

Things to know before operating London and Big Sur

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I. A tour of an NMR lab.

Figure 1 shows a picture of “London”, the 400 MHz Bruker AVIIIHD in Malott 3002. Although each NMR lab is configured a little bit differently, you should be able to walk into an NMR lab and recognize several important piece of instrumentation in Figure 1. The most critical components are the magnet, the console, the autosampler and the workstation.

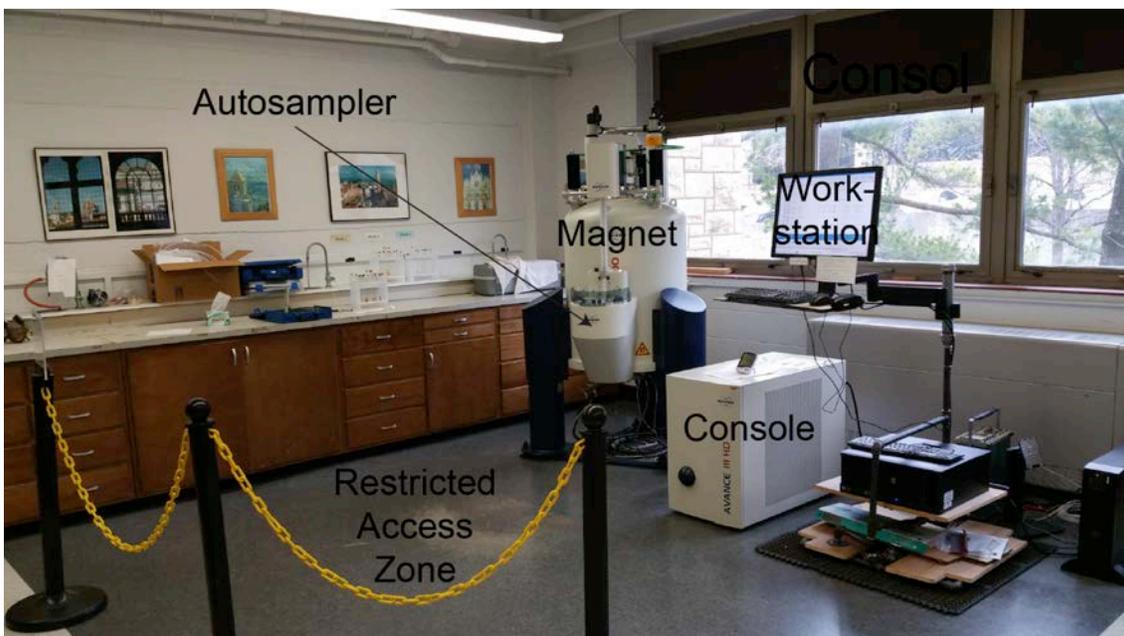


Figure 1. “London”, the 400 MHz Bruker AVIIIHD Spectrometer in Malott 3002.

II. Safety

The magnet is the single most important piece of hardware in the NMR spectrometer. At KU we have superconducting magnets at fields of ~9.4 Tesla (400 MHz), 11.7 T (500 MHz), 14.1 T (600 MHz) and 18.8 T (800 MHz). For comparison the magnetic field of the Earth is only ~0.000030 - 0.000060 T.

It is absolutely critical to understand that NMR magnets cannot be turned off and are always live and at field. Hence, it is very important to be in the habit of making sure that you do not take ferromagnetic objects within the “5 gauss line” (0.0005 T) of the superconducting magnet. In general, the KU NMR core lab has

applied some sort of markings on the floor of the labs to designate the 5 gauss line. Additionally, there are some sorts of barricade to attempt to restrict access within the 5 gauss line (See Fig. 1). In the worse case scenario, the magnetic force near the magnet can pull objects out of your grasp and (potentially) damage the spectrometer or injure a person. Ferromagnetic objects include: tools (hammers, wrenches, screwdrivers), metal buckets, metal chairs, etc. Items such as iPads, cell phones, jump drives, fitness trackers and watches can be damaged by the magnetic field. The magnetic strip on credit cards, ATM cards and student ID cards can be erased. Finally if you have certain medical devices, such as a pacemaker or metal plates, you should not approach the magnet.

III. Loading a sample

In our experience, improper loading of the sample is the single largest cause of downtime to the spectrometers at KU. Hence, it is critical that each user understand how to load and unload their sample properly.

The sample sits in a sample holder, called the “spinner”. We use a tool called the “depth gauge” to control the placement of the NMR tube in the spinner. Figure 2 shows a picture of the spinner and depth gauge. It is crucial that you always use a spinner and always use the depth gauge to position the NMR tube in the spinner. A tube that is not placed in the spinner correctly will not sit in the spectrometer properly. This situation could lead to no NMR signals or (in the most extreme cases) damage.

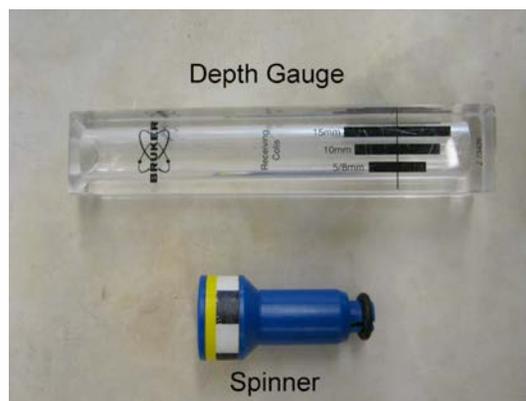


Figure 2. Depth gauge and spinner.

Figure 3A shows how to use the depth gauge to load a NMR tube into the spinner. Please note that these spinners work for 5 mm NMR tubes only. Do not use another size of NMR tube! Set the spinner in the depth gauge and gently push the tube to the bottom. You should be mindful of a few things. First, cheap NMR tubes sometimes will not fit snugly into the spinner. Moreover, sometimes the O-rings on the spinner crack. Whatever the case, you should pay attention to how the tube sits in the spinner. If the spinner does not grip the tube tightly then the sample will not be loaded into the magnet properly. Hence you should not use an NMR tube that doesn't fit snugly. Second, the KU NMR lab recommends that your sample volume is $\geq 600 \mu\text{L}$ for routine spectroscopy. We realize that occasionally smaller sample volumes are necessary. In this case (and only in this case) do not push your NMR tube to the bottom of the depth gauge because the sample will not be in the proper position. Fig. 3B illustrates this situation. An NMR tube with $300 \mu\text{L}$ sample volume adjusted to the maximum depth will not position the sample throughout detected region of the spectrometer. The thin black horizontal line on the depth gauge denotes the center of the detected region. In

Fig. 3C an NMR tube with 400 μL sample volume is centered in the detected region by raising the NMR tube slightly. Note that this sample will be more difficult for the automation to lock and shim on which may cause the experiment to fail. Therefore the sample position in Fig. C is not recommended unless absolutely necessary.

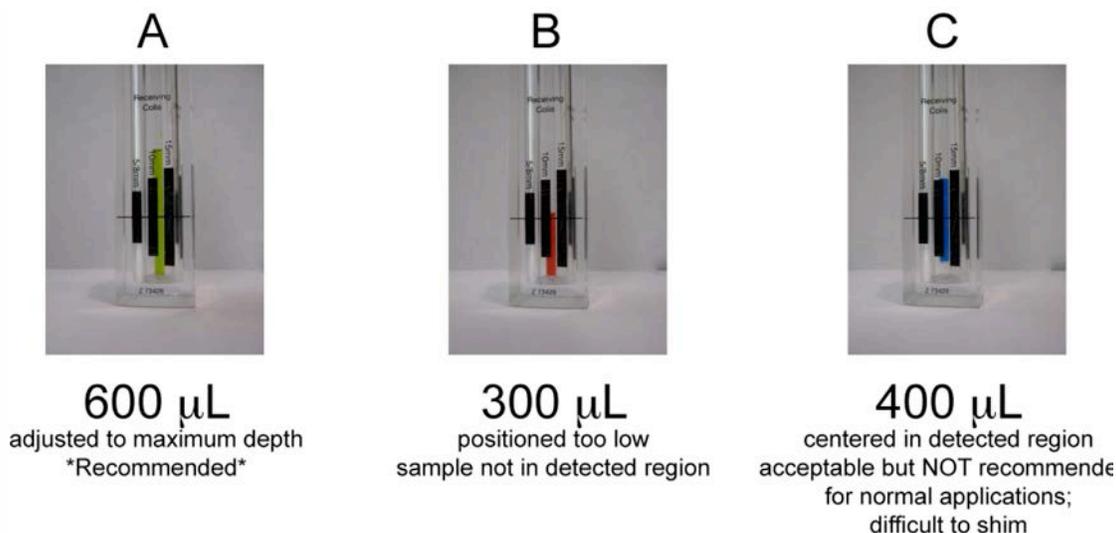


Figure 3. Pictures demonstrating a properly gauged sample (A), an improperly gauged sample (B) and an acceptable, but not recommended, gauged sample (C).

Once your tube has been appropriately placed in the spinner you will add your sample to the autosampler. London has a SampleCase autosampler. Place the sample next to the arrow or in the closest available slot and take note of the slot number. Big Sur is equipped with the BACS autosampler. Place your sample in the next available spot on the queue.

IV. Queuing Your Sample

After placing your tube on the autosampler you will enter your information in the IconNMR automation window, shown in Figure 4. The very first step is to click on “Change User” and select your name. Then double click on your slot number and fill in the line of information. The first piece of information you will enter is the name of your dataset. A dataset is just a folder that contains all of the experiments run on a specific sample. Speak with your advisor, a senior Graduate Student or Post-doc, regarding any specific recommendations on dataset names. The most common convention is to use initials, notebook number and page. For instance, if the name of the user is “Barack Obama”, the notebook number is “1” and the page for this specific reaction is “14”, the dataset name could be “BO-1-14”. Keep in mind that this dataset is actually a folder in the Linux operating system, so avoid spaces and special characters (the dash and

underscore are acceptable). Also note that variation in capitalization can present problems accessing data in OSX or Windows (e.g. Linux can distinguish “BH-1-14” from “bh-1-14”, but other operating systems cannot), so be consistent. Once you have appropriately named your dataset, tab over to the column labeled “No.”. This is the experiment number. Each experiment is a subfolder in the dataset folder, which is denoted by a number. For instance, 1D ¹H is usually experiment number 1. Any additional experiments such as 19F or 19FCPD could be experiment number 2 and 3, respectively. After selecting the deuterated solvent (or “NoD” for non-deuterated solvents) and the experiment from the following list, click on “Submit” to queue your sample. Samples will run in the order in which they were submitted. After sample submission is complete click on “Change User” once more. You can either leave the user list open without choosing a user or you can select “malott” to prevent any users after you from accidentally running their sample in your account.

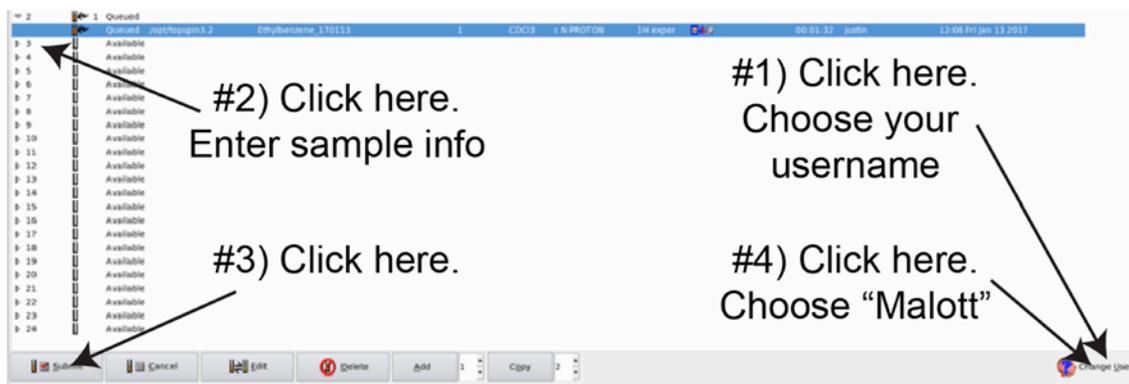


Figure 4. The iconnmr automation page

V. Data Processing

Iconnmr automatically processes the data, creates a PDF and emails it to the user. Any additional processing or analysis can be completed using either MestReNova or TopSpin, which can be downloaded and installed to personal computers. For NMR data processing software information, please see our website: nmrlab.ku.edu.

VI. Instrument Time Restrictions

London and Big Sur are intended for samples to be run with a quick turnaround. Long experiments such as 1D ¹³C should not be run on these instruments unless they can be run in under 15 minutes. No one person should utilize the instrument for more than 15 minutes at a time. If it is necessary to submit multiple samples at once, queue up 15 minutes worth then skip 2-3 positions on the sample and submit the next 15 minutes worth. Samples violating the time restriction will be deleted from the queue without notice. If a 1D ¹³C or any multidimensional experiments are required, it is recommended the sample be submitted to the cryoprobe on Siena or run in non-automated mode on Paris or Yosemite.

Questions

- 1) Which of the following is not OK to take inside the 5 gauss line of the magnet: cart for moving gas cylinders, cell phone, your new Ipad, a couple of quarters in your pocket.
- 2) You can't find the depth gauge. Will you be able to do NMR?
- 3) You place your NMR tube in the spinner. The spinner is loose on the tube, but it seems to hold the tube in place. What do you do?
- 4) You made a sample using CHCl_3 (instead of CDCl_3) as the solvent. Will the instrument be able to lock on this solvent? Will you be able to record a 1D ^{19}F spectrum on your fluorinated product?
- 5) You have 6 samples to submit which will run for 5 minutes each. How do you queue these on the autosampler?