Shimming an NMR Magnet

Shimming is a procedure to generate homogeneous magnetic field along the sample volume to obtain pure lorentzian line shapes of various resonances in the spectrum. This is accomplished by manual shimming of various axial (Z^1, Z^2, Z^3, Z^4, Z^5 and Z^6) and radial (X, Y, XY, XZ, YZ, X^2-Y^2, etc.) shims by iterative method.

Shimming of Bruker NMR magnet can be accomplished by three methods:

1. Manual shimming of individual shims.
2. Simplex shimming (semi automatic shimming procedure)
3. Gradient autoshimming

Manual Shimming of individual shims

This can be accomplished by altering shim currents (by changing individual values of different shims such as Z^1, Z^2, etc.) in an iterative process. In Bruker spectrometers, both lock display window (***lockdisp***) or FID window (in ***gs*** mode) can be used for obtaining best set of shims. For users, it is advised to use lock display window for optimizing shims in day-to-day operations of the spectrometer.

Note: *FID shimming is only advisable in case samples are recorded without deuterated lock solvent*

Shimming on lock signal

Two horizontal sweeping lines in the lock display window represent height of the lock signal (deuterium signal of the solvent). With shimming, field homogeneity around the sample tube increases resulted in increase in intensity of the lock signal as represented by vertical height of the sweeping lines. Step-by-step shimming procedure is described below

The shim buttons on most of the modern BSMS keyboard (spectrometer equipped with BOSS II shims) looks like

following figure. However, you might see a little different layout of shim buttons depending on the instrument model and year

Shims are categorized into two categories:

(a) axial shims: **Z^1**, **Z^2**, **Z^3**, **Z^4**, **Z^5**, **Z^6**, etc.

(b) radial shims: **X**, **Y**, **XY**, **XZ**, **YZ**, **X2-Y2**, etc.

Note: Radial shims are optimized without spinning while axial shims can be optimized with or without spinning

Note: In the BSMS keyboard layout displayed above, each shim are selected by pressing two buttons (one after another) e.g. axial shims (**z**, **z2**, etc.) are selected by pressing ONAXIS and **Z^n** (n = 1-10).

Similarly, radial shims are selected by pressing combination keys of **X**, **Y** component button followed by **Z** component button (e.g. **X** = **X** + **Z^0** and **XZ** = **X** + **Z^1**)

- Select **X** by pressing appropriate combination buttons, current value of **X** appears in the display panel of BSMS keyboard
- Turn knob (make sure *FINE* light is off) either clockwise or anti-clockwise to optimize **X** shim
- Observe changes in the lock level (represented by increasing/decreasing vertical height of sweeping lines). If it increases by turning knob in a particular direction, turn the knob in that direction till you see lock level starts deceasing after reaching a particular height. Slightly turn the knob in the opposite direction to maximize the lock level. If there is no change i.e. turning knob in either direction forces lock level to decrease, then press ***STD BY***
- Once **X** shims optimized, start optimizing **Y** shims exactly following the procedure described above
- If there is change in **Y** shim value during optimization, then go back to **X** shim and start optimizing **X** shim again as both **X** and **Y** are interdependent
- Repeat this process of **X** and **Y** shims optimization till you don't see any changes (increase) in lock level
- Start spinning at this point by pressing SPIN ON-OFF button once (if you want to acquire spectrum without spinning (e.g. 2D spectrum), then skip this step)

- Select Z^1 shim by pressing appropriate combination buttons and optimize as described above (make sure FINE light is on while optimizing Z^1 and Z^2 shims)

- Once Z^1 gets optimized, select Z^2 and optimize Z^2 and if there is changes in Z^2 value, then return to Z^1 and optimize by readjustment. Repeat this process of optimization for both Z^1 and Z^2 iteratively till you don't see any changes (increase) in lock level

- Once you satisfied with the shimming, go back to Section 6.8 and acquire a spectrum with 1 scan (**NS** = 1) to check the line shape

- Check line shape as described in Section 6.9 and if it is not completely Lorentzian, see the figure below to find out which shim or set of shims need to optimized to get ideal line shape

- Once you select one shim or set of shims to optimize, start optimizing shims as described above

Attn: Whenever you change any higher order shim values, lower order shims also changes so you need to optimize all lower order shims also (e.g. changing Z^2, you need to iteratively adjust both Z^1 and Z^2, similarly changing Z^3, all three (Z^1, Z^2 and Z^3) needs to be iteratively optimized)

Optimization of other higher order shims so called second order shims such as Z^3 and Z^4 are little bit more complicated. Adjustments of these two shims are not recommended on routinely basis. However, if the line shapes from figure indicates that Z^3 and/or Z^4 shims needs to be adjusted then follow following guidelines for optimization

Gradient Shimming in Bruker NMR Spectrometer:

Manual shimming method is fairly time consuming and sometimes even with best efforts it is hard to find suitable set of shims for ideal line shapes if higher-order shims needs to optimized.

Gradient shimming provides rapid, automatic adjustment of room temperature shims (mostly z shims). Typically it takes couple of minutes to obtain quality shims without much user intervention and all steps can be followed by the simple procedure written below.

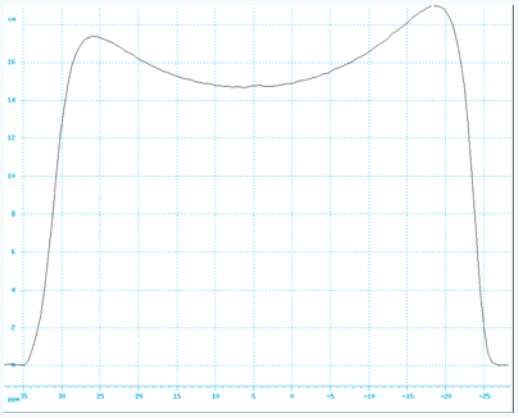
Gradient shimming of all z shims (axial shims) can be implemented with spectrometer equipped with z-gradient probe and amplifier. However, if the spectrometer is equipped with xyz gradient probe and amplifier, then one can

obtain a complete shim map including both radial and axial shims. Gradient shimming method supports shimming on a wide variety of samples with different volumes and solvents as long a single, strong resonance is present in any part of the spectrum.

This manual is a practical guide to perform **gradient shimming** on Bruker spectrometers at University of Pennsylvania, Department of Chemistry running ***XWinNMR version 2.6***.

Gradient shimming method consists of two steps:
1. Shim mapping
2. Auto shimming based on shim map.

1. **Shim Mapping**: These are maps of the effects of each shim as a function of sample position within the magnetic field. This procedure usually done once during the installation of the probe and as long as there is a reasonable shim map, it is undesirable to perform shim mapping again. However, for advanced users, this process can be repeated at any time to check the quality of shim map.



1D proton image from BBI probe on 2mM sucrose in 90% H_2O, 10% D_2O

1H 1D Shim Map: Remember shim map is probe specific and so users need to be aware of current probe in the spectrometer and it should be properly defined in **edhead** subroutine.

- Use a sample with one strong solvent line (e.g. ***2 mM sucrose sample in 90% H_2O, 10% D_2O***)
- Shim the probe to achieve best possible shim (both radial and axial shims) and write the shim set into the shim file **GSHIM** (***wsh GSHIM y***)
- Create a new data set (***edc***) and read in (***rpar***) the parameter set **gradshim1d1h** (pulse program: **imgegs1d**) (***rpar gradshim1d1h all***)
- Enter the value of ***O1P*** (exact position of the solvent resonance)
- Enter desired pulse length ***P0***/***PL1*** to achieve a 30-45 deg pulse width (e.g. ***P0*** = 2 us, ***PL1*** = 16 dB)

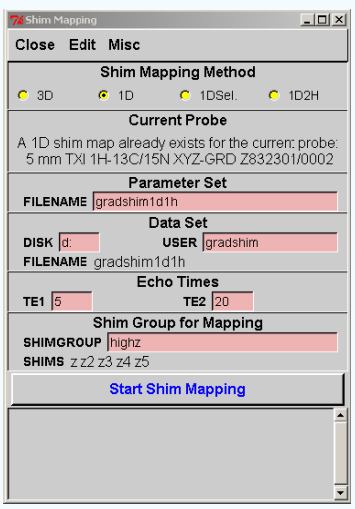
- Optimize ***RG*** with ***rga***

- Acquire the spectrum with ***zg*** (default ***TE1*** and ***TE2*** are 5 and 25 ms) and process it with *fmc*

- Optimize the ***SW*** to the gradient profile such that profile looks like spectrum below

Once the image is optimized, save the parameter set with *wpar <probe specific name>*

- Open gradient shimming window by typing ***gradshim***



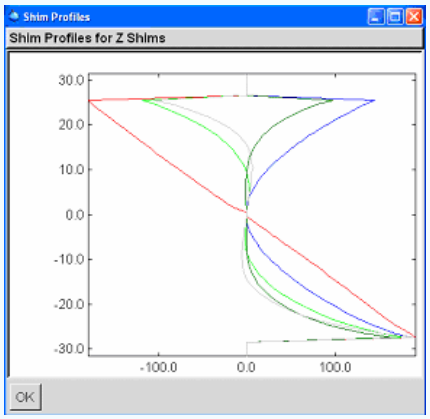
Shim mapping window layout

- Open shim mapping function from ***Setup|Shim mapping***

- Select **1D** in ***Shim Mapping Method*** tab

- Enter name of the parameter set created in ***Filename*** tab

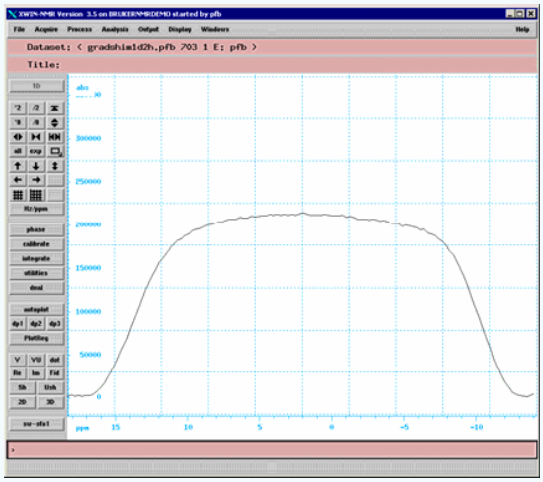
- Start shim map acquisition by clicking on **Start Shim Mapping** tab



Well behaved map from 1D proton shim mapping with BBI probe. Window size here is +/- 26

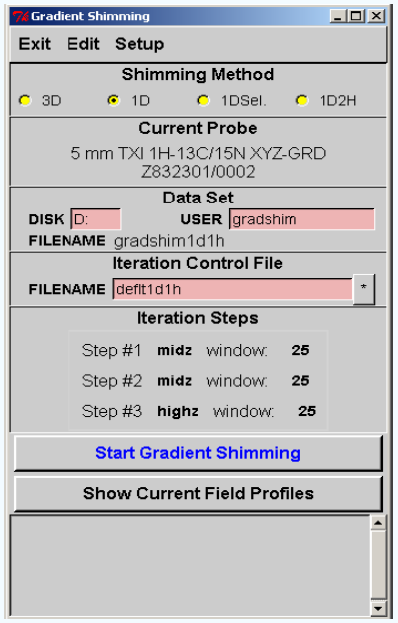
- Once shim mapping is done, check it. If satisfied, close the window

<u>2H 1D Shim Mapping</u>: This is similar procedure, however, a separate *2H lock switch unit* or *BSMS 2H-TX board* is required for deuterium gradient shimming. Ideal sample for this purpose is routinely used line shape sample (***1% $CHCl_3$ in Acetone-d_6***)



Acceptable profile from 1D 2H parameters

- Use a sample with one strong deuterium line (usually deuterated solvent signal)
- Shim the probe to achieve best possible shim (both radial and axial shims) and write the shim set into the shim file GSHIM (*wsh GSHIM y*)
- Create a new data set (*edc*) and read in (*rpar*) the parameter set gradshim1d2h (pulse program: imgegs1d2h) (*rpar gradshim1d2h all*)
- Enter the value of O1P (exact position of the deuterium resonance)
- Enter desired pulse length P0/PL1 to achieve a 30-45 deg pulse width (e.g. P0 = 12 us, PL1 = 10 dB)
- Other parameters are TD = 512, D1 = 100 ms, D27 (gradient time) = 6 ms, NS = 32/64, SWH = 5000-9000 Hz
- Optimize RG with *rga*
- Acquire the spectrum with *zg* (default TE1 and TE2 are 5 and 150 ms)and process it with *fmc*
- Optimize the SW to the gradient profile such that profile looks like spectrum below
- Once the image is optimized, save the parameter set with *wpar <probe specific name>*
- Open gradient shimming window by typing *gradshim*
- Open shim mapping function from *Setup|Shimmapping* (Figure 2)



Gradient shimming window layout

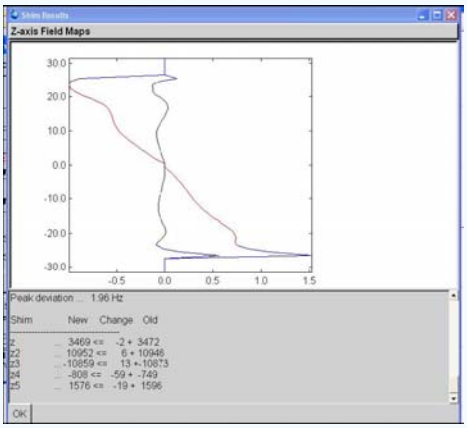
- Select 1D2H in *Shim Mapping Method* tab
- Enter name of the parameter set created in *Filename* tab
- Start shim map acquisition by clicking on *Start Shim Mapping* tab
- Once shim mapping is done, check it. If satisfied, close the window

2. **Gradient Auto shimming based on stored shim maps**:

- Open gradient shimming window with *gradshim*. Check that spinning remains turned off!

- Select desired method (1D or 1D2H) in *Shim Mapping Method* tab

- Select suitable iteration control files i.e. select or generate a suitable shim iteration control subroutine (for inverse probe head, typically **z1-z5** (highz) shims are iterated simultaneously) (e.g. Iteration Steps: *Step #1 highz window: 26*)



Changes to the shim current values after gradient shimming

- Start with a window size of **22-26** data points

- Start gradient shimming by clicking *Start Gradient Shimming* tab

- Check the following after one iteration

    - The changes in the shim values (*generally changes to the shim values on the order of < ±5 for z1 and z2, < ±50 for z3 and < ±200 for z4 and z5 indicate convergence*)

    - The standard deviation

Troubleshooting:

- If changes in shim values are higher than defined above (e.g. for iteration 3 the change of z3 was +1554, in iteration 4 -743 and in iteration 5 again +899), this indicates a problem in the shimming procedure.
    - Either stored shim map is wrong or the acquired profile is too noisy to allow shimming of the higher z gradients.
    - Check if shimming improves by changing iteration control steps to midz (z1-z3) instead of highz (z1-z5).
- Line shape is bad even after gradient auto shimming
    - Check that radial shims are correctly shimmed.
    - Check sample quality (make sure sample height is at least 5 cm and well made solution)