**Introduction to NMR Spectroscopy:**

NMR Spectroscopy can be poorly understood concepts by undergraduate students. As it is critical for understanding the structure & behaviour of molecules, herein we highlight the key concepts for solving NMR spectra & simple sample elucidation.

**NMR Spectra Decoded:**

There are 3 basic types of information that can be garnered from $^1$H NMR spectra. It is important to consider all 3 pieces of information together or the interpretation will not be accurate.

1) **What type of protons you have.**

$^1$H NMR spectroscopy is based on the interaction of protons with magnetic fields. Protons interact with both the spectrometer’s magnetic field and local fields (i.e., surrounding electron density).

Because the local electronic environment is functional group dependent, protons of the same ‘type’ resonate at the same frequency (e.g., alkyl, alcohol, aldehyde). As in most types of spectra, resonance frequencies appear as ‘peaks’. The position of each peak is referred to as the *chemical shift* ($\delta$).

Peaks in a spectrum can be identified by comparing the observed $\delta$ with a reference chart, such as the one on back of this brochure. **TAKE HOME MESSAGE:** The position of the peak suggests which potential functional groups are present.

2) **How many protons of each type you have.**

The area under each peak (i.e., its *integral*) is proportional to the number of protons contributing to the signal.

3 important points: (a) integrals are relative values. They are not absolute so watch for symmetry in the molecule; (b) *exchangeable protons* (i.e., -OH, -NH$_2$) integrate low; (c) carbon ALWAYS has 4 bonds. Use of integrals can aid identification of the correct functional group. This will be illustrated in further detail later. **TAKE HOME MESSAGE:** When integration is taken in conjunction with the possible functional groups, you have more information regarding the structure framework.

3) **How the protons are connected.**

NMR signals may appear as 1 peak (singlet) or may be split into 2 (doublet), 3 (triplet), 4 (quartet), etc closely grouped peaks. This is referred to as *multiplicity, coupling* or *splitting pattern*.

(a) The *multiplicity* of a peak is governed by the number of H *neighbours* these protons ‘see’. Thus it tells you the connectivity of the protons within the molecule.

For the important case of protons (spin = 1/2), peak splitting follows the equation:

$$\text{multiplicity} = n + 1$$

where $n =$ # of equivalent H’s adjacent to signal protons

It is important to remember that the number & type of protons represented by a peak are irrelevant its own multiplicity.

(b) Lines split into multiplets appear at intensities covered by Pascal’s Triangle. If the intensities do not follow this pattern then likely you have overlapping signals - NOT coupling.

**Worked Example:**

Comparing with the $\delta$ table offers a number of potential functional groups. To narrow this down - apply your knowledge of Lewis structures, the octet rule & other types of spectroscopy (e.g., mass or IR spectra). This will become easier with practice.
3) Proton Connectivity: Now that you have pieces, use splitting patterns to connect them.

- H NMR Spectrum
- Singlet = no coupling information
- ~ 1:3:3:1 quartet
  - n = splitting: 1 = 4 - 1 = 3
  - So: CH₂ bonded to CH₂

Now connectivity is trivial. You have two terminal groups (i.e., -C₄H₄ & -CH₃ can only form one bond) & one linking group (-CH₂-). Double check to make sure coupling agrees.

Data Accessibility:
NMReady outputs to a networked drive and has a print option. Students can process and print in third party software, like Mestrelab™, or use the NMReady directly. An example of data to be incorporated into a lab report processed and printed directly from the NMReady is presented below:

References:

Procedure:
Measure a spectrum for each pre-prepared 30% v/v 1-propanol & 2-propanol sample (labelled “isomer 1” & “isomer 2”) on the NMReady by inserting the sample and hitting “Go”. Workup each spectrum (i.e., baseline correct, peak pick & integrate) as instructed by your TA. Use what you’ve learned above to distinguish between the two isomers.

(ii) For each isomer, how many types of protons would you expect?
(iii) How many types do you see in each spectrum (i.e., how many peak groups are there if you ignore multiplicity)?
(iv) Look at chemical shift of each peak - what possible functional group does each peak represent?
(v) What integrations would you expect for each isomer?
(vi) Do the predicted values agree with observed integrations?
(vii) Propose “puzzle pieces” of the molecule using experimental information.
(viii) Assign the splitting patterns of each peak.
(ix) Propose a structure for each spectrum to distinguish between the unknown isomers.

For additional ideas of how to incorporate the NMReady™ benchtop spectrometer into undergraduate laboratories please see:

1) Synthesis of Aspirin
2) Aldol Condensation
3) Biodiesel
available at:
www.nanalysis.com/experiments.html