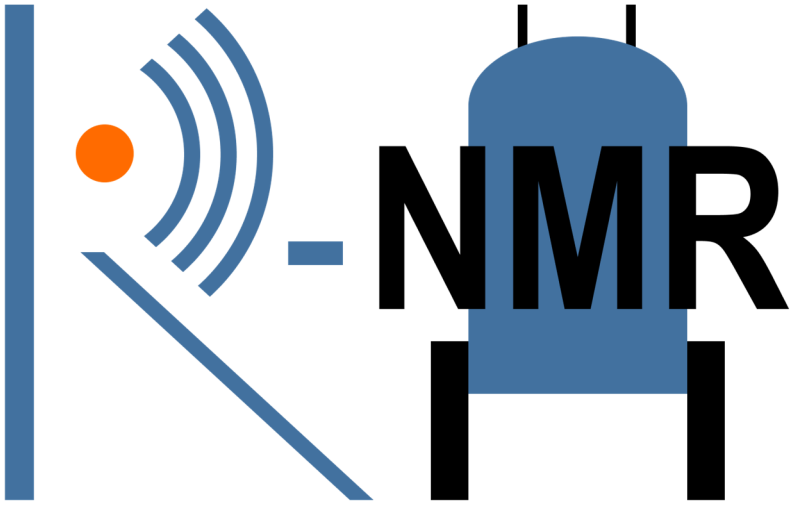


Training Material for remote access to NMR Facilities

Training material for on site facility manager

Provided by Remote-NMR (R-NMR):

Moving NMR infrastructures to remote access capabilities

This project has received funding from the European Union's Horizon Europe research and innovation programme under Grant Agreement N. 101058595

CONTENTS

1. Preamble

General guidelines and standard operating procedures are developed in the course of the R-NMR project. This file does not provide the final solution, but is the state of the discussion as of June 2023.

Remote NMR access has always been an option, but it has gained particular significance during the COVID-19 pandemic when travel is greatly restricted. During this period, the ability to access research facilities remotely is especially valuable.
When users access a research infrastructure, their interaction with the scientific and technical staff for support and guidance is often just as important as the actual instrument time. These interactions contribute to the success of the measurements. Consequently, we are currently developing new methods to maintain productive interpersonal communication through remote access procedures.
User support can encompass various aspects, ranging from providing suggestions and advice during the initial planning stages of measurements to assisting with experimental setup. Additionally, we offer training on specific areas of interest to users.
However, the level of assistance and supervision required varies depending on the expertise of the visiting scientist using the requested technology remotely.

In this file we provide training material for **facility managers covering subjects such as safety, liquid helium refill and cryoprobes** not particularly tethered towards remote NMR.

2. SOP: Safety for area containing High Field Nuclear Magnetic Resonance Spectrometer

1. Entrance is not permitted in such area to person with an implanted pacemaker. Also, a person with implanted magnetic material must not enter the NMR areas because magnetic materials in the body might move upon exposure to the large magnetic fields.
2. Carrying instrumentation, devices, cylinders containing compressed gas, PCs, electrical equipment of any kind or other items containing ferromagnetic material or other

materials that can be attracted by the large magnetic fields in the NMR premises is not permitted.

3. Moving close to such magnetic fields with credit cards, ATM cards and similar cards erases these cards. Moreover, watches might be destroyed inside the 5 Gauss line.

4. It is forbidden the usage and exposure of any magnetic materials, like for instance a staple, a screwdriver and other small tools in the NMR room.

5. Very seldom a fast magnet quench does occur. A fast magnet quench is seen by a large plume of gas coming out of the top of the magnet. Evacuate the room immediately because the liquid is extremely cold, and the individuals present can sustain frostbites and will not be able to take up sufficient oxygen and must leave immediately to avoid suffocation. The NMR personnel must be informed immediately.

6. No documentation indicates harmful effects upon fetus or mother in pregnancy from the permanent magnetic fields around NMR magnets. Moreover, no harmful effects from the radio signals involved in NMR spectroscopic experimentation have mentioned.

7. The magnet contains large amounts of liquid helium and nitrogen. These are cryogens and serve to keep the magnet core at a very low temperature. Because of the very low temperatures involved, gloves, a long-sleeved shirt or lab coat and safety goggles should always be warned when handling cryogens (i.e. during He/N refills). Do not attempt to refill the magnet with helium or nitrogen unless you have been trained in the correct procedure

3. SOP: Turn On/Off Cryo Platform and Console

The cryoprobe, when existed, is a very important part of the NMR equipment, it operates at extremely low temperature and it considerably enhance the sensitivity by ~5 times compared with a typical room temperature probe. The cryoprobe should always be connected with a nitrogen gas tank to work properly and a special procedure should be followed to warm up and cool down the probe. Moreover, the spectrometer, which is consisting of many different parts that are controlling different procedures during a measurement, is the core of the entire NMR acquisition.

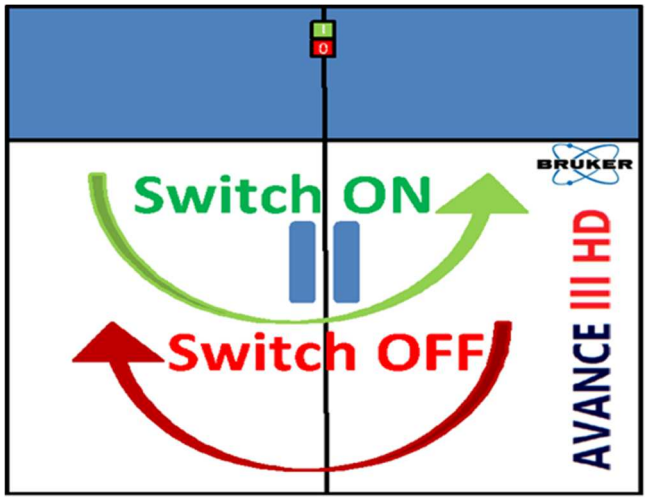
Turn on spectrometer Console:

1. Switch-on air compressor at the basement of the Pharmacy department to provide nitrogen supply to all the systems. Compressor typically takes some minutes to reach the working procure of 6-8 bar.
2. Turn-on the Bruker Cooling Window (BCU), which is behind the magnet.
3. Turn-on the power supply to the console, by pressing the green button with the symbol I written on it.
4. Switch-on the workstation PC, which is connected to the spectrometer.
5. Switch-on AQS/3+ board inside the console.
6. Switch-on the IPSO board inside the console.
7. Switch on the BSMS board inside the console.
8. Switch-on the 2 amplifiers are found at the left side of the console.
9. Check Cryoprobe. Must be WARM!
10. Open the Topspin software at the PC and type edte. A temperature control window opens and the BCU should be turned-on by pressing the ‘‘On’’ button at the top of the window.
11. A COOL DOWN of the probe must be done before a measurement acquired.
12. If the probe is COLD (check cryoplatform) open a new dataset and type ii (or ii restart) to be sure that the spectrometer is functioning correctly.

Turn off NMR spectrometer Console:

1. Take out the sample of the magnet.
2. Check Cryoprobe. Must be WARM! If not, a WARM UP of the probe must be done
3. Switch-off Cryo-platform by pressing the on/off button for few seconds.
4. Open the Topspin software at the PC and type edte. A temperature control window opens and the BCU should be turned-off by pressing the ‘‘Off’’ button at the top of the window.
5. Switch-off the 2 Amplifiers are found at the left side of the console.
6. Switch-off BSMS board inside the console.
7. Switch-off AQS/3+ board inside the console.
8. Turn-off the power supply to the console, by pressing the red button with the symbol 0 written on it.
9. Switch-off Workstation PC which is connected to the spectrometer.
10. Turn-off the Bruker Cooling Window (BCU), which is behind the magnet.
11. Switch-off Compressor at the basement of the Pharmacy department.

Important: Even if the spectrometer is turned-off the magnet is operating normally, so it is important to check the liquid Helium and Nitrogen level very often, because it is important for the magnet material to functioning properly and to avoid the destruction of the magnet. For this reason, the console must be turned on to check frequently the levels of the cryogen inside the magnet. If the level of any of them go lower than 50% a refill must be done immediately



Warm up the Cryoprobe of the spectrometer:

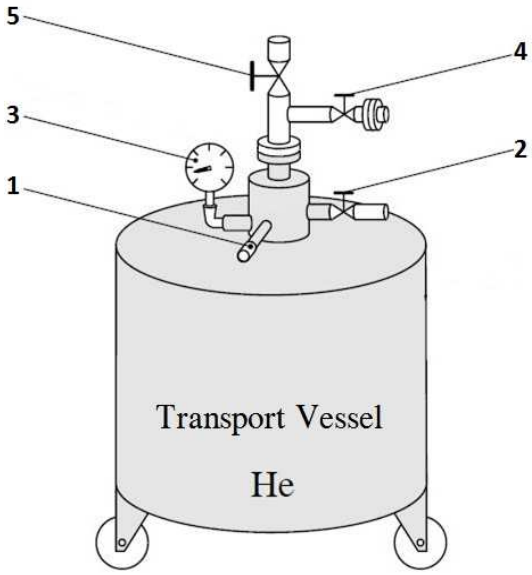
1. Check the cryoplatform Cold green LED must be on.
2. Take out the sample of the magnet.
3. Press the warmup white button on the platform.
4. Sometimes the emergency lift opens and the flow rate inside the probe increases. Ask for a NMR engineer to turn it off.
5. Wait about 3 hours for the warmup to finish. When the Green Warm LED lights on, the probe is warm.
6. Press the Unplug white button to clear the errors from the platform.
7. Check the CryoTool notification. A ‘‘System is warm’’ message must be written.
8. Let the cryoplatform to work for at least 8 hours before you turn it off.
9. Press the On/Off button to close the Cryoplatform.

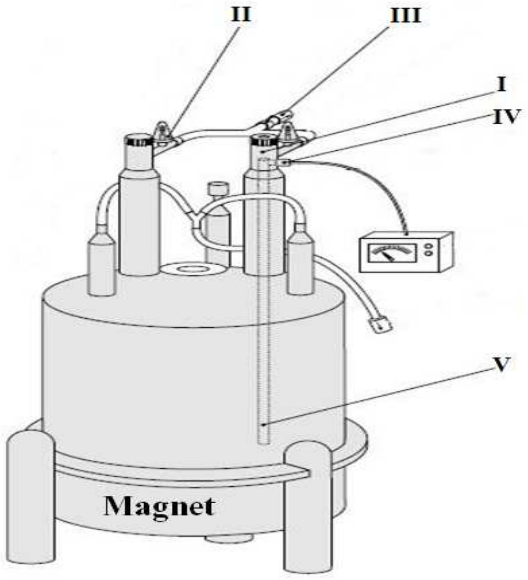
Cool down the Cryoprobe of the spectrometer:

1. Press the On/Off button to open the Cryoplatform.
2. Let the cryoplatform to work for at least 3 hours before you start the cool down.

3. Check Helium bottle and the air compressor that are working properly.
4. Check the cryoplatform Warm green LED must be on.
5. Press the Cool down white button on the platform.
6. Wait about 4 hours for the cool down to finish. When the Cold green LED lights on, the probe is cold.
7. Check the CryoTool notification. A ‘‘System is Cold’’ message must be written.
8. Open a dataset that is containing all the channels and type ii.
9. Insert a sample inside lock and match and tune the sample.
10. Sometimes Deuterium channel needs also matching and tuning.
11. Shim the sample using TopShim software.
12. If the Error LED is on press the Unplug white button and wait for 15 minutes before do any measurement.

4. SOP: Liquid and Helium Refill





1. Security Valve
2. Pressure release Valve
3. Manometer
4. Pressure release/increase Valve
5. Liquid He outlet

I. Right liquid He import with sifone
II. Quench Valve
III. One-Way Valve
IV. Liquid He Sensor
V. Sifone

Check:

1. Close the He regulator and the N2 liquefier some hours before the refill
2. Check the percentage of liquid Helium inside the magnet at the BSMS control suite.
3. Calculate the amount of liquid He needed to refill at 100%.
4. Measure the amount of He in the transport vessel with the dipstick.

Preparation:

1. Open the pressure release valve 2 to decrease the pressure in transport Vessel.

2. Connect the clamp to the entrance 5 and pressure release/increase valve 4.
3. Close all the valves at the He transport vessel (2,4,5).
4. Regulate the gas flow of He gas dewar at low pressure 0,1-0,2 bar.
5. Connect all the parts at the entrances 4 and 5 of the transport vessel without opening the valves.
6. Attach the long side of the transfer line in the outlet of liquid He 5 and tighten a bit the 'O' ring to hold it at this position without opening the valve 5.
7. Untighten but not disconnect the screw at the right side of the magnet I.
8. Open liquid He outlet 5 valve and slowly push down the long side of the transfer line inside the transport vessel until you hear and see stable He gas flow coming from the short side of the transfer line.
9. Tighten 'O' ring at Liquid He outlet 5 to stabilize the transfer line.

IMPORTANT 1: In case ice appears on the transfer line and/or no He gas comes from the short side of the transfer line, He refill should immediately stop until the vacuum inside the transfer line improves or the transfer line changes!!!

10. After some minutes instead of He gas, liquid He will start coming out from the short side like a flame. Only at that time you are allowed to put the short side of the transfer line inside the magnet to start the He transfer.

IMPORTANT 2: In case flame does not appear in the short side of the transfer line, but side is inserted into the magnet, a QUENCH may become!!!

11. If liquid He flow at the short side of transfer line is huge, open the pressure release valve 2 to decrease it, but do not stop it completely. FLAME SHOULD APPEAR!!!

Transfer of liquid He:

1. Open immediately the right liquid He import I of the magnet and put the short side of the transfer line into the siphon V in less than 5 seconds and tighten it to the middle of the siphon. Wait until the starting pressure inside the magnet decreases a little bit and push the transfer line into the magnet until it is almost all inside. Pull 1 cm backwards and tighten well to stabilize it in this position.

- Optionally: At the time you put the short side of the transfer line into the magnet

someone could remove the one-way valve III to release the starting pressure inside the magnet faster and safer. At this occasion you should have a constant He gas flow rate or liquid He during the time valve is out. If the gas flow stops at the outlet the one-way valve III should be reconnected in less than 5 seconds!

2. Push the long side of the transfer line into the transport vessel slowly to the end and pull it 5cm up. Tighten well the transfer line to the liquid He outlet 5 of the transport vessel.
3. The liquid He transfer starts. Constant He gas rate should be coming out of the one-way valve III.
4. Whenever needed you may open the pressure increase valve 4 to build pressure inside the transport vessel and push liquid He into the magnet.

IMPORTANT 3: At any time the pressure inside the transport vessel should not exceed 0,3 bar!!!!!

5. Measure the He level from the BSMS control suite. Normally, after a few minutes the indication should steadily increase. If this is does not happen, immediately STOP the He transfer.
6. When the He level reaches 100%, wait a few minutes and then the He flow rate at the one-way valve outlet will dramatically increase and probably a small amount of liquid He will appear like flame.

IMPORTANT 4: In case the pressure of the transport vessel decreases, even though dewar provides He gas, this is an indication that liquid He is almost finishing in the vessel. Moreover the transfer line will generate a characteristic sound. At this point the He transfer must stop, even if the indication of He level in the magnet is not 100%.

7. Open the pressure release valve 2 and close the pressure increase valve 4. Detach and close the He gas flow rate from the dewar. Then, open the pressure decrease valve 4 to neglect the pressure inside the vessel.
8. Untighten the 2 sides of transfer line. Use the heat gun whenever needed.
9. Whenever possible, unscrew completely the ‘O’ ring at the right import of the magnet I and pull out completely the short side of transfer line from the magnet. In less than 5 seconds the right import of the magnet I should close with any possible way.

- Optionally: Additionally at these 5 seconds the one-way valve

III should be putted on if it was removed.

10. Tighten well the 4 parts at the right import of the magnet I with the correct order as indicated in the picture.

IMPORTANT 5: Wipe all the remaining water or humidity that appears during the next hour at the top of the magnet, to prevent their entrance into the He deposit of the magnet.

IMPORTANT 6: During and after the He refill, ice should appear at all the pipes connected with the He deposit, located at the top of the magnet. If this did not happen, probably a frost block is somewhere inside the pipes. Contact a Bruker engineer to assist you immediately and stop the He transfer.

11. The liquid He transfer is done. Detach all the remaining connections of the transfer line at the transport vessel and store the transfer line in a safe place till it is completely dry.

IMPORTANT 7: During the He transfer the transfer line should never be inflected, otherwise it will be destroyed.

Last Check of the Systems:

1. Check if all the parts of the magnets are closed.
2. Check if Quench Valves II are at their positions.
3. Check the He level of the magnet with BSMS Control Suite.
4. Fill the He transfer log file of the program MICS.
5. Ensure that the frozen parts of the magnet will start unfreeze and wipe frequently the water.
6. Measure with the deep stick the remaining liquid He in the transport vessel.
7. Acquire a 1D-1H measurement to check that the system is normally working.
8. At least 1 hour after the He refill has finished, reconnect and turn on the He regulator at the one-way valve III.
9. At least 1-2 hours after the He refill has finished, you may turn on the N2 liquefier from the Cryo Tool program from the laptop.

Next Days:

1. Check frequently the rate that the liquid N2 evaporates from the system. It should be stable if the liquefier is on for many days and when it is off, N2 should evaporate about 3% per day. If the evaporation of N2 changes after some days, contact a Bruker engineer.
2. Check daily the liquid He evaporation rate. After some days it should be back to normal (~0.3%/day).