



**Important:** When refilling cryogenic liquids the magnet should not be left unattended. Protective clothing, including leather or PVC gloves and eye protection should be worn at all times. Liquid Nitrogen (LN<sub>2</sub>) spilling out of the magnet filling ports could fall onto the top or bottom magnet flange. The risk is that of the O-rings freezing and the vacuum level decreasing to the point of a magnet quench, making a new installation of the magnet necessary.

### Checking and Refilling Liquid Helium

5.3.1

The Helium vessel should be checked weekly for boil off and helium level. Use a helium flow meter or a helium gas counter. These values should be recorded (see "Magnet Refill Record" on page 55). If the boil off falls to zero for a period greater than 24 hours, the tower tubes should be checked for the presence of ice. The procedure for checking the tower tubes and removing any blockage should be attempted only by a trained technician with considerable experience on cryogenic systems.

It is recommended that you refill the helium vessel within the specified hold time period and certainly before the level falls below the allowed minimum level. Refer to the Magnet System User Manual (Table 5.1) that is delivered with the magnet for details.

#### Guidelines for checking the Helium level

- Routinely (at least once per week) check the helium level gauge with help of the B-SN 18, BSMS or BSMS/2 electronic measuring devices.
- When the electronic measurement is not possible due to a malfunction, you can check the helium level with help of the dipstick or otherwise contact the nearest Bruker Service Representative.
- The measured value should be recorded in a table or graph, for example "Magnet Refill Record" on page 55.
- A software tool is also available, "helevtransfer" to automatically check and record the helium level.
- A regular greasing of the O-rings at the LHe refill neck tube is recommended.

#### Refilling Liquid Helium (LHe)

When refilling liquid helium the following safety points should be observed:

- Leather or PVC gloves and eye protection should be worn at all times.

## Maintenance Procedures for Cryomagnets

- The **refill opening** of the helium dewar **should not be left open** for extended periods of time, as this may result in excessive icing of the magnet dewars.
- When refilling the He-dewars a **maximum pressure of 0.25 bar** should be used.
- During the transfer the **helium transfer line should not be allowed to ice up**, as then only helium gas will reach the magnet, which may result in a magnet quench. When the transfer line begins to ice up, the refill must immediately be stopped and the transfer line evacuated or exchanged.
- The **O-ring** sealing the syphon entry port should be checked approximately 10-20 minutes after every transfer, once the ice buildup on the towers has defrosted. The **helium vessel should never be left open** to atmosphere for more than 5 seconds.
- Check that there is a gas flow through the flow meter after the refill of helium.



**Refer to the Users Manual for UltraShield NMR Magnet Systems (Table 5.1.) for complete details on refilling and safety aspects when refilling!**

The LN2 and LHe refill procedures are the same for Standard and UltraShield Magnets.

Table 5.1. Nitrogen/Helium Refill Procedures

Refill Procedure	Document	Part Number
Liquid Nitrogen LN2	Users Manual for UltraShield NMR Magnet Systems	Z31326 (English) Z31320 (German) Z31366 (French) Z31367 (Italian) Z31368 (Spanish)
Liquid Helium LHe	Users Manual for UltraShield NMR Magnet Systems	Z31326 (English) Z31320 (German) Z31366 (French) Z31367 (Italian) Z31368 (Spanish)

To obtain a copy of these manual please visit our Switzerland Website at <http://www.bruker.ch/magnetics.html> or contact your nearest Bruker Service Representative for a copy of these manuals.

## General Spectrometer/Magnet Maintenance



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The transfer of cryogenic liquids should be stopped immediately when the vessel is full. Failure to observe this can lead to the freezing of O-rings and a subsequent vacuum loss of the NMR magnet system, which may result in a magnet quench.

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### Checking and Refilling Nitrogen

5.3.2

The **Nitrogen vessel** should be **checked daily** for boil off and nitrogen level. These values should be recorded. If the boil off drops to zero, the filling and exhaust ports should immediately be checked for the presence of ice.

The Nitrogen vessel will normally need to be **refilled every 7-10 days**. When the vessel is being refilled, liquid nitrogen should not be allowed to spill onto the room temperature bore closure flange. Use teflon tubes on the nitrogen filling ports during refill.



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**Over-pressured LN2 should never be used for refilling nitrogen. A maximum pressure of 0.5 bar or less must be used!**

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### Refilling Liquid Nitrogen

- Leather or PVC **gloves and eye protection** should be worn at all times.
- When refilling the LN2-dewars a **maximum pressure of 0.5 bar** should be used.
- Generally, liquid nitrogen should be filled **once a week**. This will enable you to experience on how long each refill takes.
- After the refill, check that the nitrogen filling ports are free of ice.

When you notice that the refill takes longer than normal it is quite possible that an **ice stricture has formed in the LN2 refill neck**. This stricture can be removed quite easily with a 6 mm diameter plastic or fiberglass rod. This rod should be ap-

prox. 50 cm long and must be secured to prevent it falling into the N2 dewar. The rod should be inserted deep into the stricture until it begins to enter the N2 dewar.



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Refer to the Users Manual for UltraShield NMR Magnet Systems ("[Nitrogen/Helium Refill Procedures](#)") for complete details on other safety aspects for refilling.

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### **Moving an NMR Magnet System after Installation**

5.3.3



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**Do not shift or transport the NMR magnet system after installation! Transportation without a transport fixture may lead to damage or even destruction of the NMR magnet system!**

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## **Adjustments**

5.4

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The following are adjustments that may need to be made **periodically** to optimize instrument performance.

### **Frequent Routine Adjustments**

5.4.1

#### **Locking and Shimming**

**Before running an NMR experiment**, it is necessary to optimize the homogeneity of the magnetic field. This is done by a procedure commonly referred to as "**locking and shimming**". Refer to the **Avance Beginners Guide** (P/N Z31633) for instructions on how to perform locking and shimming.

#### **Tuning and Matching**

Each probe is fitted with as many resonant circuits as there are nuclei indicated on the probe label (e.g., one for <sup>1</sup>H and one for <sup>13</sup>C in a dual <sup>1</sup>H/<sup>13</sup>C probe; one for <sup>1</sup>H and one for a wide range of nuclei in BBO or BBI probes). A resonant circuit for the lock nucleus is also fitted, even though the standard user will never need to adjust it. Each of the circuits has a frequency at which it is most sensitive (the resonance frequency). **Once the sample is inserted, the probe should be**