Rh(I)-Catalyzed Direct Arylation of Azines

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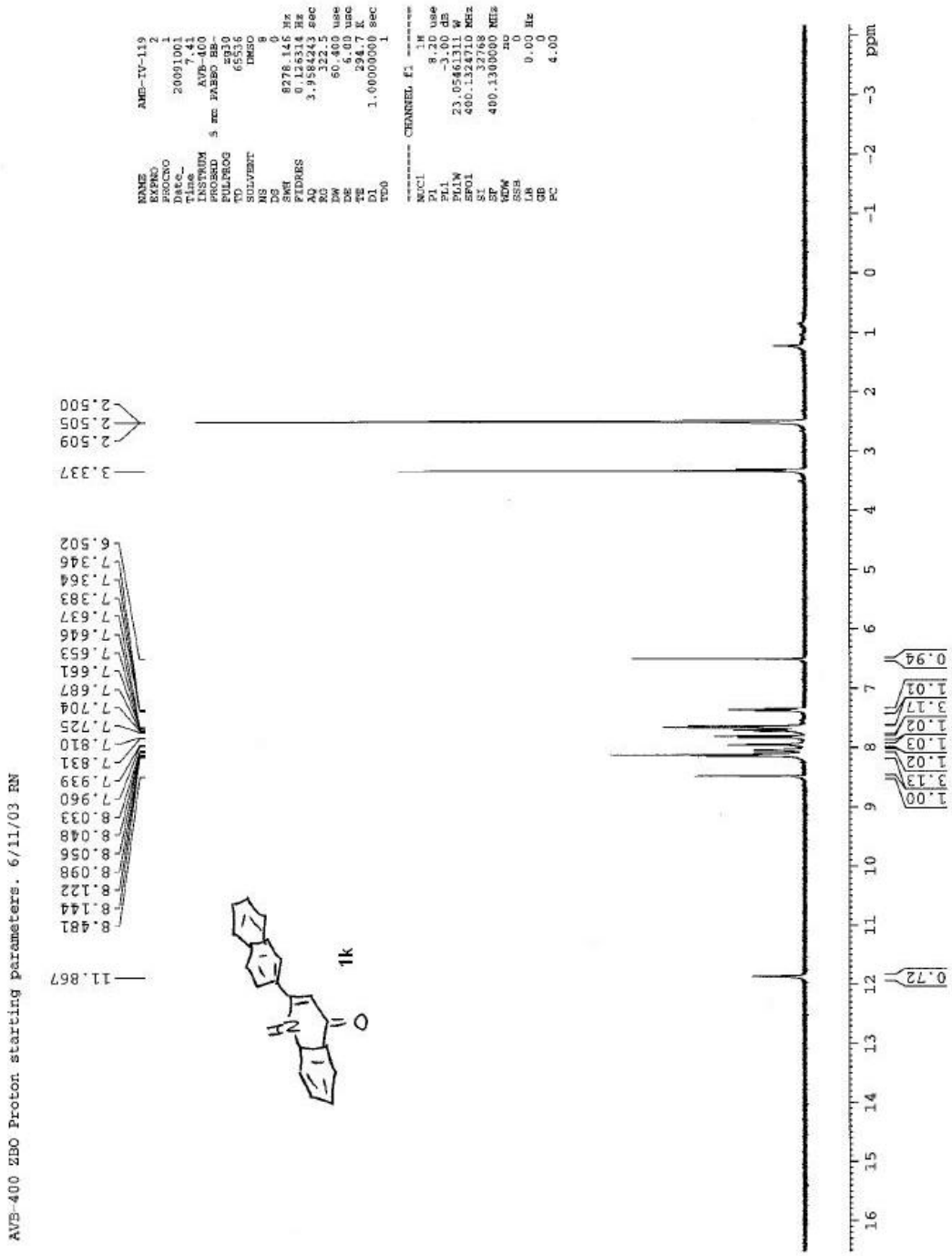
General Procedures. All reagents were degassed and handled under an inert nitrogen atmosphere using syringe and cannula techniques. Unless otherwise noted, all organic preparations were carried out in flame- or oven-dried glassware under a nitrogen atmosphere and all catalytic arylation reactions were assembled in a nitrogen-filled inert atmosphere box. Flash column chromatography was carried out using hexanes/ethyl acetate or dichloromethane/methanol gradients ^{1}H NMR chemical shifts are reported in ppm relative to TMS as an internal standard ($Si(CH_3)_4$, 0.00 ppm) in $CDCl_3$ unless otherwise noted, and coupling constants are reported in Hz. Reported tabular yields are the average of at least two experimental runs; yields reported in the following experimental procedures might vary slightly from those found in the tables. Experimental procedures and full analytical data for compounds **1a – 1f**, **1h – 1j**, **1o – 1x** have been previously reported.[9]

Materials. Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. $[RhCl(CO)_2]_2$ was purchased from commercial suppliers and stored in the glove box. All liquid reagents were thoroughly degassed using three freeze-pump-thaw cycles prior to introduction to the glove box. The 1,4-dioxane and toluene were obtained were dried over alumina under a nitrogen atmosphere prior to introduction to the glove box.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PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
11.867
8.481
8.144
8.122
8.098
8.056
8.048
8.033
7.960
7.939
7.831
7.810
7.725
7.704
7.687
7.661
7.653
7.646
7.637
7.383
7.364
7.346
6.502
2.509
2.505
2.500
3.337
1k
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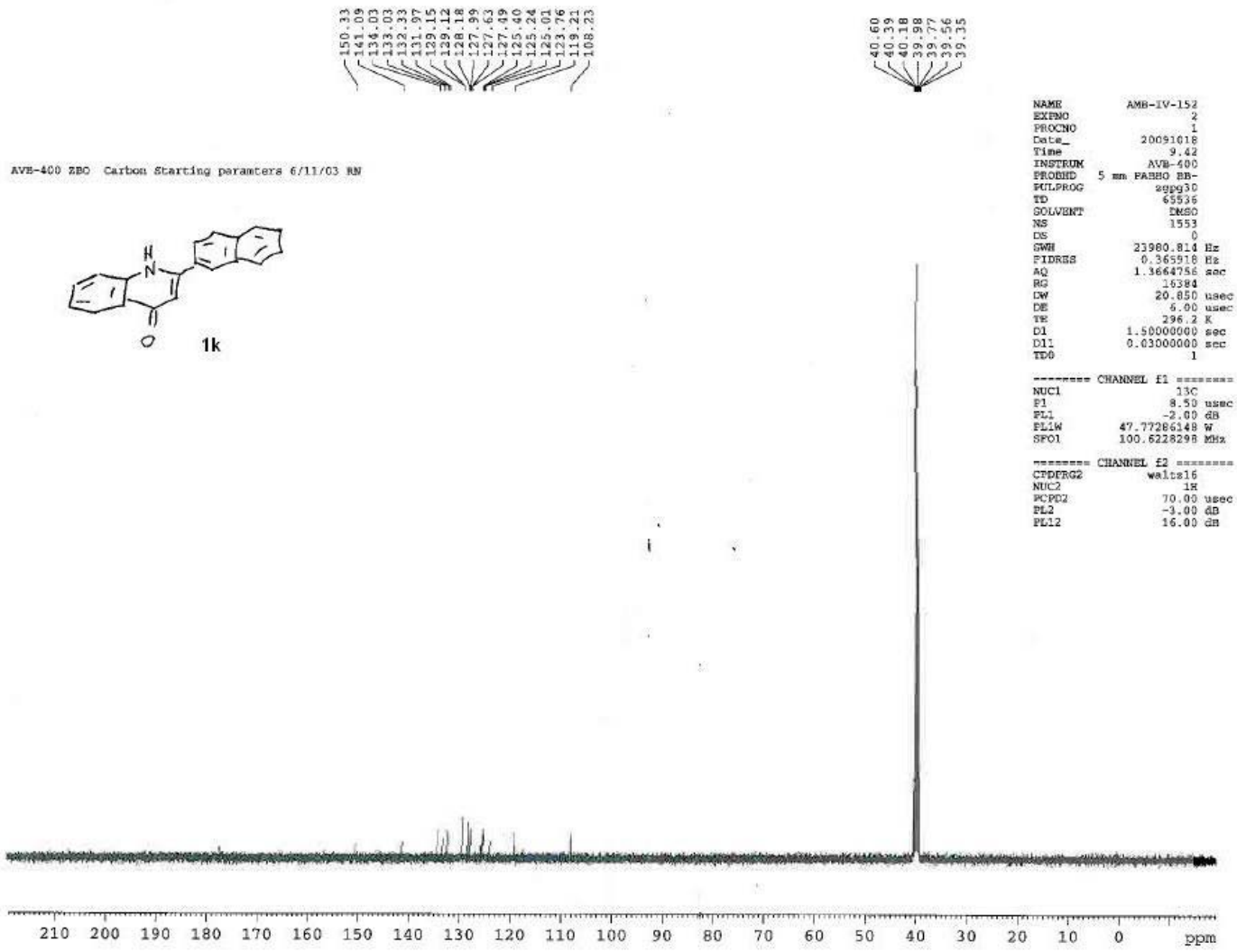
1.00
3.13
1.02
1.03
1.02
3.17
1.02
1.01
0.94
ppm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131.97
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128.18
127.99
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125.24
125.01
123.76
119.21
108.23
40.60
40.39
40.18
39.98
39.77
39.56
39.35
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1k
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PROCNO 1
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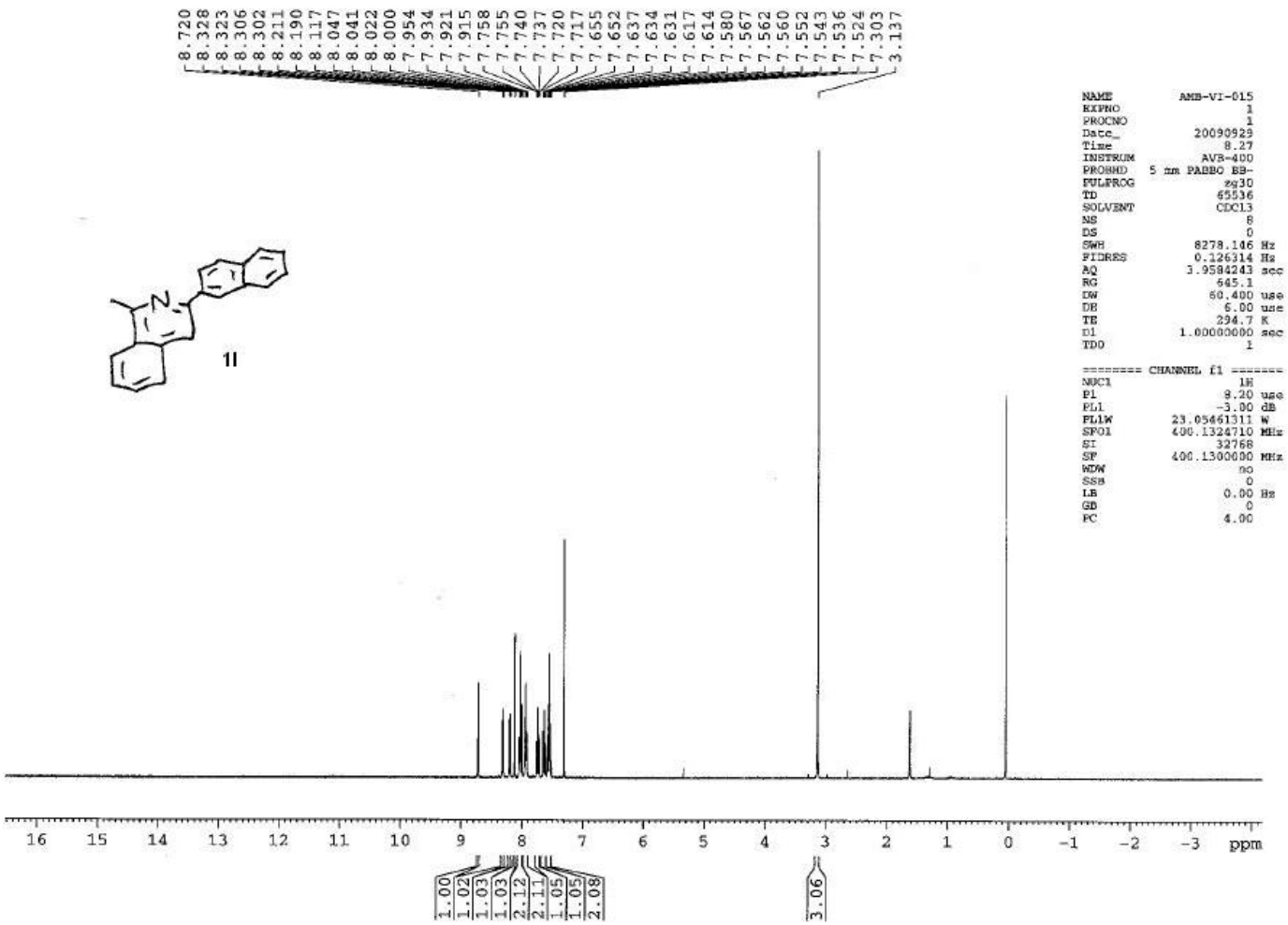
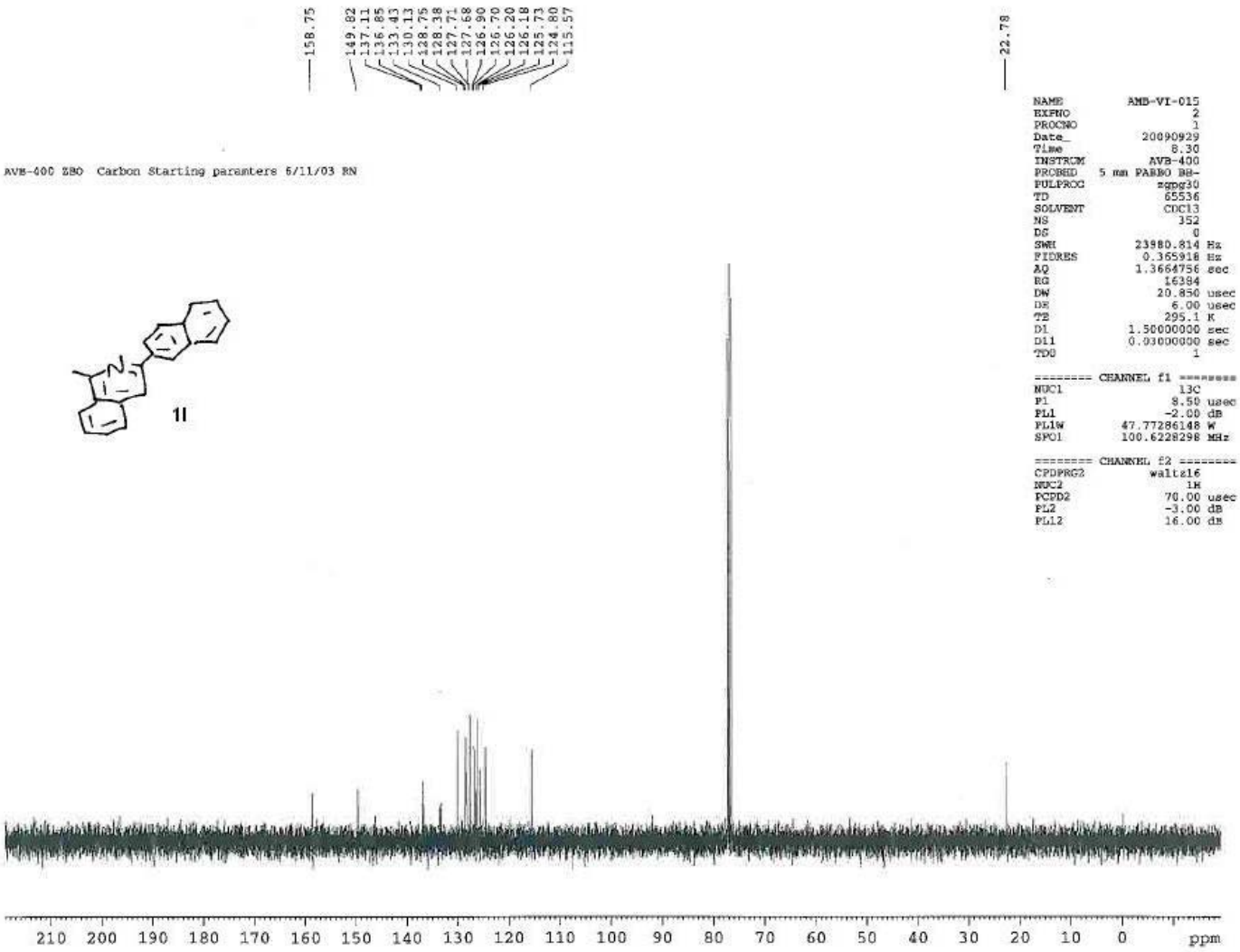
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1553
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 296.2 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

AVB-400 ZBO Proton starting parameters. 6/11/03 RN

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126.20
126.18
125.73
124.80
115.57
22.78
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11
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PROCNO 1
Date_ 20090929
Time 8.30
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 352
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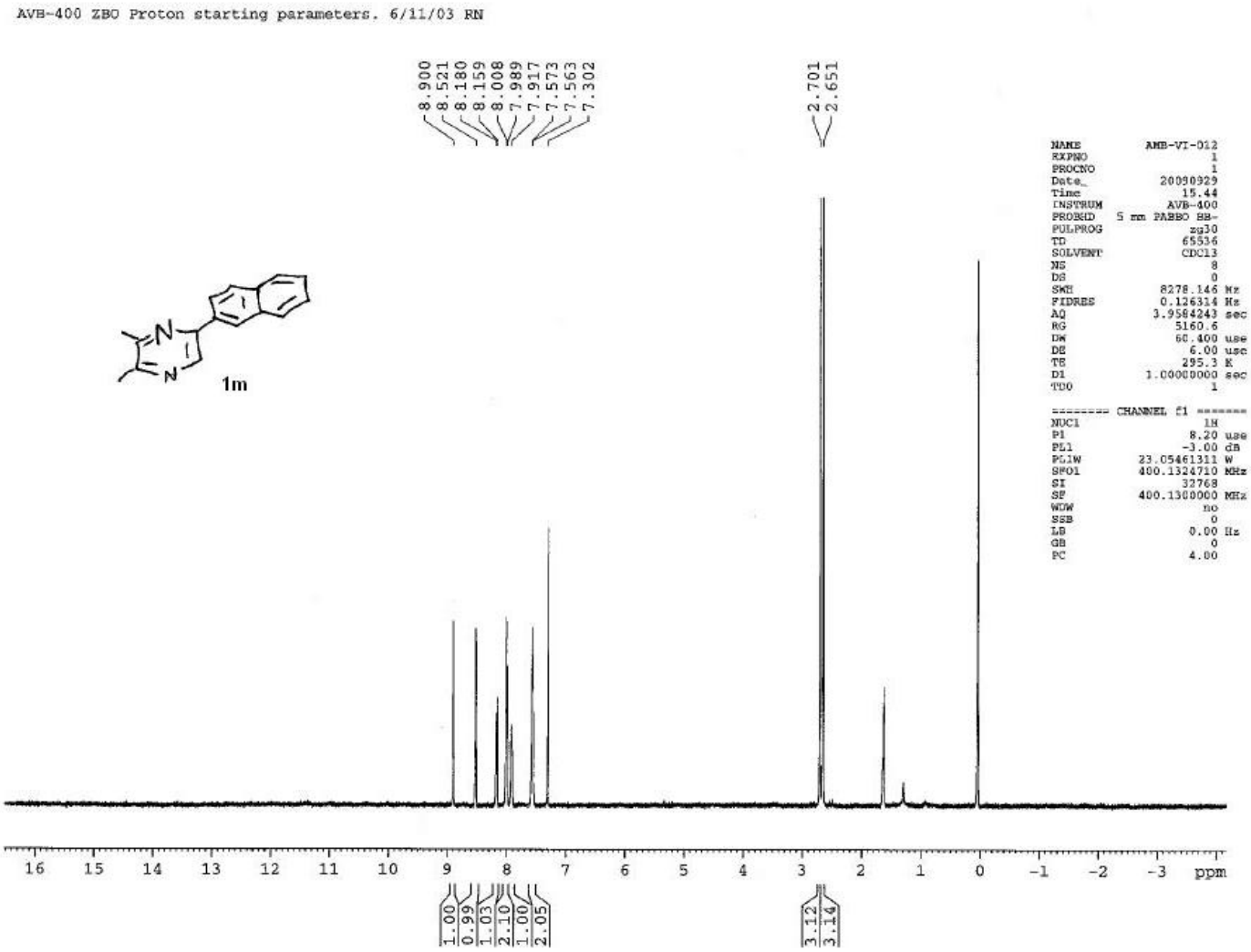
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.1 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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8.180
8.159
8.008
7.989
7.917
7.573
7.563
7.302
2.701
2.651
1m
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EXPNO 1
PROCNO 1
Date_ 20090929
Time 15.44
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 5160.6
DW 60.400 use
DE 6.00 use
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 1H
P1 8.20 use
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 ppm
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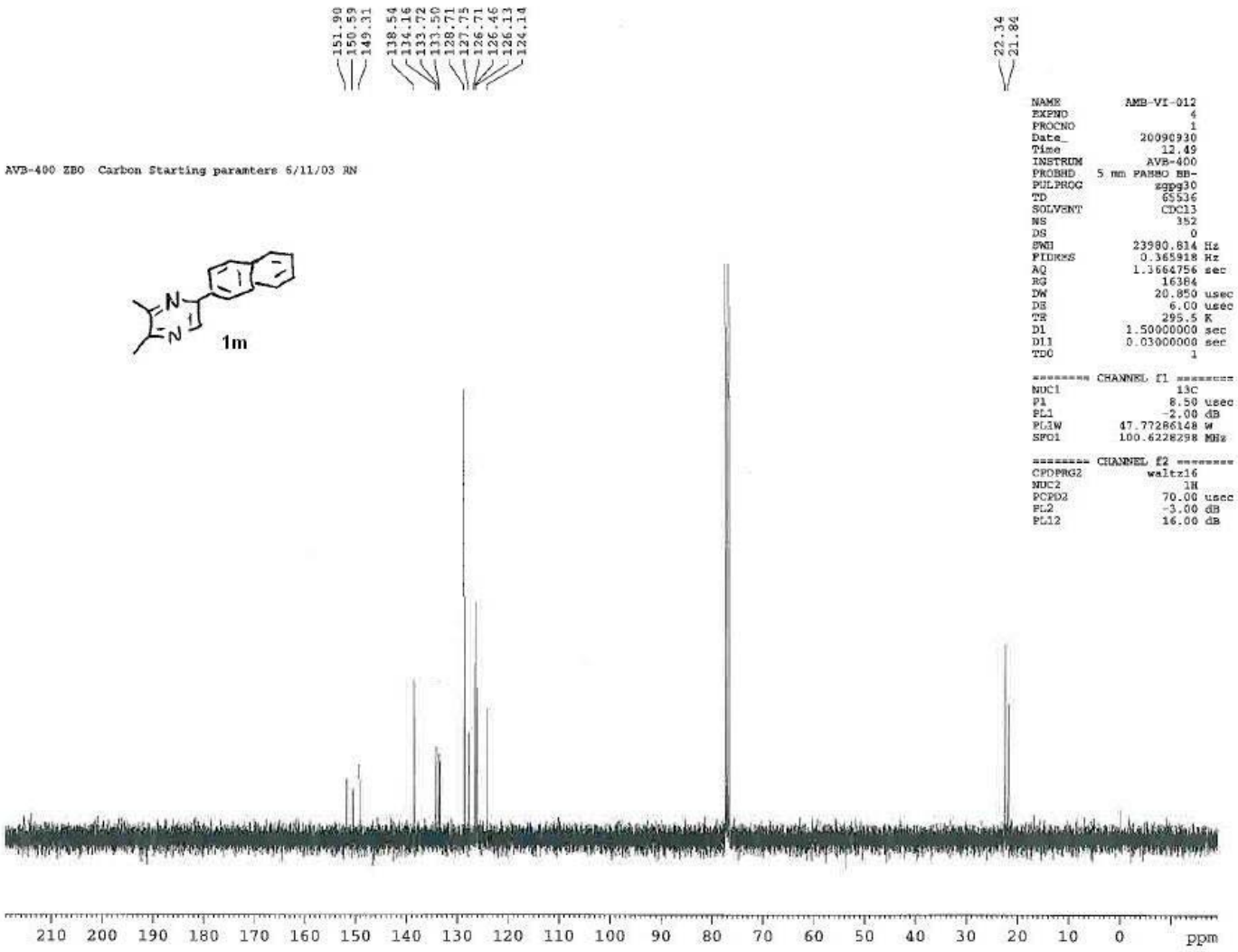
0.99
1.03
2.10
1.00
2.05
3.12
3.14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134.16
133.72
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128.71
127.75
126.71
126.46
126.13
124.14
22.34
21.84
AVB-400 ZBO Carbon Starting paramters 6/11/03 RN
1m
NAME AMB-VI-012
EXPNO 4
PROCNO 1
Date_ 20090930
Time 12.49
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 352
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.5 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

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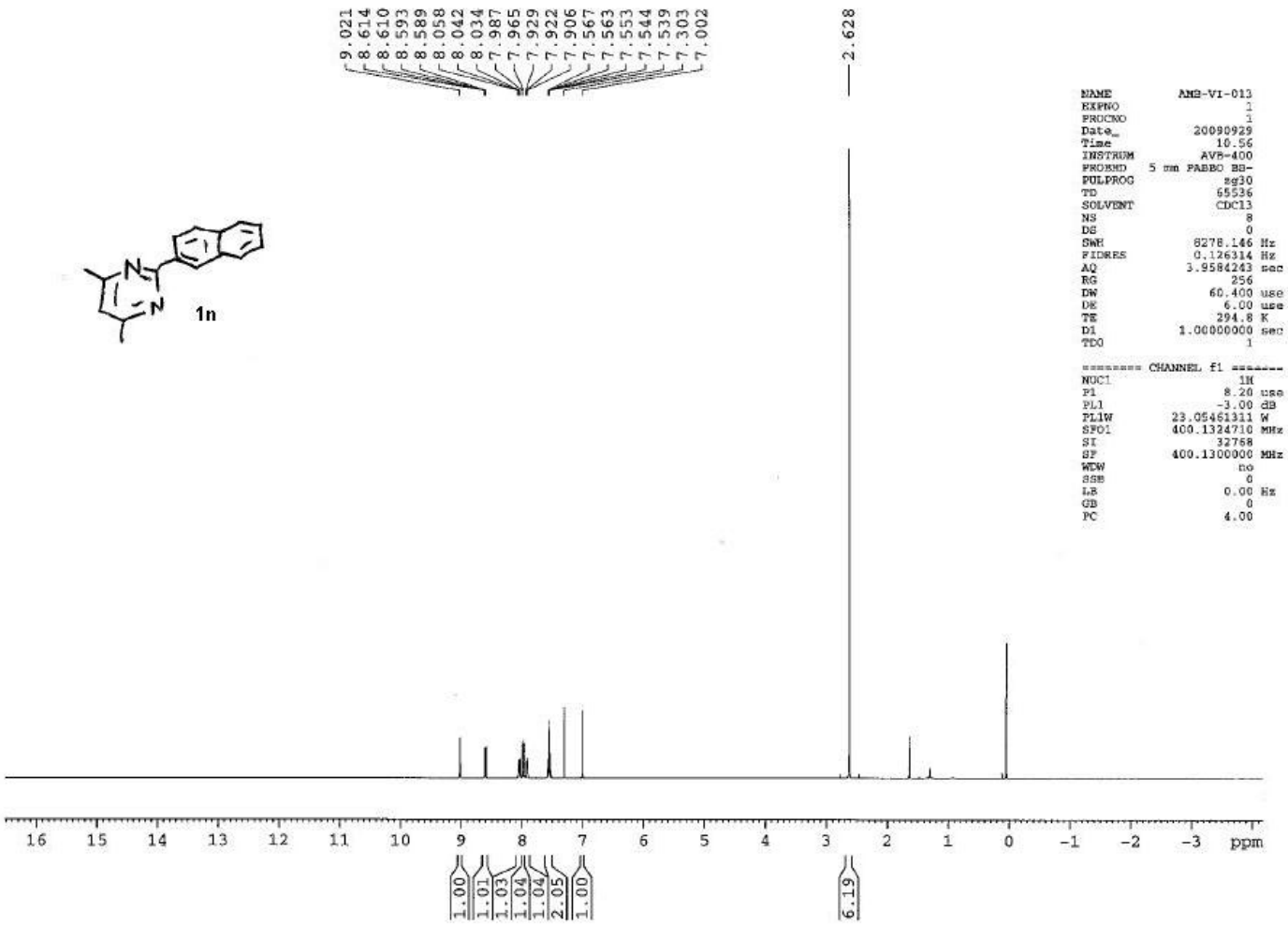

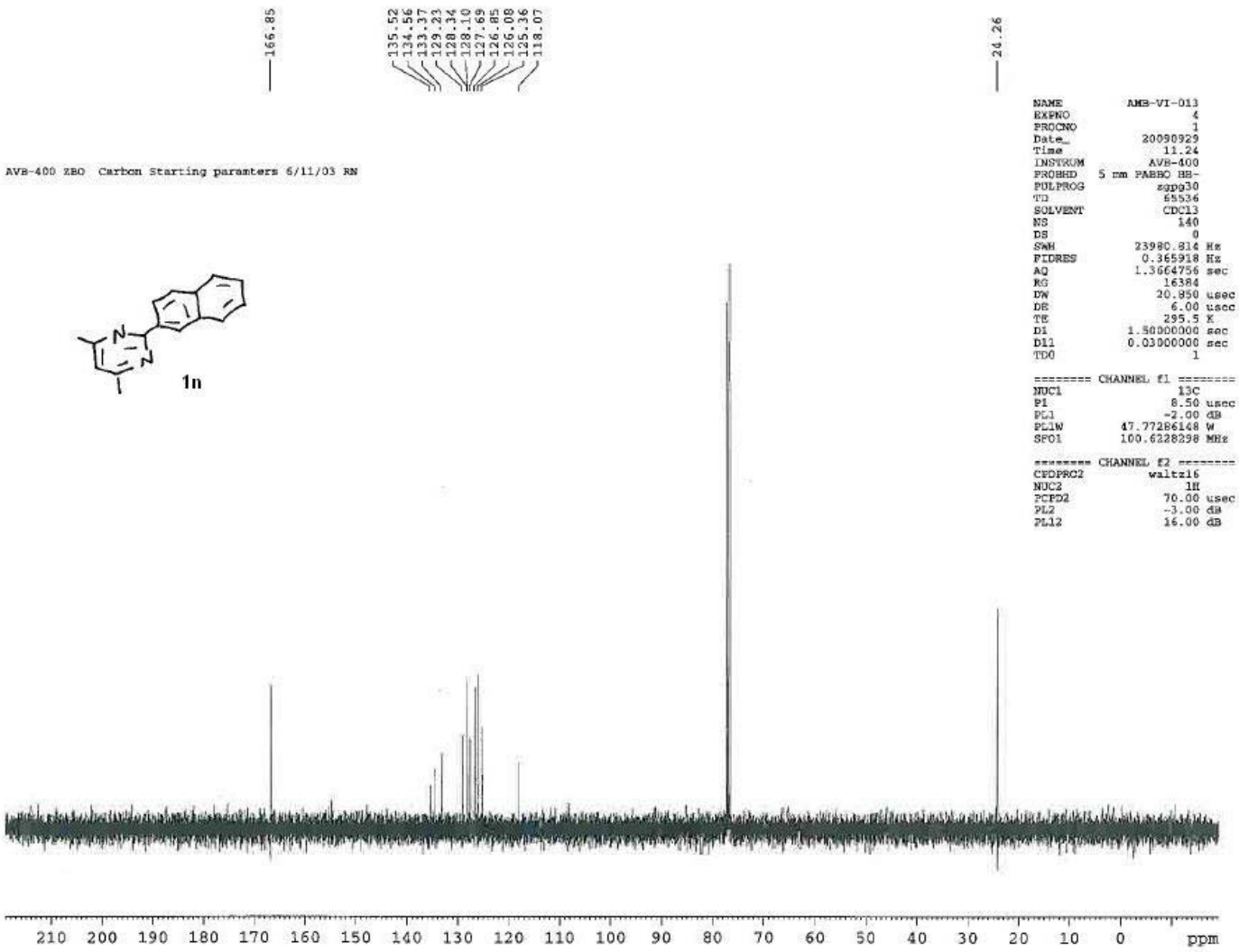
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133.37
129.23
128.34
128.10
127.69
126.85
126.08
125.36
118.07
24.26
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1n
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EXPNO 4
PROCNO 1
Date_ 20090929
Time 11.24
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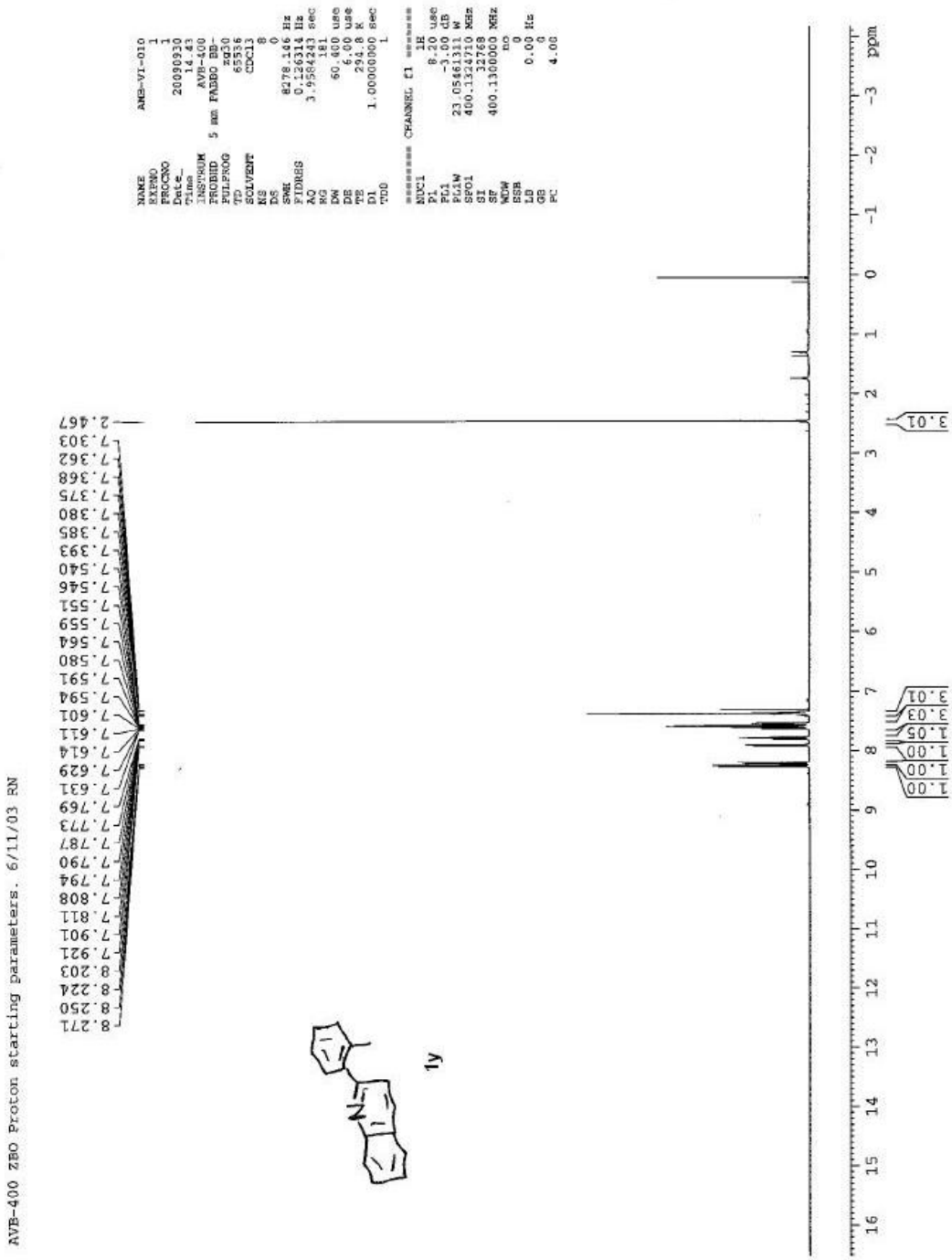
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 140
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.5 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.00 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
2.467
7.303
7.362
7.368
7.375
7.380
7.385
7.393
7.540
7.546
7.551
7.559
7.564
7.580
7.591
7.594
7.601
7.611
7.614
7.629
7.631
7.769
7.773
7.787
7.790
7.794
7.808
7.811
7.901
7.921
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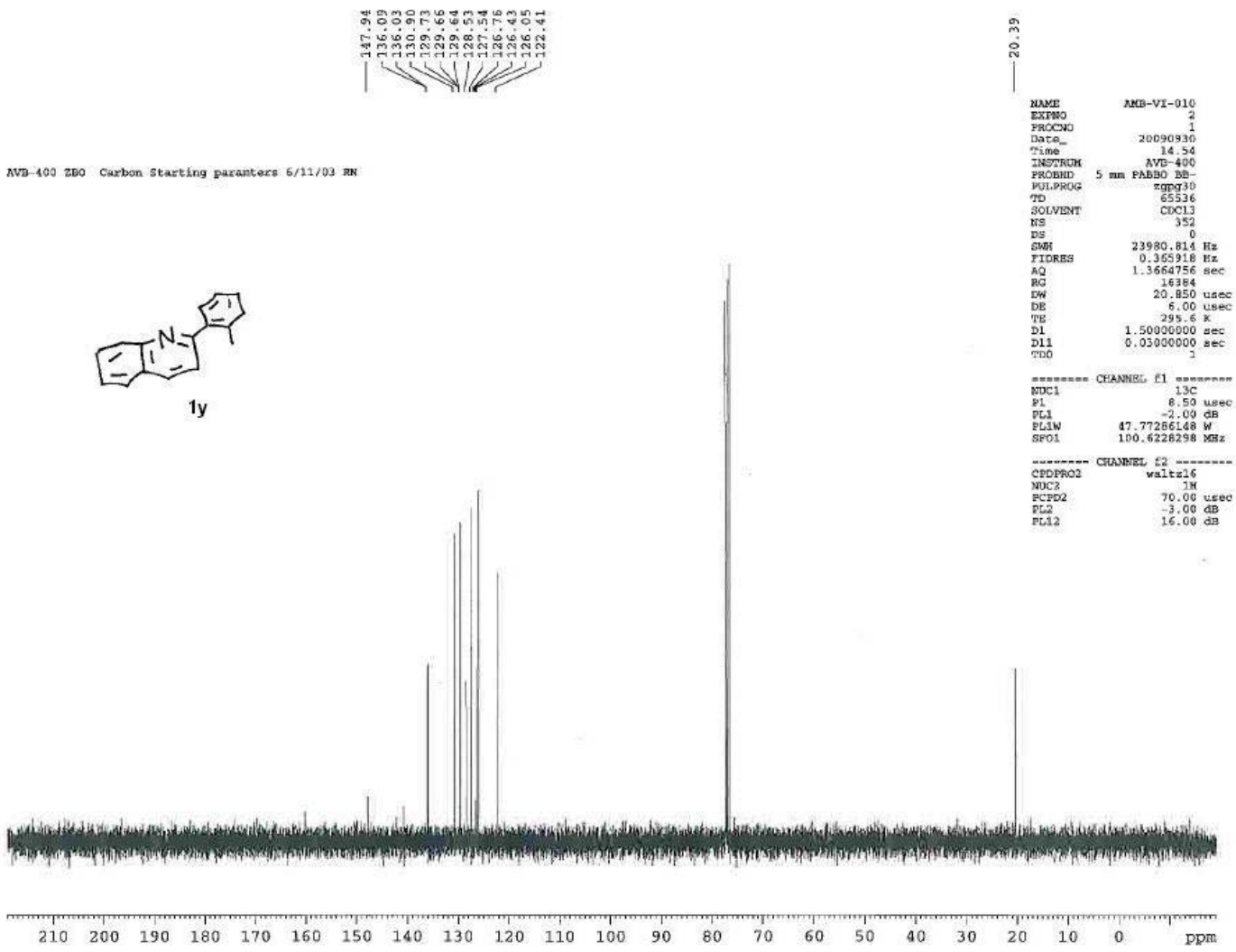
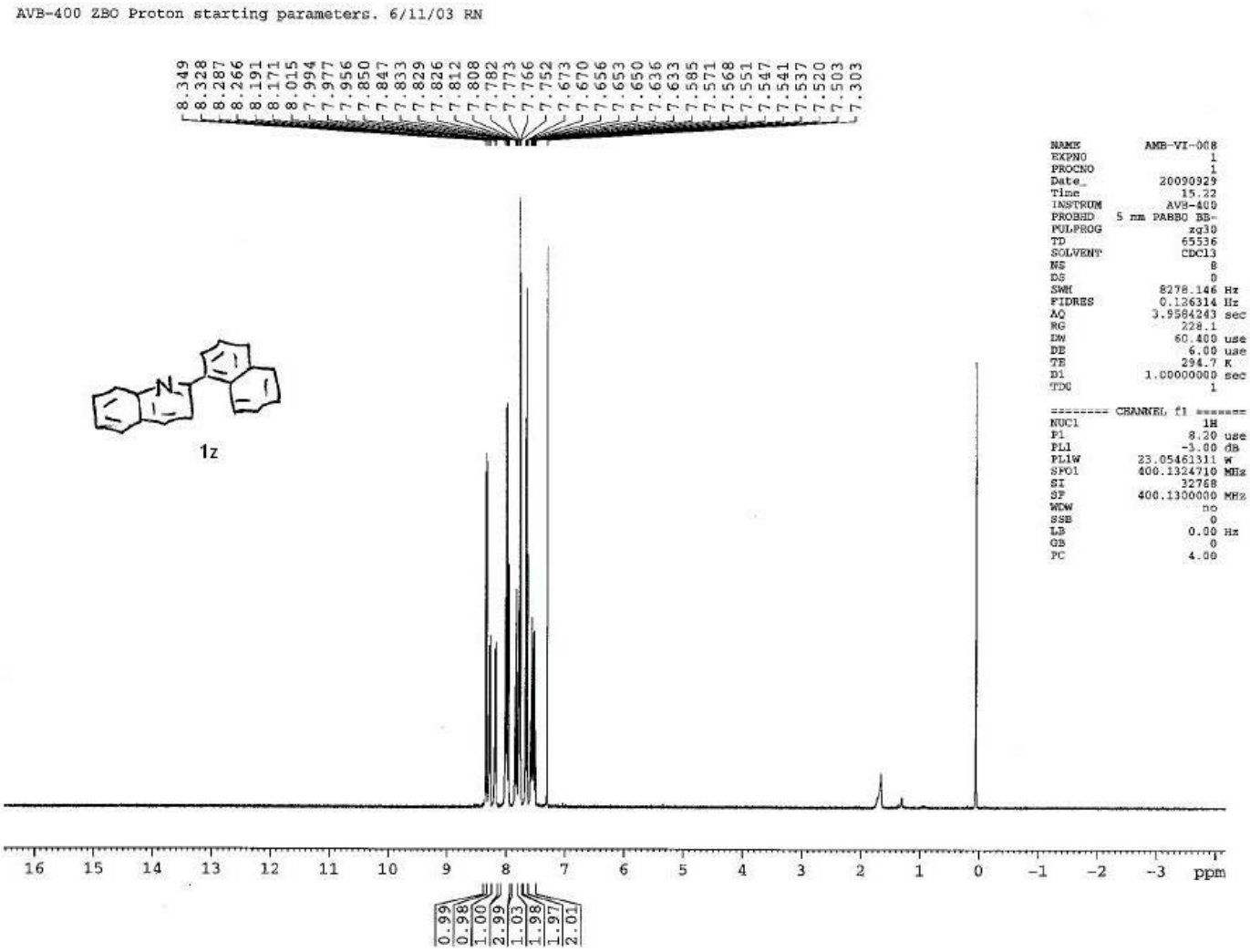
1.00
1.00
3.05
3.03
3.01
ppm

1y


147.94
136.09
136.03
130.90
129.73
129.66
129.64
128.53
127.54
126.76
126.43
126.05
122.41
20.39
AVB-400 ZBO Carbon Starting paramters 6/11/03 RN
1y
NAME AMB-VI-010
EXPNO 2
PROCNO 1
Date_ 20090930
Time 14.54
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 352
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.6 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRO2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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8.328
8.287
8.266
8.191
8.171
8.015
7.994
7.977
7.956
7.850
7.847
7.833
7.829
7.826
7.812
7.808
7.782
7.773
7.766
7.752
7.673
7.670
7.656
7.653
7.650
7.636
7.633
7.585
7.571
7.568
7.551
7.547
7.541
7.537
7.520
7.503
7.303
1z
NAME AMB-VI-008
EXPNO 1
PROCNO 1
Date_ 20090929
Time 15.22
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 use
DE 6.00 use
TE 294.7 K
D1 1.00000000 sec
TDO 1
======== CHANNEL f1 =======
NUC1 1H
P1 8.20 use
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 ppm
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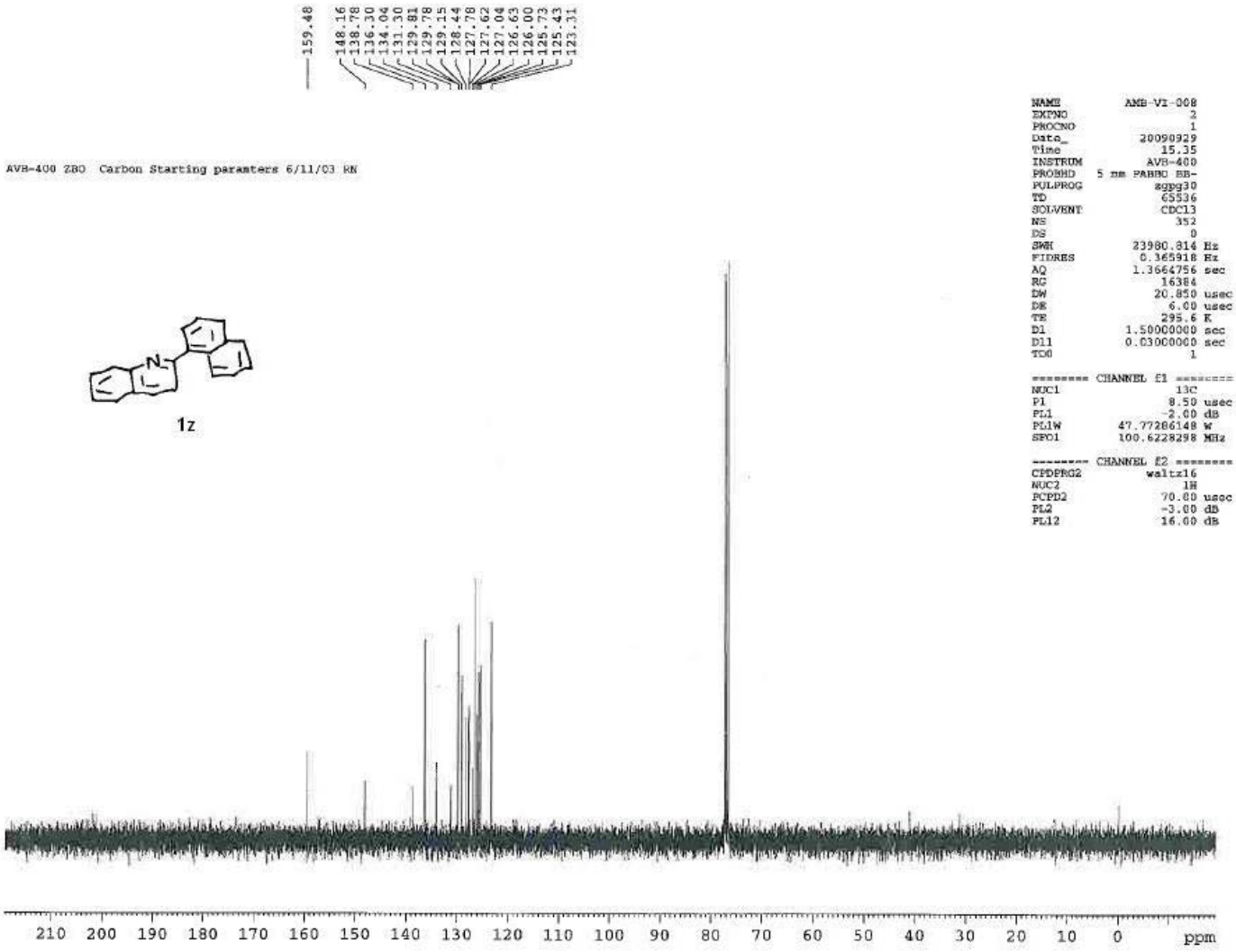
0.98
1.00
2.99
1.03
1.98
1.97
2.01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134.04
131.30
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129.78
129.15
128.44
127.78
127.62
127.04
126.63
126.00
125.73
125.43
123.31
AVB-400 ZBO Carbon Starting paramters 6/11/03 RN
1z
NAME AMB-VI-008
EXPNO 2
PROCNO 1
Date_ 20090929
Time 15.35
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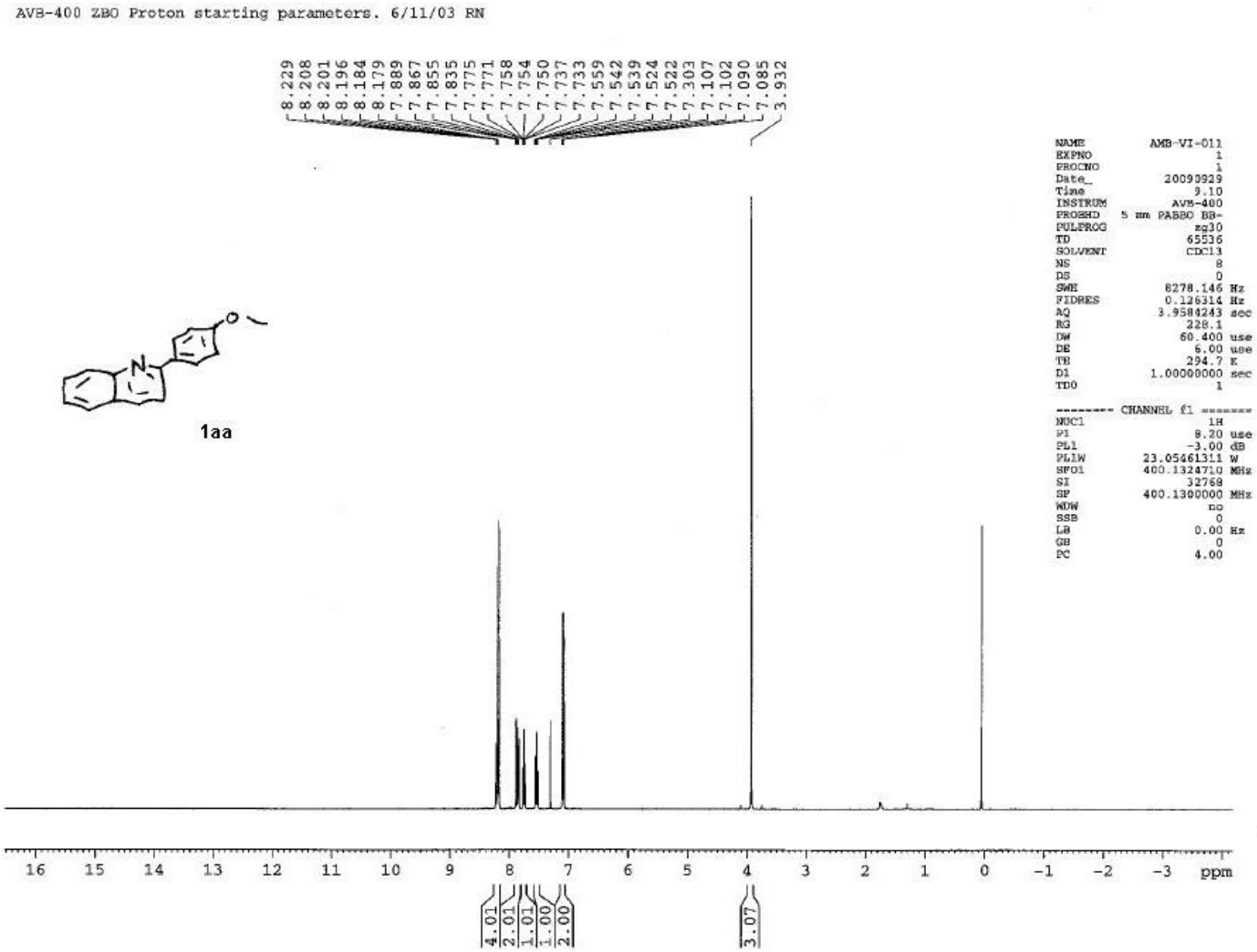
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 352
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.6 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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8.201
8.196
8.184
8.179
7.889
7.867
7.855
7.835
7.775
7.771
7.758
7.754
7.750
7.737
7.733
7.559
7.542
7.539
7.524
7.522
7.303
7.107
7.102
7.090
7.085
3.932
1aa
NAME AMB-VI-011
EXPNO 1
PROCNO 1
Date_ 20090929
Time 9.10
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 use
DE 6.00 use
TE 294.7 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 8.20 use
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
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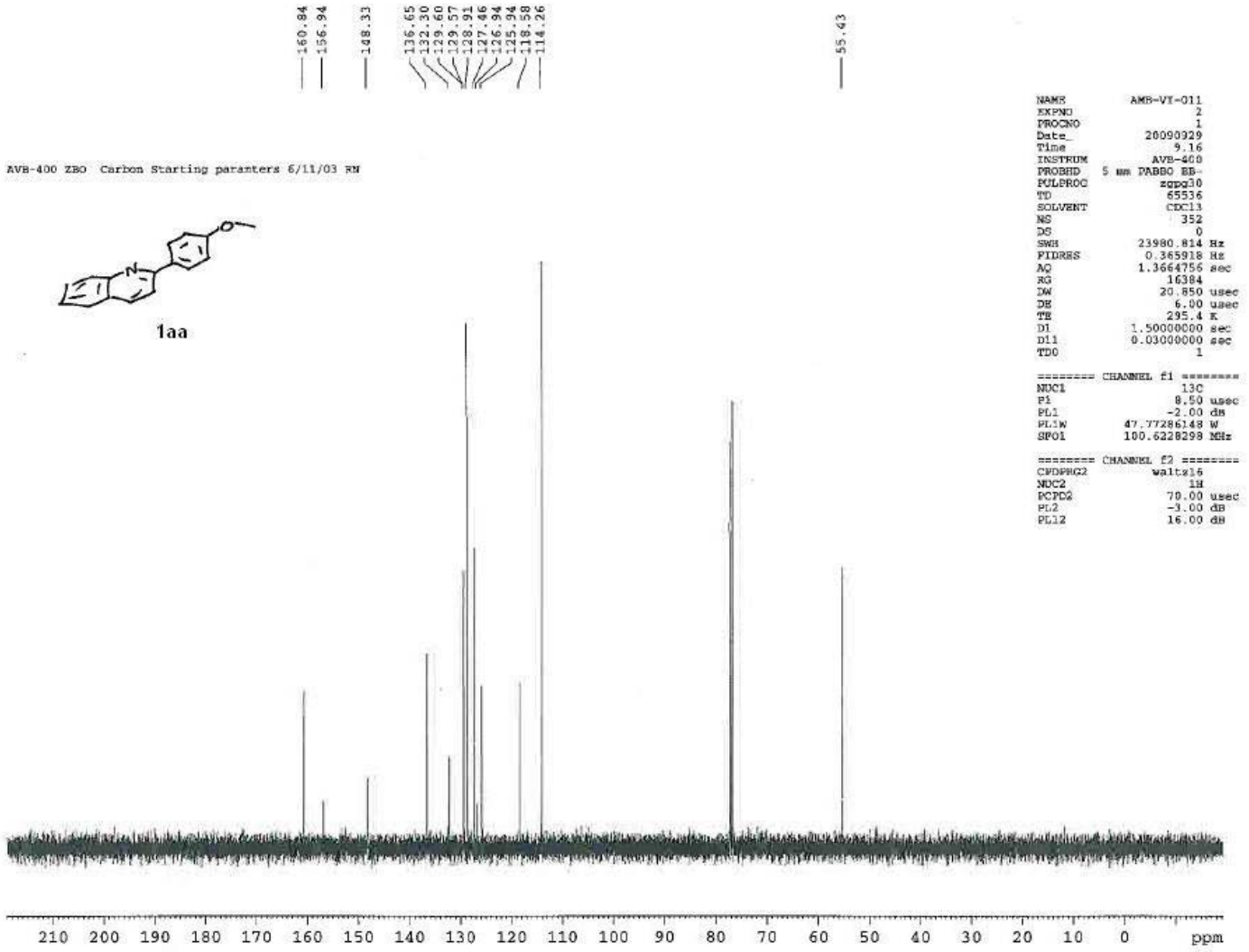
4.01
2.01
1.01
1.00
2.00
3.07


160.84
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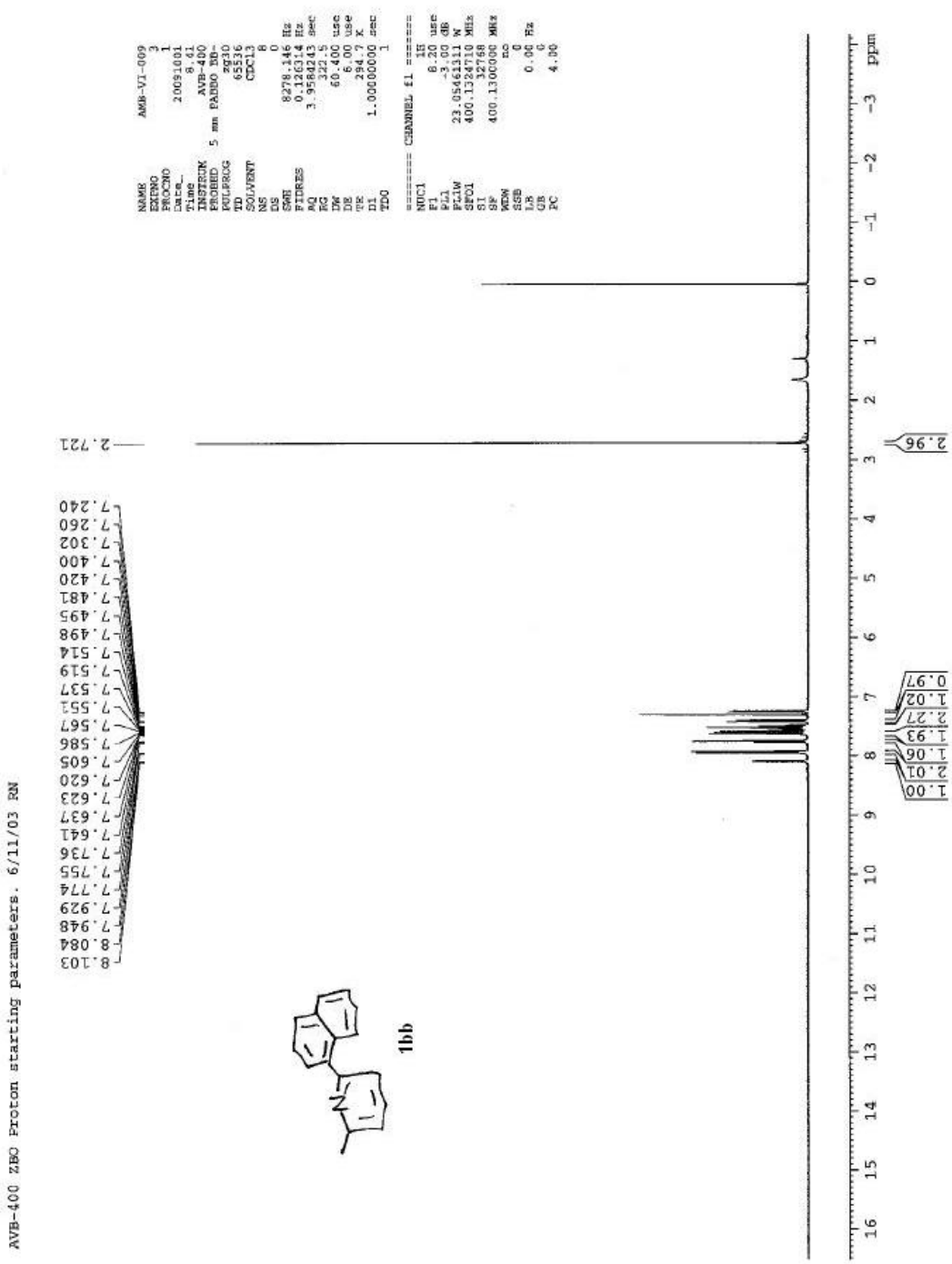
136.65
132.30
129.60
129.57
128.91
127.46
126.94
125.94
119.58
114.26
55.43
AVB-400 ZBO Carbon Starting paramters 6/11/03 RN
1aa
NAME AMB-VI-011
EXPNO 2
PROCNO 1
Date_ 20090329
Time 9.16
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 352
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.4 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


AVB-400 ZBO Proton starting parameters. 6/11/03 RN
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7.641
7.637
7.623
7.620
7.605
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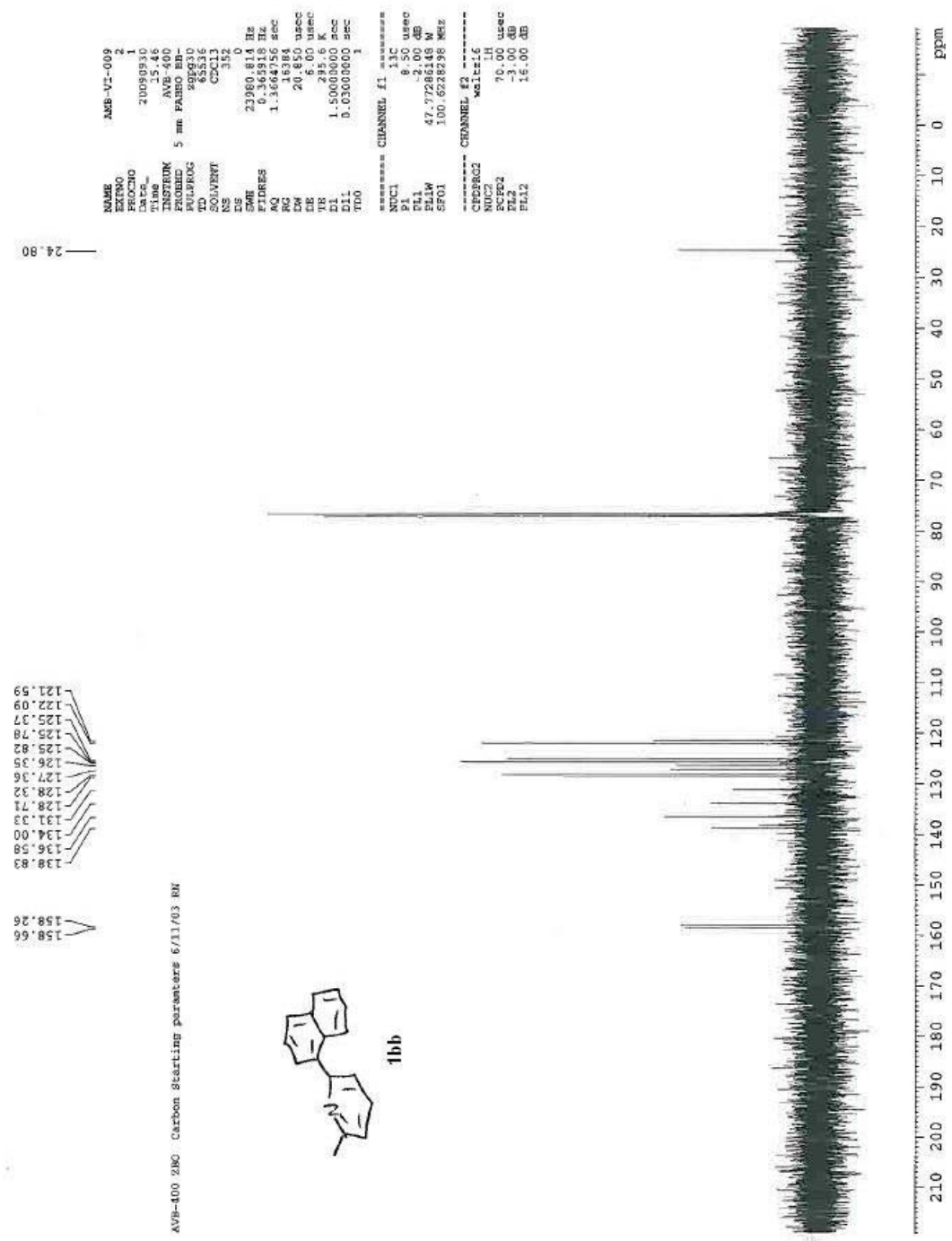
7.551
7.537
7.519
7.514
7.498
7.495
7.481
7.420
7.400
7.302
7.260
7.240
1.00
2.01
1.06
1.93
2.27
1.02
0.97
ppm


AVB-400 SBO Carbon Starting parameters 6/11/03 RN
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158.26
138.83
136.58
134.00
131.33
128.74
128.32
127.36
126.35
125.82
125.78
125.37
122.09
121.59
24.80
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EXPNO 2
PROCNO 1
Date_ 20090930
Time 15.46
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 352
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664736 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.6 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
ppm