Dissimilar
This manual is intended to be only a very brief introduction for using the Bruker 400 MHz NMR spectrometer (PS751) at the California State University LA NMR Facility. For complete information about the TOPSPIN software or running experiments, please refer to the documentation that Bruker provides. Most of the information in this manual was taken from different Bruker manuals and modified and edited to fit our needs in this facility.

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Short instructions for the basic operation of the AVANCE II 400 MHz Bruker NMR Using TOPSPIN (PS751)
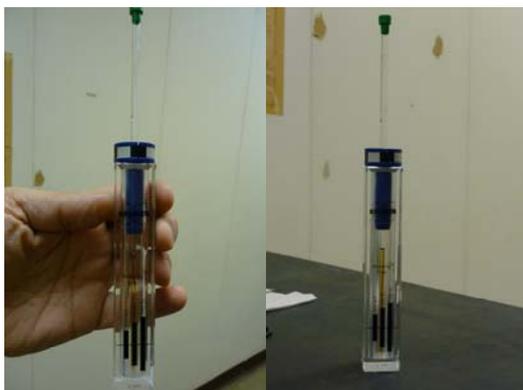
Table of Content

1.<u>Loading The sample</u>:

STEP 1   **Gently place your sample inside the spinner turbine. Use the depth gauge to measure the sample height as it is shown below.**

CAUTION: Do not use excessive pressure to insert your sample inside the spinner turbine

CAUTION: DO not forget to remove your sample from the depth gauge prior to inserting it into the magnet.
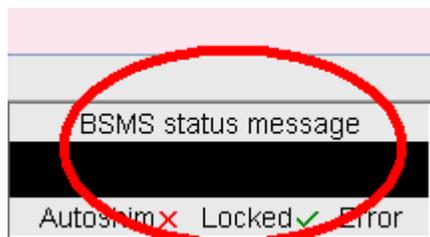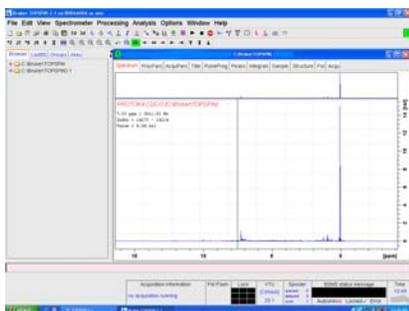
STEP 2   **Log into the account on the spectrometer's workstation.**


STEP 3   **Double click on the TOSPIN icon** **on your desktop.**

STEP 3   **Once the TOPSPIN window appears, click on the "BSMS status message" icon or at the prompt in the bottom left corner of TOPSPIN window type "bsmsdisp".**

STEP 4   Once the BSMS Control Suite window appears click on the **"LIFT"** button to left the air. Now, you should be able to feel (holding your hand over the top of the magnet) and hear the whistling sound of gas coming from the top of the magnet.
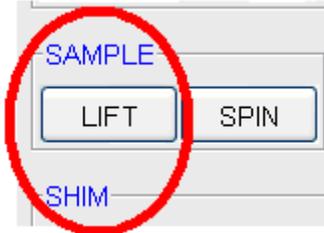
NOTE: If there is no air, stop your experiment and notify the NMR facility staffs.

STEP 5   Gently place the sample on the air cushion on top of the magnet and then push the "LIFT" button again to lower your sample. Sample will descend softly into the NMR magnet. Once the sample settles down inside the magnet, on the BSMS window, you will see the **red** light indicator for "Missing" the sample is off, and the **green** light indicator for the sample being "Down" comes on.
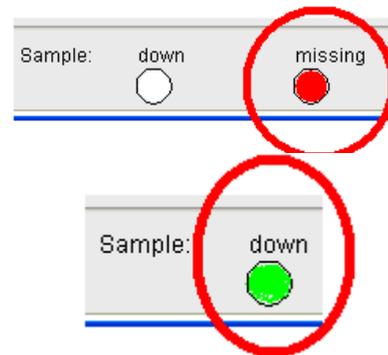
STEP 6   After you see the **green** "sample down" light is on, press the **"Spin"** button. The light will be red untill the sample is at the set speed (20 Hz) and will turn **green.**

4
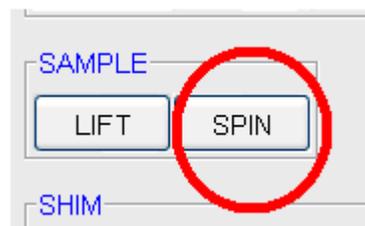
2.Creating a new data set and loading standard parameter:

STEP 1   On the TopSpin toolbar, under file menu select "New" or alternatively type "new" **at the prompt in the bottom left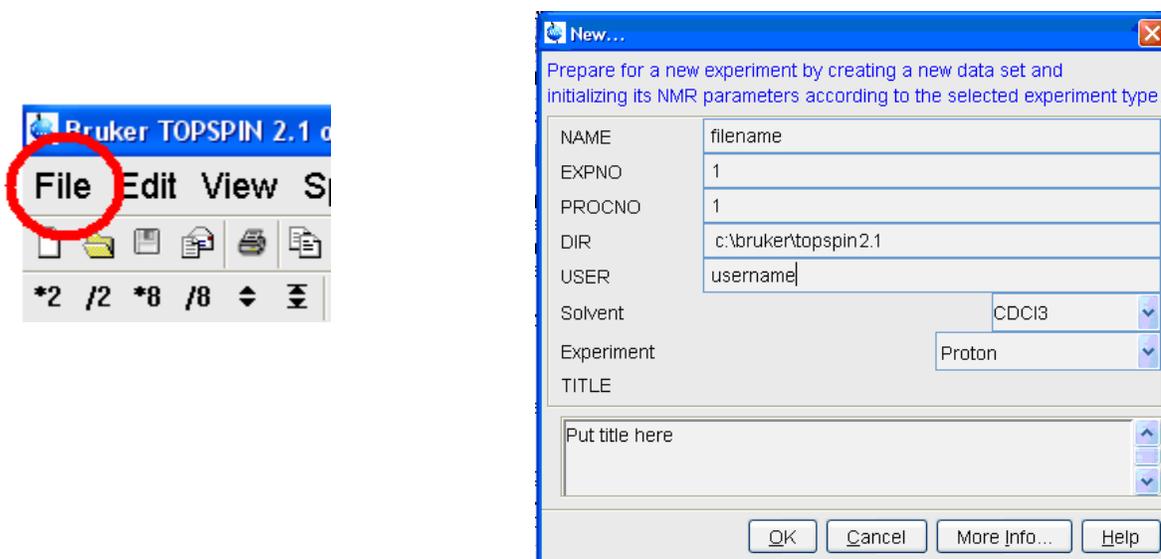 corner of the TOPSPIN window**. Make sure that under "USER" in this menu is your login name and under "DIR" is /opt/topspin2.1. Put the file name, experiment number, processing data number, solvent and title. Once you are done click the "OK" button.

STEP 2   **At the prompt in the bottom left corner of the TOPSPIN window type "rpar PROTON all" to load standard ^{1}H experiment parameters, or type "rpar C13CPD all" to load standard ^{13}C experiment parameters (proton decoupled).**

Alternatively you may create a new data set from the previous parameters belonging to an old file according to the following procedures:
-read the old data set by typing "re *old-filename*"
-with the old data set open, create a new file by typing "wr *new-filename*"
-to proceed you need to read the new data by typing "re *new-filename*"

STEP 3   **Then at the prompt type "getprosol" to load the probe dependent parameters.**

3.Locking, tuning, and shimming:

STEP 1   At the prompt you may type "lock" to invoke the automatic locking procedure in which you need to select the solvent you are using by clicking on the solvent and then the "OK" bottom as it is shown below.
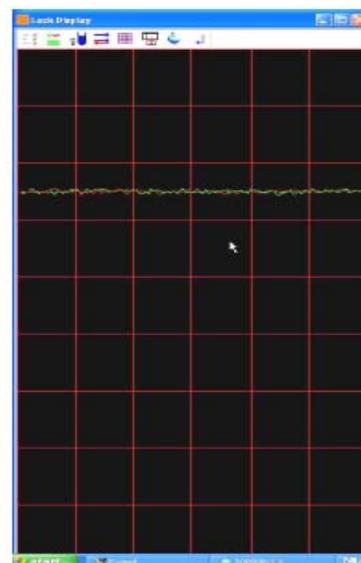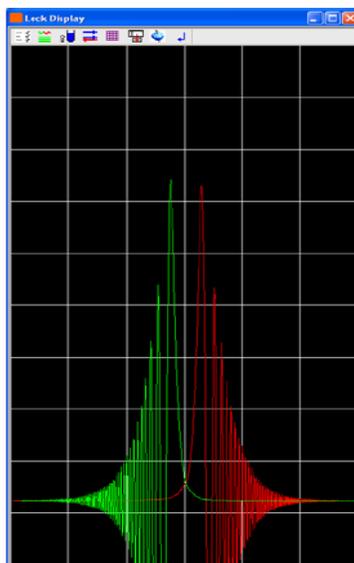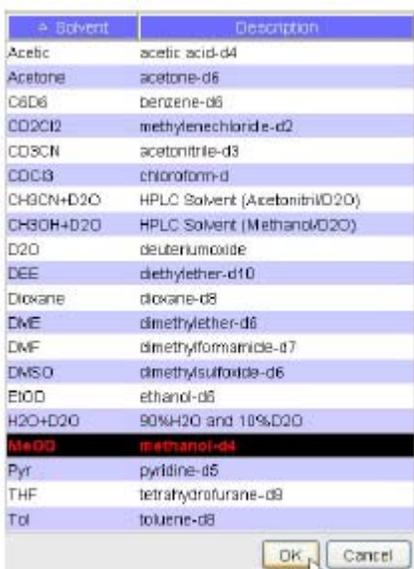
NOT LOCKED   LOCKED

To invoke the lock display (above window) type lockdip at the prompt.

STEP 2   At the prompt type "atma" to invoke the automatic tuning procedure which may takes a few seconds. Wait until the tuning is completed (you will see the "finished" message).

STEP 3   Start the automatic shimming routine by typing "topshim" at the prompt. Wait until the shimming is completed (you see will the "finished" message).

4.Acquisition.

STEP 1 Type "rga" for automatic receiving gain adjustments (wait until it is "finished").

Type "ns" at the prompt to set the number of scans (usually it is multiple of 4).
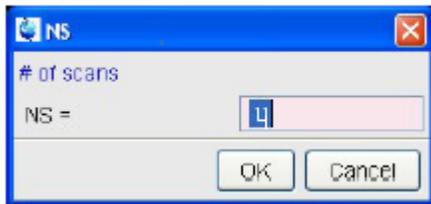
STEP 2 Type "d1" at the prompt to set the delay between pulses (usually 1 to 4 sec).

STEP 1 Type "zg" (Zero Go) to start the acquisition. Wait until the acquisition finishes.

5.<u>Processing:</u>

STEP1   Type "ef" or "efp" to process your data.

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e comes from em = *For exponential multiplication set by* lb *command for line broadening (usually* lb= .5Hz *for proton and* lb= *10 HZ for carbon).*
f comes from ft = *For Fourier transformation of time domain data to frequency domain.*
p comes from pk = *For phase correction using the last applied phase*

STEP2   Type "apk" for automatic phasing.

Manual phase correction: Click ⌁ to phase. Using left button, scroll **0** and

1 to phase. Click save+return ⮐ .

STEP3   To calibrate your spectrum manually [only if it is needed]. Click calibrate ⋀ . Place cursor on peak and click left button.

STEP4   **To integrate your spectrum click integrate ⌐ . Click ⊔ to define beginning and end of the integral; press left the button and drag. Right click under integral to select. Adjust selected integral tails to be parallel with baseline by scrolling bias and then slope ∫b ∫s . To calibrate, right click under integral and select calibrate. Click save+return ⮐ .**

STEP5   **To pick peak your spectrum Click pick peak ⊥ . Make sure a defined region is selected ⊔ . With the left button drag the box to include the top of all peaks to be picked. To add a peak, click ⊥ and select the peak with the cursor and left click. Click save+return ⮐ .**

8

6.Command list.

(All the command (bold words) must be typed at the prompt as they appear)

Loading the sample Commands:

bsmsdisp - starts BSMS windows
ej - lift the air up (eject sample)
ij - lift the air off (insert the sample)

Locking, tunning and shimming Commands:

lockdisp - it generates the lock window.
Lock *solvent-name* – it will lock on the named solvent after several seconds. Picking the wrong solvent may result in the lock failure
topshim – starts automatic shimming
gradshim – invokes gradient shimming windows in which the gradient shimming routine can be started.
atma - automatic tuning and matching
atmm – invokes manual tuning and matching window in which the automatic routine can be started
wobb- invokes tuning and matching window.

Creating a new data set Commands:

new – creates new data set
edc – creates new data set
re filename – read the file
wr filename – write a file

Acquisition Parameters Commands:

eda – it will display acquisition window
ased – display short list of acquisition window
sw – display spectral width
o1p – display transmitter center (center of spectrum)
ns – display number of scans
rg – display receiver gain
rg – automatic receiver gain adjustment
d1 – display relaxation delay
p1 – display pulse width (for simple 1D acquisition)
aq – FID acquisition time
td – display number of points used to define FID
si – display number of points used during FT (can be different from td)
zg – zero and go

Processing Commands:

edp – it will display processing window
em - exponential multiplication on the FID, uses the parameter LB. This improves signal to noise at the expense of resolution.
lb - this controls the degree of broadening added and affects your signal-to-noise. To see its effect, simply change its value and re-Fourier Transform with **ef**.
ft - fouier transform
ef - combines **em** and **ft**
pk - phase correct, applies the last phase correction to the spectrum. Useful when you have phased a primlimary spectrum, (with only a few scans) and wish to apply the same phase correction to the final spectrum.
efp - combines **em, ft**, and **pk.**
abs- automatic baseline correction
apk – automatic phase correction

7.Frequently asked questions:

Q1. How should I prepare my NMR sample?

Q2. Can I run my sample neat (or without deuterated solvent)? or What if I can not use the deuterated solvent due to special circumstances?

Q3. Why don't I see anything coming up the magnet, when I press the lift button?

Q4. Why doesn't my sample spin?

Q5. Why I cannot lock to the deuterium signal after I put my sample into the magnet?

Q6. How do I improve the resolution for my spectrum? The peaks look awful!

Q7. Why are the chemical shifts for the peaks in my spectrum incorrect?

Q1. How should I prepare my NMR sample?

Dissolve your compound in a suitable deuterated solvent. You may have to filter the solution because there should not be any undissolved particles. Place the solution in an NMR tube with a cap. The sample height in the tube should be around 5.0 cm. Wipe and clean the outside of the sample.

Click to go back to the menu

Q2. Can I run my sample neat (or without deuterated solvent)? **or What if I can not use the deuterated solvent due to special circumstances?**

You can still run your sample; however, you need to make sure that the lock is in standby mode and the field sweep is off, and you also need to shim the magnet using the FID instead of using the deuterated lock signal. Additionally, you may need to use the external reference for assigning the chemical shifts.

Click to go back to the menu

Q3. Why don't I see anything coming up the magnet, when I press the lift button?

1-There is no air

2- Air flow is too low.

3- There is no sample in the probe to be ejected by pressing the lift button.

4- In the worst scenario; the sample is broken inside the magnet.

Click to go back to the menu

Q4. Why doesn't my sample spin?

1-There is no Air.

2- Air flow is too low.

3- Air spin rate on BSMS Control Window is set to zero. You can adjust that from BSMS Control Window.

4- The black and white tape on the spinner turbine is worn or the spinner turbine is damaged. Use another one.

5- Sample height is not adjusted properly. Use the depth gauge to measure it.

6- The sample is too heavy. If you are using the heavy wall NMR tubes or NMR tubes with heavy values, you may have a spinning problem. You may be able to fix this problem by increasing the gas flow, or increasing the spinner rate form the BSMS Control Window.

7- If you are doing a low temperature experiment, most possibly there is some ice formation due to moisture condensation. You should raise the temperature and flush the system with dry air or preferably with nitrogen gas.

[Click to go back to the menu](#)

Q5. Why I cannot lock to the deuterium signal after I put my sample into the magnet?

1- Deuterated solvent was not used.

2- The sample has not settled down into the probe properly because of (a) too much airflow, and (b) the sample height either was not adjusted prior to putting the sample into the magnet, or the sample height changed and moved due to the sample being loose inside the spinner.

3- Make sure the field sweep (on the BSMS Window) is on.

4- Someone has done some radical shimming and has ruined the minimal homogeneity required for observing the lock signal. You should try to reload the shims by calling their values from the shim files. Use **rsh** to invoke the saved shim library list and then click on " Current_bbo".

5- Worst scenario; there is no magnetic field because the magnet has quenched *OUCH!*

[Click to go back to the menu](#)

Q6. How do I improve the resolution for my spectrum? The peaks look awful!

1- Make sure you are using the lock, or alternatively, if you can not use the lock, the lock sweep is off.

2- Bad shimming could cause this problem. Try to shim by typing **topshim** again.

3- Bad sample. Sample preparation is important. Make sure that you have enough sample solution; the sample height in the tube should be around 5.0 cm. Sometimes metallic impurities in the sample can cause this problem. Make sure you sample looks clear; any undissolved solid in your sample may have an undesirable effect on your spectrum.

4- Poor quality NMR tube.

[Click to go back to the menu](#)

Q7. Why are the chemical shifts for the peaks in my spectrum are incorrect?

You need to set the reference peak (TMS or your solvent peak).

[Click to go back to the menu](#)