

¹H 1D Experiments on Homer (AC-300)

You will be using Homer to collect data for this lab. Sign up for HW time using the Spectrometer Calendar web page. *Ask questions if you are uncertain about what you are doing!* This homework assignment should take no longer than 2 hours. Get help if it is taking longer. A lab partner is recommended, but make sure to acquire and work up your own data.

Use the Bruker AC Step-by-Step guide during your lab practice session. Two other guides may be useful to you as well. All are available from the class website → **Bruker UG** link:

- **Step-by-Step Intro to the AC Spectrometers** [use while on spectrometer]
- 2. Bruker AM/AC Acquisition Basics**
- 4. Primers: Locking and shimming Primer #1**

Naming of files on the Bruker ACs:

- a) All datasets (*except those for your unknown; see b*) should have **your initials** as the first three characters of the filename. Filenames on Bruker AC spectrometers are limited to 8 characters followed by a period and (typically) 3 numbers: e.g., CGFSAMPL.001. Do not use any special characters in the filename, and do not start it with a number.
- b) **Save all raw data (FID) collected on your unknown compound using the following format. Failure to do so will result in a significant reduction of your HW scores.**

IIII EESS . FFN

where

- IIII** – sample code (supplied on the tube as either a 3- or 4-letter code)
- EE** – experiment code:
 - H1** – standard 1d ¹H spectrum (including 1D setup-up for 2D spectra)
 - HR** – high-resolution 1d ¹H spectrum (increased AQ)
 - C3** – standard 1d ¹³C spectrum
 - CQ** – quaternary-enhanced 1d ¹³C spectrum (increased RD/D1)
 - GD** – coupled 1d ¹³C spectrum (GATEDEC)
 - IG** – quantitative 1d ¹³C spectrum (INVGATE)
 - D4** – DEPT-45 1d ¹³C spectrum
 - D9** – DEPT-90 1d ¹³C spectrum
 - D1** – DEPT-135 1d ¹³C spectrum
 - I1** – INEPT (-135-like) 1d ¹³C spectrum
- SS** – first and last initials of your name
- FF** – ¹H frequency in 10's of MHz:
 - 29** for Athena, data acquired manually
 - 30** for data acquired on Homer
- N** – experiment number (somewhat arbitrary, 0-9)

Save all worked-up spectra in NUTS onto a temp drive folder, **S:\temp\Chem636\yourname**, as

IIIIIEESS.FFN-nuts

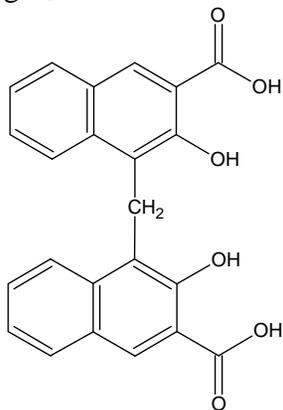
All data taken on the unknowns are being compiled into a database. These formal naming conventions will allow simpler retrieval from that database.

Acquire routine ^1H 1D data from 3 different samples:

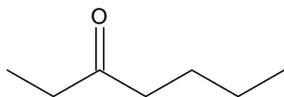
Follow the procedure for acquiring data using the on-line guide:

Step-by-Step Intro to the AC Spectrometers

1. Acquire a ^1H 1D FID of **pamoic acid in DMSO- d_6** . Note how this solvent's lock signal reacts to shim changes; do similar with the next two samples/solvents.



2. Acquire a ^1H 1D spectrum of **your unknown compound in CDCl_3** .
3. Acquire a ^1H 1D spectrum of **3-heptanone in acetone- d_6** .



Q → Describe the differences you noted for shimming with DMSO- d_6 , CDCl_3 and acetone- d_6 . Which solvent do you prefer working with on the spectrometer? Why? Which do you believe would lead to better shims? Why? (There is a correct answer to the 3rd & 4th questions, but don't worry overly about this now; the question is intended to get you thinking about how the different solvents behave.)

PLOT-1,2,3 – Plot all three spectra with integrals (normalized) and proper annotation. Include an inset plot in each plot showing an expansion of the TMS region, expanded vertically to show the ^{29}Si doublet. Turn in with a brief answer to the questions above before your 4th wk lab session.

Note: Save all your raw data to the **S:\temp\Chem636\yourname** folder. You should also regularly back up this data—as well as (all useful) research data—to your own computer and/or storage media (e.g., memory stick or CD/RW). The **S:\temp** folder is intended for temporary data storage (it undergoes regularly, monthly deletions). We will not intentionally delete the class folder data, but accidents happen. Regularly backing up data is important for the class, and (even more so) as a normal research practice.