

## SOP: filling liquid helium on NMR magnets

*This is only to be done by authorized personel*

This SOP has to be read in connection with the SOP for filling N<sub>2</sub>(l) above (the first 15 pages of the Bruker manual)



UltraShield™ NMR Magnet System

Refilling Procedure

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### Helium Refilling Procedure

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#### 7 Transport Vessel for Liquid Helium

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There are various forms of transport vessels for liquid Helium (chemical formula He). Described here are the characteristics which are valid for all vessel implementations and which you should be aware of for a safe execution of the refilling procedure.

##### 7.1 Danger source: Ultra-low Temperature

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The transport vessel contains liquid Helium with a temperature of -269°C. Please observe the warning notes in chapter 1 "Safety during Refilling Procedure", page 3 when dealing with the transport vessel.

##### 7.2 Requirements for Helium Transport Vessel

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The transport vessel must fulfil the following requirements:

- It must not be ferromagnetic. That means it must not be made up of any material, which is susceptible to magnetic fields.
- It must possess a fixed, mounted safety valve which cannot be externally influenced.
- It must possess a pressure release valve, which releases evaporating Helium.



Warning

**Danger of injury: Magnetic transport vessels could be pulled uncontrollably towards the magnet system and could trap or crush people.**



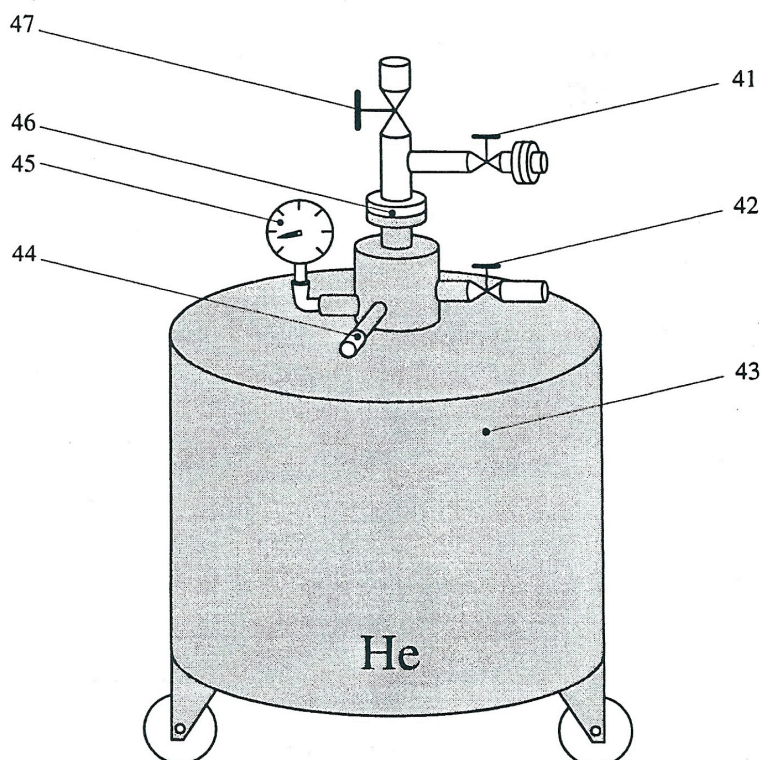
Caution

**Danger of destruction of the magnet system. Magnetic transport vessels could be pulled uncontrollably towards the magnet system and lead to destruction.**



### 7.3 Main components

A transport vessel for liquid Helium consists of the following main components:



Transport vessel for liquid Helium

#### Legend

- 41 Gas release valve (and /or valve for pressure generation)
- 42 Pressure release valve (with locking tap)
- 43 Transport vessel
- 44 Safety valve
- 45 Manometer
- 46 Connection flange
- 47 Extractor tap (ball-bearing valve) for liquid helium



## UltraShield™ NMR Magnet System

## Refilling Procedure

### 8 Transfer Line

Due to the extremely low temperature of liquid helium, a special transfer line is required for the filling procedure. It is double-skinned, insulated and evacuated.



Note

Do not use any other type of pipe or a damaged transfer line. The pipe would ice up and the helium would evaporate due to the faulty isolation.



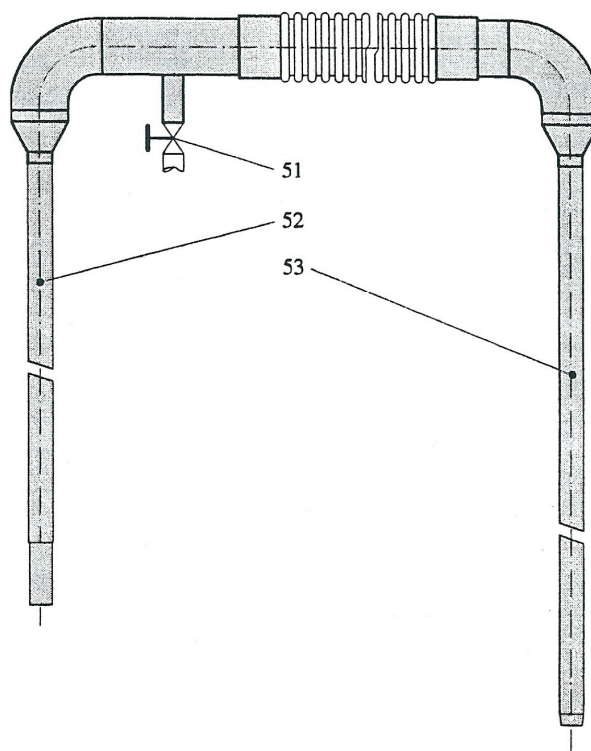
Caution

In case of problems with a transfer line, please contact your local Bruker Service Department.



Note

You can recognise a defective transfer line in that the defective area ices up (becomes frosty white) during the helium transfer process.



Transfer Line

#### Legend

- 51 Transfer line evacuation valve
- 52 Connector tube - Magnet side
- 53 Connector tube - Transport vessel side



## UltraShield™ NMR Magnet System

## Refilling Procedure

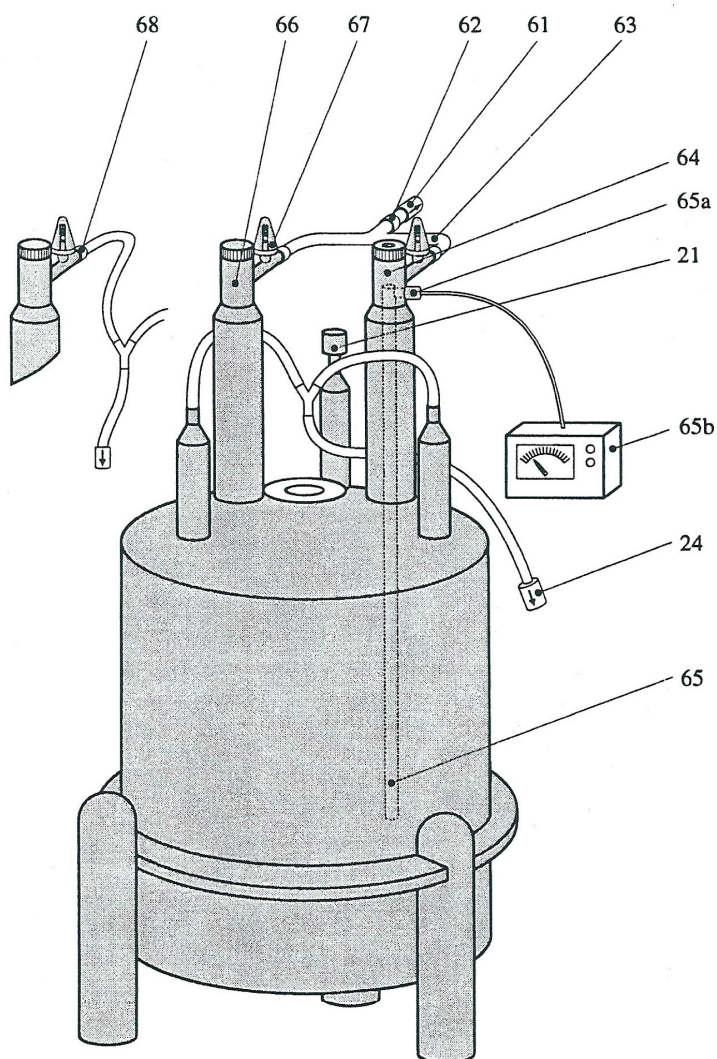
### 9 Magnet System

Described here after are the individual elements common to all magnet systems, which you should know about and observe to ensure safe execution of the helium refilling procedure.



#### Caution

The magnet system contains liquid helium and a magnet that produces a very strong magnetic field. Please observe the safety measures as described in chapter 1 "Safety during Refilling Procedure", page 3.



Magnet System

Legend



UltraShield™ NMR Magnet System

Refilling Procedure

- 21 Safety valve (for nitrogen)
- 24 Check valve (for nitrogen)
- 61 Check valve (for helium)
- 62 Helium oscillation damper
- 63 U-tube
- 64 Right helium turret with siphon
- 65 Helium level sensor
- 65a Connector to the helium level sensor
- 65b Helium level meter
- 66 Left-hand helium turret
- 67 Quench valve
- 68 Adapter with hose



Note

Not all magnetic systems are equipped with a U-tube (63). As an alternative to this, there are systems which possess adapters with hoses (68) at both helium turrets. These hoses are connected together by means of a Y-piece and ventilated via the check valve (61).



## 10 Fluid Level Control

The fluid level measurement in the magnet system indicates how much Helium needs to be added. Fluid level measurements in the transport vessel enables you to determine the effective quantity of helium used. During the filling procedure you can only check whether the transport vessel still contains any helium.

### 10.1 Measuring the Fluid Level in the Magnet System

The magnet system provides a fixed, built-in helium level sensor to measure the fluid level (65). You can connect a helium level meter (65b) to the standardised connector (65a). Please read the operating instructions of your helium level meter.

### 10.2 Measuring the Fluid Level in the Transport Vessel

Measurement of the fluid level in the transport vessel is only possible before or after the filling procedure. During the filling procedure you can only check whether the transport vessel still contains any helium.



Note

You will need a dip-stick to measure the fluid level. This is a long tube with a round end-piece, available as Part Number Z27451 from Bruker.

#### Measuring the fluid level

To measure the fluid level in the transport vessel you need finger-tip feeling in every sense of the word! Follow the procedure outlined below:

1. Release the pressure in the transport vessel by opening the gas release valve (41).
2. Open the helium extraction tap (47) and insert the dip-stick carefully until it reaches the floor of the transport vessel.
3. Mark the position of the dip-stick by holding it at the entrance of the extraction tap.
4. Seal the upper end of the dip-stick with the thumb of your other hand (moistened beforehand) or a small piece of plastic, so that you can feel and hear a fine sizzling of the helium gas.
5. Slowly pull the dip-stick out until you notice a clear change of the "sizzling frequency".
6. Mark the position of the dip-stick at the transition point, by holding it directly at the extraction tap.
7. Measure the distance between both markings on the dip-stick and determine the fluid level on the fluid level table of the transport vessel.
8. Close the gas release valve (41) and the liquid helium extraction tap (47).



Note

The frequency of the sizzling sound will become significantly higher as soon as the end of the dip-stick surfaces above the level of the liquid helium. If you are not certain whether you have passed this transition point or not, you can repeat the process by pushing the dip-stick deeper down in the tank and trying again.



## UltraShield™ NMR Magnet System

## Refilling Procedure

### Checking of the fluid level

During the refilling procedure, only an indirect checking of the presence of remaining fluid in the transport vessel is possible.

The easiest way of checking this is by reading the helium level meter (65b) on the magnet system. If the fluid level indicator slowly and continually increases, you can be sure that the transport vessel still contains fluid.



#### Note

The helium level sensor is very sensitive on icing. In case of icing it does not function any more and will indicate an arbitrary but stable value. Some helium level meters are equipped with a de-icing function. Please consult the respective manual.



#### Caution

If the icing can not be removed, please contact your local Bruker Service Department.

A slight over pressure can be generated in the transport vessel with the help of a rubber bladder (e.g. the inside skin of a football). You can determine whether the transport vessel is empty by observing the rubber bladder. If it does not stay firmly pressurised, the vessel must be empty, as no more over pressure can be generated.

In order to generate the over pressure externally with helium gas, follow the procedure outlined below:

1. Close the pressure generation valve (41) and remove the hose which delivers the external helium gas.
2. Open the pressure generation valve (41) and observe whether the transport vessel is under pressure.



#### Note

No pressure can be generated in an empty transport vessel, as the gas can escape directly into the magnet system via the transfer line.

3. Close off the hose for delivering external helium gas and open the pressure generation valve (41) to continue the filling procedure.



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## 11 Preparation for Transfer

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Prepare the magnet system for helium transfer by following the procedure outlined below:

1. If the magnet system is equipped with vibration dampers: Let the air escape out of the dampers by turning the switch on the base plate to the "Down" position.
2. Make sure that the nitrogen outlets are correctly closed with a check valve, or if necessary, close the outlets with a rubber stopper.



Caution

**Danger of excess pressure in the magnet system when using rubber stoppers. Remove the rubber stoppers after the filling procedure has been completed.**

Closing the nitrogen outlets makes sure that the super cooled magnet system does not suck air into the nitrogen tank. A correctly mounted check valve (24) (arrow pointing outwards) is sufficient for this purpose.

3. Make certain that the N<sub>2</sub> safety valve (21) is mounted onto the rear nitrogen turret.



Caution

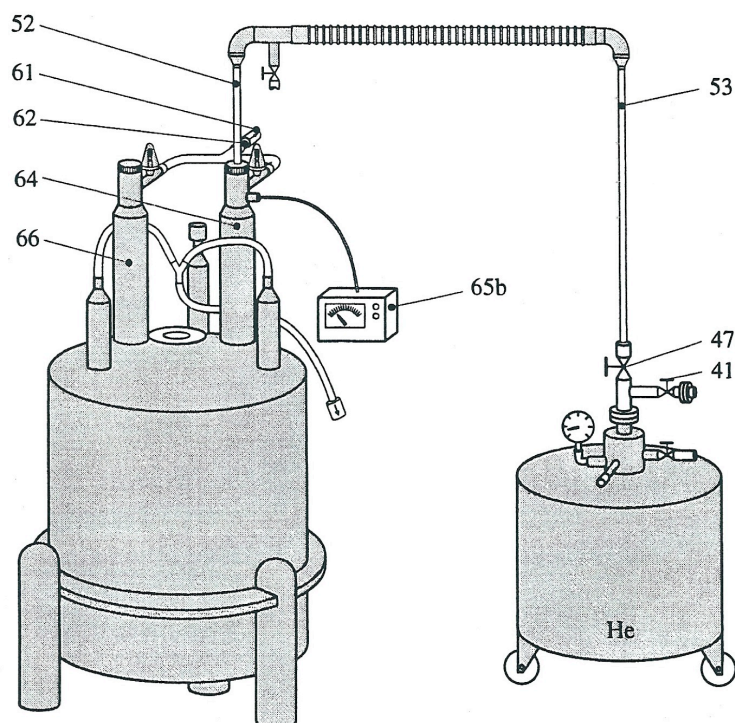
**Danger of the magnet system bursting. Always leave the safety valve and quench valves in their correct positions.**





## 12 Filling procedure

Before starting the filling process the transfer line must first be cooled down. After the transfer line has been cooled down you can make the connection between the transport vessel and the magnet system. The helium will be drawn from the transport vessel into the magnet system via the transfer line when a slight over pressure established. The filling process is finished when liquid helium starts coming out of the outlet.



System during filling procedure

### Legend

- 41 Gas release valve (and /or valve for pressure generation)
- 47 Extractor tap (ball-bearing valve) for liquid helium
- 52 Connector tube - Magnet side
- 53 Connector tube - Transport vessel side
- 61 Check valve (for helium)
- 62 Helium oscillation damper
- 64 Right helium turret with siphon
- 65b Helium level meter



66 Left-hand helium turret

### 12.1 Cooling the Transfer Line

If a warm transfer line is inserted into the magnet system, the warm helium gas coming out of the transfer line, could cause a very strong reaction called a quench. You can prevent this by first cooling the transfer line. Follow the procedure outlined below.



Caution

**Carefully read the following instruction steps before carrying out this procedures!**

1. Remove the check valve (61) and if applicable the oscillation damper (62), or
2. remove the adaptor with hose (68) from the left-hand helium turret (66).
3. Close the outlet opening by pressing a tissues lightly into the opening.



Note

This minimises the entrance of air into the helium vessel until you start to fill it with helium. Air entering the vessel will freeze.

4. Release the slight over-pressure in the transport vessel by opening the gas release valve (41) for a short period.
5. Close all taps on the transport vessel.
6. Insert the connecting tube (53) of the transfer line into the liquid helium extraction tap (47) until it reaches the ball-bearing valve.
7. Prepare the right-hand helium turret on the magnet system (64). Remove the screw cap with stopper, washer and O-ring and immediately seal the connector again with the stopper to minimise the entrance of air.
8. Prepare the transfer line for connecting with the magnet system by placing the screw cap with washer and O-ring onto the connecting tube (52).
9. Open the liquid helium extraction tap (47). Push the transfer line slowly into the transport vessel until helium in the form of gas, starts escaping at the other end.
10. Allow the transfer line to cool down until liquid helium starts to exit the open end of the connecting tube (52)



Note

You can easily recognise when liquid helium starts coming out. The stream exiting the open end looks like the flame of a candle.

### 12.2 Connecting the Transfer Line

As soon as the transfer line has cooled down, you can establish the connection between the transport vessel and the magnet system - then the helium transfer can begin.



Caution

**Danger of a quench:- When inserting an insufficiently cooled transfer line. Cool the transfer line until liquid helium comes out of the open end.**





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11. Insert the connecting tube (52) of the transfer line (without extension piece) carefully into the right-hand helium turret (64) and fix it with the nut.



### Caution

The end of the transfer line must not be submerged into the siphon in the helium tank. Check whether you are using the correct transfer line if you notice that it touches the siphon. Should the transfer line submerge into the siphon and reach the bottom of the siphon, pull back the transferline 2-3 cm (1 inch) and fix it with the screw cap.

### 12.3 Generating Over Pressure in the Transport Vessel

The helium is transferred from the transport vessel with the help of a slight over pressure. This pressure must not exceed 0.35 bar under any circumstances. You can generate the required pressure as described below:

12. Connect clean helium gas to the pressure generation tap (41) and open the tap until a slight over-pressure is built up in the transport vessel, or...
13. Connect a rubber bladder from the inside of a football, and knead the bladder. By circulating the warm helium gas in the transport vessel, this disturbance leads to sufficient pressure build up.



### Note

The insulation of the transport vessel is not perfect. Pressure is built up naturally by permanently evaporating helium. In some cases this is sufficient to generate the required over pressure.



### Note

The filling procedure can take up to one hour if you refill when reaching the minimum allowed helium level after the maximum hold time.

### 12.4 Helium Transfer



### Note

As soon as you insert the connecting tube (52), the helium transfer process begins. You can clearly recognise this in that the Kleenex tissue is blown out of the outlet opening.

14. Push the connecting tube (53) into the transport vessel until it reaches the bottom and then pull it back about five centimetres (2 inches). This will prevent the transfer line from becoming blocked during the filling procedure.
15. Switch the helium level meter (65b) to continuous measurement, if possible.
16. Provide a slight over pressure in the transport vessel as required (see section 12.3 "Generating Over Pressure in the Transport Vessel", page 26).



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### 12.5 Monitoring the Helium Transfer

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17. Make sure that the pressure does not exceed 0.35 bar during the entire filling procedure.



Caution

**Danger of explosion due to excess pressure build-up. Respect the maximum pressure of 0.35 bar at all times.**



Note

A pressure of 50 to 100 mbar is usually enough for a satisfactory transfer of helium.

18. During the filling process, check whether the transport vessel contains enough fluid (see section 10.2 "Measuring the Fluid Level in the Transport Vessel", page 21).



Note

You can easily recognize that there is still fluid in the transport vessel by observing the indicator on the helium level meter (65b). As long as it is continually rising, the transport vessel cannot be empty.



Note

The helium level sensor is very sensitive on icing. In case of icing it does not function properly and will indicate an arbitrary but stable value. Some helium level meters are equipped with a de-icing function. Please consult the respective manual.



Caution

**If the icing can not be removed, please contact your local Bruker Service Department.**

19. Finish the helium transfer process as soon as the helium tank is full.

You can recognise a full helium tank with the following methods:

- The indicator on the helium level meter shows 100%.
- Liquid air (nitrogen and oxygen) drops from the U-tube.



Note

Please observe the warning notes in section 1.4 "Protection against Fire and Explosion Risks", page 4.

- Liquid helium starts to escape from the outlet opening. This looks like a small flame.



Caution

**If the icing can not be removed, please contact your local Bruker Service Department.**



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### 12.6 Ending the Filling Procedure and Removing the Transfer Line

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The filling procedure is ended as soon as liquid helium starts escaping from the outlet opening, which you can recognise by the small "flame" as already described.

End the process as follows:

1. If you have connected an external helium supply for the pressure generation: Close the pressure generation tap (41) and remove the corresponding hose.
2. Release the pressure from the transport vessel by opening the gas release valve (41).
3. Loosen the screw caps and pull the transfer line simultaneously from the helium tank and the transport vessel.



Caution

**Danger - Do not touch the ultra-cold connecting tubes of the transfer line. Wear protective gloves when removing the transfer line.**



Note

4. Immediately close the right-hand helium turret with the stopper.  
Frozen parts made of metal can be thawed out with a fan heater.  
Do not use excessive temperatures on parts made of plastic.
5. When the transfer line has thawed out, remove the O-ring, washer and screw up and check the O-ring for damage.

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### 12.7 Return to Standard Operation after the Filling procedure

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After successful execution of the filling procedure, reverse the procedure as follows:

1. Close all taps and valves on the transport vessel.
2. Open the pressure release valve (41).
3. Close the right-hand helium turret (64) properly with the screw cap, O-ring, washer and valve.
4. Mount the helium check valve (61) on the magnet system such that the arrow is pointing outwards or
5. mount the adaptor with the hose (68) on the left-hand helium turret.



## 13 Final stages

You should conclude the helium filling procedure by checking whether the magnet system works correctly again and recording the filling procedure.

### 13.1 Recording the Filling Procedure

The filling procedure replaces the helium lost through evaporation. If the filling procedure is accurately recorded, the average consumption can be estimated. Significant changes in helium consumption is an early warning signal that the magnet system is not in order.

1. Determine the quantity of helium left in the transport vessel (either by weighing, or measuring the fluid level with a dip-stick).
2. Record the date and quantity used on the magnet system and on the transport vessel.
3. Record the helium level before and after the filling procedure.
4. Make sure the nitrogen outlets are correctly fitted with a check valve and if applicable remove the rubber stopper from the outlets.



**Caution**

**Danger of magnet system bursting - This can occur if you forget to remove the rubber stoppers from the nitrogen outlets. After conclusion of the refilling procedure, it is essential that you remove the rubber stoppers.**

5. If the magnet system is standing on vibration dampers: Activate the dampers by turning the switch on the base plate to the "up" position.

### 13.2 Control Checks

After the filling procedure has been completed, carry out the following checks to ascertain whether the magnet system functions correctly.

6. After the helium filling procedure, make sure that the U-tube thaws out. You can help this process by carefully using a fan if necessary.
7. After a few hours, check that evaporating nitrogen is exiting from the nitrogen outlets.



**Caution**

**Danger of magnet system bursting - This can occur if the nitrogen outlets are blocked through icing up. Make sure that the nitrogen outlets are free.**



**Note**

If after a few hours, evaporated nitrogen is not escaping, the cause could be iced up nitrogen turrets. Check that they are free according to 5.2 "Preparing the Magnet System", page 11.



**Caution**

**Never try to remove ice from the nitrogen turrets without prior contact with your local Bruker Service Department.**



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## Appendix

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### 14 Important terminology

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In this section, the meaning of the most important terminology is explained, in the form of a glossary.

<b>Anti Vibration Feet</b>	Air cushioned damper elements on or in the magnet stand
<b>Cryogenic fluid</b>	Super-cooled gas in fluid form
<b>Dip Stick</b>	Long metal tube with a round end-piece for measuring fluid level
<b>Epoxy Rod</b>	Long fibre glass stick with a diameter of around 1 mm for measuring fluid level.
<b>Flame</b>	Visible appearance of liquid helium escaping from a pipe.
<b>Helium</b>	Light, colorless, odorless and tasteless gas with the chemical formula "He". Liquid helium has a temperature of -269°C
<b>Helium level meter</b>	Measuring device for indicating the fluid level in the helium tank.
<b>Helium level probe</b>	Measuring probe for determining the fluid level in the helium tank
<b>Helium oscillation damper</b>	Part of the U-tube for suppressing thermo-acoustic gas oscillations.
<b>Magnet System</b>	Super-conducting magnet which stands in a tank of liquid helium, surrounded by a second isolation tank containing liquid nitrogen.
<b>Manometer</b>	Gas pressure measuring device.
<b>Nitrogen</b>	Heavy, colorless, odorless gas with the chemical formula "N <sub>2</sub> ". Liquid nitrogen has a temperature of -196°C
<b>N2 Flow System</b>	Assembly on the magnet system, which releases evaporated nitrogen into the atmosphere and at the same time prevents air and moisture from entering the nitrogen tank.
<b>Quench</b>	Very fast discharging of the magnet due to loss of its super-conducting properties.





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A quench happens when the stored magnetic energy is converted to heat, due to loss of super-conductivity. The heat produced results in rapid evaporation of large quantities of helium and nitrogen.

<b>Quench valve</b>	Pressure release valve with large outlet capacity.
<b>Siphon</b>	Metal regulator with tube in the magnet system, which reaches to the floor of the helium tank.
<b>Transfer Line</b>	Vacuum isolated transfer pipe for liquid helium.
<b>Transport vessel</b>	Vacuum isolated vessel for the transportation of liquid helium or nitrogen.
<b>U-tube</b>	Connecting piece which connects the outlets of the two helium turrets.

## 15 Warning Signs / Pictograms



**Caution:** Extremely high magnetic stray fields.

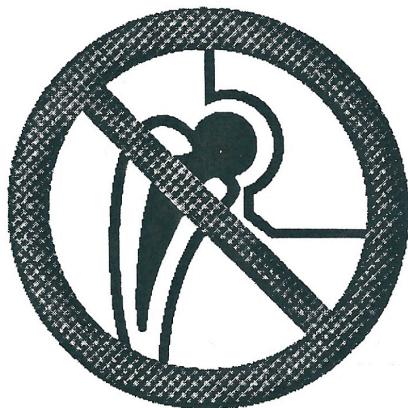


**Danger:** No entrance for people carrying pace-makers.



UltraShield™ NMR Magnet System

Refilling Procedure



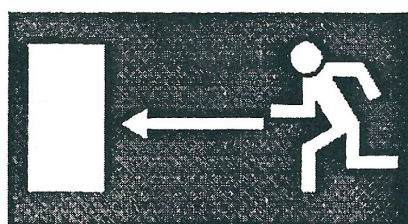
**Danger:** No entrance for people carrying medical implants.



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



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



**Note:** Exit in case of emergency.



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