

Using XWIN-NMR to manipulate NMR spectra

1. Acquire a spectrum using the ICON-NMR program.
2. Wait until spectrum acquisition and work-up are done (ICON-NMR shows you the progress on screen. Also, once acquisition and work-up is over, you get a printout of your spectrum).
3. Click on the XWIN-NMR button (bottom of screen).
4. The XWIN-NMR screen offers a wide variety of functions, many of which are self-explanatory, such as expansion of region, moving spectrum up, down, left, right, etc.
5. Expanding a particular region – you can expand a particular portion of the spectrum. To do that, click on the button for expansion (it has an image of rectangle). Press continuously the left mouse button on screen and drag until the desired region is covered. Click the right mouse button. The expansion will appear on the screen.
6. Spectrum standardization – it is important that the spectrum is standardized properly, so you can report correct chemical shifts. The procedure is as follows: Expand the area containing the peak to be used as a reference (TMS or the corresponding solvent signal). Click **calibrate** and position the cursor on the top of the peak. Click with the middle button. A new screen appears, asking you to input the numerical chemical shift value for the standard (0.0 for TMS, values for the common solvents can be found in a Table behind and to the right of the computer monitor). Type the value and hit enter. Your spectrum is standardized.
7. Setting spectrum limits – the default spectrum that comes from ICON-NMR includes a large span of ppm, often regions that have no information. It is useful to set limits so that only the pertinent part of the spectrum is shown/printed. To do that, click **dp1** (define plot). A new screen appears, asking you to introduce numerical values for the limits (in ppm). The first number is for the left-hand (downfield) limit, click enter, then type in the right-hand (upfield) limit value, hit enter twice. Your spectrum with set limits appears on the screen. Any time you modify the limits (through expansion or contraction) you can get back to the preset ones by clicking on **PlotReg** button.
8. Integration – you can integrate each meaningful signal separately and then compare relative intensities. The procedure is as follows: Expand the region that contains the most downfield or most upfield signal(s). Click **integrate** and position the cursor on the left-hand side of a peak to be integrated. Click with the left mouse button. Click with middle mouse button

once: A white arrow (pointing downward) appears. Move the cursor to the right-hand side of the peak and click with the middle button again. An integral curve should appear. The assigned value for the first integral is always one (*If you want to change the value, to correspond to an actual number of H-atoms, this can be done. Please ask me and I will show you how*). Repeat the same process for all other signals (you can get them on screen by clicking the buttons with arrows pointing to the left or right, whichever necessary. When you have integrated all signals, click **Return**. The computer will give you three options. Select **Save as Integrated and Return**.

9. Setting threshold for peak-picking – it is convenient to have chemical shift values (in ppm or Hz) printed on the spectrum, above the corresponding peaks. Here is how to do it: Get the spectrum on screen and click **utilities**. Type **pscal**. Hit **enter**. Select **global**. Click **MI** (If the computer gives you a warning that your Y-scale should be in cm units, click **Seen**, click **YU**, then click **MI** again). A horizontal line appears, which can be moved up or down by simply moving the mouse. Move it to the desired height, then click with the left button. Click **return** (Lower left corner). The threshold is set.
10. Previewing your spectrum – you can now preview the spectrum, prior to plotting. However, the computer has some annoying defaults, which necessitate further action, as follows:
 - i. On top of the screen, go to **Output**, **Setup**, **Select Plotter**. Click on the **curplot** button. Select **HP Laser Jet 1300**. Click on **curprin** button. Select **HP Laser Jet 1300**. Click **Save**.
 - ii. Type **edg**. A new screen appears, with various options related to your spectrum appearance and print output. At the bottom of the list there are two buttons controlling the peak peaking. One of them determines whether labels are to be printed or not. The default is **No**. Click on the button to change it to **Yes**. Then click on the **edplabl** button below. It takes you to a new screen, where you can choose the units for the peaks (ppm or Hz). The default is **Hz** but in order to report signals you need values in ppm. Click on the corresponding button to change to **ppm** (*However, values in Hz are also useful, when reporting coupling constants. Therefore, if you have defined multiplets in the spectrum, i.e. doublets, triplets, etc., my advise would be to print the spectrum twice, with peak values in ppm and Hz*). Then click on the button controlling the color of the

labels. Change it to **black**. Click **Save**. Click **Save** again. You are now ready to preview.

iii. Type **view**. Your spectrum should appear in a new screen. Inspect the spectrum's features. If there is something you do not like then go back and change it accordingly (*e.g. some peaks were not reported and you have to change the MI value, you forgot to integrate a signal, etc.*). When finished, click **Quit**.

11. Plotting your spectrum: Type **plot**. Hit **enter**.

12. Plotting a selected region – in some cases it is useful to expand a particular region of the spectrum, in order to see some details, recognize patterns, etc. To do this on screen, you only have to click on the button for expansion and expand the desired region. However, if you want to plot the same region, this is not sufficient. The computer will always plot the region defined with the **dpl** command (see above). Thus, you have to perform **dpl** again, this time setting the limits such that the desired region will appear on screen. All subsequent manipulations are analogous to the procedure above.