Refilling Procedure

User Manual

Version 07

Innovation with Integrity

NMR

This manual was written by

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Faellanden, Switzerland

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For further technical assistance on the NMR Magnet System, please do not hesitate to contact your nearest BRUKER dealer or contact us directly at:

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Please refer to the model No., serial No. and internal order No. in all correspondence regarding the NMR system or components thereof.

Contact

1 Introduction

1.1 General Information

This manual contains important information about the refilling procedure on magnet systems used for NMR analyses. The compliance with all safety and handling instructions, the applicable local accident prevention and general safety regulations are necessary for safe work.

This manual is part of the technical documentation of the magnet system. It must be kept nearby the magnet system and free access must be ensured at any time. Read the manual carefully before handling the magnet system or its components.

1.2 Limitation of Liability

The information in this manual will take into account the current state of the technology.

The manufacturer assumes no liability for damages resulting from:

- non-compliance with the instructions and all applicable documentation,
- use for purposes not intended,
- not sufficiently approved persons,
- arbitrary changes or modifications and
- use of unauthorized spare parts or accessories.

1.3 Customer Service

Technical support is provided by Bruker Service via telephone or e-mail. For contact information see page 5 of this document.

1.4 Warranty

The warranty terms can be found in the sales documents of the magnet system and in the Terms and Conditions of Bruker Corporation.

1.5 Copyright

1.6 General View

1.6.1 Refilling Nitrogen

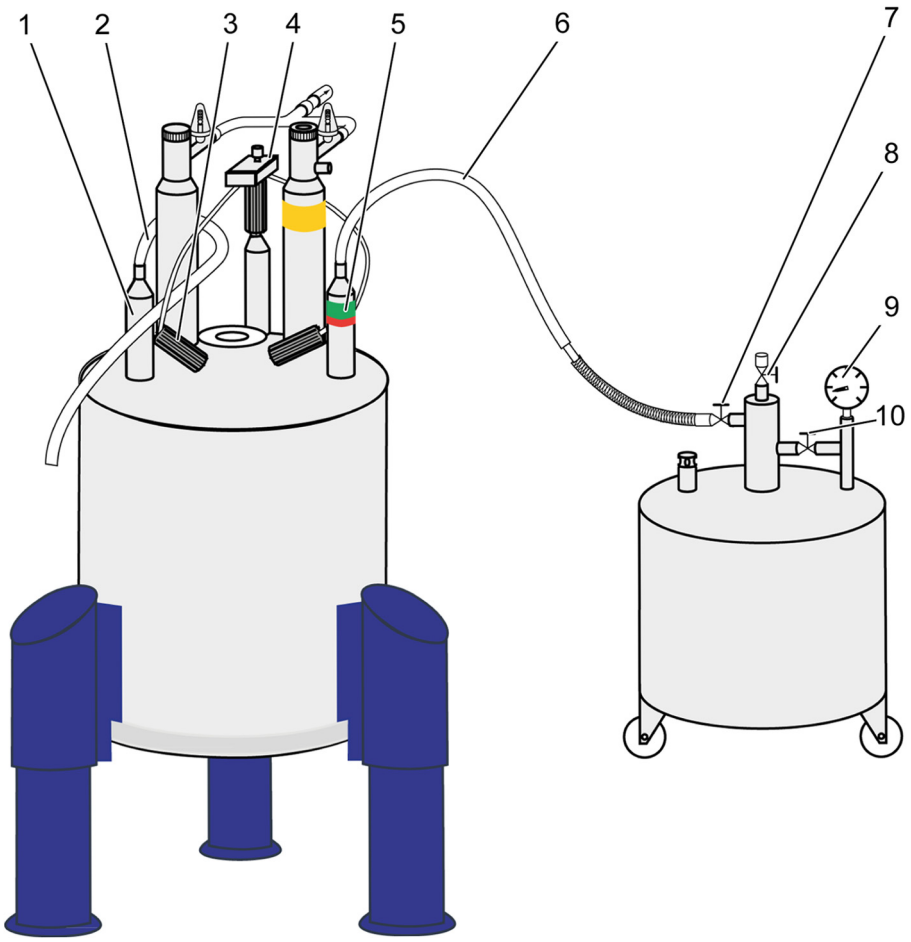


Figure 1.1: Setup of the magnet system during the nitrogen refilling procedure

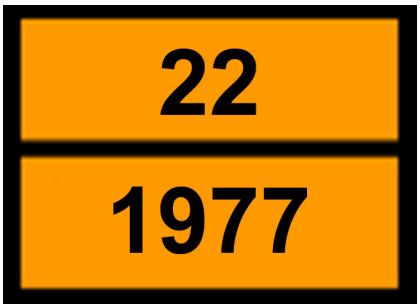
1 Nitrogen turrets (three or four turrets possible)
2 Teflon® or PFA hose used as exhaust during refilling
3 Nitrogen flow system with heat exchangers
4 Nitrogen Level Sensor
5 Nitrogen fill-in turret
6 Transfer hose
7 Liquid nitrogen extraction tap
8 Pressure release valve
9 Pressure gauge
10 Pressure regulator valve

The nitrogen fill-in turret on the magnet system is marked with a green and red colored label.

Figure 1.2: Label of the nitrogen fill-in turret

Physical Properties of Nitrogen

Liquid nitrogen is boiling at a temperature of –196 °C. Nitrogen is colorless, odorless and not inflammable. Refer to “Cryogenic Agents“ on page 23 for further safety advice.



For storage and transportation of liquid nitrogen refer to the list of hazardous substances and the hazard class data sheet.

Figure 1.3: Nitrogen hazard identification number and UN number

1.6.2 Refilling Helium

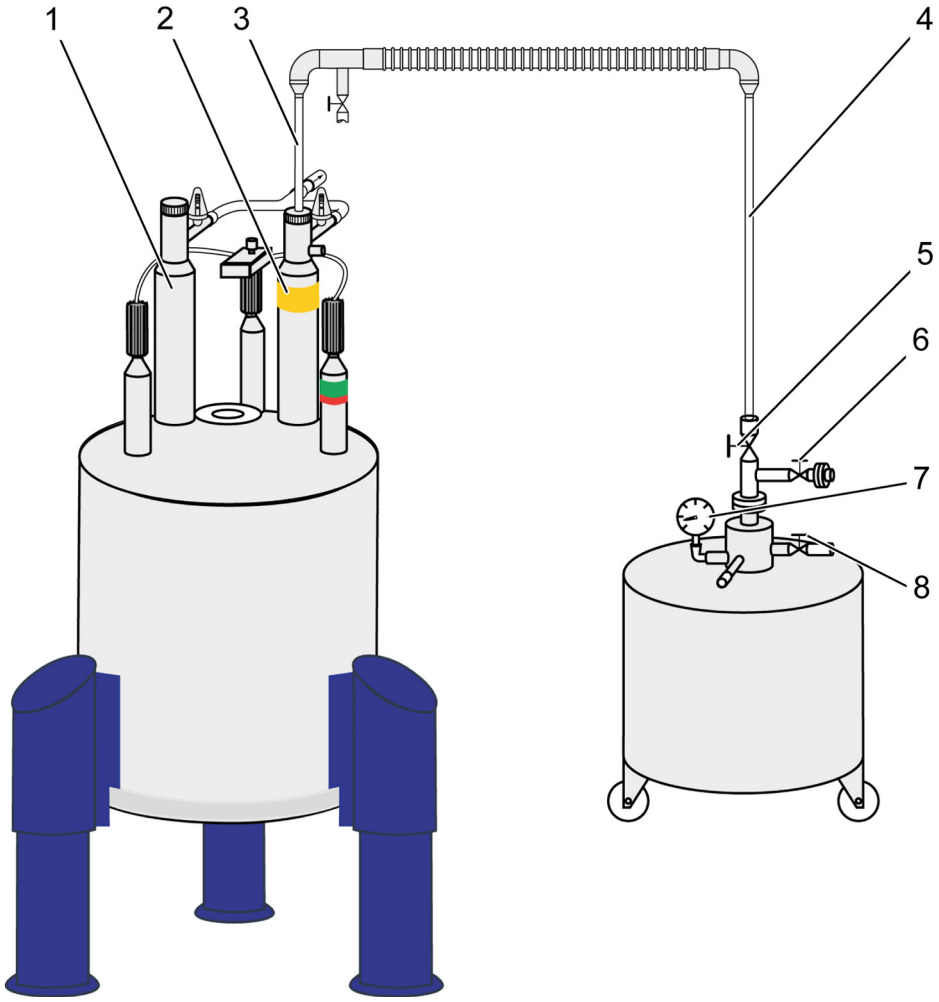


Figure 1.4: Setup of the magnet system during helium refilling procedure

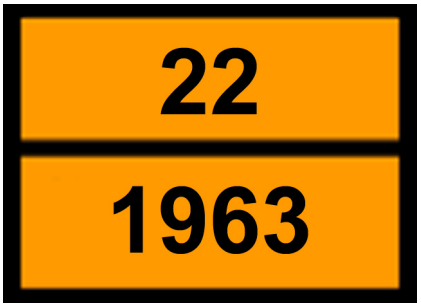
1 Helium turrets (two or three turrets possible)
2 Helium fill-in turret with Helium Level Sensor
3 Transfer line connector tube – magnet system side
4 Transfer line connector tube – transportation dewar side
5 Extraction tap for liquid helium (globe valve)
6 Pressure regulator valve
7 Pressure gauge
8 Pressure release valve

The helium fill-in turret is marked on the magnet system with a yellow label.

Figure 1.5: Label of the helium fill-in turret

Physical Properties of Helium

Liquid helium is boiling at a temperature of –269 °C. Helium is colorless, odorless and not inflammable. Refer to “Cryogenic Agents“ on page 23 for further safety advice.



For storage and transportation of liquid helium refer to the list of hazardous substances and the hazard class data sheet.

Figure 1.6: Helium hazard identification number and UN number

Introduction

2 Safety

During the refilling procedure it is necessary to enter the marked danger area of the magnet system. In order to eliminate the danger associated with the refilling procedure the customer must obey the rules for safety and accident prevention given in this chapter.

2.1 Approved Persons

Bruker BioSpin AG identifies the following qualifications for personnel performing tasks on the magnet system or its components:

Approved Customer Personnel:

As a result of professional training by Bruker Service Personnel, experience and knowledge of applicable regulations these persons are qualified to perform the refilling procedure described in this manual. Approved Customer Personnel are qualified to identify possible hazards and risks associated with the tasks assigned to them and to perform all possible steps to eliminate or minimize these risks.

Bruker Service Personnel:

These persons are qualified by appropriate qualification and professional training and experience (including all necessary knowledge of applicable regulations and regulatory requirements) to perform specific tasks on the magnet system and its components. Bruker Service Personnel are qualified to identify possible hazards and risks and to perform all possible steps to eliminate or minimize these risks.

2.2 Customer Responsibilities

The customer must obey the security advice and the rules for safety, applicable local accident prevention and environmental protection correctly for the refilling procedure, recommended equipment and for the magnet system. Furthermore, the customer is responsible for keeping the refilling equipment in good technical condition.

In particular:

- The customer must determine additional dangers resulting from the working conditions at the site of the magnet system and provide applicable safety measures.
- The customer must ensure that the site plan meets the specified conditions for operating and refilling the magnet system.
- The customer must check the marked danger area around the magnet system and the corresponding instruction plates before refilling.
- The customer has to ensure the intended use of the refilling procedure and the recommended equipment.
- The customer must clearly define the responsibilities for refilling the magnet system.
- The customer has to organize appropriate training to approve personnel for refilling the magnet system.
- The customer must ensure that all employees approved for performing the refilling procedure have read and understood the manual.
- The customer has to provide the necessary personal protective equipment for all employees approved for performing the refilling procedure.
- The customer has to instruct his employees at regular intervals on hazards and safety measures.
- The customer has to instruct other persons not approved for performing the refilling procedure but carrying out work in the same room, for instance cleaning staff or guards about the possible danger at the site of the magnet system.

2.3 Key Words

DANGER

Indicates a hazardous situation which, if not prevented, will result in death or serious injury.

WARNING

Indicates a hazardous situation which, if not prevented, could result in death or serious injury.

CAUTION

Indicates a hazardous situation which, if not prevented, may result in moderate or minor injury.

NOTICE

Hazard, which could result in property damage.

Information and links for efficient and trouble-free handling and operation.

2.4 Residual Risks

In the following chapter, the residual risks from the risk analysis according to ISO 14971 are summarized. To prevent health hazards and hazardous situations obey all safety instructions and warnings in the manual.

2.4.1 Persons

⚠ WARNING

Risk of injury and property damage due to handling by not approved persons.

Incorrect refilling or incorrect handling of the magnet system by not approved persons may result in significant bodily injury and property damage.

Thus:

- Work must only be carried out by approved persons with applicable qualifications.
- In case of doubt, contact Bruker Service. Contact information see page 9 of this document.

2.4.2 Intended Use

⚠ WARNING

Risk of damage to life and limb by incorrect refilling of the magnet system.

Incorrect refilling of the magnet system can lead to life-threatening situations and destruction of the magnet system.

Thus:

- Only refill the magnet system according the described refilling procedure.
- Do not change the magnet system or recommended equipment for refilling.
- Do not exceed specified values for refilling the magnet system.

Damage claims from damages caused by other than the intended way of the refilling procedure are excluded and the customer is held liable.

2.4.3 Safety Devices

⚠ WARNING

Risk of damage to life and limb due to not sufficient safety devices.

Several safety devices ensure safe operation of the magnet system. They must always be in correct working condition.

Thus:

- Do not block safety devices.
- Do not remove safety devices.
- Check the operational reliability of the safety devices of the magnet system before the refilling procedure.
- Check the operational reliability of the safety devices of the transportation dewar before the refilling procedure.

2.4.4 Spare Parts

⚠ WARNING

Risk of injury and property damage from using incorrect or defective spare parts and accessories.

Incorrect or defective spare parts can cause serious injuries. They may cause damaging, malfunctioning and the destruction of the magnet system.

Thus:

- Only use original equipment manufacturer spare parts.
- Only use original equipment manufacturer accessories.

2.4.5 Signs and Labels

⚠ WARNING

Risk of damage to persons and property due to not readable signs and labels.

Signs and labels with advice may become not readable.

Thus:

- Maintain signs and labels in a readable state.
- Replace damaged or not readable signs and labels immediately. New signs and labels can be ordered from Bruker Service.

2.4.6 Technical Risks

Magnetic Field

⚠ WARNING

Risk of damage to life and limb due to high magnetic fields.

A magnetic field of more than 0.5 mT (5 Gauss) is life-threatening for people with pacemakers or metal implants. Exposure to more than 8 T or duration of exposure (8 h/day) above the limit of 200 mT can cause damage to health. Ferromagnetic tools in the magnetic field are significantly hazardous. Disks and electronic devices may be damaged.

Thus:

- Check the marked magnetic field of more than 0.5 mT (5 Gauss) before start up.
- The permanent workplace must be outside the 0.5 mT area.
- Do not stay or work at magnetic fields of more than 8 T.
- Prevent exposure of more than 200 mT for more than 8 h/day.
- Keep people with pacemakers and metal implants away from the identified area.
- Keep disks, credit cards and electronic devices away from the identified area.
- Do not use ferromagnetic tools or items within the identified area.
- Only use non-ferromagnetic transportation dewars or pressure cylinders for the cryogenic agents.
- Only use non-ferromagnetic ladders or steps.

Cryogenic Agents

⚠ WARNING

Risk of damage to life and limb due to cryogenic agents.

Risk of damage to life and limb due to not correct handling of liquid cryogenic agents and its transportation dewars. Within the transition from liquid to gas, helium and nitrogen expand their volume, causing closed vessels to burst.

The evaporating cryogenic agents will displace the air. In case of not sufficient ventilation this may result in death by suffocation. Helium displaces oxygen in the upper part of the room, nitrogen displaces oxygen in the lower parts of the room.

Liquid and gaseous cryogenic agents are extremely cold. Contact with liquid or gaseous cryogenic agents will lead to cold burns. Contact with the eyes may cause blindness. Refer to Warning: Low Temperature on page 21.

Thus:

- Only use cryogenic agents in well ventilated rooms. In case of doubt ask Bruker Service.
- Wear an oxygen monitor on the body during service and maintenance work.
- Prevent any skin contact with liquid or gaseous cryogenic agents.

Electricity

⚠ WARNING

Risk of damage to life and limb due to electricity.

Risk of damage to life and limb due to contact with electrical lines and damaged insulation.

Thus:

- Work on electrical equipment must be done by an approved electrical technician.
- Keep moisture away from electrical lines to prevent short-circuits.
- Check the magnet system electrical grounding before start.

Quench

⚠ WARNING

Risk of suffocation during a quench of the magnet system.

A quench is the very fast de-energizing of the magnet by loss of its superconductivity. The stored magnetic energy is converted into heat and thus large quantities of helium evaporate. The evaporating helium will displace oxygen. In case of not sufficient ventilation this may result in death by suffocation.

Thus

- The magnet system site must be well ventilated. In case of doubt contact Bruker Service.
- The evaporating gas may resemble smoke. Never pour water on the magnet system.

Gas under Pressure

⚠ WARNING

Risk of injury due to gas under pressure inside the cryostat and further equipment.

The helium or the nitrogen vessel of the cryostat may get sealed off due to ice formation inside the helium or the nitrogen turrets in case of non-compliance with the instruction given in this manual. This may lead to overpressure and damage of the helium or the nitrogen vessel.

Manipulations of components with gas under pressure may lead to injury and property damage.

Thus:

- In case of icing inside the helium or the nitrogen turrets contact Bruker Service immediately.
- Release the pressure to the recommended value before working on components with gases under pressure.
- Do not seal cryogenic agent vessels of the magnet system or the transportation dewars.
- Do not connect high pressure transportation dewars to the magnet system. Completely eliminate the high pressure from the transportation dewars before connecting and transferring cryogenic agents.
- Always keep control of the maximum pressure during the nitrogen refilling procedure and during the helium refilling procedure.
- Do not exceed the maximum pressure of 0.35 bar in the nitrogen transportation dewar or 0.1 bar in the helium transportation dewar during the refilling procedure.

Low Temperatures

⚠ WARNING

Risk of injury due to low temperatures of liquids and metal parts.

Physical contact with extremely cold liquids and metal parts may cause serious injuries. Contact with the skin may cause cold burns. Contact with the eyes may cause blindness.

Thus:

- Always wear protective goggles, protective gloves and protective clothes while handling with liquid cryogenic agents or metal parts in contact with liquid cryogenic agents.
- Only use specified hoses made of PFA (e.g. Teflon® PFA) for refilling.
- Protect temperature sensitive components such as O-rings from contact with liquid cryogenic agents.

Spontaneous Ignition and Explosion

⚠ WARNING

Risk of injury from spontaneous ignition and explosion caused by liquid oxygen.

Pure oxygen condenses on extremely cold metal pieces. Together with oil it may ignite spontaneously. In case of fire the pure oxygen may cause an explosion.

Thus:

- Do not smoke near the magnet system.
- Do not use open flames near the magnet system.
- Keep the environment around the magnet system clean.
- Do not leave oily rags near the magnet system.

Risk of Slippage

⚠ WARNING

Risk of injury from slippage.

The accumulation of condensed water on the floor and ladders causes slippery surfaces.

Thus:

- Always wear safety shoes with an anti-slip sole.
- Be careful using ladders.
- Clean floor and ladders regularly.

2.5 Personal Protective Equipment

The personal protective equipment must be worn at any time while working on the magnet system and to prevent health hazards.

Protective Goggles

Used to protect the eyes from injury due to flying cold liquids and parts.

Protective Gloves

Used to protect the hands from injury caused by contact with extremely cold liquids or surfaces and for protection from injury caused by rough edges.

Protective Clothes

Used to protect the body from injury caused by contact with extremely cold liquids or surfaces and for protection from wounds.

Safety Shoes

Used to protect the feet from injury from falling of heavy objects. An anti-slip sole protects from injury caused by slipping and falling on slippery floor and steps. Only use safety shoes with non-ferromagnetic toe caps.

Portable Oxygen Monitor and Alarm

Used to warn against low oxygen concentrations in surrounding air.

2.6 Description of Signs and Labels

Signs and labels are always related to their immediately vicinity. The following signs and labels are found on the magnet system and in the vicinity.

Prohibition sign: No person with pacemakers!

People with pacemakers are endangered in the identified area of 0.5 mT (5 Gauss) and are not allowed to enter these areas.

Prohibition sign: No person with implants!

People with metallic implants are endangered in the identified area of 0.5 mT (5 Gauss) and are not allowed to enter these areas.

Prohibition sign: No watches or electronic devices!

Watches and electronic devices may be damaged in the identified area of 0.5 mT (5 Gauss).

Prohibition sign: No credit cards or other magnetic memory!

Credit cards and magnetic memory may be damaged in the identified area of 0.5 mT (5 Gauss).

Prohibition sign: Do not touch! Do not block!

Do not touch or block identified area.

Hazard warning sign: Strong magnetic field!

- No magnetic memory.
- No jewelry.
- No metallic items.

Emergency exit!

- Always keep the emergency exit clear.
- Follow the arrows if necessary.
- Doors must pushed open in escape direction.

2.7 Safety Devices

For detailed information on the safety devices of the cryostat refer to chapter “Safety Devices” in the supplied User Manual of the magnet system.

2.8 Behavior in Danger and Emergency Situations

Preparations

- Keep the emergency exits free at all times.
- Check the list of emergency telephone numbers in the magnet system area.

In Case of Emergency

- Leave the danger zone immediately.
- Check for sufficient ventilation in the room before entering, especially if people are showing symptoms of suffocation.
- Rescue persons from the danger zone.
- Provide medical attention for people with symptoms of suffocation.
- Start first aid immediately.
- Call the responsible contact.
- Call for medical assistance.
- Call the fire department.

First Aid for Cold Burns

- Help the injured persons to lie down comfortably in a warm room.
- Loosen all clothing which could prevent blood circulation in the injured area.
- Pour large quantities of warm water over the affected parts.
- Cover the wound with dry and sterile gauze.
- In case of contact of liquid cryogenic agents with the eyes rinse thoroughly with clean water.
- Call for medical assistance.

2.9 Fire Department Notification

For information on the fire department notification refer to the User Manual of the magnet system.

3 Refilling Nitrogen

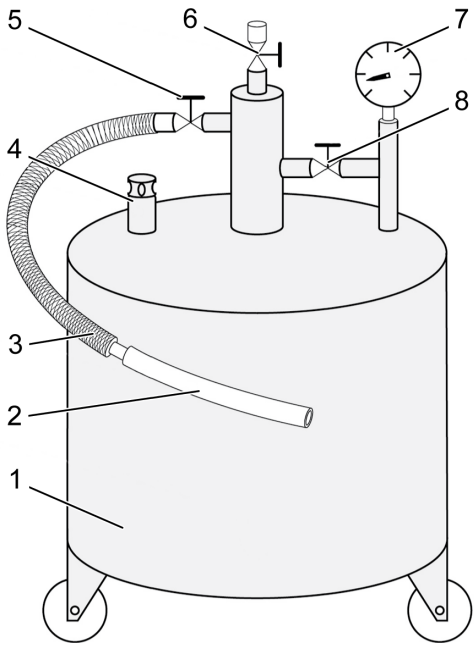
3.1 Introduction

Illustrations in the following chapters are intended for basic understanding while handling cryogenic agents during the refilling procedure. Some illustrations of transportation dewars and accessories given in this manual may differ from the actual design. However, this does not affect the safety advice given in chapter "Safety" nor the function.

3.1.1 Nitrogen Transportation Dewars

The nitrogen transportation dewars contain liquid nitrogen at a temperature of – 196 °C. Approved personnel handling with cryogenic agents must obey the security advice and the rules for safety and accident prevention (see chapter "Safety").

A transportation dewar for liquid nitrogen consists of the following main components:



1 Transportation dewar

2 Teflon® transfer hose or PFA hose

3 Transfer hose

4 Safety valve and vacuum connection flange

5 Liquid nitrogen extraction tap

6 Pressure release valve

7 Pressure gauge

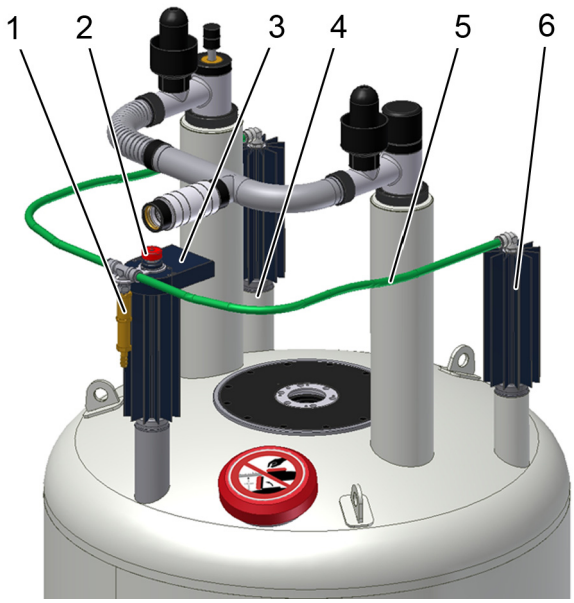
8 Pressure regulator valve

Figure 3.1: Transportation dewar for liquid nitrogen

A complete **Nitrogen Refilling Set** together with PFA hose and the necessary adaptors is available from Bruker BioSpin AG as part number Z53144.

3.1.2 Nitrogen Flow System

This chapter describes the components that are common to all superconducting magnet systems cooled with cryogenic agents.



1 One-way valve of the nitrogen vessel

2 Safety valve of the nitrogen vessel

3 Nitrogen Level Sensor

4 Nitrogen fill-in turret

5 Green tube

6 Heat exchangers

Figure 3.2: Nitrogen flow system

Do not remove the Nitrogen Level Sensor for refilling.

3.2 Safety

WARNING

Magnetic field (see page 22)

Low temperature (see page 25)

Cryogenic agents (see page 23)

Gas under pressure (see page 24)

3.3 Fluid Level Measurement

3.3.1 Nitrogen Level Sensor

The magnet system provides a Nitrogen Level Sensor to measure the fluid level. It is connected with the BSMS Service Web, where the measured value can be displayed.

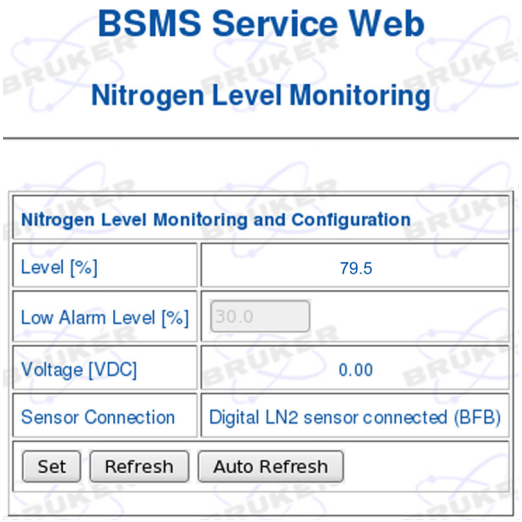
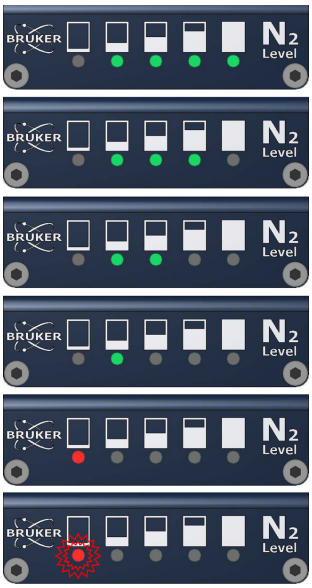


Figure 3.3: User interface of the BSMS Service Web – nitrogen level monitoring

3.3.2 Nitrogen Level Display

The Nitrogen Level Sensor itself provides a display, which shows the current fluid level in the nitrogen vessel by red and green LEDs.



Nitrogen level at:

≥ 90 %

70 % – 90 %

50 % – 70 %

30 % – 50 %

10 % – 30 %

Schedule refilling of nitrogen

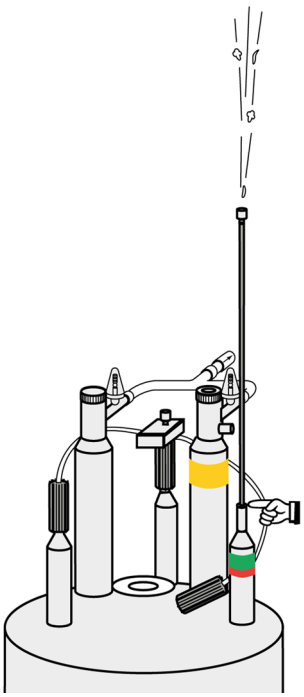
≤ 10 %

Refill immediately

Figure 3.4: Display of the Nitrogen Level Sensor

3.3.3 Dip-Stick

Use the provided dip-stick (Bruker BioSpin AG part number Z27451) to measure the fluid level in the nitrogen vessel following the procedure as described below.

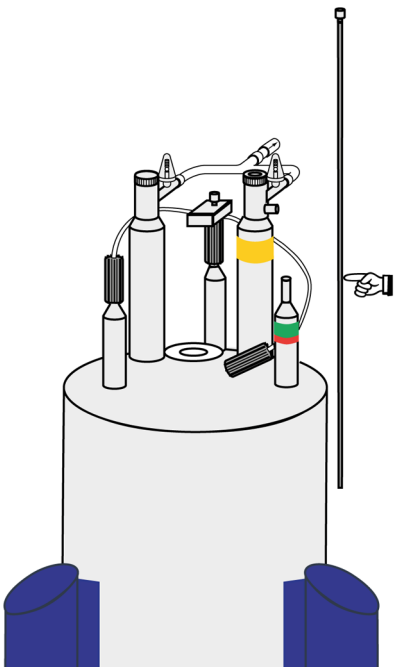


1. Remove the heat exchanger from the nitrogen fill-in turret.

⚠ WARNING:
Low Temperature

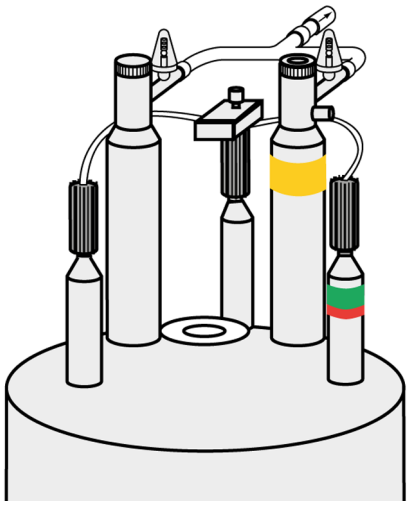
2. Slowly insert the dip-stick into the nitrogen fill-in turret and watch the top of the dip-stick.
3. Stop inserting the dip-stick any further as soon as nitrogen starts to spray out at the top of the dip-stick.
4. Mark this position on the dip-stick at the top of the nitrogen fill-in turret.

Figure 3.5: Fluid level measurement in the nitrogen vessel – step 1



5. Pull out the dip-stick from the nitrogen vessel.
6. Put the dip-stick near the cryostat with the mark on the dip-stick in the same vertical position as the top of the nitrogen fill-in turret. The lower end of the dip-stick shows the liquid nitrogen level in the nitrogen vessel.

Figure 3.6: Fluid level measurement in the nitrogen vessel – step 2



7. Remount the heat exchanger on the nitrogen fill-in turret.
8. Refer to the nitrogen level graph supplied with the User Manual of the magnet system to determine the amount of liquid nitrogen in the nitrogen vessel.
9. Calculate the required volume of liquid nitrogen to refill the nitrogen vessel. Refer to the chapter "Technical Data" of the User Manual for necessary data.

Figure 3.7: Fluid level measurement in the nitrogen vessel – step 3

i The method described above can also be used to measure the amount of liquid nitrogen in the nitrogen transportation dewar if necessary.

3.4 Nitrogen Refilling Procedure

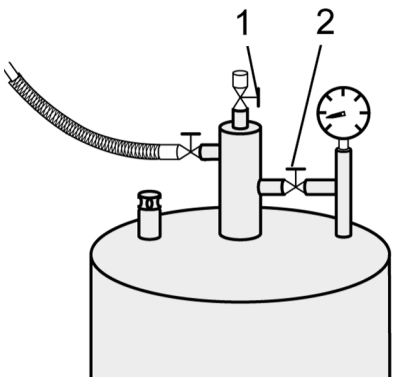


Figure 3.8: Nitrogen refilling procedure – step 1

1. Open the pressure release valve (1) to reduce the pressure in the transportation dewar to maximum 0.35 bar.
2. Close all other valves and taps.
3. Check the amount of liquid nitrogen in the nitrogen transportation dewar. Follow the description given in chapter "Dip-Stick" on page 32.

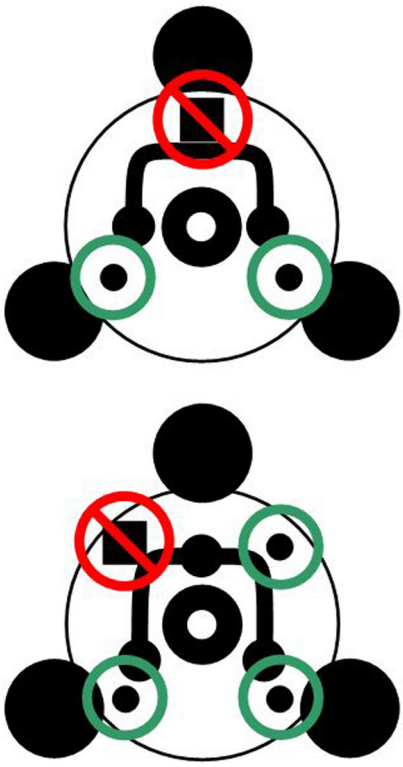
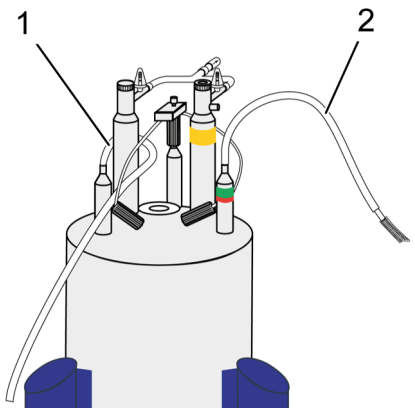


Figure 3.9: Nitrogen refilling procedure – step 2

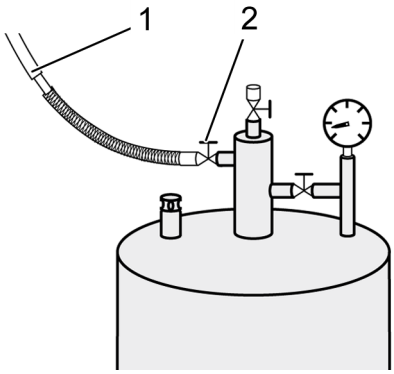
4. If the magnet system is equipped with a vibration isolation system, turn the pneumatic controller switch to the "DOWN" position. Wait until the pressurized air has escaped from the pneumatic isolation system.
5. Record the nitrogen level of the nitrogen vessel in the user interface of the BSMS Service Web. Fill the nitrogen refill report in the User Manual of the magnet system.
6. Remove the heat exchangers from the nitrogen fill-in turret and another recommended nitrogen turret (indicated by green color in figure left) without disassembling the nitrogen flow system.
7. Check that the outlets of the two nitrogen turrets are free.

i To check if the outlets are free watch the open nitrogen turrets if any evaporating nitrogen can be seen. Another option is to carefully insert a thin rod through the nitrogen turrets into the nitrogen vessel.



8. Attach the transfer hose (2) firmly to the nitrogen fill-in turret.
9. Check that the transfer hose is not creased or kinked.
10. Attach a Teflon® hose to the other nitrogen turret (1) as exhaust. Fasten the end of the hose pointing away from the magnet system towards the floor.

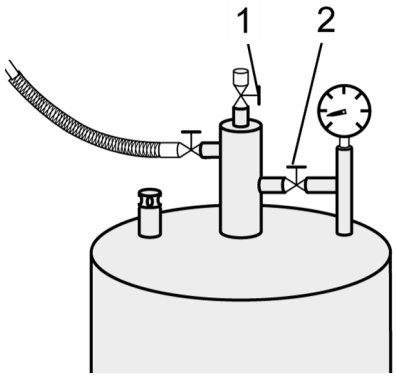
Figure 3.10: Nitrogen refilling procedure – step 3



11. Open the liquid nitrogen extraction tap (2).
12. Apply a pressure of **maximum 0.35 bar** in the transportation dewar.

➪ Transfer of liquid nitrogen will start through the transfer hose (1).

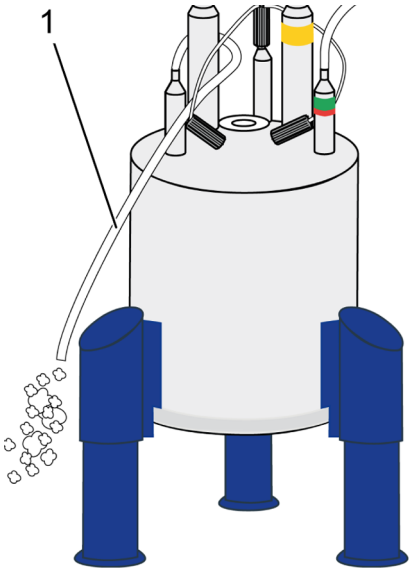
Figure 3.11: Nitrogen refilling procedure – step 4



13. Check the pressure regularly during the entire refilling procedure:
    - To decrease the pressure open the pressure release valve (1).
    - To increase the pressure open the pressure regulator valve (2).
    - Do not exceed a pressure of 0.35 bar.

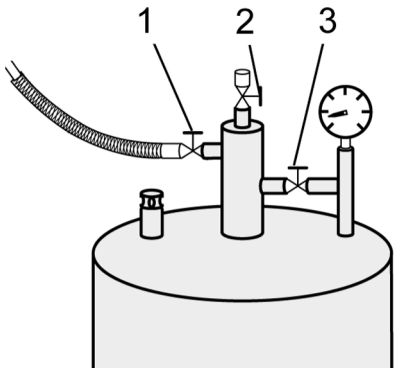
Figure 3.12: Nitrogen refilling procedure – step 5

i The pressure displayed on the pressure gauge of the transportation dewar will sink rapidly as soon as the transportation dewar becomes empty.



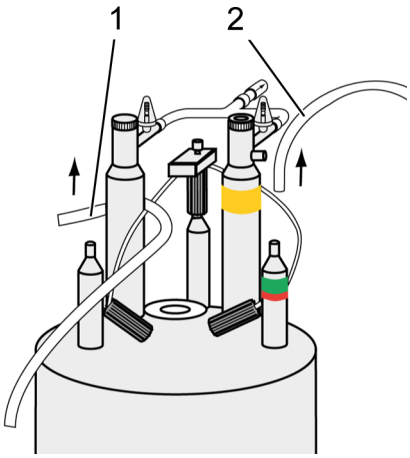
14. Check if gas is escaping from the Teflon® hose (1) at the nitrogen turret used as exhaust. This indicates that the turret is free and not blocked with ice.
15. Check the increasing nitrogen level displayed at the user interface of the BSMS Service Web or at the Nitrogen Level Sensor during refilling.
16. At the end of the refilling procedure:
    - the value of the nitrogen level displayed at the user interface of the BSMS Service Web is 100%,
    - the value at the display of the Nitrogen Level Sensor is 100% and
    - liquid nitrogen starts splashing out of the Teflon® hose on the nitrogen turret used as exhaust.

Figure 3.13: Nitrogen refilling procedure – step 6



17. Close the pressure regulator valve (3).
18. Use the pressure release valve (2) to release the pressure in the transportation dewar.
19. Close the liquid nitrogen extraction tap (1) of the transportation dewar.

Figure 3.14: Nitrogen refilling procedure – step 7



20. Let the transfer hose (2) and the exhaust thaw or carefully warm the end of the transfer hose with a fan heater.
21. Remove the transfer hose (2) from the nitrogen fill-in turret.
22. Remove the Teflon® hose (1) from the nitrogen turret used as exhaust.
23. Check the visible escaping of gas at free all nitrogen turrets showing that they are free of ice.

Figure 3.15: Nitrogen refilling procedure – step 8

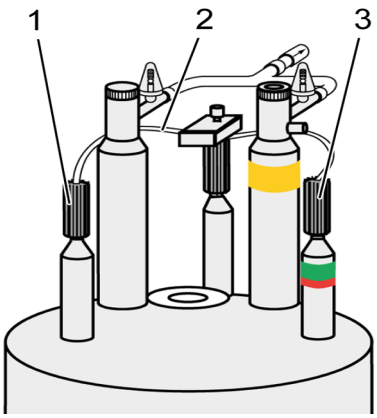


Figure 3.16: Nitrogen refilling procedure – step 9

24. Mount the heat exchangers on the nitrogen turret (1, 3) as quickly as possible to prevent air from entering.
25. Check the correct assembling of the nitrogen flow system (2).
26. If the magnet system is equipped with a vibration isolation system, turn the pneumatic controller switch to the “UP” position.
27. Record the date and quantity used on the transportation dewar in the supplied report sheets.
28. Record the nitrogen level of the nitrogen vessel displayed at the user interface of the BSMS Service Web after refilling. Fill the nitrogen refill report of the User Manual.

i The average nitrogen consumption can be estimated, if the refilling procedure is accurately recorded. Significant change in nitrogen consumption is an early warning signal that the magnet system is not working properly.

3.5 Control Checks

After the nitrogen refilling procedure has been completed, carry out the following control checks to make sure that the magnet system is working properly.

1. After a few hours, check that nitrogen gas is escaping from the nitrogen turrets. If no nitrogen gas is escaping, check if the nitrogen turrets are free (e.g. by inserting a thin rod into the nitrogen turrets).

Refilling Nitrogen

4 Refilling Helium

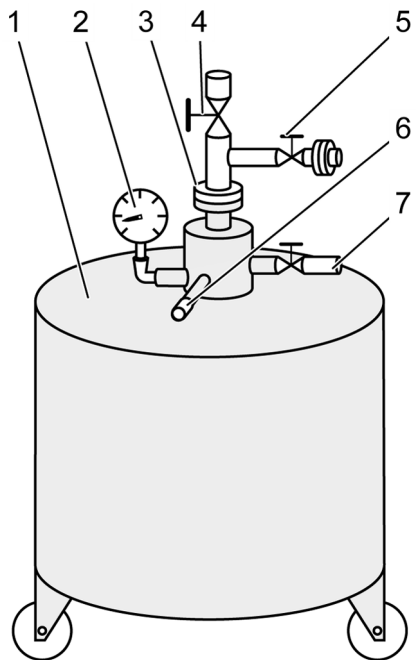
4.1 Introduction

Illustrations in the following chapters are intended for basic understanding while handling cryogenic agents during the refilling procedure. Some illustrations of transportation dewars and accessories given in this manual may differ from the actual design. However, this does not affect the safety advice given in chapter "Safety" nor the function.

4.1.1 Helium Transportation Dewars and Transfer Line

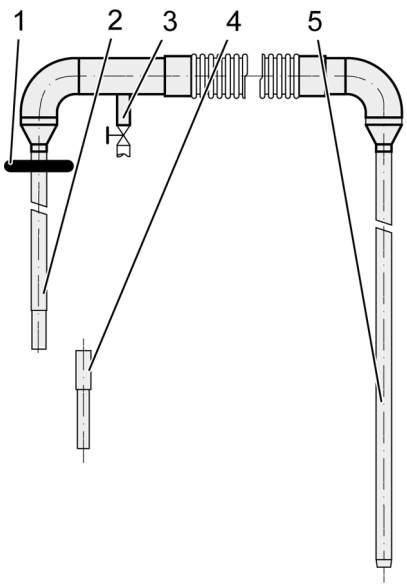
The helium transportation dewars contain liquid helium at a temperature of – 269 °C. Approved personnel handling with cryogenic agents must obey the security advice and the rules for safety and accident prevention (see chapter "Safety").

A transportation dewar for liquid helium consists of the following main components:



1 Transportation dewar
2 Pressure gauge
3 Connection flange
4 Helium extraction tap (globe valve)
5 Pressure regulator valve
6 Safety valve
7 Pressure release valve

Figure 4.1: Transportation dewar for liquid helium



For the transfer of liquid helium a helium transfer line is necessary. The helium transfer line is vacuum-insulated and the vacuum has to be refreshed regularly using a vacuum pump at the vacuum valve (3) of the transfer line.

1 Sealing ring (Z53950)

2 Connector tube – magnet system side

3 Transfer line vacuum valve

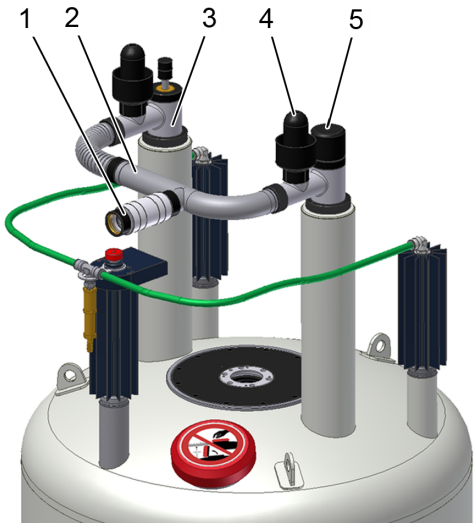
4 Extension piece magnet system side (only used for cooling down the magnet system)

5 Connector tube – transportation dewar side

Figure 4.2: Helium transfer line

4.1.2 Helium Flow System

This chapter describes the components that are common to all superconducting magnet systems that are cooled with cryogenic agents.



1 One-way valve of the helium vessel

2 Helium flow system

3 Helium fill-in turret with siphon

4 Quench valves

5 Helium oscillation damper

Figure 4.3: Helium flow system

4.2 Safety

⚠ WARNING

Magnetic fields (see page 18)

Gas under pressure (see page 20)

Cryogenic agents (see page 19)

Low temperature (see page 21)

Quench (see page 20)

4.3 Fluid Level Measurement

4.3.1 Helium Level Sensor

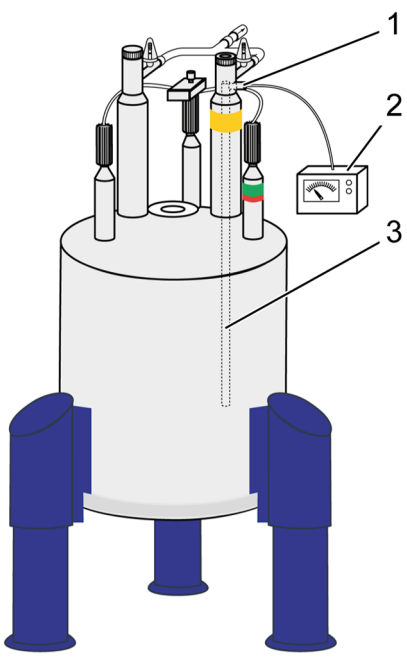


Figure 4.4: Helium Level Sensor

The magnet system provides a built-in Helium Level Sensor (3) to measure the fluid level. It is connected to the BSMS Service Web, where the measured value can be displayed.

Optionally a Helium Level Measurement Unit (HLMU, (2)) can be connected to the socket (1) on the helium fill-in turret.
Please contact Bruker Service for further information.

1 Socket to the Helium Level Sensor (3)

2 Helium Level Measurement Unit (HLMU) (optionally).

3 Helium Level Sensor

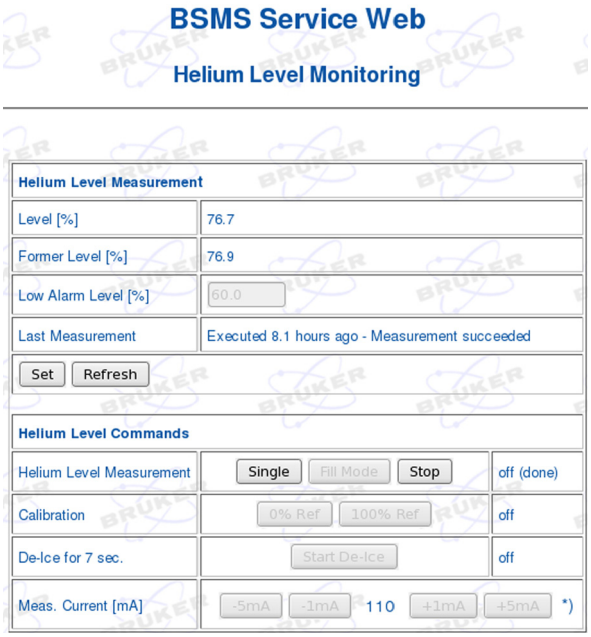
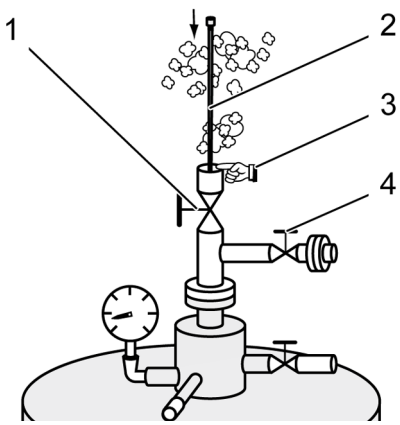


Figure 4.5: User interface of the BSMS Service Web – helium level monitoring

4.3.2 Fluid Level Measurement in the Transportation Dewar

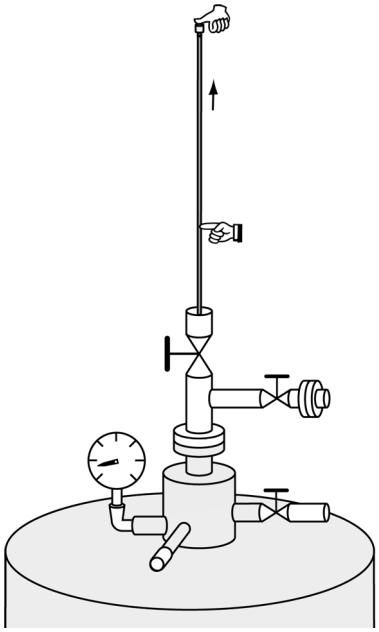
Fluid level measurements in the transportation dewar are necessary to determine the effective quantity of helium used during refilling. Measurement of the fluid level in the transportation dewar is only possible before or after the refilling procedure. During the refilling procedure it can only be checked if the transportation dewar still contains any helium. Measure the fluid level in the transportation dewar either by weighing or with the dip-stick.

The dip-stick is standard equipment of the magnet system. In case of loss or damage the dip-stick is available from BRUKER BioSpin AG.



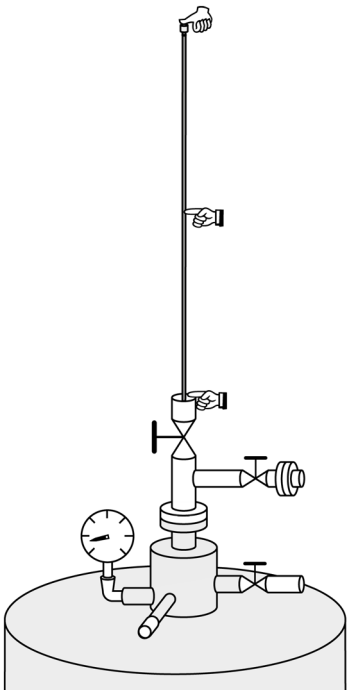
1. Open the pressure regulator valve (4) to release the pressure in the transportation dewar.
2. Open the helium extraction tap (1) and insert the dip-stick (2) carefully until it reaches the bottom of the transportation dewar.
3. Mark this position of the dip-stick near the entrance of the helium extraction tap (3).

Figure 4.6: Fluid level measurement in the transportation dewar – step 1



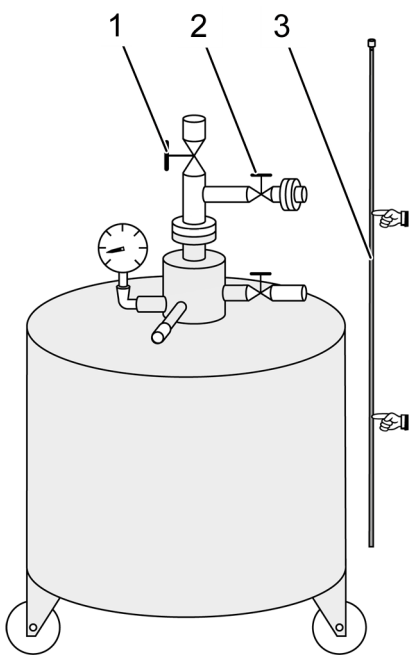
4. Seal the upper end of the dip-stick with the moistened thumb or with a plastic sheet, so that a fine sizzling of the helium gas can be heard and felt.
5. Slowly pull out the dip-stick until a clear change of the sizzling frequency can be heard and felt.
   The frequency of the sizzling sound will become significantly higher as soon as the end of the dip-stick is at the surface of the liquid helium (transition point between liquid and gas).

Figure 4.7: Fluid level measurement in the transportation dewar – step 2



6. Mark this second position of the dip-stick near the entrance of the helium extraction tap.

Figure 4.8: Fluid level measurement in the transportation dewar – step 3



7. Pull out the dip-stick of the transportation dewar.
8. Close the helium extraction tap (1) and the pressure regulator valve (2).
9. Measure the distance between both marks on the dip-stick (3).
10. Determine the amount of liquid helium from the fluid level table of the transportation dewar.

Figure 4.9: Fluid level measurement in the transportation dewar – step 4

---

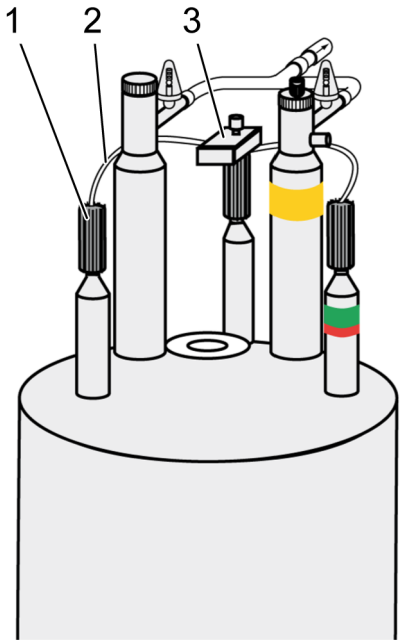
i During the refilling procedure, only an indirect checking of the presence of remaining fluid in the transportation dewar is possible:

Method No. 1
Check the fluid level displayed on the interface of the BSMS Service Web or the HLMU (optionally) for some time. An increasing value shows that helium is being transferred to the magnet system and that the transportation dewar is not empty.

Method No. 2
A whistling noise of the transfer line indicates that helium gas is flowing through the transfer line and that the transportation dewar is empty.

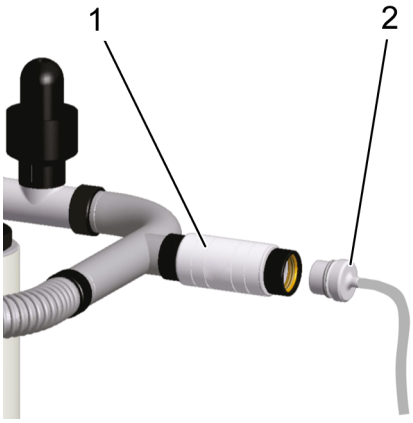
4.4 Helium Refilling Procedure

The refilling of helium should only be performed if the nitrogen vessel is full. Thus, each time before refilling helium check the nitrogen fluid level and refill the nitrogen vessel if necessary.



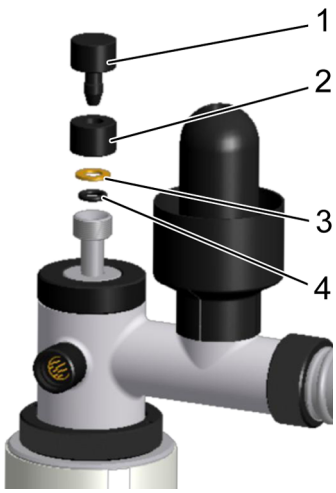
1. If the magnet system is equipped with a vibration isolation system, turn the pneumatic controller switch to the “DOWN” position. Wait until the pressurized air has escaped from the pneumatic isolation system.
2. Check if the heat exchangers (1) and the nitrogen flow system (2) are correctly mounted on the nitrogen turrets to prevent air from entering the nitrogen vessel during refilling.
3. Check if the Nitrogen Level Sensor (3) is mounted correctly on the rear nitrogen turret to prevent air from entering the nitrogen vessel during refilling.
4. Record the helium level displayed at the user interface of the BSMS Service Web. Fill the helium refill report of the User Manual.

Figure 4.10: Helium refilling procedure – step 1



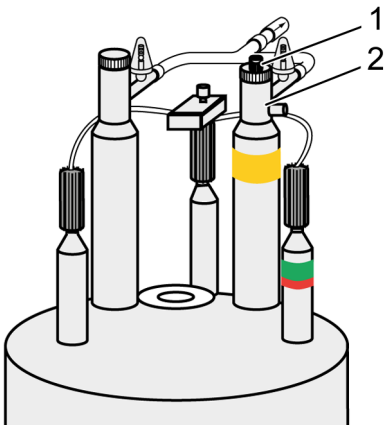
5. If equipped with an Atmospheric Pressure Device (APD) switch the APD OFF and wait until the over pressure is released (no more helium escaping the exaust of the APD; this can take 8 hours).
6. Remove the socket with hose (2) from the one-way valve (1) of the helium vessel. Do **not** remove the one-way valve (1).
7. If a helium recovery line is available connect the outlet of the one-way valve (1) of the helium vessel to the helium recovery line.

Figure 4.11: Helium refilling procedure – step 2



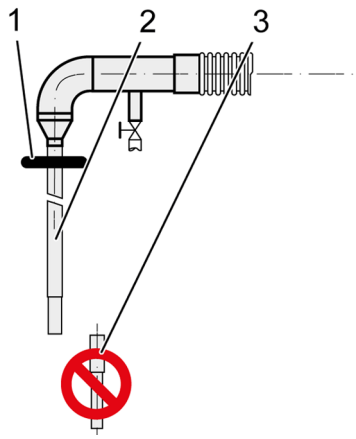
8. Remove the union nut (2) with the plug (1), washer (3) and O-ring (4) from the helium fill-in turret (5).
9. Check the O-ring (4) for visible damage. If necessary replace the O-ring. Contact Bruker Service for further information.

Figure 4.12: Helium refilling procedure – step 3



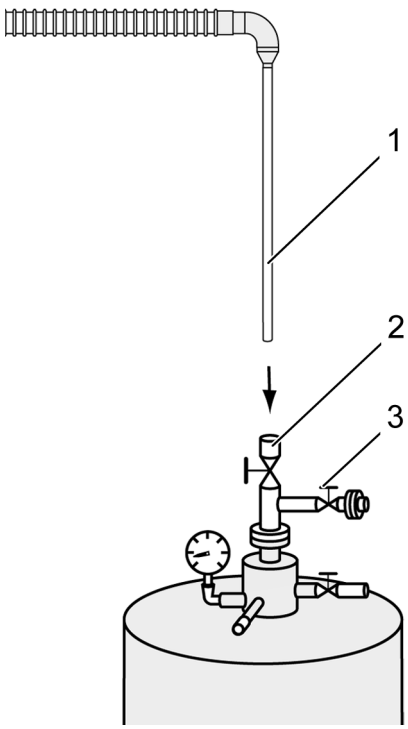
10. Remount the plug (1) to close the helium fill-in turret (2) immediately to prevent air from entering the turret.

Figure 4.13: Helium refilling procedure – step 4



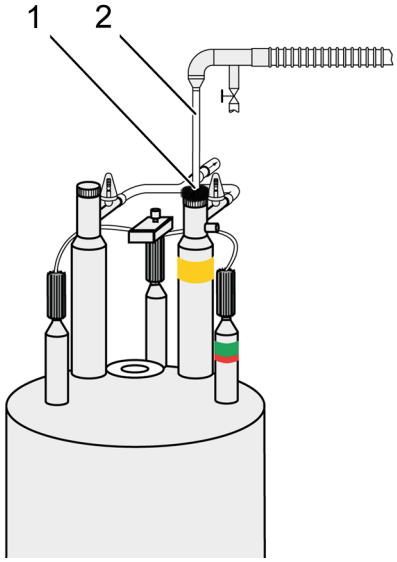
11. Check if the sealing ring (1) is mounted on the magnet system side of the transfer line (2).
12. Check if the extension piece (3) is **not** mounted on the magnet system side of the transfer line (2).

Figure 4.14: Helium refilling procedure – step 5



13. Open the pressure regulator valve (3) of the transportation dewar for a short moment to release the pressure in the transportation dewar.
14. Close all taps on the helium transportation dewar.
15. Insert the transfer line (1) into the helium extraction tap (2) of the transportation dewar until it reaches the closed valve.
16. Open the helium extraction tap (2).
17. Push the transfer line (1) carefully and slowly into the transportation dewar to make a small helium gas flow at the open end of the transfer line.
18. Allow the transfer line to cool down until liquid helium is coming out at the open end of the transfer line, looking like a bluish flame.
19. Open the pressure regulator valve (3) of the transportation dewar to release the pressure.

Figure 4.15: Helium refilling procedure – step 6



20. Remove the plug out of the helium fill-in turret.
21. Carefully insert the cold transfer line (2) without extension piece completely into the helium fill-in turret.
22. Push the transfer line (1) into the transportation dewar until it reaches the bottom of the transportation dewar.
23. Pull out the transfer line 50 – 80 mm and fix it in this position.

⚠ WARNING: Quench

24. Check that the pressure in the transportation dewar is maximum 100 mbar. High pressure during refilling helium can cause a quench. Release pressure by opening the pressure release valve.
25. Seal the helium fill-in turret with the sealing ring (1).

Figure 4.16: Helium refilling procedure – step 7

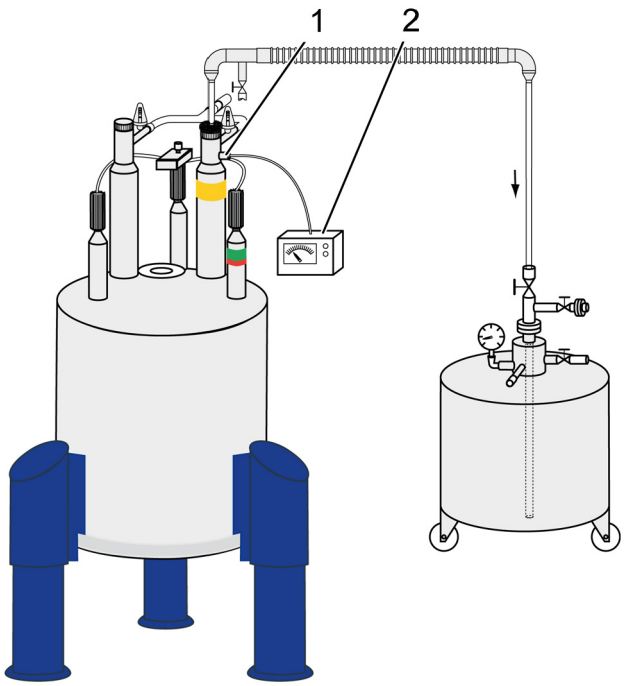


Figure 4.17: Helium refilling procedure – step 8

26. Switch the helium level measurement at the BSMS Service Web to continuous measurement mode (“Fill Mode”).

    Optionally a Helium Level Measurement Unit (HLMU (2)) can be connected to the socket (1) on the helium fill-in turret. Please contact Bruker Service for further information.

27. Apply a moderate pressure of 50 to 100 mbar in the transportation dewar using one of the following methods:

Method A

The insulation of the transportation dewar is not perfect. Pressure is built up by permanently evaporating helium. In some cases this is sufficient to generate the required pressure in the transportation dewar.

Method B

Connect a pressure cylinder with pure helium gas to the pressure regulator valve at the transportation dewar. Open the pressure regulator valve until a moderate pressure of 50 to 100 mbar is built up in the transportation dewar.

Method C

Connect a rubber bladder (e.g. from the inside of a football) to the pressure regulator valve at the transportation dewar and knead the bladder. By circulating the warm helium gas inside the transportation dewar a sufficient pressure will build up.

➪ Transfer of liquid helium will start.

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i The refilling procedure can take up to one hour.

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28. Check the pressure in the transportation dewar regularly during the entire refilling procedure:
    - To decrease the pressure open the pressure release valve.
    - To increase the pressure open the pressure regulator valve.
    - Do not exceed a pressure of 100 mbar.
29. Check the helium gas flow escaping at the one-way valve of the helium vessel or the ice covering at the recovery line if connected.
30. Check the increasing helium level displayed at the user interface of the BSMS Service Web or at the HLMU (optionally) during refilling.
31. At the end of the refilling procedure check:
    - the value of the helium level displayed at the user interface of the BSMS Service Web or the HLMU (optionally) is above 95 % with constant value,
    - liquid air (nitrogen and oxygen) drops from the helium flow system and
    - a large helium gas flow at the one-way valve of the helium vessel or massive ice covering at the recovery line.

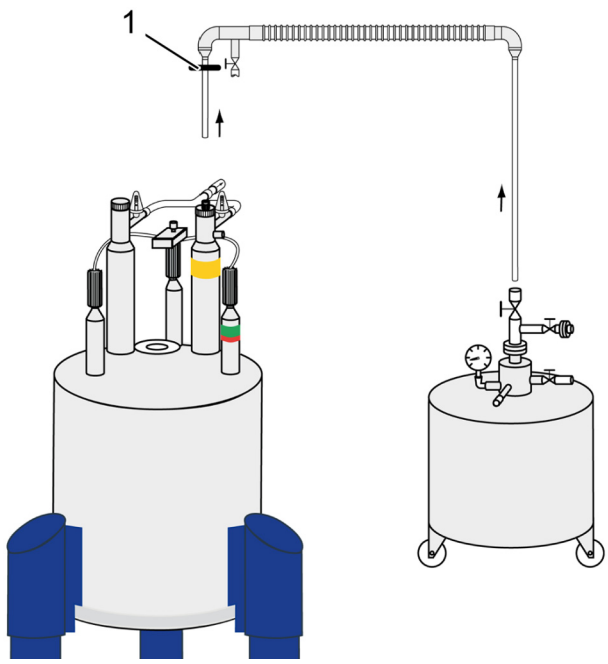
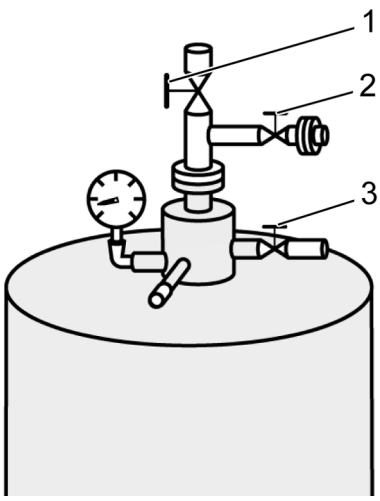


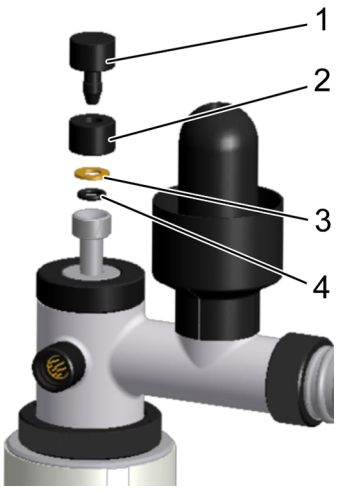
Figure 4.18: Helium refilling procedure – step 9

32. Pull out the transfer line from the transportation dewar halfway.
33. Pull out the transfer line with sealing ring (1) of the helium fill-in turret completely.
34. Pull out the transfer line from the transportation dewar completely.
35. Immediately close the helium fill-in turret with the plug.



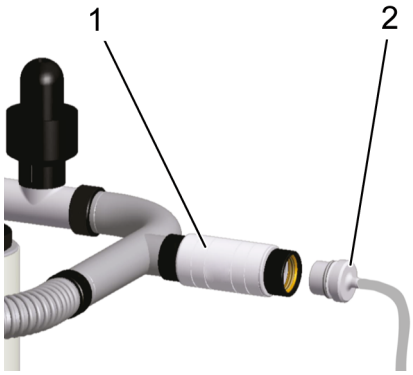
36. Close the helium extraction tap (1) and the pressure regulator valve (2) on the transportation dewar.
37. Remove the hose to the helium pressure cylinder if connected (Method B).
38. Open the pressure release valve (3).

Figure 4.19: Helium refilling procedure – step 10



39. Remove the plug (1) from the helium fill-in turret.
40. Remount the union nut (2), washer (3) and O-ring (4) back to the plug (1) as shown and close the helium fill-in turret immediately.

Figure 4.20: Helium refilling procedure – step 11



41. Remove the helium recovery line if connected.
42. Remount the socket with hose (2) to the one-way valve (1) of the helium flow system.

Figure 4.21: Helium refilling procedure – step 12

43. If the magnet system is equipped with a vibration isolation system, turn the pneumatic controller switch to the "UP" position.
44. Determine the quantity of helium left in the transportation dewar (either by weighing or measuring the fluid level with the dip-stick).
45. Write down the date and quantity used on the transportation dewar in the supplied data sheets.
46. Write down the helium level displayed at the user interface of the BSMS Service Web after refilling. Fill the helium refill report of the User Manual.

i The average helium consumption can be estimated, if the refilling procedure is accurately recorded. Significant change in helium consumption is an early warning signal that the magnet system is not working properly.

4.5 Control Checks

After the helium refilling procedure has been completed, carry out the following control checks to make sure if the magnet system is working properly.

1. Make sure that the helium flow system thaws out. This step can be supported by carefully using a fan-heater.
2. After a few hours, check that nitrogen gas is escaping from the nitrogen turrets. If no nitrogen gas is escaping, check if the nitrogen turrets are free (e.g. by inserting a thin rod into the nitrogen turrets).
3. Check the helium flow.

Refilling Helium

5 Troubleshooting

The problems described in this chapter may occur during the refilling procedure.

Contact Bruker Service in case of problems not described in this chapter (for contact information see page 9 of this document).

Indicator	Possible Reason	Solution	By
Transfer of liquid helium does not start.	The transportation dewar is empty.	Change the transportation dewar. To check if the transportation dewar is empty or not: 1. Close the pressure regulator valve on the transportation dewar and remove the hose that provides external helium gas. 2. a) Open the pressure regulator valve to release pressure. If no helium gas escapes, the transportation dewar is empty, as the helium gas can escape directly into the magnet system via the transfer line. b) Mount a rubber bladder (e.g. from inside a football) on the socket of the pressure regulator valve and open the pressure regulator valve slowly. If the rubber bladder does not stay firmly pressurized, the transportation dewar is empty.	Approved Customer Personnel
	The pressure in the transportation dewar is too low.	Increase the pressure in the transportation dewar (max. pressure 100 mbar).	Approved Customer Personnel
	The transportation dewar is leaky; no pressure is built up.	Check the transportation dewar for leakage. Tighten all connections.	Approved Customer Personnel

Continued on next page

Continued from page before

Indicator	Possible Reason	Solution	By
Continue of: Transfer of liquid helium does not start.	The helium transfer line is blocked with ice.	Remove ice with warm helium gas.	Approved Customer Personnel
		If the problem is still not solved, contact Bruker Service.	Approved Customer Personnel
	The siphon is blocked with ice.	Check the siphon for blockage, remove ice with warm helium gas.	Bruker Service
Displayed helium level value at the BSMS Service Web is arbitrary and stable.	Icing of the helium level sensor.	Use the de-icing option of the helium level sensor in the BSMS Service Web. If not equipped with, contact Bruker Service.	Approved Customer Personnel
		Remove the helium level sensor out of the helium fill-in turret and let it warm up. Remount the helium level sensor.	Bruker Service
	Helium level sensor is defective.	Refer to the Service Manual of the magnet system.	Bruker Service
The helium level does not reach 100 %.	The transportation dewar is empty, no more helium is transferred.	Change the transportation dewar.	Approved Customer Personnel
	The helium level sensor is disturbed by the transfer line.	1. Stop the transfer of liquid helium. 2. Remove the transfer line and wait a few minutes. 3. Repeat the helium level measurement.	Approved Customer Personnel

A Appendix

A.1 Warning Signs

Danger

Warning

Caution

Notice

Warning Signs

A.2 Figures

Figures

A.3 Tables

Tables

A.4 Glossary and Abbreviations

Used term	Description
Cryogenic Agents	Fluid (helium or nitrogen) in cold liquid or cold gaseous form.
Dip-Stick	Long metal tube with a round end-piece for measuring the fluid level in cryogenic liquids.
Helium	Light, colorless, odorless and tasteless gas with the chemical formula He. Liquid helium has a temperature of –269 °C / 4 K.
Helium Flow System	Assembly on the magnet system, which connects the helium turrets in order to releases evaporated helium into the atmosphere and to prevent air and moisture from entering the helium vessel; it builds the interface to the helium vessel.
Helium Level Sensor	Measuring device for measuring the fluid level in the helium vessel.
Helium Oscillation Damper	Part of the helium flow system for suppression of thermo-acoustic gas oscillations.
Nitrogen	Heavy, colorless, odorless gas with the chemical formula N_2. Liquid nitrogen has a temperature of –196 °C / 77 K.
Nitrogen Flow System	Assembly on the magnet system, which connects the nitrogen turrets in order to releases evaporated nitrogen into the atmosphere and to prevent air and moisture from entering the nitrogen vessel; it builds the interface to the nitrogen vessel.
Nitrogen Level Sensor	Measuring device for measuring the fluid level in the nitrogen vessel.
Quench Valve	Pressure release valve of the helium flow system.
Siphon	Metal tube in the helium fill-in turret of the magnet system to guide liquid helium to the bottom of the helium vessel.
Transfer Line	Vacuum-insulated transfer pipe for liquid helium.
Transportation Dewar	Vacuum-insulated container for storage and transportation of liquid cryogenic agents (e.g. helium and nitrogen).

Table A.1: Glossary

Abbreviations	Description
BSMS	Bruker Smart Magnet control System
HLMU	Helium Level Measurement Unit
RT	Room Temperature; used as prefix of parts which are at room temperature

Table A.2: Abbreviations

A.5 Refilling Reports

Nitrogen level before refill [%]	Nitrogen level after refill [%]	Refill Volume [l]	Date, Time	Signature

Refilling Report

Helium level before refill [%]	Helium level after refill [%]	Refill Volume [l]	Date, Time	Signature

A.6 Index

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Revision History List

Index:	Date:	Alteration Type:
01 - 06	October 2004	Previous releases
07	June 2013	Updated layout in accordance with Bruker Corporate Guidelines; updated safety signs and hazard symbols.

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