

Supporting Information
for

Amine-linked diglycosides: Synthesis facilitated by the enhanced reactivity of allylic electrophiles, and glycosidase inhibition assays

Ian Cumpstey^{1,2,*}, Jens Frigell¹, Elias Pershagen¹, Tashfeen Akhtar¹, Elena Moreno-Clavijo³, Inmaculada Robina³, Dominic S. Alonzi⁴ and Terry D. Butters⁴

Address: ¹Department of Organic Chemistry, The Arrhenius Laboratory, Stockholm University, 106 91 Stockholm, Sweden, ²Institut de Chimie des Substances Naturelles, Centre National de la Recherche Scientifique, 91198 Gif-sur-Yvette CEDEX, France, ³Department of Organic Chemistry, Faculty of Chemistry, University of Seville, Prof. García González, 1, 41012 Seville, Spain and ⁴Glycobiology Institute, Department of Biochemistry, Oxford University, South Parks Road, Oxford, OX1 3QU, England

Email: Ian Cumpstey - ian.cumpstey@sjc.oxon.org

* Corresponding author

¹H and ¹³C NMR spectra of compounds 9–17

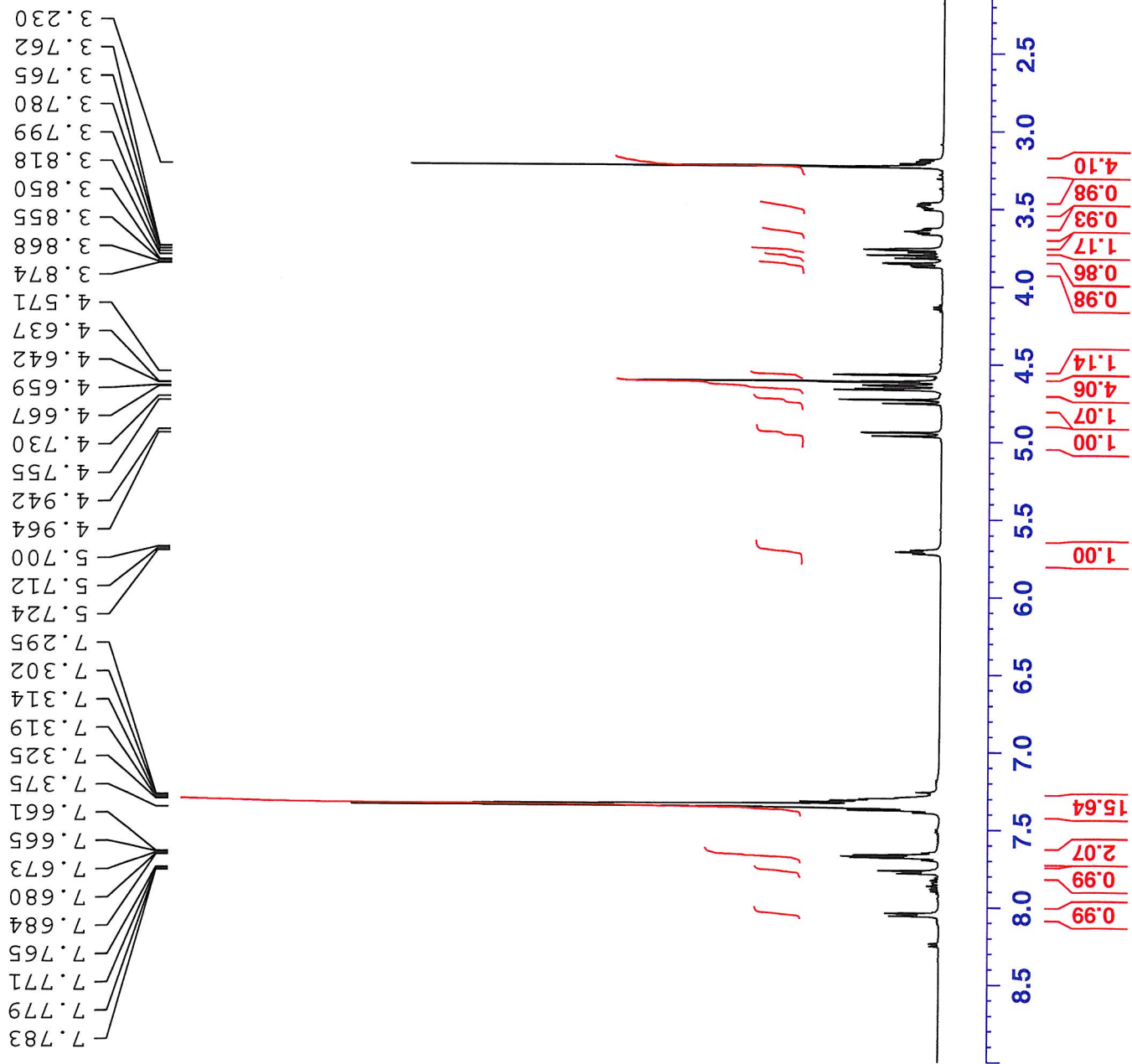
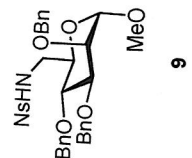


Current Data Parameters
NAME JF-38-03-fr-4-28-noylation
EXPNO 10
PROCNO 10

P2 - Acquisition Parameters
Date_ 20100506
Time 8.42

INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 90.5
RW 62.400 usec
DE 0.0000000 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.80 usec
PL1 2.00 dB
PL1W 15.76968765 W
SFO1 500.1325007 MHz
P2 - Processing parameters
SI 32768
SF 500.1300133 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





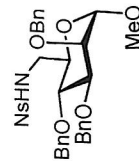
Current Data Parameters
NAME JF-38-03-Oct2010
EXPNO 30
PROCNO 10

F2 - Acquisition Parameters
Date_ 20101012
Time_ 4.27
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 262144
SOLVENT CDCl3
NS 852
DS 0
SWH 27573.529 Hz
FIDRES 0.841477 Hz
AQ 0.5942430 sec
RG 2050
DM 18.133 usec
DE 12.00 usec
TE 298.2 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

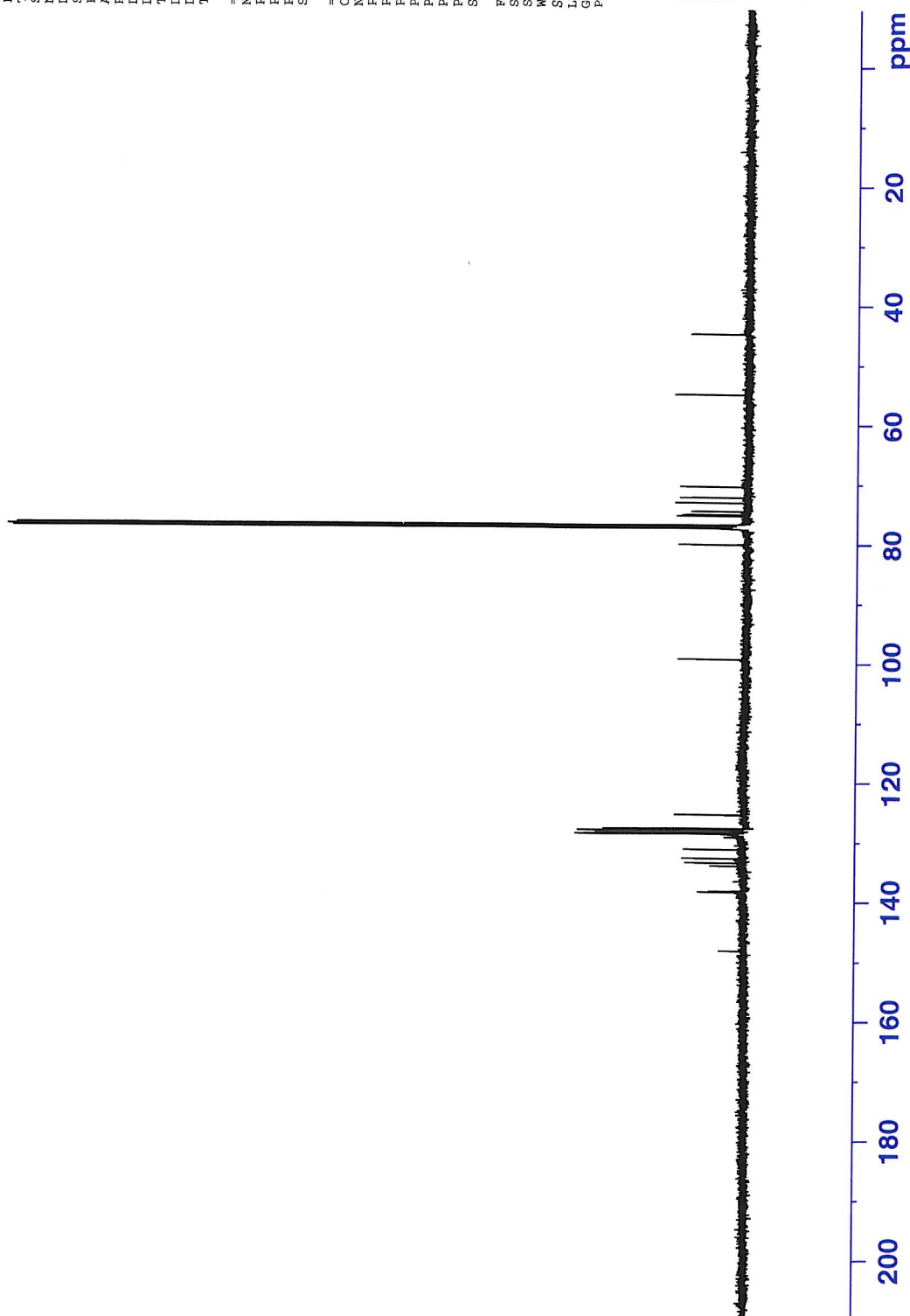
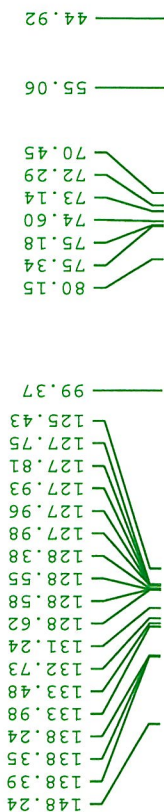
===== CHANNEL f1 =====
NUC1 13C
P1 14.75 usec
PL1 -0.30 dB
PL1W 89.89726257 W
SFO1 125.7703648 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 22.50 dB
PL13 22.50 dB
PL2W 24.99327087 W
PL12W 0.14054748 W
PL13W 0.14054748 W
SFO2 500.1321306 MHz

F2 - Processing parameters
SI 32768
SF 125.7577748 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



9



STANDARD PROTON

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Mercury-400BB "nmr4"

Relax. delay 1.000 sec

Pulse 39.2 degrees

Acq. time 2.733 sec

Width 5995.2 Hz

16 repetitions

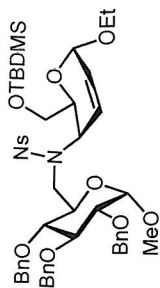
OBSERVE H1, 399.9356487 MHz

DATA PROCESSING

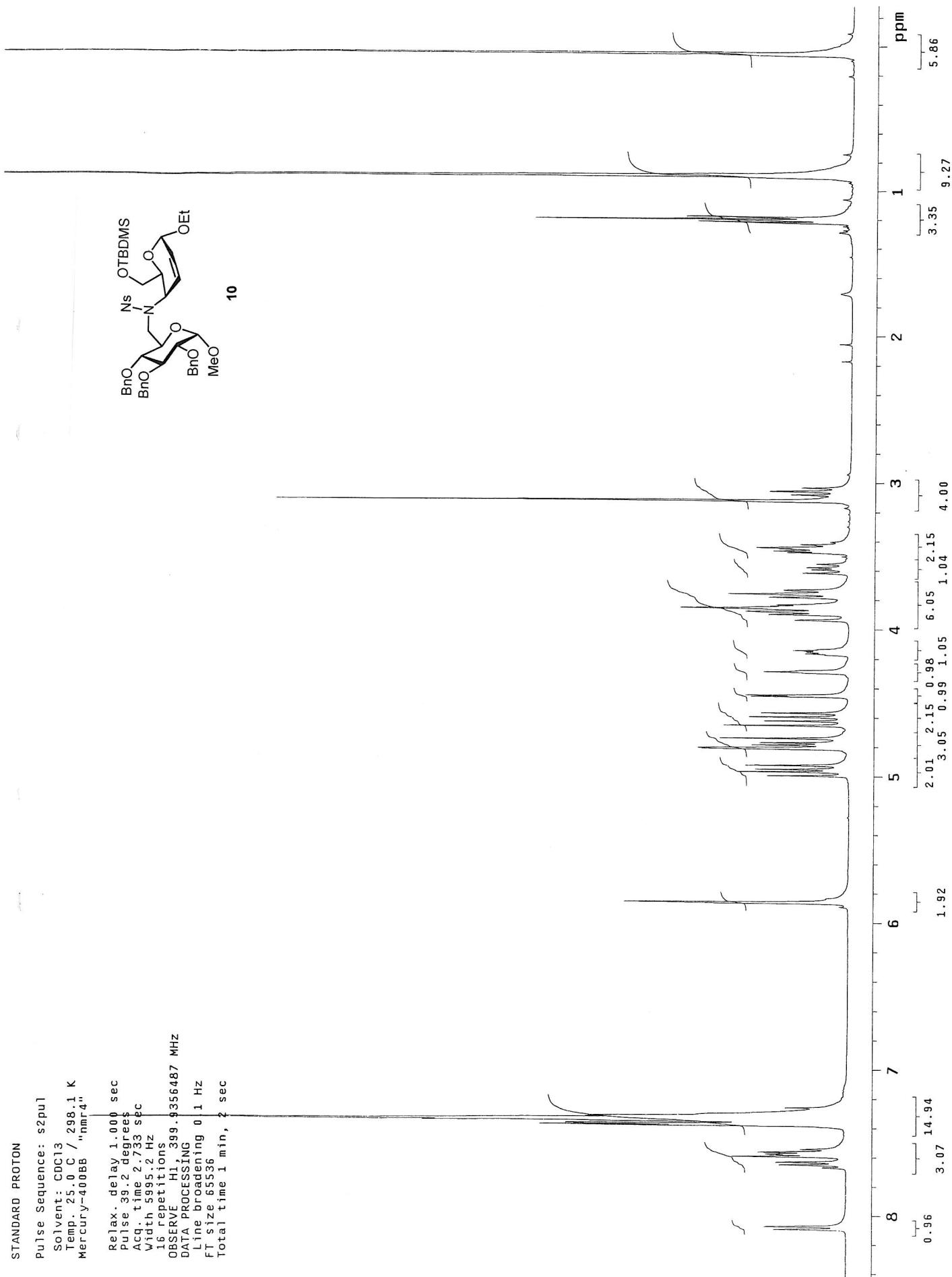
Line broadening 0.1 Hz

FT size 65536

Total time 1 min, 2 sec



10



Pulse Sequence: zgpg30
 Solvent: CDCl₃
 Temp.: 25.0 C
 Mercury-400BB
 Relax. delay: 1.000 sec
 Pulse: 48.9 degrees
 Acq. time: 1.199 sec
 Width: 25000.0 Hz
 148 repetitions
 OBSERVE C13, 100.5638952 MHz
 DECOUPLE H1, 399.9376152 MHz
 Power: 48 dB
 continuously on
 VAL12-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FI size 64536
 Total time 42 min, 5 sec

148.021 N1-C
 138.682 N1-C
 138.211
 138.638
 134.638
 133.621
 132.369
 131.770
 130.526
 127.704
 127.013 C-21 or C-36
 123.994 N5-CN
 127.893

81.940 C-3a
 80.362 C-4a
 79.960 C-2a
 77.479
 77.160
 76.841
 75.703
 75.385
 73.245
 71.880
 69.148
 63.246
 63.140

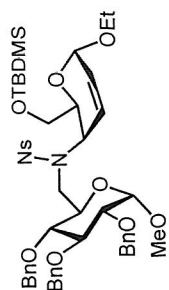
55.727

50.902

47.905

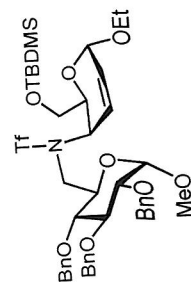
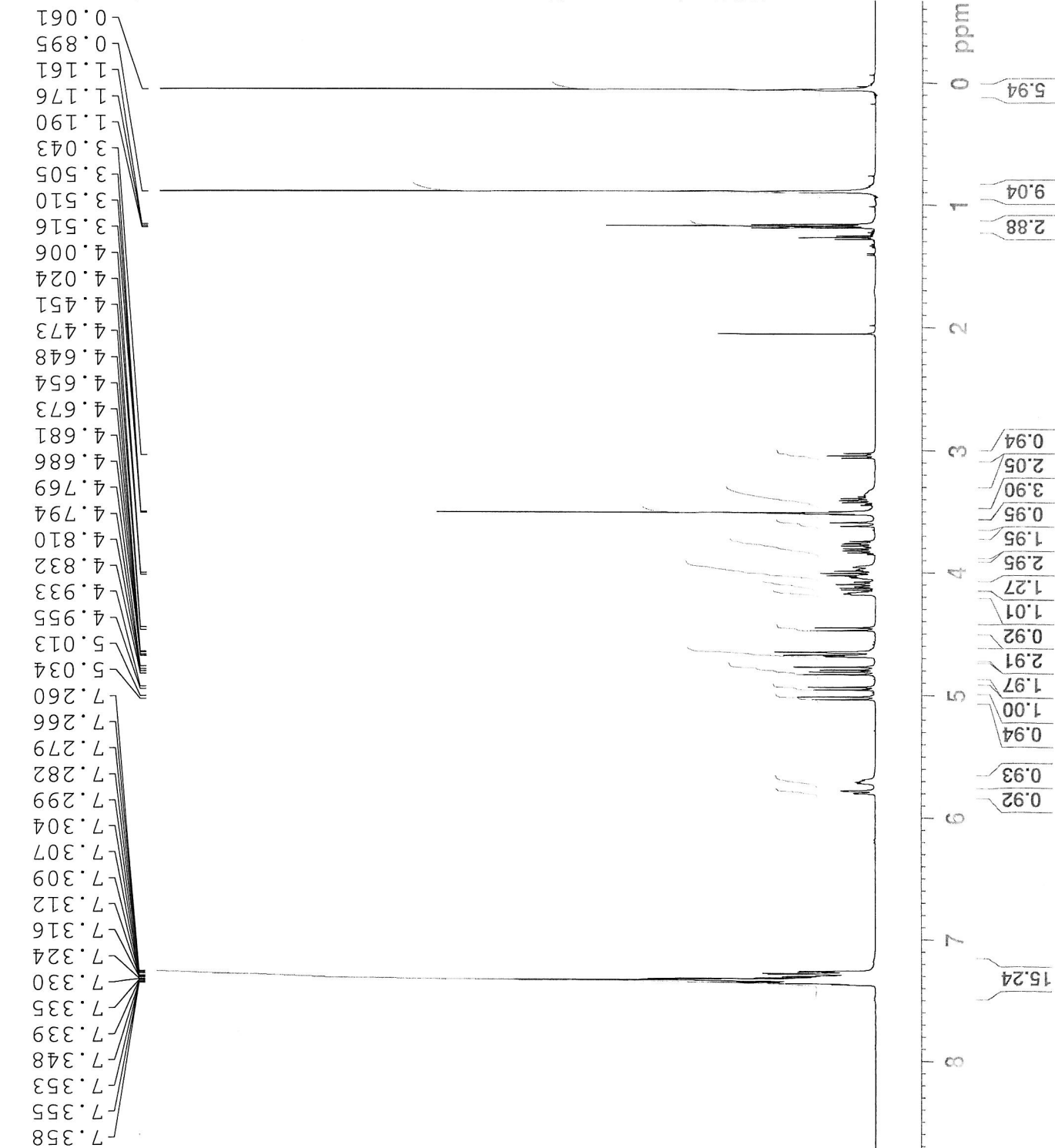
18.286
 15.130
 5.195
 5.226

c(m)5



10

ppm



11



Current Data Parameters
NAME JF-38-04-FL2-fr-24-37
EXPNO 10
PROCNO 10

F2 - Acquisition Parameters
Date_ 20100511
Time 09:25

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zg30

TD 65536

SOLVENT CDCl3

NS 16

DS 0

SWH 8012.820 Hz

FIDRES 0.132466 Hz

AQ 4.089466 sec

RG 90.5

DW 62.400 usec

DE 6.00 usec

TE 298.2 K

D1 2.0000000 sec

TD0 1

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

NUC1 1H

PL1 11.80 usec

PL2 2.00 dB

PL1W 15.76968765 W

SFO1 500.1325007 MHz

F2 - Processing parameters

SI 32768

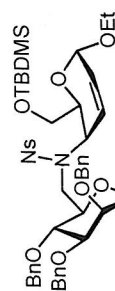
SF 500.1300133 MHz

WDW EM

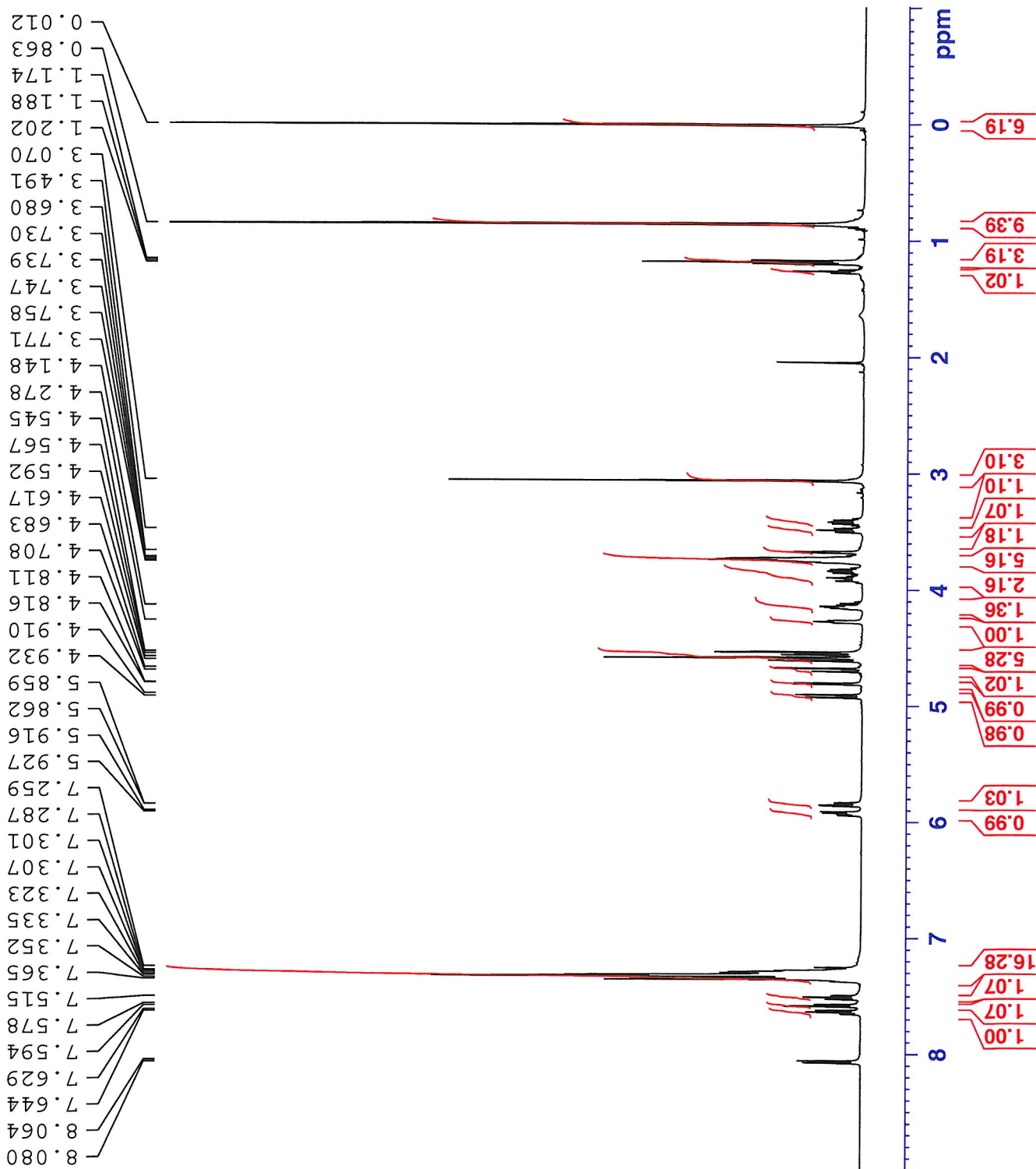
SSB 0

GB 0.30 Hz

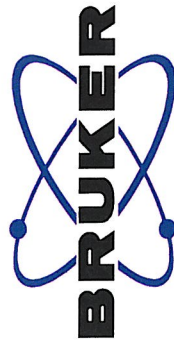
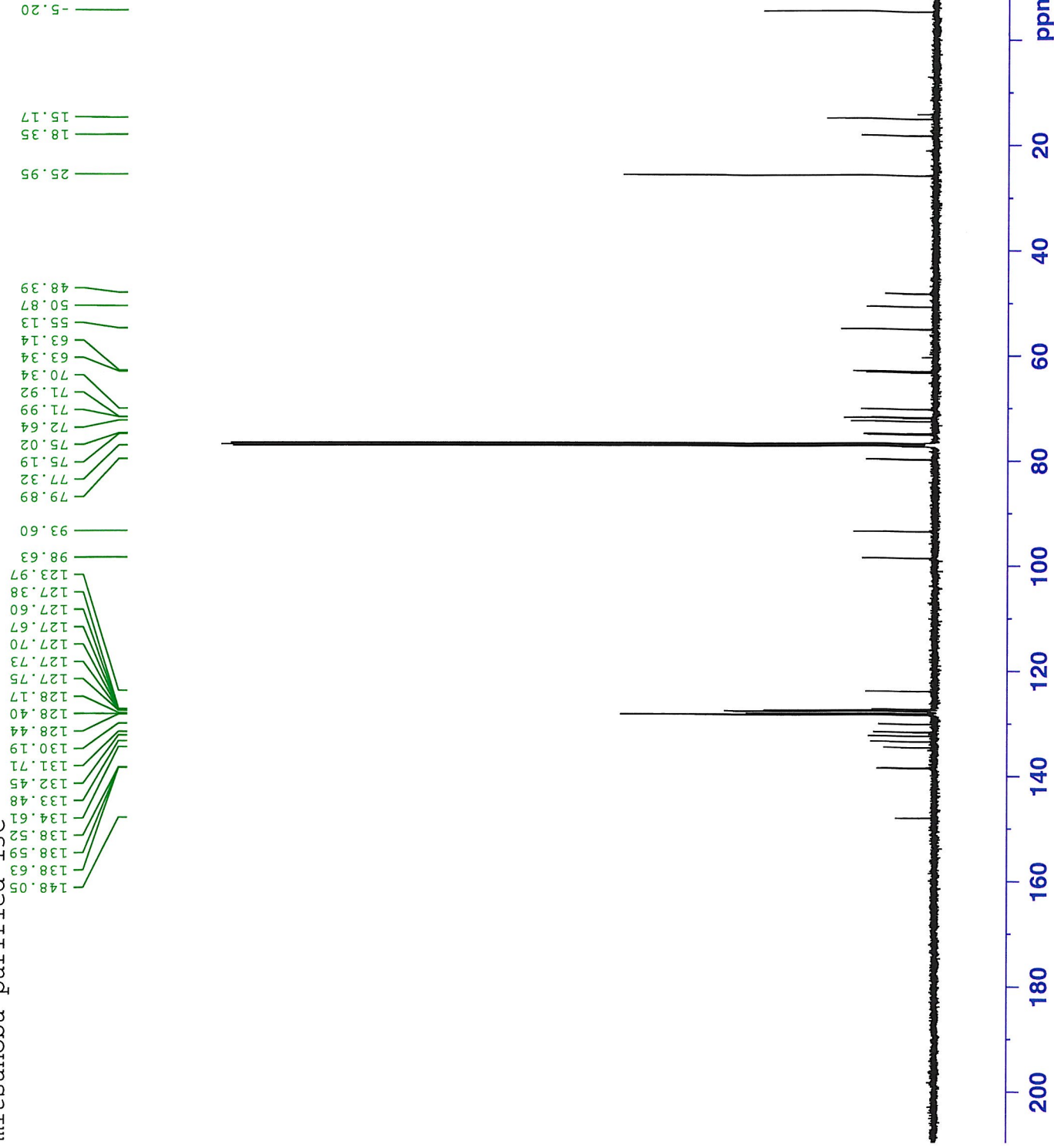
PC 1.00



12



mitsunobu purified 13C



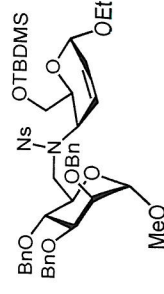
Current Data Parameters
NAME JF-38-04-Flu2-fr-24-37
EXPNO 30
PROCNO 10

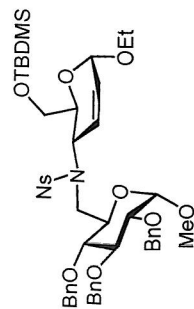
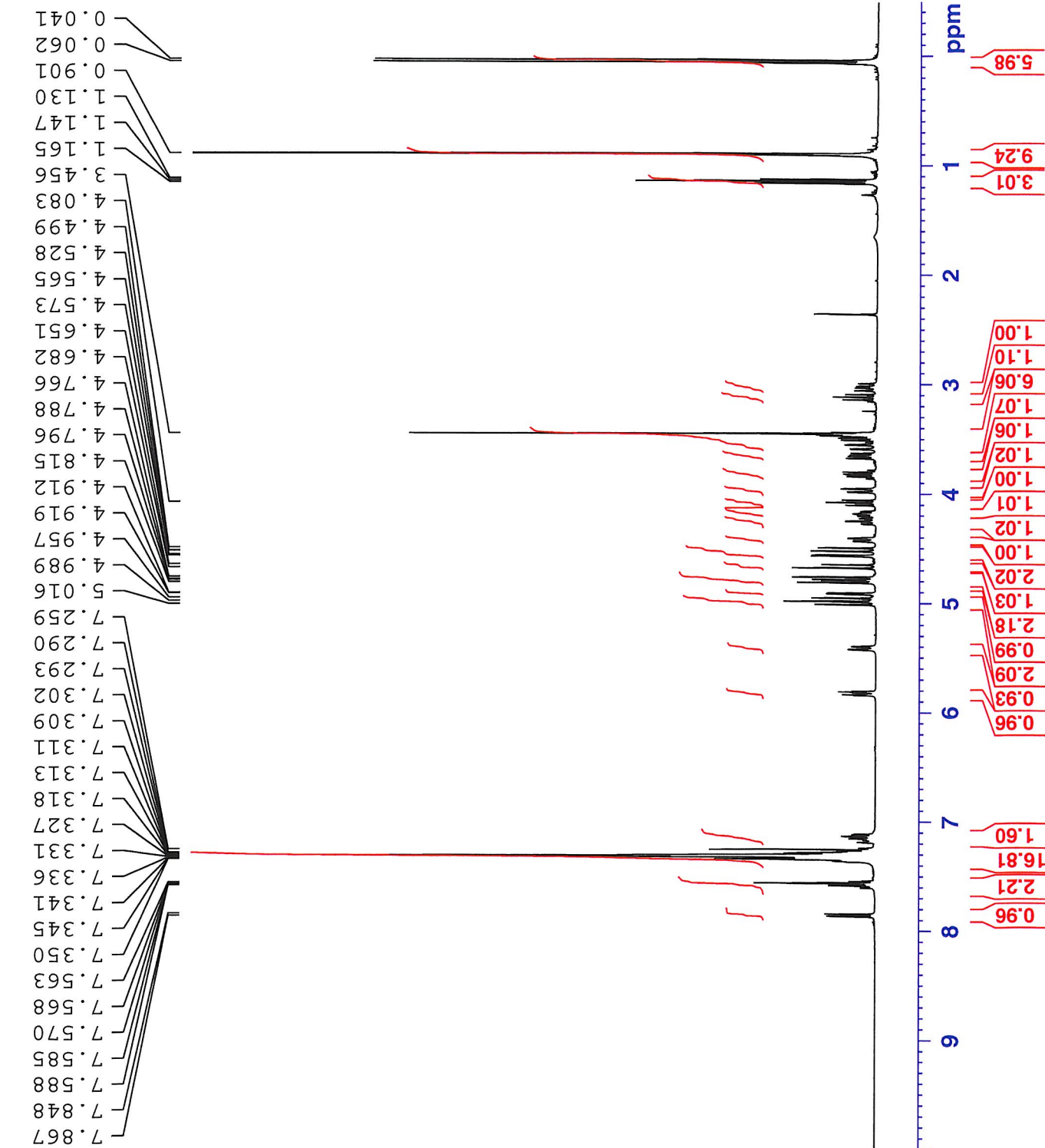
F2 - Acquisition Parameters
Date_ 20100911
Time 5.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 279
DS 0
SWH 27573.529 Hz
FIDRES 0.84177 Hz
AQ 0.5942430 sec
RG 2050
DW 18.133 usec
DE 12.00 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 8

===== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 0.00 dB
PL1W 83.89700317 W
SFO1 125.7703648 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 18.62 dB
PL13 21.00 dB
PL2W 15.76968765 W
PL12W 0.34341794 W
PL13W 0.19852860 W
SFO2 500.1321306 MHz

F2 - Processing parameters
SI 32768
SF 125.7577683 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40







Current Data Parameters
NAME JF-32-13-FL2-fr-4-7
EXPNO 20
PROCNO 20

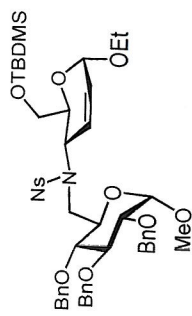
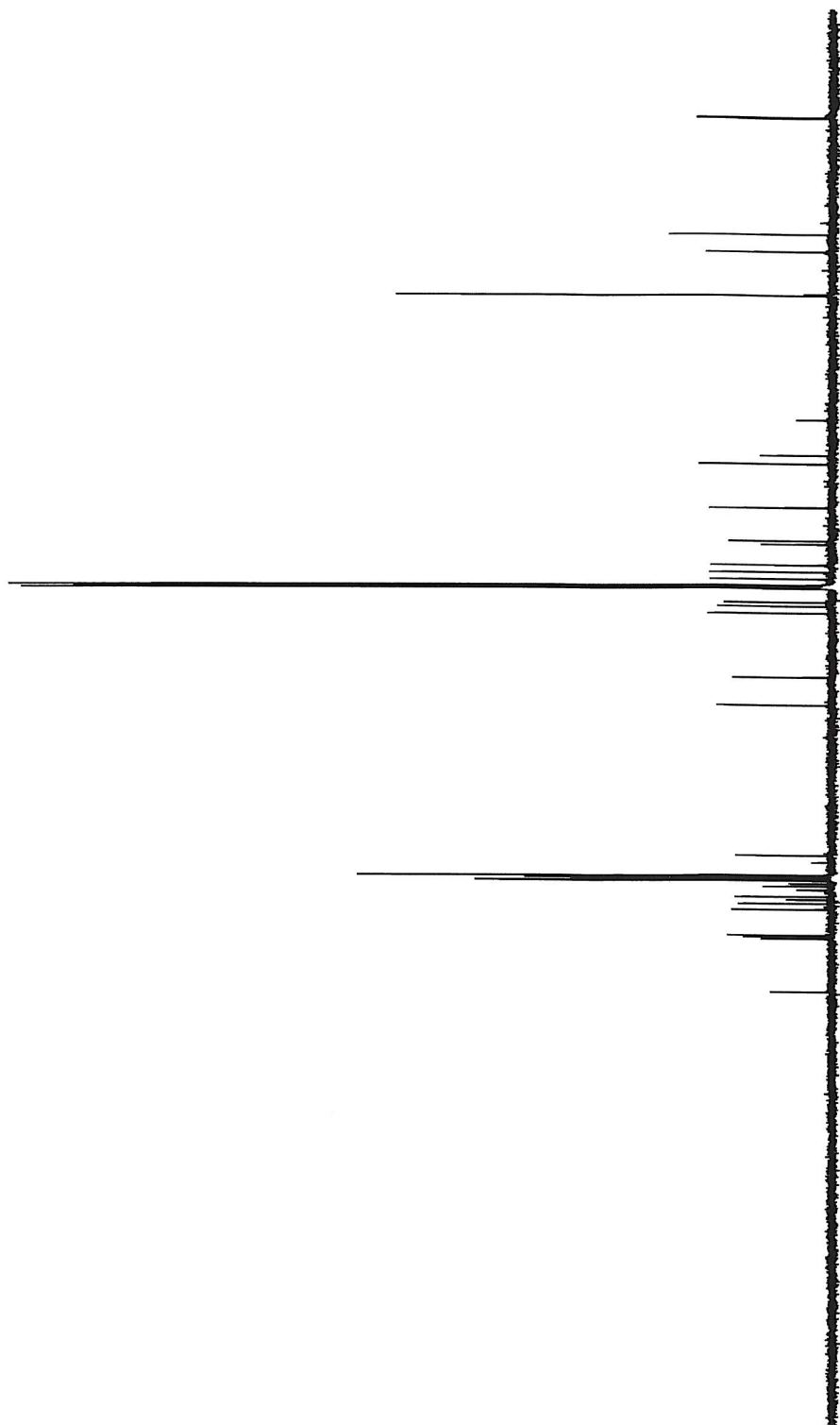
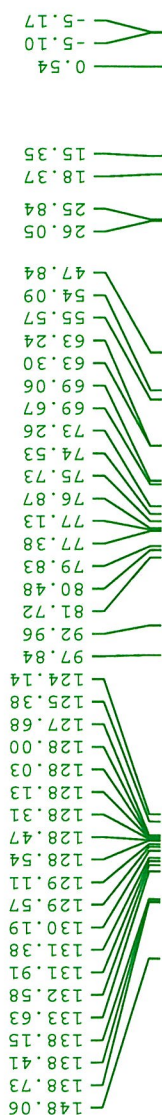
F2 - Acquisition Parameters

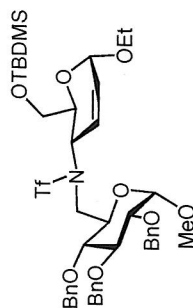
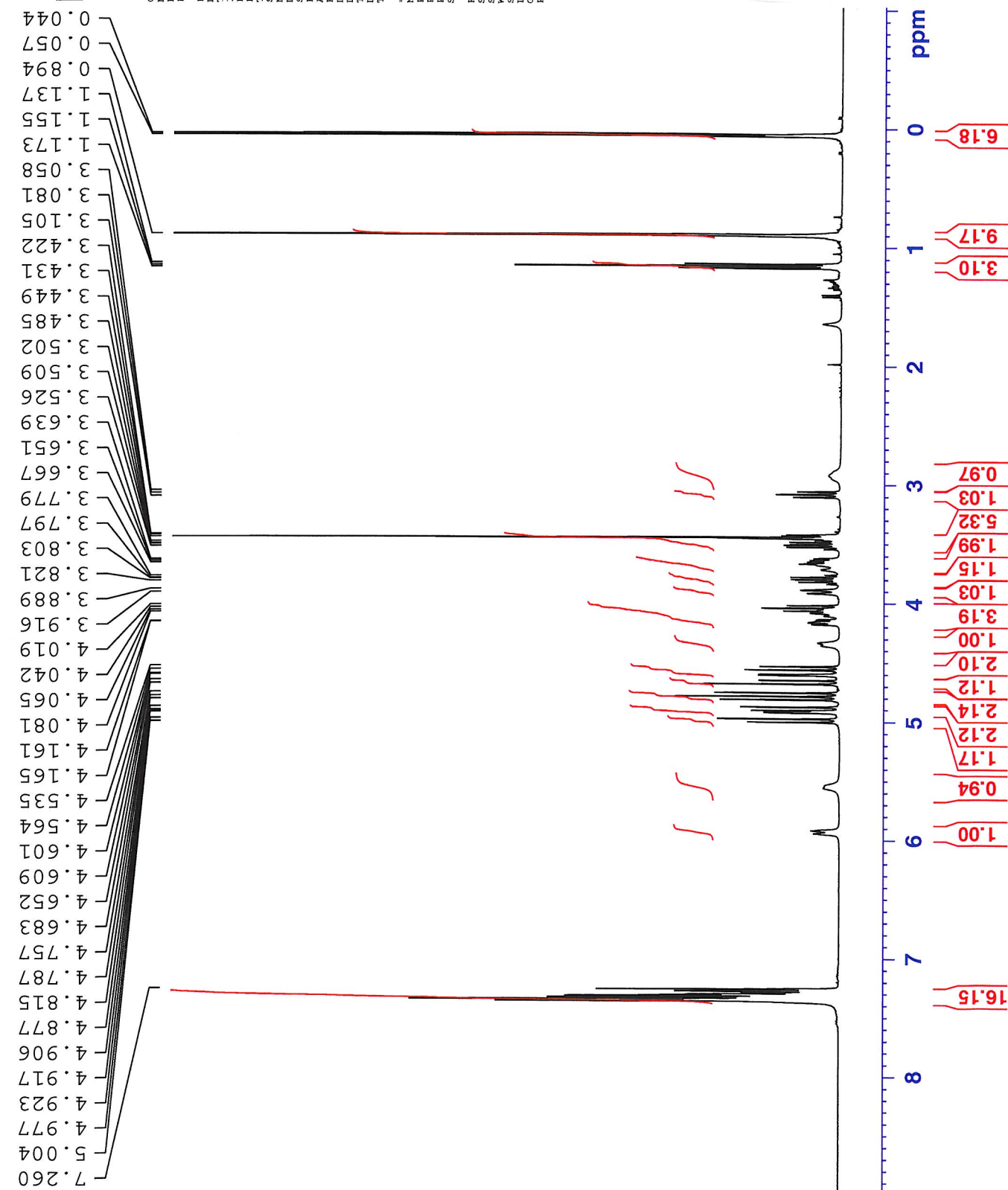
Date_ 20090821
Time 4.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 477
DS 2
SWH 31250.000 Hz
FIDRES 0.476837 Hz
AQ 1.0486259 sec
RG 32800
DW 16.000 usec
DE 6.00 usec
TE 298.2 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
PL 7.5 usec
PL1 0.00 dB
PL1W 83.89700317 W
SF01 125.7703648 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 18.62 dB
PL13 21.00 dB
PL2W 15.76968765 W
PL12W 0.34341794 W
PL13W 0.19852860 W
SF02 500.1320000 MHz

F2 - Processing parameters
SI 32768
SF 125.7577938 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





14

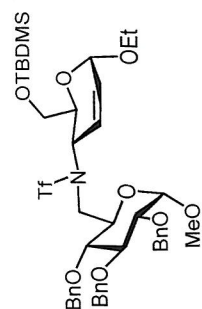


Current Data Parameters
NAME JF-32-12-ft-8-18-disach
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090818
Time 21.46
PULPROG zgpg30
PROBHD 5 mm PARAB-1
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 24038.451 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 51.93 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 ¹³C
P1 13C usec
PL1 -3.00 dB
PL1W 69.66502380 W
SFO1 100.6228298 MHz
===== CHANNEL f2 =====
PULPROG zgpg30
PROBHD 5 mm
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 24038.451 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 51.93 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

F2 - Processing parameters
SI 32768
SF 100.6127576 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



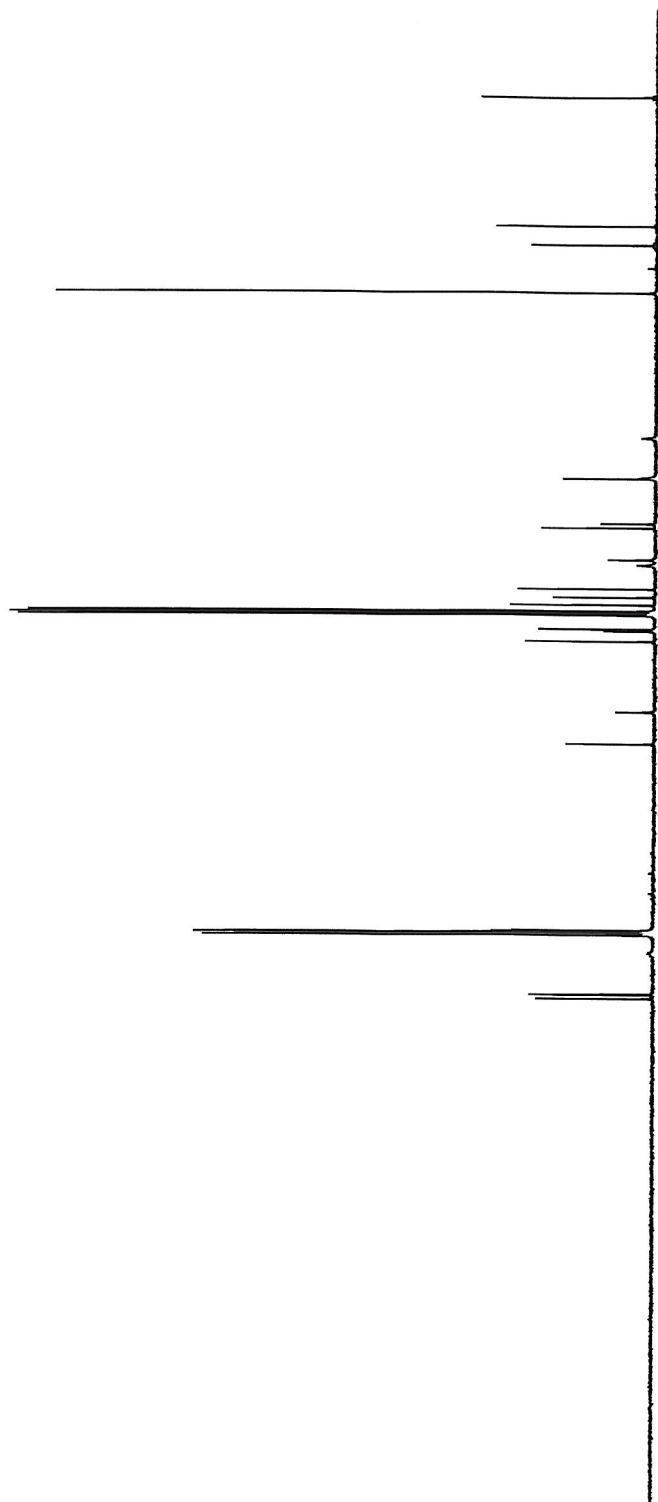
14

5.17
5.14

26.04
22.08
18.41
15.39

98.05
92.97
81.66
80.03
79.75
77.48
77.37
77.16
76.85
76.87
74.64
73.33
69.49
68.63
63.59
62.87
55.70
55.53
49.23

138.73
138.10
138.01
128.61
128.59
128.55
128.15
128.12
128.09
127.94
127.80



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

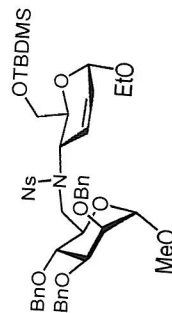
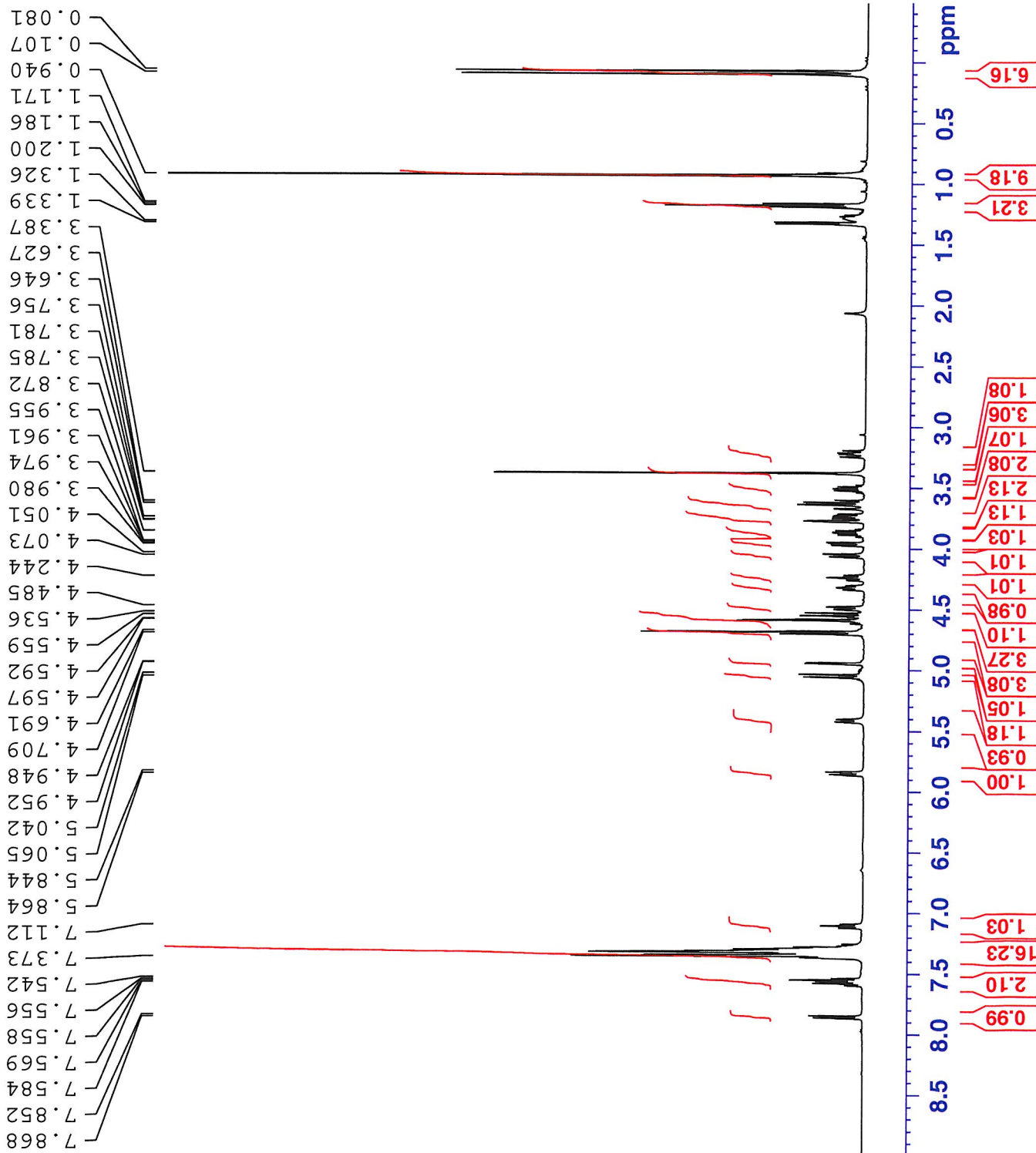


Current Data Parameters
NAME JF-38-05-fr-48-61
EXPNO 10
PROCNO 10

F2 - Acquisition Parameters
Date_ 20100510
Time_ 1.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 36
DM 62.400 usec
DE 6.00 usec
TE 298.2 K
D1 2.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.80 usec
PL1 2.00 dB
PL1W 15.76968765 W
SFO1 500.1325007 MHz

F2 - Processing parameters
SI 32768
SF 500.1300130 MHz
WDW EM
SSE 0
LB 0.30 Hz
GB 0
PC 1.00

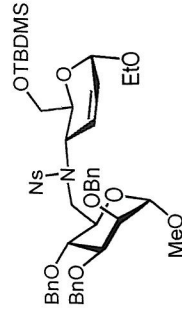




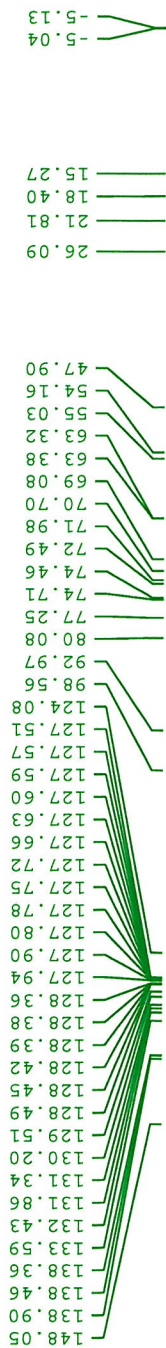
Current Data Parameters
NAME JF-38-05-fr-48-61
EXPNO 30
PROCNO 30

F2 - Acquisition Parameters
Date_ 20100510
Time 1.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDC13
NS 2048
DS 0
SWH 27573.529 Hz
FIDRES 0.841477 Hz
AQ 0.5942430 sec
RG 2050
DW 18.133 usec
DE 12.00 usec
TE 298.2 K
TD0 2.00000000 sec
D1 0.03000000 sec
D11 1
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 0.00 dB
PL1W 83.89700317 W
SFO1 125.7703648 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 18.62 dB
PL13 21.00 dB
PL2W 15.76968765 W
PL12W 0.34341794 W
PL13W 0.19852860 W
SFO2 500.1321306 MHz
F2 - Processing parameters
SI 32768
SF 125.7577816 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



15



180 160 140 120 100 80 60 40 20 ppm



0.032
0.018

1.138
1.120
1.103
0.873

3.424

3.871

5.027
5.001

7.258

Current Data Parameters
NAME JF-32-17-fr-21-32-dihydrox
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters

Date_ 20090918

Time 4.12

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 16

DS 2

SWH 8223.695 Hz

FIDRES 0.125483 Hz

AQ 3.9846387 sec

RG 80.6

DW 60.800 usec

DE 1.500 usec

TE 297.2 K

D1 1.00000000 sec

TD0 1

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

NUC1 1H

P1 8.50 usec

PL1 -4.00 dB

PL1W 24.73352814 W

SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768

SF 400.1300102 MHz

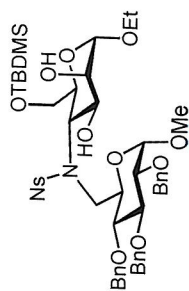
WDW EM

SSB 0

LB -0.20 Hz

GB 0

PC 1.00



16

ppm

6.35

9.61

3.10

2.03

6.41

2.30

5.44

0.96

1.10

3.26

3.23

2.00

1.92

16.94

1.22

1.21

0.92



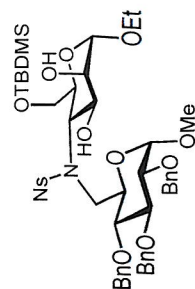
Current Data Parameters
NAME JF-32-22-october2010
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101005
Time 15.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.363376 sec
RG 32050
DM 20.800 usec
DE 51.93 usec
TE 298.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

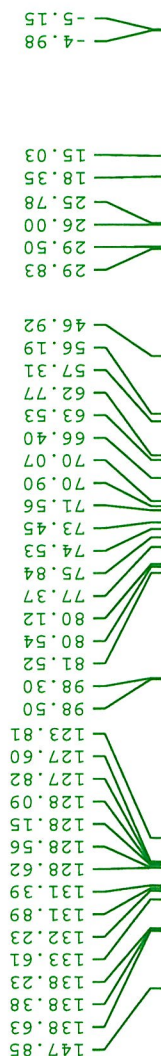
===== CHANNEL f1 =====
NUC1 13C
P1 6.43 usec
PL1 -1.00 dB
PL1W 69.66502380 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CFPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 16.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL2W 0.24733528 W
PL12W 0.31137666 W
PL13W 0.31137666 W
SFO2 400.1316003 MHz

F2 - Processing parameters
SI 32768
SF 100.6127572 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



16



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



Current Data Parameters
NAME JF-38-06-fr-18-26-dihydrox
EXPNO 10
PROCNO 10

F2 - Acquisition Parameters

Date_ 20100519

Time_ 5.18

INSTRUM spect

PROBHD 5 mm PABBO BB-

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 16

DS 0

SWH 8012.800 Hz

FIDRES 0.122266 Hz

AQ 4.0894966 sec

RG 90.5

DW 62.400 usec

DE 2.00 usec

TE 298.0 K

D1 2.00000000 sec

TD0 1

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

MUCL 11.80 usec

PL1 2.00 dB

PULP 15.76968765 W

SFO1 500.1325007 MHz

F2 - Processing parameters

SI 32768

SF 500.1300131 MHz

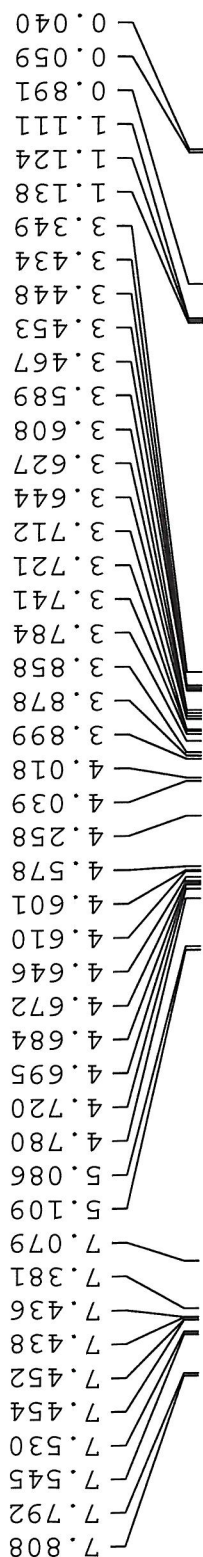
WDW EM

SSB 0

LA -0.10 Hz

GB 0

PC 1.00



ppm

0

1

2

3

4

5

6

7

8

6.74

10.40

3.28

1.03

0.96

1.01

1.29

3.23

1.21

2.39

3.53

6.81

1.06

6.93

1.13

1.00

1.03

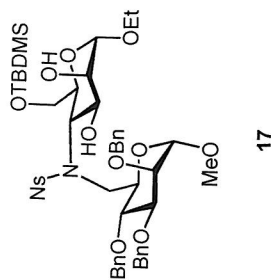
0.48

17.38

1.12

1.11

0.96





Current Data Parameters
NAME JF-38-06-fx-18-26-dihydrox
EXPNO 30
PROCNO 10

F2 - Acquisition Parameters

Date_ 20100519
Time 5.41
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2048
DS 0
SWH 27573.520 Hz
FIDRES 0.841477 Hz
AQ 0.5942430 sec
RG 2050
AQ 18.133 usec
DM 0.0000000 sec
DE 2.0000000 sec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

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

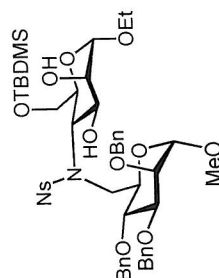
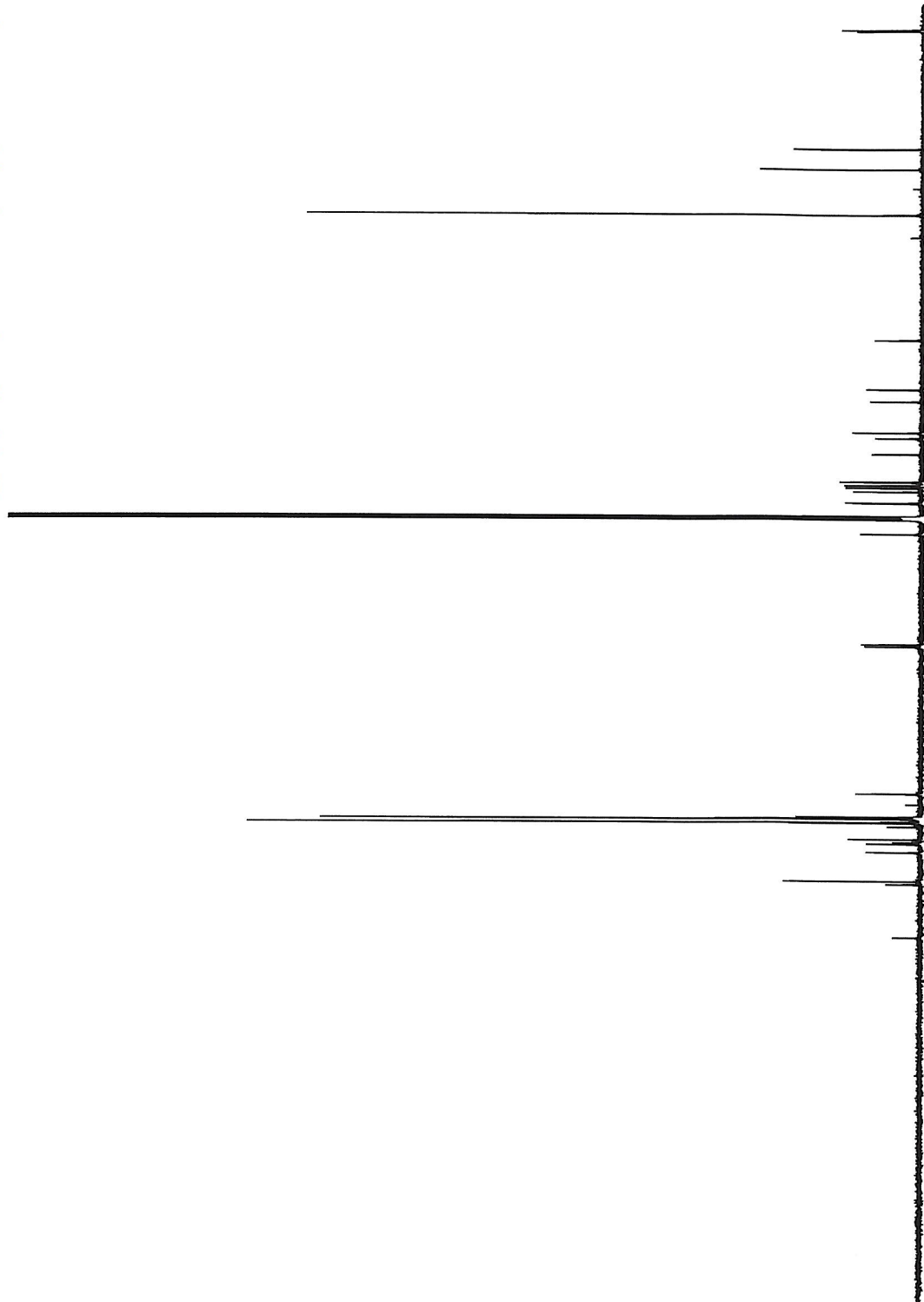
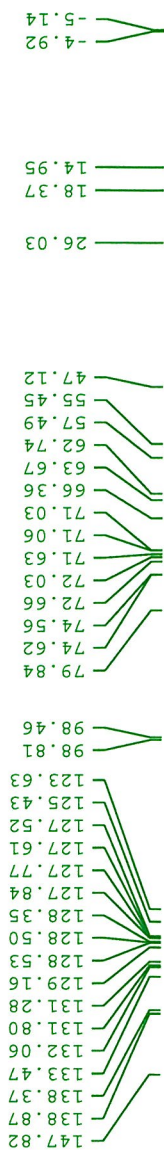
NUC1 13C
P1 7.50 usec
PL1 0.00 dB
PL1W 83.89700317 W
SFO1 125.7703686 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
P2 8.00 usec
PL2 0.00 dB
PL2W 2.00 dB
PL12 18.62 dB
PL13 21.00 dB
PL1W 15.76968765 W
PL2W 0.34312528 W
PL13W 0.39852860 W
SFO2 500.1321306 MHz

F2 - Processing parameters

SI 32768
SF 125.757785 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
FC 1.40



17