Stereoselective synthesis of oxazolidinonyl-fused piperidines of interest as selective muscarinic (M_1) receptor agonists

Kenneth J. Broadley, Maxime G. P. Buffat, Erica Burnell, Robin H. Davies, Xavier Moreau, Stephen Snee and Eric J. Thomas

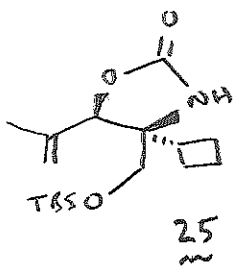
Supplementary Data

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

M Buffat
MB161570C
cdcl3
Inova 400(JF)
Aramis
Proton 400MHz
10.10.02
1002041noe

Solvent: cdcl3
Ambient temperature
User: 1-12-87
File: 1002041
DATE Oct 10 2002
INOVA-300 "solids"

PULSE SEQUENCE
Relax. delay 30.000 sec
Pulse 36.0 degrees
Acq. time 4.008 sec
Width 3559.2 Hz
64 repetitions
OBSERVE H1, 399.9584369 MHz
DECOUPLE H1, 399.9598074 MHz
Power 0 dB
off during acquisition
on during delay
single frequency
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 74 minutes



25

Integral Scale = 25000.0
Vertical Scale = 2500.0

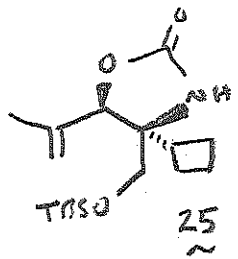
7 6 5 4 3 2 1 -0 ppm

3.34 3.33 3.41 2.98 5.96 2.96 30.40 29.72 17.91

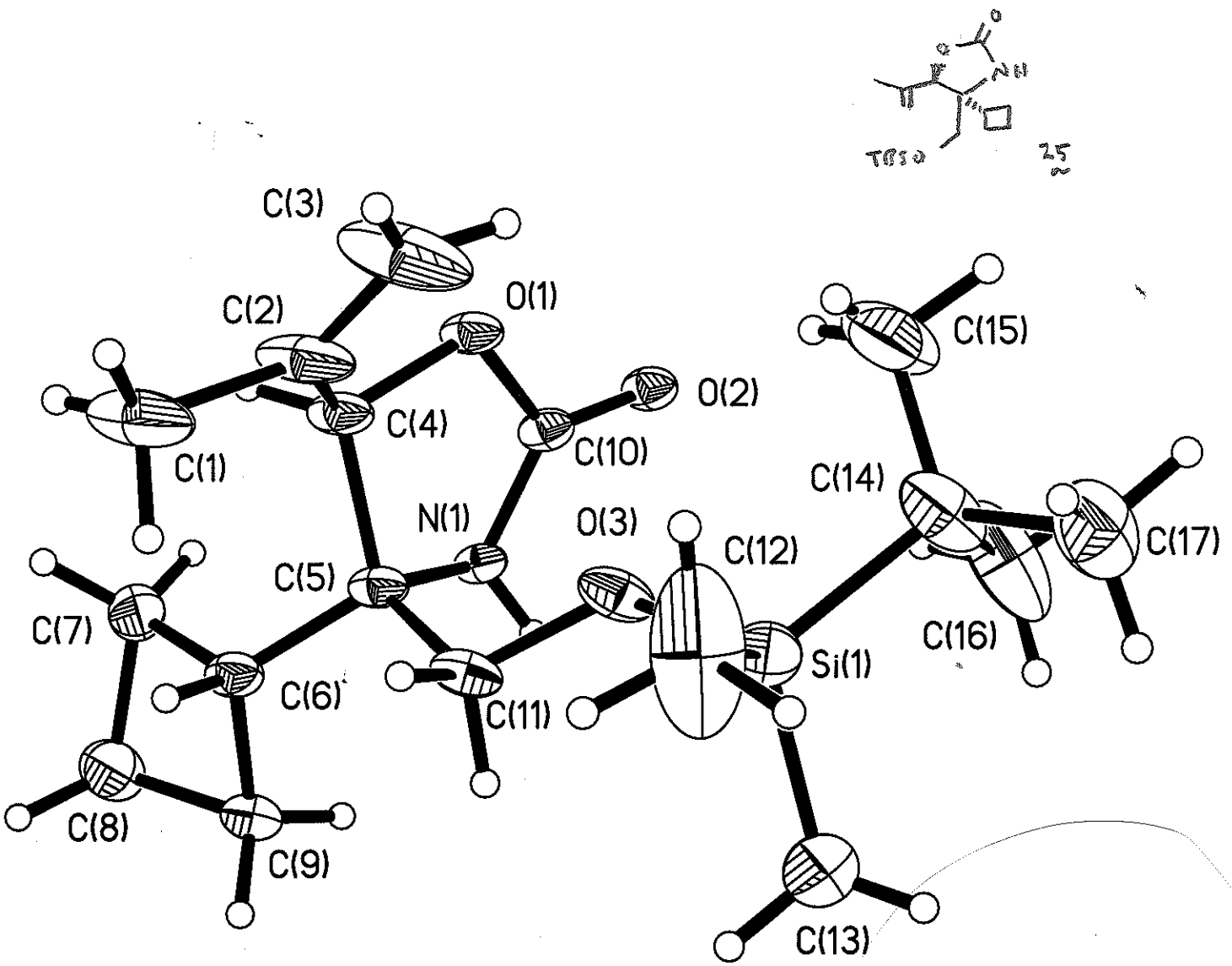
Buffat Maxime
MB/6/570/C
cdcl3
Inova 400(GS)
Aramis
13C 100MHz
09.10.02
1002041c

Solvent: cdcl3
Ambient temperature
File: 1002041
DATE Oct 9 2002
INOVA-300 "solids"

PULSE SEQUENCE
Pulse 36.0 degrees
Acq. time 1.311 sec
Width 25000.0 Hz
37712 repetitions
OBSERVE C13, 100.5696317 MHz
DECOUPLE H1, 399.9597752 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 13.7 hours



25

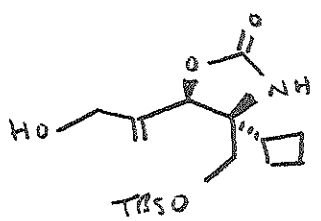

C(3)
C(2)
O(1)
C(4)
O(2)
C(10)
C(1)
N(1)
O(3)
C(12)
C(5)
C(7)
C(6)
C(11)
Si(1)
C(8)
C(9)
C(13)
C(15)
C(14)
C(16)
C(17)

Maxime BUFFAT
MB/6/594/C
cdcl3
Inova 400(MB)
Aramis
Proton 400MHz
02.11.02

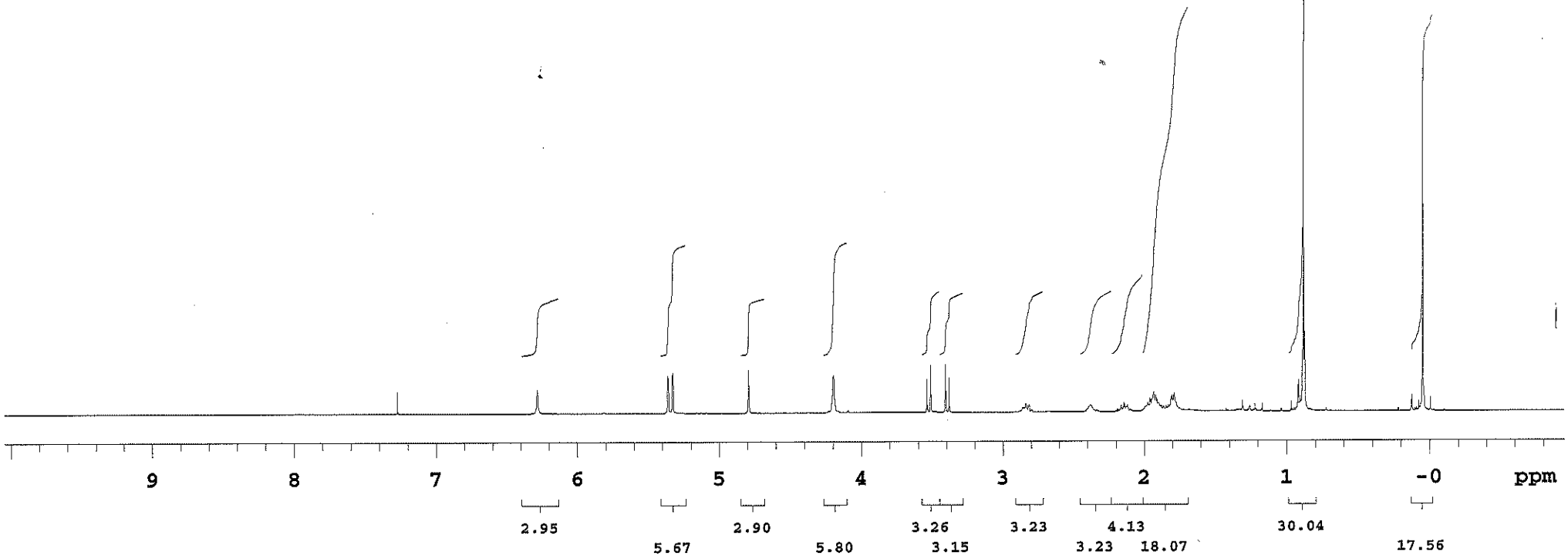
Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
User: 1-12-87
INOVA-400 "aramis"

Relax. delay 5.000 sec
Pulse 36.0 degrees
Acq. time 4.008 sec
Width 6387.7 Hz
96 repetitions
OBSERVE H1, 399.9584369 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 14 min, 25 sec



31

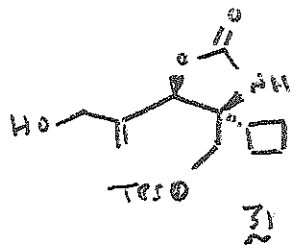


Maxime BUFFAT
MB/6/594/C
cdcl3
Inova 400(mb)
Aramis
13C 100MHz
02.11.02

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
File: MB.6.594.C-400C-02.11.02
INOVA-400 "aramis"

Pulse 36.0 degrees
Acq. time 1.311 sec
Width 25000.0 Hz
1282 repetitions
OBSERVE C13, 100.5696317 MHz
DECOUPLE H1, 399.9597752 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 366 hr, 32 min, 34 sec

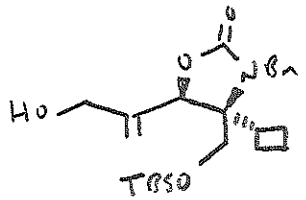


MAXIME BUFFAT
MB/6/598/C
cdcl3
Inova 400(mb)
Aramis
Proton 400MHz
03.11.02

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
User: 1-12-87
INOVA-400 "aramis"

Relax. delay 5.000 sec
Pulse 36.0 degrees
Acq. time 4.008 sec
Width 6387.7 Hz
96 repetitions
OBSERVE H1, 399.9584369 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 14 min, 25 sec



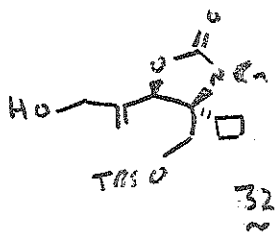
3.2

Maxime BUFFAT
MB/6/598/C
cdcl3
Inova 400(mb)
Aramis
13C 100MHz
03.11.02

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
INOVA-400 "aramis"

Pulse 36.0 degrees
Acq. time 1.311 sec
Width 25000.0 Hz
4544 repetitions
OBSERVE C13, 100.5696332 MHz
DECOUPLE H1, 399.9597752 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 36 hr, 39 min, 15 sec



32

158.812
144.064
138.465
128.466
127.472
127.351
115.493
78.017
77.319
77.000
76.681
68.351
63.891
62.768
45.827
38.567
25.769
23.099
22.848
18.160
17.120
-5.813
-6.026

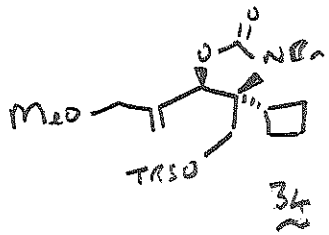
200 180 160 140 120 100 80 60 40 20 ppm

Maxime BUFFAT
MB/6/599/A
cdcl3
Inova 400(mb)
Aramis
Proton 400MHz
03.11.02

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
User: 1-12-87
INOVA-400 "aramis"

Relax. delay 5.000 sec
Pulse 36.0 degrees
Acq. time 4.008 sec
Width 6387.7 Hz
96 repetitions
OBSERVE H1, 399.9584369 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 14 min, 25 sec



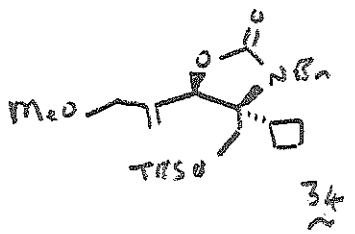
34

Maxime BUFFAT
MB/6/599/A
cdcl3
Inova 400(mb)
Aramis
13C 100MHz
03.11.02

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
INOVA-400 "aramis"

Pulse 36.0 degrees
Acq. time 1.311 sec
Width 25000.0 Hz
8175 repetitions
OBSERVE C13, 100.5696340 MHz
DECOUPLE H1, 399.9597752 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 36 hr, 39 min, 15 sec

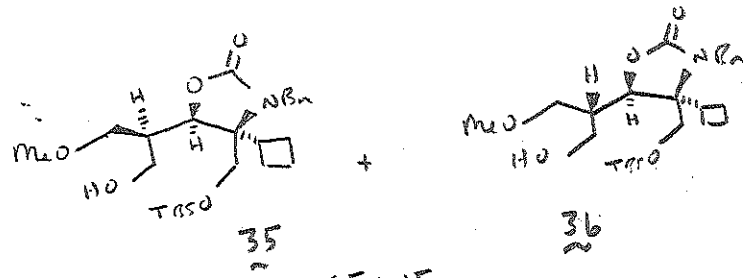


34

Maxime BUFFAT
MB/6/595/AB
cdcl3
Inova 400(MB)
Aramis
Proton 400MHz
02.11.02

Solvent: cdcl3
Ambient temperature
User: 1-12-87
File: MB.6.595.AB-400H-02.11.02
DATE Nov 2 2002
INOVA-300 "solids"

PULSE SEQUENCE
Relax. delay 5.000 sec
Pulse 36.0 degrees
Acq. time 4.008 sec
Width 6387.7 Hz
96 repetitions
OBSERVE H1, 399.9584369 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 14 minutes



35

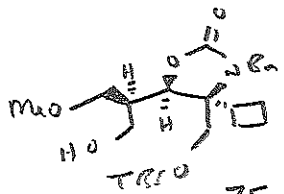
+

36

85 : 15

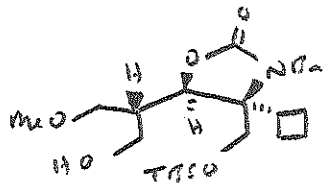
Maxime BUFFAT
MB/6/595/AB
cdcl3
Inova 400(mb)
Aramis
13C 100MHz
02.11.02

Pulse Sequence: s2pul



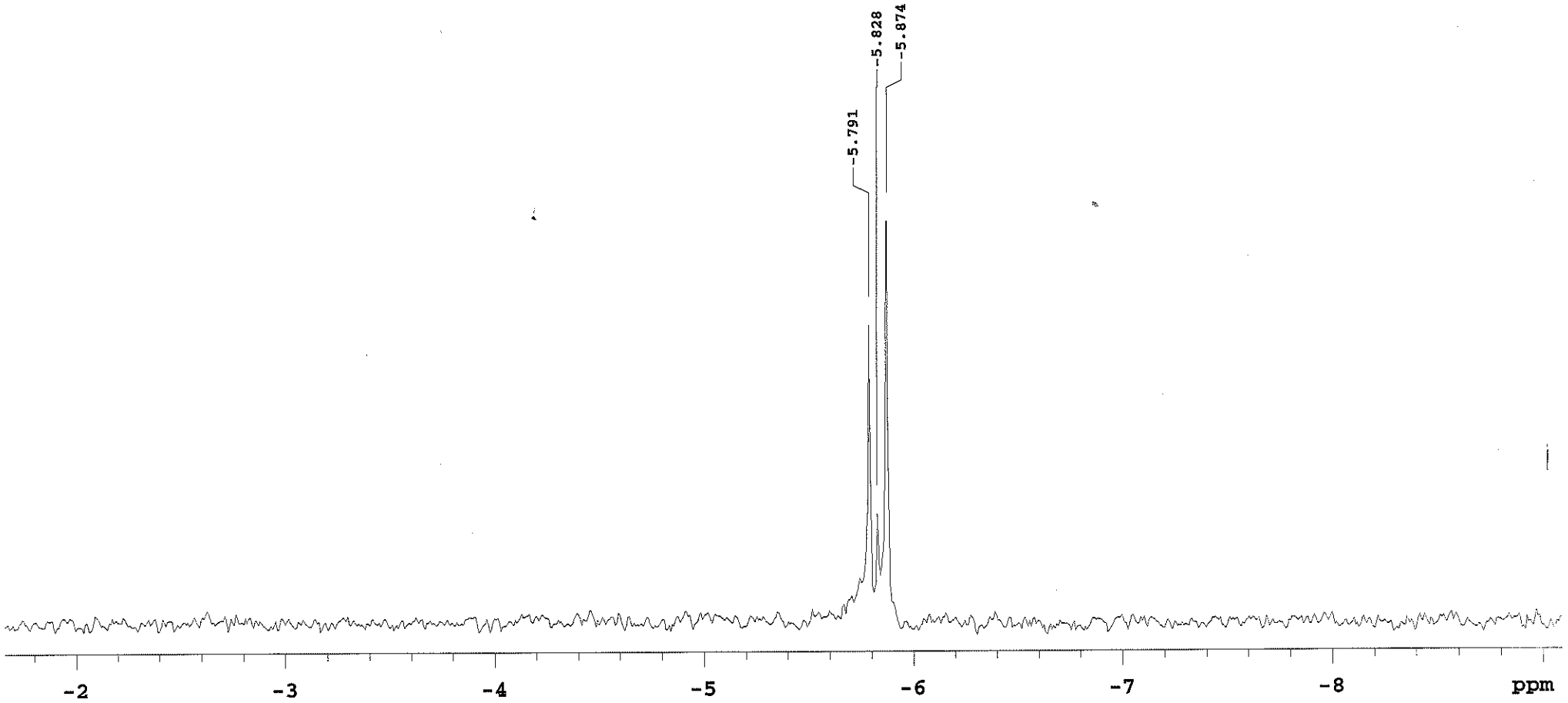
35

+



36

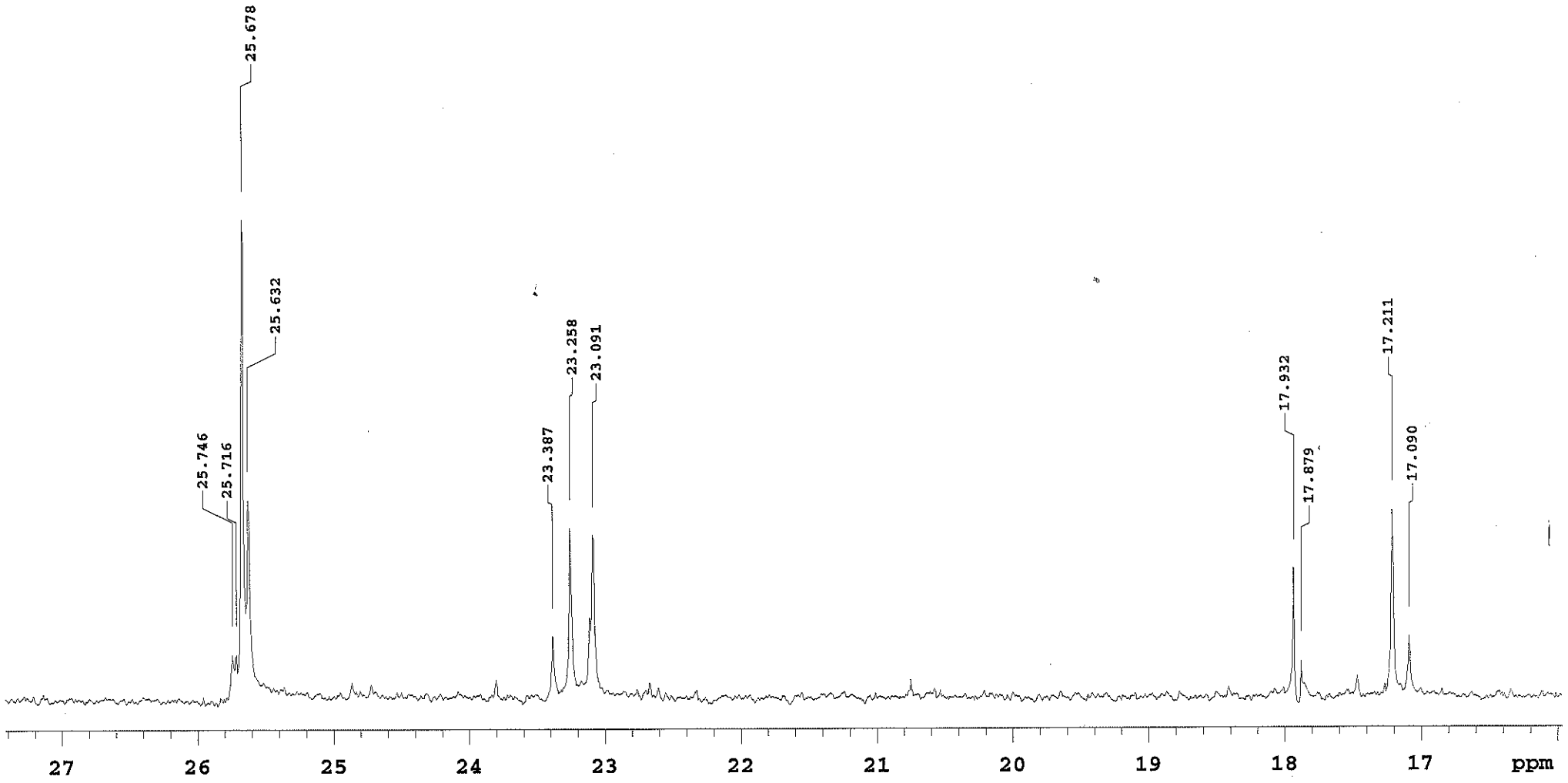
85 : 15



Maxime BUFFAT
MB/6/595/AB
cdcl3
Inova 400(mb)
Aramis
13C 100MHz
02.11.02

Pulse Sequence: s2pul

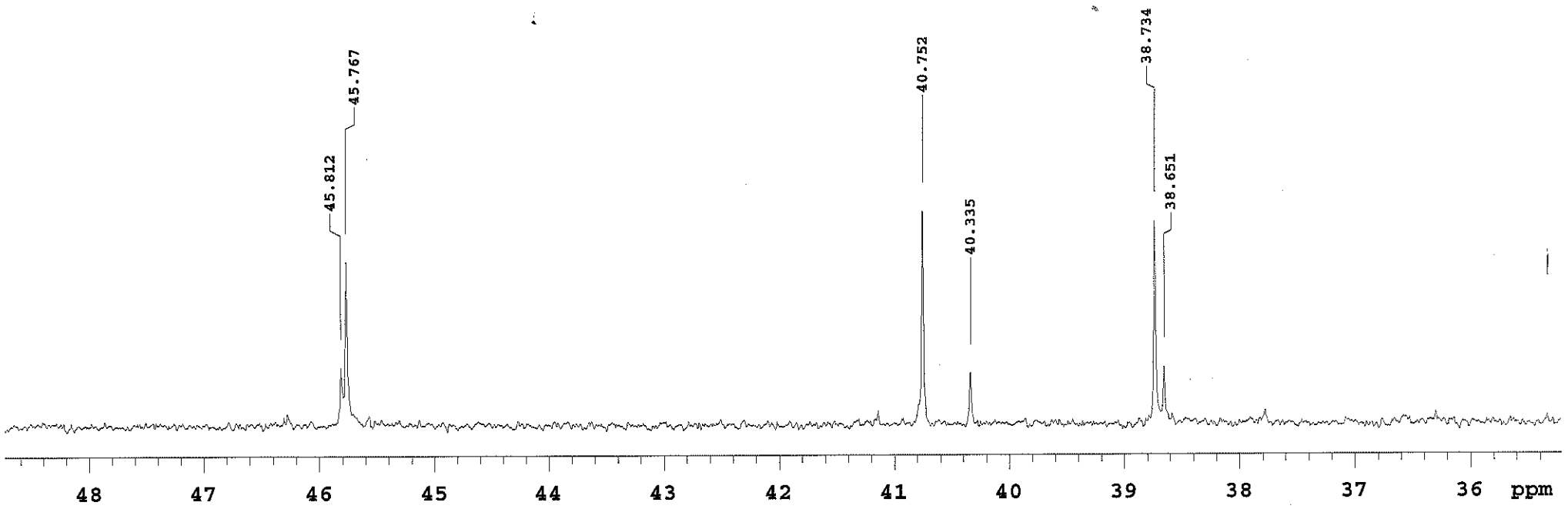
35 + 36

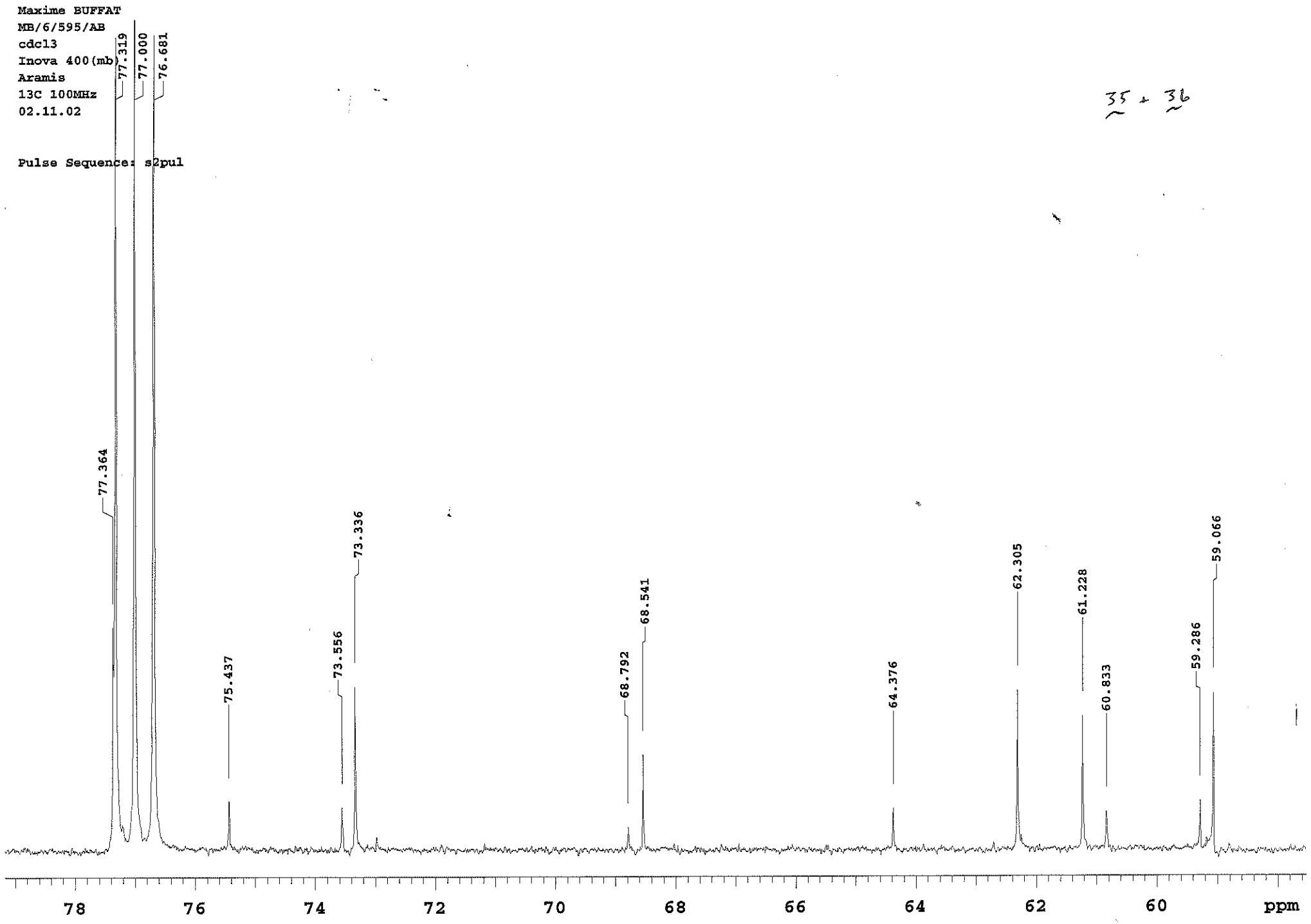


Maxime BUFFAT
MB/6/595/AB
cdcl3
Inova 400(mb)
Aramis
13C 100MHz
02.11.02

Pulse Sequence: s2pul

35 + 36

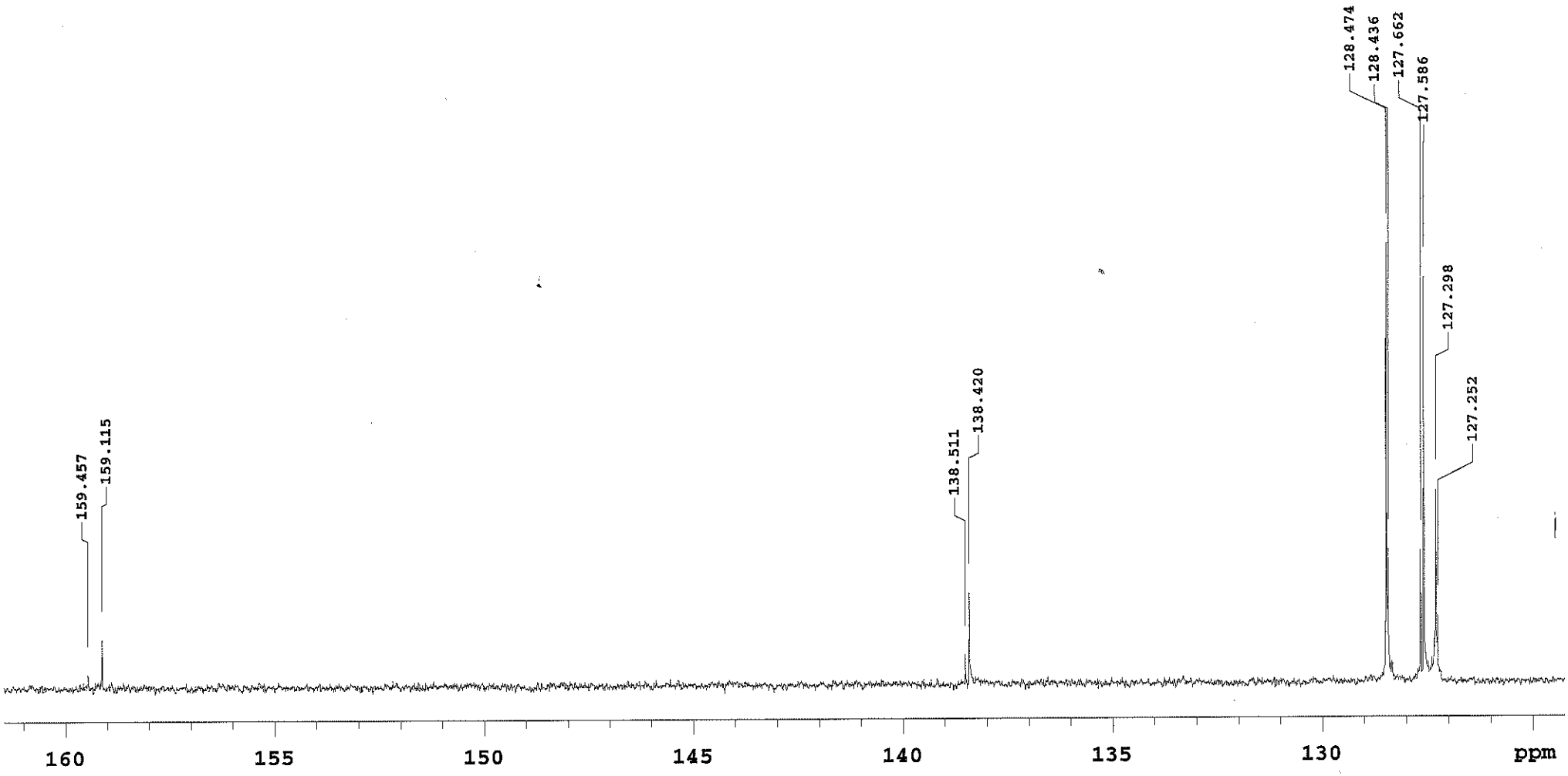



Maxime BUFFAT
MB/6/595/AB
cdcl3
Inova 400(mb)
Aramis
13C 100MHz
02.11.02
Pulse Sequence: s2pul
35 + 36
77.319
77.000
76.681
77.364
75.437
73.556
73.336
68.792
68.541
64.376
62.305
61.228
60.833
59.286
59.066
78
76
74
72
70
68
66
64
62
60
ppm

Maxime BUFFAT
MB/6/595/AB
cdcl3
Inova 400(mb)
Aramis
13C 100MHz
02.11.02

Pulse Sequence: s2pul

35 + 36

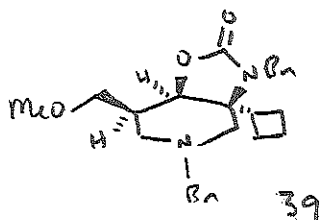


Maxime BUFFAT
MB.7.610.AB
cdcl3
Inova 400(MB)
Aramis
Proton 400MHz
23.11.02

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
User: 1-12-87
INOVA-400 "aramis"

Relax. delay 5.000 sec
Pulse 36.0 degrees
Acq. time 4.008 sec
Width 6387.7 Hz
96 repetitions
OBSERVE H1, 399.9584369 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 14 min, 25 sec



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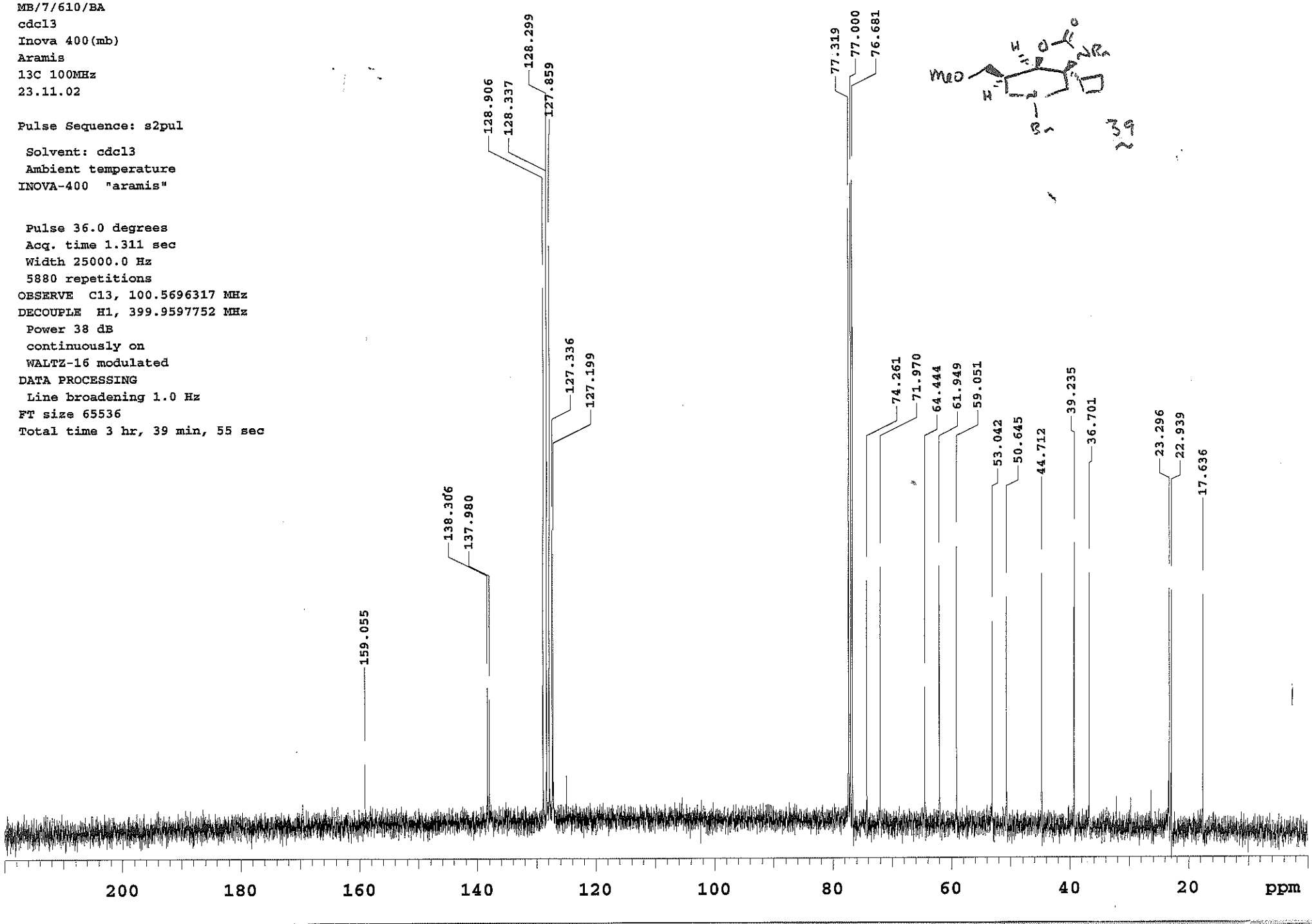
Maxime BUFFAT
MB/7/610/BA
cdcl3
Inova 400(mb)
Aramis
13C 100MHz
23.11.02

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
INOVA-400 "aramis"

Pulse 36.0 degrees
Acq. time 1.311 sec
Width 25000.0 Hz
5880 repetitions
OBSERVE C13, 100.5696317 MHz
DECOUPLE H1, 399.9597752 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 3 hr, 39 min, 55 sec

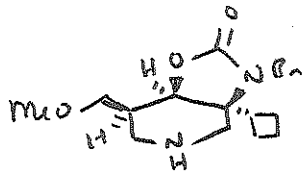


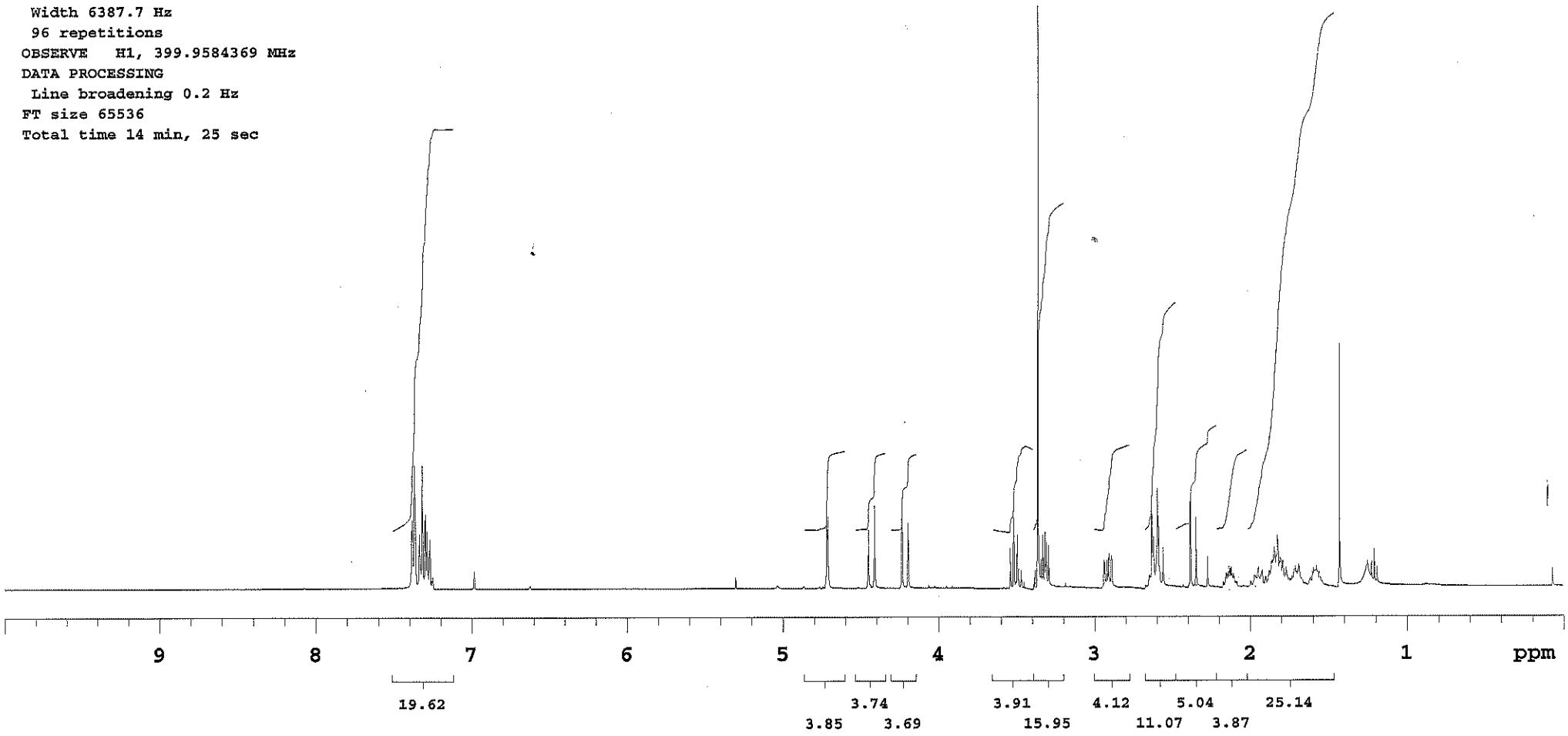
Maxime BUFFAT
MB.7.621.B
cdcl3
Inova 400(MB)
Aramis
Proton 400MHz
29.11.02

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
User: 1-12-87
INOVA-400 "aramis"

Relax. delay 5.000 sec
Pulse 36.0 degrees
Acq. time 4.008 sec
Width 6387.7 Hz
96 repetitions
OBSERVE H1, 399.9584369 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 14 min, 25 sec



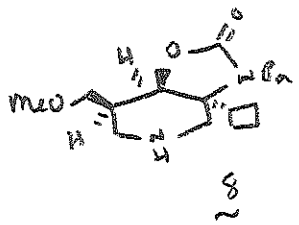


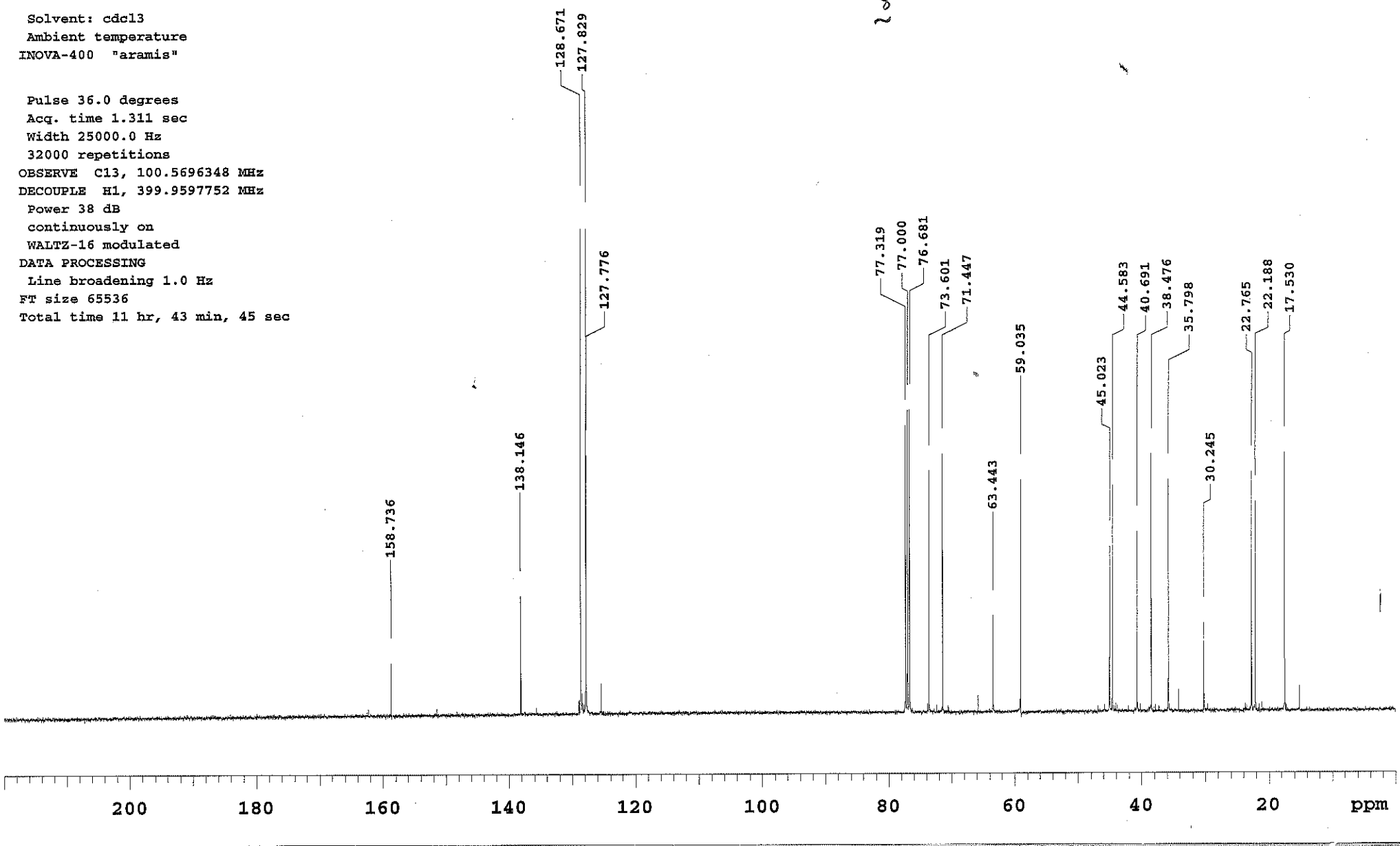
Maxime BUFFAT
MB.7.621.B
cdcl3
Inova 400(MB)
Aramis
13C 100MHz
29.11.02

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
INOVA-400 "aramis"

Pulse 36.0 degrees
Acq. time 1.311 sec
Width 25000.0 Hz
32000 repetitions
OBSERVE C13, 100.5696348 MHz
DECOUPLE H1, 399.9597752 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 11 hr, 43 min, 45 sec

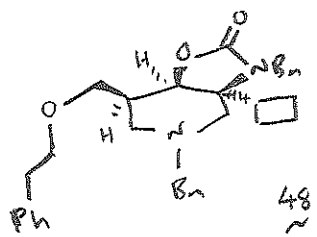




Maxime BUFFAT
MB.9.801.B
CDCl3
proton 300MHz
Inova 300
porthos
11.08.03

Solvent: cdcl3
Ambient temperature
File: MB.9.801.B-300H-11.08.03
DATE Aug 11 2003
INOVA-300 "solids"

PULSE SEQUENCE
Relax. delay 5.000 sec
Pulse 58.1 degrees
Acq. time 3.636 sec
Width 4506.0 Hz
96 repetitions
OBSERVE H1, 300.1031975 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 13 minutes

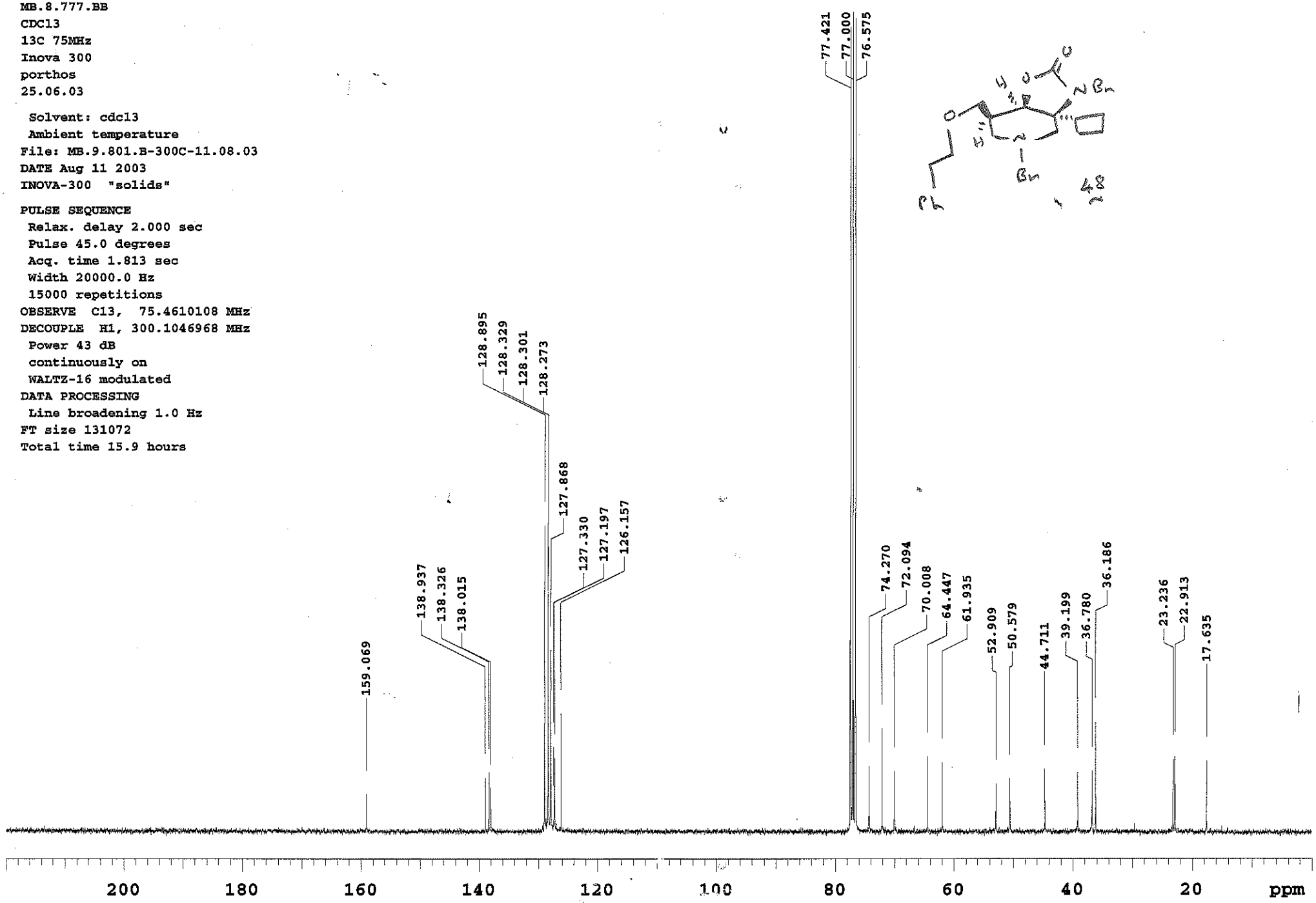


48

Maxime BUFFAT
MB.8.777.BB
CDCl3
13C 75MHz
Inova 300
porthos
25.06.03

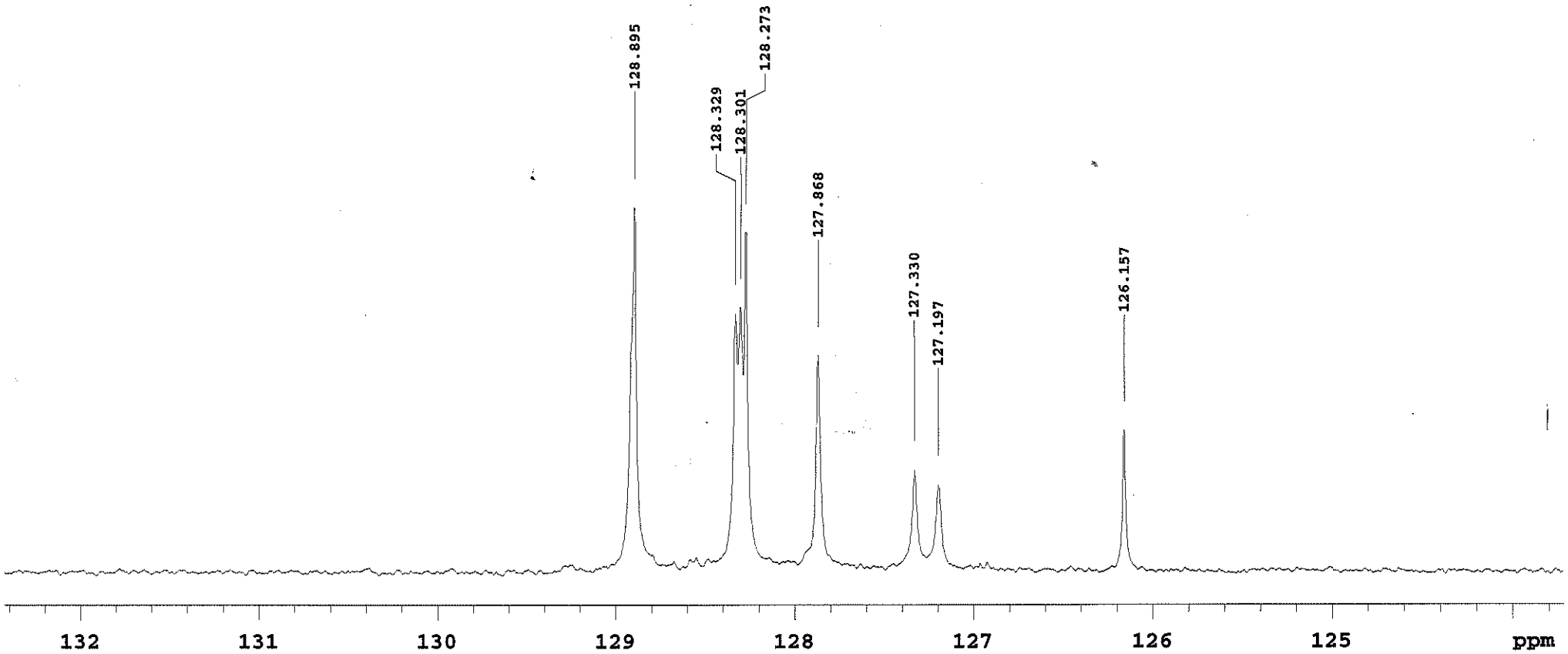
Solvent: cdcl3
Ambient temperature
File: MB.9.801.B-300C-11.08.03
DATE Aug 11 2003
INOVA-300 "solids"

PULSE SEQUENCE
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.813 sec
Width 20000.0 Hz
15000 repetitions
OBSERVE C13, 75.4610108 MHz
DECOUPLE H1, 300.1046968 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 15.9 hours



Maxime BUFFAT
MB.8.777.BB
CDCl3
13C 75MHz
Inova 300
porthos
25.06.03

48

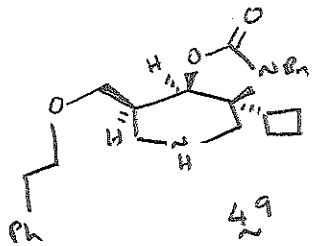


Maxime BUFFAT
MB.9.814.B
C6D6
Inova 400(MB)
Aramis
Proton 400MHz
02.09.03

Pulse Sequence: s2pul

Solvent: c6d6
Ambient temperature
User: 1-12-87
INOVA-400 "aramis"

Relax. delay 5.000 sec
Pulse 36.0 degrees
Acq. time 4.008 sec
Width 6387.7 Hz
39 repetitions
OBSERVE H1, 399.9584369 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 14 min, 25 sec

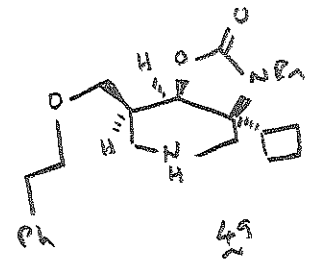


Maxime BUFFAT
MB.9.814.B
C6D6
Inova 400(MB)
Aramis
13C 100MHz
02.09.03

Pulse Sequence: s2pul

Solvent: c6d6
Ambient temperature
INOVA-400 "aramis"

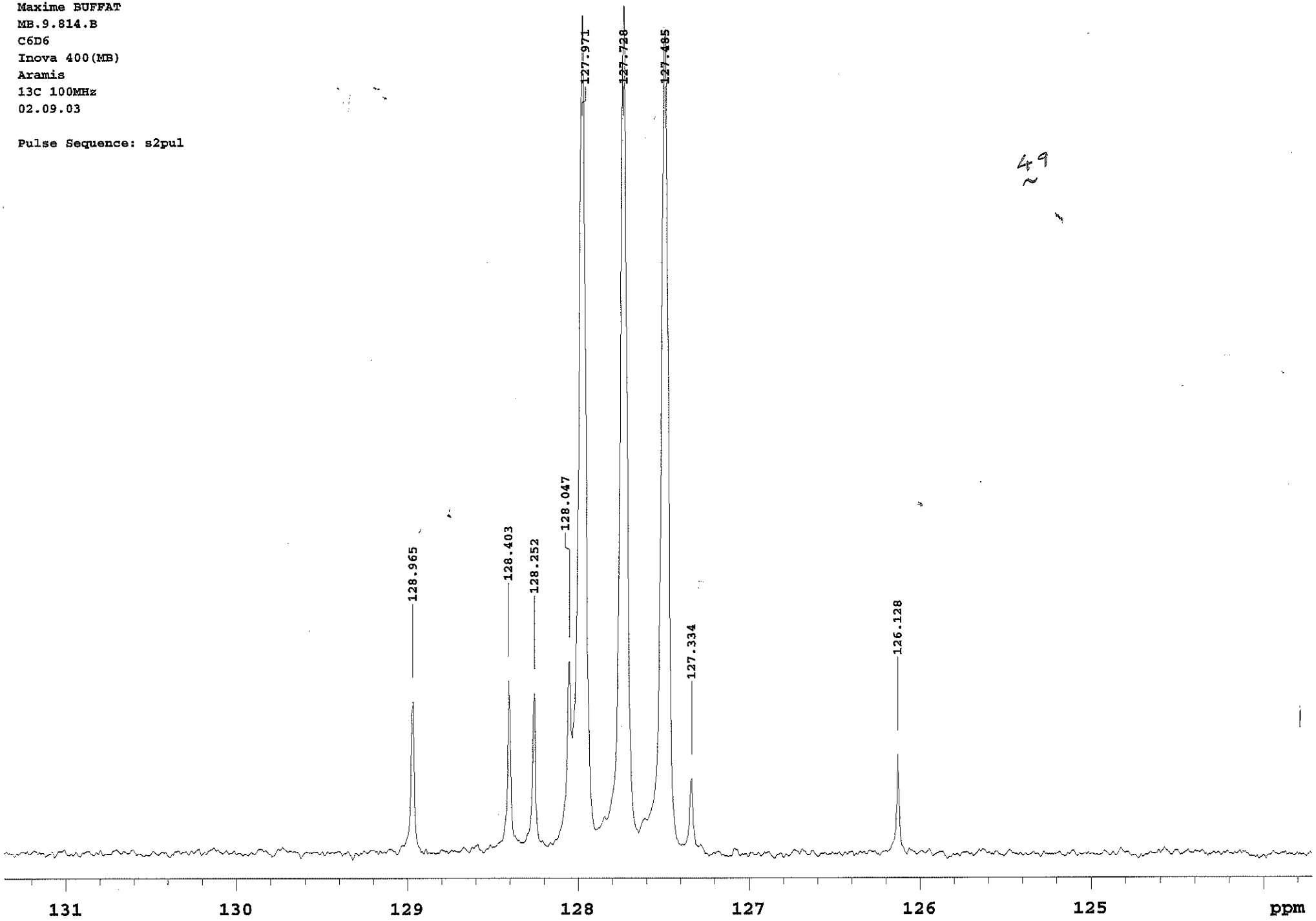
Relax. delay 2.000 sec
Pulse 36.0 degrees
Acq. time 1.311 sec
Width 25000.0 Hz
300 repetitions
OBSERVE C13, 100.5696337 MHz
DECOUPLE H1, 399.9598112 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 13 min, 15 sec



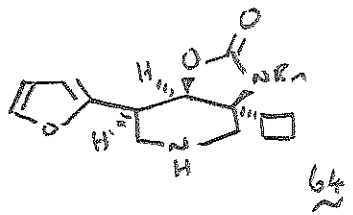
Maxime BUFFAT
MB.9.814.B
C6D6
Inova 400(MB)
Aramis
13C 100MHz
02.09.03

Pulse Sequence: s2pul

49



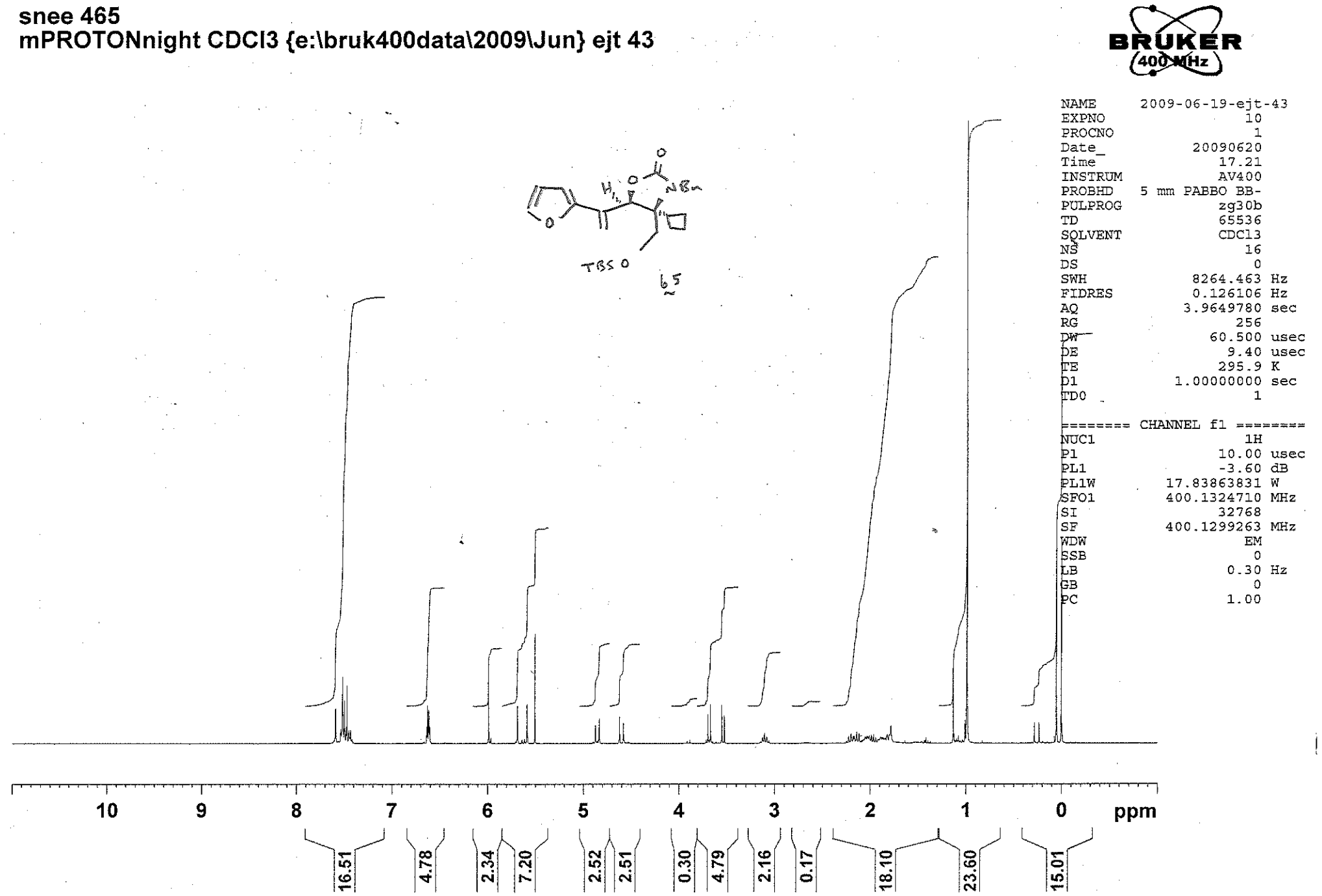
snee 461
mPROTONnight CDCl3 /opt/bruk500data/2009/Jun ejt 52



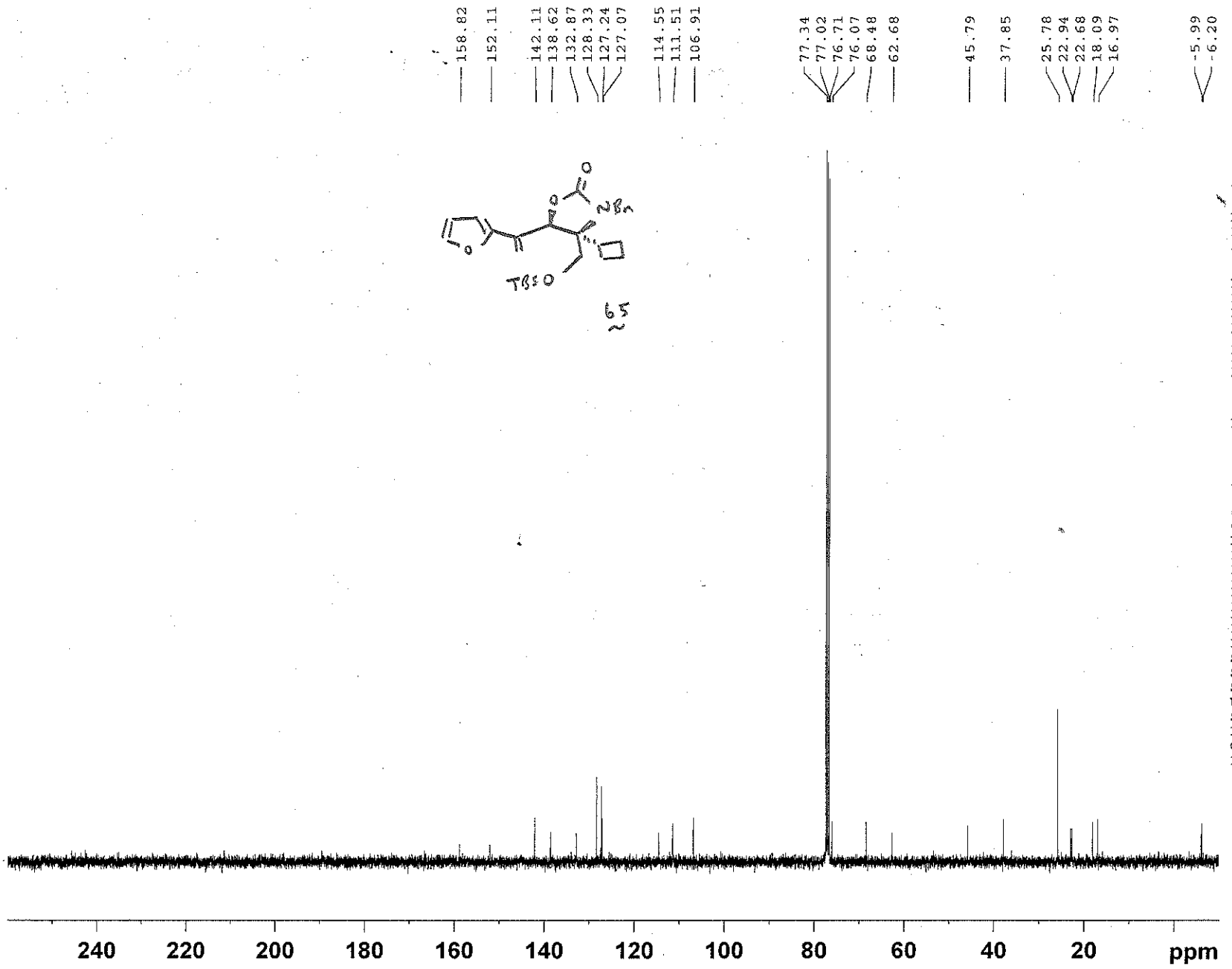
64

NAME	2009-06-02-ejt-52
EXPNO	23
PROCNO	1
Date_	20090602
Time	17.21
INSTRUM	spect
PROBHD	5 mm TXI 1H/D-
PULPROG	zg30b
TD	65536
SOLVENT	CDCl3
NS	32
DS	0
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	575
DW	48.400 usec
DE	13.38 usec
TE	291.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.20 usec
PL1	3.25 dB
PL1W	12.12272263 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300463 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

snee 465
mPROTONnight CDCl3 {e:\bruk400data\2009\Jun} ejt 43

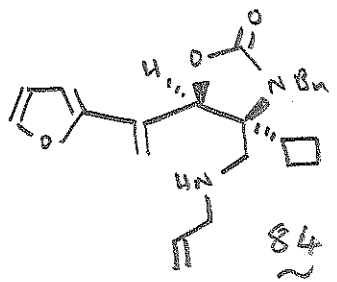


snee 465
mCARBONnight CDCl3 {e:\bruk400data\2009\Jun} ejt 43



NAME	2009-06-19-ejt-43	
EXPNO	11	
PROCNO	1	
Date_	20090620	
Time	17.35	
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	296.4	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	

snee 255
mPROTONnight CDCl3 /opt/bruk500data/2008/Oct ejt 4



NAME	2008-10-07-ejt-4
EXPNO	10
PROCNO	1
Date_	20081007
Time	18.32
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	3.1719923 sec
RG	57
DW	48.400 usec
DE	13.38 usec
TE	292.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.20 usec
PL1	3.25 dB
PL1W	12.12272263 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300474 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

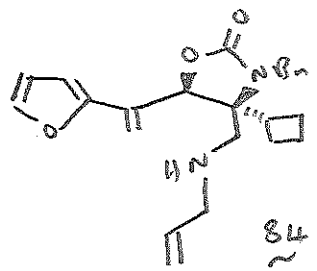
snee320
STANDARD 1H OBSERVE

Automation directory: /data/nmrdata/Aug04
File : 0102

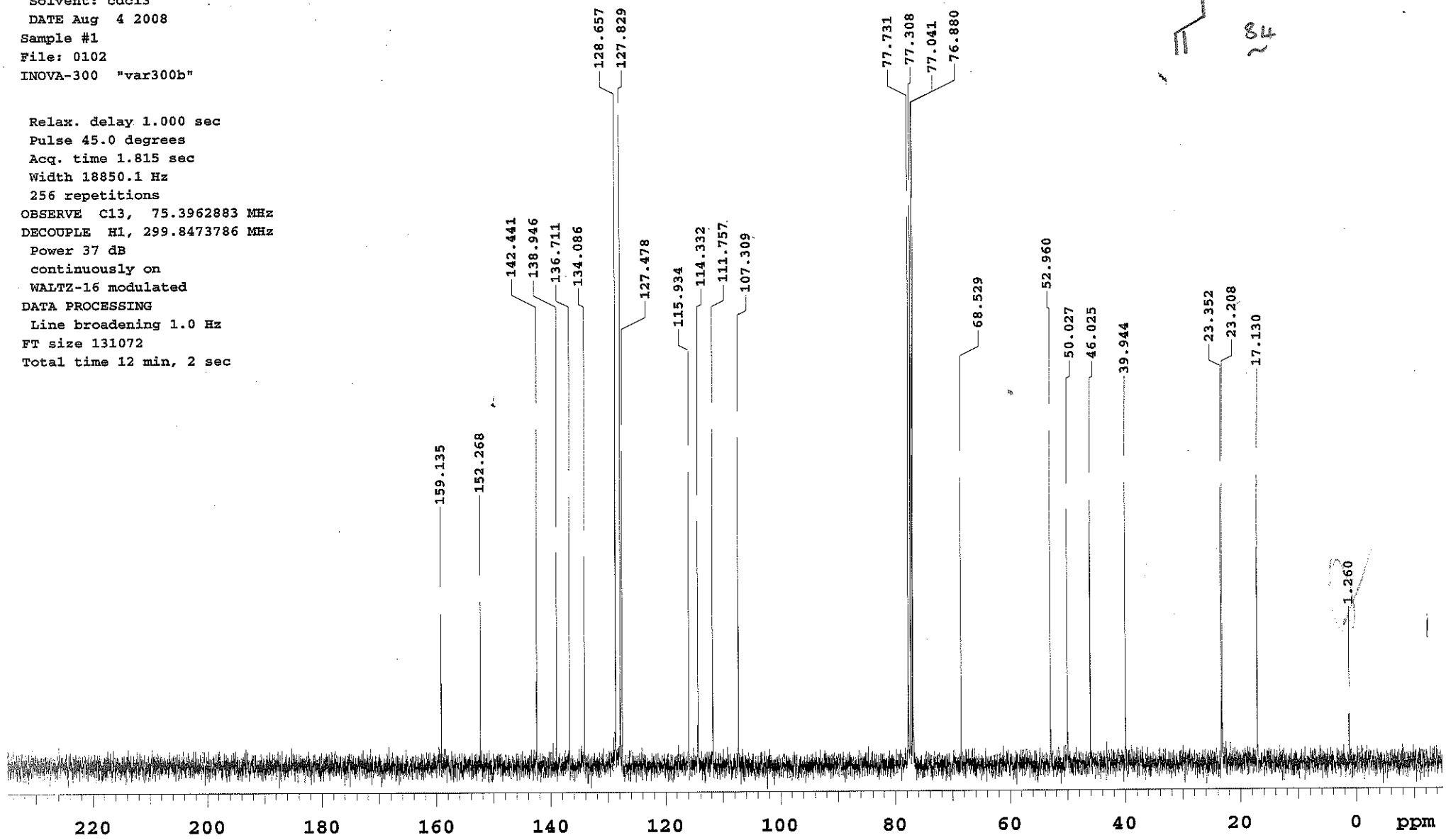
Pulse Sequence: s2pul

Solvent: cdcl3
DATE Aug 4 2008
Sample #1
File: 0102
INOVA-300 "var300b"

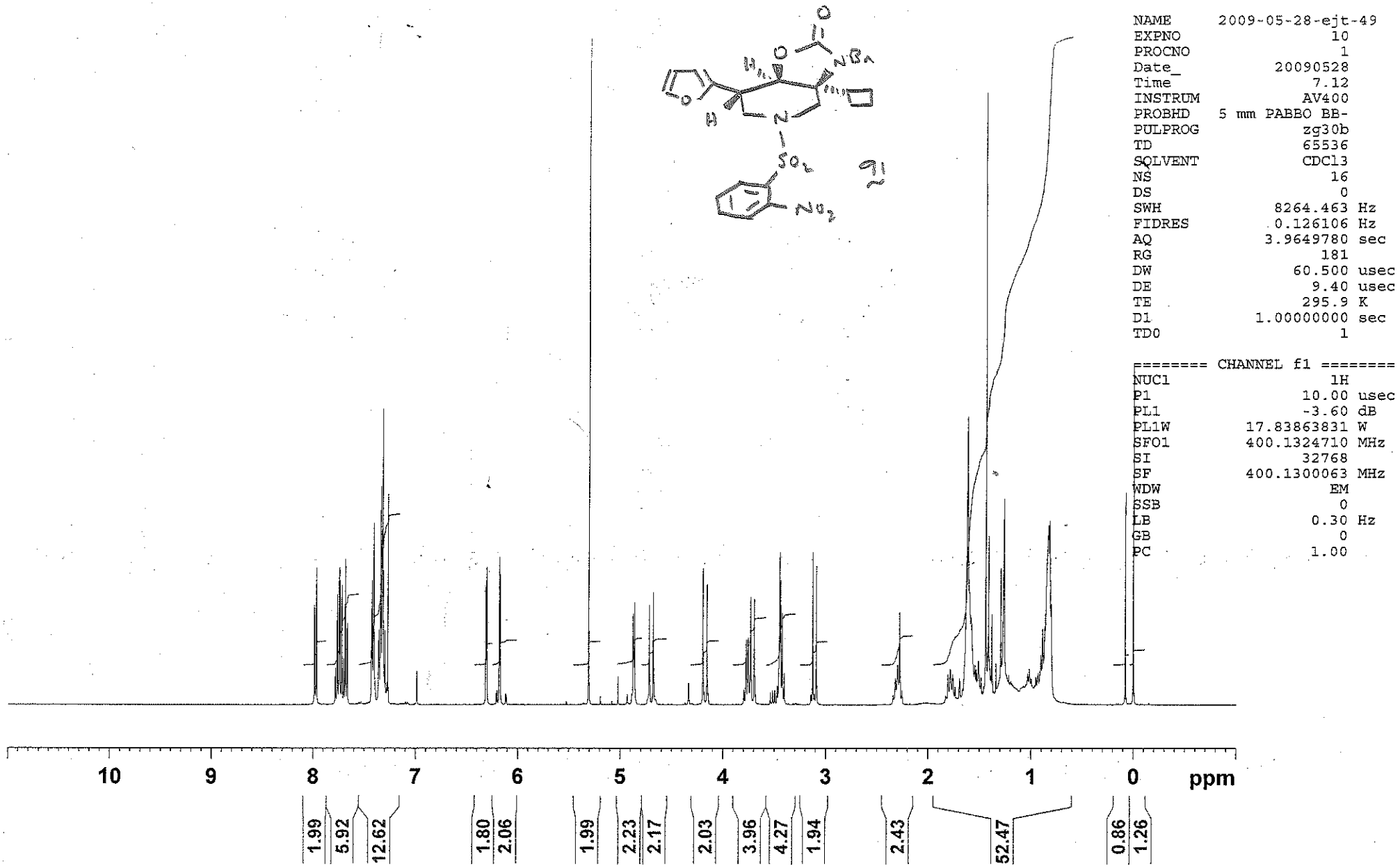
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18850.1 Hz
256 repetitions
OBSERVE C13, 75.3962883 MHz
DECOUPLE H1, 299.8473786 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 min, 2 sec



84



snee446
mPROTON CDCl3 {e:\bruk400data\2009\May} ejt 49

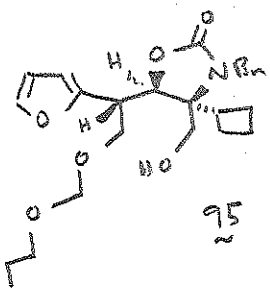


NAME	2009-05-28-ejt-49
EXPNO	10
PROCNO	1
Date_	20090528
Time	7.12
INSTRUM	AV400
PROBHD	5 mm PABBO BB-
PULPROG	zg30b
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	8264.463 Hz
FIDRES	0.126106 Hz
AQ	3.9649780 sec
RG	181
DW	60.500 usec
DE	9.40 usec
TE	295.9 K
D1	1.00000000 sec
TD0	1

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

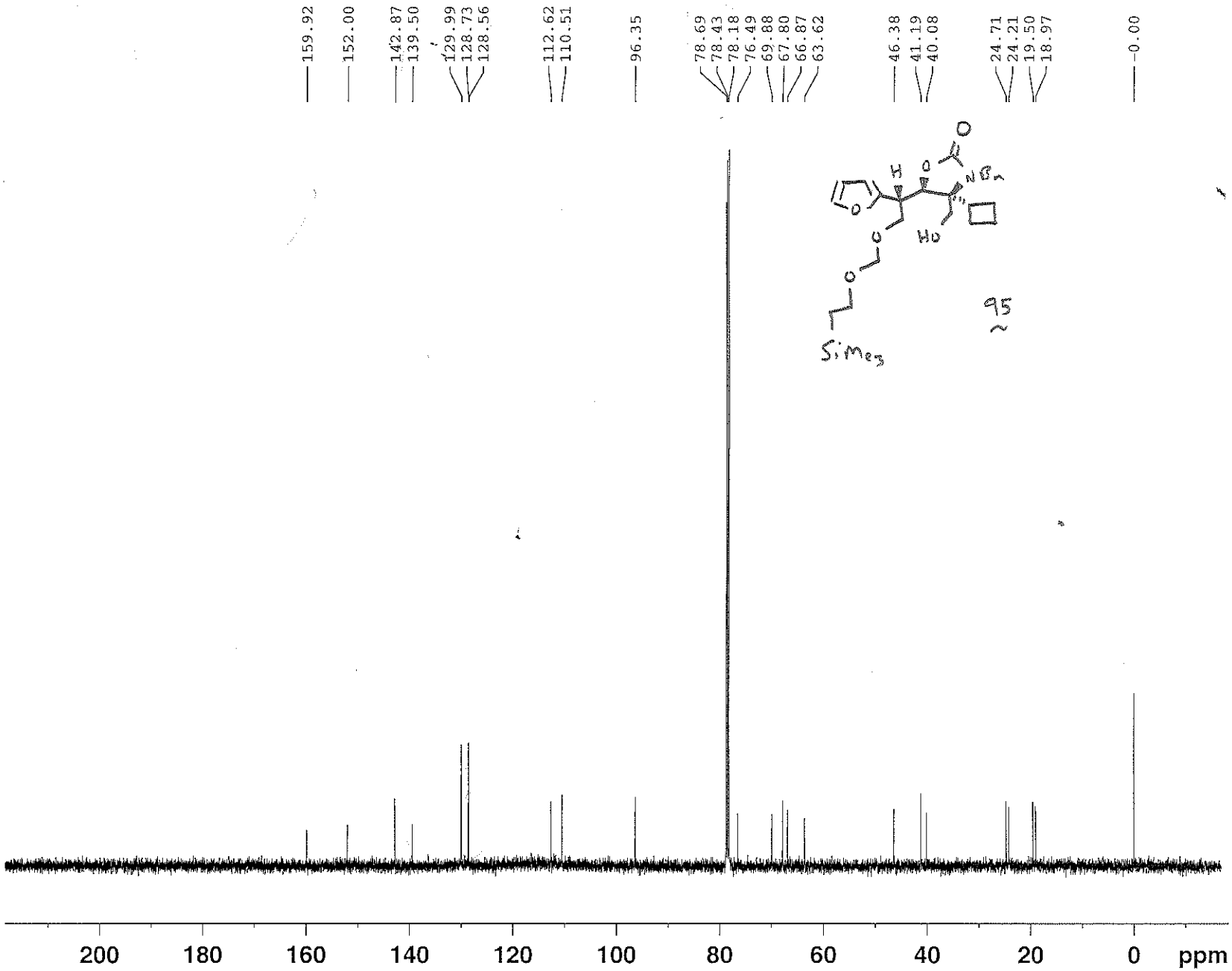
NUC1	1H
P1	10.00 usec
PL1	-3.60 dB
PL1W	17.83863831 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300063 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

snee 436 rf 0.84
mPROTONnight CDCl3 /opt/bruk500data/2009/Apr ejt 15



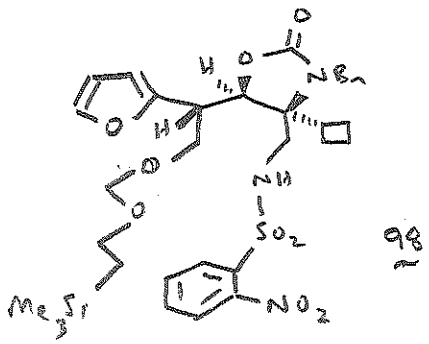
NAME	2009-05-05-ejt-15
EXPNO	10
PROCNO	1
Date_	20090505
Time	19.04
INSTRUM	spect
PROBHD	5 mm TXI 1H/D-
PULPROG	zg30b
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	161
DW	48.400 usec
DE	13.38 usec
TE	296.5 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.20 usec
PL1	3.25 dB
PL1W	12.12272263 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300210 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

snee 436 rf 0.84
mCARBONnight CDCl3 /opt/bruk500data/2009/Apr ejt 15



NAME	2009-05-05-ejt-15
EXPNO	11
PROCNO	1
Date_	20090505
Time	19.18
INSTRUM	spect
PROBHD	5 mm TXI 1H/D-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	2
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	512
DW	16.800 usec
DE	32.21 usec
TE	296.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	-4.20 dB
PL1W	218.02882385 W
SFO1	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
PL2W	9.74092484 W
PL12W	0.10223514 W
PL13W	0.12841040 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7576121 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

snee 441
mPROTONnight CDCl3 {e:\bruk400data\2009\May} ejt 54



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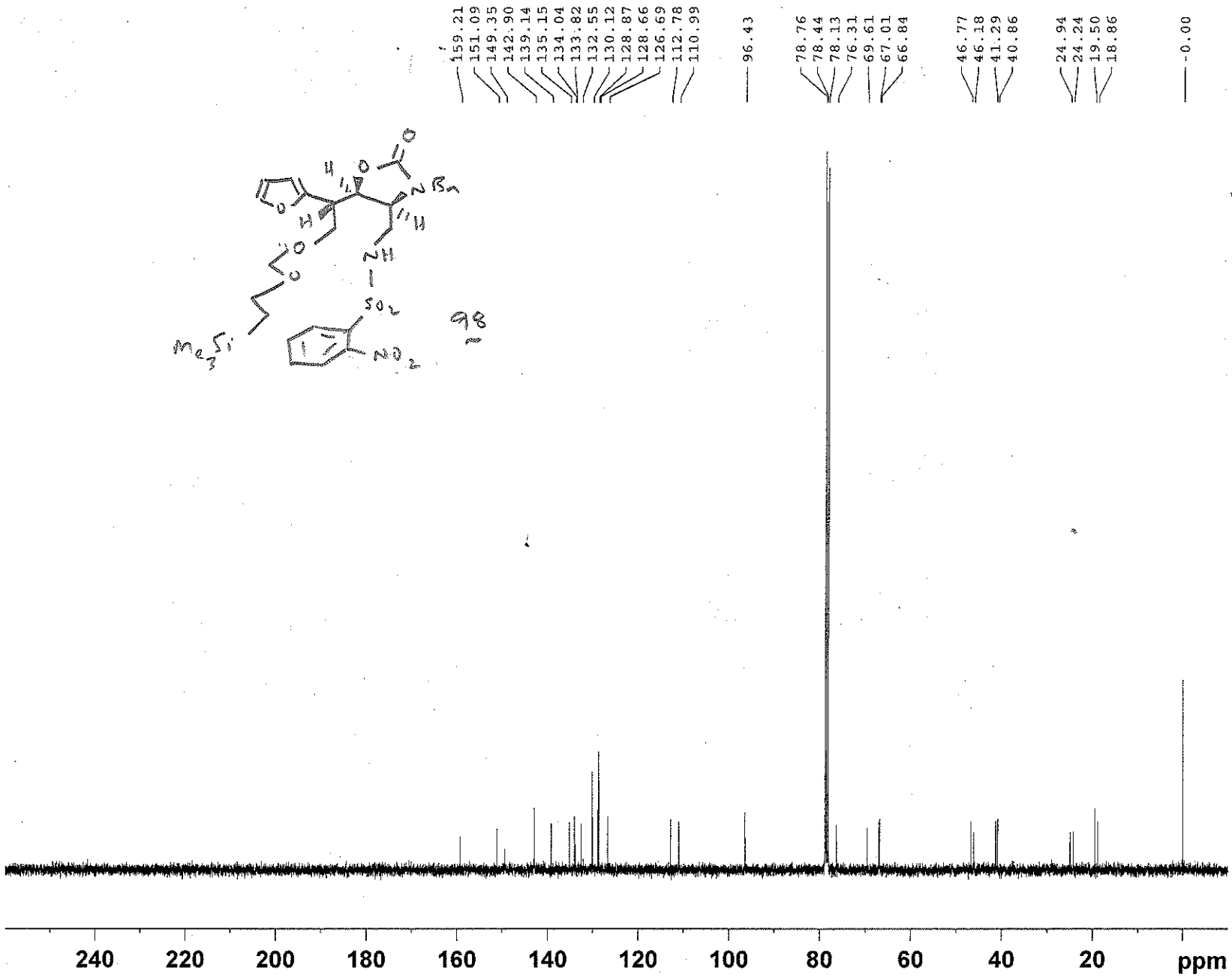
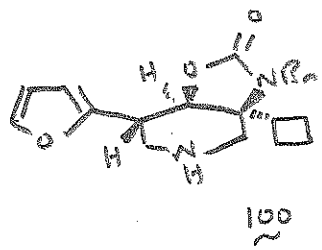
NAME	2009-05-07-ejt-54
EXPNO	10
PROCNO	1
Date_	20090508
Time	0.02
INSTRUM	AV400
PROBHD	5 mm PABBO BB-
PULPROG	zg30b
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	8264.463 Hz
FIDRES	0.126106 Hz
AQ	3.9649780 sec
RG	203
DW	60.500 usec
DE	9.40 usec
TE	295.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	10.00 usec
PL1	-3.60 dB
PL1W	17.83863831 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300181 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

snee 441
mCARBONnight CDCl3 {e:\bruk400data\2009\May} ejt 54



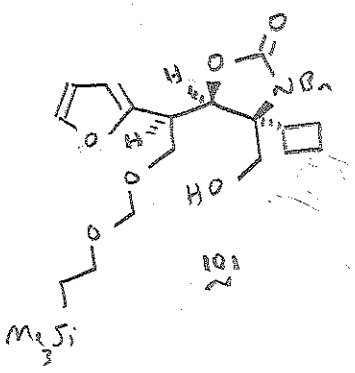
NAME	2009-05-07-ejt-54
EXPNO	11
PROCNO	1
Date_	20090508
Time	0.16
INSTRUM	AV400
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	2
SWH	30241.936 Hz
FIDRES	0.461455 Hz
AQ	1.0835786 sec
RG	512
DW	16.533 usec
DE	7.83 usec
TE	296.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== 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	100.6126270 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

snee 462 clean
mPROTONnight CDCl3 /opt/bruk500data/2009/Jun ejt 52



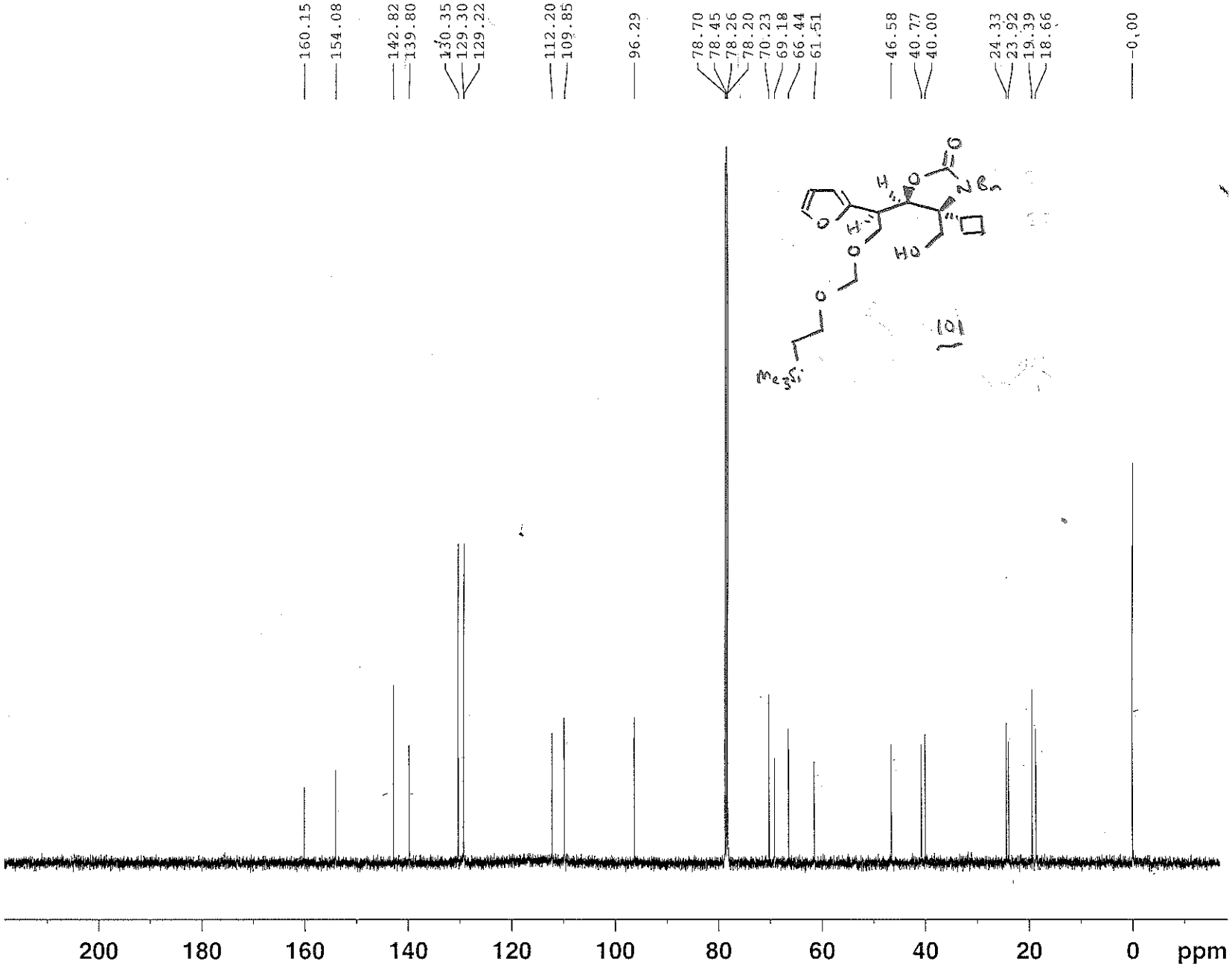
NAME	2009-06-08-ejt-52
EXPNO	10
PROCNO	1
Date_	20090608
Time	18.45
INSTRUM	spect
PROBHD	5 mm TXI 1H/D-
PULPROG	zg30b
TD	65536
SOLVENT	CDCl3
NS	64
DS	0
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	724
DW	48.400 usec
DE	13.38 usec
TE	294.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.20 usec
PL1	3.25 dB
PL1W	12.12272263 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300470 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

snee 436 rf 0.53
mPROTONnight CDCl3 /opt/bruk500data/2009/Apr ejt 17



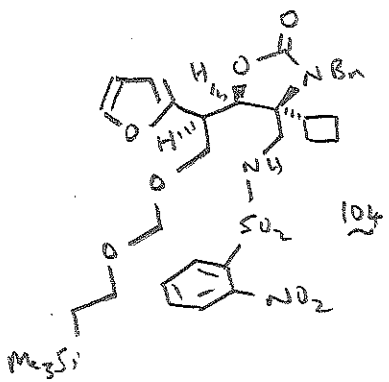
NAME	2009-05-05-ejt-17
EXPNO	10
PROCNO	1
Date_	20090505
Time	19.55
INSTRUM	spect
PROBHD	5 mm TXI 1H/D-
PULPROG	zg30b
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	90.5
DW	48.400 usec
DE	13.38 usec
TE	296.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.20 usec
PL1	3.25 dB
PL1W	12.12272263 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300056 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

snee 436 rf 0.53
mCARBONnight CDCl3 /opt/bruk500data/2009/Apr ejt 17



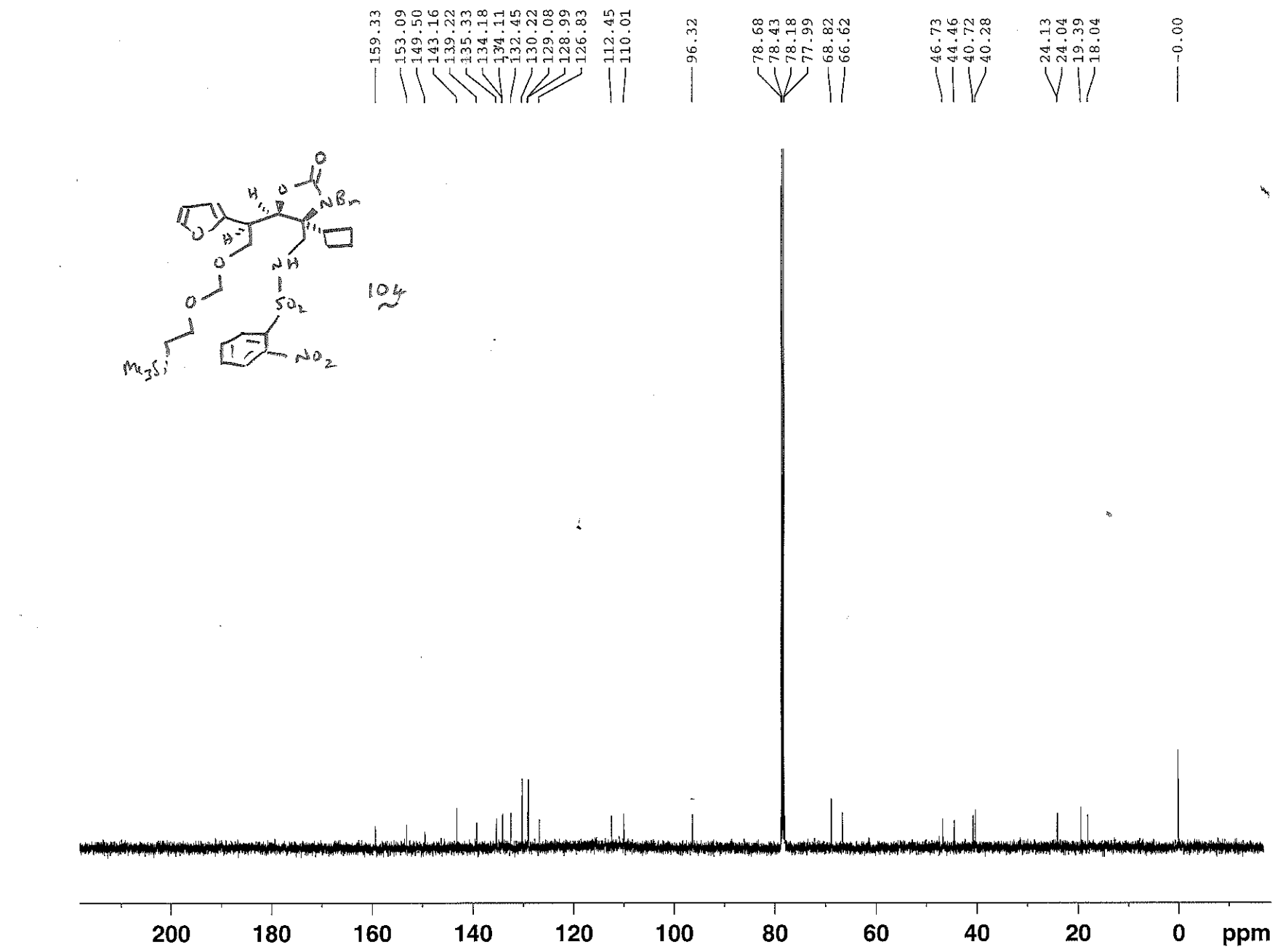
NAME	2009-05-05-ejt-17
EXPNO	11
PROCNO	1
Date_	20090505
Time	20.09
INSTRUM	spect
PROBHD	5 mm TXI 1H/D-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	2
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	512
DW	16.800 usec
DE	32.21 usec
TE	296.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	-4.20 dB
PL1W	218.02882385 W
SFO1	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
PL2W	9.74092484 W
PL12W	0.10223514 W
PL13W	0.12841040 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7576125 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

snee 485
mPROTONnight CDCl3 /opt/bruk500data/2009/Jul ejt 17



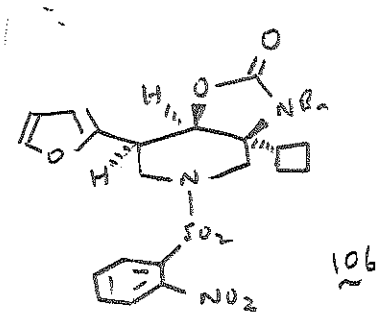
NAME	2009-07-20-ejt-17
EXPNO	10
PROCNO	1
Date_	20090720
Time	18.47
INSTRUM	spect
PROBHD	5 mm TXI 1H/D-
PULPROG	zg30b
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	203
DW	48.400 usec
DE	13.38 usec
TE	294.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.20 usec
PL1	3.25 dB
PL1W	12.12272263 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300100 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

snee 485
mCARBONnight CDCl3 /opt/bruk500data/2009/Jul ejt 17



NAME	2009-07-20-ejt-17
EXPNO	12
PROCNO	1
Date_	20090720
Time	19.09
INSTRUM	spect
PROBHD	5 mm TXI 1H/D-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	2
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	512
DW	16.800 usec
DE	32.21 usec
TE	294.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	-4.20 dB
PL1W	218.02882385 W
SFO1	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
PL2W	9.74092484 W
PL12W	0.10223514 W
PL13W	0.12841040 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7576135 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

snee 460
mPROTONnight CDCl3 /opt/bruk500data/2009/Jun ejt 56



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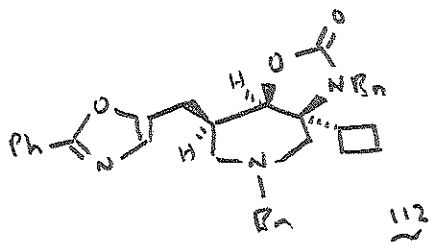
NAME	2009-06-01-ejt-56
EXPNO	10
PROCNO	1
Date_	20090601
Time	17.05
INSTRUM	spect
PROBHD	5 mm TXI 1H/D-
PULPROG	zg30b
TD	65536
SOLVENT	CDCl3
NS	32
DS	0
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	256
DW	48.400 usec
DE	13.38 usec
TE	291.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.20 usec
PL1	3.25 dB
PL1W	12.12272263 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300122 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Maxime BUFFAT
MB.8.775.BB
CDCl3
proton 300MHz
Inova 300
porthos
25.06.03

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
File: MB.8.777.BB-300H-25.06.03
INOVA-300 "porthos"

Relax. delay 5.000 sec
Pulse 58.1 degrees
Acq. time 3.636 sec
Width 4506.0 Hz
96 repetitions
OBSERVE H1, 300.1031975 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 13 min, 49 sec



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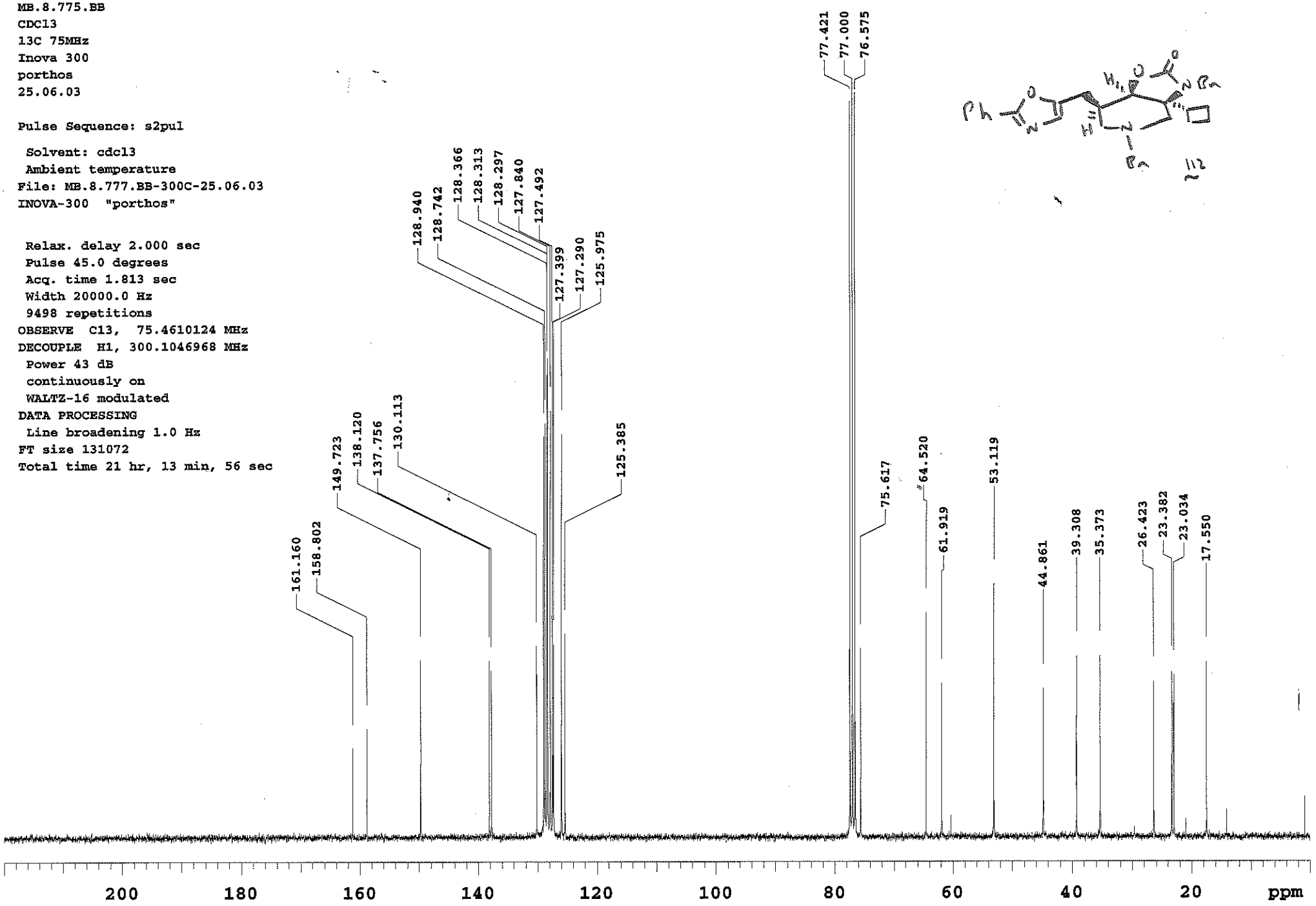
Maxime BUFFAT
MB.8.775.BB
CDCl3
13C 75MHz
Inova 300
porthos
25.06.03

Pulse Sequence: s2pul

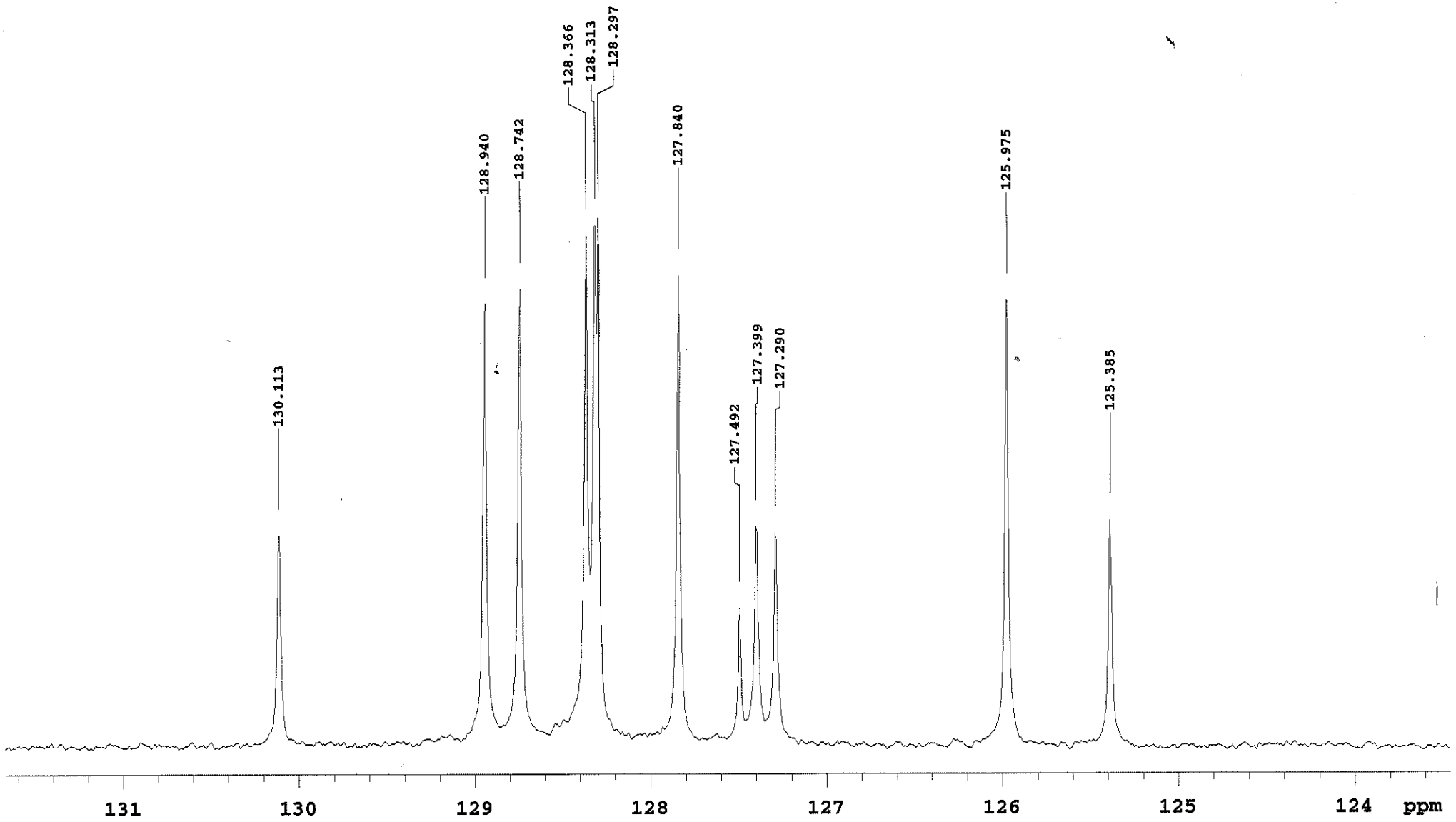
Solvent: cdcl3
Ambient temperature
File: MB.8.777.BB-300C-25.06.03
INOVA-300 "porthos"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.813 sec
Width 20000.0 Hz
9498 repetitions
OBSERVE C13, 75.4610124 MHz
DECOUPLE H1, 300.1046968 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 21 hr, 13 min, 56 sec



Maxime BUFFAT
MB.8.775.BB
CDCl3
13C 75MHz
Inova 300
porthos
25.06.03

Pulse Sequence: s2pul

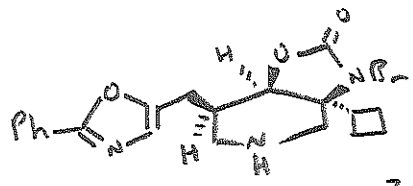


Maxime BUFFAT
MB.8.777.D
cdcl3
Inova 400(MB)
Aramis
Proton 400MHz
28.06.03

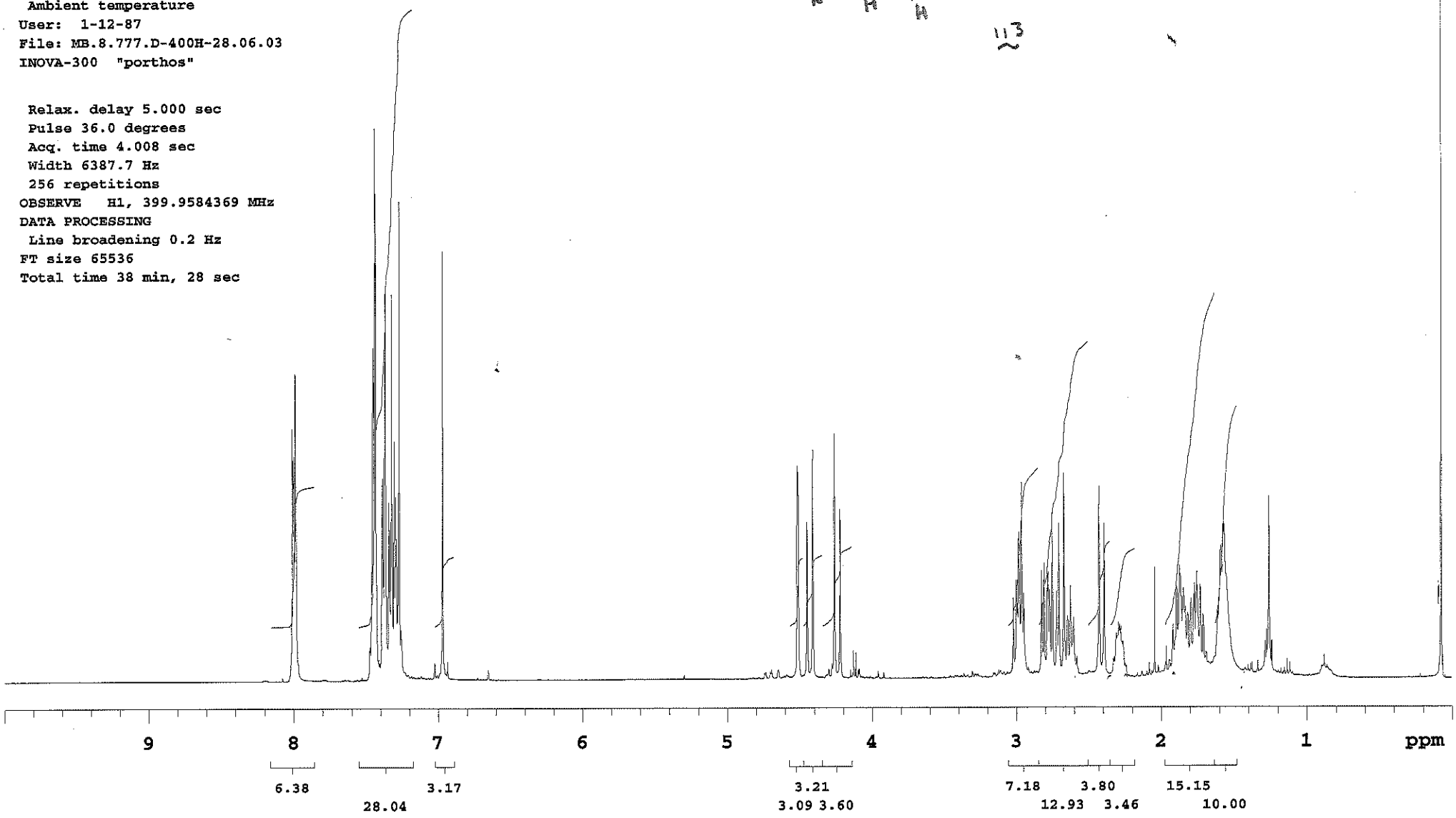
Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
User: 1-12-87
File: MB.8.777.D-400H-28.06.03
INOVA-300 "porthos"

Relax. delay 5.000 sec
Pulse 36.0 degrees
Acq. time 4.008 sec
Width 6387.7 Hz
256 repetitions
OBSERVE H1, 399.9584369 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 38 min, 28 sec



113

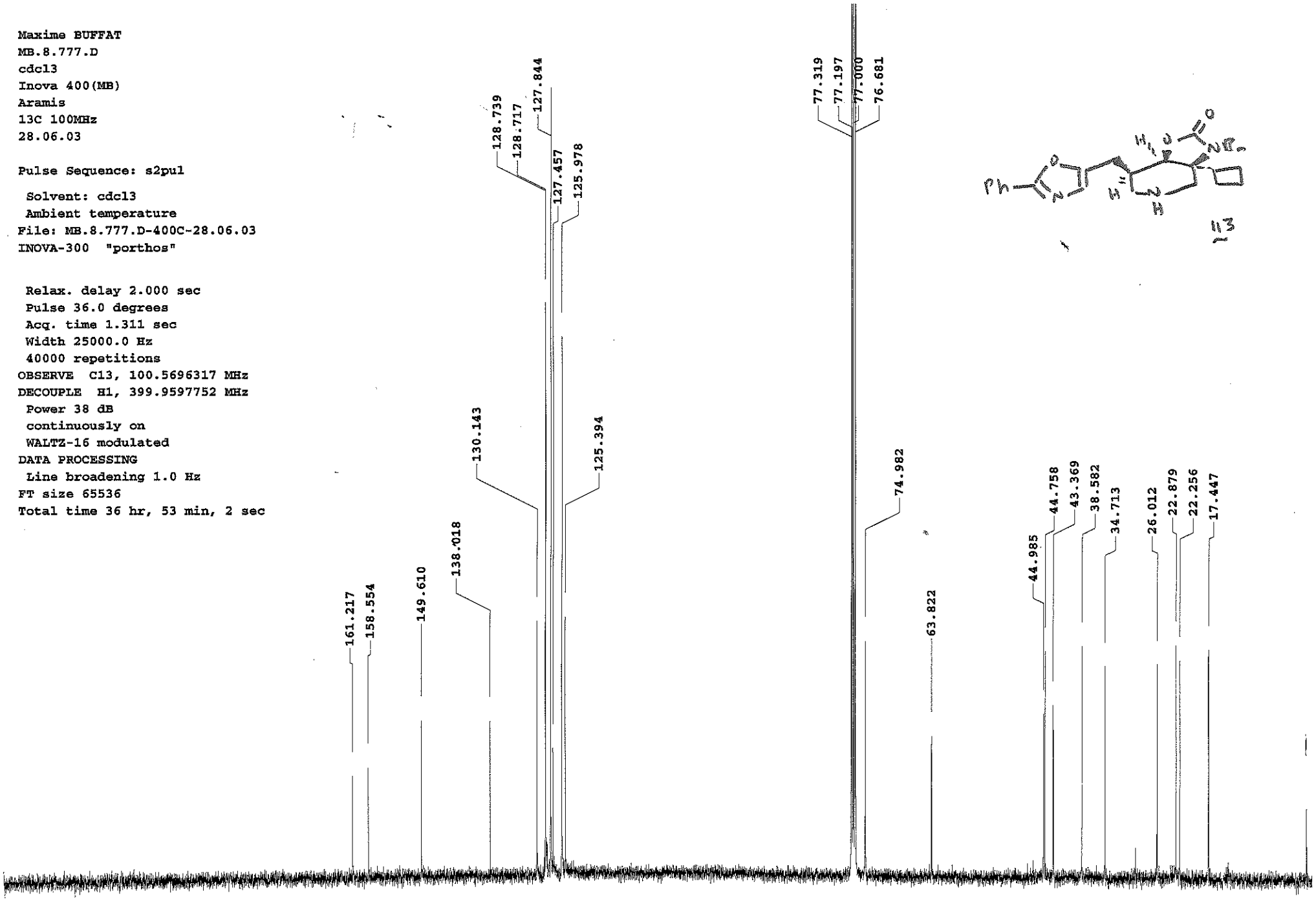


Maxime BUFFAT
MB.8.777.D
cdcl3
Inova 400(MB)
Aramis
13C 100MHz
28.06.03

Pulse Sequence: s2pul

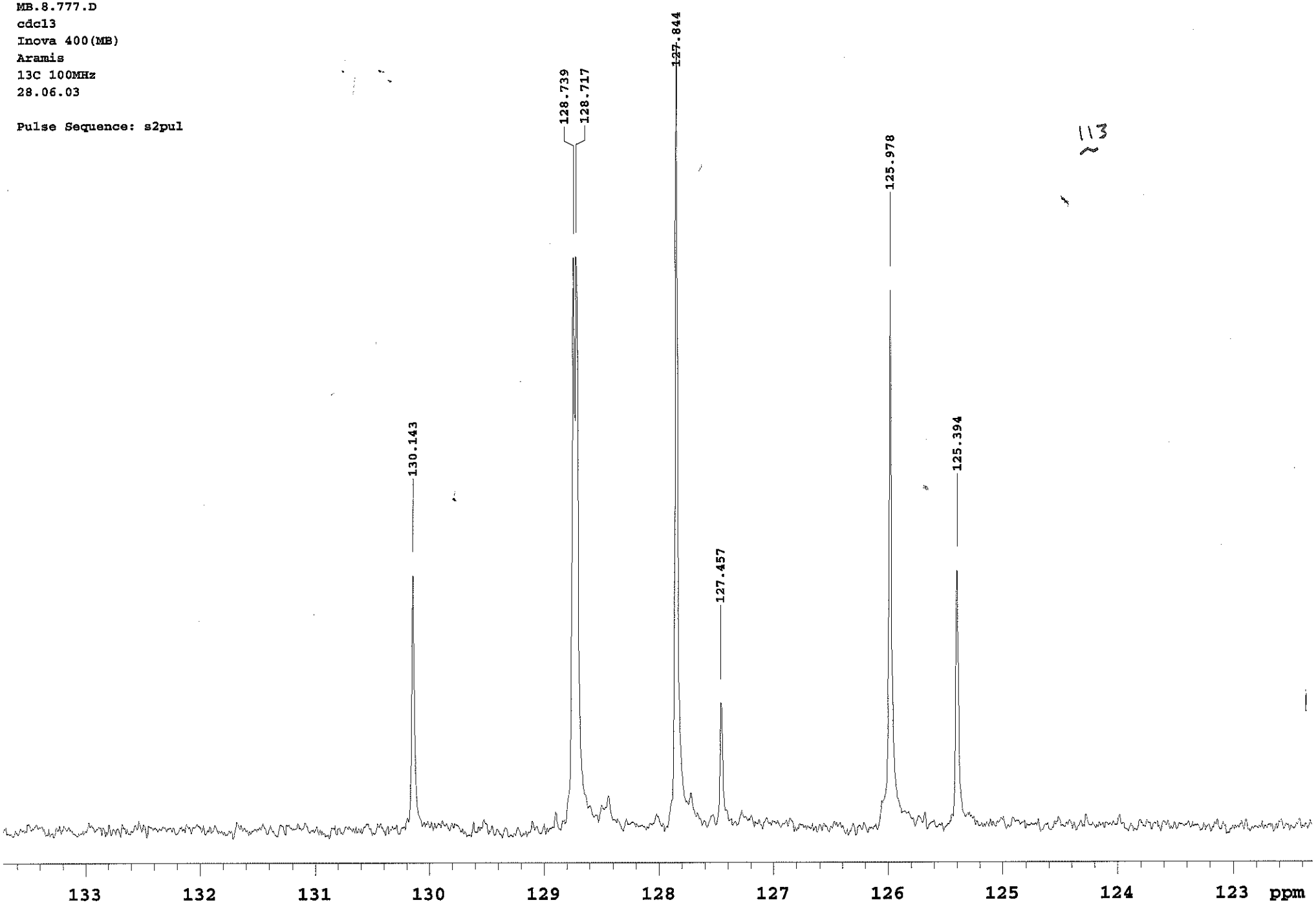
Solvent: cdcl3
Ambient temperature
File: MB.8.777.D-400C-28.06.03
INOVA-300 "porthos"

Relax. delay 2.000 sec
Pulse 36.0 degrees
Acq. time 1.311 sec
Width 25000.0 Hz
40000 repetitions
OBSERVE C13, 100.5696317 MHz
DECOUPLE H1, 399.9597752 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 36 hr, 53 min, 2 sec



Maxime BUFFAT
MB.8.777.D
cdcl3
Inova 400(MB)
Aramis
13C 100MHz
28.06.03

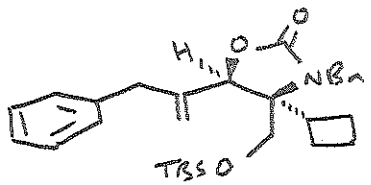
Pulse Sequence: s2pul



113

clear oil

Eb suzuki
mPROTON CDCl3 {e:\bruk400data\2012\Oct} ejt 46



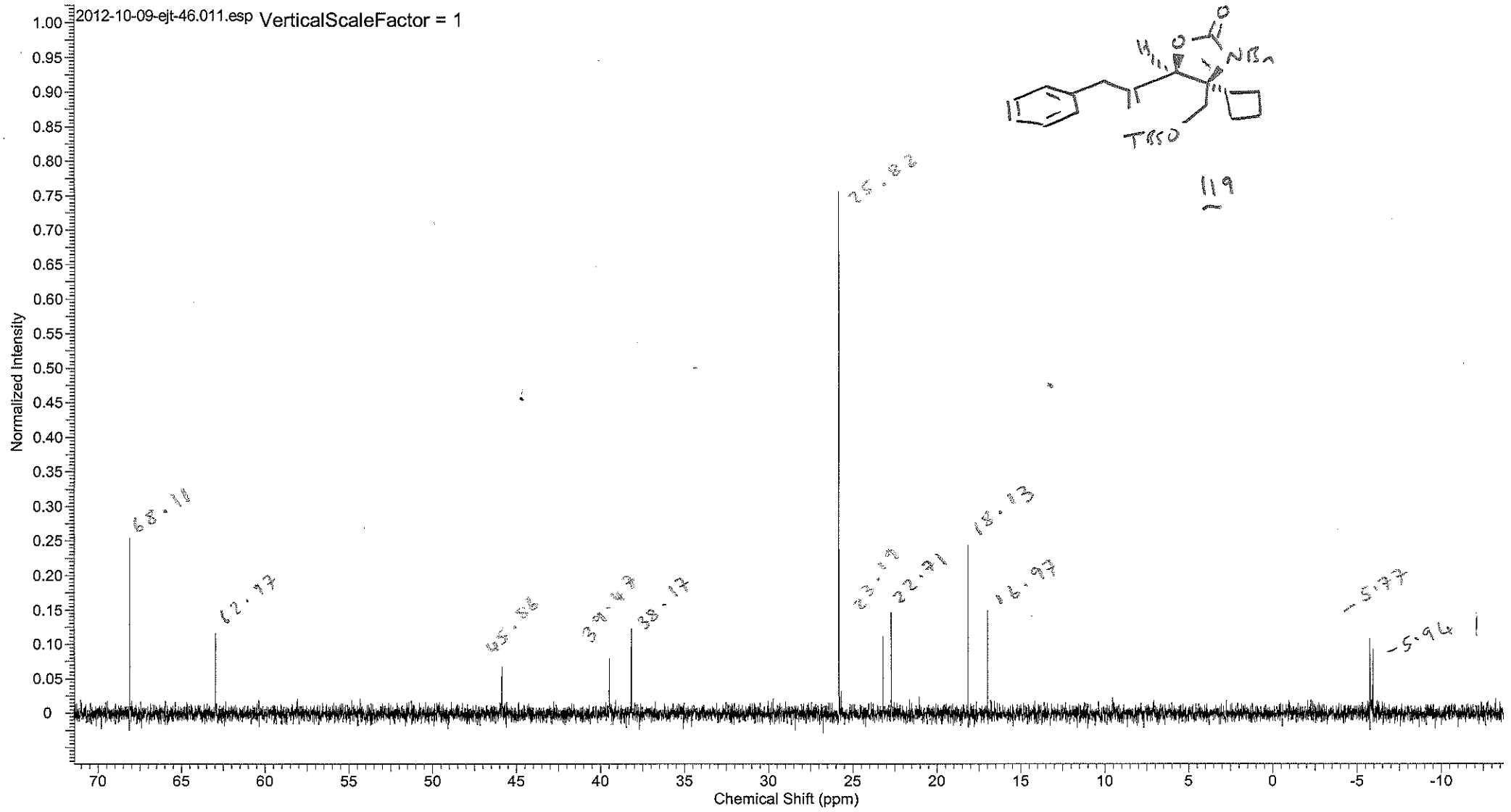
119

NAME	2012-10-09-ejt-46
EXPNO	10
PROCNO	1
Date_	20121009
Time	16.26
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	128
DW	60.500 usec
DE	9.40 usec
TE	294.1 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	400.1300104 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

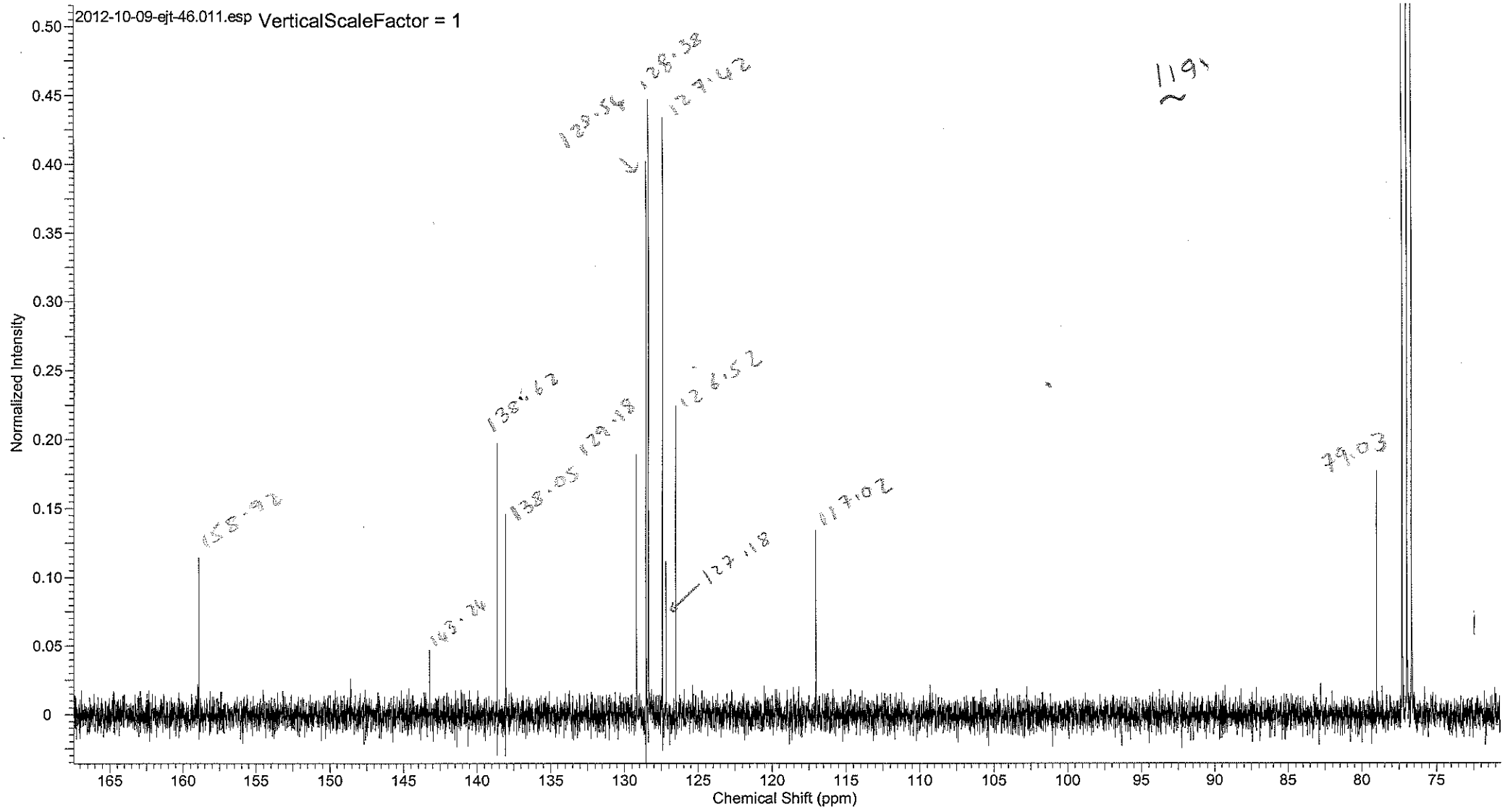
24/10/2012 17:15:01

Acquisition Time (sec)	1.0835	***Comment***	Eb suzuki mCARBON CDCl3 {e:\bruk400data\2012\Oct} ejt 46			***Date***	09 Oct 2012 16:40:48
Date Stamp	09 Oct 2012 16:40:48						
File Name	\\ss7a.ds.man.ac.uk\vol5\vol3\users\snmrdata\bruk400data\2012\Oct\data\ejt\nmr\2012-10-09-ejt-46\11\fid					***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	256	***Origin***	AV400	***Original Points Count***	32768
Owner	Administrator	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	512.00
SW(cyclical) (Hz)	30241.94	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11329.4600	***Spectrum Type***	STANDARD
Sweep Width (Hz)	30241.01	***Temperature (degree C)***	21.900				



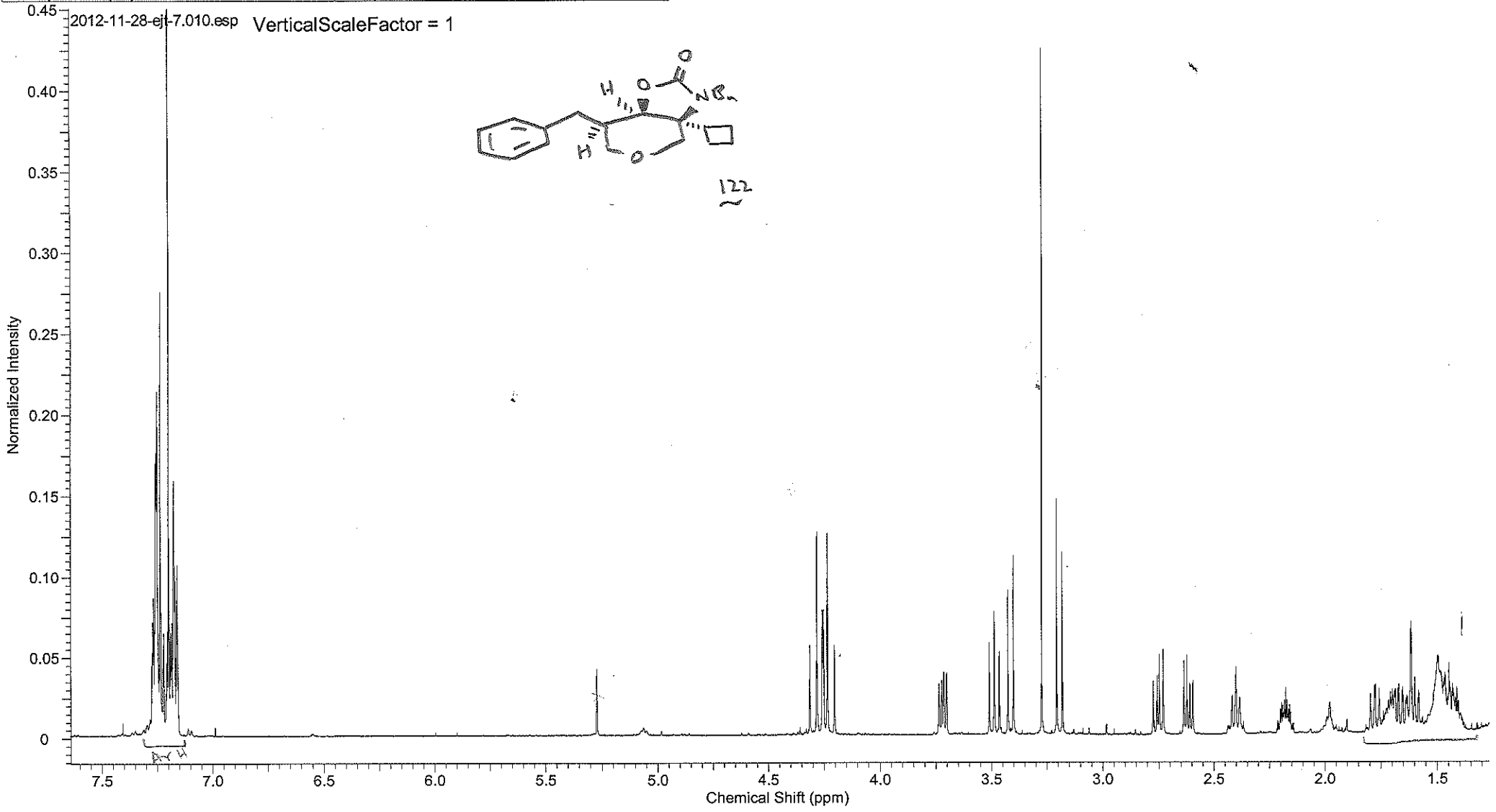
24/10/2012 17:16:16

Acquisition Time (sec)	1.0835	***Comment***	Eb suzuki mCARBON CDCl3 {e:\bruk400data\2012\Oct} ejt 46			***Date***	09 Oct 2012 16:40:48
Date Stamp	09 Oct 2012 16:40:48						
File Name	\\ss7a.ds.man.ac.uk\vol5\vol3\users\snmrdata\bruk400data\2012\Oct\data\ejt\nmr\2012-10-09-ejt-46\11\fid					***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	256	***Origin***	AV400	***Original Points Count***	32768
Owner	Administrator	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	512.00
SW(cyclical) (Hz)	30241.94	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11329.4600	***Spectrum Type***	STANDARD
Sweep Width (Hz)	30241.01	***Temperature (degree C)***	21.900				



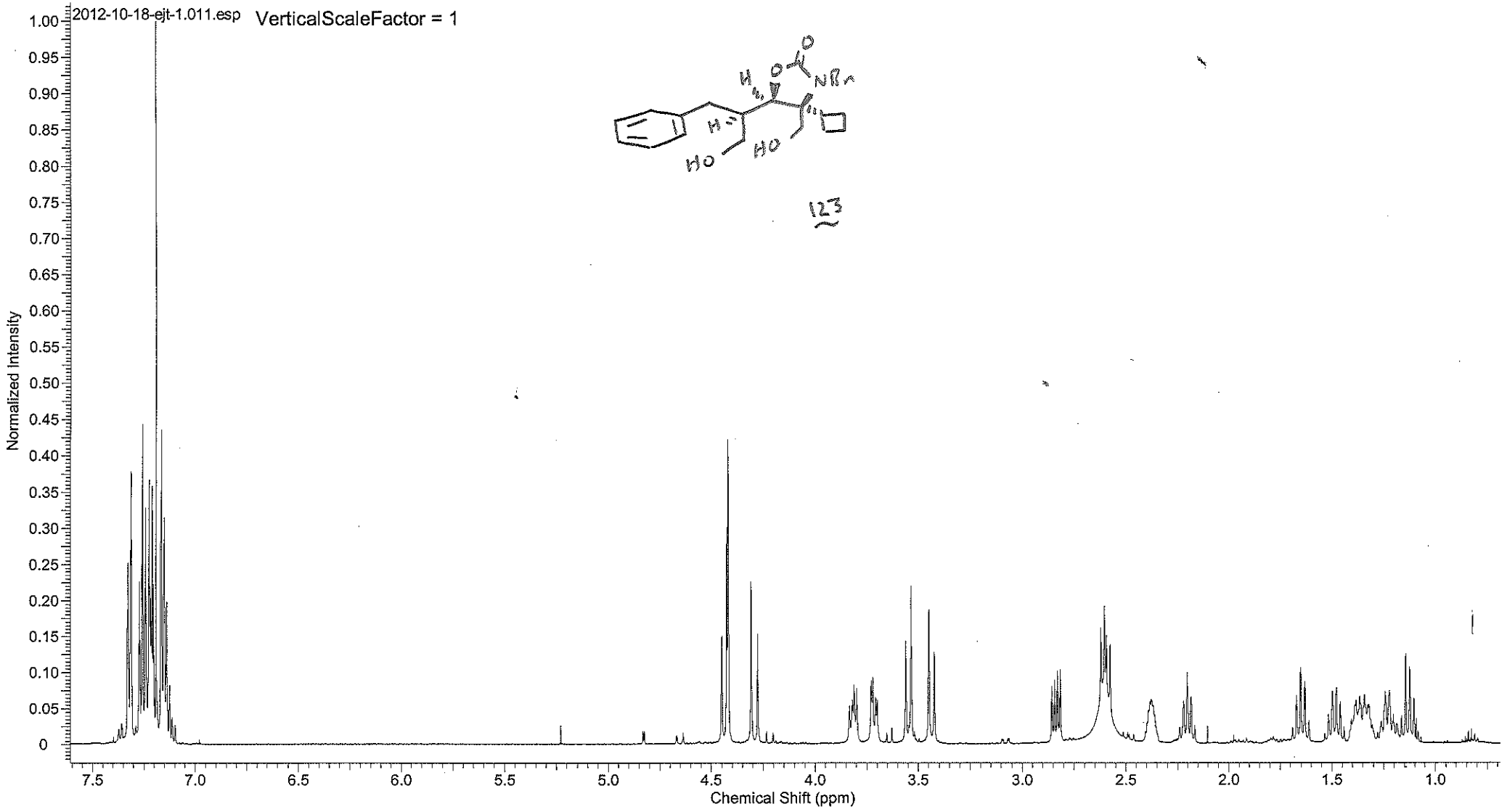
28/11/2012 12:47:27

Acquisition Time (sec)	3.1719	***Comment***	EB m1 ether mPROTON CDCl3 /opt/bruk500data/2012/Nov ejt 7			***Date***	28 Nov 2012 09:27:28
Date Stamp	28 Nov 2012 09:27:28						
File Name	\\ss7a.ds.man.ac.uk\vol5\vol3\users\snmrdata\bruk500data\bruk500data\2012\Nov\data\ejt\nmr\2012-11-28-ejt-7\10\fid					***Frequency (MHz)***	500.13
Nucleus	1H	***Number of Transients***	50	***Origin***	spect	***Original Points Count***	32768
Owner	vnmr1	***Points Count***	32768	***Pulse Sequence***	zg30b	***Receiver Gain***	256.00
SW(cyclical) (Hz)	10330.58	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	3044.7351	***Spectrum Type***	STANDARD
Sweep Width (Hz)	10330.26	***Temperature (degree C)***	25.029				

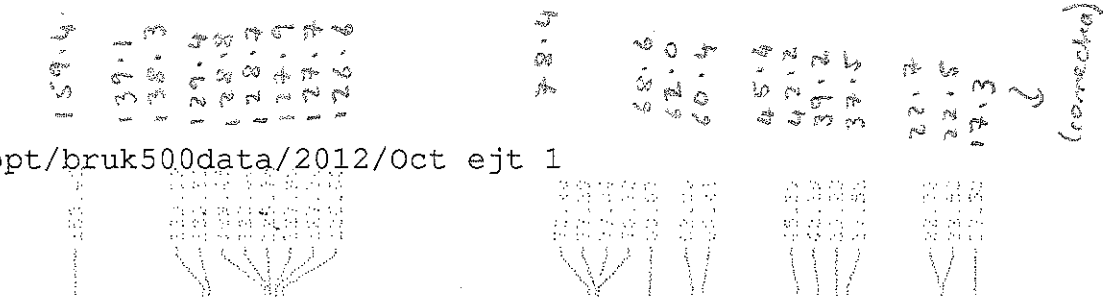


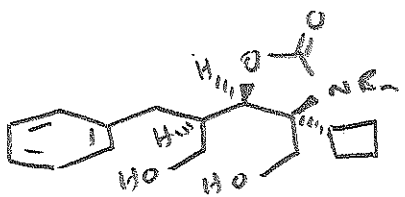
18/10/2012 17:13:23

Acquisition Time (sec)	3.1719	***Comment***	EB TBS deprot mPROTON CDCl3 /opt/bruk500data/2012/Oct ejt 1			***Date***	18 Oct 2012 09:42:40
Date Stamp	18 Oct 2012 09:42:40						
File Name	\\ss7a.ds.man.ac.uk\vol5\vol3\users\snmrdata\bruk500data\bruk500data\2012\Oct\data\ejt\nmr\2012-10-18-ejt-1\11\fid					***Frequency (MHz)***	500.13
Nucleus	1H	***Number of Transients***	16	***Origin***	spect	***Original Points Count***	32768
Owner	vnmr1	***Points Count***	32768	***Pulse Sequence***	zg30b	***Receiver Gain***	181.00
SW(cyclical) (Hz)	10330.58	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	3040.7544	***Spectrum Type***	STANDARD
Sweep Width (Hz)	10330.26	***Temperature (degree C)***	21.973				



EB TBS deprot
mCARBON CDCl3 /opt/bruk500data/2012/Oct ejt 1





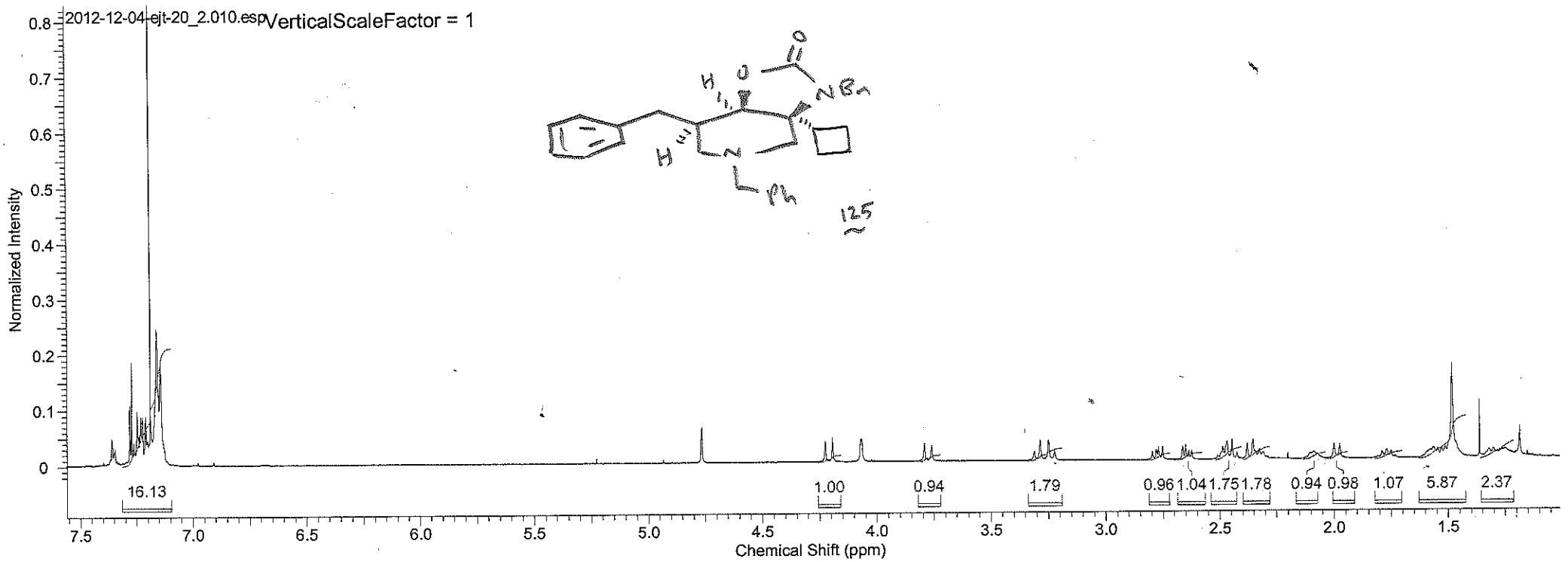
123

BRUKER

NAME	2012-10-18-ejt-1	
EXPNO	10	
PROCNO	1	
Date_	20121018	
Time	9.40	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	256	
DS	2	
SWH	29761.904	Hz
FIDRES	0.454131	Hz
AQ	1.1010548	sec
RG	512	
DW	16.800	usec
DE	32.78	usec
TE	295.0	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	8.80	usec
PL1	-1.00	dB
PL1W	104.35516357	W
SFO1	125.7703643	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	0.00	dB
PL12	17.23	dB
PL13	120.00	dB
PL2W	25.62124252	W
PL12W	0.48484197	W
PL13W	0.00000000	W
SFO2	500.1320005	MHz
SI	32768	
SF	125.7577890	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

06/12/2012 09:45:49

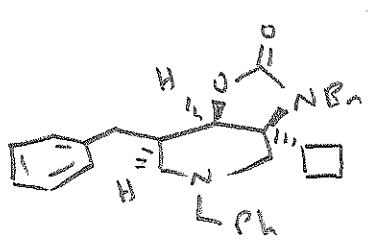
Acquisition Time (sec)	3.1719	***Comment***	EB cyclisation f2 mPROTON CDCl3 /opt/bruk500data/2012/Dec ejt 20			***Date***	04 Dec 2012 13:56:16
Date Stamp	04 Dec 2012 13:56:16						
File Name	\\ss7a.ds.man.ac.uk\vol5\vol3\users\snmrdata\bruk500data\bruk500data\2012\Dec\data\ejt\nmr\2012-12-04-ejt-20_2\10\fid					***Frequency (MHz)***	500.13
Nucleus	1H	***Number of Transients***	16	***Origin***	spect	***Original Points Count***	32768
Owner	vnmr1	***Points Count***	32768	***Pulse Sequence***	zg30b	***Receiver Gain***	256.00
SW(cyclical) (Hz)	10330.58	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	3039.4541	***Spectrum Type***	STANDARD
Sweep Width (Hz)	10330.26	***Temperature (degree C)***	25.029				



No.	(ppm)	Value	Absolute Value	Non-Negative Value
1	2141 .. 1.352	2.36910129	2.74777549e+9	2.36910129
2	4239 .. 1.625	5.87223387	6.81084467e+9	5.87223387
3	7051 .. 1.821	1.07191539	1.24324902e+9	1.07191539
4	9417 .. 2.030	0.98170584	1.13862054e+9	0.98170584
5	0399 .. 2.130	0.94085318	1.09123802e+9	0.94085318
6	2810 .. 2.391	1.77637959	2.06031398e+9	1.77637959
7	4060 .. 2.511	1.74655259	2.02571942e+9	1.74655259

No.	(ppm)	Value	Absolute Value	Non-Negative Value
8	5801 .. 2.701	1.04228306	1.20888026e+9	1.04228306
9	7229 .. 2.811	0.96146834	1.11514829e+9	0.96146834
10	1917 .. 3.331	1.78715038	2.07280627e+9	1.78715038
11	7229 .. 3.821	0.93704081	1.08681626e+9	0.93704081
12	1604 .. 4.251	0.99897206	1.15864653e+9	0.99897206
13	0964 .. 7.311	16.12777328	1.87056169e+10	16.12777328

EB ncycle - do not touch sample.
mCARBON CDCl3 /opt/bruk500data/2013/Feb ejt 2



125

NAME	2013-02-21-ejt-2	
EXPNO	10	
PROCNO	1	
Date_	20130221	
Time	13.18	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	256	
DS	2	
SWH	29761.904	Hz
FIDRES	0.454131	Hz
AQ	1.1010548	sec
RG	512	
DW	16.800	usec
DE	32.78	usec
TE	291.7	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	8.80	usec
PL1	-1.00	dB
PL1W	104.35516357	W
SFO1	125.7703643	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	0.00	dB
PL12	17.23	dB
PL13	120.00	dB
PL2W	25.62124252	W
PL12W	0.48484197	W
PL13W	0.00000000	W
SFO2	500.1320005	MHz
SI	32768	
SF	125.7577890	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

17.5
23.1
23.8
36.3
38.3
39.3
44.8
52.6
53.7
61.9
66.5
75.5
126.31
127.20
127.3
127.8
128.2
128.3
128.5
128.9
129.0
132.0
138.3
138.9
159.2