Checklist for recording NMR spectra

Insert sample tube into spinner

For ALL UNC SOM Biomolecular NMR spectrometers, the bottom of the NMR tube should be 21mm below the center of the coils. Using the depth gauge, the bottom of the gauge is at 21mm – gently slide the tube through the spinner until the tube reaches the bottom of the gauge. If you have a small sample volume, you may have to raise the sample tube to center the solution around the detection coils shown on the gauge.

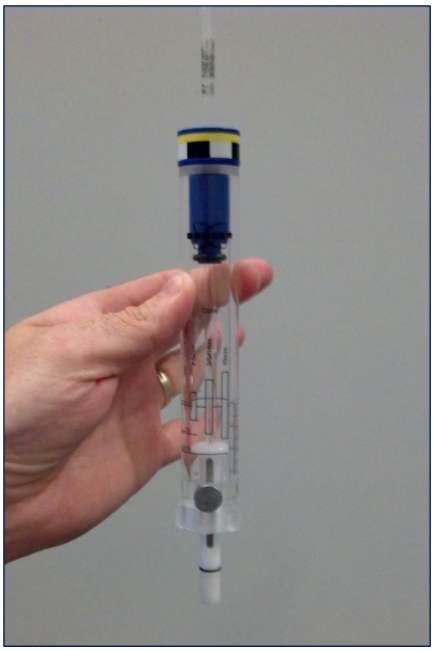


Figure 1. NMR sample and depth gauge.

Place sample tube and spinner in magnet

Turn on the air flow using the grey button labeled “Lift on/off” at the top left of the shim keypad or by typing “ej” in TopSpin. Place the sample tube and the spinner in the bore so that it floats on the air. Then press the “lift on/off” button again or type “ij” in TopSpin. You should see a ‘Sample down’ indicator when the sample seats itself properly.

Set temperature

In TopSpin enter the command “edte” or double click on the temperature shown in the display panel. This will start a new internal window allowing you to set the temperature and air flow and monitor and record the temperature. For all spectrometers, the air flow should be 640 l/h at 298K when using a white spinner but may be reduced to 535 l/h if using a blue spinner.

Lock

In TopSpin enter the command “lock” and select the solvent you are using.

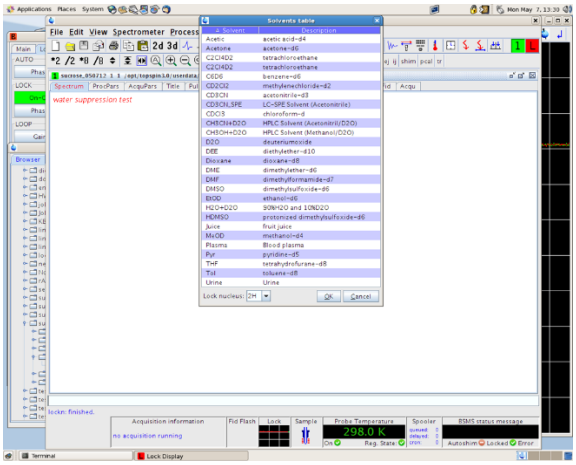


Figure 2. Solvent lock screen.

Create a new dataset

In TopSpin enter the command “new” to create a new dataset. Choose whether to read in Bruker standard parameters or to copy the parameters from the current dataset. Many standard experiments have been set up and tested. These can be used as the basis for new experiments.

Tune and match the probe

All Biomolecular NMR Core spectrometers have cryoprobes fitted with an automatic tune and match unit, you can enter the command “atmm” to manually shim the probe and adjust the tune and match at the console. This method is more accurate and will produce better results. Alternatively, you can enter the command ‘atma’ and allow Topspin to tune the probe for you. Generally, this will produce a tune profile that is ‘acceptable’ while the command ‘atma exact’ will produce a more precise tune of the probe.

***Note: You must tune the probe for each nucleus that you intend to use! ***

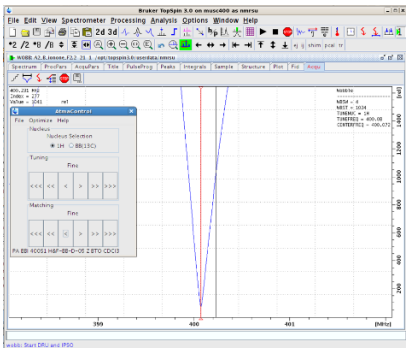


Figure 3. Manual tune of probe using ‘atmm’

Read shim file

In TopSpin enter the command “rsh” and select a file corresponding to your probe and sample conditions. The shim fields are remapped every 3 months, for the latest 3D H_2O water suppression shim file order the files by date and look for CPTCI_h2o3Dshim_<date>.sp.

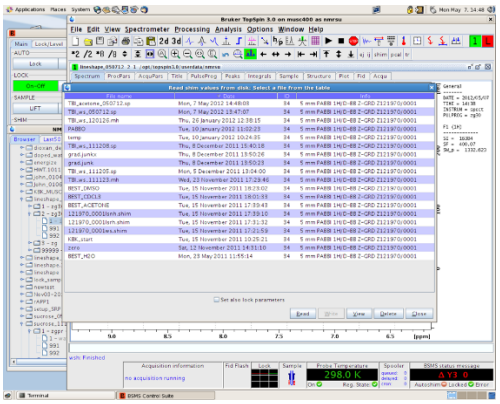


Figure 4. Read shim file screen.

Shim

In TopSpin enter the command “tsg”, select 1D or 3D shimming and click “Start”. 1D shimming takes between one and five minutes and is probably enough for acquiring 1D and 2D spectra. 3D shimming takes between ten and twenty minutes and should be done before acquiring multidimensional spectra. 3D shimming can only be done on samples dissolved in H2O.

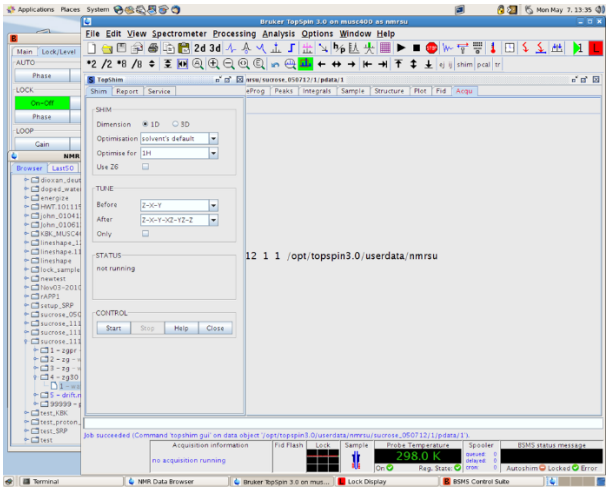


Figure 5. Bruker topshim screen.

Measure 1H 90^{o} pulse

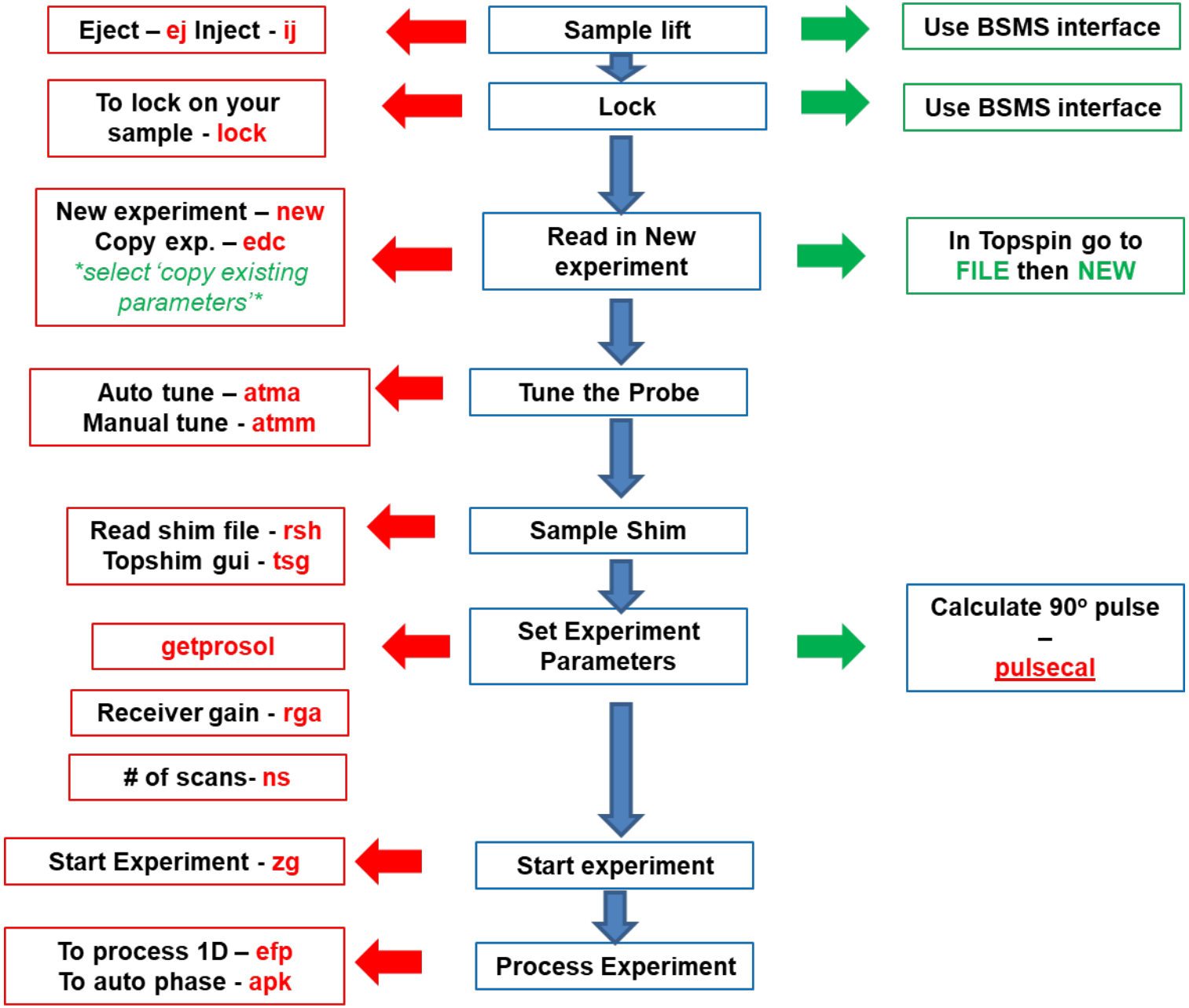
In TopSpin enter the command “pulsecal”. After a short delay a small window will popup containing the pulse lengths and power levels for a 1H 90^{o} pulse at a reduced power level and at a high-power level. The values at the high-power level will be automatically entered into your current parameter set.

Optimise parameters

In TopSpin enter the command “gs”. The display will switch to show a panel which allows you to adjust parameters on the left and a panel showing the FID or transformed spectrum on the right. Parameters such as delay lengths (D0,D1,...), transmitter offset (O1), receiver gain (RG) and power levels for presaturation (PL9) and shaped pulses (SP1,SP11,...) can be modified while the spectrometer is pulsing. This enables their effects on the data to be monitored. After all parameters are set, RG can be optimized automatically by entering the command “rga”.

Start acquisition

In TopSpin enter the command “zg”.


Eject – ej Inject - ij
Sample lift
Use BSMS interface
To lock on your sample - lock
Lock
Use BSMS interface
New experiment – new
Copy exp. – edc
select 'copy existing parameters'
Read in New experiment
In Topspin go to FILE then NEW
Auto tune – atma
Manual tune - atmm
Tune the Probe
Read shim file - rsh
Topshim gui - tsg
Sample Shim
getprosol
Receiver gain - rga
# of scans- ns
Set Experiment Parameters
Calculate 90° pulse – pulsecal
Start Experiment - zg
Start experiment
To process 1D – efp
To auto phase - apk
Process Experiment