# SHIMMING

# BRUKER NMR INSTRUMENTS

## (DPX200-, DPX300-, DRX500-, AV600- and AVII600-MHz)

## USING

## BSMS

## (BOSS 1 and BOSS 2)

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**INTRODUCTION:**

## What is shimming?

The curious term "shimming" has its roots in the early history of NMR when superconducting magnets where not existing yet.

The term is derived from the engineering practice of using thin bits of metal (called shims) as spacers between the coils of ordinary electromagnets in order to make their magnetic fields homogeneous.

In the context of modern NMR the shims are small variable magnetic fields used to cancel out in-homogeneities in the static main field. These small magnetic fields are produced by little electromagnetic coils, so-called shim coils, surrounding the probe which is holding the sample. During shimming the electric currents in this shim coils are adjusted, so that the resulting small magnetic field gradients cancel the residual in-homogeneity of the main field as completely as possible.

The superconducting magnet is producing a strong, but quite inhomogeneous main magnetic field at the centre of the superconducting coil where the sample is placed.

The field in-homogeneities are caused mainly by the inhomogeneous distribution of (magnetic) objects within its near surrounding, like steel armour in the walls, floor and roof, but is also due to the sample itself.

In addition, the "static" field itself is not 100% constant. It is always drifting a little bit in time and through space, that means is changing both its strength (by loosing energy) as well as its degree of homogeneity.

## Why bother with shimming?

A distorted, inhomogeneous field will produce low-quality spectra with poor resolution.

Depending on the degree of in-homogeneity you will either get:

- Unsymmetrical signals displaying a shoulder
- Split signals, where the splitting is *not* due to coupling
- Broad signals with only rudimental coupling pattern or no coupling pattern at all.

Even worse, you might not be able to lock in the first place.

Therefore every time a new sample is inserted into the magnet it is necessary to readjust the shims (or at least some of them).

The better you shim, the better the homogeneity of the magnetic field, the better the quality of your spectra will get, regarding both sensitivity and resolution.

## FACTORS AFFECTING RESOLUTION:

There are several factors concerning the sample which affect the resolution to the negative and *which have nothing to do* with "bad shimming". Most of these factors can easily be avoided or eliminated:

### Correct Liquid Height and Position of the Sample Tube:

Make sure that you have enough of your sample in the tube.
The optimal liquid height is 4.0 cm (0.55 ml total volume) for the BRUKER instruments.

Further make sure that you place the tube in the correct position, which means that the bottom of the tube is *touching* the white Teflon plate in the position measuring device.
There are several reasons, why these two conditions **must** be fulfilled:

- At the top and the bottom of the liquid column, where there is a transition from solution to air or glass, are found considerable changes of magnetic susceptibility which induce distortions of the static magnetic field, e.g. in-homogeneity. The disturbing influence of these regions will decrease as their distance to the centre of the observation window is increasing.

- The shim parameters stored in the shim file (which you retrieve by using the command **rts**) were achieved by using a sample which had the correct fill height and which was positioned correctly.
  That means that the shim file **is only valid for a sample that meets the same conditions.**

- If the fill height is too low, the vortex produced by rotation of the sample tube will reach down into the observation window. This again is generating changes of magnetic susceptibility (transition solution/air).
  If the fill height is too large, the spinning might not work properly, because the sample has got too heavy. In addition it is a waste of solvent.

In case you are forced to reduce the sample volume because you only have a few milligram of compound, you should place the liquid volume symmetrically around the centre of the observation window (see sample depth measuring device).
In this case you probably will have to shim all four Z gradients.
But be aware that reduction of sample volume to attain higher concentration usually fails, because the signal is found to be increased around the base of the signal, not in its narrow portion (e.g. the signal/noise ratio is not really affected).

### Sample Spinning:

Make sure that the sample is spinning properly, for the BRUKER instruments indicated by the LED on the button SPIN ON/OFF located on the BSMS tool.

The indicator lamp should burn steadily. The spinner speed should be 20-25 r.p.s. (Hz)
By rotation of the sample around its vertical axis in-homogeneities of the static field are averaged to zero.

**Sample Concentration:**

"The higher the concentration of my sample is the shorter the acquisition time?"
Yes - in principle.
But there are limits. A too high concentration of the sample will result in an increase of its viscosity. In other words, the molecules are not longer able to tumble free in the solution. This causes dipol-dipol interactions and chemical shift anisotropy, both resulting in line broadening.

Suitable concentrations, which give a good resolution in an acceptable effort of time, are:

**0.10 mmol for $^1$H and**
**0.25 mmol for $^{13}$C   for compounds with a molecular weight < 500.**

**Make sure that the solution is homogeneous to avoid concentration gradients which again may cause changes in the magnetic susceptibility.**

**Solid Particles:**

Solid particles, like fibres of filter paper, dust, silica or aluminium oxide, suspended in the solution are lowering the resolution and should therefore be removed by filtration. The reasons for their negative influence are changes in the magnetic susceptibility.

**Viscosity of the Solvent:**

Solvents of higher viscosity, like DMSO is giving broader signals than lower viscous solvents, like Acetone. This is because of dipol-dipol interactions and chemical shift anisotropy.

**Radicals and Paramagnetic Materials:**

The presence of radicals and paramagnetic materials in the sample, if only in traces, is leading to very broad and unresolved signals with a bad signal to noise ratio. The reason is that they are relaxing the nuclei of the sample very fast and the FID has no time to evolve.

**Solutions of Salts:**

Salts in the NMR sample are giving broad signals. The charged ions produce small magnetic fields on their own while moving through the solution. These fields are disturbing the general field homogeneity.

## ABOUT THE SHIMS:

The shim assembly consists of different types of gradient coils.

a) **Axial** (or **on-axis**) shims
(Pure "Z" shims, e.g. Z1, Z2, Z3, Z4, Z5, Z6)

The gradients produced by axial shims affect the field homogeneity along the Z-axis, which means parallel to the length of the tube.
The on-axis shims are not averaged by sample spinning, which means they affect the line shape whether the sample is spinning or not.
By convention the on-axis shims are adjusted with spinning (**on-axis = spinning on**)
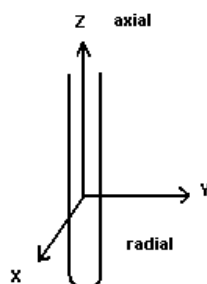


b) **Radial** (or **off-axis**) shims
(All shims containing the letters "X" or "Y", e.g. X, Y, XZ, YZ, XZ2, YZ2, XZ3, YZ3, …X2-Y2, XY etc.)

The gradients produced by radial shims gradient affect the field homogeneity in the X/Y-plane, perpendicular to the Z-axis or the length of the tube.

The off-axis shims are averaged by sample spinning and are therefore adjusted without sample rotation. Off-axis shims affect the line shape when the sample is not spinning, and the size of spinning sidebands when it is spinning.

Rotation of the sample tube, the so-called sample spinning, is used to improve the quality of the signal. It makes the signal become less broad all the way from its base to its top and is therefore increasing the signal intensity (think about the signal being made of rubber and you are tearing at its top).
That is because rotating the sample does average or cancel out field inhomogenities along the X- and Y-axis, but not along the Z-axis, e.g. the axis about which the sample is rotated.
Therefore the off-axis shims are influenced by sample spinning and have therefore to be adjusted without spinning (**off-axis = spinning off**)

## IMPORTANT TERMS AND ABBREVIATIONS:


**LOCK POWER**   **Lock (transmitter) power.** Controls the amount of energy being transferred into the sample by the applied radio-frequency field.

**LOCK GAIN**   **Lock (receiver) gain.** Controls the amplification of the received signal.

**LOCK PHASE**   **Lock phase.** Controls the phase of the lock signal.

**LOCK LEVEL** - Displays the magnitude of the lock signal in % relative to saturation

**BSMS**   **B**ruker **S**mart **M**agnet control **S**ystem, is the keyboard unit with all the knobs and the wheel, which you find beside the terminal to your right above the mouse. The BSMS is used to control various functions associated with the magnet, magnetic field and sample.

For the use of The BSMS note generally:
By activating a key function on the BSMS the light on the key will turn on.
At the same time red LED's on the display shows its actual value.
By turning the wheel, e.g. changing the value, the original value is shifted to the left of the display, while the (new) actual value is displayed at the right.
If you want to accept, e.g. to save, the new value either press STD BY or press any other key.
If you want to return to the original value press the activated key once more again.

**BOSS**   **B**ruker **O**rthogonal **S**him **S**ystem, is the system of small electromagnetic coils arranged in the bore of the magnet around the upper part of the probehead where the sample is sited.

Note that we have to types of BSMS being slightly different concerning function and design:
**BOSS 1 on DPX 300**
(18 shims available, which are on-axis: Z1, Z2, Z3, Z4, Z5, Z6 and off-axis: X, Y, XZ, YZ, XZ2, YZ2, XZ3, YZ3, X2-Y2, XY, X3, Y3)

**BOSS 2 on DPX 200, DRX 500 and AV 600**
(28 shims available, which are on-axis: Z1, Z2, Z3, Z4, Z5, Z6 and off-axis: X, Y, XZ, YZ, XZ2, YZ2, XZ3, YZ3, XZ4, YZ4, X2-Y2, XY, (X2-Y2)Z, XYZ, (X2-Y2)Z2, XYZ2, (X2-Y2)Z3, XYZ3, X3, Y3, X3Z, Y3Z)

**Differences regarding on-axis shims (Z1, Z2 etc.):**

On BOSS 2 you have to activate the key **ONAXIS** before you activate the on-axis shims.
Z1, Z2, Z3, Z4, Z5 are available as $1^{st}$ function.
Z6 is a $2^{nd}$ function, e.g. the **orange key marked "2$^{nd}$"** has to be activated before being able to use this function. $2^{nd}$ functions are marked under the keys.

On BOSS 1 the **ONAXIS** key does not exist. Here you activate the shim keys directly (Z, Z2, Z3, Z4 as $1^{st}$ function, Z5 and Z6 as $2^{nd}$ function)

**Differences regarding off-axis shims (X, Y etc.)**

On BOSS 2 you have to press first the key for the respective off-axis shim and then the corresponding Z-key.
For ex., if you want to shim "X", first press **X**, then **Z0.**
To shim "XZ", first press **X**, then **Z1.**
In other words: All shims are combined $1^{st}$ function.

On BOSS 1 you activate the shim keys directly, either $1^{st}$ or $2^{nd}$ function.

**HPPR**          High **P**erformance **Pr**eamplifier

**LED**          **L**ight **E**mitting **D**iode

## PREREQUISITES:

1. Lock yourself into the computer, call up **XWIN-NMR 3.5**, define a new dataset (**edc**), select a parameter set for proton experiment (**rpar PROTON all**) and get all probehead and solvent dependent parameters (**getprosol**).

2. Insert your sample into the magnet.

   Standard sample used for shimming: 10% $CHCl_3$ in acetone-d6 on DPX 200
   3% $CHCl_3$ in acetone-d6 on DPX 300
   1% $CHCl_3$ in acetone-d6 on DRX 500
   1% $CHCl_3$ in acetone-d6 on AV 600

   These are the Bruker standard test samples for which instrument and probehead specifications are defined. Line shape data and test spectra for comparative quality control do exist.
   The shape of the $CHCl_3$ single line resonance signal is observed.

   It is, of course, also possible to shim on any other sample.
   In unknown samples you should observe the solvent signal. This because you <u>know</u> how this signal is supposed to look like.

3. Check on the BSMS if the sample is **DOWN** (which is the case when the green light above the display window is on)

4. Check if the magnet is lifted up on its pneumatic vibration damping cushions. If not, move the switch on the small box beside the magnet to "On" and wait one minute for the magnet to rise.

5.  Check if there are any (big) movable, objects within the 5 Gauss line of the magnet that do not belong there. If yes, remove them. This is especially important for objects made from or containing magnetic, ferrous materials.

6.  Check sample temperature (the temperature is affecting the viscosity of the solvent and has therefore an influence on signal linewidth):

    Type **edte** ↵ (**ed**it **te**mperature).

    The Edte window comes up. Check the actual sample temperature (green). This should be 25 ºC / 298 K.
    If needed, change target temperature (yellow) to the right value and wait until the sample temperature (green) has stabilised. Click on **File / Exit** to close the Edte window.

7.  **Tuning and matching the probehead:**

    The last prerequisite is tuning and matching (the so-called wobbling) of the probehead.

    This procedure is normally not needed for DPX 200 and DPX 300 since the resonance frequency and impedance of the probeheads swing circuit are nearly not affected by the sample at these relatively small fields.
    **More important: We do not want you to touch the probehead on DPX 200 and DPX 300 in order to avoid damage or wear and tear !!!**

    At higher fields (DRX 500 and AV 600) wobbling is essential.

8.  Check on the BSMS if the sample **SPIN** is **OFF** (which is the case if the green light is not on).

9.  Type **acqui** ↵ to switch to the XWIN-NMR acquisition menu.

10. Type **wobb** ↵ manual tuning and matching of the probehead

    (The wobb procedure is for manual optimization of the probeheads resonance frequency (tuning) and impedance (matching). Exact tuning and matching is needed in order to get the full transmitter power transferred to the probehead. If probehead and transmitter are off-resonance you get a bad signal to noise or, in case it is the decoupled nucleus which is mistuned only incomplete decoupling).

    After some time a V-shaped signal, the so-called wobble curve appears on the XWIN-NMR data window. At the centre of the window you see a vertical line, marking the ^{1}H resonance frequency.

    (If you do not see a V-shaped signal, it lies outside the data window.
    In this case click on **wobb-SW.** A small window appears. Do **not** change to a new centre frequency in the first option coming up! Press ↵ In the next option change the wobble sweep width to some higher value, for ex. 10 (or 20). Press ↵ and wait some time for the program to update. If you still do not see the V-shaped signal, increase **wobb-SW** even further).

    Go to the magnet and have a look at the probehead (which is protruding from the centre down under the magnet, directly under the red-eloxated plate).

You will see two rods marked with yellow colour code. The longer one marked "T" (for tuning) the shorter one marked "M" (for matching).
Take the red-eloxated tool to be found on top of the HPPR (the preamplifier box) beside the magnet. Connect the tool with its open end to the end of the tuning rod (long, "T"). **Exactly vertical – don't bend!!!**
Turn the rod **slowly** and **carefully** in one direction while observing the V-shaped signal on the data screen:

(Alternatively you may observe the arrays of light diodes on top of the HPPR where the horizontal array represents tuning, the vertical array matching. The vertical array is also representing the $^1$H resonance frequency. The red arrows indicate in which direction you are tuning/matching)

Is the edge of the signal moving closer to the vertical line in the centre of the data window?
If yes, continue to turn the rod in the same direction.
If no, turn it the other direction.
Try to place the edge of the wobb signal on top of the vertical line, or at least as close as possible.
Next, you connect the tool to the matching rod (short, "M") and by turning it (again: **slowly** and **carefully**, please) in one or the other direction try to bring the edge of the wobble signal as close to the bottom scale as possible.
Check the tuning! The edge of the signal has probably migrated away from the vertical line again.
Tuning and matching are interconnected, and you have to tune and match alternately until the edge of wobb signal is lying exactly on the vertical line and is touching the bottom scale at the same.
(On the HPPR this final condition is indicated when you see only green lights in horizontal and vertical direction).

11.   Type **stop ↵** to stop the wobb procedure.

## SHIMMING ON-AXIS (Z) SHIM GRADIENTS -
## STRAIGHT FORWARD APPROACH, WHEN SHIMS ARE ALREADY CLOSE TO
## CORRECT ADJUSTMENT (STEP BY STEP):

The aim of the shim procedure is to homogenize the magnetic field at the site of the sample as much as possible.

The degree of homogeneity is measured by the height of lock level and/or the numerical value of the lock gain.

The higher you are increasing the lock level, the smaller will be the corresponding lock gain for a certain vertical position of the lock signal on the lock display (take the uppermost horizontal grid line as a reference).

In other words:

**Try to increase the lock level as much as possible in order to decrease the lock gain as much as possible.**

There are two different approaches for shimming the first order shim gradients.

The first one, which is very simple and straightforward, applies when the shims are close to correct adjustment already (which they are in most cases):

1. Log yourself into the computer, call up **XWIN-NMR 3.5**, define a new dataset (**edc**), select a parameter set for proton experiment (**rpar PROTON all**) and get all probehead and solvent dependent parameters (**getprosol**).

2. Type **ased ↵** (or **eda ↵)** and change the following acquisition parameters:

   **PULPROG: zg**
   **NS: 4**
   **TD: a multiple of 16k which results in a AQ larger than 3 s**
   (TD – *t*ime *d*omain – and AQ – *a*cquisition *t*ime – are interconnected. Changing one is changing the other automatically.

   Type **edp ↵** and change the following processing parameter:

   **SI: 4 times the value of TD**
   **LB: 0.3 Hz**

3. Insert your sample. (See above under **PREREQUISITES** for remarks about standard sample)

4. Check if the sample is really down.
   Check sample temperature.
   Check if magnet stand is lifted up.
   Check for movable magnetic objects within the 5 Gauss line of the magnet.
   (For more information, if required see in chapter about **PREREQUISITES** above)

5. Perform tuning and matching (**only** on DRX 500 and AV 600).

(For how to tune and match a probehead see page 9, point 7. to 11. in chapter about **PREREQUISITES** above).

6. Type **lockdisp** ↵ to get up the *lockdisp*lay

7. Press **SWEEP RATE,** e.g. first **the orange key marked 2ⁿᵈ**, then **SWEEP AMPL.**
   Use the wheel to adjust the sweep rate to **0.50**
   Press **STD BY** to store this value.

8. Type **lock** ↵ to *lock* the magnetic field.
   Select **Aceton(e)** (or the appropriate solvent which you used to solve your sample) from the list of solvents. Wait until the lock procedure has finished.

   If the lock signal should be unstable, that means going up and down in waves, the deuterium signal is saturated.
   In that case press on **LOCK POWER** and change the given value by using the wheel about 10 units to more negative values.

9. Adjust the main field:

   Switch **LOCK** to **OFF**
   Press **SWEEP.**
   Click the middle icon on top of the lock display (Maximize) to expand the lock display over the whole screen
   Press **FIELD**.
   Use the wheel to adjust the sweep signal in that way that the green and the red signal are crossing each other on the <u>vertical</u> grid line in the <u>centre</u> of the display.
   Press **STD BY** to store the new field adjustment.
   Click the middle icon on top of the lock display (Restore Down) to minimize the lock display again.
   Switch the **LOCK** to **ON** again.

10. Type **rsh** ↵ to *r*ead a set of *sh*imfiles.
    Select the most recently updated shim file belonging to the probehead that is inside the magnet.
    Usually you will find this file on top of the list (for ex. **QNP.040509.luf**, meaning *l*ast *u*pdated shim file for probehead *QNP*, created on **9ᵗʰ** of ***May*** 20***04***)
    You are going to use this shim file as a base platform for your own shimming.
    It contains a good set of shim gradients, which make the field already quite homogeneous.
    On base of this shim file you normally have to adjust only the shim gradients Z1 and Z2 (maybe Z3 and Z4) for your own specific sample.

11. Switch sample **SPIN** to **ON**
    (Which is the case if the green light on the button is **on**)
    and wait until the green light has stopped blinking, meaning the sample is spinning at its defined rate.
    (In case the light does not stop blinking most likely the spinner turbine is plugged with dirt. Contact the lab engineer for help).

Normally the sample should spin at 20 rounds per second. This is defined in the acquisition parameter file (eda) by the parameter **ro**.

You may check the spin rate on the BSMS by pressing first the **orange key marked 2ⁿᵈ** and then the key **SPIN RATE**. You have now activated the 2ⁿᵈ function **SPIN MEASUREMENT**: The value shown on the display to the left is the previous set spin rate. The value to the right is the actual spin rate.
Press **STD BY** to get out of this menu.

12. Press **LOCK GAIN** on the BSMS and adjust the lock signal to the uppermost red horizontal grid line by turning the wheel.
    (In case the lock signal is not seen at all it is beyond the upper limit of the lock display. Change the LOCK GAIN to smaller values to make it visible).
    Press **STD BY** to save the input and finish the function.

13. Press **LOCK PHASE** on the BSMS and try to increase the height of the lock signal by turning the wheel in one or the other direction.
    If you made an improvement press **STD BY** to save the new input and finish the function. Use **LOCK GAIN** to set the lock signal back on the uppermost horizontal grid line.
    If you were not able to make an improvement press **LOCK PHASE** again to restore the old value.

14. Press **ONAXIS** (on BOSS 2 only, to be found on DPX 200, DRX 500 and AV 600, this key is not to be found on BOSS 1, e.g. DPX 300 )

15. Press Z1 (on BOSS 2). Press Z (on BOSS 1) for shimming Z1 shim gradient

    Turn the wheel in one or the other direction in order to increase the lock signal as much as possible.
    If necessary, adjust **LOCK GAIN** in between to get the lock signal back into the visible range.
    After having found the maximum level for the lock signal use **LOCK GAIN** to readjust the lock level back to the uppermost horizontal grid line.
    **(Do always finish the adjustment of a single shim parameter by setting the lock signal back on the uppermost horizontal grid line.**
    **Make it yourself a habit! It is not strictly required, but the grid line works as a holding point or reference and helps you to decide whether there really is an improvement of the lock level or not.**
    **This will only be mentioned again one more time in the further procedure!)**

16. Press Z2 for shimming Z2 shim gradient.

    Turn the wheel in one or the other direction in order to increase the lock signal as much as possible.
    If necessary, adjust **LOCK GAIN** in between to get the lock signal back into the visible range.
    After having found the maximum level for the lock signal use **LOCK GAIN** to readjust the lock level back to the uppermost horizontal grid line.
    (Do this always after the adjustment of a single shim parameter. This will not be mentioned again in the further procedure!)

17. Return to Z1 and shim to maximum lock level.

18. Again go back to Z2 and shim to maximum lock level

19. Shim Z1 and Z2 iteratively (changing from one to the other) until the lock level cannot be improved any further.

20. Finish the procedure with $\mathbf{Z^1}$ (which is the shim parameter of highest priority).

    **Note that all the shims do influence each other. As a rule, whenever a gradient is altered <u>all</u> lower order gradients will need to be readjusted. That means, any change of the second order shim $\mathbf{Z^2}$ does require an adjustment of the first order shim $\mathbf{Z^1}$ afterwards, since that one has higher priority.**

21. You have now performed the shim procedure that should be done **at <u>any</u> case on every new sample before an acquisition is started.**
    At any case means even if you started from a freshly created, nearly perfect shim file (see point 10.). This is because the shims $Z^1$ and $Z^2$ are influenced by properties belonging to the specific sample, like concentration, solvent, total volume, glass type of the tube, quality of the tube etc.

    Acquire a spectrum to see how the signals look like:

    Type **rga** ↵ for *r*eceiver *g*ain *a*djustment.
    Type **zg** ↵ to acquire a spectrum
    Type **acqu** ↵ to get into the acquisition menu.
    > Have a look on the FID (*f*ree *i*nduction *d*ecay). You will already get an idea bout the field homogeneity. Ideally the FID will die down as a perfect exponential function.

    Type **efp** ↵ to Fourier transform the FID into the spectrum
    Type **apk** ↵ for *a*utomatic *p*hase correction
    Type **abs** ↵ for *a*utomatic *b*aseline correction

    Expand the spectrum around the $CHCl_3$ signal at ca. 8 ppm.
    Include the $^{13}C$ satellites (at about 106.5 Hz symmetrical on both sides of the signal) into the expansion.
    Click on **dp1** in the XWIN-NMR window and press ↵ 3 times to define the expansion as the plotregion.
    You can call up this spectral region easily at any time during the further process by clicking **PlotReg.**

22. Have a look on she signal and compare it with the figure below:
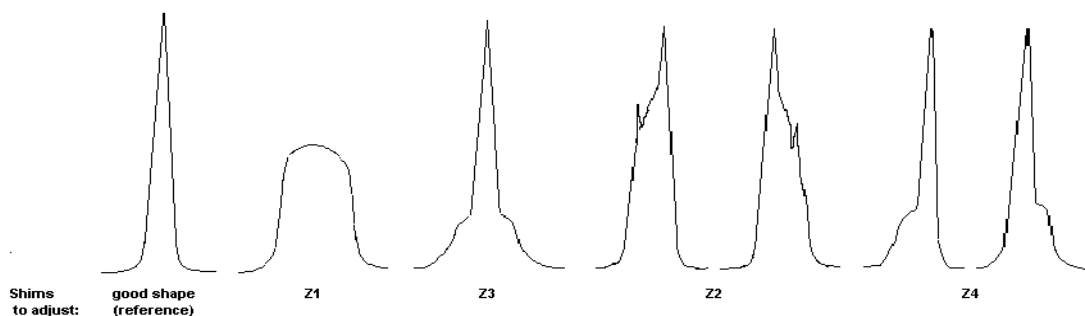


If you got a symmetrical broad signal it does most likely Z1 have to be adjusted further.
A symmetrical broadening at the base of the signal indicates that $Z^3$ needs adjustment.
An unsymmetrical broadening which starts already in the upper part of the signal (and in addition maybe displays some minor peaks) indicates that $Z^2$ has to be adjusted.
An unsymmetrical broadening or shoulder close to the base of the signal indicates that $Z^4$ is wrongly adjusted.

In addition you most likely will see $1^{st}$ order spinning sidebands at 20 Hz distance symmetrically on either side of the signal.
May be as well $2^{nd}$ order or even $3^{rd}$ and $4^{th}$ order spinning side bands at 40, 60 and 80 Hz distance are seen (although probably not at 200 and 300 MHz).
Remember that the spinning side bands are multiples of the spinning speed (expressed in Hertz).
Do not care about sidebands at this point. They are due to bad adjusted off-axis shims and will be corrected later.

23. **Check the line <u>width</u>:**

In case you already got a symmetrical signal of relative small width you might perform a quality check by using a standard procedure:

Type: **humpcal ↵** .After some time of calculation a window appears, which gives you both the linewidth at half signal height (LW) as well as the linewidth at 0.55% and 0.11% signal height (HUMP).

Note the values down on paper or type **setti ↵** (*se*t *ti*tle) and store the information into the title bar.

The linewidth at 0.55% is not chosen arbitrary as a quality measure.
The percentage equals to the relative height of the $^{13}C$ satellite signals of the $CHCl_3$ line. It further corresponds to about 13.5 times the value for the half line width (LW) of an ideally and perfectly shaped (e.g. theoretical) signal, expressed by the so-called Lorentzian line.
The value at 0.11% is set to one-fifth of that value, corresponding to 30 times the value for the half line width (LW) of a Lorentzian line.
You often will obtain 0.55% and 0.11% linewidths that are **narrower** than the ideal calculated linewidth of a Lorenzian line. Strictly speaking, this is as much an incorrect

line shape as one that is too broad. But since the resolution is affected positively, it is not worth to care about it.

If the quality of the spectrum is not quite good enough at this point the program will not be able to calculate the hump. You will get a corresponding message in that case.

If you want, you can check LW and HUMP manually:

Click on **PlotReg** in XWINNMR window. Click on the **left grid** choice.
Place the spectral baseline on the lowermost gridline.
Place the top of the signal on the uppermost gridline.
If necessary, click on **YU** to change the unit of the Y-axis from abs- to cm-mode.
Click on **utilities**, then **CY.** Place the cursor on the uppermost gridline, click with left mouse button and enter **100** (cm = %) into the upcoming window.
Click on **return** (to get back to the 1D main menu).
Expand the signal to be filling ca. one-third of the window.
Click left mouse button and place the arrow on the right side of the signal as close to the 50%-grid line as possible.
Click middle mouse to get the arrow fixed.
Click left mouse again and place the 2$^{nd}$ arrow on the opposite side of the signal as close as possible to the 50%-grid line. Do **not** click the middle mouse button.
The **Info window** shows you the half line width in Hz.
Increase the vertical intensity of the signal until you get to a suitable range that enables you to measure the hump at 0.55% and 0.11% signal height.

24. **Check the line <u>shape</u>:**

At this point you could also get a closer look on the line shape:

Expand the signal to be filling ca. one-third of the window.
Place the spectral baseline on the lowermost gridline.
Place the top of the signal on the uppermost gridline.
Press the middle mouse button, keep it pressed and move the top of the signal exactly on the centre vertical grid line. Release the button.
Press the right mouse button, keep it pressed and expand the signal in the way that is passing the second horizontal grid line (seen from the top of the window) at the cross point with the nearest vertical grid lines.
Use the pressed middle mouse again to adjust the signal to the centre position, if necessary.
Use both mouse buttons iteratively (changing from one to the other) until you have got the signal exactly on the centre vertical grid line, while at the same time it is passing at the cross point of the second horizontal grid line with the nearest vertical lines.
Examine the course of the signal going down to its base (use **\*2**, **/2**, __, __) while comparing both sides of the signal. Ideally the centre vertical grid line should work like a mirror axis.

25. Press **LOCK PHASE** and adjust it to maximum lock level

26. Type **wsh** ↵ (*w*rite a set of *sh*imfiles)
    to store your newly adjusted shim parameters into a new shim file.
    That way you can easily get back to your last good shim file later, if needed
    (by command **rsh** ↵)

    The name of the shim file should for practical reasons look similar to the following
    example: QNP041022_1DP
    where the first abbreviation is characterising the type of probehead the shimset belongs
    to (for ex. QNP, BBO, TXI etc.), followed by the date the file was created
    (year/month/day), followed by the experiment number for the spectrum your shim file
    belongs to, and finally your initials.
    You are, of coarse, free to create your own system for the name of the shim file, but
    **it <u>has</u> either to start or to end with your personal initials.** That is for identification.
    Note that only up to 15 characters are shown in the shim file list.

27. Press $Z^3$ for shimming $Z^3$ shim gradient.
    Try to increase the lock level by turning the wheel in one or the other direction.
    If there is no improvement go to the point 28.
    If there is an improvement shim $Z^2$ to maximum lock level, thereafter $Z^1$, then again $Z^2$,
    then finally $Z^1$.
    Repeat the loop ($Z^3$, $Z^2$, $Z^1$, $Z^2$, $Z^1$) until there is no more change for $Z^3$.
    **Again, note that all the shims do influence each other. As a rule, whenever a
    gradient is altered *all* lower order gradients will need to be readjusted. That
    means, any change of the third order shim Z3 does require an adjustment of the
    second order shim Z2 and the first order shim Z1 afterwards, since these have
    higher priority.**

    Check **LOCK PHASE**.

28. Type **i** ↵ (*i*ncrease experiment number) to get to Exp. No. 2
    All experiment parameters are copied into Exp. No 2 as well.
    Acquire a spectrum (**rga**, **zg**, **efp**, **apk**, **abs**) to see how the signal looks like.
    Click on **PlotReg** to get up the suitable expansion directly.

    In order to compare this spectrum with the one you took in Exp. No. 1 before,
    Click on **dual** in the XWIN-NMR window.
    A second spectrum (violet colour) is superimposed on the green one. Click on _ under
    "**spectrum2:**" to make it visible. This spectrum is identical with the one you just
    acquired in Exp. No. 2 (see small **Info window**).
    Click on **e_** under "**2$^{nd}$**". The second spectrum (still violet) is now the one you took in
    Exp. No.1, after shimming $Z^1$ and $Z^2$. Expand the signal(s) to a suitable range, if
    necessary.
    Click **return** to get out of the dual menu.

    Check half height linewidth (LW) and HUMP (**humpcal** ↵)

    Note the values down on paper or type **setti** ↵ (*se*t *ti*tle) and store the information into
    the title bar.

29. Type **wsh** ↵ and store your newly adjusted shim parameters into a new shim file.

30. Press $\mathbf{Z^4}$ for shimming $Z^4$ shim gradient.
Try to increase the lock level by turning the wheel in one or the other direction.
If there is no improvement go to point 31.
If there is an improvement shim $\mathbf{Z^3}$ to maximum lock level, thereafter $\mathbf{Z^2}$, then $\mathbf{Z1}$, then again $\mathbf{Z^3}$, $\mathbf{Z^2}$ and finally $\mathbf{Z^1}$.
Repeat the loop ($\mathbf{Z^4}$, $\mathbf{Z^3}$, $\mathbf{Z^2}$, $\mathbf{Z^1}$, $\mathbf{Z^3}$, $\mathbf{Z^2}$, $\mathbf{Z^1}$) until there is no more change for $\mathbf{Z^4}$.

   Check **LOCK PHASE**.

31. Type **i** ↵ (*i*ncrease experiment number) to get to Exp. No. 3
Acquire a spectrum (**rga**, **zg**, **efp**, **apk**, **abs**) to see how the signal looks like.
Click on **PlotReg** to get up the suitable expansion directly.

   Superimpose the spectrum from Exp. No 2 and compare.

   Check half height linewidth (LW) and HUMP (**humpcal** ↵)

   Note the values down on paper or type **setti** ↵ (*se*t *ti*tle) and store the information into the title bar.

32. Type **wsh** ↵ to store your new set of shim parameters.

33. Following the logic of the system at this point you would have to optimize the lock level further by first adjusting $\mathbf{Z^5}$, thereafter $\mathbf{Z^6}$ in loops corresponding to the procedure described above:

   $\mathbf{Z^5}$ ($\mathbf{Z^5}$, $\mathbf{Z^4}$, $\mathbf{Z^3}$, $\mathbf{Z^2}$, $\mathbf{Z^1}$, $\mathbf{Z^4}$, $\mathbf{Z^3}$, $\mathbf{Z^2}$, $\mathbf{Z^1}$)
   $\mathbf{Z^6}$ ($\mathbf{Z^6}$, $\mathbf{Z^5}$, $\mathbf{Z^4}$, $\mathbf{Z^3}$, $\mathbf{Z^2}$, $\mathbf{Z^1}$, $\mathbf{Z^5}$, $\mathbf{Z^4}$, $\mathbf{Z^3}$, $\mathbf{Z^2}$, $\mathbf{Z^1}$)

   In praxis shimming $Z^5$ and $Z^6$ is nearly never required.
   Just the opposite: you would most likely ruin the whole shim set by touching this shims.
   Skip it!

   If you at this stage got a sufficiently **narrow** and **symmetrical** signal (compare chapter **RESULTS / CRITERIA FOR GOOD SHIMMING**, page 24), proceed with shimming the off-axis shim gradients (X/Y) (go to page 21).

   **Sometimes, that is if the shims are too far off from correct values, you get to a "dead-end".**
   **You might have adjusted all on-axis gradients to maximum, but no matter how hard you try, your signal will not improve regarding to its width and/or shape.**
   **If your signal is still broad and unsymmetrical at this stage, try to shim using the iterative approach described in the next chapter.**

   Have a look on the line shape. If the irregularities are limited to the base of the signal it might be necessary only to shim either $Z^3$ or $Z^4$ iteratively.


## SHIMMING ON-AXIS (Z) SHIM GRADIENTS –

## ITERATIVE APPROACH, WHEN SHIMS ARE FAR OFF FROM CORRECT ADJUSTMENT (STEP BY STEP):

The second approach applies when the shims are far away from correct adjustment:

1.  Switch sample **SPIN** to **ON**.

2.  **Shimming $Z^1$:**

    Adjust $Z^1$ to obtain the highest lock level.
    Use **LOCK GAIN** to readjust the lock level back to the uppermost horizontal grid line and note the corresponding value for **LOCK GAIN** for later comparison.

3.  **Shimming $Z^2$:**

    Note the present values of $Z^1$ and $Z^2$.

    Now alter $Z^2$ by a small amount in *one direction* (turning the wheel either clockwise or anti-clockwise) until something happens to the lock level *without regard for what it is.* In other words, do not worry if the level falls or rises at that stage, as long as *something* is happening. Once the lock level has definitely changed one way or the other, readjust $Z^1$ for maximum signal.
    Use **LOCK GAIN** to readjust the lock level back to the uppermost horizontal grid line and note the corresponding value for **LOCK GAIN**.

    If the new combination of $Z^1$ and **Z2** did lead to a <u>higher</u> lock level (that means <u>lower</u> **LOCK GAIN** at the level of the uppermost horizontal grid line) compared with the level you obtained by optimizing $Z^1$ alone (refer point 1.), continue changing $Z^2$ *in the same direction* and readjusting $Z^1$ for maximum signal.
    Continue this iterative procedure (changing $Z^2$ into same direction and maximizing $Z^1$) until the overall maximum lock level is found**.**

    If the new combination of $Z^1$ and $Z^2$ did result in a <u>lower</u> lock level (that means <u>higher</u> **LOCK GAIN** at the level of the uppermost horizontal grid line) compared with the level you obtained by optimizing $Z^1$ alone, readjust $Z^1$ back to the optimized value which you got in the first step (at point 1.) and $Z^2$ back to the initial value.
    Now alter $Z^2$ into the *opposite direction* and follow the procedure described above.

4.  Acquire a spectrum with the new shim values.
    Check HUMP and line shape like described above.
    Have a look on she signal and compare it with the figure under point 22.on page xx.

    Type **wsh ↵** to store your new set of shim parameters, if required.

    If line shape and HUMP are still not satisfying you will also have to shim $Z^3$ and/or $Z^4$.

5.  **Shimming $Z^3$:**

Once $Z^1$ and $Z^2$ have been adjusted $Z^3$ is added to the loop.

Note the actual values for $Z^1$, $Z^2$, and $Z^3$.

Use **LOCK GAIN** to readjust the lock level back to the uppermost horizontal grid line and note the corresponding value for **LOCK GAIN**.
Change $Z^3$ a small amount in one direction (turning the wheel either clockwise or anti-clockwise).
The lock level might drop. Don't care in the first place.
Maximize the lock level using first $Z^2$, then $Z^1$.
If this new maximum is an overall improvement (which means you get a <u>lower</u> value for **LOCK GAIN** at the level of the uppermost horizontal grid line compared to the one you have been starting with), continue changing $Z^3$ in the same direction and maximizing the lock level with $Z^2$ and $Z^1$.
Continue this iterative procedure (changing $Z^3$ into same direction and maximizing $Z^2$, then $Z^1$) until the overall maximum lock level is found**.**

If you don't get an overall improvement, go back to the original values for **Z1**, **Z2** and **Z3** and change **Z3** a small amount into the opposite direction and follow the procedure described above.

6.  Acquire a new spectrum.
    Check HUMP and line shape.

    Have a look on she signal and compare it with the figure under point 22.on page xx.
    If you still observe an asymmetry or tailing close to the base of the signal you should proceed further by shimming $Z^4$ iteratively.

    Type **wsh ↵** to store your new set of shim parameters, if required.

7.  **Shimming Z4:**

    Once $Z^1$, $Z^2$ and $Z^3$ have been adjusted successfully to obtain the highest lock level possible, $Z^4$ can be added to the loop.

    Shimming $Z^4$ is done in the same way like shimming $Z^3$:
    Note the actual values for $Z^1$, $Z^2$, $Z^3$ and $Z^4$.
    Use **LOCK GAIN** to readjust the lock level back to the uppermost horizontal grid line and note the corresponding value for **LOCK GAIN**.
    Change $Z^4$ some amount in one direction (turning the wheel either clockwise or anti-clockwise).
    As a rule, which applies in most cases, $Z^4$ has to be changed into the same direction relative to the unsymmetrical distortion of the signal. That means, if the shoulder is on the right side of the signal, turn the wheel clockwise and vice versa.
    Be aware that $Z^4$ is less sensitive than $Z^3$, so that it often has to be changed some larger amount.
    The lock level might drop.
    Maximize the lock level using first $Z^3$, then $Z^2$, then $Z^1$**.**
    In case of overall improvement continue in the same direction.

Otherwise try opposite direction.

Once $Z^1$, $Z^2$, $Z^3$ and Z4 have been adjusted successfully to obtain the highest lock level possible, your signal obtained under spinning conditions hopefully will resemble a Lorenzian line with a symmetrical line shape and an acceptable HUMP (compare chapter **RESULTS / CRITERIA FOR GOOD SHIMMING**, page 24).
In that case proceed with shimming the off-axis shim gradients (X/Y):

## SHIMMING OFF-AXIS (X, Y) SHIM GRADIENTS (STEP BY STEP):

**Before you start, some general information about**
**How to activate the off-axis shim keys on BOSS 1 (DPX 300) and BOSS 2**
**(DPX200, DRX 500 and AV 600):**

On **BOSS 2** you have to press first the key for the respective off-axis shim and then the corresponding Z-key.
For ex., if you want to shim "X", first press **X**, then $\mathbf{Z^0}$.
To shim "XZ", first press **X**, then $\mathbf{Z^1}$.
In other words: All shims are combined 1$^{st}$ function.

On **BOSS 1** you activate the shim keys directly, either 1$^{st}$ or 2$^{nd}$ function.

1. Switch sample **SPIN** to **OFF**
   (This is the case if the green light on the button is off).

   In case the off-axis shims are badly adjusted the lock level will drop considerably. Another indication for misadjusted off-axis shims are, like mentioned before, large spinning side bands in the spectra you acquired above while adjusting the on-axis shims.
   Spinning side bands should ideally have a height less than 0.1% of the main signal, e.g. considerably below the height of the $^{13}$C satellites.

2. Shim $\mathbf{Z^1}$ and $\mathbf{Z^2}$ iteratively to maximum lock level like described above. (While spinning, the sample is lifted up 1-2 mm by the bearing air stream of the spinner turbine. After spinning is switched off the sample drops to the bottom of the probehead. The difference of sample depth makes it necessary to readjust the on-axis gradients $\mathbf{Z^1}$ and $\mathbf{Z^2}$ before starting to shim the off-axis shim gradients).

3. Press **X**, then $\mathbf{Z^0}$ (on BOSS 2) / Press **X** (on BOSS 1)
   For shimming X shim gradient.
   Shim to maximum lock level.

4. Press **Y**, then $\mathbf{Z^0}$ (on BOSS 2) / Press $^Y$ (on BOSS 1)
   For shimming Y shim gradient.
   Shim to maximum lock level.

5. Press **ONAXIS** (on BOSS 2 only), to be found on DPX 200, DRX 500 and AV 600, this key is not to be found on BOSS 1, e.g. DPX 300 )

6. Press $\mathbf{Z^1}$ (on BOSS 2) / Press **Z** (on BOSS 1) for shimming $Z^1$ shim gradient.

7. Press **X**, then $\mathbf{Z^1}$ (on BOSS 2) / Press **XZ** (on BOSS 1)
   For shimming XZ shim gradient.
   Shim to maximum lock level.

8  Press **Y**, then $\mathbf{Z^1}$ (on BOSS 2) / Press **YZ** (on BOSS 1)
   For shimming YZ shim gradient.
   Shim to maximum lock level.

9. Readjust first **X**, then **Y** to maximum lock level.

10. Press **X**, then $\mathbf{Z^2}$ (on BOSS 2) / Press $\mathbf{XZ^2}$ (on BOSS 1, first orange 2nd key, then XZ)
    for shimming $XZ^2$ shim gradient.
    Shim to maximum lock level.

11. Press **Y**, then $\mathbf{Z^2}$ (on BOSS 2) / Press $\mathbf{YZ^2}$ (on BOSS 1, first orange 2nd key then YZ)
    for shimming $YZ^2$ shim gradient.
    Shim to maximum lock level.

12. Readjust first $\mathbf{XZ^1}$, then $\mathbf{YZ^1}$, followed by **X**, then **Y** to maximum lock level.

13. Press $\mathbf{X^2\text{-}Y^2}$, then $\mathbf{Z^0}$ (on BOSS 2) / Press $\mathbf{X^2\text{-}Y^2}$ (on BOSS 1, first orange 2nd key then Y) for shimming $X^2\text{-}Y^2$ shim gradient.
    Shim to maximum lock level.

14. Press **XY**, then $\mathbf{Z^0}$ (on BOSS 2) / Press **XY** (on BOSS 1, first orange 2nd key then X) for shimming XY shim gradient.

15. Readjust first **X**, then **Y** to maximum lock level.

16. Check **LOCK PHASE.**

17. Type **wsh** ↵ to store your new set of shim parameters

18. Shim the following loop (which is a summary of points 35 to 48.) several times until you are not able to increase the lock level anymore:

    1. $Z^1$
    2. $Z^2$, $Z^1$
    3. X, Y, $Z^1$
    4. $XZ^1$, $YZ^1$, X, Y
    5. $XZ^2$, $YZ^2$, $XZ^1$, $YZ^1$, X, Y
    6. $(X^2\text{-}Y^2)$, XY, X, Y

    Check **LOCK PHASE** after every loop.
    Store your new set of shim parameters after every loop

19. Type **i ↵** (*i*ncrease experiment number) to get to Exp. No. 4
Acquire a spectrum (**zg**, **efp**, **apk**, **abs**) to see how the signal looks like.
Click on **PlotReg** to get up the suitable expansion directly.

Check half height linewidth (LW) and HUMP (**humpcal ↵**)

Note the values down on paper or type **setti ↵** (*se*t *ti*tle) and store the information into the title bar.

**You will observe that both the values for the half line width and the hump have increased. Nothing to worry about: This is because you were not spinning the sample. (More about sample spinning see above).**

Check the line shape. (See point 24 on page 24 if you do not remember how to do this).

20. Switch sample **SPIN** to **ON**.

The lock level will increase.
If the off-axis shims have been shimmed correctly, the lock level should not increase very much (more about that later in this manual).

21. Shim $Z^1$ and $Z^2$ iteratively to maximum lock level.

22. Type **i ↵** (*i*ncrease experiment number) to get to Exp. No. 5
Acquire a spectrum (**zg**, **efp**, **apk**, **abs**) to see how the signal looks like.
Click on **PlotReg** to get up the suitable expansion directly.

Check if the spinning side bands are reduced to a sufficient degree.

Check half height linewidth (LW) and HUMP (**humpcal ↵**)

Note the values down on paper or type **setti ↵** (*se*t *ti*tle) and store the information into the title bar.

Check the line shape.

## RESULTS / CRITERIA FOR GOOD SHIMMING:

The half height line width (LW) of the CHCl3 signal with sample spinning should now have a value between 0.5 and 0.6 Hz.

The HUMP should be about 13.5 times larger at 0.55% signal height (may be less, but not more) and about 30 times larger at 0.11% signal height (may be less, but not more).

Spinning side bands of 1st order (at 20 Hz) should be reduced to about 0.1% of the main signal height.
Spinning side bands of higher order (at 40, 60, 80 Hz) should not be visible at all.

The half height line width (LW) without sample spinning should be about 0.05 - 0.1 Hz larger than the LW under spinning conditions. The HUMP should correspond to that value.

When switching from spinning to non spinning the lock signal should not drop more than the distance between two horizontal grid lines on the lock display.
This corresponds to a difference of two units at maximum for the lock gain at spinning – non spinning, measured at the uppermost horizontal grid line of the lock display.

Both signals (spinning and not spinning) should be almost perfectly symmetrical from the top to the base.

There might be some artefacts to be found in the spectrum that have nothing to do with bad shimming:
Close to the base, within a distance up to ca 11 Hz on both sides from the top of the signals (both spinning and not spinning) there might be some irregularities.
These are due to vibrations of the floor or the environment. Nothing can be done about that.
At more than 11 Hz should be no irregularities visible, since the cushions of the pneumatic damping system are eliminating all vibrations down to that frequency.
You might observe tiny "spikes" at 50 Hz distance from the top of the main signal.
They are due to induction from the normal electrical network, which is alternating at that frequency.

**In case that your results do not agree to one or several of the mentioned criteria, you should consider to repeat shimming the on-axis and/ or off-axis shim gradients.**

## SHORT SUMMERY OF SHIM OPERATION FOR MEMORY SUPPORT:

### A. Adjusting the on-axis (Z) shims (with sample rotation).
Straight forward approach for shim gradients being close to correct values:

Adjust the on-axis shims in the following order:

1. Adjust the main field, lock the system and start sample spinning.
2. Shim $Z^1$ to maximum lock level
3. Repeat the loop ($Z^2$, $Z^1$) until there is no more change for $Z^2$.
4. Repeat the loop ($Z^3$, $Z^2$, $Z^1$, $Z^2$, $Z^1$) until there is no more change for $Z^3$.
5. Repeat the loop ($Z^4$, $Z^3$, $Z^2$, $Z^1$, $Z^3$, $Z^2$, $Z^1$) until there is no more change $Z^4$


### B. Adjusting the on-axis (Z) shims (with sample rotation).
Iterative approach for shim gradients being far off to correct values:

Adjust the on-axis shims in the following way:

1. Shim $Z^1$ to maximum lock level.
   Note **LOCK GAIN** at uppermost horizontal grid line.

2. Note present values of $Z^1$ and $Z^2$.
   Misadjust $Z^2$ deliberately in one or the other direction
   Shim $Z^1$ to maximum lock level
   Note **LOCK GAIN** at uppermost horizontal grid line.
   Compare with value for previous noted **LOCK GAIN.**.
   Improvement? ➜ Continue changing $Z^2$ into same direction and maximizing lock level by readjusting $Z^1$. Continue iteratively until maximum lock level is found.
   No improvement? ➜ Misadjust $Z^2$ in opposite direction. Shim $Z^1$ to maximum etc.

3. Note **LOCK GAIN** at uppermost horizontal grid line.
   Note present values of $Z^1$, $Z^2$.and $Z^3$.
   Misadjust $Z^3$ deliberately in one or other direction.
   Shim first $Z^2$, then $Z^1$ to maximum lock level.
   Note **LOCK GAIN** at uppermost horizontal grid line.
   Compare with value for previous noted **LOCK GAIN.**
   Improvement? ➜ Continue changing $Z^3$ into same direction and maximizing lock level by readjusting first $Z^2$ then $Z^1$. Continue iteratively until maximum lock level is found.
   No improvement? ➜ Misadjust $Z^3$ in opposite direction. Shim first $Z^2$, then $Z^1$ to maximum etc.

4. Note **LOCK GAIN** at uppermost horizontal grid line.
   Note present values of $Z^1$, $Z^2$, $Z^3$.and $Z^4$.
   Misadjust $Z^4$ deliberately in one or other direction.
   Shim first $Z^3$, then $Z^2$, then $Z^1$ to maximum lock level.
   Note **LOCK GAIN** at uppermost horizontal grid line.

Compare with value for previous noted **LOCK GAIN.**
Improvement? ➔ Continue changing $Z^4$ into same direction and maximizing lock level by readjusting first $Z^3$ then $Z^2$, then $Z^1$. Continue iteratively until maximum lock level is found.
No improvement? ➔ Misadjust $Z^4$ in opposite direction. Shim first $Z^3$, then $Z^2$, then $Z^1$ to maximum etc.

**C. Adjusting the off-axis (X/Y) shims (no sample rotation**):

Adjust the off-axis shims in the following order.
Note that although Z1 and Z2 are not off-axis shims, it is necessary for them to be set correctly before adjusting the off-axis shims.

1. Stop sample spinning if necessary.
2. $\mathbf{Z^1}$
3. $\mathbf{Z^2, Z^1}$
4. $\mathbf{X, Y, Z^1}$
5. $\mathbf{XZ^1, YZ^1, X, Y}$
6. $\mathbf{XZ^2, YZ^2, XZ^1, YZ^1, X, Y}$
7. $\mathbf{(X2-Y2), XY, X, Y}$

Repeat steps 1. through 7. several times until no further increase of the lock level is observed.

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