Appendix B: Instructions for Using the picoSpin Nuclear Magnetic Resonance (NMR) Spectrometer

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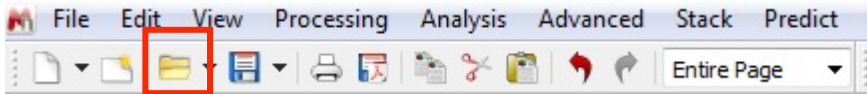California State University, Bakersfield

Recommended Resources

- Website ~ The Basics of NMR, by Josef P. Hornak
  http://www.cis.rit.edu/htbooks/nmr/

- Website ~ Nuclear Magnetic Resonance Spectroscopy
  http://www2.chemistry.msu.edu/faculty/reusch/VirtTxtJml/Spectrpy/nmr/nmr1.htm

- Video ~ NMR Made Easy! Part 6A - NMR to Molecule Structure - Organic Chemistry
  http://www.youtube.com/watch?v=5Uy7GiRaV2A

Safety Notes

- Do not take any metal objects near the NMR.

- Do not get too close to the NMR if you have a pacemaker or any medical implants.

- The magnetic field may damage watches, calculators, cell phones, or credit cards.

Directions

1. Prepare your sample.

    a. If your solvent is a liquid, you can inject the sample neat (i.e., you do not need to add solvent). If your solvent is a solid, add a minimum amount of deuterated chloroform ($CDCl_3$). If your sample does not dissolve in $CDCl_3$, you may need to try another NMR solvent such as deuterated dimethylsulfoxide (DMSO-*d6*).

    b. Check to make sure that your sample is completely dissolved and does not contain any solid contaminants. If you see any particles floating in your sample, filter to remove them (a glass pipette with a bit of cotton in the tip works great for this).

2. Inject your sample into the picoSpin.

    a. Fill a 1 mL plastic syringe approximately half way. Be sure to remove any air bubbles that may be present.

    b. Check to make sure the filter is properly connected and a collection flask is present in the "out" port.

    c. Slowly inject your sample into the "in" port until you observed a few drops coming out of the "out" port. (You do not need to inject the entire sample.)

3. Run a test spectrum.

    a. If you need to login to the computer, login as "picoSpin" using the password "nmr".

    b. In the Firefox web browser, go to http://192.168.42.31 (this should be the same IP address on the picoSpin display).

    c. On the picoSpin page, go to "Files".

    d. Select the "Standard Run" file.

    e. Click "Use these settings at run". You will be transferred to the "Run" page.

    f. Check to make sure the tx frequency is up to date. (It should be the same as the value posted on the computer monitor. This value is updated every few months.)

    g. Adjust the scans to 4.

    h. Click "Start Run".

    i. If the spectrum looks good, proceed to step 4. If the spectrum looks bad, inject more sample into the machine and click "Start Run" again. (Sometimes air bubbles will get into the machine. This is easily solved by injecting more sample to remove the air bubble.) If the spectrum still does not look good, ask your instructor for help.

4. Run a full spectrum.

    a. Adjust the scans to 16.

    b. Click "Start Run".

    c. Once the run is complete, go to the "Files" page.

    d. Click on "Last Run".

    e. Scroll down to the bottom on the page and click on the fid-avg.jdx file. This will save the file to the "picoSpin Runs" folder.

    f. Double click on the file to begin MestReNova. If the file opens, skip to step 6.

5. Open your spectrum.

    a. Open MestReNova.

    b. Click the "Open" icon located on the toolbar.

    c. Locate the "picoSpin Runs" folder on the desktop, and click on the most recent fid-avg.jdx file. (Note: if someone has run a spectrum before you, it may have a number at the end.)

    d. Click on the "Open" button.

    e. The spectrum will automatically open already processed.

6. Reference your spectrum.

a. Click the "Reference" icon located on the toolbar.

b. Click on the peak you wish to reference. (For example, if you added TMS to your sample, you would click on the TMS peak. You can also pick any other peak where the ppm value is known.)

c. In the "New Shift:" box, enter the desired value. (For TMS, the value would be 0.)

d. Click on the "OK" button.

7. **Phasing:** Ensure that the baseline of your spectrum is flat. (If the baseline of your spectrum is already flat, skip to step 8.)

    a. Select "Manual Correction" from the pull down menu located to the right of the "Phase Correction" icon in the toolbar.
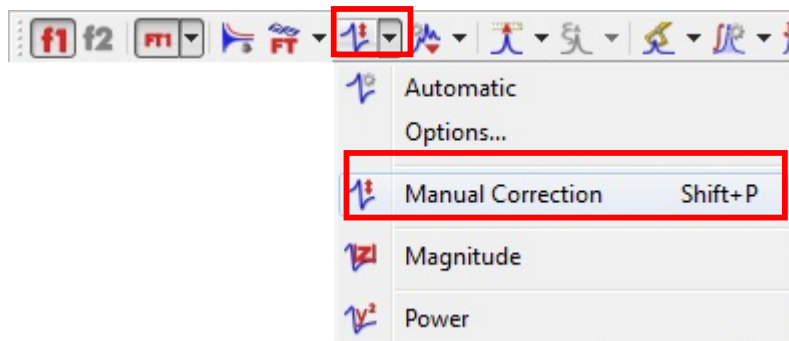

    b. Click and drag the mouse up and down in the blue box until the baseline of your spectrum looks flat.

    c. Close the window when you are done.

8. **Peak Picking:** Label the peaks on your NMR Spectrum.

   a. Clicking the "Peak Picking" icon located on the toolbar. Peaks will automatically be selected.
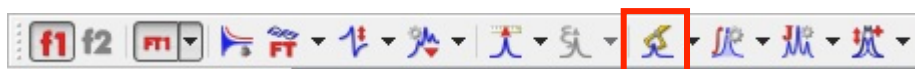
    b. To adjust peak picking sensitivity, select "Options…" from the pull down menu located to the right of the "Peak Picking" icon in the toolbar. Adjust the "Refinement Level" to increase or decrease the sensitivity as needed. Click on the "OK" button.
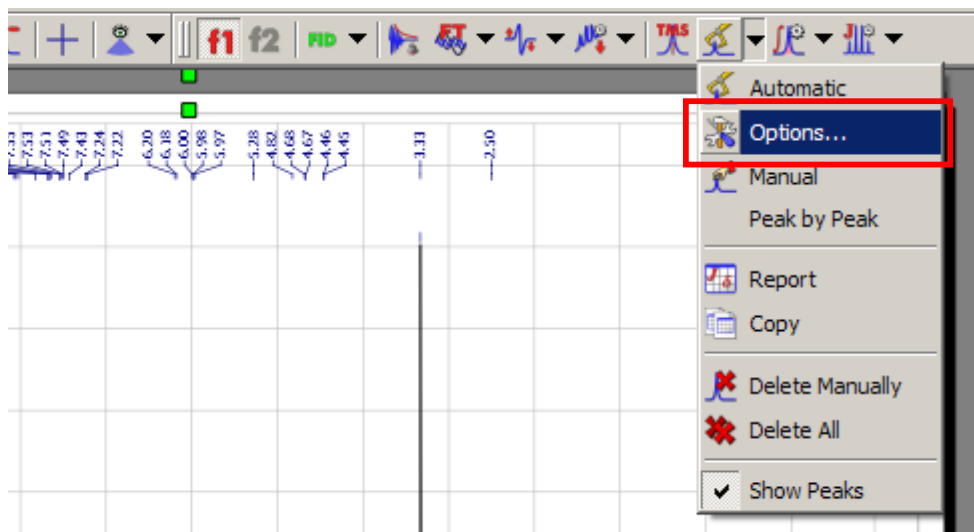
9. **Integration:** Determine the integration of each peak in your spectrum.

   a. Click the "Integration" icon located on the toolbar. The spectrum will be integrated automatically.
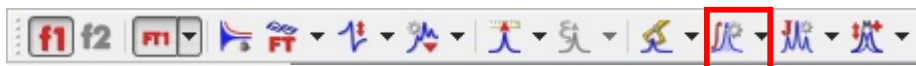
   b. To adjust integral values, run the mouse over an integral until it is highlighted pink. Right click and then select "Edit Integral…" Adjust the value in the "Normalized:" box as needed. Click on the "OK" button.

10. **Generating a report:** Format your NMR data for a lab report or publication.

   a. Click the "Multiplets Analysis" icon located on the toolbar. Multiplets will automatically be analyzed.
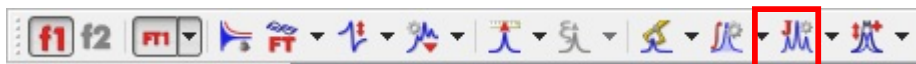
   b. To generate a report that can be pasted into a Microsoft Word document, select "Copy Multiplets" from the pull down menu located to the right of the "Multiplets Analysis" icon.

      c.   Click on the "OK" button.

      d.   Paste the report directly into your document (Ctrl+V).

11. **Saving your Spectrum:** To save a copy of your spectrum in pdf form, go to "File" and select "Save as PDF".