

Basic Solution-state 1D NMR Training Manual
for the Bruker 700 MHz Avance III HD NMR Spectrometer



Scheduling NMR Time:

Any use of the 700 MHz NMR spectrometer requires prior booking of a time slot using the faces scheduling system. After completion of the basic training course a user will be given a login name and password, via email, for the faces system. This login information can then be used to log in to the faces scheduling system at <http://faces.ccruc.uga.edu/> under the group name of ulethnmr. Due to the high demand for time on the 700 MHz NMR spectrometer, the instruments time has been broken down into peak and non-peak hours. Each of these time periods has a specific set of rules on how the instrument may be booked. Please consult the most recent version of the 700 MHz NMR policies for these rules and follow them at all times. In addition, the 700 MHz NMR has three different probes that are utilized in the instrument and each of these is designed to carry out specific experiments. The schedule for these probes can be viewed at: <http://www.uleth.ca/artsci/magnetic-resonance/booking/>. Users must check this schedule and ensure that the probe which they require is installed during the time slot they are booking. If you have any questions regarding these policies please contact the NMR Facility manager for

clarification. The most recent version of the 700 MHz policies, as well as this and other manuals, can be found at: <http://www.uleth.ca/artsci/magnetic-resonance/information-students>.

Safety Concerns:

(1) Magnet Awareness

The magnetic field of an NMR magnet is approximately 1 million times as intense as the earth's magnetic field - so treat the magnet with respect. Read and understand the warning notices posted at the entrance to the spectrometer room. The magnet will attract objects containing steel, iron, or other ferromagnetic metals. Such items include tools, electronic equipment, gas cylinders, keys, and steel chairs. Never take these items near the magnet. The magnet can corrupt the information contained on bank cards (or magnetic storage media) and may damage watches. Leave your wallet and watch outside the 5G line. To prevent accidental damage to the magnet the probe should always remain in the magnet and students should not adjust or touch the probe. Make sure any visitor or anyone carrying out maintenance or cleaning work in the spectrometer room is aware of the magnet and what it can do. In the unlikely event of a magnet quench (or if the oxygen alarm sounds), leave the room immediately. Do not re-enter until permission to do so is given by the NMR Facility Manager.

(2) Electrical Safety

High voltages exist within the spectrometer. All maintenance of spectrometer equipment should be carried out by the NMR Facility Manager. Under no circumstances should you remove any spectrometer parts from the console. Do not adjust or touch the RF cabling on the 700 MHz instrument, as doing so while an experiment is running can result in severe injury or death.

(3) Handling cryogens

Be sure to follow the directions provided in the University of Lethbridge cryogen safety manual at all times. This document can be found at the facility website here:

<http://www.uleth.ca/artsci/magnetic-resonance/information-students>

Liquid nitrogen (and helium) can cause severe burns so use it with caution. Never directly handle anything that has been immersed in liquid nitrogen (use insulated gloves or tweezers). Always use eye and face protection when transferring liquid nitrogen under pressure (such as when filling the magnet). If the oxygen-level alarm sounds leave the room immediately. Do not re-enter until permission to do so is given by the NMR Facility Manager.

(4) Working Alone

Some students and staff choose to work after hours. Those individuals who choose to work after hours must take responsibility for ensuring someone knows that they are working alone in a known location. They are also advised to make appropriate use of the campus Safewalk service. It is also recommended that anyone choosing to work outside regular hours consider having a personal cell phone. For the U of L working alone policy refer to:

http://www.uleth.ca/information-technology/labs/SCL-Working_Alone.htm

If you are working after hours within the NMR facility you need to log in to the Working alone system <https://www.uleth.ca/WorkingAlone> to let campus security know when and where you are working. Don't forget to logout of this program when you leave the building.

Step-by-Step Instructions on Using the Spectrometer with IconNMR and the SampleCase:

NOTE: This is the normal operation mode for the 700 MHz NMR spectrometer

(1) Once you have been trained to use the 700 MHz instrument you will be given login credentials for IconNMR. To begin using the 700 MHz NMR spectrometer you must log into the IconNMR system seen below Figure 1. If no one is logged into IconNMR you should see the user login box show in figure 2. If someone is logged into IconNMR you can simply left click on the change user button located in the bottom right corner of figure 1. This will log them out and bring up the login box.

The screenshot displays the IconNMR Automation software interface. The main window shows an 'Experiment Table' with columns for Hold, Type, Status, Disk, Name, No., Solvent, Experiment, Pri, Par, Title/Orig, Time, User, and Start Time. The table lists several experiments, including '1H NOESY on Asphatene with n' and 'Shimming Test'. A 'Change User' button is visible in the bottom right corner. Below the experiment table, there is a 'Preceding Experiments' section with a table of previous runs.

HoL...	Type	Status	Disk	Name	No.	Solvent	Experiment	Pri	Par	Title/Orig	Time	User	Start Time
1	U	Available											
2	U	Available	C:\	RM-2014-07-23-Asp	2	CDCl3	chlo - C 2D_HI_NOESY			1H NOESY on Asphatene with n	07:05:25	RM	Set Start
F2	U	Available	C:\User	RM-2014-07-23-Asp	1							RM	
3	U	Available	C:\	RM-2014-07-23-Asp	3	chloroform-d	C 2D_HI_ROESY			1H ROESY on Asphatene with n	06:56:50	RM	Set Start
F2	U	Available	C:\User	RM-2014-07-23-Asp	1							RM	
3	U	Available											
4	U	Available											
5	U	Available											
6	U	Available											
7	U	Available											
8	U	Available											
9	U	Available											
10	U	Available											
11	U	Available											
12	U	Available											
13	U	Available											
14	U	Available											
15	U	Available											
16	U	Available											
17	U	Available											
18	U	Available											
19	U	Available											
20	U	Available											
21	U	Available											
22	U	Available											
23	U	Available	C:\	-2014-07-23-Linesha	1	acetone-d6	no Decoupling			Shimming Test	00:01:07	TM	Set Start
U	U	Available	C:\	TM-2014-07-23-Linesha	2	Acetone	ace - C 2D_CL3H4_H5			Shimming Test		TM	Set Start
F2	U	Available	C:\	TM-2014-07-23-Linesha	1							TM	
U	U	Available	C:\	TM-2014-06-19-HI-ROE	17	CDCl3	2D_HI_ROESY				00:10:50	TM	Set Start
24	U	Available											

#	Date	Holder	Name	No.	Experiment	Load	ATM	Lock	Shim	Acq	Proc	User	Disk	Title/Orig	Remarks
2	2014-07-23 13:51:21	2	RM-2014-07-23-Asp	2	2D_HI_NOESY	✓	✓	✓	✓	✓	✓	RM	C:\User\Dat a\Genken	1H-NOESY on Asphatene with no C4Ac	
1	2014-07-23 13:35:18	2	RM-2014-07-23-Asp	1	1D_HI_NoDec	✓	✓	✓	✓	✓	✓	RM	C:\User\Dat a\Genken	1D-Protion of Asphatene No C4Ac	ref: reference peak not found default calibration done

Search Preceding include previous runs

BACS/SampleCase/SamplePro (R5232) Busy until: No Job! Day Experiments: 00:00 Night Experiments: 00:00 User: TM

Figure 1 IconNMR home page with a user logged in.

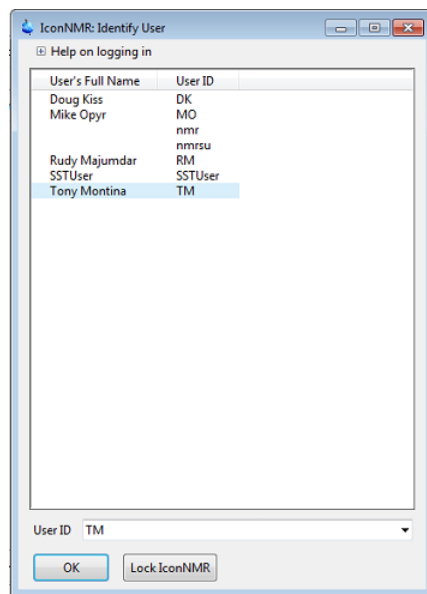


Figure 2 IconNMR login box

(2) You need to put your sample into a spinner and adjust it to the correct height. This can be done with the following instructions:

(a) The tube height guide (at right) has both a blue plastic component (the “spinner”) and a colorless glass component (the “guide”). Place the spinner into the top of the guide as shown, then carefully load your sample tube into the spinner so it touches (but is *no lower than*) the bottom of the glass guide. ***If your sample tube is too low when it is placed into the instrument, it will cause damage.***



(b) ***Without*** removing your NMR tube from the blue spinner, lift it out of the guide.
 (c) Wipe the outside of both the spinner and NMR tube with a Kimwipe.

NOTE: A user should always handle the NMR tube by the top of the tube and not the body. Doing so prevents the grease and dirt from your hands getting into the instrument and probe.

(3) You should now go into the lab and look to see which sample holder locations are available. Just because no experiment is listed in a location does not necessarily mean that a sample is not in that location. For example, slots 23 and 24 are always reserved for the lineshape and temperature calibrations test samples, respectively, but there is not always experiments listed under slots 23 and 24. Place your sample into one of the free sample holder locations.

(4) You now need to left click on your sample holder number in the IconNMR window and left click on the “Add” button located just below the experiment table (shown in figure 3). Be sure to specify how many experiments you would like added to the location by changing the number immediately to the right of the “Add” button. Alternatively, you can simply double click on the holder location and a single new experiment will be created. You should familiarize yourself with the buttons shown in figure 3, as these will be used regularly throughout the rest of the manual and just referred to as buttons.



Figure 3 IconNMR Buttons

(5) The add button has now created a new experiment in your sample holder location. This new experiment is shown in figure 4 for the example of holder location number 1.

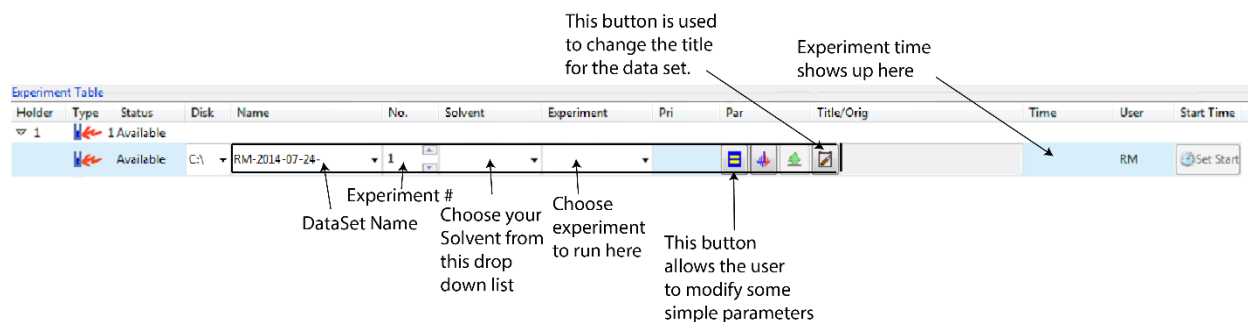


Figure 4 A new experiment created in holder location #1

You need to follow these simple steps to set up your experiment using IconNMR:

- Create a name for your data set. All data sets acquired on the 700 **MUST** start with the users initials followed by the year-month-date and finally by a sample name of some sort. All of your data gets stored under your supervisor's directory in TopSpin.
- Choose an experiment number for your experiment. By default this is set to one and will automatically increase by 1 as you make more experiments for the same sample holder location.
- Choose the experiment that you wish to run from the drop down indicated in figure 4. Each probe is capable of carrying out a specific set of experiments so be sure to check the "700 Experiments by Probe" document to ensure that the probe currently installed can carry out the experiment you are interested in.
- Create a title for your data set. This can be done by left clicking on the title button indicated in figure 4. Be sure to provide a title that provides as much information as you and your supervisor require.

- (e) The parameter button may be used to control some basic acquisition and processing parameters, such as NS and LB.
- (f) The total estimated experimental time will show up to the left of the title box.

(6) More advanced modifications of the processing and acquisition parameters can be carried out by selecting the parameters menu at the top of the IconNMR window and then left clicking on one of the following:

- (a) Edit all Acquisition Parameters (allows full access to “AcqPar” tab of the dataset)
- (b) Edit Processing Parameters
- (c) Pertinent Acquisition Parameter Edit (edit only the short list of parameters)

This will automatically open up topspin and allow you to modify the parameters. Once you have modified the parameters you **MUST** left click on the return to IconNMR button that is circled in red. Also, be sure to do this with the correct data set selected or it will open all of your data sets one by one!

(7) Additional experiments can be created for your sample location by simply selecting your data set and then left clicking on the “Add” button. This will create additional experiments under the same sample location and with both the same experiment name and solvent. The number located immediately to the left of the “Add” button allows the user to specify exactly how many additional experiments they would like to create.

(8) Submit your experiments to the IconNMR queue and you will be given an estimated completion time. This can be done by highlighting all of the experiments you want to submit and left clicking on the “Submit” button.

(9) If you wish to cancel any of the experiments that you have submitted you must highlight the experiment and left clicking on the “Cancel” button. Once the experiment has been cancelled it will remain in the experiment list for that sample changer location but it will not be editable. In order to edit or change the experiment you must left click on the “Edit” button.

If you wish to re-submit the experiment before or after editing this can be done by once again highlighting the correct experiment and left clicking on the “Submit” button.

(10) Log out of IconNMR and leave it open for the next user to setup their experiments.

(11) Once your experiments have been completed your sample will be automatically ejected from the magnet and you should be sure to remove it from the sample changer as soon as possible. The longer you leave your sample in the changer the more likely it is that it will be tying up a slot that is needed by another user. After removing your sample you should log back into IconNMR and delete your experiments. This can be done by highlighting the finished experiments and left clicking on the “Delete” button. This same “Delete” button can be used to delete experiments from your sample holder location at any given time. After deleting your experiments please log back out of IconNMR.

NOTE: Your sample should leave the lab with you!

No samples should be left in the 700 MHz NMR lab.

(12) IconNMR will automatically process all of your data using the processing parameters specified in the “ProcPars” tab of your data set. Your data can be retrieved for viewing and further processing on either of the two processing computers. One of these computers is located inside EP1301 and is shared between the 500 and 700 instruments, while the other computer is located outside of the 300 instrument room and is used primarily for processing 300 data. However, any of these processing computers can be used to process data from any of the instruments. Instructions for copying and moving data on the processing computer outside of the 300 lab are provided next to the computer on a laminated sheet. In order to process your data on the processing computer in EP1301 you must follow these steps:

- (i) Open windows explorer on the processing computer
- (ii) Open the network drive corresponding to the system with the data you are interested in. For example, if you are looking to process data from the 700 system be sure to open up the drive labelled as “700 Data”.
- (iii) The data is network mounted to this drive and is in read only mode. Therefore you must locate your data on the network drive and copy it over to the appropriate folder on the c: drive (c:\300Data or c:\500UserData or c:\700Data).
- (iv) you must now mount this data as a directory in the TopSpin Browser panel.