

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 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.

1. Analyze the spectra and solve the structure of the molecule for which data are provided. The following data are provided: exact mass; CI-MS (low resolution/accuracy); IR (solution in  $\text{CHCl}_3$  in a 0.1 mm  $\text{CaF}_2$  cell); 500 MHz  $^1\text{H}$  NMR in  $\text{C}_6\text{D}_6$ ; 125.8 MHz  $^{13}\text{C}$  NMR, DEPT 90, and DEPT 135 in  $\text{C}_6\text{D}_6$ .

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  $^1\text{H}$  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: 180.0342

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):

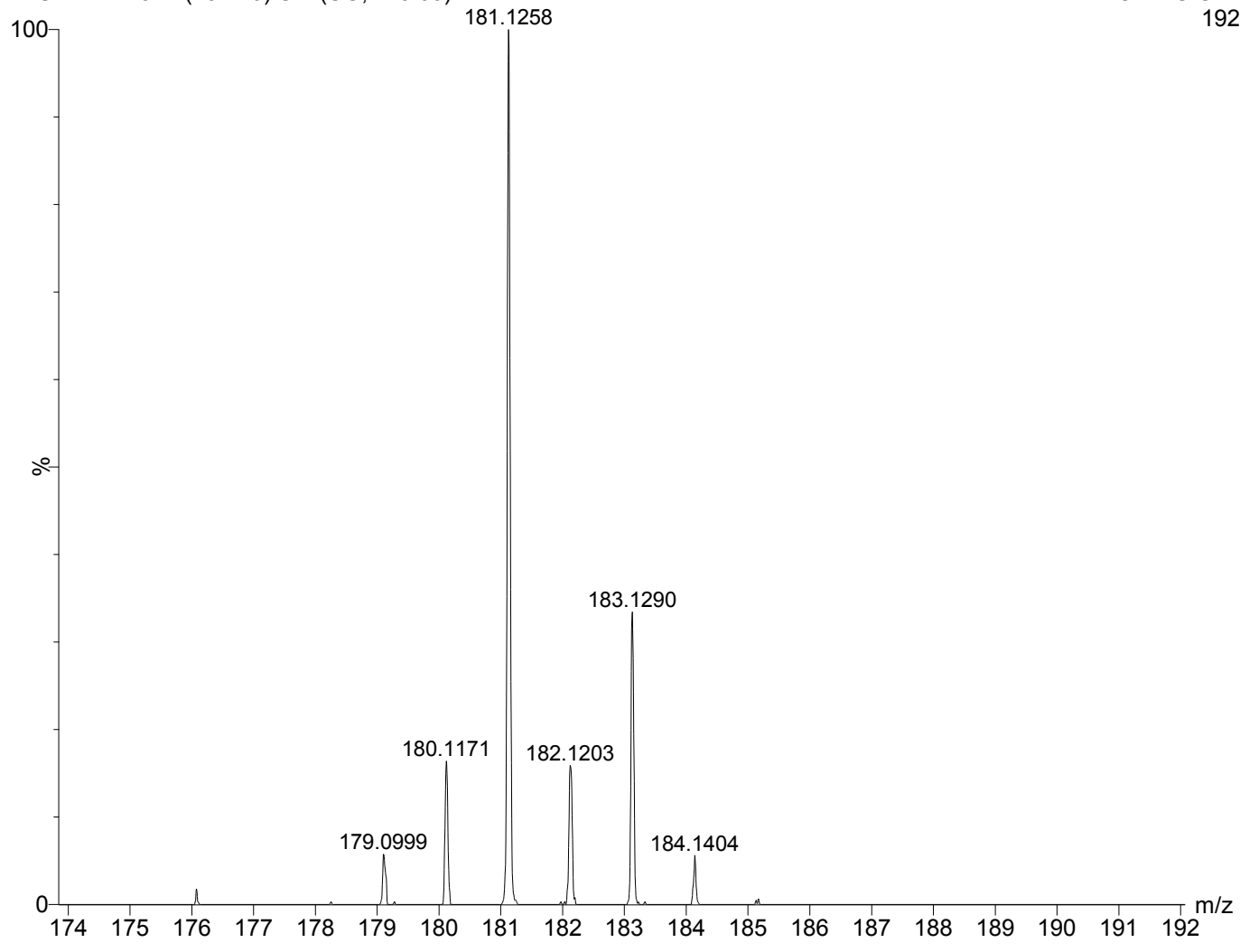
Structure with  $^1\text{H}$  NMR resonances lettered from the most downfield to the most upfield (a, b, c, d, etc.): (Note: Not all resonances can be assigned with certainty. If assignments are uncertain, indicate so by showing possible letters.)

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.)

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

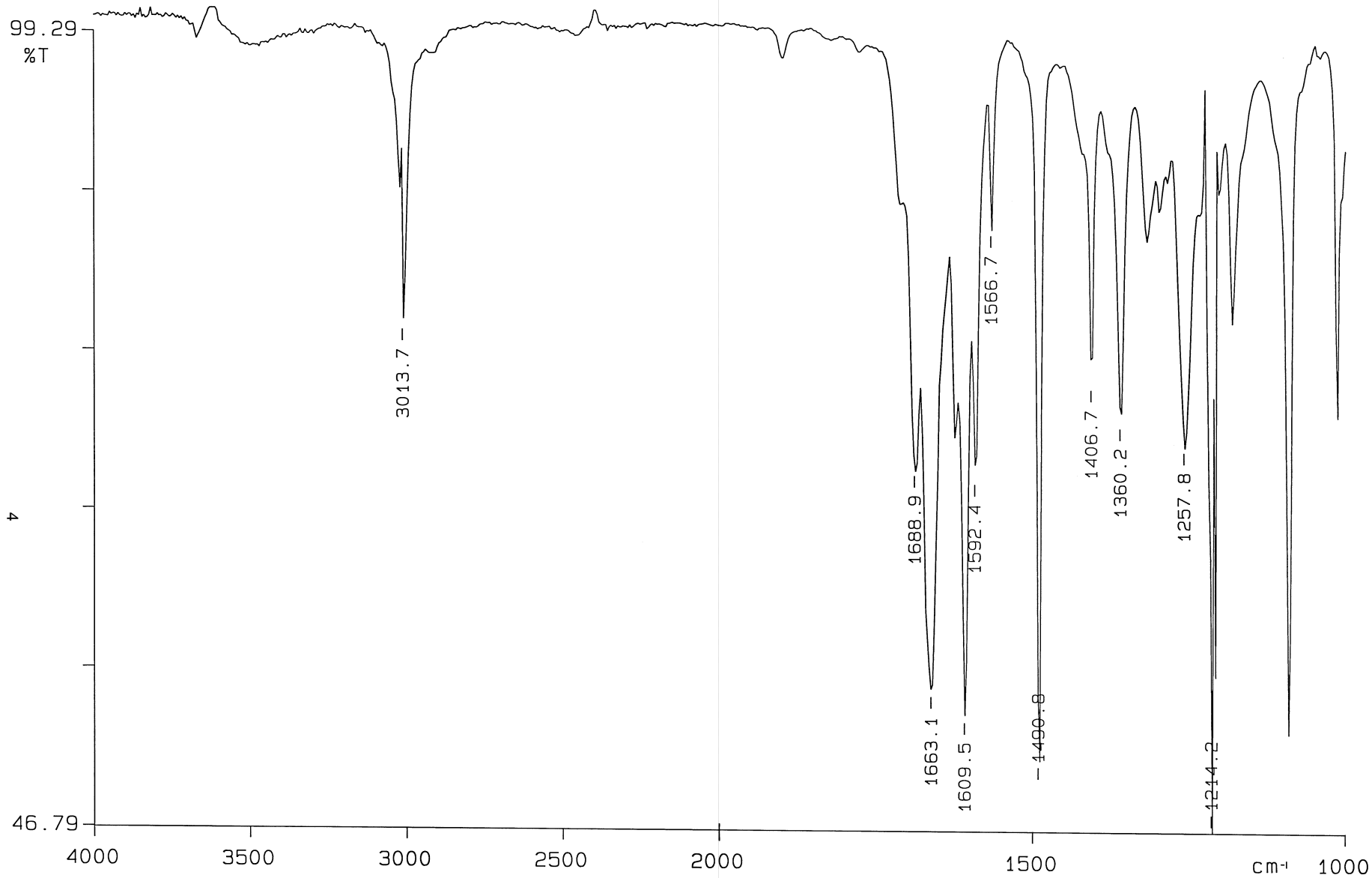
TOF MS Cl+  
192

RKS-M1-1 1371 (18.770) Sm (SG, 2x3.00)



MS

PERKIN ELMER



00/01/08 17:28

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

1H spectrum

ppm

7.15957  
7.08642  
7.03380  
6.94403  
6.81167  
6.79458  
  
6.35753  
6.32496

1.93069

C<sub>6</sub>D<sub>5</sub>H

5

Current Data Parameters  
USER nmr13t  
NAME Mid-1-3  
EXPNO 1  
PROCNO 1

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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

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P1 7.50 usec  
PL1 1.60 dB  
SFO1 500.2235015 MHz

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

1D NMR plot parameters  
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CY 14.19 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

Integral  
ppm

1.0311  
1.9500  
2.0000  
0.9388  
3.0432

9

8

7

6

5

4

3

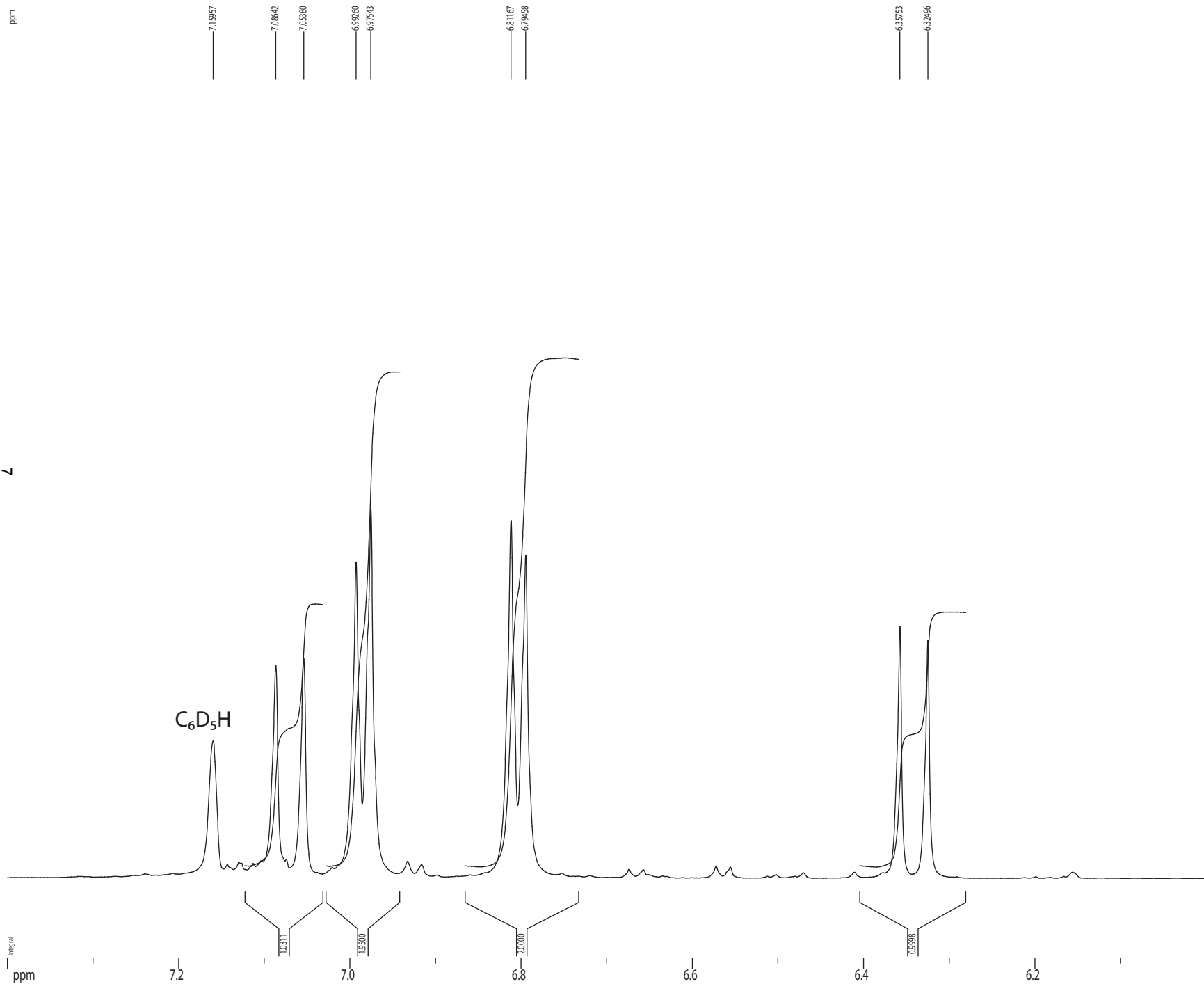
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1

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	[Hz]	[PPM]		
1	32115.2	3581.360	7.1596	2.63
2	32414.4	3544.770	7.0864	4.07
3	32547.9	3528.452	7.0538	4.19
4	32798.3	3497.840	6.9926	6.03
5	32868.5	3489.249	6.9754	7.04
6	33538.5	3407.332	6.8117	6.83
7	33608.4	3398.785	6.7946	6.17
8	35396.5	3180.162	6.3575	4.81
9	35529.8	3163.872	6.3250	4.54
10	53507.8	965.768	1.9307	14.19

# 1H spectrum



Current Data Parameters  
 USER nmr13t  
 NAME Mid-1-3  
 EXPNO 1  
 PROCNO 1

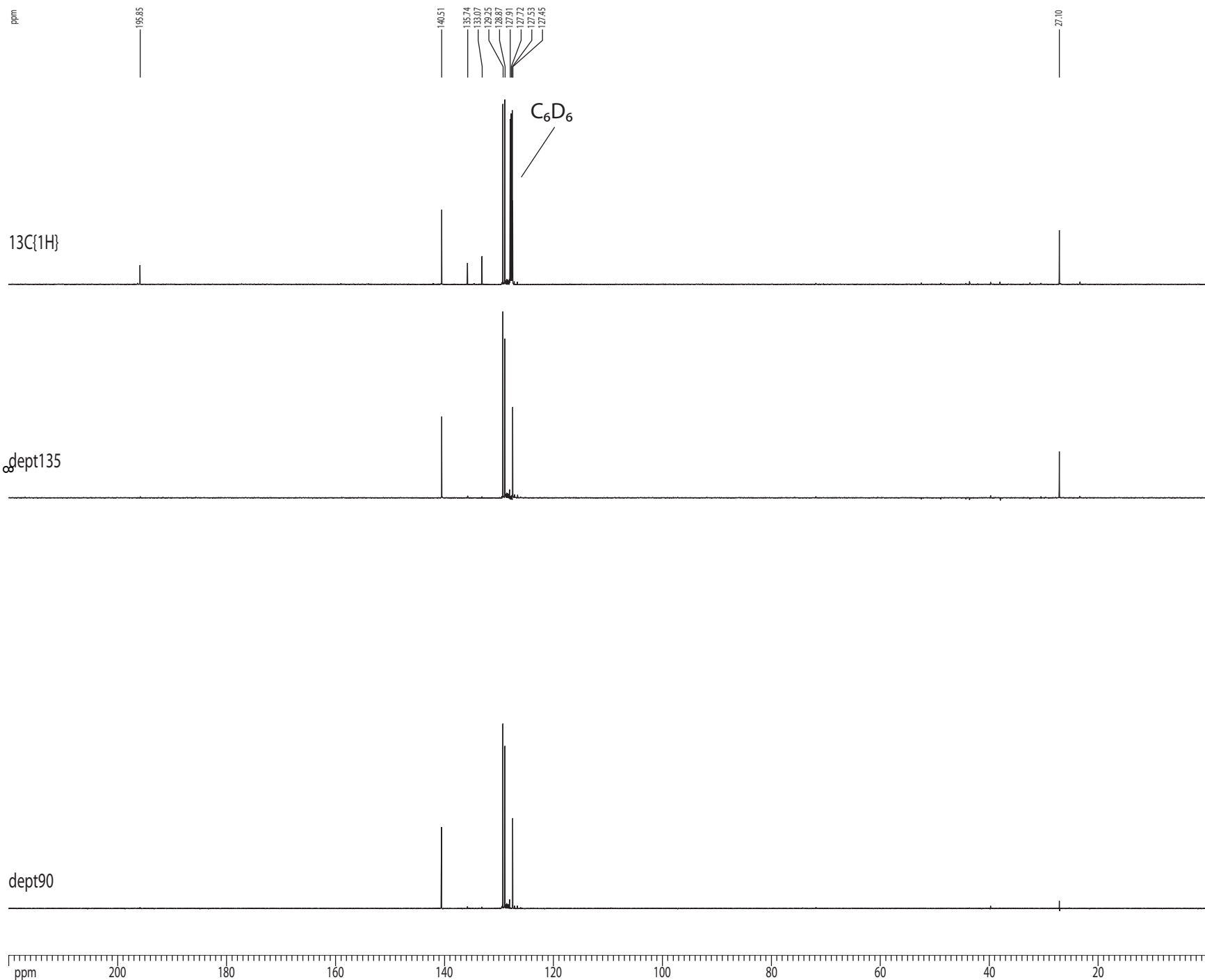
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 PROBHD 5 mm CPTCI 1H-  
 PULPROG zg30  
 TD 81728  
 SOLVENT C6D6  
 NS 8  
 DS 2  
 SWH 8012.820 Hz  
 FIDRES 0.098043 Hz  
 AQ 5.0998774 sec  
 RG 4.5  
 DW 62.400 usec  
 DE 6.00 usec  
 TE 298.0 K  
 D1 0.10000000 sec  
 MCREST 0.00000000 sec  
 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 14.19 cm  
 F1P 7.400 ppm  
 F1 3701.63 Hz  
 F2P 6.000 ppm  
 F2 3001.32 Hz  
 PPMCM 0.06140 ppm/cm  
 HZCM 30.71527 Hz/cm

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



Current Data Parameters  
 USER nmr13t  
 NAME Mid-1-3  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20131103  
 Time 12.00  
 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 5160.6  
 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  $^{13}C$   
 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 Crg60.0.5.20.1  
 SPNAM2 Crg60comp.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

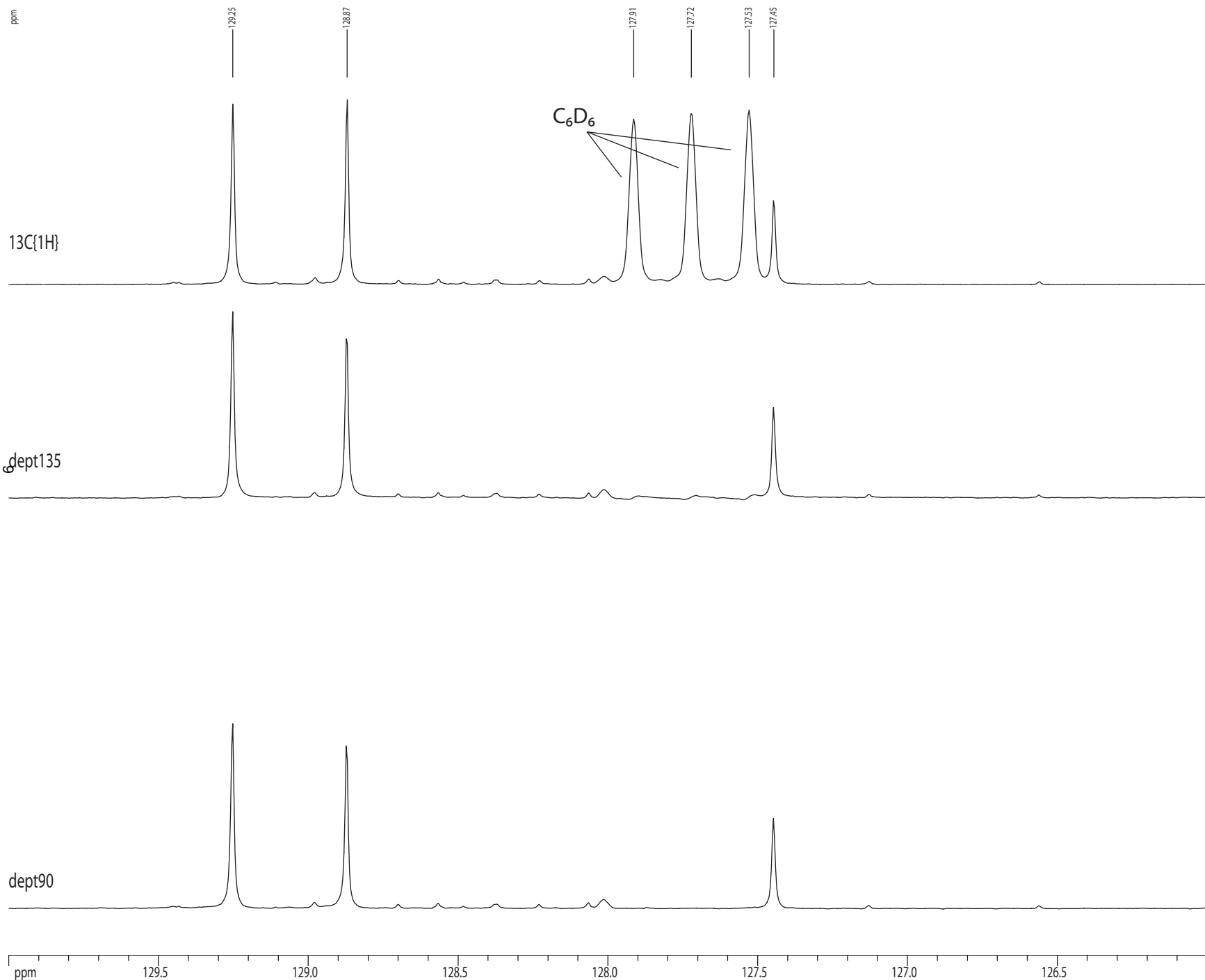
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 GPY1 0.00 %  
 GPY2 0.00 %  
 GPZ1 30.00 %  
 GPZ2 50.00 %  
 p15 500.00 usec  
 p16 1000.00 usec

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  $^{13}\text{C}$  spectrum with  $^1\text{H}$  decoupling



Current Data Parameters  
 USER nmr13t  
 NAME Mid-1-3  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20131103  
 Time 12.00  
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 TD 65536  
 SOLVENT CDCl3  
 NS 128  
 DS 16  
 SWH 30303.031 Hz  
 FIDRES 0.462388 Hz  
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 RG 5160.6  
 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  $^{13}\text{C}$   
 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  $^1\text{H}$   
 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 130.000 ppm  
 F1 16351.45 Hz  
 F2P 126.000 ppm  
 F2 15848.33 Hz  
 PPMCM 0.17544 ppm/cm  
 HZCM 22.06674 Hz/cm