

DCIF NMR TRAINING GUIDE

600 MHZ

BRUKER DRX “B600”

last edit 20061217

General Info:

The 600 NMR is equipped with a TXI Cryoprobe. The 1H coil is closest to the sample and cooled with liquid helium, the 13C and 15N coils are further away from the sample and at room temperature. Optimized for 1H detected experiments, the probe provides excellent 1H signal to noise for 1-10 mM samples, reducing acquisition time for 1D and 2D experiments. Due to the probe design, the signal to noise for direct observe 13C is lower than the Varian 501.

Variable Temperature:

Ask a staff member for training on how to use the temperature control software. The Cryoprobe requires the probe heater to be on continuously and has a limited temperature range. Researchers are responsible for calibrating the temperature inside the nmr tube as a function of sample/probe temperature and gas flow using a methanol or ethylene glycol sample. Nitrogen gas should be used for temperatures > 40 °C

Range: +10 to 60 °C (283 to 333 K)

Max. rate of change: 2 deg/min = 10 degrees every 5 min

Normal Operation

Gas:	Compressed Air (dewpoint < -70 °C)	
Temperature:	293 K	
Gas Flow:	670 L/h	
Heater:	ON	DO NOT TURN HEATER OFF!!!!
Max Heater:	10%	
Temp. Correction:	None	

Conventions used in this guide are as follows:

- **Boldface** type indicates commands that are typed into the TOPSPIN command line or in a terminal window
Unless otherwise noted, all commands typed into the command line are followed by an <↵ Enter> keystroke.
- Italic [**Boldface**] type with square brackets indicates a button in the TOPSPIN gui interface that is to be pushed.
- [**Boldface**] >[**Boldface**] type with square brackets separated by a bracket (>) indicates selections made through TOPSPIN pull-down menus.
- **LMB** indicates the Left Mouse Button
- **MMB** indicates the Middle Mouse Button
- **RMB** indicates the Right Mouse Button
- **Table format:** the left column contains the standard command line syntax, the right column shows button options.

Login, start the TOPSPIN software, sample preparation:

1. Login to B600, the HP host computer. If you do not have an account access is not permitted. No exceptions.
2. Double click the TOPSPIN icon with the **LMB**.

The screenshot shows the Bruker TOPSPIN 1.3 software interface. The main window is titled "Menthol 1 1 /u skel" and contains a "Spectrum" plot area. The plot area is currently empty, displaying the text "No raw data available" and "No processed data available". The interface includes a menu bar at the top with options like "File", "Edit", "View", "Spectrometer", "Processing", "Analysis", "Options", "Window", and "Help". A "Data Browser" is visible on the left side, showing a file tree structure. A "Command Line" is located at the bottom left, displaying the text "no acquisition running". A "Lock" button is located in the bottom right corner of the main window, with a red arrow pointing to it from a callout box that says "Right Click in the 'Lock' box to open the BSMS and Lock displays". A "Lock Display" window is open on the right side of the screen, showing a grid of plots. A "Watch for errors in the terminal window" callout box with a red arrow points to the terminal window on the left, which contains several lines of error messages. The Windows taskbar at the bottom shows the system tray with the time "19:11" and the date "Oct 17".

Menu

Data Browser

Data Window

Watch for errors in the terminal window

Right Click in the "Lock" box to open the BSMS and Lock displays

Command Line

Acquisition information	Fid Flash	Lock	VTU	Time
no acquisition running			[Kelvin]	19:11
			293.2	Oct 17

19:11

Setting up a new experiment

3. Type **new** in the TOPSPIN command prompt. The *edc* (current data parameters) window will open. **[File] > [New]** or **edc** open the same window.

Fill in the following information:

NAME: filename (NO SPACES: letters, numbers, dashes & underscores only)
 EXPNO: folder number for experiment
 PROCNO: folder number for processed data
 DIR: /u
 USER: <your-user-ID> i.e. KMbken

Data is saved to:

/u/data/<USER>/nmr/<NAME>/<EXPNO>/<PROCNO>
/u/data/KMbken/nmr/Menthol-Again/1/1

Select

Solvent

Experiment

- 1H-TXI-dcif (¹H parameters)
- 13C-CPD-TXI-dcif (¹³C parameters with ¹H decoupling)

Enter a title.

Click **[OK]**

OPTIONAL

To read parameters sets from the command line:

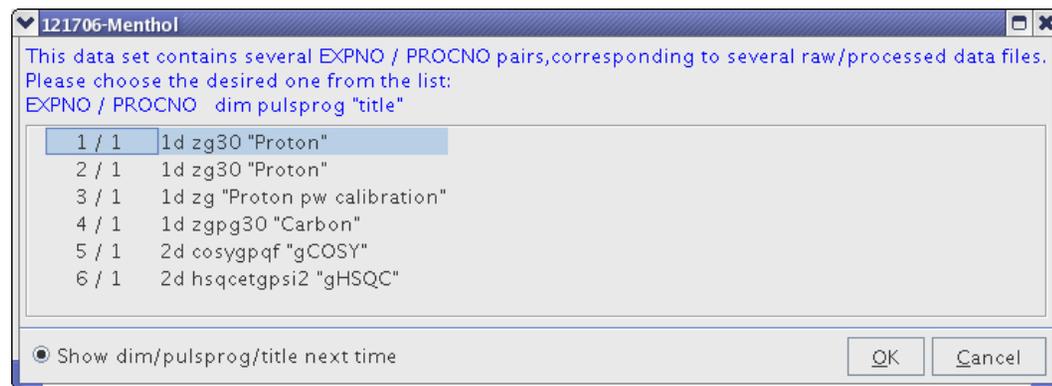
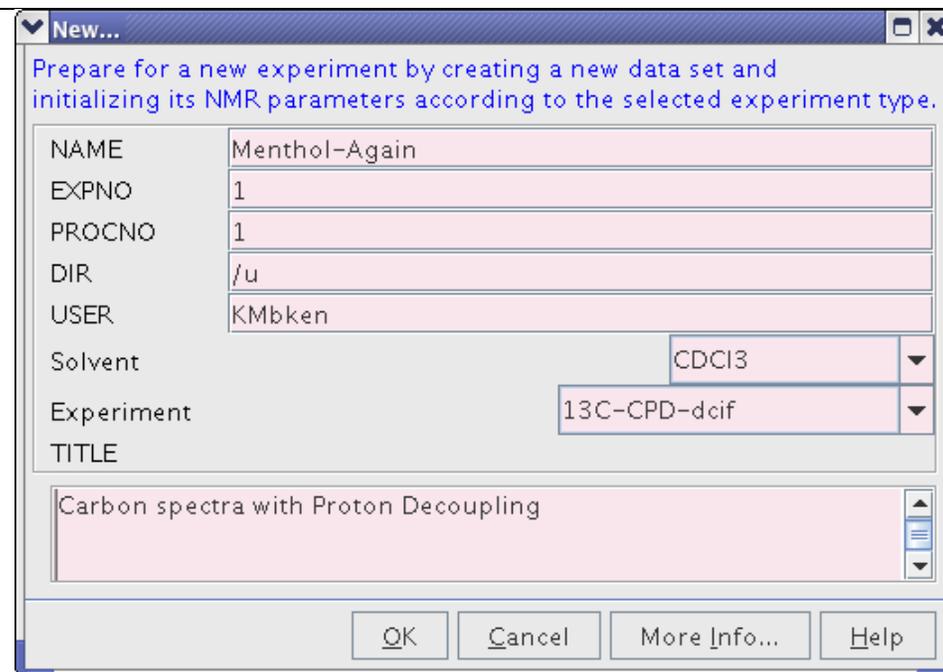
Type **rpar** (read parameter set) and select from the list, click **[OK]**

Use **rpar *dcif** to select from the DCIF parameter sets.

If you already have an existing data set with optimized parameters, read in that data set then simply change the name.

Example: Suppose you have a data set named **mysteryproduct** in experiment #1 and process #1. You may either select **[File] > [Open]** then choose the file named **mysteryproduct** or type **re mysteryproduct 1 1**. Then change the name using **edc**.

TIP: If you drag a dataset folder window from the browser to the data widow, a pop-up window will show a list of experiments by EXPNO, Pulprog and title.



Inserting your sample:

4. Insert your sample:
 - a. Type **ej** (eject sample) to turn on the eject gas. Be patient, it takes about 10 seconds for the air to come on.
 - b. Wipe off your sample, place it in the spinner, and line it up using the center line. Wipe the sample again, and then, making *sure* the eject gas has come fully on, place the tube/spinner on top of the magnet bore tube (it should float on the eject air).

BE VERY CAREFUL WHEN SETTING THE SAMPLE DEPTH!

- c. Type **ij** (insert sample) to turn off the eject gas.

Spinning:

Spinning is possible but not recommended on the Cryoprobe

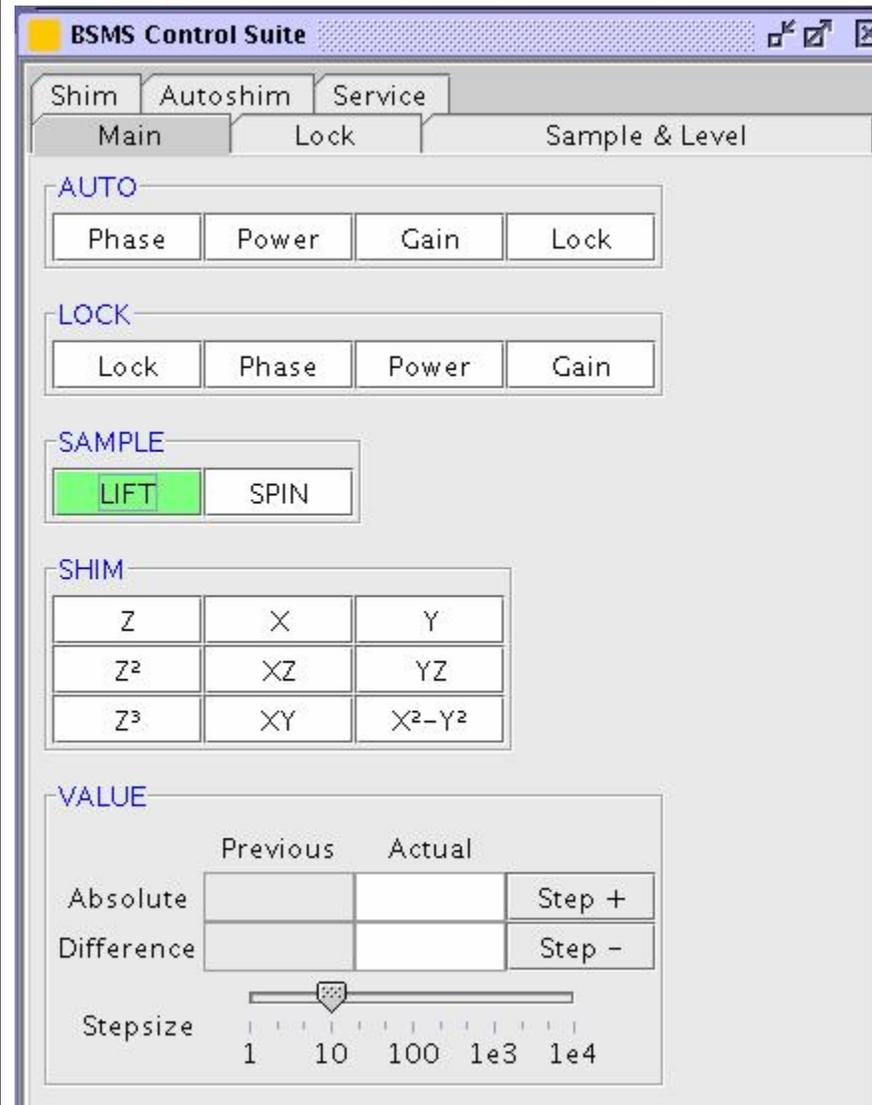
Type **ro on** (rotation on) to turn the spinner on. The default rotation speed is already set at 20Hz

NOTE:

Do not type commands into the command line when the BSMS window is open.

Alternative Button Option:

Type **bsmsdisp** to open the bsms control panel.



Click **[LIFT]** to eject, then click **[LIFT]** again to insert the sample.

Click **[SPIN]** and set the value. Close the BSMS Panel before using the command line.

5. Load the best shim file by typing **rsh best**. You can also retrieve some other shim file by typing **rsh** and selecting the file from the list.

Locking:

6. Type **lockdisp** A window will open and you will see the signal from the deuterium lock channel.

Alternative Button Option:

Click lock icon  on the top bar to open the Lock Display Window.



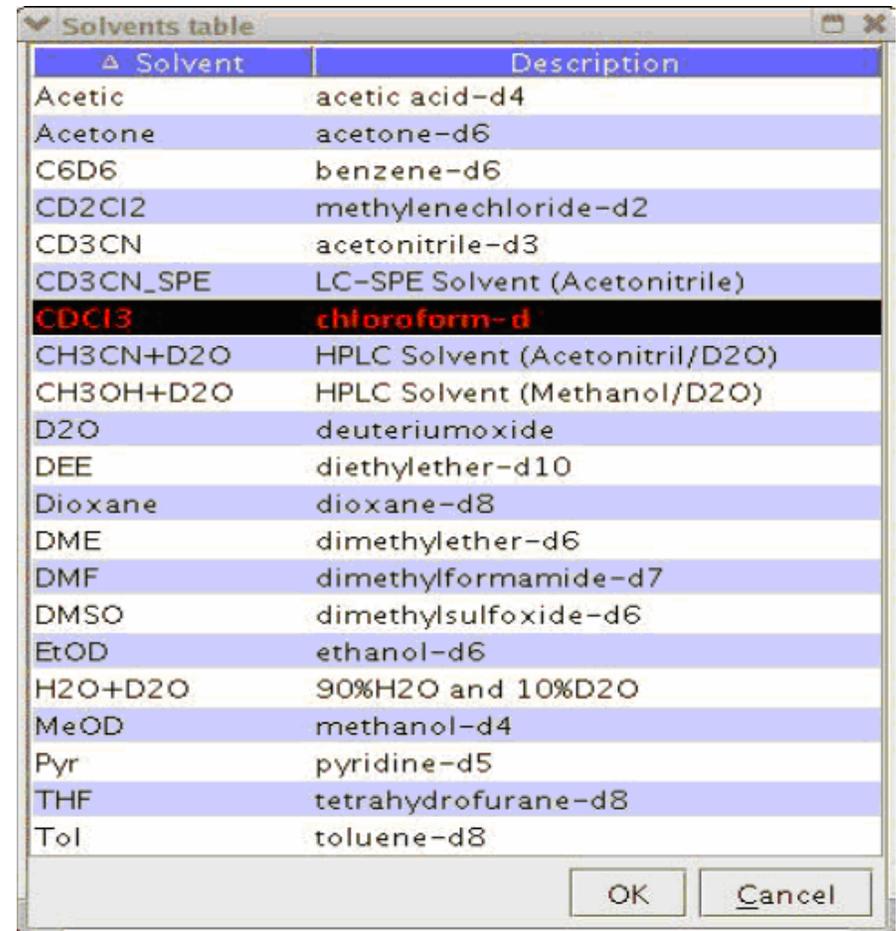
7. Type **lock**. The Lock Display Window will open. Select the deuterated solvent you are using. You can follow the progress of the locking routine by looking at the lock display. The lock display can be brought to the foreground by clicking along the edge of that window.

- Example: Suppose you would like to lock to deuterated chloroform you can also type **lock cdcl3** (lock space cdcl3) at the command prompt.
- The lock power and the lock gain are automatically determined.
- The lock level should be greater than 50%

Manual Locking:

- If the lock routine fails, open the BSMS panel Lock Tab.
- Turn the lock off
- Center the lock signal in the center of the lock display window by adjusting the field.
- Adjust the lock power to get a signal
- Adjust the lock phase, such that both signals have the same amplitude
- Turn the lock on
- Adjust the lock gain to get a lock level above 50%
- Check for lock saturation

Use manual locking for solvents with more than one type of deuterium.



Shimming:

8. Now you want to shim. To do this, follow these steps:
 - a. If you have not done so, open up the bsms display (type **bsmsdisp**).
 - b. Click the **[Shim]** tab.
 - c. If spinning, verify the spin has reached 20 Hz, the button should be green.
 - d. Click **[Z]**. Adjust the shim step size slider at the bottom of the Shim Panel, a step size of 10 is adequate for z1-z5.
 - Using the **LMB** click the **[Step +]** or **[Step -]** button to maximize the lock level in the Lock Display Window.
 - Or use the **MMB (-)** and **RMB (+)** on the shim button **[Z]** to maximize the lock signal.
 - e. If the level does "off scale", select the **[Gain]** button and reduce the lock gain to 60%.
 - f. Next, repeat steps d. and e. above to adjust the **[Z2]** and maybe **[Z3]** and **[Z4]** shims. Repeat d - f.
 - g. If the sample is not spinning, adjust the **[X]** **[Y]** **[XZ]** and **[YZ]** shims
 - h. Close the BSMS window when finished.
- If you would like to save your shim file, type **wsh** (write shim file) and give the file a name in the supplied window. This is recommended, for example, if you spend a lot of time shimming on water.
- Once the sample is locked and shimmed, multiple NMR spectra can be acquired. Locking and shimming are only needed when you change the sample.

The 600's Avance DRX Spectrometer has 28 shims:

Z¹, Z², Z³, Z⁴, Z⁵, Z⁶

X, XZ, XZ², XZ³, XZ⁴, X³, X³Z

Y, YZ, YZ², YZ³, YZ⁴, Y³, Y³Z

(X²-Y²), (X²-Y²)Z, (X²-Y²)Z², (X²-Y²)Z³, XY, XYZ, XYZ², XYZ³

The shims highlighted in blue are also in the Main Tab of the BSMS. Optimize these shims before adjusting the higher order shims.

BSMS Control Suite

Shim | Autoshim | Service

Main | Lock | Sample & Level

LOCK & SPIN

Lock | Phase | Gain | SPIN

SHIM

Z ⁰	X	Y	X ² -Y ²
Z	XZ	YZ	(X ² -Y ²)Z
Z ²	XZ ²	YZ ²	(X ² -Y ²)Z ²
Z ³	XZ ³	YZ ³	(X ² -Y ²)Z ³
Z ⁴	XZ ⁴	YZ ⁴	(X ² -Y ²)Z ⁴
Z ⁵	XZ ⁵	YZ ⁵	(X ² -Y ²)Z ⁵
Z ⁶	X ³	Y ³	XY
Z ⁷	X ³ Z	Y ³ Z	XYZ
Z ⁸			XYZ ²
Z ⁹			XYZ ³
Z ¹⁰			XYZ ⁴
			XYZ ⁵

VALUE

	Previous	Actual	
Absolute			Step +
Difference			Step -

Stepsize: 1 10 100 1e3 1e4

Sample: down missing up

9. Deuterium Gradient Shimming :

Gradient Shimming can shim Z1, Z2, Z3, Z4, Z5 simultaneously

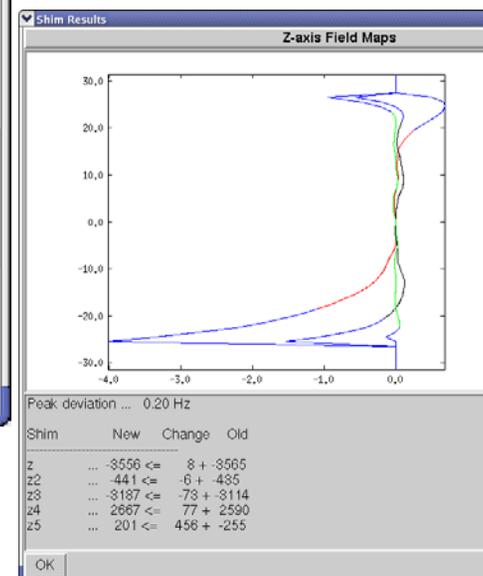
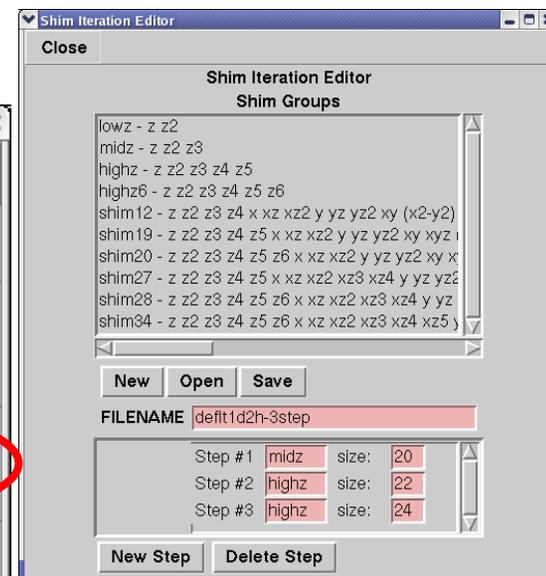
- Turn spinning off : **ro off**
- Type: **gradshim**
- Check the setup:
Method: **1D2H**
DISK: **/opt/topspin**, USER: **<your-user-ID>**
Data Set FILENAME: **gshim1d2h-dcif.CP**
- To create an iteration control file, governing which shims are optimized, open **[Edit] > [Iteration Control file]** and create the desired number of steps with shims groups and the size of the window used for the fit.
 - [Open]** select deflt1d2h.
 - Add the following steps.
 - midz 20
 - highz 22
 - highz 24
 - [Save]** file
- To open an iteration control file Press **[*]**

Always click the **LMB** inside the FILENAME window before starting gradient shimming.

- Click **[Start Gradient Shimming]**

g. The resulting field profiles should converge toward 0 ± 0.1 .

h. When finished, turn spinning on **ro on** if desired.



10. Type: **getprosol** to load the most recent pulse widths and power levels.

11. Entering a parameter name at the command prompt will open an input window. You can also assign a value to the parameter by typing
Example: **ns 4** (ns space 4)

- **ns** (number of scans)
- **solvent**
- **td** (number of data points)
- **ds** (dummy scans)
- **d1** (recycle or pre-acquisition delay)

If you are interested in more quantitative measurements you might also want to change **d1** (the recycle delay). Example: **d1 20** (d1 space 20)

- **sw** (sweep width)
- **o1p** (transmitter offset in ppm)
- **aq** (acquisition time)

Optional.

Type **ased**, to view and edit the list of the active parameters in the parameter set.

Type **eda** (edit acquisition parameters) to edit and view all acquisition parameters.

Type **edsp** to view the spectrometer routing table

12. Type **ii** (initialize interface) and wait for message 'ii finished'.

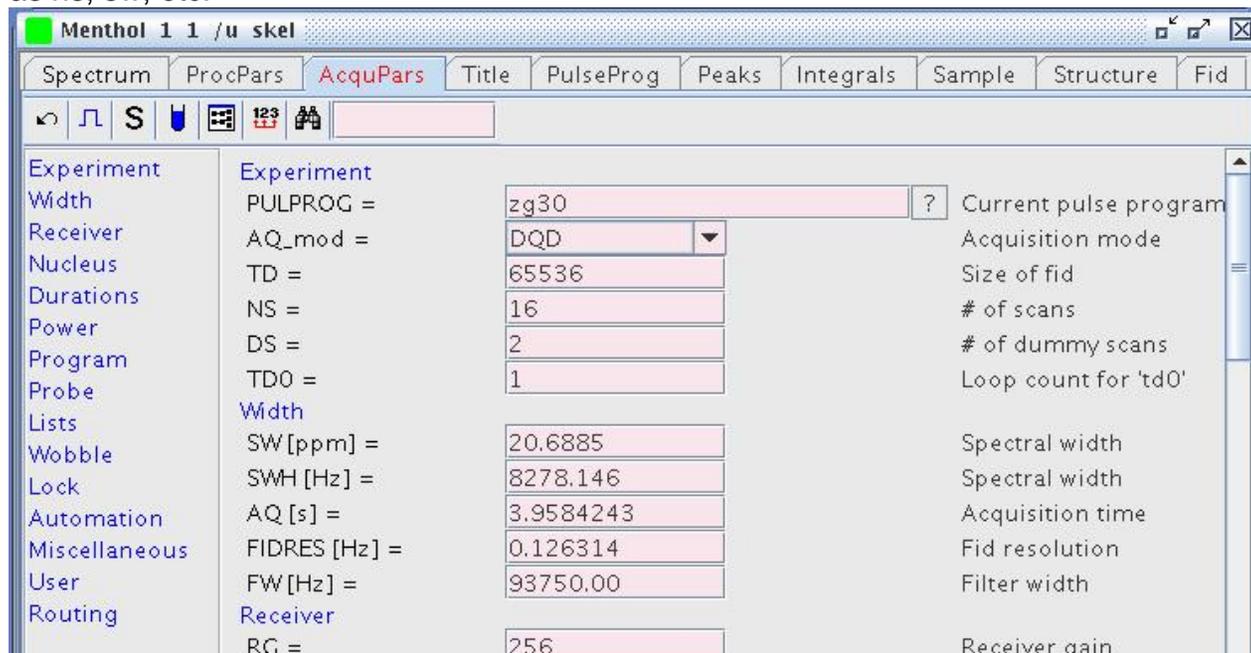
Optional.

Type **showpp** to view a graphical representation of the pulse sequence.

Type **expt** to view the acquisition time required.

Alternative Button Option:

Click [**AcquPars**] icon , you may change the experimental parameters in this window such as ns, sw, etc.



Click  to read in the correct power levels.



600 TUNING

If you have tuning questions ALWAYS ask a staff member!!! DO NOT USE FORCE when adjusting the tuning capacitors.

- To view the tune signal, type **wobb**. This starts the sweep generator which measures probe impedance as a function of frequency.
 - Use the blue screwdriver tool to adjust the Tune (T) and Match (M) knobs to center and minimize wobble curve on the monitor.
 - If the parameter set has multiple nuclei, switch nuclei by pressing the Channel Selection button on the Preamplifier or select the  button.
- Type **stop** or click  to get out of tuning mode.

TUNE & MATCH COLORS

YELLOW	1H
BLUE	13C
RED	15N

DO NOT TUNE
the **WHITE 2H**



Acquiring the FID

1. Type **rgacryo** to set the receiver gain the bottom of the TOPSIN display will read 'rga finished' when the receiver gain adjustment routine completes. (Cryoprobe ONLY)
2. Type **zg** to start the acquisition (zero data then go).

For proton only: **rga** and **xaua** will work

OPTIONAL:

- Type **tr** to transfer the currently completed transients (scans) to disk. This will allow you to process the data while the acquisition continues.
- Type **halt** to stop the experiment at the end of the current scan. The data set will be saved.
- Type **stop** to stop the acquisition immediately. Warning, the data is not saved!

To acquire another spectra with the same filename type **ixpno**, increment the experiment number and **rpar** the next parameter set

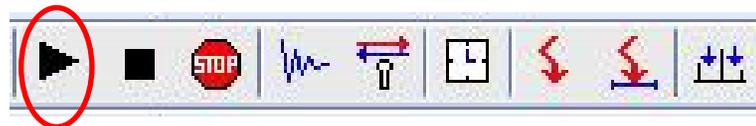
- a. To join previous experiments type **re number**, **re 1** will read experiment 1.

multizg will run multiple sequential expnos

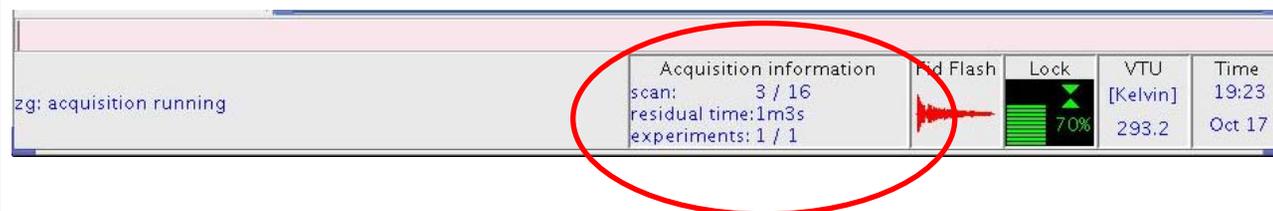
[Analysis]>[Multiple Spectrum Display] will allow multiple spectra to be overlaid.

OPTIONAL Button Commands:

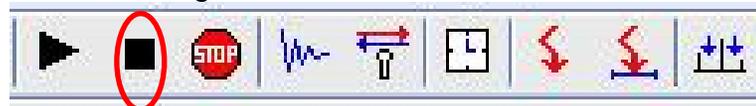
Click  to acquire a spectrum.



View the status of the experiment



To halt an acquisition and save the data. The stop button will abort the data collection without saving the data.



Researchers may process data at the spectrometer computers, see next section of processing information.

During walkon time, please use the offline data stations to process and allow others to acquire data.

Removing your sample:

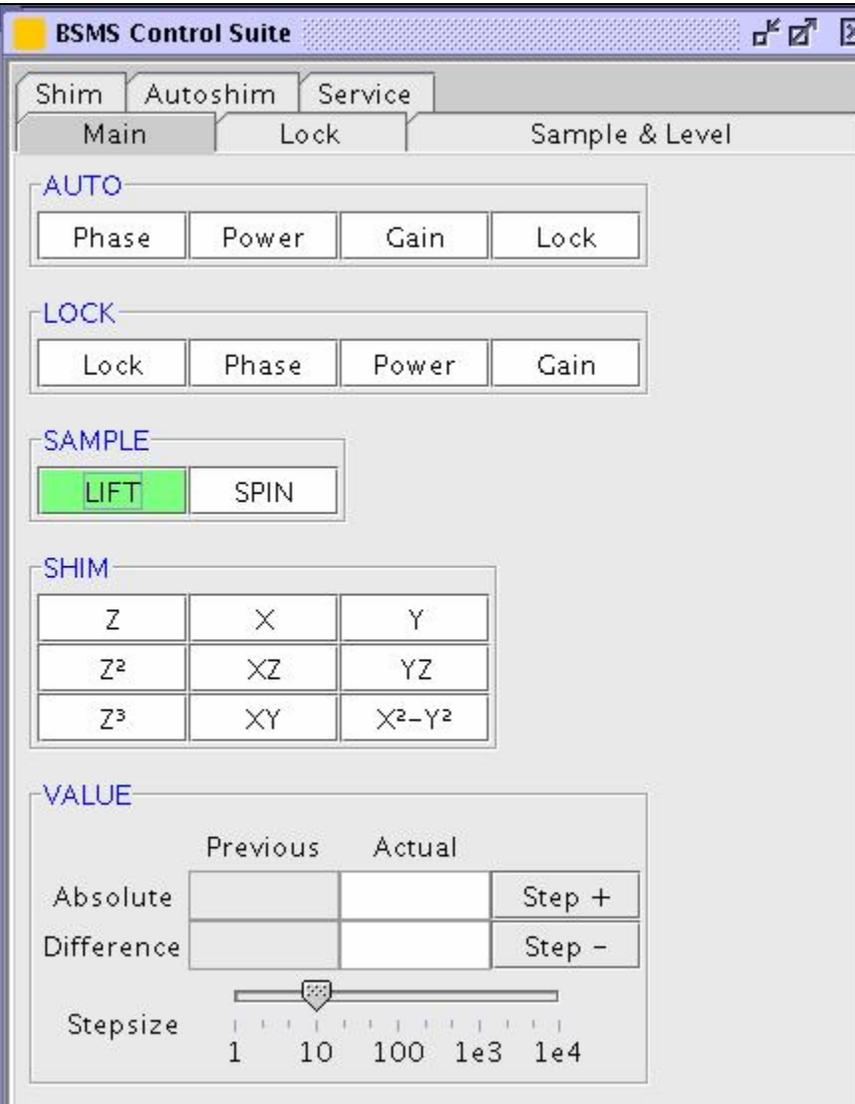
1. Type: **ro off** to turn the spin off.
2. Type **lock off** to turn off the lock.
3. Type **ej** to eject and remove the sample.
4. Type **ij** to turn off the inject gas.

Logging out:

1. Close all Bruker windows (lock display, BSMS display, TOPSPIN, PLOT EDITOR, etc) by double clicking in the top left of each window or by clicking **[File] > [Exit]** then clicking **[OK]**.
2. Click and hold the **RMB** on the workspace, drag down to **[Log Out]** and release, click **[Yes]** to confirm logging out.
3. Sign the log sheet. Please note any problems with the instrument, computer or printer.

Problems?

[Review the FAQ Sheets or the DCIF website](#)



Open BSMS type: **bsmsdisp**

Click **[SPIN]** to turn off.

Click **[LOCK]** to turn off

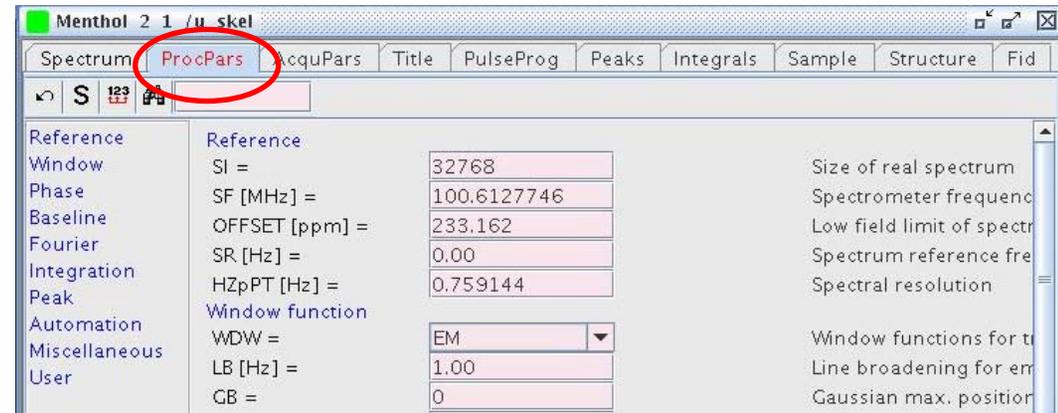
Click **[LIFT]** to eject, and remove the sample

Click **[LIFT]** again turn off the eject gas.

Processing with XWIN-NMR

A list of useful processing commands:

- **lb** line broadening (default for ^1H is 0.3Hz)
- **em** exponential window multiplication
- **ft** 1D Fourier transform
- **si** size of real spectrum should be equal to or 2x greater than **td**
- **pk** phase correction according to user defined values
- **apk** automatic phase correction
- **apks** automatic phase correction (different algorithm)
- **abs** automatic baseline correction
- **efp** em + ft + pk

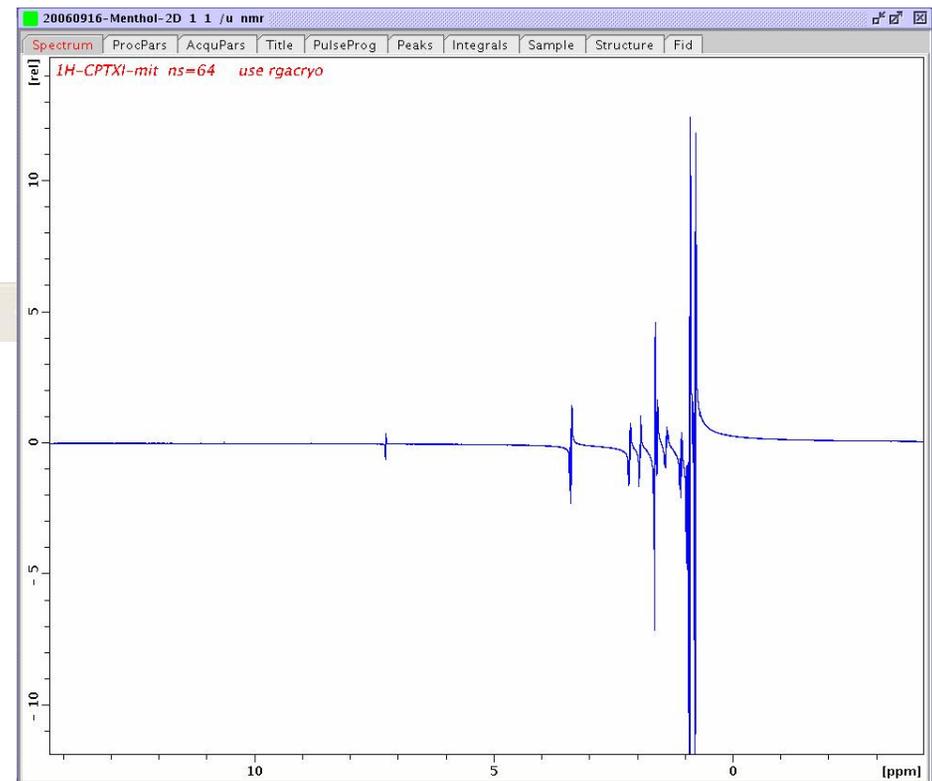


Start processing the spectrum as follows:

1. Type **ft**. This will Fourier transform the spectrum.
2. The vertical scale can be adjusted with the **[*2]**, **[/2]**, **[*8]**, and **[/8]** buttons.



3. Type **apk** or **apks** (the choice is yours) to automatically phase the spectrum. To Fourier transform data with the current phase correction type **fp**. The command **efp** will use an exponential multiplier for force the FID to zero, Fourier transform and phase using the current phasing values.



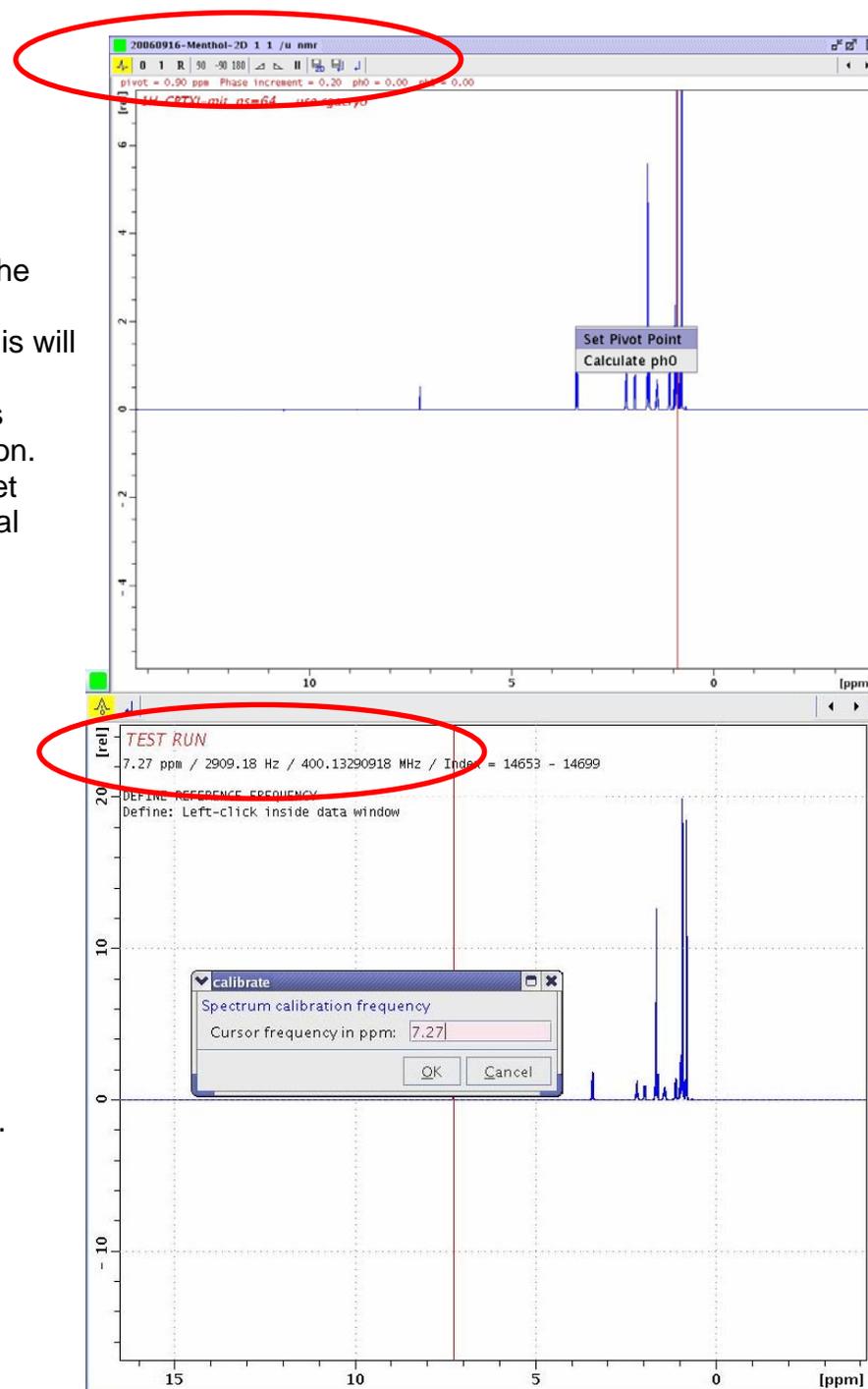
4. If automatic phasing is not satisfactory you want to manually phase the spectrum. To manually phase the spectrum do the following:

- Click  for phase correction, the phase menu open at the top of the dataset. Click the **RMB** on a peak to define the pivot point.
- Now hold the **LMB** down on the **[0]** button and move the mouse. This will adjust the phasing in the vicinity of the red vertical red line.
- Next, hold the **LMB** down on **[1]** and adjust the phasing of the peaks farthest away from the red line. This is the first order phase correction. Note that it may take several iterations of adjusting **[1]** and **[0]** to get your spectrum phased. Click **[R]** to reset the phase back to its original
- Click the  save & return button to save the phasing.

5. Baseline correct, type **abs** (automatic baseline subtraction).

6. Calibrate the spectrum:

- Click the  button or type **cal** or **[Analysis] > [Axis Calibration]**
- Move the arrow to the top of the reference peak, click the **LMB**, and assign or correct the reference value.
 - Residual protonated solvent is often used as the calibration standard, if the chemical shift is not temperature dependent.
 - TMS or TMSP can also be used.



7. Integrate the spectrum:

a. Expand the region of interest. Drag the **LMB** to create a region.

b. Click the  button or type **int** or **[Analysis] > [Integration]**

c. Click the  define regions manually button.
Follow instructions on screen.

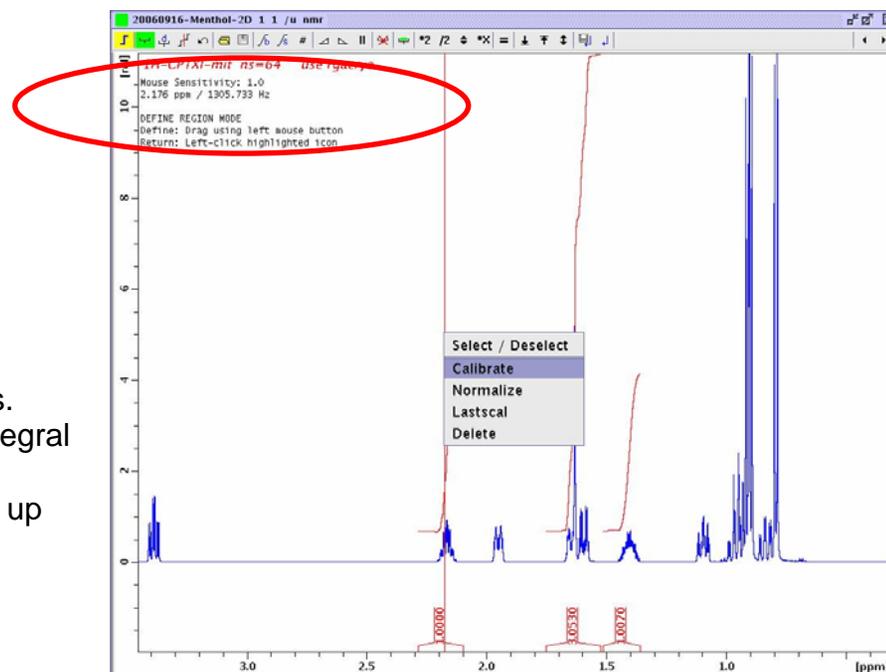
d. Drag the **LMB** over every peak to be integrated.

e. To increase or reduce the integral line size, use the **[*2]** or **[/2]** buttons.

f. To calibrate (or normalize) your integrals, click the **RMB** on a given integral line to mark it.

g. Select **[calibrate]** and enter the desired area of the integral in the pop up window. Note that all other integrals will be scaled accordingly.

h. Click the  save & return button to save the integration.

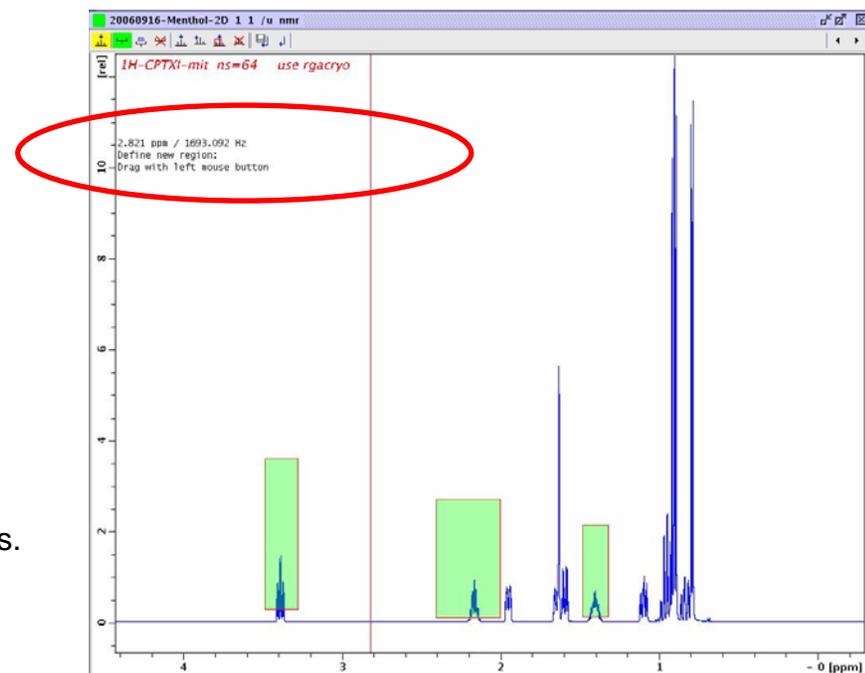


8. Peak Picking:

a. Click the  button or type **pp** or **[Analysis] > [Peak Picking]**
Define regions manually. Follow instructions on screen.

b. Drag the **LMB** over every region to be peak picked.

c. Click the  save & return button to save the peak list.



9. Printing

- prnt** : print active window in TOPSPIN, **RMB > [Display Properties]** to select viewable plot objects
- plot**: start TOPSPIN Plot Editor to create a layout.
- autoplot: print using predefined layout from TOPSPIN Plot Editor

10. **Exporting**: To export the active window, **[File]>[Export]** enter a *filename.ext* where ext is the desired extension. TOPSPIN Plot Editor also exports graphics.