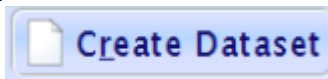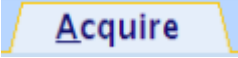# Bruker Speedy Setup Guide

Written by Monika Ivancic (1-27-2015), updated (8-25-2017, 9-3-2019 and 2-6-2020)

With releasing TopSpin software, Bruker has made it very simple to set up and acquire NMR experiments. Every command is 'click-able' and the steps you take are in 'flow mode', meaning that you start on the left and work your way to the right. Keep in mind that for every step described below, there are at least 2 other ways of achieving the same thing. In parentheses and bold letters are the command line entries (type those into the command line at bottom) that replace the click-able command.

1. Start with entering the date, time and your initials in the <u>log book</u> located to the right of the keyboard. Note the solvent you are using and the types of experiments that you will acquire. Also note if anything unusual happens during your session.

2. Log in with your NetID and password and in TopSpin under the 'start' tab ![Start], click on the 'Create Dataset' icon ![Create Dataset]. This brings up a GUI in which you will enter a sample name –NAME (no special chars!), and experiment no. EXPNO (1 is good to start with) and a process no. PROCNO (1 is good here too). Under experiment choose the one you'd like (**uvm.1H1D**). Expand the 'options' tab, set your solvent and select the 'Execute getprosol' button. The data will go into a shared data drive, but will be in your folder. Under DIR *make sure* the directory is: /opt/topspin3.6.1/data/*username*. Then click OK. (you have now successfully created a new dataset in which your current acquisition will be stored)

3. Switch to the 'Acquire' tab at the top ![Acquire]. You will now start with the left most icon and work your way to the right.
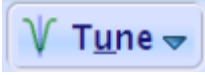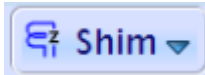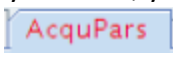
4. Click on the arrow next to 'sample' ![Sample] and turn the lift air on (**ej**). You should hear the air coming thru the bore of the magnet and the yellow dummy sample will come up through the center of the bore. Retrieve the sample and take it out of the blue spinner.

5. Wipe down your sample, then place it into the blue spinner sitting on top of the depth gauge. Holding the depth gauge/spinner with one hand, use your other hand to slide the sample into the blue spinner. Handle the sample by the top of the tube, and wipe down any residual solvent or fingerprints on the outside of the tube with a Kimwipe. Place your sample into the magnet bore (center of the red part protruding above

magnet) and it should stay floating there. The sample status at bottom will display

for an empty magnet.

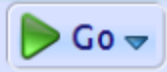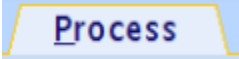6. Click on the arrow next to 'sample' **Sample** and turn the lift air off (**ij**). The sample will be sucked into the magnet and you should hear a click when it settles into the probe. The sample status on the bottom will look like when inserted.

7. Click on the lock icon **Lock** , to lock onto your deuterated solvent (**lock**). The solvent list will pop up, pick the correct solvent and click 'OK'. Wait for the message under the command line to say that lock has been achieved.

8. Click on the Tune icon **Tune** , but not on the arrow of the icon (**atma**). The spectrometer will tune and match to the frequency that you want to acquire.

9. Click on the arrow of the Spin icon **Spin** to turn spinning of the sample on (**ro on**). Once spinning is at 20Hz, the Sample status display will look like:

10. Type **rsh** in the command line, and pick 'current1' from the list of stored shims (**rsh current1**). Then click on the Shim icon **Shim** (not on the arrow). Wait several seconds or a minute or two until the message under the command line says 'Topshim Complete'.

11. Click on the Prosol icon **Prosol** (**getprosol**). This reads pulse widths and power level values from the probe file for the current experiment.

12. Click on the Gain icon **Gain** (not the arrow) or type **rga**. This optimizes the receiver gain value for the current sample and is concentration dependent.

13. Prior to acquiring your data, you may look at your acquisition parameters by clicking on the AcquPars tab **AcquPars** in the lower menu (**eda**). To view parameters for this experiment only, click on in the AcquPars submenu (**ased**). Here you may change certain parameters, such as NS, D1, SW, O1, etc. If you decide to change one of these, make sure you know what this does to your data. DO NOT change power levels or pulse widths please! (see Monika if you think you need to change these)
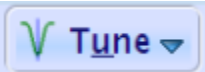
14. Acquire your data by clicking on the Go icon (**zg**). Acquisition information will be displayed at the bottom and likely the display will switch into the Acqu tab in which you see the live FID as it acquires.

15. Once the data is complete, type **efp** to quickly process your data. Follow this with an **apk** for automatic phasing. You may go to the Process tab for further processing and analyzing your data in TopSpin. It is suggested that you process the data in MestreNova, since it is simpler to use. There is a computer in W103 for this purpose.

16. If at this point you need more data on this sample, you will need to create another dataset, otherwise proceed to step 23. to finish. To create another data set, either type '**new**' or under the Start tab, click . This brings up the familiar GUI that you saw in step 3. Since you are acquiring data on the same sample, it is sufficient to just increase the experiment number. Alternatively you could enter a new name. Under experiment choose the one you'd like (**uvm.13C1D**), for a carbon spectrum, or another parameter set if you wish to acquire data on a different nucleus.
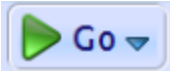
17. Since you are acquiring more data on the same sample, you DO NOT need to lock or shim again, but you DO need to tune and match for increased sensitivity on the X nucleus. Under the Acquire tab, click on the Tune icon (**atma**). You will first see the tuning dip for the X nucleus, followed by the tuning dip for 1H. Wait for the message in the bottom left corner to say 'Job Succeeded' before proceeding.

18. Click on the Prosol icon (**getprosol**), if you didn't choose this in 16.

19. If you are acquiring data on an insensitive X-nucleus, such as ^{13}C, ^{29}Si, ^{11}B, ^{15}N, then you DO NOT need to optimize receiver gain (RG). In this case, the RG value stays at the maximum of 203. IF you are acquiring data on a more sensitive X-nucleus, such as ^{19}F or ^{31}P, then do optimize receiver gain by clicking on (**rga**). In either case, optimizing receiver gain is OK, though it just takes extra time and ends up at the max for insensitive nuclei.

20. Start acquiring your data with (**zg**). Many of the X-nucleus data sets are set up to acquire A LOT of scans. For **uvm.13C1D** the number of scans, NS = 1024, takes 54 min. Likely you do not need that many scans, though you need to check your data as it is acquiring. To do this, type '**tr**' (to "transfer" data), followed by '**efp**'. While you do this, your acquisition is still in progress. If you are not happy with the signal-to-noise on your data, wait a few minutes, then re-check it with '**tr**' and '**efp**'.

21. To stop your acquisition, either type '**halt**' or click on ▮ . DO NOT use the 🛑 icon (**kill**), since it will not store your data (only stores the data until the last tr).

22. You may process your data with '**efp**' and then '**apk**' as in 15. or use MNova.

23. To get ready to take your sample out of the spectrometer, be kind to the next user and turn off spin (**ro off**) under ⚓ Spin ▾ .
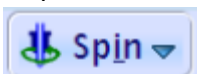
24. Click on the Sample icon 🔬 Sample ▾ to turn the lift air on (**ej**) & walk over to the magnet to get your sample. <mark>Replace your sample with the dummy sample in the blue spinner and place it in the air stream above the magnet then turn the lift air back off (**ij**).</mark>

25. Exit TopSpin by clicking on the gold stack 🗄 and click on Terminate Application. The following GUI will appear, click OK to exit the program.

> Close Application.
> This will terminate all possibly active commands.
> Exit anyway?
>
> OK   Cancel
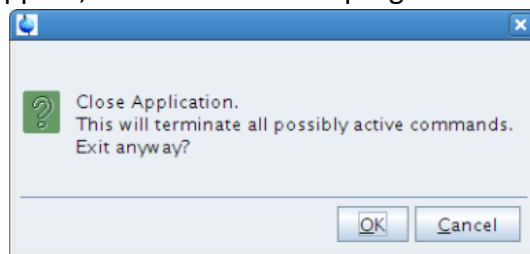
26. Last but not least, log out of the Linux account by clicking on the power symbol icon in the upper R corner, scrolling to your name, then clicking on 'Log out'.

27. You may FTP into the Bruker computer (IP address <mark>132.198.10.253</mark>) using WinSCP software and login using your username and password to get to your data (ie. transfer your data to your local computer). You will need to navigate to: <mark>/opt/topspin3.6.1/data/*username*</mark> on the Bruker computer to retrieve the data.

Some basic troubleshooting

A. If the TopSpin program is stuck or refuses to do what you are requesting, you may try typing '**ii**' in the command line (ii stands for initialize interface). This resets functions that are occurring between the computer and the console. Wait until the message below the command line says that it is done.

B. If the above doesn't complete, then exit the TopSpin software (see 18. above – you may leave your sample in the spectrometer). Re-open the software and create a new 1H 1D dataset (see 3. above). Then in the command line type '**ii restart**'. Wait until it completes and repeat (do it twice). If this doesn't fix your problem, find Monika.