Running an experiment on the autosampler

The program running the sample changer is called ICON-NMR. All users will get their own account which is issued by Kristina, after having had an introduction and signing “the rules and regulations for the sample changer”

1. Mark your NMR-tube with a water proof pen
2. Log in
   Click on and the “set user” window will appear, mark your name and click OK and give your password. If it doesn’t appear, make the ICON-NMR window a bit smaller and look for the “set user” under icon-nmr in the task bar in the bottom of the screen.
3. Mark the next available number (consecutive order, ie 1, 2, 3 etc) by double-clicking on the number. If the list is full and you can’t find anyone from the ICON-NMR responsible group, delete one of your already finished runs.
4. Take a Kleenex with ethanol and whip off the tube before you put it in a blue spinner
5. **Check the sample tube depth with the plastic depth gauge for each sample!!!**
6. Insert your marked samples into the sample rack having the corresponding rack holder number.
7. Log in again, if you have been logged out.
8. Double click on the number in order to enter your sample-specific information.

Move along with the tab key:

**Disk** Preset for each user (/opt/topspin).

**Filename** Type the filename for your sample, you can use -- or _ but not space, dots etc. It will be a folder, where you can have several runs separated by exp number and title (see below).

**Exp No** Default is 10. The system will recognize previous experiments with an identical filename and update accordingly. You can also choose an arbitrary number.

**Solvent** Choose the solvent you are using from the dropdown list by clicking on the arrow and mark it.

**Experiment** Choose experiment you wish to run from the dropdown list. Day samples for Maximum 20 minutes at a time can be added. When they are finished you are allowed to add a new set of samples/experiments for 20 minutes. To the experiment time you must add 3 1/2 – 4 min. per sample for locking, tuning, shiming and so on. That means one sample $^1$H=5min (1 ½ exp time + 3 ½), 4 samples $^1$H=20 min, one sample $^1$H + $^{13}$C (exp time10min)=20 min

**Parameters** To change number of scans (ns) or the delay time (d1) click on the icon

**Orig/Title** Clicking on the pen symbol allows you to enter a title. Finish by clicking on set title.

**Pri** If your short run samples result can wait until the next day run them during night by clicking on the sun so that it turns to a moon
9. The Submit/Delete menu

If you want to add another experiment to the same sample you should click on add. Fill in the new experiment and title. Exp number will be incremented automatically.

If you want to copy the same parameters for more samples, change the number in the box to equal the number of samples remaining and click the copy button.

Clicking on submit will put your sample into the queue. If you submit when standing on the sample position number row, all the experiments on that position will be submitted. The total experiment time, incl. shimming, acquisition is \( \approx 5 \) min for a \(^1\)H NMR with 16 scans.

If you want to change your input data, highlight the row by clicking on it and then click on Cancel. Your sample is now “un-queued”. Then click on Edit. Don’t forget to click on Submit again when you are done.

If you need to delete a sample line, highlight it and click. Don’t forget to remove the tube from the sample changer after you have removed it from the queue.

10. Log out by clicking on

11. To see when the run is finished

Here can you see when the last run in the queue is finished, included tuning locking, shimming (3 \( \frac{1}{2} \)-4 min per sample and experiment time)

To the booked experiment time, you must add 3 \( \frac{1}{2} \)-4 min per sample.
More about the program

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.

![ZG In Progress]

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.

![Error Table]

- If there is a ❌ for lock or shim:
  - Did you have a sample in that holder?
  - Was the proper solvent selected?
  - Is solvent deuterated?
  - Is the sample height about 4-5 cm, and is it free of air bubbles or precipitate?
  - Is the NMR tube being placed in the right position in the spinner?

- If there is a ❌ for acquisition:
  - See if anything was acquired.
  - Make sure that the right sample was taken from the sample changer location.
  - If no data was acquired, get Kristina to verify a hardware failure.

- 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 Kristina as soon as possible.