

# Instructions for use of the Bruker AVANCE 300

If not logged in, log in using the username “nmr” and the password “nmr”.

If TOPSPIN is not already open, then open it by double clicking the “TOPSPIN” icon on the desktop.

## Running 1D NMR spectra

If a set of buttons do not appear on the right hand side of the screen, then click “EZ-NMR” on the top menu bar and then again on EZ-NMR in the submenu. A series of buttons will appear on the right hand side of the screen.

Eject the standard sample by pressing the “EJECT” button on the right hand side of the screen. Wait for the sample to come to the top of the magnet. You should hear rushing air. Carefully remove the standard sample from the blue spinner and replace it with your sample. Set the correct depth with the depth gauge by placing the spinner on top of the depth gauge and gently pushing the NMR tube down until it touches the bottom. While the air is still rushing from the top of the magnet, place your sample at the top of the magnet. It should be supported by the rushing air.

To insert your sample into the magnet, press the “INSERT” button on the right hand side of the EZNMR button panel. The sample should be lowered slowly into the magnet. You should hear a click as the sample is engaged in the probe.

Lock your sample by clicking the “LOCK” button. A list of deuterium labelled solvents will be presented. Click the solvent you are using followed by the “OK” button. Wait for the beep and message informing you that the lock procedure is finished (approx 20 seconds). Click the “CLOSE” button to accept this message.

Shim the magnet (i.e. optimize the homogeneity) by clicking the “SHIM” button. Wait for the beep and the message indicating that the shimming process has finished (approx 2 minutes). Click the “CLOSE” button to accept this message. NOTE: DO NOT PRESS THE “SHIM” BUTTON IF THE SAMPLE IS NOT LOCKED.

Set up the dataset for the type of NMR data you wish to collect by pushing the appropriate button.

“1H”	$^1\text{H}$ NMR spectrum
“1H [31P]”	$^1\text{H}$ NMR spectrum with $^{31}\text{P}$ decoupling
“13C [1H]”	$^{13}\text{C}$ spectrum with proton decoupling
“13C”	$^{13}\text{C}$ spectrum without proton decoupling
“DEPT135”	$^{13}\text{C}$ (CH and CH3 up, CH2 down and no quaternary C)

"DEPT90"      $^{13}\text{C}$  (CH up, no CH<sub>2</sub>, CH<sub>3</sub> or quaternary C)  
"31P"          $^{31}\text{P}$  without proton decoupling  
"31P [1H]"    $^{31}\text{P}$  with proton decoupling

You will be prompted for a dataset name. Do not use spaces. As everyone's data will be kept in one directory, it is a good idea to name your datasets systematically beginning with you initials and then an underscore followed by a description of your sample. (eg. gf\_s3425). Using common names such as "sample1" will almost certainly mean that your data will be overwritten by another user. Once you have entered the dataset name, press the "OK" button. You will then be prompted for an experiment number. Choose a number between 1 and 999 then click the "OK" button. An experiment is a subdirectory of the dataset name into which NMR data can be collected. Any dataset can have as many as 999 different experiment numbers each containing different NMR data. (eg. the data set dave\_product can have a  $^1\text{H}$  spectrum in experiment 1, a  $^{13}\text{C}$  spectrum in experiment 2 and a COSY spectrum in experiment 3 etc...) If you enter a dataset name and experiment number which have been used previously, they will be overwritten. Once you have entered the dataset name and the experiment number, wait for the beep and the message saying that the dataset is set up and ready. Click the "CLOSE" button to accept this message.

If you wish, you can change the number of scans to be collected from the default value by clicking the "SCANS" button. After the button is pushed you will be prompted to input the number of scans you wish to collect. Input the number of scans desired and click the OK button.

If you wish, you can change any of the parameters used to collect the data clicking the "PARAMETERS" button. After the button is pushed you will be presented with a list of the parameters and the current values. You can change these as you see fit. After editing the parameters, return to the graphics display by clicking the "Spectrum" tab on the top of the window. Editing parameters is only necessary if you do not wish to use the default parameters.

If you wish you may edit the title that will appear on your spectrum by pressing the "EDIT TITLE" button. A text window will appear where you can edit the current title. When finished editing the title, return to the graphics display by clicking the "Spectrum" tab on the top of the window.

To start the acquisition, click the "START" button. The spectrometer will find an appropriate receiver gain to use and then start collecting data. When the acquisition is complete, the data will be displayed on the screen and a window will appear informing you that the data collection is finished. Click the "CLOSE" button to accept this message. Your data has been stored on the hard disk of the spectrometer and sent to the NMR data server. It is now available for download from the server using an external workstation.

At any time while the acquisition is running you may look at the spectrum without interrupting the acquisition by clicking the "PROCESS 1D" button. The spectrometer will wait for the next

scan to be completed and then process the data and display it on the screen. The acquisition will continue to run.

If you wish to stop your data collection before it has completed, press the "HALT" button. The data collected up until the point when the acquisition was halted will be processed and displayed and a window will appear informing you that the data collection is finished. Click the "CLOSE" button to accept this message. Even though your acquisition did not finish, the data have been stored on the hard disk of the spectrometer and the NMR server. The data are available for download from the server using an external workstation.

You can set up a series of experiments to run over an extended period of time by clicking on the "DO ALL" button. A dialogue will begin. After you answer all of the questions, the data collection will begin.

## Running 2D NMR spectra

On the top menu bar, click "EZ-NMR" and then "EZ-2DNMR" in the submenu. A series of buttons will appear on the right hand side of the screen.

With the 1D spectrum being displayed, set up the dataset for the type of 2D NMR data you wish to collect by pushing the appropriate button.

"1H COSY"	2D $^1\text{H}$ correlation spectrum via J coupling
"1H 13C HMQC"	2D $^1\text{H}$ - $^{13}\text{C}$ one bond correlation spectrum
"1H 13C HMBC"	2D $^1\text{H}$ - $^{13}\text{C}$ multiple bond correlation spectrum
"1H NOESY"	2D $^1\text{H}$ correlation spectrum via nuclear Overhauser effects
"1H EXSY"	2D $^1\text{H}$ correlation spectrum via chemical exchange
"1H TOCSY"	2D $^1\text{H}$ TOCSY spectrum
"31P EXSY"	2D $^{31}\text{P}$ correlation spectrum via chemical exchange
"1H 31P HMQC"	2D $^1\text{H}$ - $^{31}\text{P}$ correlation spectrum

You will be prompted for a dataset name and experiment number. It is conventional to use the next available experiment number of the same dataset. Once you have entered the dataset name and the experiment number, wait for the beep and the message saying that the dataset is set up and ready. Click the "CLOSE" button to accept this message.

If you wish, you can change the number of scans to be collected from the default value by clicking the "SCANS" button. After the button is pushed you will be prompted to input the number of scans you wish to collect. Input the number of scans desired and click the OK button.

If you wish, you can change any of the parameters used to collect the data clicking the "PARAMETERS" button. After the button is pushed you will be presented with a list of the parameters and the current values. You can change these as you see fit. After editing the

parameters, return to the graphics display by clicking the “Spectrum” tab on the top of the window. Editing parameters is only necessary if you do not wish to use the default parameters.

If you wish you may edit the title that will appear on your spectrum by pressing the “EDIT TITLE” button. A text window will appear where you can edit the current title. When finished editing the title, return to the graphics display by clicking the “Spectrum” tab on the top of the window.

To start the acquisition, click the “START” button. The spectrometer will find an appropriate receiver gain to use and then start collecting data. When the acquisition is complete, the data will be displayed on the screen and a window will appear informing you that the data collection is finished. Click the “CLOSE” button to accept this message. Your data has been stored on the hard disk of the spectrometer and sent to the NMR data server. It is now available for download from the server using an external workstation.

At any time while the acquisition is running you may look at the spectrum without interrupting the acquisition by clicking the “PROCESS 2D” button. The data collected will be processed and displayed. The acquisition will continue to run.

If you wish to stop your data collection before it has completed, press the “HALT” button. The data collected up until the point when the acquisition was halted will be processed and displayed and a window will appear informing you that the data collection is finished. Click the “CLOSE” button to accept this message. Even though your acquisition did not finish, the data have been stored on the hard disk of the spectrometer and the NMR server. The data are available for download from the server using an external workstation.

## **Recovering Your Data on an External Workstation**

1. Double click the WS-FTP-LE icon on your workstation. If WSFTP is not installed on your Workstation, install it from the university’s web site.

2. Verify that the following is correct:

Host Name / Address:	nmr-data.science.uottawa.ca
Host Type:	Automatic Detect
User ID:	nmr
Password:	nmr (this will appear as ***)

3. Under the “Advanced” tab, ensure that “passive transfers” is unchecked.

4. Under the “Startup” tab, set the “Initial Local Folder” (i.e. where you want to put the data on your computer). Set the “Initial Remote Site Folder” to /NMRDATA/AV300/data/guest/nmr/.

5. Click the “connect” button.

6. You should see two main panels. The one on the left is a directory listing for the local computer and the one on the right is the data directory on the nmr server. Single click on the dataset you wish to get from the server (you can select multiple datasets by holding down the Ctrl button while single clicking datasets). Once you have selected your dataset(s) click on the ← button near the center of the screen. Wait for your data to be transferred.

7. When the data transfer is complete, click “Close” then “Exit” to exit the FTP program.

8. Process or print the data using TOPSPIN, ACD NMR Processor or any other program of your choice.