NMR Magnet User Manual

AS 400/54 Family Type 3

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Contents

This User Manual details the correct use of your equipment and shows you how to contact Q.One Instruments for assistance. Please take time to read this guide to ensure that we can help you quickly whenever you require it.

Welcome

Thank you for choosing your equipment from Q.One Instruments, a company dedicated to providing first-class products and customer support. Our highly trained teams are available to help you with all your queries relating to your order, delivery or technical issues.

As a Q.One Instruments customer, you have access to a first-class service and support package providing telephone and on-site technical and repair services. In the unlikely event that your product should require repair, our technicians will initiate service under the terms of your Q.One Instruments warranty.

At Q.One Instruments we know that your expectations are at the highest level. We aim to meet and exceed those expectations in the service that we provide, and in the quality you will see when you use Q.One Instruments equipment.

We are delighted you selected Q.One Instruments as your supplier and wish you success with your new equipment.

Q.One Instruments Customer Support

The Q.One Instruments customer support warranty is available to all our customers during the first 12 months of ownership from date of delivery. This warranty provides:

- Specialist installation including project management and help with site and environment planning and logistics for complex installations
- Repair to faults which are a result of manufacturing defects
- Re-installation following a repair under warranty.

To obtain technical support you will need to quote your Q.One Instruments delivery tag number. Please contact your nearest Customer Support centre as follows:

	Telephone	E-mail	FAX
Main Office	+86-27-81773999	service@qone-inst.com	+86-27-81773999

Additional services available from Q.One Instruments

Training Solutions – a range of in-house and off-site training packages are available to cover topics including system maintenance. Please call us on +86-27-81773999 to find out more.

Q.One Instruments provides one-stop shopping for cryogenics, magnetic, vacuum and associated laboratory products as well hard to find reference for low temperature physics, optics, thermometry and laboratory safety practices. Ordering parts is easy via phone, fax or email.

Helium top-up service – available in certain territories. Ask your sales person for details.

About this Manual

This manual contains technical information pertaining to your equipment. It also contains reference information and includes details of your key contacts at Q.One Instruments who are available for help on repair matters, service and training. Please keep it close to your system.

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Chapter 1

Safety

The magnet may be operated and the cryogenic liquids handled with complete safety.

Safety procedures are vital to prevent
- Serious injury or death
- Serious damage to the equipment.

All staff, including cleaning and maintenance staff, who work in the NMR magnet room must read the Q.One Instruments booklet Safety Matters which accompanies this manual.

This section specifies on-site responsibilities.

Responsibilities

Senior Engineer

The senior engineer has responsibility for all aspects of safety on site.

1. No one may work in the magnet room alone under any circumstances. There must be at least two people working together.
2. Access to the site must be controlled.
3. There must be an oxygen monitor operating in the magnet room.
4. An illuminated sign at the entrance to the magnet room must come on automatically whenever the magnet is energised.
5. Health and safety procedures must be observed.
6. Anyone handling cryogenic liquids must be fully trained.
7. The safe operating conditions for all equipment must be understood and the suppliers' manuals kept in the control area. Safe operation relies on following procedures in the *Regular Checks* chapter.
8. Everyone either using the magnet or who regularly enters the magnet room or works within the region of the magnetic field must be trained in the safety procedures. This includes cleaning and site maintenance personnel.
9. Any visitor must be informed of the risks and of the evacuation procedures. A trained person must accompany them throughout their visit.
10. Within the 5 gauss line clear notices must warn of the possible presence of magnetic fields and of the potential hazards.

A stray field map, including the 5 gauss line, is provided in the *System Data* section of this manual.

In unforeseen circumstances or if any doubt arises over the correct procedure contact Q.One Instruments Magnetic Resonance Customer Support.

All Staff

1. No one may work in the magnet room alone for any reason.
2. Staff must observe all safety procedures, as specified in *Safety Matters*, when in the magnet room.
3. No one may operate or use the magnet system without training and permission from the Senior Engineer.
4. No one may handle cryogenic liquids without training and they must always follow the correct procedures.

Safety symbols used in this manual

Symbols are used in this manual to draw your attention to safety procedures that you must follow to protect yourself or the equipment.

Full details of the safety precautions required when working with cryogenic liquids and superconducting magnets are given in the *Safety Matters* booklet which accompanies this manual.

There are two levels of hazard

- A warning indicates a risk to people
- A caution indicates a risk to equipment.

The yellow warning triangle highlights dangers which may cause injury or, in extreme circumstances, death.

The text explains the hazard and the correct procedure. The warning triangle may be followed by specific symbols and instructions.

A white symbol in a blue circle indicates something that you must do.

The general caution symbol highlights actions that you must take to prevent damage to the equipment. The action is explained in the text.

This symbol indicates that loose fitting, insulating gloves should be worn, suitable for protection against splashes of liquid helium and nitrogen.

This symbol indicates that protective goggles or (for cryogenic use) a face mask should be worn.

This is the symbol for protective earth.

A text box draws your attention to things you should know.

Cryogenic liquids

Spilt liquid helium or nitrogen from, for example, a leaking storage dewar, may cause injury or, in large volumes, death by asphyxiation. Atmospheres containing less than 18% oxygen are potentially dangerous and entry into atmospheres containing less than 20% is not recommended.

Use a portable oxygen monitor such as the GasAlert™ to provide a visible and audible warning of low oxygen level.

Ensure that all vessels containing cryogenic liquids are provided with a means to vent safely and a separate safety valve.

For general information on the safe handling of cryogenic liquids please refer to the document *Safety Matters* that accompanies this manual.

Safety devices are coloured red and should never be modified or obstructed.

All cryostats are provided with Safety devices to protect operating personnel in case of system failure. These are clearly identifiable as they are red in colour.

NMR Magnet Quench

In the event of a magnet quench (either spontaneous or induced) the magnetic field will rapidly collapse. This may affect objects present in the room.

In the event of a magnet quench all the cryogens may be released. The volumes of gases at room temperature will be approximately 70 m^3 for every 100 litres of cryogen (helium and nitrogen). Appropriate data for individual systems is given in the* System Data *section of this document.

Use this data to calculate the worst case oxygen depletion for the room containing the NMR system.

For general information on ventilation issues please refer to the document *Safety Matters* that accompanies this manual.

Electrical Safety

Earth (ground) all equipment, including the cryostat and electronics.

Superconducting magnets usually have high inductance and operate at high currents. A large amount of energy is therefore stored in the magnet when it is at field.

where L is the inductance of the magnet and I is the current in the magnet.

If a magnet quenches a voltage is induced of magnitude

Since it is not uncommon for the current to decay at 100 As^{-1} it is possible to generate voltages of tens of kilovolts.

Protective devices are fitted to the system in order to protect users and the equipment from these hazards.

Do not disconnect the protective earth for any reason once the magnet is carrying any current.

For general information on electrical safety issues please refer to the document *Safety Matters* that accompanies this manual.

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General

- If the equipment is not used in the manner specified by Q.One Instruments Magnetic Resonance then the protection provided by the equipment may be impaired.
- The equipment is not suitable for use with explosive or flammable gases.
- The equipment is not suitable for use in explosive, flammable or other hazardous environments.
- Maintenance: only qualified and authorised persons should carry out servicing and repair work on this equipment.
- Only use genuine Q.One Instruments Superconductivity spare parts. Contact Q.One Instruments Magnetic Resonance Customer Support to obtain these.

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Chapter 2

Introduction

Description of the system

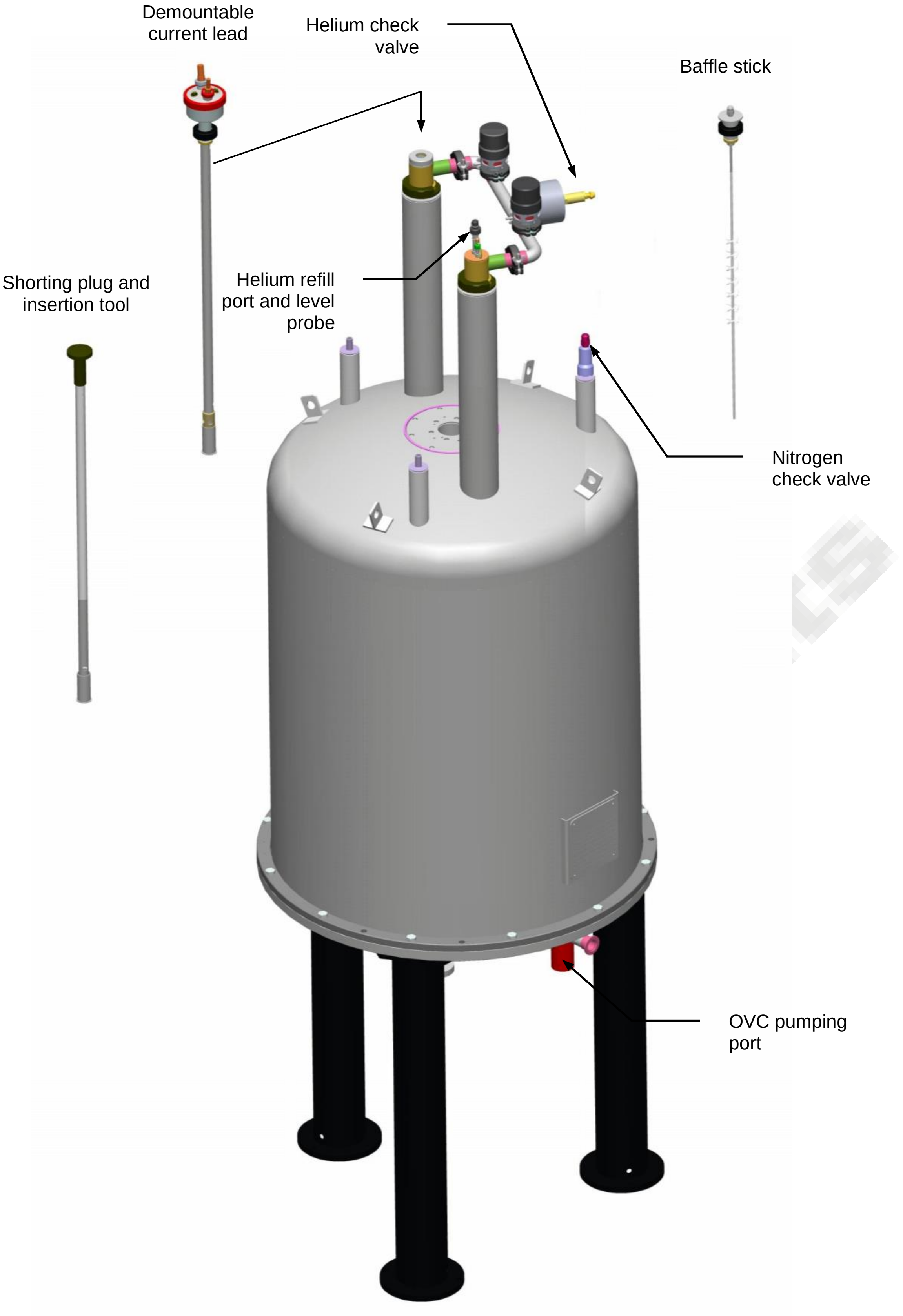
This Q.One Instruments Magnetic Resonance NMR magnet system comprises a fully persistent superconducting magnet in a low loss “Type 3” cryostat with a room temperature bore.

System designation	Room temperature bore (mm)	Central field (Tesla)
AS400/54	54	9.39

The high current needed to generate this field can only be sustained by immersing the magnet in a bath of liquid helium at 4.2 K, at which temperature the resistance of the magnet is zero. To keep the rate of helium boiloff as low as possible this vessel is shielded from room temperature by a Gas Cooled Shield (GCS) and a vessel filled with liquid nitrogen.

Use Figure 1 and Figure 2 to identify the principal components of the system.

The full list of specifications and other product data are listed in the System Data section of this document.


Demountable
current lead
Helium check
valve
Baffle stick
Shorting plug and
insertion tool
Helium refill
port and level
probe
Nitrogen
check valve
OVC pumping
port

The Magnet

The magnet consists of a number of concentric windings connected in series. The windings are manufactured using fully stabilized niobium titanium superconductor. The magnet is fitted with a superconducting switch so that it operates in true persistent mode. A set of superconducting shim coils is included in order to cancel some of the field gradients caused by winding inhomogeneities and tolerancing.

The superconducting switch consists of a length of superconducting wire and a heater. When the heater is on, the temperature around the superconducting switch rises until the switch becomes resistive and 'open'. When the heater is switched off, the temperature falls back to 4.2 K and the wire becomes a superconductor again, 'closing' the switch.

The magnet is initially energised from an external power supply with the superconducting switch 'open'. When the magnet reaches its operating current, the switch is 'closed' and the power supply gradually reduced. More and more current flows through the switch rather than the power supply. At zero amps, the power supply is removed entirely. The magnet is now in persistent mode with a high current circulating perpetually around a circuit with no resistance. The current leads are then withdrawn from the cryostat; this results in excellent field stability and very low consumption of cryogens.

If, once energised, the magnet's temperature is allowed to rise more than a few degrees above 4.2K, the coil will become resistive and generate heat, causing the liquid helium to boil-off very rapidly. This is called a quench.

Within the helium vessel, a number of low value high load resistors are connected across each section of the coil. In the event of a quench, these protection resistors prevent the development of high voltages that could cause the insulation of the windings to break down. Additionally, some of the energy released by the quench is dissipated through these resistors rather than the windings themselves, so also protecting the windings from damage.

The Cryostat

The helium and nitrogen vessels are suspended concentrically within an evacuated stainless steel container (Outer Vacuum Case or OVC) that provides room temperature access to the centre of the magnetic field.

The cryostat is equipped with pressure relief mechanisms and is therefore fully protected in case of vacuum failure or magnet quench.

At the top of the cryostat there are three ports (2 front and 1 rear) giving access to the liquid nitrogen vessel. One of these will be closed with a check valve that opens at about 150 mbar thus ensuring that the liquid nitrogen vessel can never be over-pressurised. Other ports allow gaseous nitrogen to vent freely.

There are two ports (left and right) giving access to the liquid helium vessel. The left hand neck provides electrical access to the magnet. The current lead assembly is demountable and normally removed after the magnet is energised and replaced by a simple baffle stick.

The right hand neck is used for topping up the liquid helium reservoir so contains the entry port for the helium transfer syphon. This neck is also fitted with a helium level probe.

Figure 1 also shows a set of legs that may be used to support the cryostat so that there is access to the magnet bore from below.

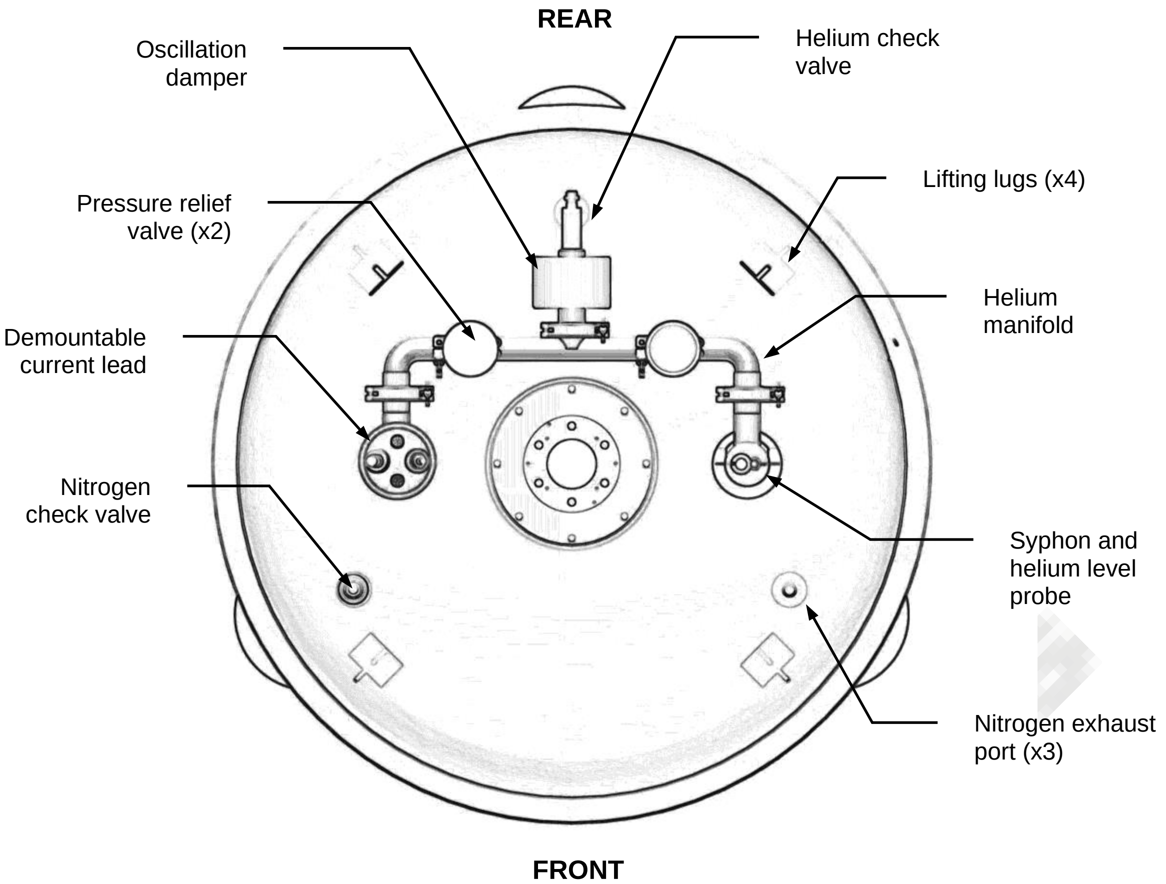


Figure 2 Top view of cryostat

Helium exhaust manifold

Helium exhaust gas from both necks is combined in a manifold at the rear of the cryostat. This manifold is fitted with two pressure relief valves (which open at 340 mbar above atmospheric pressure).

There is an “oscillation damper” fitted to the final exhaust point. This reduces the likelihood of thermo-acoustic oscillations, which can have a serious effect on the overall helium consumption and NMR performance.

Finally there is a one way (non-return) valve fitted to the oscillation damper. This allows helium gas out of the cryostat but prevents air getting in (which would freeze).

Accessories

A suitable set of accessories is supplied with all systems. This includes items for system maintenance such as:

- helium transfer syphon
- blow-out tube
- shorting plug for the magnet (and insertion/removal tool)
- baffle stick

The use of these items is fully described in the pages that follow.

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Chapter 3

Regular Checks

Objectives

This chapter describes regular checks that need to be performed by a trained member of staff. Use a system Log Book to record the results.

> You are advised to report unexpected results or errors to Q.One Instruments Magnetic Resonance (ZNMR) Customer Support. Use the final page of this section to ensure you have all the relevant information available.

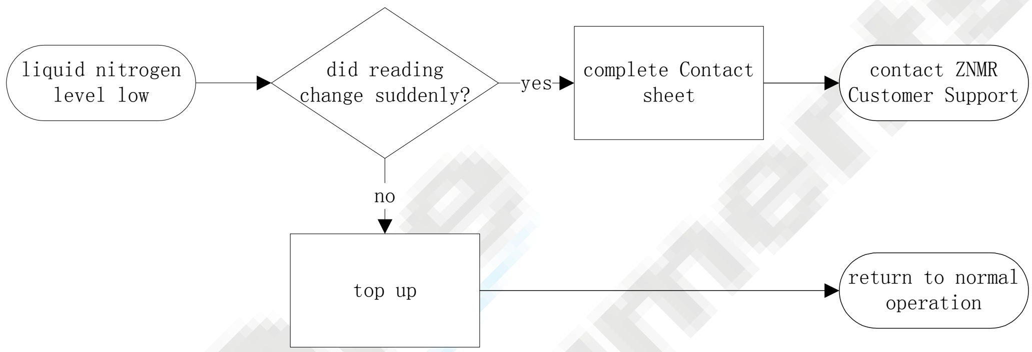
Liquid Nitrogen Level

1. Record the level of liquid nitrogen. Confirm that it is greater than the advised refill level (see *System Data*).

2. Note any alarm conditions that may be indicated.

3. Note any significant change in the rate of nitrogen consumption (increase or decrease).

Error procedure



Nitrogen Gas Flow

A check valve is fitted to at least one of the nitrogen ports so that it cannot be blocked by the entry of air or water, which will freeze. At least one other port will be open to allow nitrogen gas to vent freely (possibly outside the room or building).

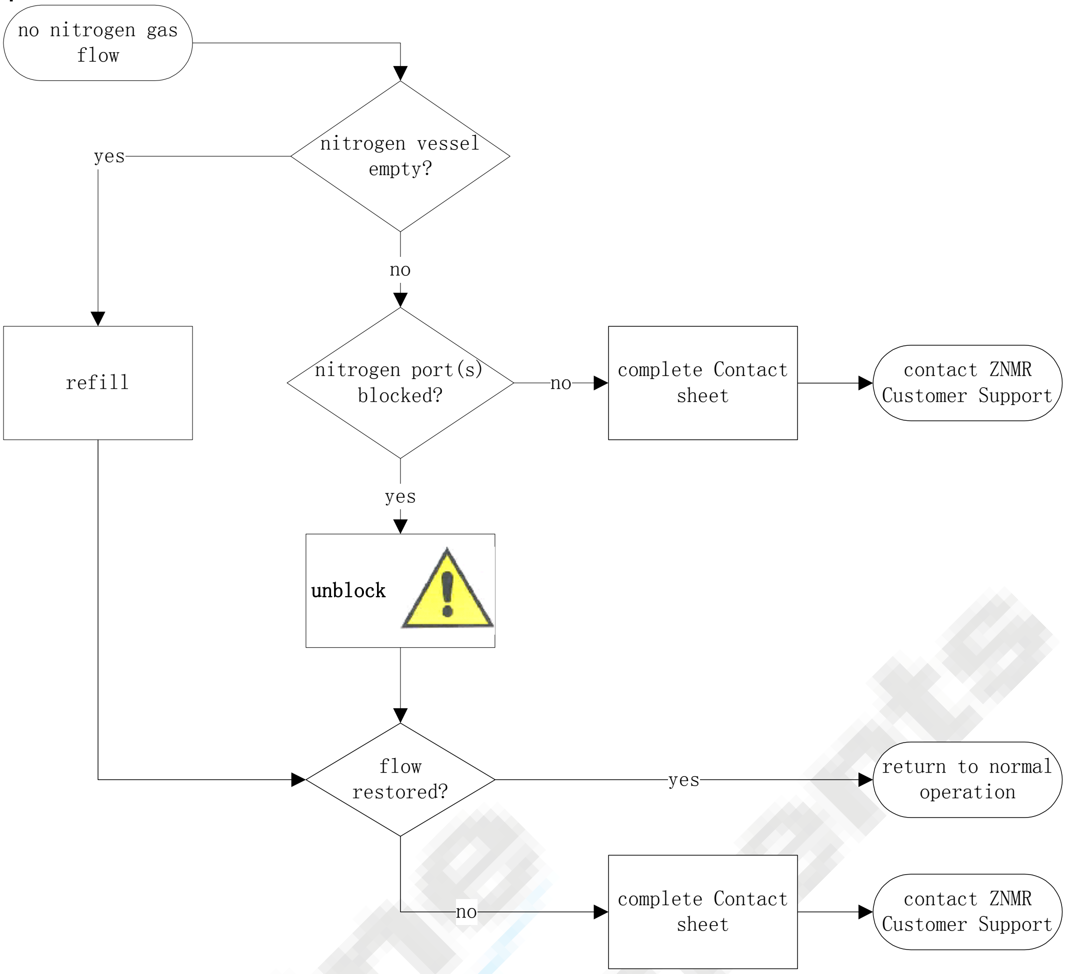
The check valve should never be removed unless advised (while the system is being filled with liquid nitrogen).

1. Check that nitrogen gas is flowing freely from the open nitrogen port(s).

2. After every nitrogen or helium top up take special care to check that gas is flowing freely from all nitrogen ports.

Wear loose fitting gloves and a face mask for this procedure.

Error procedure



Blocked nitrogen ports represent a potentially dangerous situation. It can lead to a severe build up of pressure, may rupture the nitrogen vessel and cause extensive damage to the system.

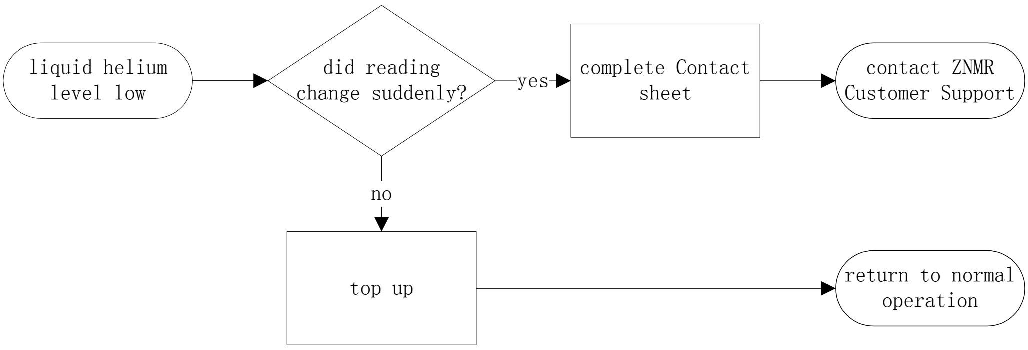
Liquid Helium Level

1. Record the level of liquid helium. Confirm that it is greater than the advised refill level (see *System Data*).

2. Note any alarm conditions that may be indicated.

3. Note any significant change in the rate of helium consumption (increase or decrease).

Error procedure



Helium Gas Flow

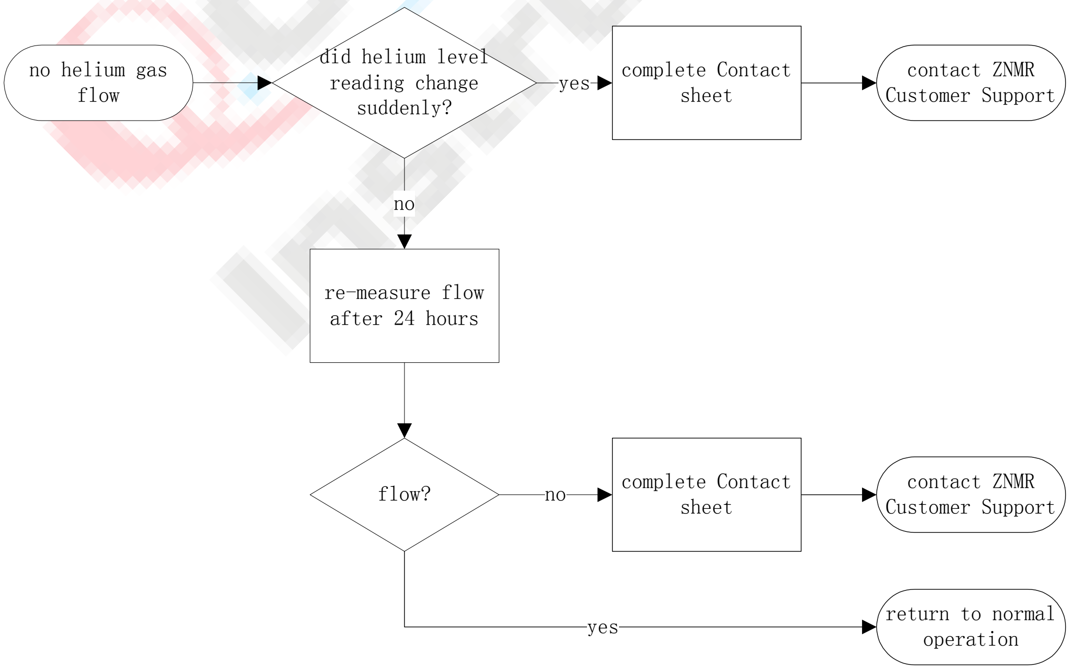
A check valve, sometimes with an oscillation damper assembly, will be fitted to the helium exhaust port so that it cannot be blocked by the entry of air or water, which will freeze. The helium exhaust from NMR systems may be allowed to vent freely into the atmosphere (possibly outside the room or building) or be connected to a helium gas recovery system.

The helium boiloff rate will be lower than normal for some hours after a helium refill.

The check valve and oscillation damper should never be removed unless advised while the system is being filled with liquid helium.

1. Check that helium gas is flowing freely from the open helium exhaust port.

Error procedure



Note that the specified helium hold time is based on the nominal refill volume and an average boil off rate measured under stable conditions of average pressure and temperature (typically 1010 mbar and 20 C). Lower atmospheric pressure causes the boiloff rate to increase and vice versa. However, these pressure induced changes in the boil off rate will average out to give the specified hold time. Normal changes to the ambient temperature will not affect the boil off.

ZNMR Customer Support Contact sheet		Please prepare answers to all the points below before contacting your nearest Customer Support centre
Identification tag numbers.		
Institute name.		
Local contact name, telephone number and email address.		
Associated NMR spectrometer.		
Installation date.		
Last helium fill or service visit.		
Last nitrogen fill.		
Date of most recent use. Has the system been idle?		
Describe environmental changes that have taken place. Has the room acquired any new equipment (particularly a magnet)? Has the building undergone any structural changes? Has air-conditioning been installed?		
What are the current % levels and flow rates?	helium	
	nitrogen	
Describe the symptoms.		

Chapter 4

Cryogen refill

Equipment required

- Personal protective equipment as detailed on the next page
- Liquid helium in a storage dewar (one large dewar is more efficient than several smaller dewars)
- Liquid helium transfer syphon
- A suitable storage dewar top fitting as a syphon adaptor
- A rubber bladder or a cylinder of 99.999% pure helium gas (UK size L) fitted with a 0 – 2 bar pressure regulator
- Liquid nitrogen in a storage dewar, ideally self-pressurising
- Liquid nitrogen transfer lines
- Standard dipstick
- Hot air gun
- A safe climbing platform as illustrated (not a ladder)

Objectives

This chapter describes how to refill (top up) the liquid nitrogen and liquid helium vessels.

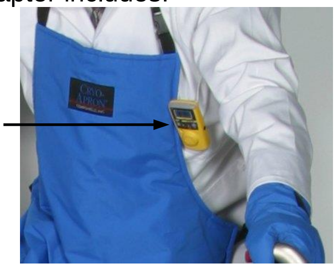
First steps

1. Please read and become familiar with the appropriate sections of the *Safety Matters* booklet before transferring cryogenic liquids.

Wear suitable personal protective equipment throughout these procedures

Personal protective equipment illustrated in this chapter includes:

- Cryogenic gloves
- Cryogenic apron
- Full face visor
- GasAlert™ personal oxygen monitor



2. Using the level meters (or a dipstick for helium) and the graphs in *System Data* calculate how much liquid helium (and nitrogen) you require to fill the vessels.

3. Use a dipstick to measure the depth of liquid helium in the storage dewar as shown in Figure 3 (the membrane vibration frequency is noticeably lower when the bottom of the tube is in liquid).

Figure 3 Using a dipstick to measure helium level

4. Use a helium dipstick and the chart supplied to confirm that you have enough liquid helium in the storage dewar.

> You can expect to use 20 litres of liquid helium just to cool the transfer syphon. Too high a transfer rate or a poor syphon vacuum will impair subsequent transfer efficiency. It is more efficient to transfer from one large storage dewar than from several smaller dewars.

Figure 4 Main items for helium refill: cryostat, climbing platform and storage dewar

Preparing the helium transfer tube (syphon)

The transfer tube consists of rigid “legs” that are inserted into the storage dewar and system cryostat. The legs are connected by a length of flexible stainless steel tube. Inside this runs the helium transfer tube itself. The entire assembly is vacuum insulated.

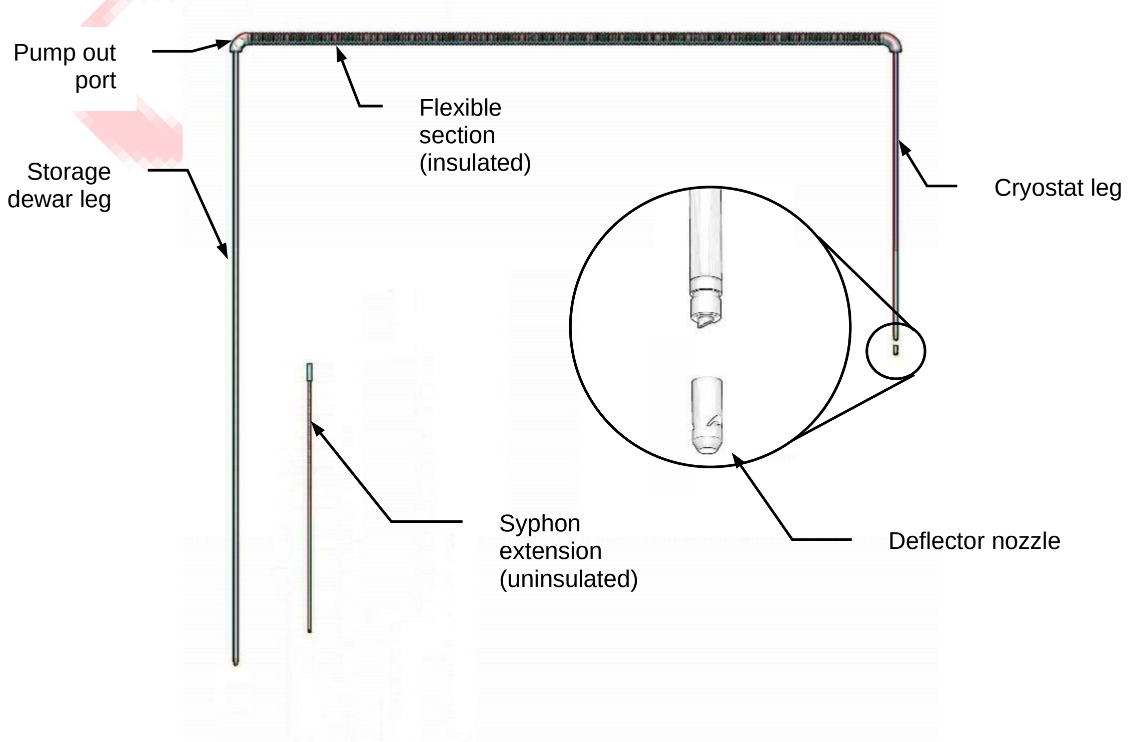


Figure 5 Typical helium transfer syphon components

1. Determine the distance from the top to the bottom of the helium vessel inside the storage dewar. This information will be supplied with the storage dewar. Include any dewar top fitting (Figure 9).

2. Choose and fit a syphon extension that will allow the storage dewar leg to reach to within about 25 mm of the bottom of the helium vessel. It should not touch the bottom.

Figure 6 Fit syphon extension to dewar leg

3. Fit the brass deflector (phase separation) nozzle to the cryostat (short) leg of the syphon as shown in Figure 7.

> The helium syphon delivers a mixture of liquid and gas. The function of the deflector nozzle is to encourage separation between the gaseous and liquid helium, allowing gas to vent upwards and liquid helium to fill the vessel.

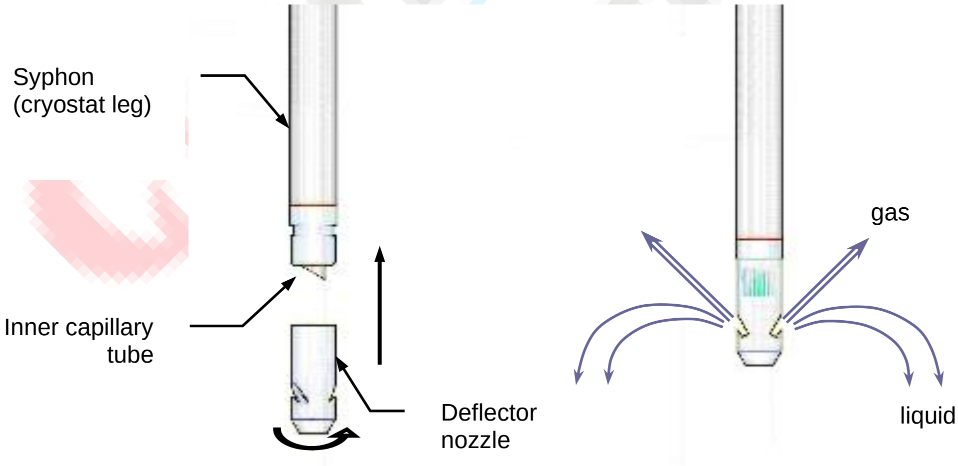


Figure 7 Fitting and function of deflector nozzle

4. Fit the knurled black ring, washer and O-ring from the spares kit over the short leg of the syphon (in this order), sliding them to the top.

Preparing the helium storage dewar

The valve configuration for safe transportation of a typical storage dewar is shown in Figure 8.

Figure 8 Storage dewar; transportation mode

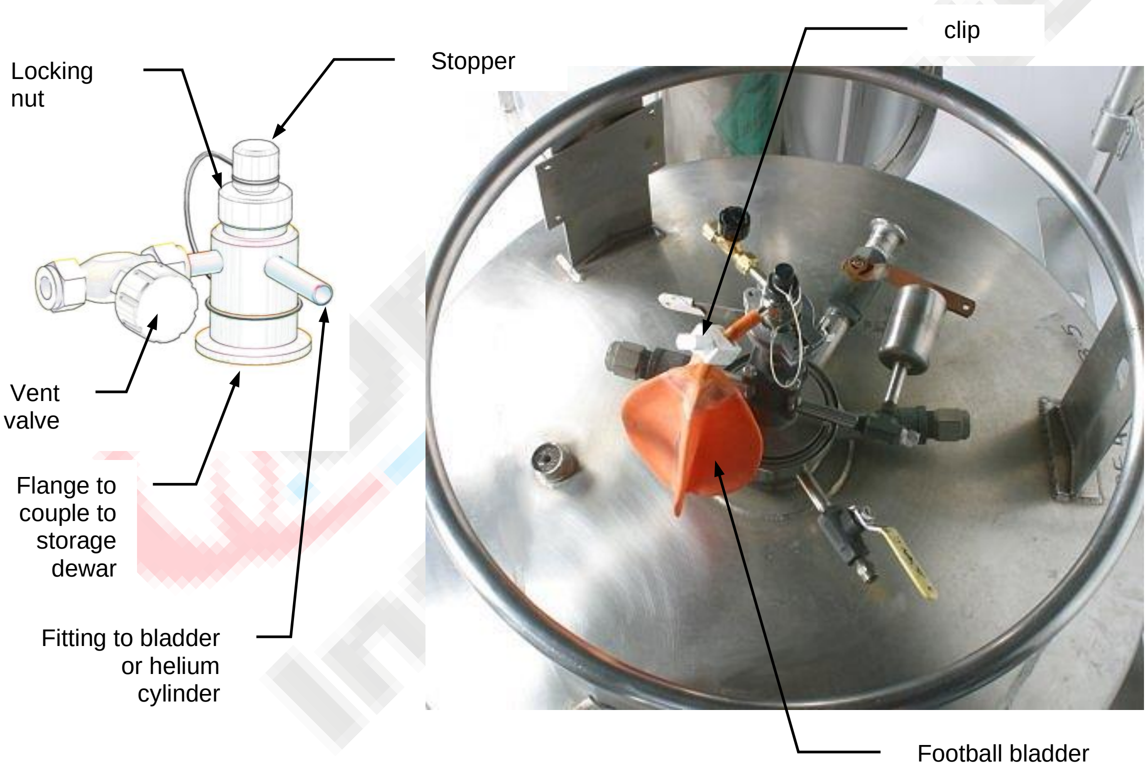


Figure 9 Storage dewar top fitting (left); fitted to dewar (right)

1. Fit a dewar top fitting to the top of the storage dewar as shown in Figure 9. Check that the small vent valve is closed.

2. Connect a helium cylinder (via the pressure regulator) to the port identified in Figure 9. Ensure that the set pressure is zero and the flow valve is closed. Alternatively connect a deflated rubber

bladder using a clip to close its neck (also shown in Figure 9). Some storage dewars are provided with a heater to promote helium boiloff.

3. Site the storage dewar so that when you insert the leg of the syphon into the dewar the other leg will reach the cryostat syphon entry port (Figure 10).

Figure 10 Siting of cryostat and storage dewar

4. Slowly release the pressure in the storage dewar by gradually opening the vent valve. For a large dewar this may take up to 30 minutes.

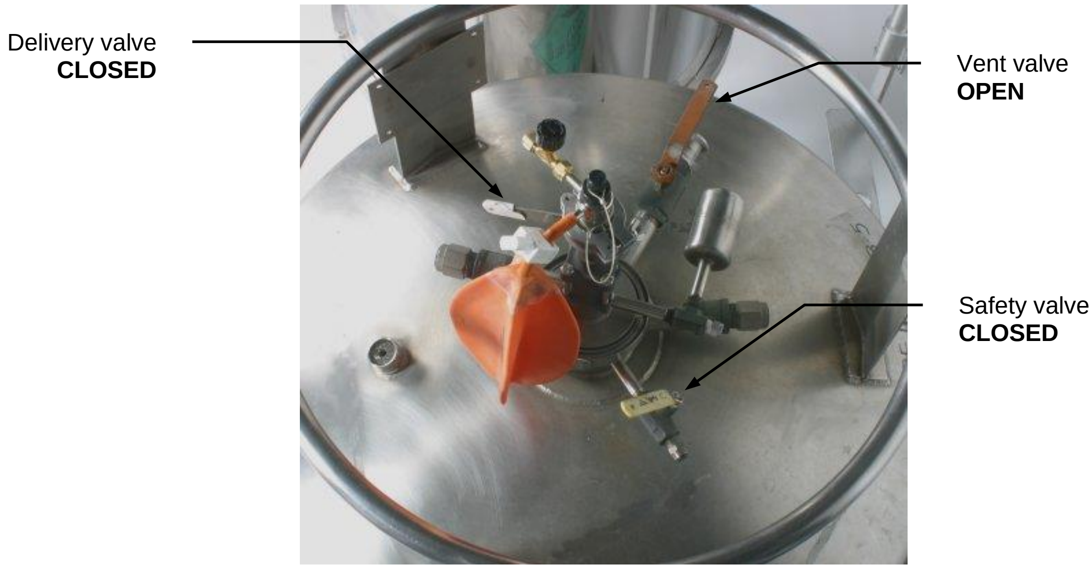


Figure 11 Storage dewar; vent mode

5. Close the dewar safety valve. The valve arrangement is shown in Figure 11.

Preparing the cryostat

Remove any helium check valve and oscillation damper (if fitted) from the exhaust manifold and connect the manifold to a helium recovery system.

Take care when releasing the cryostat pressure.

Do not use a recovery system if there is a significant back pressure. If a helium recovery system is not in use prepare a length of stainless steel tubing terminating in a high capacity check valve (ideally use flexible tubing 3 m long and 5 cm diameter; longer runs will require larger diameter tubing). This vent line should lead outside the building. Remove the manifold check valve and connect the line **when the transfer is about to start**.

Ensure that no air can enter the helium manifold

Transfer helium

1. Close the vent valve on the storage dewar.

2. Remove the stopper from the dewar top fitting.

3. Open the delivery valve on the storage dewar.

Keep clear. If the storage dewar has not been completely depressurised there will be a vertical jet of cold gas.

4. Insert the long leg of the syphon a little way into the dewar, just past the joint with the extension piece.

Figure 12 Inserting syphon leg into storage dewar

5. Tighten the locking nut on the dewar top fitting to the syphon leg to make a gas tight seal and allow the venting helium gas to purge the transfer tube.

6. Smoothly push down the syphon leg into the dewar.

If the leg does touch the bottom of the dewar it should be pulled back out by about 25 mm.

7. Set the pressure regulator on the helium cylinder to about 105 mbar (1.5 psig) and open the regulator flow valve fully. Alternatively, release the clip over the neck of the rubber bladder. A small amount of helium gas will expand the bladder. Compress this in order to force the (warm) gas into the dewar and this will boil off more liquid. When you release the bladder it will expand further. Repeat once or twice more. If the bladder is in good condition it is perfectly safe to allow it to expand to 50 cm diameter.

Figure 13 Sequence showing inflation of bladder

8. Take hold of the upper part of the syphon cryostat leg and hold it safely away from the body.

Take care. This part of the process sends jets of cold gas back along the syphon leg.

> Helium gas, initially warm, will issue as a jet from the cryostat leg. As the syphon cools this jet will become visible as a white cloud (Figure 14).

Figure 14 Pre-cooling the syphon

> After another minute this will change to a dense white “plume”(Figure 15) of liquid helium. The syphon is now cold enough. You must **not** attempt to transfer helium before this has occurred or you will inevitably force (warm) gas into the cryostat. This is bound to boil off some of the liquid helium already there and in extreme cases may cause the magnet to quench.

Figure 15 "Plume" of liquid helium

9. Release the helium pressure in the storage dewar either by opening the dewar vent valve (preferred) or the vent valve on the dewar top fitting (slow) and the jet will subside.

10. Remove the stopper, nut, washer and O-ring from the system syphon entry port, wipe any ice or air from the end of the syphon with a **gloved** hand and insert the syphon leg, pushing down until the leg reaches the cone. Then raise the leg by about 25 mm.

11. Tighten the black knurled nut to the leg to form a gas tight seal (Figure 16 – right).

12. Slowly and carefully push the long leg of the transfer syphon down into the liquid helium in the storage dewar (Figure 17).

The pressure inside the storage dewar will rise as warm parts of the syphon are pushed into the liquid helium in the storage dewar.

13. When fully inserted tighten the dewar top fitting to the syphon leg.

If there is no recovery system remove the check valve now and connect the manifold to the length of stainless steel tubing and high capacity check valve noted earlier. Ideally vent the helium gas outside the building.

Figure 16 Inserting syphon leg into cryostat and making a gas tight seal

Figure 17 Full insertion of syphon leg into storage dewar

14. As this pressure subsides maintain the pressure in the storage dewar at between 105 – 140 mbar (1.5 – 2.0 psig) using pure helium gas from the cylinder or inflate the bladder as described above.

Restrict the pressure to 35 mbar (0.5 psig) if the cryostat helium level is below 5% (Family cryostats)

During the transfer process water vapour will condense and freeze on the manifold and exhaust line and also appear in descending air that has been chilled below the dew point (Figure 18).

Figure 18 View from below the manifold showing water condensing in chilled air

When liquid (air) begins to drip from the manifold it indicates that the cryostat is full (Figure 19). The exhaust flow will also rise.

If frost on the recovery line extends beyond about 2 m the transfer rate is too high (inefficient) and the dewar pressure should be reduced.

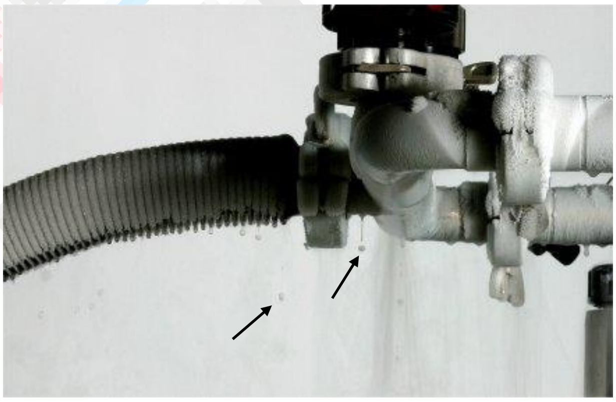


Figure 19 Cold manifold showing drips of liquid air

15. Close off the gas cylinder, if used, and release the pressure in the storage dewar by opening the vent valve.

16. If fitted, use your helium level meter to check the level in the cryostat. Alternatively, use a dipstick.

If the level is not high enough you may close the storage dewar vent valve, re-pressurise and re-start the transfer from step 14.

17. Undo the black knurled nut locking the syphon leg to the cryostat.

18. Lift the syphon leg from the cryostat in a smooth, continuous action.

Figure 20 Syphon leg just after removal from cryostat

19. Immediately replace the black knurled nut, washer, O-ring and stopper in the system syphon entry port (Figure 20).

20. Tighten the knurled nut to ensure a gas tight seal.

It is now important that the helium manifold and any other system top fittings remain sealed to prevent air entering and freezing in the system.

Restore the system

1. Loosen the black knurled nut locking the syphon leg in the storage dewar.

2. Lift the syphon leg from the storage dewar in a smooth, continuous action. Store the syphon safely.

3. Close the delivery valve on the storage dewar. Open the safety vent valve and close the main vent valve.

The storage dewar is now in a safe configuration for transport to another location.

4. Restore the helium exhaust manifold and recovery system to its normal configuration.

5. Use a dipstick to measure the depth of liquid helium remaining in the storage dewar as shown in Figure 3. Use the chart supplied to determine the volume remaining in the dewar.

6. Use a dipstick or helium level meter to measure the liquid helium level in the cryostat.

7. After every helium top up check that all nitrogen ports are free of ice blockages (which would occur within 25 cm of the top) by inserting a suitable tool such as a length of 6 mm diameter brass studding or glass fibre rod. An example is shown in Figure 21.

8. When the system top fittings are once more at room temperature check that all clamps and seals are secure.

Factors that can contribute to inefficient helium transfer

1. Using a transfer pressure that is too high will tend to force more liquid helium into suspension in the gas that passes directly to the exhaust manifold, not allowing sufficient time for it to collect. Using a transfer pressure that is too low will extend the overall time taken to fill the helium vessel. The ideal pressure is 105 – 140 mbar (1.5 – 2.0 psig).

2. If condensation or frosting appears on the surface of the transfer tube during or after cryogen transfer helium transfer efficiency will be impaired as this indicates a partial loss of vacuum. The transfer tube needs to be pumped again using the pump-out fitting supplied.

Is the storage dewar now empty?

Signs that the storage dewar has emptied include:

- Rapid deflation of the football bladder, if used
- An increase in the length of frost on the recovery line

You will have to stop the transfer process and remove the syphon. Ensure that the syphon is unblocked by blowing warm helium gas through it. Then re-start the transfer process with a full dewar.

Preparing the liquid nitrogen transfer lines

If the transfer is to be left unattended you must vent nitrogen exhaust outside the building or into an empty nitrogen storage dewar.

For liquid nitrogen transfer use only metal tubing connected by flexible metal hoses and PTFE seals. Rubber and plastic tubing may split, endangering personnel.

1. Two nitrogen ports will be open for fill and exhaust. Any others should be closed with nitrogen check valves.
2. Attach one nitrogen transfer line to the top of one of the open nitrogen ports using compression fittings.
3. Attach the other end of the line to the nitrogen storage dewar.
4. Attach the second nitrogen transfer line to the second open nitrogen port and trail the free end into an empty nitrogen dewar (or outside the building).

Route the transfer lines so that liquid air or nitrogen cannot drip on to a vacuum seal.

Top up liquid nitrogen vessel

1. Increase the pressure inside the liquid nitrogen storage dewar to about 150 mbar (2 psig) and allow nitrogen to flow to purge the lines.
2. Allow the pressure inside the storage dewar to increase to a maximum working pressure of 350 mbar (5 psig). Higher pressures will cause the relief valve to open.
3. Fill the nitrogen vessel until liquid nitrogen overflows into the empty nitrogen dewar.

> It is usually necessary to re-tighten the brass couplings after transfer begins and they cool down.

4. If a nitrogen level meter is present check that it reads 100%.
5. Release the pressure in the storage dewar and remove the transfer lines.
6. After every nitrogen top up check that all nitrogen ports are free of ice blockages (which would occur within 25 cm of the top) by carefully inserting a suitable tool such as a length of 6 mm diameter brass threaded rod or glass fibre rod. An example is shown in Figure 21.

> Take care as the nitrogen vessel is constructed using thin wall tubing.

Figure 21 Use of a simple tool to check for ice blockage in nitrogen port

Final gas flow checks

When the helium or nitrogen transfer is complete and you have restored the system to its normal configuration you must check that helium and nitrogen gases are flowing as normal. Refer to the relevant sections of the chapter *Regular Checks*.

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System Data

AS 400/54 (standard)

Mechanical data

Cryostat	Type 3
Cryostat Room Temperature Bore access (mm)	53.4 – 53.9
Bore access inside Room Temperature Shims (mm)	45
Nominal distance between cryostat base plate and magnet centreline(mm)	332 ± 5
Measured distance between cryostat base plate and magnet centreline(mm)	See Test Report
Minimum operational ceiling height (mm)	See drawings
System weight empty (Kg ± 20)	350
System weight full (Kg ± 20)	430

Cryogenic data

Liquid nitrogen required for installation (L)	400
Liquid helium required for installation (L)	500
Minimum helium refill volume (L)	72
Minimum helium refill interval (days)	183
Equivalent gas volume at 20 ℃ (L)	48.8
Minimum nitrogen refill volume (L)	78
Minimum nitrogen refill interval (days)	14
Equivalent gas volume at 20 ℃ (L)	47.3
Minimum helium level for magnet energisation (%)	100
Minimum wait after helium fill before magnet energisation (hours)	12
Minimum wait after quench and top up before magnet energisation (hours)	2
Minimum operating helium level – top up required (%)	10

Magnet data

Central field (Tesla)	9.39
Active shield	Yes
Field stability (^{1}H Hz/hr)	≤ 6
Vertical stray field 5 gauss contour (m)	1.25
Radial stray field 5 gauss contour (m)	0.8
Nominal current for central field (amps)	66.5 ± 2.5
Overfield current (amps)	Cycled (see energisation)
Nominal switch “open” resistance (ohms)	10
Nominal switch heater resistance (ohms)	100
Nominal switch heater current (milliamps)	60
Recommended power supply trip voltage (V)	4
Inductance (Henries)	100

System pre-cool table

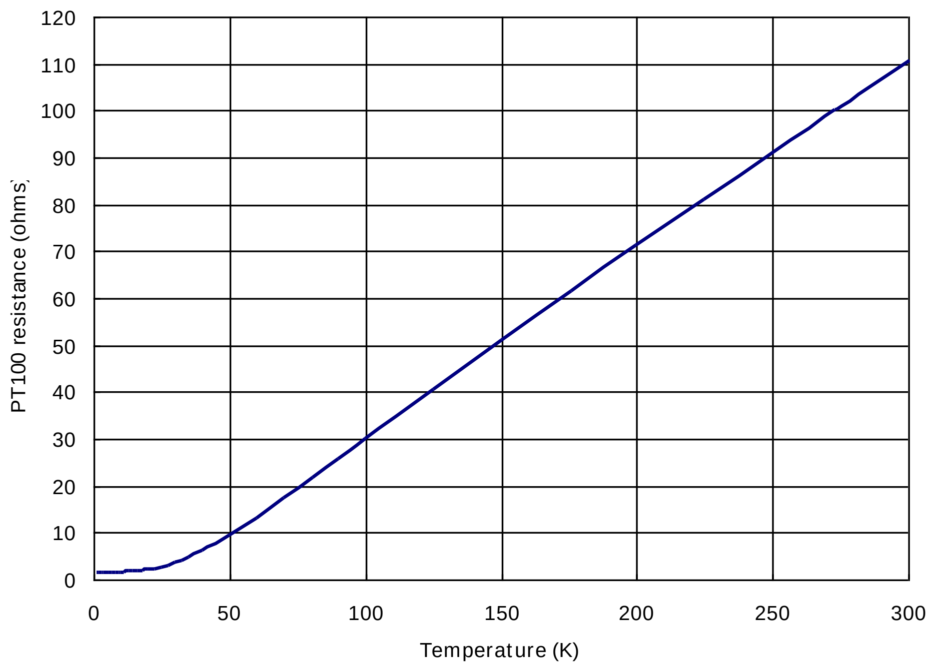
Nitrogen precool should take 10 -12 hours. After four hours the N2 cooldown unit is fitted and the nitrogen vessel refilled.
The PT100 readings should be below about 71 ohms (200 K) at the time of refilling.
Precool to 90 ± 5 K. (selected points below).
Stop LN2 transfer when PT100 reading is 26 ± 3 ohms.
Do not continue to LHe cooldown if PT100 resistance is less than 23 ohms.

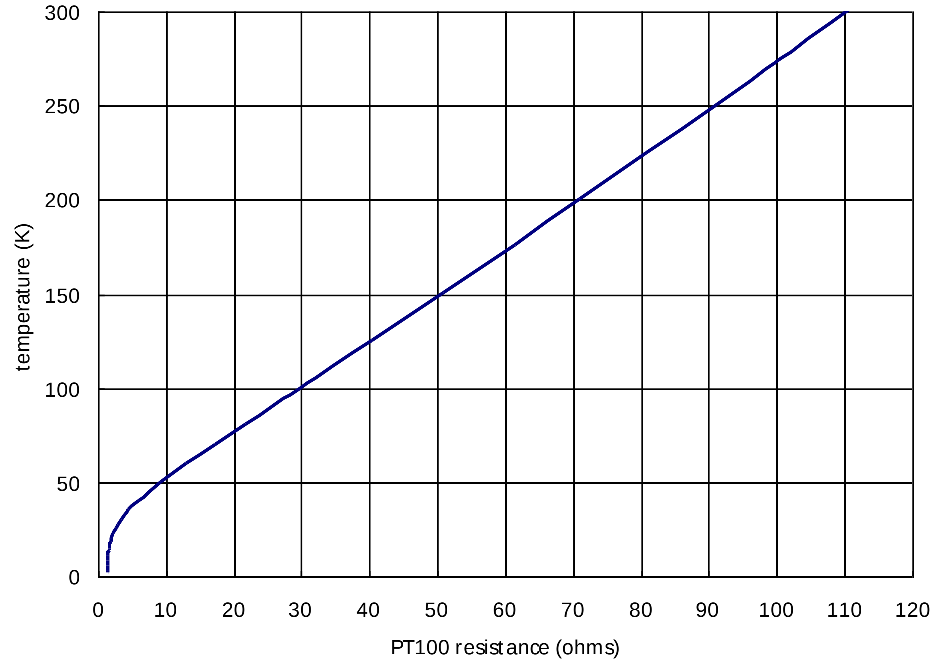
Time since start of cooldown (hours)	PT100 resistance (ohms)	Approximate Temperature (K)
0	107	290
1	100	273
2	95	260
3	83	230
4	71	200
5	67	190
6	63	180
7	59	170
8	51	150
9	30	100
10	26	90

Helium cooldown should take approximately 3 hours; system must be left for at least 12 hours after He fill before energising.

The Long Hold magnet is not fitted with any PT100s and has to be cooled down monitoring the shim resistances, from 75 ± 4 ohms at room temperature to 37 – 40 ohms at N2 temp, over the same time frame as that used for the standard Type3.

Generic calibration charts for the PT100 follow.




300
250
200
150
100
50
0
temperature (K)
0
10
20
30
40
50
60
70
80
90
100
110
120
PT100 resistance (ohms)

Magnet energisation table

Current range (A)	Rate of change (A/min)	Duration (min)
0 – 40[1]	1.00	
40 – 50	0.50	
50 – 55	0.25	
Wait at 55A		20
Wait for switch to close		5
Leads from 55 to 40	10	
Leads from 40 to 5	20	
Leads from 5 to 0	5	
Top up magnet		
Leave magnet for a minimum of 12 hours.		
Leads 0 – 55	20	
55 – 60	0.10	
60 – 62	0.05	
Top Up Magnet at 62A		
Wait after top up		30
62 – Field	0.05	
Wait at field		20
Field + 90 mA	0.05	
Wait		5
Field – 40 mA	0.05	
Wait		5
Field + 40 mA	0.05	
Wait		5
Field – 20 mA	0.05	
wait		5
Field + 20 mA	0.05	
Wait		5
Field	0.05	
Wait at field		20
Wait for switch to close		5
Leads field to 55	10	
Leads 55 – 5	20	
Leads 5 - 0	5	

Magnet de-energisation table

This magnet may be de-energised at twice the energisation rates (no need to hold at 55 A while de-energising).

Current range (A)	Rate of change (A/min)	Duration (min)
Top up magnet		
Field – 60	0.1	
60 – 55	0.2	
55 – 50	0.5	
50 – 40	1.0	
40 – 0	2.0	

[1] If a ZNMR NMR Signal kit (or equivalent) is being used then pause at (Field/6.1544) amps, measure the proton resonance frequency and calculate an accurate current for full field.

Electrical and continuity breakdown check sheet

AS400/54 T3 standard

		Assembly	N2 temp	LHe temp	After Quench	After Quench	Pins	Nominal resistance (Ω) RT	77 K	4.2 K
Continuity	Magnet Start - End						Coax-coax	0.7±0.3	0.5±0.3	0 - 0.3
	Shims Start - End{						1B-4B	75 ± 4	37 - 40	0 – 1
	{						2B-5B	75 ± 4	37 - 40	0 – 1
	Voltage taps						7B-8B	1.4±0.5	1.1±0.3	0 – 0.3
							7B-start	1.4±0.5	1.1±0.7	0 – 0.3
							7B-end	1.4±0.5	1.1±0.3	0 – 0.3
Heater resistances	Main						10B-1A	100±10	95±20	90±10
	Z0						10B-2A	100±10	95±10	90±10
	Z1						10B-3A	100±10	95±10	90±10
	Z2						10B-4A	100±10	95±10	90±10
	X						10B-5A	100±10	95±10	90±10
	Y						10B-6A	100±10	95±10	90±10
	ZX						10B-7A	100±10	95±10	90±10
	ZY						10B-8A	100±10	95±10	90±10
	C2						10B-9A	100±10	95±10	90±10
	S2						10B-10A	100±10	95±10	90±10
	PT100 number 1						6B – 3B	108 ± 2	26 ± 3	N/A
	PT100 number 2						N/A	N/A	N/A	N/A
Breakdown	Magnet-ground						coax-earth	>800 K	>800 K	>800 K
	Shims-ground{						1B-earth	>800 K	>800 K	>800 K
	{						2B-earth	>800 K	>800 K	>800 K
	Heaters-ground						10B-earth	>800 K	>800 K	>800 K
	Shims-magnet{						1B-coax	>800 K	>800 K	>800 K
	{						2B-coax	>800 K	>800 K	>800 K
	Heater-Shims{						10B-1B	>800 K	>800 K	>800 K
	{						10B-2B	>800 K	>800 K	>800 K
	Heaters-magnet						10B-coax	>800 K	>800 K	>800 K

How to use this check sheet: Use a DVM to measure the resistance across the specified pin numbers on the A and B ten pin seals and compare this resistance with the theoretical value noted in the right hand column. If the measured resistance falls within the specified limits, put a tick in the appropriate box and proceed to the next pair of pins.

Columns are identified for check results after assembly, pre-cool and initial helium fill. These values should also be checked after a quench.

Electrical and continuity breakdown check sheet

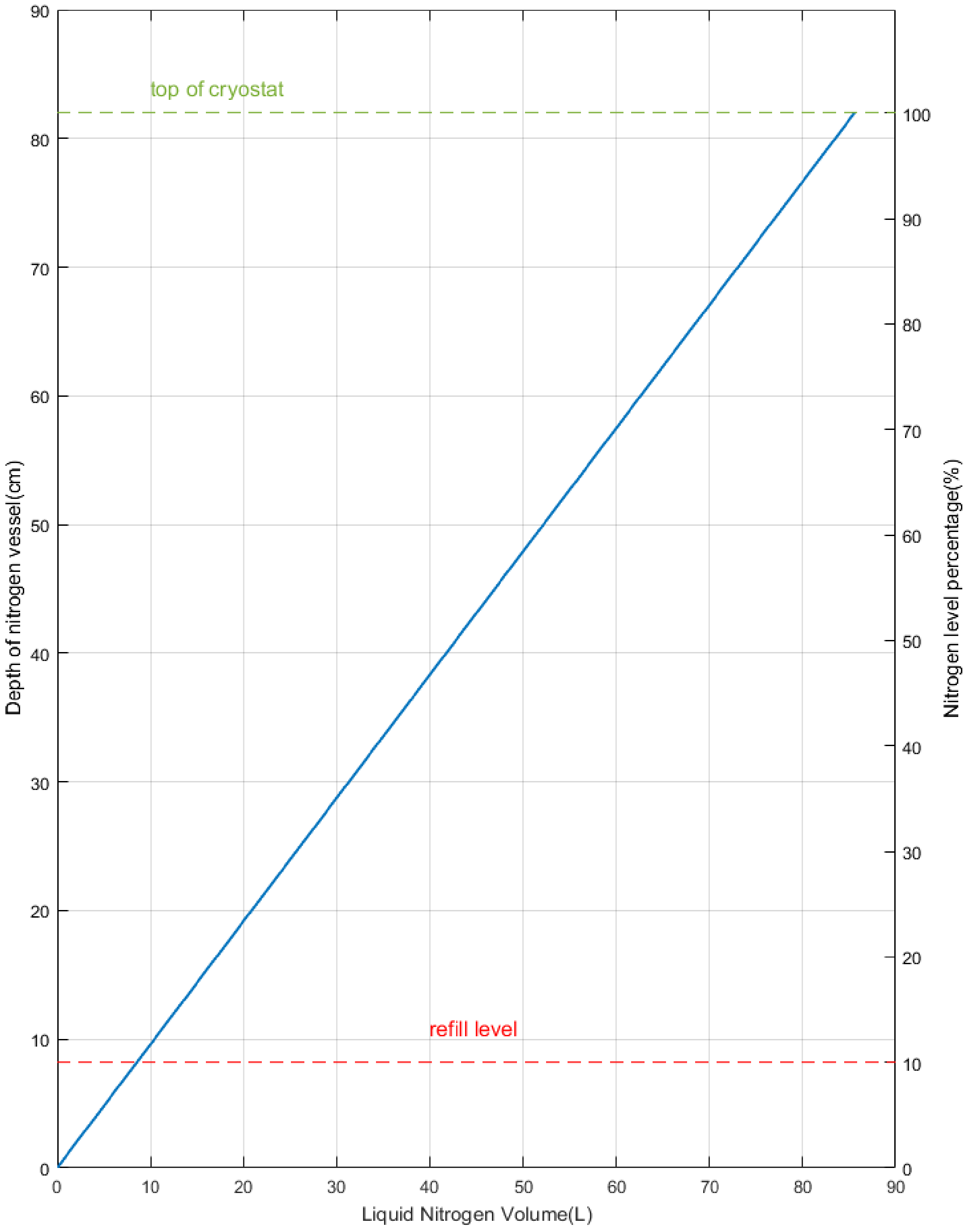
AS 400/54 T3 Long Hold

		Assembly	N2 temp	LHe temp	After Quench	After Quench	Pins	Nominal resistance (Ω)		
								RT	77 K	4.2 K
Continuity	Magnet Start - End						Coax-coax	0.7±0.3	0.5±0.3	0 - 0.3
	Shims Start - End{						1B-4B	75 ± 4	37 - 40	0 – 1
	{						2B-5B	75 ± 4	37 - 40	0 – 1
	Voltage taps						7B-8B	1.4±0.5	1.1±0.3	0 – 0.3
							7B-start	1.4±0.5	1.1±0.3	0 – 0.3
							7B-end	1.4±0.5	1.1±0.3	0 – 0.3
Heater resistances	Main						10B-1A	100±10	95±20	90±10
	Z0						10B-2A	100±10	95±10	90±10
	Z1						10B-3A	100±10	95±10	90±10
	Z2						10B-4A	100±10	95±10	90±10
	X						10B-5A	100±10	95±10	90±10
	Y						10B-6A	100±10	95±10	90±10
	ZX						10B-7A	100±10	95±10	90±10
	ZY						10B-8A	100±10	95±10	90±10
	C2						10B-9A	100±10	95±10	90±10
	S2						10B-10A	100±10	95±10	90±10
Breakdown	Magnet-ground						coax-earth	>800 K	>800 K	>800 K
	Shims-ground{						1B-earth	>800 K	>800 K	>800 K
	{						2B-earth	>800 K	>800 K	>800 K
	Heaters-ground						10B-earth	>800 K	>800 K	>800 K
	Shims-magnet{						1B-coax	>800 K	>800 K	>800 K
	{						2B-coax	>800 K	>800 K	>800 K
	Heater-Shims{						10B-1B	>800 K	>800 K	>800 K
	{						10B-2B	>800 K	>800 K	>800 K
	Heaters-magnet						10B-coax	>800 K	>800 K	>800 K

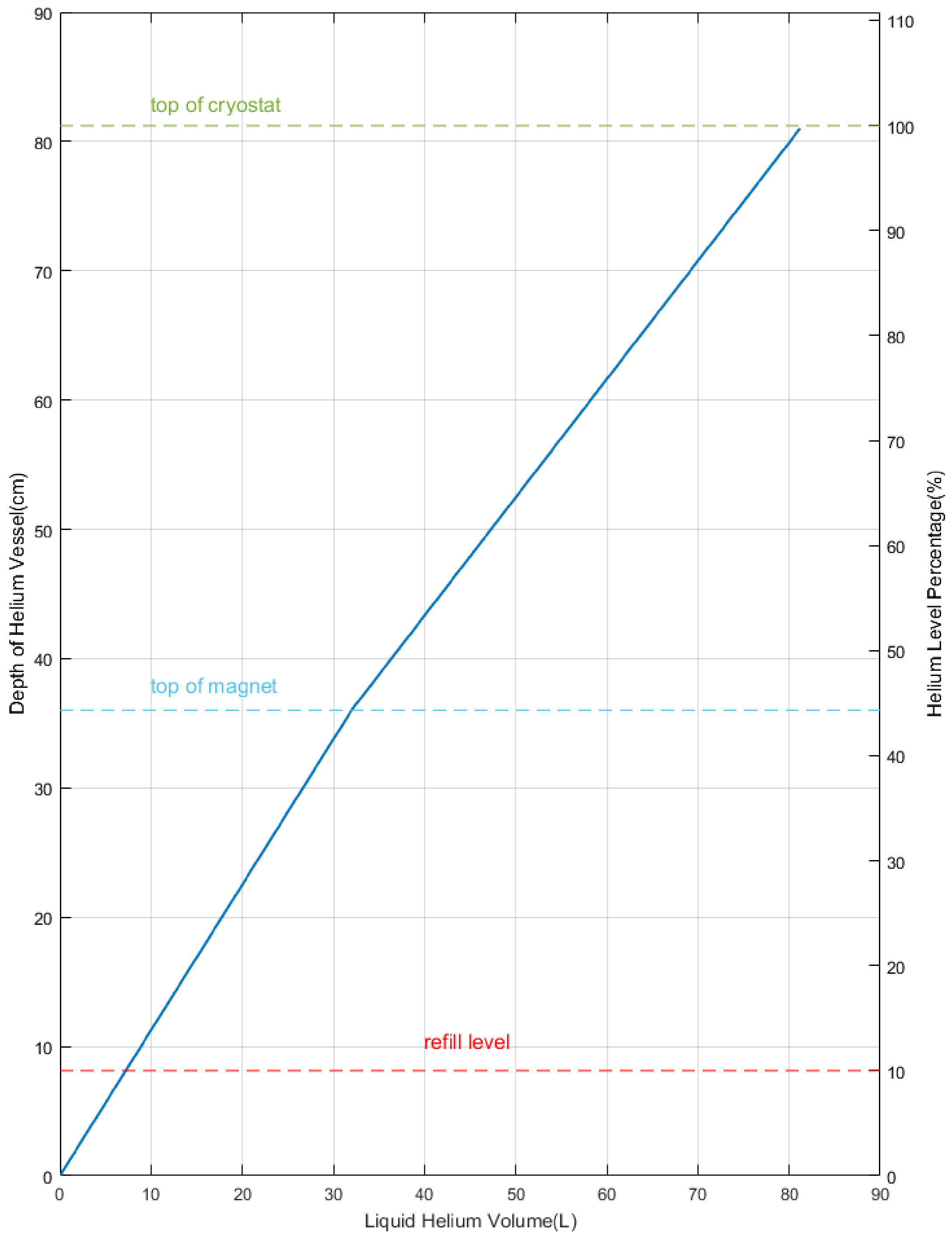
How to use this check sheet: Use a DVM to measure the resistance across the specified pin numbers on the A and B ten pin seals and compare this resistance with the theoretical value noted in the right hand column. If the measured resistance falls within the specified limits, put a tick in the appropriate box and proceed to the next pair of pins.

Columns are identified for check results after assembly, pre-cool and initial helium fill. These values should also be checked after a quench.

Nitrogen level graph

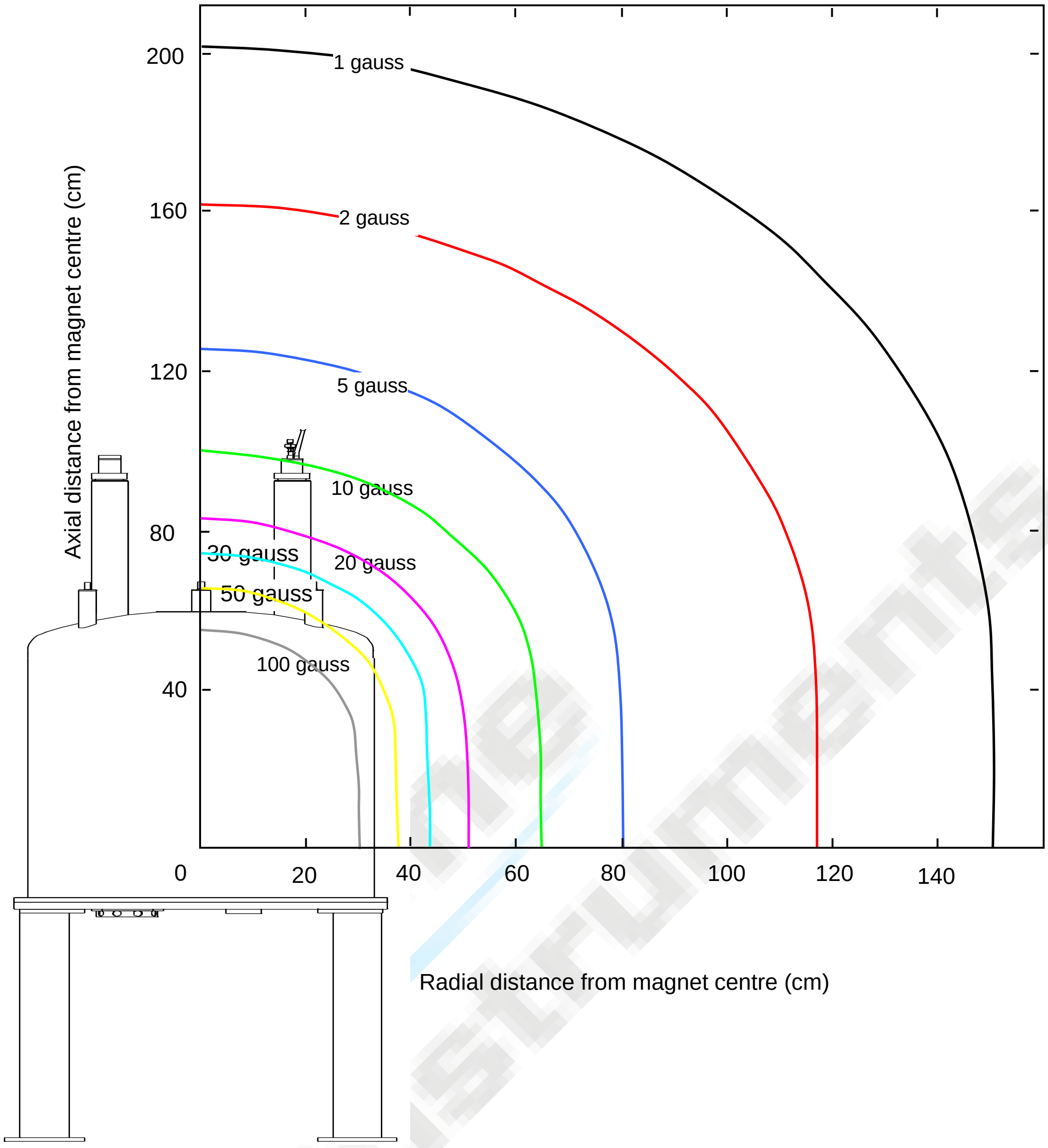


Helium level graph for AS 400/54 T3 standard



Stray Field Contour Map and intercept table

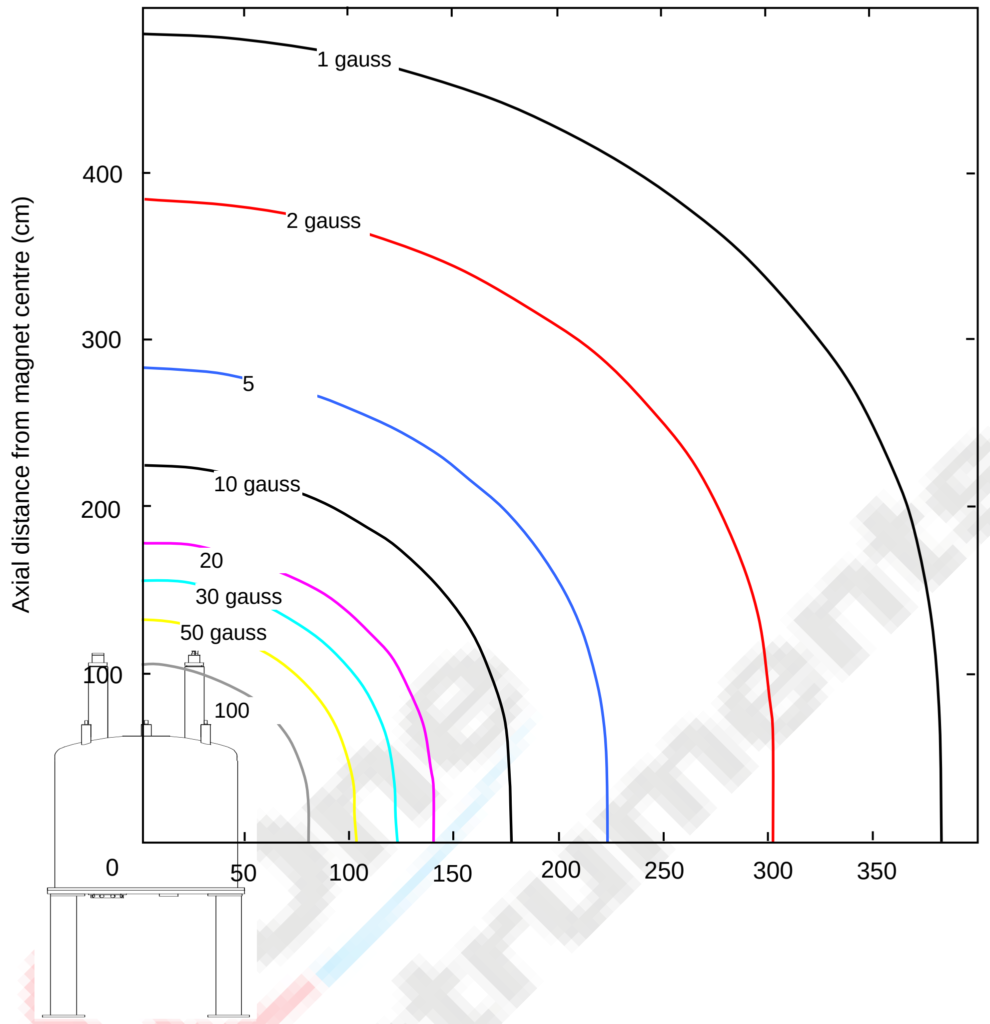
Normal operating conditions



Gauss line	Axial distance (cm)	Radial distance (cm)
1	210	150
2	165	115
5	130	80
10	100	65
20	85	55
30	75	45
50	70	38
100	60	33

Stray Field Contour Map and intercept table

Maximum stray field burst during quench



Gauss line	Axial distance (cm)	Radial distance (cm)
1	485	385
2	385	305
5	285	225
10	230	180
20	185	145
30	160	125
50	135	105
100	110	85

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Chapter 6

Glossary

This chapter defines some of the terms used in the User manual for the Q.One Instruments NMR systems and some that you will come across during the installation process. The text is modified from *Practical Cryogenics – an introduction to laboratory cryogenics,* copies of which are available from Q.One Instruments.

Backing pump A pump used to obtain the medium vacuum required for the operation of most high vacuum pumps (such as *diffusion pumps*, *booster pumps*, and *turbomolecular pumps*).
Usually a rotary vane pump is used as the backing pump.

Baffle A thin sheet of metal in the neck of the helium can of a *cryostat*, in a pumping line, or on a sample rod, to act as a *radiation shield* and to reduce convection and thermal oscillations.

Bladder The rubber lining for a 'soccer' ball, used to pressurise liquid helium storage vessels to promote the low pressure required to transfer liquid. If you squeeze the bladder with your hands warm gas is forced into the cold region of the helium vessel. This warm gas evaporates some of the liquid in the reservoir (because of its low latent heat). The gas that is released increases the pressure, and the bladder expands to larger than the original diameter.

Blow off valve Pressure relief valve.

Blow out tube Piece of stainless steel tube (often with a thread fitting on one end) used to transfer liquid nitrogen into and out of the helium vessel during the pre-cooling process. The thread screws into the siphon cone.

Boil off Evaporation from a cryostat.

Bursting disc A graphite safety device which breaks to relieve a high pressure in a pipe or vessel. Once broken, it has to be replaced with a new one.

Check valve Usually a one way valve, which allows flow in only one direction.

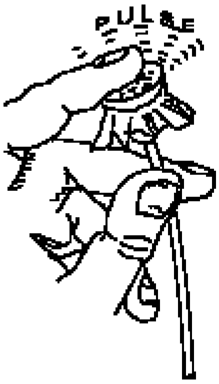
Cryogen A liquid whose normal boiling point is significantly below room temperature, used to provide refrigeration by its latent heat of evaporation.

Cryostat The terms *cryostat* and *dewar* tend to be used interchangeably to describe the vessel used to contain cryogens, usually to cool a superconducting magnet or other experimental apparatus.

Dewar See cryostat. Named after Sir James Dewar, 1842 - 1923.

Diffusion pump A high vacuum pump which works by entraining gas molecules with a flow of oil vapour which is then condensed and re-cycled. It requires a backing pump to produce a medium vacuum at the outlet of the pump. The pressure in the vessel to be evacuated must already have been reduced to an acceptable level by a roughing pump.

Dipstick Narrow tube with thin membrane over a housing at the top end, used as a simple level probe for liquid helium. The thermal gradient set up in the tube leads to thermal oscillations which are felt by the vibration of the membrane. The frequency of the oscillation with the lower end in liquid helium is noticeably lower than that when it is in cold gas, allowing the liquid level to be determined easily (in most cases). The dipstick should not be left in the cryostat when it is not in use because it introduces a large amount of heat.



Using a dipstick as a helium level probe

Drop off plate A safety device which is held in place by a vacuum, but which is allowed to fall away in the event of a high pressure building up in the vacuum space. Thus the pressure is released.

Energisation A superconducting magnet is said to be 'energised' when its current is being increased.

Gas cooled shield A radiation shield in a cryostat, cooled using some of the enthalpy from the helium gas that has evaporated from the helium reservoir. Sometimes used between a liquid nitrogen jacket and a liquid helium reservoir as a 40 K shield. More often used in liquid nitrogen free or vapour shielded cryostats.

Helium transfer Liquid helium must be transferred from one vessel to another very carefully because of its low latent heat of evaporation.

Homogeneity Uniformity. For example, the homogeneity of the field of a typical superconducting magnet may be defined as 1 part in 10^4 over a 10 mm d.s.v.

Leak detector Usually a mass spectrometer, tuned to be sensitive to helium, allowing very small vacuum. Leaks to be detected and often accurately located using helium gas.

Nb_3Sn A superconducting intermetallic compound of niobium and tin which has a very high critical field and critical temperature, and is used for the inner coils of many very high field superconducting magnets.

NbTi A superconducting alloy of niobium and titanium which is used for superconducting magnets with fields up to a maximum of approximately 9 T at 4.2 K (11 T at 2.2 K). It has the advantage of being cheaper and easier to use than Nb_3Sn.

NW fittings Nominal width. Sometimes referred to as 'ISO type DN' fittings.

O - ring Usually a rubber ring which is clamped between two surfaces to make a vacuum seal.

OVC Outer vacuum chamber, the insulating vacuum space around the liquid helium (and liquid nitrogen) reservoir(s).

Persistent mode A superconducting magnet is put into persistent mode by closing the superconducting switch that is fitted in parallel with the windings to complete the superconducting circuit.

The power supply may then be removed, and the rate at which the field decays is very low (typically 1 part in 10^4 to 1 part in 10^7 per hour, depending on the complexity of the design, and the type of superconducting joints).

Pirani Vacuum gauge for the medium vacuum range, which works by measuring the thermal conductivity of the low pressure gas.

Pre-cooling Before a system is cooled to 4.2 K using liquid helium, it is usually pre-cooled to 77 K using liquid nitrogen. This very considerably reduces the amount of liquid helium required to cool the system to 4.2 K, reducing the cost of the cool-down.

Protection circuit A resistive circuit connected in parallel with the windings of a superconducting magnet, to dump the energy stored in the magnet in the event of a quench. It protects the windings against damage if the magnet quenches.

Quench When part of the windings of a superconducting magnet goes normal (that is non-superconducting or resistive) the energy dissipated by the current flowing through this resistive part of the coil generates heat. The heat usually causes the normal region to propagate rapidly through the whole magnet (unless it is cryogenically stabilised). The stored energy of the magnet ($\frac{1}{2}LI^2$) is dumped into the helium reservoir as heat. Note that a magnet with an inductance of 100 H and current requirement of 100 A is not unusual, and that the stored energy of such a magnet is 500 kJ. The energy is dissipated within a few tens of seconds, so the liquid helium is usually evaporated very quickly.

Quench valve A relief valve which opens to release helium gas from the cryostat if a magnet quenches.

Syphon Vacuum insulated liquid helium transfer tube. Alternative spelling "siphon".

Syphon cone A fitting inside the helium reservoir of the cryostat for the *syphon* to plug into. For an efficient cooldown with liquid helium the liquid must be delivered to the lowest point so that the cold gas flows over the mass that has to be cooled, using the full enthalpy of the gas. In a complicated system, it is often impossible to arrange for a line of sight hole to the lowest point in the helium vessel. In this case a syphon cone is fitted, and there is a small tube from the cone to the bottom of the vessel.

Superconductor A material which loses its electrical resistance completely when cooled below its critical temperature. Many common metals become superconducting if their temperature is reduced sufficiently; for example, lead, tin, aluminium. However, the most useful superconductors for practical devices are alloys of niobium (Nb_3Sn and NbTi). New 'high T_c' materials are beginning to be used too.

Superconducting switch Device made from superconducting wire. It is warmed to turn the wire normal and open the switch, and it is allowed to cool to close the switch. This type of switch is often fitted across the terminals of a superconducting magnet for *persistent mode* operation.

Superinsulation Low emissivity materials used in the high vacuum insulation space of a cryostat to reduce the heat load due to thermal radiation.

Tesla The SI unit of magnetic flux density.

Thermal oscillations An acoustic frequency oscillation set up in a narrow tube which has a large temperature gradient along its length. This phenomenon is used as a cheap but effective level probe for liquid helium; see *dipstick*. However, if unwanted oscillations are allowed in a cryostat, they may introduce very large amounts of heat (perhaps watts), affecting the boil off significantly. Also known as *Taconis oscillations*.

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Magnet Data Record

Magnet data record

Use these tables to record magnet and shim currents for your NMR system. This information should remain with the system for the lifetime of the equipment.

Cryostat Number	错误!未找到引用源。
Project Number	
Magnet Number	ZA0073
Product Type	AS400**错误!未找到引用源。**
Console identification numbers	错误!未找到引用源。
Institute name	
Address	
Local contact name	
Local contact telephone number	
Email address	
Alternative contact name	
FAX number	
Lab telephone number	

Main magnet and superconducting shim currents – set 1

Main magnet and superconducting shim currents (amps)					
	value	End of phase 2			
Engineer Name	Fan				
Main magnet	+68.6 A	N / A			
Magnet Centreline Height： 332 mm					
Z1	-4.055				
Z2	-8.3324				
X	-5.454				
Y	-4.9621				
ZX	-2				
ZY	+1.66				
C2	+10.9571				
S2	0				

Magnet data record

Main magnet and superconducting shim currents – set 2

Main magnet and superconducting shim currents (amps)					
Comments					
Engineer Name					
Date					
Main magnet					
Main magnet polarity					
Z1					
Z2					
X					
Y					
ZX					
ZY					
C2					
S2					

Main magnet and superconducting shim currents – set 3

Main magnet and superconducting shim currents (amps)					
Comments					
Engineer Name					
Date					
Main magnet					
Main magnet polarity					
Z1					
Z2					
X					
Y					
ZX					
ZY					
C2					
S2					