S700X SQUID Magnetometer
USER MANUAL
Version 0.9

Contents

1 Introduction

Welcome to the Cryogenic Ltd. S700X SQUID magnetometer for the measurement of magnetic properties as a function of magnetic field and temperature. A Superconducting Quantum Interference Device (SQUID) is the most sensitive detector of magnetic flux available and is in principle capable of detecting single flux quanta variations in magnetic flux. The S700X represents the state of the art in magnetisation measurements. In the late 1980s M.V Osherov, a man of no small standing in low temperature physics, said “never use a SQUID magnetometer if you don’t need it”, but a lot has changed since then. What he meant was that the technology was so difficult to implement it should be avoided unless there was no other choice. The incredibly high sensitivity of the SQUID device meant that in the early stages of the technology it was very difficult to isolate the sample signal from other sources, however small. Now however, thanks to decades of research and development, modern SQUID susceptometers offer a number of possible measurements all with extremely high sensitivity and reliability.

Sample temperature can be controlled continuously from 1.6K to 300K as standard. Separate options are available to purchase to extend the temperature range. A furnace increases the maximum temperature to 700K and a Helium-3 sample probe lowers the base temperature to 300mK. Magnetic field is applied using a superconducting magnet with a maximum field of 7T. Under any combination of these conditions the magnetic moment of a sample is measured using a technique called extraction magnetometry, where the sample is moved through a pick up coil system. Unlike some other extraction magnetometers the signal that is measured is not dependent on the speed the sample is moved at. This is because the pick up coils are superconducting and detect flux, not changes in flux. That is, the signal is proportional to Φ and not $\frac{d\Phi}{dt}$. The advantage of this is that there is no need to move the sample rapidly through the pickup coils and so measurements can be done in a more reproducible way with the sample moved gently to avoid unwanted movement coming from fast acceleration.

The S700X has been highly engineered to remove as much of the difficulty as possible from performing magnetisation and susceptibility measurements. However, it must still be used carefully and correctly to ensure the most accurate and reliable results from your experiments. This manual is your guide through the potential pitfalls that await the unprepared.

Below is a description of the different measurement modes of the S700X. Chapter 3 gives an overview of the S700X hardware. Chapter 4 then talks you through what you need to know and do to perform a simple measurement. The remaining chapters cover each of the functions of the S700X in greater depth and chapter 13 is a software reference. Chapters 1 and 3 of this manual can be thought of as “what does it do?” and “how does it do it?” and chapters 4 onwards cover “how do I make it do what I want?”.

1.1 Modes of Operation

The S700X has three modes of operation.

DC measurement This is the most widely used. The measurement of total magnetic moment at a constant magnetic field is made by moving the sample through the pick-up coils. For materials that show magnetic hysteresis it is important that the sample remains in a highly uniform magnetic field. For this reason the normal scan length is 40mm, over which the field is uniform to 1 part in 10^4; the scan length can be varied from 2 – 130mm.

Oscillating (OSC) measurement This is useful for measurements made while the field is varying. The sample is made to oscillate up and down by a few mm and the SQUID signal appears as a sine wave whose amplitude is proportional to the magnetic moment.

AC measurement (If purchased) The magnetometer is provided with an additional set of field coils to generate an AC field of a few Oersted. This allows the measurement of AC susceptibility. The software can generate AC fields with chosen waveforms at frequencies up to 1 kHz.

1.2 Additional System Options

In addition to the AC option described above, the features and measurement range of the S700X can be augmented with the addition of a number of options. Each may be purchased separately.

The following may be delivered with the system or purchased at any time after installation:

High Temperature option This extends the measurement temperature range to 700K using a small oven that fits into the standard sample space.

Helium-3 option This is a second insert that extends the measurement temperature range down to 300mK.

Range extender Suppresses the sensitivity of the susceptometer to prevent saturation and allow larger signals to be measured. This is a modification to the system but can be done on the customer site.

The following are major modifications that must be done at Cryogenic Ltd in London. These are usually purchased and installed before delivery.

Transverse Field option Allows application of fields up to 10 Oersteds (1000 A/m) perpendicularly to the main magnetic field.

Transverse Pickup Coils A secondary set of pickup coils for detecting transverse magnetic moments. Instead of being moved vertically through the primary pickup coils the sample is rotated about the vertical axis. The rotating sample stage can also be used to perform standard DC measurements at fixed angles.

2 Safety

The following safety information should be read and understood by all users to prevent personal injury or damage to the system. This chapter is intended as a guide and any local procedures or requirements take precidence over the information contained herein.

2.1 Liquid gases

Where applicable, local training should be given in the safe use and handling of cryogenic liquids. Depending on the system model there will be liquid helium and/or liquid nitrogen involved in the operation of the magnetometer. These pose serious hazards if handled incorrectly.

2.1.1 Cold burns

- Liquid gases are extremely cold. Some metal surfaces may also become cold, especially on storage vessels and transfer tubes.
- If cold liquid splashes into your eye rinse immediately with luke warm water and seek medical advice.
- Use appropriate personal protective equipment (PPE) including gloves and eye protection.
- Avoid contact with liquid and cold metal surfaces with bare hands.
- Do not rub frozen skin as tissue damage may occur, place affected area in a warm water bath no hotter than $40°C$, do not use dry heat.
- Frozen tissue is painless and appears waxy with a possible yellow color. It will become swollen, painful, and prone to infection when thawed. If the frozen part of the body has been thawed, cover the area with dry sterile dressing with a large bulky protective covering, pending medical care. In case of massive exposure, remove clothing while showering with warm water.

2.1.2 Oxygen depletion

1 litre of liquid helium becomes 700 litres of gas when it evaporates, this will displace other gases in the air including oxygen.

- Ensure adequate ventilation.
- Use oxygen level monitors placed at head height.
- Never travel in a lift with cryogenic liquids. If a large dewar needs to be tansported by lift, put it in the lift and walk up the stairs.
- **Note:** In an oxygen poor atmosphere you will not notice any difficulty breathing until it is too late.

2.1.3 Storage and handling

- Cryogenic liquids such as helium and nitrogen should only be stored and transported in suitable vessels specifically designed for the purpose.
- Vessels must be clearly labelled with their contents.
- Vessels must have a functioning overpressure safety valve.
- Storage vessels should not be left completely closed or pressure will build up. This is not only dangerous but increases the rate of loss of helium. For the lowest loss rate, connect an open port to a return line. If no return line is available leave a length of silicon hose attached to the port and leave the other end free. This prevents fluctuations in air pressure (for example people walking past) from reaching the liquid inside the vessel and keeps the boil off as low as possible.
- never travel in a lift or other enclosed space with cryogenic liquids (see above).

2.2 Vacuum and high pressure systems

2.2.1 Pumps

- Oil-filled rotary pumps emit a fine oil mist which is a significant health hazard. Rotary pump exhausts should therefore always be fitted with an oil mist filter or routed outside the building.

2.2.2 Vacuum spaces

- All vacuum spaces in the system are fitted with suitable over-pressure relief valves. Do not obstruct or tamper with these.
- Do NOT allow air into a vacuum space in order to warm a cryostat more rapidly. This can cause serious damage.

2.2.3 High pressure gas cylinders

- Local laws and regulations should be followed at all times when using high pressure cylinders.
- Cylinders should be secured in an upright position.
- Cylinders must be used with an appropriate regulator for the gas contents.
- Only transport cylinders with a specialised trolley designed for the purpose.

2.3 Electrical Equipment

- Electrical equipment supplied by Cryogenic ltd. will be provided with a dedicated instruction manual containing specific safety guidelines that should be followed.
- All electrical equipment must be adequately earthed. Earthing should be checked regularly by a qualified technician.
- Conformity to local standards and regulations for electrical safety should be regularly certified by qualified personnel.

2.4 Lifting and Transportation

- The electronics rack is on wheels. These can be locked whilst the rack is in position.
- If a "Pump and Dump" is being used (for the recondensing and cryogen free systems) this should be lifted by two people as the pump is heavy.
- The cryostat of all models of system should only be lifted by the lifting points on the top plate. These are M6 tapped holes into which can be screwed heavy duty lifting eyes (supplied with the system).
- To move the cryostat the recommended method is to lift with a crane onto a wooden pallet and use a fork lift or hand truck.
- Always move slowly and carefully and maintain as close to vertical as possible.

2.5 Magnetic Field

- With the Cryogenic ltd. range of magnetometers, there is no risk to health or danger of corrupting magnetic storage media or credit cards etc due to stray magnetic field , because the 5 gauss surface is inside the cryostat.
- Never disconnect the magnet power supply whilst there is current in the magnet. Superconducting magnets may very rarely "quench", where a tiny part of the superconducting wire becomes normal, becomes hot from the current and starts a chain reaction, turning the rest of the magnet normal in a few milliseconds and releasing all the stored energy. The magnet power supply detects this happening and shuts off the current before any damage is done. Under no circumstances should the magnet be disconnected from the power supply whilst there is field (i.e. current in the magnet).
- The energy stored in the magnetometer system magnet is relatively small and if a quench does occur the only sign will be an increase in boil off which will last for a few minutes. In the recondensing system it may take a few hours for the pressure in the cryostat to return to normal since all of the boiled off helium must be recondensed.
- In the event of a quench there is no danger, you just have to wait for the magnet to become cold again before continuing your experiments.

- Quenches should be extremely rare. If they occur with any frequency, contact Cryogenic ltd. for support.

3 Hardware Overview

For the purposes of understanding the S700X it is convenient to divide the system into three parts; the cryostat/insert, the electronics rack and the computer/software. The following sections describe the functions of the major components of the hardware. The software is discussed briefly in chapter 4 in the context of initial set up and performing a basic measurement. The details of the software are discussed in context in each of the chapters on particular types of measurement and chapter 13 is a software look-up reference.

The standard cryostat and recondensing cryostat both house a liquid helium reservoir for keeping the superconducting components of the system cold. The helium is also used for the cooling power of the Variable Temperature Insert (or VTI). The recondensing cryostat uses a pulse tube cryocooler to keep the helium reservoir sufficiently cold that boil off gas and gas used for the VTI is recondensed. In the standard system the helium gas used by the VTI is exhausted from the VTI pump, often connected to a helium return line in the laboratory. In the recondensing system, this exhaust is instead connected to a gas reservoir (know as the “dump”) which stabilises any pressure fluctuations before the gas is returned to the reservoir to be recondensed.

The cryogen free version of the system has no liquid reservoir at all. All of the cooling is done with a pulse tube and helium is used in a closed loop, liquefied in a small chamber sufficient to run the VTI. The radiation heat load on the system is minimised with radiation shields and multilayer super-insulation. Ambient magnetic fields are also shielded in the interior to a few micro-Tesla with a Mu-metal shield. The sample is supported on a probe that is held at the top of the insert by the sample transport mechanism which moves the sample through the pick up coils of the SQUID detector circuit. An airlock allows insertion and removal of the sample probe without contaminating the interior of the insert with atmospheric gases that would freeze in the low temperatures inside, causing damage.

The rack contains all of the electronics that control the various systems needed to operate the susceptometer. Control of the electronics is almost exclusively through the software.

3.1 Cryostat / Variable Temperature Insert (VTI)

On delivery of the system the VTI will be installed in the cryostat by a Cryogenic Ltd engineer. It is secured to the cryostat top plate with five stainless steel M6 bolts. The cryostat consists of an aluminium outer shell containing a helium reservoir constructed from welded aluminium and glass fibre/epoxy composite neck and tail section. A niobium can is mounted in the tail section which, when superconducting, stabilises any ambient magnetic field that penetrates the Mu-metal shield. The base of the helium reservoir tail is fitted with a Carbon Ceramic Sensor (CCS) thermometer and heater. These are connected to the 6 pin connector in the cryostat top plate. The temperature response of these thermometers between 300 K and 4.2 K is calibrated which allows initial cool-down and subsequent operation of the cryostat to be monitored. The insert head comprises of various levels which house the connections for the electrical and gas services required to operate the susceptometer. The function of the cryostat and insert can be subdivided into the following areas.

3.1.1 Reservoirs/Shields - Standard/Recondensing versions

Figures 3.1 and 3.2 show section views of the Insert with standard and recondensing cryostats respectively. The helium reservoir serves to keep the magnets and the SQUID detection circuit in their superconducting state. It also provides the helium for the VTI temperature control system. To reduce the rate of helium boil off there is a shield system to prevent thermal radiation from the outside reaching the helium reservoir. In the standard cryostat an outer shield is kept at 77 K by the liquid Nitrogen reservoir. This shield is made from high thermal conductivity Aluminium. Heat arriving at the shield as radiation travels up the shield to the Nitrogen reservoir where it is removed to the outside as nitrogen boil off. The remaining radiation from the 77 K shield then meets a second shield kept at around 40 K by a thermal link to the Helium boil off gas in the insert. In addition to the Nitrogen cooled and gas cooled shields there are several layers of super-insulation.

There is a feedback system at work here. As the 40 K shield screens radiation it reduces helium boil off, but the helium boil off cools the shield so less boil off means less screening. This in turn leads to increased boil off and increased screening and so on. This makes the radiation shielding very stable once in equilibrium. For the same reason it takes several days for a new system to reach equilibrium on installation. If it is ever necessary to warm the system to room temperature it is very important that on cooling down again it is allowed to fully equilibriate. See chapter 4 for more information on system warm up and cool down.

In the recondensing cryostat there is no nitrogen reservoir. Constant cooling power comes from the pulse tube cryocooler on the top plate of the cryostat. The first stage of the cryocooler maintains a single radiation shield at 40 K. The second stage, which reaches around 3 K, is used to liquefy a closed loop of helium gas, referred to as the condensing loop. This is separate from the reservoir, but once cold the liquid in the condensing loop begins to circulate, cooling the liquid in the reservoir to below it's boiling point of 4.2 K and recondensing the gas which in the standard system would be exhausted from the VTI pump.

Outside the radiation shields and super-insulation is the Mu metal shield. This screens the interior of the cryostat from external magnetic fields, including the Earth's own field, to a few micro-Tesla. Fitted to the Mu metal shield is a system of "degaussing" coils to demagnetize the shield in the event of it becoming magnetized. See section 14 for more information on degaussing the shield.

3.1.2 Cryogen-Free System

The cryogen free version has no reservoir at all and is built in a different way to the other systems. The VTI and magnet are integrated into the cryostat itself and cannot be removed. The magnet and superconducting pickup circuits are kept cold with the cold head. There is a Copper radiation shield surrounding the low temperature parts of the system, connected to the first stage of the cold head at around 40 K and several layers of super-insulation surround that. A closed loop of helium gas is used for temperature control. Rather than drawing liquid helium from a large reservoir, a small pot is connected directly to the second stage of the cold head. This pot is cooled low enough to liquefy helium inside it. This small amount

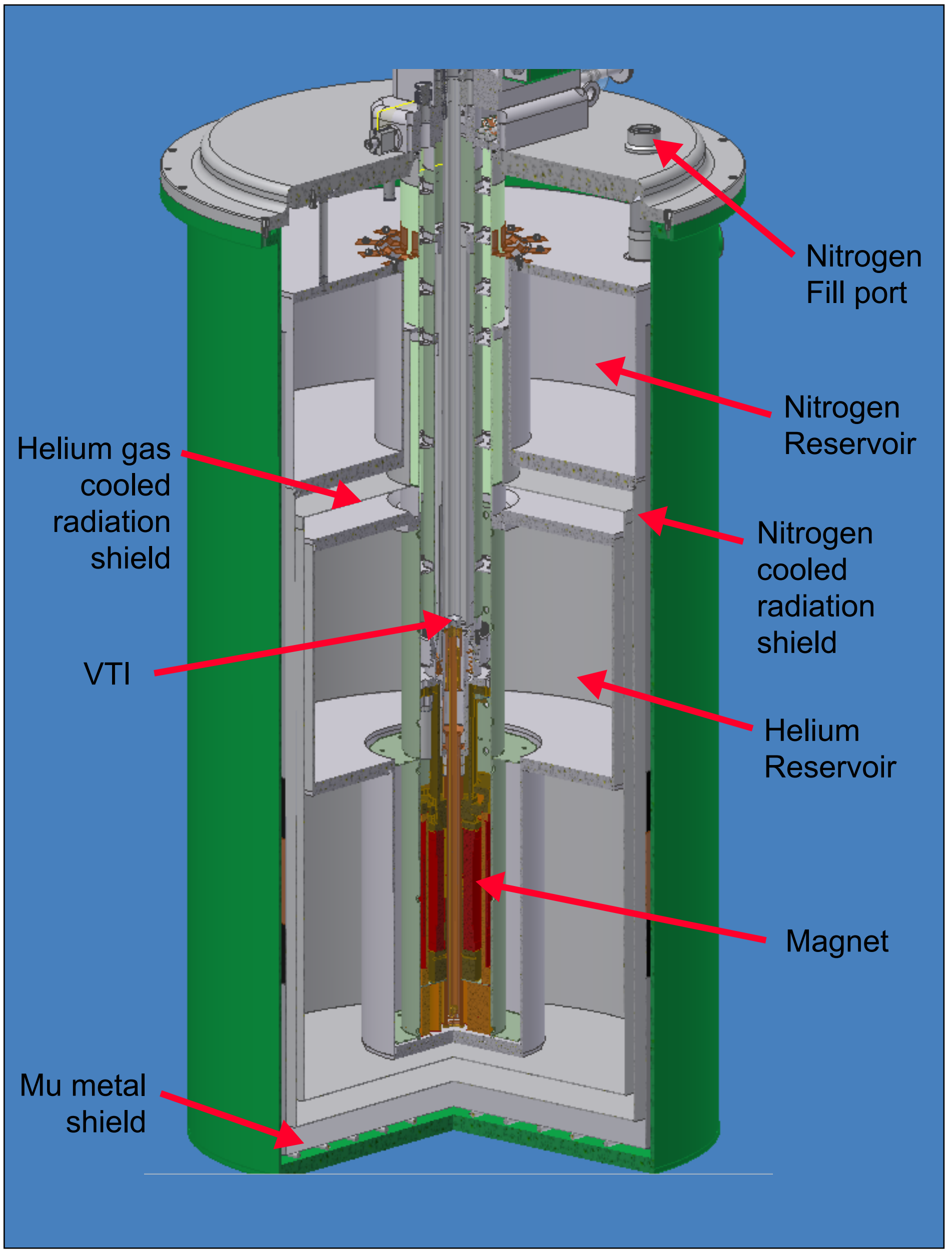


Figure 3.1: *Three quarter section view of the standard cryostat and Insert.*

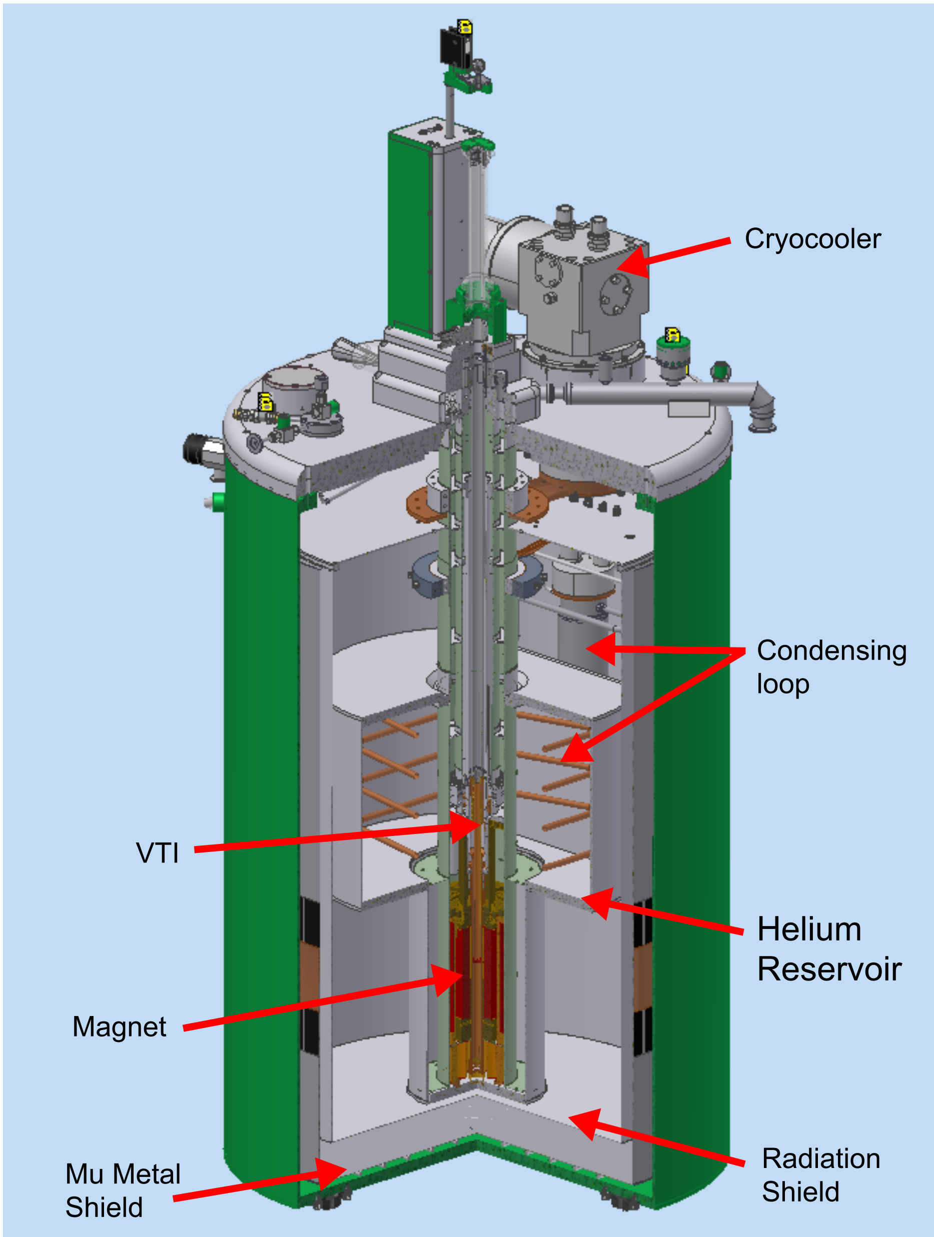


Figure 3.2: *Three quarter section view of the recondensing cryostat and Insert*

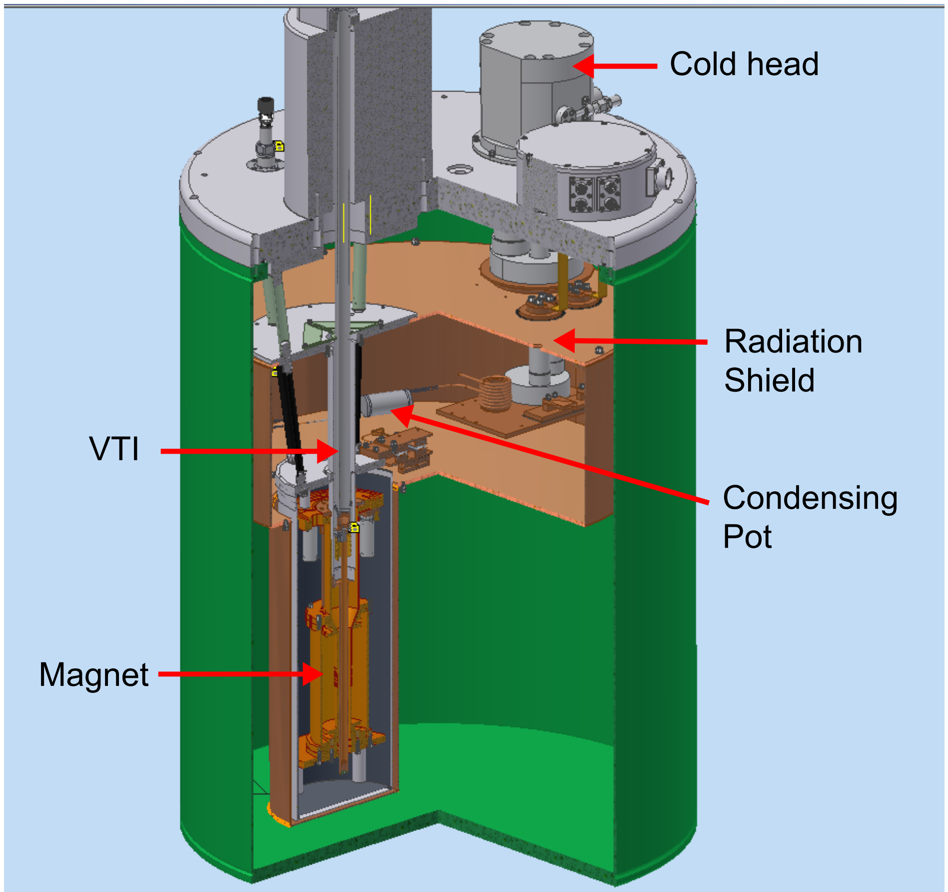


Figure 3.3: *Three quarter section view of the cryogen free system.*

of liquid is enough for the VTI to use, although it is used in a slightly different way (section 3.1.4).

3.1.3 Magnet

The superconducting magnet, shown schematically in figure 3.4, provides the bias field, up to 7 T, for sample measurements. It features high homogeneity and low drift allowing rapid field change and subsequent field stabilisation. The magnet is in two sections. The "inner" generates the bulk of the field. It has its own compensation windings to homogenize the field over a 4cm vertical region in the magnet centre. The "outer" is a compensation coil to fine tune the homogeneity of the axial field and also to minimise the stray field. The magnet features a "persistent mode" switch. The persistent mode switch is essentially a non-inductive superconducting connection across the magnet terminals. This forms a closed superconducting loop with the magnet coils and current can flow without loss, meaning the field is persistent without the need for constant current. To change the current in the inductive winding of the magnet the switch connection must first be driven into the normal (resistive) state. This is accomplished using a heater driven at 2-3V from the control cabinet. Current from the magnet power supply can then flow through the main inductive section of

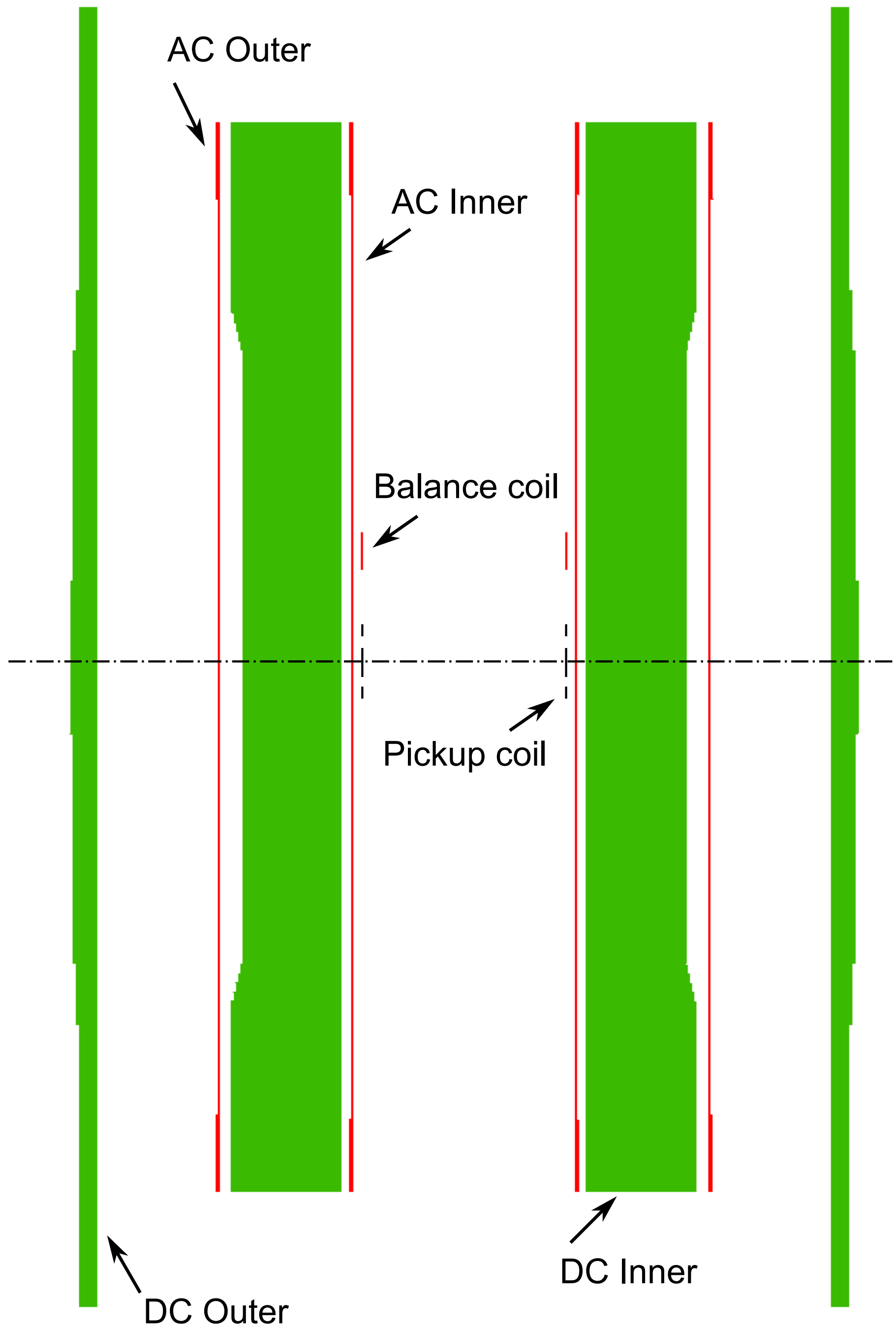


Figure 3.4: *Schematic cross-section of the magnet assembly*

the magnet and generate the required field. After the power supply has homed in on the chosen current the persistent switch heater is turned off, allowing the switch to become superconducting, and the current in the magnet current leads is ramped to zero leaving the magnet in persistent mode.

3.1.4 Temperature control

The inside of the variable temperature insert (VTI) is thermally isolated from the helium and nitrogen reservoirs by a vacuum . Temperature control is achieved in the S700X insert as shown schematically in figure 3.5. Liquid Helium is drawn from the reservoir. It passes through a constriction, in the form of a needle valve. The impedance of the valve causes a sharp drop in pressure which cools the Helium by the Joule-Thomson effect. The helium vaporizes and cools to around 1.5 K. The cold gas is then passed through a heat exchanger, where it is warmed to the desired temperature by a heater, before being passed through the sample chamber. The sample chamber and its contents are then allowed to equilibriate with this gas. The sample chamber is made from phosphor bronze, which is a poor electrical conductor, to avoid Eddie current heating. This means it is also a poor thermal conductor. Also, the gas flow is laminar, due to the low speed, and at the inner surface of the chamber the gas speed is almost zero. This reduces thermal exchange between the gas and the chamber. To minimise thermal gradients along the length of the chamber, three copper wires (a good thermal conductor) are attached vertically to the outside. To improve heating rates, the chamber is also fitted with a non-inductively wound auxiliary heater. The system is made as adiabatic as possible so that once the heat capacity of the chamber is overcome and the desired temperature is reached, the auxiliary heater can be switched off. The temperature controlled gas flow is then sufficient to maintain the temperature. There are two thermometers used for temperature control, A and B. Thermometer A is located on the heat exchanger and effectively measures the temperature of the gas coming from the heat exchanger to the sample space. Thermometer B is positioned in the sample space above the sample position. Once equilibrium has been reached thermometer B can be taken as the sample temperature.

Temperature control in the cryogen free system is subtly different. There is no reservoir, so liquid helium comes instead from a small pot connected to the cryocooler where helium gas is liquefied. This gas is in a closed loop and after it passes through the needle valve and heat exchanger, as before, it does not enter the sample space but a sleeve around the sample space. After it travels past the sample chamber, cooling the chamber on the way via a tiny amount of exchange gas inside, the gas is returned through the pump into a 50 litre gas reservoir. The pump and reservoir together are called the “Pump and Dump Station” and are located next to the main cryostat. From the Dump, the gas returns to the cryostat and after passing through several heat exchangers on it’s way to 4K arrives back at the cold pot where it is once again liquefied.

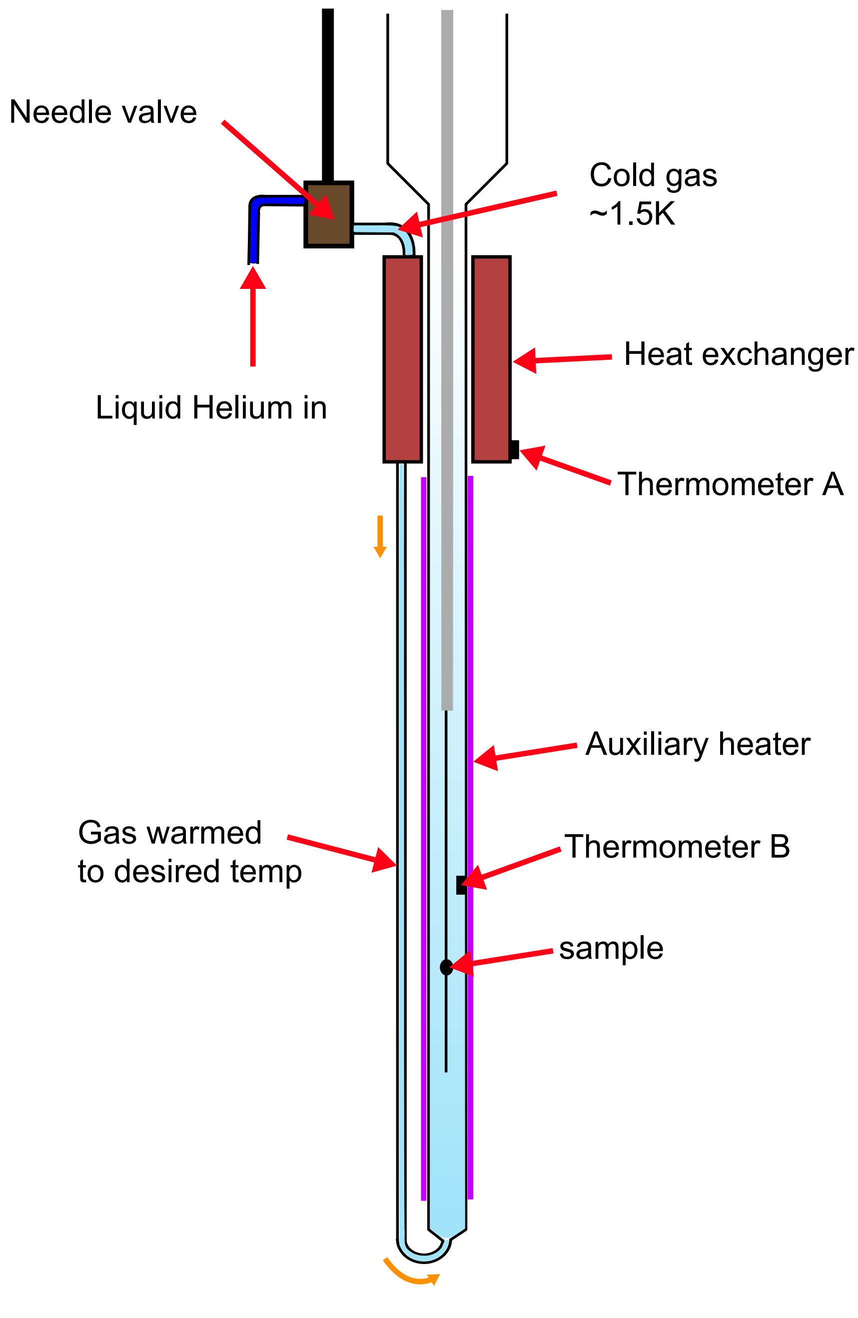


Figure 3.5: *Schematic depiction of temperature control in the S700X.*

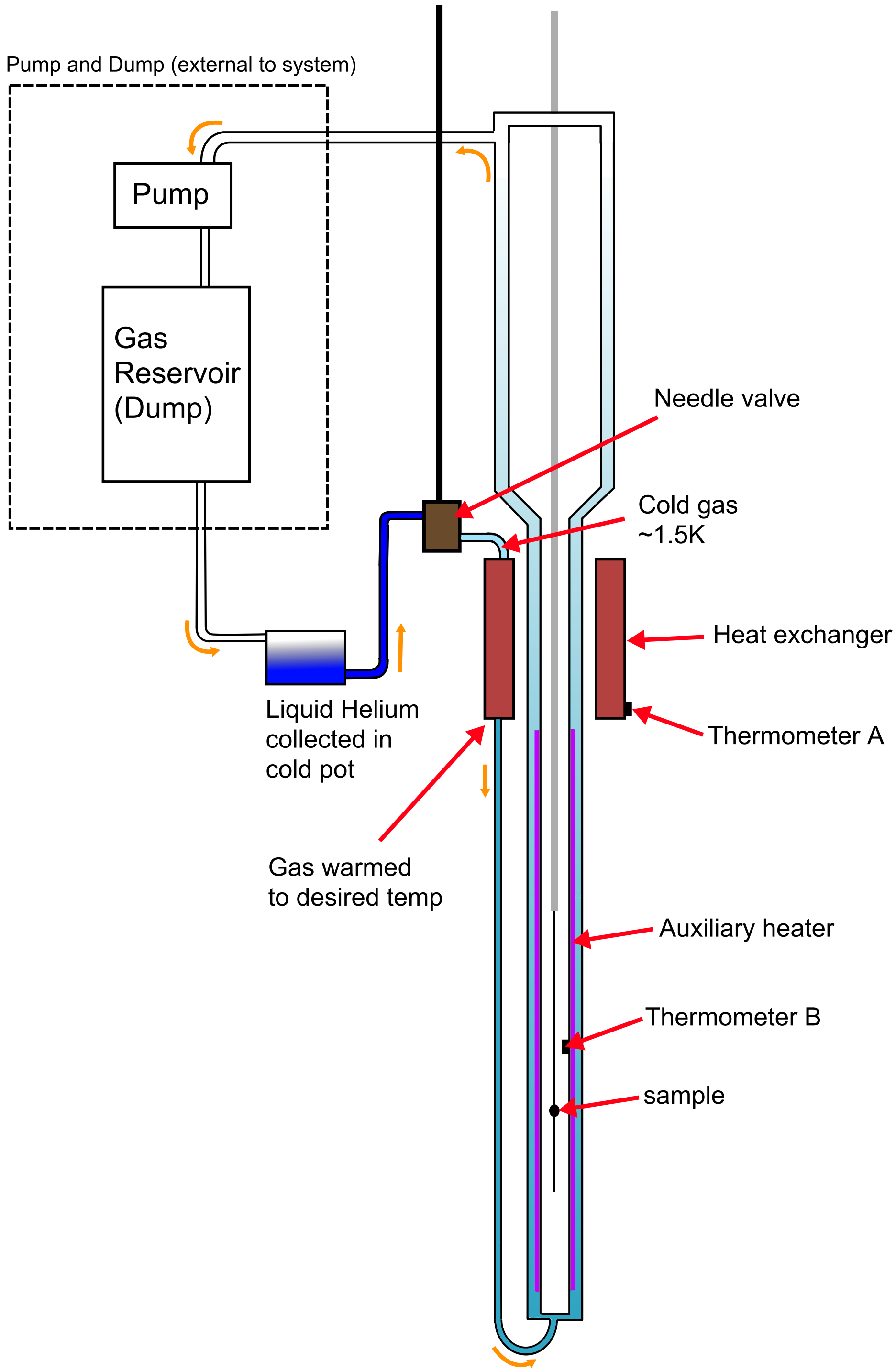


Figure 3.6: *Schematic depiction of temperature control in the cryogen free version.*

3.2 Gas Handling

Schematic diagrams of the helium circuits for all three systems are shown in figures

3.3 Measurement circuits

A SQUID is the most sensitive detector available of changes in magnetic flux. The SQUID detection circuitry is shown in Fig. 12.1 on page 123. A set of superconducting pick up coils is located centrally in the bore of the magnet. The pick up coils are inductively coupled to the input coil of the SQUID to form a flux transformer. The SQUID is driven with a DC bias current above the critical current of the device so that a voltage appears it. Quantum effects cause this voltage to depend on the flux within the SQUID. See chapter 12 for more details.

The SQUID is situated above the magnet (see Fig.10) and enclosed in a niobium can which shields it from environmental magnetic noise and the stray field from the magnet. A second niobium enclosure houses the flux transformer filter circuit (or optionally the flux transformer attenuator). The pick up coils are wound in a second order gradiometer configuration with only a few millimetres separation between adjacent turns. This configuration is chosen to reject the field from the surrounding magnet to typically 0.1% and hence desensitise the SQUID to changes in signal associated with magnet field drift. Although drift is an inevitable consequence of the non-ideal nature of the superconducting joints and wire within the magnet circuit, the control software fully compensates for any residual drift signal during a measurement routine. The SQUID measures relative changes in magnetic flux and for this reason it is necessary to move the sample through the coil set. This causes a screening current to flow in the flux transformer circuit which opposes the resultant change in flux through the pick up coils. This current is proportional to the induced magnetic moment of the sample and is detected by the SQUID via the input coil. The output from the SQUID electronics then gives a voltage directly proportional to the signal detected at the SQUID sensor.

3.4 Sample motion and limit switches

To move the sample through the pick up coils there is a transport mechanism on top of the insert. This holds the top of the sample probe and a stepper motor moves the probe vertically up and down through the pick up coils. The longitudinal motor makes 16,000 steps per cm. If the transverse moment option is installed then a second detection circuit is present in which the pick up coils are oriented perpendicular to the axis of the magnet. To measure the transverse moment the sample is rotated about the vertical instead of being moved up and down. A separate top component that can be fitted to the top of the transport mechanism contains a second stepper motor and an anti-backlash gear for precise rotation of the sample probe.

The longitudinal mechanism has three optical switches to act as reference points in case the motor ever stalls and the software loses the position of the probe. There are upper and lower limit switches at the top and bottom of the full range of the mechanism. When a tag on the motor passes through one of these switches then the software knows where the motor

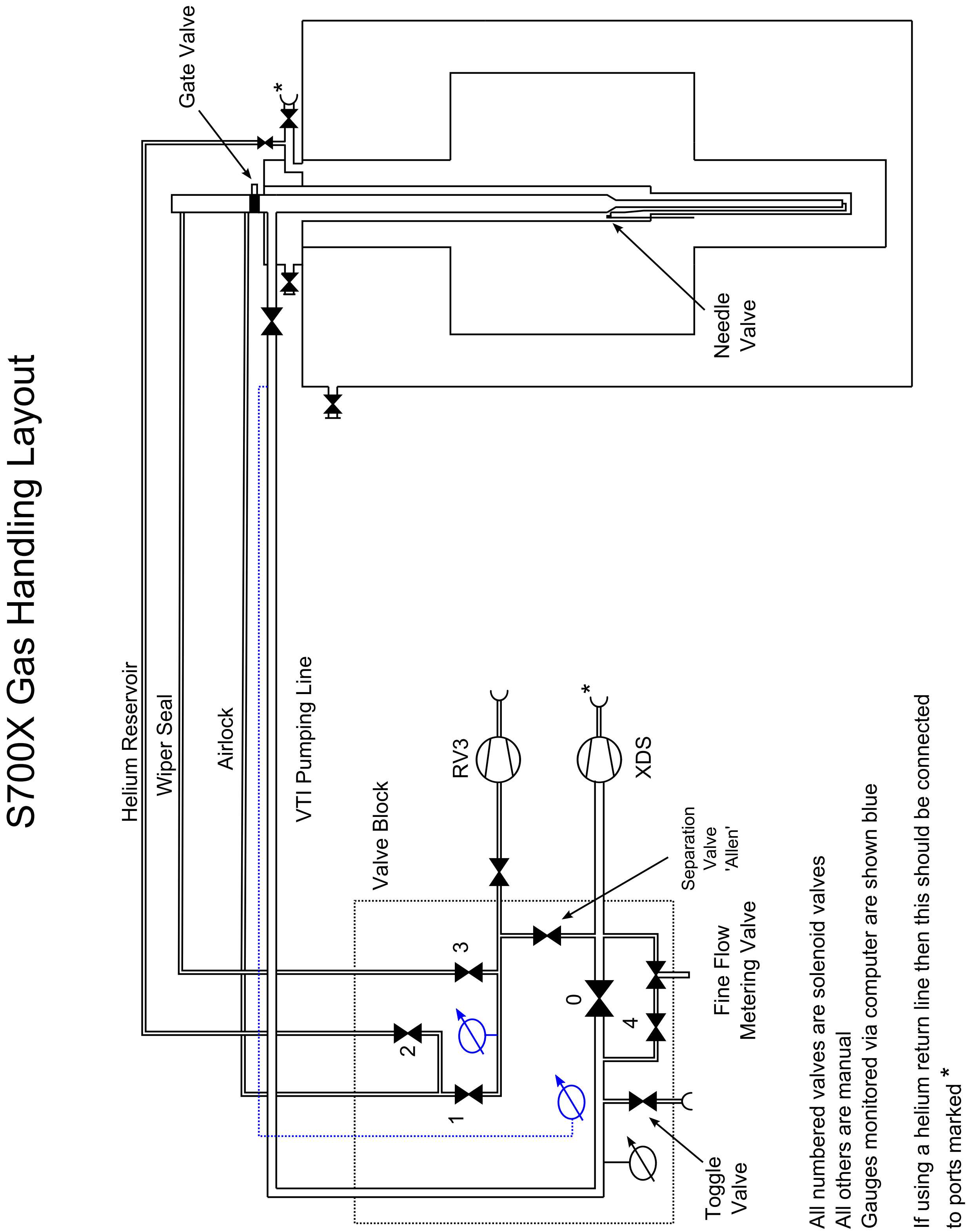


Figure 3.7: *Schematic diagram of gas handling in standard system*

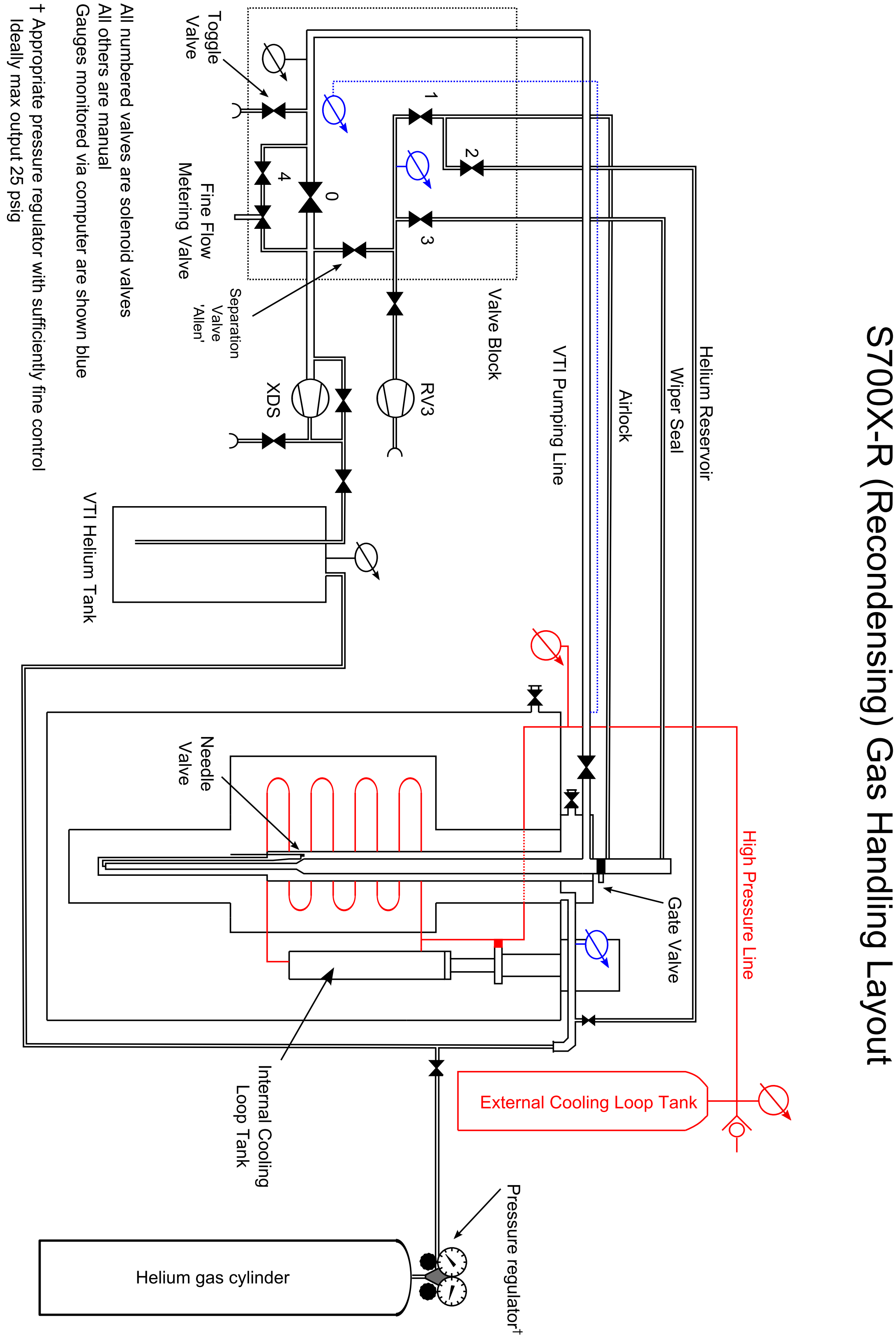


Figure 3.8: *Schematic diagram of gas handling in recondensing system*

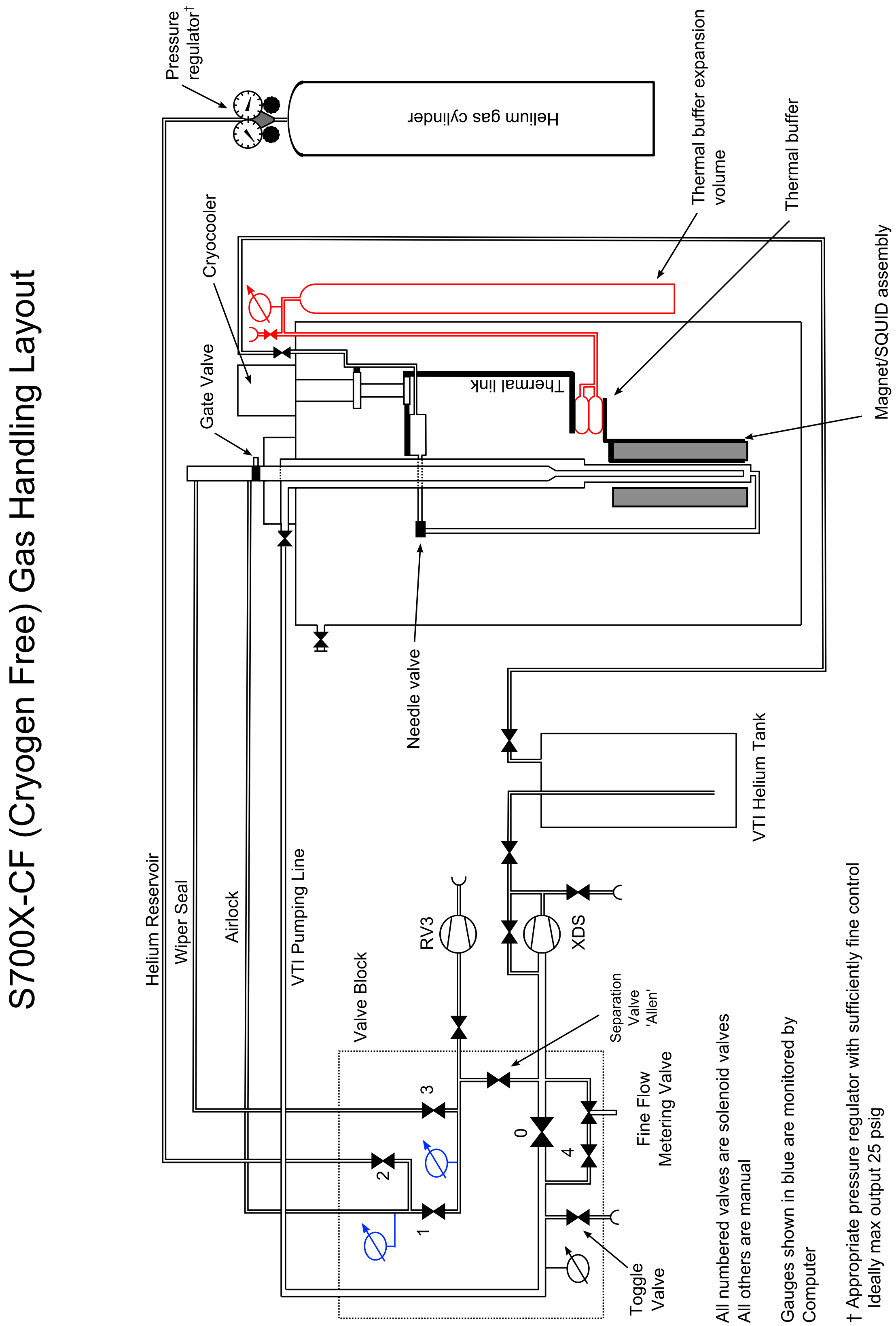
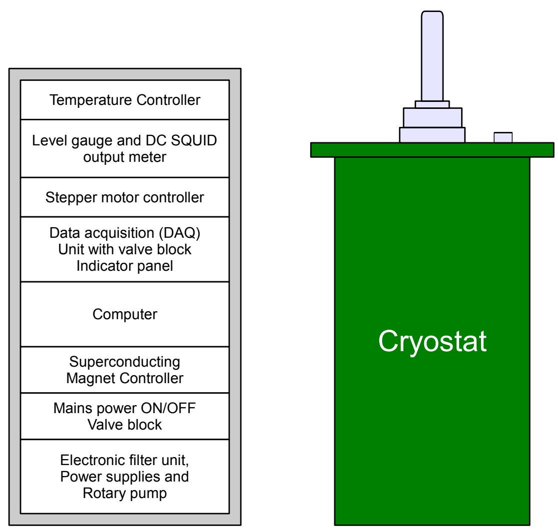


Figure 3.9: *Schematic diagram of gas handling in cryogen free system*

is. The third switch is in the centre and is referred to as the *home* position. By moving to the upper limit and reaching the upper switch then the software knows for certain that the motor is above the home position and it can then move downwards until it finds the home switch.

The rotational mechanism has one optical switch to act as a home position so that all rotations can be made relative to a known angle and therefore are consistent between measurements.

3.5 Electronics Rack



The electronic systems are housed in a standard width electronics rack. From top to bottom the rack consists of:

Temperature Controller Continually monitors all thermometers and controls the temperature of the VTI heat exchanger.

Level gauge and DC SQUID interface Measures the level of the liquid helium in the reservoir. SQUID output available via a BNC connector and also displayed on the panel meter.

Stepper motor controller This controls the sample position within the pick up coils. Front panel LEDs indicate if a limit switch is activated.

Data acquisition unit / Valve block indicator panel Controls all analogue and digital inputs and outputs to the system hardware except for the magnet power supply and temperature controller. Red and green LEDs The red LED on the lower front panel indicates power to the data acquisition unit and the green LED indicates the computer is properly connected.

Computer Built in computer that runs the S700X software and controls the various electronic systems. Details concerning the computer may be found in the computer manuals provided.

Superconducting Magnet Power supply/Controller Controls the current in the superconducting magnet. Refer to the separate manual for detailed information about this unit.

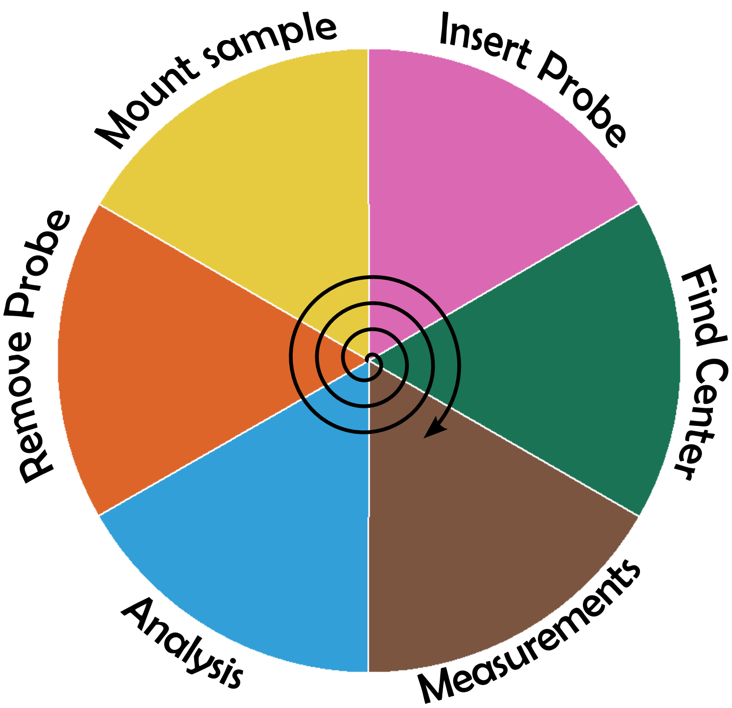
Mains power ON/OFF and Valve block The valve block contains the electronically controlled valves that operate the S700X gas systems as well as a pressure gauge for the VTI, the metering valve to control fine flow of gas and the service port for flushing the insert with gas (see chapter 4).

Electronic filter unit, power supplies and rotary pump The bottom panel houses the electronic filtering circuits for all electrical services connected to the insert, the power supplies for the VTI heaters and SQUID/magnet detection circuit and also houses the rotary pump.

4 First Steps

Getting on the Measurement Merry-go-round

Once the system is cold and operating, the process of taking data becomes an efficient cycle.



The Measurement Merry-go-round

To enter this cycle the system must be cooled from room temperature and the liquid Helium and Nitrogen reservoirs filled (if applicable). This will be carried out by a Cryogenic engineer on installation of the system, but the process is given here for reference should it ever be needed. In this chapter we will describe cooling the system from room temperature, making one complete turn of the merry-go-round and the different ways to safely exit the ride. When no measurements are being taken, fully wet systems should be left in standby mode to reduce helium consumption. Alternatively it may be necessary, for example if moving to a new laboratory, to warm the system up completely. Both of these endings are described in this chapter. So let's get the wheel turning...

4.1 Cable connections

On delivery, all cables and gas lines are stored inside the electronics rack for transportation. They are connected correctly at the rack end and on installation a Cryogenic Ltd engineer will connect them to the system hardware. All gas lines should be connected prior to cool

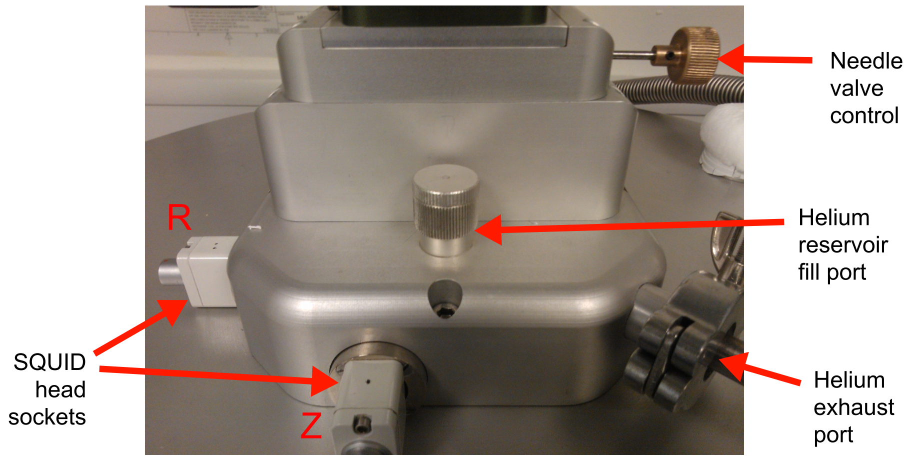


Figure 4.1: *Ports and connections on the front of the VTI.*

down. For reference, connections to the standard insert are shown in figures 4.1 and 4.2. For systems with a recondensing cryostat there is an additional 24 pin Fischer cable that plugs into the socket shown in figure 4.3.

Important: The SQUID heads (PFL-100) and their connecting cables should not be connected during cool down or Helium transfers to avoid the SQUID electronics from getting too cold. When connecting or disconnecting the SQUID heads a particular order must be followed to avoid damaging the SQUID devices themselves. This is explained in 4.3.

4.1.1 Reservoir Tail Heater and Thermometer

The S700X and S700X-R systems each have a thermometer and resistive heater on the tail of the helium reservoir. These can be used to monitor the reservoir temperature and to apply heat if required. The heater is a $30\,\Omega$ resistor.

Recondensing cryostat The recondensing cryostat has several thermometers at various points and monitoring the temperatures of these is an integrated feature with it's own dedicated software (see section 4.2.5). To apply heat to the reservoir we need to apply current to the heater. There are banana plugs coming from the back of the Keithley 2700 unit in the rack. These can be connected to either a bench top power supply or the heater output of the Lakeshore temperature controller.

Standard cryostat The thermometer and heater on the tail of the helium reservoir in the standard cryostat are wired to the 6-pin Fischer socket on the top plate of the cryostat. This is not usually used so it will not be connected in normal operation. This can be connected

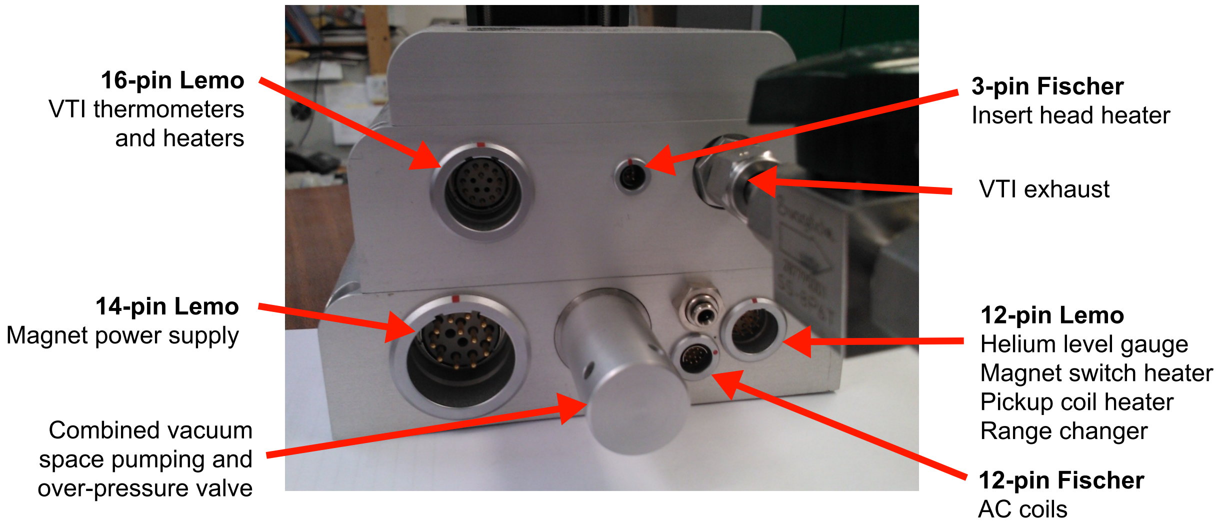


Figure 4.2: *Connections on the rear of the VTI.*

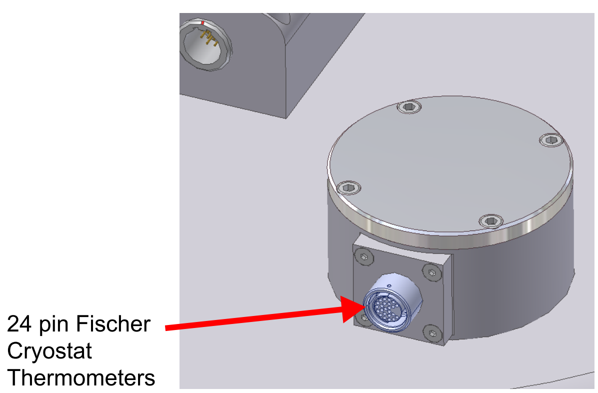


Figure 4.3: *The recondensing cryostat has several additional thermometers for monitoring the cool down. The cable for these plugs into the 24 pin socket shown here.*

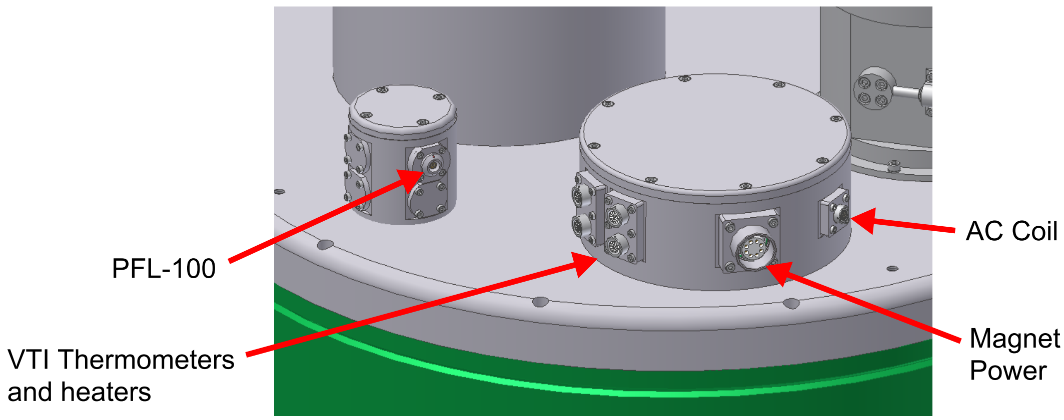


Figure 4.4: *Cable connections on top plate of the cryogen free system*

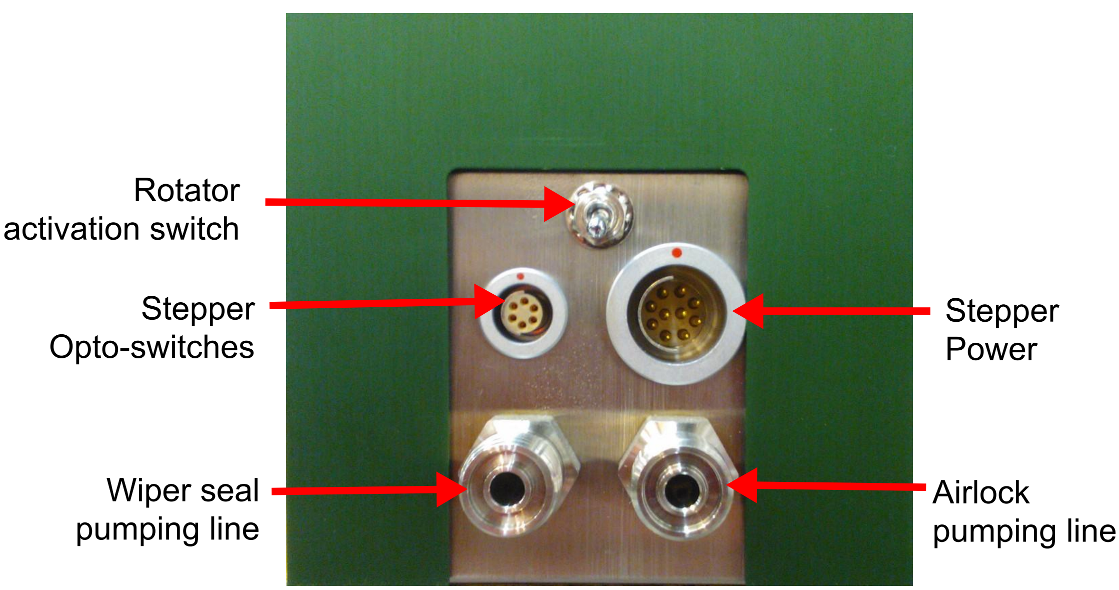


Figure 4.5: *Connections on the back of the "stepper unit", i.e. the airlock and sample movement unit.*

to the Lakeshore temperature controller with a special cable, provided with the system. The thermometer is read via one of the lakeshore inputs and the heater can be connected to the heater output. In this way the temperature can be controlled if desired. Alternatively the heater can be connected to a bench top power supply and the resistance of the thermometer monitored with a 4-terminal meter.

4.2 Cooling system from room temperature

4.2.1 Start up

Once all cables and gas lines are connected the rack may be switched on with the rack power switch located on the front panel of the valve block. After switching on the rack, perform the following three simple checks:

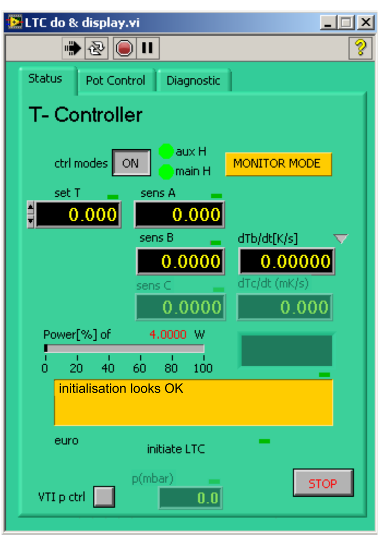
1. Check that the heat exchanger and sample thermometers on the Lakeshore 336 temperature controller are reading approximately room temperature.

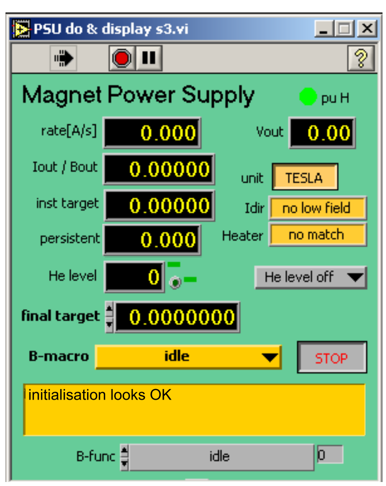
2. Check all LEDs on the front panel of the data acquisition unit are illuminated.

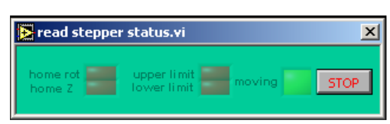
3. Check the computer is functioning properly.

If any of these checks are negative then switch off the rack and restart. If the problem persists then contact Cryogenic Ltd for support. If all these checks are positive then go ahead and start the system software "Sx_main.vi" either in the folder "S700" or via the short-cut located on the computer desktop. The main window will appear, to start the software running click the icon or select file→run. The main menu is shown in figure 4.6.

You will be asked to select a configuration file. The file "Jxxxx.ini"[1] contains all the default settings. Three more windows will appear and the software will attempt to establish communications with the temperature controller and magnet power supply.







[1]xxxx is your customer job number used for reference when contacting Cryogenic Ltd.

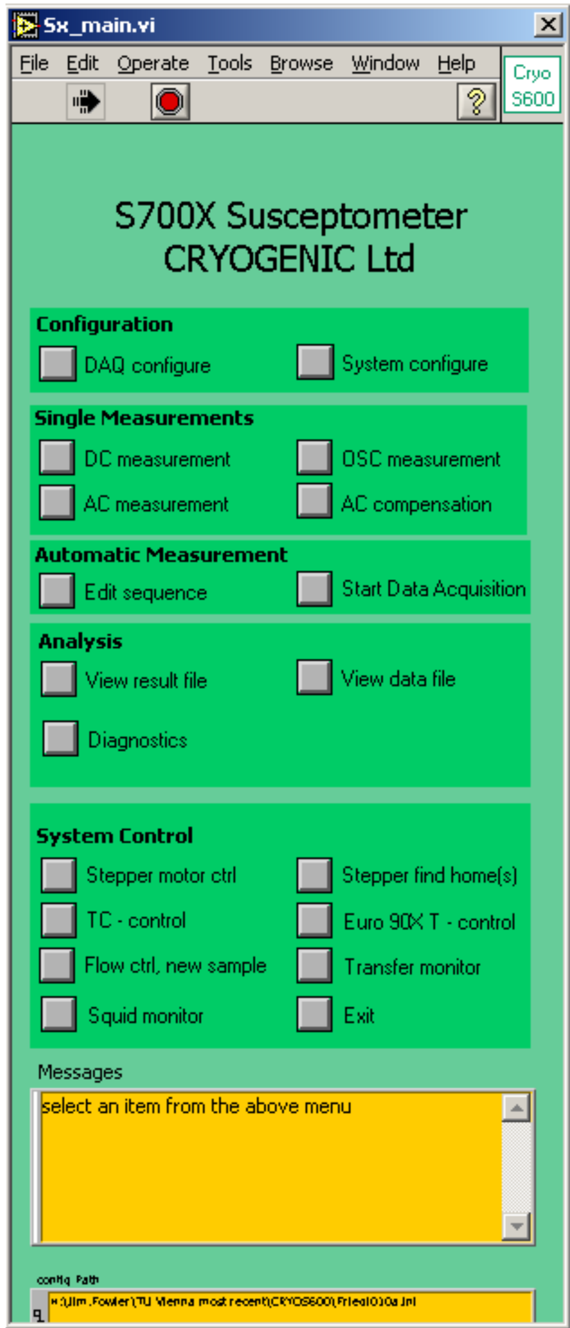
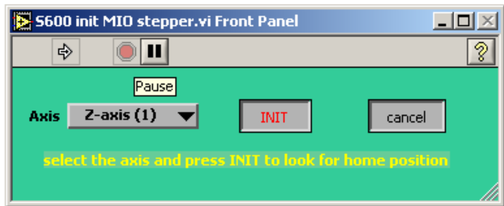


Figure 4.6: *Software main menu*

Once connected successfully, the message “Initialisation looks OK” appears in the temperature control and magnet control windows.

It is recommended at this stage to initialise the stepper motor zero position by clicking 

to open the stepper home panel shown here



Select the Z-axis and click 

.

4.2.2 Prepare for cool down

The cryostat is delivered under vacuum. Before cooling down the system, first check that the vacuum is better than 10^{-4}mbar and that you have a source of dry helium. This can be a clean cylinder of pressurised Helium or the boil-off gas from a liquid Helium transport Dewar. If there is a Helium recovery line in use in the laboratory then the exhaust ports of the cryostat and pump should be connected to it, following local guidelines.

The insert must now be flushed with helium gas for several hours. It is recommended to flush overnight. This removes air from the system which would freeze as the system becomes cold. Whenever Helium is being flushed through the Insert the cryostat must be pumped on to maintain vacuum. Helium will diffuse through the glass fibre into the vacuum space of the cryostat and this must be removed. At low temperature this diffusion becomes negligible.

The VTI vacuum space is also pumped out before delivery, typically to 10^{-5} mbar. The VTI contains charcoal that will absorb the small amount of helium that diffuses through its glass fibre outer tube at low temperatures. During any cool down of the VTI, on installation or after any time that the insert is removed from the cryostat, the VTI vacuum space should be pumped on to keep the charcoal free from gas. In this way the charcoal will remain good for a year before needing to be warmed up and pumped again. The VTI vacuum space is pumped out via a combined over-pressure and pumping valve. This is a safety over-pressure valve that can be opened using the special adapter provided in order to pump.

- Connect the source of helium to the NW16 port on the front of the electronics rack.
- Use the software to close internal valves. On the main menu click 

which opens the flow control window and choose **“Pump + Valves Shut”** from the **f-func** menu.
- Open the Helium fill port on the front of the VTI insert head.
- Start pumping on the cryostat through the vacuum port.

(a) *Lakeshore temperature controllers displaying thermometer readings. The upper controller (332 or 335) displays the temperatures of the main and mini sorbs on the ^{3}He probe (chapter 10). The lower controller (336 or 340) displays the VTI thermometers A (heat exchanger) and B (sample) , see chapter 3.1.4, and the ^{3}He probe sample temperature. Sensor D on the 336/340 is the voltage across the pressure transducer in the ^{3}He probe gas chamber.*

(b) *Front panels of the Helium level gauge, SQUID output meter and Data Acquisition system (DAQ). The DAQ front panel shows the status of the valve block. Red and green LEDs show the state of valves and pumps, green for open/on and red for closed/off.*

Figure 4.7: *Rack front panel indicators*

(a) *When not pumping via this port the cover should be screwed in place to catch the blow-off valve in the event it blows off.*

(b) *This is the pumping port for the VTI vacuum space.*

(c) *To pump on the VTI vacuum space, use this adapter. Pull back the plunger, fit the cylinder over the valve, screw the plunger into the valve, then pump out the cylinder before pulling back the plunger to open the valve.*

(d) *The vacuum inside the VTI will pull the valve closed but it can easily be held open, e.g. with the valve cover as shown.*

Figure 4.8: *Pumping on the VTI vacuum space*

- Start pumping on the VTI vacuum space as shown in figure 4.8.
- Begin helium flow to flush helium through the insert.

Flushing through the service port is the best way because the gas lines are also flushed. There is an internal safety valve so there is no risk of over-pressure. A steady flow of helium should be kept flushing through the insert for at least an hour and preferably over night.

Standard Cryostat

4.2.3 Pre-cool Helium reservoir

To cool and fill the Helium reservoir from room temperature using only liquid Helium would be very wasteful and expensive. It is therefore recommended to pre-cool the reservoir with liquid Nitrogen in one of the following two ways.

Method 1: Radiation.

The most gentle way to per-cool the Helium reservoir is by radiation from the 77K Nitrogen cooled shield. Fill the Nitrogen reservoir and leave for approximately four days, refilling each day, until the thermometer in the tail of the cryostat reads under 120K. The thermometer can be read using any four-terminal digital volt meter (DVM); a calibration table is provided. The thermometer can also be read using the Lakeshore temperature controller on the electronics rack (section 4.1.1).

It takes approximately 100 litres of liquid Nitrogen to cool and fill the Nitrogen reservoir and a further 30 litres per day whilst waiting for the Helium reservoir to cool to 120K. The Helium reservoir can now be filled with liquid Helium from a transfer Dewar. Skip to 4.2.6.

Method 2: Liquid Nitrogen

The fastest way to cool the Helium reservoir is to fill and cool it directly with liquid Nitrogen. This saves time and uses less Helium as the reservoir is colder than in method 1. The downside to this method is that it can be difficult to completely remove all of the liquid Nitrogen from the reservoir before filling with liquid Helium. Nitrogen is solid at liquid Helium temperature and so any Nitrogen that remains when Helium is added will freeze. Tiny amounts of Nitrogen ice are not problematic but significant amounts can block the VTI fill line.

Provided with the system is a “Nitrogen fill/push out tube”. This is a stainless steel tube with a PTFE end piece that fits into the top cone of the siphon extension inside the insert (see figure 4.9).

- Disconnect the Helium return line so that it can not be contaminated with Nitrogen. The Nitrogen boil-off gas must be free to escape and the room must be sufficiently well ventilated.
- The thermometer in the tail of the cryostat can be used to monitor the reservoir temperature during cool-down as in method 1.

- Insert the Nitrogen fill/push out tube into the cryostat siphon port until the PTFE cone fits into the siphon extension tube on the insert. Connect the other end of the tube to a liquid Nitrogen Dewar.
- Slowly open the main valve on the Dewar to begin transferring Nitrogen.
- Once the temperature reaches 77K add a further 25 litres of Nitrogen and leave over night.

The reservoir itself is now at 77K and can be emptied of Nitrogen and filled with Helium. However, extending the precook time to 48 hours reduces the Helium used in cooling to 4K by approximately 30 litres due to the gas cooled radiation shield reaching a lower temperature and being more effective.

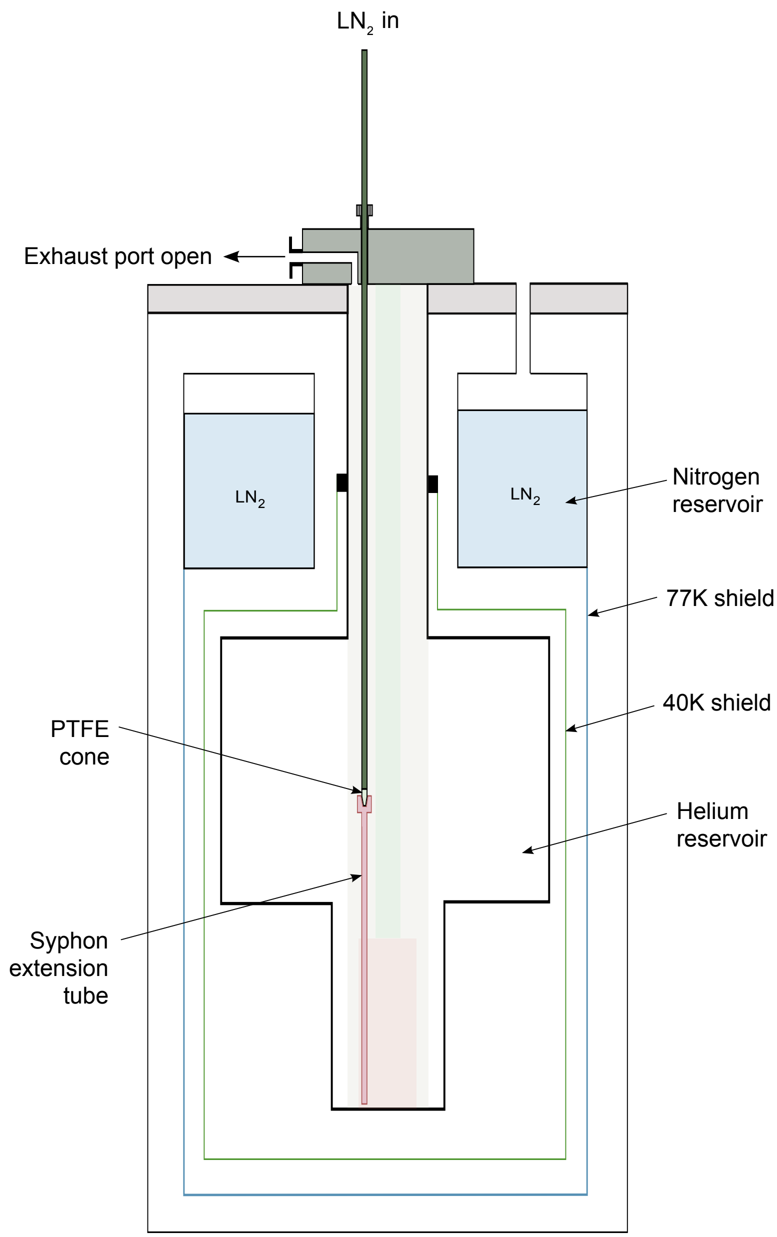


Figure 4.9: *Pre-cooling Helium reservoir with liquid Nitrogen. Ensure exhaust port is open and the room is well ventilated. Insert fill/push out tube until PTFE cone fits into siphon extension tube.*

4.2.4 Push out Liquid Nitrogen

Before the Helium reservoir can be filled with liquid Helium it must be emptied of Nitrogen to prevent damage from Nitrogen ice. It is very important to *push* the nitrogen out. **Do not *suck* it out by pumping.** Trying to pump the Nitrogen out will cause it to freeze.

If the reservoir temperature is 77K it is very likely there is a significant amount of liquid Nitrogen remaining. If the temperature begins to rise it is likely that no liquid is left. To test for liquid Nitrogen simply apply a small amount of heat to the reservoir (4.1.1) to raise the temperature slightly and then switch the heater off. If the temperature falls back to near 77K there is liquid Nitrogen present. If not then all the liquid has boiled off and Helium may be added straight away. In this case skip to 4.2.6.

To push out liquid Nitrogen:

- Insert the Nitrogen fill/push out tube into the cryostat siphon port until the PTFE cone fits into the siphon extension tube in the insert.
- Attach a length of suitable flexible tubing to the top of the push out tube to direct the liquid Nitrogen safely. The Nitrogen can be pushed out into a Nitrogen bucket but a much neater solution is to push it out straight into the Nitrogen reservoir (see figure 4.10).
- Pressurise the Helium reservoir to force liquid Nitrogen up and out of the tube. This can be accomplished in several ways:
- A nitrogen or helium cylinder set to an over-pressure of 1 bar.
- Boil-off gas from a liquid Nitrogen or Helium Dewar.
- The boil off gas from the Nitrogen in the Helium reservoir itself. Close the exhaust port and use the heater to increase boil off and create over-pressure.

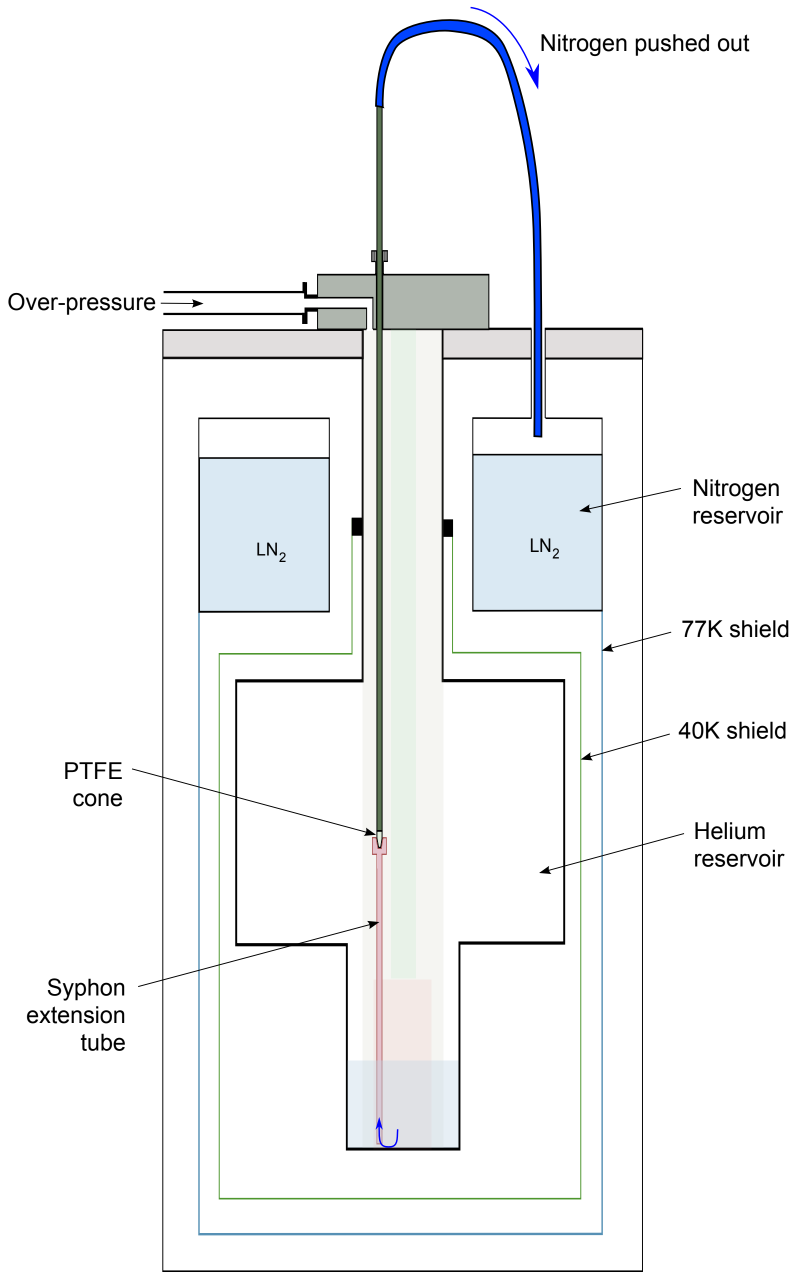


Figure 4.10: *To push out liquid Nitrogen from the Helium reservoir, insert fill/push out tube until PTFE cone fits into siphon extension tube and a suitable flexible tube to direct Nitrogen into Nitrogen reservoir or bucket. Apply over pressure as described in main text.*

Recondensing Cryostat

4.2.5 Monitor Cool-down

With the recondensing cryostat there is no need to precook with Nitrogen as the cooling is done entirely with the cryocooler. Before switching on the cryocooler it is advisable to start the temperature monitoring software located in ***S600\Keithley Software\Scanner.llb.*** When you run this software you will be asked for an initialisation file, stored in the same folder. Begin a new temperature log and then switch on the helium compressor to begin cooling down.

It is possible to let the system reach 4 K and condense helium gas into the cryostat to fill the reservoir, but if you would prefer you can fill the reservoir in the same way as the standard cryostat after it has been pre-cooled.

4.2.6 Fill Helium reservoir

Once the Helium reservoir is pre-cooled and contains no liquid Nitrogen it can be filled with liquid Helium. The volume of liquid Helium required to cool and fill the reservoir will depend on the method of pre-cooling and the temperature. The Nitrogen fill/push out tube must be removed if it was used for pre-cooling. Helium is transferred into the reservoir from a Helium Dewar via a vacuum insulated transfer siphon tube. You will need a suitable adapter, or "transfer cap", for fitting the siphon leg into the neck of the Helium Dewar. You will also need a means of maintaining overpressure in the Dewar, such as a compressed Helium cylinder.

It is important to prevent the cryostat top plate and the head of the insert from becoming iced during transfer. Rubber O-ring seals can be rapidly degraded by freezing and ice and condensation can cause significant damage to the wiring. To prevent this use a hot air gun directed at the exhaust port of the insert throughout the Helium transfer. There is also a 33 Ω resistor in the VTI head which can be used as a heater[2]. To use this, connect a bench-top power supply to the 3-pin Fischer connector on the back of the VTI (shown in figure 4.2) and put no more than 0.1A into the resistor.

The method of helium transfer outlined here is only one of several possible methods. Some laboratories have different transfer practices. Local guidelines should be followed.

- Unplug the SQUID electronics (PFL100) 10 pin LEMO connector from the VTI and store it safely. This is to save the electronics from becoming too cold.
- Position the liquid Helium Dewar close to the cryostat.
- Fit the PTFE cone from the Nitrogen push out tube to the cryostat end of the siphon transfer tube.
- Fit the other end of the siphon into the top of the Helium Dewar.
- Start the warm air gun directed at the insert exhaust port.

[2]In addition to the hot air gun, but not instead of.

- Lower the siphon until it touches the liquid helium. This will increase the boil off in the Dewar and slightly increase the pressure. The main/return line valve on the Helium Dewar should be closed to allow pressure to build up.

- Allow helium gas to flow through the siphon to expel the air.

- Insert the free end of the siphon into the cryostat Helium port until the PTFE cone fits into the insert's siphon extension tube.

- Slowly lower the siphon into the Helium Dewar until it reaches the bottom, keeping the over pressure in the Dewar below 0.1 bar for the first five minutes. Retract the siphon by a few centimetres so it is not touching the bottom.

- Use the helium cylinder to maintain the over-pressure in the Dewar between 0.1 and 0.2 bar.

- Monitor the the transfer using the thermometer in the tail of the reservoir and the liquid helium level gauge. Liquid helium begins to collect in the reservoir when the temperature falls to 4.2K, accompanied by a clear reduction in gas flow from the reservoir exhaust.

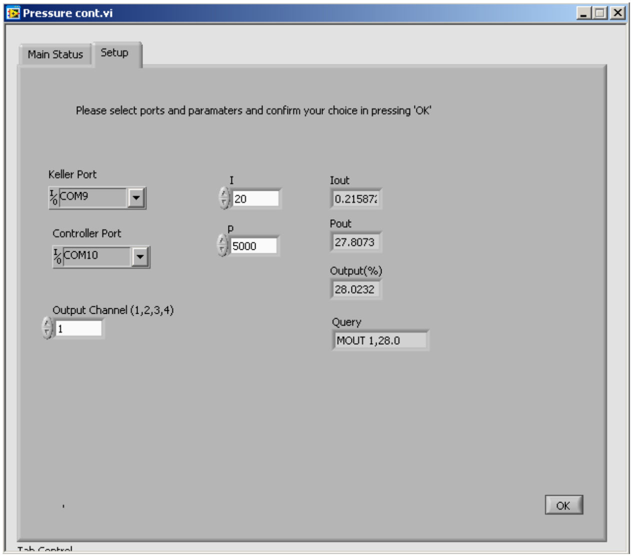
- When liquid starts to collect, the transfer rate can be increased by increasing the overpressure in the Dewar. Increase to 0.7 - 1 litre per minute maximum.

On the first transfer it is recommended to fill the reservoir to no more than 420-450mm depth. This is because equilibrium has not yet been reached and more helium than this will only boil off faster. On the second day fill the the reservoir to maximum (600mm). Leave the cryostat to reach equilibrium, approximately two to three days.

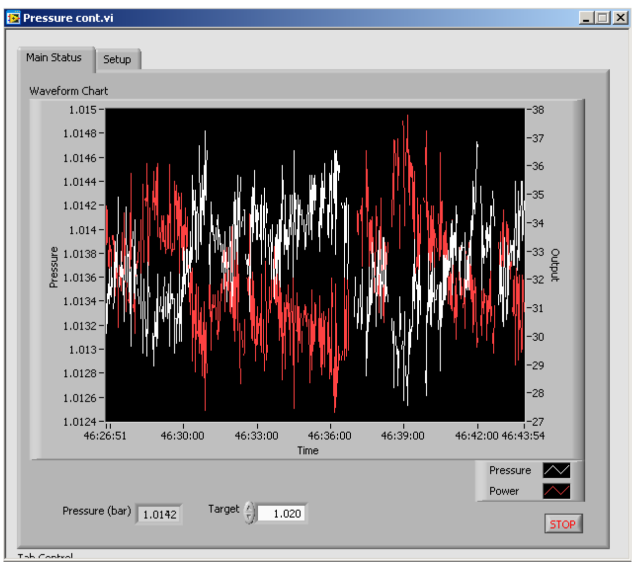
4.2.7 Reservoir Pressure

The recondensing cryostat works most efficiently when maintained at slightly above atmospheric pressure. The pressure above the reservoir is monitored with a digital manometer on the exhaust manifold. This is connected to the computer, allowing the software to automatically monitor and adjust the pressure using a resistive heater on the cryocooler to control the cooling power. This feature is controlled with the **Pressure Control** window. This vi can be found in the main software directory.

On opening the pressure control vi you see the setup tab



Use your computer's device manager to find out which ports the Keller manometer and the temperature controller to be used are plugged into and enter these values on the setup screen. Also enter which channel of the temperature controller the heater is connected to. Then press "OK". You will then see the main display screen which shows the pressure in the reservoir and the percentage heater power being put in.



Enter a target pressure in the **Target** box and hit return. The vi will now control the heater output to maintain the reservoir pressure to within a few millibar.

4.3 Connecting SQUID head

The SQUID itself is a very sensitive piece of electronics and care must be taken to prevent damaging the device. The PFL-100 head should never be connected or disconnected whilst the white cable is still plugged in to it. This prevents sharp voltage jumps across the SQUID which can damage it.

When connecting the head, plug the head into the appropriate socket and only then connect the white cable, powering up the unit. When disconnecting the head, unplug the white cable and only then unplug the head. To unplug the head, hold the unit and pull back the collar on the connector, then pull the unit away from the socket.

Connecting head

1. Head in

2. White cable in

Disconnecting head

1. White cable out

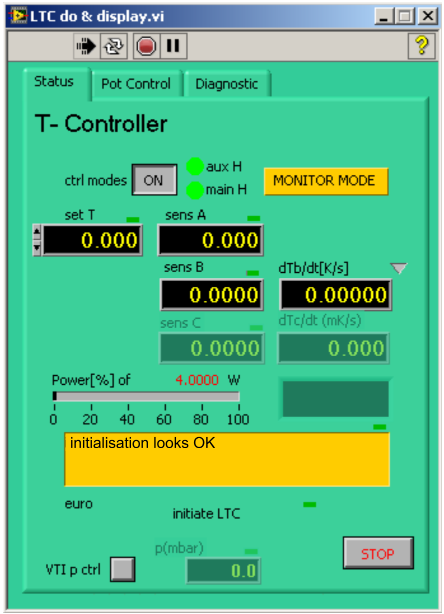
2. Head out

4.4 System Control - Temperature and Field

Before learning to take measurements we will first have a look at controlling the sample environment we will measure in. When the software is started and run, there are four windows visible, the main menu, the stepper motor status panel and windows for controlling the temperature and magnetic field. These last two, named **T-Controller** and **Magnet Power Supply**, are used to manually set the temperature and magnetic field. The LTC temperature control window, accessed from the main menu under TC control, can be used to monitor the temperature of the VTI thermometers over time but can not be used for temperature control unless using the ^{3}He insert, see chapter 10.

4.4.1 Temperature

The temperature control window looks like this



The temperatures measured at thermometers A and B are displayed in the boxes labelled **sens A** and **sens B**. To set a temperature enter the desired value in **set T** or use the scroll controls on the set T box. Temperature control is only possible with the **ctrl modes** button set to  . If this is switched off then the heaters are not used and the system will cool to the lowest temperature achievable with the current flow settings. The indicator lights **aux H** and **main H** indicate whether the auxiliary heater and main heater are active. The temperature control hardware is described in 3.1.4. The way that A and B are used to control and stabilise temperature is described in 13.1.

4.4.2 Gas flow and Needle Valve

The cooling power of the VTI is dependent on the flow rate of ^{4}He gas. This is controlled with the needle valve, labelled in figure 4.1. Opening the needle valve very wide means a large amount of gas can flow and the cooling power is increased, however the effect of expansion from liquid to gas is greatly reduced and the base temperature is higher. Conversely, a very constricted flow can achieve a lower base temperature due to the increased Joule-Thompson cooling as the liquid expands across the needle valve but the lower flow rate that results means it takes longer to cool the sample chamber. A balance must be found that is suitable for the particular temperature range you wish to measure in. This is one of the ways in which you can optimise the system to maximise the efficiency of the particular measurements you want to make.

If the optional motorised needle valve is installed for automatic control of the VTI pressure (and hence flow rate), it is activated with the **VTI p ctrl** button in the temperature control window shown in 4.4.1.

For a given temperature the software reads the reccomended pressure from a calibration file and adjusts the needle valve to achieve it. The target pressure is displayed in box shown. The pressure is checked and updated if the temperature changes by more than 5 K since the last adjustment.

4.4.3 Magnetic Field

The DC magnetic field is applied with the main magnet and controlled using the magnet power supply. The field is set by setting the current in the superconducting magnet. The magnet has a superconducting switch between the current terminals which completes the superconducting circuit so that the magnetic field is persistent and the current from the power supply can be reset to zero. When changing field the superconducting switch is driven into its normal phase with a heater and the current is ramped to the desired value. The switch heater is then turned off and the superconducting circuit is remade. The current from the power supply is then ramped back to zero.

To set a field:

- Enter the desired field in the "Final Target" box
- Select either "Set low field" or "Set field" from the "B-Macro" drop down menu
- "Set low field" is more precise than "Set field" for fields up to 30 mT
- Wait for "field OK" to appear in the status box. Whilst the field is still ramping the pick up coils are disconnected and it is not possible to make a measurement.
  - ***Do not*** *attempt to perform a scan whilst the field is ramping*

After changing the field it is advisable to monitor the SQUID output and wait for the drift to subside, see chapter 12.

Figure 4.11: *Examples of mounted samples.*

4.5 Mounting a sample for measurement

To perform a measurement, a sample must be mounted onto a holder which in turn is screwed to the sample probe for putting into the insert. The probe is then moved vertically (or rotated for transverse measurements) so that the sample moves through the pick up coils. There are many ways to mount a sample. The choice of mounting must be made with careful consideration of the sample being measured. Figure 4.11 shows three samples mounted and ready to be measured. For small samples, a good way to mount them is as shown in the centre of this figure. In this example a small sample is attached to a stiff copper wire with superglue. Superglue works very well at low temperature provided that *as thin a film of glue as possible* is used[3].

If this method is not suitable, for example with larger samples that the glue may not hold securely or for powder samples, can be placed in a standard #5 pharmaceutical capsule and held in a drinking straw as shown on the left of figure 4.11. The straw and the capsule should ideally be clear plastic and not coloured as the coloured dyes are slightly magnetic and will give an unwanted background to the measurement. The sample should not move relative to the rod or straw during a measurement. If a capsule is being used then sometimes the sample may be large enough to stay in place through friction. If not then the sample can be superglued to the inside of the capsule, or the capsule can be cut shorter to hold the

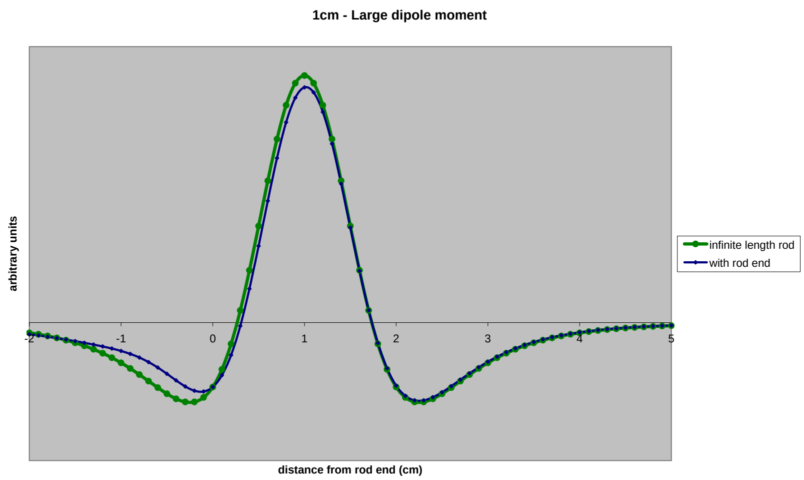
[3]It is a good idea to test the adhesion of any glue used by pouring a little liquid nitrogen over the sample before inserting the probe

sample (it will then need to be glued closed).

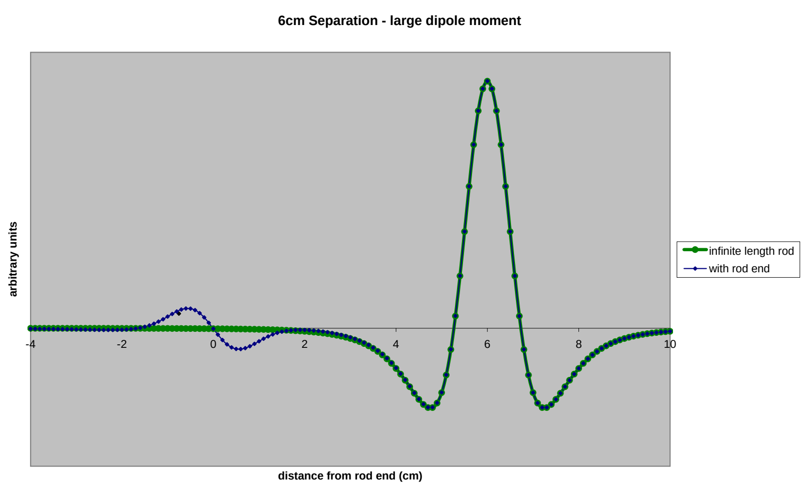
There are two types of sample rod adapter. In figure 4.11 the left hand image shows a hinged adapter, the centre a rigid one. Hinged adapters are the preferred option because they allow the sample rod to hang freely and be centred by gravity. Stiff sample rods are more prone to warping with changing temperature which reduces the reproducibility of the measurement. The hinged connector also damps out residual vibrations from the stepper motor. On the right hand side of figure 4.11 is a sample rod used for calibration. This is a length of Nylon studding with a small hole 6cm from the end. A Nylon nut screws on to the studding to cover the hole and hold the sample in place. This is used for calibration of the system using samples with extremely strong magnetic moments. The Nylon studding is attached to the rigid sample rod adapter with two interlinked lengths of wire that form a good hinge.

Whatever type of mounting is used the sample should be approximately 6cm from the bottom end of the sample rod. The susceptometer sees the end of the rod and at 6cm the signal from it is less than 1% of a typical sample signal. Figures 4.12 and 4.13 explain this in more detail. Much more than 6cm from the end of the rod and you run the risk of the rod hitting the bottom of the VTI during a scan. You also want to be as far as possible from the metal probe rod.

For the majority of samples, one of the mounting methods shown in figure 4.11 should be suitable, but with careful thought and some creativity it is possible to mount almost any sample successfully. If in doubt contact Cryogenic for advice on mounting samples.

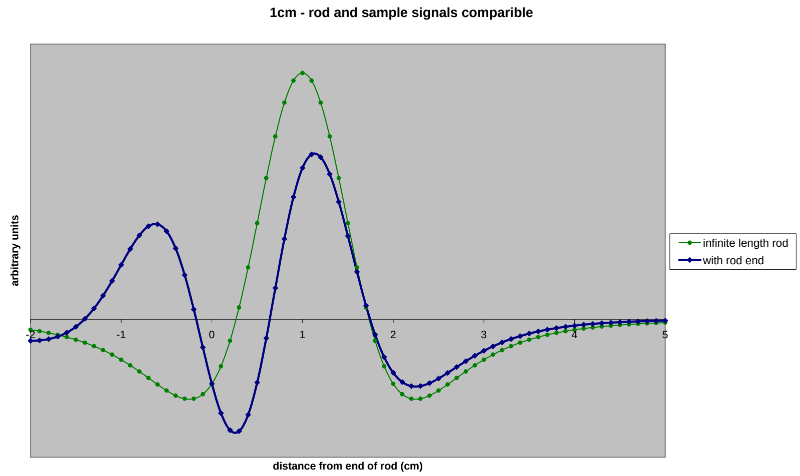


(a) *Large sample moment 1cm from end of rod*

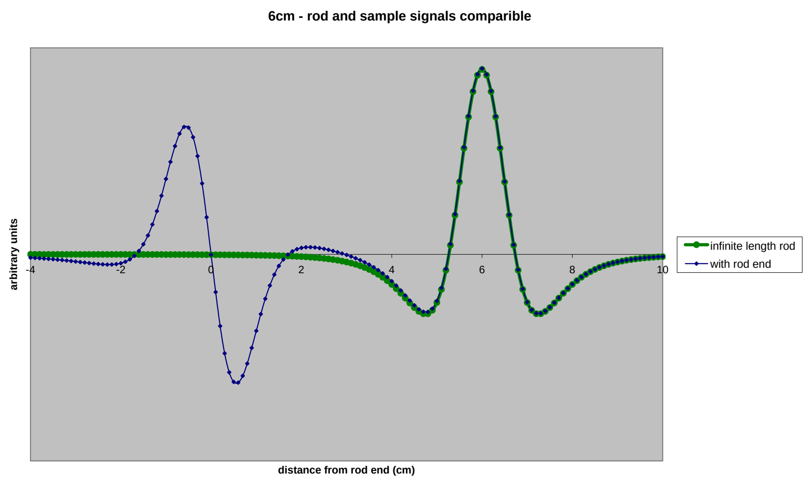


(b) *Large sample moment 6cm from end of rod*

Figure 4.12: *Simulated scans showing the relative effect of the end of the rod. The green curves show the dipole moment from an ideal sample without the presence of the end of the rod, as if the rod were infinitely long. The blue curves show the resulting scan when the end of the rod is present. If the sample has a very large moment it can be seen that the rod has little effect even if the sample is close to the end.*



(a) *Small sample moment 1cm from end of rod*



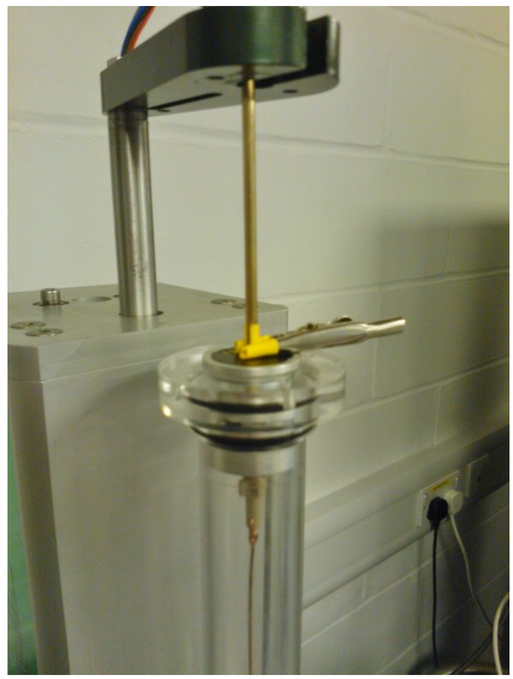
(b) *Small sample moment 6cm from end of rod*

Figure 4.13: *Curves are defined as in figure 4.12. It can be seen that for smaller sample moments the end of the rod can have a significant effect if the sample is positioned too close. At a distance of 6cm the effect of the end of the rod is less than 1% of the sample signal.*

4.6 Inserting the sample probe

With a sample mounted onto a suitable rod and rod adapter it can be screwed to the probe end. Once the adapter is securely fixed to the probe, pull the extraction rod through the airlock until the sample rod is completely within the airlock. Now insert the probe in the following way.

- Insert the airlock into the bayonet fitting on the top of the insert. Turn the locking ring clockwise to secure the airlock in place. Use a suitable clip to secure the probe and stop the rod being sucked down into the airlock when the airlock is pumped out. If this happens it can damage the sample rod or dislodge your sample.



- On the S700 software main menu click Flow ctrl, new sample. Figure 4.14 shows the flow control window that will appear and briefly describes its functionality.
- From the 'action' menu choose "load sample"
- You will be asked if the airlock is fitted. Select "OK" if you have fitted the airlock. Otherwise either fit the airlock before proceeding or click "cancel".
- The airlock will then be evacuated and flushed with Helium gas from the cryostat three times, each time pumping to progressively better vacuum. The final pressure is $\simeq 5\,mbar$.
- A simple leak test is then performed automatically. The pump is switched of and the pressure in the airlock is monitored. If it remains stable the program will continue, otherwise you will be prompted to investigate. In this case you are given the option to continue anyway or abort.

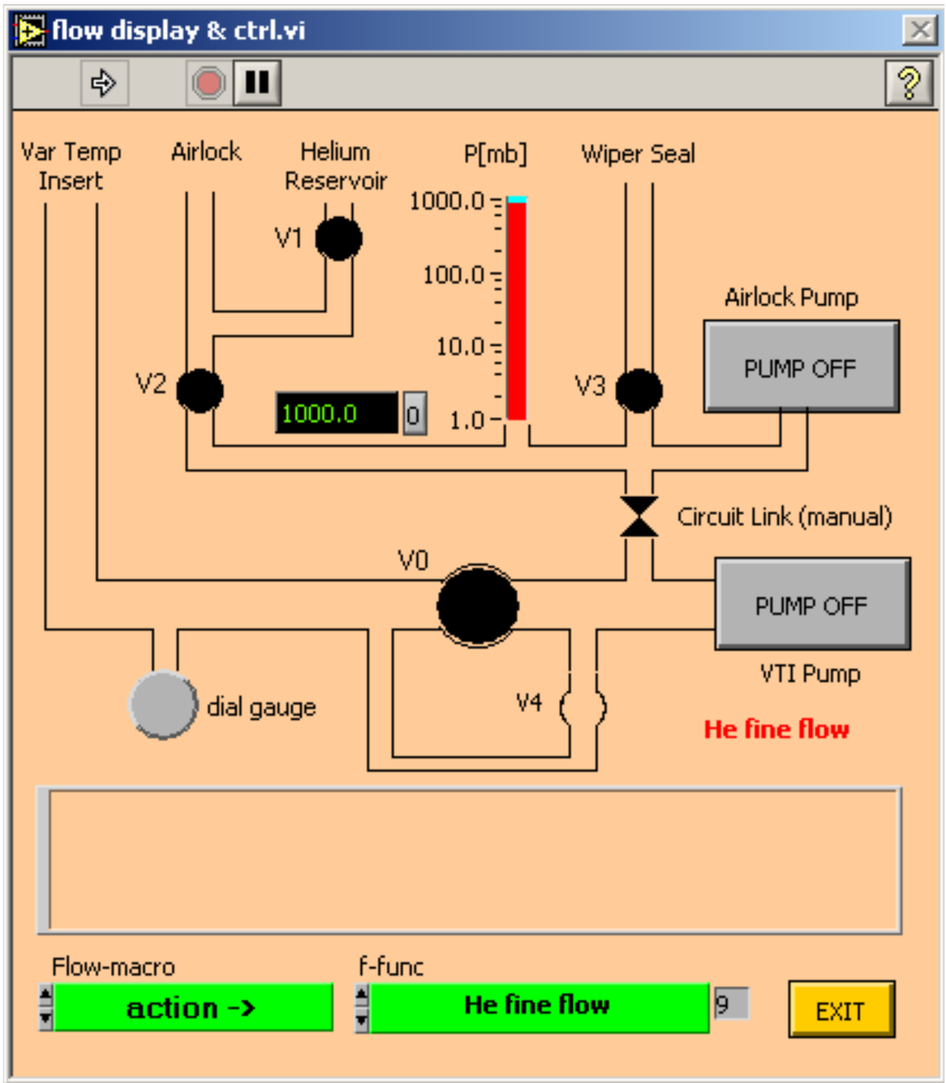
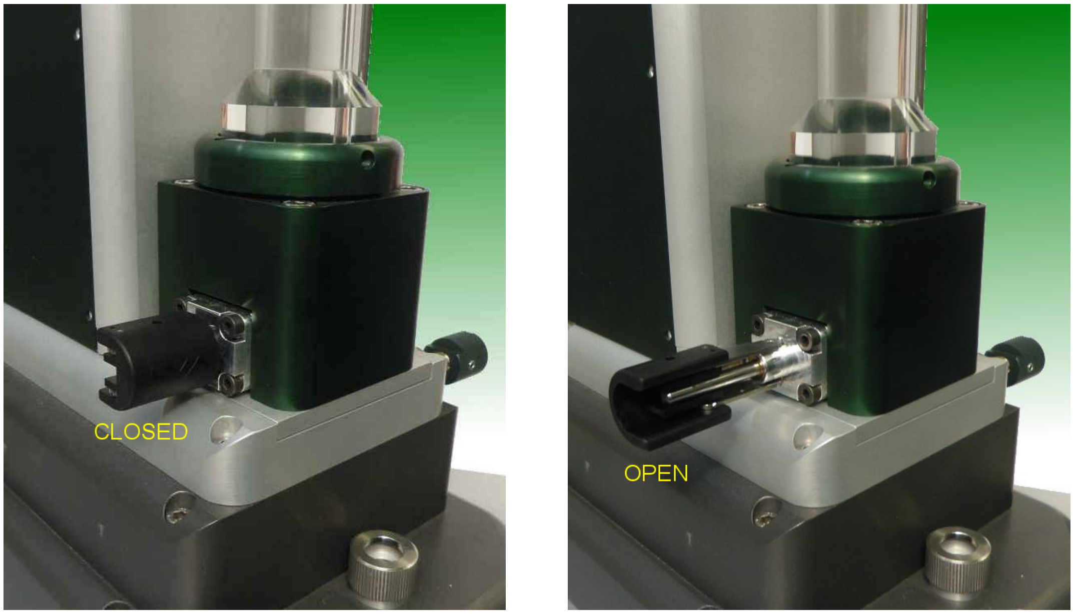


Figure 4.14: *The flow control window. Most of this window consists of a schematic diagram of the pumping system. There are three ways to interact with this window. 1) The valves can be opened and closed and the pumps turned on and off by clicking on the diagram. 2) You can select the state of the system from the "f-func" drop down menu. This sets valves and pumps automatically. 3) You can choose an action from the "Flow-macro" list. This guides you through loading or removing a sample, automatically setting valves and pumps when required.*

- On screen instructions will then guide you through the next steps.
- Open the gate valve.



- Lower the probe into the measure position. The bottom of the sample rod should be at least 2cm from the bottom of the VTI sample space.
  - *The on-screen prompt says "press OK when ready". This can be pressed before the sample is fully lowered but when it is pressed the pump will start. If the sample rod is too near the top of the VTI when the pump starts it will be pulled sideways which can damage the sample and its mounting. Make sure the sample probe is lowered at least until the brass section is no longer visible in the airlock.*
- Press "OK"

The system will now begin pumping on the Helium in the main reservoir and the temperature can be set. The default flow setting is "He Fast flow + ws" where "ws" stands for 'wiper seal'. This is the setting required to reach base temperature. Measurements may be taken in all flow settings but if the sample is to be moved, either vertically or rotationally, the wiper seal pump must be on to prevent contamination of the VTI.

A strength of the system is that because of the wiper seal the sample can be changed whilst the VTI is at any temperature. However, if the VTI is cold then the sample should be lowered slowly over a period of 5-10 minutes. If the sample is lowered quickly whilst the VTI is cold then the sample space will become warm and it will take up to several hours for equilibrium to return. By lowering the sample slowly the sample and VTI remain in equilibrium and by the time the sample is fully lowered it is at the correct temperature and ready to measure.

4.7 Centring the sample

To achieve the best results the sample should be well centred in the pick up coils. After the centring has been performed once it is possible to mount samples and adjust the sample probe such that the sample will be in the same position each time, making centring quick and simple. This can be done in several ways, for example making a mark on a work bench at the right position or, if the same sample rods are always used, marking the probe where the top nut should be. You may find your own way to reproducibly mount samples for good centring. However, for first use or if centring is lost for some reason, the centre position can be found as described below.

The extremely high sensitivity of the SQUID device means that for most samples the residual field present is sufficient to detect the sample without the need for an applied field. For some samples however, such as very small paramagnetic or antiferromagnetic samples for example, a small bias field may be required. $10\,mT$ should be sufficient in most cases. To apply a bias field use the "Magnet Power Supply" window, as described in 4.4.

Now we can perform a simple DC scan to locate the sample and centre it in the pick up coils. Select "DC Measurement" from the "Single Measurement" panel on the main menu. The measurement control window that appears is shown in figure 4.15. Use the following settings for a simple centring scan, also shown in figure 4.15. For more details on the controls in this window see chapters 7 and 13.

1. **Axis #1** (default) - Axis #1 is vertical. Axis #2 is rotational.

2. **Scan limits:** High 2.0 / Low -2.0 (default) - this gives a 4cm scan of $\pm 2cm$ about the centre of the pick up coils

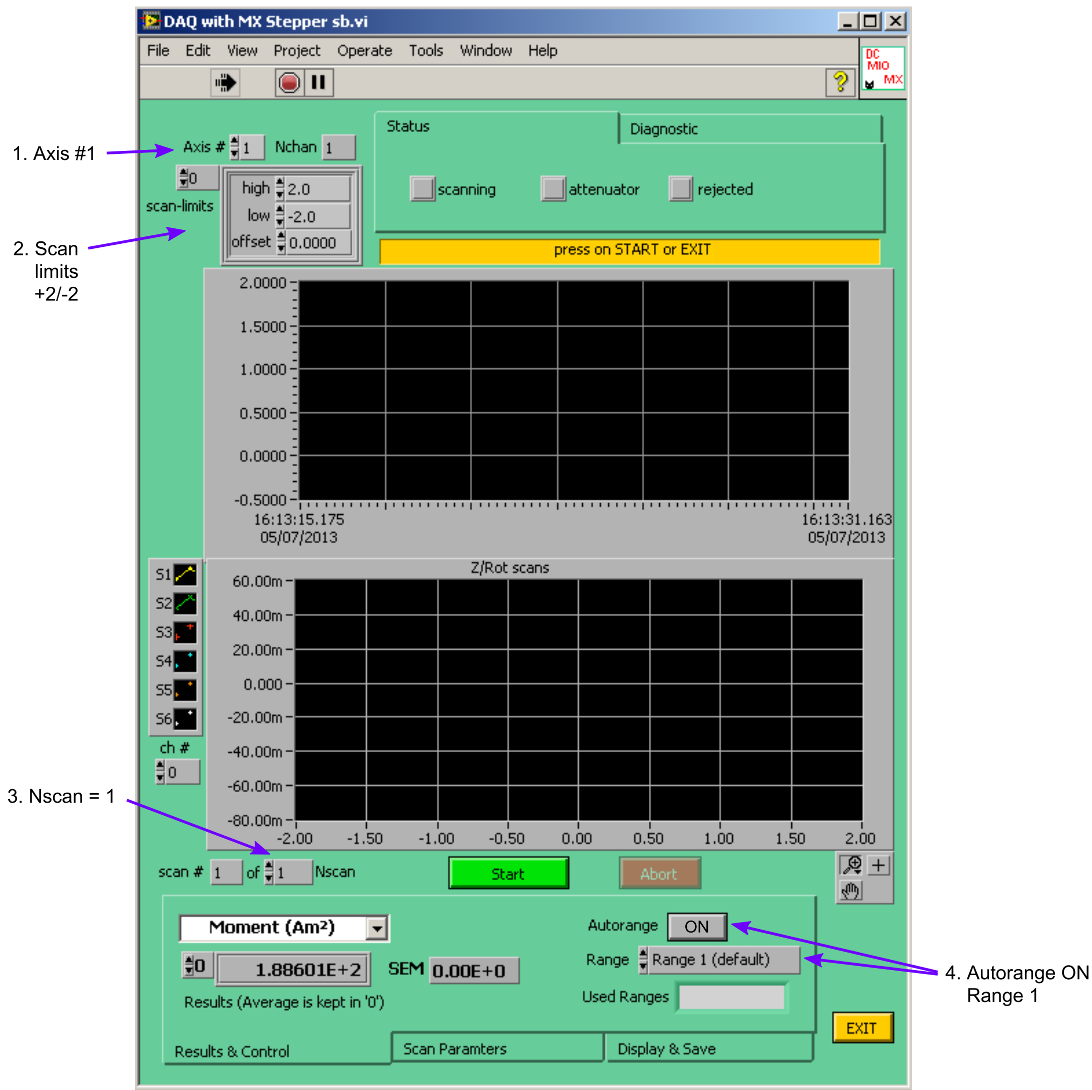
3. **Nscan = 1** (default) - the number of scans to perform. For centring one scan is sufficient.

4. **Auto range ON / Range 1** - Since we don't know how big the sample moment will be the autorange function should be switched on. The default range is 1 which is usually the right one. Starting with this range may save time.

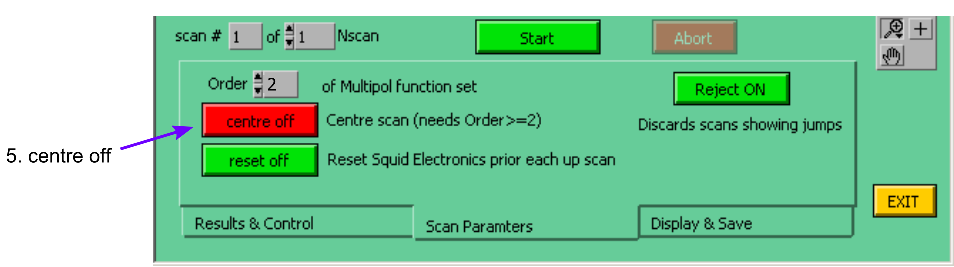
5. **Centre OFF** - Automatic centring should be turned off for the first scan when the position is not known as it is only for fine adjustment once the sample is centred to within a few millimetres.

With these settings we can now perform a scan by pressing "Start". The result is shown in figure 4.16. The upper graph shows the SQUID response with time as the sample is moved first up and then down through the coils. These scans are then both displayed in the lower graph as a function of position. Now we can use this information to find the sample position and adjust the probe to bring the sample into the centre of the pick up coils.

Finding the sample position Imagine a point at the centre of the pick up coils. This is where we want the sample to be after centring, but currently it could be above or below this point. The current centre point is moved to the bottom of the scan range as defined



(a) *First tab*



(b) *Second tab*

Figure 4.15: *The recommended settings for a simple manual centring scan.*

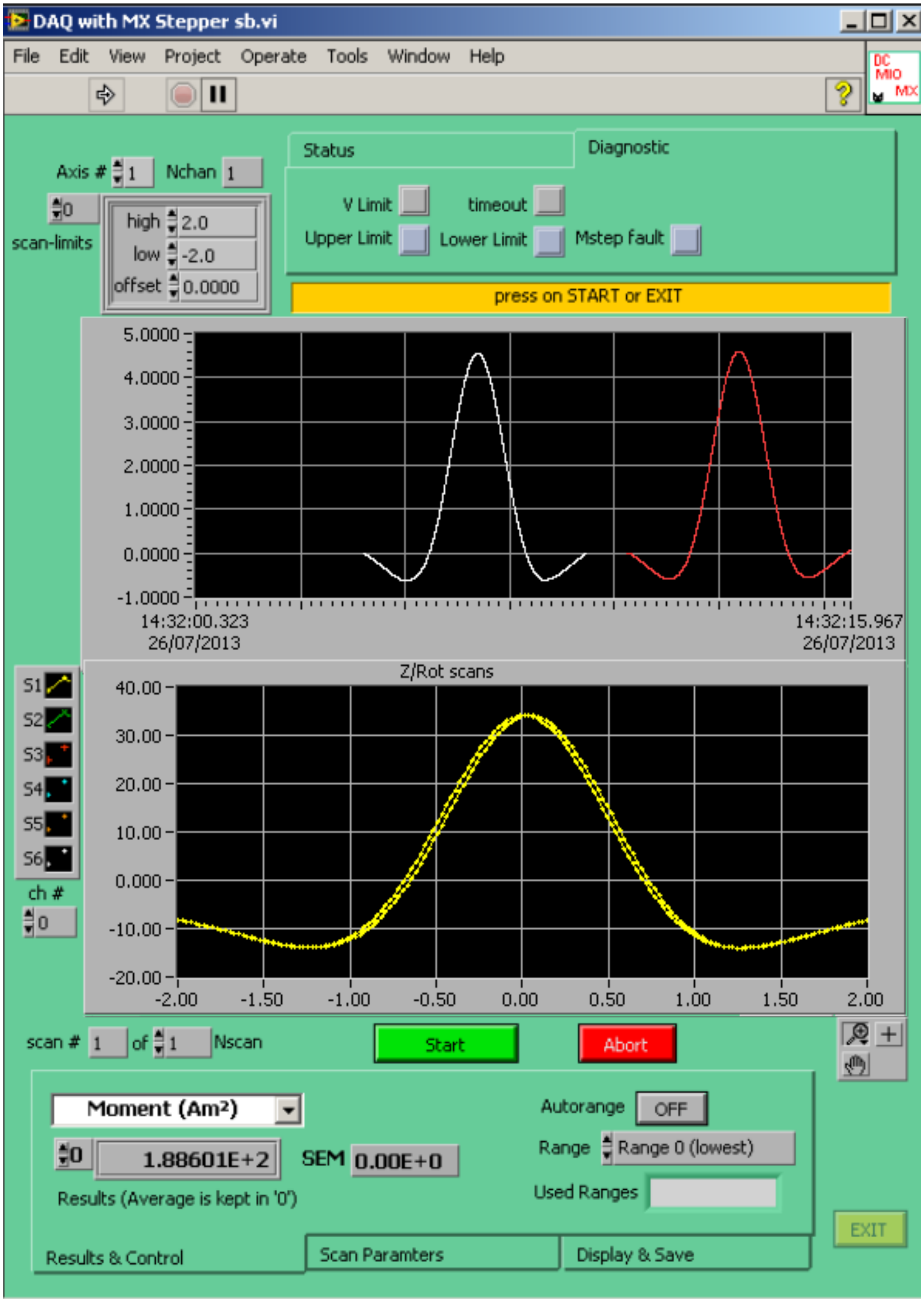
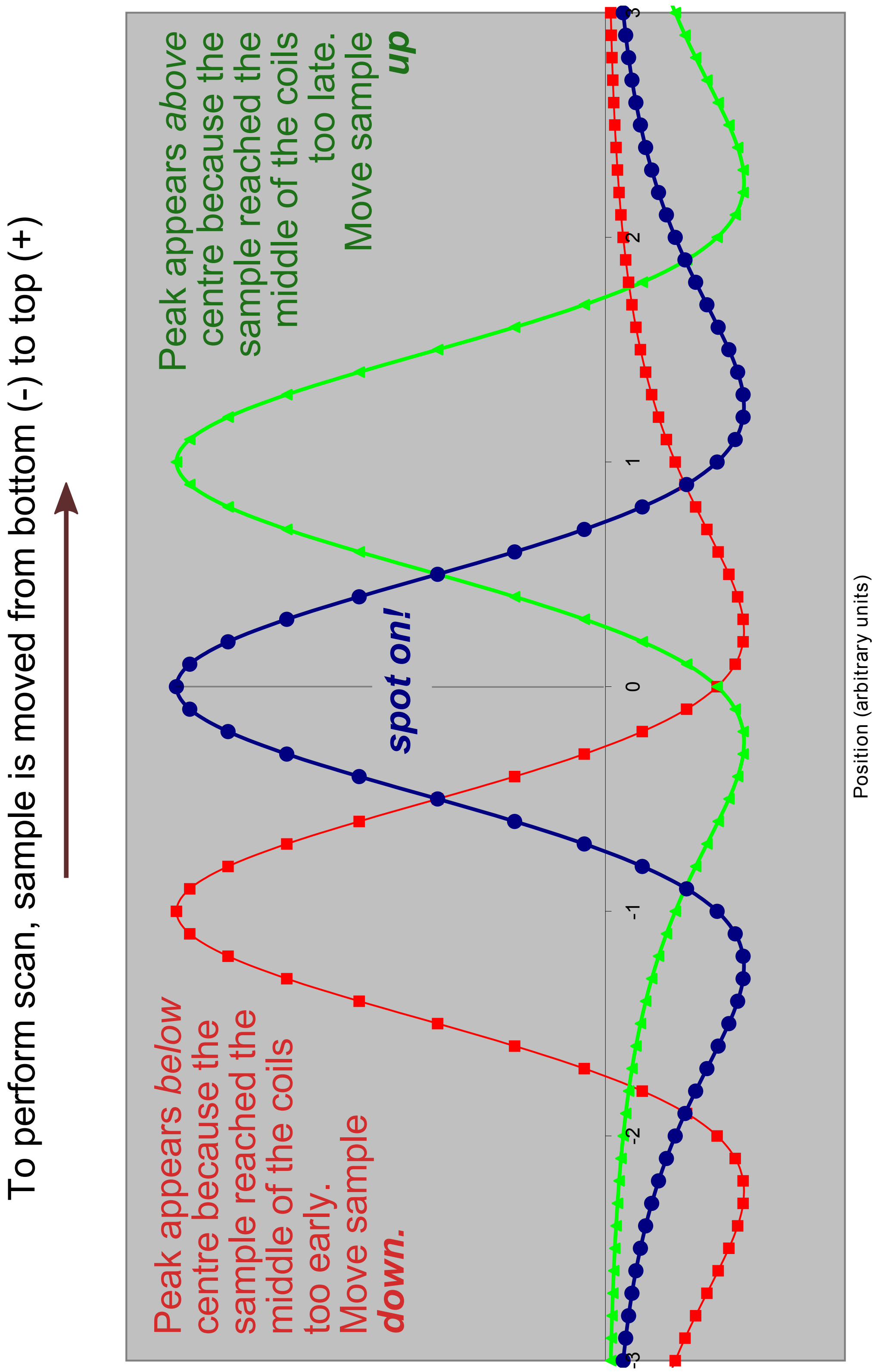


Figure 4.16: *The result of a simple centring scan. The upper graph shows the SQUID response as a function of time. The white curve represents the sample moving* up *through the coils, the red curve moving* down. *Both curves are then displayed as a function of position in the lower graph. Remember that since the red curve shows the sample moving in the opposite direction to the white curve, when they are displayed against position rather than time the red curve is 'folded' back on the white.*


To perform scan, sample is moved from bottom (-) to top (+)
Peak appears *below* centre because the sample reached the middle of the coils too early. Move sample ***down.***
spot on!
Peak appears *above* centre because the sample reached the middle of the coils too late. Move sample ***up***
-3
-2
-1
0
1
2
3
Position (arbitrary units)

by the "Low" scan limit above. Then the probe is raised smoothly until this current centre point reaches the top of the scan, defined by the "High" scan limit. The peak in the resulting scan corresponds to the sample passing through the middle of the pick up coils. On the scan, negative is below the middle of the coils and positive is above. It might appear then that if the peak is at a negative position, i.e. below centre, then the sample must be moved up, and vice versa. This is wrong because of the fact that it is the sample that moves, and not the detector.

If the sample were exactly at the centre point before the scan then the peak would appear exactly in the centre of the scan as shown by the blue curve in figure 4.17. If however the sample were too low then in would take longer to reach the middle of the coils and so the peak appears to the right of centre as shown by the green curve in figure 4.17. This means the sample must be moved up. Conversely if the sample were too high then it would take less time to reach the middle of the coils and so the peak would appear to the left of centre as shown by the red curve in figure 4.17.[4]

Manually adjusting the sample position decide which direction the sample must move as described above. For adjustments greater than a few millimetres adjust the position manually.

- Loosen the top nut of the sample probe, making sure the rod does not drop through or the sample rod may hit the bottom of the chamber.
- Carefully move the probe rod up or down through the nut the required amount.
- Tighten the top nut

Rescan and repeat the adjustment as necessary.

Automatic Sample centering Once the sample is centered to within a few millimeters the automatic centering process can do the rest. Adjust the scan settings as shown in figure 4.18. Auto-centering determines the sample position from the centre of the peak in the signal and offsets the centre position accordingly. It is a good idea to perform several scans so that you can see how reliable the calculated offset is.

Note: Auto centering will only work if the multipole function order is set to 2 or higher.

[4] Another way to think of it is that the sample moving *up* through the pick up coils is equivalent to the sample remaining still and the pick up coils moving *down* around it. In this frame of reference the peak in the scan then does show the actual position of the sample but with the sign reversed. For example, the red peak in figure 4.17 occurs where the coils (in our reversed reference frame) have travelled $3\,cm$ down, so in reality the sample has moved $3\,cm$ ***up***, i.e. it is at +1 and must be moved *down* one cm.

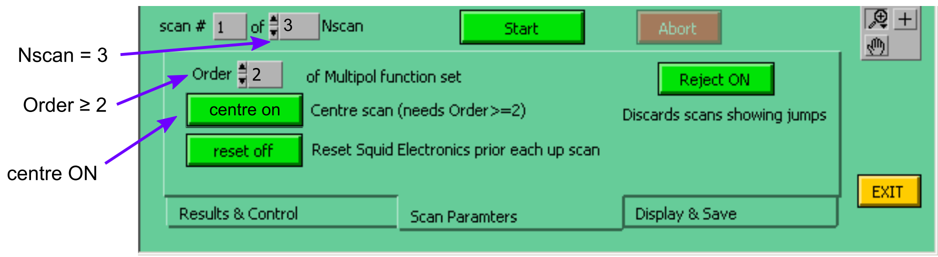


Figure 4.18: *Settings for auto-centering.*

4.8 Automated measurement sequences

Sequences allow you to perform a series of measurements on the same sample at various temperatures and fields. You may wish to measure DC magnetisation as a function of temperature in several different fields, or it could be that you wish to measure as a function of field at a single temperature, or a number of temperatures. You are not limited to performing the same type of measurement in one sequence. Perhaps you wish to measure both the longitudinal and transverse moments at each point, or maybe there are several experiments to perform on a single sample and you want to start a sequence that will run them all one after the other so the system can be left to measure say over night, or over a weekend.

Sequences are simply text files (.txt) that contain instructions to tell the system what conditions to set and what measurements to take. Sequence files can be created and edited with the built in sequence generator (see below) or written manually in any free text editor such as Notepad or Editpad lite. Sequences can also be created and saved with the built in generator and subsequently edited manually. Each row of the text file contains the instructions the system needs to measure a single data point, as in the example shown in figure 4.19.

```
0.000000  set field  10.000  Zscan  4.00  3  *  0  0
0.000000  set field  12.000  Zscan  4.00  3  *  0  0
0.000000  set field  14.000  Zscan  4.00  3  *  0  0
0.000000  set field  16.000  Zscan  4.00  3  *  0  0
0.000000  set field  18.000  Zscan  4.00  3  *  0  0
0.000000  set field  20.000  Zscan  4.00  3  *  0  0
0.250000  set field  10.000  Zscan  4.00  3  *  0  0
0.250000  set field  12.000  Zscan  4.00  3  *  0  0
0.250000  set field  14.000  Zscan  4.00  3  *  0  0
0.250000  set field  16.000  Zscan  4.00  3  *  0  0
0.250000  set field  18.000  Zscan  4.00  3  *  0  0
0.250000  set field  20.000  Zscan  4.00  3  *  0  0
0.500000  set field  10.000  Zscan  4.00  3  *  0  0
0.500000  set field  12.000  Zscan  4.00  3  *  0  0
0.500000  set field  14.000  Zscan  4.00  3  *  0  0
0.500000  set field  16.000  Zscan  4.00  3  *  0  0
0.500000  set field  18.000  Zscan  4.00  3  *  0  0
0.500000  set field  20.000  Zscan  4.00  3  *  0  0
0.750000  set field  10.000  Zscan  4.00  3  *  0  0
0.750000  set field  12.000  Zscan  4.00  3  *  0  0
0.750000  set field  14.000  Zscan  4.00  3  *  0  0
0.750000  set field  16.000  Zscan  4.00  3  *  0  0
0.750000  set field  18.000  Zscan  4.00  3  *  0  0
0.750000  set field  20.000  Zscan  4.00  3  *  0  0
1.000000  set field  10.000  Zscan  4.00  3  *  0  0
1.000000  set field  12.000  Zscan  4.00  3  *  0  0
1.000000  set field  14.000  Zscan  4.00  3  *  0  0
1.000000  set field  16.000  Zscan  4.00  3  *  0  0
1.000000  set field  18.000  Zscan  4.00  3  *  0  0
1.000000  set field  20.000  Zscan  4.00  3  *  0  0
```

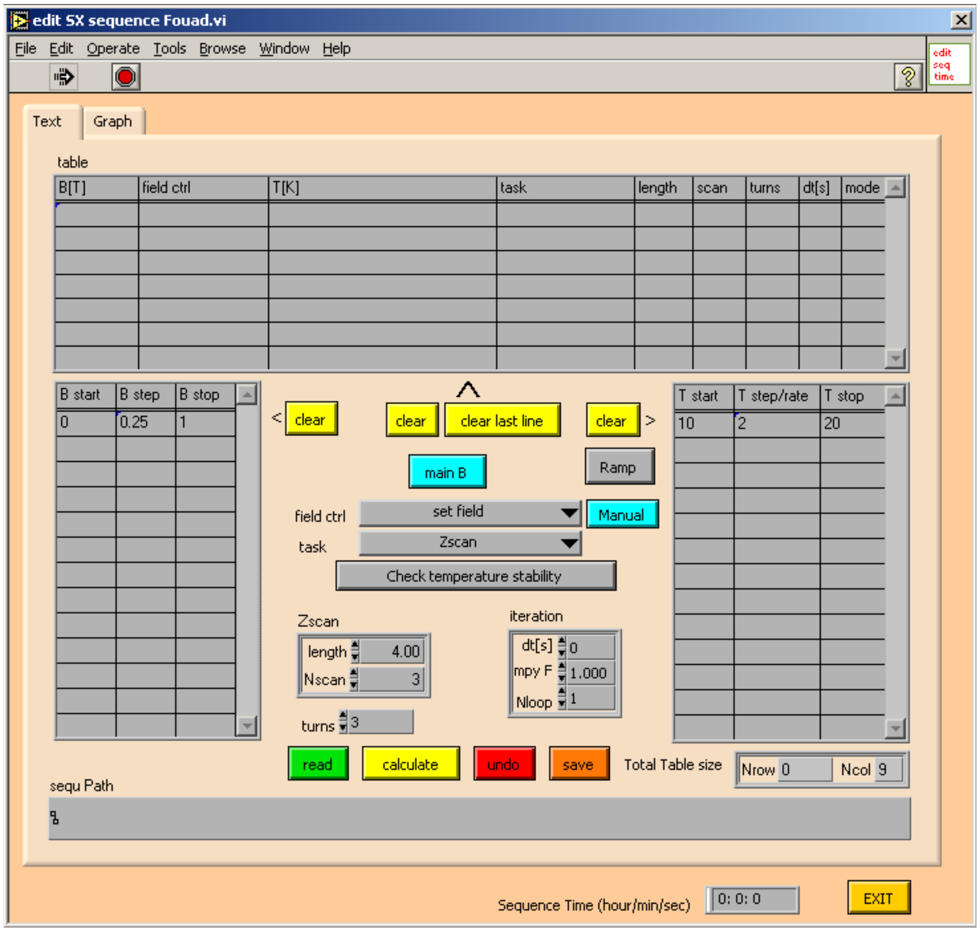
Figure 4.19: *Example sequence*

The first line of the example will set magnetic field to zero and temperature to 10K and then perform three 4cm longitudinal scans. The sequence then goes on to perform the same measurement at 12, 14, 16, 18 and 20K, all in zero field, followed by the same 6 temperatures at 0.25, 0.50, 0.75 and 1.00 Tesla. For a full description of the syntax used for manually writing or editing sequence files see appendix C.

Now let us look at how to create the sequence in figure 4.19 using the built in sequence generator.

4.8.1 Sequence Generator/Editor

To access the sequence generator choose “Edit Sequence” from the software main menu. The sequence editor window appears.



The first thing to do is decide what temperatures and fields you want to measure at and in what order. The "main" control button can be set to either "main T" or "main B". This button determines which parameter is defined by the outer loop of the sequence, see figure 4.20.

Main T Temperature is the outer loop. For each temperature set, an inner loop sets a series of magnetic fields and a measurement is taken at each.

Main B Magnetic field is the outer loop. For each field set, an inner loop sets a series of temperatures and a measurement is taken at each.

The main controls in the window are as follows:

Input/Output tables There are three tables. The left hand table is used to enter field settings, the right hand table for temperature settings and the upper table shows the sequence that is created. The input tables each have three columns. start, step[5] and

[5]The temperature table has step/rate, this is because when B is the main loop the temperature inner loop can be performed as a sweep rather than a series of discrete points. This can be used for example when using oscillating mode. The rate of the sweep is then entered in this column.

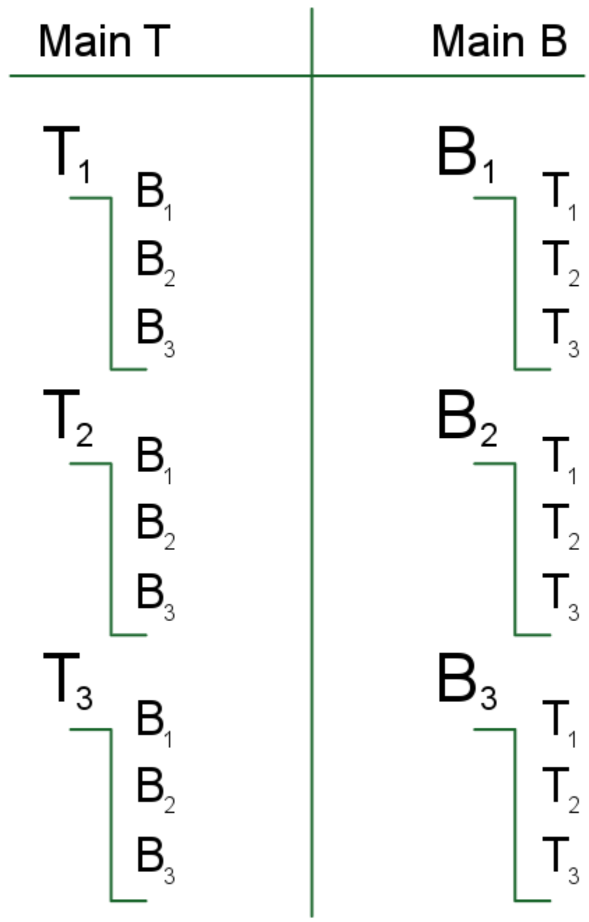


Figure 4.20: *The choice of temperature or field as main loop*

stop. Points will be generated from 'start' to 'stop' in increments of 'step'. Each table has a "clear" button to delete its contents.

main T/main B This is a toggle switch between the temperature and magnetic field as the outer loop of the sequence.

field ctrl Choose either "set field" or "set low field" from the drop down menu. This tells the program which option to use when setting the field. "Set low field" is more precise for fields up to 30 mT.

task Choose the measurement to make at each point in the sequence. The default is Zscan which performs a DC longitudinal measurement. See below for full description of tasks available.

parameters Define the length of a Zscan, the number of scans to perform and average for each point (Nscan) and for rotational measurements the number of turns.

iteration Each point can be repeated a given number of times. The iterations can be performed one after the other, or a variable delay can be imposed. See chapter 13 for more details.

Calculate When "calculate" is pressed each of the two input tables are turned into a sequence that performs the selected task at each point with the chosen settings and this sequence is appended to the upper table.

read loads a saved sequence for editing

To create our example sequence we do the following:

- Set the outer loop to "main B"
- In the left hand table enter $B\,start = 0$, $B\,step = 0.25$ and $B\,stop = 1$ (field points from 0T to 1T in steps of 0.25T)
- In the right hand table enter $T\,start = 10$, $T\,step = 2$, and $T\,stop = 20$ (temperature points from 10K to 20K in steps of 2K)
- Choose "set field" and "Zscan" from the drop down menus
- Make sure the button underneath the menus reads "check temperature stability"[6]
- Set Zscan $length = 4\,cm$ and number of scans $Nscan = 3$. Performs a 4cm scan and takes and averages three scans per point.
- Under 'iterations' set $Nloop = 1$ so that each point is only taken once.

[6]This control tells the program whether or not to wait for the temperature to be stable before making the measurement at that point. There are some circumstances when this should be set to "do not check temperature stability" but for the majority of measurements the stability should be checked. When sweeping the temperature as described in footnote 3 the program automatically selects not to check stability.

- Click “Calculate”. The sequence is now generated and appears in the upper table. Check the sequence to make sure it does what you wanted to do.
- Save the sequence.

Your sequence file is now saved and ready to use for taking measurements. The generator can also be used to edit existing sequences in several ways.

- The entries in the upper table can be edited directly.
- The “clear last line” button can be used to delete the last line of a sequence
- Sequences generated as described above are appended to what is currently shown in the upper table so if you want to extend the sequence with a new set of points you simply have to enter the new points in the input tables as before, click calculate and the new points will be added to the sequence file.

4.8.2 Tasks

idle takes no action

Zscan a DC longitudinal measurement

AC performs the measurements on the AC task list

Zscan & AC takes both measurements one after the other

Rot a DC rotational measurement. The length parameter is interpreted as an angle

Rot & AC Performs a rotation measurement and then an AC measurement

Zscan & Rot Performs a DC longitudinal measurement and then a rotation measurement...

Zscan & Rot & AC ...you get the idea

move Moves the sample to a position specified by the **length** parameter

rotate Rotates the sample rod to an angle specified by the **length** parameter

Oscilating an oscilating measurement

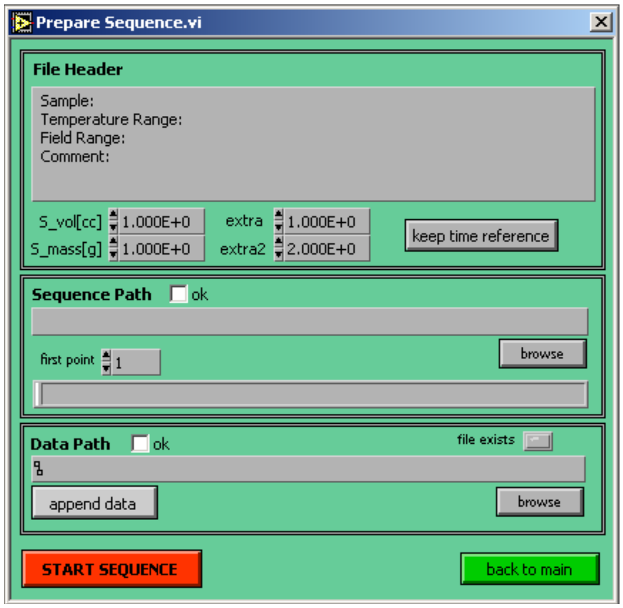
Resistivity If the resistivity option has been purchased this task takes a reading of resistivity[7]

Recondense Activates the recondense procedure if the ^{3}He probe is being used.

[7]By default this task calls the resistivity vi, but advanced users can configure the software to call any vi at this point, including external vi’s they have written for their own measurements and procedures.

4.9 Starting Data Acquisition

With a sample mounted, loaded and centred and a sequence ready to use we can begin to take data. From the main menu choose "Automatic Measurement → Start Data Acquisition". The "prepare sequence" window appears.



File Header Information entered into the "File Header" box will appear at the top of the data file where the data from the run is stored. This is a useful way to keep information about the measurement together with the data.

Time reference There is a toggle button to switch between "keep time reference" and "reset time reference". If "reset time reference" is selected then the time of the first measurement point is set to zero. If "keep time reference" is selected then the time is measured from the last time a sequence was run that did reset the time reference. This is useful for time dependent measurements where the same measurement is made in the same conditions at regular intervals a long time apart. For the vast majority of measurements it is more useful to reset the time reference.

Sequence Path Choose the sequence to use for the measurement. The tick box indicates if the given sequence exists at the specified path.

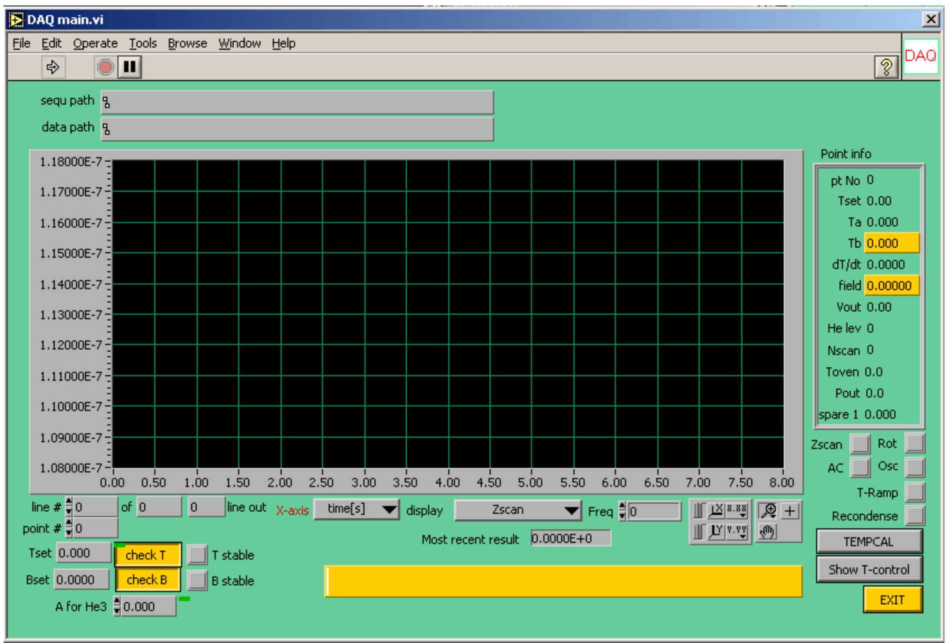
Data Path Name the file to write data to. Data can be written to a new file or appended to an existing one. The tick box indicates that you are not trying to create a new file that already exists or append to a file that doesn't. This prevents accidentally overwriting data files.

The sequence can only be started when both tick boxes are ticked. These boxes make sure that the program can find the sequence file you have specified and that you are not overwriting an existing data file. To begin taking data press 

.

4.9.1 On-screen results

As the sequence runs, the results of each measurement appear in the main data acquisition window



This window displays information about each point as well as a graph of the results. You can choose what to display on each axis via the drop down menus. If you click Show T-control so that the button turns yellow, then in between taking each point the graph will change to display the temperatures of VTI thermometers A and B as a function of time. This is useful for checking that the system parameters are producing a good, efficient temperature sweep with fast stabilisation at each set temperature. If you are planning on taking many similar measurements with the same temperature range and steps it is a good idea to spend some time optimising the system performance for this range to speed up the overall measurement process. By creating a sequence that sets each of the temperature points required but doesn't take any measurements (use **idle** as the task) then you can watch the temperature control in this window as each point is set and tweak the PID and auxiliary heater settings to optimise temperature control. Be sure to save the new settings in a different .ini file so as not to overwrite any settings you might want to keep.

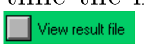
4.10 Viewing Results

The data recorded during a sequence is stored in a number of files. The file name is defined when beginning the acquisition. Several files are created with the given file name and different extensions, described below. There are two 'raw' data files and at least one "result" file. Under "Analysis" on the main menu are two options 

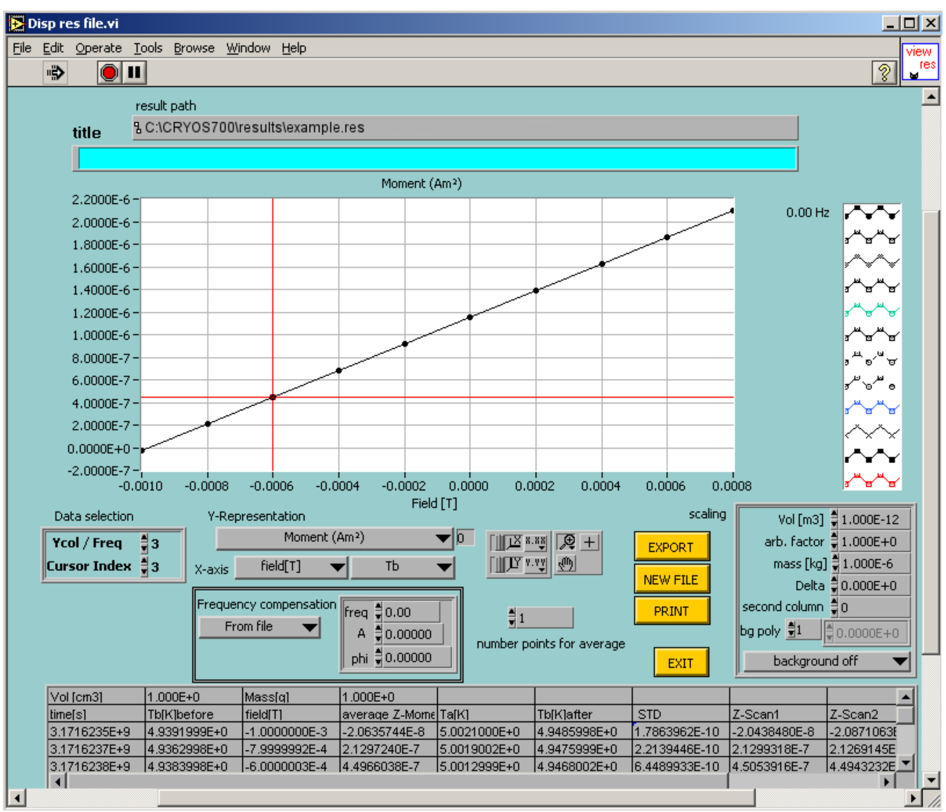
.

Result files These store the processed results of a measurement sequence. There are three types of result file. Zscan results are stored in '.res' files, rotation measurements in '.rro' files and AC measurements in '.rac' files. Each file type is only created if required so when running a sequence that involves only Zscan measurements the only result file created is a '.res', whereas if a sequence involves taking Zscan, rotation and AC measurements then you get a '.res', a '.rro' and a '.rac'.

Data files These store the raw data for more in depth analysis. There are two types of data file that together make up the raw data for every sequence. A '.dat' file contains all the information about an entire sequence in a compact, binary form and a '.wat' file contains every raw scan as measured voltage against time. The '.dat' file is partially processed in that it is scaled for certain specifics of the instrument, whereas the '.wat' file is truly *raw* data and is essential for advanced analysis.

Most of the time the first thing you will want to see after running a sequence is the result file. Choose 

from the main menu.



The most recent result file to be viewed is automatically loaded. To open a different result file click 

and choose the file via a standard dialogue box, the data is plotted in the graph panel. Use the Y-Representation and X-axis drop down menus and the Data selection box to select which data is plotted and how. The example above shows magnetic moment plotted against applied field.

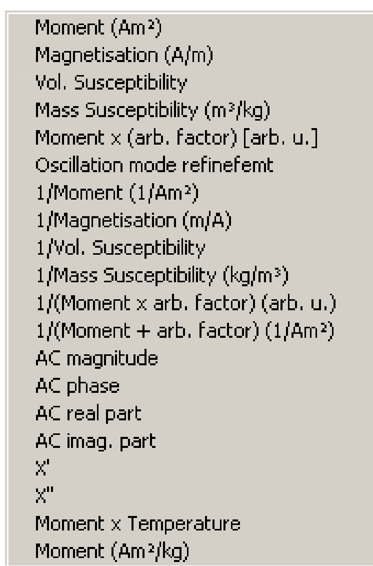
X-axis

There are three choices for the X-axis:

- Time (s)
- Temperature (K)
- Applied Field (T)

Y-axis

There are many more options for the Y-axis. What is displayed on the Y-axis depends on the choice of data column in the "Data selection" box and of representation in the "Y-Representation" drop down menu. The phrase "Y-Representation" is used rather than simply "Y-axis" because the result file viewer is more than just a graph plotter. There are some in built functions to interpret data in a number of ways commonly used by the magnetism community including:



Each representation assumes a particular data column or set of columns is used and combines them in particular ways with user-defined parameters entered in the "Scaling" box on the right hand side of the window. For example by default the representation is set to "Moment" which simply displays the selected data column, but choosing "Magnetisation" divides the selected column by the quantity "Vol" (volume). The columns are shown in a table at the bottom of the viewer. The column headings will depend on the type of result file, be it '.res', '.rro' or '.rac' but the columns are always numbered from left to right starting with time in column 0.

For now let us focus on the most common case of displaying magnetic moment as a function of temperature or field. Use the **Ycol / Freq** control to select the correct column. For a Zscan measurement the magnetic moment is stored in column 3. Select temperature or field for the X-axis.

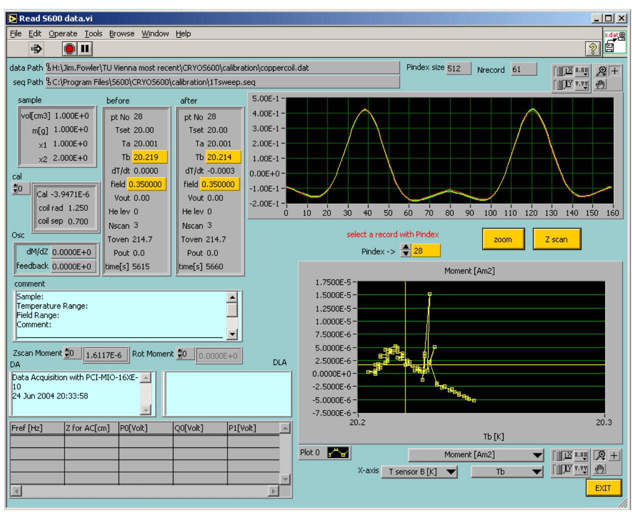
Exporting/Printing

PRINT Sends the graph in its current form to your default printer. The graph is printed along with the date, file name and title which you can enter in the blue bar under the file path.

EXPORT Saves the data currently displayed as a two column tab delimited file via a standard dialogue box. You may add the file extension of your choice, usually '.txt'.

4.10.1 A note on viewing Data files and advanced analysis

The alternative option to viewing result files is View data file, which opens the read data window.



This is used to view full information about each data point. Through this window you can use the zoom utility to assess the individual scans taken at each point and investigate the effects of various parameters on the results. This is covered in chapter 8.

4.11 Removing Sample

To remove the sample probe first make sure that no measurements are being taken. Then, if it is not already open, open Flow ctrl, new sample from the main menu. Under the **actions** menu, choose **remove sample**. An on-screen prompt will ask you to raise the sample until it is completely inside the airlock and then close the gate valve. When the airlock was first fitted, as described in 4.6, we mentioned that after pumping out the airlock the locking ring usually needs an extra quarter turn or so because the airlock is pulled down by the vacuum inside. In the reverse process it is advisable to loosen the locking ring by the same amount before pressing "OK" when the gate valve has been closed. This is so that the ring doesn't become extra tight and difficult to open when the airlock returns to ambient pressure.

Follow the instructions as prompted and press "OK". The airlock is vented and returns to atmospheric pressure. Loosen the locking ring and twist the airlock so that the steel studs line up with the slots in the airlock cradle and remove the airlock and sample probe.

- **Note:** The sample probe may be very cold as the sample is raised into the airlock.

4.12 Standby mode / Warm up - Exiting the ride safely

4.12.1 Putting the system in standby mode

Whenever the system is not being used it is recommended to put the system into standby mode to reduce the Helium boil off. This is done as follows:

- Switch off the heater
- Switch off the Helium level gauge
  - The level gauge introduces a small amount of heat each time it measures.
- Open the flow control window, figure 4.14, and set He fine flow from the f-func drop down menu.

4.12.2 Warming the system to room temperature

If it is necessary to warm the system to room temperature it should be done carefully to avoid harm to you or the system.

- The magnet ***must*** be at 0T, i.e. zero current flowing.
- Allow liquid Nitrogen and Helium to boil off naturally. This may take several days depending on the levels of liquid.

Once the system is at room temperature it is recommended to flush the Helium reservoir with Nitrogen gas to prevent Helium gas diffusing into the cryostat vacuum space.

5 Regular Maintenance

To keep your system running smoothly there are some routine maintenance tasks. Some depend on which system you have, others are common to all systems.

5.1 All Systems

5.1.1 Clean and grease sample probe

A small amount of vacuum grease needs to be applied to the sample probe where it passes up and down through the airlock. The probe needs to pass smoothly through the airlock to avoid jerky motion and bad scans. You may notice excess grease accumulating at the top of the airlock and this may be dirty. Periodically it is advisable to clean the sample probe with a small amount of ethanol to remove dirt and then reapply a thin layer of vacuum grease.

5.2 Standard System (S700X)

5.2.1 Top up liquid Helium

The helium reservoir should be filled at least once a week. Refilling the cryostat with liquid helium when cold is a relatively quick procedure, provided that a small amount of liquid remains in the dewar. The procedure is much the same as for cooling and filling the reservoir as described in 4.2.6. Re-read this section for full details. The syphon is pre-cooled by slowly lowering the arm into the storage vessel until the liquid level is reached. The storage vessel is then pressurised slightly and the exiting gas from the syphon is monitored until a strong white jet is observed. The safest procedure is then to reduce the pressure in the storage vessel to zero before inserting the syphon into the susceptometer. Experienced users may find, however, it is possible to insert the syphon directly into the system and forgo the de-pressurisation stage. In either case, the syphon should be inserted only about halfway into the main reservoir and not connected to the syphon extension cup. This prevents gas transferred through the syphon bubbling through the existing liquid and reducing the transfer efficiency. With experience the loss of only one litre of liquid helium is possible during a 'top up' procedure.

Important: remember to disconnect the SQUID head before transferring helium to prevent the SQUID electronics from becoming too cold.

5.2.2 Top up liquid Nitrogen

The nitrogen reservoir should be filled at least twice a week. The nitrogen cooled shield reduces helium boil off so keeping the nitrogen topped up regularly keeps the helium usage as low as possible. Filling the nitrogen reservoir is a simple operation. The easiest way is with a nitrogen storage dewar with a flexible hose attached to the outlet tap. Failing this, liquid nitrogen can be poured into the reservoir from a nitrogen bucket through a suitable funnel. Top up the nitrogen until you see liquid spill over the lip of the filling port.

5.3 Recondensing System (S700X-R)

The recondensing cryostat has no nitrogen reservoir and there is no need to top up the liquid helium on a regular basis, making it a very low maintenance system.

5.3.1 Check Helium Level

The S700X-R is a zero loss system when running in steady state. The only losses are the small amounts of gas used to flush the airlock when changing a sample. Depending on how frequently the sample is changed the reservoir will remain sufficiently full for 1-2 years. Once a week you should check the helium level with the digital gauge on the front of the rack. If the level begins to fall significantly then there may be a problem and you should contact Cryogenic ltd for advice on tracking down the source of the loss.

5.3.2 Compressor and Water Chiller

Refer to user manuals for the helium compressor and water chiller for guidance on regular maintenance. It is important to keep these items in good condition to prevent loss of performance of your susceptometer system.

5.4 Cryogen-Free System (S700X-CF)

5.4.1 Compressor and Water Chiller

Refer to user manuals for the helium compressor and water chiller for guidance on regular maintenance. It is important to keep these items in good condition to prevent loss of performance of your susceptometer system.

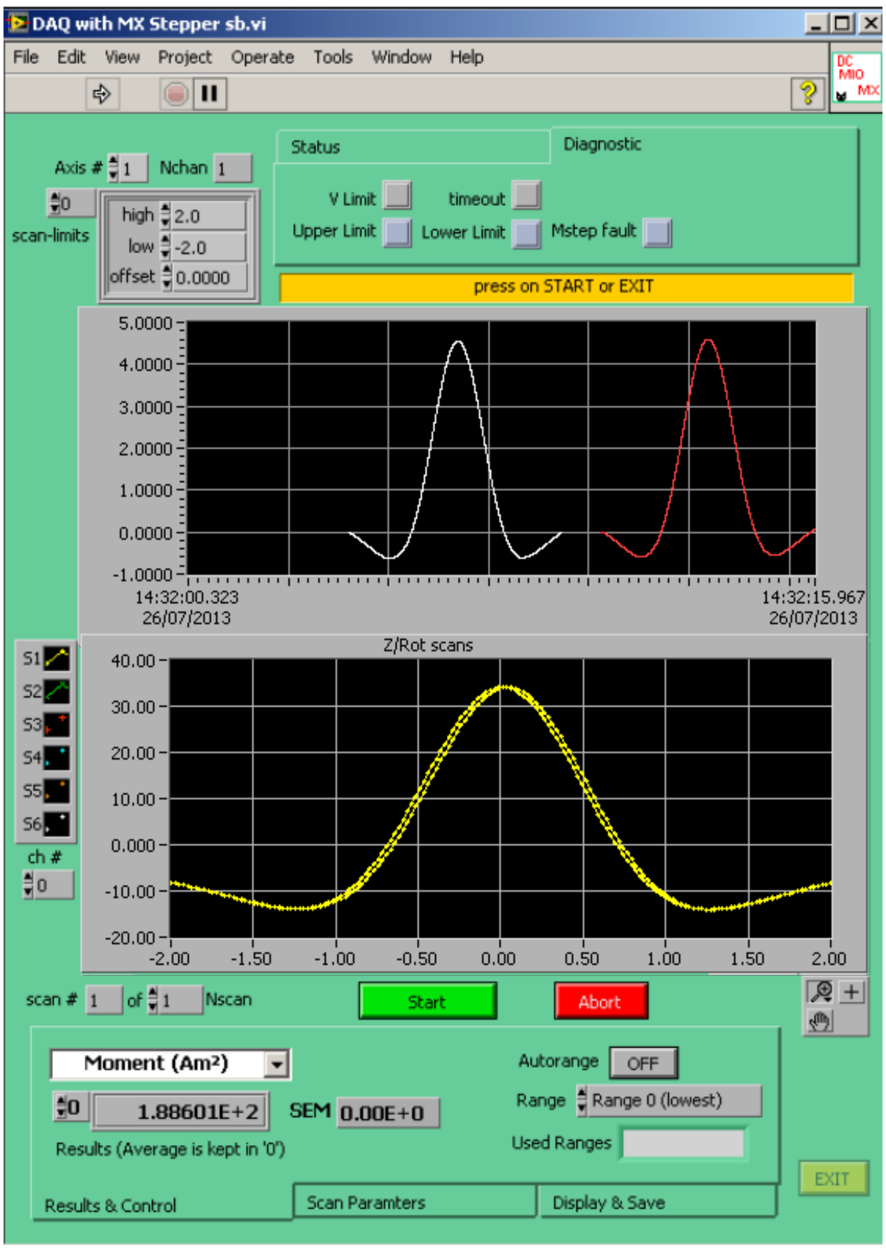
6 Anual Maintenance

Charcoal sorbs in the insert and cryostat vacuum spaces collect any helium that diffuses through the glass fibre over time. Eventually the sorb becomes saturated and further helium diffusion weakens the vacuum and hence the isolation from the outside temperature. It is reccomended once a year to take a maintenance break from experiments and warm up the system to room temperature. This will release helium from the sorbs and the vacuum spaces of the cryostat and insert can be pumped out again before cooling down. This is a good opportunity to make any planned repairs or modifications.

7 DC Measurements

DC measurements were briefly touched on in section 4.7 where we discussed centring a sample. Here we will look in more detail at what is really going on when you make a DC scan and how to utilise the available features to get the most accurate and reproducible results from your experiments. DC (direct current) refers to the current generating the applied magnetic field. The field applied in DC measurements is constant during a scan.

To set up a DC measurement open DC measurement from the **single measurement** panel in the main menu. The DC measurement window appears, entitled **DAQ with MX Stepper sb**. For a description of every control and display in this window see section 13.3.



From here you set the parameters for a longitudinal (Z) or rotational (R) scan, start the scan and see the result. In depth analysis of the results is available with the 

button. First things first, let's look at what actually happens when you make a scan.

After setting the parameters you want (which we will discuss shortly) you make a scan by pressing 

. The sample is moved to its starting position. For a Z-scan this is the bottom of the vertical scan range you have defined, for an R-scan it is usually the centre of the pickup coils[8]. In the following sections we discuss Z and R-scans separately in detail but for now we are talking of general principles common to both DC measurements.

With the sample in position, the parameters are passed to the data acquisition system, telling it what and how to measure. The stepper motor moves the sample through the defined range of motion, vertically up and back down again for a longitudinal scan and rotated about the vertical axis for a rotational one. As the sample moves, the output from the SQUID is measured and displayed in the upper graph as a function of time. There are many points taken, (16000 per cm for longitudinal scans) and a running average is used when displaying the SQUID output.

When the movement is complete, the software takes care of any *drift*, i.e. any time dependence in the background reading from the SQUID. The drift can be measured and subtracted automatically or manually. The resulting data is then scaled according to the selected range and displayed in the lower graph but this time as a function of position. This data is then fitted numerically and a magnetic moment or volume magnetisation is calculated and displayed. Several scans can be taken and the results averaged, but each scan is still stored and available for in depth analysis.

For the following sections we are assuming **channel selection** is set to **auto** mode, so that a *Z-scan* measures with the *axial* pickup coils and an *R-scan* measures with the *transverse* pickup coils. For the vast majority of measurements this will be true, but we shall see in section 7.2.1 that there may be times when you want the freedom to choose the motion and pickup coils separately. With the S700X you have this option and once you understand how DC measurements work you should be able to make use of the advanced features to meet the particular demands of your own experiments.

The significance of drift

The subtraction of drift is probably the most important part of the measurement. In an ordinary induction loop the induced current is proportional to $\frac{\partial\Phi}{\partial t}$ as a sample moves through the coil. However since our pickup system is superconducting, the SQUID output is proportional to ϕ and not $\frac{\partial\Phi}{\partial t}$, so you may ask why we need to move the sample at all. The short answer is **drift**. The background flux has several sources including the remnant field of the magnet and the metal components of the VTI amongst others. The background can be weakly time dependent. It is by measuring and subtracting the drift that the tiny moment from the sample can be distinguished from the background. This is the reason that the sample is moved through the pickup coils. In sections 7.1 and 7.2 we will see how moving

[8]you can define the measurement position for a rotational scan in the **Configure Data Acquisition** window

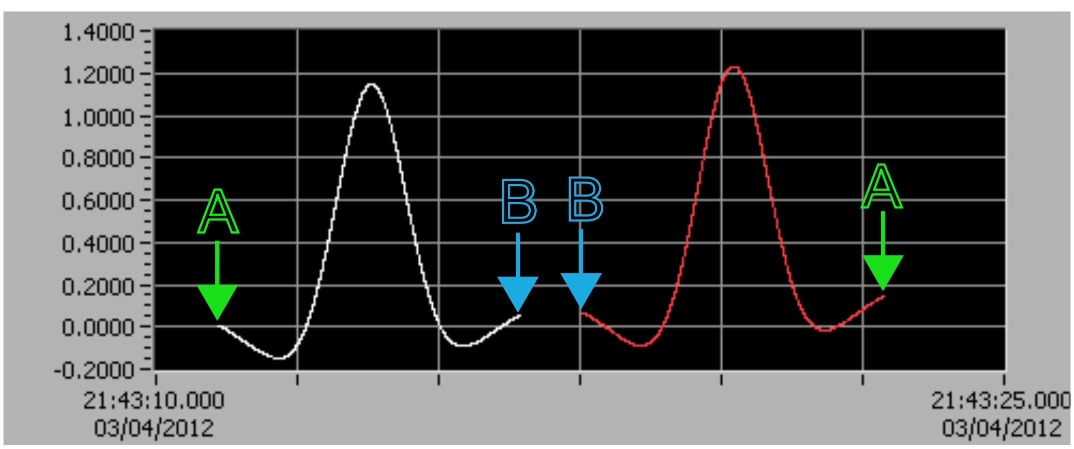
the sample allows the drift to be compensated for longitudinal and rotational scans.

For the following sections we are assuming **channel selection** is set to **auto** mode, so that a *Z-scan* measures with the *axial* pickup coils and an *R-scan* measures with the *transverse* pickup coils. For the vast majority of measurements this will be true, but we shall see in 7.2.1 that there may be times when you want the freedom to choose the motion and pickup coils separately. With the S700X you have this option and once you understand how DC measurements work you should be able to make use of the advanced features to meet the particular demands of your own experiments.

7.1 Longitudinal (Z) scan

The choice of scan type, Z or R, is made by selecting the **Axis**, for a longitudinal scan use **Axis #1**. To make a longitudinal scan the sample is moved vertically through a set of axial pickup coils, wound in a 2nd order gradiometer configuration, concentric with the sample rod. A schematic of the coils is shown in figure 7.1. A number of scans can be performed one after the other and the results averaged. To control the number of scans simply use **Nscan**.

The range of movement is defined in the **scan-limits** box. The limits will nearly always be symmetric about the centre. Let's define the range as $\pm h$. The default range is $\pm 2\,cm$. When you press Start the sample moves to $-h$ (below the centre) ready for the scan. For the scan itself the sample is moved smoothly from $-h$ to $+h$ and the SQUID output is measured at 16,000 points per cm. The output is smoothed by adjacent averaging over 40 points check number and displayed in the upper graph as a function of time.



Since this graph is produced as the sample moves up and then down through the pickup coils, the points marked A are equivalent in position as both represent the bottom of the scan. Similarly points B both represent the top of the scan. The sample starts and ends in the same position, to within less than a micron, so the signal from the sample should be the same at both points (A). Any difference between these points, as shown in the graph above, must be due to background drift.

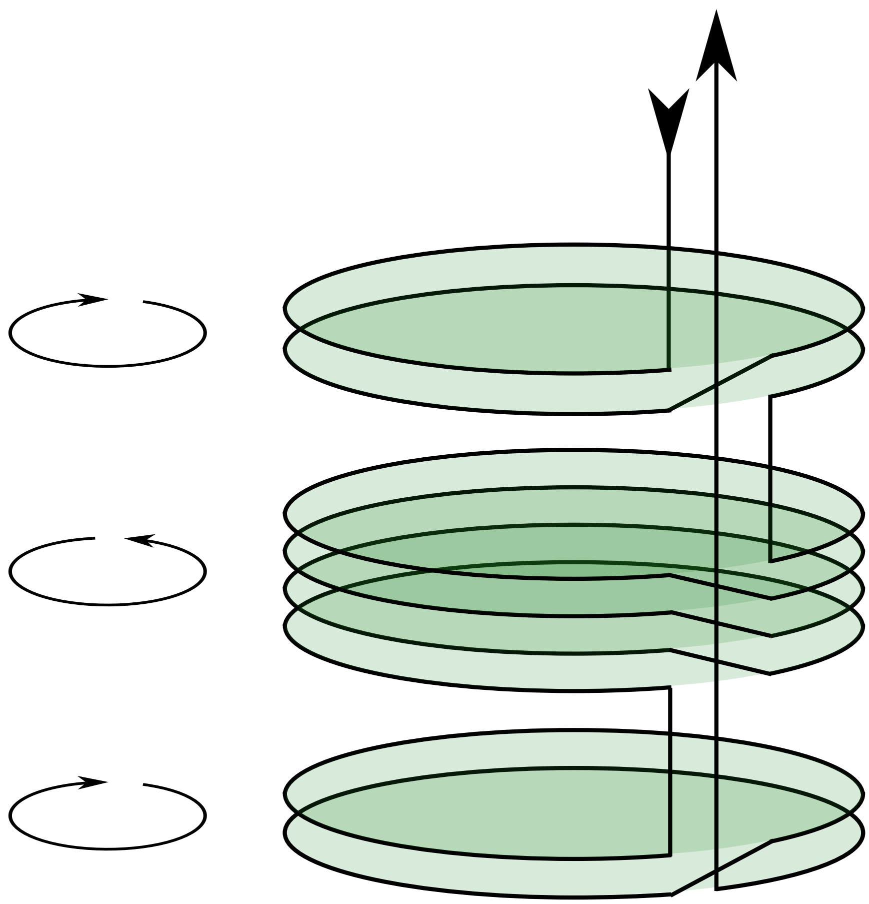


Figure 7.1: *Axial pickup coils*

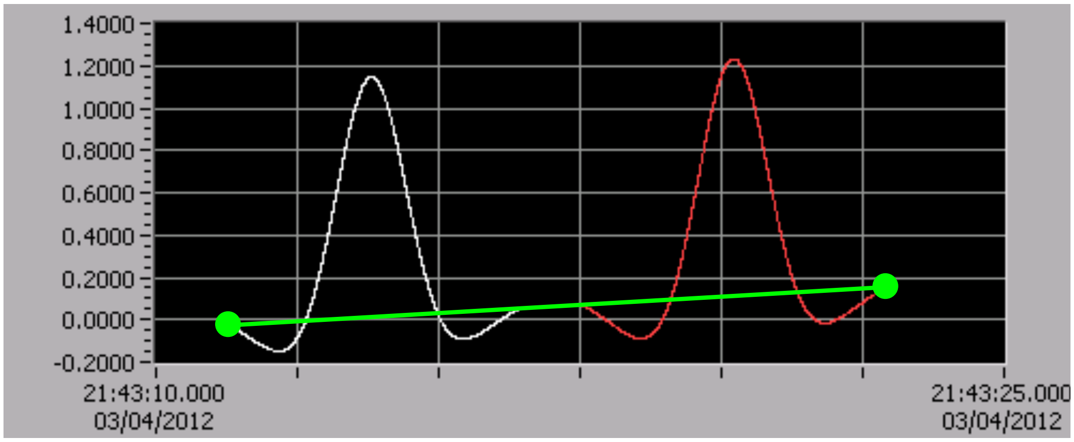


Figure 7.2: *When offset is set to* ***auto*** *the drift is measured by taking averages of the first and last three points and by linear interpolation in between. The resulting straight line function is subtracted.*

Drift compensation can be done automatically, manually or switched off altogether. Figure 7.2 shows how the drift is compensated in automatic mode. Over the few seconds it take to perform the scan the drift is linear. The first three points are averaged to give a reliable start point, the same with the last three for an end point. Then a straight line is drawn between these end points and this line is subtracted from the scan.

The default setting for drift compensation is auto offset. The drop down menu has several other options however and these are:

no offset as the name suggests, no offset is subtracted

manual offset A straight line is subtracted from the voltage-time scan but instead of measuring the drift and subtracting this, the line is defined by the user with the **m** and **dy** boxes. The line is calculated from these parameters such that **m** and **dy** represent the *intercept* and *gradient*, respectively, of a line in the voltage-position graph.

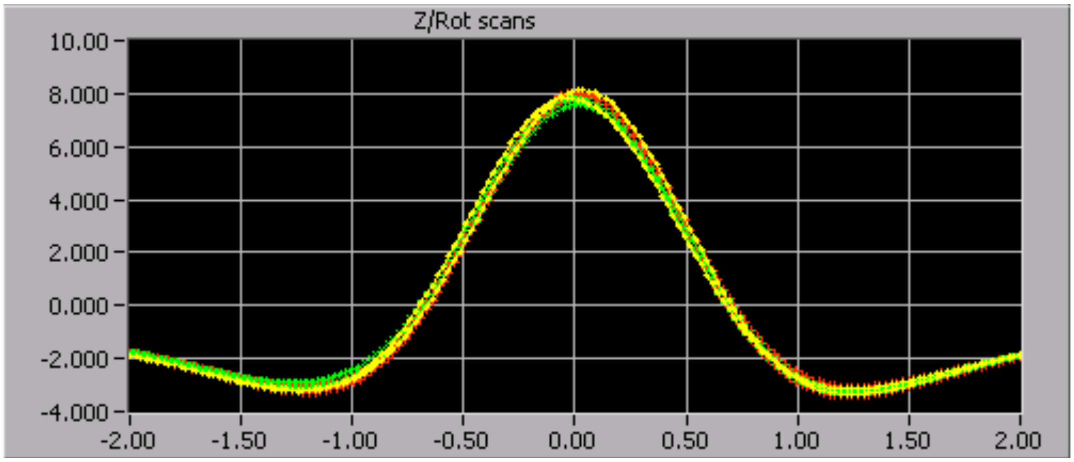
- **m** the intercept of a line in the voltage-position plane with the line $z = 0$, i.e. the centre position
- **dy** the gradient of the same line

auto offset The drift is measured as in figure 7.2 and a straight line calculated between the end points as shown. This line is then subtracted from the voltage-time scan.

Hall probe No drift compensation is subtracted but the voltage is scaled with the Hall probe calibration constant (see 13.1). If the Hall probe is inserted instead of a sample, setting **offset** to **Hall probe** means the resulting scan represents the field profile measured in Tesla.

Flux gate The same as for **Hall probe** but the **Flux gate** calibration is used instead.

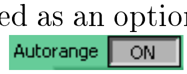
The compensated scan results are then scaled either by the hall probe or flux gate calibrations as described above, or by the appropriate settings for the selected **range**. The resulting data is then displayed in the lower graph as a function of *position.* For a Z scan this has the effect of 'folding' the red *down* scan back on top of the white *up* scan.



If several scans are performed, i.e. $Nscan > 1$, then the results of each, both up and down, are displayed here. In the example shown, three scans were performed, resulting in six curves being displayed.

Range

The range controls the sensitivity of the SQUID. Range 0 is the most sensitive but the output will easily saturate with a large sample moment. A saturated output gives a scan with a flat top making it impossible to fit accurately as a dipole. There are three ranges, 0,1 and 2, associated with gain settings on the SQUID amplifier. For a Z-scan, there are two extra ranges, 3 and 4, that come from an attenuator which decreases the sensitivity of the SQUID. The range extender is fitted as an option to the axial pickup system but not to the transverse.

The autorange button 

toggles the autorange feature on and off. With autorange ON, if at any position the voltage reaches the upper or lower limits defined for each range the scan is rejected and repeated with the next highest or lowest range.

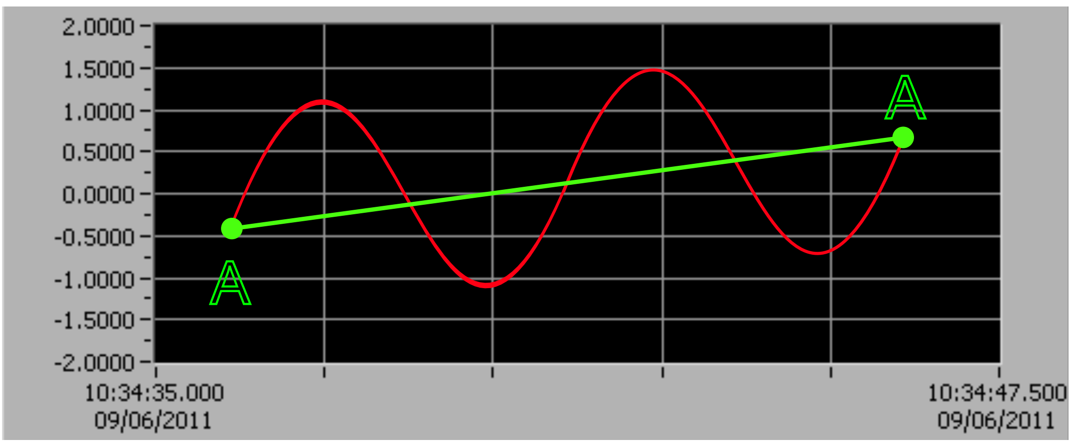
7.2 Rotational (R) scan

For rotational measurements we need to swap the head of the sample transport system for the rotating head. This can be done either before inserting the probe or whilst the probe is in the cryostat.

To make an R scan use **Axis #2**. In this mode the sample is positioned by default at the centre of the pickup coils. The axial and transverse pickup coils are located with their centres at the same point so a Z-scan can be used to centre a sample and the same position is still central for an R-scan. The transverse pickup coils are wound as shown in figure 7.3. With the sample at the centre it is rotated about the vertical axis. When axis #2 is selected the **scan-limits** box changes so that the limits are defined by a *start angle* and a *stop angle*. This should be an integer multiple of 360° for a sensible scan.

The loops of this coil set are less obvious than the nice, round loops of the axial pickup coils. In the transverse pickup coils the loops are effectively rectangular when viewed from the side. The area defined by each loop is shown in green in figure 7.3. As with the axial pickup coils the configuration makes a 2nd order gradiometer. The centre coil is twice as large in area as the end coils and wound in the opposite direction.

As the sample rotates, any transverse moment will swing around like a lighthouse beacon and sweep through the transverse coils. The resulting scan is sinusoidal.



Drift is compensated as for a Z-scan. If the sample is rotated through an integer number of complete turns then the points marked A in this case both represent the same sample orientation. The signal from the sample should therefore be the same at both points. Any difference is due to background drift during the measurement. An average of the first three points is taken to give a reliable start point, similarly the last three are averaged for an end point. A straight line is drawn in between and this is subtracted from the data. The options for drift compensation, **auto**, **manual**, **no offset**, **hall probe/flux gate**, are as described in 7.1.

The scans are then scaled as for a Z-scan, either by the hall probe or flux gate calibration or according to the set **range**. The ranges are as described for Z-scan, 7.1.

7.2.1 Centring

Centring a sample for an R-scan can often be done exactly as described in 4.7, using a Z-scan, because the transverse and axial pickup coils share the same centre position and because most samples will have a magnetic moment not exactly axial or longitudinal but somewhere in between so components of the moment can be seen in both measurements. If however you have a sample that shows no measurable longitudinal moment you will have to find another way to centre it. The best way to do this is to use manual channel selection and perform a longitudinal scan but measure with the transverse rather than axial coils.

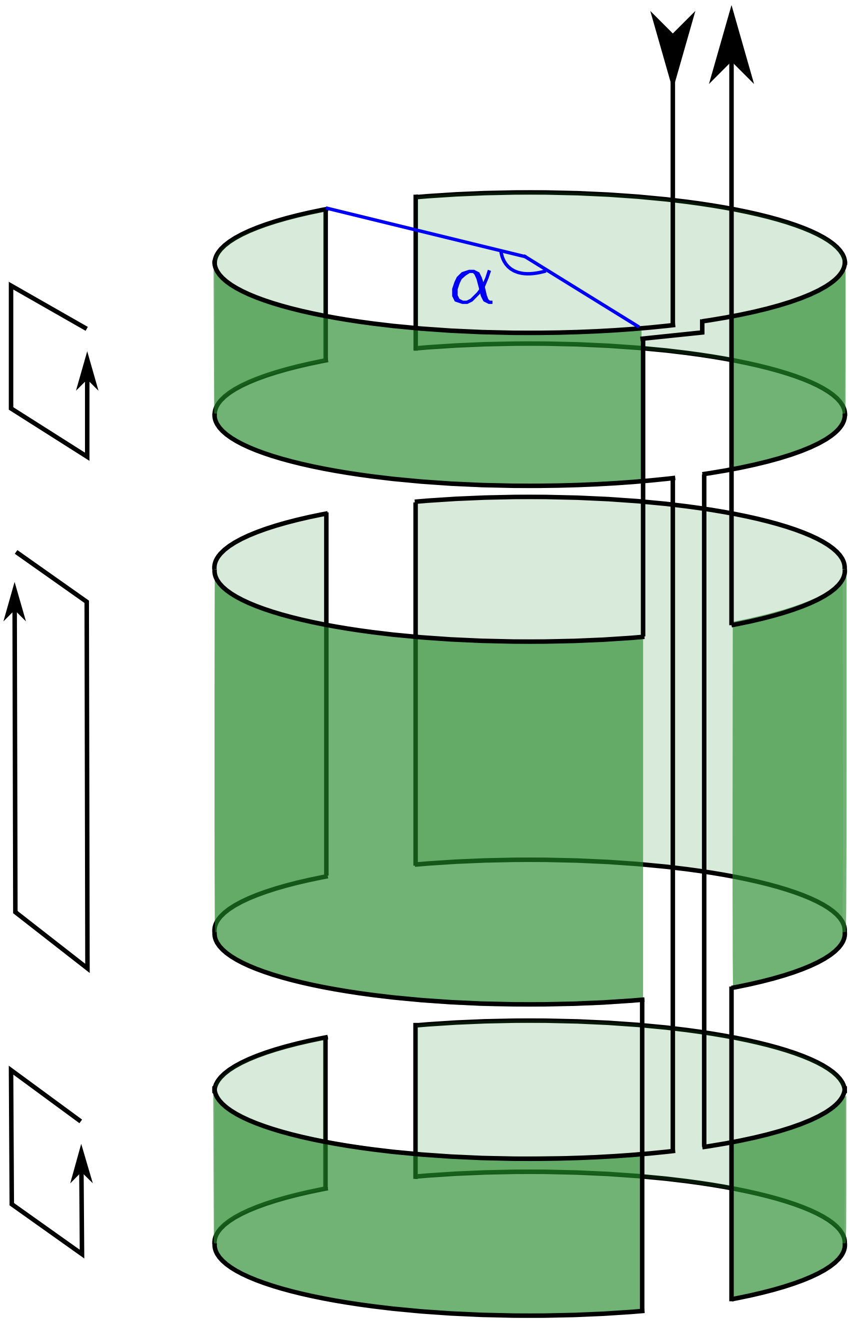


Figure 7.3: *Schematic depiction of the transverse pickup coils. The opening angle* α *has been exaggerated for clarity. In the real system* $\alpha = 90°$.

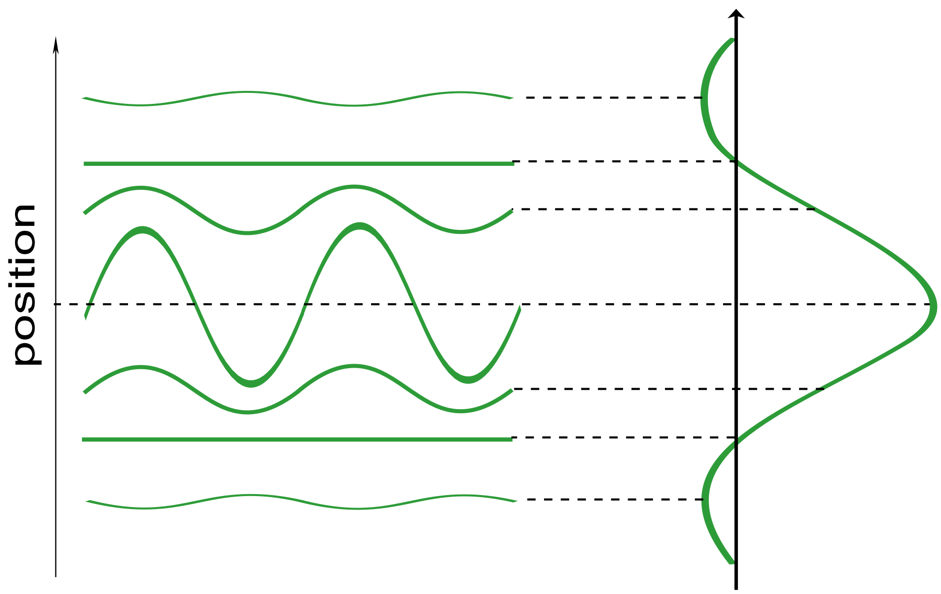


Figure 7.4: *Centring a sample for rotation measurements. If a sample has no axial moment then centring can be done with a series of rotation scans to find the largest amplitude as on the left. This is very tedious and centring is better achieved by using manual channel selection to measure the transverse moment (transverse pickup coils) whilst moving the sample vertically.*

centring a sample with a very weak or zero axial moment

Suppose we want to perform a rotation measurement to measure the transverse moment of a sample. Usually we can centre the sample with a normal Zscan because the longitudinal moment is strong enough, and the centres of the longitudinal and transverse pickup coil sets coincide. What happens then when there is no discernible longitudinal moment? We could perform a series of rotational measurements, moving the sample a small amount each time, and look for the largest amplitude to the resulting sinusoidal scan, as shown in the left hand side of figure b7.4. This is a long and frustrating method. Alternatively we could perform a Zscan but measure in the transverse pickup coils, as shown in the right hand side of figure 7.4.

- open **Configure Data Acquisition** by clicking [DAQ configure] on the main menu.
- Change **input channel selection** mode to [manual]
- click [DA change ch & ranges] to open the **DA set 8 x Ch & limits** window.
- Deselect **Z-scan** and select **R-scan** to measure with the transverse pickup coils, press [OK] to confirm.
- Click [EXIT] to confirm changes to **Configure Data Acquisition**.

- Choose DC measurement from the **single measurement** tab of the main menu
- Use **Axis #1** and **ch #1** to perform a Z-scan but measure with the transverse pickup coils and go through the centring process as normal, section 4.7.

7.3 Results

The compensated and scaled scan is used to calculate the magnetic moment of the sample. The way in which this is done is covered in chapter 8, which discusses the in depth analysis options available via the zoom button, and also in ***advanced data analysis*** (appendix **??**). The zoom function allows each individual scan to be analysed carefully to get the most reliable results.
The result of the scan or scans is displayed in this box.

The index counter to the left determines which result to display. **0** is the arithmetic mean of the results of each individual scan. **1,2,3...** are the individual results of each scan.

SEM Standard Error Mean, defined as $SEM = \frac{\sigma}{\sqrt{n}}$ where σ is the standard deviation of the results.

There are several options for the units to use when displaying the result. These are chosen by clicking on the result label **Moment (Am²)**.

Moment (Am²) Displays the calculated magnetic moment in Am^2

Moment (Am²/kg) Displays the calculated specific magnetic moment in Am^2/kg. The sample mass must be entered in the **Configure Data Acquisition** window (13.1).

Magnetisation (A/m) Displays the calculated magnetisation in A/m. The sample volume must be entered in the **Configure Data Acquisition** window (13.1).

- $10^{-3}\,Am^2 = 1\,emu$
- $1\,Am^2/kg = 1\,emu/g$

7.4 Saving Data

There are two ways to save data when performing single DC scans.

save scan saves a tab delimited text file containing the data in the lower graph, voltage (with the applied scale) against position. A standard dialogue box appears for you to choose a file name and a location.

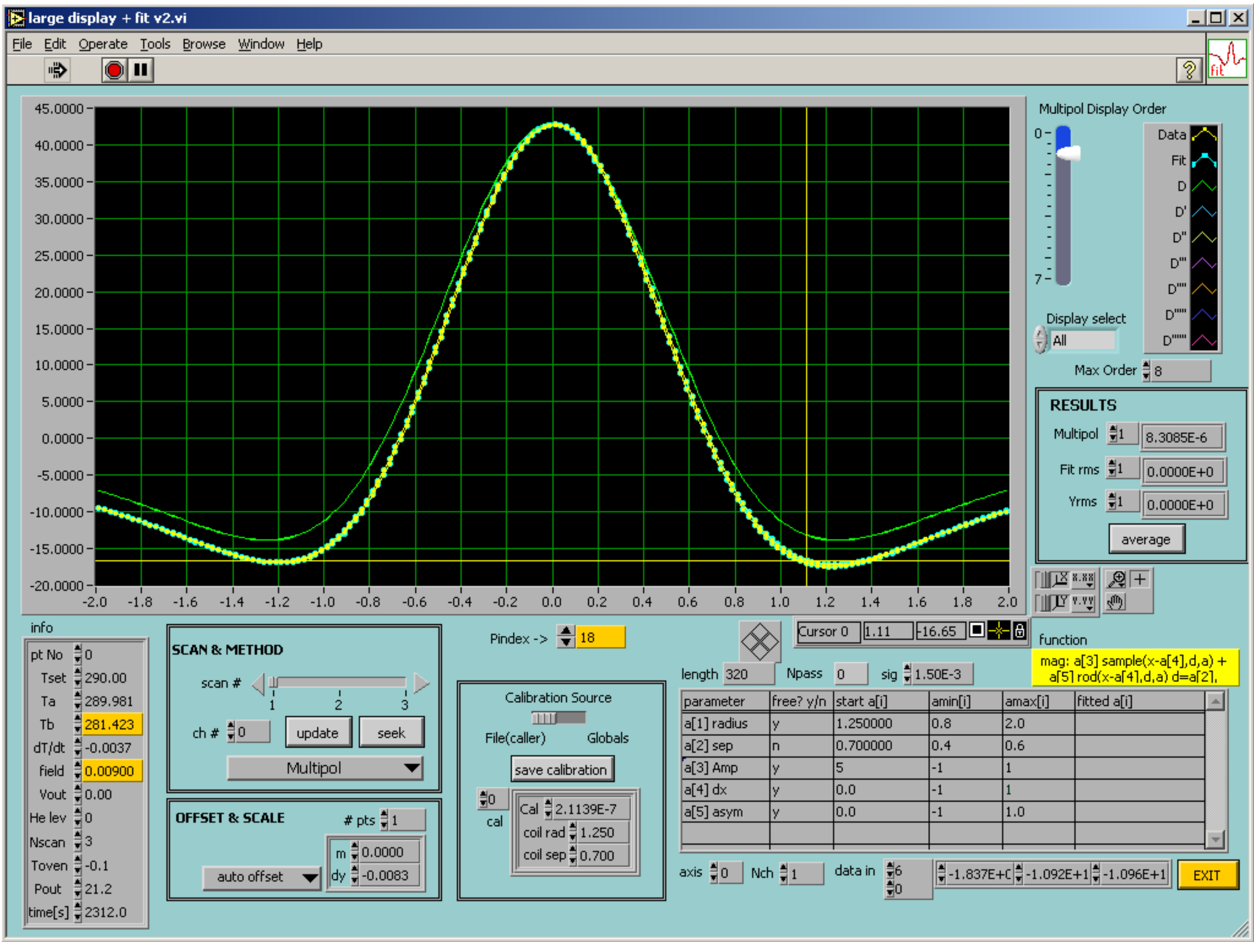
When set to ON, save data writes the data to a file in the same way as in an automatic sequence. This can be useful for manually taking data at a few B,T points without writing a sequence, or storing scans for diagnostic purposes. A standard dialogue box appears where you can choose a file name and location.

7.5 Manual channel selection

Once you understand how the measurement works, what is moving and what is measuring, and if the transverse pickup system is installed, it is possible to perform more involved DC measurements to meet more specific needs. Most users will only be interested in a small number of the possible measurement configurations. Centring a sample with no axial moment, as in section 7.2.1 serves as an instructive example to show the sort of thing that can be done with a little thought, as well as how to access the channel selection window. Several channels may be recorded at once. You can always contact Cryogenic Ltd for advice on getting the most out of your system to meet your particular requirements.

8 Zoom - Data analysis

The zoom function is accessible through either the single **DC measurement** or the **view data file** windows.



It is an environment for analysing the individual scans taken at each point of a sequence stored in a data file or the scans taken during a single DC measurement. You can look at individual scans and see how sensitive the results are to changes in certain parameters. This can tell you a lot about the quality and reproducibility of your results. Zoom is a very useful diagnostic tool and with experience you will be able to recognise problems such as a badly mounted sample simply by looking closely at the scans with this facility.

8.1 Controls and Display

One scan at a time is displayed in the large graph. If you are looking at the B,T points in a data file then you can scroll through point by point using the **Pindex** control. The individual scans taken at the selected point can be seen separately by using the **scan #** scroll bar in the SCAN & METHOD box. If you have the **view data file** window open at the same time then you will see that as you scroll through the points in the zoom window, the view data file window follows your selection. The reverse is not true however. If the zoom window is open then the view data file window will not allow you to change point until you have closed the zoom facility. If you are looking at the scans from a single measurement then there is only one B,T point to view.

The **info** box in the bottom left corner of the window displays information about the currently selected point, including the time, temperature (set and measured), field and number of scans taken. See chapter 13 for details of all the information in this box. On the right hand side is the **RESULTS** box. This displays the result in Am^2 of each of three different methods of calculating the magnetic moment from the scan currently being displayed. Next to each result is an index counter that controls the scan number of the result to display[9]. As for the result in the single DC measurement window, **0** is the average of all the scans taken at the current point, **1,2,3...** show the results of the individual scans. When changes are made to any settings that affect the results of the scans for a particular method, the average is not automatically recalculated. To recalculate the average press average.

The result that is saved in the data and result files is always the multi-pole method, calculated by *singular value decomposition* with a dipole signal and its derivatives up to n^{th} order, set with the **Order** control in the single DC measurement window. The multi-pole method greatly surpasses the others in terms of accuracy and reliability, as explained below in 8.5.

In case you wish to look at data that was taken on a different S600/S700 system or that was taken before a change was made to any calibration settings, you have the option to select the source of the calibration settings used in the analysis. In the **Calibration Source** box you can switch between two options

Globals Uses the current system calibration settings

File(caller) Uses the settings stored in the data file being examined. These are the settings that were used when the data was taken.

Finally, the **OFFSET & SCALE** box allows you to change the drift compensation method as described in chapter 7. None of these changes will affect the stored data. The tools in this window are for diagnostic purposes. By experimenting with settings and calculation methods you can test the sensitivity of results to various factors and usually determine the cause of any irregularities.

8.2 Yrms

This simply takes the **root mean square** value of every voltage value in the scan

$$RMS = \sqrt{\frac{\sum y_i}{N}} \tag{8.1}$$

where N is the number of values, and applies the appropriate scaling factor to give an estimated magnetic moment. This is a very rough estimate. The value in it is that for a

[9]When the displayed scan is changed with **scan #**, the result is automatically updated. However, when the result index is changed it does not affect which scan is displayed.

good scan it should at least approximate the real moment. If the result of the multi-pole calculation differs greatly from this value then something may be wrong either with the scan settings or with the sample mounting.

8.3 Fit rms

This is a standard *least squares* fitting process using the **Levenberg-Marquardt** algorithm. It is worth repeating that the result of this fit is *not* stored as the actual result. As will be explained shortly this technique is susceptible to error from sample geometry effects. It is useful however for investigating the sensitivity of the scan result to a number of parameters. The model used for the fit is shown in the yellow **function** box. This box is a display only, the model can not be altered except by altering the parameters. The model is made up of two contributions, from the sample and from the end of the sample rod. The effect the end of the rod can have on a measurement was discussed in section 4.5.

For a Z-scan the model is:

$$signal(x) = A \times sample(x - dx) + B \times rod(x - dx) \tag{8.2}$$

$$sample\,(y) \quad = \quad f(y - d) - 2f(y) + f(y + d) \tag{8.3}$$

$$f(z) = \frac{\mu_0 I a^2}{2(a^2 + z^2)^{3/2}} \tag{8.4}$$

$$rod(y) = g(y - d) - 2g(y) + g(y + d) \tag{8.5}$$

$$g(z) = \int\limits_{u=0}^{\infty} f(z - u)du = \frac{-\mu_0 I a^2 z}{2(a^2 + z^2)^{3/2}} \tag{8.6}$$

symbol	parameter	meaning
a	**a[1]**	radius of pickup coils
d	**a[2]**	separation of upper and lower pickup coils from centre
A	**a[3]**	Amplitude of sample signal
dx	**a[4]**	sample position offset from centre
B	**a[5]**	measure of the effect of the end of the sample rod

The parameters for fitting in the model are shown and can be edited in the table below the yellow function box.

parameter	free? y/n	start a[i]	amin[i]	amax[i]	fitted a[i]
a[1] radius	y	1.250000	0.8	2.0	
a[2] sep	n	0.700000	0.4	0.6	
a[3] Amp	y	5	-1	1	
a[4] dx	y	0.0	-1	1	
a[5] asym	y	0.0	-1	1.0	

For an R-scan the model is:

$$signal(\theta) = A\, sin(f\,\theta + \phi) \tag{8.7}$$

symbol	parameter	meaning
A	a[1]	Amplitude
f	a[2]	Frequency
ϕ	a[3]	Phase

Each parameter can be assigned as free or fixed by typing a y or and n in the second column. Free parameters will be varied during the fit whilst fixed parameters will not. Each parameter must also be given starting values as well as a range for the algorithm to explore. These are entered in the **start a[i]**, **amin[i]** and **amax[i]** columns. The result of the fit for each parameter is given in the **fitted a[i]** column.

8.4 Multi-pole analysis

Essentially we are trying to solve an overdetermined system of equations, with more data points than parameters. For every point on the scan we have an equation:

$$y_i = \sum_{j=1}^{N} a_j f_j(x_i) \tag{8.8}$$

where x_i is the position, y_i is the signal and the f_j's are a set of basis functions each with a contribution coefficient a_j. The combination of all points gives us a matrix equation to solve of the form

$$\mathbf{A} \cdot \mathbf{x} = \mathbf{b} \tag{8.9}$$

where we want to find $\mathbf{x}$ so as to minimise[10]

$$\mathbf{r} \equiv |\mathbf{A} \cdot \mathbf{x} - \mathbf{b}| \tag{8.10}$$

[10] If we could write down every basis function then the solution would be exact and we would have $\mathbf{r} = \mathbf{0}$. However, we are always working with a model, and with models come assumptions and approximations and there will always be discrepancy, however small, between models and observations.

In the conventional non-linear curve fitting method we have only one basis function, given by equation 8.2 for a Z-scan and equation 8.7 for an R-scan. In multi-pole analysis we use not only these functions but also derivatives up to the order set by the user.

For a Z-scan the maximum order is 8.

$Z(z)$	$\left(a^2+z^2\right)^{-\frac{3}{2}}$
$Z'(z)$	$-z\left(a^2+z^2\right)^{-\frac{5}{2}}$
$Z''(z)$	$\left(4z^2-a^2\right)\left(a^2+z^2\right)^{-\frac{7}{2}}$
$Z'''(z)$	$\left(3a^2-4z^3\right)\left(a^2+z^2\right)^{-\frac{9}{2}}$
$Z''''(z)$	$\left(a^4+12a^2z^2+8z^4\right)\left(a^2+z^2\right)^{-\frac{11}{2}}$
$Z^{v}(z)$	$\left(24a^4z^2-11a^4z+76a^2z^3-56z^5\right)\left(a^2+z^2\right)^{-\frac{13}{2}}$
$Z^{vi}(z)$	$\left(5a^6-120a^4z^2+240a^2z^4-64z^6\right)\left(a^2+z^2\right)^{-\frac{15}{2}}$
$Z^{vii}(z)$	$\left(-195a^6z+2760a^4z^3-120a^4z^4-2944a^2z^5-1344z^7\right)\left(a^2+z^2\right)^{-\frac{17}{2}}$
$Z^{viii}(z)$	$\left(-195a^8+4770a^6z^2-480a^6z^3-53360a^4z^4-24128a^2z^6+1560a^4z^5+50048a^2z^6+13440z^8\right)\times\left(a^2+z^2\right)^{-\frac{19}{2}}$

For an R-scan the maximum order is 4.

$Z(\theta)$	$A\,sin(b\,\theta+\phi)$
$Z'(\theta)$	$A\,b\,cos(b\,\theta+\phi)$
$Z''(\theta)$	$-A\,b^2\,sin(b\,\theta+\phi)$
$Z'''(\theta)$	$-A\,b^3\,cos(b\,\theta+\phi)$
$Z''''(\theta)$	$A\,b^4\,sin(b\,\theta+\phi)$

If we were to use an algorithm such as Levenberg-Marquardt to fit using a combination of all of these basis functions we would be confronted with a number of difficulties. Firstly, we would have to provide reasonable starting values for the coefficients a_j in equation 8.8 for all of these functions for every scan. These coefficients could potentially vary greatly for different samples, or even for the same sample as the temperature or magnetic field varies. Secondly with so many parameters to fit over we are searching a very complex vector space for a global minimum solution, greatly increasing the chance of our algorithm finding only local minima and not the real solution. Thirdly, since we have several basis functions there may exist more than one combination of functions that approximate the real solution equally well. In this case our fitted parameters would typically blow up to very large magnitude but in a way so as to almost cancel out, but the balance is very unstable. Finally, the more parameters we add the longer each fit takes. To use up to 8^{th} order would involve a lot of computing power.

So if we want to include higher orders in our solution there is no robust, stable and efficient way to find a good fit using standard non-linear curve fitting algorithms. The answer to these problems is **singular value decomposition**, or SVD.

8.4.1 Singular Value Decomposition

An excellent description and proof of this technique can be found in chapters 2 and 15 of *Numerical Recipes***. The power of SVD is that rather than search for the best solution by trial and error methods[11], the matrix equation 8.9 is "solved" directly to find $\mathbf{x}$ that minimises equation 8.10.

The key thing in SVD is that an $M \times N$ matrix $\mathbf{A}$ can be expressed as

$$\mathbf{A} = \mathbf{U}\,\mathbf{W}\,\mathbf{V}^{\mathbf{T}} \tag{8.11}$$

where $\mathbf{U}$ and $\mathbf{V}$ are **unitary** matrices, i.e. $\mathbf{U}^{\mathbf{T}}\mathbf{U} = \mathbf{V}^{\mathbf{T}}\mathbf{V} = \mathbf{I}$ (Identity matrix). $\mathbf{U}$ is $M \times N$, $\mathbf{V}$ is $N \times N$ and $\mathbf{W}$ is an $N \times N$ diagonal matrix with positive or zero diagonal elements (w_j) called the **singular values** of $\mathbf{A}$. The vector $\mathbf{x}$ that will minimise $\mathbf{r} \equiv |\mathbf{A} \cdot \mathbf{x} - \mathbf{b}|$ can be found thus:

$$\mathbf{x} = \mathbf{V} \cdot [\mathrm{diag}(1/w_j)] \cdot \left(\mathbf{U}^{\mathbf{T}} \cdot \mathbf{b}\right) \tag{8.12}$$

where $\mathrm{diag}(1/w_j)$ is a diagonal matrix whose diagonal elements are either $1/w_j$ or zero if $w_j = 0$.

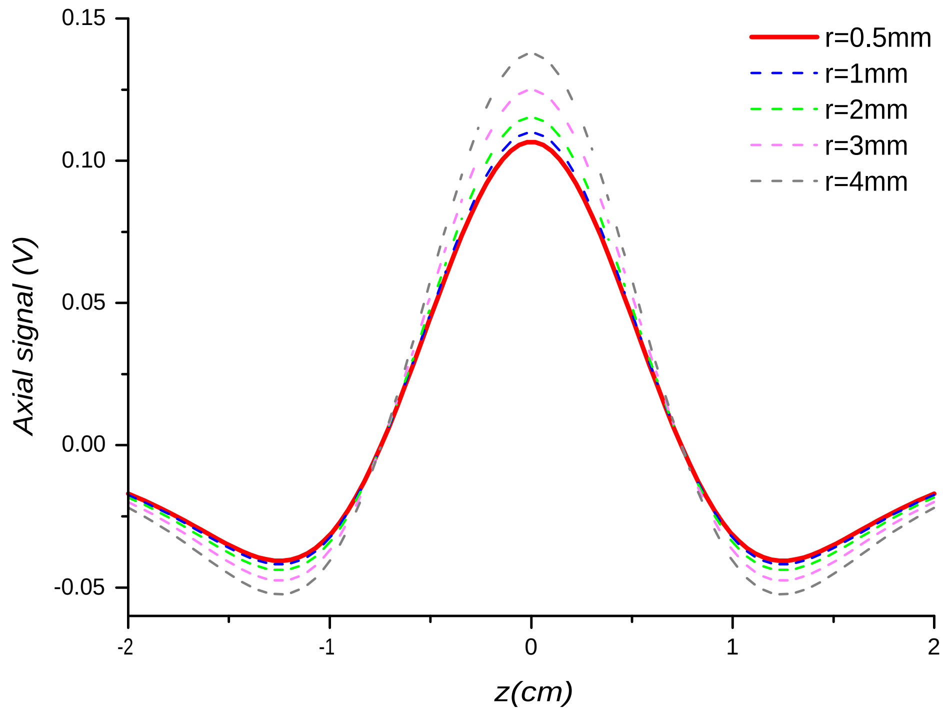
8.5 What do you gain from higher order derivatives?

There are certain effects coming from the geometry of the sample (independent of demagnetisation factors) that affect the shape of the scan in such a way that the moment calculated using only equation 8.2 will be dependent on the sample shape, even though the true moment remains the same. These effects occur via the higher order derivatives of the dipole function. By only fitting with the dipole signal model we can not distinguish the changes in the higher order moments which are falsely attributed to changes in the dipole signal. This is clearly seen in two examples from reference **

[11]sophisticated methods, but trial and error nonetheless.

8.5.1 Example 1: Axial signal of conductive loops

In this example the longitudinal signal from conductive loops was numerically simulated for loops of different radius. The magnetic moment was then calculated using the Levenberg-Marquardt algorithm (Z_{LM}) and and multi-pole analysis.



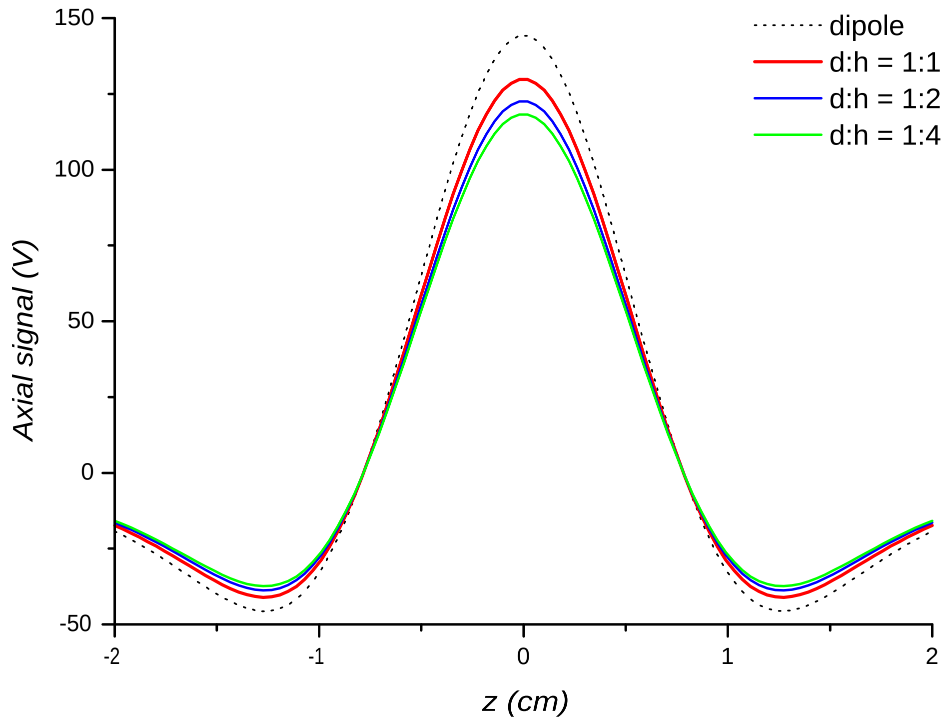
$r\,(mm)$	$Z\,(Am^2/kg)$	Z''	Z''''	$Z''/Z''_{r=0.5}$	Z_{LM}/Z
0.5	-7.8539586×10^{-7}	1.622×10^{-9}	-2.976×10^{-12}	1.0	1.002
1.0	-7.8539585×10^{-7}	6.489×10^{-9}	-3.877×10^{-11}	4.0007	1.007
2.0	-7.8539592×10^{-7}	2.595×10^{-8}	-6.234×10^{-10}	16.0025	1.027
3.0	-7.8539592×10^{-7}	5.840×10^{-8}	-3.154×10^{-9}	36.0059	1.062
4.0	-7.8539767×10^{-7}	1.038×10^{-7}	-9.979×10^{-9}	64.0069	1.113

Figure 8.1: *Magnetic dipole moment (Z) per unit mass and its second and fourth derivatives for conductive loops of different radii. It is the quadrupole (Z'') and octupole (Z'''') moments that grow with radius whilst the dipole moment (Z) remains the same. With singular variable decomposition these contributions can be distinguished. With least squares fitting such as Levenberg-Marquardt, the higher order contributions are not separable from the total signal and hence there is a geometry dependent error.*

The results are shown in figure 8.1. The table shows the dipole moment Z for each loop, along with the quadrupole (Z'') and octupole (Z'''') moments from the second and fourth derivatives of the dipole function. All moments in the table are per unit mass. The second column from the right shows how Z'' grows with the radius of the loop. At $r = 4.0\,mm$, Z'' is 64 times larger than for $r = 0.5\,mm$. For the octupole moment the change is even more significant, $Z''''_{r=4.0}/Z''''_{r=0.5} \approx 3.3 \times 10^3$. Fitting a dipole moment with a least squares method such as Levenberg-Marquardt can not distinguish the higher order contributions to the signal, leading to over-estimation of the moment. The final column in the table shows the scale of this error for each radius. Z_{fit} is the moment fitted by Levenberg-Marquardt. For the smallest loop the error is quite small, 0.2%, but the error rises quite sharply with increasing radius. At $r = 2.0\,mm$ it is almost 3% and at $r = 4.0\,mm$ the error has risen to over 11%.

8.5.2 Example 2: Stack of loops

In this example the signal from stacks of conducting loops was simulated numerically for different aspect ratios *diameter/height*.



$d:h$	Z	Z''	Z''''	$Z''/Z''_{1:1}$	Z_{LM}/Z
2:1	-0.9999966×10^{-3}	8.7246×10^{-5}	-1.0614×10^{-6}	-1.833	1.071
2:$\sqrt{3}$	-0.9999969×10^{-3}	-8.9027×10^{-7}	1.5846×10^{-6}	0.018	0.999
1:1	-0.9999886×10^{-3}	-4.7597×10^{-5}	1.8028×10^{-5}	1.0	0.989
1:2	-0.9999984×10^{-3}	-1.4679×10^{-4}	-8.9870×10^{-6}	3.084	0.886
1:4	-0.9999955×10^{-3}	-1.7156×10^{-4}	-1.7929×10^{-5}	3.604	0.867

Figure 8.2: *Magnetic dipole moment (Z) , quadrupole (Z'') and octupole (Z'''') moments from the second and fourth derivatives, solved with* singular variable decomposition. *The dipole moment per unit mass remains constant to four decimal places, but the higher order moments vary with the aspect ratio. The quadrupole moment has a minimum at $d : h = 2 : \sqrt{3}$ where it is two orders of magnitude smaller than for other ratios. At this ratio Z_{fit} is very close to the real dipole moment. For other ratios Z'' grows which least squares fitting can not distinguish, leading to a geometry dependent error.*

Just like example 1 the dipole moment per unit mass stays constant but the higher order moments vary. For the particular aspect ratio $d : h = 2 : \sqrt{3}$, the quadrupole moment is two orders of magnitude smaller than for other ratios. With this reduction in the higher moments, the moment fitted using Levenberg-Marquardt is a close match to the real dipole moment, $Z_{fit}/Z = 0.999$. For other aspect ratios however, the fitted moment is subject to error because the fitting process can not distinguish between the dipole and the higher order moments. For $d : h = 1 : 4$ the error is over 13%.

9 AC Measurements

This option allows you to measure the AC susceptibility of a sample by measuring its response to an applied AC field. The AC coils are made up of inner and outer windings and a "balance coil". The AC and DC coils together make up the entire magnet coil set as shown schematically in figure 9.1.

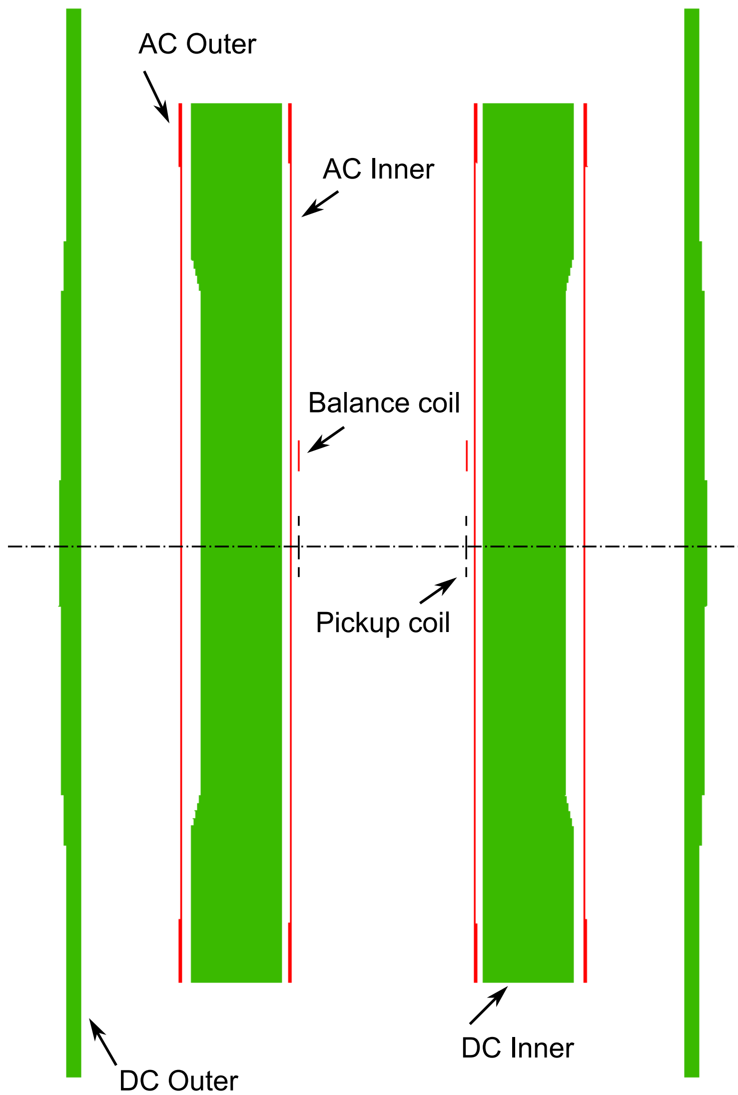


Figure 9.1: *Schematic cross section of AC (red) and DC (green) coils. The dot-dash line marks the vertical centre position where the centre of the pickup coils is located.*

The inner and outer AC coils are energised with opposite polarity to reduce the mutual inductance between AC and DC coils. This prevents the main DC coil from compensating any change in flux due to an AC excitation and allows an AC measurement to be made in the presence of an applied DC field with the DC coil in persistent mode.

To perform an AC susceptibility measurement the sample is positioned in the centre of the pickup coils, an AC current is applied to the excitation coils and the signal is detected by the SQUID via the pickup coils. In order to distinguish between the sample signal and the flux from the AC coils the following procedure is followed:

1. Move sample away from the detection region.

2. Use the balance coil to compensate the AC coil and bring the SQUID output to zero.

3. Move the sample back to the centre and measure.

4. Any signal now observed must come from the sample.

This process is depicted in figure 9.2.

The pickup coils are wound in a second order gradiometer configuration. This means that a completely uniform magnetic field, no matter how strong, will register zero output signal from the SQUID because the windings in the pickup coil exactly balance each other out. In reality we do not have a 100% uniform field. The homogeneity of the applied field is very good, yet the SQUID is so sensitive that it can be saturated even by a small AC field if the pickup system is not well balanced. To accurately measure the signal coming only from the sample we must eliminate the signal from the excitation coils.

The balance coil lets us compensate the field from the AC excitation coils so that the SQUID output is zero with no sample present. It is helpful to look at the field profiles for the coils, shown (not to scale) in figure 9.3. By controlling the current in the balance coil we can flatten out the total field profile seen by the pickup coils and thereby reduce the SQUID output to zero.

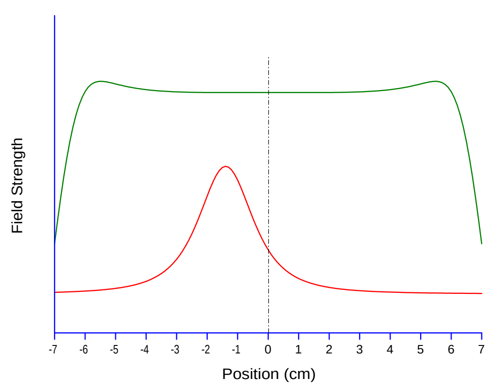
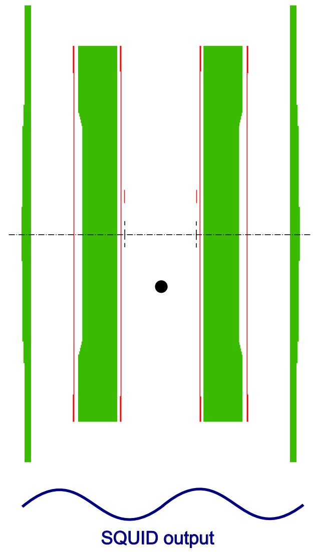
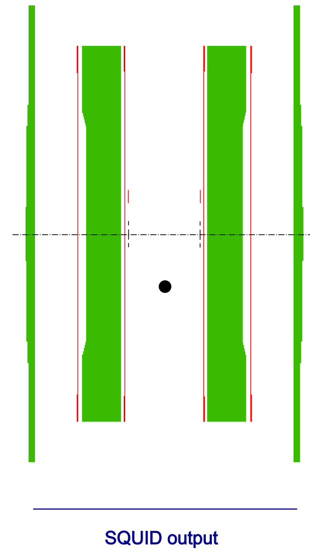


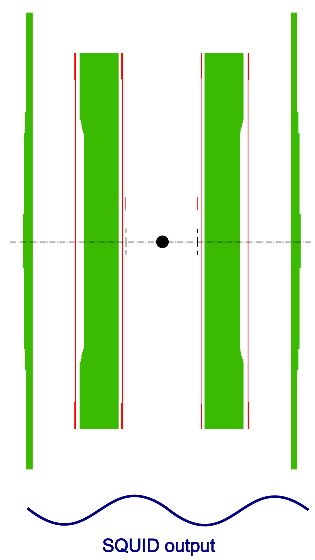
Figure 9.3: *Field profiles of the AC excitation coils (green) and the balance coil (red).* ***Not to scale.***



(a) *Unbalanced. The sample is clear of the pickup coils but the SQUID sees the AC excitation.*



(b) *The balance coil is tuned to the right amplitude and phase relative to the excitation to produce zero SQUID output.*



(c) *Now when the sample is returned to the centre position any signal that is seen must be due to the sample.*

Figure 9.2: *AC compensation distinguishes the sample signal from the AC excitation.*

The fields from the excitation and balance coils are of course both AC fields driven by complex AC currents, described by complex numbers with both amplitude and phase. It is therefore not enough to control the relative amplitude of the balance coil. The phase difference between the controlling currents must also be tuned to achieve zero signal. This must be done for each frequency that will be used during your AC measurements. The order of events when making an AC measurement is as follows:

1. Enter an **AC task list** which defines a set of frequencies to measure and the number of cycles for each (effectively giving the capture time).
2. Tune the balance coil for each frequency in the task list.
3. Perform a single measurement or begin a sequence.

9.1 Task list

The **AC Task List** contains the parameters that will be used whenever an AC measurement is made, either as a single measurement or as part of a sequence. At each (**B**,T) set point, all the measurements defined in the task list will be performed. The list can be edited via the **Configure Data Acquisition** window (section 13.1), the **System Configure** window (13.2) or in the **DLA Autotune** window (see below).

The task list displays one "task" at a time, containing the following parameters:

Fref The frequency to use for AC excitation. The signal is measured at the same frequency.

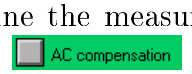
N cycles Number of cycles, effectively sets the capture time. E.g. 20 cycles at 10 Hz gives a 2 second measurement.

A0 Amplitude of main AC excitation driving signal

Ph0 Phase of main AC excitation driving signal

A1 Amplitude of balance coil driving signal

Ph1 Phase of balance coil driving signal

The frequency and number of cycles are where you define the measurements you wish to make. The amplitudes and phases are tuned using the 

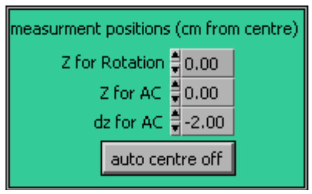
tool, located on the main menu.

9.2 Preparing system for AC measurements

To perform AC measurements the AC coils must be connected. Find the cable connected to the back of the rack in the socket labelled **AC coils**. Plug this cable into the 12-pin Fischer socket on the back of the VTI. This cable can cause noise in DC measurements if it is left plugged in when not in use. This is why it should only be plugged in when making AC measurements and removed at other times.

9.3 AC Compensation

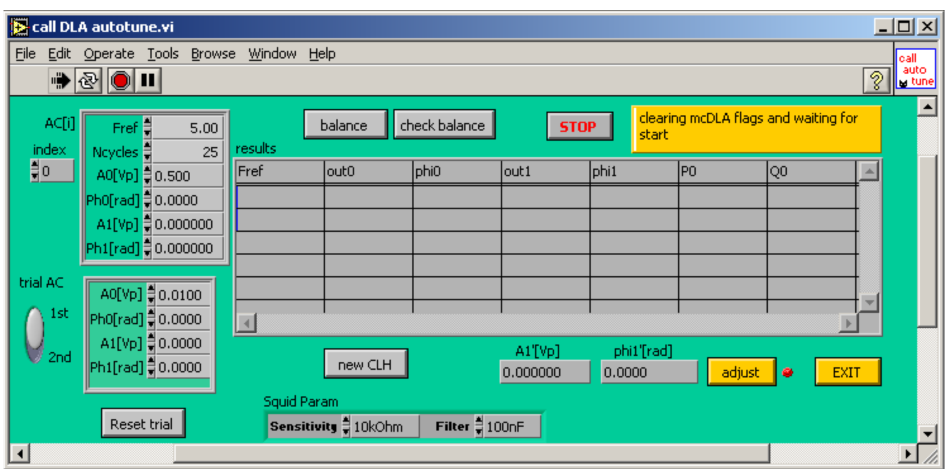
The excitation parameters required to balance the SQUID output to zero without the sample present must be set for each frequency/Ncycles task in the AC task list before a measurement is taken. These are found using the AC compensation process. Click [AC compensation] on the main menu to begin tuning the balance coil for the measurements you want to perform.The sample is also moved away from the pickup coils so that the coils can be balanced. The position of the sample during balancing can be set in the **measurement positions** box in the **Configure Data Acquisition** window.



Z for AC Defines the position of the sample during an AC measurement. By default this is at the centre position[12].

dz for AC The relative position of the sample during balancing (figure 9.2b).

The **DLA autotune** window will appear



The task list is shown in the upper left corner. Use the index control to select the task to tune the balance coil for. The tuned parameters are stored in the task list to be used when performing an AC measurement. The best parameters are found by an iterative process that will be explained below. The results of each step in the process are displayed in the **results** table.

[12] If the sample has been centred with an offset then this offset should be entered here

For each frequency we are looking for a combination of excitations of the main AC coil and the balance coil that minimises the output amplitude of the AC SQUID signal. Let's write this as

$$f_{main}\left(a_0 e^{i\phi_0}\right) + f_{balance}\left(a_1 e^{i\phi_1}\right) = A_{out} e^{i\Phi_{out}} \tag{9.1}$$

where $f_{main}(c)$ gives the complex response of the SQUID to the main AC coil being driven with a complex input and $f_{balance}$ is the same but for the balance coil. We want to make the right hand side of this equation equal to zero by choosing the right values for a_0, ϕ_0, a_1 and ϕ_1. To do this we perform a trial in which we make two measurements, exciting exciting each coil separately. This gives us two complex equations that can be solved to find an approximate solution for our parameters. The process iterates as follows.

Iteration 1 You set the values to use for the first iteration in the **trial AC** box. The switch toggles the box between the settings for first and second trial measurements. Set **A0** to a non-zero value for the 1st and **A1** to a non-zero value for the 2nd and then click 

. The system will perform the two measurements and display the results in the table. The values in the table are:

Fref Frequency being used

out0 Amplitude of the main AC coil excitation - equivalent to A0.

phi0 Phase of the main AC coil excitation - equivalent to Ph0.

out1 Amplitude of the balance coil excitation - equivalent to A1.

phi1 Phase of the balance coil excitation - equivalent to Ph1.

P0 Resulting amplitude of the SQUID output signal in phase with the main AC excitation.

Q0 Resulting amplitude of the SQUID output signal in quadrature ($\frac{\pi}{2}$ out of phase) with the main AC excitation.

We now have the following information

$$f_{main}\left(a_0 e^{i\phi_0}\right) = A_0 e^{i\Phi_0} \tag{9.2}$$

$$f_{balance}\left(a_1 e^{i\phi_1}\right) = A_1 e^{i\Phi_1} \tag{9.3}$$

where a_j and ϕ_j are the input amplitudes and phases and A_j and Φ_j are the output amplitudes and phases for the two measurements. The software solves these equations to determine new values for the amplitude and phase of the balance coil $a_1^{'}$, $\phi_1^{'}$. These are shown in the **A1'** and **Ph1'** boxes beneath the table. The values of **A1** and **Ph1** for the 1st trial in the **trial AC** box are zero for the first iteration. After the first trial has calculated $a_1^{'}$ and $\phi_1^{'}$, click 

. This adds these values to **A1** and **Ph1** for the 1st trial in the **trial AC** box, ready for the second iteration. Adjust can only be pressed once. The indicator light to the right of the button turns green when adjust has been pressed.

Iteration 2 In the first iteration the balance coil input values which were calculated were added to the values used for the 1st trial. We now run the trial measurements again by pressing check balance. Instead of equations 9.2 and 9.3, the second iteration gives us

$$f_{main}\left(a_0 e^{i\phi_0}\right) + f_{balance}\left(a_1^{'} e^{i\phi_1^{'}}\right) = A_0 e^{i\Phi_0} \tag{9.4}$$

$$f_{balance}\left(a_1 e^{i\phi_1}\right) = A_1 e^{i\Phi_1} \tag{9.5}$$

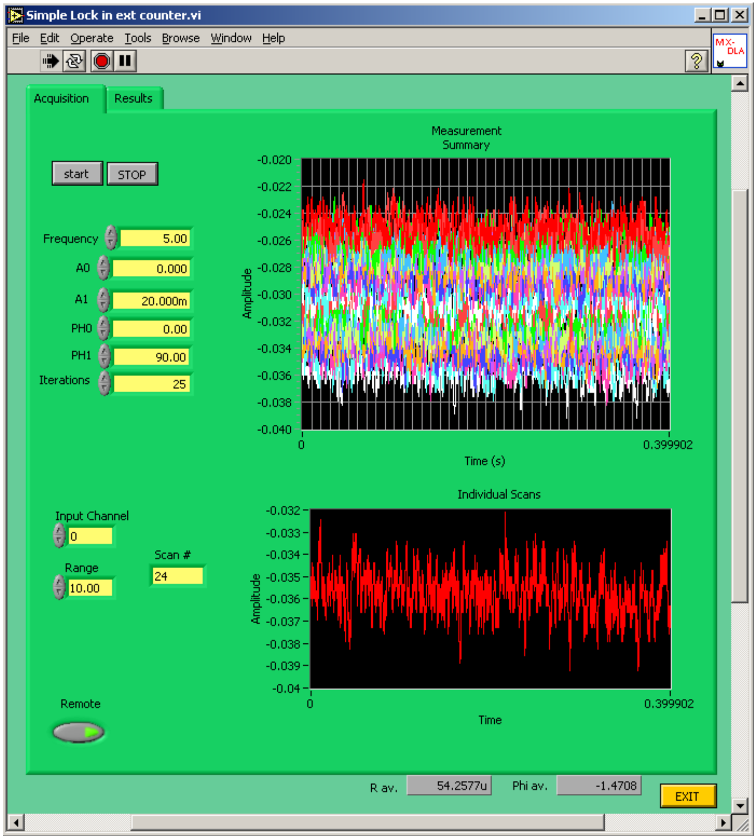
Notice that equation 9.5 is identical to equation 9.3 because the settings for the 2nd trial were not altered. After pressing check balance the system makes only one trial, for equation 9.4, and combines it with equation 9.3 from the first iteration when solving[13]. The software again solves the equations to find values for the amplitude and phase of the balance coil $a_1^{''}$, $\phi_1^{''}$. These are the values you would get if the main coil alone, in the first iteration, were as uniform as the combination of main and balance coils in equation 9.4. Therefore by clicking adjust which adds $a_1^{''}$ and $\phi_1^{''}$ to $a_1^{'}$ and $\phi_1^{'}$ respectively, we get an improved solution.

Further Iterations The process continues, using check balance for each new iteration, and in this way the solution converges to the best one. It usually only takes a few iterations to achieve sufficiently small SQUID output[14].

[13]pressing balance clears the results table and performs both trial measurements. Whilst this would lead to the same tuning parameters, the results table is cleared so the output amplitude from the previous iteration is lost. Using check balance is faster because it only makes one new measurement each iteration. The output results are also displayed on consecutive lines so the relative success of each new iteration can be compared.

[14]within the background noise

9.3.1 Acquisition window



During any AC acquisition, whether for balance or for taking a measurement, the acquisition window appears displaying the results of the acquisition. The upper graph displays all of the scans taken during the measurement and the lower graph shows the individual scans as they are taken. Each scan is two periods of the excitation frequency and the number of scans taken is determined by the length of the required acquisition as set by the number of cycles entered in the task list for that frequency.

During the balance procedure you should see the results of each of the test excitations appear as sinusoidal waves. If this is not the case then check your trial settings to make sure you are exciting one coil only on each trial. If the trial settings are OK then there may be a fault and you should contact Cryogenic.

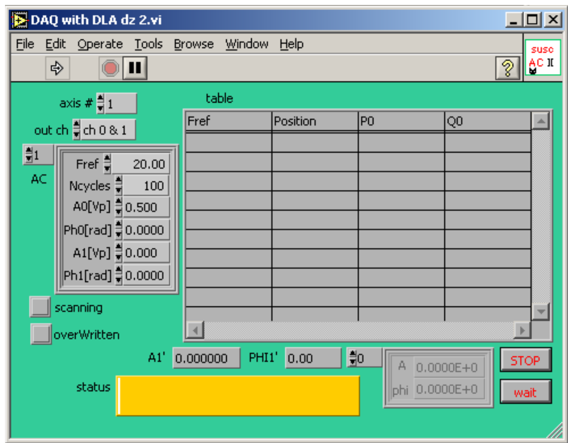
9.4 Measurements

Every time a measurement is made the following things happen:

- The sample is moved to the clear position
- The balance is checked by a test excitation at the first frequency on the task list to see if the SQUID output is still sufficiently small without the sample in place

- The sample is moved back to the measure position
- An acquisition is taken with the sample in place

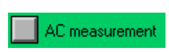
During each measurement, two windows will appear; the **acquisition window** and the **summary window**. The acquisition window is shown and described above. The summary window looks like this



The table records a summary of each of the measurements taken at each frequency. This is the raw data from which the results are calculated. As such it is not usually necessary to see this table the summary window and acquisition window will both close automatically when the measurement is finished. To prevent the windows from closing, for example so that you can see the amplitudes of each measurement in the summary window, press 

during the acquisition. This will cause the program to pause after the measurement before closing the window. It will wait until you press the same button again, there is no time-out.

9.4.1 Single AC measurements

A single measurement is performed by pressing 

from the main menu. This automatically performs each of the measurements in the AC task list. The summary and acquisition windows automatically close after the measurement unless you press 

. A single measurement is useful for determining if your sample has a strong enough signal to be detected in AC mode.

9.4.2 Sequences

AC measurements can be made as part of an automatic sequence by choosing **AC** for the “task” when generating a sequence.

10 ^{3}Helium Option

10.1 Introduction

The ^{3}He sample probe is used in place of the standard sample probe to measure at temperatures down to $300\,mK$. The sample is mounted inside a vacuum can which isolates the sample from the VTI allowing you to cool the sample below the lowest temperature of the VTI. The probe is filled with pure ^{3}Helium which is liquid below $3.19\,K$. To achieve base temperature, liquid ^{3}He is condensed in a reservoir, where it is in thermal contact with the sample. We can then pump on the liquid ^{3}He, reducing the temperature to $\sim 300\,mK$.

10.1.1 Vapour Pressure Cooling

The temperature of a liquid is pressure dependent. By controlling the vapour pressure above the liquid we can control the temperature. Liquid Helium is often used for this purpose because it is so cold even at ambient pressure and readily achievable pressures can be used to cool to around (or even below $1K$) for ^{4}He and below $300\,mK$ for ^{3}He. The vapour pressure-temperature curves for ^{3}He and ^{4}He are shown in figure 10.1. Since ^{3}He has a lower atomic mass it has less kinetic energy and therefore lower temperature than ^{4}He for any given pressure. Cooling is possible as long as some liquid remains.

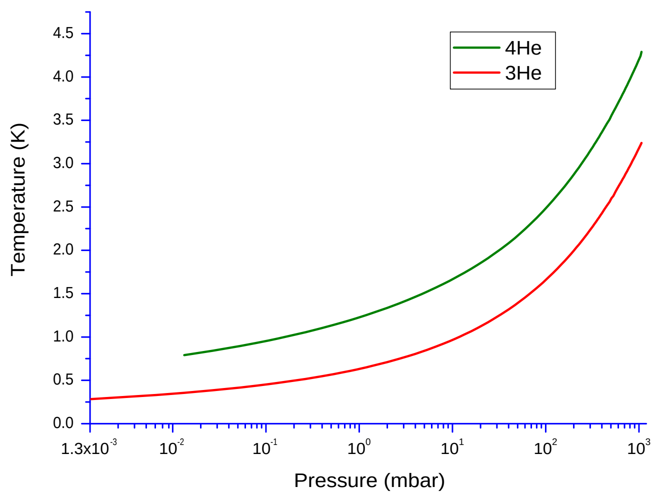
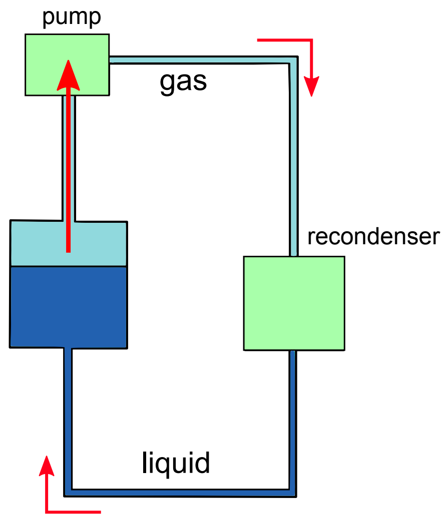


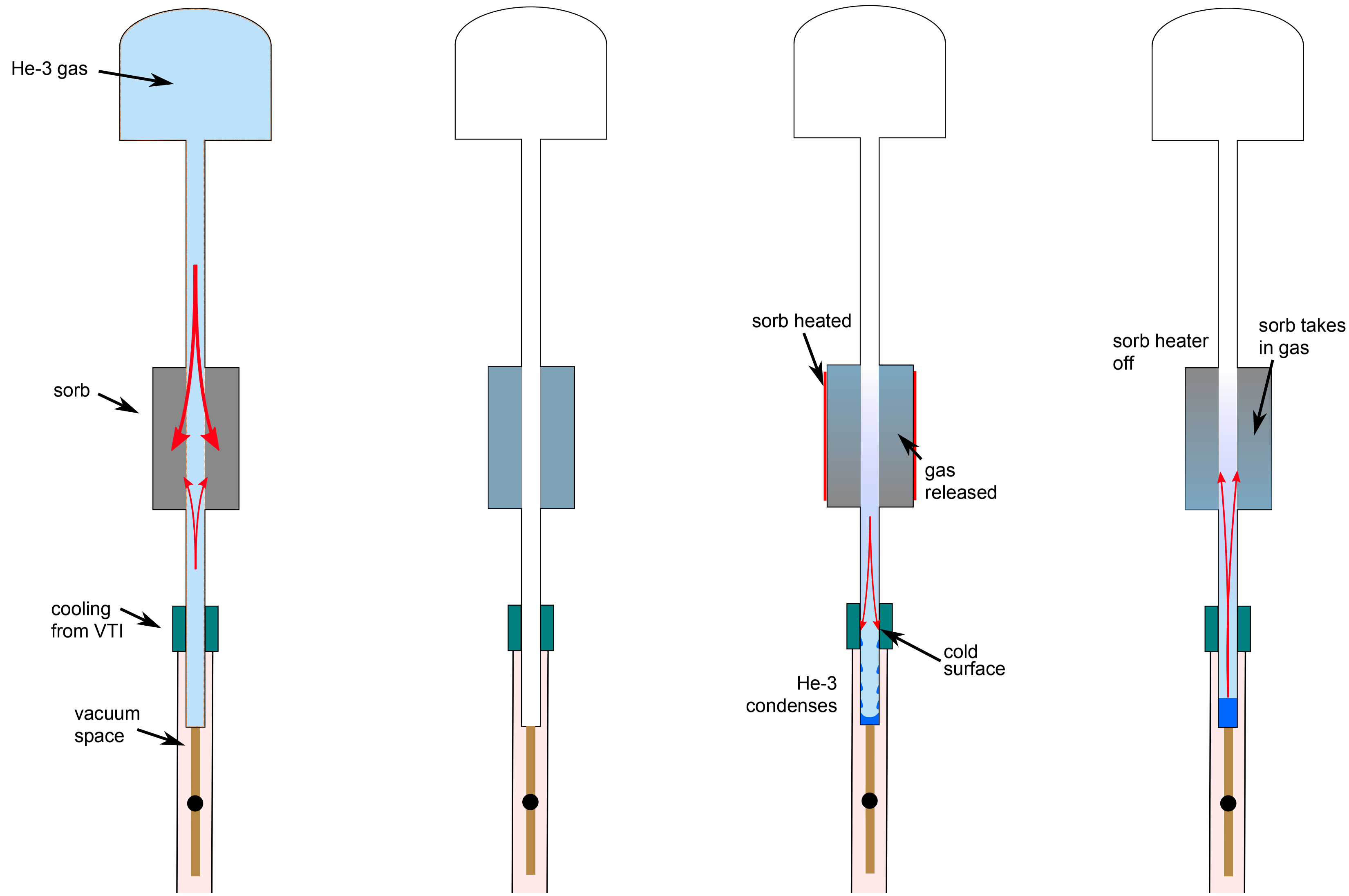
Figure 10.1: *Vapour pressure - Temperature curves for ^{3}He and ^{4}He.*

Continuous vs Single-shot

If we want to reach the lowest temperature then we must use ^{3}He and not ^{4}He. However ^{3}He is rare and very expensive so we don't want to waste any. We must therefore employ a closed system where none of the ^{3}He is lost. This could theoretically be done in a continuous fashion as shown here



This is very difficult to achieve in practice however, and the risk of losing ^{3}He through a moving seal would be ever present. A much safer option is to completely enclose the gas in a sealed container with no moving parts and somehow cycle through condensing the gas to liquid and pumping on it to achieve base temperature. This is a *single-shot* method. A temperature can be maintained as long as there is liquid remaining but the liquid is not replenished and so once it has all evaporated the temperature will increase. The process by which this is achieved in the ^{3}He sample probe is described in the following sections.



(a) *VTI cools insert. As the sorb gets cold it takes in all the He-3 gas.*

(b) *When the insert is cold, all the He-3 gas is in the sorb, leaving a vacuum.*

(c) *The sorb is heated, releasing He-3 gas. Since the bottom of the tube is colder than the top the gas travels downwards where it condenses on the cold walls of the tube.*

(d) *Sorb cools back down and acts as a pump, reducing the pressure above the liquid which reduces the temperature to* $\sim 300\,mK$.

Figure 10.2: *Helium-3 cooling. (b) shows the state of the probe before and after a recondensation and measurement cycle.*

10.1.2 Condensation in the ^{3}He sample probe

The base temperature of the VTI is sufficiently cold to condense the ^{3}He in the probe. The recondensation process is shown schematically in figure 10.2. The probe contains a charcoal *sorb*. When the charcoal is cooled below around $50\,K$ it begins to adsorb Helium. By the time the sorb is at the base temperature of the VTI it has adsorbed all of the ^{3}He, leaving a vacuum. The probe is now in the state shown in figure 10.2b. The sorb is fitted with a heater to allow us to control the gas in and out of it. By heating the sorb we release the gas. The bottom of the probe is colder than the top and so the gas travels downwards as it leaves the sorb. When it reaches the cold surface near the bottom of the tube, as shown in figure 10.2c, the gas condenses. The liquid ^{3}He now drips down into the ^{3}He reservoir at the bottom of the probe, where it is in thermal contact with the sample[15]. Once the gas has been liquefied the sorb is cooled back down. It then starts to re-adsorb the gas that evaporates from the liquid Helium, maintaining a very low pressure. When the sorb is fully cold the pressure approaches around 1.5×10^{-3} mbar, and the sample temperature reaches $\sim 300\,mK$. The exact base temperature will vary slightly between systems.

10.2 ^{3}Helium sample probe

Figure 10.3 shows a schematic diagram of the sample probe inserted into the VTI. The real probe is pictured in figure 10.4. The ^{3}He is enclosed in the dome shaped head and the central tube. The ^{3}He space is shown in blue in figure 10.3. At room temperature the ^{3}He is a gas and fills the whole of this volume. ^{3}He is loaded into the probe through the red valve. Once loaded, this valve should **never** be opened. The ^{3}He space is connected to the main sorb which is used during recondensation and to achieve base temperature by pumping as described in figure 10.2. The main sorb is surrounded by a heating coil to control the adsorption and release of ^{3}He gas. The sample is mounted on a high conductivity silver or sapphire rod which attaches to the end of the probe. This is then surrounded by a vacuum can which pushes on to a metal cone seal on the probe. This can must be pumped out to a good vacuum to thermally isolate the sample from the ^{4}He space or we will not be able to cool the sample below the temperature of the VTI. The vacuum can is evacuated through a pumping line that runs from the cone seal all the way up to the top of the probe. The pumping line passes through a second, smaller charcoal sorb. This *mini-sorb* takes care of any tiny amounts of ^{4}He that may be left in the can or the pumping line after flushing and pumping out the vacuum sample space.

The probe has three thermometers, one on each of the sorbs and one in the liquid reservoir at the bottom of the ^{3}He space. There is also a pressure transducer in the head of the probe which is used to monitor the recondensation process. The temperatures and pressure from the thermometers and transducer are displayed in the temperature control window of the software. The temperatures are also displayed on the front panels of the appropriate Lakeshore temperature controllers. These are listed in table 1.

[15]Refer to 10.4 for information about mounting samples on the ^{3}He sample probe.

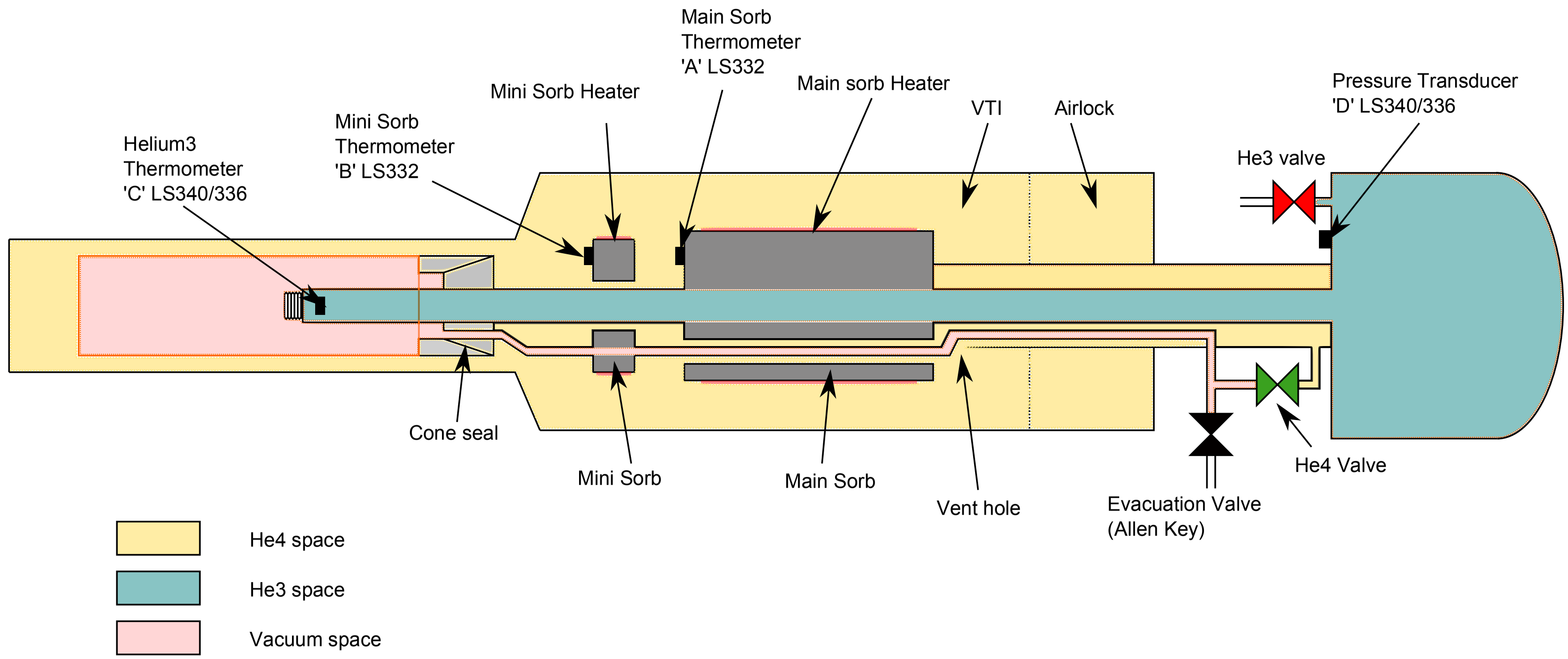


Figure 10.3: *Schematic diagram of the second generation Helium-3 sample probe. The first generation probe is the same but lacking the Evacuation valve shown in black.*

Figure 10.4: ^{3}He *probe*

Sensor	Location	Controller display
A	Main Sorb	LS332/335
B	Mini Sorb	LS332/335
C	^{3}He liquid reservoir	LS340/336/350
D	^{3}He gas chamber	LS340/336/350

Table 1: *^{3}He insert sensors and their corresponding Lakeshore Temperature Controllers. A-C are thermometers, D is a pressure transducer.*

10.2.1 Connections

There are three cables that connect to the probe to control and read the sorb temperatures, sample temperature and gas chamber pressure. When not using the probe these cables can be left connected at the rack side and hung in a convenient location ready for use. All three cables are different so it is impossible to plug them into the wrong sockets. When using the probe, wait until it is lowered into the cryostat before connecting the cables to avoid unnecessary stress on the shaft.

Sorbs 12 pin connector on the probe head, splits to 2 Lakeshore connectors and 2 pairs of wires, one with banana plugs. The lakeshore connectors plug in to the rear sockets for inputs A and B on the lakeshore 332/335 controller and these control the temperature of the Sorbs. The pairs are used for power to the sorb heaters. The 332 and 335 temperature controllers have one banana jack socket output for heaters and so the second sorb is heated using the analog output. The pair of bare ended wires are connected to the analog output "+" and "-" terminals of the screw block at the rear of the controller.

Sample 6 pin connector on the probe side, Lakeshore connector on the rack side. This is connected to input C of the Lakeshore 336/340/350.

Pressure Transducer 5 pin connector on the probe side, splits on the rack side to a Lakeshore connector and a pair of wires for powering the transducer. For the 336 and 350 controllers the output used is one of the analog outputs, usually output 3. For the 340 the digital I/O connection is used.

10.3 Collapsible Airlock

The ^{3}He probe is longer than a standard sample probe and does not fit into the standard airlock. The ^{3}He probe is fitted with a collapsible airlock to accommodate the extra length. It fits into the VTI in exactly the same way as the standard airlock. Insert into the bayonet fitting and turn the locking ring clockwise to secure the airlock[16]. Once the airlock has been

[16] After pumping out the airlock it is usually necessary to tighten the locking ring a little more.

pumped out and the probe is lowered into the VTI, the airlock can be collapsed to allow the probe head to sit on the sample transport mechanism.

10.4 Preparing probe for measurement

We've seen how the probe works in principle, now let's look at how we really use it to make a measurement. The probe is shown in figure 10.4. The probe comes with two options for mounting samples, a silver U-section tube and a sapphire tube. Both of these have very high thermal conductivity so the sample is thermally coupled to the ^{3}He liquid in the reservoir.

When using the silver sample mount, care should be taken not to bend the silver as this may cause it to touch the inside of the vacuum can and thermally couple the sample to the VTI. When using either the silver or the sapphire, the mount should be attached to the probe as straight as possible for the same reason. Now we prepare to close the vacuum can. Clean both halves of the cone seal, on the can and on the probe, using a little ethanol. Next apply a small amount of silicon vacuum grease to the inner cone on the can. To close the vacuum can, hold only by the stainless steel top section and push gently on to the steel cone on the probe.

Note: Do not grip the can tightly except on the stainless steel top section. The walls of the can are thin and easily damaged unless handled carefully. Only push the can gently onto the cone seal. When the can is pumped out the vacuum should pull the can onto the cone seal sufficiently to make a good seal. It may be necessary to rotate the can to find the smoothest fit before pumping out.

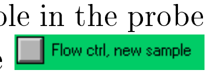
10.4.1 Pumping out

The vacuum space of the ^{3}He probe can now be evacuated. In the second generation probes this can be done in two ways, see below, but for the first generation probes only method one is applicable. The second generation probes have an additional pumping port which allows you to pump out the vacuum can using an external pump. This is opened and closed using an Allen key. If using a second generation probe, this is the recommended pump out method as it allows you to check the vacuum seal before inserting the probe.

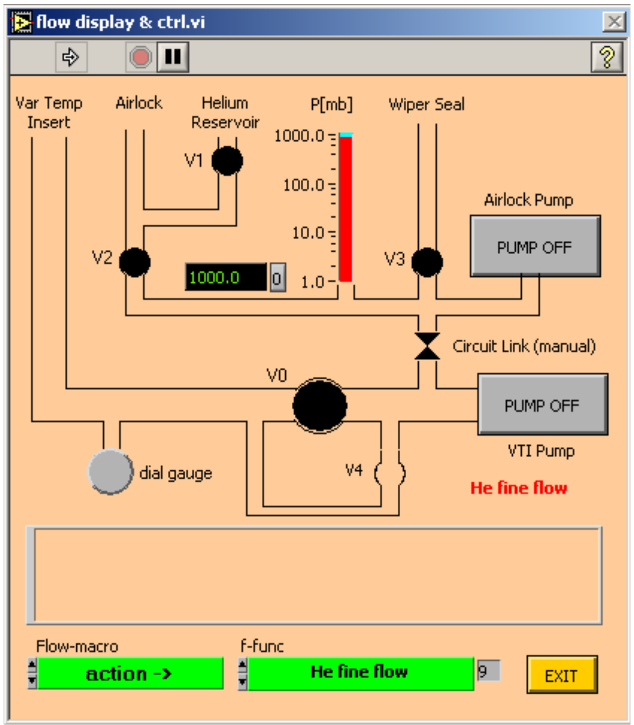
Method 1: Pump out via airlock

To use the airlock to pump out the probe, the evacuation valve (black valve in figure 10.3) must be closed if using a second generation probe, and the green ^{4}He valve must be open. Close the evacuation valve using an Allen key. The ^{4}He valve is opened by turning anti-clockwise.

Note: The ^4He valve is green. The ^3He valve is red and should **not** be opened.

With the valves set in this way the airlock can be inserted into the bayonet fitting on the VTI (do not use the **load new sample** action in the flow control window). Turn the locking ring clockwise to secure the airlock. The airlock can now pump on the vent hole in the probe (figure 10.3). If it is not already open, in the software main menu, open the 

window.



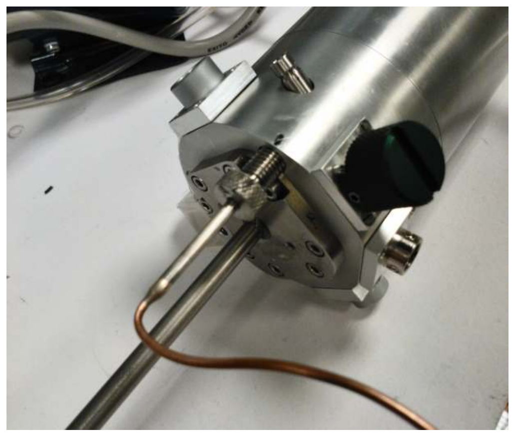
- Always pump on the wiper seal first. Switch the Airlock pump to ON and open V3.Wait for one minute.
- Open V2 to pump on the Airlock and probe.
- Leave pumping for at least 1 hour.
- Close the green ^{4}He valve to seal the vacuum space.
- Close V2 but continue pumping on the wiper seal.

It is important to close the green ^{4}He valve **before** lowering the probe. Otherwise the vacuum space is open to the VTI and the vacuum will be lost. This can lead to the vacuum can falling off into the bottom of the VTI.

The probe is now ready to be lowered. Skip to 10.6.

Method 2: Pump out with external pump

For second generation probes with the Allen key evacuation port, this is the preferred method of pumping out the probe as it allows you to check the vacuum seal before inserting the probe. This saves time if the seal has to be remade. The probe has an evacuation port and valve and comes with a special pumping line that screws into the base of the probe head. First insert the central shaft into the port, making sure it fits properly into the inner hole, and then insert and tighten the locking screw.



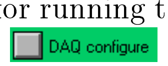
The other end of the pumping line is a standard NW16 fitting that can be attached to most standard pumps.

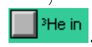
It is recommended to connect the pumping line to a leak detector and use this to pump out the vacuum can so that the seal can be checked.

1. Connect the pumping line to the probe and pump/leak detector as shown above.
2. Keep the green valve and Allen key pump out valve closed.
3. Pump out the pumping line and check for leaks.
4. When you are confident the pumping line is well connected, begin pumping out the vacuum space by opening the Allen key valve *Three full turns anti-clockwise*.
5. Wait until the pressure reaches 10^{-4} mbar, around 15-20 minutes for most laboratory pumps.
6. Check the seal with the leak detector using Helium gas. Alternatively, if using a standard turbo pump, check for leaks by spraying Helium gas and watching for spikes in the pressure.
7. If the seal is good, close the Allen key valve and disconnect the pumping line.
8. At this stage, if using a leak detector, you may wish to connect the probe to a stronger turbo pump to improve the vacuum but 10^{-4} mbar is sufficient. If pumping out again, always remember to first pump out the pumping line before opening the Allen key valve.

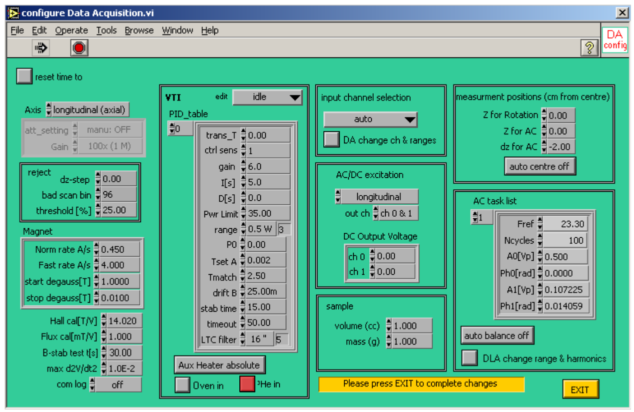
10.5 Preparing the System for ^{3}He probe

10.5.1 Software

The software automatically reconfigures for running the ^{3}He probe, however we must tell the software that the probe is present. Open 

and press 

, the button turns red.



To condense ^{3}He we need to set the VTI below 3.19 K. However, we also do not want to operate the probe with any liquid ^{4}He in the VTI as this disrupts the performance. The "Film Burner" vi prevents the build up of liquid in the VTI and should be active when using the probe. If the film burner window is not open, it can be found in the same directory as the main software. Open the vi and run it. The film burner is active when the "Burn" switch lights up green. If the switch is not lit then click it to activate the film burner.

The film burner uses the auxilliary heater to control the temperature of the sample chamber. There is an output meter showing the power, in Watts, of the the auxilliary heater. The temperature that it tries to set for sensor B comes from one of two possible sources, chosen with the "Select Source" switch.

Down The lower source is the set temperature from the standard T-Controller window. The set temperature will appear in the **Tset** box in the film burner window but you cannot change this value.

Up The upper source is a set temperature entered manually into the film burner window using the **set T** box. The "sens B" box shows the actual temperature.

The lower panel contains two controls used only with the ^{3}He probe.

Mini Sorb This sets the temperature of the mini-sorb, which is linked to the vacuum space of the probe.

A for He3 This sets the VTI set temperature for sensor A (heat exchanger), unless a sequence is running. If a sequence is running this will read the corresponding value from the **DAQ Main** window (4.9.1).

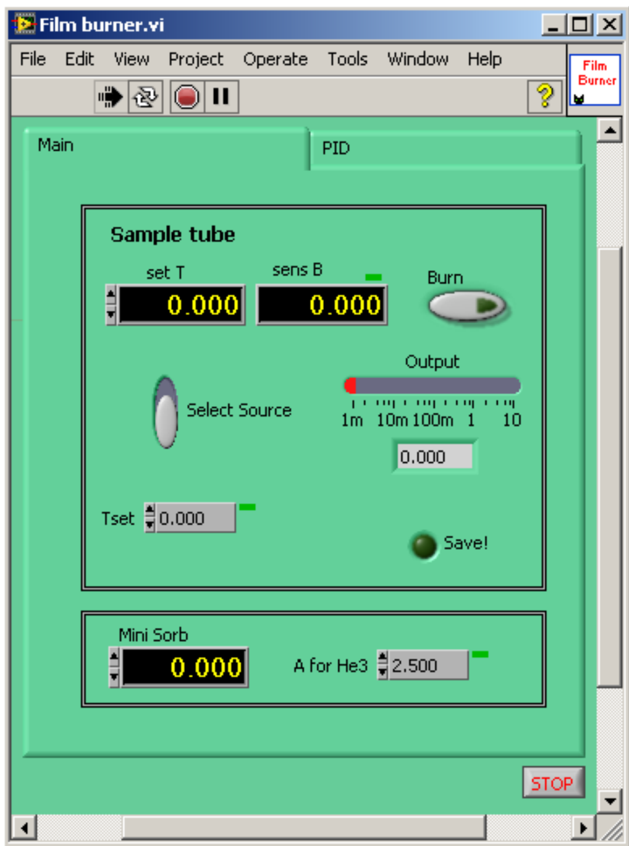


Figure 10.5: *Film Burner*

10.5.2 Cool down without collecting liquid

To avoid collecting liquid in the VTI:

1. Use the Film Burner
2. Set the temperature to 8 K and let it settle for several minutes
3. Go slowly down in 1 K steps to 4 K and then in smaller steps to 2.5 K

2.5 K is sufficiently cold to condense ^{3}He and if you are reasonably careful the VTI will not usually collect liquid at this temperature. You can tell if liquid is collecting because the VTI pressure will start to drop. Should this happen, take the VTI back to 8 K and wait for the liquid to boil off. The pressure will increase greatly as the liquid boils and will eventually settle back to where it started once all the liquid has gone.

10.5.3 Prepare stepper head

When inserted into the cryostat the ^{3}He probe rests on the green sample rod support, but the sample rod lock screws must be removed. These can be screwed into the under side of the support to prevent them from being lost.

10.6 Inserting Probe

It is recommended to insert the ^{3}He probe slowly with the VTI cold. The cables that allow the system to control and communicate with the probe should not be attached until *after* the probe is fully lowered. The probe is very long and even the weight of a cable pulling sideways from the top could cause the probe to bend.

10.6.1 Pumping out airlock

If the probe was pumped out using an external pump then the airlock needs to be fitted and evacuated. If the airlock was used to pump out the probe then the probe is ready to be lowered, in which case skip to 10.6.2. Fit the airlock into the bayonet fitting and tighten the locking ring. The collapsible airlock must be fully extended to accommodate the length of the probe. The usual procedure for inserting the sample probe is not appropriate when using the collapsible airlock. The extra volume, including the means that the automatic pumping cycle that occurs when you follow the **load sample** instructions does not pump for long enough to pump out the airlock sufficiently. Instead the airlock pumping/purging sequence must be performed manually.

1. Open 

2. Set the airlock pump to **PUMP ON**
3. Open valve V3 by clicking on the black dot to pump on the wiper seal, the pressure will drop quickly since the volume is small.

4. Open V2 to pump on the airlock. Wait for the pressure to reach 5-10 mbar.
5. Close V2 and open V1 to flush the airlock with Helium for around 1 minute, then close V1 again.
6. repeat the pump/flush cycle three times.
7. Finally, pump out the airlock until the pressure reaches $\backsimeq$ 5 mbar
8. Close V2 but leave the airlock pump ON to pump on the wiper seal.

The probe is now ready to be lowered.

10.6.2 Lowering the probe

Once the following conditions are met:

- vacuum can and airlock are both pumped out
- airlock valve V2 is closed (in the flow control window)
- Allen key valve and green ^{4}He valve both **closed** (manually)

open the gate valve and begin lowering the probe. As with the standard probe it should be lowered slowly. A good way is to lower the probe until the sorb and wires are below the level of the gate valve and secure it in place with a suitable clip. Waiting with the probe at this height allows the faster flowing gas in the upper neck of the VTI to precool the probe to around $200 - 220K$. After around 15-20 minutes continue to lower the probe until you see the VTI temperature rise, at which point stop lowering and wait for the temperature to fall. Continue in this way, not letting the temperature rise dramatically, until the probe is fully inserted. This is the most efficient way to reach a stable equilibrium with the probe inserted into the VTI. If you lower the probe quickly from room temperature you may find the VTI reaches around $250K$ and subsequently that it takes several hours to return to equilibrium at base temperature.

With the probe fully lowered, plug the three ^{3}He insert cables into their respective sockets on the head of the insert. The cables labelled and are all different so it is impossible to plug them into the wrong socket. They should also be plugged in to their corresponding outputs on the back of the electronics rack.

Before trying to measure, remember to open **DAQ configure** from the main menu and press 

to activate the probe.

Once the probe is activated the temperatures of the various thermometers are displayed. The normal **T-Controller** window that appears when the software is started usually displays only VTI thermometers A and B but after the ^{3}He probe is activated it also displays ^{3}He sensor C, i.e. the thermometer in the base of the ^{3}He space where liquid condenses. This is effectively the sample temperature. Note that the rate of change displayed next to this temperature is in mK/s. The main and mini sorb thermometer temperatures are displayed on the front panel of the Lakeshore LS332/335 temperature controller but are not shown anywhere by the software.

10.6.3 Check vacuum

After activating the probe, monitor the sorb temperatures, the sample temperature and the VTI temperature. The sorbs should come into equilibrium with the VTI, whilst the sample temperature (^{3}He sensor C) should remain at a higher temperature, up to around $100K$, because it is isolated by vacuum. If the sample temperature falls to the VTI temperature or close to it then the vacuum is probably not good enough and the probe needs to be removed and the cone seal remade. If the sample temperature remains high when the VTI and the other probe thermometers reach ~$2\,K$ then the vacuum is good and we can proceed to cool the sample down to $2\,K$ before condensing ^{3}He.

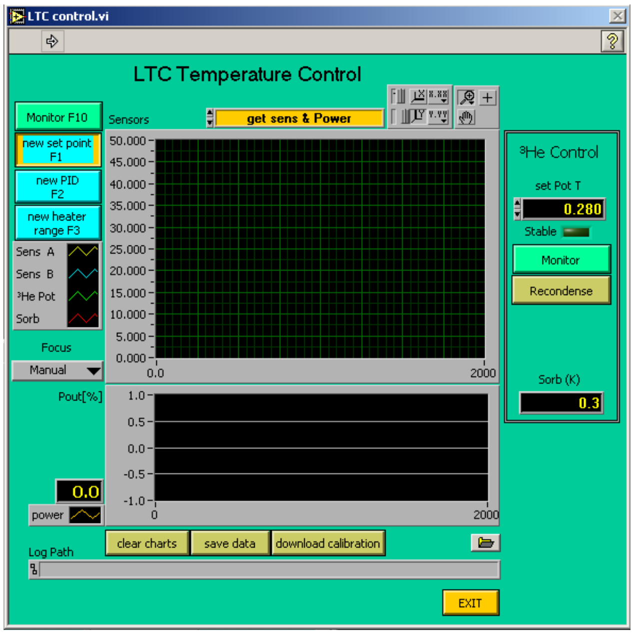
10.7 Cooling the sample to VTI temperature

Before we can reach $300\,mK$ we need to cool the sample to $2.5\,K$ (or whatever temperature you have set the VTI). To do this the main sorb is heated to release some ^{3}He which conducts heat between the sample and the walls of the probe just above the cone seal. Once the sample is cold the sorb heater is switched off and the ^{3}He gas returns to the sorb. The vacuum in the sample can is never broken during this process. To control the sorb temperature, set the temperature manually using the front panel controls of the Lakeshore LS332/335 temperature controller.

- Ramp the main sorb to $60K$ - this releases a small amount of ^{3}He gas which forms a link between the bottom of the liquid ^{3}He reservoir and the cold walls further up the tube which are cooled by the VTI.
  - *See Lakeshore manual for information about controlling the Sorb temperatures from the Lakeshore temperature controller.*
- Wait for the sample to reach VTI temperature. Leave for as long as possible to reach equilibrium, preferably overnight.
- When ready to measure, set the main sorb back to $2K$ - All ^{3}He gas is now readsorbed and the probe is ready for condensation.

10.8 Condensing Helium and Controlling Temperature below 2K

The sample temperature displayed in the **T-Controller** window is a display only. You cannot control the temperature by entering a new value the way you can with the VTI temperature. To control the temperature when using the ^{3}He you need to use the **LTC Temperature Controller** window which is accessed by clicking TC - control from the main menu.



When not using the ^{3}He insert this window is used to monitor temperature only. The ^{3}He **Control** panel on the right hand side is used to control the temperature of the liquid Helium reservoir, or pot, and hence the sample temperature. To cool below $2K$ we have to condense Helium as per figure 10.2. The whole recondensation process is automated and is performed by simply clicking Recondense. Recondensation can be done at any time.

The temperature of the ^{3}He reservoir, or pot, is shown in the upper graph along with the main Sorb temperature and the temperatures of the VTI thermometers A and B. The lower graph shows the percentage of maximum heater power. With the VTI sitting at its base temperature this should read zero.

Since cooling and temperature control are achieved by controlling the pressure above a pot of condensed ^{3}He, cooling and temperature control are only possible for as long as some liquid remains. Once all the liquid has evaporated from the pot the temperature will begin to rise and return to the temperature of the VTI.

The holding time at base temperature for the first recondensation after inserting the probe is often not as long as subsequent recondensation cycles because a true equilibrium has not been reached, unless the probe is allowed to cool for a long time, for example overnight. For the longest holding times, either leave the probe to equilibriate overnight after insertion or perform the first condensation and then wait for the temperature to rise before recondensing.

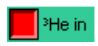
10.8.1 Setting temperature

To set a temperature using the ^{3}He Control panel of the LTC Temperature Control window, you must be in  rather than  mode. Click the button to toggle between the two. When in **Control** mode simply set a temperature in the **set Pot T** box. When the temperature is stable the **Stable** indicator light will come on.

The temperature is controlled by adjusting the temperature of the Sorb heater such that it will release or absorb Helium, thus adjusting the pressure above the liquid and therefore the temperature. The feedback loop monitors only the temperature of sensor C but the PID settings take into account the temperature dependent rate at which the Sorb releases and absorbs gas so that temperature stability is maintained at all temperatures.

10.9 Removing Probe

To remove the probe safely from the cryostat, follow the following procedure

- Deactivate the probe by clicking  in **DAQ Configure**.
- Unplug the cables from the head of the probe.
- Open the **flow control** window ()
- Stop pumping on the VTI by closing valves V0 and V4 and stopping the VTI pump
- Raise the probe until the vacuum can is completely in the airlock.
  - *The probe has been sat at 2K for some time so take care when raising the probe as it will be very cold.*
- Close the gate valve
- Flush the airlock with Helium to remove the vacuum by closing valve V2 and opening valve V1 for 30 s.
- Wait for the probe to warm up before removing the airlock to avoid moisture or ice condensing on the wiring.
- Turn the locking ring anti-clockwise and remove the airlock.

11 Furnace

The furnace, or oven, extends the temperature range of the system up to 700K. Like the ^{3}He insert, it is a special probe that replaces the standard sample probe for the extended temperature range. To use the furnace, the sample is mounted into a cavity in a solid silver rod, or glued to a silver "U" tube as for the ^{3}He insert, and the rod or tube is screwed to the probe.

Conceptually the furnace is much simpler than the ^{3}He probe. There is a resistive heating coil, wound around a ceramic former, which provides heating power. This is directly coupled to the sample rod. To eliminate thermal gradients along the length of the sample rod there is a Silver shield that is coupled directly to the heater at one end. This shield surrounds the sample rod and heats it by radiation. To isolate the furnace interior from the VTI the furnace is surrounded by a vacuum can. Unlike the ^{3}He probe which has charcoal sorbs to maintain vacuum at low temperature, the furnace probe must be continually pumped out during operation as the elevated temperature causes out-gassing from materials inside.

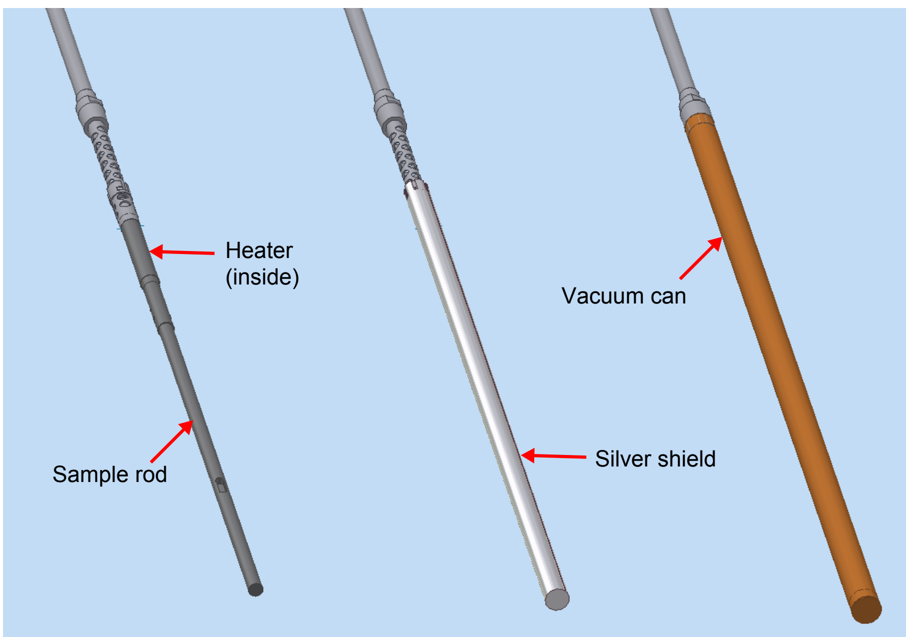


Figure 11.1: *Three views of the furnace, showing silver shield and vacuum can.*

The airlock is the same design as for the standard probe, except it is a little longer, and the furnace is inserted and lowered as normal.

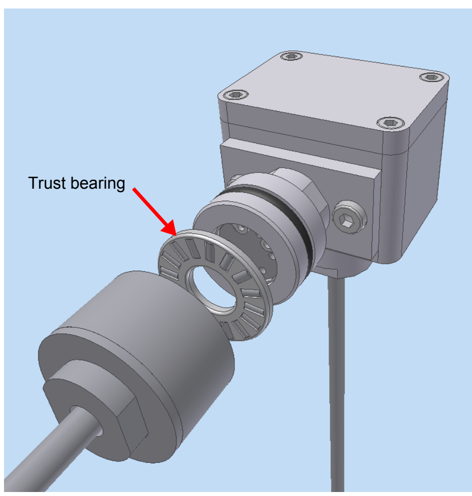
11.1 Preparing the Furnace

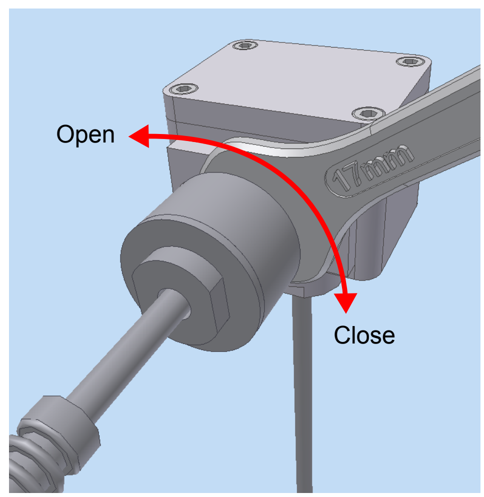
- Mount the sample on the silver rod or silver "U" section sample tube and screw to the probe.
- Push the silver shield over the top as shown in figure 11.1.

- Apply vacuum grease to the cone seal and push the vacuum can onto the probe. Do not grip the walls of the can tightly as they are thin and may bend.
  - *Vacuum grease should be applied to the cone seal slightly more liberally than with the ^{3}He vacuum can because the higher temperatures can cause some of the grease to burn away.*
- The furnace is now ready to be pumped out.

11.1.1 Pumping out

The head of the furnace probe has a pumping valve. To connect the pumping hose to the valve, make sure the trust bearing is in the cap fitting on the hose and push the cap onto the valve as shown. The valve is opened and closed with a 17mm spanner.





The recommended way to pump out the furnace initially is with a leak detector as this allows you to check the cone seal. The procedure then would be as follows:

1. Prepare the furnace and close the vacuum can as above.
2. Connect the pumping hose to the furnace head and open the valve.
3. Connect the other end of the hose to the pumping port of a leak detector and begin pumping.
4. Pump until the pressure falls to 10^{-4} mbar.
5. Check the seal with Helium gas, looking for a signal from the leak detector.
6. If the seal is good, close the valve and disconnect the leak detector.

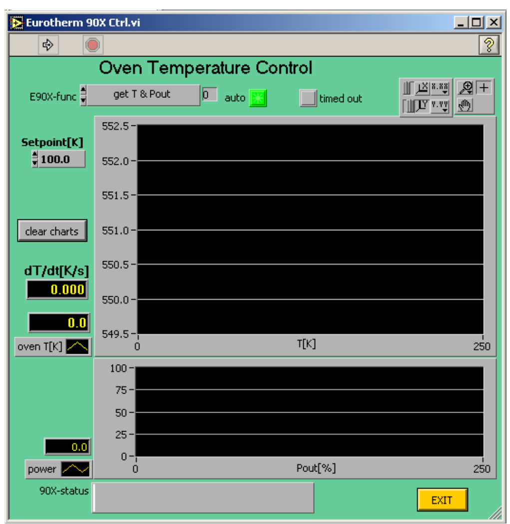
11.2 Inserting the probe

When the sample chamber is under vacuum the furnace can be inserted into the cryostat in the same way as the regular sample probe, using the **load sample** macro in the **flow control** window. When the probe is inserted, plug the Furnace cable into the 6-pin Fischer socket on the probe head, located on the opposite side to the pumping valve.

Once the furnace is in the cryostat and the cable is plugged in, open **DAQ configure** from the main menu and press Oven in, which tells the software that the furnace is installed. The VTI temperature is automatically set to 150K and the sample temperature control switches to the furnace thermometer. This is now the temperature that is monitored and recorded during measurements. The button turns red to indicate that the Furnace is active.

11.3 Temperature Control and Making Measurements

The furnace is used for making measurements at temperatures up to 700K. You can also measure down to the temperature of the VTI. This is automatically set to 150K when the **Oven in** button is pressed. To control the temperature manually whilst using the oven, open the **Oven Temperature Control** window by clicking Euro 90X T - control on the main menu.



To set a temperature, enter the desired value in the **Setpoint[K]** box. The upper graph displays the temperature and the lower graph displays heater power, the horizontal axis is time in both graphs. The rate of temperature change, current temperature and heater power are displayed down the left hand side. To reset the graphs click clear charts. The drop down menu "E90X-func" is obsolete.

Measurements can be made in the normal way, the software will take account of the furnace when setting temperatures provided you have pressed **Oven in** in the **DAQ Configure** window.

Overheating If the B thermometer on the VTI becomes too hot, the power to the furnace is automatically cut. If this happens it is likely that the vacuum is not good. The probe should be removed and the vacuum checked and remade if necessary. If the vacuum is good then contact Cryogenic for assistance.

12 SQUID Tuning / Monitor

To achieve maximum sensitivity and stability from the SQUID, the device must be carefully tuned. To see what this means let's look more closely at how the SQUID is used to detect flux. Figure 12.1 shows a schematic diagram of the flux detection system. The pickup coils form a closed superconducting loop with the input coil, which couples the flux in the pickup coil to the SQUID. The SQUID is a superconducting device and is capable of carrying a current up to a critical value without resistance. A bias current is applied to the SQUID that is larger than this critical current so that a voltage appears across the device. The bias current is a combination of a DC current and an AC modulation. The modulation is used by a digital lock-in amplifier to isolate the SQUID signal from external noise sources. There is a transformer at low temperature to prevent thermal gradients causing unwanted voltages across the SQUID.

The current-voltage characteristics of the SQUID are schematically shown in figure 12.2a. Above the critical current the device shows a voltage that is dependent on current and flux. By applying a bias current we can operate the device in this region. The voltage across the SQUID is a periodic function of flux threading the ring, not necessarily sinusoidal. The period of the function is one flux quantum, as shown in figure 12.2b. There are many texts that explain the underlying physics behind these properties of the DC SQUID (references) but here we will just use what we know about the current-voltage-flux characteristics to achieve what we want; the most sensitive and stable flux detector we can get.

Flux Locked mode

During a measurement the SQUID is operated in flux locked mode, where the output signal is fed back to the SQUID to apply flux in the opposite direction, keeping the SQUID output constant. The result that we actually measure is the voltage across a resistor in the feedback loop. Figure 12.1 shows that there are actually three resistors, corresponding to the three standard range settings, see 13.3.

In order that the feedback system works reliably we want to operate it in a region where the voltage response to flux is linear, as in figure 12.2b. If were were at one of the peaks in this figure instead then if the squid output voltage increased, would that be because the flux went up or down? how could the feedback loop tell whether to add more or less flux to balance out the change? Similarly any significant curvature in the region we lock to causes the feedback loop to not function properly. If the SQUID is not well tuned then the feedback system can allow the flux in the device to jump in flux quanta steps. We then jump from one locked mode to another. In figure 12.2b this would mean jumping forward or back by one period of the function. Tuning the SQUID amounts to finding the bias and modulation current settings that, for the given range, produce a good $V - \Phi$ function with the desired linear region being as wide as possible so that the locked mode is stable.

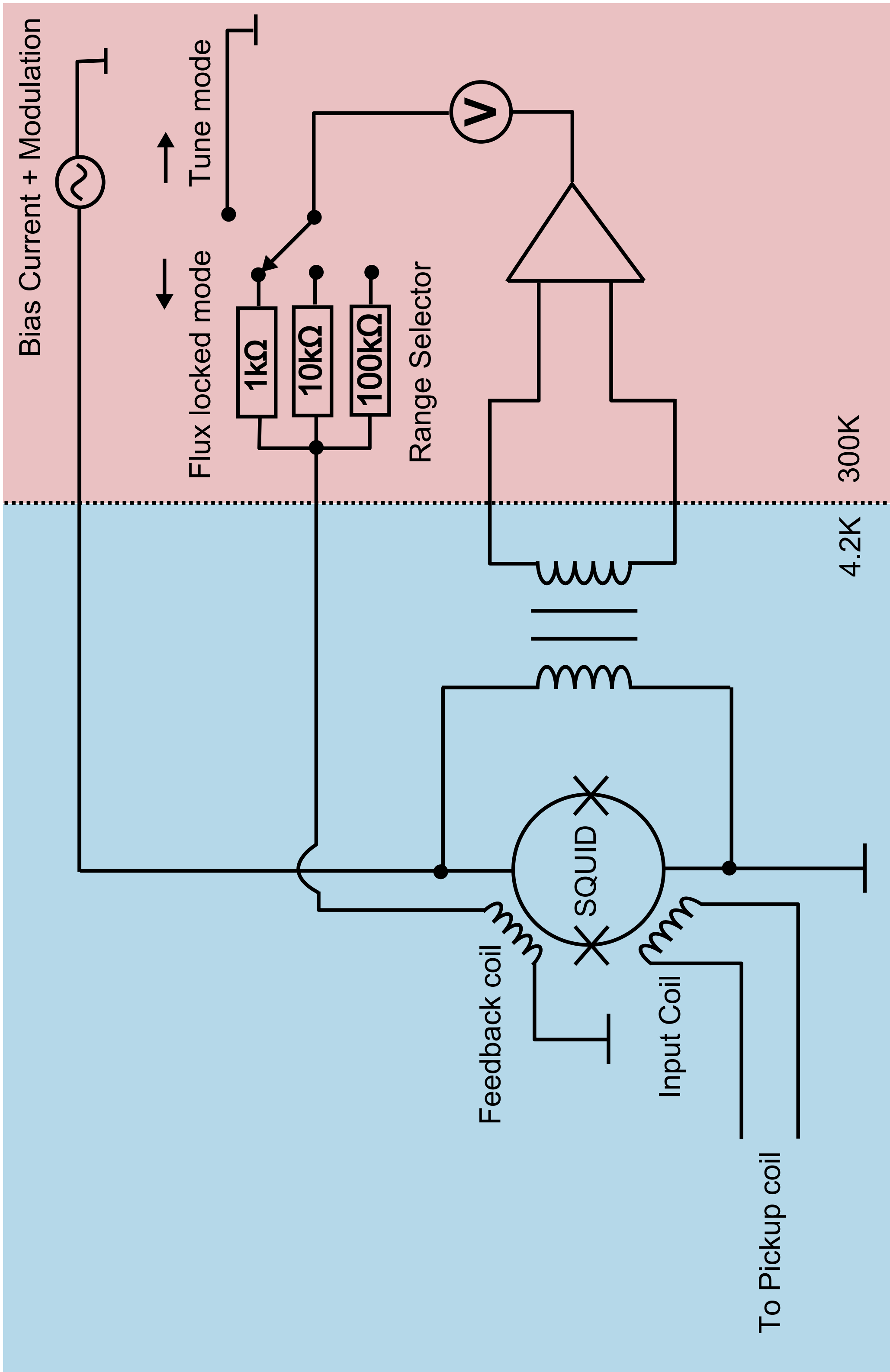
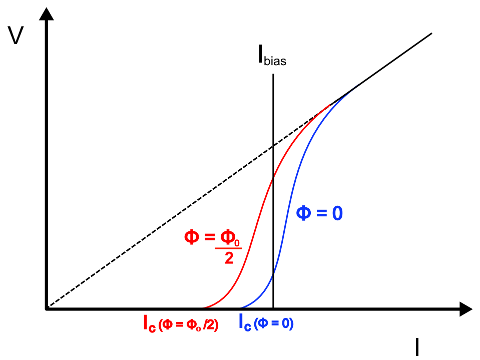
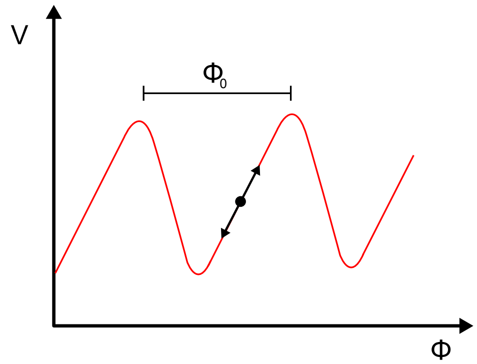


Figure 12.1: *Schematic diagram showing the operation of the flux detection system.*



(a) *typical current-voltage curve for a DC SQUID device. The curve is dependent on the magnetic flux* Φ *through the ring. The bias current is larger than the critical current so the device always shows a voltage across it during operation.*

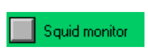


(b) *The voltage across the SQUID is a period function of flux* Φ *through the ring. The period of the function is one flux quantum* Φ_0*. A well tuned SQUID has a linear region to the flux response which makes* ***flux locked mode*** *more stable.*

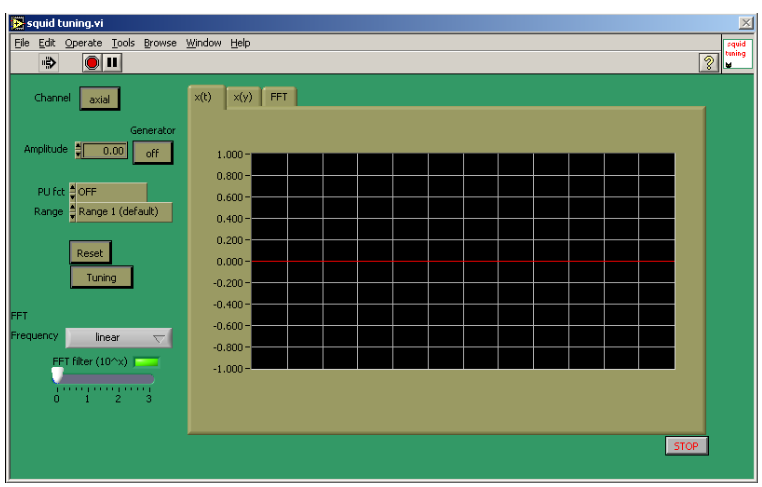
Figure 12.2

12.1 Tuning the SQUID

As stated above, tuning the SQUID is about finding the right bias and modulation current to give a $V - \Phi$ curve that will support a stable feedback loop and prevent flux jumps. We want a linear region around zero volts so that increasing or decreasing SQUID output voltage corresponds to a well defined change in flux and the feedback loop can react accordingly and in a stable fashion.

To monitor or tune the SQUID output, open 

from the main menu, the following window appears



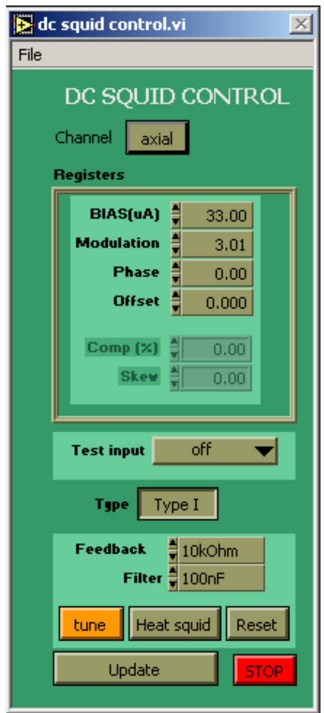
The graph displays the output signal from the SQUID in three different ways, selected by the three tabs above the graph.

x(t) displays the output as a function of time, in the currently selected range. In locked mode this is useful for monitoring the drift on the SQUID.

x(y) displays the output as a function of the test excitation applied with the generator. This is used for tuning the SQUID.

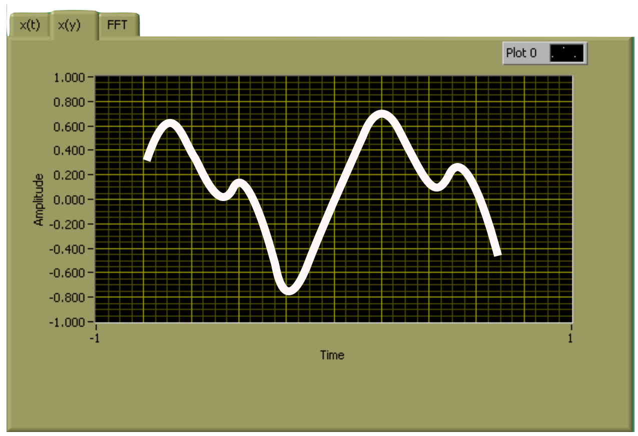
FFT displays the Fourier transformation of the output signal. This is useful for identifying the frequencies of any sources of noise.

To tune the SQUID, select **x(y)** and click Tuning. This brings up the **DC SQUID control** window.

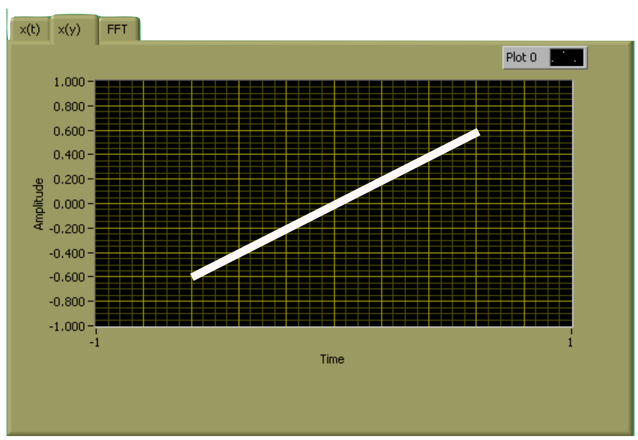


In order to observe the behaviour of the SQUID and tune it accordingly we need a test signal. The test signal is applied by switching on the **Generator** button in the **SQUID tuning** window. The amplitude is controlled with the **Amplitude** scroll buttons or by entering a value in the box. In order to enable the test signal to be read by the SQUID we have to enable the **Test input** in the SQUID control window. Select **0.5V/phi0** to begin with. Next, we must be in tune mode. The orange button in the DC SQUID control window toggles between tune and lock modes. In tune mode we follow the output of the SQUID as a function of applied flux, as in figure 12.2b.

The test signal from the generator is sinusoidal, but the response of the SQUID is not necessarily so. It is periodic in applied flux, and the period is one flux quantum, Φ_0. The amplitude of the test signal should be such that only one or two periods appear. To tune the SQUID, vary the **BIAS**, **Modulation**, **Phase** and **Offset** settings in the DC SQUID control window until the SQUID shows a linear response over as wide a flux range as possible in the



(a) *Tune mode*



(b) *Lock mode*

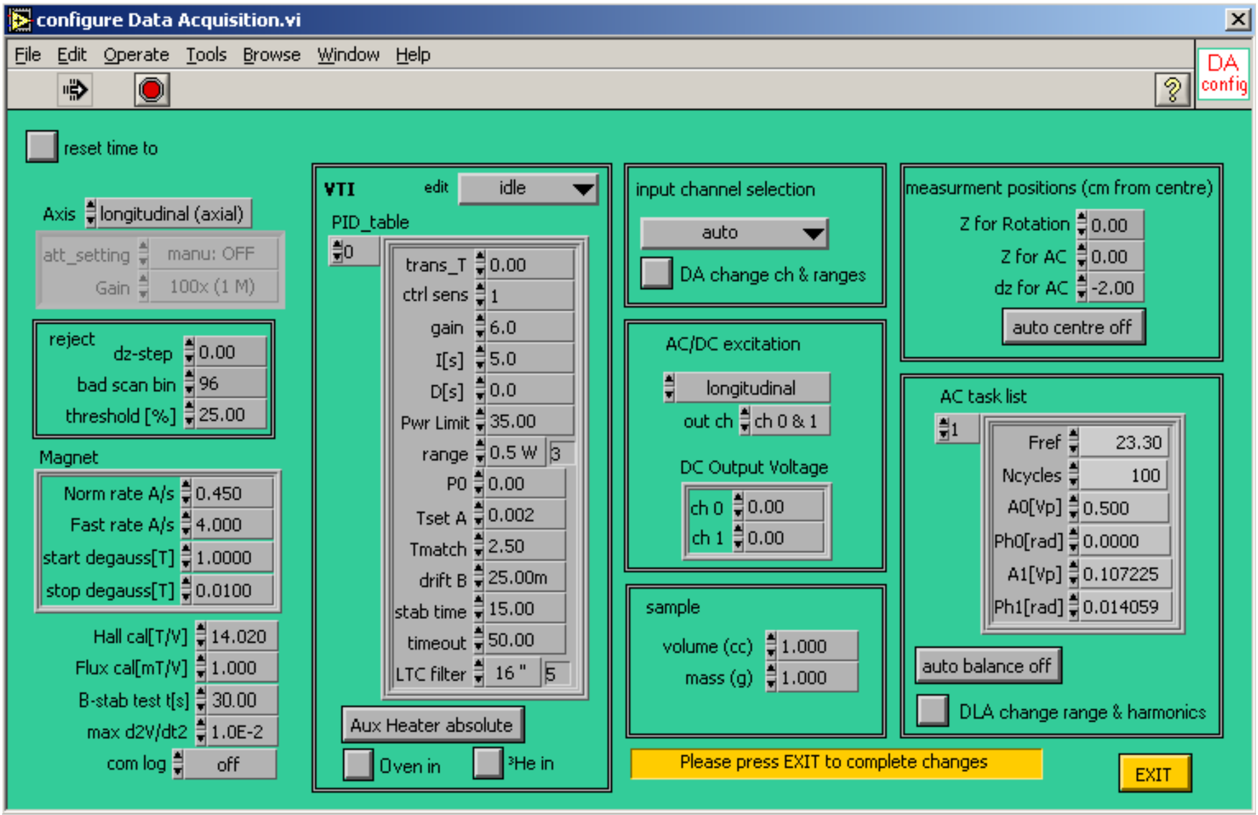
Figure 12.3: *A well tuned SQUID has a linear response to flux near to zero applied flux. This makes the feedback loop well defined and stable.*

centre of the graph where the test signal flux passes through zero, as in the example shown in figure 12.3a. Once you think the tuning looks good, put the SQUID into lock mode, with the test signal still active. The response should now be purely linear, as in figure 12.3b.

Once you are happy with the tuning, always remember to leave the SQUID in lock mode and switch off the test signal by setting **Test input** and **Generator** to **off**.

13 Software Reference

13.1 Configure Data Acquisition



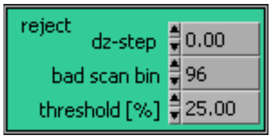
The configure data acquisition window is not required for routine DC operation of the system. It is mostly used as a testing area for making non-permanent changes to system settings related to data acquisition. Settings that are changed with this window will remain in effect until the software is next restarted and the default settings are read from the calibration file. The changes made here will be reflected in the S700 system configure window and to make permanent changes, use the system configure window to save the current settings in a new calibration file.

There are four columns of controls in the configure data acquisition window. To confirm and use settings click 

.

This button resets the time reference, stored when taking data, to the current system time.

Reject



These settings control the rejection of "bad scans". Every now and then you may get a flux jump during a scan that appears as a discontinuity in the voltage. The software can recognise this has happened and disregard and repeat the scan. This is achieved by a process that programmers call "binning", which produces a histogram of the gradient during the scan, see diagram below. The derivative of voltage against position is calculated at each point displayed on the scan and the range of gradients is divided into a number of 'bins'. Each gradient is sorted into the appropriate bin and the number of gradients in each bin is counted. A good scan is a smooth continuous curve. There is a range of gradients from zero at the minima and maxima to a maximum gradient at the slopes of the central peak and the gradient varies smoothly in between. This gives a histogram like the one shown in the upper half of the diagram below, where all the bins are connected. If a flux jump occurs during a scan however, there is a point where the gradient is much larger than any other point on the scan. This gives a histogram like the one shown in the lower half of the diagram below. Remember the whole range is divided into the given number of bins, so in this case each bin will cover a wider sub-range. Since only one point on the curve has the much larger gradient, the next lowest gradient is much lower, leaving a number of empty bins. The user can adjust the sensitivity to bad scans with the **bad scan bin** and **threshold[%]** controls.

dz-step Obsolete. Not used by the software and will be removed in future software updates.

bad scan bin This sets the number of bins to use when determining the quality of scan.

threshold[%] This sets the acceptable percentage of empty scans.

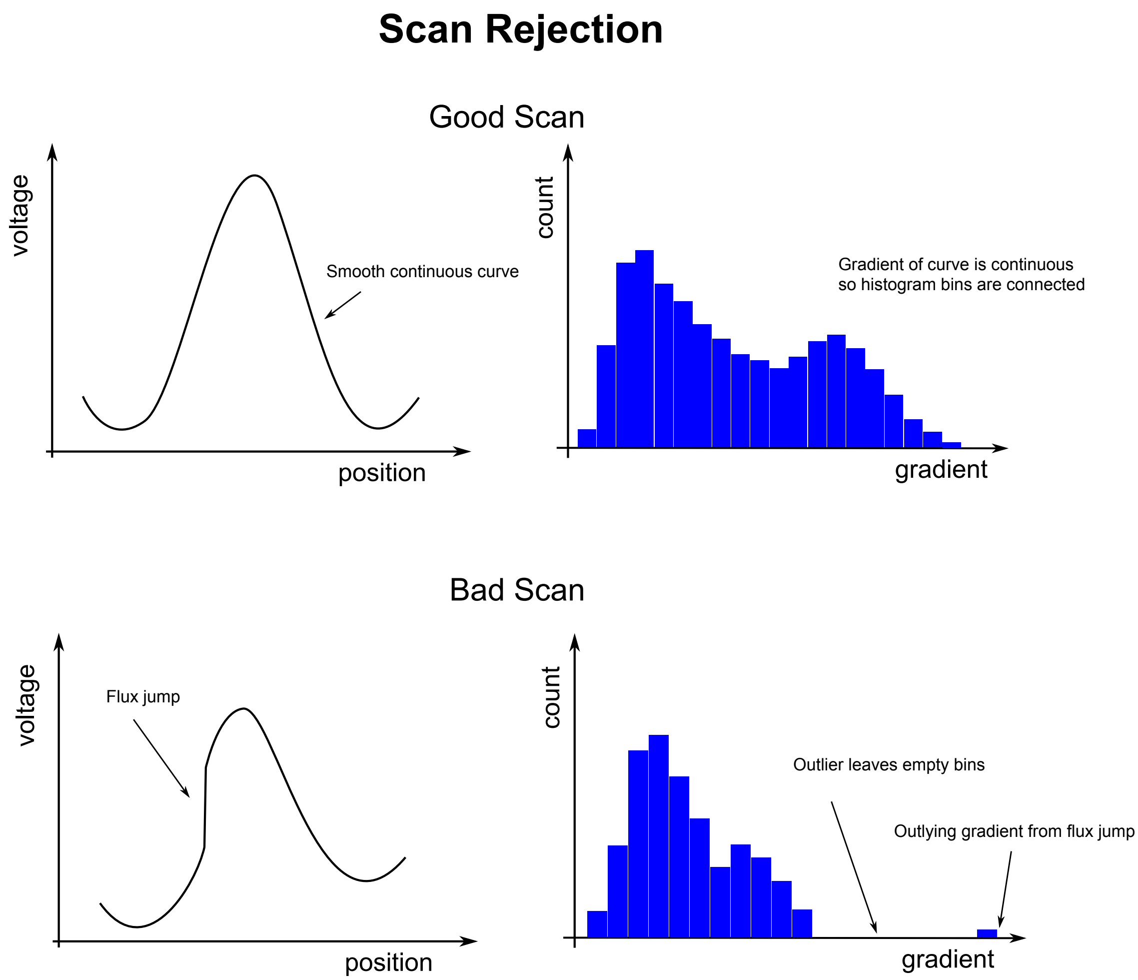
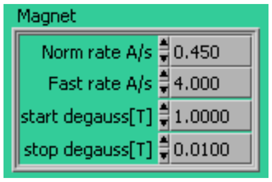


Figure 13.1: *The binning process for detecting bad scans. The gradient is taken at a large number of points on the scan and the range of gradients is divided into a given number of bins. The number of points whose gradient falls into each bin is counted, producing a histogram. If the scan is smooth then the bins will be connected. If there is a flux jump, which shows as a discontinuity in the voltage, then there will be one point with a much larger gradient than the rest. This gives a number of empty bins. The number of bins and the acceptable percentage of empty bins are user defined.*

Magnet



It is strongly recommended that the Magnet and field measurement settings (see below) be left at their factory values unless otherwise advised by a Cryogenic engineer. Unsuitable settings can cause serious damage to the system.

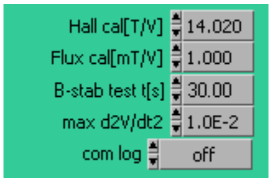
Norm rate A/s This defines the rate of change of current in the magnet, in Amps per second, used during a field sweep when the superconducting persistant mode switch heater is on.

Fast rate A/s If the magnet is set to a particular field and put into persistant mode, the current leads are ramped down with the rate defined by this control. The same rate is also used during the degaussing procedure.

start degauss[T] In order to reduce the remnant field a degaussing procedure is available in the magnet control window. This option can also be performed within a sequence. The **Start Degauss** control defines the magnitude of the first magnetic field set during this procedure.

stop degauss[T] During the degaussing procedure the field is successively reduced by 15% whilst alternating polarity. When the field becomes lower than the value defined by **stop degauss** the procedure is terminated.

Field Measurement



It is strongly recommended that the Field measurement and Magenet settings (see above) be left at their factory values unless otherwise advised by a Cryogenic engineer.

Hall cal[T/V] Calibration constant for the hall probe. Do not alter unless the probe is changed.

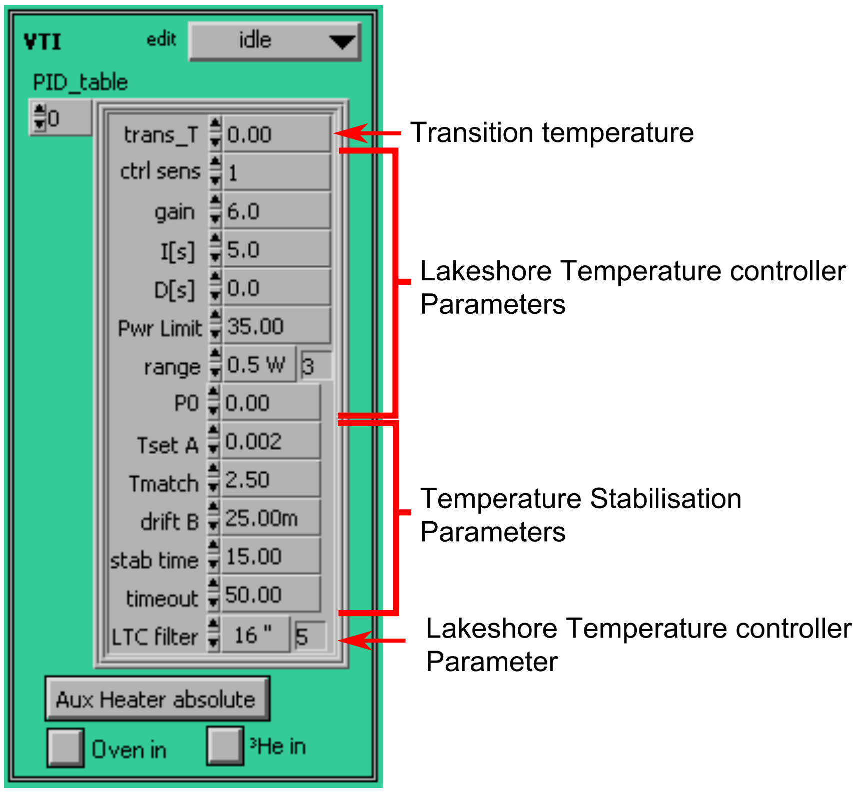
Flux cal[mT/V] Calibration constant for the fluxgate probe. Do not alter unless the probe is changed.

B-stab test t[s] This sets the time over which the field stability is measured. If the gradient of the field with time is less than **max d2V/dt2** for the duration of **B-stab test t** then the field is deemed stable.

max d2V/dt2 This sets the target gradient for the field stability test (see **B-stab test t**)

com log setting **com log** to ON writes a record of all communications between the software and the VTI and magnet power supply. Normally this is only used for diagnostic purposes.

VTI



PID table This table contains the parameters that the software and Lakeshore temperature controller use for setting and stabilising temperatures. PID stands for **Proportional - Integral - Derivative**, each representing a parameter in the control equation used by the Lakeshore temperature controller to set and maintain temperature.

$$Heater\,Output = P\left[e + I\int(e)dt + D\frac{de}{dt}\right] \quad \textbf{where}\,(\mathbf{e}) = \textbf{set}\,\mathbf{T} - \mathbf{T} \tag{13.1}$$

The parameters in this table can be set for different temperature ranges for optimisation. The preset factory values have been set to give good temperature control accross the full temperature range. The user can optimise the settings for the particular temperature range that is of interest. The table displays one 'entry' at a time, each showing the parameters to use in a given temperature range. The parameter **trans_T** stands for "transition temperature" and sets the temperature at which the settings in each entry become active.

Some parameters vary continuously with temperature. For these parameters a value is given for each transition temperature and the value is then determined by linear interplation between these set points. This is shown in figure 13.2. Other parameters can take discrete values, like **range** which tells the Lakeshore temperature controller which power range to use. For parameters that take discrete values the transition temperature defines the *upper* temperature of the range the value is used for. For example to use the 5W range between

Trans_T (K)	0	10	30	100	120	190	300
I (s)	1	105	3	8	18	24	25
range (W)	0.5	0.5	5	5	50	50	50

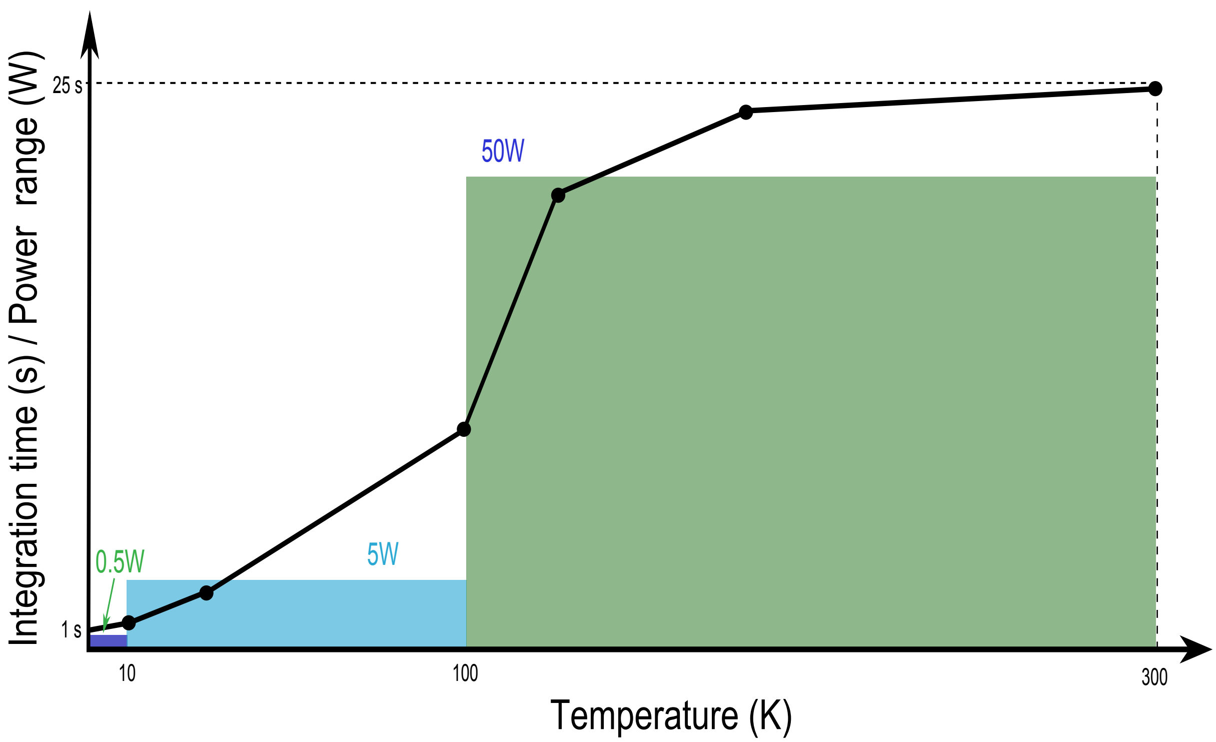


Figure 13.2: *Continuous and discrete variables in PID parameters. Continuous variables are determined by linear interpolation from a set of points given by the PID table entries. Discrete variables are defined at the upper bound of the temperature range they apply to. In this example the 5W power range is used between 10K and 60K. The PID entry that contains the 5W range is at 60K. 5W is then used for temperatures below 60K until the next entry with a different range setting which here is at 10K. The 10K entry sets the range at 500mW which is then used from 10K downwards.*

10K and 100K there should be a PID table entry with **trans_T** set to 100K and **range** set to 5W. The 5W range is then used for all temperatures from 100K down until the next PID entry temperature. This is also shown in figure 13.2.

edit This drop down menu contains controls for editing the PID table. The scroll to the left of the PID table is used to navigate through the entries.

- **idle** no action. The **edit** control returns to this state after performing an action.
- **insert entry** One entry is displayed at a time. **Insert** creates a duplicate copy of the currently displayed entry immdediately after the current entry.
- **delete entry** Deletes the currently displayed entry
- **clear table** It, um...well, it .. clears the table

Lakeshore temperature controller parameters

These parameters are passed to the Lakeshore temperature controller. For more details of their meaning see the Lakeshore manual supplied with the system.

ctrl sens The lakeshore 340 temperature controller has two inputs on its rear panel for reading temperature from sensors A and B. This parameter tells the controller which sensor to use when setting a temperature.

gain This is the P or **Proportional** part of PID. This is the constant of proportionality P in the control equation used by the the Lakeshore 340. This must be greater than zero for the control loop to work. P has a range of 0 to 1000 with a resolution of 0.1.

I[s] Integration time, in seconds. The I or **Integral** part of PID. The control equation has a term containing an integral of the difference between set point and measured temperature over this integration time.

D[s] This is the D or **Derivative** part of PID. The control equation contains a term that is proportional to the rate of change of the difference between set point and measured temperature. D is the constant of proportionality.

Pwr Limit Power limit. The maximum power that can be applied. This differs from **range** because range determines the resolution of the power that can be applied[17] whereas the power limit allows you to set a maximum power that can be applied.

range This is the range setting for the power to be applied. The range determines the resolution with which power can be applied. See footnote 17.

P0 This controls the "manual output" setting of the Lakeshore temperature controller. Manual output is a constant output power that is added to the PID output from equation 13.1, making the PID output effectively operate about **P0**. The manual output is defined as a percentage of maximum power in the currently selected range. Values are from 0% to 100% with a resolution of 0.01%.

[17] The power from the temperature controller has a finite resolution (see Lakeshore manual) which means on a high range such as 50W the smallest change in power that can be made is larger than the whole range of powers available at the 500mW range.

Temperature stabilisation parameters

These parameters are used by the software when testing for temperature stability. When a temperature is set, either manually or by a sequnce, the software requires three conditions to be met before the temperature is said to be stable. These are shown in figure 13.3.

1. $|T_{SET} - T_A| < Tset\,A$. The temperature from sensor A must be within a given window around the set temperature.
2. $|T_B - T_A| < Tmatch$. The temperatures from sensors A and B must be within **Tmatch** of each other.
3. $\frac{dT_B}{dt} < \frac{drift\,B}{stab\,time}$ for the duration of **stab time**. When the inequality is met it is monitored for a period given by stab time. If the inequality is met for the duration of that time then the temperature is stable. If not the test starts again the next time the inequality is met.

Tset A This controls the first condition that must be satisfied when setting and stabilising a temperature, that the temperature from sensor A must be within a given windown around the set temperature, i.e. $|T_{SET} - T_A| < Tset\,A$. (see figure 13.3)

Tmatch This controls the second condition that must be satisfied when setting and stabilising a temperature, that the difference in temperature from sensors A and B must be smaller than **Tmatch**, i.e. $|T_B - T_A| < Tmatch$.

drift B This, along with **stab time** controls the third and final condition that must be satisfied when setting and stabilising a temperature, that the rate of change of temperature from sensor B with time must be smaller than a given value for a given period of time. The condition to be met is $\frac{dT_B}{dt} < \frac{drift\,B}{stab\,time}$ for the duration of **stab time**. **drift B** is the amount that T_B can vary during **stab time** and still satisfy the condition. (see below)

stab time stands for *stabilisation time* and is used along with **drift B** for controling the final condition that must be satisfied for setting and stabilising a temperature, see **drift B**.

timeout If the stabilisation conditions are not met within a time defined by **timeout** then the software assumes stabilisation and continues with the sequence. This prevents the sequence from hanging if the stabilisation conditions are set to be too strict.

drift B/stab time

These two parameters warrant a little further explanation by way of an example. Say we wish the third stabilisation condition to be for the gradient of T_B to be smaller than $1\,mKs^{-1}$ for $60\,s$. The stabilisation time, **stab time**, is straightforward, it is $60s$ from the definition of **stab time** above. **drift B** is the the amount T_B can vary during the stabilisation time and still meet the criteria. This can be seen below.

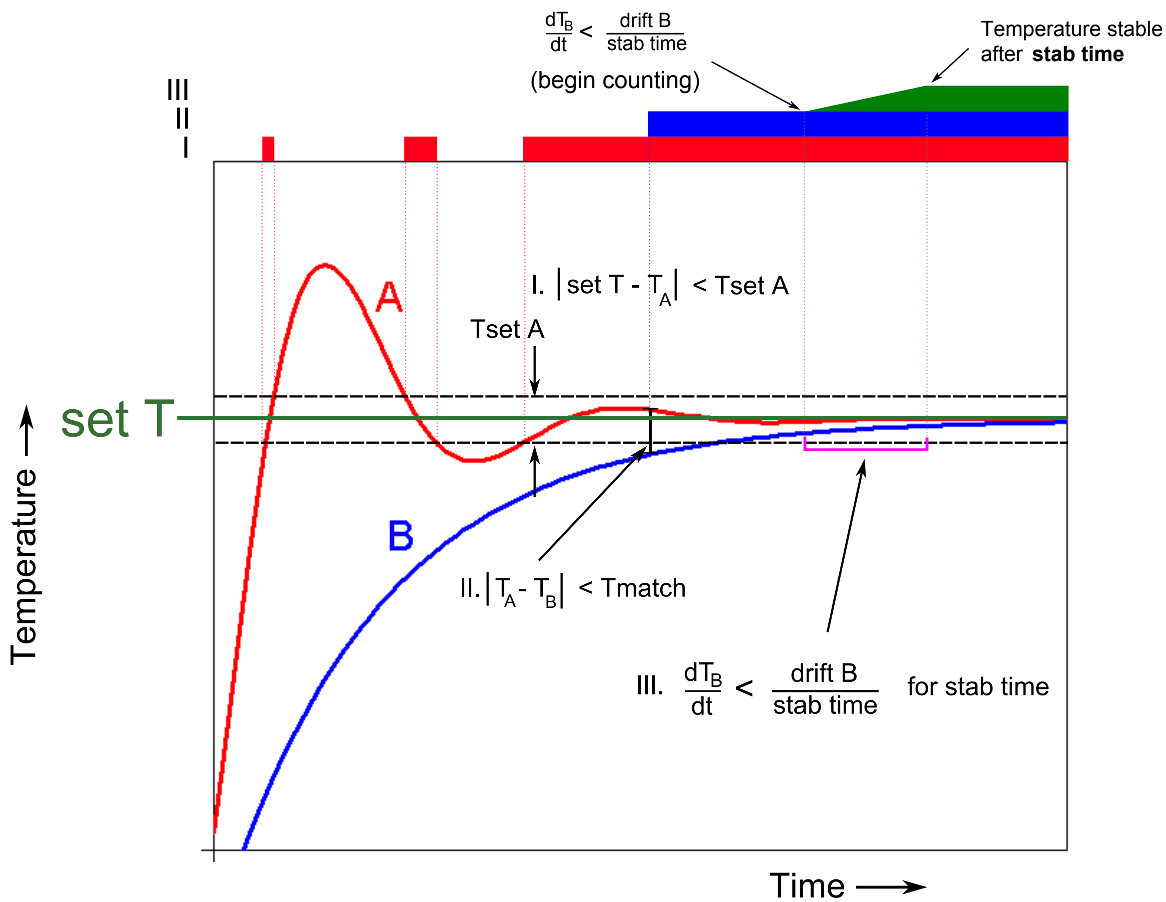
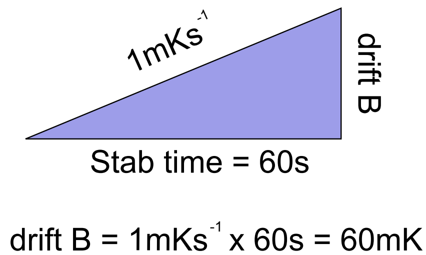


Figure 13.3: *Stabilisation conditions for setting temperature. These three conditions are tested in the order shown. When all three conditions are met the temperature is deemed to be stable. If all three conditions are not met within a given time (**timeout**) then the software assumes the temperature is stable and continues with the measurement. This prevents overly strict conditions causing the sequence to hang. Care should be taken to set these parameters carefully if they are changed from the factory settings.*

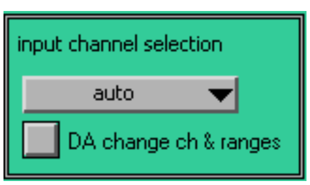


The maximum gradient permissible is our figure of $1mKs^{-1}$. This gradient is **drift B/stab time**, so as long as T_B doesn't vary by more than **drift B**, during the stabilisation time the gradient condition is met. In this way **drift B** and **stab time** are used to define the gradient condition on T_B.

If the oven, or ^{3}He insert are present then the software needs to know about it. When these buttons are pressed they turn red, indicating that the oven or ^{3}He insert are in.

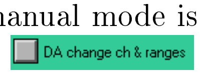
Aux Heater absolute/relative Obsolete. Not used by the software, will be removed in future software updates.

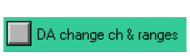
Input Channel Selection



The channel selection drop down menu sets the assignment of the input channels to the sample movement. There are two options:

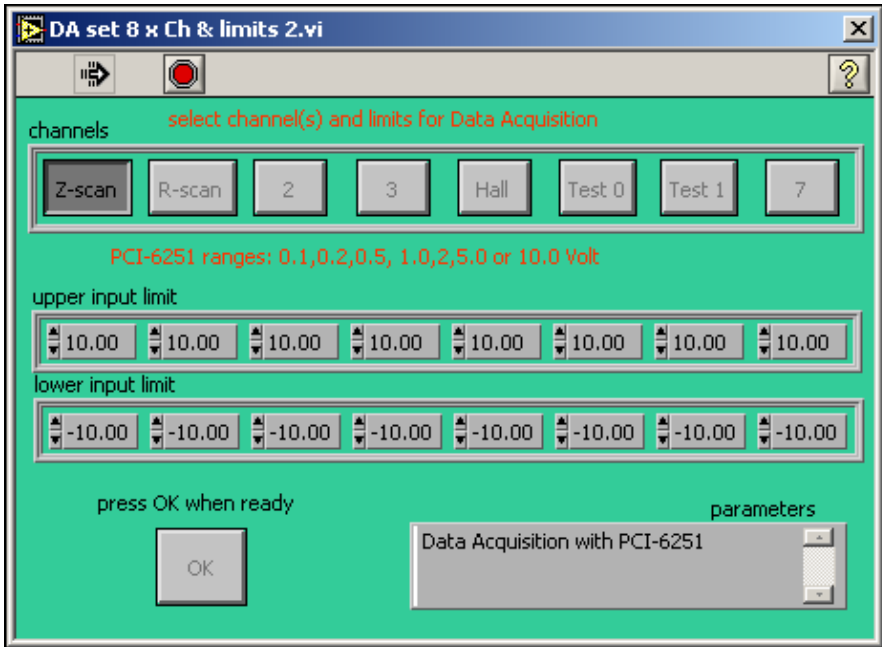
auto A Z-scan will cause the data acquisition to store the signal of the axial pickup system. A rotation measurement will collect data from the transverse pickup system.

manual To give more flexibility the manual mode is an alternative to **auto**. In this mode all input channels selected via the  button are stored in the data file. The file can be analysed with the 'view data file' option after a completed sequence.



This brings up the **DA set 8 x Ch & limits** window for configuring which channels are read by the data acquisition system.

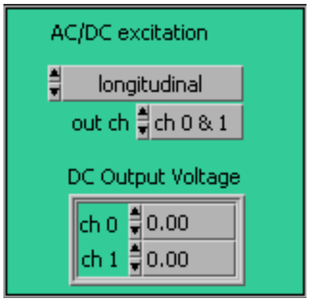
DA set 8 x Ch & limits



This window allows configuration of the number of channels read by the data acquisition system. Normally only one channel is read i.e. the Z-scan output (axial pickup coil), however if a Hall probe or fluxgate probe is used the probe output may be read instead of or in addition to the SQUID output. The additional channels are accessed by clicking the channel buttons which toggle between on and off states. The voltage limit for each channel may be defined between -10V and +10V. This is the default setting for all channels and is equivalent to the full scale voltage range available from the SQUID electronics. The voltage range set for a fluxgate or Hall probe is at the users discretion and will depend on the field range used for a particular measurement. The voltage limits may be set for a purely positive or negative voltage swing which may be useful if measurements are made when the magnetic field is not swept through zero.

As supplied, the data acquisition card is hard wired to read the two channels of the two different pickup systems (axial and, if purchased, transverse) on channels 0 and 1 and the Hall probe or fluxgate on channel 4. The two test inputs are connected to the outputs of the sources of the DLA (Digital Lock-in Amplifier). If more than one channel is selected it depends on the channel selection mode (auto or manual) whether the data is recorded simultaneously.

AC/DC Excitation



This box allows a DC excitation to be applied to the AC coils. It utilises two **D**igital **A**nalogue **C**onverters in the **D**igital **L**ock in **A**mplifier and a voltage controlled current source to apply small DC fields of up to a few Gauss. The maximum voltage is 10V which gives 330 mA and the exact field that this will produce is dependent on the specifics of each unit. The field profile of each coil set is provided with the system. You can also refer to appendix **??** on how to measure the field profiles. The DACs have 16-bit resolution which means that the field can be altered in very small steps. The excitation can be applied to the main **longitudinal** or **transverse** coils or to the balance coil.

- *N.b. This box is the only way to apply a DC transverse field.*

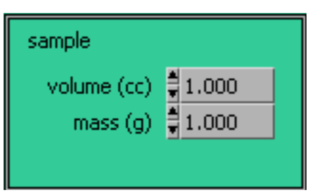
The choice of which coils to excite is made with the **longitudinal/transverse** and **out ch** buttons.

channel 0 Excites the main longitudinal or transverse AC coil depending on the direction selected.

channel 1 Excites the balance coil. This is independent of the direction selected.

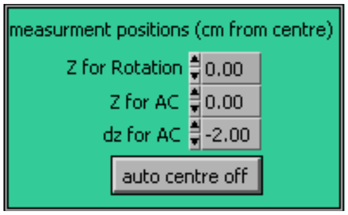
The output voltage for each channel is set in the **DC Output Voltage** box. Integer numbers of volts can be set with the up/down buttons, other values can be set by typing the value into the relevant field.

Sample



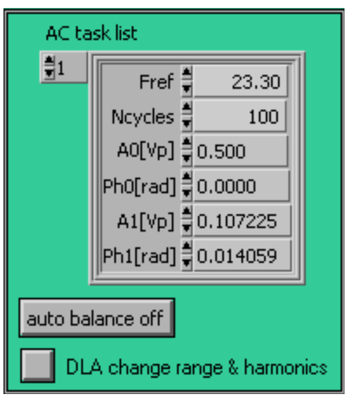
When launching a sequence you have the option of entering sample volume and mass for use when calculating various properties such as volume or mass susceptibility. If you are performing many measurements on the same sample you may wish to change the default values to save having to enter them every time. To do so enter the new values here.

Measurement positions



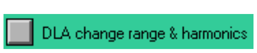
These controls define the sample position for AC and rotation measurements. **Z for Rotation** and **Z for AC** are absolute positions measured in centimeters from the centre of the longitudinal pick up coils. Positive is above centre, negative is below. **dz for AC** is a reletive displacement from the centre defined by **Z for AC** and defines how far the sample is moved out of the pick up coils for AC balancing before each scan (see 9). **auto centre ON/OFF** toggles the DC measurement auto centre feature on and off.

AC task list



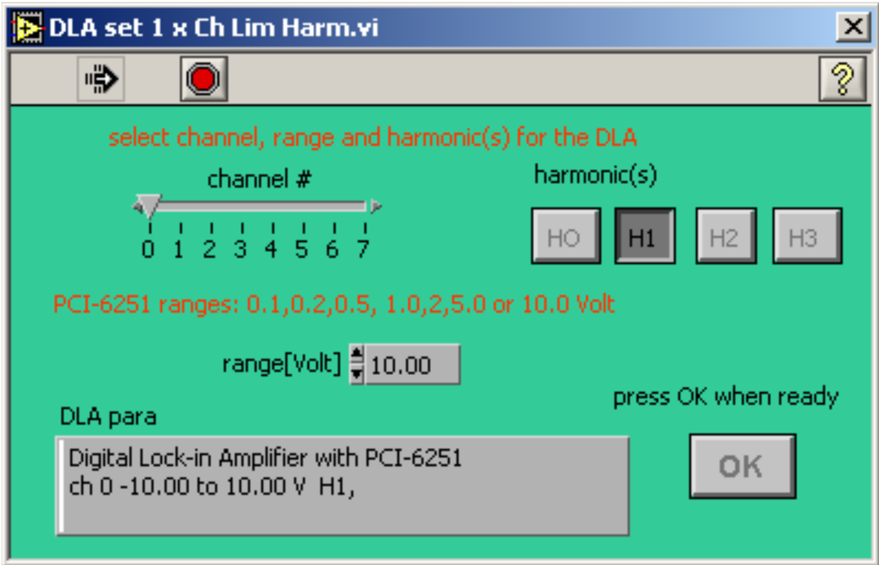
This box is used to create a list of measurements to take at each B,T point during an AC measurement. You can specify a sequence of AC measurements to take with different frequency, number of cycles, amplitudes and phases. See chapter 9 for details on performing AC measurements. Like the PID table, one 'entry' is displayed at a time. To view different entries use the up/down buttons next to the entry counter. Scrolling past the last entry adds a new entry which is greyed out. Editing one of the parameters and clicking away again activates the entry and it becomes not greyed out. To add an entry at a place other than the end, right click on the table and click "insert element before".

auto balance optimises the AC compensation during a measurement.



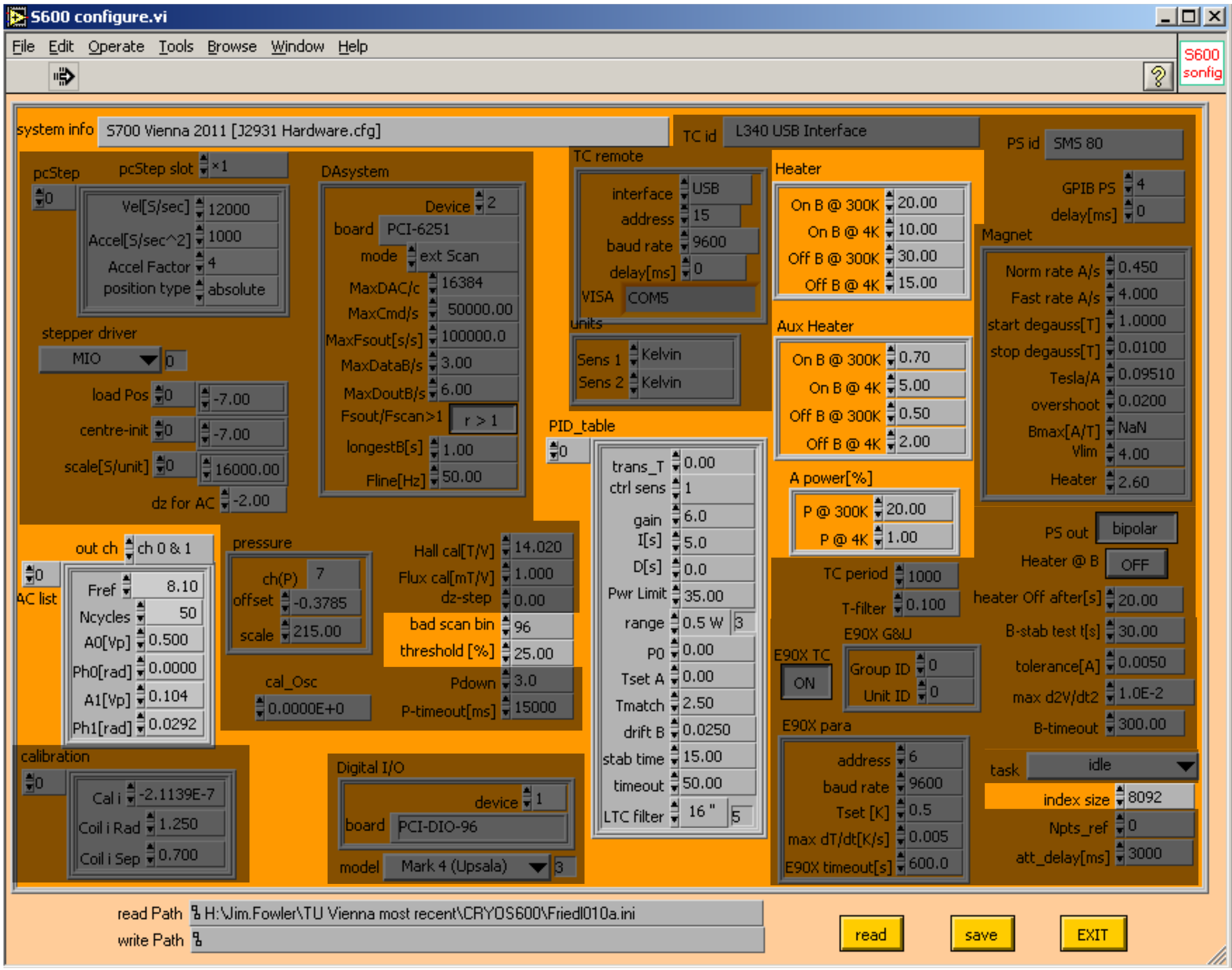
opens the **DLA st 1 x Ch Lim Harm** window for changing the channel, voltage limits and harmonics settings of the digital lock-in amplifier for AC measurements.

DLA set 1 x Ch Lim Harm



This window is used to change the channel, voltage limits and harmonics settings for the **D**igital **L**ock-in **A**mplifier used for AC measurements. As well as being able to set a list of frequencies with amplitude and phase for AC measurements (above) it is also possible to set the output channel(s) used to synthesize the AC reference signal, and which harmonics the lock-in amplifier uses when locking. Please refer to the user manual for the DLA for more details.

13.2 System Configuration



Altering shaded settings can cause damage to the system. Do not alter any settings shaded here without consulting Cryogenic.

When the software is loaded, many global parameters required for operation of the system are loaded. These parameters can be viewed in the System configuration window. Most of the parameters in this window however should not be changed by the user and are only visible for reference and for system maintainance by a cryogenic engineer, these are shaded in the picture. They relate to hardware and calibration settings and making changes without consulting Cryogenic can lead at best to loss of system performance and at worst serious harm to the system.

Parameters are loaded from two files. “Hardware.cfg” contains fundamental hardware parameters that are not visible in this window. The settings shown in this window are loaded from a ‘.ini’ file. The default file that contains the factory settings is “Jxxxx.ini” where xxxx is the Cryogenic “job number” for the system. This can be found on the cover of this manual. Changes can be made to some settings to optimise the system for your particular requirements and these changes can be saved as a **.ini** file for future use. To save settings click 

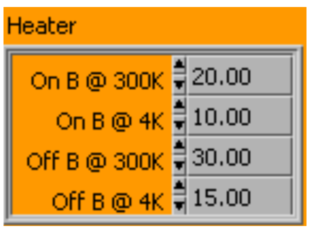
, to load a different .ini file click 

. In both cases a standard dialogue box will appear where you can choose the location to save to or to read from respectively.

AC list, **bad scan bin**, **threshold[%]** and **PID table** are as described in section 13.1, configure data acquisition, the difference being that you can save your settings with the system configure window. Configure data acquisition can be used to make non-permanent changes for one time use or for testing parameter values. The next time the software is loaded however the chosen .ini file will overwrite any changes made in the configure data acquisition window.

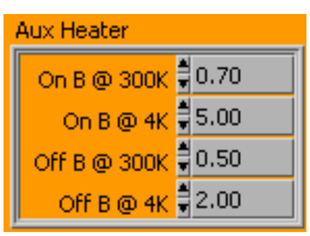
index size this controls the maximum number of data points in a data file. Values must be of the form 2^n where n is an integer.

Heater



These are the parameters that control the behaviour of the heater during cooling. To save time when cooling to a set temperature, if the temperature at sensor B is far enough above the set point then the heater is turned off. The temperature at sensor A will then fall towards base temperature, increasing the difference between A and B and speeding up the rate of cooling at B. The heater must be turned on again before B reaches the set point or all of the time saved and more will be wasted while A warms up as both sensors must be close enough to each other to stabilise the temperature (section 13.1). This is shown in figure 13.4. The magintudes of $|set\,T - T_B|$ that will cause the heater to turn off and on are temperature dependent. Two values are set at 300K and 4K, for both **on** and **off**, and linear interpolation is used to calculate all values in between.

Aux Heater



As described in section 3.1.4 the sample chamber is fitted with an auxilliary heater because it is made of poorly conducting phosphor bronze and heating from the gas flow alone would take a long time. The auxilliary heater makes warming to a set temperature much faster. The parameters in this box control when the auxilliary heater is switched on and off. When the temperature at sensor B is far below the set temperature the aux heater is switched on.

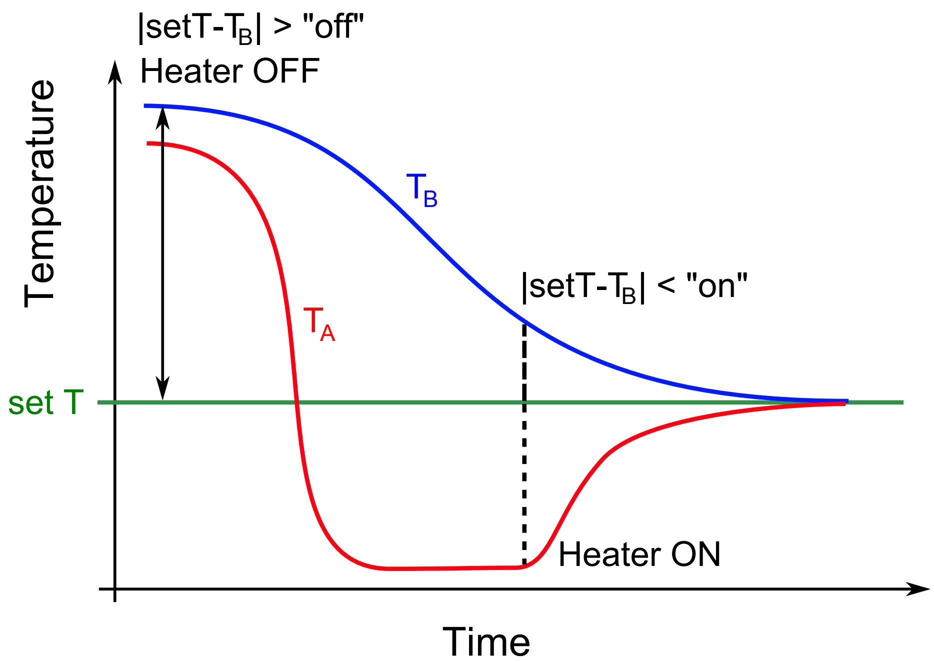
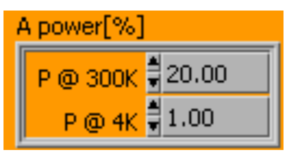


Figure 13.4

When the temperature at B gets close enough to the set point the aux heater is turned off to prevent overshoot.

A power[%]



This controls the maximum power put into the auxilliary heater. Two values are set at 300K and 4K and values in between are calculated using an Einstein function that closely matches the temperature dependance of the heat capacity of the sample chamber.

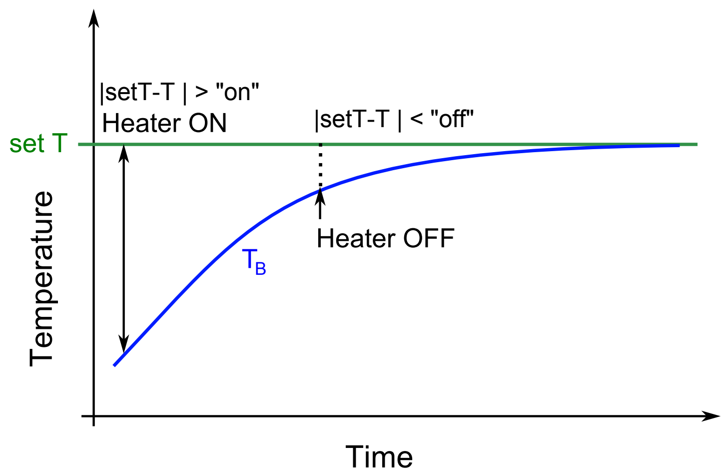


Figure 13.5: *The behaviour of the aux heater when warming up to a set temperature. The heater is on while T_B is far enough below the set point but is switched off when it gets close enough to prevent overshoot. The values of $|setT - T_B|$ for switching the heater on and off are temperature dependent. Two values are set in the config window for 4K and 300K for* ***on*** *and* ***off****. Linear interpolation is used to calculate values in between.*

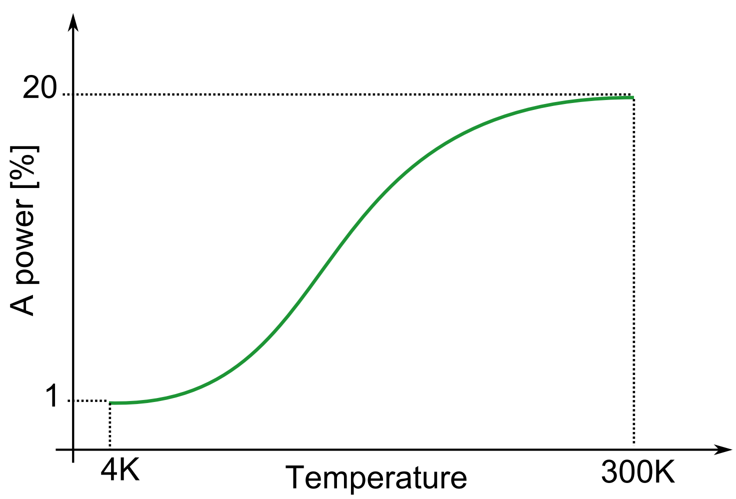
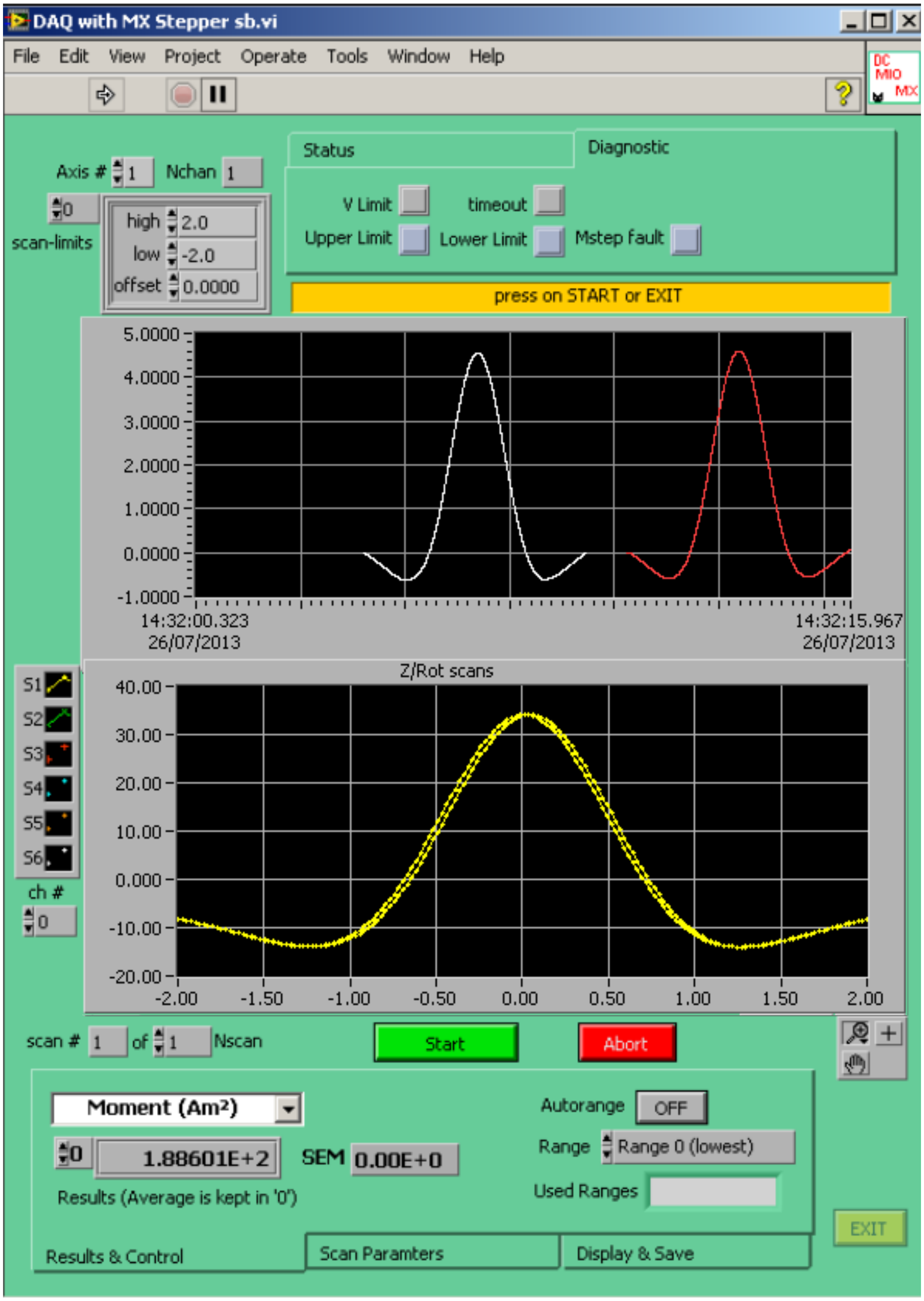


Figure 13.6: ***A power[%]*** *is interpolated between values set at 4K and 300K using an einstein function that resembles the heat capacity of the sample chamber.*

13.3 DC Measurements - reference

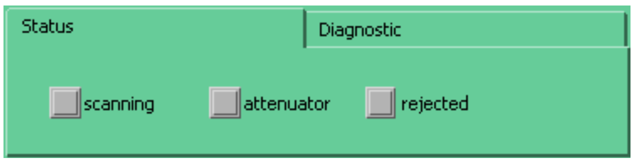
Single DC measurements are made with the **DAQ with MX stepper sb** window. This was covered briefly in section 4.7 when discussing centering a sample and in more detail in section 7. This section is a reference for each of the controls and displays in the window.



DC measurements include longitudinal (Z-scan) and rotational (R-scan) measurements, DC (direct current) refers to the current generating the applied magnetic field. The field applied in DC measurements is constant during a scan.

13.3.1 Indicators

There are three indicator lights in the **Status** tab at the top of the window.

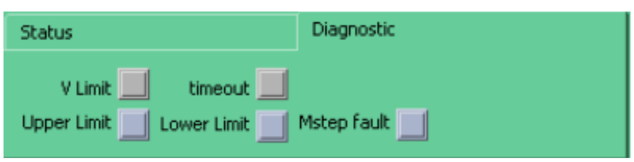


scanning Indicates that a scan is in progress

attenuator Indicates that the attenuator is active. The attenuator is used to decrease the sensitivity of the SQUID and prevent saturation when measuring large moments, giving you the extended ranges 3 and 4.

rejected Indicates that the previous scan was rejected and repeated either because of the gradient binning process described in 13.1 or because the upper or lower voltage limit was reached.

And five more in the **Diagnostic** tab.



V Limit Indicates that the voltage reached the upper or lower limits for the current range. When this happens the scan is repeated with the next highest or lowest range depending on the limit reached

timeout Indicates the scan took too long and was aborted. If the scan takes 20% longer than expected from the given limits then there must be a problem with the stepper motor and the scan is aborted

Mstep fault Indicates there was a problem with the number of steps the stepper motor took, meaning the motor probably stalled during the scan.

There are also several numerical indicators. These display information and are not controls.

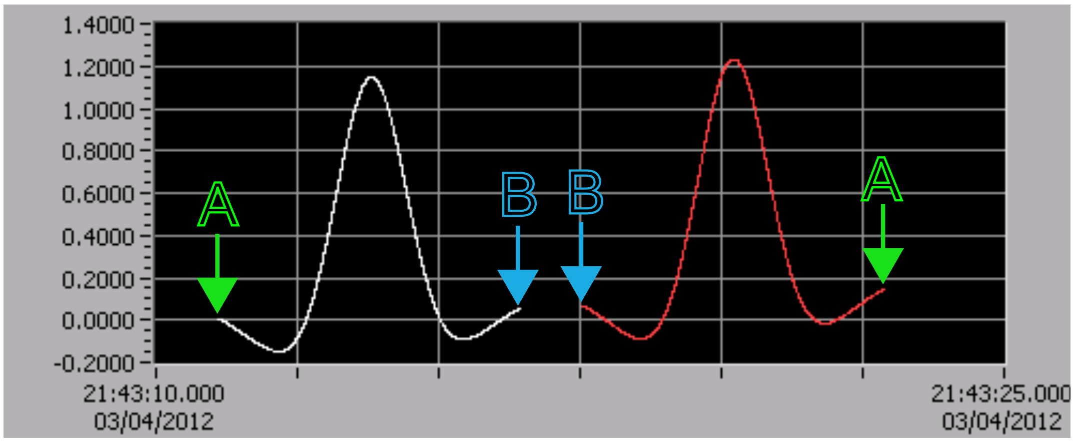
Nchan Displays the number of channels being recorded. Usually $Nchan = 1$ unless channel selection is set to manual mode (see 13.1).

scan # This displays the number of the scan currently being performed.

Ranges Not to be confused with **Range**, this is simply a display box for diagnostic purposes that shows which range was used for each scan if $Nscan > 1$. Once **autorange** has found the right range for the first scan, each subsequent scan should use the same range, as the sample is the same. If this is not the case then something is wrong and should be investigated.

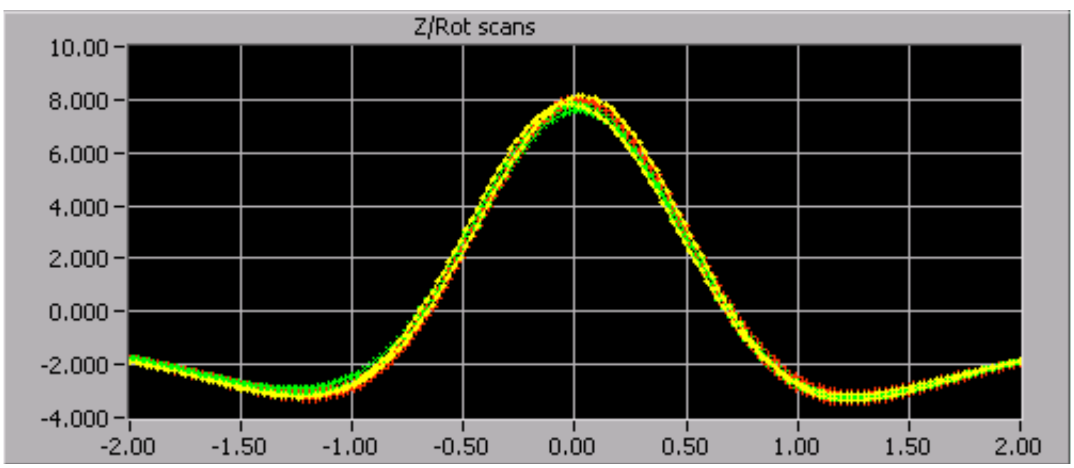
Graphs

The upper graph displays the voltage response of the SQUID as a function of time.



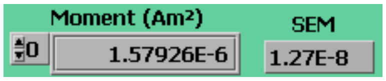
This graph is produced as the sample moves up and then down through the pickup coils in a Zscan measurement. Points marked A are equivalent in position as both represent the bottom of the scan. Similarly points B both represent the top of the scan.

The lower graph displays the result of up and down scans as a function of position, taking into account an offset for any drift in background flux. As a function of position, the red 'down' curve from the upper graph is folded back on the white 'up' curve.



 Standard Labview graph controls for zooming, panning, and configuring the displayed graphs. See the Labview help files for information on these controls.

Result



The result of the scan or scans is displayed in this box. The index counter to the left determines which result to display. **0** is the arithmetic mean of the results of each individual scan. **1,2,3...** are the individual results of each scan.

SEM Standard Error Mean, defined as $SEM = \frac{\sigma}{\sqrt{n}}$ where σ is the standard deviation of the results.

There are several options for the units to use when displaying the result. These are chosen by clicking on the result label **Moment (Am²)**.

Moment (Am²) Displays the calculated magnetic moment in Am^2

Moment (Am²/kg) Displays the calculated specific magnetic moment in Am^2/kg. The sample mass must be entered in the **Configure Data Acquisition** window (13.1).

Magnetisation (A/m) Displays the calculated magnetisation in A/m. The sample volume must be entered in the **Configure Data Acquisition** window (13.1).

- $10^{-3}\,Am^2 = 1\,emu$
- $1\,Am^2/kg = 1\,emu/g$

13.3.2 Controls

Axis # Selects which axis to use for sample movement during the scan.

#1 longitudinal - sample is moved vertically up and down

#2 rotational - sample is positioned vertically in the centre of the pick up coils (or at a non central position defined in the **Configure Data Acquisition** window), and rotated about the vertical axis.

Scan limits Set the range of a longitudinal scan.

high the position of the top of the scan relative to the centre, in centimeters.

low the position of the bottom of the scan relative to the centre, in centimeters.

offset artificially adjust the centre position. If the sample is not exactly at the centre of the pickup coils then the offset can be used to adjust for this when making a scan (see 4.7). If the offset is used then the **high/low** positions are relative to this position.

Nscan The number of scans to perform.

Starts the scan

Stops the scan when pressed. The stepper motor resets to its initial position.

The controls in the lower panel are grouped into three tabs

Results & Control

Range Selects the range to use. 0 is the most sensitive range, 4 is the least sensitive. In the more sensitive ranges a large moment would saturate the signal, giving a flat topped scan that can't be fitted as a dipole. This is when you need a higher range. Ranges 0,1 and 2 are gain settings on the SQUID amplifier and are standard in both pick up coil systems (longitudinal and transverse). Ranges 3 and 4 come from an attenuator that decreases the sensitivity further, which is fitted as standard to the longitudinal pick up system but not the transverse.

Autorange With autorange ON, if at any position the voltage reaches the upper or lower limits defined for each range the scan is rejected and repeated with the next highest or lowest range.

Scan Parameters

Toggles auto-centering on and off. Auto-centering finds the centre of the sample signal by fitting a dipole response and adjusts the position offset accordingly to bring the sample to the centre of the pickup coils.

When activated the button turns black. This defines whether the PFL100 (SQUID amplifier) is reset before each scan or only before the first.

black reset activated. PFL100 reset before each scan

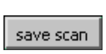
green reset deactivated. PFL100 reset before first scan only

Enables or disables the automatic scan recection based on gradient binning described in section 13.1.

Order This is the order of differentiation, $\frac{dV}{dx}$, $\frac{d^2V}{dx^2}$, $\frac{d^3V}{dx^3}$ etc, used in the mathematical expansion and fitting of the scan to obtain a result. See 8.

Display & Save

Opens the **large display + fit v2** window for detailed analysis of the scans. See 8 for details. Does not zoom on displayed graph, for this use the graph controls (see below).



Saves a tab delimited text file containing the data in the lower graph, voltage (with the applied scale) against position. A standard dialogue box appears for you to choose a file name and a location.

When set to ON, save data writes the data to a file in the same way as in an automatic sequence. This can be useful for manually taking data at a few B,T points without writing a sequence, or storing scans for diagnostic purposes. A standard dialogue box appears where you can choose a file name and location.

And finally...

Closes the window.

14 Trouble Shooting Guide

The following trouble shooting guide outlines some problems that can be experienced by the user, together with their possible causes and remedies.

14.1 Inefficient Transfer

System not thoroughly pre cooled

The system requires at least 8, but preferably 12 hours to pre cool properly.

Transfer syphon "soft"

If the syphon becomes frosted during the transfer it needs re-evacuation

Transfer too fast or intermittent

If the transfer is performed at faster than the recommended transfer rates it will be less efficient in terms of helium usage. Use of a gas meter to monitor the transfer rate can be beneficial.

Syphon not located in the mating cup.

For the initial cool down of the system below pre cool temperature ensure that end of the syphon locates properly in the syphon mating cone mounted on the insert. This ensures the full cooling enthalpy of the gas is used to cool the system. To be certain that the syphon is correctly located, it may be helpful to mark the syphon with tape when fully inserted.

Liquid Nitrogen left in from the helium reservoir

If the liquid helium used in the pre cool is not fully removed from the helium reservoir the temperature drop of the reservoir tail thermometer will be slow and the onset of a helium level may be severely delayed even having achieved close to 4.2K. Refer to 4.2.4 and remove all traces of remaining liquid nitrogen.

14.2 High System Boil-off

Level gauge switched on

The power supply limits the pulsing of the level gauge to once every 15 minutes. If the power supply is switched off, the level gauge will pulse at the rate determined by the MAX or MIN setting on the device, which leads to an increased helium boil-off.

Leak in the vacuum spaces.

Investigate using a mass-spectrometer leak detector.

14.3 Higher than normal noise level in SQUID output

External magnetic noise

Trace and eliminate

External vibrational noise

Trace and eliminate

Poorly tuned SQUID

Refer to 12 for details of SQUID tuning

14.4 Drift in SQUID output

Helium level low

Check and refill if necessary

Pressure fluctuations in the cryostat

Pressure fluctuations which can be generated by some recovery line systems can cause drift in the SQUID output.

High magnet ramp rate

The faster the ramp rate used for a change in magnet field the longer it is usual for the magnet to drift before field stabilisation. This drift will be detected by the SQUID

14.5 SQUID losing lock - flux jumps

Level gauge pulsing

Switch off the level gauge when measuring.

Poorly tuned SQUID

Refer to 12 for details of SQUID tuning

Helium level low

Check and refill if necessary

External vibrational, magnetic or R.F. noise

Trace and eliminate

Syphon in the cryostat

Complete the transfer and remove

14.6 Contamination of the VTI

Sample detached from sample rod.

Warm the VTI to a 310K (note - it is best to do this using the Temperature Control option from the main menu of the software package, as this program will utilise the auxiliary heater to speed the warming of the VTI). Once at 310K close off the helium exhaust line ensuring the cold needle valve is open. This will produce a positive pressure in the cryostat reservoir which will generate a flow a helium gas up through the VTI. Switch off the pump using the Flow Control option from the main menu and open the gate valve at the top of the VTI. Using a rod or tube of small enough diameter to fit comfortably inside the VTI with a "ball" of double side adhesive tape attached firmly to one end, gently try retrieving the sample from the base of the VTI. Do not push the retrieval rod hard into the VTI, as this will only damage the sample capsule and cause further contamination. DO NOT FORGET TO RE-OPEN THE HELIUM EXHAUST LINE TO THE CRYOSTAT

The sample capsule is broken inside the VTI when using powdered sample.

If the sample capsule containing a powdered sample breaks inside the VTI then it will be necessary to clean the VTI thoroughly. A rotary vacuum pump with a thin hose attached can be used to suck the sample from the VTI. Having removed the majority of the sample use cotton wool wet in acetone and attached firmly to a retrieval rod to clean any remaining residue. After removal of the sample close the gate valve, start the pump and flush the VTI (still at 310K) with helium for 5 minutes before cooling the VTI to lower temperature.

14.7 VTI Blocked

If air is allowed into the cryostat, then there is the danger that the VTI will become blocked from the ingress of ice or frozen air. If the blockage is in the bore of the VTI (this may be seen/felt as a restriction in the bore) then it can usually be cleared by raising the temperature of the VTI to 310K using the temperature controller. This is best achieved using Temperature Control option available on the main menu which will utilise the auxiliary heater in warming the VTI. If the blockage is closer to the helium inlet of the VTI it may not be possible to unblock it by warming only the VTI. In these cases it may be necessary to let the helium level drop below the inlet and re-try the above procedure. If the blockage is still present it may be necessary to let the insert warm above 77K or even 273K to remove the blockage. The insert can be removed from the cryostat and warmed separately so the whole system does not need to be warmed up.

14.8 Software detects leak in the wiper seals or airlock

If the software detects a leak in the wiper seals, this may be due to the seals being damaged. This can occur if the sample rod is withdrawn too quickly when the rod is cold. This will cause the wiper seals to tear and give rise to a leak in the stem. If damage to the seals is suspected they should be inspected and if necessary replaced. If a leak into the airlock is suspected check the two concentric O-rings on the airlock seating plate and the base of the airlock. If necessary clean or replace if damaged.

14.9 Frequent bad scans during measurements.

Bad scans are usually caused by one of the following:

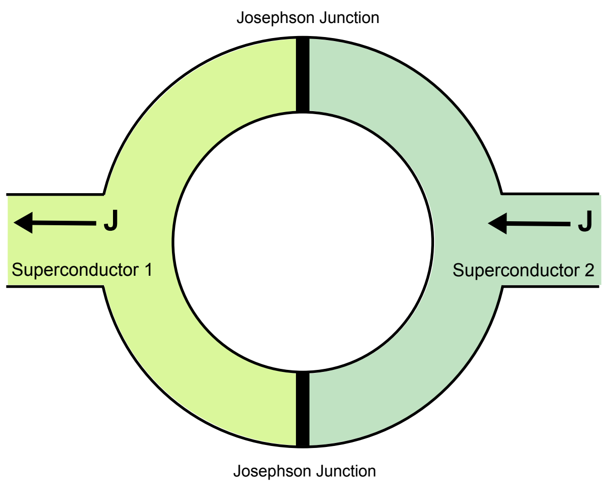
- The sample is not rigidly mounted and is moving in the magnetic field as it passes through the flux transformer.
- The sample tube has been contaminated with magnetic material which is being moved by the travel of the sample rod.
- The sample rod is hitting ice or solid air attached to wall of the sample column.
- An external source of RF noise or mechanical vibration is effecting the measurements.
- The sample rod is not moving smoothly through the airlock wiper seals.

14.10 Mu metal shield becomes magnetised

This is rare but in the event it does happen, the shield has been pre-fitted with a degaussing coil. Since the necessity to use this facility is so uncommon it is not supported as an automatic feature in the software, degaussing must be done manually via the 3-pin Fischer connector on the cryostat. It is strongly reccomended that you contact Cryogenic limited for assistance in degaussing the Mu metal shield.

A DC SQUID

There exist a number of textbooks that describe, in detail, the underlying physics behind the DC SQUID device. Here we shall give only an outline of the key concepts. SQUID stands for Superconducting QUantum Intererence Device. The fundamental phenomenon behind the device is the **Josephson Effect** which is where a supercurrent can penetrate through a thin insulating barrier between two superconductors, called a **Josephson Junction**. It is a **quantum tunnelling** effect involving the coherent tunnelling of cooper pairs through the barrier. The The DC SQUID has two such junctions in a superconducting ring.

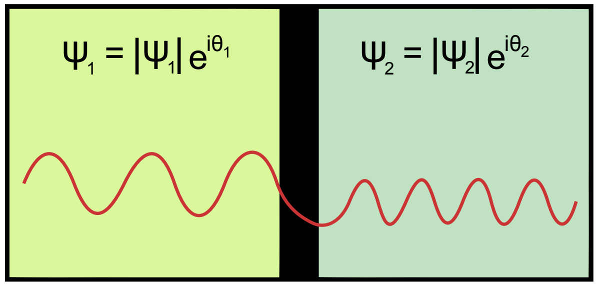


DC SQUID in three sentences

The supercurrent through each Josephson junction depends on the *phase difference* between the complex order parameters of the superconductors that form the two halves of the ring. The flux through the ring leads to assymetric phase differences across the two junctions which causes an interference pattern in the current across the device. A bias current is applied that is greater than the critical current of the junctions (the maximum current that can flow without dissipation, i.e. voltage) so that a Voltage appears accross the device which depends on the flux in the ring.

A.1 Josephson Effect

The current in a superconductor is carried by a Bose-Einstein-Condensate of Cooper pairs. The condensate groundstate is described by a wavefunction $\psi = |\psi|e^{i\theta}$. Tunnelling of Cooper pairs involves the penetration of the wavefunction through the barrier. The two groundstates must necessarily be coupled so that pairs can survive the tunnelling process. A collective groundstate exists between both superconductors which is in essence a superposition of their individual groundstates.



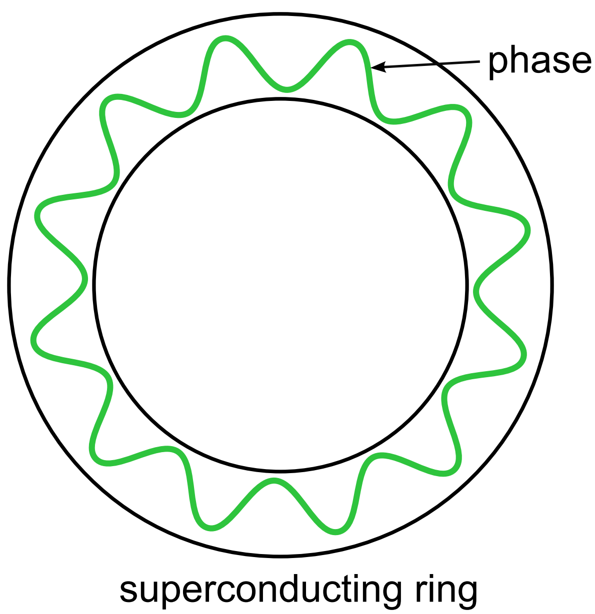
The tunnelling current is related to the difference in phase between the two groundstates

$$j = j_c sin(\theta_1 - \theta_2) \tag{A.1}$$

where j is the current, θ is the phase and j_c is the **critical current** of the junction. The critical current is the maximum current that can flow accross the junction without loss, i.e. without a Voltage drop.

A.2 Flux quantisation

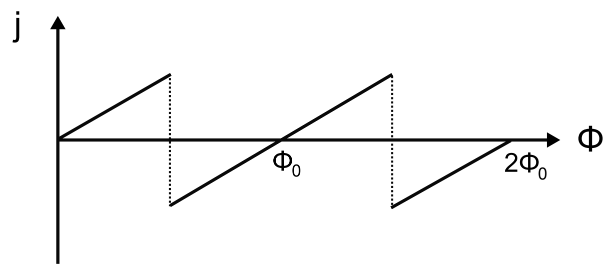
Consider a superconducting ring, with no Josephson junctions. The phase of the groundstate wavefunction must be continuous around the ring.



When there is flux passing through the ring the phase of the condensate is affected as it travels around the loop. This is related to the **Aharanov-Bohm** effect. The field in the superconductor is zero but the **Vector Potential** is non-zero. It is this vector potential, describing the flux in the ring, that affects the phase. Since the phase must be continuous, only certain amounts of flux can exist inside the ring. This is flux quantisation.

Note: Flux itself is not quantised, it is continuous. It is only the flux allowed to pass through a superconducting ring that is quantised.

In zero applied field there is no spontaneous supercurrent in the ring. As we apply flux however, a screening current appears in the superconductor that generates flux in the opposite direction to cancel that being applied. This current increases with the applied flux until we are applying half a flux quantum, $\Phi_0/2$. Now if we apply any more flux it costs the superconductor less energy to stop screening the applied flux and set up a supercurrent in the opposite direction to **add** flux to the ring to make it up to the allowed value of one flux quantum Φ_0. As we keep increasing flux it now takes less and less current to top up the flux to the allowed value until we reach Φ_0 at which point the supercurrent is zero. Now if we increase the applied flux still further the supercurrent once again begins to screen the applied flux keeping the flux in the ring to Φ_0 until we apply $\frac{3}{2}\Phi_0$ and the current changes direction again. This goes on and on, giving us a periodic function for the supercurrent as we vary the applied flux.



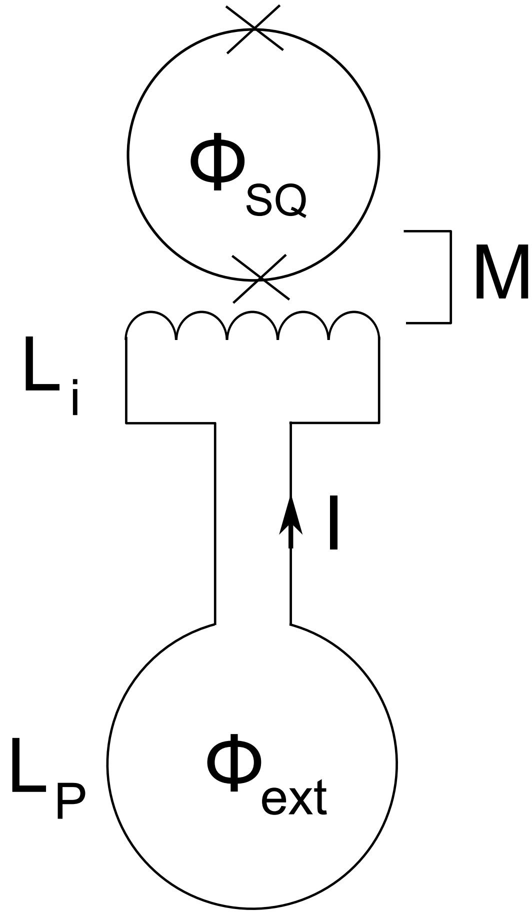


Figure B.1: *Superconducting pickup coil couples flux from the sample to the SQUID*

B DC Pickup coils

The SQUID does not see flux directly from the sample. A superconducting pickup coil system is used to couple the flux from the sample to the SQUID.

To maximise the efficiency of flux transfer to the SQUID we must consider the inductance of the circuit shown in figure B.1. The figure shows a superconducting pickup coil with inductance L_P and an input coil (also superconducting) with inductance L_i that couples to the SQUID. The total flux within the superconducting loop is a conserved quantity. The mutual inductance between the input coil and the SQUID is M. The pickup coil, with N turns, detects flux $N\Phi_{ext}$ from a sample which induces a current I in the superconducting loop. Since $\Phi = LI$, the induced current is inversely proportional to the inductance of the loop so to maximise the signal we could make both coils as low inductance as possible, right? Well, this reduces the mutual inductance between the input coil and the SQUID so clearly there needs to be some compromise. Let's now calculate the optimum balance of inductance

in the pickup system.
The two coils are two inductors in series, so the total inductance is

$$L_{total} = L_i + L_P \tag{B.1}$$

The flux Φ_{ext} coming from the sample induces a current I in the loop that satisfies

$$N\Phi_{ext} = (L_i + L_P)I \tag{B.2}$$

We can also relate the current to the flux in the SQUID

$$\Phi_{SQ} = MI \tag{B.3}$$

which, rearranging B.2 for I and substituting into B.3 gives

$$\Phi_{SQ} = \frac{MN\Phi_{ext}}{L_i + L_P} \tag{B.4}$$

we can express the mutual inductance M between the SQUID and the input coil as $M = k\sqrt{L_i L_{SQ}}$ where L_{SQ} is the inductance of the SQUID itself. Now we can write

$$\Phi_{SQ} = \frac{k\sqrt{L_i L_{SQ}} N\Phi_{ext}}{L_i + L_P} \tag{B.5}$$

What we are interested in is the combination of L_i and L_P that maximise Φ_{SQ} for a given Φ_{ext}. So, treating everything but L_i and L_P as constant we write

$$\Phi_{SQ} = \frac{C\sqrt{L_i}}{L_i + L_P} \tag{B.6}$$

and to maximise this we differentiate with respect to L_i and set the result equal to zero

$$0 = \frac{C(L_P - L_i)}{2\sqrt{L_i}(L_i + L_P)^2} \tag{B.7}$$

From which we see that to get the best efficiency from our system we must have

$$L_i = L_P \tag{B.8}$$

The number of turns in the pick up coil set and the design of the input coil are therefore carefully chosen to best match this criterion.

B.1 Second order Gradiometer

C Sequence Files

A sequence file is simply a tab delimited text file with the file extension **.seq**. The fastest way to create a sequence file is with the sequence editor as described in 4.8. Once you have a sequence created however, you might want to make another one that is very similar, or perhaps add or edit points in the sequence as it is running. This is possible by simply editing the text file in a basic text editor like notepad, which can be done even when the sequence is running. This is because the file is only opened briefly after each line is completed to load the parameters for the new line.

When you open a **.seq** file in a text editor you cannot see the column headings but they are the same as the columns in the sequence editor, namely

B(T)	field ctrl	T(K)	task	lengh	scan	turns	dt(s)	mode

B(T) and T(K), The set points for field and temperature. These are *floats*, you can enter any numerical value.

field ctrl This selects whether the magnet is in normal or low field mode. You can enter either

set field For fields up to the maximum 7T

set low field For small fields up to 30mT. This is more precice for such small fields.

task Selects which measurement(s) to take at each point.

length the length of a Zscan or peak to peak amplitude of an oscilating measurement

scan number of scans to perform at each point for a Zscan measurement. Enter an integer value

turns number of complete turns to use in a rotation measurement. Enter an integer value.

dt(s) sets a delay before moving on the the next line in the sequence. Enter any numerical value, in seconds.

mode For advanced users. **mode** is an integer that tells the system to use non-default settings for various advanced features, such as not checking temperature stability to measure on the fly, or using custom channel selection for measurements. These modes are configurable, for details speak to Cryogenic ltd.

C.1 Editing sequence files in a text editor

Sequence files can be opened in any basic text editor. As they are not **.txt** files you may need to choose *all files* from the file type menu when opening a sequence file. You can make any changes you want by simply deleting the commands you want to change and writing the new one in its place. Take care to keep the tab structure the same. Many times it is faster to create a new sequence rather than make sweeping changes to an existing one. For example, you have a sequence that measures from 5 K to 50 K in 0.5 K steps in zero field. At a later time you want to measure the same temperature sweep but at 1 T. In this case it is faster to use the sequence editor to create the new sequence than to replace 0.00 with 1.00 on every line in the field column. If however you have a complicated sequence of measurements that took some time to create with the editor and you want to repeat the sequence but with some different field or temperature values it could be much easier to use **find + replace** to make the changes directly in the text file than to use the editor.