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

NMR NEWS April 2009

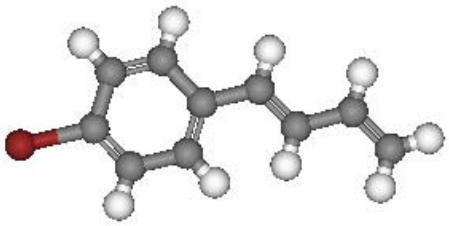
* To check on the **instrumental status** and **reservation system**, and find tutorials, links and more, visit our website www.chem.utk.edu/nmr

* **Bruker 400's BBI probe status:** No progress so far with the probe. Still, it is not ejecting the samples. It was sent back to Bruker on March 31st.

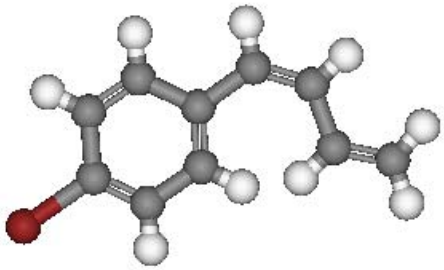
* **Varian 400 Solid State** instrument is up and running. Whenever you have an insoluble sample, then Solid State NMR can help characterizing your sample. In SSNMR, you cannot measure ^{1}H. You acquire high-resolution ^{13}C 1D spectra. If you need to characterize a solid sample, let me know and we can discuss whether SSNMR could be of any help.

∗ **What is the structure of the molecule I have sensitized? Is it trans or cis? That was Mike Quinn's question…**

TRANS

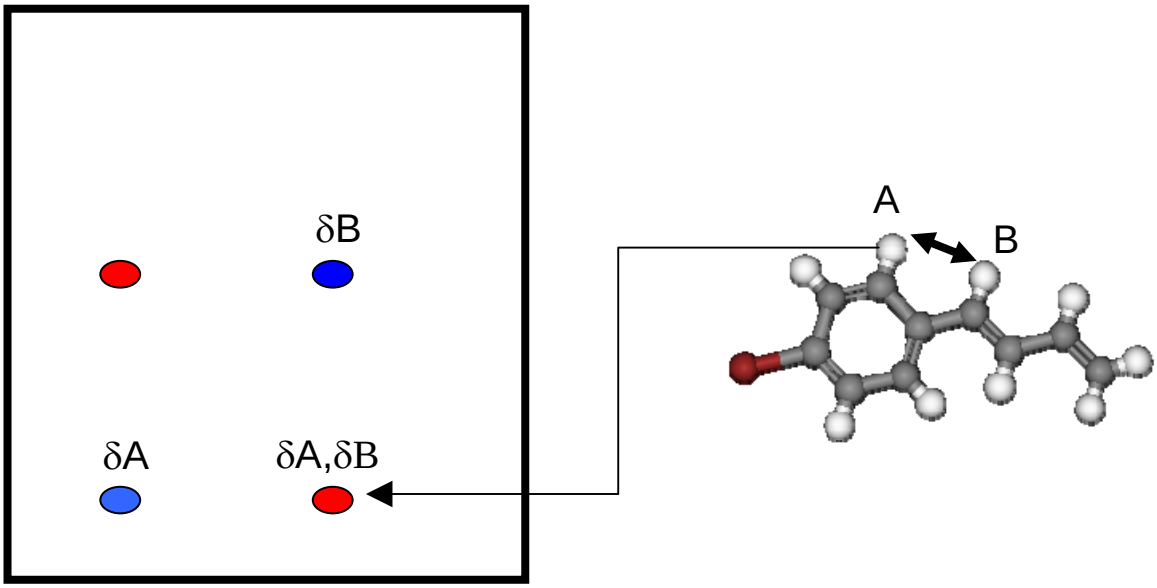


CIS



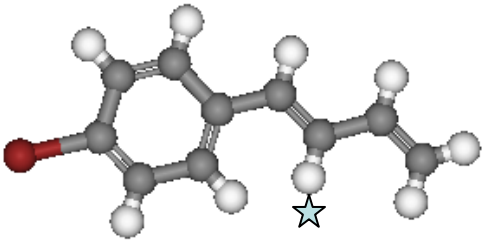
2D-NOESY is a powerful tool to tackle a problem like this one. A NOESY cross peak (also called an NOE) reflects a through space correlation between two protons that are no more than 5 Å (Angstroms) away.

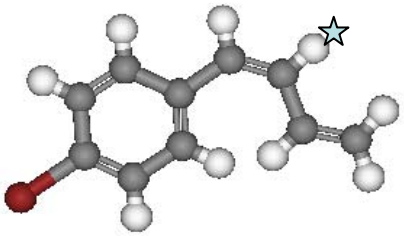
NOESY



In Mike's case, a way to verify the structure could be by confirming whether the ^{1}H indicated with a **light blue star** in the figure below, shows an NOE to the aromatic ortho proton. If an NOE is observed, then the molecule has a TRANS conformation. If the NOE is not present, then the conformation of the molecule must be CIS.

TRANS **CIS**



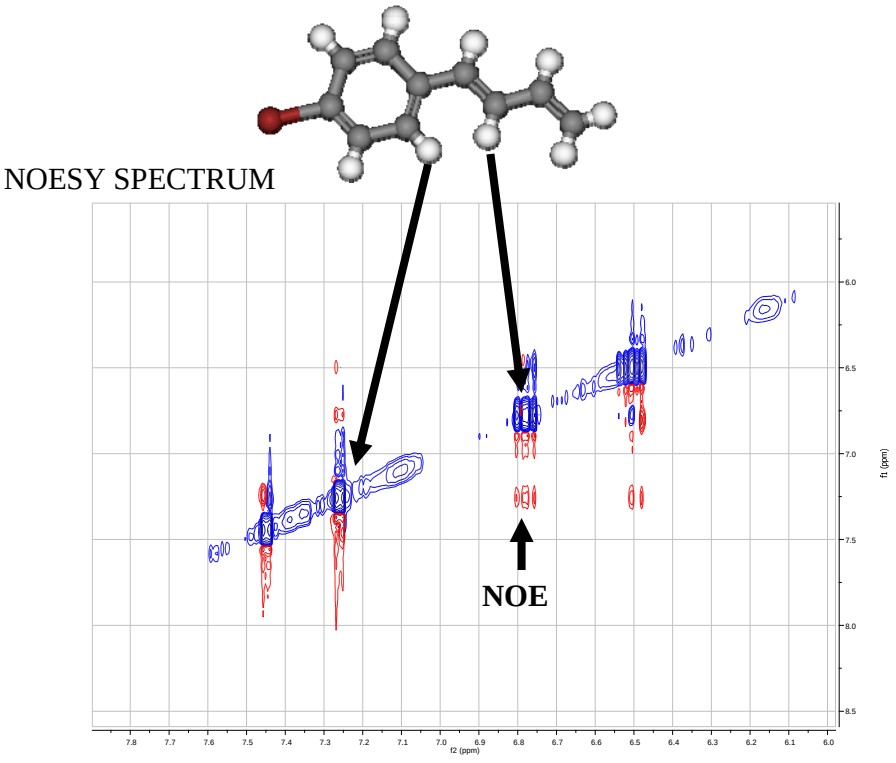


To be able to analyze a NOESY spectrum, first you need to assign the chemical shifts to the protons of the molecule. This was done with the help of HSQC, COSY and HMBC spectra recorded on the sample (not shown here).

It was determined that the chemical shift of the proton labeled with a "star" is δ=6.78 ppm, and that of the aromatic ortho protons is δ=7.28 ppm.

Then, the NOESY spectrum was analyzed. The relevant section of this spectrum is shown below. It can clearly be observed an NOE at coordinates 6.78ppm,7.28ppm, indicating that the protons at 6.78ppm and at 7.28ppm are close in space.

Henceforth, it was concluded that the molecule has a TRANS conformation. Problem solved!



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