

NAME _____

Chem 203

Organic Spectroscopy

Midterm Examination, Part II (60 points total)

Problem 2 of 4 (three out of four required, 20 points)

Saturday, November 9, 2013, 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.

2. Analyze the spectra and solve the structure of the molecule for which data are provided. The following data are provided: exact mass; EI-MS and CI-MS (low resolution/accuracy); IR (solution in CHCl_3 in a 0.1 mm CaF_2 cell); 500 MHz ^1H NMR in C_6D_6 ; 125.8 MHz ^{13}C NMR, DEPT 90, and DEPT 135 in C_6D_6 .

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: data not provided

Noteworthy Heteroatoms:

Molecular Formula:

Unsaturation Number:

Functional Groups (be as specific as possible):

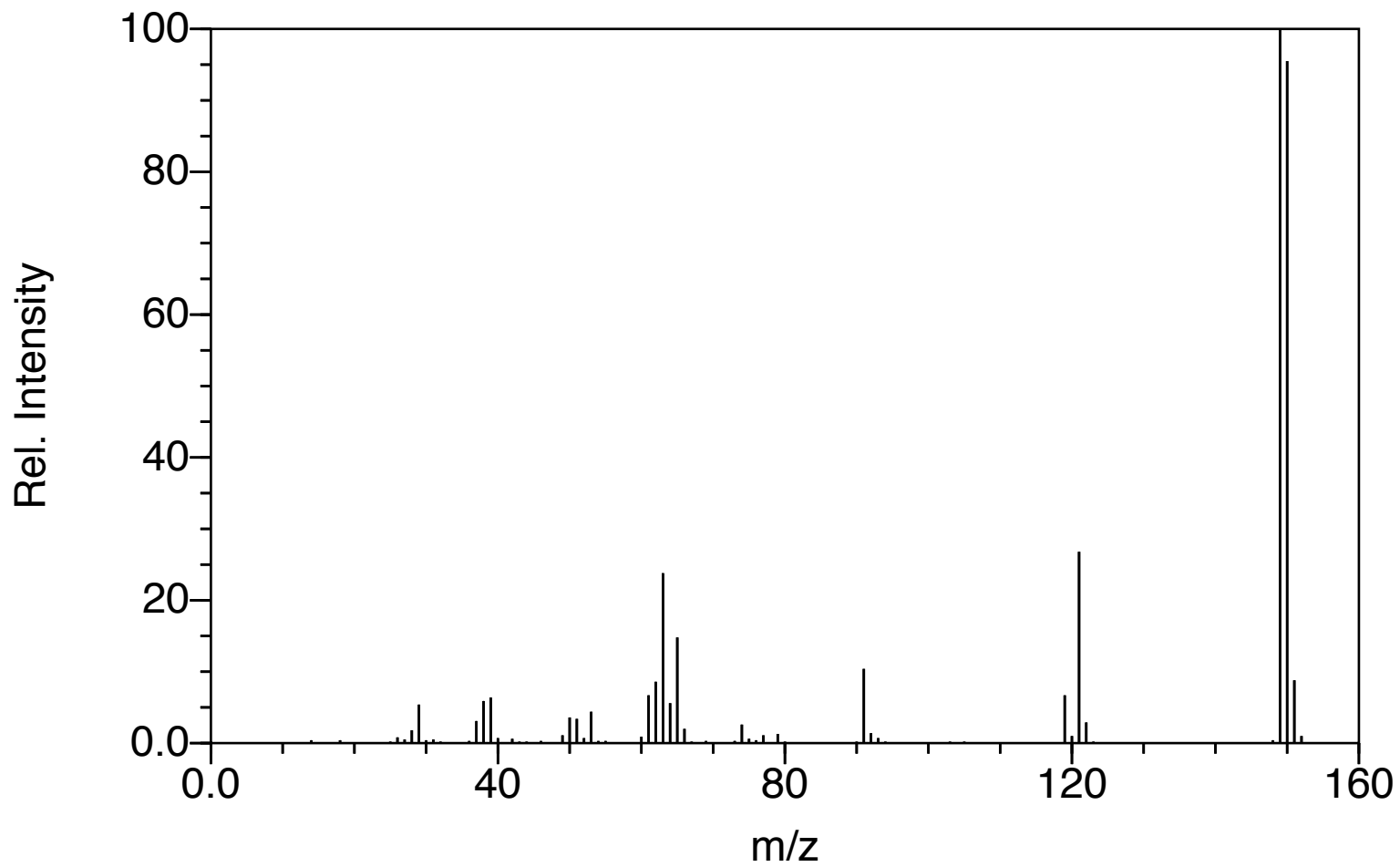
Fragments (from NMR):

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.)

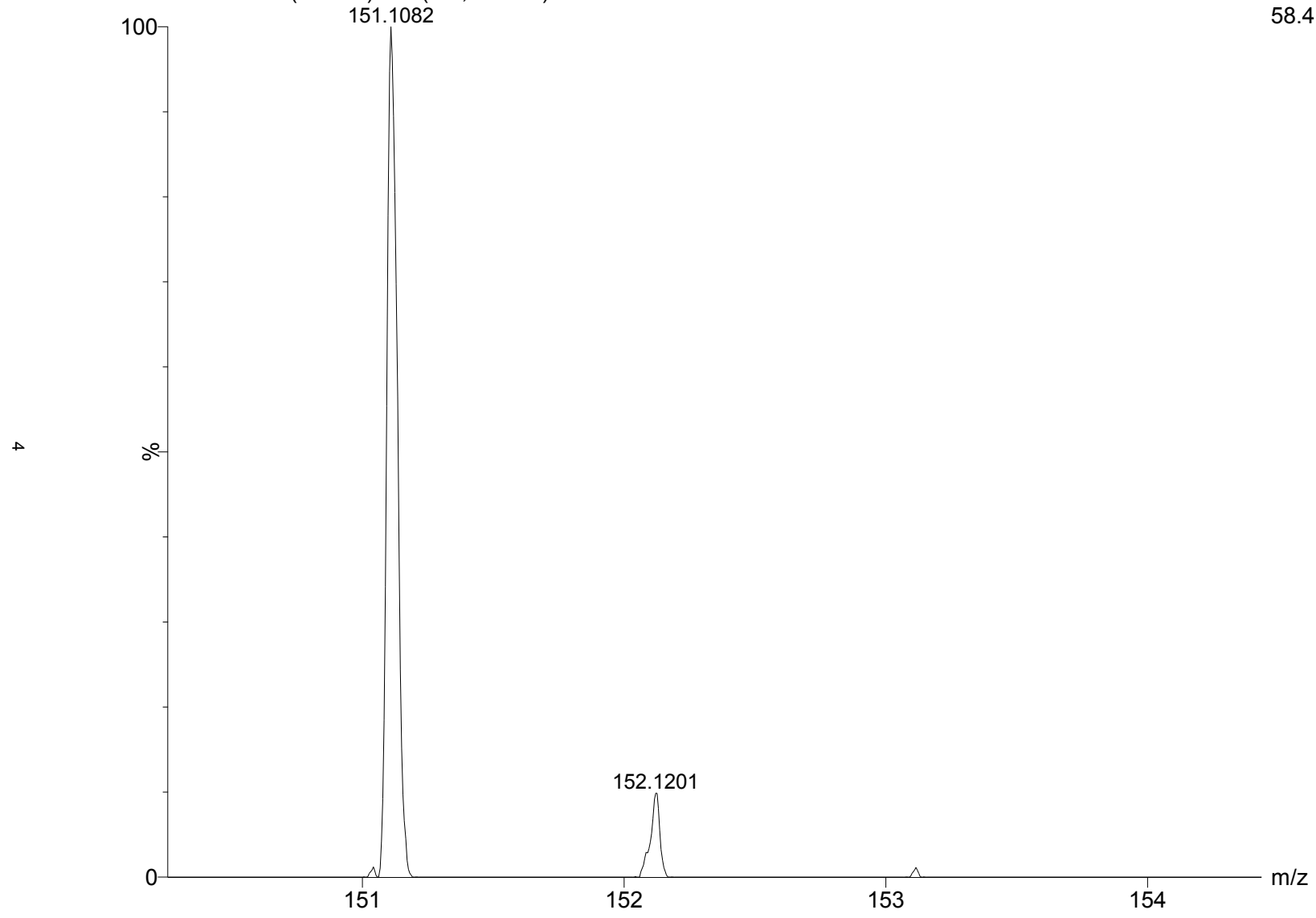
EI-MS (electron ionization)



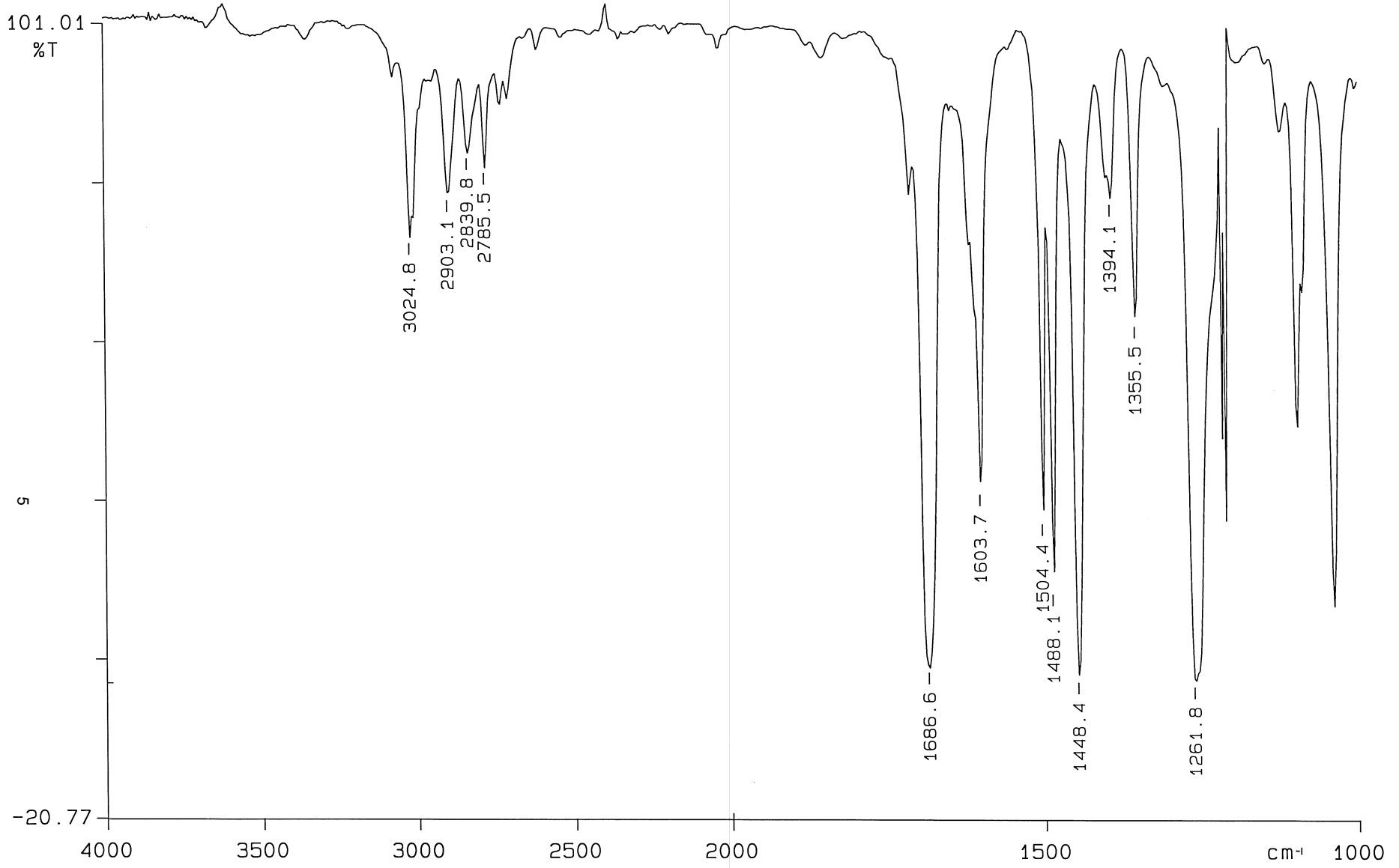
CI-MS (Chemical Ionization, low accuracy/resolution)

TOF MS Cl+
58.4

RKS-M2-1 1334 (18.400) Sm (SG, 2x3.00)

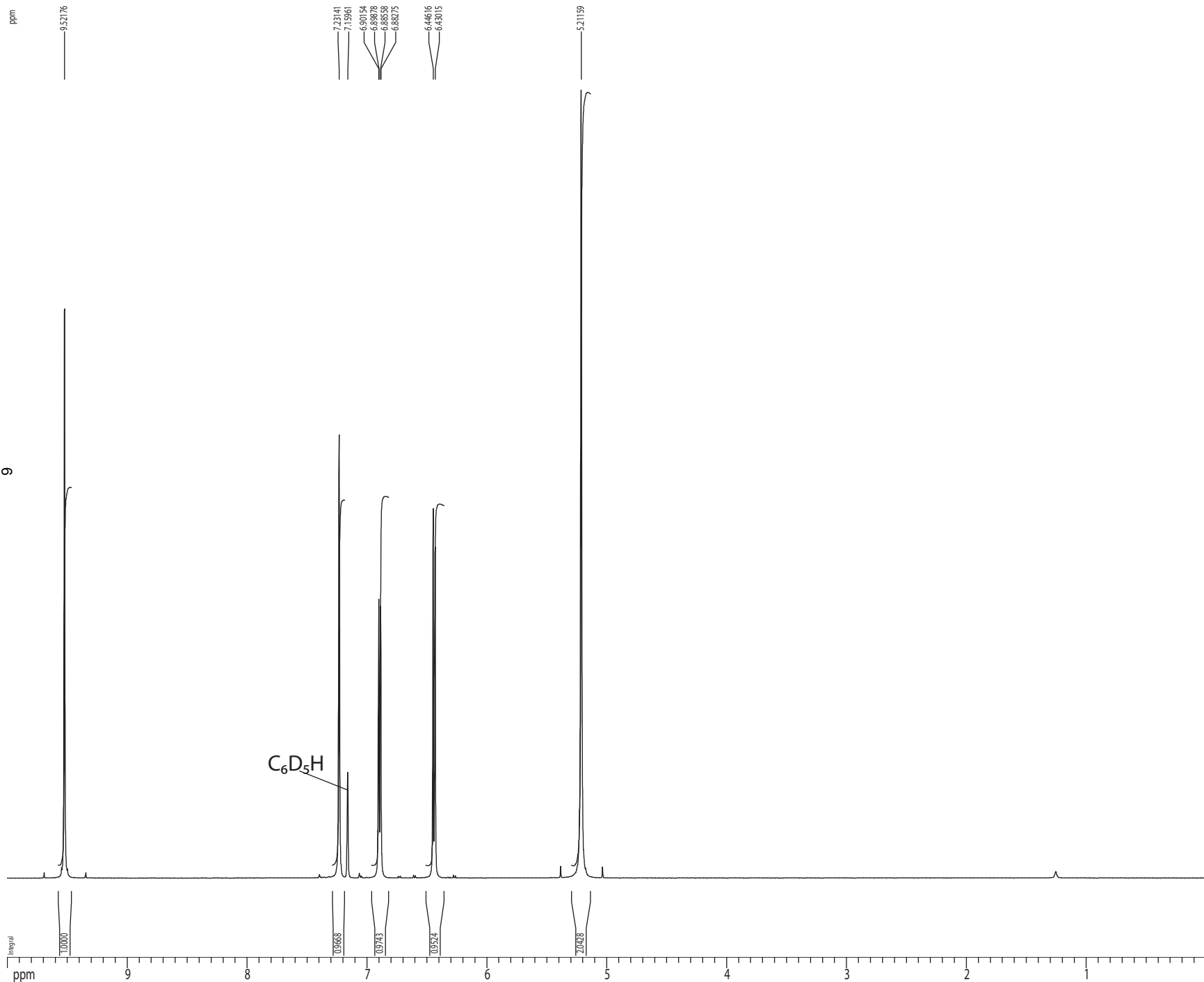


PERKIN ELMER



00/01/08 17:33
X: 4 scans, 4.0cm⁻¹

1H spectrum



Current Data Parameters
 USER nmr13t
 NAME Mid-2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20131024
 Time 16.09
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG zg30
 TD 81728
 SOLVENT C6D6
 NS 8
 DS 2
 SWH 8012820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0998774 sec
 RG 4.5
 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 =====
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 P1 7.50 usec
 PL1 1.60 dB
 SFO1 500.2235015 MHz

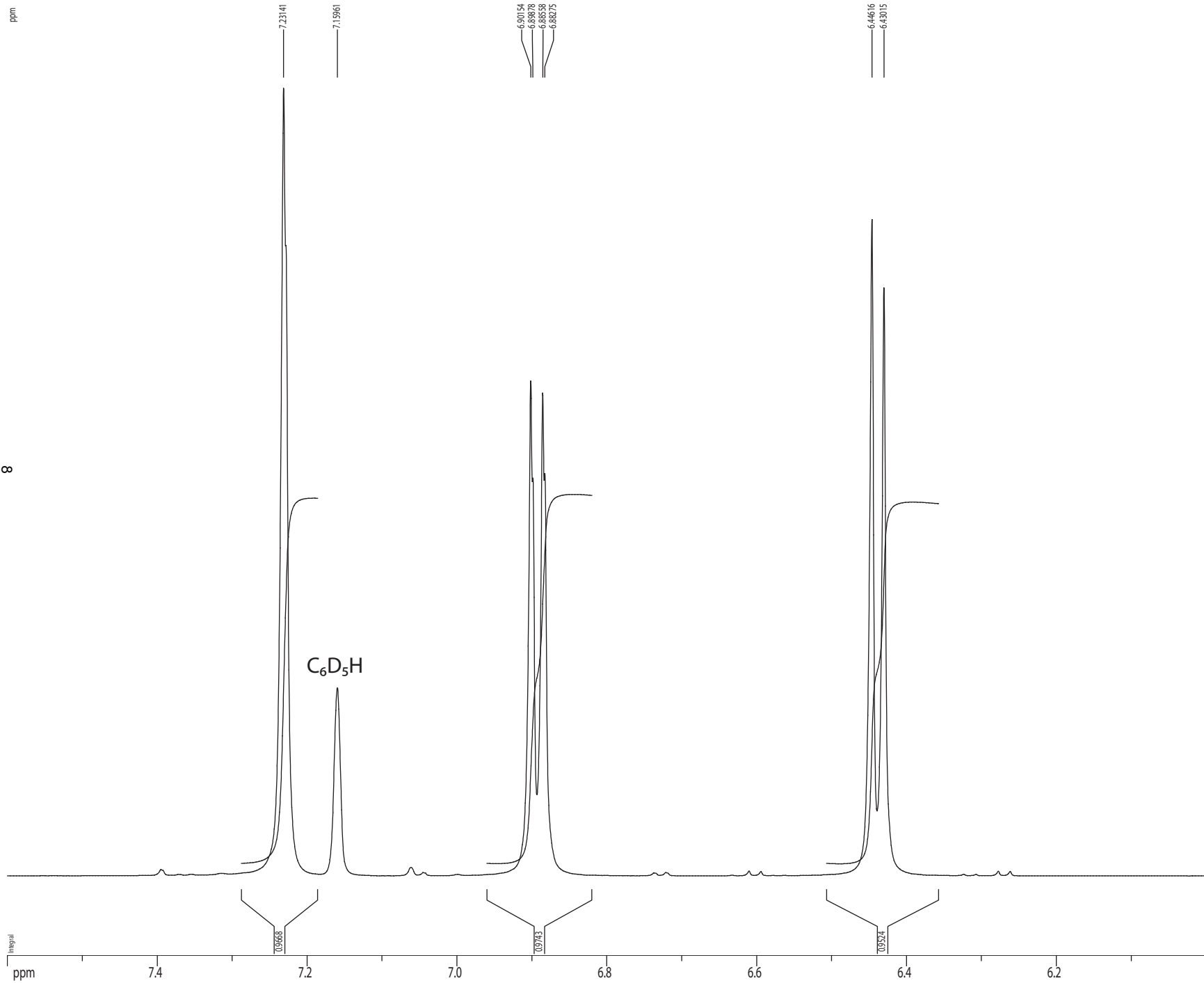
F2 - Processing parameters
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 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 F1P 10.000 ppm
 F1 5002.20 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.43860 ppm/cm
 HZCM 219.39474 Hz/cm

DU=/v, USER=nmr13t, NAME=Mid-2, EXPNO=1, PROCNO=1
F1=15.009ppm, F2=-1.009ppm, MI=0.50cm, MAXI=10000.00cm, PC=4.000

| # | ADDRESS | FREQUENCY | INTENSITY | |
|----|---------|-----------|-----------|-------|
| | [Hz] | [PPM] | | |
| 1 | 22450.9 | 4762.976 | 9.5218 | 10.84 |
| 2 | 31821.3 | 3617.294 | 7.2314 | 8.45 |
| 3 | 32115.0 | 3581.382 | 7.1596 | 2.02 |
| 4 | 33170.8 | 3452.289 | 6.9015 | 5.31 |
| 5 | 33182.1 | 3450.908 | 6.8988 | 4.27 |
| 6 | 33236.1 | 3444.306 | 6.8856 | 5.18 |
| 7 | 33247.7 | 3442.891 | 6.8828 | 4.32 |
| 8 | 35033.9 | 3224.498 | 6.4462 | 7.04 |
| 9 | 35099.4 | 3216.492 | 6.4302 | 6.32 |
| 10 | 40084.8 | 2606.941 | 5.2116 | 15.00 |

1H spectrum



Current Data Parameters
 USER nmr13t
 NAME Mid-2
 EXPNO 1
 PROCNO 1

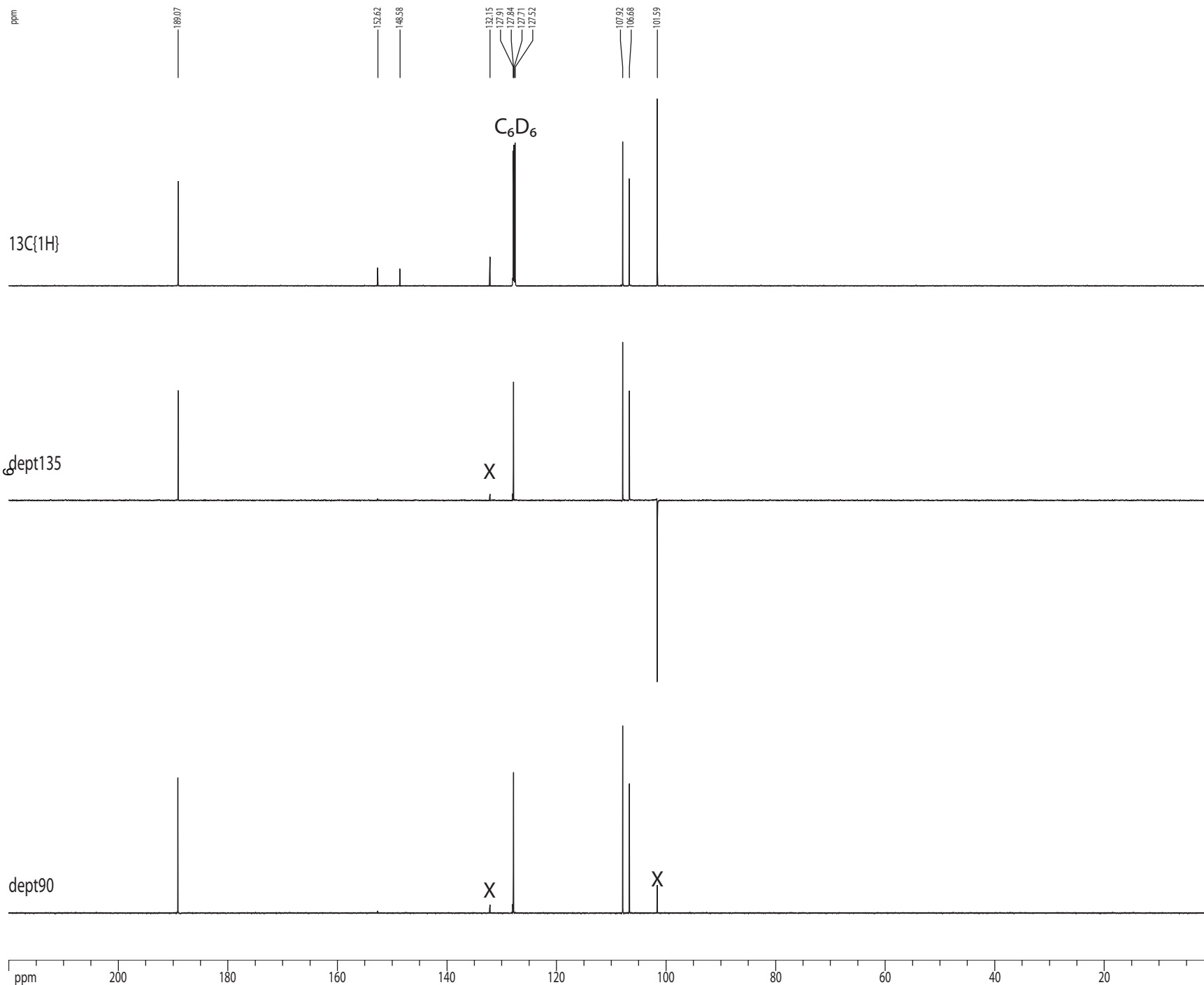
F2 - Acquisition Parameters
 Date_ 20131024
 Time 16.09
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG zg30
 TD 81728
 SOLVENT C6D6
 NS 8
 DS 2
 SWH 8012820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0998774 sec
 RG 4.5
 DW 62.400 usec
 DE 6.00 usec
 TE 298.0 K
 D1 0.10000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

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

F2 - Processing parameters
 SI 65536
 SF 500.2200000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 22.80 cm
 CY 15.00 cm
 F1P 7.600 ppm
 F1 3801.67 Hz
 F2P 6.000 ppm
 F2 3001.32 Hz
 PPMCM 0.07018 ppm/cm
 HZCM 35.10316 Hz/cm

Z-restored spin-echo ¹³C spectrum with ¹H decoupling



Current Data Parameters
 USER nmr13t
 NAME Mid-2
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20131024
 Time 16.11
 INSTRUM cryo500
 PROBHHD 5 mm CPTCI 1H-
 PULPROG SpinEchogg30gpprd
 TD 65536
 SOLVENT CDCl3
 NS 128
 DS 16
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 2580.3
 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
 MCWRK 0.01500000 sec
 P2 31.00 usec

===== CHANNEL f1 =====
 NUC1 ¹³C
 P1 15.50 usec
 P11 500.00 usec
 P12 2000.00 usec
 PLO 120.00 dB
 PL1 -1.00 dB
 SFO1 125.7942548 MHz
 SP1 3.20 dB
 SP2 3.20 dB
 SPNAM1 Crp60.0.5.20.1
 SPNAM2 Crp60comp.4
 SPOFF1 0.00 Hz
 SPOFF2 0.00 Hz

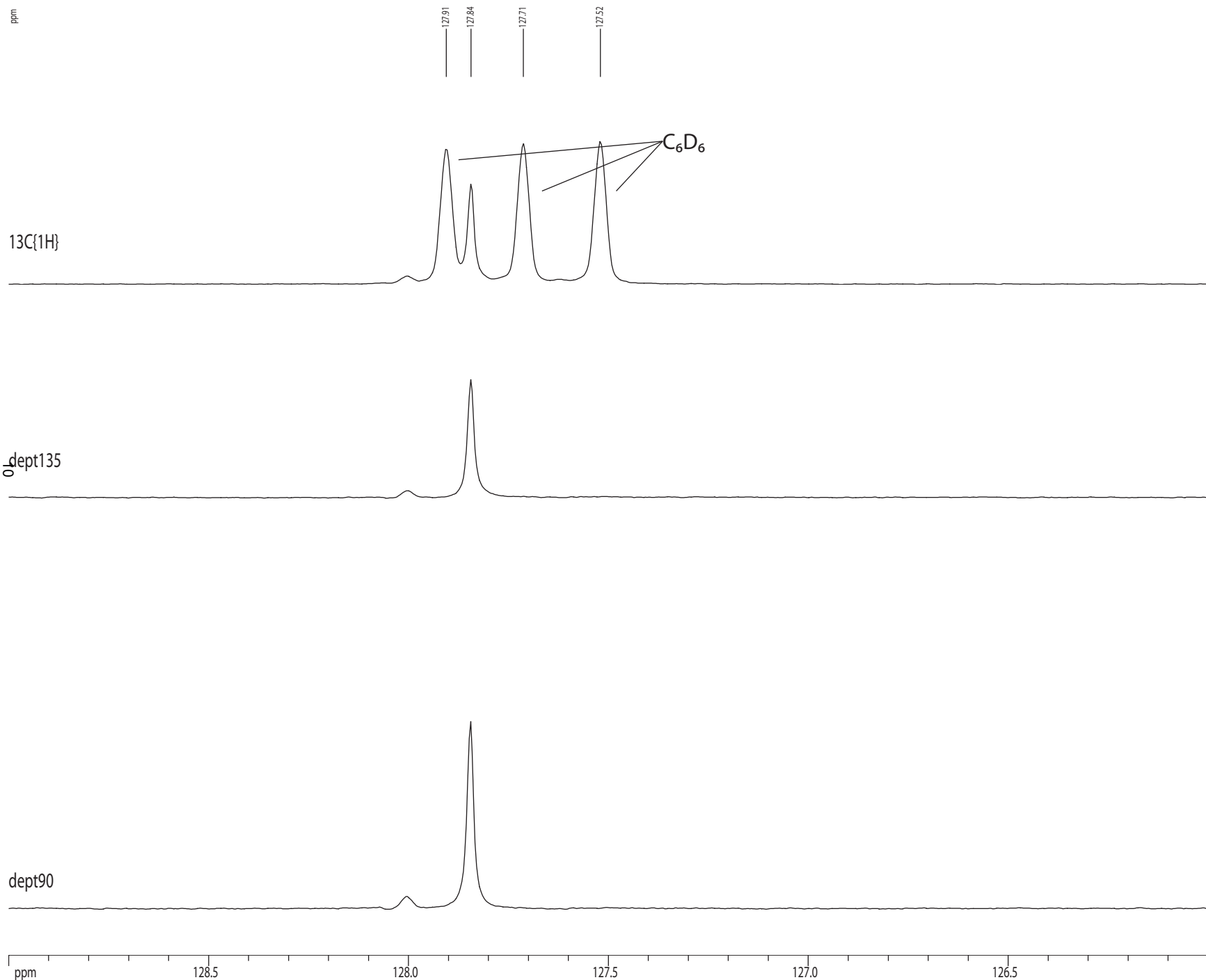
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ¹H
 PCPD2 100.00 usec
 PL2 1.60 dB
 PL12 24.60 dB
 SFO2 500.2225011 MHz

===== GRADIENT CHANNEL =====
 GPNAM1 SINE.100
 GPNAM2 SINE.100
 GPX1 0.00 %
 GPX2 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
 SI 65536
 SF 125.7804190 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.80 cm
 CY 3.56 cm
 F1P 220.000 ppm
 F1 27671.69 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 9.64912 ppm/cm
 HZCM 1213.67078 Hz/cm

Z-restored spin-echo ¹³C spectrum with ¹H decoupling



Current Data Parameters
 USER nmr13t
 NAME Mid-2
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20131024
 Time 16.11
 INSTRUM cryo500
 PROBHD 5 mm CPTCI 1H-
 PULPROG SpinEchogg30gp.prd
 TD 65536
 SOLVENT CDCl3
 NS 128
 DS 16
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 2580.3
 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
 MCWRK 0.01500000 sec
 P2 31.00 usec

===== CHANNEL f1 =====
 NUC1 ¹³C
 P1 15.50 usec
 P11 500.00 usec
 P12 2000.00 usec
 PLO 120.00 dB
 PL1 -1.00 dB
 SFO1 125.7942548 MHz
 SP1 3.20 dB
 SP2 3.20 dB
 SPNAM1 Crp60.0.5.20.1
 SPNAM2 Crp60comp.4
 SPOFF1 0.00 Hz
 SPOFF2 0.00 Hz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ¹H
 PCPD2 100.00 usec
 PL2 1.60 dB
 PL12 24.60 dB
 SFO2 500.2225011 MHz

===== GRADIENT CHANNEL =====
 GPNAM1 SINE.100
 GPNAM2 SINE.100
 GPX1 0.00 %
 GPX2 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
 SI 65536
 SF 125.7804190 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.80 cm
 CY 3.56 cm
 F1P 129.000 ppm
 F1 16225.67 Hz
 F2P 126.000 ppm
 F2 15848.33 Hz
 PPMCM 0.13158 ppm/cm
 HZCM 16.55006 Hz/cm