



DEPARTMENT OF
CHEMISTRY & BIOCHEMISTRY

University of Maryland, College Park
Analytical NMR Service & Research Center



*Updated by Fu Chen
January 2023*

NMR Facility Safety Orientation

How to get help: Contact Us !

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How to look NMR Basic information and Training notes.

What is in a Basic Level Check Out?

How to reserve NMR time?

How to keep all of us **SAFE** !

How not to break NMR tubes!

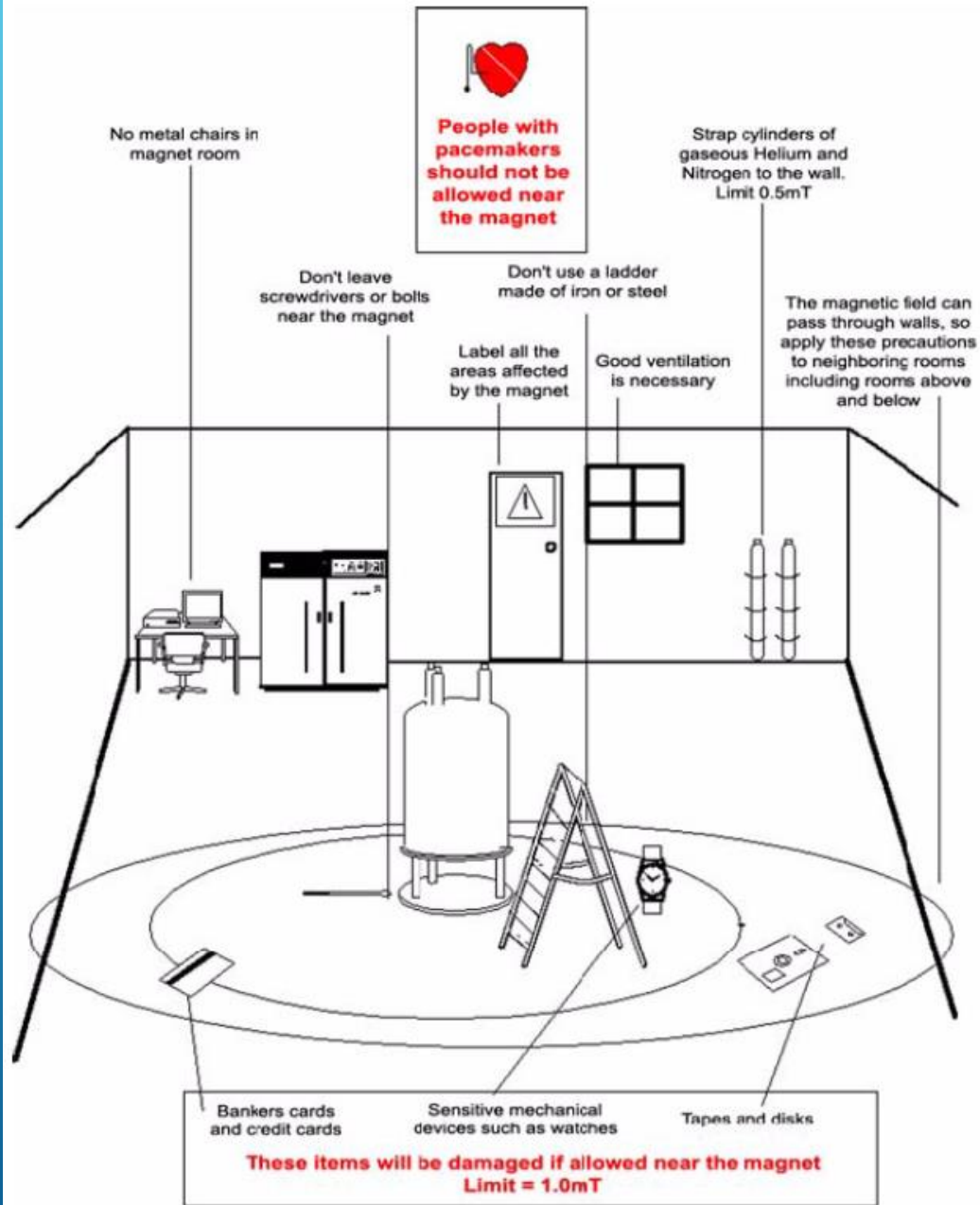


<https://blog.umd.edu/nmr/>

NMR Spectrometers Safety Aspects

- Magnetic Safety
 - Safety Precautions within the Inner Zone
 - Safety Precautions within the Outer Zone
 - Magnetic field strength: 4.7 – 14.1 T in our NMR facility
 - Earth field strength: 25 to 65 μT
- Cryogenic Safety
 - Liquid N₂ (77K) refill once a week
 - Liquid He (4.15K) refill once for a few months
- Electrical Safety
 - High voltage: 208 V
- Chemical Safety

Figure 2.1



Potential Risks of All Super-Conducting Magnets!



Ascend
Magnet
2019



Non-
shielded
Magnet
1996



Ultra-
shielded
Magnet
2007

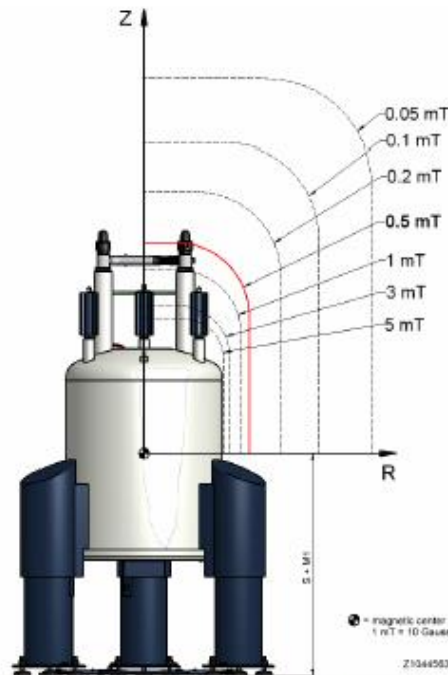


NEW BRUKER 500 MHz NMR MAGNET: 2019



Bruker BioSpin

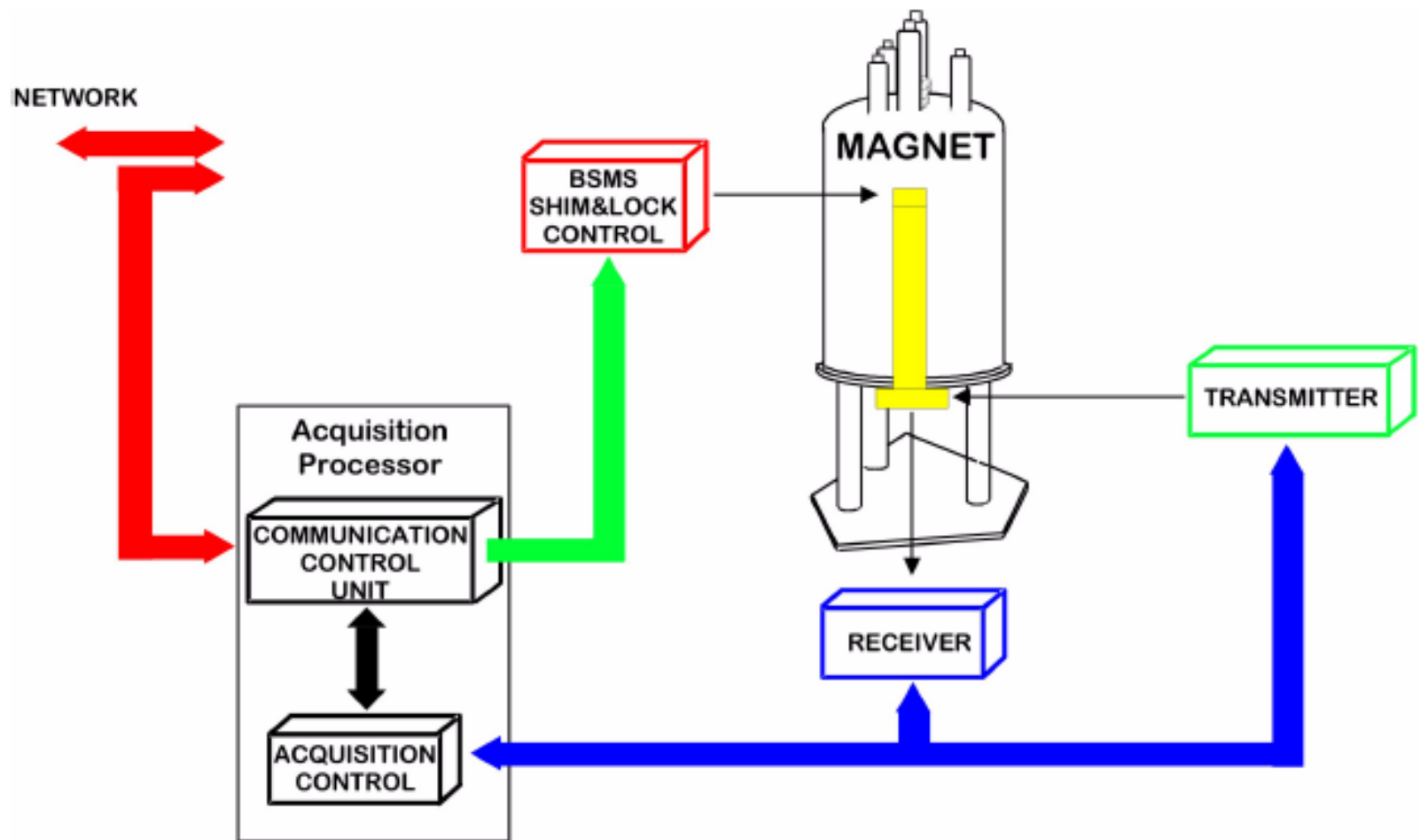
Fringe Field Plot



Fringe field contour	Radial [R]	Axial [Z]
200 mT (Directive 2004/40/EC)	Inside cryostat	Inside cryostat
5 mT	0.47 m	0.80 m
3 mT	0.50 m	0.87 m
1 mT	0.56 m	1.06 m
0.5 mT (5 Gauss)	0.60 m	1.20 m
0.2 mT	0.74 m	1.48 m
0.1 mT	0.96 m	1.75 m
0.05 mT (~Earth magnetic field)	1.26 m	2.09 m

2.7 AVANCE Architecture Overview

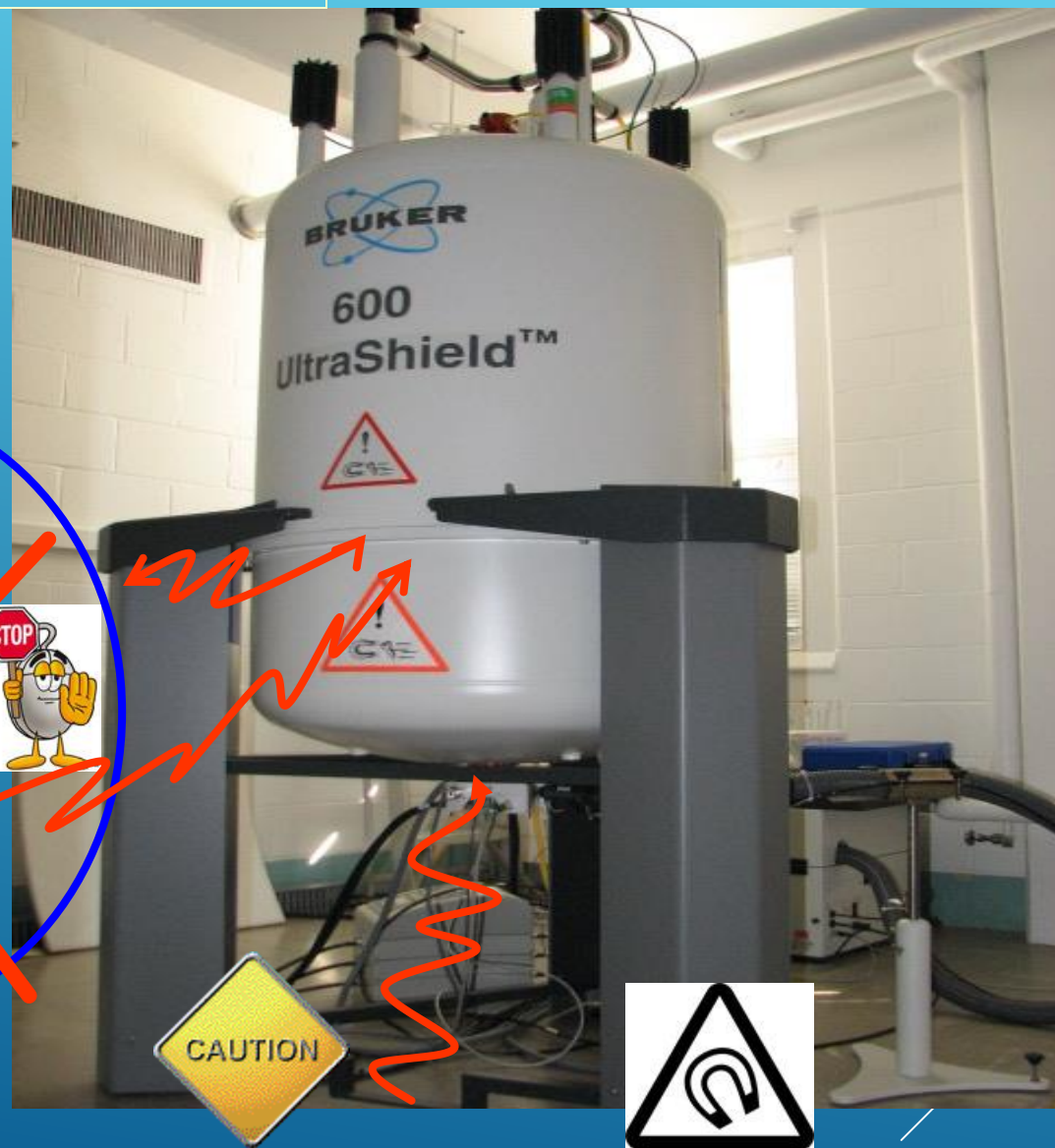
Figure 2.2

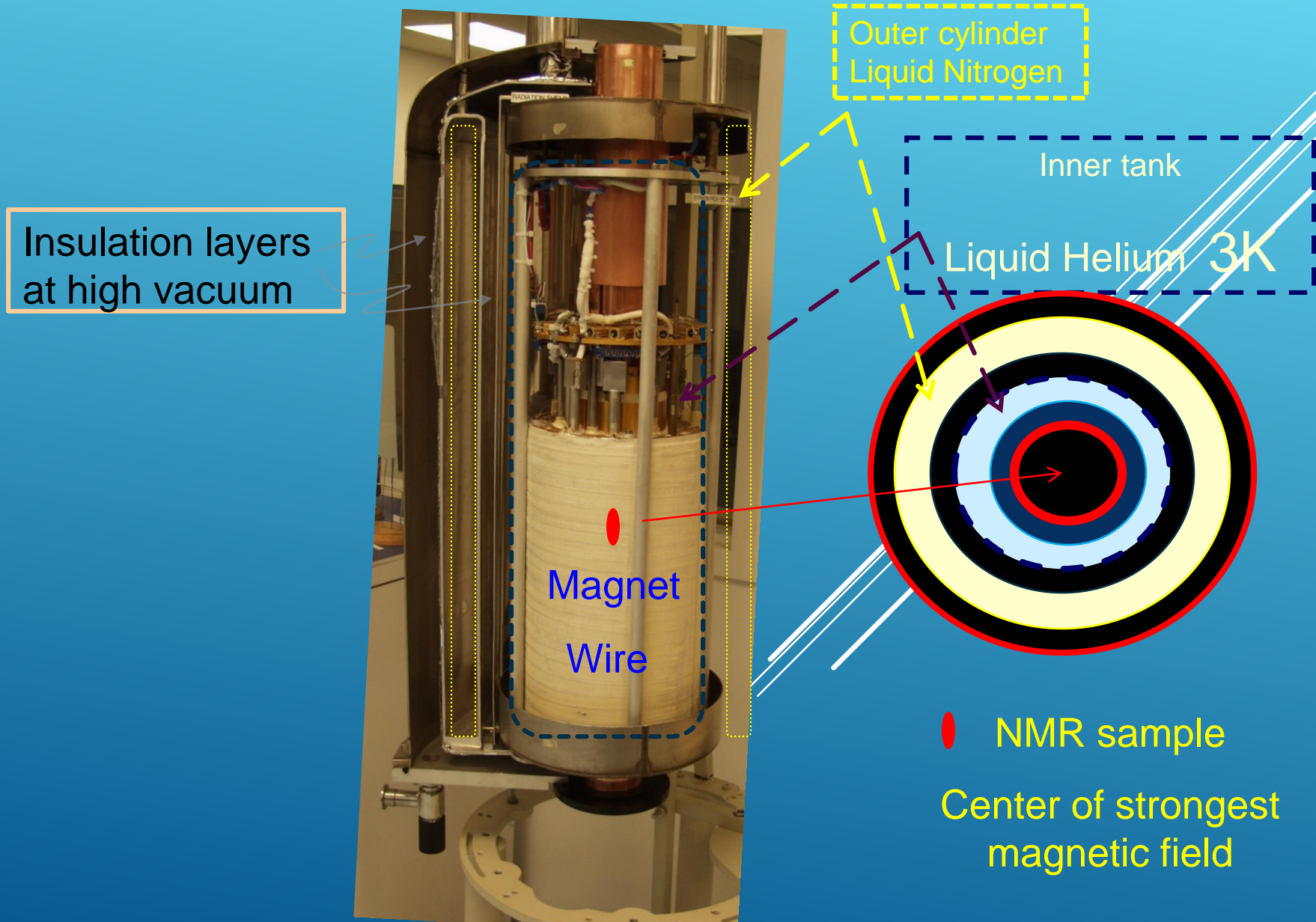


LAB SAFETY

- ▶ **never** go into back of spectrometer consoles (high voltages: 208V)
- ▶ use **care** around antivibration platform/legs
- ▶ **always be vigilant with VT**
 - use appropriate care with cryogenics; cryogen fill rooms are intrinsically unsafe!!
 - no ice build-up on magnets; heating tape on
 - keep probes within specified VT range
BBFO probe: -150 °C temp +120 °C
- ▶ correct use of decoupler (watch pl1 pl)
- ▶ staples, paper clips, tools

STRONG MAGNET FIELD:
EVEN FOR A SHIELDED
MAGNET





Refill Liquid Nitrogen

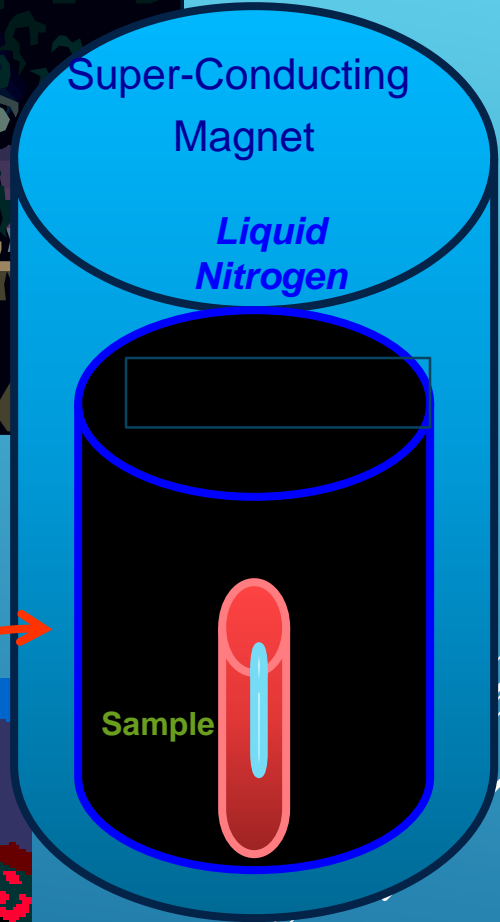
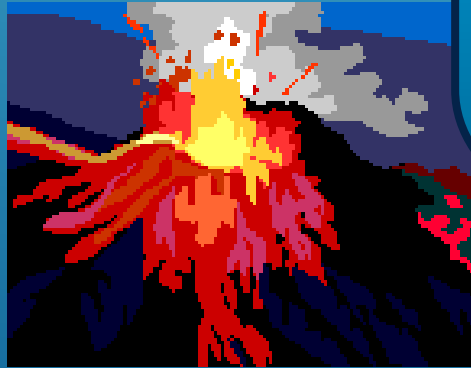
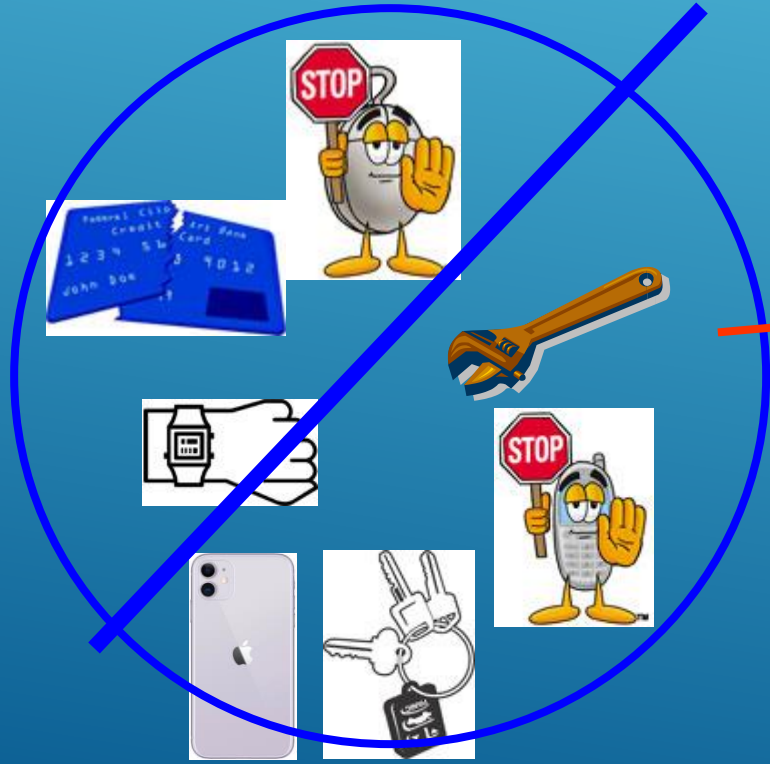


Refill Liquid Helium



WHEN TANKS TOUCH (SHORT) — INSULATION IS THEN BROKEN —
CRYOGEN BOILS OFF ALL AT THE SAME TIME —

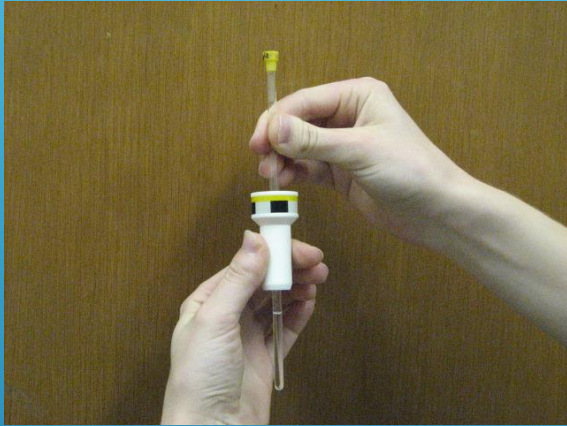
HIGH PRESSURE BUILDS UP!



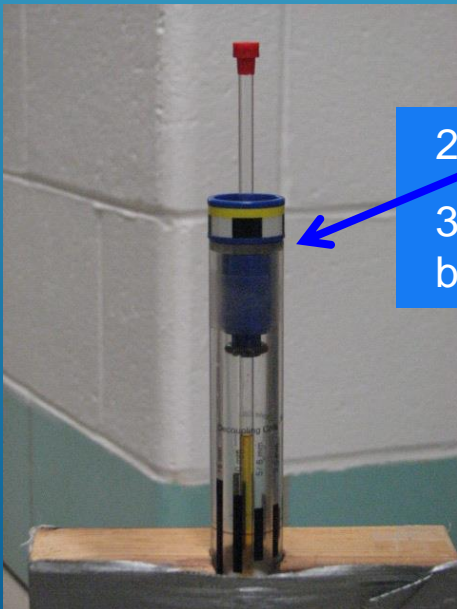
HOW TO (NOT TO) BREAK A NMR TUBE?

RECOMMENDED WAY TO *INSTALL NMR SAMPLE INTO A ROTOR*

- Select the blue plastic spinner (rotor) from the bench

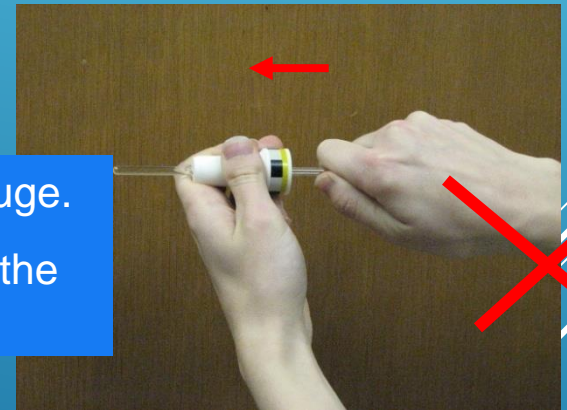


1. Hold the NMR tube steady and gently rotate the spinner & push it upward along the NMR tube!



2. Place the spinner on the depth gauge.

3. Push gently the tube till it touches the bottom of the gauge



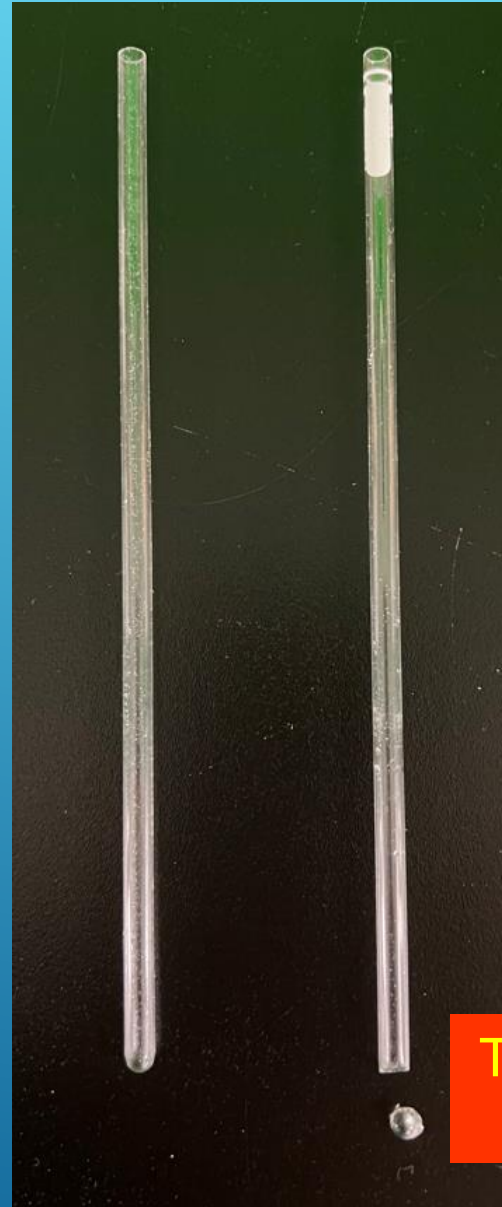
Fragile!



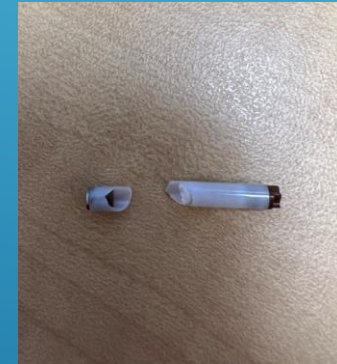
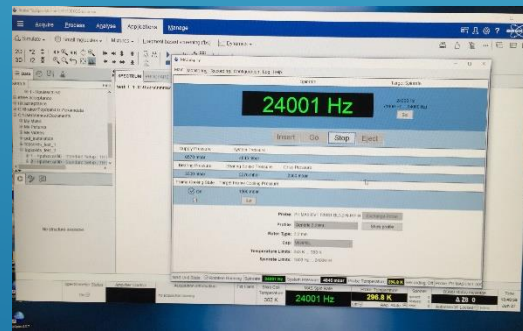
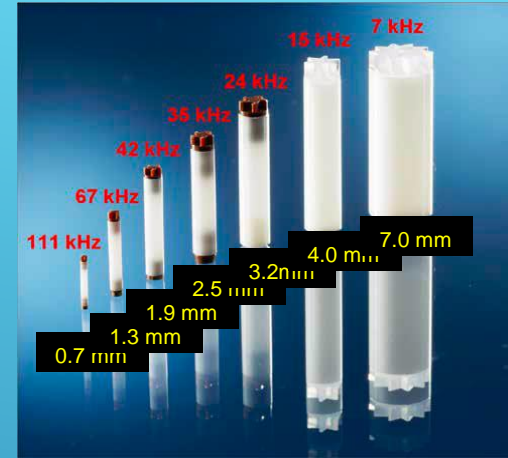
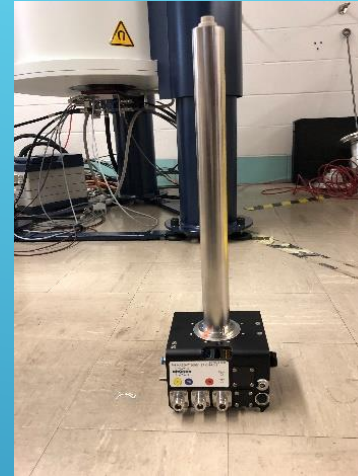
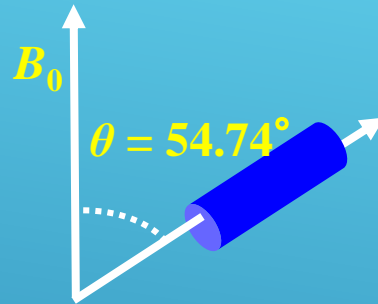
Paint on Spinner
washed away by
solvent



Tube broken
in probe



Bruker solid-state NMR spectrometers and probes



https://www.youtube.com/watch?v=VE-raM5o_Yc

Available BRUKER MAS rotor diameter ranging from 7 mm down to 0.7 mm, providing MAS frequencies as high as 111 kHz.

HOW TO LOAD NMR SAMPLE INTO THE MAGNET !

Extreme care
when step up and/or down
(backward only) to change your
sample !

Open Topspin Software, type **ej**, it will
release a strong compressed air at the
top opening of the magnet.



UM Setup II. Basic Optimization Quick

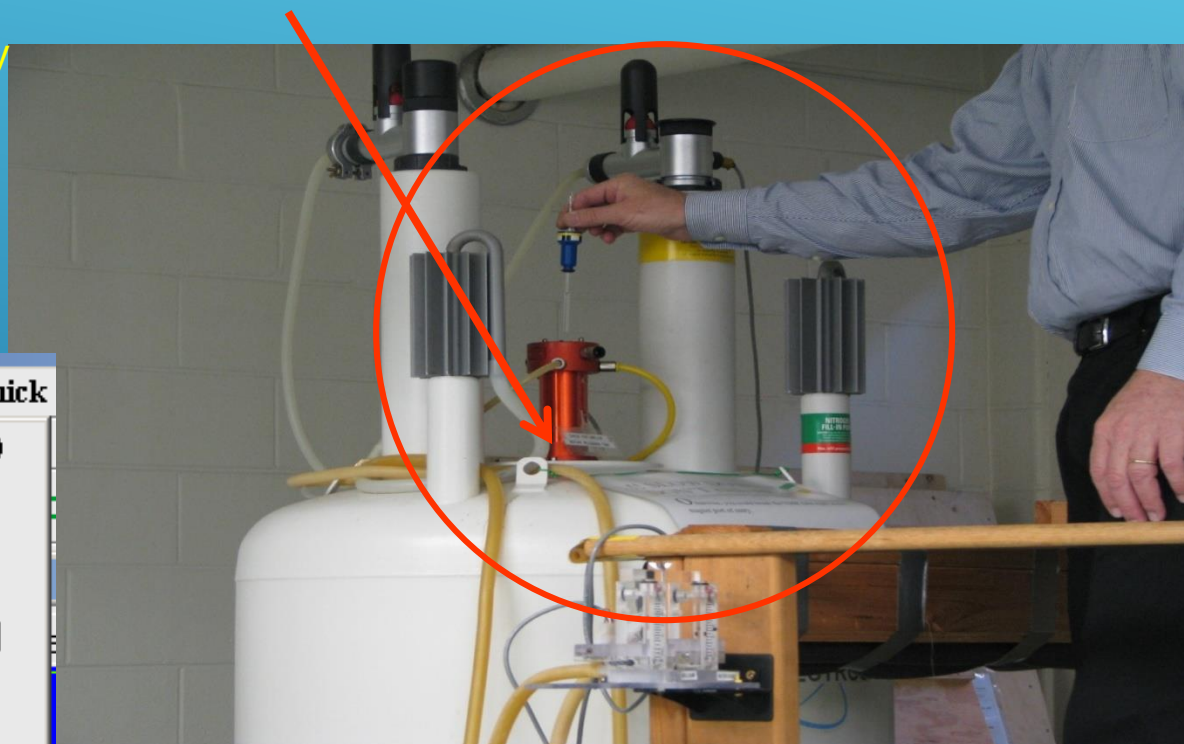
- Check Temperature (advanced only)
- sample up [ej]**
- sample down [ij]**
- create data set [new]
- display all UM applications [rpar A*]
- **select application Ac* only
- **select application Ah* only
- ** select only 2D* [rpar A2d*]
- Load hardware [getprosol]

1. Take the standby dummy sample & spinner out (for basic NMR 400 only) .
2. Carefully place your sample & spinner into the opening.

Before releasing the NMR tube & spinner, ensure there is enough air pressure at the insertion port of the magnet!

3. Type **ij**, sample will slowly transfer into the magnet.

```
UM Setup  II. Basic Optimization  Quick
Check Temperature (advanced only)
sample up [ej]
sample down [ij]
create data set [new]
display all UM applications [rpar A*]
**select application Ac* only
**select application Ah* only
** select only 2D* [rpar A2d*]
Load hardware [getprosol]
```





NMR sample and spinner

Shim stacks

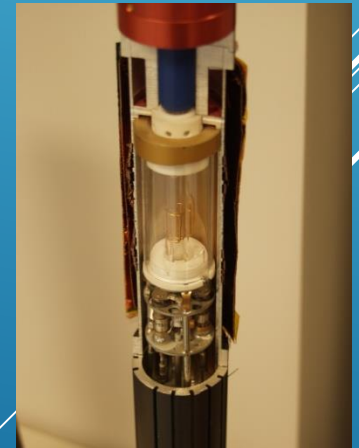
~ 3 feet

Magnet

A free fall without air support will break a NMR sample !



Probe cross-cut enlarged



Log on

Lockout --- Version 1.3-AMX500
Written by Steven G. Smith
UMCP Department of Chemistry & Biochemistry

User Functions	09/29/120 12:03 PM
<ol style="list-style-type: none">1) Log on to instrument2) Lock instrument3) Unlock instrument4) Log out from instrument5) Return to NMR Menu <p>Press # of item to select</p>	The instrument is not in use.

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User Functions	09/29/120 12:03 PM
<ol style="list-style-type: none">1) Log on to instrument2) Lock instrument3) Unlock instrument4) Log out from instrument5) Return to NMR Menu	The instrument is not in use.

Enter your User ID > staff
Enter your Password > _

Lockout --- Version 1.3-AMX500
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User Functions	09/29/120 12:03 PM
<ol style="list-style-type: none">1) Log on to instrument2) Lock instrument3) Unlock instrument4) Log out from instrument5) Return to NMR Menu	The instrument is not in use.

Press '*' or '-' to adjust the approximate time needed for your experiment
(You can use the number keys to quickly set the hours from 0 to 9.)
Other users cannot log in during this time. Press return when done.
Note that the keyboard lock will not work after this time elapses.
Approximate Experiment Time => 0 Hours 15 Minutes.

Log out

Lockout --- Version 1.3-AMX500
Written by Steven G. Smith
UMCP Department of Chemistry & Biochemistry

User Functions	09/29/120 12:04 PM
<ol style="list-style-type: none">1) Log on to instrument2) Lock instrument3) Unlock instrument4) Log out from instrument5) Return to NMR Menu <p>Press # of item to select</p>	The instrument is in use. User: staff Group: aaa-system The instrument is not locked

Lockout --- Version 1.3-AMX500
Written by Steven G. Smith
UMCP Department of Chemistry & Biochemistry

User Functions	09/29/120 12:04 PM
<ol style="list-style-type: none">1) Log on to instrument2) Lock instrument3) Unlock instrument4) Log out from instrument5) Return to NMR Menu	The instrument is in use. User: staff Group: aaa-system The instrument is not locked

Enter your Password > _

SUMMARY:

SAFETY IN NMR LABORATORY:

- Magnetic** field is **permanent**; exists even when electric power is out.
- Pined or caught between the magnet and any external metal object could be **FATAL!**
- Person with **heart pacer** and/or metal implant cannot get access to NMR due to potential health hazard.
- Keep** all metal objects at least 10 ft away from magnet.
- Keep** all credit cards, watches, keys, cell phones also away from the magnet.
- Admit** students taking this course only.

CONCLUSION OF NMR ORIENTATION

- ▶ Sign your liability weaver in the User registration Log!
 - ▶ Criteria for basic NMR check out:
 - ▶ Proper Shimming (Manually).
 - ▶ Obtain a routine H-1 1D spectrum within ten minutes.
 - ▶ Basic processing (proper use of layouts; list parameters, shifts and integration).
 - ▶ Transfer raw data to NMR workstation.
 - ▶ Dead-line for check out:
 - ▶ Four weeks from date of orientation.
 - ▶ Late penalty: \$100 per user.
 - ▶ Contact me via e-mail: fchen127@umd.edu.
- 