I. A tour of the BioNMR lab in Mary Sue Coleman Hall (MSCH) 3214

The BioNMR Core Lab located in MSCH 3214 houses two NMR spectrometers: a Varian 400 MHz spectrometer with an inverse probe and a Bruker 600 MHz spectrometer with a prodigy probe. The users of these instruments are mainly composed of organic chemists who check the synthetic structures of starting material, intermediate compounds, and final products; and natural product chemists who characterize/identify unknown structures of purified samples from various biological matrices.

Figure 1 shows a picture of the Bruker NEO 600 MHz NMR spectrometer in MSCH 3214, you should be able to recognize several important pieces of the instrumentation in Figure 1. The most critical components are the magnet, the console, the autosampler and the workstation. The spectrometer is equipped with a liquid nitrogen cooled Prodigy ($^1H/^{19}F$)-X broadband probe.
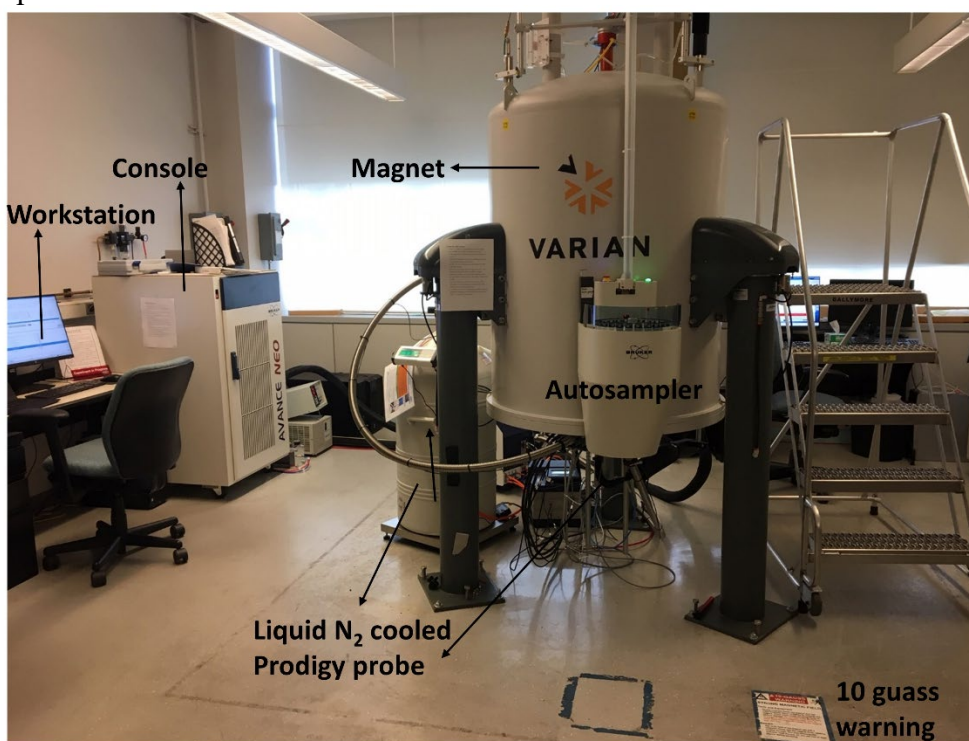

Figure 1. The 600 MHz NMR in MSCH 3214

II. Safety

A magnetic field surrounds the magnet in all directions. This field is invisible and objects made of ferromagnetic materials, e.g. iron, steel etc. will be attracted to the magnet. If a ferromagnetic object is brought too close (within the 10 guass line, see figure 1), it may suddenly be drawn into the magnet with surprising force. This may damage the magnet, or cause personal injury to anyone in the way. It is important that people working in the vicinity of the magnet fully understand the potential hazards. 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. Of critical

importance is that people fitted with cardiac pacemakers or metallic implants should never be allowed near the magnet.

III. General procedures for acquiring data using IconNMR on the Bruker 600 MHz NMR

1. Prepare your sample and label your NMR tube

2. Put the NMR tube in spinner using Bruker depth gauge

3. Load sample in next available slot in autosampler. Note the slot number

4. In the IconNMR automation window, click on "change user" and select your user name, then put your password.

5. Double click on the corresponding sample number in the IconNMR automation window.

6. Enter sample information.

7. Click on "Submit" button to complete submission process

8. Your NMR data is stored in /icondata/data/yourusername/nmr, once the experiment is completed you will get data by email.

9. Collect your samples in a timely manner

IV. Sample preparation and tube labeling

Dissolve your sample in 500-600 µL deuterated NMR solvent. Make sure there is no un-dissolved material. If there is, you will need to either centrifuge or filter your sample to remove crystals/debris, then transfer your sample into a clean NMR tube. We recommend using 5 mm high-quality NMR tubes when running experiments at 600 MHz or higher, but economy tubes will be OK for routine work. If you must use less solvent, consider using a Shigemi tube. Generally 2-10 mg of material is required for routine ^{1}H and ^{13}C NMR experiments.

Once sample preparation is done you will need to label you sample. Any sample that is not labeled will not be allowed to run on the 600. You can label your sample with permanent marker directly on the top of the tube, and DO NOT label on the cap! We also provide stickers on the 600 console, your label must stick smoothly and tightly on the tube and DO NOT leave a flap.

V. Loading your sample

Pick up a spinner turbine in the closest available slot in the authosampler and take note of the slot (figure 2). The 600 NMR has a SampleCase autosampler that can hold 24 samples. Gently place your sample inside the Bruker spinner turbine. Use the Bruker depth gauge to measure the sample height as it is shown in figure 3. If you have 500-600 µL of solvent, then push the tube to the bottom of the gauge. If you have less than 500 µL of solvent, center the solution on the center line. Note that the Bruker Spinner and depth gauge are not

interchangeable with the Varian ones used with the 400 MHz NMR. If you use cheap tubes, sometimes the tubes will not fit into the spinner, thus you should not use an NMR tube that the spinner does not grip tightly. Once your tube has been appropriately placed in the spinner you will place your sample into the autosampler.
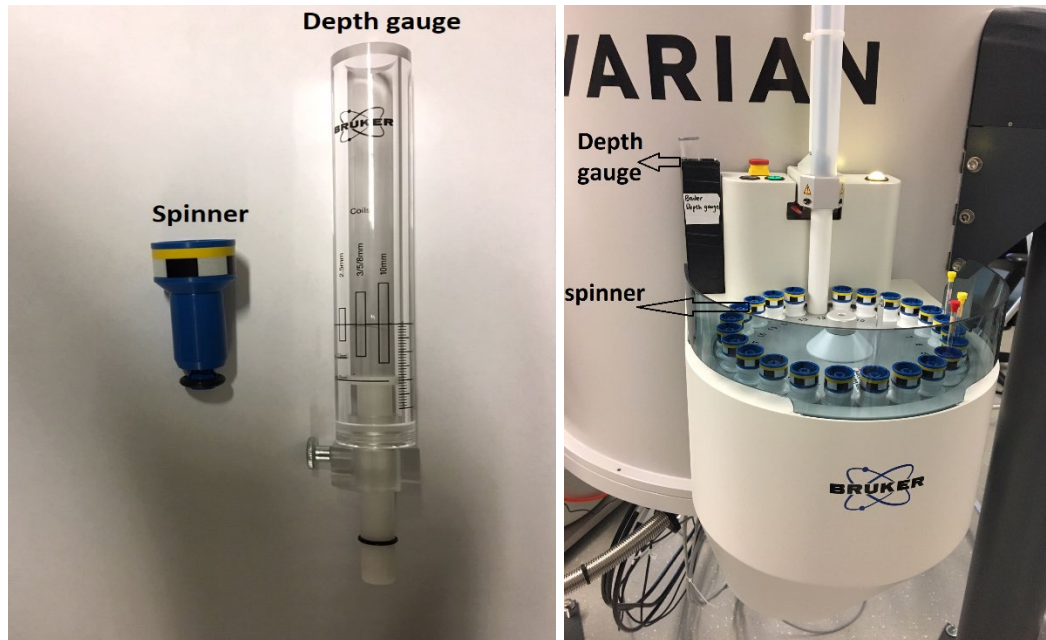

Figure 2. Bruker spinner and depth gauge

Figure 3. Placing the sample into the spinner using the depth gauge.

VI. Login and experiments setup

Figure 4 shows how to login to IconNMR automation and enter your sample information. Click on "change user", select your name, and enter your password. Then double click on your slot number and fill in the line of information including Sample Name, Experiment Number, Experiment to be run, Solvent and Experiment Parameter Set.
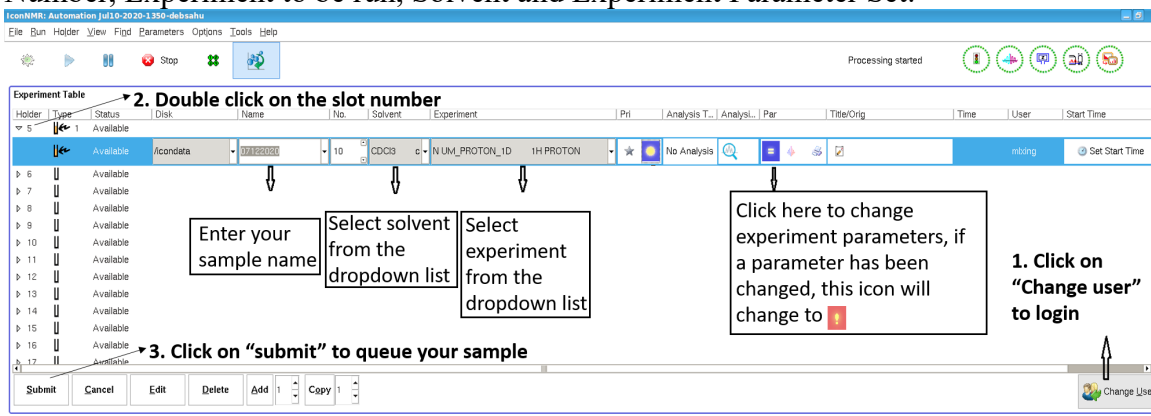
Figure 4. IconNMR login and experiments setup

1. The default disk is /icondata, you do not need to change it.
2. When entering sample names, avoid using spaces and special characters with the exception of underscores (_) or dashes (/).
3. The default experiment number is 10, you do not need to change it.
4. You can select the solvent from the dropdown list (Figure 5). If you have a special solvent and/or your solvent is not listed, please contact BioNMR staff (bionmrcore@umich.edu)
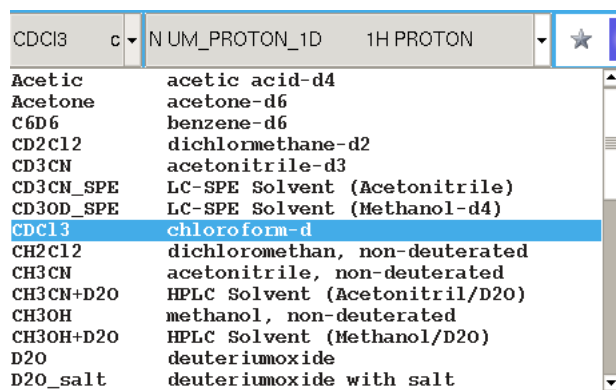

Figure 5. Solvent list

5. You can then select the experiment from the dropdown list shown in Figure 6. The experiments name started with "N UM_" are specifically designed for UM users. If there is an experiment you would like to run but it is not on the list, contact BioNMR staff (bionmrcore@umich.edu)

6. In the experiment parameter set, you can modify parameters such as number of scans, temperature, sweep width etc. if necessary (Figure 7). If a parameter has changed, the blue parameter icon will change to an exclamation point.

Figure 6. Experiment list

Figure 7. Experiment parameters

7. If you want to run more than one experiment on the sample, click on "Add" and enter the information of your additional experiment. You can also copy or edit your experiment by clicking on "copy" or "edit".
8. Once you add all information, click on "Submit" to queue your sample. IconNMR automation will load your sample, tune and match, lock, shim, acquire and process the data automatically (Figure 8).
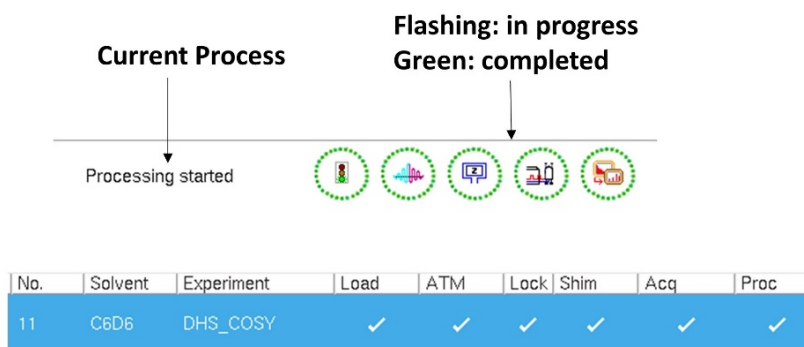
Figure 8. IconNMR automation process

VII. Remote access

A separate file describing how to access IconNMR remotely will be provided.

VIII. Data processing

IconNMR will process your data automatically. You can also use MestReNova or Topspin to analyze the data. Bruker offers free topspin processing for academia: https://www.bruker.com/service/support-upgrades/software-downloads/nmr/free-topspin-processing/nmr-topspin-license-for-academia.html Please contact BioNMR lab staff to obtain a license of MestReNova.

IX. Collecting your sample

Because the autosampler has a limited number of slots (24), it is important that you collect your sample in a timely manner. If samples are not collected in a timely manner, BioNMR lab staff will take samples from the slots at 3 pm Monday to Friday, and place the samples in the cooler by the door of MSCH 3214.