Brief Topspin 3.2 User Guide for Bruker NMR Spectrometers
Avance III 800, Avance III 600

OVERVIEW

This document is intended to be a brief, bare-bones users guide for NMR data collection using the Avance-III Bruker NMR spectrometers managed by the UC Davis NMR Facility. For detailed help with both routine and advanced NMR experiments, please consult Bruker's User Guides, which can be found within the Resources tab on our website, nmr.ucdavis.edu.

CONVENSIONS

Keyboard input is shown as **boldface** type in this manual. Note that in TS the "enter" key must be used after the command is typed; this is assumed through-out this manual and "enter" key strokes are not given explicitly. Commands in TS are typed in on the TS command line near the bottom of the TS window; again this is assumed and will not generally be stated explicitly herein. LMB, MMB, and RMB are used to indicate actions of the left, middle, and right mouse button respectively. On a PC the mouse wheel acts as the MMB. Click, and double click refer to pressing the LMB.

GENERAL PROCEDURE

You will find that the general procedure for acquiring NMR data on all NMR spectrometers is essentially the same. The general procedure is as follows:

Experiment: Routine Proton NMR

Instrument: 800 Medsci, 600 Medsci, 400 Chem

A) SAMPLE PREPARATION

1) Dissolve your sample in an appropriate deuterated NMR solvent. Make sure there is no un-dissolved material. If there is, you will need to either centrifuge or filter your sample to remove crystals/debris.

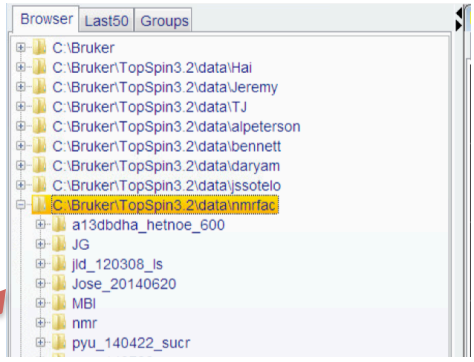
> TIP: use a small amount of KimWipe in a Pasteur Pipette to quickly filter out any undissolved material

2) Transfer about 600 uL of solvent into a clean NMR tube. We recommend high-quality NMR tubes - rated 600 MHz or higher, but economy tubes will be OK for routine work at lower fields (400 MHz and below). **Your sample height should be about 5 cm.**

B) LOGIN AND STARTUP

1) **Log In:** Log into your user account.
   a) User ID: ad3\KerberosID
   b) password: YourKerberosPassword

2) **Launch Topspin:** Launch Topspin 3.2 (or 3.5) software using the icon on the desktop

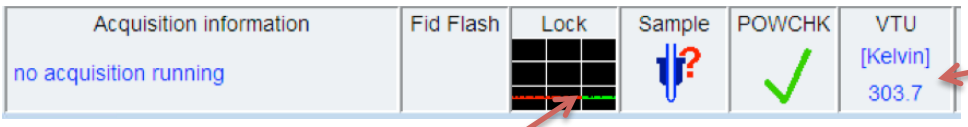


3) **Navigate to your data directory** in the file browser on the left. Example file tree: C:\Bruker\Topspin3.2\data\UserName

> NOTE: Reading in a standard shim set (step 4) is not always necessary. Usually you can get away with skipping this step, but if you end up having trouble locking or shimming, try reading in the standard shim set

4) **Open the Lock Panel:** Double-click on the Lock panel to launch the Lock display, or type **lockdisp** on the command line

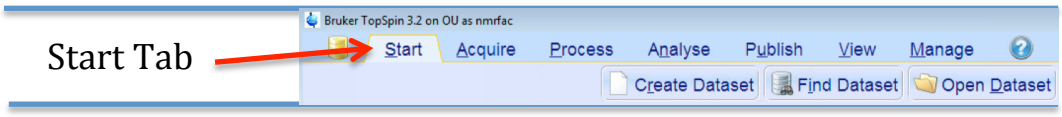
5) **Read shims:** Read in an optimal shim set by using the Read Shims command **rsh**. On the Topspin command line, type in **rsh bbo** or **rsh cptci** depending on the probe you are using (see table below). The NMR Facility staff will constantly update the **bbo** and **cptci** shim files.



Temperature Control **edte**

Lock Panel **lockdisp**

Standard Shim Files:	
800 MHz Medsci:	cptci
600 MHz Medsci:	cptci
500 MHz Medsci:	bbo
400 MHz Chemistry:	bbo



C) CREATE DATASET / SET INITIAL PARAMETERS

IMPORTANT: There are two main philosophies on how to set up your initial parameters. 1 – Copy parameters from an old data set into a new one, or 2 – Read in a generic parameter set. Both methods are described below.

Option 1: Copy Parameters From an Old Data Set (Suggested Option)

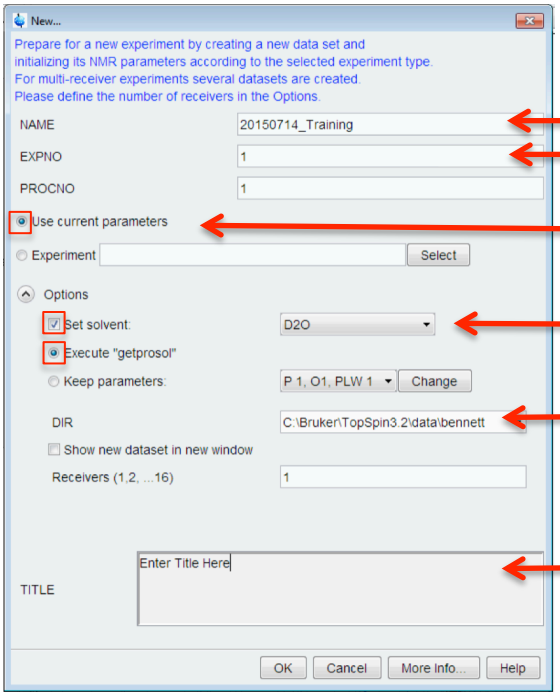
1) Use the file browser on the left to load an old experiment into the workspace. For example, double click on your most recent Proton NMR experiment. Make sure the spectrum has loaded into the viewing window.

2) From this old experiment, create a new experiment by typing **edc** or **new** on the command line, or by hitting the Create Dataset button. Make sure the Options tab is opened to display all setup options

TIP: If you use the naming format YYYYMMDD_SampleInfo, your file tree will be organized by date. Many users find this very helpful!

Create a new data set using **edc** or using Create Dataset button under the Start tab.

When you type **edc**, you are copying the major experimental parameters from an old experiment and pasting into a new data set.



Provide new experiment name
Experiment number

Select Use Current Parameters

Set Solvent (select from drop down)
Select Execute "getprosol"

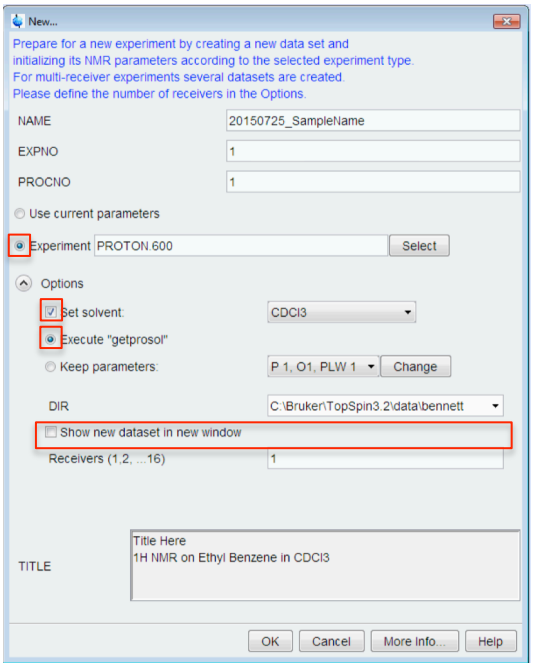
Make sure you are using your data directory!

Enter a title here. You can change the title information at any time by typing **edti** on the command line.

3) Hit OK. You now have initial Proton acquisition parameters, which are identical to the experiment from which you copied them. To check and edit these parameters, type **ased** on the command line

Option 2: Use a Standard Parameter Set

1) Type **new** on the command line, or select the Create New Dataset button under the Start tab.
2) Provide experiment name. Common example: YYYYMMDD_SampleInfo. Do not use spaces or special characters.
3) Enter experiment number. Often users start with EXPNO 1
4) Select "Experiment" option, and enter the following in the experiment selector
    a. For 800 MHz, enter PROTON.800
    b. For 600 MHz, enter PROTON.600
    c. For 500 MHz, enter PROTON.500
    d. For 400 MHz, enter PROTON.400
5) Under Options tab, set your solvent
6) Select execute "getprosol" option
7) Make sure you are in your user directory. The DIR should look like **C:\Bruker\Topspin3.2\data\username**
8) Enter a title. There are no restrictions here, type whatever you want.
9) Hit OK. You now have initial parameters from a generic Proton parameter set

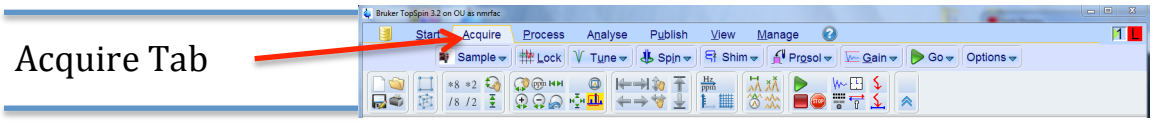


TIP: **edc** will remember your last choice. For example, if you loaded PROTON.800 experiment in the **edc** window, this same choice will be selected if you use **edc** again to create another experiment. In many cases, you will want to select "Use current parameters" instead of "Experiment".

USING PARAMETER SETS

You can also load standard parameter sets for initial acquisition parameters. For a standard Proton experiment, load parameters by typing the following on the command line

rpar PROTON.800 all; getprosol	Instrument: 800 MHz
rpar PROTON.600 all; getprosol	Instrument: 600 MHz
rpar PROTON.500 all; getprosol	Instrument: 500 MHz
rpar PROTON.400 all; getprosol	Instrument: 400 MHz



A) INSERT YOUR SAMPLE

1) Place your sample into the blue spinner
2) Adjust the sample height using the depth gauge
3) Remove fingerprints and debris from the NMR tube using a Kimwipe
4) Remove the protective black cap from the magnet, if present
5) Type **ej** on the command line to start the eject gas flow
6) Carefully place your sample into the magnet. It should float on a bed of air
7) Type **ij** to insert your sample. Wait until the spinner icon on the bottom menu indicates that your sample has been inserted correctly..

B) LOCK, TUNE, SHIM

1) LOCK: Type **lock** on the command line or hit the Lock button, then select you solvent from the list.

2) TUNE: Type **atma** to automatically tune and match the Proton channel, or type **atmm** to control the match and tune stepper motors manually. If you use **atmm**, be sure to save the probe position by selecting File – Save Position, and then File – Exit.

CAUTION: **atma** or **atmm** will only tune the nucleus that is open from your current experiment. Example, if you have a 1H experiment loaded, only the 1H channel will be tuned. Don't forget to tune 13C if you plan to do a 13C experiment later!

3) SHIM: The shimming procedure varies slightly depending on the instrument and solvent you are using. From the command line enter the indicated command depending on the instrument and solvent:

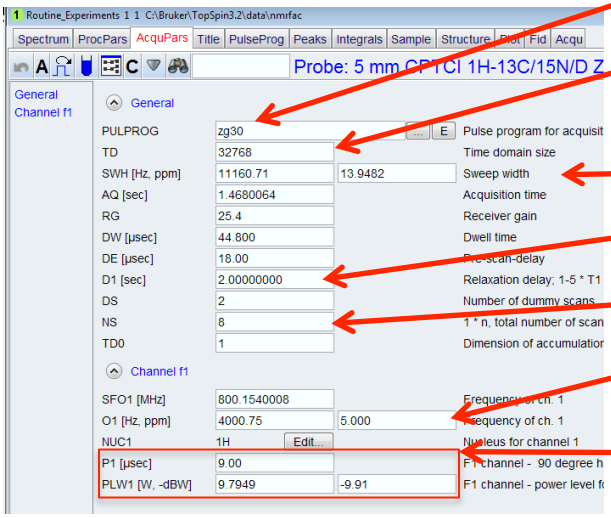
Instrument	Solvent Type	Topshim Command
800	Organic	topshim tuneb convcomp
800	Aqueous	topshim tuneb
600	Organic	topshim tuneb convcomp ordmax=4
600	Aqueous	topshim tuneb
400	Organic	topshim tuneb

NOTE: On the 800, **topshim** seems to miss the Z1 shim by about 15-20 units when the solvent is either H2O/D2O or D2O. For this reason, for optimal lineshape results on aqueous samples you may have to first perform **topshim**, and then manually add 15-20 units to the Z1 shim using the BSMS panel.

C) CHECK ACQUISITION PARAMETERS

1) Type **ased** on the command line. This will display the important acquisition parameters in a condensed table. To access all of the acquisition parameters, type **eda** on the command line.

2) Modify acquisition parameters if desired.



Pulse Program. zg30 means 30° hard pulse, and acquire

Number of points collected. Usually 32k or 64k

Sweep Width, in Hz and in ppm

Relaxation Delay, usually 2 seconds for

Number of scans

Carrier Frequency in ppm. Center of your spectrum

90° pulse length and power. The **getprosol** command will populate these values for you.

You can also use the command **pulsecal**, which will find the accurate 90° pulse

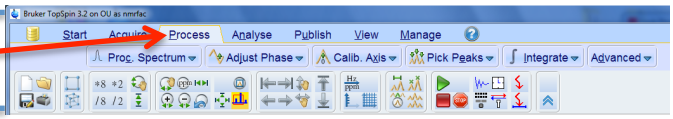
D) CHECK RECEIVER GAIN

1) Type **rga** on the command line to automatically set the receiver gain, or hit the Gain button

E) ACQUIRE YOUR DATA

1) Type **zg** on the command line, or select the Go button.

Process Tab



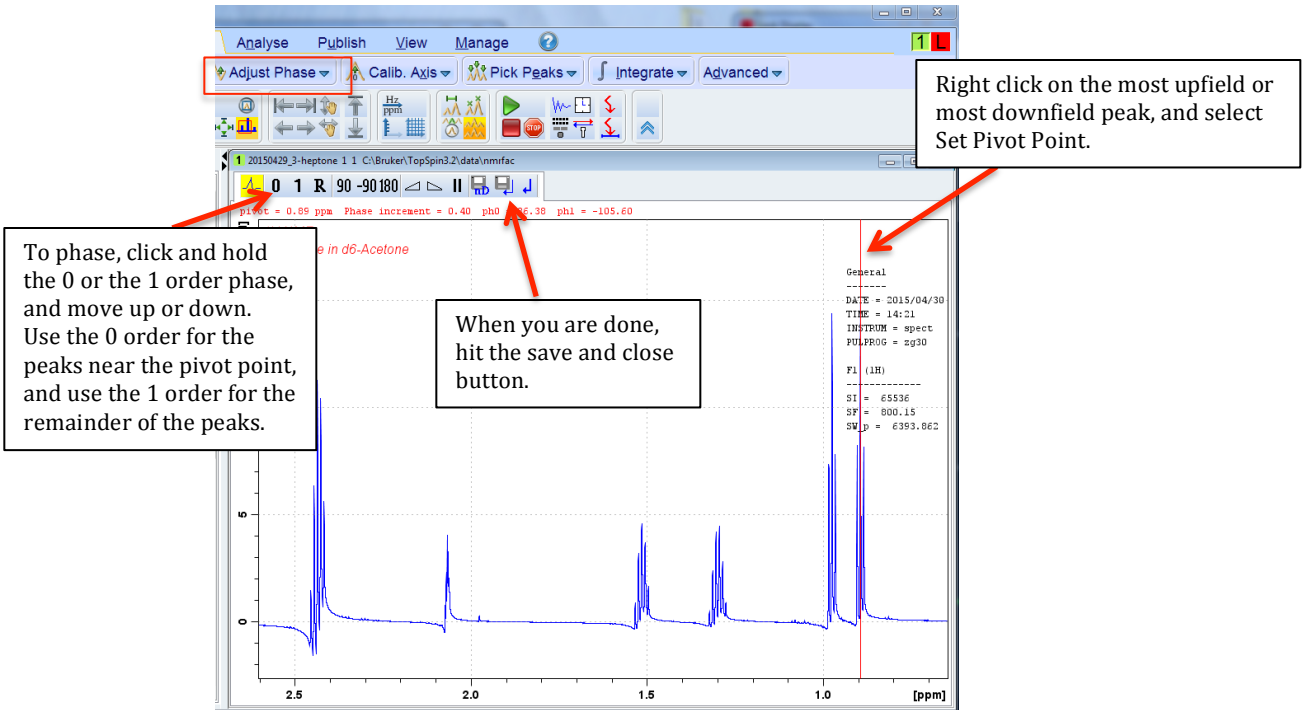
A) INITIAL DATA PROCESSING

1) Navigate to the Process tab
2) Perform a weighted Fourier transform by typing **efp** on the command line. Check the exponential weighting factor by typing **lb.** Typical weighting factors for 1H NMR are 0 to 0.3 Hz.
3) Perform automatic phasing of your data by typing **apk** on the command line
4) View your full spectral window by typing **.all** on the command line, or by using the navigation buttons.

NOTE: You can also select the Proc Spectrum button, which will perform **efp**, then **apk**, and finally an automatic baseline correction **abs**. You can change the exponential weighting factor by typing **lb** into the command line. Typically, one uses 0.1 to 0.3 Hz exponential weighting.

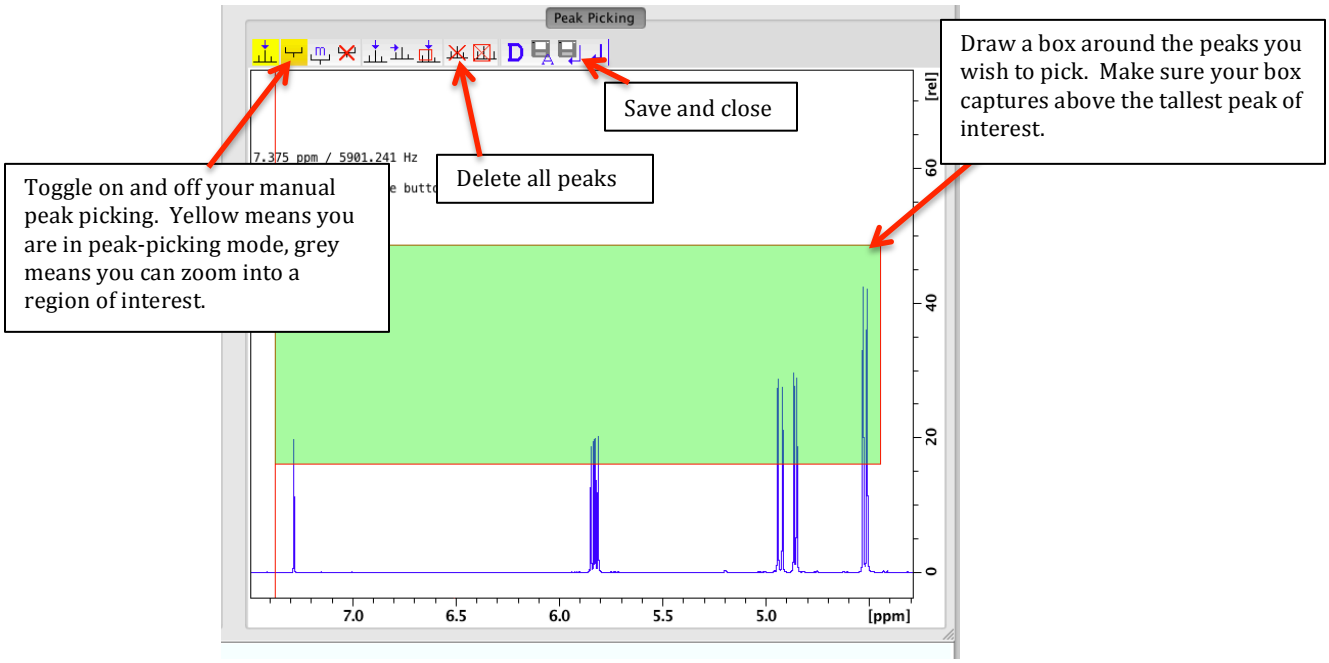
B) MANUAL PHASE ADJUSTMENT

1) Often times, automatic phasing will not do an adequate job. To perform automatic phasing, select the Adjust Phase button, or type **.ph** on the command line
2) Set the pivot point to either the most upfield or the most downfield resonance: right-click on the peak, and select Set Pivot Point
3) Use the 0 order to phase the peaks near the Pivot Point, and the 1 order to phase the other peaks.



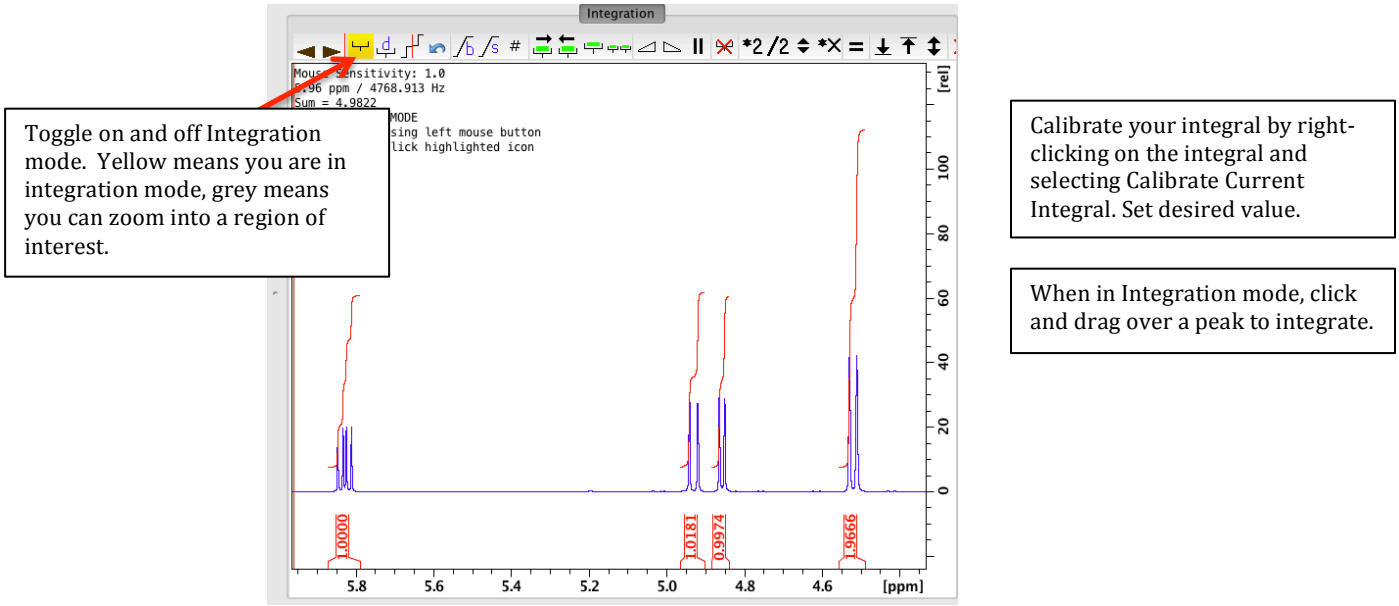
C) PEAK PICKING

1) Perform peak picking by typing **.pp** on the command line, or select the Peak Picking button under the Process tab.
2) Draw a box around the peaks you wish to pick. Make sure you catch the top of the peak.



D) INTEGRATION

1) Perform peak picking by typing **.int** on the command line, or select the Integrate button under the Process tab.



Exit Procedure

A) REMOVE YOUR SAMPLE

1) Type **ej** on the command line
2) Remove your sample, and place the black cap onto the bore of the magnet
3) Type **ij** to stop the air flow. Do not insert any blank sample
4) Place the empty spinner in the sample holder for the next user

B) COPY YOUR DATA TO KONA

1) Open up a Windows Explorer and navigate to the Shared drive, S:\\Share$
2) Open up a second Windows Explorer, and navigate to your Bruker data directory: C:\\Bruker\Topspin3.2\data\username
3) Find the experiment names in your user directory that you wish to copy to KONA
4) Copy, or drag and drop to your data to your KONA folder

C) LOGOUT AND SIGN LOGBOOK

1) Close Topspin 3.2 and any other applications
2) Log out of your account using the Start Menu
3) Sign the logbook
4) Fill out the billing slips.