TopShim

- Automatic Shimming

User Manual

Version 003

Innovation with Integrity

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1 Introduction

1.1 About TopShim

TopShim is a tool designed for easy and automatic shimming.

Functionality

The core method of TopShim is gradient shimming. This is complemented by a spectrum optimization approach, where a quality criterion for the final lineshape ensures the best results for all possible situations.

Both 1D and 3D shimming modes are provided to adjust only the on-axis or both the on- and off-axis shim functions, respectively.

The acquisition of the B0 field map data can be carried out with 1H, 2H or 19F observation, enabling the use of TopShim for protonated, deuterated or fluorinated solvents.

Optionally the additional tune functionality can be applied after gradient shimming in order to maximize the lock level by adjusting low order shims for maximum lock level.

User Interface

TopShim can either be operated via the TOPSPIN command line or, alternatively, the TopShim GUI can be used. While the GUI provides convenience and easy access for all users to perform the regular functions, expert users have the possibility to use the command line approach in order to taylor TopShim for their needs.

TopShim is also suitable to be used in NMR automation with ICON-NMR or other comparable tools.

1.2 Quick Guide

Probe Setup

- Always after changing the probe declare it with **edprobe**.
- Ensure the ^{1}H, ^{2}H and/or ^{19}F 90° hard pulses are correctly defined in **edprosol**.

Preparation

1. Open the data set of your planned NMR experiment.
2. Lock the sample using the **lock** command.

Shimming

topshim (1d)	1D shimming
topshim 3d	3D shimming (only ^{1}H or ^{19}F)
topshim hrmas	HRMAS shimming
topshim stop	abort shimming
topshim gui	open the graphical user interface

Note the shimming procedure does not have a fixed duration but is continued until the optimum homogeneity has been achieved.

Introduction

As a general rule please note that in order to work properly 1D shimming requires good 3D homogeneity. It is recommended to run **topshim 3d** on a regular basis on a water sample to assure the homogeneity. After changing back to the sample to measure, run **topshim** and adjusting the low order offaxis shims for maximum lock level.

Shimming a probe the first time

When shimming a probe the first time, for optimal performance the following preparative steps should be executed:

1. Enter a sample with a strong ^{1}H or ^{19}F signal, ideal is e.g. solvent H2O+D2O (the standard sucrose test sample). Then lock, tune and match.
2. Perform initial shimming: **topshim initial**.

The "**initial**" mode is concipated to be extremely robust and should be succesful even if all the room temperature shims are set to zero. It can also be executed without locking to the solvent by adding the option "**lockoff**", assuming the solvent signal can be detected.

If "**initial**" is not executed then TopShim will need to generate various calibration files using the sample at hand. This can be done even on a 2H sample, but TopShim will warn against doing this since this requires quite some time, and because the calibration is generally better when using samples that generate strong signals.

2 Prerequisites

TopShim works properly only if the spectrometer fulfils the prerequisites concerning hardware and software as described below.

2.1 Hardware

- **Spectrometer**
  Avance III or later
- **Shim system**
  All BOSS shim systems with electronic coding. These are:

  BOSS1-SB with 20 shim coils (not 13, 17, or 19)

  BOSS1-WB ("WB17") with plug

  BOSS2

  BOSS-WB ("WB99")

  BOSS3
- **RCB**
  3D shimming requires the real-time control board (RCB) functionality. On some systems this might be provided by a 2H-TX board, or directly by some versions of the BSMS. Otherwise, an RCB must be installed.
- **Lockswitch**
  ^{2}H shimming requires the lockswitch functionality in order to use the lock channel for standard acquisition. This feature is usually provided by a 2H-TX board, but on some systems may be part of the AQS or part of the RF amplifier. Otherwise, a lockswitch board must be installed.
- **Gradient amplifier**
  All types of GAB and GREAT are supported. The GREAT/E should have at least ECL01.
- **Probe**
  The probe must be equipped with a Z or XYZ gradient. All high-resolution NMR probes produced by Bruker Switzerland and flow probes produced by Bruker Germany are supported

2.2 Software

Make sure that all device firmware is up-to-date for the installed TopSpin version. This is required for **topshim** to work properly.

3 Setup

3.1 General Probe Setup

For the correct operation of TopShim the following standard procedures must be performed for each probe.

- After changing the probe it must always be declared using **edprobe**.
- For ^{1}H, ^{2}H and ^{19}F shimming the respective 90° hard pulses of a probe must be entered for the correct amplifier into the prosol table using **edprosol**. Note that solvents with high conductivity may require individual pulses (see *Solvent-Specific Pulses [▶ 21]*).

3.1.1 Probes with CryoFit

If your standard probe is operated as a flow probe using the CryoFit kit you need to declare that with the **edprobe** tool.

In the **Production Parameters** set **use as flow probe** to **yes**. This gives you access to the **Probe Flow Parameters** that must be set correctly.

3.2 TopShim Spectrometer Setup

All hardware information for TopShim are provided by TopSpin, no special setup is needed.

However, make sure that the TopSpin commands

cf

expinstall

Are executed after any change of hardware.

3.2.1 Shim System BOSS1-SB

Shim systems without the resistance coding through the connections are no longer supported.

3.2.2 Shim Matrix File

This section is only relevant for you if your spectrometer is equipped with a BOSS-WB matrix shim system.

A default matrix file is loaded into the BSMS from the TOPSPIN installation in the directory

`$TOPSPINHOME/conf/instr/servtool/bsmstool/boss`

There are optimized matrix files available if performance is not satisfactory. In this case please follow the instructions given in the file `wb_inst_e.pdf` located in the subdirectory `wb`.

3.2.3 Multiple Gradient Amplifier

If more than one gradient amplifier is available in your spectrometer TopShim will use the gradient amplifier selected in TopSpin.

3.3 TopShim Probe Setup

After a probe change, **edprobe** should be run within TopSpin before starting Topshim

3.3.1 Probes without PICS

Probes without PICS are no longer supported. If your PICS version is not up-to-date for the TopSpin version contact the HelpDesk.

4 Using TopShim

4.1 General

4.1.1 Basic Functionality

The core functionality of TopShim is based on gradient shimming (see sections 1-3). The two main methods are 1D and 3D shimming.

1D shimming is able to adjust the on-axis shim functions. These are the axially symmetric functions Z, Z2, Z3, etc. 1D shimming can be performed using ^{1}H, ^{2}H or ^{19}F data acquisition, hence samples with protonated, deuterated or fluorinated solvents can be used. The duration of 1D shimming will vary from several seconds up to a few minutes depending upon the available signal strength.

3D shimming uses all shim functions, i.e. the on-axis as well as the off-axis shim functions, such as X, Y, XZ, etc. 3D shimming can only be performed on samples having a strong reference signal, hence only with ^{1}H or ^{19}F data acquisition. Ideally an aqueous samples or samples providing an alternative strong ^{1}H or ^{19}F signal will do. 3D shimming takes between 5 and 30 minutes, depending on the initial B0 homogeneity. The 3D shimming includes a flowing 1D shim run including optimization.

The default shimming method is 1D, with the nucleus used for data acquisition (^{1}H, ^{2}H or ^{19}F) selected automatically based on the lock solvent name. 3D shimming, or other non-default methods, can be selected via command line options or using the graphical interface to TopShim.

4.1.2 User Interface

The principle of TopShim is to provide a push-button functionality where only the very basic decisions have to be made by the user. Advanced functionality is available but may require expert knowledge.

There are two ways of using TopShim: via the TOPSPIN command line or the graphical user interface (GUI).

The command line version allows direct operation. The basic commands are:

topshim initial	Shim new probe, including calibration steps
topshim (1d)	1D shimming
topshim 3d	3D shimming
topshim hrmas	HRMAS shimming
topshim stop	abort shimming

Furthermore, if desired a number of parameters can be added to manipulate TopShim (see *4.5 [▷ 16]*).

Alternatively, the simple and easy-to-use GUI serves to give access to the most important parameters. Furthermore, the process of shimming is documented perpetually. The GUI can be opened with the command **topshim gui** or from the TOPSPIN menu: **Spectrometer | Shim control | Automatic Shimming**. See *4.4 [▷ 13]* for a detailed description of the GUI.

4.1.3 Shimming

To successfully shim a sample, TopShim needs to have additional information as of what nucleus to use during shimming, which nucleus to optimize for and chemical shift due to the solvent. While this information can be specified explicitly in a TopShim command line (see *4.5.2 [▷ 16]* for details), the program can also determine this information automatically from the dataset to be used next and the lock solvent.

For convenience we recommend the following steps:

1. Open the data set of the planned NMR experiment
   TopShim reads the observe nucleus of the planned experiment, the NMR nucleus, to determine the coil used for the lineshape optimisation procedure performed during shimming (see *4.2 [▷ 12]*). If no data set is open, ^{1}H is taken by default.
2. Lock the sample for the correct solvent using the lock command
3. Perform shimming

Type the desired TopShim command in the TOPSPIN command line or open the GUI and start shimming.

TopShim will finish after the optimum shim has been obtained. The required number of iterations is not known a priori, hence the "residual time" in the "acquisition information" of the acquisition status bar is described as „unknown".

> **i** To take advantage of the automatic setup, TopShim requires the system to be locked in. Then TopShim reads the name of the last used lock solvent and sets other parameters accordingly (see *4.6 [▷ 20]*). It is important that the solvent name used for locking is correct. Note in particular that the shimming method for D2O or H2O+D2O will be different (^{2}H and ^{1}H respectively). If the sample is not locked, the chemical solvent shift needs to be provided by the o1p parameter, e.g. **topshim 2h o1p=4.7** for aqueous solvents.

4.1.4 Important Rules

For optimum performance of TopShim please note the following rules:

1D shimming generally requires good 3D homogeneity

> **i** Therefore, 3D shimming on a water sample should be performed at least once on a probe. After changing the sample the low order offaxis shims should be readjusted, e.g. by using the TopShim tune functionality (see *4.3 [▷ 12]*). If the sample is spinning this requirement is not quite as crucial.

Sample spinning state (only for HR-liquids, not for HRMAS)

1D shimming should always be performed in the same sample spinning state as the planned NMR experiment. For 3D shimming spinning will be temporarily switched off.

Initial homogeneity

If the initial homogeneity is bad due to the first time shim or a probe change from a different type the command **topshim initial** or **topshim 3d** should be use to establish a good overall shim.

TopShim can calibrate on any sample in principle, although time requirements may differ strongly, but the quality of the calibration data is improved by calibrating on a sample with a strong signal. The H2O+D2O solvent (90%/10%) is ideal for such purposes.

Repeating shimming

In general after a single run the optimum homogeneity should have been found. Hence usually starting TopShim with the same parameters again will not bring any further improvement and shimming should stop after acquisition of the first field map. However, there may be rare cases where repeating the identical procedure can have a positive effect.

Sample temperature

After changing the sample you need to wait for a sufficiently long time (e.g. 60 to 120 sec) to allow the sample to reach a stable temperature, depending on the difference of the room and the sample target temperature. Otherwise, a shim setting optimal at the initial temperature may not be appropriate at a later stage.

Furthermore, operating close to the boiling point of a sample should be avoided as this may cause serious signal loss during the shimming acquisition due to convection. Minor cases of convection may be counteracted by the option **convcomp**.

Temperature gradients in the probe may also cause issues as the solvent may respond differently from the signals of interest. In such cases the option **tcorr** may be used.

Gas flow

Finally, you should always make sure that the gas flow is set correctly as specified for your probe. This means both the gas type (air or nitrogen) and the flow rate. Deviating from recommended values may cause issues with shimming.

4.1.5   Shimming a HR Liquids Probe the First Time

As noted above, 1D shimming generally requires good 3D homogeneity. Therefore, 3D shimming should be performed on a water sample before 1D shimming, in case the off-axis shims are not optimal yet.

Furthermore, during the first 1D run of TopShim for a probe several calibration steps will be performed. TopShim can calibrate on any sample in principle, although time requirements may differ strongly, but the quality of the calibration data is improved by calibrating on a sample with a strong signal. The H2O+D2O solvent (90%/10%) is ideal for such purposes.

Hence, when using TopShim the first time on a probe the following procedure should be executed:

1. Use a sample with the solvent H2O+D2O, e.g. the standard sucrose sample used for water suppression tests. The sample should have the largest diameter used with this probe. Furthermore it should be filled not less than given by the filling height specified for the probe. The spinning state does not matter. Lock, tune and match.

2. Perform 3D shimming: **topshim initial**

The command **topshim calinfo** shows if the calibration has already been performed for the current probe. If ever the calibration procedures were performed outside the **topshim initial** command, they can always be repeated on a water sample with **topshim initial** later. Alternatively, the calibration data can be deleted by executing **topshim calreset**. It is recommended to do a **topshim initial** after this to get good calibration data.

4.1.6   Shimming a HRMAS Probe

Shimming a HRMAS probe requires only the hrmas command, irrespective of whether the probe is shimmed for the first time, or not. Any missing calibration steps will be executed if needed.

The first time a HRMAS probe is shimmed, or after deleting the calibration files, one specific parameter must be specified by the user and that is the orientation of the probe inside the shim system.

HRMAS probes have gradients that are aligned along the MAS rotor and therefore it is beneficial to work in the MAS frame of reference. For this to work the shim functions are recombined to generate on-axis shim functions in the MAS frame (off-axis shim functions are not useful due to the high spinning rates). This typically reduces shimming to only 3 shim functions, Z1', Z2' and Z3', although for BOSS3-SB shim systems Z4' is in fact allowed to be set manually using the **topshim changemasshims** command. The Z4' function is only about 90% pure, i.e. off-axis shim functions are also generated, which is good enough to be useful

4.1.7 Selection of Shim Functions Used

The set of shims actually used for shimming is selected based on the respective situation. If a shim is modified or not, depends on its effectiveness in the sensitive sample volume in relation to the current B0 homogeneity and on the current resources used. Hence also the size of the sample and the RF coil play a role for this selection.

The set of shims used for shimming is constantly updated while the homogeneity improves. Therefore, shimming can also be started with bad initial homogeneity.

There is an upper limit defined for the order of the shim functions used by TopShim. See the description of **ordmax** in *4.5.2 [▷ 16]* for further explanation.

4.2 Spectrum Optimisation

Based on the mapped B0 field TopShim performs an optimisation of a criterion for the quality of the spectral line [4]. Of course the aim is to obtain a narrow and smooth line without the presence of false peaks. Within this framework there is still some freedom to shape the line in order to emphasize particular properties, such as a narrow line width or hump, or good solvent suppression.

By using an additional parameter, **ws<n>,** for the TopShim command the lineshape may be influenced by focussing on the best full width at half height, or by reducing the base of the line. Reducing <n> gives the better half width whereas increasing **<n>** will reduce the width at the base of the line. The parameter files **ws0, ws5, ws10, ws15, ws20, ws30, ws40, ws50, ws60, ws70, ws80, ws90, ws100** are provided.

By default, **ws5** is used for 1D, 3D and HRMAS shimming.

> **i** When using **ws20** or higher the convergence criterion is changed somewhat as TopShim assumes a solvent suppression application.

> **i** **ws0** may induce instabilities in the shimming process and should be used with caution.

4.3 Tune (HR Liquids Only)

In some cases it is advantageous to support the standard TopShim gradient shimming methods by the integrated tune procedure. In fact, for non-spinning experiments it is always beneficial to apply the tune procedure, whenever the sample has been changed.

The **tune** procedure modifies shim values and searches for the maximum lock level. The assumption is that the higher lock signal corresponds to the better lineshape, which is often true, but not always (e.g. when a probe has a significant temperature gradient and the solvent signal responds differently to that than the signals of interest).

By default tune is switched off when performing 1D or 3D shimming.

The tune parameters can be added after any TopShim command, e.g.

topshim <various parameters> tunea<command>

The duration of the tune procedure depends on the number of shims and the response time of the system. It ranges from a few seconds to several minutes.

The tune procedure can also be used without gradient shimming by using the parameter **tune**, e.g.

topshim tune tunea<command>

Note that if the sample is spinning, only on-axis shims will be used by tune.

In the table shown below the sets of shims provided for tune, the respective com- mands, and a short description are summarized.

Set of shims	Command
Z	**tuneaz** remove possible residual Z gradient
Y	**tuneay** remove possible residual Y gradient
X	**tuneax** remove possible residual X gradientno specific purpose
Z-XY-XZ-YZ	**tunea** From experience this is a good balance between cost (time) and reward.
Z-X-Y	**tuneaxyz** remove gradients of rank 1
Z2-XZ-YZ-(X2-Y2)-XY	**tuneaxyz2** remove gradients of rank 2
Z3-XZ2-YZ2-(X2-Y2)Z-XYZ-X3-Y3	**tuneaxyz3** remove gradients of rank 3

4.4 Graphical User Interface (GUI)

You can open the GUI in two ways:

- from the TOPSPIN command line by typing
  topshim gui
- from the TOPSPIN menu with
  Spectrometer | Shim control | Automatic Shimming.

The GUI can be opened even if TopShim is currently running.

In the following the elements of the GUI shown in Figure *Figure 4.1 [▷ 14]* are shortly described. Each element is also supported by a tool tip.

4.4.1 The Shim Panel

The **Shim** panel gives access to the basic parameters, displays real-time information about the shimming process, and shimming can be started or aborted.

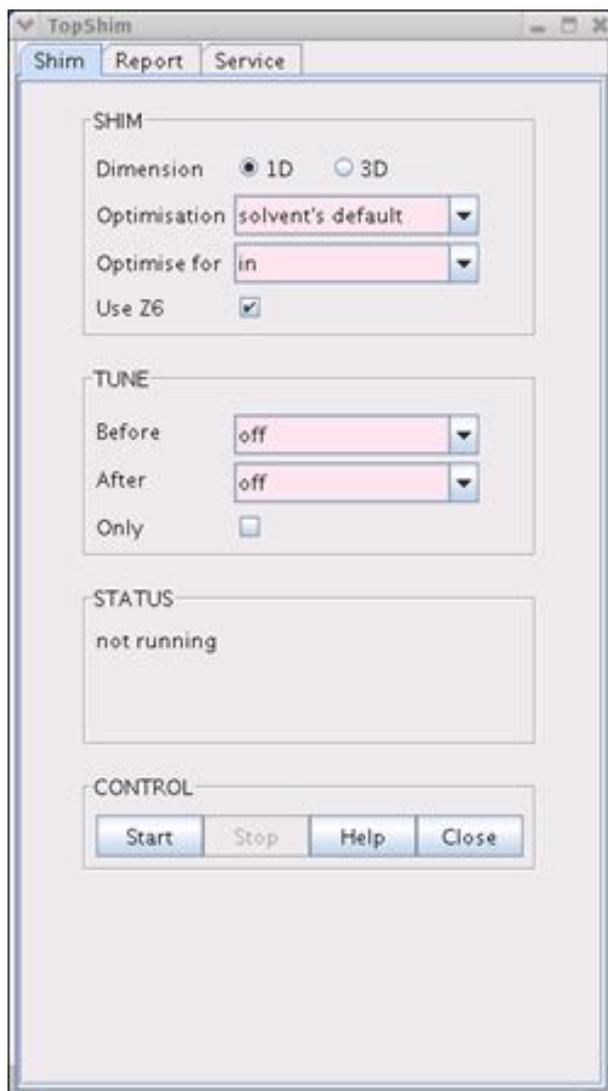In the **SHIM** box, parameters concerning gradient shimming are accessible:

Figure 4.1: The TopShim GUI.

The **Dimensio**n radio button selects between 1D or 3D shimming (see *4.1.2 [▷ 9]*).

The Optimisation pull-down menu gives access to the various types of spectrum optimisation (see *4.2 [▷ 12]*).

The parameter labelled with **Optimise for** should be set to the observe nucleus of the planned NMR experiment. From this nucleus the RF coil B1 profile is derived that is used for the spectrum optimization. Normally this is set automatically, according to the NMR dataset from which the GUI was started.

Use Z6 can be activated to enable changes of this shim.

The **TUNE** box enables application of the TopShim tune functionality Before or After gradient shimming (see *4.3 [▷ 10]*). With the Only box being selected, gradient shimming is skipped completely.

The box **PARAMETERS** is only accessible if it is enabled in the **PREFERENCES** in the **Service** panel (*4.4.3 [▷ 15]*). The text line serves to define additional parameters as described in *4.5.2 [▷ 16]*. Multiple parameters are separated by a space. Note that options (see *4.5.1 [▷ 16]*) cannot be entered here. The additional parameters will only be used if the check box is enabled. Thus you can leave frequently used parameters in the line and just disable them in case they are not required.

The **STATUS** lines inform the user if TopShim is currently running or not, display important parameters (e.g. the used solvent), show the task currently dealt with, and keep the user posted about acquisition and processing steps.

The **CONTROL** box provides four buttons with basic control functionality:

After pressing **Start** the shimming procedure is started immediately with the parameters set above. You can also start from the command line while the GUI is open. However, in this case TopShim is started with the parameters as set in the command line and the GUI is used only as a display tool.

By pressing **Stop** the shimming procedure is aborted immediately. The best shim values found so far will be set.

Clicking **Help** will display this manual.

The **Close** button closes the GUI. If TopShim is running, it will not be aborted.

4.4.2 The Report Panel

The **Report** panel collects the most important information about parameters and results of the current shimming run. Alternatively, the most recent report can always be displayed with **topshim report** or with the **Report** button in the **Service** panel.

4.4.3 The Service Panel

The **Service** panel serves to give direct access some of the options described in *4.5.1 [▶ 16]* and to setup the GUI.

The box **GENERAL** provides the buttons **About** to show the current TopShim version and **Help** to display this manual also from this panel.

The buttons in the box **GUI DEFAULTS** enable the user to define new default values for the parameters settings in the **Shim** panel (see *4.4.1 [▶ 9]*). With the button **Save** the current settings are stored as default values. They will be loaded when the GUI is opened. Alternatively, they can always be reloaded with the **Load** button. The button **Restore** enables to restore the factory default settings. By clicking this button the values are loaded into the GUI. In order to make them the default settings again, you have to click **Save** afterwards.

The box **SETUP** contains three buttons **Cf, Edhead**, and **Gradamp** for the

TopShim specific spectrometer setup (see *3.2 [▶ 7]*).

In the box **PROBE CALIBRATION** the **Info** button displays the calibration status for the current probe. With **Reset** these calibration values can be deleted. This is required if a) the calibration was performed with a bad 3D homogeneity (see *4.1.4 [▶ 10]*) or b) hardware components such as magnet, gradient system or shim system have been changed since the last calibration. After the reset shimming on a water sample must be performed to redo the calibration (see *4.1.5 [▶ 11]*).

In the box **DIAGNOSTICS** with the button **Report** the results of the last TopShim run can be displayed. Pressing the button **Logfile** shows the disk location of the current TopShim logfile. This is useful if problems occur during shimming and you want to report them to the Bruker service (see *5.2 [▶ 23]*).

Finally, in the box **PREFERENCES** the functionality of the GUI can be modified. Setting the check box **External** releases the GUI from the TOPSPIN main window. The check box **Additional parameters** controls if in the **Shim** panel the optional parameter line is available (see *4.4.1 [▶ 13]*). The chosen preferences will be remembered after closing the GUI.

4.5 Options and Parameters

Options and parameters serve to change the behaviour of TopShim in the desired manner. They can be added after **topshim** in the TOPSPIN command line.

4.5.1 Options

The following options do not start shimming, but execute other tasks such as e.g. performing configuration or providing information. Options must be added directly after the **topshim** command separated by a space:

topshim <option>

The following options are available:

gui	open graphical user interface
stop	abort shimming
	A parameter can be added to determine the shim values set after abortion:
	optimum | best setting found so far (default)
	current | current setting
	original |original setting
	Example: **topshim stop original**
help	display this manual
about	display the version information
report	display the report of the latest run of TopShim
logfile	display the location of the TopShim log-files
calinfo	display calibration information for the current probe (see *4.1.5 [▷ 11]*)
calreset	reset all calibration results for the current probe (see *4.1.5 [▷ 11]*)
edpar	create or modify parameter file (see *4.5.3 [▷ 19]*)
changemasshims	provides a basic GUI for setting shims manually in the MAS frame.

4.5.2 Parameters

Although not required in most cases, there are a number of parameters that can be set by the user to control the behavior of TopShim.

An arbitrary number of parameter expressions can be added after the **topshim** command in the command line, each of them separated by a space:

topshim <par1> <par2> …

The priority of the parameter expressions increases from left to right, i.e. the last expression has the highest priority. This hierarchy is required if a parameter occurs more than once.

There are two types of parameter expressions: parameter file names and explicit parameter settings. With parameter files one or multiple parameters can be set. An example is the parameter file **lockoff** that changes several parameters for the spectrum optimisation. In contrast explicit parameters serve to set the value of a single parameter. The syntax for setting a parameter explicitly is:

<parameter_name>=<parameter_value>

Note that there must not be spaces in such an expression. An example for performing Top-Shim for a flow application using both types of parameter expressions is:

topshim lockoff o1p=1.95 rga

Here, TopShim is executed with the lock switched off, the resonance frequency set to 1.95 ppm, and with receiver gain optimisation.

In the following table a number of available parameter expressions are listed.

1h	use ^{1}H field mapping
	requires strong 1H signal, e.g. in a water sample default: defined by solvent.
2h	use ^{2}H field mapping requires a deuterated solvent
	default: defined by solvent
19f	use ^{19}F field mapping
coil=	sets the NMR nucleus
	NMR nucleus = observe nucleus of the planned NMR experiment that defines the RF B1 coil profile used in the spectrum optimisation (see *4.2* *[▶ 12]*)
	possible values: all nuclei available on the probe;
	default: observe nucleus of the active data set; if no data set is active, the inner coil is used
	example: **coil=13c**
ws<n>	Influence the lineshape by focussing on the best full width at half height, or by reducing the base of the line. Reducing **<n>** gives the better half width whereas increasing **<n>** will reduce the width at the base of the line. The parameter files **ws0, ws5, ws10, ws15, ws20, ws30, ws40, ws50, ws60, ws70, ws80, ws90, ws100** are provided.
	By default **ws5** is used for 1D, 3D and hrmas shimming.
	NOTE: When using **ws20** or higher the convergence criterion for TopShim assumes a solvent suppression application
	NOTE: **ws0** may induce instabilities in the shimming process and should be used with caution.

ordmax=0	set maximum total order of on-axis shim functions
	By default the highest on-axis order is limited by the shim system (e.g. 6 for BOSS1-SB and BOSS2-SB, and 8 for BOSS3-SB), the coil length and the magnetic field strength of the NMR system.
	Reducing the maximum from 8 to 5 may, for example, reduce the time needed by TopShim and limit changes in the field offset (due to Z6 and Z8). Naturally, limiting the maximum order may result in broader lineshapes.
	Examples:
	ordmax=3 | use Z1 … Z3 for shimming default**: ordmax=0**, Topshim selects maximum shim order

Table 4.1: General

cal	force the execution of all calibration procedures for the current probe; a water sample must be used
	executed as part of **initial**
phcal	force probe position calibration
	May be used after reinserting the probe.
	executed as part of **initial**

Table 4.2: Calibration

lockoff	enable shimming while the lock is switched off
	The user must ensure that the excitation frequency is on resonance. Hence the lock field value must be set on resonance and/or the resonance frequency o1p must be set manually (see below).
	Furthermore the solvent must be set correctly, or the shim nucleus must be defined using **1h, 2h or 19f..** By default the solvent is determined according to the last **lock** command performed. Alternatively, it can be set using the **solvent** parameter (see below).
o1p=	set the excitation frequency o1p [ppm] manually
	Example: **o1p=1.95**
	Default: depending on solvent, usually BSMS lock shift
o1pget	read the excitation frequency o1p [ppm] from the currently active data set
	Performing the shimming acquisition on a desired peak can be accomplished by 1) acquisition of a spectrum, 2) setting o1p in the acuired spectrum to the desired peak using **.cal**, and 3) by starting shimming with **topshim o1pget**.
solvent=	set the solvent
	Example: **solvent=CH3CN+D2O**
	Default: auto, i.e. determined from last **lock** command
rga	force receiver gain optimisation before shimming
autogain	perform autogain for the lock after shimming
	By default autogain is executed only before the tune procedure.
selwid=	set width of selective excitation [ppm] and switch it on
	Example: **selwid=0.5**
	Default: depending on solvent, or 1 ppm

shigemi	use B1-profile to detect shim region and restricts to higher relative values. This is particular useful for Shigemi tubes but can be used for any sample.
astm	perform ^{1}H shimming on ASTM sample Used for shimming of 10 mm probes. See *4.7.2 [▷ 21]*.

Table 4.3: Various

convcomp	modifies the TopShim experiment to make it more robust against effects of convection.
tcorr=x,y	allows setting an offset for Z and Z2, respectively, that may be useful in cases where a probe has a significant temperature gradient and the solvent responds differently to the temperature gradient than the signals of interest.

Table 4.4: Temperature Effects

map	acquire field map without shimming Serves for diagnostics. Can be performed for 1D and 3D. The raw, magnitude and field map data is stored in data sets for the user **topshimData**.
plot	create graphical output during shimming Serves for diagnostics. The intermediate and final raw, magnitude and field map data is stored in data sets for the user **topshimData**. Should not be used routinely as large amounts of data are created, in particular for 3D.

Table 4.5: Diagnostics

4.5.3   Creating Parameter Files

i TopShim allows modifying a large set of parameters, which can lead to many unwanted results. Therefore, parameter files should be created only by experienced users.

System parameter files are located in:

`$TOPSPINHOME/conf/instr/topshim/parameters`

and they should not be modified. Modified files in this directory may be deleted or overwritten during update procedures.

If a certain setting of one or multiple parameters should be used repeatedly it can be stored in a new parameter file. New parameter files should be stored in the subdirectory user. The general procedure for creating a new parameter file should be:

1. Open an available parameter file, e.g. `default.par`, that contains all possible parameters.
2. Save the file with a new name `myfile.par` in the subdirectory user.
3. Delete all lines with parameters that should not be modified.
4. Make the desired changes to parameter values.
5. Save and exit.

In principle, any text editor can be used. However, TopShim offers the option

topshim edpar that leads though the described procedure.

The new parameter values can be used by adding the parameter file name without the extension in the command line, i.e. **topshim myfile**.

4.6 Solvents

Knowledge of the solvent is the most important source of information for TopShim. Therefore, it is essential to lock correctly before shimming. The following parameters are derived from the solvent.

- nucleus used for field mapping (^{1}H or ^{2}H)
- optimum 2H flip angle
- use of selective excitation
- receiver gain
- type of spectrum optimisation (see *4.2 [▷ 12]*)

For most solvents, the shim nucleus is ^{2}H and spectrum optimisation is performed for lineshape with narrow width. Only for the protonated solvents H2O+D2O, CH3CH+D2O, and CH3OH+D2O, the nucleus is 1H and the lineshape optimisation aims at solvent suppression.

The optimum flip angle is determined from the pulse repetition time of the field mapping sequence and from calibration data specific for each solvent (see 4.6.1).

Selective excitation is used in cases where multiple lines of comparable size are expected. An example is MeOD.

The receiver gain is calculated based on the expected relative signal strength.

The required information is available for all solvents found in the default solvents table that can be accessed using **edsolv.**

4.6.1 Solvent Setup

To allow optimum TopShim performance specific solvent properties have to be known. These have been pre-defined for all standard solvents (those named in the default Bruker solvent table), but to use other solvent names with TopShim they must first be configured. In all cases the new solvent name must first have been created with **edsolv**, and a suitable entry made in the lock table with **edlock**.

The command for creating the TopShim solvent settings is

topshim solvcal

The associated procedure has two parts, A) definition the solvent properties and B) the flip angle calibration (^{2}H only).

Part A only

In the following cases it is sufficient to execute part A only:

- ^{1}H is used for field mapping (part B be will automatically be skipped). Exam- ple: H2O+D2O
- Concerning shimming the new solvent has the same properties as an existing one. Example: New solvent Urine+D2O, copy from H2O+D2O.
- Only minor changes have to be applied with respect to an existing solvent. Example: New solvent: H2O+EtOH+D2O, copy from H2O+D2O and change to selective excitation.

There are two ways to start the setup procedure for part A only. Either you lock on the new solvent and then execute **topshim solvcal**, or you tell TopShim the solvent name in the command line with

topshim solvcal solvent=<newsolventname>

(where **<newsolventname>** is the name of your solvent exactly as entered in

edlock). Then you enter the requested information. In case of ^{2}H shimming the setup procedure must be cancelled before the calibration.

Part A and B

For a novel solvent used for ^{2}H shimming and with unknown properties the fol- lowing procedure applies:

1. The 3D homogeneity should be already good.

2. Insert a sample with the new solvent.

3. Lock for the previously defined solvent.

4. Perform basic shimming, either manually or using a tune.

5. Start the solvent setup using **topshim solvcal**.

   a) Initially select a solvent that is closest in character to your new solvent, and answer other questions.

   b) Start the flip angle calibration test (note this can take between 10 and 60 minutes to complete).

4.6.2 Solvent-Specific Pulses

TopShim calculates pulses depending on the 90° hard pulse given in the **edprosol** tables. These pulses must be correct to obtain the optimum efficiency of the shimming procedure. Therefore it is important to provide solvent-specific pulses in cases where considerable differences are expected for the same nucleus.

For example this may be necessary for salty water samples with high conductivity. In such a case a new solvent must be defined and the pulse must be determined and entered in the **edprosol** table for this specific solvent.

4.7 Specific Probes and Tubes

4.7.1 10 mm Probes

Samples with a large diameter are prone to convection in the sample which may hamper the gradient shimming procedure. Therefore, it is advantageous to perform 3D shimming on a sample which has a higher viscosity than H_2O. A very good candidate is the Bruker standard sample ASTM constisting of 40% p-dioxane in 60% C6D6. With this sample field mapping is performed on the ^{1}H peak at 3.475 ppm which is achieved with the following command:

topshim 3d 1h o1p=3.475

Alternatively, the already provided parameter file can be used

topshim 3d astm

See also *4.5.2 [▷ 16]* for a detailed description of the parameters.

4.7.2 Shigemi Tubes

For shimming Shigemi tubes please follow these rules:

- The filled tube range should not be less than about 7 mm.
- Centre the filled tube range well with the probe centre using the sample gauge.
- For 1D shimming add the parameter **shigemi** to the TopShim command, which will eliminate unreliable data at the axial tube walls.

4.8   Automation

TopShim is ready for easy shimming in automated NMR. The command line interface is suitable for this purpose without modifications. The basic command with or without parameters can be used with tools such as macros or AU programmes.

In ICON-NMR the default 1D TopShim method is already provided by the shim command **TOPSHIM**. Additional new methods can be defined by adding parameters to this command

TOPSHIM <par1> <par2> ... e.g.

TOPSHIM 3d tunea

or by describing an new shim command with

CPRW topshim <par1> <par2> ... e.g.

CPRW topshim 3d tunea

Alternatively, user-defined macros or AU programmes can be used, e.g.

CPRW my_topshim_macro XAU my_topshim_au

5 Various

5.1 Support

In case of problems please first check for known bugs and possible fixes on the Bruker knowledge base

To report problems please email

making sure to describe the problem as fully as possible and including the TopShim logfile

```
topshim<date>.log
```

as well as the TOPSPIN history file history

both located in

```
$TOPSPINHOME/prog/curdir/<user>
```

5.2 References

The following references are the original publications about gradient shimming for high resolution liquid NMR and about the spectrum optimisation method.

1. M.G. Prammer, J.C. Haselgrove, M. Shinnar, and J.S. Leigh. A new approach to automatic shimming, J. Magn. Reson. 77 (1988) 40-52

2. P.C.M. Van Zijl, S. Sukumar, M. Johnson, P. Webb, R.E. Hurd, Optimized shimming for high-resolution NMR using three-dimensional image-based field mapping, J. Magn. Reson. A 111 (1994) 203-207

3. S. Sukumar, M.O. Johnson, R.E. Hurd, P.C.M. Van Zijl, Automated shimming for deuterated solvents using field profiling, J. Magn. Reson. 125 (1997) 159- 162

4. M. Weiger, T. Speck, M. Fey, Gradient shimming with spectrum optimisation, J. Magn. Reson. 182 (2006) 38-48

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