LEM Operation Instructions

Z. Salman, A. Suter and T. Prokscha

May 25, 2021

Contents

| 1 | Safe 1.1 | ety First Definition of Hazards |
|----------|--------------------|-------------------------------------------|
| | 1.2 | Safety Measures |
| | 1.3 | Definition of Responsibilities |
| 2 | Qui | ick start guide |
| | 2.1 | Introduction |
| | 2.2 | Overview - Running a LEM experiment |
| | | 2.2.1 Sample mounting/changing |
| | | 2.2.2 Preparation of magnet and detectors |
| | | 2.2.3 Preparation of a moderator |
| | | 2.2.4 Beam Line transport settings |
| | | 2.2.5 Running measurements |
| 3 | San | nple Mounting/Changing 11 |
| | 3.1 | Introduction |
| | 3.2 | Preparing the sample |
| | 3.3 | Warming up the sample cryostat 12 |
| | 3.4 | Dismounting the cryostat |
| | 3.5 | Mounting the sample plate 13 |
| | 3.6 | Mounting the cryostat |
| | 3.7 | Update the Setup Configuration |
| | 3.8 | Preparations for the measurements |
| 4 | The | e Moderator |
| | 4.1 | Introduction |
| | 4.2 | Preparing for a new moderator |
| | 4.3 | Growing a new moderator |
| | 4.4 | Liquid He refilling |
| 5 | The | e LEM spin rotator 23 |
| - | 5.1 | introduction |
| | 5.2 | Design considerations |
| | 5.2 | Operation 27 |
| 6 | Cor | bling and Warming the Cryostat |
| Ŭ | 6.1 | Introduction |
| | 6.2 | Cooling down |

| CONTENTS | |
|----------|--|

| | 6.3 | Warming up | 31 |
|---|-----|-------------------------------------------------|----|
| | 6.4 | | 32 |
| 7 | San | nple Environment | 35 |
| | 7.1 | Introduction | 35 |
| | 7.2 | The cryostats | 35 |
| | 7.3 | General description of the Konti flow cryostats | 35 |
| | | 7.3.1 Konti-1 specific information | 39 |
| | | - | 40 |
| | | 7.3.3 Konti-3 specific information | 41 |
| | | - | 43 |
| | 7.4 | - | 43 |
| | | | 48 |
| | | - | 52 |
| | | | 54 |
| | | | 54 |
| | 7.5 | | 55 |
| | 7.6 | | 55 |
| 8 | Hig | h Voltage Table | 61 |
| | 8.1 | - | 62 |
| | 8.2 | 0 | 62 |
| 9 | Oth | ner Technical Details | 65 |
| | 9.1 | Thermal Contact Considerations | 65 |
| | 9.2 | | 66 |
| | 9.3 | | 67 |

Chapter 1 Safety First

For safety reasons and PSI's legal responsibility, users are not allowed to enter the μ E4 without being accompanied by their local contact. If you must go into the area in the absence of your local contact, please make sure that you inform him in person or by phone.

The μ E4 area is equipped with two alarm systems. The first monitors radiation (X-ray or radioactive) in the area when it is not in "Red" or prohibited access mode. The second is an oxygen depletion monitor. If you are in the area and you hear the sound of an alarm then simply leave the area immediately and call your local contact. Please do not try diagnose or understand the source of the problem, just leave immediately.

1.1 Definition of Hazards

High voltage (up to 20 kV) power supplies and high DC currents (up to 600 A) are used for the low energy muons (LEM) spectrometers. Liquid helium and nitrogen cryogens are used in moderator and sample cryostats to achieve the necessary low temperatures. The magnets on the LEM spectrometer generates high magnetic fields up to 0.35 Tesla. Radio frequency (RF) signals at low to moderate power levels (< 1 mW - 500 W) in the range of 0.1-30 MHz can be generated in the RF electronics of the LEM spectrometer. Various class IV lasers and high power LED light sources with wavelength between 365 nm and 635 nm and power up to 4.5 W are sometimes used to illuminate the sample in the LEM spectrometer.

1.2 Safety Measures

All samples which are intended to be studied in the LEM spectrometer must be solid and stable in air in its the full temperature range. They should not pose radioactive, fire or any other toxic hazards at any time under normal handling conditions. Since the spectrometers operate at ultra high vacuum (UHV), gloves should be worn primarily to prevent contamination of the studied samples and cryostats, and to prevent chemical (or potentially radioactive) contamination of experimenter's hands during mounting, removal and handling of samples.

Normal precautions with regard to high voltage or high current power supplies and cabling should be observed. The high voltage power supply for the detectors should be turned off before connecting or disconnecting the high voltage cable, and using only appropriately rated cables which are in optimal working conditions. The high current power supplies for the magnets should be turned off and disabled before connecting or disconnecting the magnets. All cryogens should be stored in approved dewars and transferred into cryostats using suitable transfer lines. Craning helium dewars in and out of the μ E4 area should be performed only by trained and authorized crane operators together with local PSI personnel. Appropriate safety equipment, i.e. suitable protective gloves and face mask, should be used when handling cryogens. Experimenters should remove any loose magnetic objects such as tools from the vicinity of magnets in the μ E4 area.

Setting up the laser or light sources must be performed by the local contact. Users are not permitted to perform this procedure alone and they must wear certified laser safety glasses (provided by the local contact) if they are present during setup. The light power during setup and alignment must be kept as low as possible, in the mW range, to avoid accidental eye or skin damaging exposure. Gloves must be worn to prevent damaging or smudging glass lenses and light sources. Once the setup and alignment process is completed, the entire laser/light path should be contained within an interlocked black box, preventing hazardous exposure to high light intensities outside the LEM spectrometer.

1.3 Definition of Responsibilities

The Experiment Leader (the main proposer) is the person primarily responsible for the safe operation of the experiment. She/He is responsible for ensuring that the apparatus and applied methods are safe, that appropriate procedures are established and implemented throughout the duration of the experiment and that members of the involved team are made familiar with the safety systems and procedures for the apparatus and experimental area. Responsibilities include ensuring that the apparatus and procedures satisfy any Swiss national or cantonal safety codes and regulations. The Experiment Leader should be aware of the training status of each experimenter in her/his team and for ensuring that they are all appropriately trained. Each experimenter is responsible for employing only safe practices while at PSI.

Chapter 2

Quick start guide

2.1 Introduction

The LEM spectrometer [1, 2] is a unique instrument that enables muon spin precession and relaxation (μ SR) measurements in thin films and multilayers. This is possible due to the tunable implantation energy (1-30 keV) of the muons, allowing depth resolved μ SR measurements between 1-300 nm. A schematic of the LEM beam line is shown in Fig. 2.1. Fully polarized surface

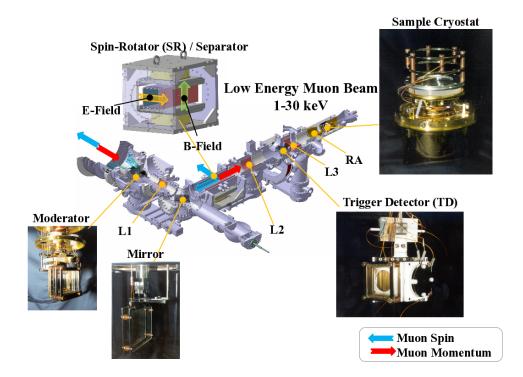


Figure 2.1: A schematic of the LEM beam line showing the different components starting from the moderator until the sample cryostat.

muons at ~ 28 MeV/c are stopped in a solid gas moderator (see Chapter 4 for details). They are then re-accelerated using a set of grids at a high voltage (see Moderator in Fig. 2.1), focused using an Einzel lens, L1, and deflected by 90° using an electrostatic mirror. This deflection insures that only slow muons (at a selected energy) continue in the direction of the sample. The muons then pass through a spin rotator (SR, see Chapter 5 for details) and then into an Einzel lens (L2) which focuses the beam onto the trigger detector (TD). Due to the divergence of the beam when passing the TD we use another lens, L3 to refocus the beam into the last focusing element which we call the ring anode (RA), which in turn focuses the muons onto the a sample mounted on the cold finger cryostat or oven.

The geometry of the LEM beam line results in a muon spin polarization that is nominally parallel to the surface of the studied samples, as shown in Fig. 2.1. The muon spin can be rotated horizontally using the SR to any angle between $\pm 90^{\circ}$ relative to the surface of the sample. Two magnets can be used on the LEM spectrometer, the WEW (B perpendicular) magnet produces fields in the range -350 to +350 mT along the muon beam direction while the AEW (B parallel) magnet produces fields in the range 0 to 25 mT along the up-down direction. Therefore, one can perform zero field (ZF), transverse field (TF) as well as longitudinal field (LF) μ SR measurements on the LEM spectrometer. A summary of important characteristics of the beam line, muons beam and spectrometer are summarized in Table 2.1.

| Beam line | |
|----------------------|-------------------------------------------------------------|
| Vacuum | UHV system, $\sim 10^{-10}$ mbar |
| | some parts are cooled with liquid N_2 |
| $\mu E4$ beam | |
| Rate | $\sim 1.9 \times 10^8 \ \mu^+/s$ |
| Energy | "surface" μ^+ beam, ~ 4 MeV |
| Beam after moderator | |
| Rate | $\sim 1.1 \times 10^4 \ \mu^+/s$ |
| Energy | $1-30 { m keV}$ |
| Implantation depth | 1-200 nm |
| Energy spread | $\Delta E \sim 400 \text{ eV}, \Delta t \sim 5 \text{ ns}$ |
| Polarization | $\sim 100\%$ |
| Beam spot | $\sim 12~\mathrm{mm}$ diameter (FWHM) |
| Sample environment | |
| Temperature | 2.5 - 500 K |
| Magnetic field | $0-25 \text{ mT} \parallel \text{surface (AEW) or}$ |
| | $0 - 350 \text{ mT} \perp \text{surface (WEW)}$ |
| Rate | at sample $\sim 4500 \ \mu^+/s$ |

Table 2.1: A summary of some important characteristics of the LEM beam line and spectrometer.

2.2 Overview - Running a LEM experiment

In preparation for a LEM experiment, the following general steps are taken,

- 1. Mount sample on the cryostat/oven.
- 2. Insert sample into the ultra high vacuum (UHV) beam line and select sample environment equipment.
- 3. Grow a fresh moderator.

2.2. OVERVIEW - RUNNING A LEM EXPERIMENT

- 4. Select the appropriate beam line transport setting, i.e. the initial muon kinetic energy.
- 5. Adjust the muon spin rotation angle.
- 6. Run the measurements.

Less experienced users will be gently guided by a local contact to help them get started and run the experiment. In this chapter we give a quick and general overview on running an experiment. Further details on the operation of various aspects of the experiment are given in the following chapters.

2.2.1 Sample mounting/changing

Samples for LEM measurements should be flat, plate-like such that they can be glues safely onto the flat sample plate. The samples should be "UHV clean", i.e. should not have any greasy or organic residues on them and should always be handled while wearing appropriate gloves. The samples are generally glued using silver paint onto a silver or nickel coated sample plate (72 mm diameter). For cryostats operating below 325 K aluminum sample plates are used, while stainless steel sample plates are used for the oven. To determine the appropriate type of sample plate for your experiment, please consult with your local contact. Note that we recommend that you prepare your sample in coordination with your local contact before the "official" start date of your experiment and at least *one day in advance*.

After gluing, the sample plate is mounted onto a cryostat using insulating Vespel (polyimidebased plastic) screws. When using the oven, stainless steel screws are used to mount the sample plate. Typically the cryostat/oven will be sealed with a specially designed vacuum sleeve and pumped until the start of your experiment. At the official start date of the experiment (9:30 am), the cryostat/oven is inserted into the UHV beam line and pumped to a level of $< 10^{-8}$ mbar before it is cooled and measurements are started.

2.2.2 Preparation of magnet and detectors

Once the cryostat is mounted into the beam line, one can select the appropriate magnet for the experiment. For measurements requiring a field applied perpendicular to the surface of the sample (as well as ZF measurements), we use the WEW magnet (-350 to +350 mT). On the other hand, if you require fields applied parallel to the surface of the sample, then we use the AEW magnet (0 to 25 mT). Each magnet comes with its own detector set. The magnet can be installed before or after inserting the cryostat/oven (except in the case of the LowTemp cryostat). Make sure that the positron detectors are connected after installing the magnet.

2.2.3 Preparation of a moderator

The efficiency of the moderator degrades with time, typically loosing up to 30% efficiency within a week. However, this depends on the vacuum level and sample or other out-gassing elements in the beam line, which can cause a significant drop within a short time. Therefore, we deposit a new moderator layer at the beginning of each experiment, usually during the sample pumping time. Your local contact will help you with the moderator deposition, however, we give here a short description of the process. Further details can be found in Chapter 4.

To deposit a new moderator layer, the moderator cryostat (Moddy) is warmed up to 150 K, both the cold finger and He radiation shield. The temperature is maintained at 150 K for at least 30 minutes to ensure that the old moderator layer is evaporated completely. Moddy

is cooled back down to ~ 10 K on the cold finger and ~ 40 K on the radiation shield. Once the temperature is stable, a small amount of Ar gas is introduced into the moderator vacuum chamber. Some of the Ar gas freeze on the cold finger of Moddy and the thickness of the layer is monitored during this process. A typical thickness of ~ 250 nm (250 Å on the XTC monitor) solid Ar is deposited followed by a ~ 10 nm (10 Å on the XTC monitor) layer of solid N₂ by introducing N₂ gas into the moderator chamber.

2.2.4 Beam Line transport settings

At the beginning of each year we tune the LEM beam line, optimizing the transport of muons for different kinetic energies. The results of this optimization are saved in "transport setting" files. The choice of most suitable energy for your specific experiment depends on your sample composition and thickness. Your local contact will be happy to help in deciding the right transport settings for your experiment. The task of adjusting the transport settings is then reduced to simply loading the appropriate transport setting file using hvEdit program, which you will be able to access from all desktops in the area or control cabin.

2.2.5 Running measurements

We try our best to automate all aspects of LEM, meaning that for a user, running a LEM experiment is as simple as writing a script to adjust different parameters and perform measurements. These scripts, also called autoruns, are a text file with a sequence of commands. The data acquisition systems (DAQ) performs these commands sequentially. Examples of commonly used commands are,

| To change | e sample temperature: |
|-----------|--------------------------------------------------------------------------------------|
| TEMP | t,dt,timeout,rate |
| t | - the requested temperature in K |
| dt | - tolerance on requested temperature |
| timeout | - time out time to reach stability in seconds. |
| rate | - is the ramp rate of the temperature to reach the destination temperature in K/min. |
| To change | e magnetic field |

FIELD b G

.

b - is the requested magnetic field in Gauss

To start a new run TITLE title
START N
title - the full title of the run which will be saved in the data file
N - The number of positron events to be saved in the run.

These are only a few examples to get you started, however, you can find a full list of available commands can be found in the complete LEM AutoRun Documentation. Alternatively, you may want to have a look at old autorun scripts for more examples or templates.

Chapter 3

Sample Mounting/Changing

3.1 Introduction

This chapter presents a detailed description for preparing and mounting a sample for a LE- μ SR experiment. An inexperienced user will be assisted by her/his local contact throughout this process. The preparation of sample change should start well in advance of the planned change, since it involves gluing the sample on an aluminium plate which requires a few hours (preferably 6h) to dry before introducing it into the UHV beam line of the LE- μ SR apparatus. Please use appropriate UHV compatible gloved during this whole process to avoid contamination of your sample or any other components that go into the UHV sample chamber. Below, we provide a detailed description of the process.

3.2 Preparing the sample

The sample can be either glued or clamped onto the sample plate. Only relatively large circular samples can be clamped. To achieve good thermal contact between the sample and the sample plate, we recommend using silver paint to glue the sample. The silver pain will be provided by your local contact. Alternatively, you may use Apiezon N grease in special cases, but this is not recommended for measurements requiring high or low temperatures. For a detailed comparison between different glues see section 9.1.

The sample plates are available in two variants, silver (Ag) or nickel (Ni) coated plates. The Ag plates are recommended if you have large samples (¿2cm diameter) or if you would prefer to work with non-relaxing background signal. The Ni plates are recommended if you have small samples and would like to suppress background signals from muons missing the samples. Consult your local contact for the best option for your specific measurement.

Once you have chosen the type of sample plate, follow theses steps to glue the sample,

- 1. Clean the sample plate with acetone and ethanol and orient it as it is mounted in the cryostat, i.e. the high voltage (HV) connection hole is pointing to the top right (2 o'clock).
- 2. If the sample plate has no indium foil at the bottom side, glue a new and clean indium foil using Apiezon N grease on the bottom side of the plate. Only a very small amount of grease is needed in the middle of the sample plate. Rub the foil gently using a lint free cloth with some Acetone to flatten the foil on the plate. Make sure that the foil remains exactly centred on the sample plate, no parts of the foil should be sticking outside the plate since this may cause HV problems.

3. Glue your sample in the center of the plate using silver paint. A few droplets should be sufficient (see Fig. 3.1). Make sure that the sample is perfectly flat on the plate with no

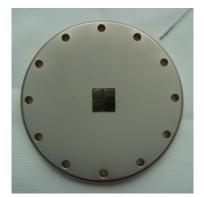


Figure 3.1: A sample plate with a mosaic samples of four pieces, each $5 \times 5 \text{ mm}^2$. The metallic needle marks the position of the HV hole on the sample plate.

air bubbles between them.

- 4. Optionally, create and electrical connection between the surface of your sample and the plate using a small drop of silver paint on the edge of the sample. If you have a mosaic as a sample, connect all individual pieces to the sample plate.
- 5. Let the sample dry for at least six hours, possibly while pumping in a special transport desiccator. Do not stop pumping during the whole drying procedure to ensure that the solvents from the paint is pumped out. This will significantly shorten the required pumping time in the beam line.

3.3 Warming up the sample cryostat

The sample should be mounted onto a warm and dry cryostat. Typically, you will have at least one warm cryostat already available for use. However, if the cryostat is being used then it has to be warmed up before you open the UHV sample chamber. Please follow these steps below carefully (requires about two hours).

- 1. Run the warmup_vent.lar autorun, which ramps down all HVs of the LEM beam line, closes the valves BPVX (between MC and TD) and BPVY (between TD and SC), and then it warms the sample up to 300 K. When the sample reaches ~ 150 K, the autorun will automatically vent the SC with dry N₂ gas.
- 2. Stop He pumps and open the He recovery valve between the pump and the He recovery system, see Fig. 3.2.
- 3. Once the sample have reached 300 K, wait until all parts of the cryostat are warm; in the vented SC the heater power on the sample should be ~ 20 % or less.

3.4 Dismounting the cryostat

Once the cryostat is warm, you may start dismounting it by following these steps,



Figure 3.2: The LEM pumping system.

- 1. Remove the transfer line from the cryostat and replace it immediately with the plug. You may remove the transfer line from the dewar and leave it to dry before the next use, or simply close the needle valve and leave it in the dewar.
- 2. Disconnect the pumping line from the cryostat and seal it using a KF blind flange to prevent air from entering into the He recovery system.
- 3. Disconnect the red sample HV cable from the cryostat, then disconnect the grey thermometry cable and *only then* disconnect the heater cable from cryostat.
- 4. Remove the cryostat from the apparatus by opening the 100CF flange. Avoid hitting the cryostat against the SC tube while extracting the cryostat.
- 5. Leave the cryostat with the sample facing upwards on the cryostat trolley in the clean area. If the cryostat is not needed immediately seal it with a sleeve and pump it to keep it clean until the next use.
- 6. Close the vacuum tube as soon as possible with a 100CF blind flange. Four screws and an old but clean copper gasket are sufficient for this.

In rare cases, it may be difficult to remove the old copper gasket from the end of the beam pipe or in the cryostat flange. In this case you may need to extract it carefully using a screw driver. Insert the screw driver into the designated groove (see Figs. 3.3) and twist it gently.

3.5 Mounting the sample plate

Please use UHV compatible gloves when working with the sample and the cryostat. Also, avoid making any scratches on the sample plate and cryostat surfaces, since they may produce HV stability problems. Finally, always remember to connect the sample HV wire to the sample plate.

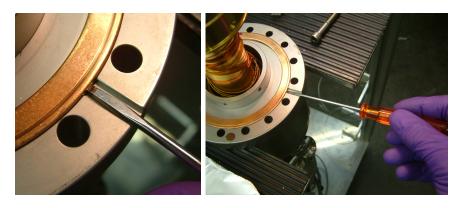


Figure 3.3: Gently insert a small screwdriver into the designated groove, under the copper gasket. *Do not push it too deep* to avoid damage to the knife edge. Twist the screwdriver slightly or use it very gently as a crowbar.

Follow carefully the steps below to mount your sample (which is already glued onto a sample plate) onto a cryostat. Whenever in doubt, do not hesitate to ask for assistance from your local contact. All tools that you will need are in/on the work bench in the clean area. Please keep these tools clean (gloves!) and keep them in the clean area.

1. Remove the screws attaching the radiation shield to the cryostat and remove the shield carefully (Figs. 3.4). Put all screws in the designated tray and the shield in a safe and clean place, e.g. on a clean aluminium foil.

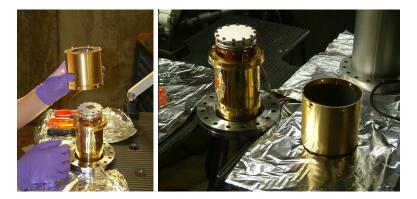


Figure 3.4: Removing the radiation shield from the cryostat.

- 2. Pull out carefully the HV plug from the currently mounted sample plate.
- 3. Unscrew the brown Vespel screws using the appropriate size screw driver and put them in the designated tray. A non-fitting screw driver head will will damage these fragile screws.
- 4. Remove the old sample plate from the sapphire (Fig. 3.5). This may require some force if the indium foil is stuck to the sapphire, gently twisting may also help.
- 5. Put the new sample plate onto the sapphire such that the HV hole is at the desired position ($\sim 45^{\circ}$ relative to the top of the cryostat). The top of the cryostat is marked by a sticker with the name of the cryostat. Make sure that no indium foil is visible between the sapphire and sample plate.

3.6. MOUNTING THE CRYOSTAT

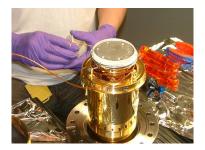


Figure 3.5: Sample cryostat with removed sample plate

6. Attach the sample to the cryostat using the brown Vespel screws. Initially use your fingers and do not tighten too much. Then use the fixed torque screw driver (20 cNm, Fig. 3.6) to tighten the screws fully following a star pattern. After tightening all screws, make *one full round* to ensure that all screws are tight using the same torque screw driver. Any broken screws can be removed using a flat nose pliers and replaced by a clean spare Vespel screw from the workbench of the clean area.



Figure 3.6: Tightening Vespel screws with torque screw driver to fix the sample plate

- 7. Push the sample HV plug into the corresponding hole in the sample plate until the metal plug is completely in. Check the electric connection between the outer HV plug and the sample plate with the help of a multi-meter.
- 8. Remount the radiation shield, rotating the shield counter clockwise may help getting it into position. Make sure that no wires (thermometry, HV etc.) touch the sample stack or the radiation shield. Fix the radiation shield using the appropriate screws.

3.6 Mounting the cryostat

Now you are ready to remount the cryostat into the SC by following these steps,

- 1. Remove the 100CF blind flange from the SC vacuum tube.
- 2. Place a new 100CF copper gasket at the vacuum tube. Typically, we use a small strip of sticky tape (3 cm \times 0.5 cm, Fig. 3.7) to fix the gasket to the tube. Make sure that the tape does not cover more than 2 mm of the gasket from its outer edge.
- 3. Insert the cryostat into the vacuum tube with the new gasket. Hold and push the flange of the cryostat against the SC tube, then screw in two or three screws by hand which should be enough to hold everything safely in place. At this stage, it is still possible to rotate the

CHAPTER 3. SAMPLE MOUNTING/CHANGING



Figure 3.7: Preparing a new 100CF copper gasket with sticky tape.

cryostat so that its upper side, marked by a sticker, is in the correct orientation (Fig. 3.8).

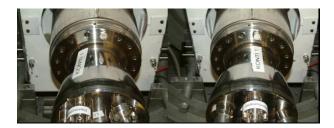


Figure 3.8: Konti-1 positioned incorrectly (left) and correctly (right). The exact orientation does not affect the operation of Konti cryostats.

4. Screw all remaining screws one after the other, following a star pattern, by hand or using the electric screw driver. Finally, use the fixed torque wrench (see Fig. 3.9) with 20 Nm to fully tighten the screws, again following a star pattern to ensure that the cryostat and sample are perfectly centered in the beam line.



Figure 3.9: Fixed torque wrench

- 5. At the main "LEM Automatic" screen of the vacuum control touch screen (Fig. 3.10), press on the "Gas" button (near "Sample Chamber"). This will get you to the "Sample Chamber" control screen, press on "No, back!" which closes the SC vent valve and takes you back to the main screen.
- 6. Press on the "Sample Chamber" to start the pumping the SC, first using the pre-pump and then the turbo pump (when you hear the big gate valve open).

For a clean sample and a cryostat which was previously kept pumped in the vacuum sleeve, the pumping will take about two hours. You may open the BPVY valve (between SC and TC)

3.7. UPDATE THE SETUP CONFIGURATION



Figure 3.10: Main screen (left) and sample chamber (right) of the vacuum control touch screen.

only when the SC pressure, SC₋GJ2, is below 8×10^{-7} mbar. This is done by clicking on the BPVY value in the lemvac control or on the touch screen of the vacuum control. After another ~ 15 min you may start to cool down the cryostat. We recommend that you wait long enough to get to 1×10^{-7} mbar before cooling or opening BPVX. This will keep a high moderation efficiency at the moderator and prevent the formation of frozen gas layers on your samples. The latter is particularly important if you plan to work with low implantation energies/depths.

3.7 Update the Setup Configuration

It is important to set up the correct configuration in the data acquisition (DAQ) system. This includes the name of the cryostat being used, the magnet etc. This ensures that the correct calibration tables of the thermometers are used by the temperature controller and that the correct power supply is used for the magnets. This is easily done by clicking on the "ConfigSetup" link in the navigation bar at right side of the main experiment status page, see Fig. 3.11. The

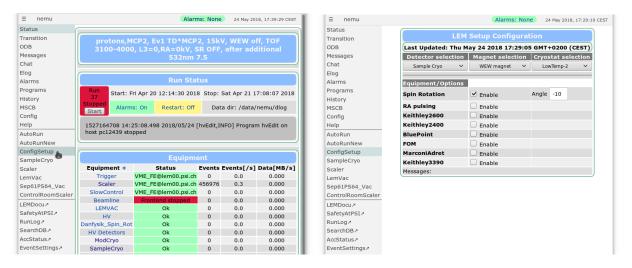


Figure 3.11: (Right) Midas main status page (right) with the "ConfigSetup" link highlighted. (Left) The "ConfigSetup" page.

configuration can be changed (if needed) in the following way

- 1. Select "Sample Cryo" from the "Detector selection" drop-down menu.
- 2. Select the appropriate magnet for your setup from the "Magnet selection" drop-down menu.
- 3. Select the appropriate cryostat from the "Cryostat selection" drop-down menu.

Having selected the new cryostat and magnet, the DAQ will automatically update all the necessary settings. You can follow and diagnose the initialization process for the new cryostat on the Midas "Messages" link in the navigation bar.

3.8 Preparations for the measurements

While the vacuum of the SC is being pumped, you should start preparing the cryostat for cooling and resetting the HV vacuum interlocks.

1. Reconnect the grey thermometry cable and red sample HV cable to the cryostat, see Fig. 3.12.



Figure 3.12: Connected Konti cryostat

- 2. Connect the He pumping tube to the cryostat. Do not forget the heating element which prevents the formation of ice on the cryostat.
- 3. Close the He recovery valve.



Figure 3.13: The HV interlocks for the SC, TC and MC.

4. There are three HV interlocks to prevent application of HV when the vacuum level in the SC, TC or MC is above 10^{-6} mbar (see Fig. 3.13). Once the vacuum level is below this value, you will be able to reset the respective interlock in each chamber. Simply press on the corresponding *black push button* to release the interlock. The light on these interlocks is red when the vacuum level is above 10^{-6} mbar. It turn orange when the vacuum reaches the required level, and becomes green when the interlock is released.

Now the system is ready to start cooling cooling the cryostat and begin with your measurements.

Chapter 4

The Moderator

4.1 Introduction

The muons in a LE- μ SR experiment are moderated using a thin cryo-solid layer. The moderator is composed of a thin, structured Ag foil (~ 125 μ m) on which a much thinner (< 1 μ m) layer of condensed van der Waals gas is deposited[3, 4]. In order to keep the gas layer intact, it is held at a temperature below 13 K using a specially designed cryostat. The gas layer degrades slowly over time, i.e. its moderation efficiency decreases, requiring a fresh layer to be grown roughly once a week in standard experiments. The aim of this chapter is to guide you through the procedure of growing a new moderator layer. It will also provide information regarding the operation of the moderator cryostat.

4.2 Preparing for a new moderator

A new moderator can be grown following the steps described below.

- 1. If there is an active measurement, pause or stop it.
- 2. Close the vacuum valve BPVX separating MC and TC using the vacuum control touch screen near the moderator cryostat (Fig. 4.1). You should hear a hissing sound when the valve is closed and its color on the touch screen will change from green to red.



Figure 4.1: Main screen of the vacuum control touch screen.

3. Run hvEdit from the laptop in the μE4 area, then select "FUG" from "Devices" dropdown menu. Zero the voltages of "Moderator", "Mod_Guard", "Mod_Grid", "Lense_1", "Mirror". Then select "NHQ" from "Devices" drop-down menu and set "HV MCP1" to 0 (see Fig. 4.2). Before you continue, make sure that all read-back values of these elements are actually 0, if not, please do not proceed and call your local contact.

| Pevices Connection FUG (kV/mA) r | | | | | | | |
|------------------------------------------------------------|-----------|---------|----------|------------------|----------|---------|--|
| н١ | / Current | t Limit | | Trip Rate Source | | | |
| | Name | Demand | Measured | Current | | | |
| 1 | RightRo | -0.713 | -0.728 | 0.00(| Switch | All Off | |
| 2 | LeftRods | 0.713 | 0.718 | -0.000 | Diricei | | |
| 3 | LeftPlate | 1.426 | 1.438 | -0.00(| Input: | - | |
| 4 | RightPl | -1.426 | -1.436 | 0.000 | | | |
| 5 | Modera | 15.000 | 15.019 | 0.000 | | Set | |
| 6 | Mod G | 12.000 | 12.001 | 0.000 | | | |
| 7 | Mod Grid | 10.400 | 10.391 | 0.00(| -1.0 | +1.0 | |
| 8 | Lense 1 | 9.000 | 8.992 | 0.00(| -0.1 | +0.1 | |
| 9 | Mirror | 15.000 | 15.005 | 0.000 | | يعتب | |
| 10 | Lense_2 | 10.500 | 10.484 | 0.00(| -0.01 | +0.01 | |
| 11 | Lense_3 | 11.500 | 11.483 | 0.00: | | | |
| 12 | RA-L | 11.900 | 11.884 | 0.000 | Restore | Zero | |
| 13 | RA-R | 11.900 | 11.896 | 0.000 | Sele | ct All | |
| 14 | RA-T | 11.900 | 11.888 | 0.000 | Dere | | |
| 15 | RA-B | 11.900 | 11.910 | 0.00(| | | |
| 16 | Sample | 0.000 | -0.005 | 0.00: | | | |
| | | | | | Scale by | | |

Figure 4.2: The hvEdit window with the "FUG" devices selected.

The remaining steps are optional, you may grow up to three moderators without warming up to remove a previously grown moderator. You may now go directly to Section 4.3 or follow the remaining steps to start with a fresh moderator.

4. Warm up the moderator to evaporate previous gas layers (if present). From the experiment laptop, run lemModCryo shown in Fig. 4.3. Set "CF1 demand" (CF = Cold Finger), as well as "He Shield demand" to 150 K. Set the He-pressure "demand" on the right side of the window to 0.

| ile P | eferences <u>H</u> elp | | |
|----------|-----------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------|
| mea O | CF1 demand He shield der sured temperatures LS_ModCryo_CF_1 LS_ModCryo_CF_2 LS_ModCryo_F_2 LS_ModCryo_He_Shield | 10.00 • 40.00 • 10.00 9.67 9.72 40.00 84.35 | Needle Valve Controll auto mode sta manu- pressure demand 3.8 measure 3.8 needle valve positio demand 2.0 measure 0.0 |
| Heate | r Outputs in (%) | | |
| cold | finge | 40 | 60 80 100 |
| | 0 20 | 40 | 60 80 100 |

Figure 4.3: The lemModCryo window to control the moderator cryostat.

- 5. Wait for at least half an hour, until all gasses have evaporated and are pumped. You can monitor this on the pressure gauges in the moderator chamber which will increase as the moderator is evaporated and decrease again to below 10^{-8} mbar when the evaporated gases are pumped out.
- 6. Cool down the moderator cryostat by setting "CF1 demand" to 10 K and "He Shield demand" to 40 K. These values are correct for the moderator types currently used, but may be different for other types. Currently, we are using the **Eurotherm 2416** controller to control the needle value of the moderator cryostat. Use the automatic mode for this controller (uncheck "manual"). To speed up cooling, increase the pressure value to about a demand value of 100. Once the cryostat is cold reduce this value gradually until you reach normal operation value of ~ 6, resulting in a heater power of ~ 10%.

4.3. GROWING A NEW MODERATOR

7. Wait until the temperature and pressure readings are stable.

Now you are ready to evaporate and grow a fresh moderator.

4.3 Growing a new moderator

Different types of solid gasses can be used in the moderator, each with a different moderation efficiency and stability. The most commonly used moderator is a bilayer of N_2 on top of Ar. It is also possible to use a single layer of N_2 moderator. The Ar/N₂ moderator is more efficient but less stable with application of high voltages compared to N₂. These moderators are grown under different conditions, which are summarized in the Table 4.1. The following steps guide

| Moderator | Gas | Pressure | Thickness | Time | Cold | Shield |
|----------------|----------------|----------|-------------|-------|--------|--------|
| | | | | | Finger | |
| | | (mbar) | (kÅ) | (min) | (K) | (K) |
| Ar/N_2 | Ar/N_2 | 1E-6 | 0.230/0.012 | 3/0.5 | 10 | 40 |
| N ₂ | N ₂ | 1E-6 | 0.125 | 3 | 10 | 40 |

Table 4.1: Growth conditions for different types of moderators.

you through the moderator deposition process,

- 1. Make sure that BPVX is closed, all HV in the MC are at zero, and that the temperature of the moderator cryostat is stable.
- 2. Pump and flush the gas pipes, shown in Fig. 4.4, in order to make sure that there are no residual gases. Use the small electronic pressure gauge attached to these pipes (marked in Fig. 4.4) to monitor the pressure in the gas inlet tubes. This gauge may be off and can be turned on by simply pressing on the little black button of the right side of its display.
 - Open the pumping valve gently and pump the gas inlet tubes down to until the gauge shows zero.
 - Close the pumping value and open the appropriate gas value to introduce $\sim 500-1000$ mbar into the tubes and then close the value.



Figure 4.4: View of the gas inlet tubes and pumping system

• Repeat this process three times for each gas type/layer.

- At this point, and also during growth, it is possible to take a look at the quadropole mass spectrometer (QMS) to make sure that there are no undesired gases in the MC. To do this, follow these steps:
 - Verify that the QMS is connected to the laptop.
 - Run the program RGA4WIN from the desktop.
 - From the toolbar, turn on one of the filaments (filament button,1 or 2).
 - Start collecting data (bar graph button). To speed up the data collection start also the M-Multiplier.

For example, with N_2 the two highest peaks should occur at A=18,28 and 40 for H_2O , N_2 and Ar, respectively.

3. Move the trumpet (Fig. 4.5) from the "OUT" to "IN" position. Pull it until the line marked "IN" and fasten the butterfly knot.

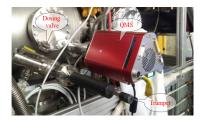


Figure 4.5: View of the trumpet, QMS and dosing valve.

4. Using the "LEM Automatic" vacuum control touch screen, press on the "Gas" button (near "Moderator Chamber", Fig. 4.6), then confirm by pressing "Yes, I want" to activate the HV interlock for the MC.



Figure 4.6: Main screen (left) and moderator chamber (right) of the vacuum control touch screen.

- 5. From the "LEM Automatic" screen (should be back) press on one of the pressure gauges of the MC to obtain a detail view of the vacuum level in the different chambers, as shown in Fig. 4.7.
- 6. The XTC device, shown in Fig. 4.8, is used to estimate the thickness of the grown gas layer. Reset it by clicking "2" ("Zero").
- 7. Open gently the dosing valve near the trumpet (counterclockwise, see Fig. 4.5) to allow gas flow from the gas pipes into the MC. Open the dosing *slowly* until the MC gauges reach $\sim 10^{-6}$ mbar.

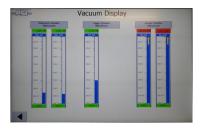


Figure 4.7: Detailed view of the vacuum level in the different chambers.



Figure 4.8: The XTC unit for measuring the thickness of moderator layers

- 8. When the target pressure is reached, press the "START" button of the XTC (see Fig. 4.8), to start counting growth time. If the QMS is running, it is expected that the strongest peaks now will correspond to the gas being grown.
- 9. When XTC reading approaches the desired value corresponding to the grown gas layer, start closing the dosing valve slowly (clockwise). This is a very fragile value, so please **do** not tighten too much.
- 10. When all layers are finished remember to move the trumpet back to its "OUT" position and fasten the butterfly knot.
- 11. From the vacuum touch screen, press the back arrow to go to the "LEM Automatic" screen again. Press on the "Gas" button near the MC, and then press on "No, back" to disable the MC HV interlock.
- 12. Switch off the QMS filament if it is on.
- 13. Create a new ELOG entry: don't forget to mention Gas, XTC thickness, growth time (XTC), and growth pressure.
- 14. Wait until the pressure drops to below 10^{-6} mbar and reset all HV interlocks (see Fig. 3.13).
- 15. Open BPVX and BPVY vacuum valves. Ramp up all HV to the desired values; don't forget to also set the "MCP1" HV to +2.6 kV (in "NHQ").

4.4 Liquid He refilling

The moderator requires liquid He refilling at least once a week. This should be done before the liquid He level reaches 1.5 (check using History \rightarrow Mod Cryo \rightarrow LHe level). Follow these steps to refill the moderator,

- 1. Stop or Pause current run. Zero the HV on "Moderator", "Mod_Guard", "Mod_Grid1", "Lense_1", "Mirror" and "MCP1". Close BPVX from the vacuum control touch screen.
- 2. Open He gas bottle to pressurize to the LHe dewar at the moderator side. Open the upper of three black valve of the He gas line below the moderator cryostat/Mirror.
- 3. Set LHe level meter for the dewar and for Moddy from "pulse"/"sample mode" to "continuous mode"/"update".
- 4. If the transfer line is not already in the dewar/cryostat then
 - Close the valve to the He recovery system to build up pressure and insert slowly the transfer line into the dewar.
 - When the pressure in the dewar increases, you should feel the He gas flow on the other end of the transfer line.
 - Insert the transfer line into the dewar until it hits the bottom, then lift it up by a few centimetres.
 - Purge the transfer line for at least 30 s before inserting it into the moderator cryostat.
- 5. Close the recovery value of the He dewar and open the value to the He gas line to apply about 0.3 bar pressure in the dewar.
- 6. Check the fill levels at the laptop in the Midas history, transfer LHe until the measured level in Moddy is 6.0 V. Normally, this requires about 25-30 % of the dewar.
- 7. When the filling is done, set the LHe level meters for Moddy and the dewar back to "sample"/"pulse" mode, and keep the transfer line in the dewar and in Moddy.
- 8. Close the black value of the He gas line, open the value to the recovery system and close the He gas bottle.
- 9. Make sure that the moderator temperature and the pressure in MC stabilizes. Wait until the pressure drops to below 10^{-6} mbar and reset all HV interlocks (see Fig. 3.13).
- 10. Open BPVX vacuum valves and ramp up all HV values to the desired values; don't forget to also set the "MCP1" HV to +2.6 kV (in "NHQ").

Chapter 5

The LEM spin rotator

5.1 introduction

In 2012 a spin rotator (SR) for the LEM spectrometer was developed to enable LF measurements. An ideal spin rotator is formed by a combination of static magnetic (B) and a perpendicular electric (E) fields which satisfy v = E/B, where v is the velocity of the muons. This ensures that the beam is not deflected when passing through the SR. When passing through the SR, the spin of the muon is rotated by an angle $\theta = \gamma B t_0$, where γ is the muon's gyromagnetic ratio and t_0 is the time it spends in the SR. Typically, for a surface muon beam line (~ 28 MeV/c), a rotation of 90° for TF measurements requires magnetic fields of ~ 0.1T and electric fields of ~ 7.5 kV/mm over a length of ~ 1.5 meters. Such requirements make the design and construction of these SRs a technically challenging task, and usually only a rotation of $40^\circ - 50^\circ$ is achieved with a single spin rotator. Fortunately, the low energy of the muons after the moderator on the LEM beam line simplifies the SR requirements considerably. In this chapter we present the various aspects for the design of the LEM SR and its operation. The SR allows for a spin rotation in the range -90° to $+90^\circ$.

5.2 Design considerations

Without rotation, the initial spin of the beam as it approaches the sample, is perpendicular to its momentum due to a 90° bend in the beam line between the moderator and the sample position. For LF μ SR measurements using the available magnets and spectrometer, one needs a spin rotator (SR) after the moderator. However, space limitations impose a small footprint of ~ 40 × 40 × 40 cm³ on the SR. Taking these limitations into consideration we designed a SR which is composed of a dipole magnet, producing a vertical magnetic field, and a set of conducting plates to produce a corresponding transverse horizontal electric field (see Fig. 5.1). The main considerations in the magnet design were its physical length, uniformity/homogeneity and sufficient magnetic field amplitude (using a reasonable current) to achieve a full 90° spin rotation at typical muon energies (< 20 keV). For the electrostatic plates, we aimed at providing an electric field that compensates the deflection of the beam due to the magnetic field while maximizing beam transmission through the SR.

Figure 5.2 we plot an example of the asymmetry between the left/right detectors as a function of SR magnet current using 12 kV transport settings (potential on the moderator). The potential on the SR plates in these measurements was of course adjusted to keep the beam on axis. One can clearly see that the asymmetry follows a sinusoidal function of the current (which is proportional

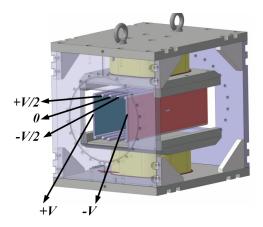


Figure 5.1: A schematic of the SR showing the magnet and electrostatic plates and rods. $\pm V$ is the potential applied on the plates.

to the SR field and the rotation angle). This clearly shows that continuous rotation of the muon

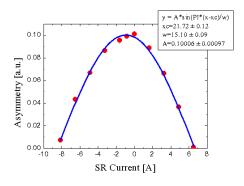


Figure 5.2: The asymmetry as a function of the current in the SR magnet. The asymmetry which is proportional to the projection of the polarization is a sinusoidal function of the current due to the spin rotation. The offset is due to the initial $\sim 10^{\circ}$ rotation in the LEM separator.

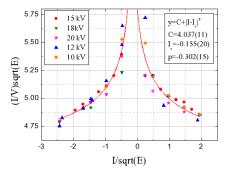


Figure 5.3: The asymmetry as a function of the current in the SR magnet. The asymmetry which is proportional to the projection of the polarization is a sinusoidal function of the current due to the spin rotation. The offset is due to the initial $\sim 10^{\circ}$ rotation in the LEM separator.

spin in the SR between -90 and 90 degrees.

In order to simplify the use of the SR for the user, it has been calibrated extensively for the different transport settings (8.5kV up to 20 kV). Let us denote V the absolute value of the potential on the electric field plates, I the current in the SR magnet, and E the kinetic energy of the muons passing through the SR. Naturally, each transport setting (E) requires different values of magnetic (I) and electric (V) field to achieve a certain spin rotation. Moreover, due to the finite size of the SR and the non-ideal nature of the electric and magnetic fields within its volume, these also depend on the required rotation angle. However, as can be seen in Fig. 5.3, we find that plots of $I/(V\sqrt{E})$ as a function of I/\sqrt{E} fall on the same curve for all transport settings. The logic behind this scaling is that as the muon passes through the SR its spin is rotated by $\phi = Bt$, where B is the magnetic field and t is the time it spends in the magnetic field. This time is inverse proportional to the velocity of the muons, \sqrt{E} , while the magnetic field is proportional to the current I. Therefore, ϕ is proportional to I/\sqrt{E} and plotting $I/(V\sqrt{E})$ as

5.3. OPERATION

a function of I/\sqrt{E} will give us the relation between the ratio of I/V for a given rotation angle regardless of beam energy, which leads to the scaling curve for all transport settings in Fig. 5.3. This allows us to determine I and V of the SR for a certain rotation ϕ at a given transport setting.

5.3 Operation

For the user, setting a rotation angle is a fairly simple task. Once the transport settings are loaded (i.e. the high voltage settings for the moderator and lenses), it is possible to set the muon spin angle in two different ways,

- 1. Preferably, using the SPIN_ROT command in an autorun.
- 2. Using the MIDAS web interface, click on ConfigSetup button, enable "LEM Spin Rotation" and set the "Rotation Angle" to the desired value.

The first option is the safest and best option for spin rotation. It is also recommended that the user uses a spin rotation of -10° even in TF experiments. This operation mode effectively uses the SR in separation mode, i.e. filters the beam from charged ions and other contamination and reduces the number of false hits on the trigger detector.

CHAPTER 5. THE LEM SPIN ROTATOR

Chapter 6

Cooling and Warming the Cryostat

6.1 Introduction

All cryostats used in the LEM sample environment are He flow cryostats, i.e. liquid He or cold He gas are pumped from a dewar through the cryostat. A heater mounted on the cold finger of the cryostat is controlled by a LakeShore340 temperature controller to provide a small amount of heating to stabilize the temperature. The amount of liquid or cold gas should be just enough to reach the desired temperature. The so-called Konti cryostats have one circuit for the cold gas reaching a base temperature of ~ 4 K, while the LowTemp cryostats have two circuits. In the latter, an additional small vessel inside the cryostat acts as a phase separator, which is continuously filled with liquid He under low pressure. This feature allows the LowTemp cryostats is the complexity of their operation compared to Konti cryostats. In all cases, care has to be taken to prevent moisture from entering the cold parts of the cryostat and remove the blockage. Please wear protective gloves when working with cryogenic liquids.

6.2 Cooling down

The cryostat is connected to the liquid He dewar by a transfer line, which generally has a needle valve built into it to adjust He flow through it. This valve is in the cold part of the transfer line, at the lower end of the He dewar side. To start cooling down the cryostat

- Open the needle value of the transfer line fully (100 % on the electric ones, 3 turns on the manual one).
- Close the recovery value of the dewar (Fig. 6.1), this allows for pressure built up.
- Insert the transfer line into the dewar slowly until you notice a pressure increase to about 0.1 0.2 bar. If the pressure gets too high (> 0.3), simply open the recovery value on the dewar to release some pressure. Continue inserting the transfer line until it reaches the bottom of the dewar, then pull it back up by ~ 3 cm.
- Let He gas flow through the transfer line to completely flush it, then insert the other end of the transfer line into the cryostat. Turn the brass nut 180° counterclockwise (Fig. 6.2, 6.3), remove the aluminum plug and insert the transfer line fully, you should then feel it hitting something. Tighten the brass nut (clockwise).



Figure 6.1: Red Recovery-line-valve on the He dewar

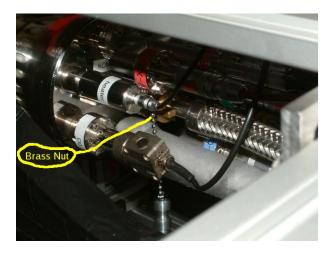


Figure 6.2: Brass nut holding the transfer line in Konti cryostat

- Close the pump bypass, switch on the pump by plugging the power cord and open the flow bypass (Fig. 6.4).
- Open the recovery line of the dewar to release any remaining pressure in the dewar. This valve should remain open during cryostat operation.
- Set the Bronkhorst flow regulator to some high demand value (e.g. 10,000).
- Close the flow bypass. Now the measured flow should slowly increase. Bear in mind that the viscosity of He gas is strongly temperature dependent, as long as the transfer line is warm, the flow will be small. The easiest way to stabilize the temperature and start a measurement is to use the AutoRun. Simply load an AutoRun file with a command TEMP <value>, <accuracy>, <timeout>, <ramp>

See for example 10K.lar. If you choose to stabilize the temperature manually, wait until the correct temperature is reached, adjust the flow and the needle valve and try to reach stability.

• Nevertheless, if you are interested in the lowest temperature possible with a Konti cryostat then

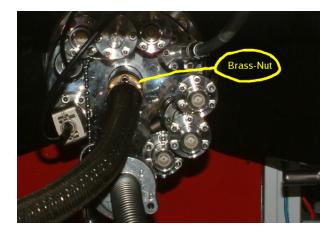


Figure 6.3: Brass nut holding the transfer line in Konti cryostat

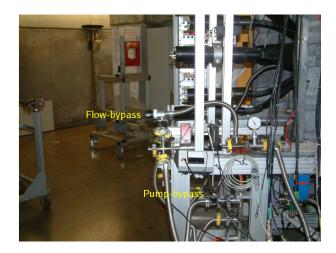


Figure 6.4: Location of Bypass Valves

- leave the cryostat at 10 K for more than an hour.
- for the real lowest temperature open the flow by pass, or set the Broncos demand value to 32,000.
- Set the demand temperature low, e.g. 1 K.
- Adjust the needle valve SLOWLY until the pressure is 0.4 bar with open bypass or 0.5 bar with closed bypass.
- For the LowTemp cryostat, please refer to section 7.4.

6.3 Warming up

The easiest way to warm the cryostat is to use the WARMUP command in an AutoRun. If you have to do it manually then

- Close the vacuum valves BPVX and BPVY.
- Ramp down the high voltage on the sample, the grids and on the ring anode.
- Set the Bronkhorst flow demand to zero and close the flow bypass valve if it is open.

- Turn off the He pump.
- Open the pump bypass valve.
- Set the demand temperature to 300 K. If the LS340 is not in ZONE mode, set the heater range to 5.
- Wait until room temperature has been reached and stable, with heater power below 25 %.
- Close the needle valve on the transfer line and remove it from the cryostat by turning the brass nut 180° counter clockwise, replace the transfer line by the aluminum plug and fasten the brass nut.
- If you intend to use a different He dewar remove the transfer line from the dewar. Otherwise, you may keep the transfer line in the dewar.
- If you wish to open the sample chamber, e.g. for sample change, follow the instructions in chapter 3.

6.4 He dewar change

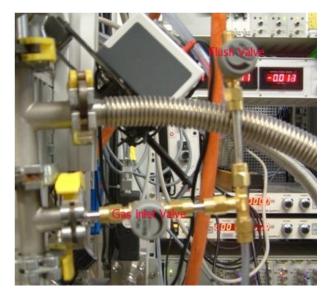


Figure 6.5: He gas inlet valves

The He dewar used to cool down the sample cryostat can be changed without warming up the cryostat to room temperature. To do this,

- Close the vacuum valves BPVX and BPVY.
- Ramp down all high voltages in the sample region, including the sample, the grids and the ring anode.
- Set the Bronkhorst flow demand to zero (and close the flow bypass valve if it is open).
- Set the demand temperature to 20 K or higher.

6.4. HE DEWAR CHANGE

- Wait until that temperature has been reached and then turn off the He pump.
- Open the main value of the He-gas bottle and flush value at least 20 s (see Fig. 6.5). Then close the flush value and open the He-gas inlet value.
- Wait until the pressure shows > 1.2 bar.
- Close the needle valve on the transfer line.
- Remove the transfer line from the cryostat by turning the brass nut 180° counter clockwise, replace the transfer line by the aluminum plug and fasten the brass nut.
- Remove the transfer line from the empty dewar.
- Gently dry and warm the transfer line with tissues and a hairdryer.
- Open the needle value of the transfer line fully (100 % on the electric ones, 3 turns on the manual one).
- Close the recovery value of the full dewar, this allows for pressure built up.
- Insert the transfer line into the full dewar slowly until you see the pressure increase (to about 0.1 0.2 bar).
- Pull back the transfer line by 5 cm. Now there is pressure in the dewar, and thus the transfer line will be flushed with He gas.
- Wait until you can feel (best with your cheek) the flow of cold gas coming out of the transfer line.
- Insert the transfer line fully in the dewar until it is a few centimetre above the bottom. Never empty a dewar completely, it is difficult and costly to fill a warm dewar.
- If the pressure gets too high (> 0.3) just open the recovery line a while.
- If the gas from the top of the transfer line gets too cold (ice forming) just close the top of the transfer line with your finger. If you burn your finger, you were too late.
- Once the transfer line is in the dewar, you can insert it into the Cryo. Turn the brass 180° counterclockwise, remove the aluminum plug and insert the transfer line fully until it stocks.
- Fasten the brass nut (clockwise).
- Close the gas-inlet, and open the flow bypass.
- Open the recovery line of the dewar.
- Close the main valve of the He bottle.
- Set the Bronkhorst flow regulator to some high demand value (e.g. 10,000).
- Close the flow bypass. Now the measured flow should slowly increase. Bear in mind that the viscosity of He gas is strongly temperature dependent, as long as the transfer line is at room temperature, the flow will be small.

- The easiest way to reach the correct temperature where the measurement should start is to let AutoRun do the job by running a .lar file with TEMP <value>, <accuracy>, <timeout>, <ramp>. see e.g. 10K.lar. Otherwise, if you are ill advised to do it manually, wait until the correct temperature is reached, adjust the flow and the needle valve and try to reach stability.
- Nevertheless, if you are interested in the lowest temperature possible with a Konti cryostat then
 - leave the cryostat at 10 K for more than an hour.
 - for the real lowest temperature open the flow bypass, set the Bronkhorst demand value to 32,000.
 - Set the demand temperature low, e.g. 1 K.
 - Adjust the needle valve *slowly* until the pressure is 0.4 bar with open bypass or 0.5 bar with closed bypass.
- For the LowTemp cryostats refer to chapter 7.4.

Chapter 7

Sample Environment

7.1 Introduction

In this section the sample environment for the LEM experiment is described. This includes the used nomenclature, a description of the different setups available and the used devices (device description, calibration tables, ranges, etc.). Before starting, we ask you to wear protective gloves whenever working with cryogenic liquids.

7.2 The cryostats

All cryostats used in the LEM sample environment are of the "flow" type, that is: a dewar with liquid helium is permanently connected and a pumping system sucks the liquid helium or cold helium gas through the system. An additional heating system, connected to the LakeShore340 temperature controller supplies a small amount of heat for final temperature stabilization. The amount of liquid or cold gas should be just enough to reach the temperature, too much will -if possible- be evaporated by the heater. The so-called Konti-cryostats (for short Konti) have one circuit for the cold gas, the LowTemp type have two circuits. In the latter, there is a small vessel, called phase separator, inside the cryostat that deals with the liquid helium flow. Hence, the LowTemp's can reach a lower temperature (~ 2 K at the base-plate), but are more difficult to operate and regulate. In all cases, care has to be taken that no moisture or air enters the cryostat while it is cold. An immediate block (freezing) will inhibit the cryostat from working and will cost you an hour or more to solve.

7.3 General description of the Konti flow cryostats

The Konti are the working horses for the LEM experiment. They have minimal consumption while being very robust. The typical temperature ranges (at the base-plate) of the different Konti cryostat is 3.8 K to 320 K. The upper limit is a protective measure due to indium seals in various positions in the Konti cryostats. The liquid helium consumption of a Konti is 2 l/hour (liquid) at 5 K, decreasing down to 0.06 ltr/hour at room temperature, almost following an inverse temperature curve, consumption (l/hour) = 10/T (K). Below 5 K the consumption increases more rapidly with decreasing temperature. The factory manual for the Konti's can be found at http://lmu.web.psi.ch/docu/manuals/device_manuals/CryoVac/Konti-2.pdf

The Konti flow cryostats consist of a cold finger, surrounded by a radiation shield. The socalled *base-plate* is directly screwed onto the cold finger (Fig. 7.1). There is a heater of 25 Ohm wound around the cold finger and two Si-diode thermometers are mounted on the back of the base plate. These diodes are called CF1 (or D1) and CF2 (or D2), normally these are connected to A and B input channel of the LakeShore340 temperature controller. A third diode (CF3 or D3) is mounted on the radiation shield, while a fourth one (D0) is factory mounted below the cold finger. Since we need the possibility to have the sample at high electric potential (+ or - 12.5 kV), there is a 6 mm thick sapphire plate between the base plate and the sample plate. The high voltage also implies that no thermometers can be mounted on the sample plate.



Figure 7.1: The low temperature part of the Konti

The effect of gluing the sample on the sample plate, while it is subjected to quit some heat load due to thermal radiation in the sample-chamber was measured in a separate experiment. A copper plate $(25 \times 25 \text{ mm}^2)$ was equipped with a heater and a calibrated thermometer as in Fig. 7.2. The effect of different glues on the thermal contact of the sample to the sample plate can be seen in Fig. 7.3. Nevertheless, be aware of the fact that the heat load on a $20 \times 20 \text{ mm}^2$ sample is of order of 0.1 W, resulting in a temperature difference between set-point as shown in Fig. 7.4. Note that for shiny samples this effect is much smaller.

The sample mounting procedure is described in Chapter 3. There is a radiation shield around the sample assembly. Inside this shield there are two guard rings, which can be put on a high electrical potential as well. However, this possibility is rarely used, but can be important when the electric field near the sample has to be decreased in order to avoid sparking. Calibration tables for the thermometers and parameters for the temperature regulation are automatically uploaded to the LakeShore340 depending on the configuration screen (Fig. 7.5).

Pumping of the He gas from the Konti is done by an Alcatel Dry Pump, 20 m^3/h via a Bronkhorst flow regulator. It is important to have the correct amount of flow through the cryostat to reach the demand temperature.

The He gas flow on the exhaust of the cryostat is controlled by a flow meter (Bronkhorst on the sketch) which measures the flow and regulates it via an automatic needle valve. The flow should be such that a slightly lower temperature can be reached, the heater, governed by the LakeShore340, then takes care of the last adjustments. In the Midas status page the flow is given in "Bronkhorst Units" (BH), 1 BH = 0.1 l/hour of He gas. Note that 1 l liquid helium is

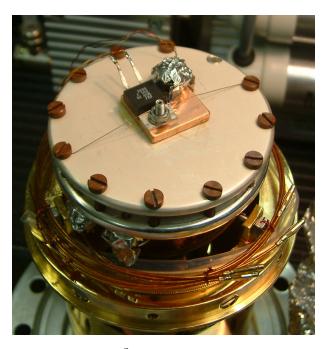


Figure 7.2: A copper plate $(25\times25~{\rm mm^2})$ was equipped with a heater and a calibrated thermometer

equivalent to 740 l gas at normal pressure and room temperature. In other words, 7400 BH is equivalent to 1 l liquid helium per hour. The flow controlled temperature regime is applicable for temperatures T > 4 K, where the relation between the flow and the temperature is given as

flow
$$\simeq (10^5/\text{temperature setpoint}) + 300.$$

In BH-units the flow can vary between 0 and 32000. Never use a flow smaller then 600, otherwise the transfer line will warm up, which kills the flow completely. For the lowest temperature the manual bypass valve needs to be opened (Fig. 7.7).

There is a pressure gauge between the Konti exhaust and the Bronkhorst flow meter which input is fed to the LakeShore340 so that this pressure can be seen from within the Midas system. It is there for diagnostic purposes. The transfer line, bringing the liquid helium from the dewar

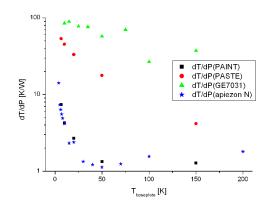


Figure 7.3: Heat resistance of different glues, silver-paint or Apiezon N provide a suitable option

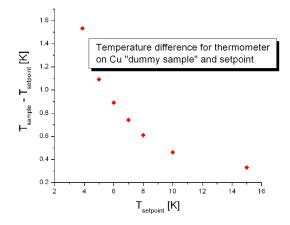


Figure 7.4: Temperature difference for thermometer

| Status LEM Setup Configuration D708 Last Updated: Thu May 24 2018 17:29:05 GMT+0200 (r Messages Detector selection Magnet selection Chat Sample Cryo WEW magnet LowTemp-2 Elog Sample Cryo WEW magnet LowTemp-2 Alarms Equipment/Options Fraable Angle 10 | | | | |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------|--|--|--|
| DBB Last Updated: Thu May 24 2018 17:29:05 GMT+0200 (r Messages Detector selection Magnet selection Chat Sample Cryo WEW magnet LowTemp-2 Elog Narms Equipment/Options Programs Spin Rotation Fnable Angle | | | | |
| Messages Detector selection Magnet selection Cryostat selection Chat Sample Cryo WEW magnet LowTemp-2 Elog Alarms Equipment/Options Programs Spin Rotation V Enable Angle 10 | LEM Setup Configuration | | | |
| Chat Sample Cryo V WEW magnet V Low Remp-2 Clog Alarms Equipment/Options Programs Spin Rotation V Enable Angle -10 | Last Updated: Thu May 24 2018 17:29:05 GMT+0200 (CEST) | | | |
| Alarms Equipment/Options Spin Rotation Finable Angle 10 | ectior | | | |
| Marms Equipment/Options Programs Spin Rotation ✓ Enable Angle -10 | ~ | | | |
| Programs Spin Rotation School Angle -10 | | | | |
| Spin Rotation Spine Angle -10 | | | | |
| History | | | | |
| | | | | |
| MSCB RA pulsing Enable | | | | |
| Config Keithley2600 Enable | | | | |
| Help Keithley2400 Enable | | | | |
| AutoRun BluePoint Enable | | | | |
| AutoRunNew FOM Enable | | | | |
| ConfigSetup MarconiAdret Enable | | | | |
| SampleCryo Keithley3390 Enable | | | | |
| Scaler Messages: | | | | |
| LemVac | | | | |
| Sep61PS64_Vac | | | | |
| ControlRoomScaler | | | | |
| LEMDocu > | | | | |
| SafetyAtPSI > | | | | |
| RunLog > | | | | |
| SearchDB A | | | | |
| AccStatus A | | | | |
| EventSettings / | | | | |

Figure 7.5: "LEM Setup Configuration" page

to the Konti contains a needle valve (manually for transfer line 1 and electric for 2 and 3), which should be set such that the pressure in the pumping line is between 0.8 atm and 0.9 atm for T> 20 K, and lower (down to 0.5 atm) for lower temperatures. For the transfer line 1 (manual) this means that for an optimal operation, the needle valve should be opened about 0.3 on the scale shown on it. For the lowest temperature (T < 5 K), it can be gradually reduced to smaller values (see also FAQ).

The temperature itself is regulated by a LakeShore340 temperature controller. We are using the ZONE setting facility of the LakeShore340 in order to ease the handling of parameters like PIDs and heater range. Here is the table with the currently used ZONE settings, with the temperature given as the upper limit of a ZONE (see also the LakeShore340 manual p. 6-5, 9-42 at http://lmu.web.psi.ch/docu/manuals/device_manuals/LakeShore/). The heater range is translated to power (the Konti-1 the heater has a resistance of 25 Ohm). All the parameters of the LakeShore340 and the Bronkhorst flow meter can be set manually if needed. However, it is strongly advised to use the TEMP command of the autorun sequence (see section 2.2.5) to take over these boring jobs.

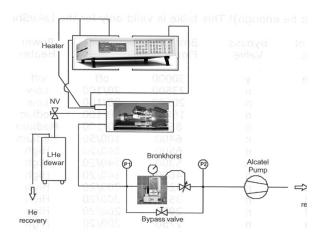


Figure 7.6: Cryogenic connections of the Konti cryostats

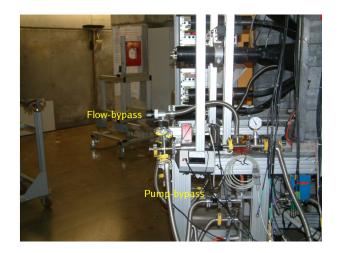


Figure 7.7: The bypass valves of the Konti setup

7.3.1 Konti-1 specific information

Konti-1 is the oldest of the Konti flow cryostats. Konti-1 has been equipped with extra electrical connections (at the cost of easy and quick operation) that enable the experimenter e.g. to apply electric fields to his sample and/or to measure electrical resistance at zero or at high potential, etc. The construction of the high vacuum voltage feed-troughs is such that they are in principle extendable ad infinitum.

The electrical connection are shown in Figs. 7.8-7.10. Since Konti-1 has many (high-voltage) wires running from room temperature to the cold finger, it is important to minimize the possible thermal contact these wires (in particular when they are unused) can make between the cold finger and the thermal shield. Best is to fasten the wires at the base of the thermal shield, see Fig. 7.11.

The base temperature (3.9 K on the base-plate, 4.0 K on the sample plate) has been reached with Konti-1 with flow bypass closed and a BH flow of 28000 (corresponding to 38 l/min) at a pressure of 0.6 bar. This corresponds to a helium consumption of 3.1 litre liquid He per hour. When such low temperatures have to be maintained for a prolonged period of time one better uses the LowTemp-1 or LowTemp-2, which will consume 'only' 2 to 4 litres liquid He per hour.

The thermometers in Konti-1 are

| Zone | Max T | PID | Heater Range |
|------|-------|-----------|--------------|
| 1 | 7 | 100/300/0 | 4 - 2.5 W |
| 2 | 10 | 100/200/2 | 4 - 2.5 W |
| 3 | 15 | 500/100/2 | 4 - 2.5 W |
| 4 | 20 | 500/100/2 | 4 - 2.5 W |
| 5 | 30 | 500/10/2 | 4 - 2.5 W |
| 6 | 320 | 500/10/2 | 5 - 25 W |

Table 7.1: The heater power range for the different temperature ranges.

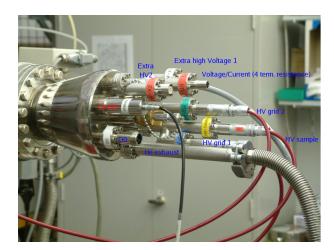


Figure 7.8: The room temperature part of the Konti-1.

- CF1: KC1CF1/D71714.
- CF2: KC1CF2/D71713.
- All other diodes are of the DT470 type.

7.3.2 Konti-2 specific information

Konti-2 and Konti-3 are identical twins. They are the easy going workhorses of the sample environment and provide the basic needs for the Low Energy Muon Experiment. The temperature range is between 4 K and 300 K. High Voltage connections to the sample and the two grids are provided. Due to the low mass of the inserts temperature sweeps between room temperature and base require less than one hour.

The thermometers in Konti-2 are

- CF1: KC2CF1/D43414.
- CF2: KC2CF2/D43412.

7.3.3 Konti-3 specific information

This cryostat is used mainly for small samples. The sample is mounted at the small round (10 mm diameter) copper sample plate screwed into the end of the long cold-finger tail (made of



Figure 7.9: Connection for the 4-point resistance measurement.

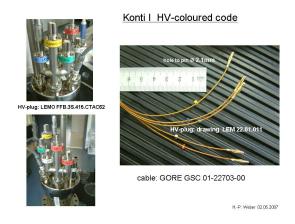


Figure 7.10: Color code for the extra HV connections.

copper ~ 355 mm length 6 mm diameter). The cold finger tail is surrounded by Al tube (thermal shield) 10 mm in diameter. All the parts are covered by Ni. Konti-3 is shown in Fig. 7.12. The cryostat is mounted using special long tube (Fig. 7.13). There are three peculiarities of Konti-3 related with the long tail, big thermal mass and big distance between sensors and the sample position.

- 1. The lowest temperature at the sample position is 6.56 K (Fig. 7.14).
- 2. There is a temperature hysteresis in the temperature range 120-160 K. Thermometers are calibrated to guess the temperature at the sample position in the direction of WARMING UP. When cooling down, the real temperature of the sample is LOWER by the value shown in the Fig. 7.15 (curve is to guide the eye).
- 3. At high temperature, the real sample temperature follows the sensor read back with a big delay tau (see Fig. 7.16 circles and Fig. 7.17).

The cryostat time constant can be approximated by:

$$\tau = 460 \times (1 - 1/(1 + T/91) \times 3)) \tag{7.1}$$

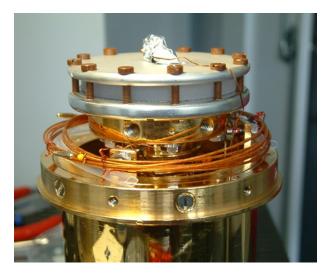


Figure 7.11: Cold part of Konti-1, note the fastening of the (spare) wires.

| Zone | Max T | PID | Heater Range |
|------|-------|-----------|--------------|
| 1 | 7 | 100/300/0 | 4 - 2.5 W |
| 2 | 10 | 100/300/2 | 4 - 2.5 W |
| 3 | 15 | 100/300/2 | 4 - 2.5 W |
| 4 | 20 | 200/200/2 | 4 - 2.5 W |
| 5 | 30 | 200/200/2 | 4 - 2.5 W |
| 6 | 90 | 400/100/2 | 4 - 25 W |
| 7 | 320 | 400/20/2 | 5 - 25 W |

Table 7.2: The heater power range and PID settings for Konti-1.

In the autorun one has to put the WAIT command with rather big value as a parameter, e.g. WAIT $1200\,$

The wait time depends (in the first approximation) on the Konti-3 time constant tau, the ramping rate, and the temperature tolerance delta. This can be estimated by:

$$t = \tau \times \ln(\tau \times \text{rate} \times \delta). \tag{7.2}$$

The practical (recommended) WAIT time for a ramping rate of 1 K/min and δ of 0.5 K (where δ is the maximum deviation from the set point and the real temperature) is shown in Fig. 7.16 by magenta line.

The thermometers in Konti-3 are

- CF1: KC3CF1/D70773.
- CF2: KC4CF2/D70776.

The Bronkhorst parameters for the Konti-3 are:

- BHFlow = $a/(T + T_{offset}) + b$
- $a = 1.5 \times 10^5$ (a is bh_flow_temp in the XML)

7.4. LOWTEMP-2 SPECIFIC INFORMATION



Figure 7.12: Konti-3 setup

- b = 300 (b is bh_flow_offset in the XML)
- $T_{\text{offset}} = -1.9 \ (T_{\text{offset}} \text{ is bh_flow_temp_offset in the XML})$

7.3.4 Konti-4 specific information

The Konti-4 setup is identical to the one of Konti-2 (see Section 7.3.2). The thermometers in Konti-4 are:

- CF1: KC4CF1/D56452.
- CF2: KC4CF2/D51453.

7.4 LowTemp-2 specific information

The LowTemp-2 consists of a long and hollow high-vacuum insert (Fig. 7.18) which ends in a cold finger with base-plate and sample mounting assembly. A flow cryostat can be inserted in this chamber (Figs. 7.19).

The cold gas or liquid from this flow cryostat then run along the inside of the cold finger, thereby cooling the sample. The heater again is mounted on the cold finger, this time of 10 Ohm resistance (**MAXIMUM CURRENT** = 1 **A**); the thermometers are on the back of the baseplate as in the Konti's. When mounting the flow cryostat insert into the UHV part and the UHV part into the sample chamber, take care that the holes for the screws of the MANGO



Figure 7.13: Konti-3 tube

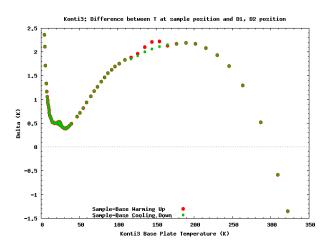


Figure 7.14: Difference between temperatures at the sample position (end of the tail) and the base plate

insert are pointing upward and that the big helium pumping line is pointing down, 45° to the left, since otherwise it will collide with the photo-multipliers.

The MANGO insert has a so-called phase-separator which holds liquid helium of 4.2 K (or colder). At the bottom of the phase-separator is a capillary with a needle valve. This capillary ends in the hollow high vacuum insert just behind the inside of the cold finger. By pumping this space liquid helium is sucked at of the phase separator and evaporates at low pressure (1 Torr), and thus at low temperature (1.2 K). In order to keep the phase separator at the desired 4.2 K the incoming helium is also evaporated directly and pumped by the secondary pump. The MANGO cryostat has been designed to reach the lowest temperature possible at the lowest helium consumption, but is not able to regulate temperatures between 1 K and 6 K, since the needle valve and the phase-separator are in thermal contact with the gas pumped from the space in the insert. Heating the cold finger then results in warmer gas -> a warmer needle valve -> larger helium viscosity -> smaller flow -> less cooling power -> higher temperature -> still warmer gas -> etc. The connection diagram is shown in Fig. 7.20 and the different parts at the room temperature side on Fig. 7.21.

Note that the phase-separation depends on gravity: the top must be upwards when using

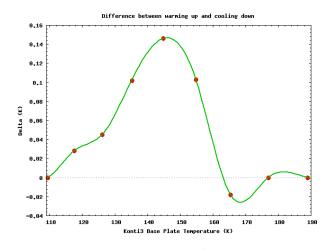


Figure 7.15: Konti-3 hysteresis

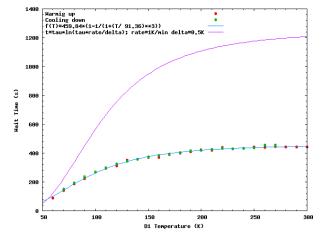


Figure 7.16: Konti-3 τ

the MANGO cryostat (Figs. 7.21 and 7.22).

The final position of the main (=lower) needle valve is rather crucial for the correct operation of MANGO, that is low temperature at moderate helium consumption. Therefore, a multiturn potentiometer has been mounted on the shaft of this needle valve. The potentiometer is connected to the potentiometer cable of the transfer line needle valve (Fig. 7.23). It's position is read by the SCS900, ADC1-2 (in the area) or by the LS340, channel C3 (in the tent).

Since the MANGO cryostat is relatively short and uses about 2 l liquid helium per hour, the 'room-temperature' part gets really cold and would collect a lot of ice. This would damage to O-rings and would inhibit the rotation of the needle valves. Four heaters (each 20 Ohm) have been mounted on the flange. They are connected, two in series and these sets parallel so that the equivalent resistance is again 20 Ohm. A thermal switch is connected in series with the heaters. The thermal switch closes below 13°C and opens above 30°C. The anti-icing heater should be connected to the anti-icing-heater-supply (Fig. 7.24). The maximum voltage on the MANGO heaters is 40 Volt, the pin layout of the supply is such that this voltage is automatically connected (Fig. 7.25).

The insert has been designed and built at the University of Birmingham. It had to be designed within the restrictions of the LEM UHV beam line. Different views of the LowTemp-2

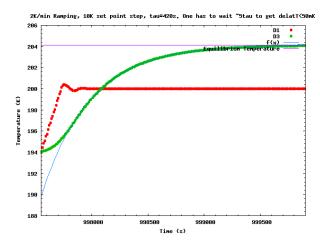


Figure 7.17: Typical example of the temperature relaxation for Konti-3.

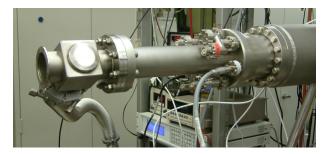


Figure 7.18: The UHV insert for LowTemp-2.

cooler are shown in Figs. 7.26 to 7.29.

Additionally, the back flow of the helium from the phase-separator is now used to pre-cool the transfer line and the needle valve. A new sinter improves the heat contact between the cold finger and the cold liquid/gas coming from the phase-separator. A turbine shaped section in the return line improves the cooling of the radiation shield of the UHV insert.

Installation of LowTemp-2 is almost the same as that of MANGO, but with a few important differences.

- Insert LowTemp-2 fully into the UHV insert. The two O-rings fit difficult. Apply a small amount of Apiezon-N grease on them and use adjustable-joint pliers (see figure) to fully insert LowTemp-2. Again, as with all phase-separators, gravity plays a role. The aluminum peace holding the needle valve should be horizontal with the needle valve to the right if you are facing the beam (see Fig. 7.30). Fasten the flange with four screws. (The screws with the springs as used sometimes, have no meaning at all). The distance should be 5.55 mm, not more and never less (Fig. 7.31).
- Connect the pumping lines via two Swagelocks (do not interchange them). The transfer line JC needs to be connected to the Bronkhorst flow controller, and the needle valve JC to the NV2 block (see schematic overview in Fig. 7.32).
- Make all electrical connections. Connect the thermometer cables as shown in Figs. 7.33 and 7.34. Do not forget to connect the two cables for the anti-icing heaters to the power supply (Fig. 7.35). One is connected directly to the LowTemp-2 cryostat, the other one in

7.4. LOWTEMP-2 SPECIFIC INFORMATION



Figure 7.19: The opening where LowTemp-2 is inserted (looking towards to beam). Note the TOP.

front of the Bronkhorst flow meter assembly. The maximum voltage on the LowTemp-2 heaters is 60 Volt.

- Close the return line of the helium-dewar.
- Slowly insert the transfer line into the dewar.
- Stop at the moment to pressure in the dewar increases and wait for the transfer line to nicely flush.
- Continue inserting the transfer line. Open the return line if the pressure gets too high and close the nozzle of the transfer line with your finger if cold gas is starting to flow through the transfer line. Insert the transfer line fully into the dewar.
- Now insert the transfer line into LowTemp-2. Slide the black sealing nut and the o-ring up to the thread around the outside of the sheath and tighten.

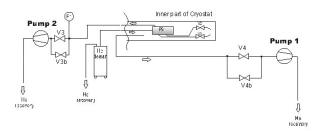


Figure 7.20: Schematic of the MANGO cooler.

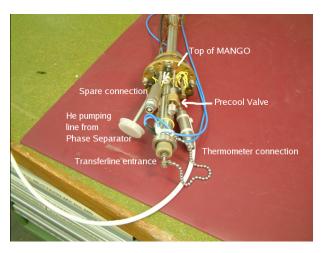


Figure 7.21: Room Temperature part with position of top upwards.

7.4.1 Operation

This cryostat operates in two modes; low and high temperature modes. The low temperature mode covers the temperature range 2.7 - 20 K, while the high temperature mode covers the temperature range 20 - 270 K. We start by describing the low temperature mode.

Low temperature operation mode (2.7-20 K)

In this mode the temperature is stabilized by the LakeShore340 heater only. To start working in this mode, cool down the cryostat following these steps

- Turn on the Anti Icing Heater Power Supply.
- Open the needle valve 3 turns (100 percent).
- Open the BH1/BH2 bypass (BH = Bronkhorst).
- Close the recovery line flush valve underneath the BH1 flow controller, and above the ACP120 (big pump). Switch on the pumps. The phase-separator temperature and sample temperature should drop rapidly. One can most conveniently observe the parameter in the sample cryostat status screen.

Wait until the temperature of the phase-separator is below 4.2 K and the sample is below 4 K (after about 10 - 15 minutes from the initial cooling), then:

• close both the BH1/BH2 bypass.

7.4. LOWTEMP-2 SPECIFIC INFORMATION



Figure 7.22: Room Temperature part with position of top downwards.

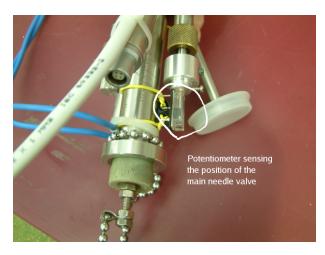


Figure 7.23: Potentiometer sensing the position of the main needle valve.

- set the BH1 flow to 11500, the BH2 flow to 700, and the temperature to 2 K.
- adjust the needle valve, NV, such that the flow after the ACP120 (big pump) is about 17 l/min He gas (this will be the final flow after about 2 3 hours of operation, initially it might be as high as 40 l/min He gas). It is important that there are no flow oscillations. In case flow oscillation are observed, play with NV and BH1/BH2 until they disappear. Typically only subtle tweaking is needed initially. LowTemp-2 will reach the real base temperature only after about 3 hours operation in the described regime (when starting from room temperature). Within these 3 hours the flow can gradually reduced (BH1, NV) until one reaches about 2.7 l/h LHe consumption (typical flows: 17 l/min after ACP20/28; 171/min after ACP120). An important note: for high temperature stability it is crucial that the flow through the BH1 (Siphon JC) is always much bigger than through BH2 (needle valve JC). This valve is just needed to keep the needle valve cold enough such that it cannot dump any heat into the phase separator.
- if the temperature starts oscillating, try first to reduce the BH1 flow slightly, e.g. from 11000 to 10500.

Some typical operation values are shown in Table 7.3.



Figure 7.24: Anti-Icing power supply (the color of the connectors have no meaning).



Figure 7.25: Thermal switch for anti-icing heater.

Some ice will form on the tubes, however the anti-icing heaters keep the o-rings and the needle valve knob at room temperature (Fig. 7.36). Note that the temperature of the sample is higher then the temperature of the base-plate (Fig. 7.37). After the base-temperature has been reached, higher temperatures, up to 20 K, can be obtained using the LakeShore340 temperature controller, without changing anything at the flow rates. The proper PID values are in Table 7.4

| Time | T_{sample} | Phase Sep Flow | sample flow | BH1 flow | BH2 flow |
|---------|---------------------|----------------|-------------|----------|----------|
| 30 min | 2.8 | 20 l/min | 20 l/min | 12000 | 700 |
| 120 min | 2.68 | 19 l/min | 15 l/min | 10000 | 700 |

Table 7.3: Typical operation conditions for the LowTemp-2 cryostat.

7.4. LOWTEMP-2 SPECIFIC INFORMATION

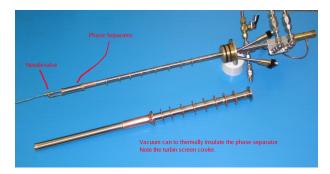


Figure 7.26: LowTemp-2 cooler

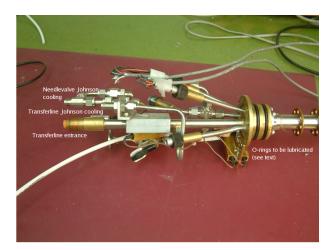


Figure 7.27: LowTemp-2 cooler, top part. Both, the transfer line Johnson cooling (JC) and the needle valve JC are pumping also the phase separator.

High temperature operation mode (20-270 K)

Although the LowTemp-2 has been designed for the use at low temperatures, normal operation, without changing anything the in flow and NV settings, is possible up to room temperature. If one wishes to run at temperatures above 20 K, go the high temperature mode in which the cryostat is operated as a mere flow cryostat.

- Set the BH2 to a flow of 0 and set the BH1 flow to a value of 1800. This results in reducing the flow at the ACP20/28 (small pump) to about 2 l/min.
- Adjust the NV such that you reach a flow of about 2 3 l/min at the ACP120 (large pump). It is very important to tweak NV and BH1 flow such that no flow oscillations are

| Zone | Upper Zone Temp | PID | Heater Range |
|------|-----------------|-----------|--------------|
| 1 | 8.5 | 250/400/1 | 4 - 0.86 W |
| 2 | 20.0 | 100/400/1 | 5 - 8.6 W |
| 3 | 60.0 | 250/400/1 | 5 - 8.6 W |
| 4 | 320.0 | 250/50/1 | 5 - 8.6 W |

Table 7.4: Table of PID values for the LowTemp-2 cryostat.



Figure 7.28: LowTemp-2 cooler, lowest part with sinter for maximal thermal contact

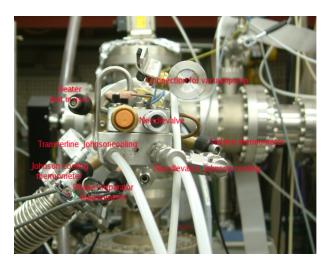


Figure 7.29: LowTemp-2 cooler, connections

present!

• If proper conditions are reached, the LakeShore340 heater is handling the rest for the temperature range from 20 K to room temperature.

Note that the flow values quoted above are correct for $T \sim 20$ K. At higher temperature these will generally be lower. This mode is mostly useful for measurements while warming up. However, in an autorun one can run in this mode going up and down in temperature between 20 - 150 K. When the temperature is higher than 150 K, cooling down to low temperature will require an increase in the Bronkhorst flow. For example, you can cool down from room temperature to 20 K by setting the Bronkhorst to a value of 9000. Once the required temperature is reached, this should be reduced back to 1800 for normal temperature stabilization.

7.4.2 Changing dewar

Although it is recommended to use a 250 litre He dewar so that a dewar change during the experiment can be avoided, it is possible to use 100 litre dewars (the typical life-time is about 27 hours) and to change the dewar while LowTemp-2 is rather cold.

7.4. LOWTEMP-2 SPECIFIC INFORMATION

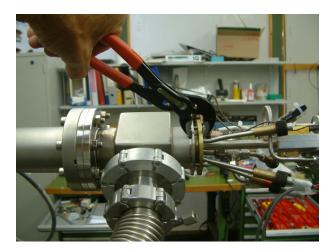


Figure 7.30: Exert force gently at different places of the circumference.



Figure 7.31: Fastened flange of LowTemp-2

- Stop both helium pumps and open their respective bypasses.
- Quickly pull out the transfer line (leave the nut and the O-ring on the transfer line).
- Immediately close the opening with a rubber cork.
- Take the transfer line from the 'empty' dewar.
- Gently dry the transfer line with a heat gun.
- Insert the transfer line into the 'full' dewar.
- Close after some time the output of the transfer line with your finger (or something else) to avoid forming of ice on the output of the transfer line.
- Remove the cork and quickly insert the transfer line.
- Be aware that the transfer line should be completely inserted before fastening the nut.
- Close the recovery line flush valves of the pumps.
- Start the pumps.

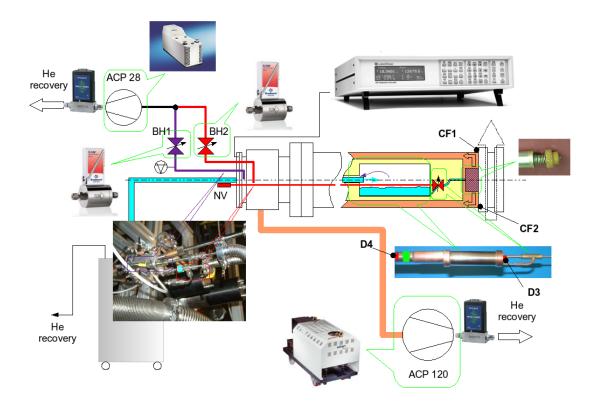


Figure 7.32: Schematic diagram of the LowTemp-2 cryostat and pumping system.

7.4.3 Warming up

- Switch off the pumps.
- Open bypasses.
- Quickly pull out the transfer line (leave the nut and the O-ring on the transfer line).
- Immediately close the opening with a rubber cork.
- Remove the transfer line from the dewar.
- When the cryostat is warm (> 270 K), remove the cork, put back the nut and the O-ring and close the opening with the rubber cork.

7.4.4 Maintenance

Bear in mind that the vacuum space has to be pumped about twice a year. This can be done by connecting the vacuum pump connector (Leybold KF6) to e.g. the rough pump of the vacuum unit in the tent. The same holds for the transfer line.

7.5. B PARALLEL MAGNET

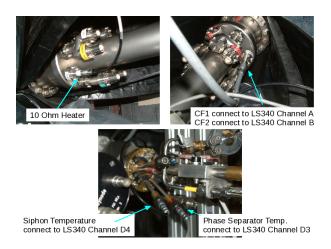


Figure 7.33: LowTemp thermometry connections needed

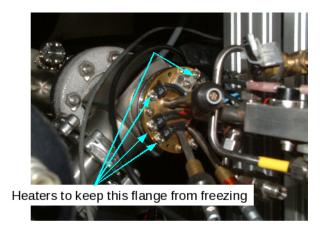


Figure 7.34: LowTemp-2 flange heaters

7.5 B parallel magnet

Remanent field after running degauss_danfysik: Bx = 0.01 G, By = -0.17 G, Bz = 0.12 G, see http://lem00:8000/LEM_Experiment/4901.

Figure 7.40 gives a field calibration table which results in

$$B(G) = 1.63 + 32.376 \times I(A)$$

In-plane field maps at the sample position are shown in Fig. 7.41 and the field along the beam axis (Fig. 7.42).

7.6 B perpendicular magnet (WEW)

Remanent field after running degauss_bruker: Bx = By = 0.01 G, Bz = -0.08 G, see http://lem00:8000/LEM_Experiment/4927.



Figure 7.35: Anti-Icing power supply (the color of the connectors have no meaning)

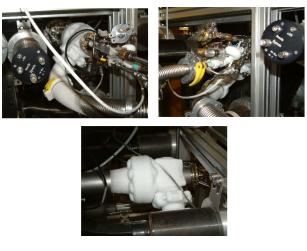


Figure 7.36: Typical view after some time

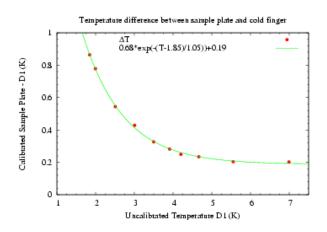


Figure 7.37: Temperature between the sample and the cold finger as function of the cold finger temperature. This difference is included in the calibration of the diodes - so the sensors show the temperature as expected at the sample position



Figure 7.38: Connection for pumping the vacuum space of LEMON



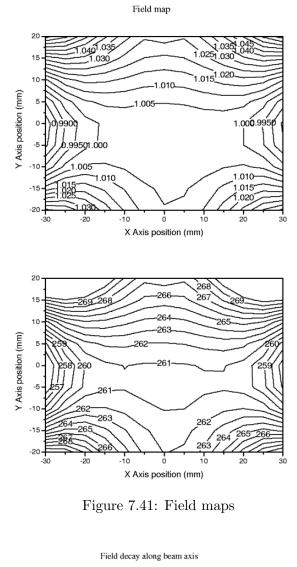
Figure 7.39: Correct way of closing the vacuum valve, including security wrap

4 coils 270 turns each, 1.8mm diameter Cu wires
 Shunt – 2.005mOhm

3. Zero Flux Meter x 50 = Current [A]

| | Calibration | |
|-----------|-------------|---|
| Field (G) | Current (A) | Τ |
| 0 | -0.05035 | Τ |
| 1 | -0.01946 | Τ |
| 2 | 0.01143 | Т |
| 3 | 0.04232 | Т |
| 4 | 0.0732 | T |
| 5 | 0.10409 | Т |
| 6 | 0.13498 | T |
| 7 | 0.16586 | Т |
| 8 | 0.19675 | Т |
| 9 | 0.22764 | Т |
| 10 | 0.25852 | Т |
| 20 | 0.5674 | T |
| 30 | 0.87627 | Т |
| 40 | 1.18514 | Т |
| 50 | 1.49401 | T |
| 60 | 1.80288 | T |
| 70 | 2.11175 | Т |
| 80 | 2.42062 | T |
| 90 | 2.72949 | T |
| 100 | 3.03836 | Т |
| 110 | 3.34723 | Т |
| 120 | 3.6561 | Τ |
| 130 | 3.96497 | Τ |
| 140 | 4.27384 | Т |
| 150 | 4.58272 | Т |
| 160 | 4.89159 | Т |
| 170 | 5.20046 | Τ |
| 180 | 5.50933 | Τ |
| 190 | 5.8182 | |
| 200 | 6.12707 | Τ |
| 210 | 6.43594 | Τ |
| 220 | 6.74481 | Ι |
| 230 | 7.05368 | Ι |
| 240 | 7.36255 | Τ |
| 250 | 7.67142 | Τ |
| 260 | 7.98029 | Ι |
| 270 | 8.28916 | Γ |
| | | |

Figure 7.40: Magnetic field calibration curve



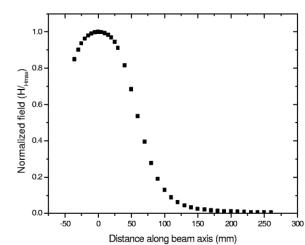


Figure 7.42: Field decay along the beam axis

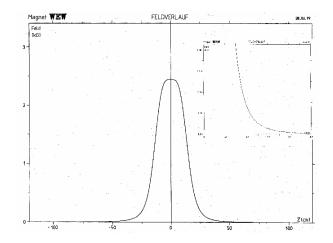


Figure 7.43: Field distribution of WEW

Chapter 8

High Voltage Table

Tuning the energy of incoming muons in LEM is done primarily by applyinh a high voltage (HV) onto the sample plate. Therefore, any manipulation on the sample that requires the use of direct contacts to it becomes complicated. For example, in order to run a current through or apply an electric field on the sample, the power supply and contacts have to be on the same HV as the sample. A simple way to achieve this is to place the power supply (or any other standard lab equipment or device) on an insulated platform outside the cryostat and bias both the sample and the device by the same HV. Although the idea is simple, its application requires serious safety and reliability considerations.

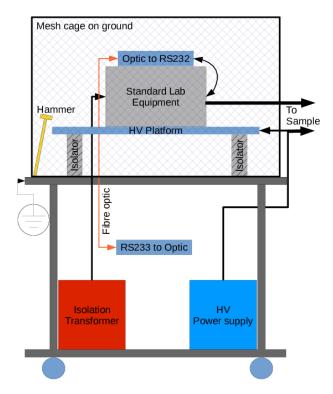


Figure 8.1: A schematic of the design of the HV table.

8.1 The Design of the HV Table

A schematic of the design on the HV table is shown in Fig. 8.1. A high volatge platform is isolated from a grounded table and placed inside a grounded cage for safety. The HV platform a fed via an isolation transformer mains electricity to power the standard lab equipment, which will be connected to the sample. The sample HV bias is connected to both, the sample plate and the HV platform to ensure that they are at the exact same HV at all time. Hence, even in case of HV discharge we expect that both will remain at the same bias. We have also implemente serial RS232 communication between our DAQ and the device using fibre optic connection. This is nessesary to isolate our DAQ electronics from the HV. Other safety precations include a grounded hammer which is realease when the cage is openned exposing the HV platform. A 1 G Ω resistor is connected between the HV platform and ground to discharge the HV platform within a second once the HV is turned off. The output from the device (e.g. current or voltage leads) are delivered via suitably insulated cables to the cryostat. During operation, the electrical power which feeds the isolation transformer is interlocked with the mains electricity for the HV power supplies used for all sample chamber equipment. This ensures that the HV table and device are turned off whenever an interlock is fired due to vacuum degradation or any other issues.

8.2 Connecting and Using the HV Table

Before we start, for your own safety and to comply with PSI's safety regulations, please do this only when accompanied by your local contact.

Below are a step by step instructions for connecting and preparing the HV table for an experiment. Since this setup is used only for few experiments, you will probably have to do this in preparation for your experiment.

• Start by making sure that the mesh cage is connected to the grounding copper plate at the bottom of the trolley, as shown in Fig. 8.2. Connect the copper plate to the platform of the spectrometer to make sure that the whole HV table is properly grounded.

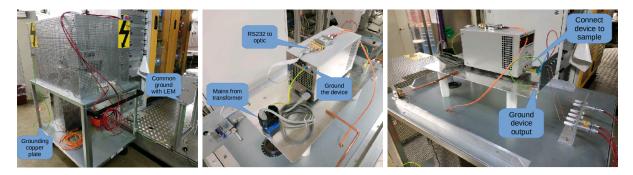


Figure 8.2: (left) Grounding connections. (center) Device grounding, power and communication. (right) Grounding connections.

• Remove the cage from the trolley and place the device that you want to use on the HV platform (Fig. 8.2). Currently, only serial RS232 devices are supported and only the current/voltage power supply TTi QL564P have been tested. Please do not connect any other device before consulting with your local contact.

8.2. CONNECTING AND USING THE HV TABLE

- Connect the metal body of the device to the aluminium HV platform (Fig. 8.2(center)). If relevant/available, also connect the ground output port to the HV platform (Fig. 8.2(center)) to make sure that the output of the device is biased at the same HV as the sample.
- Connect the RS232 to optic box to the RS232 port of the device (Fig. 8.2(center))
- Make sure that the RS232 to optic box and the device are both securely fastened to the HV platform, e.g. using cable ties.
- Place the mesh cage back on top of the trolley to enclose the HV platform and device properly. Make sure that the cage lifts up the grounding hammer and that it pushes the power switch. Make sure that there are nothing other than the fibre optic and the 1GΩ resistor going between the HV platform and any other parts of the trolley.
- Fasten the cage securely to the trolley using four butterfly screws, see Fig. 8.3(left).
- Plug the interlock switch into the SC interlocked mains electricity below the FUG HV power supplies. As an additional safety measure we use this interlock switch to power the transformer, and although not strictly needed, also the RS232 optical link. This way, HV interlocks in the SC and/or once the cage is lifted the devices on the HV platform will be immediately turned off.
- Finally, we need to make sure that the sample and HV platform of the cage are biased to the same HV at all times. To achieve this, make sure the the sample HV FUG is powered off, connect the sample HV cable (normally connected to the cryostat), to the HV plugs panel in the cage (on the right side of the panel, see Fig. 8.3(right)). Connect the next

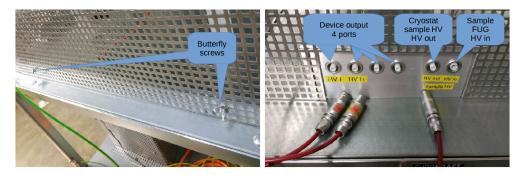


Figure 8.3: (left) Butterfly screws to secure the cage mesh. (right) HV panel.

plug from the right to the sample HV plug on the cryostat. Then connect the device ports as appropriate to the input ports on the cryostat.

At this stage, the experiment is ready to start as usual.

CHAPTER 8. HIGH VOLTAGE TABLE

Chapter 9

Other Technical Details

9.1 Thermal Contact Considerations

Figs. 9.1 and 9.2 gives an impression of the effect of different glues.

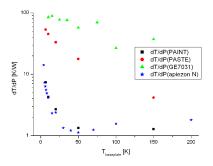


Figure 9.1: Heat resistance of different glues: silver paint or Apiezon N are OK. Nevertheless, be aware of the fact that the heat load on a 20×20 mm sample is of order of 0.1 W, resulting in a temperature difference between set-point and sample temperature as shown in Fig. 9.2. Note that for shiny samples this effect is much smaller.

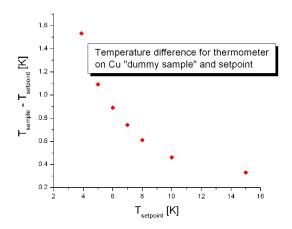


Figure 9.2: The temperature difference between the set-point and sample temperature.

9.2 Technical description of the automatic cryostat update

In this section the detailed update mechanism concerning the sample cryostat settings will be discussed. The ODB entry /Info/Sample Cryo defines the name of the cryostat that will be used. In sample_scfe.c frontend, there is a hotlink to this variable so that if it is changed, it will be transferred to the DD settings in the ODB, namely to /Equipment/SampleCryo/Settings/Devices/Lake340_S 340 Name. The device driver (LakeShore340.c) hotlinks the DD cryostat names and calls a routine called 1s340_cryo_name_changed, which is the routine which handles all the necessary changes, which are

- checks if the given name exists in the ODB DD subtree (see below for the details)
- read all settings from the cryostat specific ODB tree (e.g. DD/Cryos/Konti-I)
- set the proper heater resistance
- set the proper sensor types
- set the proper calibration curves
- set the proper zone settings

WARNING: Please be aware of the following:

- 1. The mlogger needs to be restarted if the ODB names are changed, otherwise the history will not be available.
- 2. If the number of temperature channels are different between cryostat settings (hopefully not). The frontend needs to be restarted, since the ODB will change!

For each sample cryostat there exists a subtree in the ODB which keeps all the necessary information. These subtree can be found under

/Equipment/SampleCryo/Settings/Devices/Lake340_Sample_0/DD/Cryos/CryoName where CryoName is one of the names given in Section 3.7. The cryostat subtree is structured as follows:

- 1. Heater Resistance : holds the resistance of the cryostat's heater.
- 2. **Sensor Type** : sensor types for the 10 possible channels (for sensor types see the LakeShore340 user manual p.9-33, INTYPE).
- 3. Calibration Curve : calibration curve number for the 10 possible channels (for details see the LakeShore340 user manual p.9-33 INCRV).
- 4. Channel : channel name (A, B, C1-C4, D1-D4) for the 10 possible channels.
- 5. Sensor Name : ODB Names of the 10 possible channels.
- 6. **Zone** : 10 Zone settings (see LakeShore340 user manual p.9-42 ZONE).

9.3 High voltage interlocks

This section describes the details of the high voltage interlocks employed at the LEM beam line and spectrometer. The HV interlocks are responsible for shutting down HV elements in case of pressure increase above 1×10^{-6} mbar to protect various beam line elements. Each vacuum chamber, MC, TC and SC, has its own interlock system. For example, when the moderator cryostat is warmed up, the MC pressure may exceed the preset threshold, resulting in activation of the MC interlock with the corresponding light turning red. Once the pressure goes down below the threshold the light will turn yellow. The one can release the corresponding interlock by pressing the black push button of the corresponding interlock board (Fig. 3.13). The light will then turn green and the HVs can be reset to the correct values.

Use hvEdit to load different "FUG_xxxxx_2-6ug_nosample.xml" (for a 2.6μ g/cm² carbon foil in TD) configurations with increasing transport voltage(=xxxxx). If everything is working properly, few ions are expected to hit the Trigger Detector (TD) and the Multi Channel Plate #1 (MCP1). The dark rate of both detectors is expected to be ~ 10 counts per second. The MCP1 is a muon detector which collects muons whose energy is too high to be deflected by the mirror. Test several transport settings until your desired experimental transport settings. If at some point, the read-back values fail to meet the set-point values, stop and get help.

CHAPTER 9. OTHER TECHNICAL DETAILS

Bibliography

- $\left[1\right]$ E. Morenzoni et al., Phys. Rev. Lett. 72 (1994) 2793.
- [2] T. Prokscha et al., Nucl. Instr. and Meth. A 595 (2) (2008) 317–331.
- [3] T. Prokscha et al. Appl. Surf. Sc., **172** 235 (2001).
- [4] P. Bakule and E. Morenzoni. Contemp. Phys., 45 203 (2004).