

I. Introduction to NMR-218

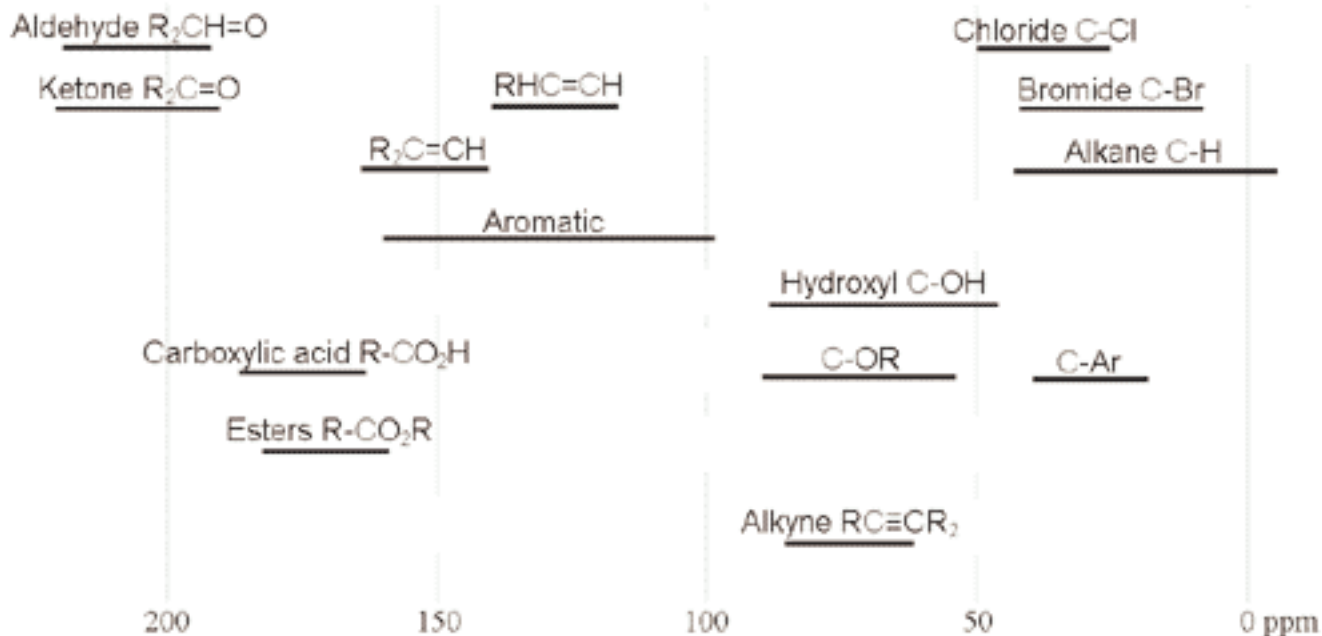
The different kinds of information one can get from an ordinary C-13 NMR spectrum can be classified in the following 3 ways: the number of different kinds of magnetically **different nuclei** in the compound, the **chemical shift** of the nuclei, and a rough indication of the number of absorbing nuclei via the **intensity** of the peaks. Each of these will be discussed in turn.

The **number of magnetically different nuclei** in a molecule will determine the number of peaks in the spectrum. For example in the C1 to C4 alcohols a spectrum that contains just two peaks in the carbon spectrum must either be t-butyl alcohol, (CH₃)₃COH, ethanol, or 2-propanol. Because all three C in the methyl groups of t-butanol are equivalent they will each show as a single peak in the carbon spectrum. How many different peaks will there be in the carbon spectra of methanol and t-butyl alcohol?

Chemical Shift- the property of the absorbing nuclei that results from the electronic environment around the atom. Measured in parts per million and **high** chemical shift numbers are associated with nuclei **low** electron density.

$$\delta = 1000000 \frac{(\text{ref } \nu - \text{sample } \nu)}{(\text{ref } \nu)} \text{ in ppm} = -1000000 \frac{(\text{ref } \nu - \text{sample } \nu)}{(\text{ref } \nu)} \text{ in ppm}$$

Carbon chemical shifts:



Intensity of the peaks.- Because of the long time it takes to quantitatively take a C-13 spectrum, carbon peaks are not normally integrated- a process that is done for proton NMR and will be discussed next week. For carbons with hydrogens attached to them, the intensity of the peaks should be a rough indication of the relative number of carbons giving that peak. Carbons without hydrogens attached to them will generally give lower intensity peaks. The reason for this is that these carbons do not enhanced by the Nuclear Overhauser Effect. More on that subject later.

Questions. Which one of the c1 to c4 alcohols will give just one peak in the carbon spectrum?

Which three of the c1 to c4 alcohols will give just two peaks in the carbon spectrum? How will you tell them apart?

Which two of the c1 to c4 alcohols will give three peaks in the carbon spectrum? How will you tell them apart?

Which two of the c1 to c4 alcohols will give four peaks in the carbon spectrum? How will you tell them apart?

NMR Report -small alcohol unknown

The possible compounds for this unknown are:

Methanol CH_3OH

Ethanol $\text{CH}_3\text{CH}_2\text{OH}$

1-Propanol $\text{CH}_3\text{CH}_2\text{CH}_2\text{OH}$

1-Butanol $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$

2-Propanol $(\text{CH}_3)_2\text{CHOH}$

i- Butyl alcohol $(\text{CH}_3)_2\text{CHCH}_2\text{OH}$

t-Butyl alcohol $(\text{CH}_3)_3\text{COH}$

2 Butanol $\text{CH}_3\text{CH}_2\text{CH}(\text{OH})\text{CH}_3$

Answer these questions on your spectra if possible. No additional sheets are necessary.

What is the known number _____ and compound.

How many different C's are present? (from the number of peaks in the C spectrum) How many C's are associated with each peak? (from the height of the peaks.

Attach the decoupled C spectrum (the normal C spectrum). Label the peaks A, B, etc corresponding to your answer above.

Draw the structure of the known alcohol and give its name and label the carbons in the structure so that the correspond to the table above.

What is the unknown number _____

How many different C's are present? (from the number of peaks in the C spectrum) How many C's are associated with each peak? (from the height of the peaks.

Attach the decoupled C spectrum (the normal C spectrum). Label the peaks A, B, etc corresponding to your answer above.

Draw the structure of the unknown alcohol and give its name and label the carbons in the structure so that the correspond to the table above.

Some facts about the EFT -60

Proton Frequency is 60 MHz (usually this frequency is in the name)

Carbon Frequency is 15 MHz (always about 1/4 the proton frequency).

Magnet is a permanent magnet.

The magnetic field is 1.38 Tesla or 13800 Gauss. (Proportional to the frequency of the proton).

Magnet temperature is 37 C or 310 K.

Directions for obtaining C13 Spectra on the EFT -60

0. Place sample in spinner to measured depth. Place in the nmr and turn the air off; then on. You should hear a "purr" at this point.

0. Enter the *PNMR* program.

0. Select C-13 parameters. <nu C13> Commands within <> are meant to be typed in the small command line towards bottom of the screen. End each command with an "enter". Parameters should be ns=16; rg= 100 for all neat samples and for the 3-96 n-Butanol standard. {If no one is waiting for the instrument, you can enter a large number for ns and then <zg> and got to steps 2 and3. Repeat 3 until the spectra "looks good then plot it. Hit Q in PNMR to stop data collection.} If you use an ns >64 you must wait until no one else has a sample to run. If you use ns >1000 you must wait until the evening.

1. Acquire data. <zg> The computer will ask for a file name. Hit return to use the default name. The FID should be above the base line and yellow.

2. Enter the *NUTS* program. Nut commands are just two letters in main program with NO following enter.

3. Process the data. [Ctrl F3].To use the default data file hit return when it asks for the data file Use line broadening of 2 when it asks. Line broadening LB=2.

4. Adjust scale by using scroll bar on right and phase if needed ai.

5. Plot the data [pl]

