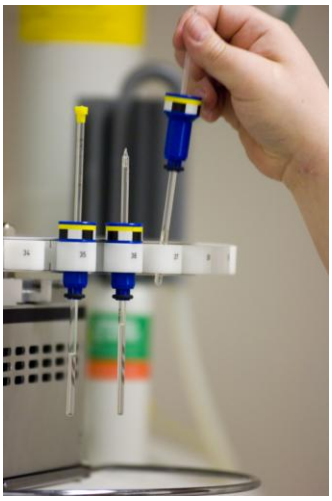
Automated Acquisition and Processing of NMR Samples on the Avance II 400 Spectrometer at the console workstation using ICONNMR

In order to submit samples for the Avance II 400 NMR spectrometer, you will need to be in a course or conducting research at Indiana State University, Rose-Hulman Institute of Technology, or Saint Mary-of-the-Woods College. In any event, you will bring your prepared sample to the NMR room and load it into the sample rack as follows.

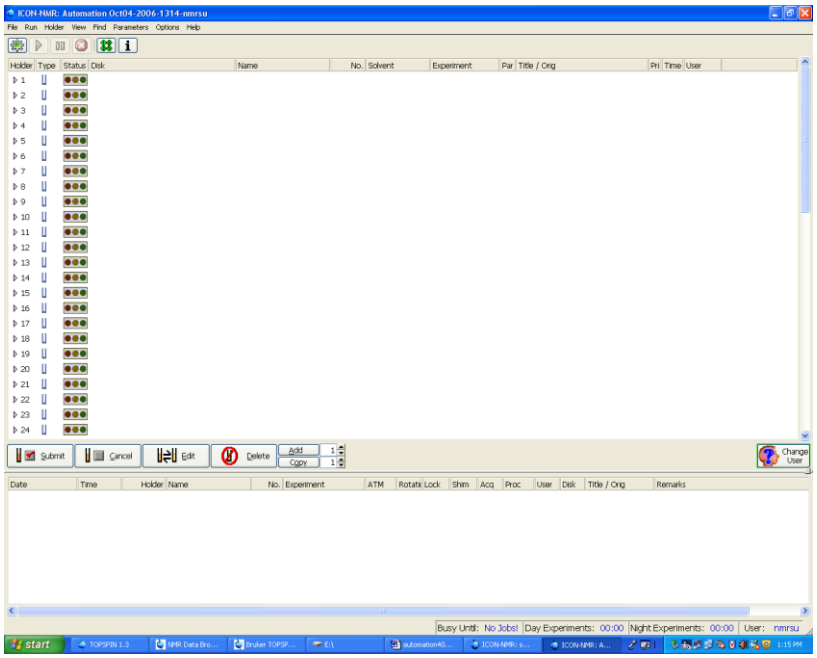
Wipe the outside of the NMR tube with a Kimwipe. Place the tube into one of the blue spinners in the blue suitcase storage rack on the counter. Only insert the sample an inch or two into the spinner and then remove the tube and spinner from the rack. Wipe the spinner off with the *same* Kimwipe and place the sample in the clear plastic depth gauge on the counter (this should be square in cross section, not round). Press the tube gently until it bottoms out in the gauge. Then remove the tube and spinner from the gauge and wipe the lower portion of the NMR tube with the *same* Kimwipe. From this point forward, only handle the tube by the upper part.



Note: once you have set the depth, do not touch the spinner. This can alter the depth of the sample and give you a poor spectrum, or (worse) cause the spinner not to spin, or (worst) bottom out the sample and break the tube in the NMR (very expensive).

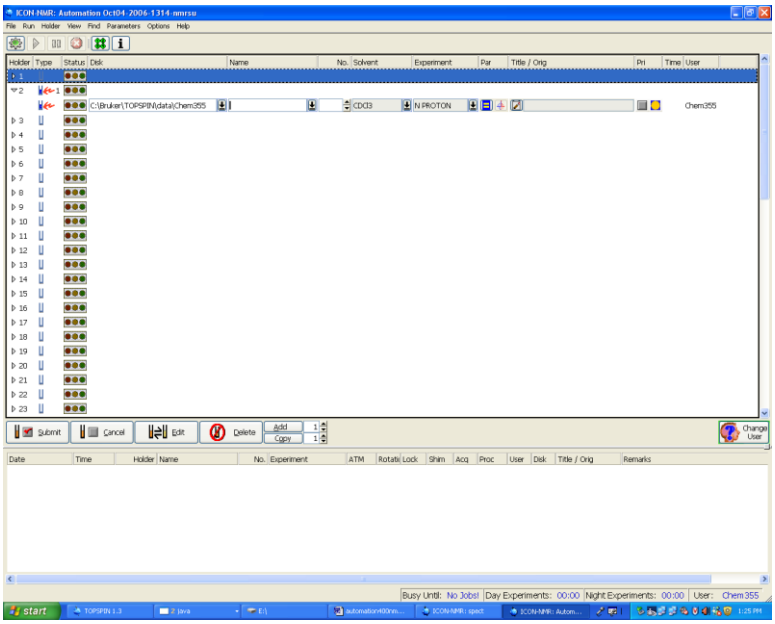
Place the sample in the Sample Changer carousel in the slot to which you have been assigned. If you do not know your slot, ask your instructor or supervisor.

To queue the sample into the automation sequence, go to the NMR console workstation and log into ICON-NMR. The program should be active and an icon should be present on the taskbar. Once you locate the icon, bring up the automation window, which should appear as it does on the right. There may be existing samples in the queue, but the general appearance will be similar. To login, click the **Change User** icon on the right side of the automation window. This will bring up the login window if it is not already in front. Select your username from the list and hit OK. Then login using the password you were assigned in class or by your research advisor.



Only use the NMR during the time to which you have been assigned and only run the sample in the holder to which you have been assigned. Otherwise you may not be taking spectra of someone else's sample instead of yours.

Select the holder to which you have been assigned. Either double click on the slot or push the **Add** button. It will bring up the line dialog on the right. The directory for the file will already be filled in for you (make sure it is the one for your class).



Type in the Name as yourname-samplename (ex. Fitch-acetylation). This will save your data under that filename. If you click the arrow to the right, a set of default filenames is available to use, but in general, you will not use them.

Next put in the experiment number (No.) to the right of Name. By default this will be 1, but use the numbering scheme which your instructor tells you (ex. 1 for proton, 2 for carbon, 3 for DEPT, etc.).

Next select the solvent. Typically this will be $CDCl_3$ (default), but other solvents are available on the dropdown menu. Make sure the solvent matches the one you are using as the referencing and shimming depend on this.

Next select the experiment you want to run from the drop down menu. There are two types of experiment. The first, preceded by N are normal experiments which are single ordinary experiments, such as proton and carbon. The C stands for composite, which include most 2D experiments. For example, a COSY is generally preceded by a proton experiment. Selecting the COSY will automatically queue up a preceding proton experiment. Once a 2D composite experiment is selected, a reference will appear following the experiment and referring to the 1D associated with it. It will not run the 1D experiment twice.

To the right of the experiment are two icons. The first which looks like an "=" allows you to modify certain parameters, typically the number of scans (NS) and the number of data points (TD). The number of scans you need will depend on the amount of sample you have. If you 20 milligrams or more, the default values of sixteen scans will be sufficient for proton and 1024 scans for carbon are sufficient. If you have less, you will need more scans. You will not need to change the number of data points, so leave TD with the default value.

The second icon controls the locking and shimming. You should not have need for this and it is generally grayed out.

Next, type your title in the title box. The title should include your name, course, description of the sample, type of spectrum, date, and your initials. An example is shown below.

Rick Fitch
Chem355
Elimination product *(do not identify the compound unless you know for sure what it is)*
Chromatographed (*it is useful to include details as to the quality of the sample)*
1H CDCl3
10-22-06
rwf *(these might be someone else's initials if they ran the experiment for you)*

When you are satisfied with the quality of the title, click on the Set and Copy Title button. This way, you can paste a title into subsequent experiments as a starting point.

You have now completed your experimental setup. If you wish to run multiple experiments on the same sample, you simply click the Add button again and a second experiment will be created.

When you have created all of the experiments you wish to run, you should submit the sample by clicking and dragging across all of the experiments you have created and then click Submit at the bottom of the queue window.

Once you have submitted your sample, you can monitor its progress in the window at the bottom. It will run and produce an automatically processed spectrum in pdf format. A pdf file is mailed to the email address you provided to your instructor/research advisor when it is generated. This is handy if you need to look at your spectrum off campus. Otherwise, you can process the spectrum directly using Topspin at the spectrometer or remotely if someone needs to acquire a spectrum immediately after you at the console. A dataset will also be emailed to your class email account as well.

Processing of the spectra may be done on the computers in S51J. The data are stored on the chemistry server at http://carbon.indstate.edu/nmr. The data are indexed by course and contain the data in the directory structure used by Topspin, the NMR spectrometer software. The data may be downloaded in a zipped format. You will need to make a directory on your computer that has the format data/user/nmr. This can be done anywhere on your computer, preferably on a flash drive (at least 128MB recommended). Your chemistry computer accounts do not have enough space to hold many spectra, especially 2D and 3D spectra.

Once you have downloaded your spectra, you may process them using Topspin just as if you were sitting at the spectrometer. Processing details are in the **Processing NMR data using Topspin** document on the chemistry department website at http://www.indstate.edu/chemistry/AVII-400.htm .