



Department of Chemistry
NMR Facilities
Director: Dr. Carlos A. Steren

NMR NEWS

June 2013

* To check on the [instrument status](#) and [reservation system](#), and find tutorials, links and more, visit our website www.chem.utk.edu/facilities/nmr

* The NMR website has changed address (www.chem.utk.edu/facilities/nmr) and format. Ideas and suggestions to help improving the website are welcome.

* **New Student Operators at the NMR Facilities.**

Kevin Gmernicki and Roger Wright are the new Student Operators starting this fall. They will start in their positions on August 1st 2013.

Mr. Kevin Gmernicki is replacing Mr. Matt Dembo and will be in charge of the Bruker 400 and Varian 400.

Mr. Roger Wright is replacing Mr. Chad LeCroix and will be in charge of the Varian 500 and Varian 600.

We wish the best to Chad and Matt in their careers. They have been a big help and an honor to have them in the NMR Facilities.

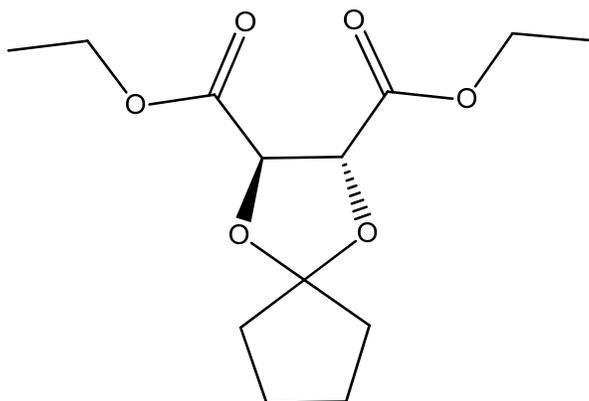
* 12 minutes that can save you a lot of time!!

A ^1H NMR spectrum is easy to acquire, it takes just a few minutes to run. Furthermore, in most cases, it contains the information that is needed.

However, what to do when the analysis of the ^1H spectrum is not straight forward? When there are more or fewer peaks than expected. When integrals do not match up the number of protons in the sample.

In that case, instead of speculating about the ^1H spectrum for hours, we suggest that you spend 12 minutes acquiring a standard ^{13}C -gHSQC. This example tries to explain why.

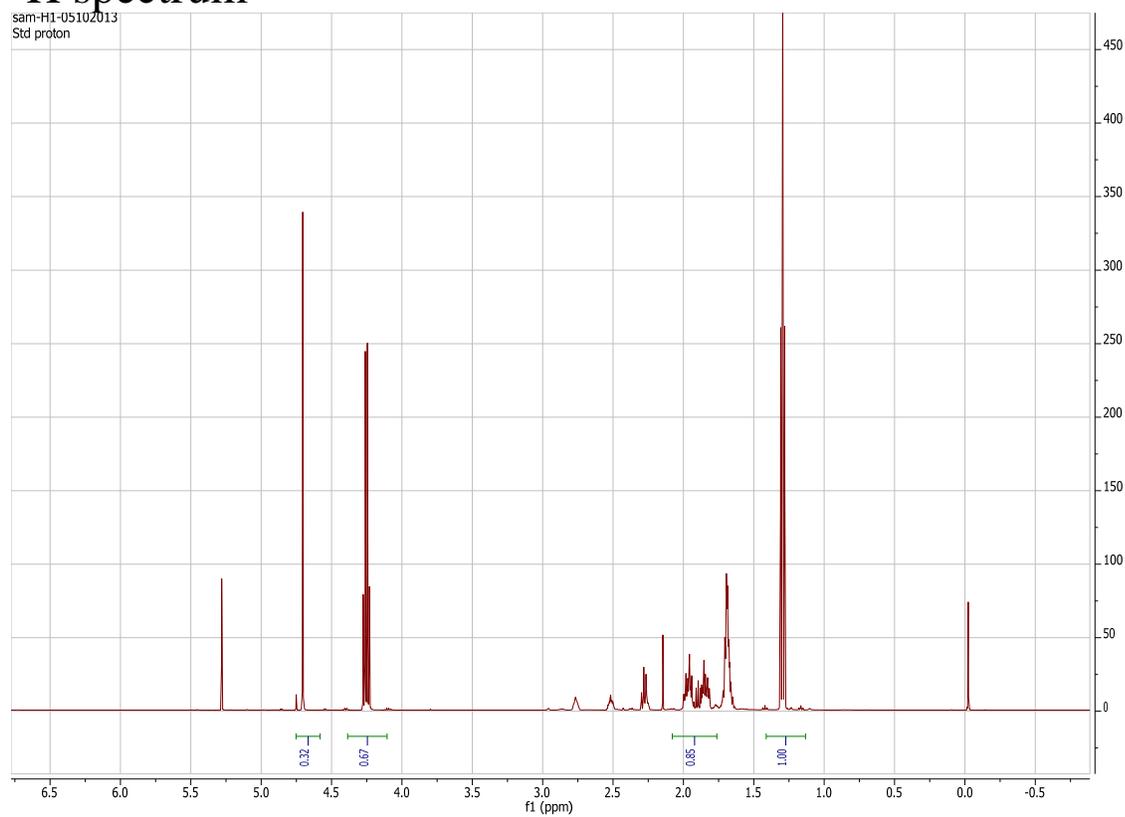
The ^1H spectrum of the complex depicted below shows too many signals. What are these peaks? Which ones belong to the compound?



diethyl (2*R*,3*R*)-1,4-dioxaspiro[4.4]nonane-2,3-dicarboxylate

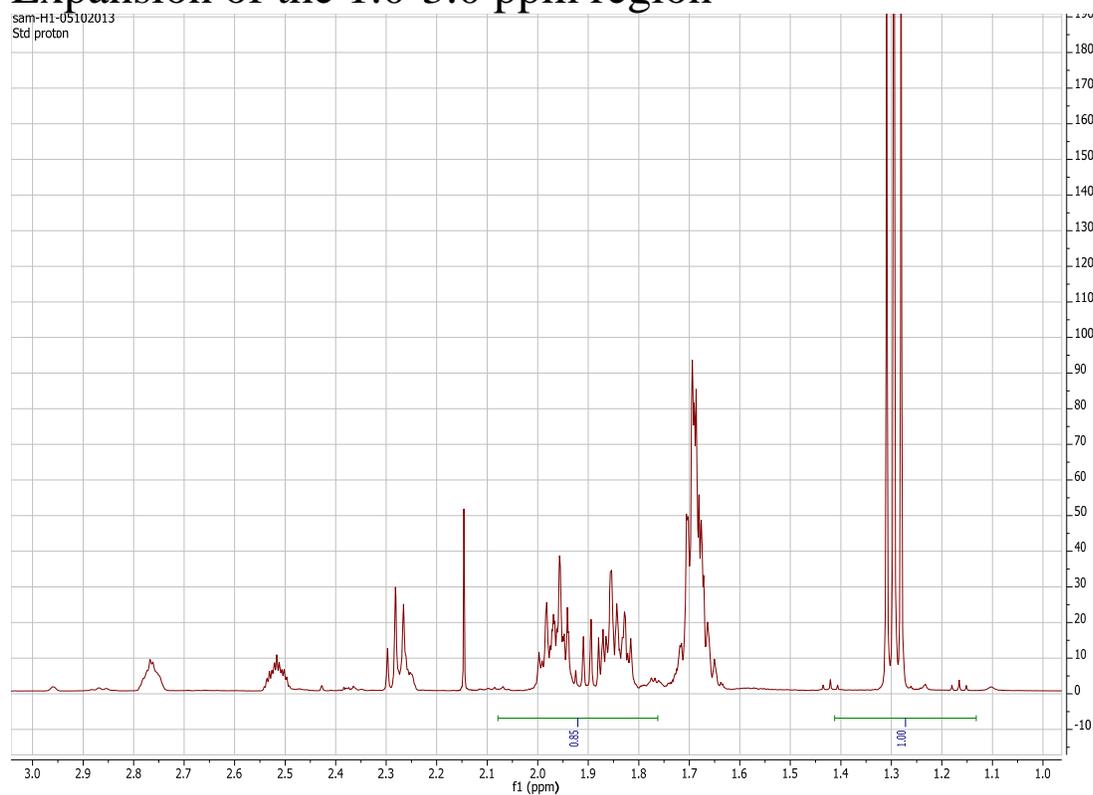
^1H spectrum

sam-H1-05102013
Std proton



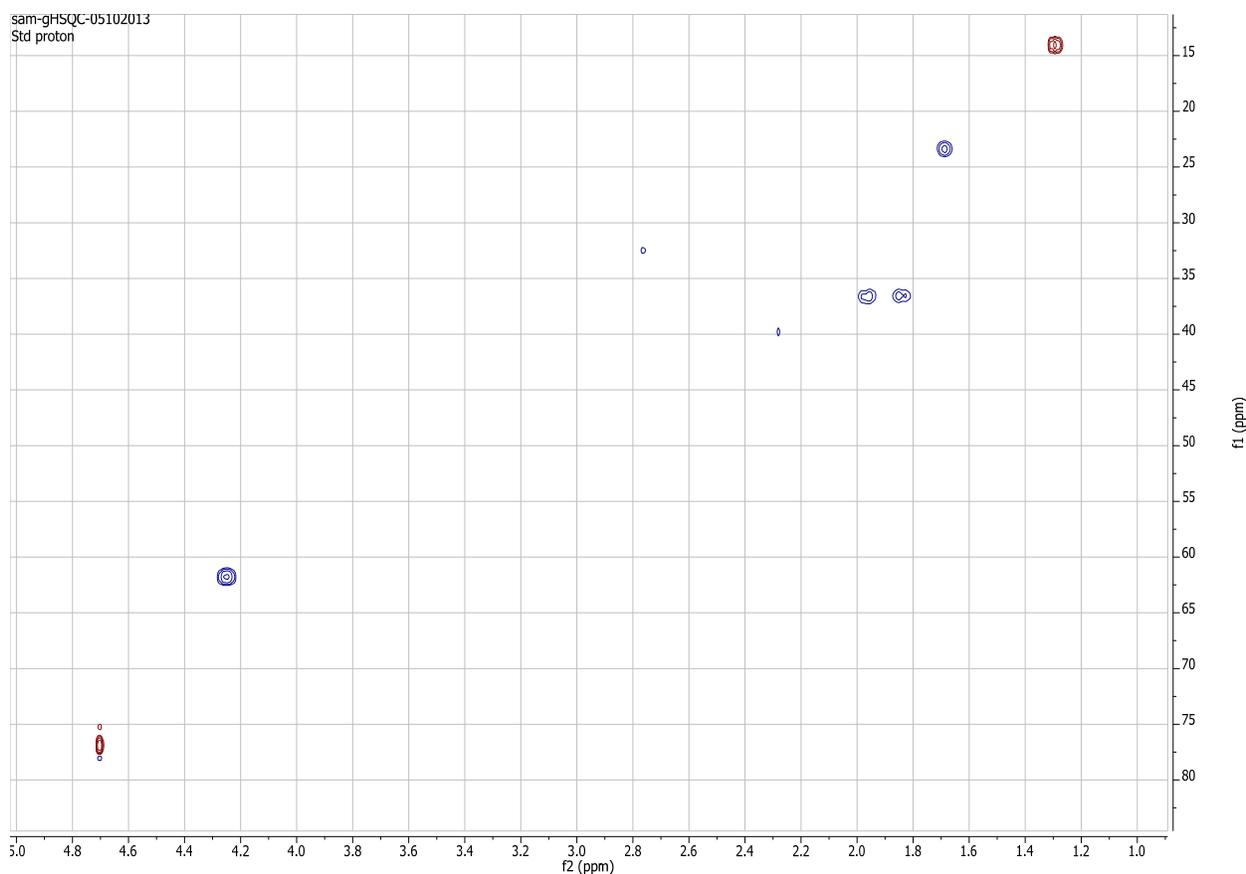
Expansion of the 1.0-3.0 ppm region

sam-H1-05102013
Std proton



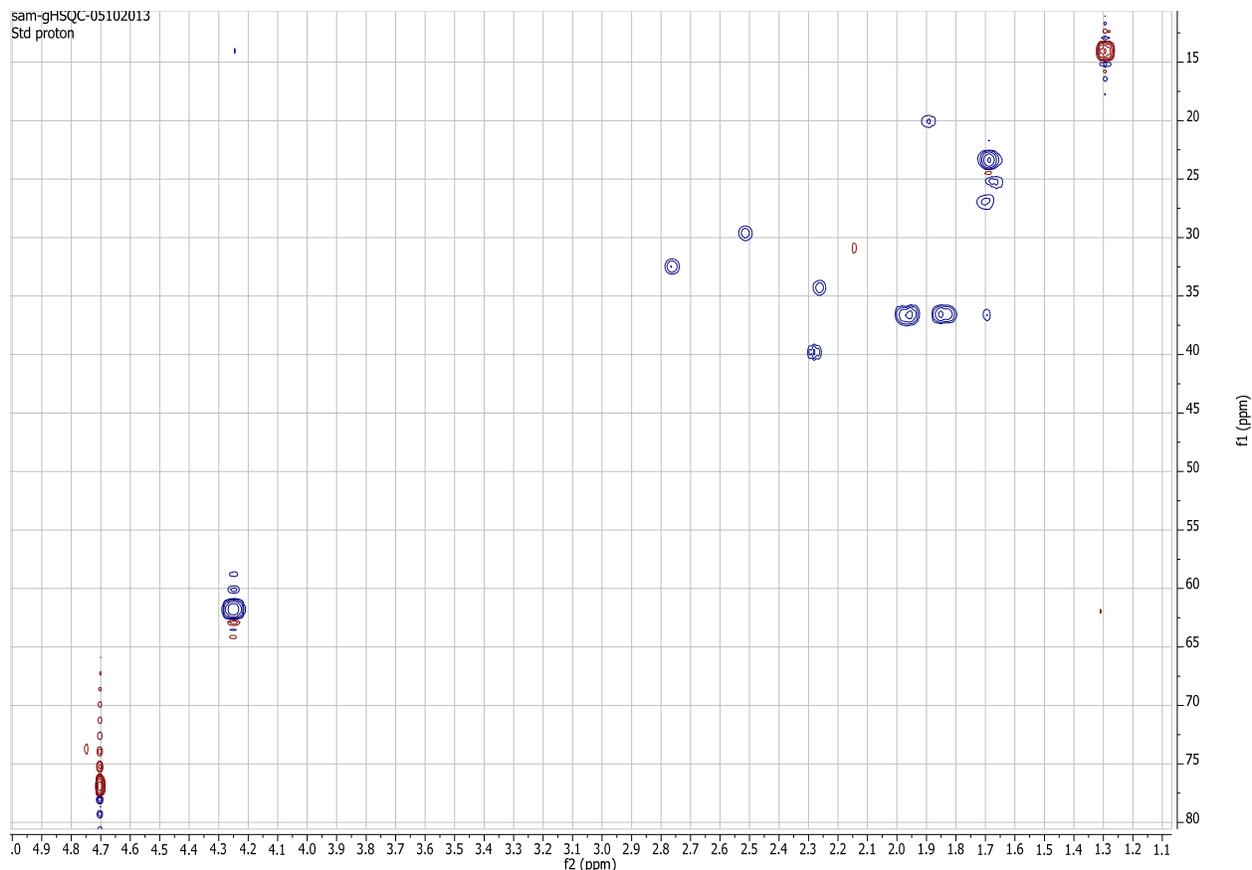
The HSQC shows the expected signals for the molecule:

- one(1) CH₃ group (**red peak**, top right),
- three (3) CH₂ groups, one with diastereotopic protons (**blue peaks**)
- one (1) CH group (**red peak**, bottom left).



What are the extra signals observed in the ¹H spectrum?

We get the answer by analyzing the HSQC spectrum at a lower contour level (displayed below). There are weaker “extra” peaks in the 2D spectrum. These peaks correspond to impurities, which are observed in the ¹H spectrum as well.



Conclusion: 12 minutes can save you a lot of time trying to guess things on a 1D ^1H spectrum.

*** Doty Probe. Solid State CPMAS probe.**

Wands for the Doty probe to observe ^{119}Sn and ^7Li are now available. Nuclei that can be detected on the Doty probe are: ^{15}N (40 MHz), ^{17}O (54 MHz), ^2H (61 MHz), ^{29}Si (79 MHz), ^{111}Cd (85 MHz), ^{13}C (100 MHz), ^{79}Br (100 MHz), ^{27}Al (104 MHz), ^{11}B (128 MHz), ^{119}Sn (149 MHz), ^7Li (155 MHz) and ^{31}P (162 MHz).

*** Cross Polarization Magic Angle Spinning (CPMAS).
CPMAS versus VACPMAS.**

Increasing spinning rates makes the standard Cross Polarization sequence less efficient. Then, ^{13}C signals are weaker, requiring a lot of scans to get a spectrum with a reasonable signal to noise.

It was found, around the year 1993, that varying the pulse strength during CP, improves the polarization transfer at fast spinning rates (faster than 3KHz). This pulse sequence, called Variable CP (VACP), is more efficient than CPMAS at spinning rates faster than 3KHz. There are several versions of the variable CP pulse sequence. The sequence available in our spectrometer is shown below,

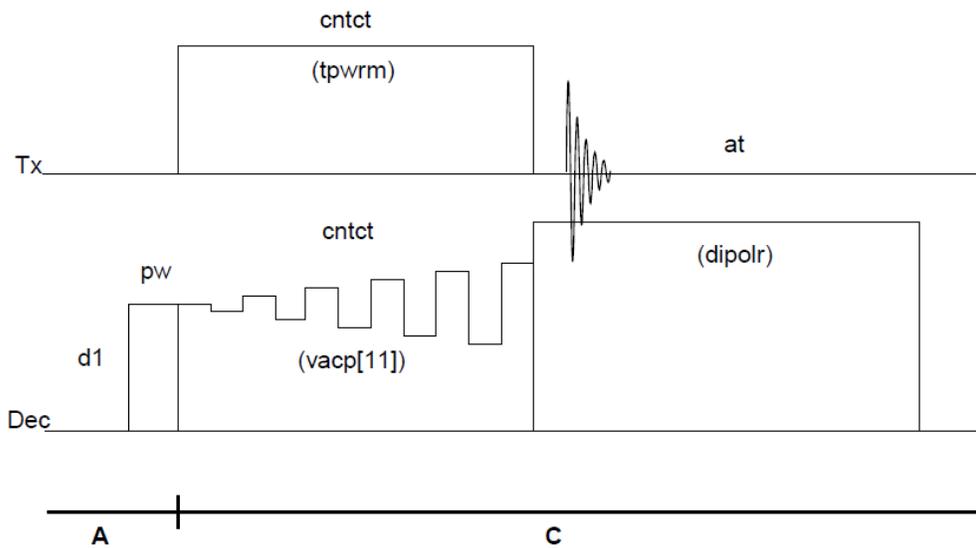


Figure 39. VACP Pulse Sequence

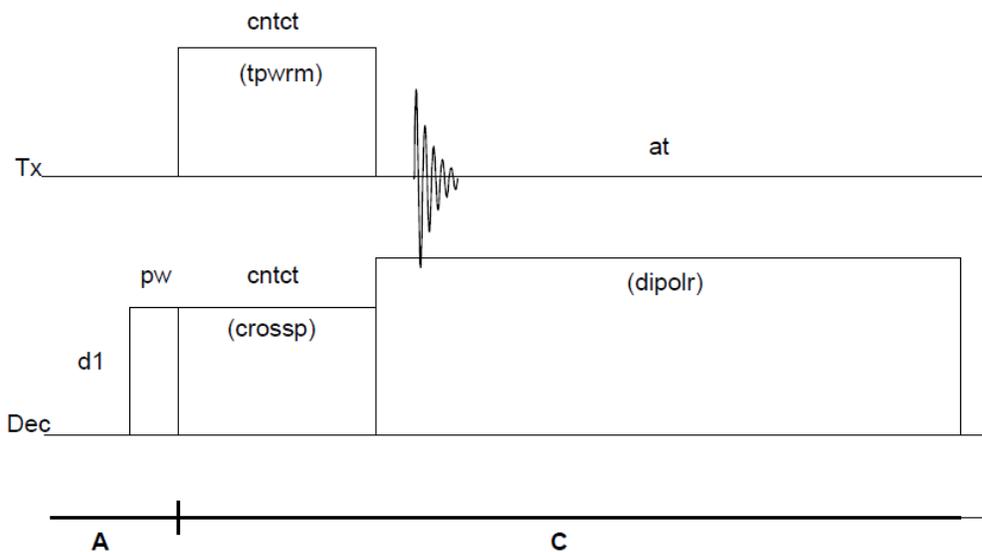


Figure 34. XPOLAR1 Pulse Sequence

In the following graph, the integral area of the methyl signal of hexamethylbenzene (HMB) was plotted as a function of the spinning rate. Data acquired with both CP and VACP are compared. Parameters for CP and VACP experiments were the same. The better performance of VACP is clearly demonstrated.

