

The synthesis of a series of adenosine A₃ receptor agonists

Kenneth J. Broadley, Erica Burnell, Robin H. Davies, Alan T. L. Lee, Stephen Snee and Eric J. Thomas

Supplementary Data

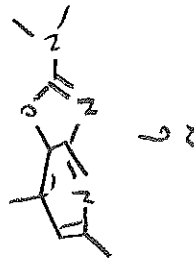
Copies of ¹H and ¹³C NMR spectra of key compounds – in numerical order.

atl1502
STANDARD 1H OBSERVE

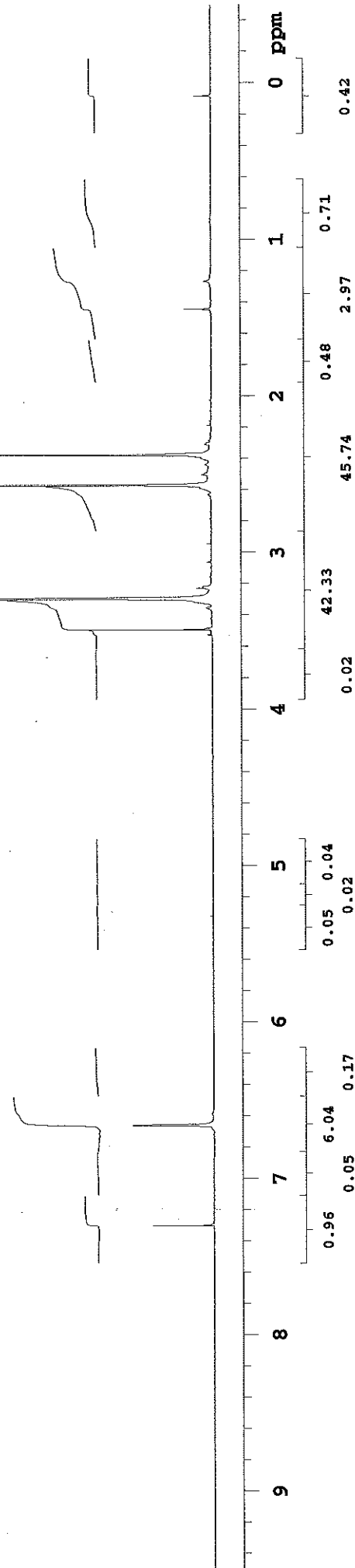
Automation directory: /data/nmrdata/Nov20
File : 1201

Pulse Sequence: s2pul

Solvent: cdcl3
DATE Nov 20 2006
Sample #12
File: 1201
INOVA-300 "var300b"

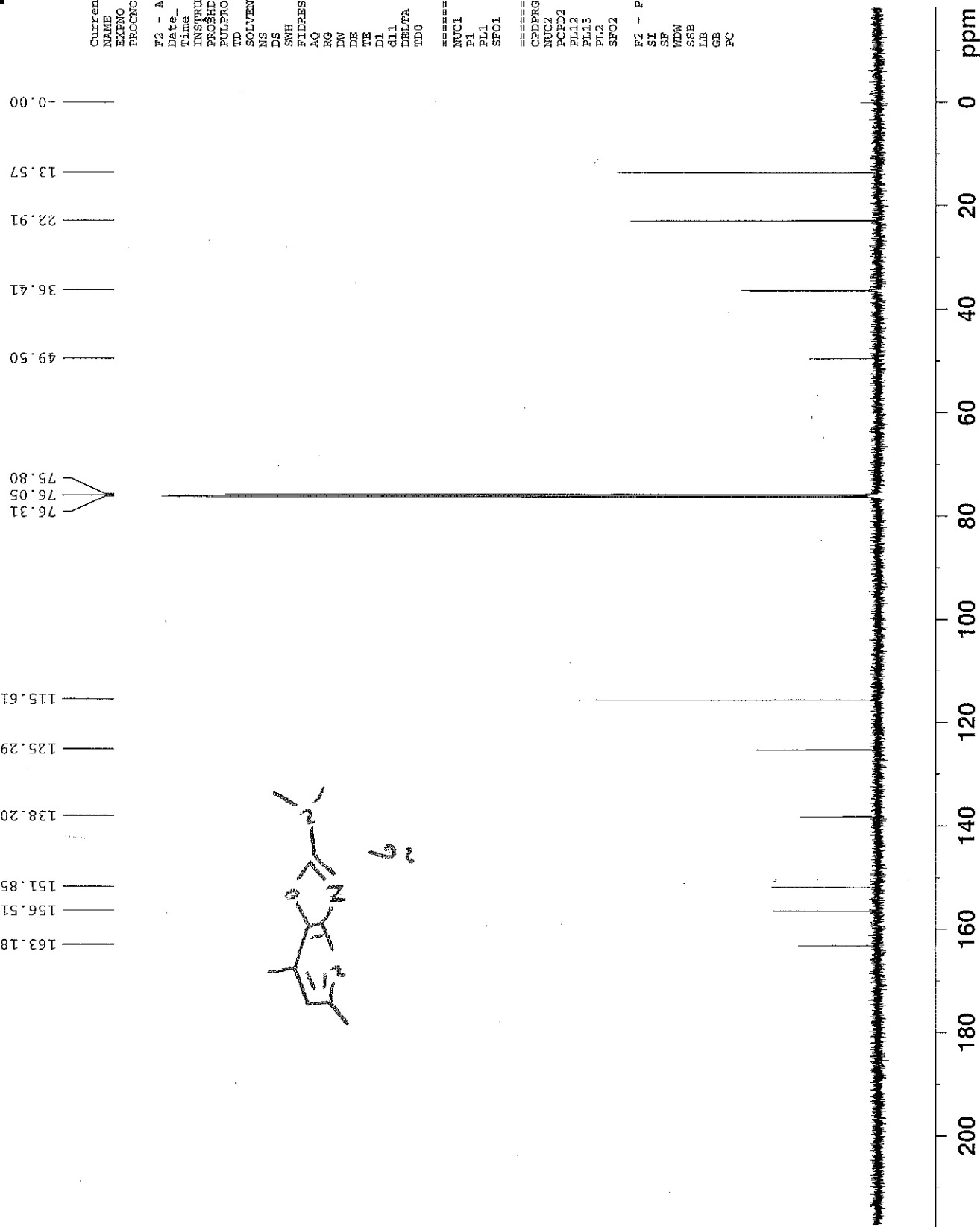


Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.744 sec
Width 4797.6 Hz
16 repetitions
OBSERVE H1, 299.8458792 MHz
DATA PROCESSING
Ft size 65536
Total time 1 min, 16 sec



atl1517

mCARBONnight CDCl3 /opt/topspin ejt 15



```

Current Data Parameters
NAME      2006-12-10-ejt-15
EXPNO     21
PROCNO    1

F2 - Acquisition Parameters
Date_     20061211
Time      4.13
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         2
SWH        29761.904 Hz
FIDRES     0.454131 Hz
AQ         1.1010548 sec
RG         512
DE         16.800 usec
TE         300.0 K
DL         2.0000000 sec
d11        0.0300000 sec
DELTA     1.89999998 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         7.30 usec
PL1        -4.50 dB
SFO1       125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL12       17.75 dB
PL13       120.00 dB
PL2        -0.30 dB
SFO2       500.1320005 MHz

F2 - Processing parameters
SI         32768
SF         125.7579164 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

atll n-oxide
mPROTON MeOD /opt/topspin ejt 35

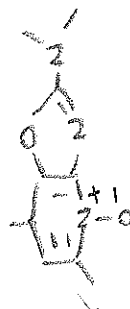


Current Data Parameters
NAME 2007-06-24-ejt-35
EXPNO 10
PROCNO 1

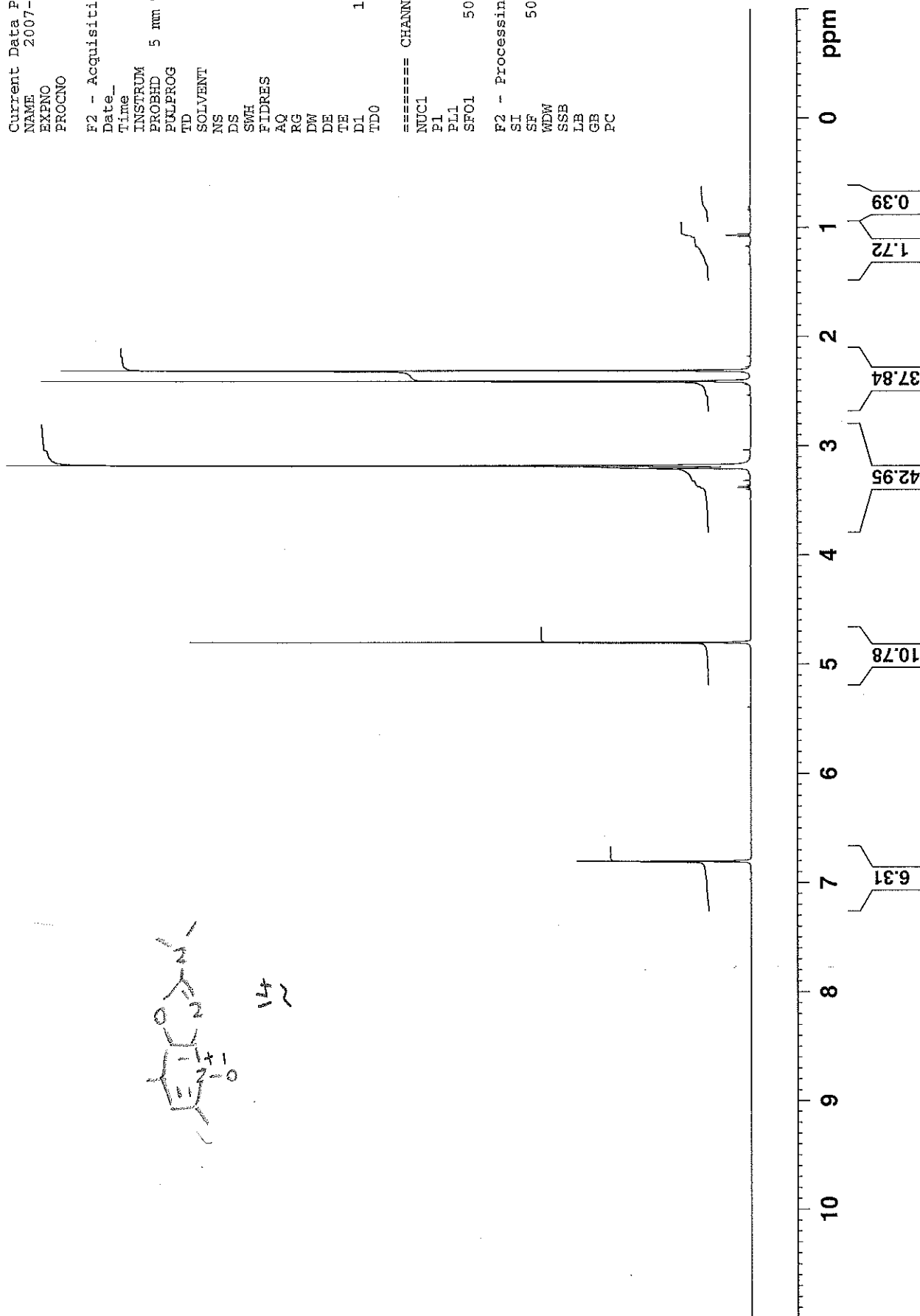
F2 - Acquisition Parameters
Date_ 20070625
Time 7.04
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zg30b
TD 65536
SOLVENT MeOD
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 203
DW 48.400 usec
DE 13.52 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

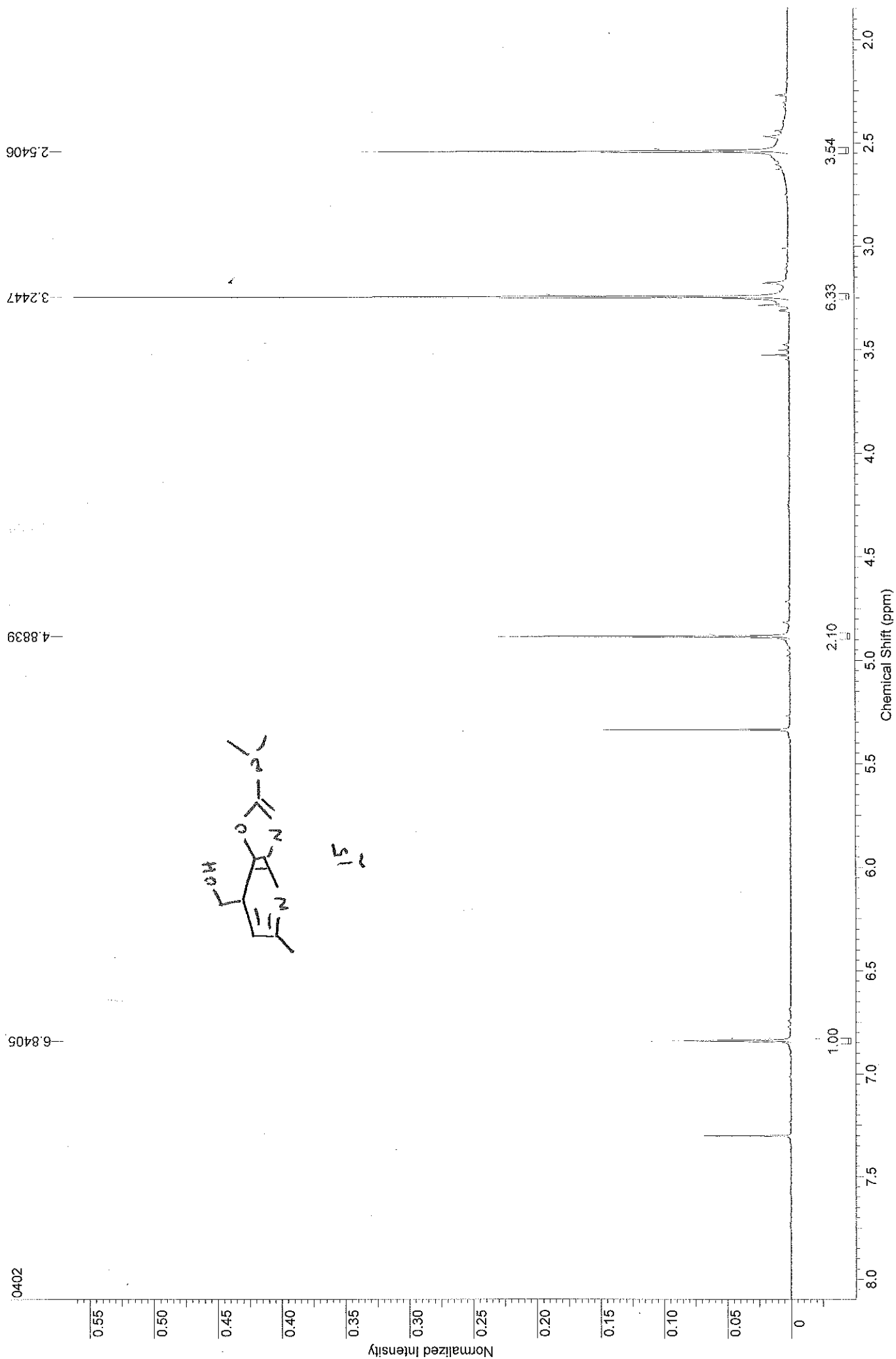
==== CHANNEL f1 =====
NUC1 1H
P1 37.76 usec
PL1 3.25 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300629 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



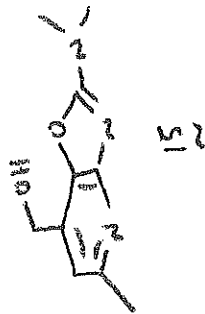
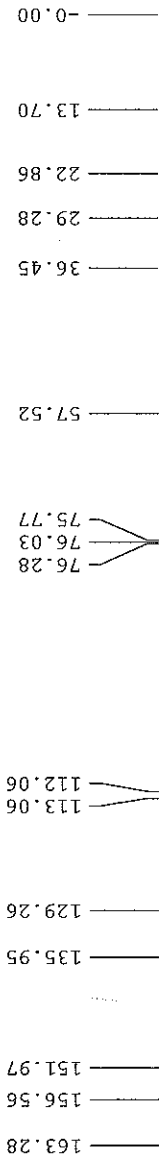
142





atl522

mCARBONnight CDCI3 /opt/topspin ejt 14



Current Data Parameters
NAME 2006-12-10-ejt-14
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061210
Time 23.41
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 512
DW 16.800 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 7.30 usec
PL1 -4.50 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 17.76 dB
PL13 120.00 dB
PL2 -0.30 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7579174 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

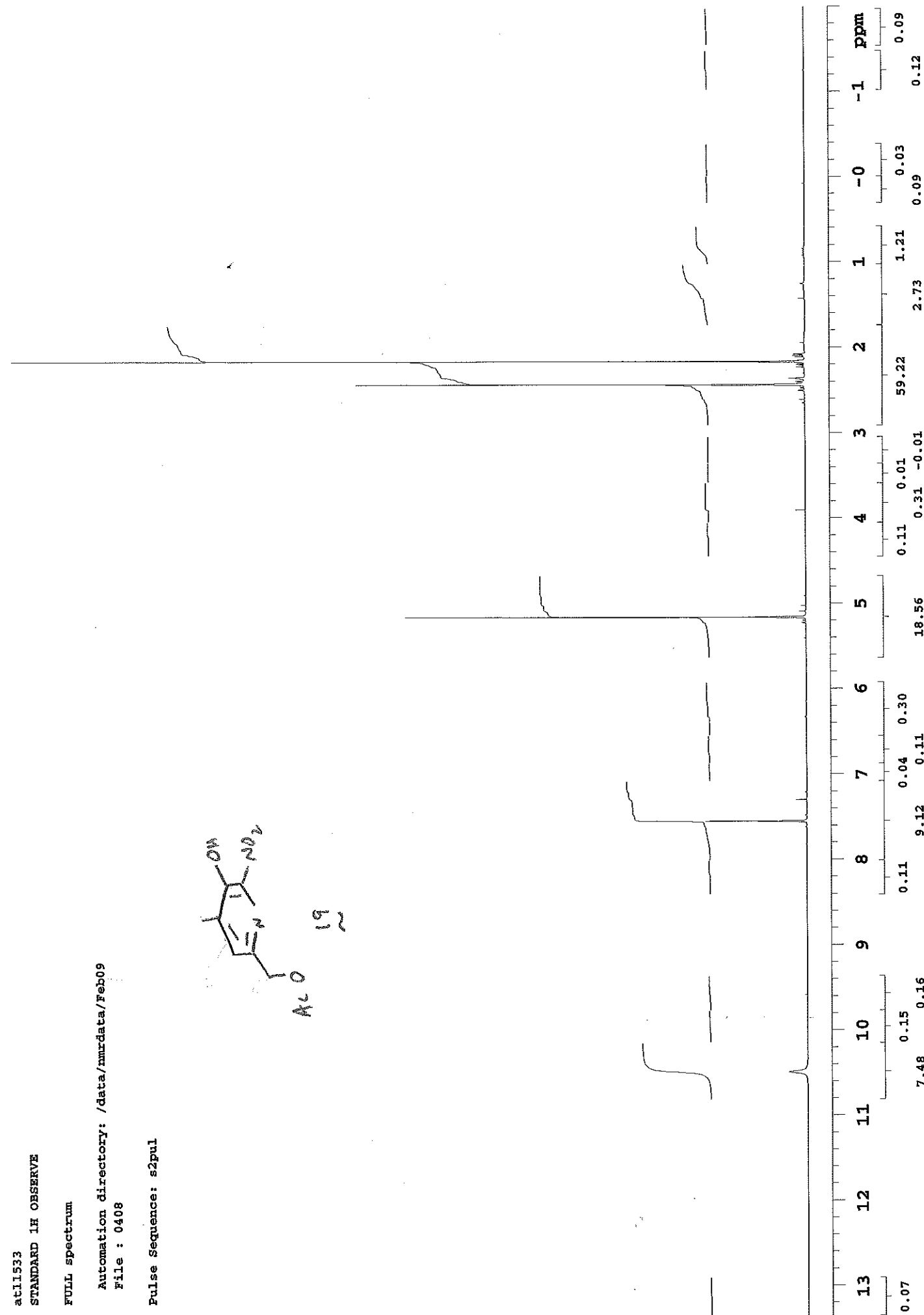
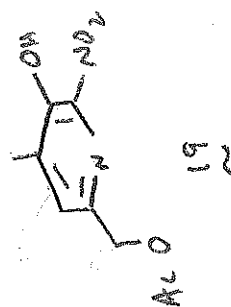
200 180 160 140 120 100 80 60 40 20 0 ppm

atl1533
STANDARD 1H OBSERVE

FULL spectrum

Automation directory: /data/nmrdata/feb09
File : 0408

Pulse Sequence: s2pul



at11533

STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/feb09

File : 0410

Pulse Sequence: s2pul

Solvent: cdcl3

DATE Sun 11 Feb 2007 22:21:29 GMT

Sample #4

File: 0410

INOVA-300 "var300b"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.815 sec

Width 18850.1 Hz

256 repetitions

OBSERVE C13, 75.3962883 MHz

DECOUPLE H1, 299.8473786 MHz

Power 37 dB

continuously on

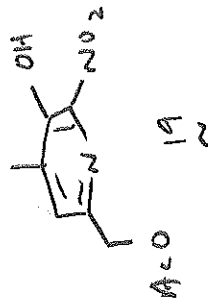
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 12 min, 2 sec



131.614

65.851

16.249

21.079

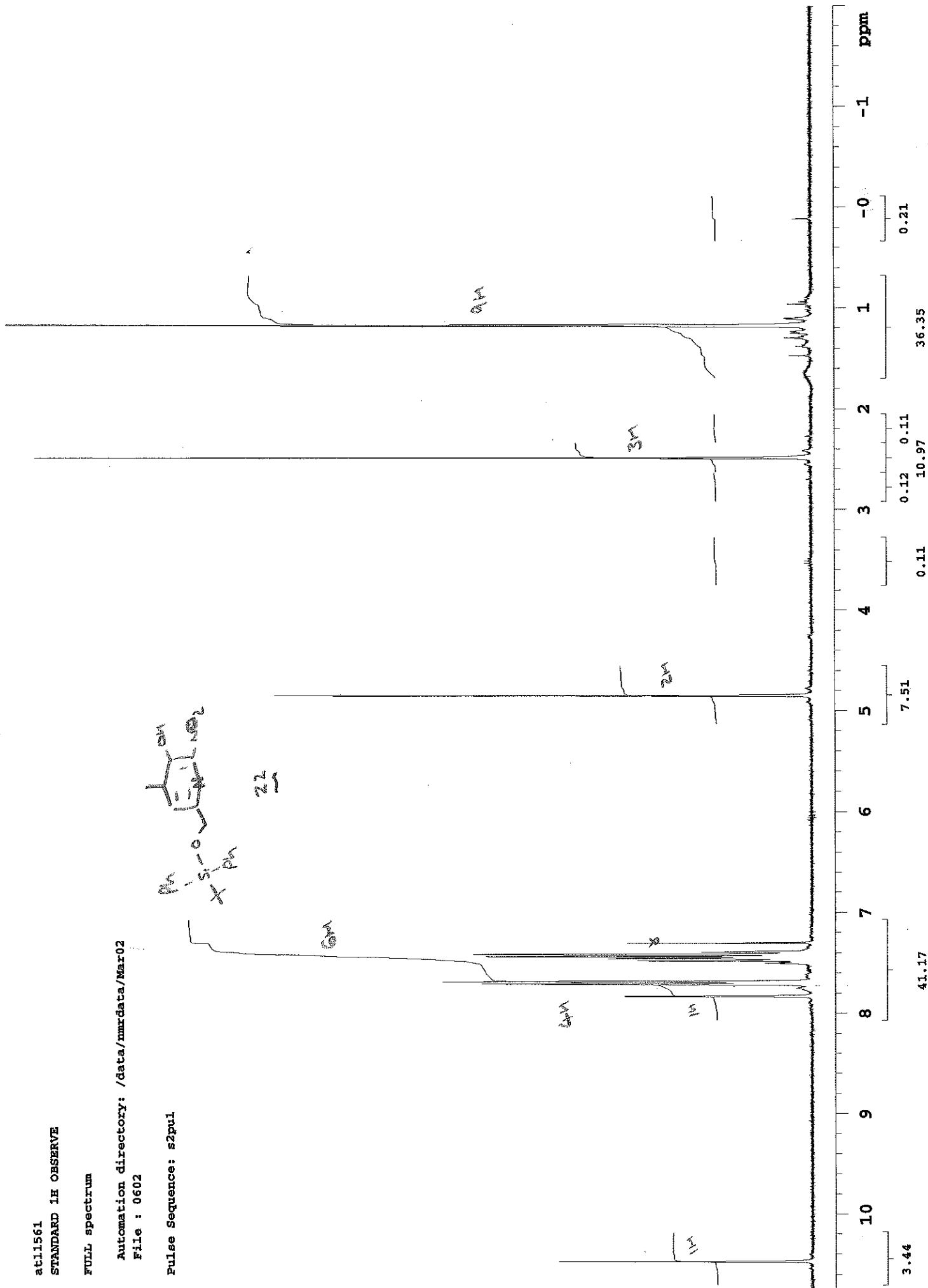
77.781
77.357
76.930

149.601
146.286
142.574
141.227

170.794

220 200 180 160 140 120 100 80 60 40 20 0 ppm

atl1561
 STANDARD 1H OBSERVE
 FULL spectrum
 Automation directory: /data/nmrdata/Mar02
 File : 0602
 Pulse Sequence: s2pul

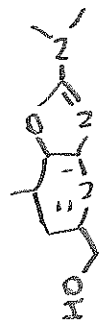


atl1567
STANDARD 1H OBSERVE

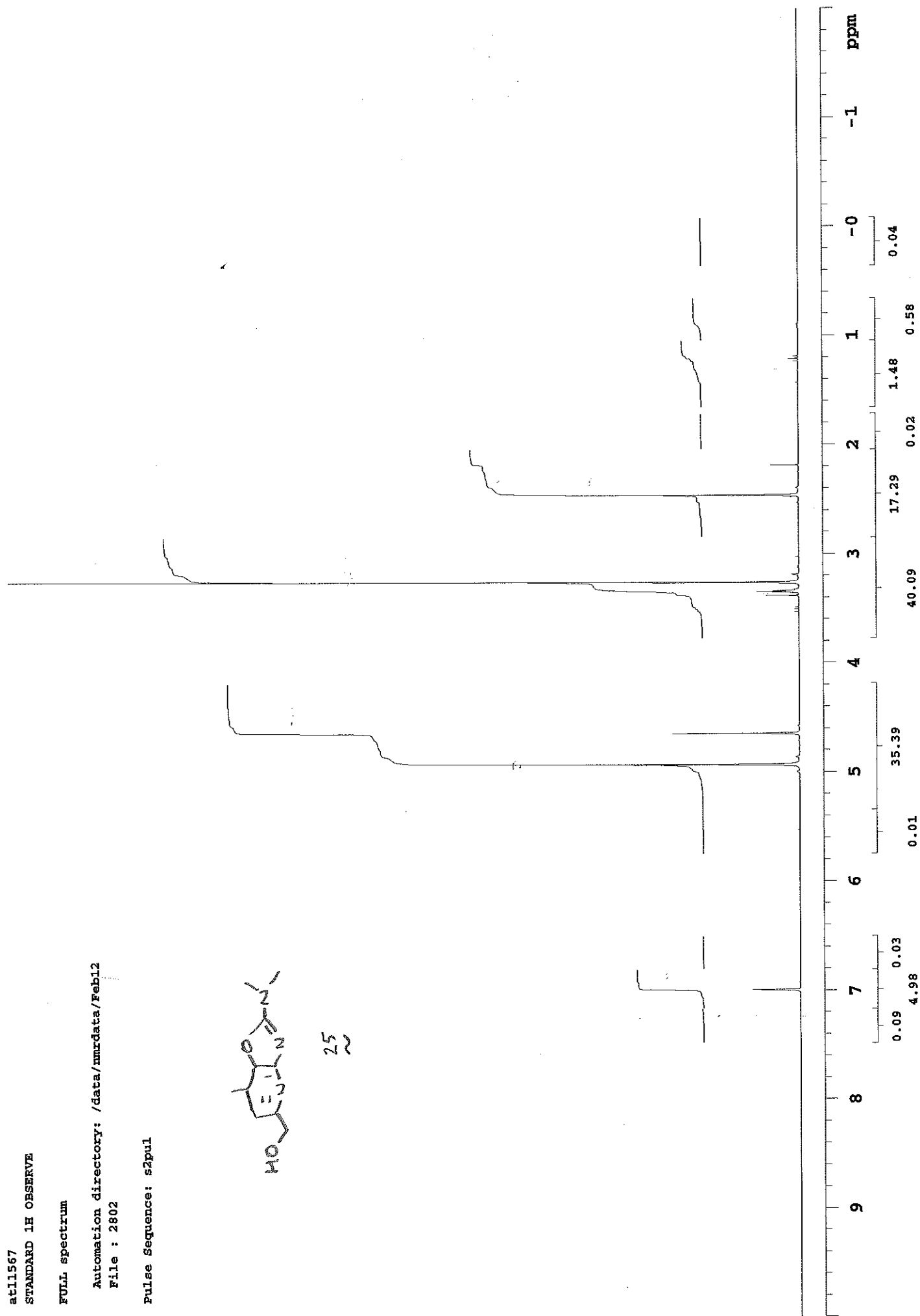
FULL spectrum

Automation directory: /data/nmrdata/Feb12
File : 2802

Pulse Sequence: s2pul



25
~



atl1567

13C OBSERVE

pad=2 run with findz0 before acquisition

Automation directory: /data/nmrdata/Feb23

File : 1304

Pulse Sequence: s2pul

Solvent: CD3OD

DATE Mon 26 Feb 2007 02:06:29 GMT

Sample #13

File: 1304

INOVA-300 "var300b"

Relax. delay 5.000 sec

Pulse 45.0 degrees

Acq. time 1.815 sec

Width 18850.1 Hz

2048 repetitions

OBSERVE C13, 75.3965854 MHz

DECOUPLE H1, 299.8485600 MHz

Power 37 dB

continuously on

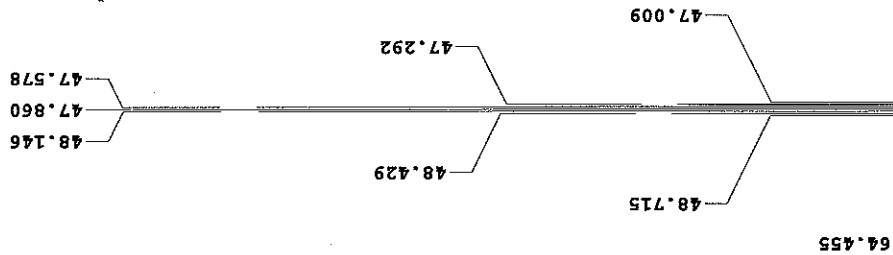
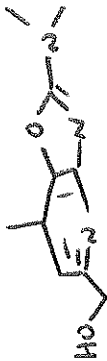
WALTZ-16 modulated

DATA PROCESSING

line broadening 1.0 Hz

FT size 131072

Total time 3 hr, 52 min, 55 sec

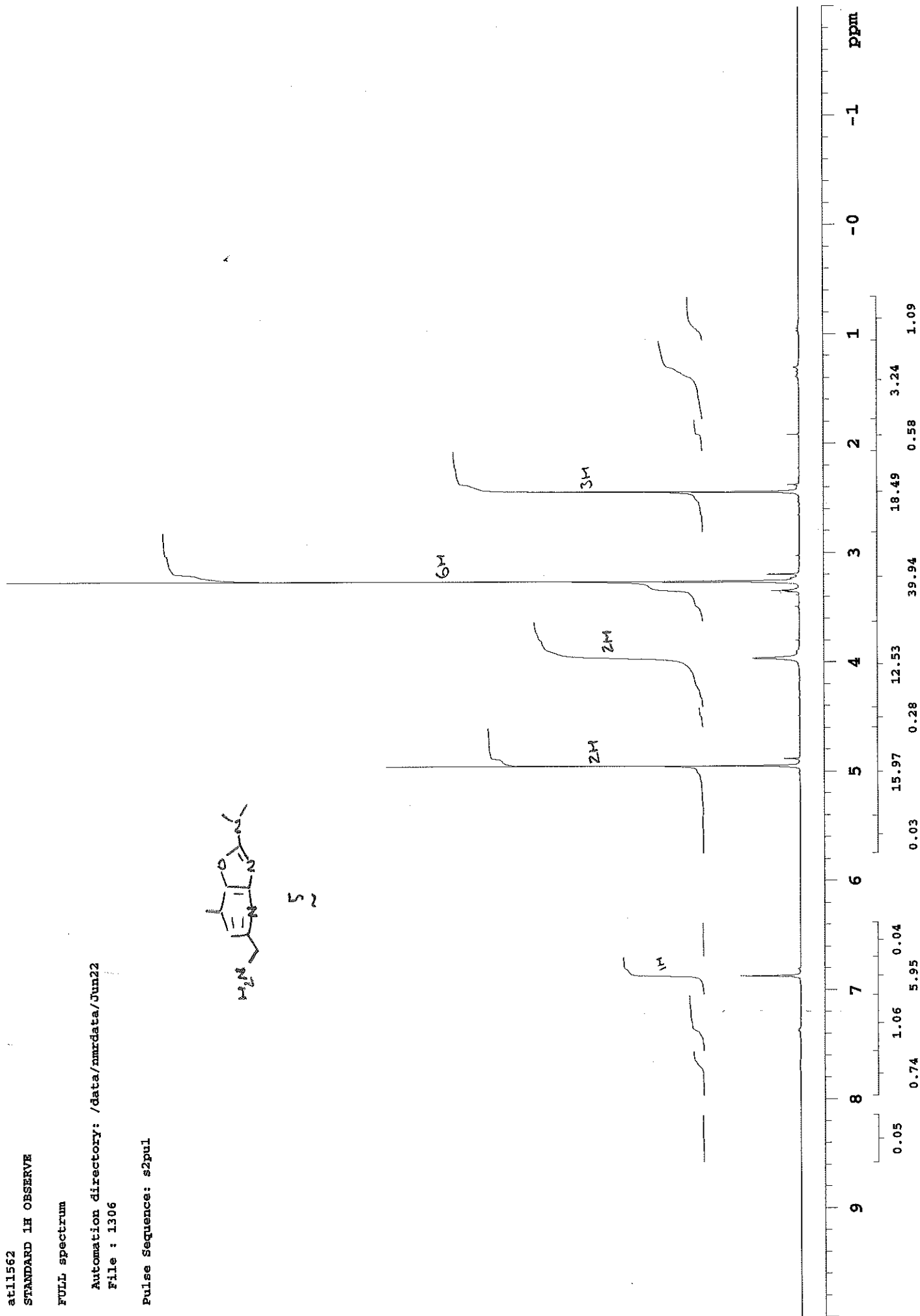


220 200 180 160 140 120 100 80 60 40 20 0 ppm

at11562
 STANDARD 1H OBSERVE
 FULL spectrum
 Automation directory: /data/nmrdata/Jun22
 File : 1306
 Pulse Sequence: s2pul



5



atl1562

STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Jun22
File : 1309

Pulse Sequence: s2pul

Solvent: CD3OD

DATE Jun 24 2007

Sample #13

File: 1309

INOVA-300 "athos"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.815 sec

Width 18850.1 Hz

256 repetitions

OBSERVE C13, 75.3965854 MHz

DECOUPLE H1, 299.8485600 MHz

Power 37 dB

continuously on

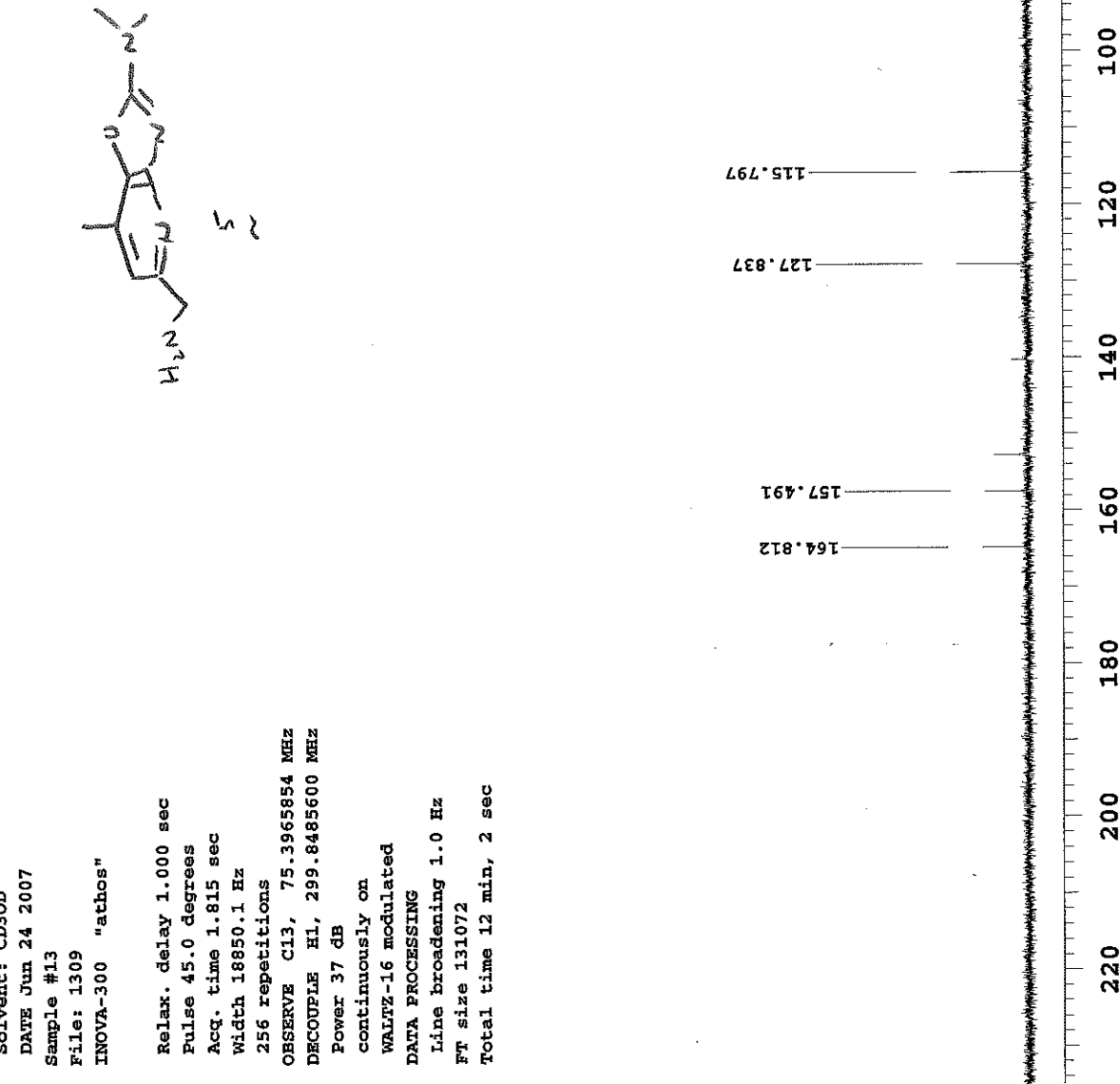
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 12 min, 2 sec



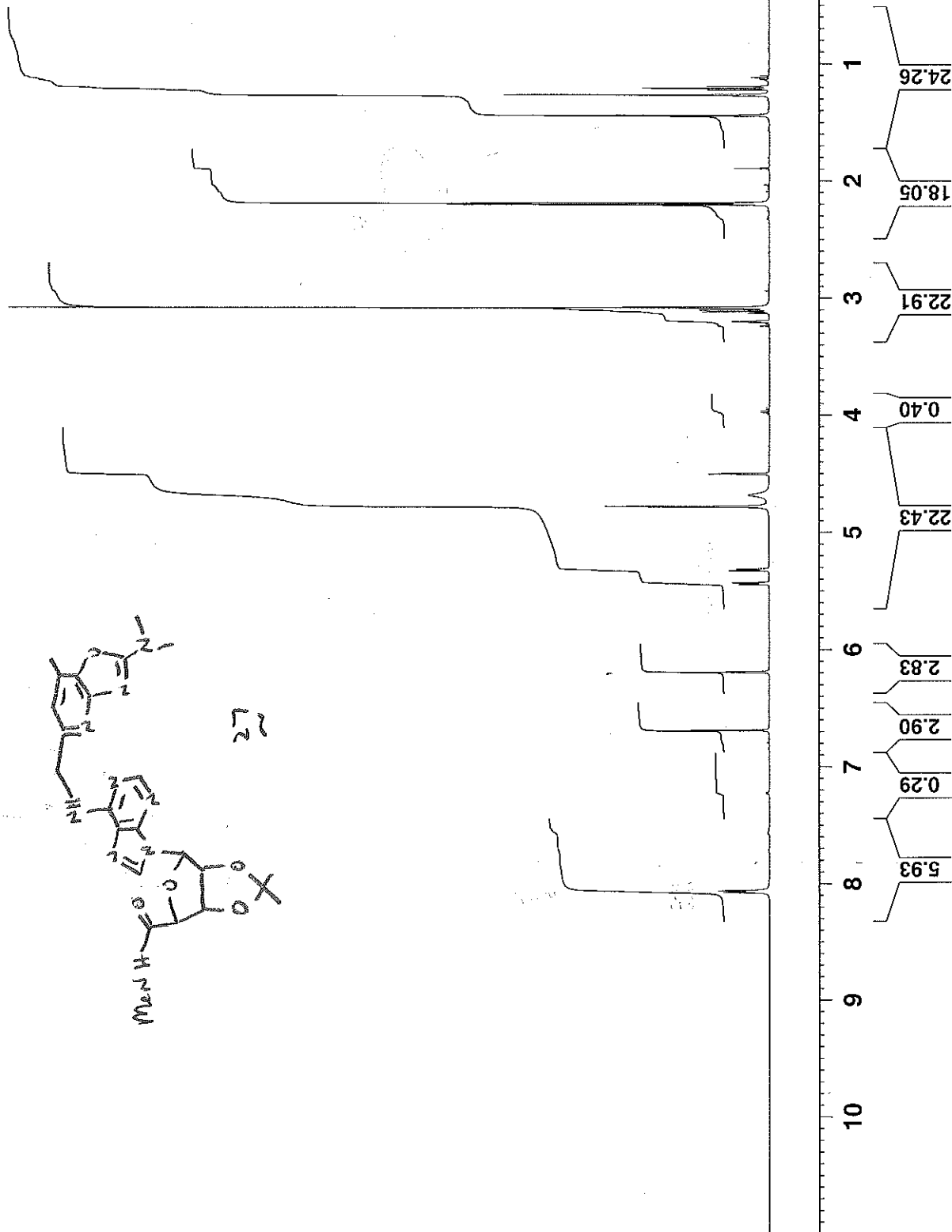


Current Data Parameters
NAME 2007-07-04-ejt-15
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070705
Time 1.41
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zg30b
TD 65536
SOLVENT MeOD
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 90.5
DW 48.400 usec
DE 13.52 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 37.76 usec
PL1 3.25 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300639 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



atl1564

13C OBSERVE

pad=2 run with findz0 before acquisition

Automation directory: /data/nmrdata/Jul13

File : 4101

Pulse Sequence: s2pul

Solvent: CD3OD

DATE Jul 14 2007

Sample #41

File: 4101

INOVA-300 "var300b"

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 1.815 sec

Width 18850.1 Hz

2048 repetitions

OBSERVE C13, 75.3965854 MHz

DECOUPLE H1, 299.8485600 MHz

Power 37 dB

continuously on

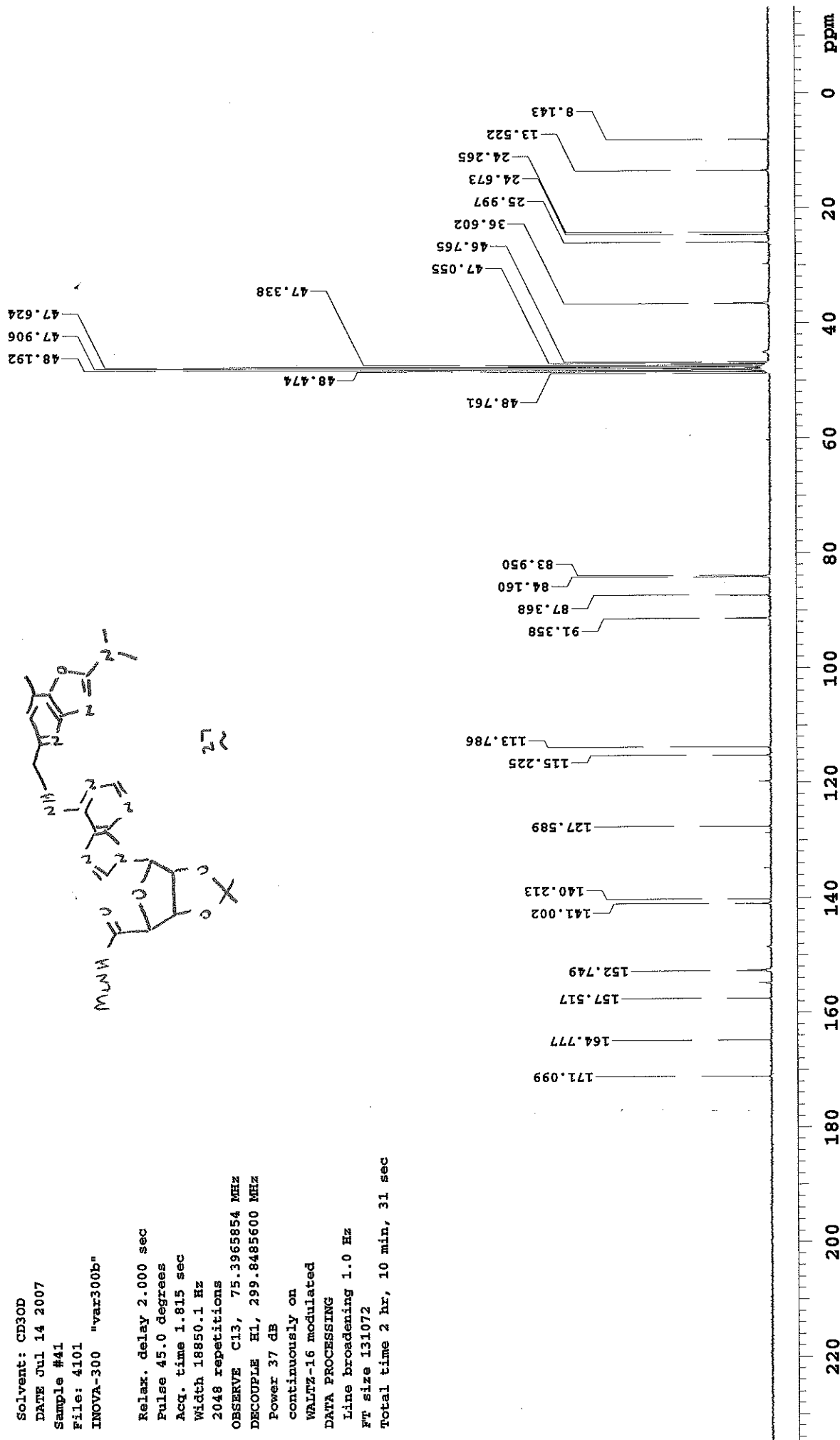
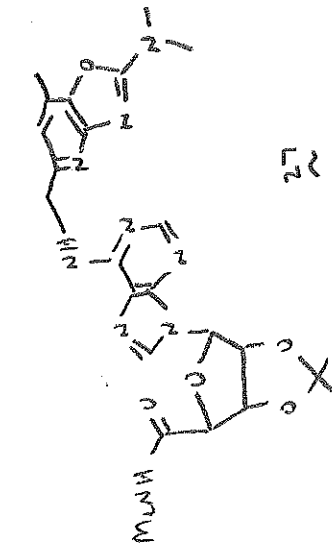
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

Ft size 131072

Total time 2 hr, 10 min, 31 sec



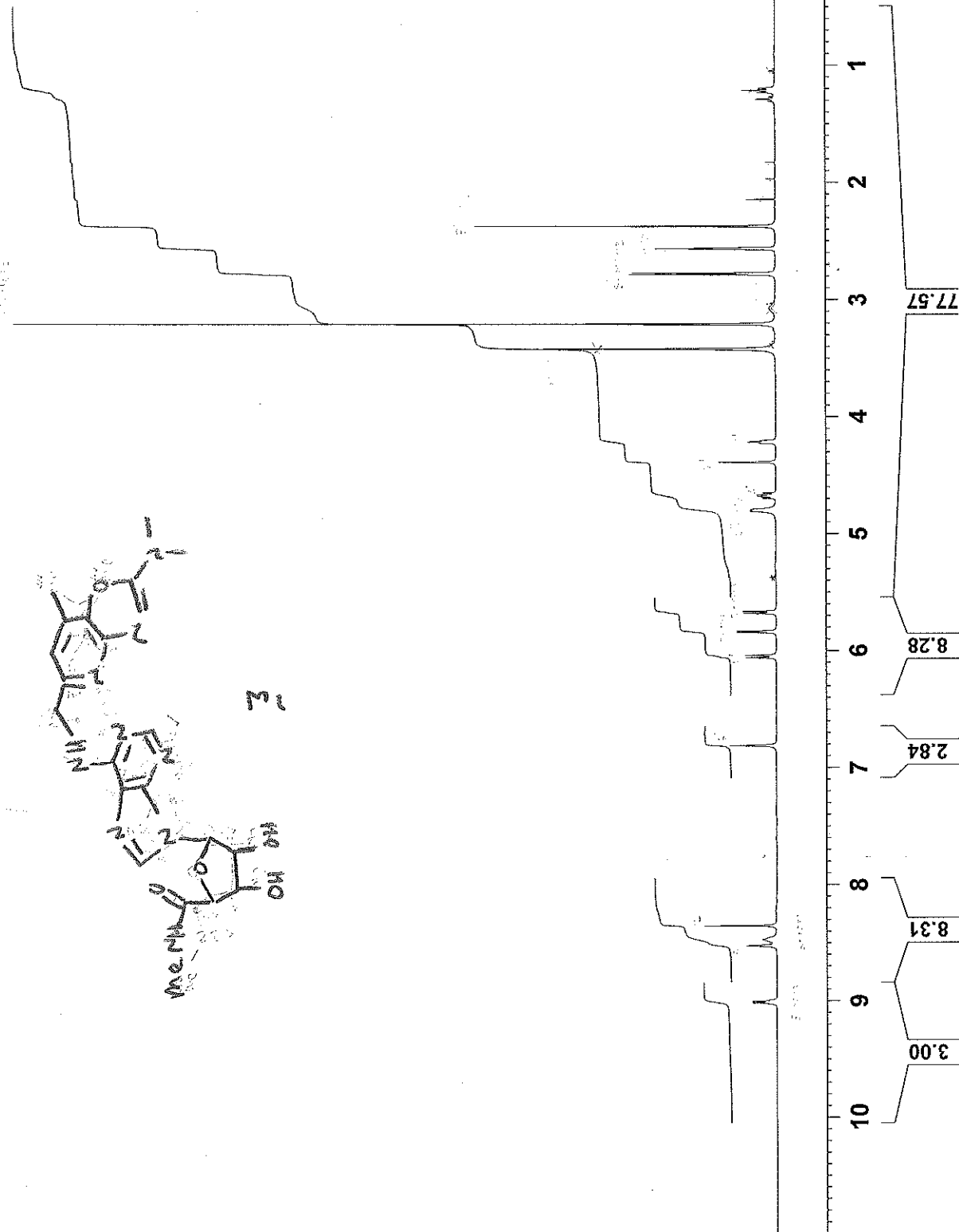
atl1a3
mPROTONnight DMSO {e:\bruk400data\2011\Sep} ejt 32

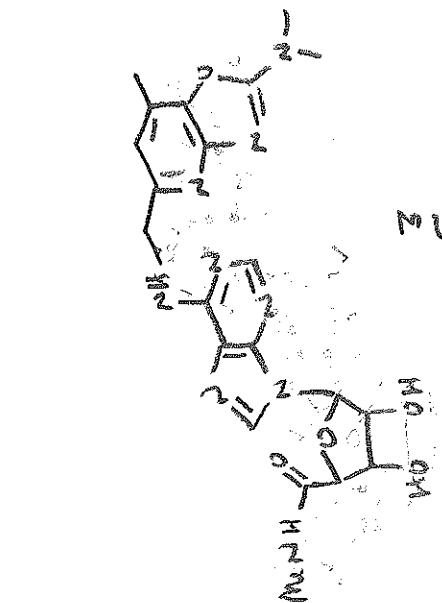
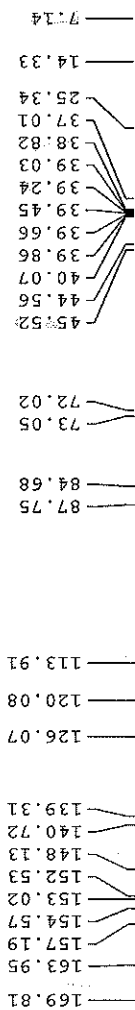


2011-09-23-ejt-32

NAME
EXPNO 11
PROCNO 1
Date_ 20110924
Time_ 12.00
INSTRUM AV400
PROBHD 5 mm PABEO BB-
PULPROG zg30b
TD 65536
SOLVENT DMSO
NS 256
DS 0
SWH 8264.463 Hz
FIDRES 0.126106 Hz
AQ 3.9649780 sec
RG 322
DW 60.500 usec
DE 9.40 usec
TE 292.3 K
D1 1.00000000 sec
D11
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -3.60 dB
PL1W 17.83863831 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1299778 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



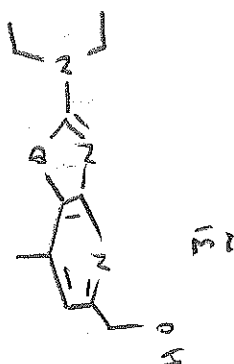


NAME 2011-09-23-ejt-32
EXPNO 10
PROCNO 1
Date_ 20110924
Time_ 11.38
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 3072
DS 2
SWH 30241.936 Hz
FIDRES 0.461455 Hz
AQ 1.0835786 sec
RG 512
DW 16.533 usec
DE 7.83 usec
TE 293.1 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 0.00 dB
PL1W 34.9152217 W
SFO1 100.6241042 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.60 dB
PL12 15.31 dB
PL13 18.00 dB
PL1W 17.83863831 W
PL12W 0.2292761 W
PL13W 0.12341322 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

240 220 200 180 160 140 120 100 80 40 20 ppm

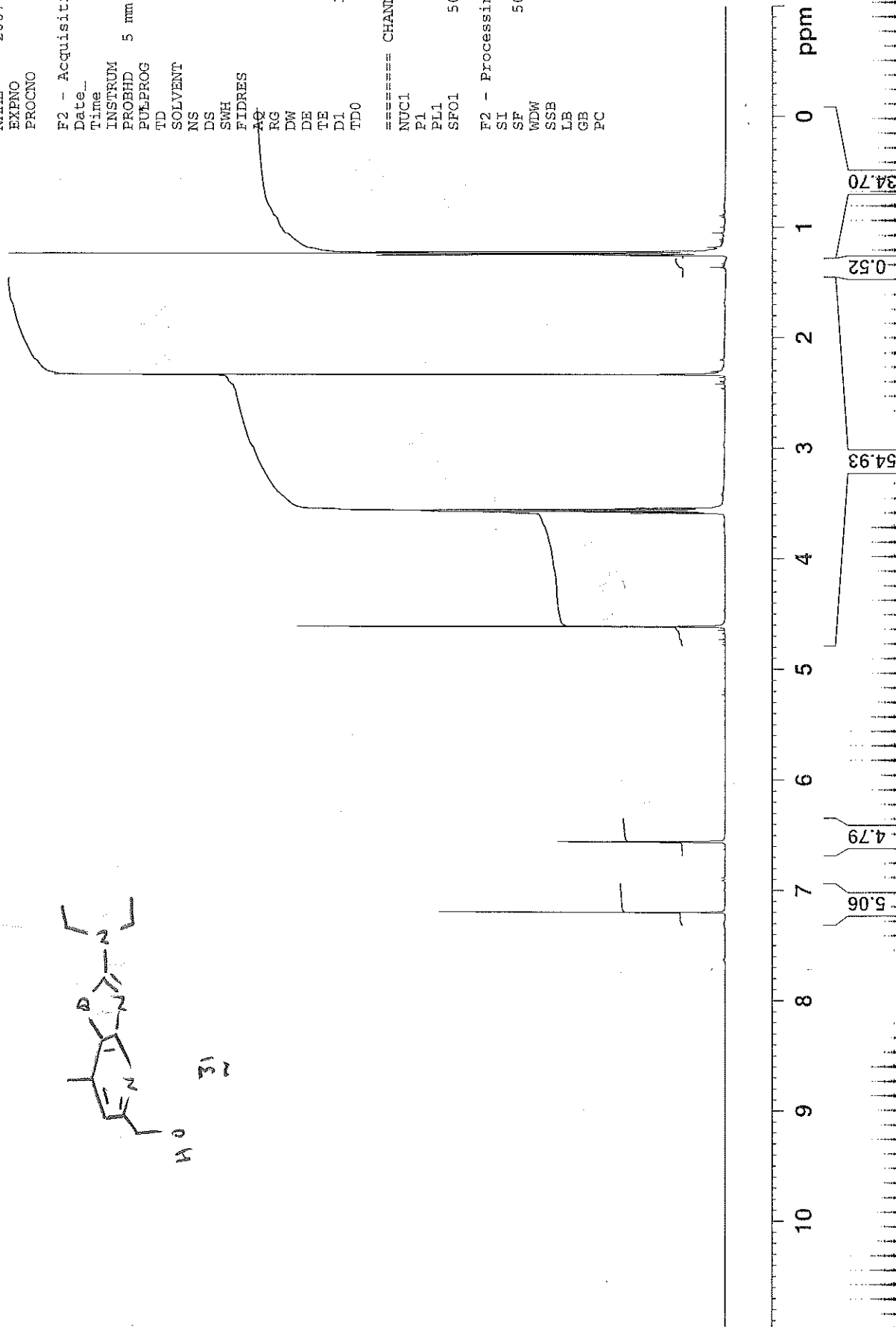


Current Data Parameters
 NAME 2007-08-10-ejt-3
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070812
 Time 21.11
 INSTRUM spect
 PROBD 5 mm TXI 1H/D-
 PULPROG zg30b
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.171923 sec
 RG 228
 DW 48.400 usec
 DE 13.76 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.80 usec
 PL1 3.25 dB
 SFO1 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300393 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



ss095

STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Mar29

File: 1205

Pulse Sequence: s2pul

Solvent: cdcl3

DATE Fri 30 Mar 2007 01:09:08 BST

Sample #12

File: 1205

INOVA-300 "var300b"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.815 sec

Width 18850.1 Hz

2048 repetitions

OBSERVE C13, 75.3962883 MHz

DECOUPLE H1, 299.8473786 MHz

Power 37 dB

continuously on

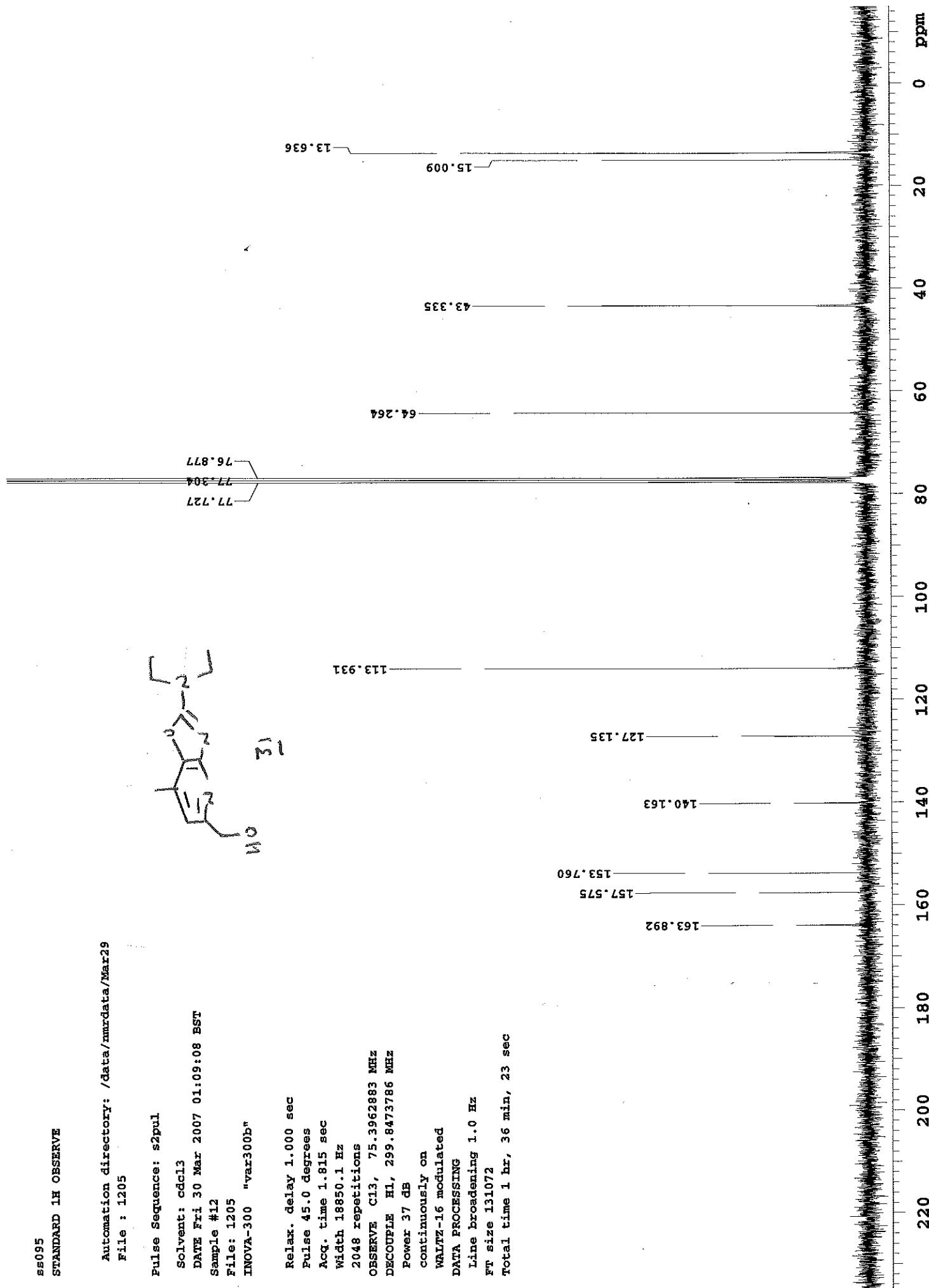
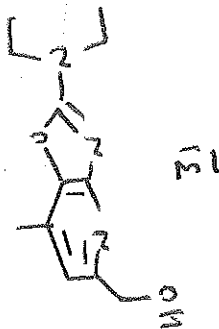
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 1 hr, 36 min, 23 sec



sneel75
STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/aug17
File : 0601

Pulse Sequence: s2pul

Solvent: cdcl3

DATE Aug 17 2007

Sample #6

File: 0601

INOVA-300 "var300b"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 3.744 sec

Width 4797.6 Hz

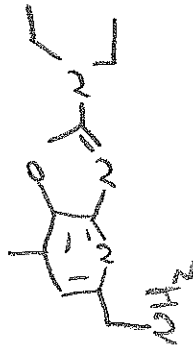
16 repetitions

OBSERVE H1, 299.8458792 MHz

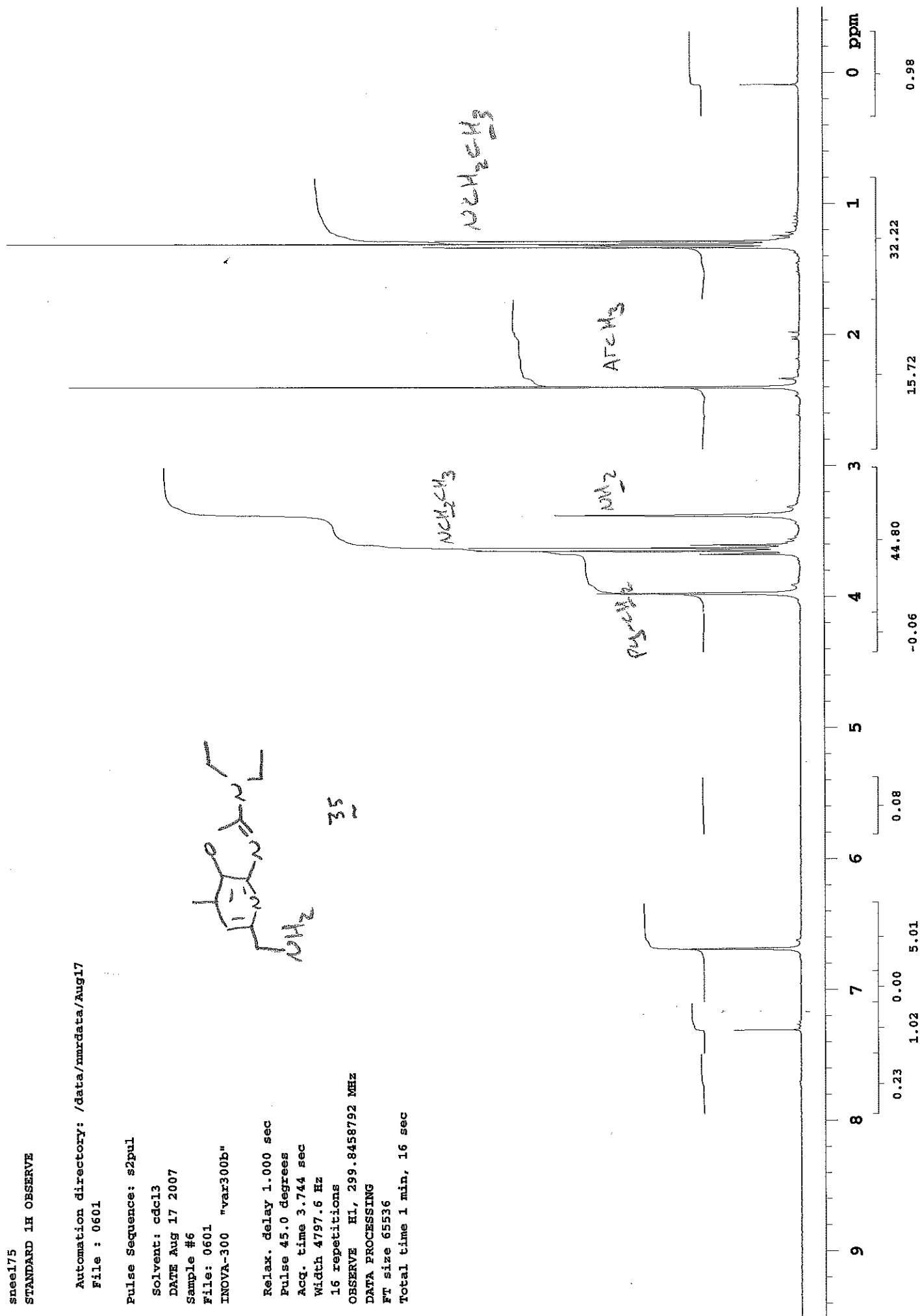
DATA PROCESSING

FT size 65536

Total time 1 min, 16 sec



35



sneel75
STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Aug17
File : 1103

Pulse Sequence: s2pul

Solvent: cdcl3

DATE Aug 17 2007

Sample #11

File: 1103

INOVA-300 "var300b"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.815 sec

Width 18850.1 Hz

256 repetitions

OBSERVE C13, 75.3962883 MHz

DECOUPLE H1, 299.8473786 MHz

Power 37 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

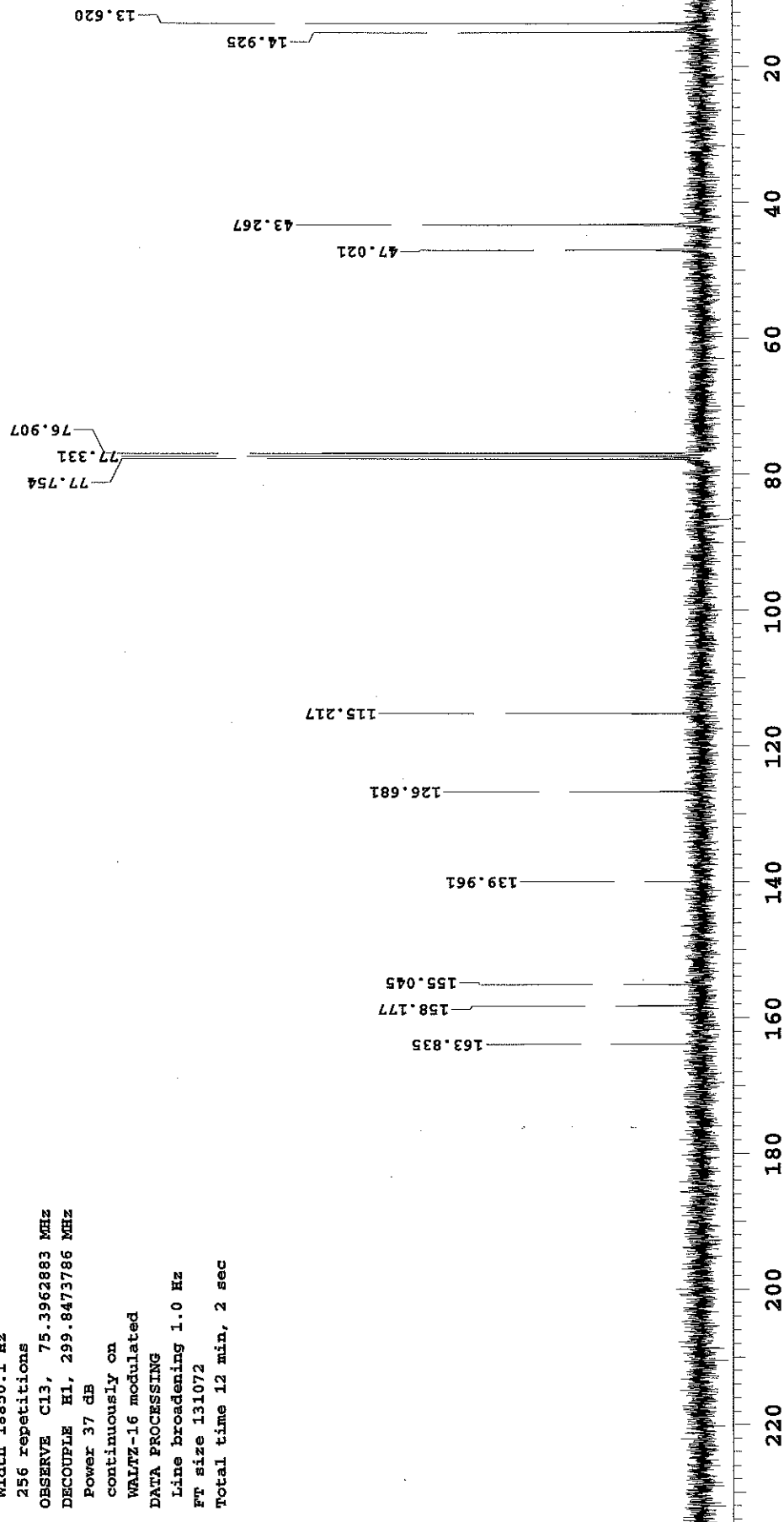
Line broadening 1.0 Hz

FT size 131072

Total time 12 min, 2 sec



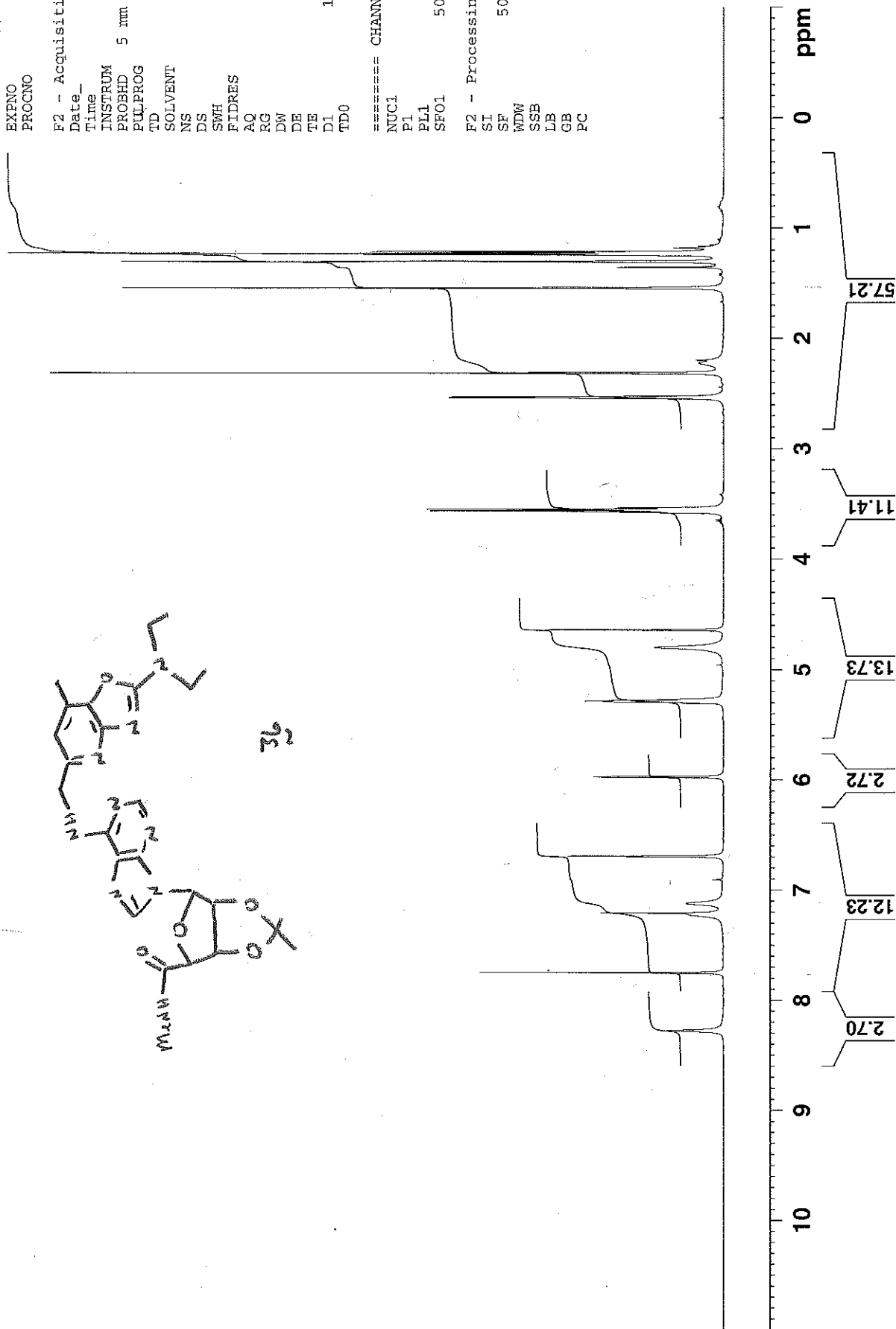
35





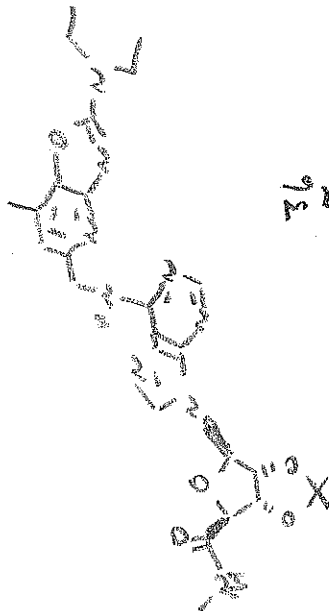
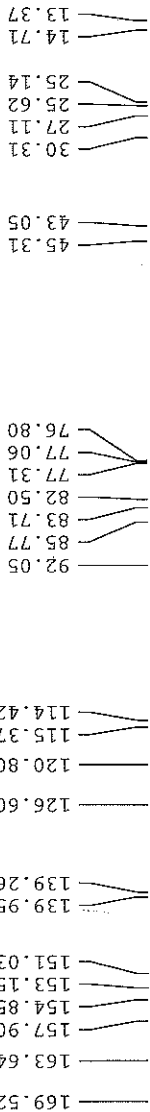
Chemical structure of a complex organic molecule, possibly a steroid or a related compound, with numbered atoms (1-10) indicating specific sites of interest. The structure includes a benzene ring, a five-membered ring, and a six-membered ring, with various functional groups and substituents. A handwritten number '52' is present in the upper right corner.

F2 - Processing parameters	
SI	32768
SF	500.1300372 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



snee176

mCARBONight CDCl3 /opt/topspin ejt 36



Current Data Parameters
NAME 2007-08-23-ejt-36
EXPNO 11
PROCNO 1

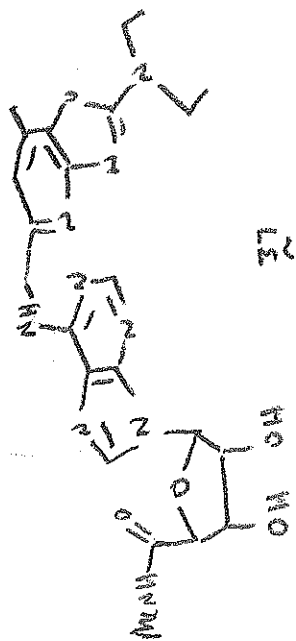
F2 - Acquisition Parameters
Date_ 20070824
Time 9.16
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 512
DW 16.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 11.50 usec
PL1 -4.20 dB
SFO1 125.7703643 MHz

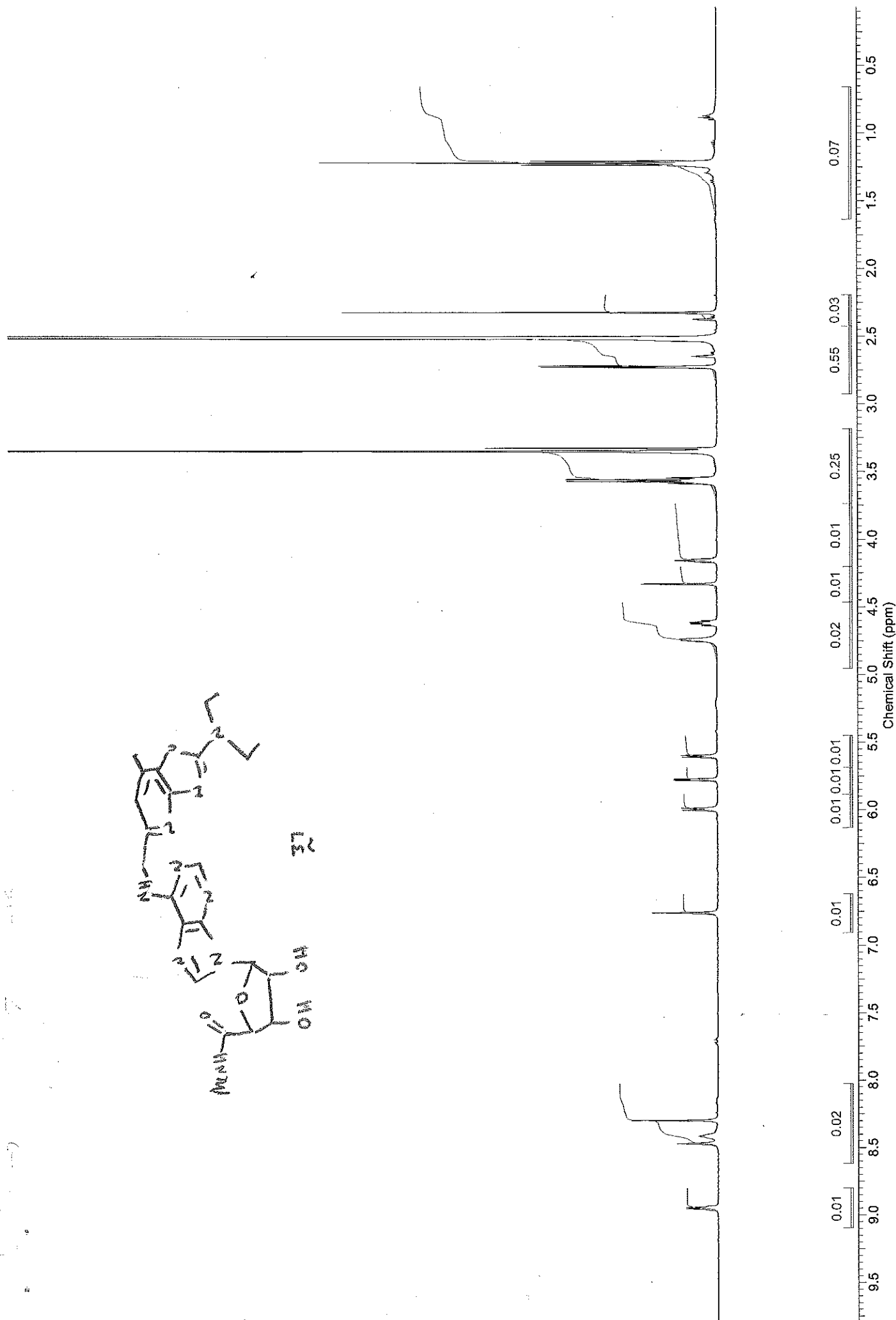
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 20.00 dB
PL13 23.00 dB
PL2 3.25 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

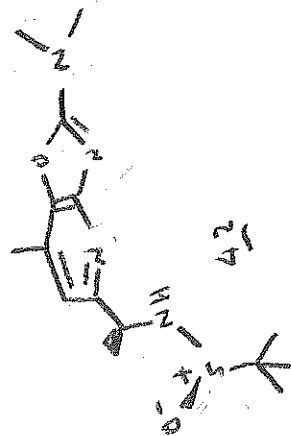
200 180 160 140 120 100 80 60 40 20 0 ppm



32



snee162
mPROTON CDCl3 /opt/topspin ejt 49

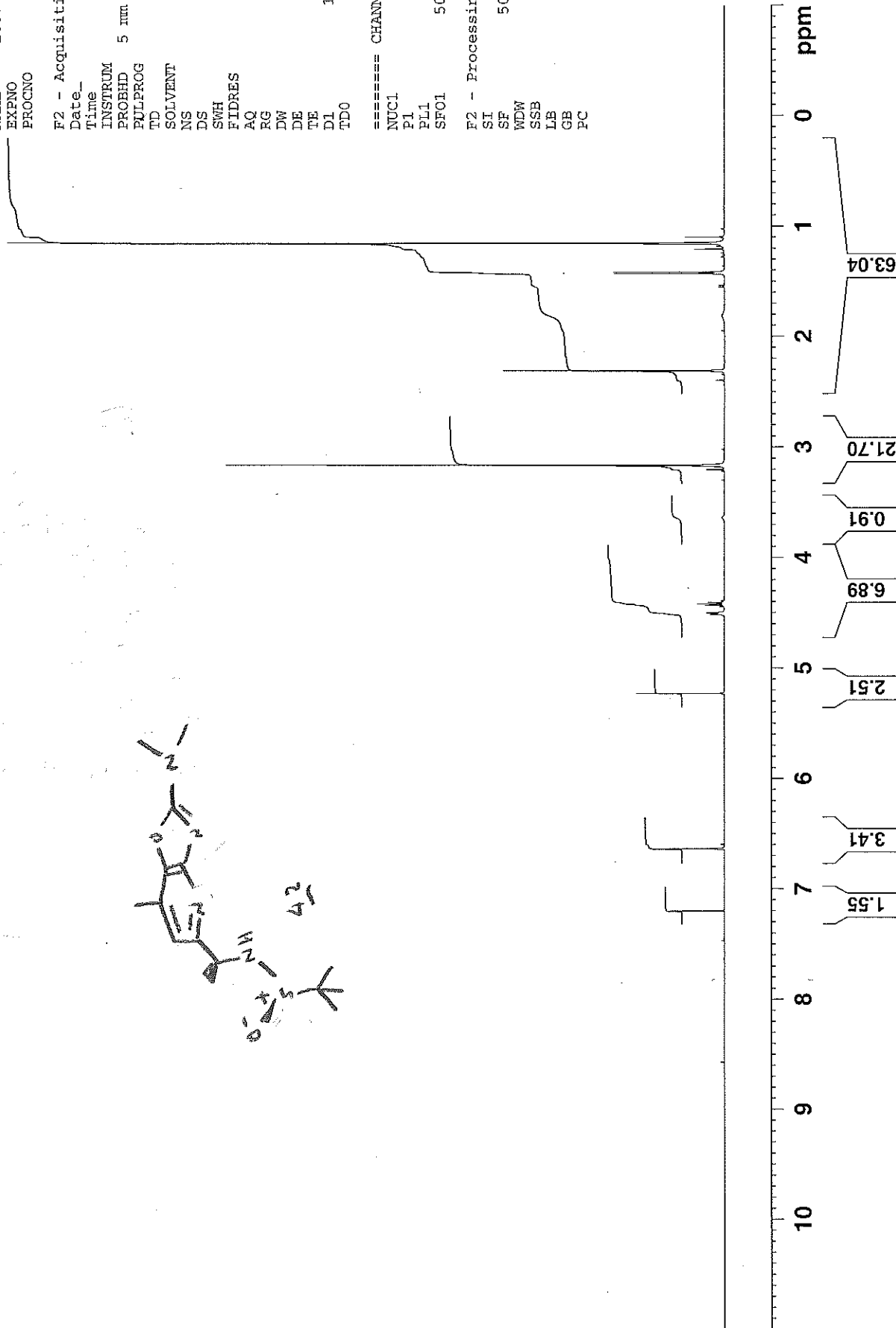


Current Data Parameters
NAME 2007-07-16-ejt-49
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070716
Time 17.38
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zg30b
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.171923 sec
RG 90.5
DW 48.400 usec
DE 13.76 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 7.80 usec
PL1 3.25 dB
SF01 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300376 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



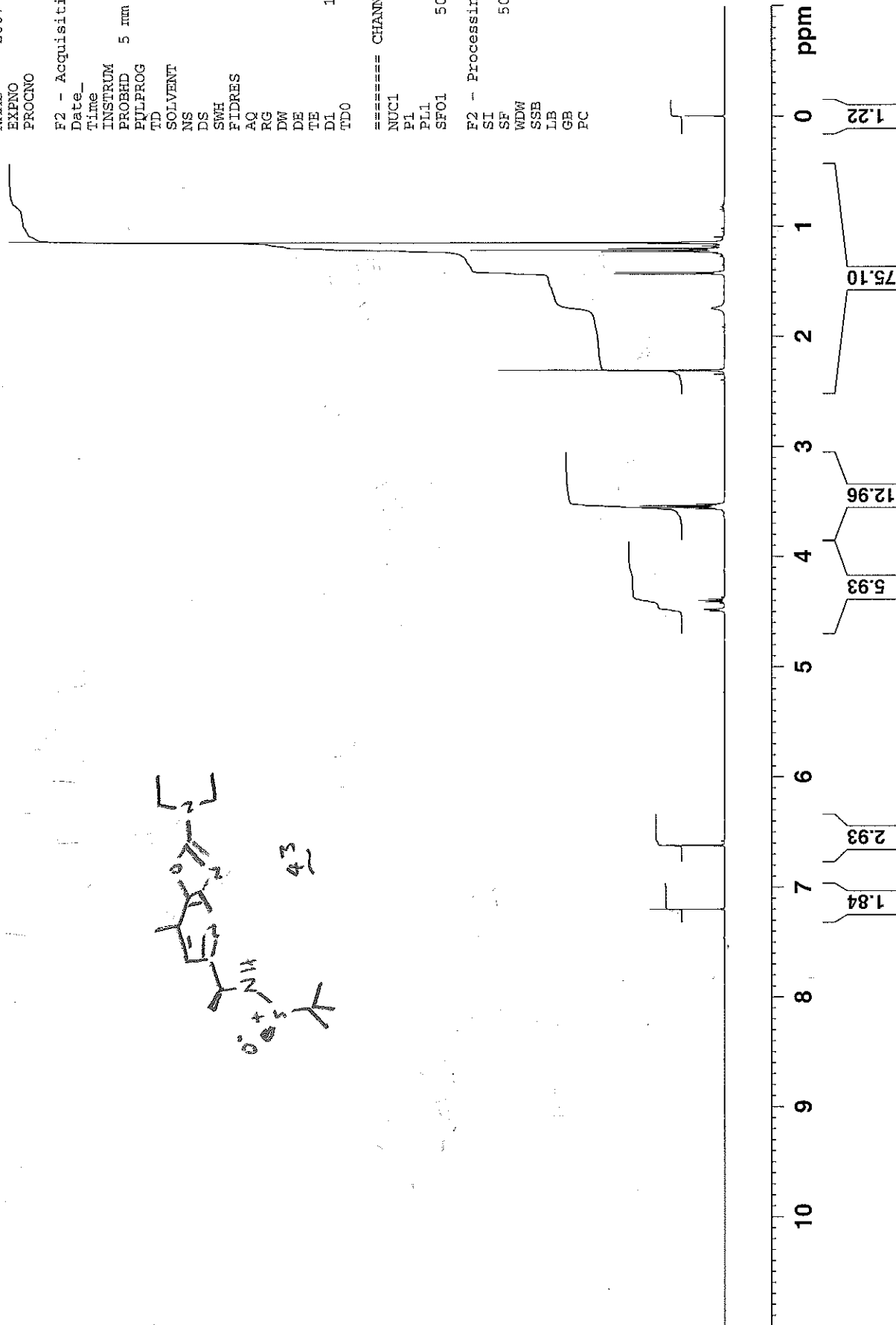
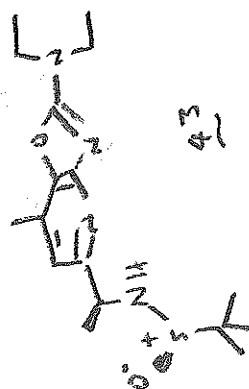


Current Data Parameters
NAME 2007-07-04-ejt-45
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070704
Time 15.40
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zg30b
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 161
DW 48.400 usec
DE 13.76 usec
TE 300.0 K
D1 1.0000000 sec
TD0 1

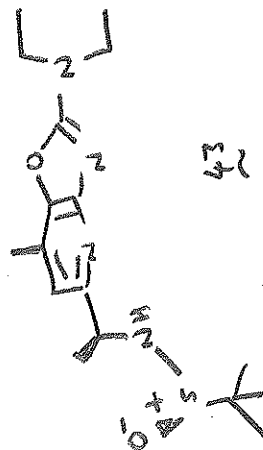
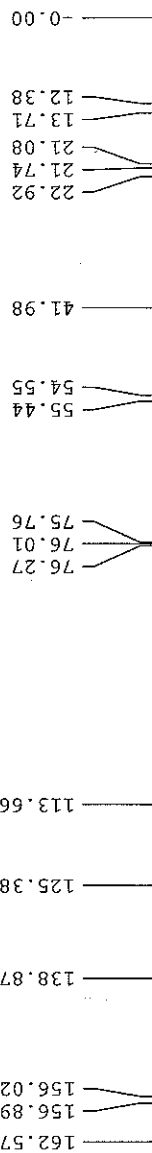
==== CHANNEL f1 =====
NUC1 1H
P1 7.80 usec
PL1 3.25 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300389 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



snee153

mCARBONnight CDCl3 /opt/topspin ejt 45



Current Data Parameters
NAME 2007-07-04-ejt-45
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070704
Time 21.53
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 512
DW 16.800 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 11.50 usec
PL1 -4.20 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 23.47 dB
PL13 120.00 dB
PL2 3.25 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7579183 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

200 180 160 140 120 100 80 60 40 20 0 ppm

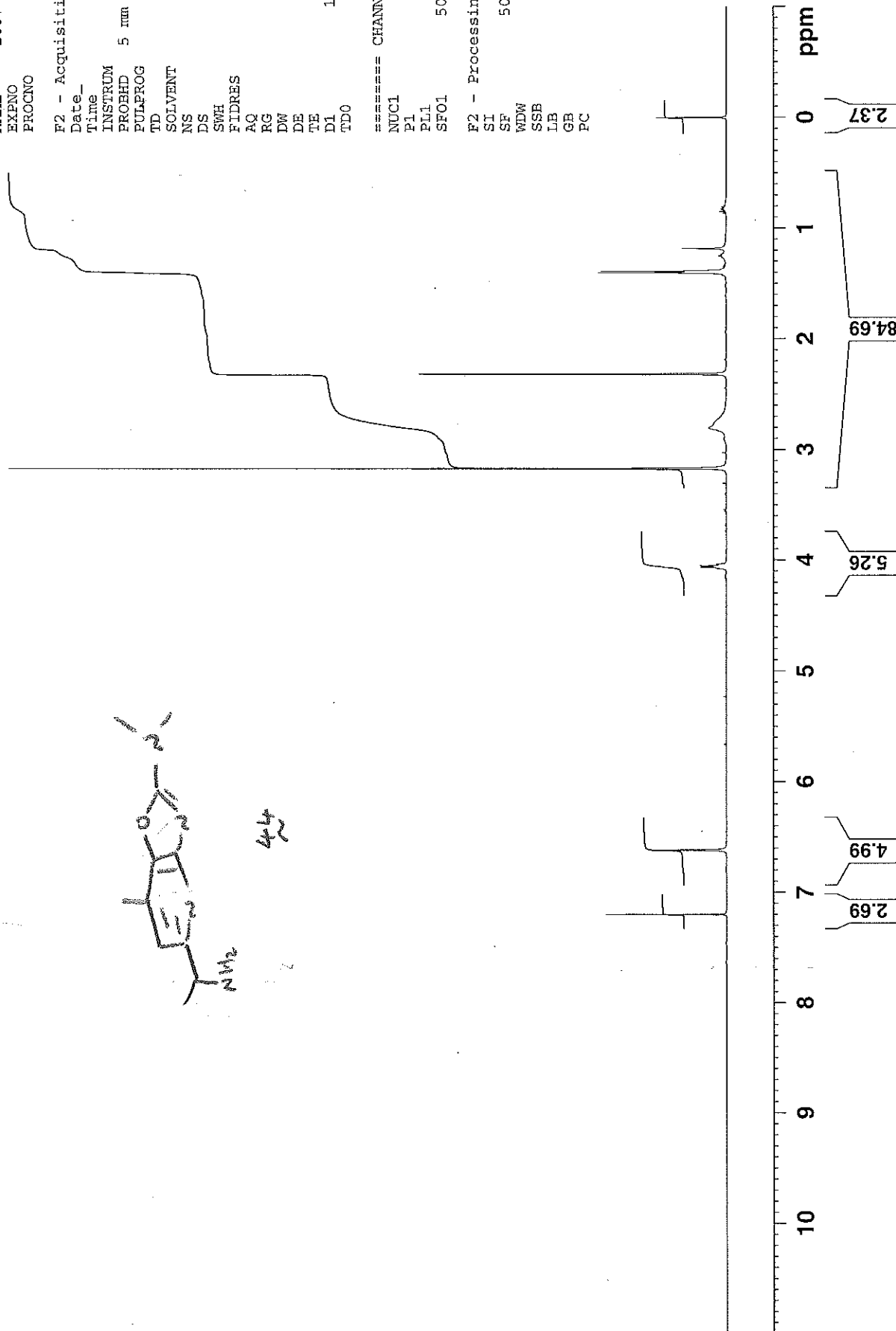
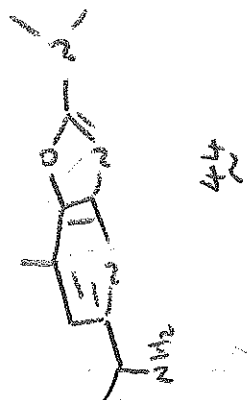


Current Data Parameters
NAME 2007-07-18-ejt-53
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070718
Time 12.40
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zg30b
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 228
DW 48.400 usec
DE 13.76 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 7.80 usec
PL1 3.25 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300387 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



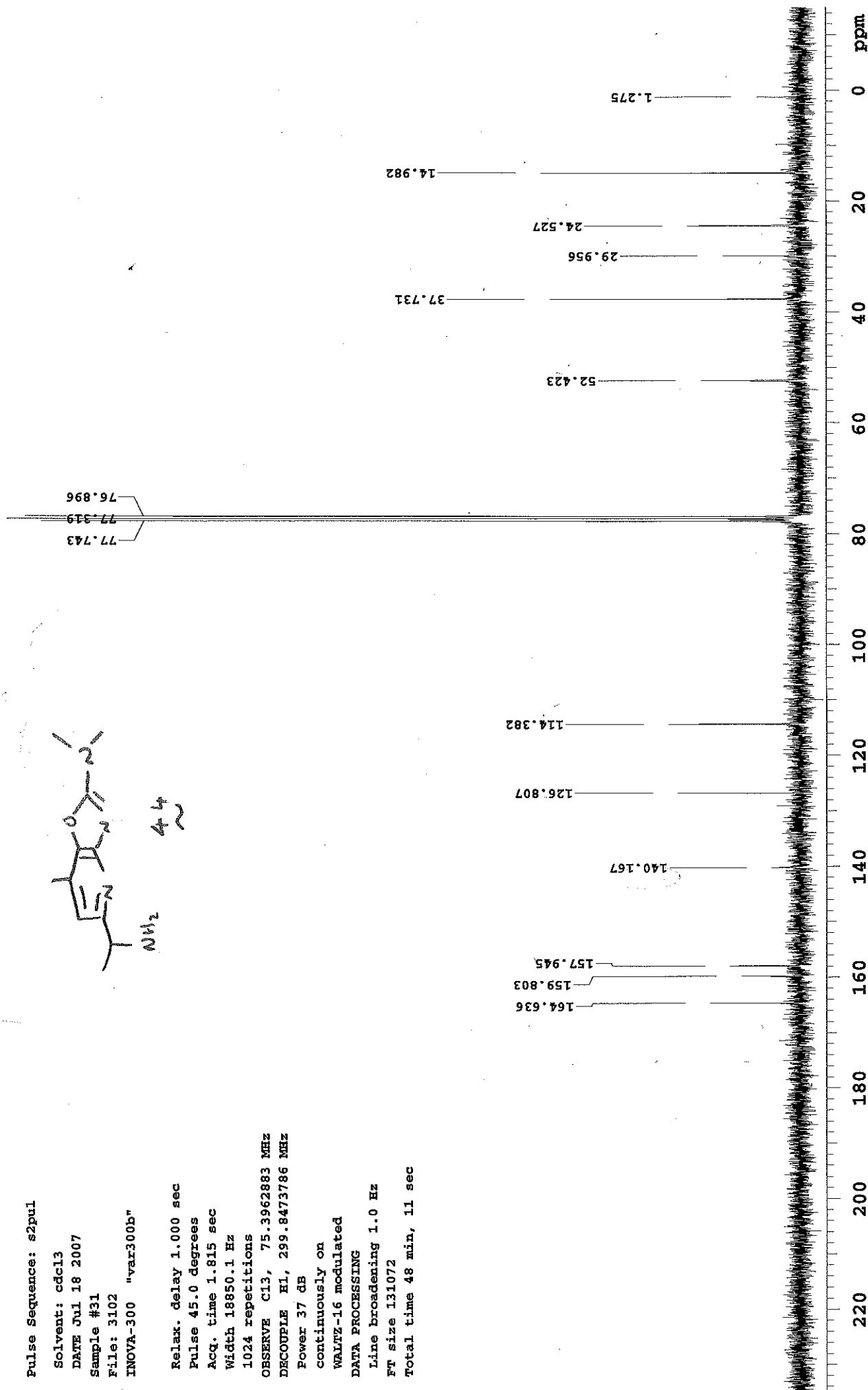
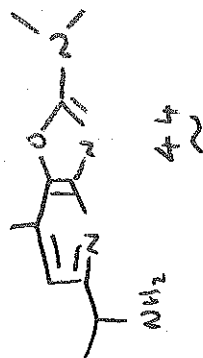
sneel64
STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Jul17
File : 3102

Pulse Sequence: s2pul

Solvent: cdcl3
DATE Jul 18 2007
Sample #31
File: 3102
INOVA-300 "var300b"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.1 Hz
1024 repetitions
OBSERVE C13, 75.3962883 MHz
DECOUPLE H1, 299.8473786 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 131072
Total time 48 min, 11 sec

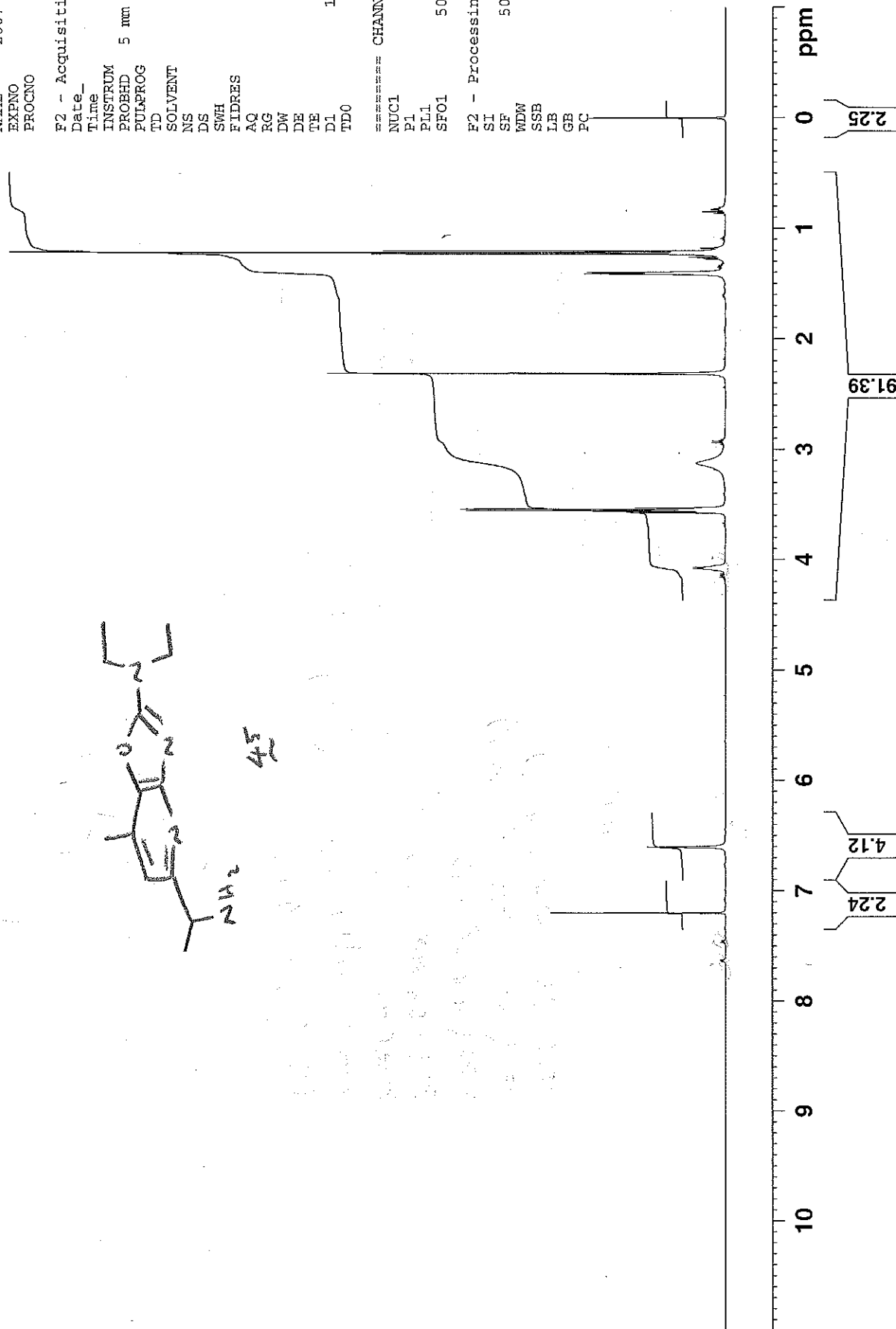




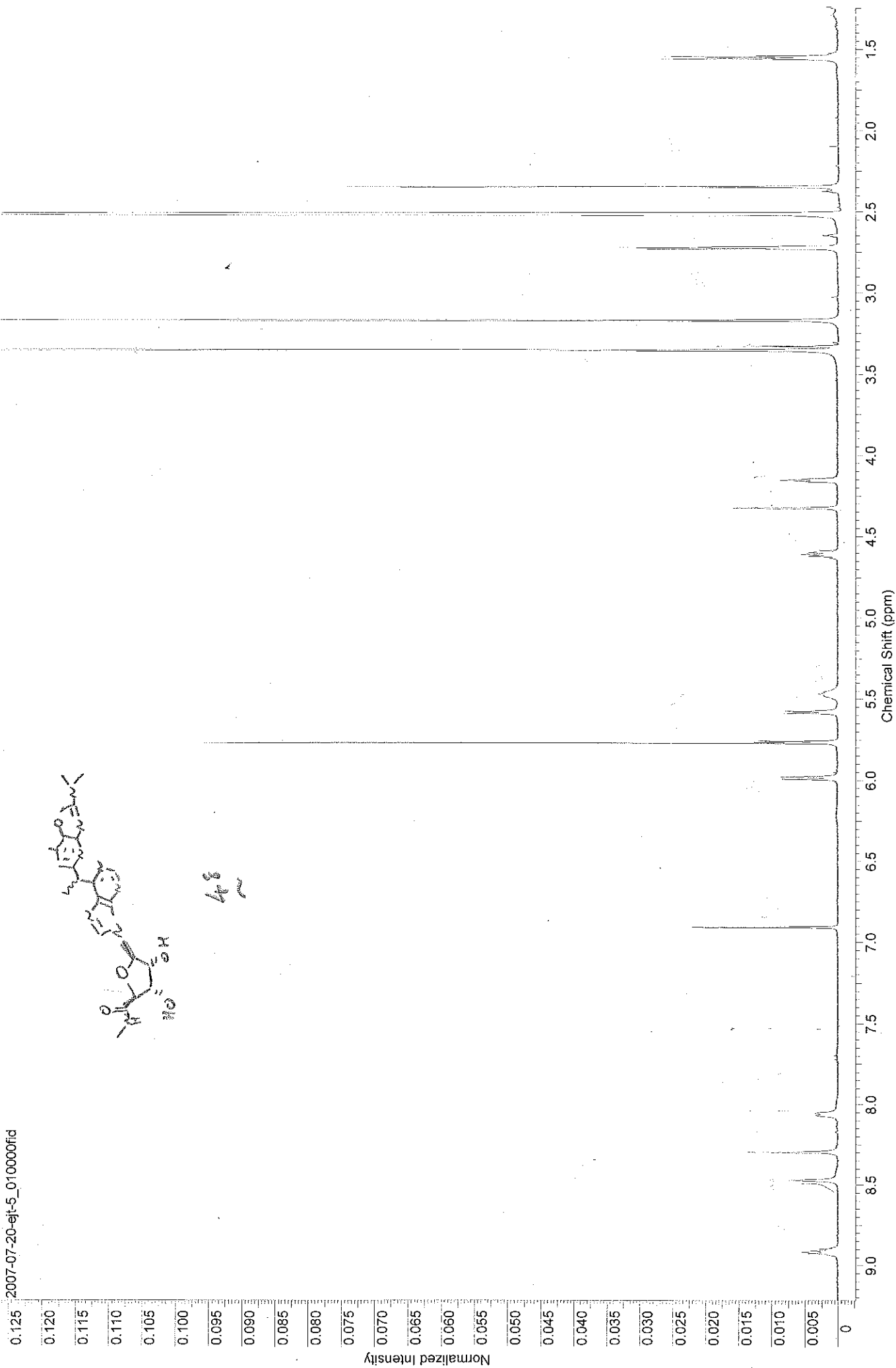
Current Data Parameters
NAME 2007-07-09-djd-48
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070709
Time 15.32
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zg30b
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.171923 sec
RG 181
DW 48.400 usec
DE 13.76 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 7.80 usec
PL1 3.25 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300385 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



2007-07-20-ejt-5_010000fid



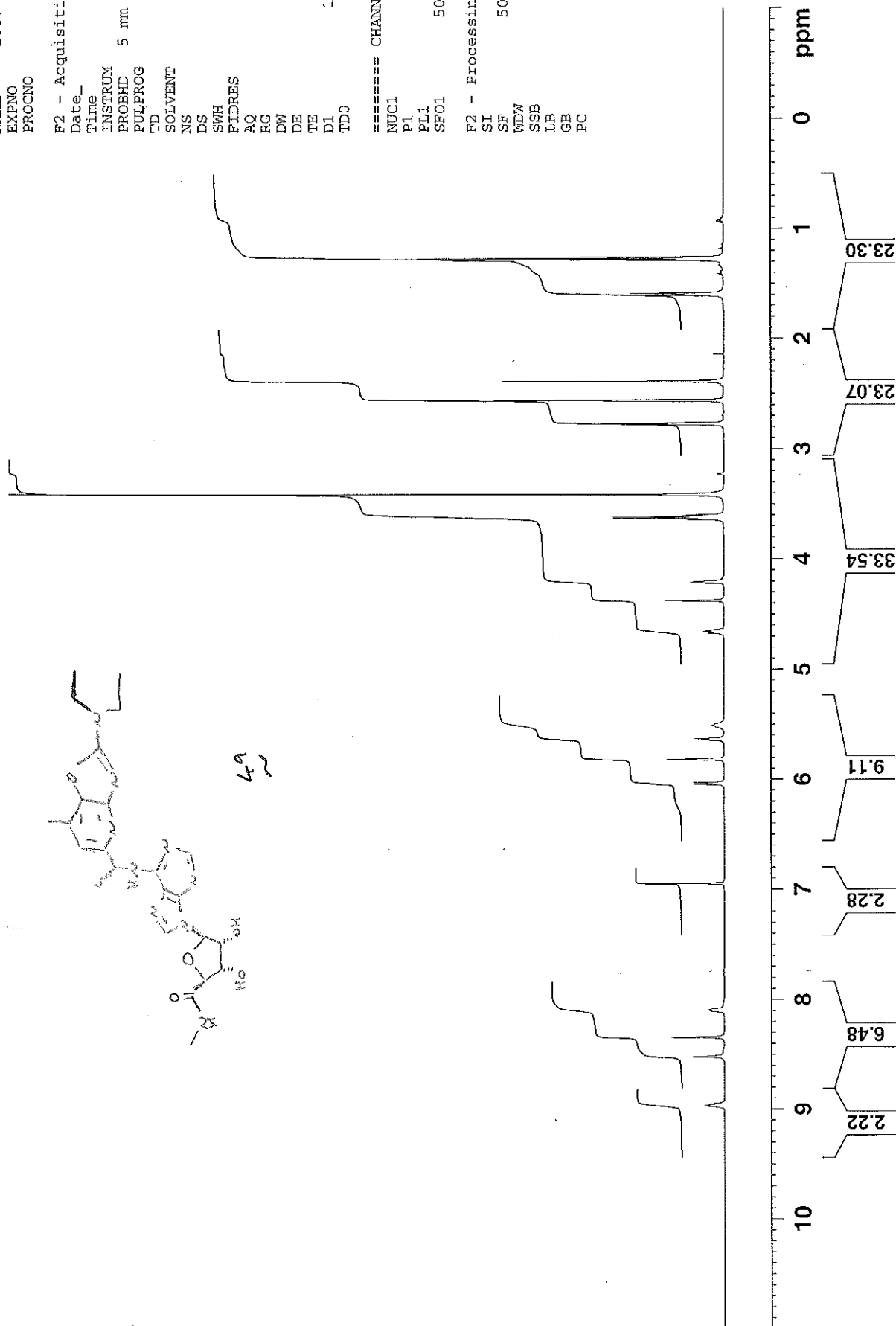
```

F2 - Acquisition Parameters
Date_      20070716
Time       19.37
INSTRUM    spect
PROBHD     5 mm TXI 1H/D-
PULPROG    zg30b
TD          65536
SOLVENT     DMSO
NS          500
DS          0
SWH         10330.578 Hz
FIDRES     0.157632 Hz
AQ         3.1719923 sec
RG         322
DW         48.400 usec
DE         13.07 usec
TE         300.0 K
D1         1.00000000 sec
TD0        1

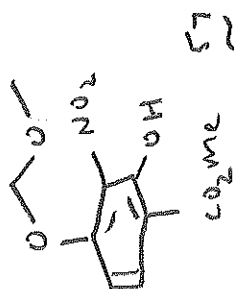
```

```
===== CHANNEL f1 =====
NUC1          1H
P1            39.89 usec
PL1           3.25 dB
SFO1          500.1330885 MHz
```

F2 - Processing parameters	
SI	32768
SF	500.129748 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

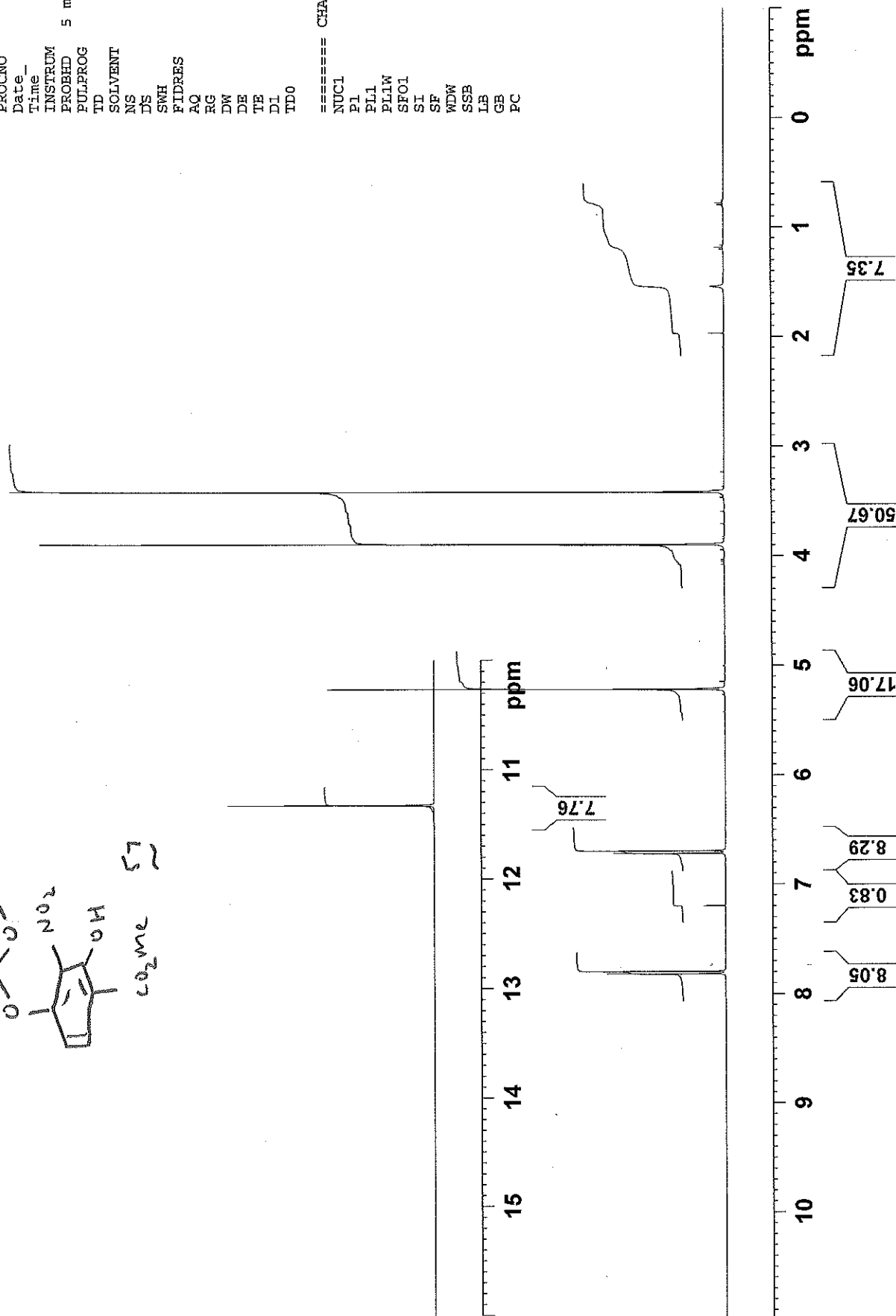


EB091
mPROTONight CDCl3 {e:\bruk400data\2010\Jun} ejt 8



NAME
EXPNO 2010-06-23-ejt-8
PROCNO 10
Date_ 20100624
Time_ 11.01
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zg30b
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8264.463 Hz
FIDRES 0.126106 Hz
AQ 3.9649780 sec
RG 203
DW 60.500 usec
DE 9.40 usec
TE 294.9 K
D1 1.00000000 sec
D1 1
TD0

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -3.60 dB
PL1W 17.83863831 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300356 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

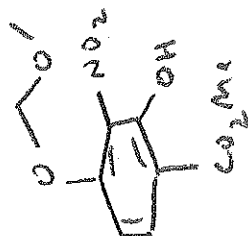


EB091

mCARBONight CDCI3 {e:\bruk400data\2010\Jun} ejt 8



169.40
154.50
153.97
132.35
131.52
107.60
105.77
94.79
77.36
77.05
76.73
56.92
52.78



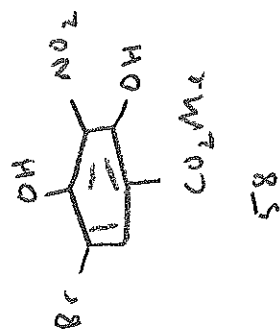
NAME 2010-06-23-ejt-8
EXPNO 11
PROCNO 1
Date_ 20100624
Time_ 11.26
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 2
SWH 30241.936 Hz
FIDRES 0.461455 Hz
AQ 1.0835786 sec
RG 512
DW 16.533 usec
DE 7.83 usec
TE 295.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 0.00 dB
PL1W 34.91522217 W
SF01 100.6241042 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.60 dB
PL12 15.31 dB
PL13 18.00 dB
PL1W 17.83863831 W
PL12W 0.22927761 W
PL13W 0.12341322 W
SF02 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

240 220 200 180 160 140 120 100 80 60 20 ppm

EBstep4
mPROTON CDCl3 {E:\bruk300data\2010\Dec} ejt 4

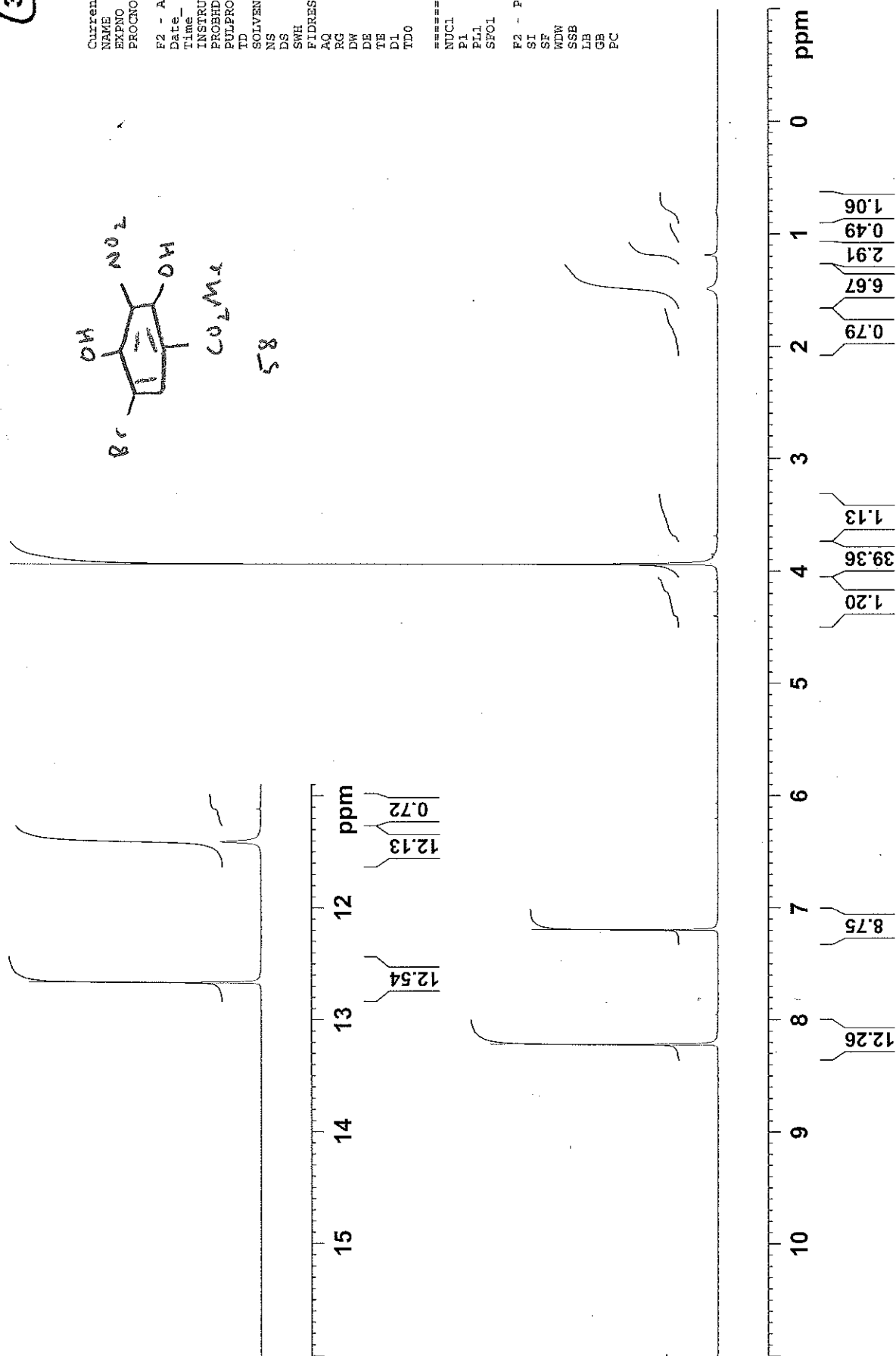


Current Data Parameters
NAME 2010-12-13-ejt-4
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101213
Time 10.23
INSTRUM DPX300
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 1448.2
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -2.60 dB
SFO1 299.9518523 MHz

F2 - Processing parameters
SI 32768
SF 299.9500266 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

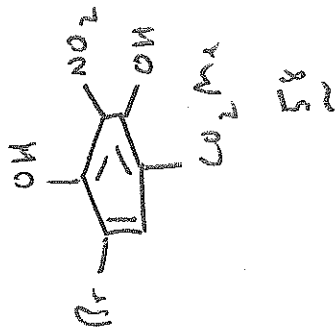


EB084

mCARBONnight CDCI3 {e:\bruk400data\2010\Jun} ejt 42



169.04
158.86
157.07
139.20
126.08
106.62
101.04
77.36
77.04
76.72
53.27



NAME 2010-06-16-ejt-42
EXPNO 11
PROCNO 1
Date_ 20100617
Time 6.26
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCI3
NS 256
DS 2
SWH 30241.936 Hz
FIDRES 0.461455 Hz
AQ 1.0835786 sec
RG 512
DW 16.533 usec
DE 7.83 usec
TE 294.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

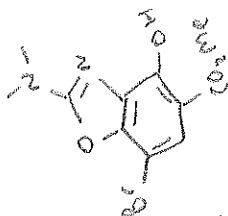
===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 0.00 dB
PL1W 34.91522217 W
SFO1 100.6241042 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.60 dB
PL12 15.31 dB
PL13 18.00 dB
PL2W 17.83863831 W
PL12W 0.22927761 W
PL13W 0.12341322 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

240 220 200 180 160 140 120 100 80 60 40 20 ppm

EB087

mPROTONnight CDCl3 {e:\bruk400data\2010\Jun} ejt 31



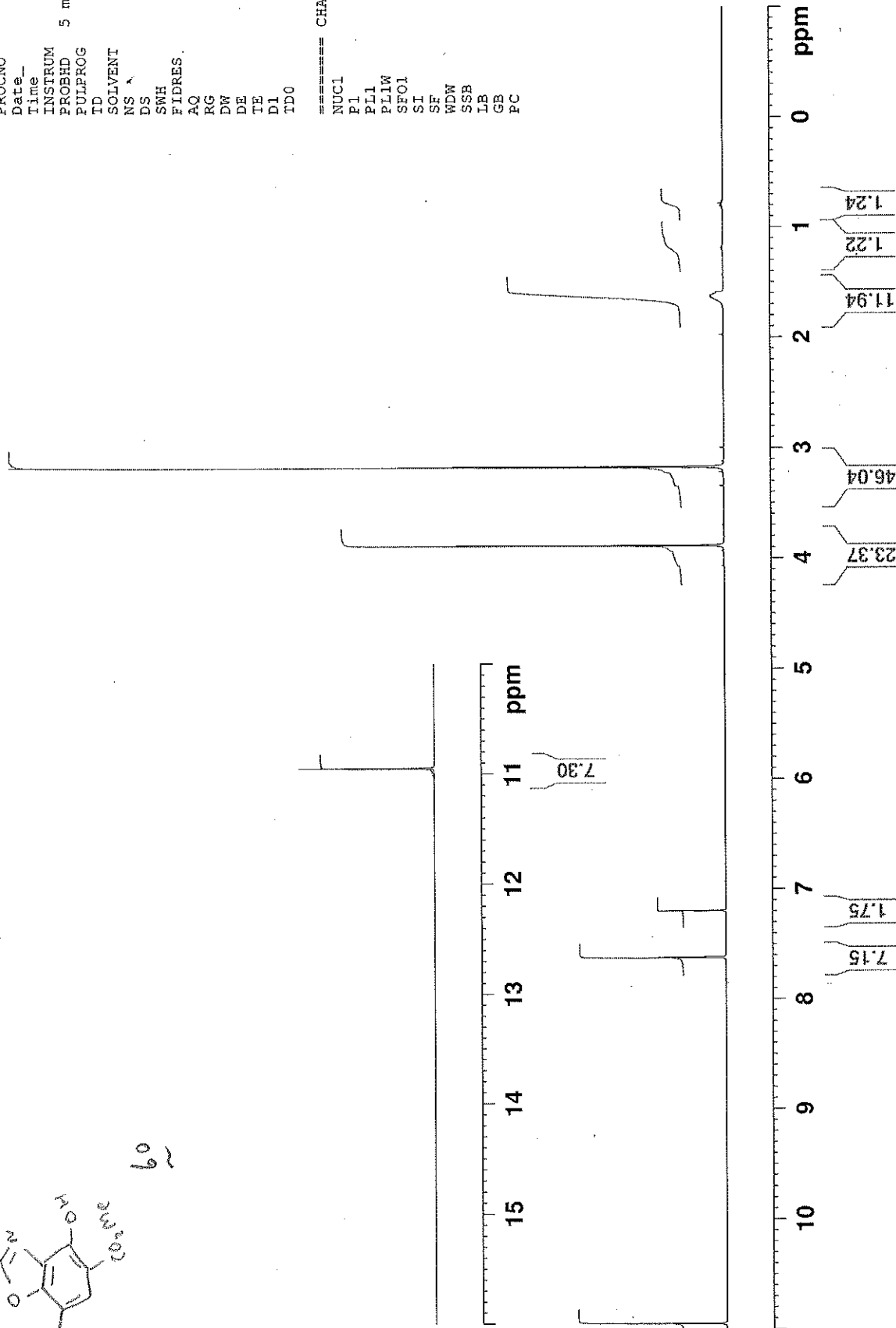
60



2010-06-14-ejt-31

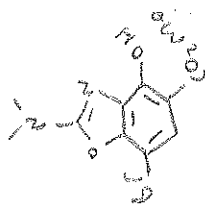
NAME
EXPNO 10
PROCNO 1
Date_ 20100614
Time_ 20.58
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zg30p
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8264.463 Hz
FIDRES 0.126106 Hz
AQ 3.9649780 sec
RG 362
DW 60.500 usec
DE 9.40 usec
TE 294.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -3.60 dB
PL1W 17.83863831 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300350 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

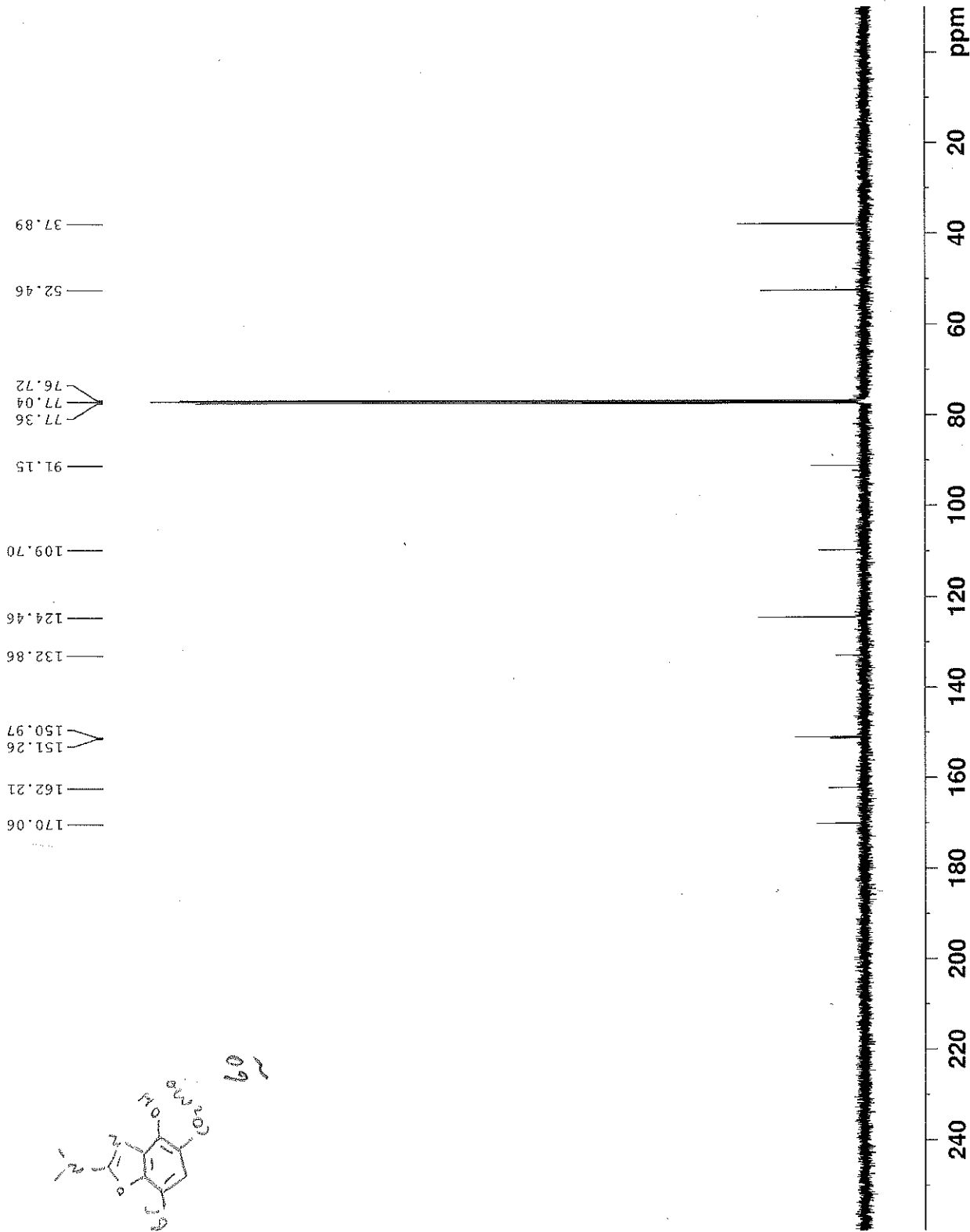


EB087

mCARBONnight CDCI3 {e:\bruk400data\2010\Jun} ejt 31



60



```

NAME      2010-06-14-ejt-31
EXPNO      1
PROCNO      1
Date_       20100614
Time        21.12
INSTRUM     AV400
PROBHD      5 mm PABCO BB-
PULPROG     zgpg30
TD          65536
SOLVENT      CDCl3
NS          256
DS          2
SWH          30241.936 Hz
FIDRES      0.461455 Hz
AQ          1.0835786 sec
RG          512
DW          16.533 usec
DE          7.83 usec
TE          294.6 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1        13C
P1          8.00 usec
PL1         0.00 dB
PL1W        34.9152217 W
SFO1        100.6241042 MHz

===== CHANNEL f2 =====
CPDPRG2     waltz16
NUC2        1H
PCPD2       90.00 usec
PL2         -3.60 dB
PL12        15.31 dB
PL13        18.00 dB
PL2W        17.83863931 W
PL12W       0.22927761 W
PL13W       0.12341322 W
SFO2        400.1316005 MHz
SI          32768
SF          100.6127690 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

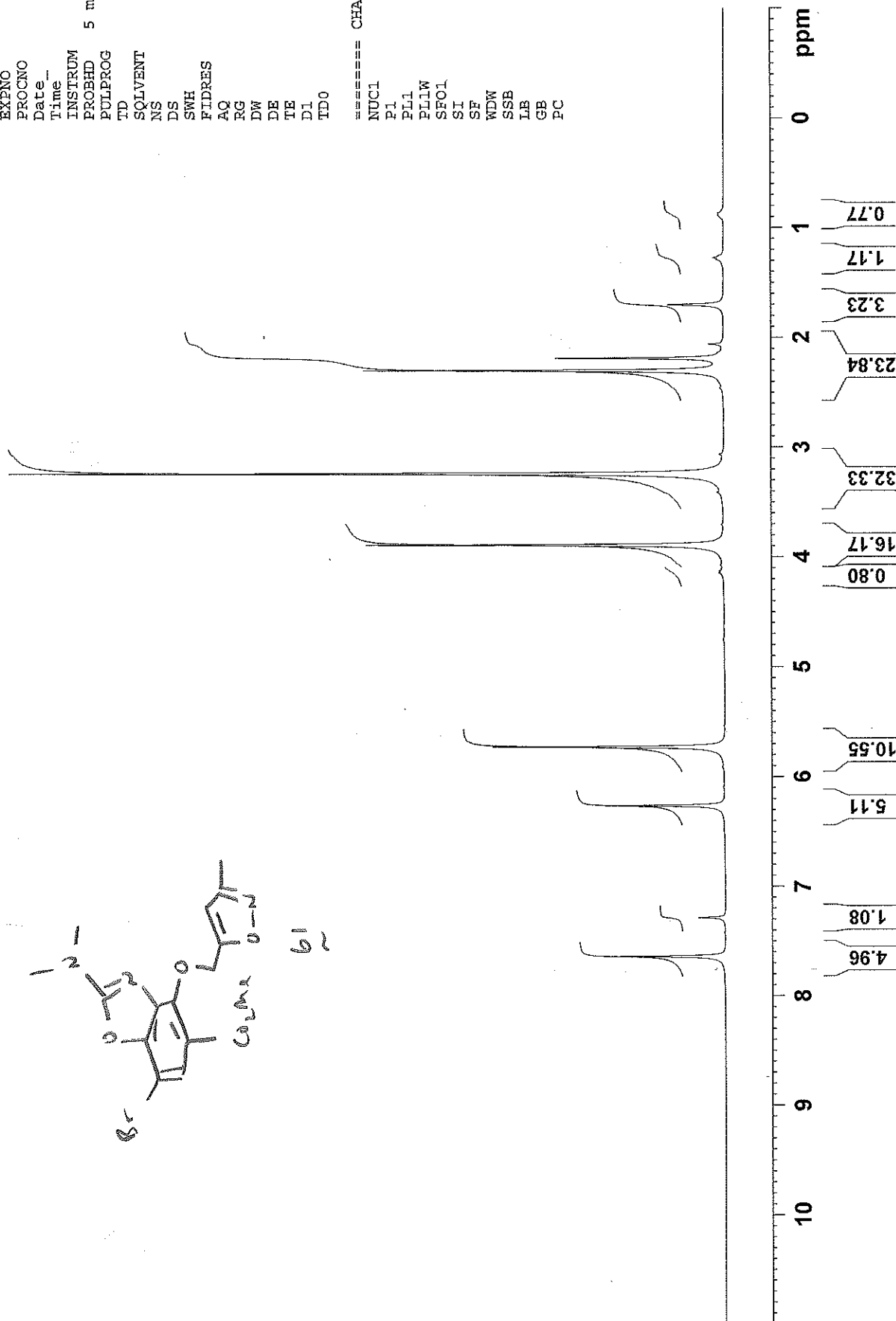
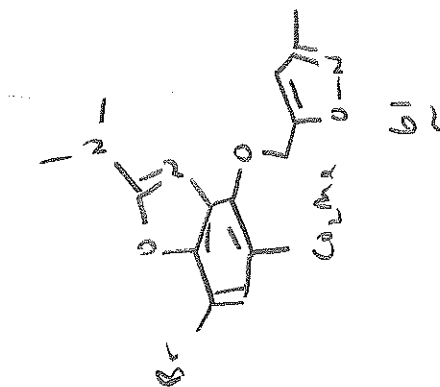
EB112

mPROTON CDCI3 {e:\bruk400data\2010\Sep} ejt 42



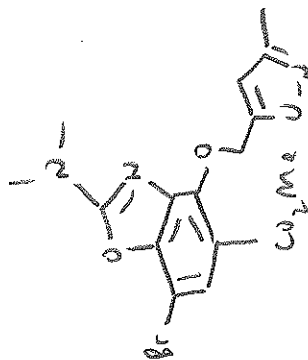
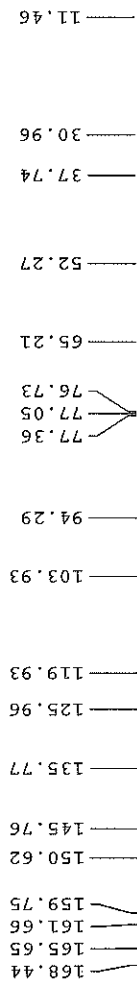
NAME 2010-09-15-ejt-42
 EXPNO 10
 PROCNO 1
 Date_ 20100915
 Time_ 17.22
 INSTRUM AV400
 PROBHD 5 mm PABO BB-
 PULPROG zg30b
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 8264.463 Hz
 FIDRES 0.126106 Hz
 AQ 3.9649780 sec
 RG 256
 DW 60.500 usec
 DE 9.40 usec
 TE 293.7 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.00 usec
 PL1 -3.60 dB
 PL1W 17.83863831 W
 SF01 400.1324710 MHz
 SI 32768
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



EB112

mCARBONight CDC13 {e:\bruk400data\2010\Sep} ejt 42



61

NAME 2010-09-15-ejt-42
 EXPNO 11
 PROCNO 1
 Date 20100916
 Time 7.22
 INSTRUM AV400
 PROBD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 *SOLVENT CDCl3
 NS 256
 DS 2
 SWH 30241.936 Hz
 FIDRES 0.461455 Hz
 AQ 1.0835786 sec
 RG 512
 DW 16.533 usec
 DE 7.83 usec
 TE 294.4 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 0.00 dB
 PL1W 34.91522217 W
 SFO1 100.6241042 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P1 90.00 usec
 PL1 -3.60 dB
 PL12 15.31 dB
 PL13 18.00 dB
 PL2W 17.83863831 W
 PL12W 0.22927761 W
 PL13W 0.12341322 W
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

240 220 200 180 160 140 120 100 80 60 40 20 ppm

E. Burnell

0910-047

EB111

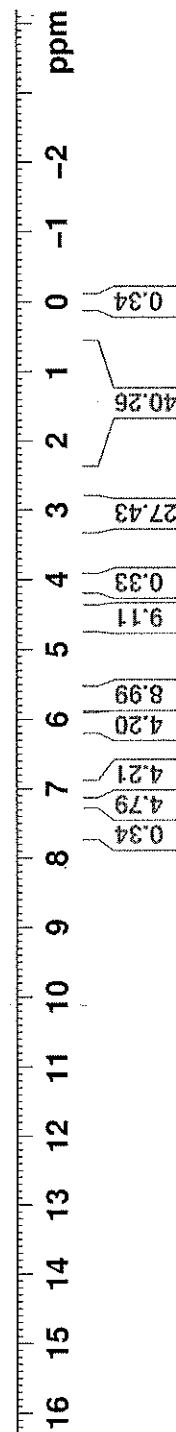
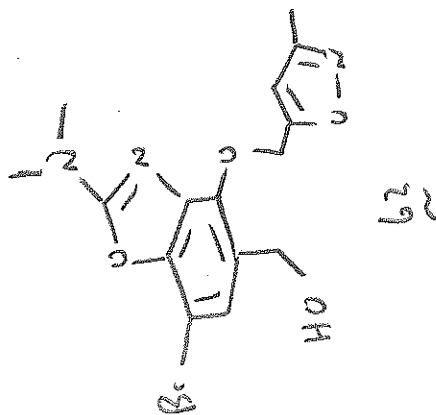
mPROTON CDCl3 {E:\bruk400service_data\2010\Sep} Administrator 45



2010-09-21-Administrator-45

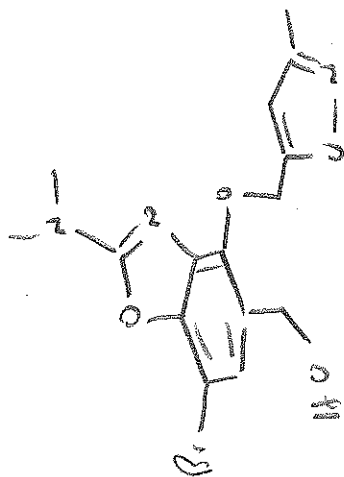
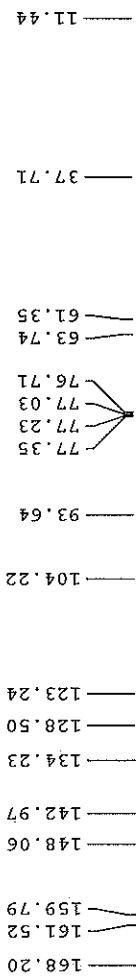
NAME
EXPNO 1
PROCNO 1
Date_ 20100921
Time 10.17
INSTRUM AV400-S
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT A CDCl3
NS 64
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 512
DW 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -3.60 dB
PL1W 18.98951721 W
SF01 400.2324716 MHz
SI 32768
SF 400.2300392 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



EB111

mCARBONnight CDCI3 {e:\bruk400data\2010\Sep} ejt 26



```

NAME      2010-09-17-ejt-26
EXPNO     20
PROCNO     1
Date_      20100918
Time       17.27
INSTRUM    AV400
PROBHD     5 mm PABBO BB-
PULPROG    zgpg30
TD         65536
SOLVENT     CDCl3
NS         3072
DS         2
SWH         30241.936 Hz
FIDRES     0.461455 Hz
AQ         1.0835786 sec
RG         512
DE         16.533 usec
TE         7.83 usec
D1         295.1 K
D11        2.00000000 sec
TD0        0.03000000 sec
          1

===== CHANNEL f1 =====
NUC1       13C
P1         8.00 usec
PL1        0.00 dB
PL1W       34.91522217 W
SFO1       100.6241042 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        -3.60 dB
PL12       15.31 dB
PL13       18.00 dB
PL2W       17.83863831 W
PL12W      0.22327761 W
PL13W      0.12341322 W
SFO2       400.1316005 MHz
SI         32768
SF         100.6127690 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

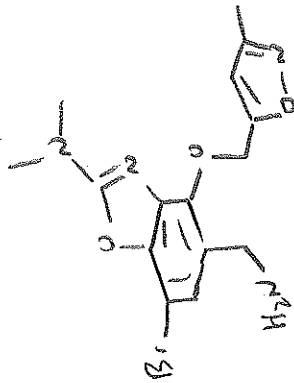
240 220 200 180 160 140 120 100 80 60 40 20 ppm

EB step 10 deprotection
mPROTONnight CDC13 /opt/bruk500data/2011/Mar ejt 22

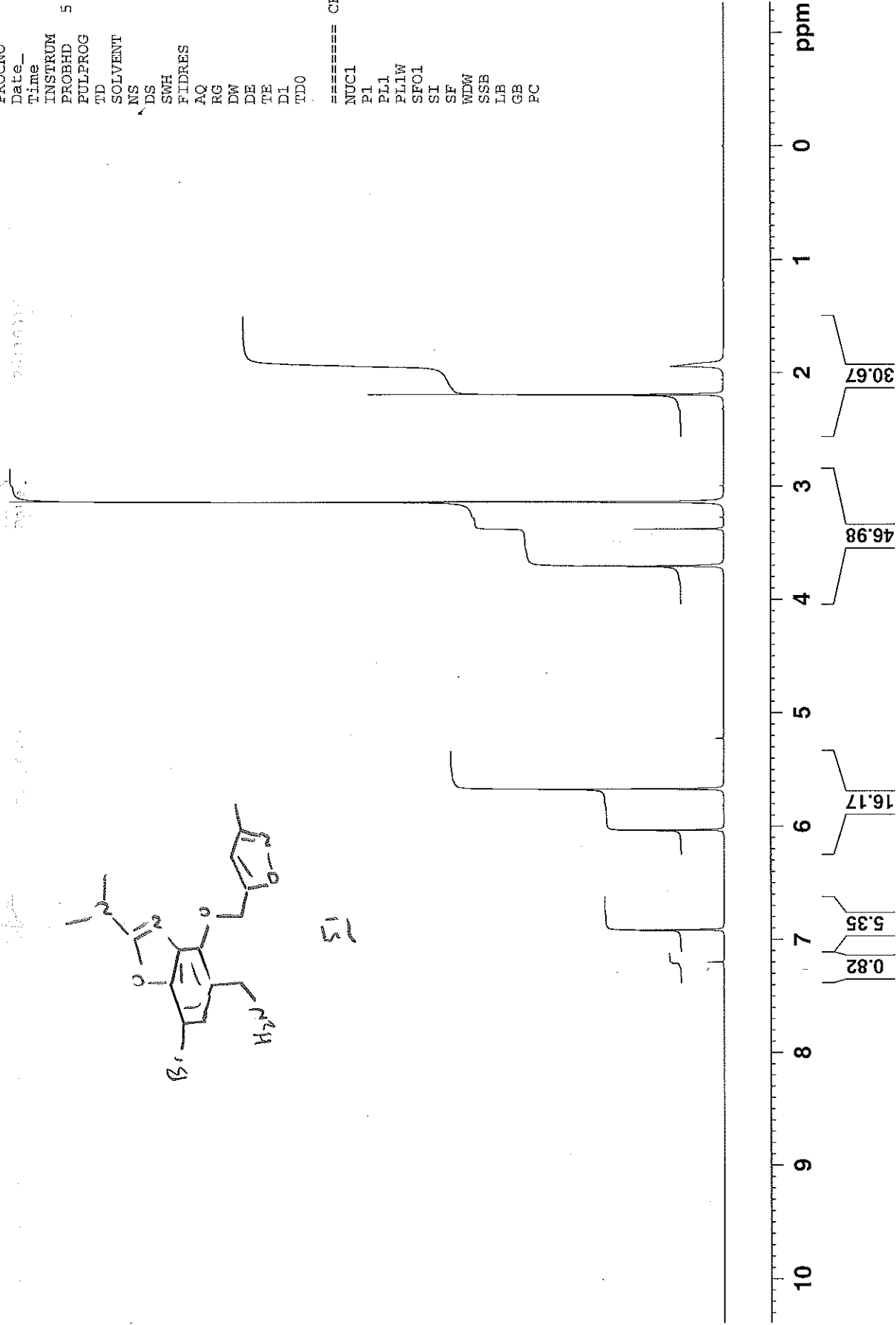


NAME 2011-03-15-ejt-22
EXPNO 10
PROCNO 1
Date_ 20110316
Time 1.30
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zg30b
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 90.5
DW 48.400 usec
DE 13.38 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.20 usec
PL1 3.25 dB
PL1W 12.12272263 W
SF01 500.1330885 MHz
SI 32768
SF 500.1300428 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



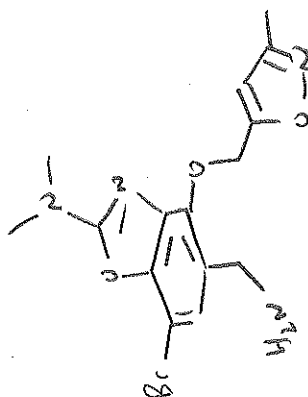
13



EB step 10 deprotection mCARBONight CDC13 /opt/bruk500data/2011/Mar ejt 22



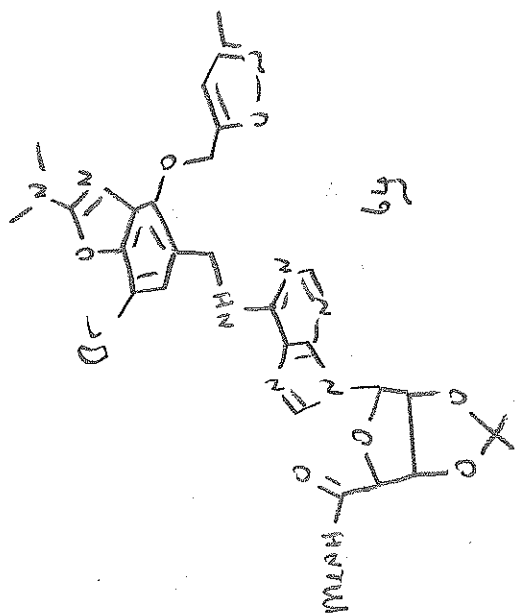
168.42
161.45
159.73
147.54
142.95
134.31
130.59
123.00
104.12
93.43
77.32
77.06
76.81
63.66
42.09
37.69
11.43



```

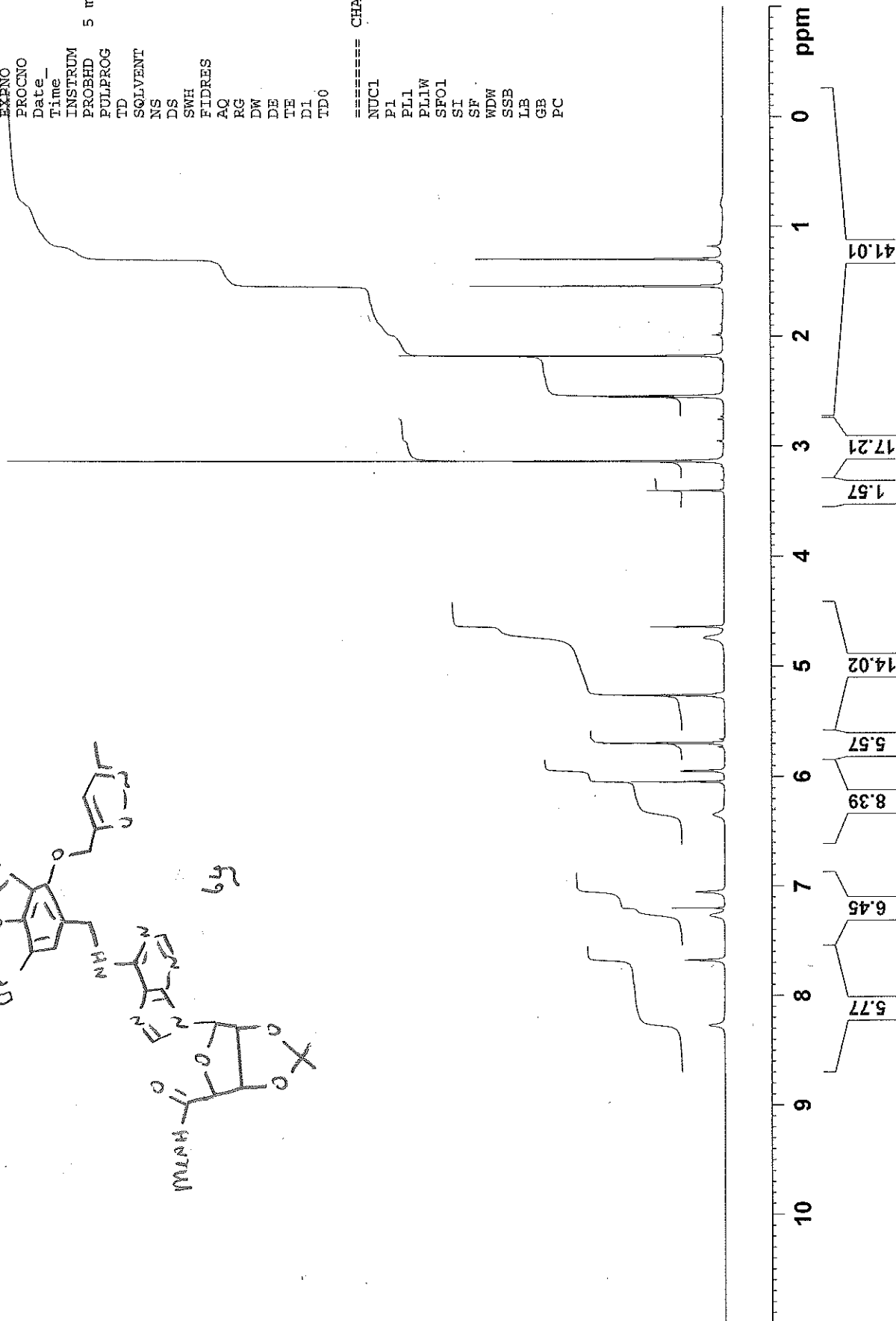
NAME      2011-03-15-ejt-22
EXPNO     11
PROCNO    1
Date_     20110316
Time      1.43
INSTRUM   spect
PROBHD    5 mm TXI 1H/D-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         256
DS         2
SMH        29761.904 Hz
FIDRES    0.454131 Hz
AQ         1.1010548 sec
RG         512
DW         16.800 usec
DE         32.21 usec
TE         295.7 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
===== CHANNEL f1 =====
NUC1       13C
P1         11.50 usec
PL1        -4.20 dB
FL1W       218.02882385 W
SF01       125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        4.20 dB
PL12       23.99 dB
PL13       23.00 dB
PL1W       9.74092484 W
PL12W      0.10223514 W
PL13W      0.12841040 W
SFO2       500.1320005 MHz
SI         32768
SF         125.7577890 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

200 180 160 140 120 100 80 60 40 20 0 ppm



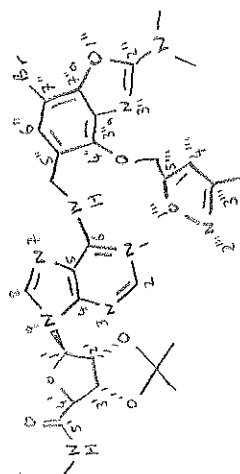
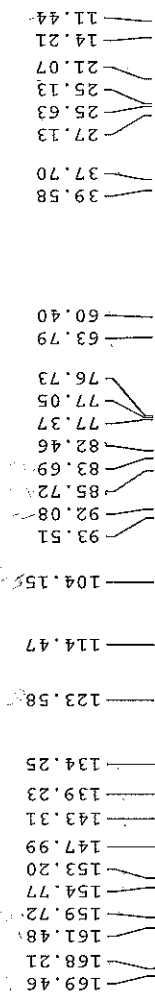
NAME EXPNO 2011-04-06-ejt-54
PROCNO 10
Date_ 20110407
Time_ 18.36
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zg30b
TD 65536
SOLVENT CDCI3
NS 16
DS 0
SWH 8264.463 Hz
FIDRES 0.126106 Hz
AQ 3.9649780 sec
RG 228
DW 60.500 usec
DE 9.40 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -3.60 dB
PL1W 17.83863831 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300343 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



EB step11

mCARBONnight CDC13 {e:\bruk400data\2011\Apr} ejt 21

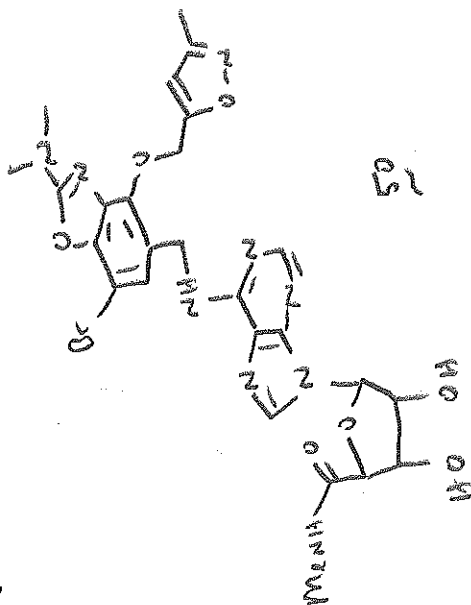


64

2011-04-13-ejt-21
NAME EXPNO 11
PROCNO 1
Date_ 20110414
Time_ 8.26
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1000
DS 2
SWH 30241.936 Hz
FIDRES 0.461455 Hz
AQ 1.0835786 sec
RG 512
DE 16.533 usec
TE 7.83 usec
D1 295.3 K
D11 2.0000000 sec
D11 0.0300000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 0.00 dB
PL1W 34.91522217 W
SFO1 100.6241042 MHz
===== CHANNEL f2 =====
CDPRG2 waltz16
NUC2 1H
POPD2 90.00 usec
PL2 -3.60 dB
PL12 15.31 dB
PL13 18.00 dB
PL1W 17.83863831 W
PL12W 0.22927761 W
PL13W 0.12341322 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

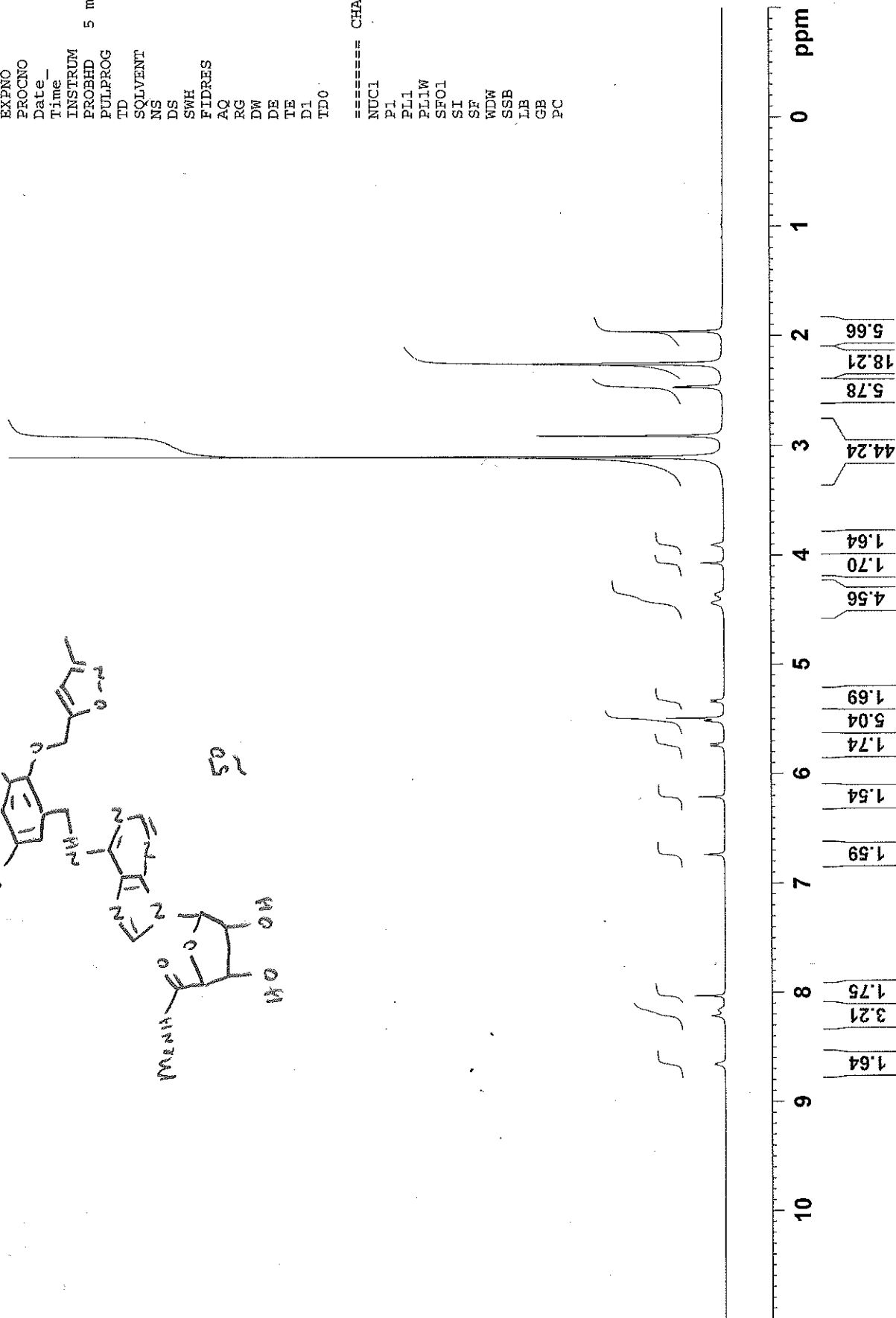
240 220 200 180 160 140 120 100 80 60 40 20 ppm

EBdeprotection
mPROTON DMSO {e:\bruk400data\2011\Feb} ejt 35

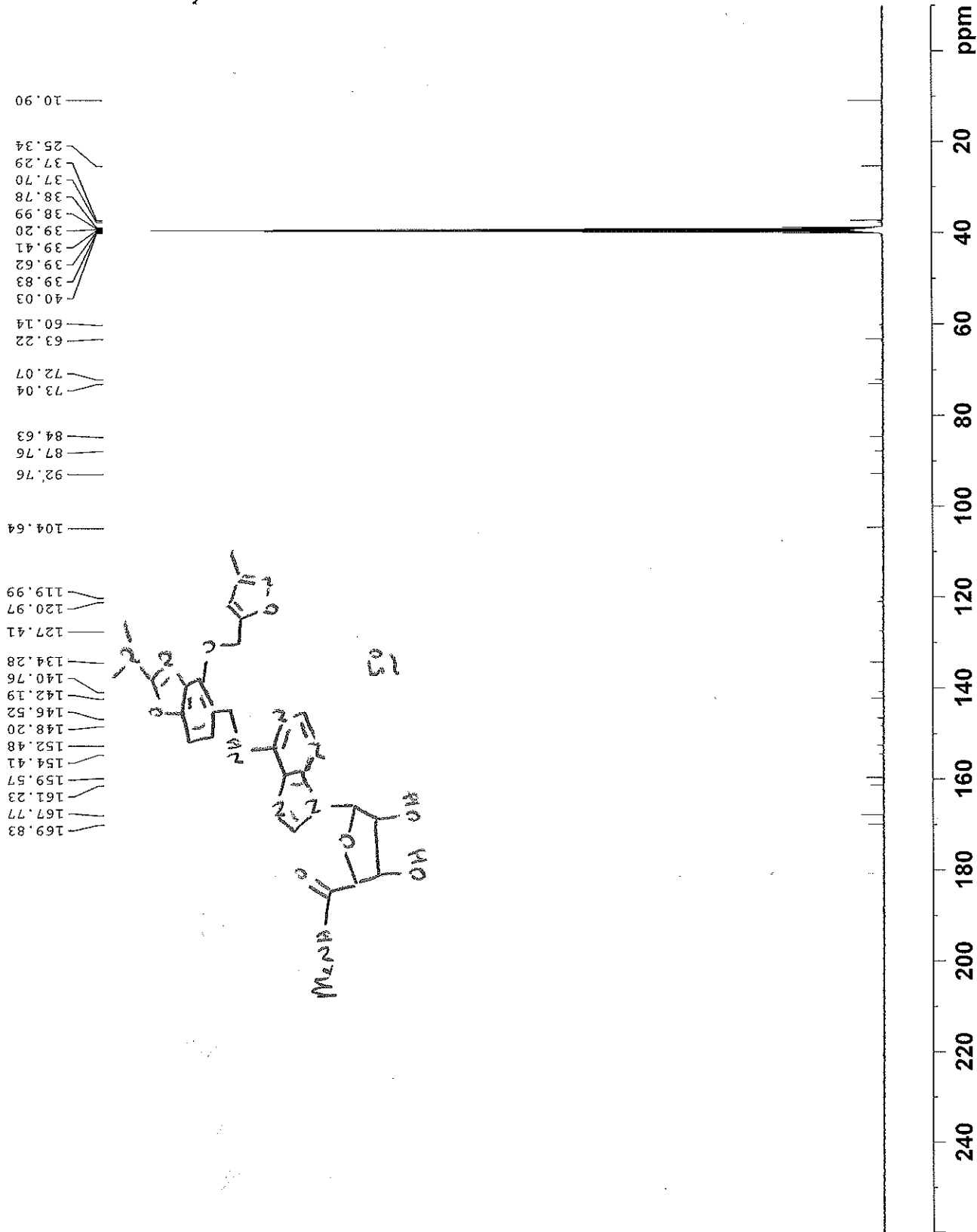


NAME 2011-02-04-ejt-35
EXPNO 10
PROCNO 1
Date_ 20110204
Time_ 9.56
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zg30b
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 8264.463 Hz
FIDRES 0.126106 Hz
AQ 3.9649780 sec
RG 322
DW 60.500 usec
DE 9.40 usec
TE 293.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -3.60 dB
PL1W 17.83863831 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300996 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



EB step 12
mCARBONight DMSO {e:\bruk400data\2011\May} ejt 15



```

NAME          2011-05-10-ejt-15
EXPNO         10
PROCNO        1
Date_         20110515
Time          1.49
INSTRUM       AV400
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            3072
DS            2
SWH           30241.936 Hz
FIDRES       0.461455 Hz
AQ           1.0835786 sec
RG           512
DW           16.533 usec
DE           7.83 usec
TE           293.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0          1

===== CHANNEL f1 =====
NUC1          13C
P1           8.00 usec
PL1          0.00 dB
PL1W         34.91522217 W
SFO1         100.6241042 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
P2           90.00 usec
PL2          -3.60 dB
PL2W         15.31 dB
PL13         18.00 dB
PL12W        17.83863831 W
PL12W        0.22927761 W
PL13W        0.12341322 W
SFO2         400.1316005 MHz
SI           32768
SF           100.6128193 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
  
```