

China Users' Meeting – 2019

Tips and Troubleshooting for NMR Experiments



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Service & Lifecycle Support (SLS)
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Innovation with Integrity

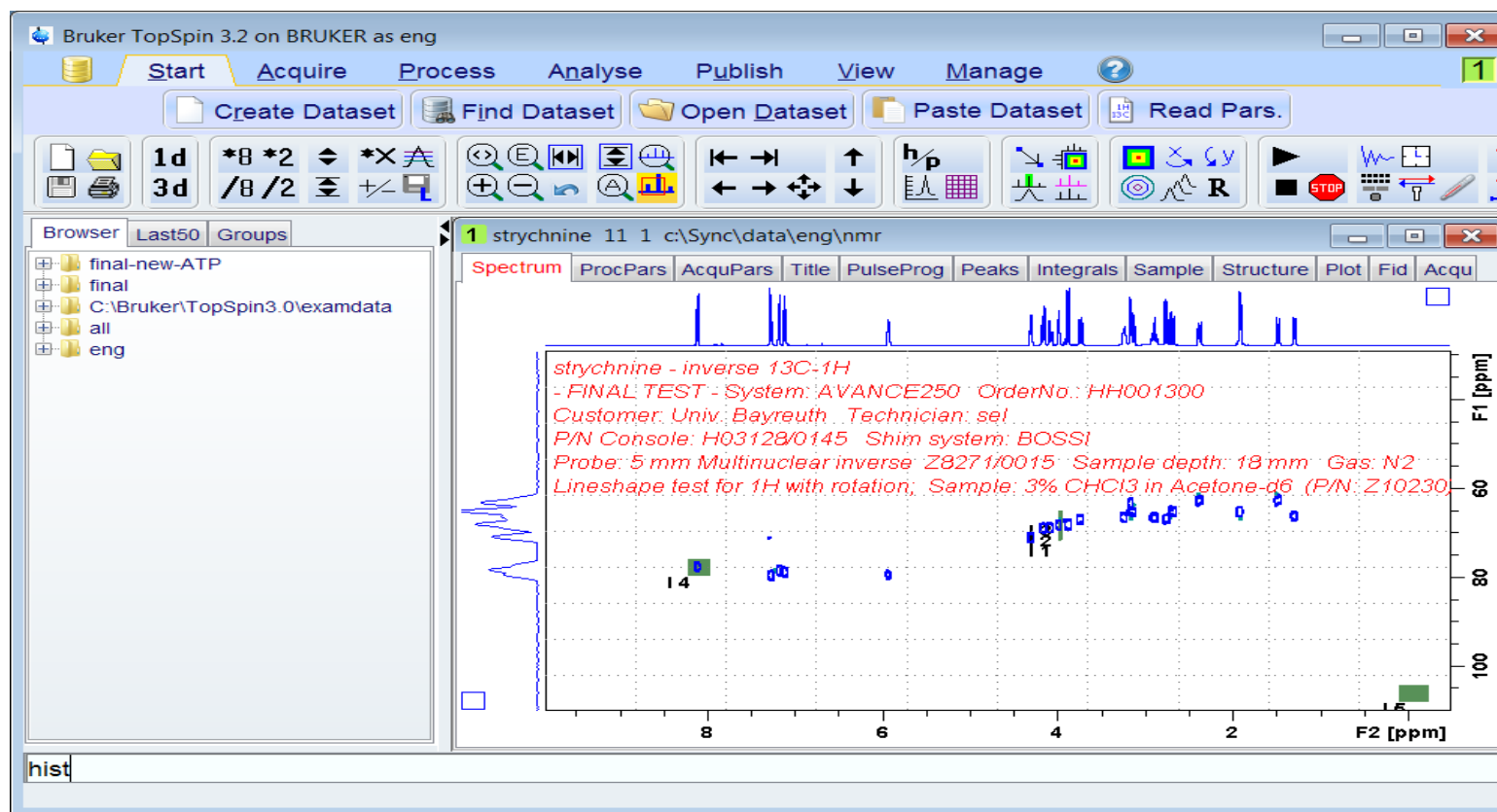
General hints



A few hints on how to get more information from the system:

- Read the error messages appearing in TopSpin carefully, often they help in tracking down the problem.
- Enter '**hist**' in TopSpin and check the latest error messages.
- Is it a problem which is always there or only from time to time?
- Can you reproduce the problem? E.g. the problem occurs only in a certain data set.
- Create a backup of the history files right after the problem occurred with the command '**savelogs**'. That file can be sent to Bruker if required:
 - Enter 'savelogs' in TopSpin
 - As Support Token, enter your company name and your order number, e.g. 10234567_BrukerCH

If you feel like it, have a look at the history file ...

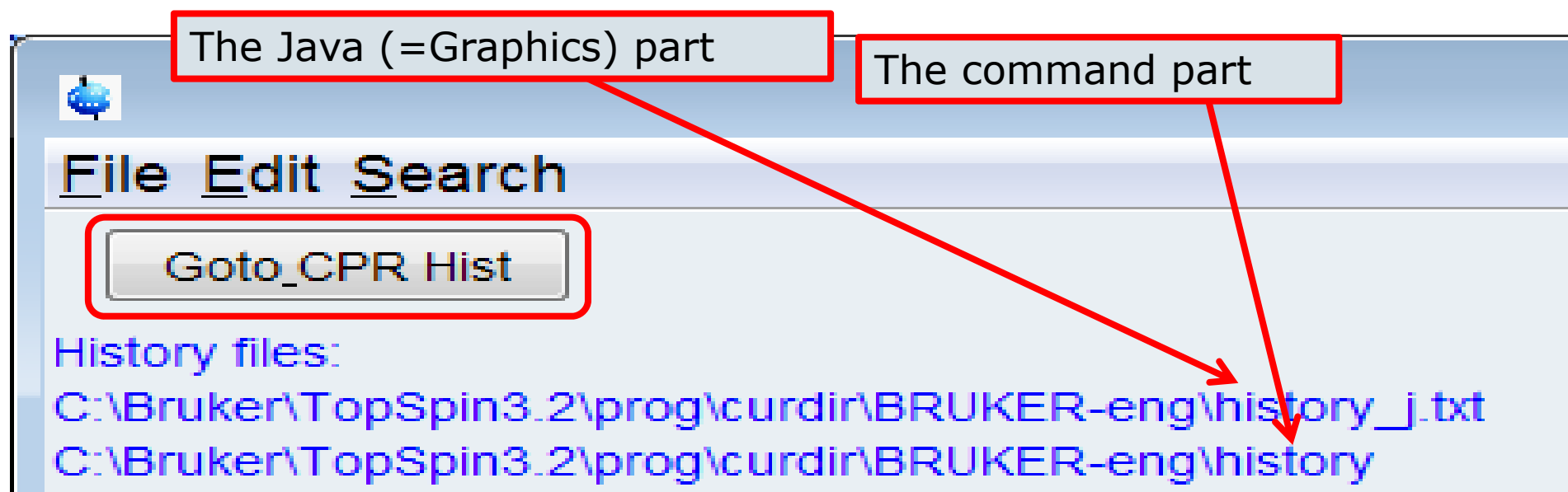


There are two parts to the history file ...

A screenshot of a text editor window titled "File Edit Search". It contains a "Goto_CPR Hist" button and a list of "History files" with two entries: "C:\Bruker\TopSpin3.2\prog\curdir\BRUKER-eng\history_j.txt" and "C:\Bruker\TopSpin3.2\prog\curdir\BRUKER-eng\history". The main text area shows a log file with a date separator "2013-10-19" followed by a series of timestamped messages. The messages describe the startup of the graphical user interface, system information (Windows, 32-bit JVM), user properties, connection details (host=127.0.0.1, port=5500), session key import, GUI session creation, and the initialization of the CPMR listener. It details the successful activation of the CPMR listener server, contact configuration services, and the login of a CPMR client. The log ends with a message about the inability to get last used acquisition data as no acquisition is running.

```
1
2 2013-10-19
3 -----
4 19-02:58:00.086 Graphical User Interface started.
5 19-02:58:00.101 Operating system = WINDOWS, JVM type (bit) = 32
6
7 19-02:58:00.101 User properties directory=C:\Users\eng.BRUKER\.topspin-BILBY\prop
8 19-02:58:00.663 To connect to this TOPSPIN instance via Remote Connection use host=127.0.0.1, port=5500
9 19-02:58:00.663 Start: opening file for local session key at 'C:\Bruker\TopSpin3.2\prog\curdir\BRUKER-eng\keystore/localSes:
10 19-02:58:00.663 Start: local session key imported successfully.
11 19-02:58:00.663 Start: GUI session key created
12 19-02:58:01.100 GUI Version = 3.2 / 1791
13 19-02:58:01.271 cmd=create_drag_transfer_handler
14 19-02:58:01.568 cmd=init_cpr
15 19-02:58:01.568 Start ORB: recommended port=5090.
16 19-02:58:02.379 Start ORB: successful on port 5090
17 19-02:58:02.379 cmd=cprlistener
18 19-02:58:02.379 Start CprListenerServer: activate.
19 19-02:58:02.441 Start CprListenerServer: activation successful, id = corbaloc:iiop:127.0.0.1:5090/%0000%00%FEPOA%FEctrlPOA%
20 19-02:58:02.582 Contact configuration service: URL=corbaloc:iiop:127.0.0.1:5500/ConfigurationServer, IOR=IOR:01000000240000:
21 19-02:58:02.582 Contact configuration service: local contact, using URL: corbaloc:iiop:127.0.0.1:5500/ConfigurationServer
22 19-02:58:02.628 Contact configuration service: successful.
23 19-02:58:02.628 Contact configuration service: local session, authentication done.
24 19-02:58:02.675 LOCKCONF, SamChgEth[0-3]: [DLMT, no, no, no, no]
25 19-02:58:02.691 Datastation: false, Fourier: false, ELCB: true, EVTU: true, Fourier-BSMS: false,
26 MAS2: false, VTU: false, RCU: false, Multi-receiver: false, number of receivers: 1,
27 Multiplex lock: true, NMR thermometer: true, 19F lock: false, SampleXpress over Ethernet: false
28 19-02:58:02.691 Trying to start hardware access servers...
29 19-02:58:02.847 Contact CPR: local contact, narrowing object for: corbaloc:iiop:127.0.0.1:5500/CPMRmsg, IOR=IOR:010000001d00:
30 19-02:58:02.894 Contact CPR: object is valid.
31 19-02:58:02.894 Contact CPR: address of CprListener is corbaloc:iiop:127.0.0.1:5090/%0000%00%FEPOA%FEctrlPOA%FECPClient
32 19-02:58:02.894 Contact CPR: login at CPR with "corbaloc:iiop:127.0.0.1:5090/%0000%00%FEPOA%FEctrlPOA%FECPClient"
33 19-02:58:02.906 Unable to get last used acquisition data: No acquisition running
```

There are two parts to the history file ...





A real life example:

```
C:\Sync\powerp\TopSpin-Training\Service-Training-BII-June-2008\pyc_history_250108.txt - metapad
File Edit Favourites Options Help
[Icons]
2008-01-25
TIME +0200 PID OBJ CMD TYPE COMMAND
18:54:34.293 15137 TOPSPIN Version 2.1
/opt/topspin/prog/mod/cpr on ext3
version 2007-10-17 07:11:26Z build 2007-10-23 04:26:46
gcc 3.4.6 with glibc 2.3 compiled tag topspin_2_1
on AV600 (Linux 2.6.9-67.0.1.ELsmp glibc 2.3.4 1 System
with Message Queue Id 98304
started in /home/pyc
by pyc with home directory /home/pyc
18:54:34.293 FLEXlm license is valid until 2022-03-13
18:54:34.294 15137 0 cmd enter: cprserver
18:54:34.319 15149 proc start: cprserver (module cprserver)
18:54:34.423 15149 0 cmd sent: cprserver
18:54:34.454 15137 0 cmd enter: cprclient /tmp/msg.cprclient.15137
18:54:34.466 15154 proc start: cprclient (module cprclient)
18:54:34.524 15157 java virtual machine
/opt/topspin/jre/bin/java
started with arguments:
-DJAVA_JVM_PARENTID=15137 -splash:/opt/topspin/classes/p
18:54:34.524 15154 0 cmd sent: cprclient /tmp/msg.cprclient.15137
18:54:36.920 15149 0 cmd enter: dataserver id 0
DOS INS Line: 1/1361 Col: 1
```


A real life example:



```
client changed object to "/opt/topspin/data/pyc/nmr/pyc_AI_005/89/pdat
10 new object "/opt/topspin/data/pyc/nmr/pyc_AI_005/89/pdata/1"
10 cmd enter: rpar "dipsi_tbi.pyc" all
   proc start: rpar (module edparproc)
10 cmd sent: rpar "dipsi_tbi.pyc" all
   error msg: rpar
dipsi_tbi.pyc: File not found
   cmd term: rpar; status=-1
   proc term
```

New object indicates a new dataset. Here, the new dataset gets the number "10". In all following lines commands that show a "10" in the front refer to and work on this dataset. Like the **rpar** command in this example.

A real life example:



pyc_history_250108.txt - Notepad

File Edit Format View Help

```
cmd enter: int m atmaserver halt
cmd enter: int m topshi
cmd term: halt; status=0
10 cmd enter: checklocksh
10 cmd(o) sent: checklocksh
cmd enter: sendgui clientid=all
cmd enter: sendgui clientid=all
cmd term: zg; status=0
proc term
cmd term: checklocksh
2 cmd enter: zg yes
proc start: zg (module g
2 cmd sent: new yes
error msg: zg
Error during BirdRack flush: Error in flush: AxisException 12 during
getPossibleRoutings: HTTPTransportException:
Client failed to open Failed to open connection to server:
hostname='149.236.99.250'
port='10000'
Error Message='No route to host'
cmd enter: sendgui clientid=all acqstop end POWCHK_ON
cmd term: zg; status=-1
```

An acquisition is "halt"ed successfully and the "zg" finishes with no problem (indicated by the "status=0" message).

The next acquisition fails with a network problem "no route to host" !!

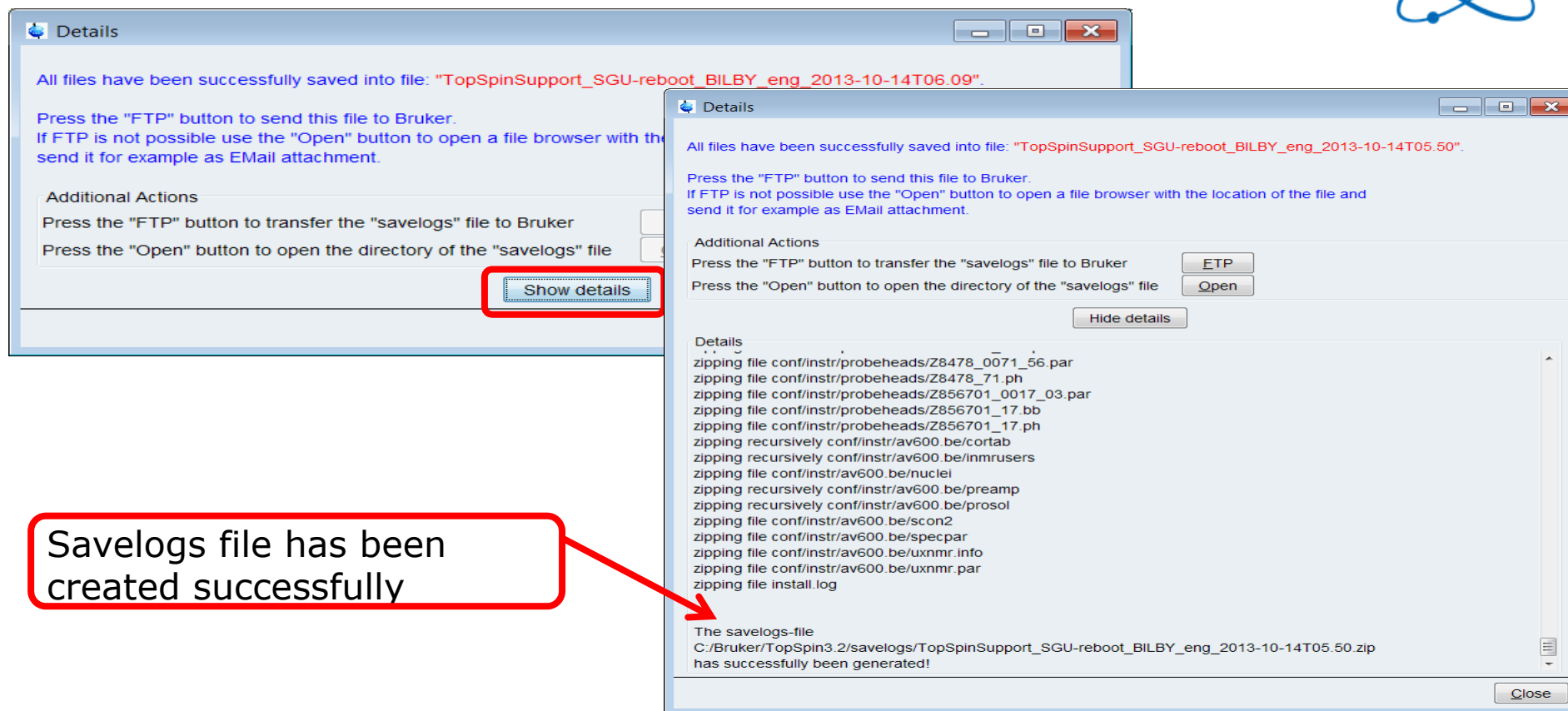
And the solution was:



Firmware update on the BLAXH amplifier !!

The amplifier occasionally lost its Ethernet connectivity due to a race conflict in the firmware.

savelogs details



Details

All files have been successfully saved into file: "TopSpinSupport_SGU-reboot_BILBY_eng_2013-10-14T06.09".

Press the "FTP" button to send this file to Bruker.
If FTP is not possible use the "Open" button to open a file browser with the location of the file and send it for example as EMail attachment.

Additional Actions

Press the "FTP" button to transfer the "savelogs" file to Bruker

Press the "Open" button to open the directory of the "savelogs" file

Show details

Details

All files have been successfully saved into file: "TopSpinSupport_SGU-reboot_BILBY_eng_2013-10-14T05.50".

Press the "FTP" button to send this file to Bruker.
If FTP is not possible use the "Open" button to open a file browser with the location of the file and send it for example as EMail attachment.

Additional Actions

Press the "FTP" button to transfer the "savelogs" file to Bruker

Press the "Open" button to open the directory of the "savelogs" file

ETP

Open

Hide details

Details

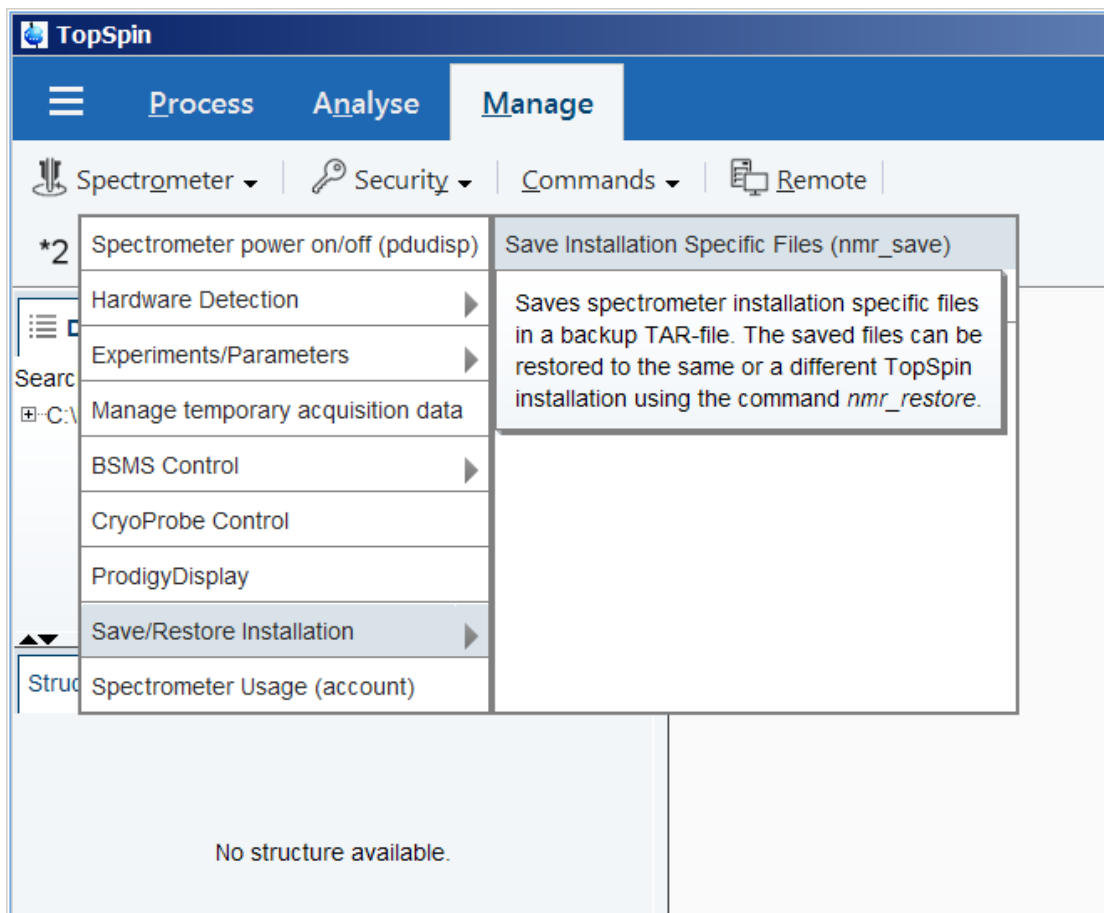
- zipping file conf/instr/probeheads/Z8478_0071_56.par
- zipping file conf/instr/probeheads/Z8478_71.ph
- zipping file conf/instr/probeheads/Z856701_0017_03.par
- zipping file conf/instr/probeheads/Z856701_17.bb
- zipping file conf/instr/probeheads/Z856701_17.ph
- zipping recursively conf/instr/av600.be/cortab
- zipping recursively conf/instr/av600.be/inmrusers
- zipping file conf/instr/av600.be/nuclei
- zipping recursively conf/instr/av600.be/preamp
- zipping recursively conf/instr/av600.be/prosol
- zipping file conf/instr/av600.be/scon2
- zipping file conf/instr/av600.be/specpar
- zipping file conf/instr/av600.be/uxnmr.info
- zipping file conf/instr/av600.be/uxnmr.par
- zipping file install.log

The savelogs-file
C:/Bruker/TopSpin3.2/savelogs/TopSpinSupport_SGU-reboot_BILBY_eng_2013-10-14T05.50.zip
has successfully been generated!

Close

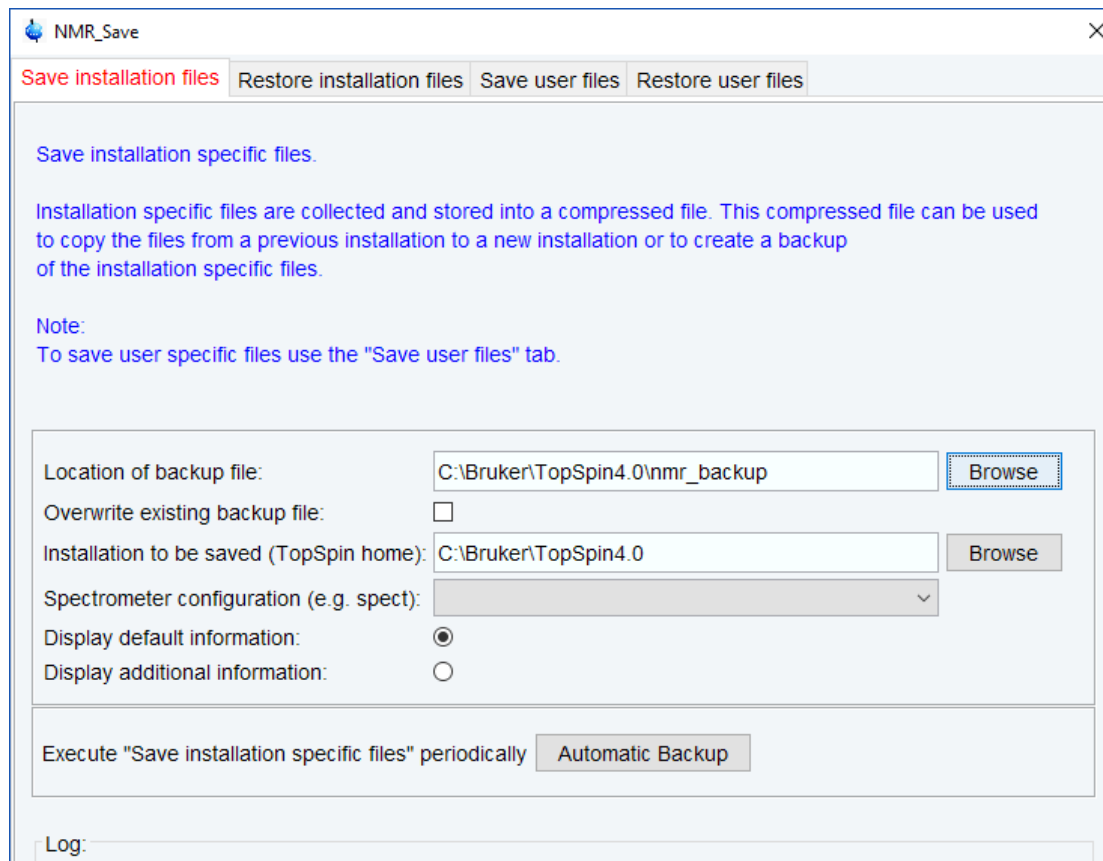
Savelogs file has been created successfully

nmrsave



**nmrsave:
VERY IMPORTANT !!!**

nmrsave

A screenshot of the "NMR_Save" dialog box. The window has a title bar with a close button. Below the title bar are four tabs: "Save installation files" (highlighted in red), "Restore installation files", "Save user files", and "Restore user files". The main area contains instructions: "Save installation specific files." followed by a paragraph explaining that installation files are collected into a compressed file for backup or migration. A note states: "Note: To save user specific files use the 'Save user files' tab." Below this is a form with several fields: "Location of backup file:" with a text box containing "C:\Bruker\TopSpin4.0\nmr_backup" and a "Browse" button; "Overwrite existing backup file:" with an unchecked checkbox; "Installation to be saved (TopSpin home):" with a text box containing "C:\Bruker\TopSpin4.0" and a "Browse" button; "Spectrometer configuration (e.g. spect):" with a dropdown menu; "Display default information:" with a selected radio button; and "Display additional information:" with an unselected radio button. At the bottom, there is a checkbox for "Execute 'Save installation specific files' periodically" and a button labeled "Automatic Backup". A "Log:" label is at the very bottom left.

nmrsave:
Archives AVANCE NEO
Configuration !!!

General hints



- Is it a hardware failure?
Check the power supply voltage LEDs in the console.
- SampleChanger is not running?
Check the input pressure from the SampleChanger.
- Do other systems in the lab also have problems?
It could be a power issue.

Tracking the problem



Here are some questions you could ask yourself

- Is the magnet on field?
- Is there ice on the magnet?
- Can we communicate with the spectrometer?
- Does tuning and matching work ('wobb', 'atma')?
- Is there a lock? Does it look normal?
- Can you run TopShim? (on 1H and 2H)
- Do we get a spectrum? On more than one channel?

Is the magnet on field?



If there is neither a lock nor a spectrum can be acquired, the magnet could have quenched or the main switch could have opened and the magnet either lost field or is still discharging.

- Try feeling the magnetic field by using a magnetic tool.
Be careful!
- If there is ice on top of the magnet, or the magnet is wet, this could indicate a severe problem! Contact Bruker immediately!

Can you communicate with the hardware?



- Try to run 'ii' and 'ii restart'
- Use 'ha' to check which units are available in the spectrometer network

What could help?

- Restart TopSpin
- Reboot the spectrometer
- Reboot the workstation
- Disable the firewall on the workstation

Can you acquire a spectrum?



- If you cannot acquire a spectrum, try a different nucleus, usually ^{13}C .
- A different nucleus requires probably a different channel (SGU/TRX) and a different HPPR slice.
- A cable might not be connected or a frequency might not be produced by the REF unit.
- You can also try to use a different SGU, if your spectrometer is SGU based (change routing in 'edasp').

Can you run 'wobb' / 'atma'?

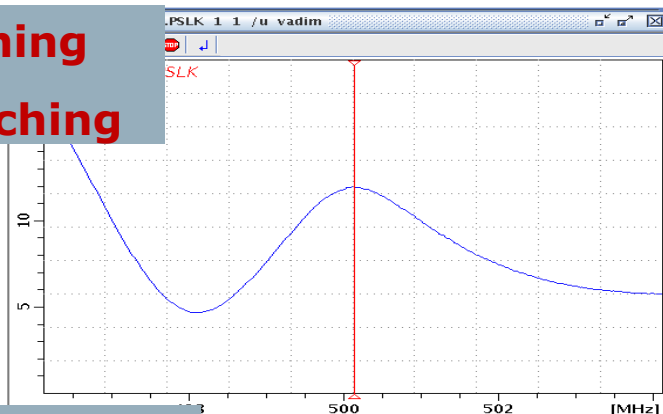


- The 'wobb' routine uses many components of the spectrometer and is therefore generally a good test. If it runs fine, it means that the receiver as well as parts of the frequency generation works fine.
- If 'wobb' does not run, try a different nucleus.
- Do 'atma' or 'atmm', does it work?



Tuning/matching and wobble curve

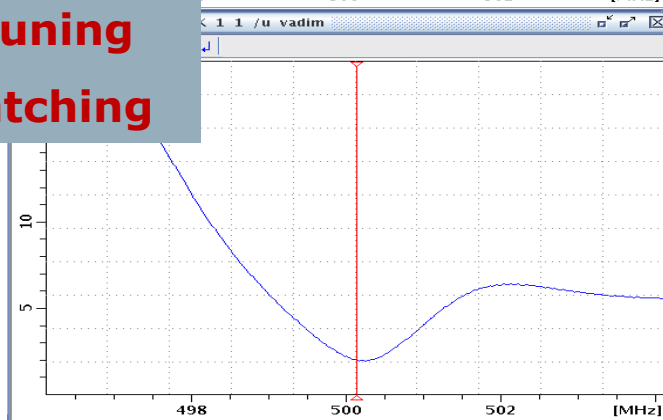
**bad tuning
bad matching**



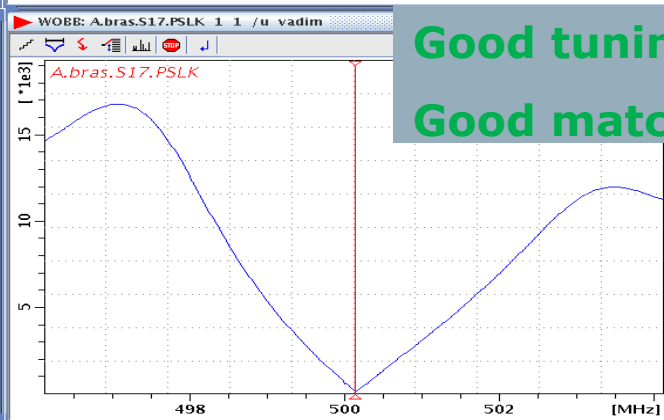
**bad tuning
good matching**



**good tuning
bad matching**

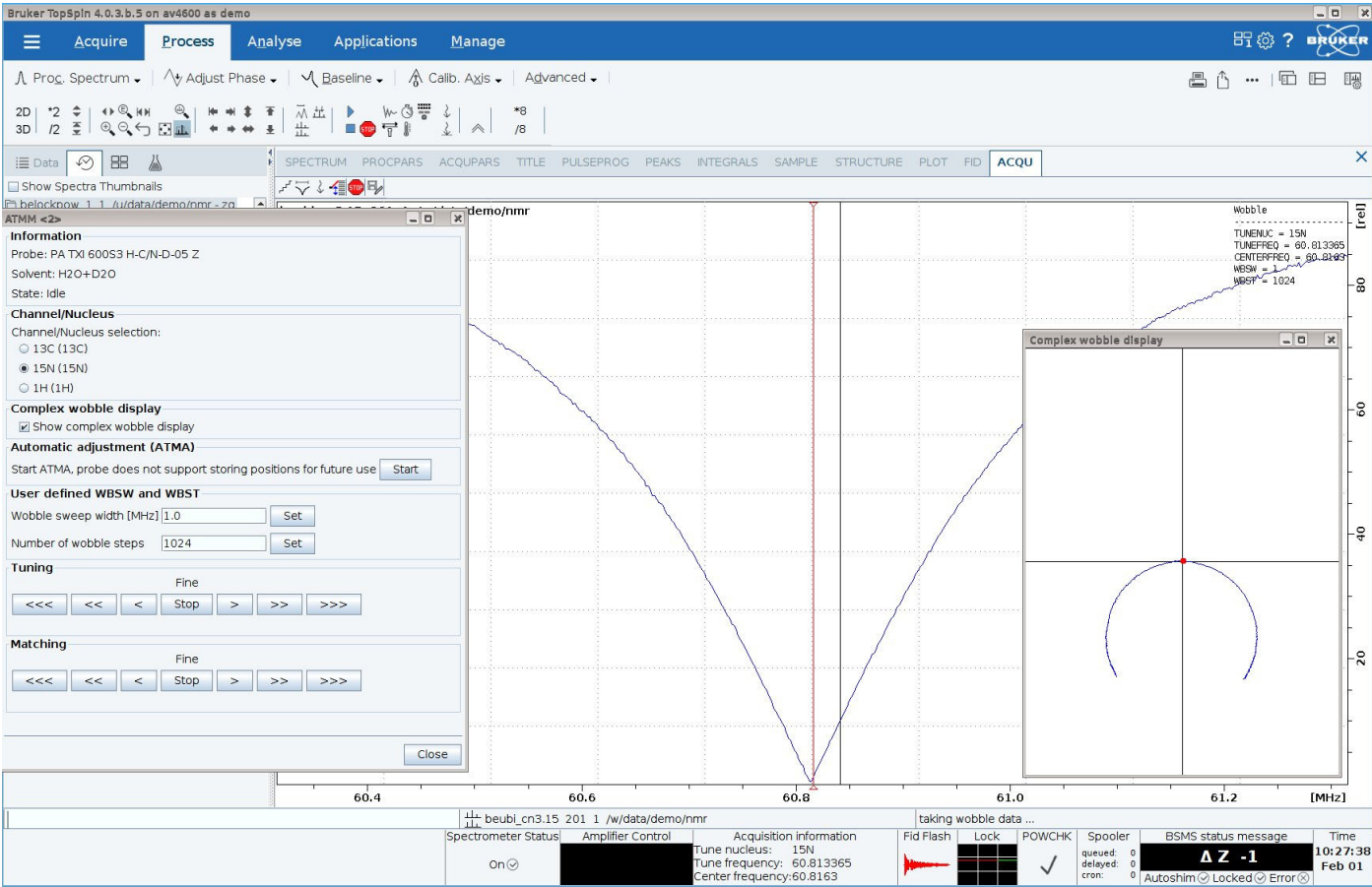


**Good tuning,
Good matching**





Tuning/matching and wobble curve



Does the lock signal look fine?

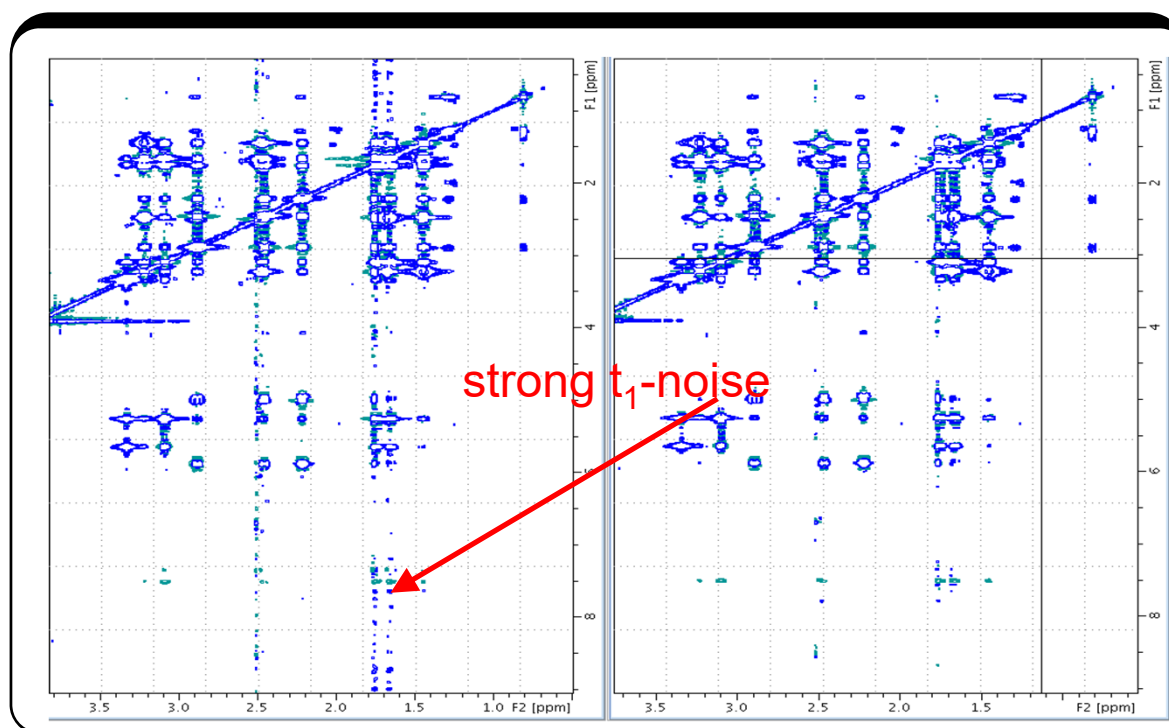


- Check the LOCK signal carefully in the swept mode and check whether it is locking-in normally for the correct solvent and whether the lock is stable.
- It is not locking?
 - Is a sample in the magnet?
 - Is the sample depth ok?
 - Use a Bruker test sample, e.g. the Lineshape sample or the Sucrose sample for your tests.
 - Create a new data set, read a standard parameter set, e.g. 'rpar PROTON', do 'ii' and try to lock again, e.g. 'lock acetone'.
 - Is the 2H tuning ok?
Can be checked with: 'rpar gradshim1d2h' – 'wobb'

TOCSY without gradients: t_1 -noise



- TOCSY:
AUTOSHIM during
acquisition can
influence t_1 -noise
Especially while
measuring salty
aqueous Samples

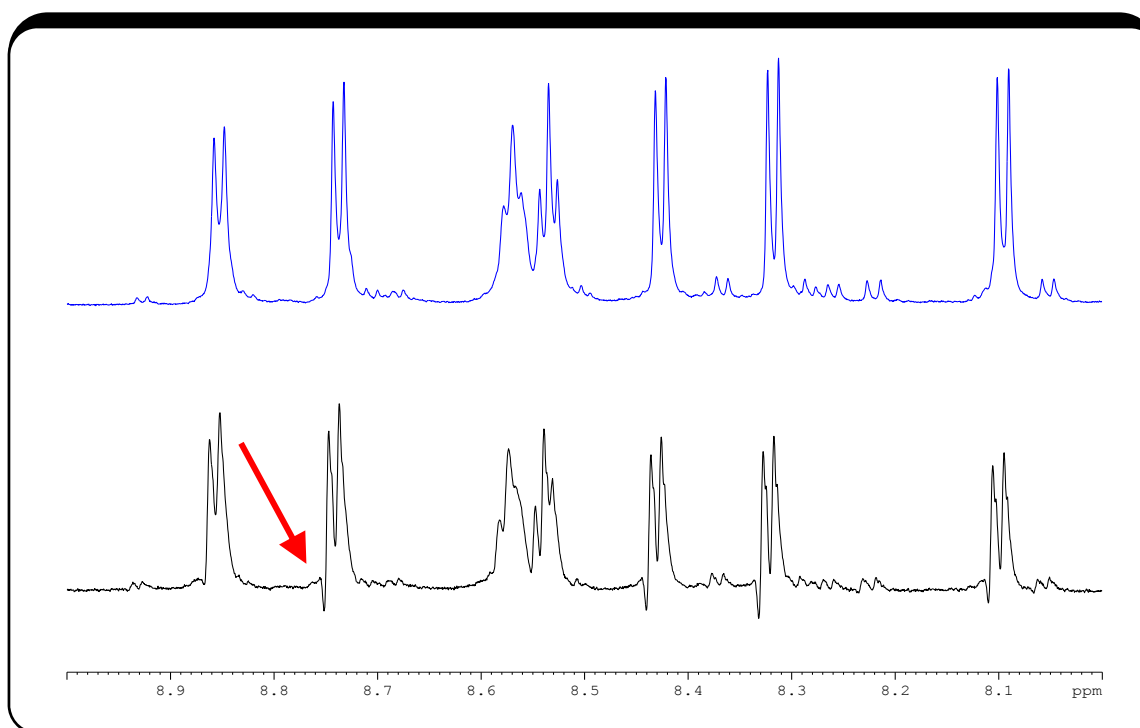


WATERGATE experiment: gradients



- WATERGATE:
Lock phase wrong
approximately 30°

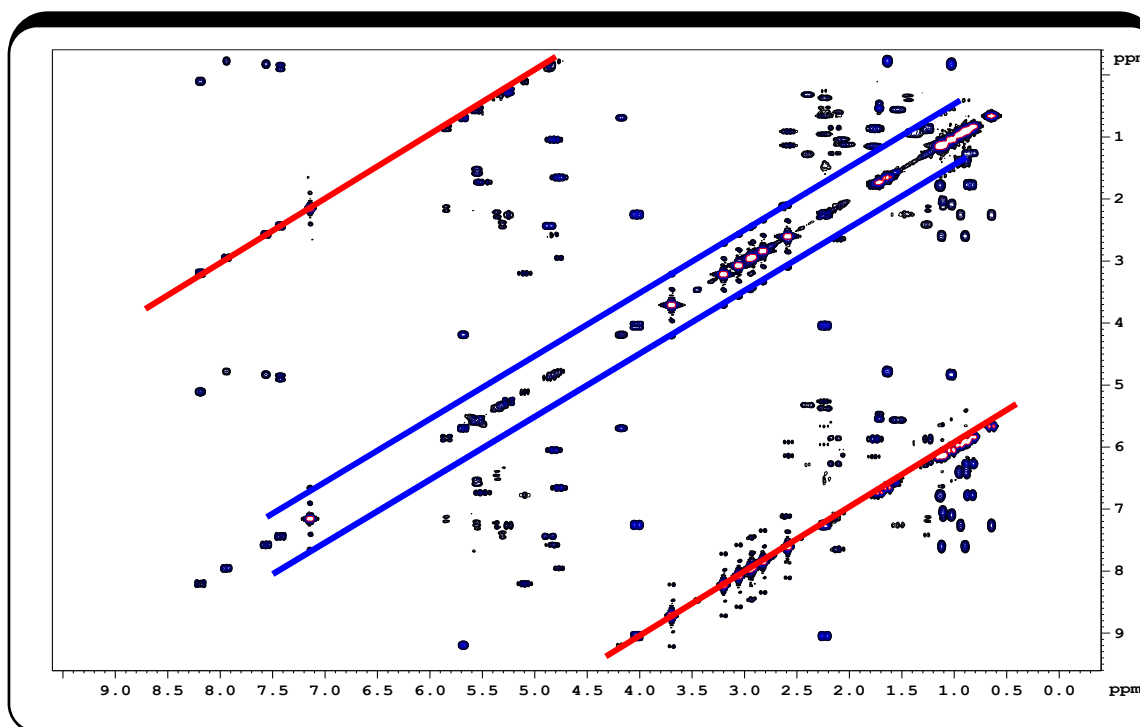
Depending on the
sign of the error
the artifacts
appear on the **right**
or **left** side of the
signal



COSY with gradients



- COSY with gradients:
Lock phase wrong
by approximately
40°



Also check the other lock parameters ...



- The “Loop Gain”, “Loop Phase” and “Loop filter” values are the control parameters relative to the lock current regulation loop. These values have an important influence on time average lock level stability. Their accurate setting is important when recording 2D homo-nuclear or hetero-nuclear experiments in the absence of Pulsed Field Gradients.
- A bad setting of these parameters will increase the t1 noise in these 2D experiments.
- The best way to set them is to adjust as good as possible the “Lock Power” (find the 3dB limit before solvent deuterium nucleus eigenstates saturation occur). If this value of the “Lock Power” is found you can use the automation routine “loopadj”. This is an AU program that calculates the “Loop Gain”, the “Loop Filter”, the “Loop Time” and adjusts the “Lock Gain” and the “Lock Phase” parameters.



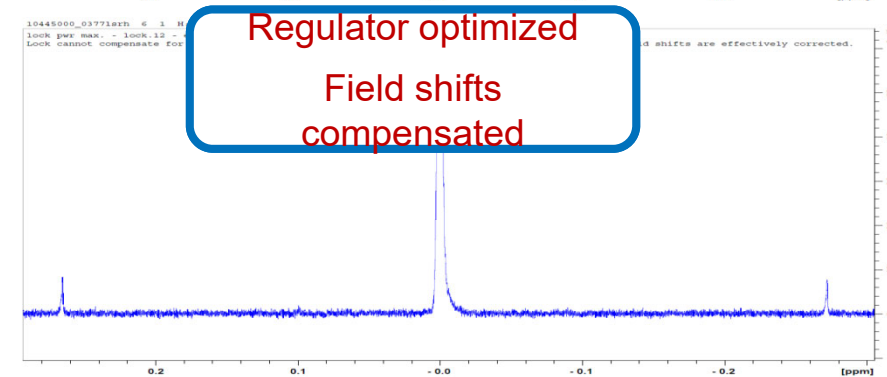
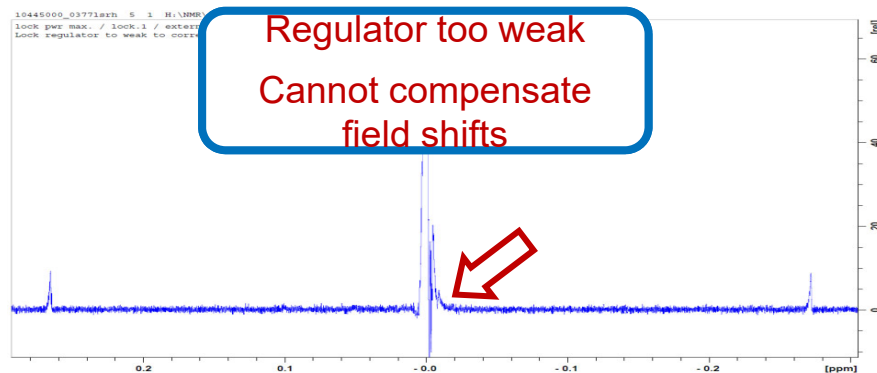
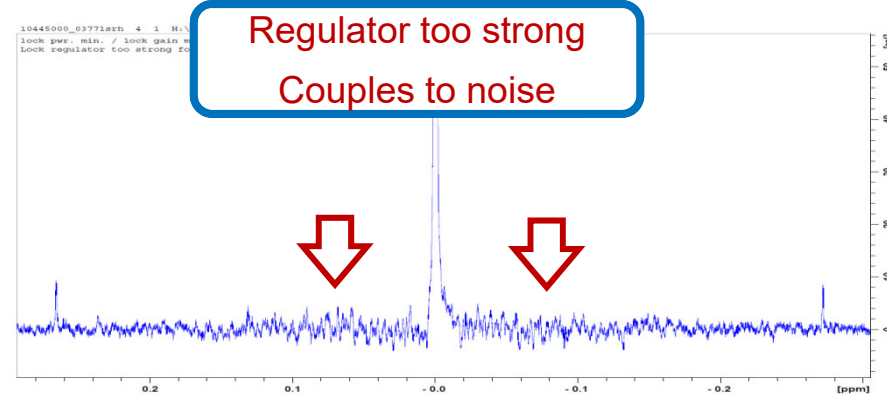
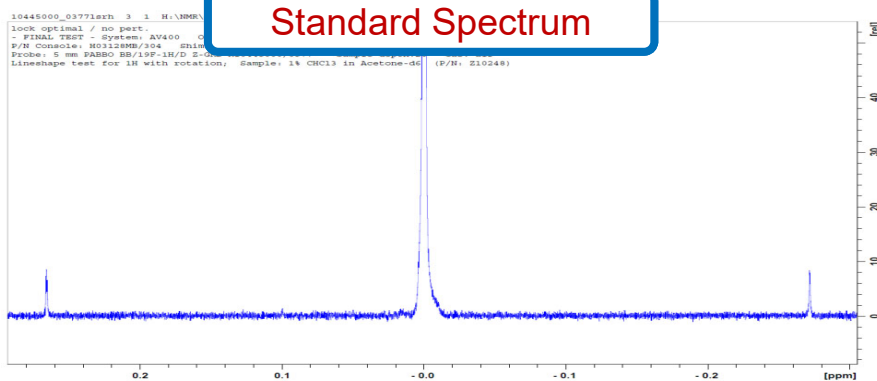
Also check the other lock parameters ...

- The "loopadj" command does not work if the "Lock Gain" is higher than 120.0.
- A "Lock Gain" value higher than 120.0 does not match with the "Loop Gain", "Loop Time" and "Loop Filter" values defined in the following table

Lock parameters			
Macro	Loop Filter Hz]	Loop Gain [dB]	Loop Time [s]
lock.1	20	-17.9	0.681
lock.2	30	-14.3	0.589
lock.3	50	-9.4	0.464
lock.4	70	-6.6	0.384
lock.5	100	-3.7	0.306
lock.6	160	0.3	0.220
lock.7	250	3.9	0.158
lock.8	400	7.1	0.111
lock.9	600	9.9	0.083
lock.10	1000	13.2	0.059
lock.11	1500	15.2	0.047
lock.12	2000	16.8	0.041

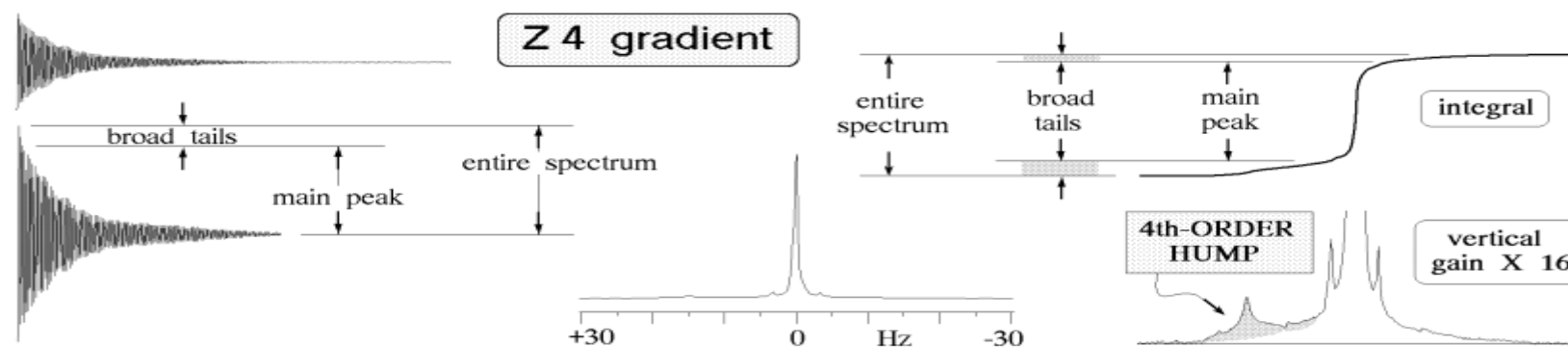
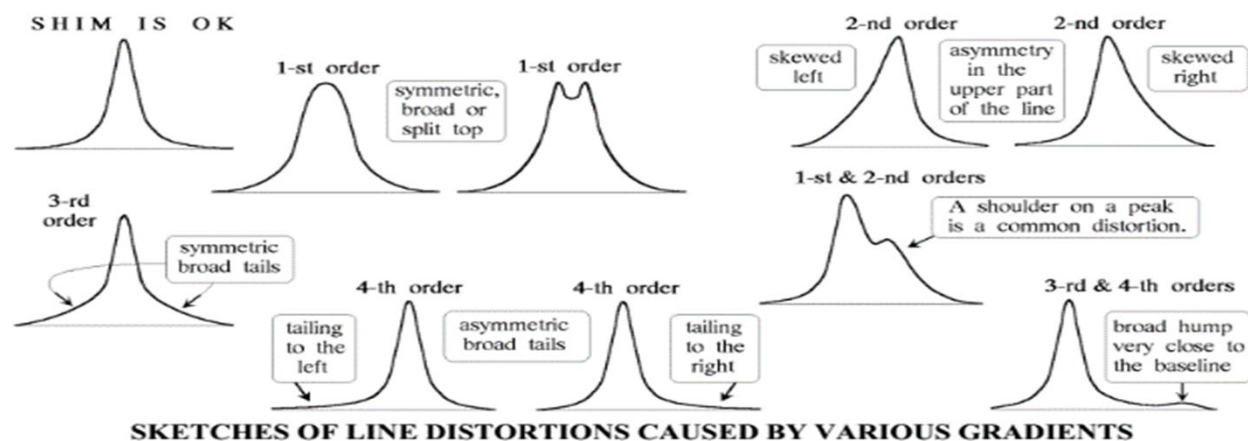


Also check the other lock parameters ...



Courtesy of Dr. Christoph Freudenberger BRUKER BIOSPIN Fällanden

Principles of shimming and shim errors



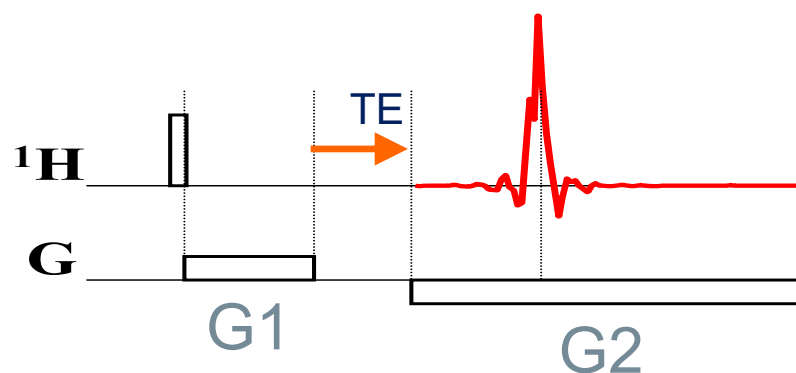
From «Shimming an NMR Magnet» by G. A. Pearson. Highly recommended!

Shimming: Preparation

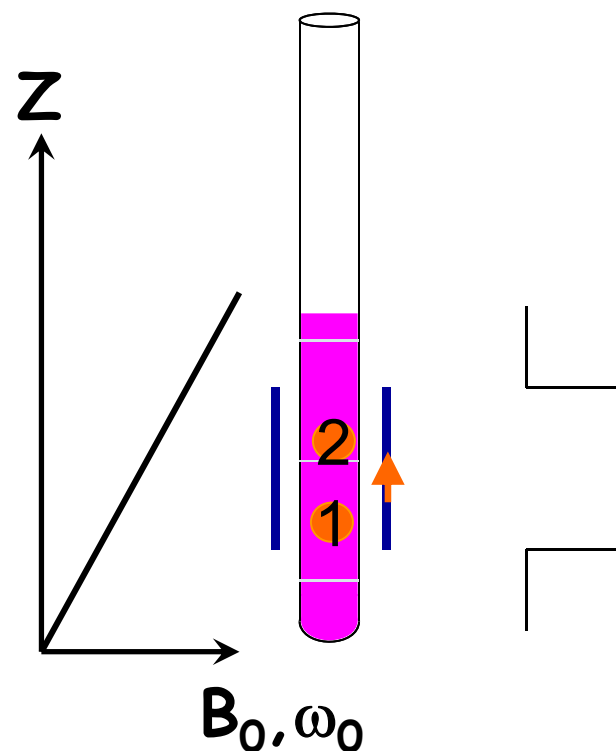


- Manual Shimming:
 - Adjust the LOCK PHASE to maximize the lock level
 - Adjust the LOCK POWER according to the solvent
 - Lock level is the criterion for evaluation of magnet homogeneity

Gradient shimming: Convection



1. Convection causes a wrong information according to the position in the sample.
2. Lost of the signal because of a incomplete focusing caused by the second gradient.



Gradient shimming: Convection



Counteractions:

1. Avoid convection:
 - Sample filling height of 40 mm.
 - Increase the airflow.
 - Rotate the Sample.
 - Lower the temperature (if possible).
 - Use a solvent with a higher viscosity (if possible).
2. Version of TopShim with convection compensation:
 - TopShim in TopSpin 2.1 patch level 5 (or higher)
3. Version of Gradshim with convection compensation:
 - Pulse programs on request

Gradient shimming: Convection



Common problems with TopShim:

- Situation:
 - bad result without sample rotation
 - Good result with sample rotation
- Reason:
 - Convection

Topshim window [topshim gui]

A screenshot of the TopShim GUI window. The window has a title bar "TopShim" and three tabs: "Shim", "Report", and "Service". The "Shim" tab is active. It contains several sections: "SHIM" with radio buttons for "1D" (selected) and "3D", a dropdown for "Optimisation" set to "solvent's default", a dropdown for "Optimise for" set to "1H", and a checkbox for "Use Z6" which is unchecked. "TUNE" section has dropdowns for "Before" (off) and "After" (Z), and an unchecked "Only" checkbox. "PARAMETERS" section has a checked checkbox and a text field containing "ordmax=8 convcomp". "STATUS" section shows "not running". "CONTROL" section has four buttons: "Start", "Stop", "Help", and "Close". Red arrows point from the explanatory text on the right to specific controls in the GUI.

• 1D shimming (Z-Z5 only, on every sample) 3D (all shims, only 90% H₂O sample using LOCK H₂O+D₂O)

• Optimization criterium

• For which coil optimize

• Z6 also (usually no)

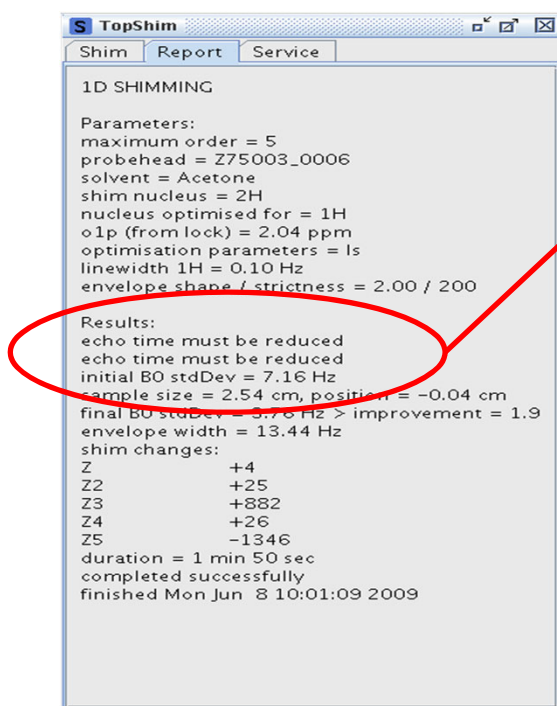
• Shimming of Z,X,Y,XZ,YZ by lock before and after gradient shimming (before makes real sense)

• Start button

TopShim: Remarks during shimming



Remark: **echo time must be reduced**



```
TopShim
Shim Report Service

1D SHIMMING

Parameters:
maximum order = 5
probehead = Z75003_0006
solvent = Acetone
shim nucleus = 2H
nucleus optimised for = 1H
o1p (from lock) = 2.04 ppm
optimisation parameters = ls
linewidth 1H = 0.10 Hz
envelope shape / strictness = 2.00 / 200

Results:
echo time must be reduced
echo time must be reduced
initial B0 stdDev = 7.16 Hz
sample size = 2.54 cm, position = -0.04 cm
final B0 stdDev = 5.76 Hz > improvement = 1.9
envelope width = 13.44 Hz
shim changes:
Z      +4
Z2     +25
Z3     +882
Z4     +26
Z5     -1346
duration = 1 min 50 sec
completed successfully
finished Mon Jun  8 10:01:09 2009
```

Results:
echo time must be reduced
echo time must be reduced
initial B0 stdDev = 7.16 Hz

Reason and solution:

Second Echo time was too long. As a result the magnetization relaxes during the pulse sequence.

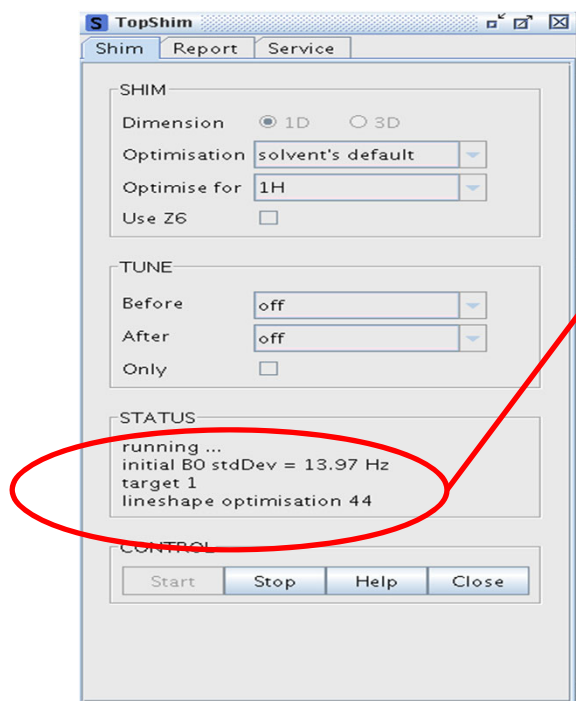
Reason:

- 1.The 3D homogeneity is not good (bad off-axis shim).
- 2.Convection.

TopShim: Remarks during shimming



Remark: Shimming takes too much time



running ...
initial B0 stdDev = 13.97 Hz
target 1
lineshape optimisation 44

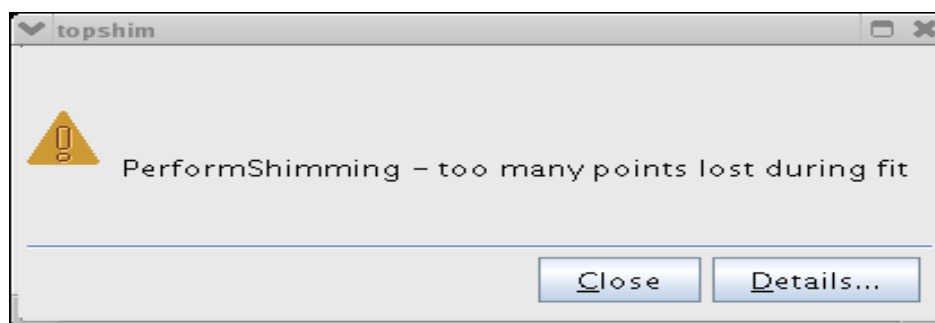
Reason and solution:

1. The 3D homogeneity is not good (bad off-axis shim).
2. Convection.

TopShim: Remarks during shimming



Remark: **Too many points lost during fit**



Reason and solution:

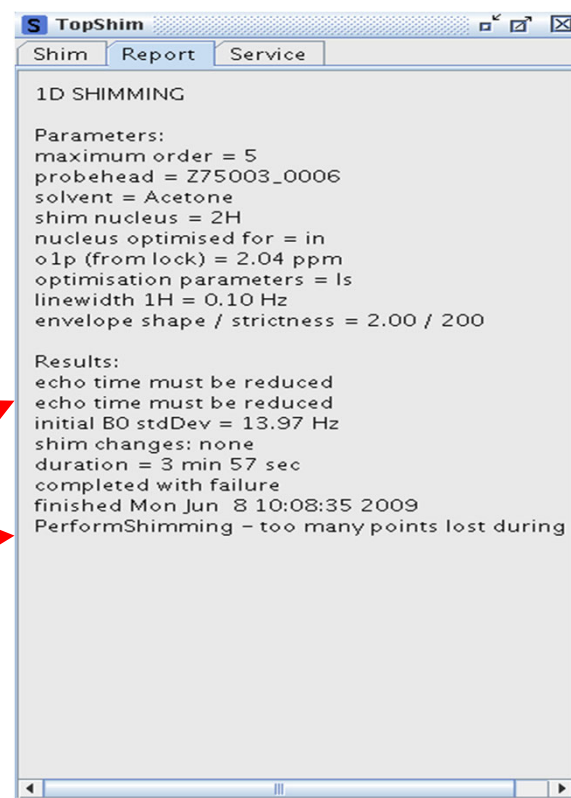
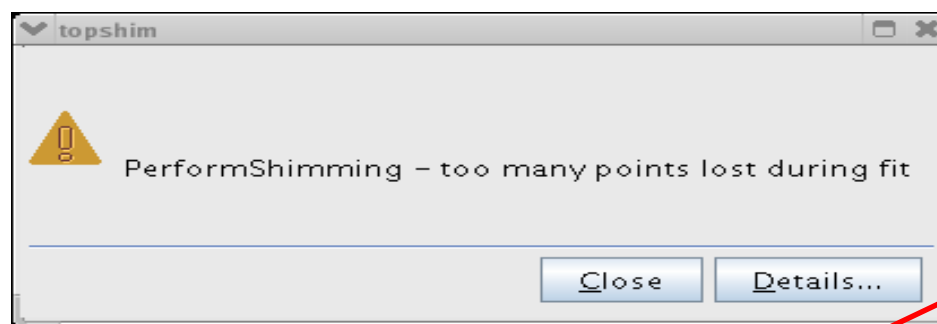
The basic homogeneity may not be sufficient.

1. Improve the homogeneity by manual or automated shimming on the lock.
2. Set very large values of high order shims to zero.



TopShim: Remarks during shimming

Remark: Too many points lost during fit

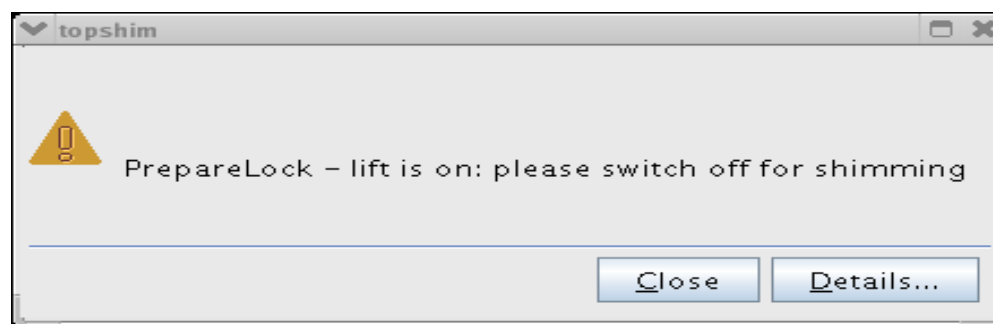


One error can cause another one



TopShim: error message

Error message:

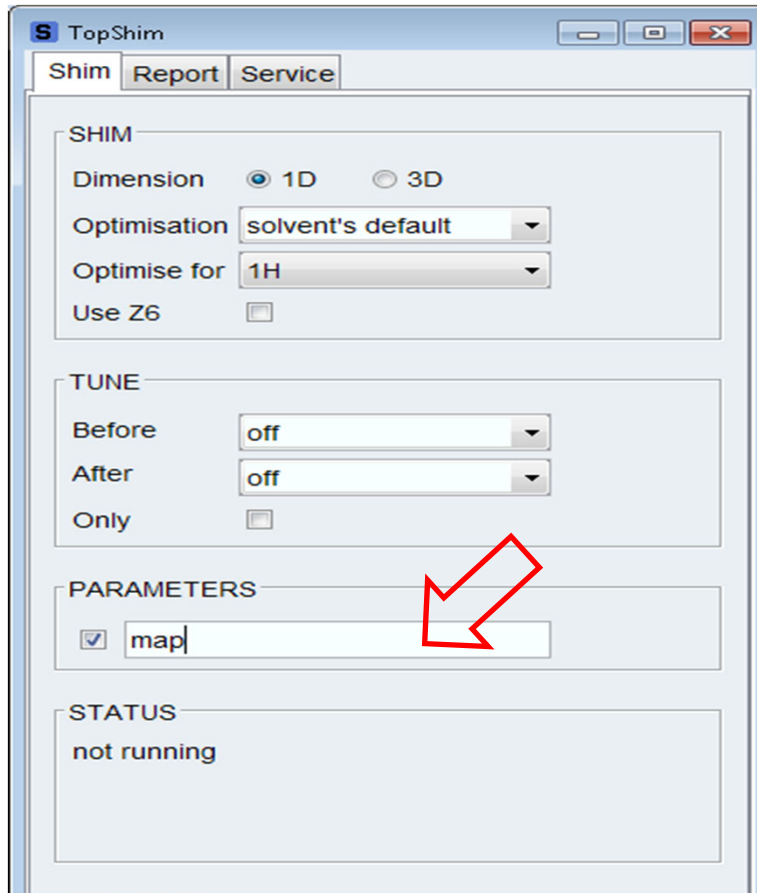


Reason and solution:

Lift is on.

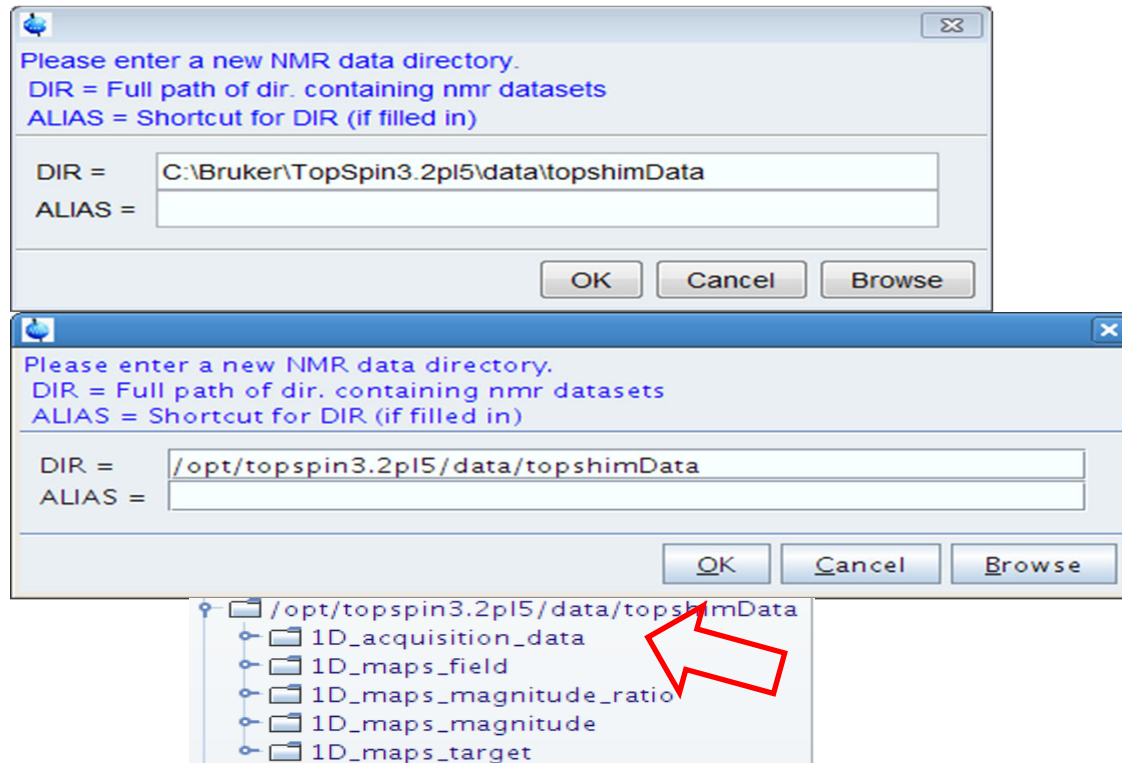
Sample is not locked.

TopShim:

A screenshot of the TopShim software interface. The window has a title bar with "S TopShim" and standard Windows controls. Below the title bar are three tabs: "Shim", "Report", and "Service", with "Shim" being the active tab. The main area is divided into four sections: "SHIM" with radio buttons for "1D" (selected) and "3D", a dropdown for "Optimisation" set to "solvent's default", a dropdown for "Optimise for" set to "1H", and a "Use Z6" checkbox; "TUNE" with dropdowns for "Before" and "After" both set to "off", and an "Only" checkbox; "PARAMETERS" with a checked checkbox and a text field containing "map"; and "STATUS" showing "not running". A red arrow points to the "map" text field in the PARAMETERS section.

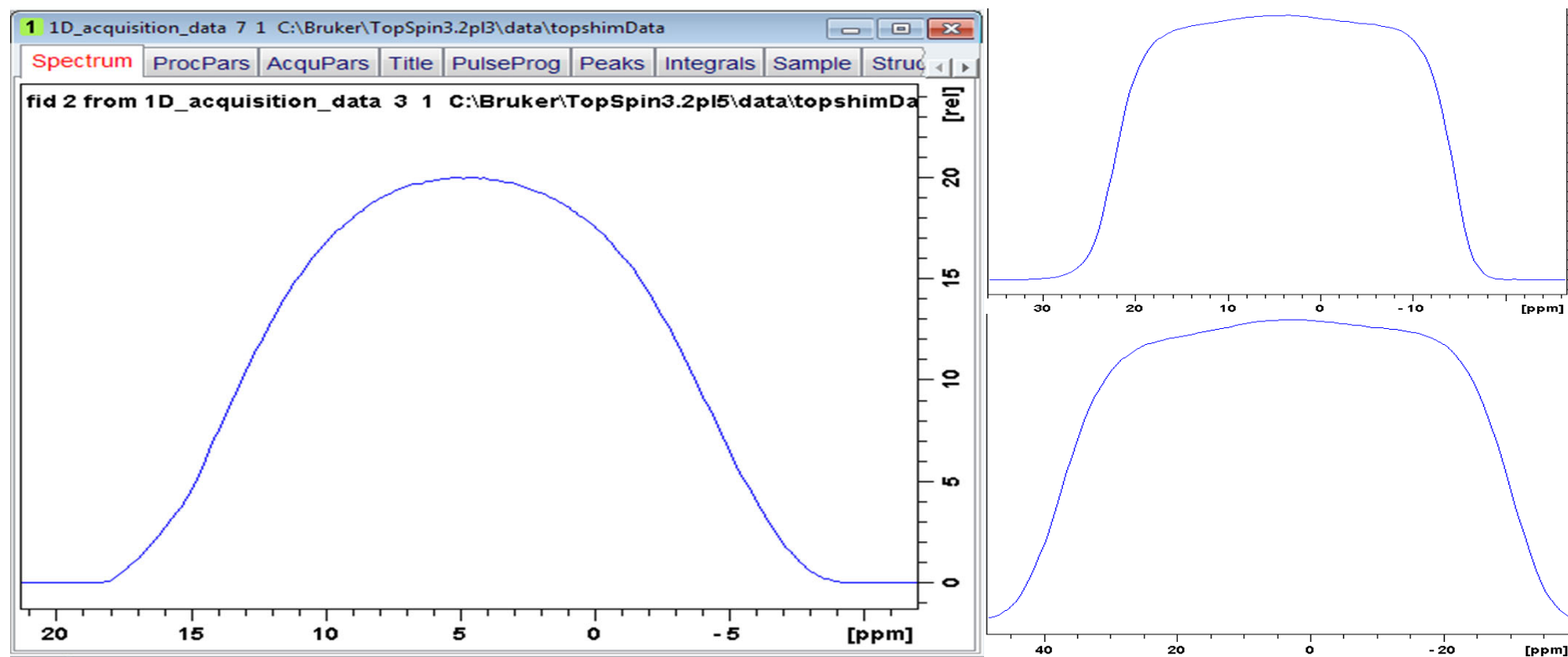
- Use e.g. Sucrose in H₂O/D₂O
- Start TopShim with the option "map"
- A Shim optimization will not be executed
- The current state will be documented with a Z-gradient profile.

TopShim



- The results will be stored in the folder: topshimData
- This folder is a sub folder of TopSpin
- Please Note: These data will be removed with the next TopShim run !!
- Send us this dataset directory for investigation

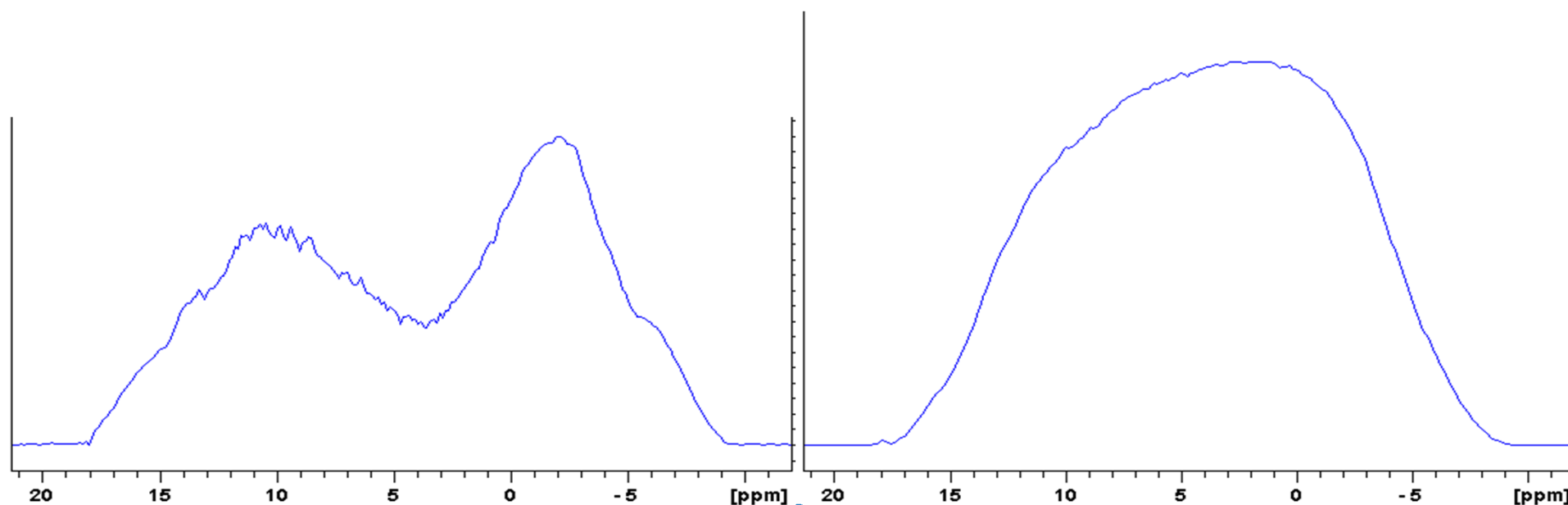
TopShim – good Z-gradient profile



TopShim – critical Z-gradient profile



- no hardware defect!
- Off-Axis Shims are really bad !



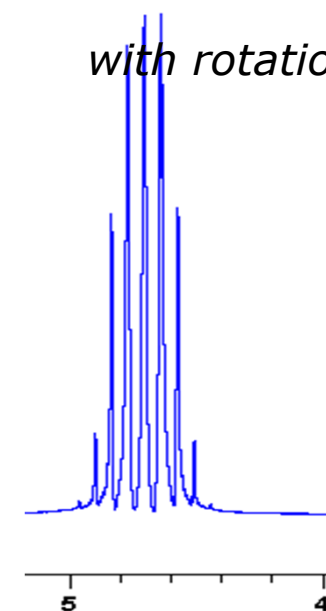
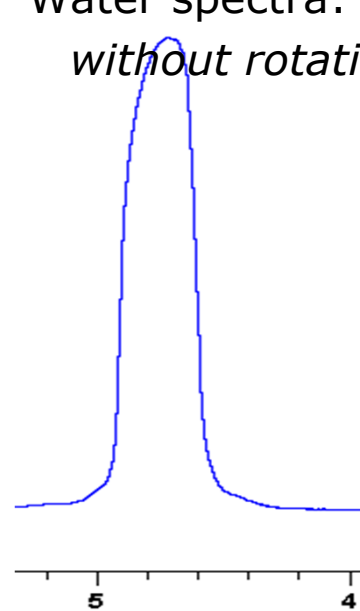
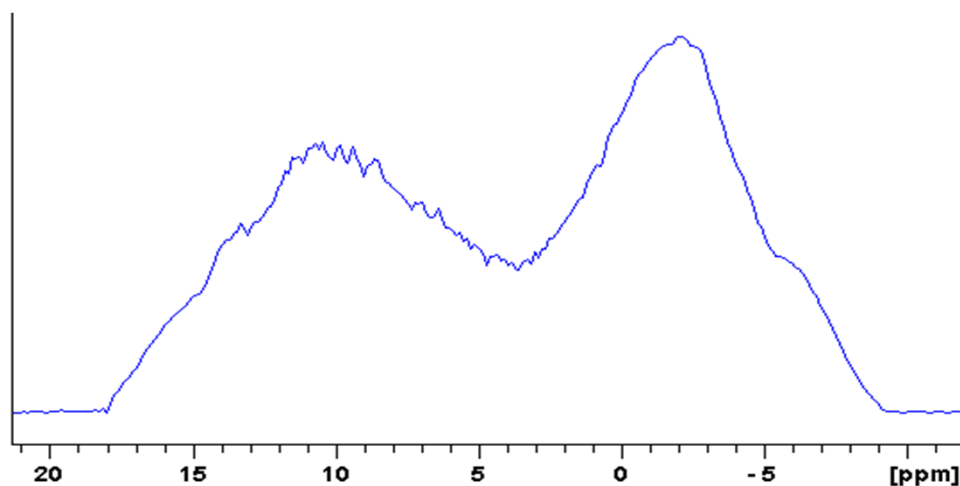
TopShim – critical Z-gradient profile



- no hardware defect!
- Off-Axis Shims are really bad
- Water spectra:

without rotation

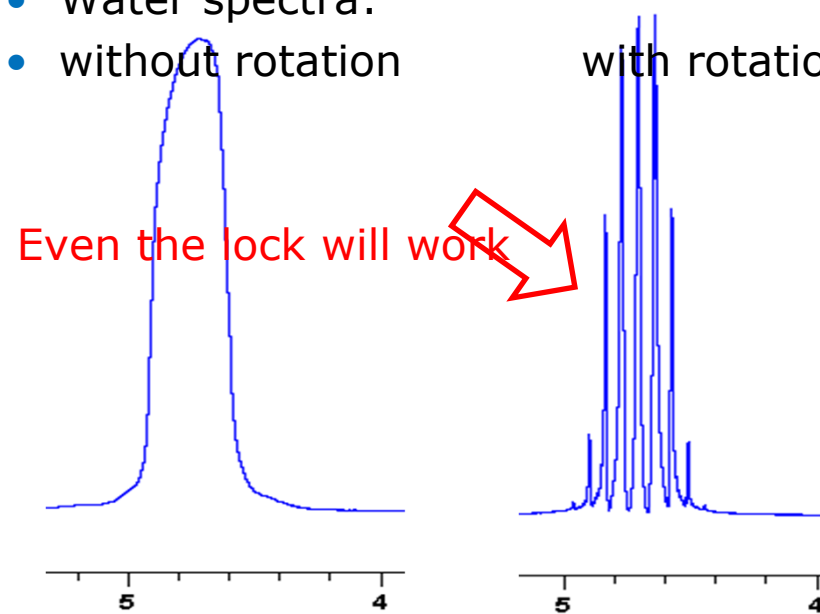
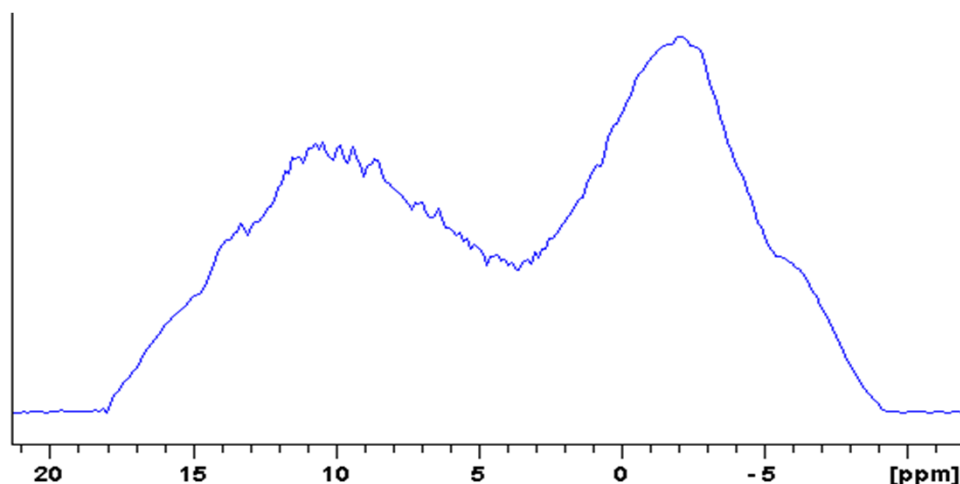
with rotation



TopShim – critical Z-gradient profile



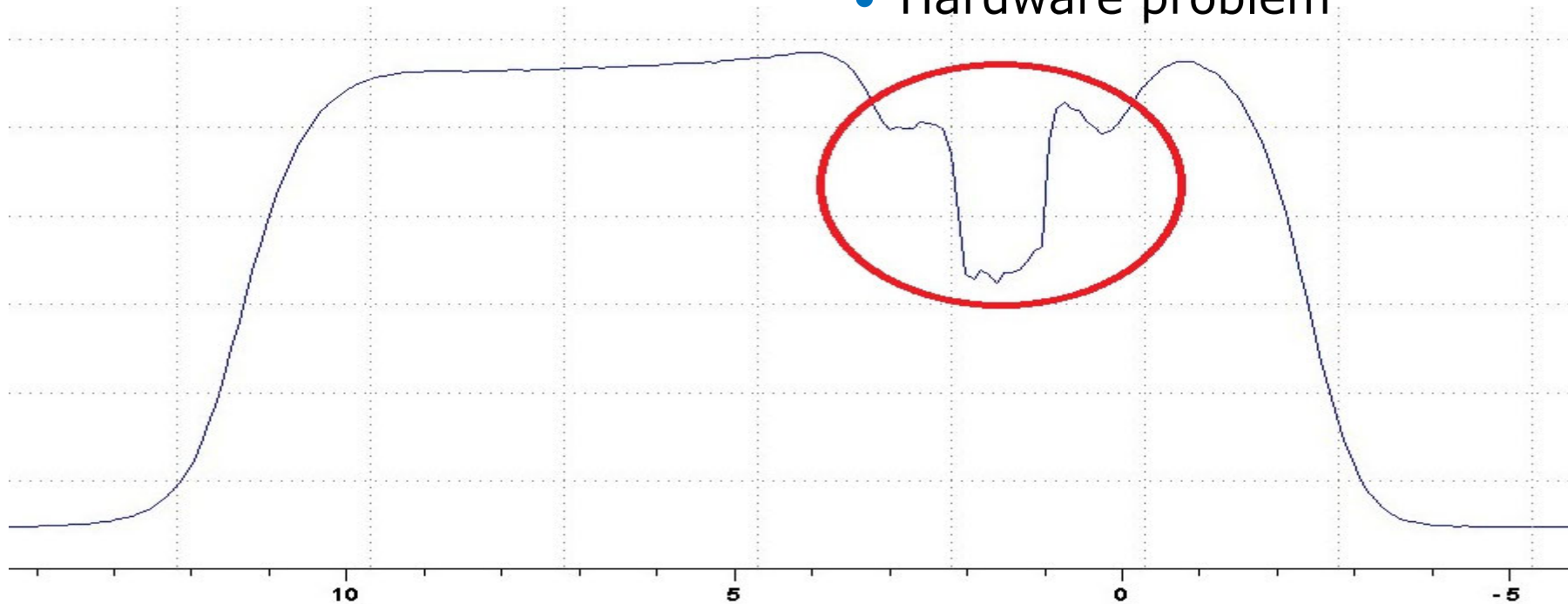
- no hardware defect!
- Off-Axis Shims are really bad
- Water spectra:
- without rotation
- with rotation



TopShim – critical Z-gradient profile



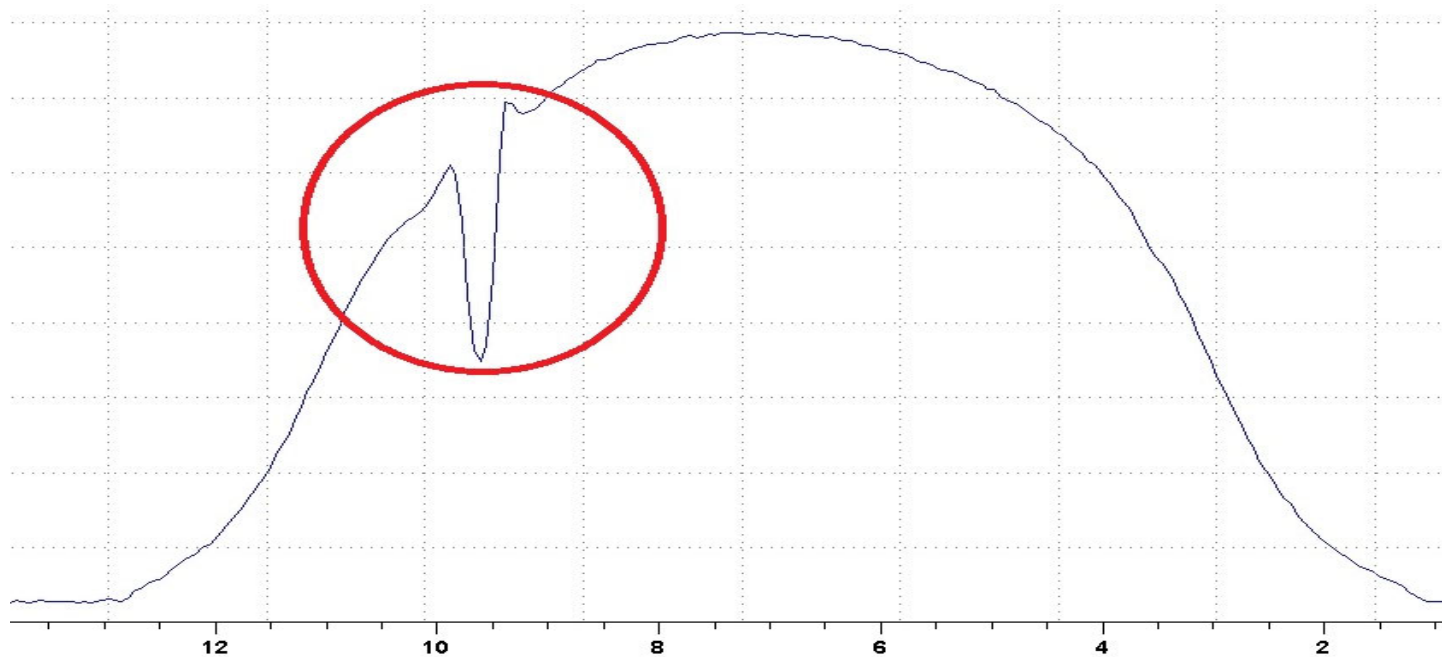
- Hardware problem



TopShim – critical Z-gradient profile



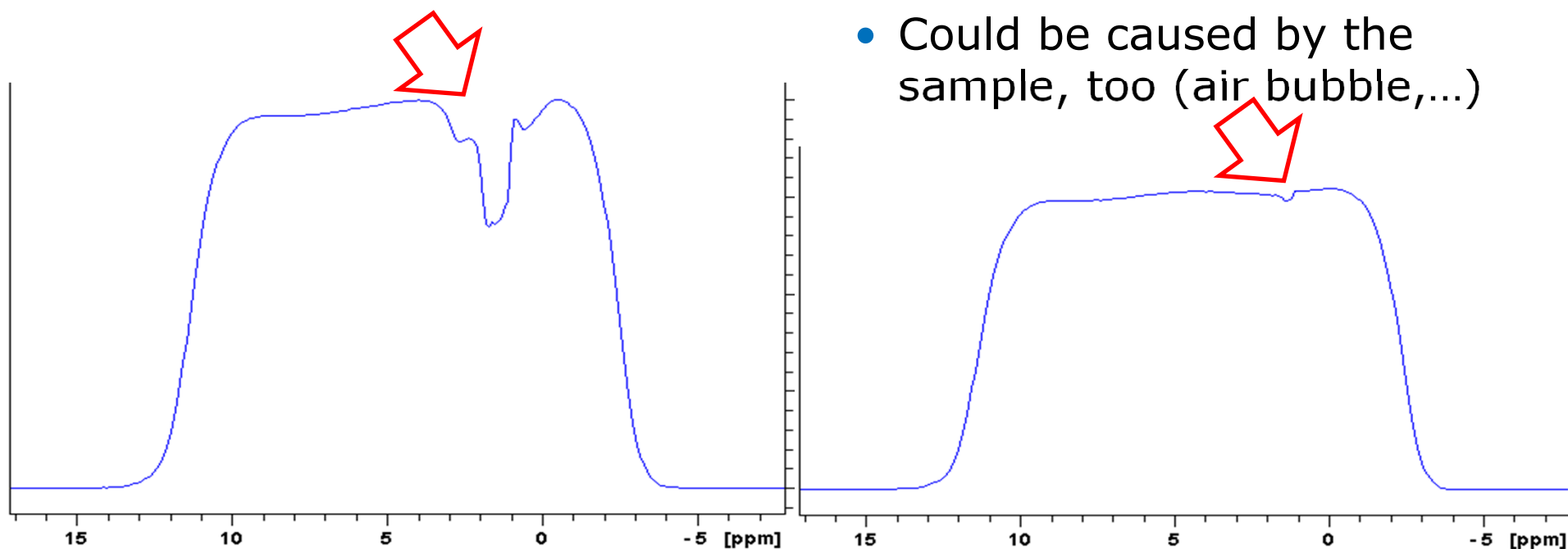
- Hardware Problem



TopShim – critical Z-gradient profile



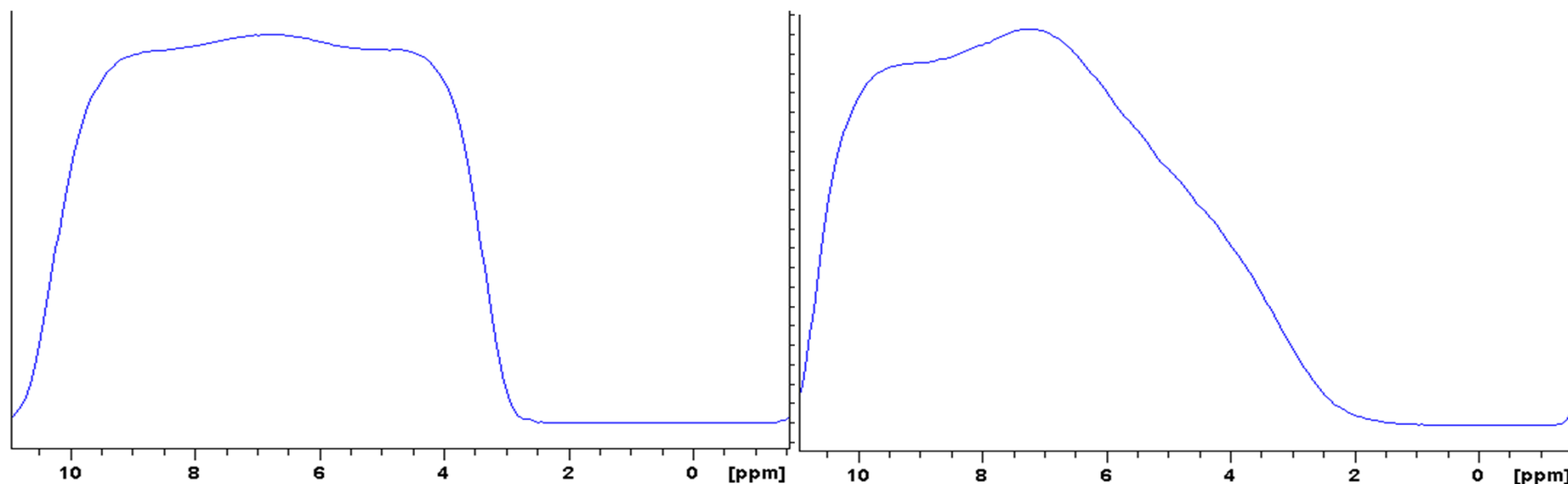
- Hardware problem
- Could be caused by the sample, too (air bubble,...)



TopShim – critical Z-gradient profile



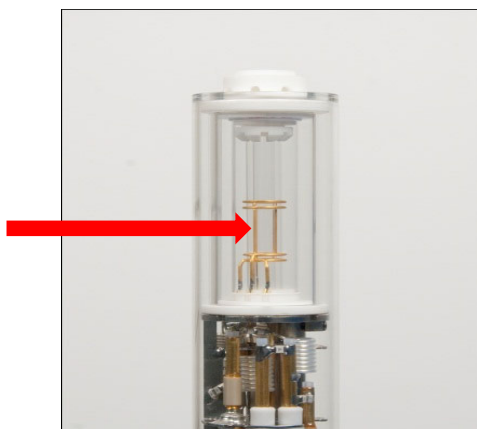
- Hardware problem
- Please contact Bruker!



NMR Thermometer



High Resolution



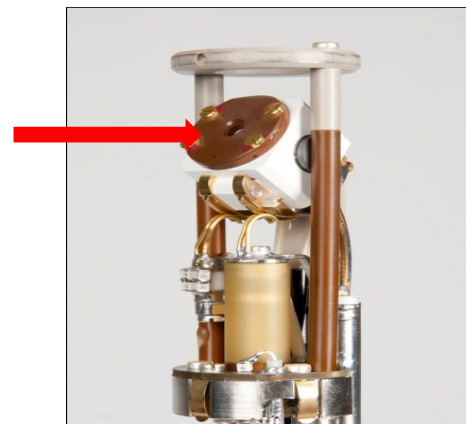
Heating of the sample by RF $\sim 2\text{K}$

0 – 12 Hz shifts

H_2O : $\sim 0.01\text{ppm} / \text{K}$

@600MHz = 6Hz / K

HR-MAS



**Heating of the sample by MAS
up to 30K**

NMR Thermometer



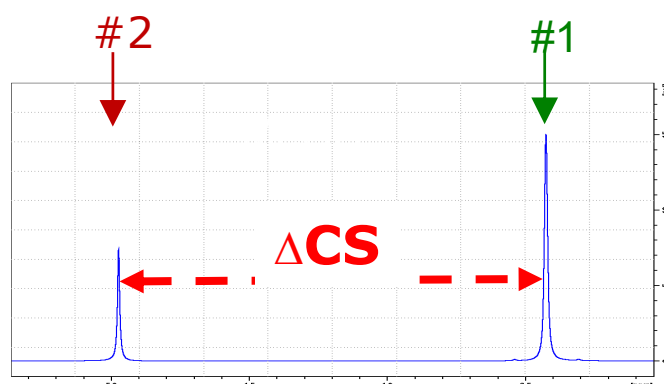
- **Measuring**
- **Regulation** of the temperature **in** the sample
- **Monitoring**

Method of measurement is known:

Measure the temperature dependent Chemical Shift

Regulation

NMR Thermometer



Solvent with at least two ^2H lines with different temperature dependent behavior of the CS.

Measure ΔCS

L-TRX (2G DigiLock) transmits and detects two frequencies simultaneously.

#1 is fixed = **Lock**

Defined CS of the Lock-**Peak**:. Field is regulated, so that the **Peak** is on Resonance.

#2 is variable = **Temperature**

CS of the second **Peak**. Frequency is regulated, so that the **Peak** is on Resonance.

NMR Thermometer - Advantages



The advantages are analogue to the solvent as Lock-substance

Solvent has exact the temperature of the sample

Solvent is spectroscopically independent of the sample

No additional spectra have to be recorded

Update of the temperature with high rate (as Lock, about 6600/s)

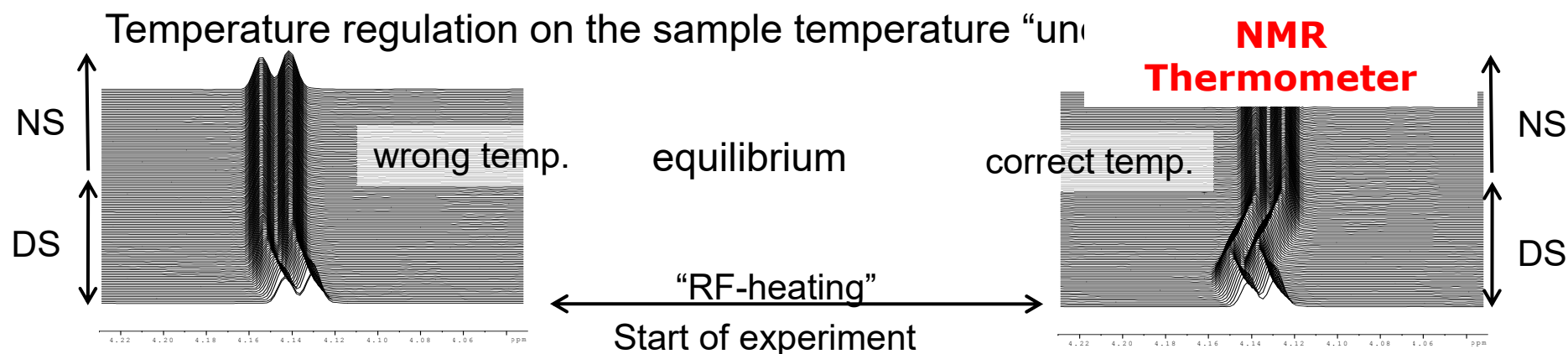
Fast and easy data transfer between BSMS and SmartVT unit because of integrated infrastructure. L-TRX ELCB BSVT

NMR Thermometer

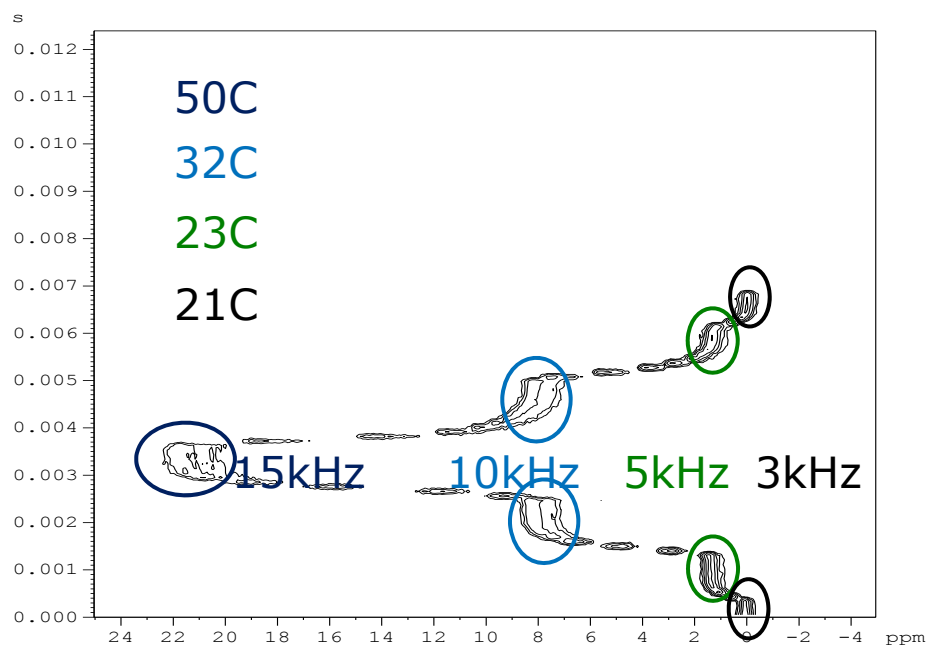


“Correct” temperature (if calibrated correctly).

Identical temperature on different probes and spectrometers.

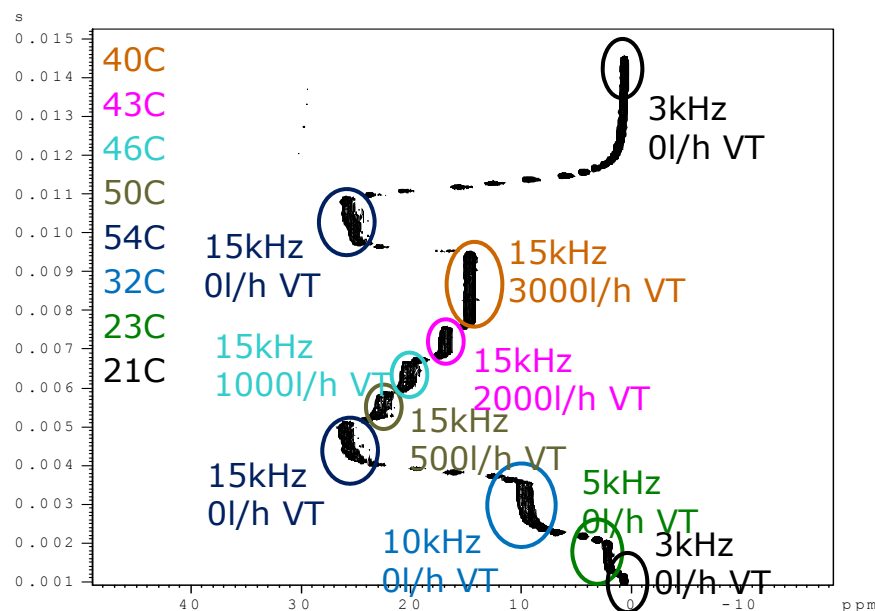


Influence of Magic Angle Spinning on the Temperature



- Center packed sample (4mm, ZrO powder)
- 21C at 3kHz (room temperature, referencing)
- Lead nitrate for temperature calibration, 0.753ppm/K
- Spin rate was changed within running experiment
- Maximum temperature difference about 30K above RT

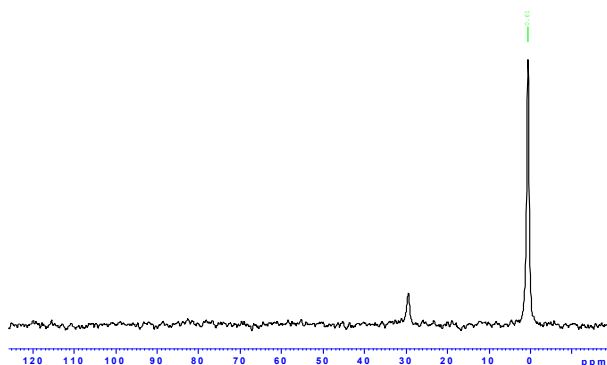
Influence of Magic Angle Spinning and VT Gas on the Temperature



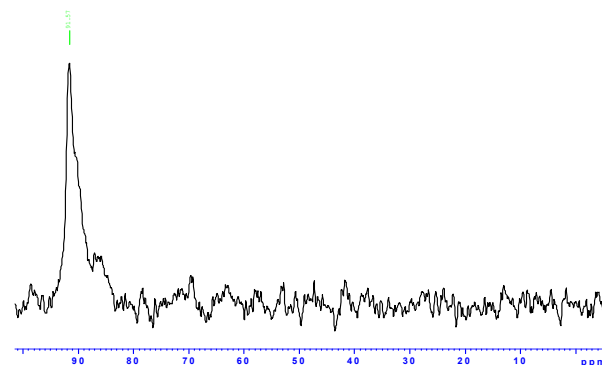
- Center packed sample (4mm, ZrO powder)
- 21C at 3kHz (room temperature, referencing)
- Lead nitrate for temperature calibration, 0.753ppm/K
- Spin rate was changed within running experiment and VT gas flow was adjusted
- Maximum temperature difference about 34K above RT



Temperature Calibration

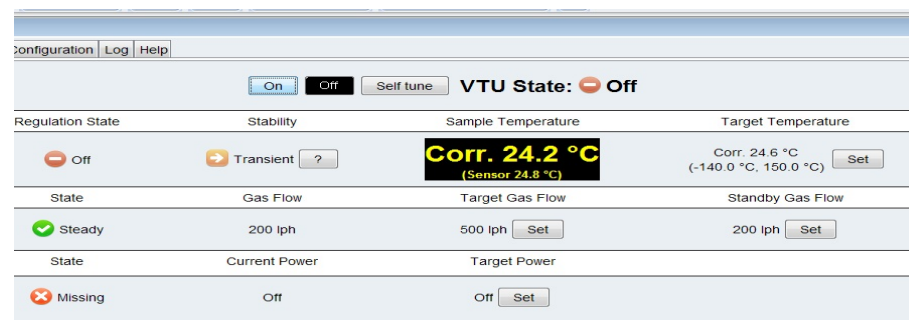


Tset=294K: 0.61ppm = 20.3C



Tset=413K: 91.55ppm = 141.8C

- Calibration with 2 or more temperatures
- Calibration curve is calculated either by EXCEL/ORIGIN or TS
Slope:0.979424 / offset: 6.734979
- After activation EDTE shows both values



Temperature Calibration – edte



Temperature Control Suite

TemperatureMonitoringRecordCorrectionConfigurationLogHelp

Temperature correction

Use temperature correction if you want to display the real sample temperature instead of the probe temperature sensor value. Please check the manual how to perform temperature measurements with NMR (to determine the real sample temperature).

Note: Temperature correction is not applied to temperature limits (safety checks).

☒ Enable temperature correction with these values

Name: DUL_PHIP

Probe: 5 mm FIDUL 13C/2H-1H Z-GRD H8902/0201

Temperature range [K]: 288.00 – 303.00

Slope: 0.939850

Offset: 14.766917

Comment:

Available correction settings

Δ Name	Probe	Temperature Range	Slope	Offset	Comment
DUL_PHIP	5 mm	288.00 – 303.00	0.939850	14.766917	
DUL_PHIP	5 mm PAI	303 – 373	1.113	21.4	

NewEditSetDelete

VTU State: On✔Sample Temperature: Corr. 293.0 KProbe Regulation State: Transient⚡Recording: Off⏻Probe: 5 mm PABBI 1H-BB Z-GRD Z810701/0040



Temperature profiles – edte

Temperature Control Suite

Temperature | Monitoring | Record | Correction | Configuration | Log | Help

General configuration

Temperature unit used in VTU display:
☒ Kelvin [K]
☐ Celsius [°C]

Power unit used in VTU display:
☒ Watt [W]
☐ Percent [%]

Location of VTU display (internal/external):
☒ external Window
☐ internal Window

Profile configuration

VTU related settings (e.g. temperature unit, names limits etc.) can be stored in a profile. You can load a profile to apply the stored VTU settings to different hardware configurations or users.

Create new profile

Load profile

Delete profile

Channel mapping

Set mapping between te-parameters (e.g. te2) or te-commands (e.g. te2set, te2get) and hardware channels. This determines which channel is used for each command or parameter.

Hardware channel	Logical channel
1	1
2	2
3	3
4	4

Channel configuration

Channel	Regulation Mode	Min. Temperature	Max. Temperature	Max. Safety Temperature	Max. Heater Power
1 5 mm PABBI 1H-BB Z-GRD Z810701... <input type="button" value="Set"/>	Standard <input type="button" value="Set"/>	180.0 K <input type="button" value="Set"/>	400.0 K <input type="button" value="Set"/>	500.0 K <input type="button" value="Set"/>	20.00 W (max. 20.00 W of 210.0 W) <input type="button" value="Set"/>

VTU State: On ☒ | Probe Temperature: 290.1 K | Probe Regulation State: Transient ☒ | Recording: Off ☐ | Probe: 5 mm PABBI 1H-BB Z-GRD Z810701/0040

User can store probe profiles. This way, it is easy to handle different settings.

BSMS shim cooling and probe flush



Experiments at very high or very low temperatures need some care.

- The probe electronics should be protected from excessive heat or icing.
 - This can be done by using flush gas (on newer probes)
- The shim system and the O-rings of the magnet (vacuum sealing) must be protected from excessive temperature.
 - This can be done by using shim flush gas. A special adapter (clamp ring) is necessary for the Shim Upper Part.

Conclusion - Solids NMR



- Calibrate your probe
- Choose carefully the flow/hardware conditions
- Use center packed samples whenever possible
- Higher flow rates can improve the overall performance
- The probe/shim needs time for thermal equilibrium (30min to 1h)
- The most versatile cap is the ZrO cap
- Keep an eye at the magnet's shim temperature

Conclusion - Solution NMR



- Run "self tune" with your probe (different temperature ranges >30-50K may require different PID parameters).
- Choose carefully the flow to optimize temperature gradients (higher flow typically reduces gradient but be careful not to "lift" the sample).
- If absolute temperature is of interest, calibrate the probe with a thermal standard (depends on flow rate) and set the correction in edte accordingly.
- The probe/sample needs time for thermal equilibrium (>5min).
- Choose the best hardware option get cold gas.
- Keep an eye at the magnet's shim temperature.

The Importance of Air!



- Are you sure what kind of gas you get out of your supply? Air or clean N₂?
- Does it make a difference?
- How about quality of the supplied air?

Air or N₂:

The answer is important, because your probe is most likely optimized for one or the other. Most new RT-PH (Room Temperature Probes) will only work nicely if used with N₂ -> shimming will be difficult if used with air!

All RT HR-PH >400 MHz + all RT small volume PH:s (1mm/1.7mm)

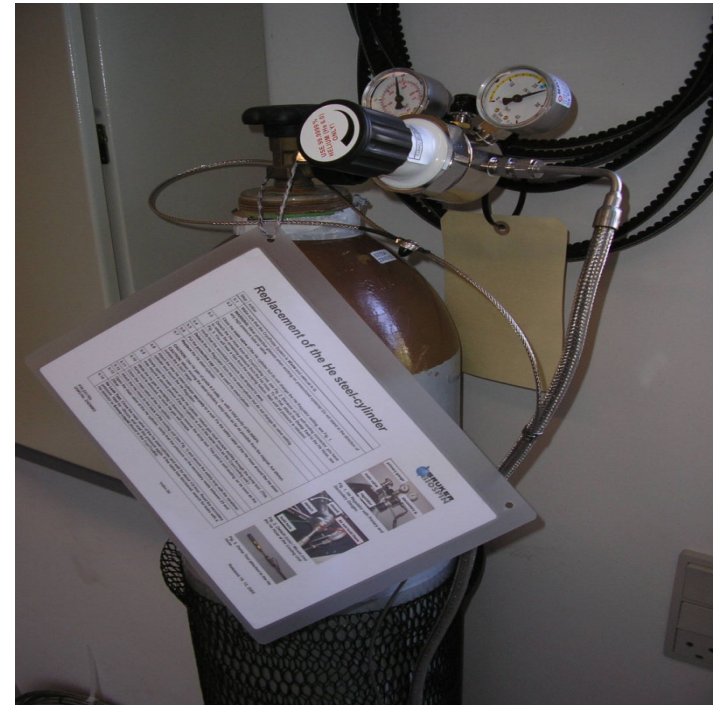


The Importance of Air!



Cryoprobe platforms

- Only use grade 6 helium quality (99.9999 % He)!
- Make sure the regulator is not manipulated: 24-25 bar
- Follow the instructions when replacing bottle
- In case of Shimming problems: try to increase the airflow. Watch wobble curve and lock !!



The Importance of Air!



What is the pressure? Is it stable?

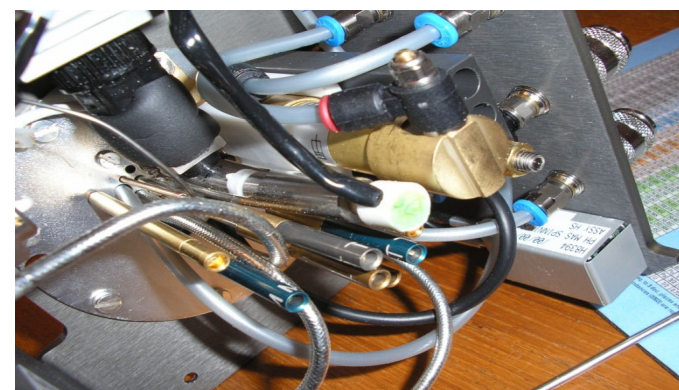
Magnets on air posts will move up/down!
Sample changer may fail.

Solution: Connect a **buffer tank**, possibly along with a **higher capacity compressor**.

What is the dew point?

Bruker recommends a dew point $< -40^{\circ}\text{C}$. Install an efficient and silent dryer!

Condensation! Corrosion! Ice build-up!



The Importance of Air!



What flow can you use without pressure drop?

MAS and automation will need a larger flow (>100l/minute). Measurement may be necessary upon installation of new accessories.

How clean is the air? Amount and size of particles?

Clogging of MAS-PH and MAS-drive. Think about installing an **oil separators** and/or a **filter** at the outlet! The extra cost is small compared to the repair costs of a probe!



The Importance of Air!



What temperature has the incoming air?

If you're using air directly for the VTU (no pre-cooling), you may see stability problems.

Solution: Install a **pre-cooler** or a large **buffer tank**.

If you're uncertain or have questions, please contact your Bruker Service team.



The Importance of Air!

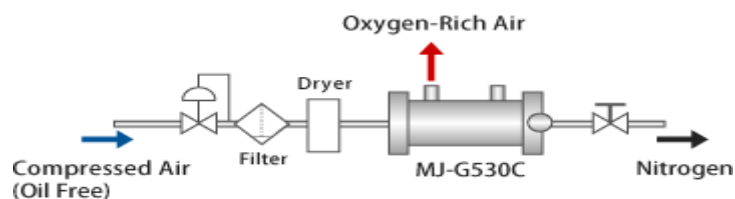


What if I only have compressed air and need N₂ for a new system or PH?

Solution : **N₂-Separator**.

The separator is using a membrane (passive, and only built for low flow applications)

Separate benefit: dew point will be reduced!



High/low temperature experiments



What are “extreme temperatures”?

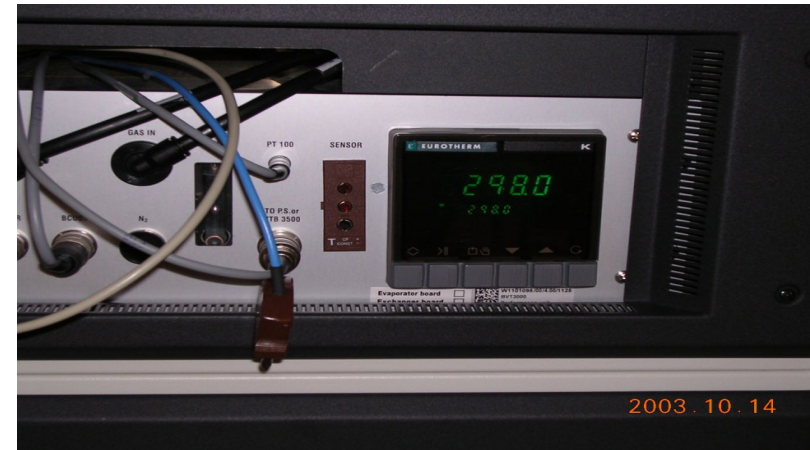
No strict definition, but outside [0-60] C would make sense 😊

But my probe specs say [-150- +150] C?

Yes, but it takes more than a PH to make a system.

Does this mean that I should not exceed [0-60] C?

Not necessarily, but if you do, take extra precaution and supervise the system during the experiment.



High/low temperature experiments



Why are there restrictions?

1. The PH itself has limitations (material & design).
2. The shim system has a very well defined upper limit: +80C. Condensation of water could also damage it at temperatures below +4C.
3. The magnet will be in danger if parts of it are frozen - the vacuum will be softened and the insulation capacity decreased. Higher boil off, in the worst case sudden loss of cryogen and a quench, may occur.
4. The spinners have a limited temperature range (plastic ones about +60C, ceramic ones should be used at higher temperatures).

High/low temperature experiments



There are soft solutions:

1. If you need to run long time experiments at high temperatures or low temperatures, you can think about special probes (ET/LTB).
2. Supervise the shim tube temperature using “coiltemp” (an AU program in Topspin) and make sure it stays (well) below +80C. Future versions of Topspin will have a protection in the software.
3. Inspect the top and bottom magnet flanges and upper part for signs of freezing. This should be done continuously during the experiment at least some time after an equilibrium state.
4. And remember the ceramic spinner ...

High/low temperature experiments



There are more/hardware solutions:

1. From 2nd half of 2008 on (in some cases earlier) most RT-PH are equipped with a built-in frame cooling that will flush the interior parts using a constant flow of air/N₂
2. Upgrade with the cooling ring set that will flush the space between shim tube and RT-bore (the visible inner tube of the magnet) using air or N₂

Two version are available: one to retrofit systems delivered before 2nd half of 2008 (the shim tube needs to be taken out of the magnet), one for new systems already prepared for cooling rings (only some pieces are added, no need to remove the shim tube)

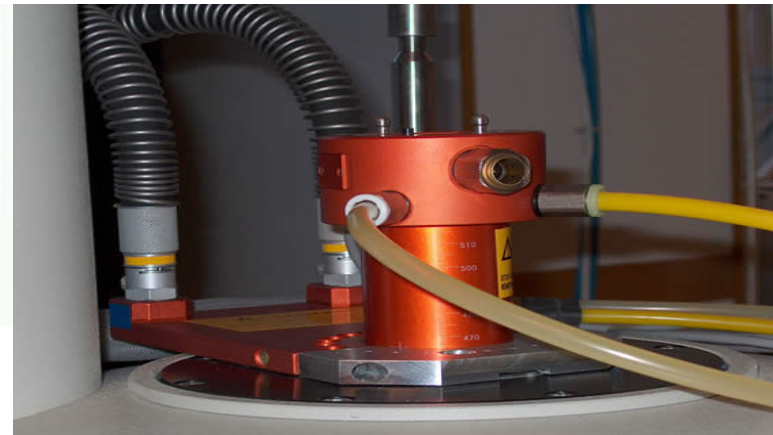
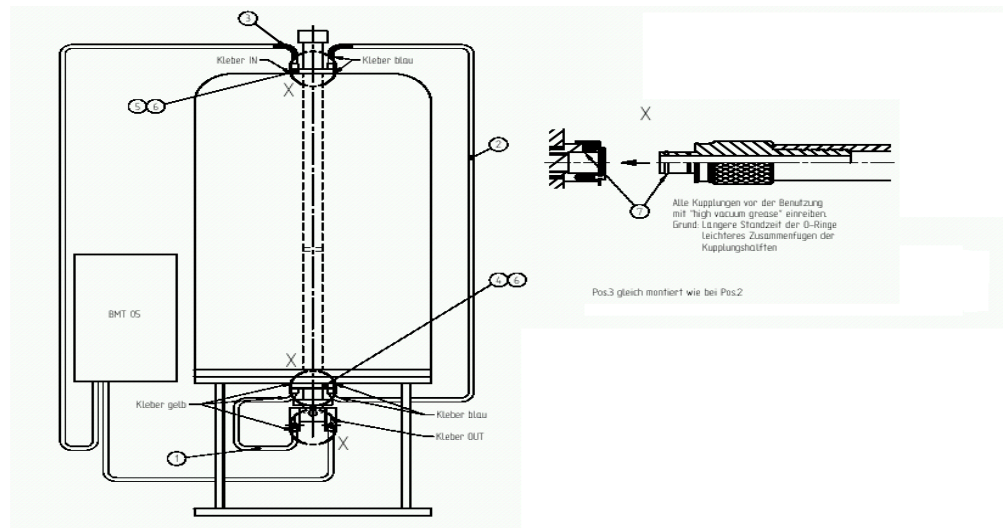


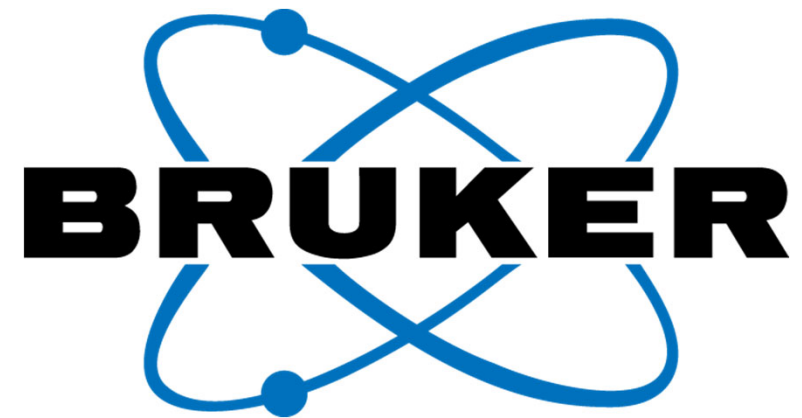
High/low temperature experiments



There are more/hardware solutions:

3. Upgrade with water heated system that will keep the upper/lower flanges at a constant (warm) temperature





Innovation with Integrity