Supporting Materials

Palladium-Catalyzed Methylation of Aryl C-H Bond by Using Peroxides

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Contents

1) Experimental details and characterization data for all new compounds

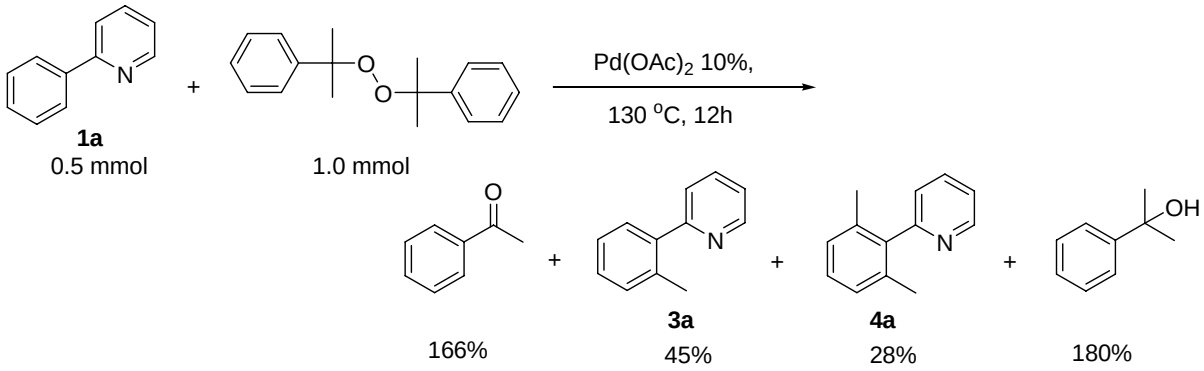
General information:

^{1}H NMR spectra were recorded on 400 MHz spectrometer in $CDCl_3$ solution and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm). The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; dd, doublet of doublet; ddd, doublet of doublet of doublet; m, multiplet; q, quartet; dq, doublet of quartet. The coupling constants, *J*, are reported in Hertz (Hz). ^{13}C NMR spectra were obtained at 100 MHz and referenced to the internal solvent signals (central peak is 77.00 ppm). MS data were obtained by Varian Saturn 2100D GC/MS Spectrometer. HRMS were made by McGill University. IR spectra were recorded by a Nexus 670 Avator FTIR Spectrometer and an ABB Bomem MB100 instrument. Flash column chromatography was performed over SORBENT silica gel 30-60 μm. Thin layer chromatography was performed by using Sorbent Silica Gel 60 F_{254} TLC plates and visualized with ultraviolet light.

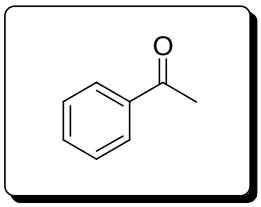
Substrates **1a**, **1b**, **1c**, **1d**, **1h** and **1i** were purchased from Aldrich Corp. Other substrates **3a**, **1e**, **1f** and **1g** were prepared via Suzuki coupling of the corresponding boronic acid and 2-bromopyridine according to the literature procedure. [1] **1j** was prepared according to the literature[2].

General procedure:

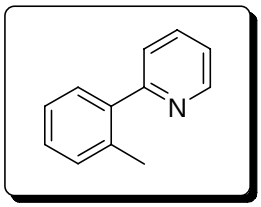
Methylation of 2-phenylpyridine with dicumyl peroxide:



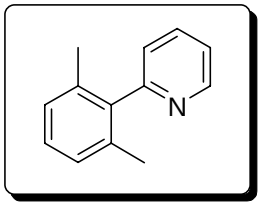
To a mixture of 2-phenylpyridine (**1a**) (79 mg, 0.5 mmol) and dicumyl peroxide (270 mg, 1.0 mmol), $Pd(OAc)_2$ (11.2 mg, 0.05 mmol) were added. Then the reaction mixture was stirred at 130°C for 12hrs under nitrogen atmosphere. After that, the resulting mixture was filtered through a short silica gel in a pipette eluting with methylene chloride. The solvent was evaporated and the residue was purified by silica gel column separation (eluted with hexane/methylene chloride = 1:2) to give acetophenone (99 mg, 166% based on 2-phenylpyridine), the desired product **3a** (35 mg, 41% based on 2-phenylpyridine), a second product **4a** (29 mg, 32% based on 2-phenylpyridine) and 2-phenylpropan-2-ol (122 mg, 180% based on 2-phenylpyridine).



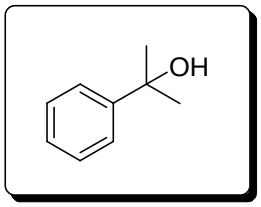
Acetophenone.[3] Register Number [98-86-2]. An oil; 99 mg (166% based on 2-phenyl pyridine); ^{1}H NMR (400 MHz, ppm) δ 7.80-7.95(m, 2H), 7.60-7.54(m, 1H), 7.50-7.44(m, 2H), 2.61(s, 3H); ^{13}C NMR (100 MHz, ppm) δ 197.9, 136.8, 132.9, 128.3, 128.1, 26.4.



2-*o*-Tolylpyridine (3a).[4] Colorless oil; 35 mg (41% based on 2-phenylpyridine). Register Number [10273-89-9]. ^{1}H NMR (400 MHz, ppm) δ 8.71-8.69(m, 1H), 7.74(ddd, J = 7.6, 7.6, 2.0 Hz, 1H), 7.41-7.39(m, 2H), 7.31-7.22(m, 4H), 2.37(s, 3H); ^{13}C NMR (100 MHz, ppm) δ 160.2, 149.4, 140.6, 136.4, 136.0, 131.0, 129.8, 128.5, 126.1, 124.3, 121.9, 20.5.



2-(2,6-Dimethylphenyl)pyridine (4a).[5] Colorless oil; 29 mg (32% based on 2-phenyl pyridine). Register Number [10273-91-3]. ^{1}H NMR (400 MHz, ppm) δ 8.66-8.62(m, 1H), 7.68(ddd, J = 8.0, 8.0, 2.0 Hz, 1H), 7.23-7.10(m, 3H), 7.04-7.01(m, 2H), 1.97(s, 6H); ^{13}C NMR (100 MHz, ppm) δ 160.1, 149.9, 136.5, 136.0, 128.1, 127.7, 124.7, 121.9 (possibly overlapped), 20.4.

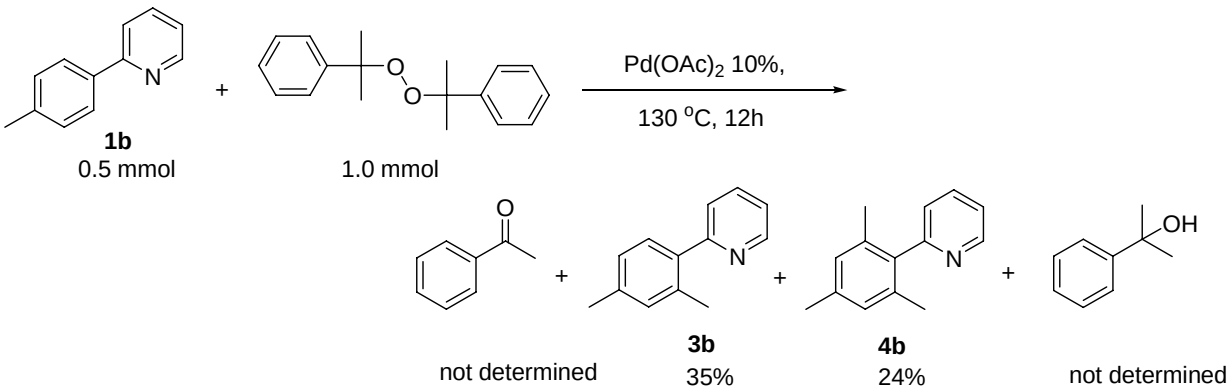


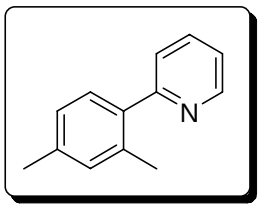
2-Phenylpropan-2-ol.[3] Colorless oil; 122 mg (180% based on 2-phenyl pyridine). Register Number [617-94-7]. An oil; 122 mg (180% based on 2-phenylpyridine); ^{1}H NMR (400 MHz, ppm) δ 7.52-7.48(m, 2H), 7.38-7.32(t, J = 15 Hz, 2H), 7.28-7.22(m, 1H), 1.59(s, 6H); ^{13}C NMR (100 MHz, ppm) δ 149.0, 128.0, 126.5, 124.3, 72.4, 31.6.

Methylation of other substrates with dicumyl peroxide

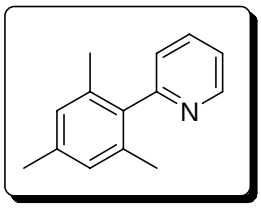
The characteristic data for methylation products were provided in the following. Acetophenone and 2-phenylpropan-2-ol were observed by-products in all cases; however, their yields were not determined during the separation by column.

Methylation of 1b with dicumyl peroxide:



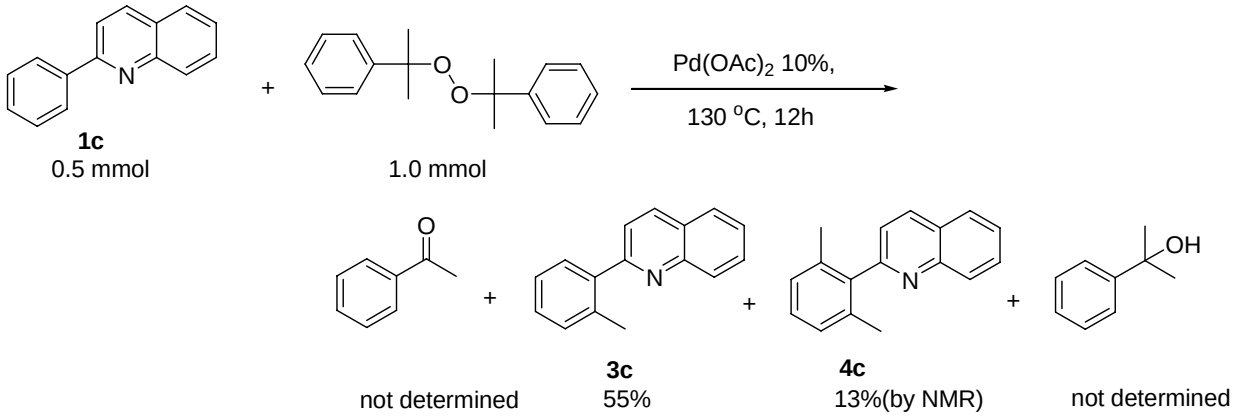


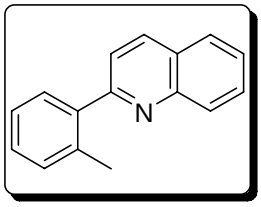
2-(2,4-Mimethylphenyl)pyridine (3b).[5] Following the general reaction procedure by using **1b** as the substrate, after separation by column chromatography on silica gel compound **3b** was obtained as a colorless oil (35 mg, 35% based on **1b**). . Register Number [914253-86-4]. ^{1}H NMR (400 MHz, ppm) δ 8.69-8.67(m, 1H), 7.72(ddd, J = 8.0, 8.0, 2.0 Hz, 1H), 7.38(d, J = 7.6 Hz, 1H), 7.31(d, J = 7.6 Hz, 1H), 7.23-7.20(m, 1H), 7.10-7.08(m, 2H), 2.36 (s, 3H), 2.34(s, 3H); ^{13}C NMR (100 MHz, ppm) δ 160.2, 149.3, 138.2, 137.8, 136.3, 135.8, 131.7, 129.8, 126.8, 124.3, 121.6, 21.4, 20.5.



2-Mesitylpyridine (4b).[6] By following the general reaction procedure, **4b** was obtained from **1b** as a colorless oil (24 mg, 24% based on **1b**). Register Number [75722-64-4]. ^{1}H NMR (400 MHz, ppm) δ 8.72-8.70(m, 1H), 7.74(ddd, J = 8.0, 8.0, 2.0 Hz, 1H), 7.26-7.21(m, 2H), 6.93 (s, 2H), 2.32 (s, 3H), 2.01(s, 6H); ^{13}C NMR (100 MHz, ppm) δ 160.2, 149.8, 137.9, 137.6, 136.4, 135.9, 128.5, 124.9, 121.7, 21.3, 20.3.

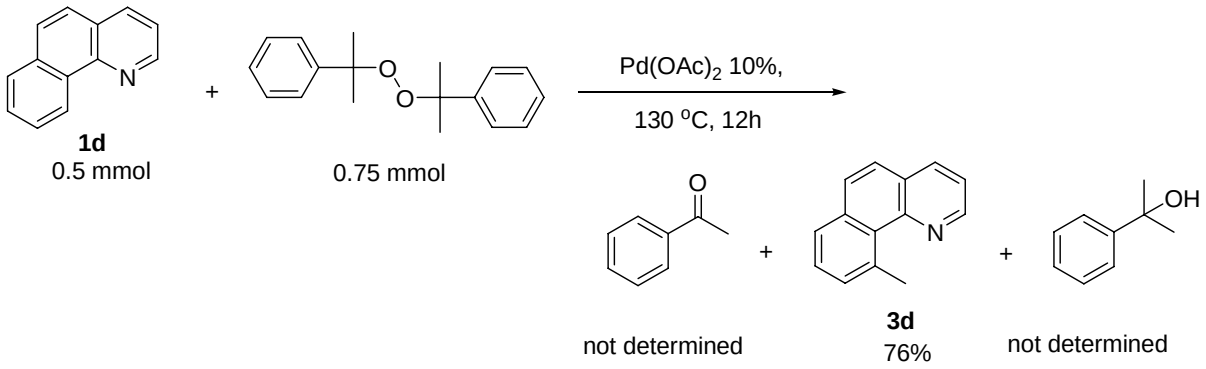
Methylation of 1c with dicumyl peroxide:

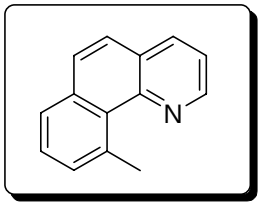




2-*o*-Tolylquinoline (3c).[7] By following the general reaction procedure, **3c** was obtained from **1c** as a viscous oil (60 mg, 55% based on **1c**). Register Number [52146-06-2]. ^{1}H NMR (400 MHz, ppm) δ 8.21(d, J = 8.4 Hz, 1H), 8.17(d, J = 8.4 Hz, 1H), 7.86(d, J = 8.4 Hz, 1H), 7.74(ddd, J = 7.6, 7.6, 1.2 Hz, 1H), 7.58-7.49(m, 3H), 7.37-7.30(m, 3H), 2.42(s, 3H); ^{13}C NMR (100 MHz, ppm) δ 160.5, 148.1, 140.9, 136.3, 136.2, 131.1, 129.9(two peaks), 129.8, 128.8, 127.7, 127.0, 126.7, 126.3, 122.6, 20.6.

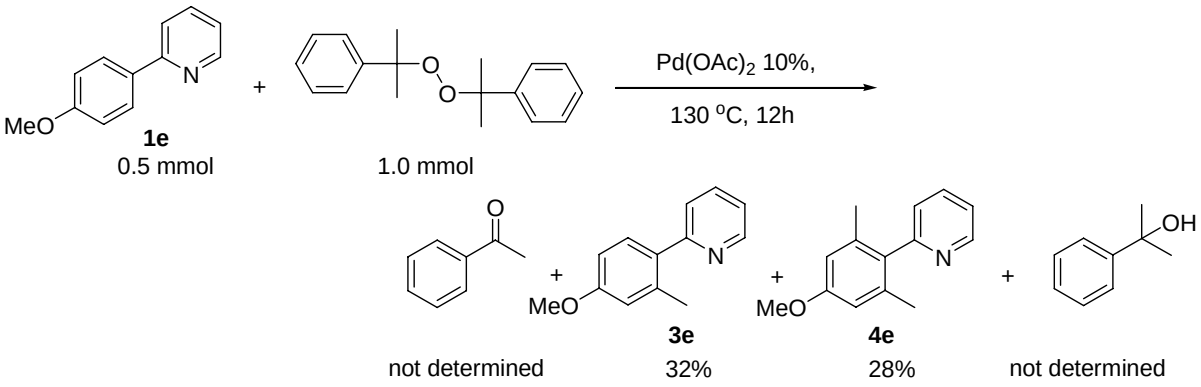
Methylation of 1d with dicumyl peroxide:

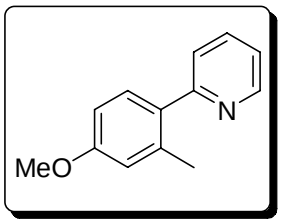




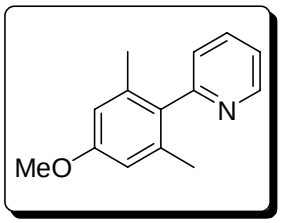
10-Methylbenzo[h]quinoline (3d).[5] By following the general reaction procedure except that 0.75 mmol of dicumyl peroxide was used, compound **3d** was obtained as a white solid from **1d** (73 mg, 76% based on **1d**). Register Number [914253-93-3]. ^{1}H NMR (400 MHz, ppm) δ 9.01(dd, J = 4.8, 2.0 Hz, 1H), 8.11(dd, J = 8.0, 2.0 Hz, 1H), 7.77-7.75(m, 2H), 7.61(d, J = 8.4 Hz, 1H), 7.57-7.52(m, 2H), 7.44(dd, J = 8.0, 4.4 Hz, 1H), 3.35(s, 3H); ^{13}C NMR (100 MHz, ppm) δ 149.2, 147.4, 139.0, 135.5, 135.4, 131.4, 130.1, 129.0, 127.7, 127.5, 127.0, 125.7, 120.8, 27.5.

Methylation of 1e with dicumyl peroxide:



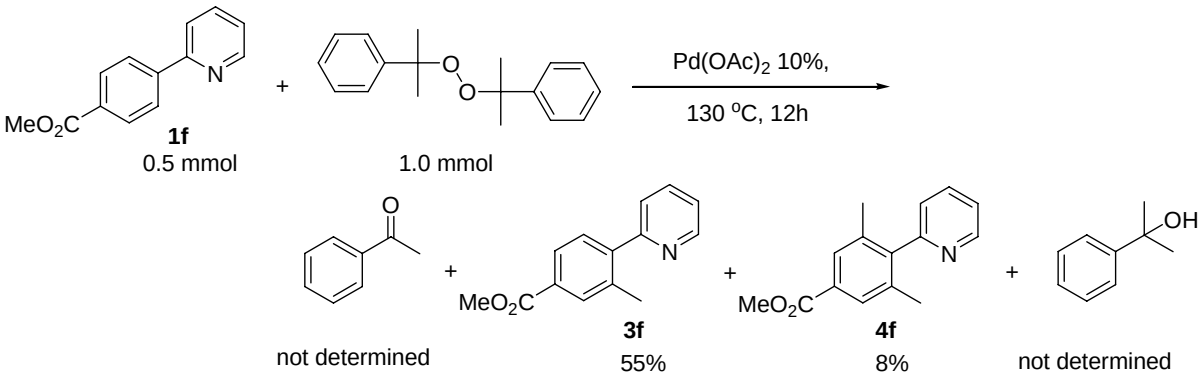


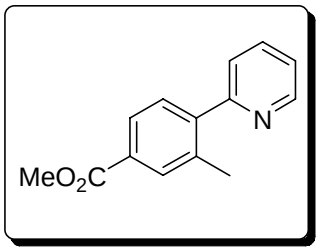
2-(4-Methoxy-2-methylphenyl)pyridine (3e).[4] By following the general reaction procedure, **3e** was obtained from **1e** as a colorless oil (32 mg, 32% based on **1e**). Register Number [521958-77-0]. ^{1}H NMR (400 MHz, ppm) δ 8.68-8.66(m, 1H), 7.71(ddd, J = 7.6, 7.6, 2.0 Hz, 1H), 7.38-7.35(m, 2H), 7.22-7.19(m, 1H), 6.83-6.81(m, 2H), 3.83(s, 3H), 2.37(s, 3H); ^{13}C NMR (100 MHz, ppm) δ 159.9, 159.7, 149.3, 137.6, 136.3, 133.4, 131.2, 124.3, 121.5, 116.3, 111.5, 55.5, 20.9.



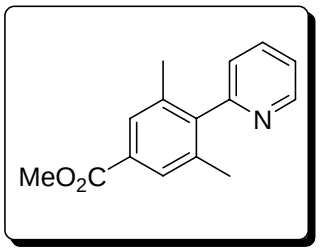
2-(4-Methoxy-2,6-dimethylphenyl)pyridine (4e). By following the general reaction procedure, **4e** was obtained from **1e** as a colorless oil (30 mg, 28% based on **1e**). IR(liquid): 3047, 3000, 2954, 2922, 2837, 1607, 1586, 1563, 1465, 1424, 1376, 1318, 1283, 1193, 1156, 1075, 1021, 935, 855, 794, 752 cm^{-1}; ^1H NMR (400 MHz, ppm) δ 8.69-8.68(m, 1H), 7.74-7.69(m, 1H), 7.25-7.18(m, 2H), 6.64(s, 2H), 3.79(s, 3H), 2.02(s, 6H); ^{13}C NMR (100 MHz, ppm) δ 160.0, 159.1, 149.8, 137.6, 136.4, 133.6, 125.3, 121.7, 113.1, 55.4, 20.7; MS (EI) *m/z* (%) 212(100), 197, 181, 168, 154, 141. HRMS calcd for $C_{14}H_{15}ON$: 213.1154; found: 213.1139.

Methylation of 1f with dicumyl peroxide:



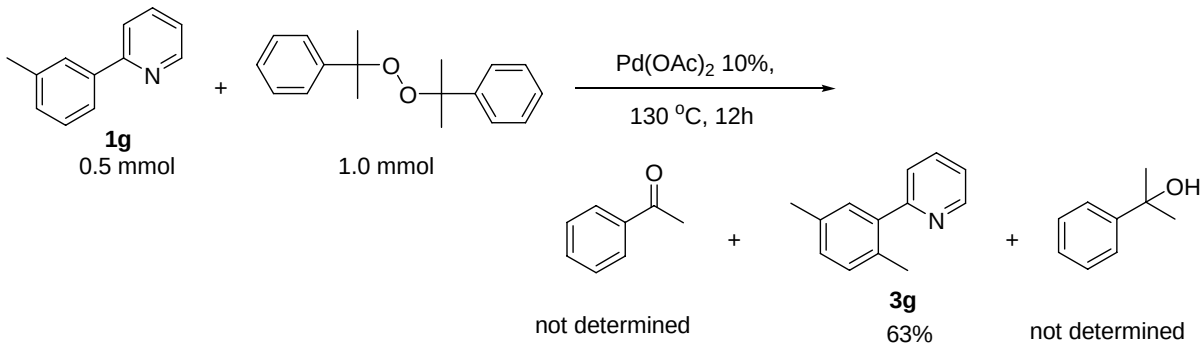


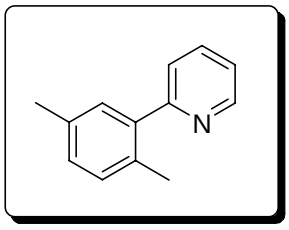
2-(4-Methoxycarbonyl-2-methylphenyl)pyridine (3f). By following the general reaction procedure, **3f** was obtained from **1f** as a colorless oil (62 mg, 55% based on **1f**). IR (KBr pellet): ν_{max} 2952, 1720, 1586, 1468, 1290, 1251, 1198, 1112, 764 cm^{-1}; ^{1}H NMR (400 MHz, ppm) δ 8.70-8.69(dd, *J* = 5.2, 0.8 Hz, 1H), 7.96(s, 1H), 7.93(d, *J* = 7.6 Hz, 1H), 7.77(m, 1H), 7.46(d, *J* = 8 Hz, 1H), 7.40(d, *J* = 7.6 Hz, 1H), 7.28(m, 1H), 3.91(s, 3H), 2.39(s, 3H); ^{13}C NMR (100 MHz, ppm) δ 167.0, 158.9, 149.3, 144.6, 136.2, 136.1, 131.8, 129.7, 129.7, 127.0, 124.0, 122.1, 52.0, 20.2; HRMS calcd for $C_{14}H_{14}NO_2^{+1}$(M+1): 228.1019; found: 228.1012.



Methyl 3,5-dimethyl-4-(pyridin-2-yl)benzoate (4f). By following the general reaction procedure, **4f** was obtained from **1f** as a colorless oil (10 mg, 8% based on **1f**). IR (KBr pellet): ν_{max} 2951, 1722, 1586, 1436, 1315, 1219, 1121, 771 cm^{-1}; ^{1}H NMR (300 MHz, ppm) δ 8.70-8.69(dd, *J* = 4.2, 0.9 Hz, 1H), 7.76(s, 2H), 7.73-7.72(m, 1H), 7.28-7.23(m, 1H), 7.19-7.16(d, *J* = 7.5 Hz, 1H), 3.88(s, 3H), 2.04(s, 6H); ^{13}C NMR (100 MHz, ppm) δ 167.1, 158.9, 149.7, 144.8, 136.4, 136.1, 129.2, 128.6, 123.9, 122.0, 51.9, 20.0. HRMS calcd for $C_{15}H_{16}NO_2^{+1}$(M+1): 242.1181; found: 242.1186.

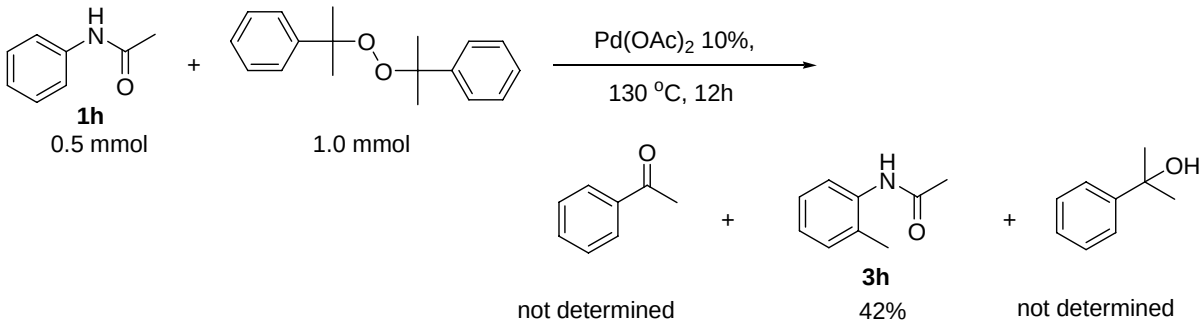
Methylation of 1g with dicumyl peroxide:

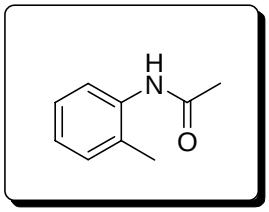




2-(2,5-Dimethylphenyl)pyridine (3g). By following the general reaction procedure, **3g** was obtained from **1g** as a colorless oil (58 mg, 63% based on **1g**). IR (KBr pellet): ν_{max} 2922, 1588, 1465, 1426, 793, 750 cm^{-1}; ^1H NMR (400 MHz, ppm) δ 8.71(m, 1H), 7.75(dt, *J* = 8, 2 Hz, 1H), 7.41(d, *J* = 7.6 Hz, 1H), 7.25-7.11 (m, 4H), 2.37(s, 3H), 2.33(s, 3H); ^{13}C NMR (100 MHz, ppm) δ 160.0, 149.2, 140.2, 135.9, 135.2, 132.4, 130.6, 130.2, 128.9, 124.0, 121.4, 20.8, 19.7; HRMS calcd for $C_{13}H_{14}N^{+1}$(M+1): 184.1121; found: 184.1118.

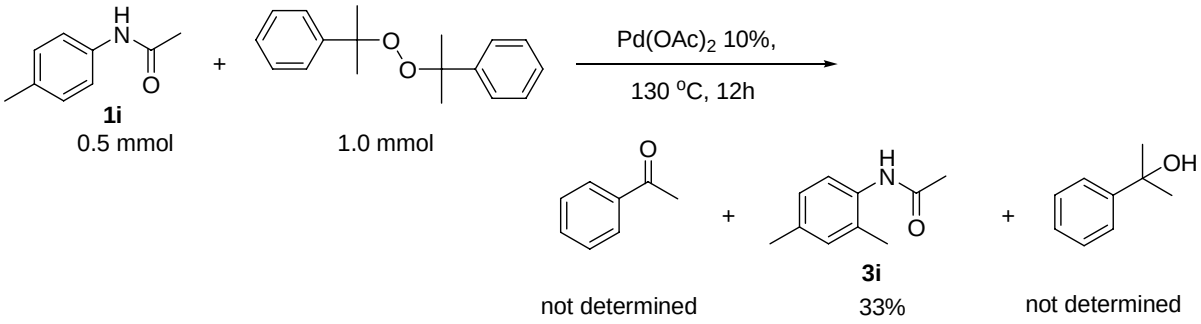
Methylation of 1h with dicumyl peroxide:

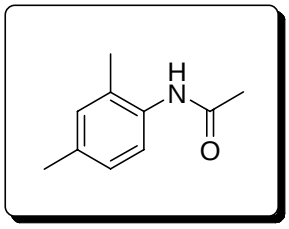




***N*-*o*-Tolylacetamide (3h).**[8] By following the general reaction procedure, **3h** was obtained from **1h** as a pale yellow solid (31 mg, 42% based on **1h**). Register Number [120-66-1]. ^{1}H NMR (400 MHz, ppm) ^{1}H NMR (400 MHz, ppm) δ 7.68(d, J = 8.0 Hz, 1H), 7.20-7.13(m, 3H), 7.07(dd, J = 6.8, 7.6 Hz, 1H), 2.23(s, 3H), 2.17(s, 3H); ^{13}C NMR (100 MHz, ppm) δ 168.8, 135.8, 130.7, 129.9, 126.9, 125.6, 123.9, 24.4, 18.0.

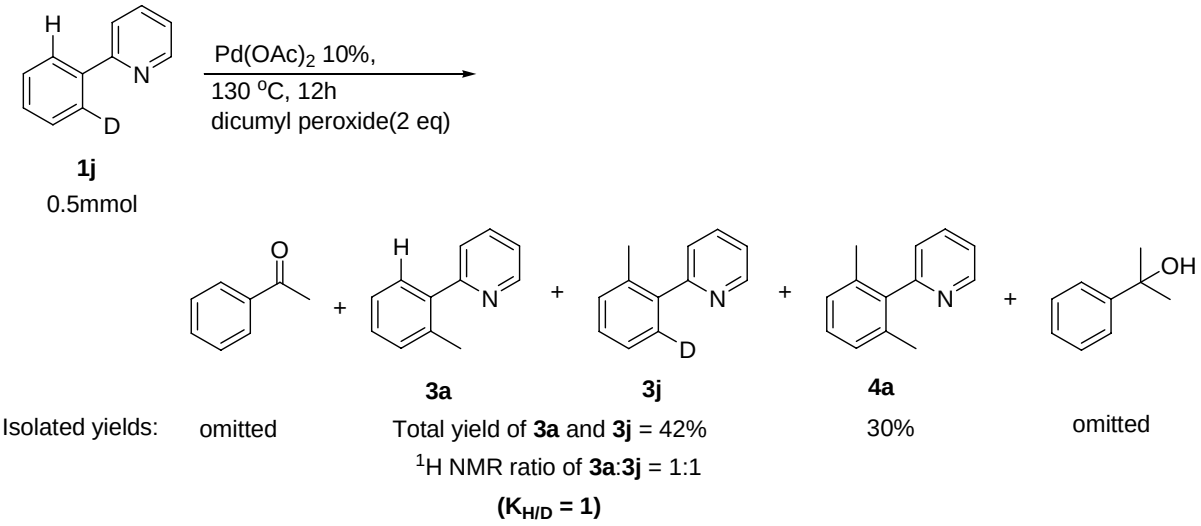
Methylation of 1i with dicumyl peroxide:



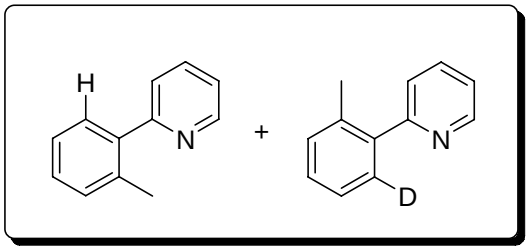


***N*-(2,4-dimethylphenyl)acetamide (3i).**[9] By following the general reaction procedure, **3i** was obtained from **1i** as a pale yellow solid (27 mg, 33% based on **1i**). Register Number [2050-43-3]. ^{1}H NMR (400 MHz, ppm) δ 7.48(d, J = 8.8 Hz, 1H), 7.14(s, 1H), 7.01-6.97(m, 2H), 2.28(s, 3H), 2.19(s, 3H), 2.16(s, 3H); ^{13}C NMR (100 MHz, ppm) δ 168.9, 135.5, 133.1, 131.4, 130.5, 127.4, 124.3, 24.3, 21.1, 18.0.

2) Mechanistic investigation: Isotope effect



To a mixture of 2-phenyl pyridine **1j** (80 mg, 0.5 mmol) and dicumyl peroxide (270 mg, 1.0 mmol), $Pd(OAc)_2$ (11.2 mg, 0.05 mmol) were added. Then the reaction mixture was stirred at 130°C for 12hr under nitrogen atmosphere. After that, the resulting mixture was filtered through a short silica gel in a pipette eluting with methylene chloride. The solvent was evaporated and the residue was purified by silica gel column chromatography (eluting with hexane/methylene chloride = 1:2), to give acetophenone (its yield was not determined), the mono-methylated product **3a** and **3j** (overall 35 mg, 42% based on **1j**), ^{1}H NMR analysis showed that the ratio of *ortho*-proton product **3a** to *ortho*-deuterium product **3j** is 1:1 (Compared with the ^{1}H NMR spectrum of pure **3a**, the integration of the peaks at 7.40 was 1.48 instead of 2). Then the second product **4a** (28 mg, 30% based on **1j**), and 2-phenylpropan-2-ol (its yield was also omitted) were eluted successively.



3a and **3j** (crude NMR ratio of **3a** and **3j** = 1:1)

Colorless oil; 35 mg (42% based on **1j**). ^{1}H NMR (400 MHz, ppm) δ 8.71-8.69(m, 1H), 7.76-7.72(m, 1H), 7.41-7.39(m, 1.5H), 7.31-7.22(m, 4H), 2.37(s, 3H); ^{13}C NMR (75 MHz, ppm) δ 160.0, 149.2, 140.3, 136.1, 135.7, 130.7, 129.6, 128.2, 125.8, 125.7, 124.1, 121.6, 20.3. MS (EI) *m/z* (%) 170, 169(100), 168, 167.

References:

[1] Littke, A. F.; Dai. C. Y.; Fu. G. C. *J. Am. Chem. Soc.* **2000**. *122*, 4020-4028.

[2] Chen, X.; Hao, X.-S.; Goodhue, C.E.; Yu, J.-Q. *J. Am. Chem. Soc.* **2006**, *128*, 6790**.**

[3] Spectral data were identical to those of the commercially available products.

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[5] Chen, X.; Goodhue, C. E. Yu, J.-Q. *J. Am. Chem. Soc.* **2006**, *128*, 12634-12635.

[6] Korn, T. J.; Schade, M. A.; Cheemala, M. N.; Wirth, S.; Guevara, S. A.; Cahiez, G.; Knochel. P. *Synthesis* **2006**, *21*, 3547-3574.

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[8] Ramalingan, C.; Park, Y.-T. *J. Org. Chem.* **2007**, *72*, 4536-4538.

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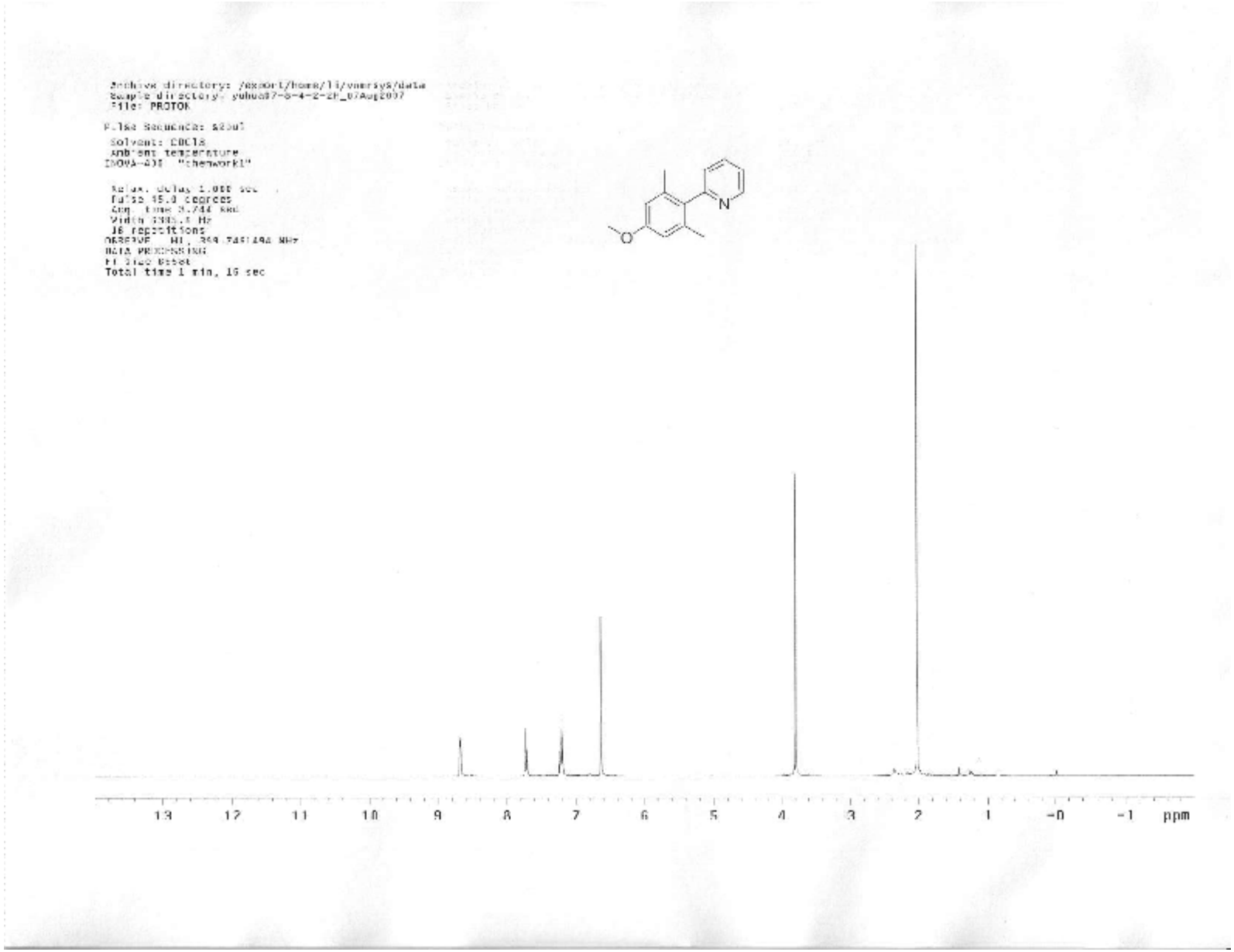
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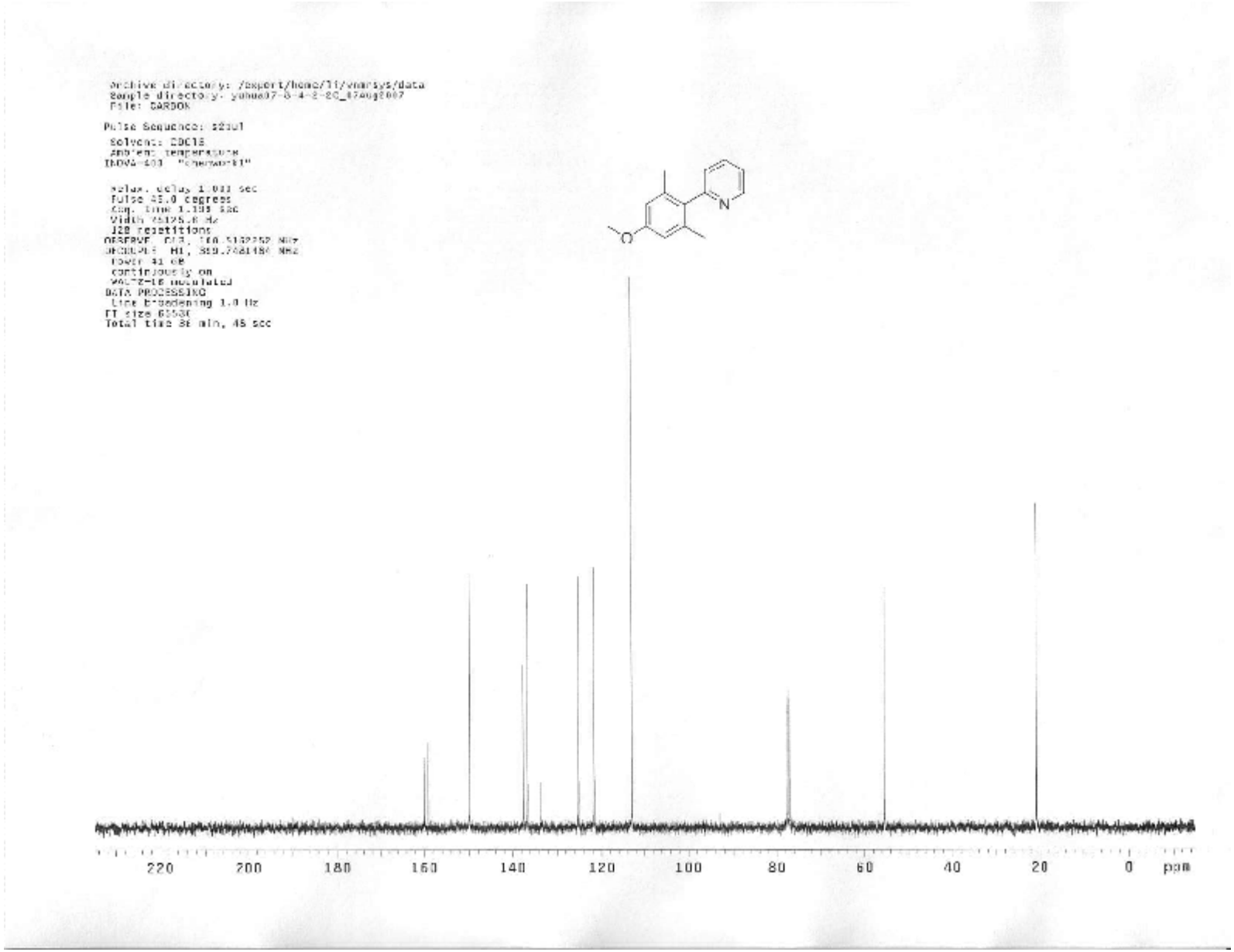
Ambient temperature
INOVA-400 "chemwork1"
Relax. delay 1.000 sec
Pulse 15.0 degrees
Acq. time 3.744 sec
Width 3385.5 Hz
16 repetitions
OBSERVE H1, 399.7451494 MHz
DATA PROCESSING
FT Size 65536
Total time 1 min, 16 sec
N
O
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm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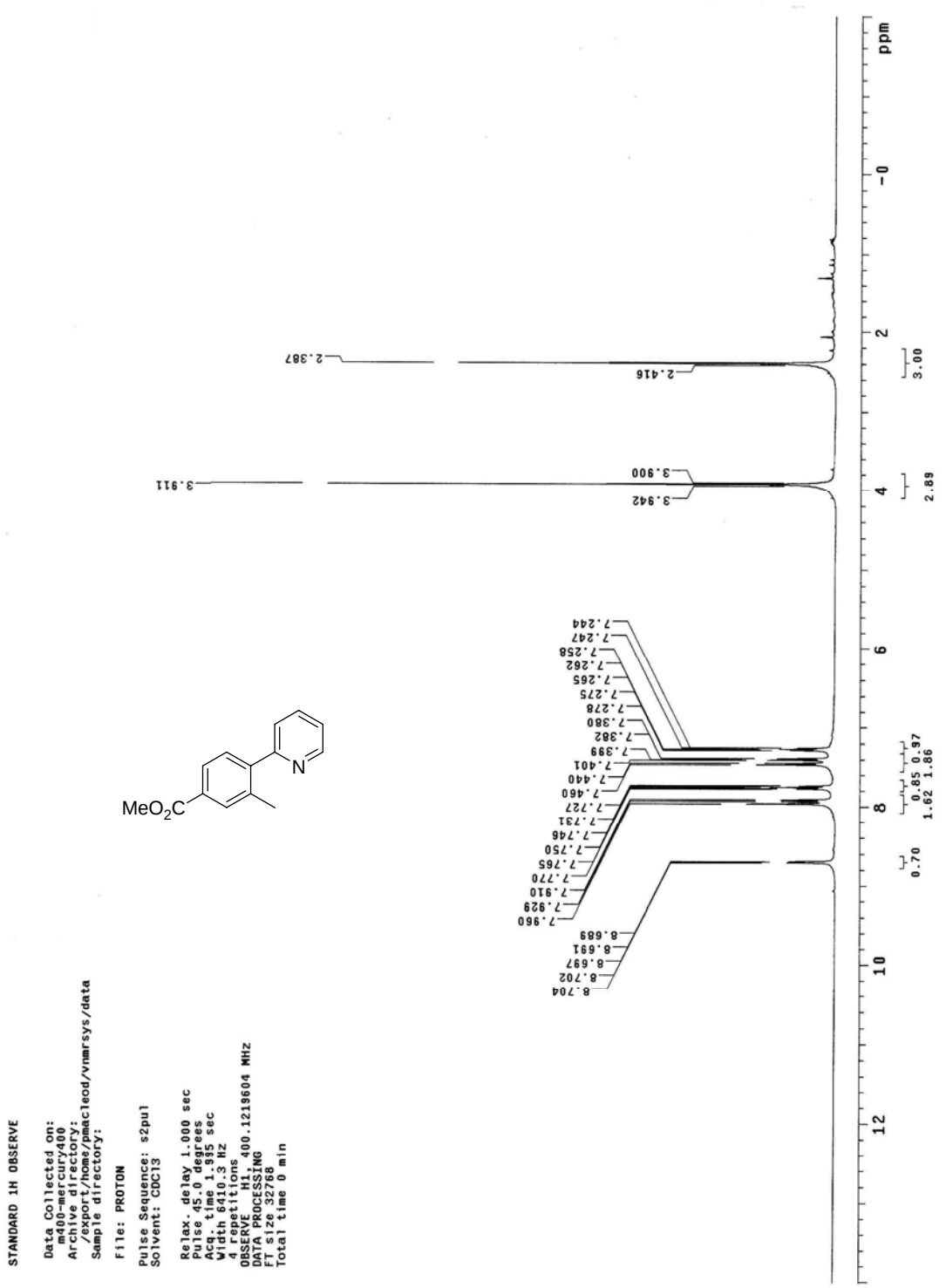
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-400 "chemwb41"
relax. delay 1.000 sec
pulse 45.0 degrees
acq. time 1.185 sec
width 25125.8 Hz
128 repetitions
OBSERVE C13, 100.5197252 MHz
DECOUPLE H1, 399.7681480 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 8 min, 45 sec
O
N
220
200
180
160
140
120
100
80
60
40
20
0
ppm


STANDARD 1H OBSERVE
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m400-mercury400
Archive directory:
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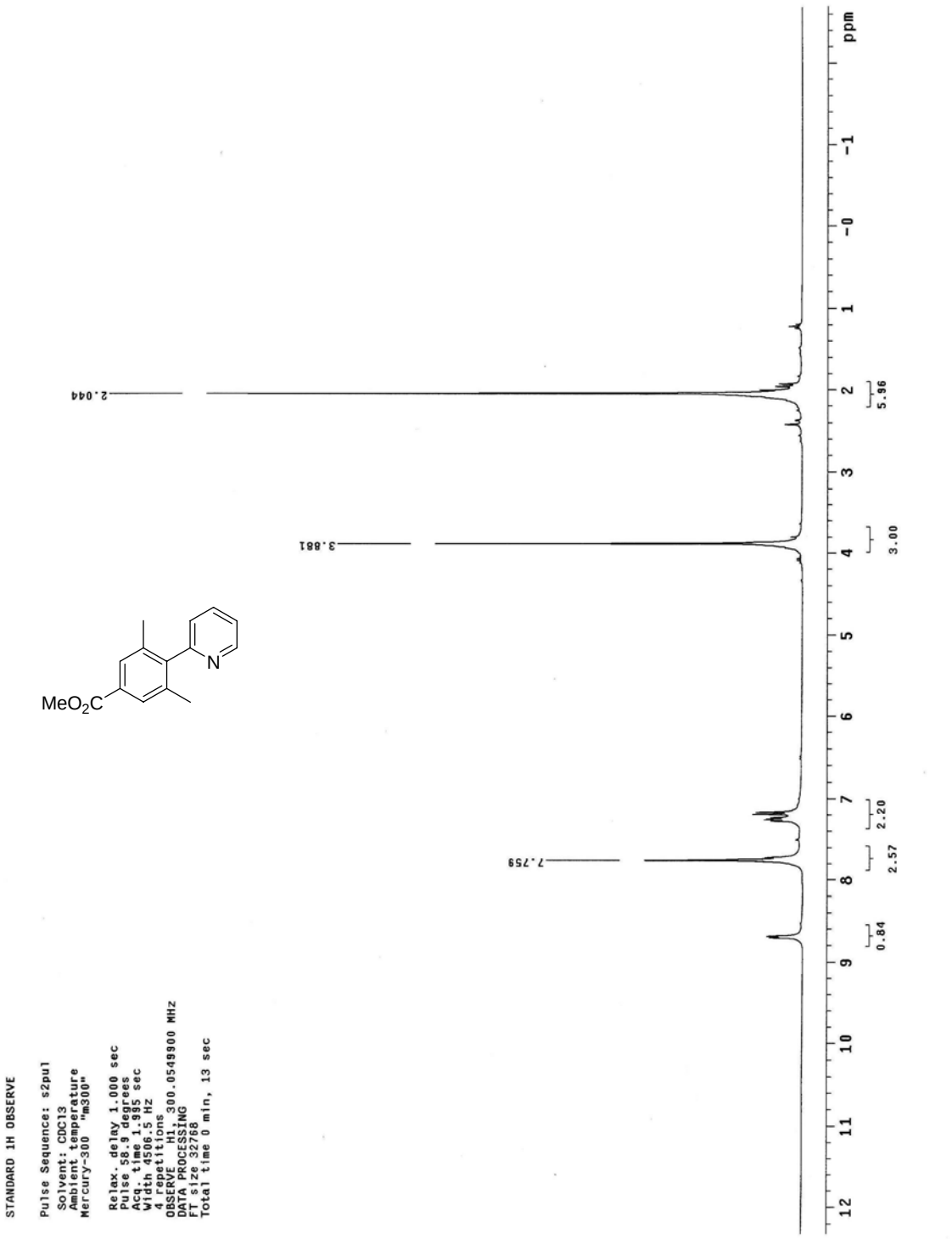
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.995 sec
Width 6410.3 Hz
4 repetitions
OBSERVE H1, 400.1219604 MHz
DATA PROCESSING
FT size 32768
Total time 0 min
MeO2C
N
ppm
2.387
2.416
3.911
3.900
3.942
8.704
8.702
8.697
8.691
8.689
7.960
7.929
7.910
7.770
7.765
7.750
7.746
7.731
7.727
7.460
7.440
7.401
7.399
7.382
7.380
7.278
7.275
7.265
7.262
7.258
7.247
7.244
0.70
0.85 0.97
1.62 1.86
3.00
2.89

13C OBSERVE
Data Collected on:
m400-mercury400
Archive directory:
/export/home/pmacleod/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 23.6 C / 296.8 K
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
640 repetitions
OBSERVE C13, 100.6107539 MHz
DECOUPLE H1, 400.1239934 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 48 min
MeO2C
N
166.960
158.897
149.295
144.623
136.217
136.072
131.842
129.685
129.655
126.980
123.954
122.095
77.312
77.000
76.680
52.033
20.176
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300 "m300"
Relax. delay 1.000 sec
Pulse 58.9 degrees
Acq. time 1.985 sec
Width 4506.5 Hz
4 repetitions
OBSERVE H1, 300.0549900 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 13 sec
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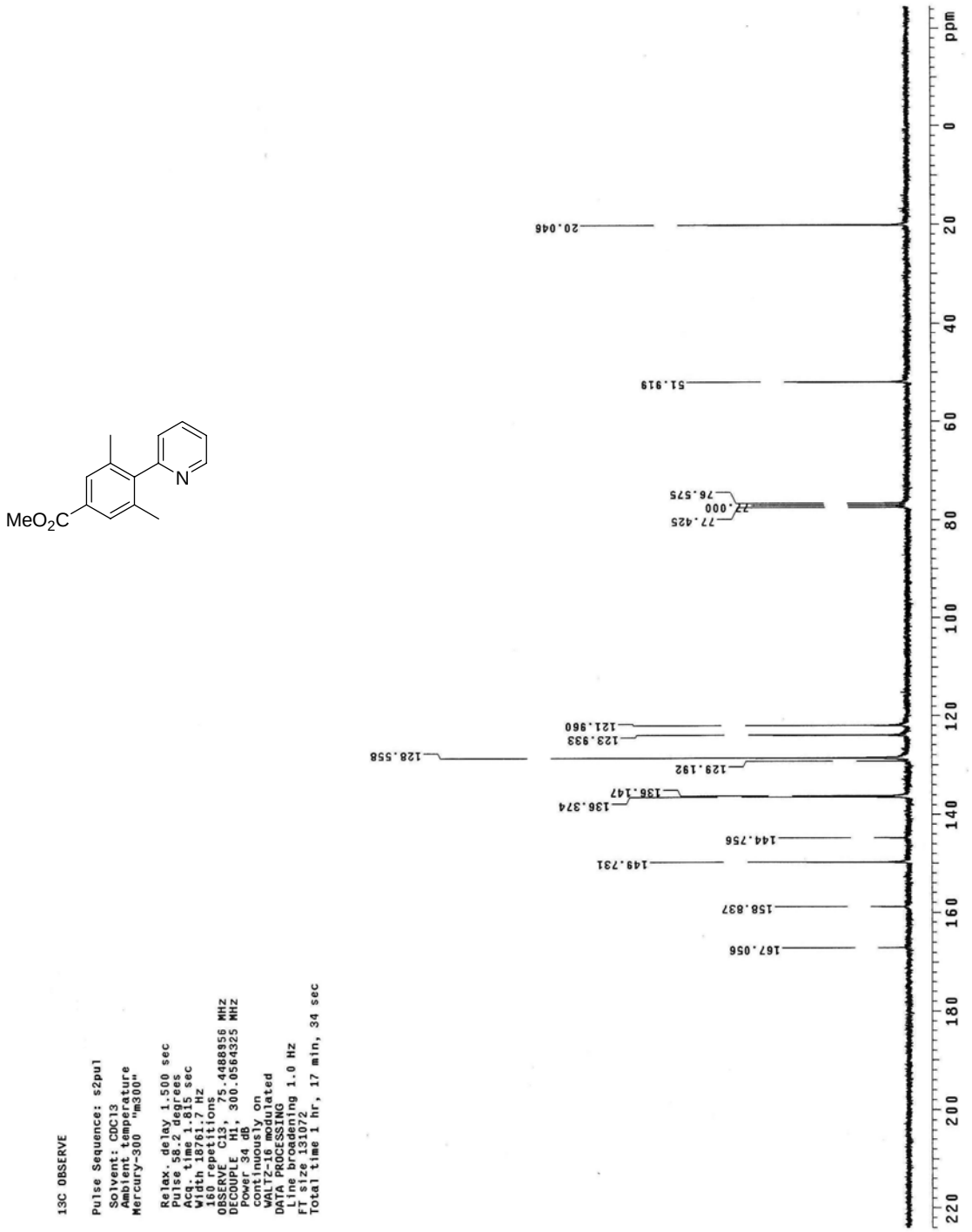
3.881
7.759
5.96
3.00
2.20
2.57
0.84
ppm
MeO2C
N


MeO2C
N
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300 "m300"
Relax. delay 1.500 sec
Pulse 58.2 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
160 repetitions
OBSERVE C13, 75.4488956 MHz
DECOUPLE H1, 300.0564325 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 17 min, 34 sec
20.046
51.919
77.425
77.000
76.575
121.960
123.933
128.558
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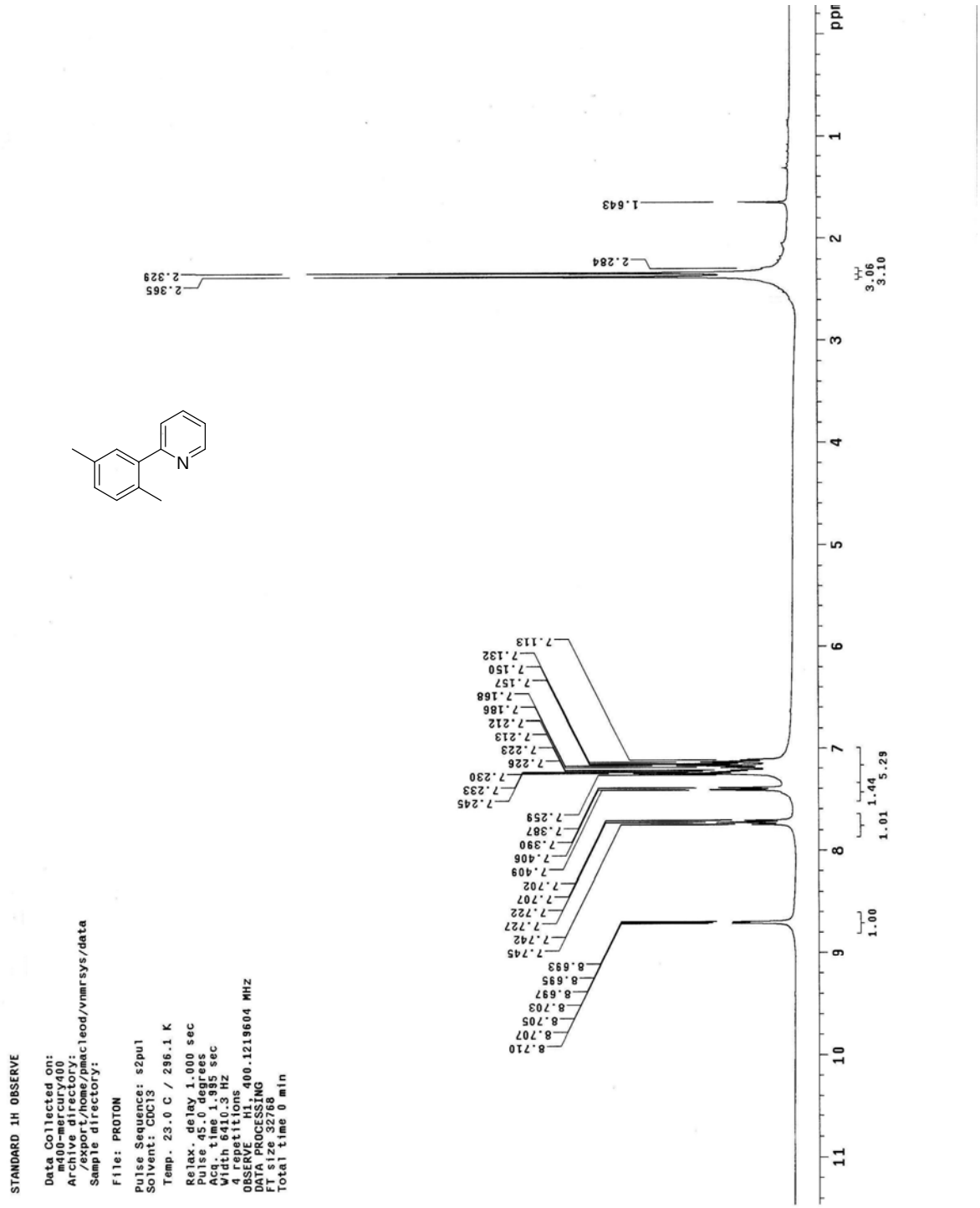
136.147
136.374
144.756
149.731
158.837
167.056
ppm


STANDARD 1H OBSERVE
Data Collected on:
m400-mercury400
Archive directory:
/export/home/pmacleod/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 23.0 C / 296.1 K
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.985 sec
Width 6410.3 Hz
4 repetitions
OBSERVE H1, 400.1219604 MHz
DATA PROCESSING
FT size 32768
Total time 0 min
N
2.365
2.329
2.284
1.643
8.710
8.707
8.705
8.703
8.697
8.695
8.693
7.745
7.742
7.727
7.722
7.707
7.702
7.409
7.406
7.390
7.387
7.259
7.245
7.233
7.230
7.226
7.223
7.213
7.212
7.186
7.168
7.157
7.150
7.132
7.113
1.00
1.01
1.44
5.29
3.06
3.10
ppm

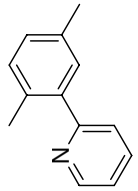
13C OBSERVE

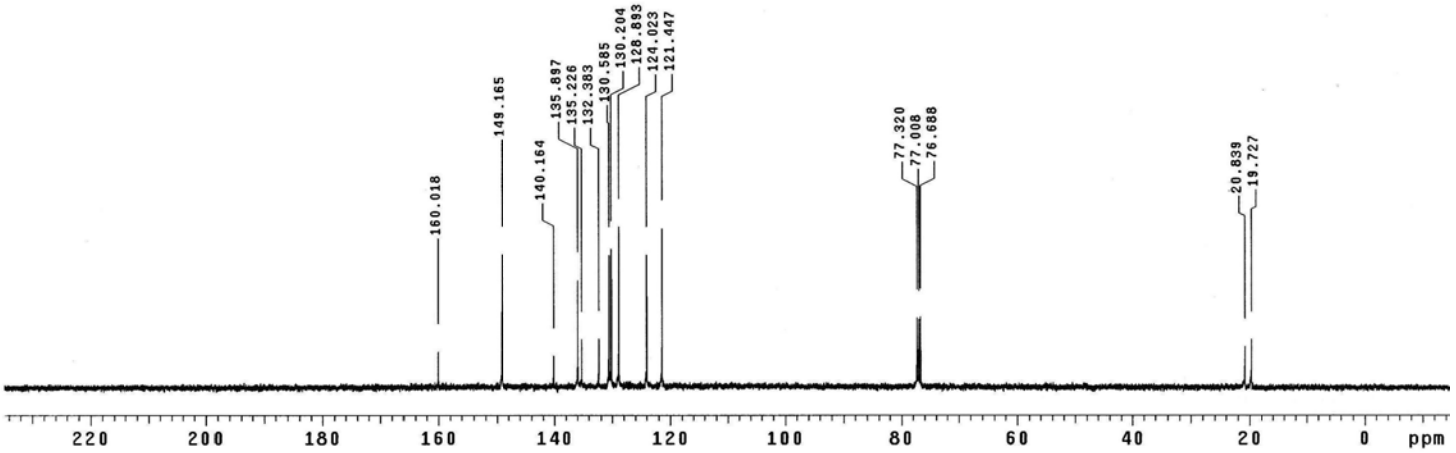
Data Collected on:
m400-mercury400
Archive directory:
/export/home/pmacleod/vnmrsys/data
Sample directory:

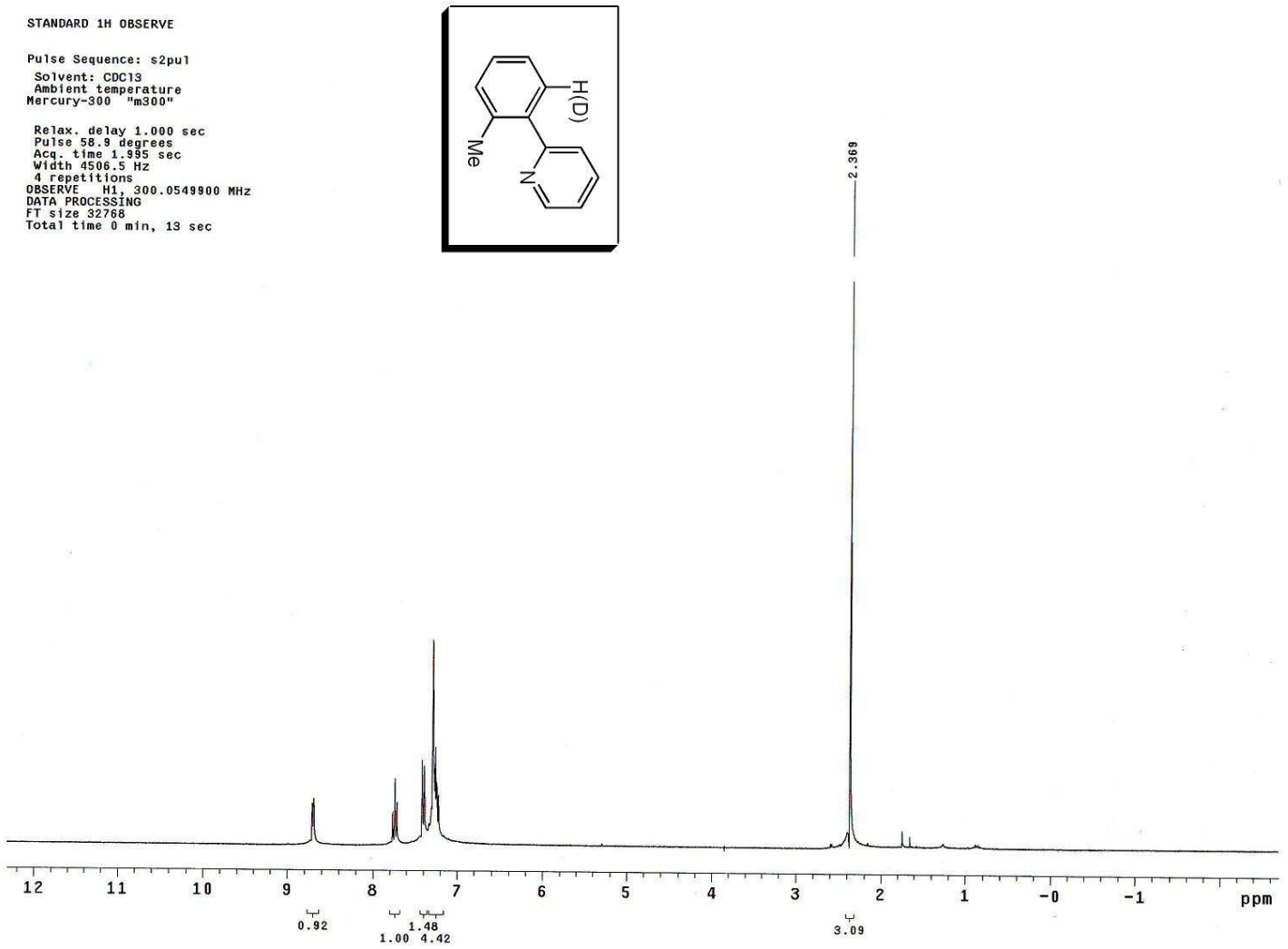
File: CARBON

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 23.5 C / 296.6 K

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
1160 repetitions
OBSERVE C13, 100.6107554 MHz
DECOUPLE H1, 400.1239934 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr



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Ambient temperature
Mercury-300 "m300"
Relax. delay 1.000 sec
Pulse 58.9 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
4 repetitions
OBSERVE H1, 300.0549900 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 13 sec
H(D)
Me
N
2.369
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
0.92
1.00
1.48
4.42
3.09

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300 "m300"

Relax. delay 1.500 sec
Pulse 58.2 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
984 repetitions
OBSERVE C13, 75.4488895 MHz
DECOUPLE H1, 300.0564325 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 13 hr, 31 min, 10 sec