

Bruker DRX-Avance NMR

Bare Bones User's Manual

Revised February 04, 2003

Note: This document covers basic operation of the instrument and data processing. It is not meant to be a comprehensive guide for advanced experiments, nor does it present all optional methods of doing everything. In many cases, there exists more than one way to accomplish a task.

CONTENTS:

- Section I: Setup, sample insertion, lock, shim, acquire data.
- Section II: Moving data to the workstation // Exporting for Processing using Nuts® // Quitting
- Section III: Basic Data Processing using Xwin NMR
- Section IV: Printing/Plotting from within Xwin NMR.

SECTION I: Acquiring your NMR spectrum.

- 1) **SIGN IN** the logbook before you start.
- 2) **<Ctrl><Alt>** to login.
 - a) Type in your login-ID (group) and passwd to begin.
 - b) ****If asked for a password for //phase2, enter "nmr <Return>"****
 - c) XwinNMR will start automatically.
- 3) **Click in the command-line area** and then type "**cpan <Ret>**" to view/use the Command Panel (Buttons). Remember, you can either use the buttons, or use the command-line equivalent in all cases.
 - a) *Note*, if your Button-Panel disappears, you can bring it back to the top by *clicking* on [cpan-default] icon on the taskbar on the bottom of the screen.
- 4) *Click* on the **[Lock Display]** *cpan* button (or...pull-down Windows menu, and select "Lock Display")
- 5) **Press the [Lift On/Off]** button on the BSMS unit to eject the sample, and remove the sample from the magnet.
 - a) *Note: Take care to keep the rotor CLEAN, and clear of Finger Oils!*
 - b) If your experiment is going to require changing or re-tuning the probe (i.e., you need the PFG probe, or the H/F probe, or you need to re-tune the broadband channel to 31P), please seek assistance for special training.
- 6) **Insert the NMR tube** into the rotor, and set the depth as indicated, using the depth gauge.
 - a) **IMPORTANT:** Use ONLY high-quality NMR tubes in the 400, 500 and 600!

	MHz	New Era	Wilmad	Norell
Bruker	600	NE-UL5	541-PP-7 or 535-PP-7	509-UP
Bruker	500	NE-UL5	535-PP-7 or 528-PP-7	509-UP or 508-UP
Bruker	400	NE-HL5	528-PP-8, 527- PP-8, 526-PP-8	508-UP 8 in. or 507- HP 8 in.

- 7) **Insert the sample in the top of the magnet**, then push the **[Lift On/Off]** button to insert the sample. Watch the sample, to ensure that it goes down (sometimes they get stuck at the top).

- a) One-More-Time: Oil from fingers on the tube, and the spinner-rotor can build up and cause serious problems with the instrument. Take EXTREME care to clean the NMR tube and the rotor with a clean, dry Kim-wipe before inserting the sample.
 - b) If you are using a J-Young tube or any high pressure NMR tube, DON'T spin the sample (turn the spinner off).
- 8) **Setting the Solvent and Locking:** If your sample is in CDCl₃, the sample might lock-up automatically.
- a) To Lock, click the [Lock/Solvnt] *cpan* button {command= "lock"} then <Click> on the solvent of choice...instrument should lock-up and set the lock-gain automatically. NOTE, remember the exact name of the solvent that you used because you may need to enter it from the keyboard later.
- 9) You may need to optimize the Lock Phase, and Gain to achieve a stable lock level in the LockDisplay window.
- (1) Press the [Lock Phase] button on the BSMS unit, and maximize the lock level with the knob
 - (a) Press the [Standby] button on the BSMS unit when done.
 - (2) Press the [Lock Gain] button on the BSMS unit, and adjust the lock level to ~80% of the display
 - (a) Press the [Standby] button on the BSMS unit when done.

10) **Shimming:**


- a) Manually Adjust the **Z1** and **Z2** shims using the BSMS buttons and wheel.
 - i) Press the [Standby] button on the BSMS unit when done.
- b) **Click on the [Shim] *cpan* button** (command = tune Z1Z2) for a quick auto-shim.
 - i) A better shim can be obtained by using the command “ tune Z3 ”, which optimized Z1, Z2, and Z3; however, this takes significantly more time.
- c) Watch for the "tune: finished" prompt at the bottom-left corner of the window (or "cmdpanaux: finished" if you used the command panel button). Also, the auto-tune LED on the BSMS unit so be on when the tune/shim is complete.
- d) For more thorough shimming, **type** the command “**tune**”. This brings up a menu of shimming procedures.
 - i) “Z2Z3”, does a simplex optimization of Z1 -Z3, takes quite awhile
 - ii) “Z2Z4”, does a simplex optimization of Z1, Z2, and Z4...takes about as long as i) above.
 - iii) “Full_Shim” does Z1 and Z2, turns the spinner off, optimizes the low-order nonspinning shims, turns the spinner back on, and full optimizes the “Z” shims. This takes up to 15 minutes to perform....not recommended for routine samples.
- e) If you wish, you may **SAVE** your shim values for a particular sample. It is recommended that you use the following convention for naming shim files: *Initials_Probe_Solvent* (Eg. JDoe_BBO_cdcl3).
 - i) Using the example name above, you would **type**: “ wsh JDoe_BBO_cdcl3 <Return> ”.
 - ii) To recall these shim values (or other *standard* shims):
 - (1) Type: “ rsh <Return> ”, then select the shim-file from the menu.
 - (a) Standard Shims include:
 - (i) In most cases you can type “rfshim” to recall the standard shims for the probe
 - (ii) QNP_cdcl3 : (GE broadband (default) probe w/ CDCl₃)
 - (iii) TXI-cdcl3 : (Bruker TXI-gradient probe, CDCl₃)
 - (iv) GE-5HF-cdcl3: (GE H/F probe, CDCl₃)
 - (b) **DO NOT** attempt to overwrite existing, standard shim files with *wsh*!

11) **Create a New File for Acquisition**

- a) **Click on the [NewFile] *cpan* button** (command = new) to define your working file:
 - i) NAME: the filename for your sample (NOTE: Filename limited to ONLY 14 CHARACTERS!)
 - ii) EXPNO: a number for this experiment (any integer from 1-999)
 - iii) PROCNO: a number for the processed data (usually 1)
 - iv) DU: should already be "D:"
 - v) USER: should be your user/group nameTYPE: should always be "nmr"
- b) **Click on [Save] to create the file.** You should see your filename at the top of the XwinNMR window, just below the pull-down menus.
- c) **Setup your experiment:**
 - i) [Setup H1] *cpan* button (command = 1h) for proton observe
 - ii) [Setup C13] *cpan* button (command = 13c) for carbon observe
 - iii) [Setup P31] *cpan* button (command = 31p) for phosphorous-31 observe
 - iv) [Setup F19] *cpan* button (command = 19f) for fluorine-19 observe

NOTE: You will be asked to enter the solvent name after performing any of the steps below



















- 12) *Optional:* **Type** the command “**setti**” to define a plot title for your data.



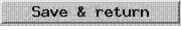
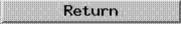
- 13) **Tune the probe** (if necessary) on your sample:
 - a) NOTE: You MUST receive proper training for proper probe tuning procedures. Improper tuning can degrade your spectrum quality (at best) and/or severely damage the instrument (at worst).
 - b) **Click on the [TuneProbe]** *cpan* button (command = wobb) to enter the probe tuning routing
 - c) Adjust the appropriate tuning-stick to center and minimize the reflected power, using the LEDs on the probe-interface module (next to the magnet).
 - d) [Quit Probe Tune] *cpan* button (command = halt) then click on  .
 - 14) **Click the [Set Rcvr Gain]** *cpan* button (command = rga) to automatically adjust the receiver gain.
 - 15) **Click the [Set # Scans]** *cpan* button (command = ns) to set the number of scans desired.
 - 16) **Click the [Start Acq.]** *cpan* button (command = zg) to begin acquiring.
 - 17) You can read the data as it acquires, and either wait for completion of "ns" scans, or stop it yourself.
 - a) **Click** the [Read Data] *cpan* button to read the data while acquiring (command = tr).
 - b) **Click** the [Process FID] *cpan* button to see the spectrum (command = efp).
 - c) **Click** the [AutoPhase] *cpan* button to phase the spectrum (command = apk).
 - d) **Click** the [Stop Acq.] *cpan* button if you wish to stop before reaching "ns" scans (command = halt).
 - i) You must repeat [Process FID] each time you Read Data, Stop Acq., and/or when the experiment reaches completion.
 - 18) If you wish to do another experiment on this sample (i.e. do a 13C after completing a 1H experiment):
 - a) **Click** the [NewFile] button (command = new), and enter either a different NAME or a different EXPNO value.
 - i) *Caution:* failure to change the NAME or EXPNO may overwrite the data you just acquired
 - b) **Click** [Save] to save and exit the panel.
 - 19) If you are done with this sample, and wish to either quit, or start fresh with a new sample:
 - a) **Click** the [Clear/Reset] *cpan* button (command = rsp).
 - i) this will restore your working file to disk, and set the working file to " temp 1 1 "
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Section II. Moving Data to the Workstation // Exporting Nuts Data // Quitting

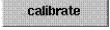
- 1) **Moving Data to the Workstation:**
 - a) On the desktop, there are two folders: "Instrument Data", and "Wrkstation Data".
 - b) <Double_Click> on "Instrument Data" to open the local data folder.
 - c) Select the File (or files) that you wish to process on the workstation, and Drag-and-Drop them from the Instrument Data folder to the "Wrkstation Data" folder.
 - d) The files will be available for processing on the workstation
- 2) **Quitting:**
 - a) Insert the reference sample (CDCl₃) into the instrument, lock, and recall reference shims by typing "rfshim". (re-tune the probe to 1H and 13C of you changed the tuning).
 - b) Copy your files to the workstation (See Section II).
 - c) Exit XwinNMR (File exit, ...or...type the command "exit" on the command line).
 - d) Log-Out of Windows-NT (<Ctrl><Alt> ... [logoff])
 - e) Answer "yes" (or <Return>) to any questions about saving files, ...etc.
 - f) **Sign-Out** in the logbook.

Section III. Processing 1D Data in XwinNMR

- 1) Do [File] [Open >] [1d spectra] to open your file (command = dirs)
- 2) Defining the Default Printer & Plotter:
 - a) **Type** the command “ edo ”, and you will see a dialog box showing the current printer/plotter.
 - b) **Hold** the **LEFT mouse button** over the small “down-arrow” beside the printer name, and **select the printer and plotter** that you wish to use for your printouts.
 - c) **Click** on [Save] to save these values.
- 3) **Window Functions // Fourier Transform**
 - a) The command " efp " performs the following: exponential apodization window, FFT, and phase correction using the most recent phase values.
 - b) Alternative (manual) window function optimization:
 - i) Pull-down the [Process] menu, and select "Manual window adjust" (command = “ winfunc ”).
 - ii) This allows you to optimize the window function, perform interactive resolution-enhancement (via the GM function) visually. For more details, see page 16 of the Data-processing tutorial.
 - (1) If you wish to use the “GM” values, process the data with the “gfp” command (instead of ‘efp’).
- 4) **Displaying the Data**...Zooming in, out , up, and down:
 - a) On the left button-bar, the top section controls the display. Here is a brief description of the buttons.
 - i)  : Increase / Decrease Vertical Scale x 2
 - ii)  : Scale Vertical Size to full-screen for largest peak (may be outside window)
 - iii)  : Increase/ Decrease Vertical Scale x 8
 - iv)  : Increase/ Decrease Vertical Scale manually (hold-down LEFT button, and drag the intensity up/down).
 - v)  : Expand Horizontally by 1/2 screen width.
 - vi)  : Contract Horizontally by 1/2 screen width.
 - vii)  : Expand full spectrum to screen width
 - viii)  : 1. Display complete spectrum, save zoom region 2. Display last zoom region.
 - ix)  : Drag a zoom-box around desired region, *click* the RIGHT button to zoom.
 - x)  : Raise/Lower Baseline (raise to middle, or lower to bottom)
 - xi)  : Move Baseline manually (hold-down LEFT button, and drag the baseline)
 - xii)  : Move/Slide spectrum left/right by 1/2 screen at a time...very useful while integrating, since your mouse buttons are busy defining break-points.
 - xiii)  : Toggle grid-lines and Y-axis labels.
 - xiv)  : Toggle the spectrum axis between Hz and PPM.
 - b) **Zooming –In/Out using the mouse:**
 - i) *Click* the LEFT mouse button anywhere in the spectrum display window, and you will see the pointer appear (the pointer “sticks” to the data-points).
 - ii) *Move* the pointer to one side of the desired region, then *Click* the MIDDLE mouse button (scroll wheel). You will see a solid-pointer-arrow marking this position.
 - iii) *Move* the pointer to the other side of the desired region, then *Click* the MIDDLE mouse button again.
 - iv) You should now see the expanded region on the screen. You can still adjust this region using the buttons described above (section 2a).
- 5) **Phasing the Spectrum:**
 - a) Auto-Phase: *Click* the [AutoPhase] *cpan* button, or type the command “ apk ”.
 - b) Manual Phasing:
 - i) *Note:* it is easier to *zoom-in* on the region of interest before initializing the phase-panel.
 - ii)  : *Click* on the phase button to enter the phase-correction panel.
 - iii)  : *Click* on this button to set the “pivot point” on the tallest peak.
 - (1)  : this button allows you to set the “pivot point” manually with the cursor.
 - iv)  : *Hold* the LEFT mouse button over this icon, and adjust the phase at the “pivot-point”.

- v)  : Hold the LEFT mouse button over this icon, and adjust the phase of the peaks furthest from the “pivot point”.
- vi)  : Click on the return button when done phasing
- (1)  : Click this button to save your phasing values and exit phasing.
- (2)  : Click this button to abort phasing and exit phasing.
- (a) Note: the changes to the phasing will be lost if you exit with [return]














6) Setting the Shift Reference (“Calibrating” the Spectrum):

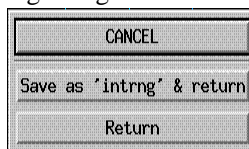
- a) First, *zoom-in* on your reference peak.
- b)  : Click on [calibrate]
- c) Move the pointer to the top of the peak (note, the pointer is tied to the data-points).
- d) Click the MIDDLE mouse button (scroll wheel), and a window will pop-up.
- e) Enter the correct chemical shift for this peak (in ppm), then press <Return>.

7) Baseline Correction:



- a) Type the command “ abs ”. This will automatically fit the baseline, and will also define default integral regions. You can change the integration regions manually, as described below.
- i) *Caution:* ‘abs’ will delete any integral regions previously defined!
- b) Manual Baseline Correction: (when the abs command doesn’t quite do the job)
- i) Type the command “basl” to enter the manual baseline correction routine.
- ii) Study the 1D Processing tutorial, Page P-21 for details.

8) Integration:

- a) It is best to *Zoom-In* on a narrow region before starting to integrate.
- b)  : Click on the [integrate] button to begin, Note the panel on the right 
- i) It will work best to use: , , , and  to move around the spectrum because the mouse buttons (in the spectrum window) are used to assign regions.
- c) Click the LEFT mouse button in the window to activate the pointer.
- d) Move the pointer to the left-side of a region to be integrated...
- e) Click the MIDDLE mouse button...this sets the beginning of the region.
- f) Move the pointer to the right-side of the region
- g) Click the MIDDLE mouse button again.
- i) REPEAT steps d, e, f, & g for all regions.
- ii) Again, you can use: , , , and  to move around within the spectrum.
- h) To *calibrate* a given integral, *point* to it and *DoubleClick* the LEFT button on it. It will be labeled with an asterisk (*). Then Click on the  button. A dialog box will appear for entering the desired integral value.
- i) You may also level any region by selecting it (*DoubleClick*), and then *HOLD* the LEFT mouse button over . Use [bias] to level the beginning (left) part, and [slope] to level the ending (right) part of the region. *NOTE: be careful not to misuse this option to alter the data improperly.*
- j) Click on  when you are satisfied with your integral regions.
- i) You will see:
- ii) Click on [Save as ‘intrng’ & return] to save your work, and allow plotting with integrals
- iii) Click on [Return] to exit integration without saving your changes (all regions will be lost)
- k) *Caution:* Other commands (i.e. “abs”) automatically overwrite the ‘intrng’ file, and can cause loss of your integral regions.
- l) Selecting the integral options for the hardcopy is described in section 8 (plotting).
- m) For a more detailed description of the integration tools, see Page P-24 in the 1D Processing Tutorial.



9) Peak Picking:

- a) *Zoom-In* on the region that you wish to Peak-Pick.
- b) Click on  (define plot region), and answer all prompts with <Return>.
- i) Note: this sets the active plot region. You can always return to this region by clicking on .
- c) Type the command: “ pscal ” and select the option for vertical scaling:
- i) *global* uses the entire spectrum, scaling the intensity to the largest peak.

- d) Click on and you will see the utilities panel on the left.
 - i) Click the LEFT mouse button on and drag the line so that it crossed all peaks of interest.
 - ii) Click the LEFT mouse button again to set the minimum intensity for peak picking.
 - iii) Click on to exit the utilities panel.
- e) Type “ pps ” on the command line, and a list of peaks will appear.
 - i) You can click on [Print] to print the list, or click on [OK] to clear the peak display.
- f) Selecting the peak-pick options for the hardcopy is described in section IV (plotting).

Section IV: Plotting Your Spectrum from within XwinNMR:

Note: This section describes plotting within XwinNMR. Another program, XwinPLOT, is available to create WSYIWYG output. XwinPLOT will be described in a separate tutorial, and once learned is usually the preferred method of plotting spectra.

- 1) **Zoom-in** on the region you wish to plot.
- 2) **Click** on (define plot region), and answer all prompts with <Return>.
 - a) *Note:* this sets the active plot region. You can always return to this region by clicking on .
- 3) **Type** the command: “ pscal ” and select the option for vertical scaling:
 - a) **global** uses the entire spectrum , scaling the intensity to the largest peak.
 - b) **preg** uses the plot region just defined in step 2...the largest peak in the plot region is set to full-scale.
- 4) **Type** the command “ edg ” (edit graph) and you will see the panel below, which allows you to select what will appear on your plot:
 - a) Most of the options are self-explanatory :
 - b) Toggle options on/off by clicking on the corresponding button.
 - c) Buttons like [edplabl] or [edinteg] will bring up a list of options (i.e. colors).
 - d) Click on [SAVE] to save your selections.
- 5) **Type “ view ”** on the command line to see how the plot will look.
 - a) Click on [Quit] to clear the view screen.
- 6) **Click** on to plot the spectrum.
- 7) You can *Zoom-in/out* to select a new region, then Click on to define a new plot region, and then repeat step 4) above.
- 8) **Caution:** Be sure to execute “rsp” (or “ re temp 1 1 ”) when you are done. Otherwise, someone else may accidentally operate on your spectrum, and damage your data!

