12BL Experiment 6 Prelab: NMR Analysis

1. Review the NMR Summary Powerpoint on our lab website under Techniques.

2. Explain if the following statement is true or not: "An NMR reports a signal for every H atom in a molecule."

3. What are equivalent H atoms? Give an example structure & circle equivalent Hs in groups.

4. What are unequivalent H atoms? Give an example structure & indicate the unequivalent Hs in some clear way.

5. Complete the following by filling in the number of neighbors where indicated AND circling equivalent or unequivalent.

a. H atom(s) that report a Singlet have ____ equivalent / unequivalent H neighbors.

b. H atom(s) that report a Triplet have ____ equivalent / unequivalent H neighbors.

c. H atom(s) that report a Multiplet have equivalent or unequivalent H neighbors?

d. H atom(s) that report a Doublet have ____ equivalent / unequivalent H neighbors.

e. H atom(s) that report a Sextet Multiplet have ____ equivalent / unequivalent H neighbors.

6. Draw any molecule that contains a Sextet Multiplet as one of its NMR signals. Use an arrow to point to the H(s) that are giving this signal.

7. Draw the structures for the following including ALL H atoms. Label the number of signals that would be reported on an NMR spectrum for each molecule.

Molecule	Structure	# Signals
2-butanone		
Propanoic acid		
Tert-butyl alcohol		
Pentanal		
Methyl butyrate		
Phenyl Chloride		

8. Directly on the structures in Question 6, use arrows to point to each UNIQUE H atom, and indicate the splitting pattern that would be observed for its signal (ie: singlet, doublet, etc…). *Do not point to every single H….. only each UNIQUE H!

12BL Experiment 6: NMR Analysis

Day 1 NMR: You may work individually or with ONE partner. But remember, YOU need to be confident as an individual as Day 2 NMRs and your Quiz are completely on your own.

After completing the packet, you are required to check the packet with your instructor. The instructor will mark any incorrect spectrums. You need to fix them and then re-check with the instructor. The instructor will Sign-Off once the packet is completely correct. Make Sure You Get Your Instructor's Signature Before Leaving!

***NMR Analysis Directions: READ carefully to ensure that you do all that is required!**

1. Every NMR must be completely analyzed. What does this mean?

   a. You are given the molecular formula – always calculate your degrees of unsaturation – sometimes it is very useful!

   b. Point with an arrow to each signal & label as singlet, doublet, etc…
   *****These are real NMRs – to help determine the splitting pattern the computer has drawn thin black lines extending up from the thick lines to make counting easier. Any "funnies" will be labeled.**

   c. Also next to each signal, label what the splitting pattern means…
   "This signal represents H(s) with _____ equivalent neighbors."
   "This multiplet represents H(s) with unequivalent neighbors."

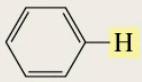
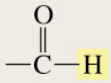
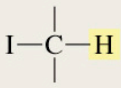
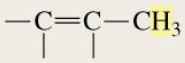
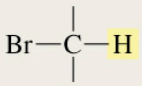
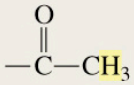
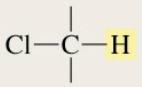
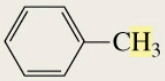
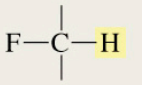
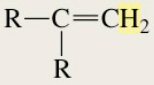
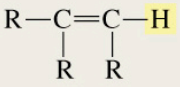
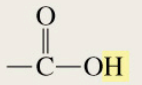
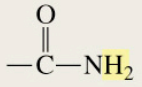
   d. And finally next to each signal, label what the approximate chemical shift indicates…
   "~11-12ppm indicates H of a carboxylic acid; ~2-2.2ppm indicates H(s) near a carbonyl bond."

2. Once you have a, b, and c completed above, you need to determine the structure of the molecule and make sure it is drawn on the NMR and CIRCLED.

3. Always, Check your Work! Does it make sense? Do the expected signals of the structure you drew actually match the NMR? Did I calculate my degrees of unsaturation from my given molecular formula?

***Make sure you learn your Chemical Shifts!**

Table 14.1 Approximate Values of Chemical Shifts for ^{1}H NMR[a]

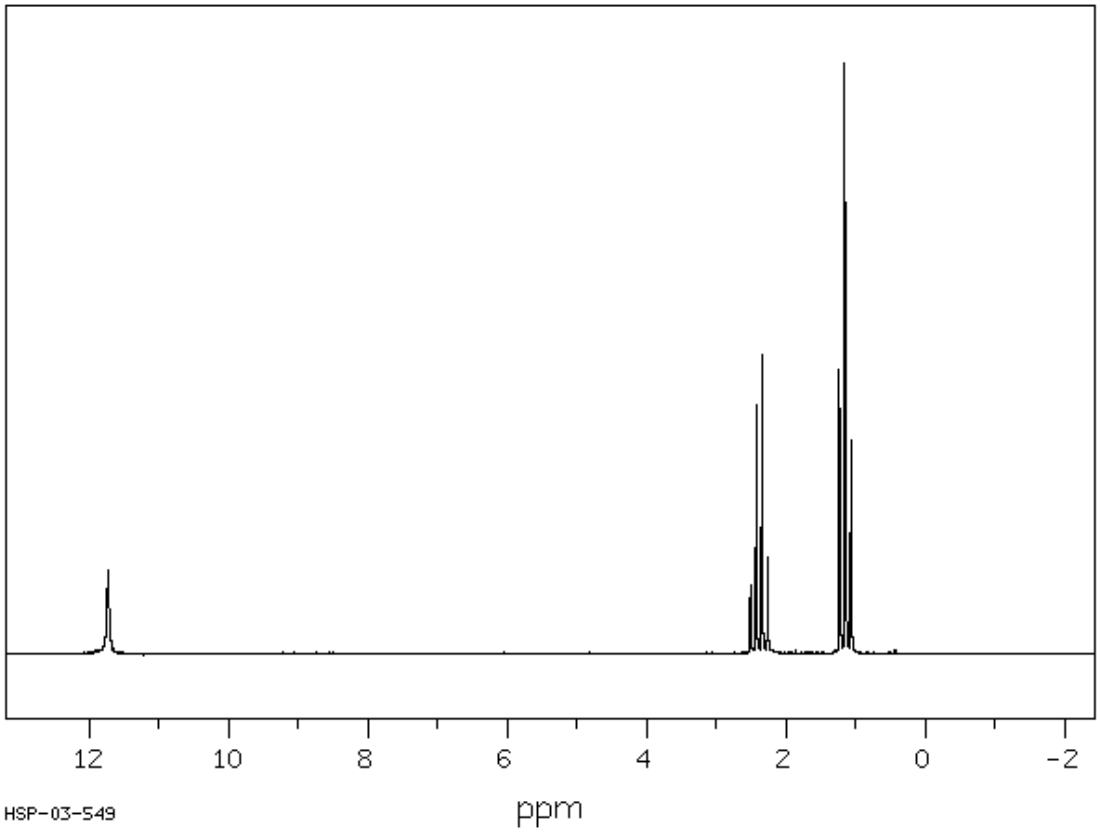
Type of proton	Approximate chemical shift (ppm)	Type of proton	Approximate chemical shift (ppm)
$(CH_3)_4Si$	0		6.5–8
$-CH_3$	0.9		
$-CH_2-$	1.3		9.0–10
	1.4		2.5–4
	1.7		
			2.5–4
	2.1		
			3–4
	2.3		
			4–4.5
$-C\equiv C-H$	2.4		
$R-O-CH_3$	3.3	RNH_2	Variable, 1.5–4
	4.7	ROH	Variable, 2–5
		ArOH	Variable, 4–7
	5.3		Variable, 10–12
			Variable, 5–8

[a]The values are approximate because they are affected by neighboring substituents.

(Note: Most Spectrums Provided by SDBSWeb : http://sdbs.riodb.aist.go.jp National Institute of Advanced Industrial Science and Technology, March 2013)

NMR Day 1

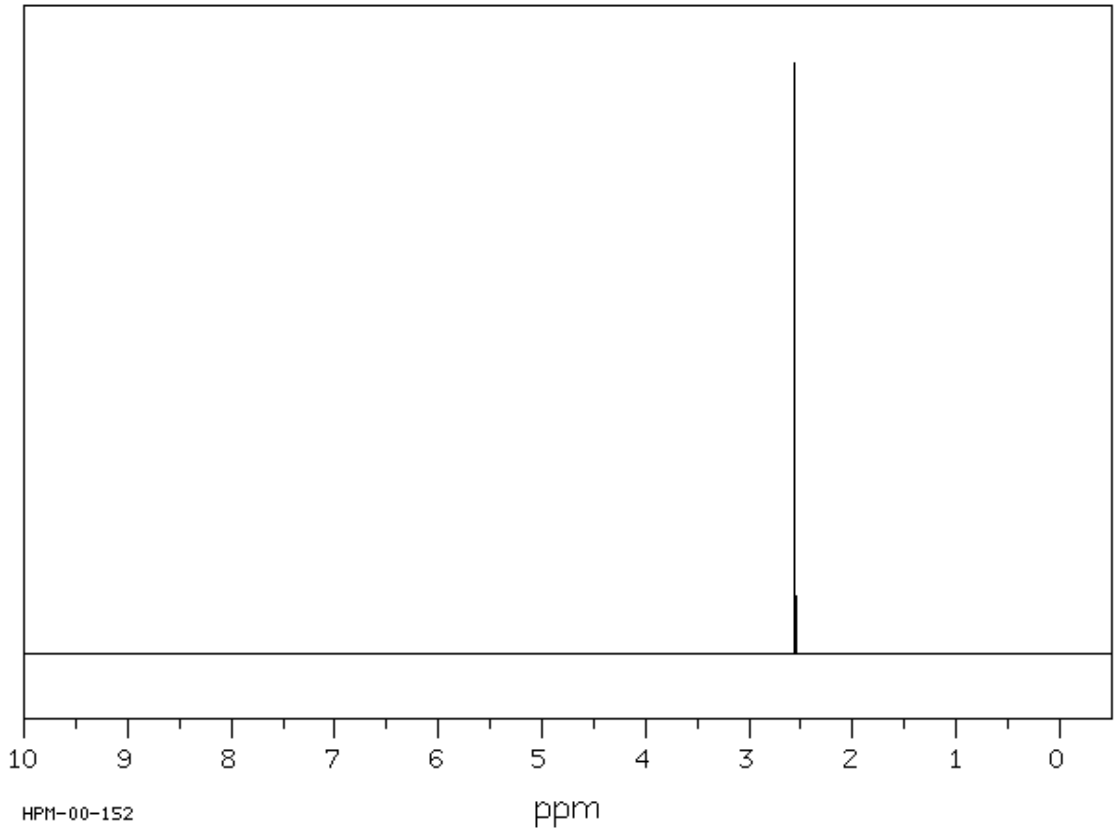
1) $C_3H_6O_2$



(signal at ~2.2ppm is quartet)

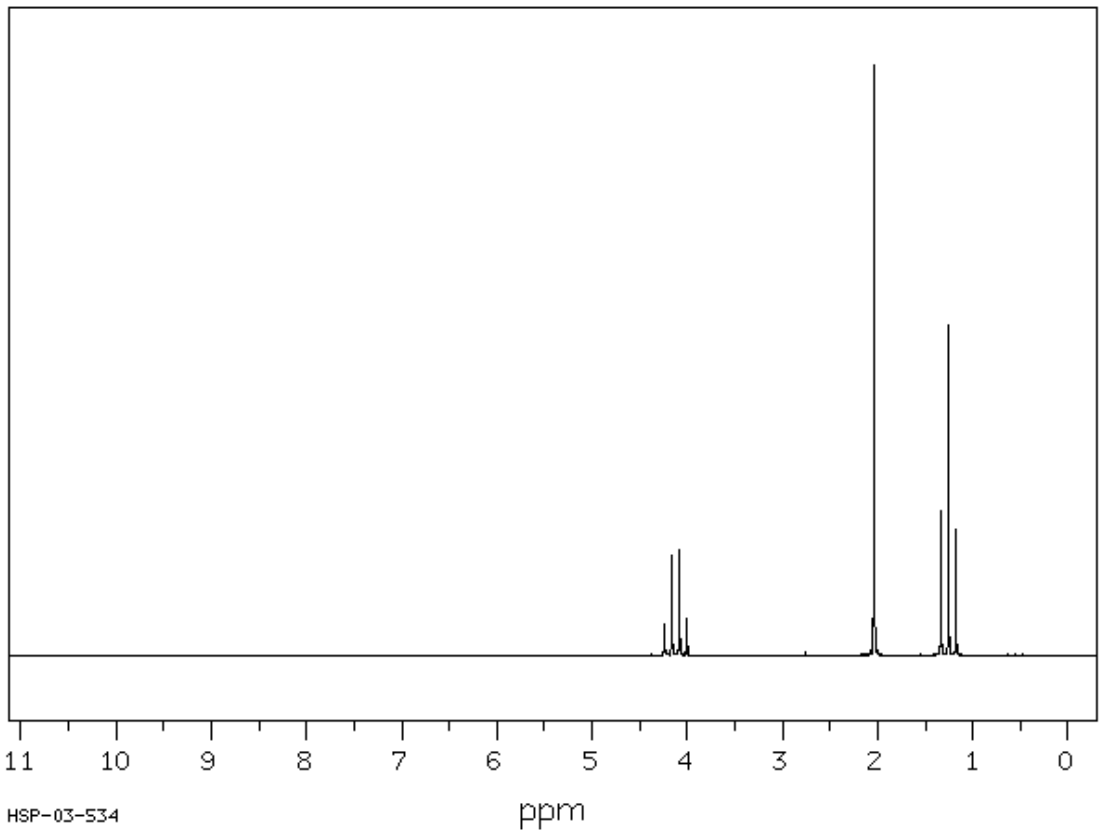
NMR Day 1

2) $C_3H_6Br_2$



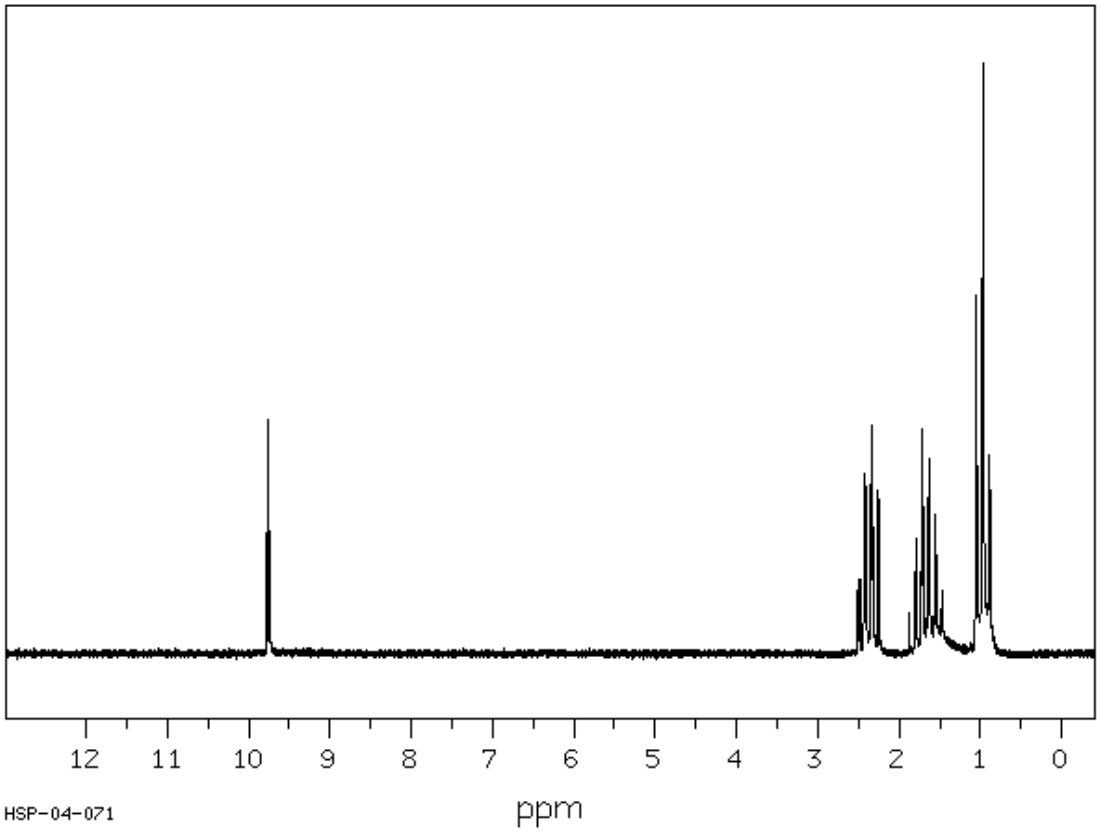
NMR Day 1

3) $C_4H_8O_2$



NMR Day 1

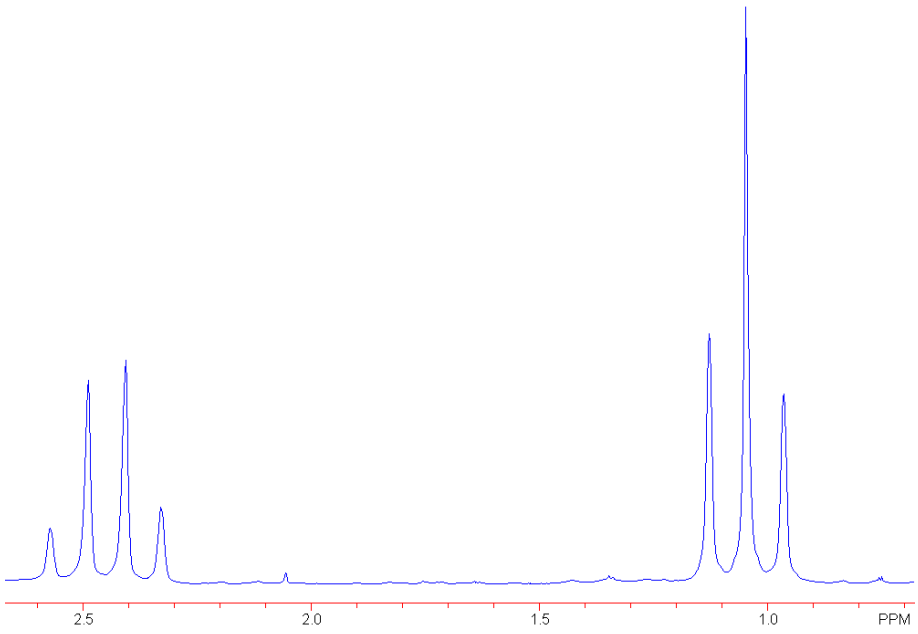
4) C_4H_8O



(signal at ~2.2ppm is considered a triplet; ~1.5ppm is considered a multiplet)

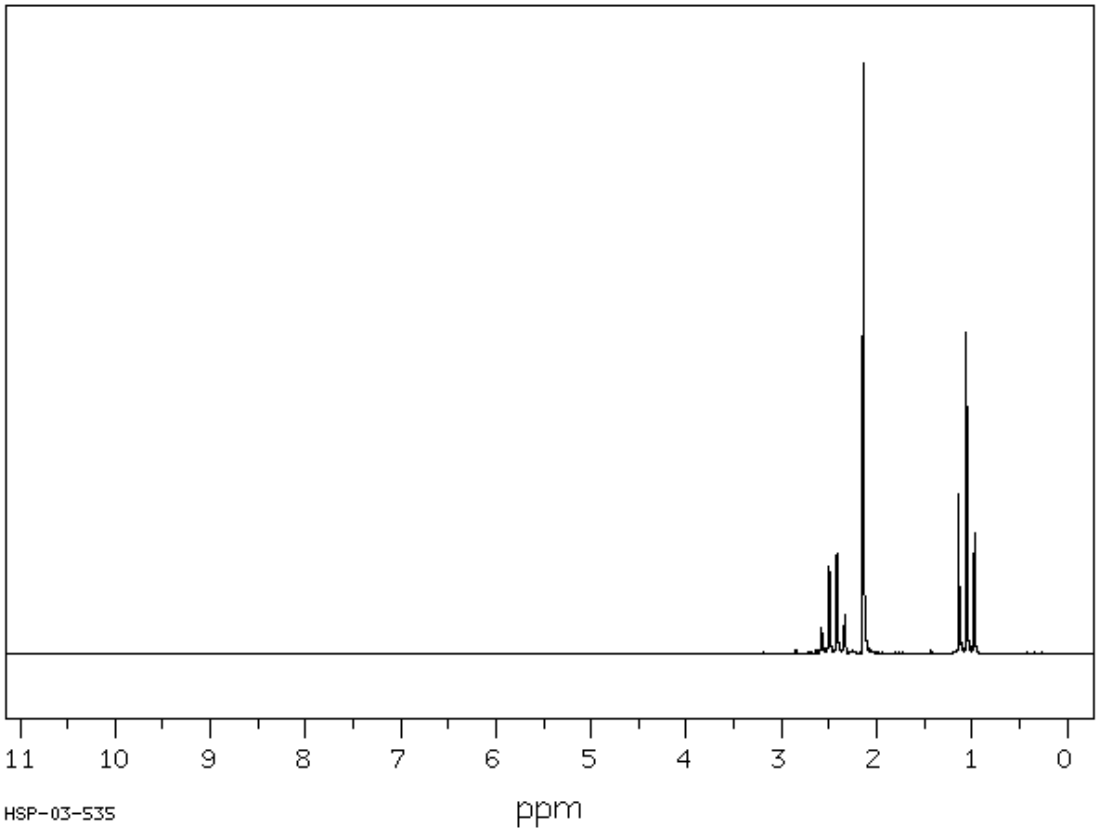
NMR Day 1

5) $C_5H_{10}O$



NMR Day 1

6) C_4H_8O



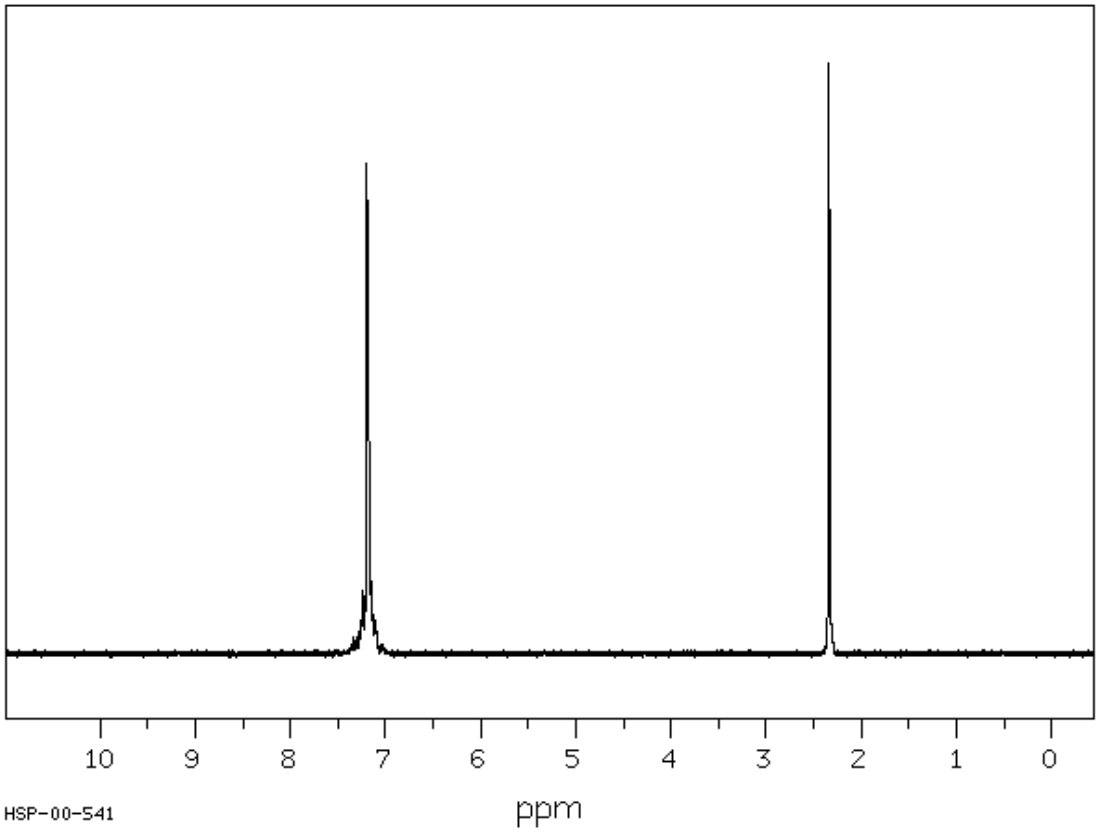
NMR Day 1

7) C_6H_{14}

Chemical Shifts Observed:	1.22	ppm	Quartet
	0.855	ppm	Singlet (very intense)
	0.84	ppm	Triplet

NMR Day 1

8) C_7H_8



(signal at ~7ppm is often referred to as a Multiplet)

12BL Experiment 6: NMR Analysis DAY 2 **VERSION 1**

Day 2 NMR: Check which Version is Assigned to you for in class analysis. The other version can serve as practice for you. **This is a completely INDIVIDUAL packet**. These spectrums must be completed in class, not prior! Once complete, turn into your instructor and you may leave for the day. This is a Graded packet so make sure you take your time and have clear complete work!

***NMR Analysis Directions: READ carefully to ensure that you do all that is required!**

1. Every NMR must be completely analyzed. What does this mean?

   a. You are given the molecular formula – always calculate your degrees of unsaturation – sometimes it is very useful!

   b. Point with an arrow to each signal & label as singlet, doublet, etc…
   *****These are real NMRs – to help determine the splitting pattern the computer has drawn <u>thin black lines extending up from the thick lines to make counting easier</u>. Any "funnies" will be labeled.**

   c. Also next to each signal, label what the splitting pattern means…
   "This signal represents H(s) with _____ equivalent neighbors."
   "This multiplet represents H(s) with unequivalent neighbors."

   d. And finally next to each signal, label what the approximate chemical shift indicates…
   "~11-12ppm indicates H of a carboxylic acid; ~2-2.2ppm indicates H(s) near a carbonyl bond."

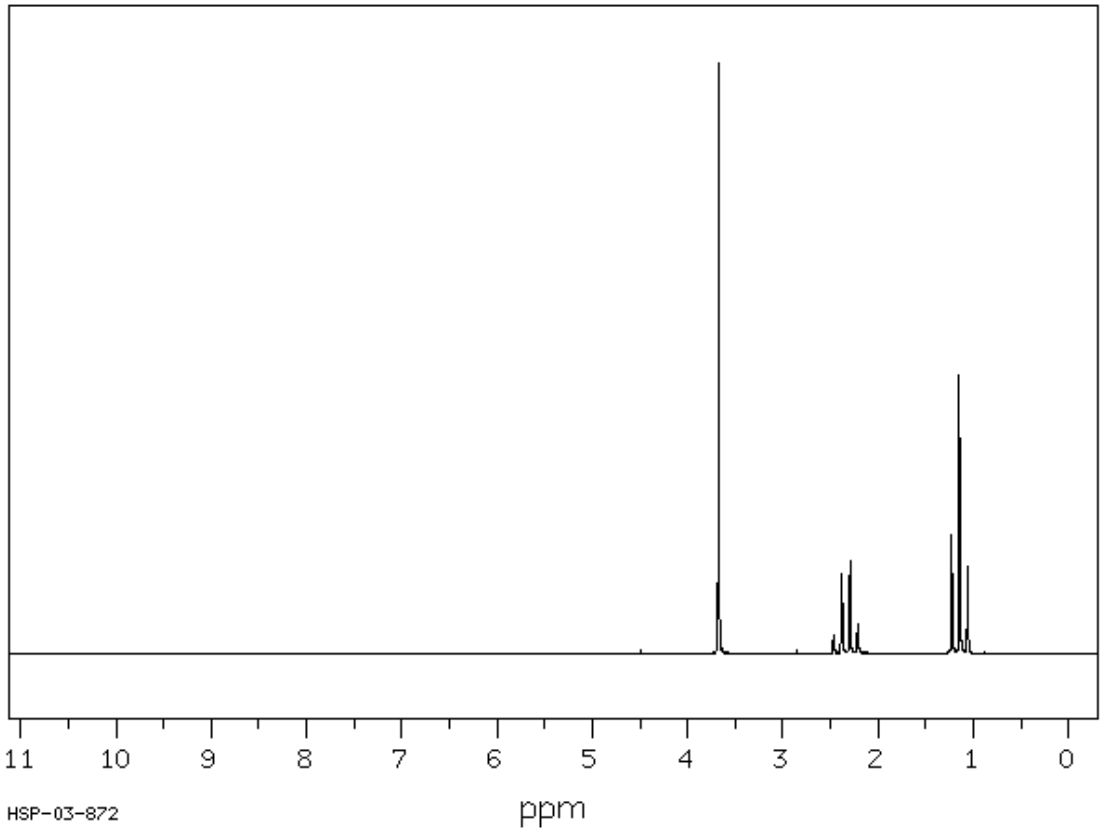
2. Once you have a, b, and c completed above, you need to determine the structure of the molecule and make sure it is drawn on the NMR and CIRCLED.

3. Always, Check your Work! Does it make sense? Do the expected signals of the structure you drew actually match the NMR? Did I calculate my degrees of unsaturation from my given molecular formula?

(Note: Most Spectrums Provided by SDBSWeb : http://sdbs.riodb.aist.go.jp National Institute of Advanced Industrial Science and Technology, March 2013)

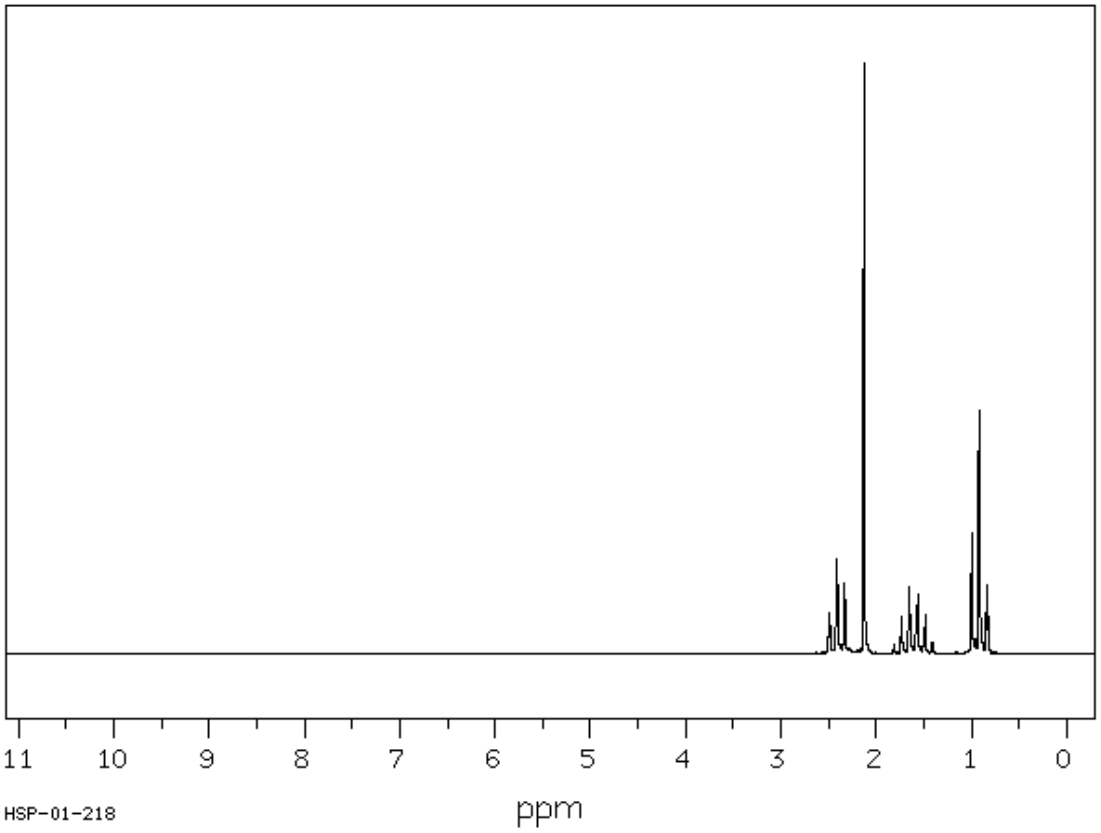
NMR Day 2 Version 1

1) $C_4H_8O_2$



NMR Day 2 Version 1

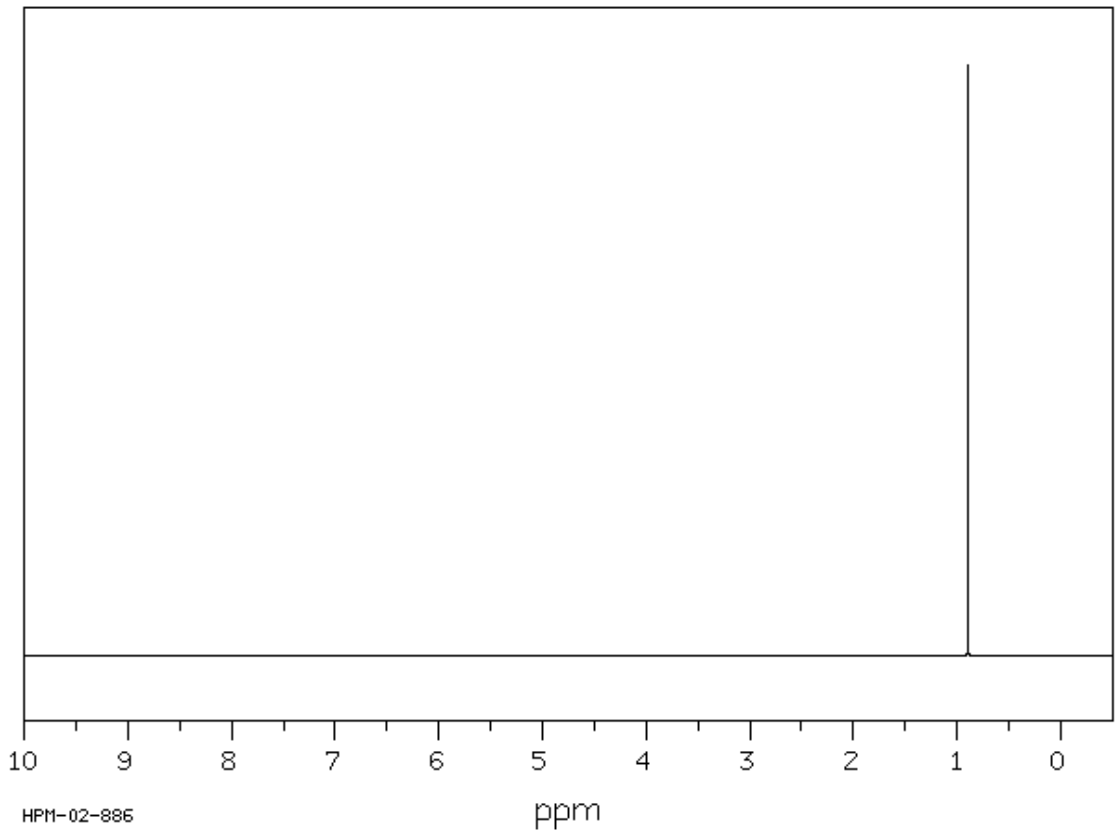
2) $C_5H_{10}O$



(signal at ~1.6ppm is a multiplet)

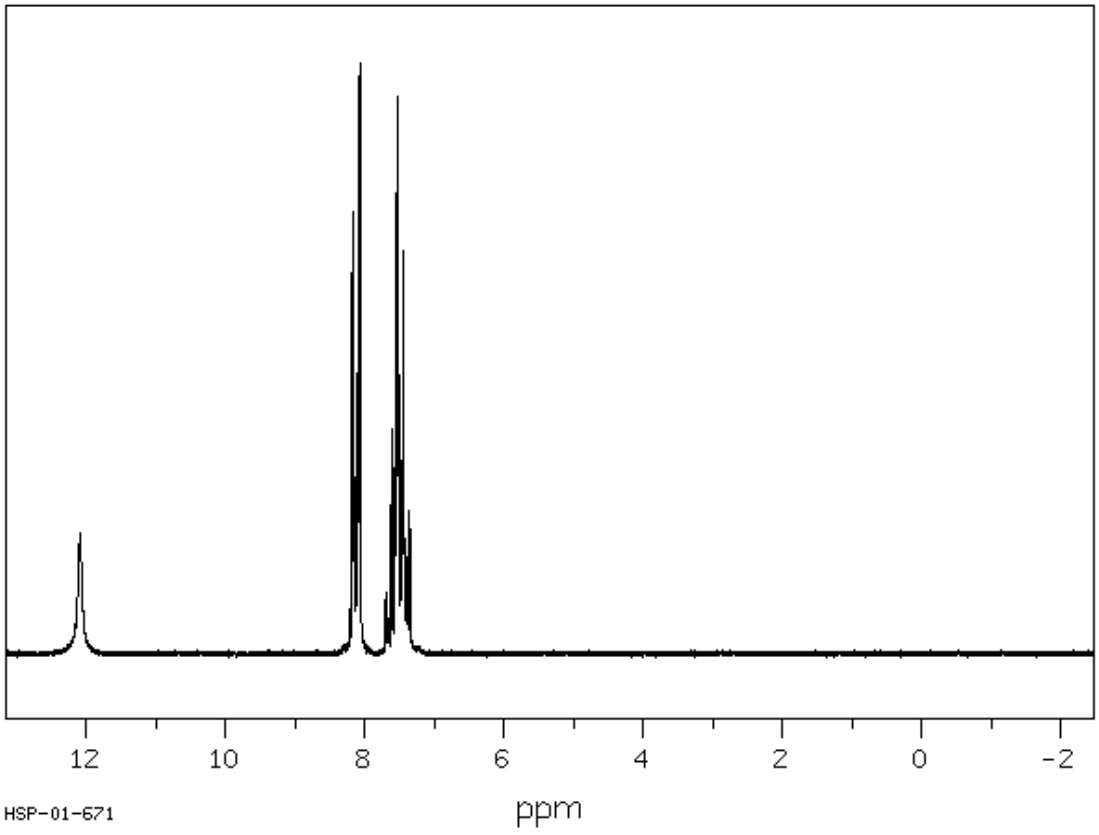
NMR Day 2 Version 1

3) C_5H_{12}



NMR Day 2 Version 1

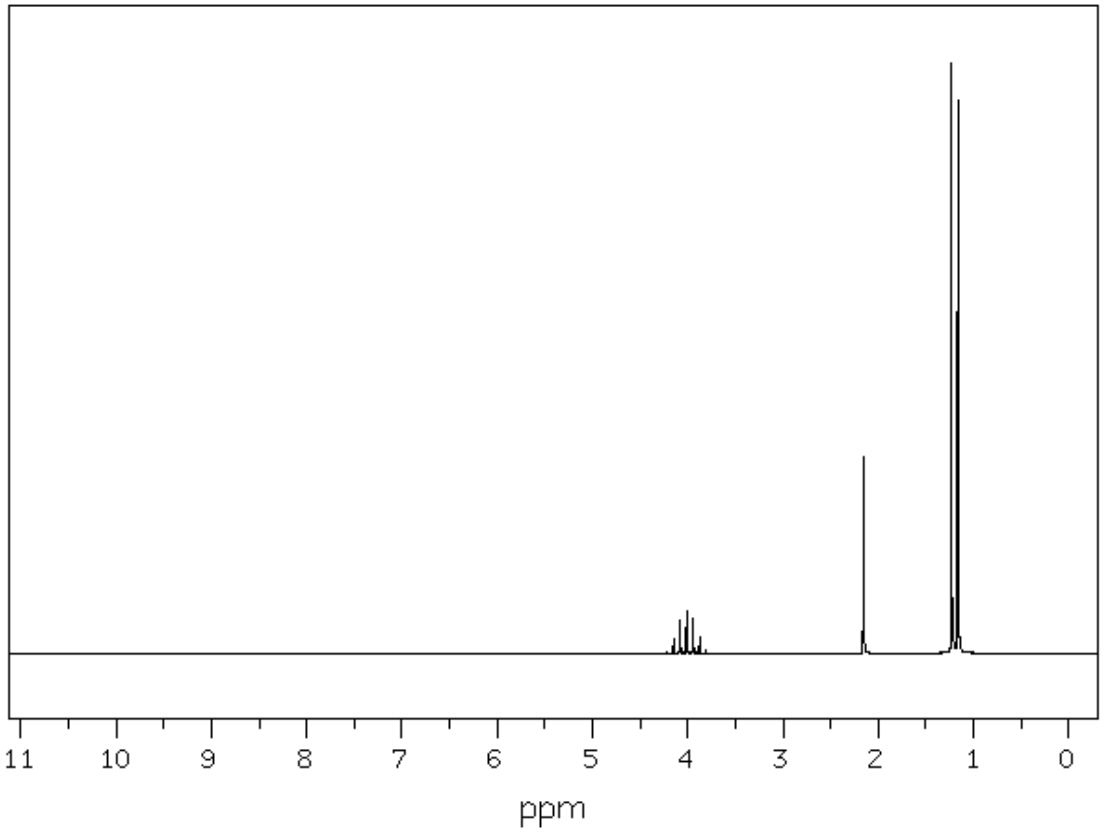
4) $C_7H_6O_2$



(signal at ~8.12ppm is a doublet; signals at ~7.5-7.9ppm are a ~triplet overlapped with a ~multiplet, respectively)

NMR Day 2 Version 1

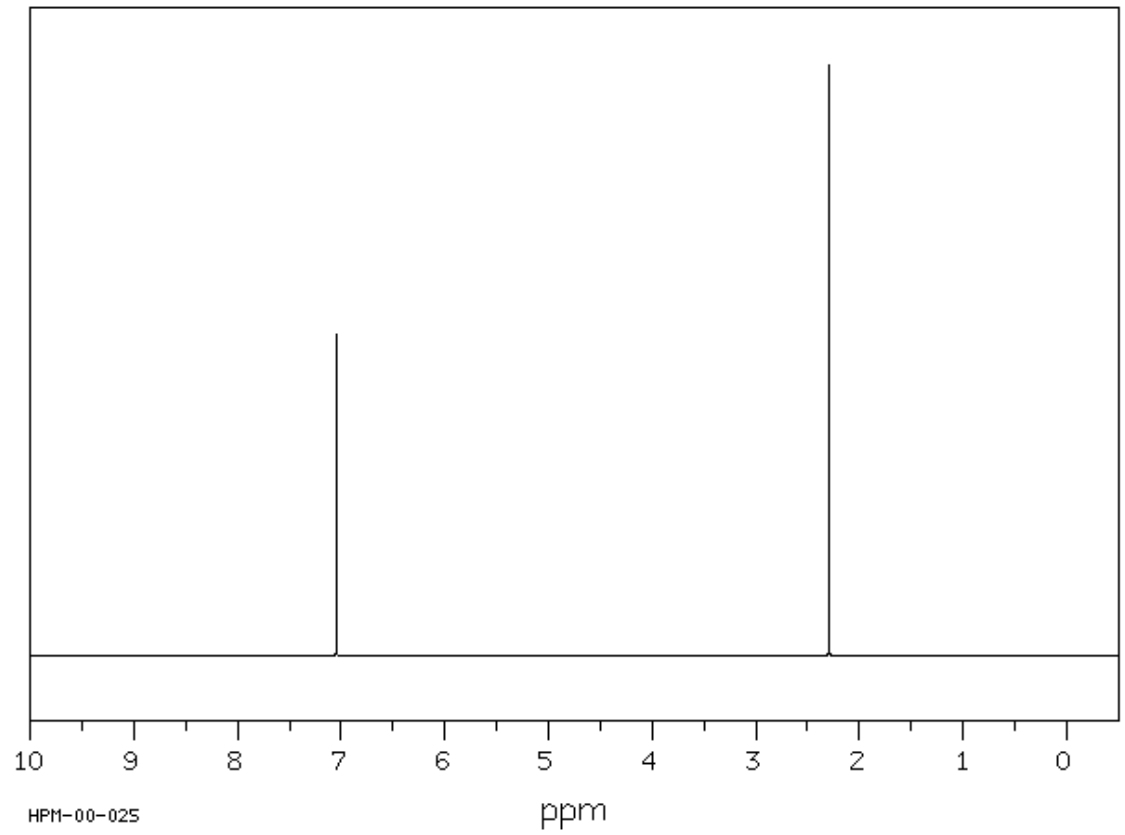
5) C_3H_8O



(signal at ~4ppm is a septet)
(Hint: It is known in H NMR spectroscopy that H atoms of aldehydes, alcohols, & amines rarely split any signals – in other words, these types of Hs are not influenced by neighboring Hs <u>nor</u> do they influence their neighbors.)

NMR Day 2 Version 1

6) C_8H_{10}



(Hint: The signal at ~7ppm is often mistakenly thought of as a doublet, but it is not. Remember, equivalent Hs do not split each other's signal)

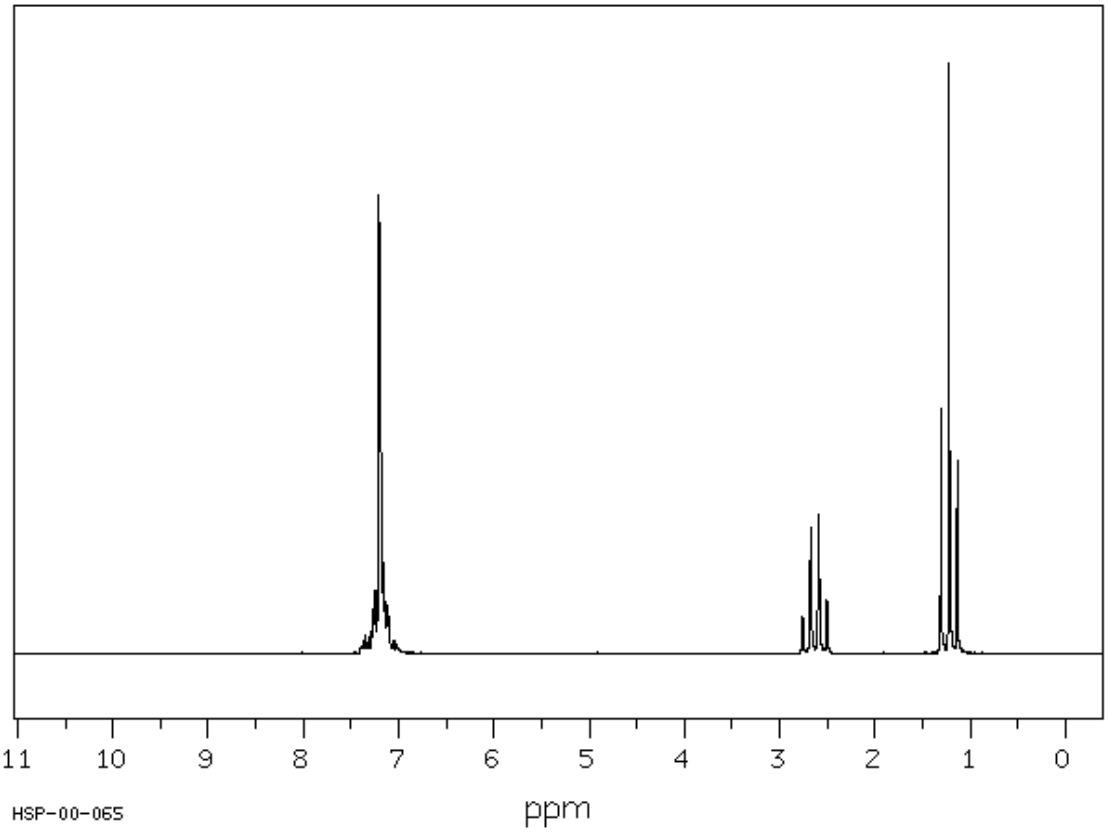
NMR Day 2 Version 1

7) $C_5H_{10}O_2$

Chemical Shifts:	4.13	ppm	Quartet
	2.30	ppm	Quartet
	1.26	ppm	Triplet
	1.14	ppm	Triplet

NMR Day 2 Version 1

8) C_8H_{10}



(signal at ~7ppm is a multiplet)

12BL Experiment 6: NMR Analysis DAY 2 **VERSION 2**

Day 2 NMR: Check which Version is Assigned to you for in class analysis. The other version can serve as practice for you. **This is a completely INDIVIDUAL packet**. These spectrums must be completed in class, not prior! Once complete, turn into your instructor and you may leave for the day. This is a Graded packet so make sure you take your time and have clear complete work!

***NMR Analysis Directions: READ carefully to ensure that you do all that is required!**

1. Every NMR must be completely analyzed. What does this mean?

   a. You are given the molecular formula – always calculate your degrees of unsaturation – sometimes it is very useful!

   b. Point with an arrow to each signal & label as singlet, doublet, etc…
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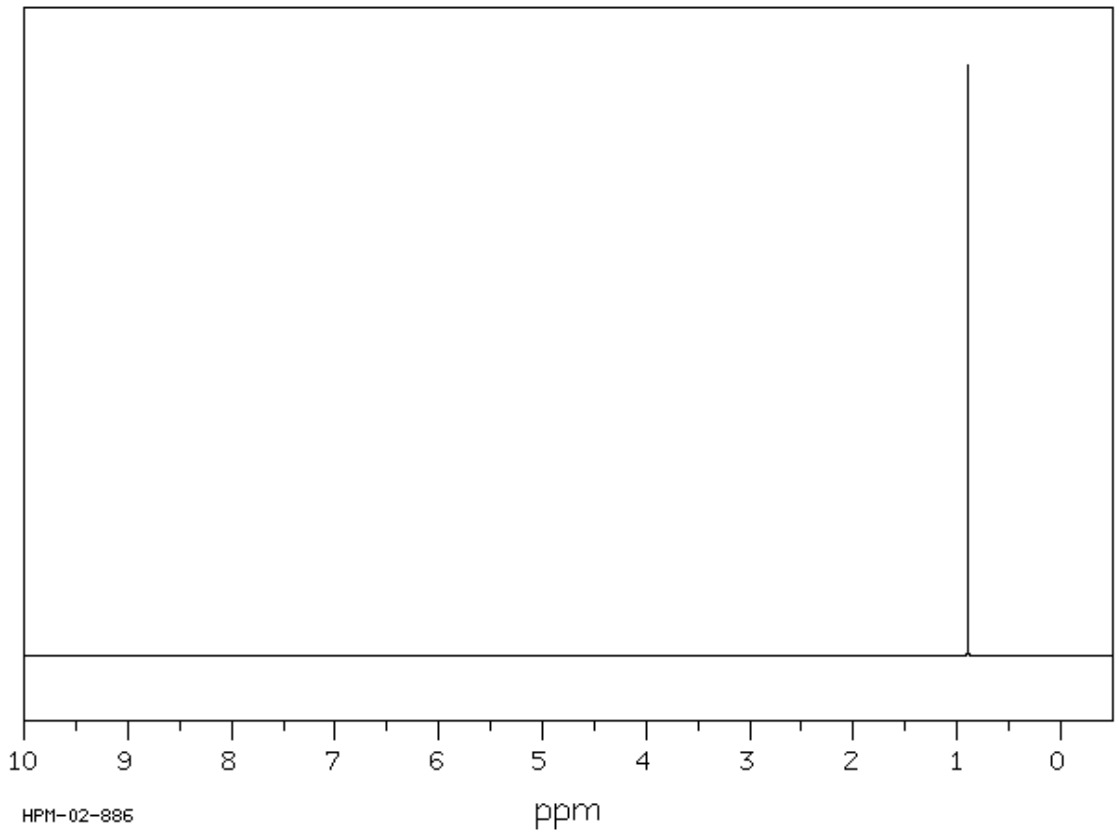
NMR Day 2 Version 2

1) C_5H_{12}



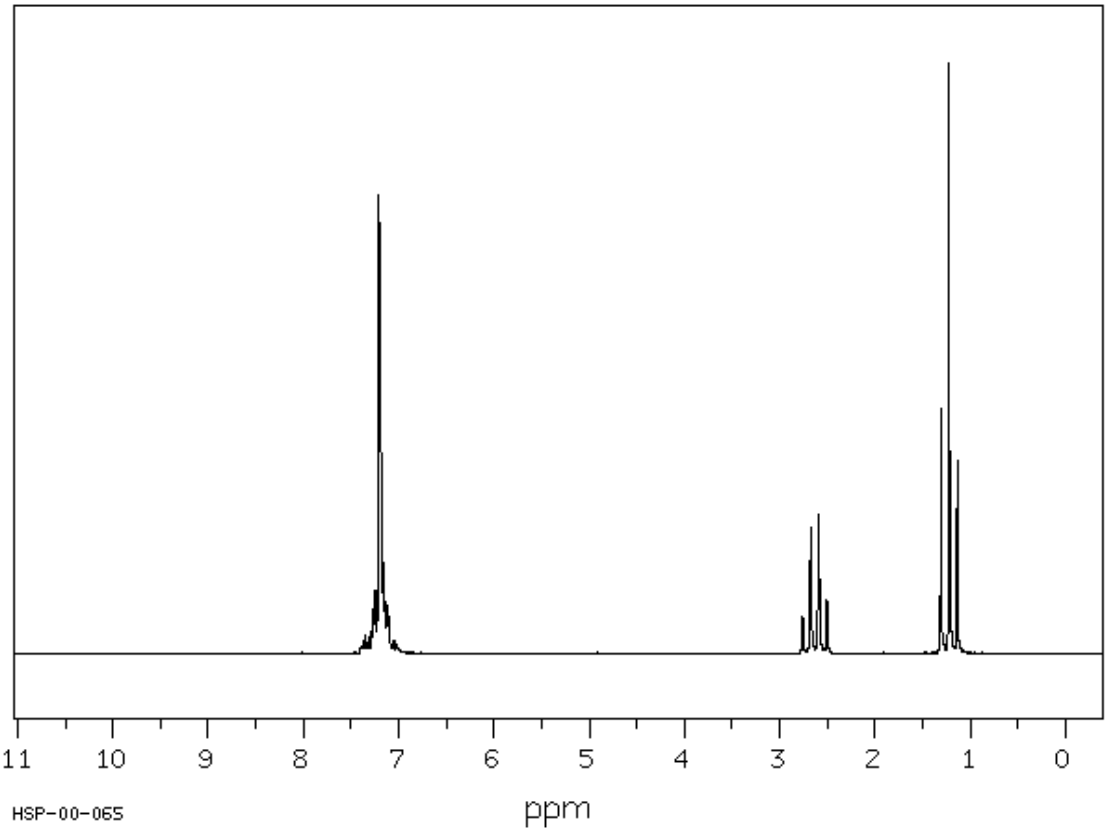
NMR Day 2 Version 2

2) $C_5H_{10}O_2$

Chemical Shifts:	4.13	ppm	Quartet
	2.30	ppm	Quartet
	1.26	ppm	Triplet
	1.14	ppm	Triplet

NMR Day 2 Version 2

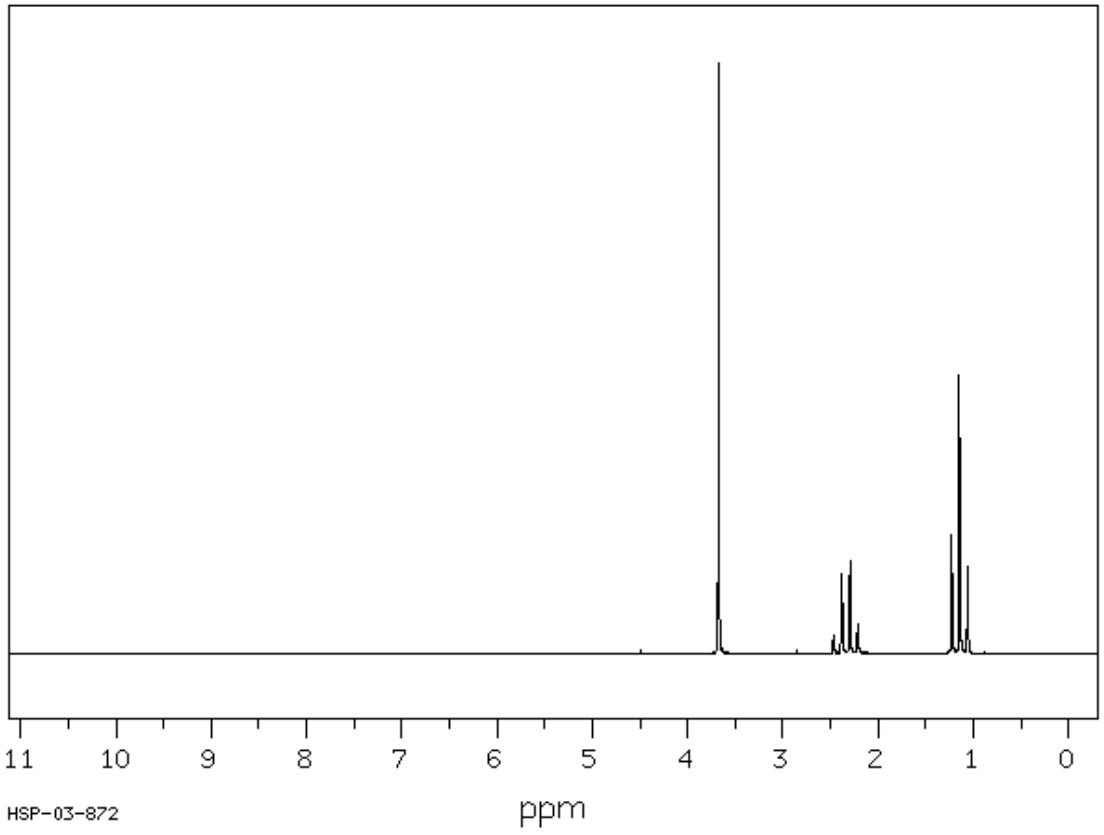
3) C_8H_{10}



(signal at ~7ppm is a multiplet)

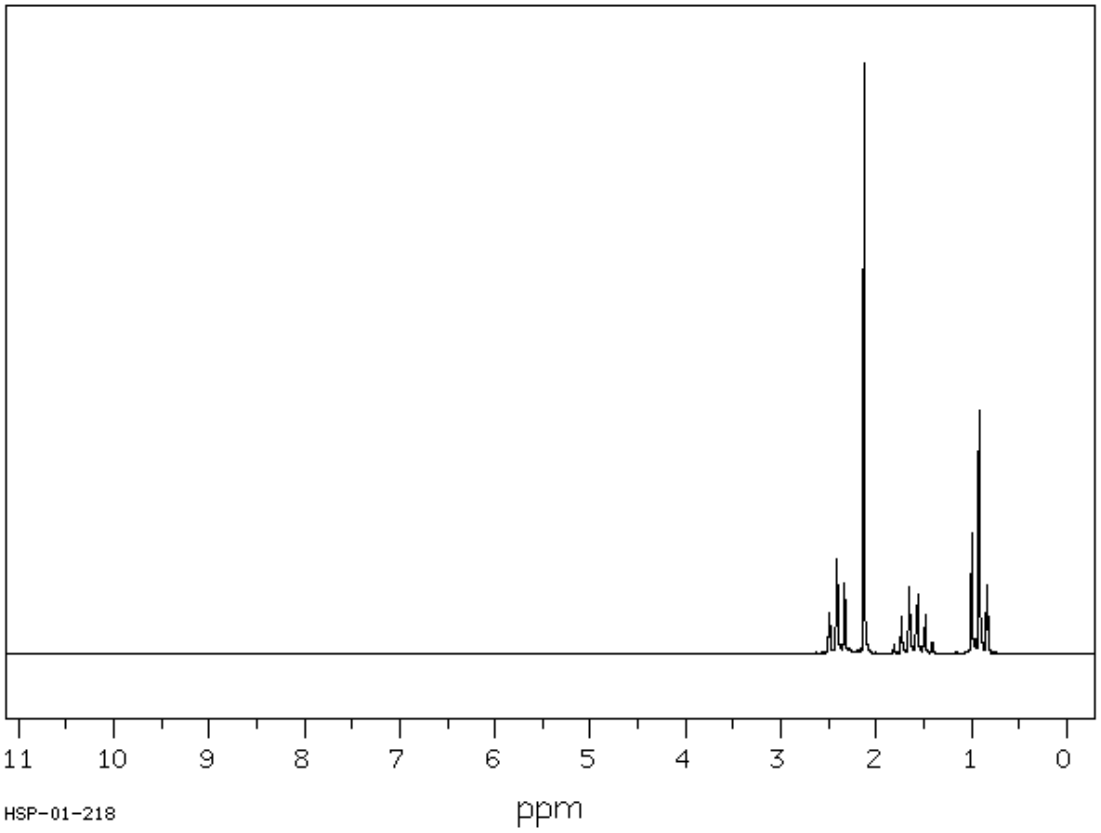
NMR Day 2 Version 2

4) $C_4H_8O_2$



NMR Day 2 Version 2

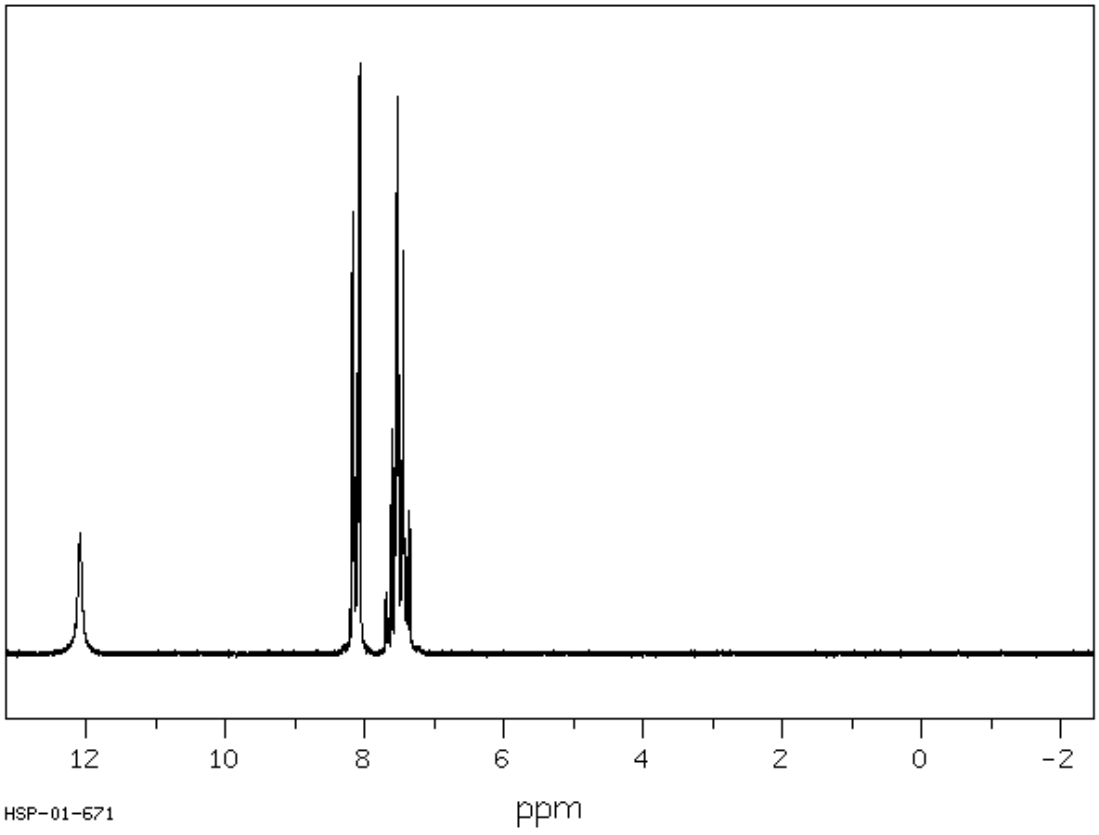
5) $C_5H_{10}O$



(signal at ~1.6ppm is a multiplet)

NMR Day 2 Version 2

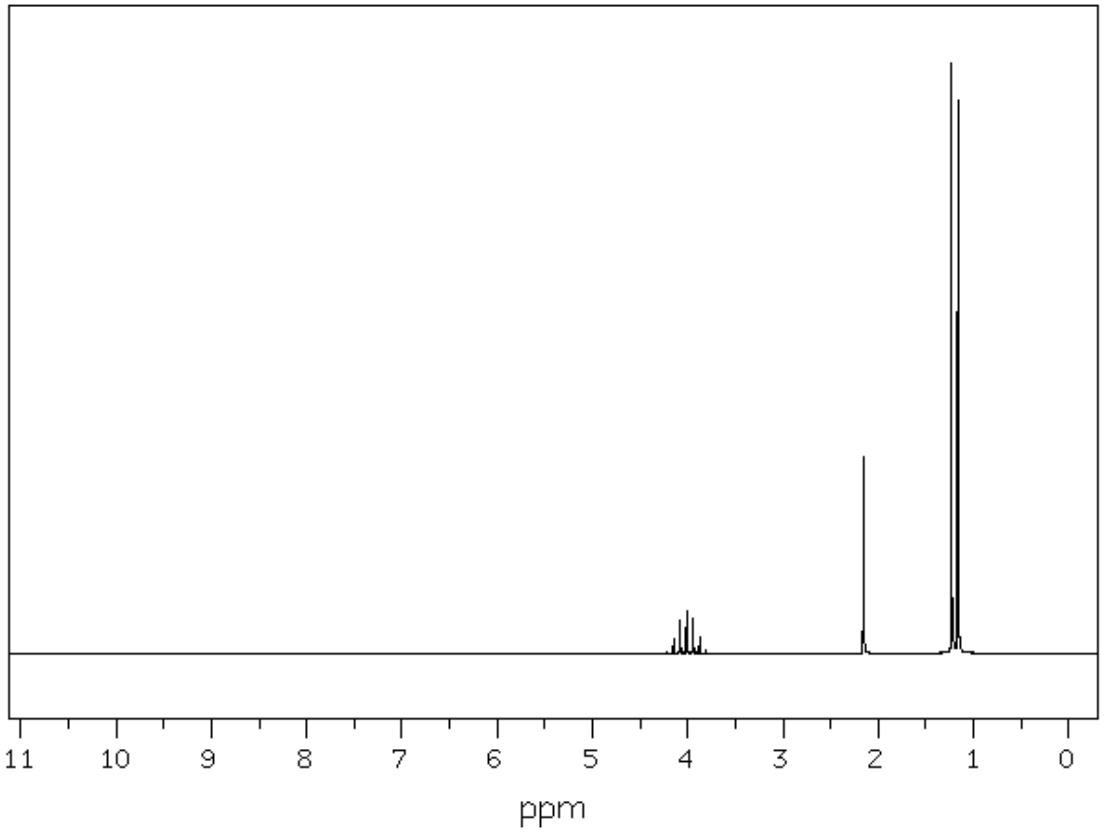
6) $C_7H_6O_2$



(signal at ~8.12ppm is a doublet; signals at ~7.5-7.9ppm are a ~triplet overlapped with a ~multiplet, respectively)

NMR Day 2 Version 2

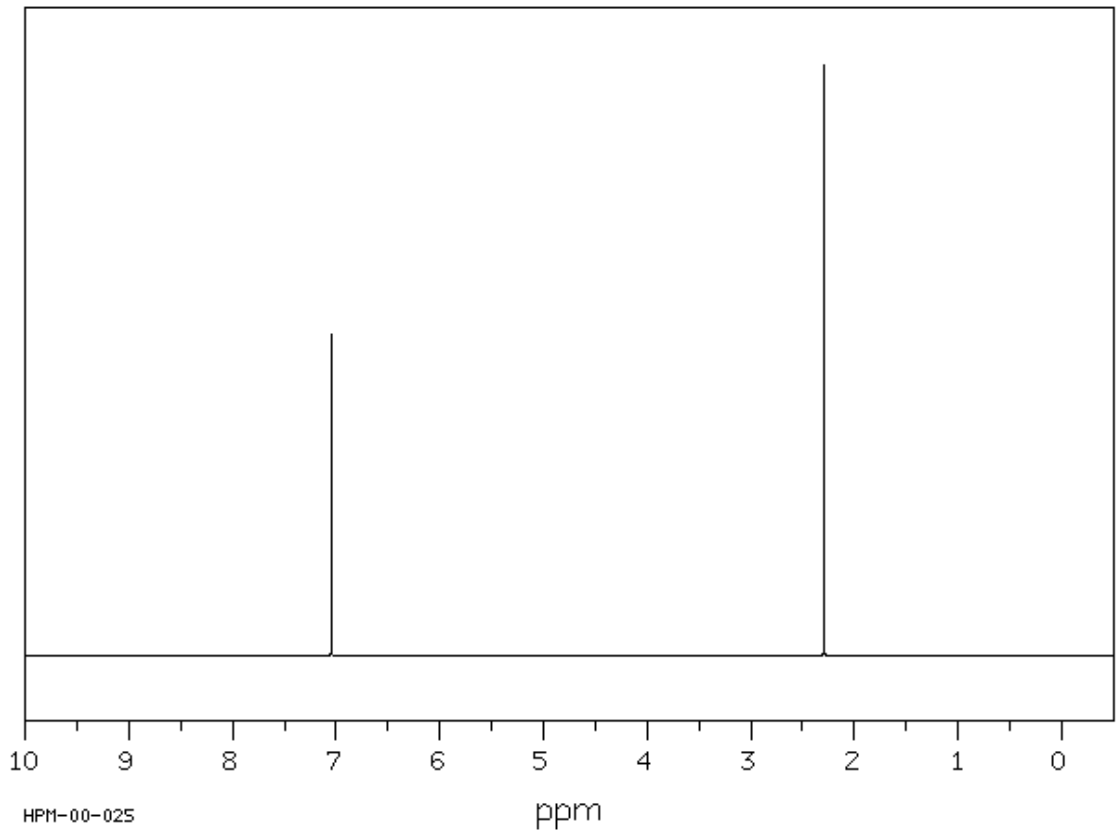
7) C_3H_8O



(signal at ~4ppm is a septet)
(Hint: It is known in H NMR spectroscopy that H atoms of aldehydes, alcohols, & amines rarely split any signals – in other words, these types of Hs are not influenced by neighboring Hs nor do they influence their neighbors.)

NMR Day 2 Version 2

8) C_8H_{10}



(Hint: The signal at ~7ppm is often mistakenly thought of as a doublet, but it is not. Remember, equivalent Hs do not split each other's signal)