

Zero Filling in 1D NMR SPECTROSCOPY

(Xwinnmr 3.5 patchlevel 6 , Windows XP)

**DPX200
DPX300
DRX500**

and

Processing computers



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1. A prerequisite for using this manual is that you know how to: **Login**, enter **password** and start the **xwinnmr** program.
2. Click on **File** followed by **search**. Select disk D: Select one of your own spectra.
3. Type **wrd**↵ This is the command for copying the dataset which is on screen to another disk or the same disk (home or other place) for permanent storage and at the same time **change the ownership** of the files.

Enter target data set **NAME:** **ZEROFILL**↵ (use your own expname)
 Enter target data set **EXPNO:** **2**↵ (use the expno seen in the top part of the xwinnmr window).
 Enter target data set **PROCNO:** **1**↵ (use the procno seen in the top part of the xwinnmr window).
 Enter target disk unit **DU:** **D:**↵
 Enter target user name **USER:** **UIO-username**↵ (your own user name)

4. Use **search** to find the ZEROFILL dataset, then **append** and **apply** it to the xwinnmr window and **efp**↵ to Fourier transform it. If the computer you are using does have another plotter attached than the one used under acquisition use **edo**↵ (edit output) to select the new plotter. The data set does regretfully remember the plotter name connected to the computer used to acquire the spectrum.
5. **efp**↵ and **apk**↵ the spectrum. Expand a sensible part of the spectrum on the screen..
6. Type **eda**↵ to see acquisition parameters. Look for the parameter **TD**. eda parameters can only be changed before the experiments starts.
7. Type **edp**↵ to see processing parameters. Look for the parameter **SI** (size). (edp parameters can be changed after the experiment is finished.)
8. Click on **Re**, **Im**, **Fid**, **Sh** and **Ush** and observe the effects on the screen. Re represents the “real spectrum” and **Im** represents the “imaginary spectrum”. They are phase shifted 90 degrees. The “**Re**” spectrum is presumably consisting of 16k or 32k data points. If so the **Im** spectrum has consists of the same number of data points 16k/32k. The whole dataset consists of the double number of points of either Re or Im. This parameter (data points) is called **SI** (size). What **SI** is set to is seen in **edp**↵ (processing parameters). When **SI=TD/2** no zero filling has taken place. The current value of **TD** is seen in **eda**↵ (acquisition parameters)

9. Use **edc** to increase the **procno** (processing numbers) for the following. Change **SI**, either by typing **SI** in the **xwinnmr** command line or by changing **SI** in the **edp** window. Select **SI** to be **2k**, **4k**, **8k**, **16k**, **32k**, **64k** and **128k**. Do an **efp** for each individual **SI** and watch the result on the expanded part of the spectrum on the screen. The expansion disappears for each **efp**. Plot if you like with **plot**.
10. When **SI = TD** you have zero filled once, when **SI = 2TD** you have zero filled twice. What have you done when **SI = TD/4** or **SI = TD/8**? Which part of the FID are you then using? Why are the spectra looking as they do with low **SI**?
11. Zero filling is important in 2D NMR. Another manual will be provided for this purpose. An alternative to zero filling in both 1D and 2D is **linear prediction** which also will have its own manuals.
12. Zero filling is important with wide sweep widths (large ppm area). The sweep width is connected to the acquisition time (AQ) in such a way that the wider the ppm area is the shorter is the AQ time for a given number of data points (TD). If you do not increase the number of data points when increasing the SW (in hertz or ppm) the AQ drops down. In certain cases the T1 relaxation is not over at the end of the AQ time. This will give bad "wiggles" at the baseline of strong peaks. Zero filling in combination with a WDW (window) function that takes the signal down to zero at the end of the FID (attenuates the signal) can make a bad looking (see above) spectrum better. The command **winfunc** brings up an interactive display for this purpose.
13. Change **procno** with **edc**. Open **edp**. Increase **TD** to a higher number. Change **LB** (line broadening) from 0.3 Hz to 3 Hz. Keep EM (exponential multiplication) as the wdw (window function). Expand and observe the differences between different **LB**'s.
14. **efp** the FID - watch the difference in appearance!!