Supplementary Materials for

A convenient synthesis of (16*S*,20*S*)-3β-hydroxy-5*α*-pregnane-20,16-carbolactam and its *N*-alkyl derivatives

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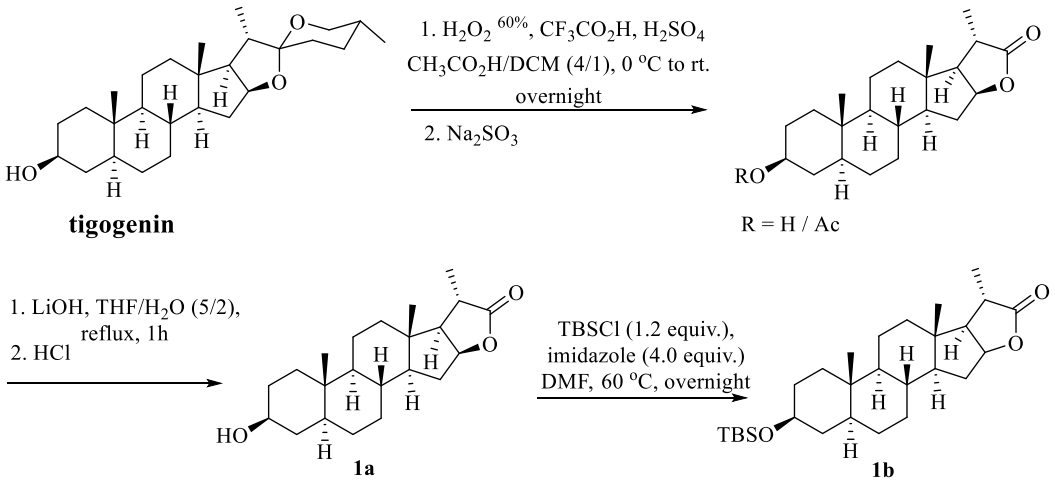
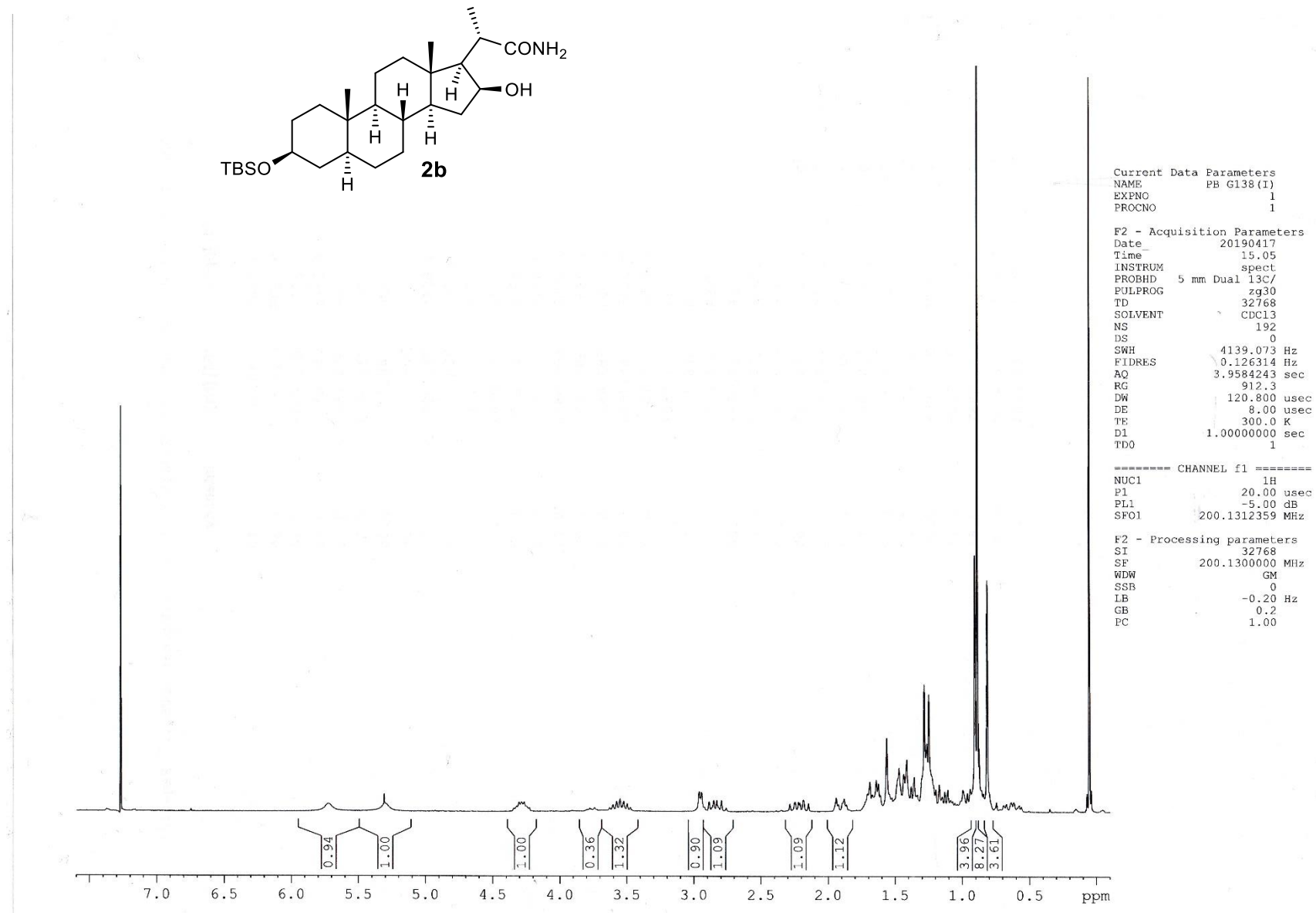
Experimental procedures for (16S,20S)-3β-*t*-butyldimethylsilyloxy-5*α*-pregnane-20,16-carbolactone (1b) synthesis from tigogenin



Trifluoroacetic acid (1 mL) and concentrated sulphuric acid (10 mL) were added to the mixture of tigogenin (10 g, 0.024 mol), molecular iodine (0.5 g, 0.002 mol), *n*-Bu_4NBr (0.100 g, 3 x 10^{-4} mol), acetic acid (120 ml) in DCM (30 ml). The multi-component solution was heated at 50° C for 1 hour, then, after cooling to 0°C, 60% aqueous solution of H_2O_2 (20 mL, 0.438 mol, 18.2 equiv.) was slowly added. After 1 h, the ice bath was removed and stirring was continued at room temperature overnight. The progress of the reaction was monitored by TLC. After completion of the reaction, a saturated aqueous Na_2SO_3 solution was carefully added to the mixture (ca. 300 mL). Stirring was continued until the purple colour of the mixture disappeared. After that, the resulting yellow solution was neutralized with 50% aqueous NaOH solution to pH 7 (ca. 120 mL). Product was extracted with DCM (5 x 30 mL). The combined organic layers were concentrated under reduced pressure. Then, LiOH (2.874, 0.120 mol) and a THF/H_2O (5/2) mixture (200 mL) were added to the residue. The resulting suspension was heated at reflux. The progress of saponification was monitored by TLC. After about 1 hour, the solution was acidified with 12 N HCl to pH 3 and stirring was continued for approx.. 10 min at 80°C. After cooling, THF was evaporated under reduced pressure. The residue was diluted with water (200 mL) and product was extracted with AcOEt (5 x 20 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered and the solvent was concentrated under reduced pressure. The crude product was purified by crystallization from AcOEt. (16S,20S)-3β-Hydroxy-5*α*-pregnane-20,16-carbolactone (**1a**) was obtained as a white crystalline material (7.591 g, 91%).

(16*S*,20*S*)-3β-*t*-Butyldimetylsillioxy-5*α*-pregnane-20,16-carbolactone (**1b**) was obtained in 95% (6.321 g) from (16*S*,20*S*)-3β-hydroxy-23,24-bis-norcholano-22,16-lactone (**1a**, 5 g, 0.014 mol, 1.0 equiv.) according to a standard procedure using imidazole (3.93 g, 0.058 mol, 4 equiv.), TBSCl (2.61 g, 0.017 mol, 1.2 equiv.) in anhydrous DMF (200 mL).

Spectra (^{1}H NMR, ^{13}CNMR) of compounds **2b** - **5b**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H
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H
H
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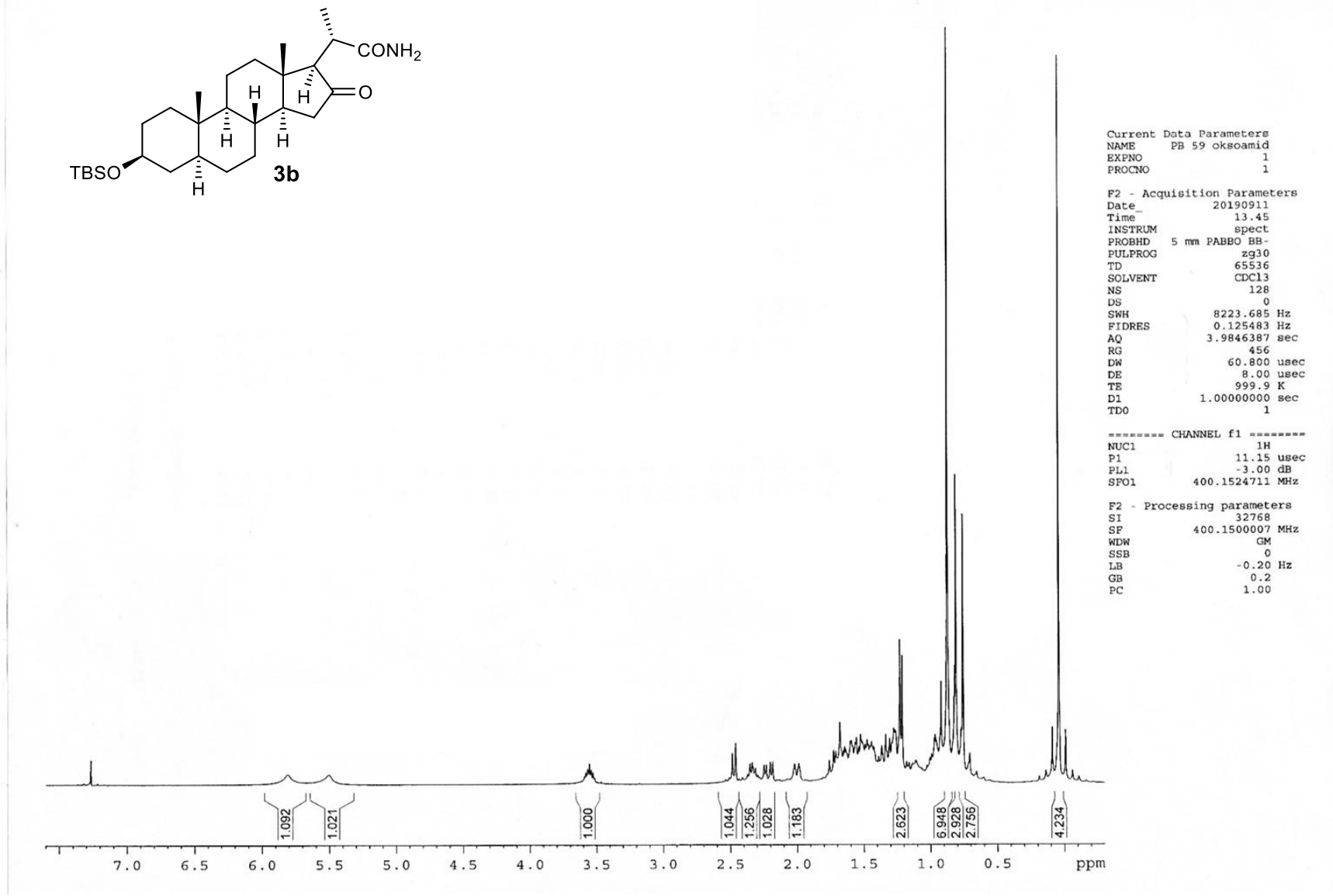
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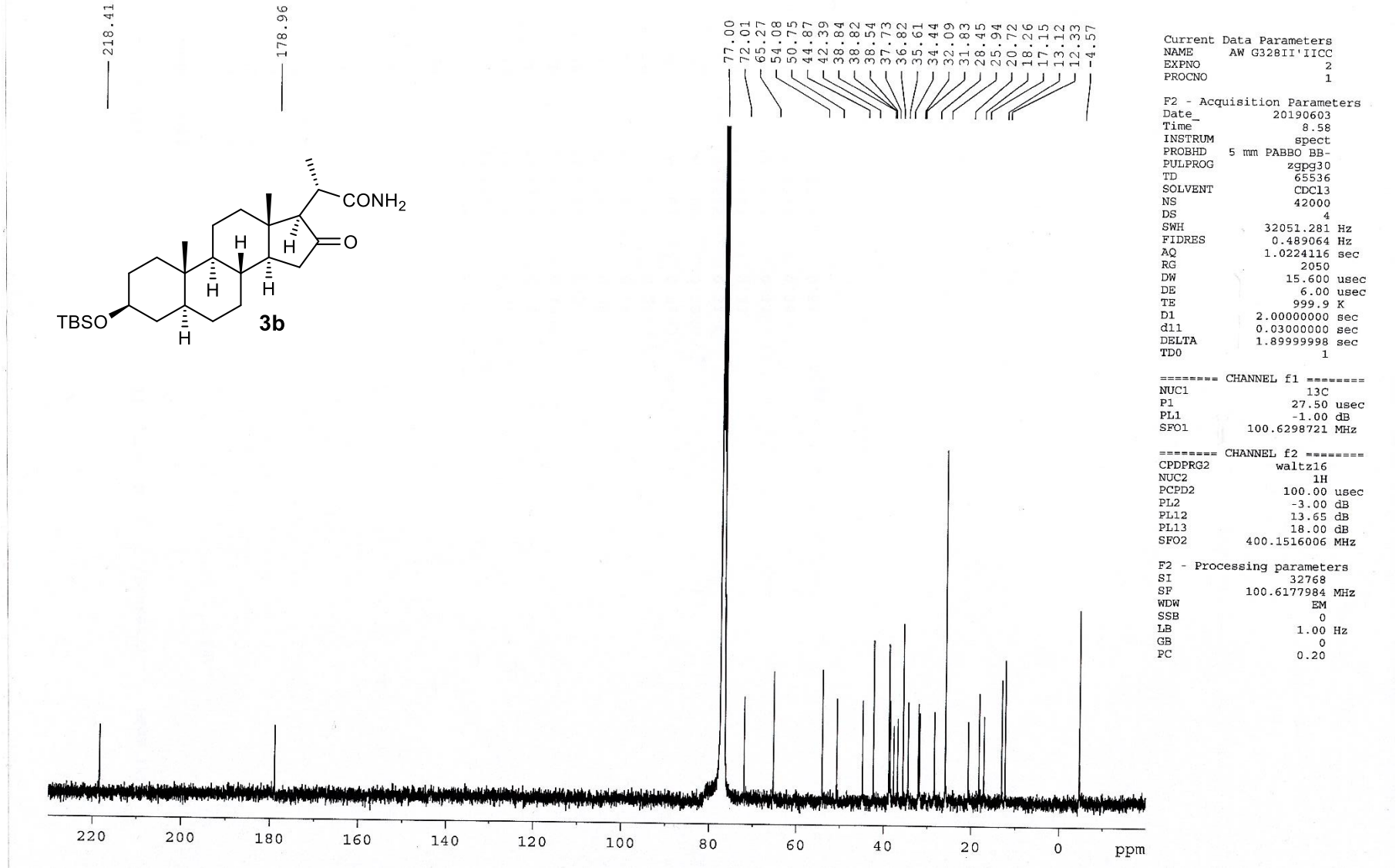
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H
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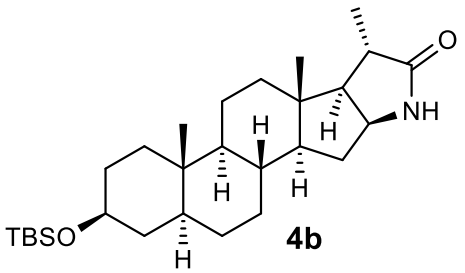

4b


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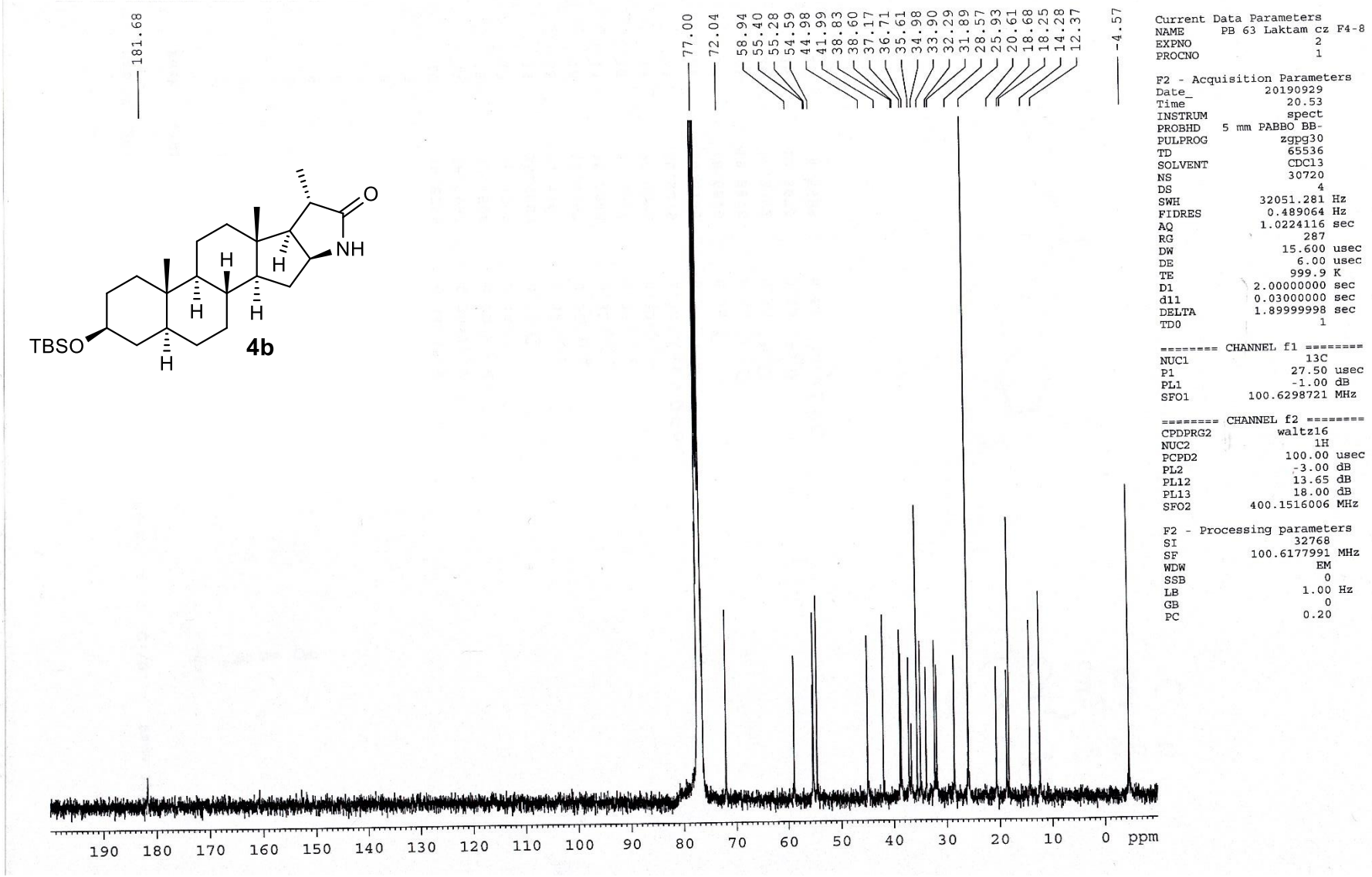

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TD0	1

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NUC1	13C
P1	27.50 usec
PL1	-1.00 dB
SFO1	100.6298721 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
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PL12	13.65 dB
PL13	18.00 dB
SFO2	400.1516006 MHz

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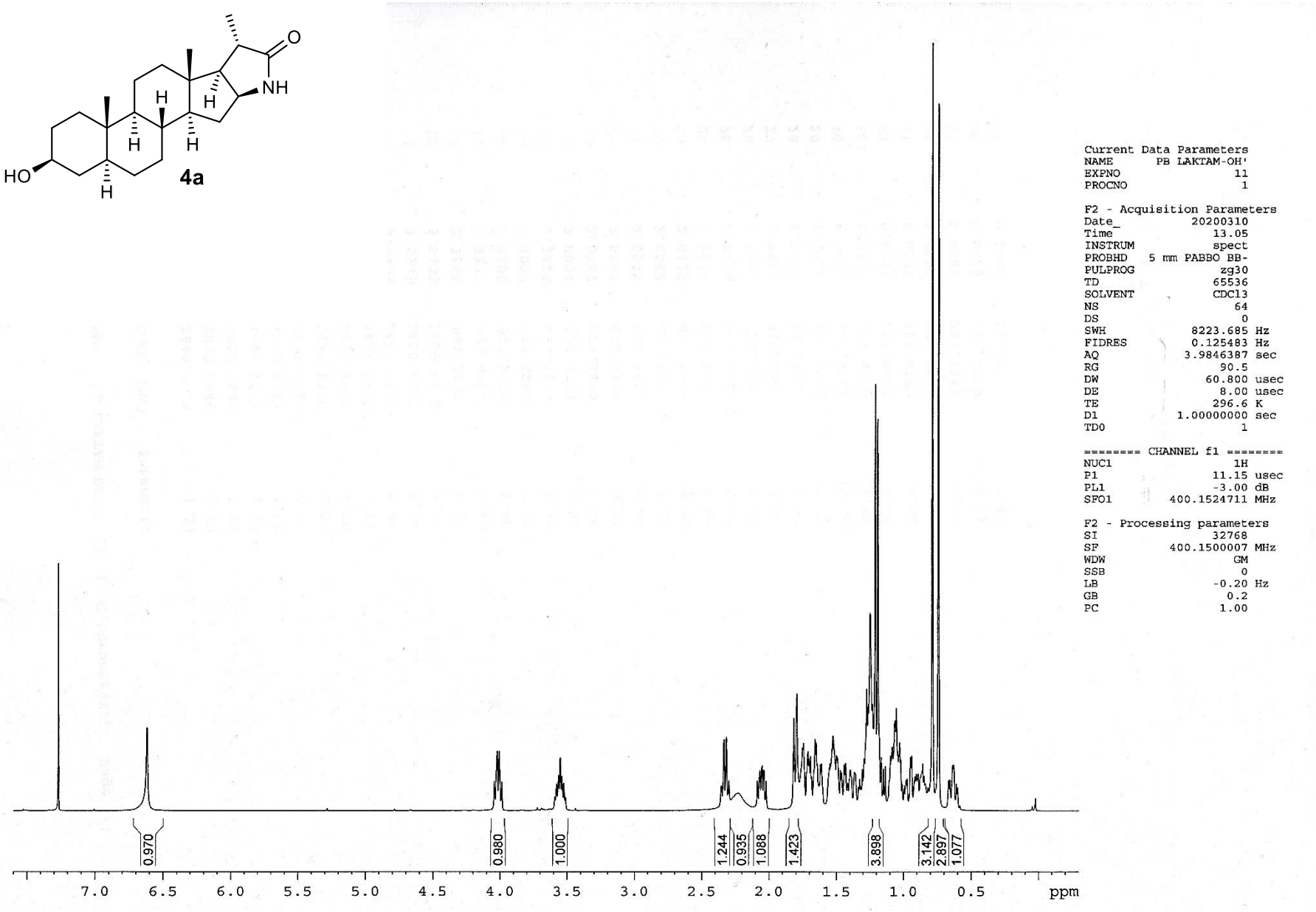


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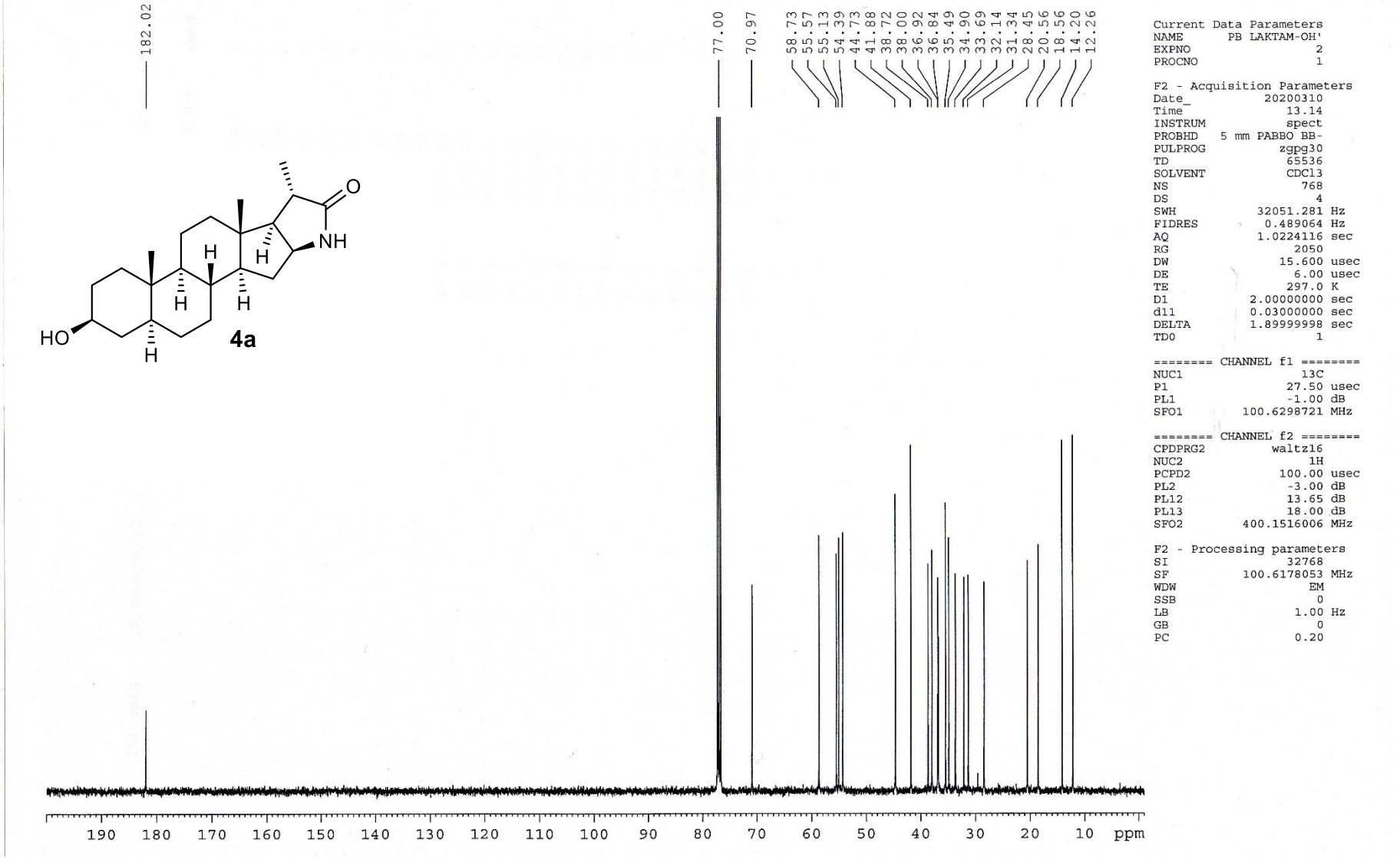


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TE               297.0 K
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TD0                  1

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PL1               -1.00 dB
SFO1        100.6298721 MHz

======== CHANNEL f2 ========
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NUC2                1H
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PL12              13.65 dB
PL13              18.00 dB
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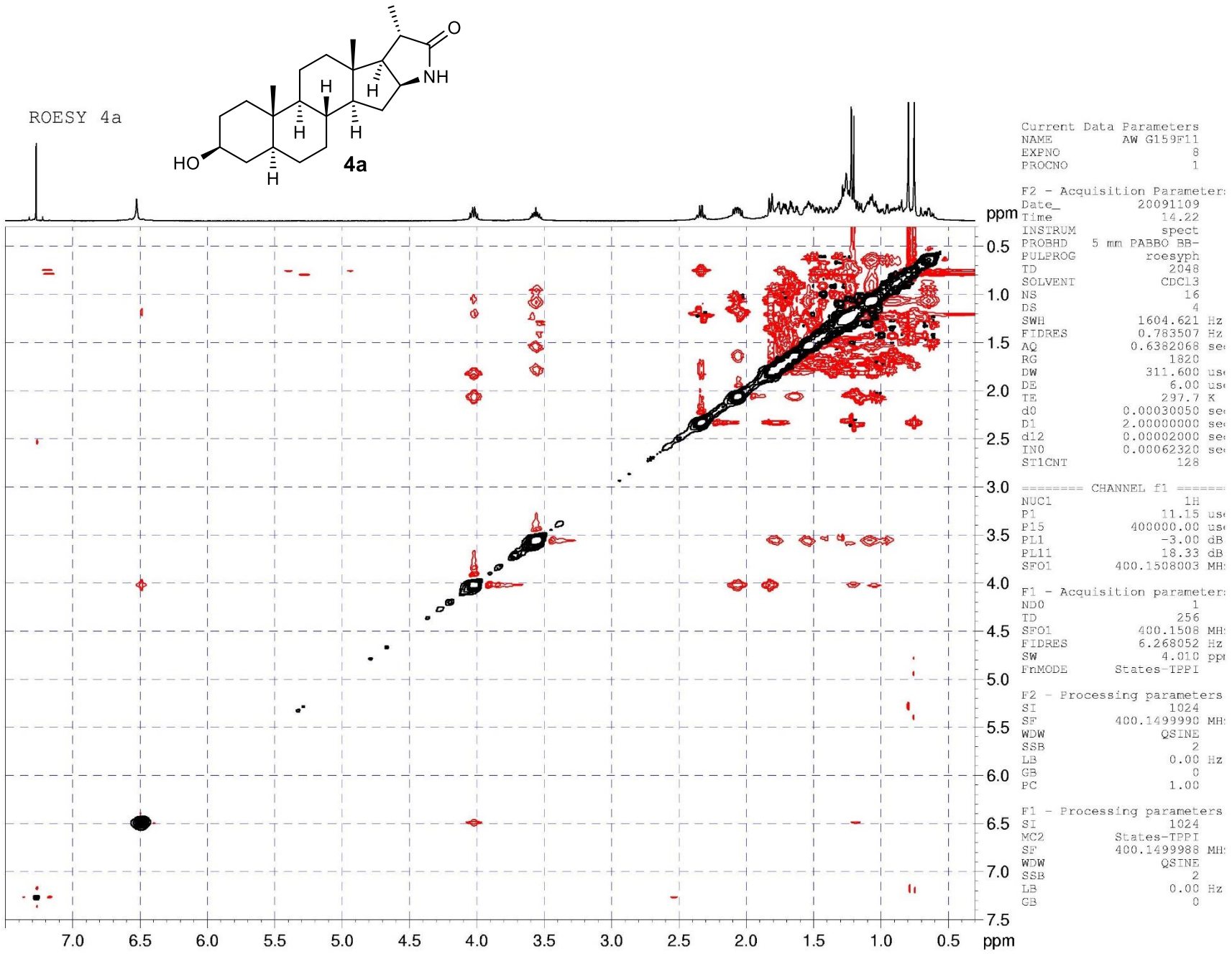
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D1 2.00000000 se
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PL11 18.33 dB
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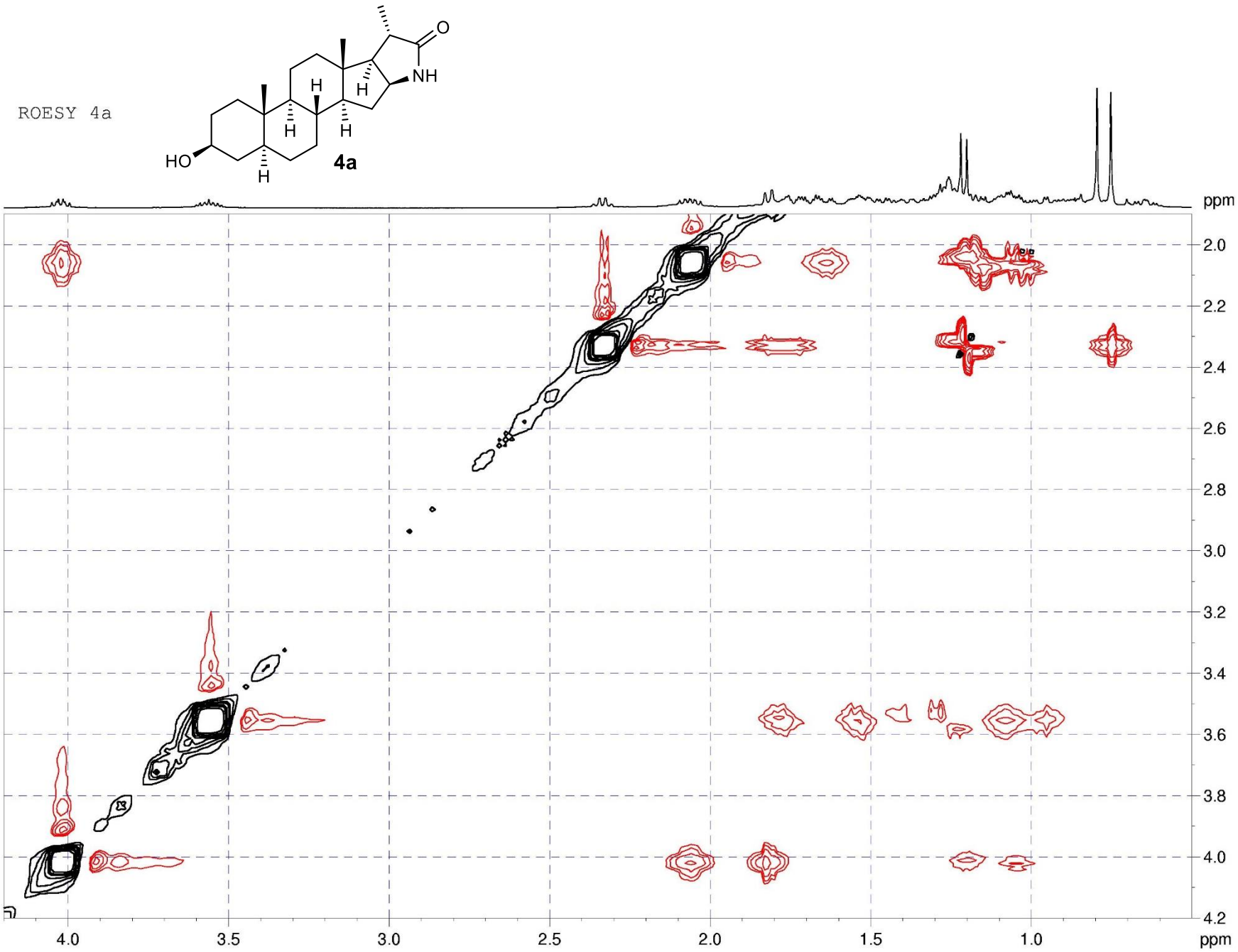
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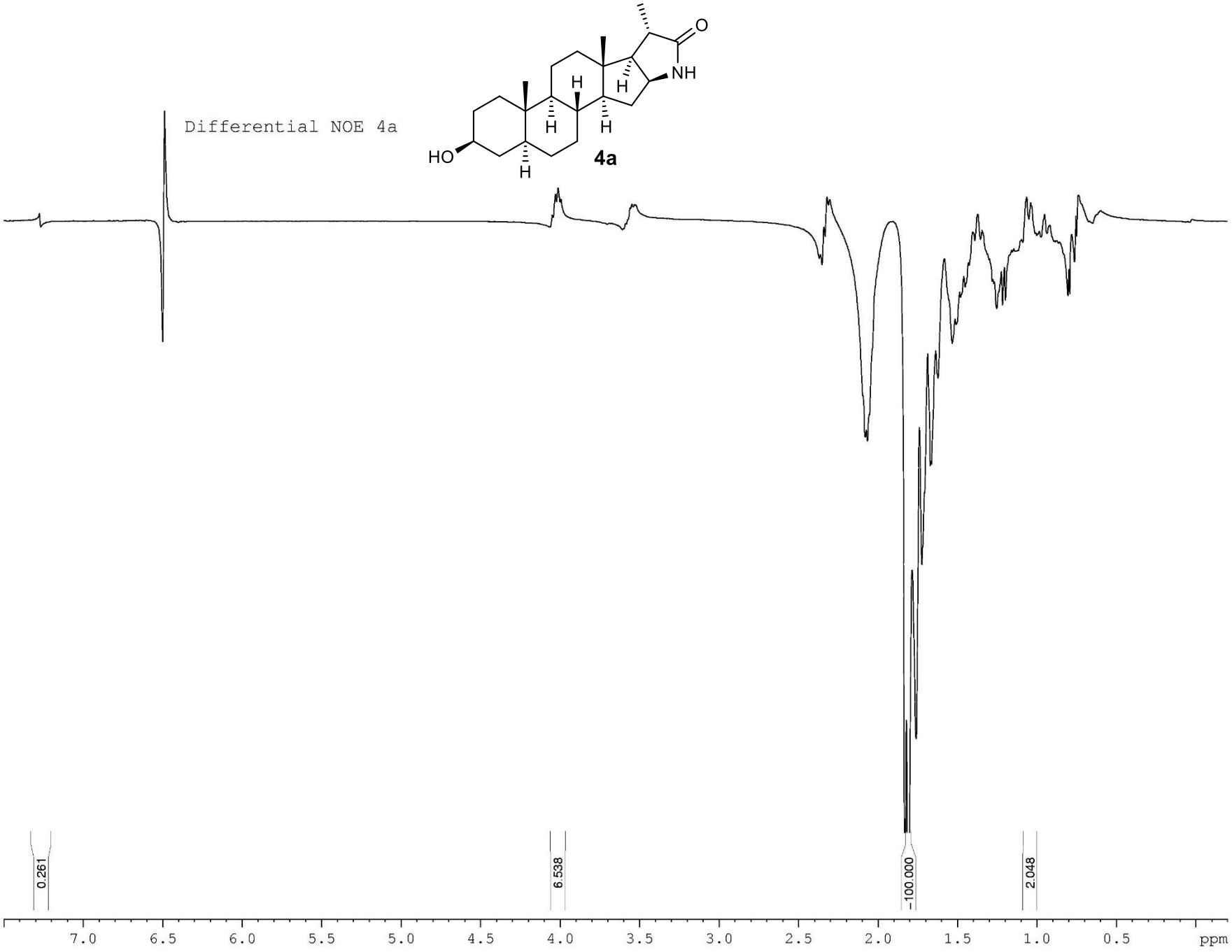
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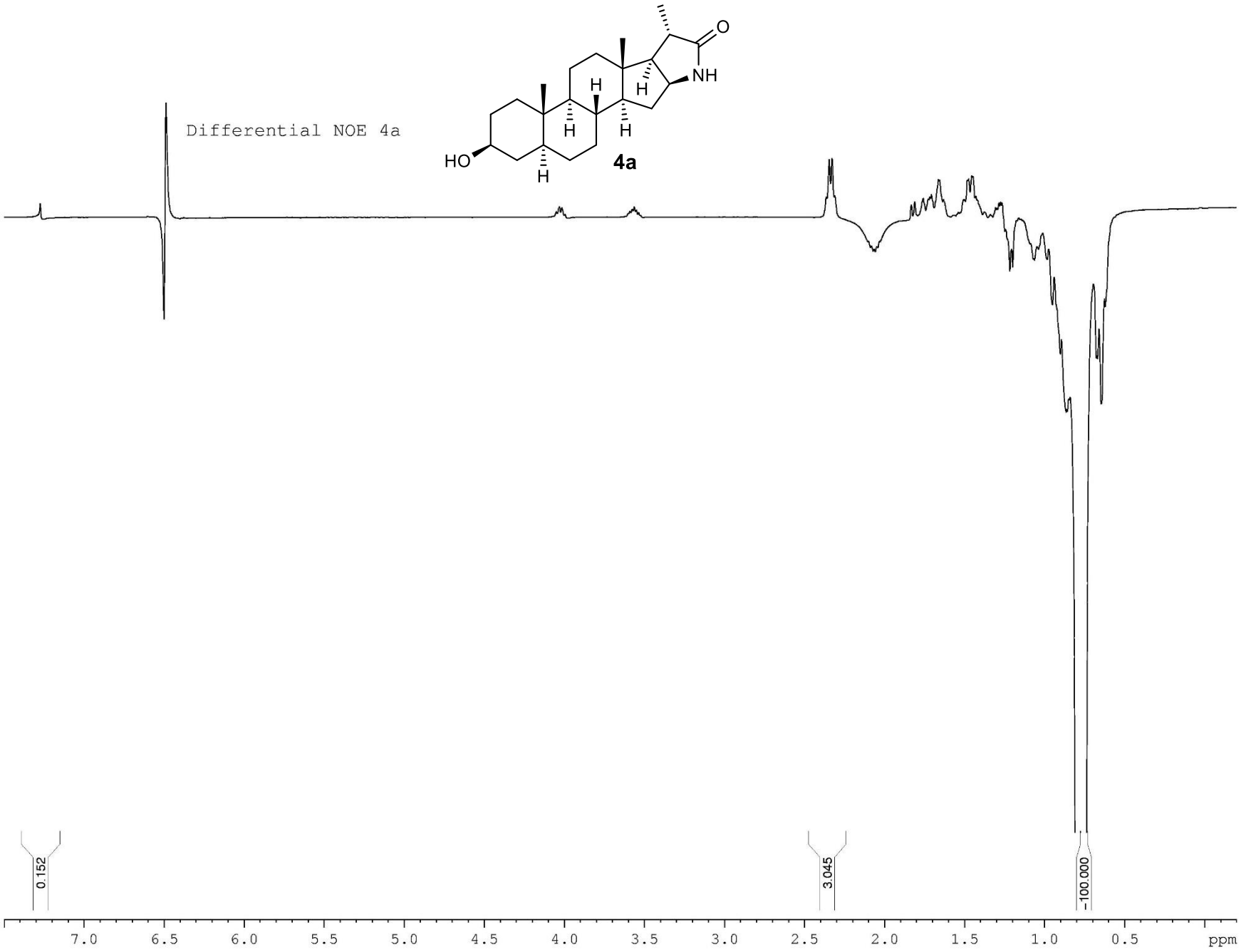
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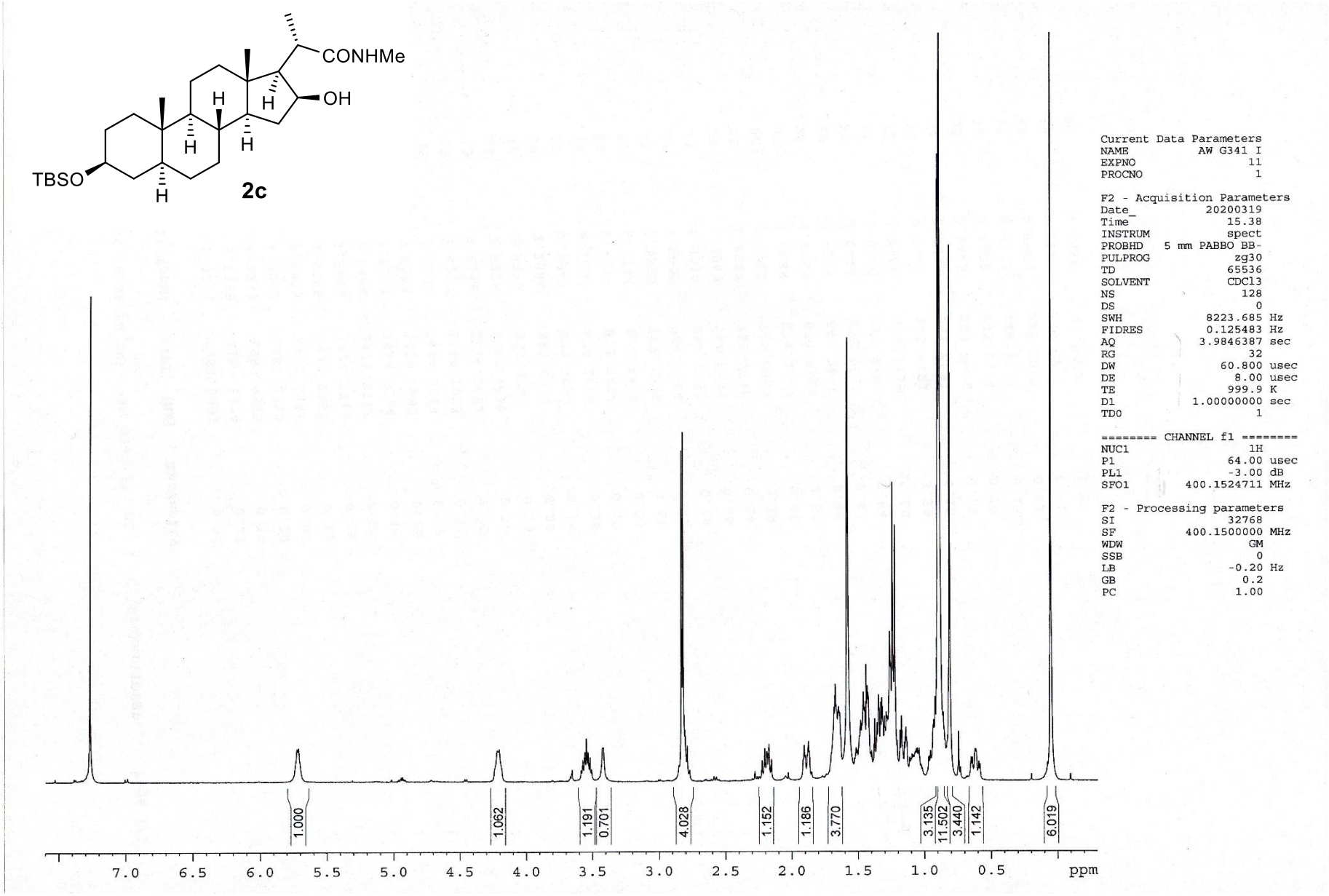
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DE 8.00 usec
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TD0 1
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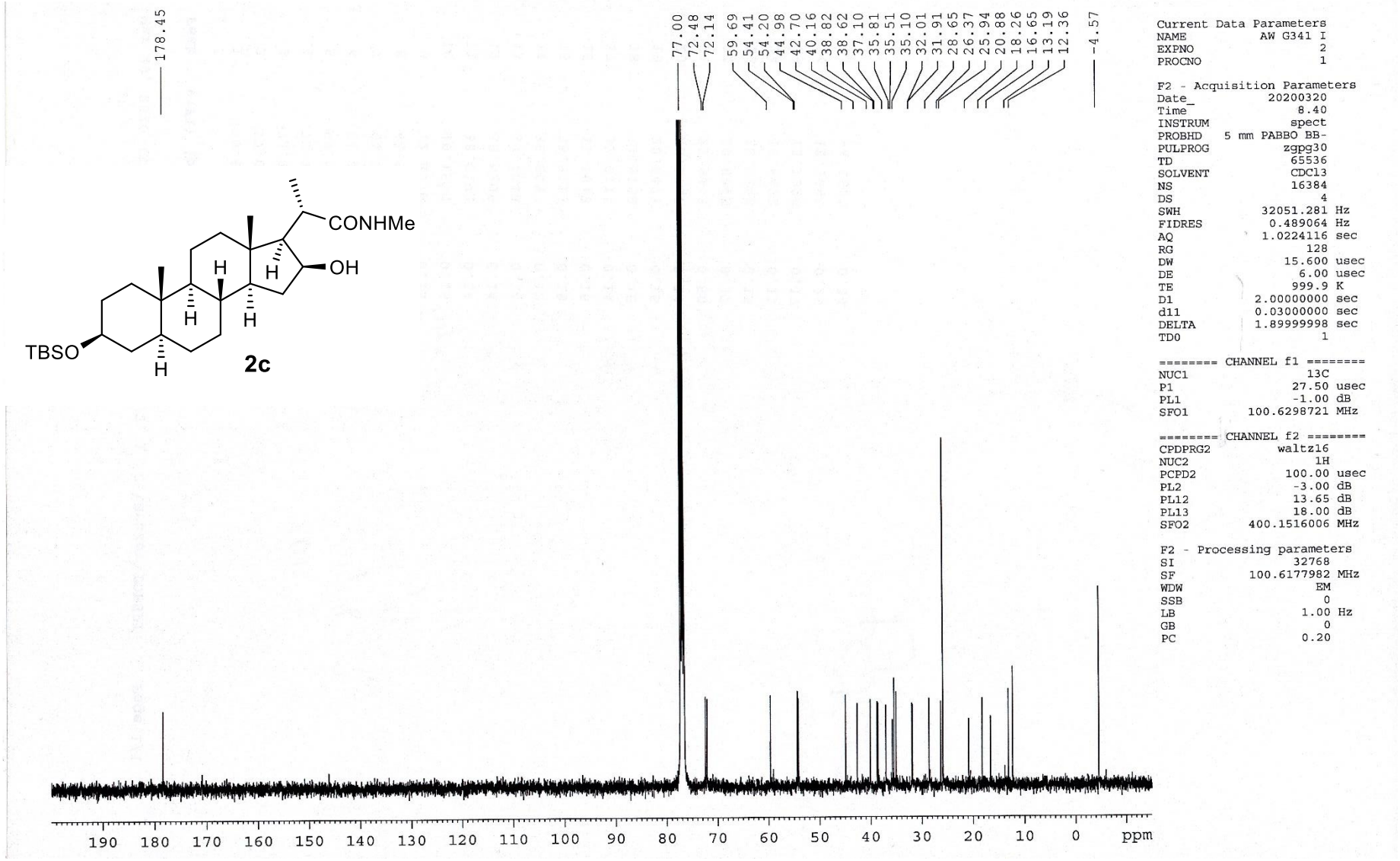
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d11 0.03000000 sec
DELTA 1.89999998 sec
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SFO1 100.6298721 MHz
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NUC2 1H
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PL12 13.65 dB
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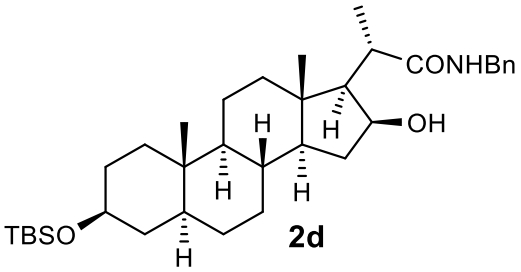

2d


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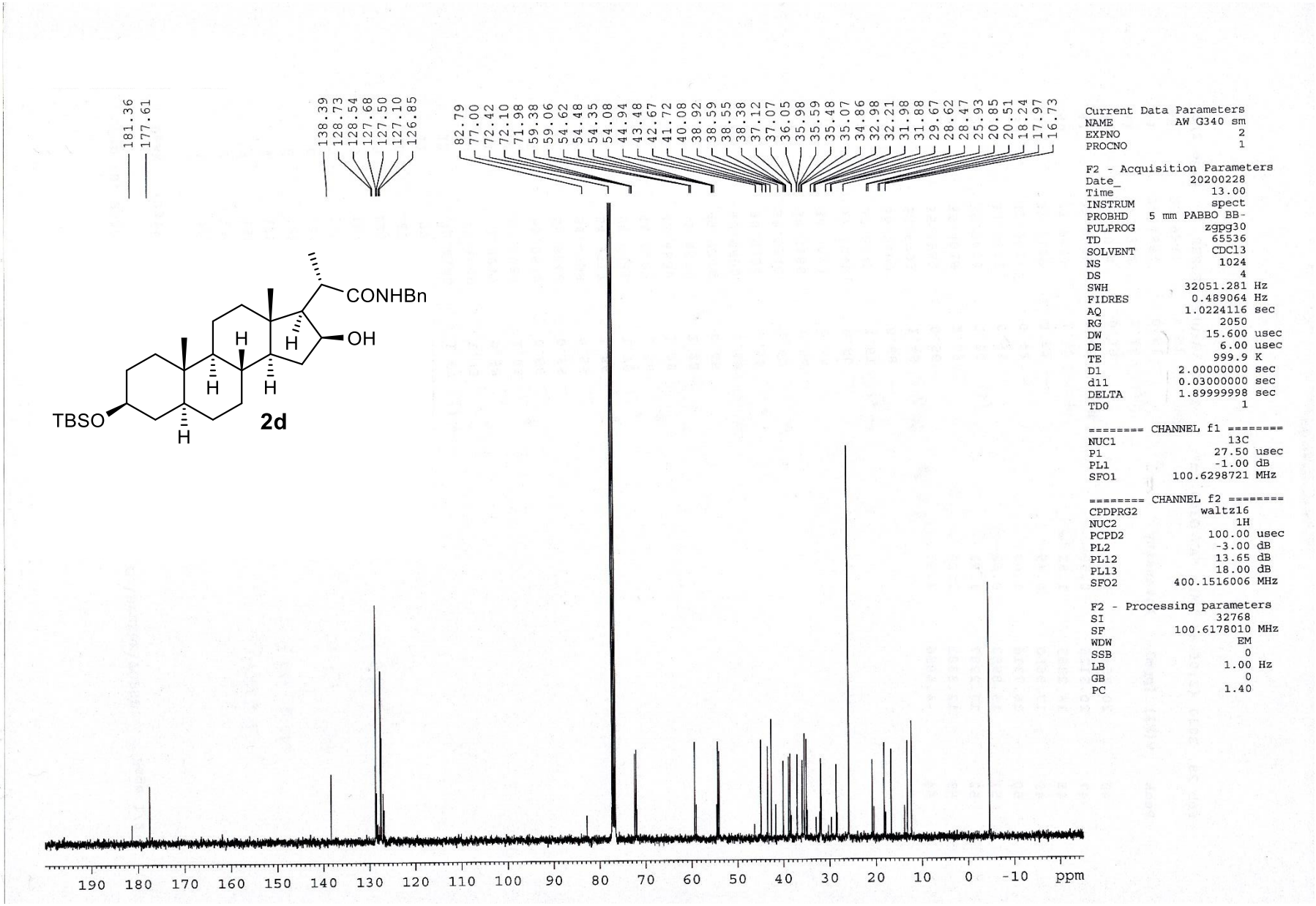
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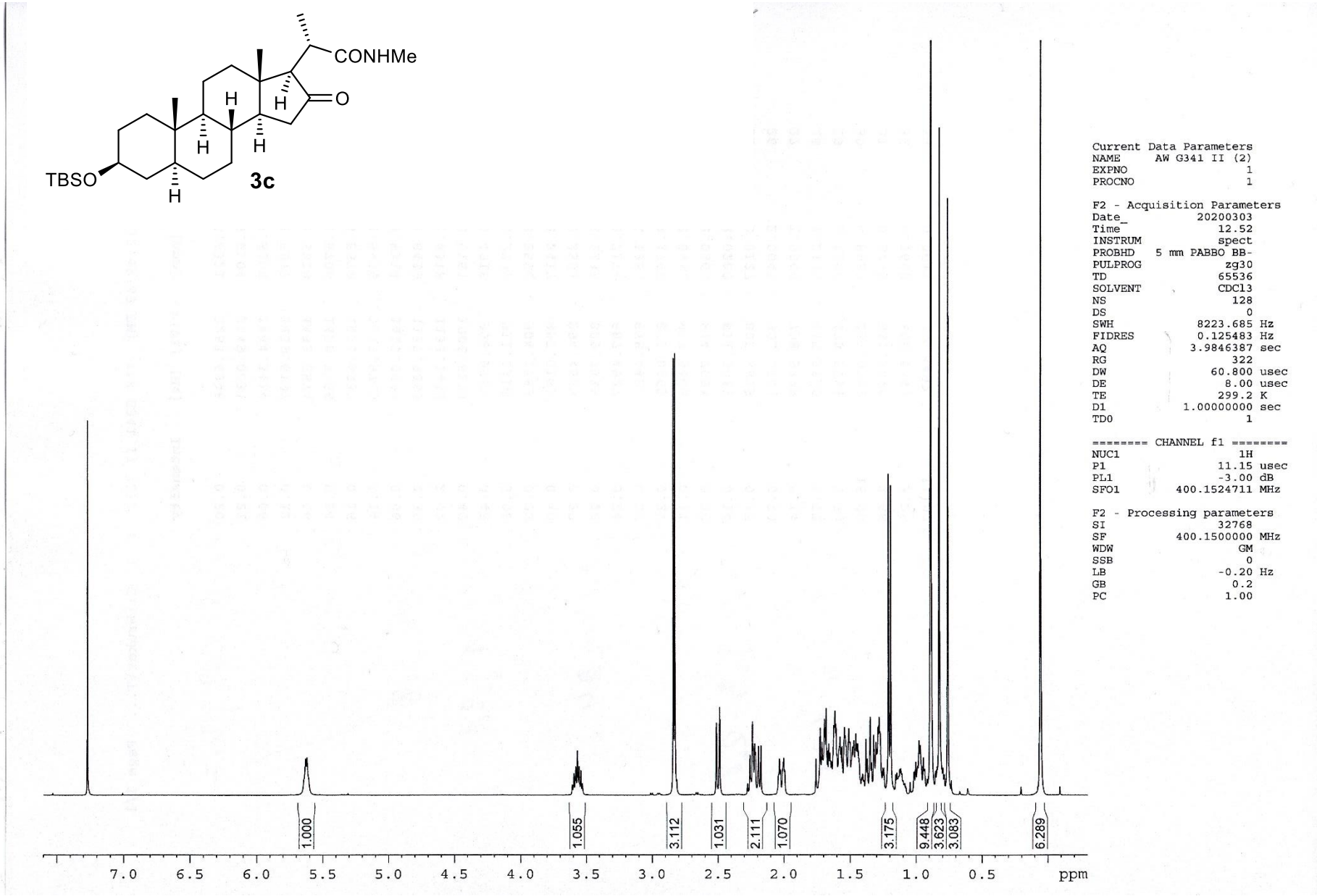
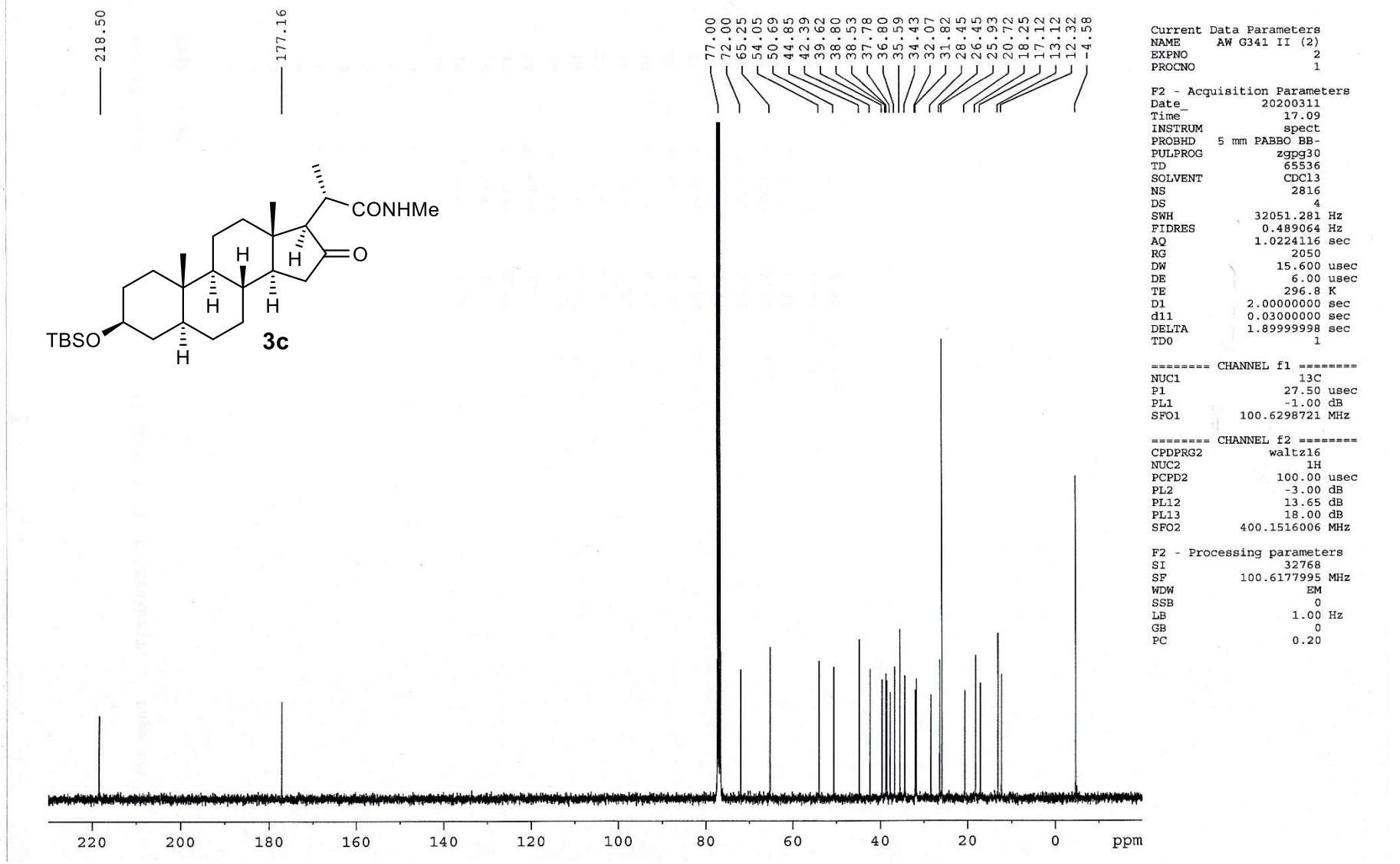
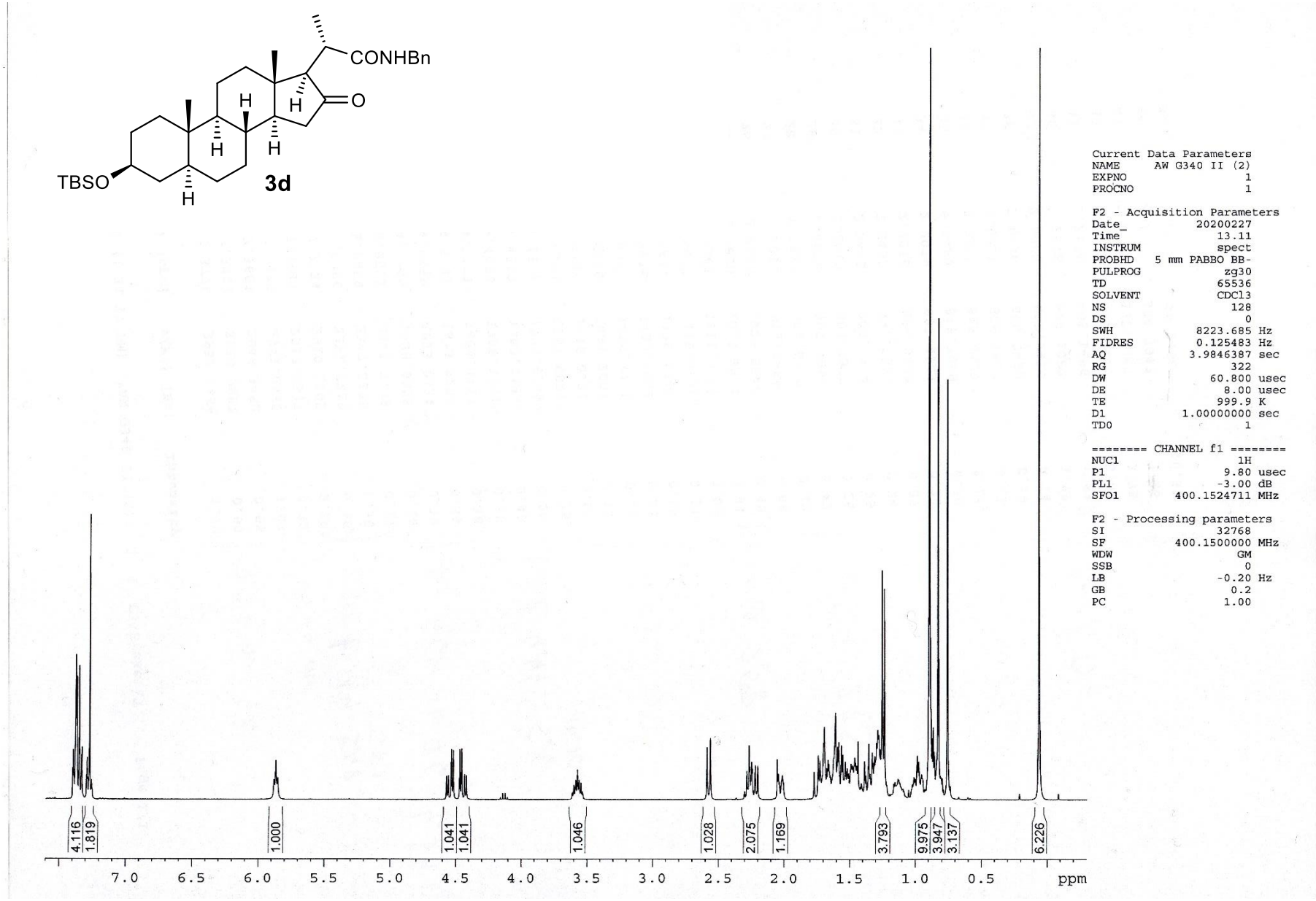
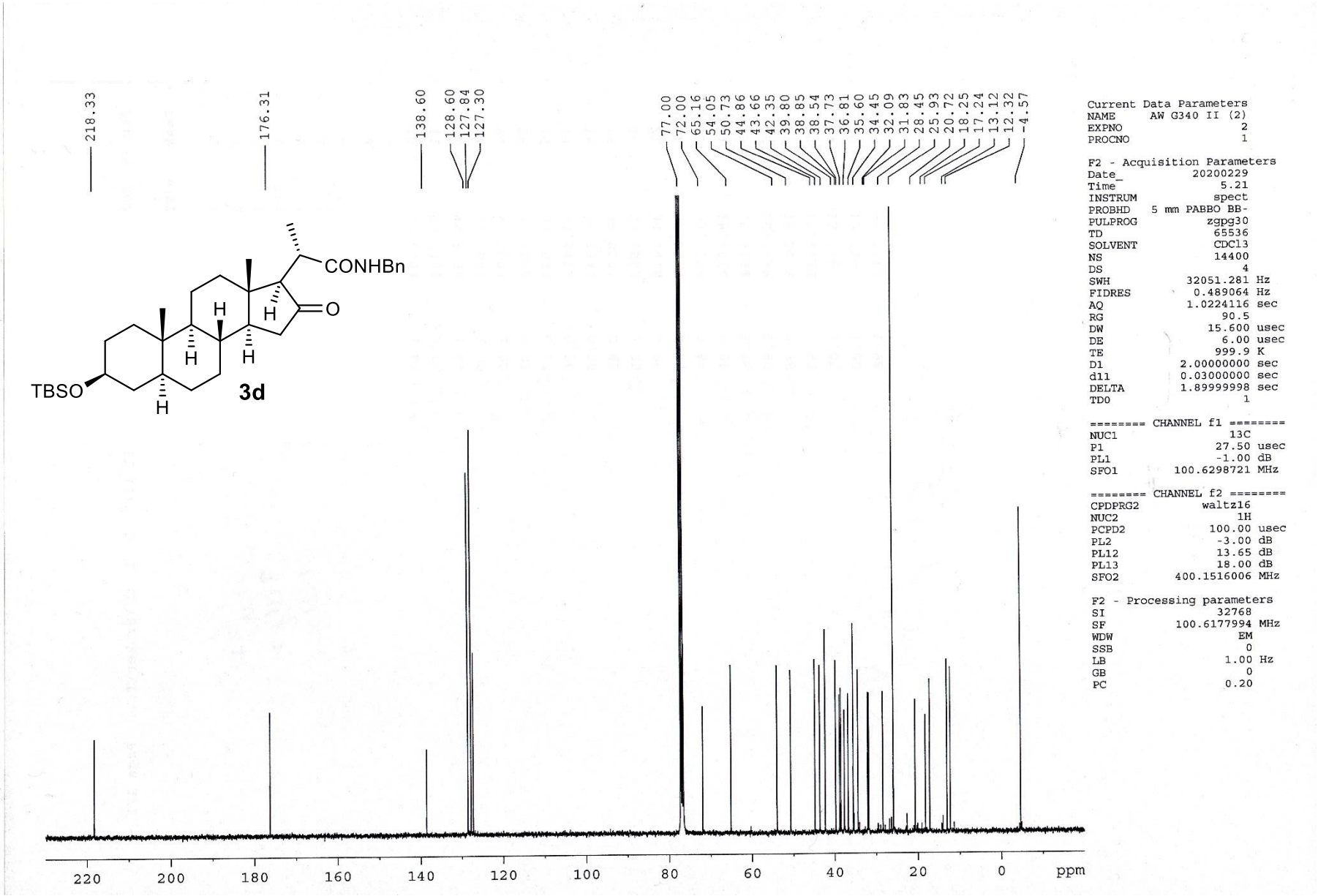
41.72
40.08
38.92
38.59
38.55
38.38
37.12
37.07
36.05
35.98
35.59
35.48
35.07
34.86
32.98
32.21
31.98
31.88
29.67
28.62
28.47
25.93
20.85
20.51
18.24
17.97
16.73
CONHBn
OH
H
H
H
H
H
TBSO
2d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm
Current Data Parameters
NAME AW G340 sm
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200228
Time 13.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 32051.281 Hz
FIDRES 0.489064 Hz
AQ 1.0224116 sec
RG 2050
DW 15.600 usec
DE 6.00 usec
TE 999.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 27.50 usec
PL1 -1.00 dB
SFO1 100.6298721 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 13.65 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
F2 - Processing parameters
SI 32768
SF 100.6178010 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

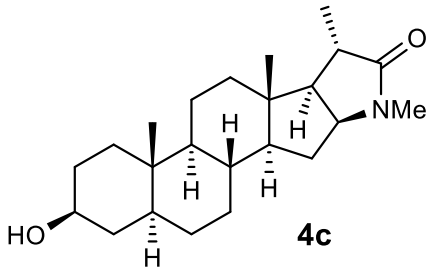

CONHMe
O
H
H
H
H
TBSO
H
3c
Current Data Parameters
NAME AW G341 II (2)
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200303
Time 12.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 322
DW 60.800 usec
DE 8.00 usec
TE 299.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.15 usec
PL1 -3.00 dB
SFO1 400.1524711 MHz
F2 - Processing parameters
SI 32768
SF 400.1500000 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.2
PC 1.00
1.000
1.055
3.112
1.031
2.111
1.070
3.175
9.448
3.623
3.083
6.289
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
0.5
ppm




```
Current Data Parameters
NAME      AW G341 II (2)
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20200311
Time              17.09
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                2816
DS                   4
SWH          32051.281 Hz
FIDRES        0.489064 Hz
AQ           1.0224116 sec
RG                2050
DW              15.600 usec
DE               6.00 usec
TE              296.8 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1               27.50 usec
PL1              -1.00 dB
SFO1        100.6298721 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2              -3.00 dB
PL12             13.65 dB
PL13             18.00 dB
SFO2        400.1516006 MHz

F2 - Processing parameters
SI                32768
SF          100.6177995 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               0.20
```

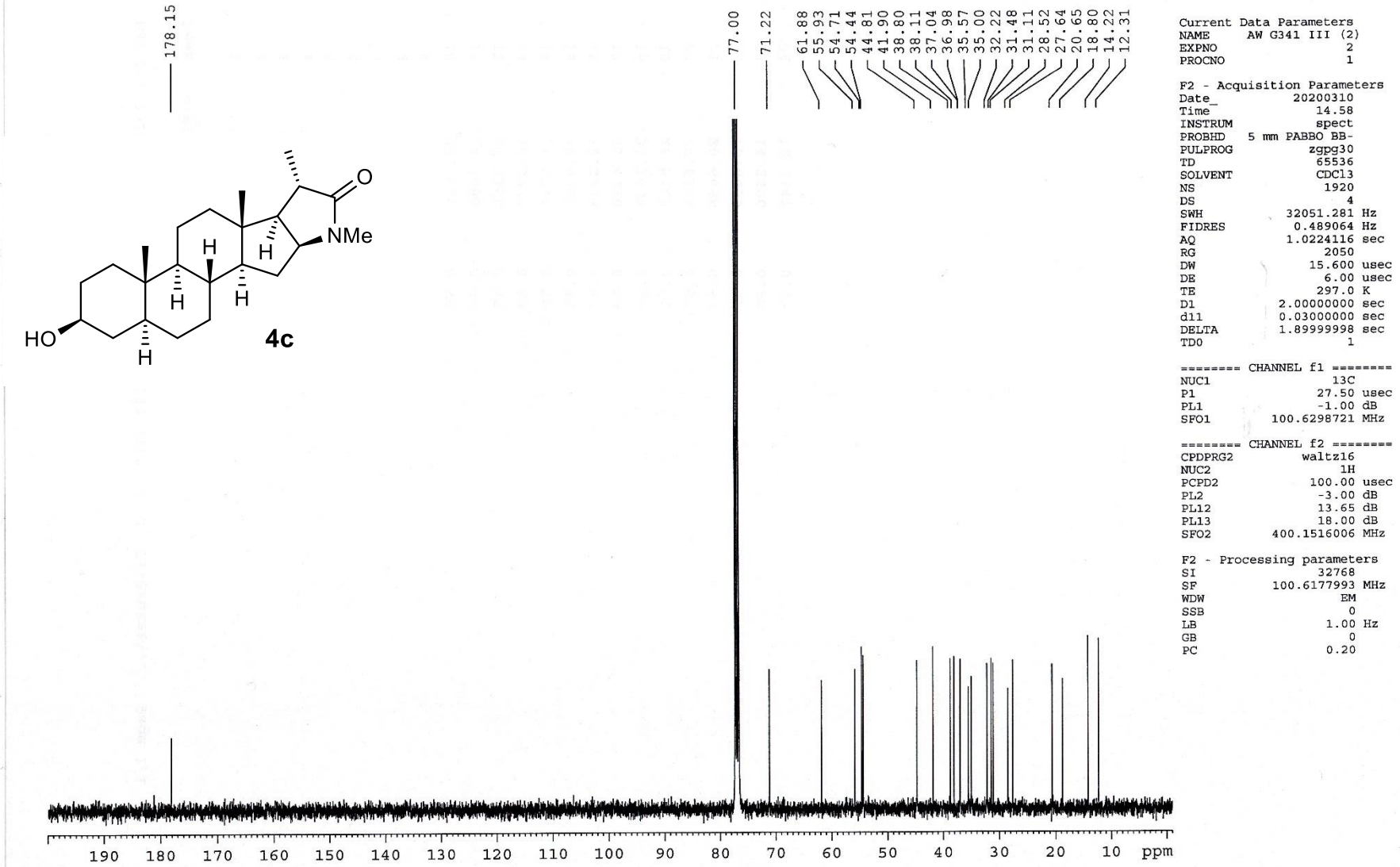

CONHBn
O
H
H
H
H
TBSO
H
3d
4.116
1.819
1.000
1.041
1.041
1.046
1.028
2.075
1.169
3.793
9.975
3.947
3.137
6.226
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
Current Data Parameters
NAME AW G340 II (2)
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200227
Time 13.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 322
DW 60.800 usec
DE 8.00 usec
TE 999.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -3.00 dB
SFO1 400.1524711 MHz
F2 - Processing parameters
SI 32768
SF 400.1500000 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.2
PC 1.00


218.33
176.31
138.60
128.60
127.84
127.30
77.00
72.00
65.16
54.05
50.73
44.86
43.66
42.35
39.80
38.85
38.54
37.73
36.81
35.60
34.45
32.09
31.83
28.45
25.93
20.72
18.25
17.24
13.12
12.32
-4.57
CONHBn
O
H
H
H
H
H
TBSO
3d
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME AW G340 II (2)
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200229
Time 5.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 14400
DS 4
SWH 32051.281 Hz
FIDRES 0.489064 Hz
AQ 1.0224116 sec
RG 90.5
DW 15.600 usec
DE 6.00 usec
TE 999.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 27.50 usec
PL1 -1.00 dB
SFO1 100.6298721 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 13.65 dB
PL13 18.00 dB
SFO2 400.1516006 MHz
F2 - Processing parameters
SI 32768
SF 100.6177994 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.20

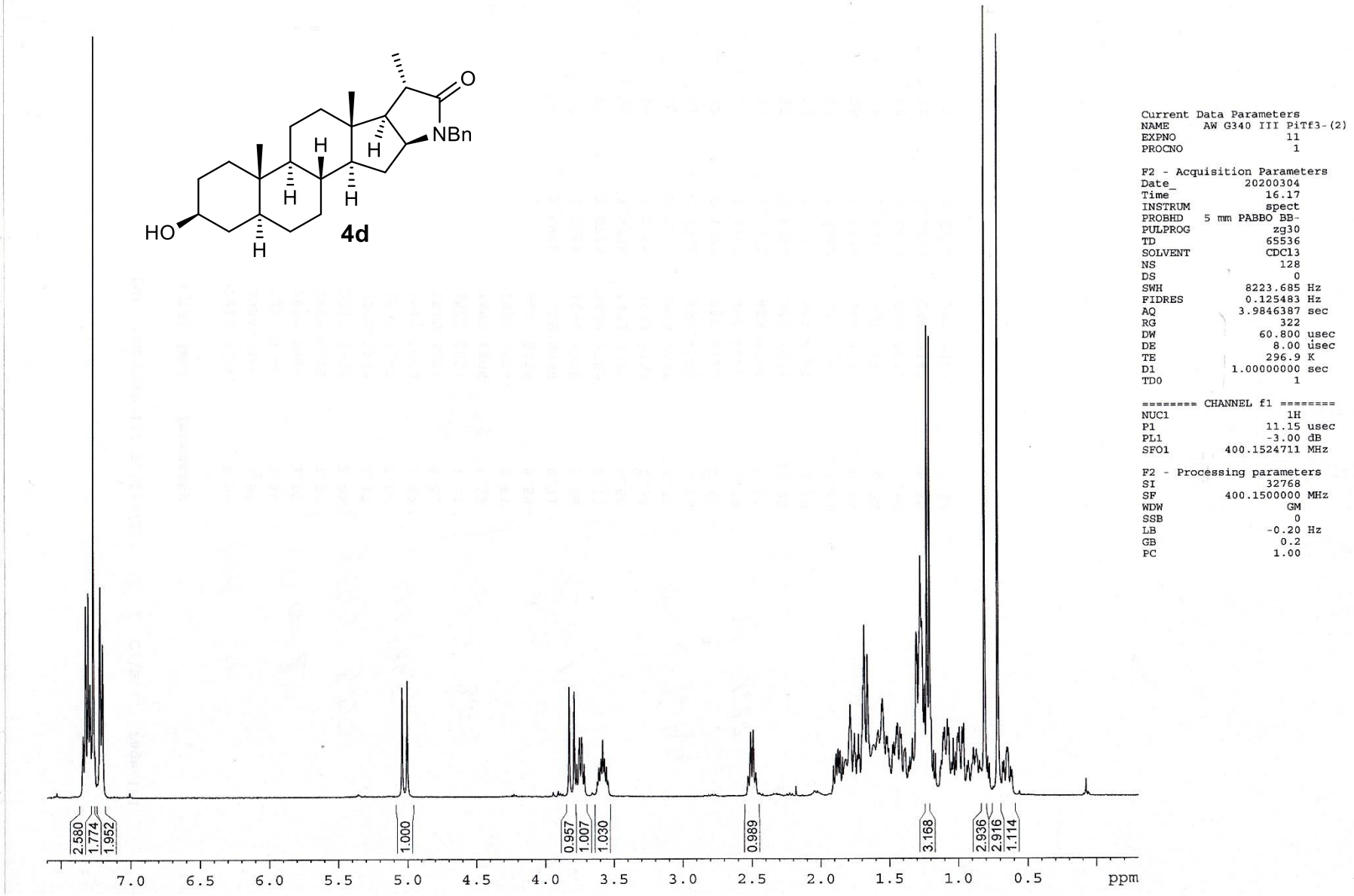


4c

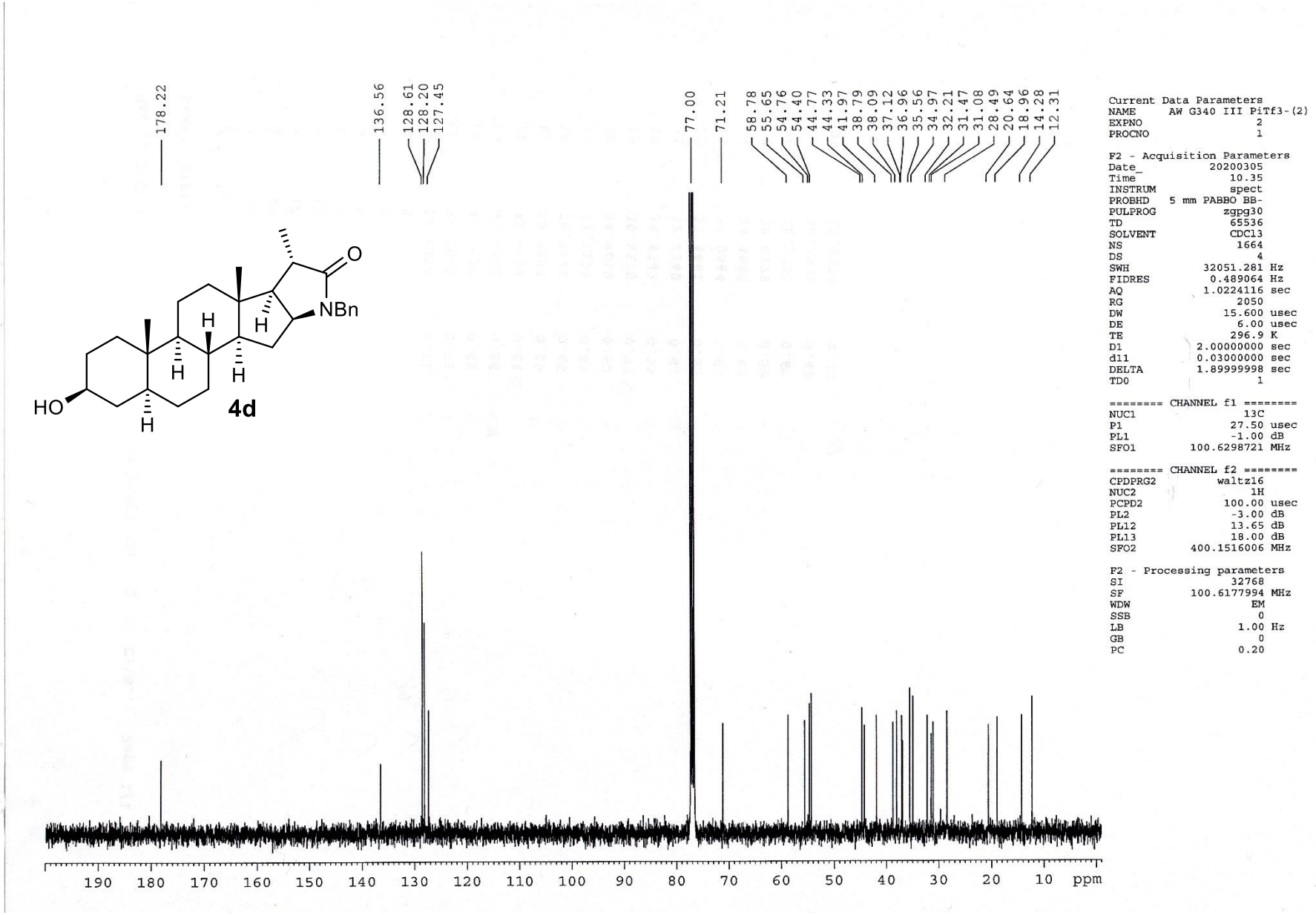

```
Current Data Parameters
NAME      AW G341 III (2)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200304
Time               17.19
INSTRUM            spect
PROBHD  5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                   128
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   322
DW                60.800 usec
DE                 8.00 usec
TE                296.9 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.15 usec
PL1                -3.00 dB
SFO1          400.1524711 MHz

F2 - Processing parameters
SI                 32768
SF          400.1500000 MHz
WDW                   GM
SSB                    0
LB                 -0.20 Hz
GB                   0.2
PC                  1.00
```




```
Current Data Parameters
NAME      AW G341 III (2)
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20200310
Time                 14.58
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1920
DS                        4
SWH              32051.281 Hz
FIDRES            0.489064 Hz
AQ               1.0224116 sec
RG                     2050
DW                 15.600 usec
DE                  6.00 usec
TE                  297.0 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                  27.50 usec
PL1                 -1.00 dB
SFO1           100.6298721 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                 -3.00 dB
PL12                13.65 dB
PL13                18.00 dB
SFO2           400.1516006 MHz

F2 - Processing parameters
SI                     32768
SF             100.6177993 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      0.20
```




```
Current Data Parameters
NAME      AW G340 III PiTf3-(2)
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_              20200304
Time                  16.17
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                      128
DS                        0
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                      322
DW                 60.800 usec
DE                  8.00 usec
TE                  296.9 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                 11.15 usec
PL1                 -3.00 dB
SFO1           400.1524711 MHz

F2 - Processing parameters
SI                    32768
SF             400.1500000 MHz
WDW                      GM
SSB                       0
LB                  -0.20 Hz
GB                       0.2
PC                      1.00
```



Current Data Parameters

NAME	AW G340 III PiTf3-(2)
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20200305
Time	10.35
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1664
DS	4
SWH	32051.281 Hz
FIDRES	0.489064 Hz
AQ	1.0224116 sec
RG	2050
DW	15.600 usec
DE	6.00 usec
TE	296.9 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

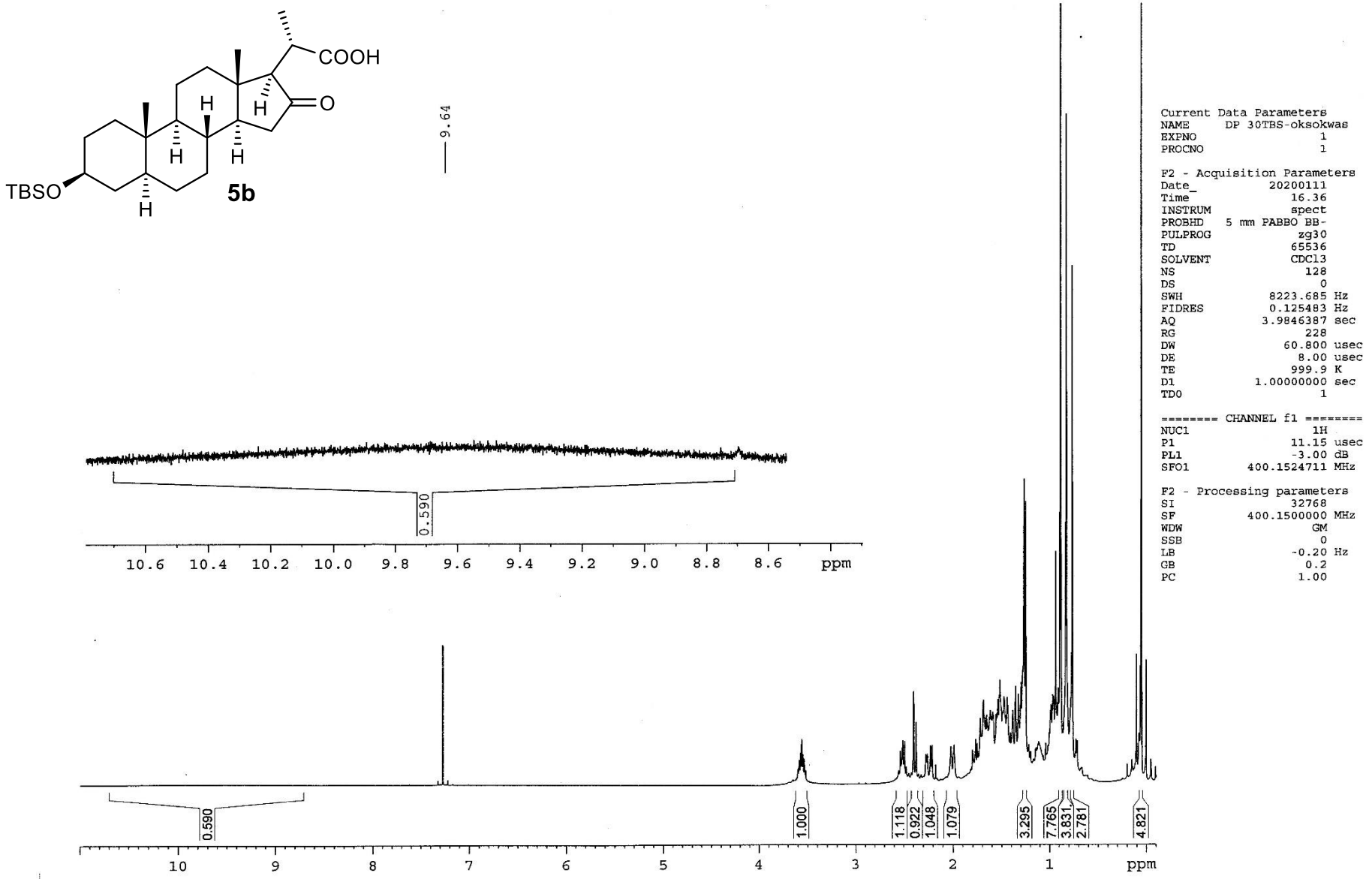
NUC1	13C
P1	27.50 usec
PL1	-1.00 dB
SFO1	100.6298721 MHz

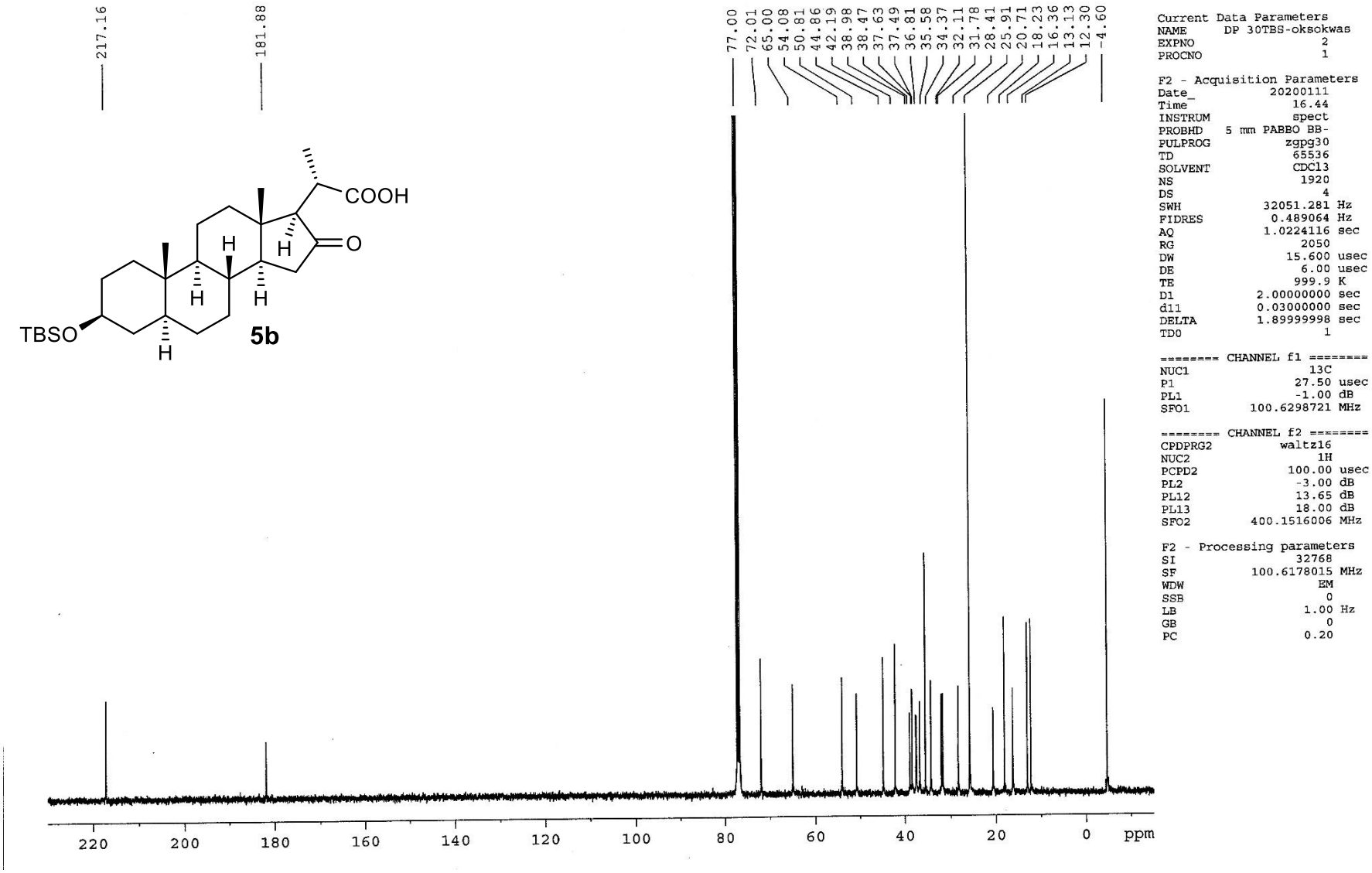
======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-3.00 dB
PL12	13.65 dB
PL13	18.00 dB
SFO2	400.1516006 MHz

F2 - Processing parameters

SI	32768
SF	100.6177994 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.20


COOH
O
H
H
H
H
H
TBSO
5b
9.64
0.590
10.6 10.4 10.2 10.0 9.8 9.6 9.4 9.2 9.0 8.8 8.6 ppm
0.590
1.000
1.118
0.922
1.048
1.079
3.295
7.765
3.831
2.781
4.821
10 9 8 7 6 5 4 3 2 1 ppm
Current Data Parameters
NAME DP 30TBS-oksokwas
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200111
Time 16.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 228
DW 60.800 usec
DE 8.00 usec
TE 999.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.15 usec
PL1 -3.00 dB
SFO1 400.1524711 MHz
F2 - Processing parameters
SI 32768
SF 400.1500000 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.2
PC 1.00



Current Data Parameters

NAME	DP 30TBS-oksokwas
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20200111
Time	16.44
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1920
DS	4
SWH	32051.281 Hz
FIDRES	0.489064 Hz
AQ	1.0224116 sec
RG	2050
DW	15.600 usec
DE	6.00 usec
TE	999.9 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

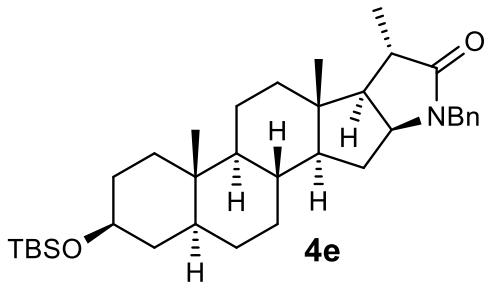
NUC1	13C
P1	27.50 usec
PL1	-1.00 dB
SFO1	100.6298721 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-3.00 dB
PL12	13.65 dB
PL13	18.00 dB
SFO2	400.1516006 MHz

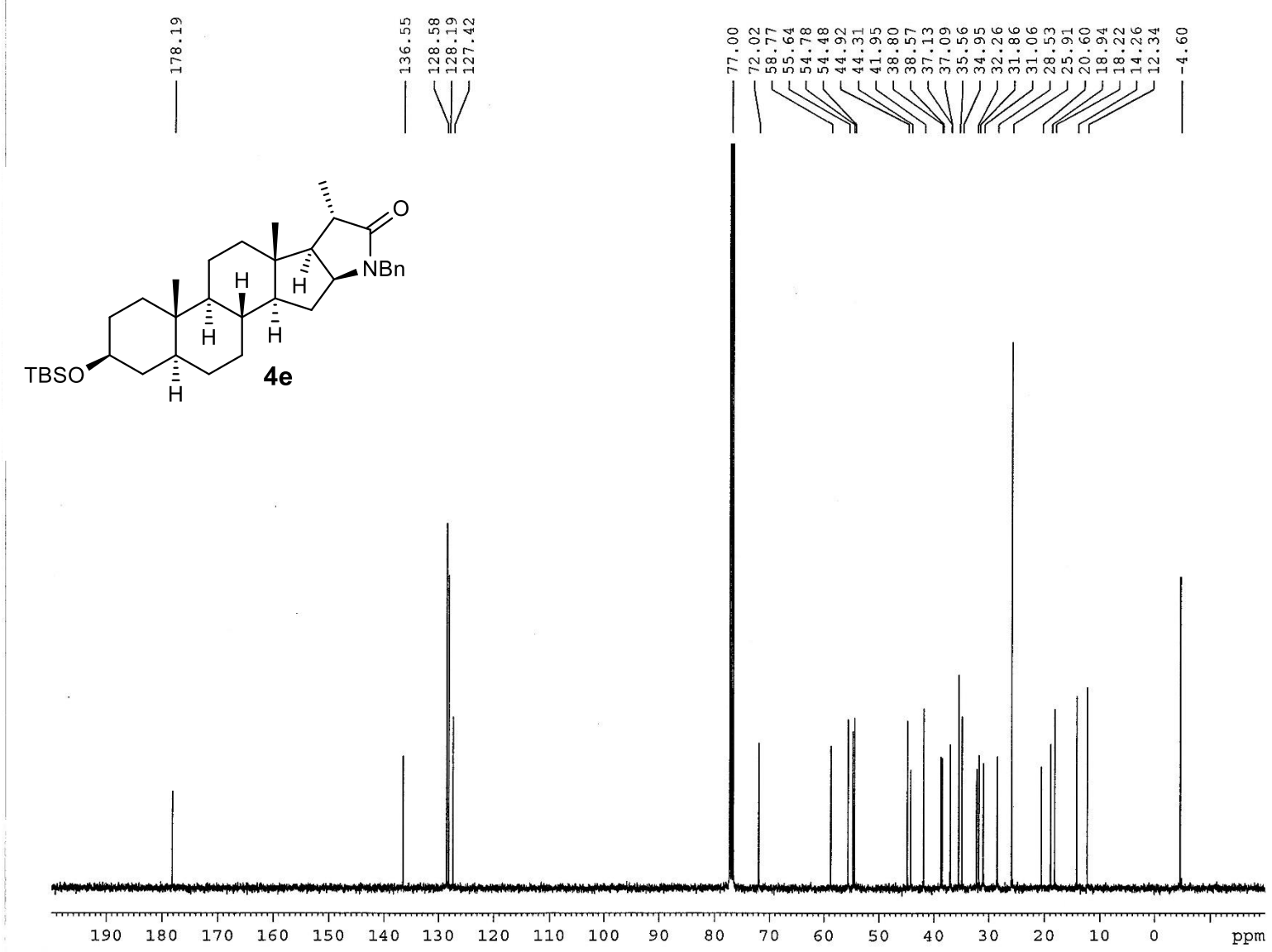
F2 - Processing parameters

SI	32768
SF	100.6178015 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	0.20

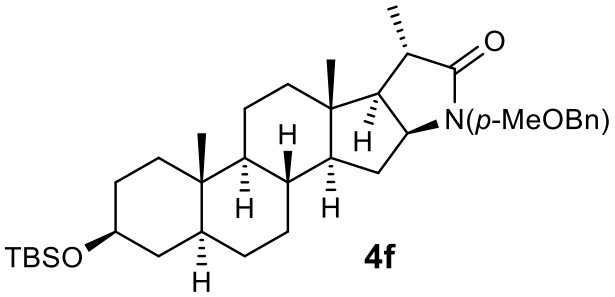


4e


```
Current Data Parameters
NAME          AB 601D-z3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200304
Time              13.46
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    0
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ            3.9846387 sec
RG                  256
DW               60.800 usec
DE                8.00 usec
TE               296.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.15 usec
PL1               -3.00 dB
SFO1        400.1524711 MHz

F2 - Processing parameters
SI                 32768
SF          400.1500008 MHz
WDW                   GM
SSB                    0
LB               -0.20 Hz
GB                   0.2
PC                  1.00
```

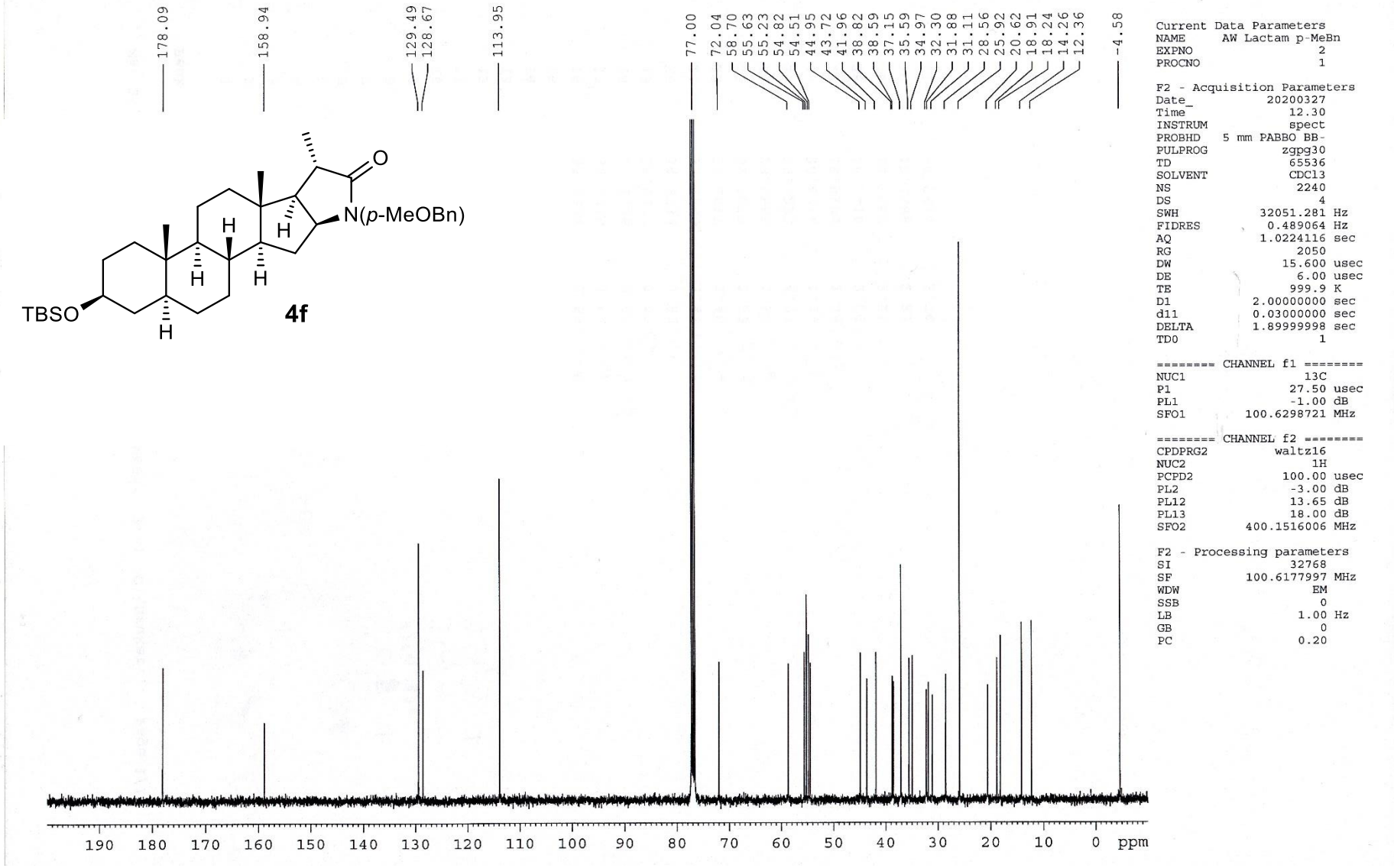



```
Current Data Parameters
NAME          AB 601D-z3
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200306
Time               18.46
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  768
DS                    4
SWH           32051.281 Hz
FIDRES         0.489064 Hz
AQ            1.0224116 sec
RG                 2050
DW               15.600 usec
DE                6.00 usec
TE               297.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 27.50 usec
PL1                -1.00 dB
SFO1        100.6298721 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                -3.00 dB
PL12               13.65 dB
PL13               18.00 dB
SFO2        400.1516006 MHz

F2 - Processing parameters
SI                 32768
SF          100.6178015 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



4f


```
Current Data Parameters
NAME      AW Lactam p-MeBn
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20200327
Time              11.54
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    0
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  161
DW             60.800 usec
DE              8.00 usec
TE              999.9 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                1H
P1             11.15 usec
PL1             -3.00 dB
SFO1      400.1524711 MHz

F2 - Processing parameters
SI                32768
SF        400.1500000 MHz
WDW                  GM
SSB                   0
LB             -0.20 Hz
GB                  0.2
PC                 1.00
```




```
Current Data Parameters
NAME      AW Lactam p-MeBn
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20200327
Time                 12.30
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     2240
DS                        4
SWH              32051.281 Hz
FIDRES           0.489064 Hz
AQ               1.0224116 sec
RG                     2050
DW                15.600 usec
DE                 6.00 usec
TE                 999.9 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                  27.50 usec
PL1                 -1.00 dB
SFO1          100.6298721 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                 -3.00 dB
PL12                13.65 dB
PL13                18.00 dB
SFO2          400.1516006 MHz

F2 - Processing parameters
SI                    32768
SF            100.6177997 MHz
WDW                      EM
SSB                       0
LB                   1.00 Hz
GB                        0
PC                     0.20
```