

SUPPORTING INFORMATION

Palladium C–N Bond Formation Catalysed with Air-Stable Robust Polydentate Ferrocenylphosphines: a Comparative Study for the Efficient and Selective Coupling of Aniline Derivatives to Dichloroarene

Mélanie Platon, Julien Roger, Sylviane Royer and Jean-Cyrille Hierso

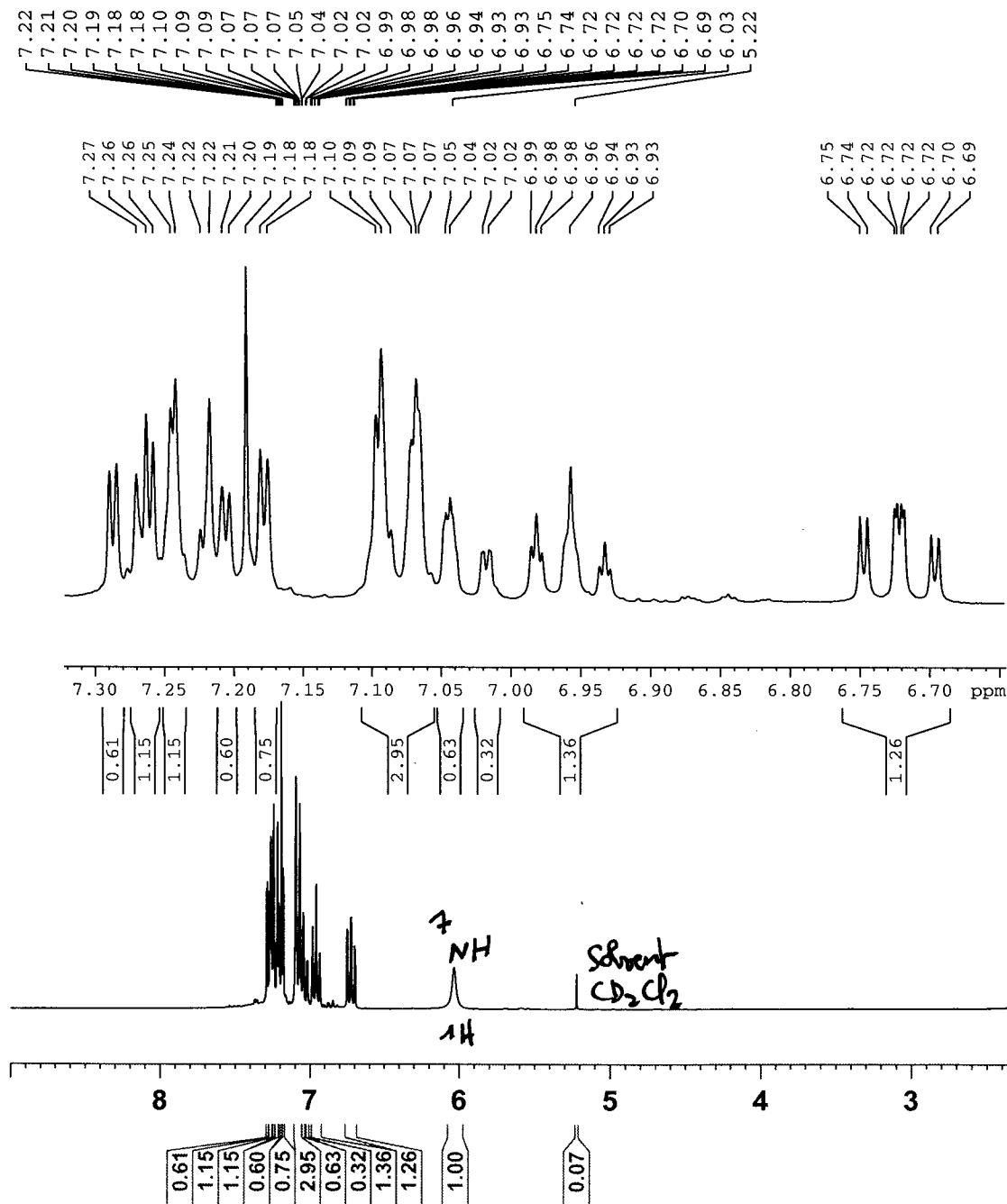
Université de Bourgogne, CNRS, UMR 6302, ICMUB, Institut de Chimie Moléculaire de l'Université de Bourgogne, 9 avenue Alain Savary, 21078 Dijon, France.

Content

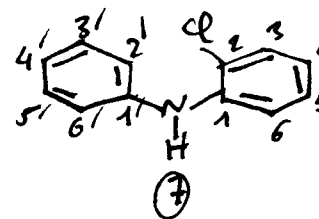
COPY of Spectroscopic data

3a	p1-3
3b	p4-8
3c	p9-12
3d	p13-17
5	p18-23
3e+3e'	p24-26
3f+3f'	p27-28
3g+3g'	p29-30
3h'	p31-34
3i+3i'	p35-38
3j+3j'	p39-42
3k+3k'	p43-46
3l'	p47-50
3m+3m'	p51-54
Ligand L5	p55-57

10mpl_1_163 brut



3a



2.09

Solvent
(CH₃)₂CO

NAME 10mpl_1_163
EXPNO 1
PROCNO 1
Date_ 20100318
Time 3.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 203.2
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300269 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10mpl_1_163 brut

206.94

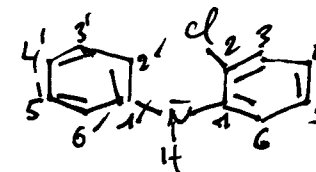
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140.29
129.73
129.43
129.32
127.41
122.63
121.50
120.57
120.36
120.17
120.08
117.78
115.57

77.48
77.26
77.05
76.63

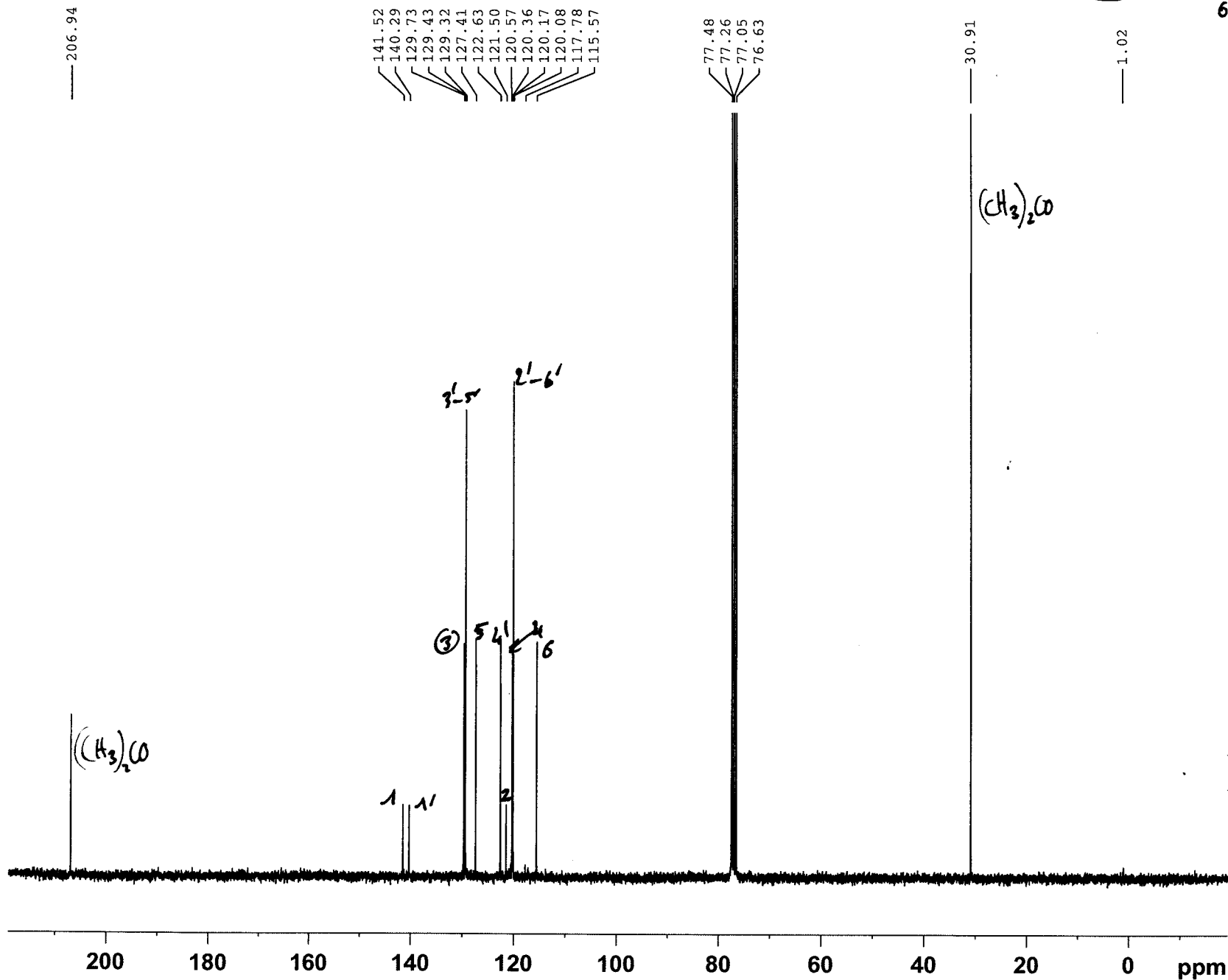
30.91

1.02

3a



2



NAME 10mpl_1_163
EXPNO 2
PROCNO 1
Date_ 20100318
Time 4.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 14596.5
DW 27.800 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 5.00 dB
PL12 27.00 dB
PL13 27.00 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10mpl_1_163 brut

— 141.52

— 140.29

— 129.73
— 129.43
— 129.32

— 127.41

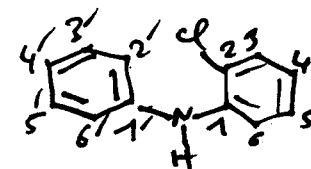
— 122.63

— 121.50
— 120.57
— 120.36
— 120.17
— 120.08

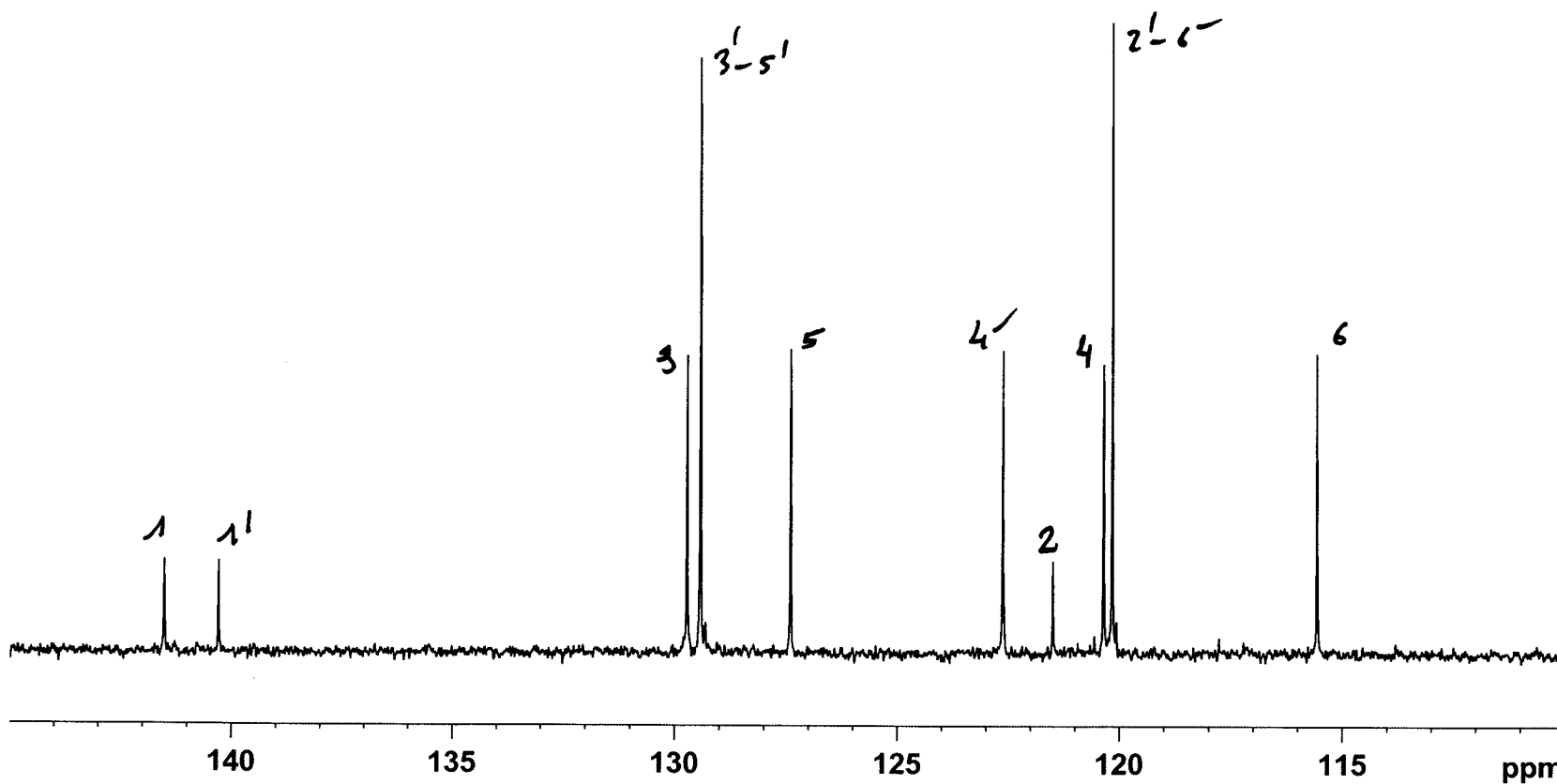
— 117.78

— 115.57

(3a)



3



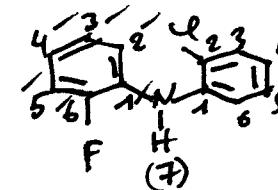
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EXPNO 2
PROCNO 1
Date_ 20100318
Time 4.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 14596.5
DW 27.800 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 5.00 dB
PL12 27.00 dB
PL13 27.00 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10mpl_2_93

(3b)

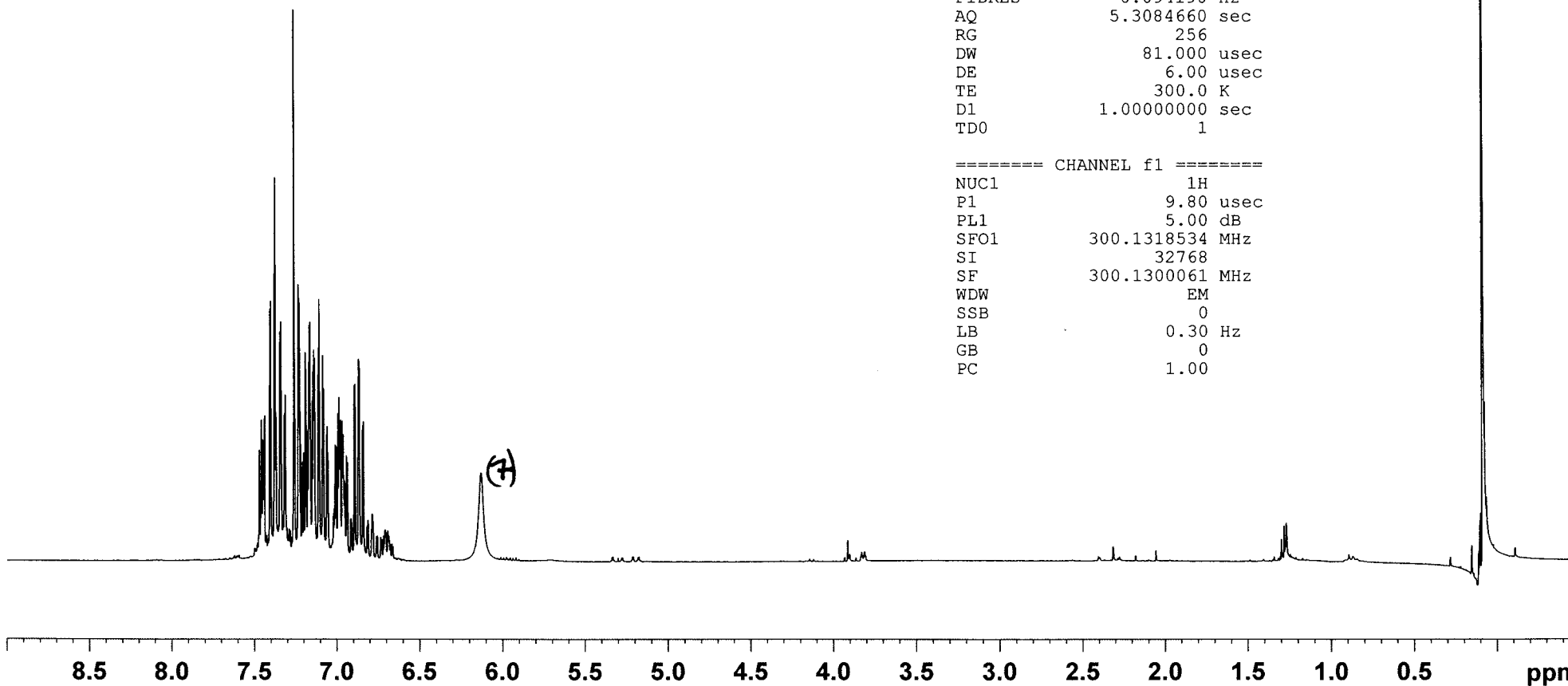


4

7.222
7.210
7.202
7.192
7.187
7.176
7.168
7.164
7.149
7.144
7.140
7.135
7.112
7.108
7.086
7.081
7.061
7.059
7.056
7.054
7.011
7.005
6.994
6.989
6.984
6.978
6.968
6.962
6.959
6.953
6.942
6.937
6.895
6.890
6.871
6.869
6.866
6.864
6.845
6.839
6.130

NAME 10mpl_2_93
EXPNO 1
PROCNO 1
Date_ 20100615
Time_ 19.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 256
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

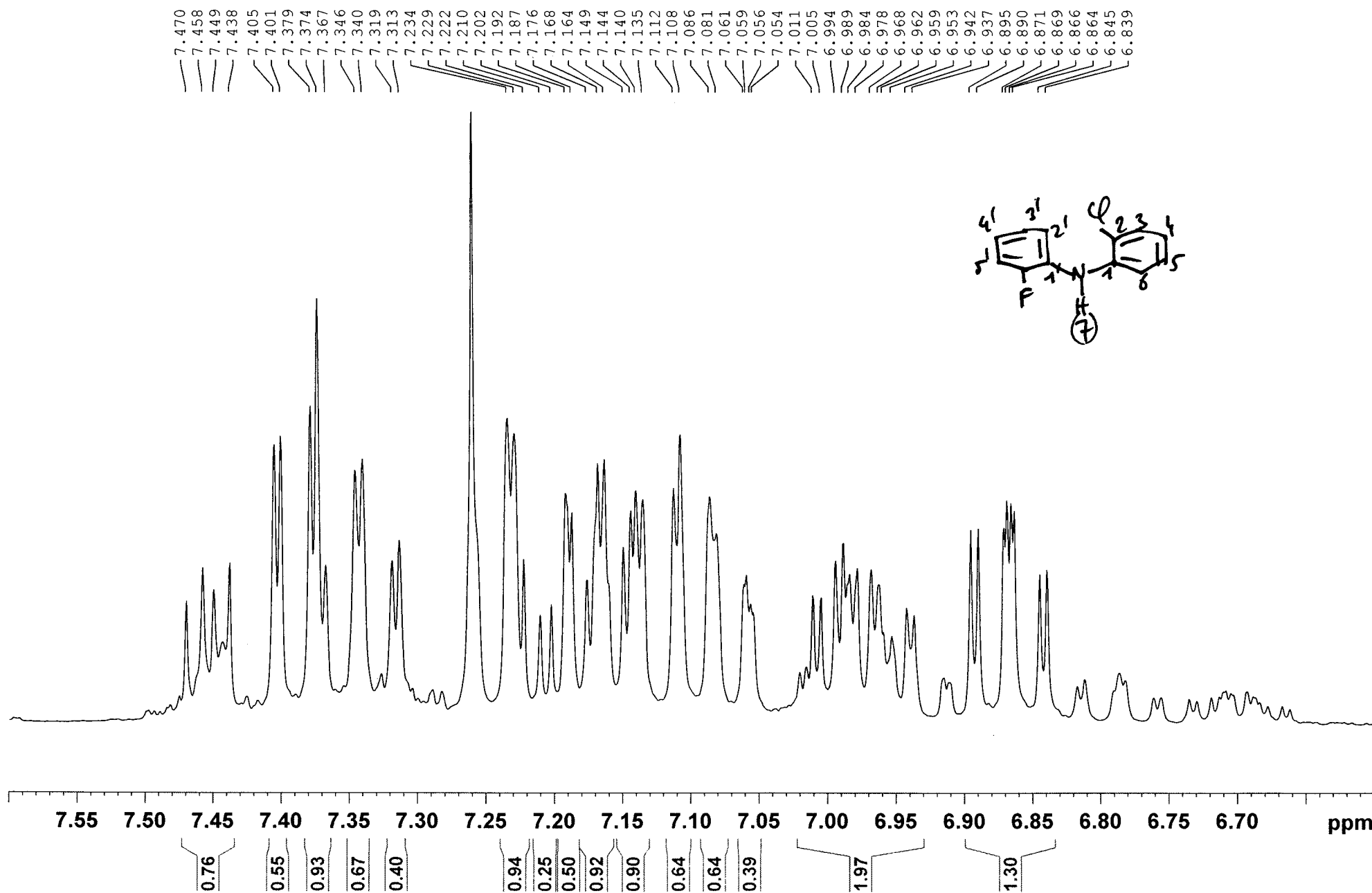


0.76
0.55
0.93
0.67
0.40
0.94
0.25
0.50
0.92
0.90
0.64
0.64
0.39
1.97
1.30
1.00

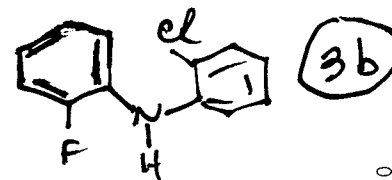
10mpl_2_93

3b

5

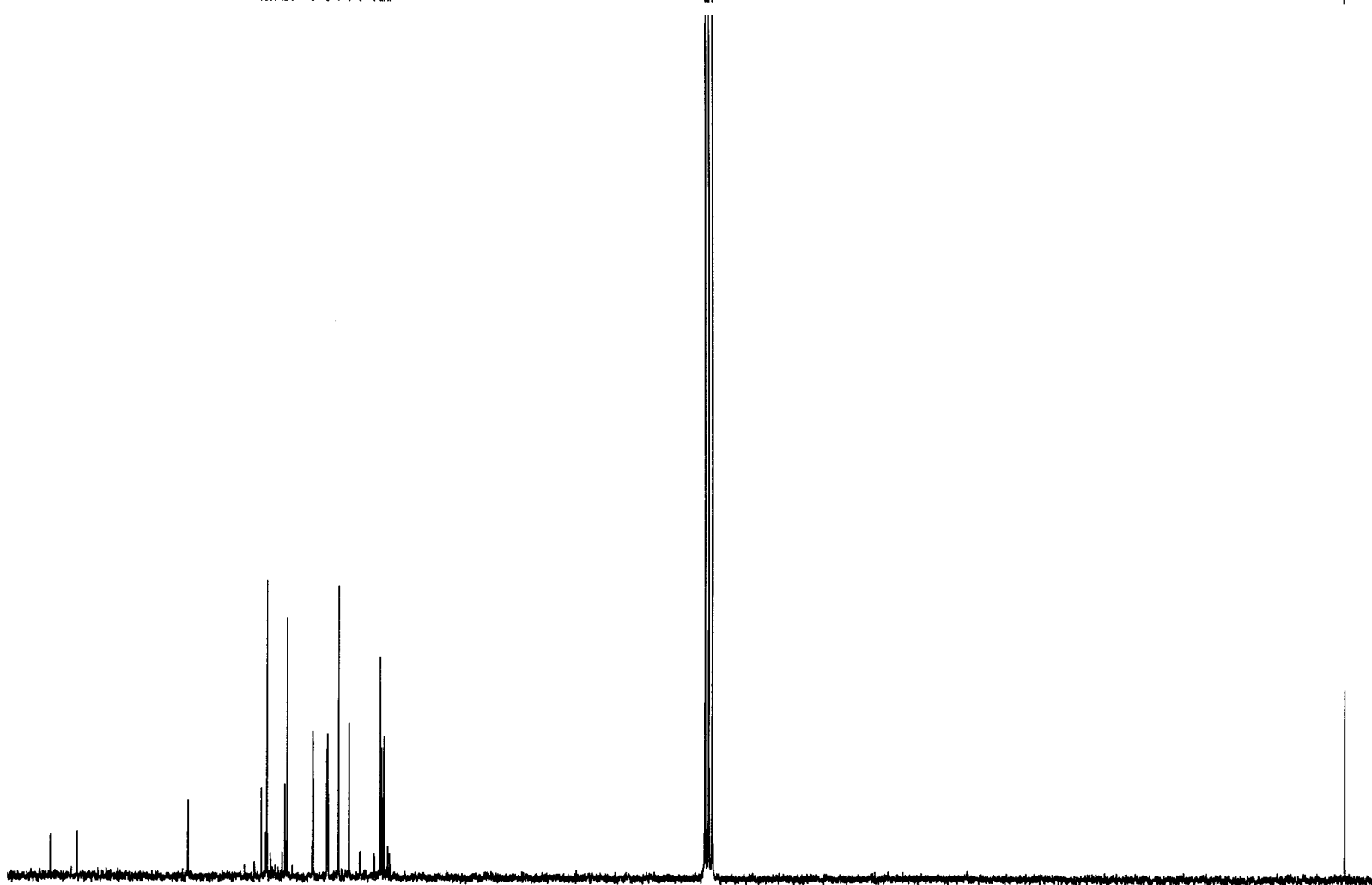


10mpl_2_93



6

129.50
128.98
128.81
128.41
128.36
127.85
127.00
126.68
126.56
126.40
125.78
123.41
123.36
123.32
123.27
121.63
121.53
121.46
120.19
118.96
118.94
117.68
117.59
115.94
115.89
115.21
115.02
114.76
114.31
114.07
76.42
76.20
76.00
75.57



0.00

NAME 10mpl_2_93
EXPNO 2
PROCNO 1
Date_ 20100617
Time_ 5.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 11585.2
DW 27.800 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 5.00 dB
PL12 27.00 dB
PL13 27.00 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4678284 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

10mpl_2_93

3b

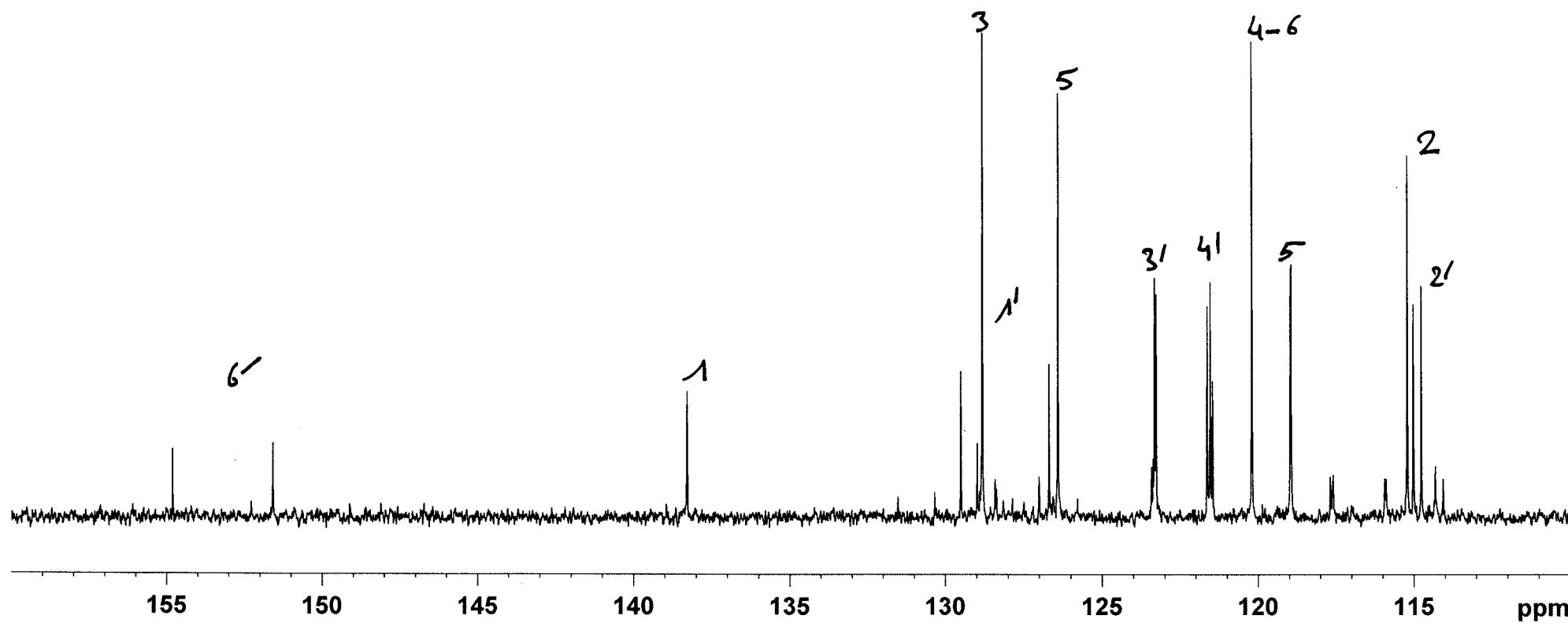
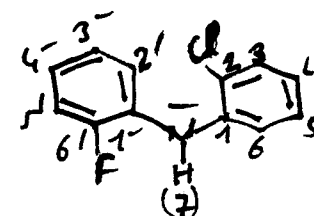
7

— 154.81

— 151.58

— 138.28

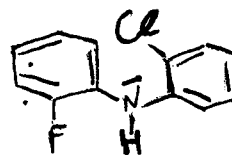
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127.85
127.00
126.68
126.56
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125.78
123.41
123.36
123.32
123.27
121.63
121.53
121.46
120.19
118.96
118.94
117.68
117.59
115.94
115.89
115.21
115.02
114.76
114.31
114.07



10mpl_2_93

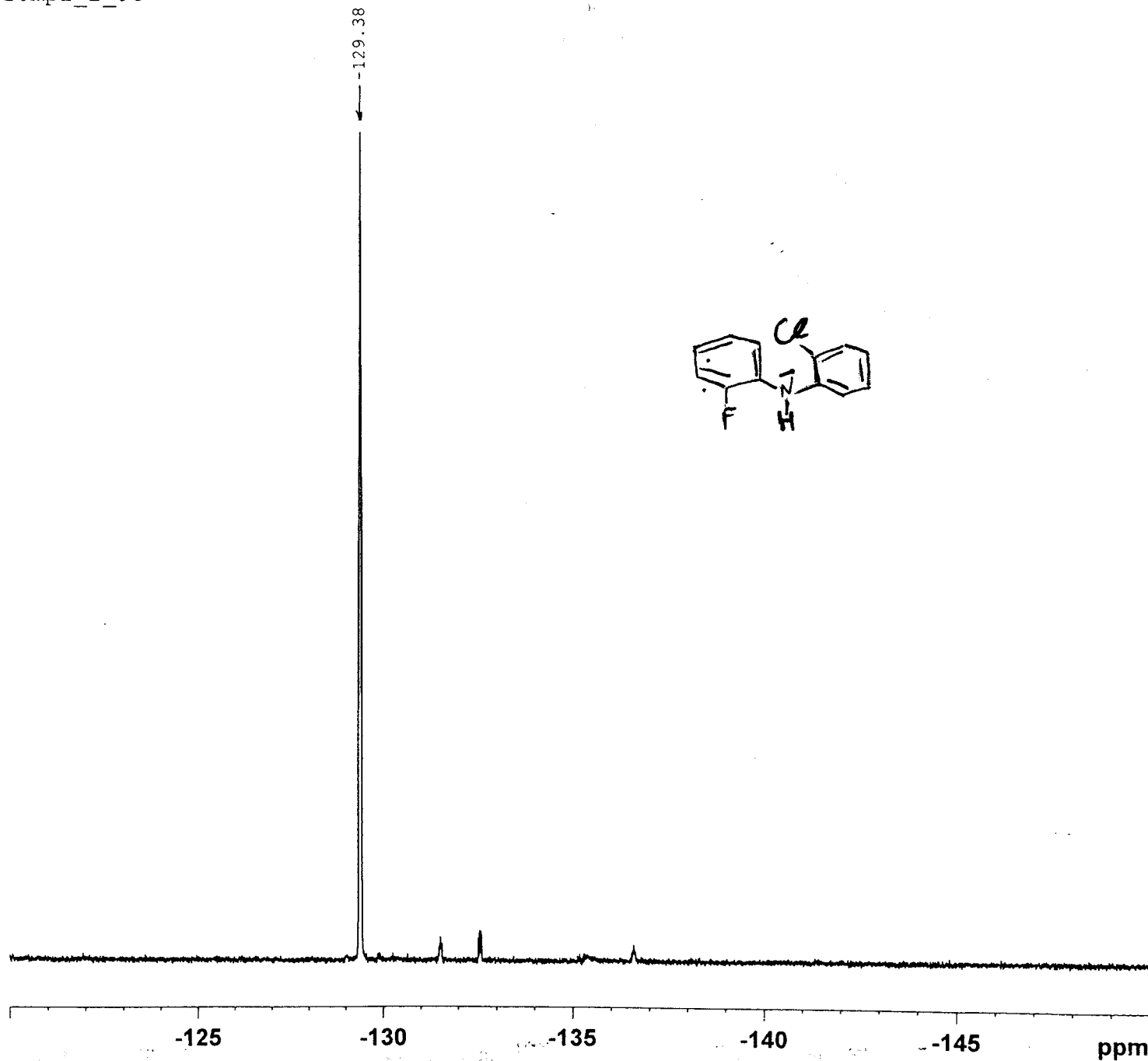
(3b)

8



NAME 10mpl_2_93
EXPNO 4
PROCNO 1
Date_ 20100617
Time_ 6.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgflqn
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 67567.570 Hz
FIDRES 0.515500 Hz
AQ 0.9699828 sec
RG 362
DW 7.400 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

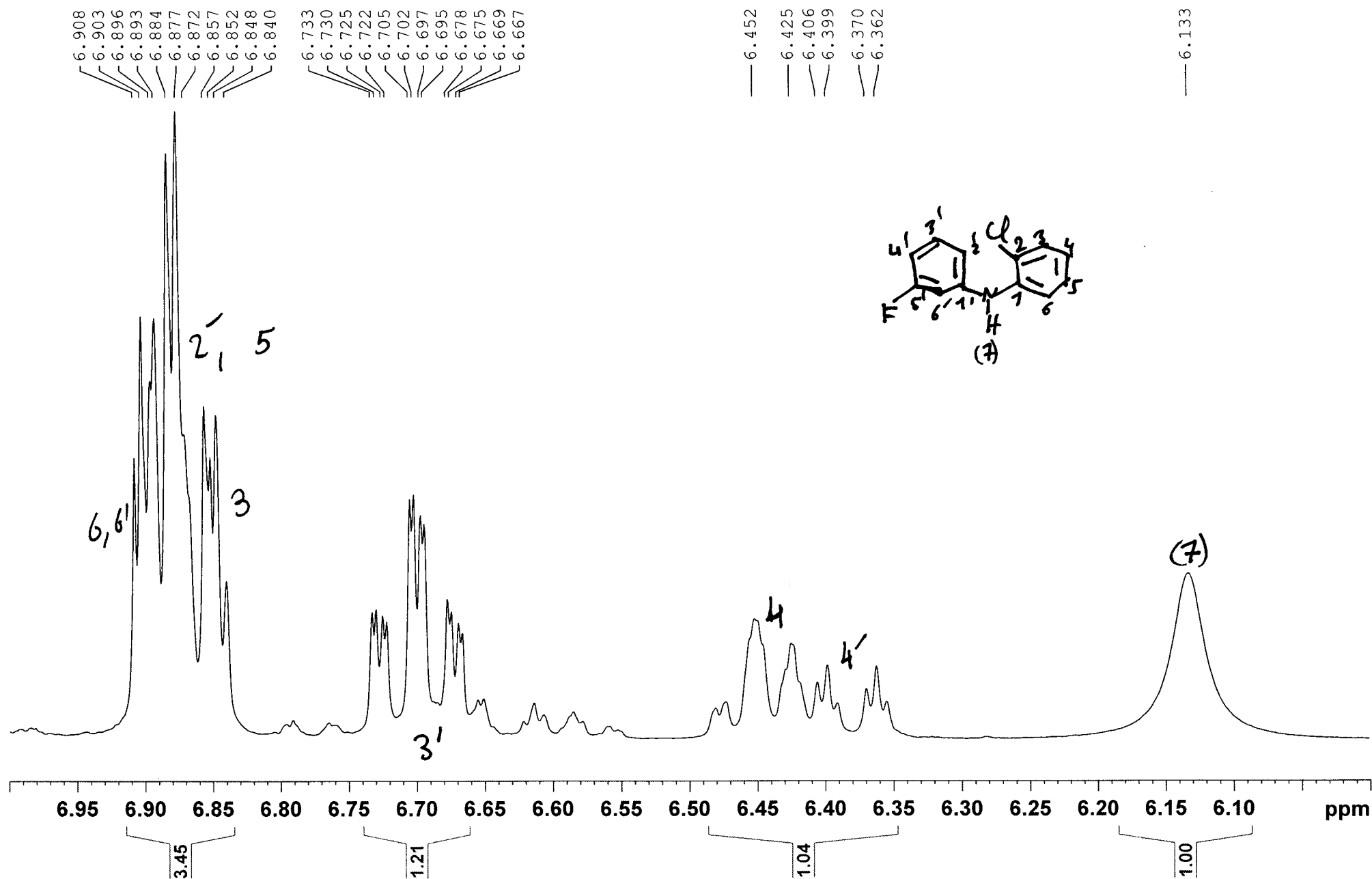
===== CHANNEL f1 =====
NUC1 19F
P1 10.00 usec
PL1 1.00 dB
SFO1 282.3761148 MHz
SI 65536
SF 282.4043550 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC .. 1.00



10mpl_2_99

3c

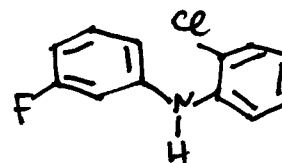
9



10mpl_2_99

— 164.23
— 160.98

142.69
142.55
138.11
129.60
129.49
129.47
128.86
126.68
126.47
121.48
120.49
115.91
113.58
113.54
107.80
107.52
104.91
104.58



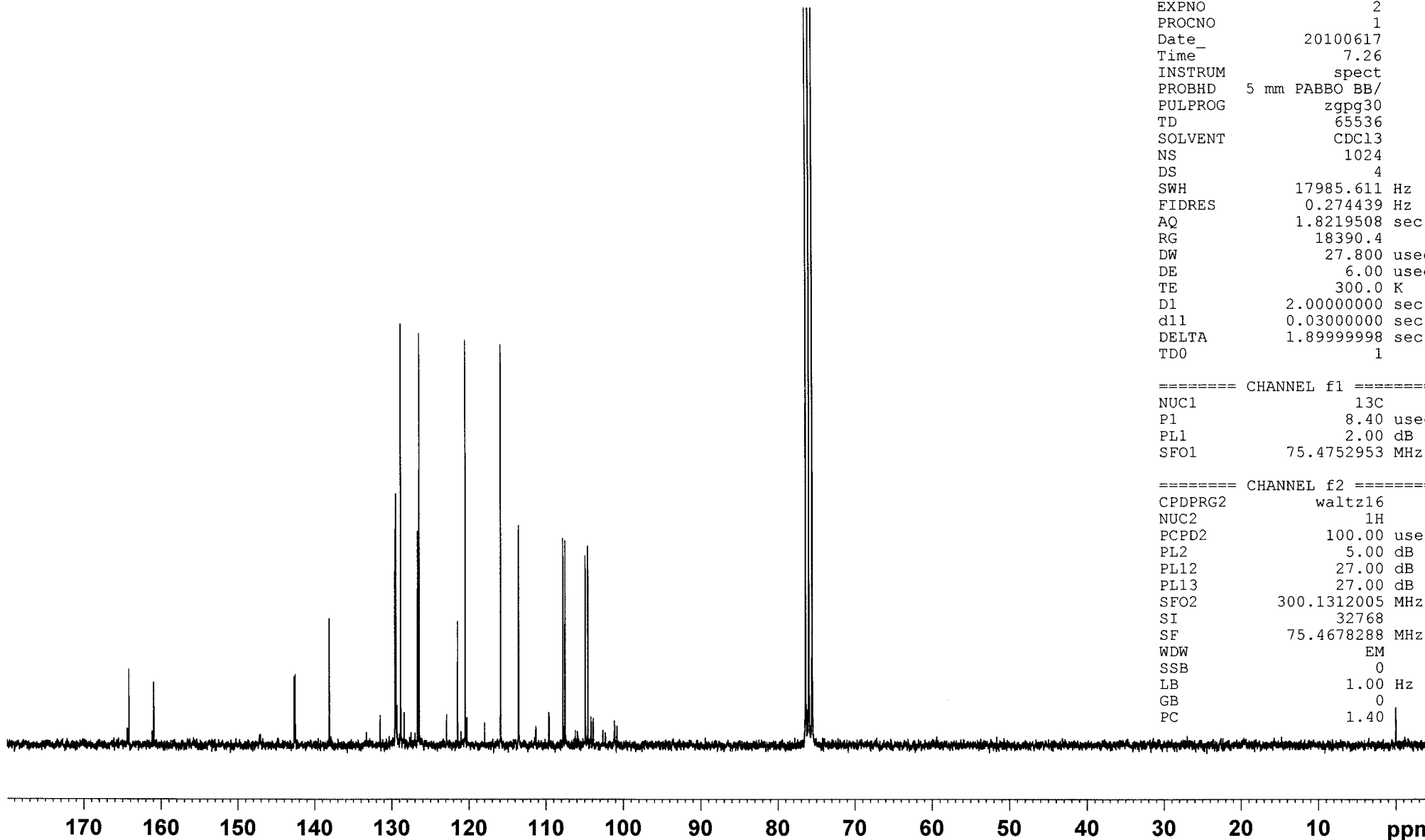
3c

10

NAME 10mpl_2_99
EXPNO 2
PROCNO 1
Date_ 20100617
Time_ 7.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 18390.4
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 5.00 dB
PL12 27.00 dB
PL13 27.00 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4678288 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



10mpl_2_99

3C 11

129.61
129.50
129.48
129.33
128.87
128.41
126.68
126.48

122.91

120.50
120.27

117.99

115.91

113.58
113.55

111.31

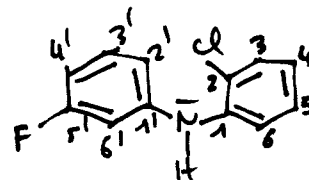
109.60

107.81
107.52

104.91
104.58
104.19
103.90

102.33

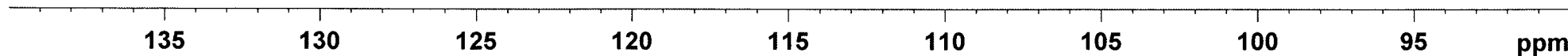
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100.83



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EXPNO 3
PROCNO 1
Date 20100617
Time 7.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001070 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
p2 16.80 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P3 10.00 usec
p4 20.00 usec
PCPD2 100.00 usec
PL2 5.00 dB
PL12 27.00 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4678288 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



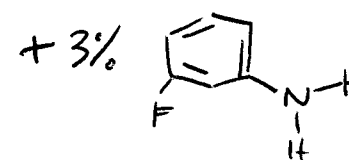
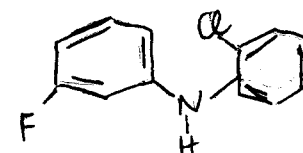
10mpl_2_99

3c

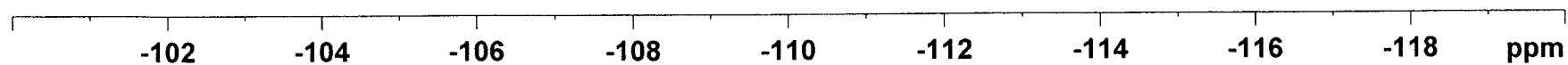
12

-111.81
-111.84
-111.84
-111.86
-111.87
-111.89
-111.90
-113.09
-113.11
-113.12
-113.15
-113.18

NAME 10mpl_2_99
EXPNO 4
PROCNO 1
Date_ 20100617
Time_ 7.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgflqn
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 67567.570 Hz
FIDRES 0.515500 Hz
AQ 0.9699828 sec
RG 362
DW 7.400 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TDO 1



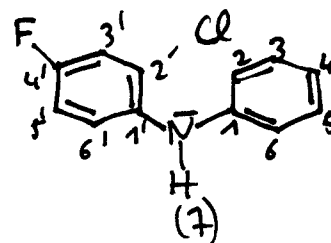
===== CHANNEL f1 =====
NUC1 19F
P1 10.00 usec
PL1 1.00 dB
SFO1 282.3761148 MHz
SI 65536
SF 282.4043550 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3d

13

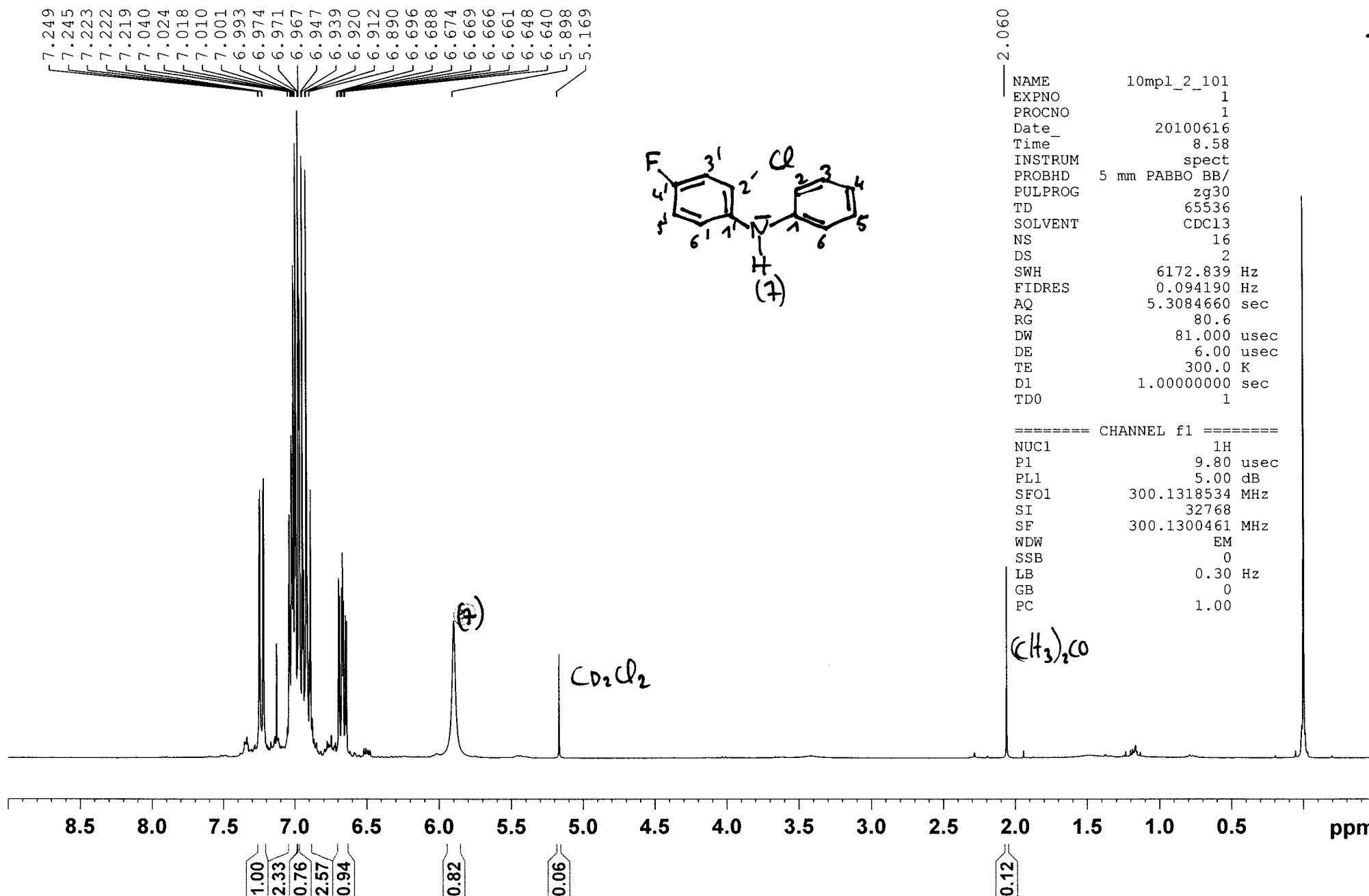
10mpl_2_101



2.060

NAME 10mpl_2_101
EXPNO 1
PROCNO 1
Date_ 20100616
Time 8.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 80.6
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300461 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



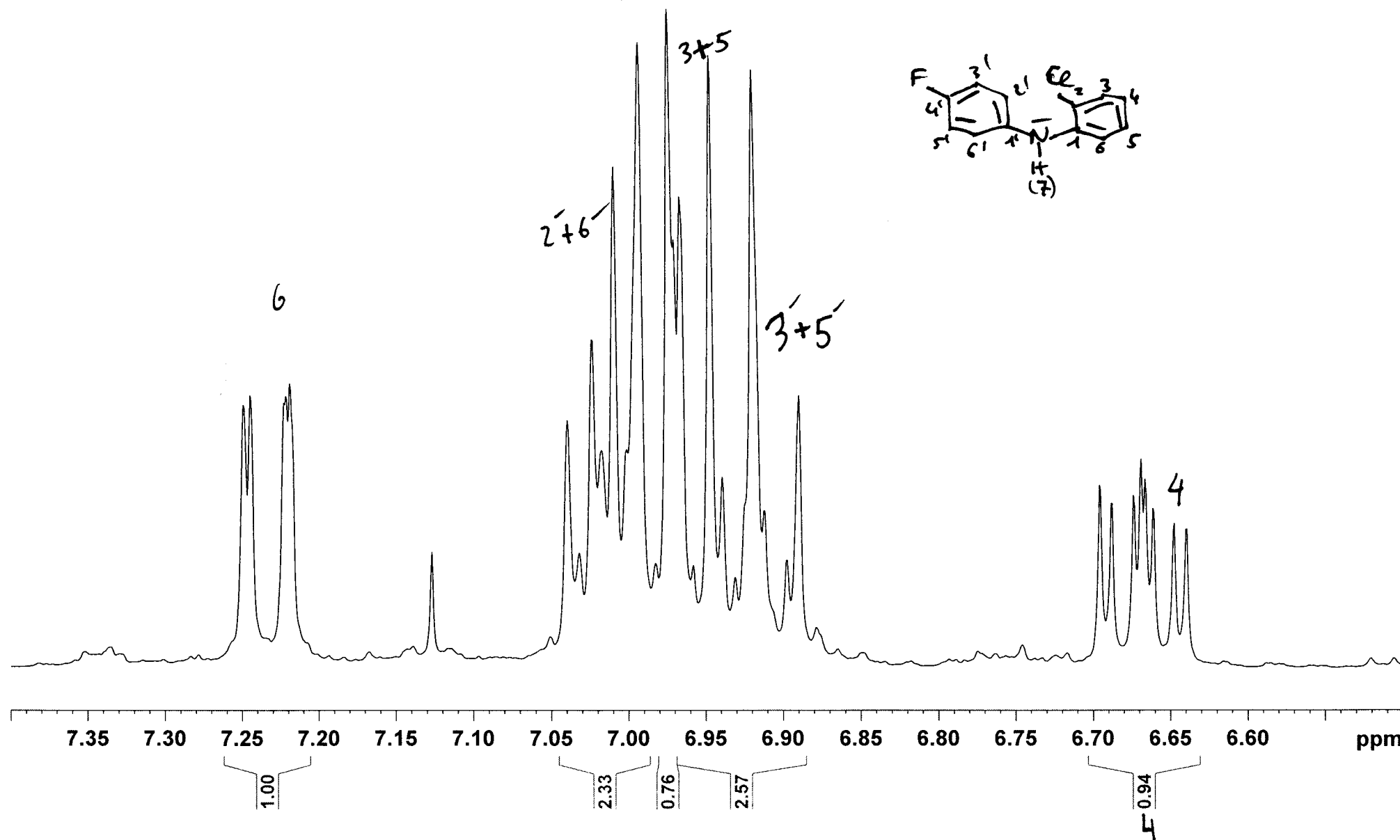
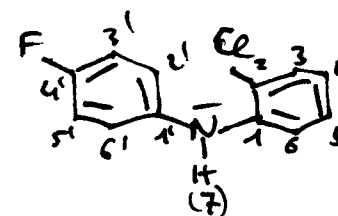
10mpl_2_101

3d 14

7.249
7.245
7.223
7.222
7.219

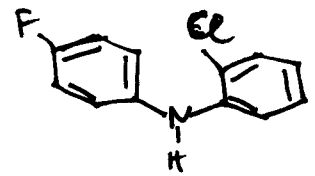
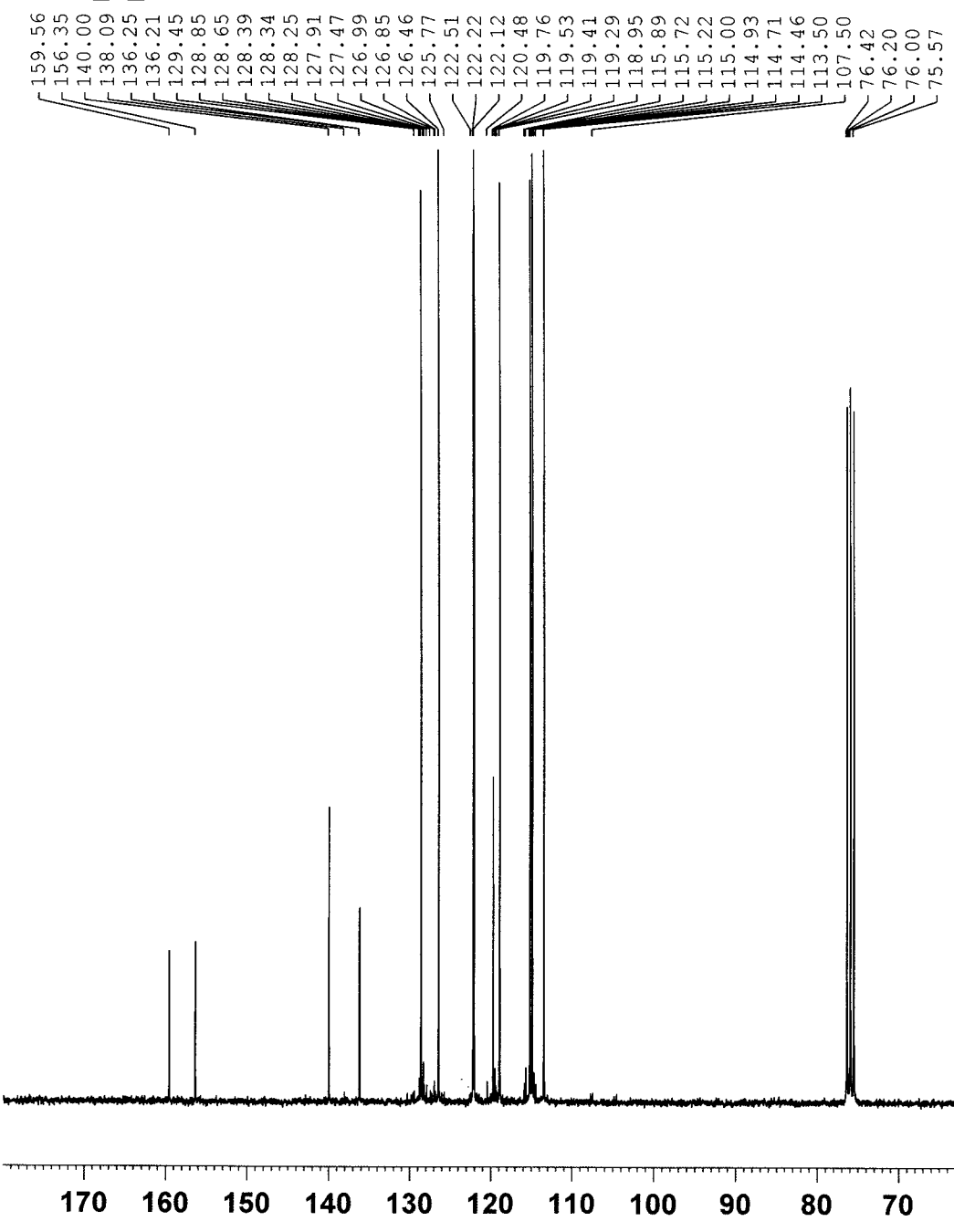
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7.024
7.018
7.010
7.001
6.993
6.974
6.971
6.967
6.947
6.939
6.920
6.912
6.890

6.696
6.688
6.674
6.669
6.666
6.661
6.648
6.640



3d

10mpl_2_101



NAME 10mpl_2_101
EXPNO 2
PROCNO 1
Date_ 20100617
Time 9.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 11585.2
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 5.00 dB
PL12 27.00 dB
PL13 27.00 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4678320 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10mpl_2_101

3d

16

— 159.56

— 156.35

— 140.00

— 138.09

— 136.25

— 136.21

— 129.45

— 128.85

— 128.65

— 128.39

— 128.34

— 128.25

— 127.91

— 127.47

— 126.99

— 126.85

— 126.46

— 125.77

— 122.51

— 122.22

— 122.12

— 120.48

— 119.76

— 119.53

— 119.41

— 119.29

— 118.95

— 115.89

— 115.72

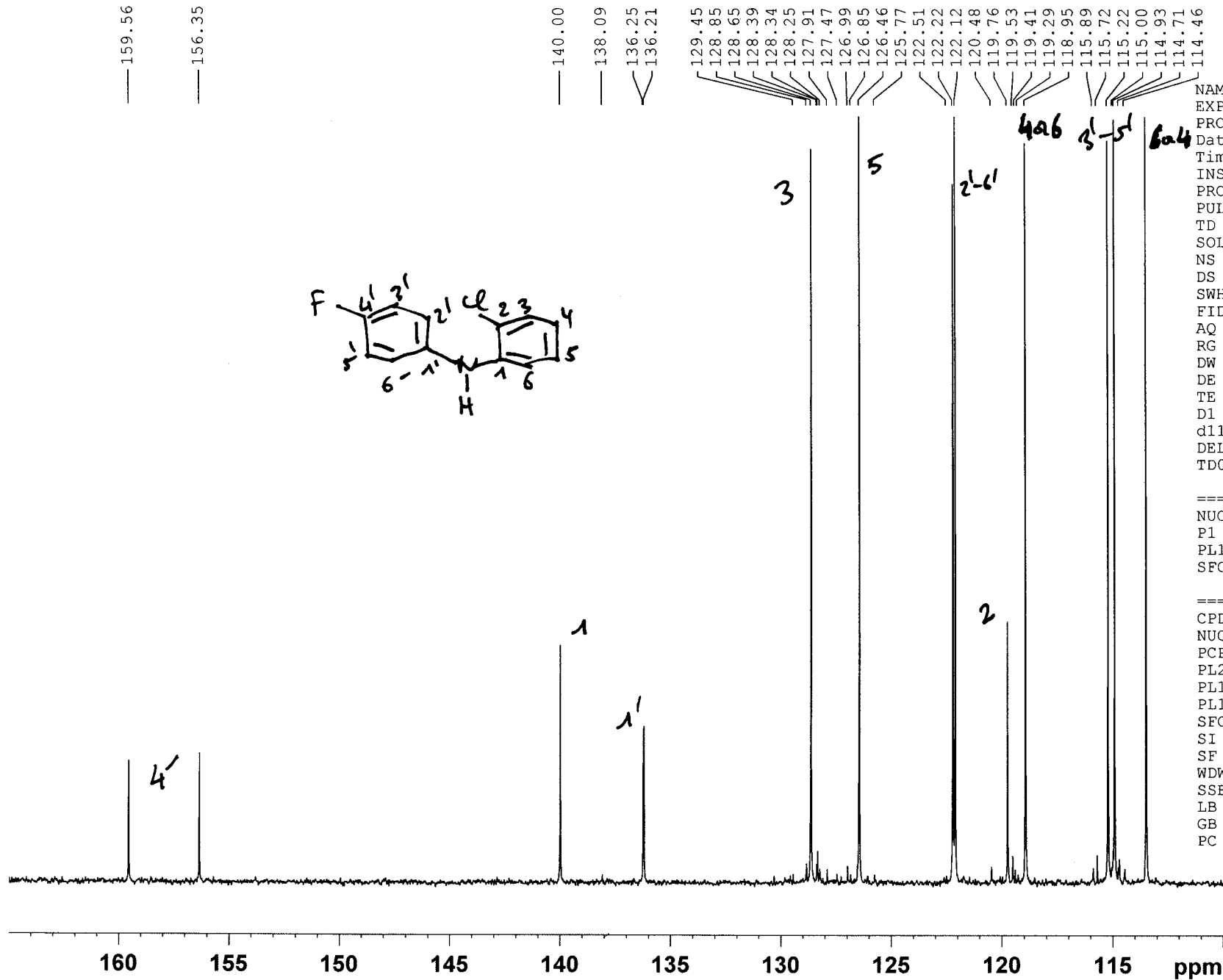
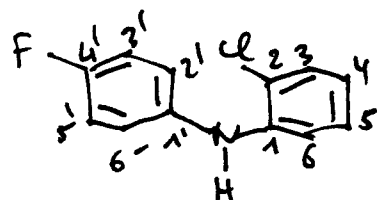
— 115.22

— 115.00

— 114.93

— 114.71

— 114.46



NAME 10mpl_2_101
 EXPNO 2
 PROCNO 1
 Date_ 20100617
 Time 9.02
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 1024
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 11585.2
 DW 27.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.40 usec
 PL1 2.00 dB
 SFO1 75.4752953 MHz

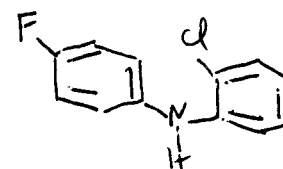
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 5.00 dB
 PL12 27.00 dB
 PL13 27.00 dB
 SFO2 300.1312005 MHz
 SI 32768
 SF 75.4678320 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10mpl_2_101

3d

17

-119.54



NAME 10mpl_2_101
EXPNO 4
PROCNO 1
Date_ 20100617
Time 9.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgflqn
TD 131072
SOLVENT CDC13
NS 16
DS 4
SWH 67567.570 Hz
FIDRES 0.515500 Hz
AQ 0.9699828 sec
RG 362
DW 7.400 usec
DE 10.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 19F
P1 10.00 usec
PL1 1.00 dB
SFO1 282.3761148 MHz
SI 65536
SF 282.4043550 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

-105

-110

-115

-120

-125

-130

-135

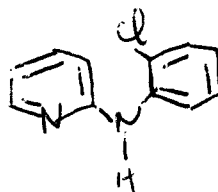
ppm

10mpl_2_40 F1

5

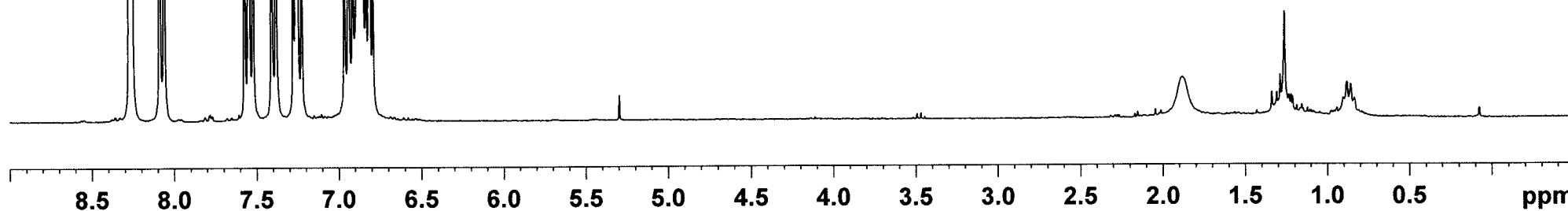
18

8.061
8.056
7.573
7.566
7.547
7.544
7.521
7.515
7.408
7.403
7.382
7.277
7.272
7.260
7.252
7.249
7.225
7.220
6.966
6.961
6.940
6.936
6.914
6.910
6.880
6.852
6.833
6.816
6.809
6.792



NAME 10mpl_2_40 F1
EXPNO 1
PROCNO 1
Date_ 20100430
Time_ 15.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 406.4
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

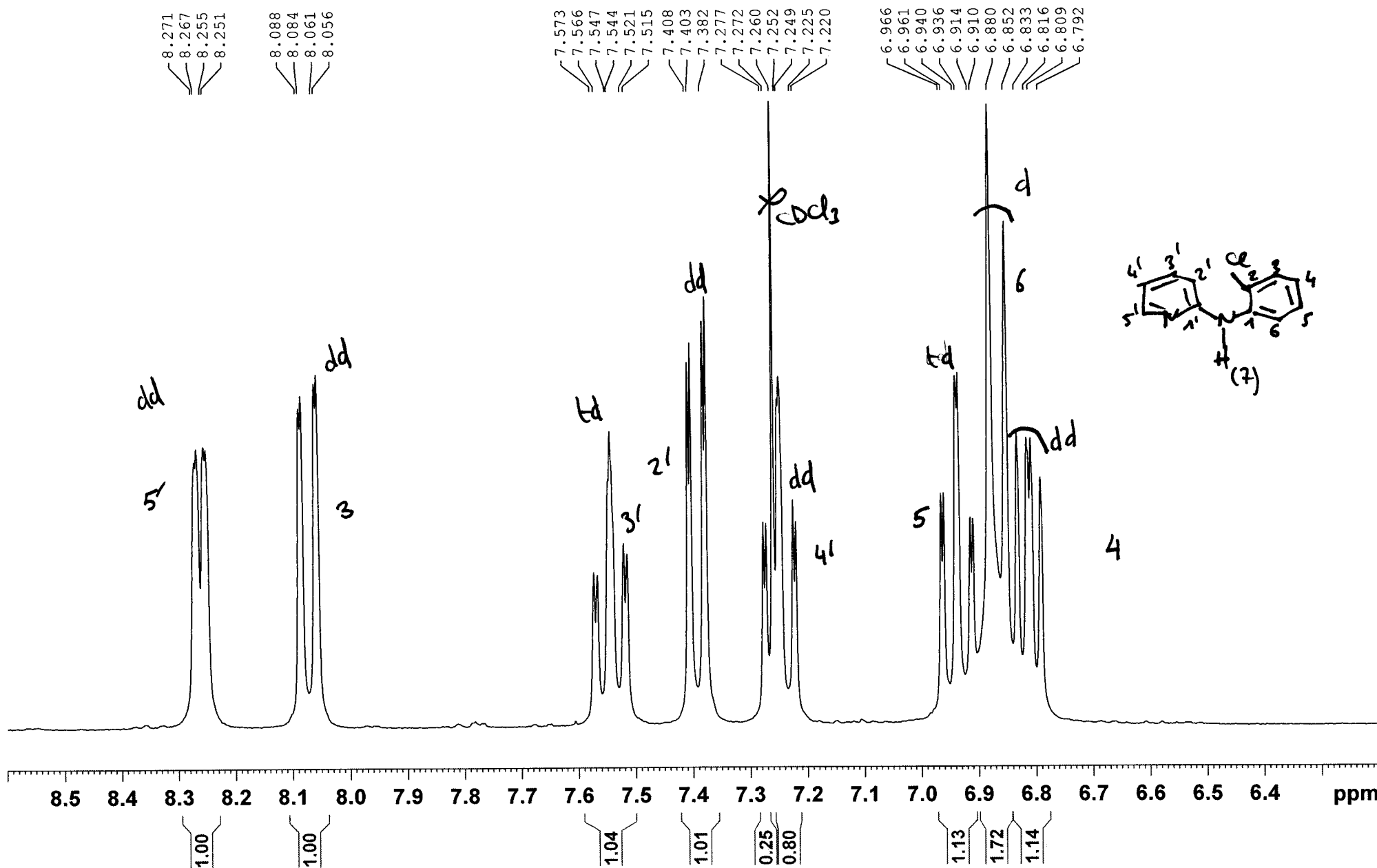
===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



1.00
1.00
1.04
1.01
0.25
0.80
1.13
1.72
1.14

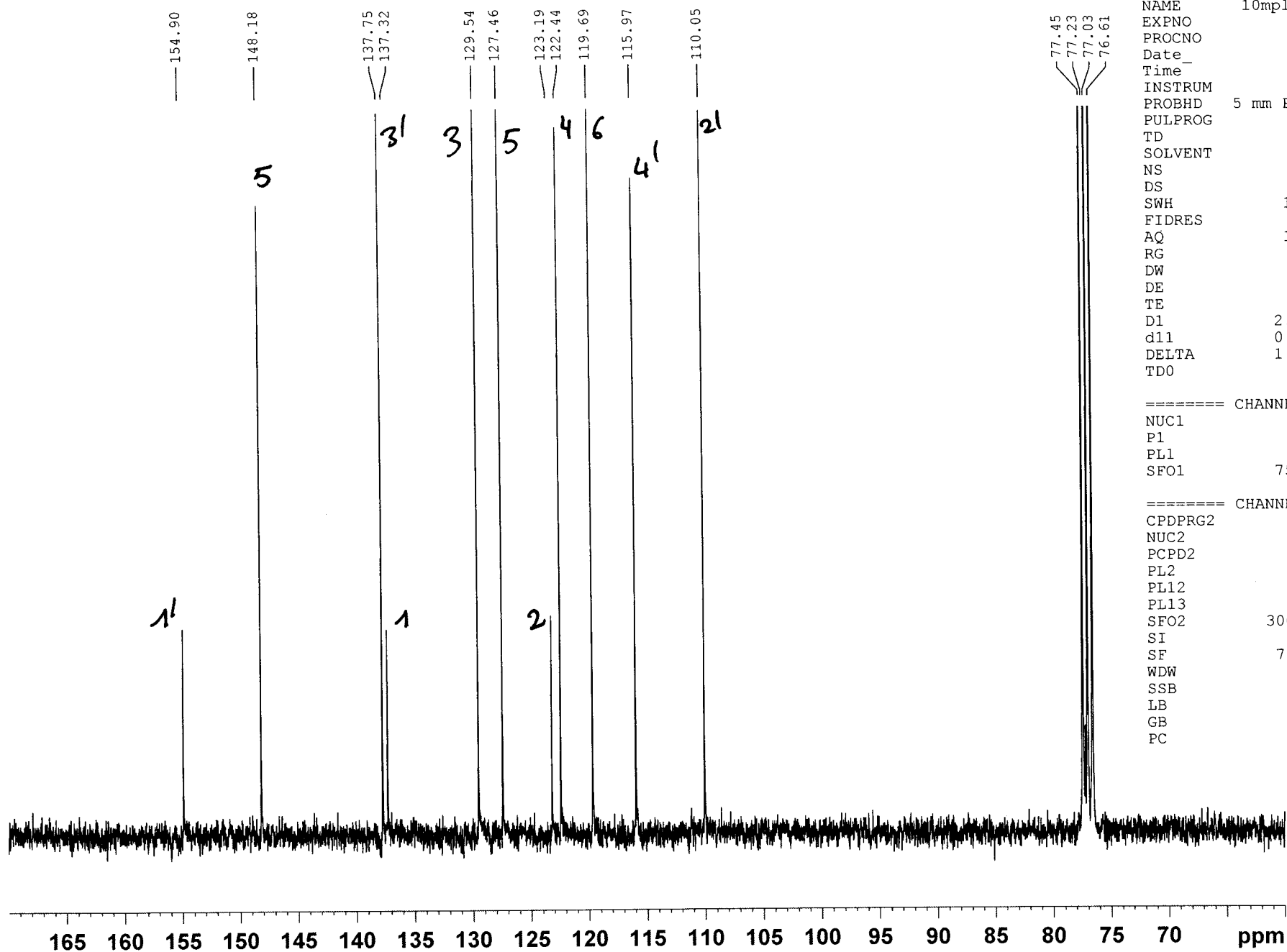
10mp1_2_40 F1

5
19



10mpl_2_40 F1

⑤ 20



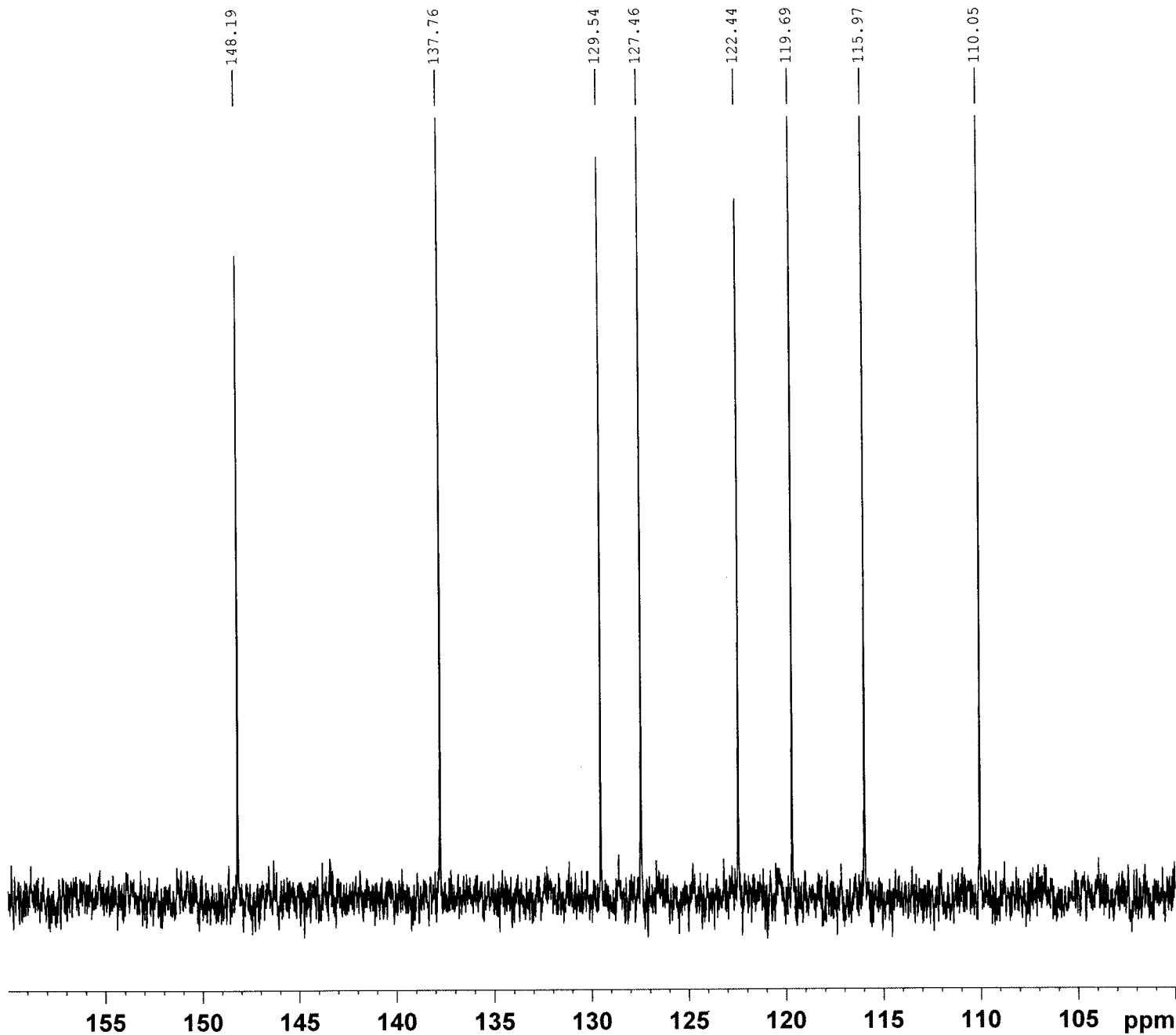
NAME 10mpl_2_40 F1
 EXPNO 2
 PROCNO 1
 Date_ 20100501
 Time 17.50
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 1024
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 20642.5
 DW 27.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.40 usec
 PL1 2.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 5.00 dB
 PL12 27.00 dB
 PL13 27.00 dB
 SFO2 300.1312005 MHz
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10mpl_2_40 F1

5
21



```

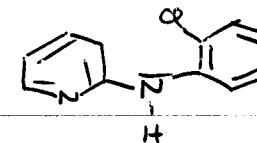
NAME      10mpl_2_40 F1
EXPNO      3
PROCNO      1
Date_      20100501
Time_      18.09
INSTRUM      spect
PROBHD      5 mm PABBO BB/
PULPROG      dept135
TD      65536
SOLVENT      CDC13
NS      256
DS      4
SWH      17985.611 Hz
FIDRES      0.274439 Hz
AQ      1.8219508 sec
RG      16384
DW      27.800 usec
DE      6.00 usec
TE      300.0 K
CNST2      145.0000000
D1      2.00000000 sec
d2      0.00344828 sec
d12      0.00002000 sec
DELTA      0.00001070 sec
TD0      1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1      8.40 usec
p2      16.80 usec
PL1      2.00 dB
SFO1      75.4752953 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2      1H
P3      10.00 usec
p4      20.00 usec
PCPD2      100.00 usec
PL2      5.00 dB
PL12      27.00 dB
SFO2      300.1312005 MHz
SI      32768
SF      75.4677490 MHz
WDW      EM
SSB      0
LB      1.00 Hz
GB      0
PC      1.40
  
```



22

Display Report

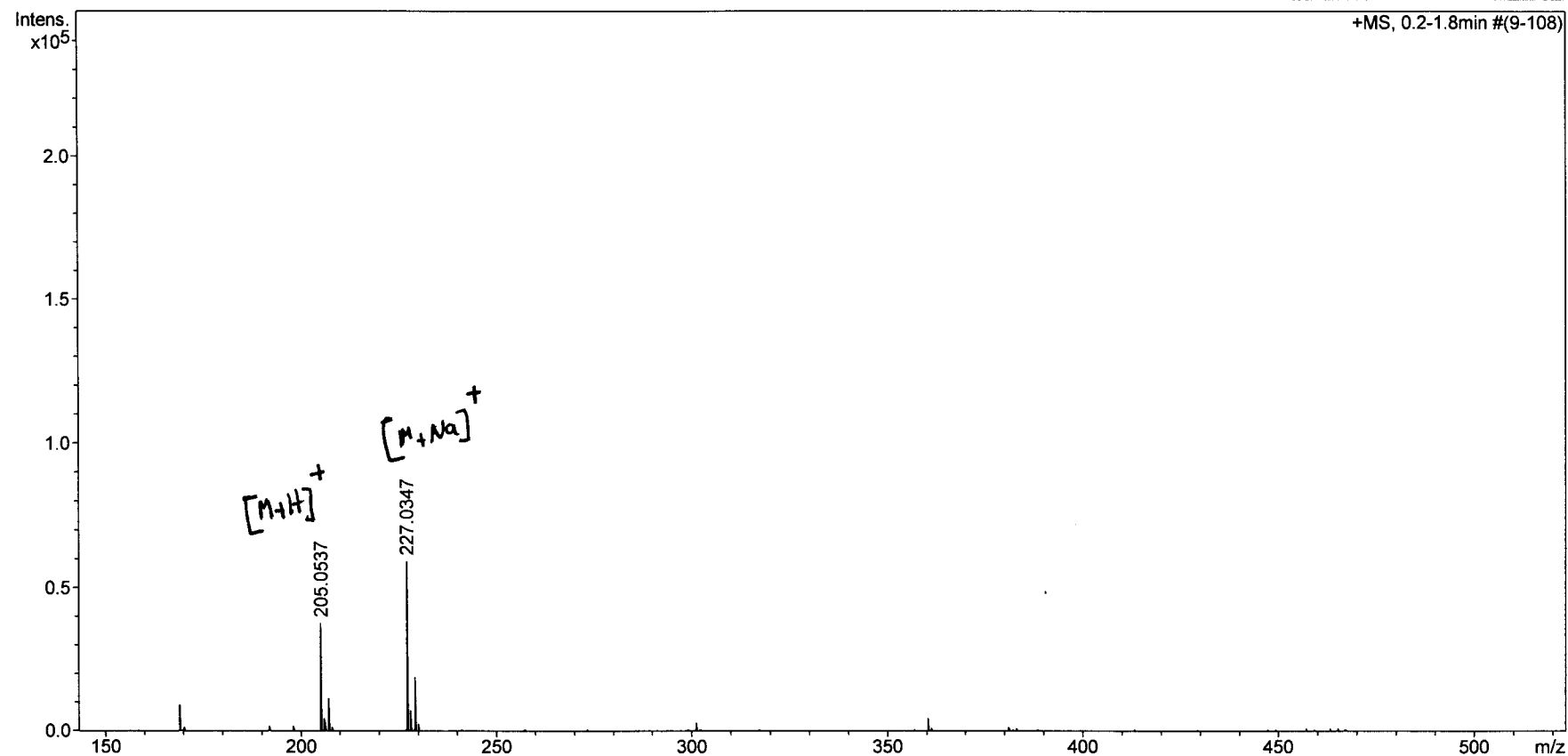
Analysis Info

Analysis Name 11mpl_2_40_b_me.d
Method tune_low.m
Sample Name
Comment DCM/MeOH

Acquisition Date 3/29/2011 2:25:02 PM
Operator Fanny Chaux
Instrument micrOTOF-Q 56

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	5.8 psi
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	190 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	160.0 Vpp	Set Divert Valve	Source



Mass Spectrum Report

Analysis Info

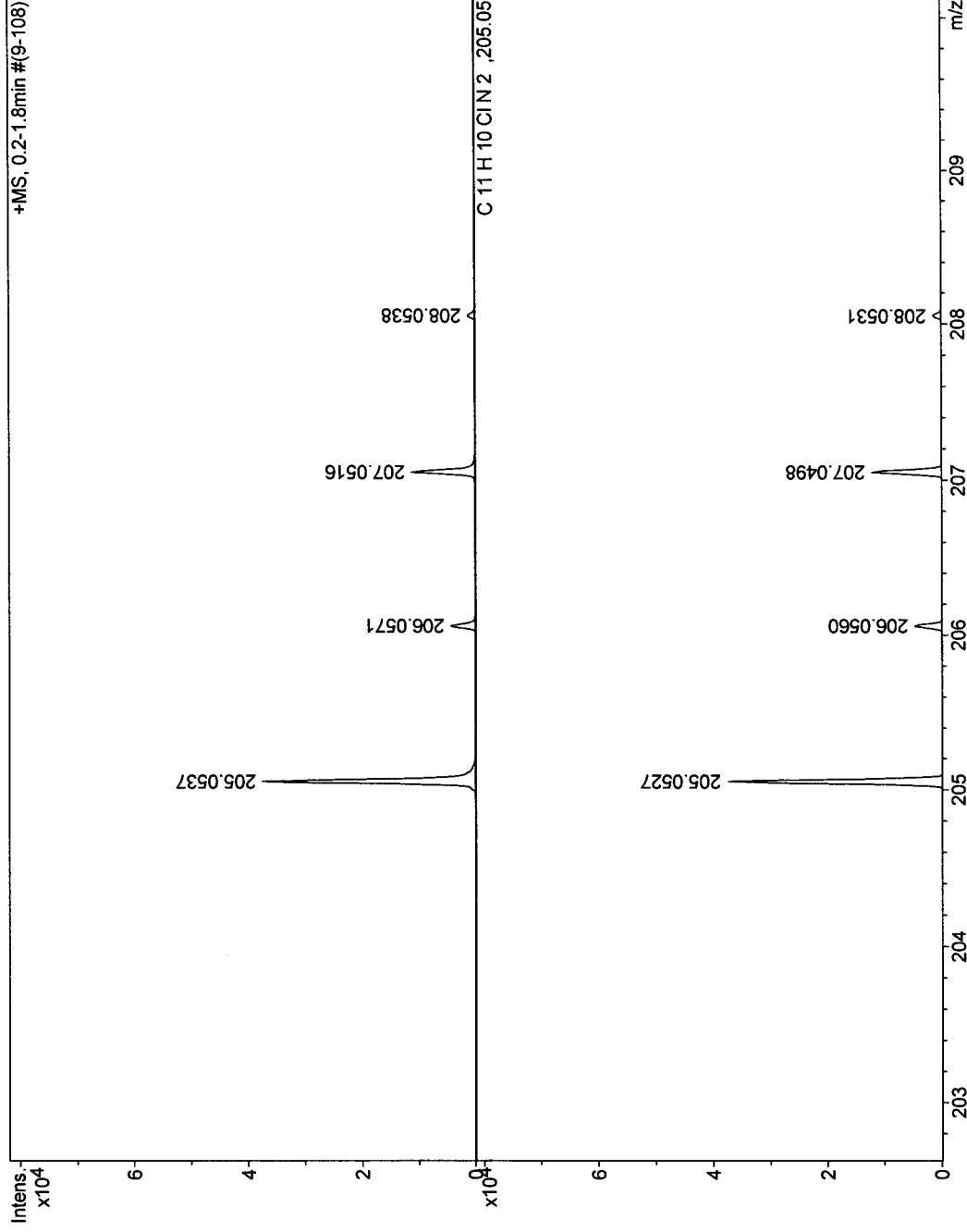
Analysis Name 11mpl_2_40_b_me.d Acquisition Date 3/29/2011 2:25:02 PM
Method tune_low.m Operator Fanny Chaux
Sample Name Instrument microTOF-Q 56
Comment DCM/MeOH

Acquisition Parameter

Source Type ESI
Focus Not active
Scan Begin 50 m/z
Scan End 3000 m/z

Ion Polarity Positive
Set Capillary 4500 V
Set End Plate Offset -500 V
Set Collision Cell RF 160.0 Vpp

Set Nebulizer 5.8 psi
Set Dry Heater 190 °C
Set Dry Gas 4.0 l/min
Set Divert Valve Source



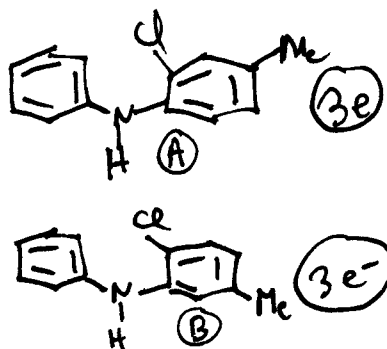
Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdB	e ⁻ Conf	N-Rule
205.0537	1	C ₁₁ H ₁₀ ClN ₂	100.00	205.0527	-1.0	-4.8	13.8	7.5	even	ok

10mpl_1_159 F1

$3e + 3e^-$

24

7.316
7.288
7.263
7.259
7.231
7.209
7.201
7.174
7.152
7.133
7.130
7.105
7.102
7.079
7.075
7.055
7.023
6.999
6.974
6.948
6.923
6.897
6.893
6.596
6.573
6.569



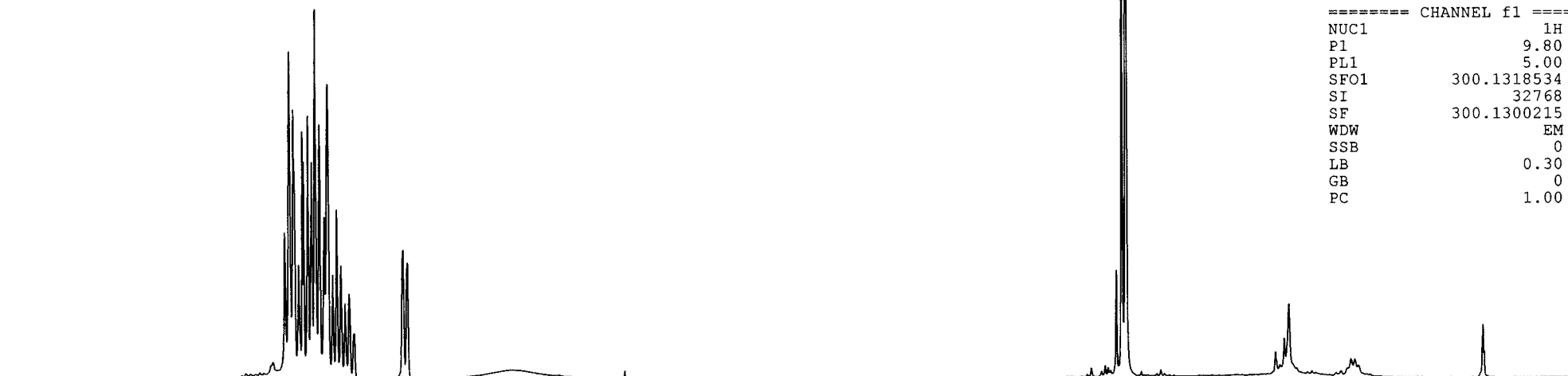
2.270
2.237
2.213

(A)
Me

(B)
Me

NAME 10mpl_1_159
EXPNO 1
PROCNO 1
Date_ 20100423
Time 14.05
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 228.1
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300215 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

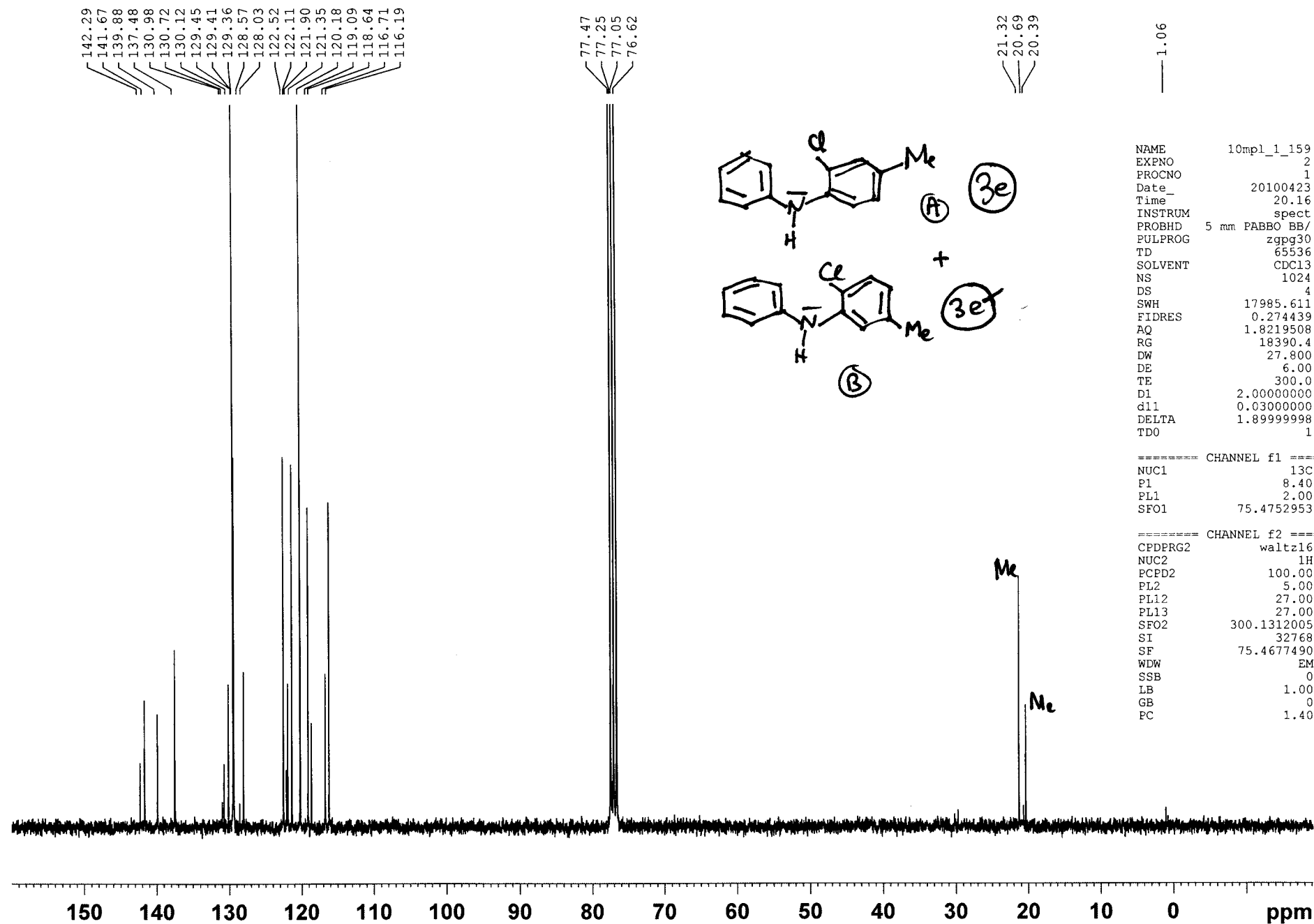
2.24
0.45
0.89
0.74
2.84
1.93
1.25
0.75
1.00

0.23
1.31
3.08

10mpl_1_159 F1

 $3e + 3e^-$

25

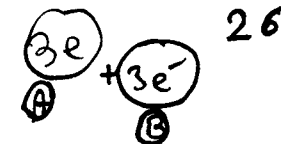


NAME 10mpl_1_159
 EXPNO 2
 PROCNO 1
 Date_ 20100423
 Time 20.16
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 18390.4
 DW 27.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.40 usec
 PL1 2.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 5.00 dB
 PL12 27.00 dB
 PL13 27.00 dB
 SFO2 300.1312005 MHz
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10mpl_1_159 F1



26

142.29
141.67

139.88

137.48

130.98
130.72
130.12
129.45
129.41
129.36
128.57
128.03

122.52
122.11
121.90
121.35

120.18

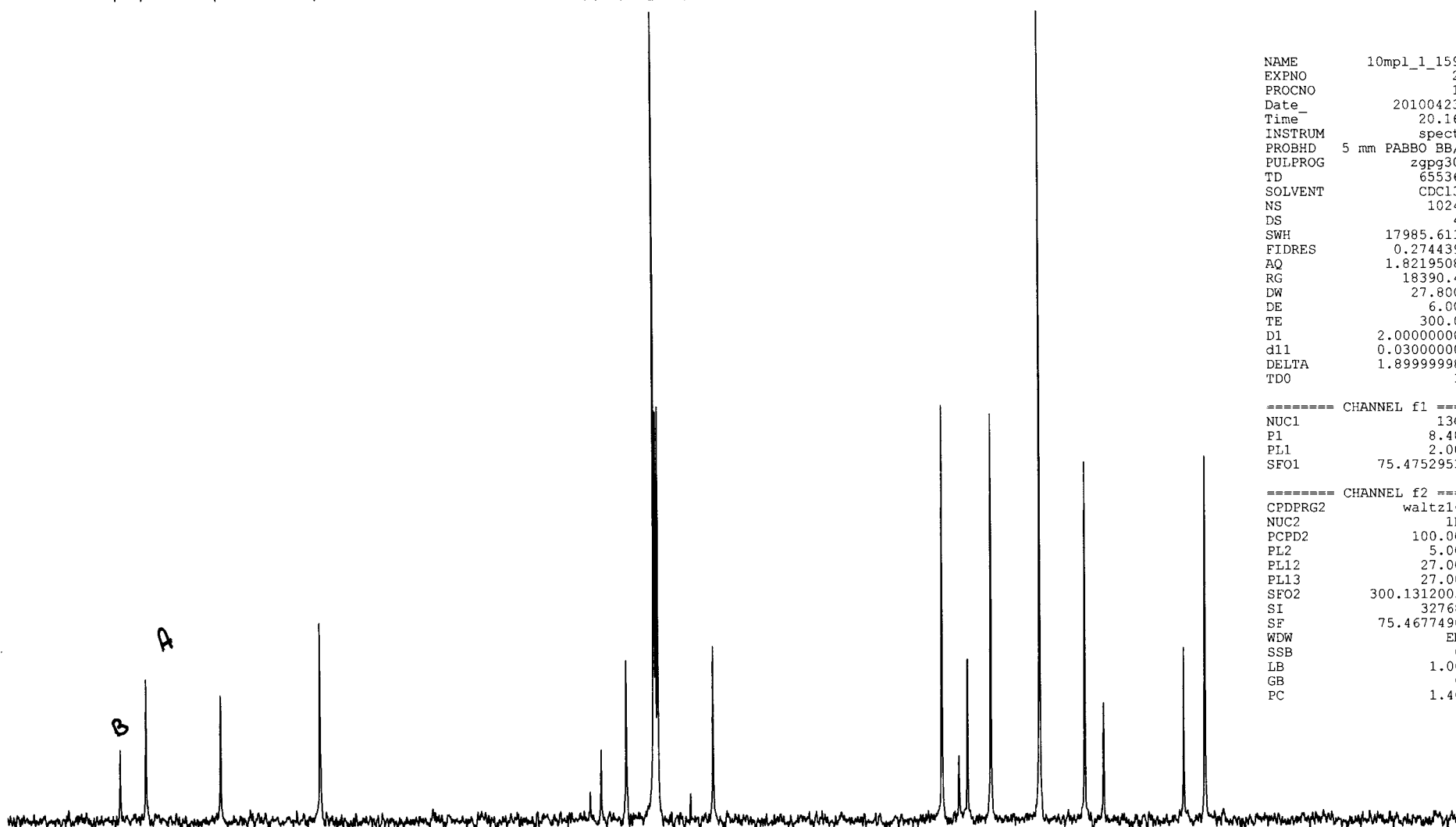
119.09
118.64

116.71
116.19

NAME 10mpl_1_159
EXPNO 2
PROCNO 1
Date_ 20100423
Time 20.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 18390.4
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 5.00 dB
PL12 27.00 dB
PL13 27.00 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



140

135

130

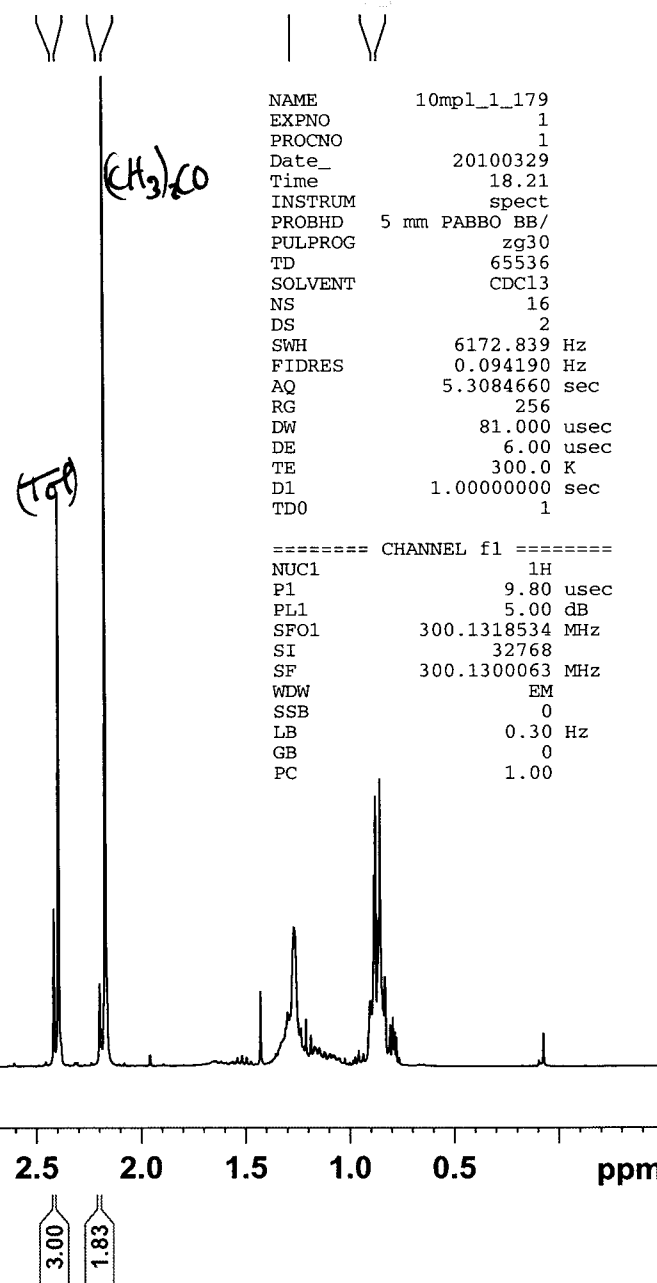
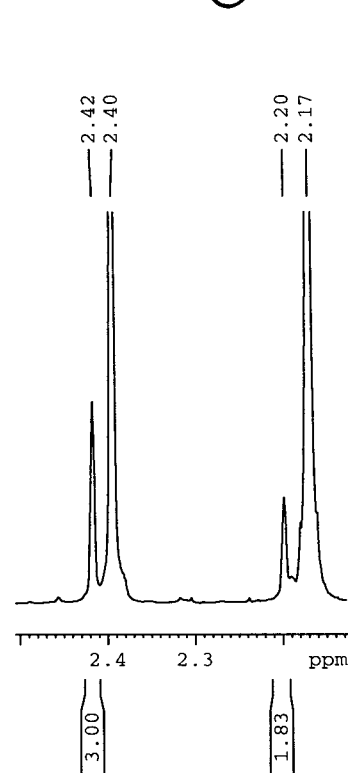
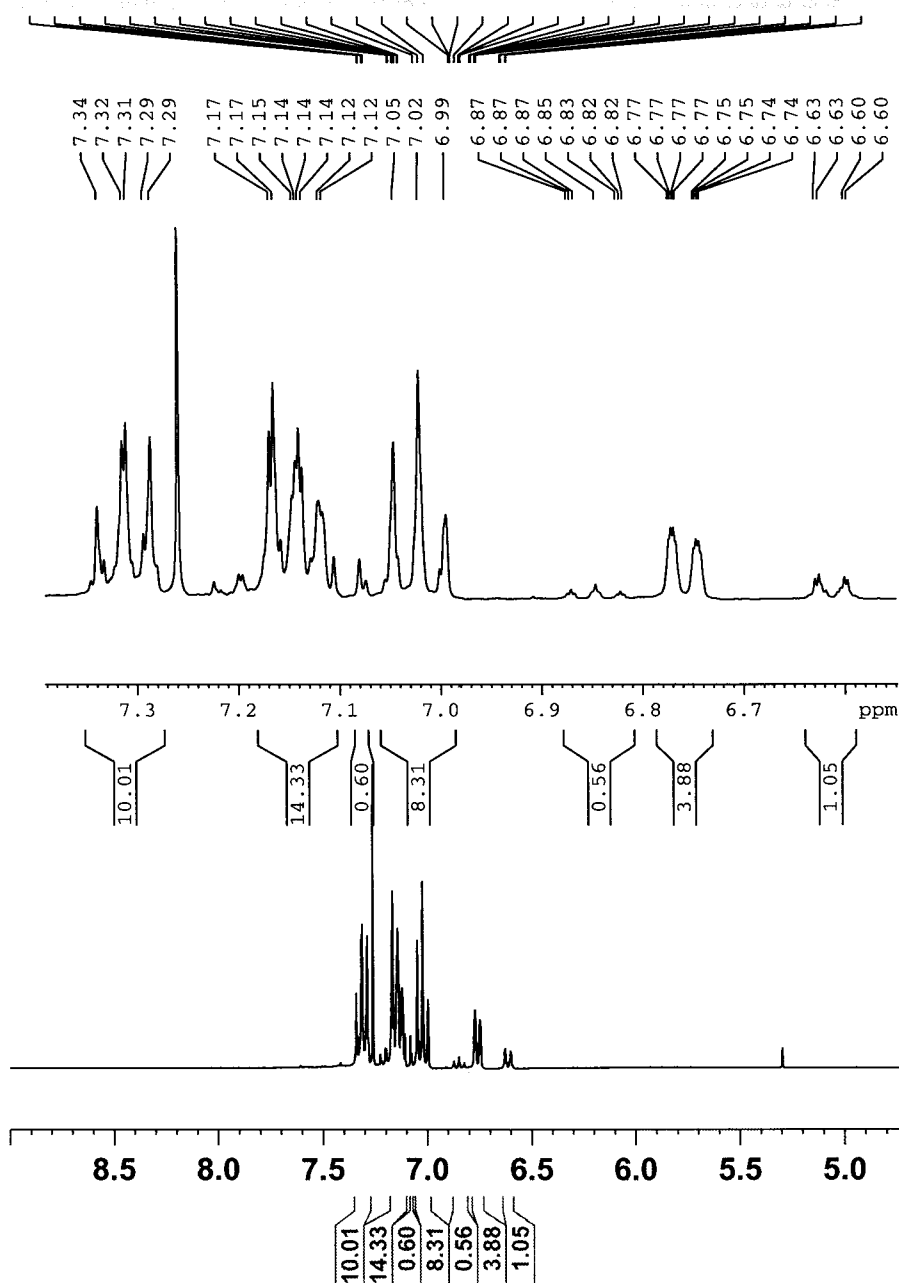
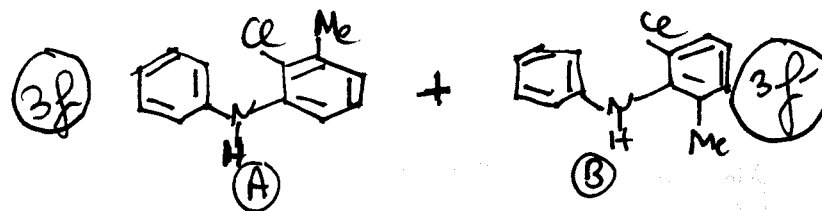
125

120

115

ppm

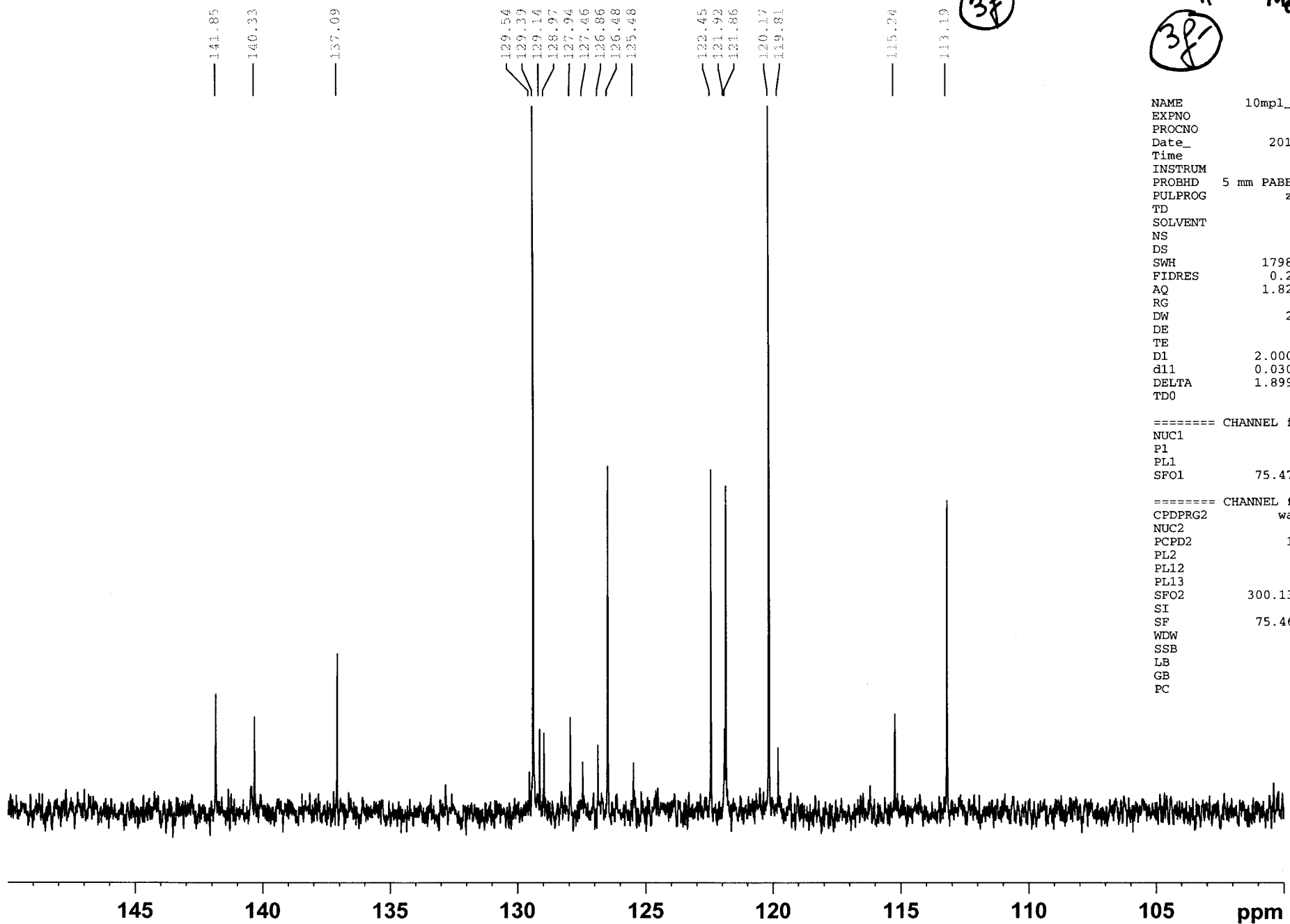
10mpl_1_179



NAME 10mpl_1_179
 EXPNO 1
 PROCNO 1
 Date_ 20100329
 Time 18.21
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.094190 Hz
 AQ 5.3084660 sec
 RG 256
 DW 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.80 usec
 PL1 5.00 dB
 SFO1 300.1318534 MHz
 SI 32768
 SF 300.1300063 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

10mpl_1_179

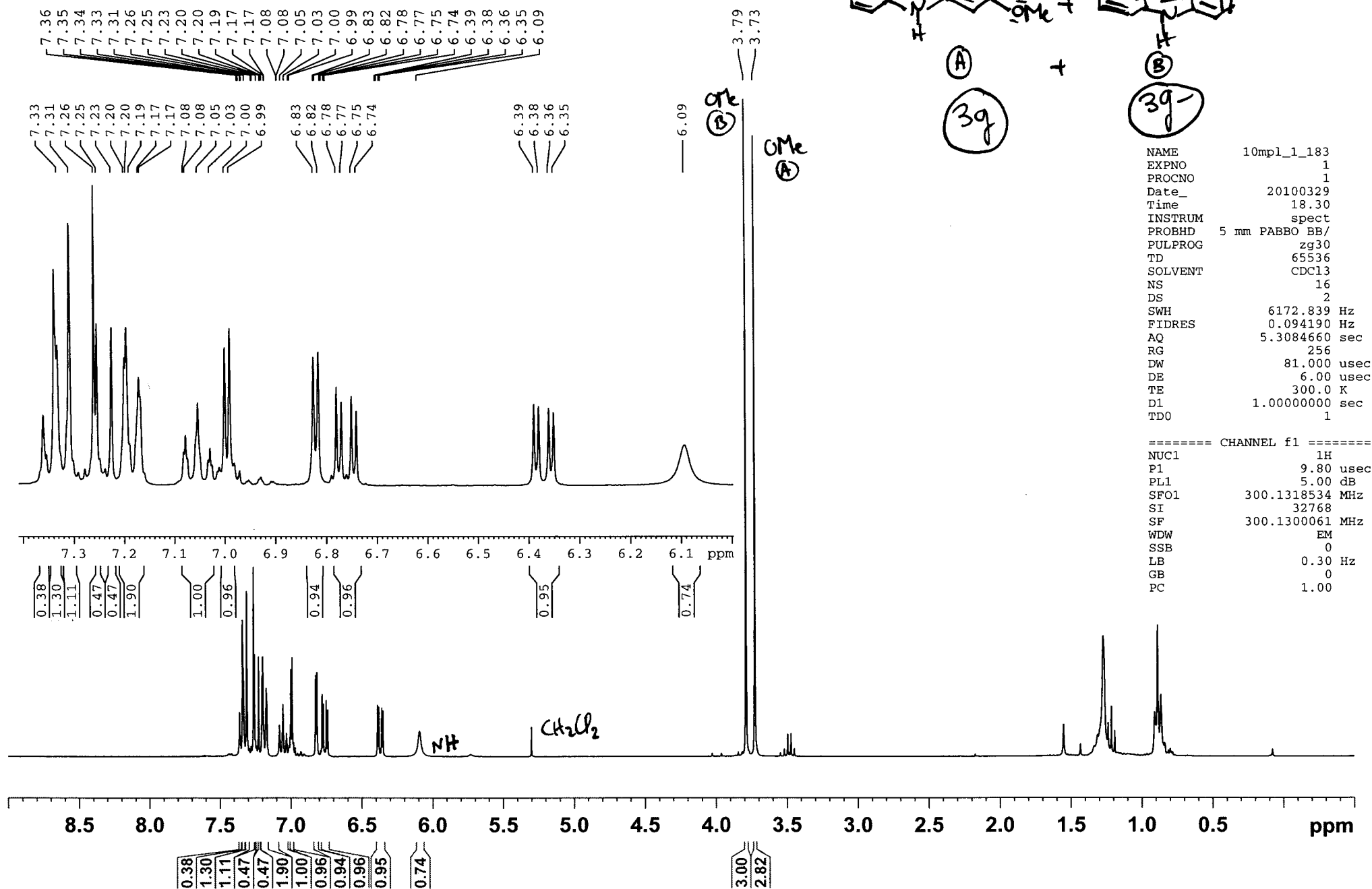


NAME 10mpl_1_179
 EXPNO 2
 PROCNO 1
 Date_ 20100330
 Time 20.22
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 1024
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 16384
 DW 27.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

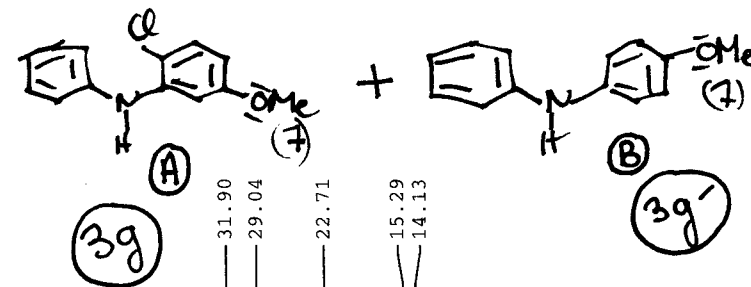
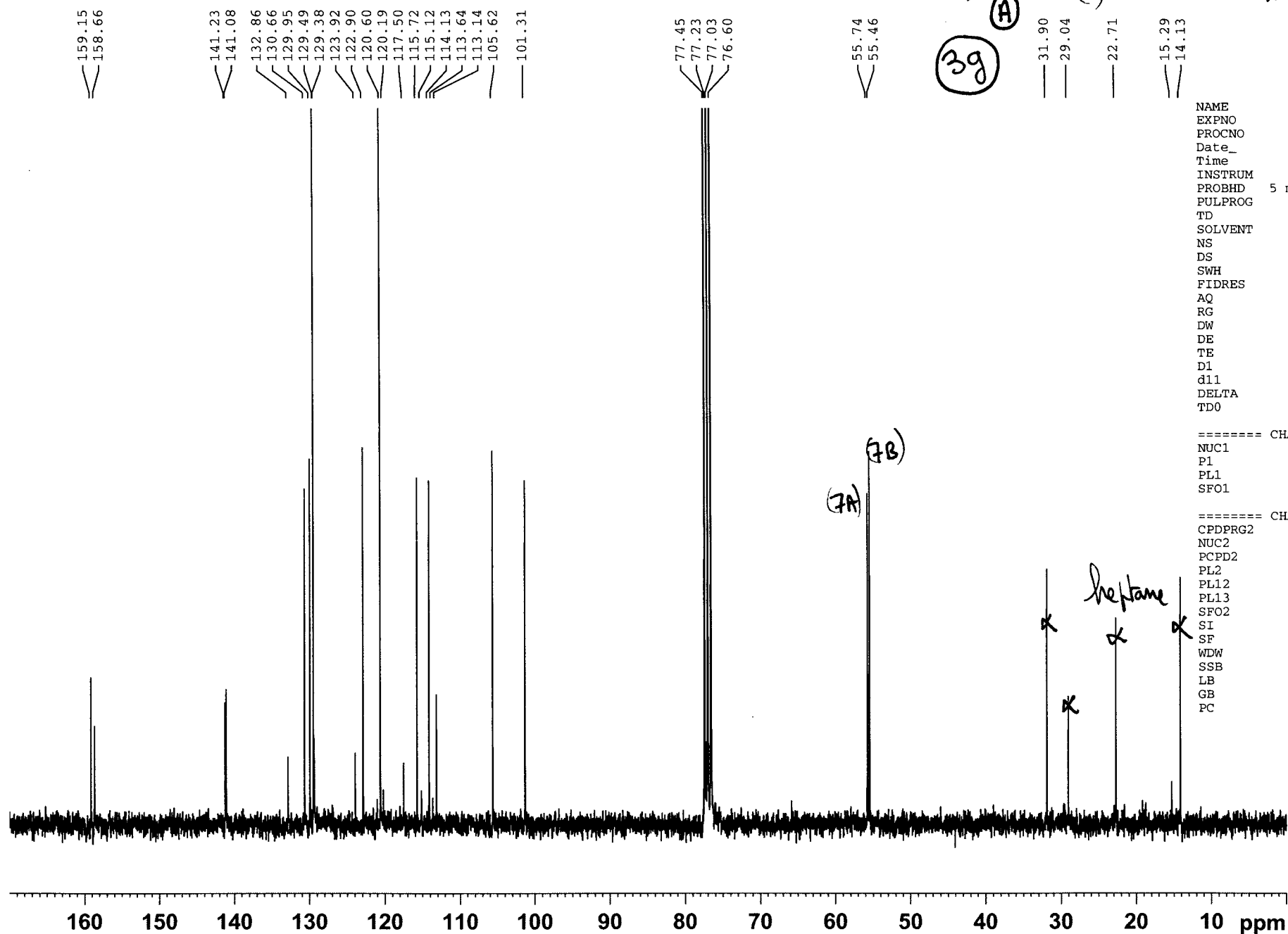
===== CHANNEL f1 =====
 NUC1 13C
 P1 8.40 usec
 PL1 2.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 5.00 dB
 PL12 27.00 dB
 PL13 27.00 dB
 SFO2 300.1312005 MHz
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10mpl_1_183



10mpl_1_183



NAME 10mpl_1_183
 EXPNO 2
 PROCNO 1
 Date_ 20100330
 Time 21.54
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 1024
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 20642.5
 DW 27.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.40 usec
 PL1 2.00 dB
 SFO1 75.4752953 MHz

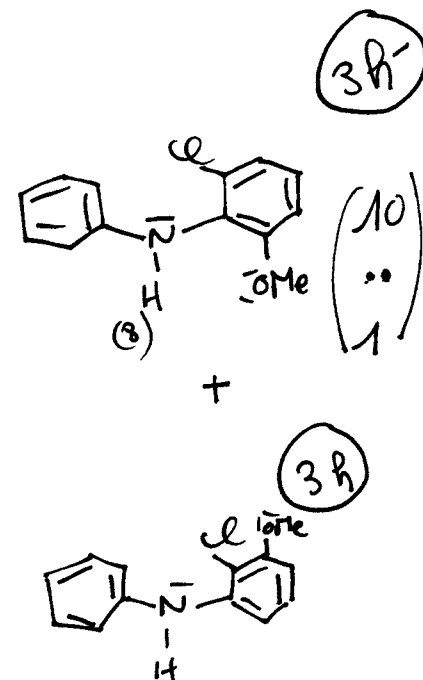
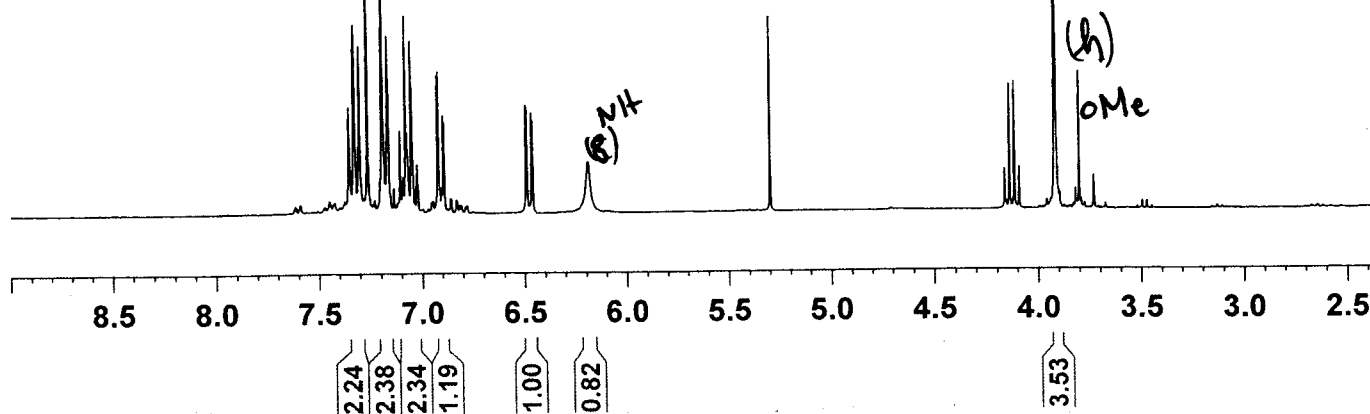
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 5.00 dB
 PL12 27.00 dB
 PL13 27.00 dB
 SFO2 300.1312005 MHz
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10mpl_2_26 F1

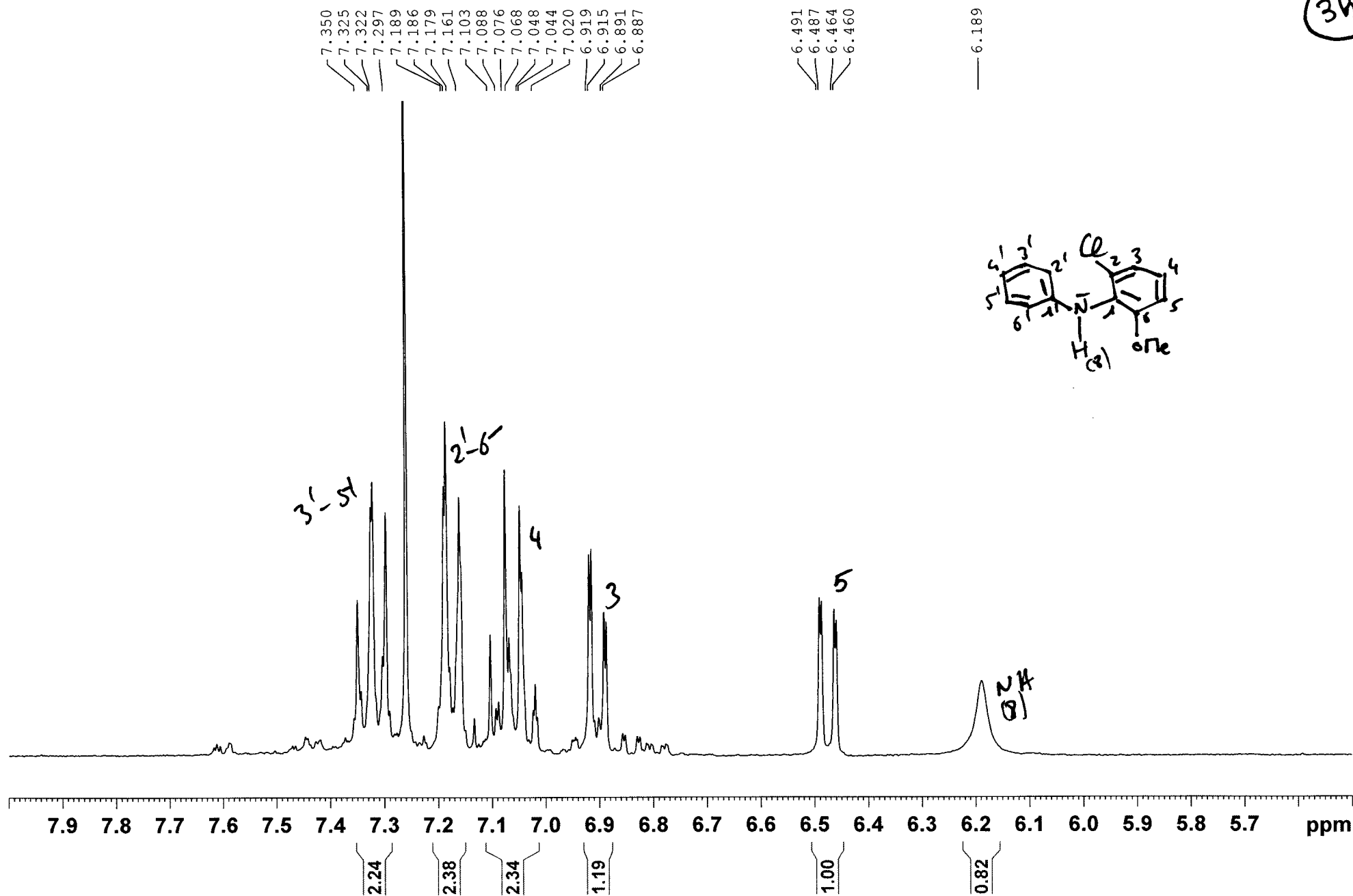
7.350
7.325
7.322
7.297
7.189
7.186
7.179
7.161
7.103
7.088
7.076
7.068
7.048
7.044
7.020
6.919
6.915
6.891
6.887
6.491
6.487
6.464
6.460
6.189

NAME 10mpl_2_26 F1
EXPNO 1
PROCNO 1
Date_ 20100423
Time 23.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1290608 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



(3H)



10mpl_2_26 F1

38

— 154.88

140.63
140.49

— 128.36

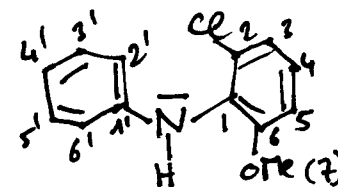
— 126.05

— 121.74

— 119.64

— 108.38
— 106.91

— 101.88

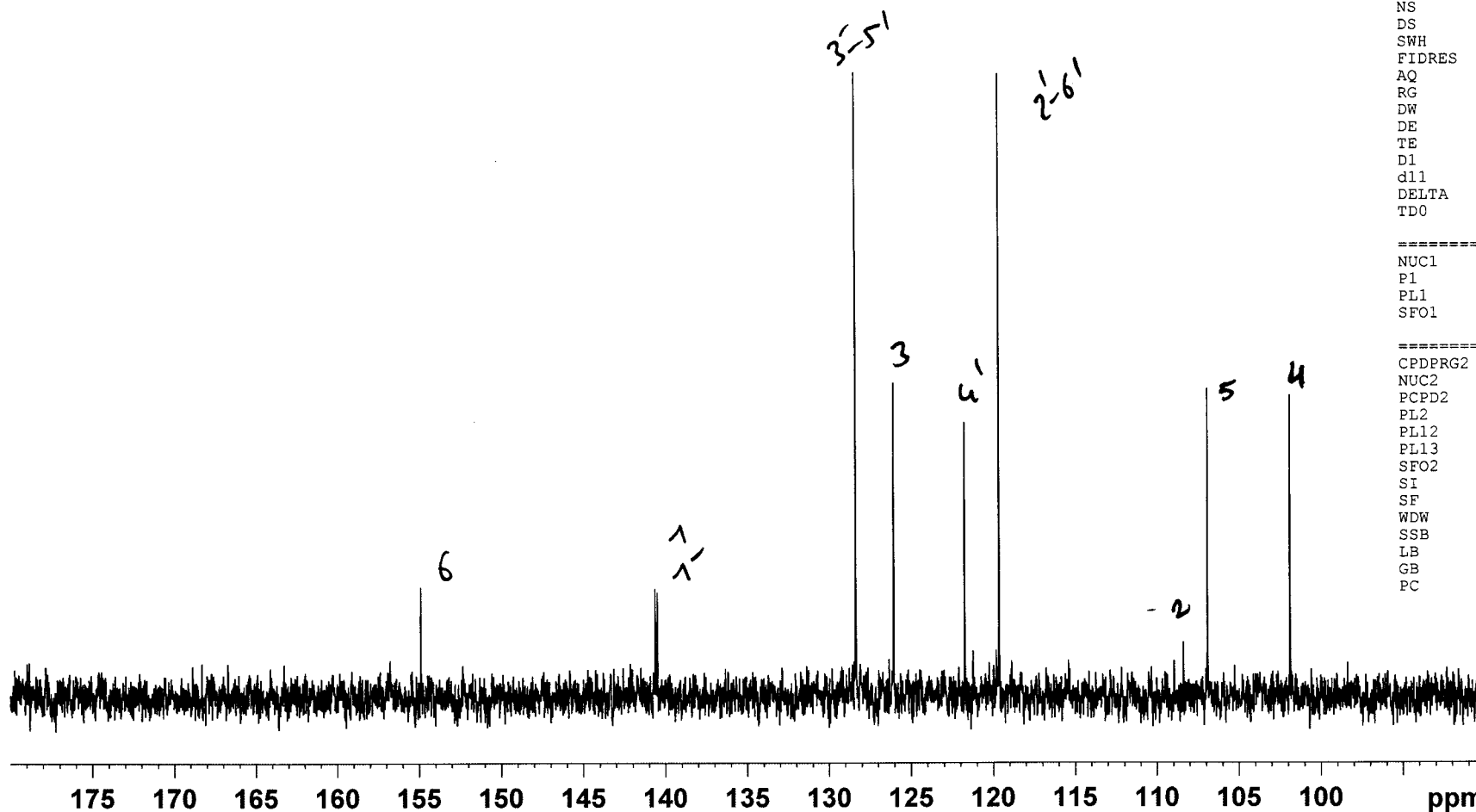


38

NAME 10mpl_2_26 F1
EXPNO 2
PROCNO 1
Date_ 20100424
Time_ 0.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 14596.5
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 5.00 dB
PL12 27.00 dB
PL13 27.00 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4675886 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Mass Spectrum Report

Analysis Info

Analysis Name 11mpl_2_26_b_me.d
Method tune_low.m
Sample Name
Comment DCM/MeOH

Acquisition Date 3/29/2011 2:28:34 PM

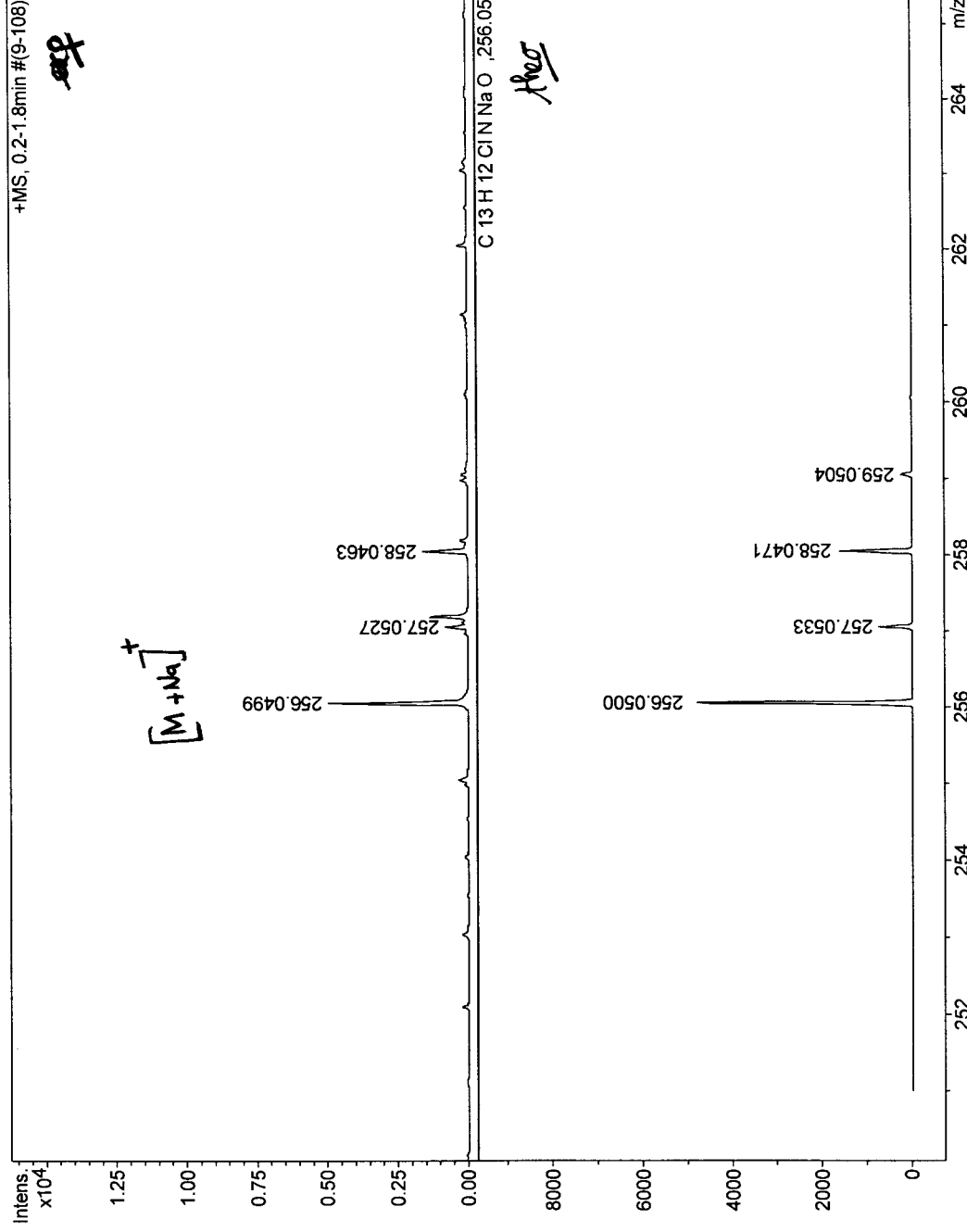
Operator Fanny Chaux
Instrument micrOTOF-Q 56

Acquisition Parameter

Source Type ESI
Focus Not active
Scan Begin 50 m/z
Scan End 3000 m/z

Ion Polarity Positive
Set Capillary 4500 V
Set End Plate Offset -500 V
Set Collision Cell RF 160.0 Vpp

Set Nebulizer 5.8 psi
Set Dry Heater 190 °C
Set Dry Gas 4.0 l/min
Set Divert Valve Source

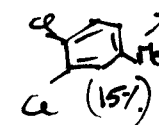
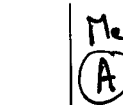
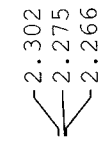
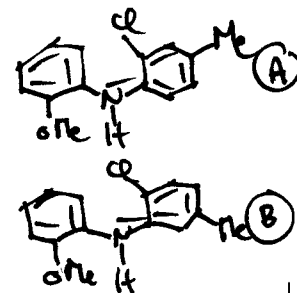
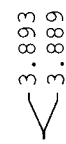
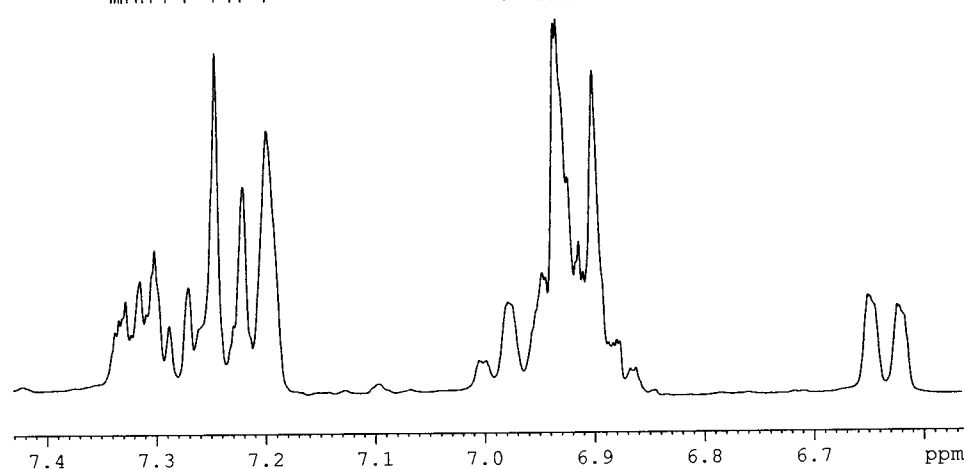
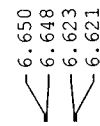
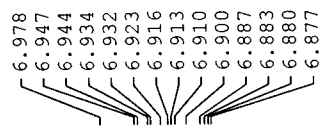
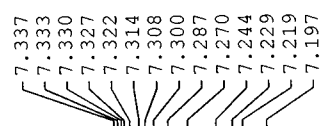
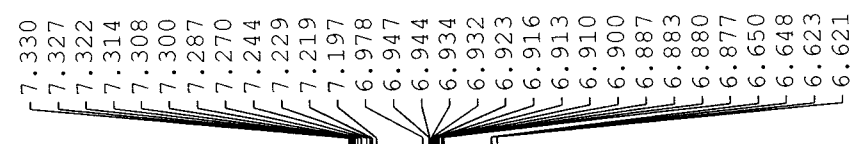


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdB	e ⁻ Conf	N-Rule
256.0499	1	C ¹³ H ₁₂ ClNNaO	100.00	256.0500	0.1	0.3	24.6	7.5	even	ok

(3c) + (3i)

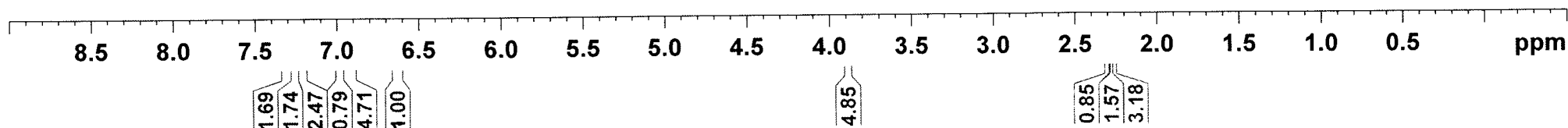
35

10mpl_2_91F1

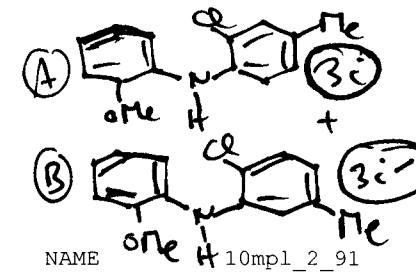
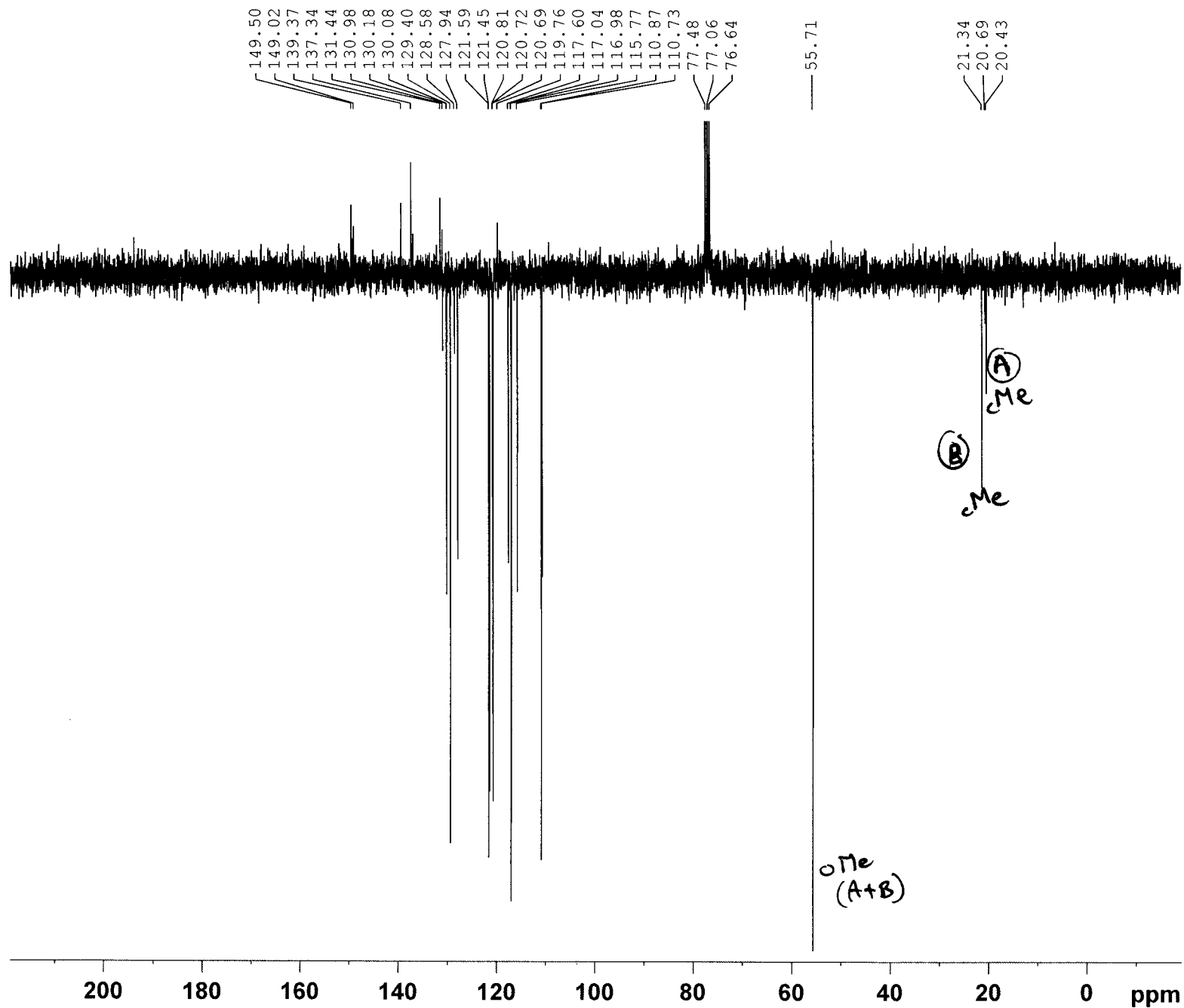


NAME 10mpl_2_91F1
EXPNO 1
PROCNO 1
Date_ 20100614
Time_ 12.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 203.2
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



10mpl_2_91



NAME 10mpl_2_91
 EXPNO 1
 PROCNO 1
 Date_ 20100622
 Time 7.01
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG jmod
 TD 65536
 SOLVENT CDC13
 NS 256
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 16384
 DW 27.800 usec
 DE 6.00 usec
 TE 300.0 K
 CNST2 145.0000000
 CNST11 1.0000000
 D1 2.00000000 sec
 d20 0.00689655 sec
 DELTA 0.00001070 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.40 usec
 p2 16.80 usec
 PL1 2.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 5.00 dB
 PL12 27.00 dB
 SFO2 300.1312005 MHz
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

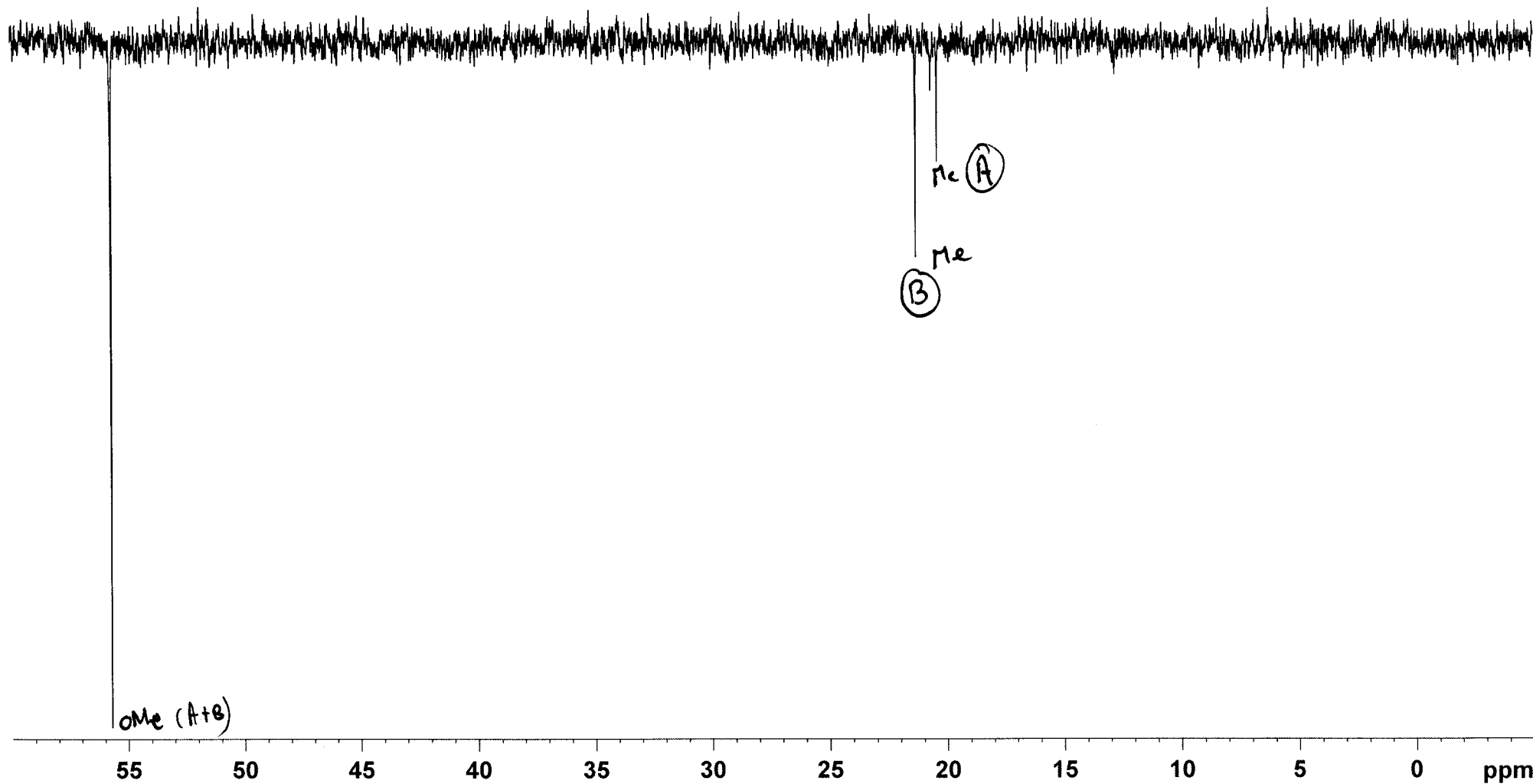
10mpl_2_91

$(3i) + (3i)$

37

55.71

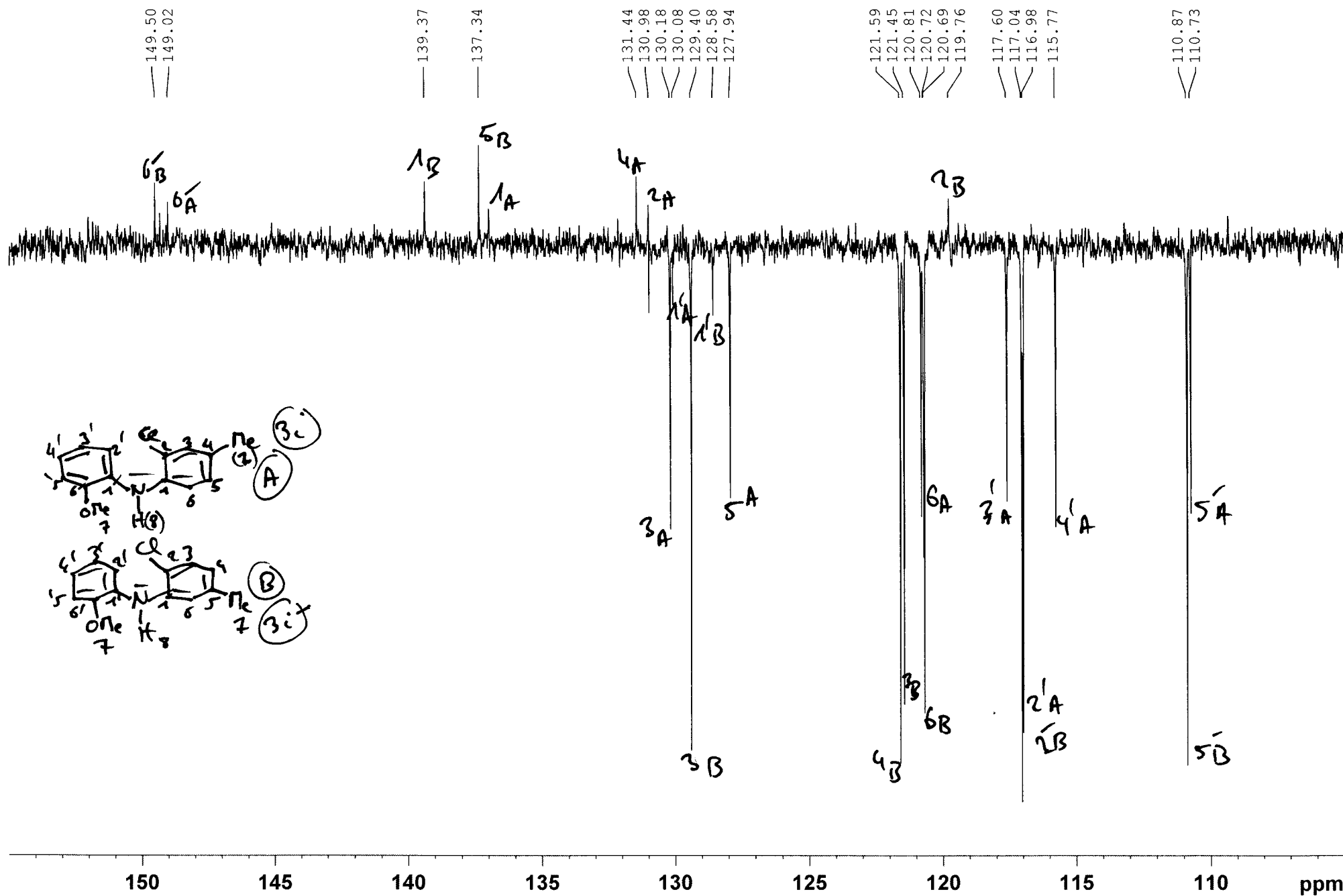
21.34
20.69
20.43



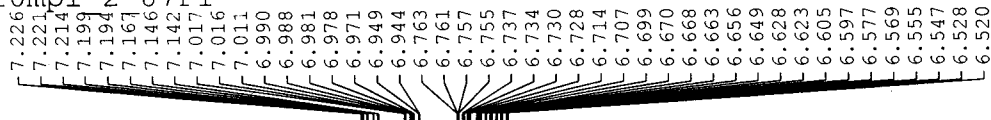
10mpl_2_91

(3i) + (3i')

38



10mpl 2 87F1



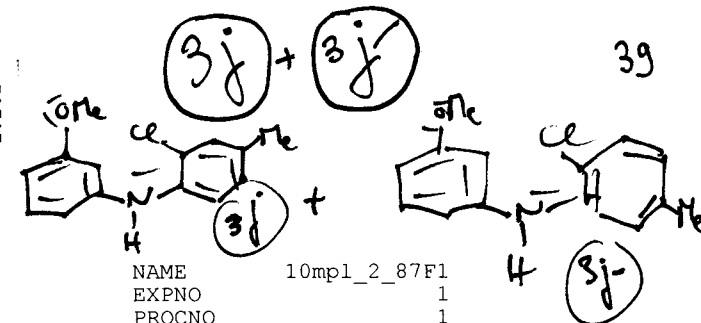
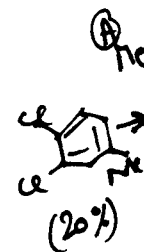
3.805
3.791

OMe (B)

OMe (A)

2.313
2.280
2.261

Me (B)



NAME 10mpl_2_87F1
EXPNO 1
PROCNO 1
Date_ 20100610
Time_ 9.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====

NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

0.96
3.34
4.25
2.13
2.20
1.89
2.36
2.79
2.61
3.32
0.94

6.46
3.00

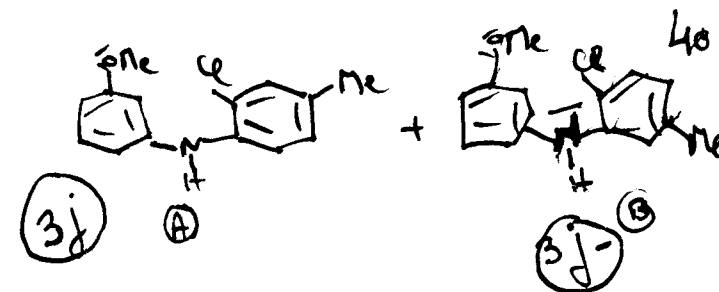
2.51
2.95
6.74

10mpl_2_87F1

7.325
7.298
7.269
7.260
7.253
7.241
7.234
7.226
7.221
7.214
7.199
7.194
7.167
7.146
7.142

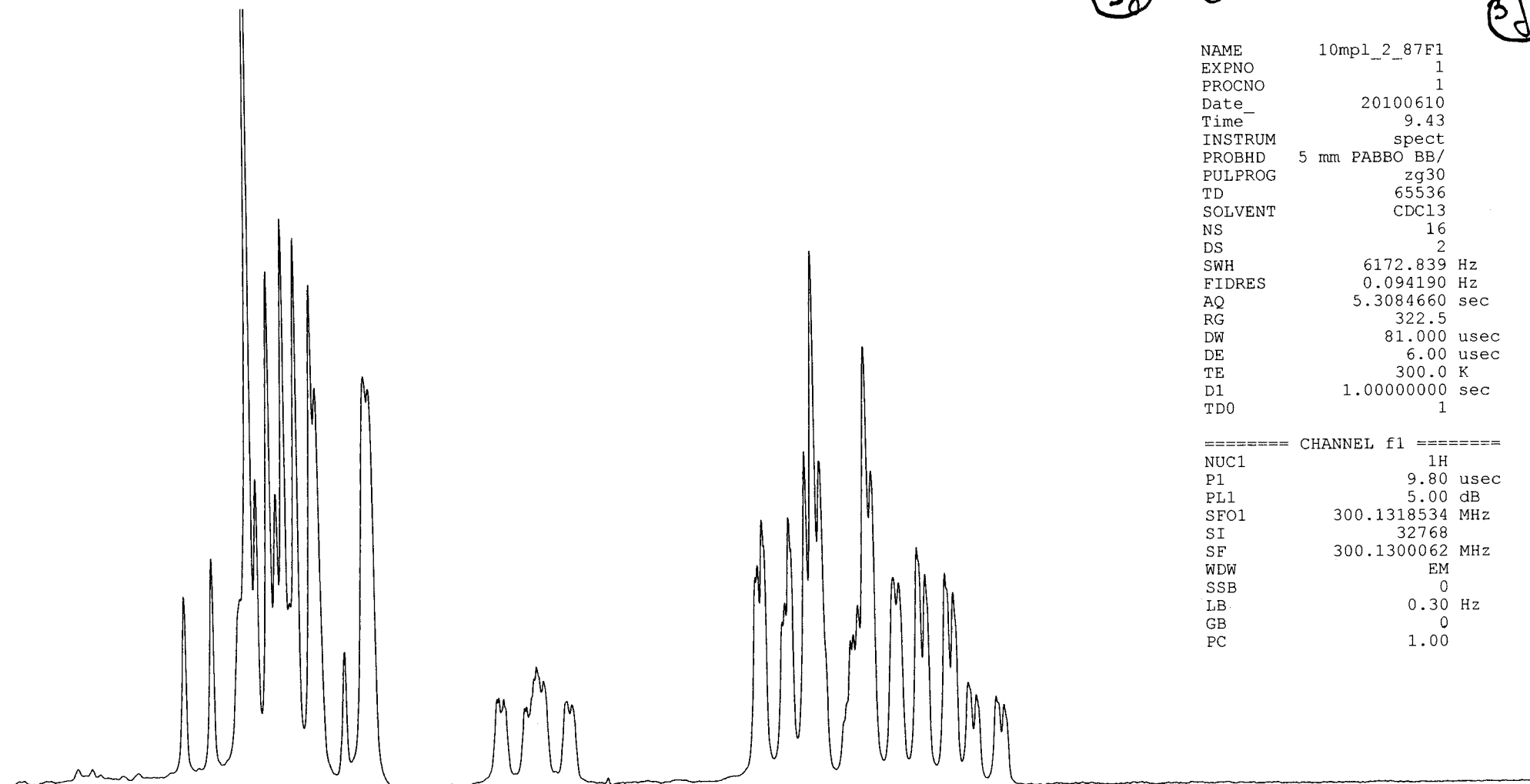
7.017
7.016
7.011
6.990
6.988
6.981
6.978
6.971
6.949
6.944

6.763
6.761
6.757
6.755
6.737
6.734
6.730
6.728
6.714
6.707
6.699
6.670
6.668
6.663
6.656
6.649
6.628
6.623
6.605
6.597
6.577
6.569
6.555
6.547
6.528
6.520



NAME 10mpl_2_87F1
EXPNO 1
PROCNO 1
Date_ 20100610
Time_ 9.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



7.4

0.96

3.34

4.25

2.13

2.20

7.1

1.89

6.9

6.8

2.36

2.79

2.61

6.6

3.32

0.94

6.5

6.4

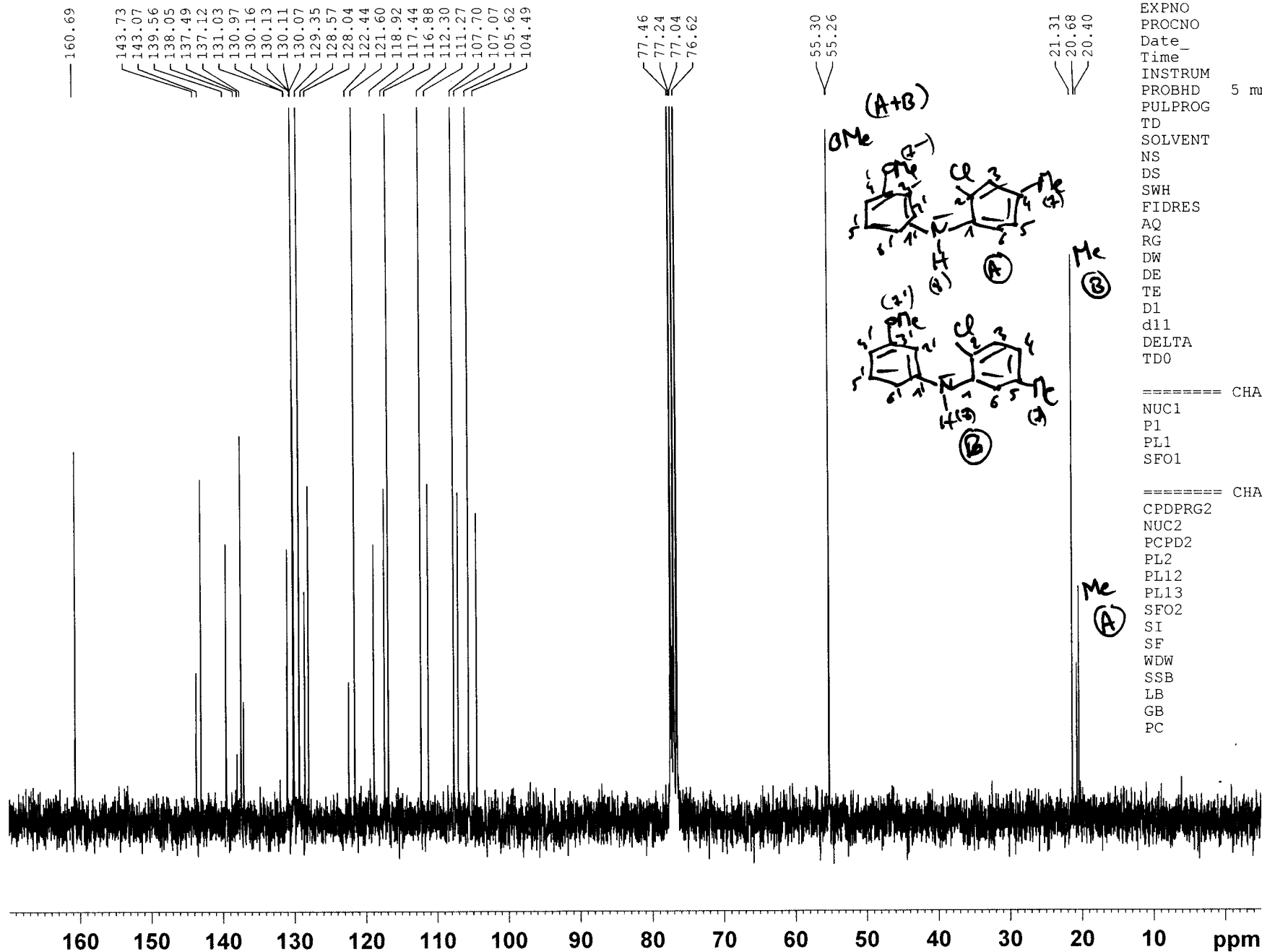
6.3

6.2

6.1

ppm

10mpl_2_87F1



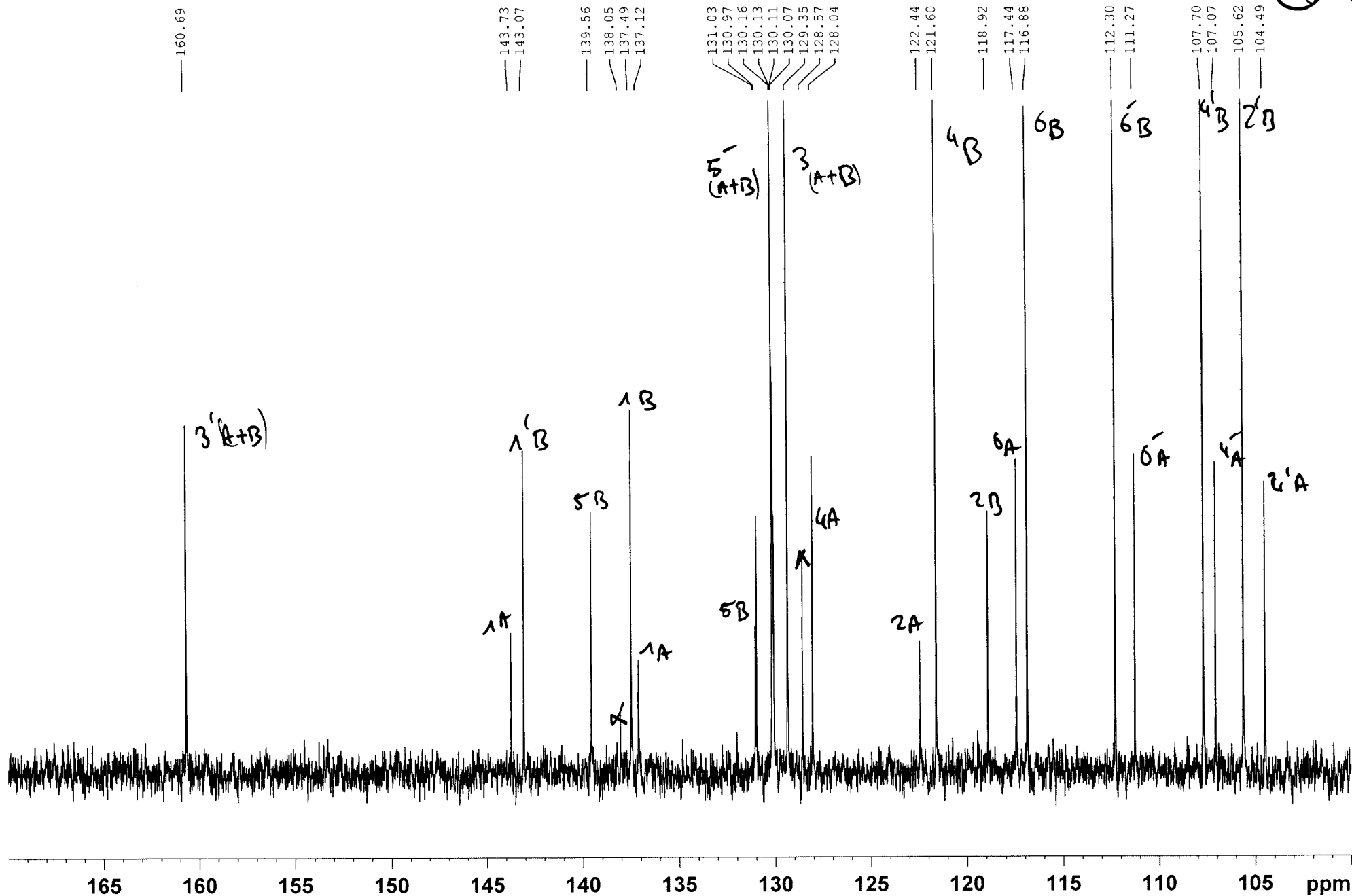
NAME 10mpl_2_87F1
 EXPNO 2
 PROCNO 1
 Date_ 20100611
 Time_ 6.10
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 18390.4
 DW 27.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.40 usec
 PL1 2.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 5.00 dB
 PL12 27.00 dB
 PL13 27.00 dB
 SFO2 300.1312005 MHz
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10mpl_2_87F1

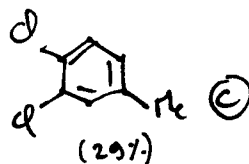
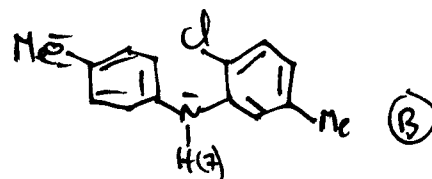
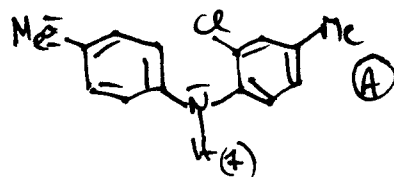
3j + 3j 42



10mpl-2_97

(3R) + (3S)

7.217
7.189
7.161
7.157
7.145
7.099
7.072
7.045
7.015
7.008
6.978
6.906
6.904
6.901
6.879
6.874
6.835
6.821
6.811
6.808
6.794
6.766
6.690
6.452
6.425
5.796



3.720
3.705

OMe
(B)

OMe
(A)

2.206
2.147
2.107

Me
(B)

(C)

Me
(A)

NAME 10mpl_2_97
EXPNO 1
PROCNO 1
Date_ 20100629
Time_ 15.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 90.5
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300409 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

(H4)(4)

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

0.69
0.87
1.12
3.92
0.68
4.46
1.00
1.01

3.00
1.59

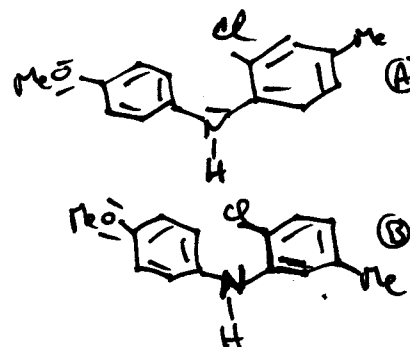
2.02
1.72
3.17
= 6.91
29% 25% 46%

10mpl-2_97

(3k) + (3k-)

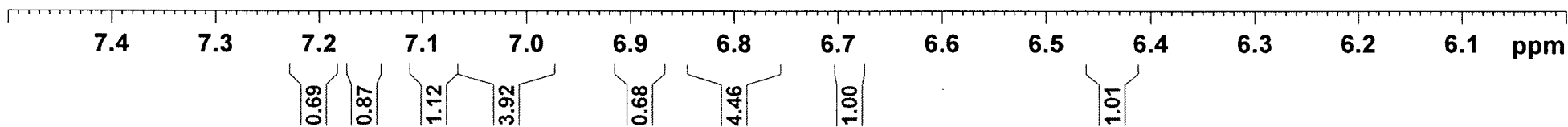
7.217
7.189
7.161
7.157
7.145
7.099
7.072
7.045
7.015
7.008
6.978
6.906
6.904
6.901
6.879
6.874
6.835
6.821
6.811
6.808
6.794
6.766
6.690

6.452
6.425



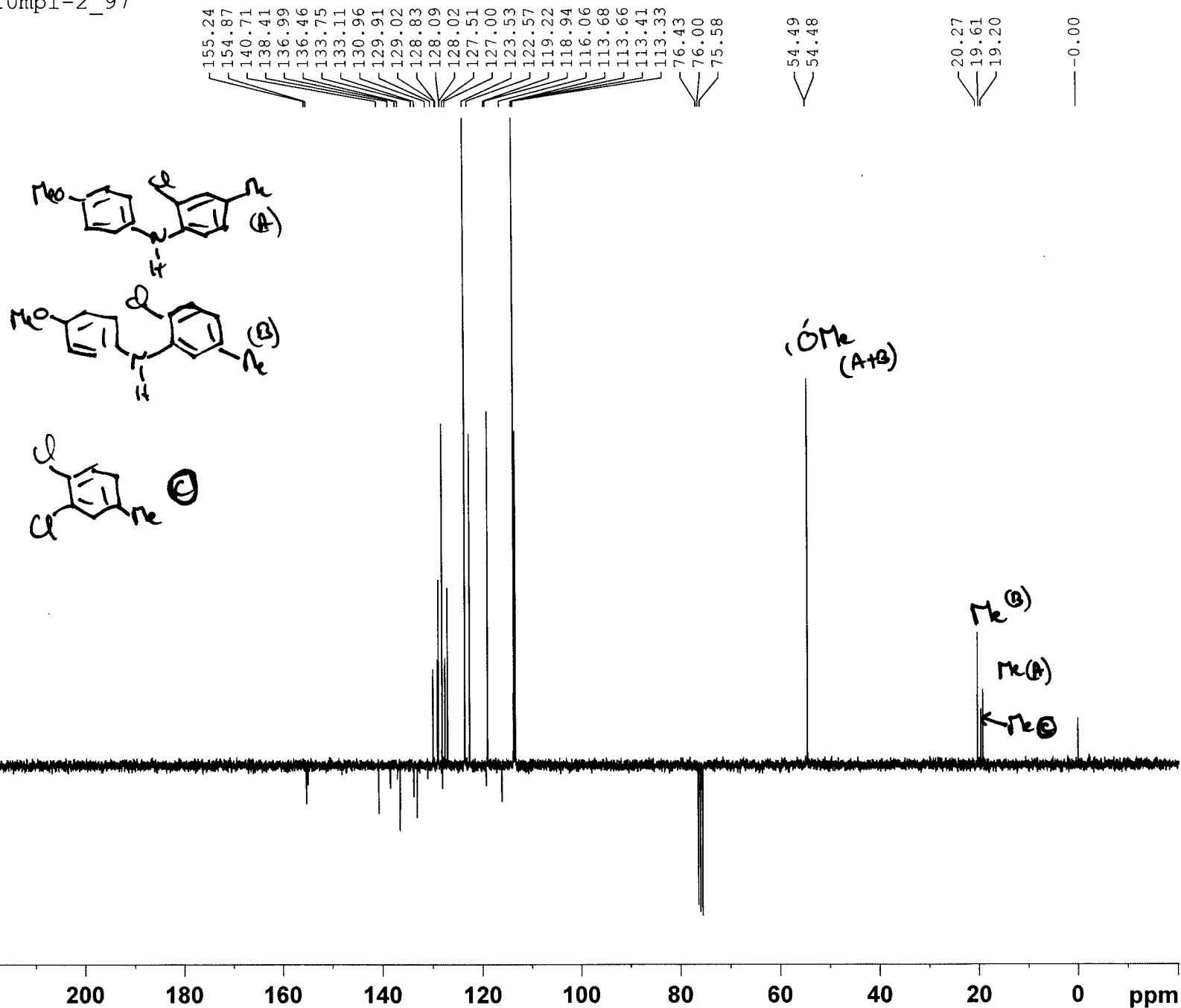
NAME 10mpl_2_97
EXPNO 1
PROCNO 1
Date_ 20100629
Time_ 15.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 90.5
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300409 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



10mpl-2_97

(3h) + (3h)



NAME 10mpl_2_97
 EXPNO 2
 PROCNO 1
 Date_ 20100630
 Time_ 8.04
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG jmod
 TD 65536
 SOLVENT CDC13
 NS 256
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 16384
 DW 27.800 usec
 DE 6.00 usec
 TE 300.0 K
 CNST2 145.0000000
 CNST11 1.0000000
 D1 2.00000000 sec
 d20 0.00689655 sec
 DELTA 0.00001070 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.40 usec
 p2 16.80 usec
 PL1 2.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 5.00 dB
 PL12 27.00 dB
 SFO2 300.1312005 MHz
 SI 32768
 SF 75.4678295 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10mpl-2_97

155.247
154.87

140.71

138.41

136.99

136.46

133.75

133.11

130.96

129.91

129.02

128.83

128.09

128.02

127.51

127.00

123.53

122.57

119.22

118.94

116.06

113.68

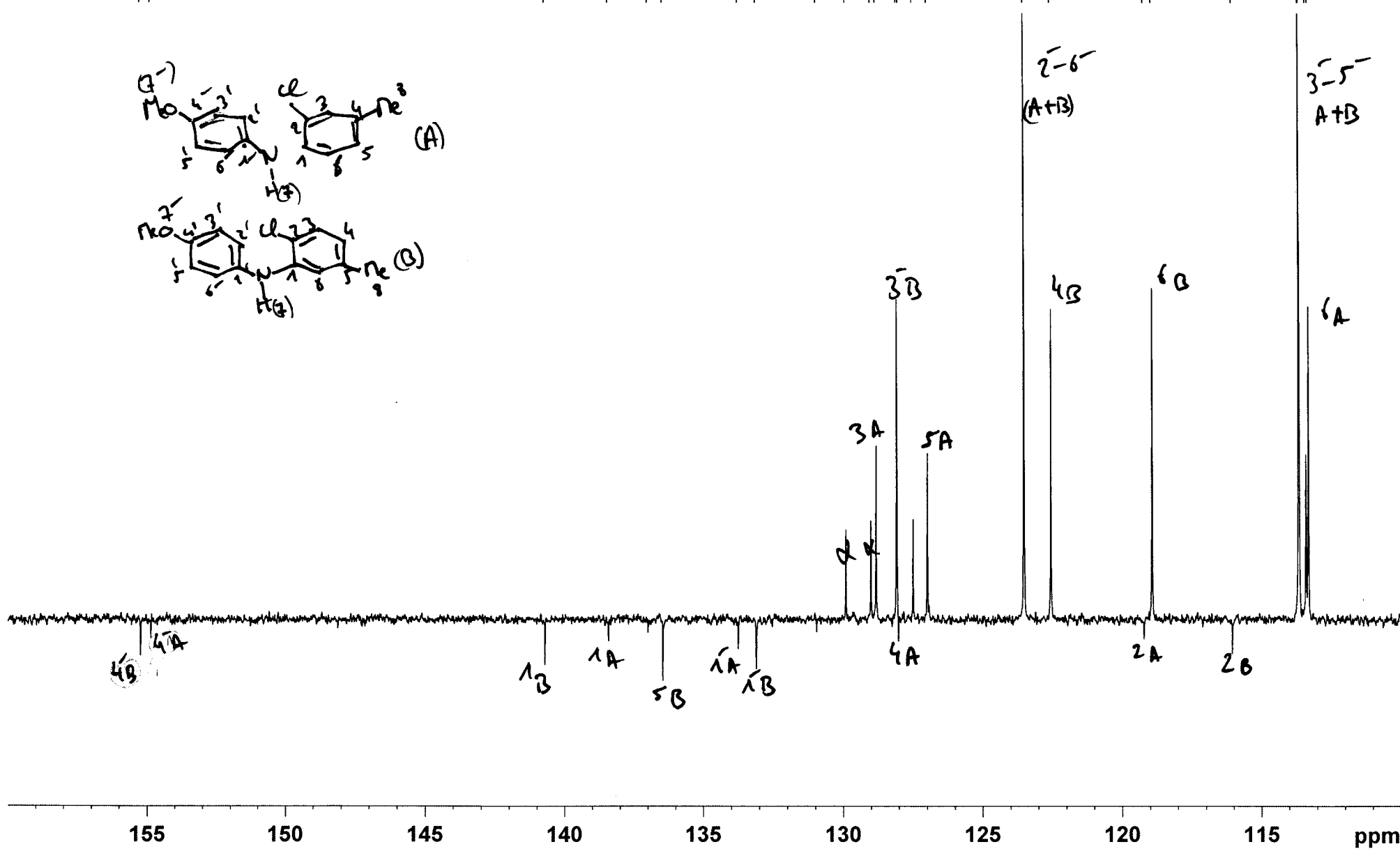
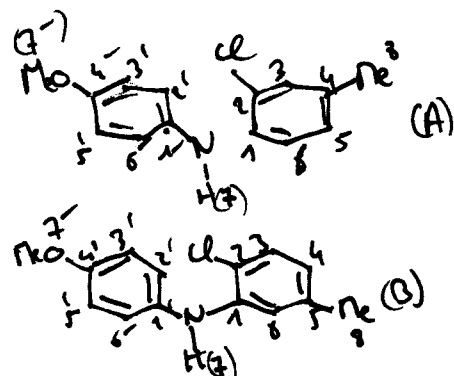
113.66

113.41

113.33

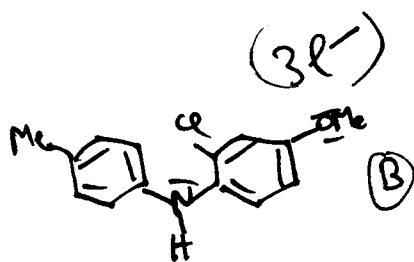
3k + 3k

46



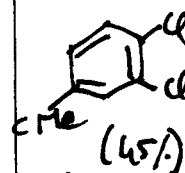
10mpl_2_95

7.338
7.308
7.260
7.232
7.203
7.162
7.134
7.102
7.074
6.999
6.990
6.955
6.779
6.770
6.750
6.740
6.723
6.714
6.347
6.338
6.318
6.309



3.789
3.775
3.710

OMe
(B) (55%)



2.337
2.305

Me
(B)

* Tol

NAME 10mpl_2_95
EXPNO 1
PROCNO 1
Date 20100613
Time 23.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 287.4
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

1.00
0.42
0.87
3.47
0.92
0.31
1.05
0.73
0.73

2.68
0.43
2.20
= 5.31

2.23
0.30

55% 8% 41%

(3l-)

47

10mpl_2_95

32

48

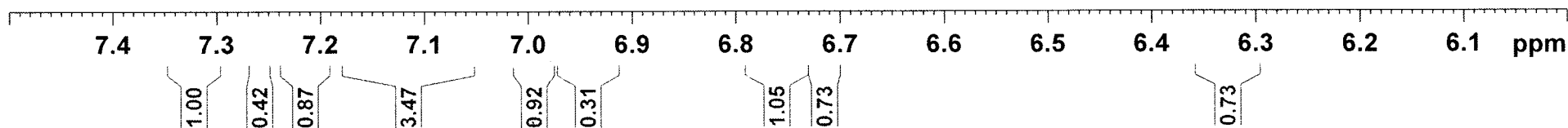
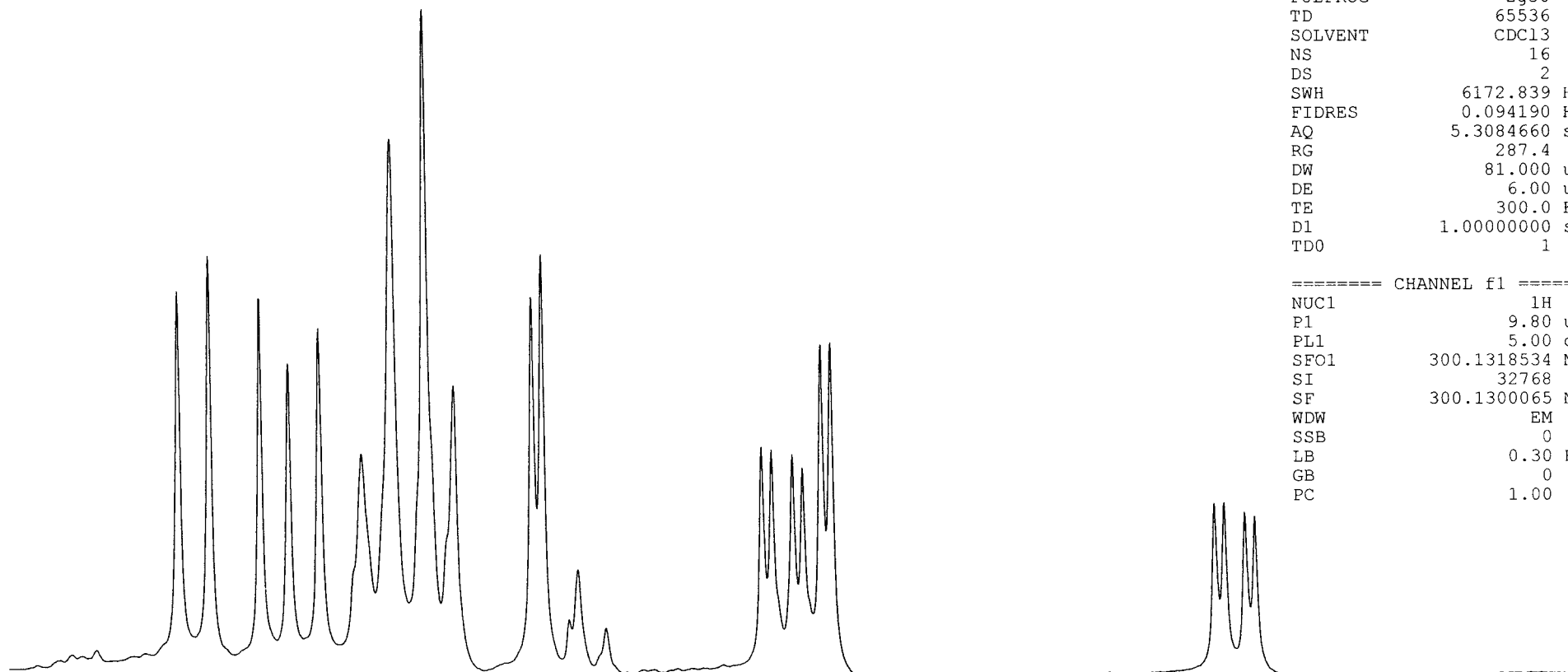
7.338
7.308
7.260
7.232
7.203
7.162
7.134
7.102
7.074
6.999
6.990
6.955

6.779
6.770
6.750
6.740
6.723
6.714

6.347
6.338
6.318
6.309

NAME 10mpl_2_95
EXPNO 1
PROCNO 1
Date_ 20100613
Time_ 23.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 287.4
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



10mpl_2_95

38

49

158.14
157.61

140.77
137.36
131.88
131.81
129.63
128.99
128.87
128.81
122.86
120.54
117.88
117.67
114.66
114.05
113.08
112.61
111.50
103.95
99.52

76.42
76.20
76.00
75.57

54.81
54.70
54.39

19.79
19.64

NAME 10mpl_2_95
EXPNO 2
PROCNO 1
Date_ 20100614
Time_ 0.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 20642.5
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999999 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 5.00 dB
PL12 27.00 dB
PL13 27.00 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4678278 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

C-OMe
C-OMe
(dichloride)

OMe(B)
OMe
(dichloride)

Me

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

10mpl_2_95

38

50

158.14
157.61

140.77

137.36

131.88
131.81
129.63
128.99
128.87
128.81

122.86

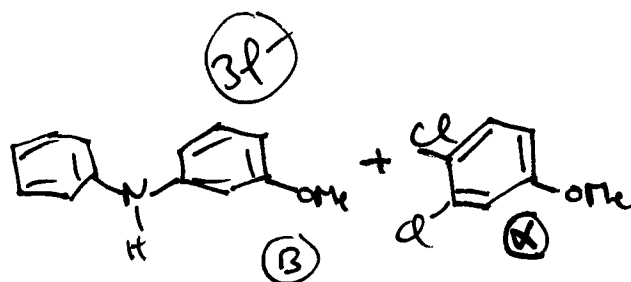
120.54

117.88
117.67

114.66
114.05
113.08
112.61
111.50

103.95

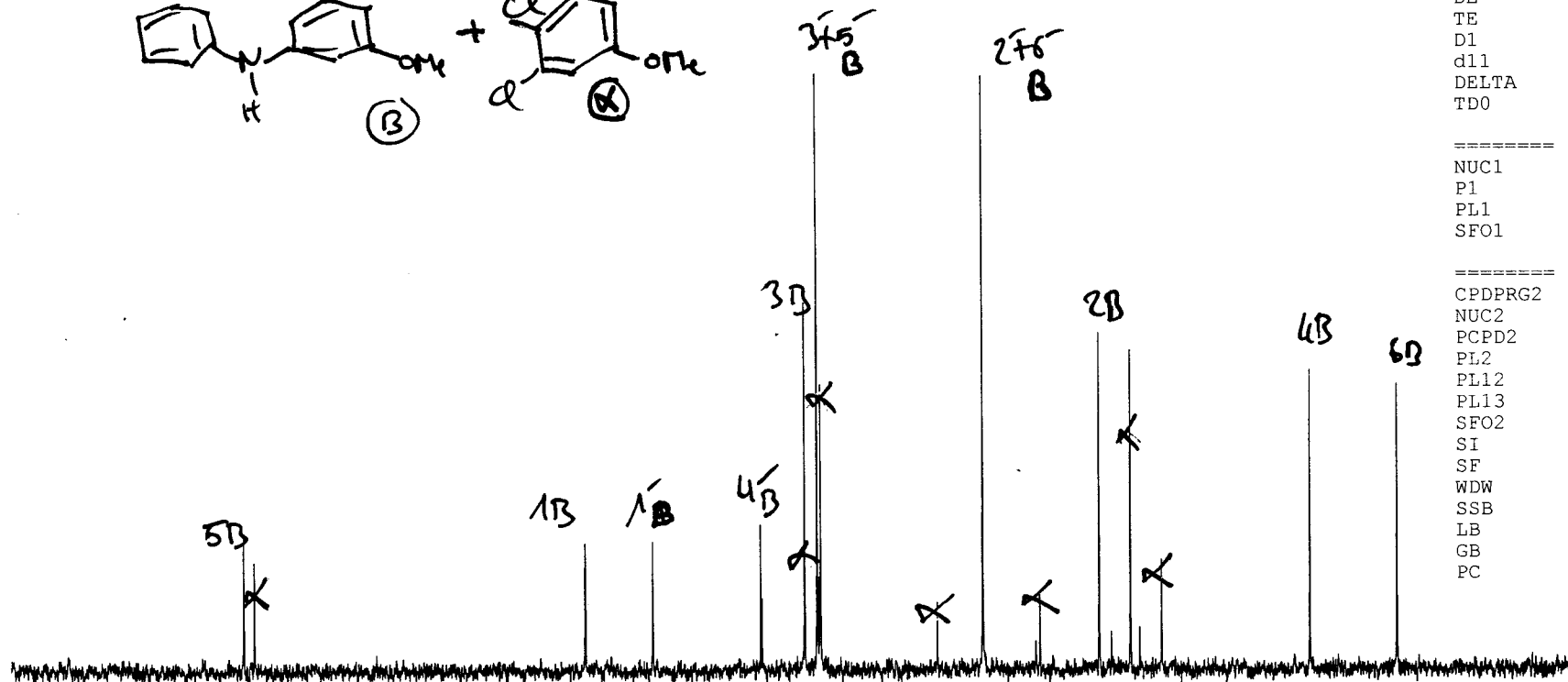
99.52



NAME 10mpl_2_95
EXPNO 2
PROCNO 1
Date_ 20100614
Time_ 0.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 20642.5
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 5.00 dB
PL12 27.00 dB
PL13 27.00 dB
SFO2 300.1312005 MHz
SI 32768
SF 75.4678278 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

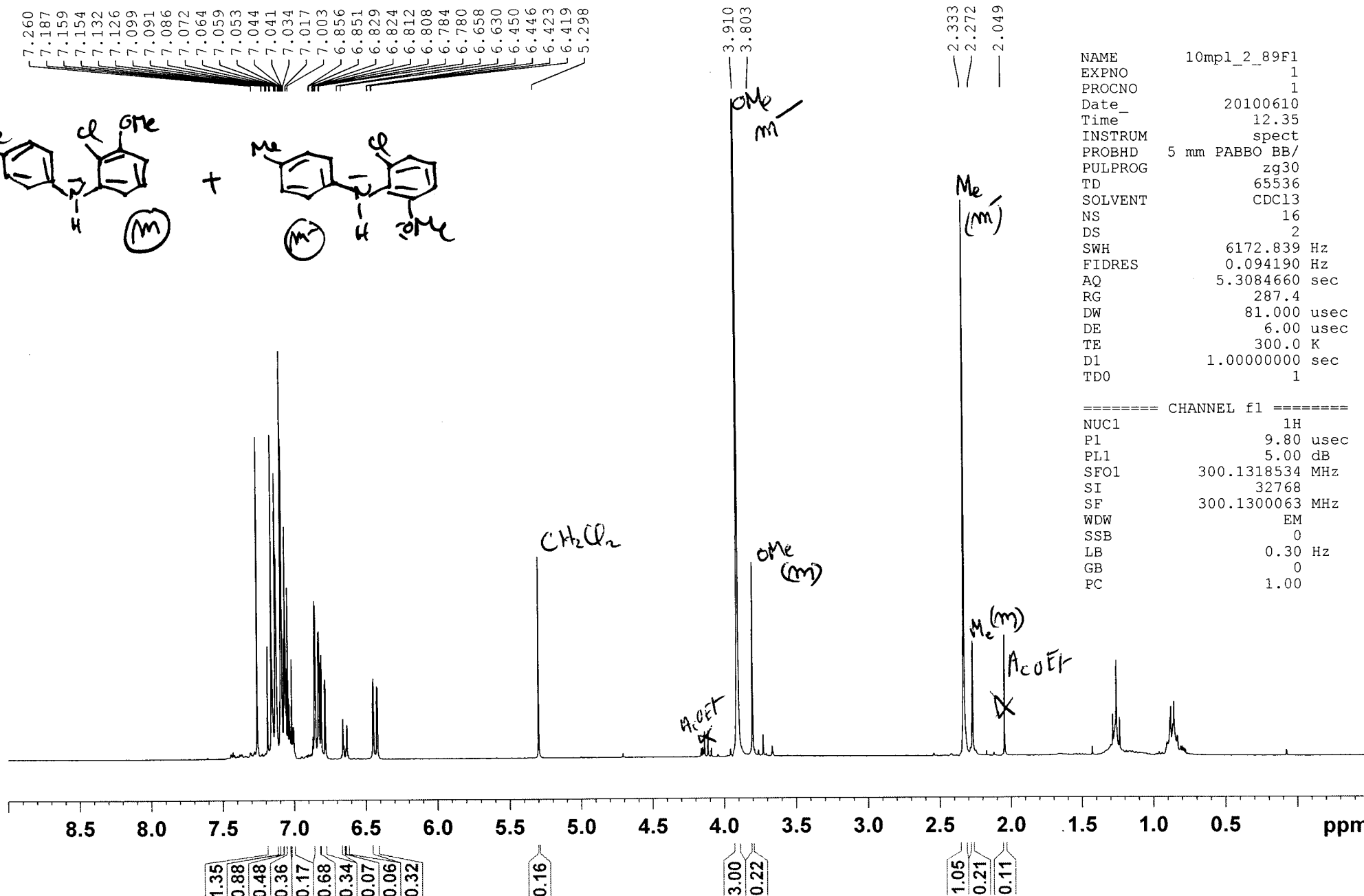
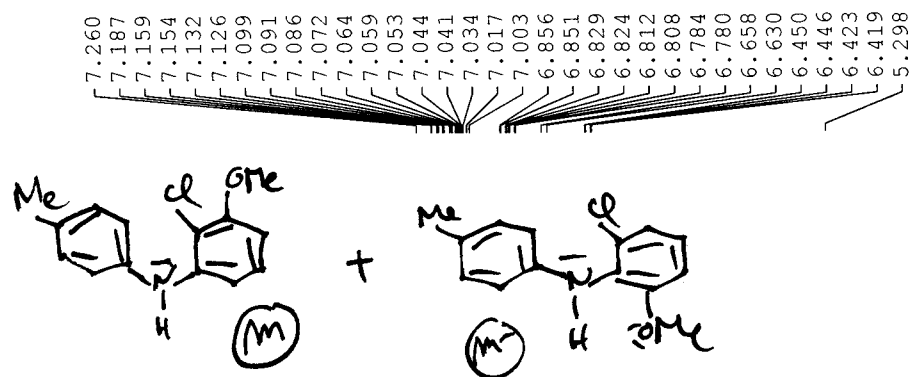


165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 ppm

10mpl_2_89F1

(3m) + (3m)

51



NAME 10mpl_2_89F1
EXPNO 1
PROCNO 1
Date_ 20100610
Time 12.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 287.4
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300063 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10mpl_2_89F1

3m + 3m

52

7.260
7.187
7.159
7.154
7.132
7.126
7.099
7.091
7.086
7.072
7.064
7.059
7.053
7.044
7.041
7.034
7.017
7.003

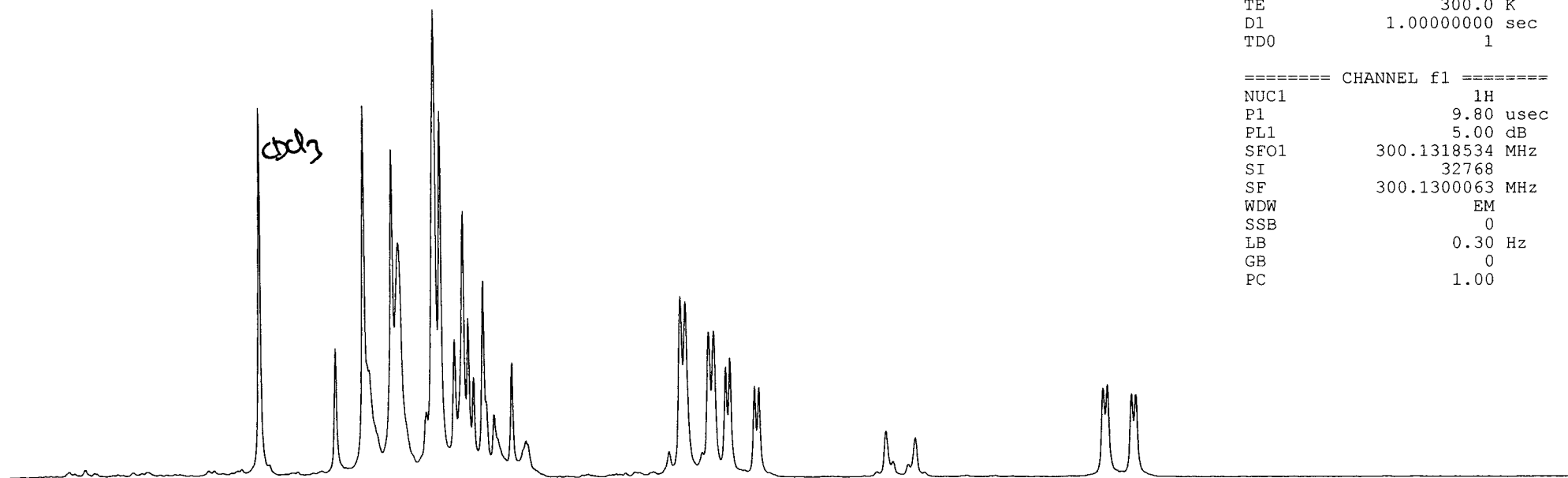
6.856
6.851
6.829
6.824
6.812
6.808
6.784
6.780

6.658
6.630

6.450
6.446
6.423
6.419

NAME 10mpl_2_89F1
EXPNO 1
PROCNO 1
Date_ 20100610
Time 12.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 287.4
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.80 usec
PL1 5.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300063 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 6.1 ppm

1.35
0.88
0.48
0.36
0.17

0.68
0.34

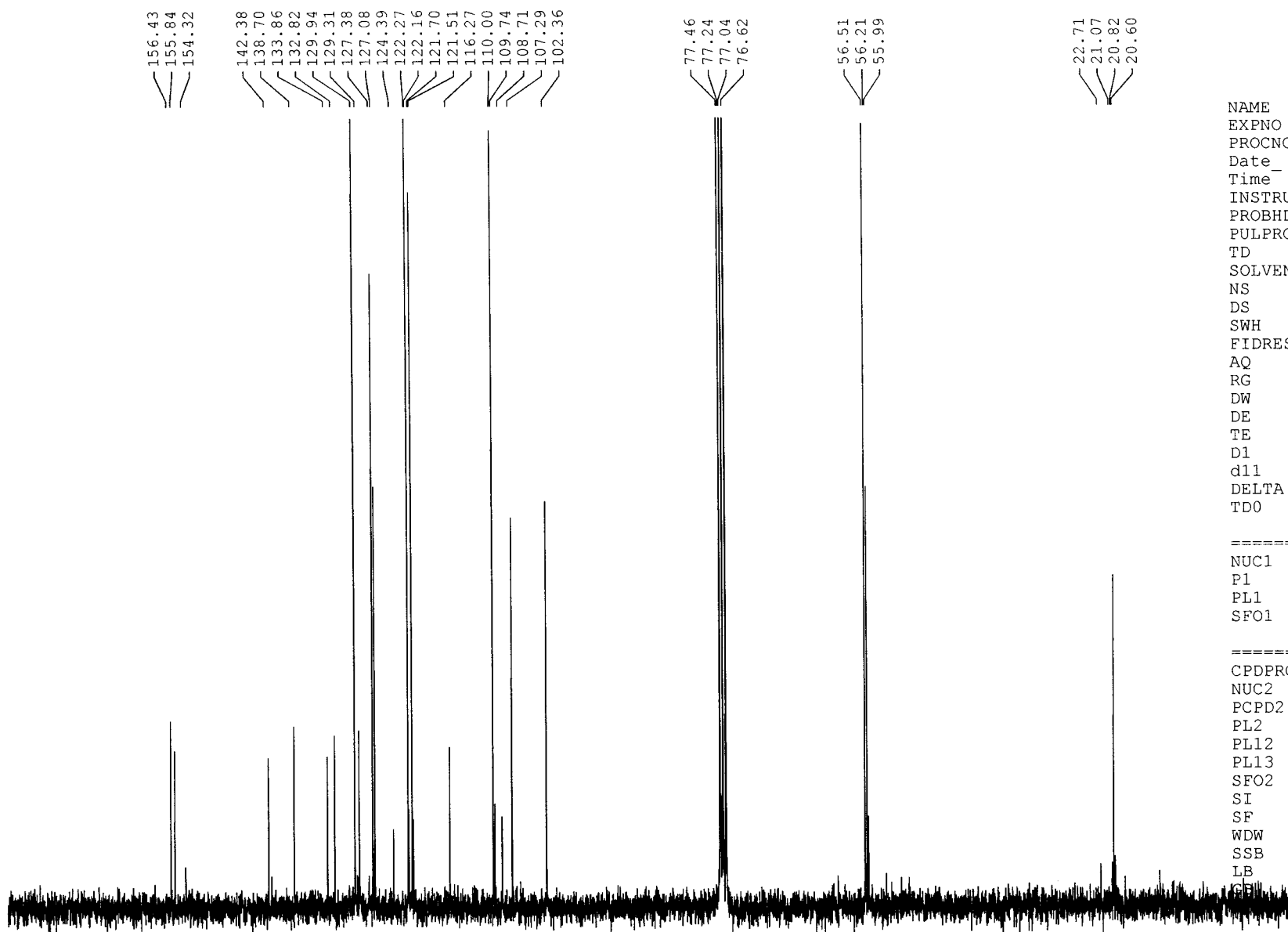
0.07
0.06

0.32

10mpl_2_89F1

3m+3m

53



NAME 10mpl_2_89F1
 EXPNO 2
 PROCNO 1
 Date_ 20100611
 Time_ 20.16
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 1024
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 20642.5
 DW 27.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.40 usec
 PL1 2.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 5.00 dB
 PL12 27.00 dB
 PL13 27.00 dB
 SFO2 300.1312005 MHz
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 1.40

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

10mp1_2_89F1

3m+3m 54

156.43
155.84
154.32

142.38

138.70

133.86
132.82

129.94
129.31
127.38
127.08

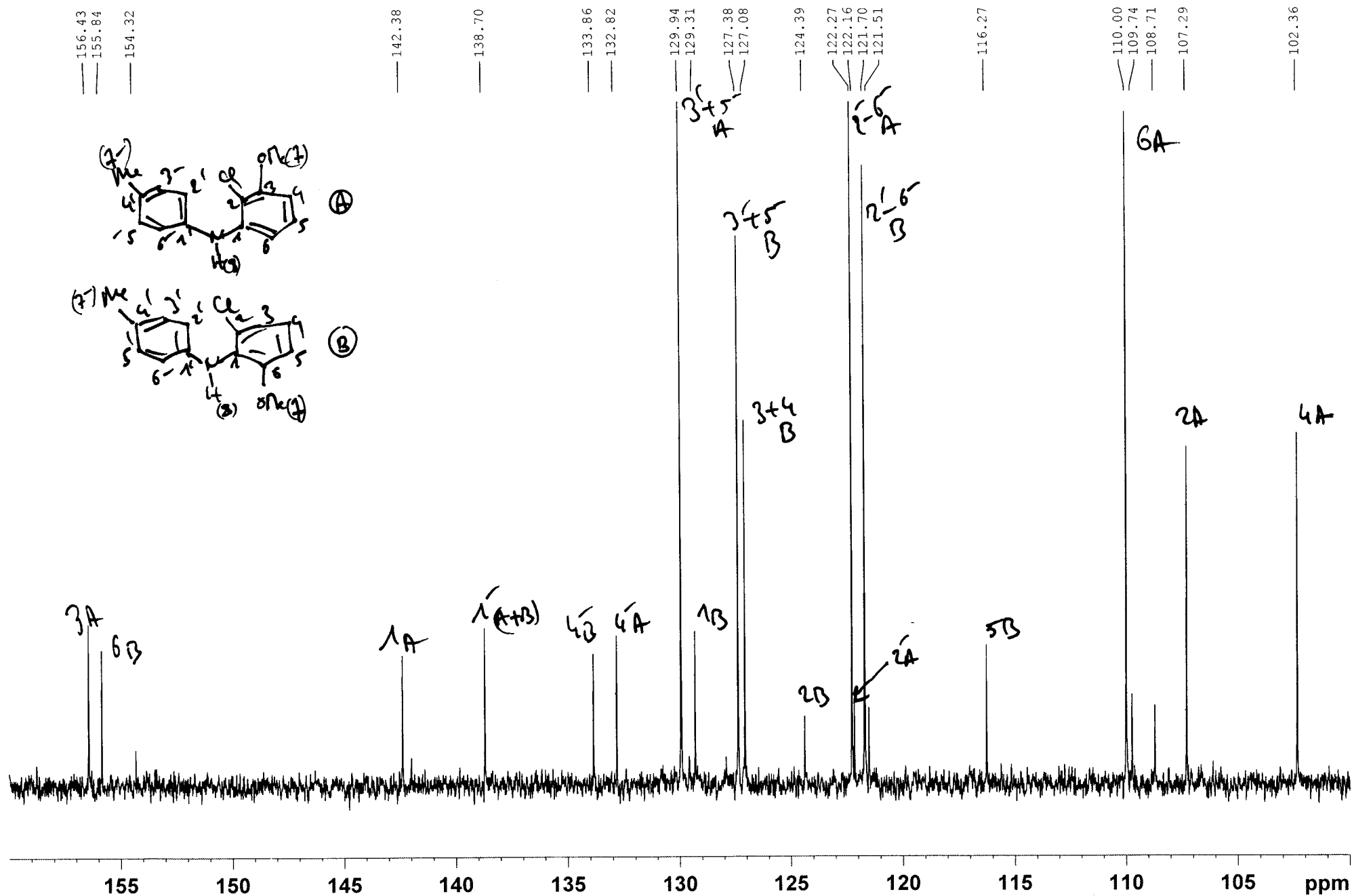
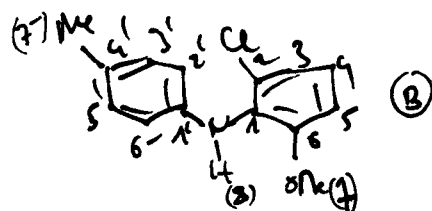
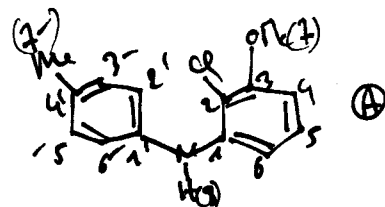
124.39

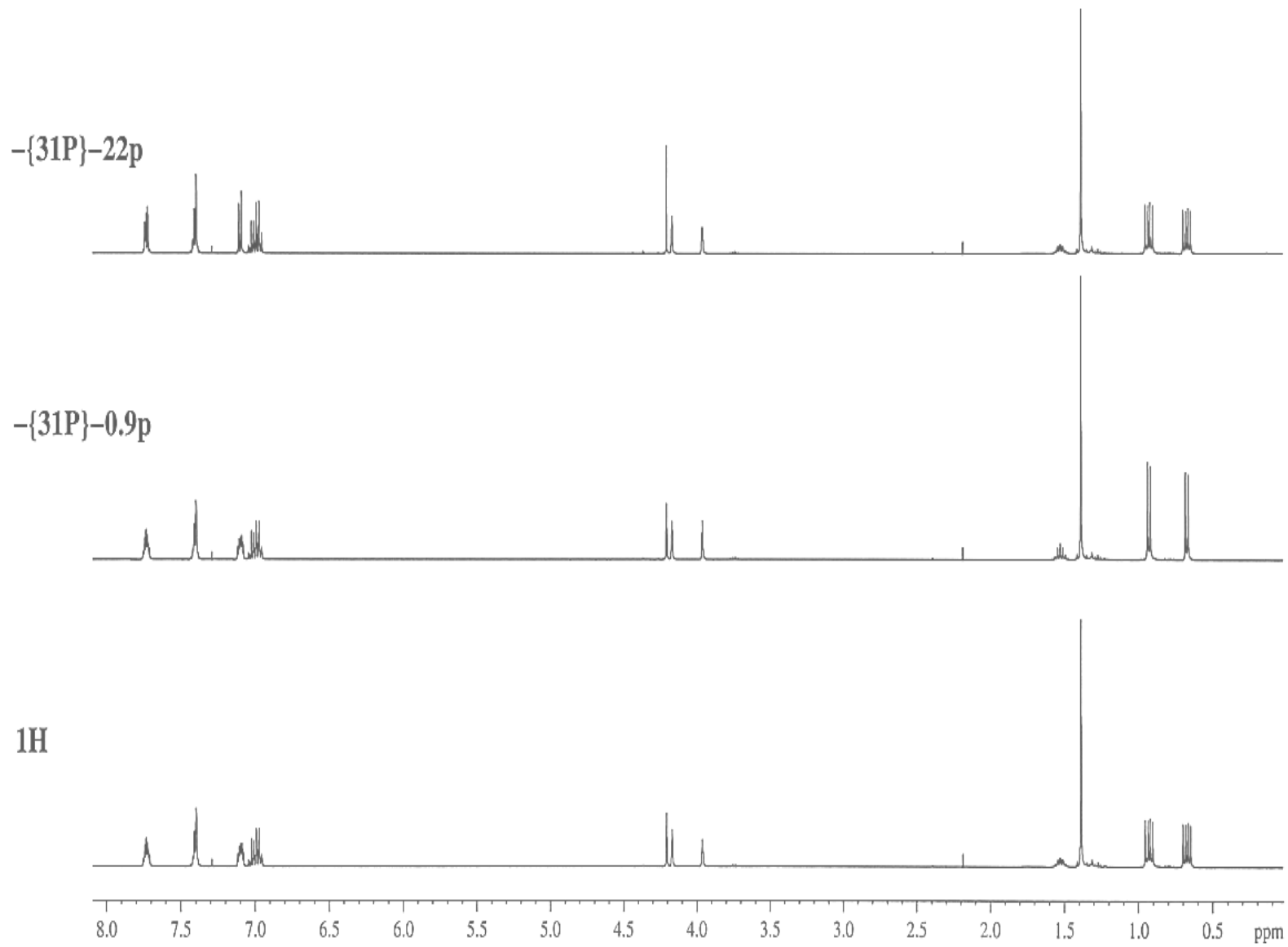
122.27
122.16
121.70
121.51

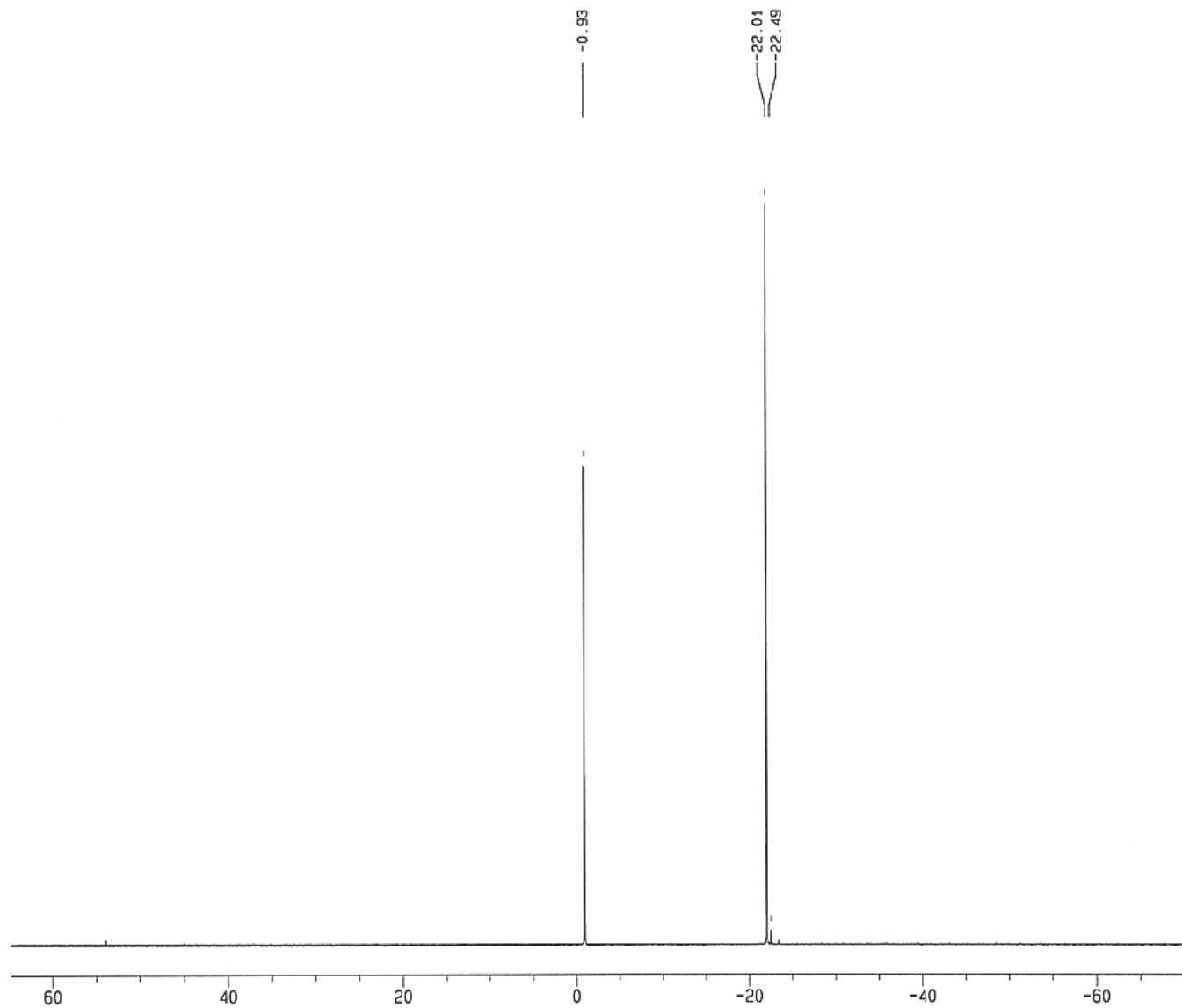
116.27

110.00
109.74
108.71
107.29

102.36



^1H NMR spectrum for L5

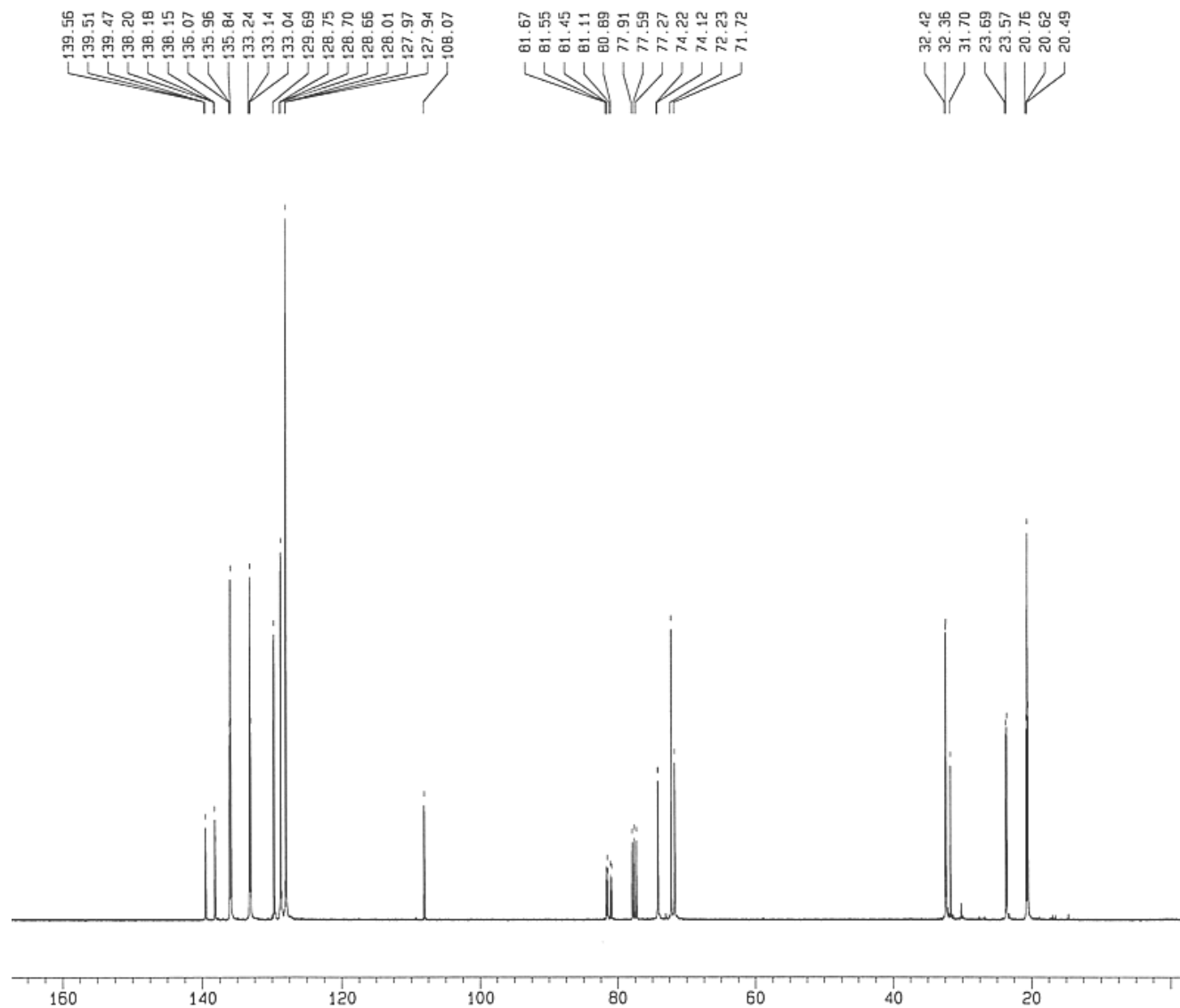
³¹P NMR spectrum for L5

Current Data Parameters
 NAME hier1117
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20031117
 Time 15.20
 INSTRUM amx400
 PROBHD 5 mm TBI Z-g
 PULPROG zg60dcsave
 TD 66662
 SOLVENT CDC13
 NS 8
 DS 4
 SWH 22727.273 Hz
 FIDRES 0.340933 Hz
 AQ 1.4666140 sec
 RG 8192
 DW 22.000 usec
 DE 31.43 usec
 TE 293.0 K
 D11 0.0300000 sec
 CPDPRG waltz16
 P31 88.00 usec
 S2 25 dB
 HL1 90 dB
 D1 1.00000000 sec
 P1 13.00 usec
 SF01 161.9751360 MHz
 NUCLEUS 31P
 L4 1

F2 - Processing parameters
 SI 131072
 SF 161.9751360 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0

1D NMR plot parameters
 CX 22.00 cm
 F1P 70.157 ppm
 F1 11363.64 Hz
 F2P -70.157 ppm
 F2 -11363.64 Hz
 PPMCM 6.37788 ppm/cm
 HZCM 1033.05786 Hz/cm

¹³C NMR spectrum for L5

Current Data Parameters
 NAME hier1117
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20031117
 Time 15.49
 INSTRUM amx400
 PROBHD 5 mm TBI Z-g
 PULPROG zg60_xcpd1hcwys
 TD 55552
 SOLVENT CDCL3
 NS 5200
 DS 4
 SWH 18518.518 Hz
 FIDRES 0.333355 Hz
 AQ 1.4999540 sec
 RG 32768
 DW 27.000 usec
 DE 38.57 usec
 TE 293.0 K
 HL1 90 dB
 D11 0.03000000 sec
 DBL0 44.00 dB
 D1 1.00000000 sec
 CPDPRG waltz16
 P31 88.00 usec
 S2 25 dB
 P1 10.40 usec
 SF01 100.6217290 MHz
 NUCLEUS 13C
 L4 26

F2 - Processing parameters

SI 131072
 SF 100.6127290 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0

1D NMR plot parameters

CX 22.00 cm
 F1P 181.481 ppm
 F1 18259.26 Hz
 F2P -2.577 ppm
 F2 -259.26 Hz
 PPMCM 8.36625 ppm/cm
 HZCM 841.75092 Hz/cm