

NAME _____

Chem 203

Organic Spectroscopy

Midterm Examination, Part II (50 points total)

Problem 2 of 3 (two out of three required, 25 points)

Saturday, November 5, 2011, 9 am - ???

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

2. 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 ^1H NMR and ^{13}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: 241.0477, ESI, $[\text{M} + \text{Na}]^+$

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 ^1H 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.)

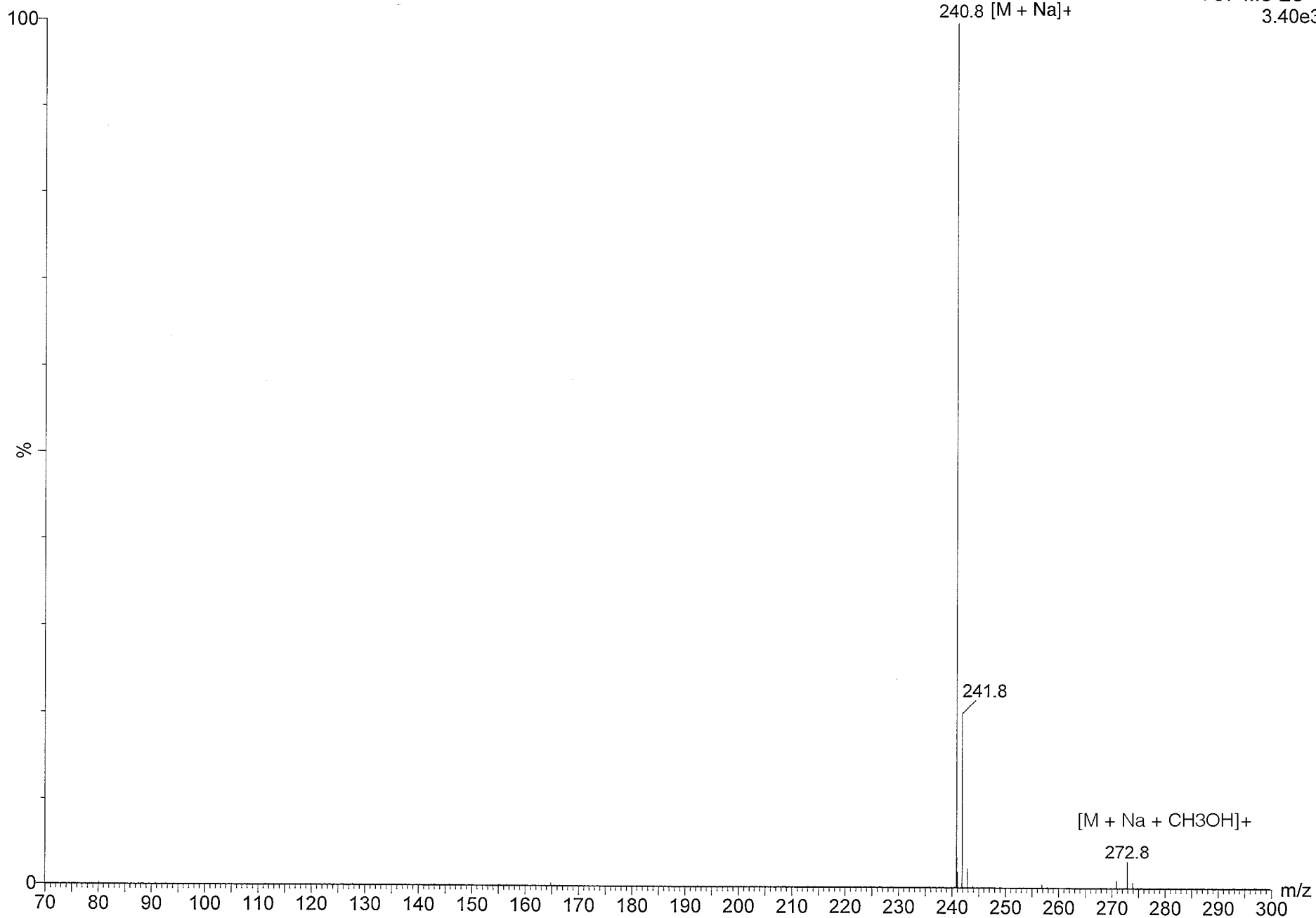
Structure with ^{13}C NMR resonances numbered from the most downfield to the most upfield (1, 2, 3, 4, etc.): (Note: Not all resonances can be assigned with certainty. If assignments are uncertain, indicate so by showing possible numbers.)

Molecular Model: Build an energy-minimized molecular model of the structure using PyMOL and the "clean" function. Save the .pse files as structure2.pse. E-mail the .pse file to me (jsnowick@uci.edu).

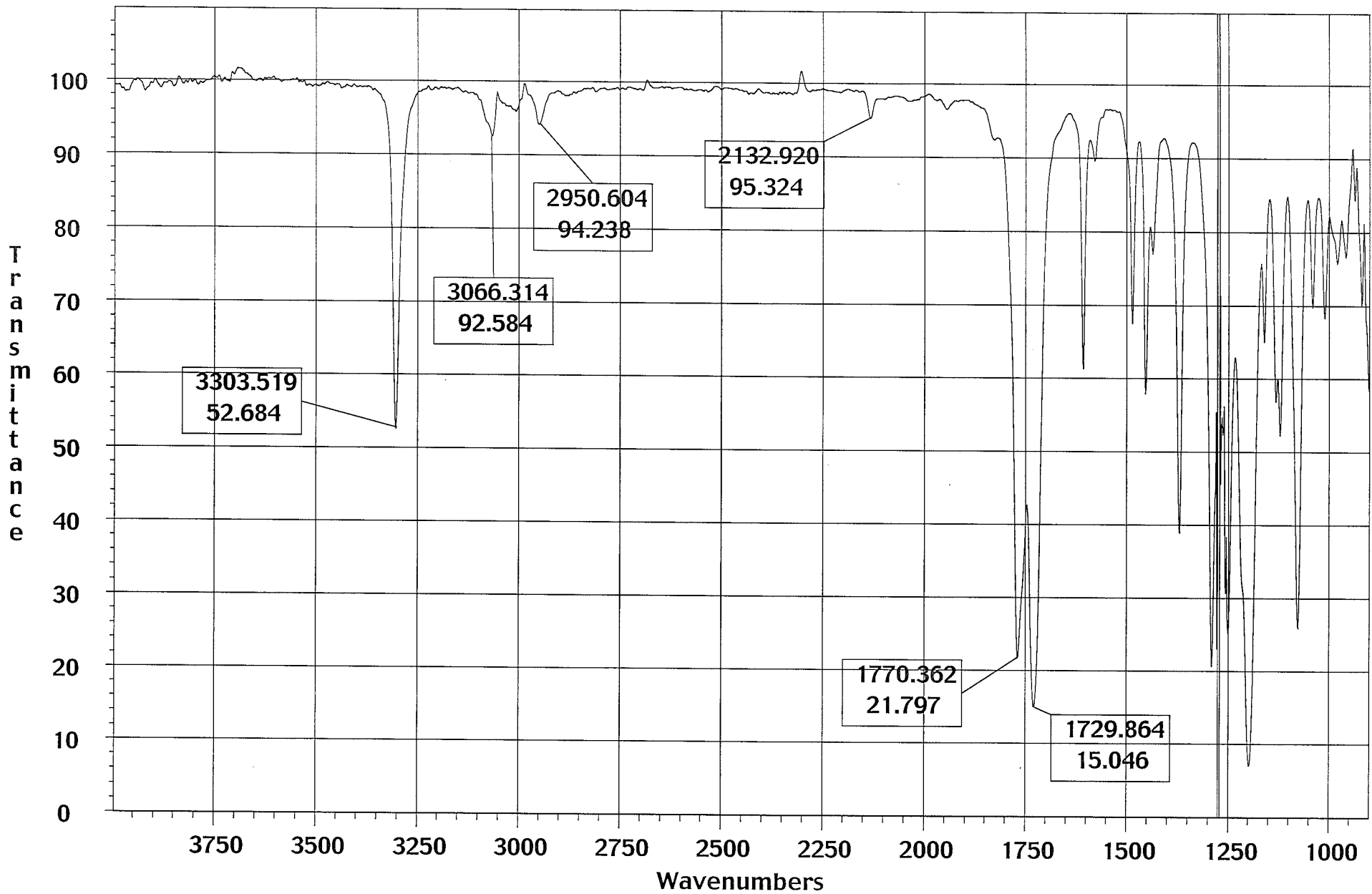
ESI MS

C2-1 19 (0.349)

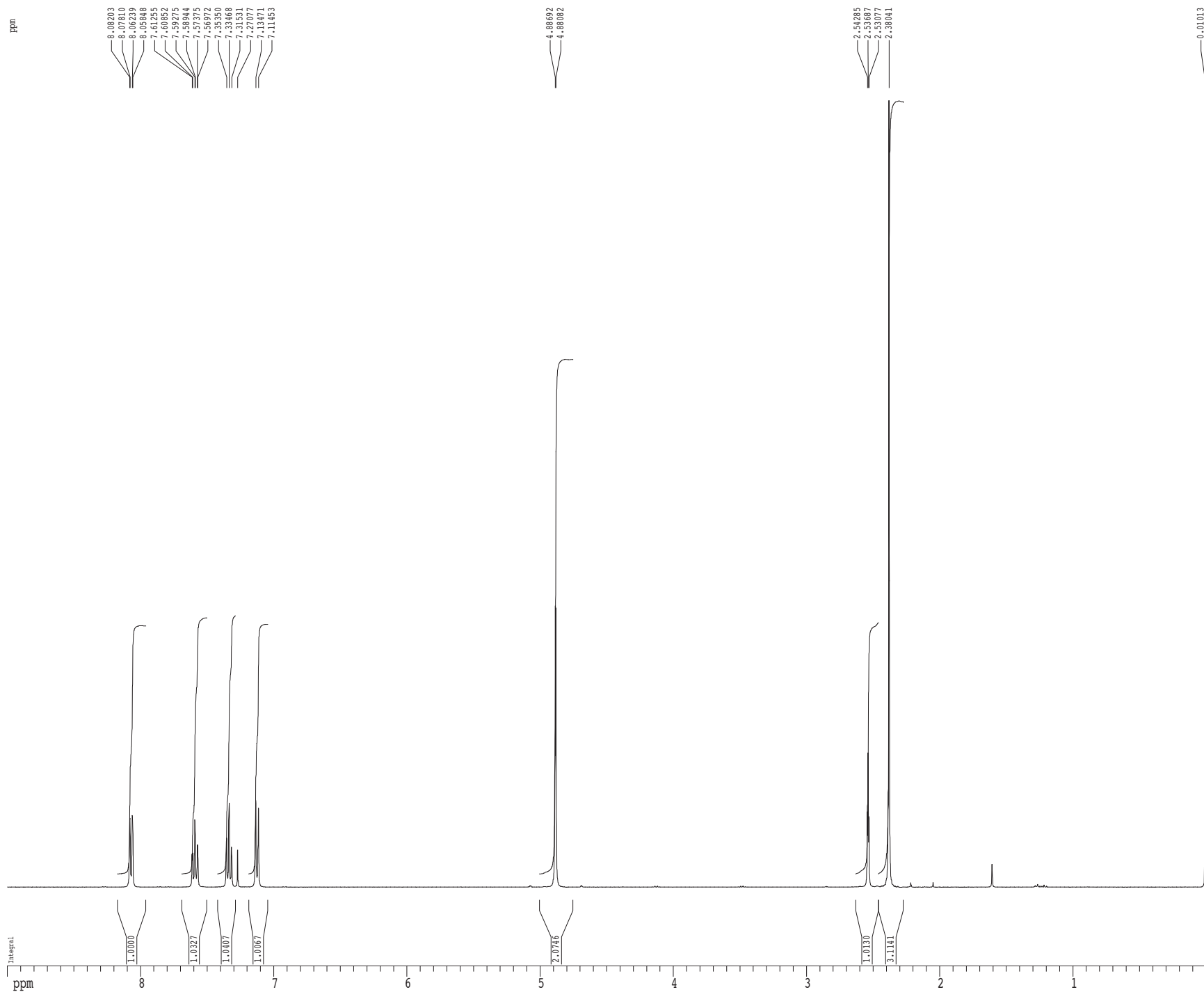
TOF MS ES+
3.40e3



5% Solution in CH₂Cl₂ in CaF₂ plates



400 MHz 1H spectrum in CDCl3



```

Current Data Parameters
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NAME          nmrl1t-X-sample4
EXPNO        3
PROCNO       1

F2 - Acquisition Parameters
Date_        20111018
Time         20.17
INSTRUM      drx400
PROBHD       5 mm QNP H/E/P
PULPROG      zg30
TD           65536
SOLVENT      CDCl3
NS           8
DS           2
SWH          6410.256 Hz
FIDRES       0.097813 Hz
AQ           5.1118579 sec
RG           203.2
DN           78.000 usec
DE           4.50 usec
TE           298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

===== CHANNEL f1 =====
NUC1          1H
P1            12.00 usec
PL1           -0.60 dB
SFO1         400.1328009 MHz

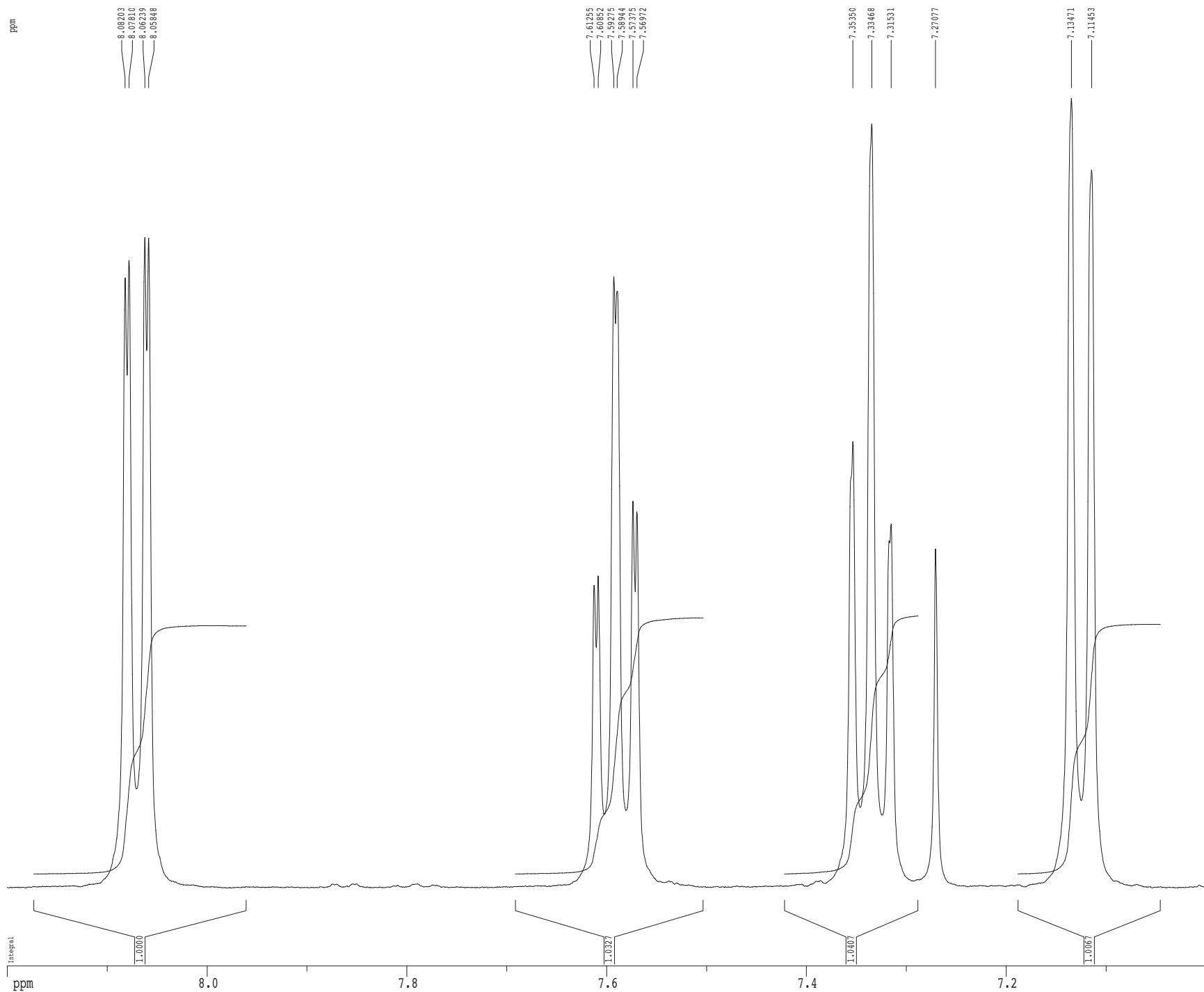
F2 - Processing parameters
SI            65536
SF           400.1300175 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            2.00

ID NMR plot parameters
CX            22.80 cm
CY            15.00 cm
FIP           9.000 ppm
F1            3601.17 Hz
F2P           0.000 ppm
F2            0.00 Hz
PPMCM         0.39474 ppm/cm
HZCM          157.94606 Hz/cm
    
```

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#	ADDRESS	FREQUENCY		INTENSITY
		[Hz]	[PPM]	
1	28162.5	3233.861	8.0820	1.28
2	28178.6	3232.292	8.0781	1.31
3	28242.8	3226.003	8.0624	1.36
4	28258.8	3224.439	8.0585	1.36
5	30083.0	3046.010	7.6126	0.63
6	30099.5	3044.397	7.6085	0.65
7	30164.0	3038.088	7.5928	1.28
8	30177.6	3036.764	7.5894	1.25
9	30241.7	3030.486	7.5738	0.81
10	30258.3	3028.870	7.5697	0.79
11	31142.8	2942.355	7.3535	0.93
12	31219.7	2934.824	7.3347	1.60
13	31299.0	2927.074	7.3153	0.76
14	31481.2	2909.254	7.2708	0.71
15	32037.8	2854.811	7.1347	1.65
16	32120.3	2846.738	7.1145	1.50
17	41233.0	1955.402	4.8869	5.35
18	41257.9	1952.964	4.8808	5.30
19	50822.0	1017.471	2.5429	1.44
20	50846.5	1015.078	2.5369	2.57
21	50871.5	1012.636	2.5308	1.35
22	51486.6	952.472	2.3804	15.00
23	61182.8	4.055	0.0101	0.73

1H spectrum



```

Current Data Parameters
USER          nmrlit
NAME          nmrlit-X-sample4
EXPNO        3
PROCNO       1

F2 - Acquisition Parameters
Date_        2011018
Time         20.17
INSTRUM     drx400
PROBHD      5 mm QNP H/E/P
PULPROG     zg30
TD          65536
SOLVENT     CDCl3
NS           8
DS           2
SWH         6410.256 Hz
FIDRES      0.097813 Hz
AQ          5.1118579 sec
RG           203.2
DN           78.000 usec
DE           4.50 usec
TE           298.0 K
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MCREST      0.00000000 sec
MCWRK       0.01500000 sec

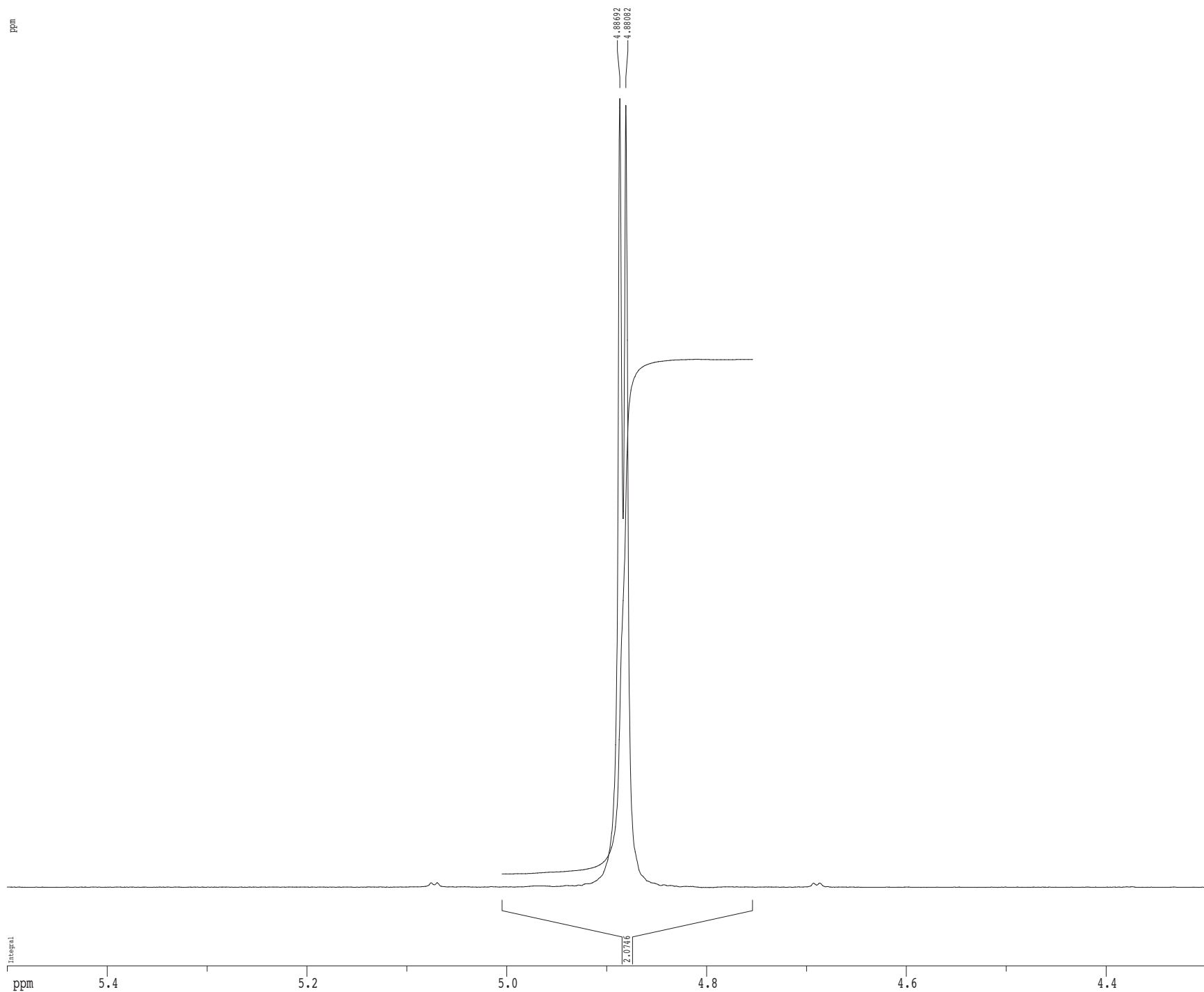
===== CHANNEL f1 =====
NUC1         1H
P1           12.00 usec
PL1          -0.60 dB
SFO1        400.1328009 MHz

F2 - Processing parameters
SI           65536
SF          400.1300175 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           2.00

ID NMR plot parameters
CX           22.80 cm
CY           15.00 cm
FIP          8.200 ppm
F1           3281.07 Hz
F2P          7.000 ppm
F2           2800.91 Hz
PPMCM       0.05263 ppm/cm
HZCM        21.05947 Hz/cm
    
```

1H spectrum

ppm



```
Current Data Parameters
USER          nmrl1t
NAME          nmrl1t-X-sample4
EXPNO         3
PROCNO        1

F2 - Acquisition Parameters
Date_         20111018
Time          20.17
INSTRUM       drx400
PROBHD        5 mm QNP H/E/P
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            8
DS            2
SWH           6410.256 Hz
FIDRES        0.097813 Hz
AQ            5.1118579 sec
RG            203.2
DN            78.000 usec
DE            4.50 usec
TE            298.0 K
D1            0.10000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

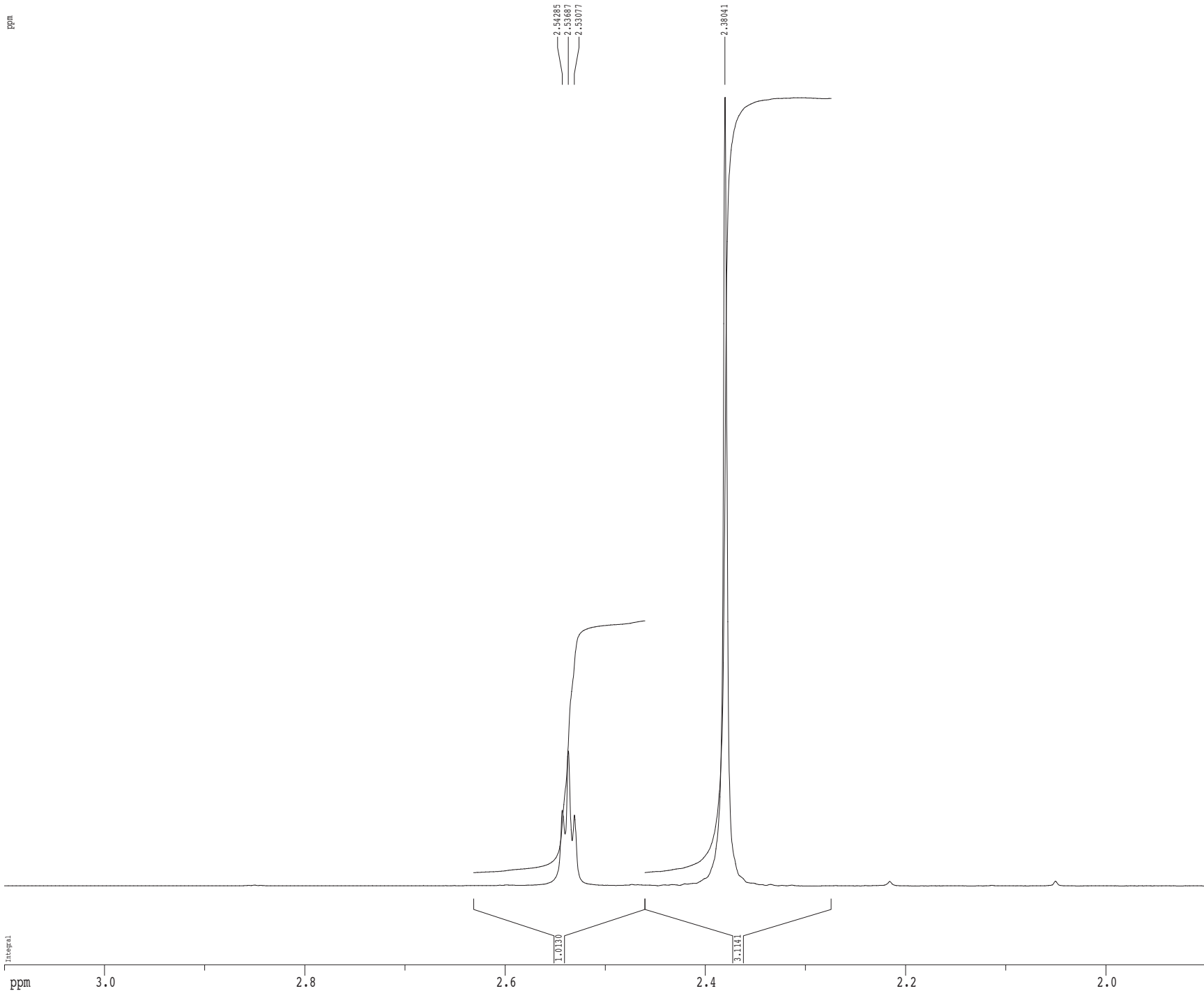
===== CHANNEL f1 =====
NUC1          1H
P1            12.00 usec
PL1           -0.60 dB
SFO1         400.1328009 MHz

F2 - Processing parameters
SI            65536
SF            400.1300175 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            2.00

1D NMR plot parameters
CX            22.80 cm
CY            15.00 cm
FIP           5.500 ppm
F1            2200.72 Hz
F2P           4.300 ppm
F2            1720.56 Hz
PPMCM         0.05263 ppm/cm
HZCM          21.05947 Hz/cm
```


1H spectrum

ppm



2.54285
2.53687
2.53077

2.38041

```
Current Data Parameters
USER          nmrl1t
NAME          nmrl1t-X-sample4
EXPNO        3
PROCNO       1

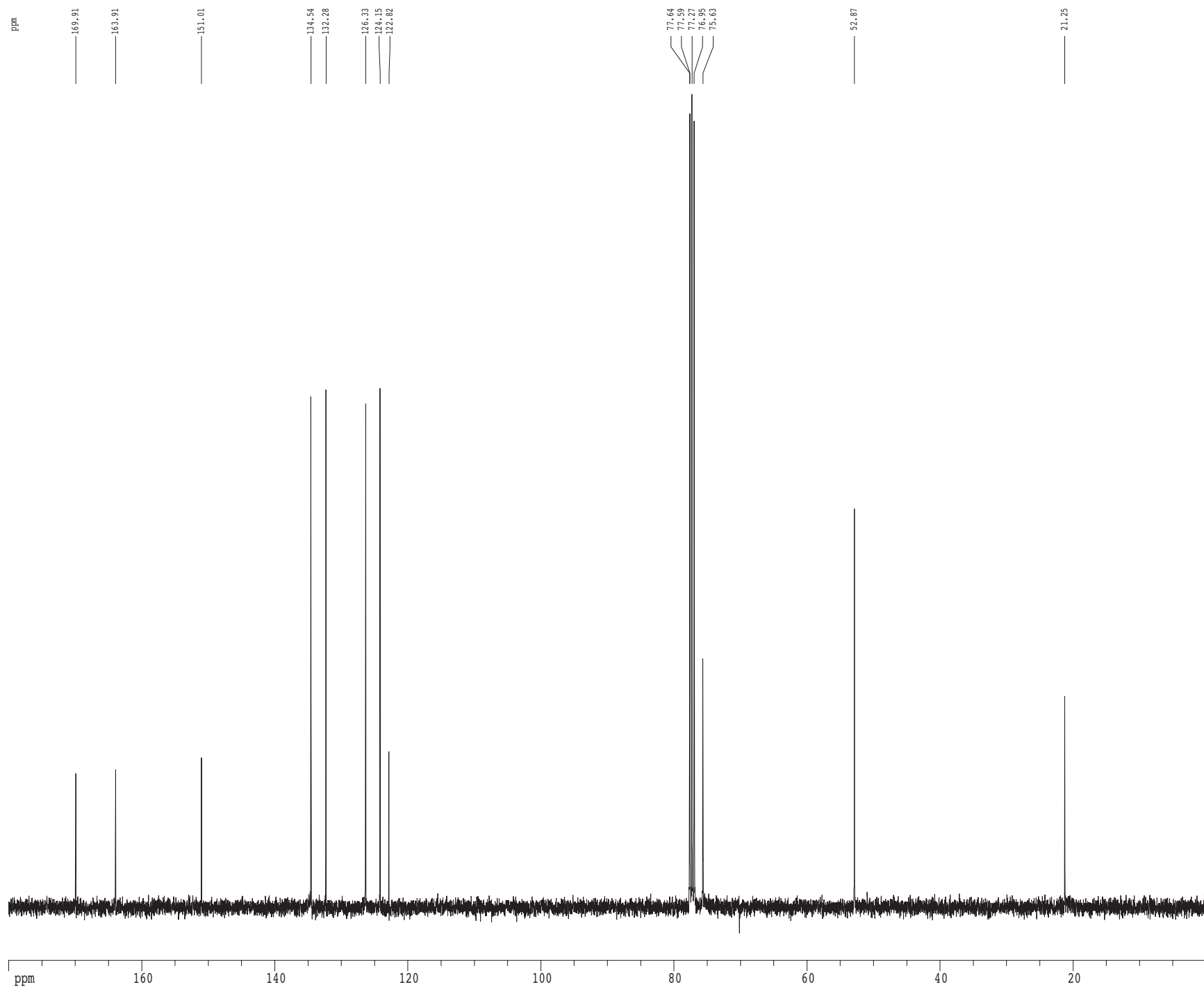
F2 - Acquisition Parameters
Date_        20111018
Time         20.17
INSTRUM      drx400
PROBHD       5 mm QNP H/E/P
PULPROG      zg30
TD           65536
SOLVENT      CDCl3
NS           8
DS           2
SWH          6410.256 Hz
FIDRES       0.097813 Hz
AQ           5.1118579 sec
RG           203.2
DN           78.000 usec
DE           4.50 usec
TE           298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

===== CHANNEL f1 =====
NUC1         1H
P1           12.00 usec
PL1          -0.60 dB
SFO1         400.1328009 MHz

F2 - Processing parameters
SI           65536
SF           400.1300175 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           2.00

1D NMR plot parameters
CX           22.80 cm
CY           15.00 cm
FIP          3.100 ppm
F1           1240.40 Hz
F2P          1.900 ppm
F2           760.25 Hz
PPMCM        0.05263 ppm/cm
HZCM         21.05947 Hz/cm
```

100.6 MHz ¹³C spectrum with ¹H decoupling in CDCl₃



```

Current Data Parameters
USER          nmrl1t
NAME          nmrl1t-X-sample4
EXPNO        4
PROCNO       1

F2 - Acquisition Parameters
Date_        2011018
Time         20.36
INSTRUM     drx400
PROBHD      5 mm QNP H/F/P
PULPROG     zgpg30
TD           65536
SOLVENT     CDCl3
NS           305
DS           4
SWH          24154.590 Hz
FIDRES       0.368570 Hz
AQ           1.3566452 sec
RG           9195.2
DW           20.700 usec
DE           20.39 usec
TE           298.1 K
D1           0.10000000 sec
d11          0.03000000 sec
MCREST      0.00000000 sec
MCMRK       0.01500000 sec

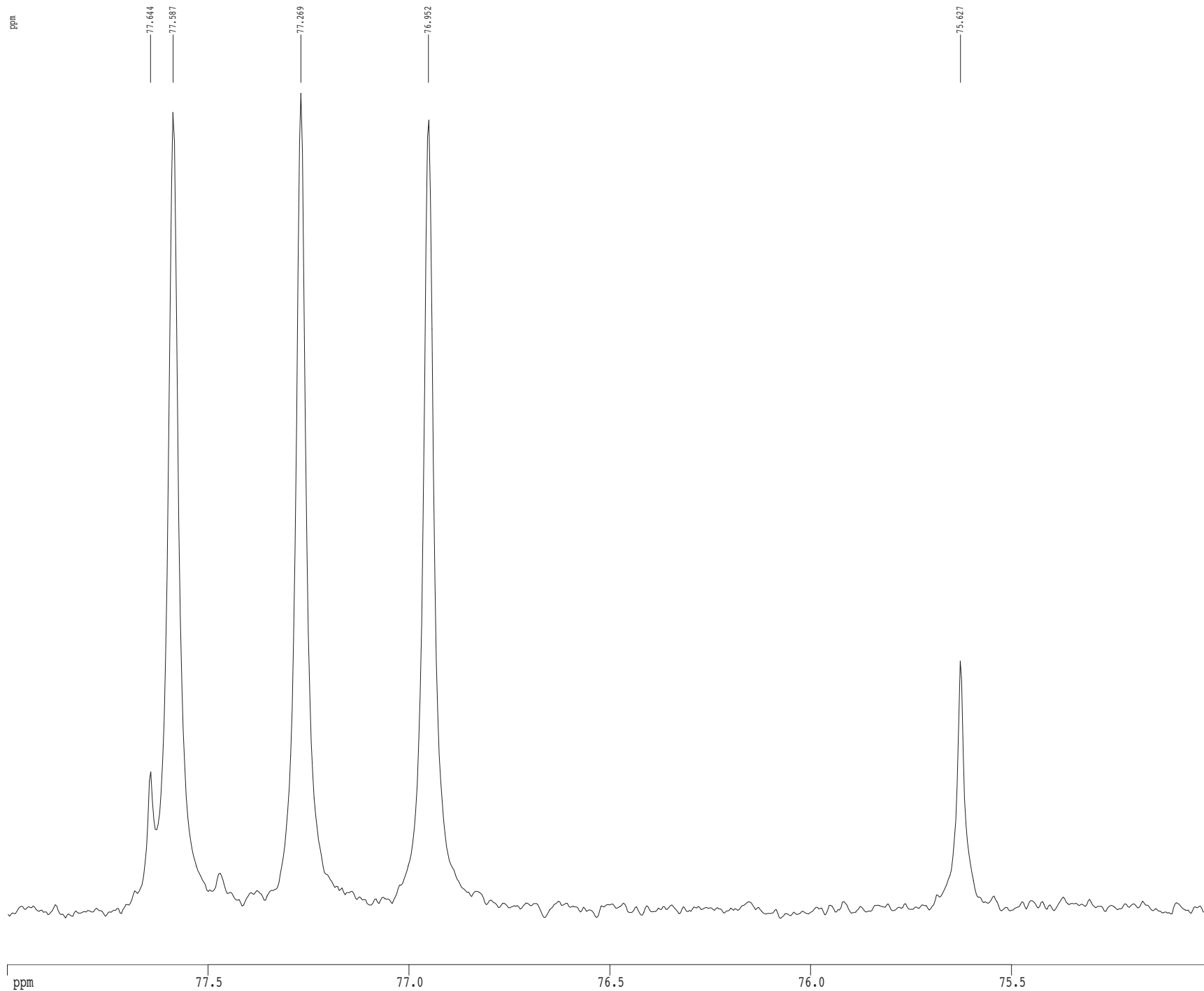
===== CHANNEL f1 =====
NUC1         13C
P1           11.00 usec
PL1          0.00 dB
SF01         100.6237964 MHz

===== CHANNEL f2 =====
CPDPRG2     mlev16
NUC2         1H
PCPD2       80.00 usec
PL2          0.00 dB
PL12         16.20 dB
SF02         400.1328009 MHz

F2 - Processing parameters
SI           65536
SF           100.6127500 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.00

1D NMR plot parameters
CX           22.80 cm
CY           15.50 cm
F1           180.000 ppm
F1           18110.29 Hz
F2           0.000 ppm
F2           0.00 Hz
PPMCM       7.89474 ppm/cm
HZCM        794.31122 Hz/cm
    
```

13C spectrum with 1H decoupling



```
Current Data Parameters
USER          nmrl1t
NAME          nmrl1t-X-sample4
EXPNO         4
PROCNO        1

F2 - Acquisition Parameters
Date_         20111018
Time_         20.36
INSTRUM       drx400
PROBHD        5 mm QNP H/F/P
PULPROG       zgdc30
TD             65536
SOLVENT       CDC13
NS             305
DS             4
SWH           24154.500 Hz
FIDRES        0.368570 Hz
AQ            1.3566452 sec
RG            9195.2
DW            20.700 usec
DE            20.39 usec
TE            298.1 K
D1            0.10000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCMRK         0.01500000 sec

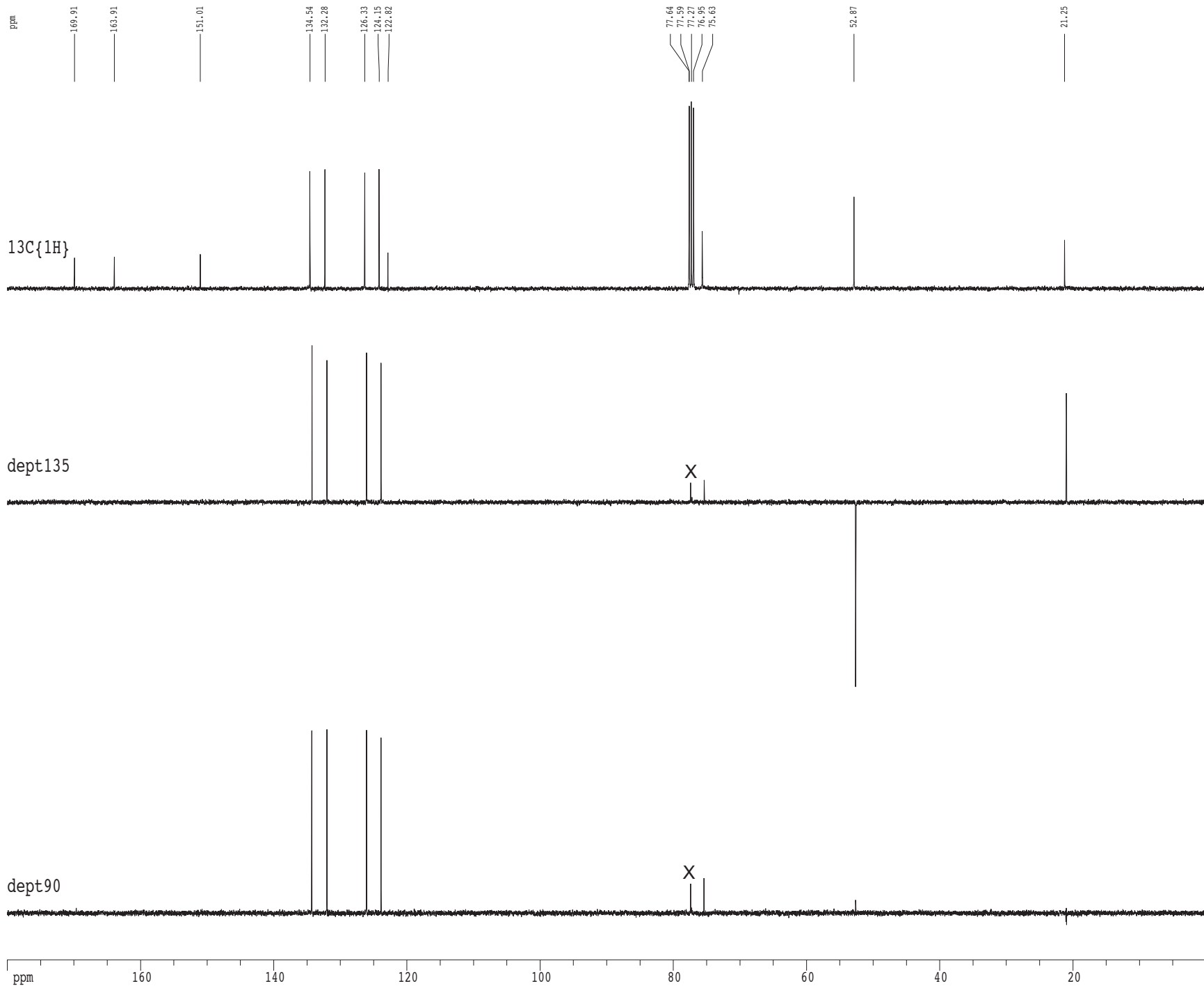
===== CHANNEL f1 =====
NUC1          13C
P1            11.00 usec
PL1           0.00 dB
SFO1          100.6237964 MHz

===== CHANNEL f2 =====
CPDPRG2       mlev16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          16.20 dB
SFO2          400.1328009 MHz

F2 - Processing parameters
SI            65536
SF            100.6127500 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.00

1D NMR plot parameters
CX            22.80 cm
CY            15.50 cm
F1P           78.000 ppm
F1            7847.79 Hz
F2P           75.000 ppm
F2            7545.96 Hz
PPMCM         0.13158 ppm/cm
HZCM          13.23852 Hz/cm
```

13C spectrum with 1H decoupling



Current Data Parameters
 USER nmrl1t
 NAME nmrl1t-X-sample4
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20111018
 Time 20.36
 INSTRUM drx400
 PROBHD 5 mm QNP H/F/P
 PULPROG zgdc30
 TD 65536
 SOLVENT CDCl3
 NS 305
 DS 4
 SWH 24154.590 Hz
 FIDRES 0.368570 Hz
 AQ 1.3566452 sec
 RG 9195.2
 DW 20.700 usec
 DE 20.39 usec
 TE 298.1 K
 D1 0.10000000 sec
 d11 0.03000000 sec
 MCREST 0.00000000 sec
 MCWPK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 11.00 usec
 PL1 0.00 dB
 SF01 100.6237964 MHz

===== CHANNEL f2 =====
 CPDPRG2 mlev16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 16.20 dB
 SF02 400.1328009 MHz

F2 - Processing parameters
 SI 65536
 SF 100.6127500 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.80 cm
 CY 3.56 cm
 F1P 180.000 ppm
 F1 18110.29 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 7.89474 ppm/cm
 HZCM 794.31122 Hz/cm