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Appendix VII: Processing NMR spectra.

All raw (automated) nmr spectra should be presented in your reports. Where appropriate, data must be reprocessed to obtain accurate peak positions, coupling constants, integration and display appropriate expansions. In which case, also include this reprocessed output in your report.

All spectra will be run on the **undergraduate 300 MHz NMR** in Ell 346. The files for each spectrum are automatically transferred to a university directory. You can access them from the drop-in access computers in Student Computing Facilities (SCF) such as those in Clearihue, Business & Economics and HSD, plus some select ones in the Library. You can book a reservation for physical access to these computers, or just drop-in.

Note that you can also use *Mercury*, *Word*, *Excel* etc by the same method. The advantages include not needing to download the various programs yourself and if you are a Mac user, you will not have to worry about the windows-based programs being incompatible.

As NMR spectra are run, the data is automatically transferred to the drive \\chemlabs.storage.uvic.ca\chemlabs\UGNMR\data\ugnmruser\nmr\ that is accessed directly by ACD on the two processing computers in Ell 346. If you are processing elsewhere, such as from home or through SCF, you must wait for the data to be manually copied (the previous day's files are transferred each weekday morning) to another drive \\labs.storage.uvic.ca\labs\CHEM\NMR\ .

Processing in the NMR lab:

Log in to the computer with your Netlink ID and password. Check that the default printer is set correctly before opening the ACD program. Always log out once you are done!

Processing in or through the Student Computing Facilities on Campus:

In the SCF, ACD is already installed on the Windows computers. Open the program and **import** your data from \\labs.storage.uvic.ca\labs\ . This drive is already mapped (L:). Go to CHEM then the NMR folder and find your date code and spectrum experiment number that corresponds to the information on the top right hand corner of your raw printed output.
Always remember to logout when you are finished on the computer.

Processing on your own computer:

You need to have the processing software installed. This can be ACD, Spinworks, TopSpin or other programs that will process the data. See below.

You then need to access the data files, and the method of access depends on your location. You can access the data from off campus by using Virtual Private Network. Go to https://www.uvic.ca/systems/services/internettelephone/remoteaccess/ and scroll to the bottom of the page. A link for the installation of the VPN client for Mac and Windows is in the Related Support window. Follow that link, and follow the instructions on that page.

You can map the data storage drive by right clicking on "This PC" or "This computer". Then click on "Map Network Drive . . .". Choose a letter for your drive (e.g. N: for NMR) and type in the drive address \\chemlabs.storage.uvic.ca\chemlabs\UGNMR\data\ugnmruser\nmr.

Sign into the drive using either "UVic\Netlink ID" or your "Netlink ID@uvic.ca", and then your Netlink password. You can then either download or process the files from that directory.

You should disconnect the VPN after you are finished transferring your data, but it will timeout on its own.

Note: If you want to transfer your data to your Mac use:
Finder > Go > Connect to Server …
Type in the address smb://labs.storage.uvic.ca/labs/CHEM
Authenticate with UVIC\netlinkid or netlinkid@uvic.ca

Processing Software:
These are the programs that have been most commonly used in our laboratories for analysis of NMR spectra.

ACD Labs NMR Processor must be downloaded from our NMR data file storage area (not directly from ACD). Look for the 'ACD download' in the folder marked 'all except current date codes'.

SpinWorks ftp://davinci.chem.umanitoba.ca/pub/marat/SpinWorks/. Windows only.

The three links below are more recent, work on Macs but may not be free.
Bruker TopSpin, now free for academic use.
https://www.bruker.com/service/support-upgrades/software-downloads/nmr/free-topspin-processing/free-topspin-download.html Windows or Mac. You need to register for the site.
NMRnotebook by NMRTEC. http://www.nmrtec.com/software/nmrnotebook/presentation/
Windows or Mac. 30 day trial, may be free for academic use.
MNova by Mestrelab Research. http://mestrelab.com/software/mnova/ Windows or Mac, the "lite" version may be free for academic use, we haven't checked.

Processing spectra from the research 300 MHz NMR Spectrometer
Occasionally, we need to run spectra on the research 300 MHz NMR. We will transfer the data to the same folder. The raw output can be distinguished by looking down the column of parameters on the right hand side in line 7 for PROBHD. The research instrument will read PABBO whereas the undergrad instrument reads QNP. An easier distinction is that the undergrad nmr uses a alpha-style date code (eg Sep21-2021) and the research nmr uses a digital version (eg 09212021). SpecView, Spinworks or ACD/1D NMR Processor will allow the processed spectrum file to be viewed, expanded and peak-picked in the normal way. To access, click through the sub-folders under *date code/spectrum number* (found on the top right hand side of the printout) until you reach the 1r file.

Instructions for ACD 1D NMR Processor software:
*Opening the file on campus in a student computing facility***:**
Login using your netlink ID and password.. Choose 'ACD Labs 12.0' from the alphabetical menu

then '1D NMR Processor' to open the software. Choose '1D NMR Processor' from the Sciences tiles.

1. *Close the Processor Window(s) that may automatically open when the program is started.*
   Click on 'file' and then 'import' (NOT 'open') from 1D NMR Directory. Look in the mapped L: drive \\labs.storage.uvic.ca\labs\ . You are now linked to the drive that stores the data. Choose CHEM and NMR. Nmr files from the current term are listed by date code (top right hand corner of your spectrum). Files that we have provided for course assignments are collated by course number in the 'all except current date codes' folder.
2. Click on the folder that has the date code shown on your spectrum (ie: Oct06-2018, see 'name' in the top right corner of your original printout). Choose the expt number from the list of folders (ie: #21, below the date code on your original spectrum). Open all subsequent sub-folders until a list of files appears in the left hand window, then double-click the '1r' file. Your spectrum will now appear along with a set of tools for manipulating the data.

Zooming in on the peaks

The magnifying glass icons at the toolbar have a variety of options, but the most useful are likely the 'rectangular zoom' tool to click and drag over a defined area and the 'show whole spectrum' option to zoom back out.

3. Click on 'view' then 'zoom area'. The area of the spectrum selected for expansion is shown in a grey box in this window. This box may now be used to further modify the expansion. Click and drag the edges of the box to change its size. The expansion window changes with the changes in the box. This can be done with both the horizontal and vertical edges of the spectrum. You should zoom in sufficiently to show the multiplicity of the signal(s) and maximize their appearance on the page. This may require a vertical expansion by dragging the top edge of the grey box down to the top of the signal. Your printed expansions of a multiplet should always be larger than observed on your original output.

Peak picking

The peak picking function is selected by clicking on 'peak picking' on the toolbar.

4. Two options for labeling the peaks are now generally used - 'auto label' and 'peak by peak'.
   a) 'auto label' lets the computer pick every peak in the entire window above a minimum level. The minimum level is adjustable from a right hand click or choosing 'peak level' directly from the tool bar. This provides a good starting point for labeling your peaks.
   b) 'peak by peak' can now be used to label any other peaks of interest that the 'auto label' function has missed. Click on 'peak by peak' and move the cursor to the spectrum window to bring up a red vertical cursor when the mouse is over a peak. When the cursor is visible, click to label the peak. To delete a peak label, click again.
5. To change the units of your labeled peaks, choose 'options' from the drop down menu at the top of the page then 'preferences' then the 'peaks' tab. You can label the peaks in

ppm or Hz according to your needs - Hz if coupling constants need to be calculated (ie: ^{1}H nmr spectra) or ppm if only shift values are needed (ie: most ^{13}C spectra).

6. Add a 'table of peaks' from the top toolbar, or select 'view' then 'tables' then 'peaks' from the drop down menu. This table will list all the chosen peaks in ppm and Hz. You now have enough information to calculate both the chemical shift (ppm) and the coupling constants (Hz) of the multiplets in your expansion.
7. When you have finished peak picking, click on the green checkmark on the left side of the peak picking toolbar to exit peak picking mode.

Integration (^{1}H nmr only)

The original output of your ^{1}H nmr spectrum has the integration values already calculated just above the x-axis on your spectrum and the lines identify the range of these integration values. If these values have not been chosen over an appropriate range you will need to re-calculate the integration.

8. Start the integration by choosing the 'integration' tool from the toolbar.
9. Click on 'manual' to bring up a red vertical cursor on the spectrum.
10. Click, drag and release over the multiplet that you want to integrate. When the mouse button is released, the integration appears numerically on the bottom of the spectrum, and as a trace across the peaks. Continue until all the peaks are integrated. To change the relative values of the highlighted integral, insert the desired value in the 'reference' window in the tool bar and press 'enter'. All the values will change in the same relative ratio.
11. When you are satisfied with the look of the integrated spectrum, click on the green checkmark on the left side of the toolbar to exit integration mode.

Printing

12. Once you have an area of expansion with peak picking, tabulation, and integration (if applicable) on the screen, click on the little printer icon in the toolbar (within the chemistry department, or at home) or the 'export to PDF' icon right beside it (on University systems computers) .
13. In the 'text' tab make sure 'document name' and 'comment' are selected.
14. In the 'tables' tab make sure 'table of peaks' is selected in both 'ppm' and 'Hz'.
15. All other printer settings are chosen at your preference.
16. Click on 'ok' to accept the selected options, and then click on 'ok' in the next dialog box to actually print the page. If you were converting to PDF, save into onto your desktop or wherever you wish, then open the .pdf file and print from there.

A useful tip to reduce printing charges (other than processing in Ell346 for free):
In ACD, each data table is printed on a separate page. If you are doing multiple expansions of the same spectrum, select the data table for the first print but then cancel that selection for subsequent expansions.