

Nuclear Magnetic Resonance spectroscopy

Normal Operation Training Course



Pre-Preatment

Solution NMR

Solute



Solvent



Solution



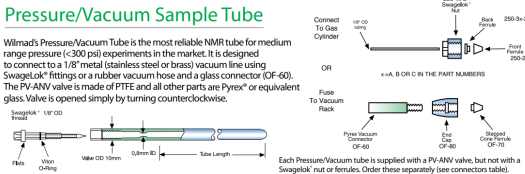
Channel with gel phase



Gas phase



Pressure tube



Wilmad's Pressure/Vacuum Tube is the most reliable NMR tube for medium range pressure (<300 psi) experiments in the market. It is designed to connect to a 1/8" metal (stainless steel or brass) vacuum line using Swagelok® fittings or a rubber vacuum hose and a glass connector (OF-60). The PV-ANV valve is made of PTFE and all other parts are Pyrex® or equivalent glass. Valve is opened simply by turning counterclockwise.

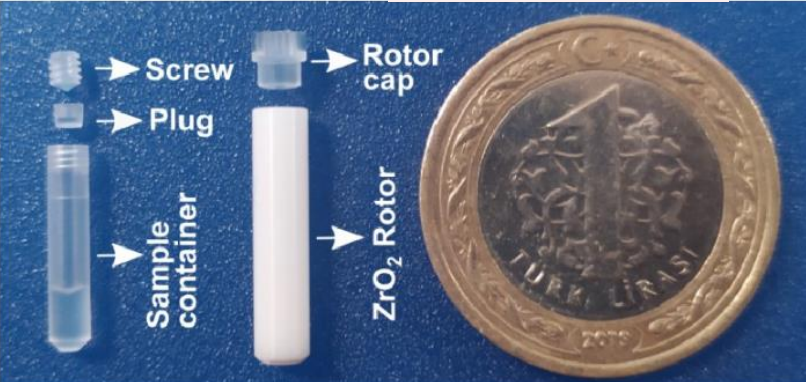
Each Pressure/Vacuum tube is supplied with a PV-ANV valve, but not with a Swagelok® nut or female. Order these separately (see connection table).

HR-MAS NMR

Gel & Paste



HR-MAS insert kit



Screw
Plug
Sample container
Rotor cap
ZrO₂ Rotor

Solid-state NMR

Solid
(Powder, film, fiber)



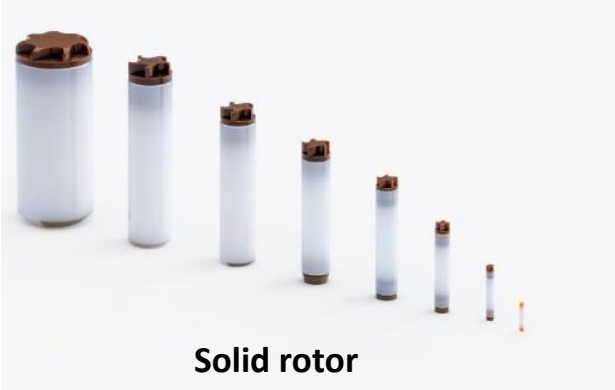
Fine powder



Flexible film



Solid rotor



Max. MAS Speed [kHz]	Rotor Diameter [mm]	Rotor Volume [μl] (Rounded)
7	7	360
15	4	106
24	3.2	47
42	1.9	13
67	1.3	3
111	0.7	0.6
160	0.4	0.12

1) Normal case

Sample(~ 20mg) + Deuterated solvent (500uL)
Deuterated solvent: ex) D₂O, CDCl₃, DMSO-D₆

2) 10% D solvent (Quantitative NMR with Calibration curve)

Sample(~ 20mg) + Non-D solvent (450uL) + Deuterated solvent (50uL)
Non-D solvent: H₂O, CHCl₃, DMSO-H₆

3) After reaction in water or solvent base without pretreatment

Sample(~ 20mg) + Non-D solvent(500uL)
Non-D solvent: H₂O, CHCl₃, DMSO-H₆

4) Coaxial tube

Inner tube: NMR solvent or Reference (Chemical shift, Concentration)
Outer tube: Sample (Non-D solvent, unknown concentration)

5) Gelation with coaxial tube / without coaxial tube

With coaxial tube

Inner tube: Deuterated solvent

Outer tube: Sample

6) Rolling flim

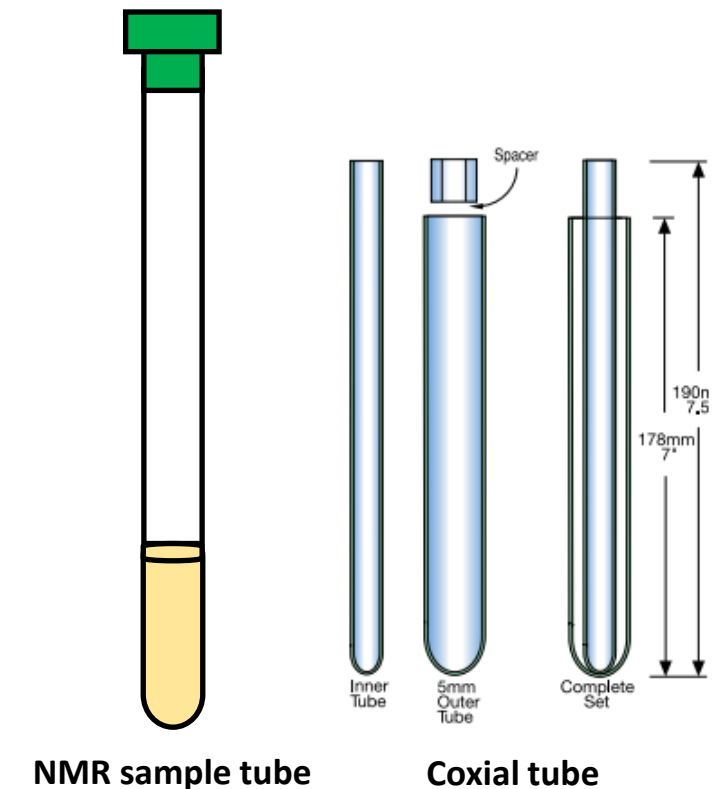




Figure 1: MAS rotors with different diameters for different spinning speeds

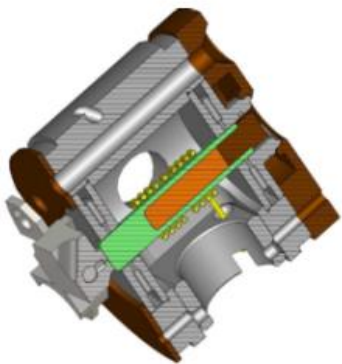
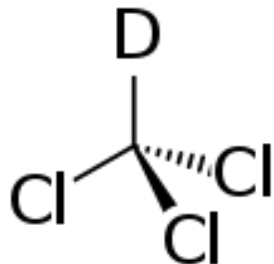


Figure 2: Schematic of a Bruker MAS stator

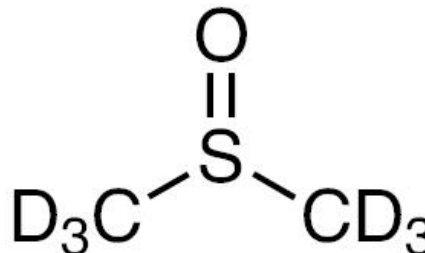
Max. MAS Speed [kHz]	Rotor Diameter [mm]	Rotor Volume [μl] (Rounded)
7	7	360
15	4	106
24	3.2	47
42	1.9	13
67	1.3	3
111	0.7	0.6
160	0.4	0.12

Dielectric constant : **Polar** > 20 > **Non-polar**



CDCl₃

- Dielectric constant: 4.8
- Advantage:
 - Cheap,
 - Easily Removed
- Disadvantage:
 - Light sensitive(Contain HCl)
 - Toxic, carcinogenic
 - Weak lock signal
 - Easily evaporated

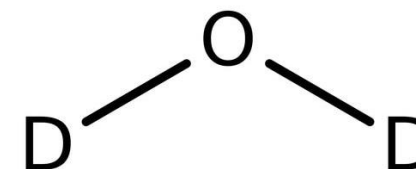


DMSO-D₆

- Dielectric constant: 46.7
- Advantage:
 - Strong lock signal
- Disadvantage:
 - High price
 - Highly viscosity
 - Easily contain water
 - M.P.: 20 ~ 22 °C (Freeze RT)
 - Hard removed

NMR Solvent Storage

Avoid light & moisture: All NMR solvent
Store refrigerator (4°C): CDCl₃, THF-D₈

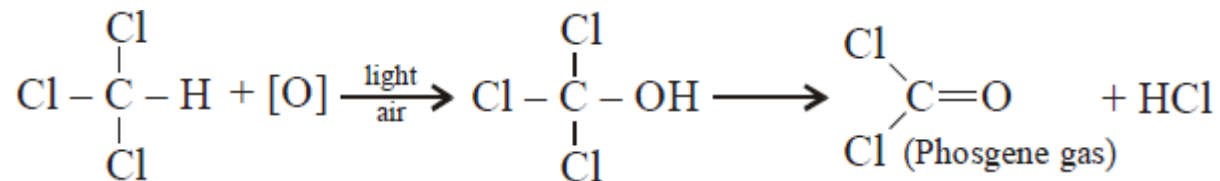


D₂O

- Dielectric constant: 78.5
- Advantage:
 - Cheap
- Disadvantage:
 - Exchange OH and NH
 - Easily contain water
 - Chemical shift (Temp. dependent)
 - Hard removed

CDCl₃ + Light -> HCl

- Use silver foil
(Stabilizer, radical scavenger)
- Use molecular sieve
(Eliminate H₂O)



Change of chemical shift

- Temperature
- pH
- Concentration

HOD peaks

- Related residual H₂O
- Slow exchange H₂O and HOD
→ two peaks
(singlet H₂O, 1:1:1 triplet HOD)
- Hydrogen bond
→ Broad peak of HOD

How to remove residual water

→ Use Molecular Sieve 3~5 g in 100g (CDCl₃)

- 3 Å: Adsorption of polar liquid (NH₃, H₂O)
- 4 Å: Adsorption of Non-polar liquid
(H₂O, CO₂, SO₂, H₂S, C₂H₄, C₂H₆, C₃H₆, EtOH)
- 5 Å: Adsorption of Linear C_xH_x to n-C₄H₁₀
Alcohols to C₄H₉OH
C₄H₉SH

Thin wall (Precision-Glass)

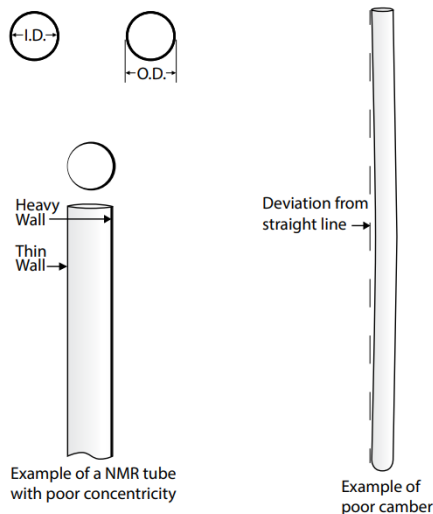
- **Temp 120 °C**
- Critical shimming quality (low Fe₂O₃)
- **Good volume reproducibility**
- High sample volume in RF coil
- 535-PP-7 (600 MHz grade, \$40)
- 527-PP-7 (400 MHz grade, \$25)

Economy (Middle MW)

- **MW<1500**, RT
- WG-1241-7 (600 MHz grade, \$10)
- WG-1228-7 (400 MHz grade, \$9)

High-Throughput (low MW)

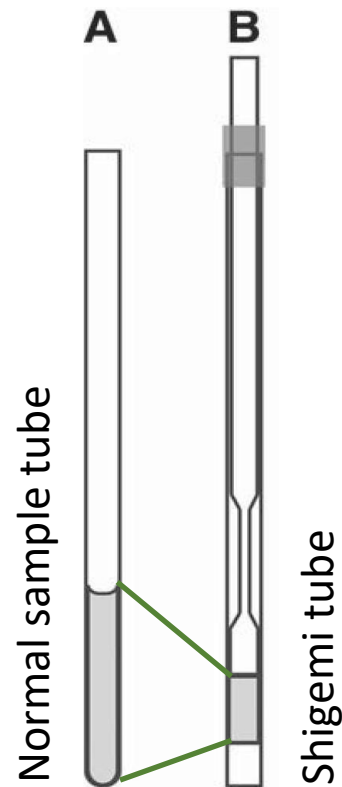
- up to 600 MHz, **MW<250**, RT
- WG-1000 (\$2)



5mm Thin-Wall NMR Tube Comparison Table

	High-Throughput	Economy	Precision (Glass)	Precision (Quartz)	Precision (Suprasil)
Material	Type 1 Class B Borosilicate Glass	Type 1 Class B Borosilicate Glass	Type 1 Class A Borosilicate Glass	Clear Fused Quartz	Synthetic Quartz
Impact On Shimming Quality By Paramagnetic Impurities	Medium (1200ppm Fe ₂ O ₃)	Medium (1200ppm Fe ₂ O ₃)	Small (400ppm Fe ₂ O ₃)	None (0.5ppm Fe ₂ O ₃)	None (<0.005ppm Fe ₂ O ₃)
Maximum Working Temperature	Ambient	Ambient	230° C	1300° C	1300° C
Sample Volume Reproducibility	10%	10%	0.5%	0.5%	0.5%
Recommended Applications	Small molecule experiments up to 600 MHz (MW<250)	1D NMR experiments with small organic molecules (MW<1500)	Experiments requiring critical shimming quality (high-field, multi-dimension, multi-nuclei)	¹¹ B NMR, rapid cooling/heating experiments, photochemistry studies	Photochemistry studies with deep UV light source

Special purpose sample tube



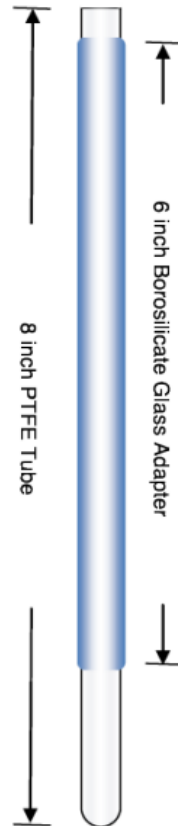
Normal sample tube

Shigemi tube

Shigemi tube

\$ 300 - 400

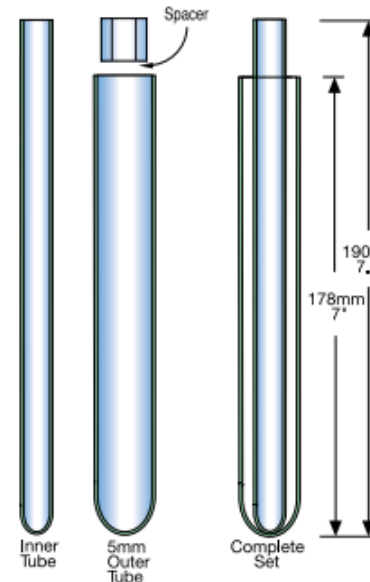
- Increase 16% SNR
- Low con. or low volume



PTFE tube

\$ 60

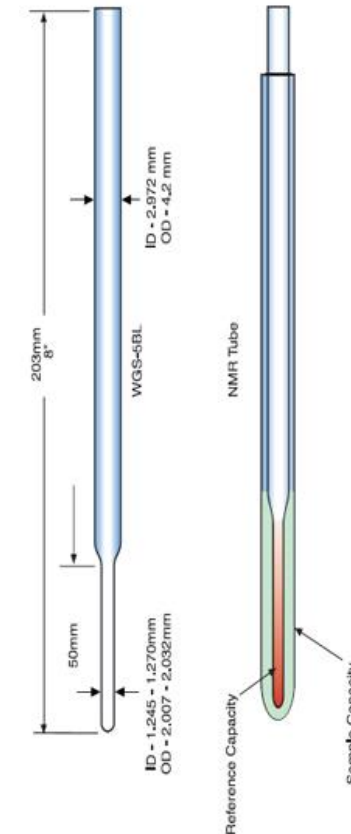
- HF or ^{29}Si NMR



Coaxial tube

\$ 40 - 50 (+ \$ 50)

- Non-deuterated sample
- Quantitative NMR
- Referencing



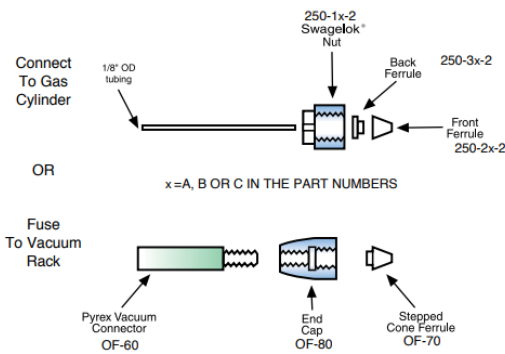
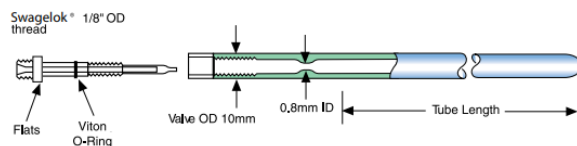
Stem Coaxial tube

\$ 40 - 50 (+ \$ 50)

- Non-deuterated sample
- Quantitative NMR
- Referencing

Pressure/Vacuum Sample Tube

Wilmad's Pressure/Vacuum Tube is the most reliable NMR tube for medium range pressure (<300 psi) experiments in the market. It is designed to connect to a 1/8" metal (stainless steel or brass) vacuum line using SwageLok® fittings or a rubber vacuum hose and a glass connector (OF-60). The PV-ANV valve is made of PTFE and all other parts are Pyrex® or equivalent glass. Valve is opened simply by turning counterclockwise.



Each Pressure/Vacuum tube is supplied with a PV-ANV valve, but not with a SwageLok® nut or ferrules. Order these separately (see connectors table).



Ethyl Vinyl Acetate Cap

- Cheap
- Avoid CDCl_3 , Acetone- D_6



Ethyl Vinyl Acetate Cap

+ Teflon tape

- Cheap
- Use CDCl_3 , Acetone- D_6



PTFE Cap

\$ 16 (or \$ 5 economy ver.)

- Use CDCl_3 , Acetone- D_6 , HF

Low Pressure/Vacuum Tubes

Wilmad's Low Pressure/Vacuum (LPV) tube is ideal for anaerobic and gas-tight NMR experiments, and offers a convenient flame-free sealing solution for air sensitive or volatile liquid samples.

- Robust sealing system allows pressure build-up inside the sample
- Greaseless PTFE piston provides a 100% contamination-free seal
- Redesigned with a 4X larger sealing surface; eliminates leaks and greatly increases lifetime when compared to traditional J. Young tubes
- Axial symmetric design guarantees application in spinning experiments
- Due to the nature of glass, Extreme Caution should be exercised when using at elevated or reduced pressures since a tiny scratch on the glass surface would significantly lower the tensile strength. Adequate safety shielding should always be used when working in these conditions.



Consumables (Recommendation)

NMR sample tube (rating ≤ 600 MHz)

WG-1000-7 (5mm High Throughput NMR tube)

WG-1003-7 (**Large writing** Area 5mm High Throughput NMR tube)



Labeling tip

Short term: **Red**

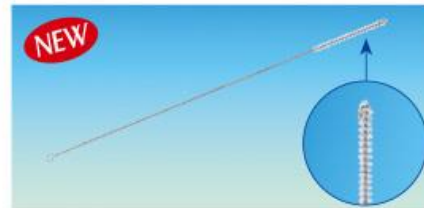
Long term: **Black**



NMR pipet – Long tip pipette (13.75 inch)

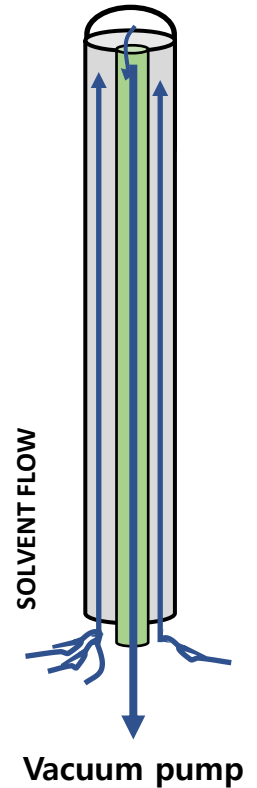


NMR tube Brush



Tube washer

(Multi-tube Jet Solvent Wash/Dryer)



DURAN® NMR Tube Brush, Suitable for ϕ 5mm NMR Tubes, L250mm **NEW**
Made of Stainless-steel Wire & Nylon Bristles, <Germany-made>, ϕ 5mm NMR, 튜브용 브러쉬

* Curved End of Bristle Trim for Cleaning the Inner Tube Bottom
* Ideal for Cleaning NMR Tubes from Stubborn Contamination

DU.2907400 Brush, for ϕ 5mm NMR Tubes, SS Wire w/Nylon Bristles, L250mm

<L1>	
Ea.	Price, W

- Sample volume = 500 ul (400 ~ 700 ul)
- Sample concentration
 1. ^1H : 0.05 mM ~ 10 mM (Too high concentration cause Line-broadening effect)
 2. ^{13}C , 2D NMR(COSY, NOESY, TOCSY, etc.): 10 mM ~ 100 mM
 3. ^{15}N , ^{17}O , Hetero 2D NMR (HSQC, HMBC, HMQC, etc.): 100 mM < Highest concentration
 - ex) Sample A (MW=200), 0.05 mg / 500 ul = 0.5 mM = 100 ppm
 - Sample A (MW=200), 1 mg / 500 ul = 10 mM = 2000 ppm
 - Sample B (MW=500), 25 mg / 500 ul = 100 mM

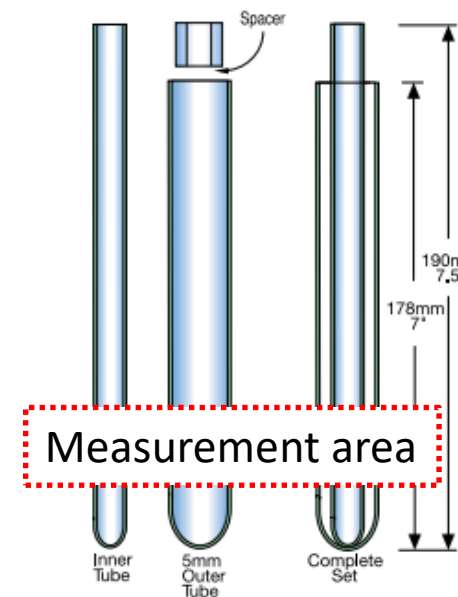
<Internal reference>

1. Sample + NMR solvent (CDCl_3 , D_2O , $\text{DMSO}-d_6$, etc.) + reference compound (TMS, DSS)

<External reference>

- 2-1. Sample + NMR solvent (CDCl_3 , D_2O , $\text{DMSO}-d_6$, etc.)
- 2-2. Reference sample (P31: TPP = -17.57 ppm, F19: TFT: -63.72 ppm)
3. Coaxial sample tube (Evans method)
 - Inner tube: NMR solvent or Reference
 - Outer tube: Sample

- None deuterated solvent condition -> No Lock / No auto shim -> Poor or not easy measurement
- NMR solvent and Referencing (+Quantitative NMR)



- Degassing

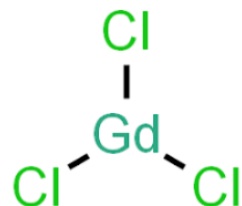
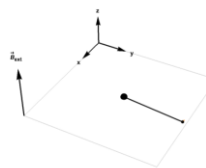
Oxygen (Paramagnetic) -> Short T1&T2 relaxation-> **Quench** for NMR signal -> Purge N2 or Ar gas

- Doping agent (Use Paramagnetic agent)

- Reduce T1 & T2 -> Short experiment time

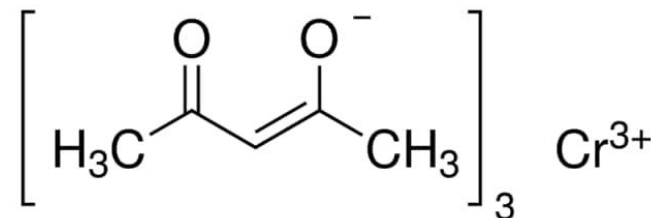
- Line broadening effect (poor shimming)

- Add paramagnetic material concentration: 1 mM (1H), 25 mM – 50 mM (13C)



GdCl₃

Gadolinium(III) chloride



Cr(acac)₃

Chromium(III) acetylacetonate

CuSO₄, MnCl₂, NiCl₂

Dielectric constant : **Polar** > 20 > **Non-polar**

- Wash sample tube to acetone or ethanol.

Cheap sample tube dry-situation tilted state in oven make possible bending
(Sample tube bending test → rolls the ground If bending sample tube spins, quartz glass in probe has some damage.)



Probe Bottom

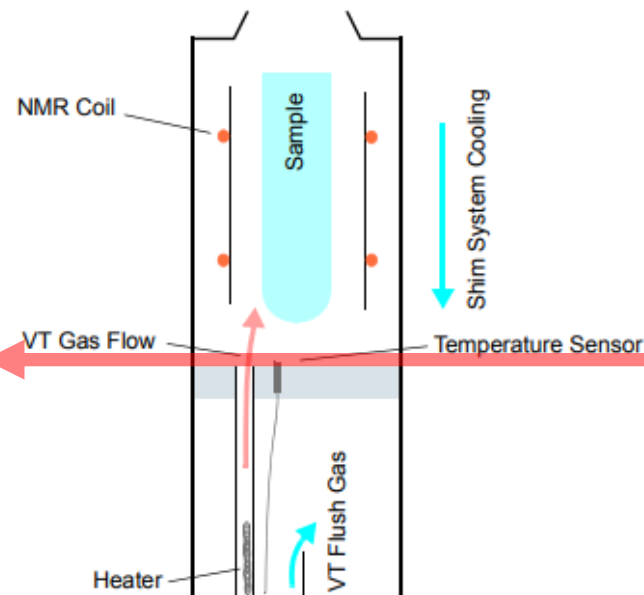
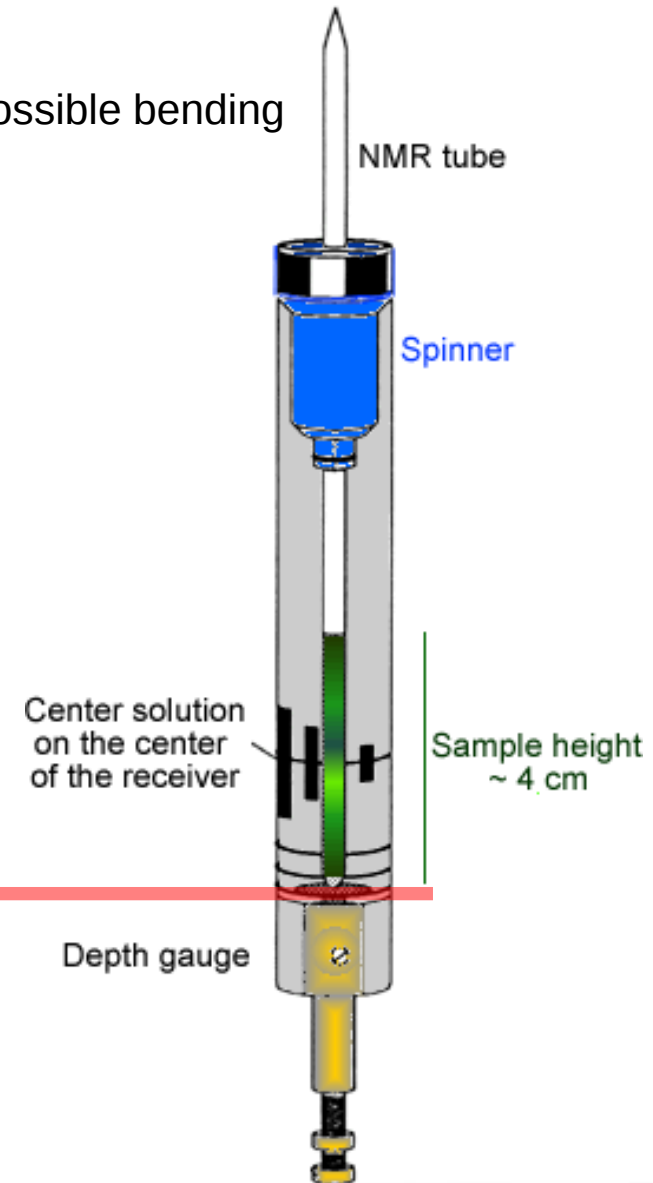
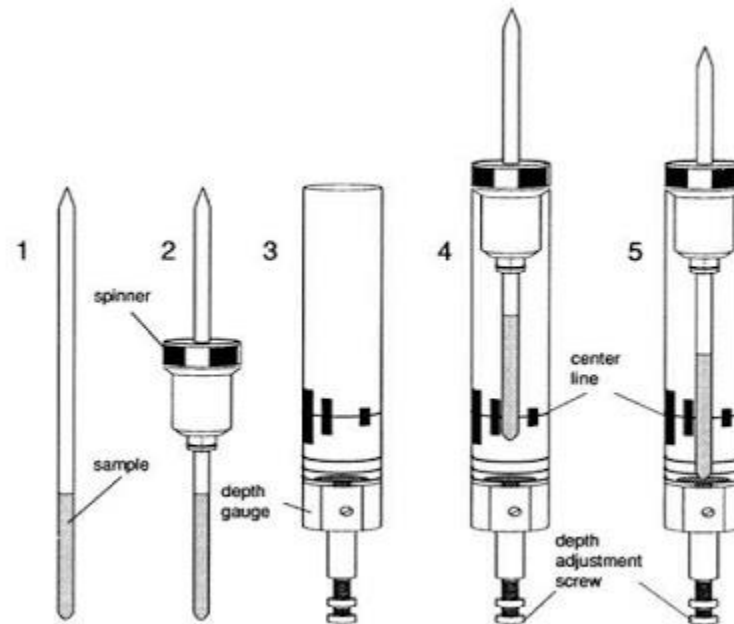


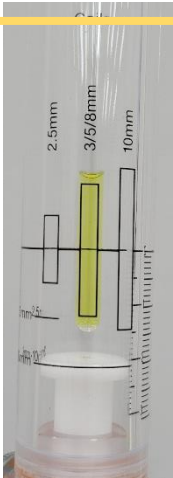
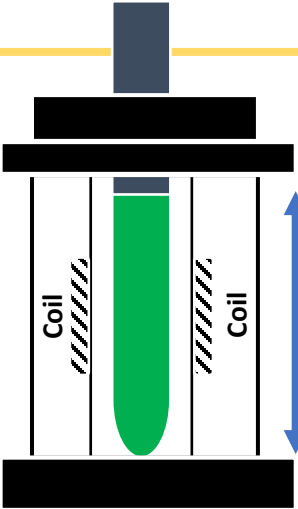
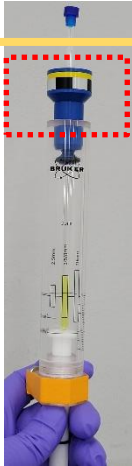
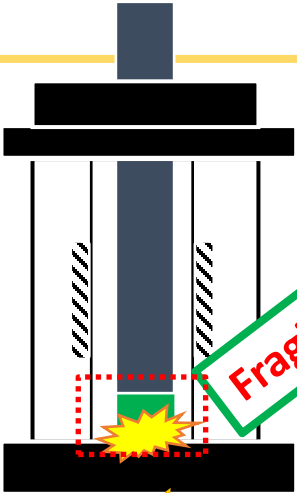
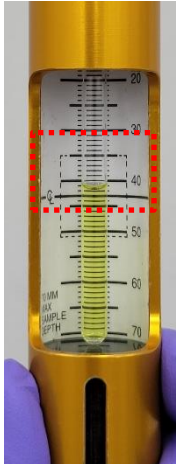
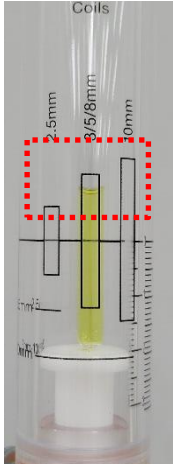
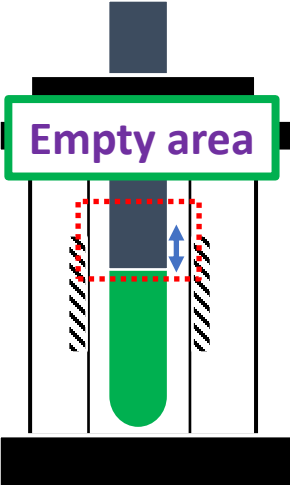
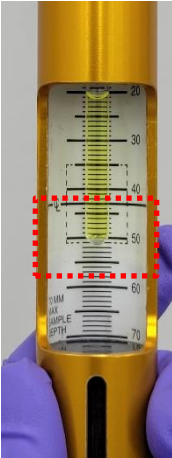
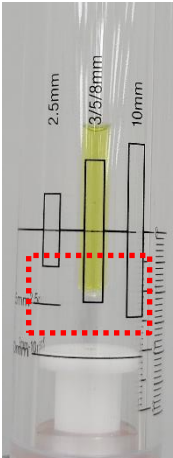
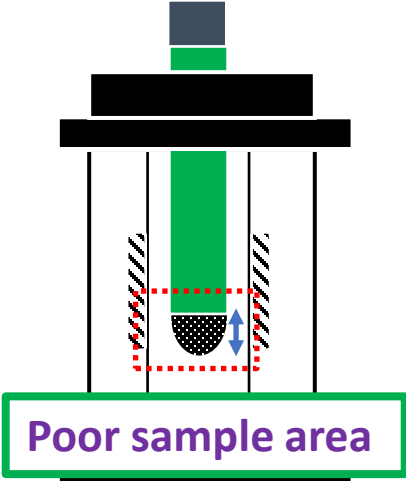
Figure 3.1 Sketch of sample temperature control in a NMR probe



1. Perfect solution sample filters syringe filter.
(If your sample has insolubility, It makes low resolution and sensitivity about peak)
2. 5 mm sample tube inserts sample 0.5 ml (500 μ l, 4cm)
3. Spinner placed on depth gauge insert sample tube
(※ Don't touch O-ring)
4. Check location depth gauge center (dash line square) with sample tube
5. Remove depth gauge.



Sample volume and height

		 40 mm 400 - 500 uL			 40 mm 400 - 500 uL
BEST			Worst		
Bad shim			Bad shim		
		 Empty area			 Poor sample area

Operation With Icon-NMR

UNIST
ULSAN NATIONAL INSTITUTE OF
SCIENCE AND TECHNOLOGY



Type **icon** on command line
Click Acquire tab > More > IconNMR Automation



Topspin 3.5



Topspin 3.5

Topspin 4.4.1



19

Icon-NMR (Routine)

Check holder number (= Auto-sampler number) **5**

6 Double-click available holder

7 Write sample name

8 Choice Solvent

9 Choice Experiment

3-4 Change user (Login/out)

10 Submit

Topspin 4.4.1

Topspin 3.5

Holder	Type	Status	Name	No.	Solvent	Experiment
1	1	Available	20250801-TEST-DMSO-D6_H1	1	DMSO	dimethylsulfoxide
2	1	Available				
3	1	Available				
4	2	Failed	20250731-PNU-THF-E-HOESY-7Li-1H_D8=1.51	15	D2O	deuteriumoxide
5	1	Running	20250731-PNU-THF-E-HOESY-7Li-1H_D8=1.7_with_Li7_T1	18	D2O	deuteriumoxide
6	1	Running	20250731-PNU-DOL-E-HOESY-7Li-1H	20	D2O	deuteriumoxide
7	1	Queued	20250731-PNU-MCP-E-HOESY-7Li-1H	1	D2O	deuteriumoxide
8	1	Available				

No.	Solvent	Experiment	Load	ATM	Rotation	Lock	Shim	Acq	Proc	User	Disk
215	2025-08-01 11:32:43	5	20250731-PNU-DOL-E-HOESY-7Li-1H	20	D2O	HOESY_7Li-1H	✓	✓	✓	✓	UCRF
214	2025-08-01 10:01:40	4	20250731-PNU-THF-E-HOESY-7Li-1H_D8=1.51	15	D2O	HOESY_7Li-1H	✓	✓	✓	✓	UCRF
213	2025-08-01 09:40:54	3	20250731-PNU-THF-S_Li7	19	D2O	Li7_UNIST	✓	✓	✓	✓	UCRF
212	2025-07-31 23:29:53	6	20250731-PNU-MCP-E_Li7_T1	21	D2O	Li7_T1	✓	✓	✓	✓	UCRF

Icon-NMR (Folder name)

1 **Folder name** 2 **Experiment number** 0 **user**

Holder	Type	Experiment number	Experiment name	Experiment details	Time	User	Start Time
5	Finished	1	42Ah_Li_sol_5th_250804_H	H2O Water, non-deutera N H1_Li_Salt Li_electrolyte	00:06:50	YK	14:30 Mon Aug 04 2025
	Finished	2	42Ah_Li_sol_5th_250804_C	H2O Water, non-deutera N C13_Li_Salt Li_electrolyte	00:15:33	YK	14:36 Mon Aug 04 2025
	Finished	3	42Ah_Li_sol_5th_250804_F	H2O Water, non-deutera N F19_Li_Salt Li_electrolyte	00:08:39	YK	14:59 Mon Aug 04 2025
6	Available	1	2D NMR_ICON COSY	CDC13 chloroform-d N PROTON		UCRF	Set Start Time
	Available	2	2D NMR_ICON COSY	CDC13 chloroform-d C COSYGPSW Gradient selected COSY		UCRF	Set Start Time
	F2	1	2D NMR_ICON COSY			UCRF	
7	Available	1	2D NMR_Full COSY	CDC13 chloroform-d N COSY_Full_range -4 to 16 ppm		UCRF	Set Start Time
8	Available						
9	Finished		20250804-HANTOK-TMAC-S6-TK-741-50802_H1	1 10%_D2O_HANTOK N PROTON_HANTOK 10 degree	00:30:46	UCRF	15:47 Mon Aug 04 2025
10	Finished						

Submit Cancel Edit Add 1 Copy 1 X Delete

0 **user** 1 **Folder name** 2 **Experiment number**

Path: D:\nmrdata\UCRF\20250801-CNERG-P1_H1

Search: 20250801-CNERG-P1_H1

Name	Date modified	Type
1	8/1/2025 4:04 PM	File folder
2	8/1/2025 4:09 PM	File folder
3	8/1/2025 4:29 PM	File folder

2 Automatically opened 1H NMR experiment
(Measure 1D 1H ->)

Available 2D NMR_ICON COSY 1 CDCI3 chloroform-d N PROTON

Available 2D NMR_ICON COSY Same No. = PROTON C COSYGPSW Gradient selected COSY

3 F2 2D NMR_ICON COSY 1

1 Choice 2D NMR

2D NMR_ICON COSY 1 (1H NMR data) -> Automatically control SW(Spectral width, Spectrum width)

Available 2D NMR_ICON COSY 1 CDCI3 chloroform-d N PROTON

Available 2D NMR_ICON COSY 2 CDCI3 chloroform-d C COSYGPSW Gradient selected COSY

F2 2D NMR_ICON COSY 1

1 Choice 2D NMR
(Modified full range)

Available 2D NMR_Full COSY 1 CDCI3 chloroform-d N COSY_Full_range -4 to 16 ppm

No need 1D PROTON

Icon-NMR (Change parameter - Simple)

IconNMR: Automation Jul21-2025-1857-UCRF

File Run Holder View Find Parameters Options Tools Help

IconNMR - Automation running UCRF

Do Acquisition

Experiment Table

Holder	Type	Status	Name	No.	Solvent	Experiment	Start Time
1	Available	Available	20250801-TEST-DMSO-D6_H1	1	DMSO	dimethylsulfo	N PROTON
2	Available	Available					
3	Available	Available					
4	Failed	Failed	20250731-PNU-THF-E_HOESY-7Li-1H_D8=1.51	15	D2O	deuteriumoxide	N HOESY_7Li-1H
5	Running	Running	20250731-PNU-THF-E_HOESY-7Li-1H_D8=1.7_with_U7_T1	18	D2O	deuteriumoxide	N HOESY_7Li-1H
6	Queued	Queued	20250731-PNU-DOL-E_HOESY-7Li-1H	20	D2O	deuteriumoxide	N HOESY_7Li-1H
7	Queued	Queued	20250731-PNU-MCP-E_HOESY-7Li-1H	1	D2O	deuteriumoxide	N HOESY_7Li-1H
8	Available	Available					

Submit Cancel Edit Add Copy Delete

1 Click 'Edit specific parameters'

Change Parameter (Simple)

2 Change simple parameter

3 Submit

#	Date	Holder	Name	No.	Solvent	Experiment	Load	ATM	Rotation	Lock	Shim	Acq	Proc	User	Disk	Title/Orig	Remarks
215	2025-08-01 11:32:43	5	20250731-PNU-DOL-E_HOESY-7Li-1H	20	D2O	HOESY_7Li-1H	✓	✓	✓	✓	✓	✓	✓	UCRF	D:\nmrdata\UCRF		
214	2025-08-01 10:01:40	4	20250731-PNU-THF-E_HOESY-7Li-1H_D8=1.51	15	D2O	HOESY_7Li-1H	✓	✓	✓	✓	✓	✓	✗	UCRF	D:\nmrdata\UCRF		ef: Command sref NUCLEUS peak for scaling get solvent re
213	2025-08-01 09:40:54	3	20250731-PNU-THF-S_U7	19	D2O	U7_UNIST	✓	✓	✓	✓	✓	✓	✓	UCRF	D:\nmrdata\UCRF		ef: Command AuditTrail.uns
212	2025-07-31 23:29:53	6	20250731-PNU-MCP-E_U7_T1	21	D2O	U7_T1		✓	✓			✓	✗	UCRF	D:\nmrdata\UCRF		

Search Preceding

Sample: 5

297.5K SampleCase™ Busy until: Fri 16:47 Day: 02:35 Night: 00:00

Icon-NMR (Change parameter - Complex)

1 Click 'Edit all Acquisition Parameters'

Change Parameter (Complex)

- Edit all Acquisition Parameters
- Edit Processing Parameters
- Pertinent Acquisition Parameter Editor
- User Specific Commands

2 Click Show pulse program parameters

3 Change parameters

4 Click Return to IconNMR

Experiment running

Do Acquisition

UCRF

Experiment

Par

Title...

Time

User

Start Time

SPECTRUM

PROCPARS

Return to IconNMR

Channel f1

General

Current pulse program

Size of fid

Sweep width in acquisition direction

Acquisition time

Receiver gain

Dwell time

Pre-scan delay

Relaxation delay; 1-5 * T1

Number of dummy scans

1 * n, total number of scans: NS * TD0

Number of averages in 1D

Channel f1

SFO1 [MHz]

Frequency of ch. 1

O1 [Hz, ppm]

Frequency of ch. 1

NUC1

Nucleus for channel 1

p0 [μsec]

p0 = p1/3

P1 [μsec]

F1 channel - 90 degree high power pulse

PLW1 [W, dB]

F1 channel - power level for pulse (default)

Experiment

Width

Nucleus

Receiver

Durations

PULPROG

AQ_mod

TD

DS

NS

TD0

Preceding Experiments

#	Date	Holder	Name	No.	Solvent
215	2025-08-01 11:32:43	5	20250731-PNU-DOL-E_HOESY-7U-1H	20	D2O
214	2025-08-01 10:01:40	4	20250731-PNU-THF-E_HOESY-7U-1H_D8=1.51	15	D2O
213	2025-08-01 09:40:54	3	20250731-PNU-THF-S_U7	19	D2O
212	2025-07-31 23:29:53	6	20250731-PNU-MCP-E_U7_T1	21	D2O

Search Preceding

IconNMR: Automation Jul21-2025-1857-UCRF

File Run Holder View Find Parameters Options Tools Help

IconNMR - Automation running

Do Acquisition

Current stage
현재 진행 중인 상태

1

2

3

4

5

6

7

8

Status	상태	Notes
Available	비어있는 홀더	사용 가능
Finish	완료된 실험	삭제가능
Running	진행 중인 실험	현재 진행 중
Queued (Ready)	대기 중인 실험	예약상태
Fail	실패한 실험	관리자계정 (Bruker 400: nmrsu Bruker 600: root) or 해당 User로만 삭제가능

Holder	Type	Status	Name	Par	Title...	Time	User	Start Time
1	1	Available	20250731-PNU-MCP-E_HOESY-7U-1H	1	HOESY_7U-1H	00:01:08	UCRF	Set Start Time
2	1	Available	20250731-PNU-DOL-E_HOESY-7U-1H	20	HOESY_7U-1H	01:27:12	UCRF	10:01 Fri Aug 01 2025
3	1	Available	20250731-PNU-THF-E_HOESY-7U-1H_08=1.51	15	HOESY_7U-1H	01:30:33	UCRF	00:00 Sat Aug 02 2025
4	2	Failed	20250731-PNU-THF-E_HOESY-7U-1H_08=1.51	15	HOESY_7U-1H	02:39:16	UCRF	11:32 Fri Aug 01 2025
5	1	Failed	20250731-PNU-THF-E_HOESY-7U-1H_08=1.51	15	HOESY_7U-1H	00:52:08	UCRF	14:16 Fri Aug 01 2025
6	1	Running	20250731-PNU-THF-E_HOESY-7U-1H_08=1.51	15	HOESY_7U-1H			
7	1	Running	20250731-PNU-THF-E_HOESY-7U-1H_08=1.51	15	HOESY_7U-1H			
8	1	Available	20250731-PNU-THF-E_HOESY-7U-1H_08=1.51	15	HOESY_7U-1H			

#	Date	Holder	Name	No.	Solvent	Experiment	Load	ATM	Rotation	Lock	Shim	Acq	Proc	User	Title/Orig	Remarks
216	2025-08-01 14:16:37	6	20250731-PNU-MCP-E_HOESY-7U-1H	1	D2O	HOESY_7U-1H	✓	✓	✓	✓	✓	✓	✓	UCRF		
215	2025-08-01 11:32:43	5	20250731-PNU-DOL-E_HOESY-7U-1H	20	D2O	HOESY_7U-1H	✓	✓	✓	✓	✓	✓	✗	UCRF		ef: Command AuditTrail upd
214	2025-08-01 10:01:40	4	20250731-PNU-THF-E_HOESY-7U-1H_08=1.51	15	D2O	HOESY_7U-1H	✓	✓	✓	✓	✓	✓	✗	UCRF		ef: Command sref: NUCLEUS peak for scalin not solvent re
213	2025-08-01 09:40:54	3	20250731-PNU-THF-E_HOESY-7U-1H_08=1.51	19	D2O	L7_UNIST	✓	✓	✓	✓	✓	✓	✓	UCRF	D:\nmrdata\UCRF	

Progress stage
완료된 과정

Run

Queue

Available

Submit

Cancel

Edit

Add

1

Copy

1

X Delete

Button	Function	Notes
Submit	Run experiments	원하는 홀더 번호 선택 후 Submit
Cancel	Queued -> Available	준비 -> 사용가능상태로
Edit	Available -> Edit	수정필요시
Add	1 Sample -> Multiple experiments	Save time, Lock/Shim just run 1 time (시간절약, Lock/Shim은 1회만 진행)
Copy	Copy Sample name, Solvent, Parameter (For Similar experiment)	실험명, 용매, 실험, 파라미터 복사 (유사한 샘플 또는 유사한 실험 진행시 활용)
Delete	Delete experiment	실험삭제 (Fail 상태는 User or Admin만 삭제가능)

Icon-NMR (Spin on/off)

The screenshot shows the Icon-NMR software interface with the following parameters:

Parameter	Value	Description
TD	65536	Size of fid
DS	2	Number of dummy scans
AQ	2.75251 [sec]	Acquisition time
NS	16	Number of scans
P1	11.8 [μsec]	Pulse
D1	1 [sec]	Delays
D8	0	Delays
RO	0 [Hz]	Rotation frequency of sample
O1P	6.175 [ppm]	Transmitter frequency offset
1SW	19.8329 [ppm]	Spectral width (F1)

An 'OK' button is located at the bottom left of the parameter table.

RO (Rotation On)
Spinning = 20
Non Spinning = 0

Icon-NMR (Parameter list)

SPECTRUM PROCPARS **ACQUPARS** TITLE PULSEPROG PEAKS INTEGRALS SAMPLE STRUCTURE PLOT FID AC

Return to IconNMR Probe: Prodigy

General

Channel f1

General

PULPROG zg30 E Current pulse program

TD 65536 Size of fid

SWH [Hz, ppm] 11904.76 19.8329 Sweep width in acquisition direction

AQ [sec] 2.7525120 Acquisition time

RG 32 Receiver gain

DW [µsec] 42.000 Dwell time

DE [µsec] 18.00 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] 600.2537065 Frequency of ch. 1

O1 [Hz, ppm] 3706.54 6.175 Frequency of ch. 1

NUC1 1H Edit... Nucleus for channel 1

p0 [µsec] 3.93 p0=p1/3

P1 [µsec] 11.8000 F1 channel - 90 degree high power pulse

PLW1 [W, dB] 25 -13.98 F1 channel - power level for pulse (default)

List	Explanation	Notes
PULPROG	실험프로그램	zg30(30도 펄스) zg (90도 펄스)
TD	Time domain	FID point 개수
SWH(ppm)	Spectral width	스펙트럼 총 폭 (X축)
AQ	Acquisition time	데이터 얻는 시간 (TD * DW)
RG	Receiver gain	Detector window size
DW	FID Point사이 간격	
DE	Acquisition 이전 delay	
D1	Relaxation delay time	Integration값과 연동 T1 * 5
DS	Dummy scan	실제스캔이전 준비운동
NS	Number of scans	실제스캔
SFO1	Resonance frequency	Target 핵종의 에너지 분리량
O1(ppm)	Center frequency	스펙트럼 중심값
NUC1	Target nucleus	측정하고자 하는 핵종
P0	Input RF pulse (time)	실제로 들어가는 RF pulse 시간 ex) zg30 (P0=P1/3)
P1	Input RF pulse (time)	입력된 RF pulse 시간
PLW1	RF power	RF pulse power level

IconNMR: Automation Jul21-2025-1857-UCRF

File Run Holder View Find Parameters Options Tools Help

IconNMR - Automation running UCRF

Do Acquisition

Experiment Table

Holder	Type	Status	Name	No.	Solvent	Experiment	Load	ATM	Rotation	Lock	Time	User	Start Time
1	1	Available	20250801-TEST-DMSO-D6_H1	1							00:01:08	UCRF	Set Start Time
2	1	Available											
3	1	Available											
4	2	Failed	20250731-PNU-THF-E_HOESY-7Li-1H_D8=1.51	15	D2O	deuteriumoxide	N HOESY_7Li-1H						
5	1	Failed	20250731-PNU-THF-E_HOESY-7Li-1H_D8=1.7_with_Li7_T1	18	D2O	deuteriumoxide	N HOESY_7Li-1H						
6	1	Failed	20250731-PNU-DOL-E_HOESY-7Li-1H	20	D2O	deuteriumoxide	N HOESY_7Li-1H						
7	1	Running	20250731-PNU-MCP-E_HOESY-7Li-1H	1	D2O	deuteriumoxide	N HOESY_7Li-1H						
8	1	Available											

Submit Cancel Edit Add 1 Copy 1 X Delete

Preceding Experiments

#	Date	Holder	Name	No.	Solvent	Experiment	Load	ATM	Rotation	Lock
216	2025-08-01 14:16:37	6	20250731-PNU-MCP-E_HOESY-7Li-1H	1	D2O	HOESY_7Li-1H	✓	✓	✓	✓
215	2025-08-01 11:32:43	5	20250731-PNU-DOL-E_HOESY-7Li-1H	20	D2O	HOESY_7Li-1H	✓	✓	✓	✓
214	2025-08-01 10:01:40	4	20250731-PNU-THF-E_HOESY-7Li-1H_D8=1.51	15	D2O	HOESY_7Li-1H	✓	✓	✓	✓
213	2025-08-01 09:40:54	3	20250731-PNU-THF-S_Li7	19	D2O	Li7_UNIST	✓	✓	✓	✓

Search Preceding

Acquisition information

Name/Expts: 20250731-PNU.../20

Scan Exp.: 4/4 200/256

Residual time: 33m50s

Fid Flash Lock 100%

TD 65536 Size of fid

DS 2 Number of dummy scans

AQ 2.75251 [sec] Acquisition time

NS 16 Number of scans

P1 11.8 [μsec] Pulse

D1 1 [sec] Delays

D8 0 Delays

RO 0 [Hz] Rotation frequency of sample

O1P 6.175 [ppm] Transmitter frequency offset

1SW 19.8329 [ppm] Spectral width (F1)

OK

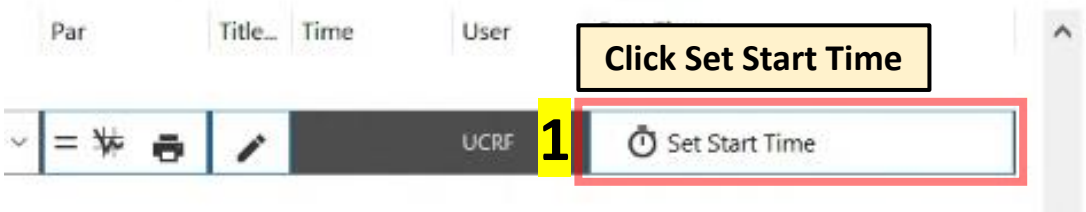
297.5K SampleCase™ Busy until: Fri 16:47 Day: 02:17 Night: 00:00

Only Show acquisition time
(Load, ATM, Lock, Shim 시간 제외)

Show time = 00:01:08
Real time = 6~7 min

Experiment time
남은 시간 확인

D8 or RO 등 0인 값에 0을 다시넣고
OK 누르면 실험시간 확인가능



overwrite - O
arrow key - O
Delete - X
Backspace - X

▼ 14	1	Queued							
		Queued	250804 KJB BA	1	Tol-H8	toluene, non-deute	N Pb207_UNIST		
▼ 15	1	Queued							
		Queued	250804 KJB DPP	1	Tol-H8	toluene, non-deute	N Pb207_UNIST		
▼ 16	1	Queued							
		Queued	250804 KJB BSA	1	Tol-H8	toluene, non-deute	N Pb207_UNIST		

3 Many samples
Just +1 min is enough

00:25:23	SMH	21:00 Mon Aug 04 2025
00:25:23	SMH	21:01 Mon Aug 04 2025
00:25:23	SMH	21:02 Mon Aug 04 2025

Icon-NMR (Open data)

Queued 20250804-HANTOK-TMAC-58-TK-753-50802_H1 1 10%_D2O_HANTOK N PROTON_HANTOK 10 degree 00:30:46 UCRF 17:30 Mon Aug 04 2025

12 Available
13 Available
1 Queued

A

Double click Preceding Experiment's list
[Finished experiment]

#	Time	Exp	Load	ATM	Rotation	Lock	Shim	Acq	Proc	User	Disk	Title/Orig	Remarks
14	2025-08-04 16:55:41	10 20250804-HANTOK-TMAC-58-TK-742-50804_H1	✓	✓	✓	✓	✓	✓	✓	UCRF	D:\nmrdata\UCRF		
13	2025-08-04 16:20:07	9 20250804-HANTOK-TMAC-58-TK-741-50802_H1	✓	✓	✓	✓	✓	✓	✓	UCRF	D:\nmrdata\UCRF		
12	2025-08-04 15:47:11	8 20250804-HANTOK-TMAH-20-TK-422-50804_H1	✓	✗	✓	✓	✓	✓	✓	UCRF	D:\nmrdata\UCRF		log_err: Cannot
11	2025-08-04 15:10:20	5 42Ah_Li_sol_5th_250804_P	4	H2O	P31_Li_Salt	✓	✓	✓	✓	YK	D:\nmrdata\YK		

Start in Preceding [Search] Sample: 10 298.3K SampleCase™ Busy until: Mon 19:34 Day: 02:15 Night: 00:00

B

Double click Experiment's list
[Finished experiment]

- + 20250725-SESL-#3_F19
- + 20250725-SESL-#3_H1
- + 20250725-SESL-#3_Li7
- + 20250725-SESL-#5_Li7
- + 20250725-SESL-#6_Li7
- + 20250725-SESL-#7_Li7
- + 20250725-SESL-#8_F19
- + 20250729-HANTOK-TMAC58-TK-750-50727_H1
- 20250729-HANTOK-TMAH20-TK-421-50726_H1
- 1 - zg
- + 20250729-SESL-#10_F19
- + 20250729-SESL-#10_H1
- + 20250729-SESL-#10_Li7
- + 20250729-SESL-#1_F19

Export

1

4 Type efp; apk; absn

3

Acquisition information

Name/Expno: 20250804-HAN.../1
Scan: 48/256
Residual time: 24m35s

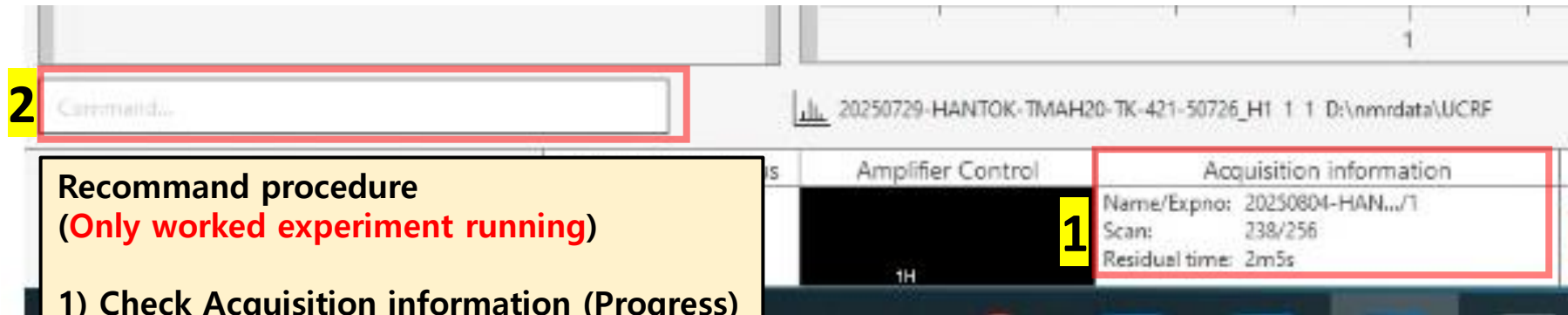
- C**
- Save & show data in progress
- 1) Type *tr* on command line
 - 2) Check Stored NS OO
 - 3) Double click Acquisition information
 - 4) Click Process Proc. Spectrum (or Type efp; apk; absn)
- efp: FID to spectrum
apk: Auto phase correction
absn: Auto baseline correction

2

stored NS 72 into /root/.topspin-BladeEpu/local_acqu/...

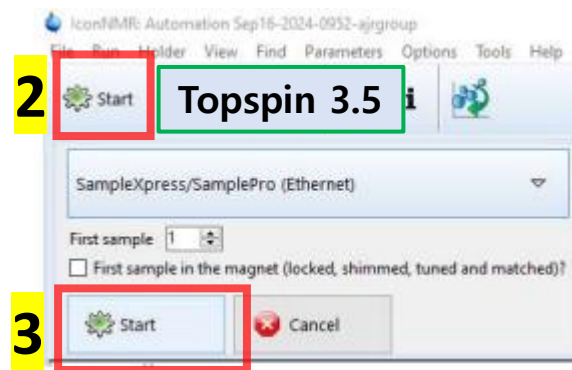
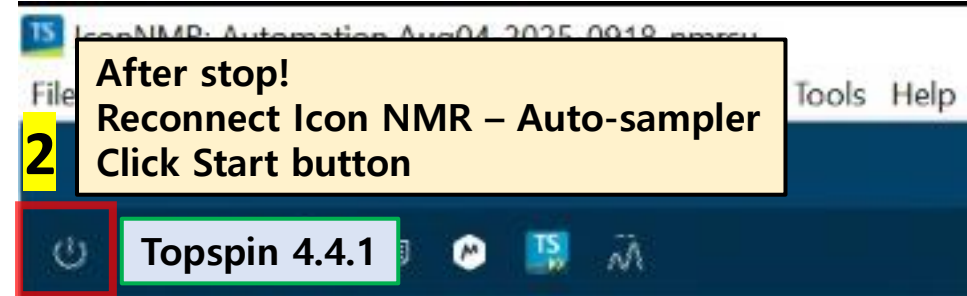
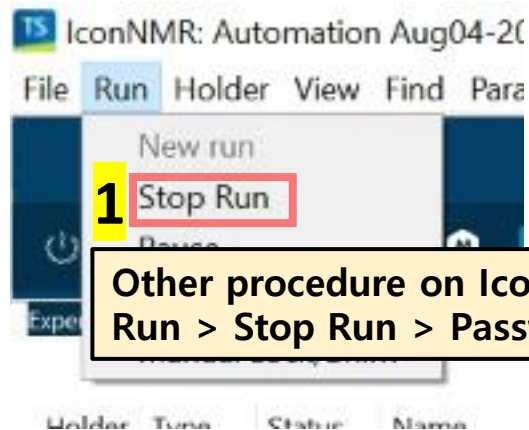
AutoDiagnose	Spooler	BSMS Auto Status	Time
Sending Status	queued: 0 delayed: 0 cron: 0		17:10:00 Aug 04

Icon-NMR (Stop run)

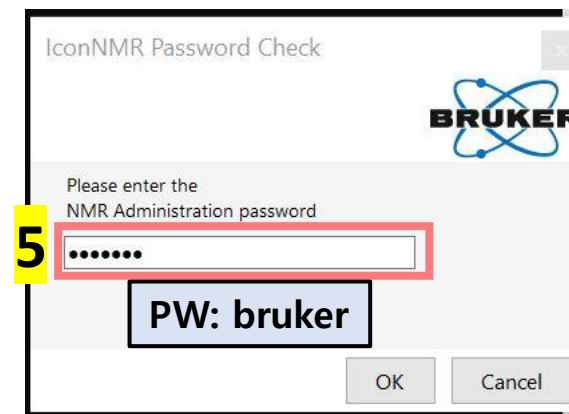
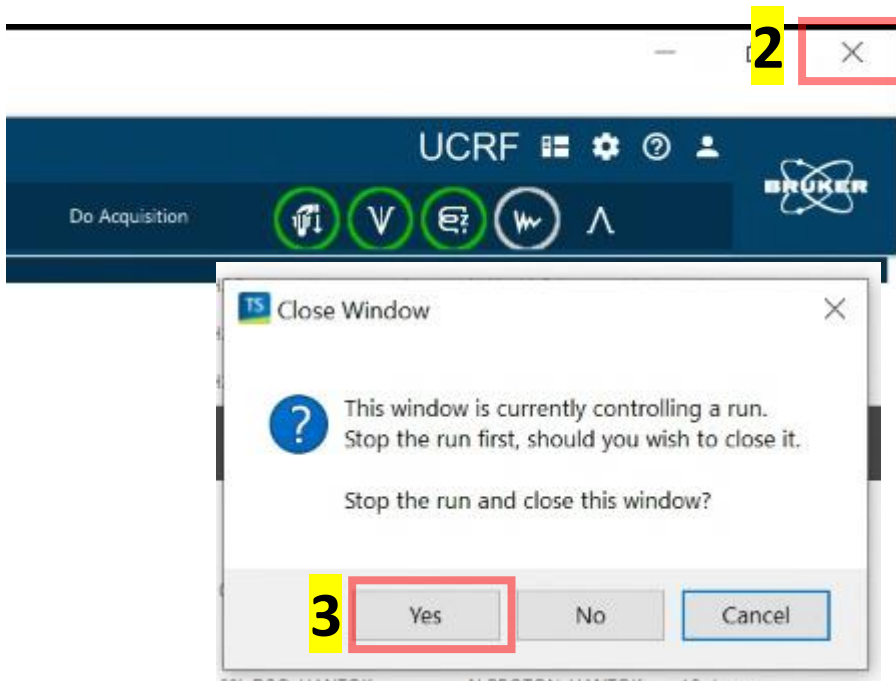
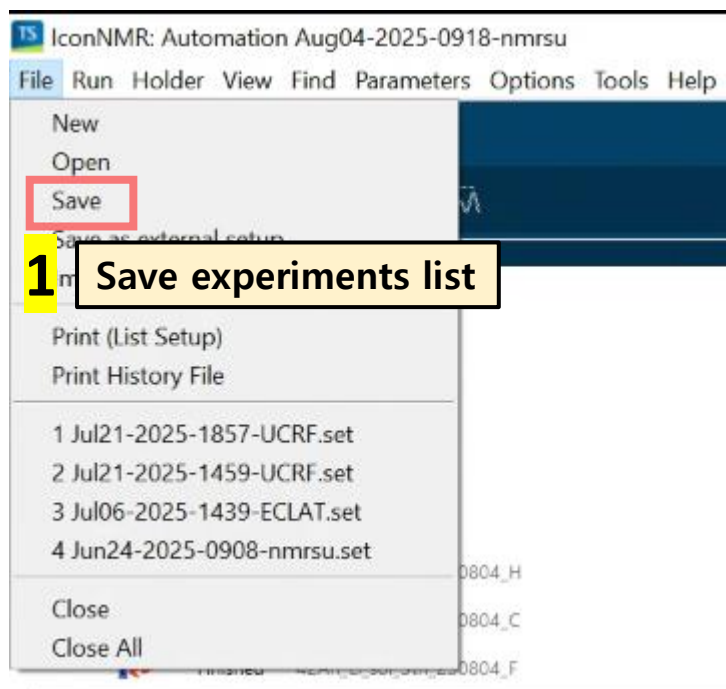


Recommand procedure
(Only worked experiment running)

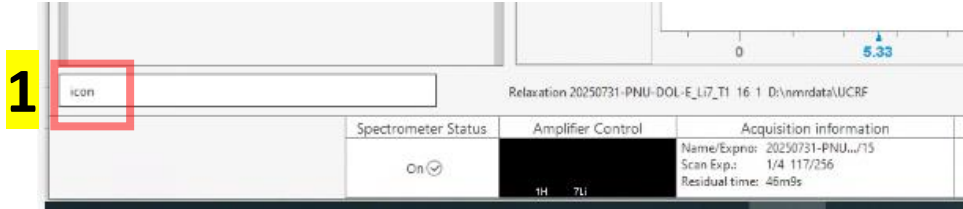
- 1) Check Acquisition information (Progress)
- 2) Type *halt* on command line
halt: Save and stop



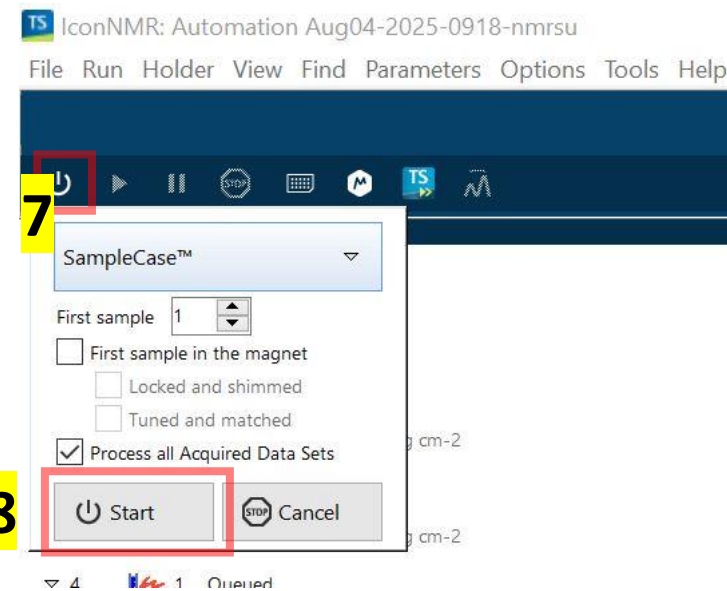
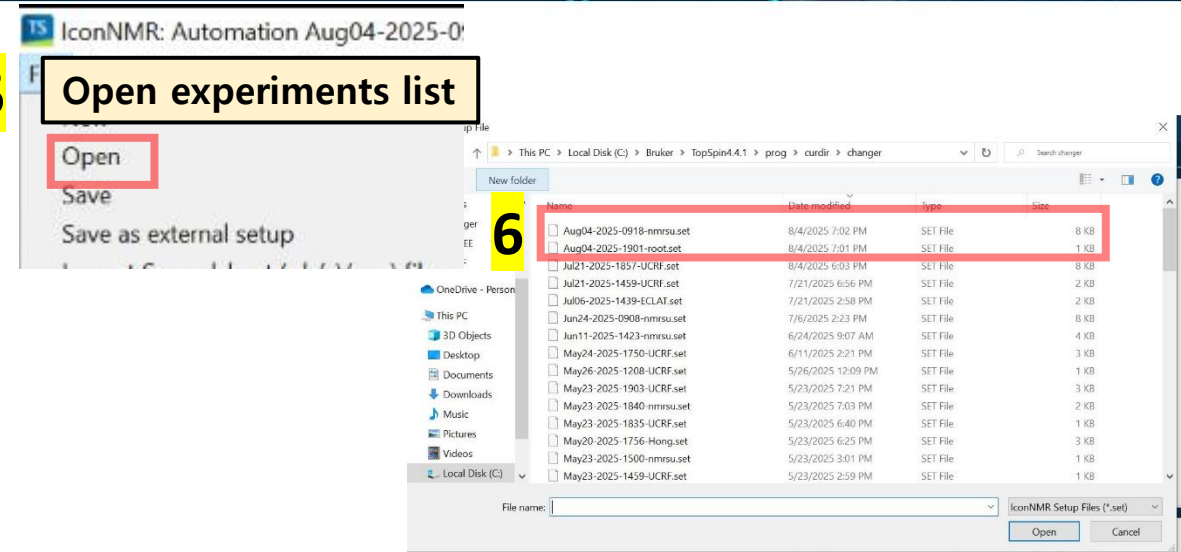
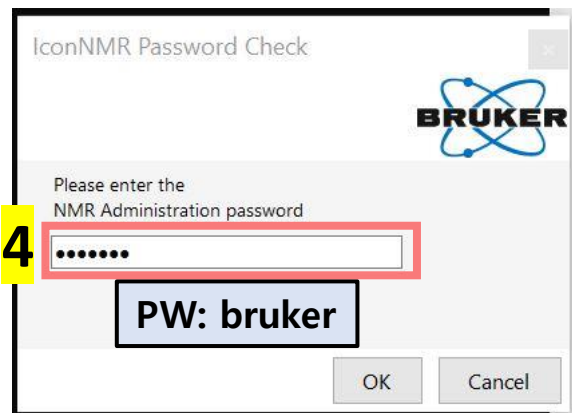
Icon-NMR (Turn off)



Icon-NMR (Turn on)



Type *icon* on command line





Bruker 600
Software: Topspin 4.4.1
Windows10

Admin (Icon-NMR)
ID: root
PW: topspin

Windows 10
ID: nmrsu
PW: topspin

Topspin/Icon-NMR config
PW: bruker

Admin (Icon-NMR)

- 1) Delete fail experiment**
- 2) Exit Icon-NMR**
- 3) Control other user list**

Windows 7/10

- 1) Turn on PC**

Topspin/Icon-NMR config

- 1) Exit icon-NMR**
- 2) Change configuration**



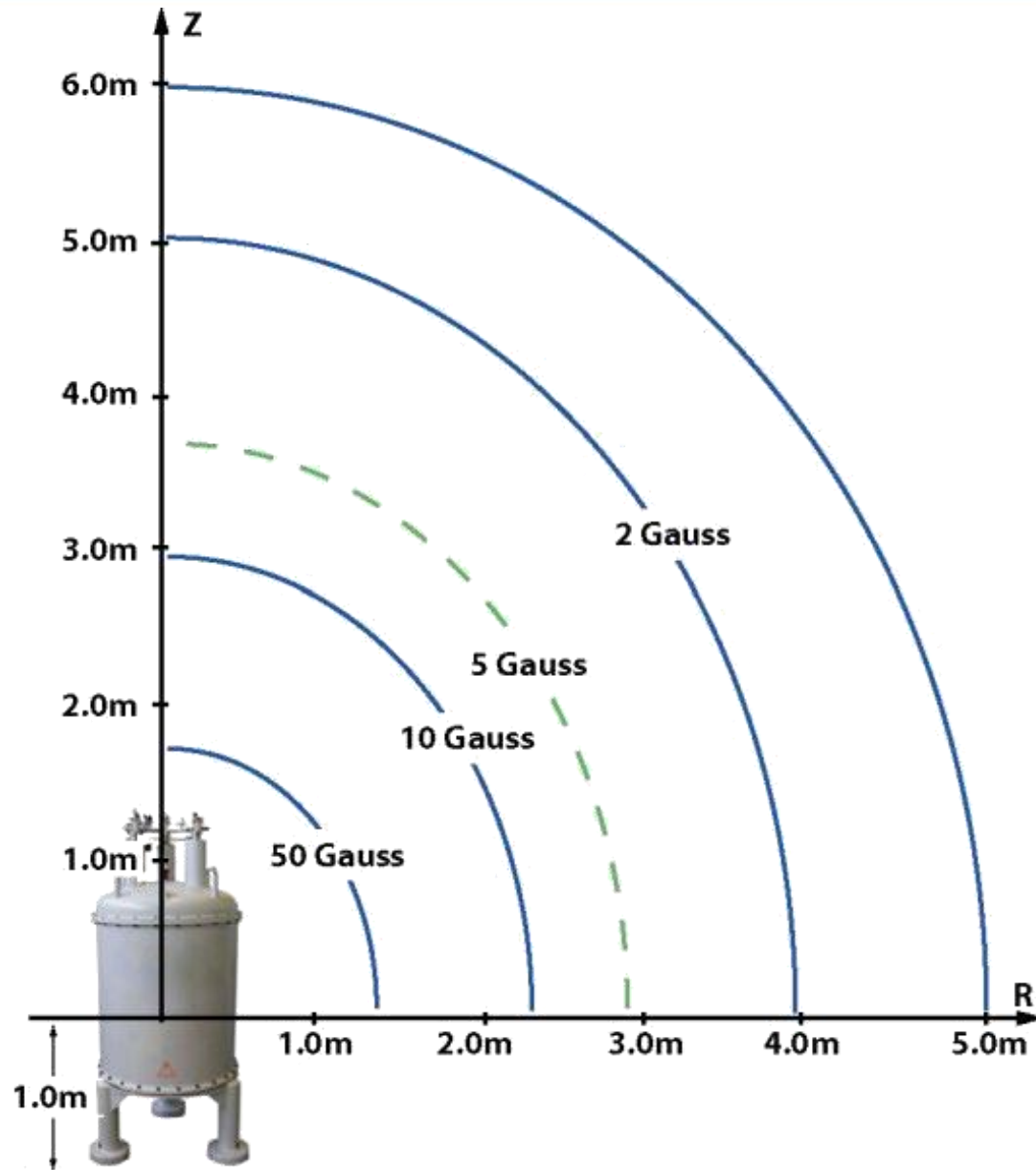
Bruker 400
Software: Topspin 3.5 pl7
Windows7

Admin (Icon-NMR)
ID: nmrsu
PW: topspin

Windows 7
ID: nmrsu
PW: topspin

Topspin/Icon-NMR config
PW: bruker

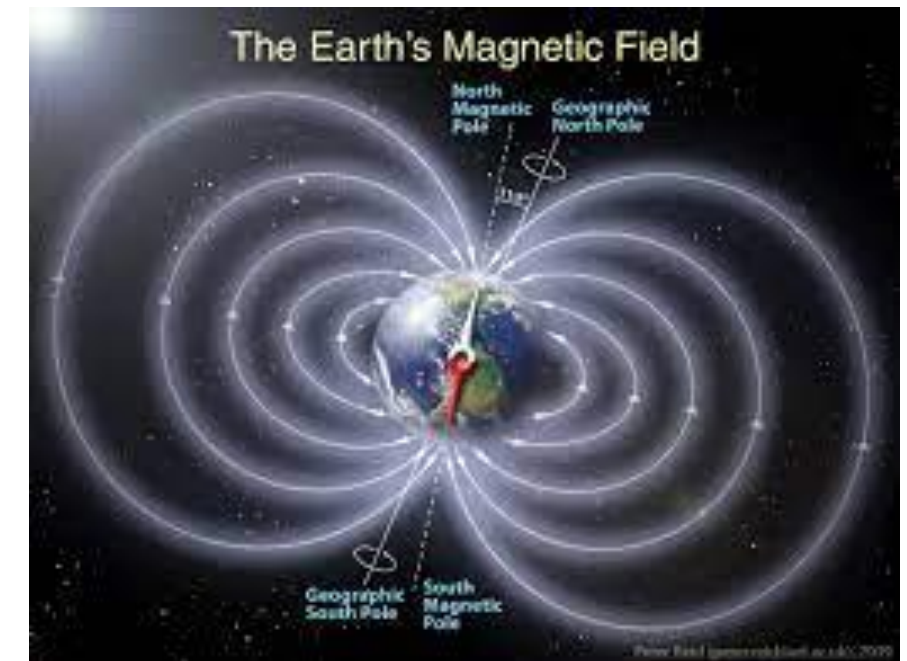
Safety



5 Gauss line

'Safe' levels of static magnetic field exposure for general purpose

Earth's Magnetic field = **0.5 gauss**





Caution: Extremely high magnetic stray fields.



Caution: Watches and electronic or electro-mechanical devices may be damaged.



Danger: No entrance for people carrying pace-makers.

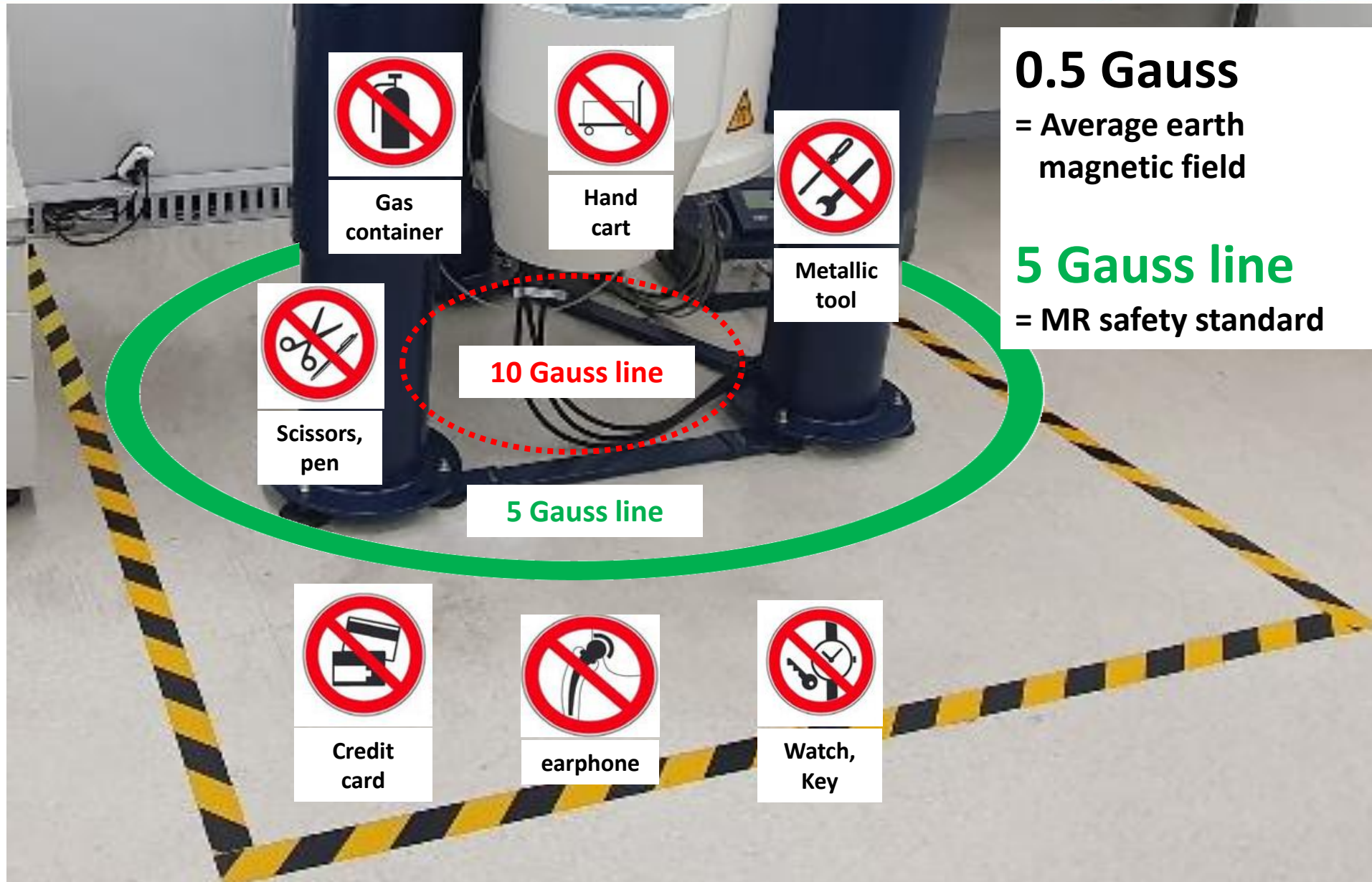


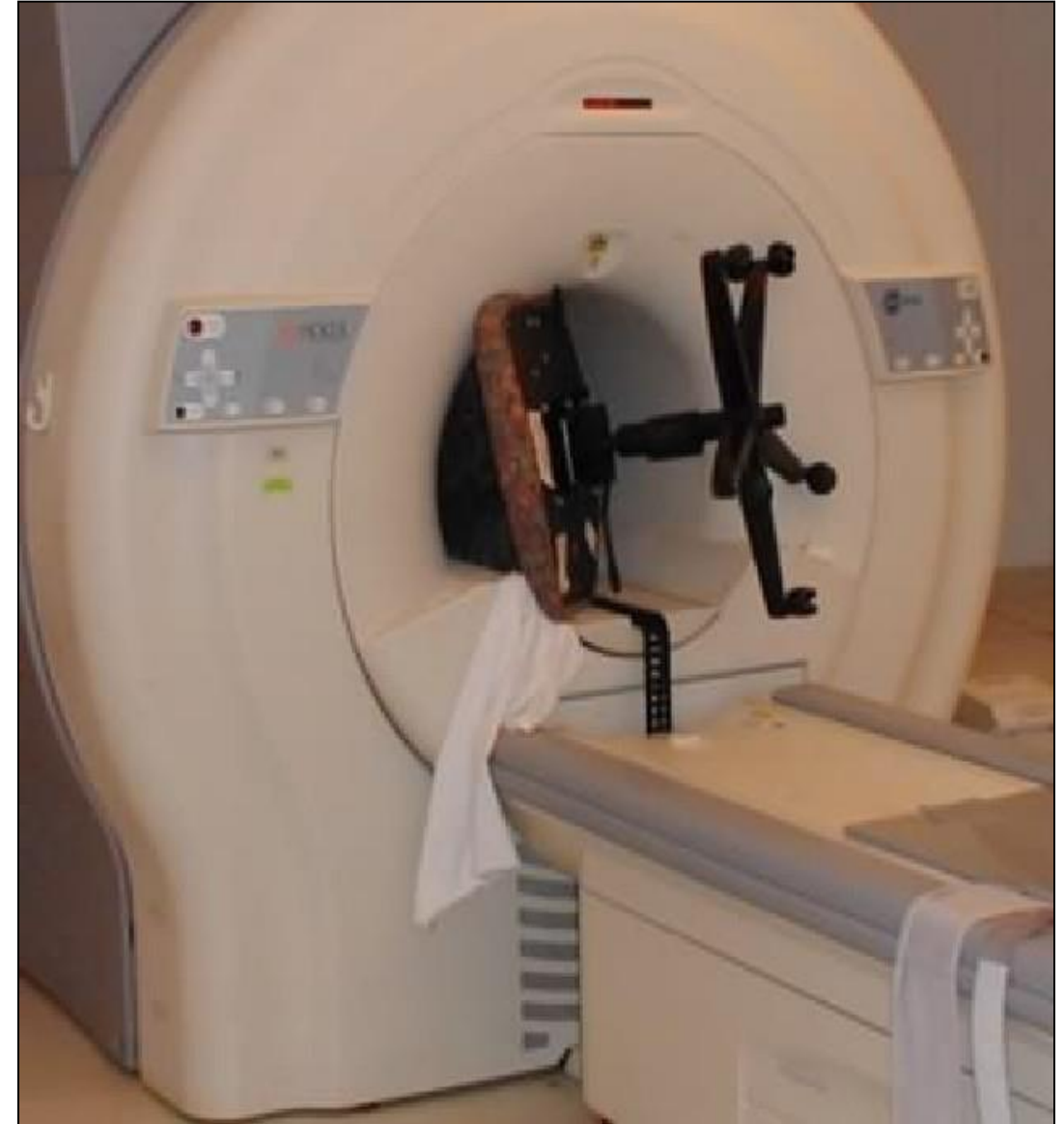
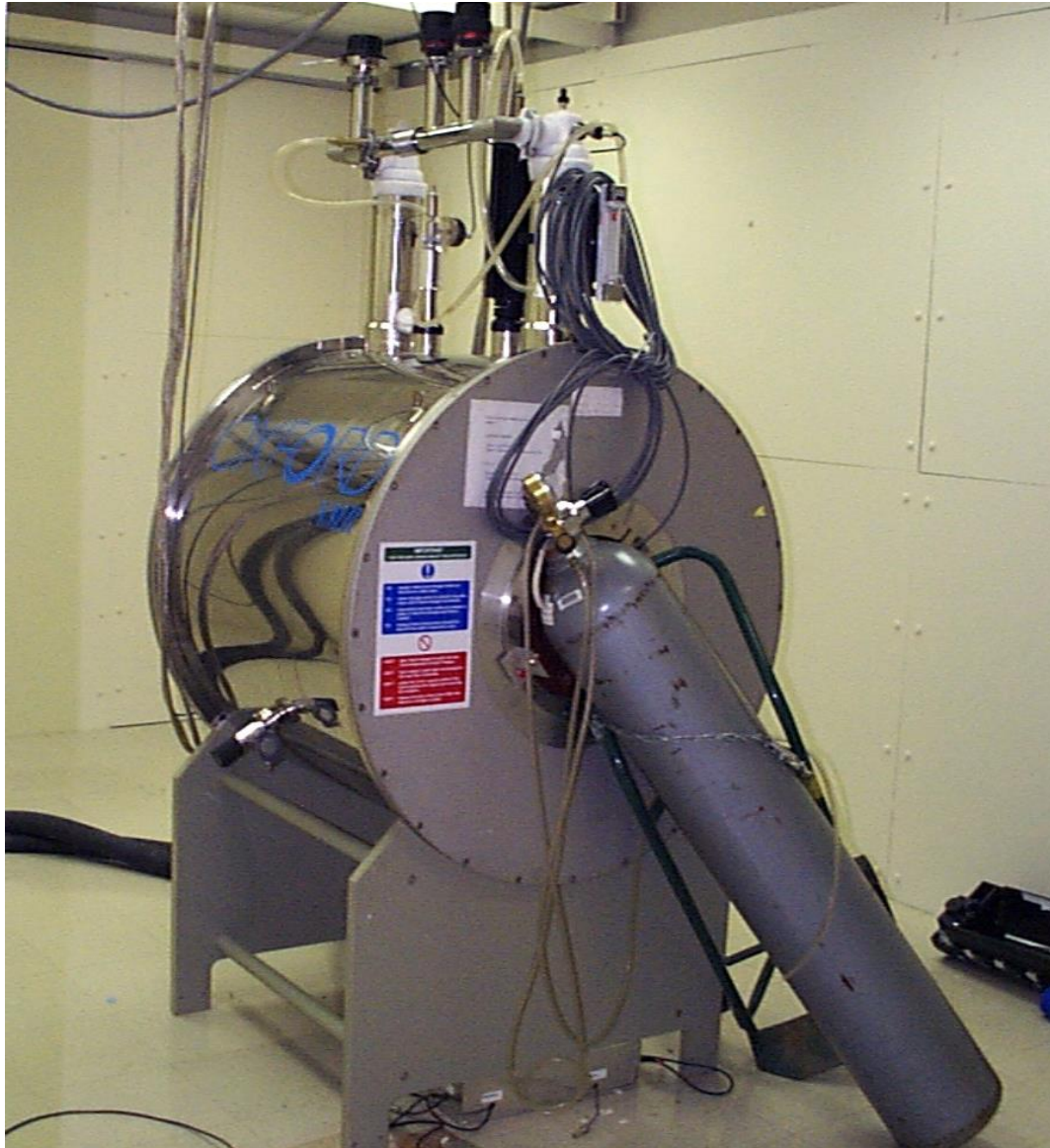
Caution: Credit cards, magnetic storage media as tapes, floppy disks or hard disks, may be damaged.



Danger: No entrance for people carrying medical implants.

Warning signs







Human Body react to Oxygen-Deficiency

O2 Concentration
21%

Symptoms
Natural air



O2 Concentration
18%

Symptoms
Limit level for not causing serious health problems. Continuous ventilation is required



O2 Concentration
16%-12%

Symptoms
Rapid breathing, Increase in pulse rate, Loss of concentration, Headache, Nausea, Ear ringing



O2 Concentration
14%-9%

Symptoms
Stupor, Headache, Nausea, Cyanosis, Faintness on the entire body



O2 Concentration
10%-6%

Symptoms
Comatose, Loss of consciousness, Muscle spasm on the entire body



O2 Concentration
6% or less

Symptoms
Unconsciousness, Comatose, Cessation of breathing, Cardiac arrest, Die in 6 minutes



https://youtu.be/d-G3Kg-7n_M?t=5