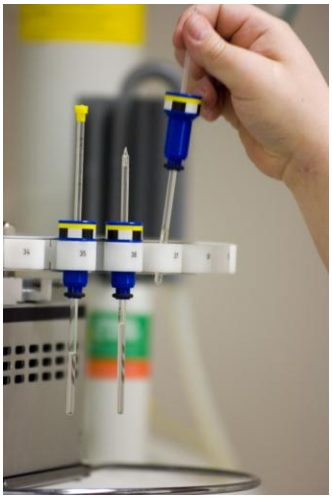
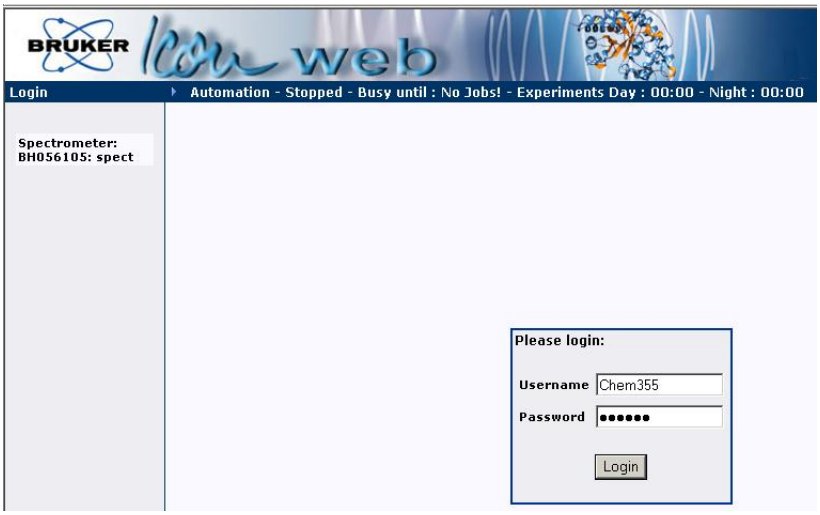
Remote Acquisition and Processing of NMR Samples on the Avance II 400 Spectrometer Using ICONWeb.

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 square plastic depth gauge on the counter. 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.



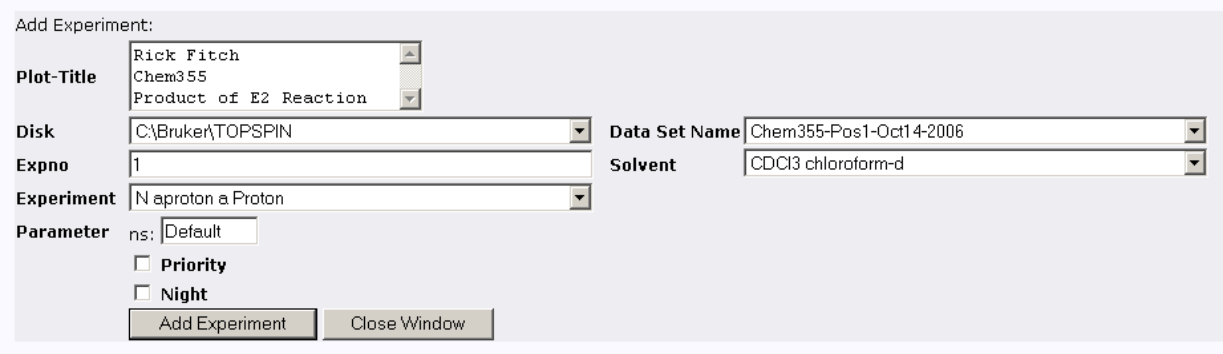
To queue the sample into the automation sequence, go to a computer and log into ICONNMR at http://nmr400.indstate.edu:8015/icon . The website is firewalled and is only available on the ISU, RHIT, or SMWC campuses. If you are unable to connect to the site from one of these locations, it generally means that ICONNMR automation is not running on the spectrometer. Let your instructor know as soon as possible if you cannot load the webpage. Once connected, you will see a login for ICONWeb as shown below. Login with the username and password your instructor gave you. Be sure to type them exactly as you were given. The username and password are case-sensitive. Also be sure to include the extension in your username (e.g. CHEMISTRY/orgn1a). Otherwise, the interface will not recognize you.



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 be taking spectra of someone else's sample instead of yours.

Once you are logged in, you will want to put in the information for your sample.

Select the holder to which you have been assigned. Push the **Add** button. It will bring up the following dialog.



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.

John Doe
Chem351L
Grignard product *(do not identify the compound unless you know for sure what it is)*
1H CDCl3
10-22-06
JD *(these could be someone else's initials if they ran the experiment for you)*

Next select the file directory from the drop down menu. For classes, there will be only one option. Likewise, the Data Set Name will have only one option (formatted as Coursename-Position-Date, see above).

The experiment number is the lowest available number for that filename, usually 1. If you wish a different number, you can input one.

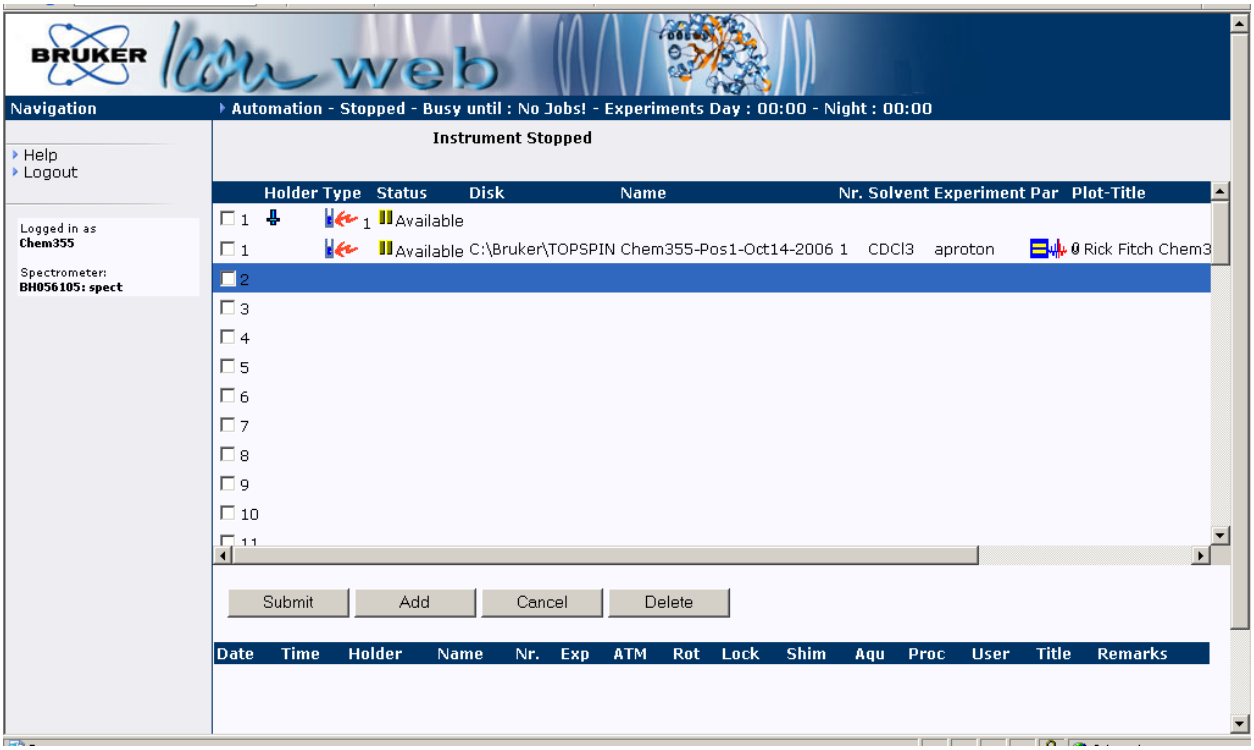
Next, select the appropriate solvent from the drop-down menu. Generally this will be $CDCl_3$.

Next, select the experiment you wish to run. Be careful, for many experiments there is more than one "flavor" of the experiment. For example, for a proton experiment, there are at least six different formats. The most common you will use is aproton, which is a 16 scan experiment with a plot output of 10.5 to -0.5 ppm. Likewise, there are several carbon experiments, but you will most often use acarbon, which is a 1024 scan carbon with a 210 to -10 ppm plot range.

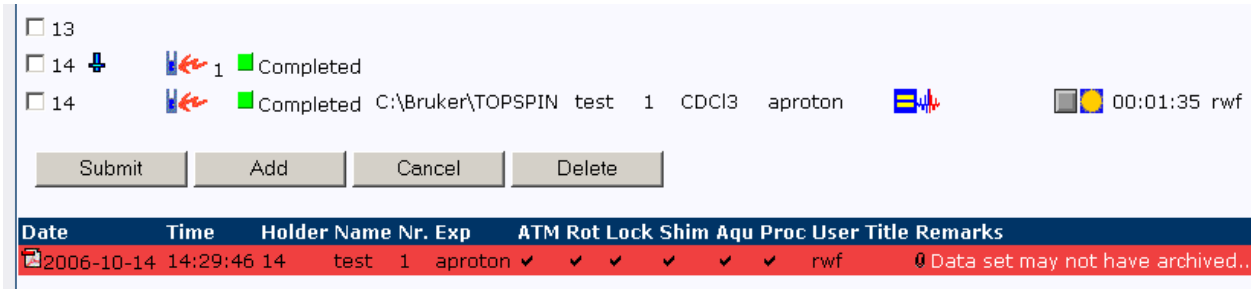
You can select priority or night runs or priority handling. However the software decides if a night run is necessary based on the length of the experiment and classes are not allowed priority handling, so it is best to leave these boxes unchecked.

Once you are satisfied with your sample information, select **Add Experiment**,

The display will now appear as follows, with your sample information. To submit your sample to run, select Submit at the bottom. Your run will begin when the instrument is available. ICONNMR will also tell you the status of the instrument. Notice it says Instrument Stopped at the top of the display. At various times it may report the instrument as Idle, or show the status of an automated acquisition. It will accept new samples as long as the automation is active.



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. **If you are using a community workstation, you must logout once you are finished.** Otherwise, the next person may inadvertently run another sample under your username or cancel your sample. The sample shown below has run and completed. Notice the pdf icon next to the dataset (shown in red). You can look at the spectrum by simply clicking on the icon. A pdf file is also mailed to you're 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.



The directions above are sufficient for basic users. A dataset will also be mailed to your class email address which you can use to reprocess the data to refine integration, phase, peak pick to

obtain coupling constants, expansions and other details of your spectrum that you may not get from the pdf alone.

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 .