FAll 2014 Chem 636 – Lab #2

Assignment due at beginning of lab, week of Sept 16-19.

Use Athena (Bruker AC-300) for this week's HW #2. Use the samples specified for this assignment.

For this lab, it is particularly important to take data yourself on all 3 assigned samples. Shimming is an empirically learned task, and the odd interface Athena presents has to be experienced to become comfortable with it. Athena provides high quality data; if you will need walk-up data, this is a very good spectrometer to use.

Reading
- **field-frequency locks: Claridge section 3.4.4 (and see the guide link below)**
- **shimming: Claridge section 3.4.5 up to Gradient shimming**

Goals
- **Learn how to acquire high-quality ^{1}H spectra on Athena, a Bruker AC+-300.**

Athena is an ancient spectrometer, with hardware and software components designed in the early 1980s. Treat it with care, please, and ask questions whenever you are uncertain.

Use the Bruker AC/AM User Guide at the link below to assist in performing the following tasks (esp. pgs 4 & 5): http://www.chem.wisc.edu/~cic/nmr/Guides/BUG/ACQuickGuide-2014.pdf

Read through the following questions prior to starting work on the spectrometer. Think about these while working, and note that Q2 suggests an experimental procedure to test the effects of solvents on shimming.

Q1 You should notice big differences between the three solvents while shimming. Which solvent gives the best signal-to-noise in the lock signal? Offer a hypothesis to explain your observations.

Q2 Which solvent seemed to react the fastest to changes in the shims?

[Remember to *not* continuously move the shim dial! You need to move a specific amount, then stop for a period of time to let the lock signal settle (the amount of time is dependent on the solvent; thus this question!).]

You can test the solvent's reaction time by moving the Z1 shim setting such that the lock signal degrades by ~ 2 vertical units on the display. [Remember to keep the lock signal always in the top ½ of the display.] Wait for the signal to settle in, then press the Z1 button on the SCM, which will immediately reset the shim to the original value. The lock signal will move back up with a "relaxation time" characteristic of the spin-lattice relaxation time, T_1, of the solvent's ^{2}H nuclei. Use a watch, or just count out the time, to get qualitative values. Doing this procedure for each solvent will provide data to answer the question.

Q3 Which solvent is "best" for shimming?

Experiments:

1☞ Acquire a standard ^{1}H spectrum of sucrose in D_2O. Perform standard processing in MNova, including applying a matched filter, phasing, baseline correction, and proper referencing. Remember to properly annotate your spectra. Plot and upload as normal.

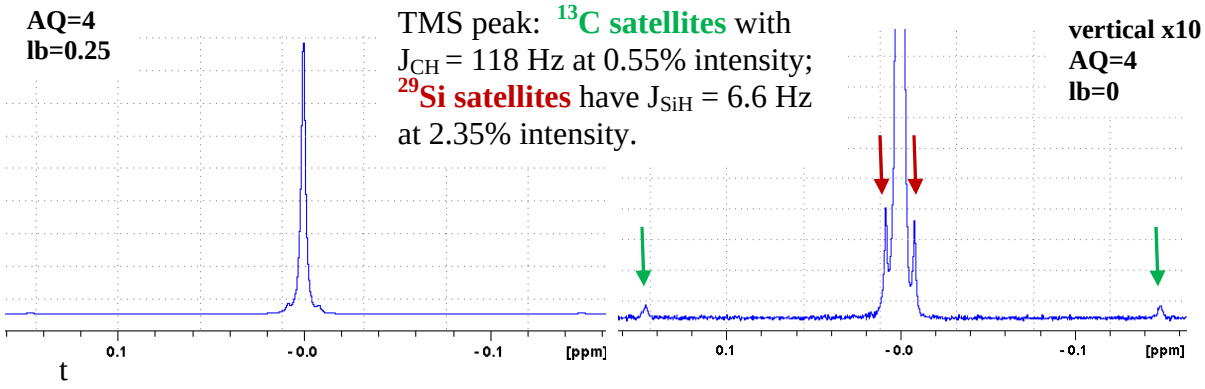
Note: As discussed in lecture, Gaussian apodization is preferred over Exponential/Lorentzian apodization when performing careful integrations; otherwise either are OK (i.e., for this lab, either are OK).

2☞ Acquire a standard ^{1}H spectrum of rotenone in $CDCl_3$. Process as described in part 1, but for this compound also perform integrations across the spectrum, doing as much as reasonable to integrate individual multiplets. Plot and upload as normal.

3☞ Acquire a standard ^{1}H spectrum of 3-heptanone in acetone-d_6. Process as described in part 1; no need to integrate here. Plot and upload as normal.

4+5☞ For any 1H 1D spectrum, it is important to ascertain the quality of the shim you have achived. When TMS is present, it provides a very good method for doing so. For the 2nd and 3rd samples above, expand about the TMS peak from approx. 0.2 to -0.2 ppm as shown below. Expand horizontally and vertically, Plot these two expansions and upload as normal.

*To quickly estimate of the quality of a ^{1}H spectrum of an organic compound, look at the TMS peak. If the ^{29}Si satellites are "resolved", and the central ^{12}C peak is symmetric, the shims are (very) good. A typical example is shown below: on the left standard parameters were used with **AQ** = 4s, **lb** = 0.25; on the right the peak is vertically expanded 10×, and no exponential multiply was applied (i.e., **lb** = 0).*



Upload 5 plots as .mnova and .pdf files, and hand in to your lab instructor either handwritten or typed answers to 3 questions.