TopSpin
QuickStart

Preliminaries

Sign Up

> Sign up for instrument time at `calendar.yahoo.com/hmcnmr` The password is `nmr`. During the summer, 1100-1200 and 1600-1700 are reserved for walk-on use (15 min maximum use).

Default State

> TopSpin should be running at all times. If this is not the case, you may need to *Log In* and *Start TopSpin*.
>
> Log In
>
> > If you are presented with the blue logon screen, click on the NMR icon. Enter the password. (Please note that passwords are case-sensitive.)
>
> Start TopSpin
>
> > If TopSpin is not already running, you can start it by double-clicking the TopSpin icon on the desktop.

Sample insertion

> Press `LIFT ON/OFF` on the BSMS keypad. Fit your sample with a spinner collar set to the correct depth, and place your sample on the stack. Press `LIFT ON/OFF` again. Wait for the SAMPLE DOWN green light on the BSMS console.

Guided Acquisition/Processing

Data Acquisition Guide

> Users with less experience may wish to be guided through the data acquisition and processing processes. The Data Acquisition Guide (shown at right) and Data Processing Guide (next page) will do this, suggesting automated procedures for each step in the process as well as allowing for more user intervention, if desired. To use this approach: from the SPECTROMETER menu, select *Data Acquisition Guide*. You can click through the
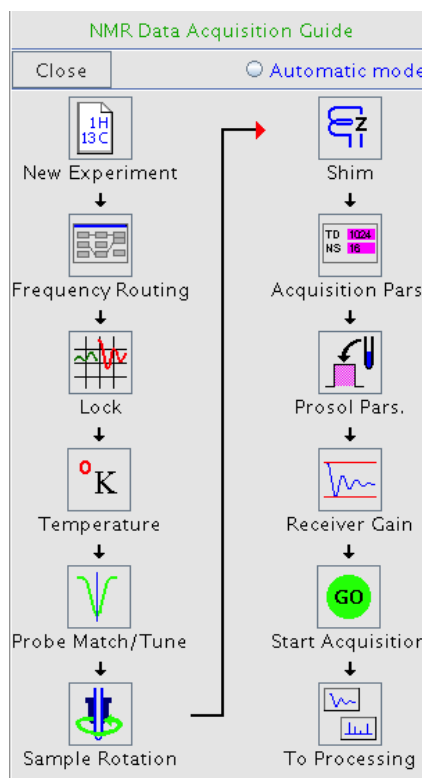
various boxes in the flowchart to acquire and process data.

If you first click on the "Automatic Mode" radio button at the top of the Guide, then clicking on an icon will apply the automated response. If you don't use automatic mode, then the first choice presented is always the automatic response.

Most users will not need to visit the Frequency Routing and Temperature settings for acquisition. In a similar vein, the processing settings for Window Function, Baseline Correction, Advanced, and Email/Archive are seldom used.

More experienced users may wish to run the entire process manually… The following discussion expounds on each set of options, in turn.
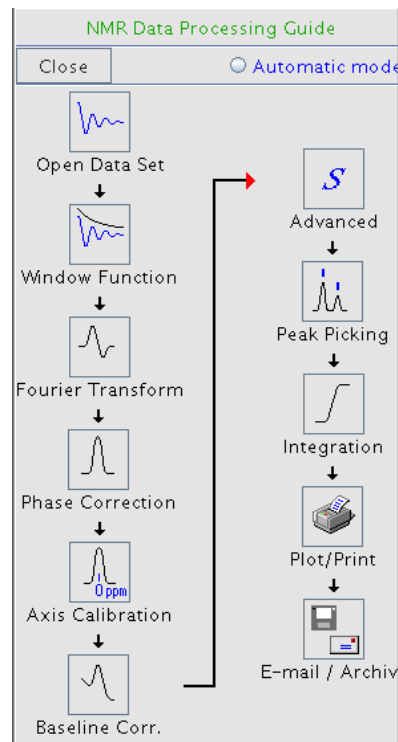
Manual Operation

New Data Set
    Type `new` on the TopSpin command line. Fill in:

`NAME`	A descriptive name for this data set.
`EXPNO`	A number to distinguish between different experiments within the same data set.
`PROCNO`	A number to distinguish between different processing within the same data set. Typically set to "1" unless you are post-processing your data.
`DU`	The physical name of the disk where the data is stored. `/opt/topspin1.3` is the default – don't change this.
`USER`	For faculty, your *lower-case* initials. For students, the *lower-case* initials of your faculty research advisor.

Enter whatever you like for the title. Click on save. You can change any of these fields at any time by typing `edc`. Of these selections, the `USER` field is most important—this is what tags the data as yours. Please follow the conventions shown in the table above!

Existing Data Set
    You can use the parameters associated with an existing data set by loading that data set, then using `edc` to change its name. The acquisition and processing

parameters associated with that data set remain until you overwrite or change them.

Select Experiment

To use one of the Bruker predefined experiments, type `rpar.` Select the parameter set you will use. Select your parameter set. (For typical proton spectra, use `PROTON`) Select all parts of the parameter file by clicking on `Copy All.`

Next you will need to read in pulse length and solvent information for the current probehead: Type `getprosol` to do so.

You may edit acquisition parameters with `ased` (for the primary parameters used in the current pulse program), or with `eda` (which lists all acquisition parameters. Alternatively, you can change individual parameters by typing their name on the command line. For example, you can change the number of scans by typing `ns`, which will prompt you for the number of scans you wish to run, or you can type `ns 64` to set the number of scans directly. You can examine the signal routing with `edsp`.

Locking

If the lock display is not already active, type `lockdisp` at the TopSpin command line. Type `lock` at the TopSpin command line to be prompted for your solvent, or you can type `lock cdcl3` (or another appropriate solvent) directly. Alternately, you can click on the lock display icon on the menu bar, or double-click on the lock icon at the bottom-right of the screen. At the end of successful locking you should have a constant lock level signal in the middle-to-upper portion of the lock screen with little noise. If this is not true, lock again. If the lock signal moves off the top of the screen, use the `lock gain` button on the BSMS keypad to move it to about ¾ of the way up the screen.

Tuning and Matching

If you are using an atypical solvent, you should tune and match. If you are working in deuterochloroform and the last user did as well, you likely will not need to tune and match.

Make certain that sample rotation is off with the `SPIN ON/OFF` button on the BSMS keypad. Type `wobb` at the TopSpin command line to start the wobble process, then type `acqu` to view the acquisition window. For protons, *gently adjust* the tune screw (yellow tape, marked with a "T") on the bottom of the probe to position the "V" of the wobble curve on the spectrometer frequency. Gently adjust the match screw (yellow tape, marked with a "M") to make the dip as deep as possible. When you are satisfied with the tune and match, click on the `return` icon at the top of the wobb window.

X-band nuclei on the BBI probe: For nuclei other than protons on the BBI probe, first read in the correct acquisition parameters with the appropriate `rpar` command followed by `getprosol`. Next, gently adjust the tune and match sliders to the values listed on the card attached to the probe. Fine-tune in order to get a deep "V" at the correct frequency. When you are satisfied with the tune and match, click on the `return` icon a the top of the wobb window.

X-band nuclei on the QNP probe: First read in a *phosphorus* acquisition parameter file with the appropriate `rpar` command e.g., `rpar P31 all`, followed by `getprosol`, and `wobb`. If phosphorus is the nucleus you wish to observe, you're done – ,type `halt` to stop wobble, then `return` to return to the main TopSpin screen.. If you want to observe ^{13}C or ^{19}F, then read in a parameter set for the appropriate nucleus with `rpar` and issue the `getprosol`, and `wobb`, commands. If you are observing carbon, adjust the blue screw to centert the "V". If you are observing fluorine, adjust the white screw. When you are satisfied with the tune and match, click on the `return` icon a the top of the wobb window.

Sample Rotation

Most two-dimensional NMR experiments are designed to be run without sample rotation. One-dimensional experiments can be run either with our without sample rotation.

Shimming

Manual shimming: Touching up the shims manually is typically good enough for most users. On the BSMS keypad click on `z1`. Adjust this shim using the knob in order to maximize the lock signal. Repeat with `z2`. Now touch up `z1` again. (If you mess up royally while turning the knob, you can always return to the initial value of that shim by pressing its key twice in rapid succession.) When finished with manual shimming, press the `standby` key.

Reload the default shim file: If manual shimming still results in substandard performance, reload the default shim file. If the current probe (shown on the sign on the side of the NMR electronics rack) is BBI, then type `rsh bbi` at the TopSpin command line. If the current probe is QNP, then `rsh qnp` is the command. Try manual shimming again.

Computer-controlled shimming: If you are still unhappy with the shims, you can start computer-controlled shimming by typing `tune.` Select the shim group; the instrument will launch a simplex shim on this group. (Best results are often obtained by shimming on `z1-z2-z3` while spinning.) The shim status will be updated on the lower line of the TopSpin window.

Full-sequence computer shimming: For particularly difficult problems, try the automated sequence `tune daily` which shims the spinning shims, the first-order non-spinning shims, then the spinning shims again.

Receiver Gain
Type `rga`. Wait for the receiver gain to be set.

Starting the experiment
Type `zg`. If you want to view the FID during acquisition, type `acqu` or click on the FID tab.

Processing the data
Line Broadening: For nuclei other than protons: Set `lb` to 1 or 2 Hz, then type `em` to invoke exponential multiplication of the FID.

Fourier Transformation: Type `ft` to transform the data.

Expansion: Drag with the left mouse button depressed to expand a region of the spectrum. To see the entire spectrum again, click on the |< >| icon above the spectrum.

Phasing: `apk` will do automated phasing, whereas `pk` will phase using the existing `ph0` and `ph1` values. To do phasing manually, click on the Phase icon. The largest peak on the display will be the pivot point (you may wish to expand a region of the spectrum before entering the phase routine). Set ph0 by clicking and holding the mouse on the 0 icon above the spectrum. As you move the mouse up and down you will vary ph0. The 1 icon adjusts ph1 in a similar fashion. Click on the Save & Return icon to keep your new phasing, or Return to cancel your new phasing.

Integration: `abs` will correct the baseline and do automated integration. `int` will give you the option of automated or manual integration. You integrate manually by selecting the first icon on the integration toolbar. You define a region by dragging with the left mouse button. You can delete an integral or define its value by right-clicking on it. You can expand by clicking off the highlighted icon, then dragging across the region of interest. Remember to Save & Return when finished (or just Return to discard your results).

Peak Picking: Type `pp` to enter the peak picking routine. `mi` and `maxi` define the minimum and maximum intensity for peak picking. Type `mi`, then enter the minimum intensity.

Adding a Title: Click on the Title tab to edit the spectrum title. You can also put sample information in the Sample tab.

Plotting: You can turn on and off the integrals and peak labels by right-clicking on the screen to access Spectral Display Preferences. Selecting Print from the FILE menu allows you to select "Print active window" (`prnt` from the command line) and plot exactly what you have on the screen. Alternately, selecting "Print with layout" (`plot` from the command line) will allow you to specify an XWinPlot format file to add acquisition parameters. If you know you won't change anything in the plot, you can use the `autoplot` command.

All Done?

When finished, tidy up any open windows. Leave TopSpin running with its main window open on the active desktop. Do not log out. If you are the last user for a time, turn the monitors off.

Basic NMR Experiments

Under the SPECTROMETER menu is the *Basic/Selective Experiments* option. Selecting this option opens the Basic NMR Experiments window. Use the options given to set up and execute a basic set of NMR experiments.

Yellow buttons help you get the instrument setup up correctly for your new sample: Selecting *Lock* will bring up the lock solvent menu. Selecting *Tune/Match* will allow you to manually tune on both ^{1}H and X. *Auto Shim* will do a linear shim on Z1, Z2, and Z3.

Blue buttons help you to run a one-dimensional experiment once the sample is inserted, locked, tuned, and shimmed: *1H* will run the basic proton experiment. *X{1H} with NOE* will run a composite pulse decoupling ^{13}C experiment. Note that the default here is 1024 scan (roughly 1 hour for acquisition) – you may wish to cancel the first window which opens, then type `NS` and set the number of scans to a more reasonable value, something between 32 and 256 scans.

Green buttons set up and run a two-dimensional experiment once you have already acquired a one-dimensional proton spectrum. Use these buttons when the spectrum window shows the completed proton spectrum: *1H-1H Gr. COSY* will run the gradient-based COSY experiment, while *1H-X Gr. HSQC* will run the gradient-based heteronuclear single quantum coherence, or proton-carbon correlation spectroscopy experiment.
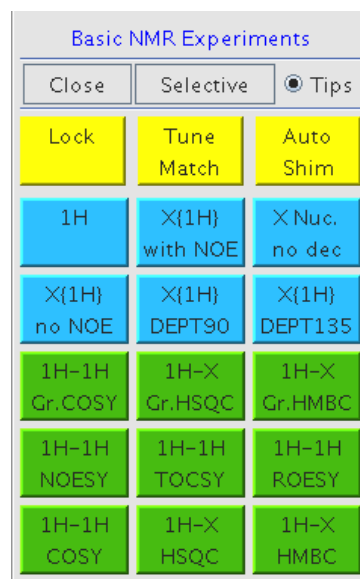
When the experiment is complete, you may wish to use the Data Processing Guide to help you work up your data (see page 1 of this document). Under the PROCESSING menu select *Data Processing Guide*.

Remote Processing

You are encouraged to work up complex data sets on a PC, allowing the instrument to be used for acquisition. There is a guide to working up data using a stand-alone copy of TopSpin. This document is available online and beside the PC in the south instrument room of SuperLab.

Abbreviated QuickStart Instructions

Task	Command	Alternatives
log on	nmr (icon or username)	
launch	topspin (unix command line)	TopSpin icon
sample insertion	LIFT ON/OFF (BSMS keypad)	ej *and* ij
display lock signal*	lockdisp	
lock	lock cdcl3	lock
shim	z1, z2 on BSMS keypad	rsh bbi *or* rsh qnp tune z1-z2-z3 tune daily
new data set	new	edc
acquisition parameters	rpar getprosol	rpar PROTON all gpro
tune & match*	wobb	
set receiver gain	rga	rg 128
run experiment	zg	
line broadening*	lb em	lb 3 em
Fourier transformation	ft	efp
phase	apk	pk *or* [phase]
baseline correction*	abs	
integration*	[int]	int
peak pick*	pp	maxi 12 mi 3
title*		
preview*	view	
plot	prnt	plot autoplot

Key:
*	= can be skipped in most instances
[phase]	= click on the 'phase' icon
FILE -> read	= pull down the 'file' menu and select 'read'