

TopSpin 3.2

Routine Operating Procedures

This guide provides basic instructions for acquiring routine NMR spectra using the TopSpin program. Because you are likely to do most of your data processing using MNova, only a few processing procedures are provided in this document.

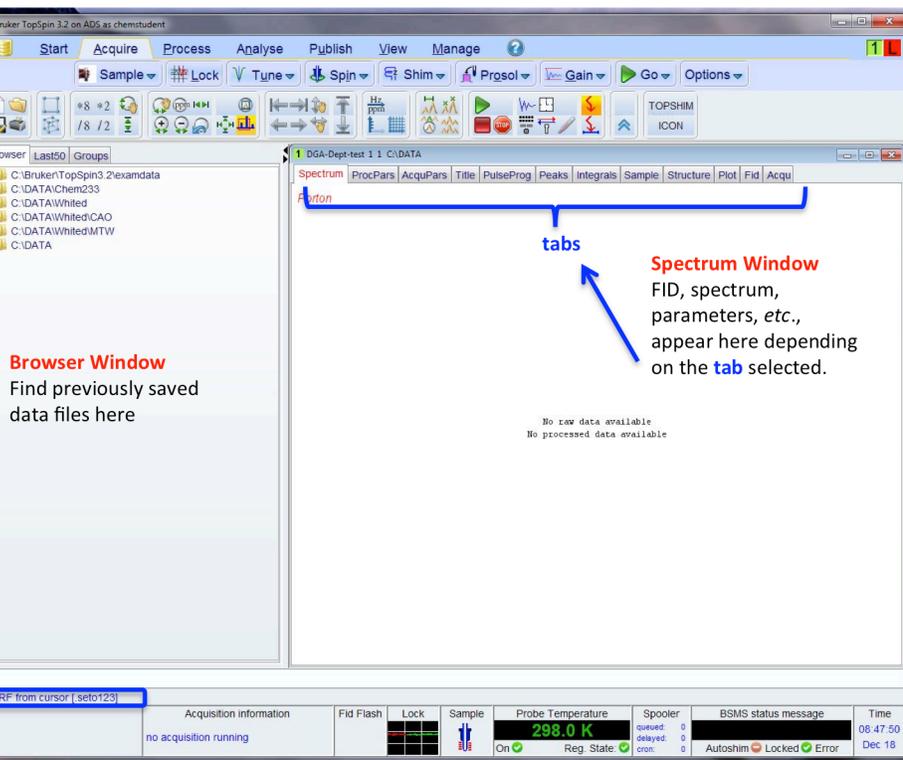
1. Insert your sample in the SampleXpress sample changer. Note the sample position number (##), in the cassette.
2. Generally, you will find the NMR PC running and logged in. If TopSpin is not running, double-click the TopSpin 3.2 icon on the desktop.



The TopSpin main window

Workflow Tabs

These tabs are activated from left to right as you move from experiment set-up through final spectrum printing (although you will probably be processing your spectrum off-line, using Mnova).



Browser Window
Find previously saved data files here

Spectrum Window
FID, spectrum, parameters, etc., appear here depending on the **tab** selected.

Command line

Click here to enter keyboard commands. Commands always require <return> to invoke the command.

Look here for information about the progress or completion of a process.

Lock signal
Double-click for large lock signal window.

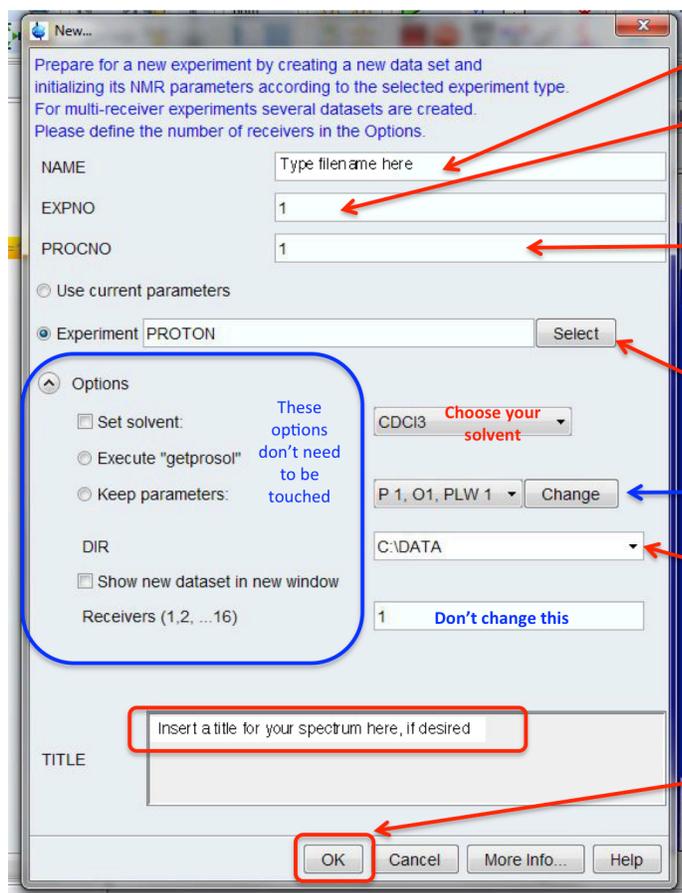
Sample Icon
You will see this icon when a sample is in the magnet.

3. To inset your sample into the magnet, type on the command line: `sx ##`, where ## is the sample position number.
4. **Create a dataset:** the first step in acquiring NMR data is to create a new dataset (actually a directory). It is important to do this right away, otherwise you run the (small) risk of overwriting a currently loaded data set. Click [Start] and then [Create Dataset] to bring up the Dataset dialog box. You can also type *new* in the command line.



The Dataset dialog box

Enter your data file and experiment information as indicated below.



You are actually creating a directory, in which you can save multiple NMR experiments for this sample

For EXPNO (experiment number), start with "1". Later, you can increment EXPNO as you add additional experiments on this sample. For instance, "1" might be a proton spectrum, "2" a carbon, "3" a COSY, etc. Thus, all your experiments will be in the same directory.

PROCNO (process number), start with "1". It is unlikely that you would enter anything but "1", here. The process number allows you to save different processing parameters for the same FID; e.g. perhaps you want to save a spectrum with and without resolution enhancement. If you plan to process your data off-line, using MNova, just enter "1".

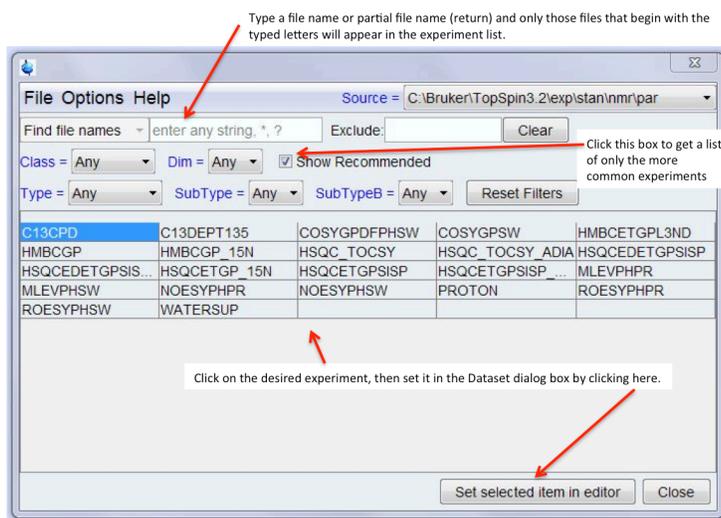
Click [Select] for a pop-up list of NMR experiment parameter sets (see below), or type the name of a parameter set, if you know it.

These acquisition parameters should **not** be changed, unless you know what you are doing.

Select the directory where you want your data to be saved, or type in a path, if you know it.

Click [OK] when you are done setting up your data set.

The Experiment dialog box



Type a file name or partial file name (return) and only those files that begin with the typed letters will appear in the experiment list.

Click this box to get a list of only the more common experiments

Click on the desired experiment, then set it in the Dataset dialog box by clicking here.

Common 1D Experiments

PROTON: standard 16 scan ¹H spectrum spectrum with 20 ppm spectral window. You may want a smaller spectral window – see p. 4.

C13CPD: standard ¹³C spectrum with ¹H 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 later, see below.

P31CPD: standard ³¹P spectrum with ¹H decoupling.

F19CPD: standard ¹⁹F spectrum with ¹H decoupling.

After selecting the experiment, close the Experiment box and then the Dataset box.

5. **Acquire data.** To begin the data acquisition process click on the [Acquire] tab.

Next, click, in order, on the tabs indicated below, allowing each process to complete before moving on to the next tab. The progress of each operation is displayed below the command line in the lower left of the TopSpin window.

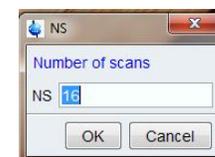
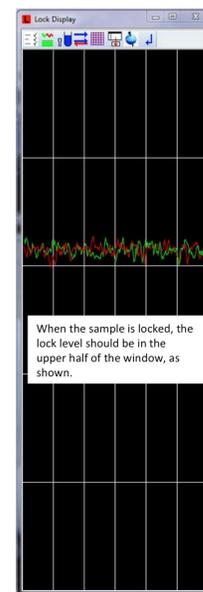


- [Lock] Left click, then select your sample solvent from the dialog box that pops up. The program will automatically lock on the solvent and the lock signal will stabilize with the level in the upper half of the Lock Display window. If desired, click on the small Lock signal display at the bottom of the main TopSpin window to invoke the large Lock window, shown here.
- [Tune] Left click and wait for the autotune to complete. It may take several minutes.
- [Spin] To turn on or off the sample rotation, **right** click on the [Spin] tab and select rotation on or off, as desired.
- [Shim] Left click to begin the automatic shimming. It may take several minutes, be patient.
- [Prosol] Left click to load the correct probe parameters for the experiment to be acquired.
- [Gain] Left click to automatically adjust the receiver gain for your sample. You can also type *rga*, to invoke the automated gain adjustment.

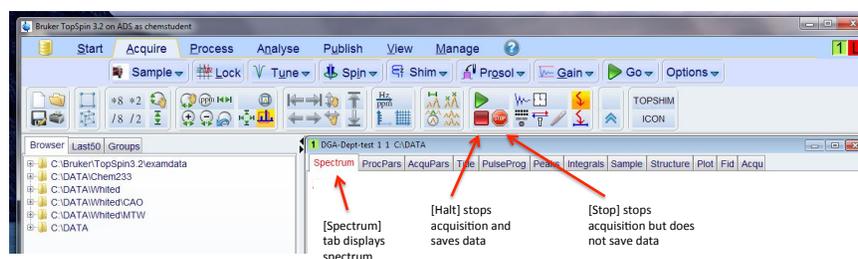
Before starting your experiment, you may want to change the number of scans, especially if you are running a ^{13}C NMR acquisition. To do so, type *ns* <return> at the command line. A dialog box will appear showing the current NS value. Enter the desired number of scans and click OK. You can also directly enter the number of scans at the command line; type: *ns ##*, where ## is the number of scans.

- [Go] Clicking this tab starts the acquisition. Alternatively, the command *zg* does the same thing.

The Lock Display



If for some reason you need to stop an acquisition before completion, use either the [Halt] button, which stops the acquisition after the current scan and saves the data, or the [Stop] button, which halts the acquisition *without* saving the data.



Halt



Stop

Basic Processing (only very basic processing is described here):

1. **Fourier transformation:** Click on the [Spectrum] tab in the spectrum window, if not already there. You'll see the raw FID. You may wish to apply a window function to the data, like line broadening, controlled by the parameter *lb*. For ^1H spectra, the default *lb* = 0.3 Hz; for ^{13}C and ^{31}P spectra *lb* = 1 Hz. For ^1H spectra applying line broadening is not critical, but it is strongly recommended for ^{13}C

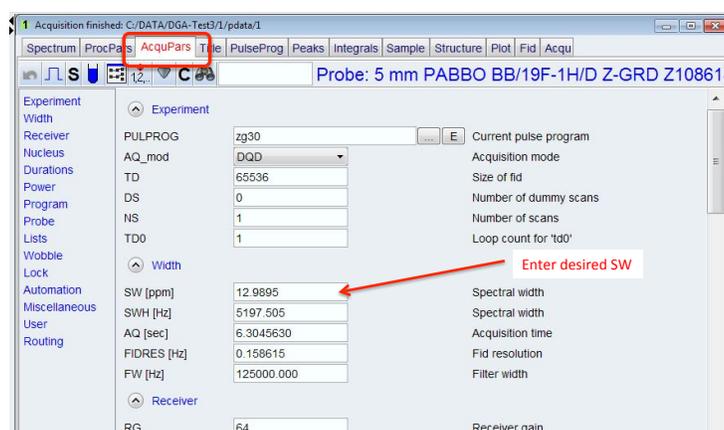
and ^{31}P spectra. Type *em* (exponential multiplication) to apply *lb*, then type *ft* to transform the data. Alternatively, typing *ef* does both.

2. **Phasing:** Type *apk* to apply the automatic phase correction.
3. **Expand the spectrum:** To expand the spectrum, click and drag the cursor over the area of interest.
4. **Changing spectral width:** There are a number of ways to change the acquisition spectral width. The relevant parameters are SW and O1 (the transmitter offset = the center of SW).

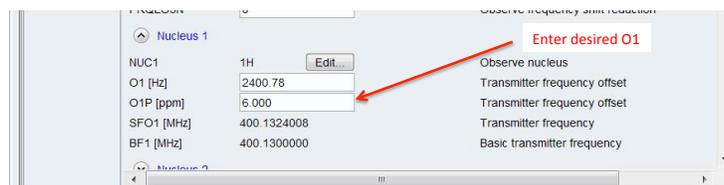
- Type *sw* (or *o1*) <return>. Enter the new value in the dialog box. Make sure to hit <return> after typing the new value.
- To interactively change SW based on the spectrum itself, simply expand the spectrum to the desired width, then click on the button shown to the right, which will use the displayed spectrum region to set O1 and SW. 
- You can also enter these parameters directly. In the Spectrum window, click on the [AcquPars] tab. Enter the desired SW and O1. You'll have to scroll down to find O1. The spectral width straddles O1. Thus, if SW = 10ppm and O1 = 4ppm, the newly acquired spectrum will run from 9 to -1 ppm.

Key parameters that can be changed in this window are:

- NS = number of scans
- DS = dummy scans – number of scans run before data is saved. Brings system to steady state before data is collected.
- TD = size of the FID – number of data points that constitute the spectrum.
- AQ = acquisition time. Note that SW, TD and SW are all dependent on one another. You may want to check AQ after changing SW. If AQ is much longer or shorter than about 4-5 sec, set it to 4-5 sec.
- D1 = relaxation delay between scans.



You'll have to scroll down to find O1



Once SW and O1 are set, reacquire the spectrum by typing *zg*, or click the Go button. You will be asked if you want to overwrite the original spectrum. If you do **not** want to overwrite the previous spectrum, the command *ixpno* increments the experiment number (EXPNO). Type *zg* to acquire new spectrum.

Other useful command:

tr will transfer data to disk during an acquisition so that you can look at the spectrum. After *tr*, type *ef*, then *apk* to process and phase the spectrum.

tr 8 will transfer data after 8 scans.