



# TopShim Advanced Tips and Tricks

Presented by Mike Brown 03192012

Bruker **BioSpin** 



## Advanced Topshim

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



S TopShim	
Shim Report Service	
GENERAL	
About Help	
GUI DEFAULTS	
Save Load Restore	
SETUP	
Cf Edhead Gradamp	
Info	
DIAGNOSTICS	
Report Logfile	
PREFERENCES Additional parameters External	

- 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



S TopShim
Shim Report Service
GENERAL
About Help
GUI DEFAULTS
Save Load Restore
SETUP
Cf Edhead Gradamp
Info
DIAGNOSTICS
Report Logfile
PREFERENCES
Additional parameters 💟 External 💟

- 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



S TopShim
Shim Report Service
GENERAL
About Help
GUI DEFAULTS
Save Load Restore
SETUP
Cf Edhead Gradamp
PROBE CALIBRATION
Info
DIAGNOSTICS
Report Logfile
PREFERENCES Additional parameters External

Top	Shim	and Report Same in	- O X
Shim	Report Servi	се	
1	SHIM		
	Dimension		
	Optimisation	solvent's default	
	Optimise for	in 🔹	6 //
	Use Z6		
	TUNE		
	Before	off	
	After	off 🔹	
	Only		
	STATUS		
	-CONTROL	Stop Help Clo	ose
	<u></u>		

 Additional parameters <u>NOT</u> checked



S TopShim
Shim Report Service
GENERAL
About Help
GUI DEFAULTS
Save Load Restore
SETUP
Cf Edhead Gradamn
PROBE CALIBRATION
Info
DIAGNOSTICS
Report Logfile
PREFERENCES
Additional parameters 📝
External

Shim	
Report Ser	vice
SHIM	
Dimension	
Optimisation	n solvent's default
Optimise for	r in 🔹
Use Z6	
TONE	
Before	off
After	off
Only	
-STATUS	
-CONTROL-	
CONTROL-	Stop Help Close

S TopShim	
Shim Report	Service
SHIM	
Dimension	● 1D ○ 3D
Optimisation	solvent's default
Optimise for	in 👻
Use Z6	
Before	off
After	off
Only	
PARAMETER	25
STATUS	
not running	
+	2
CONTROL-	
Start	Stop Help Close

• Additional parameters checked



S TopShim	
Shim Report	Service
SHIM	
Dimension	
Dimension	
Optimisation	solvent's default
Optimise for	<u>in</u>
Use Z6	
TUNE	
Before	off
After	off
Only	
PARAMETER	IS
STATUS	
not running	
CONTROL-	
Start	Stop Help Close

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

Bruker **BioSpin** 

## **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

PARAMETERS			
<b>V</b>	1H o1p=7 selwid=0.5 ordmax=3		

• 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

🖕 top	pshim
8	SetParameters - A correlation calibration will be performed for this combination of probehead and matrix. Therefore 1D 1H shimming must be performed with this probehead: 1. Insert a sample with solvent H2O+D2O. 2. Make sure the 3D homogeneity is good (e.g. by using 3D topshim). 3. Start 1D topshim.
	See the topshim manual for further explanation.
	Close

- 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



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





• Answer questions for solvent to be shimmed

🖕 topshim 🛛 💽		
Nucleus used for shimming (1 = 1H, 2 = lock) :		
1		
OK Cancel		

• Shim on 1H DMSO

🖕 topshim 📃	3	
Excitation frequency o1p [ppm]: -100.00 - 100.00		
2.49		
OK Cancel		

• Enter frequency (may vary in mixed solvent)

🖕 topshim 💽 💌
Mode for setting excitation frequency o1p (1 = lock shift, 2 = manual) :
2
UK Cancel

• Not using lock shift



 Contains 2 1H peaks so selective excitation needed Bruker BioSpin



🤤 topshim 🛛 🔀
Excitation bandwidth [ppm]: 0.01 - 10.00
0.5
OK Cancel

 Selective excitation bandwidth of 0.5 usually good

🖕 topshim 🛛 💽
Specific optimisation parameter file :
s
OK Cancel

 Lineshape optimization chosen see manual for other options



• T1 is read in from solvent file chosen

🍬 top	oshim 💽
ৃ	For 1H shimming no further information is required. The current solvent parameters will be stored now.
	Press OK to continue or CANCEL to abort.
	OK
	Press OK to continue or CANCEL to abort.

Click OK to finish



- Mixed Solvent 2H shimming (mixture of CDCl<sub>3</sub> and CD<sub>3</sub>CN)
- Add new solvent to solvent list and lock table.
- Run topshim solvcal from topspin command line and click OK to modify parameters.



Recent Items	Acetone		D2O	С DM С DM	
Desktop	<ul> <li>File name:</li> <li>Files of type:</li> </ul>	CDCI3			select file Cancel

 Choose solvent closest to that used for shimming



• Answer questions for solvent to be shimmed

🤹 topshim 🛛 💓	X
Nucleus used for shimming (1 = 1H, 2 = lock) :	
2	
OK Canter	

• Shim on 2H (lock nucleus)

🤹 topshim 🛛 💌
Excitation frequency o1p [ppm]: -100.00 - 100.00
7.26
OK Cancel

• Enter frequency

🖕 topshim	×
Mode for setting excitation frequency o1p (1 = lock shift, 2 = m	anual) :
2	]
ОКС	ancel

• Not using lock shift

🤹 topshim	×
Selective excitation (1 = off, 2 = on) :	
2	
ОКС	ancel

 Contains 2 2H peaks so selective excitation needed Bruker BioSpin



🖕 topshim	<b>—</b>
Excitation bandwidth [ppm]: 0.01 - 10	.00
0.5	
(	DK Cancel

 Selective excitation bandwidth of 0.5 usually good

🖕 topshim	<b>—</b>
Specific optimisation parameter fi	ile :
Is	
	OK Cancel

 Lineshape optimization chosen see manual for other options



• T1 is read in from solvent file chosen

🖕 topshim	×
Density [g/l] : 1.0 - 10000.0	
1500.0	
	OK Cancel

• Enter density of solvent to be shimmed on



🖕 topshim	<b>—</b>
Molecular mass [u] : 1.0 - 1000.0	
120.38	
	OK Cancel

• Enter Molecular mass of solvent to be shimmed on

🧅 topshim	<b></b>
Fraction of volume engaged by so	lvent : 0.01 - 1.00
0.5	
	OK Cancel
	-

• Enter fractional volume of solvent to be shimmed on

🤹 topshim 🛛 💌
Number of nuclei contributing to signal :
1
OK Cancel

• Enter number of nuclei in 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



## **Topshim in Automation**



- Automation
  - In an au program

**GETCURDATA** 

CPR\_exec("topshim ordmax=4 tuneaxyz",WAIT\_TERM);

RGA

ZG

QUIT

Iconnmr

4 ICON: Confic	uration	
File Help		
User Setting User Ma Compos	is nager te Experiments	Lock Program LOCK   Lock only after a solvent change
Shim Program	TOPSHIM of	rdmax=4 tuneaxyz;#NEW Gradient Shimming 💌
Solv Depa ⊤Tuning/ Priority	ent/Probe ndencies Matching	Always shim after a QNP change  Total time allocated for tune command (minutes) 10



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





- Troubleshooting Tips
  - Check <sup>2</sup>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



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



Data located in [topspin\_home] /data



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

🤹 fmc		
You are about to execute a 1D processing command on multi-dimensional acquisition data (ser file). Please specify the fid number in the ser file to be processed and the destination PROCNO for the result.		
FID # [12] =	1	
PROCNO =	998	
OK Cancel Help		





- 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?



## www.bruker-biospin.com

Bruker **BioSpin**