Training Material Number : UMAL-1507-01
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Date : 2022-11-10

ULSAN NATIONAL INSTITUTE OF SCIENCE AND TECHNOLOGY

Varian NMR Manual (Agilent NMR)

Standard Operation Training Course

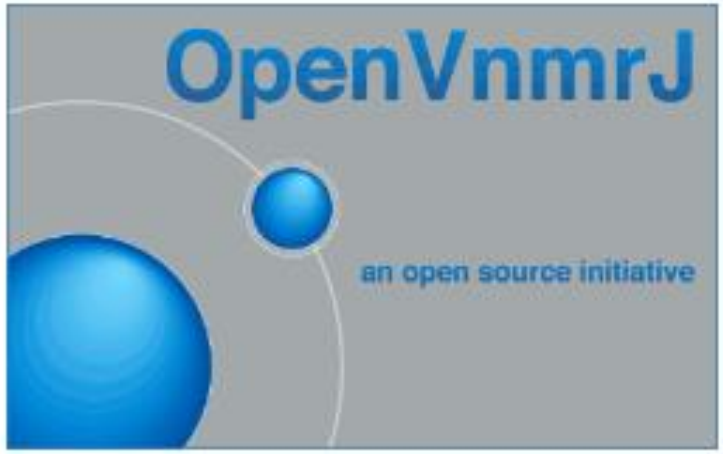
1. Software

OPENVNMRJ

https://github.com/OpenVnmrJ/OpenVnmrJ/blob/master/Install.md

OpenVnmrJ 3.1A

CentOS: Versions 7.5, 7.6, 7.7, 7.8, 7.9
RHEL: Versions 8.4
AlmaLinux: Version 8.4
Ubuntu: Version 20.



OpenVnmrJ 2.1A

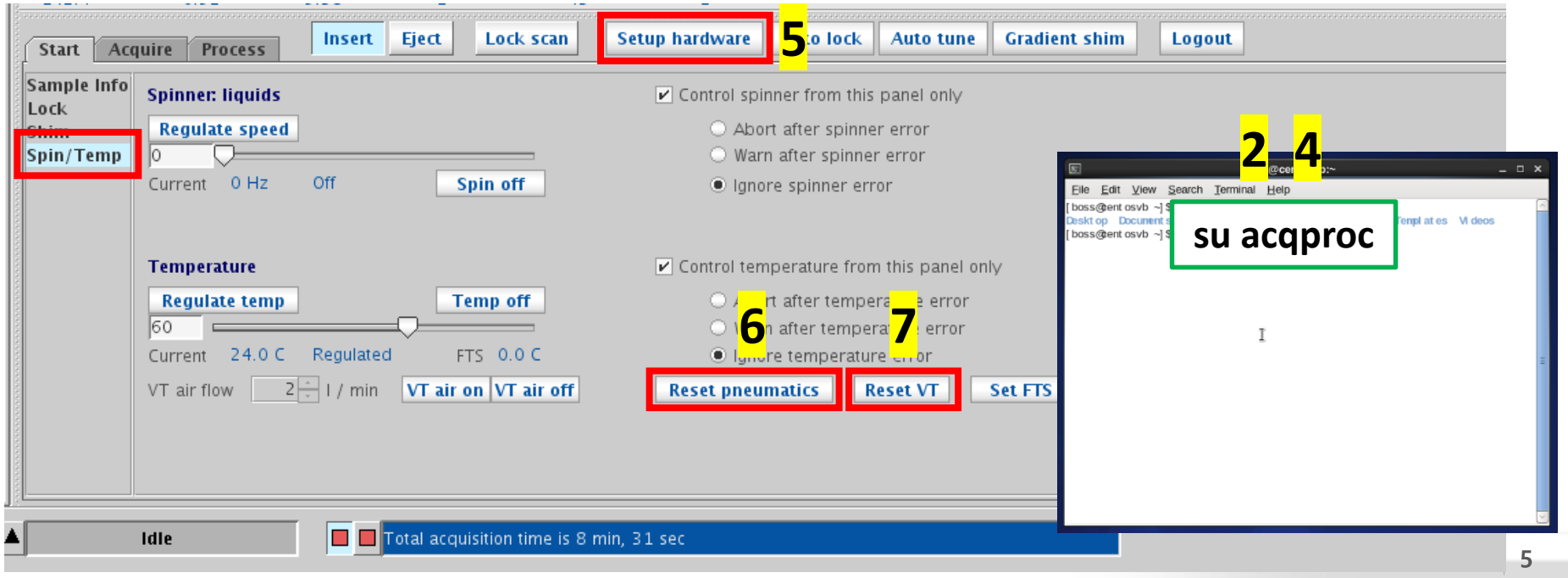
RHEL/CentOS: Versions 6.8, 6.9, and 6.10
RHEL/CentOS: Versions 7.5, 7.6, 7.7, 8.1, and 8.2

https://openvnmrj.org/

2. Turn on /off & Reset Hardware

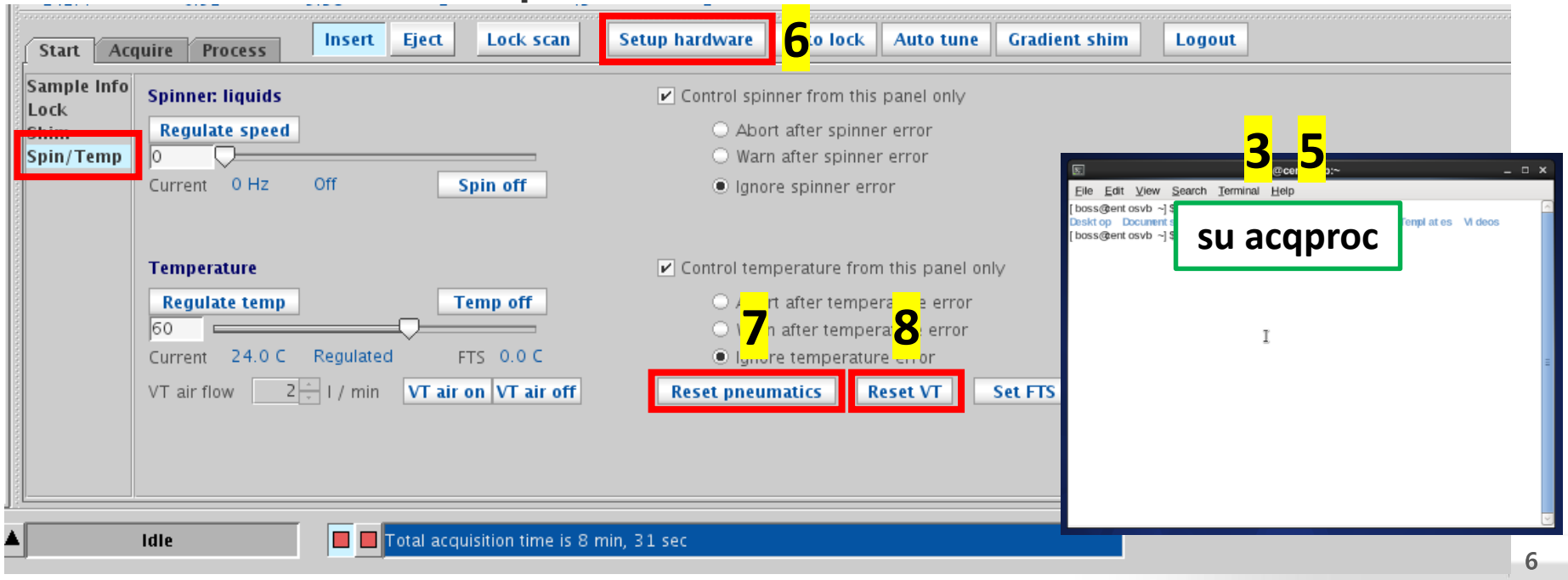
How to reset Console

1. **Open terminal**
2. **Type *su acqproc* on terminal**
   (Disconnect Workstation <-> NMR console communication)
3. **Push reset switch on mater board of NMR Console**
4. **Type *su acqproc* on terminal**
   (Connect Workstation <-> NMR console communication)
5. **Click Setup hardware (reset software/hardware configuration)**
6. **Click Reset pneumatics (reset air flow control unit)**
7. **Click Reset VT (reset Temperature unit)**



How to reset Workstation (PC)

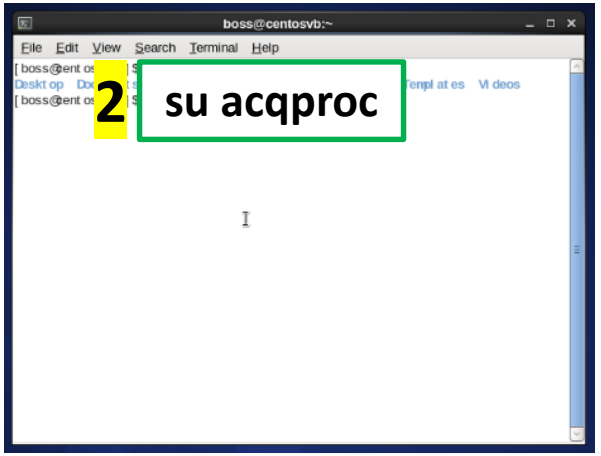
1. **Open terminal**
2. **Type *su acqproc* on terminal**
   (Disconnect Workstation <-> NMR console communication)
3. **Reset Workstation (PC)**
4. **Push reset switch on mater board of NMR Console**
5. **Type *su acqproc* on terminal**
   (Connect Workstation <-> NMR console communication)
6. **Click Setup hardware (reset software/hardware configuration)**
7. **Click Reset pneumatics (reset air flow control unit)**
8. **Click Reset VT (reset Temperature unit)**



How to Turn on-off Console & Workstation

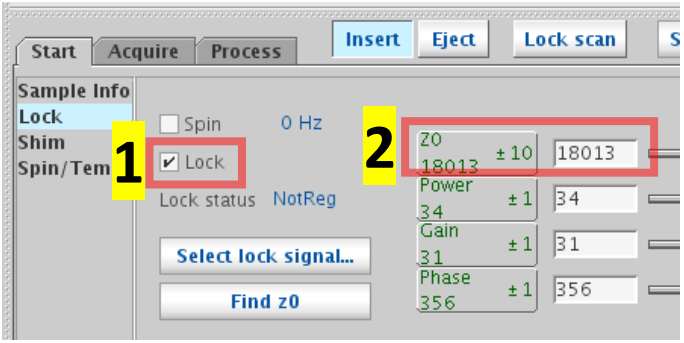
<Turn off Console & Workstation>
1. Open terminal
2. Type *su acqproc* on terminal
   (Disconnect Workstation <-> NMR console communication)
3. Turn off Workstation (PC)
4. Turn off NMR Console

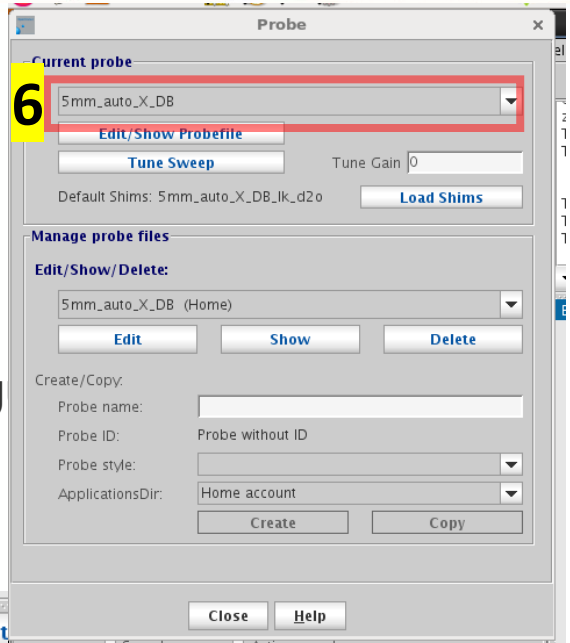
<Turn on Console & Workstation>
1. Turn on NMR Console
2. Turn on Workstation (PC)
3. Double click 'openvnmrj2.1' (Automatically run 'su acqproc')
4. Click Setup hardware (reset software/hardware configuration)
5. Click Reset pneumatics (reset air flow control unit)
6. Click Reset VT (reset Temperature unit)

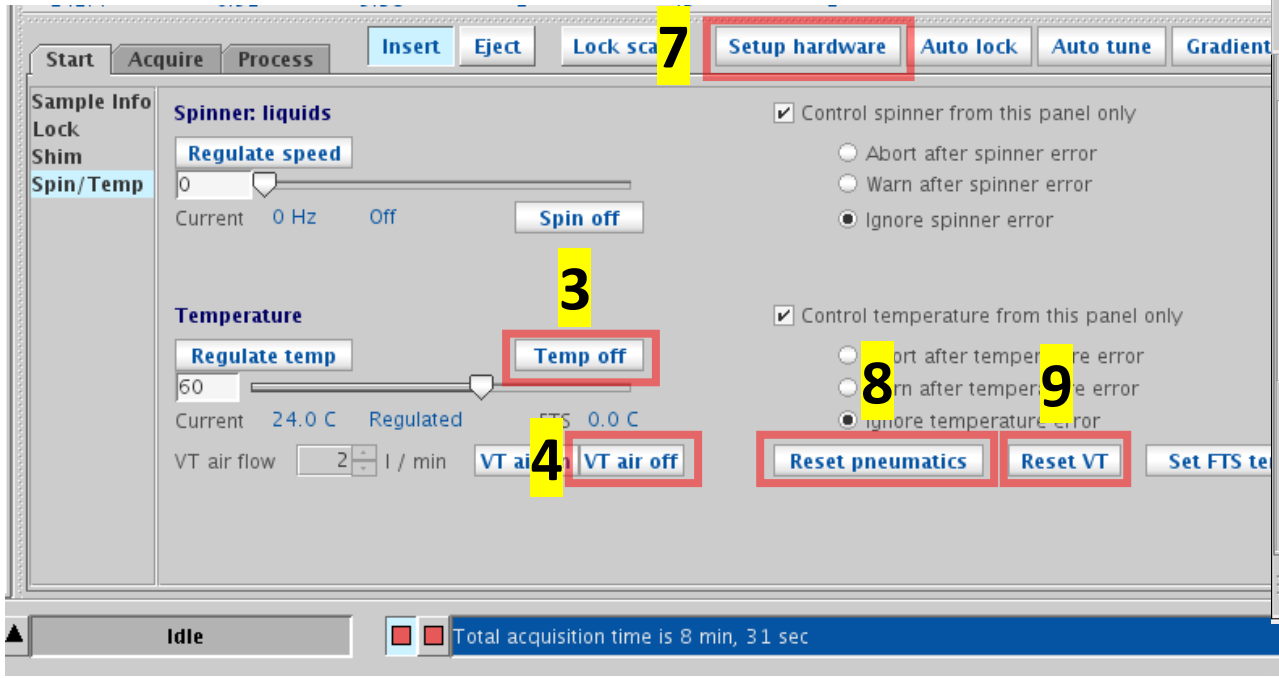


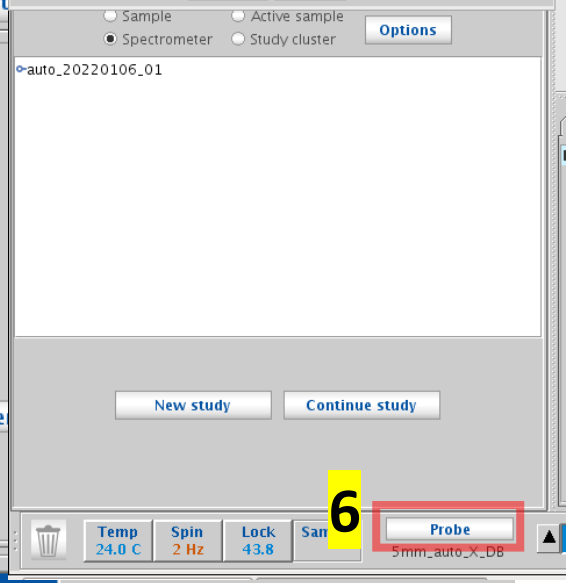
How to change NMR probe

1. Lock off
2. Z0 is 0
3. Regulate Temp off
4. Air flow off
5. Replace NMR probe
6. Choice Current probe
7. Click Setup hardware (reset software/hardware config
8. Click Reset pneumatics (reset air flow control unit)
9. Click Reset VT (reset Temperature unit)
10. If it is not work, follow 'How to reset Console'.









3. Quick Guide

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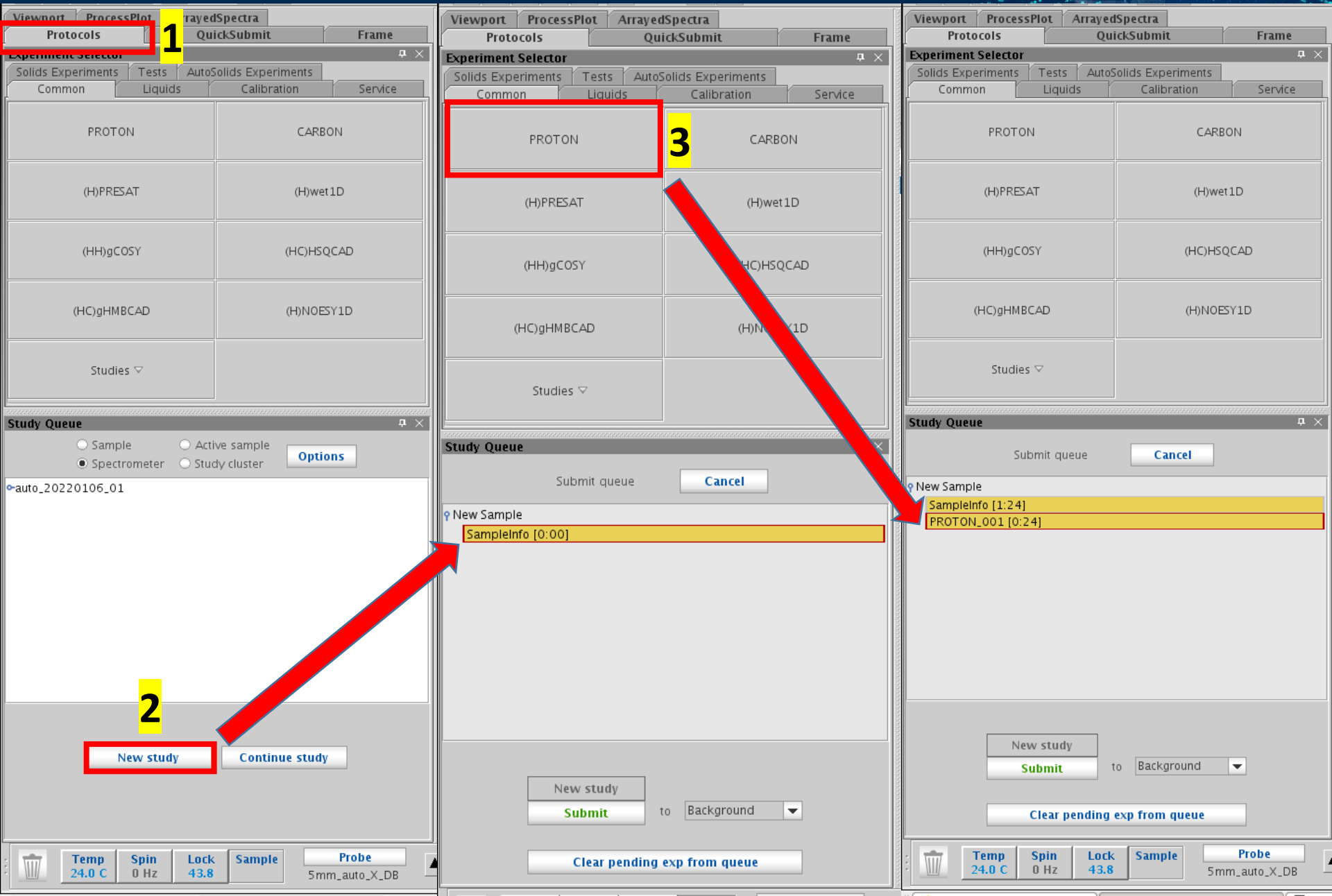
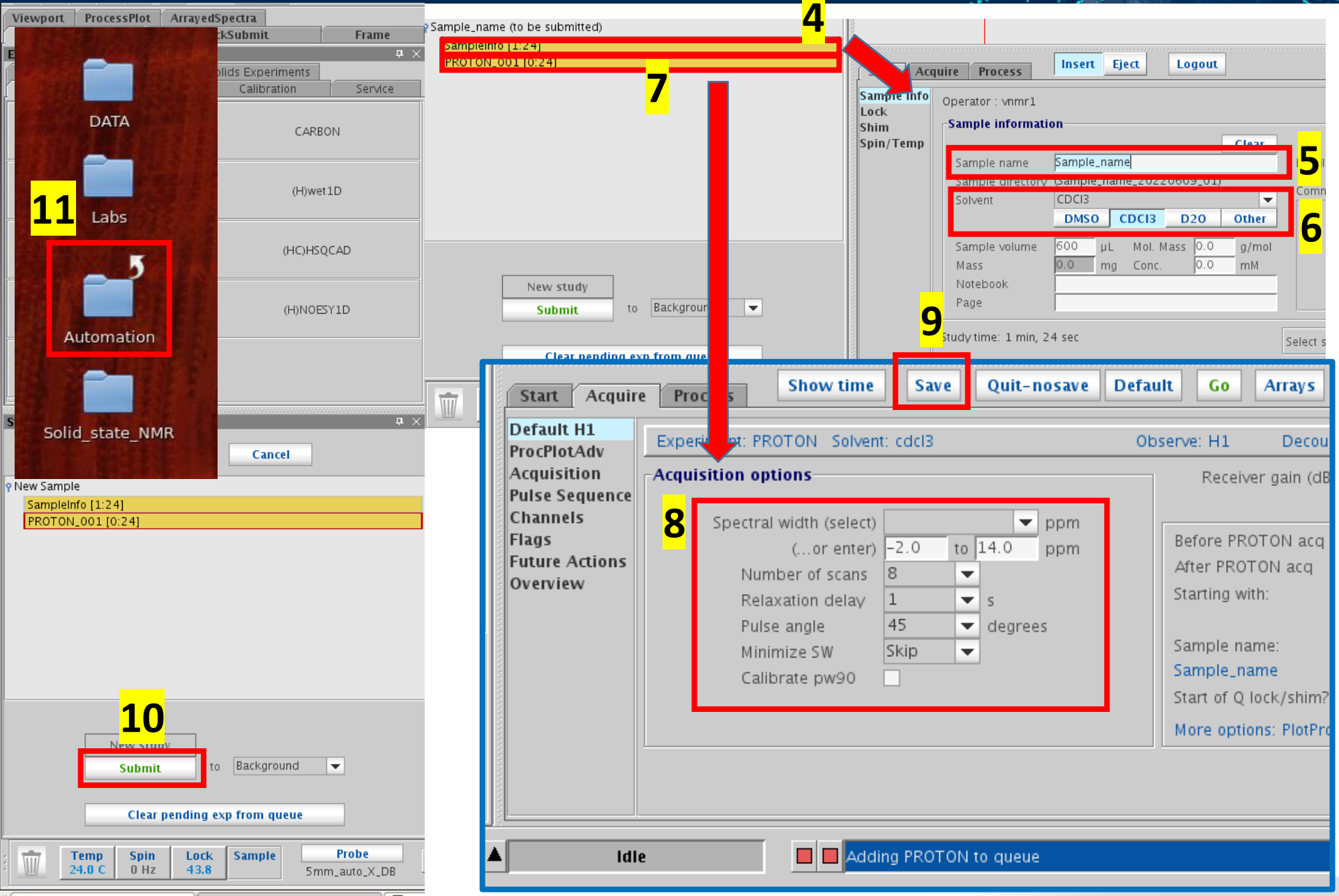
3-1. Automation

Automation


Viewport
ProcessPlot
ArrayedSpectra
Protocols
QuickSubmit
Frame
1
Experiment Selector
Solids Experiments
Tests
AutoSolids Experiments
Common
Liquids
Calibration
Service
PROTON
CARBON
(H)PRESAT
(H)wet1D
(HH)gCOSY
(HC)HSQCAD
(HC)gHMBCAD
(H)NOESY1D
Studies
Study Queue
Sample
Active sample
Spectrometer
Study cluster
Options
auto_20220106_01
2
New study
Continue study
Temp
24.0 C
Spin
0 Hz
Lock
43.8
Sample
Probe
5mm_auto_X_DB
3
Submit queue
Cancel
New Sample
SampleInfo [0:00]
Submit
to
Background
Clear pending exp from queue
SampleInfo [1:24]
PROTON_001 [0:24]

Automation


DATA
Labs
Automation
Solid_state_NMR
11
Viewport
ProcessPlot
ArrayedSpectra
Frame
Calibration
Service
CARBON
(H)wet1D
(HC)HSQCAD
(H)NOESY1D
Cancel
New Sample
SampleInfo [1:24]
PROTON_001 [0:24]
10
New study
Submit
to
Background
Clear pending exp from queue
Temp
24.0 C
Spin
0 Hz
Lock
43.8
Sample
Probe
5mm_auto_X_DB
Sample_name (to be submitted)
SampleInfo [1:24]
PROTON_001 [0:24]
4
7
Acquire
Process
Insert
Eject
Logout
Sample Info
Lock
Shim
Spin/Temp
Operator : vnmr1
Sample information
Clear
Sample name
Sample_name
5
Solvent
CDCl3
DMSO
CDCl3
D2O
Other
6
Sample volume
600
µL
Mol. Mass
0.0
g/mol
Mass
0.0
mg
Conc.
0.0
mM
Notebook
Page
9
Study time: 1 min, 24 sec
Start
Acquire
Show time
Save
Quit-nosave
Default
Go
Arrays
Default H1
ProcPlotAdv
Acquisition
Pulse Sequence
Channels
Flags
Future Actions
Overview
Observe: H1
Acquisition options
8
Spectral width (select)
ppm
(...or enter)
-2.0
to
14.0
ppm
Number of scans
8
Relaxation delay
1
s
Pulse angle
45
degrees
Minimize SW
Skip
Calibrate pw90
Receiver gain (dB
Before PROTON acq
After PROTON acq
Starting with:
Sample name:
Sample_name
Start of Q lock/shim?
More options: PlotPro
Idle
Adding PROTON to queue

3-2. Manually operation

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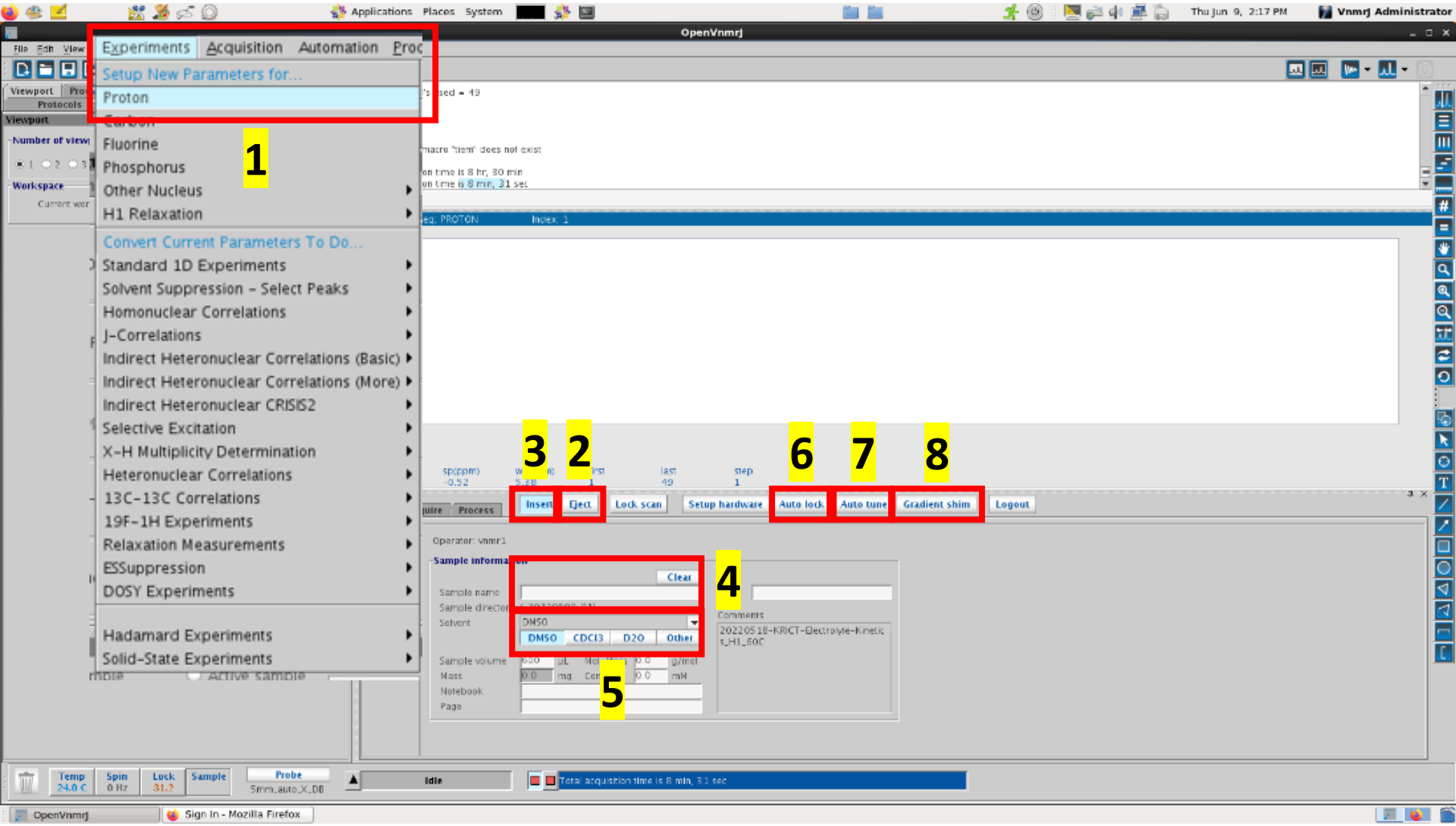
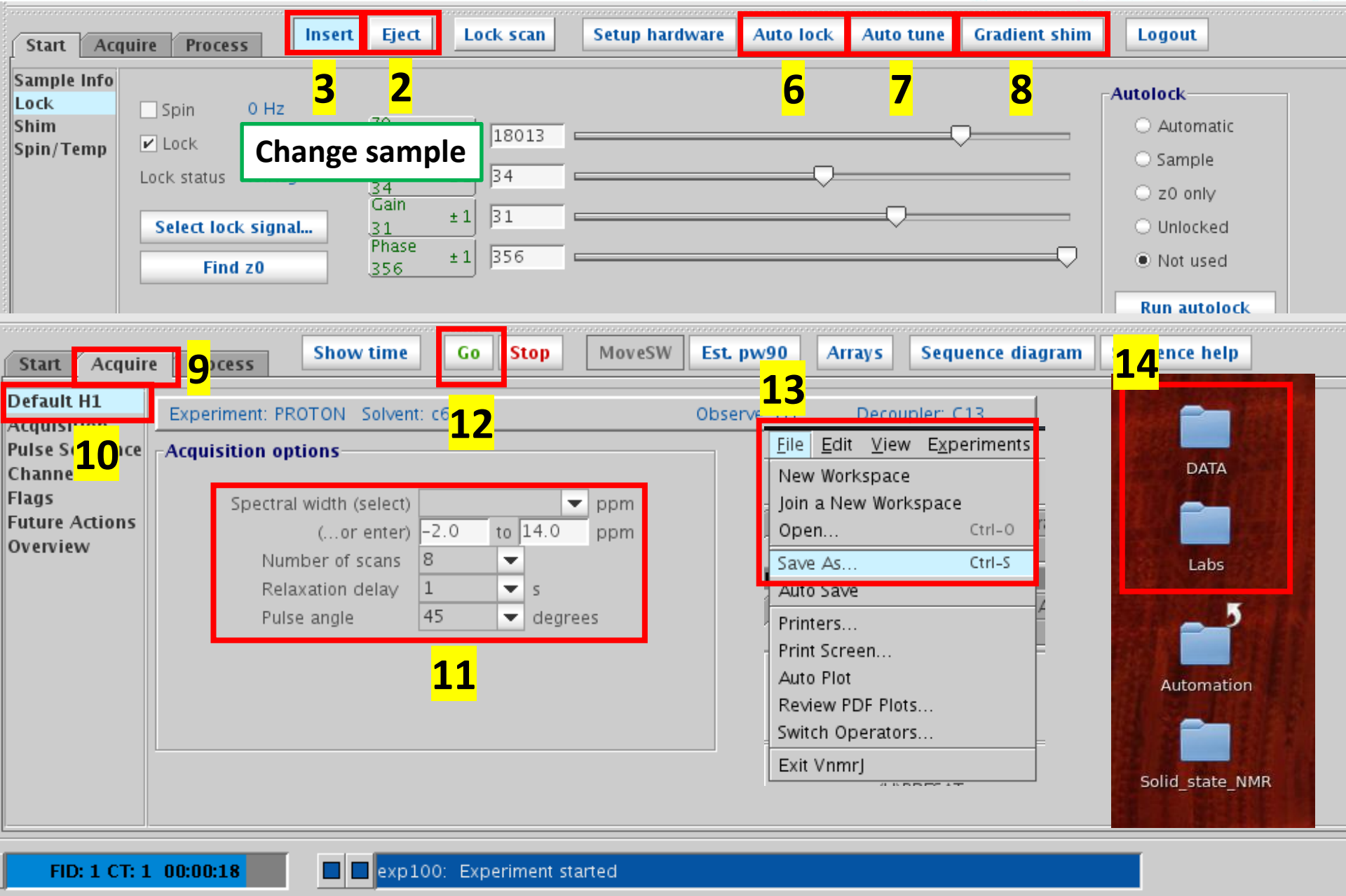
Process


Experiments
Acquisition
Automation
Setup New Parameters for...
Proton
Fluorine
Phosphorus
Other Nucleus
H1 Relaxation
Convert Current Parameters To Do...
Standard 1D Experiments
Solvent Suppression - Select Peaks
Homonuclear Correlations
J-Correlations
Indirect Heteronuclear Correlations (Basic)
Indirect Heteronuclear Correlations (More)
Indirect Heteronuclear CRISIS2
Selective Excitation
X-H Multiplicity Determination
Heteronuclear Correlations
13C-13C Correlations
19F-1H Experiments
Relaxation Measurements
ESSuppression
DOSY Experiments
Hadamard Experiments
Solid-State Experiments
1
2
3
4
5
6
7
8
Insert
Eject
Lock scan
Setup hardware
Auto lock
Auto tune
Gradient shim
Logout
Clear
DMSO
CDCl3
D2O
Other

Process


Start
Acquire
Process
Insert
Eject
Lock scan
Setup hardware
Auto lock
Auto tune
Gradient shim
Logout
3
2
6
7
8
Sample Info
Lock
Shim
Spin/Temp
Spin
0 Hz
Lock
Lock status
Change sample
Select lock signal...
Find z0
Gain
31
± 1
Phase
356
± 1
18013
34
31
356
Autolock
Automatic
Sample
z0 only
Unlocked
Not used
Run autolock
Start
Acquire
9
Show time
Go
Stop
MoveSW
Est. pw90
Arrays
Sequence diagram
14
Default H1
10
Pulse
Channe
Flags
Future Actions
Overview
Experiment: PROTON
Solvent: c6
12
Decoupler: C13
13
Acquisition options
Spectral width (select)
ppm
(...or enter)
-2.0
to
14.0
ppm
Number of scans
8
Relaxation delay
1
s
Pulse angle
45
degrees
11
File
Edit
View
Experiments
New Workspace
Join a New Workspace
Open...
Ctrl-O
Save As...
Ctrl-S
Auto Save
Printers...
Print Screen...
Auto Plot
Review PDF Plots...
Switch Operators...
Exit VnmrJ
DATA
Labs
Automation
Solid_state_NMR
FID: 1 CT: 1 00:00:18
exp100: Experiment started

4. Start tab

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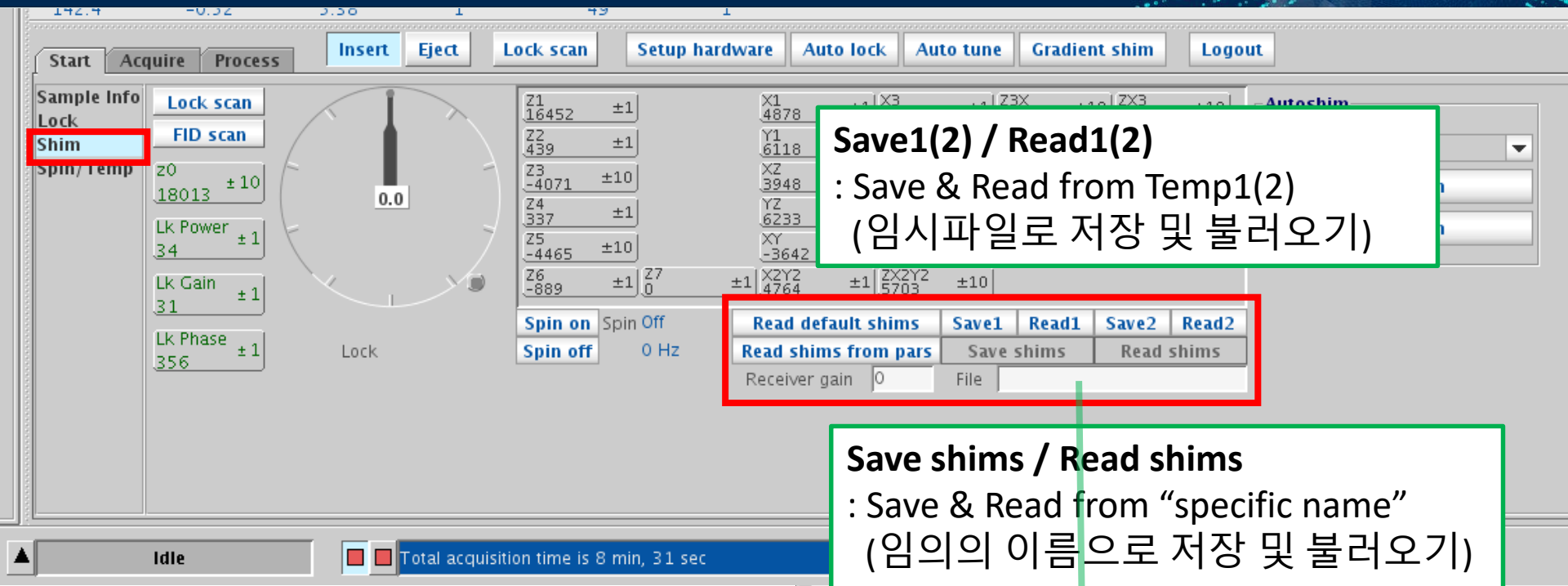
Start - Shim



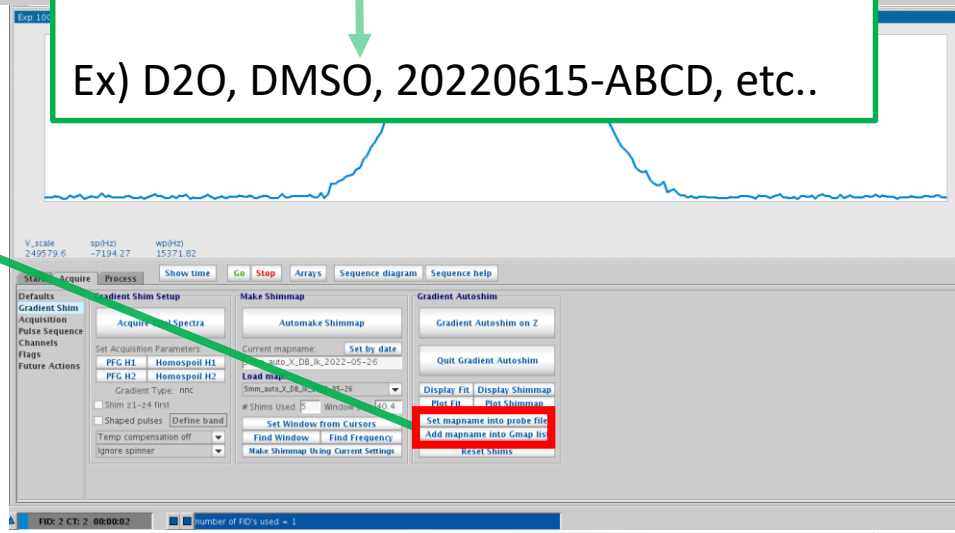
Save1(2) / Read1(2)
: Save & Read from Temp1(2)
(임시파일로 저장 및 불러오기)

Save shims / Read shims
: Save & Read from “specific name”
(임의의 이름으로 저장 및 불러오기)

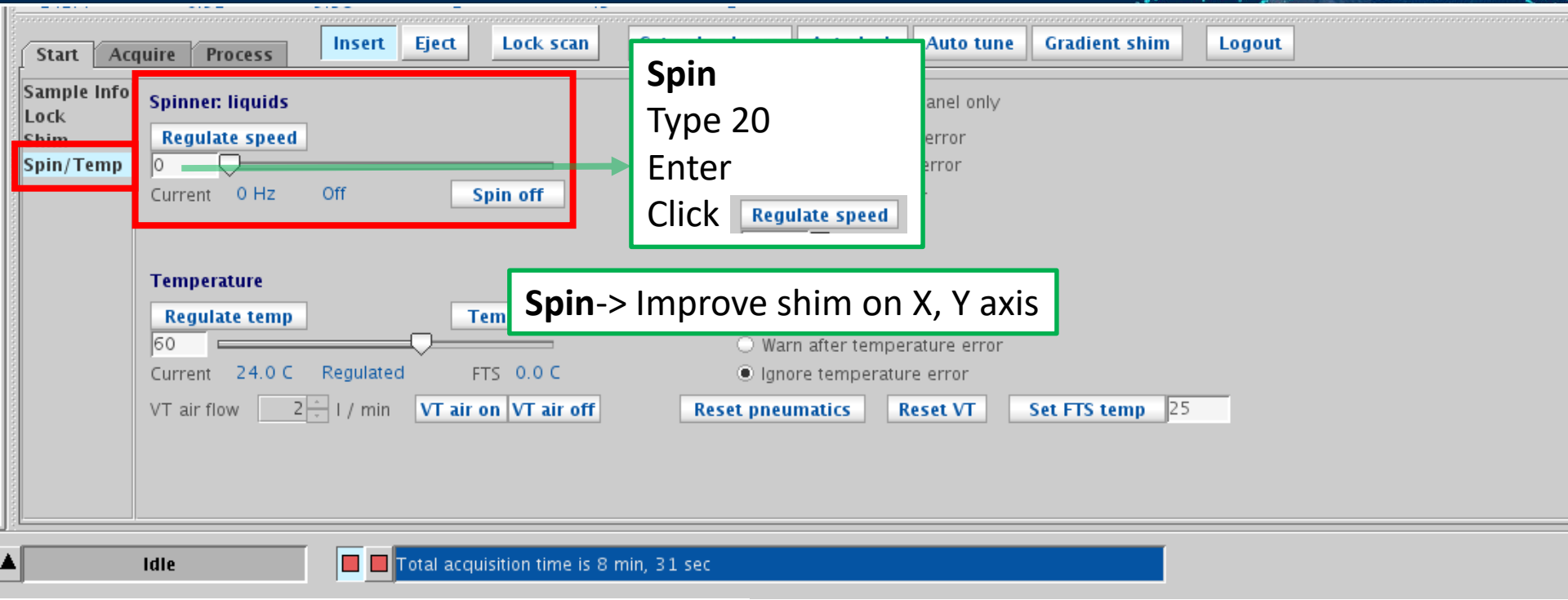
Ex) D2O, DMSO, 20220615-ABCD, etc..

Read default shims
: Read shim from Gradient shim-map
(Gradient shim-map을 가져옴)

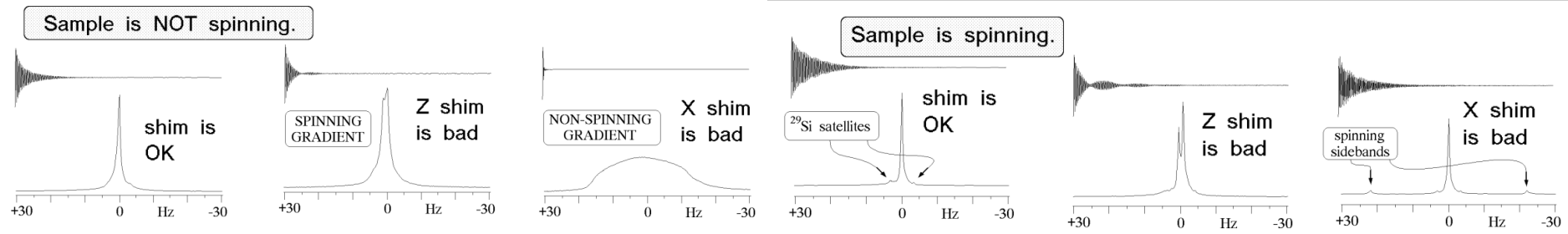
Read shims from pars
: Read shim from experiments
(실험방에서 shim-map을 가져옴)



Start – Spin



Spin-> Improve shim on X, Y axis



https://medicine.uiowa.edu/nmr/sites/medicine.uiowa.edu.nmr/files/wysiwyg_uploads/Shimming%20an%20NMR%20Magnet.pdf

Start – Temp

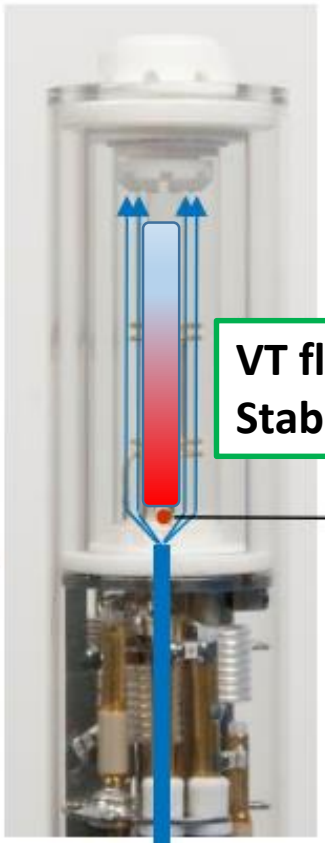
Temperature
Type 50 (target temp)
Enter
Click

Start | Acquire | Setup hardware | Auto lock | Auto tune | Gradient shim | Logout

Sample Info
Lock
Spin/Temp

Temperature
Regulate temp | Temp off
60
Current 24.0 C Regulated FTS 0.0 C
VT air flow 2 l / min | VT air on | VT air off

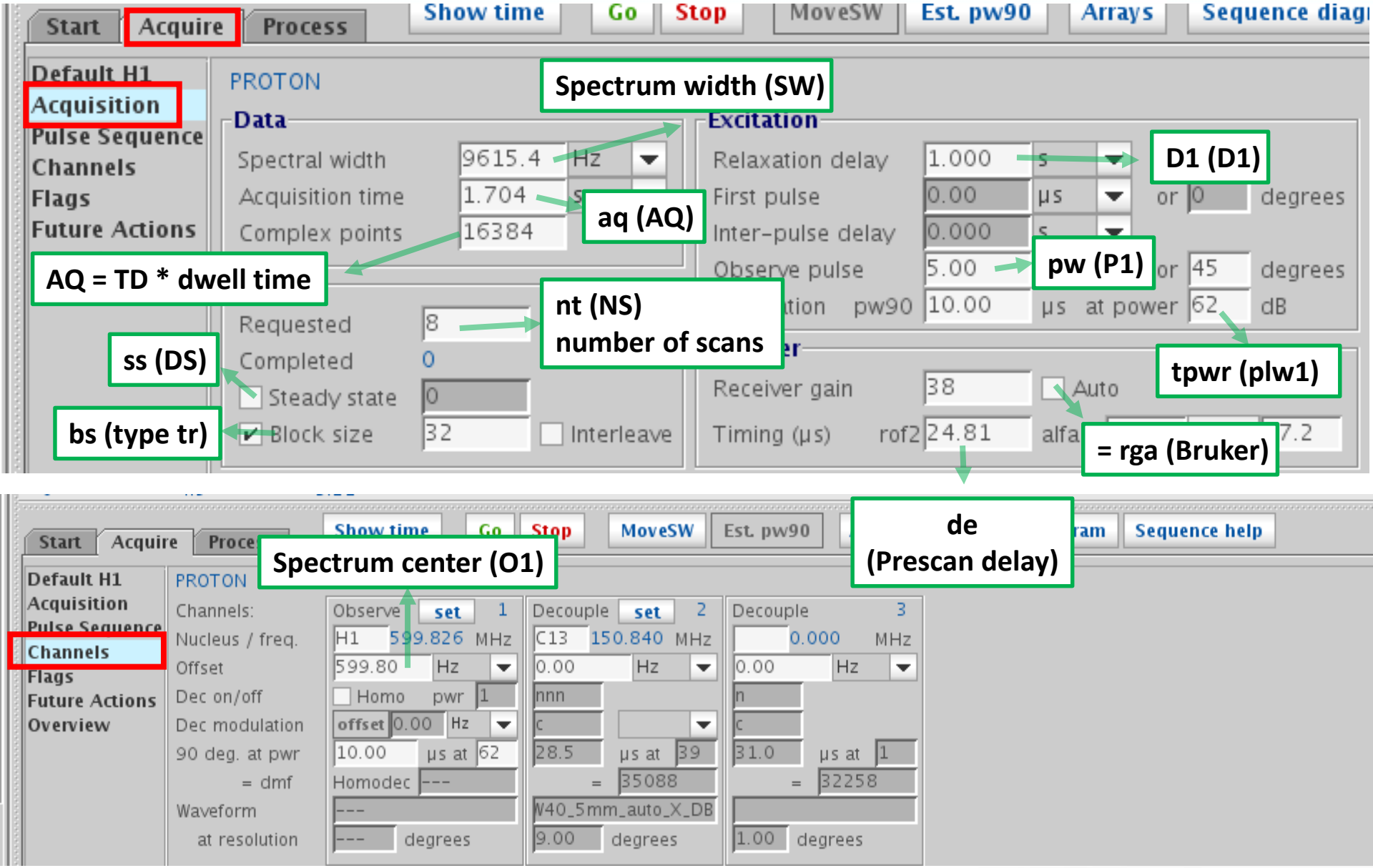
Limit VT air flow -> 6 °C / min
Extremely temperature change could break Quartz in Probe.



VT flow cause temp gradient.
Stabilization time: 5 – 15 min

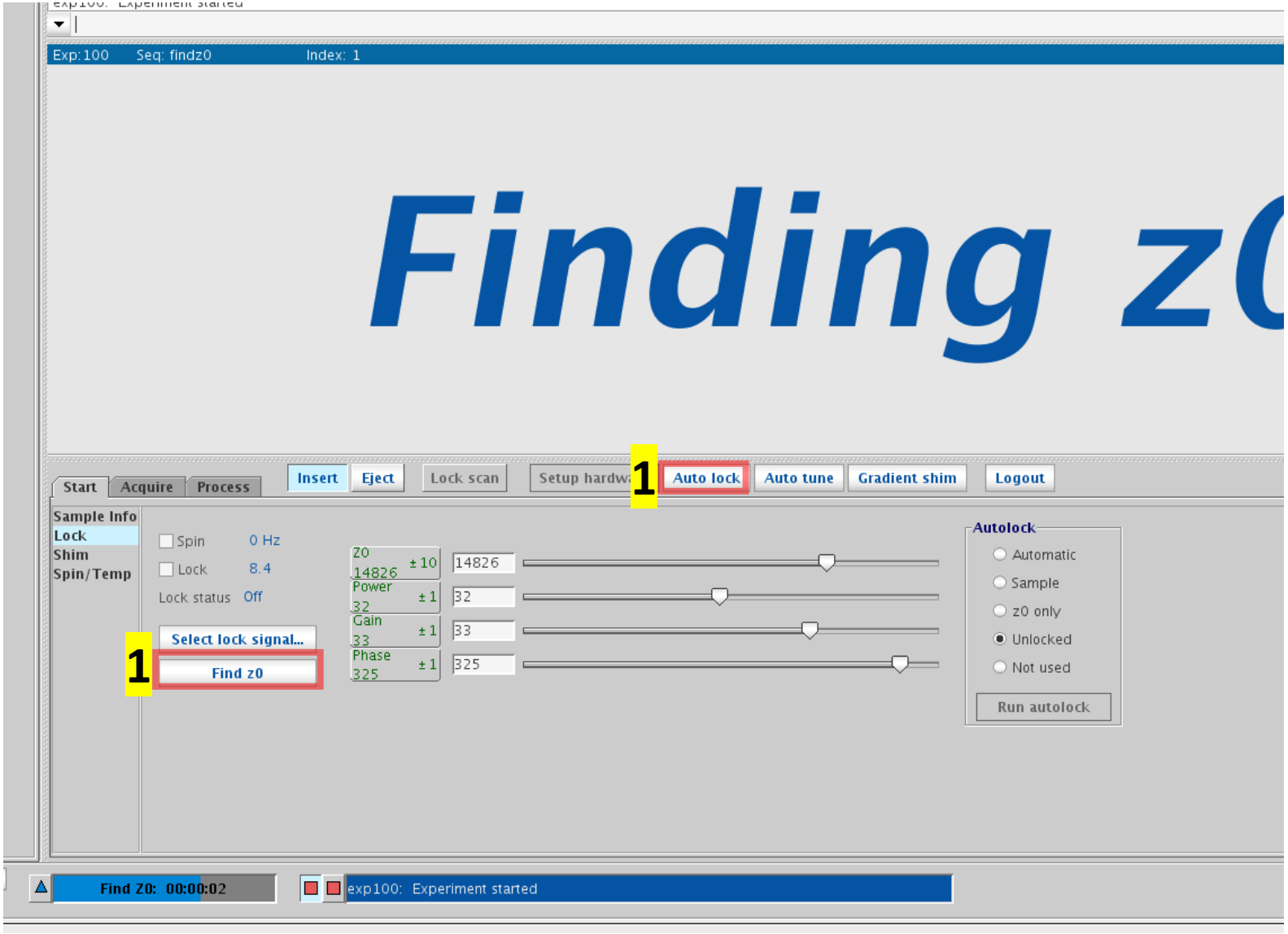
5. Acquire tab

Acquire tab

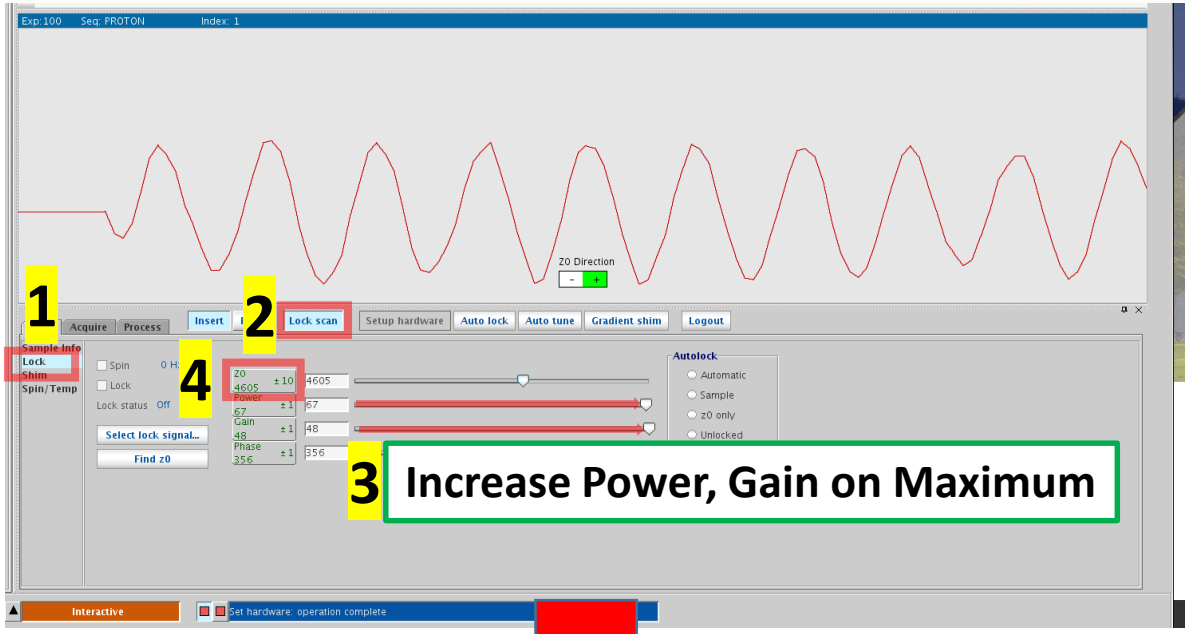

Start
Acquire
Process
Show time
Go
Stop
MoveSW
Est. pw90
Arrays
Sequence diagr
Default H1
Acquisition
Pulse Sequence
Channels
Flags
Future Actions
PROTON
Data
Spectral width
9615.4
Hz
Acquisition time
1.704
Complex points
16384
Spectrum width (SW)
aq (AQ)
AQ = TD * dwell time
Excitation
Relaxation delay
1.000
s
D1 (D1)
First pulse
0.00
µs
or
0
degrees
Inter-pulse delay
0.000
Observe pulse
5.00
pw (P1)
or
45
degrees
pw90
10.00
µs
at power
62
dB
Requested
8
nt (NS)
number of scans
Completed
0
ss (DS)
Steady state
0
bs (type tr)
Block size
32
Interleave
Receiver gain
38
Auto
tpwr (plw1)
Timing (µs)
rof2
24.81
alfa
= rga (Bruker)
de
(Prescan delay)
Start
Acquire
Show time
Go
Stop
MoveSW
Est. pw90
Sequence help
Spectrum center (O1)
Default H1
Acquisition
Channels
Flags
Future Actions
Overview
PROTON
Channels:
Nucleus / freq.
Offset
Dec on/off
Dec modulation
90 deg. at pwr
= dmf
Waveform
at resolution
Observe
set
1
H1
599.826
MHz
599.80
Hz
Homo
pwr
offset
0.00
10.00
µs at
62
Homodec
---
degrees
Decouple
set
2
C13
150.840
0.00
nnn
28.5
µs at
39
35088
W40_5mm_auto_X_DB
9.00
Decouple
3
0.000
31.0
32258
1.00

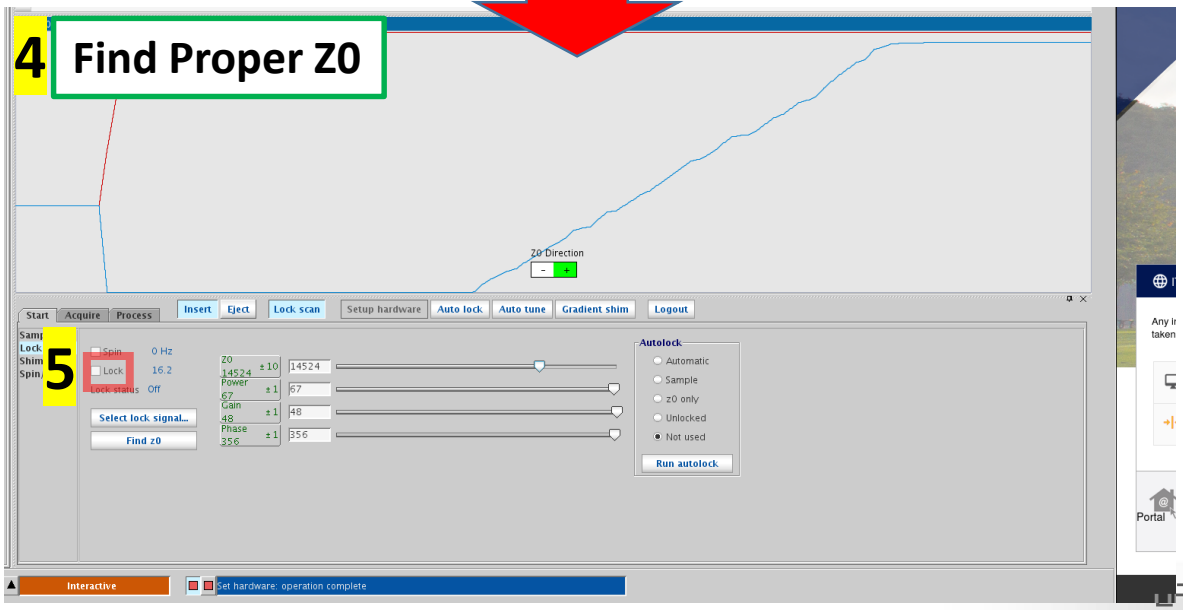
6-1. Lock

Lock (Auto)


Exp:100 Seq: findz0 Index: 1
Finding z0
Start
Acquire
Process
Insert
Eject
Lock scan
Setup hardw
1
Auto lock
Auto tune
Gradient shim
Logout
Sample Info
Lock
Shim
Spin/Temp
Spin 0 Hz
Lock 8.4
Lock status Off
Select lock signal...
1
Find z0
z0 14826 ±10
14826
Power 32 ±1
32
Gain 33 ±1
33
Phase 325 ±1
325
Autolock
Automatic
Sample
z0 only
Unlocked
Not used
Run autolock
Find z0: 00:00:02
exp100: Experiment started

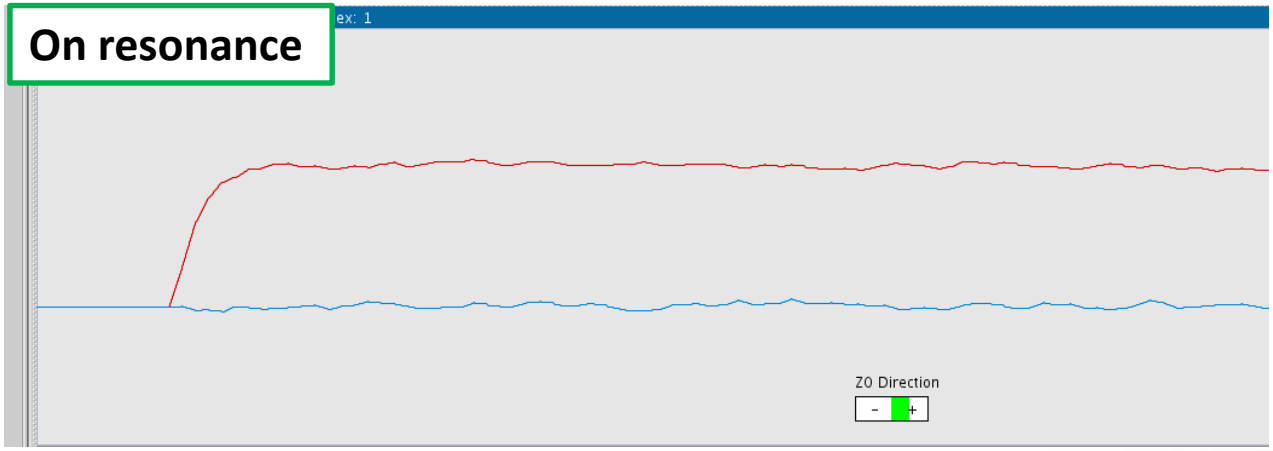
Lock (Manual)


Exp: 100 Seq: PROTON Index: 1
Z0 Direction
1
2
Insert
Lock scan
Setup hardware
Auto lock
Auto tune
Gradient shim
Logout
Acquire
Process
Sample Info
Lock
Shim
Spin/Temp
4
Spin
Lock
Lock status Off
Select lock signal...
Find z0
Z0 4605 ±10
Power 67 ±1
Gain 48 ±1
Phase 356 ±1
Autolock
Automatic
Sample
z0 only
Unlocked
3
Increase Power, Gain on Maximum
Interactive
Set hardware: operation complete


4
Find Proper Z0
Z0 Direction
Start
Acquire
Process
Insert
Eject
Lock scan
Setup hardware
Auto lock
Auto tune
Gradient shim
Logout
5
Spin
Lock
0 Hz
16.2
Lock status Off
Select lock signal...
Find z0
Z0 14524 ±10
Power 67 ±1
Gain 48 ±1
Phase 356 ±1
Autolock
Automatic
Sample
z0 only
Unlocked
Not used
Run autolock
Interactive
Set hardware: operation complete
Portal

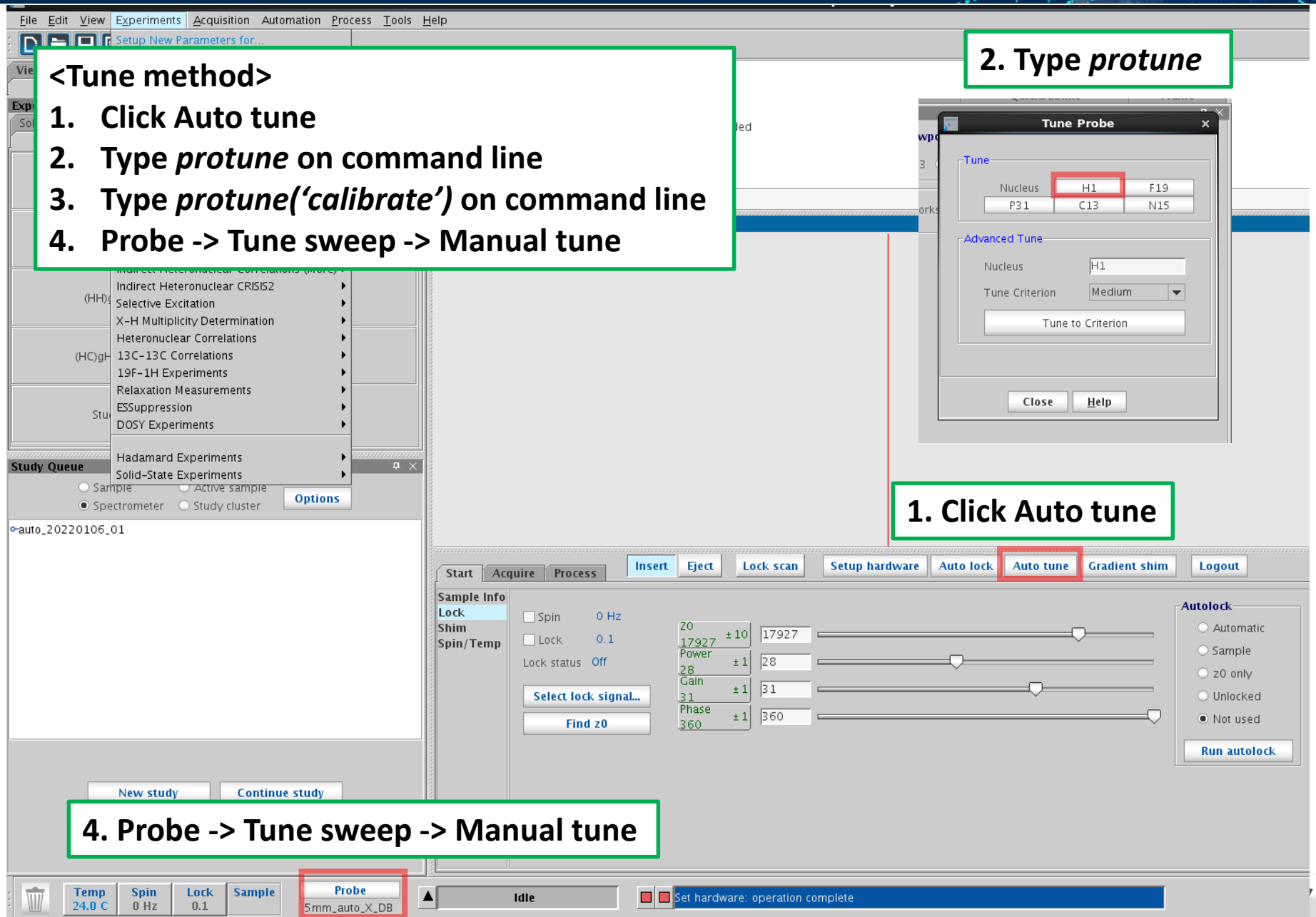
Lock (Manual)


Exp: 100 Seq: PROTON Index: 1
7
Z0 Direction
Start Acquire Process
Insert Eject Lock scan Setup hardware Auto lock Auto tune Gradient shim Logout
Sample Info
Lock
Shim
Spin/Temp
Spin 0 Hz
Lock 67.2
Lock status Regulated
Select lock signa
Find z0
7
Z0 12665 ±10 12665
Power 46 ±1 46
Gain 36 ±1 36
Phase 359 ±1 359
6
Autolock
Not used
Run autolock
Decrease Power, Gain
Interactive
Set hardware: operation complete

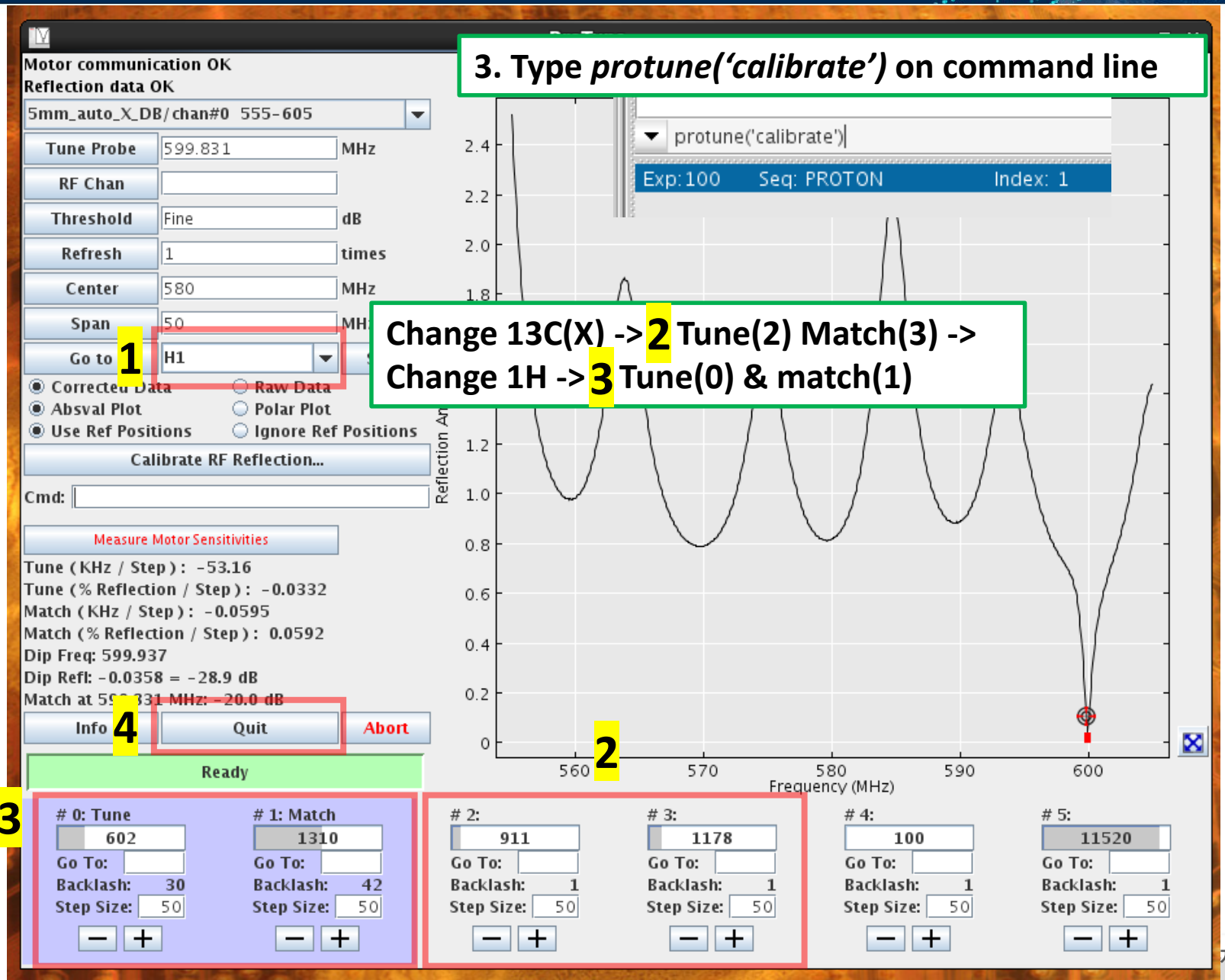

On resonance
Z0 Direction

6-2. Tune & Match

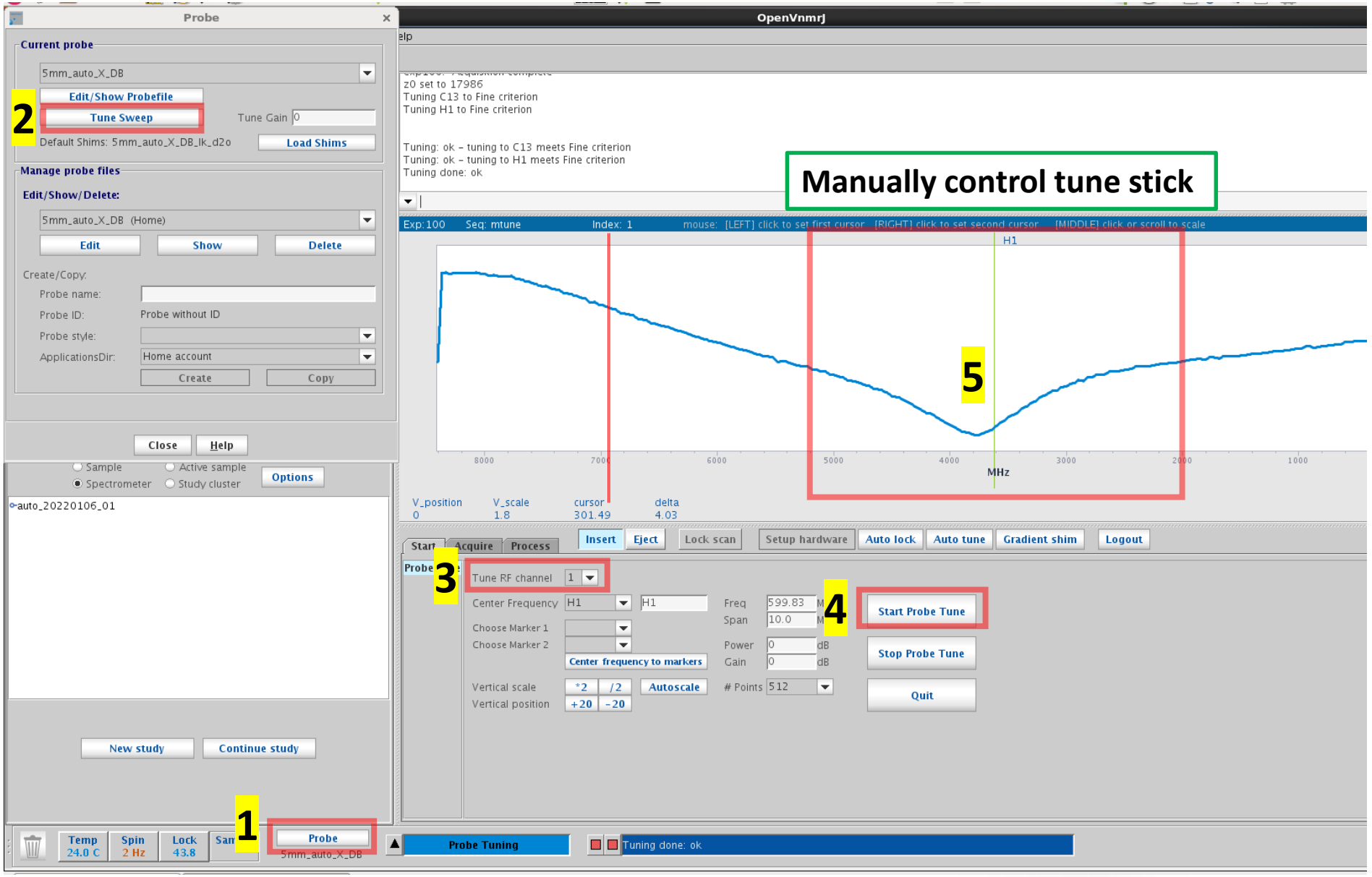
Tuning and Matching



Protune('Calibrate')

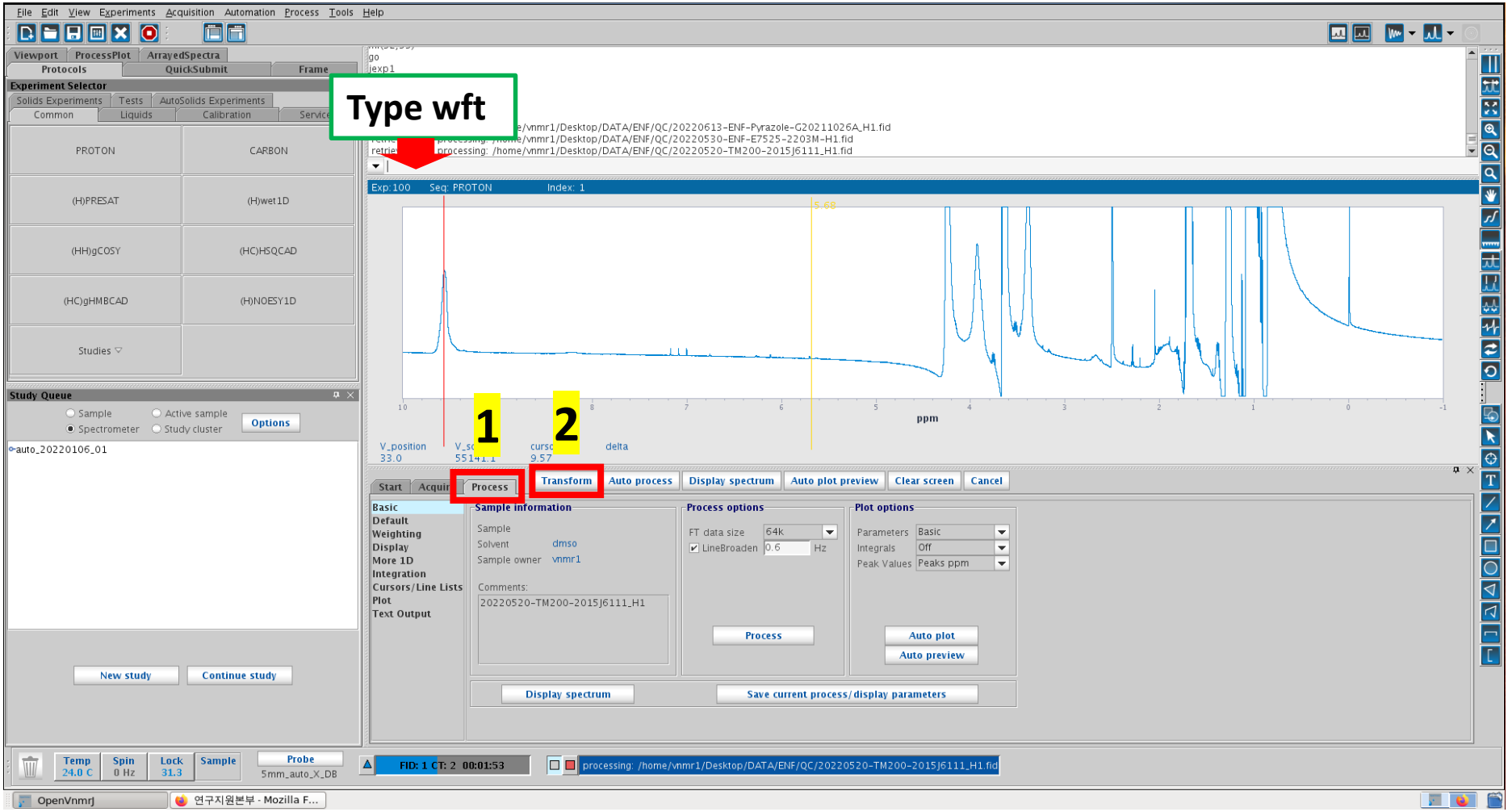


Manual tune (Original process)

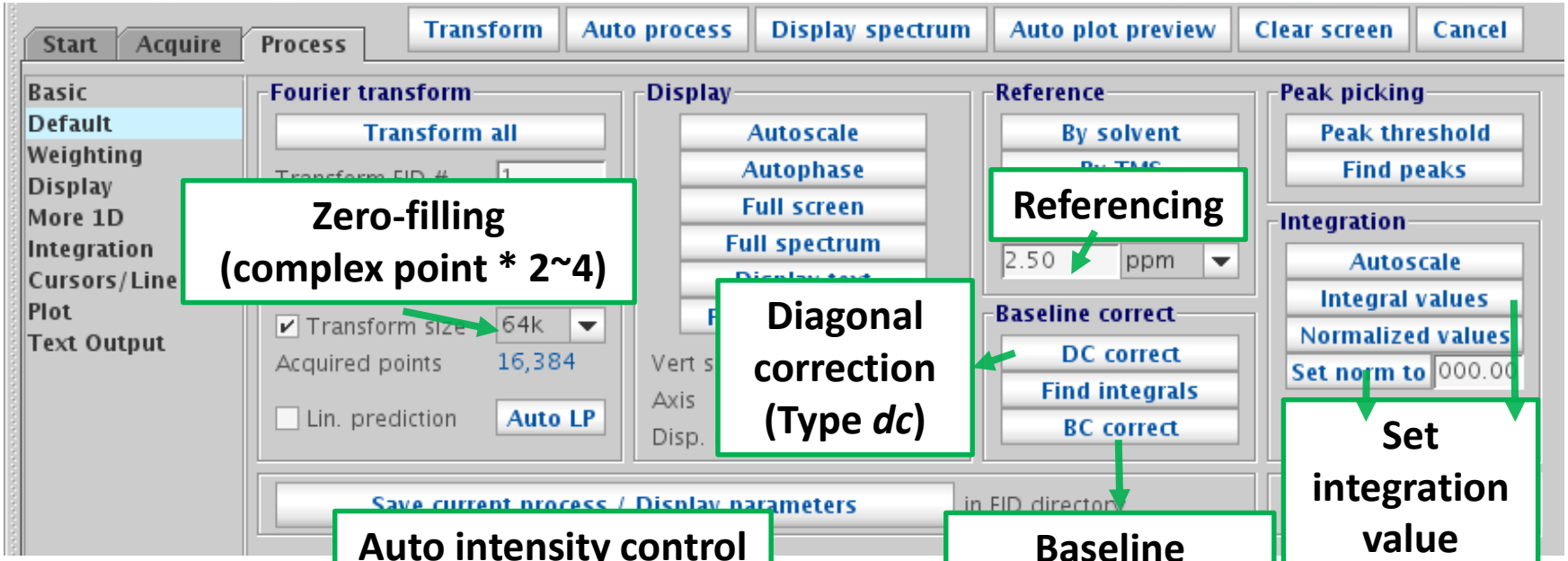


6-3. Processing

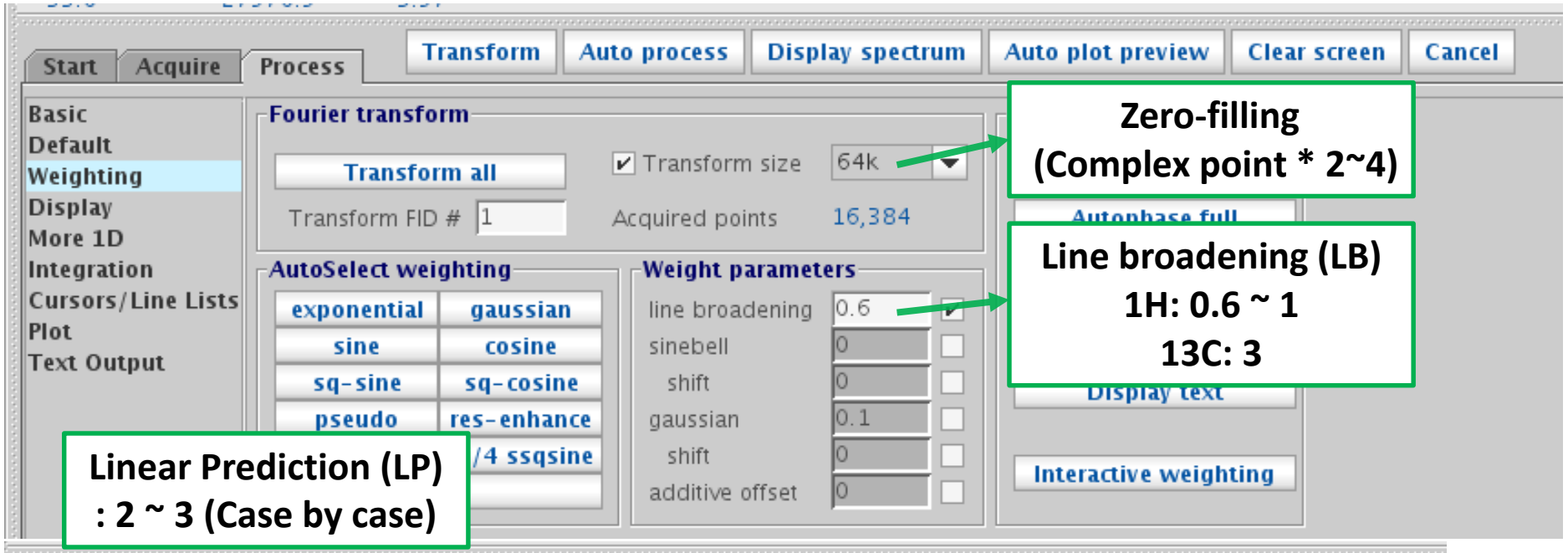
Transformation



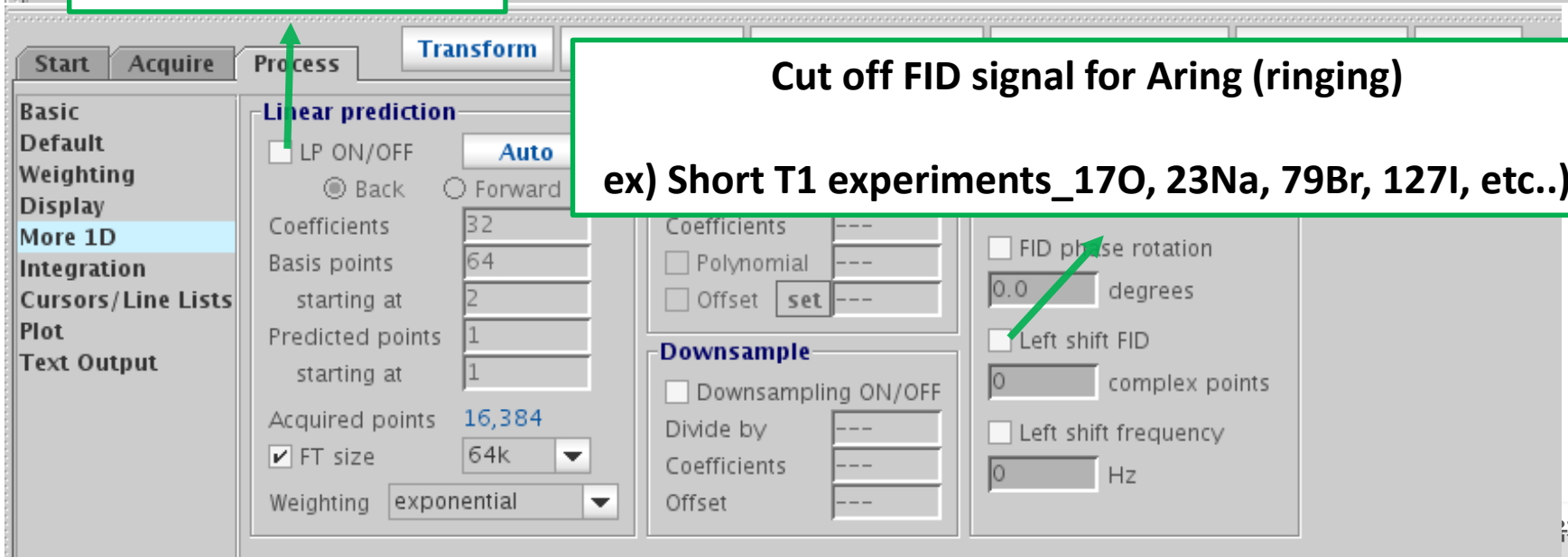
Process tab


Start
Acquire
Process
Transform
Auto process
Display spectrum
Auto plot preview
Clear screen
Cancel
Basic
Default
Weighting
Display
More 1D
Integration
Cursors/Line
Plot
Text Output
Fourier transform
Transform all
Transform size
64k
Acquired points
16,384
Lin. prediction
Auto LP
Display
Autoscale
Autophase
Full screen
Full spectrum
Reference
By solvent
2.50
ppm
Baseline correct
DC correct
Find integrals
BC correct
Peak picking
Peak threshold
Find peaks
Integration
Autoscale
Integral values
Normalized values
Set norm to
Save current process / Display parameters
Zero-filling
(complex point * 2~4)
Referencing
Diagonal
correction
(Type dc)
Set
integration
value
Auto intensity control
(Type vsadj)
Baseline
correction
on
Integration area
(Type bc)
Auto phase correction
(Type aph)
Display
Autoscale
Autophase
Full screen
Full spectrum
Display text
Find nearest line
Vert scale
Absolute
Axis
PPM
Disp. mode
Phased
Display parameters
Fine Maximum intensity on peak
(Type nl)

Process tab



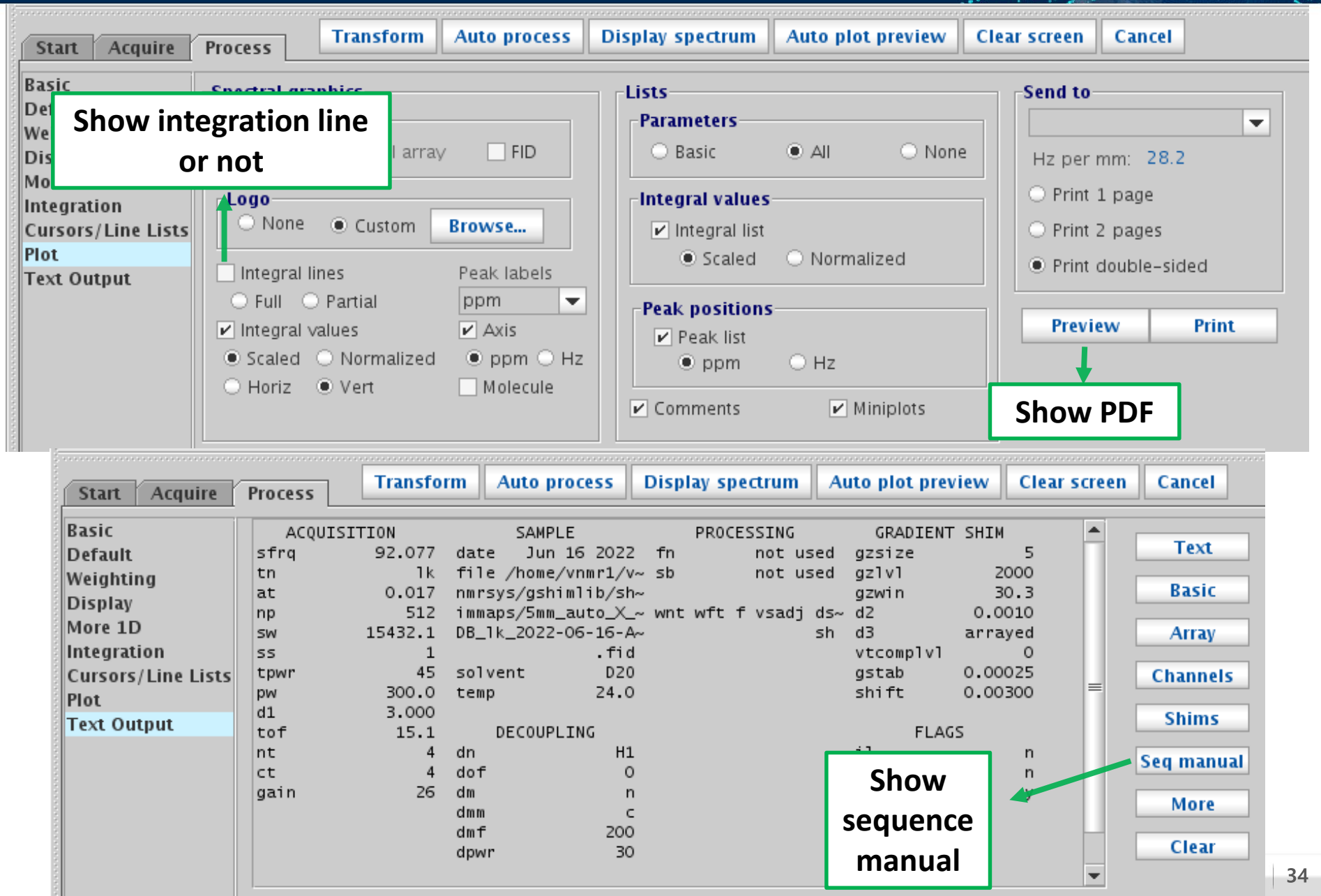
Linear Prediction (LP)
: 2 ~ 3 (Case by case)



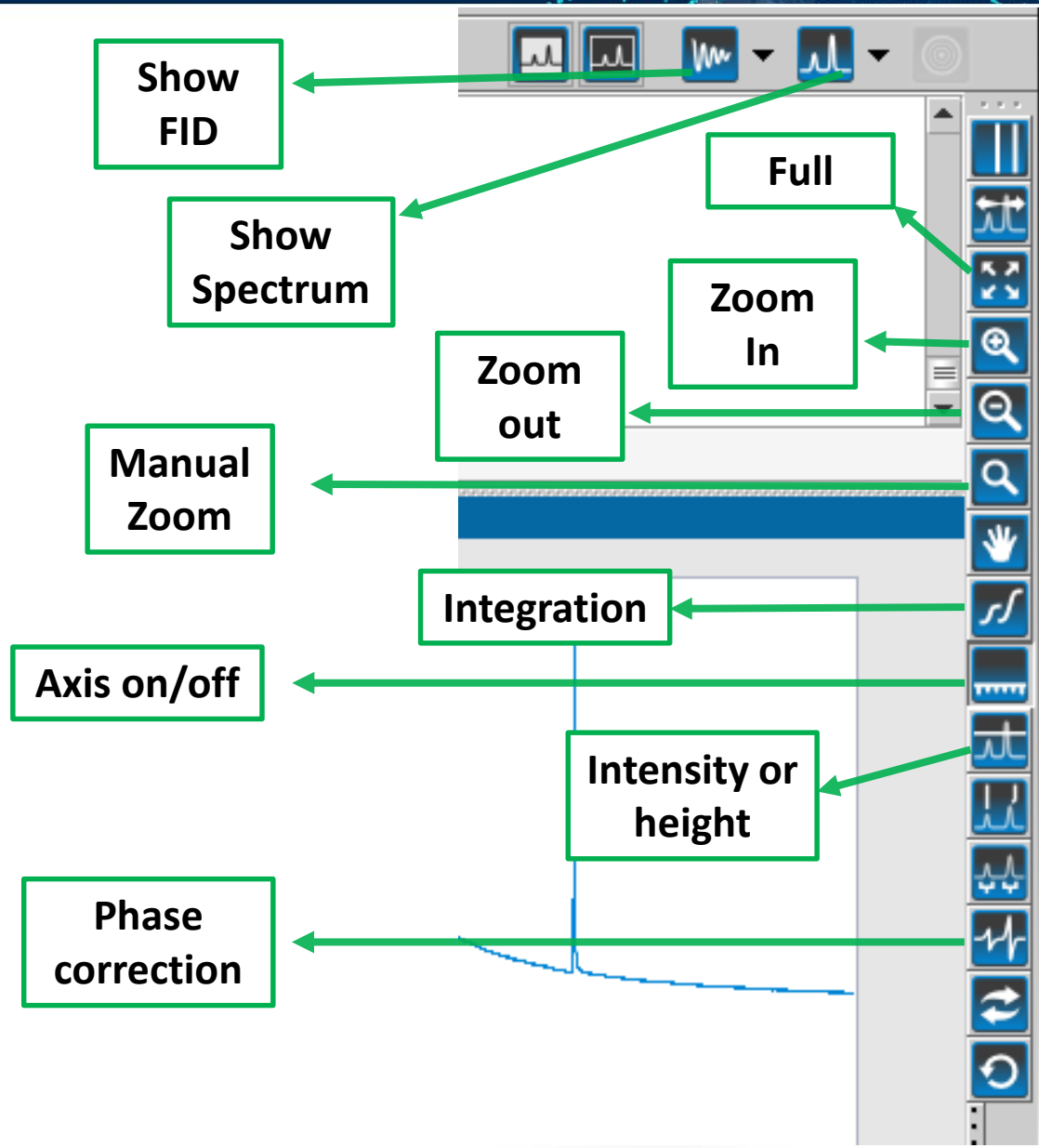
Cut off FID signal for Aring (ringing)

ex) Short T1 experiments_17O, 23Na, 79Br, 127I, etc..)

Process tab


Start
Acquire
Process
Transform
Auto process
Display spectrum
Auto plot preview
Clear screen
Cancel
Basic
Integration
Cursors/Line Lists
Plot
Text Output
Show integration line or not
Logo
None
Custom
Browse...
Integral lines
Full
Partial
Integral values
Scaled
Normalized
Horiz
Vert
Peak labels
ppm
Axis
ppm
Hz
Molecule
FID
Lists
Parameters
Basic
All
None
Integral values
Integral list
Scaled
Normalized
Peak positions
Peak list
ppm
Hz
Comments
Miniplots
Send to
Hz per mm: 28.2
Print 1 page
Print 2 pages
Print double-sided
Preview
Print
Show PDF
Start
Acquire
Process
Transform
Auto process
Display spectrum
Auto plot preview
Clear screen
Cancel
Basic
Default
Weighting
Display
More 1D
Integration
Cursors/Line Lists
Plot
Text Output
ACQUISITION
sfrq 92.077
tn 1k
at 0.017
np 512
sw 15432.1
ss 1
tpwr 45
pw 300.0
d1 3.000
tof 15.1
nt 4
ct 4
gain 26
SAMPLE
date Jun 16 2022
file /home/vnmr1/v~
nmrsys/gshimlib/sh~
immaps/5mm_auto_X_~
DB_1k_2022-06-16-A~
.fid
solvent D20
temp 24.0
DECOUPLING
dn H1
dof 0
dm n
dmm c
dmf 200
dpwr 30
PROCESSING
fn not used
sb not used
wnt wft f vsadj ds~
sh
GRADIENT SHIM
gzsize 5
gzlvl 2000
gzwin 30.3
d2 0.0010
d3 arrayed
vtcomplvl 0
gstab 0.00025
shift 0.00300
FLAGS
Text
Basic
Array
Channels
Shims
Seq manual
More
Clear
Show sequence manual

Side tab


Show FID
Show Spectrum
Full
Zoom In
Zoom out
Manual Zoom
Integration
Axis on/off
Intensity or height
Phase correction

7. Maintenance

Shim

Affecting factors for Shimming

1. Temperature gradient (Convection effect)
2. Sample tube (Camber, Thickness, Scratch, Material quality, Impurity contents)
3. Sample height
4. Sample (Concentration, Paramagnetic species, Impurity, Ion/salt, Particle)
5. Solvent (Viscosity, Dielectric constant)

Axial shims(on axis): Z1, Z2, Z3, Z4, Z5, Z6, etc..

Radial shims(off axix): X, Y, XY, XZ, YZ, X2Y2, etc..

Ex) X = X+Z0, XZ = X + Z1

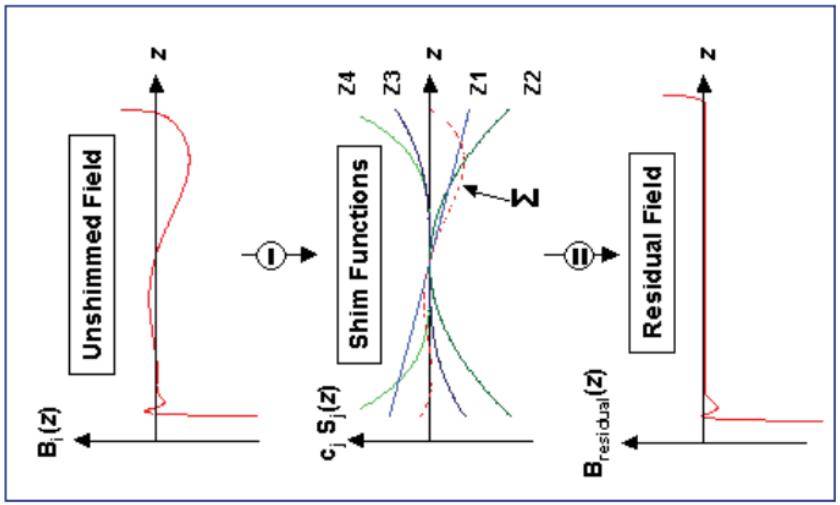
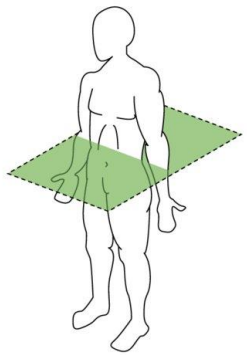
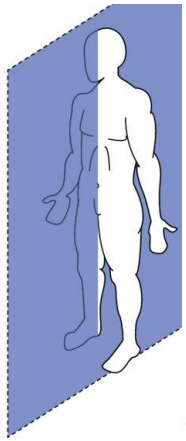


Fig. 7: The 1D gradient shimming algorithm. In a one-time calibration experiment it is necessary to use the method of Fig. 5 to measure the actual

Transverse plane (Radial) X, Y shims



Longitudinal plane (Axial) Z shims

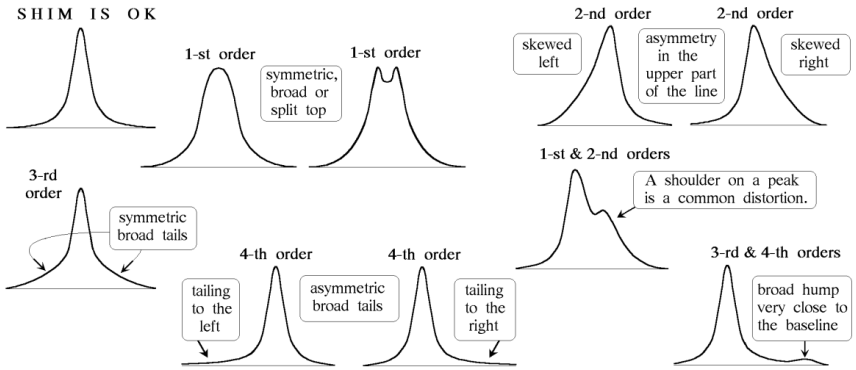


Class of shim coil

SHIM CONTROLS ON THE SPECTROMETERS					spinning/ non-spin	high/ low	odd/ even	order
FX-90Q	WM-360	MSL-300	AC-300	AMX-600				
Y	Z	Z	Z	Z	spinning	low	odd	1-st
C	Z^2	Z^2	Z^2	Z^2	spinning	low	even	2-nd
Y^3	Z^3	Z^3	Z^3 Z^5	Z^3 Z^5	spinning spinning	high high	odd odd	3-rd 5-th
F	Z^4	Z^4	Z^4	Z^4	spinning	high	even	4-th
Z X	X Y	X Y	X Y	X Y	non-spin non-spin	low low	odd odd	1-st 1-st
ZX XY YZ X^2-Y^2	XY YZ XZ X^2-Y^2	XY YZ XZ X^2-Y^2	XY YZ XZ X^2-Y^2	XY YZ XZ X^2-Y^2	non-spin non-spin non-spin non-spin non-spin	low low low low low	even even even even even	2-nd 2-nd 2-nd 2-nd 2-nd
X^3 Z^3	X^3 Y^3	X^3 Y^3 XZ^2 YZ^2	X^3 Y^3 XZ^2 YZ^2	X^3 Y^3 XZ^2 YZ^2 ZXY $Z(X^2-Y^2)$	non-spin non-spin non-spin non-spin non-spin non-spin	high high high high high high	odd odd odd odd odd odd	3-rd 3-rd 3-rd 3-rd 3-rd 3-rd
				XZ^3 YZ^3	non-spin non-spin	high high	even even	4-th 4-th
Y	Z				<--- spinning axis for NMR tubes			

Doped 1% H2O in D2O
(0.1 mg/ml GdCl3 in sample)

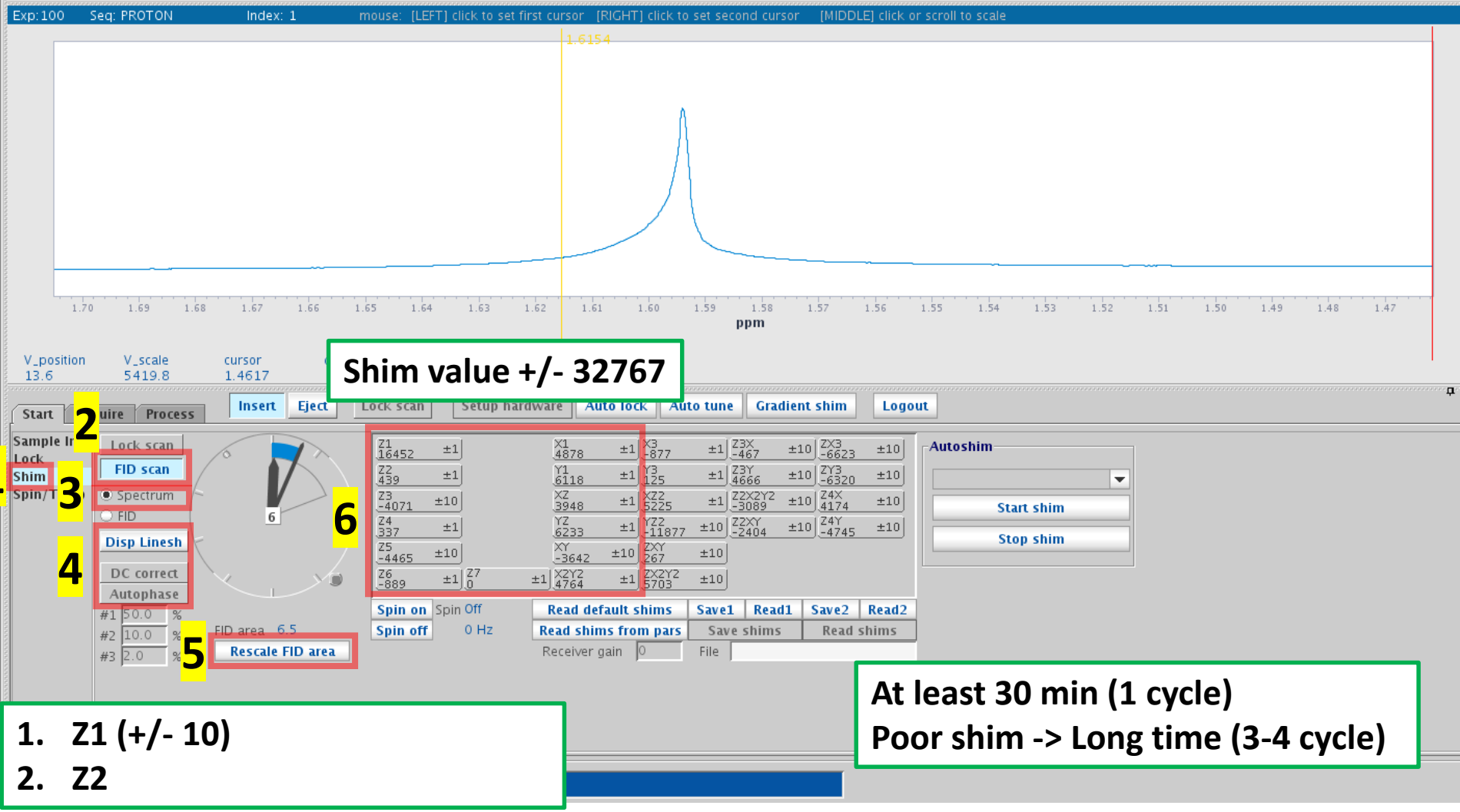
Bruker 400 MHz linewidth 4 Hz
Varian 600 MHz linewidth 6 Hz



SKETCHES OF LINE DISTORTIONS CAUSED BY VARIOUS GRADIENTS

https://scs.illinois.edu/system/files/inline-files/ShimmingNMR_Magnet.pdf

Manual shim



1. Z1 (+/- 10)
2. Z2

At least 30 min (1 cycle)
Poor shim -> Long time (3-4 cycle)

Manual shim

<Line shape sample>
1H: CHCl3 in Acetone-D6
13C: Dioxane in Benzene-D6

<Routine shim>
1) Spin off
2) Adjust Lock level 80
3) Z1 -> Z2 -> Z1
4) Z3 -> Z1 -> Z2 -> Z3 -> Z1 -> Z2
5) Z4 -> Z1 -> Z2 -> Z4 -> Z1 -> Z2
6) Turn off spin -> Repeat step 3), 4), 5)
7) Turn on spin -> 3)

<Remove spinning sideband>
1) Spin off
2) Adjust Lock level 80
3) X-Y-X-Y
4) X-ZX-X
5) Y-ZY-Y
6) X
7) XY-ZXY
8) X-Y-X-Y
9) Spin on
10) Z4 (asymmetry peak)
11) Z3-Z1-Z2

Typical interactions for axial shims:

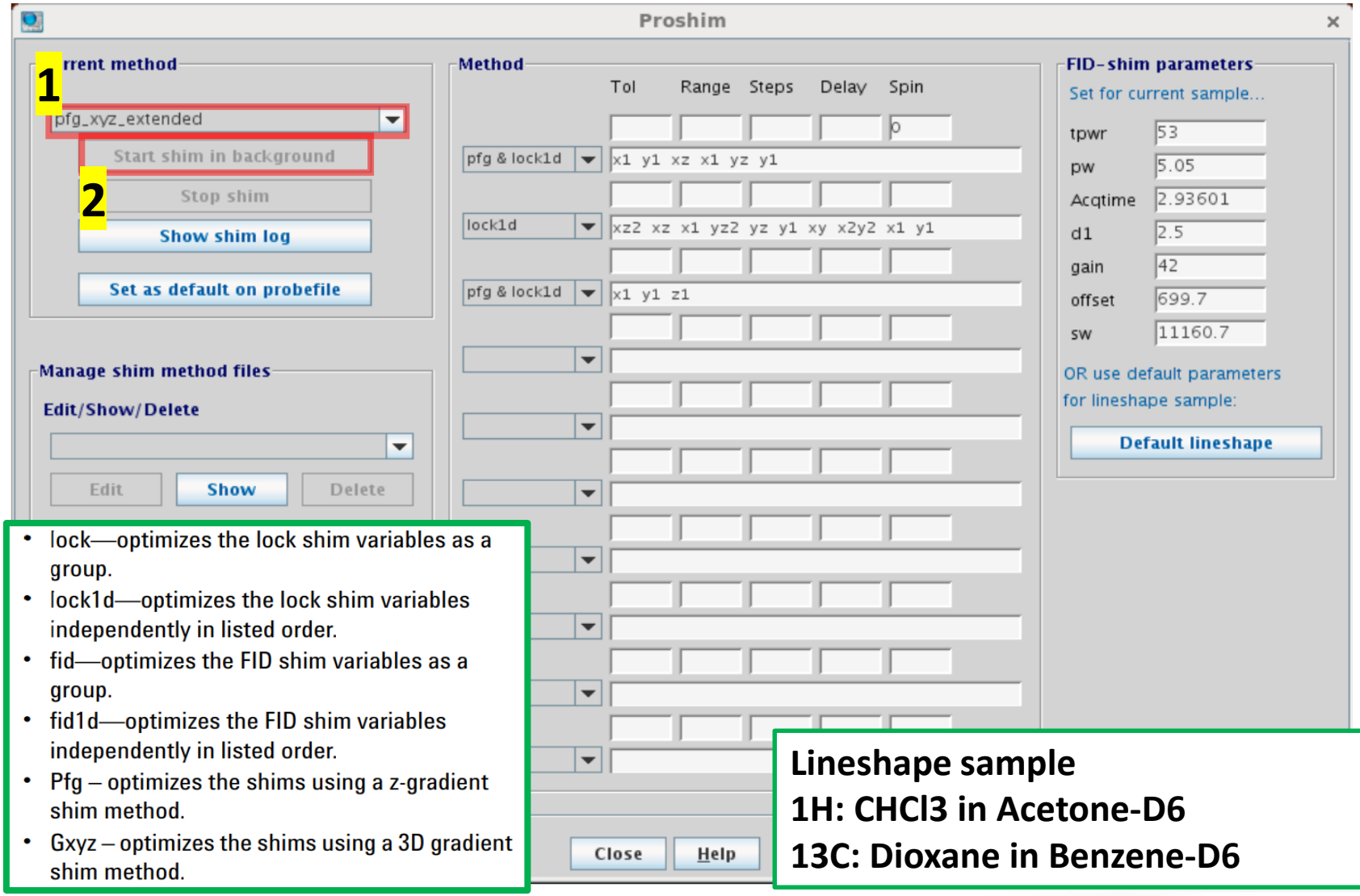
- Z1 and all other axial shims, to some extent
- Z2 and Z1
- Z3 and Z1
- Z4 and Z2 (with large delta Z4s: Z4 and Z3)
- Z5 and both Z3 and Z1 (Z5 not available on 13-channel shim systems)

Table 42. Lineshape Effects and Their Associated Shims

Lineshape Effect	*Shims*
Split peak	Z4 and Z1
Asymmetry greater than half-way up	Z2
Asymmetric foot	Z4
Symmetric feet and or low broad base	Z5
Symmetrically broad base	Z3
Spinning sidebands	Low-order radials X1, Y1
Symmetric broad base	High-order radials X3, Y3, etc.

Shim editors

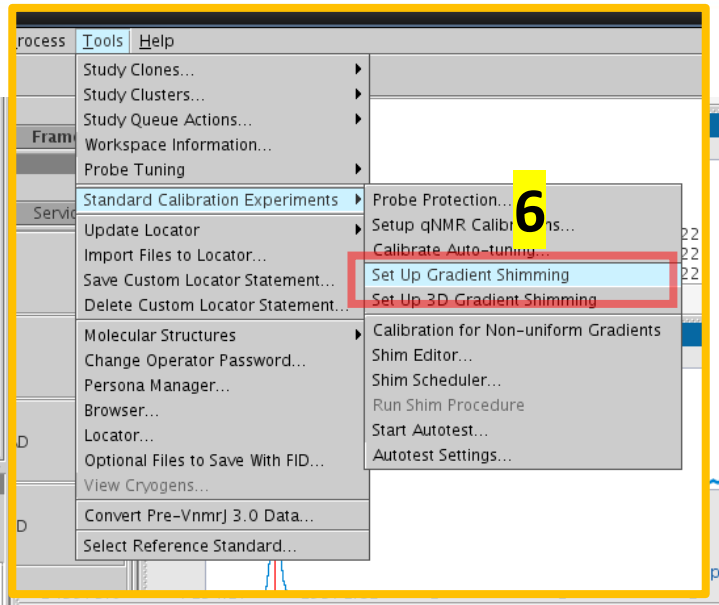
Tools > Standard Calibration Experiments > Shim Editor



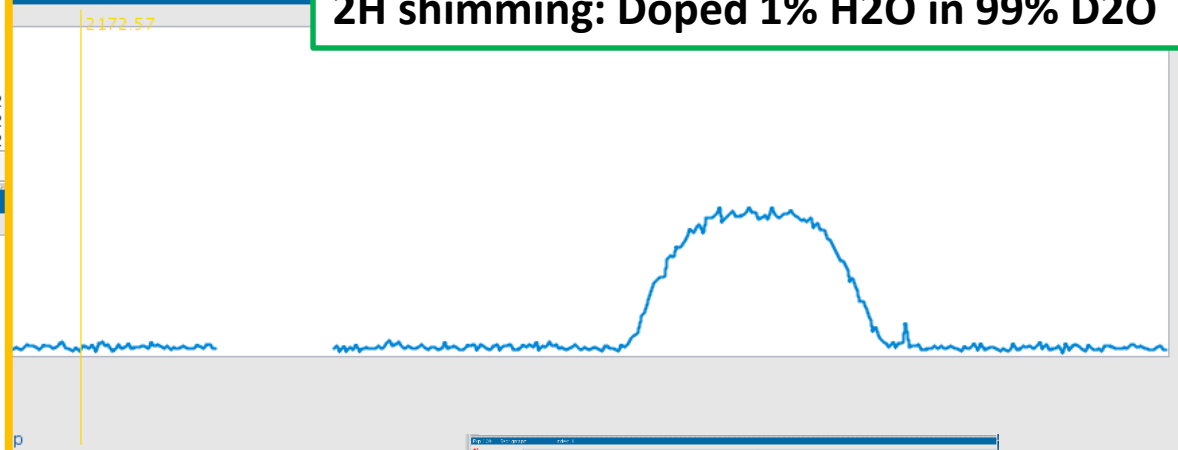
- lock—optimizes the lock shim variables as a group.
- lock1d—optimizes the lock shim variables independently in listed order.
- fid—optimizes the FID shim variables as a group.
- fid1d—optimizes the FID shim variables independently in listed order.
- Pfg – optimizes the shims using a z-gradient shim method.
- Gxyz – optimizes the shims using a 3D gradient shim method.

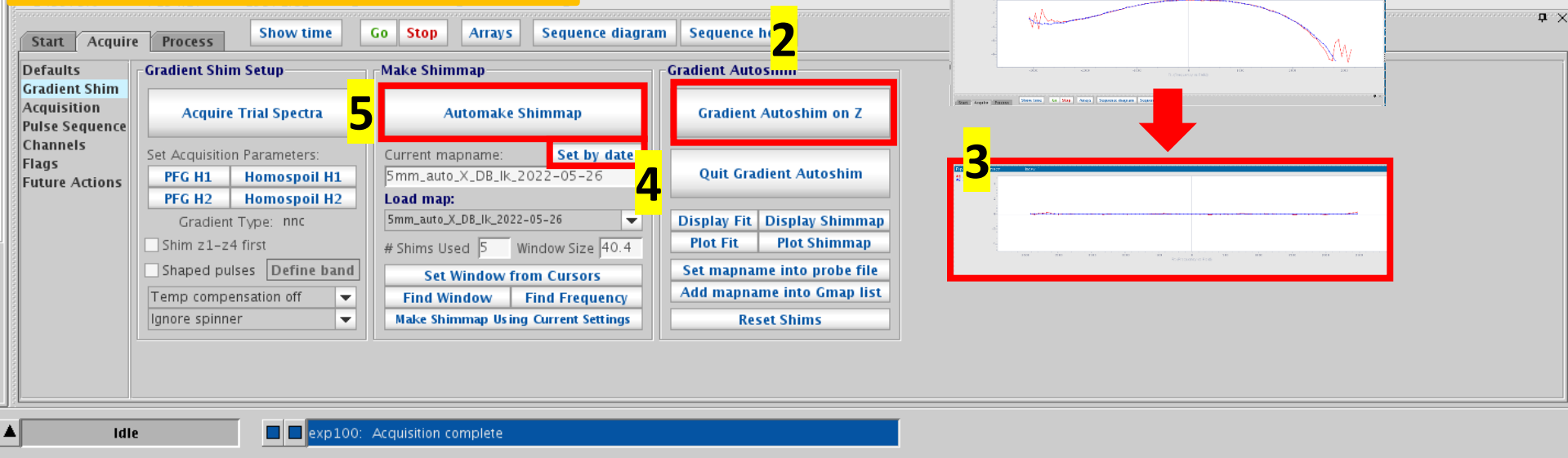
Lineshape sample
1H: CHCl3 in Acetone-D6
13C: Dioxane in Benzene-D6

Create Gradient shim-map (Simple)

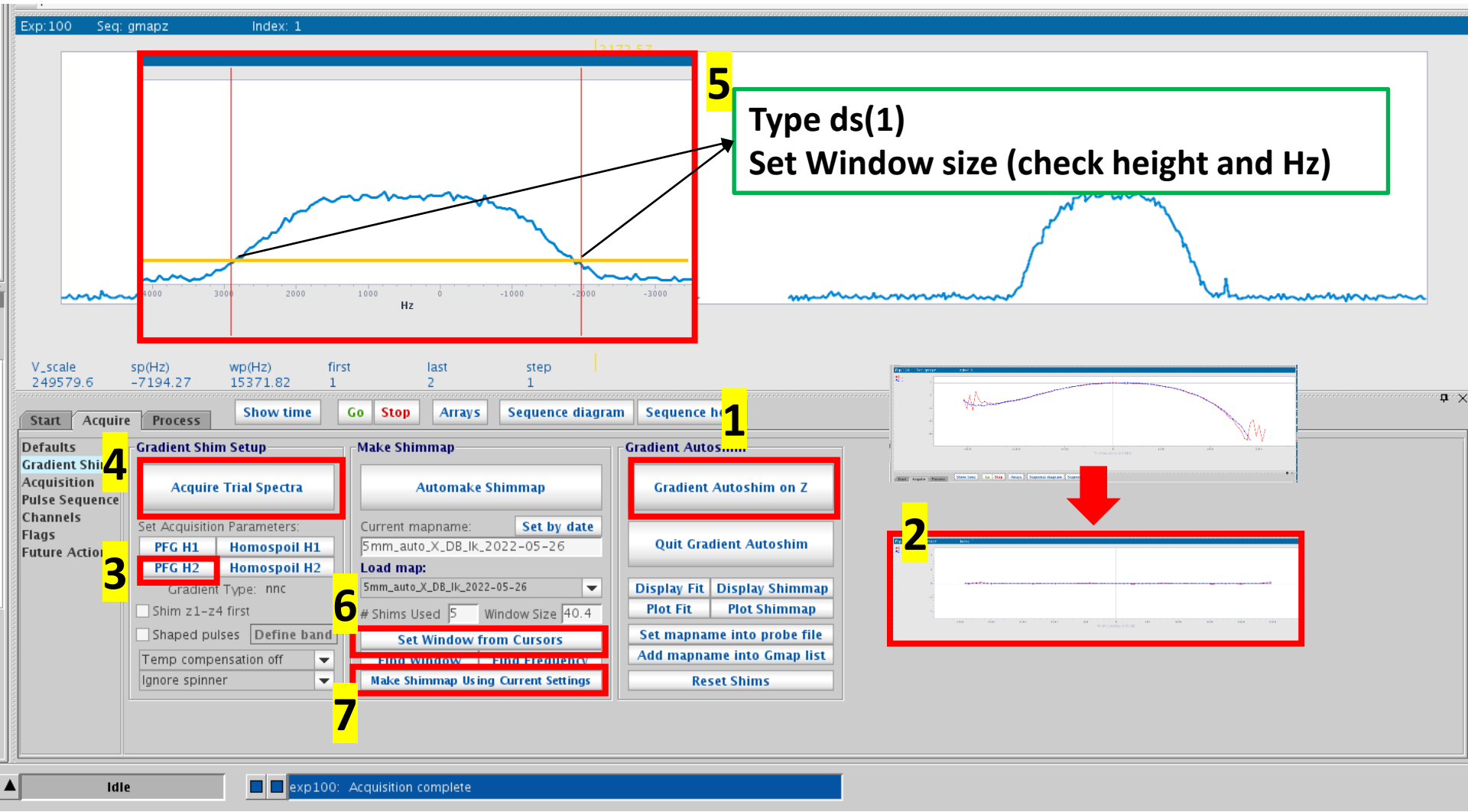


1H shimming: 10% H2O in 90% D2O
2H shimming: Doped 1% H2O in 99% D2O

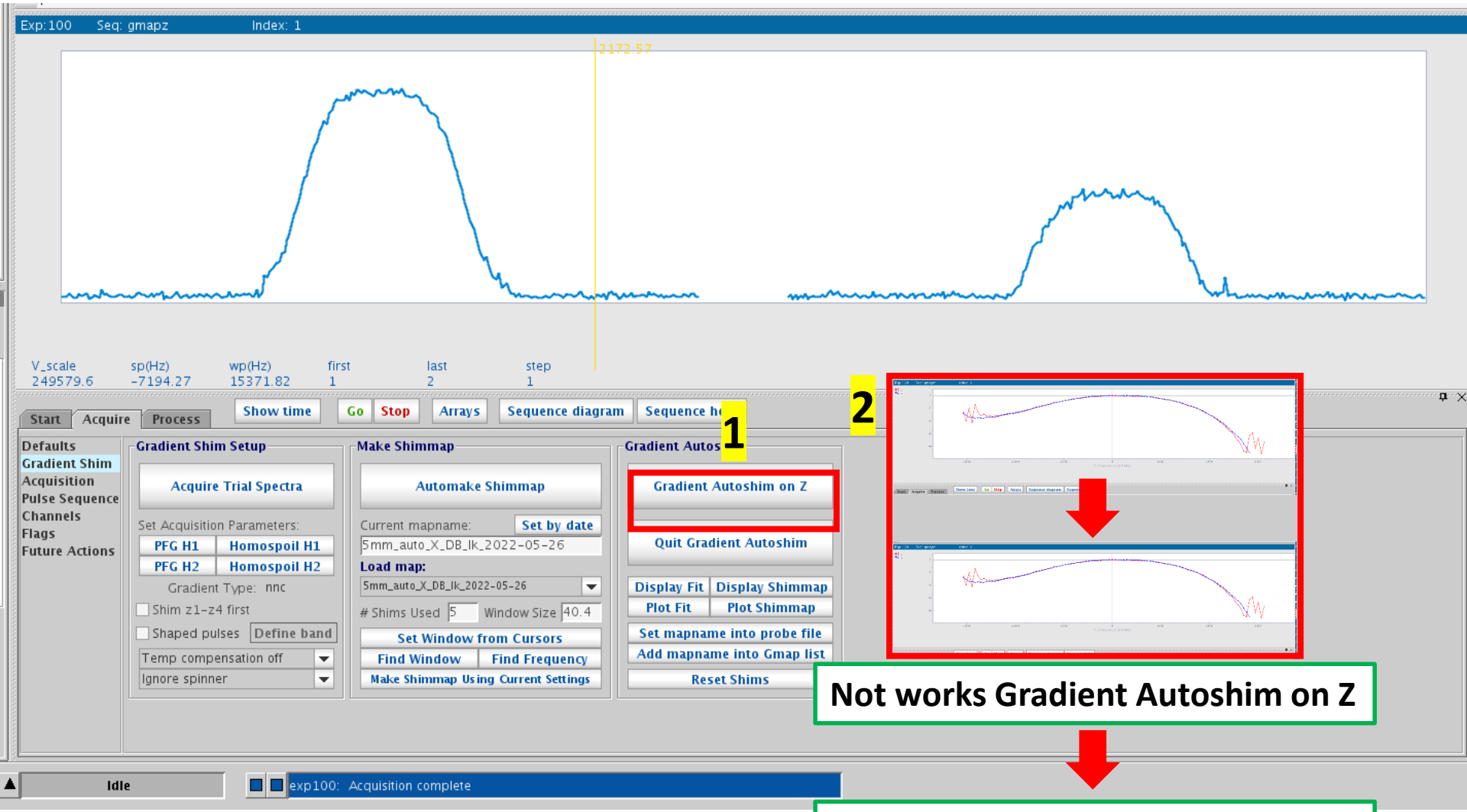




Create Gradient shim-map (Manually)



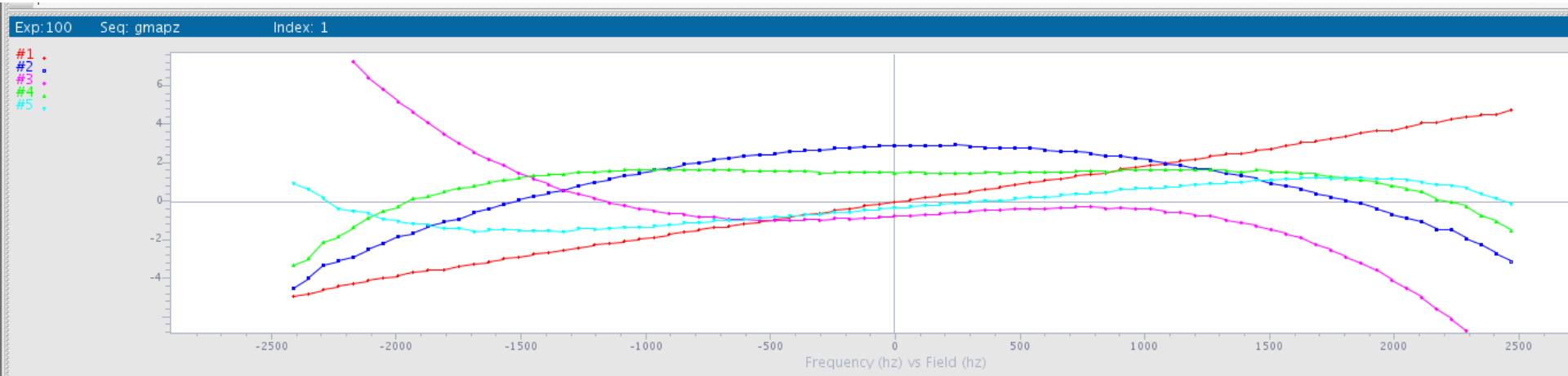
Create Gradient shim-map (Trouble shooting)

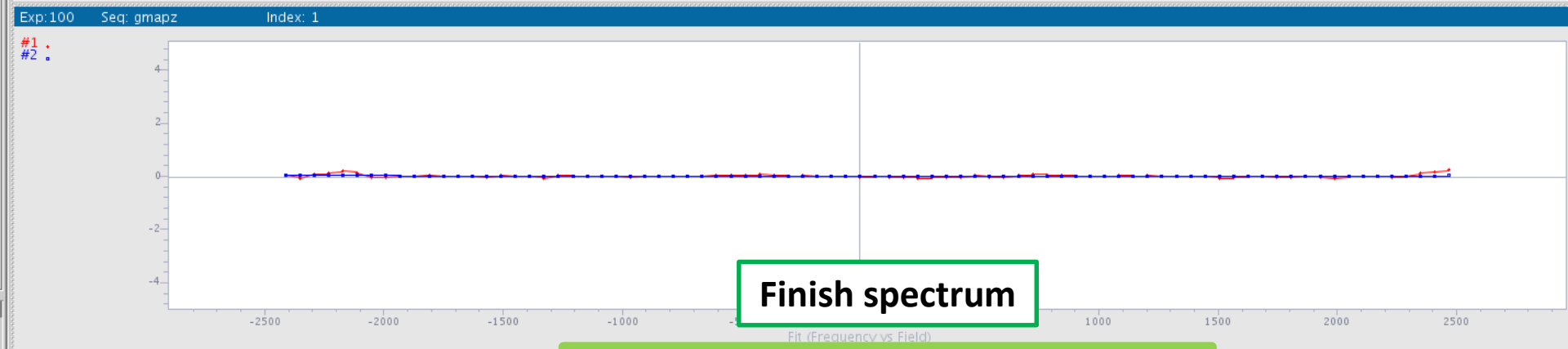


Not works Gradient Autoshim on Z

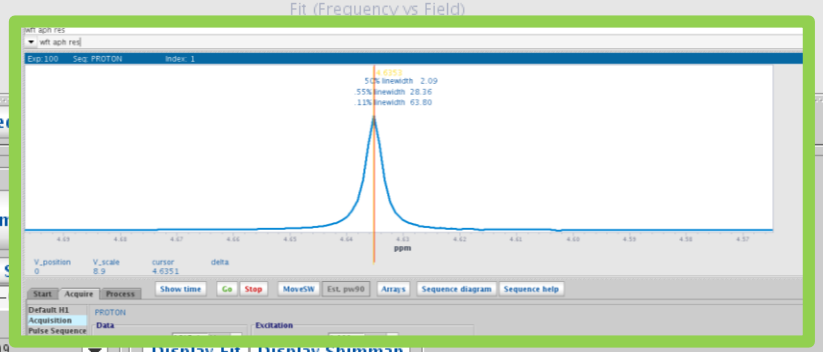
Try to use 3D shim or Shim editor

Finish shim-map

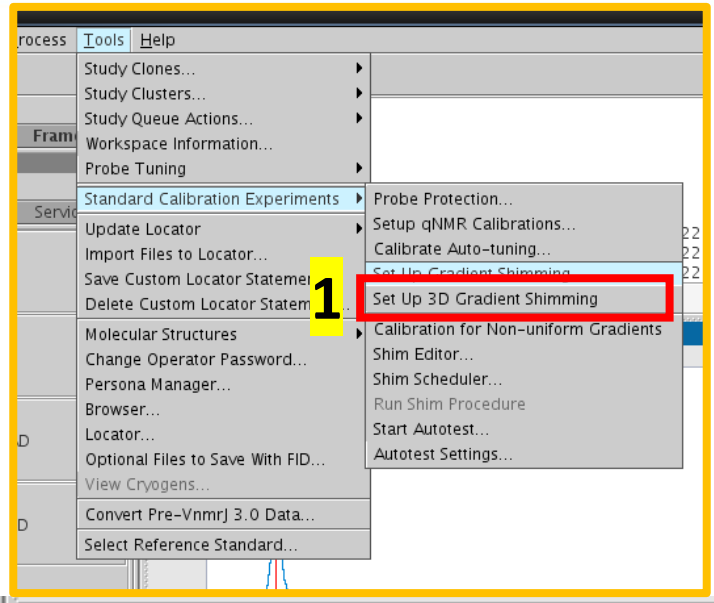




Finish spectrum



3D shim



At least 30 min for 1 cycle
Poor shim -> Try to use manual shim or shim editors