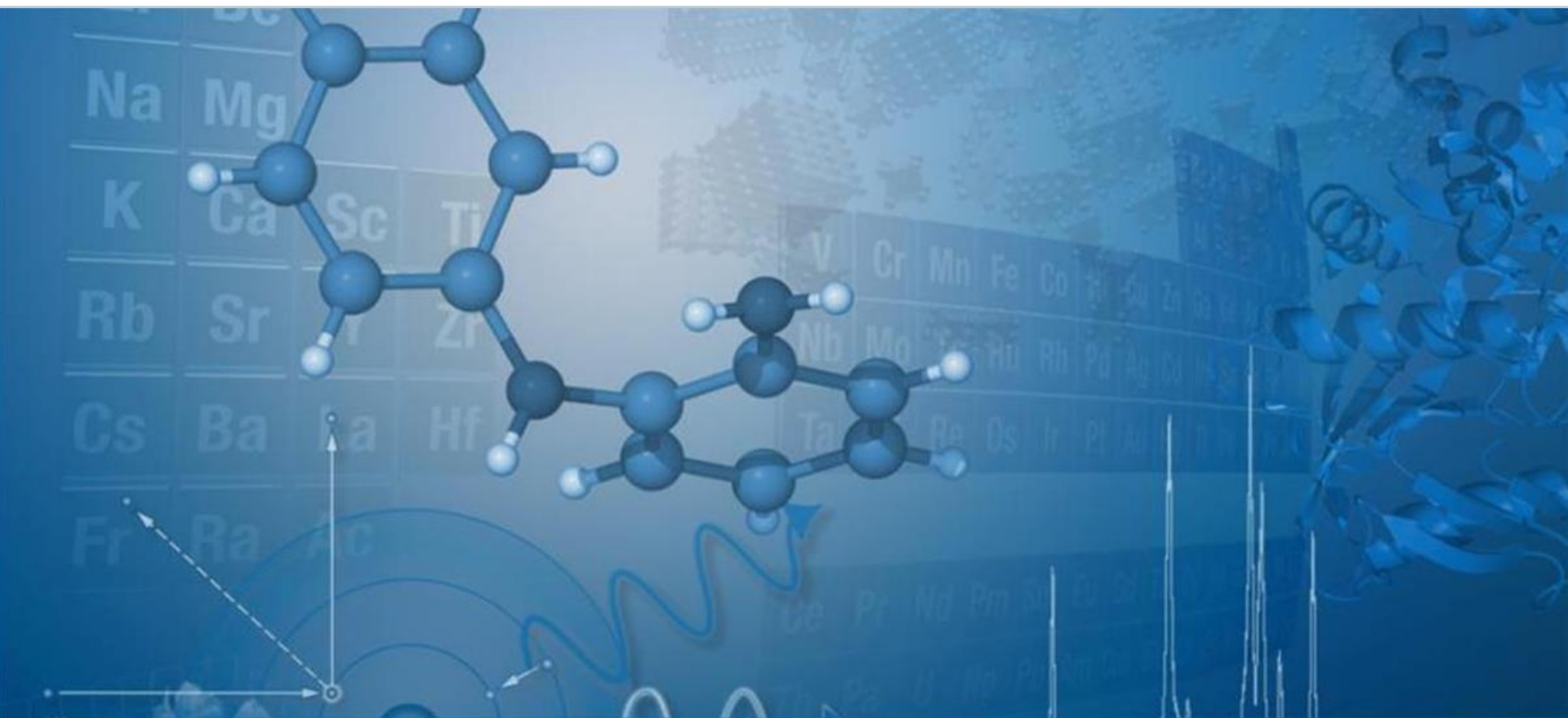


1D Acquisition

Dr. Benjamin Görling

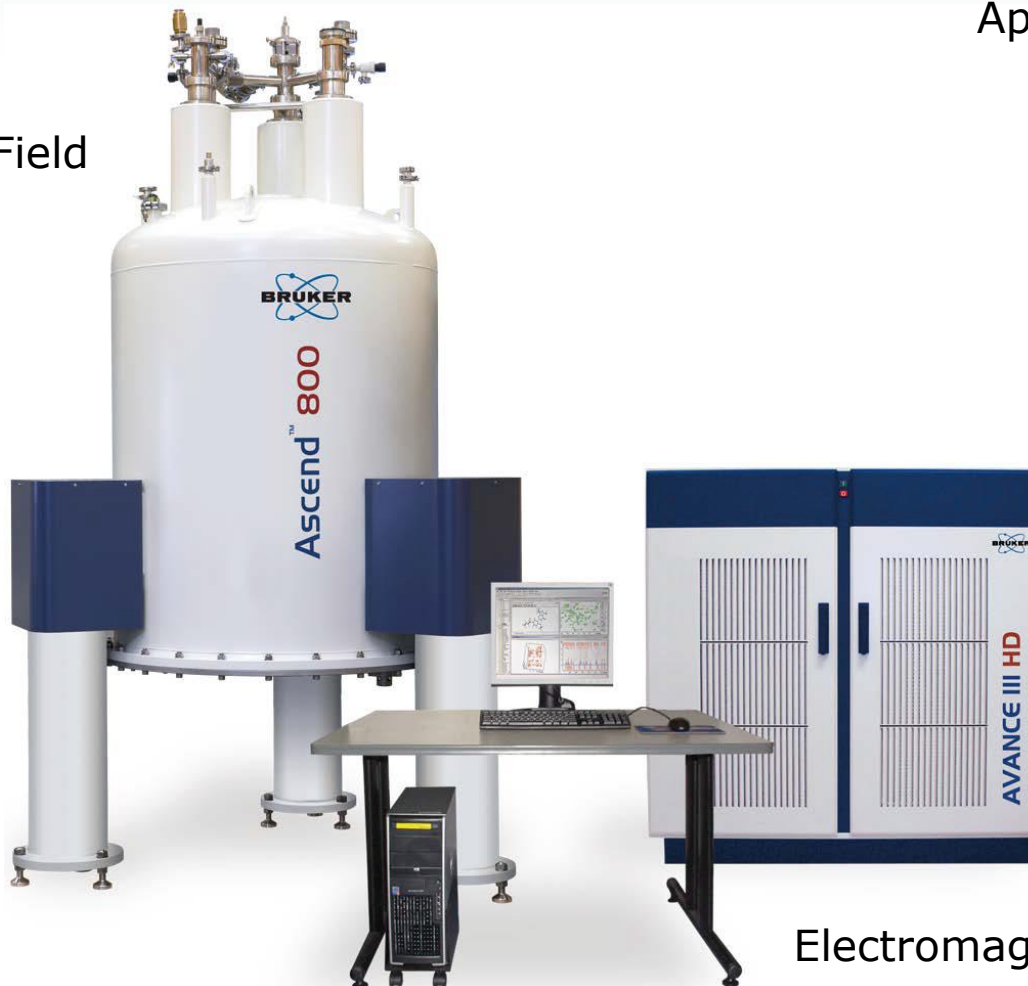


- **What is NMR?**

What do we need for NMR?



Magnetic Field



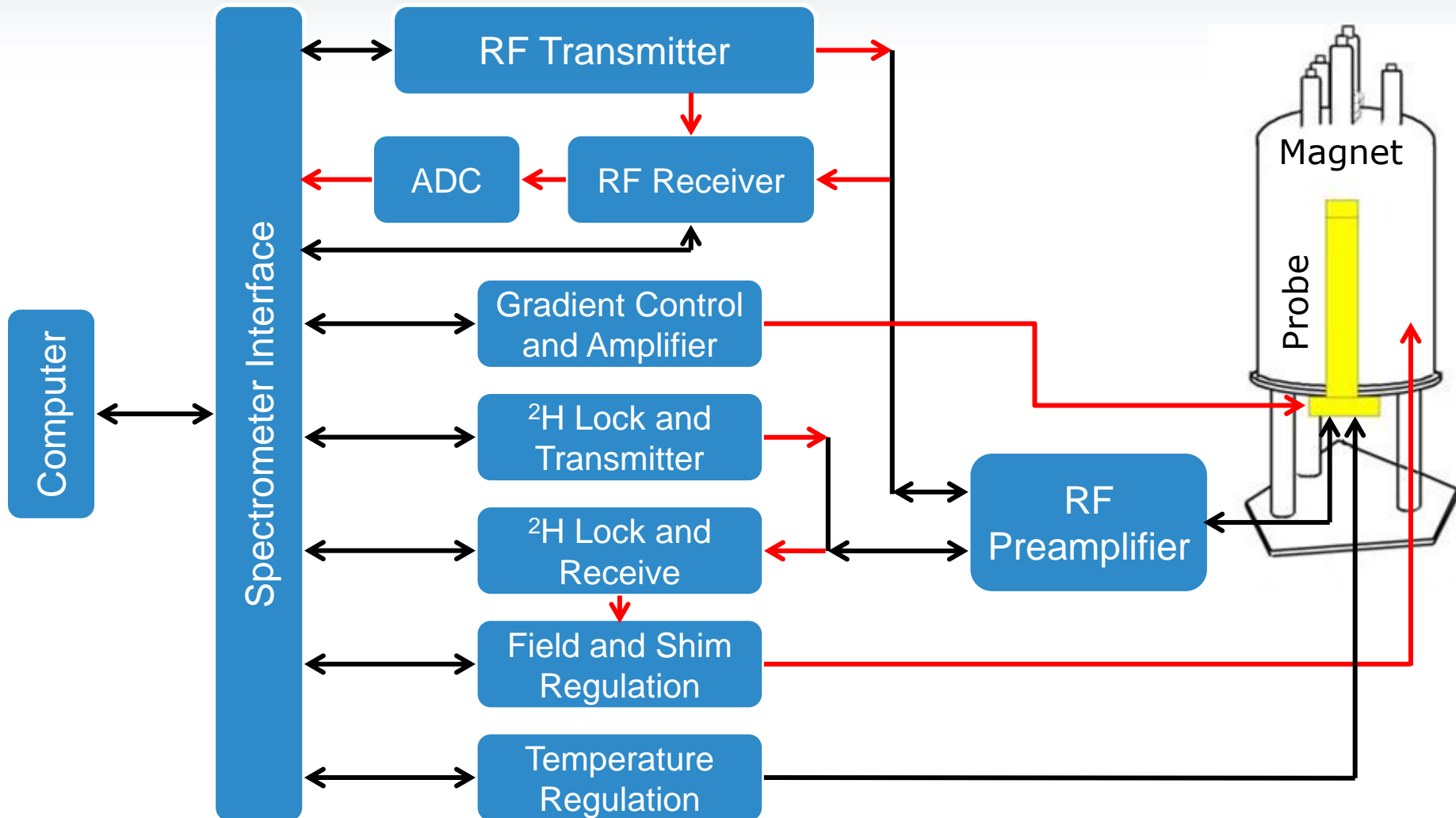
Appropriate Nucleus



Probe

Electromagnetic Frequencies

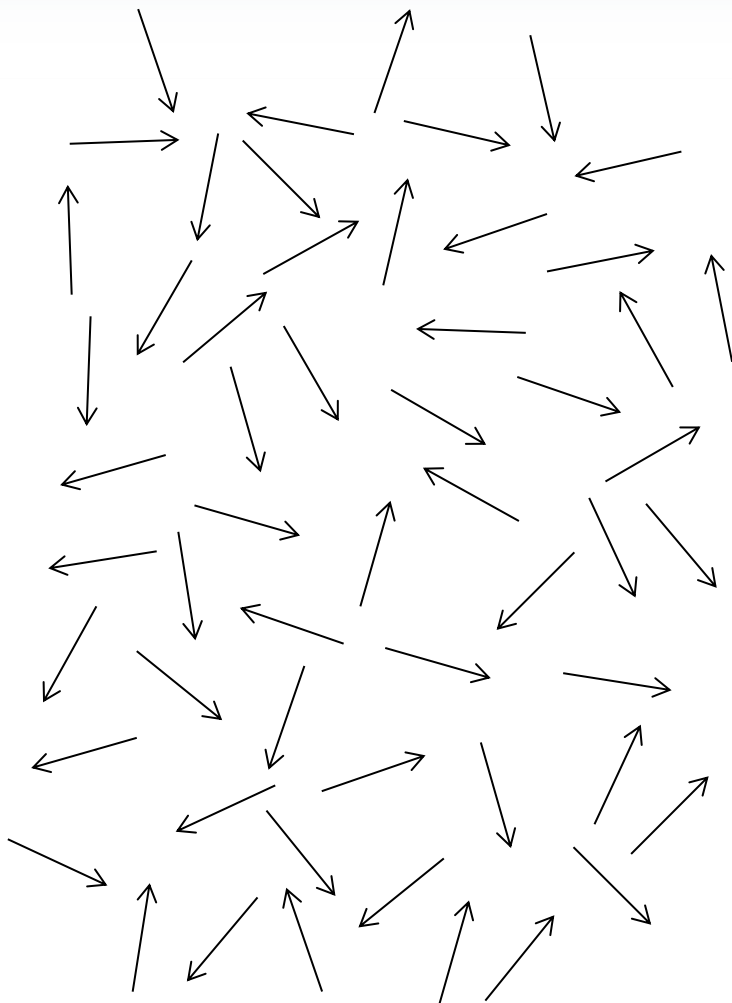
The NMR spectrometer



What happens in the magnet?



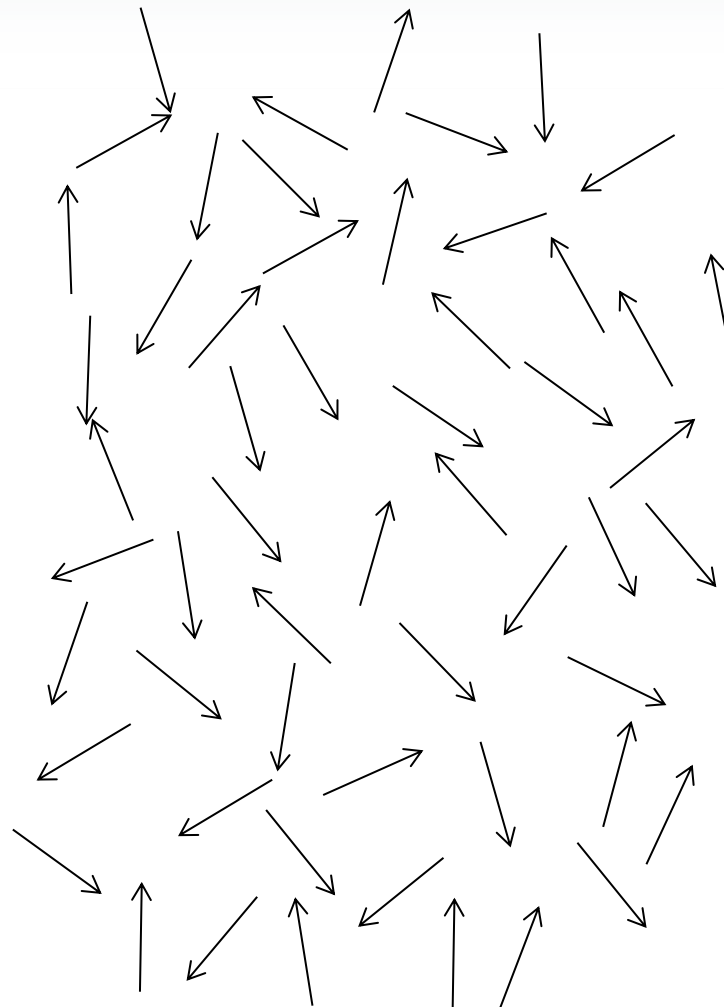
No magnetic field



B_0



External magnetic field

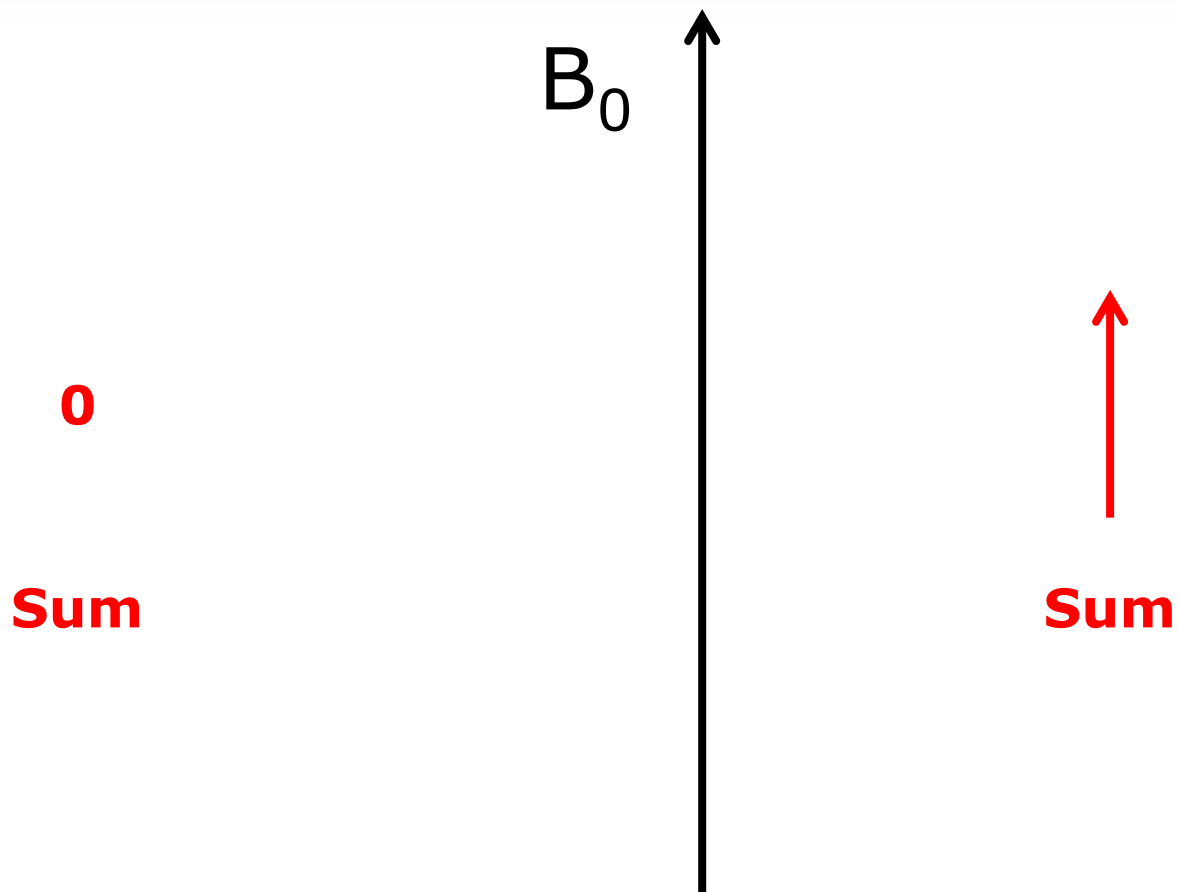


What happens in the magnet?

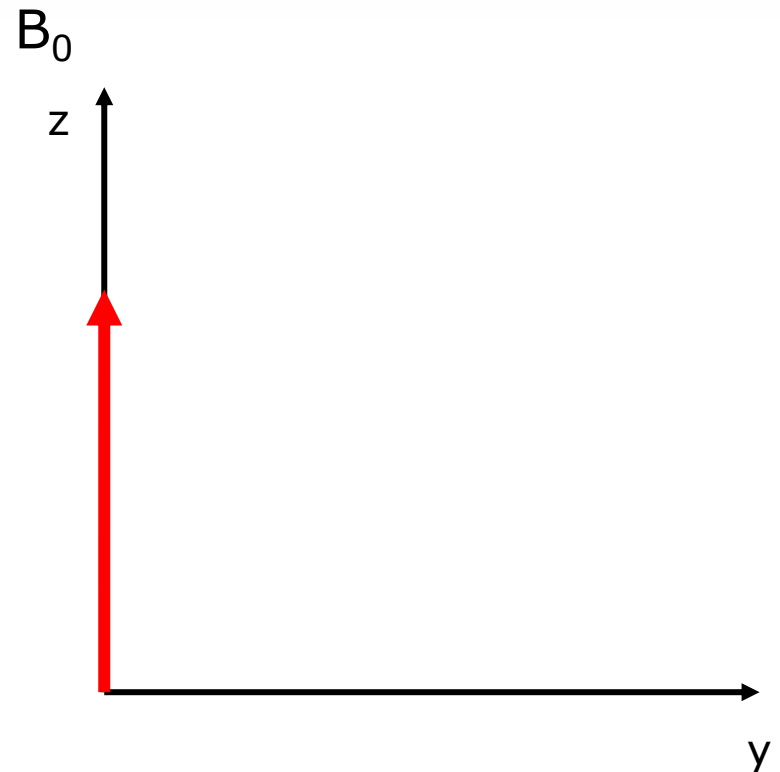


No magnetic field

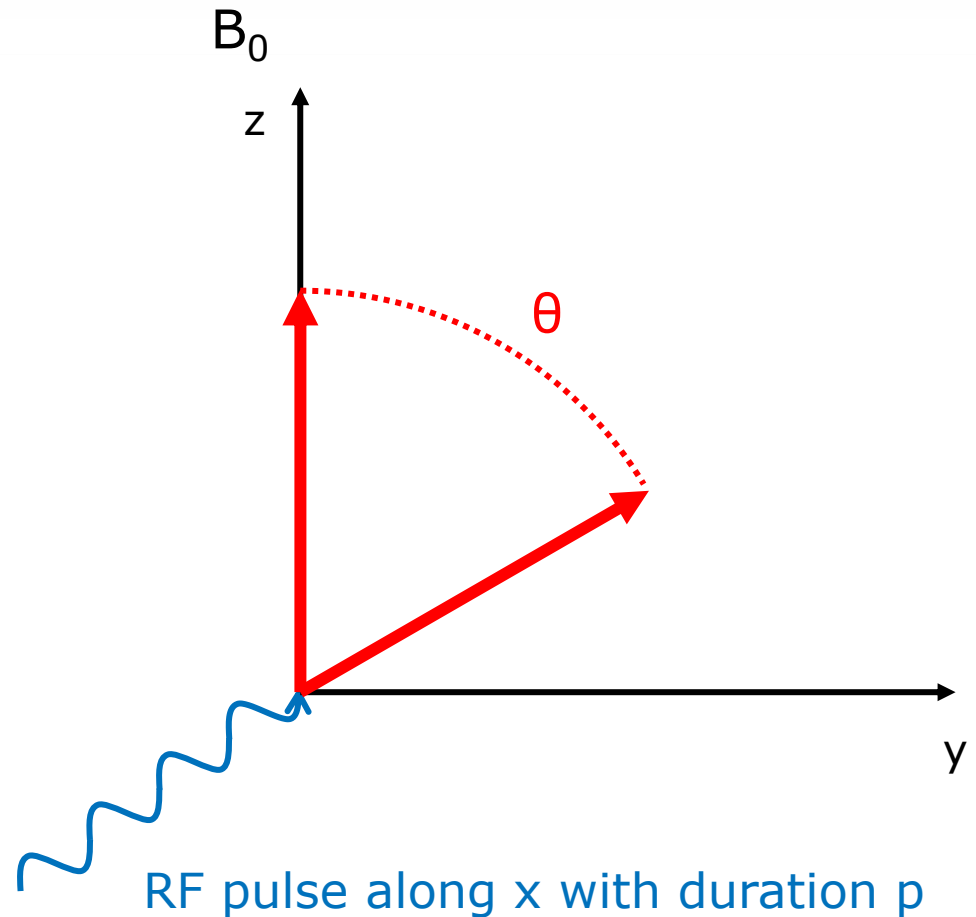
External magnetic field



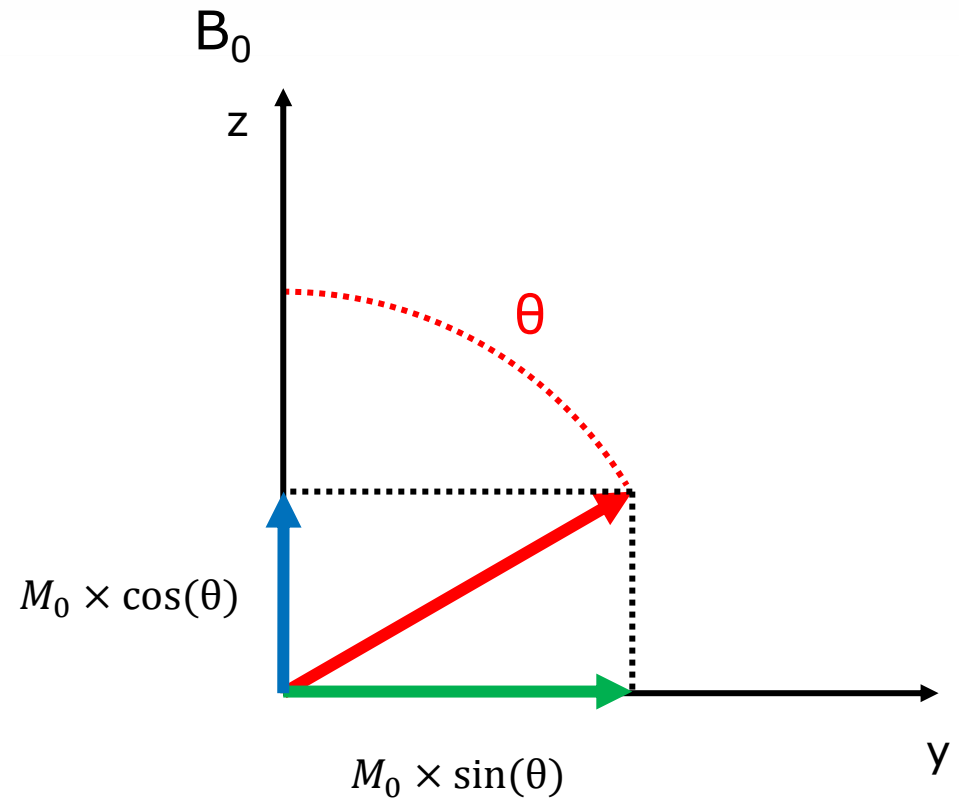
- Bulk magnetization is aligned along +z
- Can be rotated with radio frequency pulses



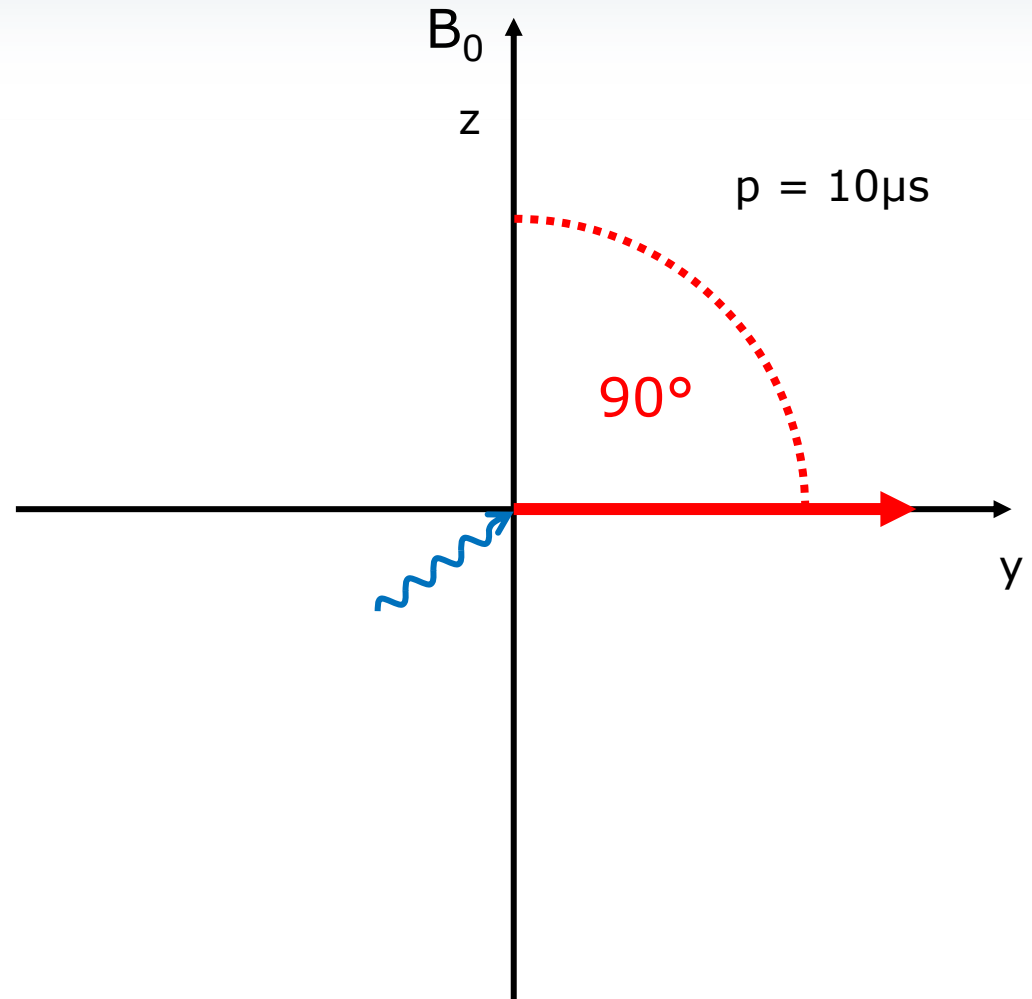
- Bulk magnetization is rotated around the axis of the pulse
- Angle θ depends on the duration (p) and the power of the pulse



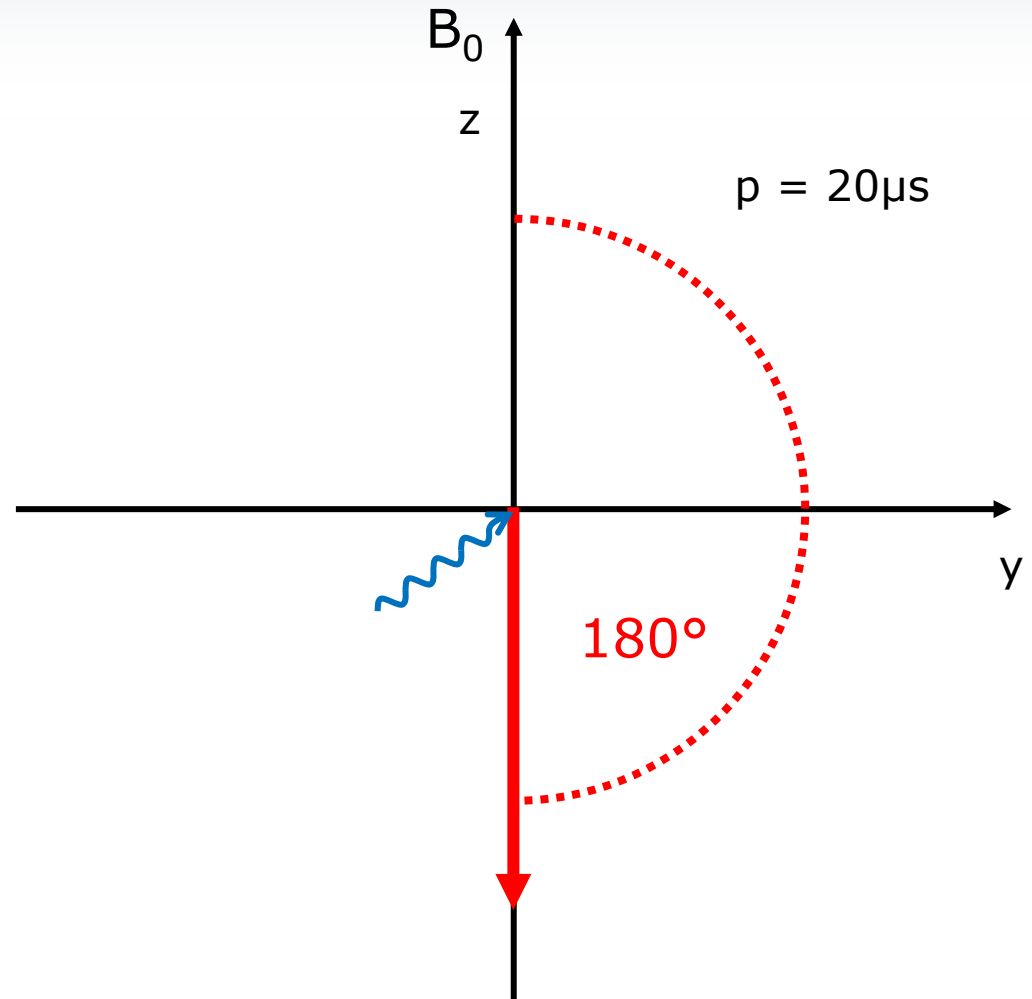
- x/y-component of the magnetization can be measured



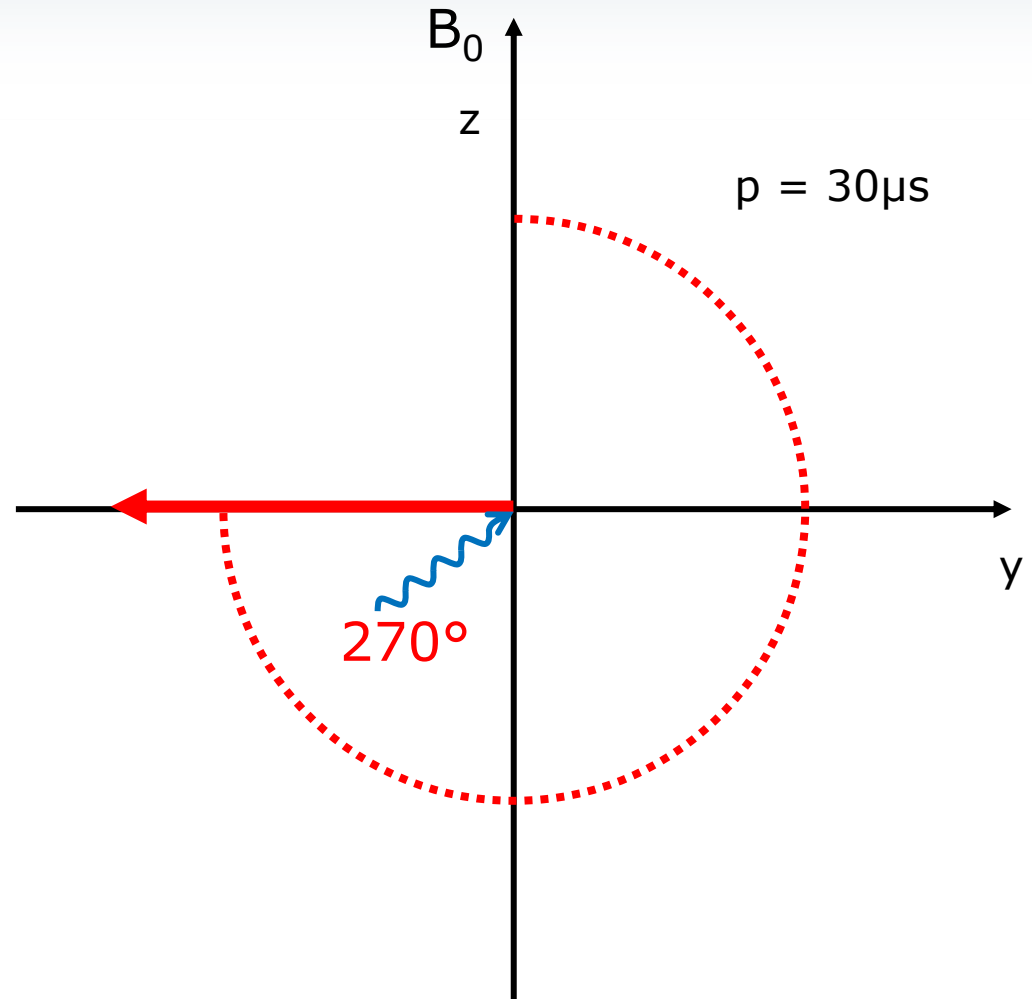
- Pulse angle is proportional to the duration p of the pulse (at constant power level)



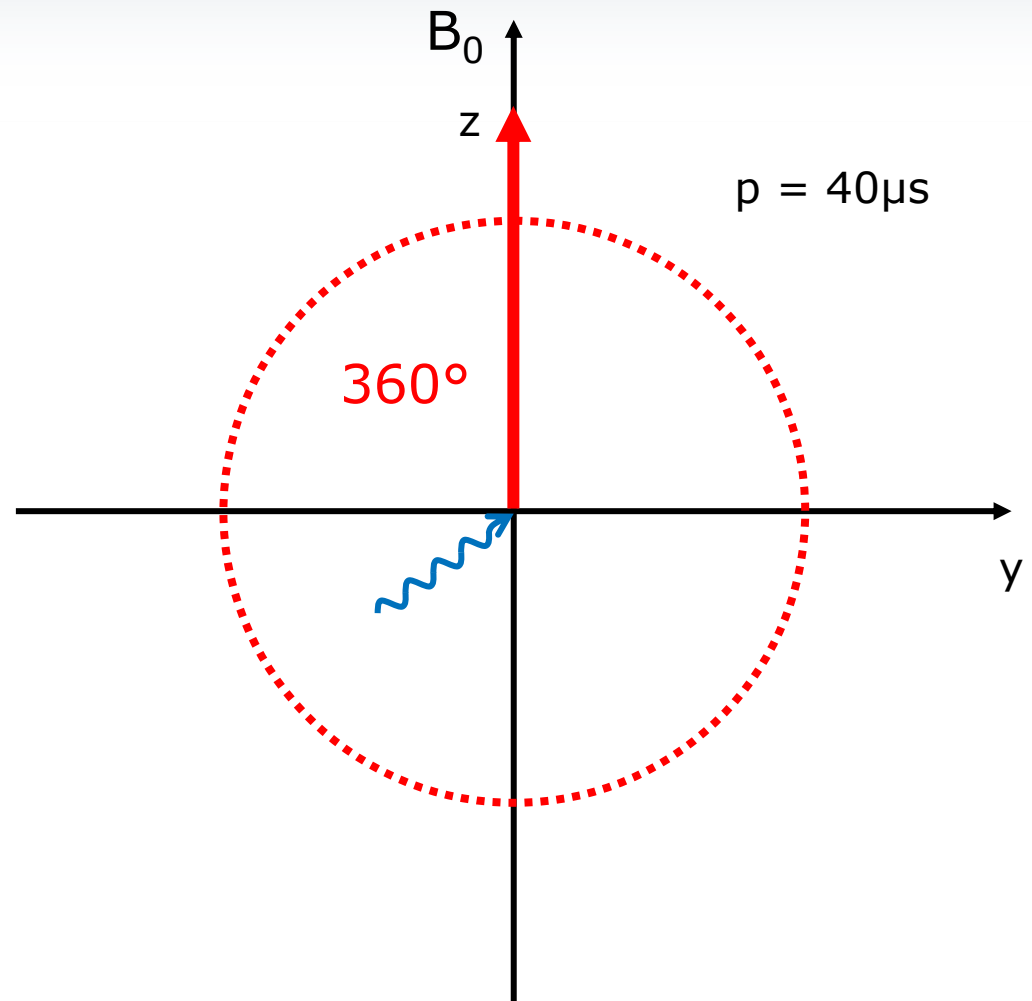
- Pulse angle is doubled if pulse length is doubled



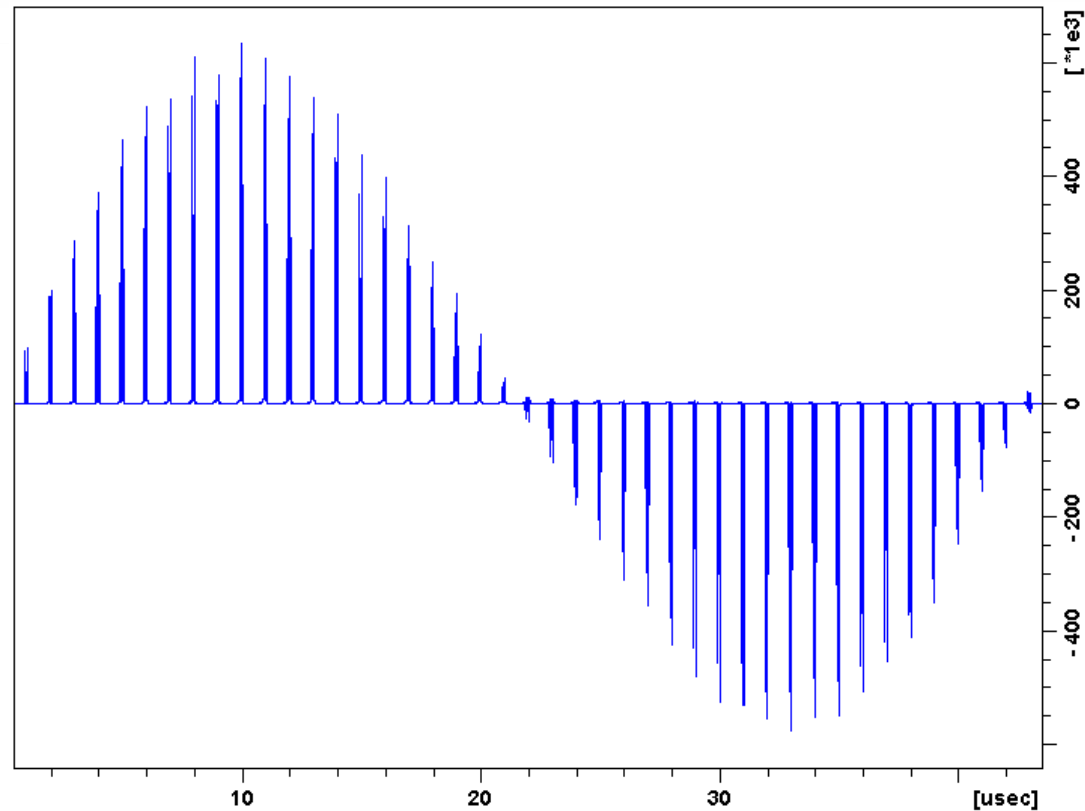
- Three times the duration results in a 270° pulse



- Four times the duration results in a 360° pulse



- Maximum signal intensity can be achieved with a 90° pulse
- Length is changed to find maximum
- In practice zero crossing for 180° or 360° is used

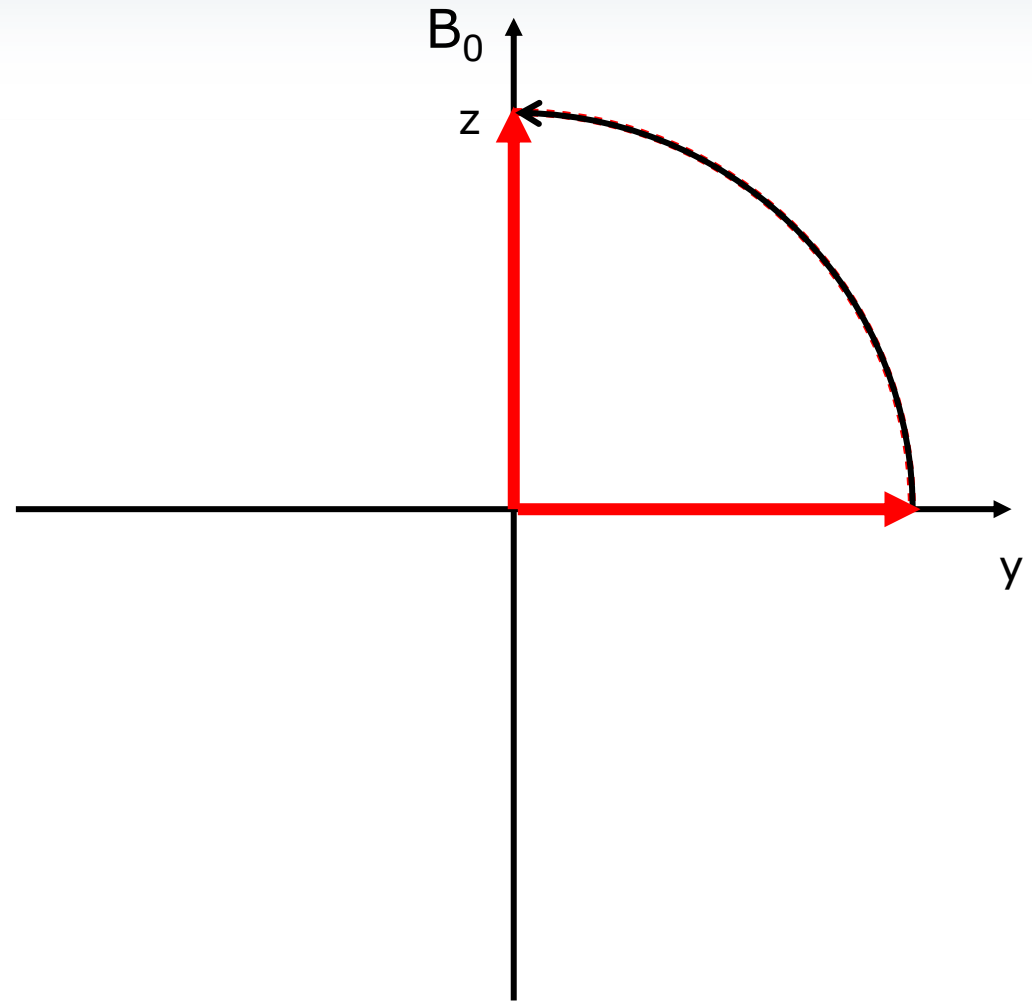


- Two parameters define a pulse: the duration of the pulse **P1** and its power level **PLDB1/PLW1**.
- **P1** is the duration of a 90° pulse (typical values are 8-10 μ s for a ^1H 90° pulse)
- **PLDB1/PLW1** is the power level (given by its attenuation (dB) or in W)

Relaxation



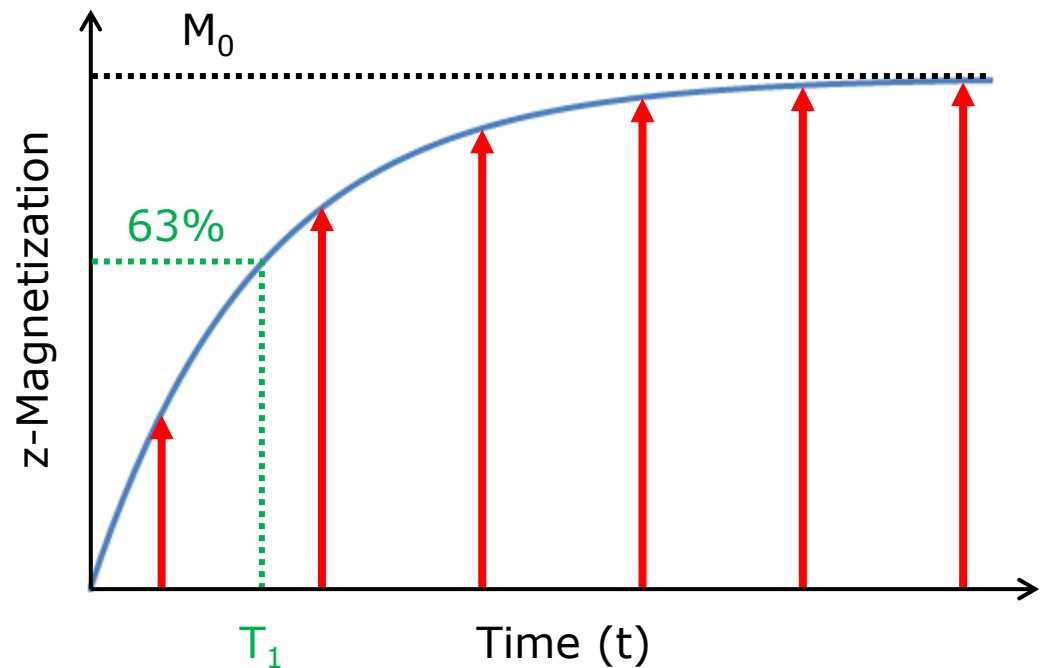
- After the excitation with the pulse, the magnetization returns back to starting position
- This is called relaxation



Relaxation



- Magnetization needs to be at starting position for next experiment to get maximum intensity
- Characterized by so called T_1 time
- Fully relaxed at $>5 \times T_1$

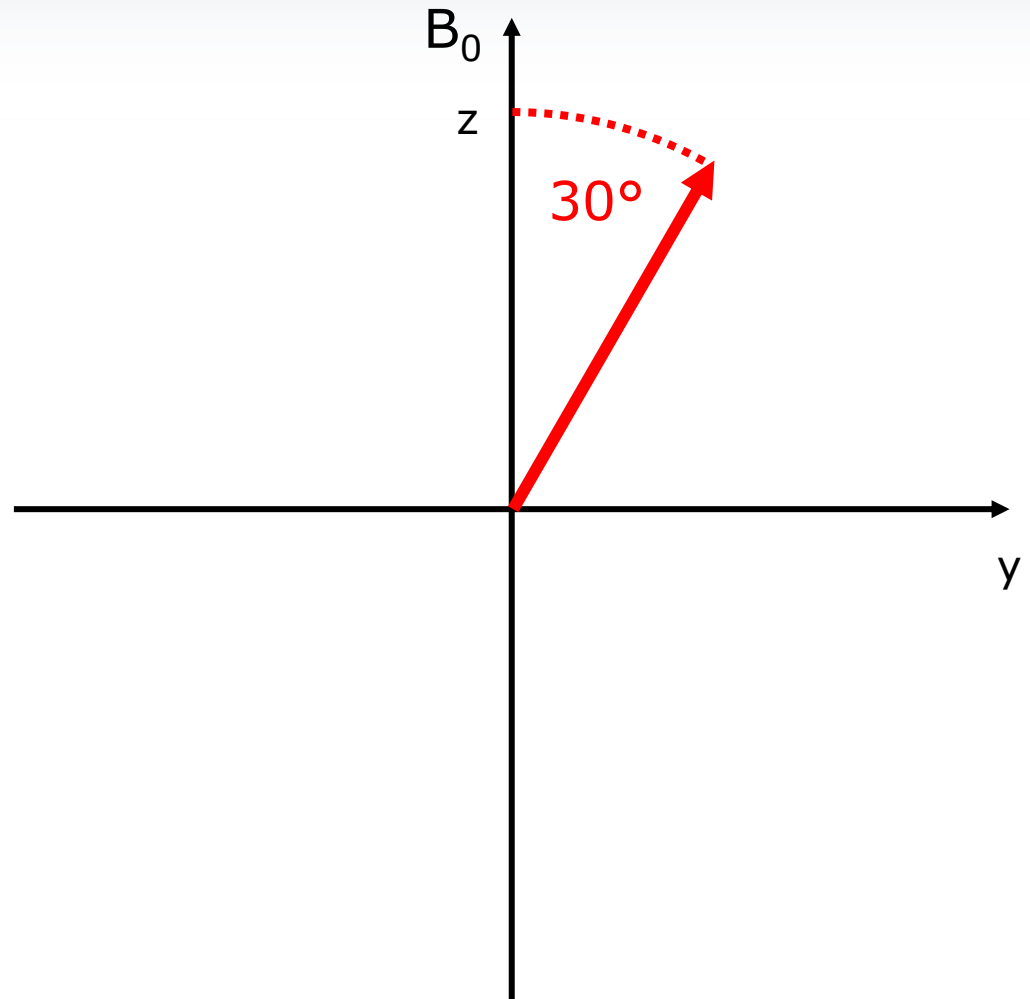


$$M = M_0 \times \left(1 - e^{-\frac{t}{T_1}}\right)$$

30° pulse



- For normal 1D experiments pulse angles of 30° are typically used
- 2D- and special 1D- experiments need well defined pulse angles



30° pulse

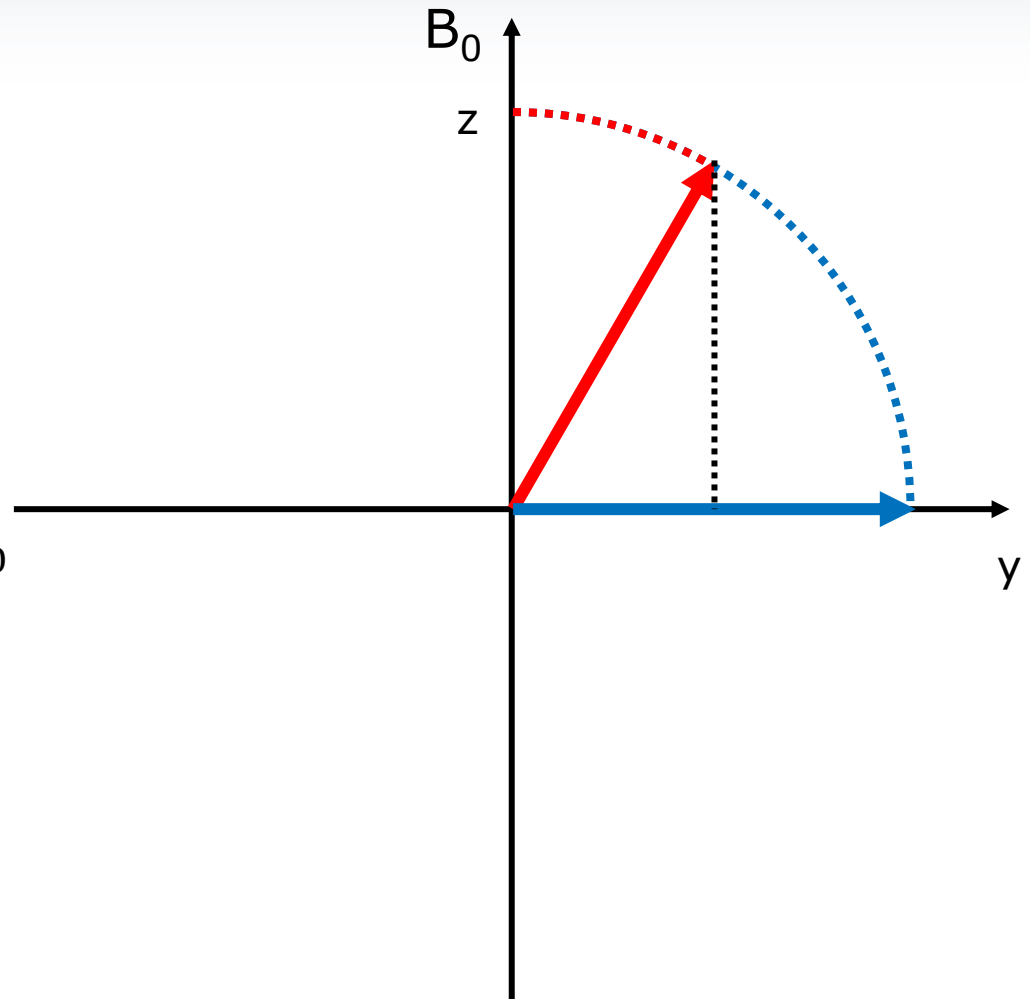


- Half the intensity of a 90° pulse

$$M_y = M_0 \times \sin \theta$$

$$M_y = M_0 \times \sin 30^\circ = 0.5 \times M_0$$

- Shorter relaxation delay necessary

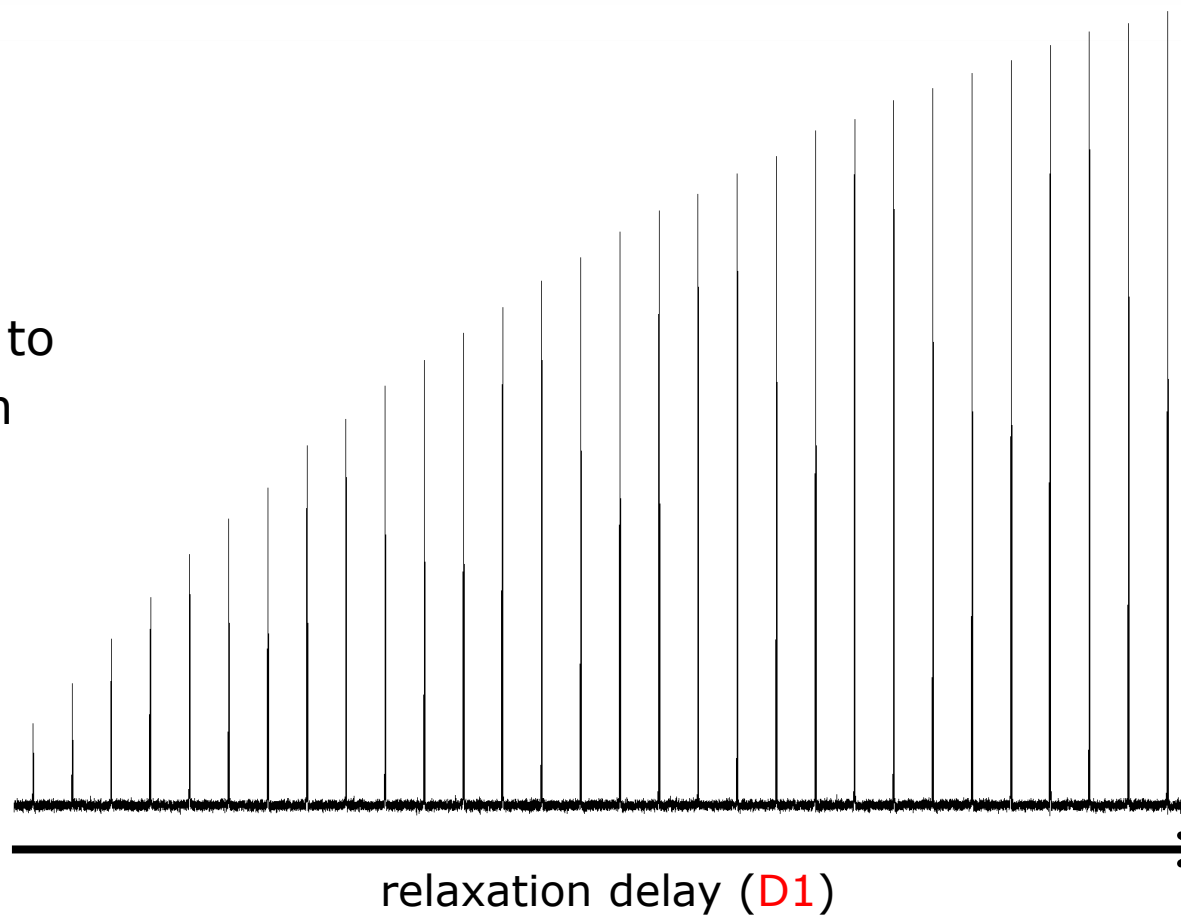


- Other parameters are :
 - the delay **D1**
 - number of scans **NS**
 - number of dummy scans **DS**
- **D1** is executed before pulsing and is the time the sample needs to relax; typically in the range of 1 to 5s (special experiments need a longer delay like T_1 measurement).
- **NS** means that the pulse sequence is executed several times; typical for a ^1H 1D-experiment are 16 scans.
- **DS** means the number of scans which are not saved. They are needed to get steady state (equilibrium).

Relaxation delay



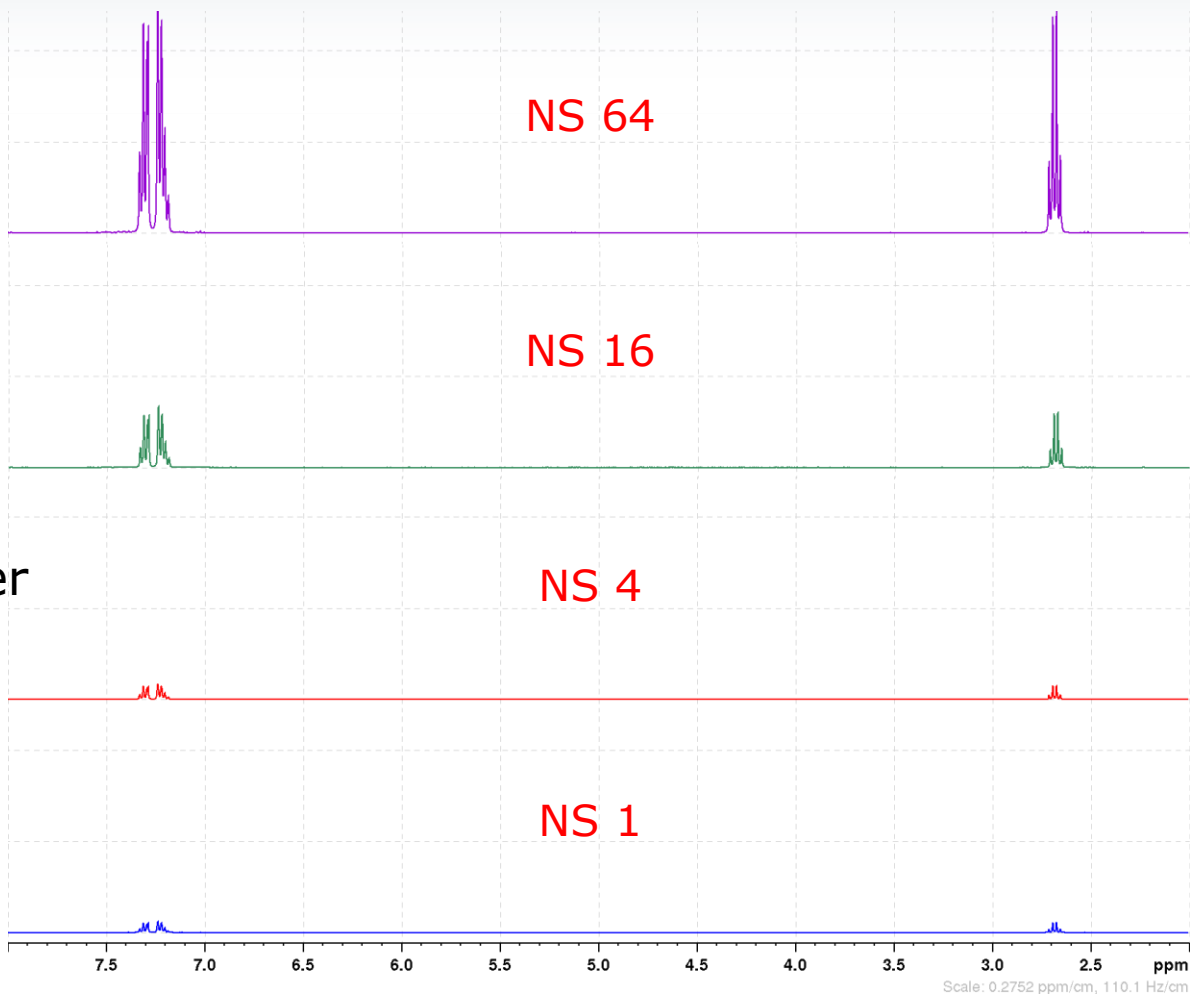
- Loss in intensity due to insufficient relaxation delay



Number of scans



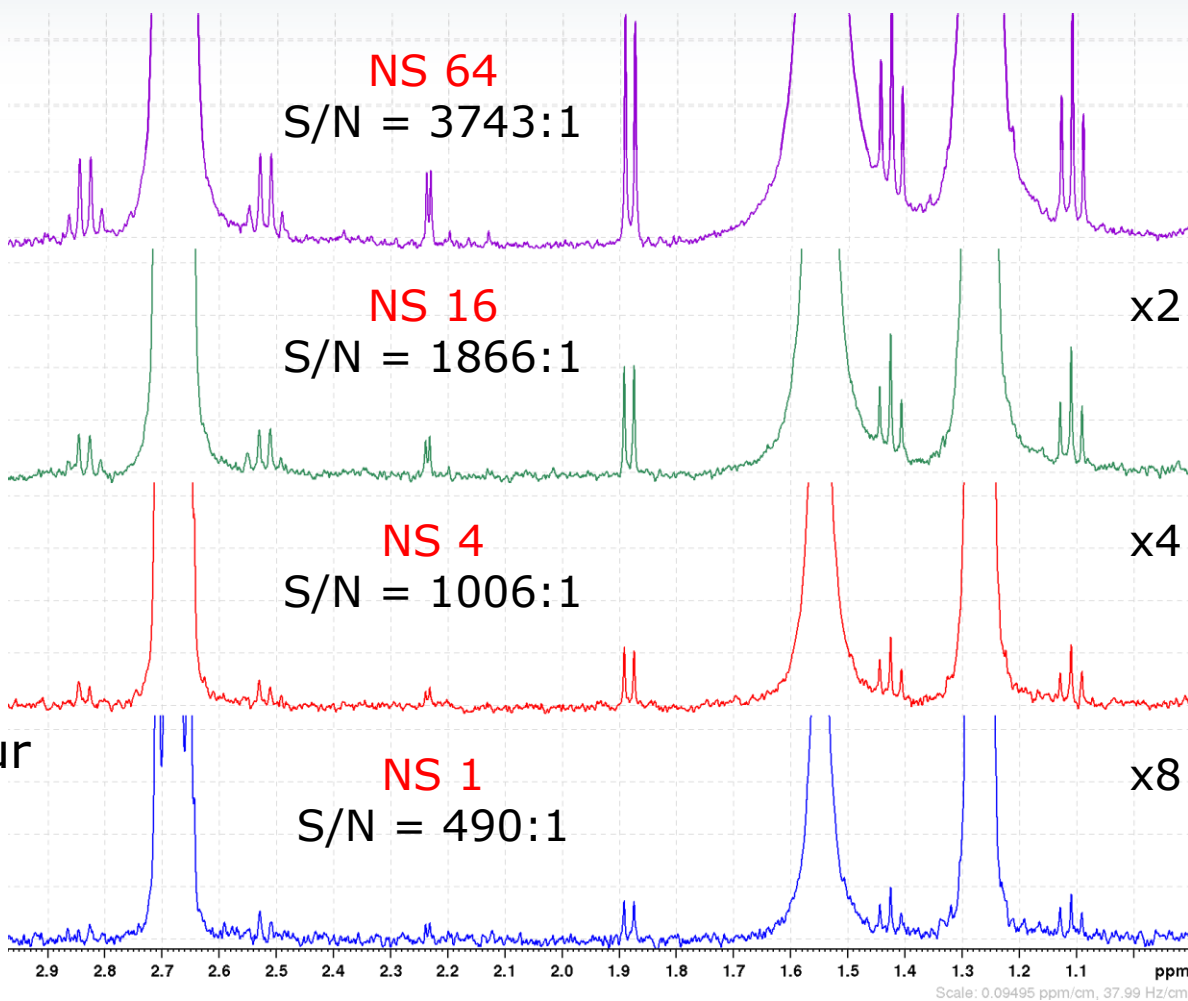
- Signal intensity increases with number of scans



Number of scans



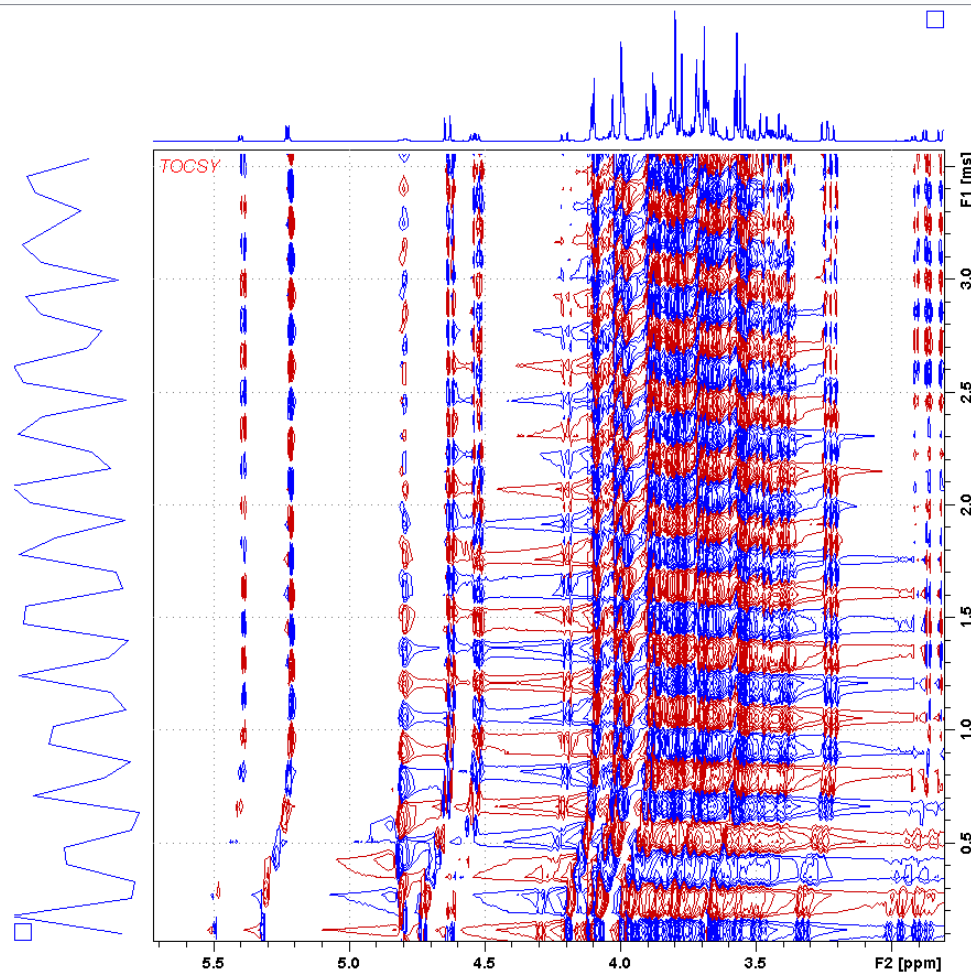
- Noise increases with square root of **NS**
- S/N increases with square root of **NS** as well
- Double S/N needs four times as many scans



Number of dummy scans



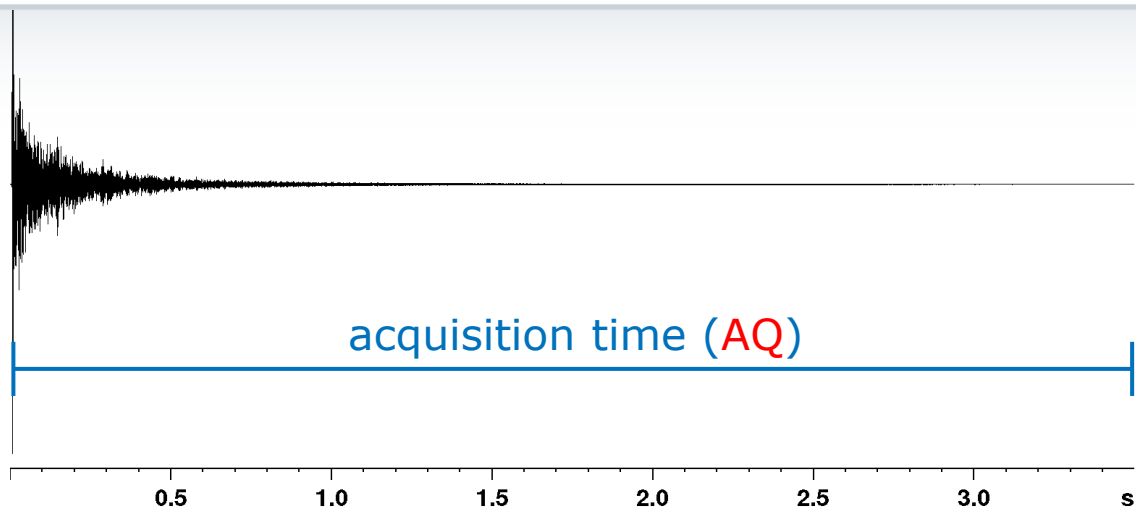
- Not enough dummy scans
- Sample not at equilibrium



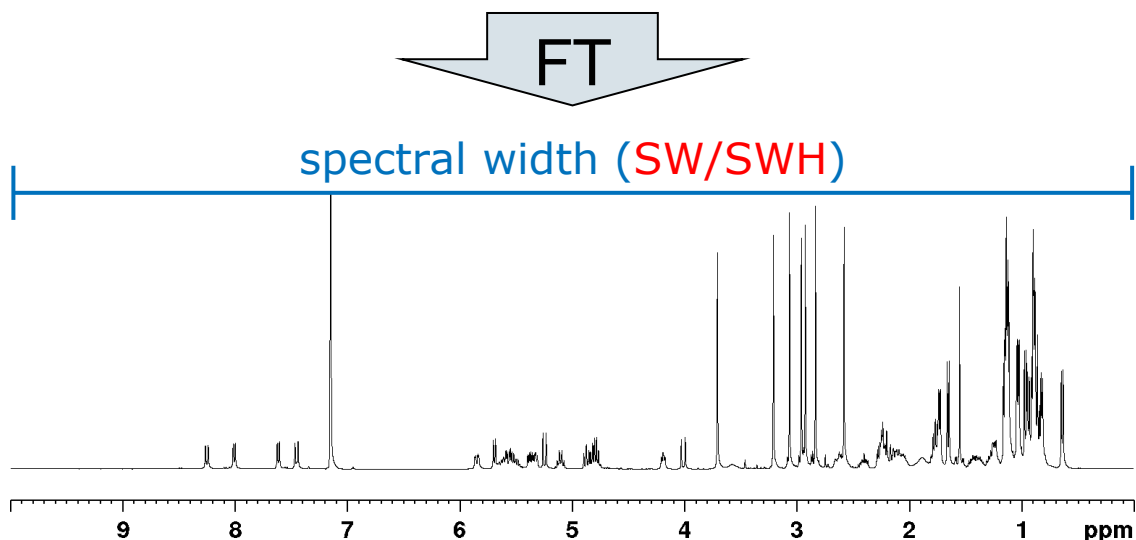
Resolution



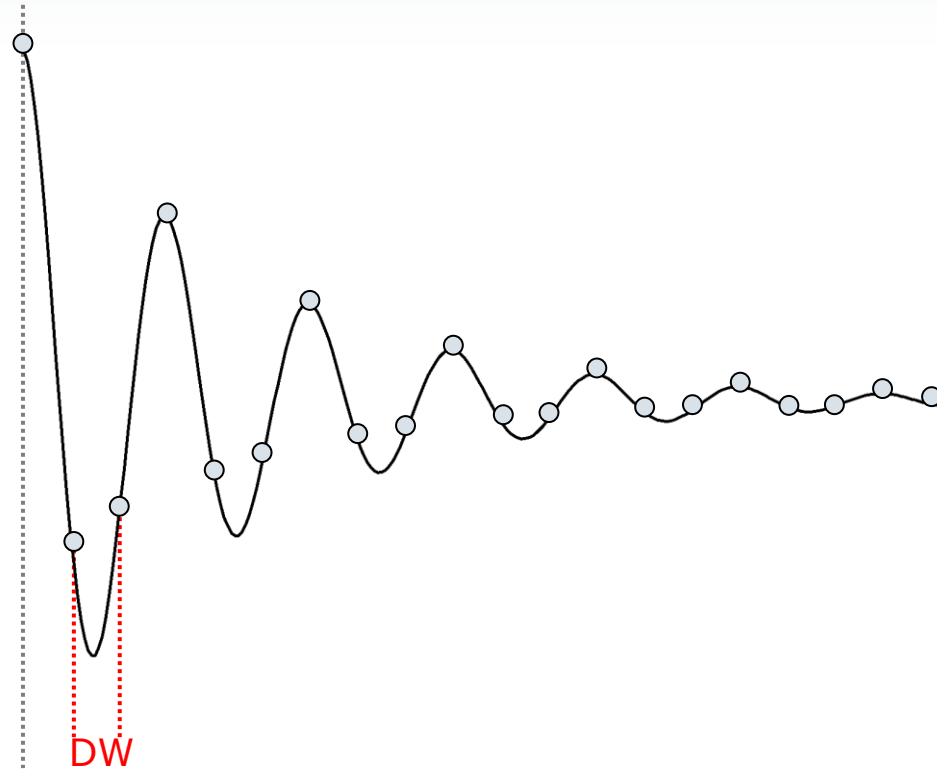
- After the acquisition you will get the so called FID, a function of time $f(t)$.



- With the Fourier Transformation you will get a function of frequency $f(\nu)$, the spectrum.



- Data points must be equidistant
- the distance between two data points is called dwell time (**DW**)
- **TD** is the amount of data points that are acquired

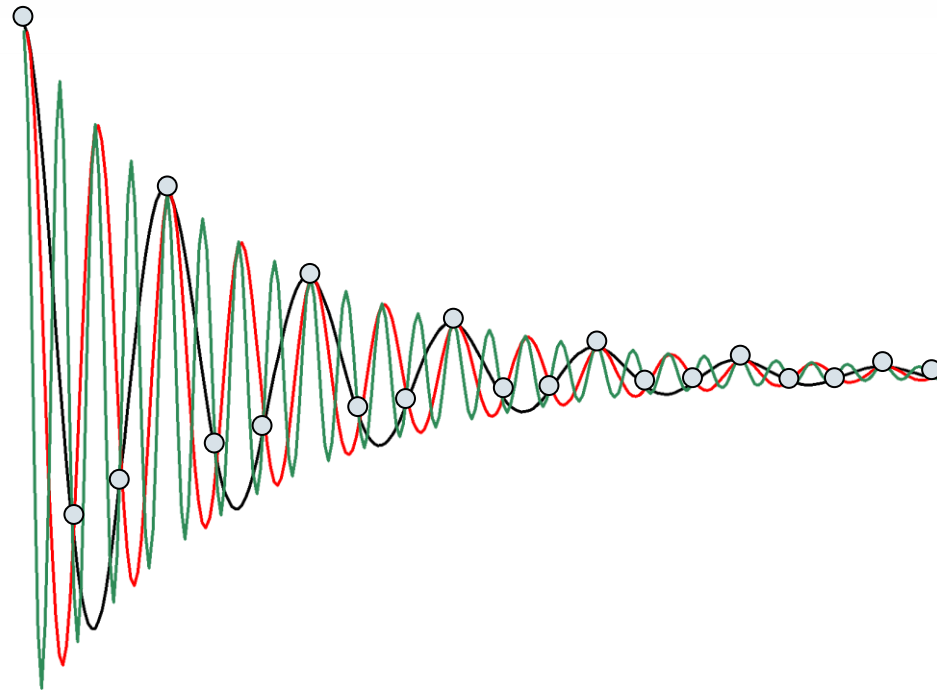


- To find a frequency f , at least two points per sine wave need to be measured

- Nyquist theorem:

$$SWH = \frac{1}{2 \cdot DW}$$

- spectral width **SWH** determines **DW**



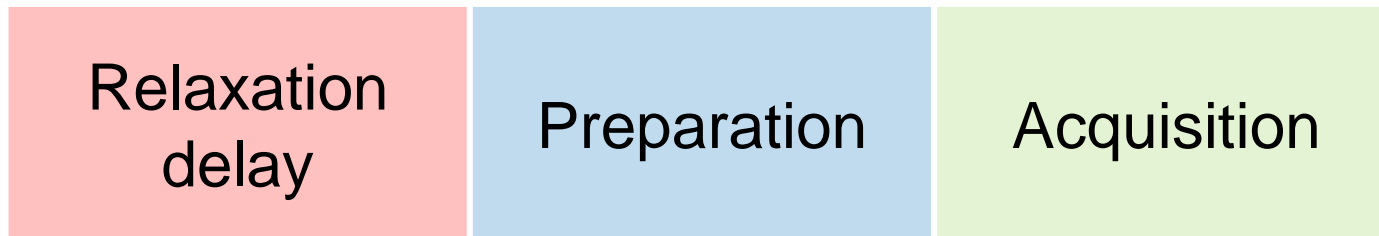
Parameters



- Parameters are :
 - time domain **TD**
 - spectral width **SW/SWH**
 - irradiation frequency offset **O1/O1P**
 - dwel time **DW**
 - acquisition time **AQ**
- **TD** number of raw data points that are acquired in one scan. For a 1D-experiment is typically set to 64k (for a 2D-experiment 1k, 2k or 4k are typical values).
- **SW/SWH** is the spectral width in ppm/Hz. Depends on nucleus (15ppm for 1H, 240ppm for 13C). Defines dwell time **DW**.
- **O1/O1P** represents the irradiation frequency offset in Hz/ppm
- **DW** (dwell time) is the time between two data points.
- **AQ** represents the time to acquire one FID. Defined by **TD** × **DW**.

- **Pulse programs**

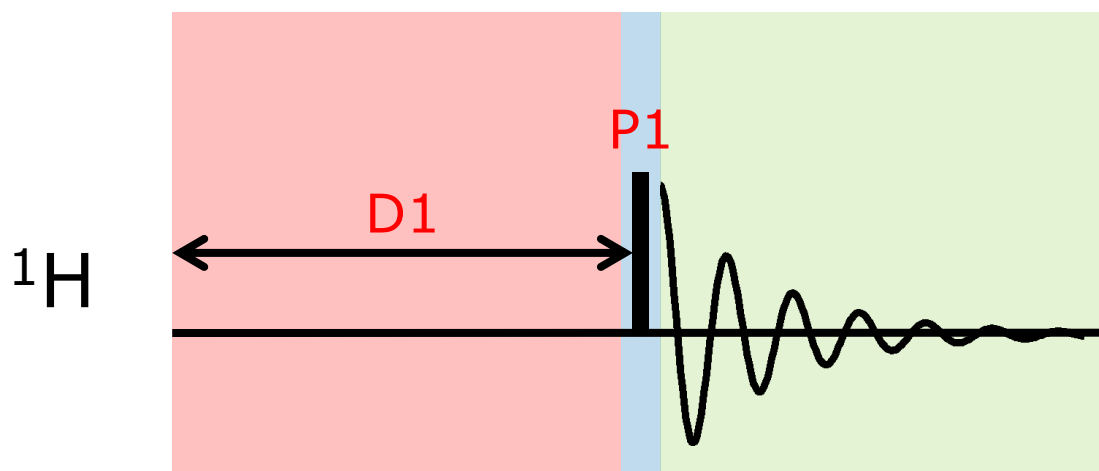
- Pulse programs tell the spectrometer what to do when.
- Building blocks are:



- **Relaxation delay**: time needed for relaxation
- **Preparation**: spins are excited by one or more pulses
- **Acquisition**: Signal is detected as a function of time

Pulse programs

ZG



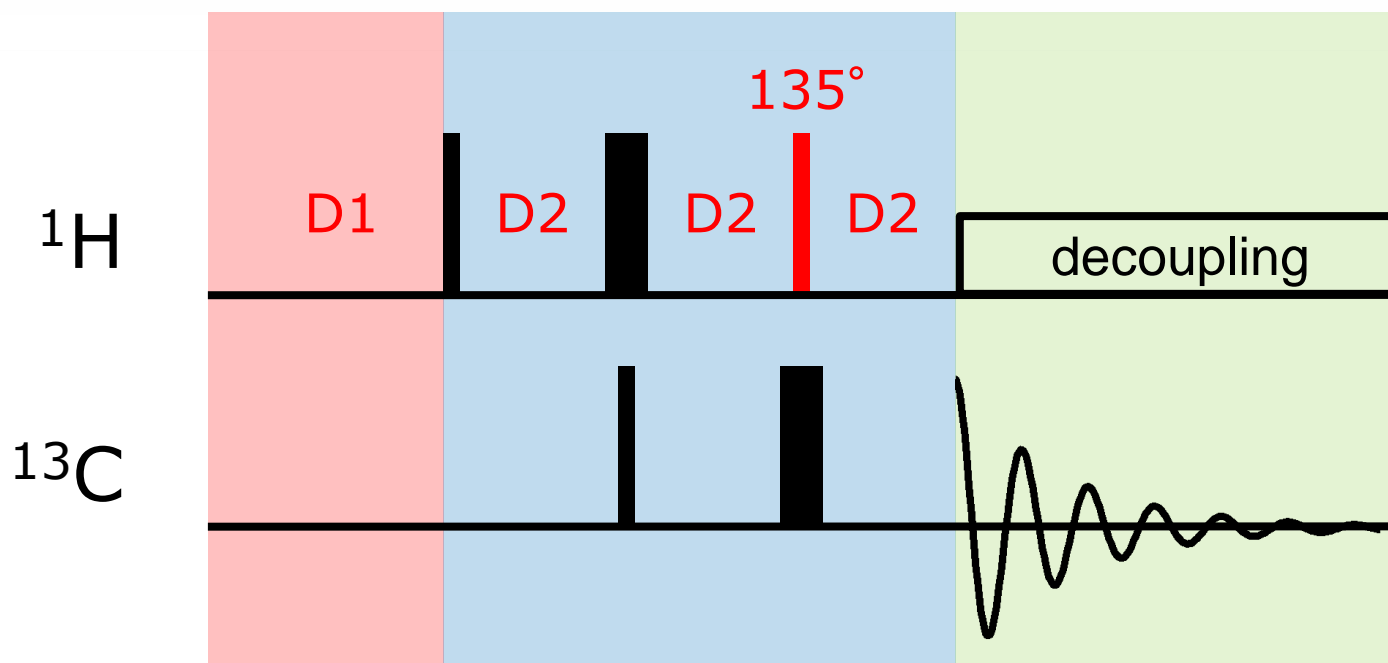
Relaxation
delay

Preparation

Acquisition

Pulse programs

DEPT135



- **How to set up the spectrometer?**

- Configuration of the spectrometer [**cf**]
- Installation of pulse programs, parameter sets etc. [**expinstall**]
- Choosing probe [**edprobe**]

- The commands [**cf**] and [**expinstall**] have to be executed when new software is installed!
- [**expinstall**] has to be executed after changing the routing of the spectrometer.
- The command [**ii restart**] can be used when there is a problem with the instrument.

Install Standard Experiments [**expinstall**]



A screenshot of the Bruker software interface. The top menu bar includes 'File', 'Start', 'Process', 'Analyse', 'Publish', 'View', and 'Manage'. Below the menu bar are buttons for 'Preferences', 'Spectrometer', 'Security', 'Commands', and 'Remote'. The 'Security' button is highlighted with a red box. A dropdown menu is open from 'Security', showing a list of options. The 'Experiments/Parameters' option is highlighted with a red box. A sub-menu is open from 'Experiments/Parameters', and the 'Install Standard Experiments (expinstall)' option is highlighted with a red box. Other options in the sub-menu include 'Convert Parameter Set (paracon)', 'Shape Tool (stdisp)', 'Probe/Solvent Dependent Params (edprosol)', 'Edit Solvent Table (edsolv)', 'Edit Lock Table (edlock)', 'Edit Nuclei Table (ednuc)', and 'Edit customer/system information (edcstm)'. The background shows a toolbar with various icons for file operations and data analysis.

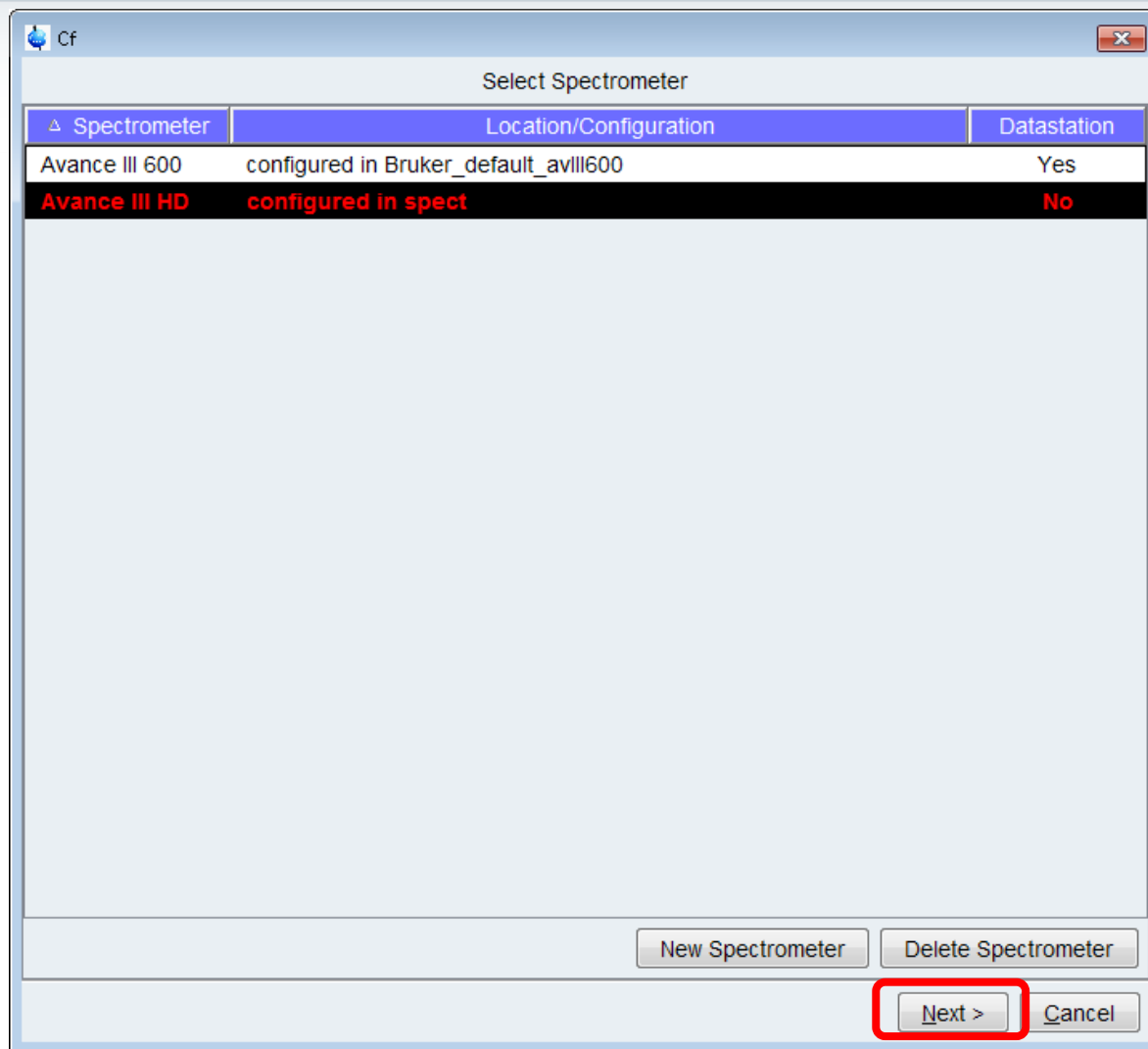
Configure hardware [cf]



The screenshot shows the Bruker software interface with the 'Manage' menu open. The 'Security' option is highlighted in the menu bar. The 'Security' dropdown menu is open, showing several options. The 'Hardware Detection' option is highlighted, and its submenu is open, showing 'Configure Hardware (cf)' as the first option. Other options in the 'Security' menu include 'Experiments/Parameters', 'BSMS Control', 'CryoProbe Control', 'ProdigyDisplay', 'Save/Restore Installation', and 'Spectrometer Usage (account)'. Other options in the 'Hardware Detection' submenu include 'Initialize Spectrometer Interface (ii)', 'Edit the Probe Table (edprobe)', 'Setup Linearization Correction Tables (cortab)', and 'Find Ethernet Addresses (ha)'. The 'Configure Hardware (cf)' option is highlighted in yellow.

Menu Item	Submenu Item
Security	Hardware Detection
Security	Experiments/Parameters
Security	BSMS Control
Security	CryoProbe Control
Security	ProdigyDisplay
Security	Save/Restore Installation
Security	Spectrometer Usage (account)
Hardware Detection	Configure Hardware (cf)
Hardware Detection	Initialize Spectrometer Interface (ii)
Hardware Detection	Edit the Probe Table (edprobe)
Hardware Detection	Setup Linearization Correction Tables (cortab)
Hardware Detection	Find Ethernet Addresses (ha)

Configure hardware [cf]



Configure hardware [cf]



The screenshot shows a software dialog box titled "Edit Configuration Parameters" with a "Cf" icon in the top-left corner. The dialog is organized into three sections:

- Spectrometer Description:** A text field labeled "Description" contains the text "Avance III HD".
- Spectrometer Data:** A text field labeled "1H Spectrometer frequency" contains the value "400.130" followed by "MHz".
- Security Options:** A checkbox labeled "enable power check" is checked.

At the bottom of the dialog, there are three buttons: "< Previous", "Next >", and "Cancel". The "Next >" button is highlighted with a red rectangular box.

Configure hardware [cf]



```
Cf
wait for server to handle parameters

start a new configuration
reinitialize objects
determine instrument name
create directories
read old configuration
send user input to server
wait for server to handle parameters
get permission to continue configuration
continue configuration
parse input from user
check for questions from server
check hardware
there are 12 DHCP controlled devices and 18 devices with fixed IP to check
try to connect 30 devices at the spectrometer subnet
connected: BLA_W1345092_0117 at IP 149.236.99.253
connected: BLA_W1345096_0166 at IP 149.236.99.252
connected: LNP PRODIGY UNIT 2127349/100 at IP 149.236.99.244
connected: DRU1 at IP 149.236.99.89
connected: BACS2_H15000-01_0304 at IP 149.236.99.139
connected: DRU2 at IP 149.236.99.88
connected: ELCB_Z100818_3992 at IP 149.236.99.20
connected: IPSO at IP 149.236.99.243
read configuration data from BSMS/2
Connecting to ipso server at IP 149.236.99.243 ... done
IPSO: connected to spectrometer subnet.
configure AQS racks
read configuration from AQS
read BIS from AQS_SGU1 ... done
read BIS from AQS_RX1 ... done
read all RG values from AQS_RX1 ... done
read BIS from AQS_RX2 ... done
read all RG values from AQS_RX2 ...
```

Cancel

Configure hardware [cf]



Cf

Edit Configuration Parameters

Optional Standard Devices

MAS Pneumatic Control Unit connected to	no
Bruker Automatic Changer (BACS) connected to	no
Cryo Controller connected to	no
Variable Temperature Unit connected to	149.236.99.20

Optional Amplifier Devices

19F Lockswitch connected to Amplifier at Blanking Signal	0
2H Lockswitch connected to Amplifier at Blanking Signal	0

Optional Gradient Control Devices

Gradient Temperature Unit (BCU-20) connected to	no
Preemphasis/Gradient Unit connected to	no
Gradient Power Supply Control Unit connected to	no

Miscellaneous Optional Devices

PC running LC-NMR Software HyStar connected to	no
Radio Frequency Supervisor connected to	no
TOSI connected to	no

< Previous **Next >** Cancel

Configure hardware

[cf]

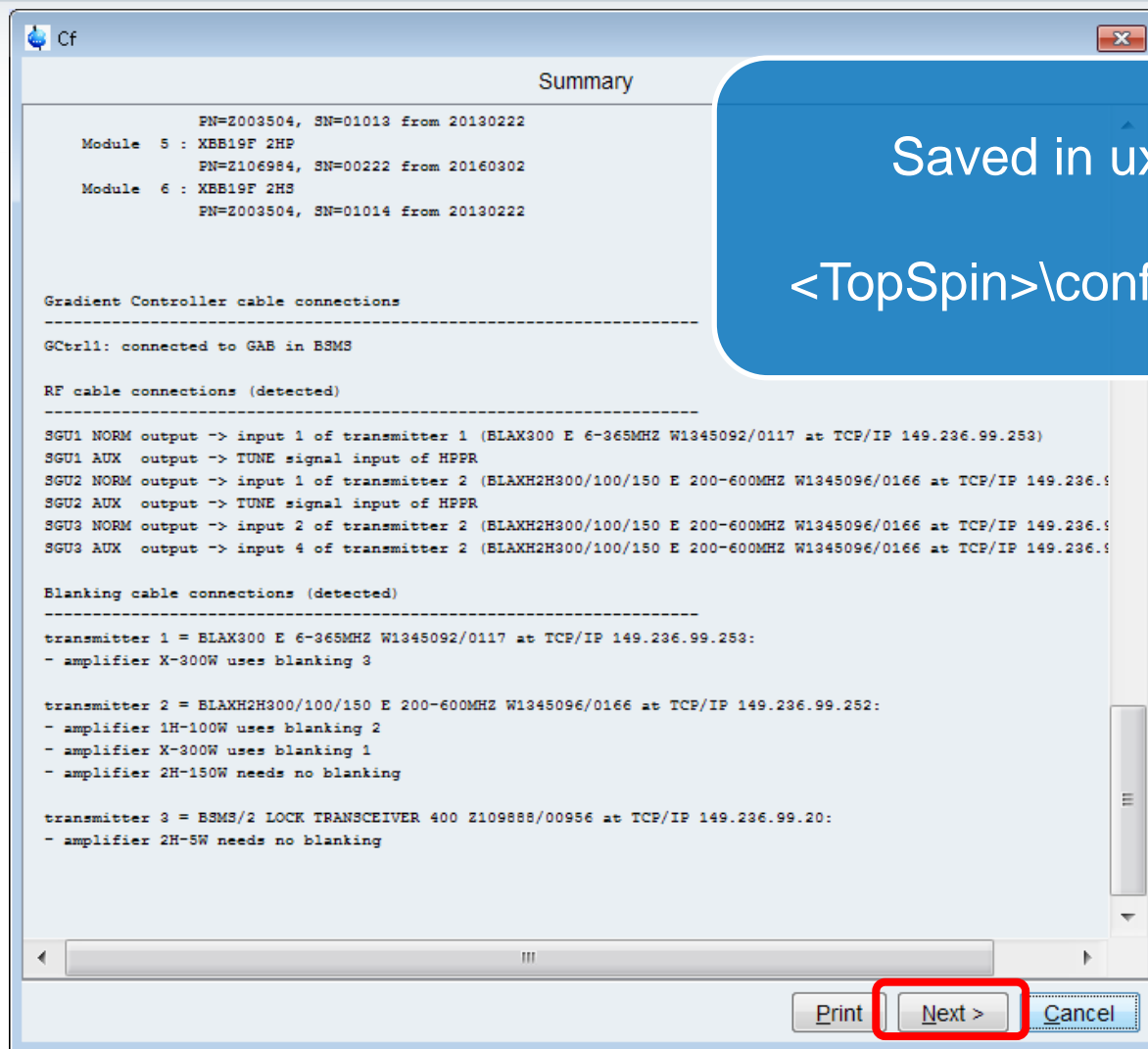


The screenshot shows a terminal window titled "Cf" with a standard Windows-style title bar (minimize, maximize, close buttons). The window content displays a list of configuration steps, many of which are followed by "... done", indicating successful completion. The steps include parsing user input, checking for server questions, finishing configuration, configuring remaining units, and checking HPPR preamplifier configurations. A significant portion of the log is dedicated to detecting RF wiring between SGUs and amplifiers, with specific checks for 186 MHz and 400 MHz signals from various SGU modules (e.g., SGU1-NORM, SGU1-AUX, SGU2-NORM, SGU2-AUX, SGU3-NORM). A "Cancel" button is visible in the bottom right corner of the window.

```
wait for server to finish hardware configuration

continue configuration
parse input from user
check for questions from server
get permission to finish configuration
finish configuration
configure remaining units
check HPPR preamplifier configuration
read HPPR/2 controller configuration
read preamplifier module configuration
read BIS from HPPR/2 module C1 ... done
read BIS from HPPR/2 module P1 ... done
read BIS from HPPR/2 module P2 ... done
read BIS from HPPR/2 module P3 ... done
read BIS from HPPR/2 module P4 ... done
read BIS from HPPR/2 module P5 ... done
read BIS from HPPR/2 module P6 ... done
detect wiring and connections
detect RF wiring between SGUs and amplifiers:
wake up AQS rack 1 ... done
wait 1 second for boards in AQS rack to boot ... done
check if a transmitter detects a 186 MHz signal from SGU1-NORM ... done
check if a transmitter detects a 186 MHz signal from SGU1-AUX ... done
check if a transmitter detects a 400 MHz signal from SGU1-NORM ... done
check if a transmitter detects a 400 MHz signal from SGU1-AUX ... done
check if a transmitter detects a 61 MHz signal from SGU1-NORM ... done
check if a transmitter detects a 61 MHz signal from SGU1-AUX ... done
check if a transmitter detects a 186 MHz signal from SGU2-NORM ... done
check if a transmitter detects a 186 MHz signal from SGU2-AUX ... done
check if a transmitter detects a 400 MHz signal from SGU2-NORM ... done
check if a transmitter detects a 400 MHz signal from SGU2-AUX ... done
check if a transmitter detects a 61 MHz signal from SGU2-AUX ... done
check if a transmitter detects a 186 MHz signal from SGU3-NORM ...
```

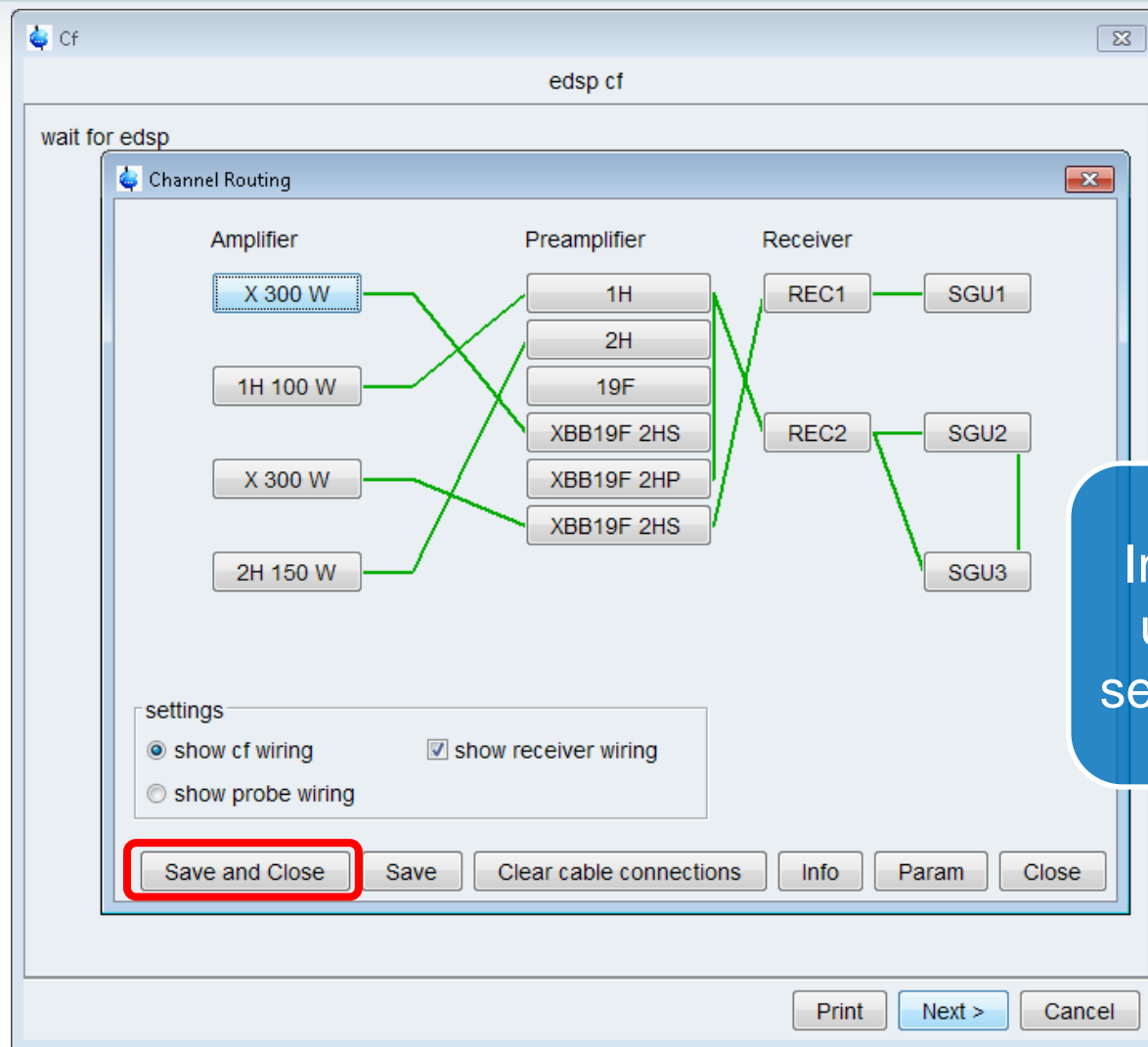
Configure hardware [cf]



Saved in uxnmr.info

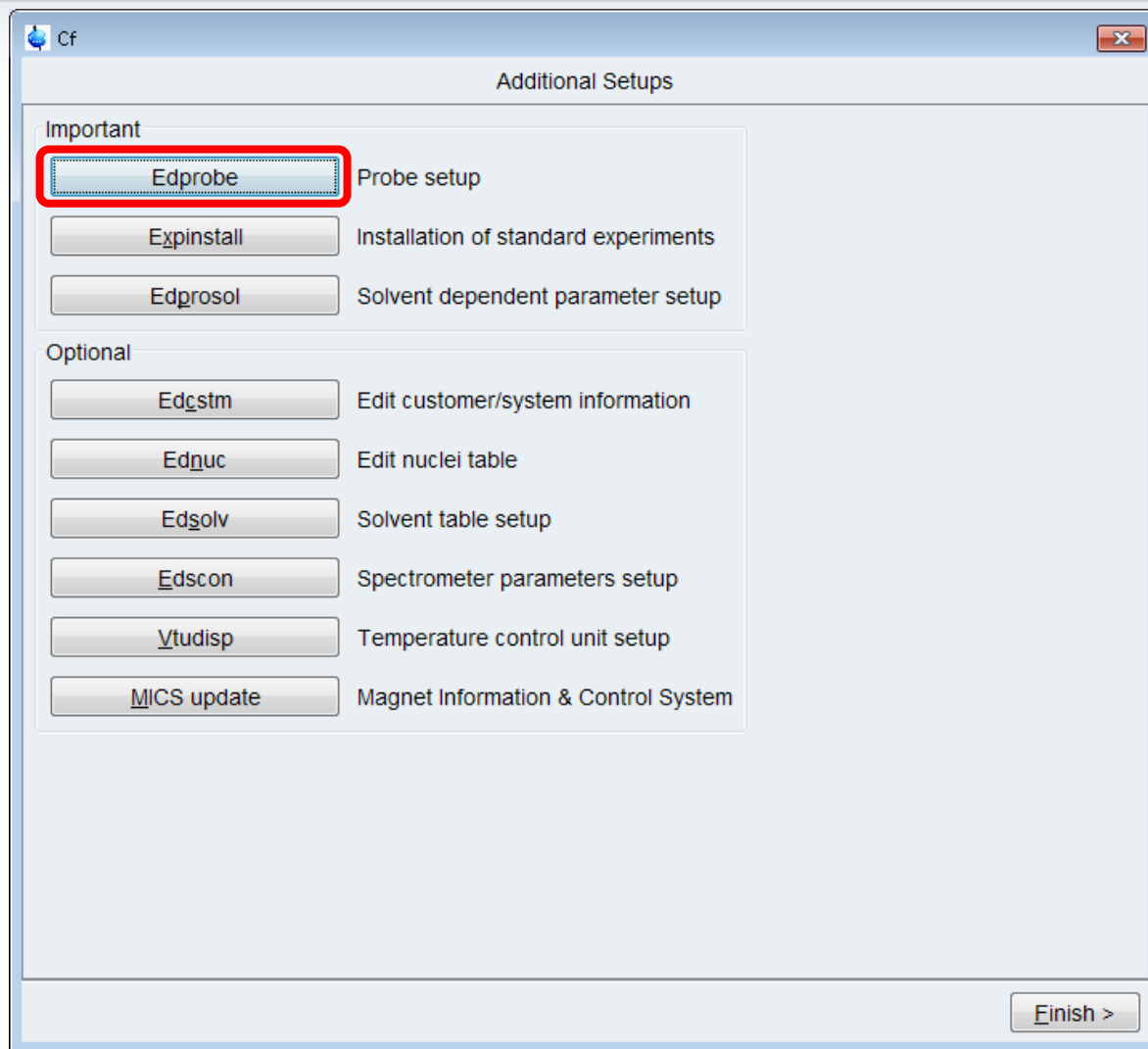
<TopSpin>\conf\instr\<spec>

Configure hardware [cf]



Internal routing usually set by service engineer.

Configure hardware [cf]



Edit Probe [edprobe]



Additional Setups

Important

Edprobe

Manage Help

Current probe: (Automatically detected)
Nickname: BBFOSP
Probe ID: Z116098_0002
Description: PA BBO 400S1 BBF-H-D-05 Z PLUS SP

Nickname	Probe ID	Description
MASBL4	H13383_0003	MAS
HR-MAS	B7110_0564	PH HRMAS 400S3 CHD 4G
CPPBBO	Z122623_0005	CPP BBO 400S1 BB-H&F-D-05 Z
BBI-2	Z157523_0001	PA BBI 400S1 H-BB-D-05 Z N
BBI	Z820201_0176	PA BBI 400S1 H-BB-D-05 Z
BBFOSP	Z116098_0002	PA BBO 400S1 BBF-H-D-05 Z PLUS SP
	H153169_0004	PI MAS-400-S1-4.0MM-BB/H
	K3166_0118	PH MAS 400SB BL4 N-P/H VTN

Edit Properties Edit RF Connections Set as current Delete Close

Finish >

Current probe usually automatically detected.

Edit Probe [edprobe]



Additional Setups

Important

Edprobe
Manage Help

edprobe: Edit RF Connections

Amplifier Preamplifier Probe: Z116098_0002

X 300 W

1H 100 W

X 300 W

2H 150 W

1H

2H

19F

XBB19F 2HS

XBB19F 2HP

XBB19F 2HS

19F-109Ag

1H

2H

connector 1 on coil 1

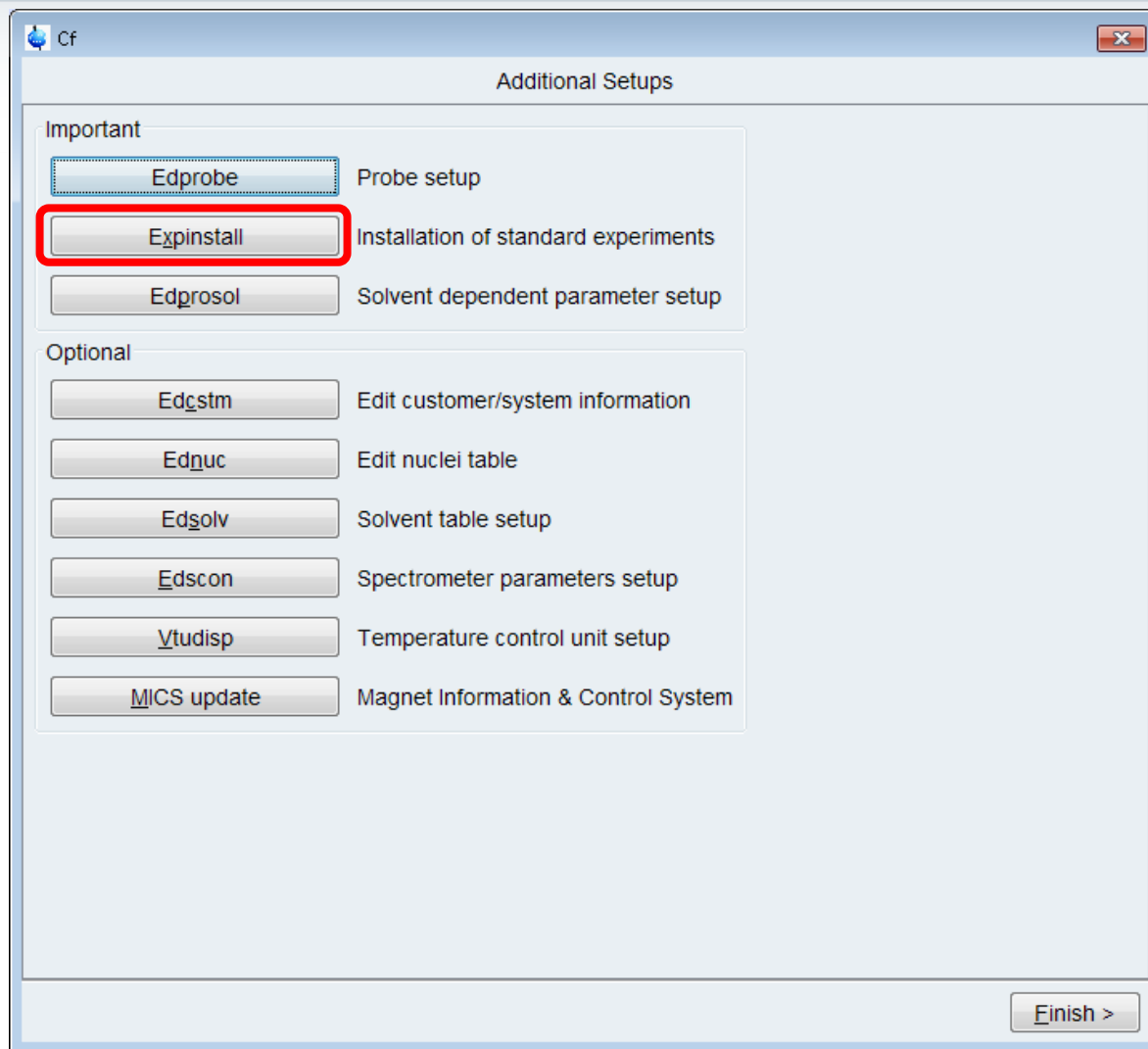
connector 1 on coil 2

connector 2 on coil 2

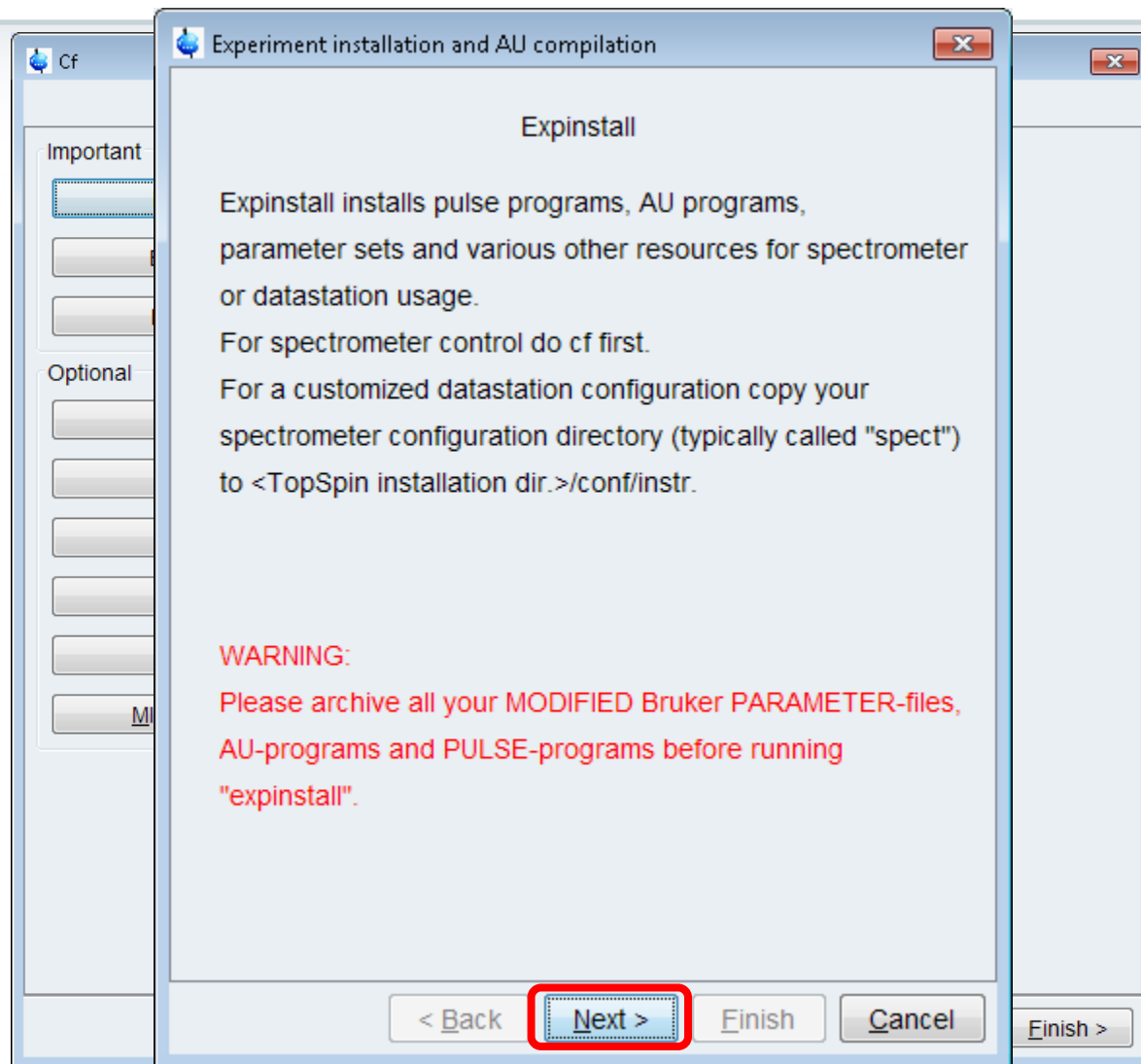
Save and Close Clear cable connections Info Param Close

Connections between preamplifier and probe.

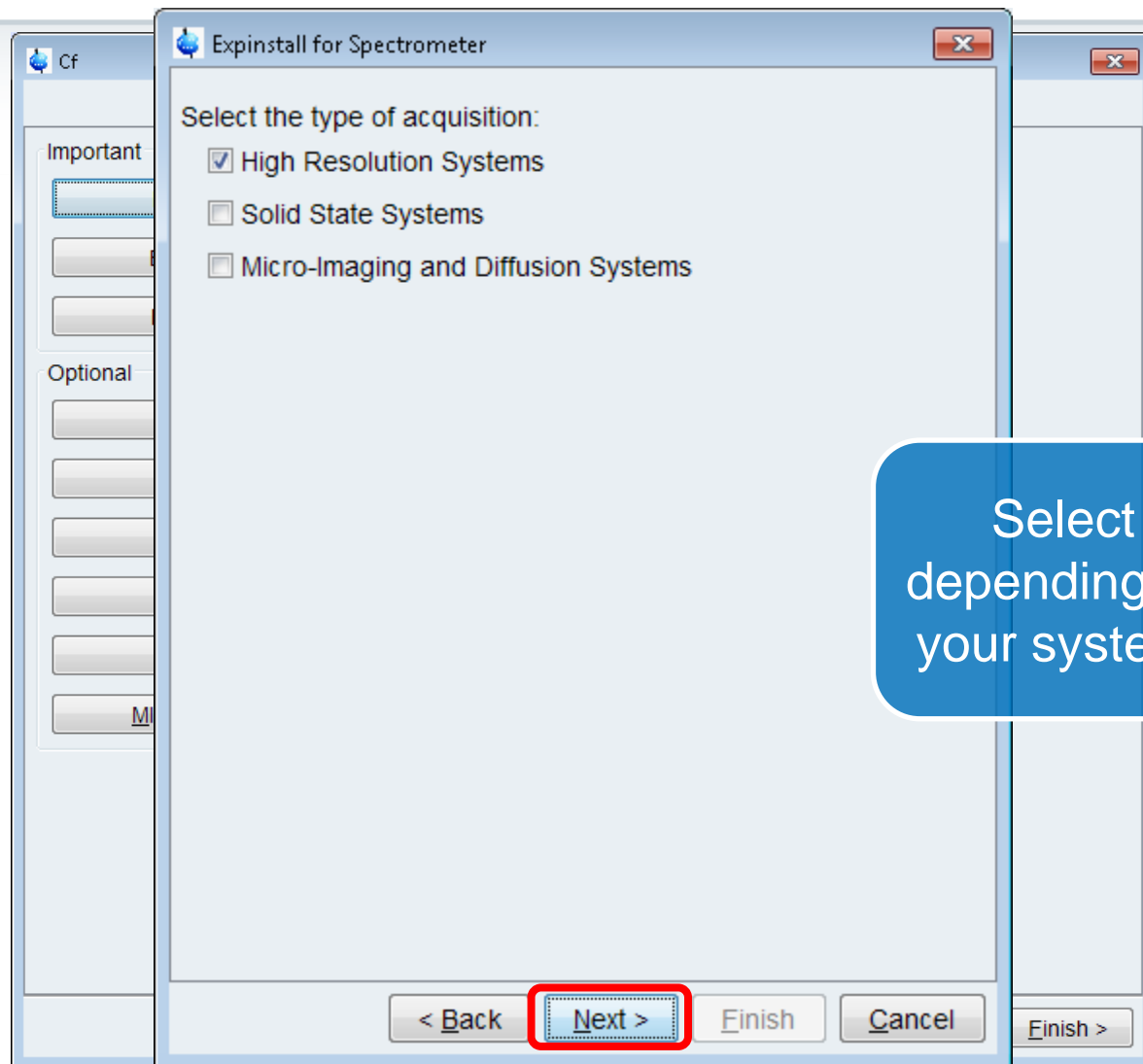
Configure hardware [cf]



Install Standard Experiments [**expinstall**]

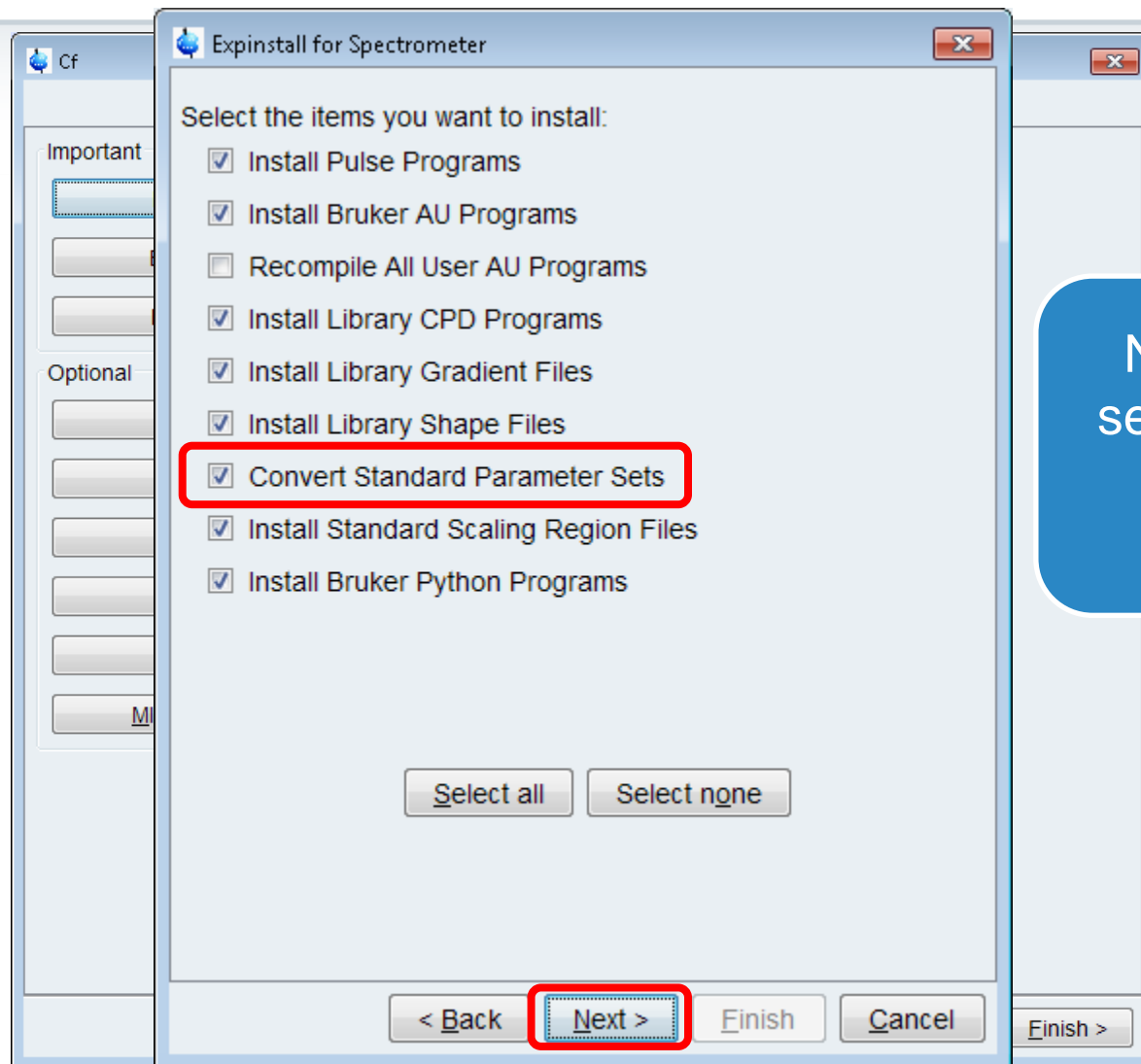


Install Standard Experiments [**expinstall**]



Select depending on your system.

Install Standard Experiments [**expinstall**]



Needs to be selected when routing is changed

Install Standard Experiments [**expinstall**]



Expinstall for Spectrometer

Select the basic frequency of your spectrometer:

Basic frequency (MHz):

Select the pre-scan-delay DE:

Default pre-scan-delay (μ s):

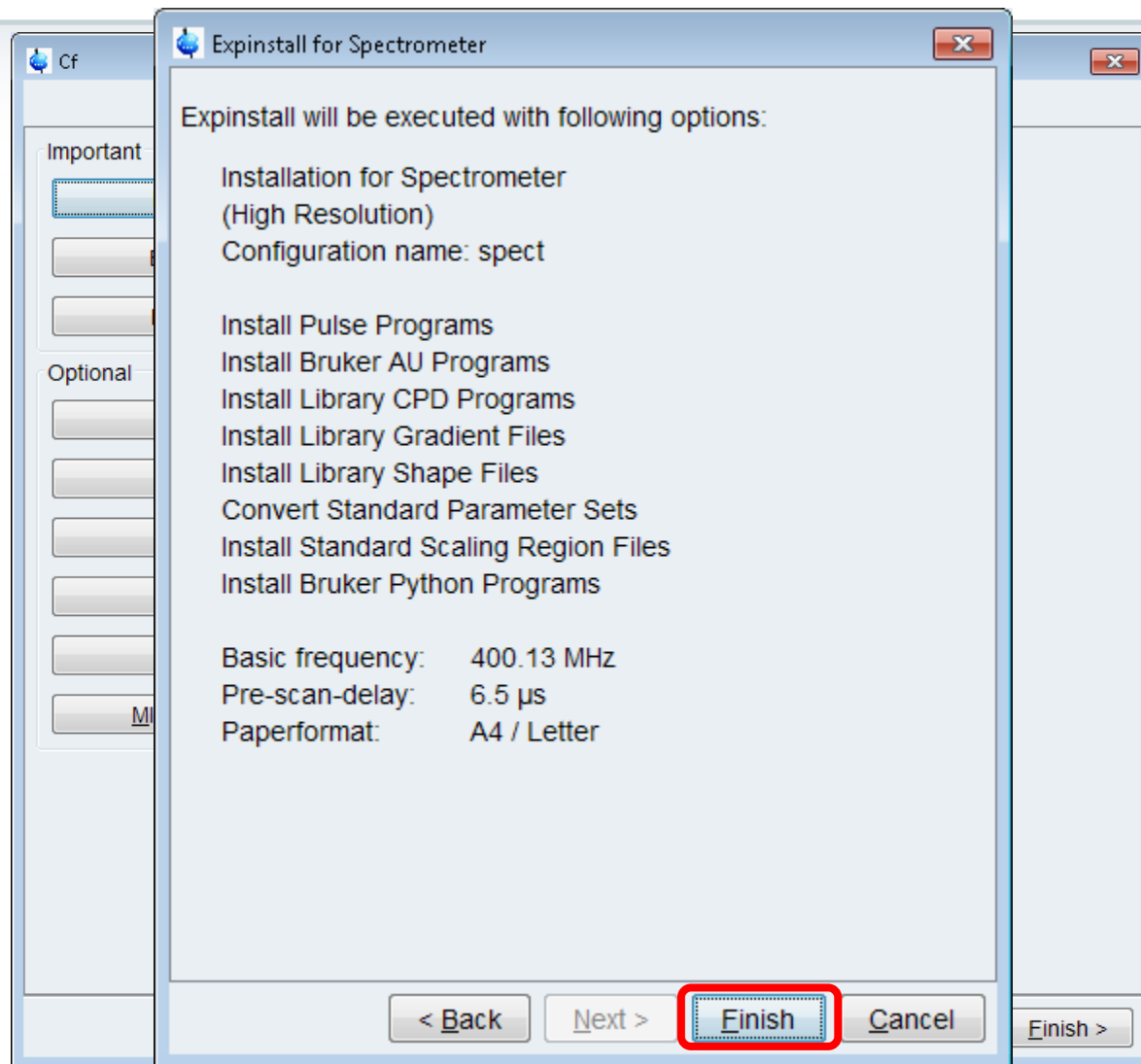
Select the plotter paper format:

Paper format:

< Back **Next >** Finish Cancel Finish >

The image shows a software dialog box titled "Expinstall for Spectrometer". It contains three sections for configuration: "Select the basic frequency of your spectrometer:" with a text input field containing "400.13"; "Select the pre-scan-delay DE:" with a text input field containing "6.5"; and "Select the plotter paper format:" with a dropdown menu showing "A4 / Letter". At the bottom, there are five buttons: "< Back", "Next >" (highlighted with a red dashed border), "Finish", "Cancel", and "Finish >".

Install Standard Experiments [**expinstall**]



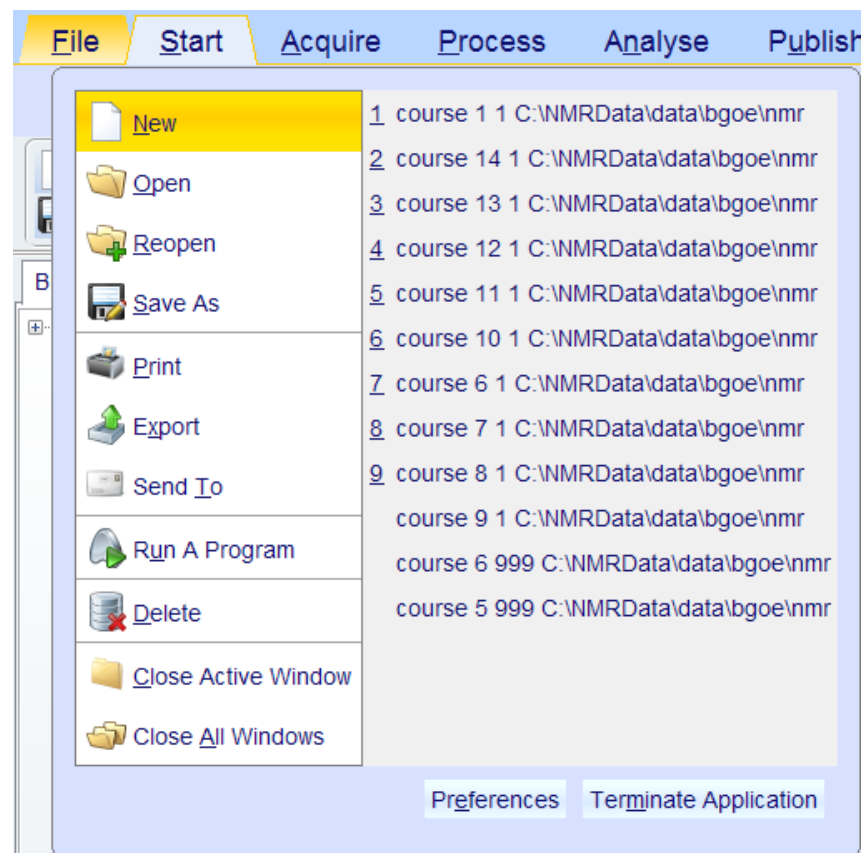
- **How to create a new data set?**

Create new data set [new/edc]



[new]

[edc]



Create new data set [**new**/**edc**]



The screenshot shows the Bruker software interface with the 'Create New Dataset - new' dialog box open. The dialog box contains the following fields and options:

- NAME:** Avance_Training
- EXPNO:** 1
- PROCNO:** 1
- Use current parameters
- Experiment **Select** (highlighted with a red box)
- Options:**
 - Set solvent: DMSO
 - Execute 'getprosol'
 - Keep parameters: P 1, PLW 1 **Change**
 - DIR:** C:\NMRData
 - Show new dataset in new window
 - Number of additional datasets: (1,2, ...16) 1
- TITLE:**

At the bottom of the dialog box are buttons for **OK**, **Cancel**, **More Info...**, and **Help**.

Experimental parameters are saved in parameter sets



The screenshot shows the Bruker software interface. The 'Create New Dataset - new' dialog box is open, with the 'NAME' field set to 'Avance_Training' and 'EXPNO' set to '1'. Below it, the 'File Options' dialog box is open, showing a list of experiment types. The 'Show Recommended' checkbox is checked and highlighted with a red box. The 'PROTON' experiment type is selected in the list. At the bottom of the 'File Options' dialog, the 'Set selected item in editor' button is also highlighted with a red box. To the right, there are two yellow callout boxes with '1H 13C Read Pars.' text, connected by red lines to the 'Create New Dataset' dialog.

File Options Help Source = C:\Bruker\TopSpin3.5pl7\exp\stan\nmr\par

Find file names enter any string, *, ? Exclude: Clear

Class = Any Dim = Any Show Recommended

Type = Any SubType = Any SubTypeB = Any Reset Filters

C13CPD	C13DEPT135	C13DEPTQ135	C13UDEFT	COSYGPDPFPHSW
COSYGPSW	HMBCETGPL3ND	HMBCGP	HMBCGP_15N	HSQC_TOCSY
HSQC_TOCSY_ADIA	HSQCEDETGPSISP	HSQCEDETGPSISP_ADIA	HSQCETGP_15N	HSQCETGPSISP
HSQCETGPSISP_ADIA	MLEVPHPR	MLEVPHSW	NOESYPHPR	NOESYPHSW
PROTON	ROESYPHPR	ROESYPHSW	WATERSUP	

Set selected item in editor Close

[rpar]

Experimental parameters are saved in parameter sets

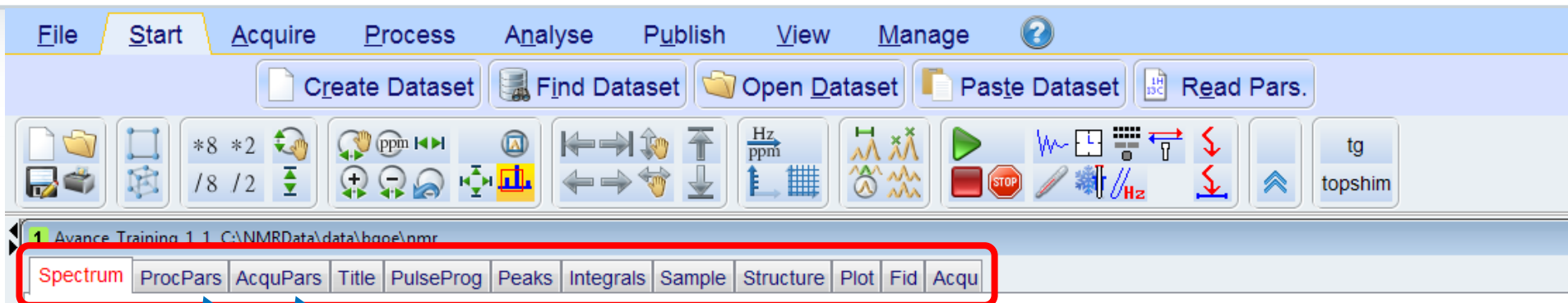


The screenshot displays the Bruker software interface with a "Create New Dataset - new" dialog box open. The dialog box contains the following fields and options:

- NAME:** Avance_Training
- EXPNO:** 1
- PROCNO:** 1
- Use current parameters
- Experiment: PROTON (with a "Select" button)
- Options:**
 - Set solvent: DMSO (dropdown menu)
 - Execute 'getprosol'
 - Keep parameters: P 1, PLW 1 (dropdown menu) with a "Change" button
 - DIR: C:\NMRData (dropdown menu)
 - Show new dataset in new window
 - Number of additional datasets: (1,2, ...16): 1
- TITLE:** (empty text box)

The "OK" button at the bottom of the dialog box is highlighted with a red square.

Data set tabs



All parameters related to the **acquisition** of data

All parameters related to the **processing** of data

No raw data available
No processed data available

Acquisition parameters [eda]



1 Avance_Training_1 C:\NMRData

Spectrum ProcPars **AcquPars** Title PulseProg Peaks Integrals Sample Structure Plot Fid Acq

Probe: BBFOSP

Parameter	Value	Description
Experiment		
PULPROG	zg30	Current pulse program
AQ_mod	DQD	Acquisition mode
TD	65536	Size of fid
DS	2	Number of dummy scans
NS	16	Number of scans
TD0	1	Loop count for 'td0'
Width		
SW [ppm]	20.0254	Spectral width
SWH [Hz]	8012.820	Spectral width
AQ [sec]	4.0894465	Acquisition time
FIDRES [Hz]	0.244532	Fid resolution
FW [Hz]	4032000.000	Filter width
Receiver		
RG	32	Receiver gain
DW [usec]	62.400	Dwell time
DWOV [usec]	0.025	Oversampling dwell time
DECIM	2496	Decimation rate of digital filter
DSPFIRM	sharp(standard)	DSP firmware filter
DIGTYP	DRU	Digitizer type
DIGMOD	digital	Digitization mode

Not all parameters are needed for each experiment.

Shorter list with used parameters available!

Short list can be shown with:



Acquisition parameters – short list [ased]



1 Avance_Training_1 1 C:\NMRData

Spectrum ProcPars **AcquPars** Title PulseProg Peaks Integrals Sample Structure Plot Fid Acqu

Probe: BBFOSP

General
Channel f1

PULPROG	zg30	...	E	Pulse program for acquisition
TD	65536			Time domain size
SWH [Hz, ppm]	8012.82	20.0254		Sweep width
AQ [sec]	4.0894465			Acquisition time
RG	32			Receiver gain
DW [µsec]	62.400			Dwell time
DE [µsec]	6.50			Pre-scan-delay
D1 [sec]	1.000000000			Relaxation delay; 1-5 * T1
DS	2			Number of dummy scans
NS	16			1 * n, total number of scans: NS * TD0
TD0	1			Number of averages in 1D
Channel f1				
SFO1 [MHz]	400.1324708			Frequency of ch. 1
O1 [Hz, ppm]	2470.80	6.175		Frequency of ch. 1
NUC1	1H	Edit...		Nucleus for channel 1
P1 [µsec]	10.000			F1 channel - 90 degree high power pulse
PLW1 [W, dB]	0	1000.00		F1 channel - power level for pulse (default)

Parameters that are shown in the short list are defined by the pulse program.

Complete list can be shown with:



Acquisition parameters



```
zg30 (C:\Bruker\TopSpin3.5pl7\exp\stan\nmr\lists\app)
File Edit Search
Graphical_Edit Set PULPROG
1 ;zg30
2 ;avance-version (12/01/11)
3 ;1D sequence
4 ;using 30 degree flip angle
5 ;
6 ;$CLASS=HighRes
7 ;$DIM=1D
8 ;$TYPE=
9 ;$SUBTYPE=
10 ;$COMMENT=
11 ;$RECOMMEND=y
12
13
14 #include <Avance.incl>
15
16
17 "acqt0=-p1*0.66/3.1416"
18
19
20 1 ze
21 2 30m
22 d1
23 p1*0.33 ph1
24 go=2 ph31
25 30m mc #0 to 2 F0(zd)
26 exit
27
28
29 ph1=0 2 2 0 1 3 3 1
30 ph31=0 2 2 0 1 3 3 1
31
32
33 ;p1 : f1 channel - power level for pulse (default)
34 ;p1 : f1 channel - 90 degree high power pulse
35 ;d1 : relaxation delay; 1-5 * T1
36 ;ns: 1 * n, total number of scans: NS * TD0
37
```

Only parameters
that are
mentioned here
will be used.

Channel Routing [edasp]



1 Avance_Training 1 1 C:\NMRD...

Spectrum ProcPars **AcquPars** Title PulseProg Peaks Integrals Sample Structure Plot Fid Acqu

Probe: BBFOSP

General Channel f1

General

PULPROG	zg30	E	Pulse program for acquisition
TD	65536		Time domain size
SWH [Hz, ppm]	8012.82	20.0254	Sweep width
AQ [sec]	4.0894465		Acquisition time
RG	32		Receiver gain
DW [µsec]	62.400		Dwell time
DE [µsec]	6.50		Pre-scan-delay
D1 [sec]	1.000000000		Relaxation delay; 1-5 * T1
DS	2		Number of dummy scans
NS	16		1 * n, total number of scans: NS * TD0
TD0	1		Number of averages in 1D

Channel f1

SFO1 [MHz]	400.1324708		Frequency of ch. 1
O1 [Hz, ppm]	2470.80	6.175	Frequency of ch. 1
NUC1	1H	Edit...	Nucleus for channel 1
P1 [µsec]	10.000		F1 channel - 90 degree high power pulse
PLW1 [W, dB]	0	1000.00	F1 channel - power level for pulse (default)

Routing of the spectrometer is saved in each parameter set.

Can be opened with [edasp] or



Channel Routing for 1D ¹H experiment [edasp]



Channel Routing

Frequency	Logical Channel	Amplifier	Preamplifier	Receiver	Observe Channel
BF1 400.13 MHz SFO1 400.132471 MHz OFS1 2470.8 Hz	NUC1 F1 1H	SGU1 X 300 W 1H 100 W	1H 2H 19F	REC1 SGU1	NUC1 F1 1H
BF2 400.13 MHz SFO2 400.132471 MHz OFS2 2470.8 Hz	NUC2 F2 off	SGU2 X 300 W	XBB19F 2HS XBB19F 2HP	REC2 SGU2	NUC2 F2 off
BF3 400.13 MHz SFO3 400.132471 MHz OFS3 2470.8 Hz	NUC3 F3 off	SGU3 2H 150 W	XBB19F 2HS	SGU3	NUC3 F3 off

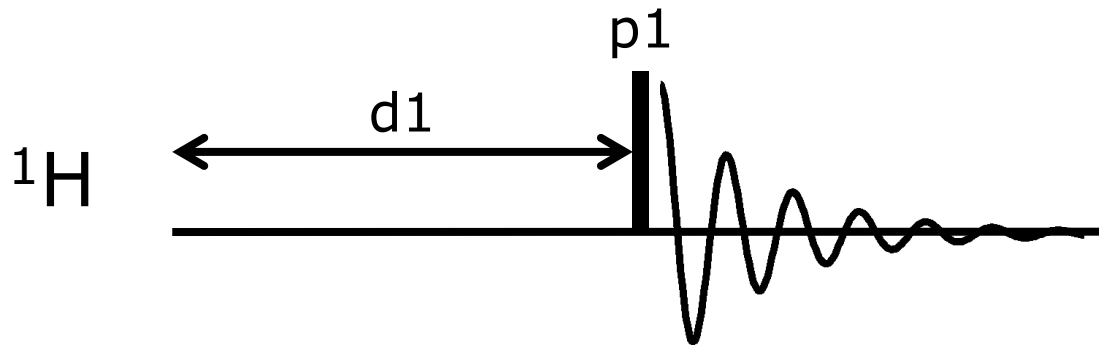
— : cable wiring
- - : possible RF routing
● : cortab available

settings

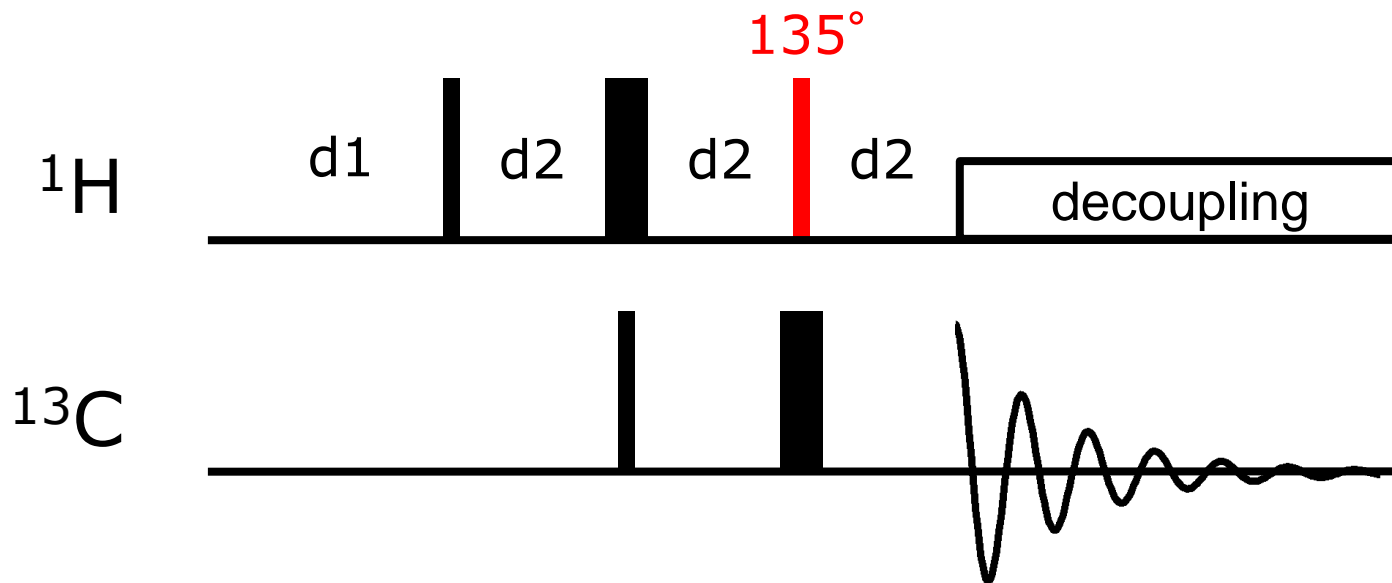
- show selected routing
- show receiver wiring
- show probe wiring
- show receiver routing
- show RF routing
- show power at probe in

2 Save and Close Switch F1/F2 Switch F1/F3 Add logical channel Remove logical channel Default 1 Info Param Close

Channel Routing for 1D ^1H experiment



Channel Routing for 1D ^{13}C experiment with proton decoupling



Channel Routing for 1D ^{13}C experiment with proton decoupling



Frequency	Logical Channel	Amplifier	Preamplifier	Receiver	Observe Channel
BF1 100.612769 MHz SFO1 100.620818 MHz OFS1 8049.02 Hz	NUC1 F1 13C	SGU1 X 300 W	1H 2H	REC1 SGU1	NUC1 F1 13C
BF2 400.13 MHz SFO2 400.131601 MHz OFS2 1600.52 Hz	NUC2 F2 1H	SGU2 1H 100 W X 300 W	19F XBB19F 2HS XBB19F 2HP	REC2 SGU2	NUC2 F2 1H
BF3 400.13 MHz SFO3 400.138049 MHz OFS3 8049.02 Hz	NUC3 F3 off	SGU3 2H 150 W	XBB19F 2HS	SGU3	NUC3 F3 off

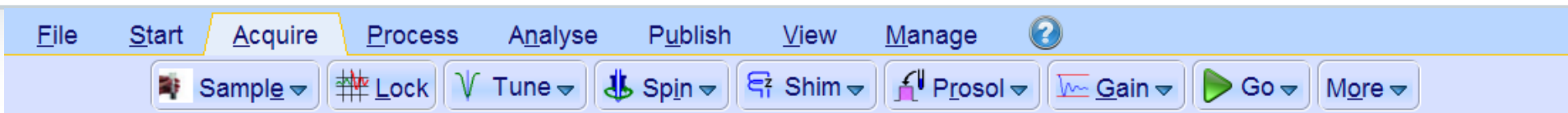
Legend:
— : cable wiring
- - - : possible RF routing
● : cortab available

settings:
 show selected routing show receiver routing
 show receiver wiring
 show probe wiring
 show RF routing show power at probe in

Buttons: Save and Close (2), Switch F1/F2, Switch F1/F3, Add logical channel, Remove logical channel, Default (1), Info, Param, Close

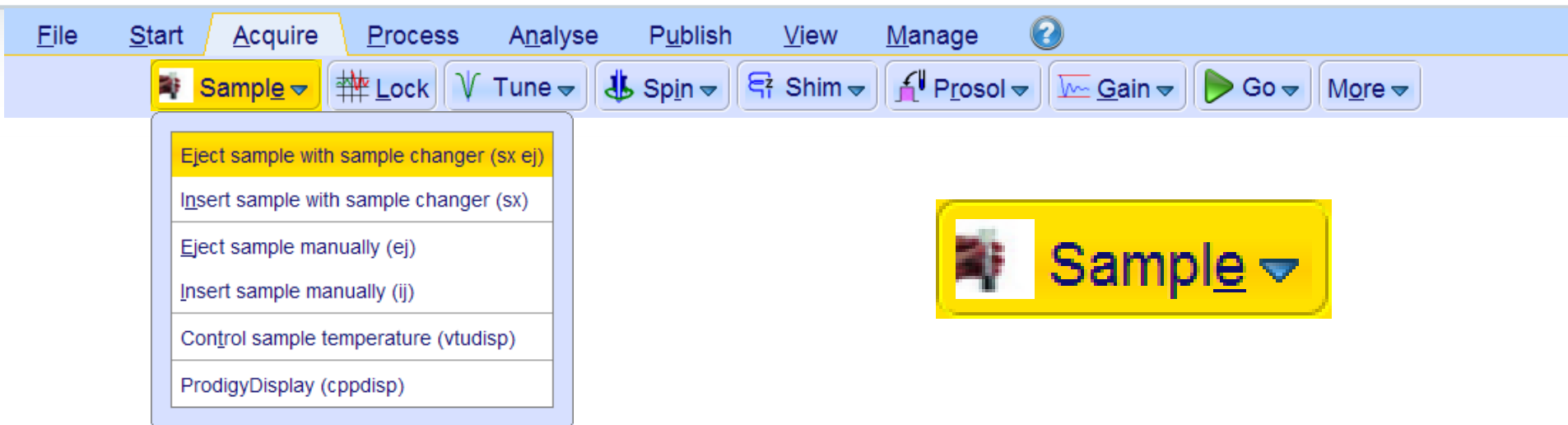
- **How to acquire a spectrum?**

Acquire Toolbar

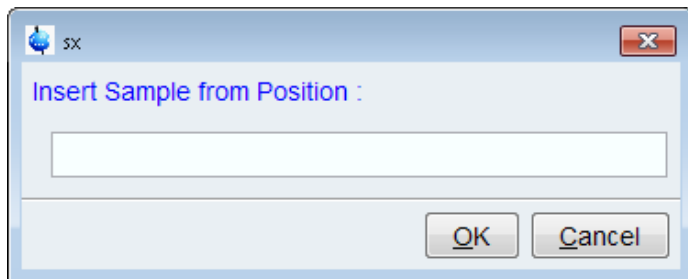


All steps that are needed to acquire a spectrum can be done one after another from left to right.

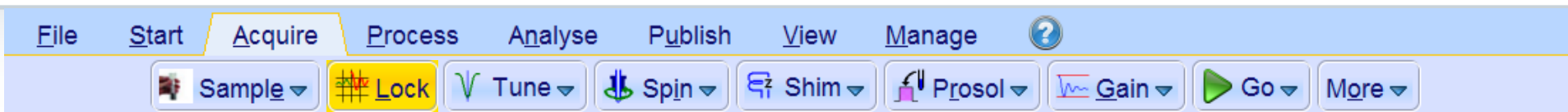
Acquire Toolbar – Sample



- Eject [**ej**] or insert [**ij**] sample manually
- If you have a sample changer you have to use [**sx ej**] to eject the sample and [**sx**] or [**sx <number>**] to insert the sample .



Acquire Toolbar – Lock



Δ Solvent	Description
Acetic	acetic acid-d4
Acetone	acetone-d6
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
D2O_salt	deuteriumoxide with salt
Dioxane	dioxane-d8
DMF	N,N-dimethylformamide-d7
DMSO	dimethylsulfoxide-d6
EtOD	ethanol-d6
H2O+D2O	90%H2O and 10%D2O
H2O+D2O_salt	90%H2O and 10%D2O with salt
HDMSO	90%DMSO and 10%DMSO-d6
Juice	fruit juice

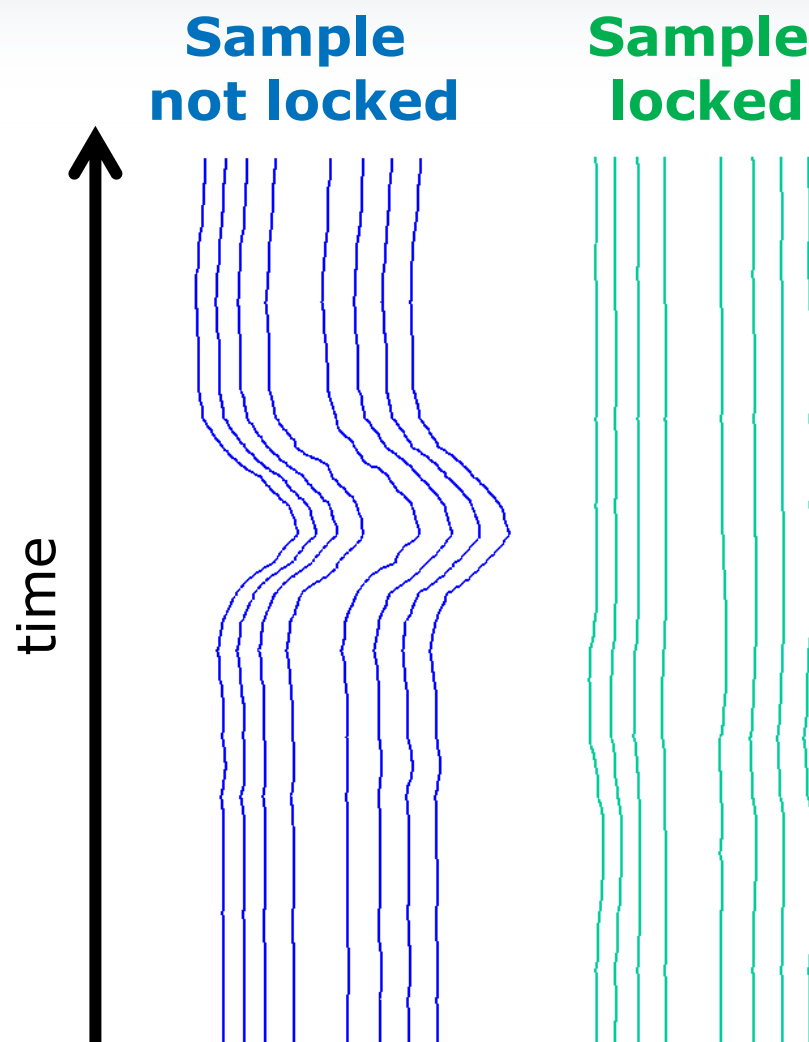


- [**lock**] or directly [**lock <solvent>**]
- Locks the spectrometer frequency to the deuterium signal of the solvent
- Eliminates influence of field drift and reduces influence of disturbances

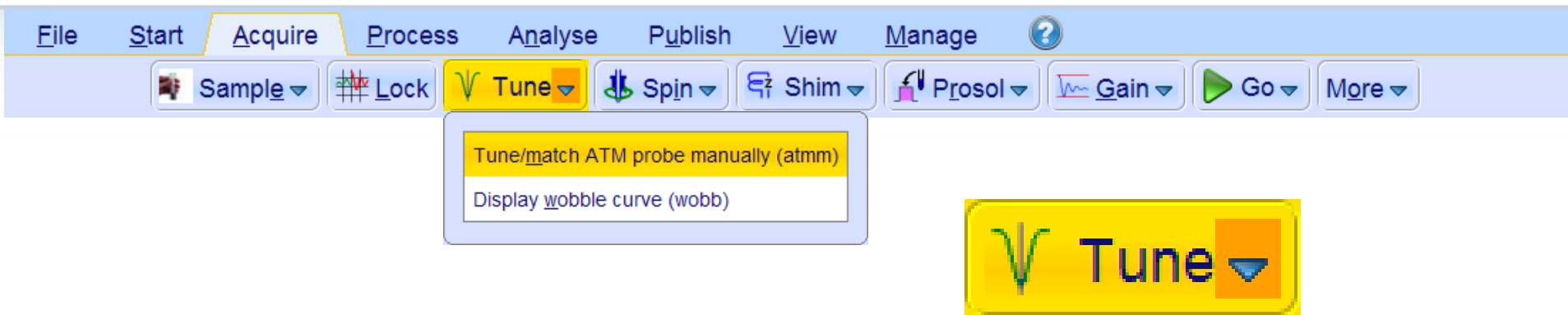
Why lock?



- The lock is in principle an extra spectrometer
- Monitors deuterium frequency continuously
- Adjusts field to compensate disturbances

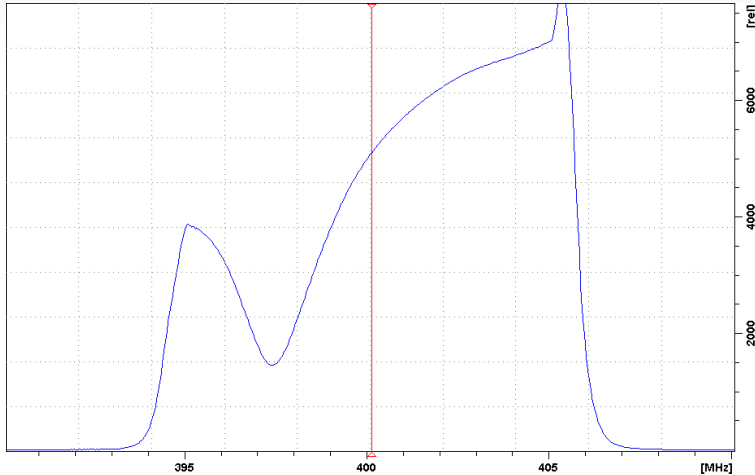


Acquire Toolbar – Tune



- Probe need to be tuned at matched to be most sensitive
- Automatic tuning and matching [**atma**]
- Can be tuned and matched manually as well [**atmm**]
- Needs to be done for each nucleus and when sample matrix is changed (different solvent, salty sample).

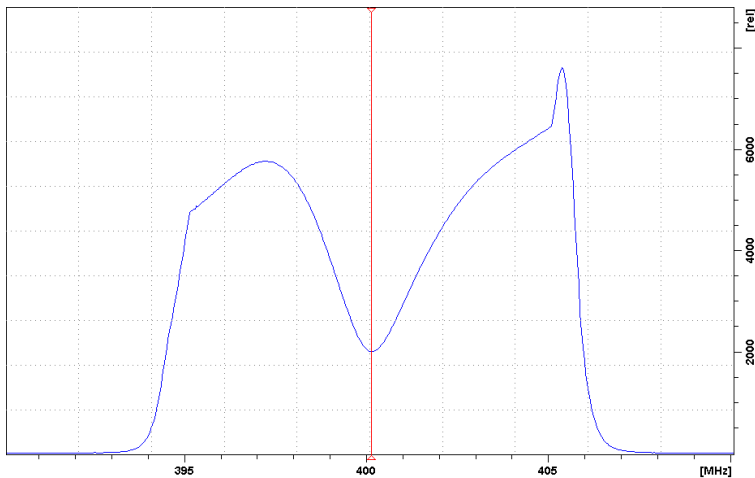
Tuning and Matching



Tuning:



Matching:



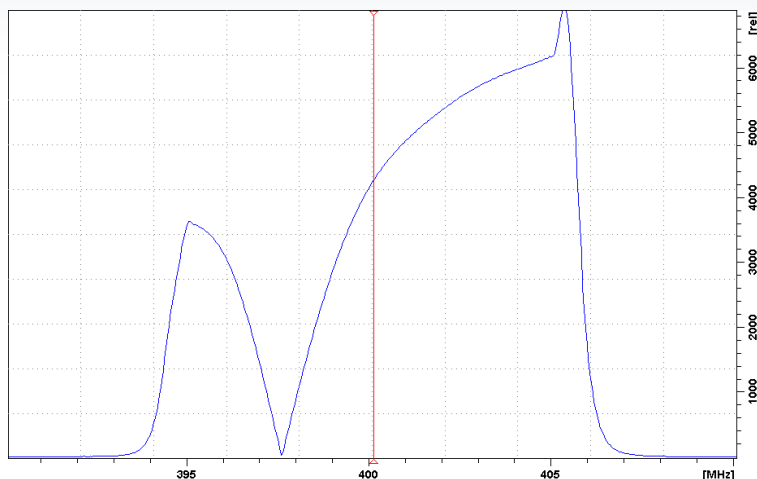
Tuning:



Matching:



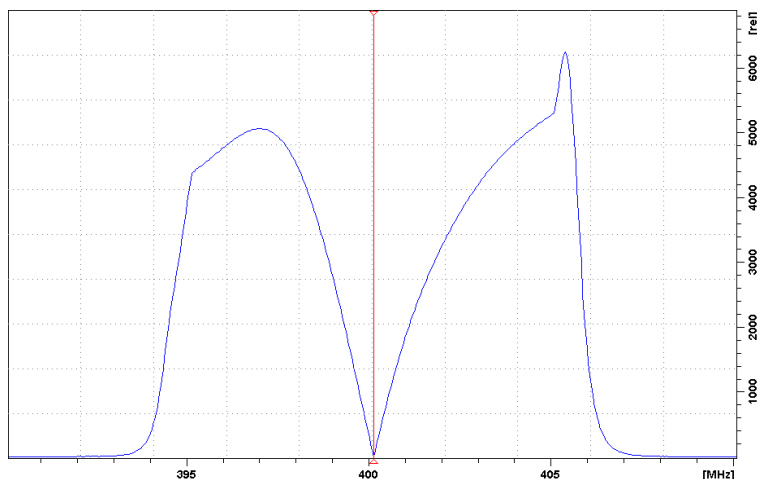
Tuning and Matching



Tuning:



Matching:



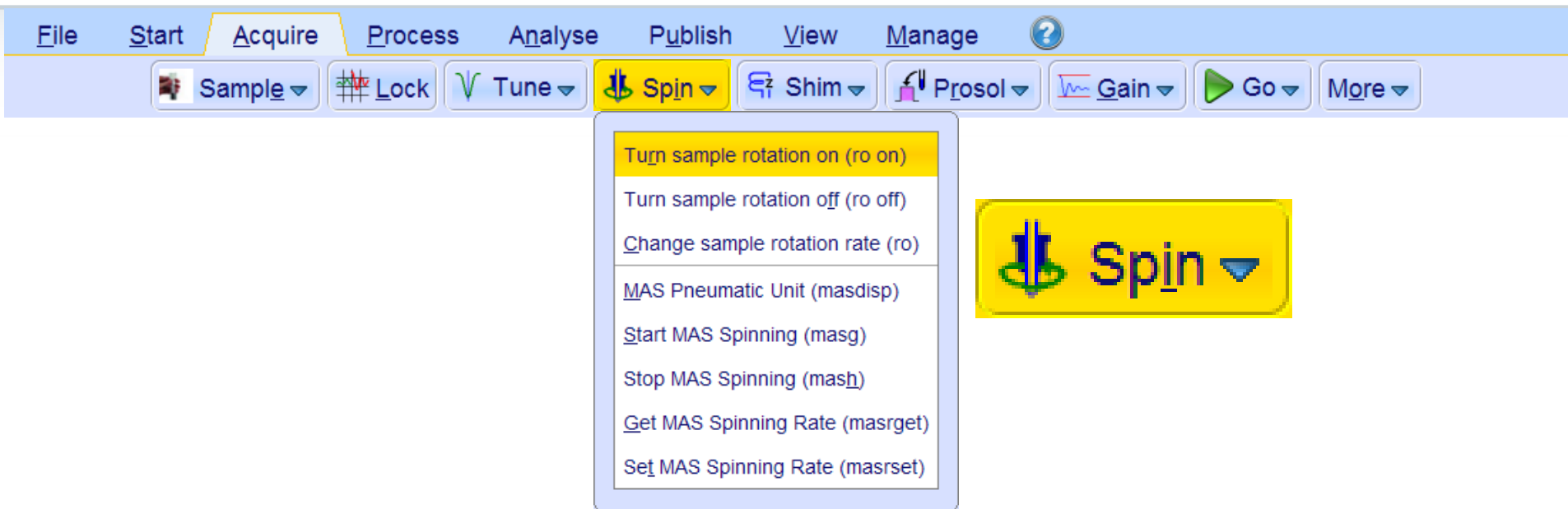
Tuning:



Matching:



Acquire Toolbar – Spin



- Usually, sample rotation is not needed anymore
- Forbidden for some experiments (e.g. water suppression)
- MAS rotation for solids/semi-solids can be controlled as well

Acquire Toolbar – Shim



The screenshot displays the Bruker software interface with the 'Acquire' menu selected. The 'Shim' button in the toolbar is highlighted in yellow. A dropdown menu is open, showing various shim-related options. The option 'Open topshim graphical user interface (topshim gui)' is highlighted with a red border. Below the main menu, a larger yellow button labeled 'Shim' is also shown.

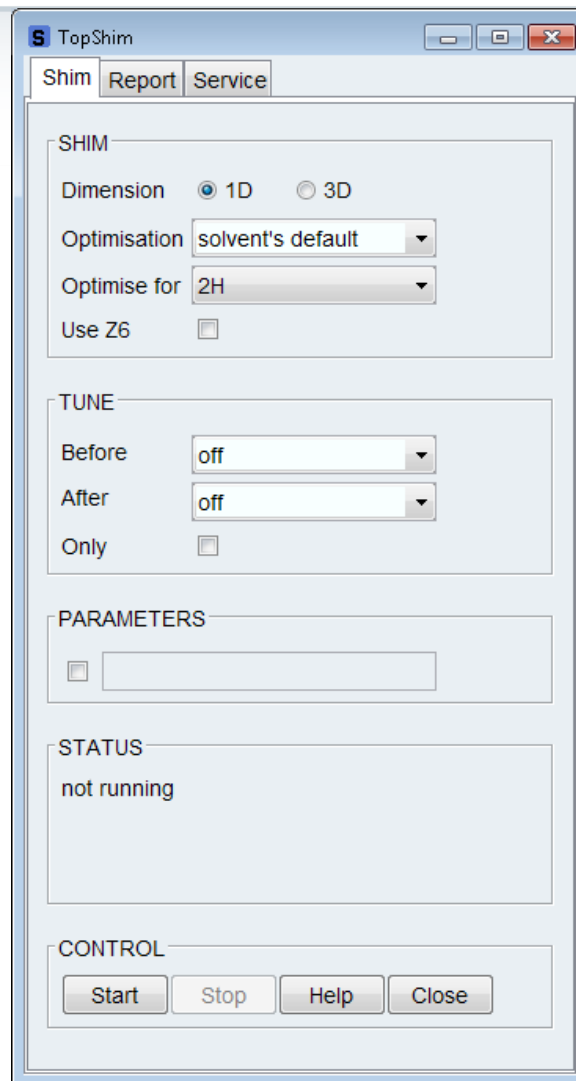
Menu Item	Command
Display topshim report (topshim report)	Open topshim graphical user interface (topshim gui)
Additional topshim options	Stop topshim optimization (topshim stop)
Shim manually using BSMS panel (bsmsdisp)	3D topshim (H2O sample) (topshim 3d)
Traditional gradient shimming (gradshim)	Tune shim after topshim (topshim tunea)
Set Shim Values (setshim)	Run topshim unlocked (topshim lockoff)
Read shim values (rsh)	
Write shim values (wsh)	
View Shim Values (vish)	
Delete Shim File (delsh)	
Autoshim using tune file (tune)	
Autoshim using tune file for current probe (tune .sx)	
Edit automshim definition (tune) file (edtune)	

- Each sample needs to be shimmed for best homogeneity
- Automatic shimming with [**topshim**]

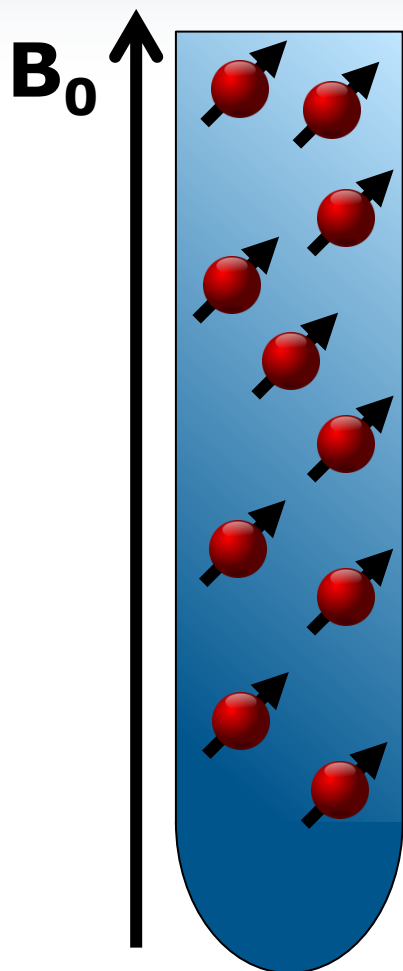
Topshim GUI



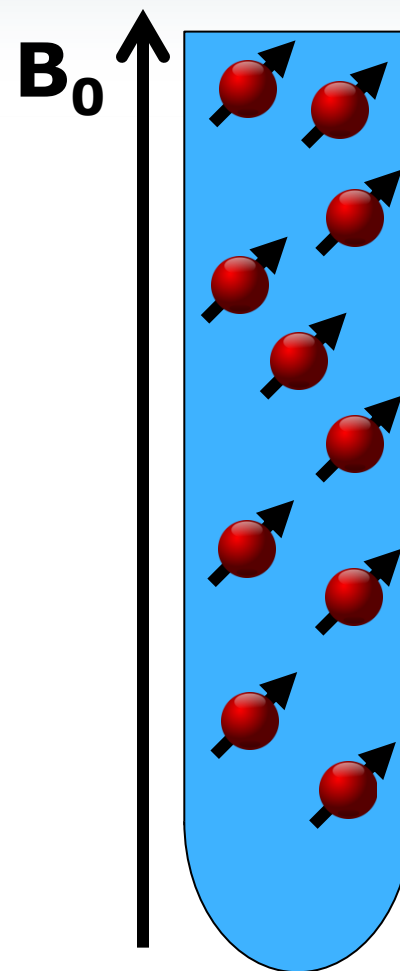
- Different options for shimming can be selected
- Standard 1D shimming is along z
- 3D shimming is only available for non-deuterated water as solvent (90% H₂O + 10% D₂O)



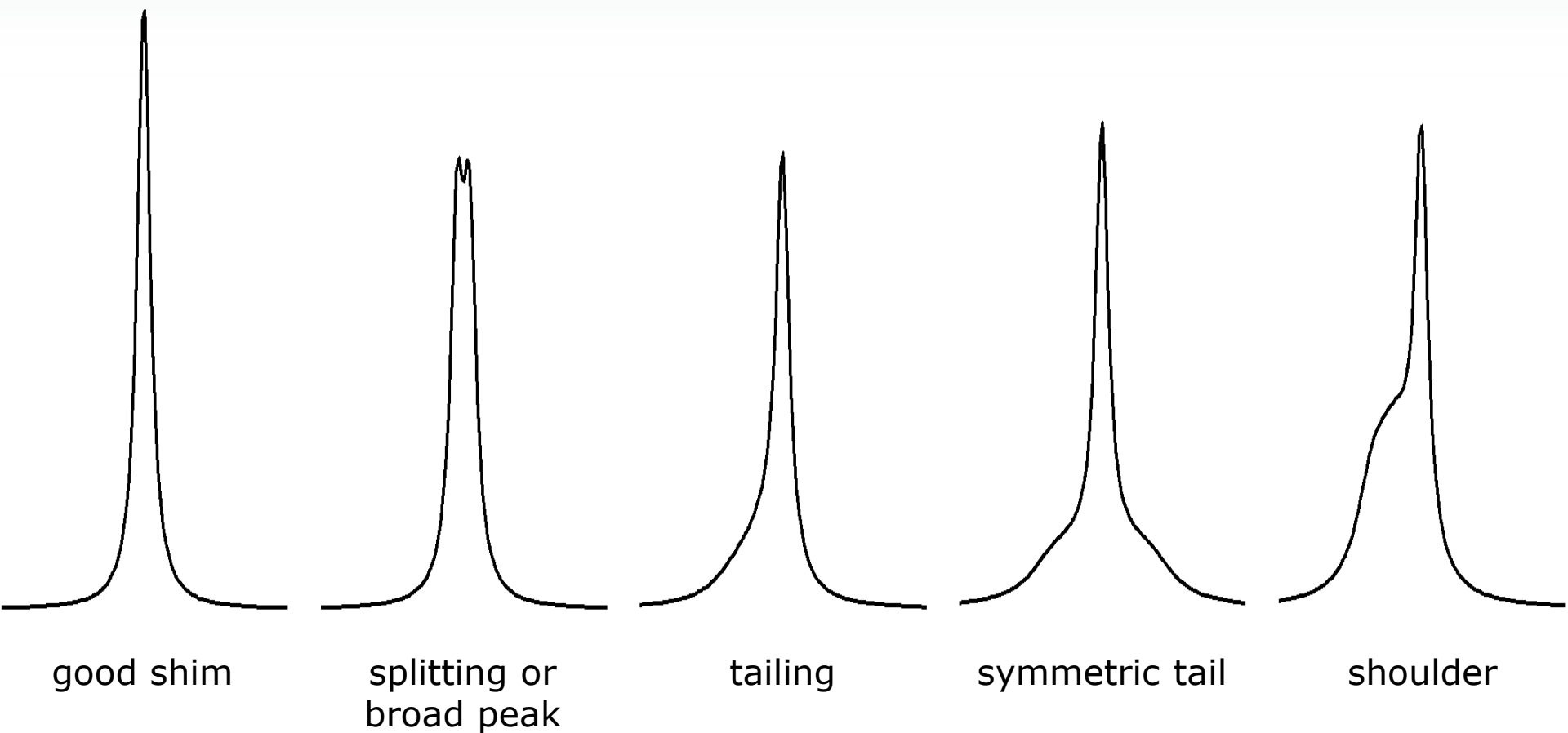
Why shimming?



- Magnetic field strength determines resonance frequency
- External magnetic field not 100% homogeneous
- External field: $\sim 500\text{MHz}$
- Resolution: $< 1\text{Hz}$
- Additional magnetic fields applied by shim coils



What is a good shim?



good shim

splitting or
broad peak

tailing

symmetric tail

shoulder

More information about shimming:

G. Chmurny, D. Hoult, "The ancient and honorable art of shimming." *Concep. Magnetic Res.*, **1990**, 2, 131-149.

Acquire Toolbar – Prosol



- Copies probe & solvent specific parameters to data set



- It is possible to execute getprosol with a specific pulse
[getprosol <nuc> <pulse length> <pulse power in dB>]

Prosol table [edprosol]



edprosol

File Edit View Help

Saved Observe and Saved Decouple Prosol Parameter Set for:

Probe: BBFOSP Z116098_0002 PA BBO 400S1 BBF-H-D-05 Z PLUS SP Solvent: generic

Observe: Nucleus: Decouple:

Observe Comment: Decouple Comment:

90 deg. Pulses | HR Square Pulses | HR Shape Pulses | Others

Observe				Decouple			
Nucleus	Pulse Width[μs]	Power[W]	Set	Pulse Width[μs]	Power[W]	Set	Nucleus
1H	11.00	16.911		11.00	16.911		1H
2H	300.00	5.9113		300.00	5.9113		2H
3He	0.00	0.0000		0.00	0.0000		3He
7Li	0.00	0.0000		0.00	0.0000		7Li
10B	0.00	0.0000		0.00	0.0000		10B
11B	8.00	100.00		0.00	0.0000		11B
13C	10.00	80.929		10.00	80.929		13C
14N	0.00	0.0000		0.00	0.0000		14N
15N	21.00	80.455		21.00	80.455		15N
17O	10.00	100.00		0.00	0.0000		17O
19F	18.00	19.036		18.00	19.036		19F
21Ne	0.00	0.0000		0.00	0.0000		21Ne
23Na	0.00	0.0000		0.00	0.0000		23Na
25Mg	0.00	0.0000		0.00	0.0000		25Mg

Prosol table [edprosol]



edprosol

File Edit View Help

Saved Observe and Saved Decouple Prosol Parameter Set for:

Probe: Solvent:

Observe: Nucleus:

Observe Comment: Decouple Comment:

90 deg. Pulse:

	Observe					Decouple				
	α [°]	RFF[Hz]	PuW[μ s]	Pw[W]	#	α [°]	RFF[Hz]	PuW[μ s]	Pw[W]	
cpd	90.00	2777.78	90.00	0.25262	0	cpd	90.00	2777.78	90.00	0.25262
TOCSY spinlock	90.00	8333.33	30.00	2.2736	1	TOCSY spinlock	90.00	8333.33	30.00	2.2736
ROESY spinlock (cw, RF field)	90.00	1923.08	130.00	0.12108	2	ROESY spinlock (cw, RF field)	90.00	1923.08	130.00	0.12108
presat. (cw irradiation, RF field)	90.00	25.00	10000.00	2.0462e-05	3	presat. (cw irradiation, RF field)	90.00	50.00	5000.00	8.1849e-05
					4	2nd cpd (power gated)	90.00	1970.06	126.90	0.12707
					5	low power cpd	90.00	1388.89	180.00	0.063155
					6	bilev cpd (cw part)	90.00	5555.56	45.00	1.0105
TOCSY/hetero T. (med. selection)	90.00	4807.69	52.00	0.75674	7	TOCSY/hetero T. (med. selection)	90.00	4807.69	52.00	0.75674
TOCSY/hetero T. (high selection)	90.00	3205.13	78.00	0.33633	8	TOCSY/hetero T. (high selection)	90.00	3205.13	78.00	0.33633
					9	TOCSY/hetero T. (very high selection)	90.00	666.67	375.00	0.014551
cleanex spinlock	90.00	4807.69	52.00	0.75674	10					
ROESY pulsed (90°)	90.00	1851.85	135.00	0.11228	11	ROESY pulsed (90°)	90.00	1851.85	135.00	0.11228
low power presat. (cw irradiation, RF field)	90.00	10.00	25000.00	3.2740e-06	12	low power presat. (cw irradiation, RF field)	90.00	10.00	25000.00	3.2740e-06
					13	NOE diff. irradiation (RF field)				

Prosol table [edprosol]



edprosol

File Edit View Help

Saved Observe and Saved Decouple Prosol Parameter Set for:

Probe: BBFOSP Z116098_0002 PA BBO 400S1 BBF-H-D-05 Z PLUS SP Solvent: generic

Observe: Nucleus:

Observe Comment: Default 1H obs 400 Decouple Comment: Default 1H dec 400

90 deg. Pulses | HR Square Pulse | **HR Shape Pulses** | Others

Observe								Decouple							
Filename	α [°]	RFF[Hz]	Ali	PuW[μ s]	Pw[W]	#		Filename	α [°]	RFF[Hz]	Ali	PuW			
selective excitation	Gaus1_270.1000	270.00	22.78	1.0	80000.00	1.6987e-05	0	selective excitation	Gaus1_270.1000	270.00	22.78	1.0	80		
selective refocussing	Gaus1_180r.1000	180.00	15.19	0.5	80000.00	7.5497e-06	1	select. inversion/refocussing	Gaus1_180r.1000	180.00	15.19	0.5	80		
bandsel. excitation	Q5.1000	90.00	458.63	1.0	10000.00	0.0068866	2	bandsel. excitation	Q5.1000	90.00	458.63	1.0	10		
bandsel. inv./refoc.	Q3.1000	180.00	330.08	0.5	10000.00	0.0035670	3	bandsel. inv./refoc.	Q3.1000	180.00	330.08	0.5	10		
off-resonance presat. (powe	Squa100.1000	90.00	2.50	0.5	100000.00	2.0462e-07	4								
90° flip back (H2O)	Sinc1.1000	90.00	424.52	0.5	1000.00	0.0059003	5	90° flip back (H2O)	Sinc1.1000	90.00	424.52	0.5	10		
2nd 90° flip back (H2O)	Sinc1.1000	90.00	106.13	0.5	4000.00	0.00036877	6								
90° WET	Sinc1.1000	90.00	21.23	1.0	20000.00	1.4751e-05	7								
120° NH region	Pc9_4_120.1000	120.00	740.72	1.0	3600.00	0.017963	8								
180° NH region I	Rsnob.1000	180.00	1949.81	0.5	1200.00	0.12447	9								
90° NH region I	Pc9_4_90.1000	90.00	606.04	1.0	3300.00	0.012025	10	90° NH region I	Q5.1000	90.00	1652.73	1.0	2		
90° NH region I timerev.	Pc9_4_90.1000	90.00	606.04	0.0	3300.00	0.012025	11	90° NH region I timerev.	Q5tr.1000	90.00	1652.73	0.0	2		
180° NH region II	Reburp.1000	180.00	2983.27	0.5	2100.00	0.29138	12	180° NH region II	Reburp.1000	180.00	2983.27	0.5	2		
00° NH region II	Eburn2.1000	90.00	1606.43	1.0	2550.00	0.004488	13	00° NH region II	Eburn2.1000	90.00	1606.43	1.0	2		

Last Save Print Copy to Solvent Copy to Probe Save

Prosol table [edprosol]



edprosol

File Edit View Help

Saved Observe and Saved Decouple Prosol Parameter Set for:

Probe: BBFOSP Z116098_0002 PA BBO 400S1 BBF-H-D-05 Z PLUS SP Solvent: generic

Observe: Nucleus:

Observe Comment: Decouple Comment:

90 deg. Pulses | HR Square Pulses | HR Shape Pulse | **Others**

Saved Prosol Parameters Depending on Probe and Observe Nucleus.				Saved Gradient Durations Depending on Probe only.			
Name	Value	Unit		Name	Value	Unit	
pre-scan delay DE	<input type="text" value="6.500000"/>	µsec		grad. recovery delay D_grad	<input type="text" value="0.000200"/>	sec	
trim pulse mlev P_mlev	<input type="text" value="2500.000000"/>	µsec		grad. pulse 1 P_grad1	<input type="text" value="1000.000000"/>	µsec	
trim pulse hsqc P_hsqc	<input type="text" value="1000.000000"/>	µsec		grad. pulse 2 P_grad2	<input type="text" value="600.000000"/>	µsec	
Name	Value	Unit		Name	Value	Unit	

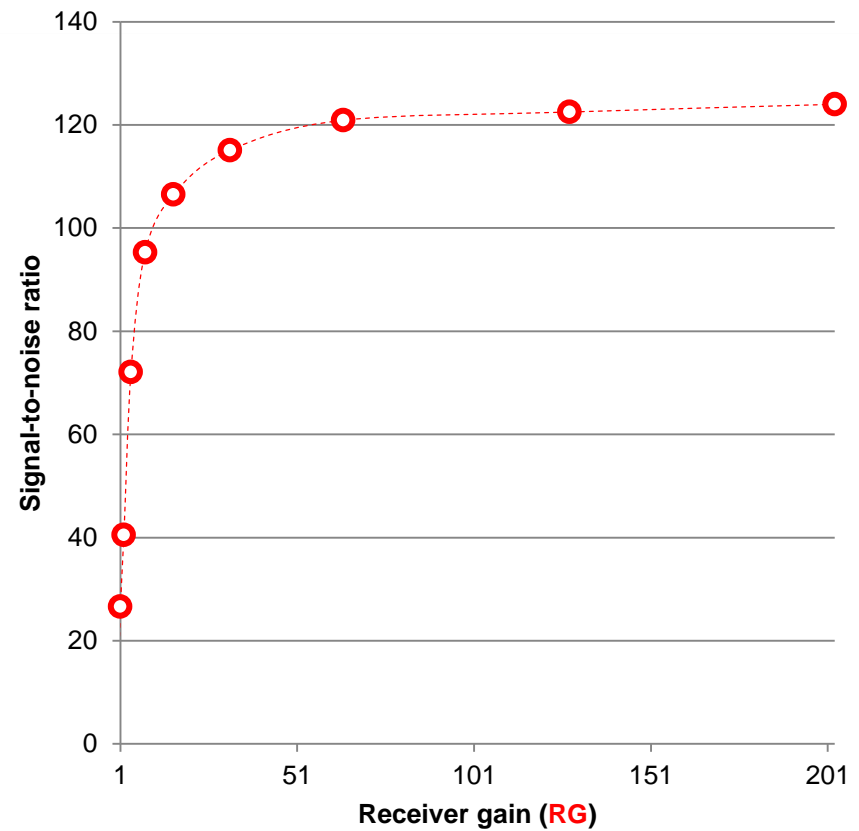
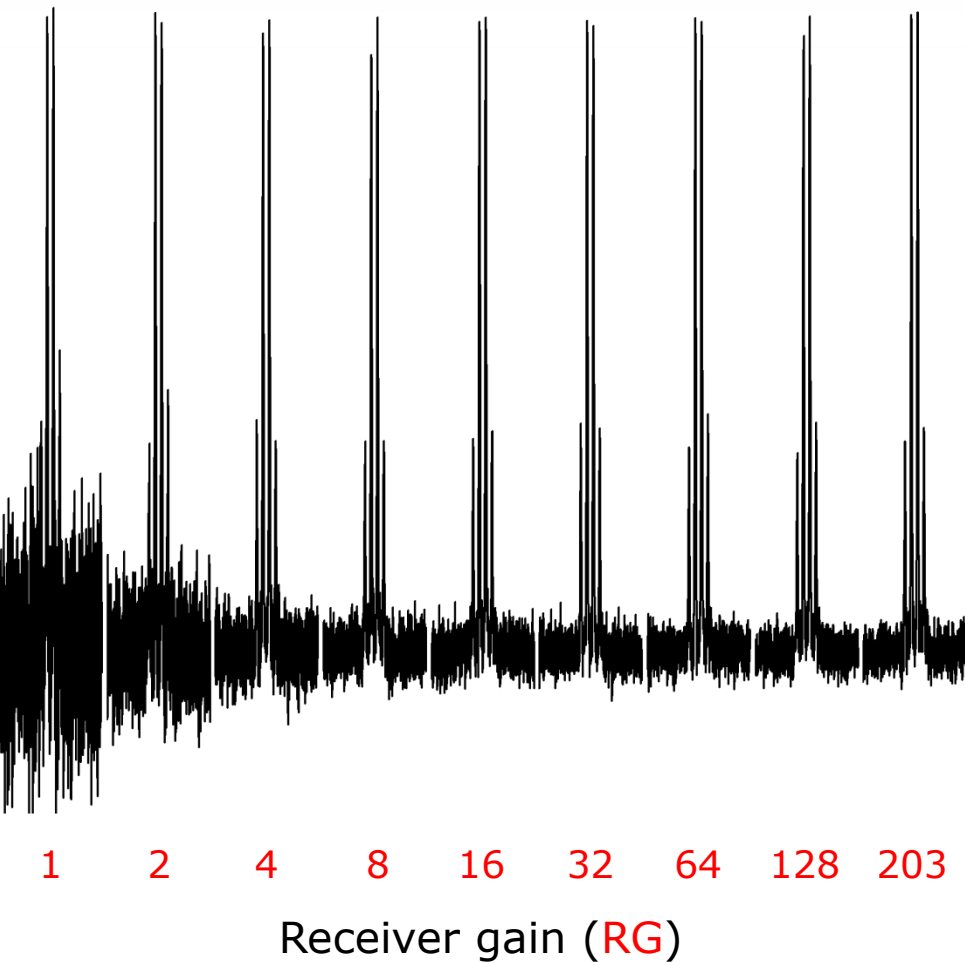
- [**pulsecal**] AU-program to determine the 90° 1H-pulse in F1
- [**pulsecal fast**] pulse determination without receiver gain adjustment and without search for the biggest signal
- [**pulsecal quiet**] the result is not shown
- [**pulsecal sn**] uses em instead of gm for processing
- [**pulsecal c13, p31, f19**] checks the C13, P31, F19-pulse in F1

Acquire Toolbar – Gain

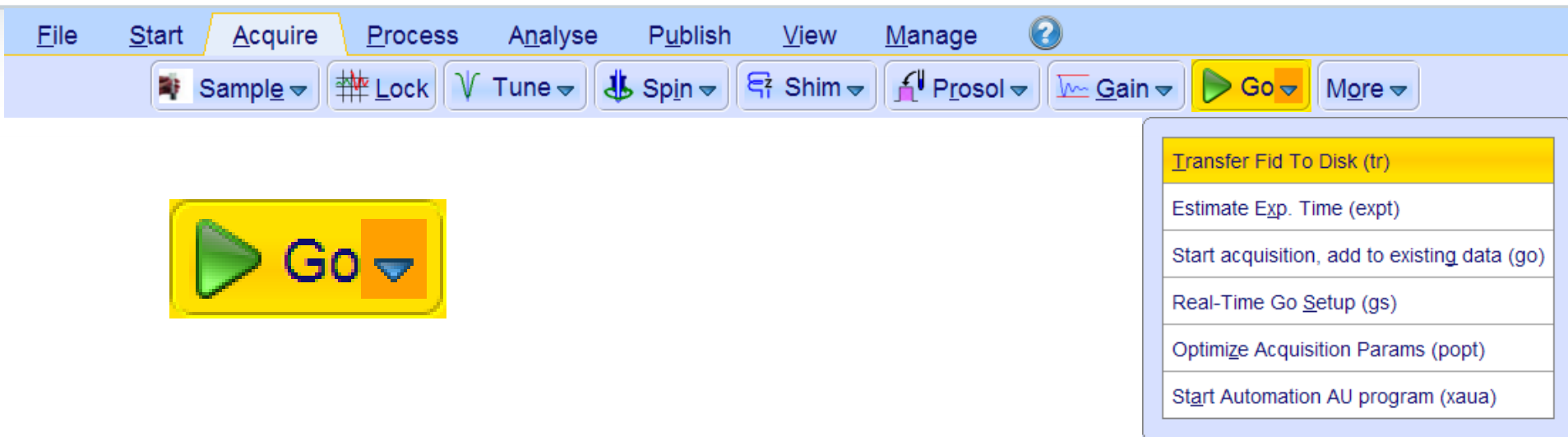


- [**rga**] runs automatic determination of optimum receiver gain (RG)
- RG controls the amplitude of the FID signal before it enters the digitizer
- Higher RG values will improve S/N (up to a certain RG value)
- Too high RG will result in distorted spectra

Effect of RG

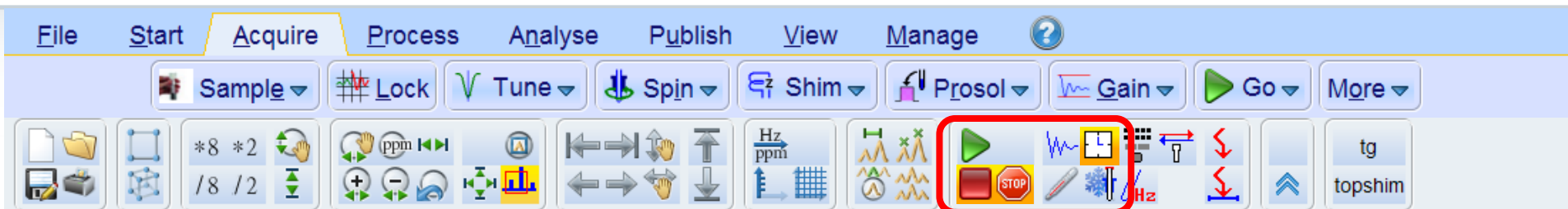





Acquire Toolbar – Go



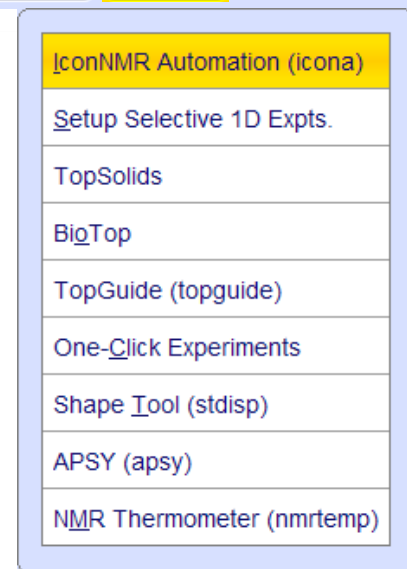
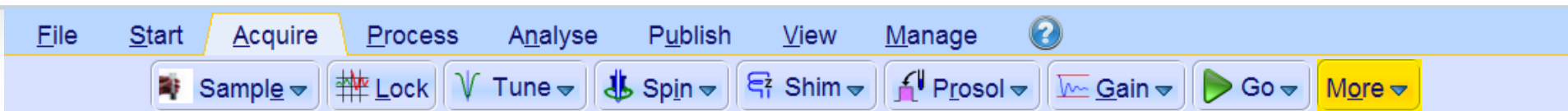
- [**zg**] zeroes data (!) and starts acquisition
- [**go**] starts acquisition and adds to existing data
- [**xaua**] executes AU program for acquisition

Acquire Toolbar – Go



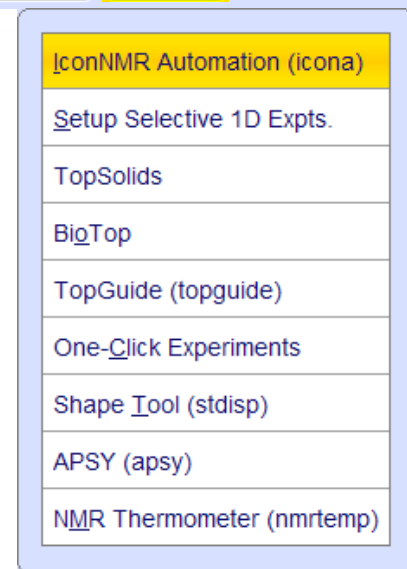
- [**stop**] stops the measurement without storing the FID ()
- [**halt**] stops the measurement and stores the FID ()
 - [**halt 4**] stops at a multiple of 4
- [**tr**] stores the FID without stopping the measurement
 - [**tr 2**] stores FID at a multiple of 2 without stopping
- [**expt**] estimates experiment time ()

Acquire Toolbar – More



- IconNMR: Automation program for routine use
- Easy setup for selective 1D/2D experiments from 1D Proton /Carbon
- TopSolids: Assisted user interface for solid-state experiments
- BioTop: Assisted user interface for high resolution experiments for biological samples

Acquire Toolbar – More



- TopGuide: Interactive software to guide through acquisition and processing of 1D/2D experiments
- One-click execution of a series of 1D and 2D experiments
- Shape Tool: Design and manipulation of pulse shapes; calculation of excitation profiles
- APSY: Automated Projection Spectroscopy; provides access to N-dimensional correlations by lower-dimensional projections

One more thing...



- Digitizer mode: **DIGMOD**
- Can be set in [**eda**]
- Typically set to digital (oversampling and digital filtering)

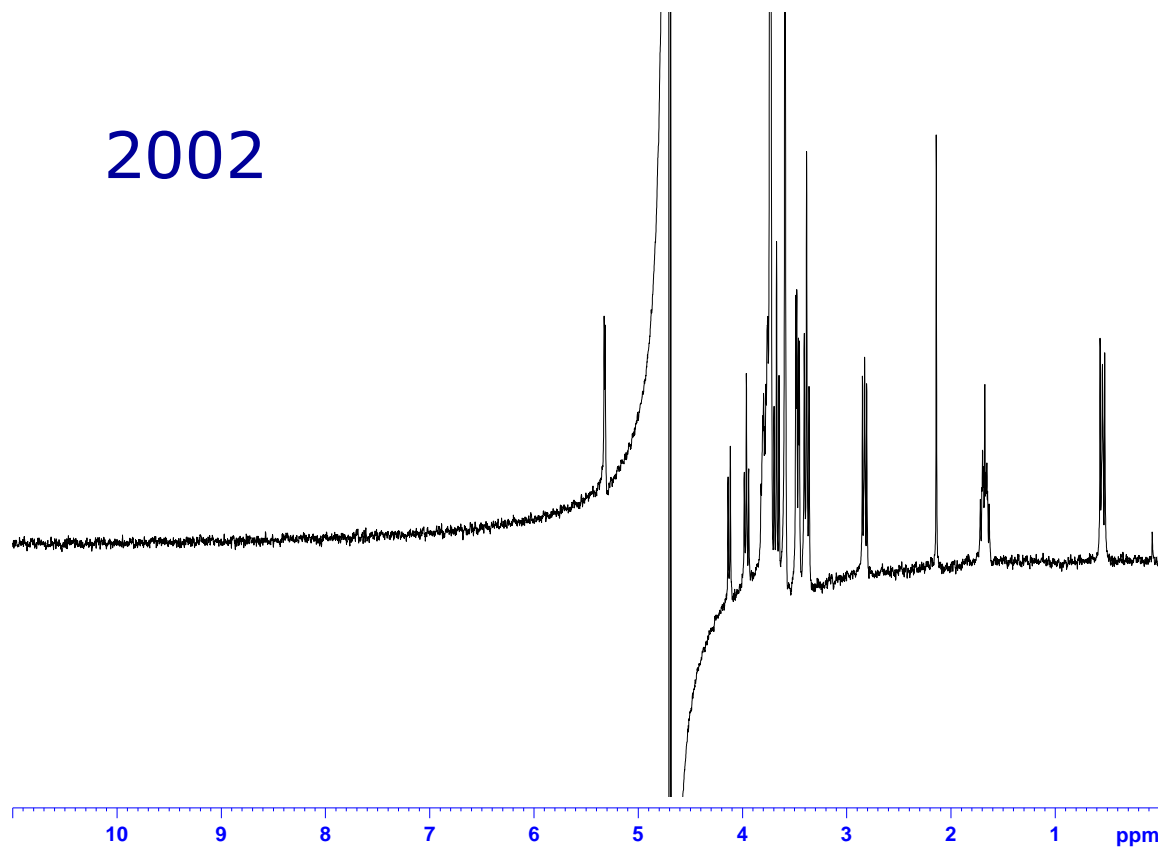
- New since TopSpin 2.0: baseopt
 - Flat baseline at 0
 - No 1st order phase correction
 - No distortions at the edge of the spectrum

- **FILCOR** parameter needs to be determined

One more thing...



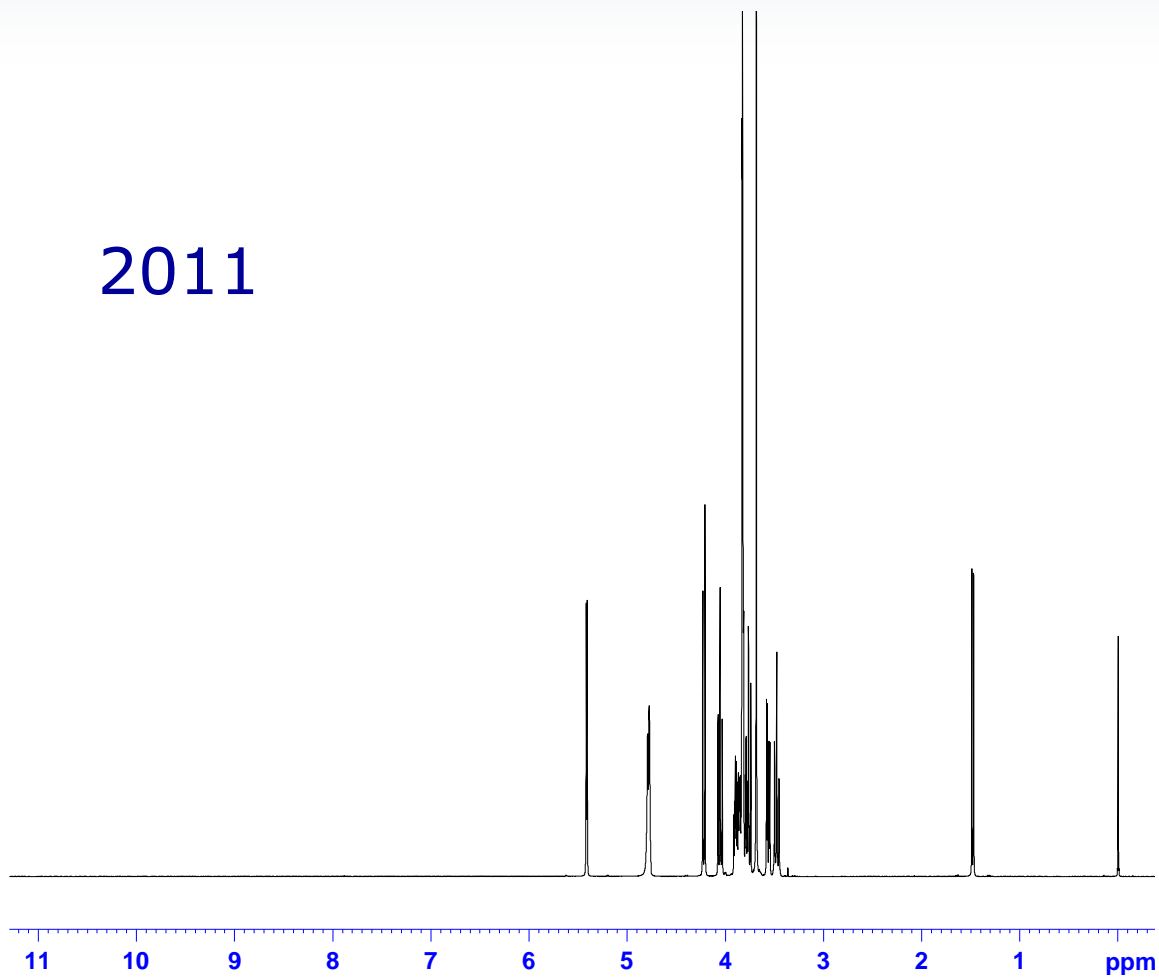
2002



One more thing...



2011



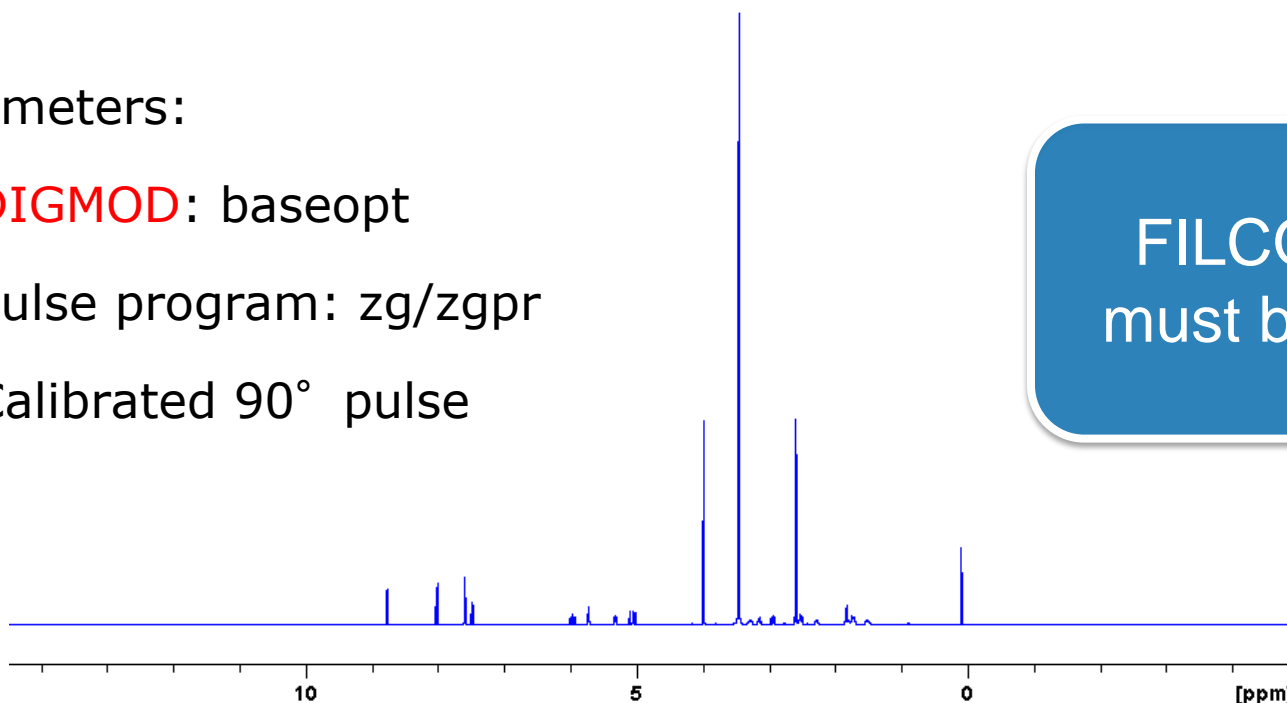
Determine FILCOR parameter



- **FILCOR** is a hardware specific parameter
- Needs to be determined for each probe separately
- Use a sample with a wide chemical shift range

- Parameters:

- **DIGMOD**: baseopt
- Pulse program: zg/zgpr
- Calibrated 90° pulse



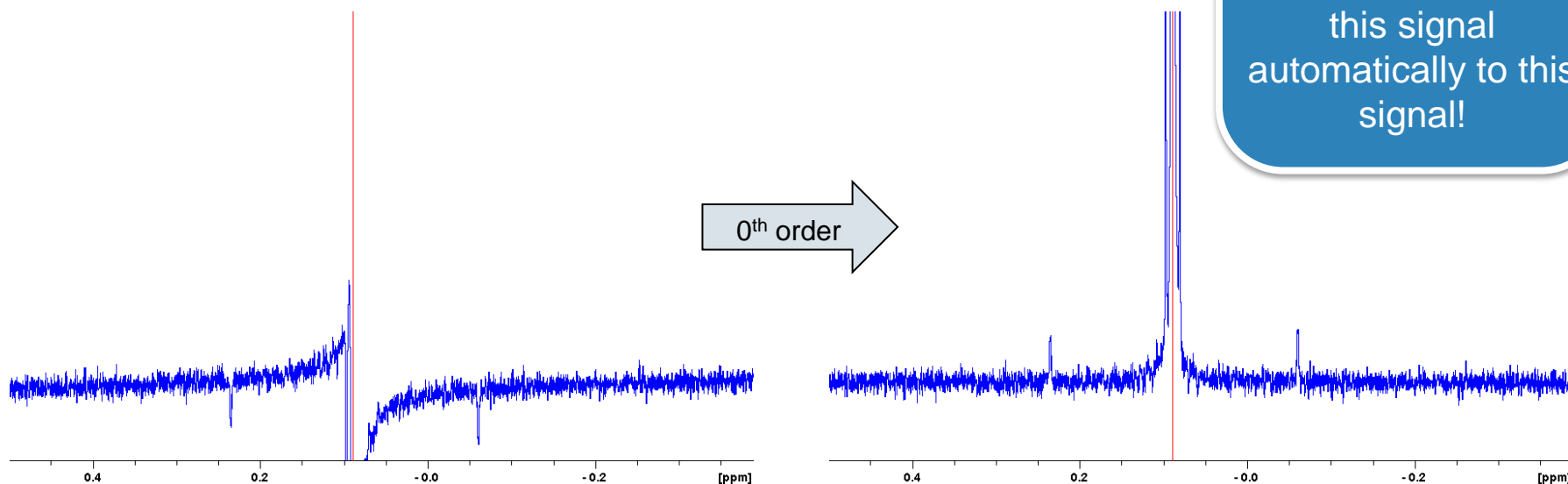
FILCOR
must be 0!

Determine FILCOR parameter



- Zoom into the rightmost signal and phase it with 0th order only
- Pivot point should be set to this signal

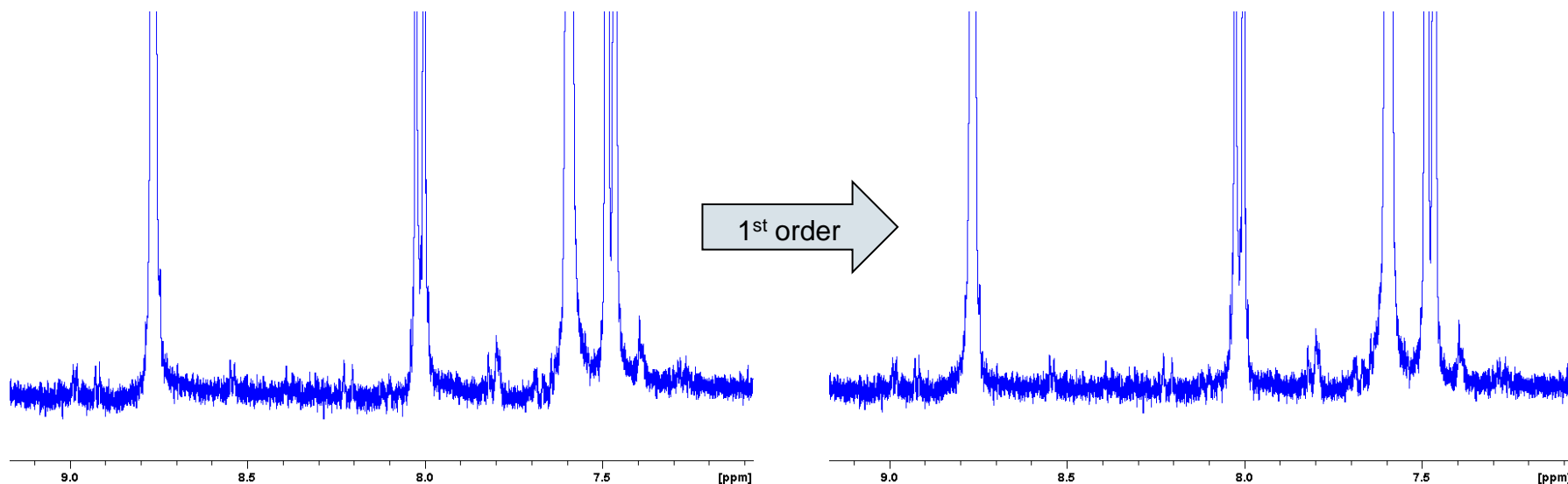
If you enter phasing mode after zooming into the signal, the pivot point is set to this signal automatically to this signal!



Determine FILCOR parameter



- Do not exit phasing mode!
- Zoom into the leftmost signal and phase it with 1st order only
- 1st order phase correction should be small



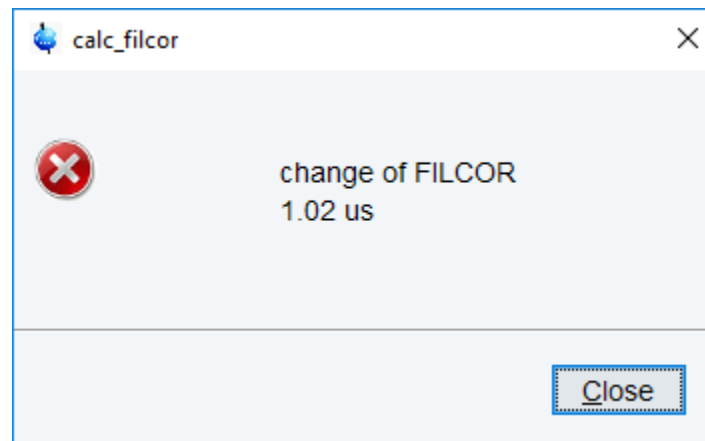
Determine FILCOR parameter



- **FILCOR** parameter is calculated by:

$$FILCOR = \frac{PHC1 \times DW}{180}$$

- AU program available on your USB stick for automatic calculation:
calc_filcor



- **FILCOR** parameter is set as a spectrometer constant with [**edscon**]

Set FILCOR parameter



Edscon

Spectrometer Parameters

Spectrometer parameters

BLKTR [μsec]	Edit...	Preset time for amplifier blanking
DE1 [μsec]	4.50	Time between LO switching and start of FID
DERX [μsec]	1.50	Time between receiver enable and start of FID
DEADC [μsec]	0.50	Time between ADC enable and start of FID
DEPA [μsec]	4.50	Time between preamplifier switching and start of FID
FILCOR [μsec]	1.02	Correction for filter delay
GRADCHAN	GCtrl1	Used gradient channel
GRADPRE [μsec]	Edit...	Pre-delay of gradient channels

Homodecoupling spectrometer parameters

HD_BLKTR [μsec]	Edit...	Preset time for amplifier blanking for homodecoupling
HD_DE1 [μsec]	5.00	Time between LO switching and start of FID for homodecoupling
HD_DEADC [μsec]	0	Time between ADC enable and start of FID for homodecoupling
HD_DEPA [μsec]	2.50	Time between preamplifier switching and start of FID for homodecoupling
HD_DERX [μsec]	0	Time between receiver enable and start of FID for homodecoupling

Undo Close

Protected by super user password.

Edscon

Warning:

You have modified the spectrometer parameters! Any changes will be lost if you continue.

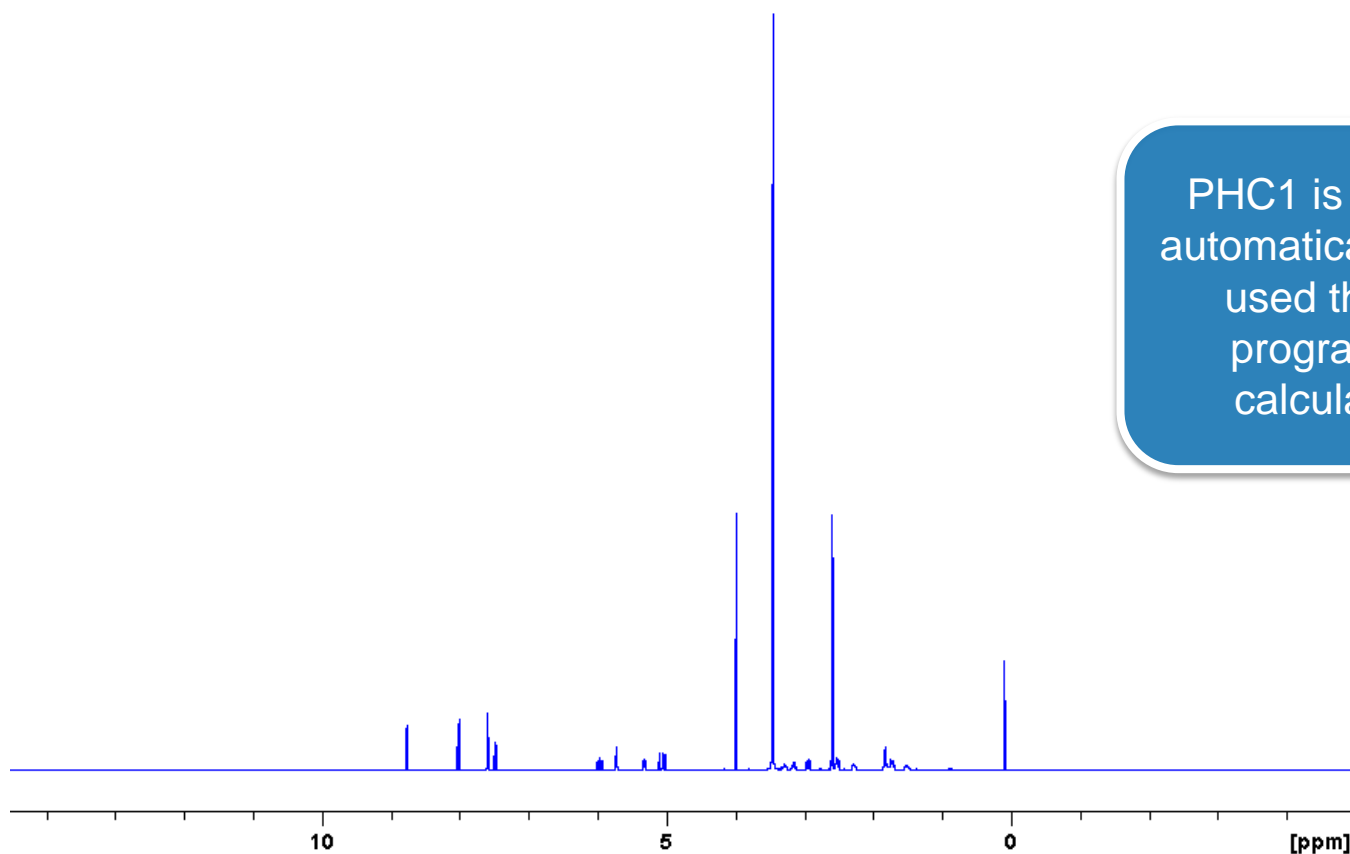
Press "OK" if you want to continue and discard all changes, press "Save" if you want to save the changes, press "Cancel" if you want to return to the edscon dialog.

OK Save Cancel

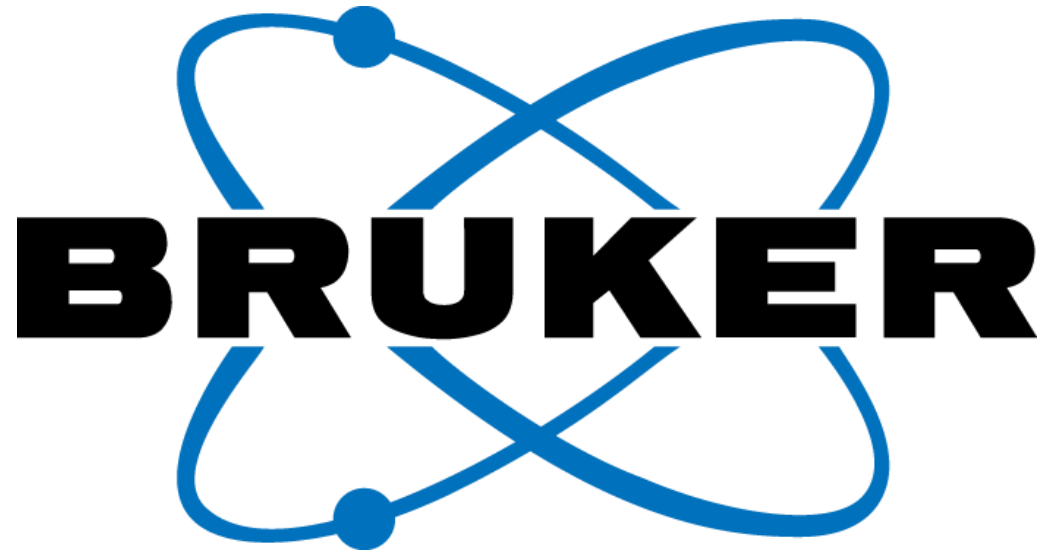
Check FILCOR parameter



- Set **PHC1** to 0 and repeat experiment
- Phasing should be possible with 0th order phase correction only



PHC1 is set to 0 automatically if you used the AU program for calculation.



www.bruker.com