

# Automation using ICON-NMR

For use with Topspin 1.3      Revised 04-13-05

**NOTE: If you do NOT hear the cryoprobe “chirping” and/or the “WARM” or “ERROR” lights are lit on the cryopump unit, IMMEDIATELY tell Joe or Sara and DO NOT PROCEED! You can damage the probe by trying to acquire data on it while it is warm.**

## Part I: Logging Into the Computer

1. **If the computer is logged in and Icon is already running, skip to Part II.** If the computer is not logged into already, log in as the user “icon”.



2. Open Topspin by double-clicking the icon on the desktop.
3. Type “rfshim” to be sure the reference shims are loaded.
4. Type “iconnmr” to start the Icon software.
5. Click on Automation.



6. In the Icon-NMR Set User window click on the username “icon”.
7. Enter the Icon user password (same as to log into computer) and press Return.

## Part II: Setting up an Automation Run

1. Place your samples in the sample changer.

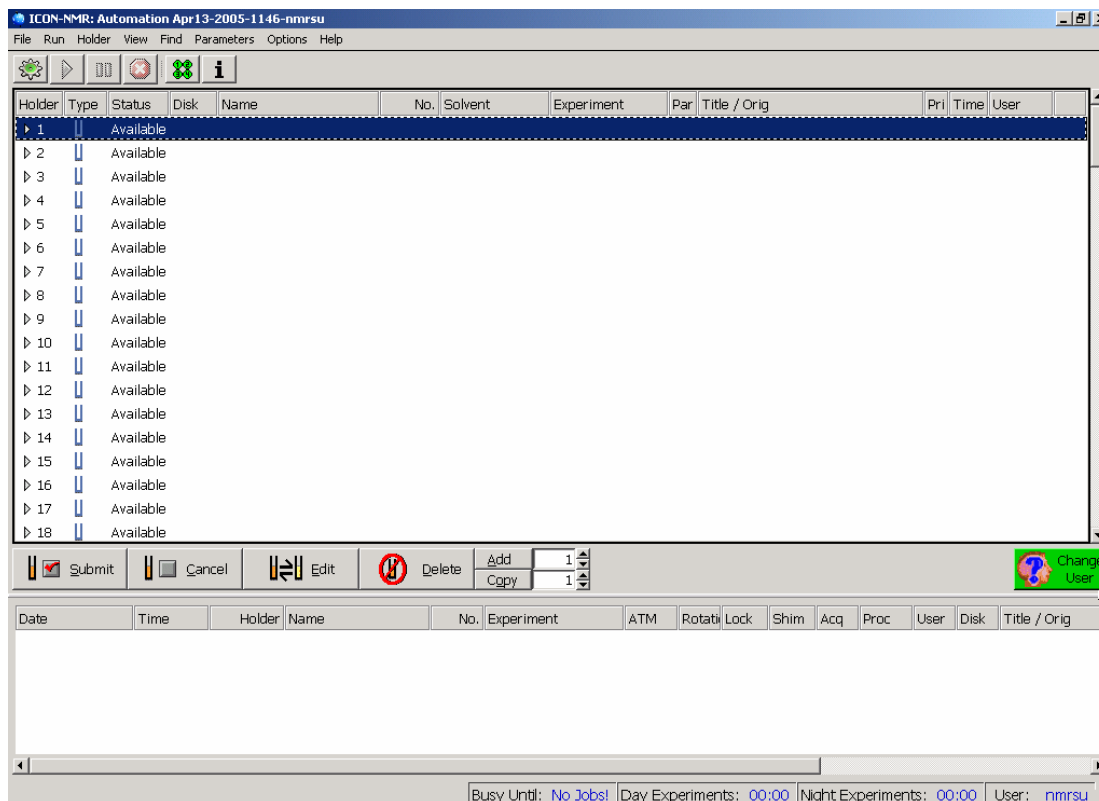
***Inner rack: Samples 1-60***

***Outer rack: Samples 61-120***

**WARNING: Check the sample tube depth with the depth gauge for each sample!!!**

**WARNING: Tubes must not be longer than 7 inches**

2. The Setup screen will open:



3. Double-click on the number in the “Holder” column to indicate the first sample’s location.
4. Type a filename for your sample under the “Name” column.
5. The experiment number will default to “10”. You can change it by typing “1” if you prefer.
6. Choose the solvent you are using from the dropdown list.
7. Choose the experiment you wish to run from the dropdown list, the most common experiments are shown below.
  - a. PROTON = standard 1D proton with 32 scans
  - b. COSYGPSW = COSY with gradients, magnitude mode (similar to “ez\_cosy”)
  - c. HMQCGP = HMQC with gradients (similar to “ez\_hmqc”)
  - d. LC1D12 = similar to “multsolvsup” Note: The software will select which peaks to suppress.

**NOTE:** If you select a 2D experiment it will automatically run the standard 1D proton experiment first. You may alter the number of scans, sweep width and offset (o1) on the “pre-experiment” if needed to get proper setup for your 2D experiment.




13. If you want to perform multiple experiments on a sample (ie COSY and HMQC):


e. Click .

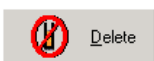
f. Repeat steps 7-9 for the new line added to that sample.

g. To delete an experiment, highlight that line and click .

14. Repeat steps 3-13 to add more samples to your run.


If you want to copy the same parameters for all your samples, change the number in the box to equal the number of samples remaining next to “copy” and click the  button.

15. Highlight your new sample information and click . You can now change your sample names, experiments, etc. following steps 4-13.

16. If you need to delete a sample line, highlight it and click .

### Part III: Starting the Acquisition

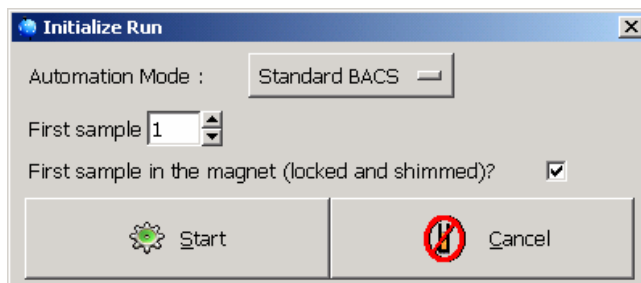
1. Highlight all the samples using the mouse.


2. When you are ready to perform the acquisition, click .

3. Click the  button.

**NOTE:** If you have added samples to an existing, queue that is already running, you do not need to click “submit” but you do not need to click the “Go” button again.

4. If your first sample is not in holder one, change the number next to “First Sample”.

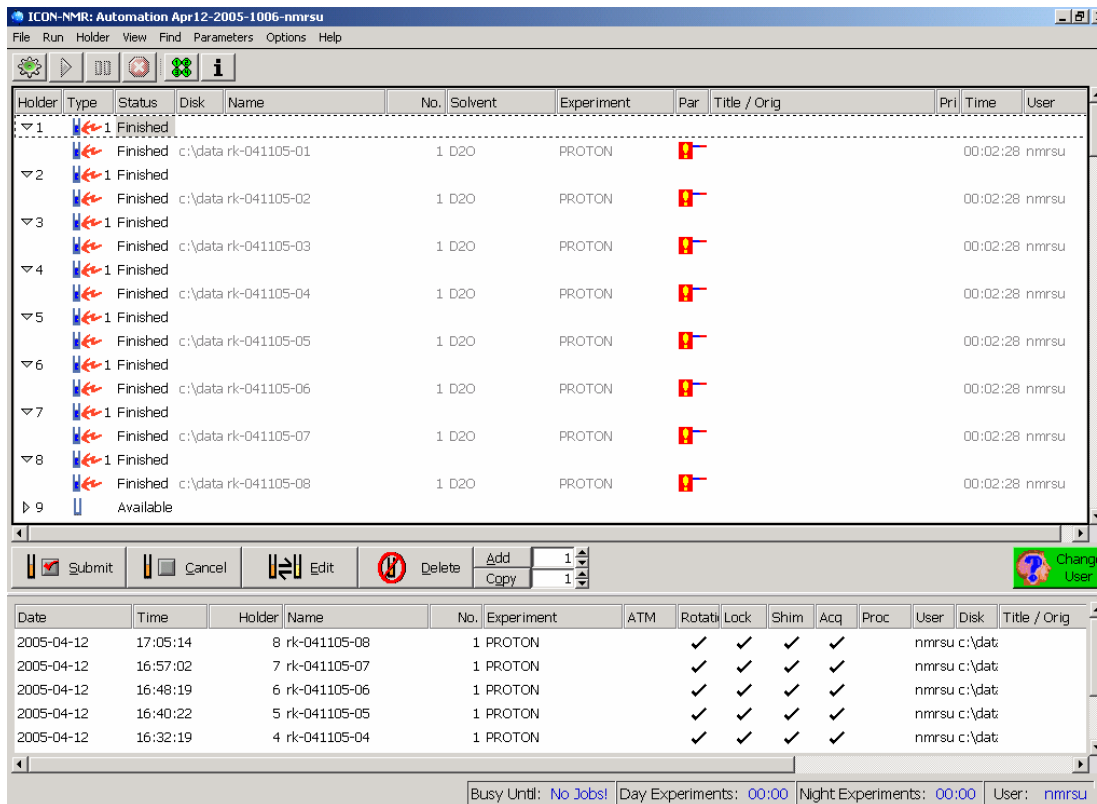


5. Click on  to start the acquisition.

6. While Icon is running, the status of the acquisitions can be seen in the upper right corner of the setup screen. The process running will be listed on the left, and the icon representing the process will be flashing.






7. You can check on the current acquisition while it runs by clicking on Topspin in the taskbar (Icon minimizes it).
  - a. Open the filename you wish to look at.
  - b. Type “tr”, “efp” and “apk” if you are running a 1D experiment.
  - c. Type “xfb” to look at your 2D experiment data.
8. When the acquisition is complete, the screen should look similar to this:




9. Things to look for when it's done:

- All the boxes in the bottom of the screen have a ✓ in them.
- If you ran multiple experiments on the same sample (ie Proton and COSY) there will not be a ✓ for Lock and Shim on those lines.

- If there are any , look at which column the error is in.
- If there is a  for lock or shim:
  - Did you have a sample in that holder? Remember that samples 1-60 are the inner rack, 61-120 are the outermost rack
  - Was the proper solvent selected?
  - Is solvent deuterated?
  - Is the sample height about 1.5-2", and is it free of air bubbles or precipitate?
- If there is a  for acquisition:
  - Open the data file in Xwin-NMR. Type "efp" if it was 1D data or "xfb" if it was 2D data. See if anything was acquired.
  - Make sure that the right sample was taken from the sample changer location.
  - If no data was acquired, get Joe or Sara to verify a hardware failure.

10. If all of your samples are done running and no one has queued a sample for after yours, click

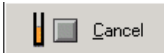
on the  button to release the instrument.

11. Remove your samples from the sample changer. Sign the logbook.

**Note:** DO NOT log out of the computer and DO NOT leave a sample in the magnet when you are done with an automation run.

## Possible Problems with Icon-NMR

- The cryoprobe “chirping” noise cannot be heard or the “WARM” or “ERROR” light on the cryoprobe pump unit is lit or there are errors in the window on the laptop.
  - ***IMMEDIATELY*** notify Joe or Sara.

***WARNING: DO NOT attempt to acquire data or you WILL damage the probe.***
- The “Lock On/Off” light is flashing during an acquisition.
  - The Lock light will flash during a 2D experiment that involves gradients.
  - If the light is flashing during a 1D experiment, the gradient shimming routine did not relock when it was done. (This is a known bug in the software that Bruker will fix in the new version.) Let the experiment finish, and look at your data. You may need to reacquire that sample if your lineshape is poor or your peaks have drifted during the acquisition.
- When Icon is done, it does not necessarily put the tube back from where the sample changer got it. It is *highly recommended* that you label your tube a fine-point Sharpie.
- What if you realize that your sample tube is too long or in a J-Young tube?
  - Samples must be in standard 7” long NMR tubes, such as the New Era NE-UL5-7, Norell 509-UP, or Wilmad 535-PP-7 or 541-PP-7. The NE-UL5-7 tubes are available in the stockroom. The probe cannot be used with any other tubes, and the sample changer will break any that are longer than 7 inches.
  - You can remove sample from the sample changer and transfer the liquid to a 7” long 500 NMR-rated tube. If the sample isn’t in the correct holder, Icon will skip it. Samples can be added to the queue later by following steps 8-19.
  - You can “un-queue” your sample by selecting the holder number with the mouse and clicking on . Remove the tube from the sample changer after you have removed it from the queue.
- If the sample changer begins to do anything strange, such as drop tubes on the floor, move the racks while the arm is down, or if your sleeve gets caught in the rack or arm, you can disable the sample changer by pressing the large red “panic button” on the front. Notify Joe or Sara as soon as possible.