

**Operating
the
Bruker Avance DRX-400
NMR**

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Revised September 7, 2006

INTRODUCTION

The procedures for the recording and processing of a routine proton or carbon NMR spectrum using a standard set of parameters are described here. The topics covered are:

- Starting the NMR Program
- Setting Up an Experiment
- Loading, Locking and Shimming a Sample
- Data Acquisition
- Data Processing
- Expanding the Spectrum
- The XWinNMR Toolbar
- Referencing the Spectrum
- Integration
- Plotting
- Exiting the program
- XWinNMR Parameters
- XWinNMR Commands
- Probe Calibration Results

STATUS BAR

The status bar at the bottom of the screen displays both a short description of the menu item highlighted and the status of a the current command.

HELP

There is help available by choosing Help from the main menu and selecting either Contents or Index.

STARTING THE NMR PROGRAM

Starting XWinNMR.

From the desktop, single click the XWinNMR icon.

SETTING UP AN EXPERIMENT

Open a new file.

NEW (or EDC)	Create a new data set for your sample by typing <u>n</u> ew (or <u>e</u> dc) at the XWinNMR command prompt. This function can also be accessed by choosing <u>F</u> ile and then <u>N</u> ew... from the main menu bar.
NAME name	Enter a name for your data set. You can save several NMR experiments (^1H , ^{13}C etc) for one compound under one name.
EXPNO 1	Enter an experiment number for your data set. This is a number between 1 and 998. You might use expno 1 for ^1H , 2 (or 13) for ^{13}C etc..
PROCNO 1	Enter a processing number for your data set (usually 1). You can process your data several different ways and compare the results.
DU /opt/xwinnmr	The disk unit must always be <u>/opt/xwinnmr!!!</u> .
USER username	Enter a username. This must be your user name (the name you used to log on with).
TYPE	The type must always be <u>nmr!!!</u>
[SAVE]	Save the data set.

Read a standard parameter file.

RPAR	Read in a standard parameter file by typing rpar at the XWinNMR command prompt. Pick "+proton" (or "+carbon") from the list. Click the [Copy All] button to read in the parameters. The computer alphabetizes the "+" sign before the letters, so that the standard files are at the top of the list. Note that parameter files also contain an entry for the solvent, and reading a parameter file after locking the sample may replace the solvent parameter with an incorrect value.
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Editing acquisition parameters.

ASED (or EDA)

Edit the acquisition parameters. A listing of a few of the parameters and their meaning is shown below. A more complete listing is included at the end of this document. Note that the auto setup editor, ased, displays only the acquisition parameters relevant to your experiment, while the edit acquisition parameters editor, eda, displays all of the acquisition parameters. Both editors are useful.

PULPROG	zg	PULse PROGram (experiment)
TD	32768	number of Time Domain samples (real+imaginary)
NS	16	Number of Scans
SWH	8000	Sweep Width in Hz
SW	20	Sweep Width in ppm
D[1] (or d1)	3	relaxation Delay

LOADING, LOCKING AND SHIMMING A SAMPLE

Loading the sample.

The following functions are carried out using the BOSS keyboard.

[Lock On/Off] Turn the lock off. Press the [Lock On/Off] button. The light should change from on to off.
 [Lift On/Off] Eject the sample. Press the [Lift On/Off] button. The light should change from off to on).

Replace the current sample with your own. Make sure that your sample spins freely in the "glass bearing" before inserting into the spinner. Carefully set the depth.

[Lift On/Off] Load the sample. Press the [Lift On/Off] button. The light should change from on to off.
 [Spin Rate] Adjust the spin rate. Use the knob to set the desired value (usually 18 rps).

Wait for the sample to spin. The light in the [Spin On/Off] button will blink until the set spinning speed is achieved, then stay on.

Lock the sample.

LOCK Lock your sample by typing lock at the command prompt. Choose a solvent from the list. The [Lock On/Off] light will blink until the sample is locked, then stay on.

Automatic Shimming of the sample.

TUNE QZ1Z2 Shim your sample automatically by typing tune qz1z2 (or tune sh12) at the command prompt. Wait for the “tuning” routine to finish.

Manual Shimming of the sample.

LOCKDISP Open the lock display window by typing lockdisp on the command line. The light on the [FINE] button on the BOSS keyboard should be lit. If it is not, press the [FINE] button to turn it on. To manually shim, press the [Z] button and then turn the knob until the maximum signal is obtained. Repeat for [Z2]. If the signal goes off scale, press the [LOCK GAIN] button and adjust the gain with the knob until the signal is back on scale.

DATA ACQUISITION

RGA Set the receiver gain. This will take a few seconds.
 ZG Start the acquisition.
 ACQU Open the acquisition window to display the FID as it is acquired. A dialog box in the upper right hand corner of the window displays the number of scans acquired so far, and the time remaining.

DATA PROCESSING

LB Set the argument for exponential multiplication.
 EM Multiply the FID by an exponential with an argument of LB.
 FT Fourier transform the spectrum.
 PK Phase this spectrum with a phase correction determined in a previous experiment.
 APK Automatically phase correct the spectrum.

Composite commands	These “macros” execute multiple commands.
EFP	EM + FT + PK.
FP	FT + PK.
ZGA	ACQU + ZG.
[phase]	Clicking the [phase] button in the XWinNMR tool bar will cause the display to change to the phase correction window.

Before phasing the spectrum, you should first define the phase pivot. The phase pivot is the point in the spectrum about which the first order phase correction is applied. The zero order correction is applied equally to the entire spectrum. The first order correction is zero at the phase pivot and increases for points farther and farther away. The [biggest] button will set the phase pivot to the position of the largest peak and attempt to phase the spectrum. The [cursor] button will allow you to define any point in the spectrum as the phase pivot. The phase pivot is marked on the display by a dashed vertical line. Phasing is accomplished by positioning the mouse cursor over the [PH0] button, holding the LEFT mouse button down, and then dragging the mouse. When you are satisfied with the phase correction, release the LEFT mouse button. Repeat for [PH1].

When you are finished, press the [return] button. The pop up window gives you several choices. The cancel button returns you to the phase correction window. The Save & return button applies the phase correction to the spectrum and then returns you to the normal display window. The Save as 2D & return button also saves the phase correction to the most recently displayed 2D spectrum. The Return button returns you to the normal display window without applying the phase correction.

EXPANDING THE SPECTRUM

Clicking the LEFT mouse button inside the spectral display will cause the cursor to change to a vertical arrow pointing down and it will track with the spectrum. As the mouse is moved back and forth, the cursor moves across the spectrum with it. A dialog box also appears in the upper right hand corner of the window and displays information about the cursor position. Clicking the MIDDLE mouse button will anchor a cursor at the current position. The dialog box now shows information about the cursor position with respect to the anchor point. Clicking the MIDDLE mouse button a second time causes the spectrum to be expanded between the two points. To turn the tracking cursor off, click the LEFT mouse button.

THE XWinNMR TOOL BAR



The left hand margin of the XWinNMR window has three columns of buttons that provide additional functionality to the program.



The six buttons of the top two rows provide for vertical scaling. The [*2] [/2] and [*8] [/8] buttons increase or decrease the vertical scaling by 2 or 8. The right button on the top row resets the vertical scale so that the largest peak in the spectrum is displayed full scale. The right button on the second row is used to interactively set the vertical scaling. This is accomplished by placing the cursor over the button, holding down the left mouse button and then dragging the mouse (up and down) to adjust the scale.



The next row of buttons are for horizontal scaling. The left button expands the spectrum. The middle button contracts the spectrum and the right button resets the horizontal scale so that the complete spectrum is displayed.



These buttons shift the spectrum vertically. The left button moves the baseline of the spectrum to the middle of the screen. The middle button moves the baseline of the spectrum to the bottom of the screen. The right button is used to interactively set the vertical shift. This is accomplished by placing the cursor over the button, holding down the left mouse button and then dragging the mouse (up and down) to adjust the scale.



These buttons shift the spectrum horizontally. The left button shifts the spectrum to the left. The middle button shifts the spectrum to the right.



The plot buttons are used to define and display a plot region. The [dp1] button is the most commonly used to define a plot region. The [PlotReg] button forces the program to display the portion of the spectrum defined as the plot region.



These buttons affect the display. The [Y] button toggles the display of the y scale. [YU] toggles the units of the y scale. [dot] displays the spectrum as dots. [Re] and [Im] display the real or imaginary parts of the spectrum. [fid] displays the free induction decay. [Sh] and [Ush] shuffle and un-shuffle the real and imaginary parts of the spectrum.



This button sets the spectrometer frequency and sweep width to match the values of the display.

REFERENCING THE SPECTRUM

[calibrate] Clicking the [calibrate] button will cause the display to change to the calibration window.

Upon entering the calibration window, there will be a cursor (vertical arrow pointing down) tracking the spectrum. As the mouse is moved back and forth, the cursor moves across the spectrum with it. Position the cursor at the reference point and click the MIDDLE mouse button. Type in the value of the reference and then press the Enter key.

INTEGRATION

ABS Apply an automatic baseline correction followed by an automatic integration. To see the results of the integration, type xwinplot, xwp or view.

[integrate] Clicking the [integrate] button will cause the display to change to the manual integration window.

Integration is performed in a manner similar to expanding the spectrum. Click the LEFT mouse button. Move the cursor to the left of the region to be integrated. Click the MIDDLE mouse button. Move the cursor to the right of the region to be integrated. Click the MIDDLE mouse button. The integral will be displayed. Move the cursor to the left of the next region to be integrated ...

When you are finished, click [return]. The pop up window gives you several choices. The CANCEL button returns you to the integration window. The "Save as 'intrng' & return" button, saves the integrations with the spectrum and then returns you to the normal display window. The Return button returns you to the normal display window without applying the integrations.

PLOTTING

SETTI	Set the title for the spectrum. This command starts a text editor. Enter a title to go with your spectrum. The title can have multiple lines, and each line will be centered in the spectral window of the plot.
[dp1]	Define a plot region. You can either enter a chemical shift range to be plotted, or set the range to coincide with the range displayed on the screen by answering the questions with "Enter".
XWP or VIEW File / Print	Display the spectrum as it will be plotted on the paper. Plot the spectrum along with the title, parameters, and integration.
File / Close	Close the xwinplot program.

EXITING THE PROGRAM

IMPORTANT: You must exit XWinNMR before logging out. If you do not, the next user will NOT be able to use the instrument.

[quit]	If the <u>lock display</u> window is open, close it by pressing the [quit] button.
EXIT	Exit XWinNMR. This function can also be accessed by choosing <u>File</u> and then <u>Exit</u> from the main menu bar.
[OK]	Answer "Do you really want to leave the program?" with [OK].
[OK]	If you get the "Process still active" dialog box, (instead of the "Do you really want to leave the program?" dialog box), answer with [OK]. This message usually means that the lock display window is still open.

XWinNMR Parameters	
File Parameters	
NAME	data set NAME
EXPNO	EXPeriment Number (1-998, 999 is used internally)
PROCNO	PROCeSSing Number (1-998)
DU	Disk Unit. (this must always be <u>/opt/xwinnmr</u>)
USER	your USER name (the name you used to login)
TYPE	this must always be <u>nmr!</u>
Note:	The raw data is saved in the directory /DU/data/USER/nmr/NAME/EXPNO
Acquisition Parameters	
TD	number of Time Domain samples (real+imaginary)
NS	Number of Scans (number of acquisitions to be averaged)
DS	number of Dummy Scans
SWH	Sweep Width in Hz
SW	Sweep Width in ppm
AQ	AcQuisition time (calculated from TD and SW)
D[1] (or d1)	Delay 1 (usually the relaxation delay)
P[1] (or p1)	Pulse 1 (usually the length of the transmitter pulse)
FIDRES	temporal resolution of the FID
DW	DWell time (sampling interval, 1/SW or 1/2SW)
SOLVENT	Solvent
O1P	Offset 1 from the base frequency in Ppm (center of the spectrum)
PULPROG	PULse PROGram (experiment)
PL[1] (or pl1)	Power Level 1 (attenuation, larger #'s => lower power)

XWinNMR Parameters	
Processing Parameters	
SI	Size of the data in the frequency domain (usually TD/2)
LB	Line Broadening (used by EM, usually 0.1 for ^1H)
GB	Gaussian Broadening (used by GM)
TM1	Trapezoid point 1 [range 0.0 - 1.0, usually 0.0] (used by TM)
TM2	Trapezoid point 2 [range 0.0 - 1.0, usually 0.1, {1.0 - TM2 used}]
PHC0	Zero order phase correction in degrees
PHC1	First order phase correction in degrees

XWinNMR Commands	
File Commands	
NEW	create a NEW data set (EDC also works)
Lock Commands	
LOCK solvent	LOCK the sample using parameters for solvent specified
LOCKDISP	LOCK DISPlay (displays lock signal as a function of time)
RSH	Read in a SHim file.
Acquisition Commands	
EDA	EDit Acquisition parameters (displays all acquisition parameters)
ASED	Auto Setup Editor (displays only the acquisition parameters used for this experiment)
RGA	automatically adjust the Receiver GAin
ZG	Zero Go, start accumulation
ACQU	display the ACQUisition window (shows data as it is collected)
TR	TRansfer data from the spectrometer to the computer

XWinNMR Commands	
EXPT	estimates the amount of EXPeriment Time
EDHEAD	EDit the information for the probe HEAD
Processing Commands	
TM	Trapezoid Multiplication (uses TM1, TM2)
EM	Exponential Multiplication (uses LB)
GM	Gaussian Multiplication (uses GB)
WINFUNC	interactively adjust WINdow FUNCtion parameters (LB, GB, etc.)
FT	Fourier transform the data
PK	Phase the spectrum using previous values of PH0, PH1
APK	Automatically Phase the spectrum
FP	same as Ft + Pk
EFP	same as Em + Ft + Pk
EDP	EDit Processing parameters
Analysis Commands	
SREF	automatically Set the REFerence
ABS	Automatic Baseline Subtraction (and integration)
Plot Commands	
EDG	EDit Plot parameters
SETTI	SET (edit) the TItle of the spectrum
XWINPLOT	Starts the xwinplot program.
Macros	
VIEW	Executes the xwinplot command.
ZGA	Executes the commands ACQU + ZG.

PROBE CALIBRATION RESULTS

Probe	Nucleus	90° PW (μ Sec)	Power Level (dB)	90° Dec (μ Sec)	Power Level (dB)
5 mm QNP $^1\text{H}/^{13}\text{C}/^{31}\text{P}/^{19}\text{F}$ Z-gradient (#07)	^1H	11.3	-6.0	100.0	16.0
	^{13}C	6.0	-6.0	60.0	15.0
	^{31}P	5.6	-3.0		
	^{19}F	14.5	-6.0		
5 mm Multinuclear (#09)	^1H	14.2	0.0	100.0	18.5
	^{13}C	9.3	0.0		
	^{31}P	6.9	0.0		
	^{15}N	11.9	-6.0		
5 mm Multinuclear Inverse XYZ-gradient (#11)	^1H	7.4	-3.0		
	^{13}C	14.0	-3.0	100.0	14.0
	^{31}P				
	^{15}N	(25.5)	-3.0		

PARAMETERS FOR COMMON 1D EXPERIMENTS

Experiment	Setting
Homonuclear Decoupling (zgpd)	<p>pl24 = 40 - 50 dB DIGMOD = homodecoupling-digital Set O2 for frequency of peak to be decoupled Set NUC2 to ^1H (edasp)</p>
Presaturation (zgpr)	<p>pl9 = 40 - 50 dB d1 = 2.0 sec Set O1 for frequency of the solvent peak</p>