

Summer 2012 Chem 637 – Lab #1

Assignment due at beginning of lab, week of 27 June - 2 July.

Use the AVANCE-400 for this week's HW #1.

Always login to your own account when working: your practice grade will be based on your login time. Although you may be working in groups, each of you should setup and acquire your own data, and process and hand in that individual set of data.

Save all of your data, and back it up weekly, e.g., to a USB key.

Reading – *Preparing Samples*: Claridge section 3.3 pgs 75-81

Use the guide from the class website: “Automation on Av400 using IconNMR and SampleJet robot”

Assignment: Use IconNMR on the Avance-400 for this assignment.

The experiments can be run on facility samples — provided in the SampleJet as documented on the sheet next to the spectrometer host computer — or on your own sample(s). One or more samples can be used (i.e., the same sample need not be used for each part).

IconNMR makes acquiring data almost too easy a task: it would be bad habit to not *think* about your data when setting up experiments. In particular, three parameters critically affect data quality in all NMR experiments. The questions below touch on these issues.

1. Collect one ^1H 1D dataset: adjust the parameter discussed in Q1.a to twice its default value.

Q1. There is one parameter fundamentally important to the quantitative behavior of ^1H 1D NMR spectra. A number of parameters actually are involved, but only one is regularly changed in quantitative experiments, as it varies as a function of sample conditions.

a) What parameter is this?

b) State the physical property of a sample that is directly related to this parameter (if you measure the property's value, you can calculate the correct setting for the parameter).

2. Collect one ^{13}C , ^{31}P or ^{19}F 1D dataset.

Q2. Specify which experimental parameter, involved in all NMR experiments, that is fundamentally important to sensitivity and signal-to-noise (S/N). Provide a mathematical relationship that relates this parameter to S/N.

The dependence of concentration on the length of NMR experiments, sufficient to attain usable S/N — is a topic we'll cover in lecture. See Claridge for more detail.

Q3. The parameter described in Q1.a is critically important to normal ^1H -decoupled ^{13}C spectra. Describe what you would expect to happen as you increase the size of this parameter (e.g., from 2 to 5 sec).

3. Collect one 2D dataset (recommended are cosy, hsqc or hmbc experiments).

Q4. The acquisition time, AQ, is critically important to resolution in NMR spectra. Obtainable resolution is $\leq 1/AQ$. Note then the relationship: $AQ = TD/(2 \times SW)$.

What is the corollary to this equation for the indirect dimension in a 2D experiment?

SW1 is set automatically by the software using the acquired proton spectrum adjoining the 2D spectrum. Find the analogous parameter to TD for your experiment (using Parameters → Edit All Acquisition Parameters), and list its value on the 2D plot. Provide a calculation of the digital resolution in the indirect dimension (along F1) in Hz / point for your spectrum.

4. Process the data using NUTS, MNova or TopSpin (your choice). Always document all plots by including the following information:

- your name
- the sample and solvent (provide sample amount or concentration if known)
- the spectrometer
- the date
- the operating temperature
- any changed parameters [you can use the defaults, but please feel free to change parameters that you understand (i.e., do **not** change any you are uncertain about!)]

Q5. The final question points to issues that can arise with decoupling. A ^{19}F decoupler's bandwidth, **bw** —the spread of frequencies over which the decoupler will provide “good” decoupling — is inversely proportional to the ^{19}F 90° pulse width (at the decoupler power), **pw90**. For waltz-16 decoupling (Bruker's CPD):

$$\mathbf{bw} \approx 1/[2 \times \mathbf{pw90}]$$

What is the approx. bandwidth in ppm on the 400 when $\text{pw90} = 85 \mu\text{s}$?

Hopefully, you will find the answer to be surprising. We'll discuss this more in lecture. For more details, see Claridge section 10.3.

Hand-in 3 plots, and answers to 5 questions.