

# IconNMR Operating Procedures

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

1. Insert your sample(s) in the SampleXpress sample changer. Note the sample position number(s) (##), in the cassette.

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 3.2 icon on the desktop.



3. To start Icon NMR, type *icon* at the command line of TopSpin.

4. Click on the [Automation] button in the Icon NMR dialog box that appears.



5. The Icon window and a dialog box will appear and you'll be asked to select your username. Note that another annoying pop-up will appear saying that it can't find the domain username. Just click *OK* to shut that window. If Icon is already open, click on the [Change User] button and select your username.

The main Icon window. Each row corresponds to a sample position in the SampleXpress autosampler.

Click Start to begin an Icon run of all Submitted samples

Click here to login or to logoff from your Icon account. You should always logoff after you Submit your sample.

Copies current experiment. Useful if you are running the same experiment on multiple samples. Change the number to the number of additional samples.

Adds a new experiment to the current sample

Click to Submit a sample to be acquired, after inputting the sample information

Click to Edit an entry.

The actions of these buttons can also be accessed by right-clicking on a sample number.

This window will show the status of the samples acquired during an Icon run.

Clicks a submitted sample entry, but does not delete the entry. Required if you want to edit an already submitted entry.

Click to Edit an entry.

Click to Submit a sample to be acquired, after inputting the sample information

6. Double-click on the sample *Holder* number from the list of available entries to open the sample *Holder* corresponding position of your sample in the sample changer. In the figure below, sample *Holder* 5 is selected.

Directory where data will be stored. Normally you should not have to change this.

Click to choose your experiment. The available experiments depending on the account.

Click here to add a title for your spectrum here, if you choose.

Click here to change basic parameters, like number of scans, etc.

Experiment number defaults to 10. Value will increment for additional experiments added to this sample.

7. Use the pulldown menus or type in the boxes to provide the following information for your experiment (from left to right in the figure above; only the indicated items need be set).
- **Disk:** the data folder where your experiment will be saved. Normally you should not have to change this from the default for your icon account.
  - **Name:** Enter a name for your data file (really a directory)
  - **No.** This is the Experiment Number (EXPNO, in TopSpin). It defaults at 10 and will increment if you add additional experiments to this sample. Each different experiment you add is stored in its own subdirectory (the *No.*) under your main data folder.
  - **Solvent:** Select your sample solvent from the pulldown menu.
  - **Experiment:** Select the desired experiment from the list. The available experiments depend on your icon account (the Chem233/234 accounts have an abbreviated list of experiments).

### Common 1D Experiments

**PROTON:** standard 16 scan  $^1\text{H}$  spectrum with 20 ppm spectral window. You may want a smaller spectral window –see p. 4. There is no sample rotation with this experiment.

**PROTONRO:** standard  $^1\text{H}$  spectrum, as above, except with sample rotation.

**C13CDP:** standard  $^{13}\text{C}$  spectrum with  $^1\text{H}$  decoupling. Default is 1024 scans, which takes ~1 hour. This is likely more scans than you need. The number of scans (*NS*) can be changed; see below.

**P31CDP:** standard  $^{31}\text{P}$  spectrum with  $^1\text{H}$  decoupling.

**F19CDP:** standard  $^{19}\text{F}$  spectrum with  $^1\text{H}$  decoupling.

**C13DEPT45, 90, 135p:** DEPT experiments.

### Common 2D Experiments

**Note:** The “C” in front of an experiment name indicates a “composite” experiment. A composite experiment is one in which a preliminary “scout” experiment is run to automatically determine the correct spectral width for an experiment. If you want to define your own spectral window(s) use a “normal” experiment (name preceded by an “N”), and set the parameters as you choose (see below for details).

### **Recommended routine COSY, NOESY, HSQC and HMBC experiments:**

**C COSYGPSW:** composite gradient COSY using a spectral width set by a  $^1\text{H}$  scout experiment; can do single scan, default 1 x128, 5 min.

**C NOESYPHSW:** phase-sensitive NOESY with spectral width set by the  $^1\text{H}$  scout experiment; use multiples of 2 scans, default 4x256, 44 min.

**C HSQCEDETGPSISP\_ADIA:** gradient HSQC, phase sensitive ( $\text{CH}_2$  opposite phase from CH and  $\text{CH}_3$ ), sensitivity enhanced with adiabatic pulses; can do single scan; default 4x256, 39min.


**HMBCGP:** gradient HMBC with decoupling during acquisition; multiples of 2, default 4x128, 15 min.

### **Other common 2D experiments**

**HSQCEDETG:** gradient HSQC, phase sensitive:  $\text{CH}_2$  opposite phase than CH and  $\text{CH}_3$ ; can do single scan; default 2x256, 14min.

**HMQCGP:** gradient HMQC; can do single scan, default 4x128, 14min.

**HMBCGPND:** gradient HMBC without decoupling; see single-bond CH peaks as doublets; multiples of 2, default 8x128, 32 min.

- **Par:**  Select this button to change a subset of standard parameters, as shown for a  $^{13}\text{C}$  experiment. See below in this document for instructions on how to access all experimental parameters.

NS = number of scans.

SW = spectral width (ppm).



O1P = transmitter offset (ppm), the center of the spectrum.

D1 = relaxation delay between scans.

AQ = acquisition time (s). This should be about 4s for a  $^1\text{H}$  spectrum and about 1-2 sec for a  $^{13}\text{C}$  spectrum.



If any parameters are changed, the *Par* icon will change to an exclamation point:

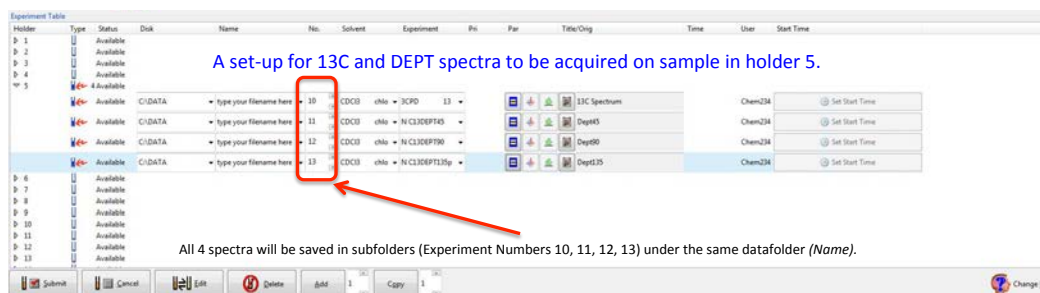
- *Title/Orig*: enter a title for your spectrum, if desired.
- *Set Start Time*: If you want your experiment to start at some later time (like in the evening or night time, for a long experiment, you can set the desired start time by clicking on this button.



8. To add additional experiments to this same sample, click the Add button, or right-click on your sample number and select *Add*.

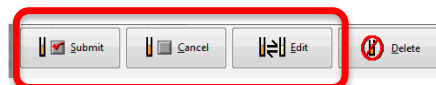


Shown in the figure below is a set-up for a DEPT experiment. The original experiment was a  $^{13}\text{C}$  spectrum (Experiment No. = 10). The [Add] button was invoked 3 times to add the DEPT45 (No. 11), DEPT90 (No. 12), and DEPT135 (No. 13).



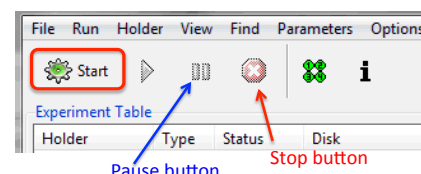
9. When you have finished setting up the desired experiment(s), click the submit button. The status of the experiment will be changed to "Queued".

If you need to edit a selected Queued experiment, click [Cancel] and then [Edit]. After editing, click [Submit], again. These commands can also be invoked via a right-click on the desired sample position.



10. Begin the automation by clicking the [Start] button. If automation is already running, your sample will be run in turn.

Automation can be halted with the [Stop] button, which also stops the currently running experiment. If you don't want to interfere with the current experiment, use the [Pause] button. All experiments that have not been completed remain in the queue and can be run by restarting the automation. Note that only the user who started the Icon automation run (or a superuser) and stop a run in progress.



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



## Advanced Options

### ➤ Giving your sample “Priority”

Except for the Chem233 and Chem234 Icon accounts, all other accounts have the option of giving a sample *priority* in terms of the order of acquiring *queued* samples. To do so, click on the [Priority] button when you set up your experiment.



Giving your sample priority means that it will run after the currently running acquisition is complete. Your sample has priority over samples not designated priority, but it will not move your sample ahead of any other samples which are also designated “priority”.

### ➤ Set up an Icon run of multiple samples with the same experiment, such as a batch of CHEM 233 lab samples.

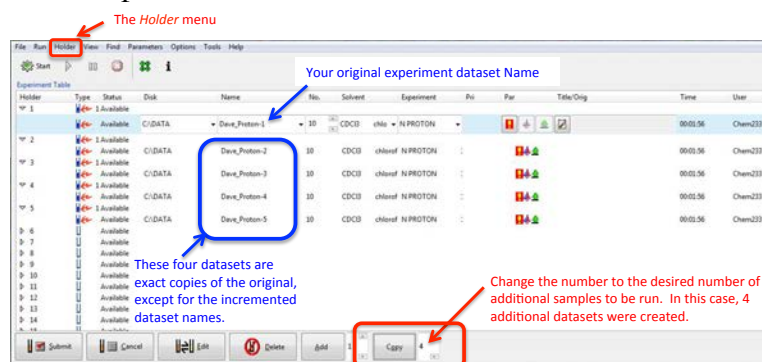
1. Set up the first sample in the batch as outlined above.

Hint: When naming your dataset (directory), end the name with the number “1”. For example, the dataset name could be: Dave\_Proton-1. The file increment feature of Icon will recognize the number at the end of your dataset name and will iterate from this number when it creates the incremented data files. Thus, the second dataset will be named Dave\_Proton-2, the third, Dave\_Proton-3, *etc.* If you don’t end the original filename with a number, then your dataset names will be, for instance: Dave\_Proton (the original file), Dave\_Proton-1, Dave\_Proton-2, *etc.* Likewise, if your original dataset name ended with, say, a “4” (Dave\_Proton-4), then the dataset names would increment from 4; *i.e.* Dave\_Proton-5, Dave\_Proton-6, *etc.*


2. In order for Icon to automatically increment the file names for each sample in the batch, select *Holder > Increment Name*, from the menu at the top of the icon window.

3. Select the sample holder of the first sample, which you have set-up. Next to the [Copy] button, change the number to the number of additional samples in the batch (4, in the example below), then click [Copy]

You can see in this example that four additional sample holders are populated exactly like the original one, except the dataset names are incremented.



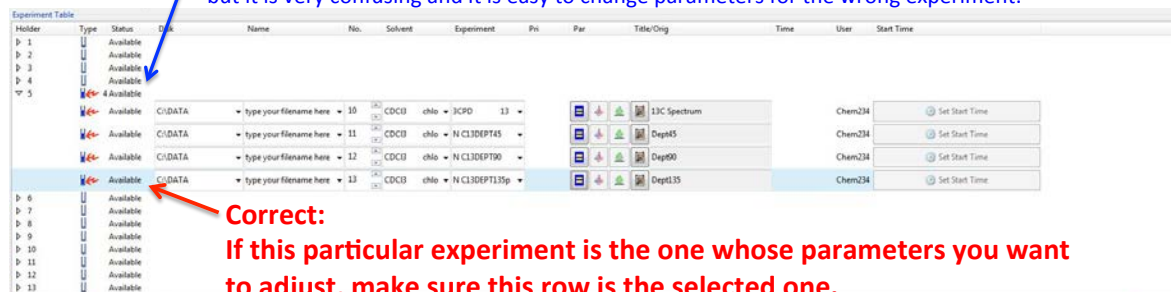
### ➤ Customizing parameters for more advanced experiments.

If you are using Icon to queue up more advanced experiments, you may need to adjust acquisition parameters that are not among the limited set available from the parameters button. 

In this case, you can access acquisition parameters from the *Parameters* menu, at the top of the Icon window. However, if you have multiple experiments set up for a sample, first make sure to select,

explicitly, the row that corresponds to the experiment you are working on. If, instead, the “parent” row is selected, you’ll get the parameters for all of the sub-experiments, which can be toggled through, but will easily cause confusion and cause you to make mistakes.

**Don't highlight this “parent” row:** If the “parent” row is selected, when you view the parameters in TopSpin, you’ll get the parameters for all of the sub-experiments, which can be toggled through, but it is very confusing and it is easy to change parameters for the wrong experiment.

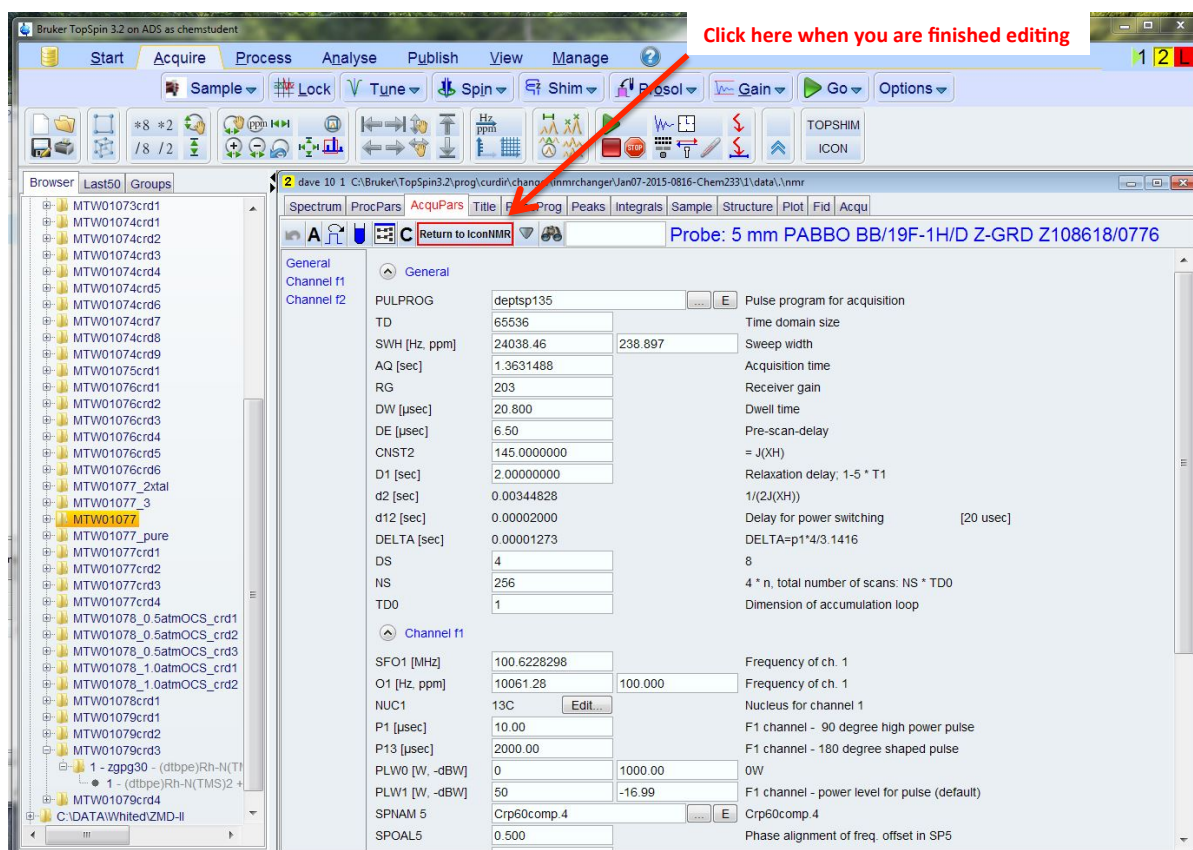


**Correct:**  
If this particular experiment is the one whose parameters you want to adjust, make sure this row is the selected one.

After you select the correct experiment row, go to the *Parameters* menu: select *Parameters > Pertinent Acquisition Parameter Editor*, for a set of the relevant acquisition parameters for your experiment, or *Parameters > Edit all Acquisition Parameters* for access to **all** acquisition parameters.

The TopSpin window will open to the acquisition parameters (see the figure below). When you are done editing parameters, click on the flashing **[Return to IconNMR]** button.

**NOTE: do not change any parameters in this window unless you are sure you know what you are doing. Ask for help if you are unsure.**



**Click here when you are finished editing**

Probe: 5 mm PABBO BB/19F-1H/D Z-GRD Z108618/0776

Parameter	Value	Description
PULPROG	deptsp135	Pulse program for acquisition
TD	65536	Time domain size
SWH [Hz, ppm]	24038.46	Sweep width
AQ [sec]	1.3631488	Acquisition time
RG	203	Receiver gain
DW [µsec]	20.800	Dwell time
DE [µsec]	6.50	Pre-scan-delay
CNST2	145.0000000	= J(XH)
D1 [sec]	2.00000000	Relaxation delay, 1-5 * T1
d2 [sec]	0.00344828	1/(2J(XH))
d12 [sec]	0.00002000	Delay for power switching
DELTA [sec]	0.00001273	DELTA=p1^4/3.1416
DS	4	8
NS	256	4 * n, total number of scans: NS * TD0
TD0	1	Dimension of accumulation loop
SFO1 [MHz]	100.6228298	Frequency of ch. 1
O1 [Hz, ppm]	10061.28	100.000
NUC1	13C	Nucleus for channel 1
P1 [µsec]	10.00	F1 channel - 90 degree high power pulse
P13 [µsec]	2000.00	F1 channel - 180 degree shaped pulse
PLW0 [W, -dBW]	0	0W
PLW1 [W, -dBW]	50	-16.99
SPNAM 5	Crp60comp.4	Crp60comp.4
SPOAL5	0.500	Phase alignment of freq. offset in SP5