

## Supplementary Materials for

### Synthesis of demissidine analogues from tigogenin via imine intermediates<sup>†</sup>

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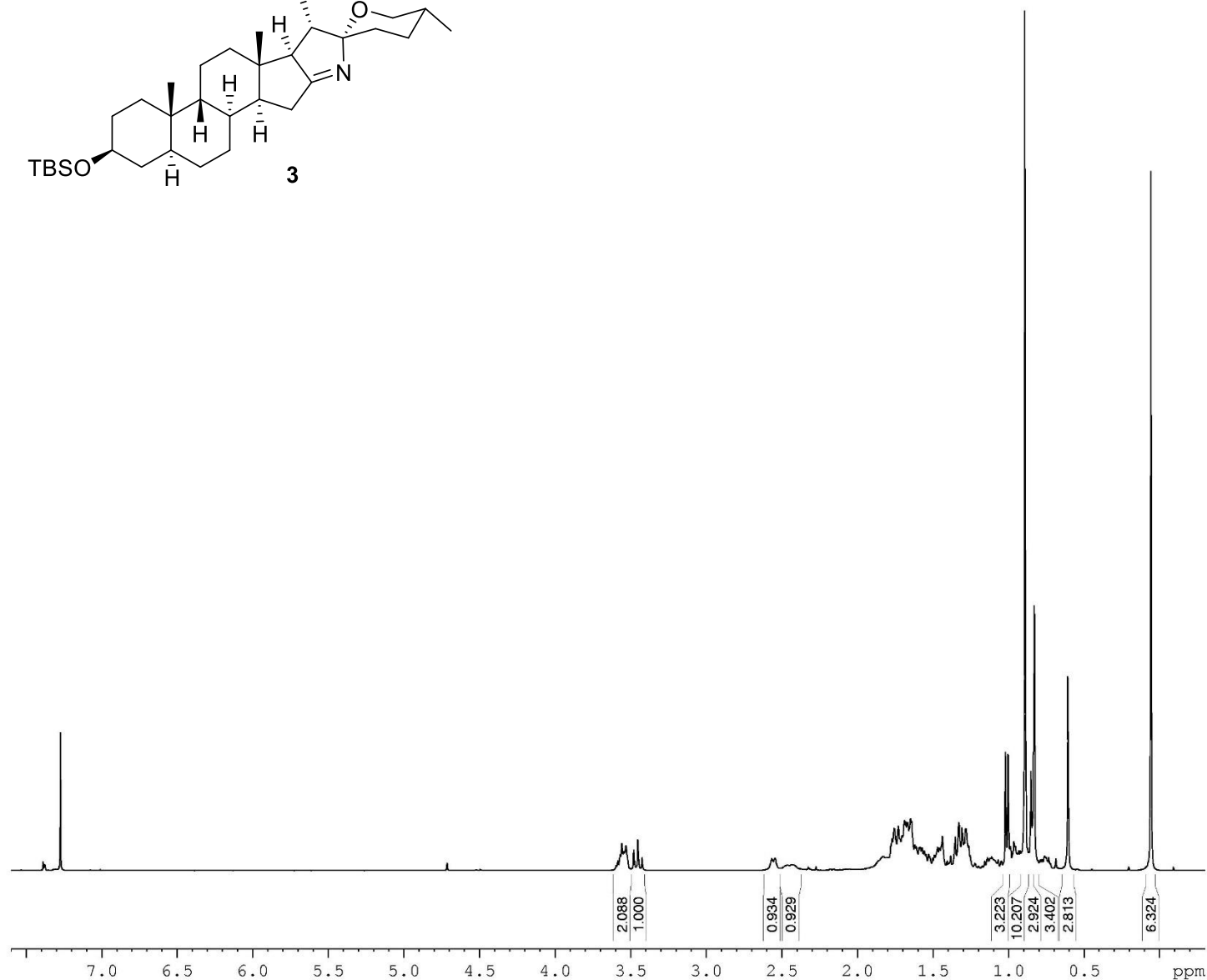
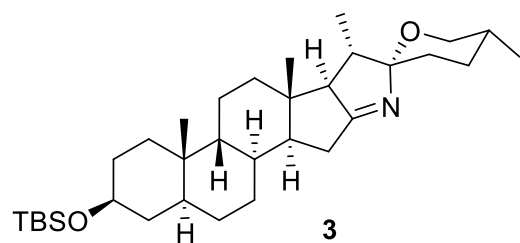
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† Dedicated to Prof. Dr. Ludger Wessjohann on the occasion of his 60th birthday

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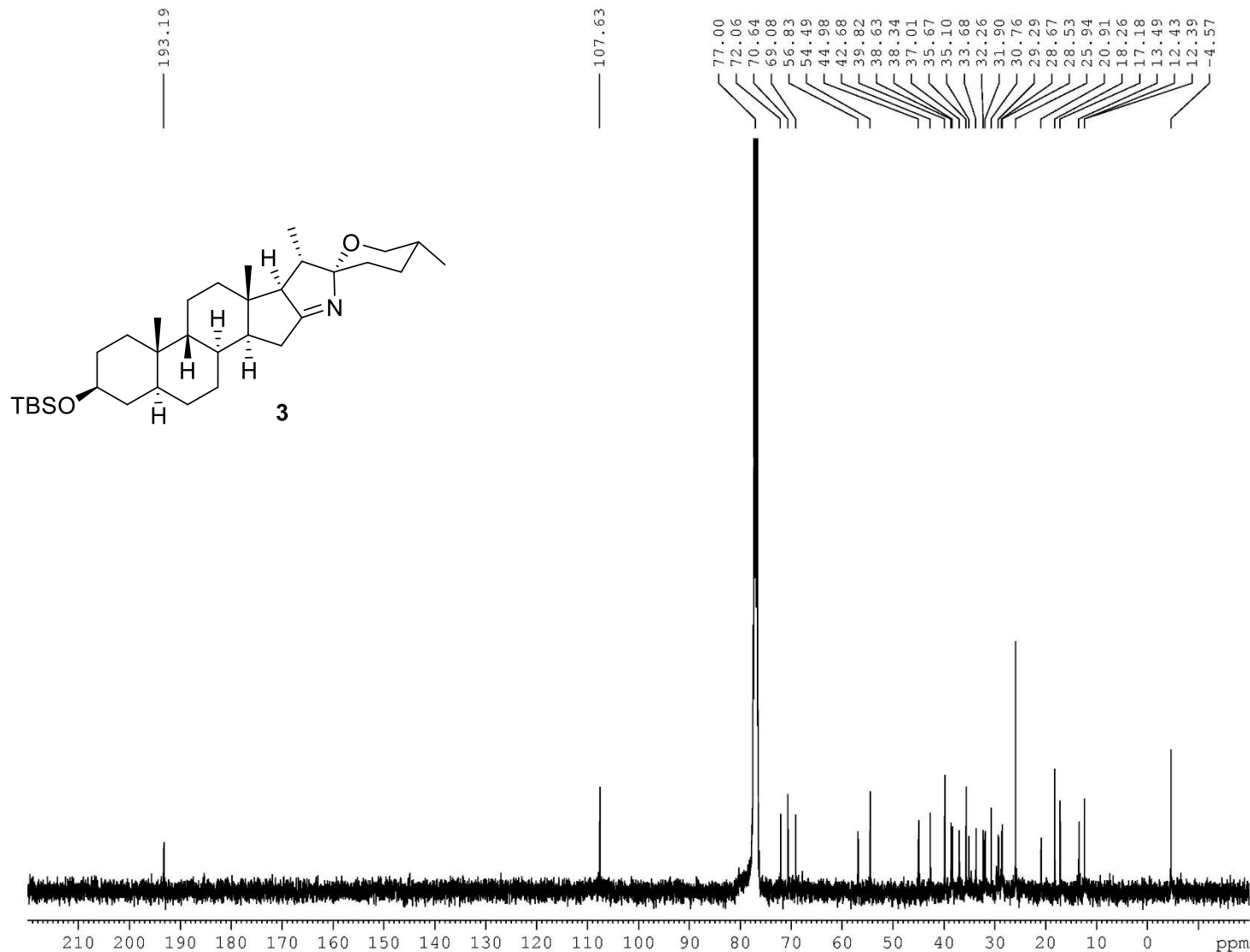


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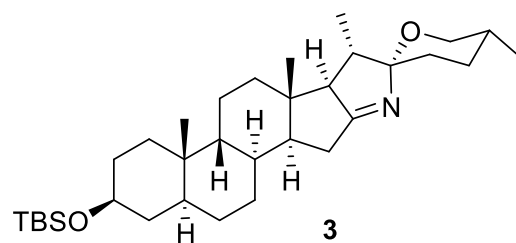
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 AQ 1.0224116 se  
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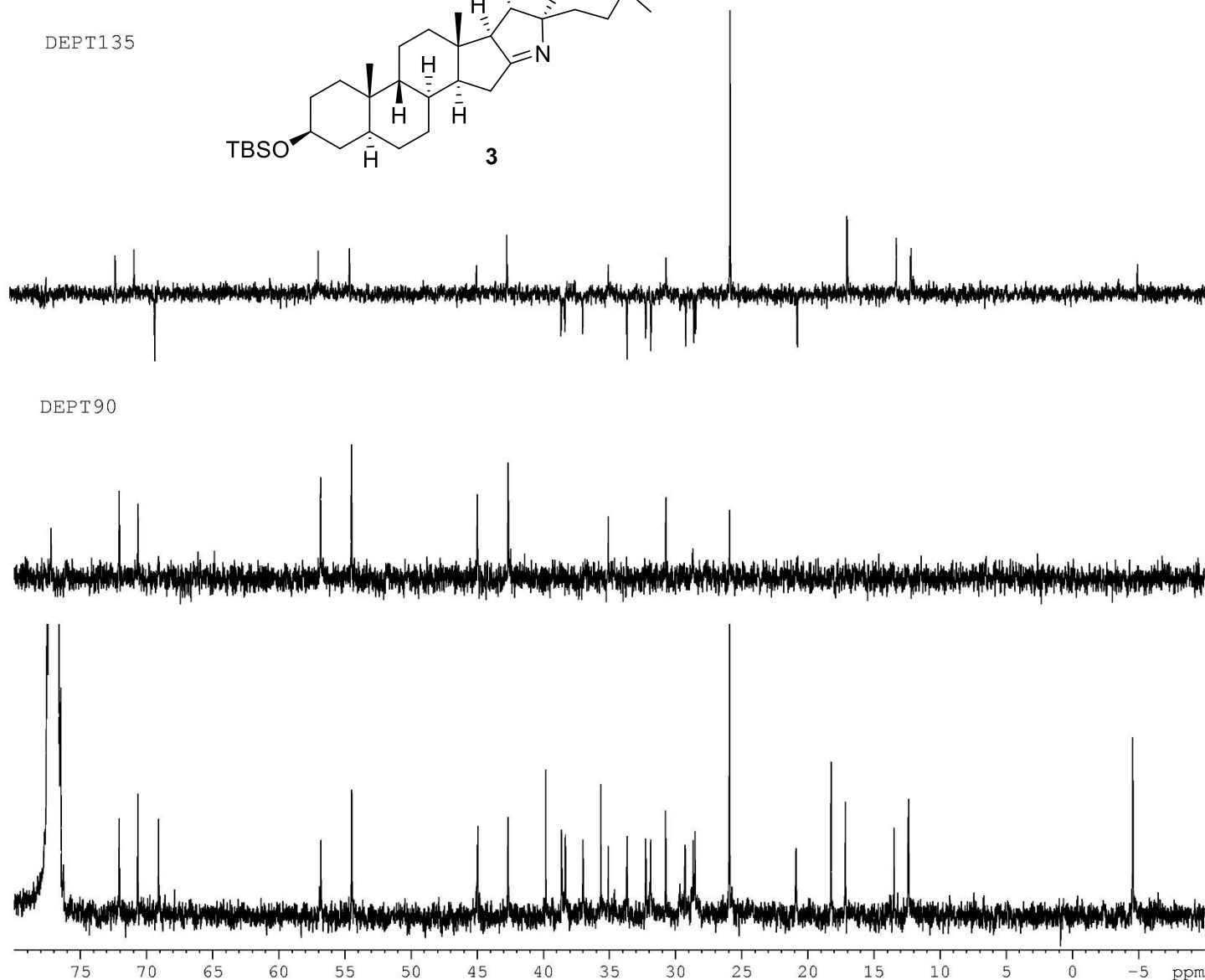
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DEPT135



DEPT90



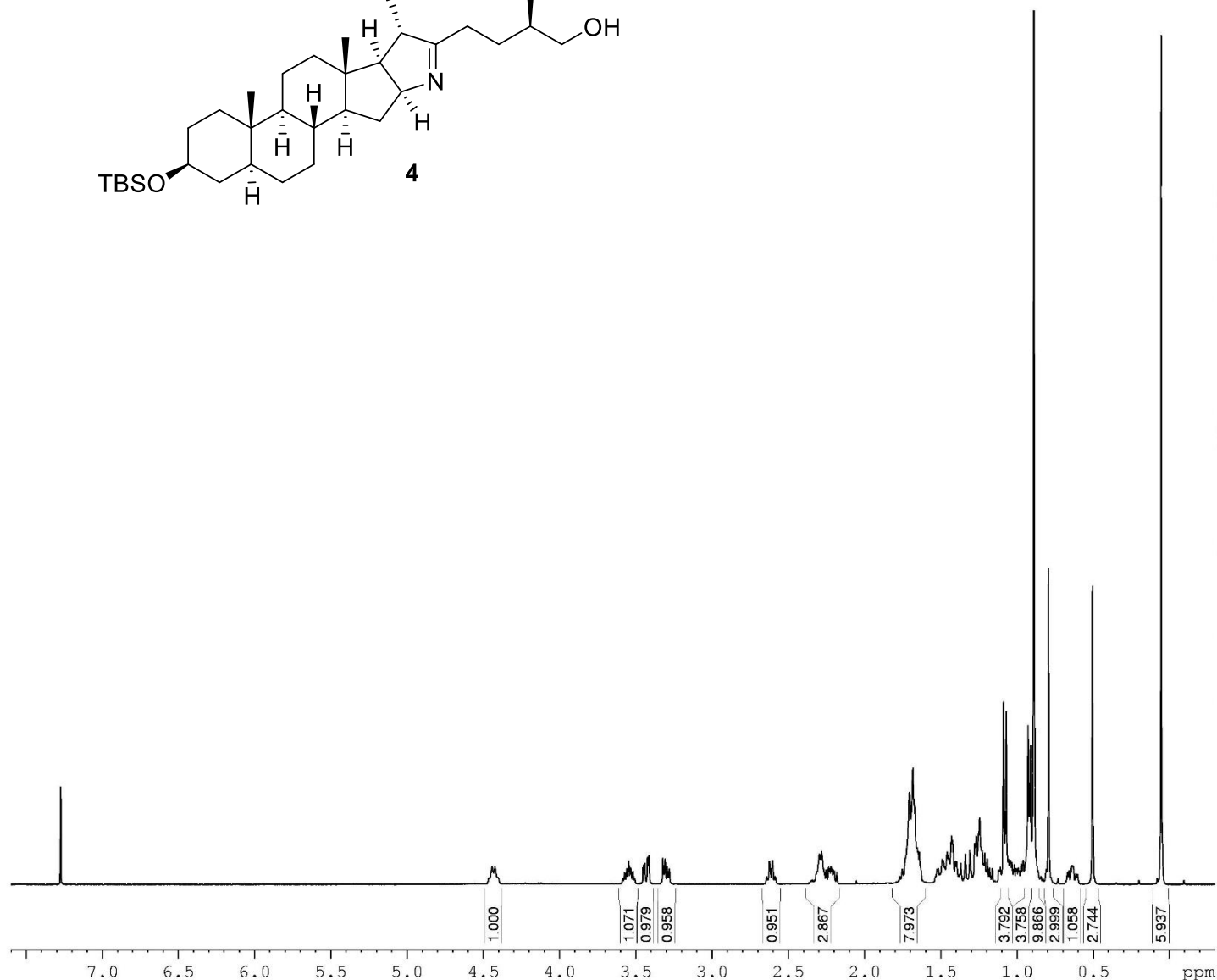
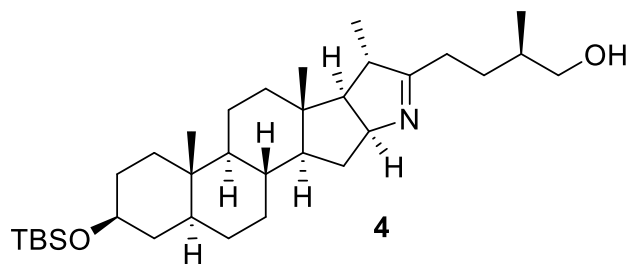
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DS 4  
SWH 36231.883 Hz  
FIDRES 0.552855 Hz  
AQ 0.9044468 se  
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DE 6.00 us  
TE 999.9 K  
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PL12 13.65 dB  
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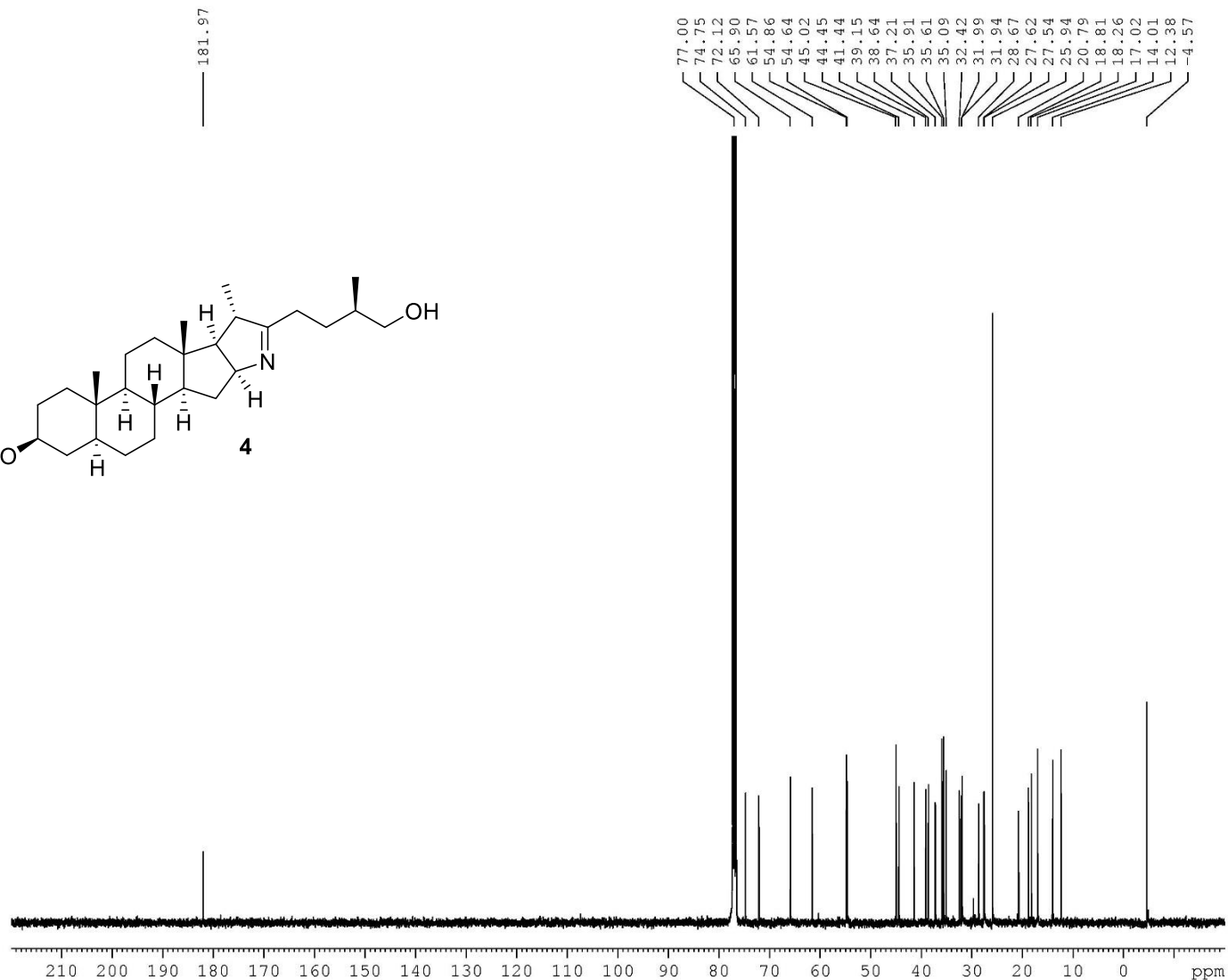
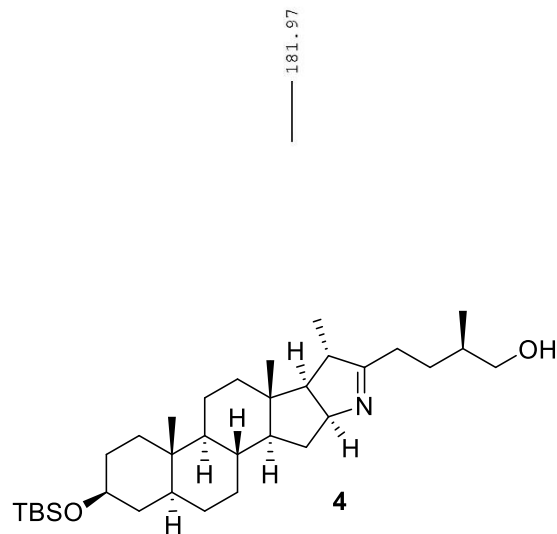


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 SOLVENT CDCl3  
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 DS 0  
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 FIDRES 0.125483 Hz  
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 RG 322  
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 D1 1.00000000 se  
 TD0 1

===== CHANNEL f1 =====  
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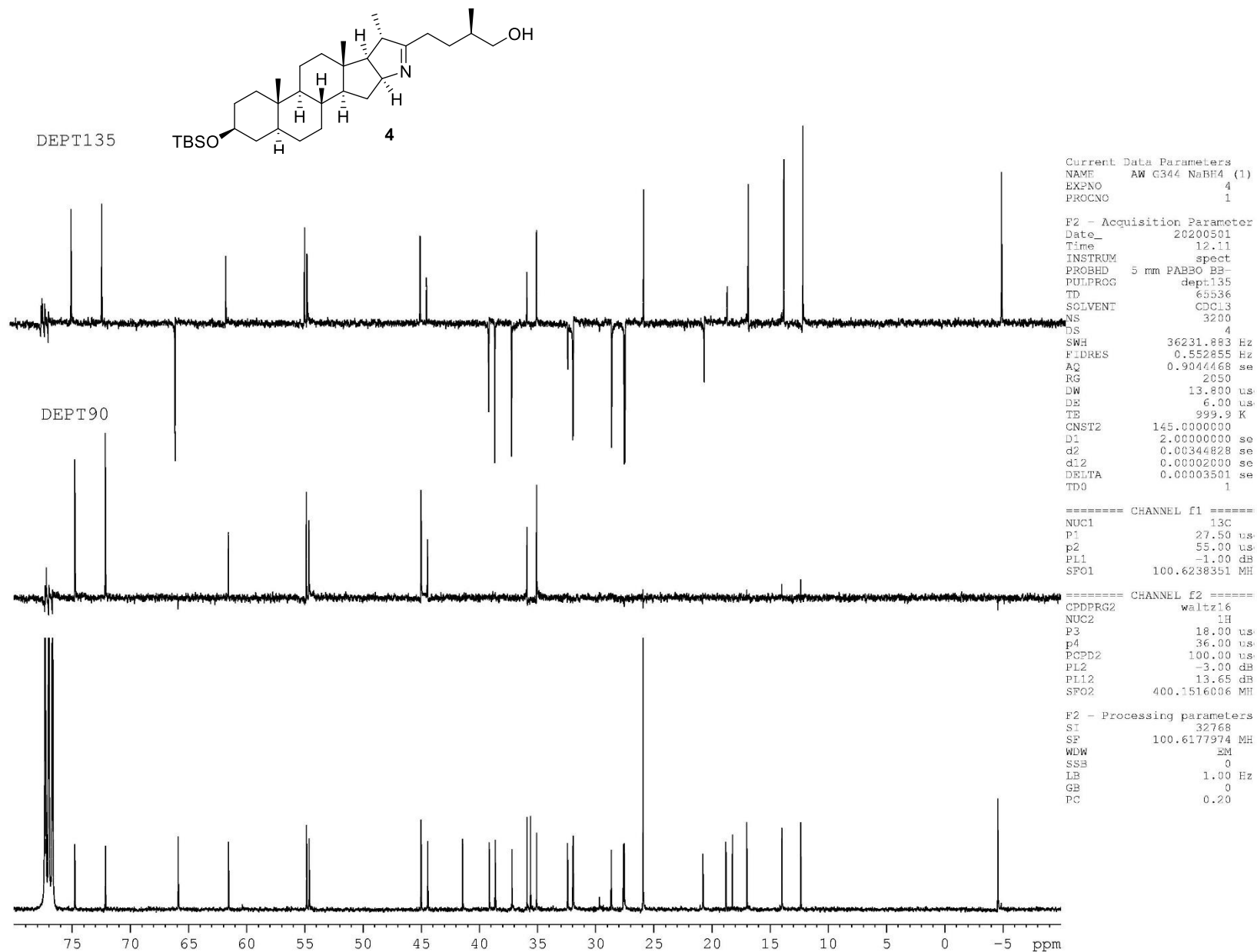
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 SOLVENT CDCL3  
 NS 14400  
 DS 4  
 SWH 32051.281 Hz  
 FIDRES 0.489064 Hz  
 AQ 1.0224116 se  
 RG 114  
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 TE 999.9 K  
 D1 2.0000000 se  
 d11 0.0300000 se  
 DELTA 1.89999998 se  
 TD0 1

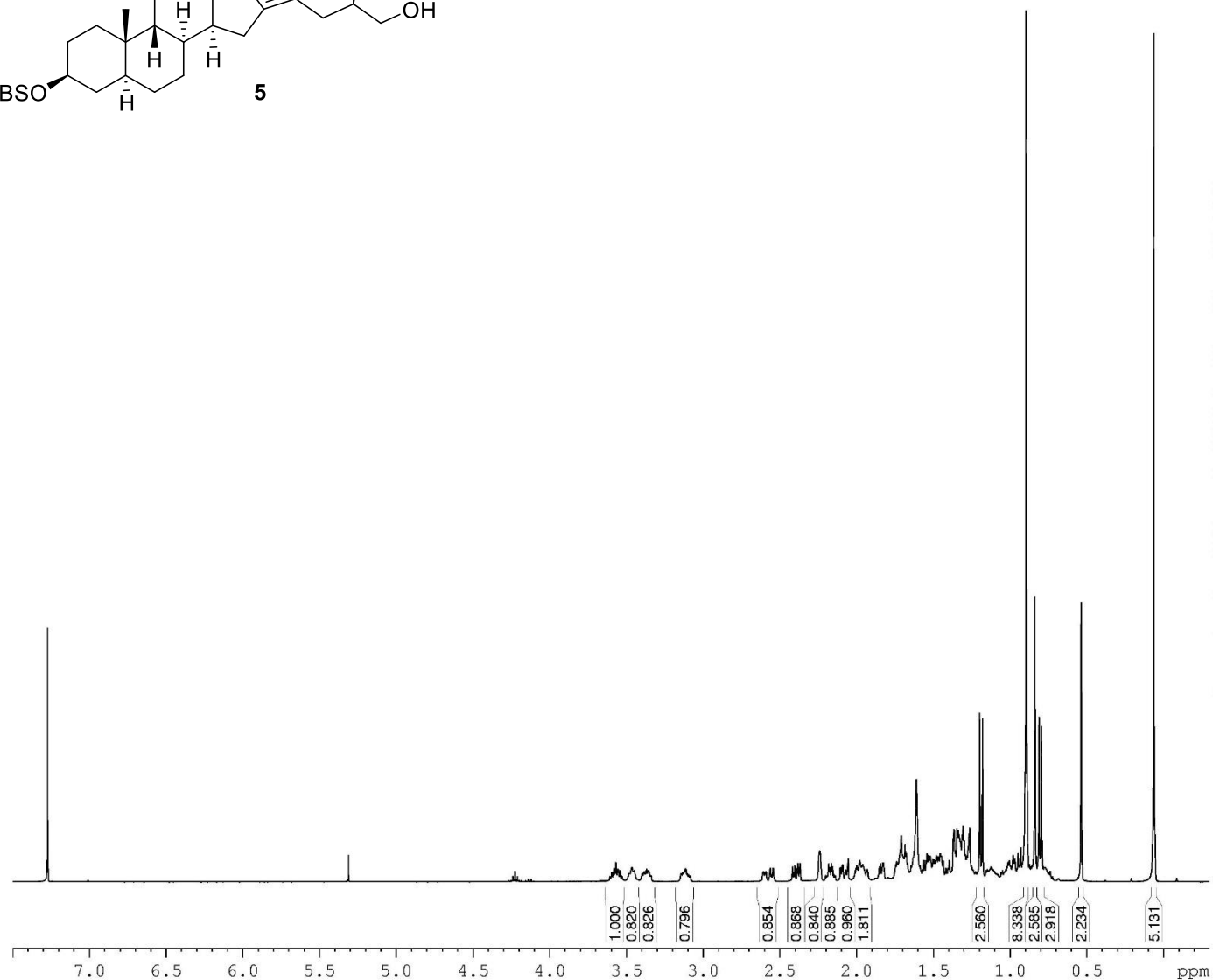
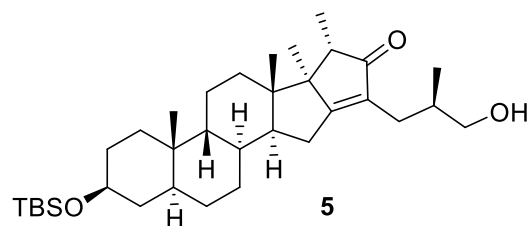
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===== CHANNEL f2 =====  
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 NUC2 1H  
 PCPD2 100.00 us  
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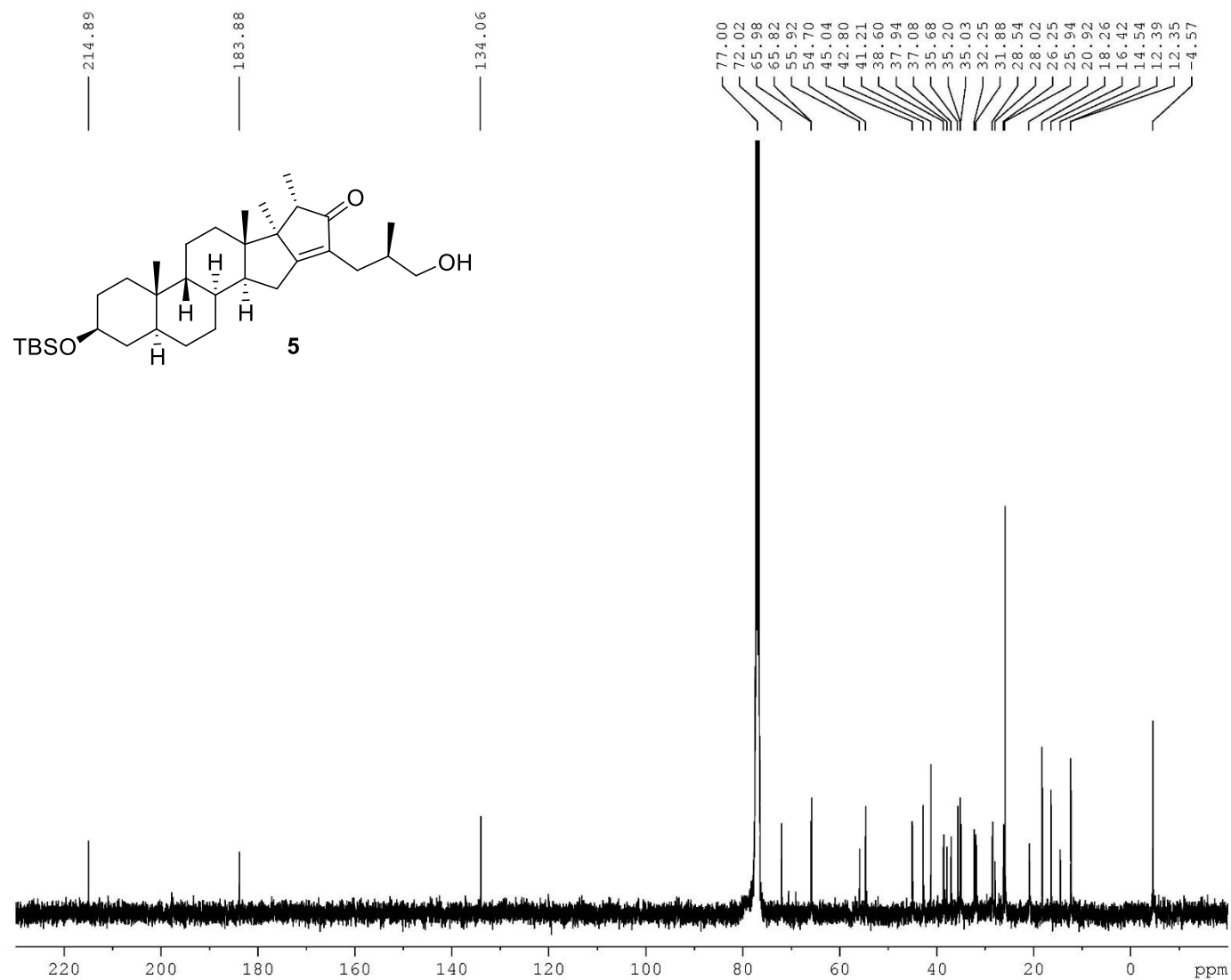


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 SOLVENT CDCl3  
 NS 128  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 se  
 RG 322  
 DW 60.800 us  
 DE 8.00 us  
 TE 999.9 K  
 D1 1.00000000 se  
 TDO 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 20.00 us  
 PL1 -3.00 dB  
 SFO1 400.1524711 MH

F2 - Processing parameters  
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 SF 400.1500000 MH  
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 GR 0.2  
 PC 1.00



Current Data Parameters  
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 EXPNO 2  
 PROCNO 1

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 DS 4  
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 FIDRES 0.489064 Hz  
 AQ 1.0224116 se  
 RG 144  
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 DE 6.00 us  
 TE 999.9 K  
 D1 2.0000000 se  
 d11 0.0300000 se  
 DELTA 1.89999998 se  
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===== CHANNEL f1 =====  
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 P1 27.50 us  
 PL1 -1.00 dB  
 SFO1 100.6298721 MH

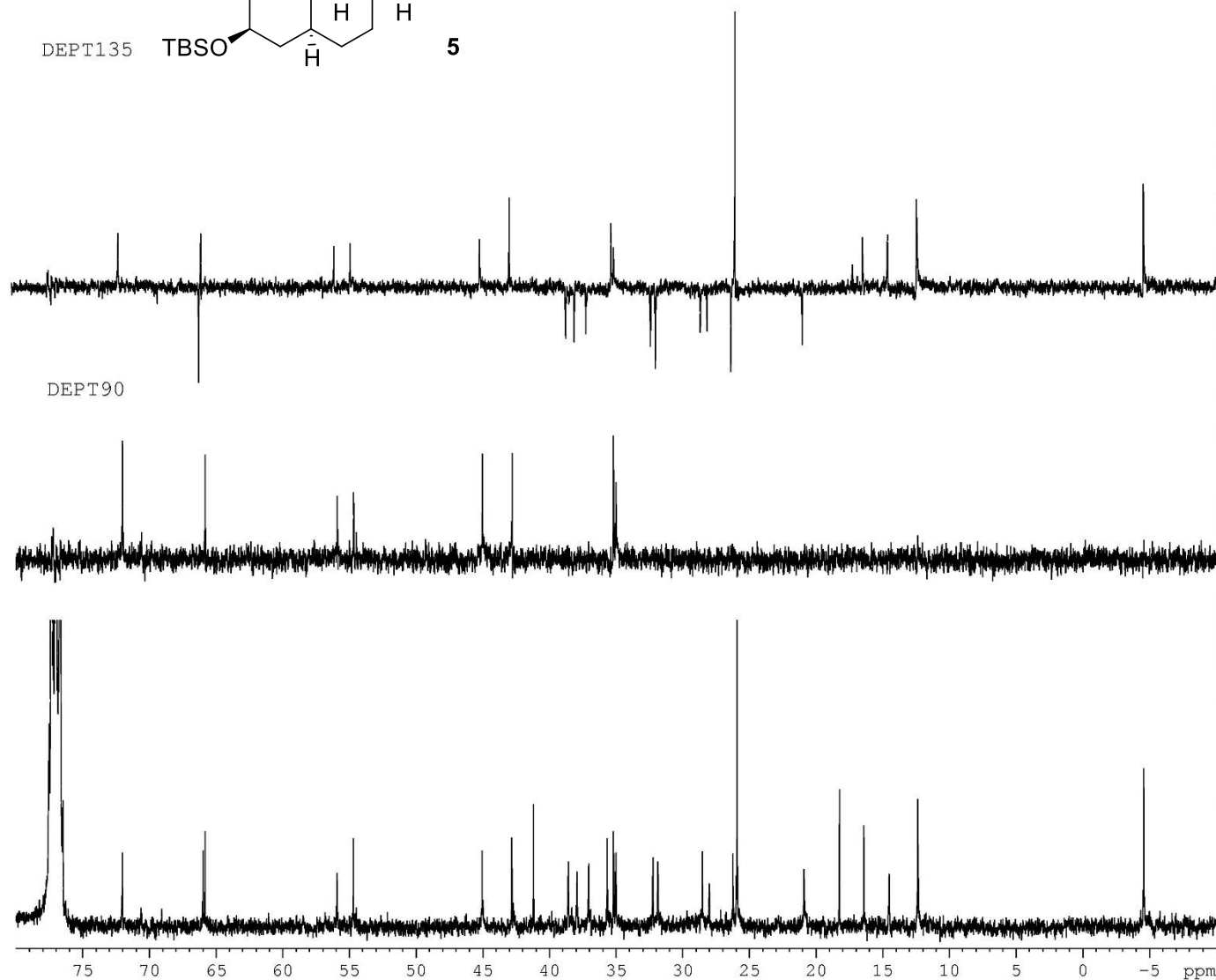
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 SFO2 400.1516006 MH

F2 - Processing parameters  
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DEPT135

TBSO

5



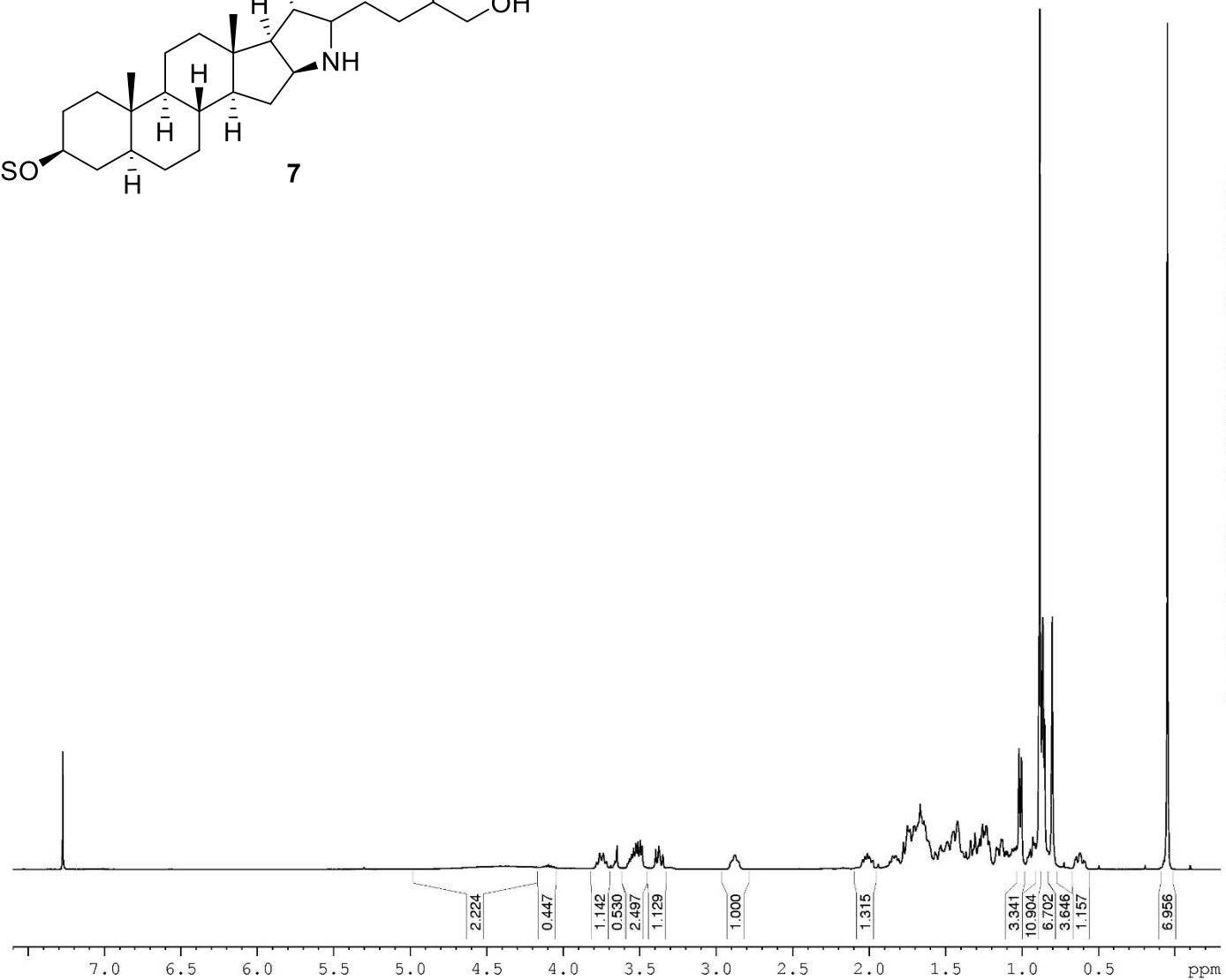
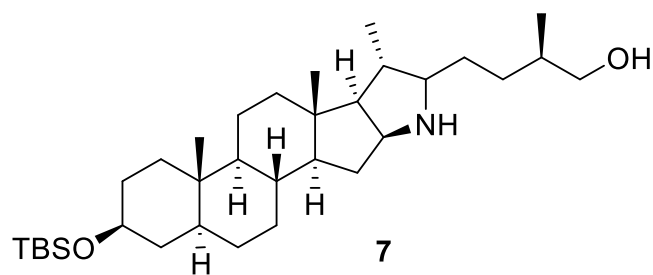
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 FIDRES 0.552855 Hz  
 AQ 0.9044468 se  
 RG 2050  
 DW 13.800 us  
 DE 6.00 us  
 TE 999.9 K  
 CNST2 145.0000000  
 D1 2.0000000 se  
 d2 0.00344828 se  
 d12 0.00002000 se  
 DELTA 0.00003501 se  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 27.50 us  
 p2 55.00 us  
 PL1 -1.00 dB  
 SFO1 100.6213196 MH

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 P3 25.00 us  
 p4 50.00 us  
 PCPD2 100.00 us  
 PL2 -3.00 dB  
 PL12 13.65 dB  
 SFO2 400.1516006 MH

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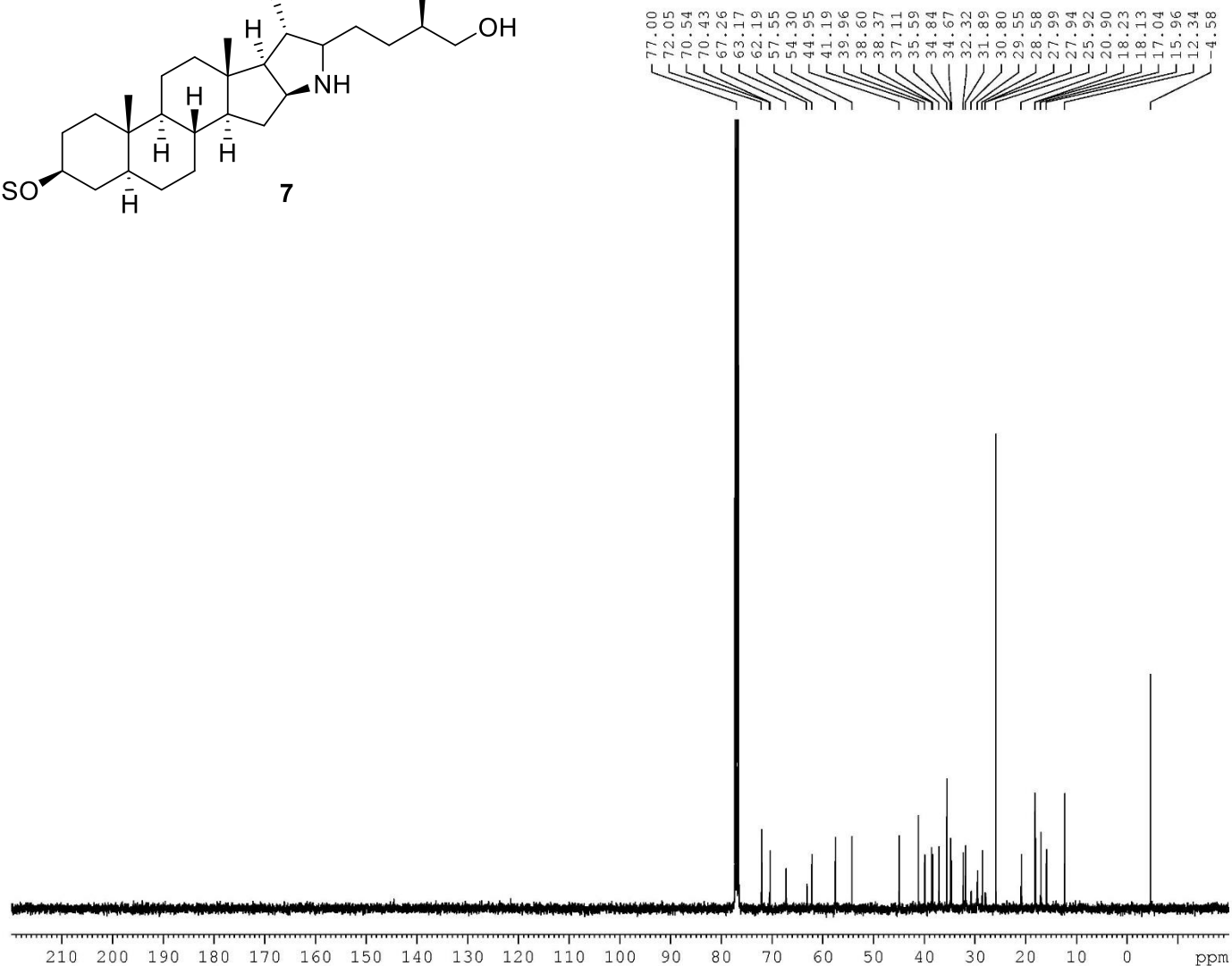
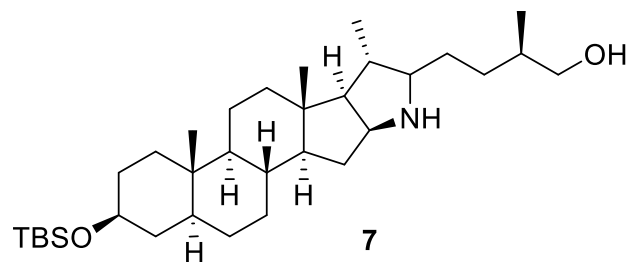


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 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 192  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 se  
 RG 144  
 DW 60.800 us  
 DE 8.00 us  
 TE 999.9 K  
 D1 1.00000000 se  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 11.15 us  
 PL1 -3.00 dB  
 SFO1 400.1524711 MH

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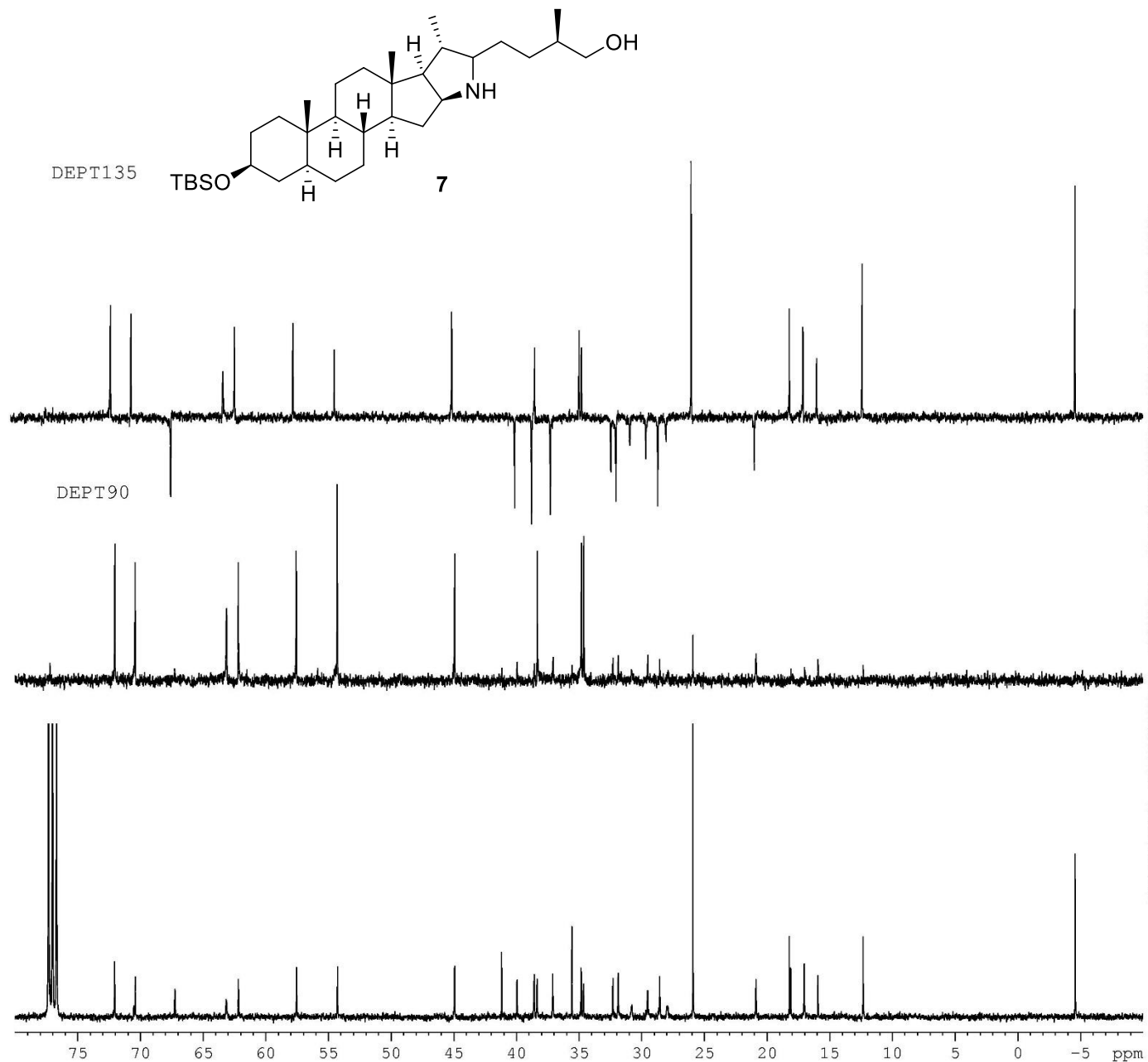
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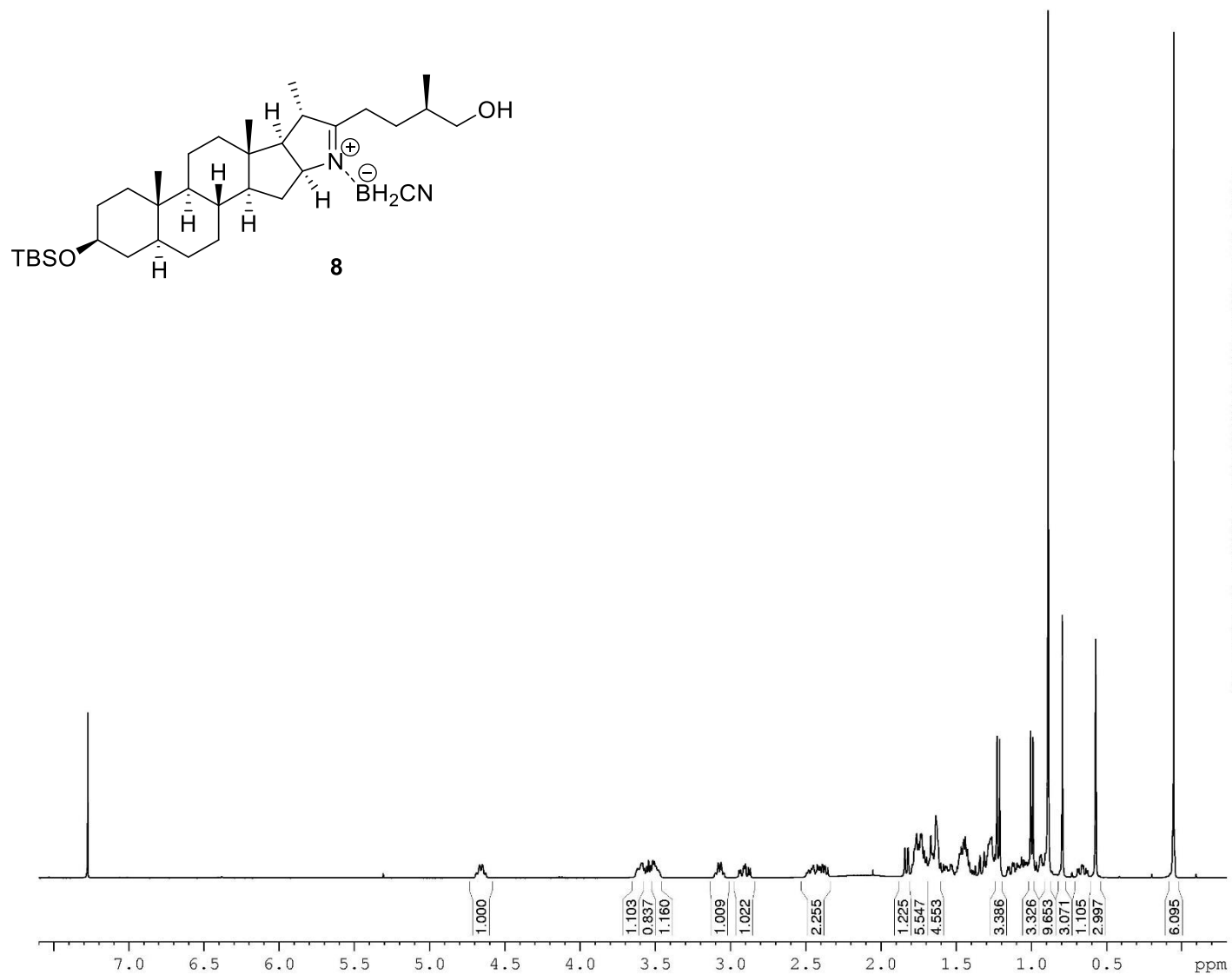
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 NS 1440  
 DS 4  
 SWH 32051.281 Hz  
 FIDRES 0.489064 Hz  
 AQ 1.0224116 se  
 RG 2050  
 DW 15.600 us  
 DE 6.00 us  
 TE 999.9 K  
 DI 2.0000000 se  
 d11 0.0300000 se  
 DELTA 1.89999998 se  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 27.50 us  
 PL1 -1.00 dB  
 SFO1 100.6298721 MH

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

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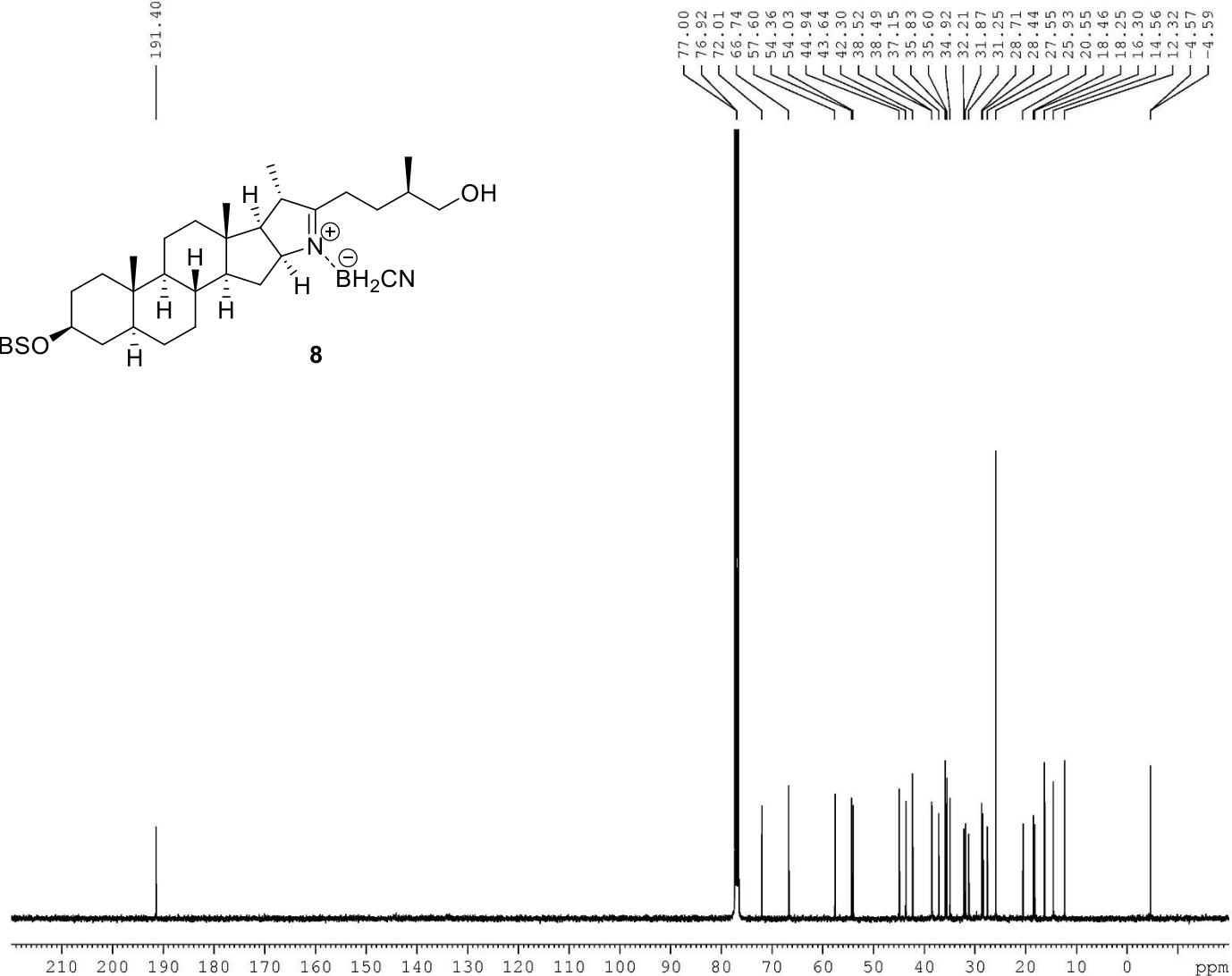
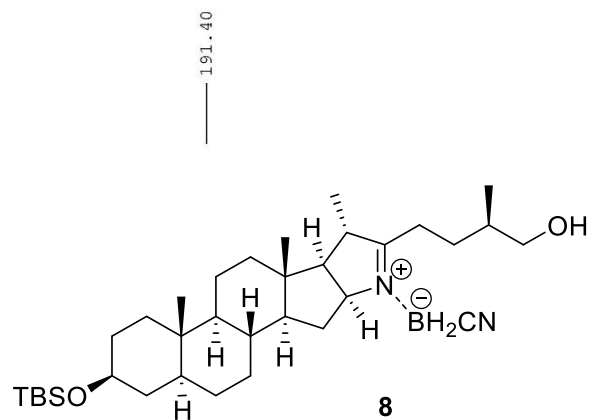


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 SOLVENT CDCl3  
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 FIDRES 0.125483 Hz  
 AQ 3.9846387 se  
 RG 144  
 DW 60.800 us  
 DE 8.00 us  
 TE 999.9 K  
 D1 1.00000000 se  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 20.00 us  
 PL1 -3.00 dB  
 SFO1 400.1524711 MH

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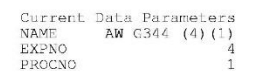
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PULPROG zgpg30  
TD 65536  
SOLVENT CDCL3  
NS 12800  
DS 4  
SWH 32051.281 Hz  
FIDRES 0.489064 Hz  
AQ 1.0224116 se  
RG 71.8  
DW 15.600 us  
DE 6.00 us  
TE 999.9 K  
D1 2.0000000 se  
d11 0.0300000 se  
DELTA 1.89999998 se  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 27.50 us  
PL1 -1.00 dB  
SFO1 100.6298721 MH

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

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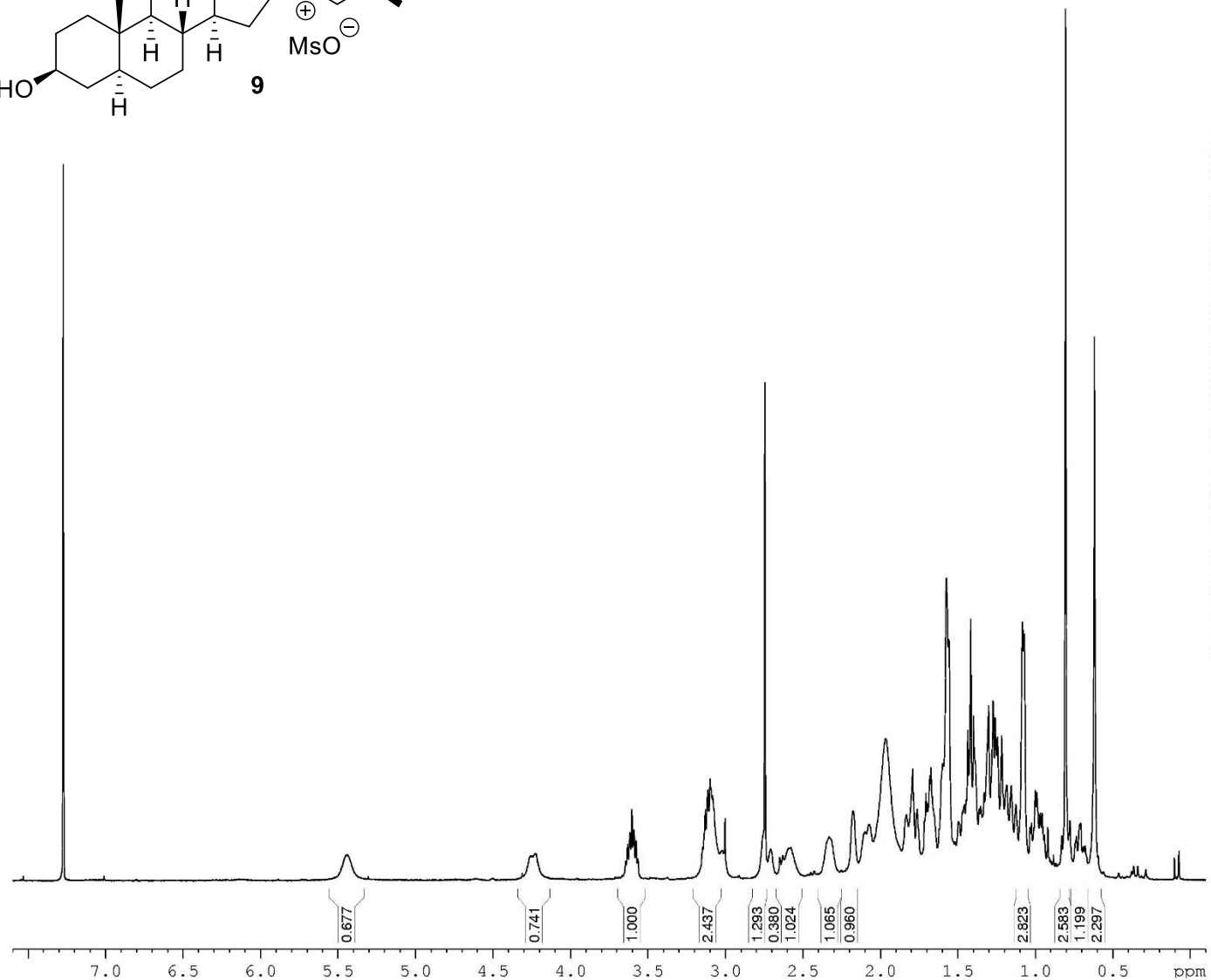
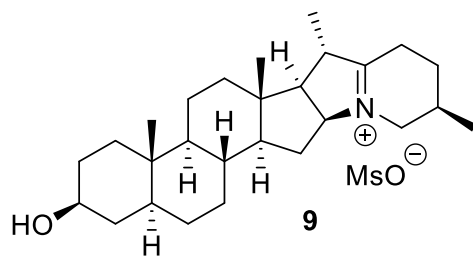
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SOLVENT        CDCl3
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DS             4
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FIDRES        0.552855
AQ            0.9044468
RG            2050
DW            13.800
DE            6.00
TE            999.9
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D1            2.00000000
d2            0.00344828
d12           0.00020000
DELTA         0.00003501
TD0           1

```

```
===== CHANNEL f1 =====
NUC1                13C
P1                  27.50 us
p2                  55.00 us
PL1                 -1.00 dB
SFO1               100.6228289 MH
```

```
===== CHANNEL f2 =====
GPDPRG2          waltz16
NUC2              1H
P3                18.00 us
p4                36.00 us
PCPD2            100.00 us
PL2              -3.00 dB
PL12              13.65 dB
SFO2             400.151606 MHz
```

```
F2 - Processing parameters
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WDW                      EM
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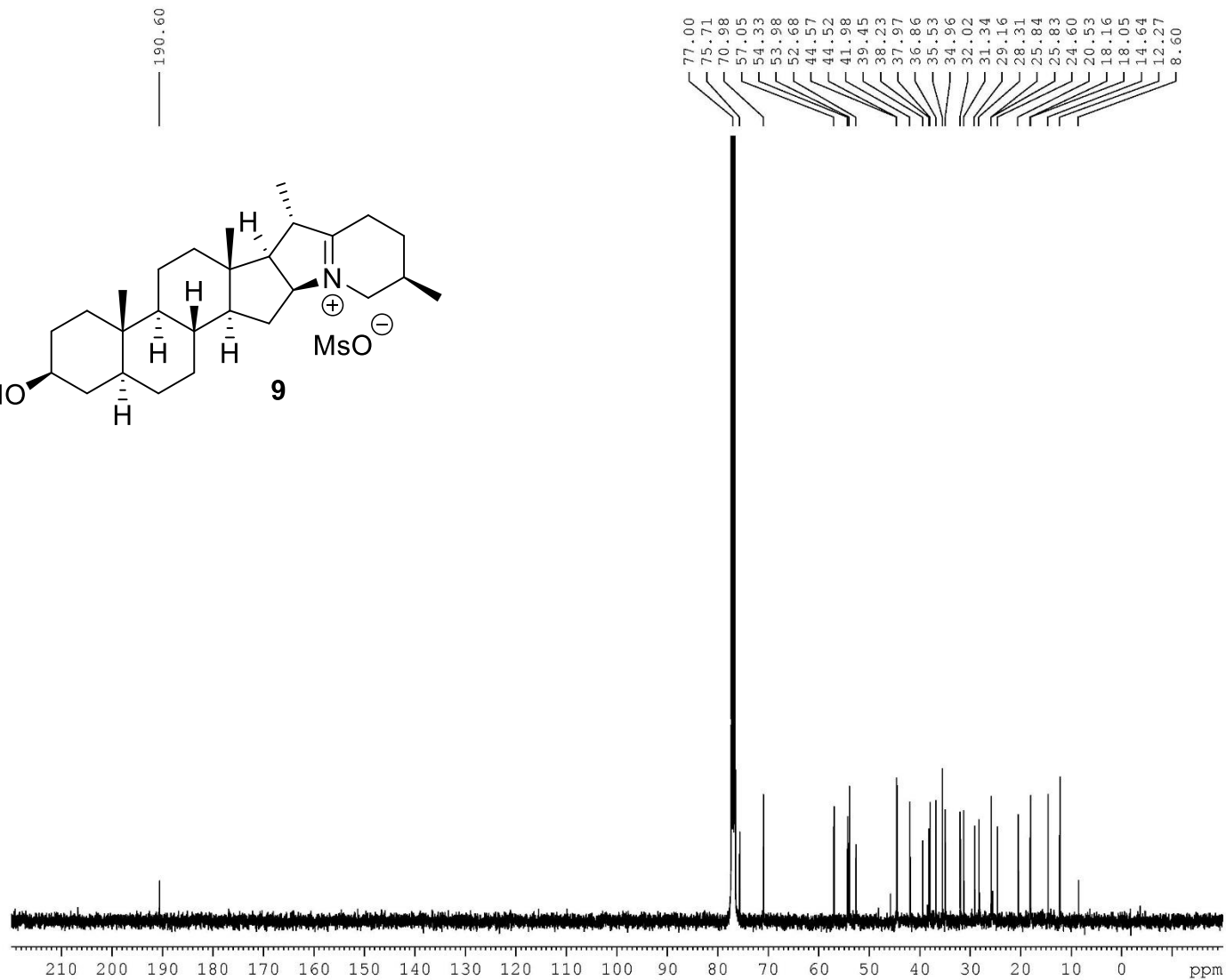
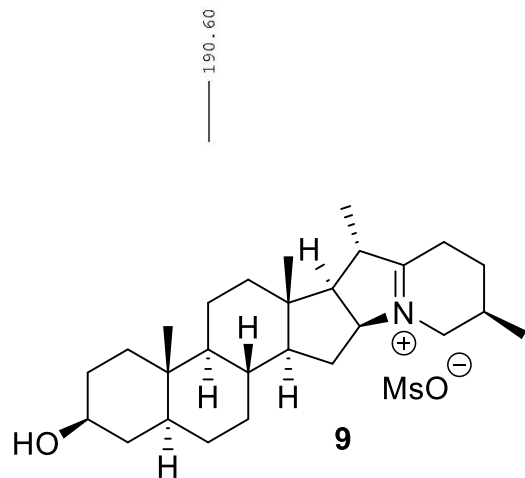


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 SOLVENT CDCl3  
 NS 192  
 DS 0  
 SWE 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 se  
 RG 287  
 DW 60.800 us  
 DE 8.00 us  
 TE 298.6 K  
 D1 1.00000000 se  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 25.00 us  
 PL1 -3.00 dB  
 SFO1 400.1524711 MH

F2 - Processing parameters  
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 SF 400.1500000 MH  
 WDW GM  
 SSB 0  
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 PC 1.00



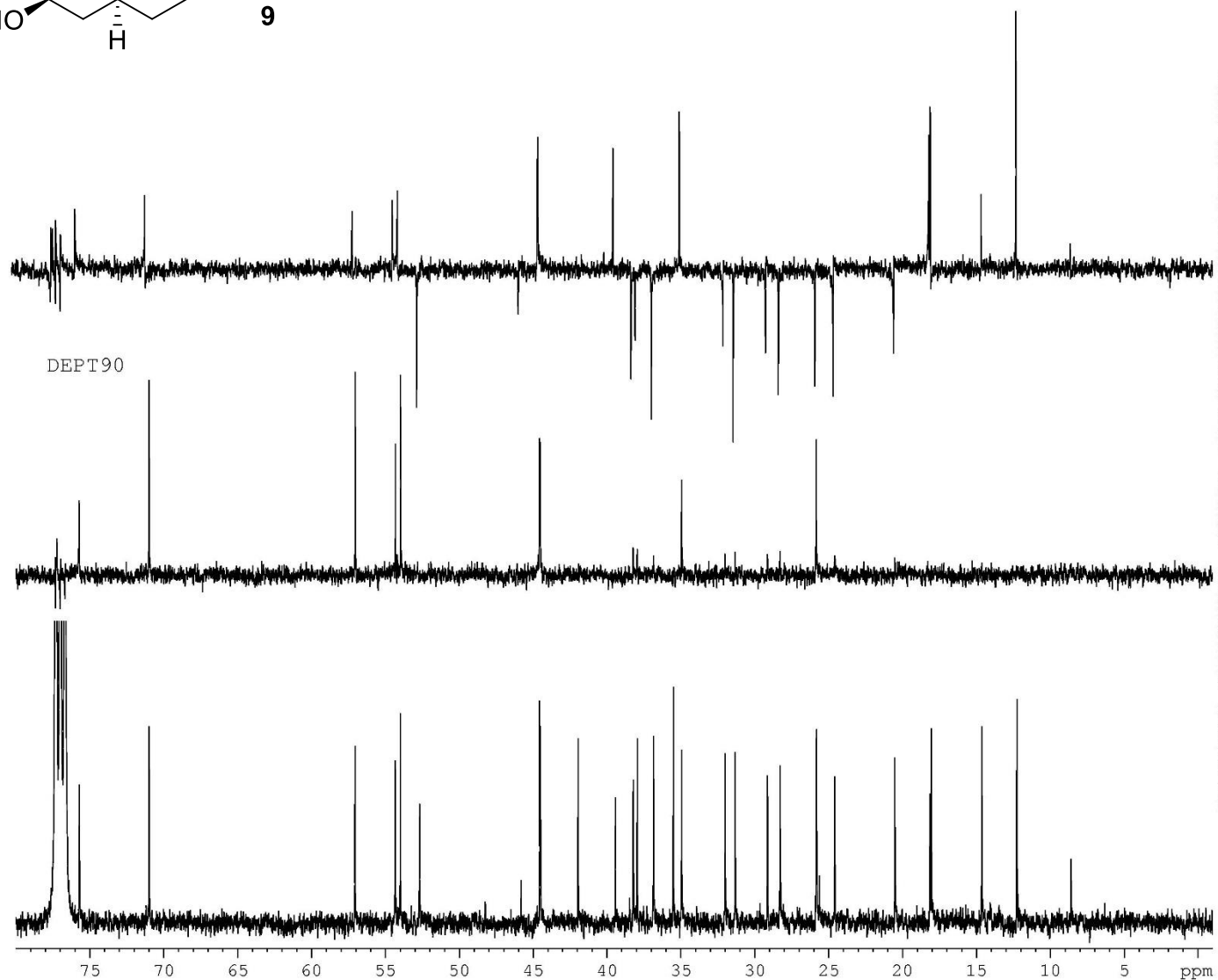
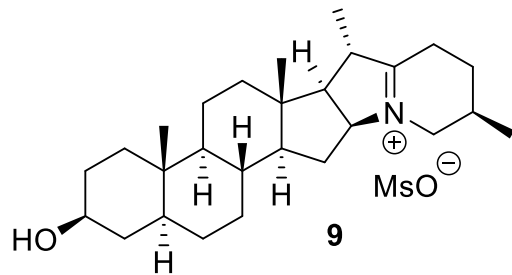
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 TD 65536  
 SOLVENT CDCL3  
 NS 16384  
 DS 4  
 SWH 32051.281 Hz  
 FIDRES 0.489064 Hz  
 AQ 1.0224116 se  
 RG 114  
 DW 15.600 us  
 DE 6.00 us  
 TE 299.0 K  
 D1 2.0000000 se  
 d11 0.0300000 se  
 DELTA 1.89999998 se  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 27.50 us  
 PL1 -1.00 dB  
 SFO1 100.6298721 MH

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

F2 - Processing parameters  
 SI 32768  
 SF 100.6177990 MH  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 0.20



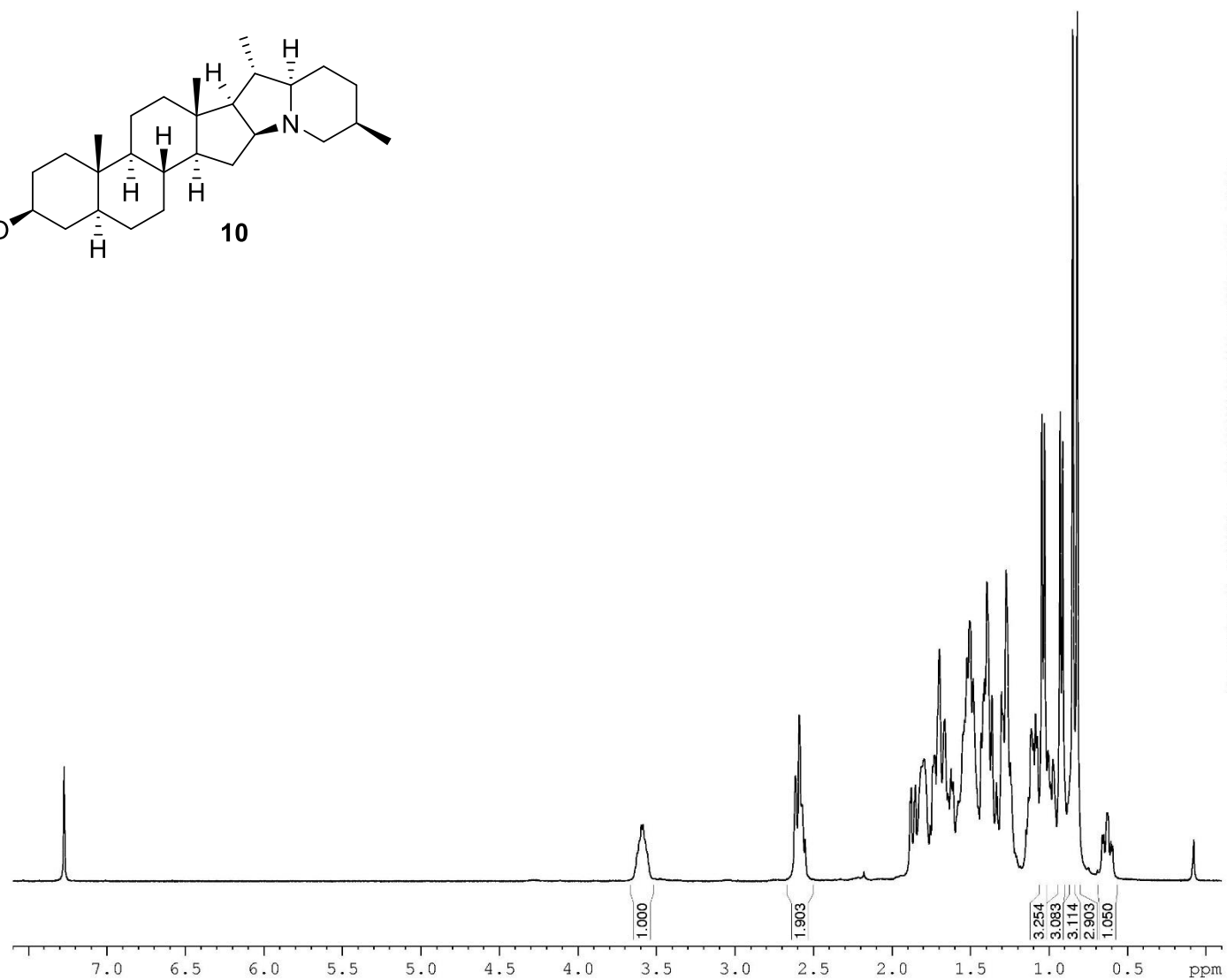
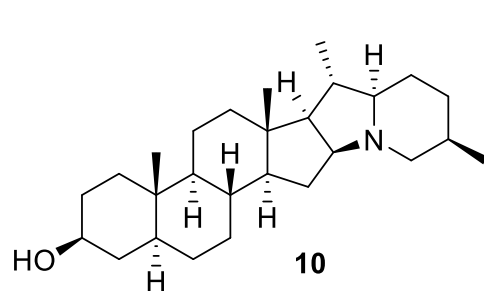
Current Data Parameters  
NAME AW G354c (5) susz  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameter  
Date\_ 20210417  
Time 13.08  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG dept135  
TD 65536  
SOLVENT CDCl3  
NS 4096  
DS 4  
SWH 36231.883 Hz  
FIDRES 0.552855 Hz  
AQ 0.9044468 se  
RG 2050  
DW 13.800 us  
DE 6.00 us  
TE 299.1 K  
CNST2 145.000000  
D1 2.0000000 se  
d2 0.00344828 se  
d12 0.00002000 se  
DELTA 0.00005501 se  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 27.50 us  
p2 55.00 us  
PL1 -1.00 dB  
SFO1 100.6218227 MH

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
P3 18.00 us  
p4 36.00 us  
PCPD2 100.00 us  
PL2 -3.00 dB  
PL12 13.65 dB  
SFO2 400.1516006 MH

F2 - Processing parameters  
SI 32768  
SF 100.6177997 MH  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

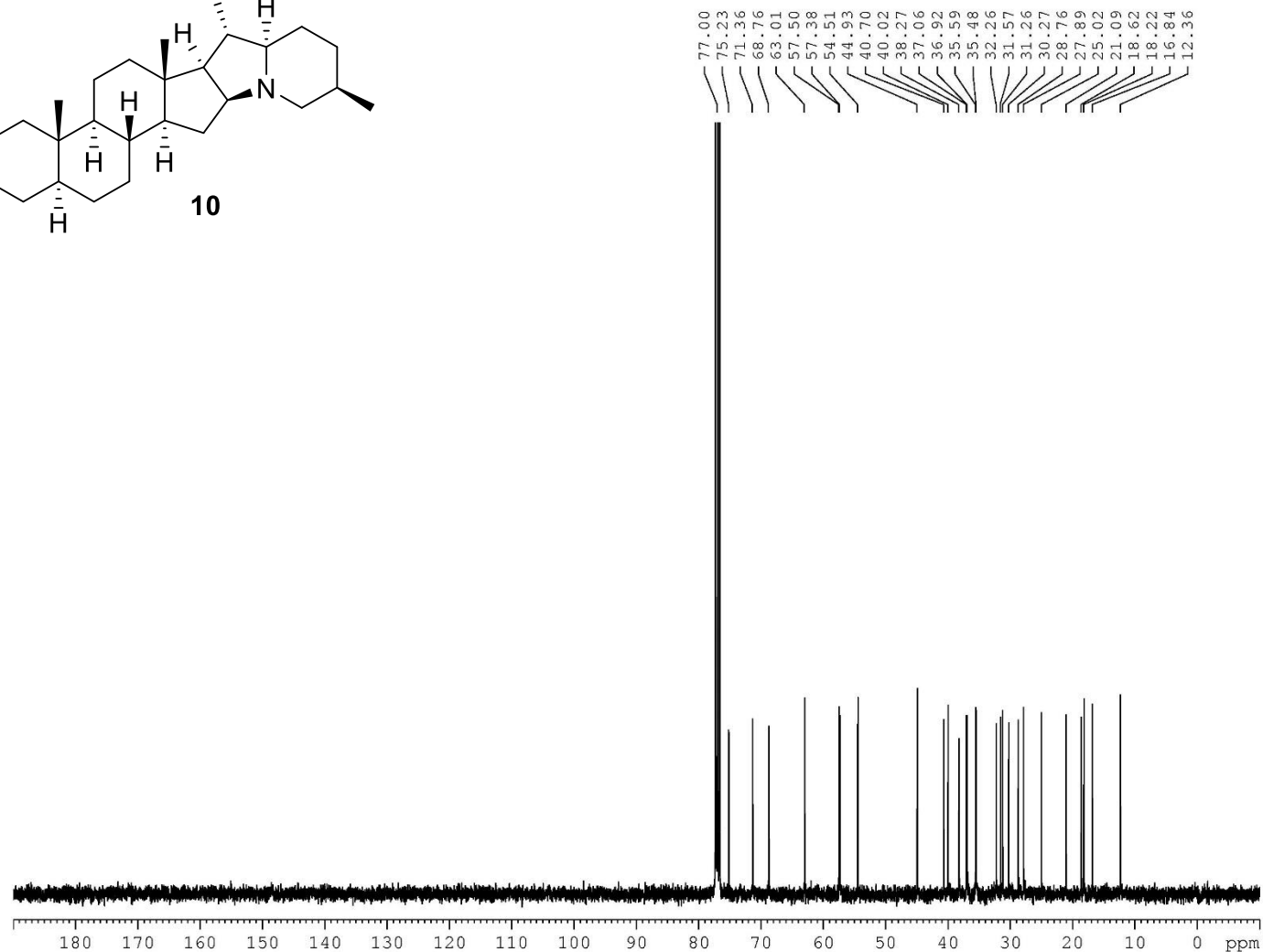
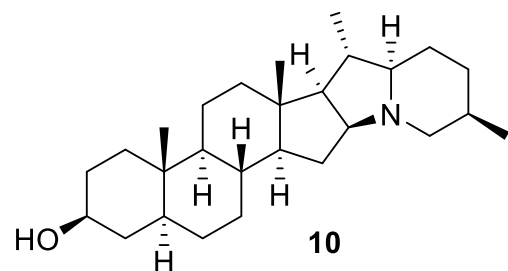


Current Data Parameters  
NAME MSZ R13-P  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameter  
Date\_ 20210219  
Time 11.59  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 128  
DS 0  
SWE 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 se  
RG 256  
DW 60.800 us  
DE 8.00 us  
TE 298.9 K  
D1 1.00000000 se  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 11.15 us  
PL1 -3.00 dB  
SFO1 400.1524711 MH

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



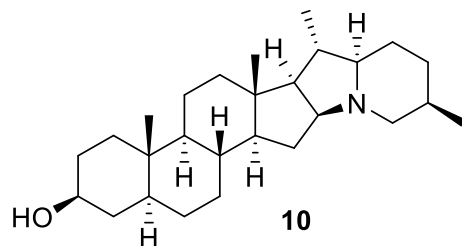
Current Data Parameters  
NAME MSX R13-P  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameter  
Date\_ 20210219  
Time 12.07  
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 se  
RG 2050  
DW 15.600 us  
DE 6.00 us  
TE 299.2 K  
D1 2.0000000 se  
d11 0.0300000 se  
DELTA 1.89999998 se  
TD0 1

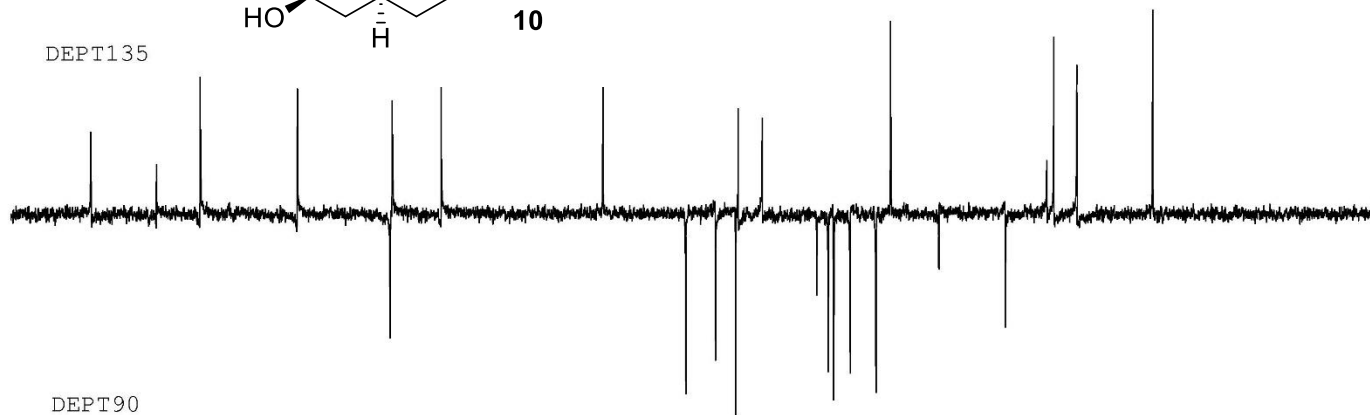
===== CHANNEL f1 =====  
NUC1 13C  
P1 27.50 us  
PL1 -1.00 dB  
SFO1 100.6298721 MH

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

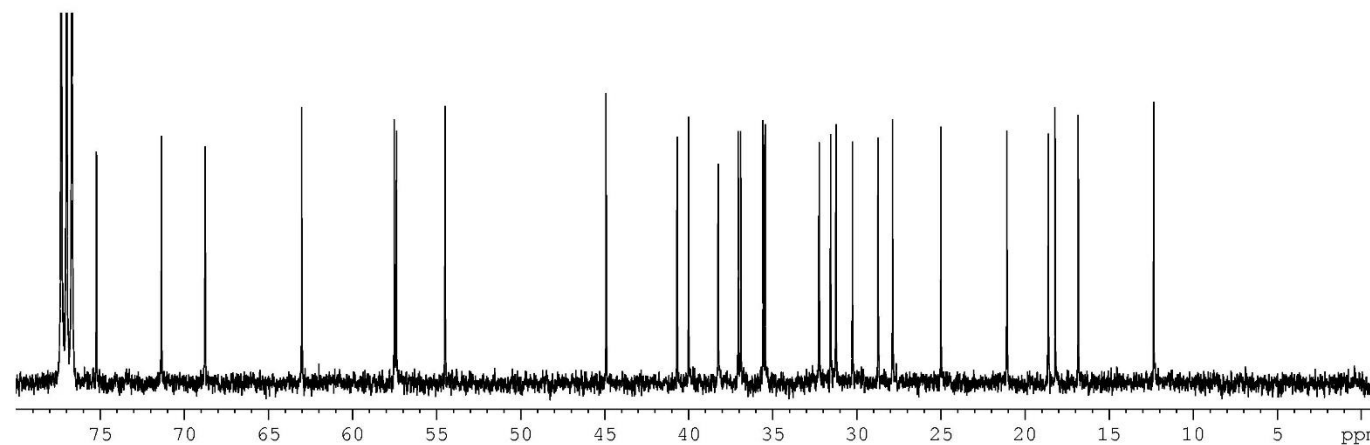
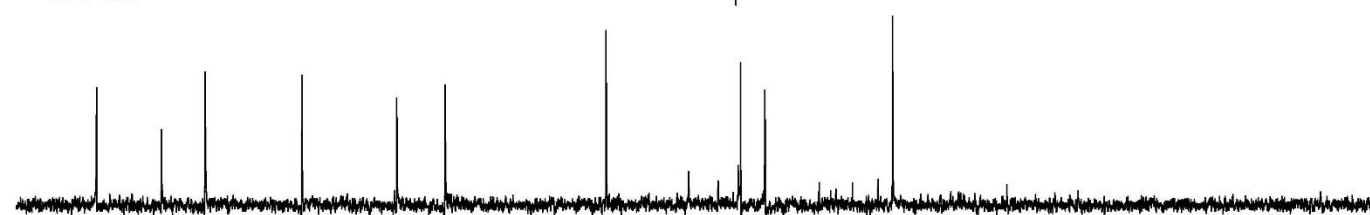
F2 - Processing parameters  
SI 32768  
SF 100.6177973 MH  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 0.20



DEPT135



DEPT90



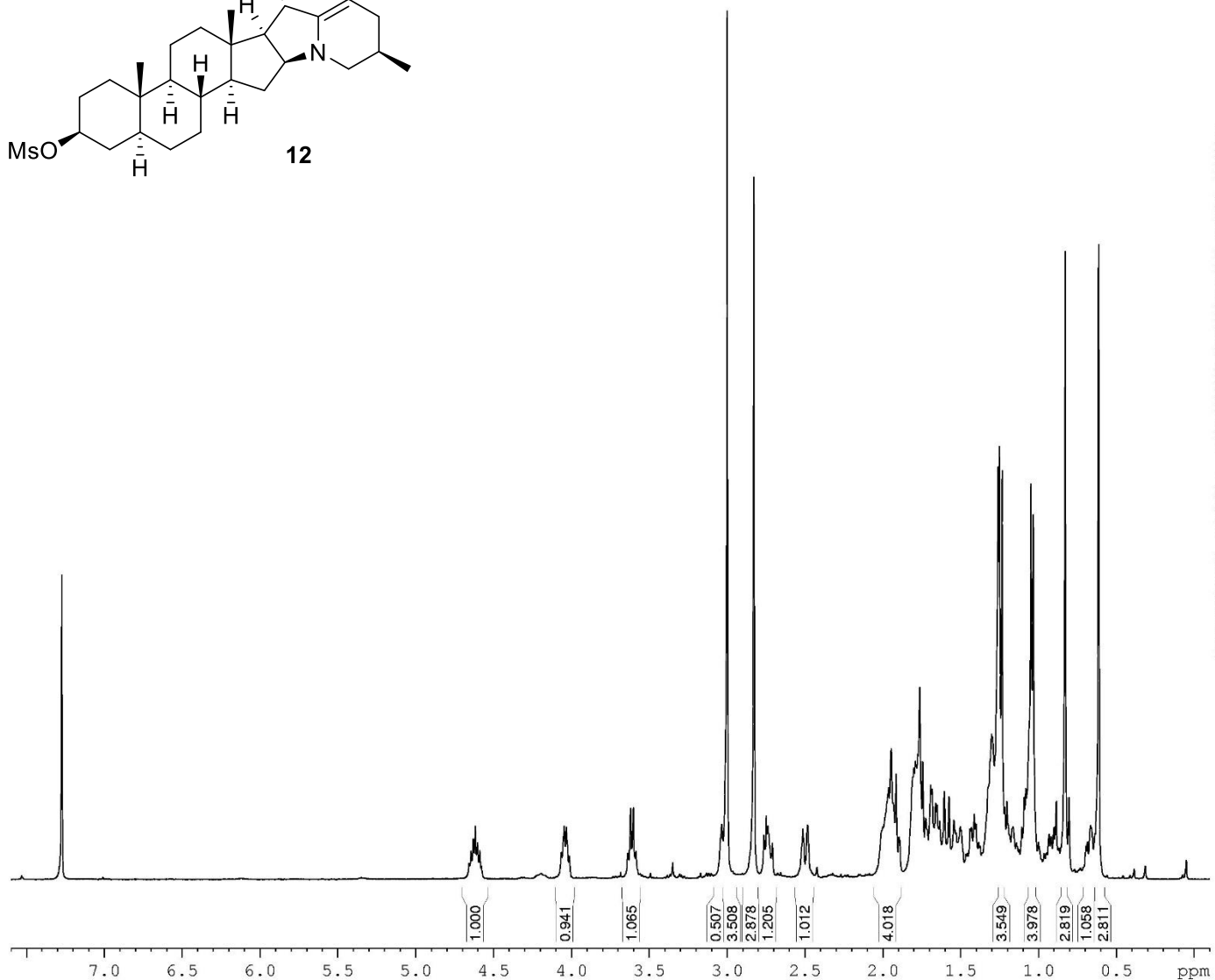
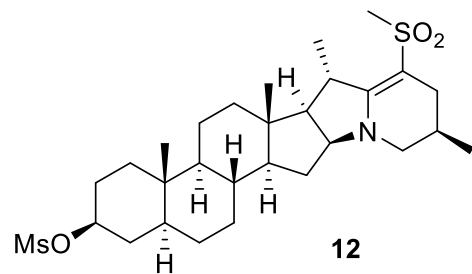
Current Data Parameters  
NAME MSX R13-P  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameter  
Date\_ 20210219  
Time 14.41  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG dept135  
TD 65536  
SOLVENT CDCL3  
NS 480  
DS 4  
SWH 36231.883 Hz  
FIDRES 0.552855 Hz  
AQ 0.9044468 se  
RG 2050  
DW 13.800 us  
DE 6.00 us  
TE 299.9 K  
CNST2 145.000000  
D1 2.0000000 se  
d2 0.00344828 se  
d12 0.00002000 se  
DELTA 0.00005501 se  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 27.50 us  
p2 55.00 us  
PL1 -1.00 dB  
SFO1 100.6218227 MH

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
P3 18.00 us  
p4 36.00 us  
PCPD2 100.00 us  
PL2 -3.00 dB  
PL12 13.65 dB  
SFO2 400.1516006 MH

F2 - Processing parameters  
SI 32768  
SF 100.6177973 MH  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 0.20



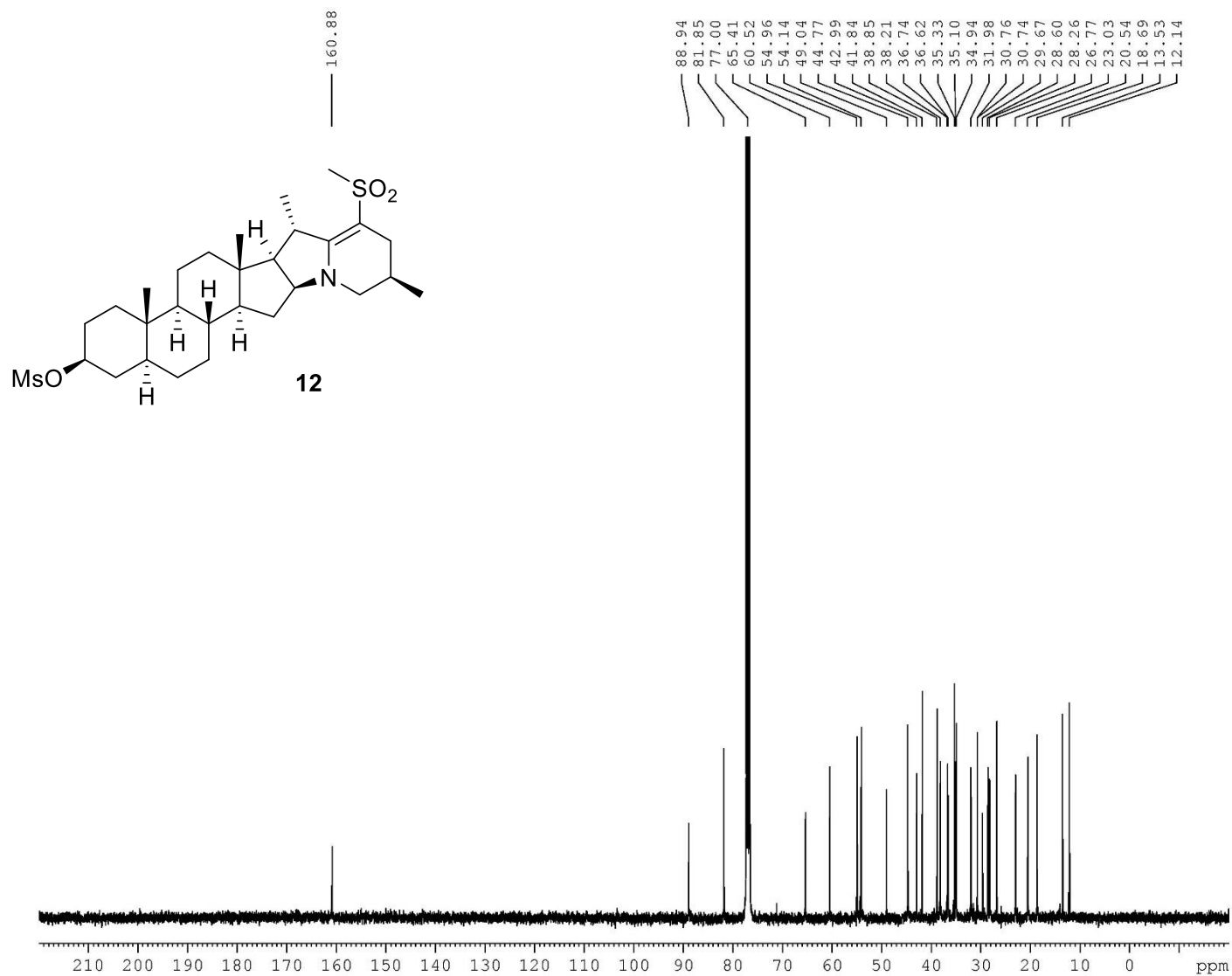
Current Data Parameters  
 NAME AW G354b 11 (1)  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameter  
 Date\_ 20210401  
 Time 13.28  
 INSTRUM spect  
 PROBED 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 128  
 DS 0  
 SWE 8223.685 Hz  
 FIDRES 0.125483 Hz  
 AQ 3.9846387 se  
 RG 144  
 DW 60.800 us  
 DE 8.00 us  
 TE 299.2 K  
 D1 1.00000000 se  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 25.00 us  
 PL1 -3.00 dB  
 SFO1 400.1524711 MH

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





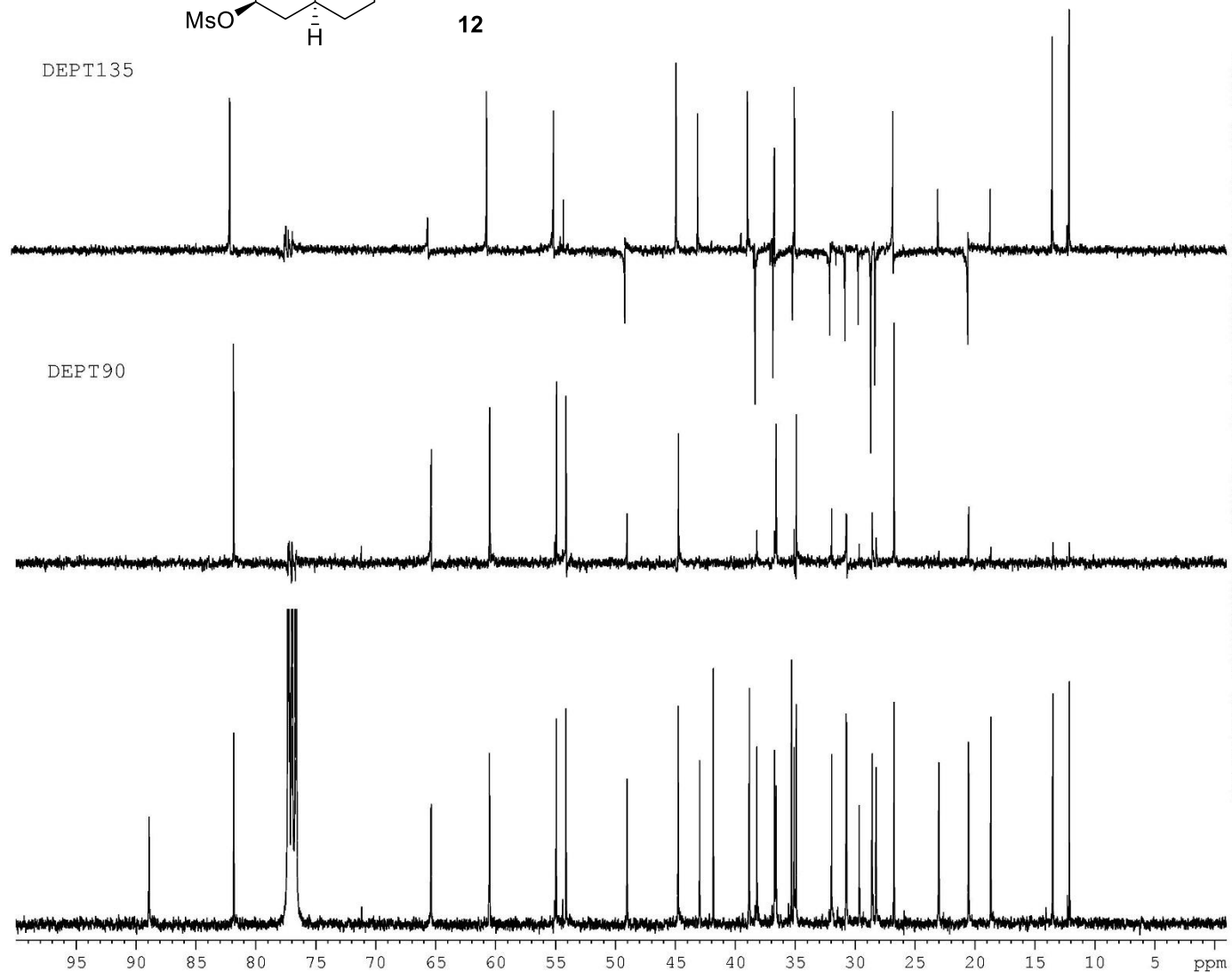
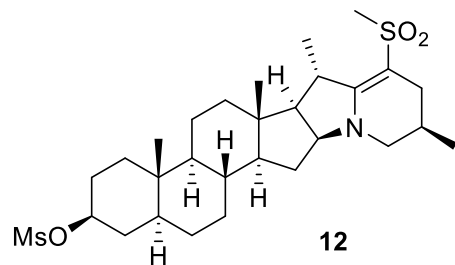
Current Data Parameters  
 NAME AW G354b II (1)  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameter  
 Date\_ 20210402  
 Time 6.49  
 INSTRUM spect  
 PROBED 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCL3  
 NS 16384  
 DS 4  
 SWH 32051.281 Hz  
 FIDRES 0.489064 Hz  
 AQ 1.0224116 se  
 RG 101  
 DW 15.600 us  
 DE 6.00 us  
 TE 299.1 K  
 D1 2.00000000 se  
 d11 0.03000000 se  
 DELTA 1.89999998 se  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 27.50 us  
 PL1 -1.00 dB  
 SFO1 100.6298721 MH

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

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



Current Data Parameters  
 NAME AW G354b T1 (1)  
 EXPNO 4  
 PROCNO 1

F2 - Acquisition Parameter  
 Date\_ 20210402  
 Time 10.58  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG dept135  
 TD 65536  
 SOLVENT CDCL3  
 NS 6400  
 DS 4  
 SWH 36231.883 Hz  
 FIDRES 0.552855 Hz  
 AQ 0.9044468 se  
 RG 2050  
 DW 13.800 us  
 DE 6.00 us  
 TE 298.8 K  
 CNST2 145.000000  
 D1 2.0000000 se  
 d2 0.00344828 se  
 d12 0.00002000 se  
 DELTA 0.00005501 se  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 27.50 us  
 p2 55.00 us  
 PL1 -1.00 dB  
 SFO1 100.6218227 MH

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 P3 18.00 us  
 p4 36.00 us  
 PCPD2 100.00 us  
 PL2 -3.00 dB  
 PL12 13.65 dB  
 SFO2 400.1516006 MH

F2 - Processing parameters  
 SI 32768  
 SF 100.6178003 MH  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 0.20