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NMR Facilities
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NMR NEWS October 2008

* To check on the **instrumental status** and the **reservation system**, please, visit our website www.chem.utk.edu/nmr

* Do you want to save time? ...then run 2D experiments!

It is true that a 1D-^{1}H spectrum takes only few minutes to be acquired. A 2D spectrum, on the other hand, with the exception of a gcosy, would require 20 minutes or more to be completed. However, as it will be shown below, 2D experiments give accurate information and much faster than 1D-^{1}H spectra. Two-dimensional NMR makes it easier to assign the lines of a 1D-proton spectrum, and is a robust method for structure elucidation and confirmation of small organic molecules.

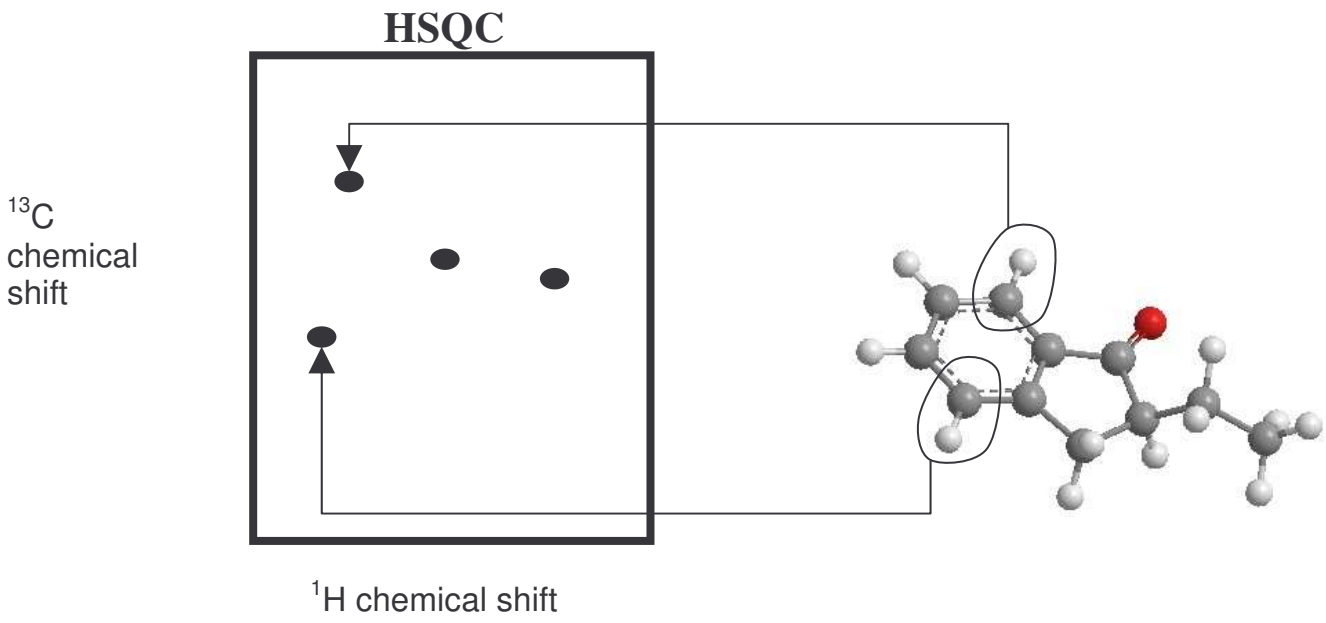
I hope that what it is explained below will make you think whether spending a couple of hours of instrument time (not even your time!) is worth the time **you** will save in the analysis of the data.

In this issue, I will introduce the experiment that you should always try first, the ^{13}C-HSQC.

What is an ^{13}C-HSQC spectrum? As shown in the sketch below, it is basically a xy plot containing peaks that correspond to the protons bonded to carbon of your molecule. The positions (x,y) of these peaks have coordinates,

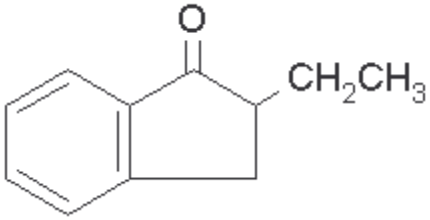
x = ^{1}H chemical shift of the proton,

y = ^{13}C chemical shift of the carbon attached to the proton.

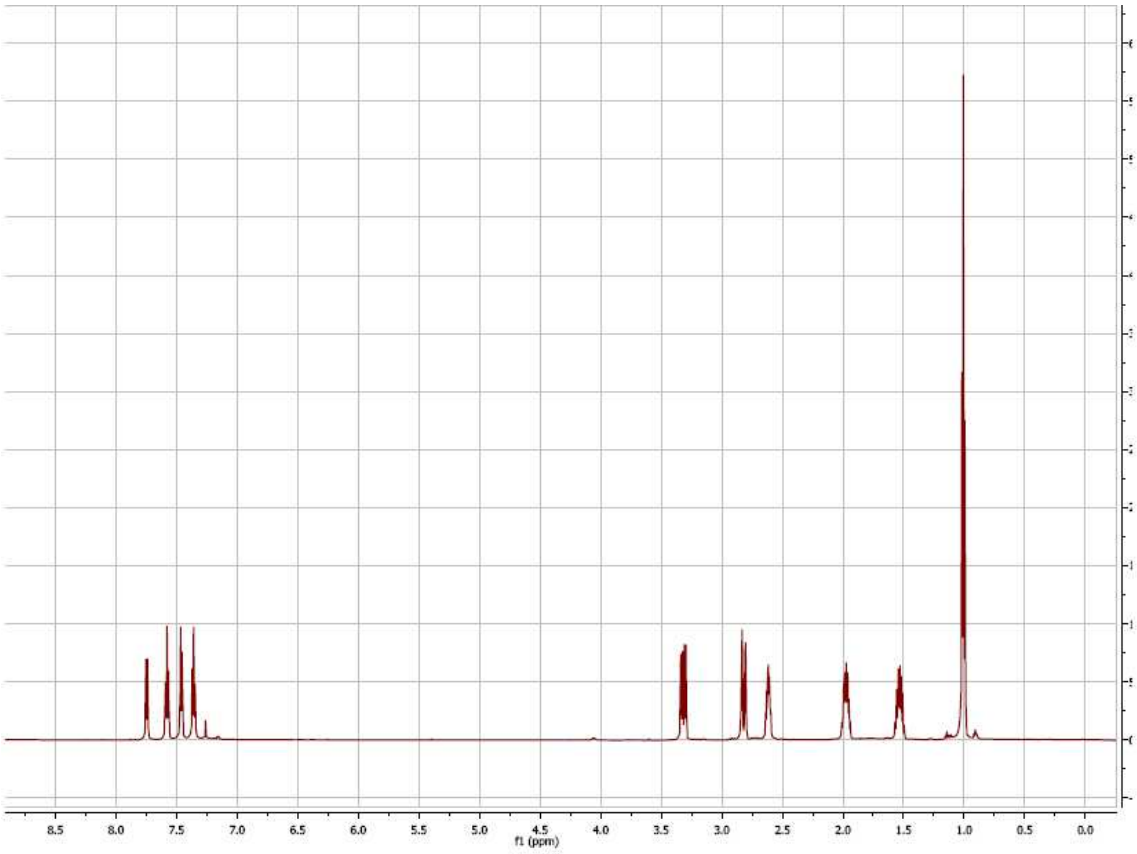


One first conclusion we can draw from what it has been said above is that quaternary carbons are not present in an HSQC spectrum.

Now, we will analyze a real HSQC spectrum acquired for the molecule 2-ethyl-1-indanone.

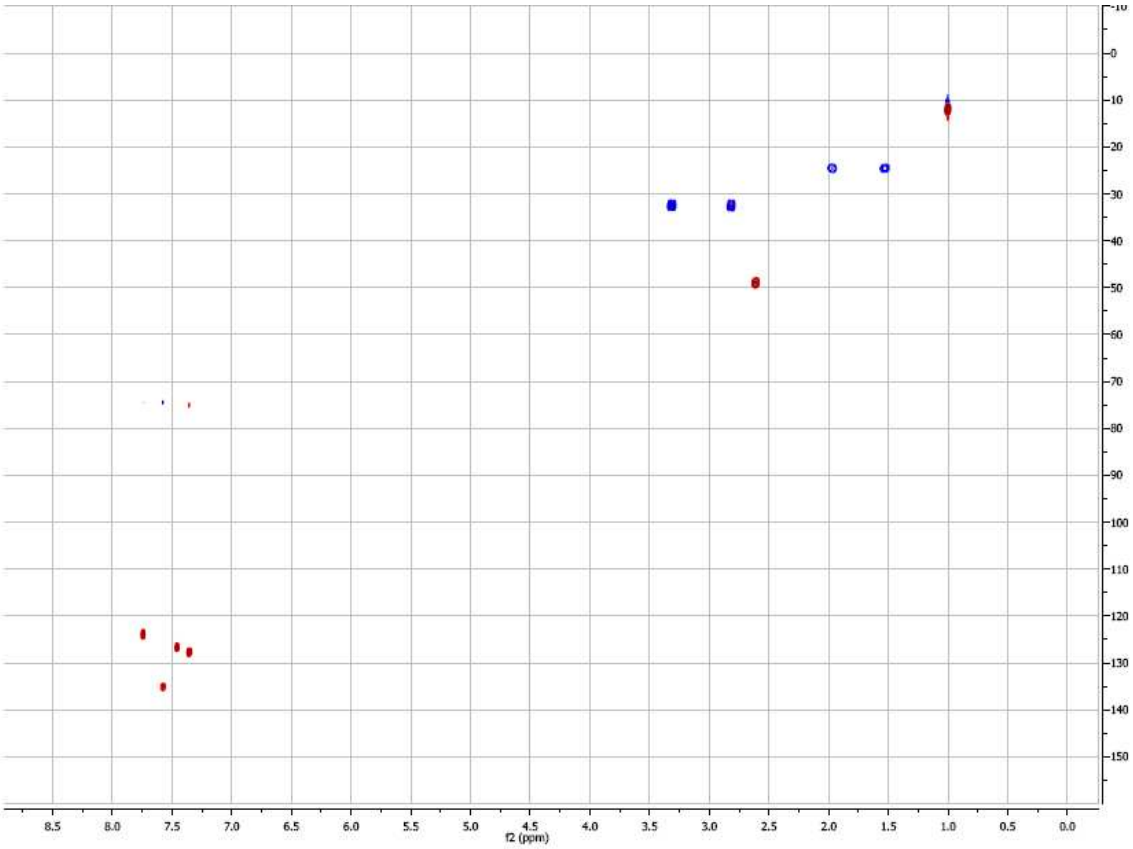


First, though, let us take a look at the 1D ^{1}H spectrum of 2-ethyl-1-indanone.



The spectrum clearly shows an aliphatic and an aromatic region consistent with 2-ethyl-1-indanone. Furthermore, the methyl group can be easily assigned. However, the analysis of the 5 NMR lines between 1.5 to 3.5 ppm, would have required looking into the splitting patterns and homonuclear J couplings. Integrals do not help in this case since each NMR line corresponds to a single proton.

Let us analyze the HSQC spectrum now.



1) There are **4 blue peaks** and **6 red peaks.**

How do we interpret the red and blue peaks?

Simple tips,

a) different colors means peaks pointing in opposite directions (up and down). CH and CH_3 peaks point in the same direction whereas CH_2 peaks point in opposite direction. This is called an "edited HSQC", and it is somewhat similar to a 135^{O} DEPT experiment.

b) Pair of peaks at the same ^{13}C chemical shift (same carbon position) corresponds to the two diastereotopic protons of a methylene (CH_2) group.

c) A strong peak typically corresponds to a methyl (CH_3) group.

2) With the simple tips detailed above, it is easy to recognize that; **red peaks** are protons belonging to CH_3 and CH functional groups while **blue peaks** are protons in CH_2 groups. Now, we can count;

five CH , two methylene (CH_2) and one methyl (CH_3) groups.

3) The four aromatic CH peaks are easy to distinguish because of their ^{1}H and ^{13}C chemical shifts. Therefore, the fifth CH peak at 2.61ppm is the aliphatic CH group of the molecule.

A table of chemical shifts (δ) of protons and bonding carbons can be constructed.

	δ(^{1}H) ppm	δ(^{13}C) ppm
CH_3	1.00	12.1
CH_2	1.97, 1.53	24.5
CH_2	3.31, 2.82	32.5
CH	2.61	49.1
CH	7.74	124.0
CH	7.46	126.7
CH	7.36	127.7
CH	7.57	135.1

In summary, the information we get from an HSQC is straightforward and does not require any elaborate analysis of the data.

Just a reminder, an HSQC experiment is more sensitive than a 1D-^{13}C experiment. This means that you need less time and/or sample to run an HSQC than to acquire a 1D-^{13}C spectrum.

Now, which aromatic peak corresponds to which proton in the molecule? Which CH_2 group is which in the molecule? Again, you can answer those questions by analyzing the splitting pattern of the NMR lines and J couplings on the 1D ^{1}H spectrum, or just by running a COSY (it takes only 3 minutes) and get all the connectivities in a much simple manner….

Well, we will talk about COSY in the next issue.