# Gradient shimming non deuterated solvents with Vnmrj 2.2/2.3

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Shimming non-deuterated solvents is usually possible with gradient shimming when a proton shim map is used. Clicking on the [Gradient Autoshim] button on the Start parameter panels uses the deuterium or lock channel maps and will fail for non deuterated solvents. For these solvents, you have to select from the main menu: Acquisition > Do Gradient Shimming > Use 1H/gradient map. Make sure the probe is tuned to detect protons before you start. This simple procedure may work fine, but unfortunately it gives no indication about "how good" the shimming was. For this reason, for "difficult" solvents (like those with multiple peaks), it is preferable to use the full gradient shimming interface. This is done by selecting Tools > Standard Calibration Experiments > Set Up Gradient Shimming from the main menu. The parameters and panels for gradient shimming will be loaded as shown below.
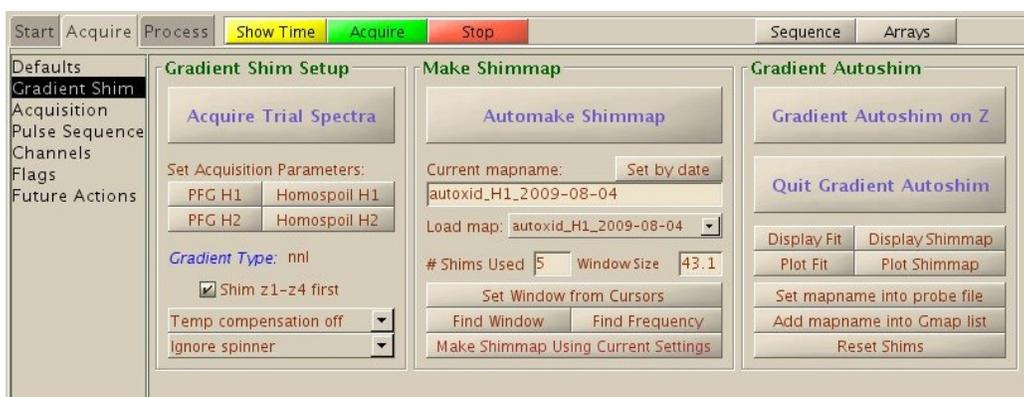

The first step is to make sure an appropriate shim map is already loaded under the Current mapname: field. Map names are made up of the probe name, followed by the nucleus (lk or H1), followed by the date, for example: autoxid_H1_2009-08-04. If a proton map is not already loaded, select the most recent map available for H1 shimming from the Load map: drop down menu.

Solvents with multiple resonances, particularly THF, may require fine tuning of the parameters in order to improve shimming performance. Perhaps the most important parameter to adjust is a delay called d3[2]. In solvents with multiple peaks, the resonances may interfere with each other, reducing the signal needed for proper gradient shimming. The amount of interference depends on the solvent and the value of d3[2]. The default value is 5 milliseconds. This value may be fine for some solvents, but for THF, 15 milliseconds works better. To check if the parameters are appropriate, click the [Acquire Trial Spectra] button. Two test "spectra" (or gradient profiles) similar to the ones shown below should appear. The second one should be about 30% to 70% the height of the first one. If the second profile is too weak and noisy or if it has almost the same height as the first one, then the default value of d3[2] should be changed. To find a good value, make an array of delays by typing on the command line: d3=0,0.005,0.010,0.015,0.020,0.025,0.030 and type go (make sure the probe is tuned to proton before you start). When the acquisition stops, type wft dssh to display the array. Select a profile that is about half as intense as the first one in the series and notice its value of d3. For example, if you selected the fourth profile, it corresponds to a d3=0.015. Then modify d3 by typing: d3=0,0.015 (this sets up a new array where the first element has a value of 0, and the second element, or d3[2], has the desired value of 0.015). Typical values of d3[2] for protonated solvents are 5 to 30 ms, and 30 to 200 ms for deuterated solvents.

Other parameters can also be adjusted. For example, if the profiles show ridges on both sides of their bases, the relaxation delay d1 should be increased or the gain decreased. Or, if the profiles are noisy (common when using lock maps), increase the number of scans: nt=nt*2.
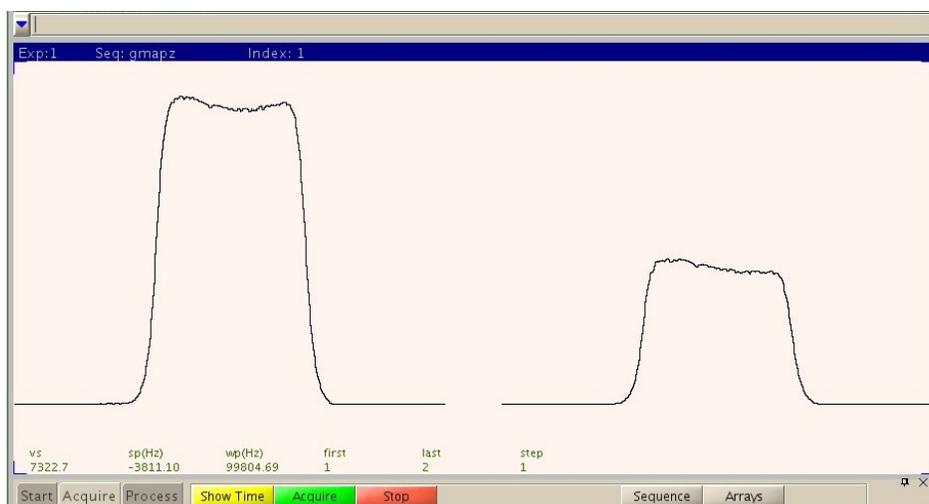

After adjusting parameters, click [Gradient Autoshim on Z]. The shimming process then starts and after each iteration, it displays a plot of the magnetic field homogeneity similar to the one shown below. A successful shimming should show a red curve that is as flat and horizontal as possible. Some noise or deviation at the edges is common. If not satisfied, the shimming process can be repeated. To finish, click [Quit Gradient Autoshim].
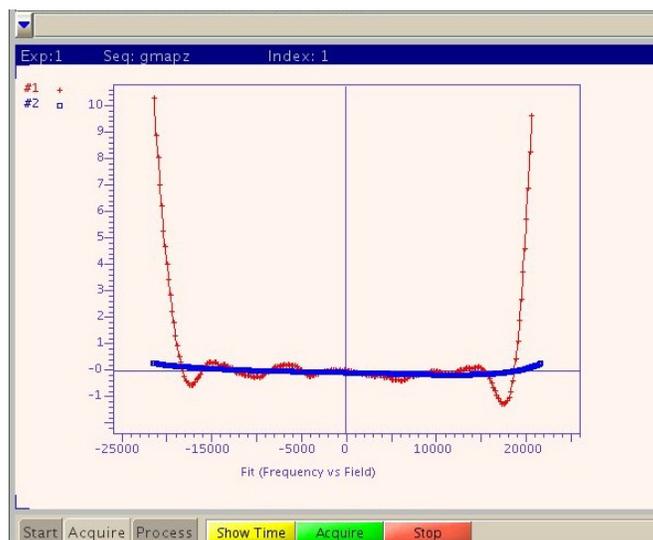
Shimming on the FID

If gradient shimming didn't work well, or if you want to improve the homogeneity, shimming can also be done manually by looking at the free induction decay after each scan. Load parameters for proton spectra and set a

short acquisition time of 1 second (at=1). Next, go to the **Start, Shim** parameter panel (shown below) and click [**Fid scan**]. After a few seconds, the FID of your sample will appear and will be refreshed every second. You can now start shimming manually. The goal is to increase the FID's intensity as much as possible, and to produce an FID with a long decaying tail. As a guide, the "clock display" in the parameter panel shows the area of the FID. If the area is past 999, click [**Rescale FID area**] to reset its value to 500. Adjust the shims very slowly, watching the result of every change before continuing. When you are done, click the [**Stop**] button or type aa and reload the acquisition parameters.
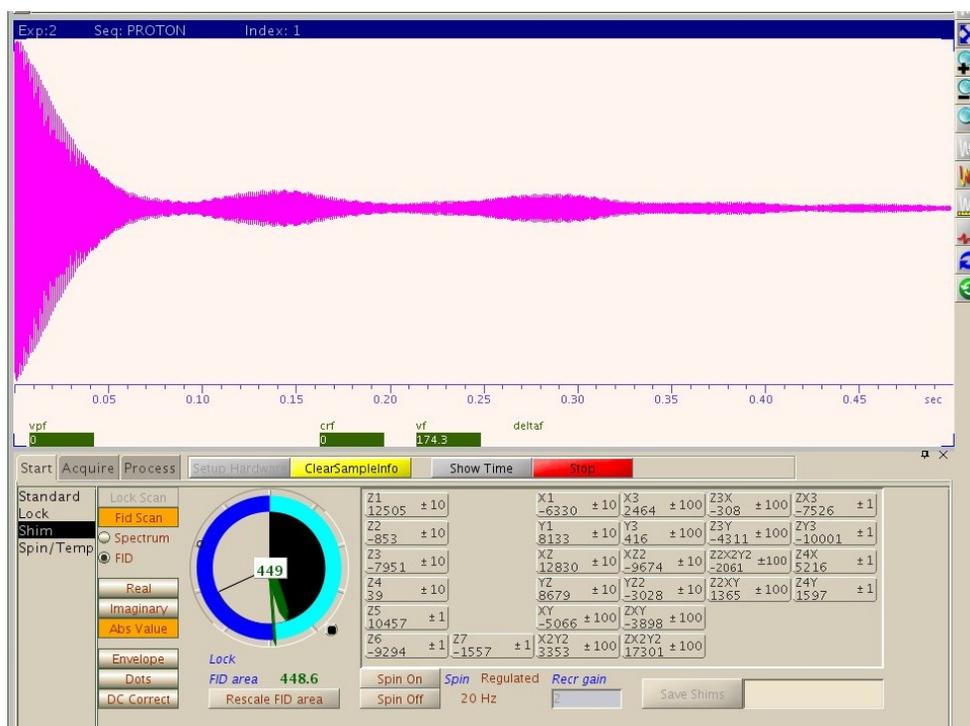
Shimming on the proton spectrum

It is also possible to shim a sample by watching an actual peak in the proton spectrum. First, load parameters for proton spectra and reduce the acquisition time (at) to 1 or 2 seconds. To be able to achieve narrow line widths, the spectrum needs high resolution (long acquisition times) but the process will go more slowly. If your solvent is protonated set the gain to 0. Then take a spectrum with only 1 scan (nt=1) and process it. Now zoom in to a single peak that you can see clearly and you want to use for monitoring the line shape, but do not choose the solvent peak (it is probably broadened by *radiation damping*). Go to the **Start, Shim** parameter panel, click [**Fid Scan**] and then select the **Spectrum** option below that button. The acquisition will start and after each scan, the zoomed region will be shown. You can now shim as usual. The goal is to obtain a peak that is as narrow, and symmetrical as possible while maintaining a Lorentzian line shape. A good understanding of the effect of each shim in the line shape is essential. Consult textbooks on experimental NMR, for example the books by Claridge or Derome or the internet. When you are done, click the [**Stop**] button or type aa and reload the acquisition parameters.