Shimming

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JEOL UK Demo Lab

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How to setup 1D gradient shimming

Gradient shim calibrations (shim mapping)

- It is important that all the following gradient shim calibrations are done during installation

System Type	Nucleus	Comments
Homospoil	2H	By far the most used method; for deuterated solvents that have only one deuterium resonance (e.g. DMSO-D6, chloroform-D, acetone-D6)
Homospoil_selective	2H	Very important to calibrated, needed for solvents that have more than one deuterium resonance, e.g. Methanol-D4
Homospoil_selective	1H	Needed for non-deuterated solvents (No-D NMR)
Fast_homospoil_CC	2H	Needed for variable temperature (VT) measurements. Depending on solvent, can be needed for even modest temperatures. **The default supplied pulse sequence does not work well**
Fast_homospoil_selective_CC	2H	As above; for solvents with more than one deuterium resonance **The default supplied pulse sequence does not work well**

Gradient shim calibrations (shim mapping)

Samples

× 1H lineshape sample is not ideal (long T_1 relaxation, needs long relaxation delay, time consuming)

× 1H sensitivity sample is not ideal (low SNR for 2H, more scans needed, time consuming)

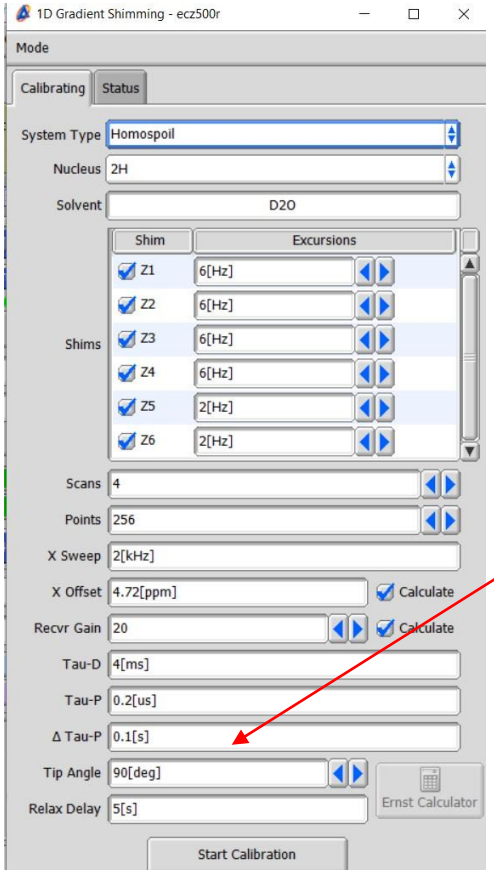
✓ Doped 1% H2O in 99% D2O is an excellent choice

✓ ASTM (40% p-dioxane) is also a good choice, and can be used for 1H calibration

System Type	Nucleus	Sample	Scans* (1H sensitivity)	Relaxation delay (1H lineshape)
Homospoil	2H	Doped H2O; ASTM; 1H lineshape; 1H sensitivity	4 (8)	2 (5)
Homospoil_selective	2H	As above	4 (8)	2 (5)
Homospoil_selective	1H	ASTM	4 (8)	2 (5)
Fast_homospoil_CC	2H	Doped H2O; ASTM; 1H lineshape; 1H sensitivity	8 (16)	2 (5)
Fast_homospoil_selective_CC	2H	Doped H2O; ASTM; 1H lineshape; 1H sensitivity	8 (16)	2 (5)

*For ROYAL/HFX probes. Older or direct-style probes may need more scans

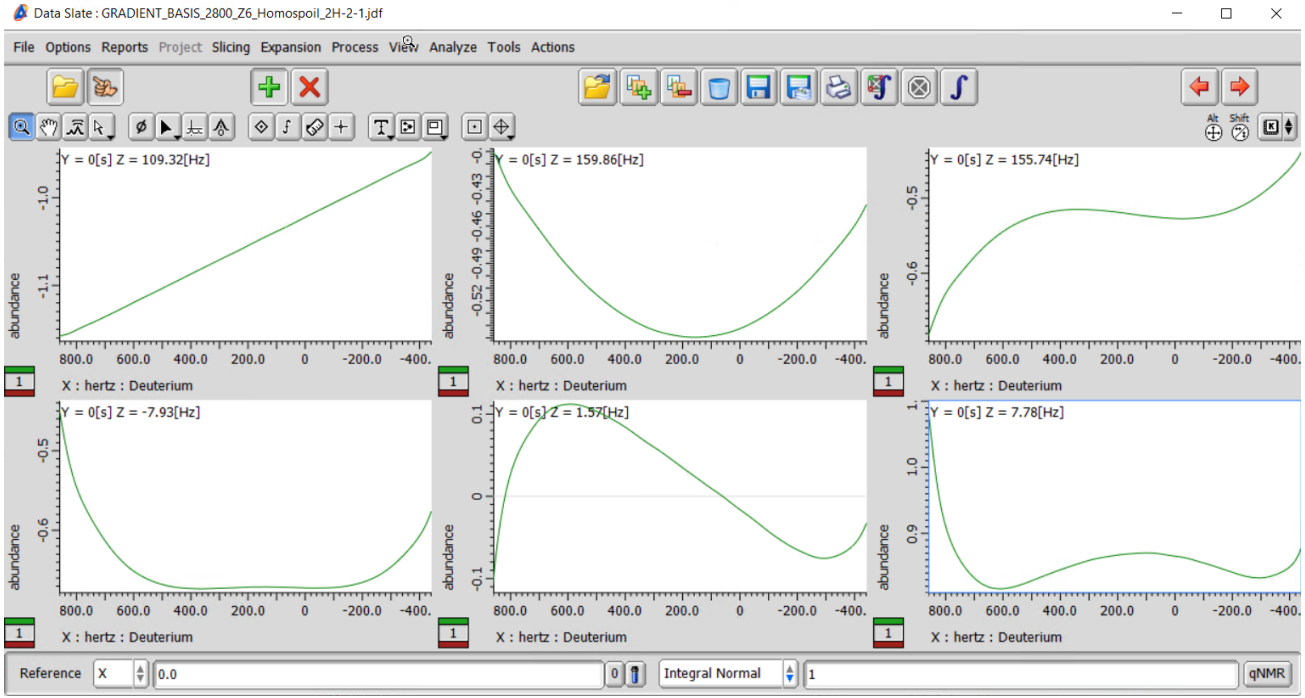
Gradient shim calibrations (shim mapping)



- Use a 90-degree tip angle for all calibrations
- Don’t bother with the Ernst calculator, no one uses it
- Leave the defaults as they are, including excursions (too large excursions will cause aliasing in the maps)
- For the 1H homospoil selective calibration, it can be useful to reduce Δ Tau-P from the default 20[ms] to e.g. 5[ms] to reduce issues with radiation damping

Gradient shim calibrations (shim mapping)

- After calibration, check the maps in a data slate
- “Trim” the edges so that Z1 (top left) is straight with no wiggles displayed either side
- The shim maps should show good symmetry
- A small asymmetry is acceptable but a large one could indicate a problem, e.g. probe vertical misalignment, issue with mapping



Setting up shimming parameters

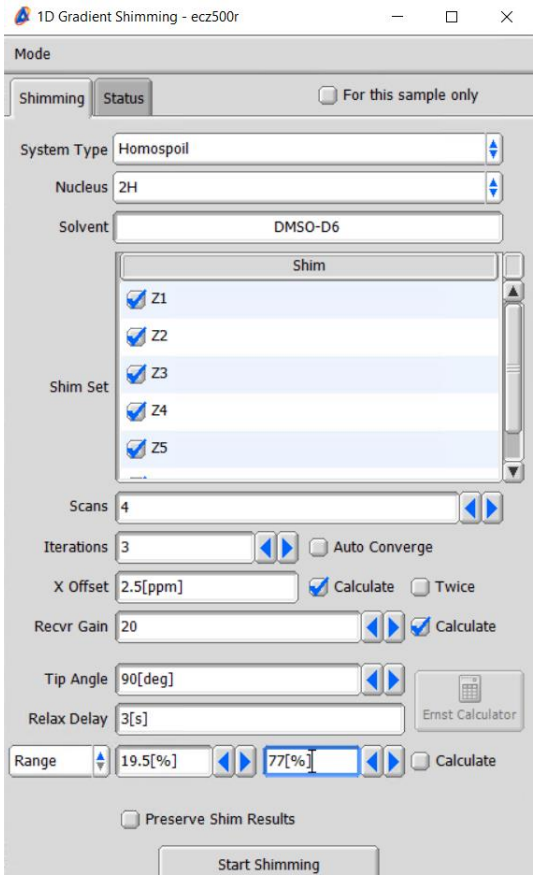
Setting parameters for gradient shimming

- Calibrating the shims (shim mapping) is only half the job

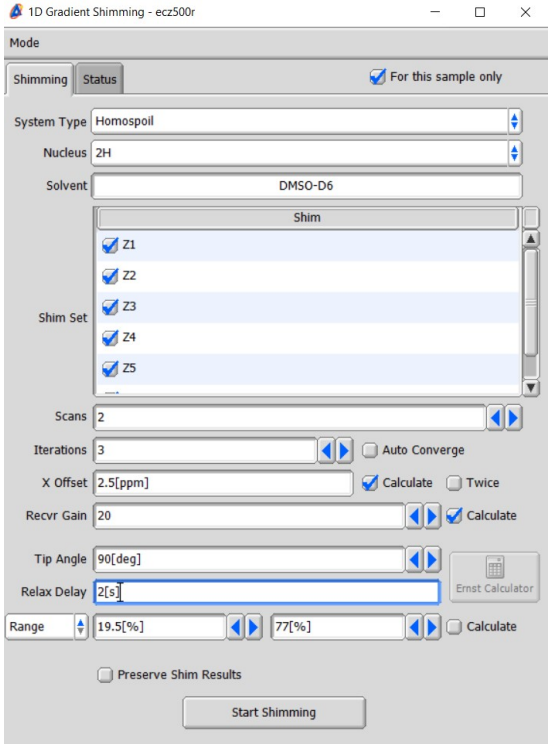
- Parameters for shimming different solvents need to be set for the different “System Types” (shim methods)

What does unchecking “For this sample only” do?

- Requires administrator (console) privileges
- Sets all default parameters, **including the “System Type” (shimming method)** , **<u>for the current solvent</u>**
- They apply ONLY to the current solvent/”System Type” combination
- **Checks and settings need to be made to ensure parameters are set for every solvent/method combination**



Sensible default parameters for different solvents

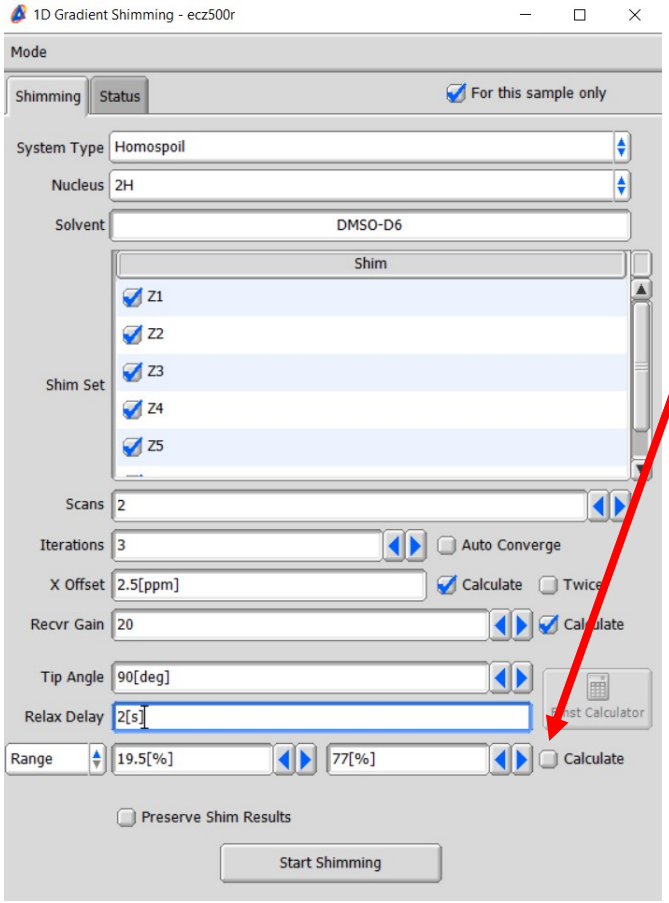


Solvent	Default shim method (System Type)	Scans* (CC methods)	Relaxation delay
Acetone	Homospoil	2 (4)	5
CDCl3	Homospoil	4 (8)	2
MeOD	Homospoil selective	2 (4)	2
DMSO-d6	Homospoil	2 (4)	2
D2O	Homospoil	2 (4)	2
Benzene-d6	Homospoil	2 (4)	2

*For ROYAL/HFX probes. Older or direct-style probes may need more scans

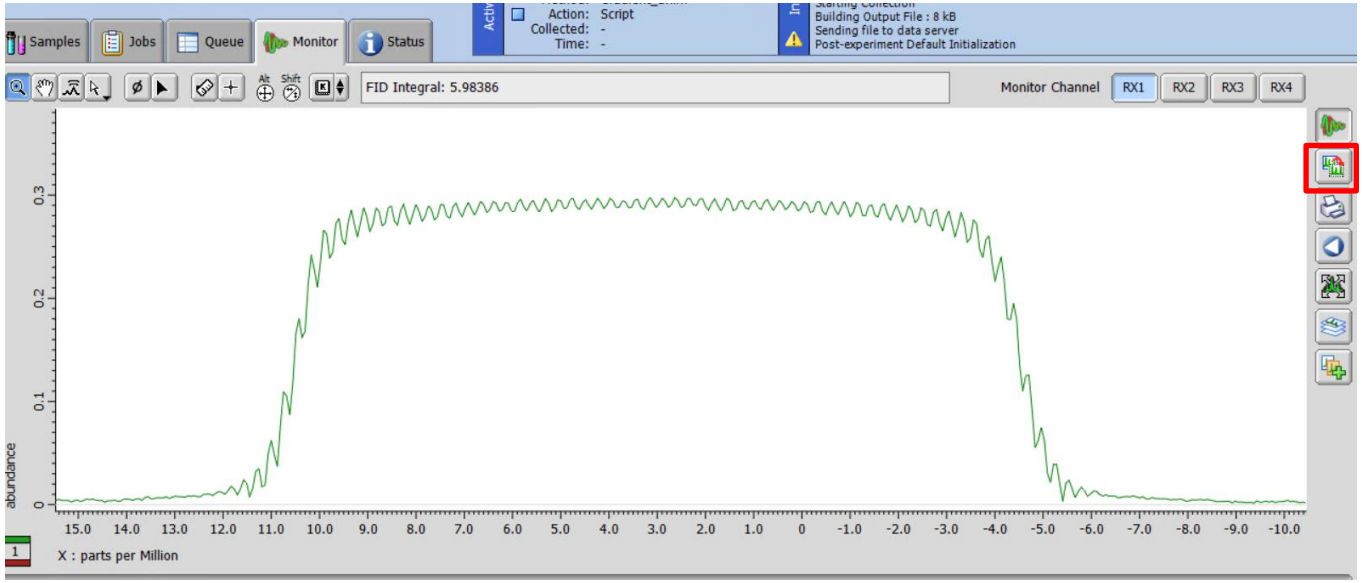
- Uncheck “Auto Converge”
- Set 3 iterations
- 90-degree tip angle for all methods
- Uncheck “Calculate” range and set manually (next steps)

Setting Range



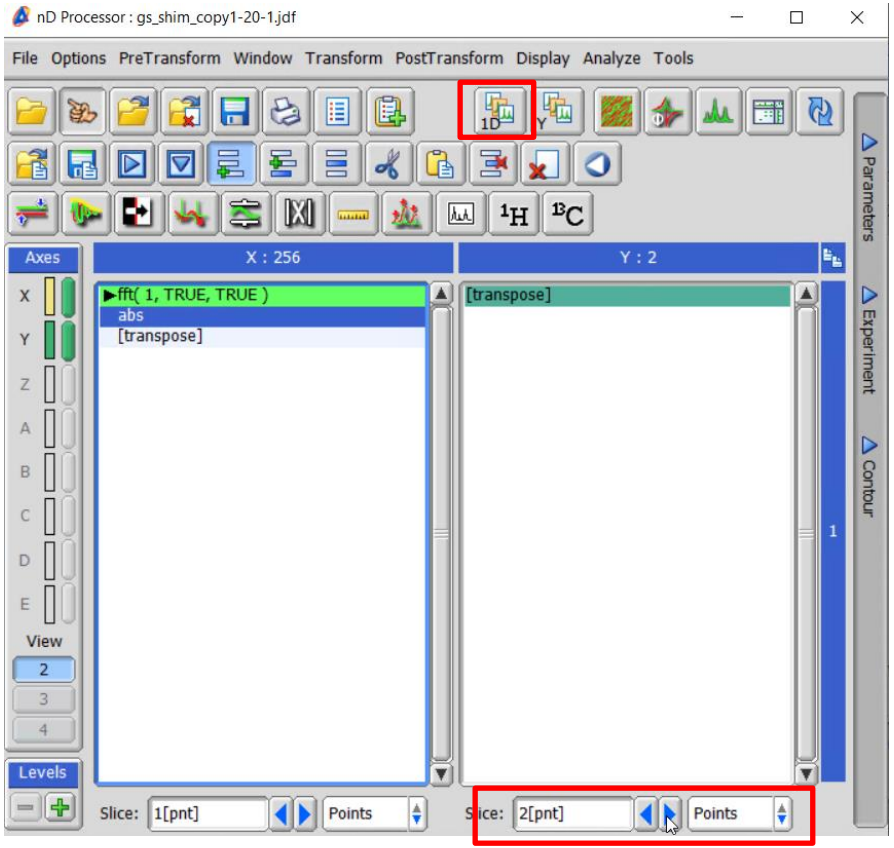
- "Calculate" option sets range too wide
- Better to determine range manually and set for all samples

Setting Range



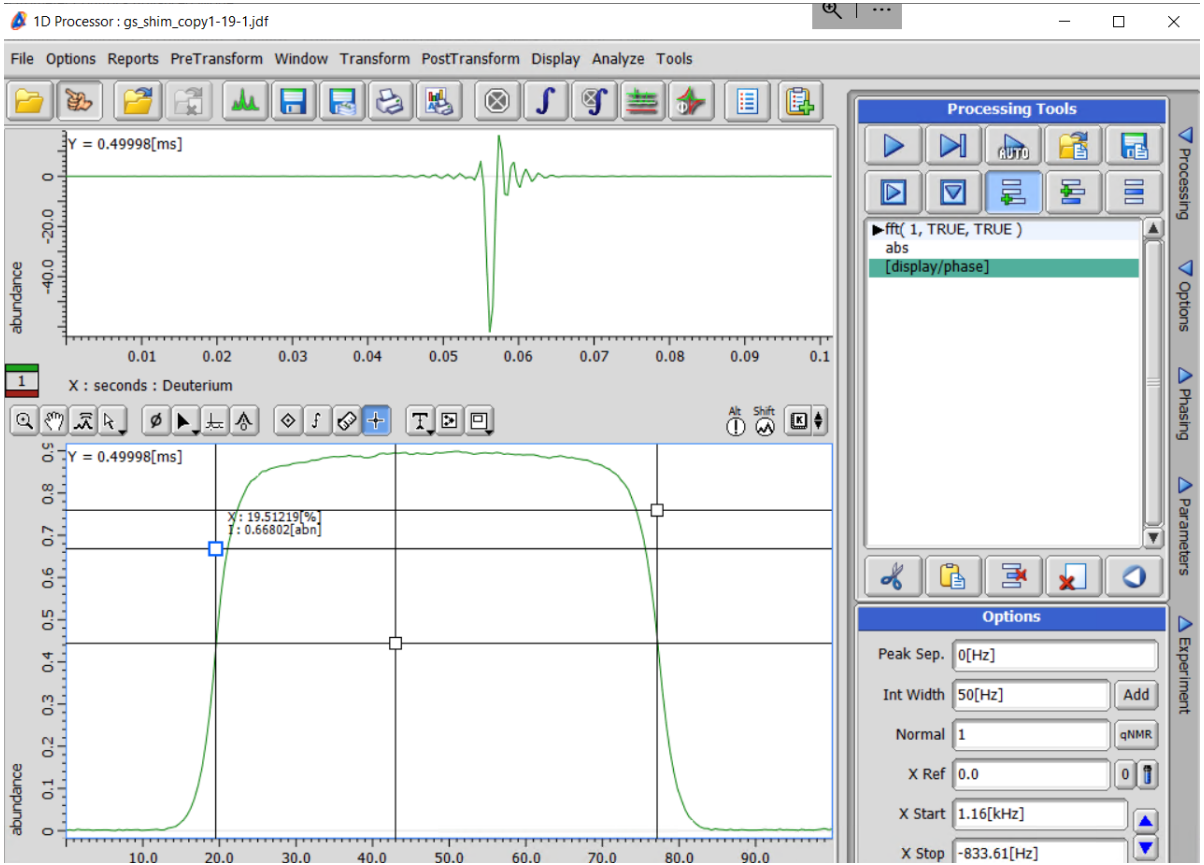
- A robust way to set range is to use ~50% amplitude points on second profile from gradient shimming
- Start gradient shimming with “calculate” option checked
- Go to “Monitor” window

Setting Range



- In nD Processor window, add FFT and Abs steps to X dimension
- Select 2[pnt] slice for Y dimension (i.e. second profile)
- Click 1D button

Setting Range



- Place cursor at top of profile and measure amplitude
- Place cursor at half that amplitude (i.e. 50% point)
- Place left and right cursors to intersect profile at 50% point
- Set X-axis units to %
- Read low and high range values
- IMPORTANT: range needs to be set for every method (“System Type”). E.g. setting range for “Homospoil” does not get automatically applied for “Homospoil selective”

Testing the gradient shimming

- After everything has been set up, it is essential to check that the shimming is working well

- A good test:

    - Remove and recreate sample definitions for standard test samples in the “Samples” tab. This will ensure that the default gradient shimming parameters will be used

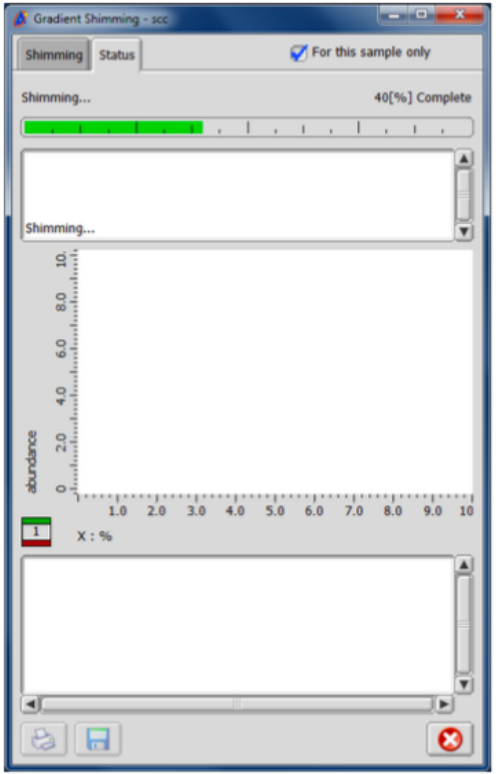
    - Run 1H lineshape, 1H sensitivity and 13C sensitivity GLP tests under automation – do the samples shim well and do the tests pass?

Troubleshooting

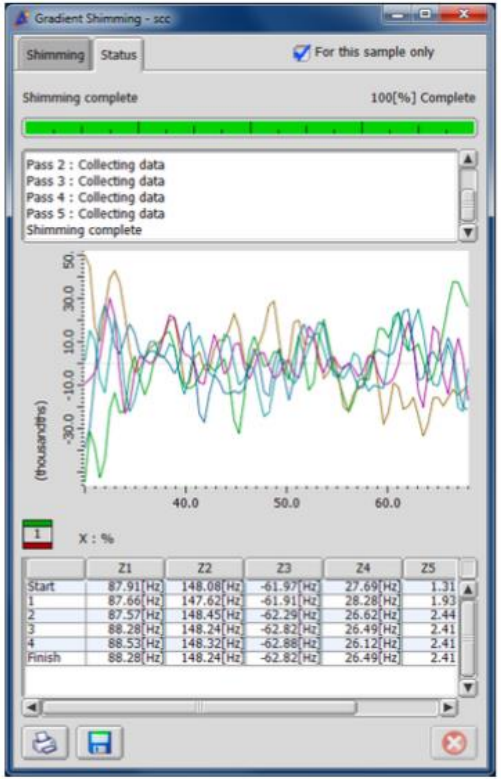
Occasionally, gradient shimming can fail to produce well adjusted line-shape.

Possible causes can include,

- Insufficient sample depth. Aim for a minimum 40mm sample depth. If this isn't possible for a particular sample, use the depth gauge to centre the sample against the centre of the RF coil.
- Dirty or scratched sample tube. Contaminated sample material.
- Temperature gradient. Insufficient wait time between VT on and starting gradient shimming. Remember, the temperature shown is that of the thermocouple NOT the sample. It takes time to catch up & settle(gradient shimming gives better results waiting 5 minutes than if just waiting 3 minutes, do not expect it to shim well without any temp_delay).
- Erratic spinning. Is the spinning speed stable? Try another rotor.
- Have the shim values been excessively adjusted. Try reloading the system shim file or a known good user shim file for the probe in use
- It can also help to check the gradient shimming is configured correctly and also the FG shim residual map.

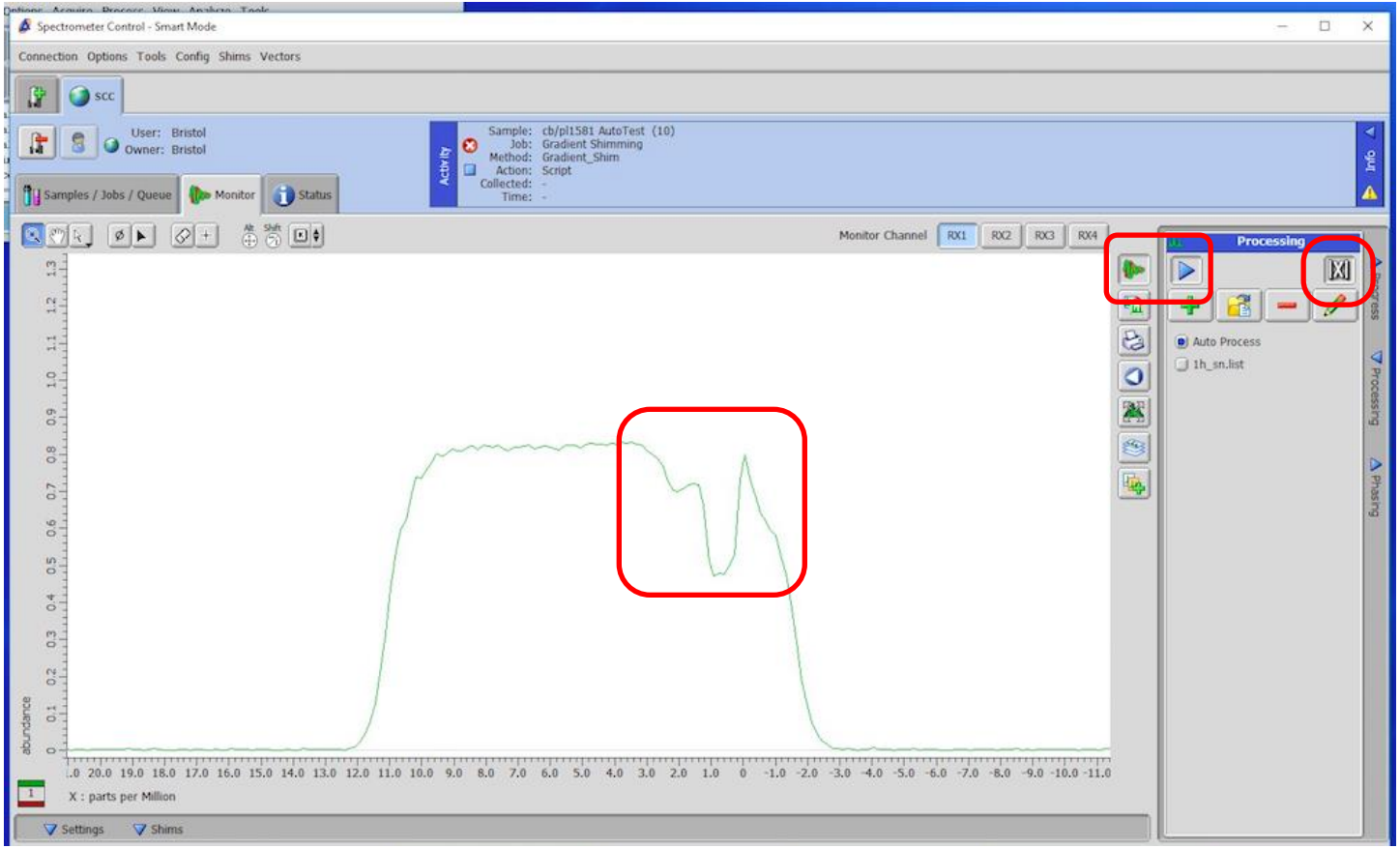


Click Start Shimming, the status tab is shown.

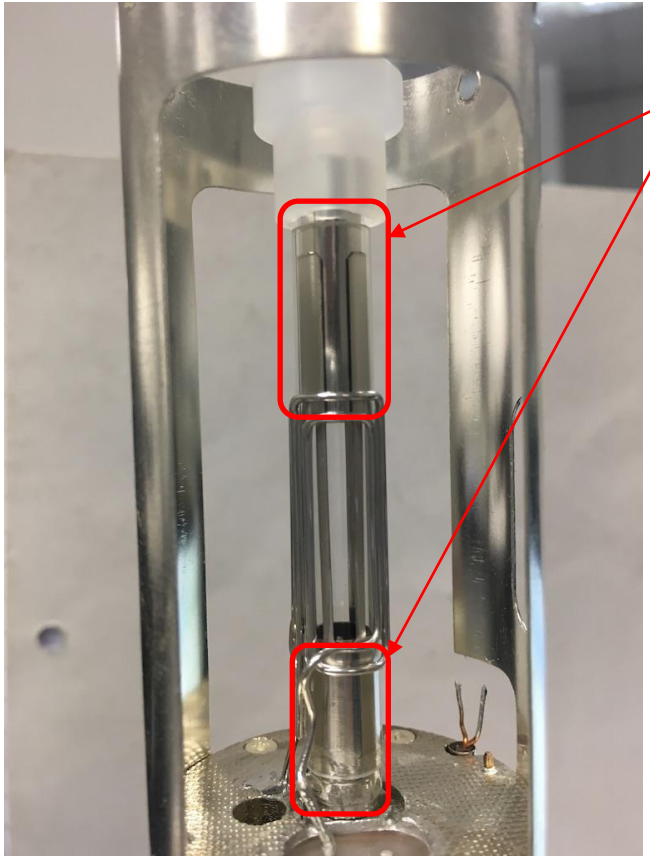


The residual plot should flatten out after each iteration.
Aim for less than +/- 0.1 abundance.

- The residual can bee seen in real time, from the monitor tab.
- Turn on FID, FT and ABS.
- Modern probes should have a square like profile, no notches as below.



The previous image indicates that either the sample tube is scratched/dirty, or that the detection coil area is dirty and needs cleaning. BUT…..

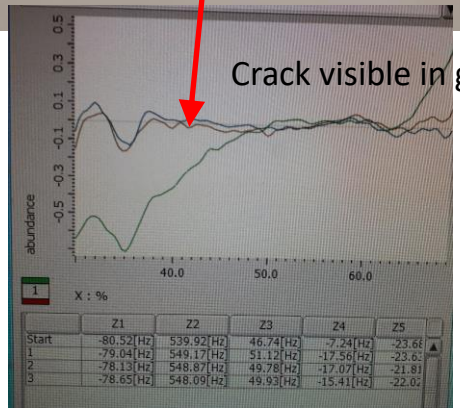


Modern sample coils have RF shielding above and below the detection coils.

They improve achievable line-shape by reducing end effects.

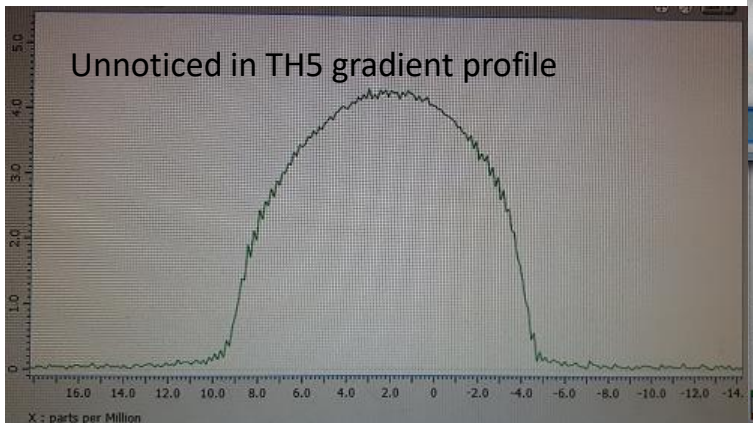
However, they make cleaning the detection coil area tricky.

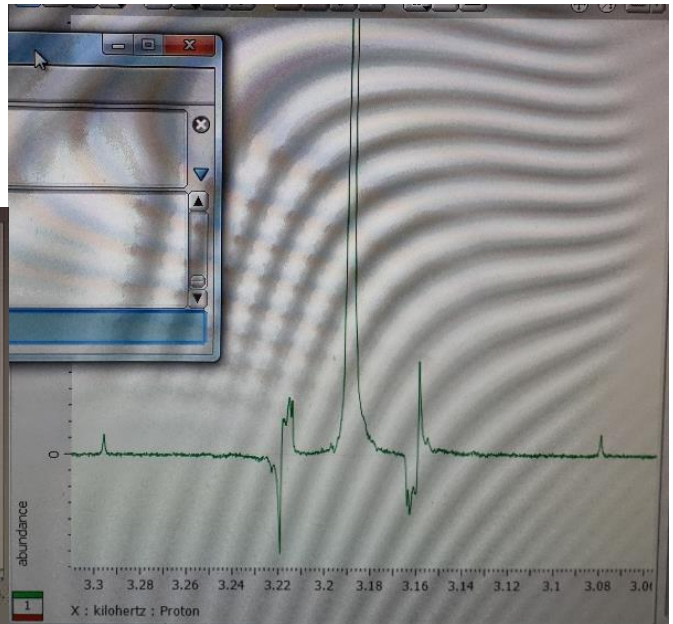
Vigorous cleaning with an aggressive solvent (e.g. Acetone) can lift the foil and prevent samples from spinning, as well as affecting the FG shimming performance.



Crack visible in gradient shimming fitting

Non-spin lineshape: cracked glass leading to bad coil fixing leading to vibrations





And finally…

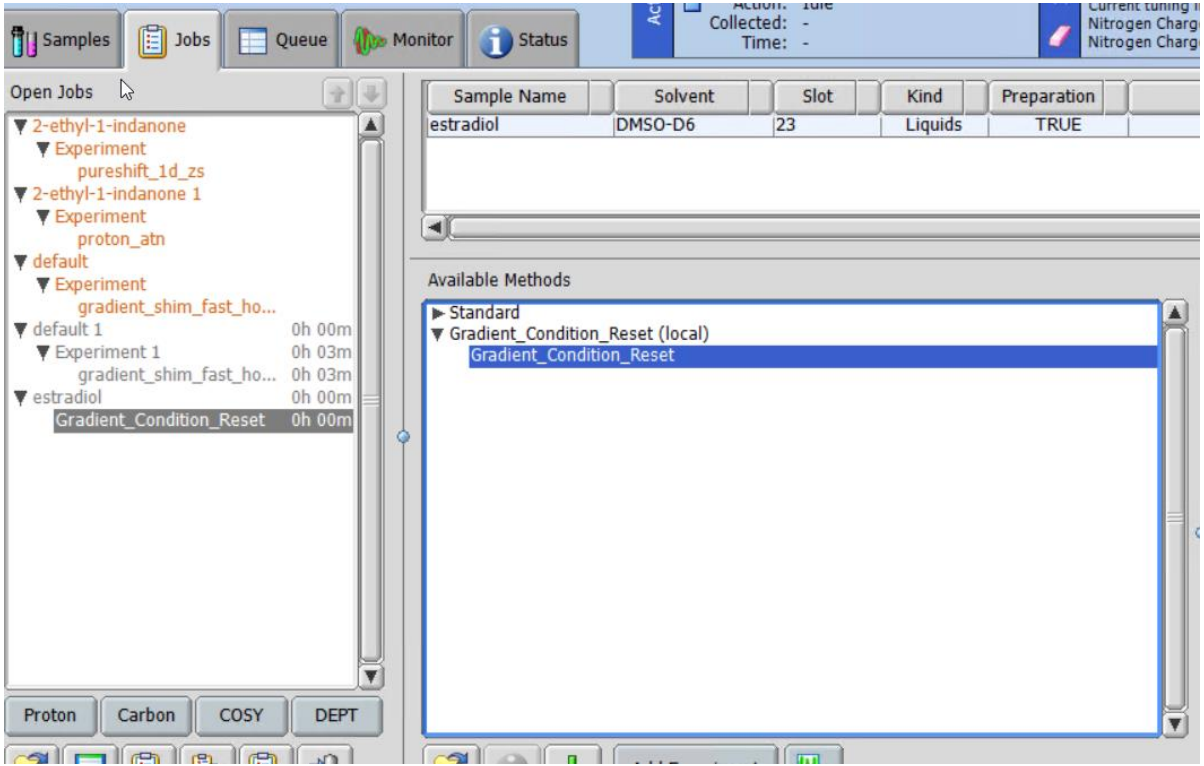
When you update the system shims with an HFX probe, remember to do so for both single and dual mode as each mode uses a different system shimset in Delta 5. In Delta 6 this is not needed.

- At the end of the installation remember to do a backup of everything, including data. If the database becomes corrupted (which can happen with power cuts), it needs to be restored otherwise the gradient maps will be lost.

How to restore normal configuration for 1D gradient shimming

Resetting to defaults

- Ashok has written an automation script Gradient_Condition_Reset.jaf
- Tested in UK applab, works
- Load script, add method to a job, click “Submit”
- Use as a last resort, but can be useful



Gradient shimming files

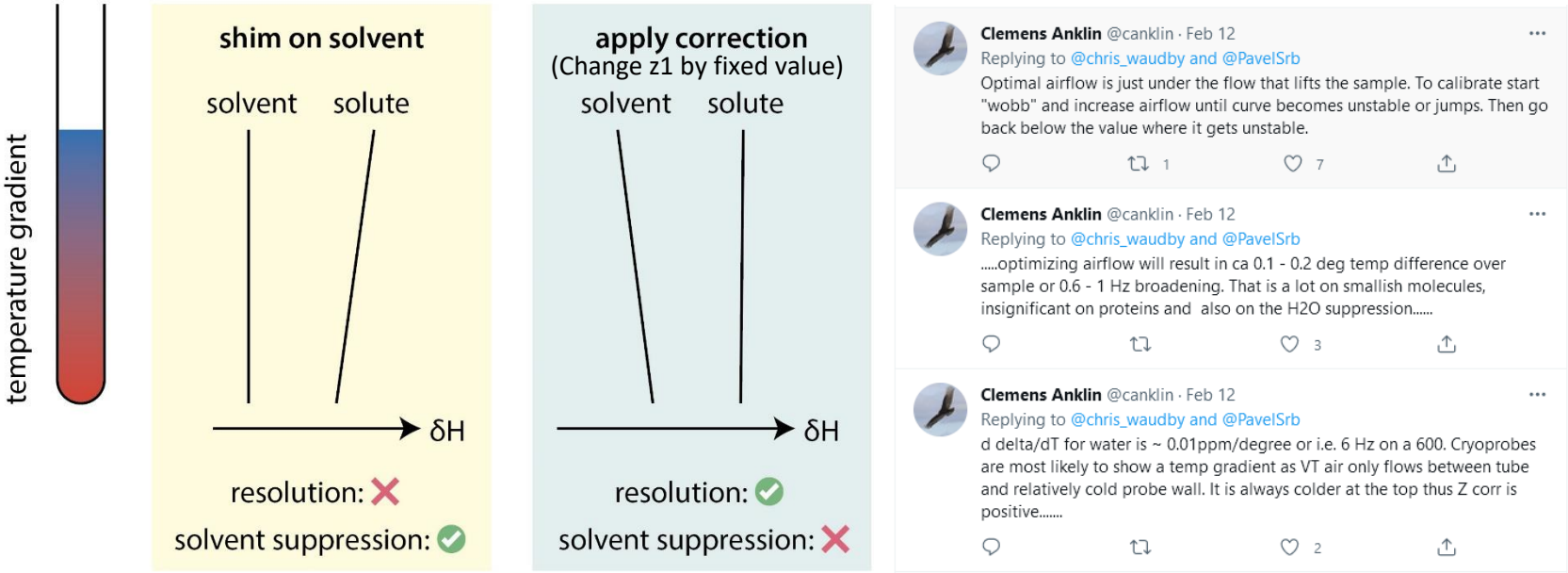
For a completely new and never used system, if you uncheck “For this sample only” and setup parameters, changes will be reflected for all samples except for those with different predefined parameters. Used systems may have other conditions and different solvents can have different parameters, and different specific samples can have different parameters.
A dive into the configuration of control in the console can clarify what is happening. Locate *C:\Program Files\Common Files\JEOL\Control 5.3*

This folder contains several files related to the shimming:

- *Solvent_reference.jnv*: It tells the system which type of shimming it should do for each solvent.
- *gradient_shim.jnv*: It tells which nucleus and shims to use in shimming
- *gradient_shim-homospoil-2h.jnv*: Contains the shimming settings for each solvent with this type of gradient shimming
- System shims are in probe file
- Shimmaps are in the database!

Temperature gradients

Temperature gradients are common

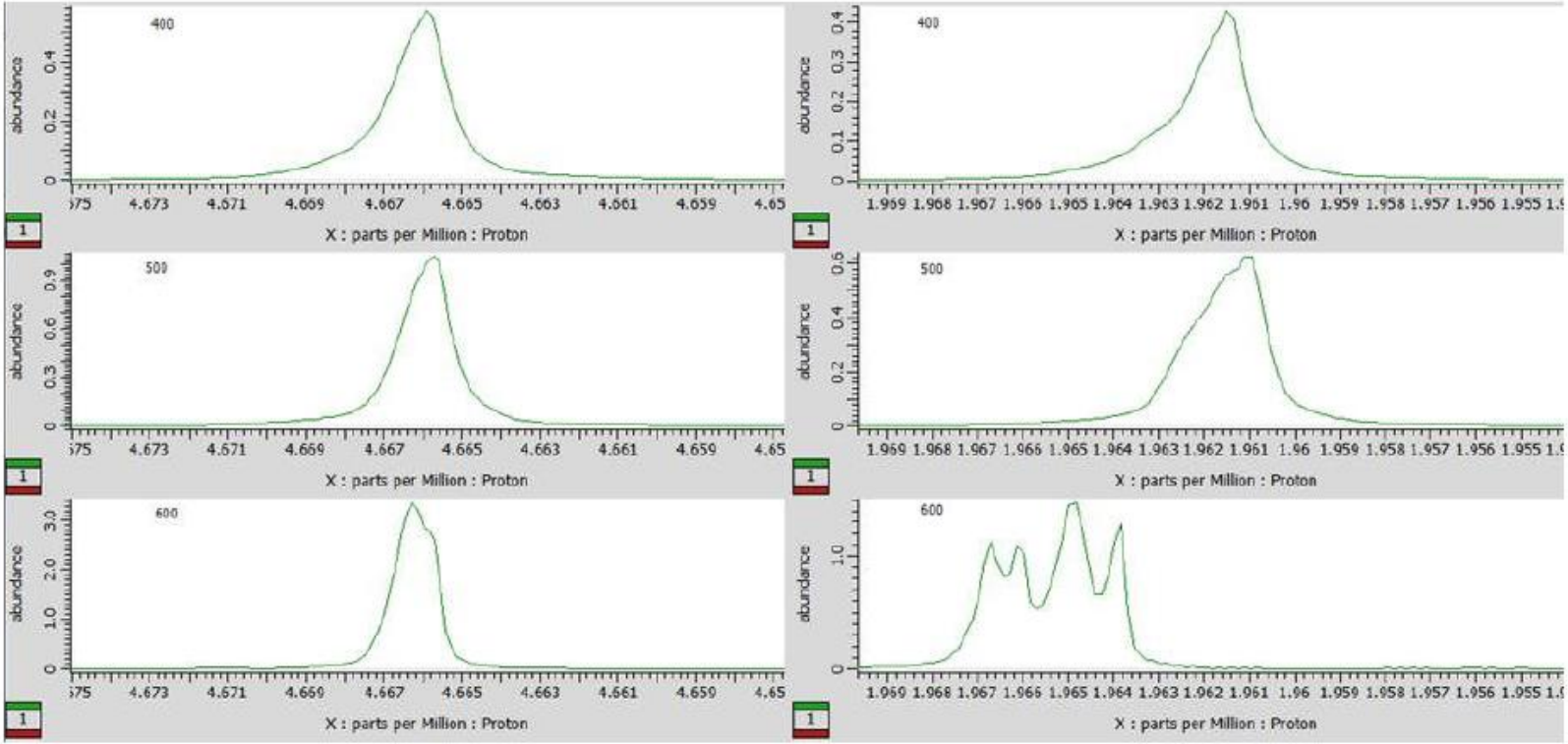


https://twitter.com/chris_waudby/status/1492474822932275201

Solvent vs solute: 400 Demo HFX vs 500 Demo HFX vs 600 RO

	LW_D2O(Hz)	LW_ACN(Hz)
400	0.64	0.54
500	0.73	0.95
600	0.75	1.95

Varian temperature gradient standard
H2O vs CH3CN signals

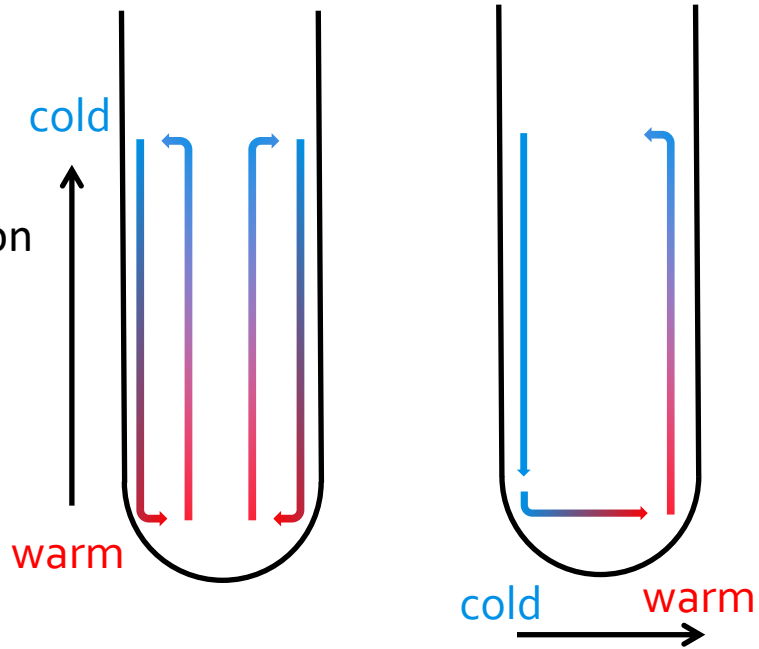


Temperature gradients lead to convection

- Once a critical Raleigh number is reached (1700 for a Benard configuration, i.e., two parallel horizontal boundaries separated by a distance *d*), natural convection appears.

$$\mathrm{Ra} = \frac{\rho\beta\Delta T l^3 g}{\eta\alpha}$$

g: gravity,
β: coefficient of thermal expansion
ΔT: temperature difference
l: length
η: viscosity
α:thermal diffusivity

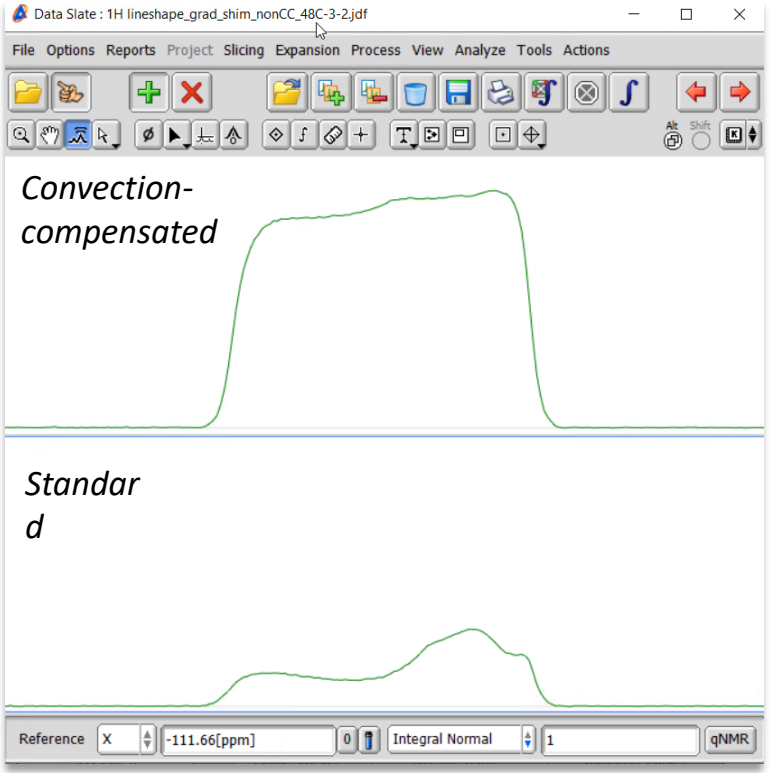


Convection-compensated gradient shimming

- At temperatures significantly away from room temperature, sample convection will occur

- This will cause the signal profiles collected by gradient shimming to be heavily attenuated and/or distorted

- Solution: use a pulse sequence that compensates for convection
  - Double spin echo refocuses additional phase shifts that accumulate due to convection



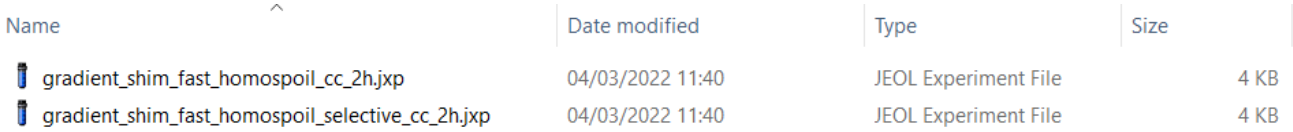
Acetone-d6 sample, 48° C

Convection-compensated gradient shimming

- Calibrate “Fast_homospoil_CC” and “Fast_homospoil_selective_CC” during installation

- Standard sequences do not work properly. Ashok has supplied “Non-fast” versions that have been verified to work.

- Recommended use:
  - Working sequences have been renamed as “Fast” versions



Name	Date modified	Type	Size
gradient_shim_fast_homospoil_cc_2h.jxp	04/03/2022 11:40	JEOL Experiment File	4 KB
gradient_shim_fast_homospoil_selective_cc_2h.jxp	04/03/2022 11:40	JEOL Experiment File	4 KB

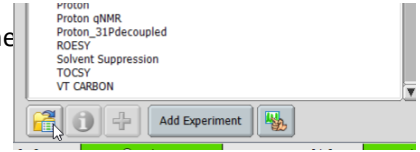
  - Upload to “Experiments folder on spectrometer and then calibrate these methods as described

3D gradient shimming

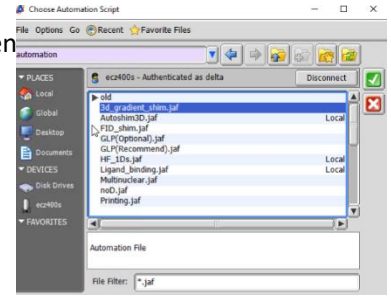
3D gradient shimming

Transverse shims need to be shimmed from time to time with a period ranging from every 3 days to every 3 months depending on magnet stability and nature of work.

1. Load the standard 1H lineshape sample if you have it
2. Use the gradient shimming tool to Z-gradient shim first
3. Create a job for this sample
4. Load the 3D_gradient_shim.jaf script by clicking on the button to the bottom-left of the “Available Methods” window as shown below



5. Navigate to the spectrometer using the window that opens and then select the 3D gradient shimming script as shown below



6. The 3D gradient shimming method should now be available

7. Load the method and then set shimXY_array_pnts to 8x8 (4x4 is acceptable on a 400 JEOL magnet)
8. Click the “Submit Job” button. The 3D shimming will take around half an hour or so to complete.
9. After this check the lineshape. Save the system shims as console.

When to run it

- At installation
- Every week (if it works)