



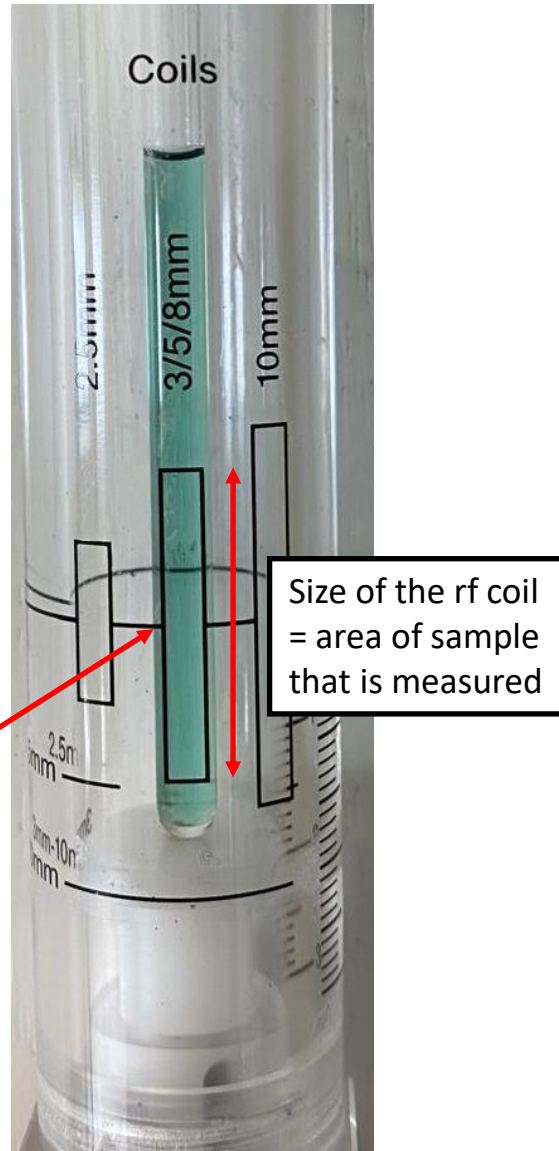
av400 NMR spectrometer

Getting started + Troubleshooting

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Sample centering



Place spinner on top of insertion tool and push sample through to the bottom

The rectangle marking the size of the rf coil should be “filled” with sample

For samples with lower filling height, you can carefully pull the tube out of the spinner until the rectangle is “filled” with liquid. If the bottom of the tube is inside the rectangle then, add more solvent

There should be no liquid-gas or liquid-solid interfaces within the rectangle, otherwise this might lead to lineshape distortions

Do not push the tube further into the spinner after taking it out of the insertion tool! The bottom of the tool marks the bottom of the probe, if the sample sticks too deep in the spinner, the tube might break while inserting it into the magnet!

Browser Last50 Groups

- /opt/topspin3.6.2/data/roesky/mn
- /opt/topspin3.6.2/data/schneider/MT
- /opt/topspin3.6.2/data/schneider/RvA
- /opt/topspin3.6.2/data/schneider/acg
- /opt/topspin3.6.2/data/schneider/bs
- /opt/topspin3.6.2/data/schneider/dd
- /opt/topspin3.6.2/data/schneider/dd-mh
- /opt/topspin3.6.2/data/schneider/dd_mh
- /opt/topspin3.6.2/data/schneider/fs
- /opt/topspin3.6.2/data/schneider/gs
- /opt/topspin3.6.2/data/schneider/ja
- /opt/topspin3.6.2/data/schneider/js
- /opt/topspin3.6.2/data/schneider/kw**
- /opt/topspin3.6.2/data/schneider/la
- /opt/topspin3.6.2/data/schneider/mf
- /opt/topspin3.6.2/data/schneider/mh
- /opt/topspin3.6.2/data/schneider/na
- /opt/topspin3.6.2/data/schneider/sb
- /opt/topspin3.6.2/data/schneider/sf
- /opt/topspin3.6.2/data/schneider/sj kf
- /opt/topspin3.6.2/data/schneider/tsr
- /opt/topspin3.6.2/data/schneider/yz
- /opt/topspin3.6.2/data/siewert/ad
- /opt/topspin3.6.2/data/siewert/if
- /opt/topspin3.6.2/data/siewert/ktk
- /opt/topspin3.6.2/data/siewert/lp
- /opt/topspin3.6.2/data/sindlinger/cps
- /opt/topspin3.6.2/data/sindlinger/cth
- /opt/topspin3.6.2/data/sindlinger/js
- /opt/topspin3.6.2/data/sindlinger/l n
- /opt/topspin3.6.2/data/sindlinger/mg
- /opt/topspin3.6.2/data/sindlinger/th
- /opt/topspin3.6.2/data/stalke/acf
- /opt/topspin3.6.2/data/stalke/ad
- /opt/topspin3.6.2/data/stalke/ak
- /opt/topspin3.6.2/data/stalke/bachelor

Data browser

Depending on the TopSpin version, the design might be somewhat different, but the operation stays the same

Sample status

Sample temperature

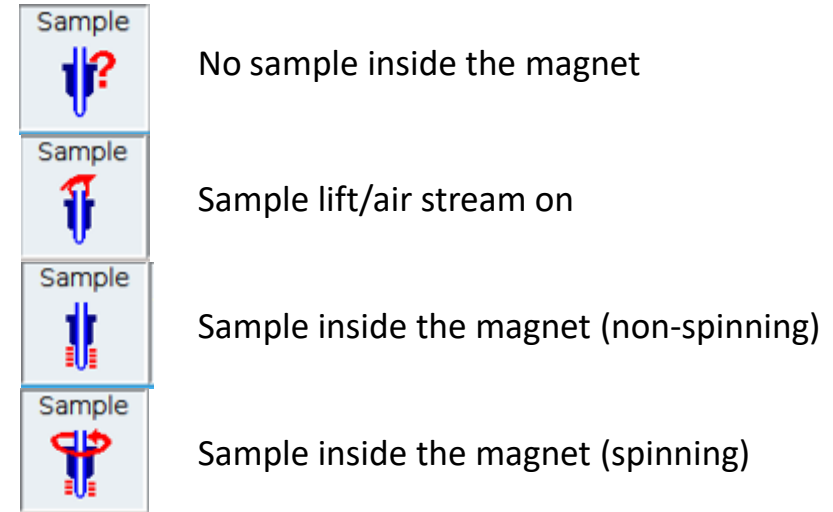
Measurement queue

Command line

Amplifier Control	Acquisition information	Fid Flash	Lock	Sample	Shim Coil Temperature	POWCHK	VTU	Spooler	BSMS status message	Time
	no acquisition running		12%		303 K	✓	[Celsius] 25.0	queued: 0 delayed: 0 cron: 0	Δ Y3 0 Autoshim Locked Error	15:06:40 Mar 16

Sample insertion and ejection

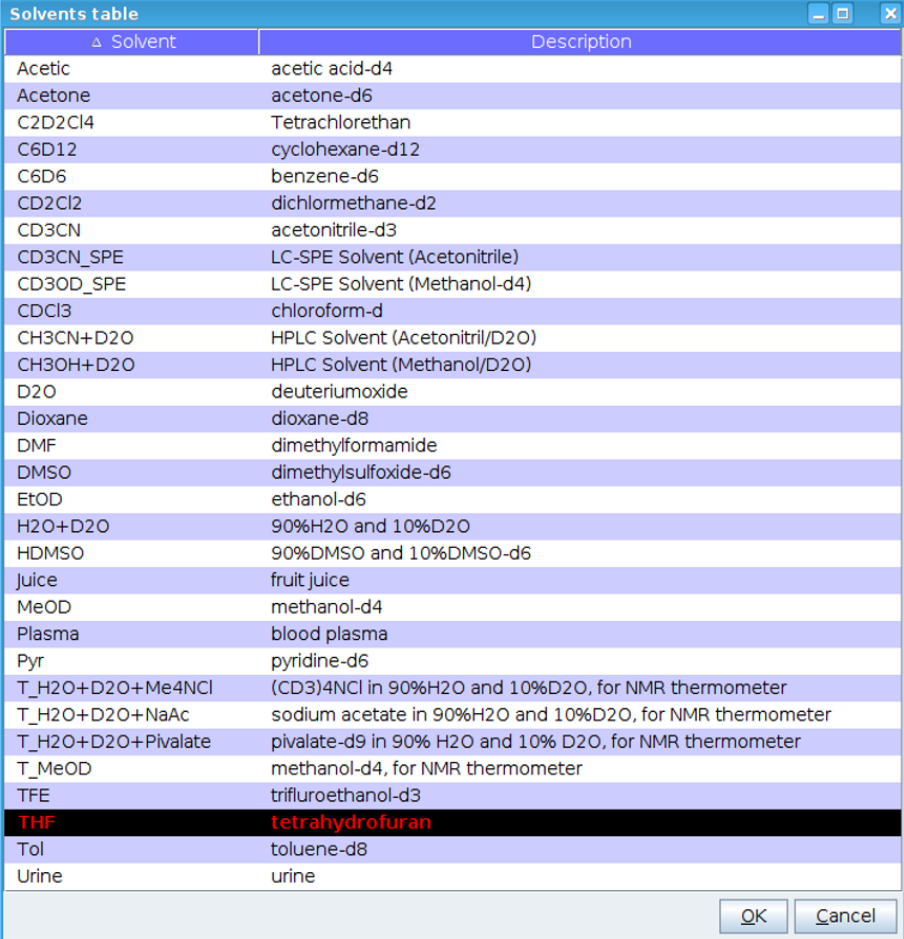
- > first check whether there already is a sample inside
- > enter command **ej** in the command line to activate air stream
- > wait until previous sample is ejected (if necessary)
- > put down sample onto air stream and enter command **ij**
- > after ejection of your own sample, turn off the air stream with **ij**, even if you don't inject a new sample



Young NMR tubes are heavier than conventional tubes and sometimes take longer to eject. If the sample eject does not work and you are sure that there still is a sample inside, you can increase the air flow carefully stepwise to max. 1200 lph. To achieve this, double-click on sample temperature and change the target gas flow. Please reset the air flow to the initial value afterwards (usually 400 to 600 lph). If you are uncertain about anything, do not hesitate to ask the technicians!

Sample lock

- > field frequency locking is performed on the deuterium signal of the solvent and is required for gradient shimming and to counteract the magnetic field drift for longer measurements
- > enter command **lock** into the command line, a table with all available solvents opens, select your solvent and press ok
- > alternatively, enter command **lock *solvent name*** and press enter (e.g. **lock thf** for locking on THF-d₈, for names of solvents as saved on the device enter **lock** or **edlock** to open solvent table, entry in command line is not case sensitive)
- > locking usually does not take longer than 1 min
- > repeat for every new sample with a different solvent than the previous one

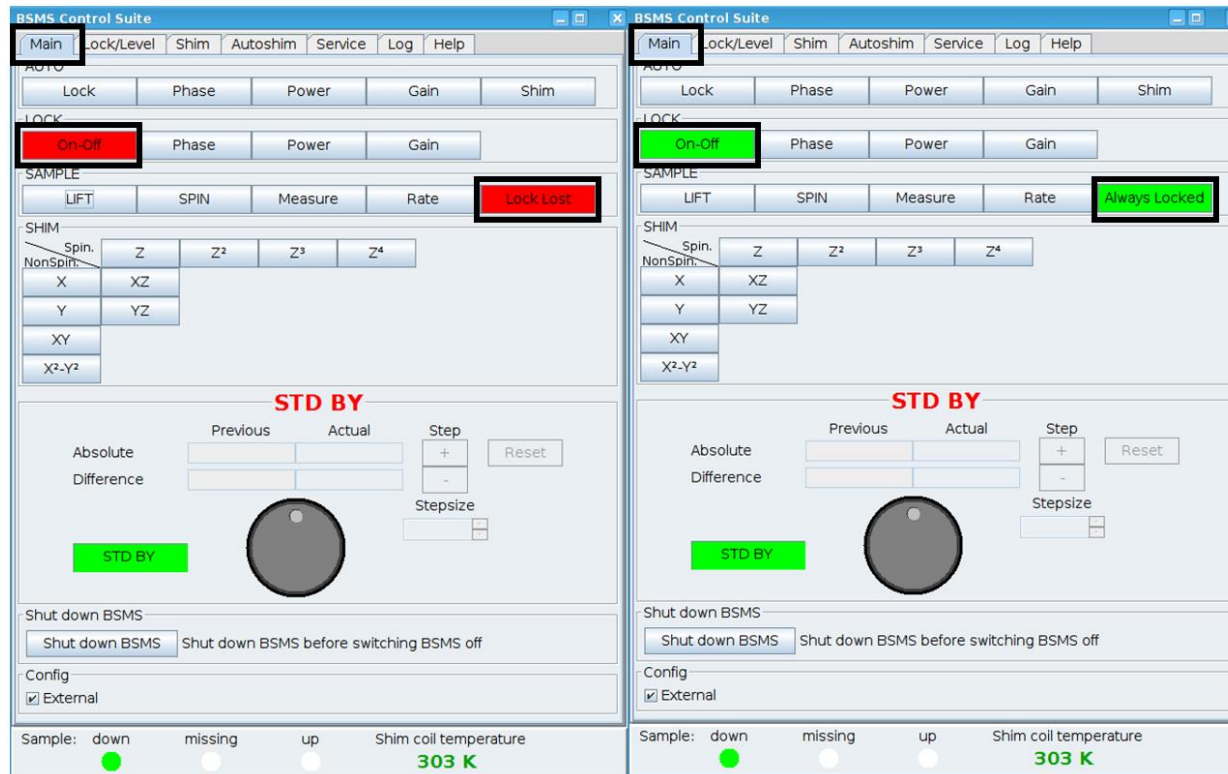


Δ Solvent	Description
Acetic	acetic acid-d4
Acetone	acetone-d6
C2D2Cl4	Tetrachlorethan
C6D12	cyclohexane-d12
C6D6	benzene-d6
CD2Cl2	dichlormethane-d2
CD3CN	acetonitrile-d3
CD3CN_SPE	LC-SPE Solvent (Acetonitrile)
CD3OD_SPE	LC-SPE Solvent (Methanol-d4)
CDCl3	chloroform-d
CH3CN+D2O	HPLC Solvent (Acetonitril/D2O)
CH3OH+D2O	HPLC Solvent (Methanol/D2O)
D2O	deuteriumoxide
Dioxane	dioxane-d8
DMF	dimethylformamide
DMSO	dimethylsulfoxide-d6
EtOD	ethanol-d6
H2O+D2O	90%H2O and 10%D2O
HDMSO	90%DMSO and 10%DMSO-d6
Juice	fruit juice
MeOD	methanol-d4
Plasma	blood plasma
Pyr	pyridine-d6
T_H2O+D2O+Me4NCl	(CD3)4NCl in 90%H2O and 10%D2O, for NMR thermometer
T_H2O+D2O+NaAc	sodium acetate in 90%H2O and 10%D2O, for NMR thermometer
T_H2O+D2O+Pivalate	pivalate-d9 in 90% H2O and 10% D2O, for NMR thermometer
T_MeOD	methanol-d4, for NMR thermometer
TFE	trifluoroethanol-d3
THF	tetrahydrofuran
Tol	toluene-d8
Urine	urine

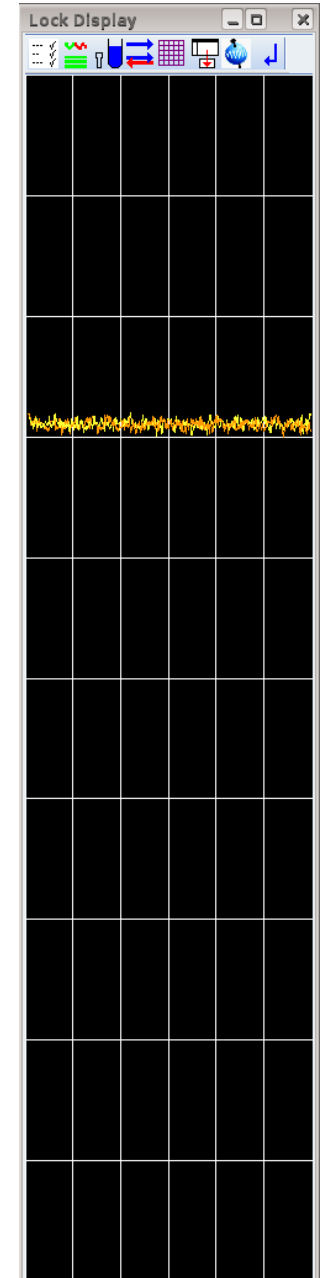
Sample lock

How do I know whether my sample is locked?

- > open BSMS window (double-click on sample button)
- > check the color of the Lock On-Off button in the main menu (red = lock off, green = lock on)



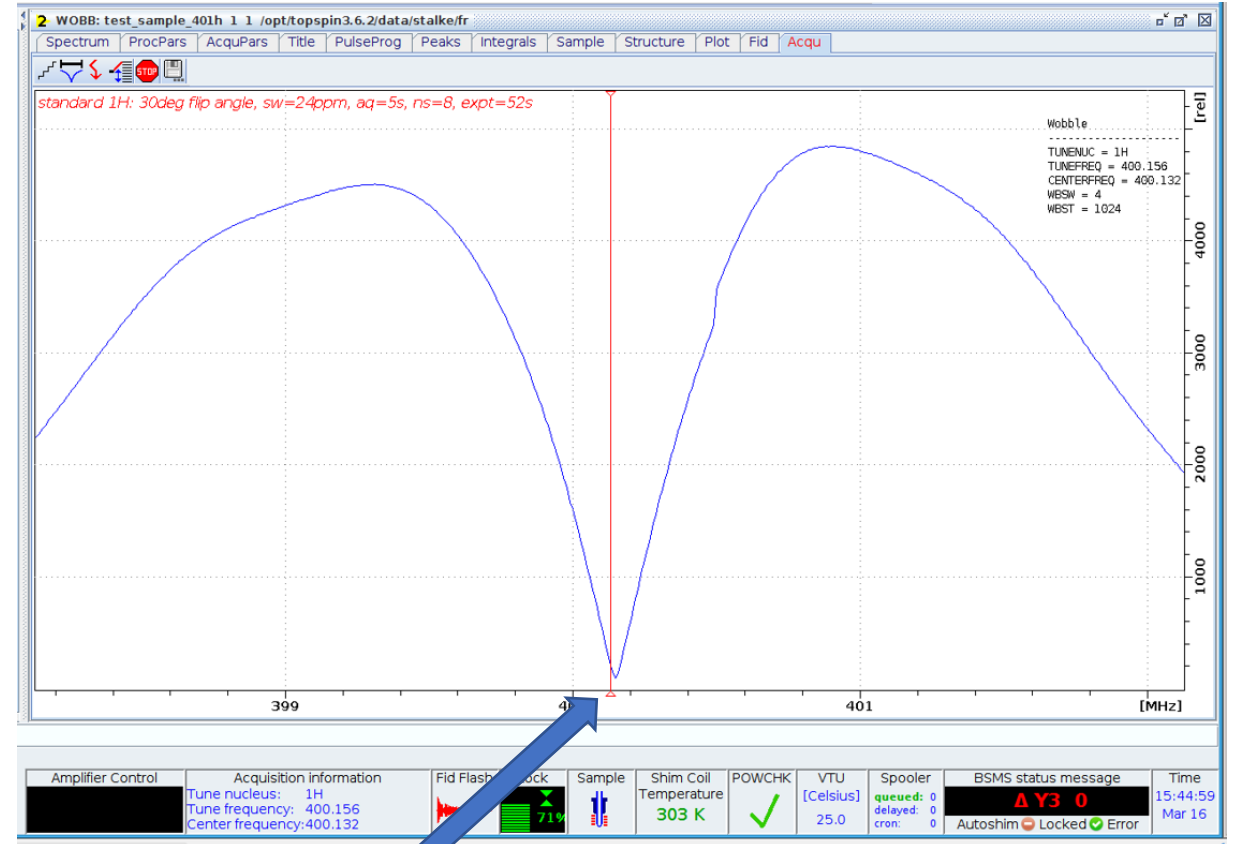
Lock display (usually on the right side of the screen)



If the lock display should be closed for some reasons, enter **lockdisp** into the command line

Tuning and matching the probe

- > adjustment of the probe to the correct radiofrequency (e.g. 400 MHz for ^1H , 100 MHz for ^{13}C etc.)
- > open data set which you would like to use, then enter command **atma**
- > automatic tuning and matching will be performed for the nuclei contained in the open data set (one for non-decoupled 1D experiments, two for decoupled 1D and 2D experiments)
- > **atma** usually takes less than 1 min
- > repeat **atma** for each experiment containing a new nucleus



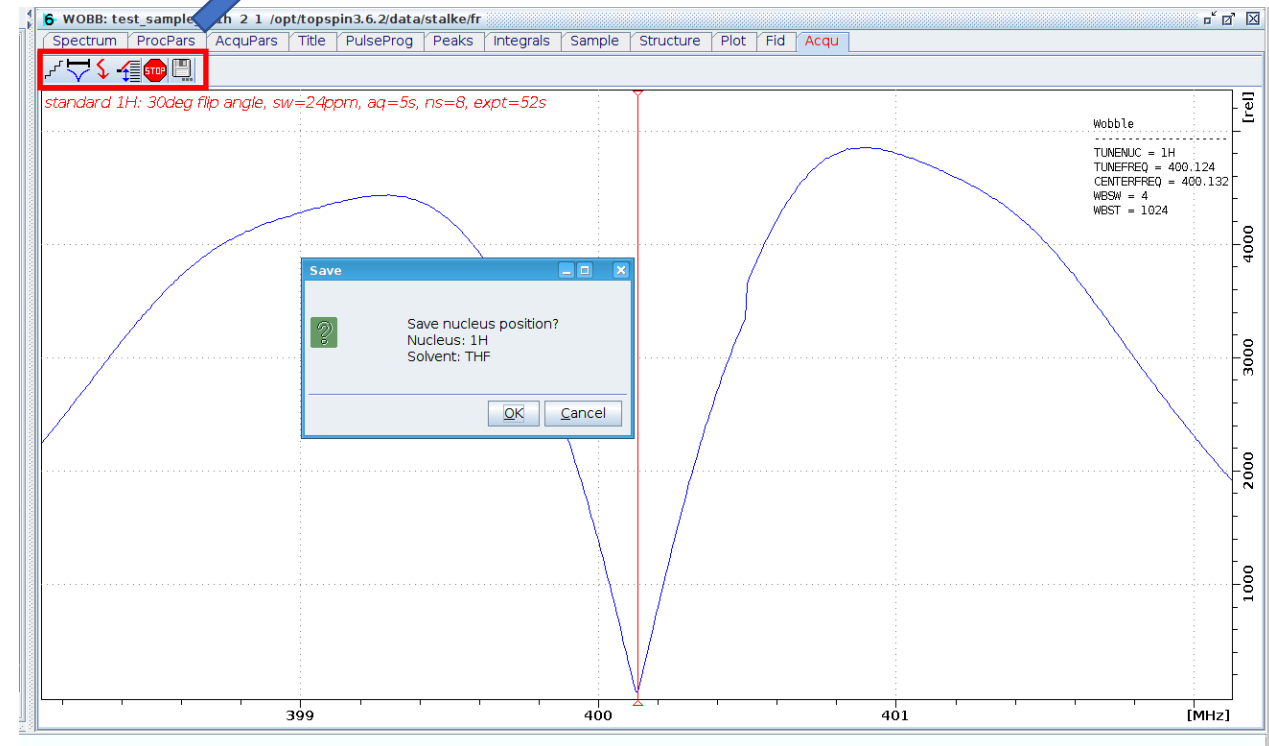
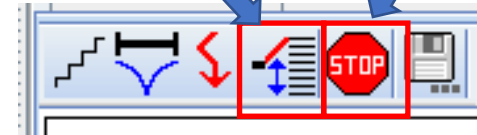
The dip is supposed to be on the red line marking the resonance frequency of the tuning nucleus and as deep as possible

Tuning and matching the probe

- > tuning and matching can also be performed manually using the command **atmm**
- > manually adjust dip by clicking on the respective buttond on the atma control panel that automatically opens when executing **atmm**
- > tuning moves the dip left and right, matching up and down
- > **atmm** is required especially for the first time measuring a certain nucleus

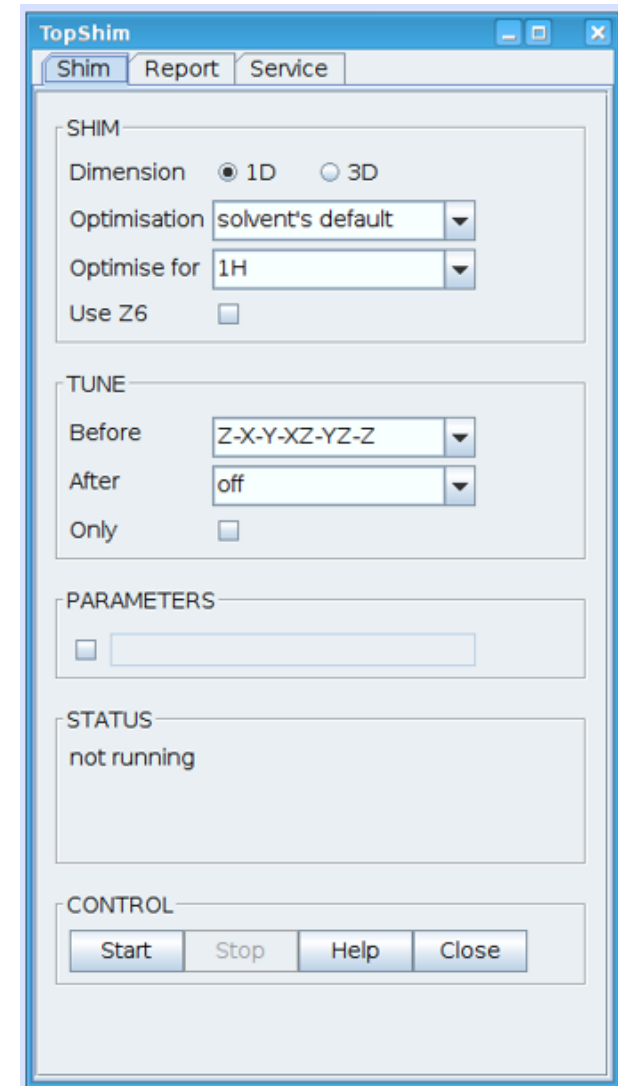
Switch to 2nd nucleus
(if available)

Exit atmm window



Gradient shimming

- > shimming is performed to increase the homogeneity of the magnetic field at the position of the sample
- > additional small magnetic fields into different directions are induced to correct residual inhomogeneities
- > fastest and easiest way to shim the sample is via the use of automatic gradient shimming (only works when the sample is locked!)
- > enter command **tg** into the command line and the TopShim window opens
- > press start button to start shimming process (usually takes approx. 2-5 min)
- > alternatively, the command **topshim** can be used (only gradient shimming will be performed without opening the TopShim window)



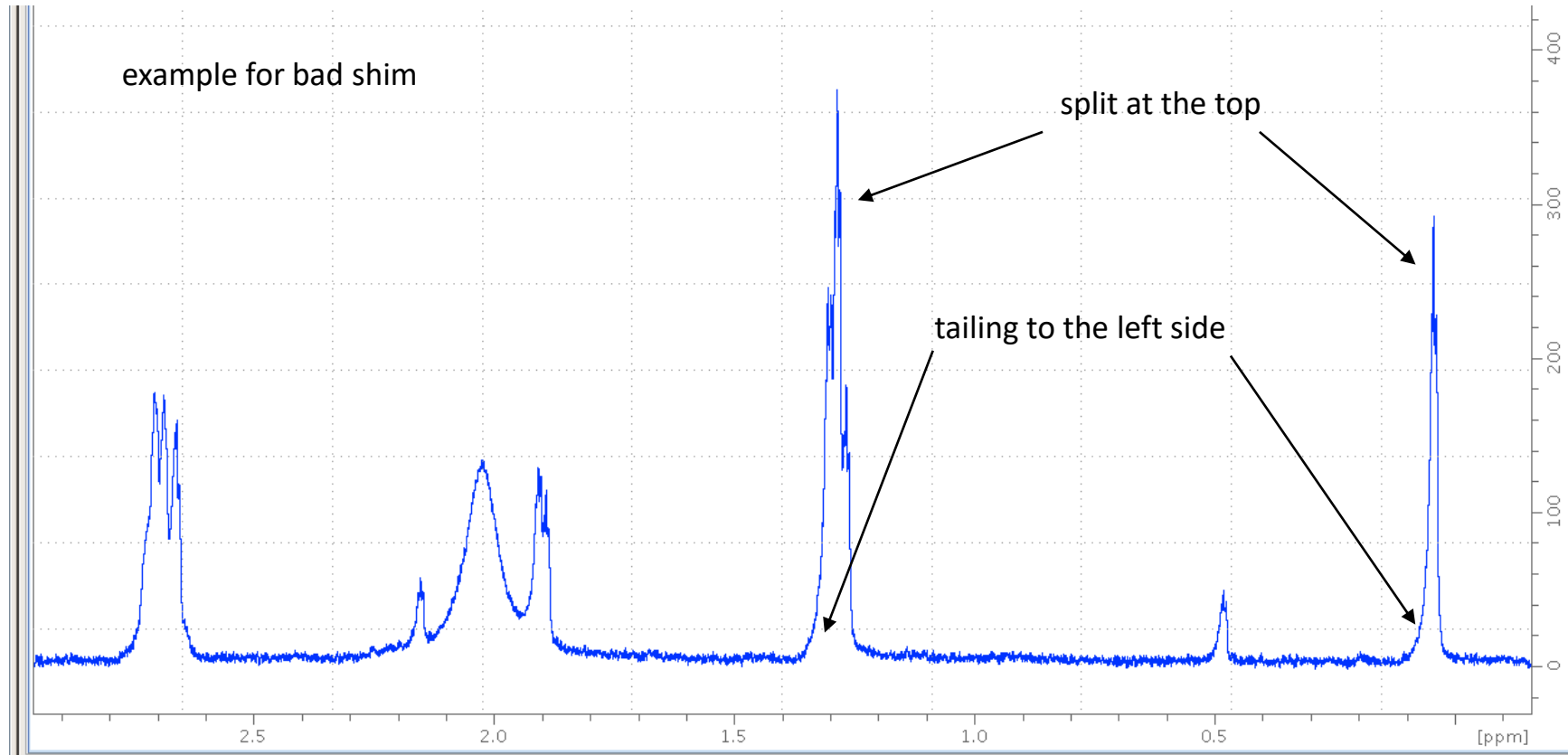
Gradient shimming

- > when shimming is successfully completed, the results can be viewed in the TopShim window under the tab „Report“
- > the final B0 stdDev indicates how well the automatic shim performed
- > the smaller the value, the more homogeneous is the magnetic field
- > for standard organic samples, the value should be lower than 0.4 Hz

```
TopShim
Shim | Report | Service
1D SHIMMING
Parameters:
maximum order = 5
probe = Z116098_0825
solvent = THF
shim nucleus = 2H
nucleus optimised for = 1H
o1p (from lock) = 1.73 ppm
selective excitation width = 1.0 ppm
optimisation parameters = ls
linewidth 1H = 0.10 Hz
envelope shape / strictness = 2.00 / 200
Results:
tune lock level increase = 1.0
initial B0 stdDev = 14.58 Hz
sample size = 2.05 cm, position = 0.00 cm
final B0 stdDev = 0.21 Hz > improvement = 68.6
envelope width = 0.46 Hz
shim changes:
Z          -37
Z2         +556
Z3         -394
Z4         +3625
Z5         +923
X          -23
XZ         +15
Y          -69
YZ         +15
duration = 1 min 35 sec
completed successfully
finished Tue Mar 16 15:37:30 2021
```

Bad shim

- > when signal distortions are observed in the spectrum, this might indicate a bad shim
- > in case of a bad shim, all signals in the spectrum are equally affected by the same distortions
- > when signals are asymmetrically distorted, an inhomogeneous sample might be the reason



What to do when the shim is bad?

1) Adjusting the shim by hand

-> open BSMS window (double-click on sample button) and go to the shim menu
-> click on the respective shim coil and turn the mouse wheel to change its value while watching the lock signal

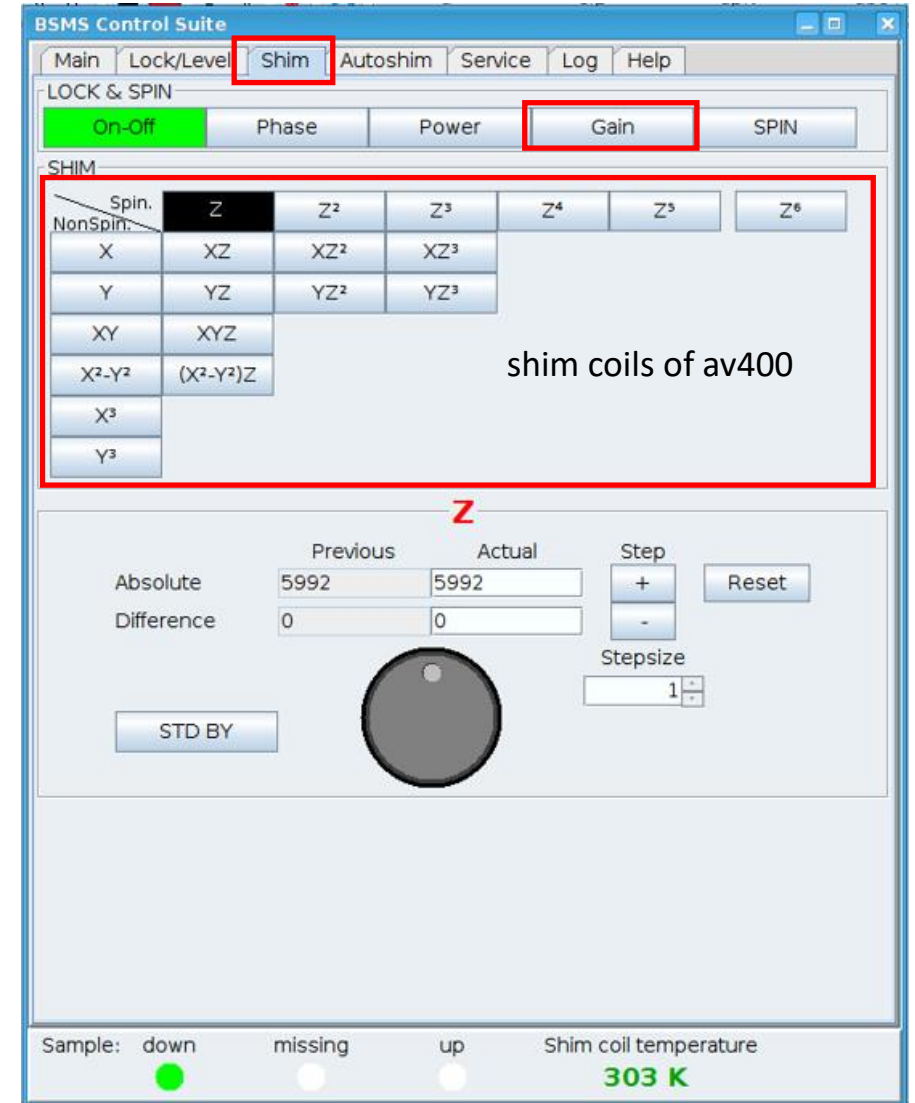
-> lock signal goes up: shim gets better, lock signal goes down: shim gets worse
-> when lock signal reaches top of lock display, click on Gain and turn the mouse wheel to adjust height of lock signal

-> usually, the adjustment of the coils Z, Z², Z³, X, XZ, XZ², Y, YZ, YZ² is fully sufficient

-> proceed like this:

- Z -> Z² -> Z³
- X -> XZ -> Z
- Y -> YZ -> Z
- X -> XZ² -> Z²
- Y -> YZ² -> Z²

-> if you see a major change after optimizing e.g. Z², go back to Z and start the row anew



What to do when the shim is bad?

2) Read a shim file performed on a calibration sample

- > enter command **rsh** into the command line, a table with saved shim files opens
- > select newest shim file called **cdcl3** or **Sucrose_3D** and click on read (make sure it was saved on the correct probe, usually, PABBI is inside av400)
- > execute TopShim again (either via **tg** or **topshim**)

File name	Date	ID	Info
cdcl3_13.02.25	Thu, 13 February 2025 10:38:19	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
s1	Thu, 6 February 2025 10:38:44	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
Sucrose_3D_15.01.25	Wed, 15 January 2025 10:44:08	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
test	Fri, 29 November 2024 11:18:13	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
mm-thf	Fri, 11 October 2024 13:06:04	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
lineshape_rot	Tue, 24 September 2024 11:58:25	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
TSR_RT	Wed, 4 September 2024 16:59:10	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
TSR_2_-85	Sun, 18 August 2024 12:18:37	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
mm	Mon, 17 June 2024 19:25:04	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
Sucrose_3D_11.04.24	Thu, 11 April 2024 12:49:13	3	5 mm PABBO BB/19F-1H/D Z-GRD Z116098/0825
cdcl3_BBI	Tue, 9 April 2024 12:33:13	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
lineshape_BBI	Tue, 9 April 2024 12:24:15	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
sucrose_3D	Tue, 9 April 2024 12:02:21	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
lineshape_BBO	Wed, 3 April 2024 10:59:54	4	5 mm PABBO BB/19F-1H/D Z-GRD Z108618/0095
ls_nospinning	Tue, 19 March 2024 11:34:23	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
Sucrose_3D	Tue, 19 March 2024 11:26:05	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
NaHMDS_thf	Fri, 15 December 2023 09:57:52	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
Bodipy	Tue, 7 November 2023 13:00:44	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
SnCl2_Stamm	Tue, 24 October 2023 14:30:08	1	5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
simplexend	Tue, 26 September 2023 10:55:21	4	5 mm PABBO BB/19F-1H/D Z-GRD Z108618/0095
tune_end	Tue, 26 September 2023 10:55:21	4	5 mm PABBO BB/19F-1H/D Z-GRD Z108618/0095
tune_beg	Tue, 26 September 2023 10:44:49	4	5 mm PABBO BB/19F-1H/D Z-GRD Z108618/0095
sh00	Tue, 26 September 2023 10:44:49	4	5 mm PABBO BB/19F-1H/D Z-GRD Z108618/0095

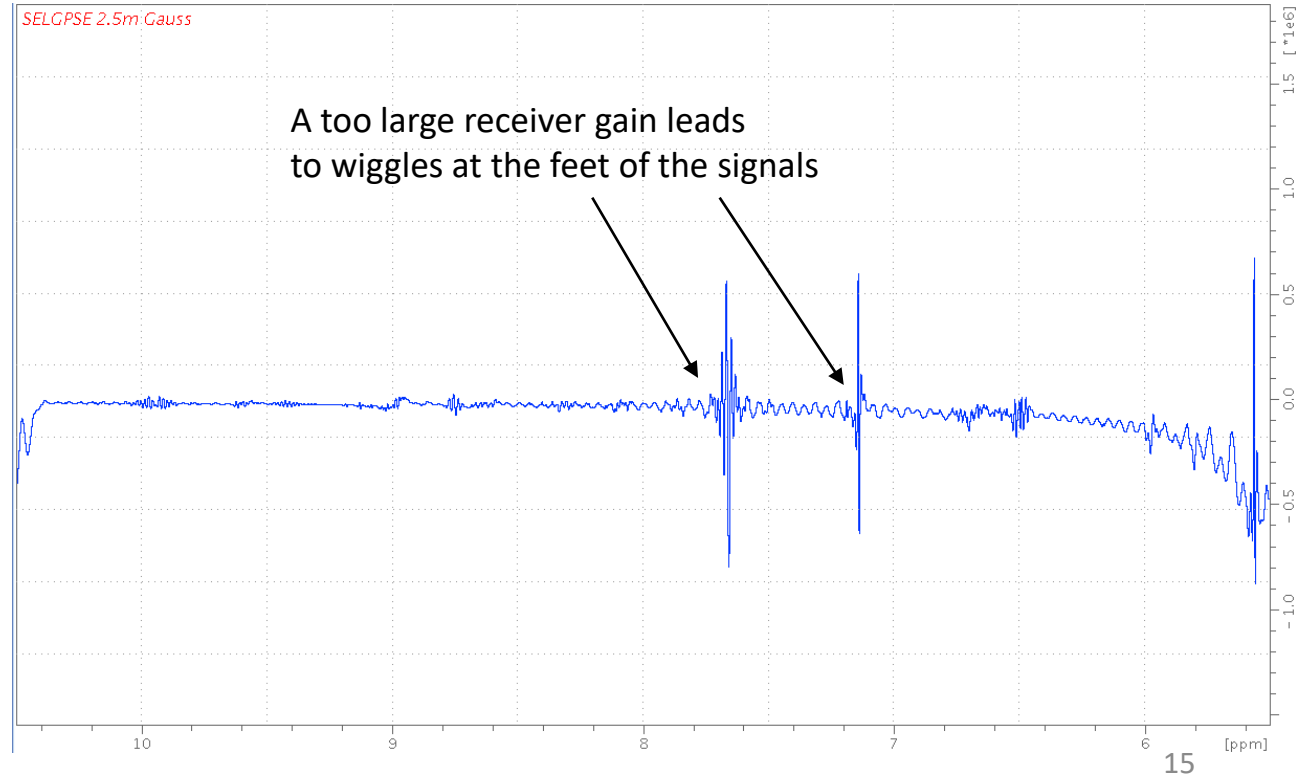
Set also lock parameters

Read Write View Delete Close

Adjusting the receiver gain

- > signal emitted from the sample is enhanced by receiver gain
- > receiver gain is automatically determined by the spectrometer when entering the command **rga**
- > alternatively, a certain value can be chosen by entering **rg**
- > the receiver gain is small for concentrated samples and high for dilute samples and varies depending on the channel (^1H , BB)
- > **rga** usually takes only a few seconds
- > repeat for each new measurement

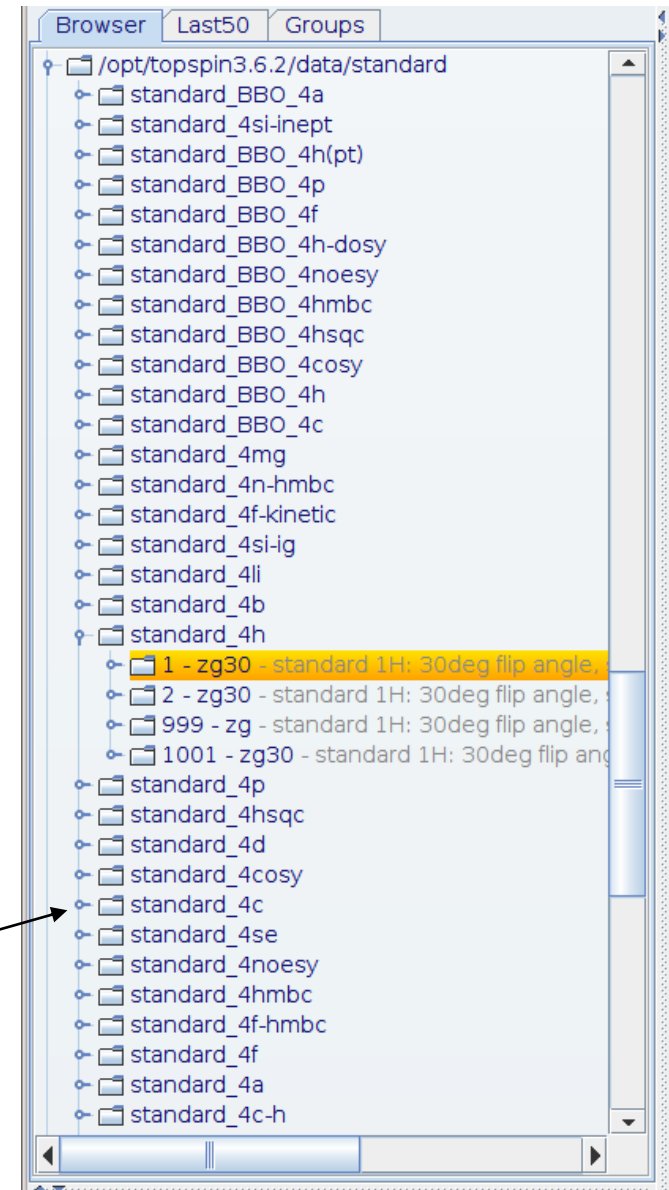
If you enter a command and the previous command is still running, the new one will be automatically added to the queue!



Creating a new dataset

- > easiest way to create a new dataset is via copying an already existing dataset
- > either copy one of your own experiments or take one from the standard file containing standard datasets with standard parameters
- > open the dataset you want to copy and enter command **new**
- > enter name of file, experiment number, directory of your research group and title (if you wish to) and press ok
- > **do not change the parameters of the standard datasets!**

The standard file contains a variation of different 1D and 2D experiments for several nuclei, e.g. standard_4c is a standard ^{13}C dataset



Creating a new dataset

Create New Dataset - new

Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type. For multi-receiver experiments several datasets are created. Please define the number of receivers in the Advanced.

Dataset

NAME: test_sample_401h

EXPNO: 1

Directory: /opt/topspin3.6.2/data/stalke/fr

Open in new window

Parameters

Use current parameters

Read parameterset

Set solvent: THF

Additional action

No additional action

Execute getprosol

Keep parameters: P 1, O1, PLW 1

Advanced

Number of datasets (receivers): 1

Title: standard 1H: 30deg flip angle, sw=24ppm, aq=5s, ns=8, expt=52s

OK Cancel More Info... Help

name of file (e.g. number of sample, acronym_4h,... , do not use spaces or special characters like ä, ö, ü)

experiment number (not always continued automatically)

directory into which the new dataset is copied (don't forget to change if you copy a standard dataset!)

enter title of your experiment e.g. with experimental details here (can be changed afterwards)

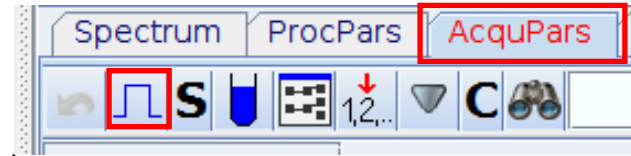
Optimizing acquisition parameters (1D)

The screenshot shows the 'AcquPars' window with the following parameters:

Parameter	Value	Description
Experiment		
PULPROG	zg30	Current pulse program
AQ_mod	DQD	Acquisition mode
TD	98304	Size of fid
DS	2	Number of dummy scans
NS	8	Number of scans
TD0	1	Loop count for 'td0'
Width		
SW [ppm]	24.0305	Spectral width
SWH [Hz]	9615.385	Spectral width
AQ [sec]	5.1118078	Acquisition time
FIDRES [Hz]	0.195625	Fid resolution
FW [Hz]	4032000.000	Filter width
Receiver		
RG	71.8	Receiver gain
DW [μsec]	52.000	Dwell time
DWOV [μsec]	0.025	Oversampling dwell time
DECIM	2080	Decimation rate of digital filter
DSPFIRM	sharp(standard)	DSP firmware filter
DIGTYP	DRU	Digitizer type
DIGMOD	digital	Digitization mode
DR	22	Digitizer resolution
DDR	10	Digital digitizer resolution

Large range of parameters that can be changed, you can ignore most of them

Optimizing acquisition parameters (1D)



Parameter	Value	Description
PULPROG	zg30	Pulse program for acquisition
TD	98304	Time domain size
SWH [Hz, ppm]	9615.38	Sweep width
AQ [sec]	5.1118078	Acquisition time
RG	71.8	Receiver gain
DW [µsec]	52.000	Dwell time
DE [µsec]	6.50	Pre-scan-delay
D1 [sec]	0.100000001	Relaxation delay; 1-5 * T1
DS	2	Number of dummy scans
NS	8	1 * n, total number of scans: NS * TD0
TD0	1	Number of averages in 1D
SFO1 [MHz]	400.1320007	Frequency of ch. 1
O1 [Hz, ppm]	2000.65	Frequency of ch. 1
NUC1	1H	Nucleus for channel 1
p0 [µsec]	3.37	p0=p1/3
P1 [µsec]	10.100	F1 channel - 90 degree high power pulse
PLW1 [W, dB]	27.733	F1 channel - power level for pulse (default)

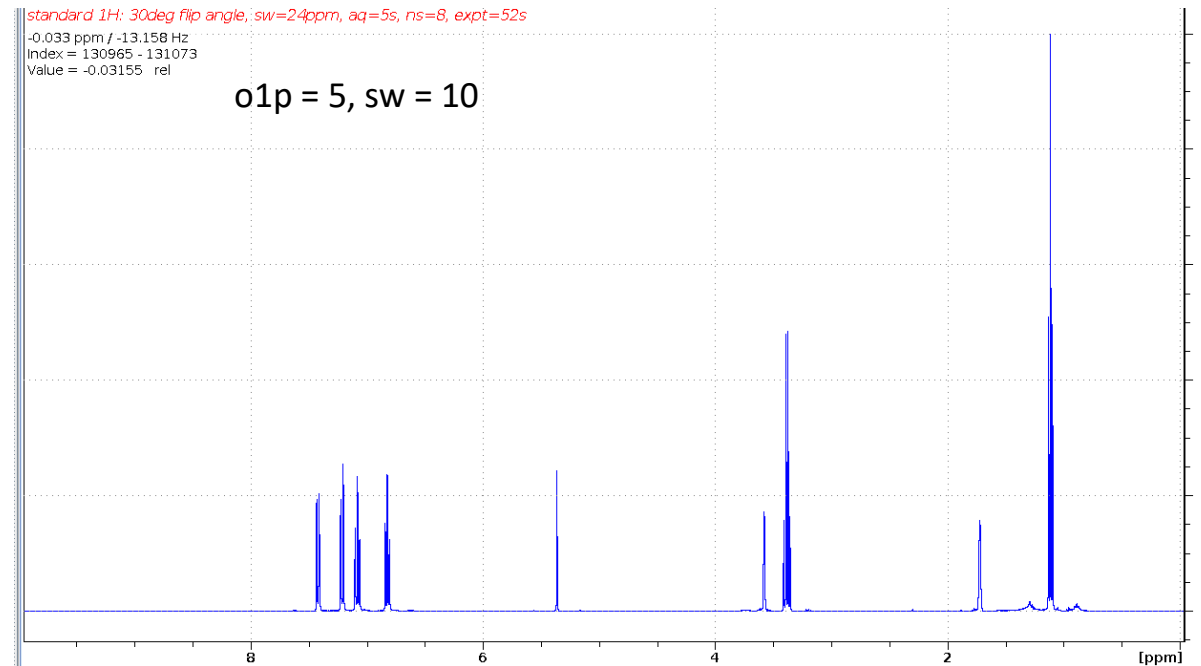
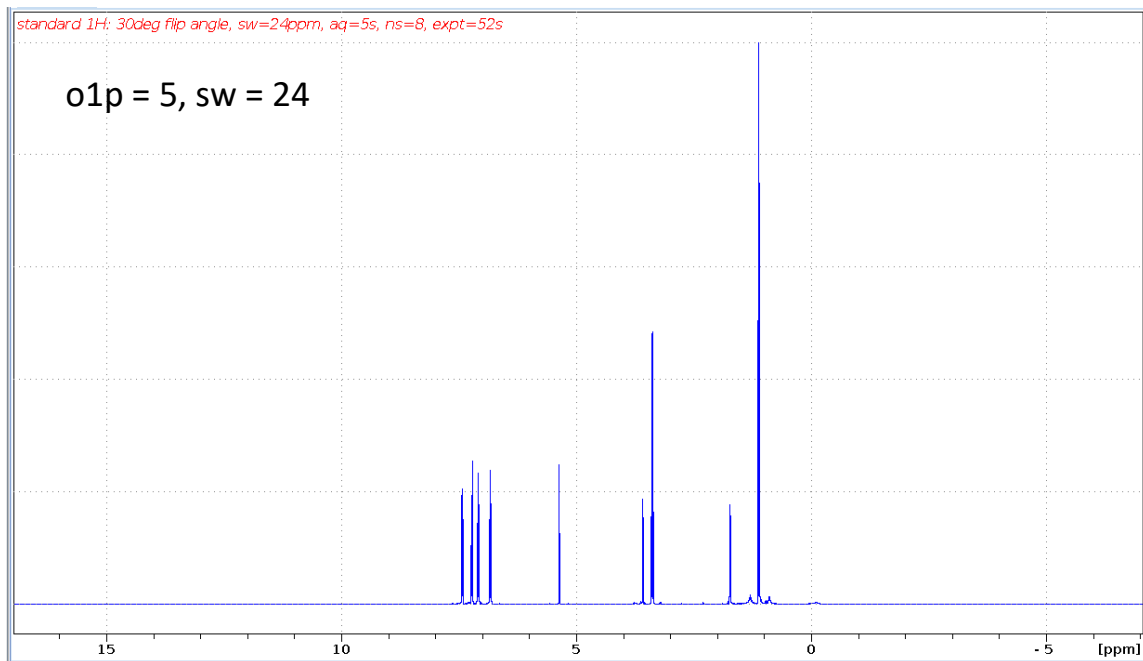
most important parameters

Parameters can be changed either in this screen or by entering the respective parameter into the command line, e.g. **o1p** for the transmitter frequency

Do not change the parameters in the standard datasets! Make a copy first!

Optimizing acquisition parameters (1D)

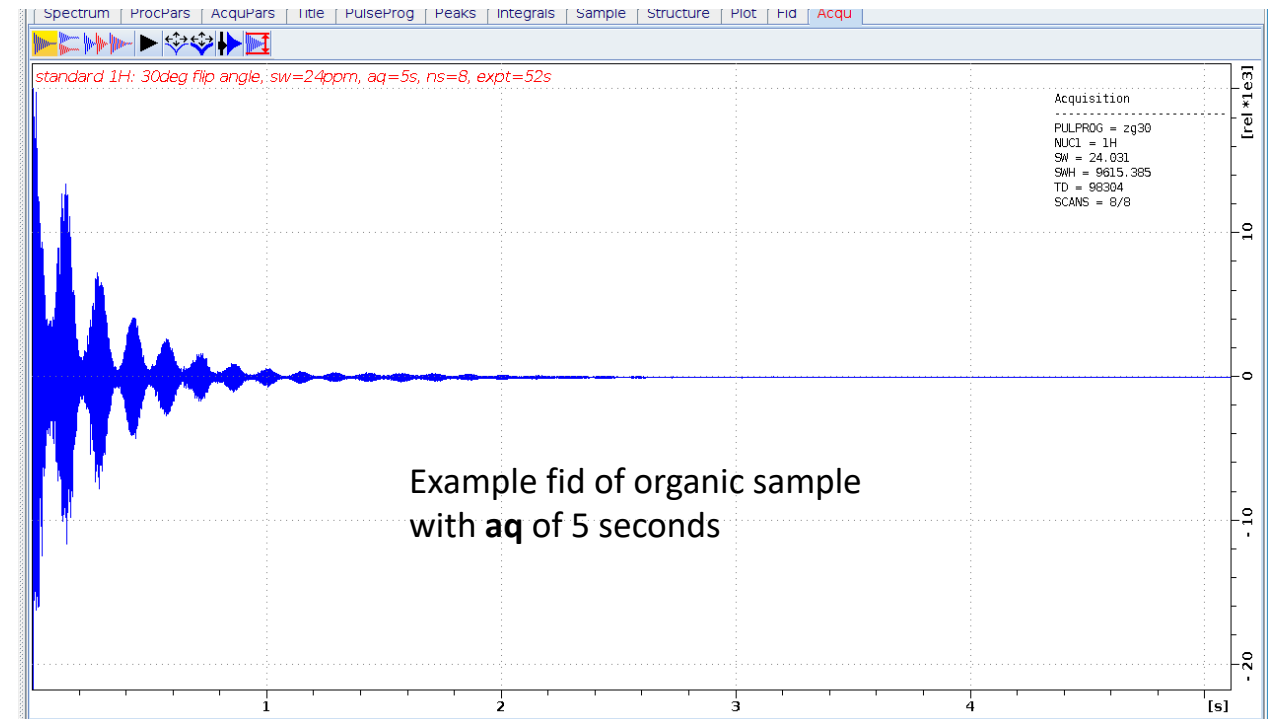
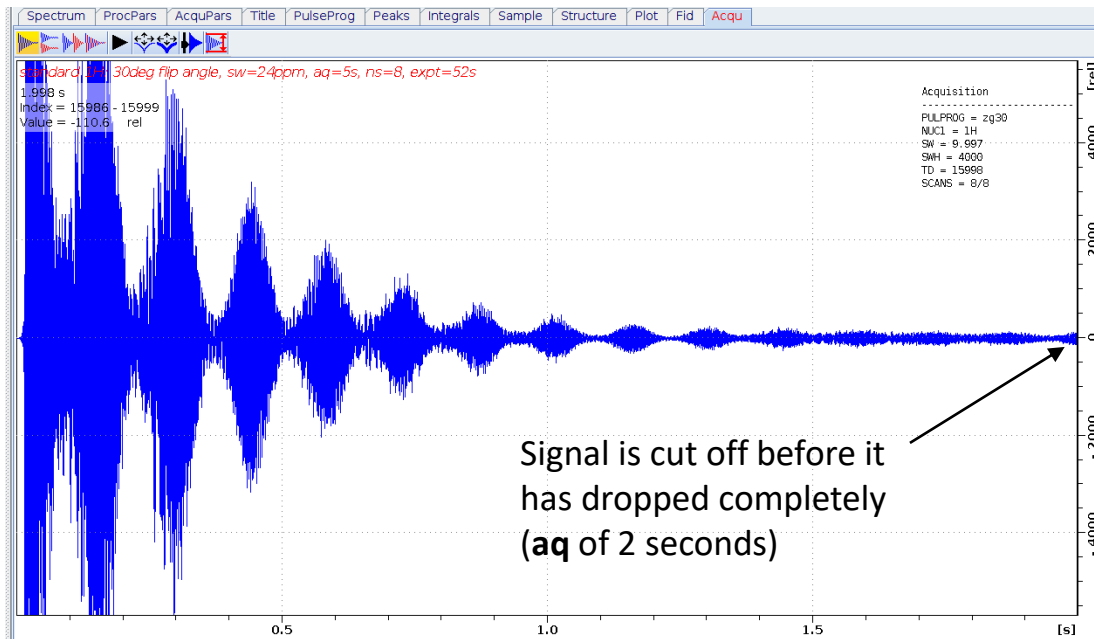
- > transmitter frequency (**o1p**, center of the spectrum) and spectral width (**sw**, width of the spectrum)
- > total ppm-region that is recorded goes from $o1p - \frac{1}{2} sw$ to $o1p + \frac{1}{2} sw$
- > can be changed either in the acquisition parameters or by typing **o1p** and **sw** into the command line



Optimizing acquisition parameters (1D)

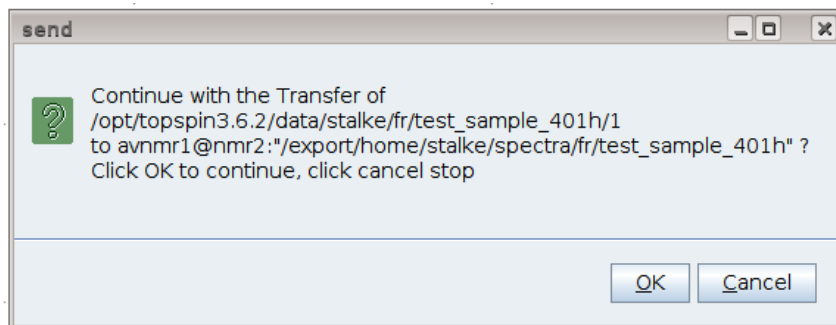
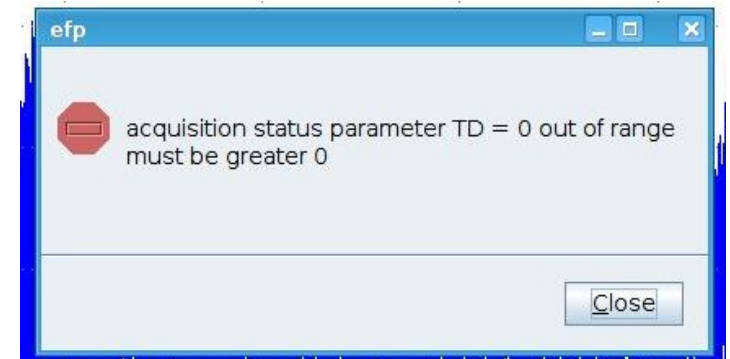
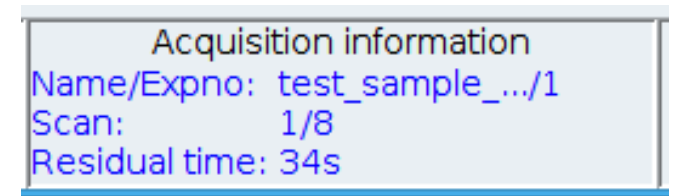
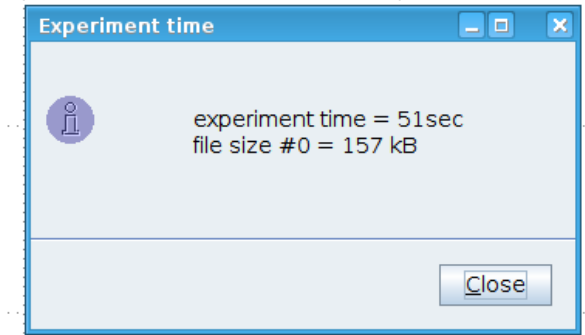
- > acquisition time **aq** is the time in seconds in which the fid is recorded
- > is the **aq** too long, much noise will be recorded, leading to lower signal intensity
- > is the **aq** too short, the fid will be cut off, leading to signal distortions
- > for standard organic samples, an **aq** of 5 seconds for ^1H experiments is a good compromise

sw and **aq** are connected!
When the **sw** is halved, the **aq** doubles, so always check the **aq** after changing the **sw**!



Starting & processing the measurement (1D)

- > use command **expt** to see how long the experiment will take (dependent on **ns**, **aq**, ...)
- > command **zg** starts the acquisition (valid for all 1D & 2D experiments except for DOSY)
- > acquisition information shows information about the currently running measurement
- > 1D fourier transformation is executed using command **efp**
- > 1D automatic phase correction (**apk**) and baseline correction (**abs n**, here the space is important) can be used
- > fourier transformation can also be performed while the measurement is still running
- > if this error occurs then, first use command **tr** to store already recorded scans to the disk before executing **efp** again
- > when you want to stop the acquisition, use either **stop** (measurement stops immediately, fid is not saved) or **halt** (stops after currently running scan, fid is saved)
- > send your data to your group server via command **send**



Optimizing acquisition parameters (2D)

standard_4cosy 1 1 /opt/topspin3.6.2/data/nmr/standard

Probe: PA BBO 400S1 BBF-H-D-05 Z PLUS

Parameter	F2	F1	Description
PULPROG	cosygppqf		Current pulse program
AQ_mod	DQD		Acquisition mode
FnTYPE	traditional(planes)		nD acquisition mode for 3D etc.
FnMODE	QF		Acquisition mode for 2D, 3D etc.
TD	1536	256	Size of fid
DS	16		Number of dummy scans
NS	1		Number of scans
TD0	1		Loop count for 'td0'
TDav	0		Average loop counter for nD experiments
Width			
SW [ppm]	9.9967	9.9967	Spectral width
SWH [Hz]	4000.000	4000.000	Spectral width
IN_F [μsec]		250.00	Increment for delay
AQ [sec]	0.1920000	0.0320000	Acquisition time
FIDRES [Hz]	5.208333	31.250000	Fid resolution
FW [Hz]	4032000.000		Filter width

Homonuclear (e.g. COSY)

standard_4hsqc 1 1 /opt/topspin3.6.2/data/nmr/standard

Probe: PA BBO 400S1 BBF-H-D-05 Z PLUS

Parameter	F2	F1	Description
PULPROG	hsqcedetgppsp.3		Current pulse program
AQ_mod	DQD		Acquisition mode
FnTYPE	traditional(planes)		nD acquisition mode for 3D etc.
FnMODE		Echo-Antiecho	Acquisition mode for 2D, 3D etc.
TD	1536	256	Size of fid
DS	16		Number of dummy scans
NS	2		Number of scans
TD0	1		Loop count for 'td0'
TDav	0		Average loop counter for nD experiments
Width			
SW [ppm]	9.9967	160.0040	Spectral width
SWH [Hz]	4000.000	16099.653	Spectral width
IN_F [μsec]		62.11	Increment for delay
AQ [sec]	0.1920000	0.0079505	Acquisition time
FIDRES [Hz]	5.208333	125.778542	Fid resolution
FW [Hz]	4032000.000		Filter width

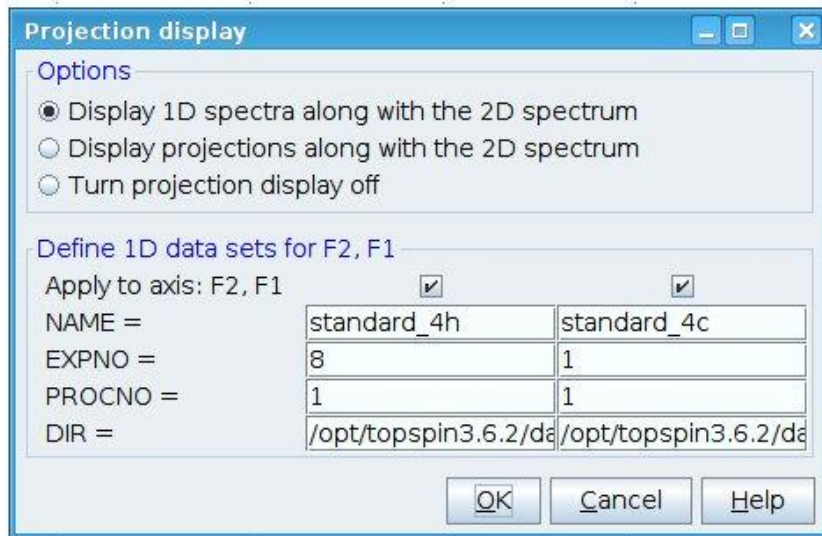
Heteronuclear (e.g. HSQC)

-> parameter optimization for 2D experiments very similar to 1D experiments, only there are often two values (F2 and F1)

-> F2 direct dimension (horizontal), F1 indirect dimension (vertical)

Processing the measurement (2D)

- > 2D fourier transformation works via command **xfb**
- > 2D baseline correction is executed either via **absb** (no space here) or separately correcting the respective dimensions via **abs1** and **abs2**
- > using command **projd**, you can project already measured 1D spectra as traces to the 2D spectrum




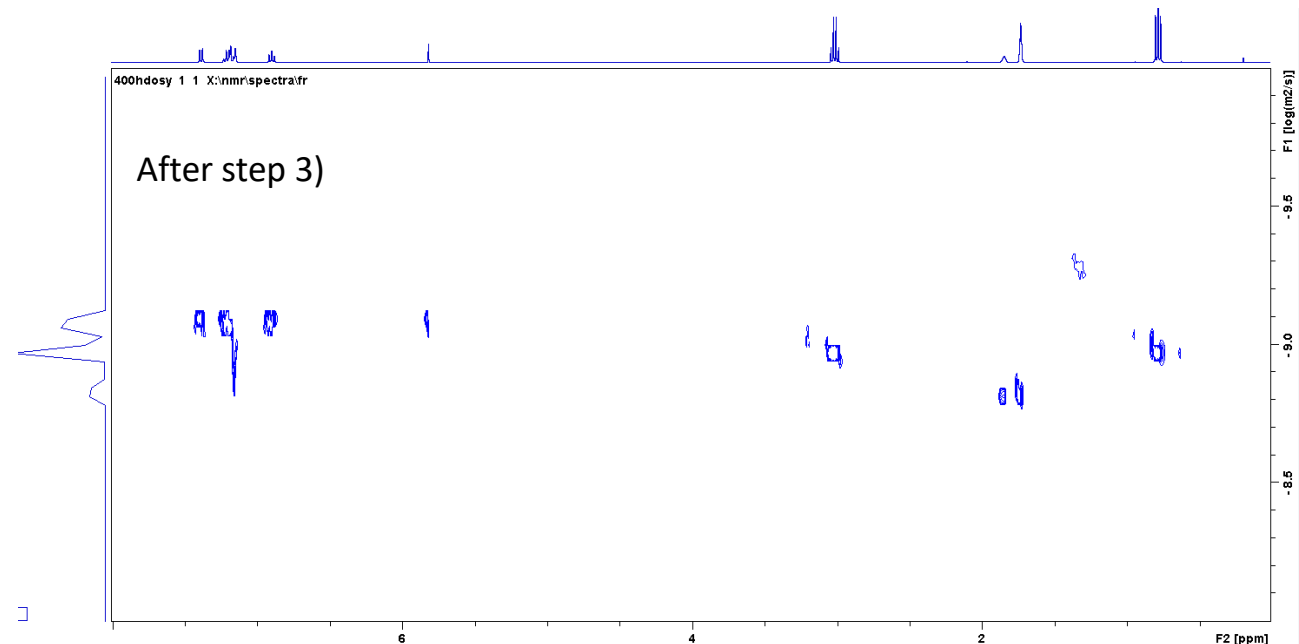
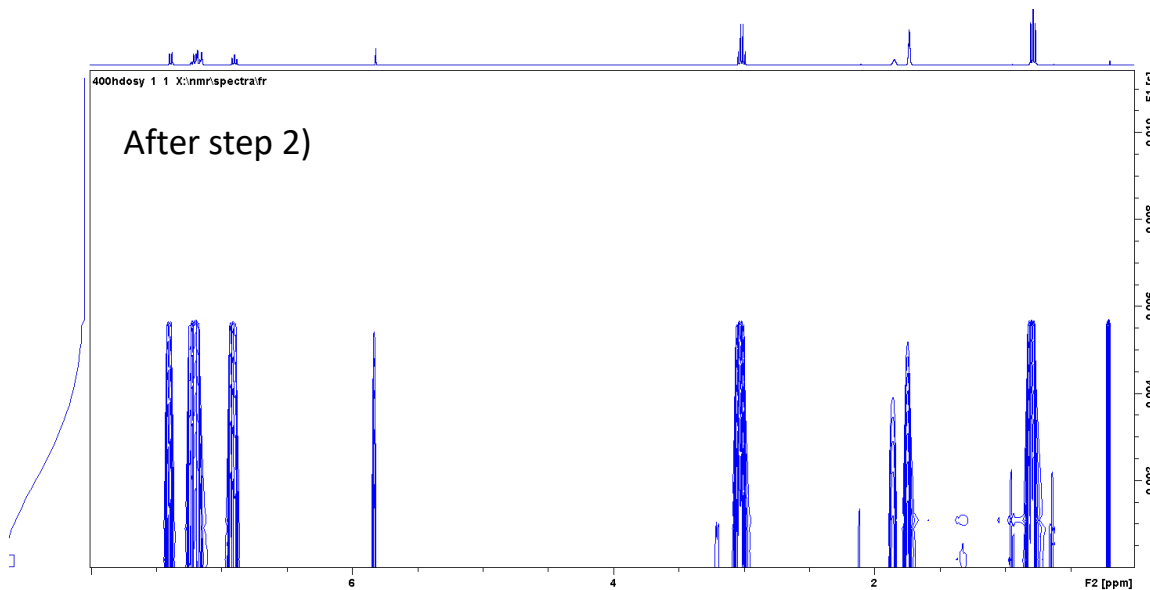
Measuring and processing DOSY

-> unlike all other experiments, DOSY is started via command **xau dosy**

-> a window opens, asking about gradient setup, just press enter until the measurement starts

-> processing DOSY requires multiple steps:

- 1) process a 1D FID via **efp** and do a phase correction using **.ph** (press  to transfer the correction to all FIDs)
- 2) enter command **to2d** to return to 2D view, then process the pseudo-2D experiment with **xf2** and **abs2** (baseline correction)
- 3) to create the diffusion dimension, enter command **setdiffparm** and afterwards **dosy2d** to obtain the typical DOSY spectrum



Variable temperature (VT) measurements

- > please make sure your chosen solvent is suitable for the temperature range you plan to measure
- > temperature is chosen and monitored via temperature control suite (double-click on probe temperature)
- > for temperatures **higher than 25 °C**, click on „set target temperature“ and enter desired value
- > the spectrometer will adjust the heater power by itself to reach the new target temperature
- > is the current temperature lower (higher) than the target temperature, the value is shown in blue (red)
- > the color of the value will change to green when the target temperature is reached and stable

Channel	Regulation State	Stability	Current Temperature	Target Temperature	Heater Power
1 bruker	Steady	Stable since 18:52:53 28 Apr 2025	25.1 °C	25.0 °C (-150.1 °C...149.9 °C)	0.3 % (max. 29.8 % of 196.1 W)

State	Gas Flow	Target Gas Flow	Standby Gas Flow
Steady	400 lph	400 lph	200 lph



Plan more time for your VT measurements than for measurements at room temperature! Your booked time should include enough time to reach your desired temperature and turn the spectrometer back to 25 °C afterwards!

Variable temperature (VT) measurements

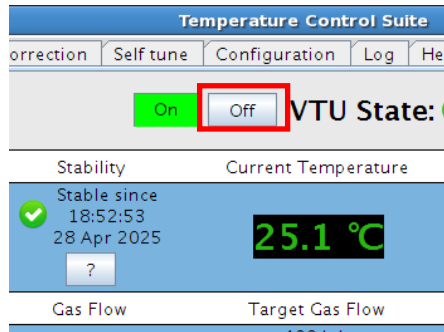
-> for temperatures **beneath 25 °C**, an external cooling with (liquid) nitrogen is necessary (N2 chiller)
-> cooling equipment ready to use next to av400

- 1) Dewar with liquid nitrogen (bring your own in case it's empty)
- 2) Nitrogen transfer line (connects dewar with probe)
- 3) Connection cable between console and chiller (for regulation)

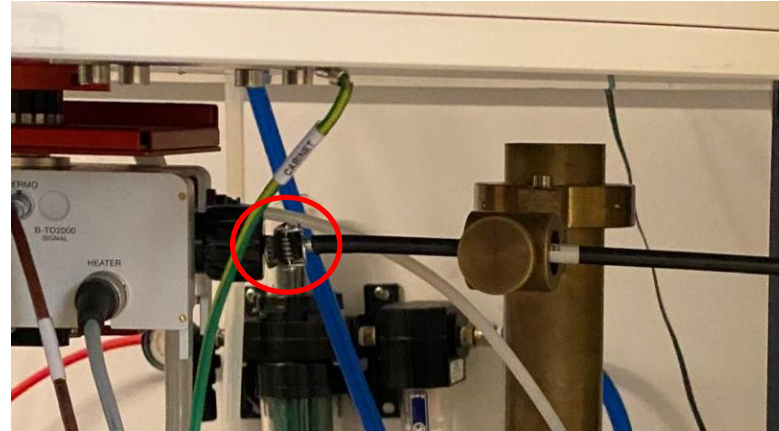


If it's your first time using the low temperature setup, let an experienced person show you how to install and use it. Don't use the setup on your own until you're 100% sure how to use it. If you are unsure about anything, do not hesitate to ask someone with experience!

Installing the N2 chiller



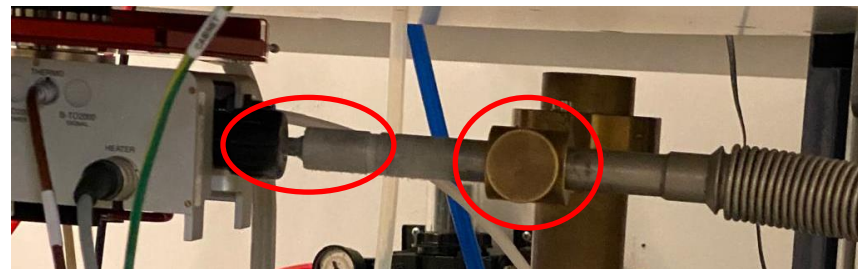
1) Open temperature control suite and press „off“



3) Remove the hose and the respective clamp regulating the air flow at the back of the probe



2) Insert the transfer line into the dewar and secure it with the clamp (don't forget the gasket)

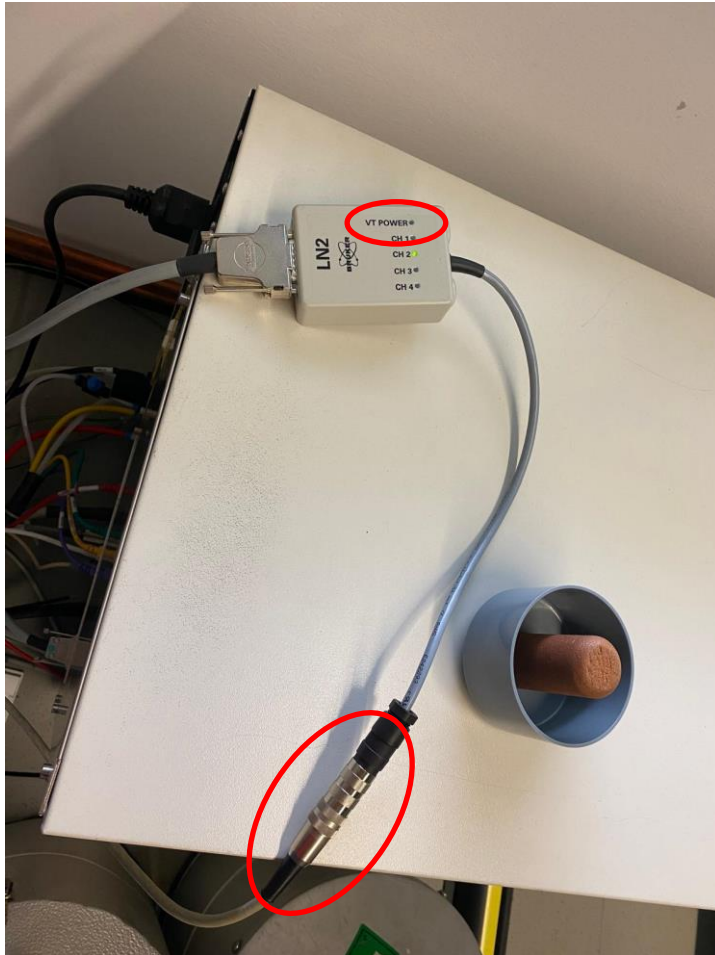


4) Put the hose of the transfer line through the bronze mount and secure it with the screw. It should sit tightly at the gas inlet to prevent leakage



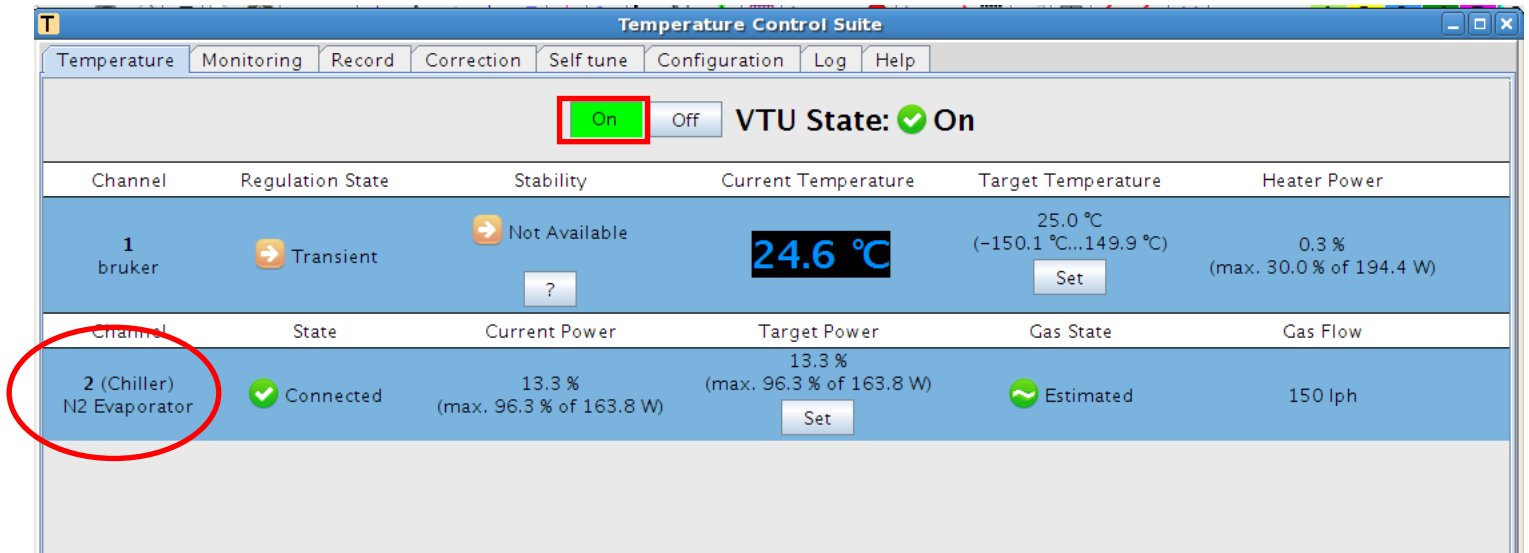
Please don't remove any equipment parts from the room and put them back to where you found them after use!

Installing the N2 chiller



5) Connect the cable of the transfer line with the port lying on the av400 console (no brute force!). The light behind „VT power“ should blink after a few seconds

6) Click on „on“ again. After successful installation, the N2 chiller should be shown in the temperature window



Low temperature measurements

- > low temperature measurements require a temperature correction
- > after installing N2 chiller, open temperature window and go to correction window
- > place tick and select available correction plot
- > click on „set“
- > the temperature window will now show a corrected (actual value) and a measured temperature value (as measured by the device)
- > when correction is active, just enter the desired temperature as target temperature
- > temperature correction works reasonably well between 0°C and -75°C
- > after going back to room temperature, just remove the tick again

Temperature Control Suite

Temperature Monitoring Record **Correction** Self tune Configuration Log Help

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: Low temperature
Probe: 5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002
Temperature range [°C]: 0.0--50.0
Slope: 0.794913
Offset: 60.892448
Comment: 0 to -50 degrees

Available correction settings

Name	Probe	Δ Temperature Rang...	Slope	Offset	Comment
Low	5 mm PABBI	0.0 - -50.0	0.794913	60.892448	0 to -50 degrees

Manual Edit **Set** Delete

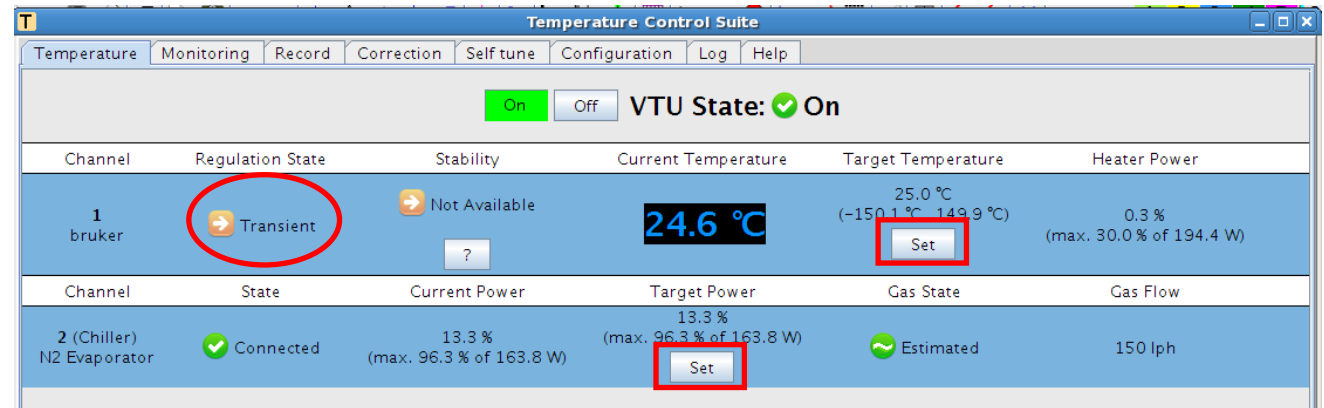
VTU: On Sample Temperature: **Corr. 4.6 °C** Probe Regulation: Transient Tune: Misfit Recording: Off Probe: 5 mm PABBI 1H/19F/D-BB Z-GRD Z120175/0002

Low temperature measurements

- > enter desired temperature and appropriate evaporation power
- > wait until target temperature is reached and stable (this may take some time, especially when cooling down from room temperature as all the equipment is still warm then)
- > regulation state should be transient

What to do if the regulation state says „insufficient chilling“?

- > check whether the transfer line hose sits tightly on the gas inlet and tighten it further if necessary
- > putting a towel around the hose helps to prevent ice formation on the magnet
- > slightly increase the evaporation power



- Use 3 % for 0 °C
- Use 5 % for -25 °C
- Use 7 % for -50 °C
- Use 10 % for -75 °C

For longer measurements (overnight) at temperatures lower than 0 °C or higher than 80 °C, switch on the shim gas to prevent ice formation in the shim system by turning the blue knob at the back of the console. Use this preferably after 3 pm as the shim gas of av400 is pretty loud



abs1/abs2	Baseline correction of F1/F2 in 2D spectrum
absb	Baseline correction of both dimensions in 2D spectrum
abs n	Baseline correction in 1D spectrum
apk	Phase correction in 1D spectrum
aq	Show/change acquisition time
atma	Automatic tuning and matching of the probe to the frequency of the nucleus to be measured
atmm	Manual tuning and matching of the probe to the frequency of the nucleus to be measured
dosy2d	Creates typical pseudo-2D DOSY spectrum
edlock	Show table of available solvents and their locking properties
efp	Performs 1D fourier transformation
ej	Ejects sample/turns on air stream
expt	Shows estimated duration of experiment
halt	Stops running experiment after currently running scan and saves fid
ij	Injects sample/turns off air stream
lock/lock *solvent name*	Select solvent for locking/starts locking on the stated solvent
lockdisp	Opens lock window

new	Creates new data set by copying open data set
ns	Show/change number of scans
o1p	Show/change transmitter frequency (center of spectrum)
projd	Project already existing 1D data sets as traces of 2D data set
rg/rga	Show/change receiver gain/automatically adjust receiver gain
rsh	Read existing shim file from disk
send	Send your data to your group server after measurement is complete
setdiffparm	Creates diffusion dimension during DOSY processing
stop	Stops acquisition immediately without saving fid
sw	Show/change spectral width
tg	Opens TopShim window
topshim	Performs gradient shimming on sample
tr	Save already recorded scans to disk while experiment is still running
xau dosy	Starts DOSY experiment
xfb	Performs 2D fourier transformation (both dimensions)
zg	Starts any 1D and 2D experiment (except for DOSY)