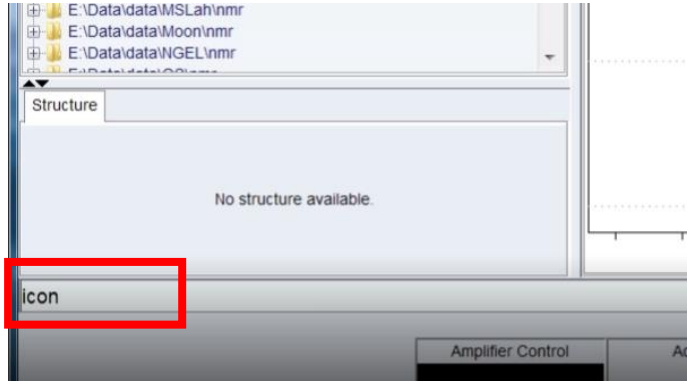
Common SOP

2022-08-17

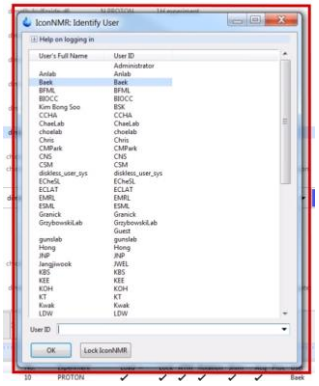
UNIST Central Research Facilities

Sun-Phil Han

Login Icon-NMR (Automation)

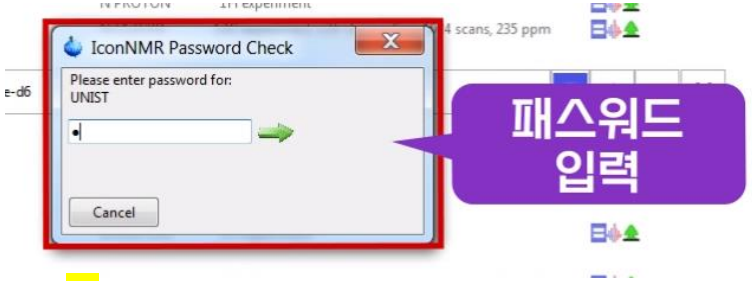


1 Type command ***icon***



3 Select Lab ID

2 Click **Automation**

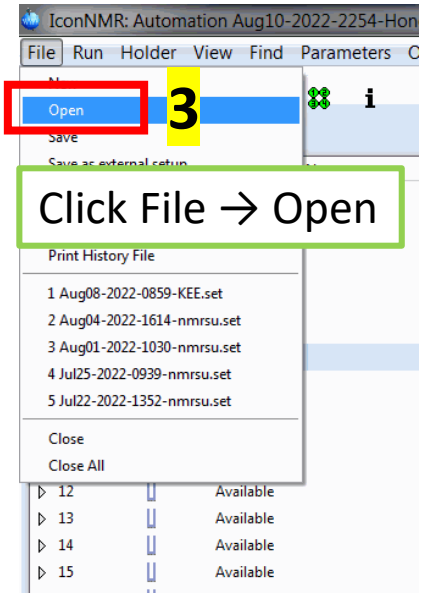


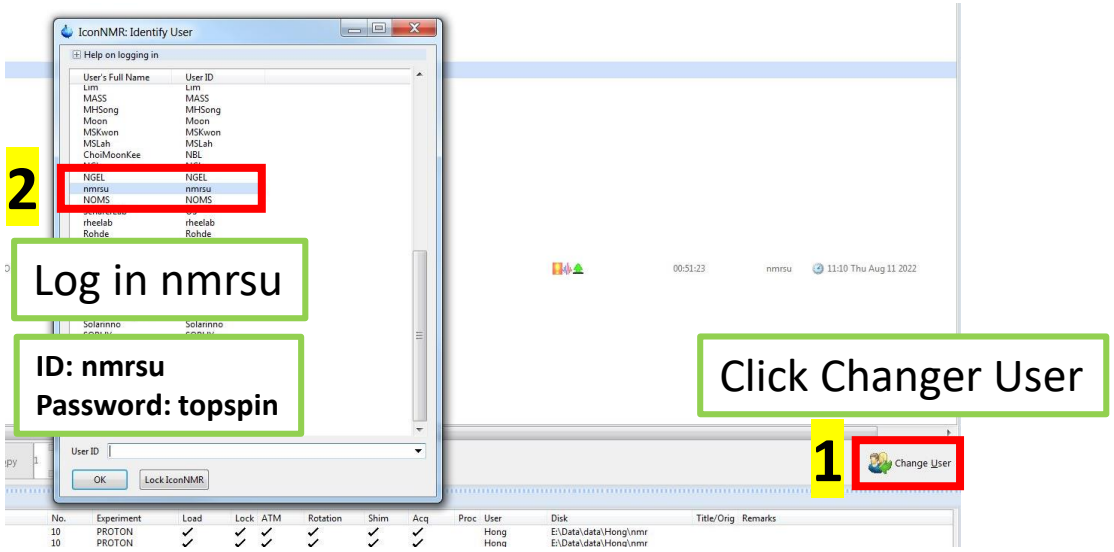
4 Type password

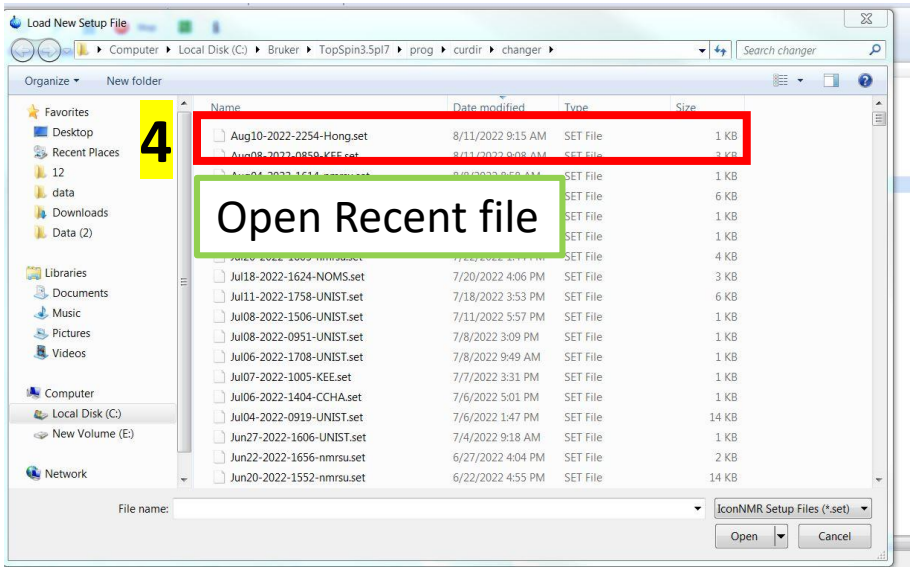
Administrator
ID: nmrsu
Password: topspin

1) Turn off **icon-nmr and topspin**
2) Cancel other user`s experiment
3) Delete fail experiment line

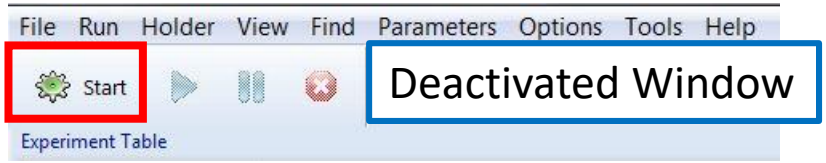
How to reopen last experiment list







5 Activate icon-nmr Window



Precaution

1. Unauthorized students (non-self-users) are prohibited to use it.

2. If subscriber and actual-user is different, the use of the equipment may be limited.

3. Don`t exceed the scheduled time up to

   2 PM to 8 PM on weekdays -> 1 hour

   8 PM to 2 PM and weekend -> unlimited

4. Equipment must use after booking **your name**.

5. Never turn-off '**Topspin 3.5**' and **NMR workstation**.

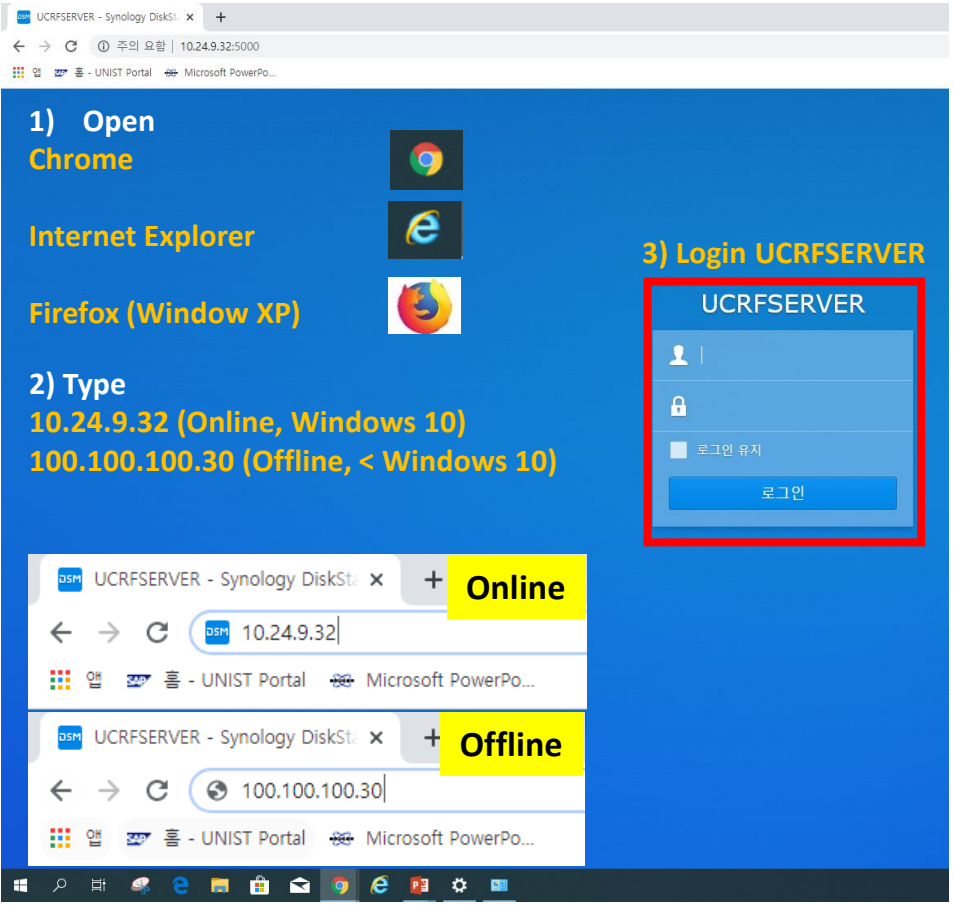
6. Don`t lean your body and attach the ladder to magnet when you replace sample.
   (You pay attention because of behavior affecting Magnet)

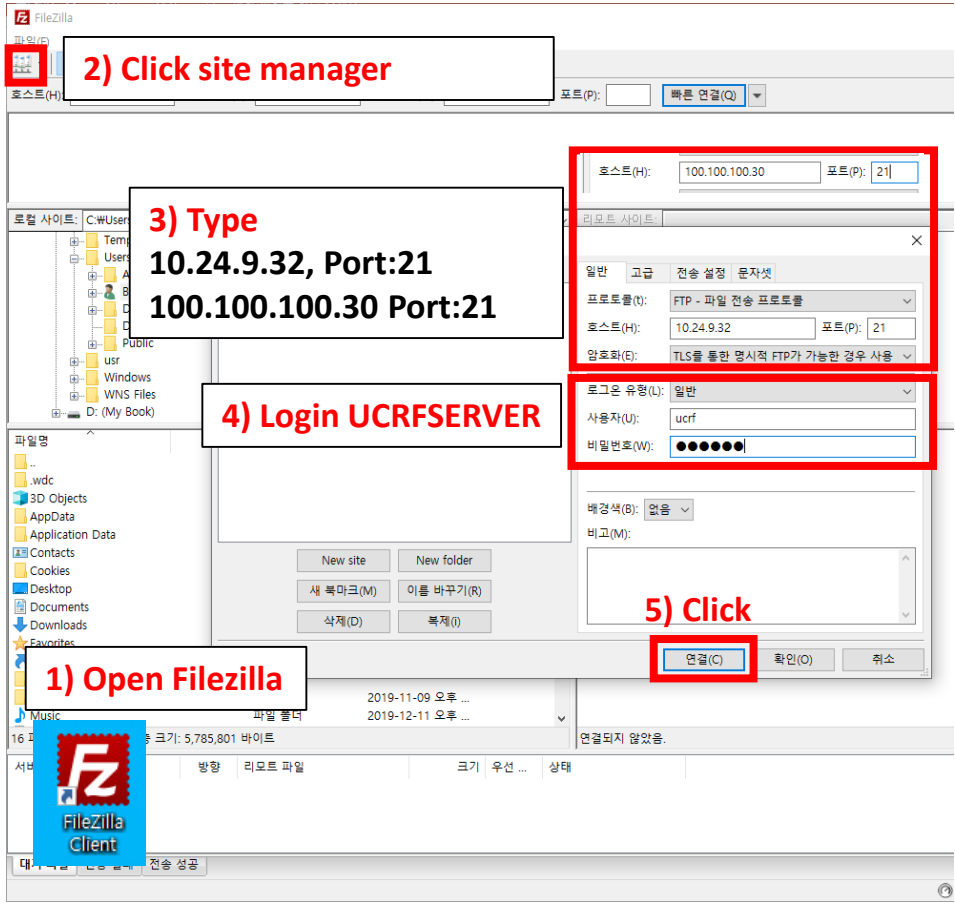
7. Your turn off the lab lights and lock up thoroughly after 6 PM and on weekends.

8. If you have some problem,

Contact me(okno1234@unist.ac.kr, 052-217-4174) or visit Bldg.102 B122(Office)

NAS system - Connection procedure





1. Sample handling

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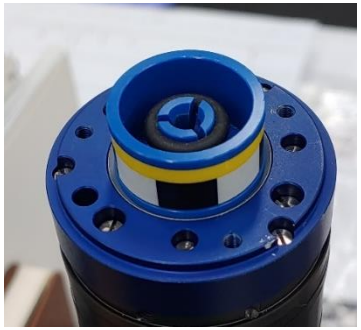
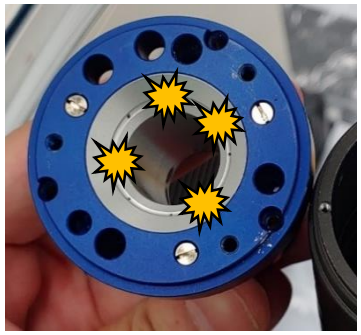
Touch for Spinner, Sample tube

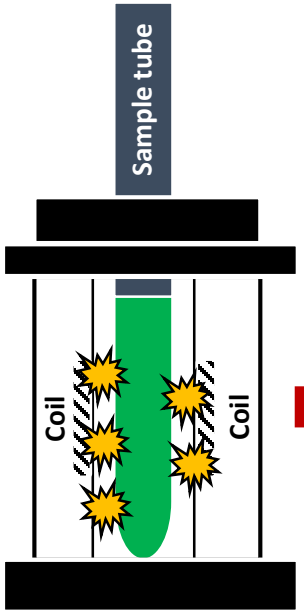
Never touch NMR spinner & sample tube bottom!



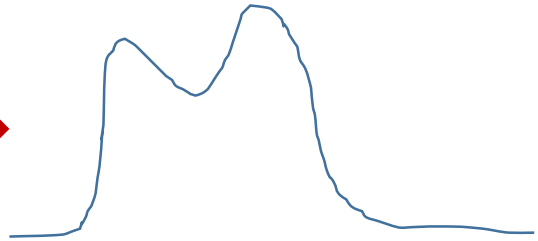


Spinning problem

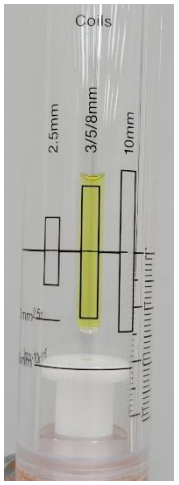
Finger-print, Oil, Dust

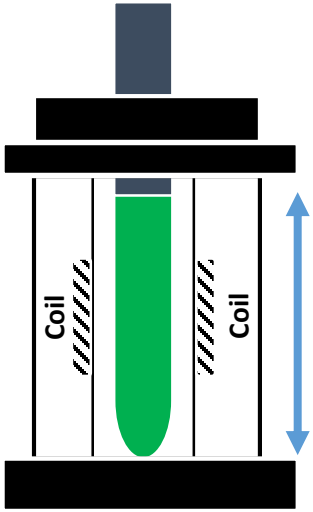


Bad shimming

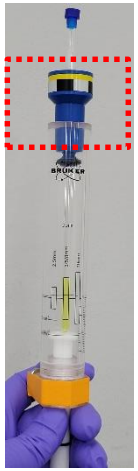


Sample volume and height

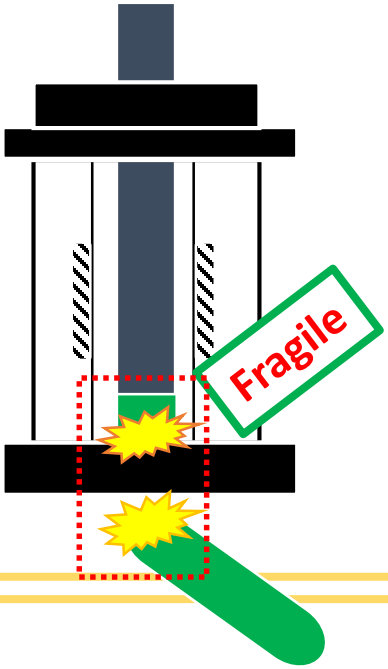




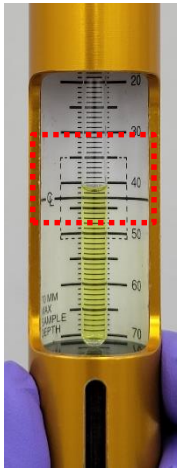
BEST

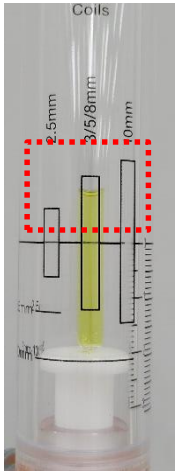


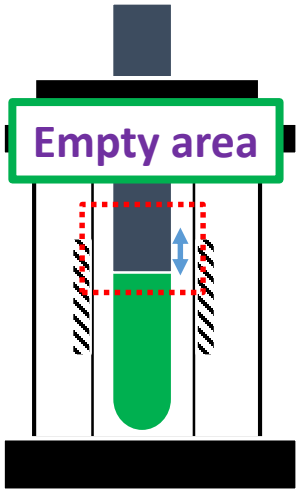
Worst



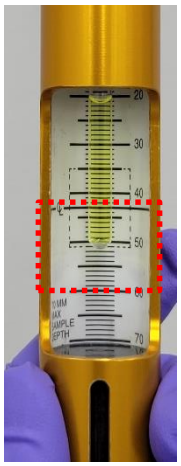
Bad shim

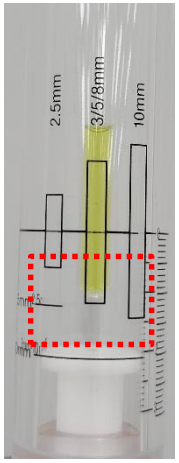


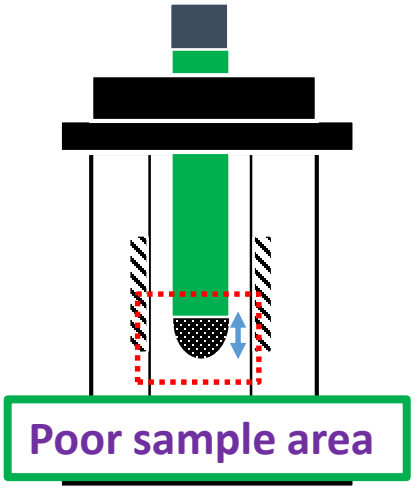




Bad shim







Standard operation procedure

1. Turn on ICONNMR (Automation system)

1) Open Topspin 3.5pl7
2) Type '***icon*** or ***iconnmr***' on command line
3) Click automation

2. Shim failure (for Manually operation)

1) Open Topspin 3.5pl7
2) Type '***rsh***' on command line
3) Double-click recent shim-map

3. To check interval data

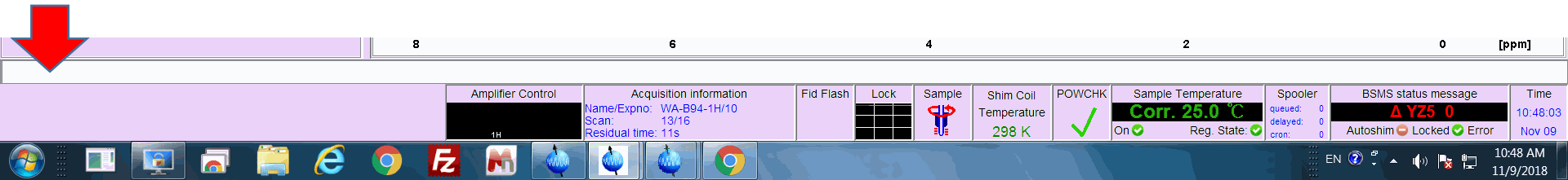
Command line !

1) Type '***tr***' on command line
2) Type '***efp; apk; absn***' on command line

efp = fourier transform

apk = Auto - phase correction

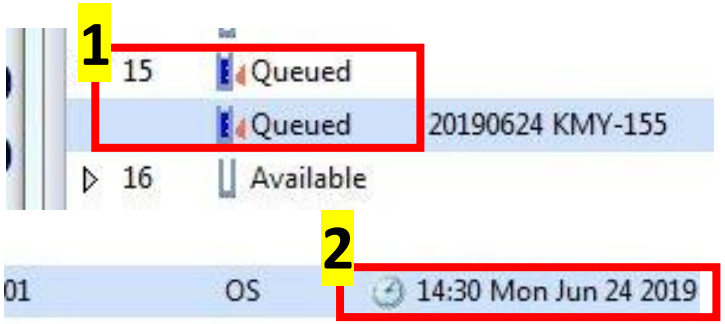
absn = Auto - baseline correction



Standard operation procedure

4. To check reservation & running experiment

1) Check **Queued**
2) Check **Start time**
3) Check **Reservation system**
4) Check **Acquisition information**
5) Check **Spooler**



5. Sava & Stop

1) Type '***halt***' on command line (Only work Acquisition condition)

   # Check Acquisition information "Scan ***n*** / 16"

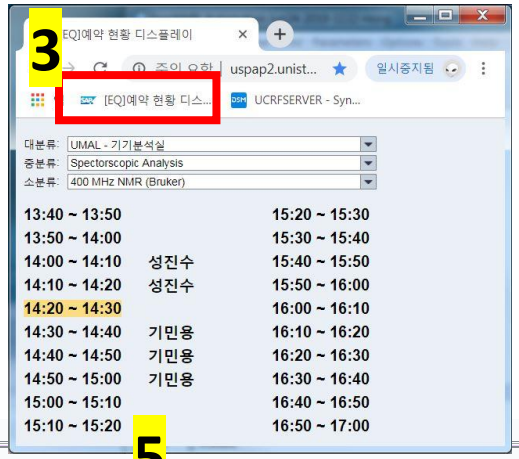
6. Emergency stop

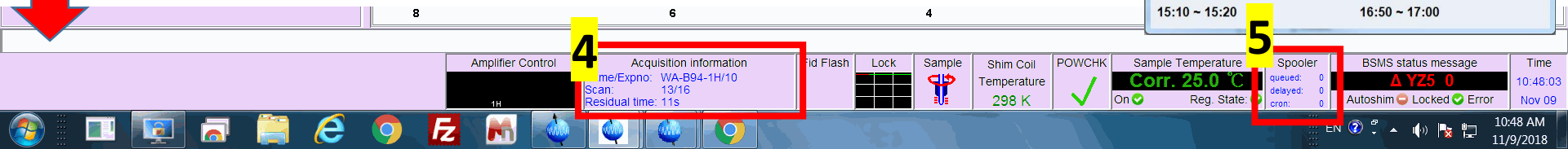
1) Type '***stop***' at least 2 times

   # shim

   # acquisition

Command line !





2. Troubleshooting

Troubleshooting

- **Shimming error**

Massage <Echo time must be reduced>

: Second echo time too long, not enough Magnetization

1. Too short T2

1) High concentration → Dilute → Low concentration

2) High viscosity → Dilute → Low viscosity

3) Paramagnetic materials → …

2. Starting shim is bad (type rsh) – only manual operation

3. Convection (Too many sample volume)

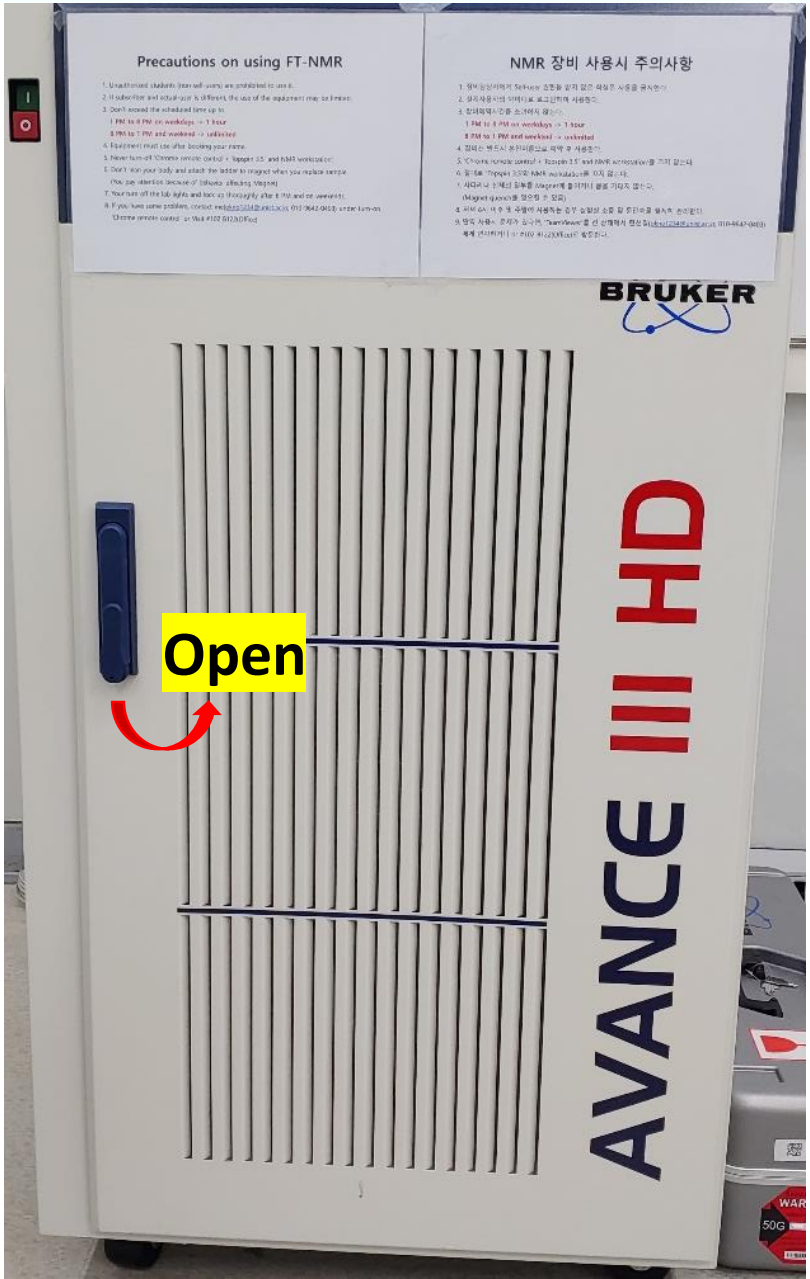
- Unstable temperature (Temperature gradient -> Cause convection)

- **Reduce convention**

- **Increase VT gas flow**
- **Spinning**
- **Low sample temperature**
- **High Viscosity solvent**
- **Shigemi or 3 mm sample tube**

3. Reset console

Reset console



Bruker BioSpin

How to reboot an AV III / AV III HD (for TopSpin 3.x)

BRUKER

2015/03/19

Communication problems between spectrometer and PC

Spectrometer and PC are up and running.

Reboot PC*, spectrometer stays active.

Turn off spectrometer (each unit) - Power Down Procedure for BSMS: **1.** Start Soft Shutdown of BSMS Software (***a)*** ≤ TopSpin 3.2: BSMS web page: ***Main → Service → Soft Shutdown Shims or b)*** ≥ TopSpin 3.5: BSMS Panel: Press ***Shut down BSMS***). **2.** Wait for completion. **3.** Power off BSMS. – Do not turn off the main power cabinet switch!

Spectrometer and PC are already turned off. Ensure that each spectrometer hardware unit has its switch in the position '**Off**'.

Press the green main power cabinet switch.
This is necessary to ensure that the Ethernet-switch is turned on.

Turn on PC*.

Turn on AQS-Rack, if the IPSO does not start, turn it on by pressing the red button.

Turn on all other units (within 20 seconds).

1. Start TopSpin. 2. Create a new data set.
3. Read in a standard Carbon parameter set with TopSpin command ***rpar C13CPD all***.
4. Read in standard pulse definition with TopSpin command ***getprosol***.

- Ignore any upcoming error message in this section.
- Execute ***edhead*** (≤ TopSpin 3.2) or ***edprobe*** (≥ TopSpin 3.5) (if necessary: select the installed probe and define the probe as current probe).
- Execute ***ii*** on the new dataset up to three times, until you do not get an error message anymore.
- Execute ***atma*** to initialize the ATM motor if you have an ATM probe.

If a temperature control unit is available, start temperature control with ***vtudisp*** *(successor of edte)*. Make sure that *heater* is turned on and *air flow* is set to the value you want.

Your spectrometer is up and running.

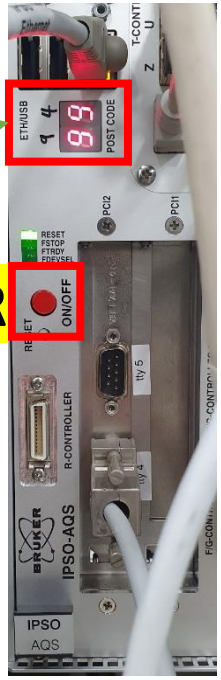
* Login and wait until all server processes have been started. Do not start TopSpin!
Necessary Services under: 1) Windows: dhcpd.exe, tftpd32.exe 2) Linux: dhcpd

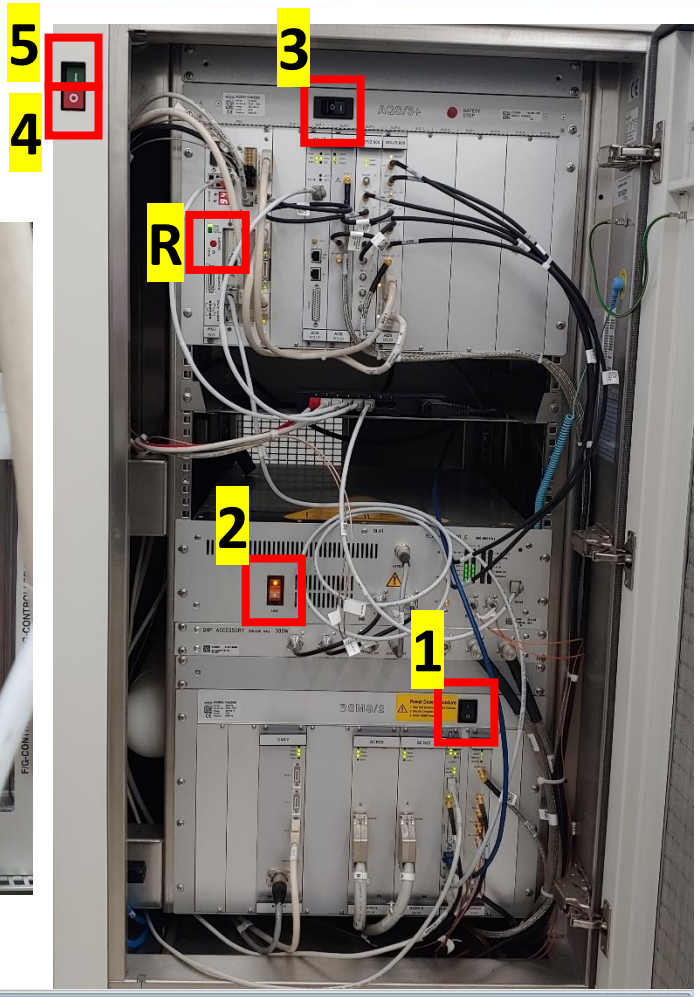
More details can be found in the TopSpin Installation Guide available from TopSpin Help menu (→ Manuals) or from the Bruker BioSpin webpage: http://www.bruker.com/service/information-communication/user-manuals/nmr/installation.html

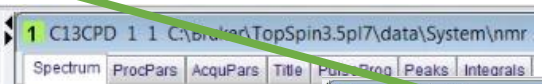
Reset console

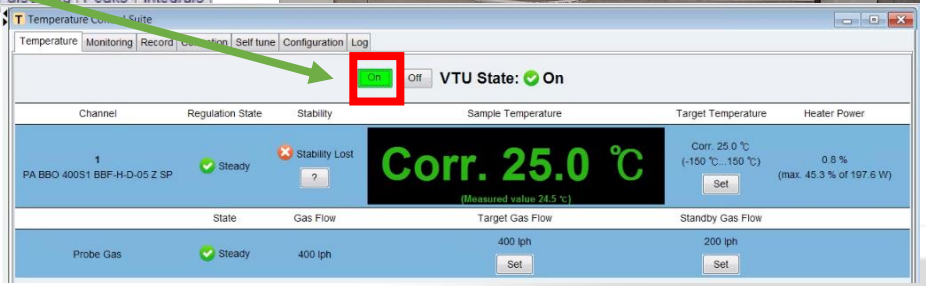
Reboot PC
(PC and Console ***are running***)

1. Reboot PC (Console is running.)
2. Turn off Console (**1** -> **2** -> **3**)
3. Turn on **3**
4. Check LED light (Stabilized 94)
   *(Don`t indicate 94 -> Press **R**)*
5. Turn on **2** -> **1** within 20 s
6. Open \Topspin3.5pl\data\system
   \nmr**C13CPD**
7. Type ***ii*** (Wait 20 s)
   Type ***atma***
8. Type ***vtudisp***
   (Turn on Temp. 25C)





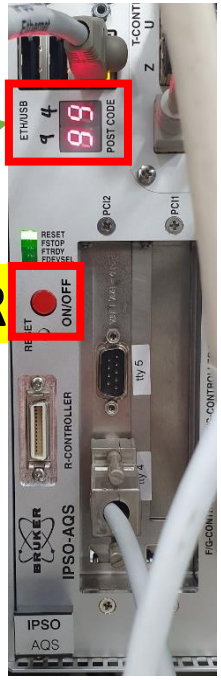


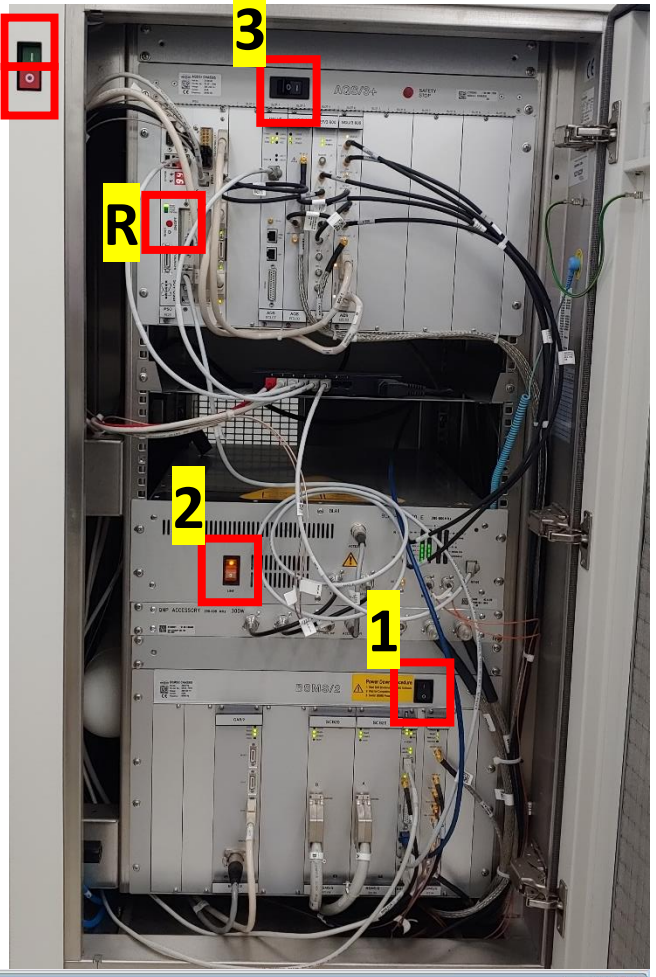


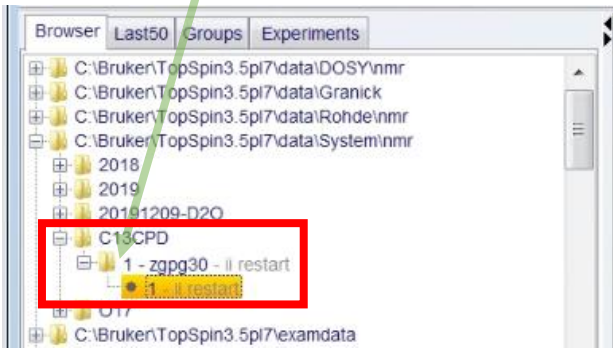
Reset console

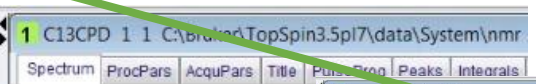
Reboot PC
(PC and Console are ***already turned-off***)

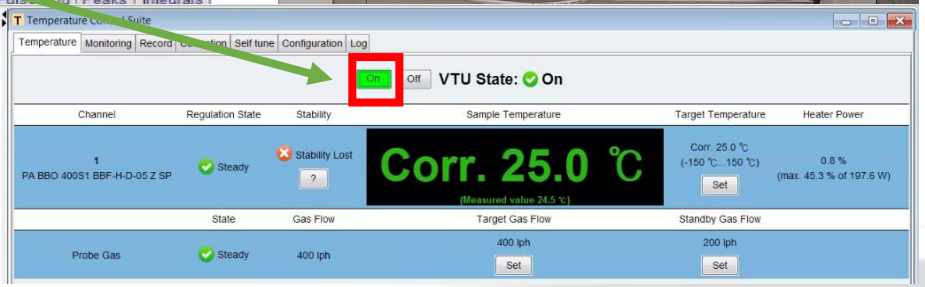
1. Press **5**
2. Turn on PC
3. Turn on **3**
4. Check LED light (Stabilized 94)
   *(Don`t indicate 94 -> Press **R**)*
5. Turn on **2** -> **1** within 20 s
6. Open \Topspin3.5pl\data\system
   \nmr**C13CPD**
7. Type ***ii*** (Wait 20 s)
   Type ***atma***
8. Type ***vtudisp***
   (Turn on Temp. 25C)











4. Command

Command list

Loading the sample

bsmsdisp - starts BSMS windows

ej - lift the air up (eject sample)

ij - lift the air off (insert the sample)

Locking, tunning and shimming

lockdisp - it generates the lock window

Lock solvent-name – it will lock on the named solvent after several seconds.

topshim – starts automatic shimming

gradshim – invokes gradient shimming windows in which the gradient shimming routine can be started.

atma - automatic tuning and matching

atmm – invokes manual tuning and matching window in which the automatic routine can be started

wobb- invokes tuning and matching window.

Command list

Create dataset

new(edc) - creates new data set

re - filename – read the file

wrpa - Copy data set

Acquisition

eda – it will display acquisition window

ased – display short list of acquisition window

sw – display spectral width

o1p – display transmitter center (center of spectrum)

ns – display number of scans

rga – automatic receiver gain adjustment

d1 – display relaxation delay

p1 – display pulse width (for simple 1D acquisition)

aq – FID acquisition time td – display number of points used to define FID

si – display number of points used during FT (can be different from td)

zg – zero and go Processing Commands:

Command list

Processing

edp – it will display processing window

em - exponential multiplication on the FID, uses the parameter LB. This improves signal to noise at the expense of resolution.

lb - this controls the degree of broadening added and affects your signal-to-noise. To see its effect, simply change its value and re-Fourier Transform with ef. ft - fouier transform

ef - combines em and ft

pk - phase correct, applies the last phase correction to the spectrum. Useful when you have phased a primlimary spectrum, (with only a few scans) and wish to apply the same phase correction to the final spectrum.

efp - combines em, ft, and pk.

abs- automatic baseline correction 9

apk – automatic phase correction

Advanced SOP

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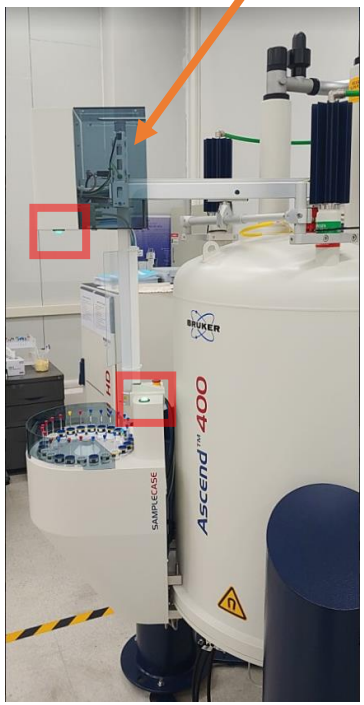
2022-08-10

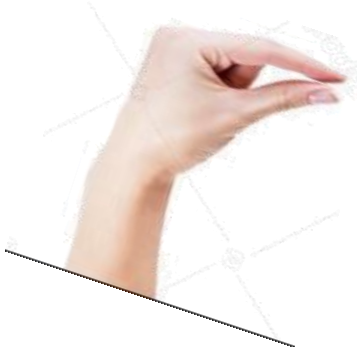
UNIST Central Research Facilities

Sun-Phil Han

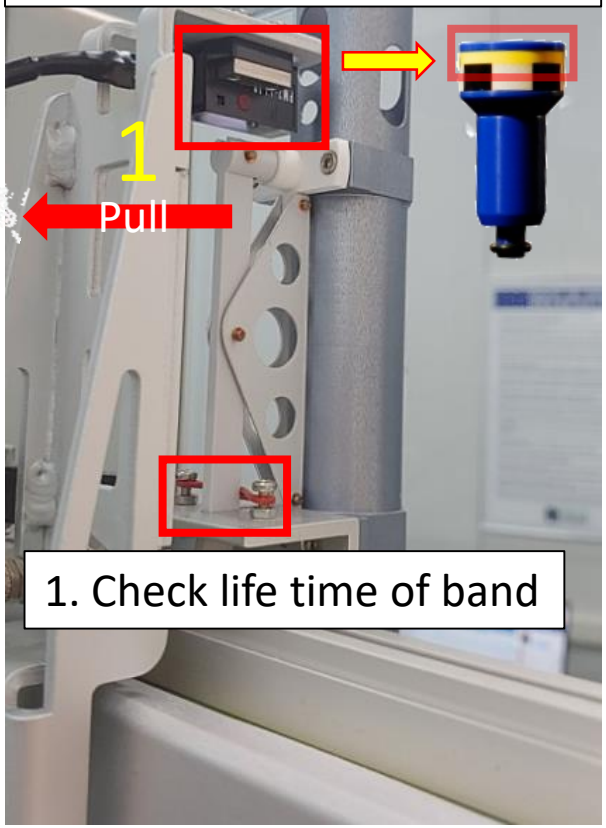
Problem of Auto-sampler

If you have a auto-sampler error,
(stuck sample here, and show red LED)

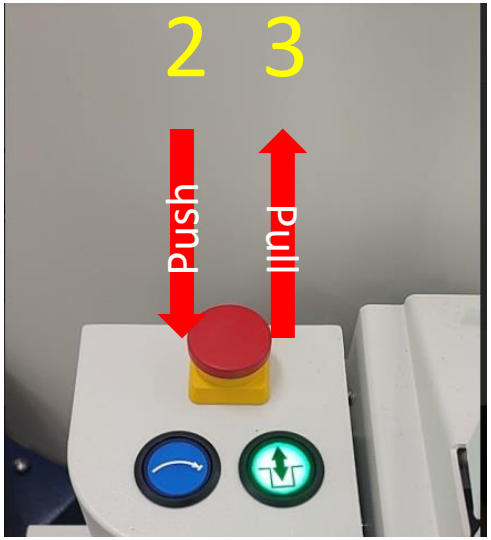




2. Check spinner(Yellow line)
or Sensor



1. Check life time of band



Remove Probe

When NMR has poor shim-map, it need to clean probe coil. (per 1 month)

1) Remove probe line connection

2H, 1H, BB

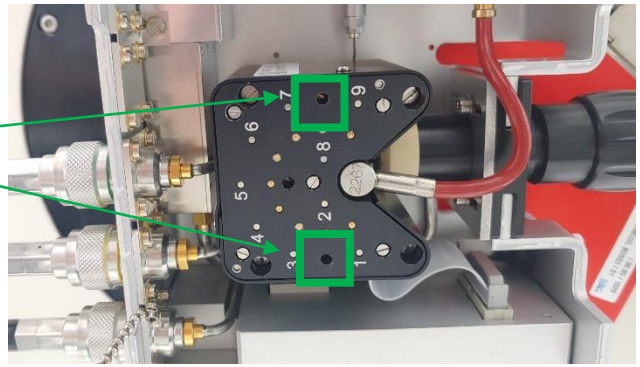
HEATER, THERMO, GRADIENT

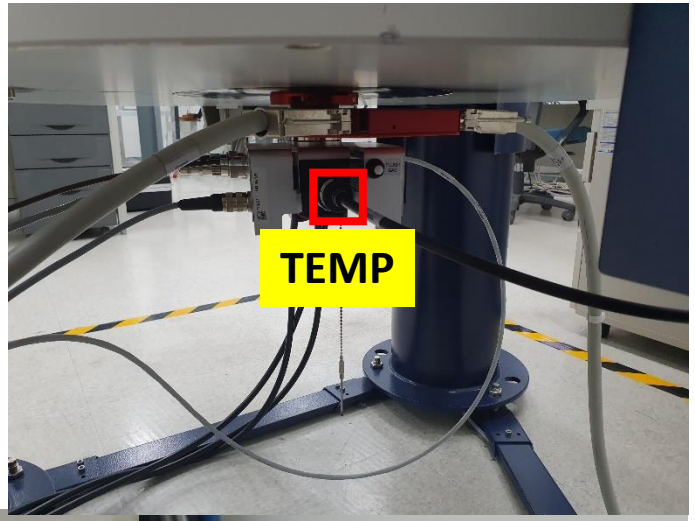
PICS

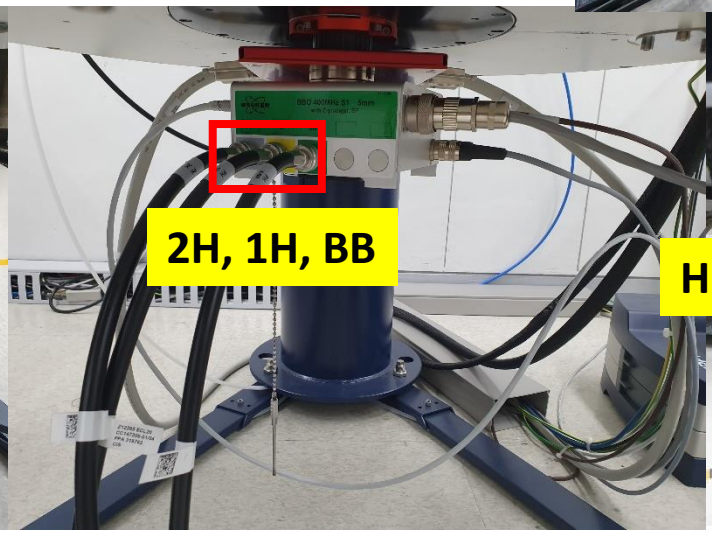
TEMP

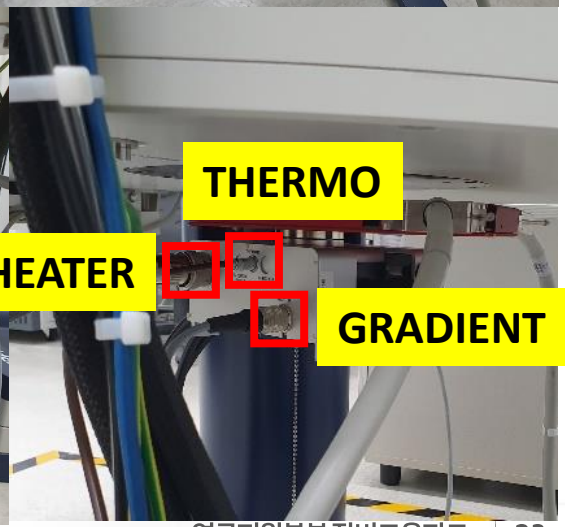
2) Remove probe

Unscrew two points





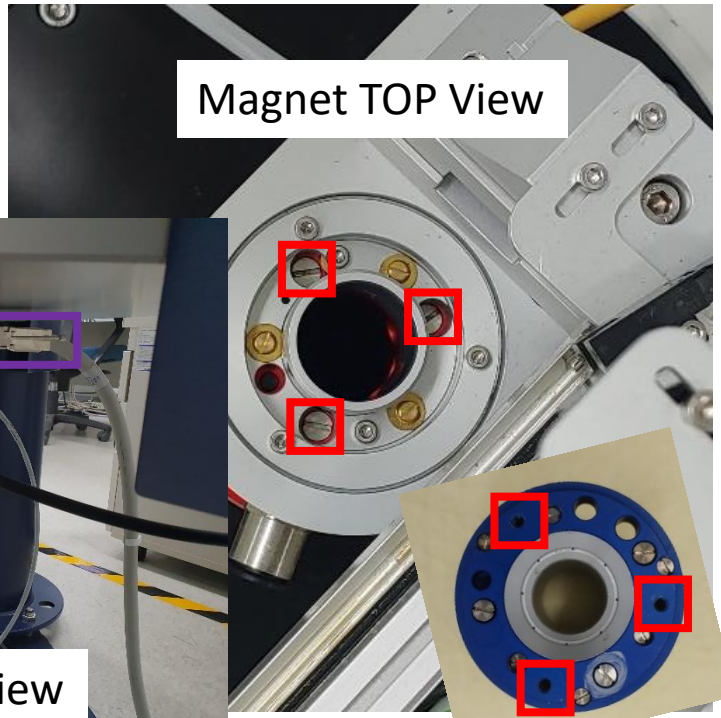


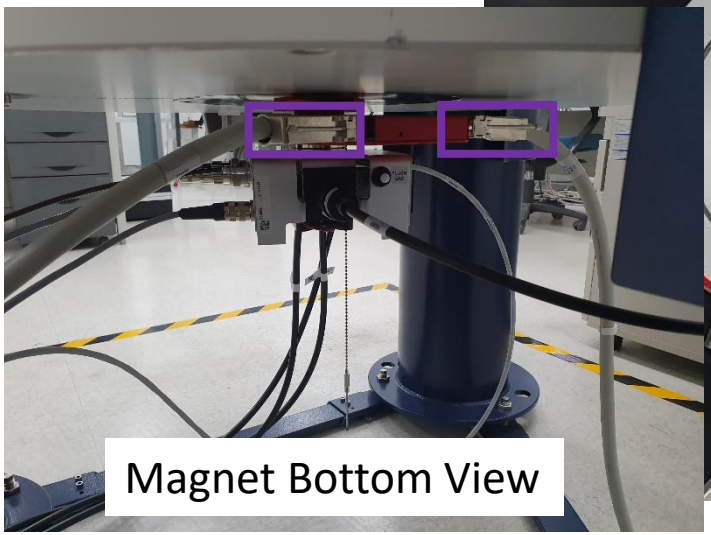


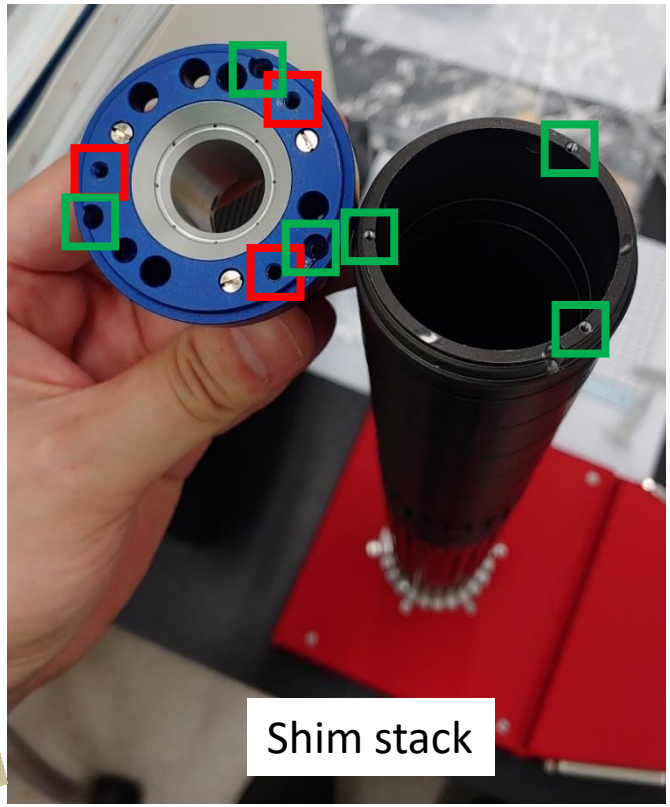
Remove Shim-stack

Must turn-off BSMS when remove shim-stack !! (for spinning error)

1) Turn-off BSMS unit
2) Remove A, B cable
3) Remove screw on NMR upper barrel
4) Remove shim-stack screw
5) Clean part







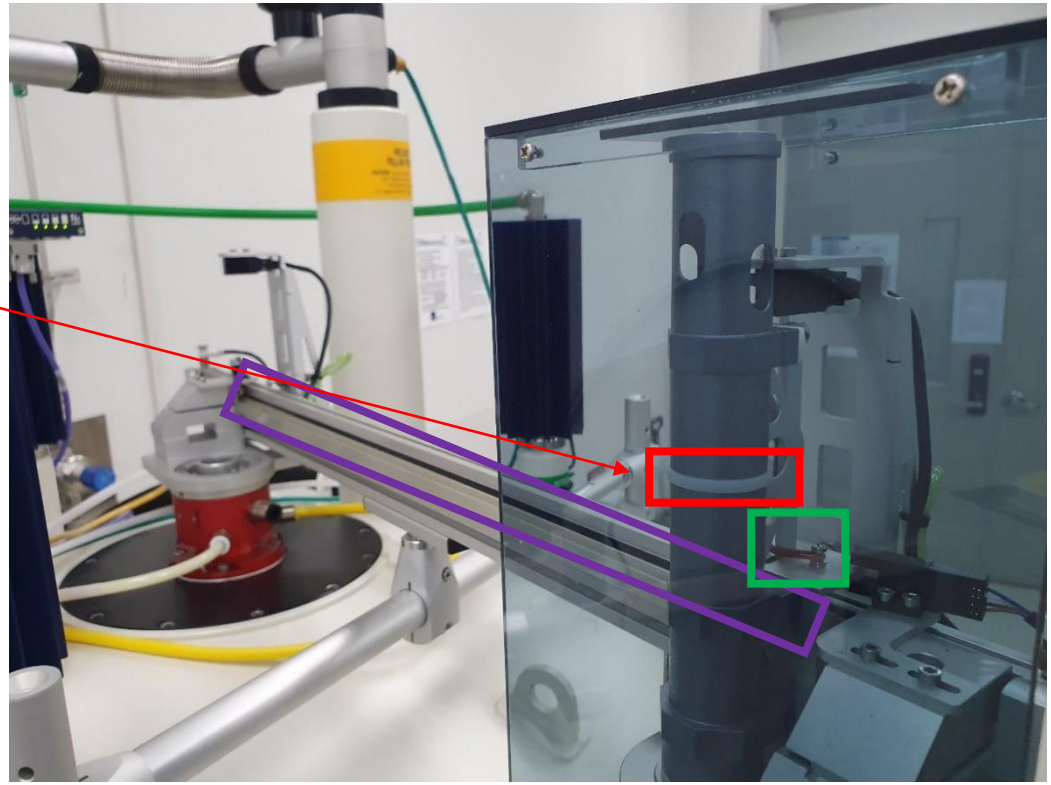
Maintain Auto-sampler

Must turn-off BSMS when remove shim-stack !! (for spinning error)

1) Coat grease on rail
2) Replace red rubber band (20,000 cycle / band)

Sample Grab 부분 유격으로 인해
Sample 인식이 불가한 경우 발생 시

-> 케이블 타이로 해당 부분 고정



Spinner 존재 유/무

Spinning rate
(Tachometer 인식부)

Tune & match error

주기적으로 시행 (장비 껐다가 켤 때 반드시 필수)

1) Open C13CPD

2) Type ii or ii restart

3) Type atma

1) 타핵종 실험 후 CH2(X, Low band)를 13C로 Tune 하지 않고 지속적으로
CH1 (1H, High band)만 진행하여 Tune 값이 한쪽으로 쏠리면
1H의 Tune 값이 무너지는 경우가 발생함

2) High concentration salt sample (or Low/High pH) 의 경우 Dieletric constant 값 변화에 따라
Tune이 제대로 진행되지 않는 경우가 발생함
(차라리 Icon-NMR 상에서 Tune을 수행하지 않고 실험 진행하는 것이 나음)

<Initialize Tune & Match>

→ atma activatedrivecalibration
→ atma selectnuc
→ atma resetconfiguration
→ atma activatedrivecalibration
→ atma seletnuc

How to Handle Lock, ATM, Shim (X)

1. ATM X (Tune & Match X)

1) Contain high concentration salt
   (add NaOH, KOH, NaCl, KCl, etc..)
2) High pH or Low pH
   (Acidic or Basic)

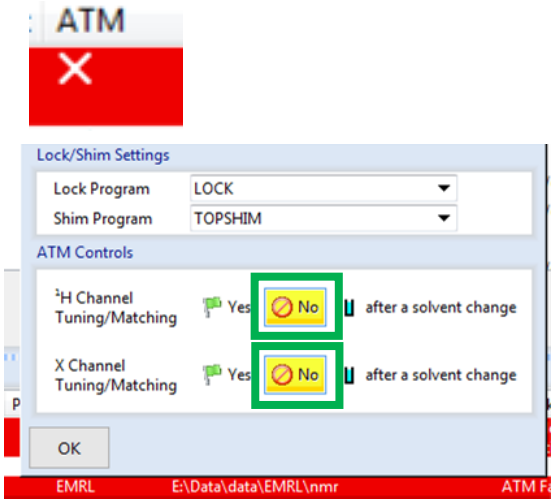
2. Only Shim X

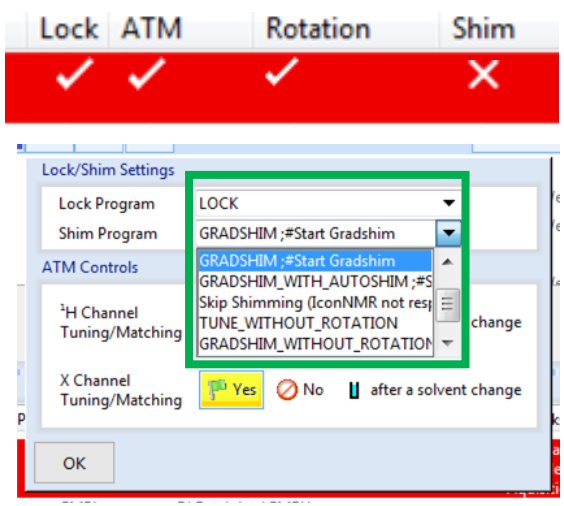
Topshim
(Optimized Single solvent)
-> Use Gradient shim

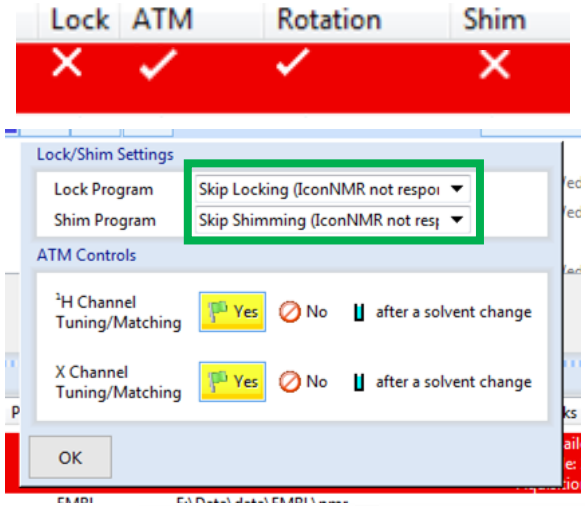
1) NMR solvent mixture
ex) D2O+DMSO-D6,
    DMSO-D6+CDCl3

3. Lock & Shim X

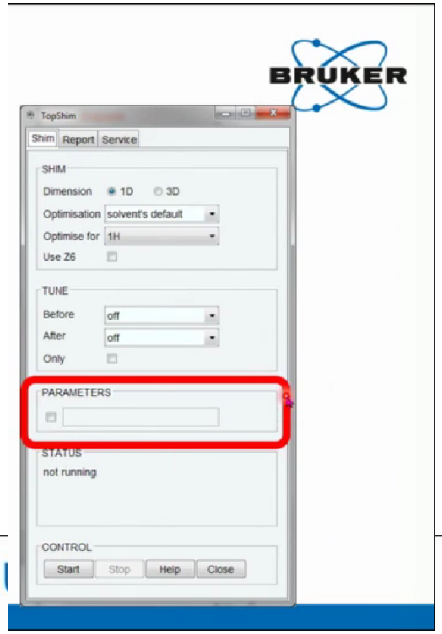
1) NMR solvent mixture
ex) D2O+DMSO-D6,
    DMSO-D6+CDCl3
2) Non-Deuterated solvent
   (H2O, DMSO-H6, CHCl3)
-> Recommend to use
   Coaxial tube (Evans method)







Topshim gui



- **3d** activates 3-dimensional X, Y, Z gradient shimming
- **1dfast** perform fast 1D shimming; only 1 iteration loop is performed
- **3dfast** perform fast 3D shimming; only 1 iteration loop is performed
- **tune** also shim on the lock before and/or after gradient shimming
  (tuneb shims X,Y,Z,XZ,YZ before running gradient shimming)
  topshim tuneaz (shims Z after running gradient shimming)
- **shigemi** Used to eliminate unreliable data at axial Shigemi tube walls when 1D shimming
- **zrange=** sets the range in cm in the Z direction used for shimming
  topshim zrange=-0.8,0.8 [below center (mm)],[above center (mm)] (short sample)
- **plot** Saves data after completion in <TopSpin_home>/data/topshimData

... read about more in the Topshim manual located in the Help(?) pulldown!

BRUKER

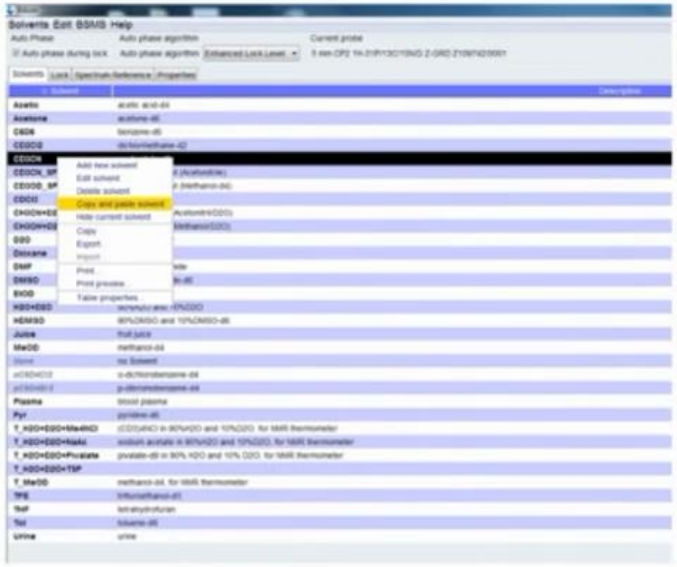
- **lockoff** Enables shimming with system unlocked
- **1h or 2h** Explicitly sets shimming nucleus
- **o1p=** Explicitly sets excitation frequency in PPM
  topshim 1H lockoff o1p=2.49 (DMSO-h6)
- **selwid=** Activates selective excitation of a bandwidth expressed in ppm; useful when shimming on a solvent with multiple strong signals
  topshim o1p=1.93 selwid=0.5 (CD3CN+D2O)
- **ordmax=** Sets the maximum shim function order used (default = 5 on axis, 7 off axis)
  ordmax=3 limits shimming to Z-Z3
  topshim ordmax=8 (SmartProbe, iProbe, newer CryoProbes)
  topshim 3d ordmax=8,7 ([on-axis],[off-axis])
- **durmax=** maximum duration per 1D field map acquisition (expressed in seconds)
  default = 7 (try 15, 30 or even 120), good for low s/n situations
- **convcomp** Used to activate convection compensation; useful when using low viscosity solvents susceptible to convection

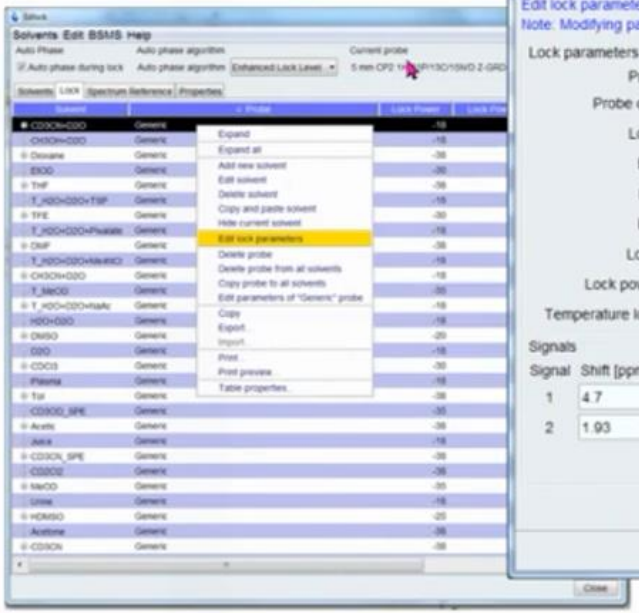
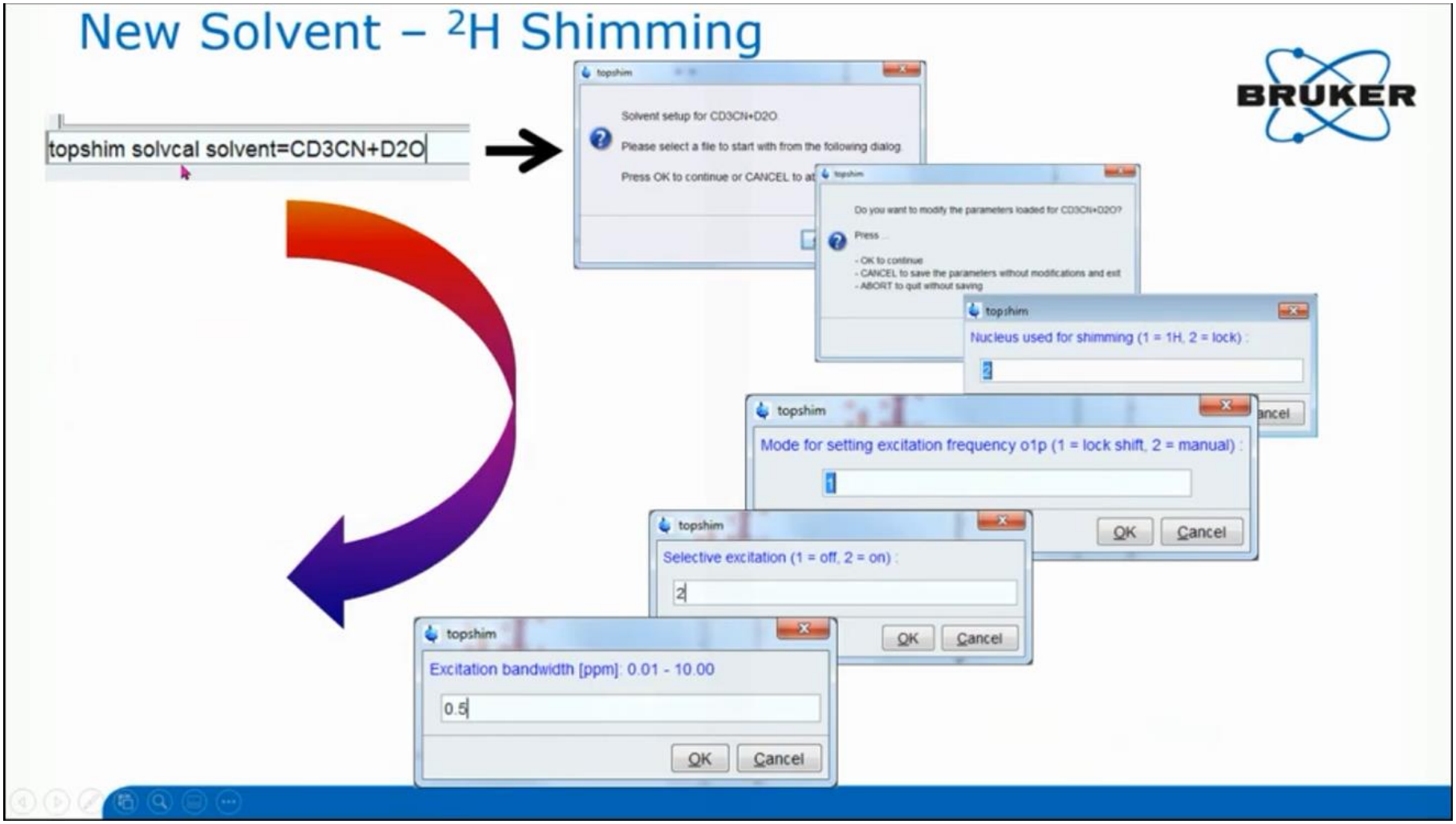
Shim for Solvent mixture

New Solvent – ^{2}H Shimming

Mixed solvent example- (mixture of CD_3CN and D_2O)

edsolv ***edlock***





Shim for Solvent mixture