

Supporting Information

Synthesis and biological activity of C-glycosides of KRN 7000 with novel ceramide residues

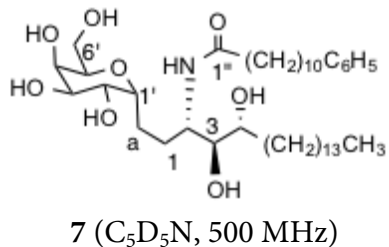
Ahmad S. Aliti,^a Xiaojing Ma,^b Lixing Zhang,^b Yi Ban,^b Richard W. Franck^a and David R. Mootoo^{a*}

^a*Department of Chemistry, Hunter College, 695 Park Avenue, New York, NY 10065 and The Graduate Center, CUNY, 365 Fifth Avenue, New York, NY 10016.* ^b*Department of Microbiology and Immunology, Weill Cornell Medicine, 1300 York Ave, NY, NY. 10065, USA.*

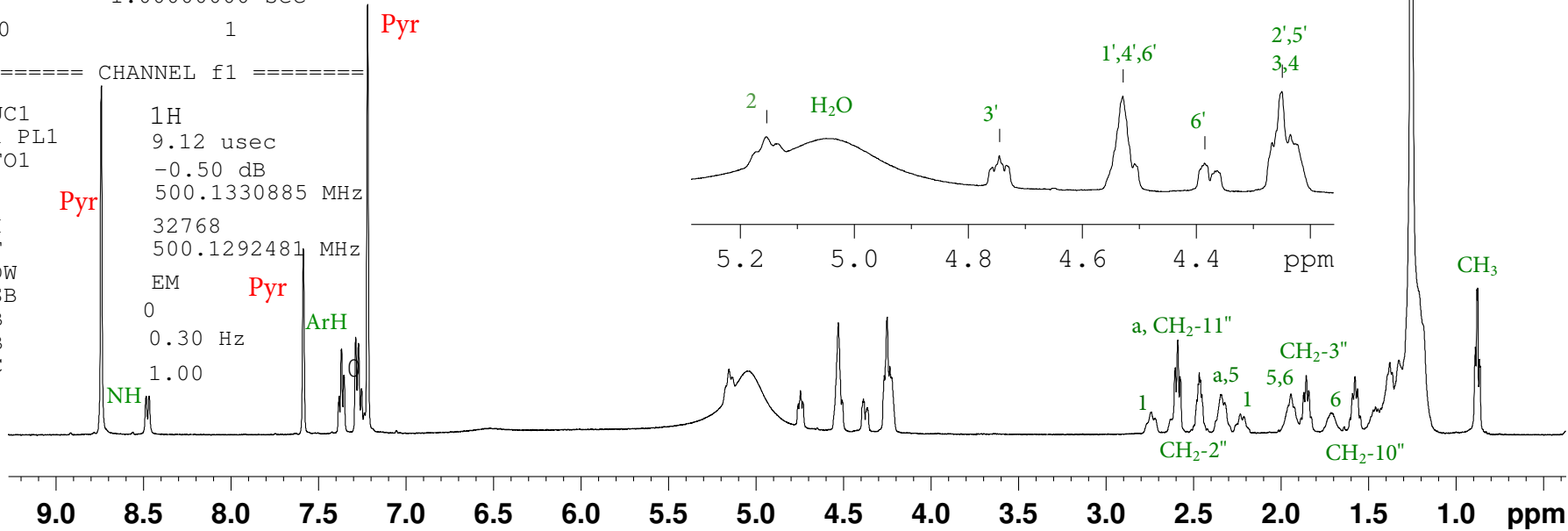
dmootoo@hunter.cuny.edu

NMR charts for compounds **7**, **8**, **9** and **10**. Data for **9** and **10** reproduced from reference 30.
PDF of cytokine assays

NAME 20130813
 EXPNO 1
 PROCNO 2
 Date_ 20130813
 Time 12.09
 INSTRUM spect
 PROBHD 5 mm CPDCH
 PULPROG 13C zg30
 TD 32768
 SOLVENT CDC13
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0448356 sec
 RG 40.3
 DW 62.400 usec
 DE 6.00 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1



===== CHANNEL f1 =====
 NUC1 1H
 P1 PL1 9.12 usec
 SFO1 -0.50 dB
 500.1330885 MHz
 SI 32768
 SF 500.1292481 MHz
 WDW EM
 SSB Pyr
 LB 0
 GB 0.30 Hz
 PC 1.00



8.738
8.482
8.465

7.586
7.367
7.355
7.287
7.271
7.219

5.154
4.745
4.528
4.385
4.249

2.740
2.603
2.588
2.575
2.466
2.342
1.941
1.854
1.576
1.377
1.256
0.874
0.865

CH₂ x 17

3.32
0.93

1.82
1.67
6.17

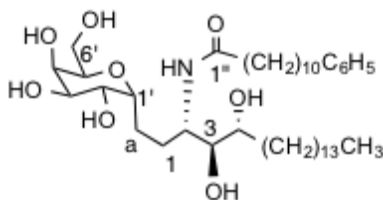
1.42

18.33
2.88
1.13
3.55

1.00
2.48
1.77
1.74
1.01
3.72
1.23
1.82
30.13
2.97

8.467
8.444
7.381
7.362
7.344
7.282
7.264
7.246
5.143
4.753
4.739
4.731
4.717
4.536
4.521
4.509
4.491
4.386
4.375
4.358
4.347
4.264
4.256
4.242
4.226
2.602
2.584
2.564
2.479
2.473
2.460
2.455
2.441
2.335
2.322
2.308
1.938
1.927
1.909
1.866
1.848
1.829
1.632
1.589
1.571
1.552
1.322
1.299
1.252
1.201
1.180
0.888
0.872
0.854

NAME Ph-Gal-Cer
EXPNO 1
PROCNO 1
Date_ 20130906
Time 18.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT Pyr
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 135.71
DW 60.800 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1

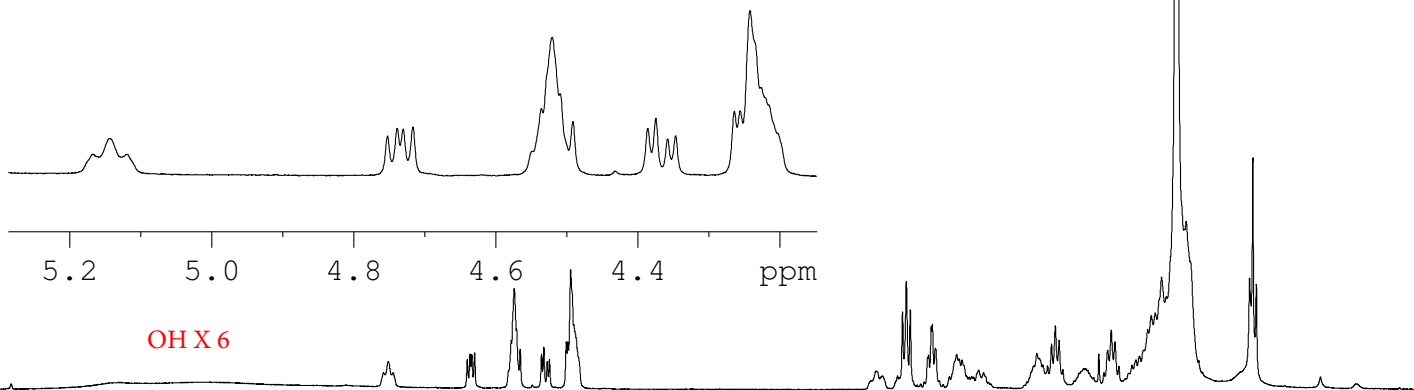


7 (C₅D₅N, 400 MHz)

sample run under anhydrous conditions

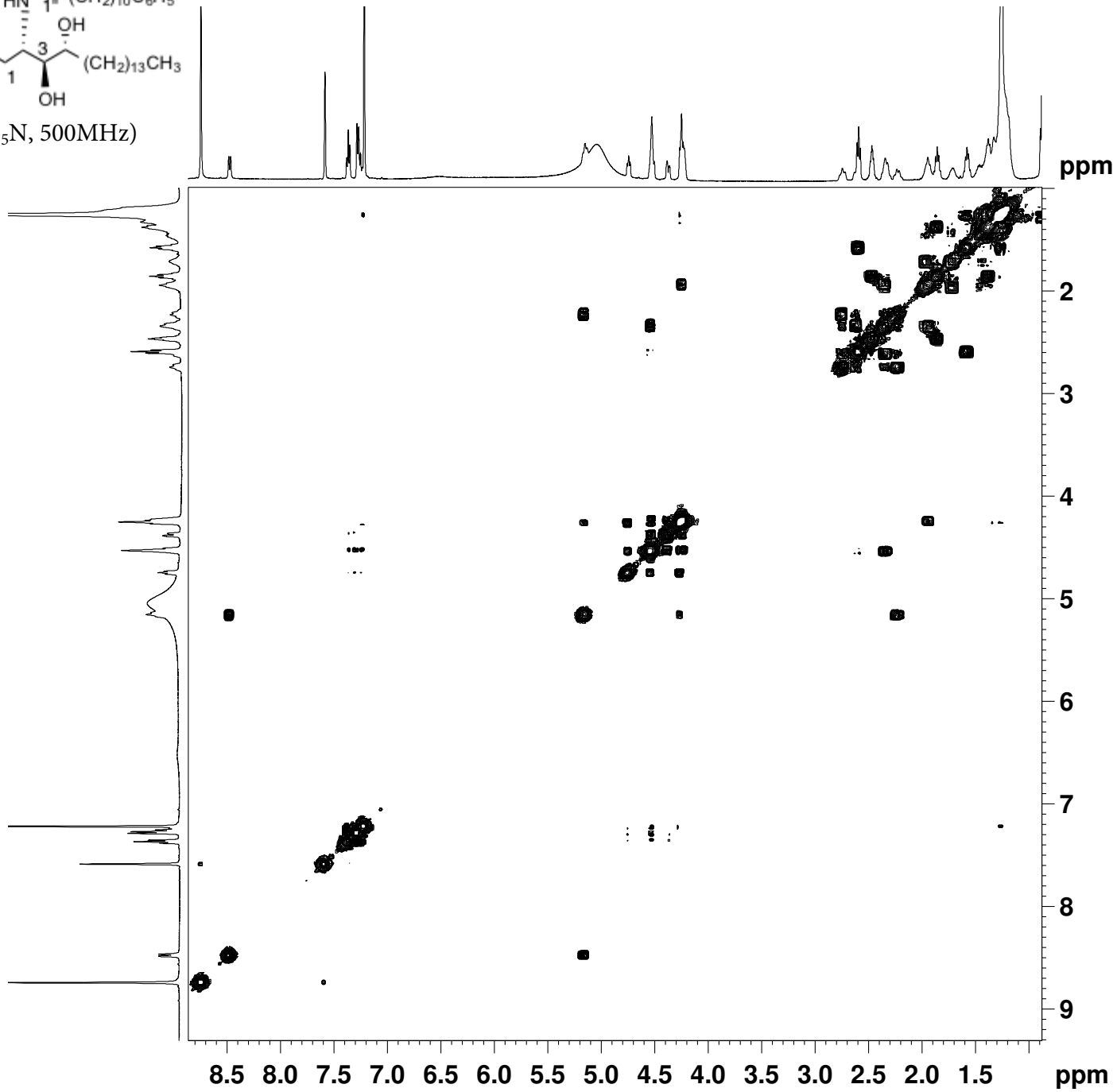
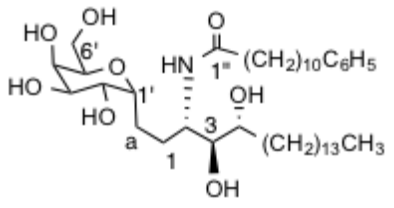
5.167
5.143
5.120
4.753
4.739
4.731
4.717
4.536
4.521
4.509
4.491
4.386
4.375
4.358
4.347
4.264
4.256

==== CHANNEL f1 =====
NUC1 1H
P1 11.00 usec
SI 65536
SF 400.1799934 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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

0.91
5.03
6.50
1.03
1.00
3.01
1.02
3.89
0.90
2.77
1.99
3.07
4.20
1.45
2.36
39.80
4.66



```

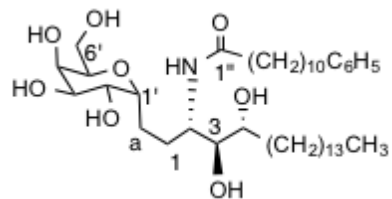
NAME          20130813
EXPNO         2
PROCNO        1
Date_         20130813
Time          12.09
INSTRUM       spect
PROBHD        5 mm CPDCH 13C
PULPROG       cosygpgqf
TD            2048
SOLVENT       CDC13
NS            1
DS            8
SWH           6666.667 Hz
FIDRES        3.255208 Hz
AQ            0.1537250 sec
RG            22.6
DW            75.000 usec
DE            6.00 usec
TE            298.0 K
d0            0.00000300 sec
D1            1.48689198 sec
d13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00015000 sec
  
```

```

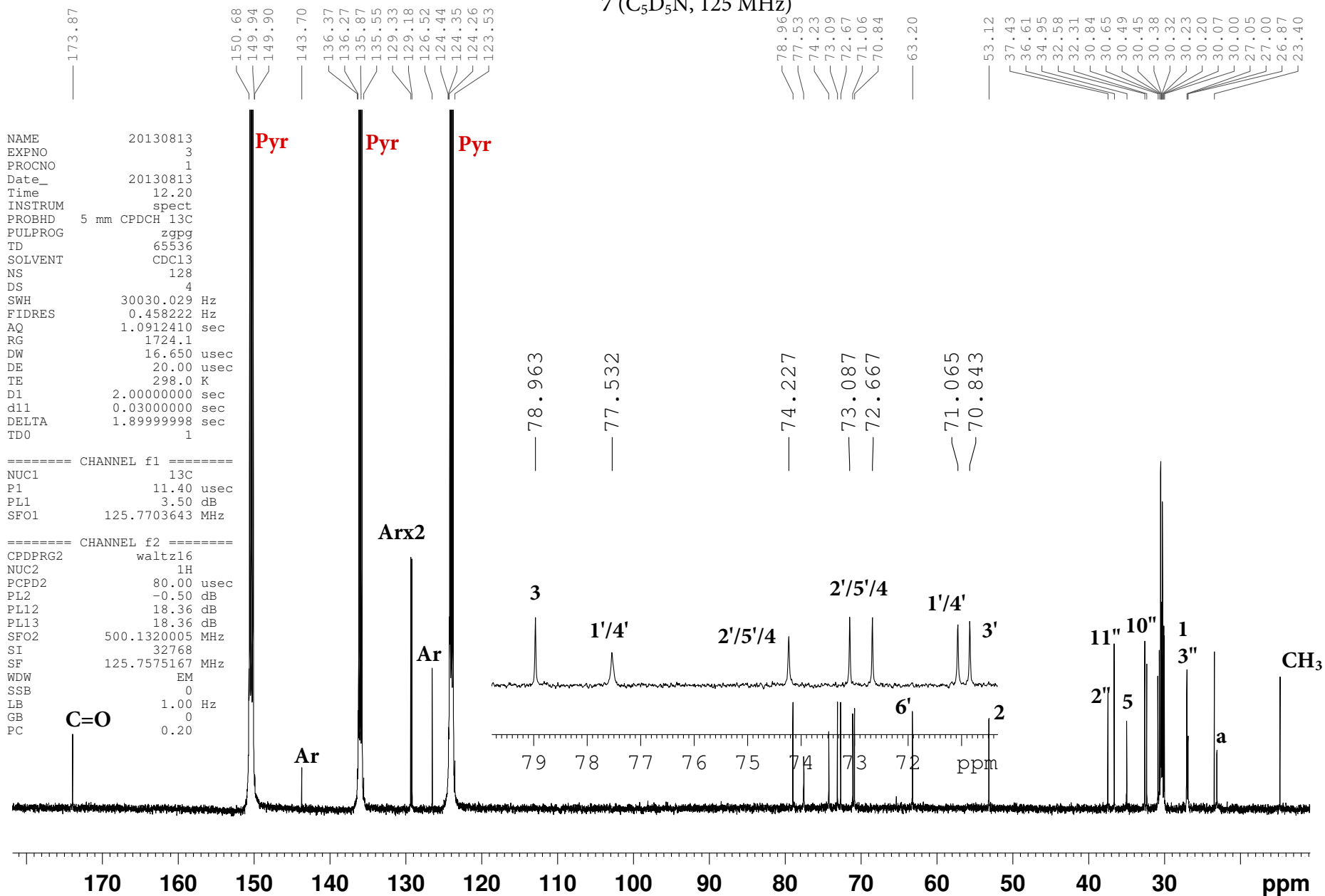
===== CHANNEL f1 =====
NUC1          1H
P0            9.12 usec
P1            9.12 usec
PL1           -0.50 dB
SFO1          500.1330069 MHz
  
```

```

===== GRADIENT CHANNEL =====
GPNAM1       SINE.100
GP21         10.00 %
P16          1000.00 usec
ND0          1
TD           128
SFO1         500.133 MHz
FIDRES       52.083332 Hz
SW           13.330 ppm
FnMODE       QF
SI           1024
SF           500.1292440 MHz
WDW          SINE
SSB          0
LB           0.00 Hz
GB           0
PC           1.40
SI           1024
MC2          QF
SF           500.1292441 MHz
WDW          SINE
SSB          0
LB           0.00 Hz
GB           0
  
```



7 (C₅D₅N, 125 MHz)



```

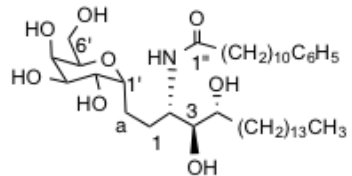
NAME          20130813
EXPNO         3
PROCNO        1
Date_         20130813
Time          12.20
INSTRUM       spect
PROBHD        5 mm CPDCH 13C
PULPROG       zgpg
TD            65536
SOLVENT       CDCl3
NS            128
DS            4
SWH           30030.029 Hz
FIDRES        0.458222 Hz
AQ            1.0912410 sec
RG            1724.1
DW            16.650 usec
DE            20.00 usec
TE            298.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0           1
  
```

```

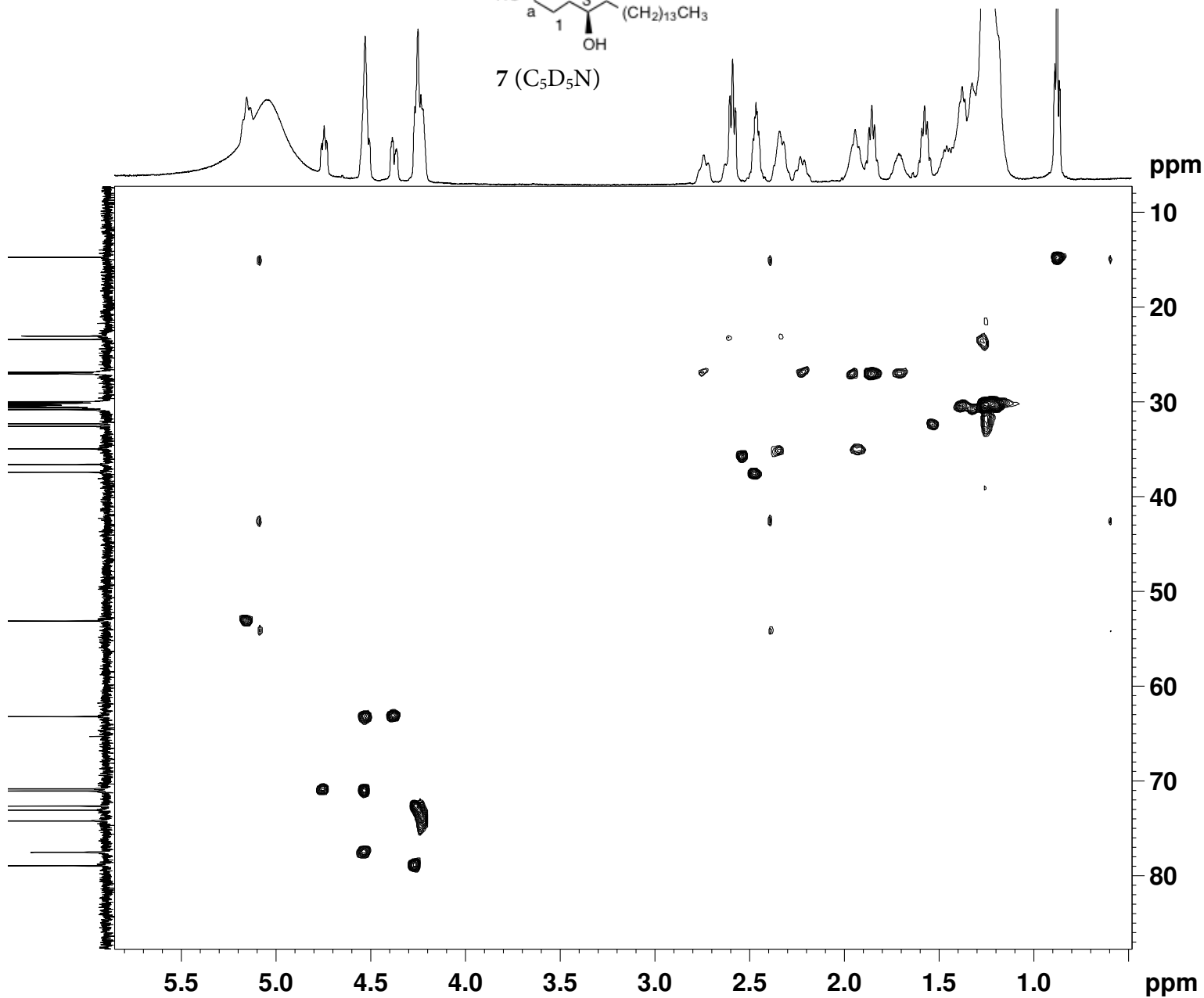
===== CHANNEL f1 =====
NUC1          13C
P1            11.40 usec
PL1           3.50 dB
SFO1          125.7703643 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -0.50 dB
PL12          18.36 dB
PL13          18.36 dB
SFO2          500.1320005 MHz
SI            32768
SF            125.7575167 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            0.20
  
```



7 (C₅D₅N)



```

NAME          20130812
EXPNO         4
PROCNO        1
Date_         20130812
Time          15.40
INSTRUM       spect
PROBHD        5 mm CPDCH 13C
PULPROG       hsqcetgp
TD            1024
SOLVENT       Pyr
NS            10
DS            16
SWH           6666.667 Hz
FIDRES        6.510417 Hz
AQ            0.0769250 sec
RG            29193
DW            75.000 usec
DE            6.00 usec
TE            298.0 K
CNST2         145.0000000
d0            0.00000300 sec
D1            1.20000005 sec
d4            0.00172414 sec
d11           0.030000000 sec
d13           0.00000400 sec
D16           0.00020000 sec
DELTA         0.00122424 sec
DELTA1        0.00071614 sec
IN0           0.00001990 sec
ST1CNT        0
ZGOFPTS

```

```

===== CHANNEL f1 =====
NUC1           1H
P1             9.12 usec
p2            18.24 usec
P28           2000.00 usec
PL1           -0.50 dB
SFO1          500.1330069 MHz

```

```

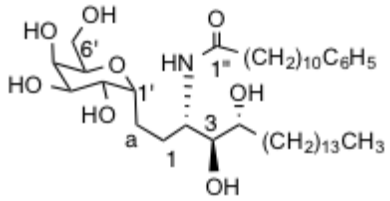
===== CHANNEL f2 =====
CPDPRG2       garp
NUC2           13C
P3            11.40 usec
p4            22.80 usec
PCPD2         70.00 usec
PL2           3.50 dB
PL12          19.26 dB
SFO2          125.7678496 MHz

```

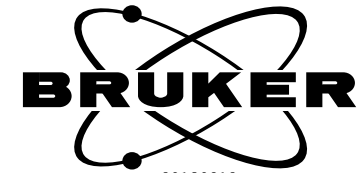
```

===== GRADIENT CHANNEL =====
GPNAM1        SINE.100
GPNAM2        SINE.100
GPZ1          80.00 %
GPZ2          20.10 %
P16           1000.00 usec
ND0           2
TD            128
SFO1          125.7678 MHz
FIDRES        196.293976 Hz
SW            199.778 ppm
FnMODE        Echo-Antiecho
SI            1024
SF            500.1299792 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           echo-antiecho
SF            125.7576890 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0

```



7 (C₅D₅N)



```

NAME      20130812
EXPNO     4
PROCNO    1
Date_     20130812
Time      15.40
INSTRUM   spect
PROBHD    5 mm CPDCH 13C
PULPROG   hsqcetgp
TD         1024
SOLVENT   Pyr
NS         10
DS         16
SWH        6666.667 Hz
FIDRES     6.510417 Hz
AQ         0.0769250 sec
RG         29193
DW         75.000 usec
DE         6.00 usec
TE         298.0 K
CNST2     145.0000000
d0         0.00000300 sec
D1         1.20000005 sec
d4         0.00172414 sec
d11        0.03000000 sec
d13        0.00000400 sec
D16        0.00020000 sec
DELTA     0.00122424 sec
DELTA1    0.00071614 sec
INO       0.00001990 sec
ST1CNT    0
ZGPTNS
  
```

```

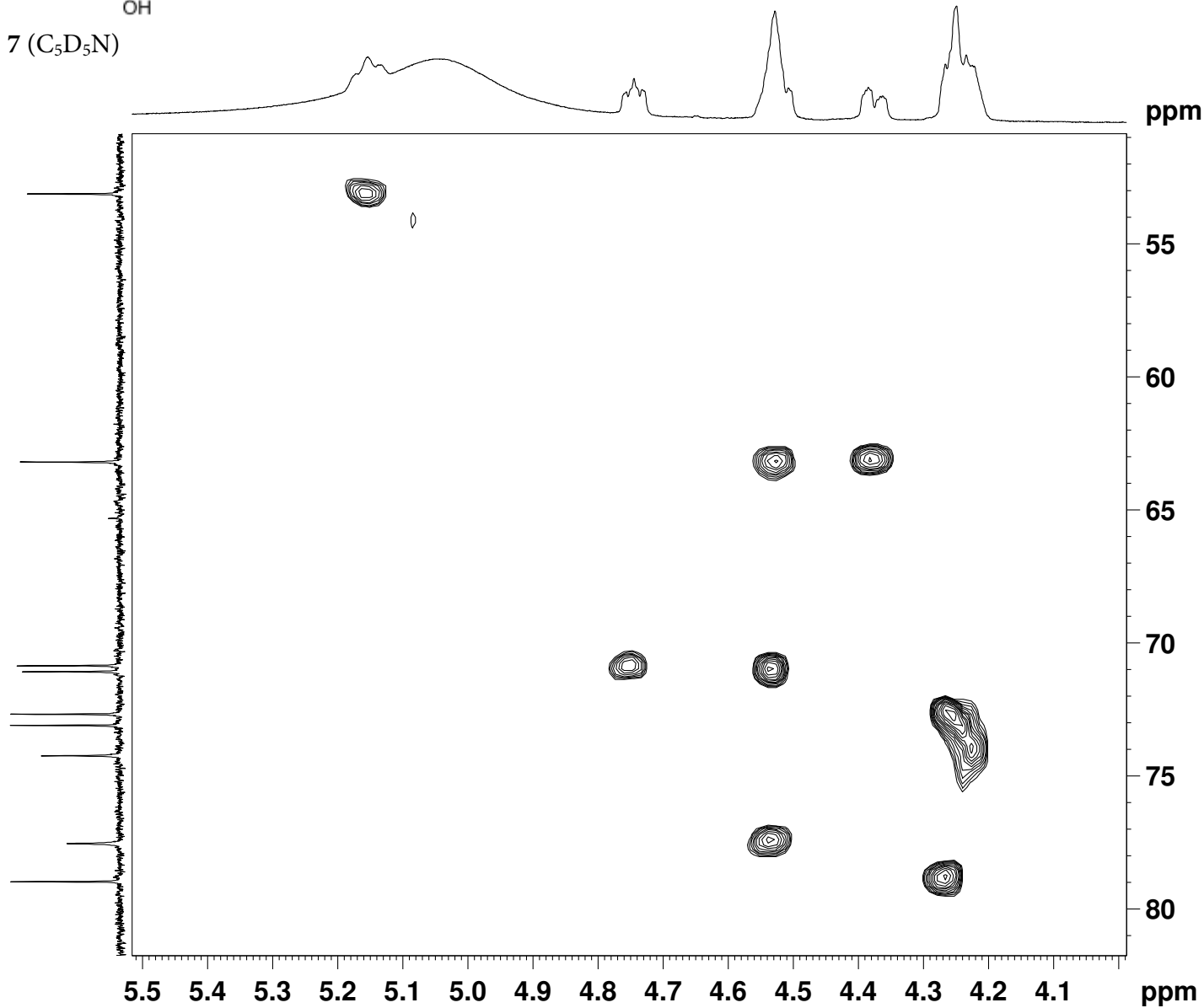
===== CHANNEL f1 =====
NUC1      1H
P1        9.12 usec
p2        18.24 usec
P28       2000.00 usec
PL1       -0.50 dB
SFO1      500.1330069 MHz
  
```

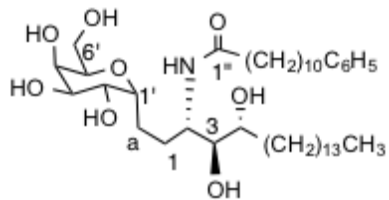
```

===== CHANNEL f2 =====
CPDPRG2   garp
NUC2      13C
P3        11.40 usec
p4        22.80 usec
PCPD2     70.00 usec
PL2       3.50 dB
PL12      19.26 dB
SFO2      125.7678496 MHz
  
```

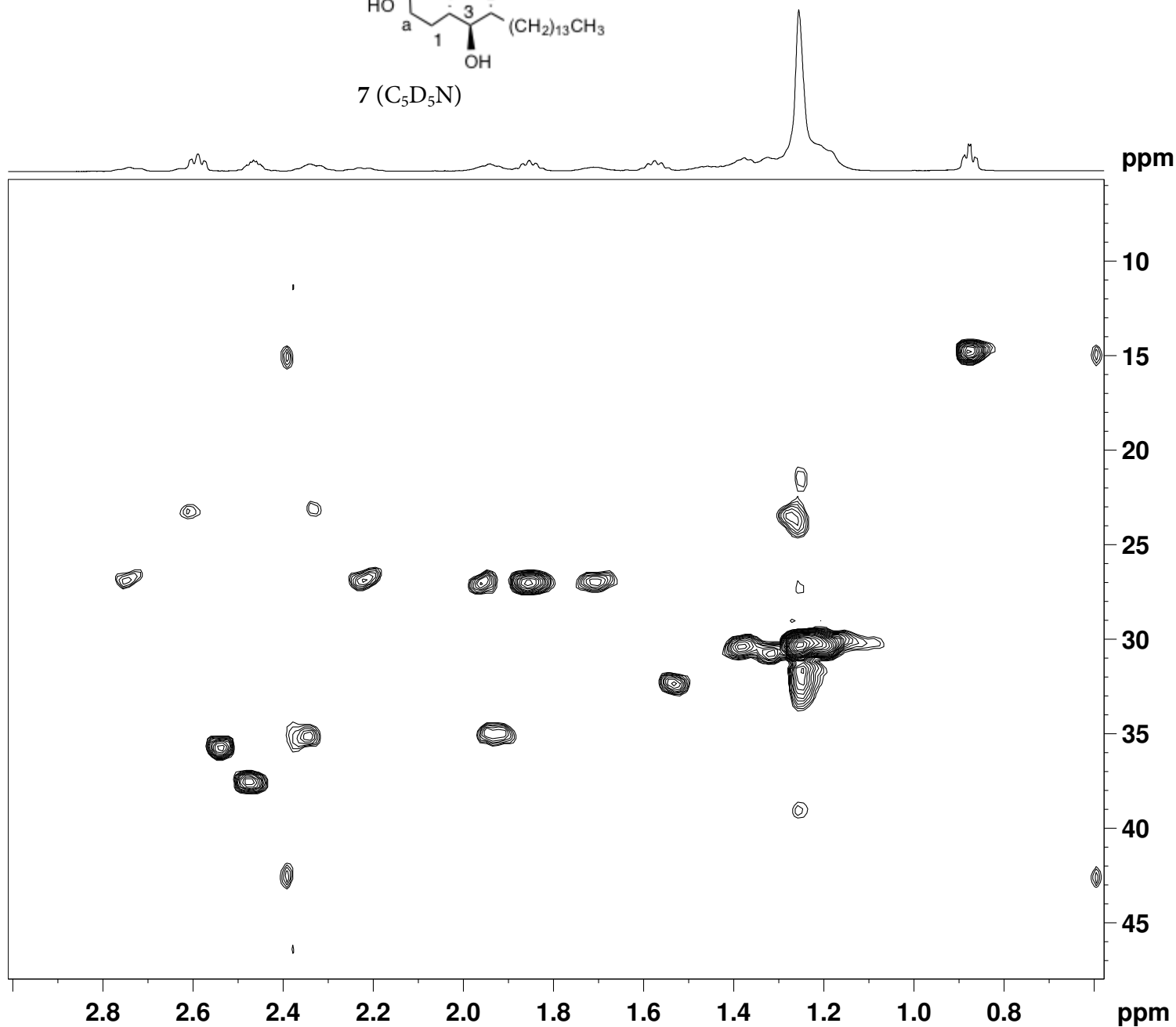
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===== GRADIENT CHANNEL =====
GPNAM1    SINE.100
GPNAM2    SINE.100
GPZ1      80.00 %
GPZ2      20.10 %
P16       1000.00 usec
ND0       2
TD         128
SFO1      125.7678 MHz
FIDRES     196.293976 Hz
SW         199.778 ppm
FnMODE    Echo-Antiecho
SI         1024
SF         500.1299792 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.40
SI         1024
MC2       echo-antiecho
SF         125.7576890 MHz
WDW        QSINE
SSB        2
LB         0.00 Hz
GB         0
  
```





7 (C₅D₅N)



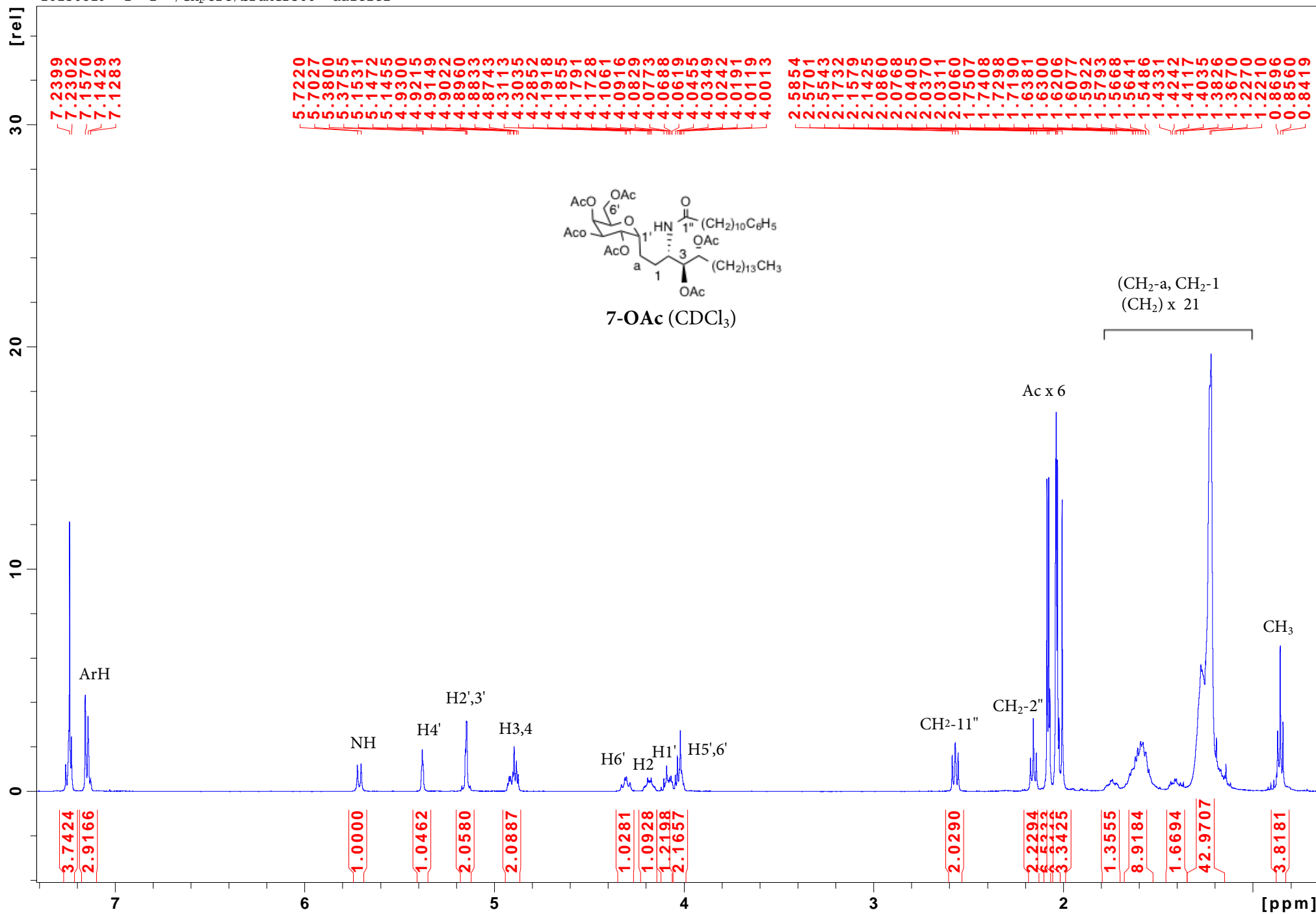
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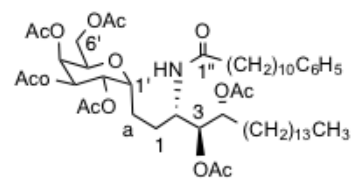
NAME          20130812
EXPNO         4
PROCNO        1
Date_         20130812
Time          15.40
INSTRUM       spect
PROBHD        5 mm CPDCH 13C
PULPROG       hsqcetgp
TD            1024
SOLVENT       Pyr
NS            10
DS            16
SWH           6666.667 Hz
FIDRES        6.510417 Hz
AQ            0.0769250 sec
RG            29193
DW            75.000 usec
DE            6.00 usec
TE            298.0 K
CNST2         145.000000
d0            0.00000300 sec
d1            1.20000005 sec
d4            0.00172414 sec
d11           0.03000000 sec
d13           0.00000400 sec
D16           0.00020000 sec
DELTA         0.00122424 sec
DELTA1        0.00071614 sec
IN0           0.00001990 sec
ST1CNT        0
ZGPTNS

===== CHANNEL f1 =====
NUC1           1H
P1             9.12 usec
p2            18.24 usec
P28           2000.00 usec
PL1            -0.50 dB
SFO1          500.1330069 MHz

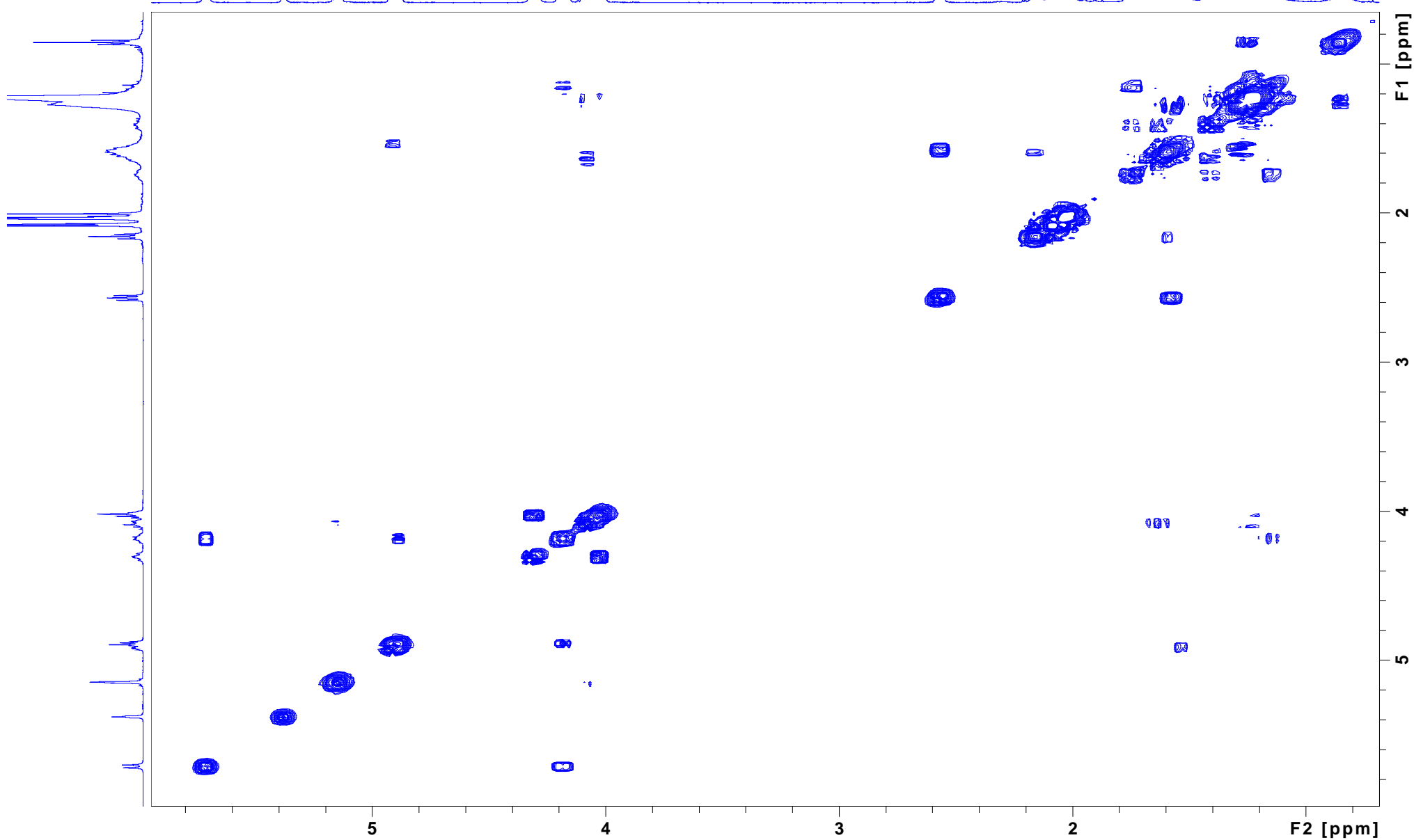
===== CHANNEL f2 =====
CPDPRG2        garp
NUC2           13C
P3            11.40 usec
p4            22.80 usec
PCPD2         70.00 usec
PL2            3.50 dB
PL12          19.26 dB
SFO2          125.7678496 MHz

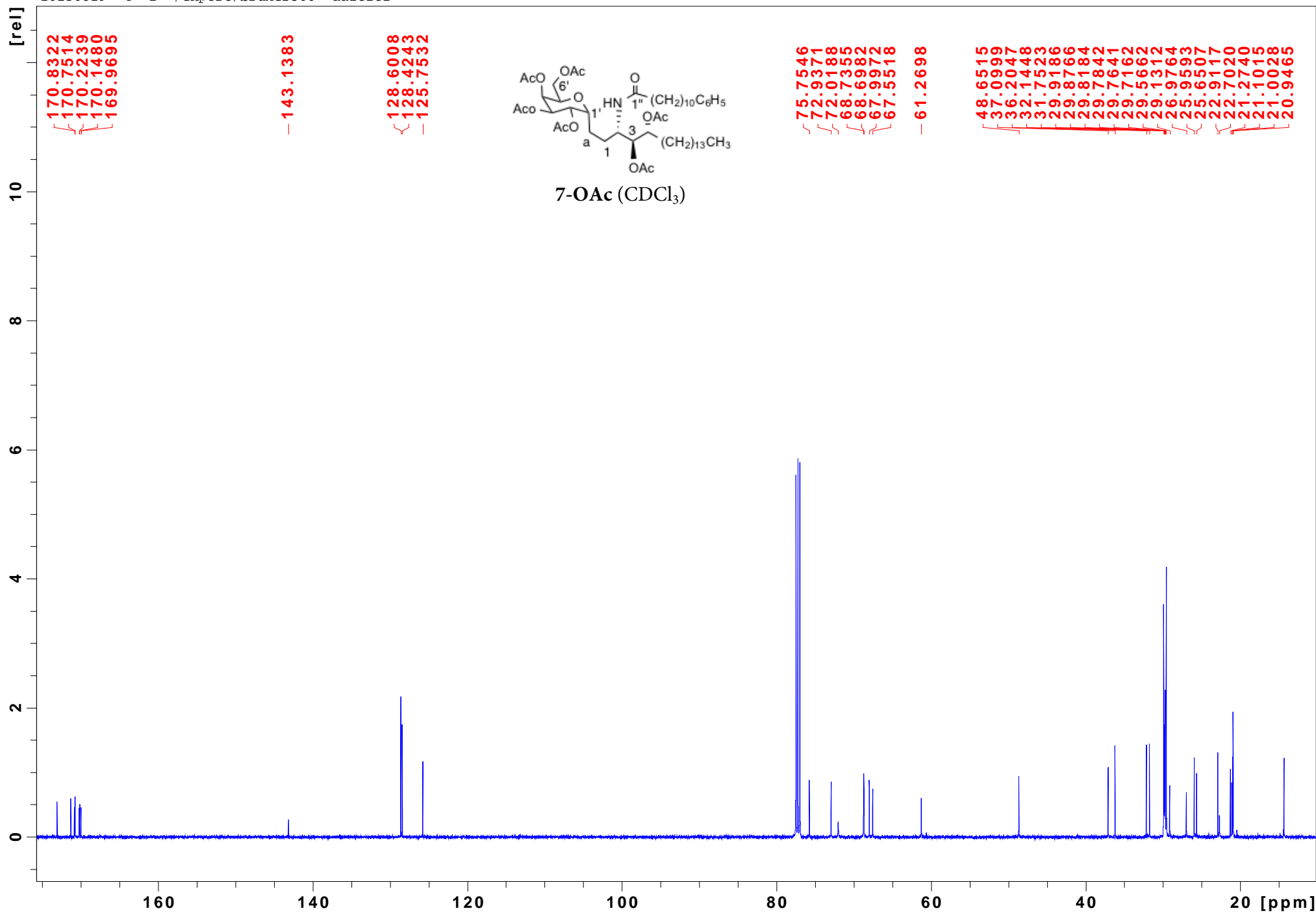
===== GRADIENT CHANNEL =====
GPNAME1        SINE.100
GPNAME2        SINE.100
GPZ1           80.00 %
GPZ2           20.10 %
P16           1000.00 usec
ND0            2
TD            128
SFO1          125.7678 MHz
FIDRES        196.293976 Hz
SW            199.778 ppm
FrMODE        Echo-Antiecho
SI            1024
SF            500.1299792 MHz
WDW           QSINE
SSB            2
LB            0.00 Hz
GB            0
PC            1.40
SI            1024
MC2           echo-antiecho
SF            125.7576890 MHz
WDW           QSINE
SSB            2
LB            0.00 Hz
GB            0
  
```

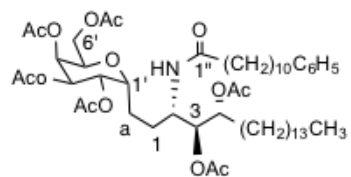





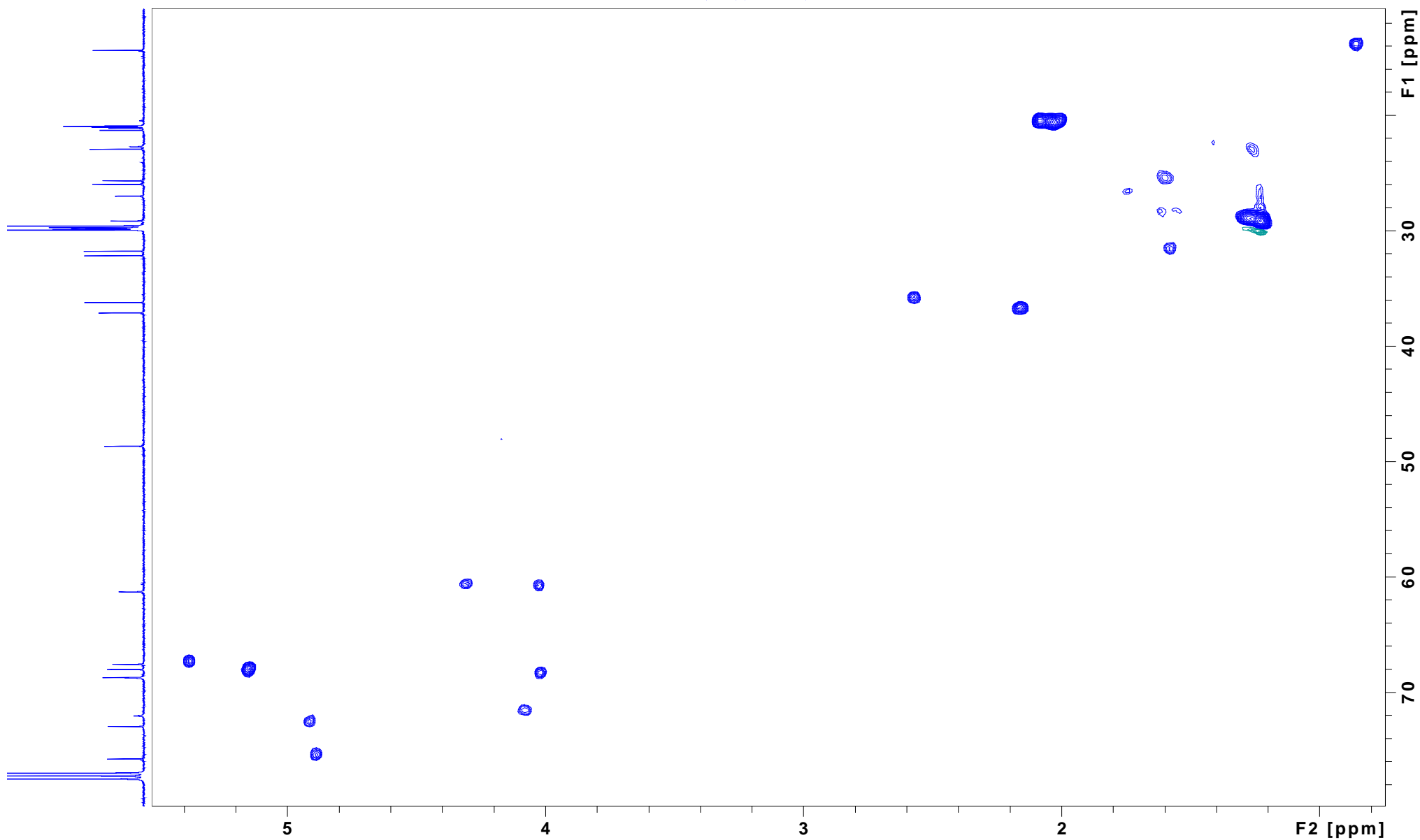
7-OAc (CDCl₃)

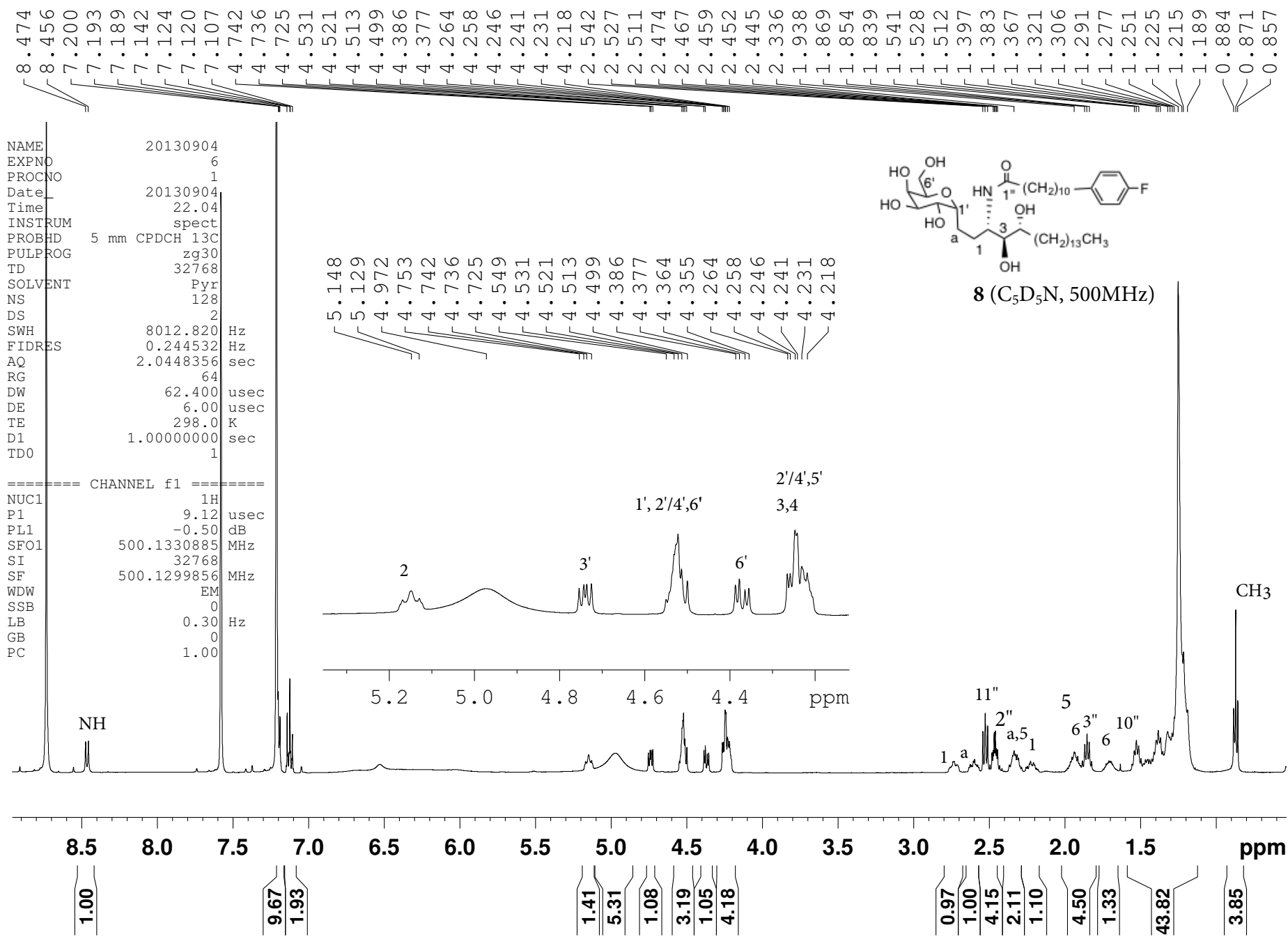






7-OAc (CDCl₃)





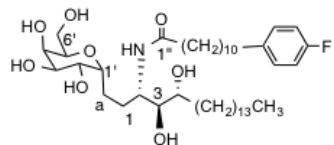
NAME 20130904
 EXPNO 6
 PROCNO 1
 Date_ 20130904
 Time 22.04
 INSTRUM spect
 PROBHD 5 mm CPDCH 13C
 PULPROG zg30
 TD 32768
 SOLVENT Pyr
 NS 128
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0448356 sec
 RG 64
 DW 62.400 usec
 DE 6.00 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 9.12 usec
 PL1 -0.50 dB
 SFO1 500.1330885 MHz
 SI 32768
 SF 500.1299856 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

5.148
 5.129
 4.972
 4.753
 4.742
 4.736
 4.725
 4.549
 4.531
 4.521
 4.513
 4.499
 4.386
 4.377
 4.364
 4.355
 4.264
 4.258
 4.246
 4.241
 4.231
 4.218
 2'/4',5'
 3,4
 1', 2'/4',6'
 6'
 2
 3'

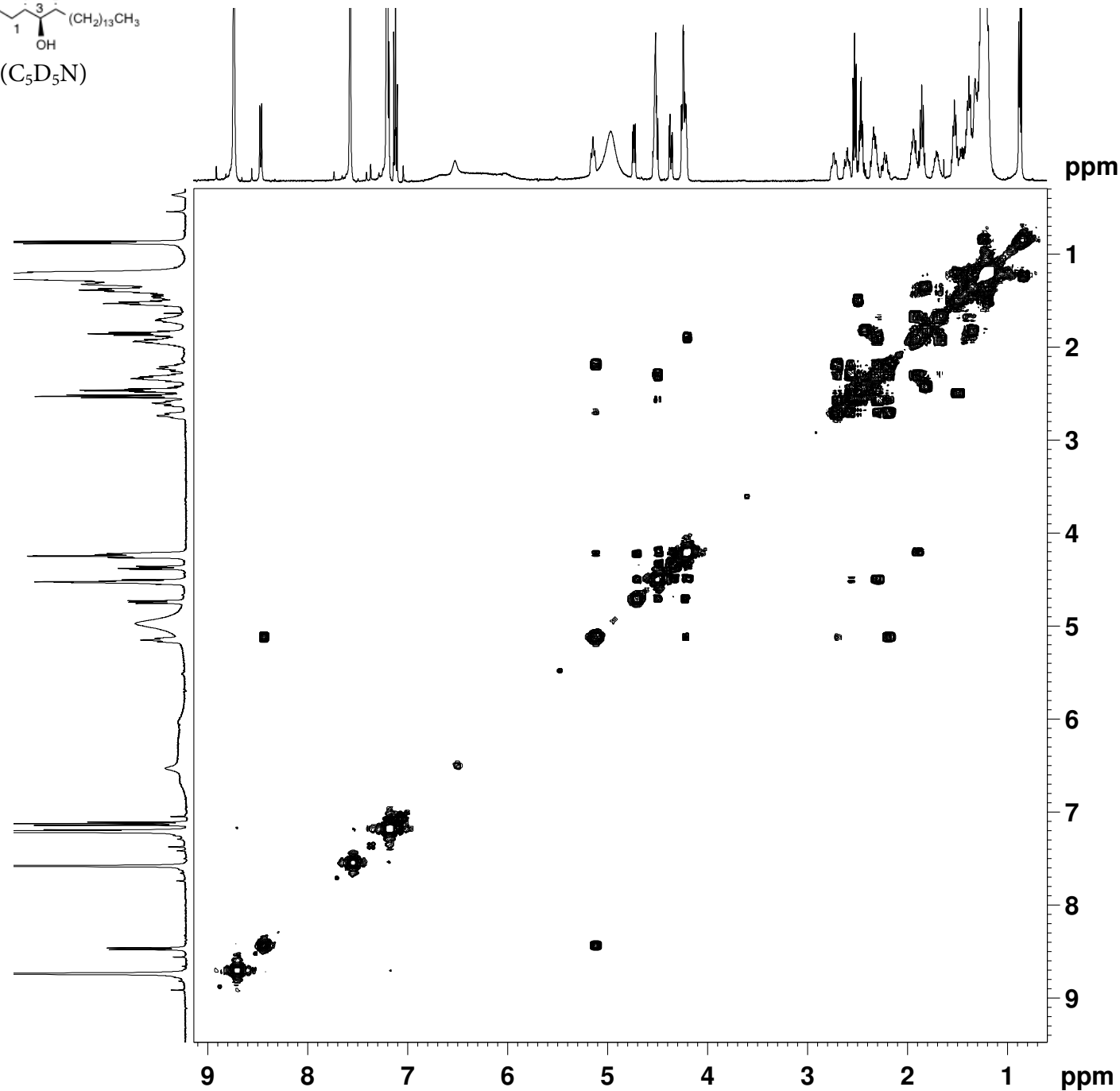
11''
 2''
 a,5
 1
 5
 6
 3''
 10''
 1
 a

CH₃

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



8 (C₅D₅N)

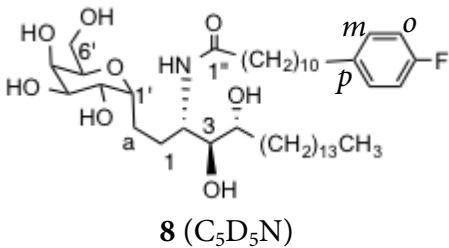
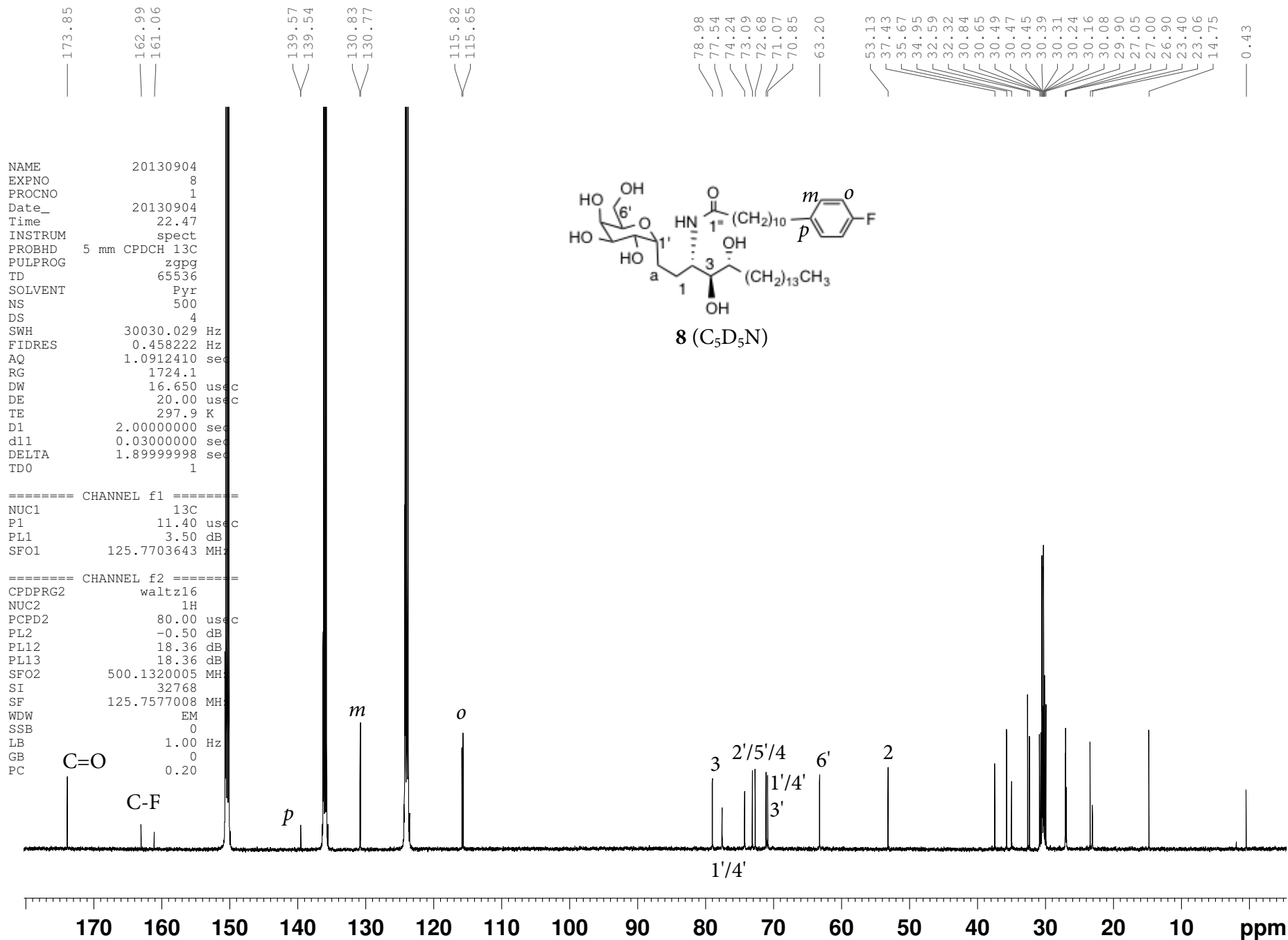


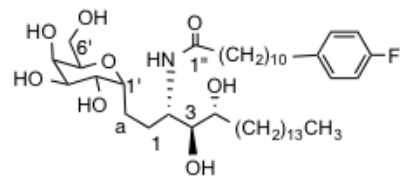
```

NAME          20130904
EXPNO         7
PROCNO        1
Date_         20130904
Time          22.04
INSTRUM       spect
PROBHD        5 mm CPDCH 13C
PULPROG       cosygpgqf
TD            2048
SOLVENT       Pyr
NS            5
DS            8
SWH           6666.667 Hz
FIDRES        3.255208 Hz
AQ            0.1537250 sec
RG            32
DW            75.000 usec
DE            6.00 usec
TE            298.0 K
d0            0.00000300 sec
D1            1.48689198 sec
d13           0.00000400 sec
D16           0.00020000 sec
IN0           0.00015000 sec

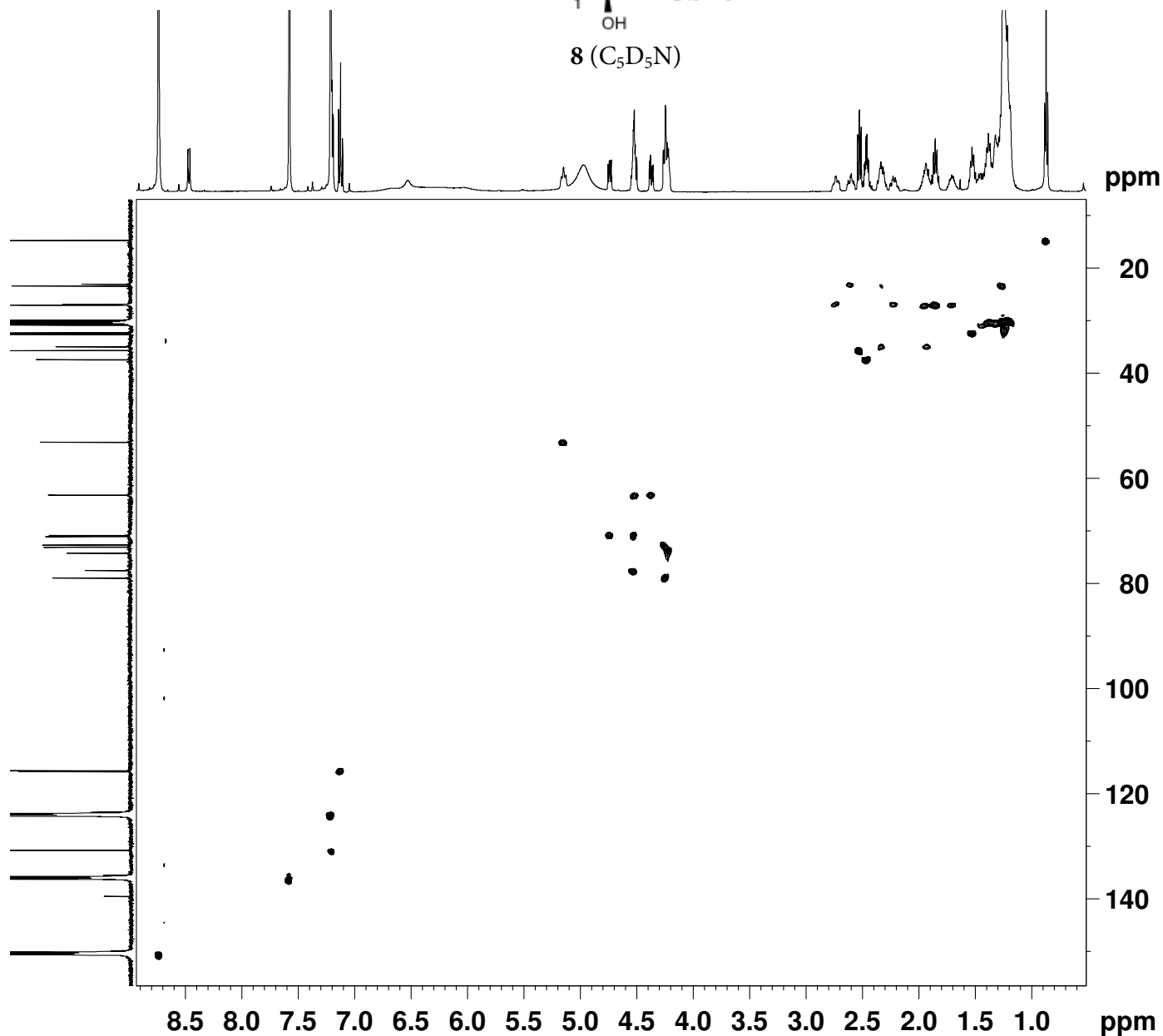
===== CHANNEL f1 =====
NUC1          1H
P0            9.12 usec
P1            9.12 usec
PL1           -0.50 dB
SFO1          500.1330069 MHz

===== GRADIENT CHANNEL =====
GPNAM1       SINE.100
GPZ1         10.00 %
P16          1000.00 usec
ND0          1
TD           116
SFO1         500.133 MHz
FIDRES       57.471264 Hz
SW           13.330 ppm
FnMODE       QF
SI           1024
SF           500.1299998 MHz
WDW          SINE
SSB          0
LB           0.00 Hz
GB           0
PC           1.40
SI           1024
MC2          QF
SF           500.1300010 MHz
WDW          SINE
SSB          0
LB           0.00 Hz
GB           0
  
```





8 (C₅D₅N)



```

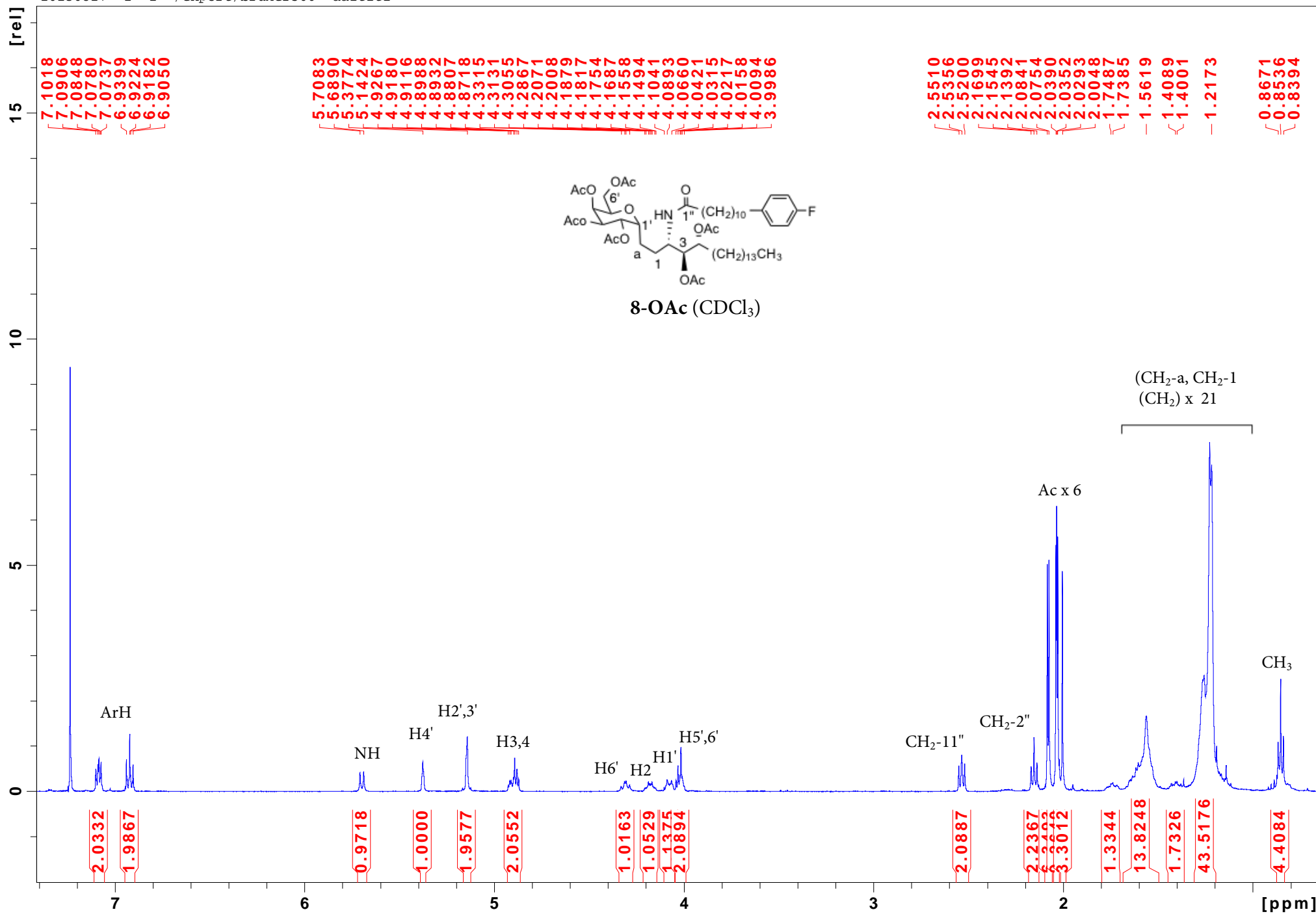
NAME          20130904
EXPNO         9
PROCNO        1
Date_         20130904
Time_         22.48
INSTRUM       spect
PROBHD        5 mm CPDCH 13C
PULPROG       hsqcetgp
TD            1024
SOLVENT       Pyr
NS            12
DS            16
SWH           6666.667 Hz
FIDRES        6.510417 Hz
AQ            0.0769250 sec
RG            29193
DW            75.000 usec
DE            6.00 usec
TE            298.0 K
CNST2         145.0000000
d0            0.00000300 sec
D1            1.20000005 sec
d4            0.00172414 sec
d11           0.03000000 sec
d13           0.00000400 sec
D16           0.00020000 sec
DELTA         0.00122424 sec
DELTA1        0.00071614 sec
IN0           0.00001990 sec
ST1CNT        0
ZGPTNS

===== CHANNEL f1 =====
NUC1           1H
P1             9.12 usec
p2            18.24 usec
P28           2000.00 usec
PL1           -0.50 dB
SFO1          500.1330069 MHz

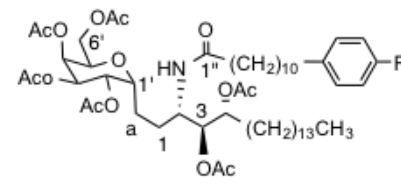
===== CHANNEL f2 =====
CPDPRG2       garp
NUC2           13C
P3            11.40 usec
p4            22.80 usec
PCPD2         70.00 usec
PL2           3.50 dB
PL12          19.26 dB
SFO2          125.7678496 MHz

===== GRADIENT CHANNEL =====
GPNAM1        SINE.100
GPNAM2        SINE.100
GPZ1          80.00 %
GPZ2          20.10 %
P16           1000.00 usec
ND0           2
TD            116
SFO1          125.7678 MHz
FIDRES        216.600235 Hz
SW            199.778 ppm
FnMODE        Echo-Antiecho
SI            1024
SF            500.1299824 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0
FC            1.40
SI            1024
MC2           echo-antiecho
SF            125.7576947 MHz
WDW           QSINE
SSB           2
LB            0.00 Hz
GB            0

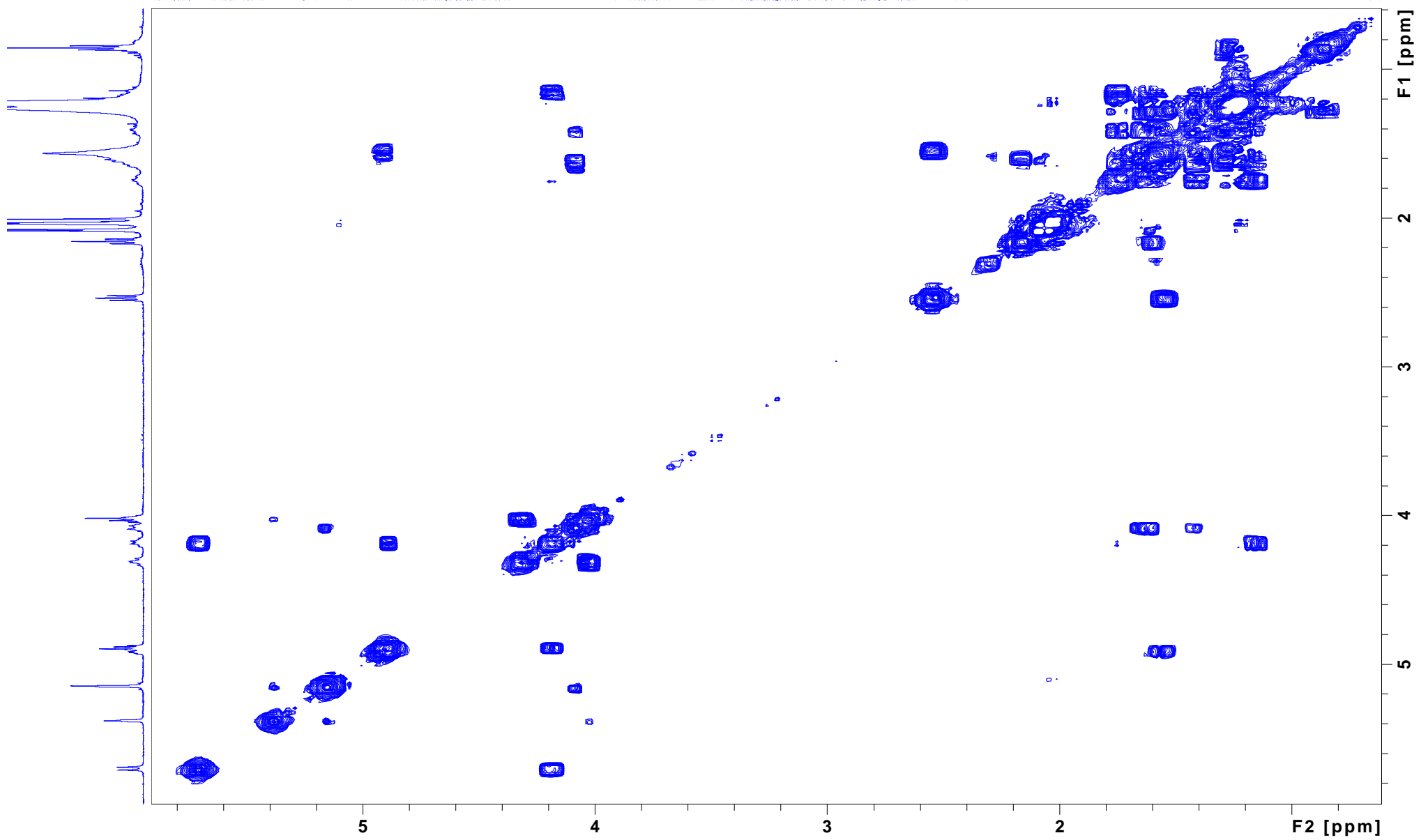
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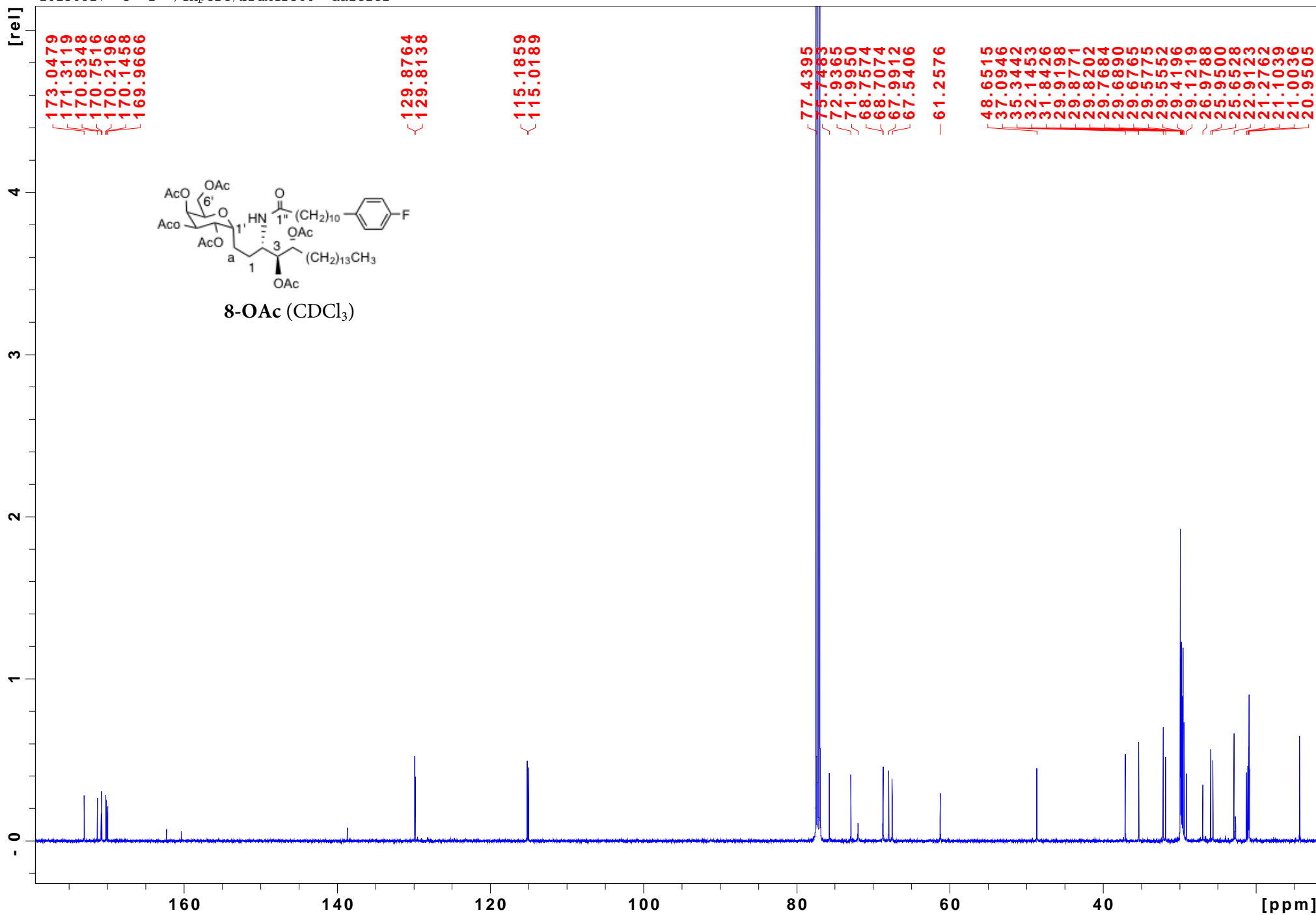



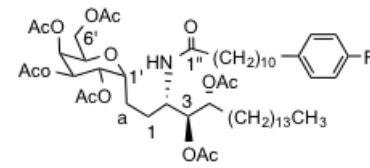
20130827 4 1 /export/bruker500 aaltiti



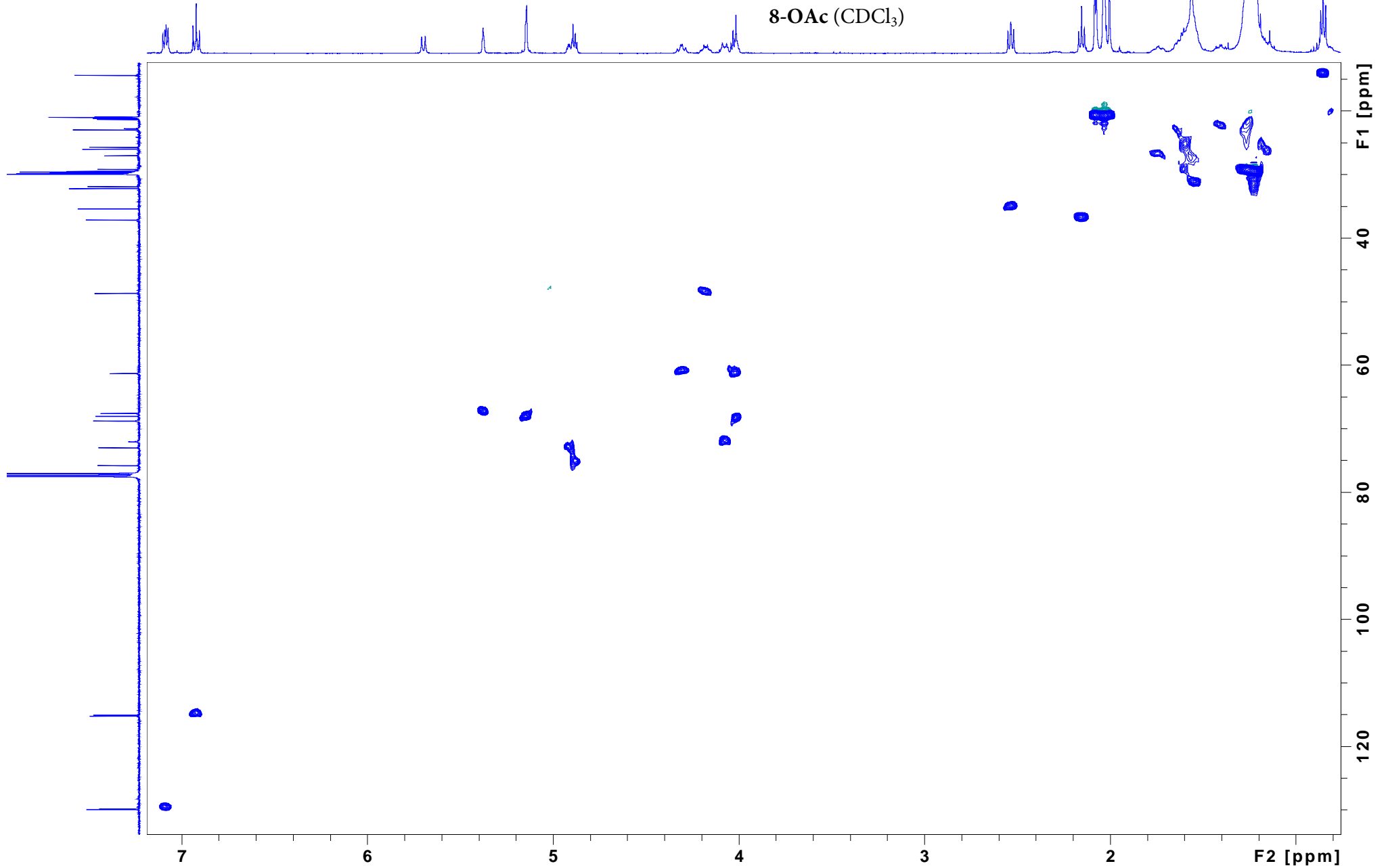
8-OAc (CDCl₃)

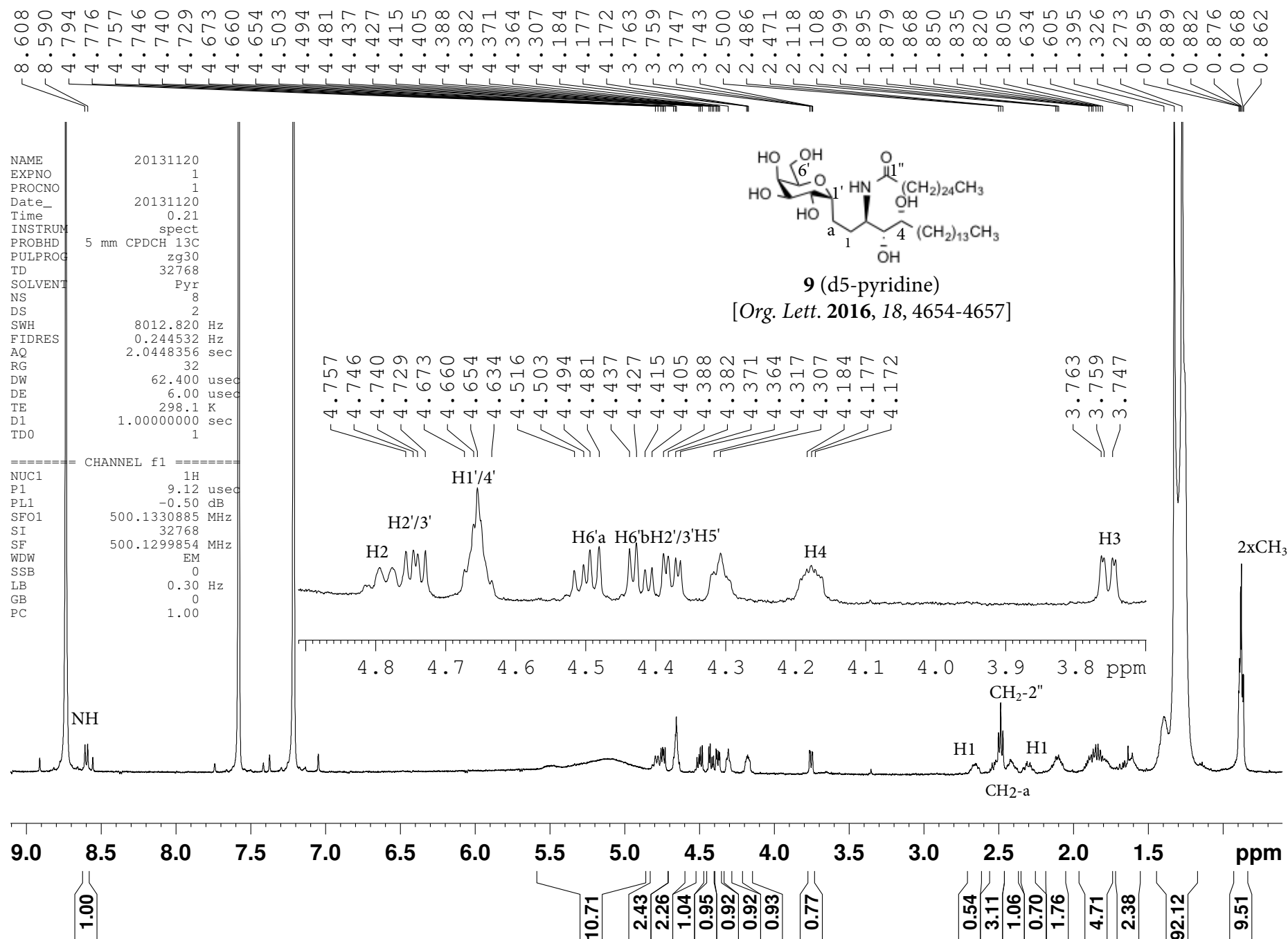




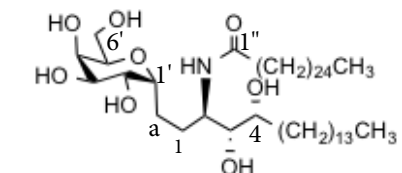


8-OAc (CDCl₃)





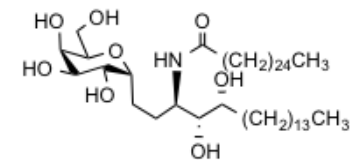
NAME 20131120
 EXPNO 1
 PROCNO 1
 Date_ 20131120
 Time 0.21
 INSTRUM spect
 PROBHD 5 mm CPDCH 13C
 PULPROG zg30
 TD 32768
 SOLVENT Pyr
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 2.0448356 sec
 RG 32
 DW 62.400 usec
 DE 6.00 usec
 TE 298.1 K
 D1 1.00000000 sec
 TD0 1



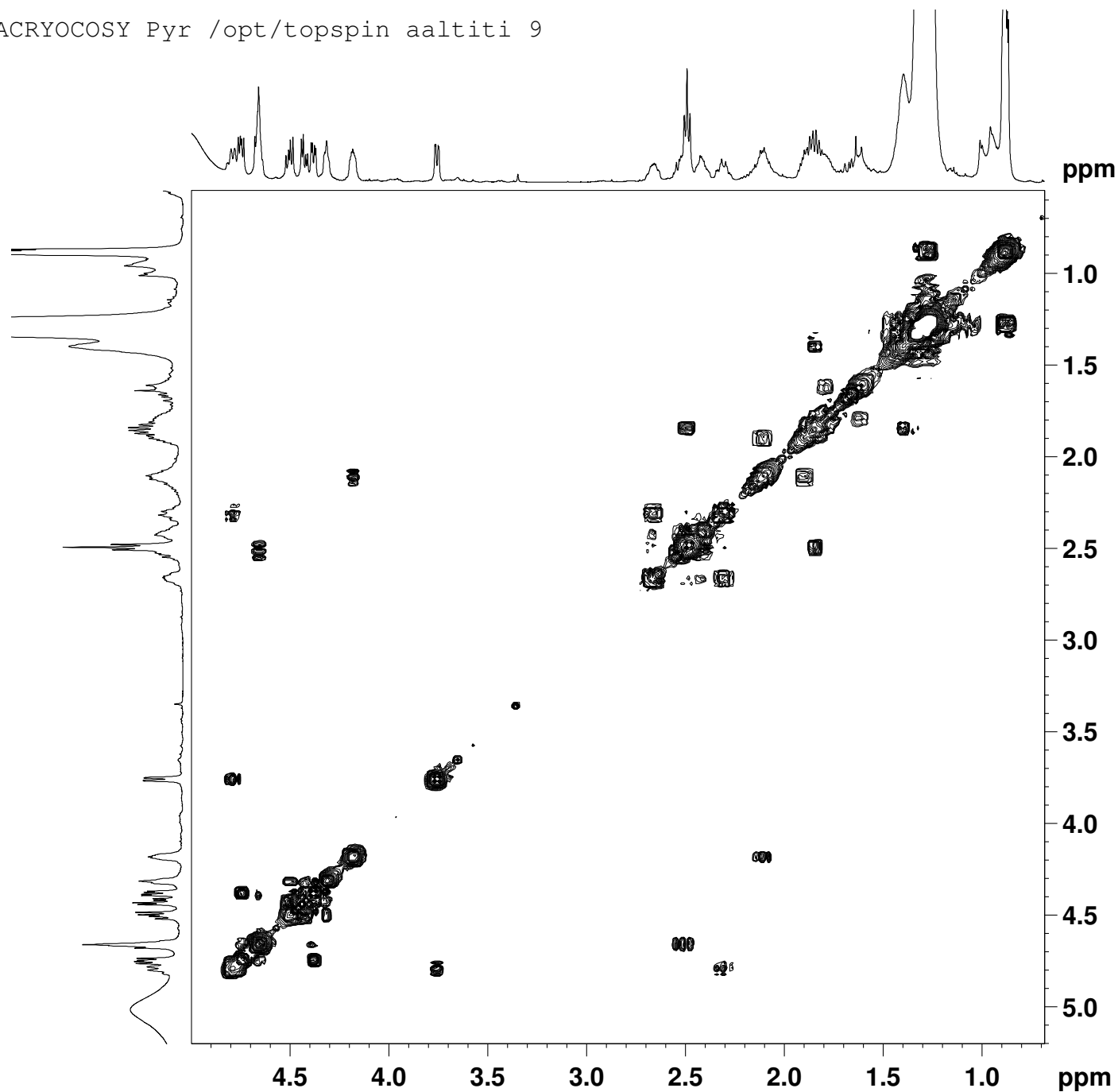
9 (d5-pyridine)
 [Org. Lett. 2016, 18, 4654-4657]

==== CHANNEL f1 =====
 NUC1 1H
 P1 9.12 usec
 PL1 -0.50 dB
 SFO1 500.1330885 MHz
 SI 32768
 SF 500.1299854 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

AAACRYOCOSY Pyr /opt/topspin aaltiti 9



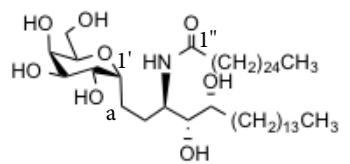
9 (d5-pyridine)



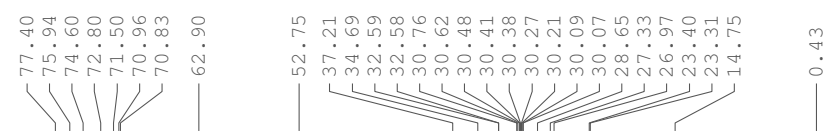
```
NAME          20131120
EXPNO         2
PROCNO        1
Date_         20131120
Time          0.22
INSTRUM       spect
PROBHD        5 mm CPDCH 13C
PULPROG       cosygpcqf
TD            2048
SOLVENT       Pyr
NS            2
DS            8
SWH           6666.667 Hz
FIDRES        3.255208 Hz
AQ            0.1537250 sec
RG            18
DW            75.000 usec
DE            6.00 usec
TE            298.1 K
d0            0.00000300 sec
d1            1.48689198 sec
d13           0.00000400 sec
d16           0.00020000 sec
IN0           0.00015000 sec

===== CHANNEL f1 =====
NUC1           1H
P0             9.12 usec
P1             9.12 usec
PL1            -0.50 dB
SFO1           500.1330069 MHz

===== GRADIENT CHANNEL =====
GPNAM1        SINE.100
GPZ1           10.00 %
P16           1000.00 usec
ND0            1
TD            128
SFO1           500.133 MHz
FIDRES         52.083332 Hz
SW             13.330 ppm
FnmODE         QF
SI             1024
SF            500.1299812 MHz
WDW            SINE
SSB            0
LB             0.00 Hz
GB             0
PC             1.40
SI             1024
MC2            QF
SF            500.1299795 MHz
WDW            SINE
SSB            0
LB             0.00 Hz
GB             0
```



9 (d5-pyridine)



```

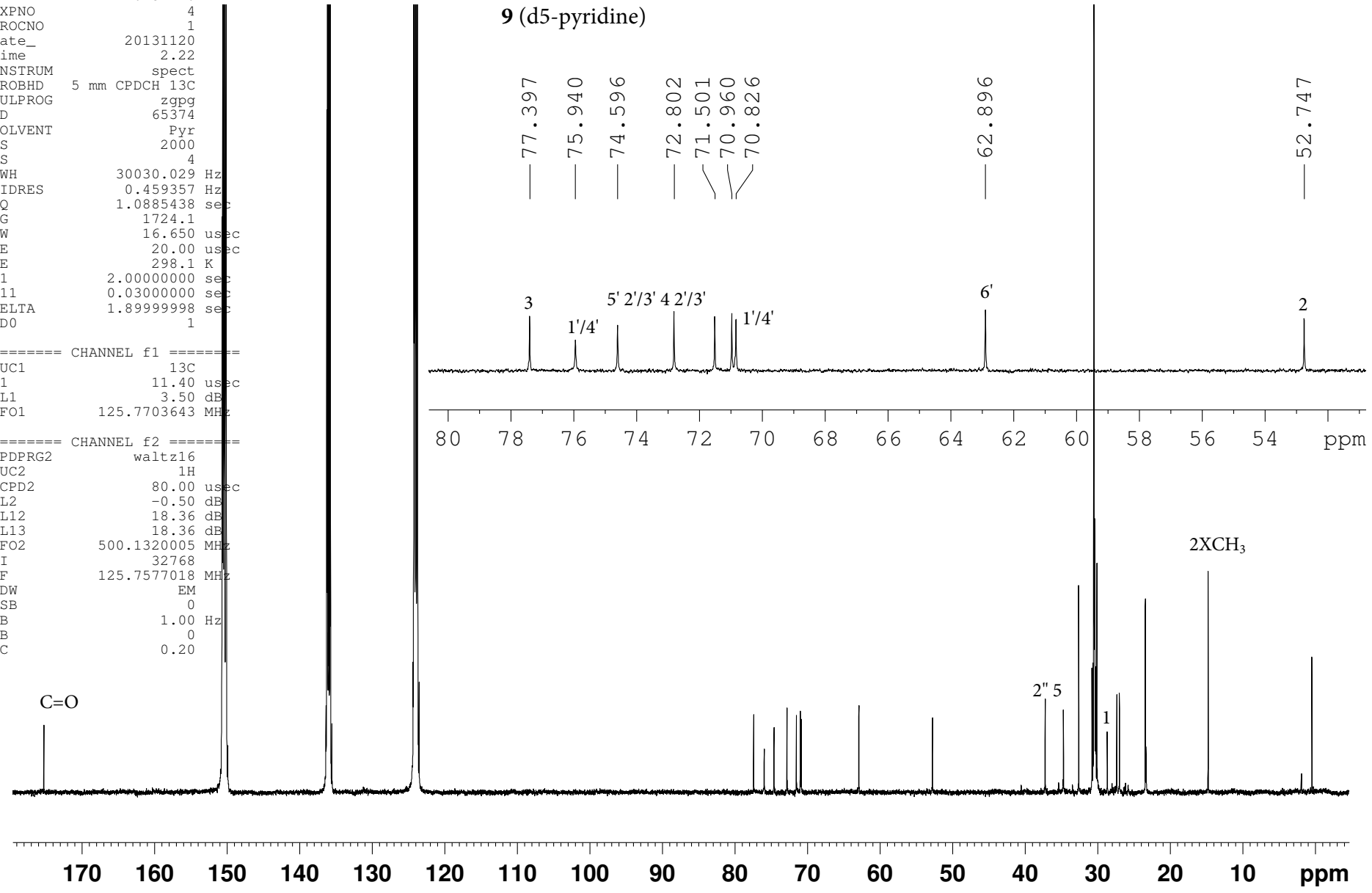
NAME      20131120
EXPNO     4
PROCNO    1
Date_     20131120
Time      2.22
INSTRUM   spect
PROBHD    5 mm CPDCH 13C
PULPROG   zgpg
TD         65374
SOLVENT   Pyr
NS         2000
DS         4
SWH        30030.029 Hz
FIDRES    0.459357 Hz
AQ         1.0885438 sec
RG         1724.1
DW         16.650 usec
DE         20.00 usec
TE         298.1 K
D1         2.0000000 sec
d11        0.0300000 sec
DELTA     1.89999998 sec
TDO        1
    
```

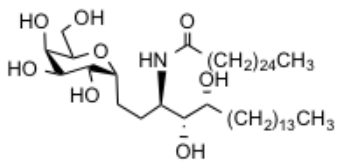
```

===== CHANNEL f1 =====
NUC1      13C
P1        11.40 usec
PL1       3.50 dB
SFO1     125.7703643 MHz
    
```

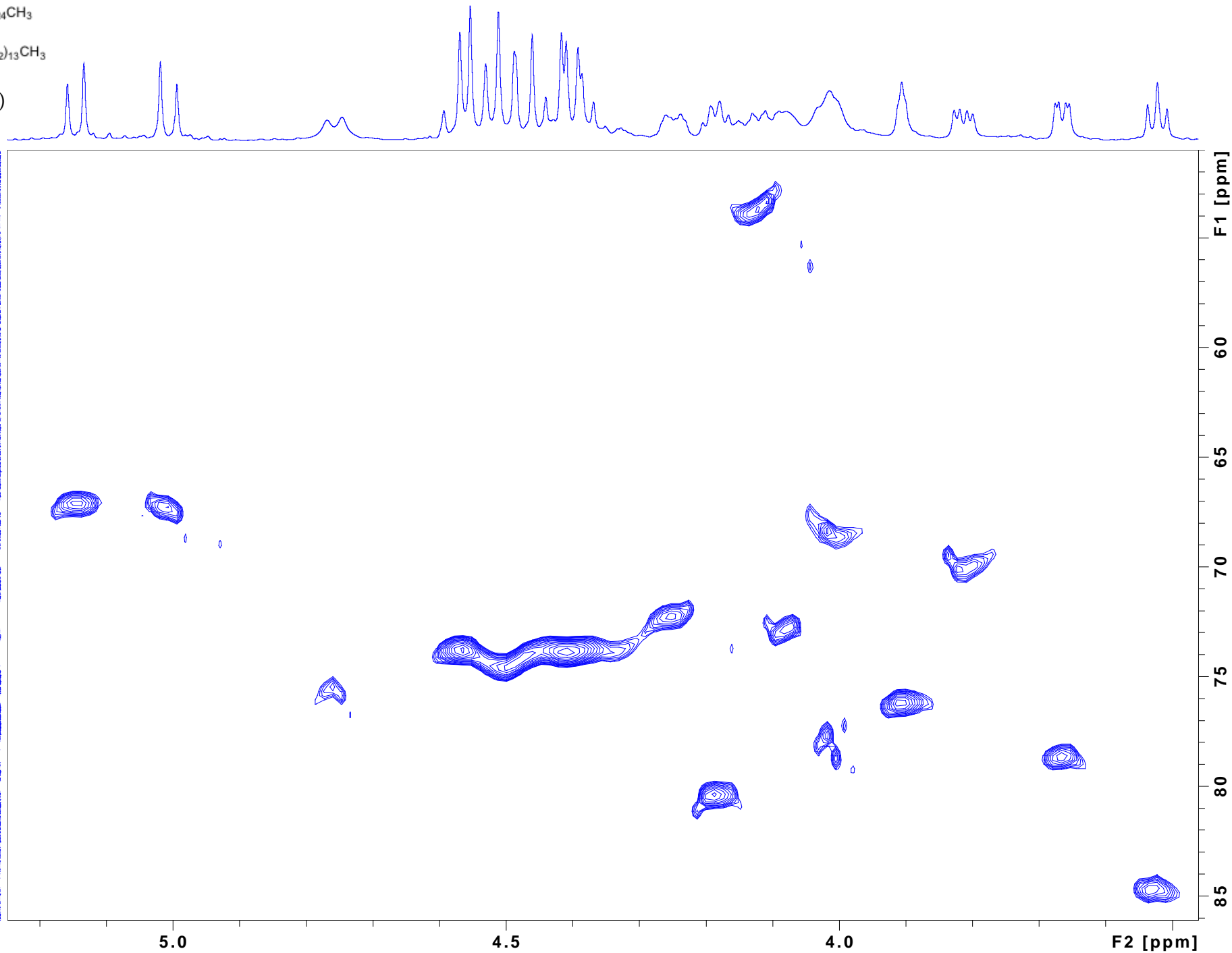
```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       -0.50 dB
PL12      18.36 dB
PL13      18.36 dB
SFO2     500.1320005 MHz
SI         32768
SF        125.7577018 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         0.20
    
```





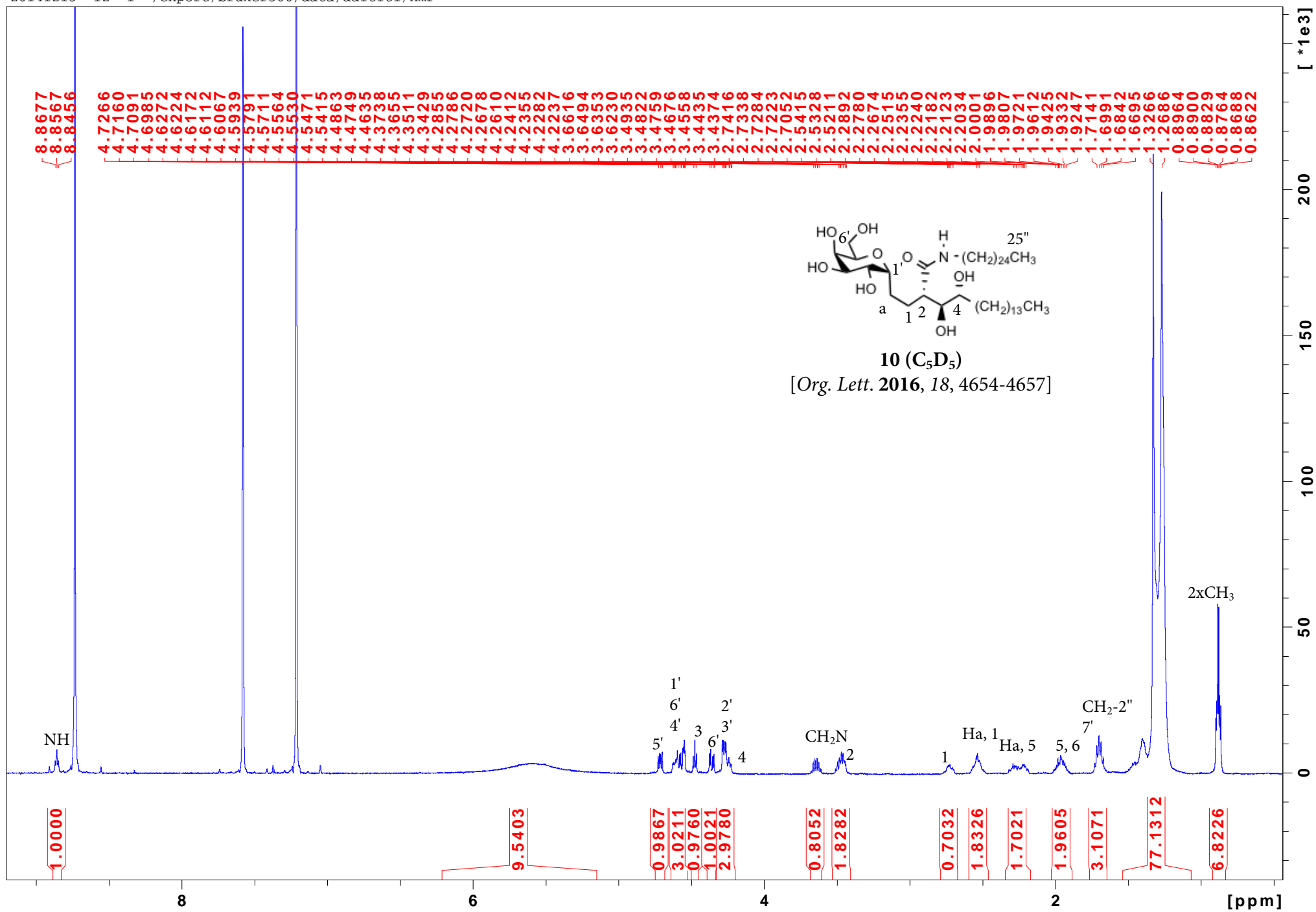
9 (d5-pyridine)



```

NAME          20130805
EXPNO         16
PROCNO        1
Date_         20130806
Time_         0.34
INSTRUM       xpc-c
PROBHD        5 mm CPDCH 13C
PULPROG       zgpg30
TD            1024
SOLVENT       c5d6
NS            50
DS            16
SWH           6666.667 Hz
FIDRES        6.510417 Hz
AQ            0.0769250 sec
RG            29193
EM            75.000 usec
DE            6.00 usec
TE            298.0 K
CST2          145.0000000
d0            0.0000000 sec
d1            1.2000000 sec
d4            0.00172414 sec
d11           0.03000000 sec
d13           0.00000400 sec
d16           0.00020000 sec
DELTA         0.00122424 sec
DELTA1        0.00071614 sec
IN0           0.00001990 sec
STICNT        0
EGOPTMS

```



20141215 13 1 /export/bruker500/data/aaltiti/nmr

