

Instructions for use of the Bruker AVANCE 300 (Undergraduate Laboratory)

If not logged in, log in using the username “chem4380” and the password “nmr”.

Open ‘TOPSPIN’ by double clicking the “TOPSPIN 1.3” icon.

Running 1D NMR spectra

On the top menu bar click on “EZ-NMR” 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. 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. 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.

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

“ ¹ H”	¹ H NMR spectrum
“ ¹³ C[¹ H]”	¹³ C spectrum with proton decoupling
“DEPT135”	¹³ C (CH and CH ₃ up, CH ₂ down and no quaternary C)
“DEPT90”	¹³ C (CH up, no CH ₂ , CH ₃ or quaternary C)
“ ³¹ P”	³¹ P without proton decoupling
“ ³¹ P[¹ H]”	³¹ P with proton decoupling
“ ¹⁹ F”	¹⁹ F without proton decoupling
“ ¹⁹ F[¹ H]”	¹⁹ F with proton decoupling

You will be prompted for a dataset name. Please provide a name with no more than 12 characters. Do not use spaces. (eg. dave_product) 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 ^1H spectrum in experiment 1, a ^{13}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.

Tune the probe by clicking the "Tune Probe" button. This needs to be done only if the last person was running a different nucleus or was using a different solvent. If you are not sure, about the last sample then you should tune the probe. Please wait for the beep and message informing you that the probe tuning has been completed (approx. 2 minutes). 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 "Change Number of 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.

To start the acquisition click the "START ACQUISITION" 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 is now available for download using an external workstation.

If you wish to stop your data collection before it has completed, press the "HALT ACQUISITION" 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 collected data is available for download using an external workstation.

Running 2D NMR spectra

Run a 1D spectrum as described above or import a spectrum if one has already been acquired. (For example, if a proton spectrum has previously been run in experiment number 1 of a dataset called "dave_product", you can type the command *re dave_product 1* to view the data.)

Before running a COSY, HMQC, HMBC, ^1H NOESY or ^1H EXSY you must be viewing a ^1H spectrum. Before running a ^{31}P EXSY you must be viewing a ^{31}P spectrum.

On the top menu bar click on "EZ-NMR" and then again on 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.

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

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.

Tune the probe by clicking the "Tune Probe" button. This needs to be done only if the last person was running a different nucleus or was using a different solvent. If you are not sure, about the last sample then you should tune the probe. Please wait for the beep and message informing you that the probe tuning has been completed (approx. 2 minutes). Click the "CLOSE" button to accept this message.

If you wish, you can change the default parameters by clicking the "Change Parameters" button. After the button is pushed you will be presented with a list of parameters, any of which can be edited. 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.

To start the acquisition click the "START ACQUISITION" 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 is now available for download using an external workstation.

If you wish to stop your data collection before it has completed, press the "HALT ACQUISITION" 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 collected data is available for download using an external workstation.

Recovering Your Data on the Workstations in Marion 313

1. If you are not logged in on one of the workstations, log on with the user name "nmr" and password "nmr".
2. Double click the WS-FTP-LE icon.
3. Verify that the following is correct:

Host Name / Address:	137.122.190.195
Host Type:	Automatic Detect
User ID:	chem4380
Password:	nmr (this will appear as ***)
4. Click the "connect" button
5. You should see two main panels. The one on the left is a directory listing for the local computer (c:/data/guest/nmr) and the one on the right is the data directory on the nmr spectrometer (/data/chem4380/nmr). Single click on the dataset you wish to get from the spectrometer (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.
6. When the data transfer is complete, click "Close" then "Exit" to exit the FTP program.
7. View process or print the data using TOPSPIN.