NMR Training
Bruker AV-300 for Advanced Users

Resolution Enhanced 1D Processing

Sometimes the resolution in a 1d proton spectrum is not enough to identify a multiplet structure. In this case a special processing function can be employed to enhance the spectrum resolution. Normally the exponential multiplication in the processing routine employs an exponential decay function well suited to avoid FID truncation and to enhance signal to noise. However this is done at the expense of losing some spectral resolution. If spectral resolution is desired a different window function e.g. Gaussian, can be employed. This will enhance the spectral resolution at the expense of signal to noise. That is why this technique can be used only when there is plenty of signal to noise such as 1H, 19F or 31P spectra. To use the function, just open the desired 1D spectrum then click NEW and increment the processing number of the same experiment (do not change the experiment name or number). This will create a new processing of the same dataset. Click on "PROC 1D HR" button. This will process the data with the new processing parameters. Notice that you now have 2 different processings of the same dataset the original one and the resolutions enhanced version.

Multiplet Analysis Tool

Multiplet analysis tool (MAT) provides help in calculating NMR coupling constants. The most important thing to remember is that this tool works based on the peak list. So before using the MAT make sure the peak list is complete and all the interested peaks are covered. Note that sometimes manual peak picking is necessary to get some peaks that are not well defined in the 1D spectrum (e.g. overlapped peaks). Once the peak list is complete proceed to MAT by typing "mana" in the command line. Note the icons at the top for defining mutiplets. When defining multiplets always start with the smaller coupling constant first and work your way out to the largest one. For the smaller coupling in a multiplet peaks are next to each other so "Define multiplet by region" is the method of choice. Going out to the larger multiplets, depending on each case, you can use "Define multiplets by coupled grid" or "Define multiplets manually" and "Couple existing multiplets". When all the multiplets are done you can click on "Show Multiplet Report" and get a list of coupling constant. All the chemical shifts are also calculated automatically. The list can be either printed directly or copied and pasted into the xwinplot program.

Peak Assignments and Structure editor

To open structure editor click on structure tab in the dataset windows. This will open the structure editor. This program is very similar to ChemDraw. Once you draw the structure you can link each peak from the 1D spectrum to the structure. In the Peak list in the Annotation column you can assign a label to each peak (e.g. C12 or CH3). This label can be printed instead or together with the chemical shift in the xwinplot program.

Multiple Experiment Acquisition

The Topspin software offers the possibility to run multiple experiments in an automated fashion. For kinetic studies you normally follow a reaction by running the same NMR spectrum at a time interval. At the beginning run the first 1D spectrum like you would normally do, please note that this is a very important step which cannot be skipped. To start the series of 1D spectra type "multi_zgvd" in the command line. You will be asked to use a fixed or variable delay in between experiments. Choose fixed delay by typing "f" then input the total number of experiments. Be aware of the total time the series will take.

Multiple Experiment Processing

Tens or hundreds of experiments could be generated as a result of reaction kinetics studies. It would be very time consuming to process all these data files manually. There is an automation routine which can deal with this much faster. In order to use multiple processing routine you must process your first dataset manually. This is a very important step. You can do this by clicking in the PROC_1D button. Please pay attention to the correct spectrum phase. If the phase needs adjustment please do it manually. Once the first spectrum looks fine, type "multiefp_abs" in the command line. Answer the questions by choosing "experiments", first experiments to process will be usually number 2 since you have processed the first one manually then input the total number of experiments.

When proper integration is required for the series of spectra you can use the "multi_integ" command. Before you can use this command the first spectrum which will be called the reference spectrum needs to be manually integrated. Follow the instructions
1. Switch to the first experiment to be integrated.
2. Define the integral region(s) you want to use in the interactive integration menu. Calibrate the integrals with an appropriate calibration factor (calibrate button) and store the intrng file (write return button).
3. Store the intrng file with the 'wmisc' command under a name of your choice.
4. Start the integration ny typing "multi_integ". The individual integration results are stored in the file integrals.txt in each dataset; the summary of all results is stored in the file "intall.txt" in the first dataset. In order to access the summary file type the command "expl" in the first dataset.

Calculate 2D projections

13C NMR spectra of dilute samples sometimes require a significant amount of time for acquisition. 13C spectra can also be acquired indirectly through an HSQC or an HMBC experiment. Please remember that some quad 13C signals may not be present in the spectrum. The 13C spectrum will be calculated as a projection of the acquired 2D spectrum. Follow the instructions:
1. Acquire a 1D proton spectrum of your sample as you would normally do verify the quality of your sample.
2. Acquire an HSCQ spectrum of your sample.
3. Once the HSQC spectrum is processed go to Processing/Calculate Projections
4. Select Projection Sum of columns since your 13C is on the indirect dimension
5. Select the destination PROCNO default is 999. Now you have your 1D 13 spectrum.

Enjoy !