

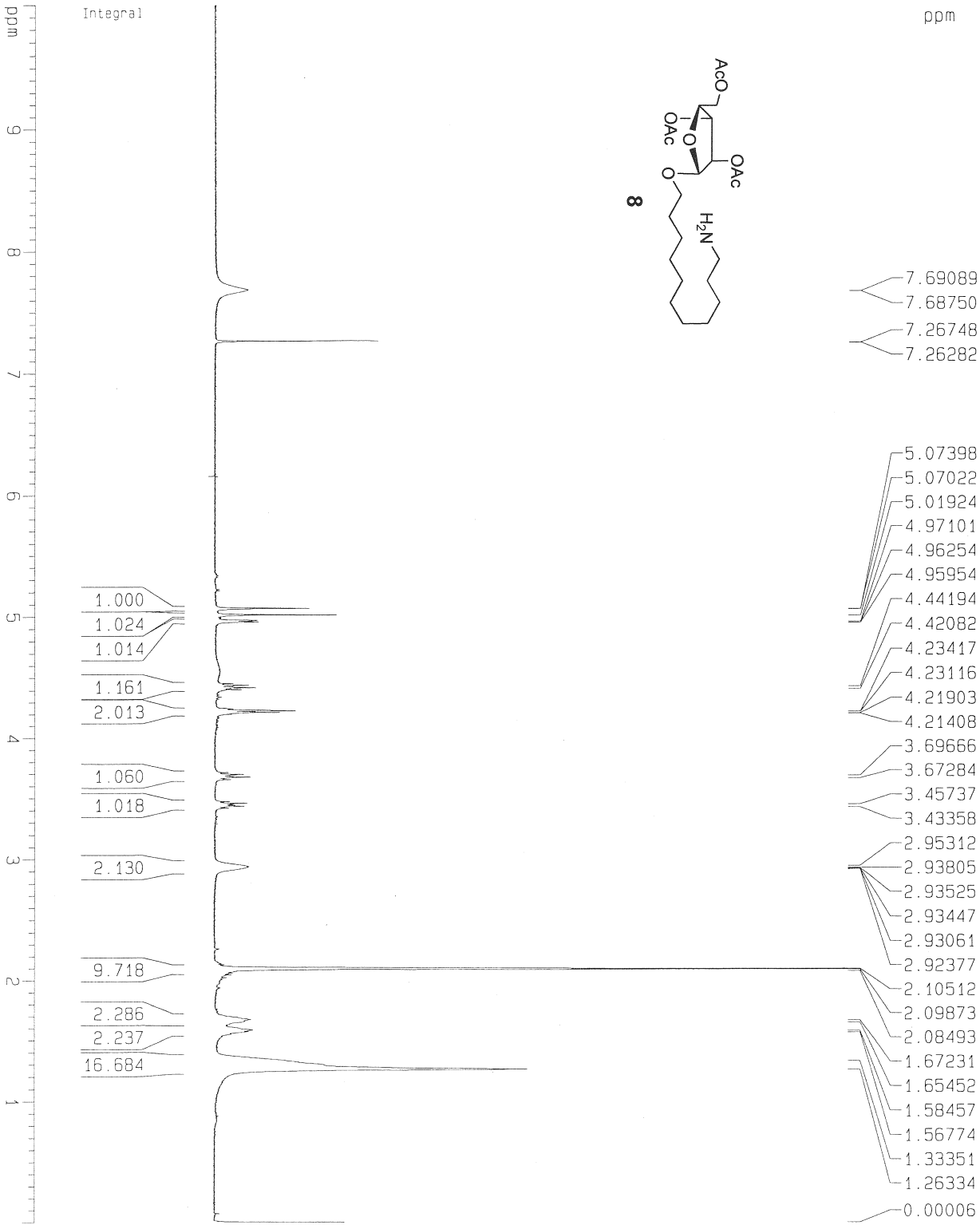
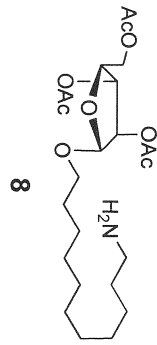
Synthesis and Evaluation of QS-21-Based Immunoadjuvants with a Terminal-Functionalized Side Chain Incorporated in the West Wing Trisaccharide

Pengfei Wang,^{*†} Dattatray A. Devalankar,[†] Qipu Dai,[†] Ping Zhang,^{*‡} and Suzanne M. Michalek[§]
[†]Department of Chemistry, [‡]Department of Pediatric Dentistry, [§]Department of Microbiology, University of Alabama at Birmingham, 901 14th Street South, Birmingham, AL 35294

Table of Content

	Page
Table of content	S1
¹ H and ¹³ C NMR spectra of 8	S2-3
¹ H and ¹³ C NMR spectra of 9	S4-5
¹ H and ¹³ C NMR spectra of 20x	S6-7
¹ H and ¹³ C NMR spectra of 21x	S8-9
¹ H and ¹³ C NMR spectra of 22x	S10-11
¹ H and ¹³ C NMR spectra of 3x	S12-13
¹ H and ¹³ C NMR spectra of 4x	S14-15
¹ H and ¹³ C NMR spectra of 5x	S16-17

1H



Current Data Parameters
 NAME dd-3-131pure1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20151221
 Time 15.54
 INSTRUM drx400
 PROBH0 5 mm Multinu
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SMH 8278.146 Hz
 FIDRES 0.126314 Hz
 A0 3.9584243 sec
 RG 645.1
 DW 60.400 usec.
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

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

NUC1 1H
 P1 6.50 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters

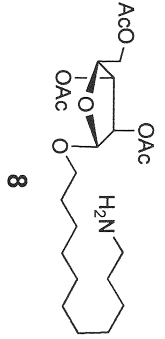
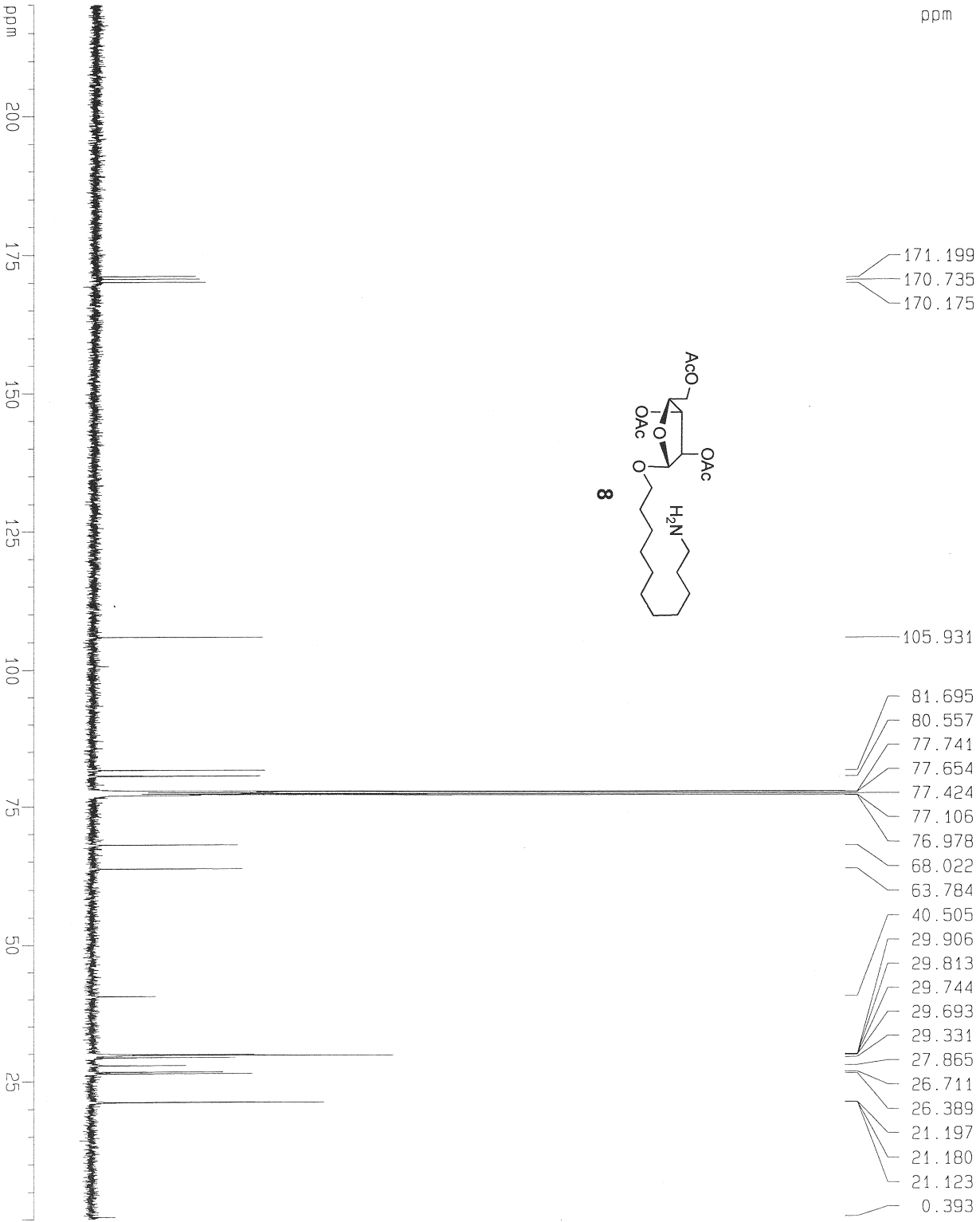
S1 131072
 SF 400.1300061 MHz
 WDM no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PRKCM 0.50000 ppm/cm
 HZCM 200.06500 Hz/cm

13C w/ 1H decoupling

ppm



Current Data Parameters
 NAME dd-3-13113c
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151222
 Time 16.07
 INSTRUM drx400
 PROBD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 8192
 DS 0
 SWH 25125.629 Hz
 FIDRES 0.383387 Hz
 AQ 1.3042164 sec
 RG 32768
 DW 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.000000000 sec
 D11 0.030000000 sec
 D12 0.000020000 sec

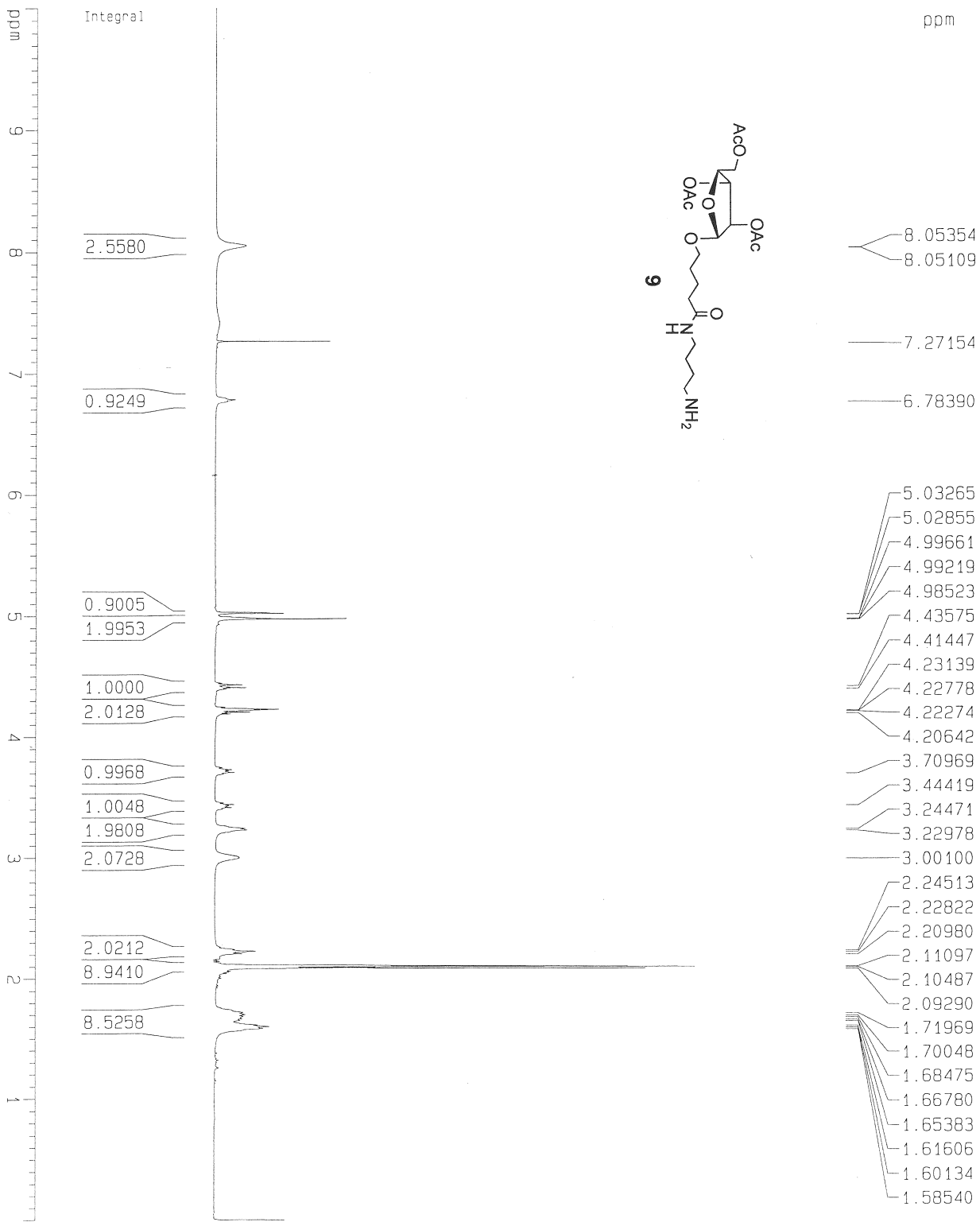
==== CHANNEL f1 =====
 NUC1 13C
 P1 16.00 usec
 PL1 0.00 dB
 SF01 100.6237959 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PGPD2 102.00 usec
 PL2 0.00 dB
 PL12 23.00 dB
 PL13 23.00 dB
 SF02 400.1316005 MHz

F2 - Processing parameters
 SI 131072
 SF 100.6127290 MHz
 KDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.80 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPRCM 11.00000 ppm/cm
 HZCM 1106.73999 Hz/cm

1H



Current Data Parameters
 NAME dd-3-133a
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151223
 Time 8:32
 INSTRUM gnx400
 PROBHD 5 mm MUltinu
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 101.6
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

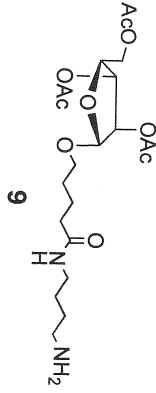
===== CHANNEL f1 =====
 NUC1 1H
 P1 6.50 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 131072
 SF 400.1300044 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.50000 ppm/cm
 HZCM 200.06500 Hz/cm

13C w/ 1H decoupling

- 174.981
- 171.237
- 170.704
- 170.579
- 105.988
- 81.956
- 80.445
- 77.762
- 77.643
- 77.444
- 77.126
- 67.509
- 63.573
- 39.866
- 38.925
- 36.159
- 31.318
- 28.930
- 26.555
- 24.808
- 22.873
- 21.164
- 21.028



Current Data Parameters
 dd-3-133c13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151223
 Time 10.02

INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT DMS-D6
 NS 1024

DS 4
 SWH 25125.629 Hz
 FIDRES 0.383387 Hz
 AQ 1.3042164 sec
 RG 32768

DW 19.900 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 D12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C

P1 16.00 usec
 PL1 0.00 dB
 SF01 100.6237959 MHz

===== CHANNEL f2 =====
 CPGPRG2 waltz16

NUC2 1H
 PCPD2 102.00 usec
 PL2 0.00 dB
 PL12 23.00 dB
 PL13 23.00 dB
 SF02 400.1316005 MHz

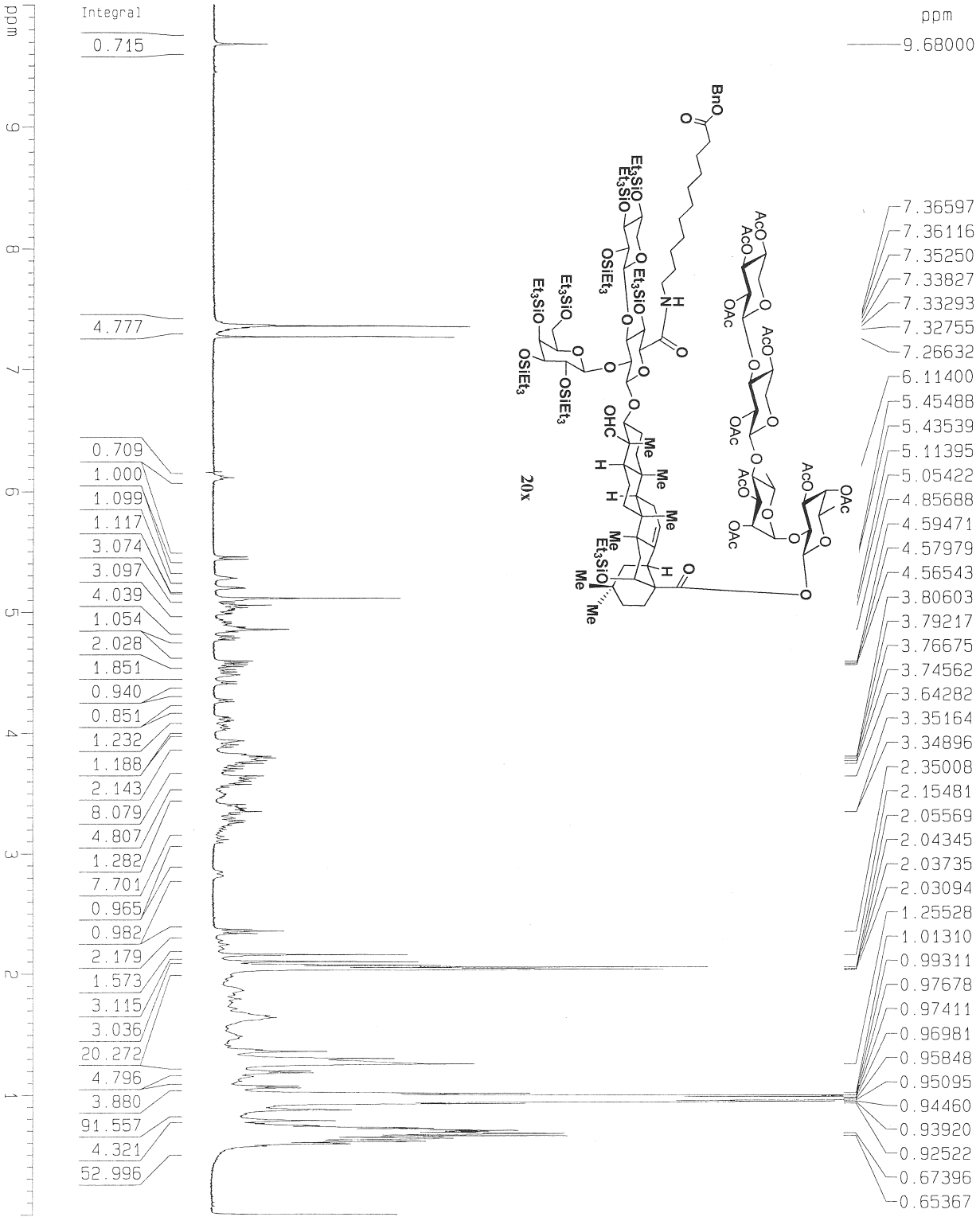
F2 - Processing parameters

SI 131072
 SF 100.6127299 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.80 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1106.73999 Hz/cm

PW-VIII-13-C1A



Current Data Parameters
 NAME PW-VIII-13c1a
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130617
 Time 9.17

INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30

TD 65536
 SOLVENT CDCl3
 NS 15
 DS 2

SMH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec

RG 256
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K

D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H

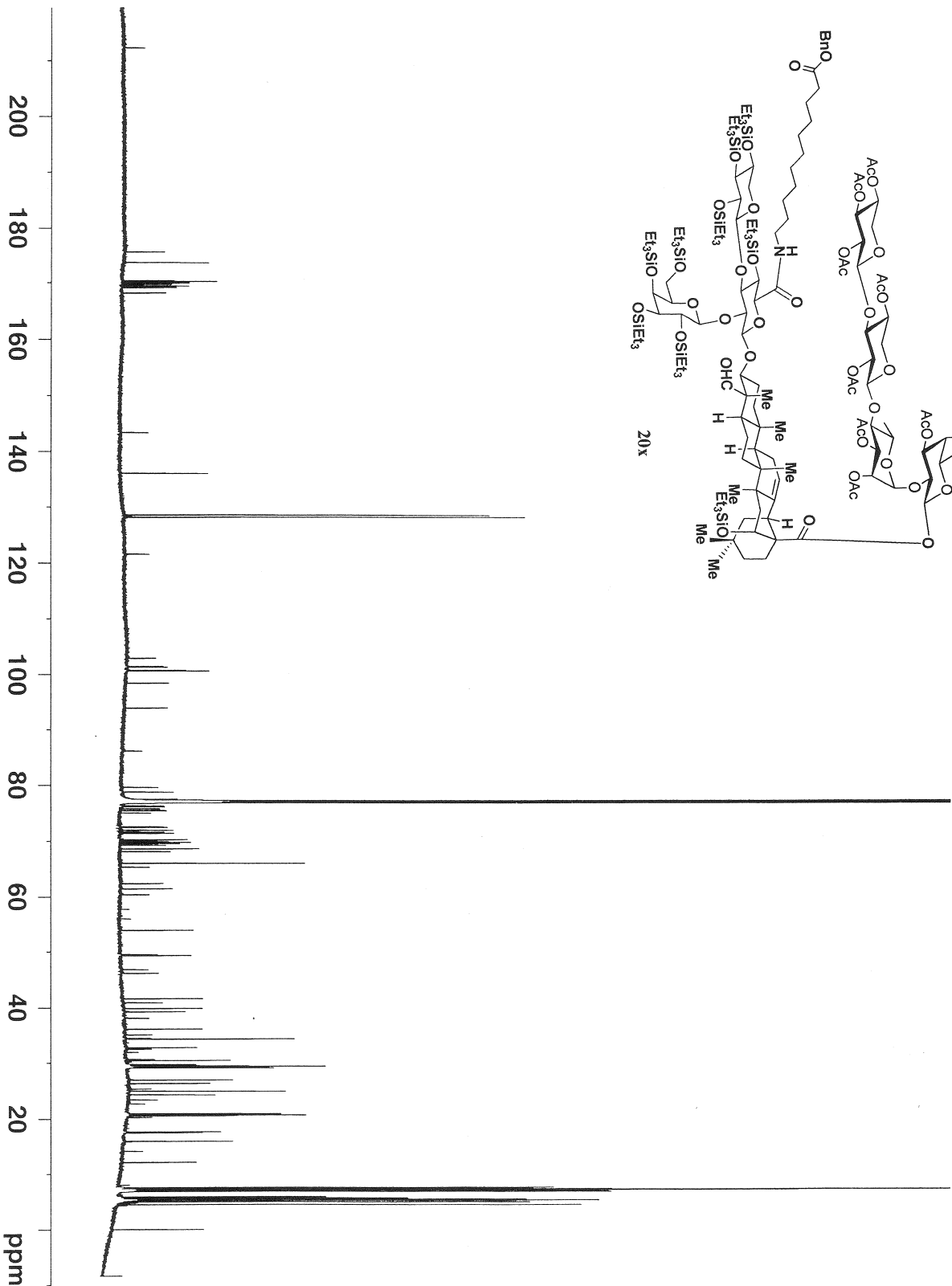
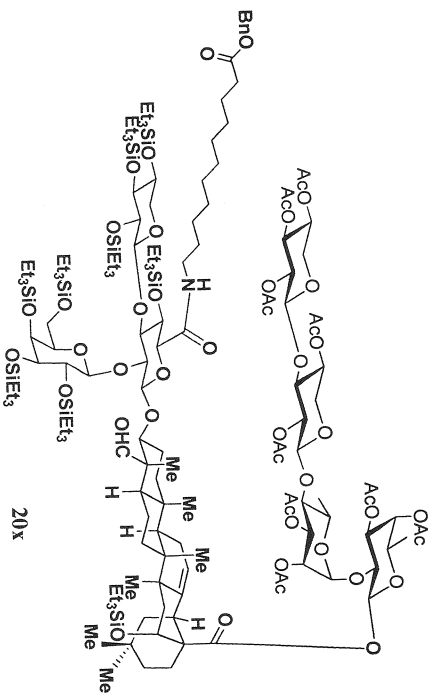
P1 6.50 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 131072
 SF 400.1300066 MHz

WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz

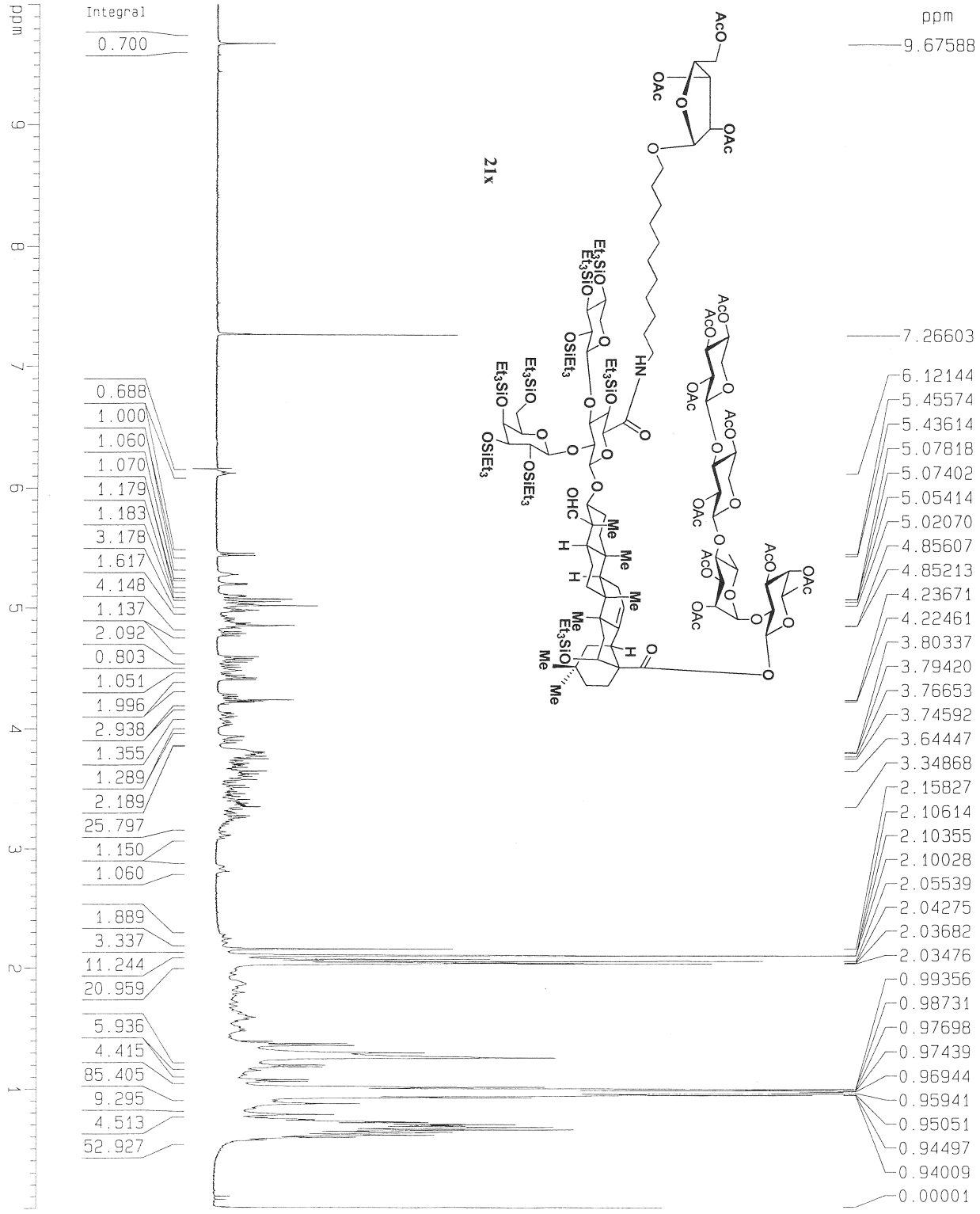
PPMCM 0.50000 ppm/cm
 HZCM 200.05500 Hz/cm



NAME	VII-13
EXPNO	702
PROCNO	1
Date_	20130619
Time	15.55
INSTRUM	spect
PROBHD	5 mm CP131 H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	4096
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 se
RG	64
DW	12.000 us
DE	20.00 us
TE	296.6 K
D1	4.19999981 se
D11	0.03000000 se
TD0	1

===== CHANNEL F1 =====	
NUC1	13C
P1	11.80 us
PL1	-1.30 dB
PL1W	128.76077271 W
SFO1	176.0395312 MHz

===== CHANNEL F2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 us
PL2	1.30 dB
PL12	19.50 dB
PL13	19.50 dB
PL2W	6.88972235 W
PL12W	0.10428017 W
PL13W	0.10428017 W
SFO2	700.0228001 MHz
SI	131072
SF	176.0201725 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



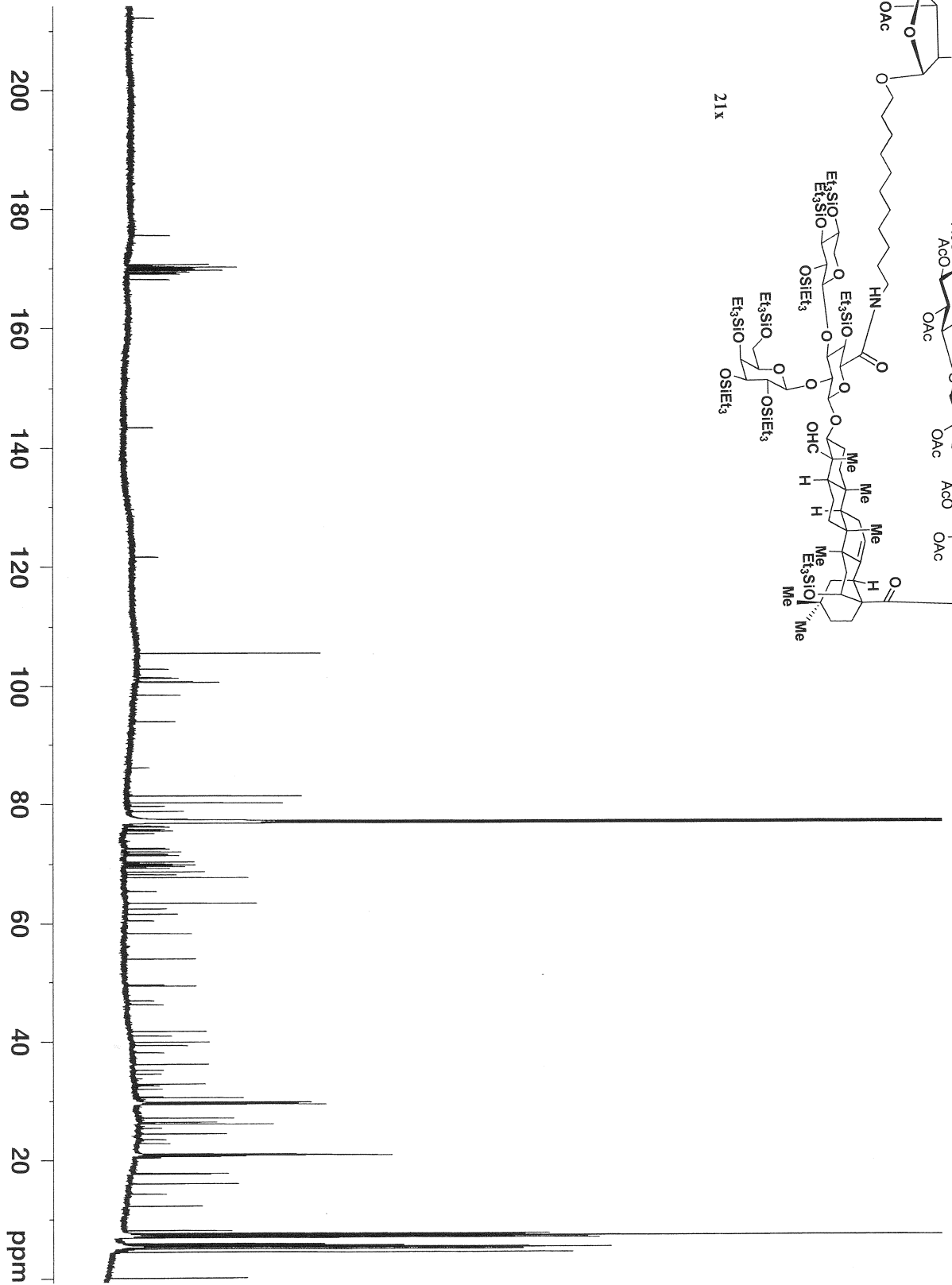
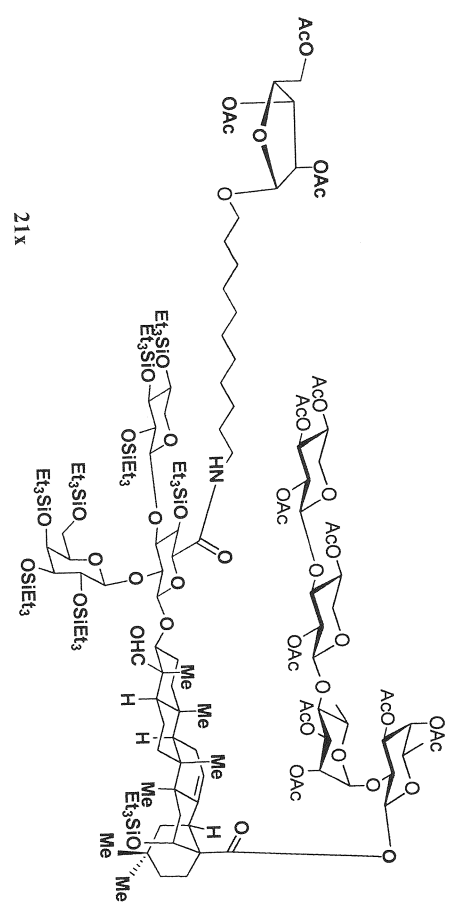
Current Data Parameters
 NAME PW-VIII-12c2a
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130617
 Time 8.52
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 HZ
 FIDRES 0.126314 HZ
 AQ 3.9584243 sec.
 RG 181
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 6.50 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 131072
 SF 400.1300055 MHz
 KW 0
 SSB 0
 LB 0.00 HZ
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1p 10.000 ppm
 F1 4001.30 HZ
 F2p 0.000 ppm
 F2 0.00 HZ
 PPMCM 0.50000 ppm/cm
 HZCM 200.06500 HZ/cm



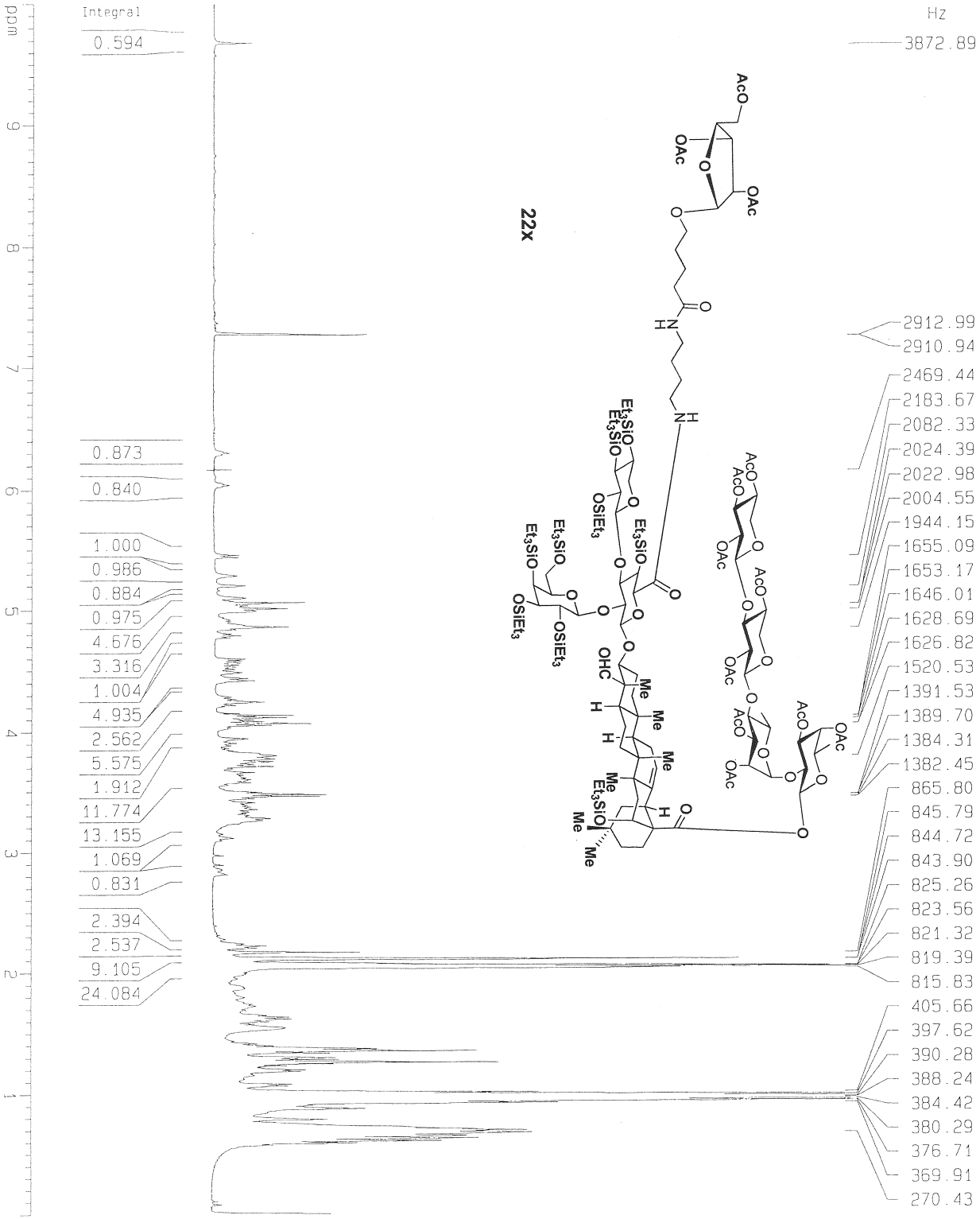
```

NAME          VII-12
EXPNO         702
PROCNO       1
Date_        20130618
Time         16.12
INSTRUM      spect
PROBHD       5 mm CPTCI 1H-
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           4096
DS           4
SWH          41666.668
FIDRES       0.635783
AQ           0.7864820
RG           64
DW           12.000
DE           20.000
TE           296.6
D1           4.19999981
D11          0.030000000
TD0          1

===== CHANNEL f1 =====
NUC1         13C
P1           61.00
PL1         91.30
PL1W        128.76077271
SFO1        176.0395312

===== CHANNEL f2 =====
CPDPRG2     waltz16
NUC2         1H
PCPD2       65.00
PL2         1.30
PL12        19.50
PL13        19.50
PL2W        6.88972235
PL12W       0.10428017
PL13W       0.10428017
SFO2        700.0228001
SI          131072
SF          176.0201727
WDW         EM
SSB         0
LB          1.00
GB          0
PC          1.40
  
```

PW-VI-50-C2



Current Data Parameters
 NAME PW-VI-50-C2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20130202
 Time 13.35
 INSTRUM drx400
 PRCBHND 5 mm Multinu
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.145 Hz
 FIDRES 0.125314 Hz
 AQ 3.9584243 sec
 RG 64
 DW 60.400 usec
 DE 5.00 usec
 TE 300.0 K
 D1 1.00000000 sec

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

NUC1 1H
 P1 6.50 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters

SI 131072
 SF 400.1300015 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPGCM 0.50000 ppm/cm
 HZCM 200.05500 Hz/cm

170.16
170.02
169.85
169.34
105.69
100.69
100.61
81.53
80.14
77.18
77.00
76.82
69.91
68.74
67.35
63.33
54.02
41.68
39.93
39.11
36.21
30.48
28.77
26.35
24.41
22.53
20.91
20.86
20.78
20.76
20.73
20.66
20.58
20.54
20.49
17.66
17.55
15.91
15.90
7.52
7.42
7.22
7.13
7.12
7.09
7.00
6.82
6.78
5.84
5.69
5.46
5.38
5.31
5.27
5.24
4.99
4.47
-0.02

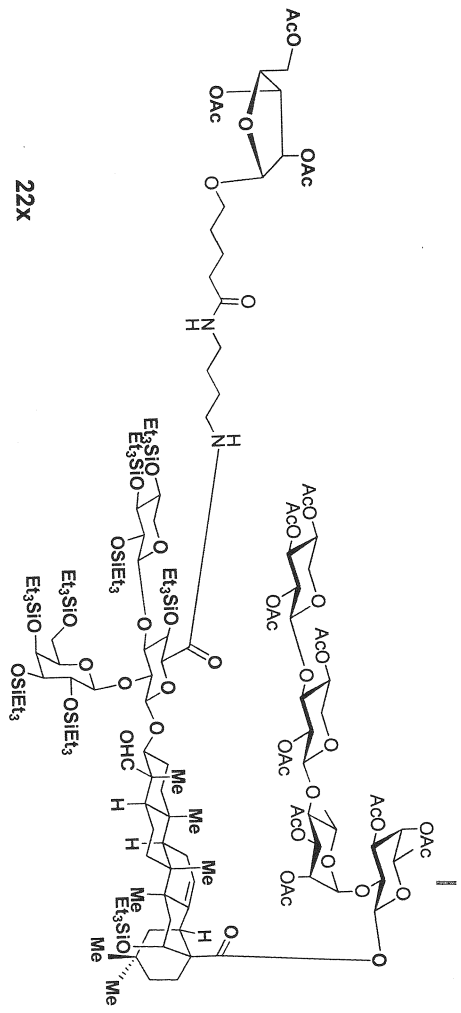
Current Data Parameters
NAME DD-3-123-C13
EXENO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151209
Time 16.20
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
DS 20657
NS 4
SWH 41666.668 Hz
FIDRES 0.635793 Hz
AQ 0.7864320 sec
RG 256
DWM 12.000 usec
DE 20.00 usec
TE 300.0 K
T1 2.00000000 sec
D11 0.103000000 sec
TD0 1

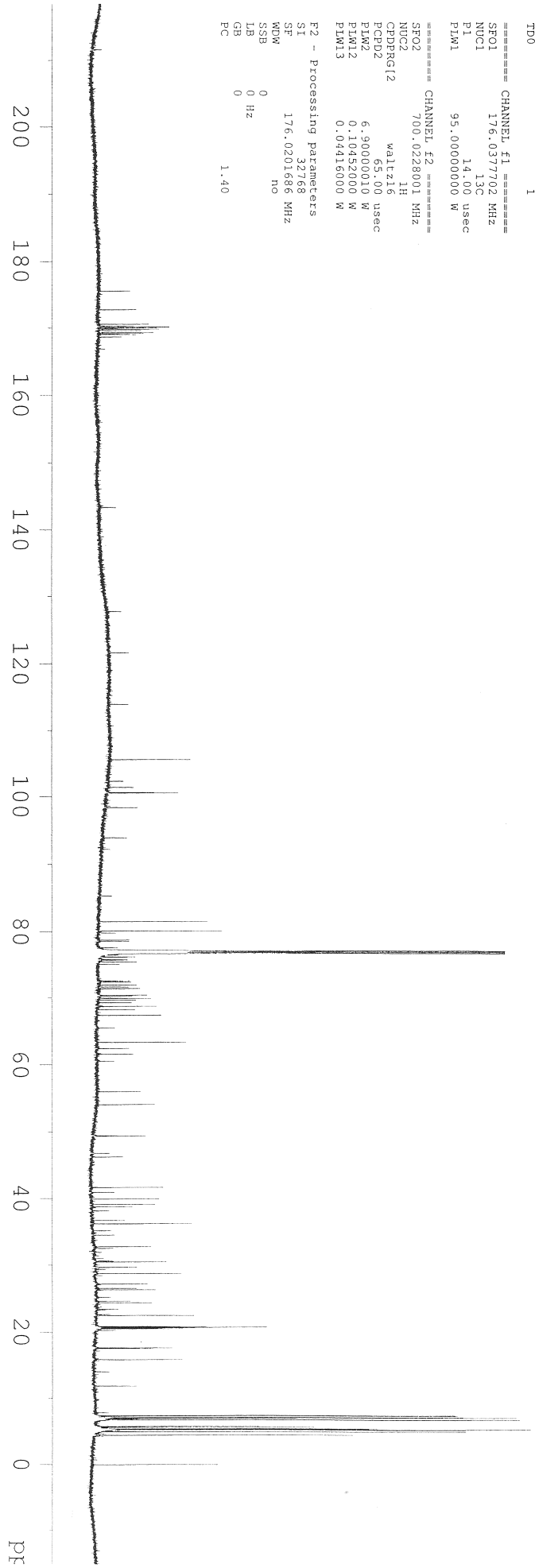
==== CHANNEL f1 =====
SF01 176.0377702 MHz
NUC1 13C
P1 14.00 usec
PLW1 95.000000000 W

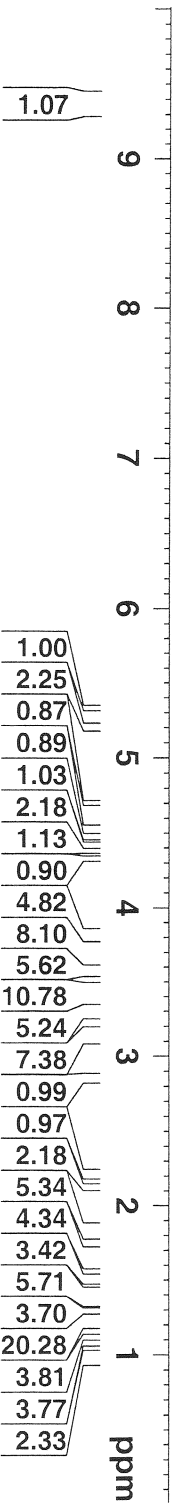
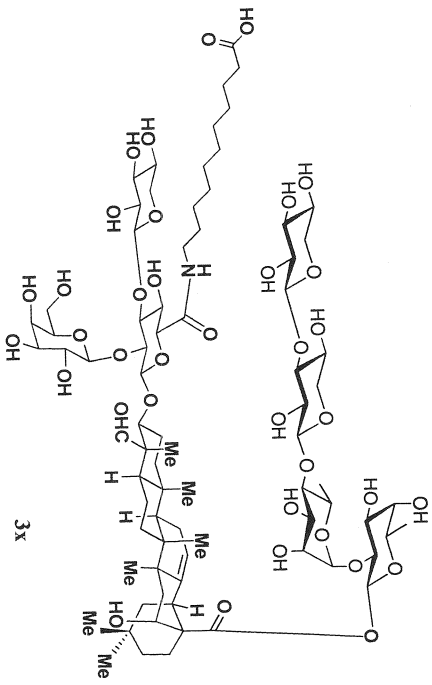
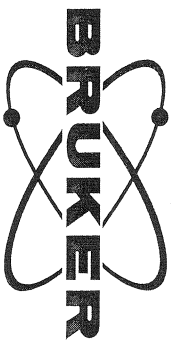
==== CHANNEL f2 =====
SF02 700.0228001 MHz
NUC2 1H
P2 1.40 usec
PLW2 6.900000010 W
PLW12 0.10452000 W
PLW13 0.04416000 W

F2 - Processing parameters
SI 32768
SF 176.0201686 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40



22X

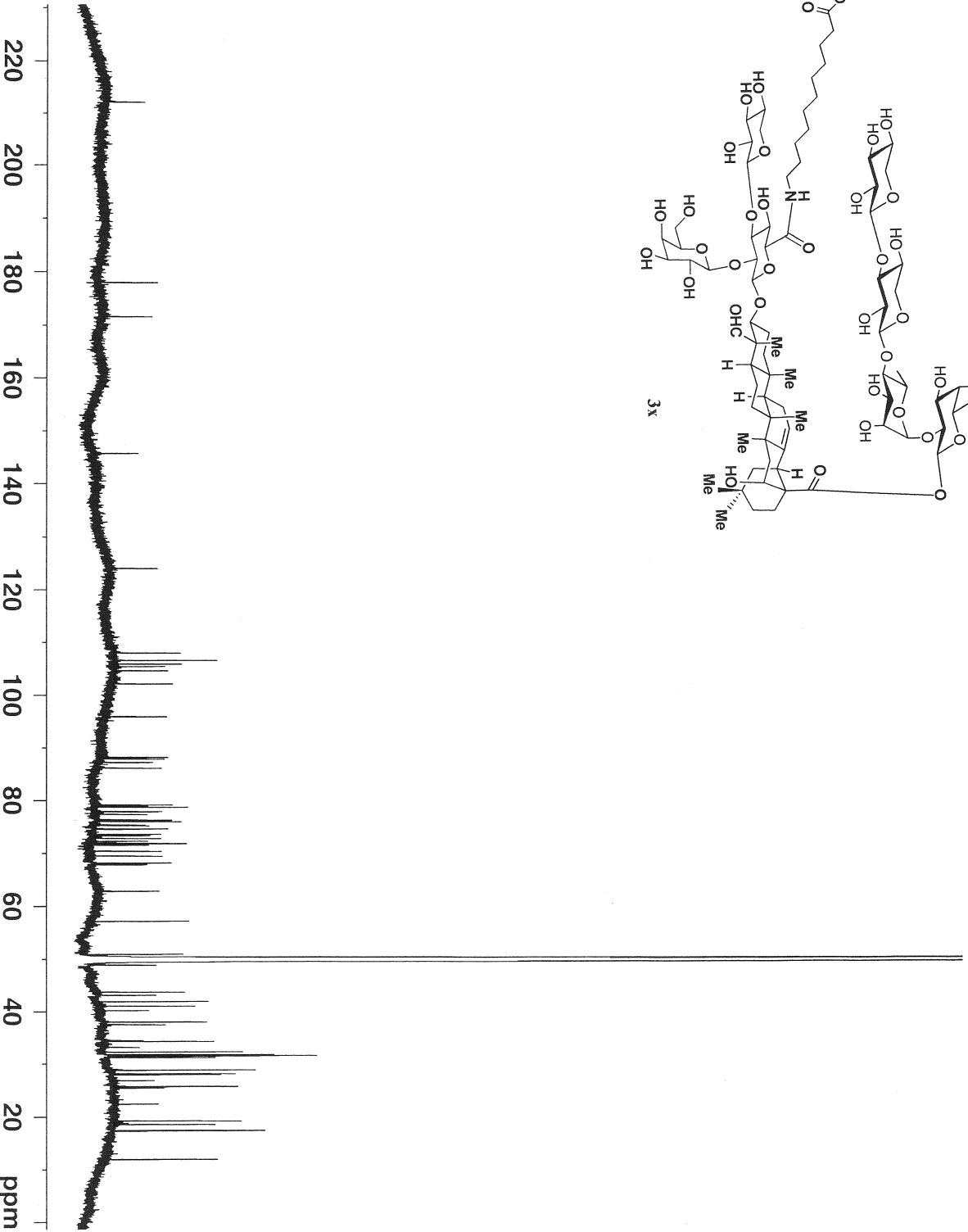
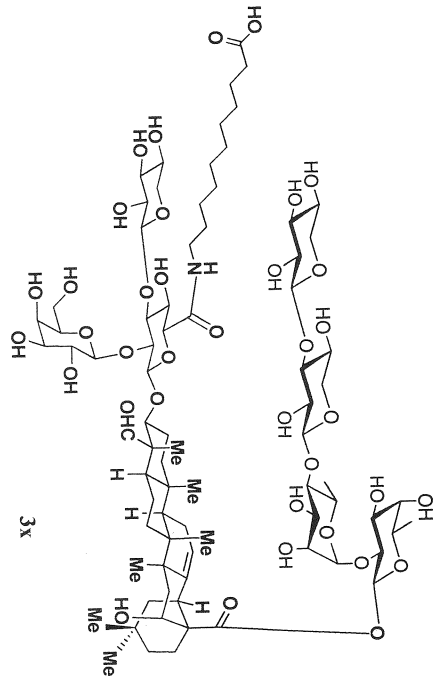




```

NAME PW-VIII-14-2
EXPNO 701
PROCNO 1
Date_ 20131028
Time 11.48
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 64
DS 0
SWH 8417.509 Hz
FIDRES 0.128441 Hz
AQ 3.8928883 sec
RG 16
DW 59.400 use
DE 6.50 use
TE 298.0 K
D1 1.00000000 sec
TD0

===== CHANNEL f1 =====
NUC1 1H
P1 8.00 use
PL1 1.30 dB
PL1W 6.88972235 W
SF01 700.0240601 MHz
SI 65536
SF 700.0200852 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
  
```

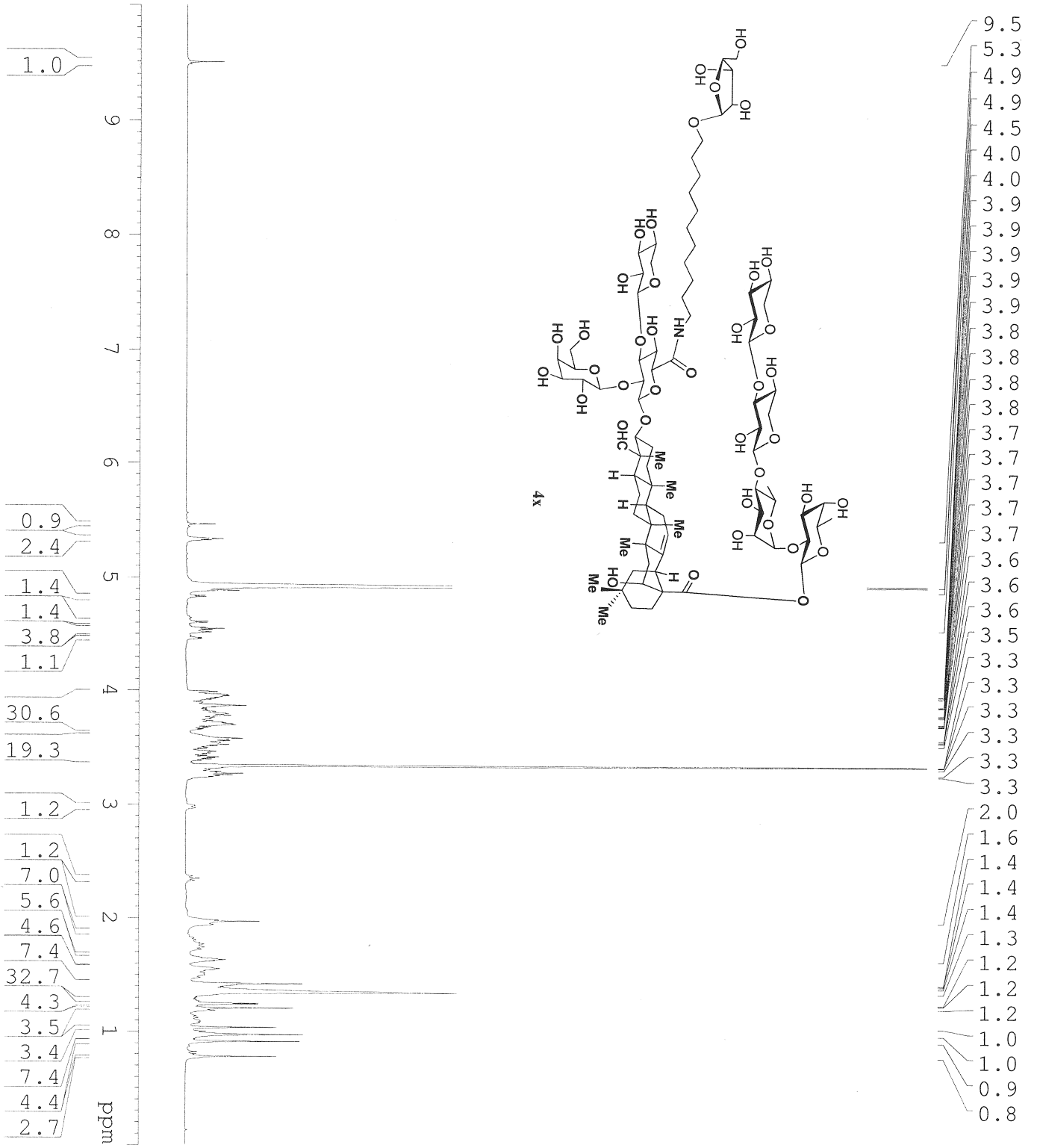


```

NAME          PW-VIII-14-2
EXPNO         702
PROCNO        1
Date_         20131029
Time         0.04
INSTRUM       spect
PROBHD        5 mm CP1H-
PULPROG       zgpg30
TD            65536
SOLVENT       MeOD
NS            8192
DS            8
SWH           41666.668 Hz
FIDRES       0.635783 Hz
AQ           0.7864820 sec
RG           256
DW           12.000 usec
DE           6.50 usec
TE           298.0 K
D1           2.20000005 sec
D11          0.03000000 sec
TD0          1

===== CHANNEL F1 =====
NUC1          13C
P1           14.00 usec
PL1          0.00 dB
PL1W         95.45168304 W
SFO1         176.0395312 MHz

===== CHANNEL F2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2        65.00 usec
PL2          1.30 dB
PL12         19.50 dB
PL13         19.50 dB
PL2W         6.88972235 W
PL12W        0.10428017 W
PL13W        0.10428017 W
SFO2         700.0228001 MHz
SI           32768
SF           176.0197706 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
  
```



Current Data Parameters
 NAME PW-IX-2H
 EXPNO 1
 PROCNO 1

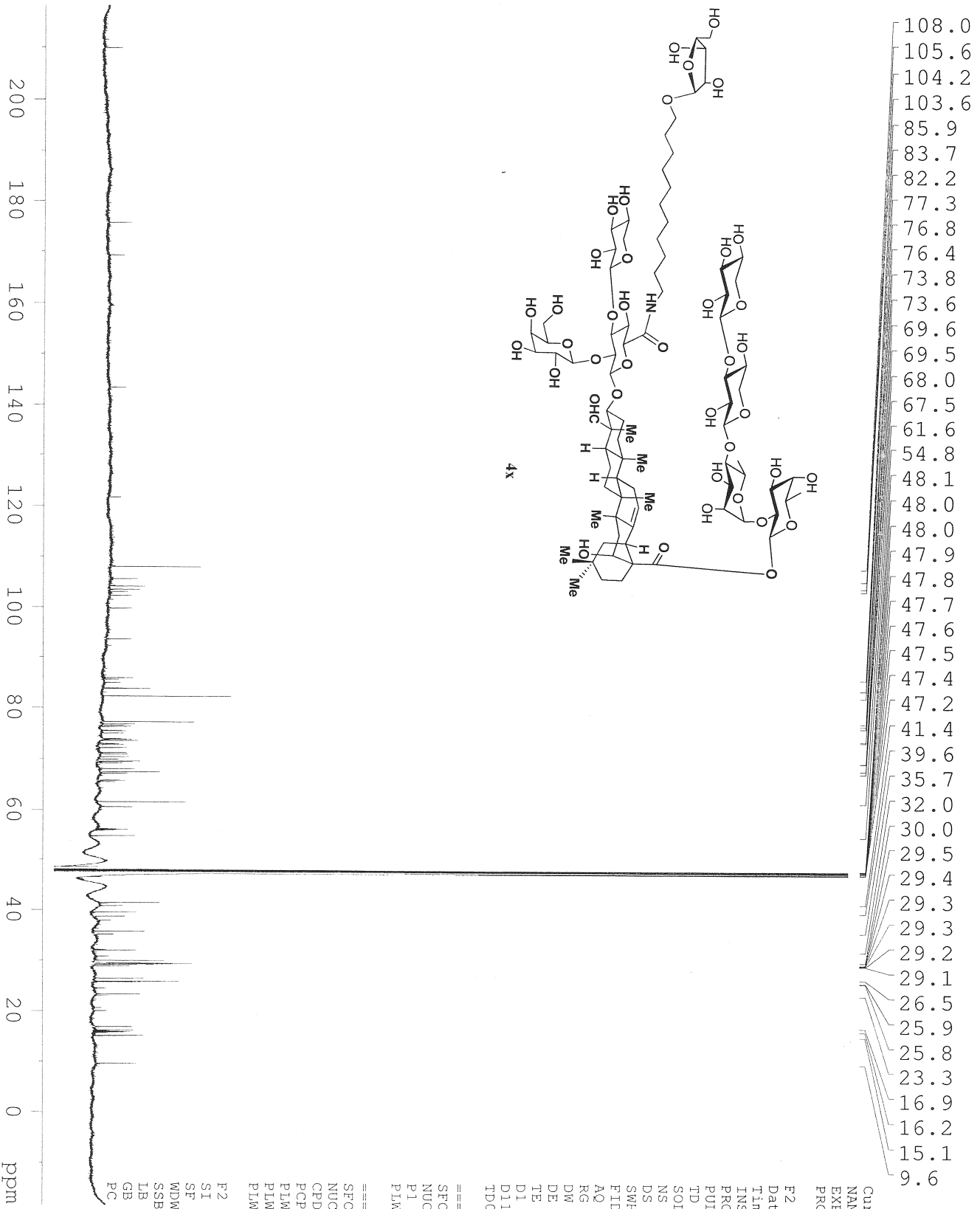
F2 - Acquisition Parameters
 Date_ 20160304
 Time 14.09

INSTRUM spect
 PROBHD 5 mm CPTCI 1H-
 PULPROG zg30
 TD 65536
 SOLVENT MeOD
 NS 16
 DS 2

SWH 14097.744 Hz
 FIDRES 0.215115 Hz
 AQ 2.3243434 sec
 RG 25.4
 DW 35.467 usec
 DE 10.00 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL F1 =====
 SFO1 700.0243229 MHz
 NUCL 1H
 P1 8.00 usec
 PLWI 6.90000010 W

F2 - Processing parameters
 SI 65536
 SF 700.0199922 MHz
 WDW no
 SSB 0 Hz
 LB 0
 GB 0
 PC 1.00



- 108.0
- 105.6
- 104.2
- 103.6
- 85.9
- 83.7
- 82.2
- 77.3
- 76.8
- 76.4
- 73.8
- 73.6
- 69.6
- 69.5
- 68.0
- 67.5
- 61.6
- 54.8
- 48.1
- 48.0
- 48.0
- 47.9
- 47.8
- 47.7
- 47.6
- 47.5
- 47.4
- 47.2
- 41.4
- 39.6
- 35.7
- 32.0
- 30.0
- 29.5
- 29.4
- 29.3
- 29.3
- 29.2
- 29.1
- 26.5
- 25.9
- 25.8
- 23.3
- 16.9
- 16.2
- 15.1
- 9.6

Current Data Parameters
 NAME PW-IX-2C13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160304
 Time 14.33

INSTRUM spect
 PROBHD 5 mm CPTCI 1H-
 PULPROG zgpg30
 TD 65536
 SOLVENT MeOD
 NS 23739
 DS 4

SWH 41666.668 Hz
 FIDRES 0.635783 Hz
 AQ 0.7864320 sec
 RG 256

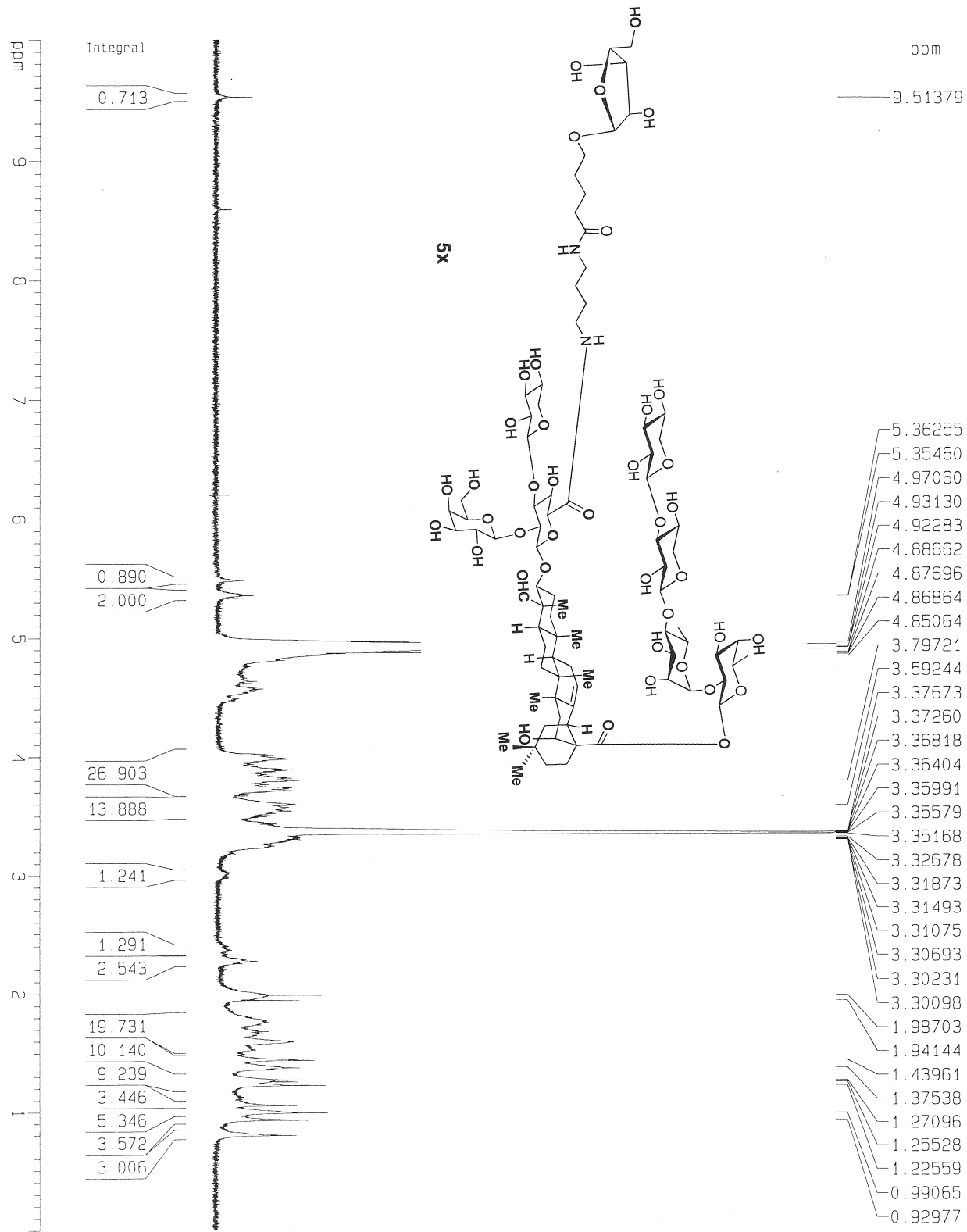
DW 12.000 usec
 DE 18.00 usec
 TE 298.05 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 ID0 1

CHANNEL F1
 SFO1 176.0377702 MHz
 NUC1 13C
 P1 14.00 usec
 PLW1 95.00000000 W

CHANNEL F2
 SFO2 700.0228001 MHz
 NUC2 1H
 CPDPRG12 waltz16
 PCPD2 65.00 usec
 PLW2 6.90000010 W
 PLW12 0.10452000 W
 PLW13 0.04416000 W

F2 - Processing parameters
 SI 32768
 SF 176.0201690 MHz
 WDW no
 SSB 0 Hz
 LB 0
 GB 0
 PC 1.40

PW-IX-1H



Current Data Parameters
 NAME PW-IX-1H
 EXPNO 1
 PROCNO 1

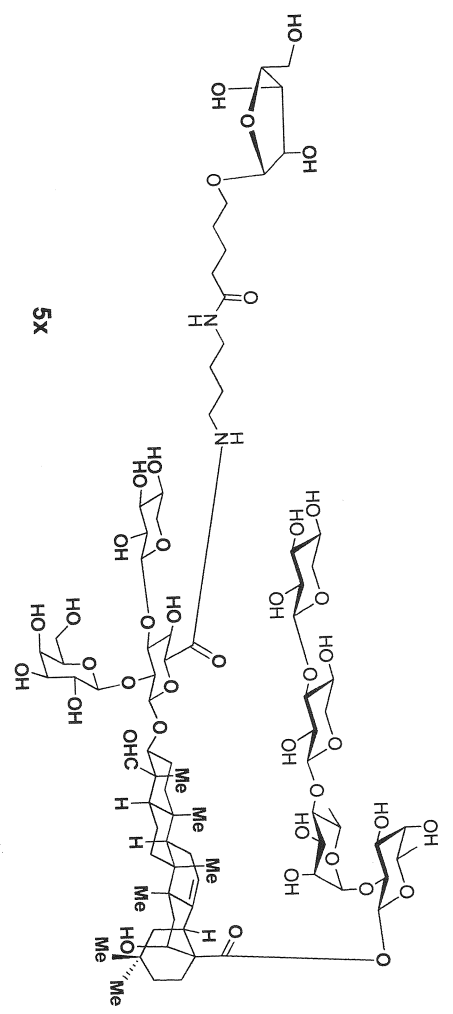
F2 - Acquisition Parameters
 Date_ 20160223
 Time 10.54
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 65536
 SOLVENT MeOH
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 512
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 6.50 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing Parameters
 S1 131072
 SF 400.1299870 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 dpm
 F1 4001.39 Hz
 F2P 0.000 dpm
 F2 0.00 Hz
 PPMCM 0.50000 dpm/cm
 HZCM 200.06499 Hz/cm

174.681
169.429
108.060
105.603
104.168
103.560
103.140
83.905
82.205
77.323
76.343
75.598
75.509
75.012
74.018
73.929
73.823
73.627
71.231
70.983
70.432
69.953
69.545
68.036
67.149
66.950
65.853
65.782
65.498
61.676
56.073
48.123
47.962
47.840
47.718
47.595
47.473
47.352
47.232
38.447
35.358
35.181
28.613
26.221
25.720
15.123
9.621



Current Data Parameters
 NAME PW-IX-1C13
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160223
 Time 12.43
 INSTRUM spect
 PROBHD 5 mm CPTCI 1H-
 PULPROG zgpg30
 TD 65536
 SOLVENT MeOD
 NS 25634
 DS 4
 SWH 41666.668
 FIDRES 0.635783
 AQ 0.7864320
 RG 129.256
 DW 18.00
 DE 18.00
 TE 293.0
 D1 2.00000000
 D11 0.03000000
 TD0 1

==== CHANNEL f1 ====
 SF01 176.0377702
 NUC1 13C
 P1 14.00
 PLW1 95.00000000

==== CHANNEL f2 ====
 SF02 700.0228001
 NUC2 1H
 CPDPRG12 waltz16
 PCPD2 65.00
 PLW2 6.90000010
 PLW12 0.10452000
 PLW13 0.04416000

F2 - Processing Parameters
 SI 32768
 SF 176.0201690
 WDW EM
 SSB 0
 LB 1.00
 GB 0
 PC 1.00