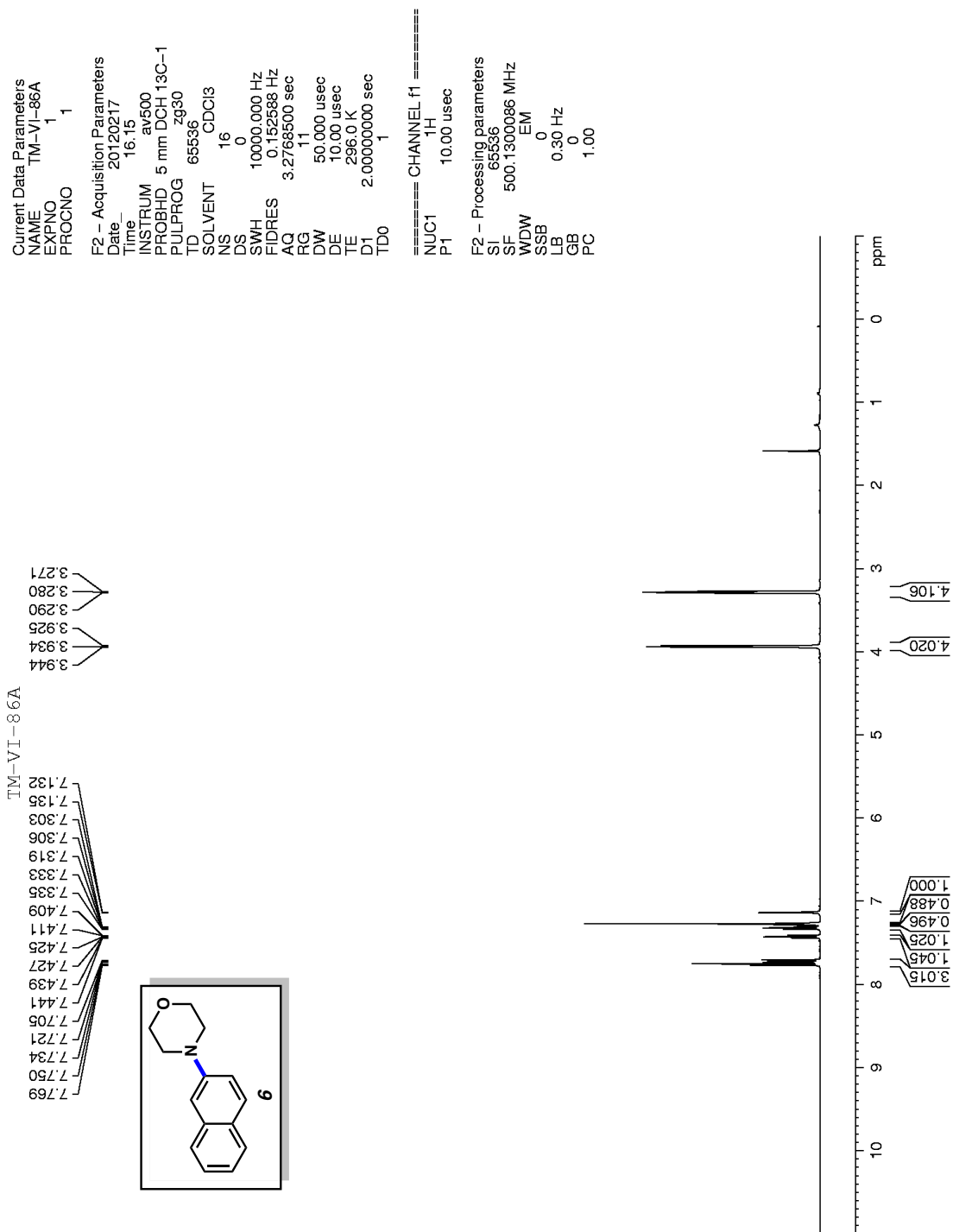
¹⁰ Wagaw, S.; Buchwald, S. L. *J. Org. Chem.* **1996**, *61*, 7240–7241.

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NMR Spectra





Current Data Parameters
 NAME LH-1-226
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120309
 Time_ 16.33
 INSTRUM av500
 PROBHID 5 mm DCH 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 1000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2768500 sec
 RG 11
 DW 50.000 usec
 DE 10.00 usec
 TE 296.0 K
 D1 2.00000000 sec
 TD0 1

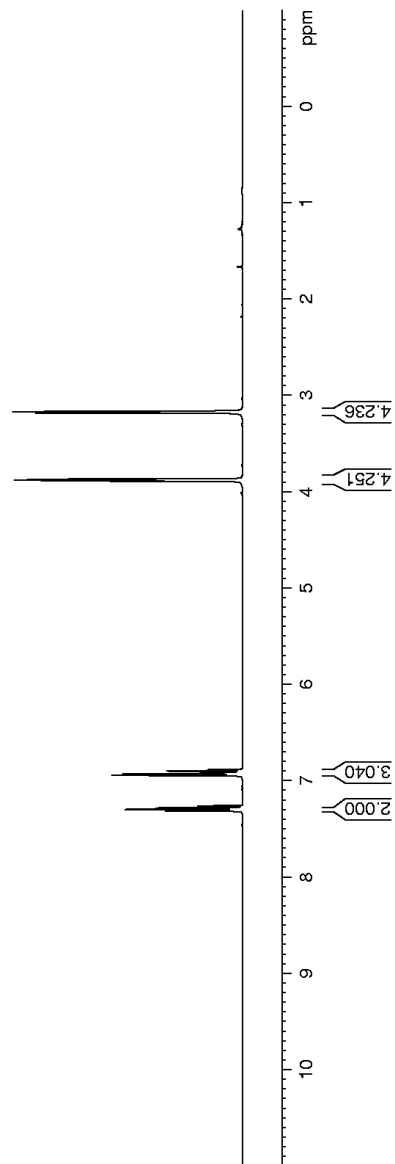
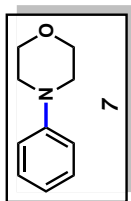
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 NUC1 1H
 P1 10.00 usec

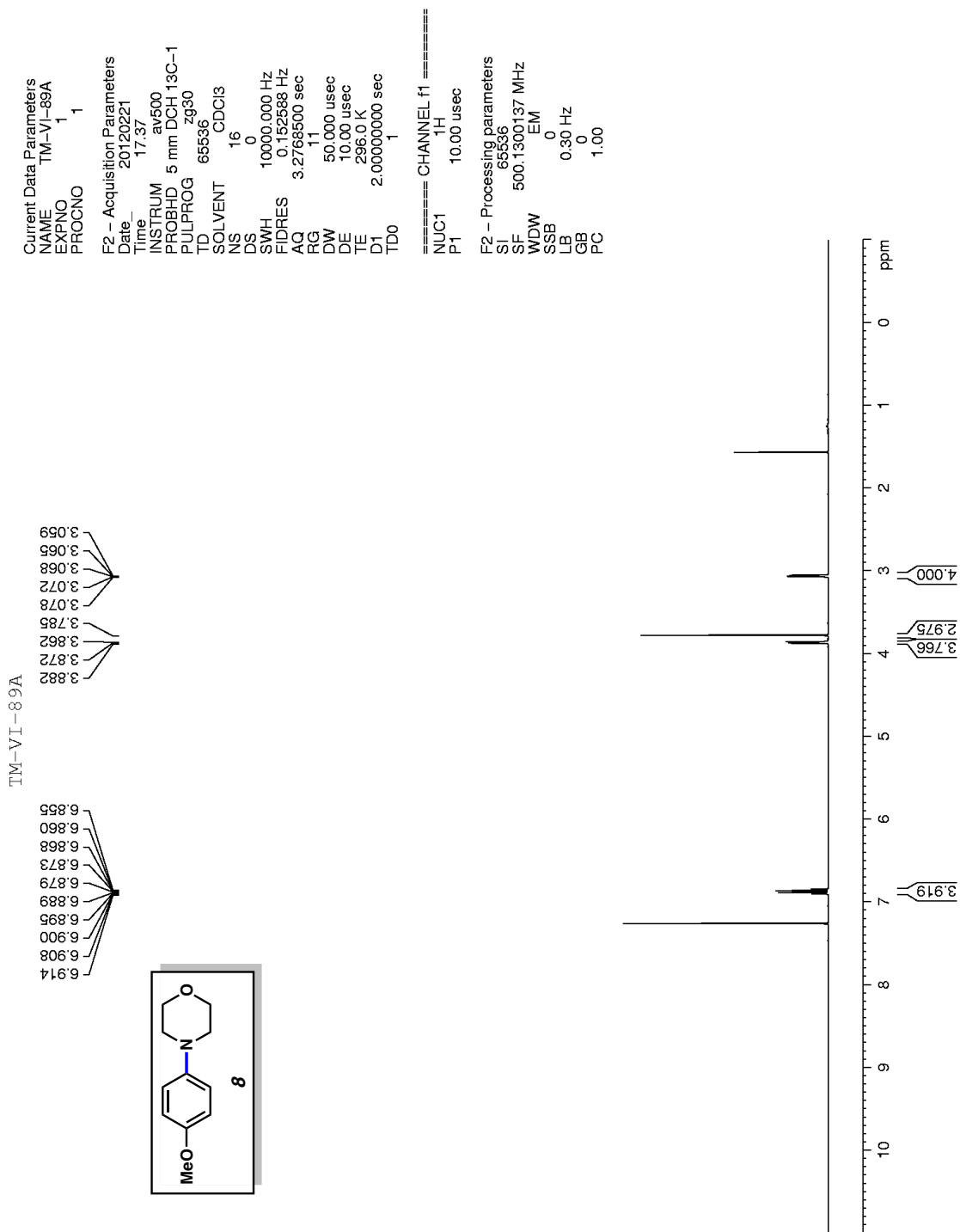
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 SF 500.1300135 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

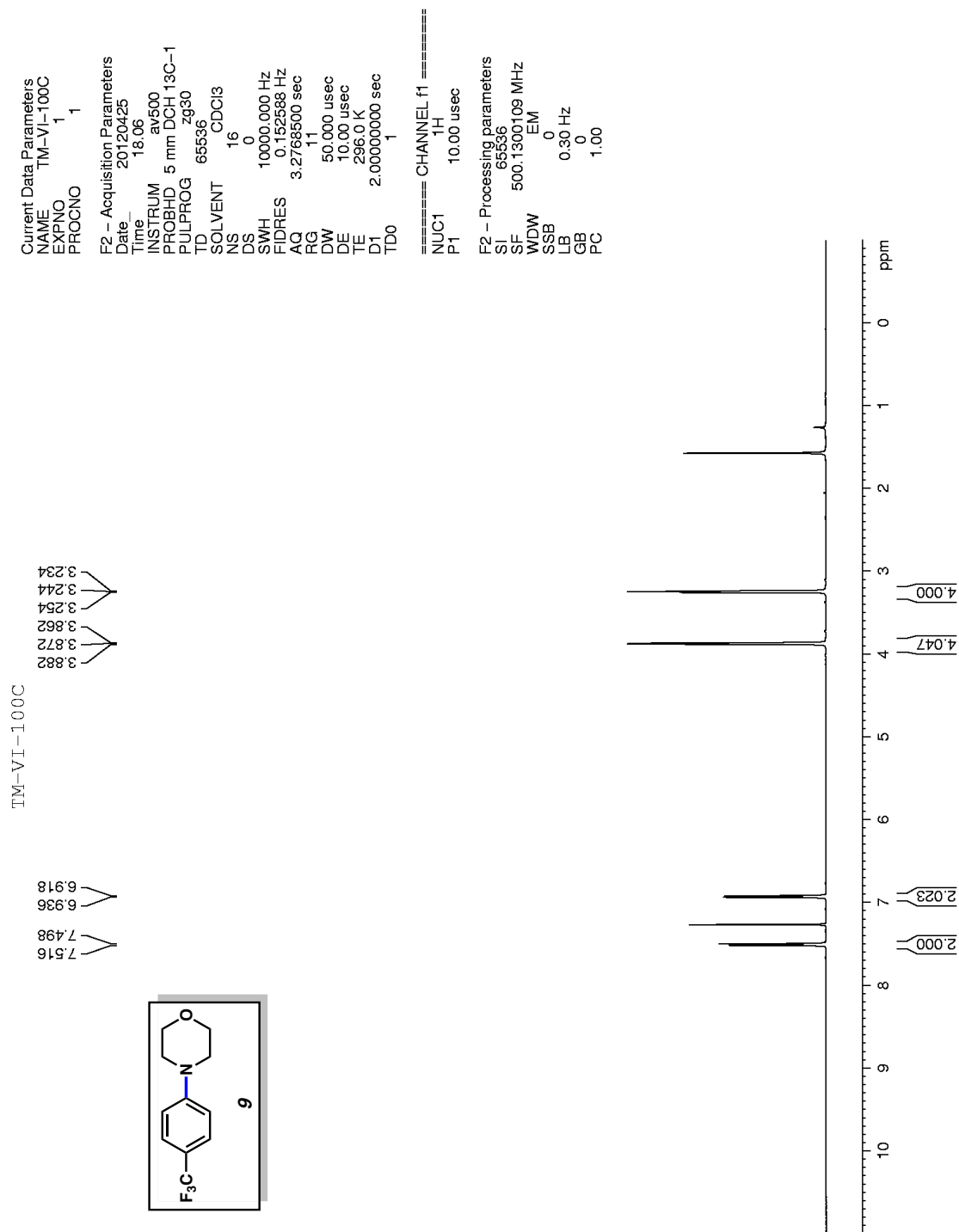
LH-1-226

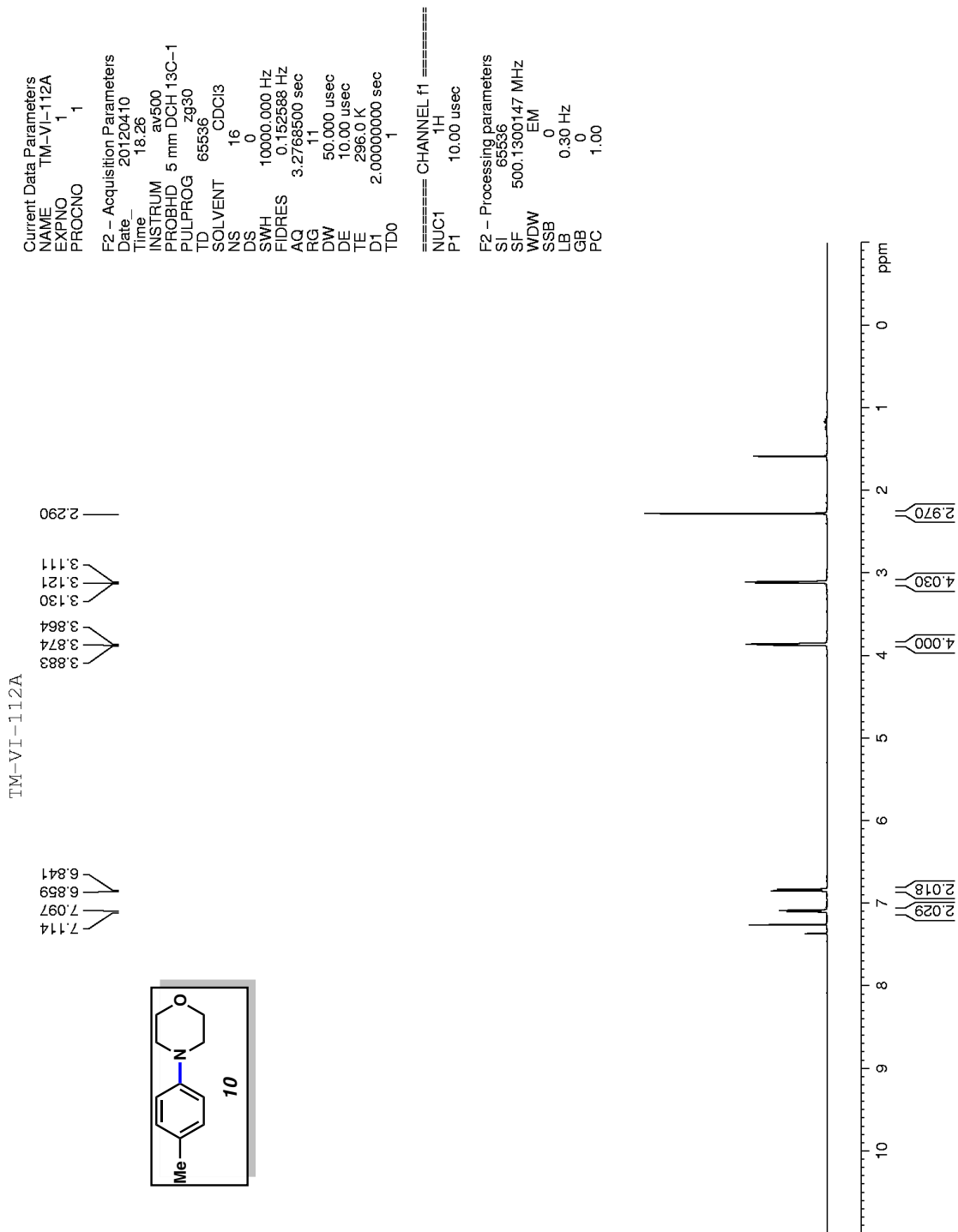
3.885
 3.875
 3.866
 3.178
 3.169
 3.159

7.312
 7.297
 7.295
 7.280
 7.260
 6.941
 6.925
 6.913
 6.899
 6.884









Current Data Parameters
 NAME TM-VI-74A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120208
 Time_ 17.18
 INSTRUM dx500
 PROBHD 5 mm bb-Z800
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 1000.000 Hz
 FIDRES 0.152568 Hz
 AQ 3.2769001 sec
 RG 228.1
 DW 50.000 usec
 DE 6.00 usec
 TE 297.6 K
 D1 2.0000000 sec
 TD0 1

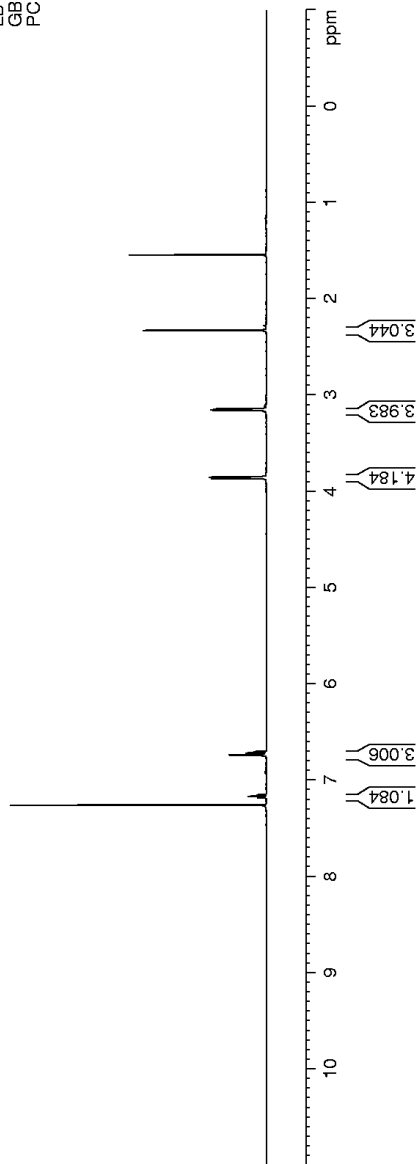
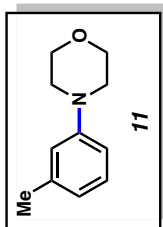
===== CHANNEL f1 =====
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 P1 12.25 usec
 PL1 0.00 dB
 SFO1 500.3330020 MHz

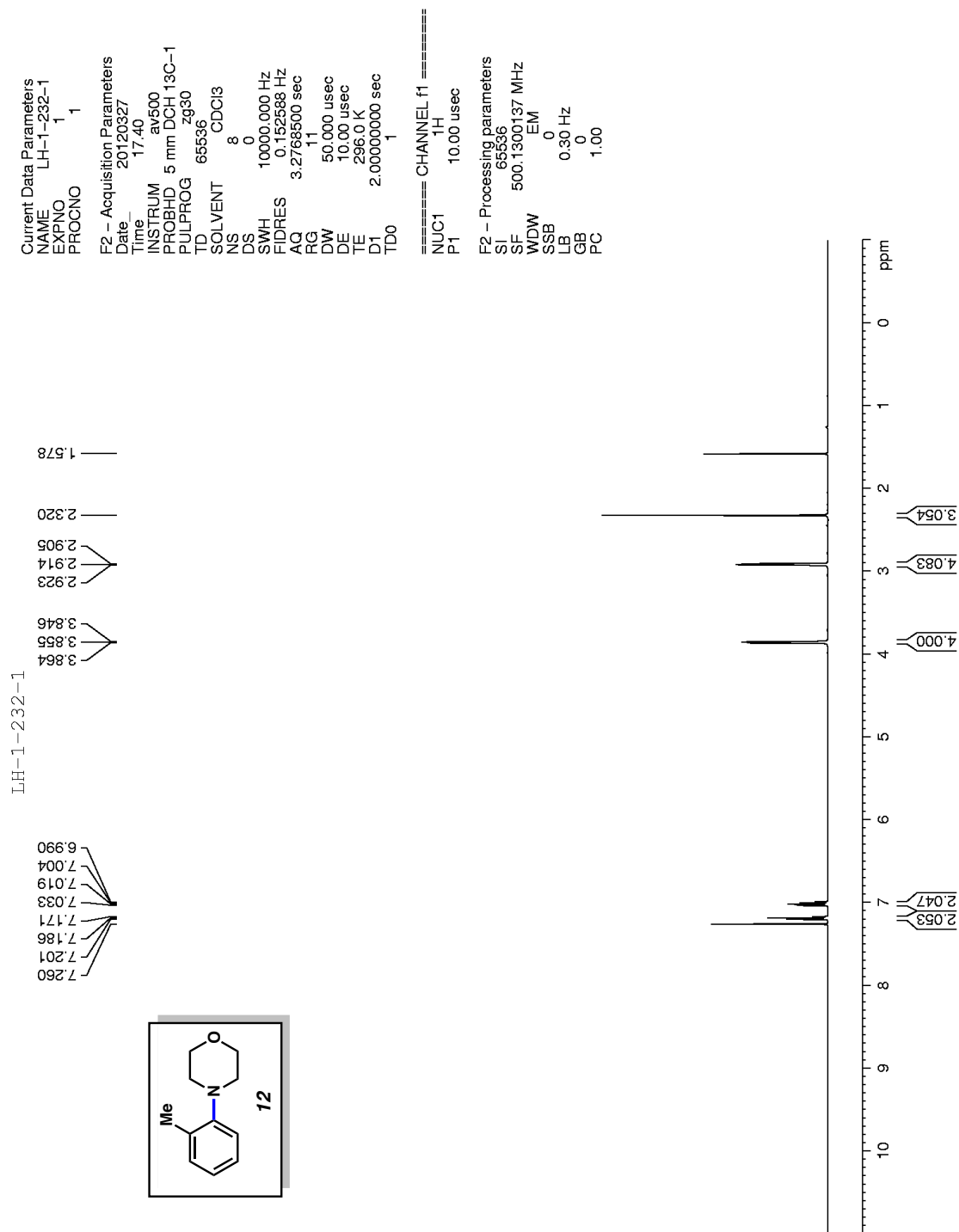
F2 - Processing parameters
 SI 32768
 SF 500.3300231 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

TM-VI-74A

1.541
 2.324
 3.138
 3.147
 3.157
 3.846
 3.856
 3.865

6.703
 6.718
 6.738
 7.151
 7.166
 7.182
 7.258



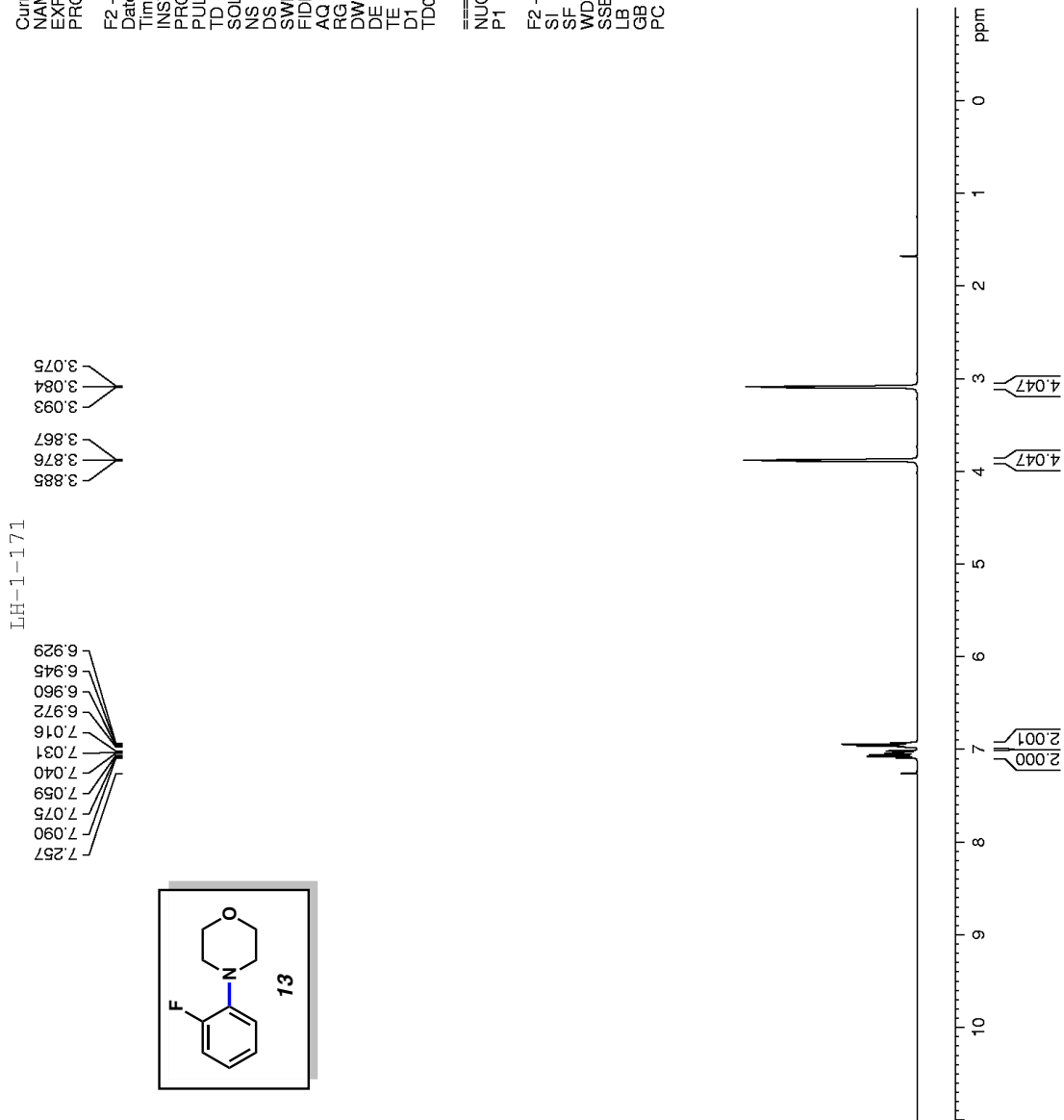


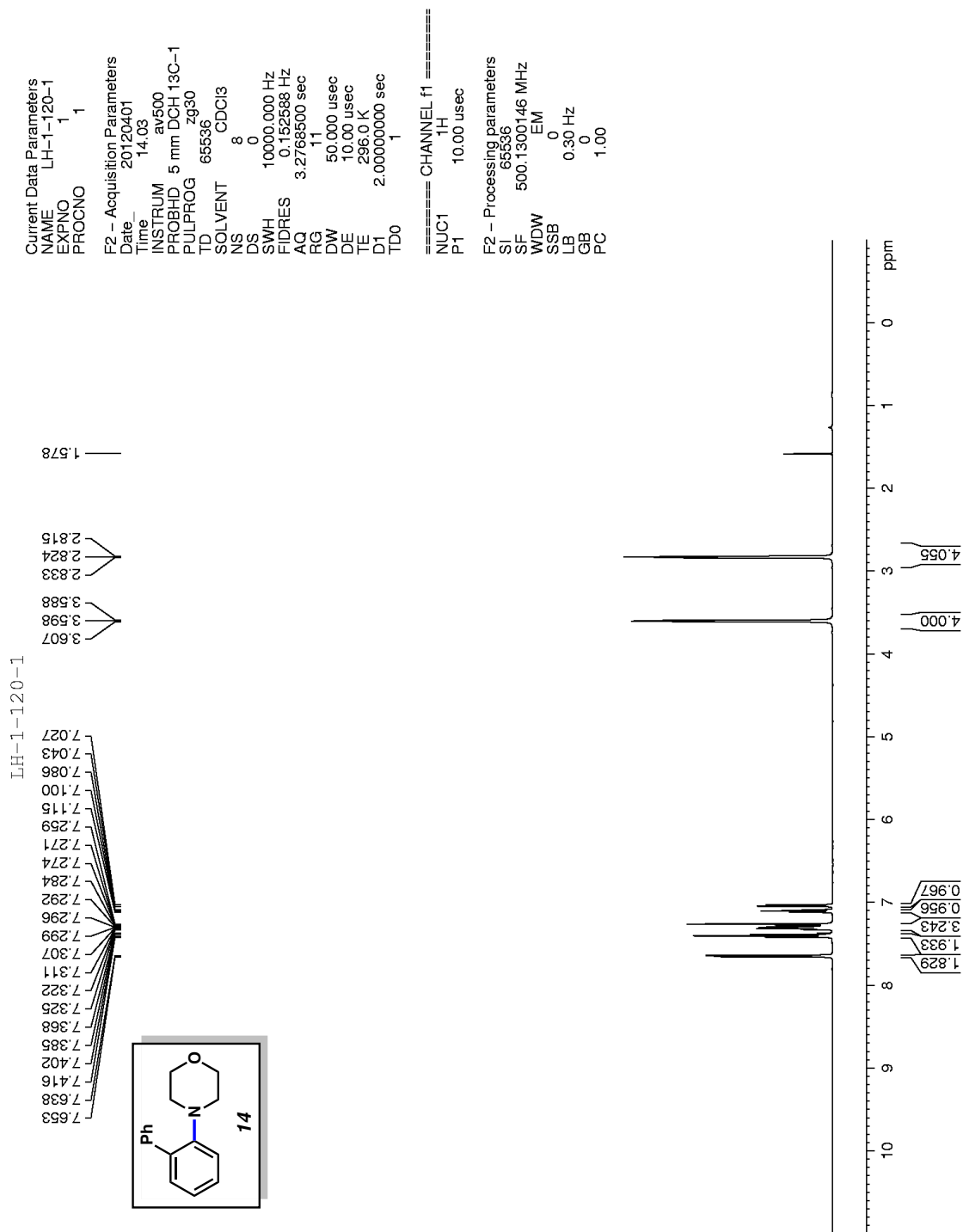
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 EXPNO 1
 PROCNO 1

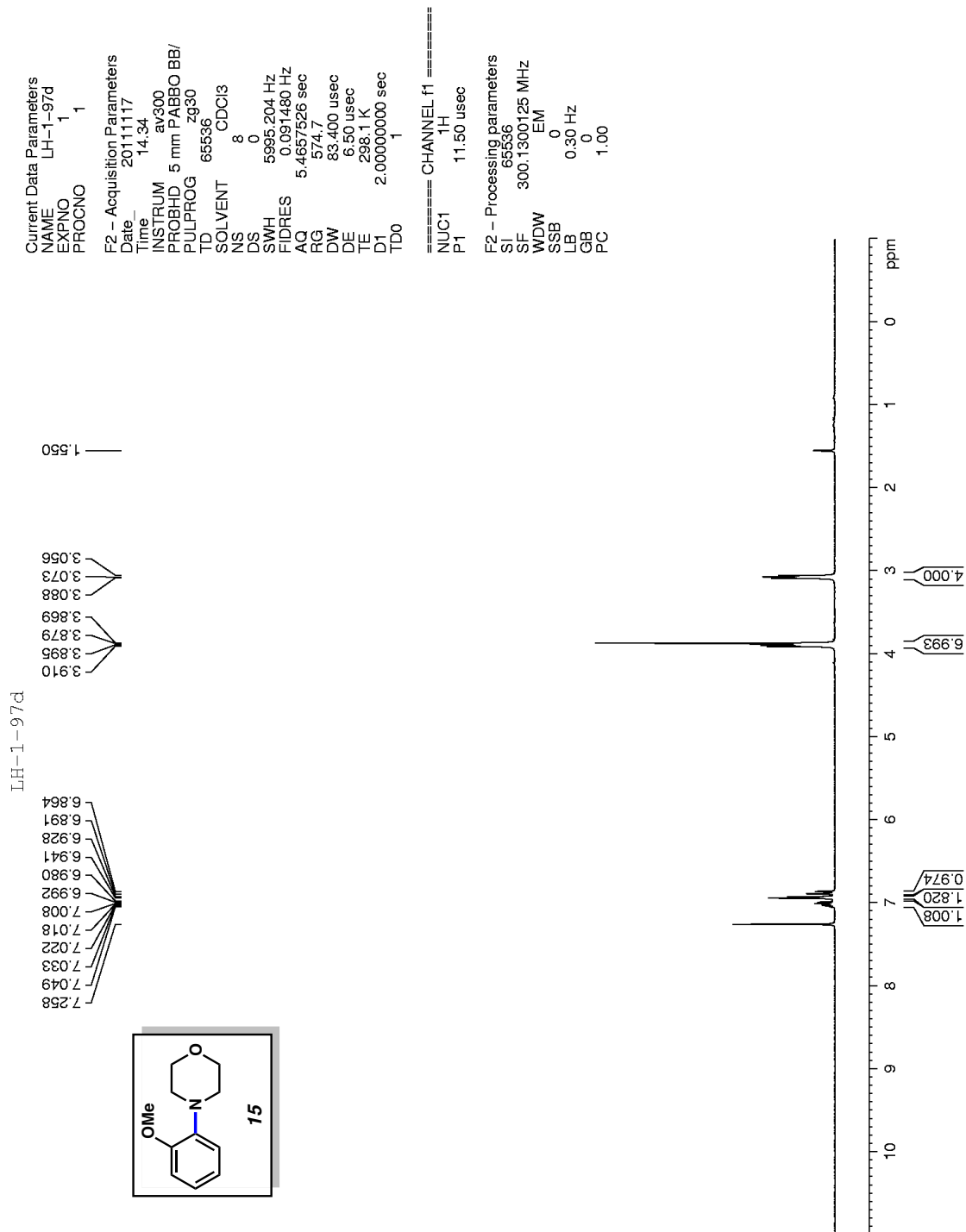
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 PROBHD 5 mm DCH 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 1000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2768500 sec
 RG 11
 DW 50.000 usec
 DE 10.00 usec
 TE 296.0 K
 D1 2.00000000 sec
 TD0 1

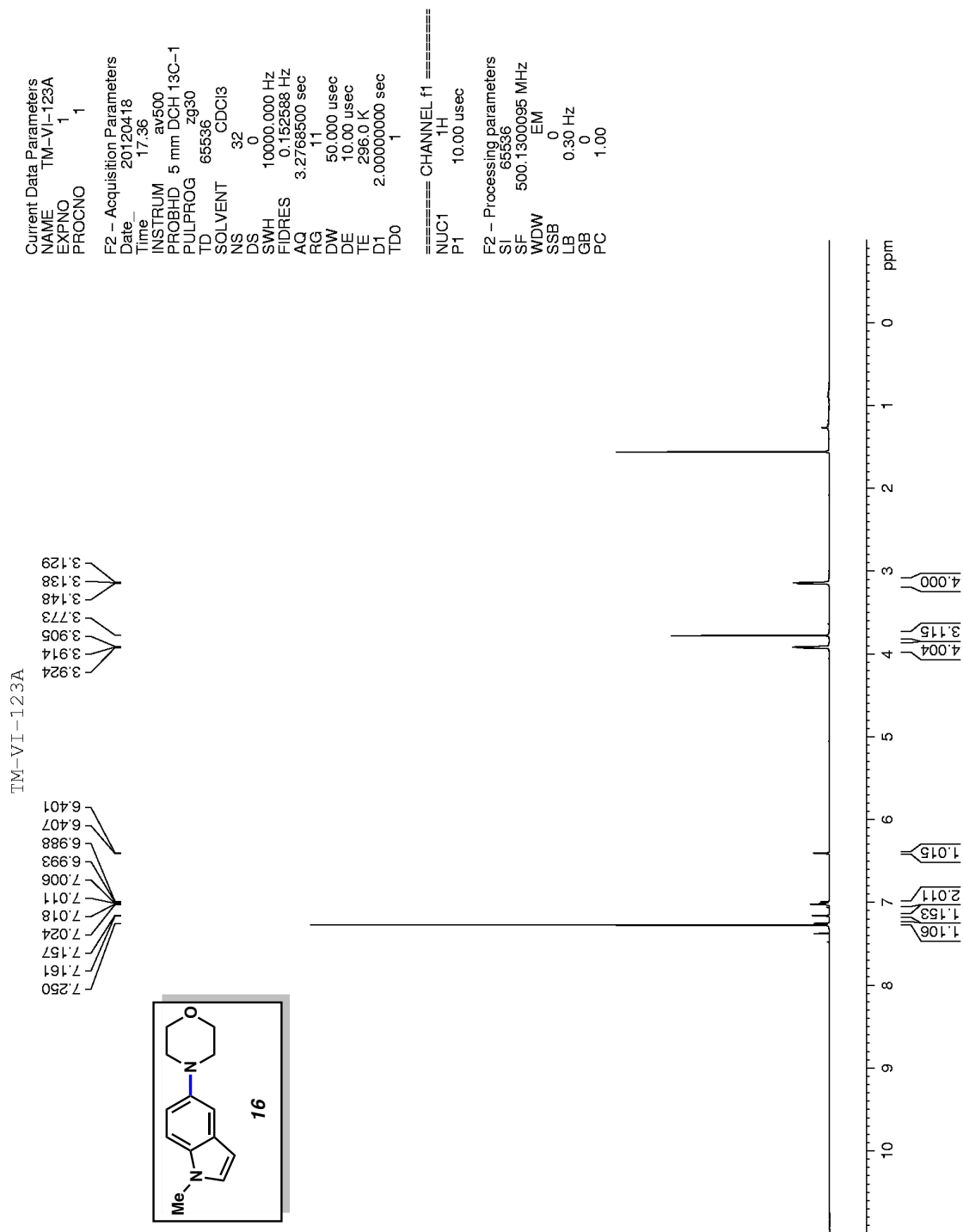
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 P1 10.00 usec

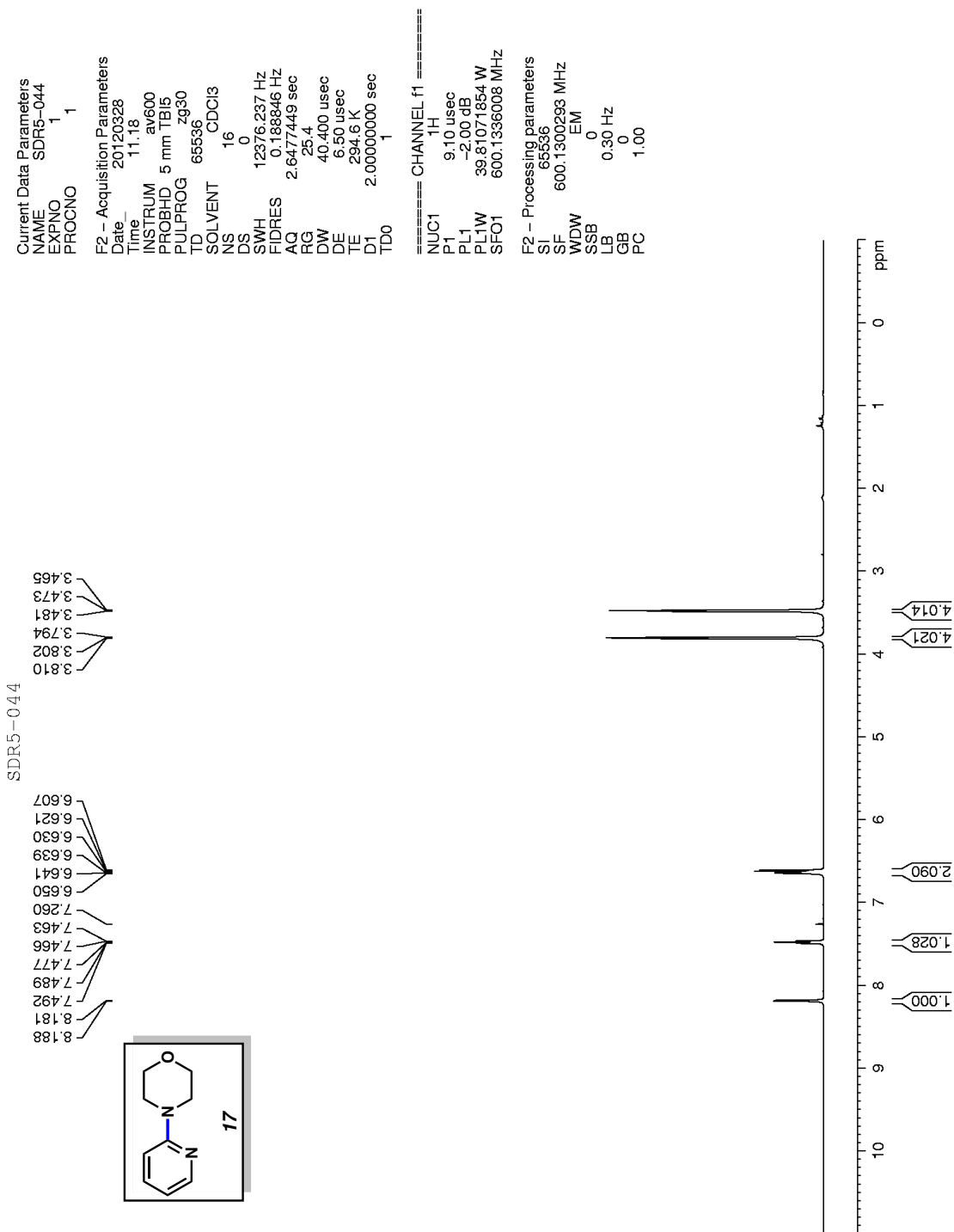
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 LB 0.30 Hz
 GB 0
 PC 1.00

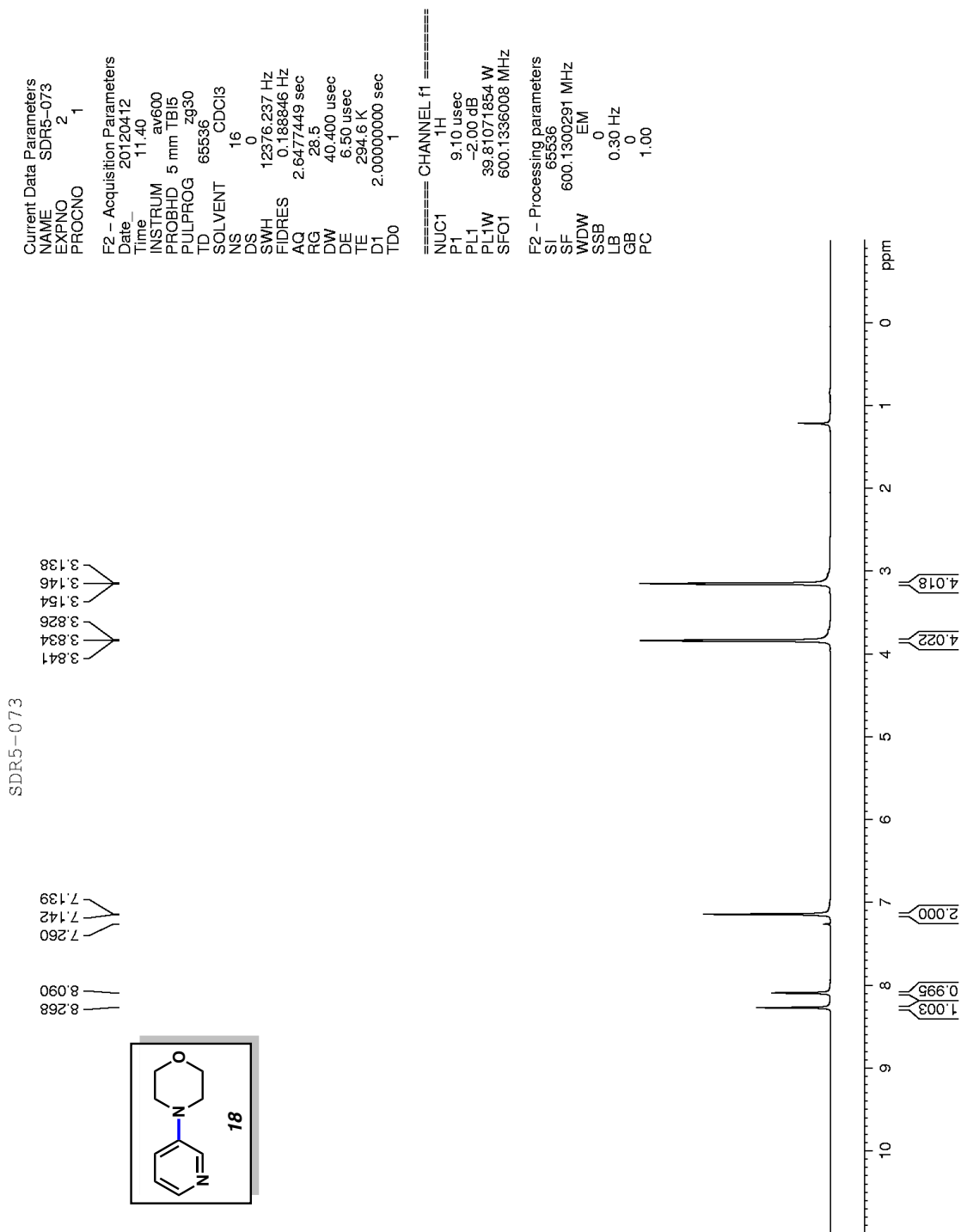












Current Data Parameters
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 EXPNO 59
 PROCNO 59

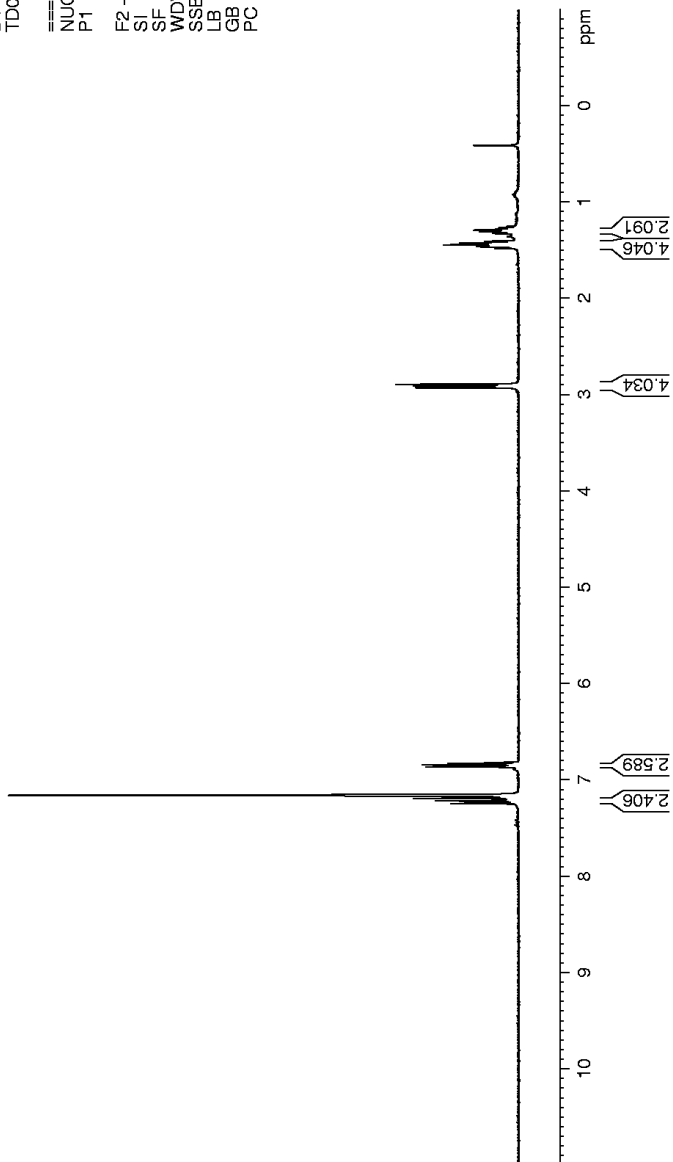
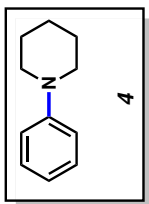
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 Date 20110830
 Time 11 40
 INSTRUM av300
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 24
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.091480 Hz
 AQ 5.4657526 sec
 RG 456.1
 DW 83.400 usec
 DE 6.50 usec
 TE 297.5 K
 D1 2.0000000 sec
 TD0 1

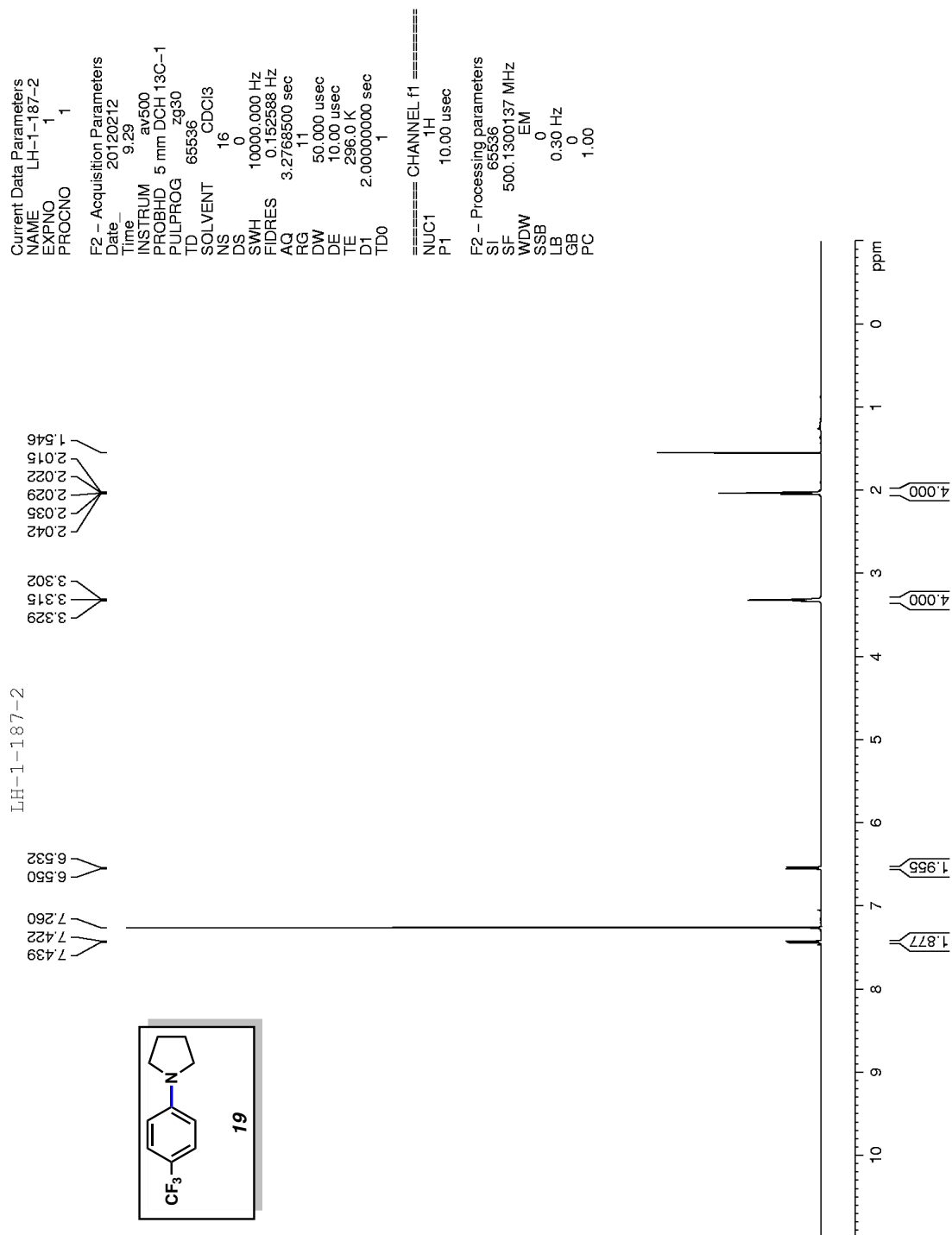
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 NUC1 1H
 P1 11.50 usec

F2 - Processing parameters
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 SF 300.1300333 MHz
 WDW EM
 SSB 0
 LB -0.10 Hz
 GB 0
 PC 1.00

1.265
1.270
1.276
1.287
1.306
1.315
1.324
1.405
1.415
1.422
1.441
1.460
1.480
2.889
2.909
2.927

6.813
6.818
6.821
6.829
6.832
6.839
6.842
6.846
6.851
6.861
6.864
6.866
7.181
7.189
7.189
7.192
7.194
7.201
7.206
7.208
7.213
7.215
7.218
7.221
7.224
7.236
7.240
7.242
7.243
7.252





Current Data Parameters
 NAME LH-1-210
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120228
 Time_ 11:56
 INSTRUM av500
 PROBHD 5 mm DCH 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 10000.000 Hz
 FIDRES 0.152568 Hz
 AQ 3.2768500 sec
 RG 11
 DW 50.000 usec
 DE 10.00 usec
 TE 296.0 K
 D1 2.00000000 sec
 TD0 1

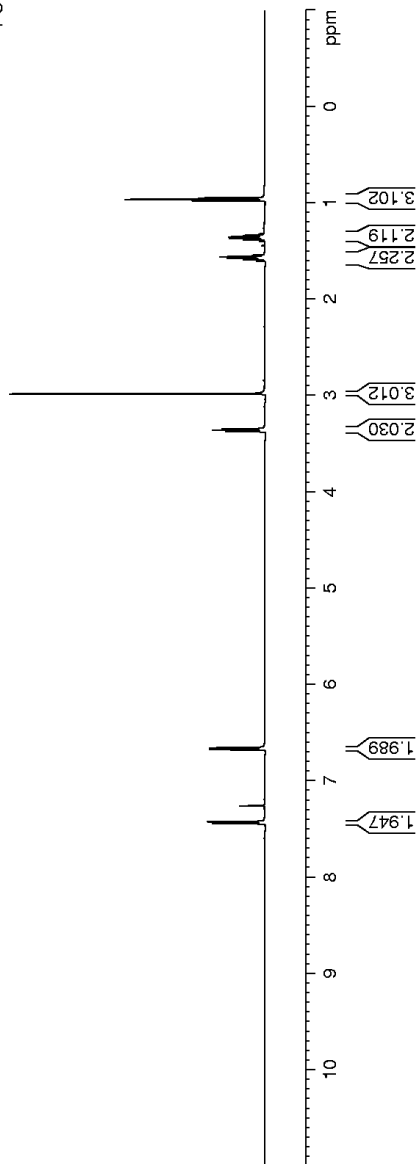
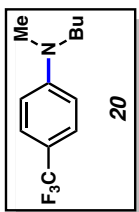
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 P1 10.00 usec

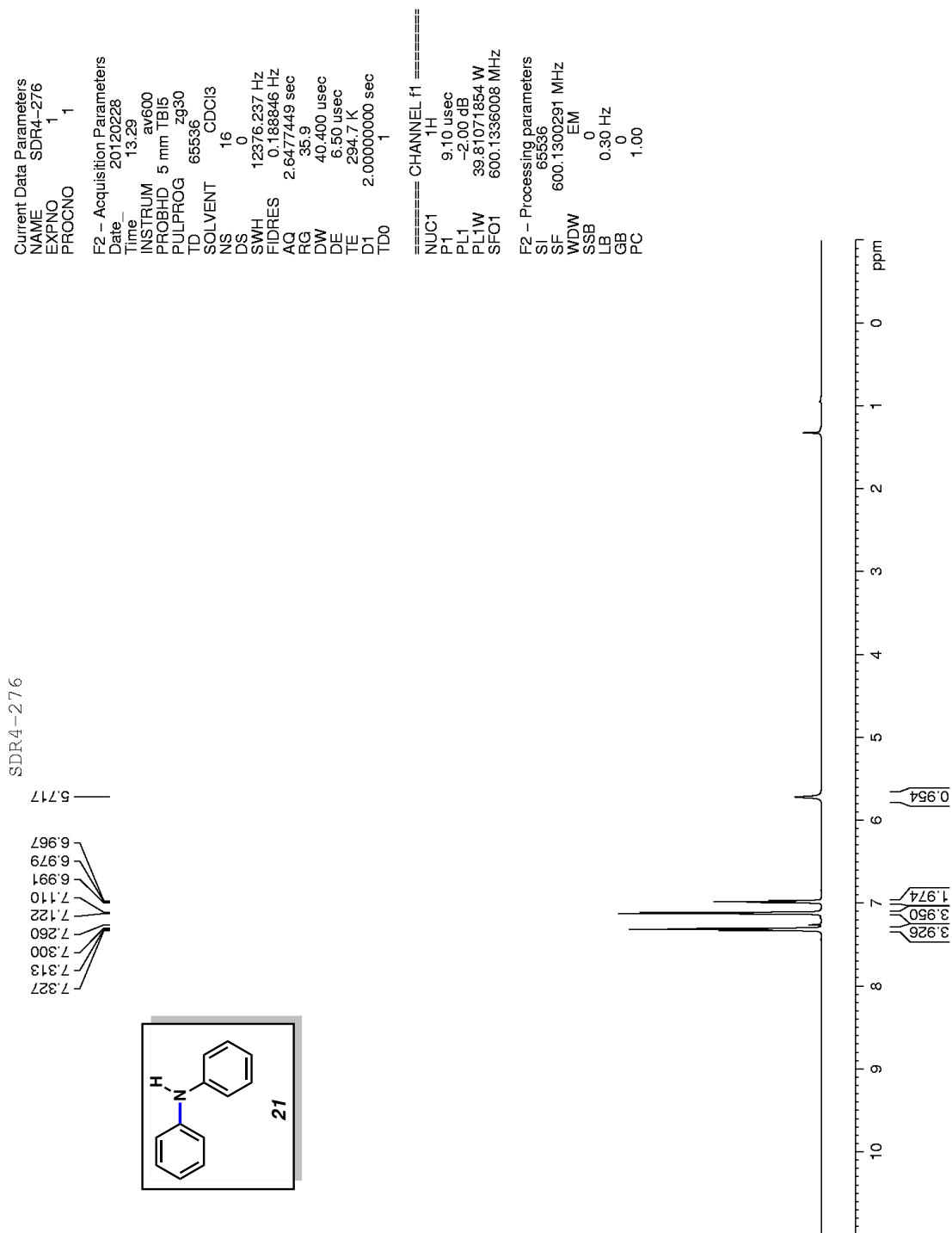
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 WDW EM
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 GB 0
 PC 1.00

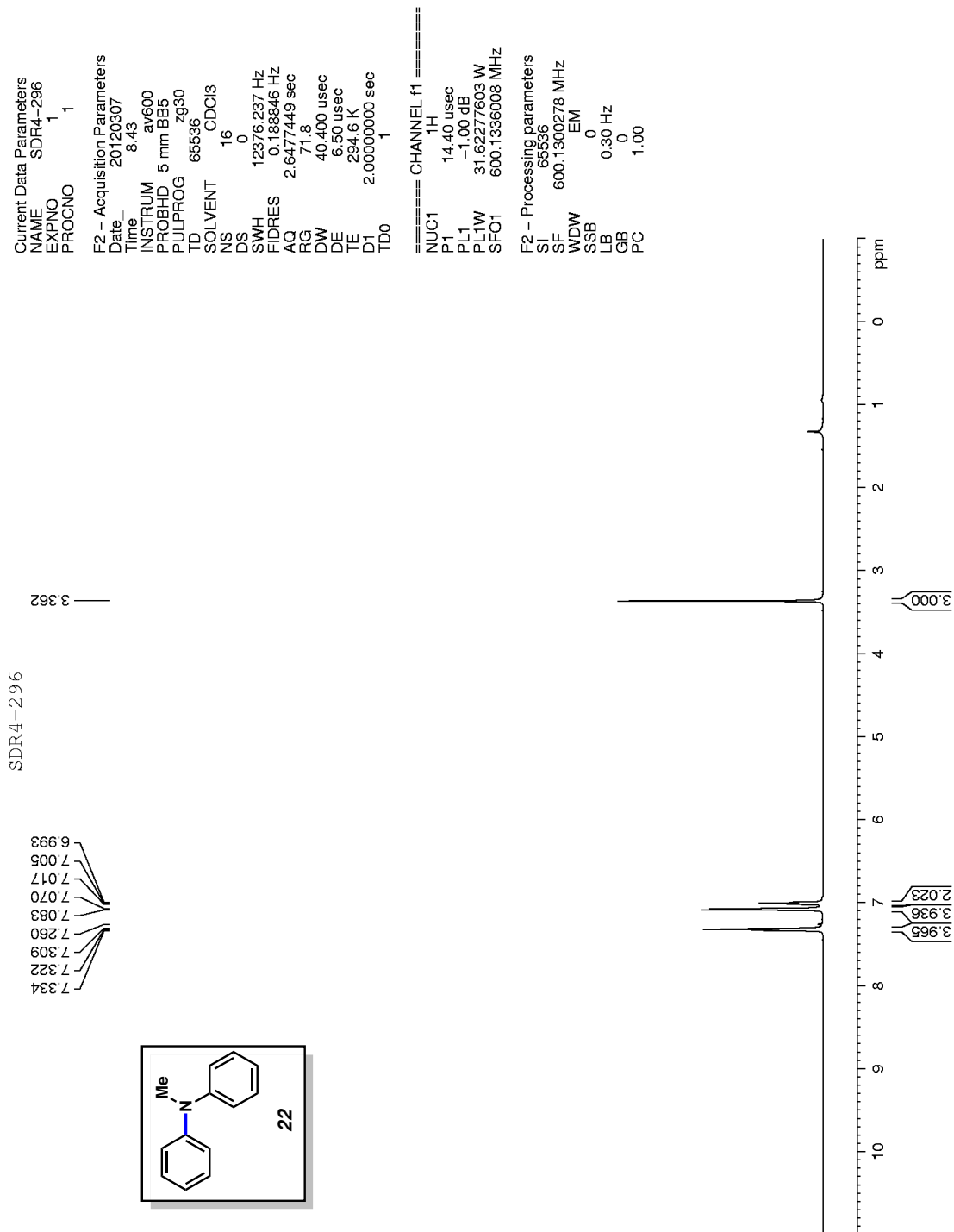
0.945
 0.960
 0.975
 1.320
 1.335
 1.350
 1.365
 1.380
 1.395
 1.541
 1.557
 1.571
 1.586
 1.601
 2.980
 3.342
 3.357
 3.373

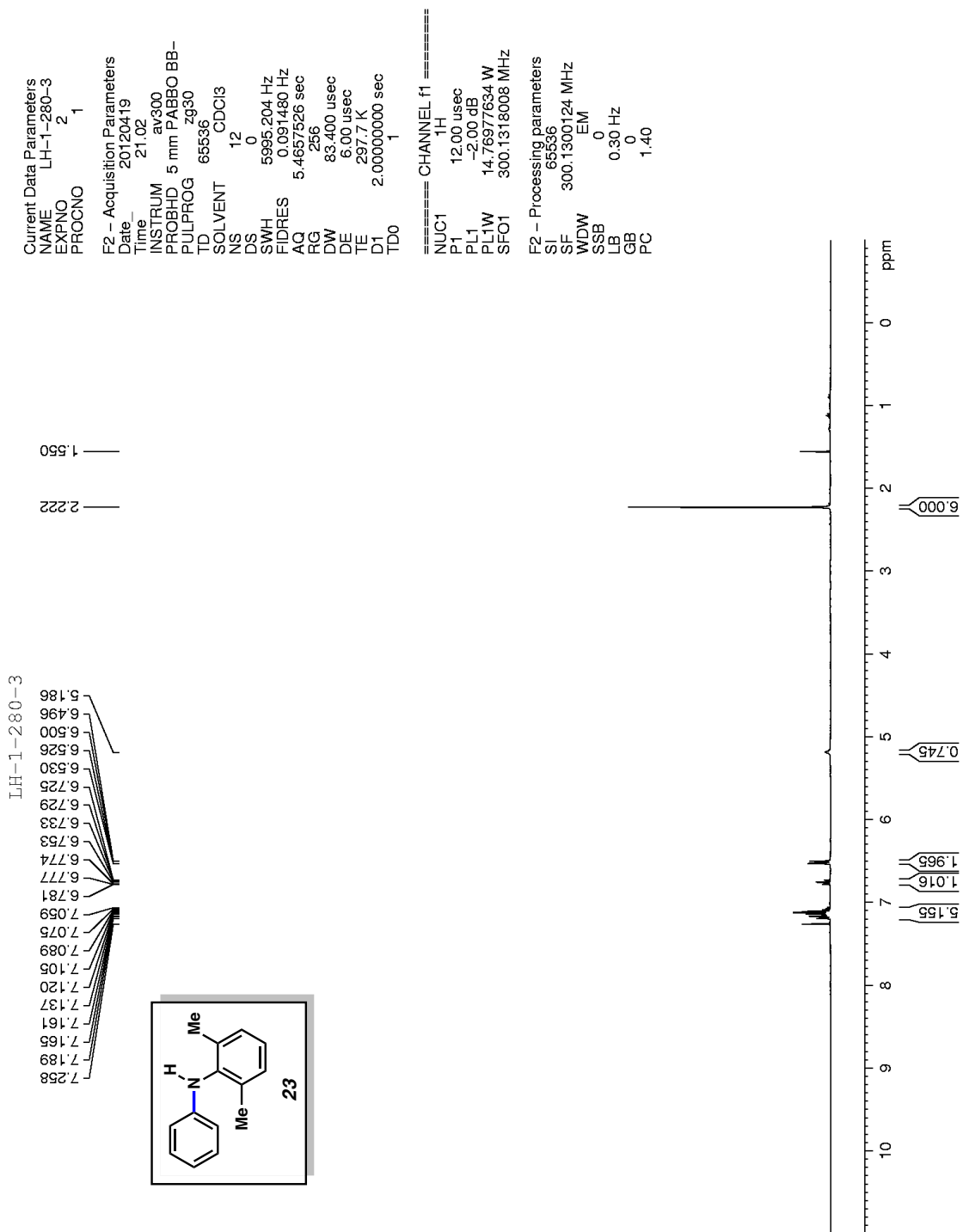
LH-1-210

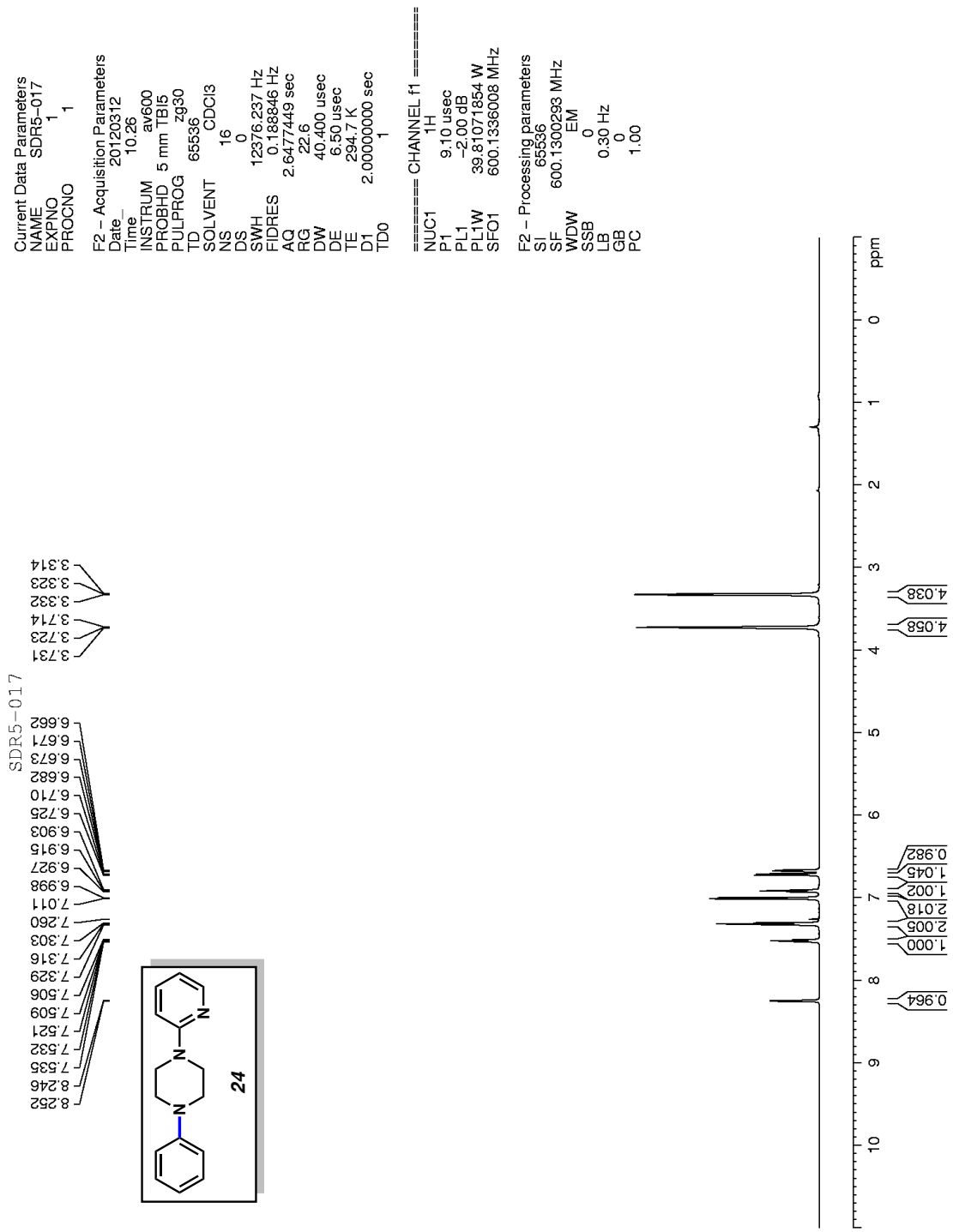
6.657
 6.674
 7.261
 7.424
 7.442











Current Data Parameters
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 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120321
 Time 10:36
 INSTRUM dx500
 PROBHD 5 mm bb-Z800
 PULPROG zg30
 TD 65536
 SOLVENT CDCI3
 NS 16
 DS 0
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2769001 sec
 RG 35.9
 DW 50.000 usec
 DE 6.00 usec
 TE 297.6 K
 D1 2.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.25 usec
 PL1 0.00 dB
 SFO1 500.330020 MHz

F2 - Processing parameters
 SI 32768
 SF 500.3300427 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

0.943
 0.928
 0.914

3.717
 3.703
 3.689
 3.675

SDR5-033
 5.160
 6.821
 6.836
 6.851
 6.907
 6.923
 6.942
 6.959
 7.058
 7.075
 7.167
 7.171
 7.188
 7.196
 7.203
 7.209
 7.213
 7.219
 7.283
 7.383
 7.398
 7.413
 7.430
 7.440
 7.744
 7.933
 7.948

