

*Supporting Information for*

**Reversal of Fluconazole Resistance by Sulfated Sterols from the Marine Sponge *Topsentia* sp.**

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*Department of Pharmacognosy, National Center for Natural Products Research, and Research Institute of Pharmaceutical Sciences, School of Pharmacy, University of Mississippi, University, Mississippi, 38677, USA*

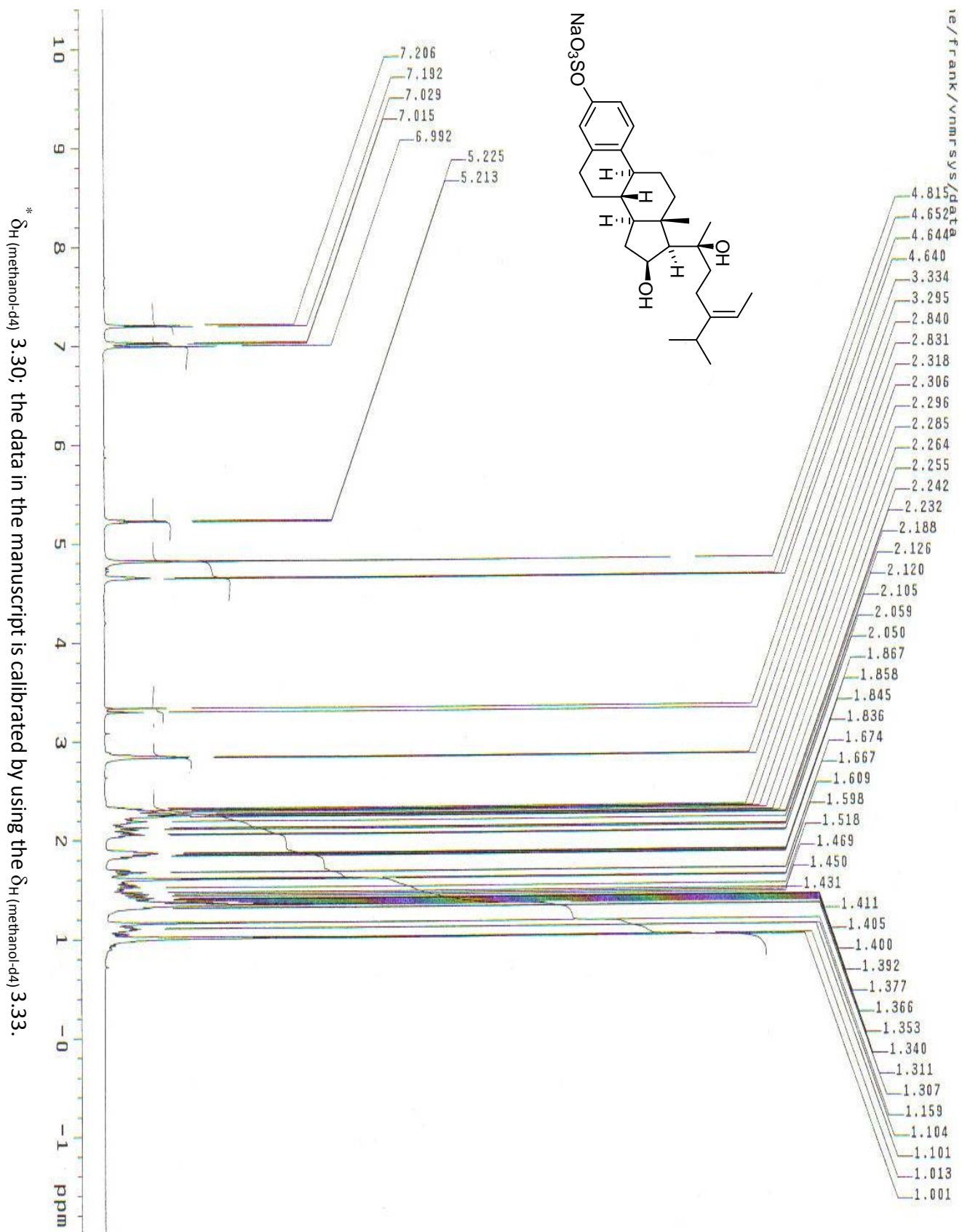
**Table of Contents:**

**S1-S12.** NMR spectra of compound **1**.

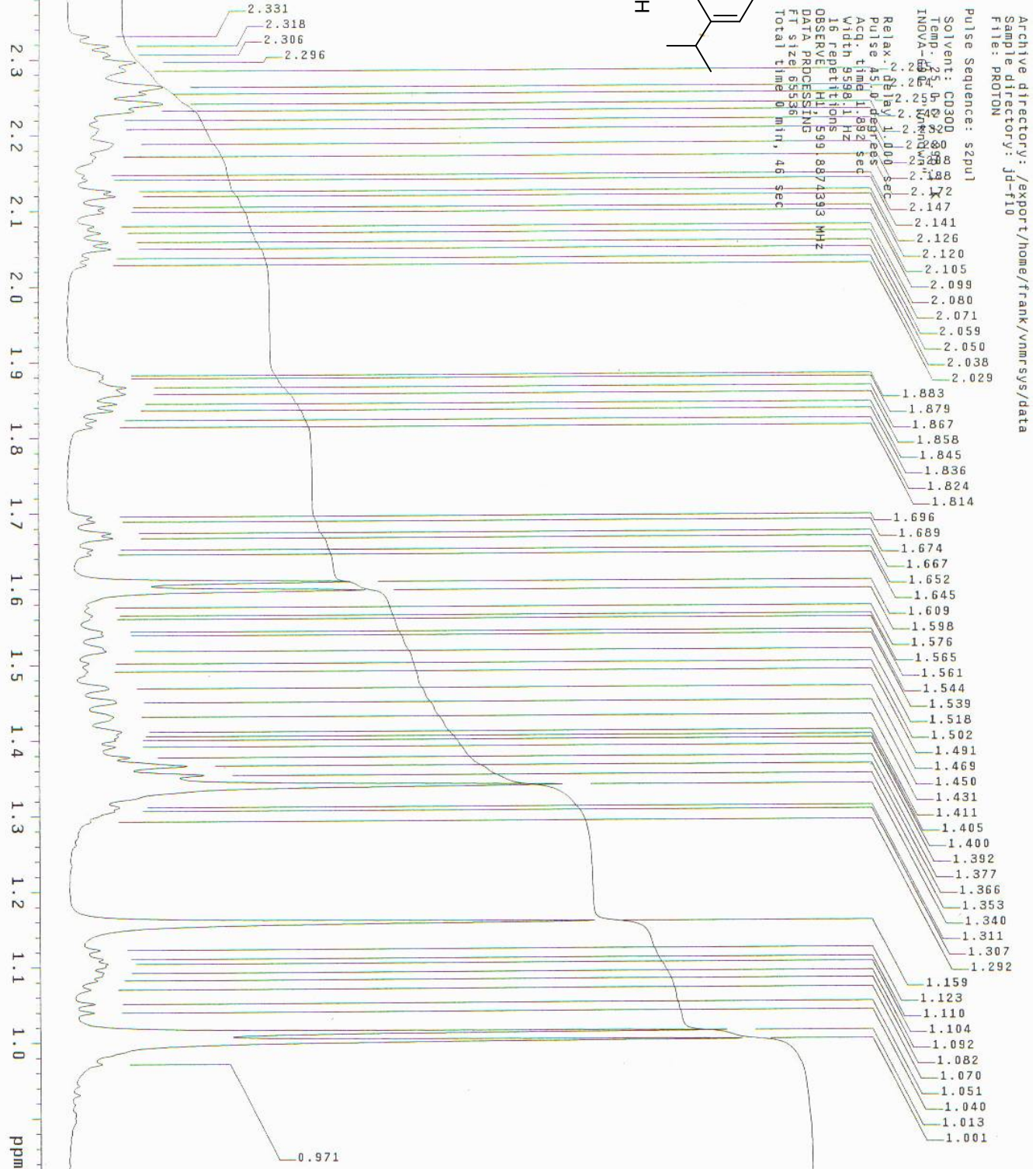
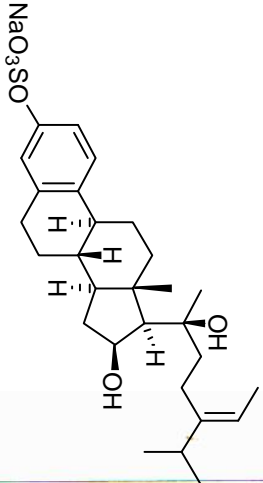
**S13-S23.** NMR spectra of compound **2**.

**S24-S27.** NMR spectra of compound **3**.

S1. 600 MHz <sup>1</sup>H NMR spectrum of compound 1 in methanol-d<sub>4</sub>\*



S2. 600 MHz <sup>1</sup>H NMR spectrum of compound 1 in methanol-d<sub>4</sub> (expansion)



S3. 150 MHz <sup>13</sup>C NMR spectrum of compound 1 in methanol-d<sub>4</sub>\*

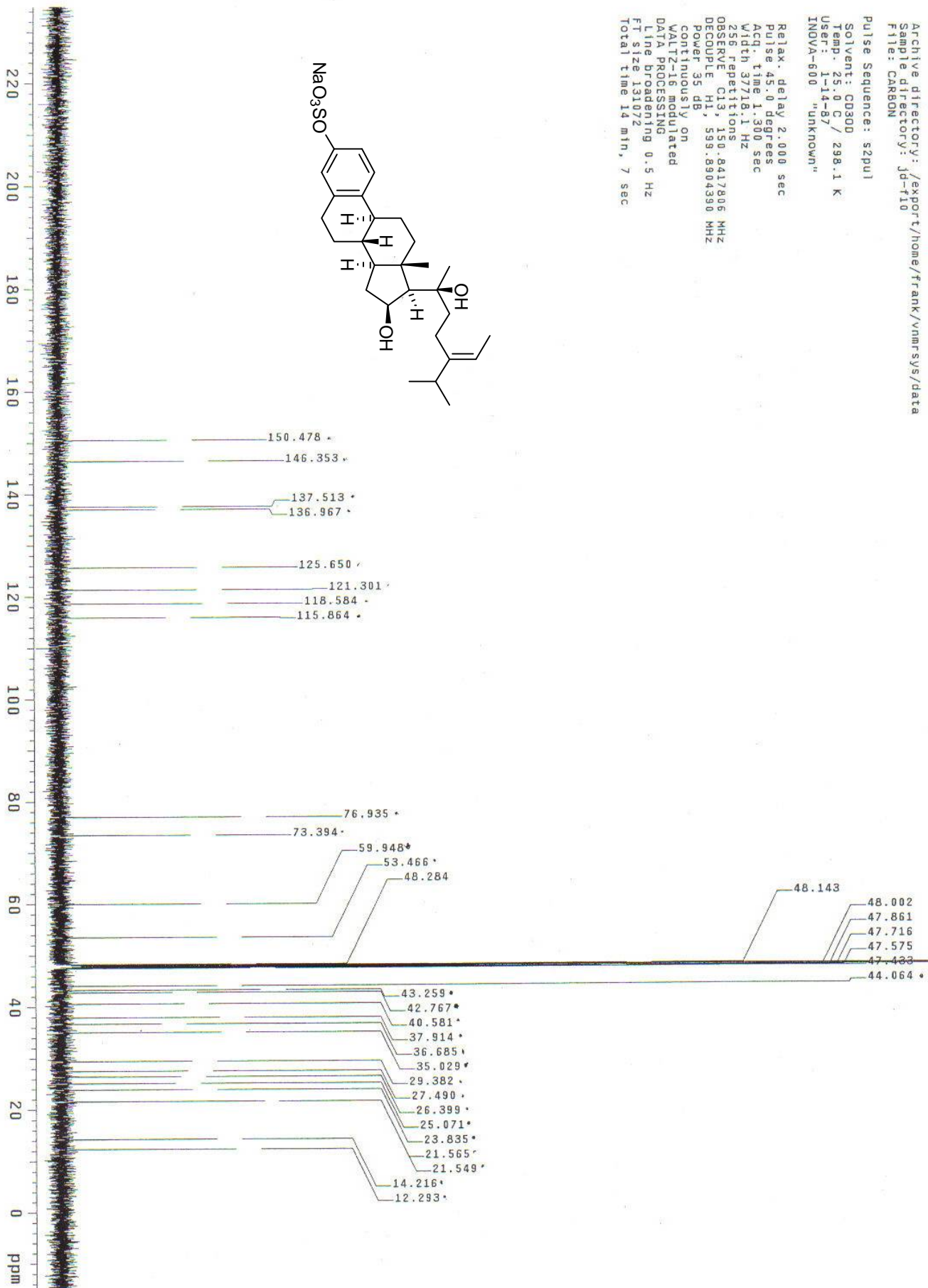
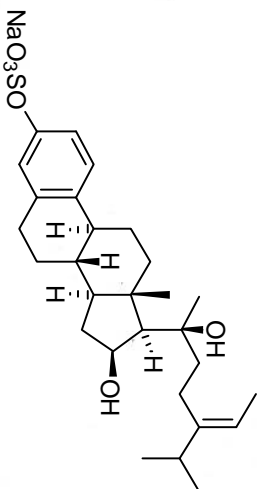
STANDARD CARBON PARAMETERS

Archive directory: /export/home/frank/vnmrSYS/data  
 Sample directory: jd-f10  
 File: CARBON

Pulse Sequence: s2pul

Solvent: CD3OD  
 Temp: 25.0 C / 298.1 K  
 User: 1-14-87  
 INOVA-800 "unknown"

Relax. delay 2.000 sec  
 Pulse 45.0 degrees  
 Acq. time 1.300 sec  
 Width 37718.1 Hz  
 256 Repetitions  
 OBSERVE C13, 150.8417906 MHz  
 DECOUPLE H1, 599.8904390 MHz  
 Power 35 dB  
 continuously on  
 WALTZ-16 modulated  
 DATA PROCESSING  
 Line broadening 0.5 Hz  
 FT size 131072  
 Total time 14 min, 7 sec



\* $\delta_C$  (methanol-d<sub>4</sub>) 47.9; the data in the manuscript is calibrated by using the  $\delta_C$  (methanol-d<sub>4</sub>) 49.0.

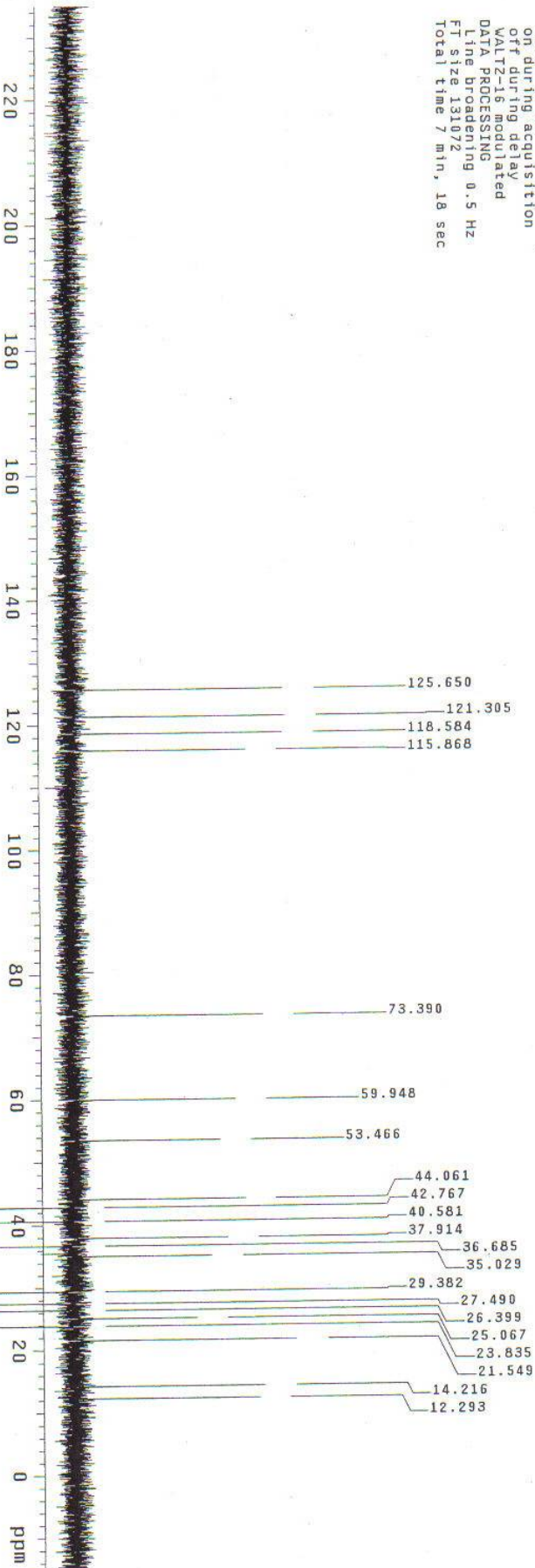
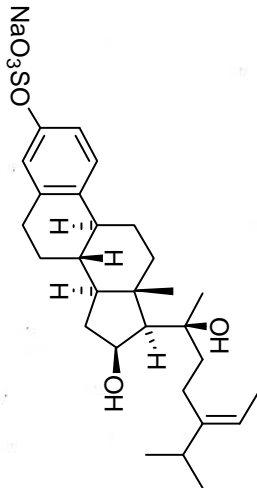
S4. 125 MHz DEPT-135 spectrum of compound 1 in methanol-d<sub>4</sub>

STANDARD CARBON PARAMETERS

Archive directory: /export/home/frank/vnmrsvs/data  
 Sample directory: jd-f10  
 File: CARBON

Pulse Sequence: DEPT  
 Solvent: CD3OD  
 Temp: 25.0 C / 298.1 K  
 User: 1-14-87  
 INOVA-600 "unknown"

Relax. delay 2.000 sec  
 Pulse 90.0 degrees  
 Acq. time 1.300 sec  
 Width 37718.1 Hz  
 128 repetitions  
 OBSERVE C13, 150.8417806 MHz  
 DECOUPLE H1, 599.8304390 MHz  
 Power 95 dB  
 on during acquisition  
 off during delay  
 WALTZ-16 modulated  
 DATA PROCESSING  
 Line broadening 0.5 Hz  
 FT size 131072  
 Total time 7 min, 18 sec



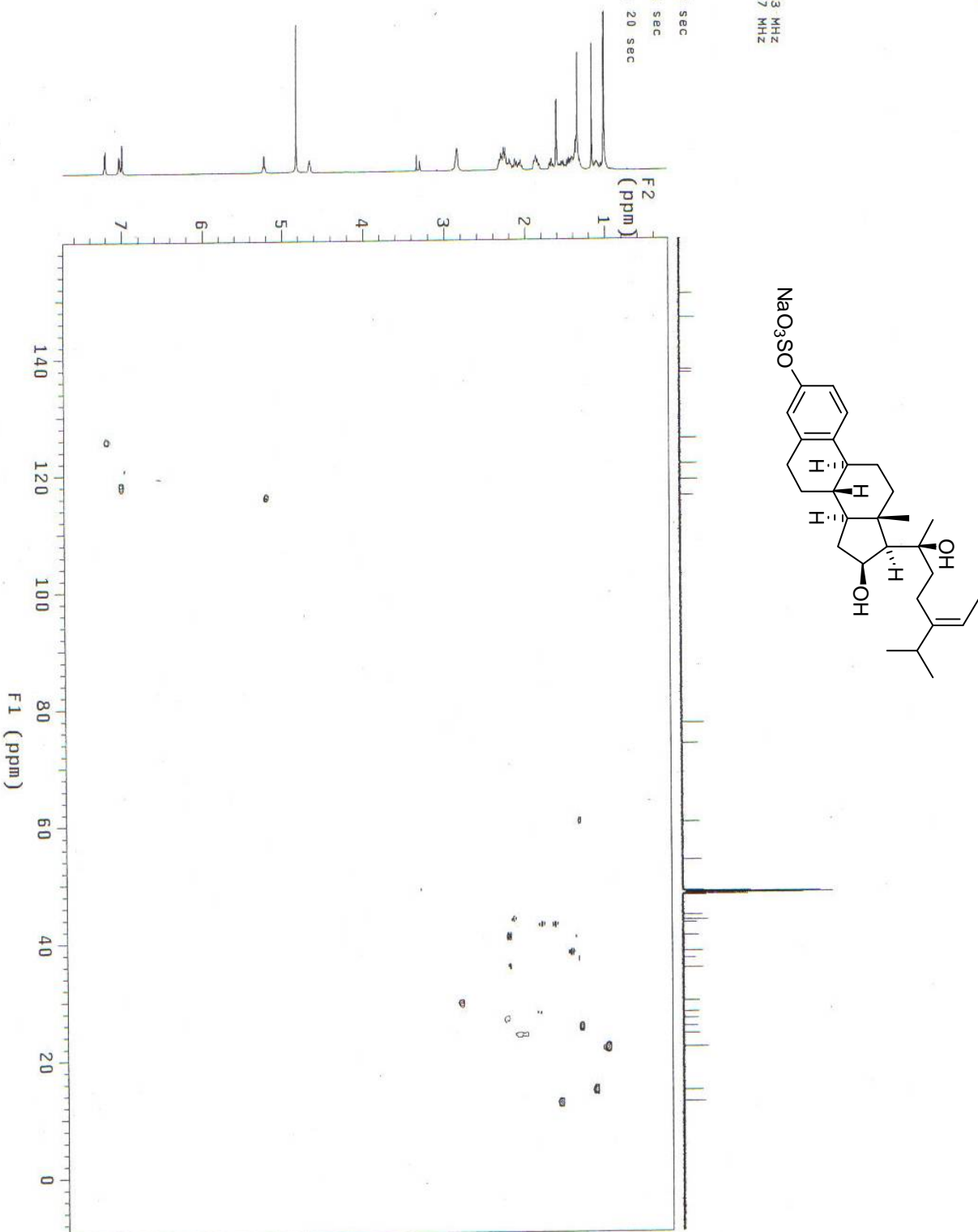
S5. 600 MHz HMQC spectrum compound 1 in methanol-d<sub>4</sub>

STANDARD PROTON PARAMETERS

Archive directory: /expnor/home/frank/vnmrSYS/data  
 Sample directory: Jd-f10  
 F1 file: PROTON

Pulse Sequence: ghmqc  
 Solvent: CD300  
 Temp: 25.0 C / 298.1 K  
 User: 1-14-87  
 INOVA-600 "unknown"

Relax. delay 1.000 sec  
 Acq. time 0.227 sec  
 Width 4513.7 Hz  
 2D Width 25641.0 Hz  
 64 repetitions  
 2 x 64 increments  
 OBSERVE H1, 599.8874393 MHz  
 DECOUPLE C13, 150.8530937 MHz  
 Power 40 dB  
 on during acquisition  
 off during delay  
 GARP-1 modulated  
 DATA PROCESSING  
 Gauss apodization 0.105 sec  
 F1 DATA PROCESSING  
 Gauss apodization 0.009 sec  
 FT size 2048 x 2048  
 Total time 2 hr, 55 min, 20 sec



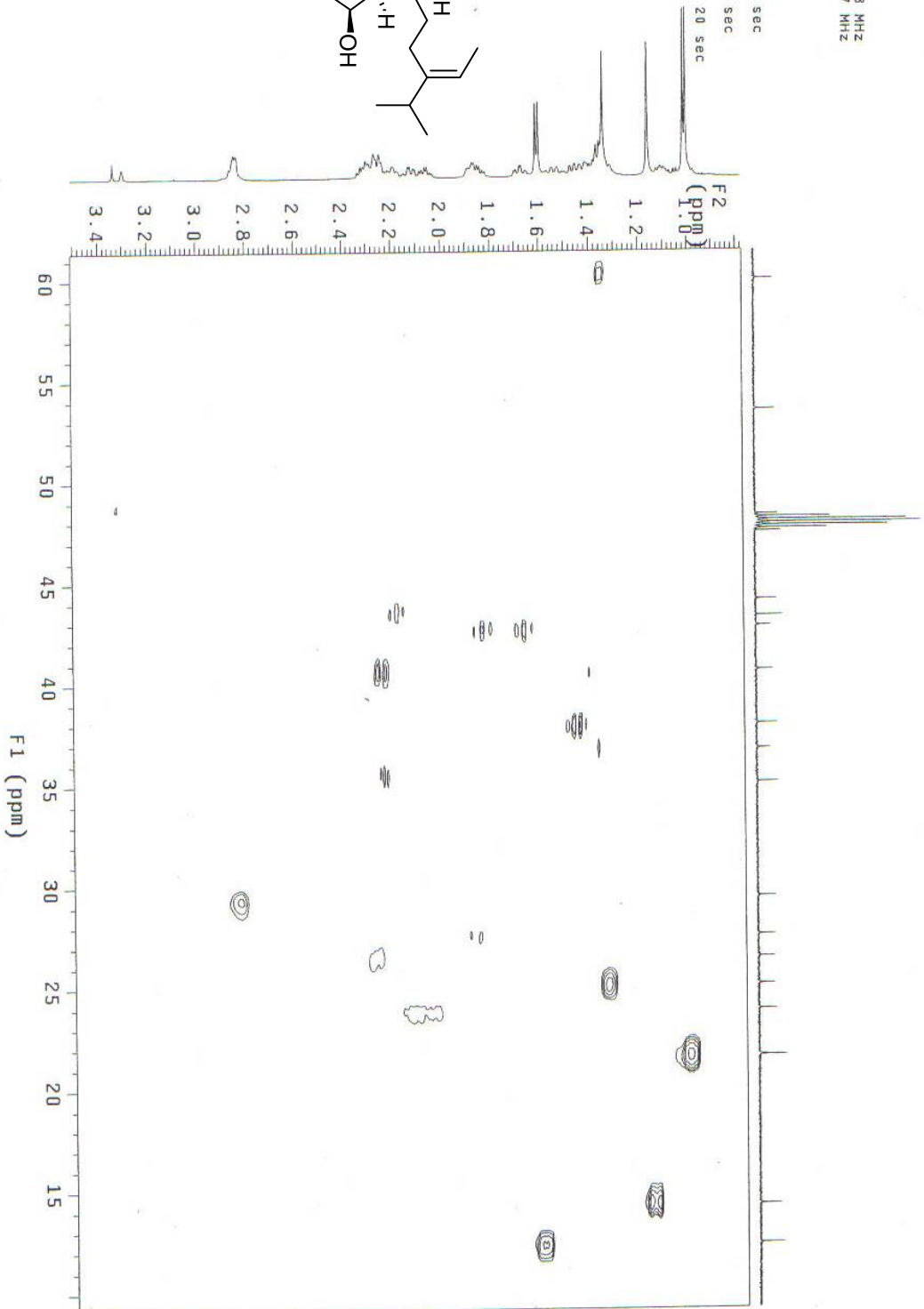
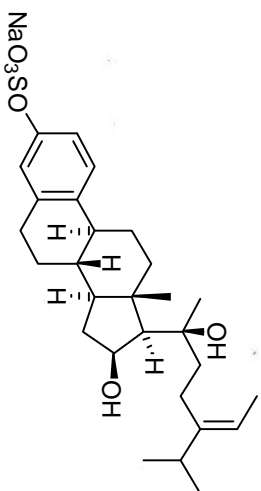
S6. 600 MHz HMOC spectrum of compound 1 in methanol-d<sub>4</sub> (expansion)

STANDARD PROTON PARAMETERS

Archive directory: /export/home/frank/vnmrSYS/data  
 Sample directory: Jd-F10  
 File: PROTON

Pulse Sequence: ghmqc  
 Solvent: CD3OD  
 Temp: 25.0 C / 298.1 K  
 User: 1-14-87  
 INOVA-600 "unknown"

Relax. delay 1.000 sec  
 Acq. time 0.227 sec  
 Width 4513.7 Hz  
 2D Width 25641.0 Hz  
 64 repetitions  
 2 x 64 increments  
 OBSERVE H1, 599.8874393 MHz  
 DECOUPLE C13, 150.8530937 MHz  
 Power 40 dB  
 on during acquisition  
 off during delay  
 GARP-1 modulated  
 DATA PROCESSING  
 Gauss apodization 0.105 sec  
 F1 DATA PROCESSING  
 Gauss apodization 0.009 sec  
 FT size 2048 X 2048  
 Total time 2 hr, 55 min, 20 sec

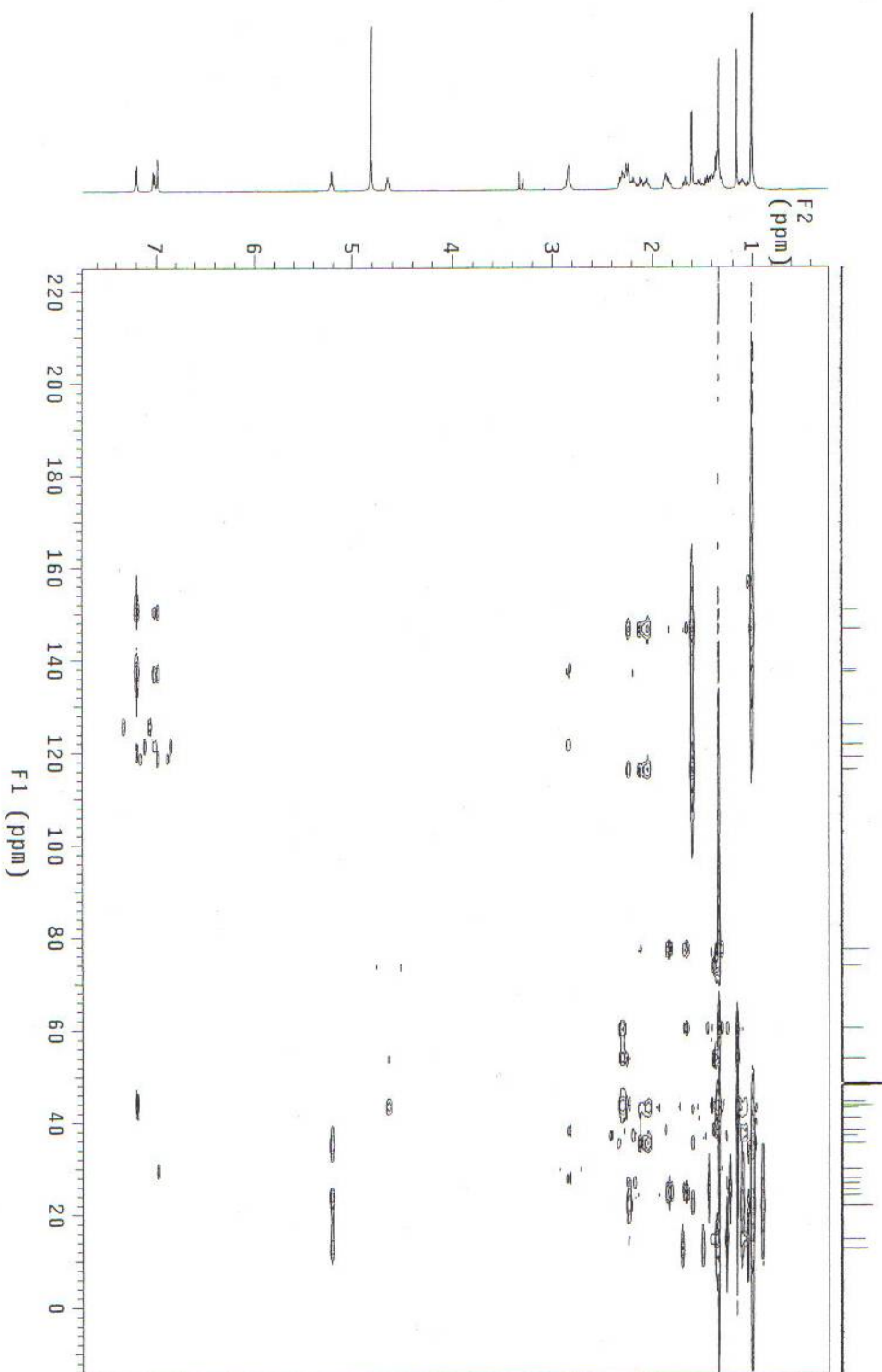
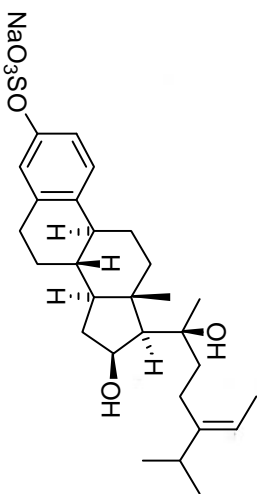


STANDARD PROTON PARAMETERS

Archive directory: /export/home/frank/vnmrSYS/data  
Sample directory: jd-f10  
File: PROTON

Pulse Sequence: ghmbrc  
Solvent: CD300  
Temp: 25.0 C / 298.1 K  
User: 1-14-87  
INOVA-600 "unknown"

Relax. delay 1.000 sec  
Acq. time 0.227 sec  
Width 4513.7 Hz  
2D Width 36199.1 Hz  
64 repetitions  
128 increments  
OBSERVE H1, 599.8874393 MHz  
DATA PROCESSING  
Sine bell 0.113 sec  
F1 DATA PROCESSING  
Sine bell 0.006 sec  
FT size 2048 x 2048  
Total time 3 hr, 12 sec



S7. 600 MHz HMBC spectrum of compound 1 in methanol-d4



S8. 600 MHz HMBC spectrum compound 1 in methanol-d<sub>4</sub> (expansion)

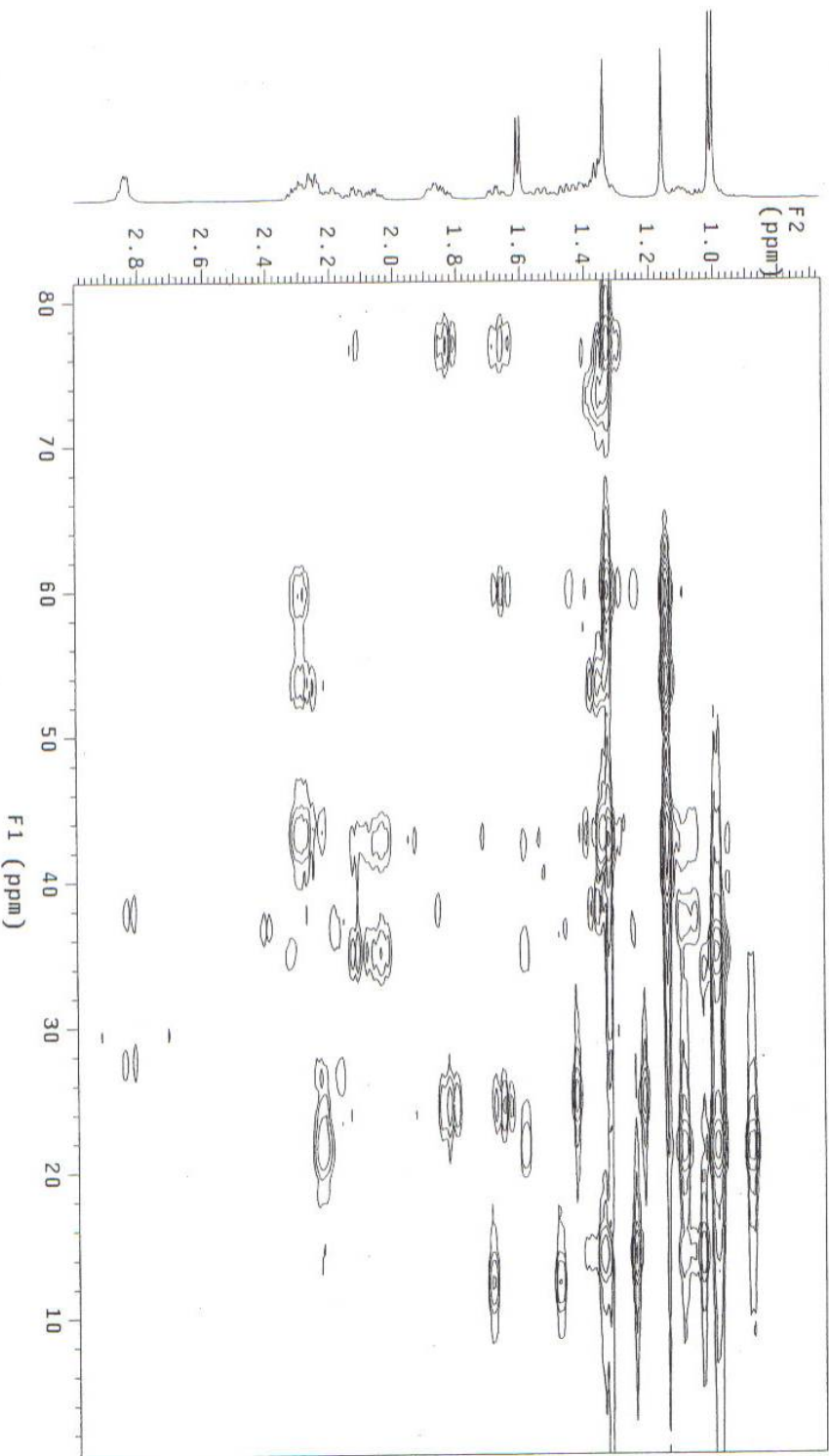
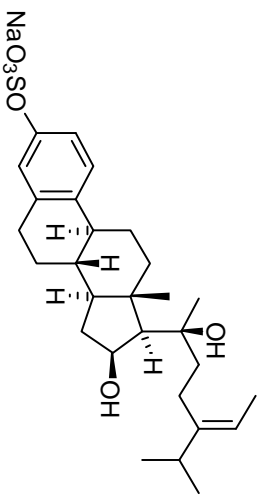
STANDARD PROTON PARAMETERS

Archive directory: /export/home/frank/vnmrSYS/data  
Sample directory: jd-f10  
File: PROTON

Pulse Sequence: gHMBC

Solvent: CD3OD  
Temp: 25.0 C / 298.1 K  
User: 1-14-87  
INDVA=600 "unknown"

Relax.: delay 1.000 sec  
Acq. time 0.227 sec  
Width 4513.7 Hz  
2D Width 36199.1 Hz  
3D repetitions  
3D increments  
OBSERVE H1, 599.8874393 MHz  
DATA PROCESSING  
Sine bell 0.113 sec  
F1 DATA PROCESSING  
Sine bell 0.006 sec  
F1 size 2048 X 2048  
Total time 3 hr, 12 sec



S9: 600 MHz COSY spectrum of compound 1 in methanol-d<sub>4</sub>

STANDARD PROTON PARAMETERS

Archive directory: /export/home/frank/vnmr sys/data  
Sample directory: Jd-f110  
File: PROTON

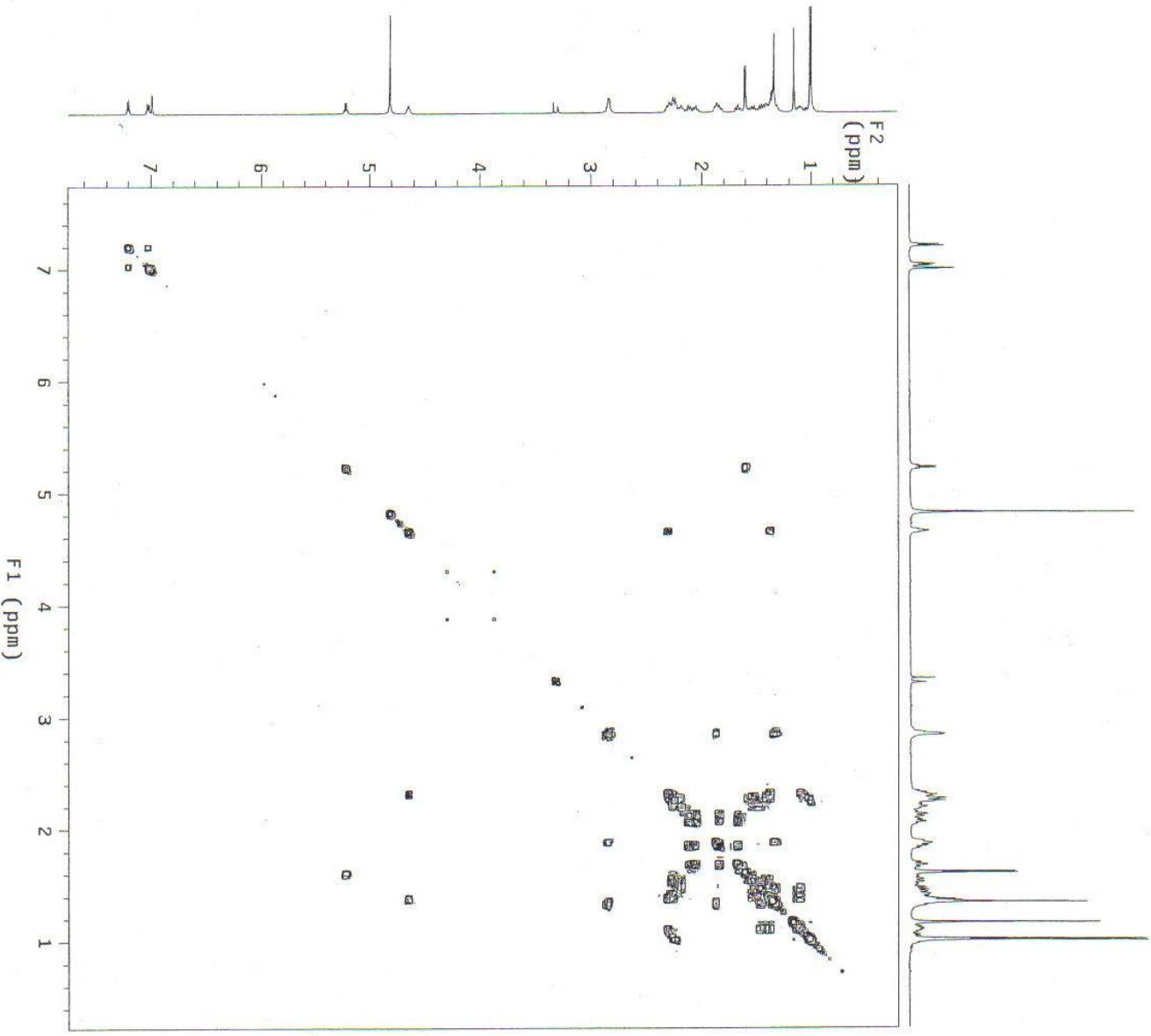
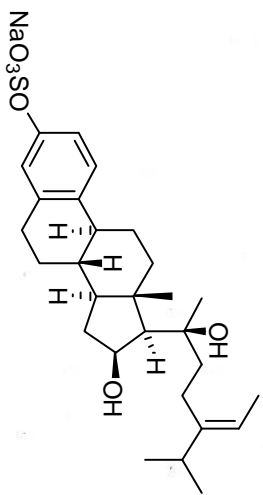
Pulse Sequence: COSY

Solvent: CD30D  
Temp: 25.0 C / 298.1 K  
INOVA-600 "unknown"

Relax: delay 1.000 sec  
Acq: time 0.227 sec  
Width 4513.7 Hz  
2D Width 4513.7 Hz  
8 repetitions  
128 increments

OBSERVE H1, 599.8874393 MHz  
DATA PROCESSING

Sq. sine bell 0.113 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.028 sec  
Ft size 2048 x 2048  
Total time 22 min, 0 sec

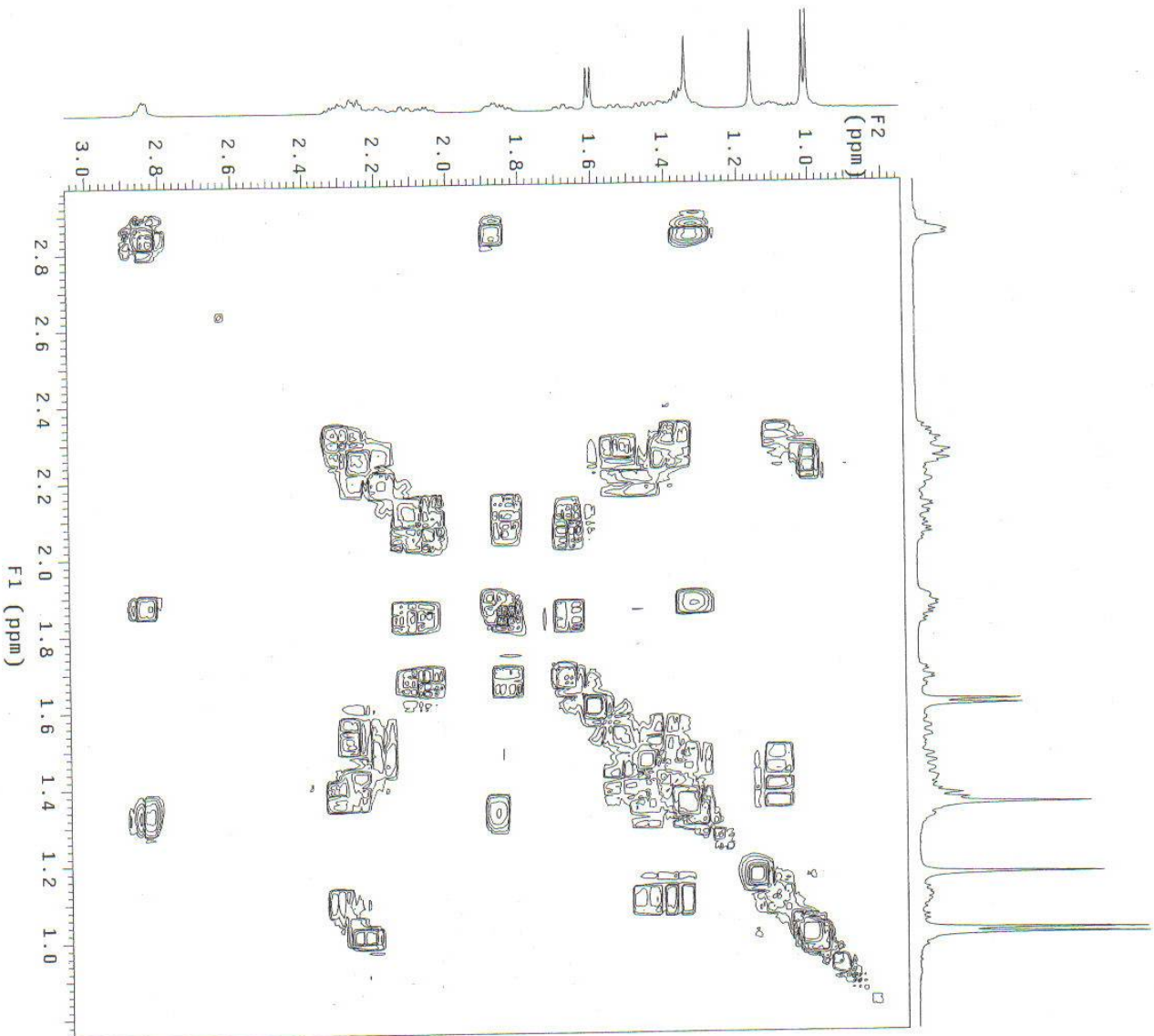
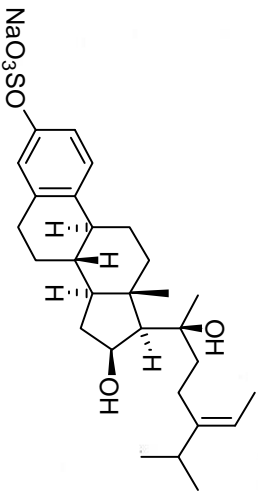


S10. 600 MHz COSY spectrum of compound 1 in methanol-d<sub>4</sub> (expansion)

STANDARD PROTON PARAMETERS  
 Archive directory: /export/home/frank/vnmrSYS/data  
 Sample directory: jd-f10  
 File: PROTON

Pulse Sequence: COSY  
 Solvent: CD3OD  
 Temp: 45.0 C / 298.1 K  
 INOVA-600 "unknown"

Relax. delay 1.000 sec  
 Acq. time 0.227 sec  
 Width 4513.7 Hz  
 2D Width 4513.7 Hz  
 8 repetitions  
 128 increments  
 OBSERVE H1, 599.8874393 MHz  
 DATA PROCESSING  
 Sq. sine bell 0.113 sec  
 F1 DATA PROCESSING  
 Sq. sine bell 0.028 sec  
 Ft size 2048 x 2048  
 Total time 22 min, 0 sec



S11. 600 MHz NOESY spectrum of compound 1 in methanol-d4

STANDARD PROTON PARAMETERS

Archive directory: /export/home/frank/vnmrSYS/data  
Sample directory: jd-f10  
File: PROTON

Pulse Sequence: NOESY

Solvent: CD3OD  
Temp: 25.0 C / 298.1 K  
INDVA-600 "unknown"

Relax. delay 3.000 sec

Mixing 0.800 sec

Acq. time 0.227 sec

Width 4513.7 Hz

2D Width 4513.7 Hz

8 repetitions

2 x 128 increments

OBSERVE H1, 599.8674393 MHz

DATA PROCESSING

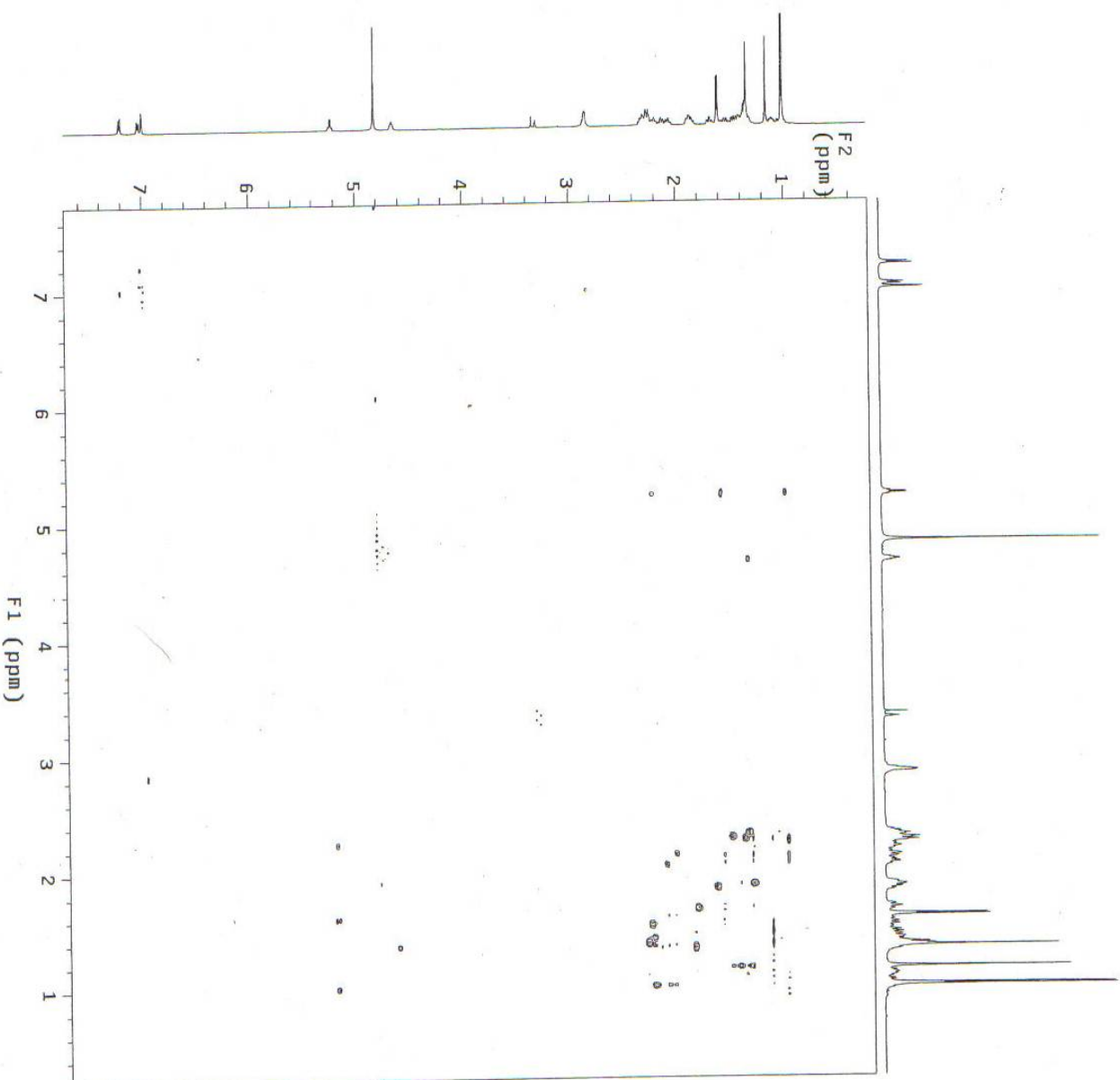
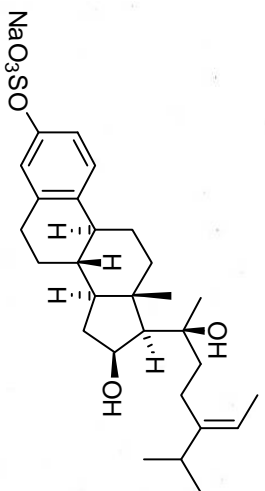
Gauss apodization 0.105 sec

F1 DATA PROCESSING

Gauss apodization 0.041 sec

FT size 2048 x 2048

Total time 2 hr, 20 min, 24 sec



S12. 600 MHz NOESY spectrum of compound **1** in methanol-d<sub>4</sub> (expansion)

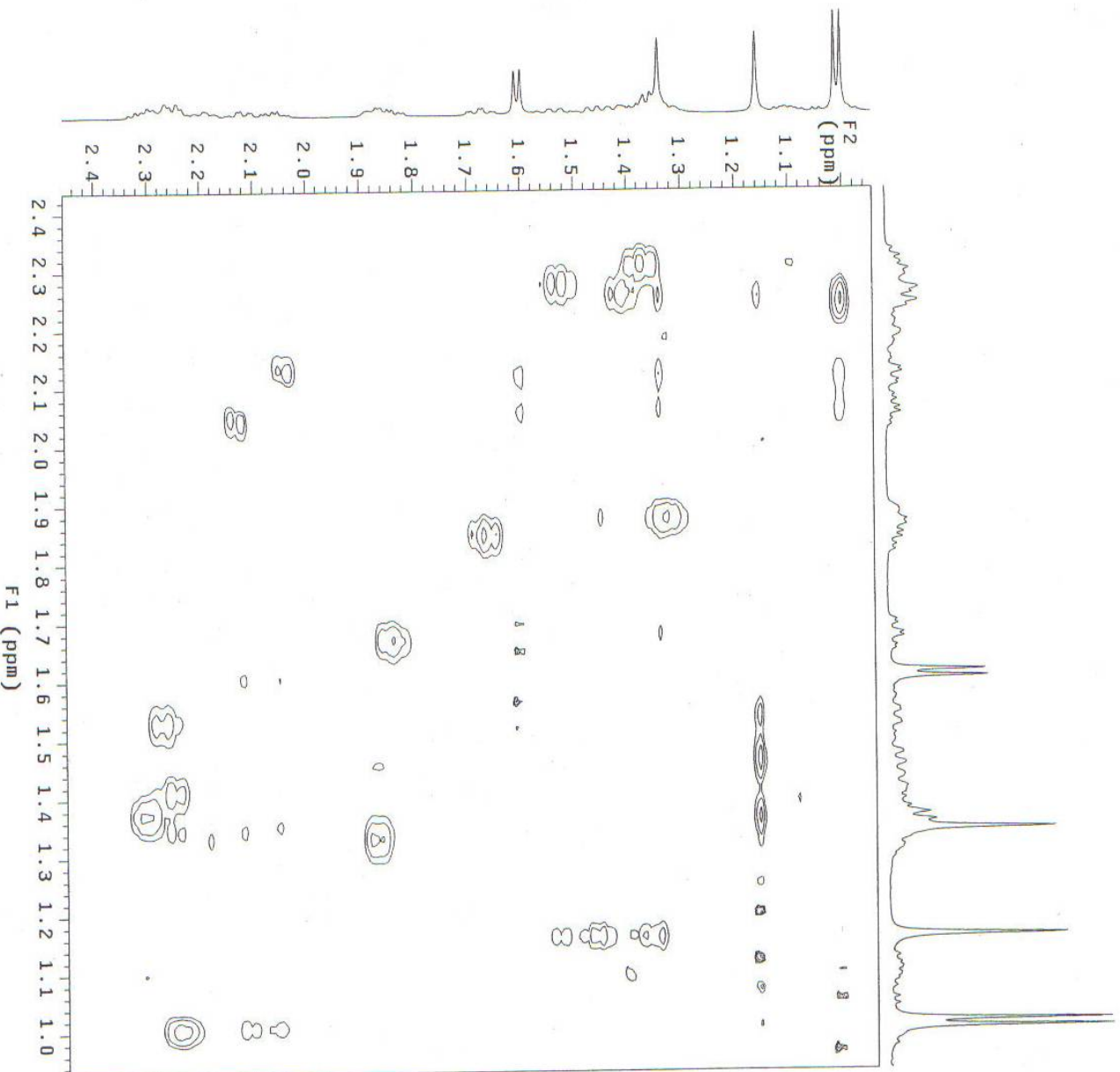
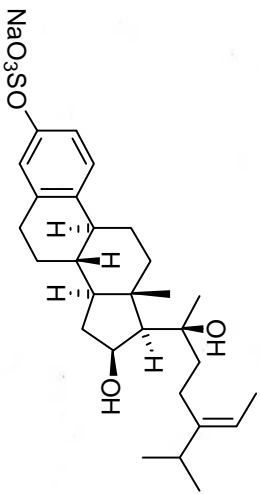
STANDARD PROTON PARAMETERS

Archive directory: /export/home/frank/vnmrsys/data  
 Sample directory: jd-f10  
 File: PROTON

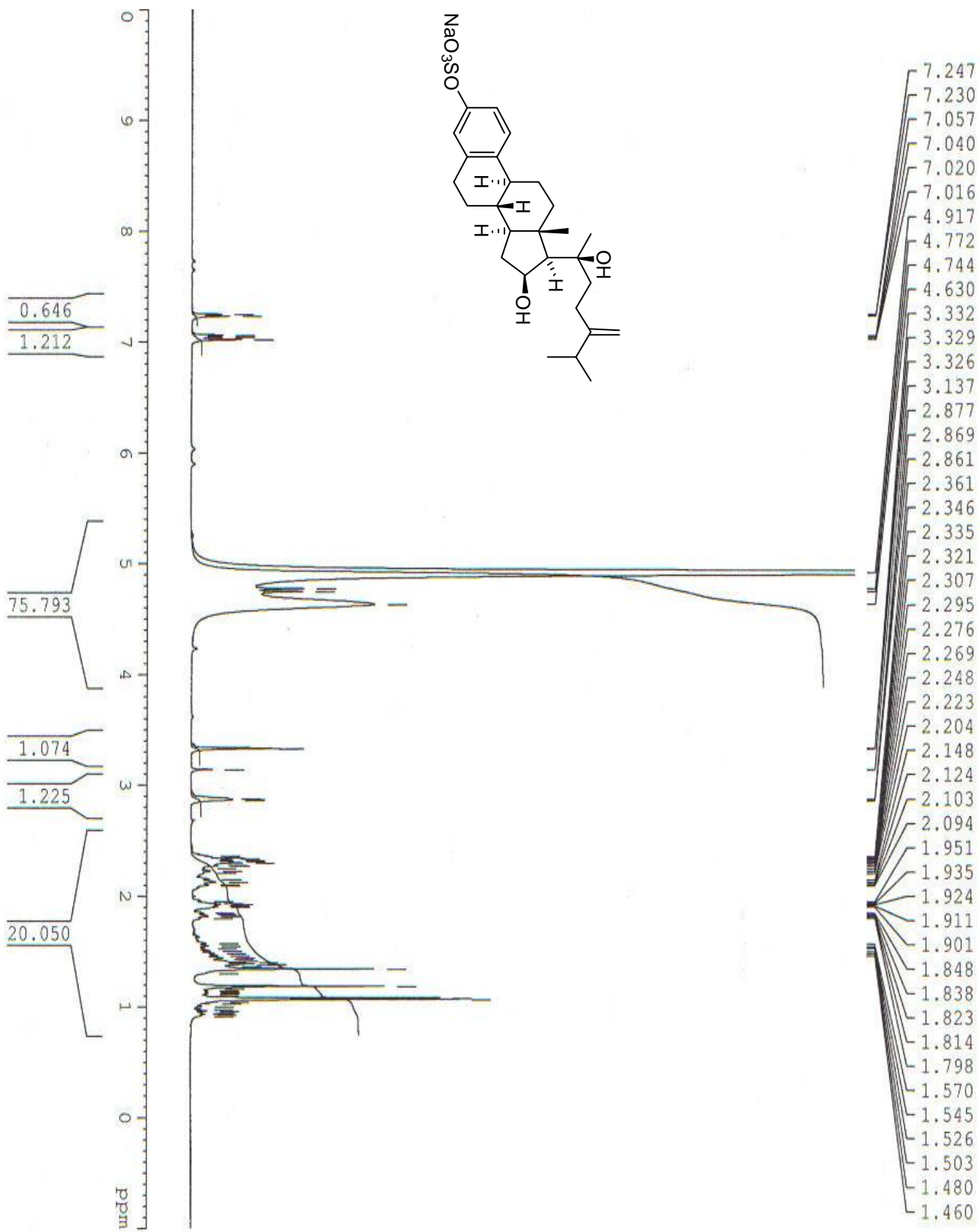
Pulse Sequence: NOESY

Solvent: CD3OD  
 Temp: 25.0 C / 298.1 K  
 INOVA-600 "unknown"

Relax. delay 3.000 sec  
 Mixing 0.800 sec  
 Acq. time 0.227 sec  
 Width 4513.7 Hz  
 2D Width 4513.7 Hz  
 8 repetitions  
 2 x 128 increments  
 OBSERVE H1, 599.8874393 MHz  
 DATA PROCESSING  
 Gauss apodization 0.105 sec  
 F1 DATA PROCESSING  
 Gauss apodization 0.041 sec  
 FT size 2048 X 2048  
 Total time 2 hr, 20 min, 24 sec

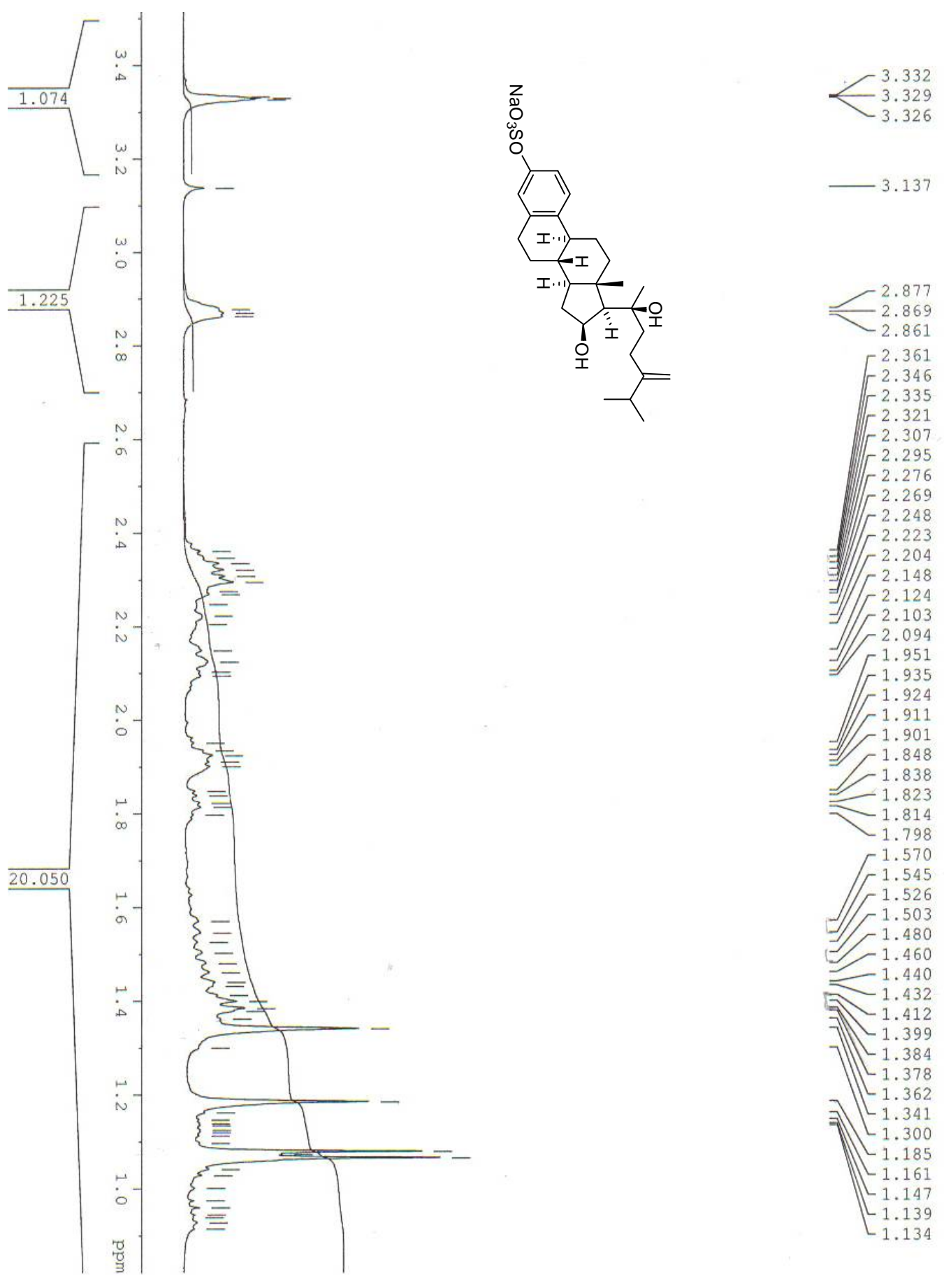


S13. 500 MHz <sup>1</sup>H NMR spectrum of compound 1 in methanol-d<sub>4</sub>.



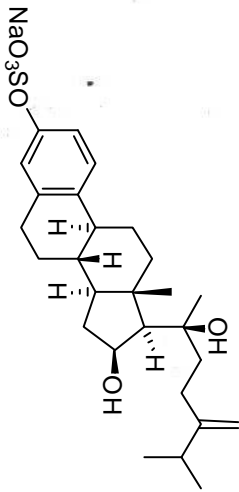
\*  $\delta_{\text{H}}$  (methanol-d<sub>4</sub>) 3.33; the data in the manuscript is calibrated by using the  $\delta_{\text{H}}$  (methanol-d<sub>4</sub>) 3.33.

S14. 500 MHz <sup>1</sup>H NMR spectrum of compound 2 in CD<sub>3</sub>OD (expansion)

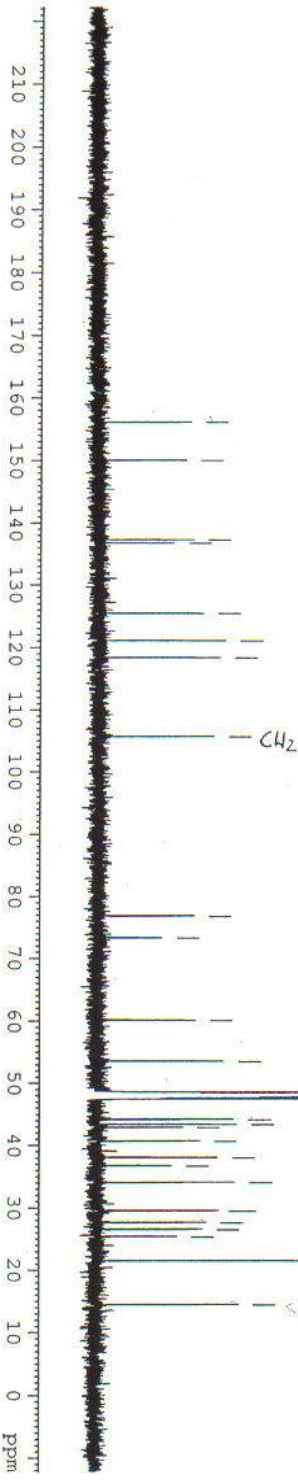


S15. 125 MHz <sup>13</sup>C NMR spectrum of compound 2 in methanol-d<sub>4</sub>

CARBON



156.066	✓
149.983	✓
137.233	✓
136.691	✓
125.389	✓
121.015	✓
118.301	✓
105.623	✓
76.776	C
73.264	CH
60.026	CH
53.459	CH
48.418	
48.248	
48.079	
47.909	
47.739	
47.569	
47.400	
44.111	CH
43.296	C
42.825	CH <sub>2</sub>
40.630	CH <sub>2</sub>
37.973	CH
36.678	CH <sub>2</sub>
34.020	CH
29.480	
29.432	
27.588	
26.500	
25.322	
21.418	
14.398	



Current Data Parameters  
 NAME: jd-trach-6-7  
 EXPNO: 2  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20050911  
 Time: 19.25  
 INSTRUM: spect  
 PROBD: 5 mm Dual 13C/  
 PULPROG: zgpgc30  
 TD: 65536  
 SOLVENT: MeOD  
 NS: 5000  
 DS: 4  
 SMH: 29498.525 Hz  
 FIDRES: 0.450112 Hz  
 AQ: 1.1108851 sec  
 RG: 11585.2  
 DE: 16.950 usec  
 TE: 300.0 K  
 D1: 3.00000000 sec  
 D11: 0.03000000 sec

CHANNEL F1  
 NUC1: 13C  
 P1: 8.00 usec  
 PL1: 3.00 dB  
 SFO1: 125.770936 MHz

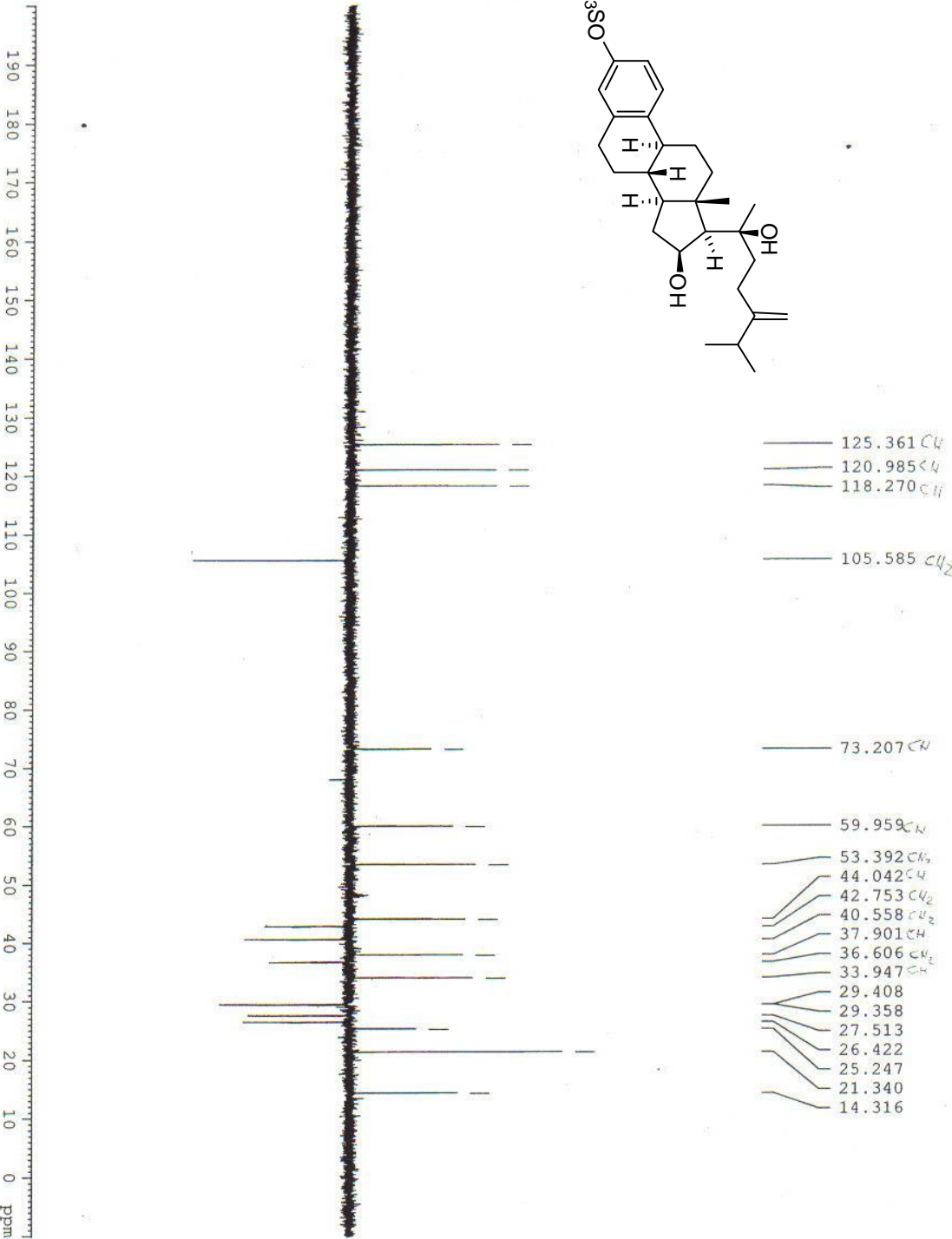
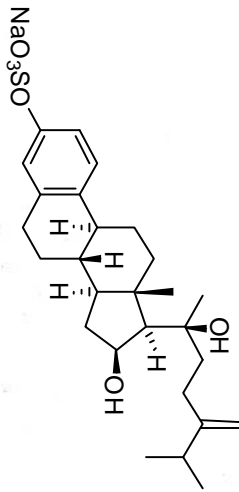
CHANNEL F2  
 NUC2: 13C  
 P2: 100.00 usec  
 PL2: 3.00 dB  
 SFO2: 500.1320005 MHz

Processing parameters  
 SI: 32768  
 SF: 125.7577890 MHz  
 MDW: EM  
 SSB: 0  
 LB: 1.00 Hz  
 GB: 0  
 PC: 1.40



S16. 125 MHz DEPT-135 spectrum of compound 2 in methanol-d4

DEPT135



Chemical Shift (ppm)	Assignment
125.361	CH
120.985	CH
118.270	CH
105.585	CH <sub>2</sub>
73.207	CH
59.959	CH
53.392	CH <sub>2</sub>
44.042	CH
42.753	CH <sub>2</sub>
40.558	CH <sub>2</sub>
37.901	CH
36.606	CH <sub>2</sub>
33.947	CH
29.408	
29.358	
27.513	
26.422	
25.247	
21.340	
14.316	

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Current Data Parameters
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EXPNO     3
PROCNO    1

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Date_     20050911
Time      22.08
INSTRUM   zgpg30
PROBHD    5 mm Dual 13C/
PULPROG   dept135
TD         65536
SOLVENT   MeOD
NS         3000
DS         4
SWH        26455.027 Hz
FIDRES     0.402672 Hz
AQ         1.226804 sec
RG         178.900
DSF         18.900 usec
DE         6.00 usec
TE         300.0 K
CNST2     145.0000000
D1         2.000000000 sec
d2         0.00344828 sec
d12        0.0002000 sec
DELTA     0.00001019 sec

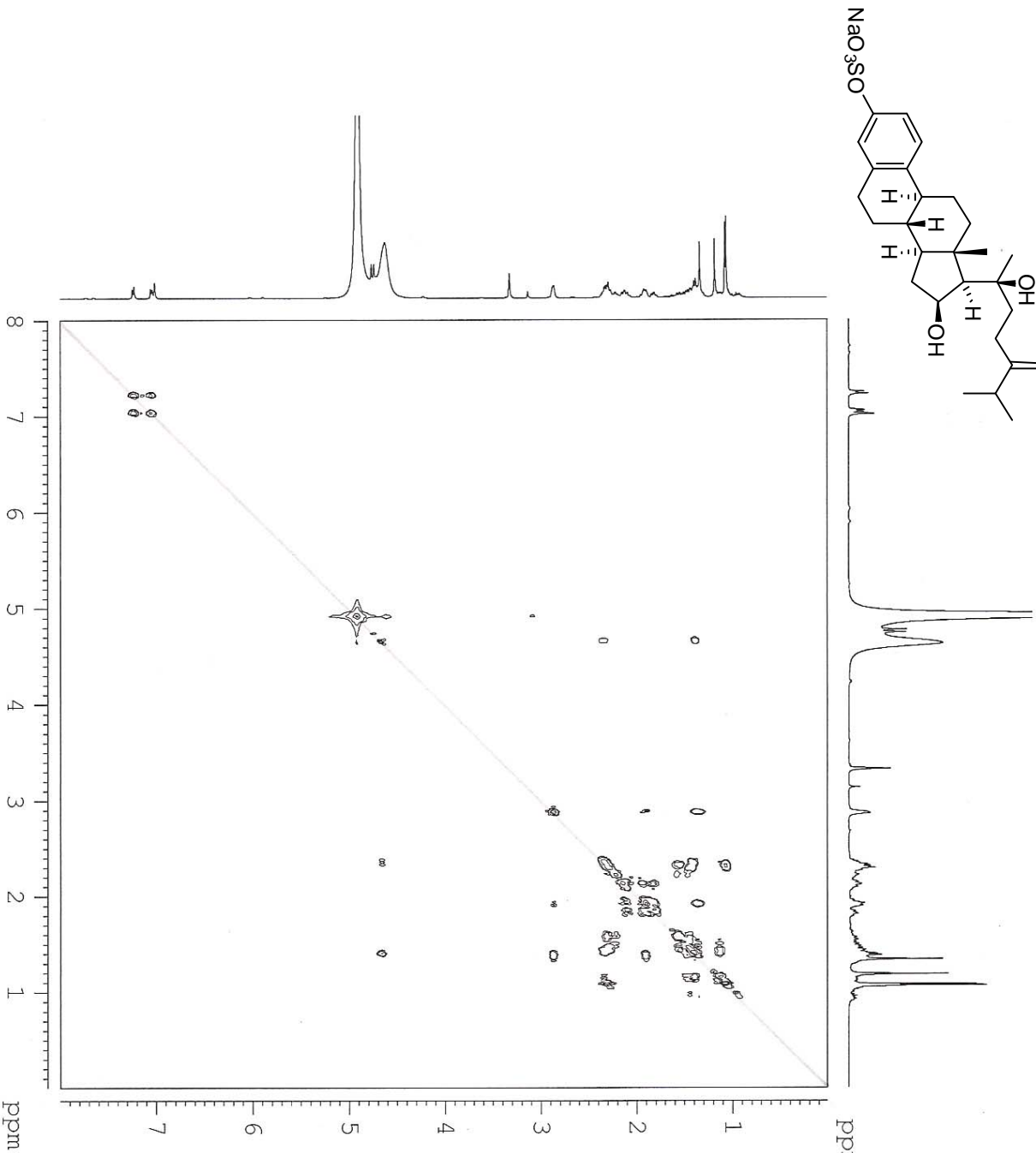
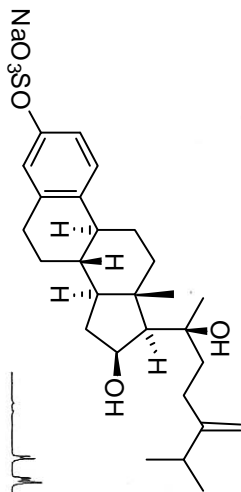
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NUC1       13C
P1         8.00 usec
PL1        16.00 dB
SFO1       125.7697360 MHz

===== CHANNEL f2 1H =====
CPDPRG2   waltz16
NUC2       1H
P2         13.00 usec
PL2        0.00 dB
SFO2       500.1320005 MHz

F2 - Processing parameters
SI         32768
SF         125.757890 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```

S17. 500 MHz COSY spectrum of compound 2 in methanol-d<sub>4</sub>

DOF COSY



Current Data Parameters  
 NAME Jd-trach-6-7  
 EXPNO 4  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20050911  
 Time 22:03  
 INSTRUM spect  
 PROBRD 5 mm Dual 13C/  
 PULPROG cosygpmrf  
 TD 2048  
 SOLVENT MeOD  
 NS 16  
 DS 8  
 SWH 4006.410 Hz  
 FIDRES 1.956255 Hz  
 AQ 0.2556404 sec  
 RG 32768  
 DW 124.800 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D0 0.00000300 sec  
 D1 0.00000000 sec  
 D13 0.00000400 sec  
 D16 0.00010000 sec  
 INO 0.00024994 sec  
 MCRBST 0.00000000 sec  
 MCWRK 1.00000000 sec

CHANNEL f1  
 NUCL1 1H  
 P1 13.00 usec  
 PL1 3.00 dB  
 SFO1 500.1320005 MHz

GRADIENT CHANNEL  
 GENAM1 SINE.100  
 GENAM2 SINE.100  
 GENAM3 SINE.100  
 GEX1 0.00 %  
 GEX2 0.00 %  
 GEX3 0.00 %  
 GEX4 0.00 %  
 GEX5 0.00 %  
 GEX6 0.00 %  
 GEX7 0.00 %  
 GEX8 0.00 %  
 GEX9 0.00 %  
 GEX10 0.00 %  
 GEX11 0.00 %  
 GEX12 0.00 %  
 GEX13 0.00 %  
 GEX14 0.00 %  
 GEX15 0.00 %  
 GEX16 0.00 %  
 P16 1000.00 usec

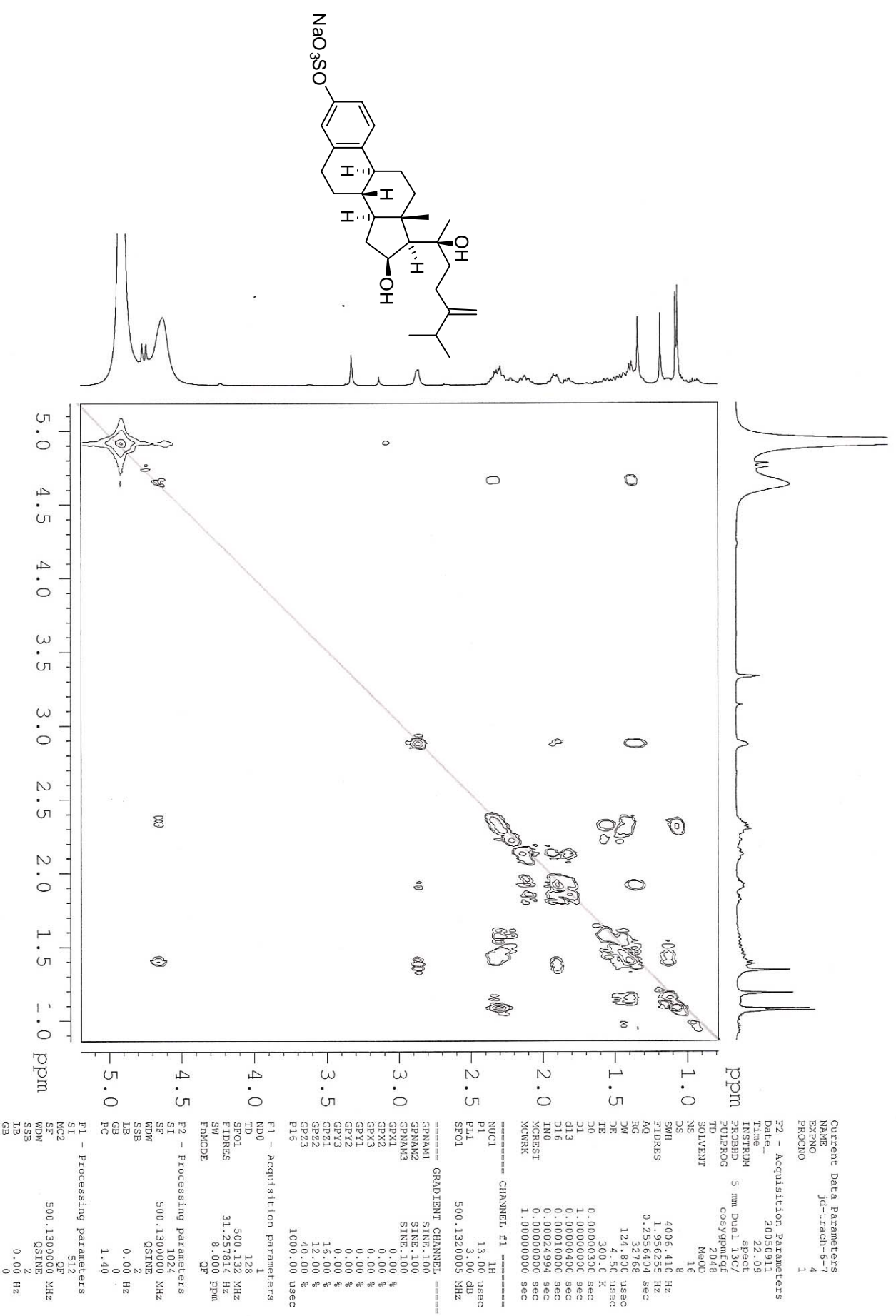
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 TD 128  
 SFO1 500.132 MHz  
 FIDRES 31.257814 Hz  
 SW 8.000 PPM  
 FMODE GE

F2 - Processing Parameters  
 SI 1024  
 SF 500.1300000 MHz  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

F1 - Processing Parameters  
 SI 512  
 MC2 GF  
 SF 500.1300000 MHz  
 WDW OSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0

S18. 500 MHz COSY spectrum of compound 2 in methanol-d4 (expansion)

DOF COSY



```

Current Data Parameters
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EXPNO        4
PROCNO       1
PROCNO       1

F2 - Acquisition Parameters
Date_        20050911
Time         22.09
INSTRUM     5 mm Dual 13C/
PROBHD      spect
PULPROG     cosygpmrfg
TD          2048
SOLVENT     MeOD
NS          16
DS          8
SWH         4006.410 Hz
FIDRES     1.356235 Hz
AQ         0.23592148 sec
RG         124.600 usec
DE         300.0 K
TE          300.2 K
DO         0.00000300 sec
D1         1.00000000 sec
d13        0.00000400 sec
D16        0.00010000 sec
IN0        0.00024994 sec
MCREST     0.00000000 sec
MCWRRK     1.00000000 sec

===== CHANNEL f1 =====
NUC1        1H
P1          13.00 usec
PL          3.00 dB
SFO1        500.1320005 MHz

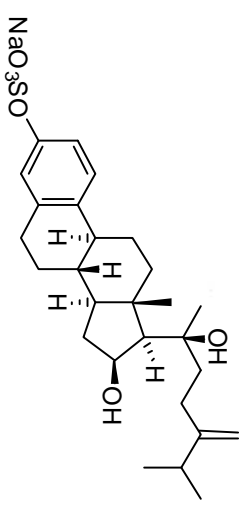
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SINE.100
GPNAM2     SINE.100
SINE.100
GPNAM3     SINE.100
SINE.100
GPX1       0.00 %
GPX2       0.00 %
GPX3       0.00 %
GPY1       0.00 %
GPY2       0.00 %
GPY3       0.00 %
GPZ1       16.00 %
GPZ2       12.00 %
GPZ3       40.00 %
P16        1000.00 usec

F1 - Acquisition Parameters
ND0        1
TD         128
SFO1       500.132 MHz
FIDRES     31.257614 Hz
SN         8.000 PPM
FMODE      QF

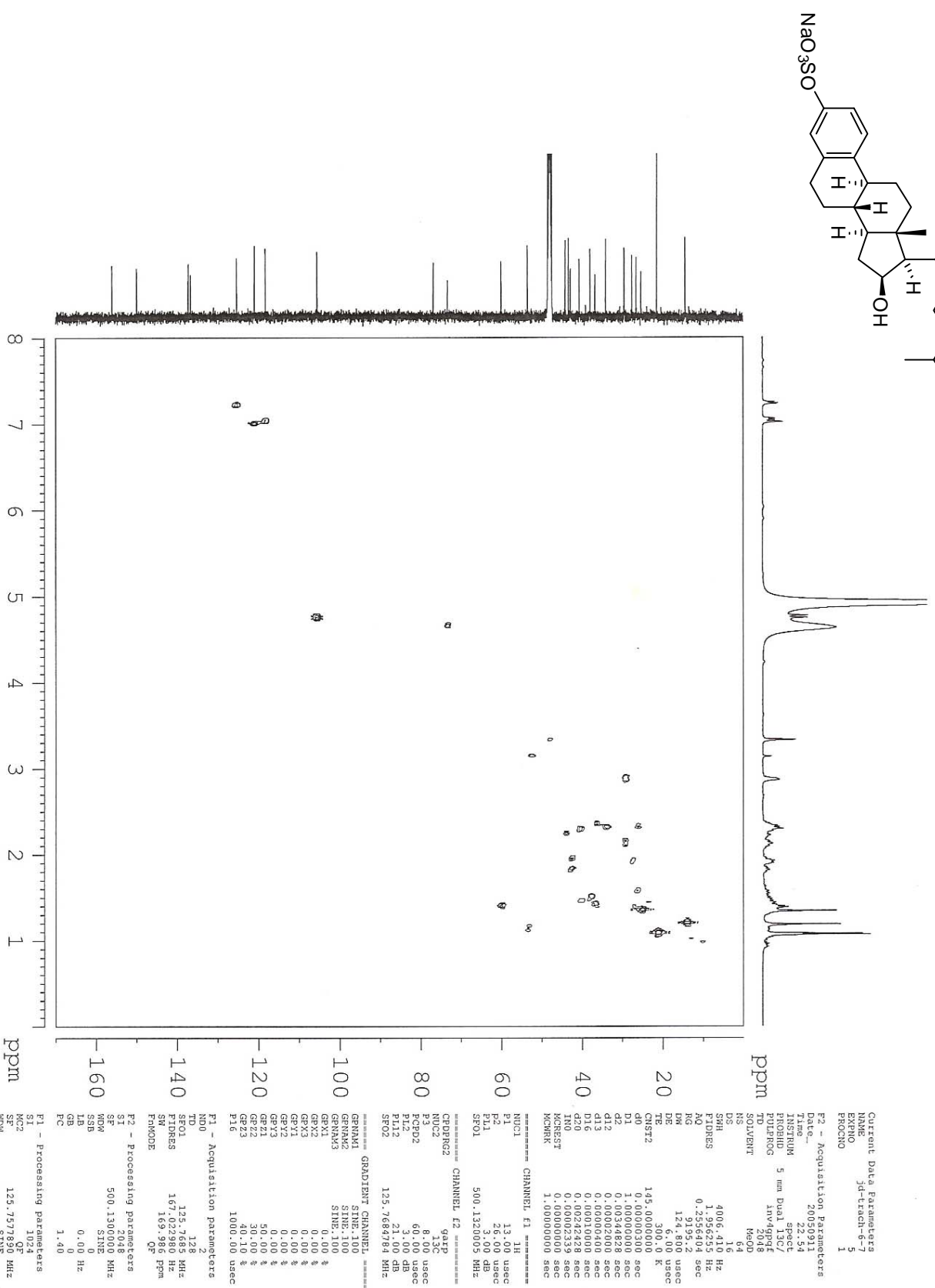
F2 - Processing parameters
SI         512
SF         500.1300000 MHz
WDW        QSINE
SSB        2
GB         0.00 Hz
PC         1.40

F1 - Processing parameters
SI         512
SF         500.1300000 MHz
WDW        QSINE
SSB        2
GB         0.00 Hz
PC         1.40
    
```

S19. 500 MHz HMOC spectrum of compound 2 in methanol-d4



HMOC



Current Data Parameters  
 EXPNO 5  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20050911  
 Time 22:27  
 INSTRUM spect  
 PROBMU 5 mm Dual 13C/  
 PULPROG zgpg30  
 TD 2048

SOLVENT ResD  
 DS 16  
 SWH 4006.410 Hz  
 FIDRES 1.956295 Hz  
 AQ 0.2556404 sec  
 RG 9195.2  
 RT 12.497000  
 DE 6.00 usec  
 TE 300.0 K

CHST2 145.0000000  
 d0 0.0000300 sec  
 d1 1.0000000 sec  
 d12 0.0000400 sec  
 d13 0.0000200 sec  
 d16 0.0001000 sec  
 d20 0.0024248 sec  
 d21 0.0000239 sec  
 d22 0.0000000 sec  
 d23 1.0000000 sec

CHANNEL F1  
 NUCL 13  
 P1 26.00 usec  
 PL1 3.00 dB  
 SFO1 500.1320005 MHz

CHANNEL F2  
 GRPP 131.4  
 NUCL 1H  
 P3 8.00 usec  
 PCPDP 60.00 usec  
 PL2 3.00 dB  
 PL12 21.00 dB  
 SFO2 125.7687084 MHz

GRADIENT CHANNEL  
 GRNAM1 SINE.100  
 GRNAM2 SINE.100  
 GRNAM3 SINE.100  
 GRX1 0.00 %  
 GRX2 0.00 %  
 GRX3 0.00 %  
 GPY1 0.00 %  
 GPY2 0.00 %  
 GPY3 0.00 %  
 GZ1 30.00 %  
 GZ2 30.00 %  
 GZ3 40.10 %  
 P16 1000.00 usec

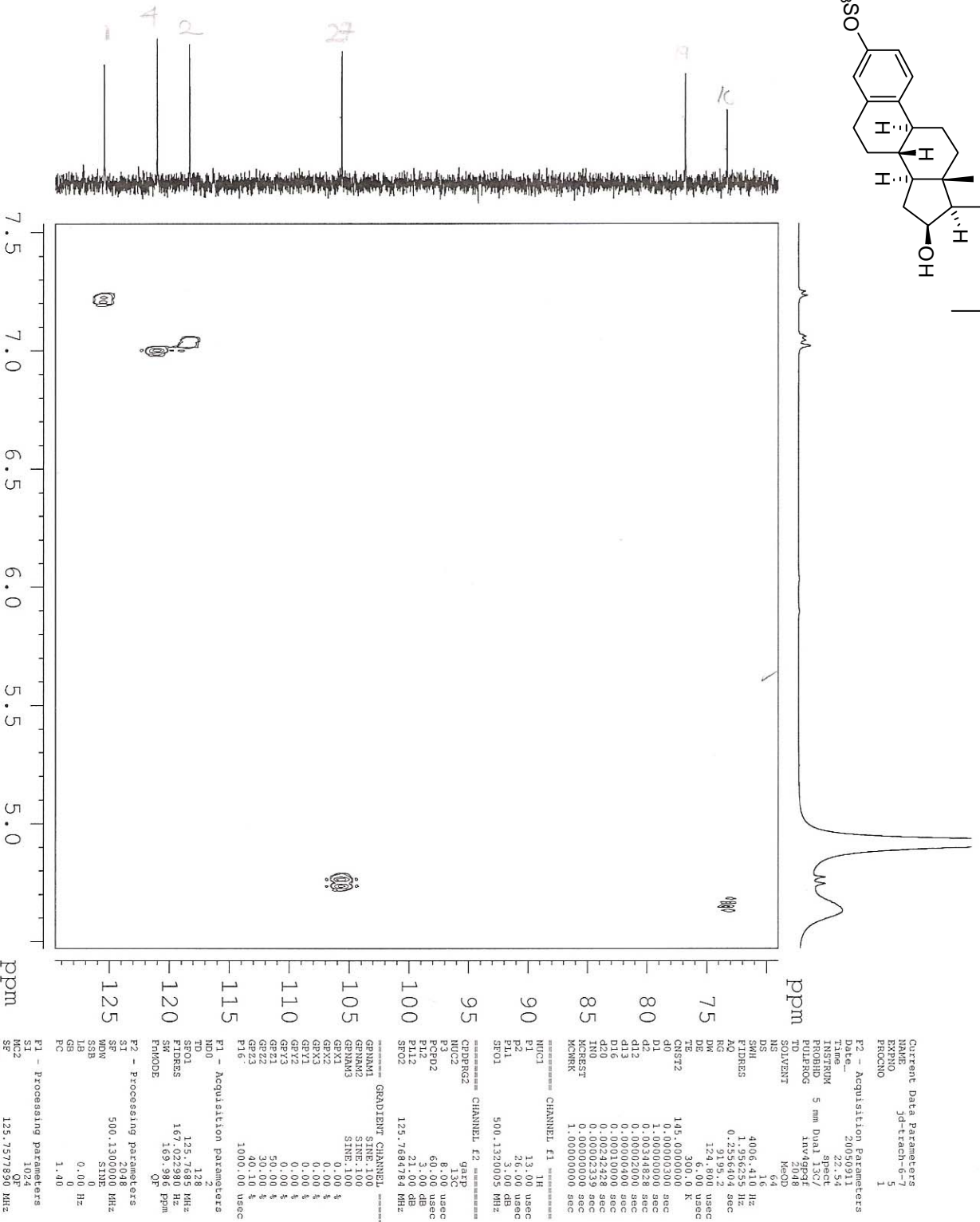
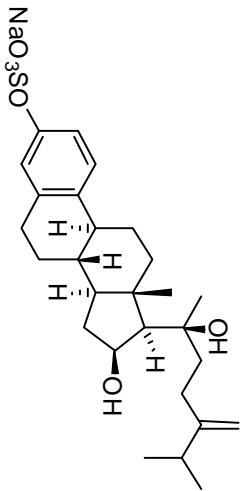
F1 - Acquisition Parameters  
 TD 128  
 SFO1 125.7685 MHz  
 FIDRES 167.022980 Hz  
 SW 169.986 Ppm  
 FWHODE QF

F2 - Processing parameters  
 SI 2048  
 SF 500.1300000 MHz  
 MDW SINE  
 SSB 0.00 Hz  
 GB 0  
 PC 1.40

F1 - Processing parameters  
 SI 1024  
 SF 125.7577890 MHz  
 MDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

S20. 500 MHz HMQC spectrum of compound 2 in methanol-d4 (expansion)

HMQC



```

Current Data Parameters
NAME          Jd-trach-6-7
EXPNO        1
PROCNO       1
F2 - Acquisition Parameters
Date_        20050911
Time         22.54
INSTRUM      spect
PROBHD       5 mm DualSpec
PULPROG      jmv4pqt
TD           2048
SOLVENT      MeOD
NS           64
DS           4
SFO1         4006.416 Hz
SFO2         1.956235 Hz
AQ           0.2558404 sec
RG           9195.2
DW           124.800 usec
DE           8.00 usec
TE           300.2 K
CHST2       145.0000000
d0           0.00000300 sec
d1           1.000000000 sec
d2           0.00344828 sec
d42          0.00002000 sec
d12          0.00310000 sec
d13          0.00310000 sec
d20          0.00242428 sec
TNO          0.00002339 sec
KCREST      0.00000000 sec
KCMKR       1.00000000 sec

CHANNEL F1
NUC1         1H
P1           13.00 usec
P2           26.00 usec
PL1          3.00 dB
SFO1         500.1320005 MHz

CHANNEL F2
CPDPRG2     garp
NUC2         13C
P3           8.00 usec
PCPD2       69.00 usec
P4           1.00 usec
PL2         21.00 dB
SFO2         125.7684784 MHz

GRADIENT CHANNEL
GPMAX1      SINE:100
GPMAX2      SINE:100
GPMIN3      SINE:100
GPX1        0.00 %
GPX2        0.00 %
GPX3        0.00 %
GPY1        0.00 %
GPY2        0.00 %
GPZ1        50.00 %
GPZ2        30.00 %
GPZ3        40.10 %
PI6         1000.00 usec

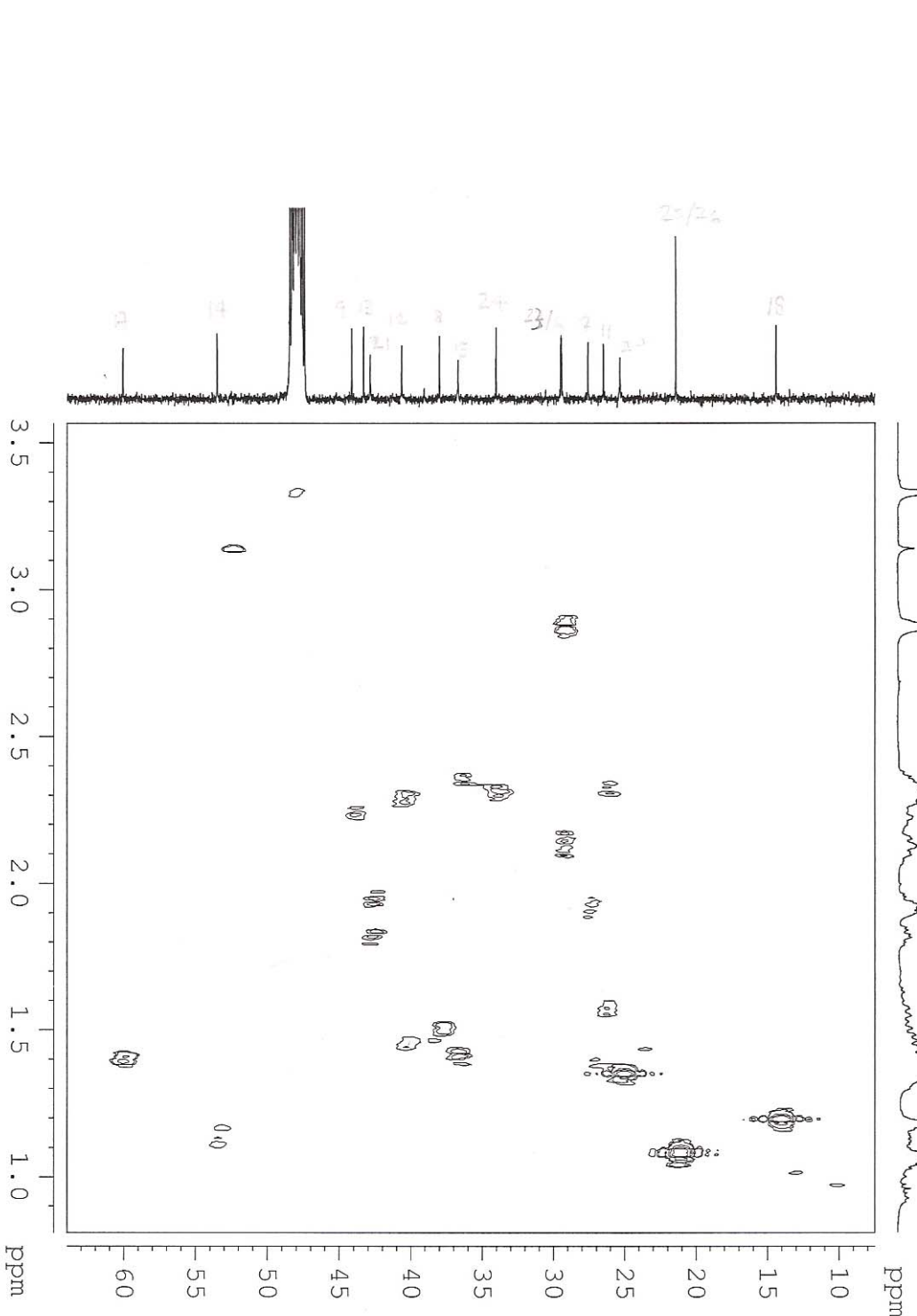
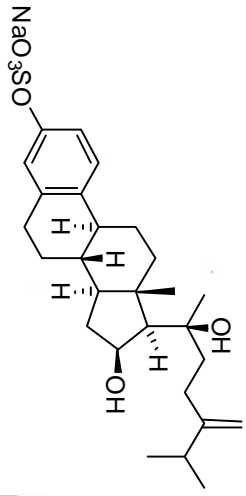
F1 - Acquisition parameters
NUC1         1H
TD           128
SFO1         125.7685 MHz
FIDRES      167.022980 Hz
SNR         169.986 ppm
FMODE       QF

F2 - Processing parameters
SI           2048
SF           500.1300000 MHz
SINE        SINE
SSB          0
GB           0.00 Hz
PC           1.40

F1 - Processing parameters
SI           1024
SF           125.7577880 MHz
SINE        SINE
SSB          0
GB           0.00 Hz
  
```

S21. 500 MHz HMQC spectrum of compound 2 in methanol-d4 (expansion)

HMQC



Current Data Parameters  
 NAME Jd-trach-6-7  
 PROCNM 1

F2 - Acquisition Parameters  
 Date\_ 20050911  
 Time 22.54  
 FIDRES 1.956235 Hz  
 PRGNAME 5 mm Dual  
 FULPRG 1drvprgf  
 TD 2048  
 SOLVENT MeOD  
 NS 64  
 SH 16  
 SI 1  
 FIDRES 1.956235 Hz  
 AQ 0.2556404 sec  
 RG 9195.2  
 DW 124.800 usec  
 DE 6.00 usec  
 KE 30.00 K  
 CASST2 145.00000000 sec  
 d0 0.000000000 sec  
 d1 1.000000000 sec  
 d2 0.00344828 sec  
 d42 0.00002000 sec  
 d12 0.00000400 sec  
 d13 0.00000000 sec  
 d20 0.00242428 sec  
 INO 0.00002339 sec  
 MCREST 0.00000000 sec  
 MCWRR 1.00000000 sec

CHANNEL F1 <sup>1</sup>H  
 P1 13.00 usec  
 P2 25.00 usec  
 P11 3.00 dB  
 SFO1 500.1320005 MHz

CHANNEL F2 <sup>13</sup>C  
 CPDPRG2 garp  
 NUCC 13C  
 P3 8.00 usec  
 PCPD2 60.00 usec  
 P12 3.00 dB  
 P14 2.00 dB  
 SFO2 125.7687784 MHz

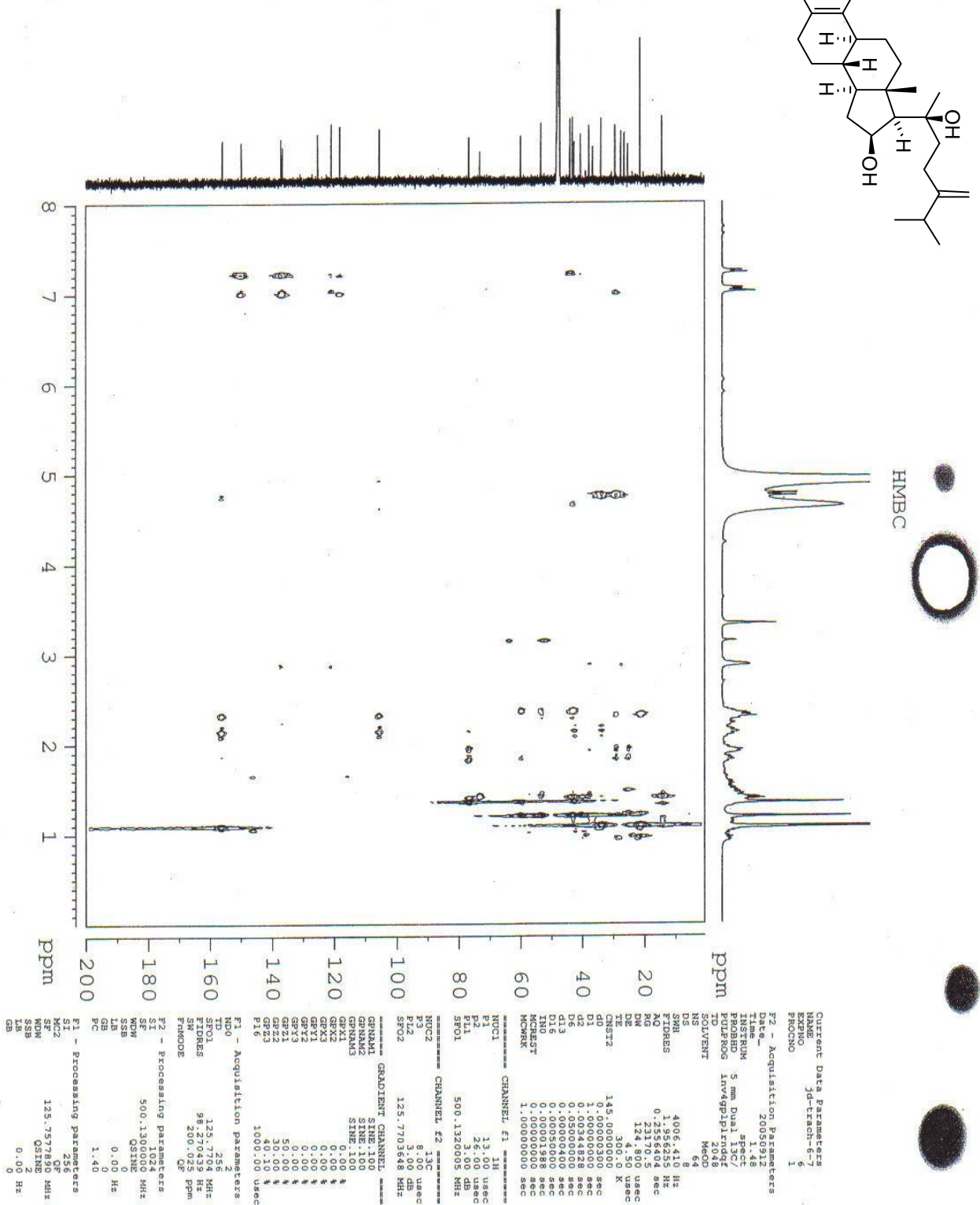
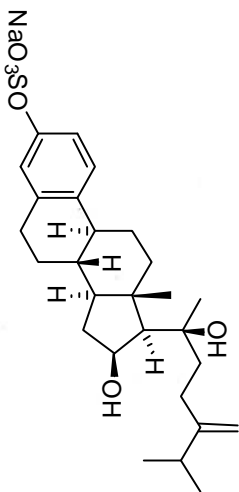
GRABNT CHANNEL  
 GENM1 SINE:100  
 GENM2 SINE:122  
 GENM3 SINE:100  
 GENM4 SINE:100  
 GENM5 SINE:100  
 GENM6 SINE:100  
 GENM7 SINE:100  
 GENM8 SINE:100  
 GENM9 SINE:100  
 GENM10 SINE:100  
 GENM11 SINE:100  
 GENM12 SINE:100  
 GENM13 SINE:100  
 GENM14 SINE:100  
 GENM15 SINE:100  
 GENM16 SINE:100  
 GENM17 SINE:100  
 GENM18 SINE:100  
 GENM19 SINE:100  
 GENM20 SINE:100  
 GENM21 SINE:100  
 GENM22 SINE:100  
 GENM23 SINE:100  
 GENM24 SINE:100

F1 - Acquisition parameters  
 NU0 100  
 SE01 125.7685 MHz  
 FIDRES 167.022980 Hz  
 SW 169.986 ppm  
 FURCODE QF

F2 - Processing parameters  
 SI 500.1300000 MHz  
 SINE SINE  
 WDW 0.00 Hz  
 SSB 0  
 LB 0  
 GB 1.40  
 PC 0

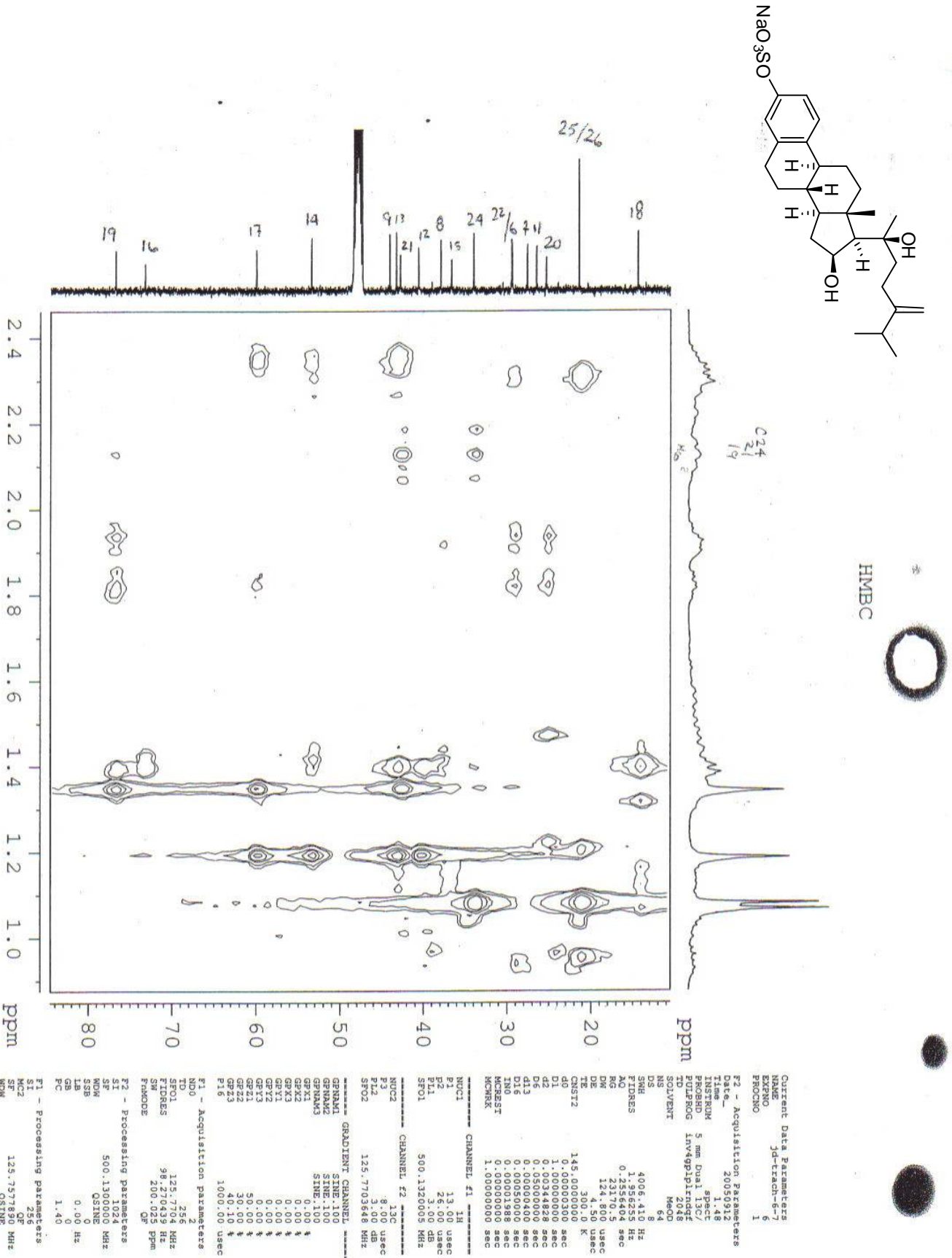
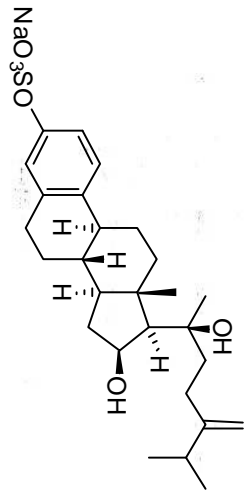
F1 - Processing parameters  
 SI 1024  
 KC2 125.7577890 MHz  
 SE SINE  
 SSB 0.00 Hz  
 GB 0

S22. 500 MHz HMBC spectrum of compound 2 in methanol-d4



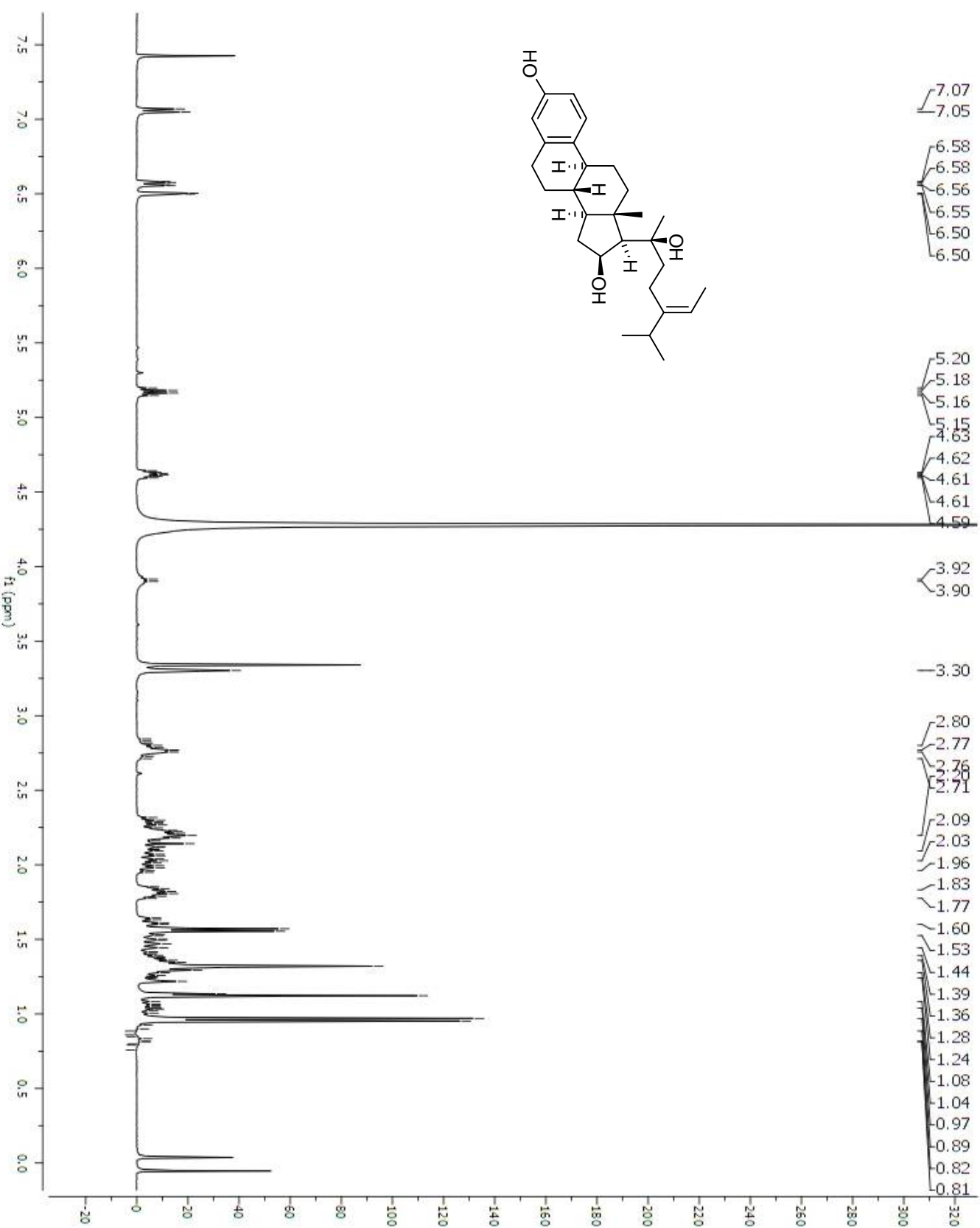
Current Data Parameters  
 Name: Jd-trach-6  
 ExpNO: 6  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_: 20060312  
 Time: 1.48  
 INSTRUM: spect  
 F1PR01: 5 mm Dual 1H/1  
 F2PR02: 500 MHz 13C/1  
 T0: 300 K  
 T1: 300 K  
 T2: 300 K  
 SOLVENT: MeOD  
 NS: 64  
 DS: 4  
 SWH: 4006.410 Hz  
 FIDRES: 1.956255 Hz  
 AQ: 0.2556404 sec  
 SFO1: 500.132005 MHz  
 DM: 124.800 usec  
 DE: 4.50 usec  
 TE: 300.0 K  
 D0: 0.00000000 sec  
 d1: 1.00000000 sec  
 d2: 0.00344828 sec  
 d3: 0.00000000 sec  
 d4: 0.00000000 sec  
 D16: 0.00050000 sec  
 INO: 0.00001988 sec  
 KCNST: 0.00000000 sec  
 NOMX: 1.00000000 sec  
 CHANNEL F1  
 NU01: 13 1H  
 P1: 13.00 usec  
 P2: 26.00 usec  
 PL1: 3.00 dB  
 SFO1: 500.132005 MHz  
 CHANNEL F2  
 NU02: 13C  
 P3: 8.00 usec  
 P4: 0.00 usec  
 SFO2: 125.7703648 MHz  
 GRAMP1: GRADIENT CHANNEL  
 SINE: 100  
 SINE: 100  
 SINE: 100  
 SINE: 100  
 GPMMS3  
 GRPX1: 0.00 %  
 GRPX2: 0.00 %  
 GRPX1: 0.00 %  
 GRPX2: 0.00 %  
 GRPX3: 0.00 %  
 GRPX4: 0.00 %  
 GRPX5: 0.00 %  
 GRPX6: 0.00 %  
 GRPX7: 0.00 %  
 GRPX8: 0.00 %  
 GRPX9: 0.00 %  
 GRPX10: 0.00 %  
 GRPX11: 0.00 %  
 GRPX12: 0.00 %  
 GRPX13: 0.00 %  
 GRPX14: 0.00 %  
 GRPX15: 0.00 %  
 GRPX16: 0.00 %  
 P16: 1000.00 usec  
 F1 - Acquisition Parameters  
 ND0  
 TD: 3256  
 SFO1: 500.132005 MHz  
 SFO2: 125.7703648 MHz  
 SW: 200.025 ppm  
 FWHM: 0.00 Hz  
 QF  
 F2 - Processing Parameters  
 SI: 1024  
 SF: 500.1300000 MHz  
 WDW: QSINE  
 LB: 0.00 Hz  
 GB: 0  
 PC: 1.40  
 F1 - Processing Parameters  
 SI: 256  
 MC2: 125.757703648 MHz  
 SFO1: 500.1300000 MHz  
 WDW: QSINE  
 LB: 0.00 Hz  
 GB: 0

S23. 500 MHz HMBC spectrum of compound 2 in methanol-d<sub>4</sub> (expansion)

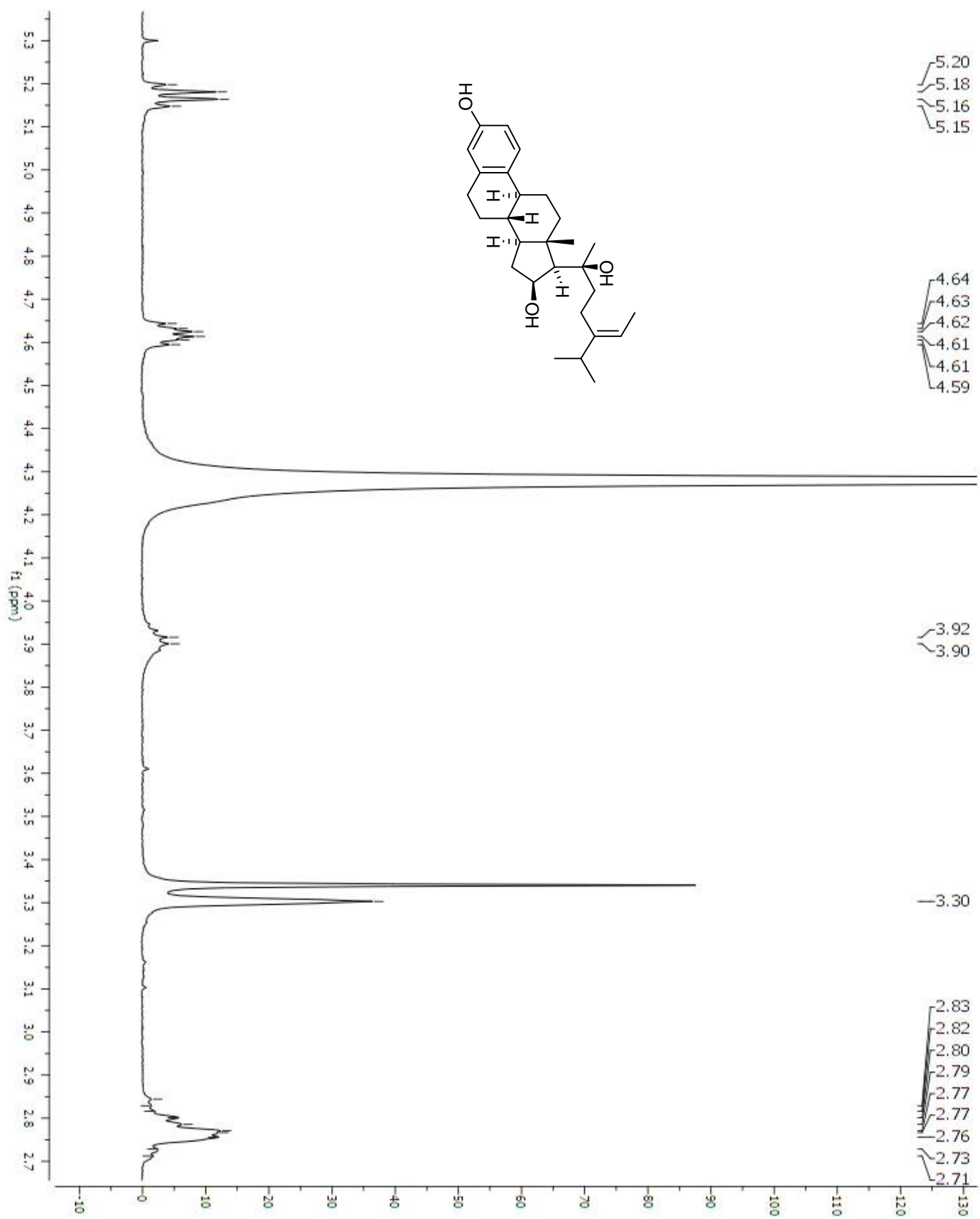




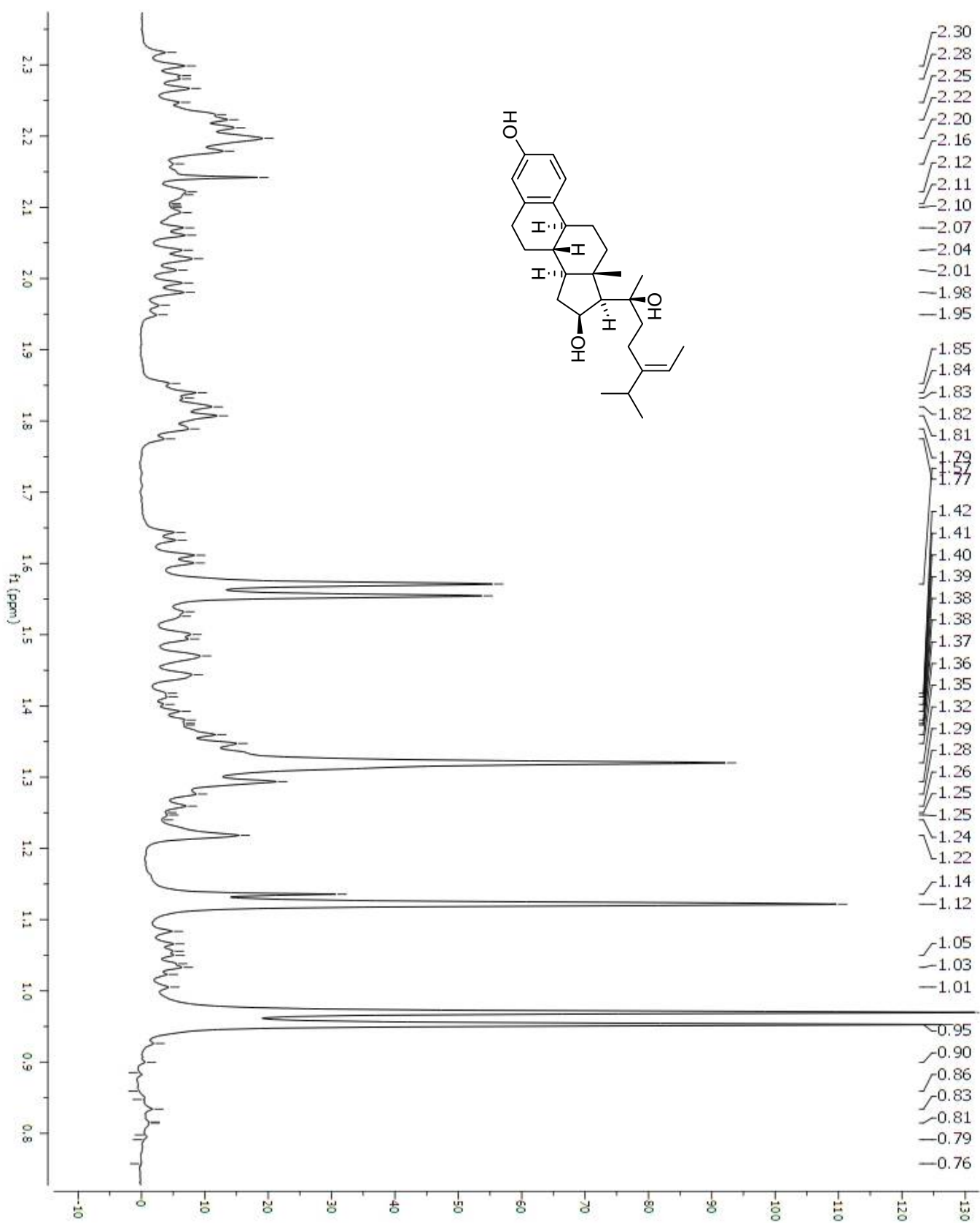
S24. 400 MHz <sup>1</sup>H NMR spectrum of compound **3** in 2:1 CDCl<sub>3</sub>:methanol-d<sub>4</sub>



S25: 400 MHz  $^1\text{H}$  NMR spectrum of compound **3** in 2:1  $\text{CDCl}_3$ :methanol- $d_4$  (expansion)



S26. 400 MHz <sup>1</sup>H NMR spectrum of compound 3 in 2:1 CDCl<sub>3</sub>:methanol-d<sub>4</sub> (expansion)



S27. 100 MHz <sup>13</sup>C NMR spectrum of compound 3 in 2:1 CDCl<sub>3</sub>:methanol-d<sub>4</sub>

