SPECTROSPIN
NMR Magnet System

SPECTROSPIN NMR Magnet System
Important Notes

Version 002

BRUKER

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2 Important Notes

2.1 Important Notes

Read this first!

Please read the following pages carefully before installation or operation of your NMR magnet system:

- Hazards associated with superconducting NMR magnet systems
- Cryostat installation
- Magnet charging procedure
- NMR experiments at extreme temperatures

2.2 Hazards

Hazards associated

The high stray field in the vicinity of a superconducting NMR magnet system is potentially dangerous. This tends to be overlooked as it is belied by a relatively harmless appearance.

Attractive forces

The stray field will exert an enormous attractive force on any iron or magnetic object in the immediate vicinity of the NMR magnet system, transforming such objects into projectiles capable of causing considerable harm to persons in the way and of course, the magnet and the objects themselves. Special care is essential in the vicinity of high field or large bore magnets because their attractive forces are effective even at rather large distances from the cryostat.

Electronic devices

It must also be realised that strong magnetic fields may adversely affect a range of electronic devices including those directly controlling or assisting human vital functions, e.g. cardiac pacemakers. Great attention must be paid to this possible hazard as even comparatively low magnetic fields may prove dangerous in this respect.

In conclusion, for the reasons outlined above, it is essential to declare the area around the NMR magnet system a hazardous zone and to limit the access. Detailed recommendations for this particular NMR magnet system are given in chapter 3 of this manual.

2.3 Cryostat Installation

Cryostat installation

Cryostat installation must be done by experienced cryogenic engineers only.
The vacuum chamber of the cryostat was closed under nitrogen atmosphere in order to avoid penetration of moisture and impurities. Please do not open the vacuum valve unless necessary.

Specified loss rates

This is an ultra low loss cryostat. Because of its excellent thermal insulation it normally takes a very long time (often several weeks), before the internal radiation shields have cooled down to their operation temperatures. During this cooling down period the helium losses can be considerably higher than specified.

In order to accelerate this cooling down process we recommend transferring liquid helium in stages at intervals of 3 to 4 days into the helium can. Each transfer will overcool the neck tubes and in this way help to remove the heat from the radiation shields.
The cryostat helium vessel must always be closed with a one way valve when cold.

Important

Cryostat warm up must be done exactly according to the procedures outlined in chapter 3 of this manual. Fast warm up by breaking the vacuum should be done by **experienced cryogenic engineers only.**

Helium recovery systems

If a helium recovery system is used, this system must be connected only with a special assembly available as an accessory from Bruker/ Spectrospin. Failure to do so or modifications of the assembly will terminate the magnet system warranty, due to high quench risks.

2.4 Handling of Superconducting NMR Magnet Systems

Magnet charging procedure

This magnet is operating at a very high field. It is possible that during the charging procedure a few training quenches may occur to accommodate the stress changes in the magnet. These quenches are usually harmless, however they must be considered as a temporarily high thermal and mechanical strain to the magnet and should be avoided as far as possible.

Therefore we recommend that the NMR magnet system is kept on field permanently, even during longer periods where no measurements are performed, such as vacation times.

Z and Z^2 Shims

Normally, the magnet is equipped with a superconducting Z shim and Z^2 shim. **The Z and Z^2 shim heaters must be permanently "ON" during the charging and discharging of the magnet to avoid quenching the magnet.**

Z^2 Shift

The magnet should be charged above or below the nominal field to compensate the Z^2 shift (some kHz) generated by the Z^2 shim.

Persistent mode

The use of the shorting connectors for permanent magnet protection in the persistent mode is described in chapters 3 and 5 of this manual.

Magnet shimming

Before starting the shimming procedure itself, set the shim currents as stated in the characteristic data table. Due to the high efficiency of the cryo shims, they are influencing strongly the magnetic field of the magnet.

Important

Change shim currents slowly.

Cycling of shims

If setting of cryo shims causes irreversible frequency shifts, cycling of shims helps to get stable conditions within the magnet system. Cycling of shims is essential for 600 MHz NMR magnet systems!

2.5 Special NMR Experiments

NMR Experiments at extreme temperatures

This spectrometer is designed to make longtime experiments at the specified lowest and highest probe temperatures. However, when preparing and running such experiments, some care must be given to avoid excessive temperatures on the magnet flanges and O rings.

For high temperature experiments, adequate cooling has to be provided to the cooling lines of the probehead.
For epoxy shim systems, adequate air cooling between the shims and the magnet bore tube has to be permitted. For low temperature experiments, cooling losses to the magnet bottom flange have to be avoided.

A good indicator of cooling losses during low temperature experiments is the formation of ice at the lower part of the probehead and its connection to the cold gas supply. A small cover of ice is normal, however this cover should not exceed 1 cm in thickness.

It must not reach the magnet's bottom flange. The following parts have to be checked if excessive ice formation is observed.

1. Nitrogen transfer tube (N_2 supply to the probehead): Evacuate (stainless steel version) or replace if necessary (glass version).
2. Vertical transfer dewar within probehead: Replace if necessary.
3. Probehead dewar (around probe and receiver coil): Replace if necessary.
4. Dewar seal on probehead dewar: Seal with teflon tape if leaky.

In extreme cases, excessive cooling losses may lead to a cool down of the magnet bottom flange. In this case the cryostat vacuum seal may get leaky and the magnet will quench due to helium loss. To prevent this, the low temperature experiment must be interrupted before the cryostat bottom flange reaches the freezing point. The bottom flange must be allowed to warm up. The reason for the cooling losses has to be eliminated before the experiment can be continued.

2.6 Discharging and Warming Up of the NMR Magnet System

Magnet discharging procedure

Spectrospin magnet systems are equipped with an internal diode for switch protection and discharging.
Before starting the discharging of a NMR magnet system, be sure to have enough helium in the helium vessel.
Prepare the cryo power supply and all cabling before extracting the shorting connector from its place.
Discharge the magnet according to the discharging rates in chapter 3 of this manual. **The Z and Z2 shim heaters must be permanently "ON" during discharging of the magnet.**

Magnet system warming up procedure

After having discharged the magnet coil, blow out the remaining cryogenic liquids from the helium vessel and the nitrogen vessel before breaking the vacuum in the vacuum chamber!

Important

Cryostat warm up must be done exactly according to the procedures outlined in chapter 47 of this manual. Fast warm up by breaking the vacuum should be done by **experienced cryogenic engineers only.**

Breaking the vacuum

The use of nitrogen gas to break the vacuum is **essential**. Never use helium gas to flood the vacuum chamber - the super insulation would be irreversibly contaminated with helium gas!