

Bruker run manual for the 500 MHz NMR

12/02/2014

Bold text is used for commands to be entered on the command line and which must be confirmed by the enter button (↵).

Underlined text is for buttons / tabs that can be clicked on.

Expressions in **[SQUARE BRACKETS]** and in bold capitals represent keys on the BSMS keyboard (the BSMS keyboard is the grey rectangular pad with buttons, a control wheel, sitting next to the computer)

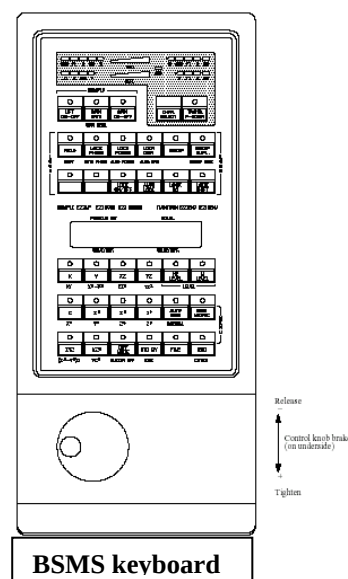
1. **To start:**

2. Log in (username, password).

3. Double-click on the Topspin icon  to start the software.

4. Load the sample:

- Turn off (on=the **green** lamp don't shine) **[LOCK]** and **[SPIN]** before changing samples.
- Press the **[Lift On/Off]** button on the BSMS unit to eject the blank sample. To confirm the sample status, check the LED indicators on the BSMS keypad, either *sample up*, *sample missing*, or *sample down* will be illuminated.
- Clean you're NMR-tube (Kleenex and ethanol).
- Insert your sample into the spinner. The spinner easily picks up dirt and oils from your hands, which end up inside the NMR, so be careful and if you need to touch the spinner do it on the stripped part.
- Use the sample depth gauge to position your sample in the spinner properly. For normal height samples the bottom of the NMR tube will go to the bottom of the depth gauge, and for shorter samples the sample must be centred on the middle line of the depth gauge. This will make shimming on small volume samples easier.
- Position your sample at top of magnet bore, supported by air flow ***Do not release your sample unless you are sure that the air flow will support it and it will not drop into the magnet.***
- Press the **[Lift On/Off]** button on the BSMS unit turns off eject air and the sample will be inserted. When the sample is fully loaded into the probe, *sample down* will be illuminated on the keypad.



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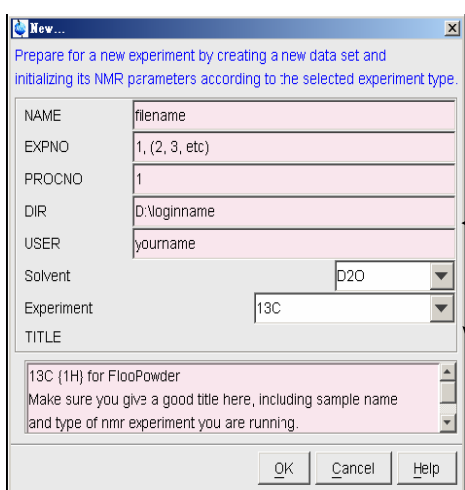
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5. Lock the sample:

- **lock ↵** Select the solvent name from the result menu. Wait until it has found the lock.
- If necessary to adjust *Lock Power* and *Lock Gain* turn on the lock display window click on  or **lockdisp ↵** to get the lockdisplay window. Press **[lockgain]** or **[lockpower]** on the BSMS and adjust the value with the wheel.
- If you attend to run more than one sample with the same solvent can you click on **[LOCK]** when the sample is down.

*If you have difficulty locking, a common problem is that the shim set values are way off. Chose and reload another shim set **rsh ↵**.*

6. Create a new data set. **new** or **edc** or click on and fill in the form.



Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type.

NAME	filename
EXPNO	1, (2, 3, etc)
PROCNO	1
DIR	D:\loginname
USER	yourname
Solvent	D2O
Experiment	13C
TITLE	13C {1H} for FlooPowder Make sure you give a good title here, including sample name and type of nmr experiment you are running.

OK Cancel Help

DIR /opt/home/user

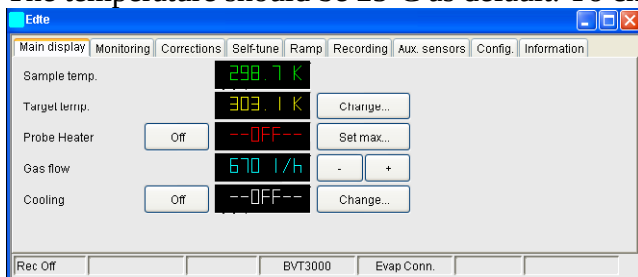
Experiment Dirs. (pathway to parametersets)
/opt/topspin21/exp/stan/nmr/par/user (selfmade)
/opt/topspin21/exp/stan/nmr/par (Brukers)

Experiment
Use current parameters, A-¹H, etc.
or
Brukers own parametersets depending on in which Experiment Dirs. you standing in

“use current parameters” uses the parameters from the dataset you started new/edc from, if Experiment Dirs. are /opt/topspin21/exp/stan/nmr/par/user.

If you change Experiment Dirs level confirm by press enter (↵)

7. The temperature should be 25°C as default. To change temperature write **edte**



Sample temp. 298.1 K

Target temp. 303.1 K Change...

Probe Heater Off --OFF-- Set max...

Gas flow 670 l/h - +

Cooling Off --OFF-- Change...

Rec Off BVT3000 Evap Conn.

and adjust the temperature.

- 8. **atma ↵** (tune and match automatically for the nuclei/nucleus involved in the actual data set) **OBS! Needs to be done on every new nucleus!** Wait until it's finished- atma finished or atma completed.
- 9. Before you shim with “topshim” turn on the spin **[SPIN]**. The spinner should be 20 Hz as default (5mm tubes). The indicator will shine steadily **green** when the sample is spinning.

10. Shim:

➤ **topshim** ↵

This programme shim on the z-shims.

To see how good the shimming are, perform a short ^1H run (go to 11) and check the half height broadband width (the resolution) and the shape of the solvent top.

➤ ***Don't shim more than you need for your purpose!!***

- If you think the peak is too broad or misshaped, try to begin from another shim set (save the shim set before you change) and run topshim again.

wsh (save actual shim set to a list),

rsh (click on the shim set you wish to use)

➤ or shim manually

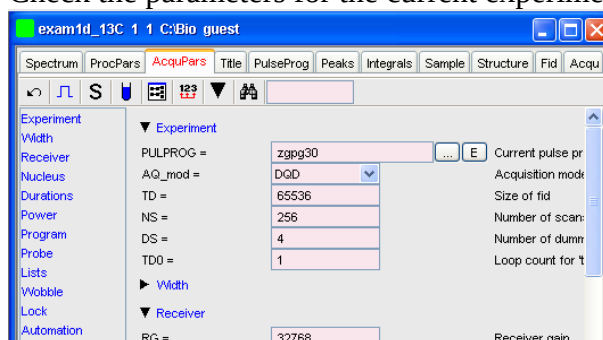
- **[ONAXIS]** (to shim on z shims) For most solvents it is advisable to ensure that **[FINE]** is selected
- **[z]** Turn knob and observe the height of the lock signal on the screen. Adjust for maximum height. **[z2]** Turn knob until maximum signal height is achieved.
- To go back to the value you began the round with, press the actual shim (e.g. **[z]**)
- To save a value press on another shim knob
- Re-adjust **[z]** and **[z2]** in turn until no further improvement is observed.
- **[z3]** Adjust knob for maximum lock signal height.
- Cycle through adjusting **[z3]**, **[z2]** and **[z]** in turn until no further improvement is achieved.
- If the lock signal goes off-scale, reduce [lockgain] to bring the lock level to one of the horizontal lines in the lock display.
- Off axis, press **[SPIN ON-OFF]** to stop spinning
- Adjust X, XZ and Y, YZ iteratively until the maximum lock level is found
- X: First press **[X]**, then **[Z0]**, and then adjust the shim value
- XZ: First press **[X]**, then **[Z1]**, and then adjust the shim value
- Y: First press **[Y]**, then **[Z0]**, and then adjust the shim value
- Y: First press **[Y]**, then **[Z1]**, and then adjust the shim value
- Optimize Z and Z2 as described above, but this time without sample spinning.
- It can be wise to save the shims now and then, see above
- **[STD BY]** Stand by mode, knob is inactive.

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11. Start the run:

- a Check the parameters for the current experiment under the AcquProc tab



e.g. ns, sw, o1p, td. To change pulse programme click on  beside PULPROG mark the choose sequence and “set PULPROG” to confirm.

You can find the parameters p1 and d1 if you go to  menu.

You can also type the parameter and the value on the command line e.g. **ns 16 ↵**

- b **getprosol** or click on  (get probe and solvent specific values for the accurate parameter set)
- c **rga** (finds receiver gain) A sample and experiment dependent parameter.
- d **zg** (start the run) or **zgefp** (start the run 1D + line broadening +Fourier transformation + take current phase parameters) or **xaau** (rga + zg) (finds receiver gain + start the run)
- e **efp** (Processing 1D data: line broadening +Fourier transformation + take current phase parameters)
xfb (processing 2D data)
- f **apk** (automatic phasing).
- g If you want to see how well you have shimmed, zoom in on e.g. the solvent peak **pp**, and choose “Calculate width of current peak”. The broad-width value should be less than 1 (CDCl₃, s 7.26 ppm) and rather as low as possible.
- h You can transform already run scans by the command **tr** and process the data with **efp**. If you want an exact number of scans, type e.g. **tr 4**.
- i If you want to stop the acquisition type **halt**, this will save already run scans. **stop** is a harder way to end the run; you will only have already saved data left.

12. You can work with and/or prepare different experiments at the same time as you run.

13. You have manuals in the Topspin software. Under Help you will find Manuals choose “1D and 2D step-by-step – basic”. Go to T1 Experiment → processing and follow the instructions. Or try NMR Guide to find information.

14. You can queue experiments and commands if you under Options have enable automatic spooling.

15. To end a session:

- Turn off [**LOCK**] and [**SPIN**] before changing samples.
- Press the [**Lift On/Off**] button on the BSMS unit to eject the blank sample. To confirm the sample status, check the LED indicators on the BSMS keypad, either *sample up*, *sample missing*, or *sample down* will be illuminated.
- Change back to the reference sample.
- Press the [**Lift On/Off**] button on the BSMS unit turns off eject air and the sample will be inserted. When the sample is fully loaded into the probe, *sample down* will be illuminated on the keypad.
- **Exit Topspin**
- To reach the data from the 500 MHz you either use the icon “copy NMR data” before you log out to place a copy on Nevyn or the SSH-programme to copy over the file to your own computer.
- Logout from your account

OBS! NMR problems should always be reported in the brown book on the booking table!!

16. Summary

This section serves as a short summary of the steps to follow to obtain a basic spectrum and may be useful when you have had some experience on the instrument.

- Log in and insert your sample.
- **lock** (on the sample)
- **new** or **edc**
- Check the acquisition parameters within the AcquPars tab (or eda) if necessary.
- **getprosol**
- **atma** (For every new nuclei)
- turn on the spinner before topshim
- **topshim** or shim manually (on the sample)
- **rga**
- **zg** or **zgefp** (efp for 1D processing)
- **efp** (1D processing) or **xfb** (2D processing)
- **apk** if needed
- Process and plot your data on the spectrometer computer or on your own.
- Turn off the lock, sample spinning (if used) and eject and remove your sample, put in the “reference sample” and then turn off the eject air.
- **Exit TOPSPIN**, when finished.
- Copy your data to the N-server if needed
- Log-off from the PC

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