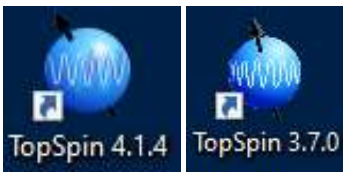
Use of ICON-NMR for Automated NMR Operation

The contents of this handout is focused on the use of IconNMR on the NMR spectrometers in the CRL basement.

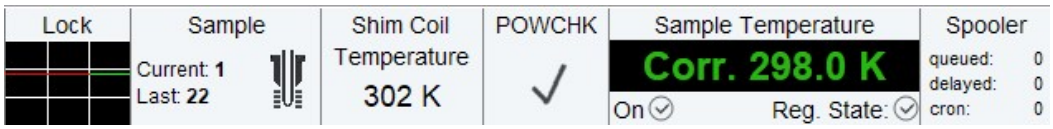
It is essential that all user time on the hands-on spectrometers is booked online using the NMR instrument booking system and users have attended a hands-on training. Please note that the ICON-NMR interface may only be used for **ambient temperature** experiments.

Opening IconNMR from TopSpin

As with manual Topspin operation, login to the spectrometer with the "*nmr-user*" domain account and open Topspin. The TopSpin icon will be available from the desktop. Click on the icon and wait until the main TopSpin window opens. Note the version may differ from the examples below:

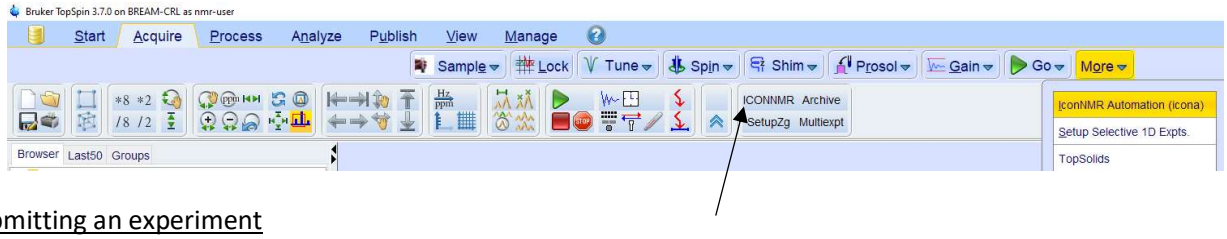


It is advisable to check the probe temperature is properly equilibrated - the current probe temperature may be seen in the lower right status display panel and the default is 298.0 K.



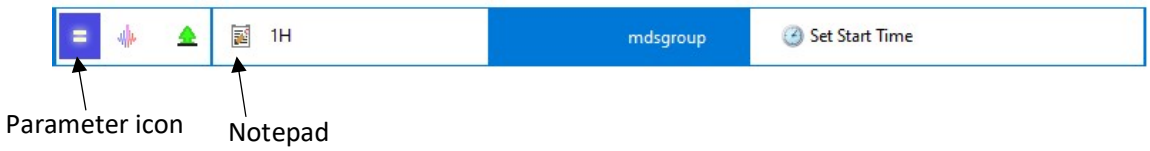
Check the autosampler is in use and is not displaying a red error light.

Check whether ICON-NMR is already open. If necessary, open a new ICON-NMR interface window by clicking on **ICONNMR** on the main toolbar, or under **Acquire - More - IconNMR** Automation, or by using the command 'icona'. Log in with your group user account.



Submitting an experiment

To submit your first sample, double click on **Available** next to an available Holder position (e.g. 1) which will open the sample entry menus. Enter your sample name, starting with your initials (**DO NOT** include non-standard characters such as * ! " ; etc and it is advisable not to include spaces in data set names). Select your solvent and experiment. If you wish to edit the number of scans (NS) click on the ***Parameter*** icon (blue box with yellow strips). Click on the ***Notepad*** icon to enter your details and experiment title; ***Set and Copy*** this to save for all experiments.

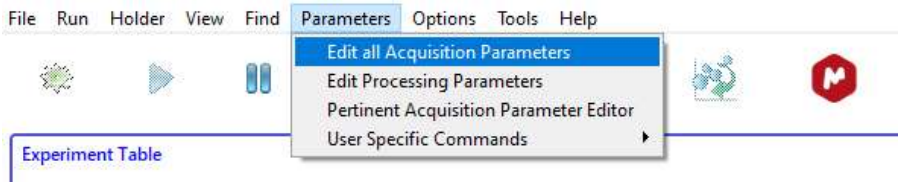


How to queue an experiment to start at a specific time

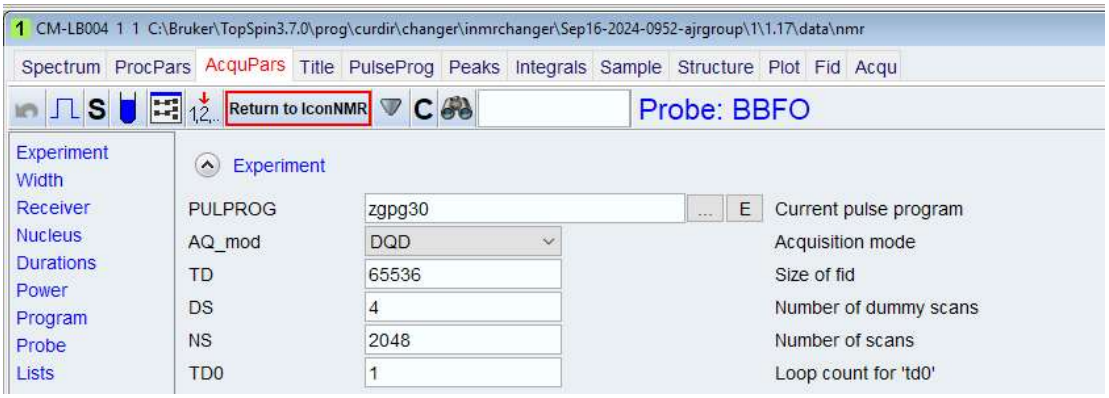
On the far right of the sample entry, click the clock icon, and select the time you wish for the experiment to start. If queuing multiple experiments, set the start time for the first experiment, then add the other experiments you wish to run. Each experiment will then have a time one minute starting after the previous experiment. To remove the start time, click the 'X' icon.

How to view/edit the full acquisition parameter set of an experiment

To view/edit the full acquisition parameter set, click on the experiment you wish to change then click on **Parameters** (top left in Icon) and scroll down to **Edit all Acquisition Parameters**.

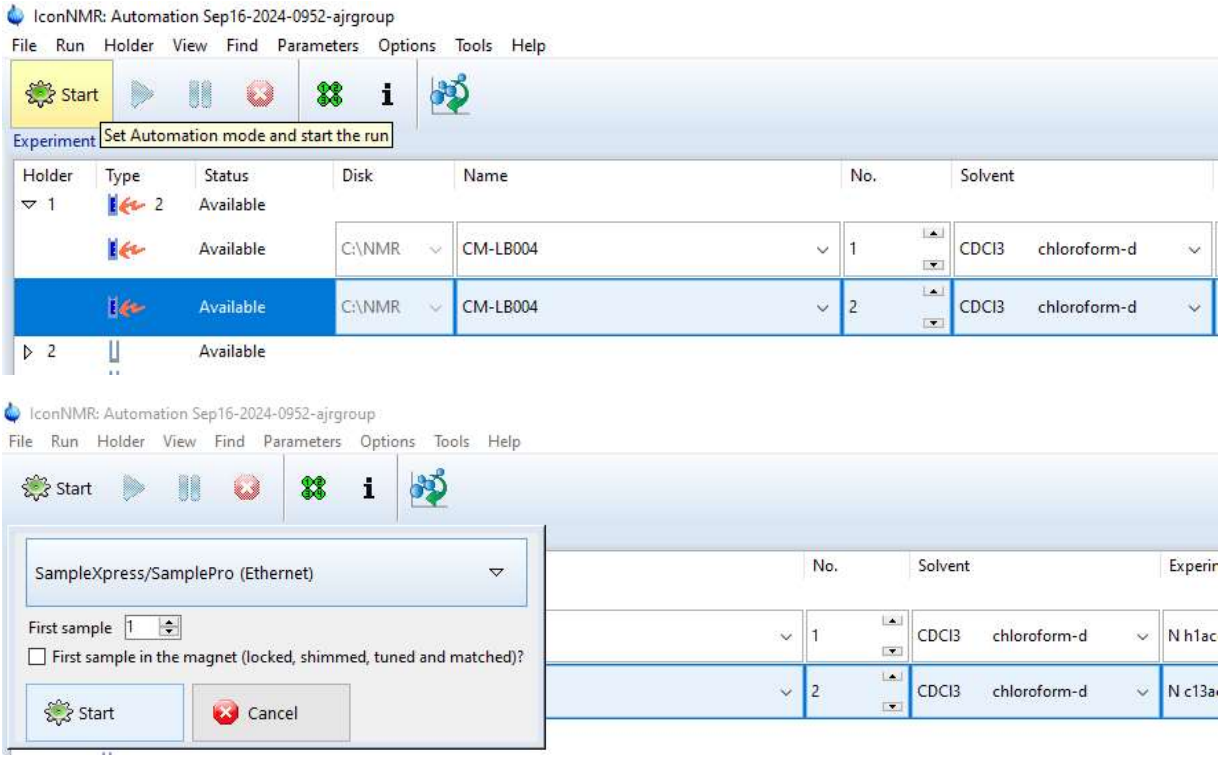


To return to ICON-NMR, you must click on **Return to ICON-NMR**, as shown below, otherwise your changes will not be saved:



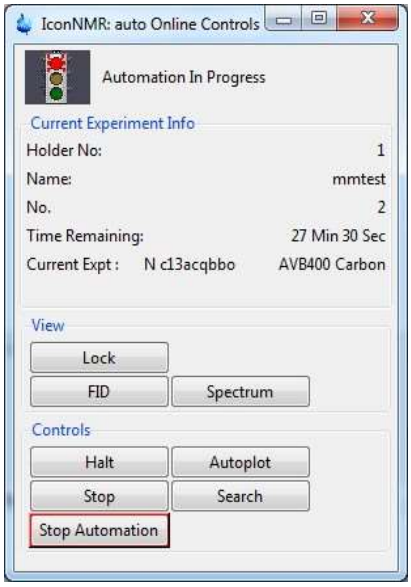
Starting the IconNMR run

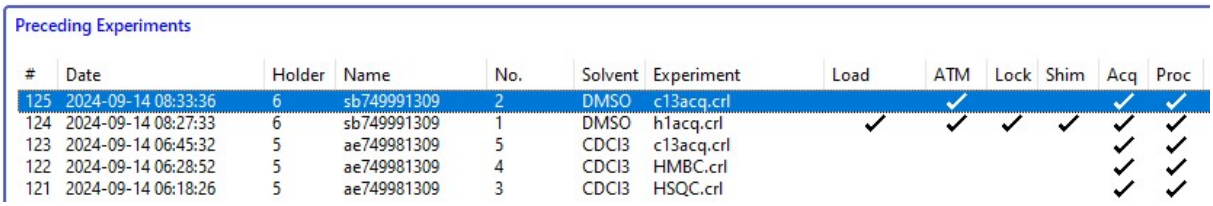
Place your sample in the corresponding position on the carousel. To queue your sample, click on the **Submit** icon (bottom left). Check that all the experiments for your sample have been queued. To start the run in ICON-NMR, click on the green ***Start*** icon (top left cog wheel, see below). In the **Initialise Run** window check that the **First sample** is set to your holder position. [If you have already collected data on your sample (eg to check the ^{1}H is OK) and it is already in the magnet locked and shimmed you may also select "**First sample in magnet (locked and shimmed)?**" to skip these processes]. Next, click on the **Start** button.



ICON-NMR will now perform automatic tuning, locking, and shimming of your sample and automatic acquisition and processing of data. All data produced through ICON-NMR will be archived to group folders on the departmental NMR data server (within the appropriate instrument folder).

To open the **ICON-NMR Online Controls** window, click on the **i** icon (top left). Here you may view the current experiment information, and detail the time remaining on the current experiment. Data may be viewed in TopSpin by double-clicking on any experiment in the lower history window.





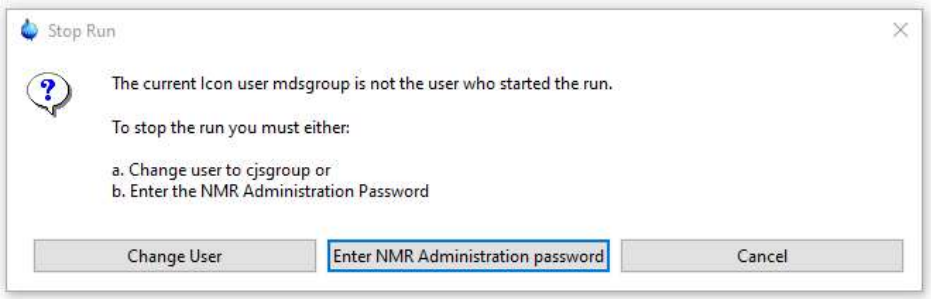
Now queue any further samples (making sure you are logged in with your group account) and place them in subsequent positions on the carousel. To ensure that you do not exceed your booking time, check the experiment run times on the ICON-NMR interface window.

<u>Halting and Stopping the IconNMR run</u>

To halt the run, click on the blue Pause icon. This will pause the run after the current experiment is finished. To continue the run, click the 'play' icon.

Start run play run pause run stop run

To stop the run, you must be in the same group account that started the run. Click the red Stop icon in the top left. If under the incorrect group account, a pop-up message will appear detailing which group account you must be under:



Change to this group account (in the above case, the cjsgroup) and repeat the process. Icon will then confirm you would like to stop the run. Click 'yes' and Icon will stop. Any experiment currently running will immediately finish.

How to halt experiments early

If an experiment is running in Icon, a user may wish to terminate the experiment earlier than expected. To do this, open the TopSpin window and type 'halt' in the command window. The experiment will finish when the next scan (1D experiments) or increment (2/3D experiments) is complete. If an experiment is halted due to another user using time that is not theirs, ensure any other experiment they have queued is cancelled before halting the current experiment.

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