Chemistry 463

Nmr Spectroscopy in the Undergraduate Laboratory

The Bruker 300 MHz nmr spectrometer is capable of automatically recording and printing a basic proton or carbon spectrum, as well as complex proton, carbon, and 2D experiments. It will be used extensively in the organic laboratory, therefore it is imperative that you learn how to operate the instrument properly. You must have your technique checked by an instructor before operating the instrument on your own.

The following sections describe sample preparation, operation of the spectrometer, use of the two programs for spectral analysis, and guidelines for the presentation of data. You are expected to have read this material, and have a basic understanding of it. If you don’t understand, ask your instructor before you try something!

Expectations

The proton (1H) and carbon (13C) nmr spectra are the primary source of data relating to the structure of your compound. It is therefore required that the preparation of the sample and analysis of the spectrum is done properly in order to obtain a good data set.

The basic printout of the 13C (carbon) spectrum should be adequate for the purposes of the undergraduate laboratory. This printout includes a peak-picking function, so analysis with SpinWorks or ACD NMR Processor is unnecessary unless the signals are poorly dispersed. The one problem to watch for is a very noisy spectrum. If the noise on the baseline is greater than 5 mm in height, the sample should be rerun, after adding more compound to the tube. This ensures that weak signals from quaternary carbon atoms won’t be lost in the noise.

The basic printout of the 1H (proton) spectrum will rarely be adequate. This spectrum is printed at approximately 225 Hz/cm, and it is impossible to discern coupling constants of a few Hz from this spectrum. Peak picking is of little value for this spectrum, since often many extra peaks (solvents, impurities) are listed. The integration regions chosen by the processing software may not be the best for your compound. The use of the basic spectrum is to qualitatively prove that the compound is present, to check if the sample was well made, and to give a rough idea of the purity of the sample.

It is expected that SpinWorks, ACD NMR Processor, or another software program will be used to analyze the spectrum. Within these programs, it is possible to integrate the appropriate regions of the spectrum, pick the peaks you want, and to consistently expand the multiplets and determine their coupling constants. For the latter, it is recommended that all expansions be done between 3–1 ppm/page. A 1 ppm/page expansion gives a printout at about 10 Hz/cm. This allows for a consistent appearance to the signals, and also allows for a qualitative visual comparison of the signals. If the expansion is less than 1 ppm/page (> 10 Hz/cm), the digital nature of the data becomes apparent and the signal shape is distorted. See the processing directions for obtaining these expansions.

The spectral data is best recorded in a table, similar to the example on the next page. Also include a structure with the atoms labelled (H_x, H_y, etc., or even better use IUPAC numbering) next to the table. Consider the possibility of impurities and solvents in the spectrum. In the report, remember that a table with the analysis of the spectrum does not show an understanding of the meaning of the spectrum. It is required that the critical features of the spectral data be discussed in the discussion section.
These are reported to 2 decimal places for $^1$H spectra, and 1 decimal place for $^{13}$C spectra. Well-resolved singlets, doublets, etc. have one exact value, while multiplets with indistinct coupling are reported over a range of values. See the paragraph on the next page.

<table>
<thead>
<tr>
<th>Chemical Shift $\delta$ (ppm)</th>
<th>Multiplicity</th>
<th>$J$-value(s) (Hz)</th>
<th>Integration</th>
<th>Assignment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Singlet (s), doublet (d), doublet of doublets (dd), triplet (t), quartet (q), multiplet (m), etc. Combinations of these are often required. Not required for $^{13}$C spectra, since $^{13}$C spectra are recorded with proton decoupling and the signals are all singlets.</td>
<td>These are the coupling constants of the multiplet, in units of Hertz (Hz). Report an average (see below) of the observed values. Not observed in $^{13}$C spectra.</td>
<td>Give the relative integration in numbers of protons (H), not just the number provided by the computer. Not meaningful for $^{13}$C spectra.</td>
<td>This is the part of the molecule that gives rise to the signal. Assignments must be clearly made—don’t just say “methyl group” if there is more than one methyl group in the compound.</td>
<td></td>
</tr>
</tbody>
</table>

Example Table #1: The $^1$H nmr spectrum of ethylbenzene in CDCl$_3$

<table>
<thead>
<tr>
<th>$\delta$ (ppm)</th>
<th>Multiplicity</th>
<th>$J$</th>
<th>Integration</th>
<th>Assignment</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.40–7.08</td>
<td>m</td>
<td>-</td>
<td>5 H</td>
<td>phenyl protons</td>
</tr>
<tr>
<td>2.66</td>
<td>q</td>
<td>$J_{ba} = 7.6$ Hz</td>
<td>2 H</td>
<td>$H_b$</td>
</tr>
<tr>
<td>1.25</td>
<td>t</td>
<td>$J_{ab} = 7.6$ Hz</td>
<td>3 H</td>
<td>$H_a$</td>
</tr>
</tbody>
</table>

![Diagram of ethylbenzene nmr spectrum]
Under the “δ (ppm)” heading, there are two ways to enter the data—either report the signal as occurring over a range of chemical shifts, or report an exact chemical shift. Remember that all protons in a molecule resonate at a particular frequency. The appearance of the signal depends on how complex the coupling is to the proton in question.

In the example, the aromatic proton signals are reported as occurring over a range of δ values. Reporting a range for the chemical shift is done when there is more than one inequivalent proton giving the signal, there is a lot of complex coupling, and/or the coupling is indistinct due to second-order effects. All three account for the appearance of the aromatic protons in this spectrum. Reporting the signal as a range gives an idea of the complexity of the coupling.

In the example the signals for the ethyl group have exact chemical shift values reported. Both of these signals give well-resolved, first-order multiplets, and the chemical shift of the signal is easily determined. The chemical shift is the centre of the splitting pattern for doublets, doublet of doublets and quartets, while the chemical shift is the position of the central line for singlets, triplets and pentets.

Under the “Integration” heading, the numbers have been rounded off to integers. Digital integration of proton spectra has an error of about 10 %, and the error is usually on the high side of the number. For example, the three methyl protons in ethylbenzene integrate to 3.04 on the expansions. Use the integration produced by the software to get the proton ratios. It’s up to you to interpret the integration value and report the number of protons giving rise to the signal.

If you have a mixture of compounds, the integration can be used to calculate the mole ratio between two or more substances. This is most often seen in a mixture of diastereomers. Be careful when reporting the integrations of a mixture of diastereomers—integrations within a compound must be whole number ratios (no quarks allowed), integrations between different compounds will be mole fractions.

With respect to the coupling constants: the coupling nuclei must couple with the exact same coupling constant. Unfortunately, because of the digital nature of the data, the coupling constants may appear to differ slightly. Thus, it is an average of the coupling that is required.

When labelling protons, be very clear in the labels. You may use either letters or numbers, but put some logical thought to the labelling. If the compound is given an IUPAC name, use the IUPAC numbers for assigning protons and carbons.

In the analysis, it is not necessary to record the nmr solvent signals in the table. Chloroform-d (CDCl₃) has a signal in the ¹H nmr spectrum at δ 7.26 due to residual CHCl₃ present and a water signal at δ 1.54. 100% deuterated chloroform is too expensive, we use 99.8% deuteration. Remember that D does not give a signal in ¹H spectra. Chloroform-d also gives a triplet in the ¹³C nmr spectrum at δ 77.2. This is due to the natural abundance of ¹³C in the solvent. Here, the coupling between the ¹³C and D atoms splits the carbon signal into a triplet.

Don’t spend a lot of time doing a full analysis of any minor impurities in the sample, and including the analysis in the table, rather make a note after the table that the impurities are present.
Preparation of NMR Samples

1. This is the important part. Good sample preparation leads to good spectra. Pay attention to what you are doing. Work with clean compounds that are free of solvent.

2. Prepare the sample in a clean vial, not directly in the tube. Use either 1 drop of a liquid sample, or 10 mg of a solid sample. Actually weigh the solid sample, don’t just estimate! If your sample will also be used for a carbon spectrum, 20–25 mg is more appropriate.

3. Add 0.7 mL of the appropriate solvent. This gives the required sample depth of 4 cm in the tube. Excess solvent leads to sample dilution and poorer signal to noise ratio in the spectra. If a different solvent than CDCl₃ is required, it is listed with the experiment.

3. Carefully filter your sample into a supplied nmr tube. The filter pipette is made by tamping a small amount of cotton wool into a second pipette. Clamp the filter pipette with the tip inside the nmr tube, don’t just put the pipette on top of the very fragile tube. Please take good care with the tubes—they are fragile.

4. Labelling is very important here in order to track the sample. If you wish to keep the sample after an overnight run, the tube must also have a paper label, which includes your name, the date, the name of the compound and the nmr sample number. You must retrieve the sample yourself within one week, otherwise the tubes will be cleaned.

5. The instrument will produce a basic spectrum, with integrals. Since the instrument decides what to integrate, it may not suit your needs. Also, expansions will not be automatically run. The use of the software for spectral manipulation is discussed later in this section, and will be demonstrated as needed.

Operation of the 300 MHz NMR Spectrometer

Once the sample has been prepared, operations in the spectrometer room must be done properly in order to obtain the nmr spectrum. Failure to follow the procedures below in order may result in a crash of the automation software. That isn’t a good thing. Remember—no gloves in the instrument rooms!

You’ve Just Come Into the Lab . . .

1. Place your sample in the rack behind the log book.

2. Fill out the log book with the required information. The date on the page must be today’s data. You must fill out the sample holder number, solvent used, your name, an ID for the sample, which experiment(s) you require, the experiment number(s). For example, recording a ¹H and a ¹³C on the same sample is two separate experiment and needs two experiment numbers. Note if you wish the sample to be retained for recovery or further experiments. Use two lines in the log book if necessary.

<table>
<thead>
<tr>
<th>Holder #</th>
<th>Solvent</th>
<th>Name</th>
<th>Spectrum title</th>
<th>Expts to run</th>
<th>Expt #</th>
<th>Retain? (y/n)</th>
<th>Printout OK?</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>CDCl₃</td>
<td>Harry Harris</td>
<td>Cyclohexanone</td>
<td>¹H, ¹³C</td>
<td>1, 2</td>
<td>No</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>acetone-d₆</td>
<td>Sally Smith</td>
<td>463 - Piperine</td>
<td>¹H</td>
<td>3</td>
<td>Yes</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>CDCl₃</td>
<td>J. Jones</td>
<td>363 - pyrazole</td>
<td>¹H &amp; COSY</td>
<td>4, 5</td>
<td>No</td>
<td></td>
</tr>
</tbody>
</table>
It is important that you completely fill out this information. In the event of a system failure, someone else may have to re-program your sample. **Note:** The next holder number after 60 is 1 (again). Contact your instructor for assistance if this happens to you, we need to make a software programming change.

3. Remove all paper labels from the tube. Place the spinner on the nmr tube. The wide part of the spinner is at the top of the tube. Gauge the depth of the spinner using the depth gauge—the spinner sits on the top of the gauge, and the tube just touches the bottom of the gauge.

4. Clean the bottom of the sample tube thoroughly with a kimwipe, taking care not to move the spinner. Throw out the kimwipe when done.

5. Place the sample in the correct holder of the sample changer. Don’t grab the thin, fragile, extremely expensive metal bar surrounding the sample changer.

**Console Time . . .**

1. The software needed to program the spectrometer to run your sample is already running. You don’t have to start anything, just enter the information required. Jiggle the mouse if the screensaver is on.

2. At any point, **if there has been as serious error in data entry**, you can escape from the set-up routine by pressing “DELETE”.

3. Programming the spectrometer is done by sample holder number. Double clicking on the holder number brings up a new set of dialog boxes which are used to tell the instrument what-to-do.

4. DON’T change the entries under “Type”, “Status”, “Disk”, “Name” (which is today’s date), “No.” (which is/are the experiment number(s)), “Par” or “User”.

5. The solvent is selected by clicking on the “Solvent” box, and selecting the correct solvent by clicking. The default solvent is CDCl₃, scroll down the list for other options.

6. The experiment is selected by clicking on the “Experiment” box, and selecting the desired experiment. The default experiment is ¹H. This is done one at a time for the 1D experiments such as ¹H and ¹³C, as well as the 2D COSY (run the ¹H first). For some of the 2D experiments (HMQC), all necessary experiments are programmed automatically, and each has a unique experiment number.

7. The title is selected by clicking on the “Title” box. You can then type in your title in the dialog box that opens. This title should include your name, and be descriptive of the sample. For example, if Will Windsor is running a spectrum of cyclohexanone for Chem 363, a useful title would be:

   “W. Windsor, Cyclohexanone, Chem 363”

This tells everyone whose spectrum it is, and allows Will to remember what it is. Conversely, a title of “WW-01” describes almost nothing about the spectrum. A longer description, including course number and type of spectrum, may be used.

Close the title dialog box by clicking either “Set Title” if you are running only one spectrum, or “Set and Copy Title” if you are running more than one spectrum.
8. If you want to run another spectrum with the same sample, click “add”. Then go back to step 6. If you are finished entering your requests, go to step 9.

9. To accept your entry, click first on your holder number and then click “Submit”. If you’ve changed your mind (after all that work), click “Delete”. DON’T click the “Cancel” button—ever!
   If you don’t click “Submit”, the automation run will skip your sample or worse, stop.

10. If you have any questions at any time, ask an instructor rather than guess.

**Can I Put My Sample in the Changer Now?**

12. It should already be there, otherwise put your sample in the correct sample holder.

13. That’s all for now.

**How Long Before I Get the Spectrum?**

For simple proton spectra, each sample requires about 10 minutes in the magnet, processing the data is almost instantaneous. Simple carbon spectra require about 18 minutes for data acquisition and processing. Cosy spectra can take 20 minutes for acquisition, and proton-carbon correlations are longer still.

During most of the lab time (1:30–4:30 PM), only simple proton spectra may be run. Other spectra (carbon and correlations) may not be placed on the instrument until after 4:30 PM.

The basic output is plotted on the laser printer at the front of the lab, or it may be placed in the output tray in the hallway. Judge the quality of the spectrum—is it acceptable, or does the quality of the sample need to be improved? Ask your instructor for guidance.

**Access to your Nmr Spectral Data**

The data sets for the nmr spectra are transferred from the spectrometer’s computer to two data servers. One is accessed within the Chemistry department, usually through the computer room (Elliott 330), and the transfer should happen soon (30 minutes or less) after the data is processed. It’s on the N:// drive.

The spectral data is also transferred to a web server the following morning. The web address is:

http://web.uvic.ca/~ugradnmr

If you plan to process your data on your own computer, go to the web site and click on the “manual” directory. This brings up a short introduction to the WinSCP file transfer program. There is also a link to the WinSCP program on the Chem 363 web page.

There are a lot of data folders for each spectrum, and the folders are only denoted by a number once they’re on your computer, so take care to organize your data.
**Processing NMR Spectral Data**

There are several programs available to analyze your spectrum for chemical shift, integration, and coupling constants. Bruker has their own proprietary software ($$$), however it is not available for undergraduate use in our computer lab. There are a couple of “freeware” programs on the internet, and we can make use of those. The results are equally good.


ACD NMR Processor (formerly “SpecView”): http://www.acdlabs.com/resources/freeware/nmr_proc/index.php (Academic use only)

Both programs are produced in Canada—it’s your choice as to which you use. There are links to these programs on the Chem 363 web site.

**Spinworks**

These notes only provide information for 1D spectra, and only for basic analysis. If you wish to try to use the program to do more advanced 1D processing, process and view 2D spectra, or to simulate spectra, consult Dr. Marat’s manual. The remainder of the instructions assume that you have the program installed and running, and that your spectrum files are available.

**Setting the Data Format**

The correct format for the Bruker 300 MHz data is set for SpinWorks version 3.17. If you are running SpinWorks 2.55, select the “Options” menu, select “Data Format” and “Bruker (UXNMR/XWinNMR)”. This data format may be set as the default format by selecting “Set Preferences” under the options menu, and clicking “OK”.

**Opening the Data**

Select “File” and then “Open” to get the standard Windows file selection menu. Navigate the directories as usual (this can include the network, if on a departmental computer), and double click on the directory of the desired experiment number. This opens the basic data directory. Click on the FID file to open it—this brings up the FID (free induction decay) for your compound.

If you have already processed your data with SpinWorks, click on the 1D_real file to directly access your spectrum (though you can reprocess your FID if you wish).

**Processing Data (if the FID file was opened)**

Select the green “Process” button on the lower right of the window, and click on. This applies a default set of parameters to the processing of the FID, does the necessary mathematical magic, and displays the spectrum. Parameters can be adjusted through the “Processing” menu, but this is far more work than necessary at this level.

**Zooming In**

You’ll need to know how to do this before phasing the spectrum, and doing the phasing on expanded peaks is much easier.

Click once in the spectrum display area, and a red cursor appears. Move away from the first cursor and click again to display a second cursor. It is the area between the cursors that is horizon-
tally expanded. Click on the blue “Zoom” in the upper right side of the screen to complete the expansion. For phasing, expansions can show a range of 0.4 ppm on the screen. For other expansions, especially those to be printed, expand to no less than 1 ppm.

To vertically expand the spectrum, use the yellow “+” and “-” tools on the toolbar—beside the word Expt: in the upper right of the screen. The scrolling wheel on a wheelmouse also works to change the vertical expansion. This is most useful for phasing, and for clearly visualizing weaker signals. Make use of the space available on the screen/page!

Fine control of the horizontal expansions can be done with the yellow “< >” and “> <” tools on the toolbar—beside the term H.Exp: in the upper right of the screen. Movement left or right of the expanded region is done with the scroll bar at the bottom of the screen.

Click on the blue “Full” button in the toolbar to get back to the complete spectrum, or on “Last Exp.” to return to the last horizontal expansion.

Phasing Data

This should not be necessary if the spectrum file was opened. You may be lucky, and the spectrum will be properly phased (all peaks are positive absorptions) once the data is processed. More likely, phasing will be required. Click on the interactive phasing button on the toolbar (on the lower right). It’s yellow in colour, and says “phase”. The use of the green “AutoPhase” button is NOT recommended.

Before you begin phasing the spectrum, you will need to zoom in on your peaks.

When you click the “Phase” button, a vertical green line appears, and this is the reference point for the zero order phase. Expand the indicated peak horizontally (keeping the baseline visible on both sides), and also expand it vertically. Adjust the upper (next to the ph0 box) coarse and fine sliders to control the zero order phasing. The number box to the left of the sliders displays the numerical value of the change. The peak should appear symmetrical with a level baseline on both sides of the peak.

Move to the peak farthest from the green vertical line by moving the slider at the bottom of the screen. You may need to adjust the vertical expansion. Adjust the lower (next to the ph1 box) coarse and fine sliders to control the first order phasing. The number box to the left of the sliders displays the numerical value of the change. When done, click on “Apply and Exit”.

What you are looking for is to have the baseline on the left side of the peak at the same level as the baseline on the right side of the peak. To see what bad phasing looks like, really move one of the coarse sliders a large distance! The better the phasing, the more accurate the integration.

Baseline Correction (if the FID was opened)

A baseline correction might be necessary. Fortunately, there is a “Fully Automatic Baseline” function under the “Processing” menu. In all but the worst cases, this should be sufficient.

Calibration

If the spectrum is calibrated incorrectly, the reported chemical shifts will be wrong. The entire spectrum may be calibrated when necessary. Expand the spectrum around the reference signal. Click once in the spectral window to put the cursor on the peak maximum, and select “Calibrate” from the “PeakPick” menu. For proton spectra, the residual chloroform peak is at δ 7.26, and in
carbon spectra, the middle peak of the triplet is defined as $\delta 77.23$. If you need to calibrate the spectrum, do this before you integrate or peak pick.

**Integration**

To start the integration, select the yellow “Integrate” button from the right-side toolbar.

Expand the spectrum horizontally as necessary. In the spectrum window, click where you want the integration to start, and click again where you want it to stop. The first integral that you define will be calibrated as 1.000, so try to select a peak that does indeed represent one proton. Move the spectrum window with the slider and repeat as required.

If you don’t want the integral traces (those curvy lines) obliterating your signals, click on the “/2” button until all the traces are flat lines. These still show the range of the integration.

To recalibrate the integrations, click once on an integral value. A signal that is expected to be one proton is best, but a methyl group (three protons) can also be used. Change the number in the box next to the “Calibrate” button, and click the “Calibrate” button.

It is possible to delete an integral, or all of them. The controls for this are on the left of the integration dialog box.

Clicking on “List” in the integration dialog box will give a Windows Notepad copy of the integrations, which may be printed.

**Peak Picking**

On the right-side toolbar, there is a button labelled “PP Minimum”. Click that, and click in your spectrum to set a horizontal pink line. Then re-click the bright pink button to “Return”. Any peak above that line will be picked by the procedure below.

On the “PeakPick” menu, select “Units” and set to Hz if analyzing a proton spectrum, or set to ppm if analyzing a carbon spectrum.

Expand the spectrum as necessary. Readjust the pink line using the procedure above. The peak picking function only works on the region displayed in the spectrum window. When you are happy with your selection, click “Peak Pick”, then select “Peak Pick and Append to List”. Move the slider at the bottom of the spectrum window to move to another region of the spectrum.

An individual peak may be picked by setting the cursor on the peak and right-clicking the mouse. Be careful, however that you are exactly under the peak you want.

Under the “PeakPick” menu, the entire peak list may be cleared, or just the peaks picked within the spectrum window.

Clicking on “List” on the “PeakPick” menu will give a Windows Notepad copy of the peaks, which may be printed.

**Printing**

Clicking on the “Print” button on the toolbar will give you a copy of what is on the screen, including integrations and peak picking. When printing, don’t expand beyond 1 ppm per page, or the signals will look distorted. Make use of the vertical space on the paper. Intense signals may be run off of the top of the page in order to see weaker signals above the baseline.
Exiting

It is not possible to save your integration and peak picking work, so finish the job the first time! Exit the program as you would exit any other Windows program.

Reference

SpinWorks Documentation, Version 2.2 (09/2003), by Dr. Kirk Marat, University of Manitoba. Just clicking around to see what worked also helped.

ACD/1D NMR Processor

Opening the file

Start the program like any other Windows program. Close the Processor Window that automatically opens when the program is started. Under the “File” menu, choose “Import” (NOT “Open”) and “From 1D NMR Directory”.

Navigate to your spectrum directory (it’s the N: drive in the computer room) and find your data by date code and experiment number (found on the top right hand side of the instrument output). Search through all the folders until you find the 1r file. Once the file is imported, your spectrum will appear along with a set of tools for manipulating the data.

You can also process the FID, but you’re on your own here, though the mechanics are similar to Spinworks.

Zooming In

Clicking on “View” and “Zoom area” brings up a small window that shows the full spectrum. The expanded area of the spectrum is shown as a grey box within that window.

You can either use the handles of this box to expand or contract the zoomed area, or you can use the magnifying glass tools near the top of the screen. If you overdo the zooming, there is a “zoom undo” button, or CTRL-Z also works to get you back to the full spectrum.

Good horizontal expansions have between 1–3 ppm on a page, any less than 1 ppm on the page and the peaks stop looking like nmr signals. Good vertical expansions make use of the space available, and it is acceptable to run intense signals (like methyl singlets) off the top of the page in order to view the weaker signals. Your wheel mouse might work for the vertical expansion.

Calibration

If the spectrum is calibrated incorrectly, the reported chemical shifts will be wrong. The entire spectrum may be calibrated when necessary. Expand the spectrum around the reference signal. Select “Reference” and click under the reference peak to open a dialog window. For proton spectra, the residual chloroform peak is at δ 7.26, and in carbon spectra, the middle peak of the triplet is defined as δ 77.23. If you need to calibrate the spectrum, do this before you integrate or peak pick. Click the green arrow at the left of the screen to continue.

Integration

The original output of your 1H 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.
These are typically useless for a detailed analysis. You will need to re-calculate and reference the integration.

On the toolbar, choose “Integration” and “Manual”. Move the red vertical cursor to the left hand side of a peak. Click, drag and release on the other side of the peaks to integrated. Move the spectrum horizontally with the slider, and continue this integration until all the desired peaks have been measured.

To reference the integrations, click on an integral value to highlight the integration in red. A signal that is expected to be one proton is best, but a methyl group (three protons) can also be used. Change the number in the “Reference” window in the toolbar, and hit “Enter”.

Click on the green arrow when you are done.

**Peak Picking**

Use the magnifying glass icons on the toolbar to re-size the spectrum (or part thereof) appropriately. A useful window can be opened under ‘View’ and ‘Zoom Area’. The grey box represents the expanded area which you can move around by clicking and dragging the edges.

Depending if you are analyzing \(^1\text{H}\) or \(^{13}\text{C}\) spectra, you need to change the units for the peak label. Click “Options” on the menu bar, then “Preferences”. Click on the “Peak” tab and select “ppm” (for \(^{13}\text{C}\)) or “Hz” (for \(^1\text{H}\)).

Select “PeakPicking” on the toolbar. Two options for labeling the peaks are available.

“Peak Level” lets you set the minimum peak level for the current expansion. Everything more intense than that level is picked. To pick a level or to change a level, just click in the window.

“Peak by Peak” can now be used to label any other peaks of interest that the “Peak Level” function has missed. Click on “Peak by Peak” and move the cursor to bring up a red vertical cursor when the mouse is over a peak. When the cursor is visible, click the mouse once to label the peak. To delete a peak label, click the mouse again.

Select the green check mark when finished peak picking.

**Printing**

Clicking on the “Print” button on the toolbar will give you a copy of what is on the screen, including integrations and peak picking. Try to avoid printing all of the tables that are generated. They don’t add anything and take away from the details of your spectra.

When printing, don’t expand beyond 1 ppm per page, or the signals get distorted. Make use of the vertical space on the paper. Intense signals may be run off of the top of the page in order to clearly see weaker signals.

**Exiting**

Hopefully by now you can exit a Windows program without explicit directions. Thanks to Dave Berry for a draft version of these notes.
On-line Resources

Links are provided on the Chemistry 363 web page (http://web.uvic.ca/~pmarrs/chem363/chem363.htm) for both SpinWorks and the ACD nmr processor, as well as SDBS (spectral data base system) and Aldrich Chemical Co. (many spectra are available) and our own nmr data web site.

Also available on the Chemistry 363 web page are links to more information about nmr spectroscopy, including:

• Spectra of organic solvents and some starting materials recorded on our spectrometer.
• The difference between chemical and magnetic inequivalence and the effect on spectra.
• Second-order effects on spectra.
• The effect of a chirality centre on spectra.
• The Karplus equation.

The pages are new, and most of them should be available in September.