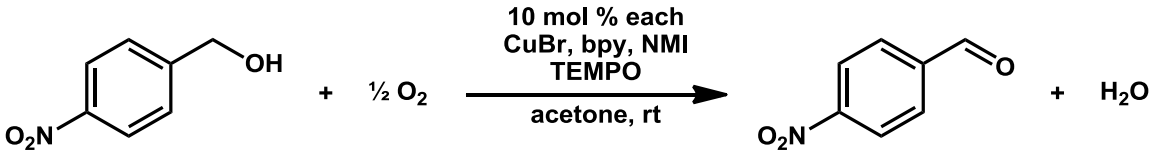
Name: **CHEM 344 Spring 2014**

Spectroscopy Problem Set

TA:

Due at start of discussion Monday the 30th June

1) Assign all signals in the ^{1}H-NMR spectrum of the crude reaction mixture from the aerobic oxidation reaction shown below. Use the standard labeling (H_a, H_b, H_c, etc.) shown in the NMR lectures and problem sets. Justify your assignments by use of the empirical parameters found in the appendix of the laboratory manual.



Use the ^{1}H-NMR spectrum of the crude product mixture to determine the % conversion of this reaction with the assumption that there are no side reactions. (**10 pts)**

2) Use the supplied spectral data to identify Compound 2, $C_8H_{14}O_4$. **(12 pts)**

3) Use the supplied spectral data to identify Compound 3 (a sulfonyl chloride) , $C_{15}H_{23}ClO_2S$. **(14 pts)**

4) Use the supplied spectral data to identify Compound 4, $C_{11}H_{14}O_2$. **(14 pts)**

Questions 2 - 4 require you to use a combination of molecular formula, NMR and MS data in order to identify each unknown compound; use all data supplied and hand in all spectra for each question.

- Clearly provide your calculation of the unsaturation number (IHD, DBE) value for each compound.

- **Draw all molecules or fragments directly onto the provided spectra**. Show all lone pairs and charges for each structure. Write and sketch clearly!

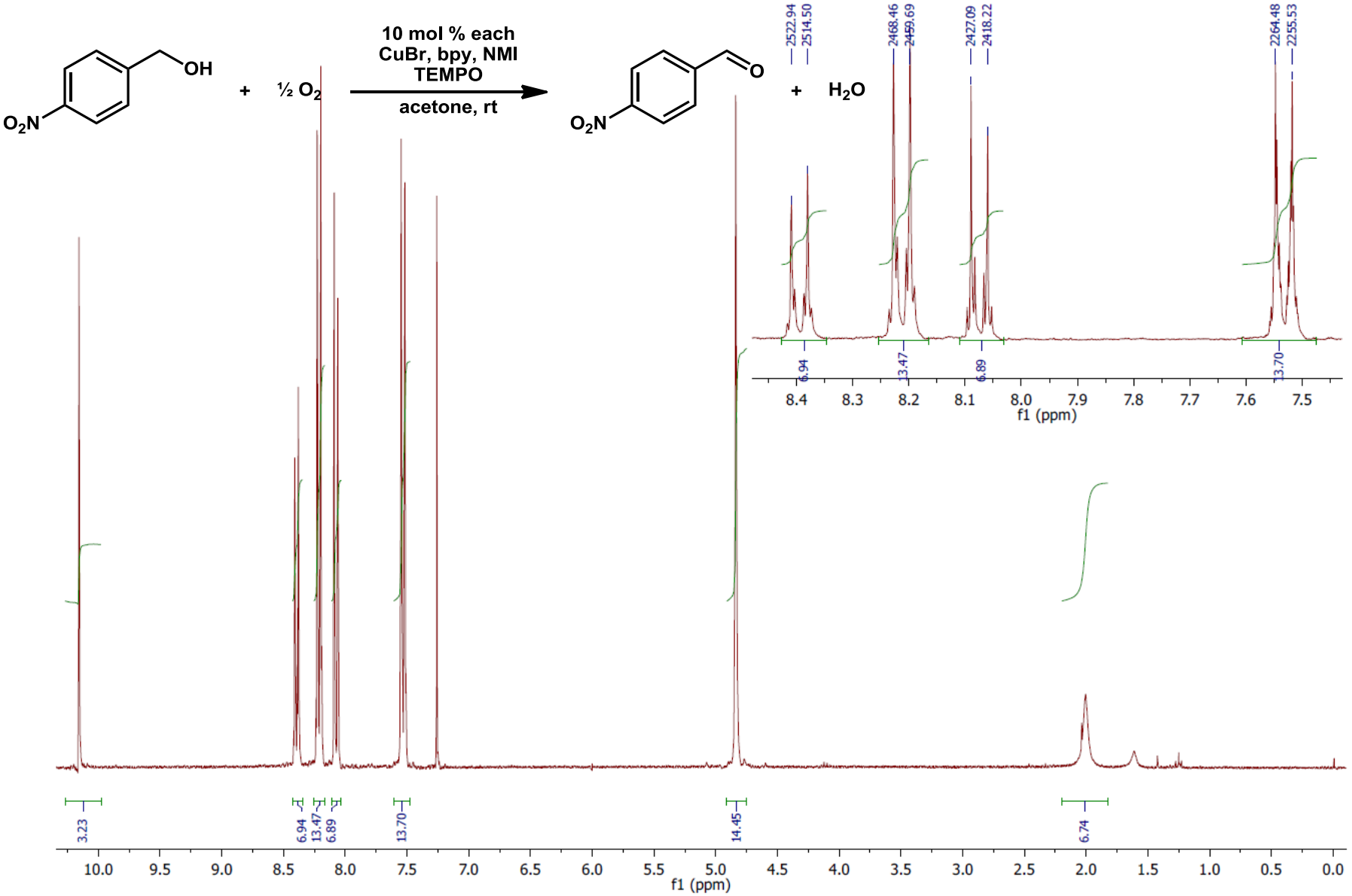
- Label each set of equivalent protons using the H_a, H_b, H_c etc. labeling system shown in the ^{1}H-NMR lectures and practice problem sets. Assign each **^{1}H-NMR** signal and write your assignments directly onto the spectrum. Justify your assignments by use of the empirical parameters found in the appendix of the laboratory manual.

- Identify each **^{13}C-NMR** signal as either alkyl, vinyl, alkynyl, aryl, nitrile, imine, or carbonyl (you do not need to assign individual carbon atoms to each signal).

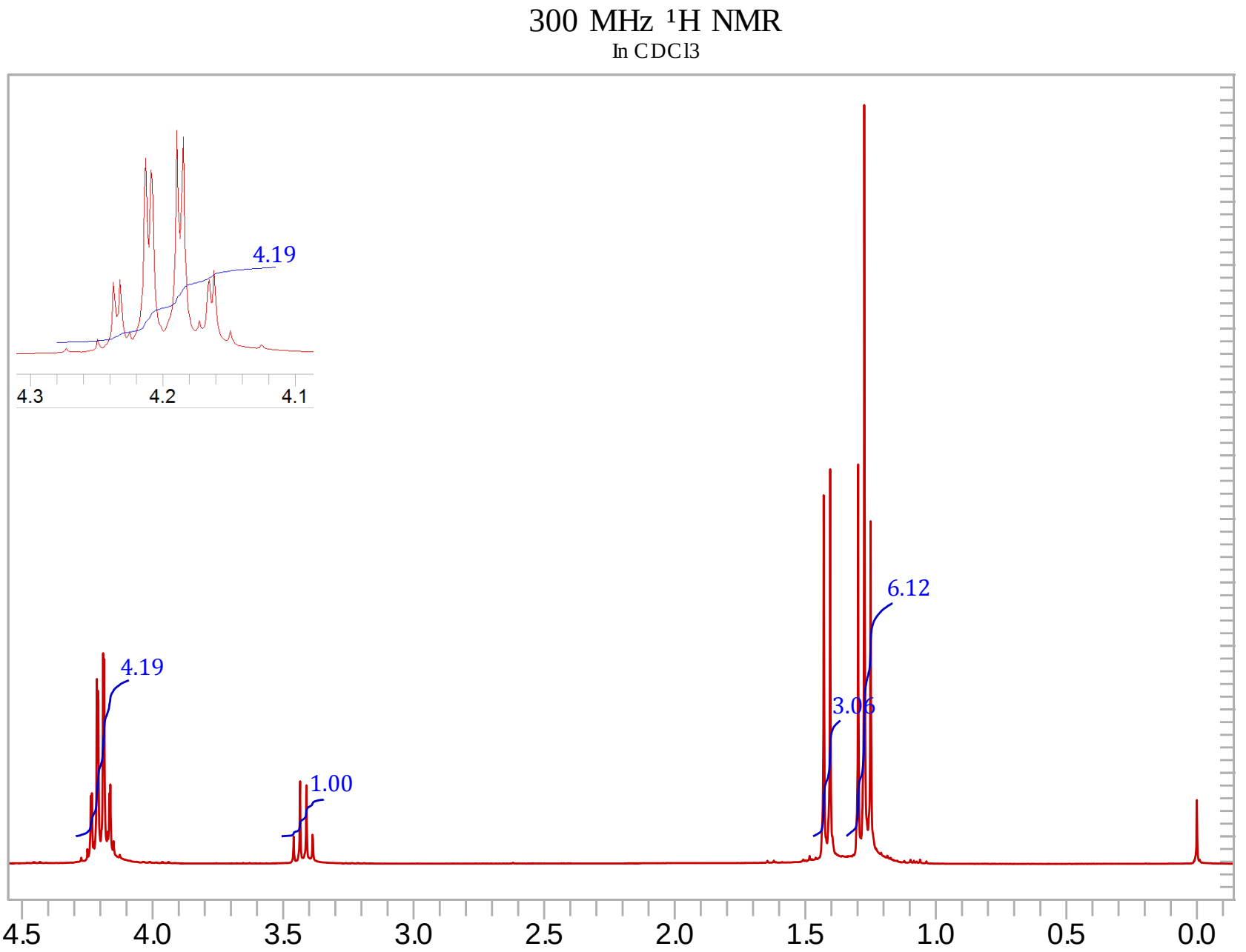
- Assign each key **IR** absorption band >1500 cm^{-1} to a specific functional group.

- Draw fragments for all labeled peaks in the **EI-MS** directly onto the spectrum (you do not need to show the fragmentation mechanism).

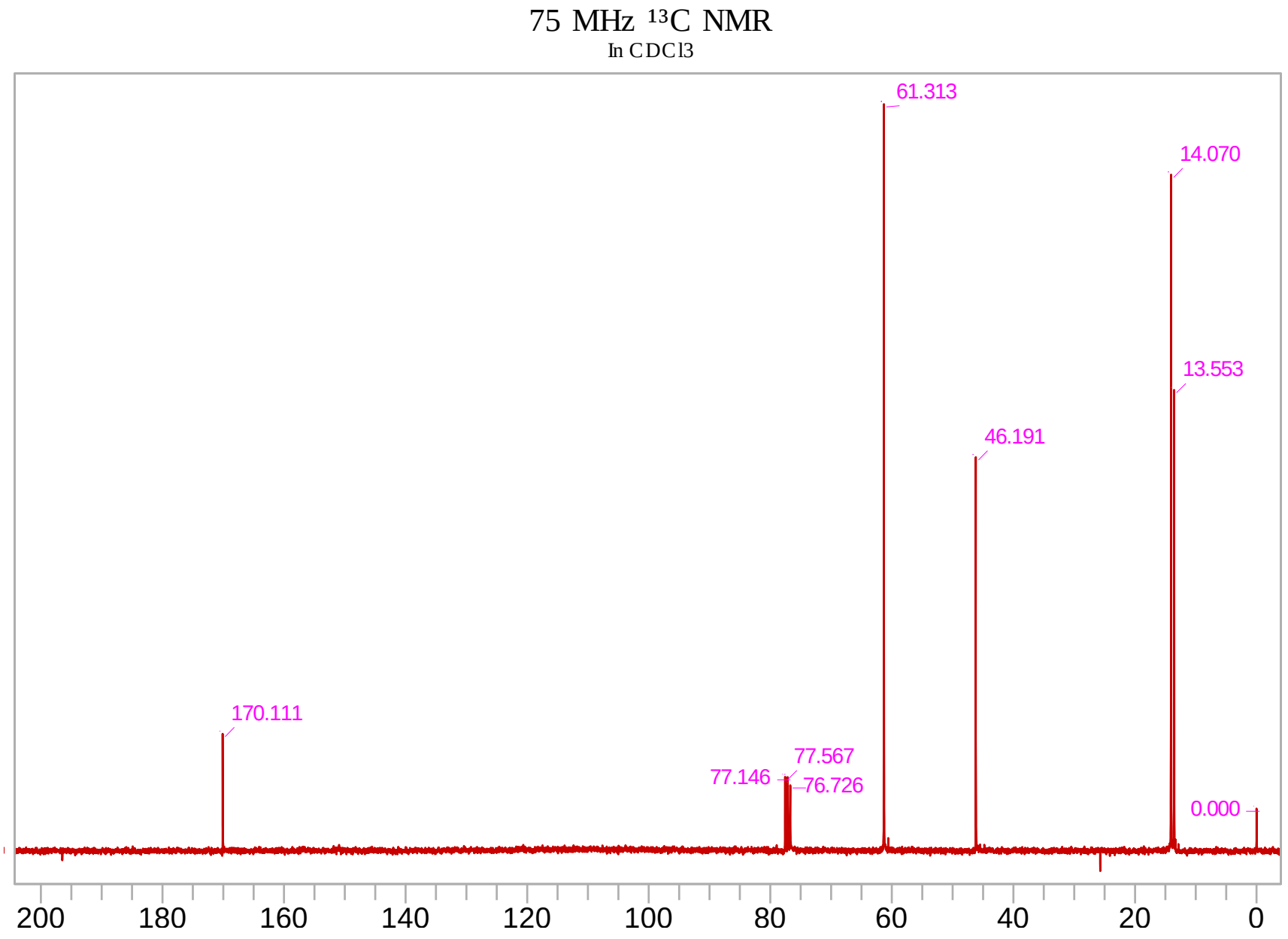
1) Aerobic Oxidation Crude Product Mixture ^{1}H-NMR



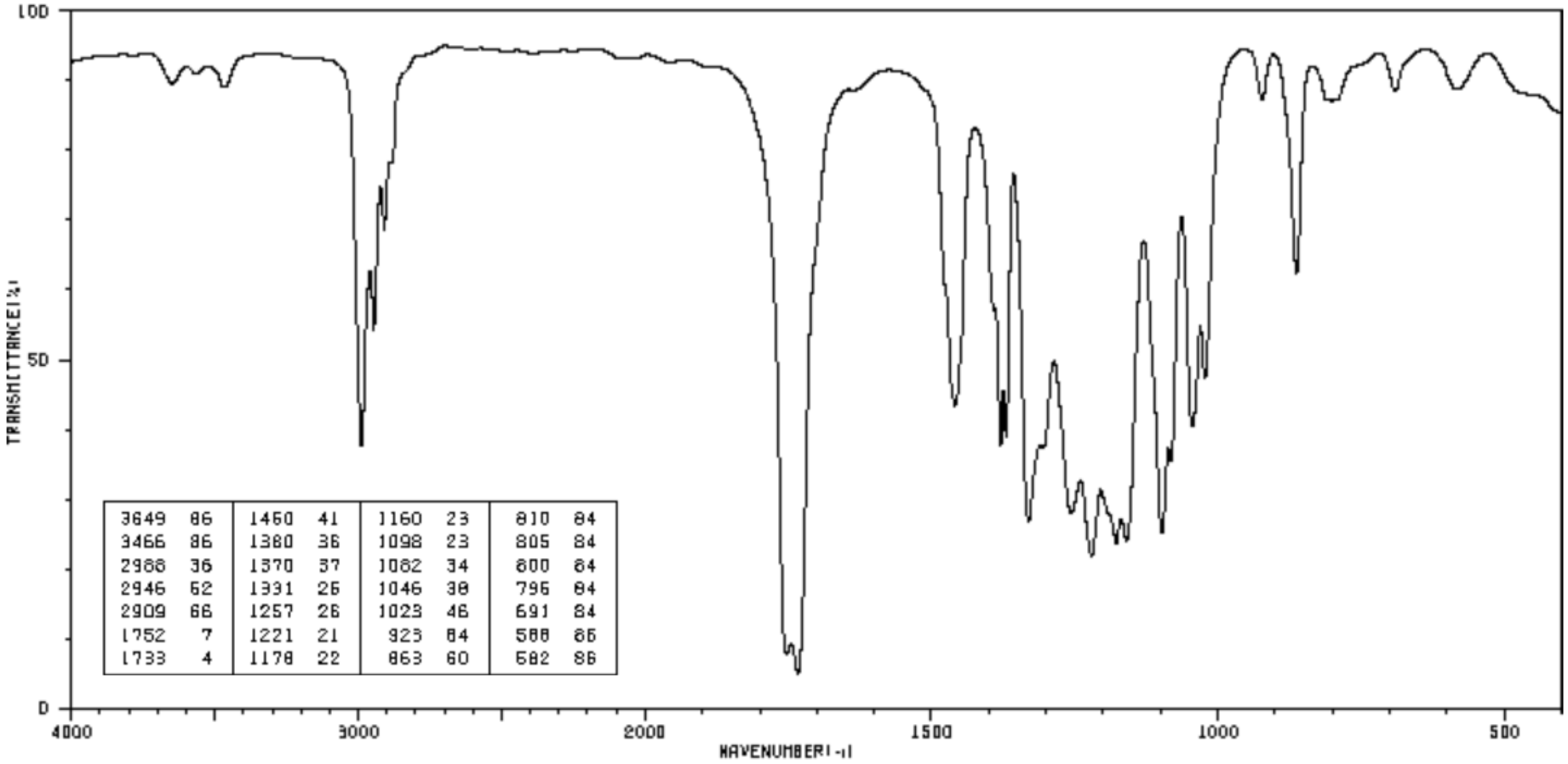
2) Compound 2 ($C_8H_{14}O_4$): ^{1}H-NMR



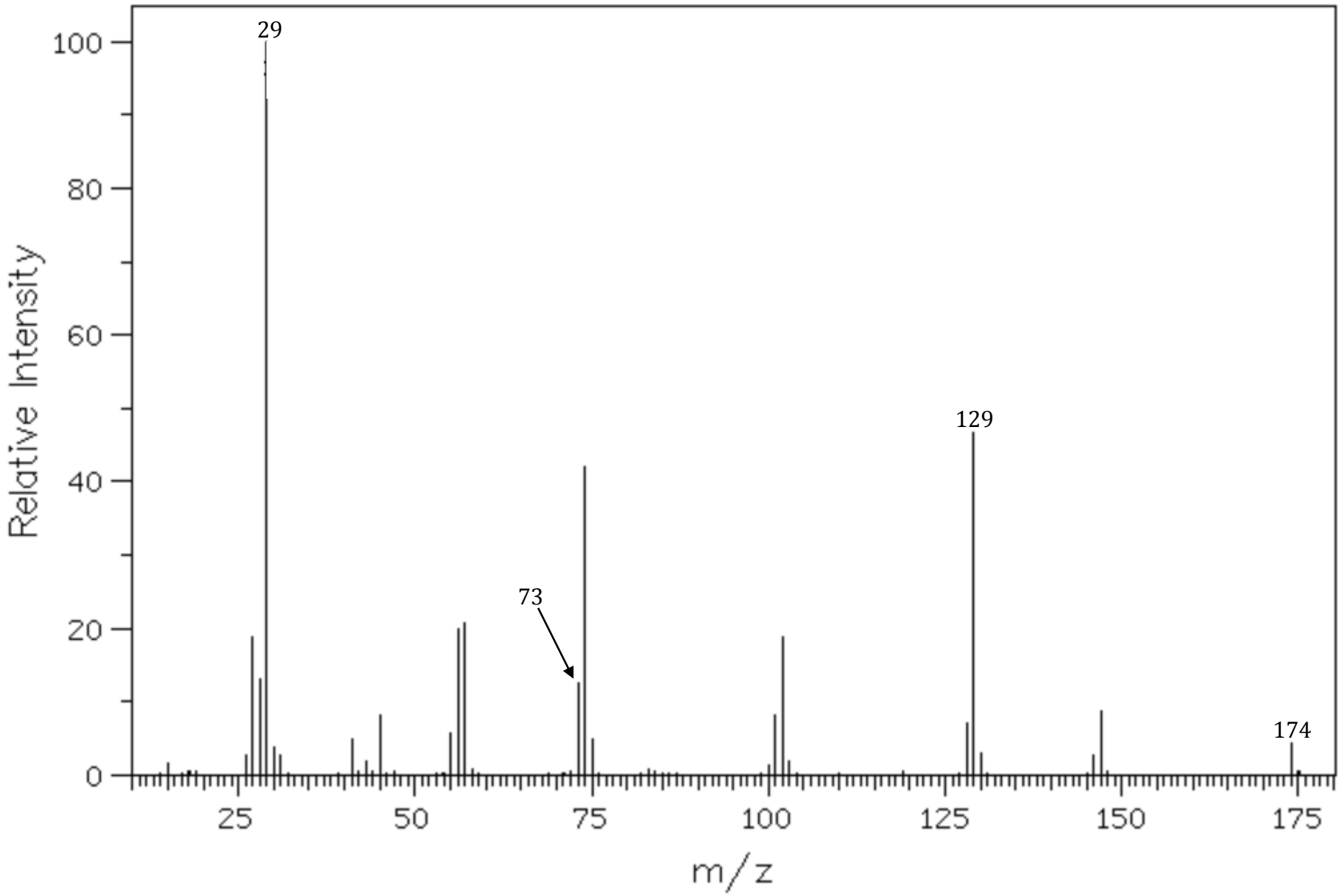
2) Compound 2 ($C_8H_{14}O_4$): ^{13}C-NMR



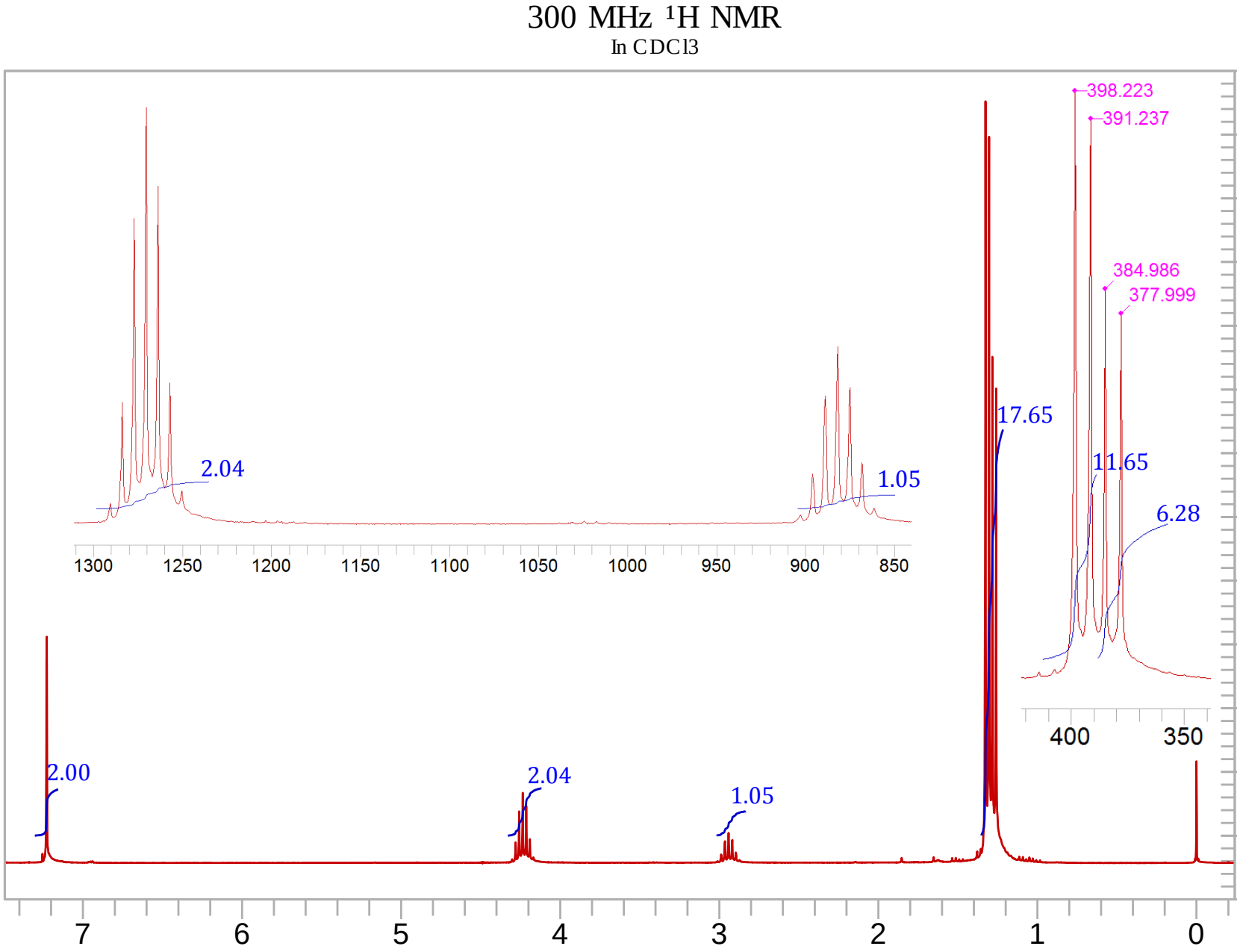
2) Compound 2 ($C_8H_{14}O_4$): IR



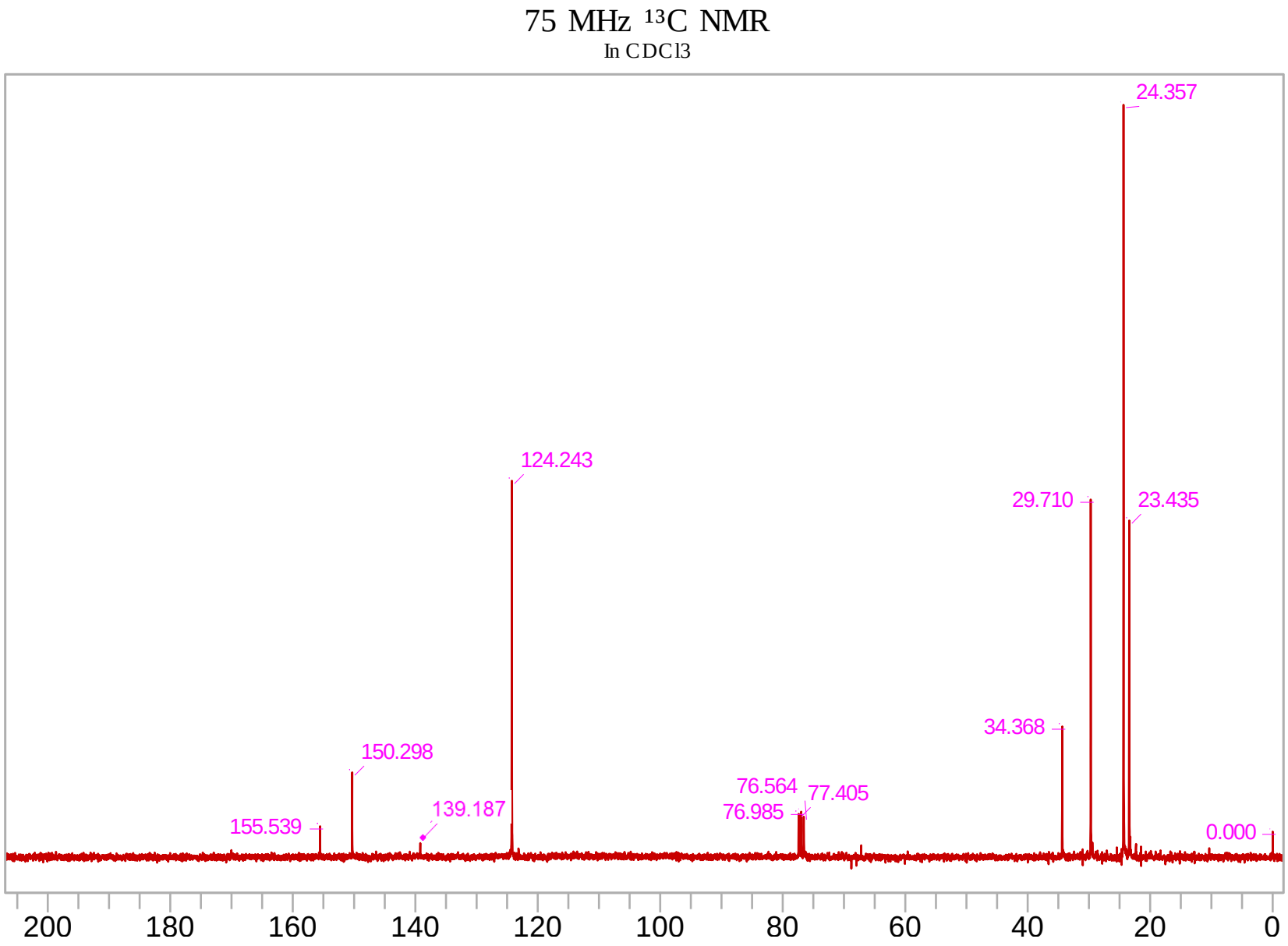
2) Compound 2 ($C_8H_{14}O_4$): EI-MS



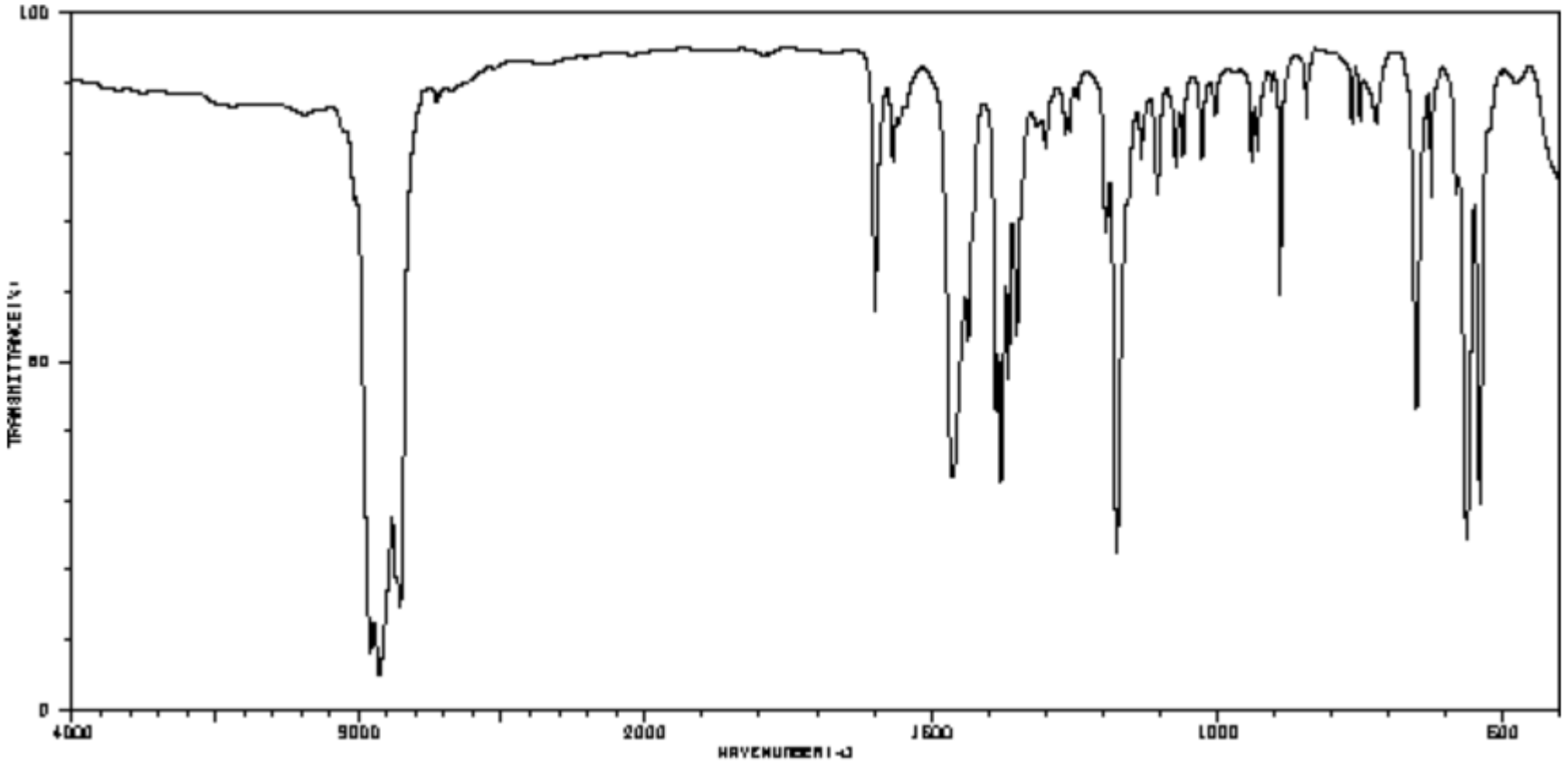
3) Compound 3 ($C_{15}H_{23}ClO_2S$): ^{1}H-NMR



3) Compound 3 ($C_{15}H_{23}ClO_2S$): ^{13}C-NMR

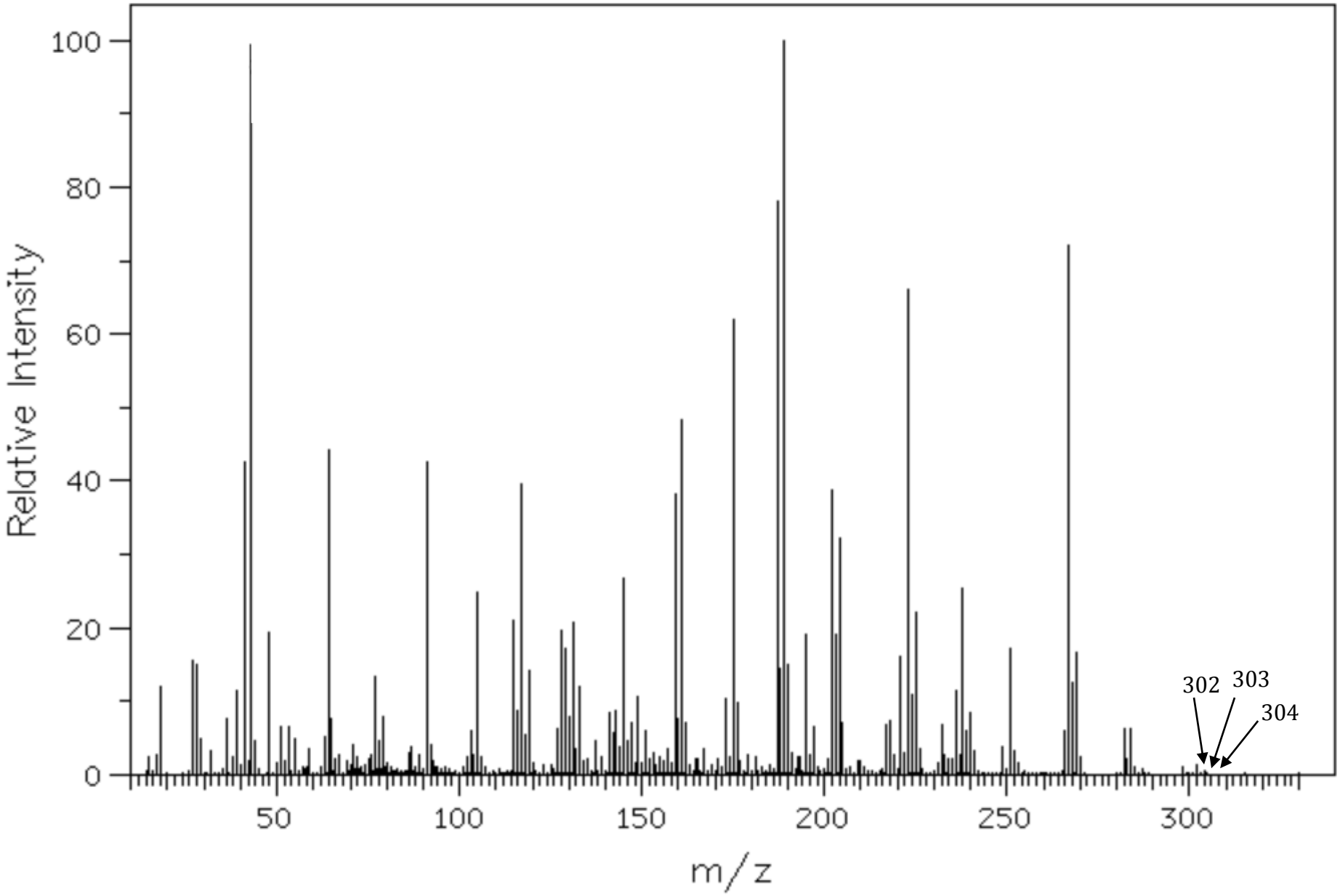


3) Compound 3 ($C_{15}H_{23}ClO_2S$): IR

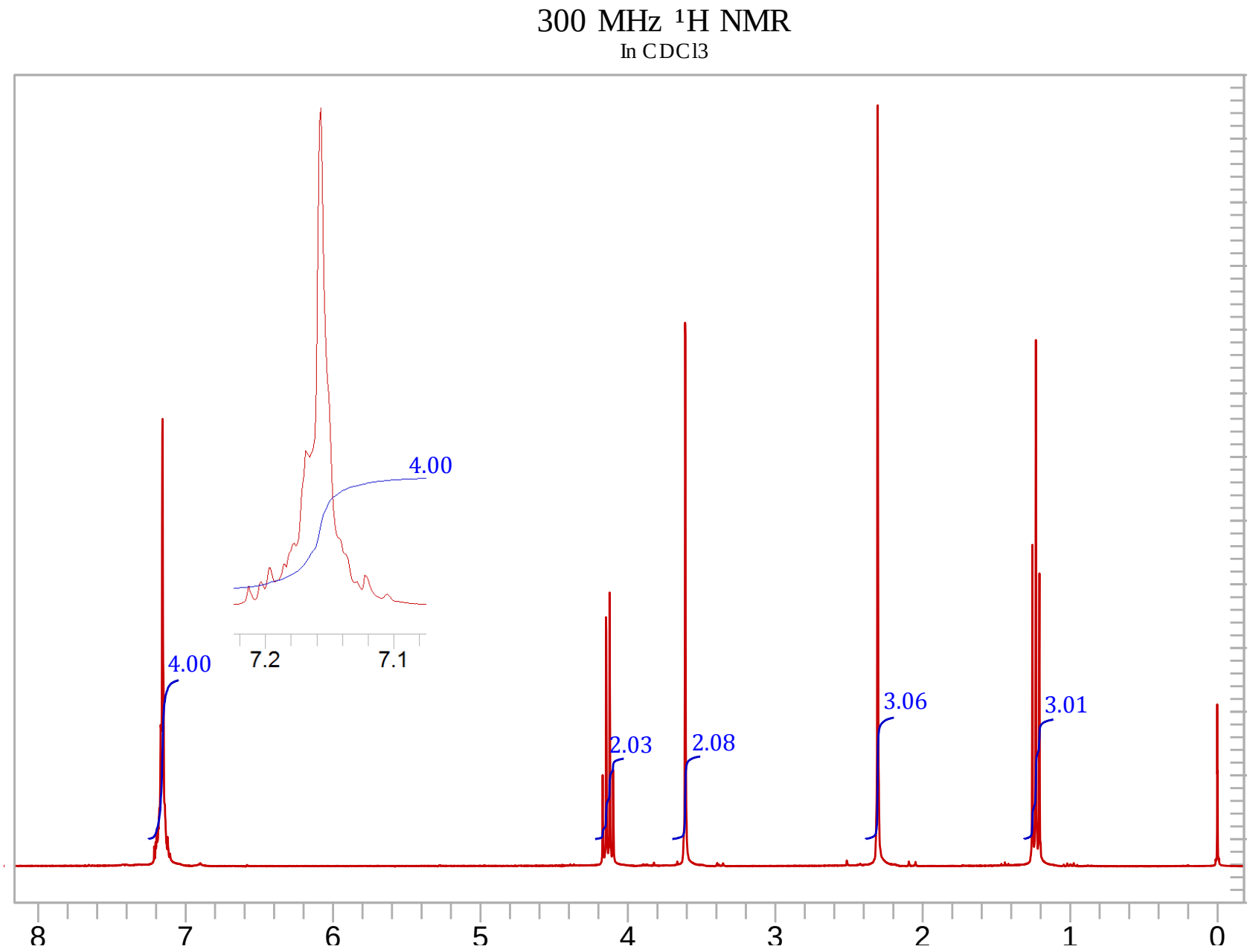


2959	7	1462	32	1286	79	1061	77	760	81
2928	4	1456	60	1268	79	1027	77	722	81
2869	18	1387	41	1196	66	940	77	662	41
2866	13	1378	31	1176	21	931	77	626	70
1698	66	1366	46	1133	77	890	67	683	70
1667	77	1360	62	1104	70	844	81	663	23
1666	81	1300	77	1079	74	766	81	640	28

3) Compound 3 ($C_{15}H_{23}ClO_2S$): EI-MS

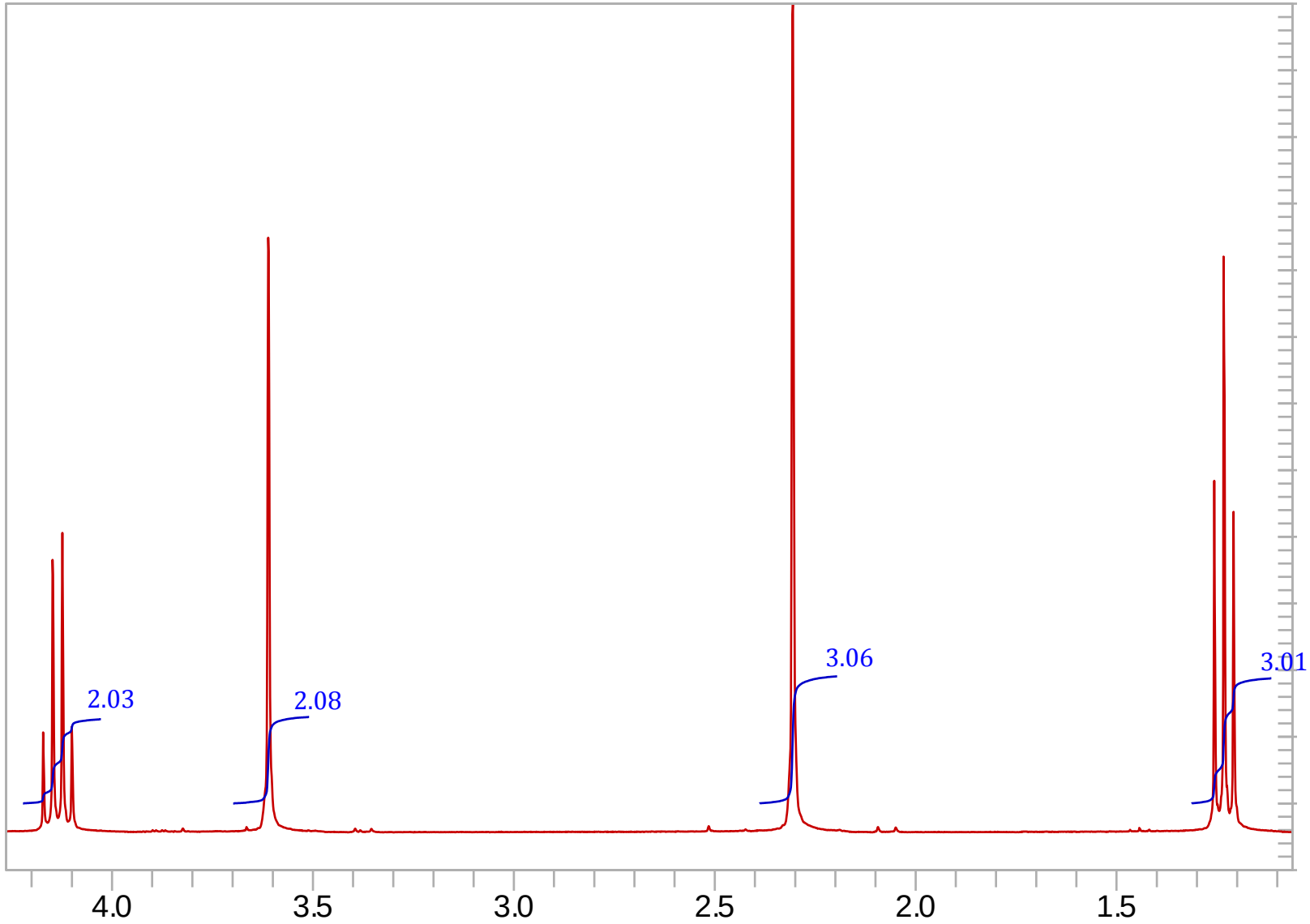


4) Compound 4 ($C_{11}H_{14}O_2$): ^{1}H-NMR

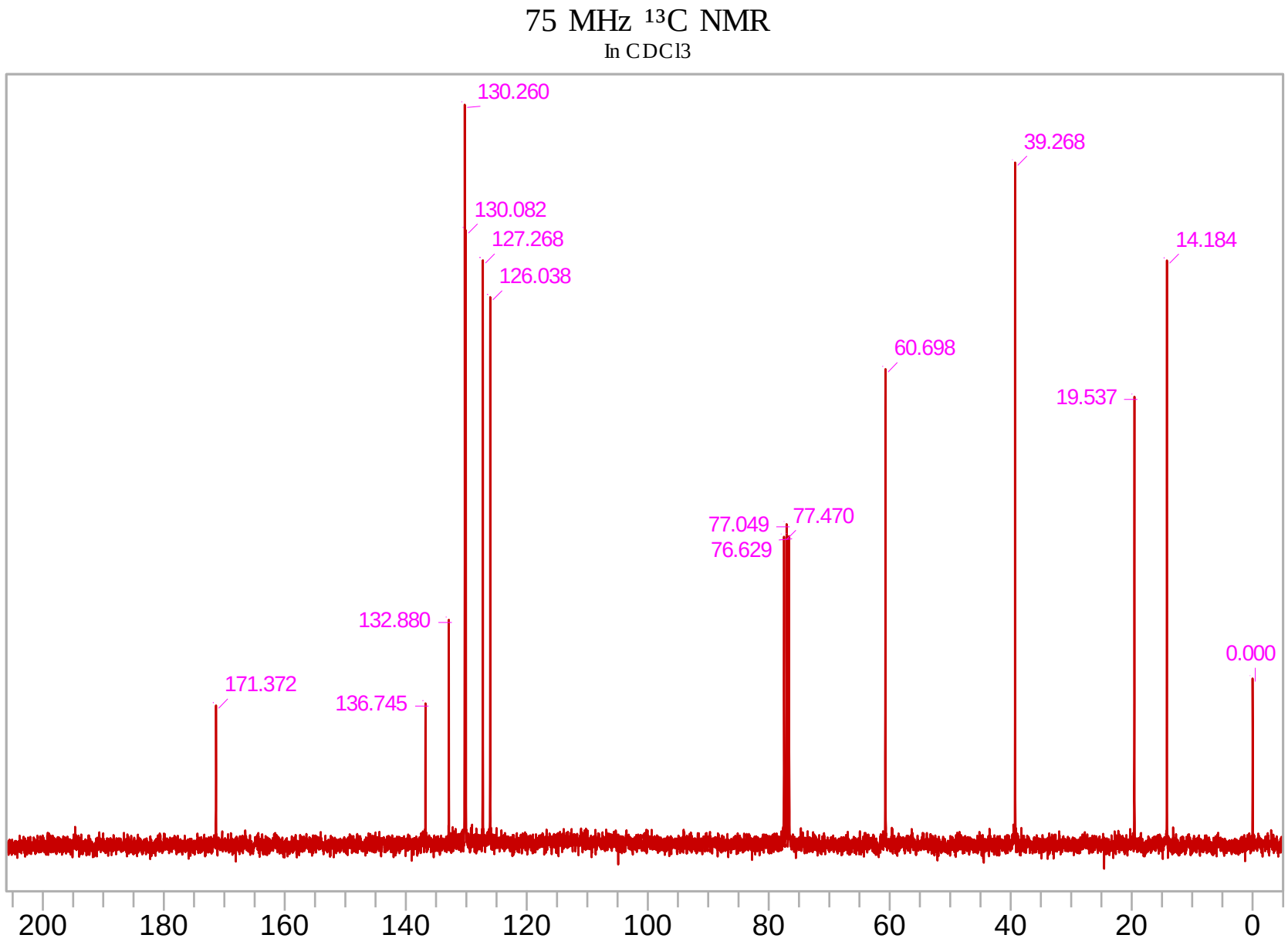


300 MHz ^{1}H NMR

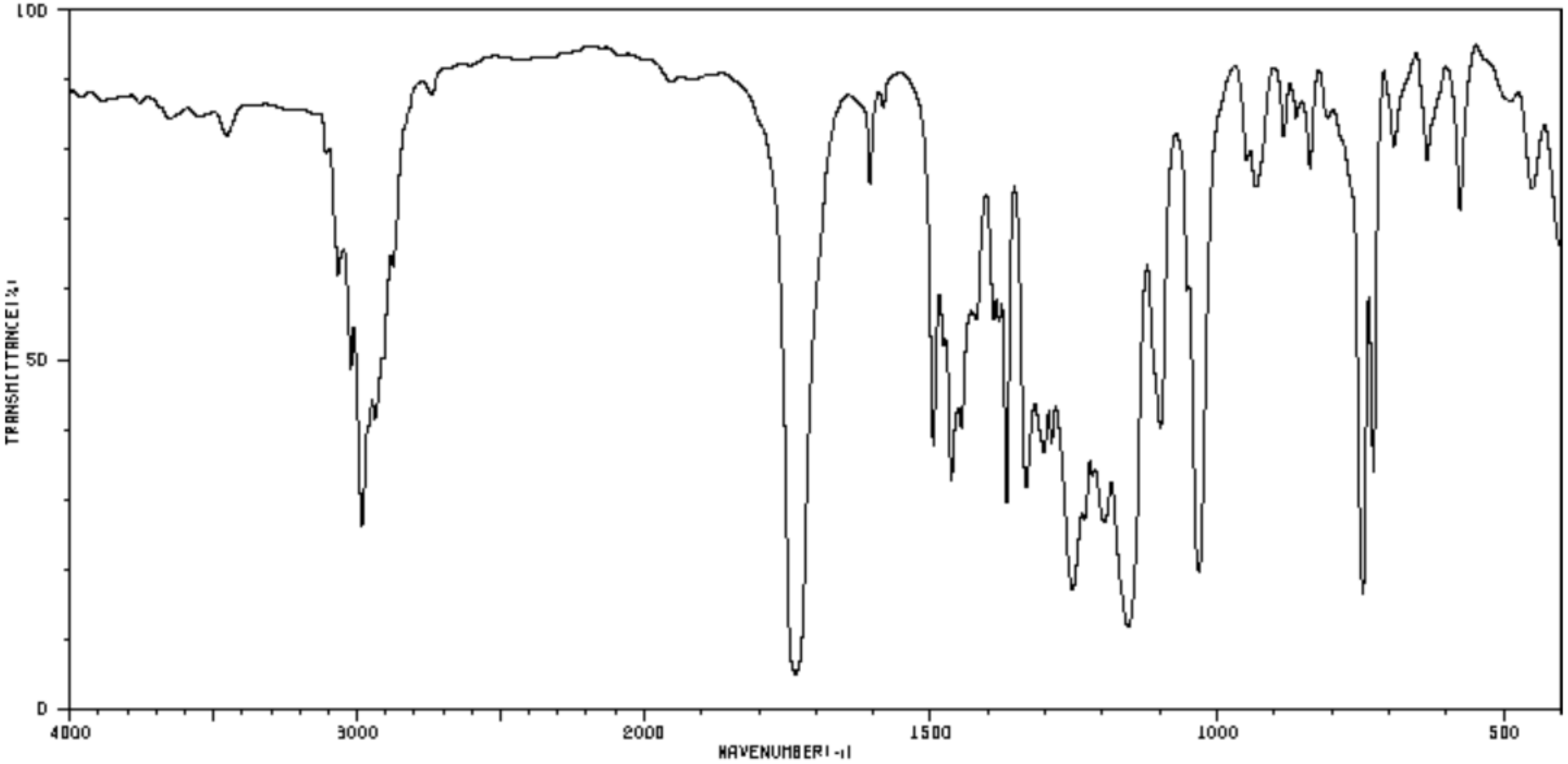
In CDCl3



4) Compound 4 ($C_{11}H_{14}O_2$): ^{13}C-NMR

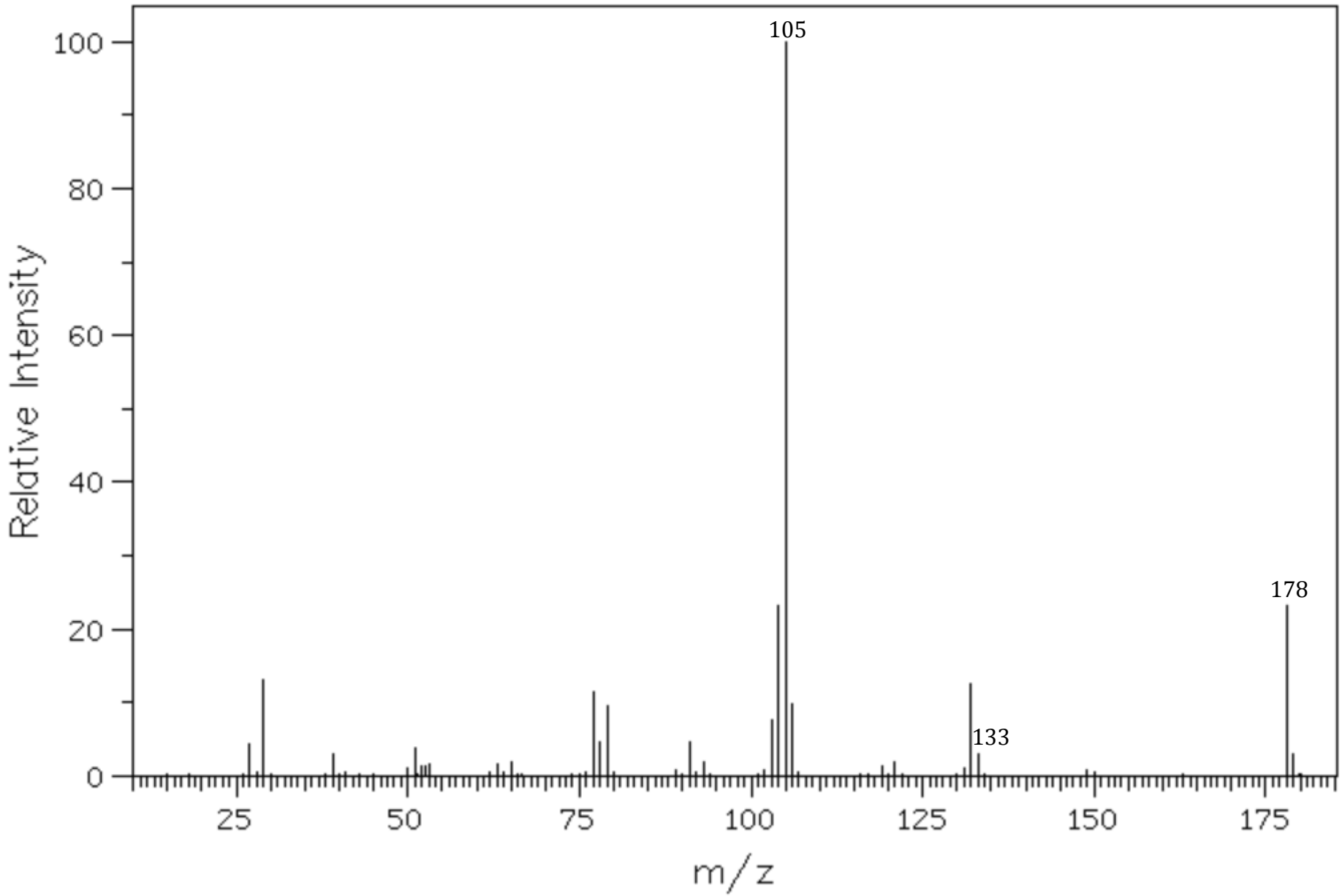


4) Compound 4 ($C_{11}H_{14}O_2$): IR



3647	81	1736	4	1381	63	1166	11	808	81
3454	79	1606	72	1368	28	1099	38	747	16
3066	80	1495	36	1334	30	1032	18	727	32
3021	46	1464	31	1302	36	949	74	693	77
2982	25	1446	38	1289	36	932	72	634	74
2936	39	1421	59	1253	16	885	79	577	68
2873	60	1391	69	1197	26	838	74	461	72

4) Compound 4 ($C_{11}H_{14}O_2$): EI-MS



Last Name (print): _____

First Name (print): _____

TA's Name: _____

CHEM 344

Summer 2014

Spectroscopy Problem Set

1) _____ **(10 pts)**

2) _____ **(12 pts)**

3) _____ **(14 pts)**

4) _____ **(14 pts)**

Total _____ **(50 pts)**

_____ **(math double-check)**