

NAME \_\_\_\_\_

**Chem 203**

**Organic Spectroscopy**

Midterm Examination, Part II (60 points total)

Problem 1 of 4 (three out of four required, 20 points)

Saturday, November 10, 2012, 9 am - ???

**SUBMIT THREE OF THE FOUR PROBLEMS FOR GRADING AND DO NOT SUBMIT THE PROBLEM THAT YOU DO NOT WANT GRADED. IF FOUR PROBLEMS ARE SUBMITTED, ONLY THE FIRST THREE (PROBLEMS 1, 2, AND 3) WILL BE GRADED**

Books, notes, calculators, rulers, and laptop computers are permitted as is wireless (or wired) internet access and appropriate software (e.g., PyMOL, Maestro/MacroModel, Excel, ChemDoodle, Chemdraw, ElComp, MolE, etc.). Communication with other students by e-mail, text, or in person is not permitted. Catalogs of molecular structures (e.g., the Aldrich catalog, the Merck Index, etc.) or databases of molecular structures (such as wireless access to SciFinder Scholar, the Sigma-Aldrich website, etc.) are NOT PERMITTED. INAPPROPRIATE COMMUNICATION OR USE OF SUCH ITEMS CONSTITUTES ACADEMIC DISHONESTY, WILL RESULT IN A FAILING GRADE (F) IN THE CLASS, AND MAY RESULT IN EXPULSION FROM THE Ph.D. PROGRAM.

If you wish to use a laptop computer, please be willing to share briefly with others when needed.

1. Analyze the spectra and solve the structure of the molecule for which data are provided.

Identify any noteworthy heteroatoms present. Determine the molecular formula and unsaturation number. Identify functional groups that are present from the IR and other spectra. Identify key fragments from NMR. Assign the  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR resonances to the respective atoms in the molecules. Mass spectra are EIMS, unless otherwise indicated.

ONLY WORK SHOWN ON THIS PAGE WILL BE GRADED.

Exact Mass: 192.1150

Noteworthy Heteroatoms:

Molecular Formula:

Unsaturation Number:

Functional Groups (be as specific as possible):

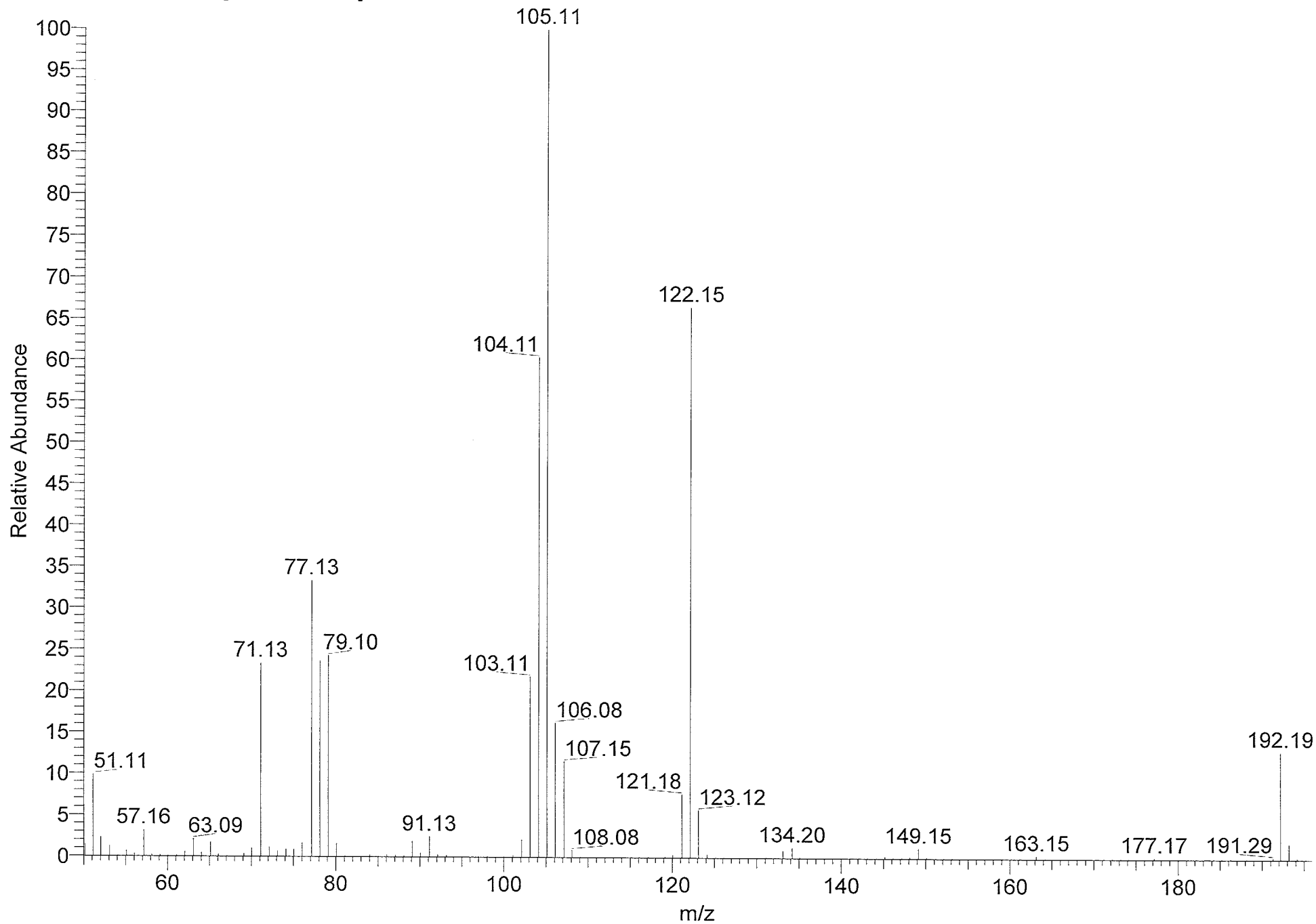
Fragments (from NMR):

Structure (Make sure to properly indicate stereochemistry, if applicable):

Structure with  $^1\text{H}$  NMR resonances lettered from the most downfield to the most upfield (a, b, c, d, etc.): (Note: Not all resonances can be assigned with certainty. If assignments are uncertain, indicate so by showing possible letters.)

Structure with  $^{13}\text{C}$  NMR resonances numbered from the most downfield to the most upfield (1, 2, 3, 4, etc.): (Note: Not all resonances can be assigned with certainty. If assignments are uncertain, indicate so by showing possible numbers.)

RKS\_1 #2411-2630 RT: 10.70-11.44 AV: 220 NL: 4.19E8  
T: {0,0} + c EI Full ms [50.00-600.00]

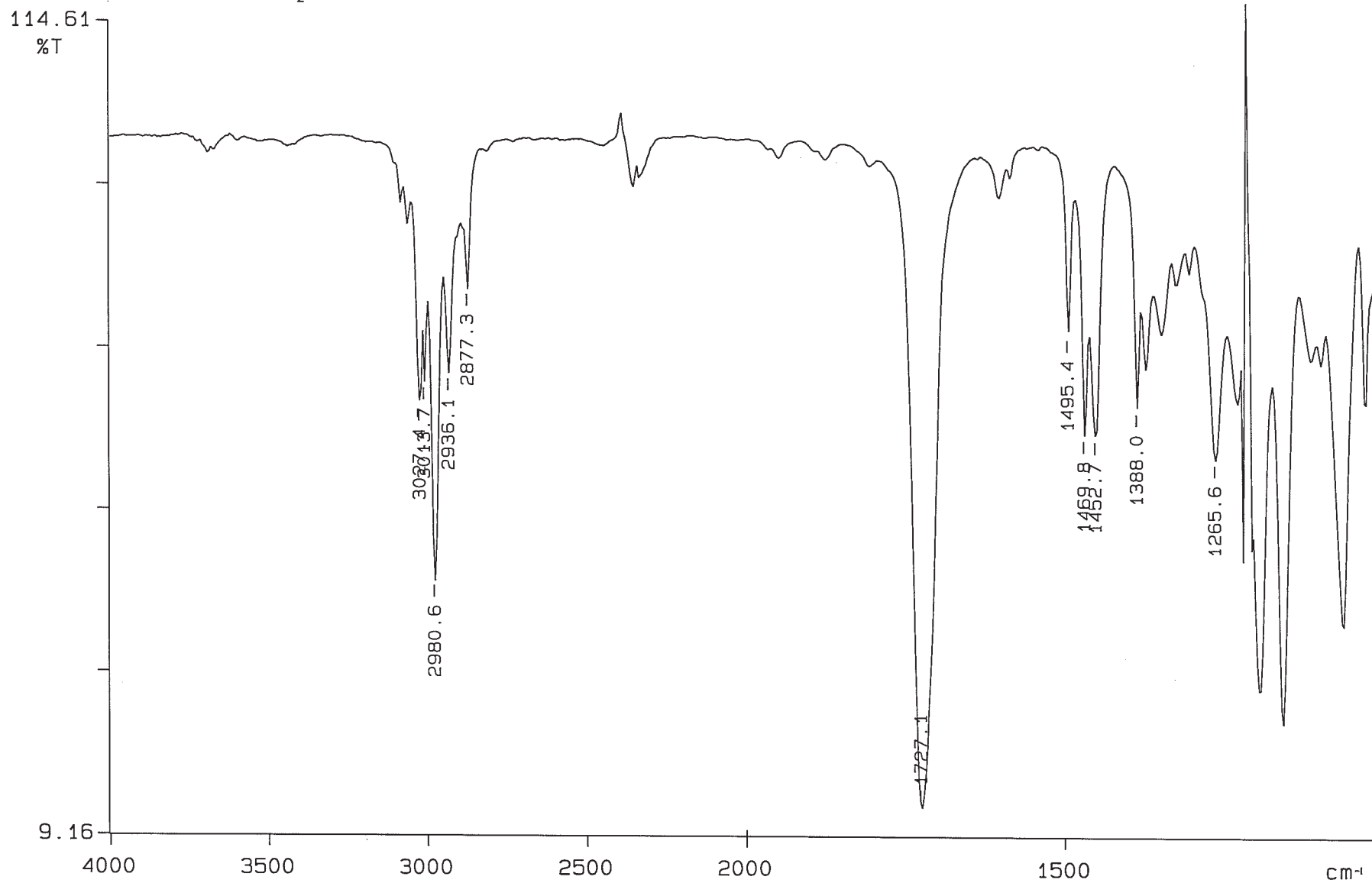


PERKIN ELMER

#1

5% solution in CHCl<sub>3</sub>

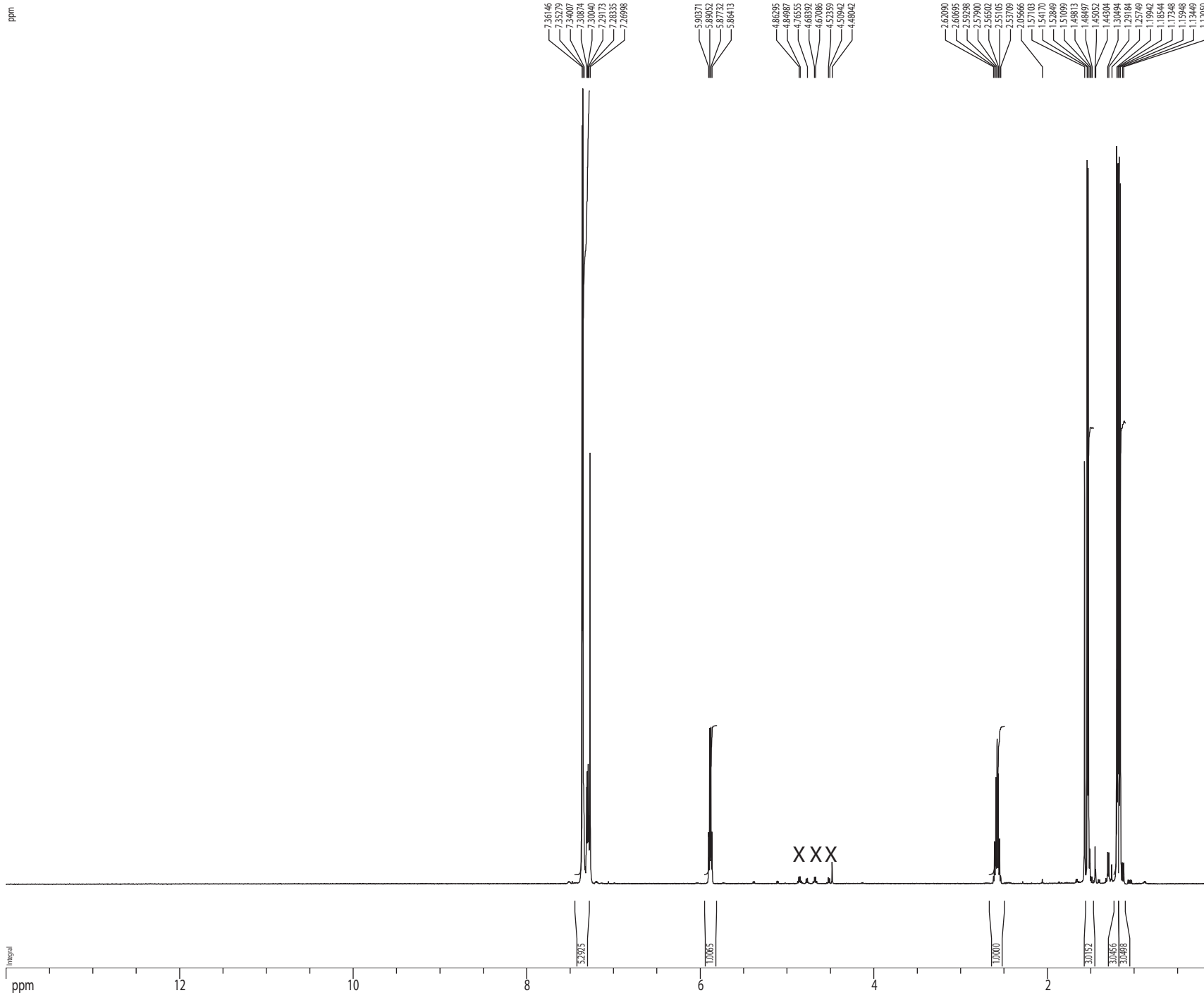
CaF<sub>2</sub> cell



12/10/31 13:06

X: 4 scans, 4.0cm<sup>-1</sup>

500 MHz <sup>1</sup>H NMR Spectrum in CDCl<sub>3</sub>



```

Current Data Parameters
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NAME      Mid_1_500_CDCL3
EXPNO     1
PROCNO    1

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Time      9.53
INSTRUM   cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG   zg30
TD         81728
SOLVENT   CDCl3
NS         32
DS         2
SWH        8012.820 Hz
FIDRES     0.098043 Hz
AQ         5.0998774 sec
RG         9
DW         62.400 usec
DE         6.00 usec
TE         298.0 K
D1         0.10000000 sec
MCREST     0.00000000 sec
MCWRK     0.01500000 sec

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P1        7.50 usec
PL1       1.60 dB
SFO1     500.2235015 MHz

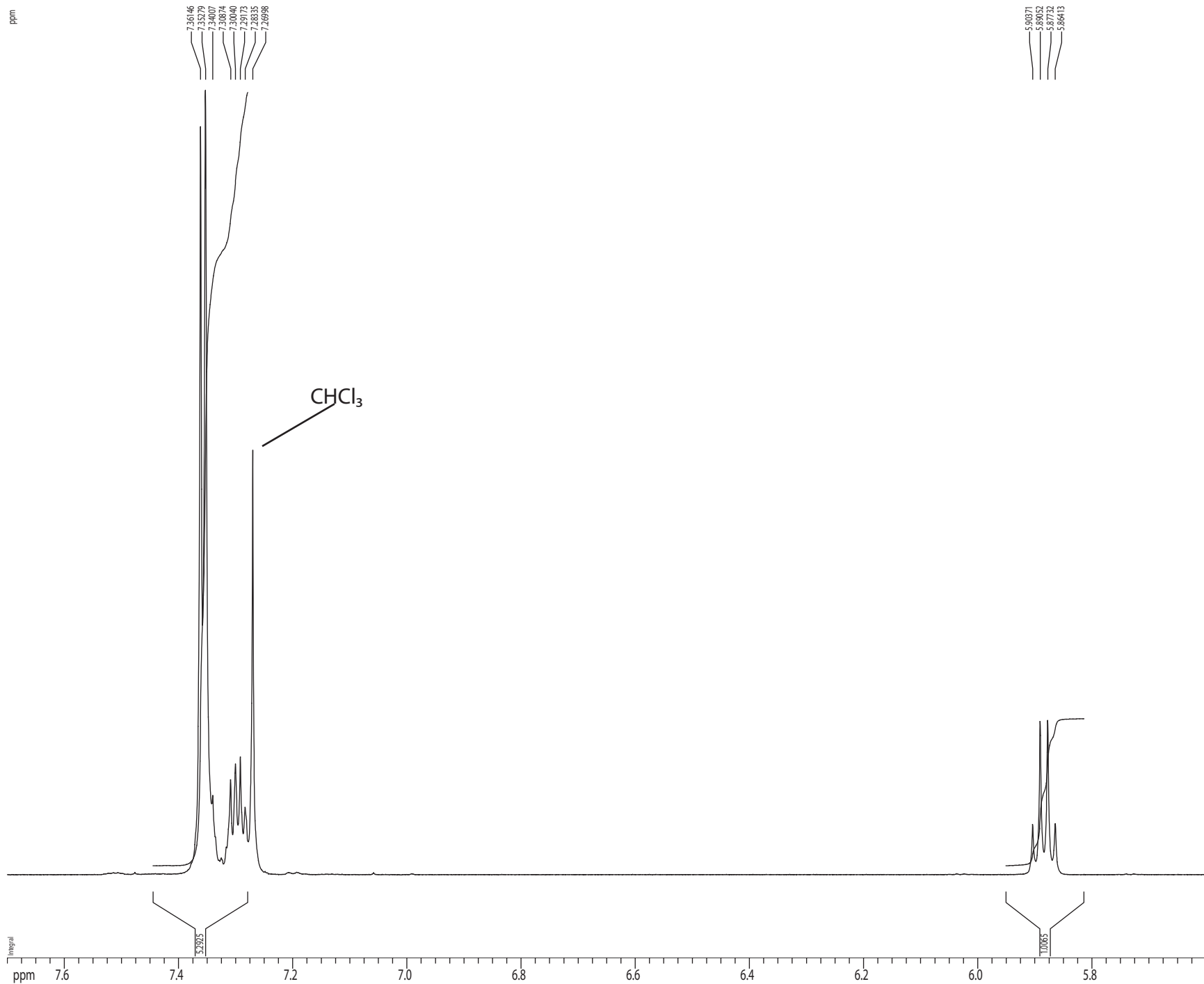
F2 - Processing parameters
SI         65536
SF         500.2200257 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         2.50

1D NMR plot parameters
CX         22.80 cm
CY         15.00 cm
F1P        14.000 ppm
F1         7003.08 Hz
F2P         0.000 ppm
F2          0.00 Hz
PPMCM      0.61404 ppm/cm
HZCM       307.15265 Hz/cm
    
```

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| #  | ADDRESS | FREQUENCY | INTENSITY    |
|----|---------|-----------|--------------|
|    | [Hz]    | [PPM]     |              |
| 1  | 54768.8 | 785.862   | 1.5710 8.63  |
| 2  | 54888.8 | 771.191   | 1.5417 14.72 |
| 3  | 54942.9 | 764.581   | 1.5285 14.55 |
| 4  | 55014.5 | 755.826   | 1.5110 0.80  |
| 5  | 55067.1 | 749.393   | 1.4981 0.24  |
| 6  | 55121.0 | 742.809   | 1.4850 0.23  |
| 7  | 55261.9 | 725.578   | 1.4505 0.83  |
| 8  | 55292.5 | 721.838   | 1.4430 0.39  |
| 9  | 55857.5 | 652.758   | 1.3049 0.73  |
| 10 | 55911.1 | 646.206   | 1.2918 0.72  |
| 11 | 56051.6 | 629.021   | 1.2575 0.48  |
| 12 | 56289.2 | 599.976   | 1.1994 15.00 |
| 13 | 56346.4 | 592.983   | 1.1854 14.70 |
| 14 | 56395.3 | 586.996   | 1.1735 14.82 |
| 15 | 56452.6 | 579.992   | 1.1595 14.30 |
| 16 | 56554.8 | 567.496   | 1.1345 0.52  |
| 17 | 56612.1 | 560.498   | 1.1205 0.50  |

1H spectrum



Current Data Parameters  
 USER nmr12t  
 NAME Mid\_1\_500\_CDCL3  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20121101  
 Time 9.53  
 INSTRUM cryo500  
 PROBHD 5 mm CPTCI 1H-  
 PULPROG zg30  
 TD 81728  
 SOLVENT CDCl3  
 NS 32  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.098043 Hz  
 AQ 5.0998774 sec  
 RG 9  
 DW 62.400 usec  
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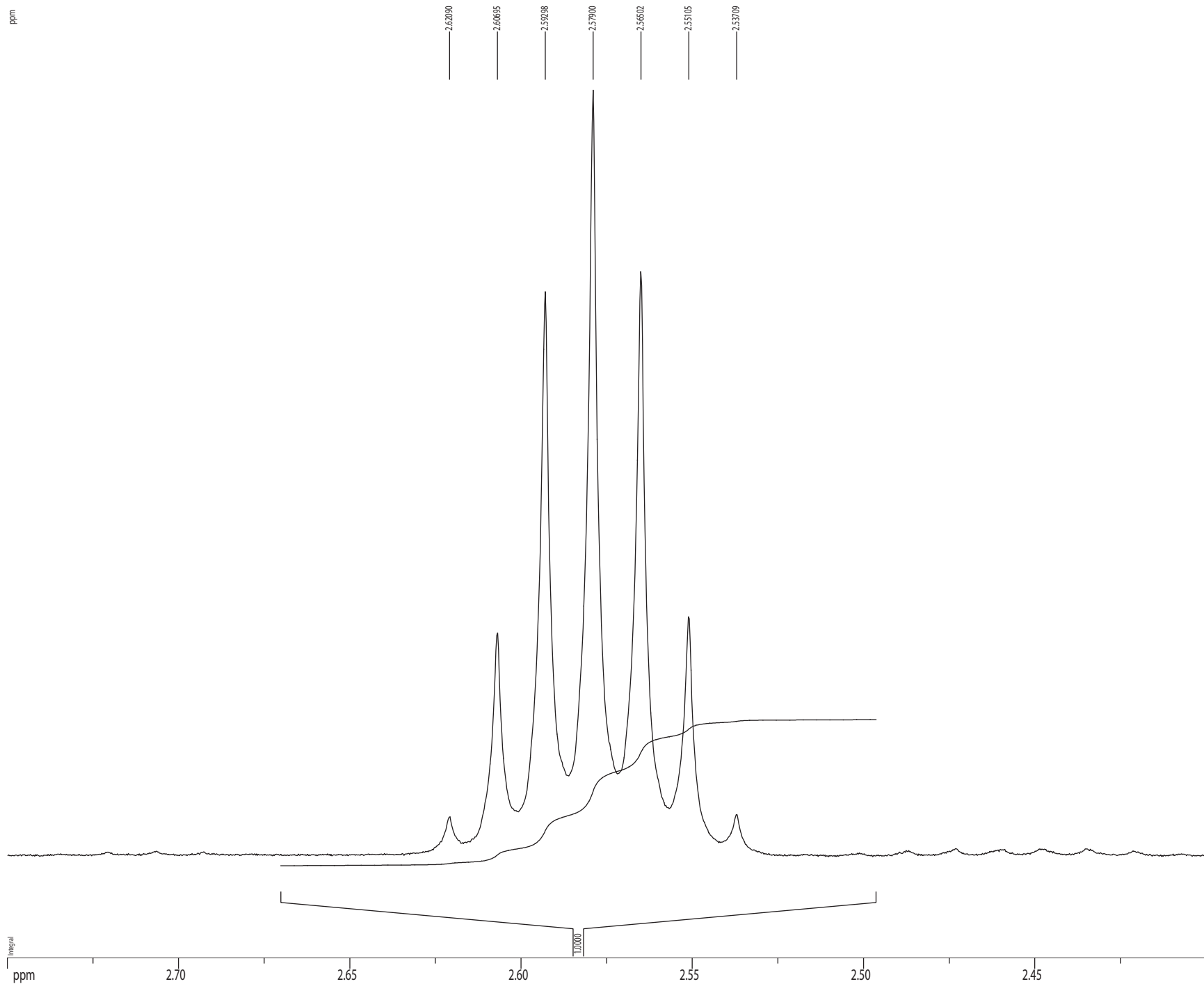
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 P1 7.50 usec  
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F2 - Processing parameters  
 SI 65536  
 SF 500.2200257 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 2.50

1D NMR plot parameters  
 CX 22.80 cm  
 CY 15.00 cm  
 F1P 7.700 ppm  
 F1 3851.69 Hz  
 F2P 5.600 ppm  
 F2 2801.23 Hz  
 PPMCM 0.09211 ppm/cm  
 HZCM 46.07290 Hz/cm

# <sup>1</sup>H spectrum

ppm



Current Data Parameters  
USER nmr12t  
NAME Mid\_1\_500\_CDCL3  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20121101  
Time 9.53  
INSTRUM cryo500  
PROBHD 5 mm CPTCI 1H-  
PULPROG zg30  
TD 81728  
SOLVENT CDCl3  
NS 32  
DS 2  
SWH 8012820 Hz  
FIDRES 0.098043 Hz  
AQ 5.0998774 sec  
RG 9  
DW 62.400 usec  
DE 6.00 usec  
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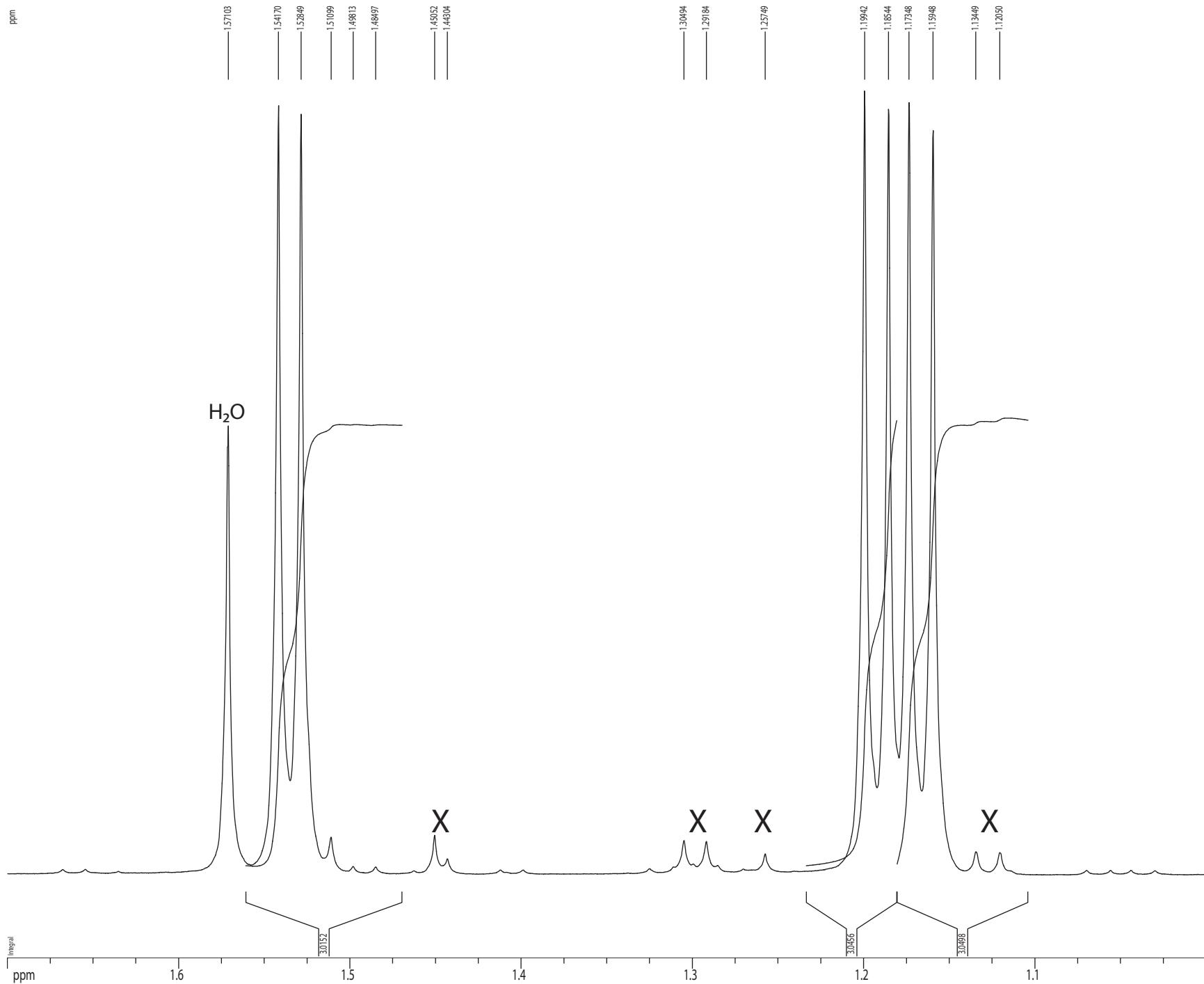
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PL1 1.60 dB  
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F2 - Processing parameters  
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SF 500.2200257 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 2.50

1D NMR plot parameters  
CX 22.80 cm  
CY 15.00 cm  
F1P 2.750 ppm  
F1 1375.60 Hz  
F2P 2.400 ppm  
F2 1200.53 Hz  
PPMCM 0.01535 ppm/cm  
HZCM 7.67881 Hz/cm



# 1H spectrum



Current Data Parameters  
 USER nmr12t  
 NAME Mid\_1\_500\_CDCL3  
 EXPNO 1  
 PROCNO 1

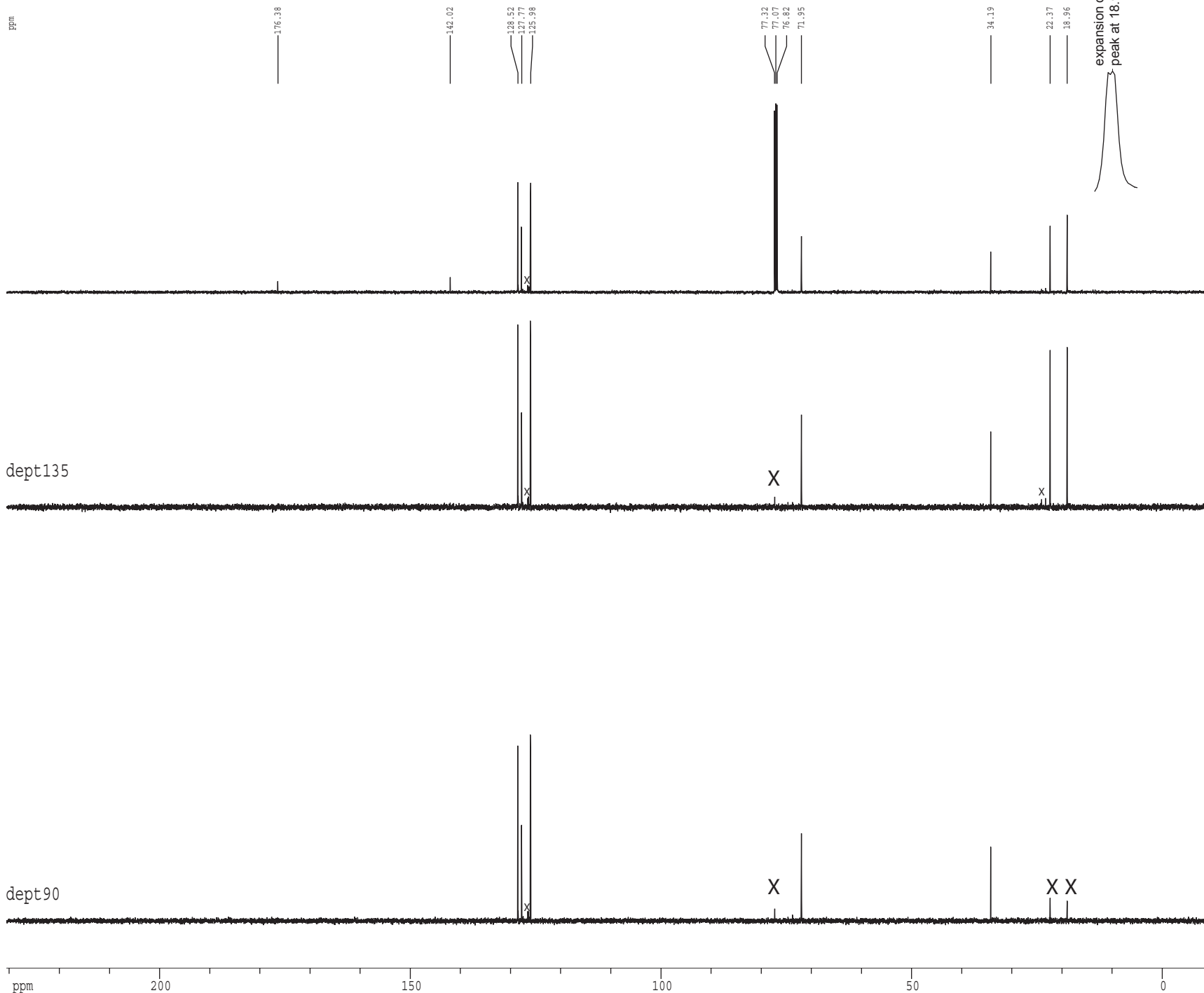
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 Time 9.53  
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 PULPROG zg30  
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 SOLVENT CDCl3  
 NS 32  
 DS 2  
 SWH 8012820 Hz  
 FIDRES 0.098043 Hz  
 AQ 5.0998774 sec  
 RG 9  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 MCREST 0.00000000 sec  
 MCWRK 0.01500000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.50 usec  
 PL1 1.60 dB  
 SFO1 500.2235015 MHz

F2 - Processing parameters  
 SI 65536  
 SF 500.2200257 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 2.50

1D NMR plot parameters  
 CX 22.80 cm  
 CY 15.00 cm  
 F1P 1.700 ppm  
 F1 850.37 Hz  
 F2P 1.000 ppm  
 F2 500.22 Hz  
 PPMCM 0.03070 ppm/cm  
 HZCM 15.35763 Hz/cm

125.8 MHz <sup>13</sup>C NMR spectrum with <sup>1</sup>H decoupling in CDCl<sub>3</sub>



Current Data Parameters  
 USER nmr12t  
 NAME Mid\_1\_500\_CDCl3  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20121101  
 Time\_ 10.02  
 INSTRUM cryos500  
 PROBHD 5 mm CPTCI 1H-  
 PULPROG SpinEchopp30gp.prd  
 TD 65536  
 SOLVENT CDCl3  
 NS 500  
 DS 16  
 SWH 30303.031 Hz  
 FIDRES 0.462388 Hz  
 AQ 1.0813940 sec  
 RG 4096  
 DW 16.500 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.25000000 sec  
 d11 0.03000000 sec  
 D16 0.00020000 sec  
 d17 0.00019600 sec  
 MCREST 0.00000000 sec  
 MCWRR 0.01500000 sec  
 P2 31.00 usec

===== CHANNEL f1 =====  
 NUC1 <sup>13</sup>C  
 P1 15.50 usec  
 P11 500.00 usec  
 P12 2000.00 usec  
 PL0 120.00 dB  
 PL1 -1.00 dB  
 SFO1 125.7942548 MHz  
 SF1 3.20 dB  
 SF2 3.20 dB  
 SFOAM1 Crp60,0.5,20.1  
 SFOAM2 Crp60comp,4  
 SFOFF1 0.00 Hz  
 SFOFF2 0.00 Hz

===== CHANNEL F2 =====  
 CPDPRG2 waltz16  
 NUC2 <sup>1</sup>H  
 PCPD2 100.00 usec  
 PL2 1.60 dB  
 PL12 24.60 dB  
 SFO2 500.2225011 MHz

===== GRADIENT CHANNEL =====  
 GENAM1 SINE.100  
 GENAM2 SINE.100  
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 GFX2 0.00 %  
 GPY1 0.00 %  
 GPY2 0.00 %  
 GPZ1 30.00 %  
 GPZ2 50.00 %  
 p15 500.00 usec  
 p16 1000.00 usec

F2 - Processing parameters  
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 SF 125.7804190 MHz  
 WMW HM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 4.00

ID NMR plot parameters  
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 CY 3.56 cm  
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 F1 29009.68 Hz  
 F2P -10.287 ppm  
 F2 -1293.96 Hz  
 PPMCM 10.56688 ppm/cm  
 HZCM 1329.10706 Hz/cm