TopShim Advanced Tips and Tricks

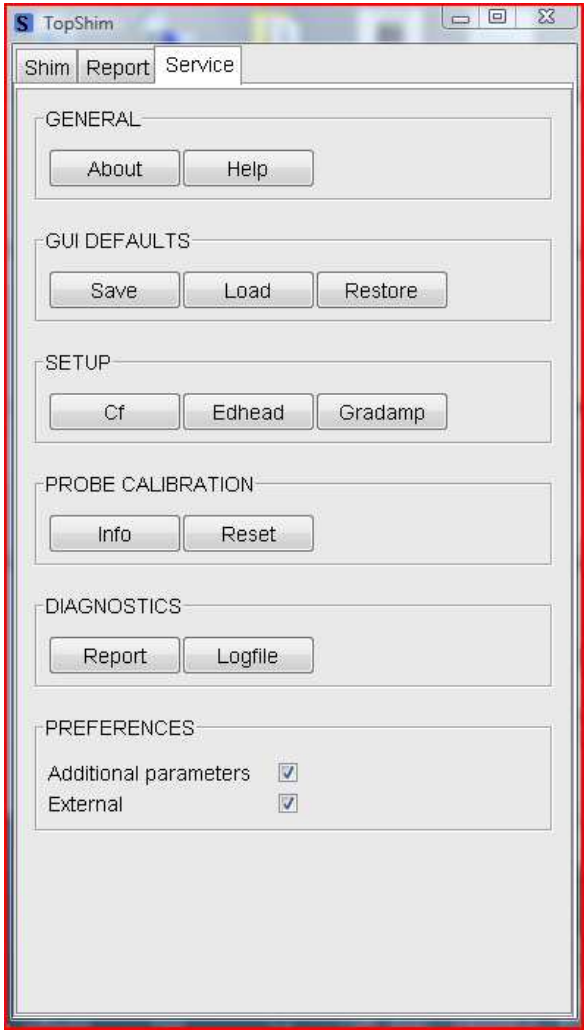
Presented by Mike Brown

03192012

Advanced Topshim

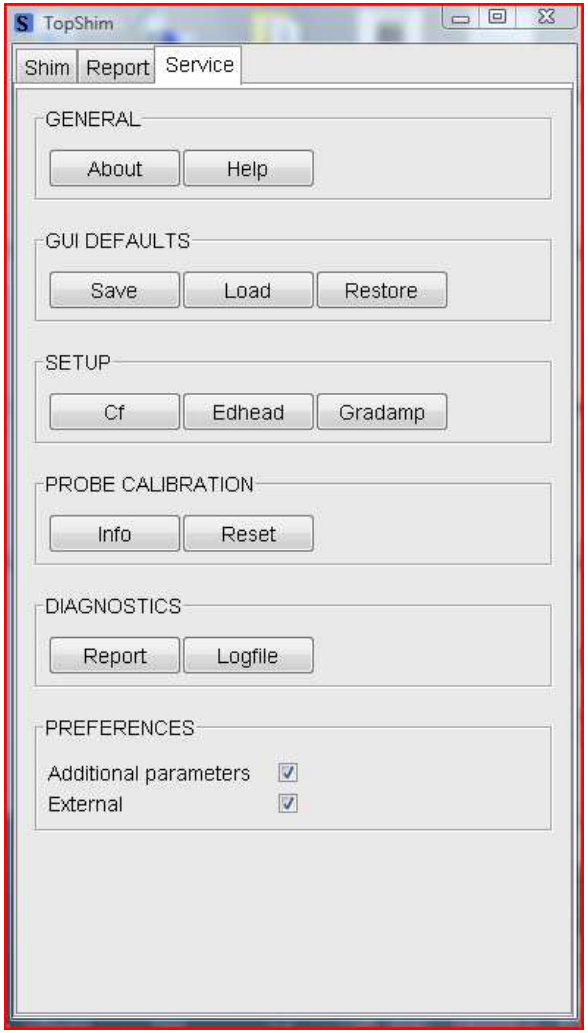
- Service tab in GUI
- Useful command line arguments
- Calibration Procedure
- Adding a new solvent
- Use in automation
- Tips

Service Features in the GUI



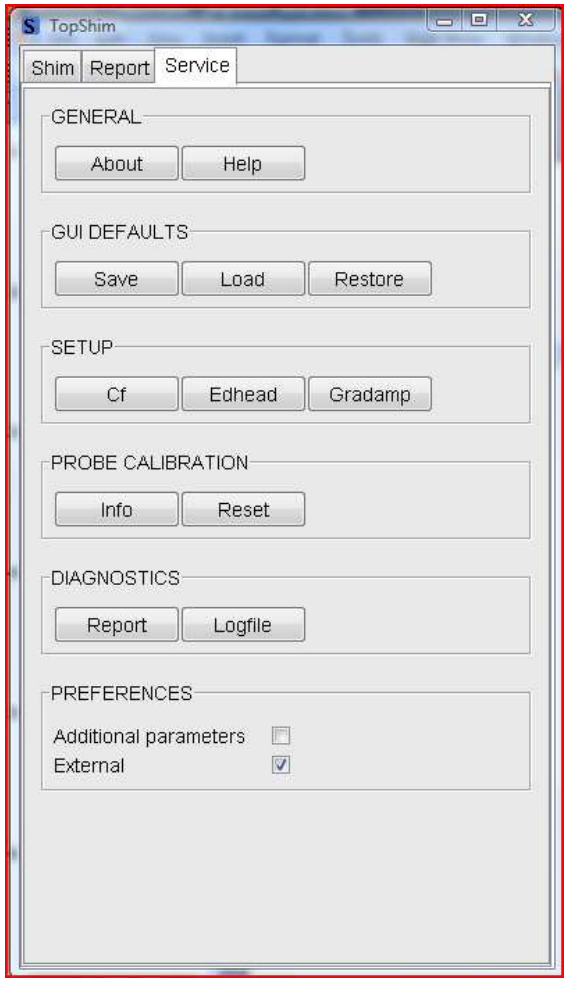
- General
  - About - Topshim version information
  - Help – Opens Topshim manual
- GUI Defaults
  - Save, Load or Restore GUI preferences
- Setup
  - Cf - Configure instrument
  - Edhead - Choose and setup probe
  - Gradamp – Choose gradient amplifier if more than one available

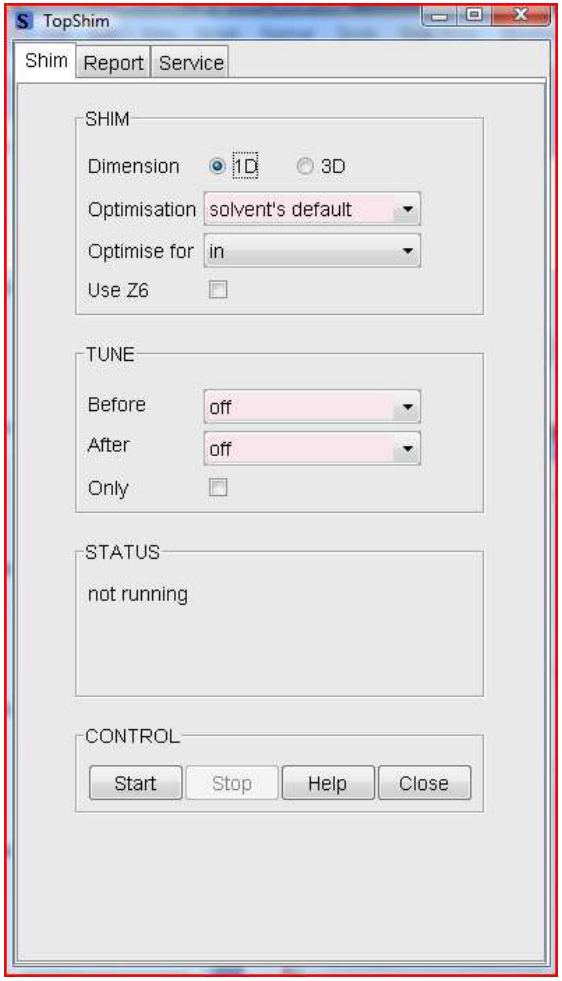
Service Features in GUI



- Probe Calibration
  - Info - Shows current probe calibration information
  - Reset - Deletes current probe head information
- Diagnostics
  - Report - Opens report that shows information about last topshim execution
  - Logfile - Opens box showing location of topshim logfile
- Preferences
  - Additional parameters
  - External - Opens topshim gui in external window

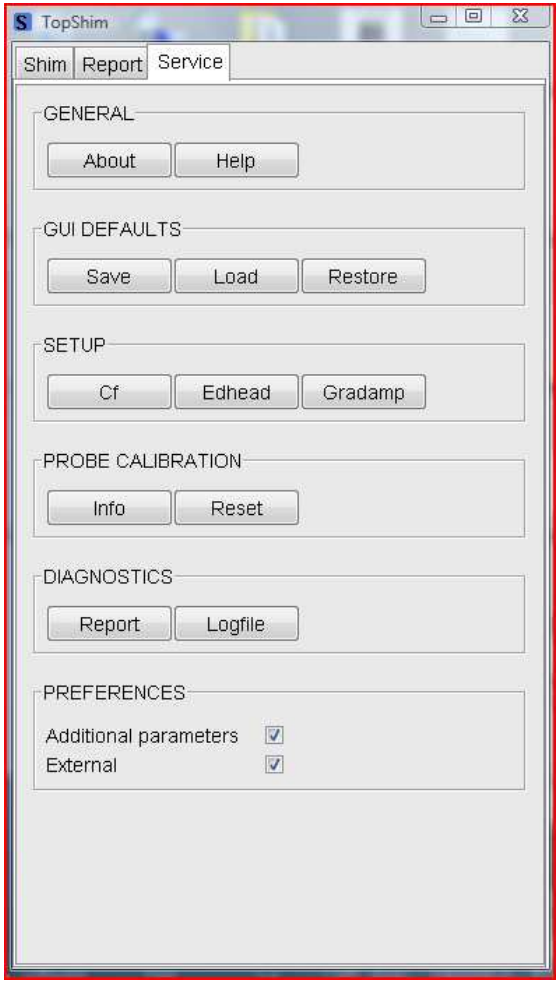
Service Features in GUI

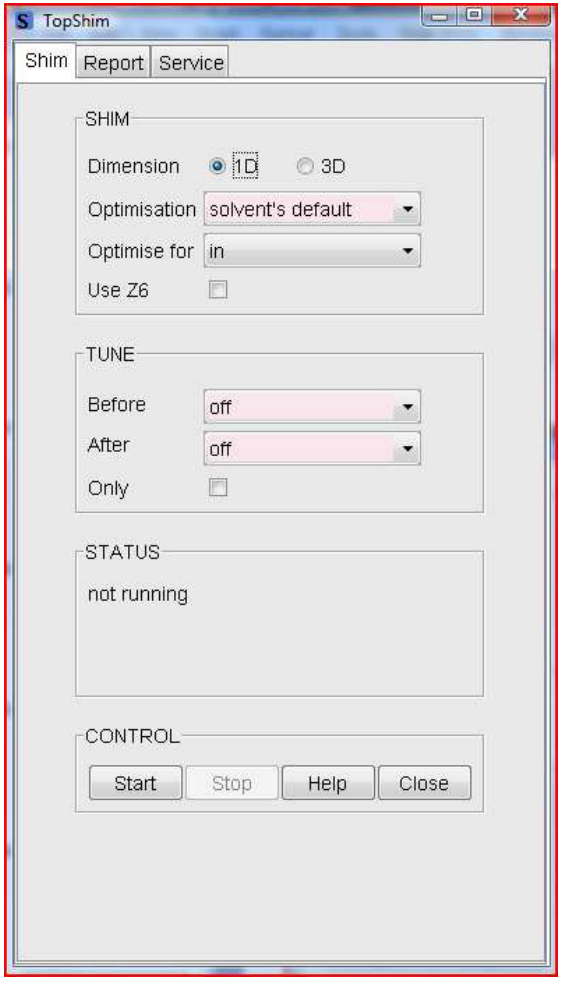


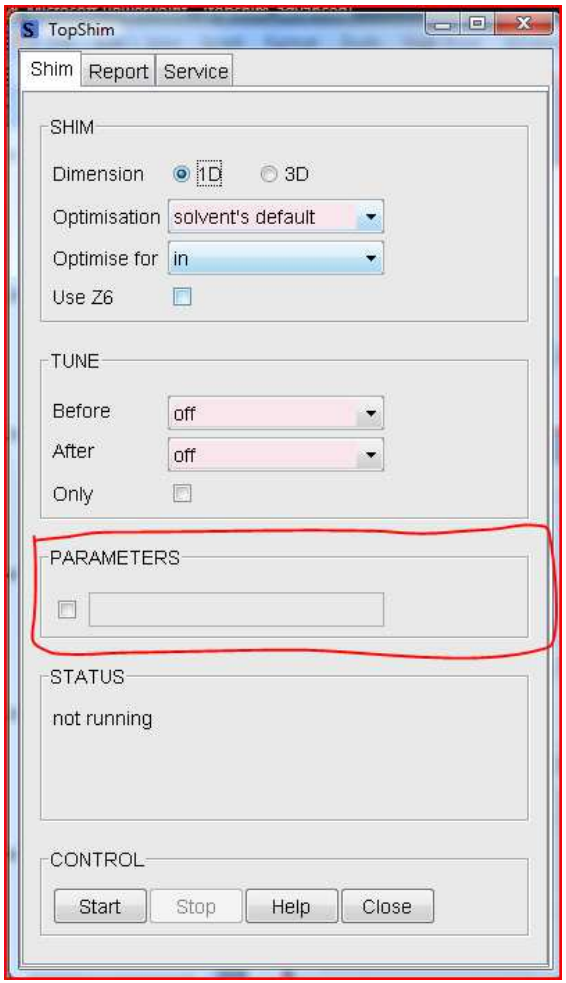


- Additional parameters **NOT checked**

Service Features in GUI

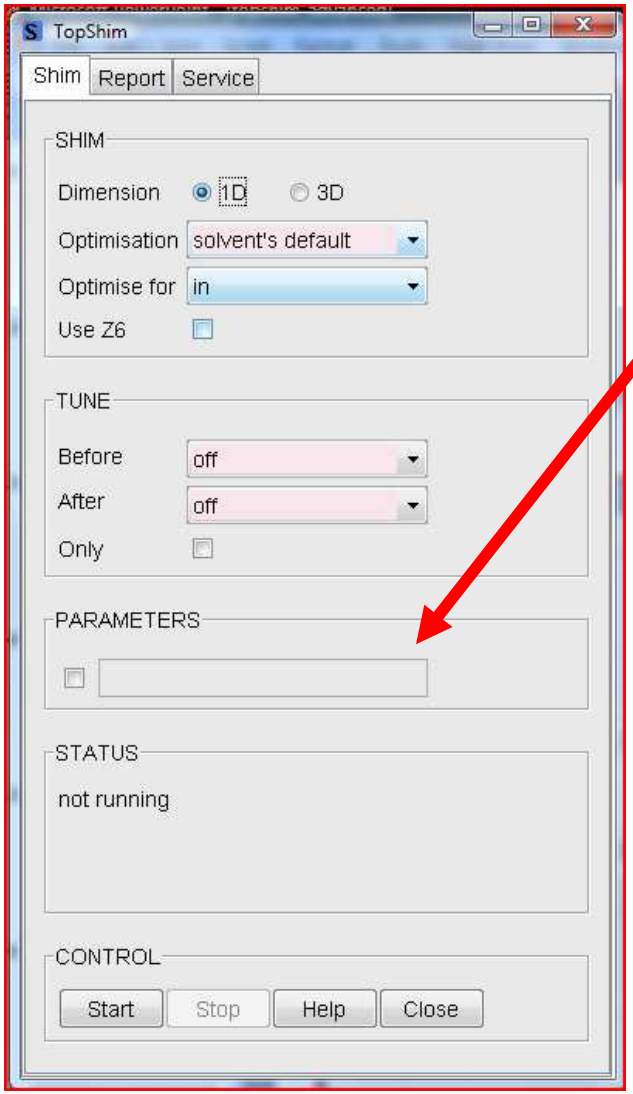






- Additional parameters **checked**

Service Features in GUI



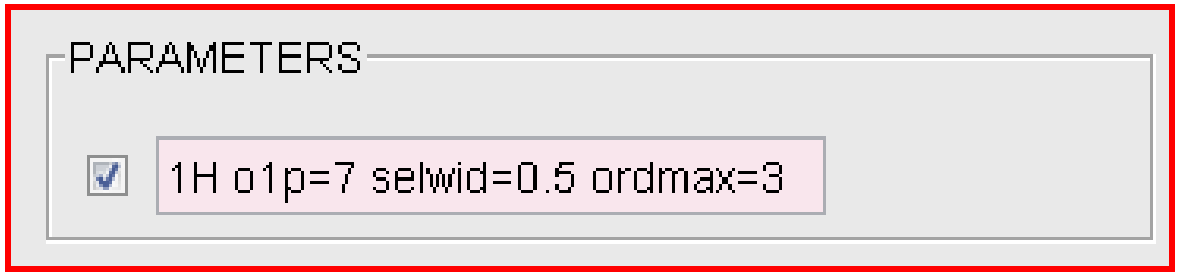
- Command line arguments can be added here when the box is checked

Command Line Arguments

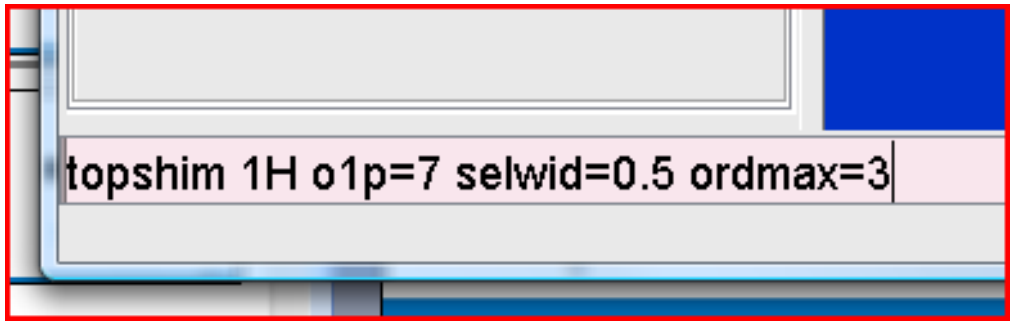
- Useful arguments
  - ordmax= Sets the maximum total order of shim functions (default = 5)
    - ordmax=3 limits shimming to Z-Z3
    - ordmax=8 limits shimming to Z-Z8
  - lockoff Enables shimming with system unlocked
  - o1p= Explicitly sets excitation frequency in PPM (o1p=2.06)
  - selwid= Enables selective excitation of a bandwidth expressed in PPM.
    - Useful when shimming on a solvent with multiple signals (selwid=0.5)
  - 1H or 2H Explicitly sets shimming nucleus
  - durmax= Increases time allowed for signal averaging (durmax=30)
  - tune* also shim on the lock before or after gradient shimming (tuneb shims X,Y,Z,XZ,YZ before running gradient shimming)
  - plot Saves data after completion
  - Convection Compensation (convcomp)

Command Line Arguments

- Command line arguments
  - Multiple arguments can be used simultaneously



  - Similarly, these arguments can be executed directly from the topspin command line.



Command Line Arguments

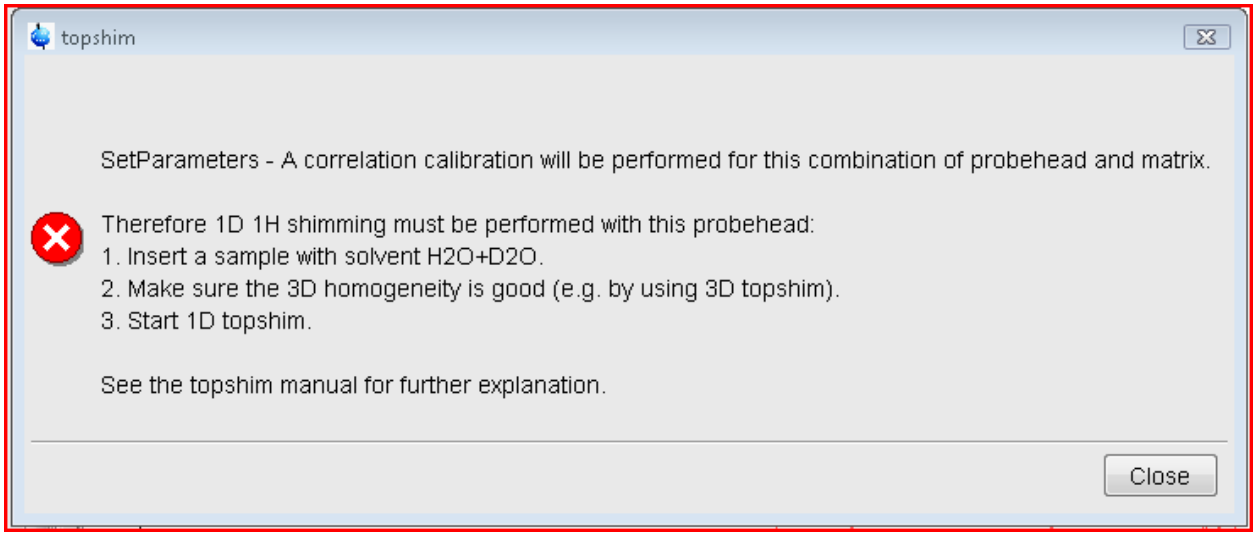
- Command line arguments (continued)
  - Topshim with arguments can be entered into a macro
    - Use command edmac
    - Use File/New pulldown to create new macro with name of your choosing
    - Enter topshim command with desired arguments
    - Use File/Save and File/Exit to save macro and exit
    - Command can now be executed using macro

Useful macros

- TOPLS (Topshim lineshape)
  - topshim ls convcomp
- TOPHU (Topshim hump)
  - topshim lshump convcomp
- TOPSS
  - topshim ss convcomp
  - or you can use “topshim 3d convcomp” as the macro to accomplish the same thing

Probe Calibration

- Calibration
  - Calibration is automatically done by topshim if no calibration data is available

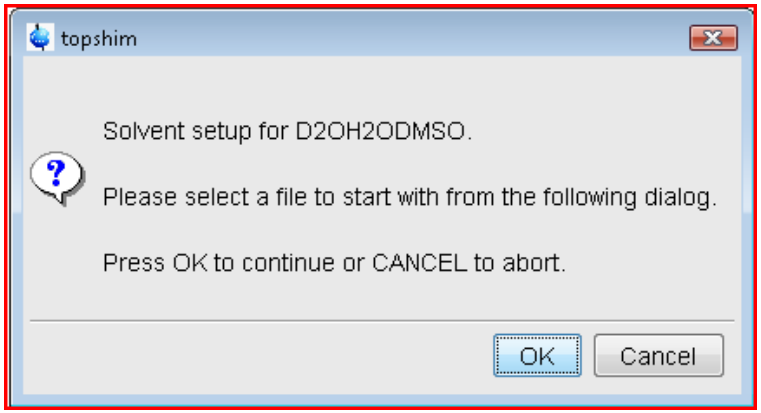


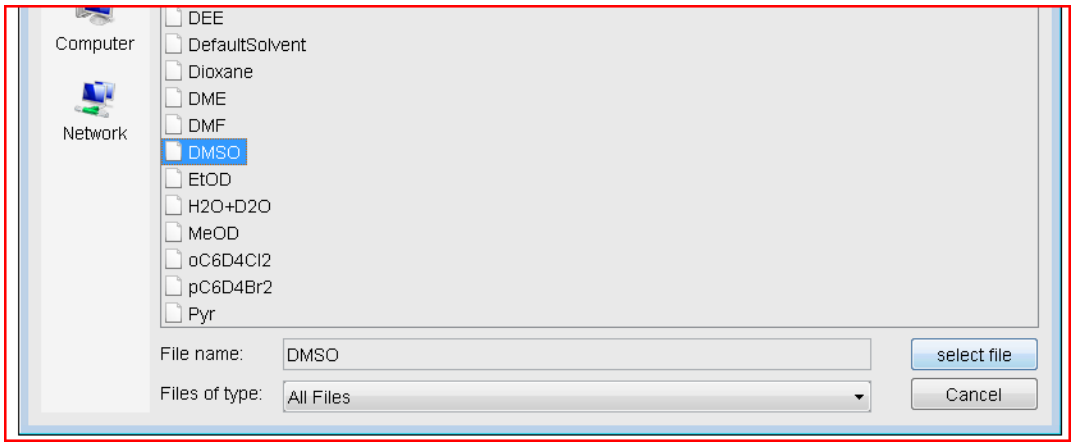
  - Must use 1H for calibration
  - Can run ***topshim 3d*** before calibration to optimize shims
  - Use command ***topshim calreset*** to delete calibration data if it is believed recalibration is required

New Solvent – 1H Shimming

- Mixed Solvent - 1H shimming (mixture of DMSO H_2O and D_2O)

- Add new solvent to solvent list and lock table.
- Run ***topshim solvcal*** from topspin command line and click OK to modify parameters.

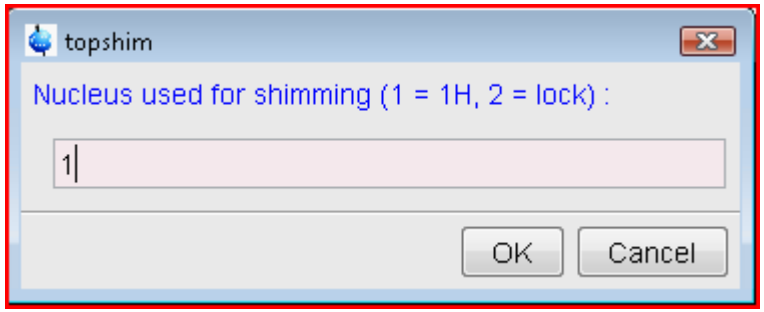




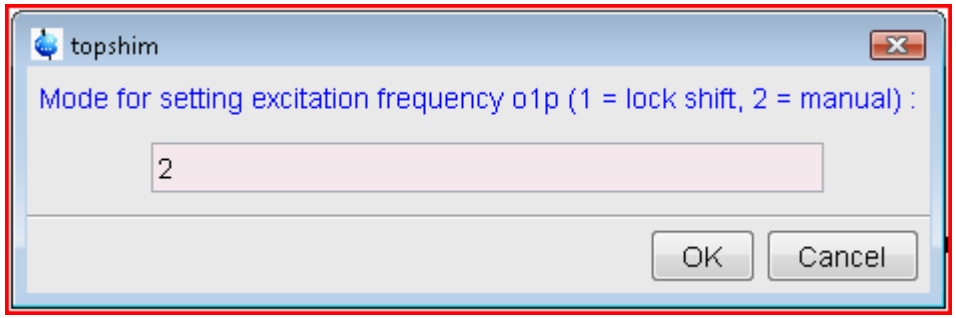
- Choose solvent closest to that used for shimming

New Solvent – 1H Shimming

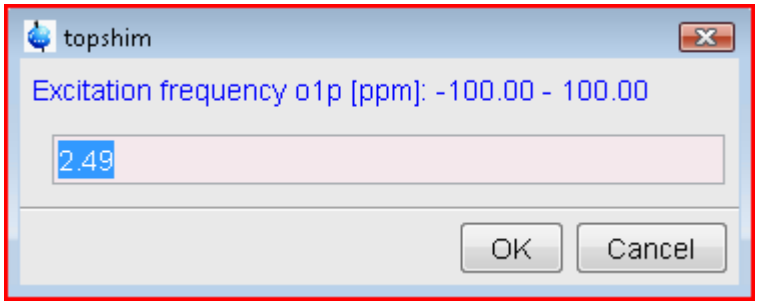
- Answer questions for solvent to be shimmed



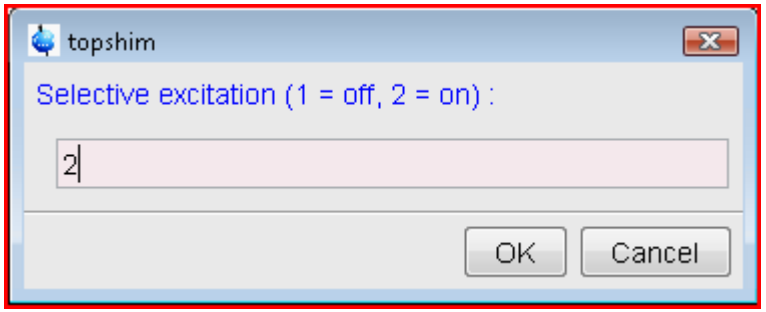
- Shim on 1H DMSO



- Not using lock shift

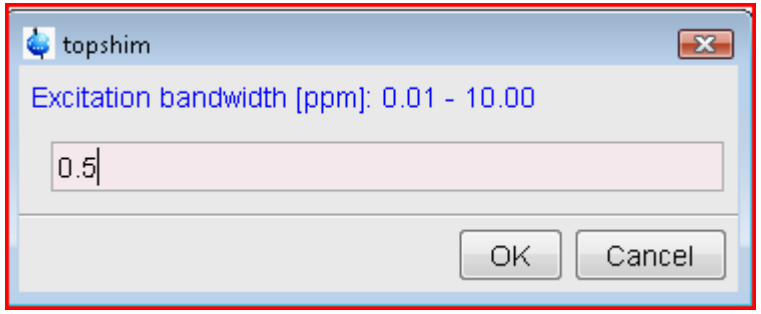


- Enter frequency (may vary in mixed solvent)

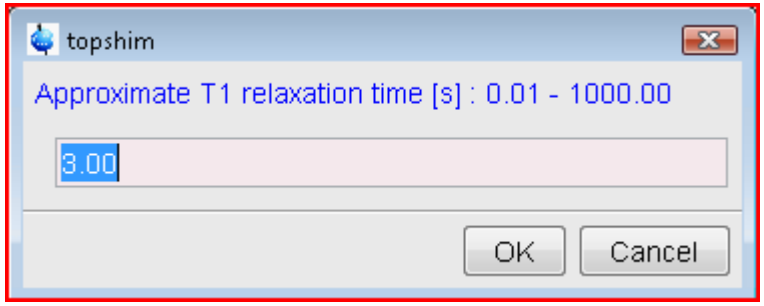


- Contains 2 1H peaks so selective excitation needed

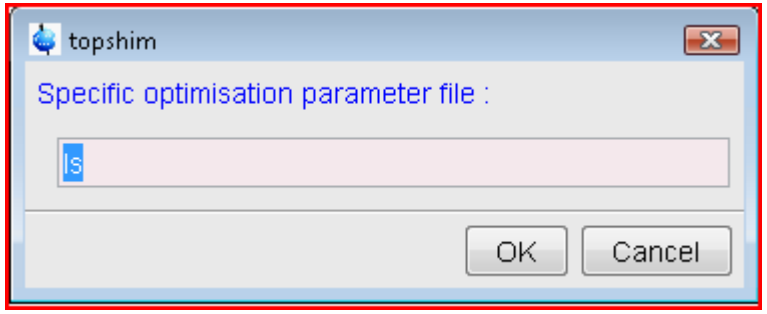
New Solvent – 1H Shimming



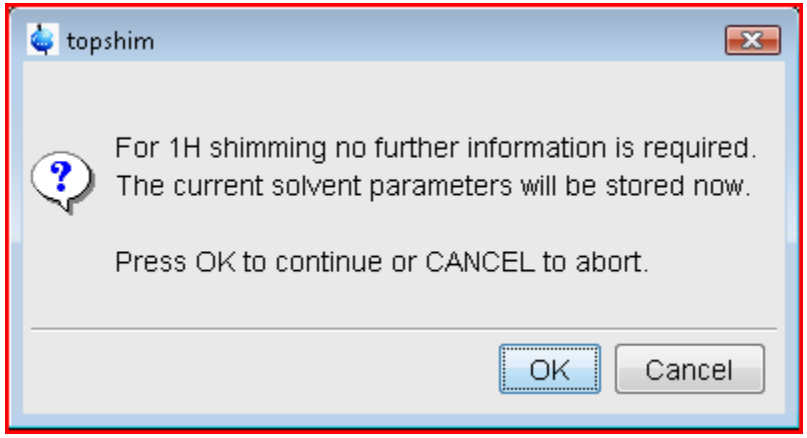
- Selective excitation bandwidth of 0.5 usually good



- T1 is read in from solvent file chosen



- Lineshape optimization chosen see manual for other options

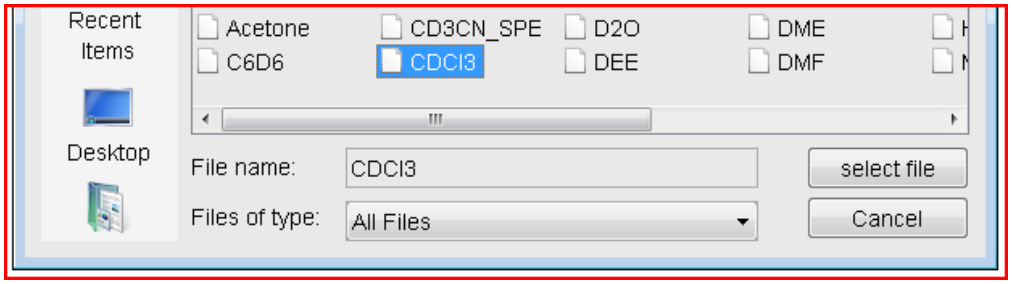


- Click OK to finish

New Solvent – 2H Shimming

- Mixed Solvent - 2H shimming (mixture of $CDCl_3$ and CD_3CN)

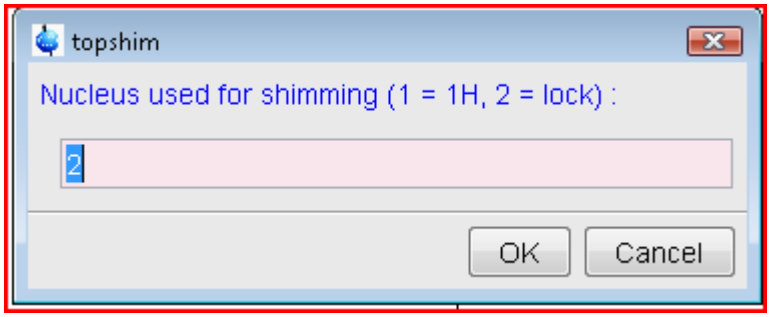
- Add new solvent to solvent list and lock table.
- Run ***topshim solvcal*** from topspin command line and click OK to modify parameters.



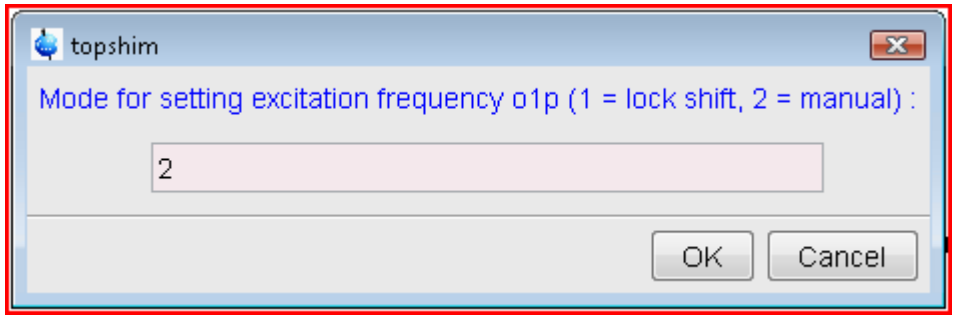
- Choose solvent closest to that used for shimming

New Solvent – 2H Shimming

- Answer questions for solvent to be shimmed



- Shim on 2H (lock nucleus)



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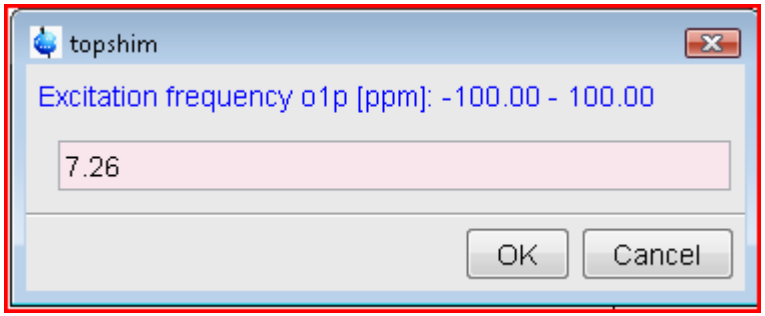
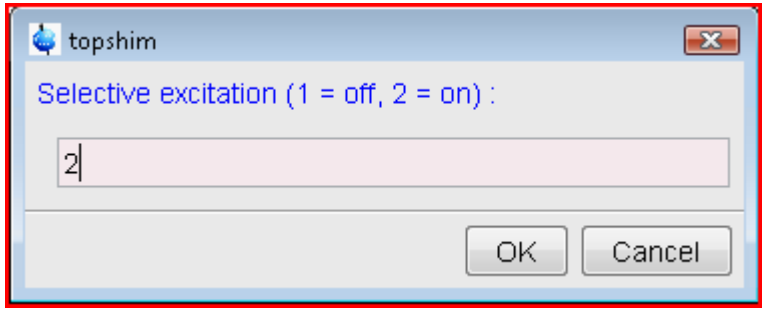
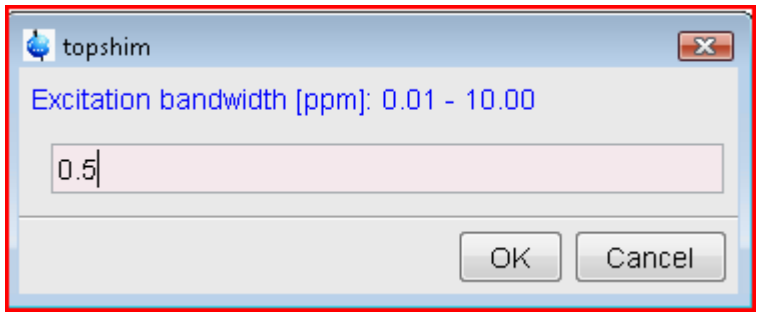


- Enter frequency



- Contains 2 2H peaks so selective excitation needed

New Solvent – 2H Shimming



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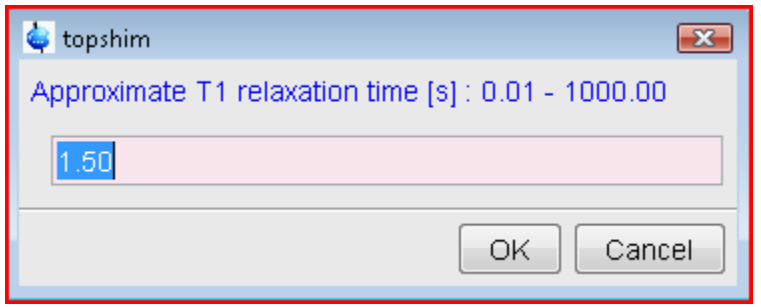

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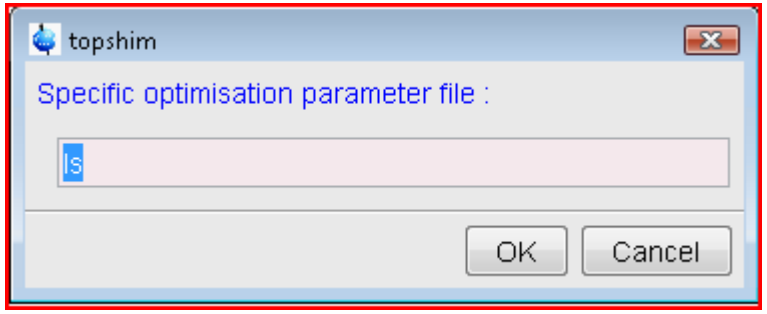

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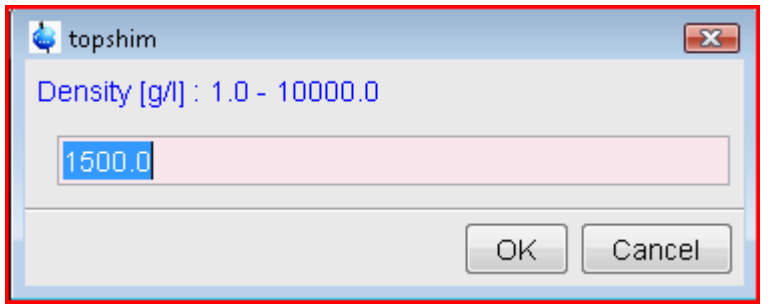
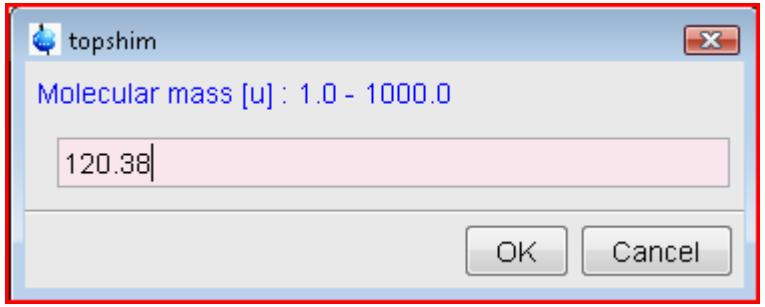
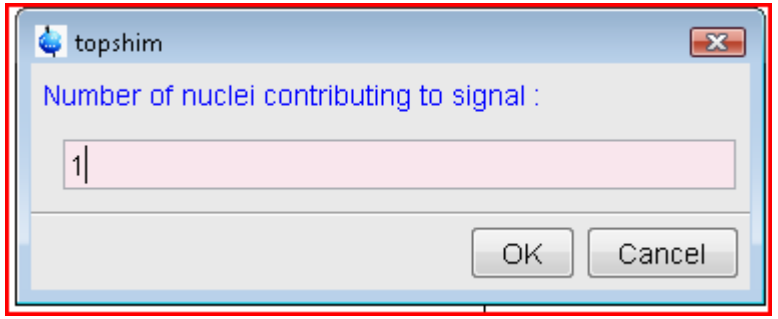


- Enter density of solvent to be shimmed on

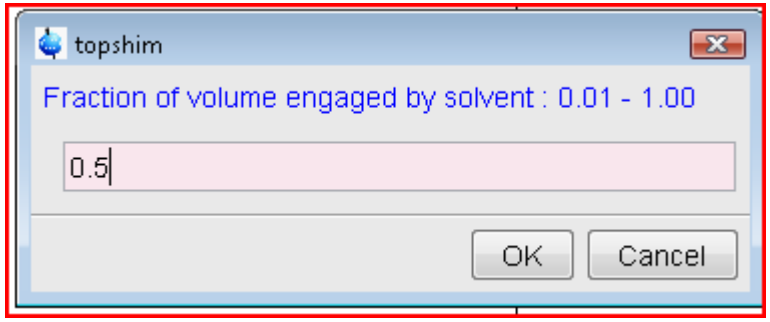
New Solvent – 2H Shimming



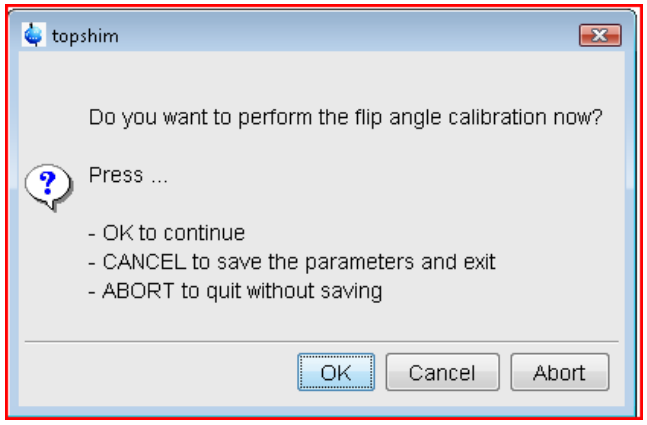
- Enter Molecular mass of solvent to be shimmed on



- Enter number of nuclei in solvent to be shimmed on



- Enter fractional volume of solvent to be shimmed on



- Click OK to preform calibration

Topshim in Automation

- Automation
  - In an au program

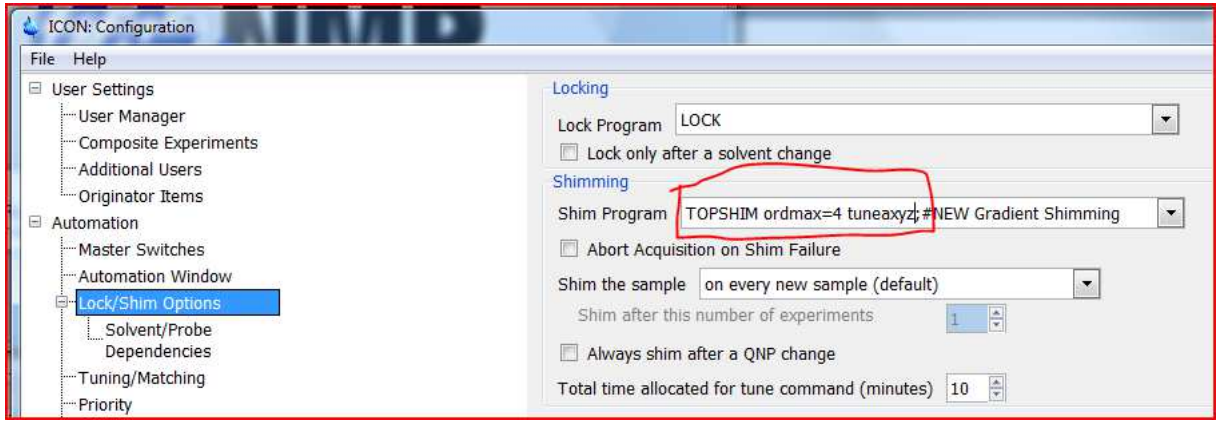
    GETCURDATA

    CPR_exec("topshim ordmax=4 tuneaxyz",WAIT_TERM);

    RGA

    ZG

    QUIT
  - Iconnmr



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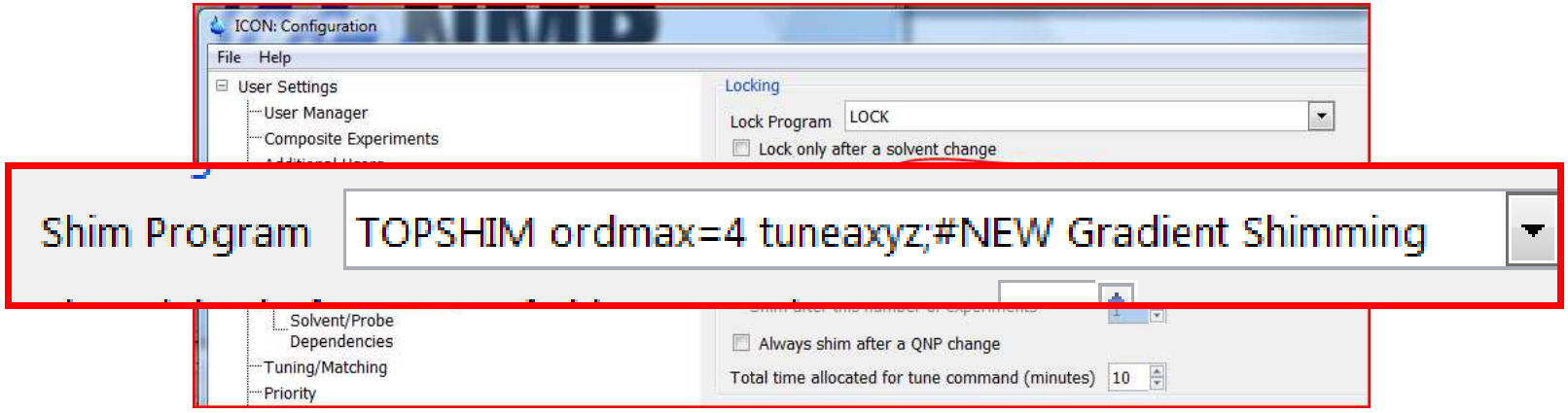
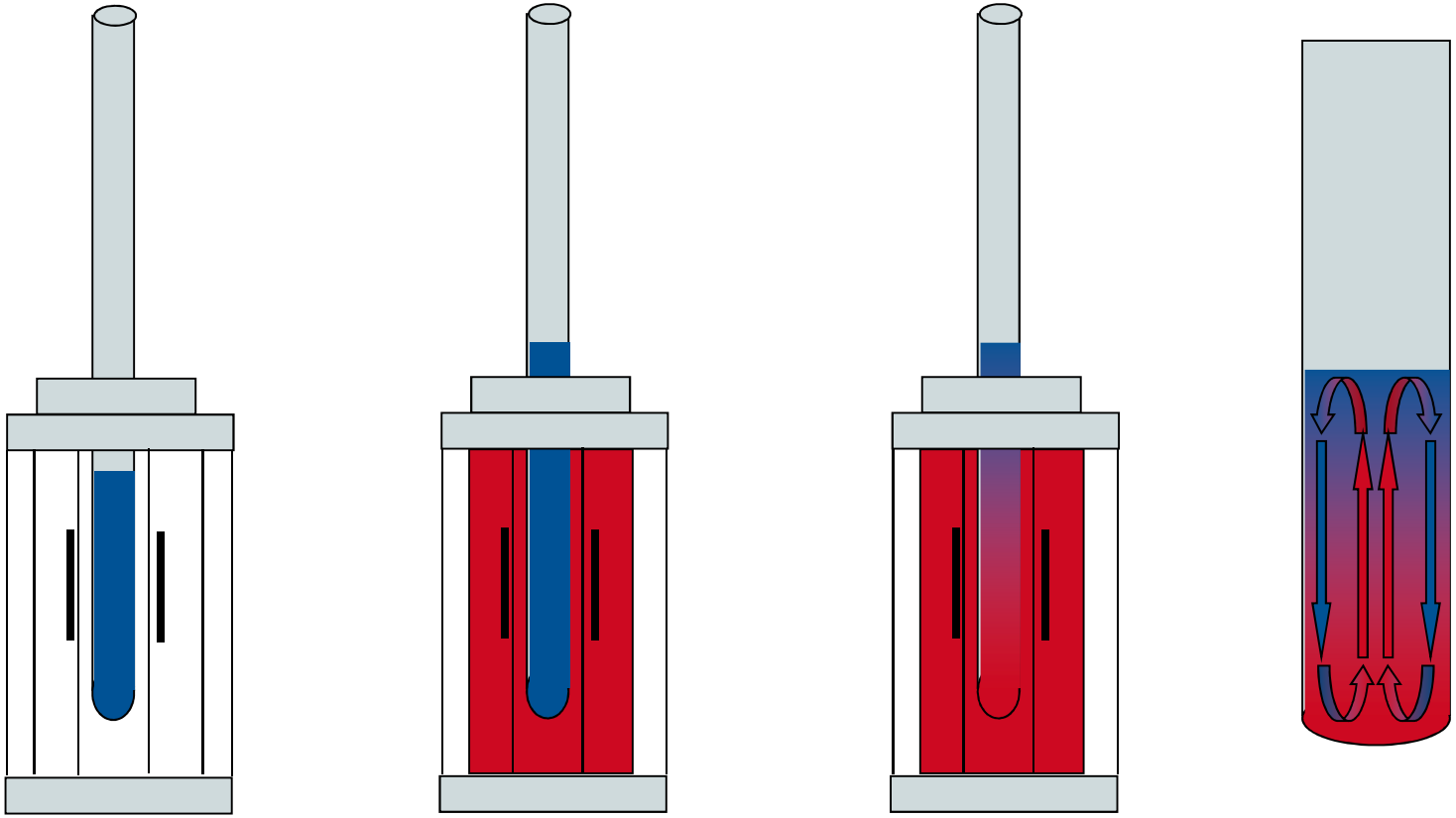


Topshim Tips

- Convection and temperature gradients
- Raising temperature + filling height too high = problems

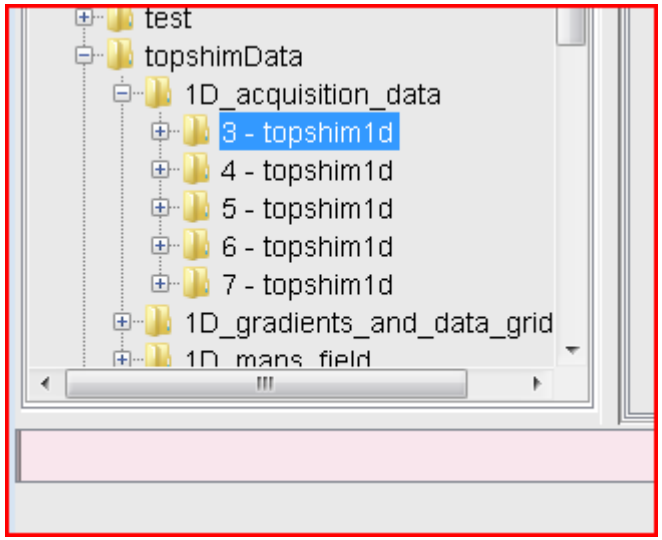


Topshim Tips

- Troubleshooting Tips
  - Check ^{2}H pulse length
  - Poor initial field homogeneity
    - Shims can be so bad that topshim cannot get data good enough to use for the calculation.
    - Try running topshim with sample spinning.
    - Try ***topshim tuneb*** to improve initial homogeneity.
  - Convection in a sample may cause topshim to fail.
    - Low viscosity solvents like chloroform and acetone are particularly susceptible.
    - Can manifest itself as "too many points lost during fit".
    - Use ***topshim convcomp*** to run topshim with convection compensation. > 2.1pl5.
  - S/N too low
    - Try running ***topshim durmax=30***, 60, or even 120

Topshim Tips

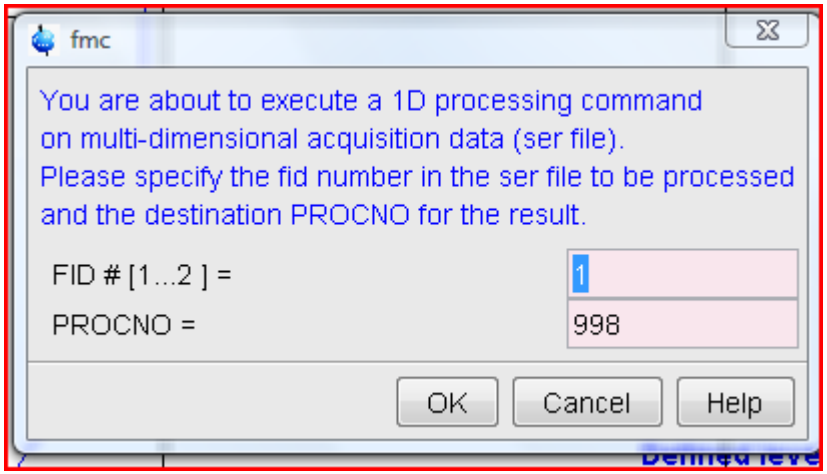
- Troubleshooting Tips
  - Topshim deletes data immediately after execution. Command ***topshim plot*** can be used to save topshim data



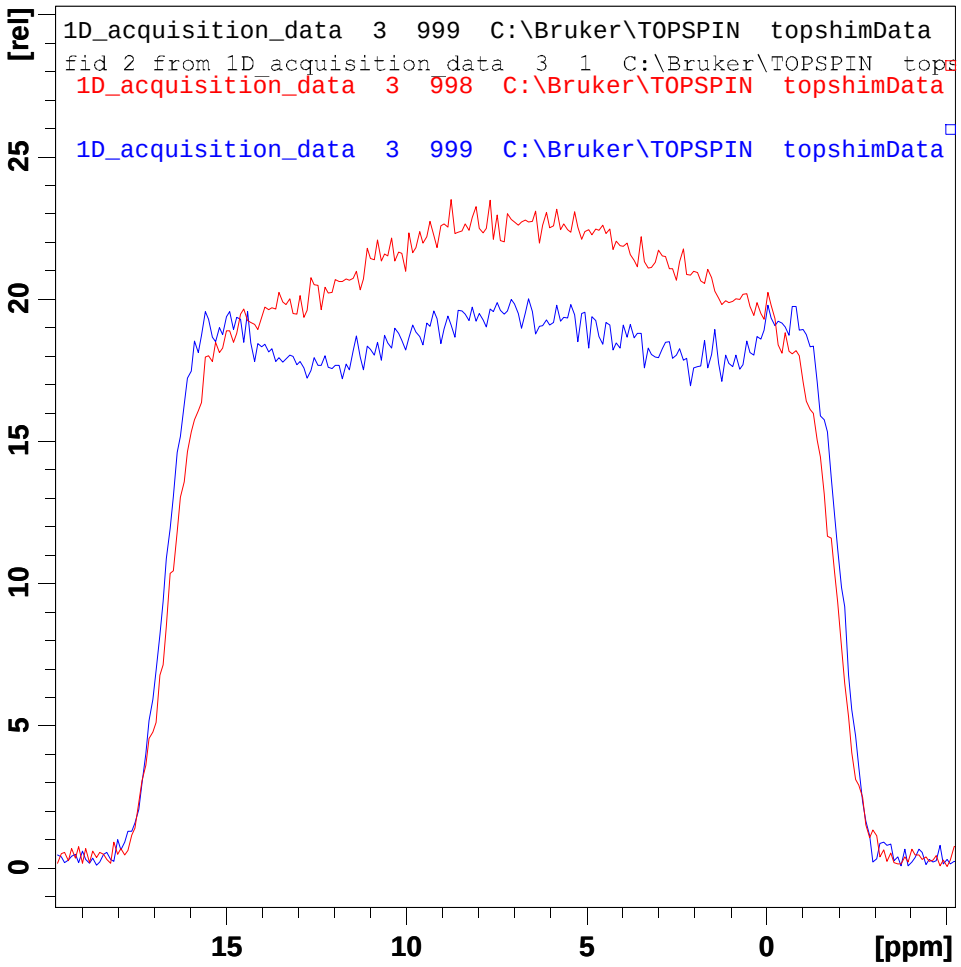
  - Data located in ***[topspin_home]*** /data

Topshim Tips

- Troubleshooting Tips
  - Data is stored in serial file. Long echo time in FID 1 and short echo time in FID 2. Process with ***fmc***



Topshim Tips

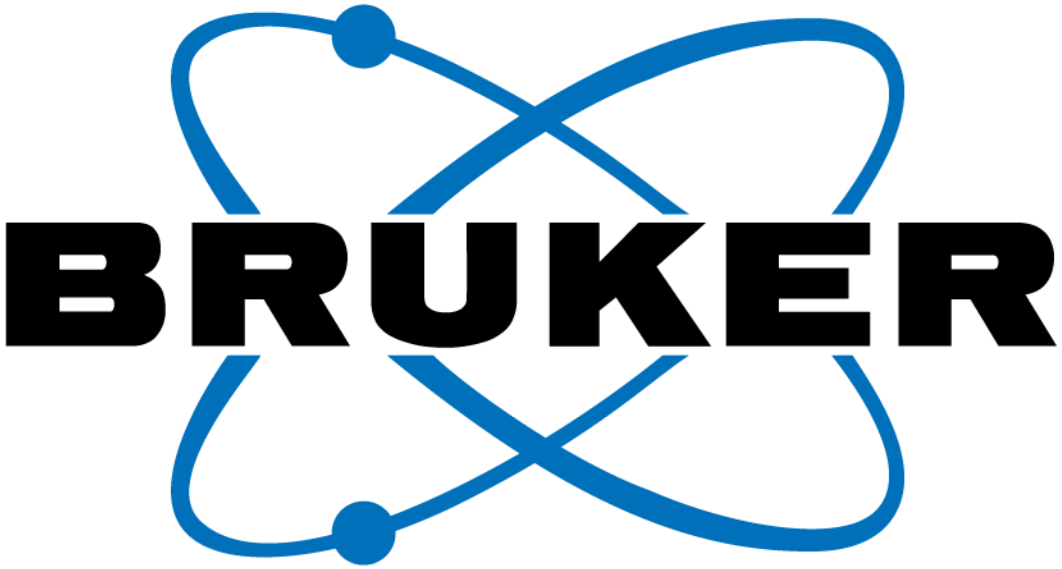


- Profiles should have similar intensity. If long echo time spectrum is <0.5 then there may be homogeneity issues.
- For 2H, check pulse length
- Type ***help topshim*** in Topspin command line to open topshim manual

How to shim a probe from scratch with TopShim

- Insert 90% H2O+10% D2O (standard sucrose sample); lock it, tune the probe, no rotation
- topshim cal (if needed)
- topshim 3d convcomp
- topshim ls convcomp
- Write the shim file
- Test presaturation if it is a BBI probe use the **WATERSUP** parameter set to test
- Insert line shape sample (1% CHCl3 in Acetone-d6)
- Tune the probe, rotate
- topshim ls convcomp
- Test with the parameter set **PROHUMP** and *humpcal*
- Write the shim file.

Are there any questions?



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