



Technical Bulletin J0020 - CapNMR™ Probe Shimming
31 Jan 2005, AGW, MRM

This is a summary of shimming methods and techniques for CapNMR™ probes and the spectral artifacts associated with specific shims.

Shimming: Lock versus FID

There are several different ways to shim. Most users opt to shim on the lock level. This is perfectly fine. However, another way to shim, which will provide more information to the shimmer, is shimming off the FID. On a Bruker, this is done by type gs and acqu, which opens a FID window. You are also given a numerical integer value for the FID. On a Varian, this is done by clicking on the acqi window and selecting FID. Be sure that you've selected FID instead of spectrum within this window. You can actually choose how to shape the FID to give you a specific peak shape. Expanding the beginning of the FID can give you a better base line, but by extending the decay you'll have a better half height. Ideally, you'll be able to optimize both.

Ensure the flowcell is clean

You should not have to reshim if you've injecting a compound in a solvent you already have a shim set for. However, to obtain an initial shim set, it is recommended to flush though at least 200 uL of your desired solvent to ensure that your probe is well rinsed. If you've been running urine, plasma, and any raw matrix, it is also recommended to run the HCl cleaning solution through the probe to ensure the flowcell is clean. Once you are certain the flowcell has been thoroughly cleaning, inject at least 100 uL of your solvent of choice to shim up on. Once the probe is tuned, you can start shimming up.

Where to start and what sample to use

During your installation, there were two shim sets saved. The first was for the lineshape sample (5% CHCl₃ in acetone-d₆) for your organic solvents and the other was for 10 mM Sucrose in D₂O for your aqueous solvents. These are both good start points for shims however you do not want to shim on D₂O since it has such a long relaxation time. For shimming, you want to use acetone since it relaxes quickly and you pulses on it repeatedly while you shim without problems. In order to load a shim set, for Bruker type rsh and you'll be given a list of labeled shim sets to choose from and for Varian, load the lineshape file and type `load='y'su` to load the shim set associated with the file.

However, if you don't have the shims set for the install or cannot locate it, you can also start from a shim set for another 5mm or 10 mm probe that is known to be good.

Overall shimming methods

You will always want to use large steps as you start your shimming and then use smaller values once you've optimized all other shims.

The flowcell is quite small so to get the field homogeneous around that small is quite easy but you want to make large dramatic changes since they don't affect the field too greatly in the small area around the coil.

Begin Shimming Here

Start by Optimizing Z1 and Z2 (increments of 1000)

- Optimize X and Y (increments of 50)
- Optimize X2Y2 (increments of 1000)
 - Touchup Z1 and Z2 shims
- You will most likely need further Z2 and Z1 adjustments (increments of 1000)
- Adjust Z2 and X2Y2 together and optimize the pair.
 - (This sometimes requires adjusting one shim several thousand and then touching up the other complimentary shim for a net improvement.)
 - Touch up Z1 and X,Y

Initial Shimming Increments

Z1, Z2, X2Y2 Change in increments of 1000
X,Y,X3,XY,Y3,XZ2,YZ2 Change in increments of 50

Fine Shimming Increments

Z1, Z2, X2Y2 Change in increments of 200
X,Y,X3,XY,Y3,XZ2,YZ2 Change in increments of 10 (or smaller depending on sensitivity)

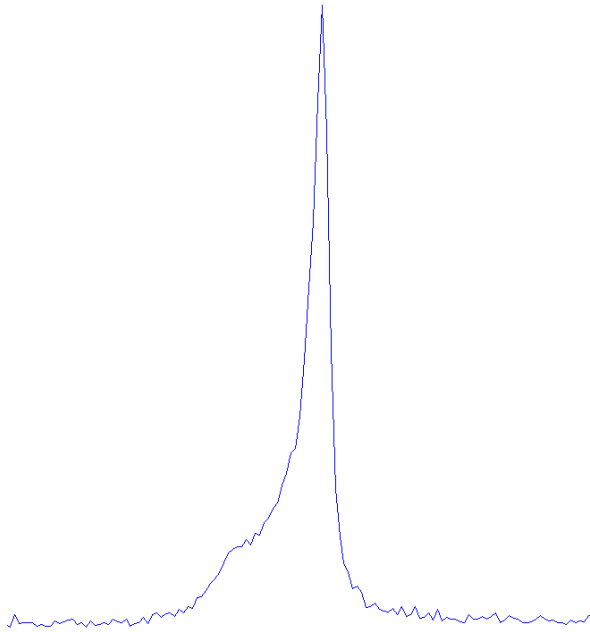
At this point you should have a defined peak. It will be broad but see below artifacts for specific artifacts and which shims to use to optimize. Generally, the CapNMR probe is able to shim up with Z1, Z2, X, Y, X2Y2, XY, X3, XZ2, Y3, YZ2. However, most users are able to shim up with simply 4-6 shims but this is just a characteristic of how the CapNMR probe reacts with your specific shim stack.

Varian Versus Bruker in Shimming Technique

We have consistently noticed a slightly different way in which the Varian and Bruker shim stacks shim the magnetic field. The significant difference generally occurs in the X, Y, and X2Y2 shims.

For Varian, the X and Y shims are very important for X & Y axis shimming and seem more 'pure'. They make drastic changes and can significantly improve the lock without additional shimming. However, optimizing X2Y2 should follow any significant change in the X & Y shims since lightly coupled.

For Bruker, the X and Y shims aren't as independent and are more closely coupled to the X2Y2 shim. It is important to start with X & Y but the dramatic change will occur with the X2Y2 adjustment. A change in X or Y may not even result in a change in lock but may allow significant improvements in X2Y2 when optimized.



This artifact is created by Z2.

If the artifact occurs on the high field side (left) as seen above then it generally requires several 1000s INCREASE. A 'shelf' on *only one side* of the peak's base characterizes Z2.

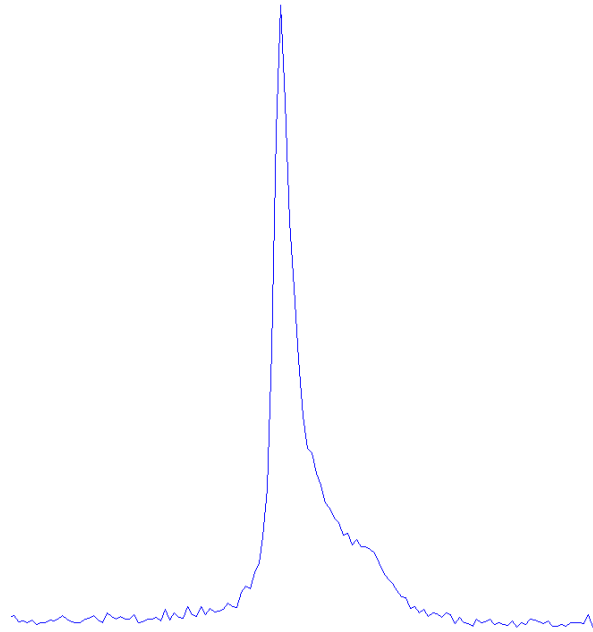
You may initially see a drop in lock/FID, optimizing the X2Y2 on the Z-axis shims will bring the lock/FID back up for a net improvement. (Adjust in steps of 200)

Continue by:

- optimizing Z2 in steps of 200
- optimizing Z1 in steps of 200
- optimizing X in steps of 10
- optimizing Y in steps of 10

Basic Strategy:

When the artifact looks like the spectra above, a drastic change in Z2 must be made and then optimizing the other shims will give you best effect. However, if you have a much less dramatic artifact, you'll only want to fine touch in steps of 200 or 500.



This artifact is created by Z2.

If the baseline artifact occurs on the low field side (right) as seen above then it generally requires several 1000s DECREASE. A 'shelf' on *only one side* of the peak's base characterizes Z2.

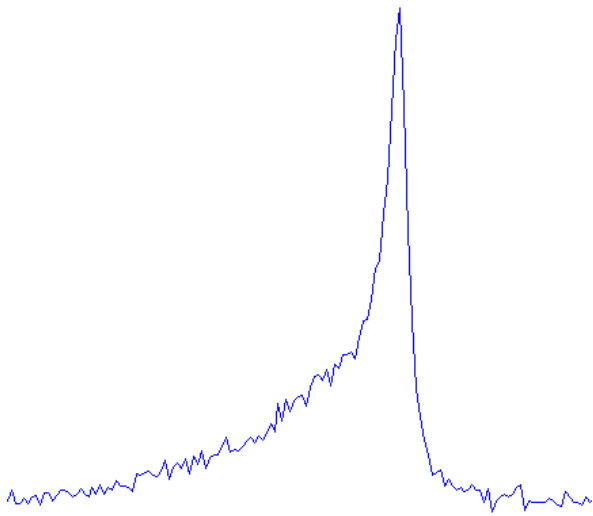
You may initially see a drop in lock/FID, optimizing the X2Y2 on the Z-axis shims will bring the lock/FID back up for a net improvement. (Adjust in steps of 200)

Continue by:

- optimizing Z2 in steps of 200
- optimizing Z1 in steps of 200
- optimizing X in steps of 10
- optimizing Y in steps of 10

Basic Strategy:

When the artifact looks like the spectra above, a drastic change in Z2 must be made and then optimizing the other shims will give you best effect. However, if you have a much less dramatic artifact, you'll only want to fine touch in steps of 200 or 500.



This artifact is created by X2Y2.

If the artifact occurs on the high field side (left) as seen above then it generally requires several 1000s INCREASE.

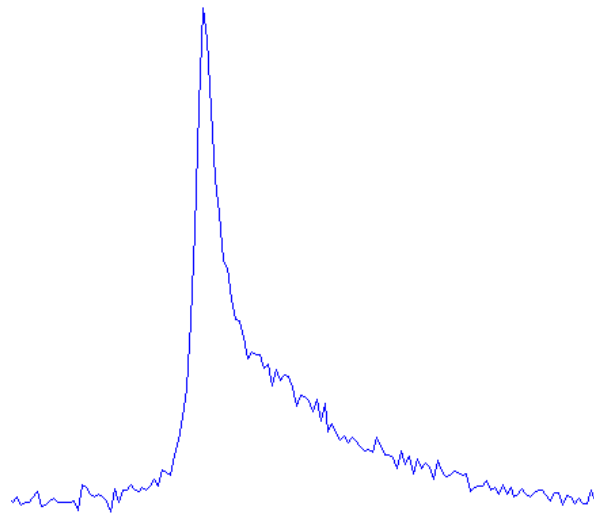
X2Y2 has a mathematical association with Z2. Whenever a large change is made to X2Y2, you'll find that Z2 always changes (either + or -) the same direction each time with a large X2Y2 change.

Continue by:

optimizing Z2	in steps of 200
optimizing Z1	in steps of 200
optimizing X	in steps of 10
optimizing Y	in steps of 10

Basic Strategy:

You'll want to have all Z2 artifacts cleared before you start working on X2Y2 artifacts. X2Y2 is almost always responsible for a sharp baseline. X2Y2 artifacts generally have a characteristic slope to them along one side on the baseline. (If there is a slope on both sides, move onto Z3 artifacts.)



This artifact is created by X2Y2.

If the artifact occurs on the low field side (right) as seen above then it generally requires several 1000s DECREASE.

X2Y2 has a mathematical association with Z2. Whenever a large change is made to X2Y2, you'll find that Z2 always changes (either + or -) the same direction each time with a large X2Y2 change.

Continue by:

optimizing Z2	in steps of 200
optimizing Z1	in steps of 200
optimizing X	in steps of 10
optimizing Y	in steps of 10

Basic Strategy:

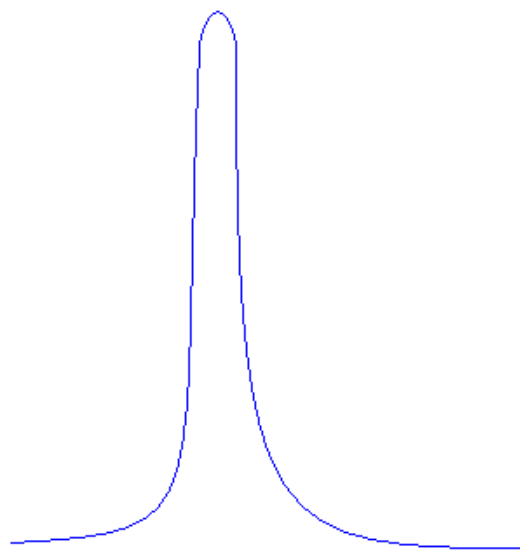
You'll want to have all Z2 artifacts cleared before you start working on X2Y2 artifacts. X2Y2 is almost always responsible for a sharp baseline. X2Y2 artifacts generally have a characteristic slope to them along one side on the baseline. (If there is a slope on both sides, move onto Z3 artifacts.)

An Important Note about X and Y Shims:

The CapNMR™ probe has a different coil than most 5 mm tube probes. The CapNMR has a horizontal solenoid coil, which is why it was important for the probe to be placed in the X or Y-axis of the shim stack. Either the X or the Y shim will always be more sensitive than the other shim.

For example, if you change the Y shim 30 points and you see a large drop/increase in your lock and then you change the X shim 30 points and you see only about 10% of the effect the Y shim caused, then you are on the Y axis. In this example, the Y-axis is what we call your “on axis shim” and will always be more sensitive than the other “off axis shim”.

The reason for this effect is that your “off axis shim” is moving along the length of the coil so a change may simply move to another part of the coil where there is just as much signal. However, your “on axis shim” is looking down the coil as you would down a telescope. The “on axis shim” can either be on the diameter of the coil, which would give maximum signal/lock level but if it's just off the diameter, that a dramatic change in signal so it reacts more to a smaller change.



This artifact is created by X and Y.

Broad peaks at half height with ‘rounded’ tops characterize the X and Y artifacts. Start with the more sensitive shim and follow-up with the ‘off axis shim’ until both have maximized lock. There is no common direction this artifact represents, so adjust the shim in one direction by 50 and see if the artifact improves or amplifies to determine the direction of shimming.

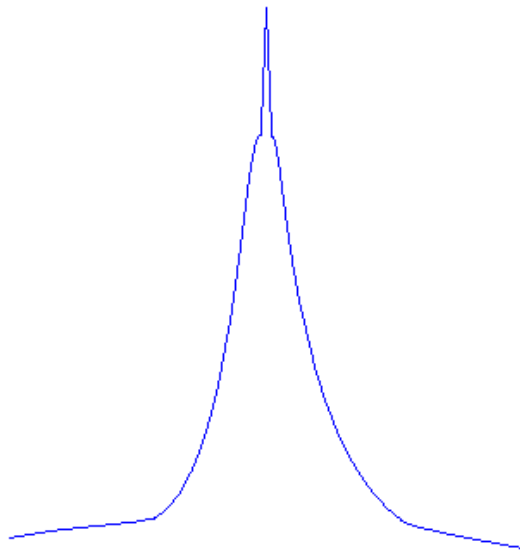
Continue by:

optimizing X2Y2	in steps of 200
optimizing Z2	in steps of 200
optimizing Z1	in steps of 200

Basic Strategy:

X and Y should both be optimized together but once they are near to their optimal values, they will basically become touchup shims for other shim adjustments.

When adjusting X and Y for the first time, only want to use Z1, Z2 and X2Y2 to optimize X and Y. However, once they are optimized and you start higher order shimming, you’ll optimize X and Y after each adjustment because higher order shims start to affect the axis geometry within the flowcell.



This artifact is created by X3 or Y3 artifact.

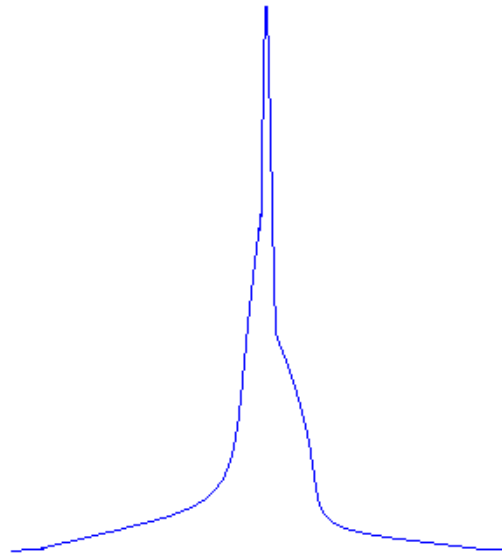
The shim is determined by the 'on axis shim'. If X is more sensitive, then this is a X3 artifact but if Y is more sensitive, then this is an Y3 artifact.

Continue by:

optimizing X	in steps of 10
optimizing Y	in steps of 10
optimizing X2Y2	in steps of 200
optimizing Z2	in steps of 200
optimizing Z1	in steps of 200

Basic Strategy:

X3 or Y3, depending on your on axis shim, generally has a spectral artifact that occurs along the sides on the spectral peak and generally occurs on both the high field side and the low field side although not even. X3 is highly coupled to XZ2, and Y3 is highly coupled to YZ2 so a change in one shim may not change anything but this would affect the other shims. Small changes sometimes don't make a dramatic affect so a large change may decrease the lock level but once the complimentary shims are optimized, there will be a net improvement.



This artifact is created by XZ2 or YZ2.

The shim is determined by the 'on axis shim'. If X is more sensitive, then this is a XZ2 artifact but if Y is more sensitive, then this is an YZ2 artifact.

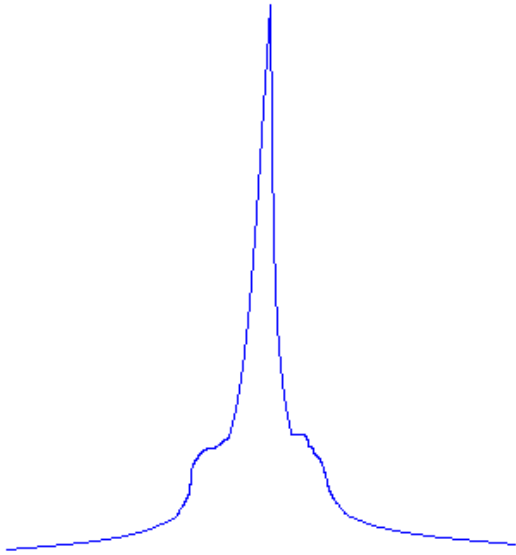
Continue by:

optimizing X	in steps of 10
optimizing Y	in steps of 10
optimizing X2Y2	in steps of 200
optimizing Z2	in steps of 200
optimizing Z1	in steps of 200

Basic Strategy:

X and Y should both be optimized together but once they are near to their optimal values, they will basically become touchup shims for other shim adjustments.

X3 is highly coupled to XZ2, and Y3 is highly coupled to YZ2 so a change in one shim may not change anything but this would affect the other shims. Small changes sometimes don't make a dramatic affect so a large change may decrease the lock level but once the complimentary shims are optimized, there will be a net improvement.



This artifact is created by Z3.

There are sometimes artifacts slightly higher than baseline on *both* sides of the peak that are due to Z3. Since this is a high order shim, it won't affect the flowcell as much so large steps in 1000's are required. There is no common direction this artifact represents, so adjust the shim in one direction by 500 and see if the artifacts improves or amplifies to determine the direction of shimming.

Continue by:

- optimizing Z2 in steps of 200
- optimizing Z1 in steps of 200
- optimizing X2Y2 in steps of 200
- optimizing X in steps of 10
- optimizing Y in steps of 10

Basic Strategy:

This shim is rather straightforward and will affect the peak between half height and baseline. These artifacts are much lower on the peak than the Z2 'shelf'.

No common descriptive artifacts except a slightly broad peak

This artifact is created by XY.

This is not a common shim but can sometimes make a difference although there aren't any common characteristics. Since this is a high order shim, it won't affect the flowcell as much so large steps in 1000's are required. There is no common direction this artifact represents, so adjust the shim in one direction by 500 and see if the artifacts improves or amplifies to determine the direction of shimming.

Continue by:

- optimizing X2Y2 in steps of 200
- optimizing Z2 in steps of 200
- optimizing X in steps of 10
- optimizing Y in steps of 10
- optimizing Z1 in steps of 200

Basic Strategy:

XY generally required a drastic change and generally never improves lineshape but requires an adjustment in X2Y2 to improve lock/FID.

If you still have difficulty shimming the CapNMR probe, please give Protasis/MRM a call at (217) 351-4359 and we'd be glad to help in any way we can. It would help us assist you if you could print out a copy of your current lineshape and artifacts and fax to (217) 352-6655.