IconNMR User Guide

This procedure provides basic instructions for acquiring NMR spectra using the IconNMR automation program.

Sample preparation

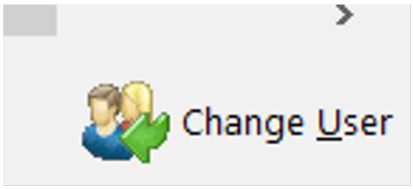
1. Dissolve your sample in an appropriate deuterated NMR solvent. Make sure there is no undissolved material. If there is, you will need to either centrifuge or filter your sample to remove crystals/debris.
2. Transfer about. 550 -700 µL of solvent into a clean NMR tube. Your sample height should be about 4-5 cm.
3. Clean the outside of your NMR tubes with Kimi wipes, then properly gauge your sample in a blue spinner.

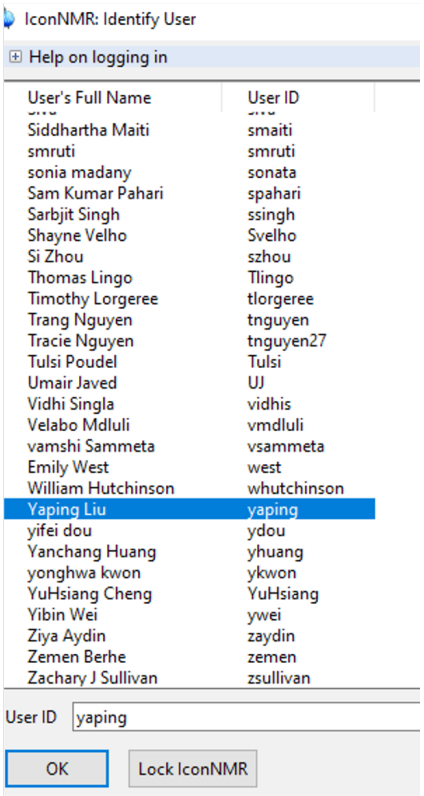
Login and experiment setup

1. Place your sample in the autosampler changer. Remember with the sample holder number you used.

2. Generally, you will find the NMR PC running with both TopSpin and IconNMR open. If so, skip to step 5. If Topspin is not running, double click the TopSpin 3.6.5 icon on the desktop.
3. To start IconNMR, type icon at the command line of Topspin.
4. Click on the Automation button in the IconNMR dialog box that appears.

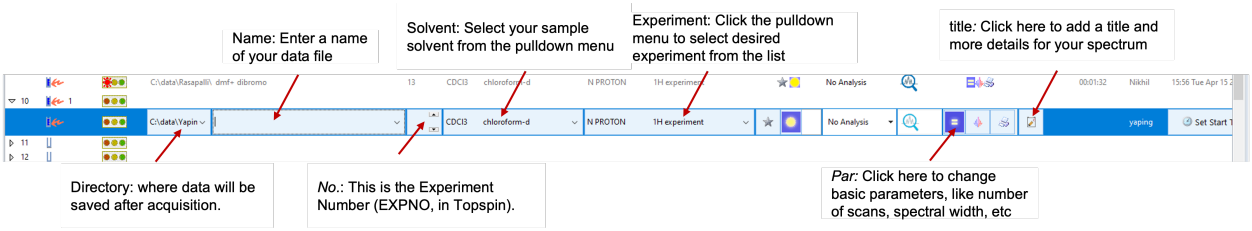
5. Log into your IconNMR account. If a previous user is currently logged in, you will need to click the “change user” button, then log into your account.





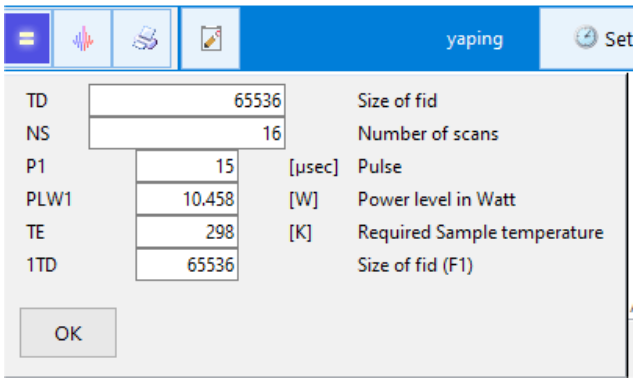
Noted: If your IconNMR account has not yet been created, please reach out to Yaping Liu: yaping.liu@umassd.edu

6. Identify the matching holder number in IconNMR menu and double-click on the corresponding holder number to add an experiment.
7. Enter your details: Data Directory, Experiment Name, Experiment Number, Solvent, Experiment/Parameter Set.

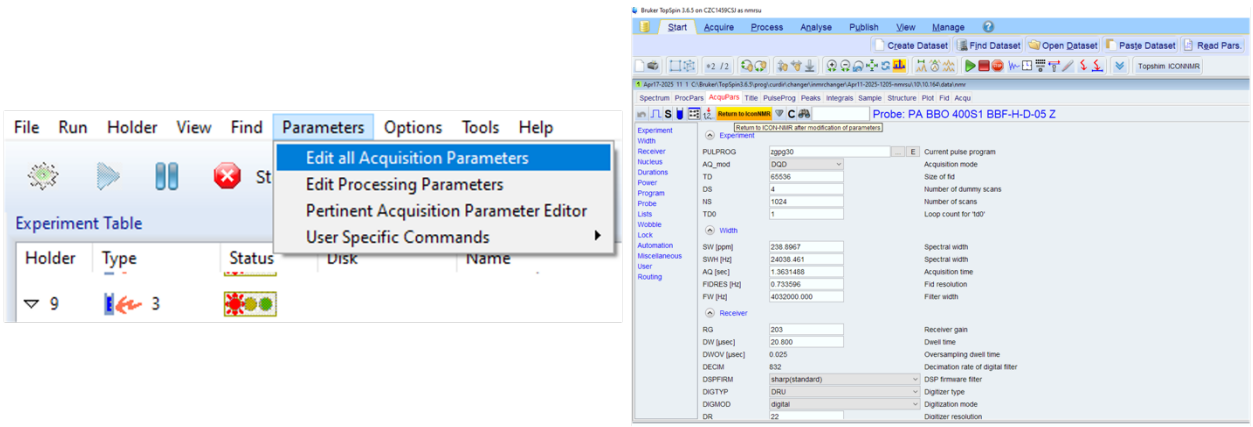


Check/Edit Acquisition Parameters

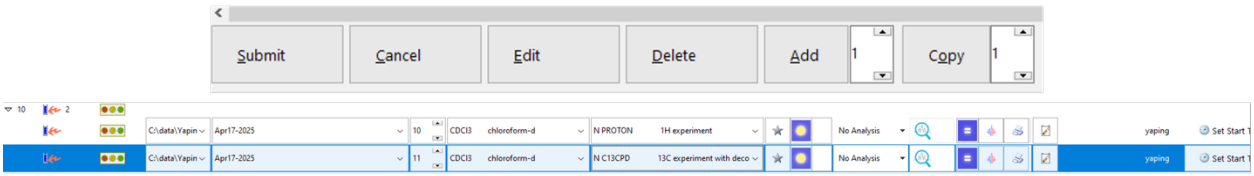
1. If desired, hit the equals button to edit standard acquisition parameters including number of scans (NS), spectral width (SW), Acquisition time (AQ), offset frequency (O1P), etc.



Note: The default acquisition parameters are good, but feel free to change them as desired. For example, if your sample concentration is too low, you could increase the number of scans. If you would like to change any other parameters that are not accessible in this list, you can also click Parameters – Edit all, which will bring you to Topspin. You may need this for 2D experiments set up.

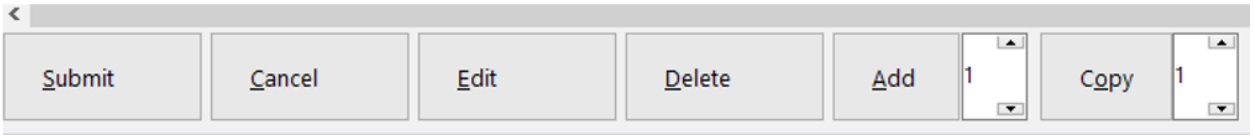


2. If using the nighttime queue (5 pm - 8 am), you may edit the start time if desired.
3. If you would like to collect another experiment on this sample (e.g., C13CPD), you can highlight the current experiment and then hit the Add button. Another experiment will appear below with a new experiment number and an empty experiment type. Select the appropriate experiment and edit acquisition parameters if necessary.



Submit your experiment

1. When you are satisfied with all experiments set up and are ready to collect your data, click the submit button to start your data acquisition.



2. When finished, select the Change User button to log out, allowing other users to place samples for automation.

Experiment

Common 1D experiments

PROTON: standard 16 scan ^{1}H spectrum with 20 ppm spectral window.

C13CDP: standard ^{13}C spectrum with ^{1}H decoupling. The number of scans (NS) can be changed according to your sample concentration.

C13DEPT45, 90, 135: DEPT experiments

Common 2D experiments

Recommended routine COSY, TOCSY, NOESY, ROESY, HSQC and HMBC

Homonuclear 2D experiments

^{1}H - ^{1}H COSY (Correlation Spectroscopy): identify spins that are coupled to each other

COSYGPSW: gradient COSY; can do single scan, default 1 x 128, 5min

COSYGPDFPHSW: phase sensitive 2D homonuclear shift correlation experiment with double quantum filter to remove uncoupled singlets. Default 4 x 256, 39 min

^{1}H - ^{1}H TOCSY (Total Correlation Spectroscopy): displays correlations between all protons within a given spin system

MLEVPHSW: default 8 x 256, 1 hour and 22 minutes

^{1}H - ^{1}H NOESY (Nuclear Overhauser Effect Spectroscopy): shows through-space interactions within the molecule

NOESYPHSW: default 4 x 256, 46 minutes

Heteronuclear 2D experiments

For heteronuclear 2D experiment, particularly those involving ^{13}C, experiment time significantly depend on the sample concentration. If a 1D ^{13}C experiment with

good quality takes 320 scans, increase the number of scans (NS) to at least 16 for the 2D experiments, which is about a factor of 20.

HSQC: determine proton-carbon single bond correlation

HSQCEDETGP: can adjust NS according to your sample condition

HMQC: shows heteronuclear correlations that are directly bonded to each other

HMQCBIPH: can adjust NS according to your sample condition

HMBC: detects heteronuclear correlations over longer rangers of about 2-4 bonds