QUICK REFERENCES
DOLLY – πE1 AREA
HELIOX INSERT

μSR Facility 2017

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This document is available at:
https://www.psi.ch/smus/InstrumentDollyEN/QUICK_REF_HELIOX_DOLLY_June_2017.pdf

The sources are available in the LMU afs disk:
/afs/psi/project/lmu/Facility/Manuals/DOLLY/Heliox_DOLLY/2017

1 IMPORTANT FACTS

Contact person:

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In case Jean-Christophe is not around, contact Hubertus Luetkens (4450).

Main computers:

pc8533:	data taking console (No web browsing in this pc, please!!!)
pc8575:	users pc (to be used for users analysis)
pc9573:	pc to control the beamline
mpc1364:	pc to control the spinrotator II
psw411:	backend workstation.

2 THE HELIOX INSERT - OVERVIEW

The `Heliox` cryostat used on Dolly is a commercial Oxford Instrument ^{3}He fridge VT. A quick documentation can be found at http://www.oxford-instruments.com/products/cryogenic-environments/3he-inserts/helium-3-refrigerator-without-1k-pot-helioxvt. The VT `Heliox` is directly inserted in the `Variox` cryostat used as the 1 K pot.
A scheme of the important part of the insert is shown below.

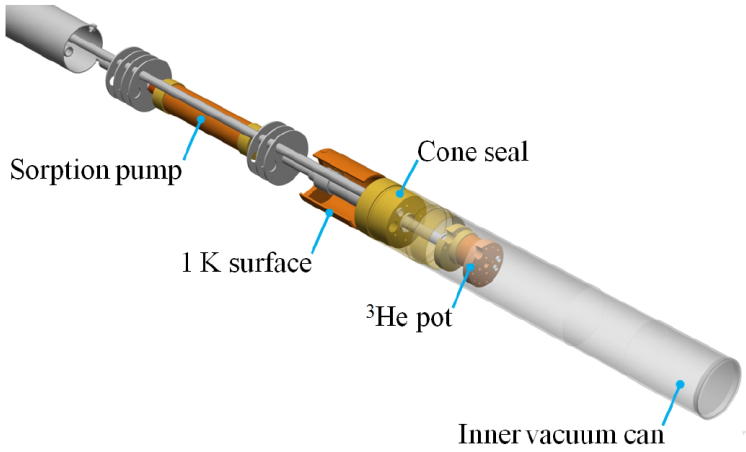


Figure 1: The HelioxVT "1 K surface" is cooled to below 2 K by the `Variox` Cryostat through exchange gas, and acts as the 1 K pot for the ^{3}He refrigerator. The inner vacuum chamber (IVC) is sealed using a silicon based paste applied to a cone seal.

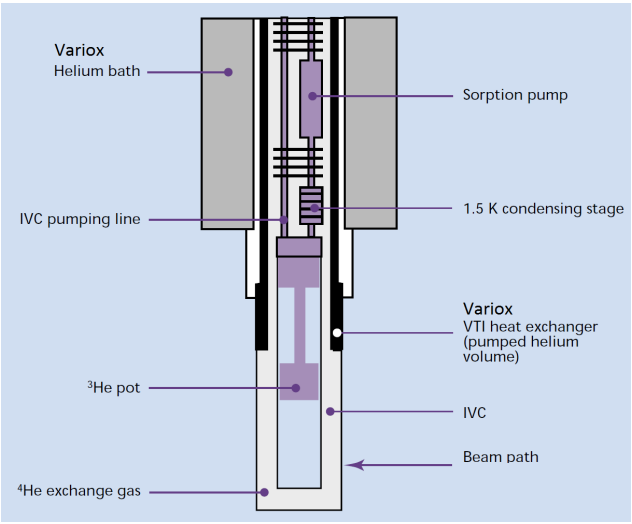


Figure 2: Schematic view of the bottom part of the `Variox` cryostat with the `Heliox` insert in position. The sample is attached on a copper rod fixed to the ^{3}He-pot. The ^{4}He excange gas in the IVC (100 mbar at room temperature) remains during the full operation. The exchange gas inside the ^{3}He insert is automatically condensed at low temperatures by a charcoal (activated carbon)

After cooling the insert, the exchange gas inside the ^{3}He insert is then automatically condensed

by a charcoal (activated carbon), whereas the exchange gas in the IVC remains in it during the full operation. The ^{3}He gas contained in a small dump sitting on top of the insert is then condensed at around 1.5 K. Once the ^{3}He pot has reached a stable temperature and condensation is completed, the sorption pump will start to cool the ^{3}He pot and the sample to below 300 mK.

To control the temperature of the ^{3}He pot between the lowest attainable temperature and up to 2 K, the temperature of the sample is just regulated by changing the pumping efficiency of the sorption pump (i.e. the `Mercury` temperature controller will automatically heat the sorption pump and change its temperature in order to regulate its pumping power and ultimately change the sample temperature).
For higher sample temperatures and up to 15 K, it is most efficient to apply electrical power directly to the ^{3}He pot heater. To provide cooling the ^{3}He sorption pump is automatically warmed to 15 K to partially release the ^{3}He charge from the sorption pump, thus providing a thermal link (through the gas) between the 1 K pot and the ^{3}He pot.
For even higher temperatures, the temperature of the `Variox` should also be changed, as the exchange-gas absorbed in the charcoal will be released.

The `Heliox`VT sorption pumped ^{3}He refrigerator is "single-shot" in the sense that there is only a finite quantity of ^{3}He condensed into the system. Experimental heat loads are taken up by the latent heat of vaporisation of the liquid as it is transformed into vapour. Once all of the liquid ^{3}He is consumed in this way, the system will start to warm. To cool the system again requires the "regeneration" of the ^{3}He sorption pump (i.e. the sorption pump will be heated up to about 32 K) such that the adsorbed gas is freed and can be re-condensed into the ^{3}He pot.

3 PRINCIPLE OF OPERATION

The heart of the temperature control is the `Mercury` temperature controller.

The electrical connections from the `Variox` and `Heliox` must be directed to the `Heliox Mercury`. This is performed by putting the switches of the switching box in the appropriate configuration.

This `Heliox Mercury` controller will on one side control the temperature of the VTI (i.e. `Variox`) by controlling its heater and the pressure in the VTI.
The closed loop control of the `Variox` temperature is achieved by servo-controlling its VTI heater.
The closed loop control of the pressure in the VTI is performed by measuring the pressure in front of the main ^{4}He pump and servo-controlling the needle valve of the `Variox`.
In normal operation, the temperature and pressure of the VTI (`Variox`) are set automatically by the `Mercury`. They must be overwritten when warming-up and cooling-down the ^{3}He-insert and measuring at temperature higher than 15 K.

On the other side, the `Heliox Mercury` will take care about the ^{3}He condensation and temperature stabilization of the ^{3}He insert.

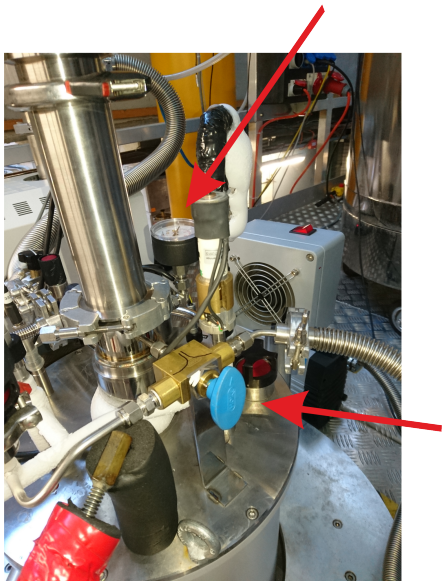
Note that even though the `Mercury` has the capability to measure the Helium and Nitrogen levels, these are always measured by the "Cryomagnetic" device *LM510*. The output of this device is used to control the automatic refilling of the Helium and Nitrogen into the `Variox`.

3.1 Initial Cooldown

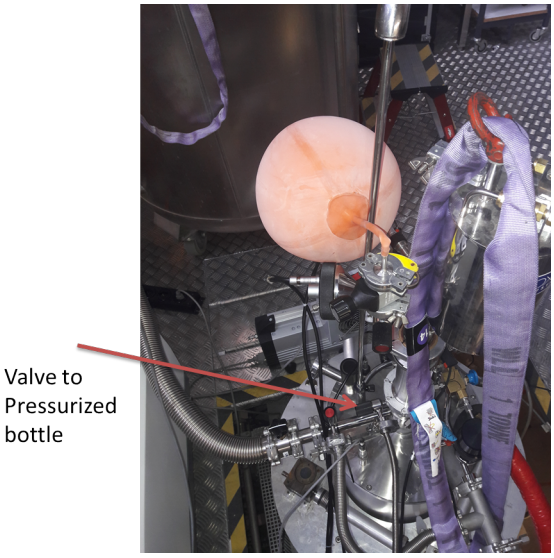
The mounting of the sample is described in Section 4.

Once loaded, the insert is cooled by the VTI (`Variox`) down to around 20 K using exchange gas.

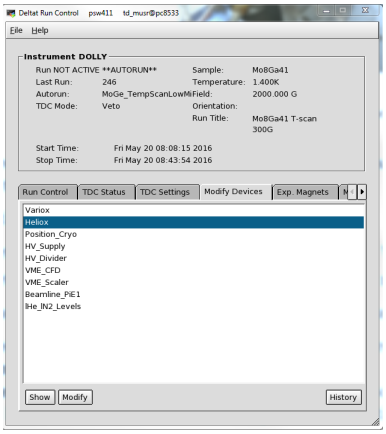
- Be sure to first evacuate the IVC using the IVC-pump by opening the 3-way blue-valve adequately (see Figure below).



- Close the 3-way blue-valve and insert 100 mbar of ^{4}He-gas using the black-valve connected to the pressurized ^{4}He bottle. One can check the pressure with the manometer (see Figures above and below).



- Be sure that all the electrical cables of the `Heliox` are connected and that the switch-box is set to the `Heliox Mercury`.
- Start the cool-down procedure. In `Deltat` go to `Modify Devices` then to `Heliox` and click on `Modify`. The procedure has to be done in two times :
  i) First set the setpoint for the `Heliox` and for the `Variox` temperatures to 10 K. Then click on OK.



ii) Second set the pressure setpoint for the VTI to 35 mbar by going back to the `Heliox` tab.

- Note that if one changes the temperatures and the pressure at the same time, the pressure will not be taken into account and the `Variox` temperature will decrease too slowly.

- After about 1 hour, the `Heliox` will reach a temperature around 30 K. As this procedure consumes quite a lot of helium, please try to keep it as short as you can.

- At this point, the first condensation can be initiated by simply setting the setpoint of the `Variox` to 1.4 K and the one of the `Heliox` to 0.1 K.
  The `Mercury` will change the status of the `Heliox` to `REGENERATE`.
  In the `REGENERATE` mode, the `Mercury` will automatically:

  - Set the pressure setpoint for the VTI to 7 mbar (corresponding to the parameter `OPTIMAL_NV_RG`)
  - Set the setpoint of the sorption pump to 32 K (parameter `He_3SORB_REGEN`)
  - Switch off the `Variox` Heater.

  The temperature of the `Heliox` will go down rather quickly down to 2 K (about 30 min) and after the cooling rate will decrease.

- When the temperature reaches 1.85 K (parameter `CONDENSED_TEMP`), the first step of the condensation is finished and the sorption pump heater is automatically switched OFF. The status of the `Heliox` remains to `REGENERATE`.

- Finally, when the `Heliox` temperature drops below the desired setpoint (if higher than 0.3 K) or below 1.65 K (parameter `ACCEPT_BASE`), then the status of the `Heliox` switches to `LOW TEMP` and the `Mercury` automatically sets the pressure setpoint for the VTI to 1.5 mbar (parameter `OPTIMAL_NV_LT`).

  At this point, assuming that the setpoint is 0.1 K, it will take less than 10 minutes to reach temperatures below 0.3 K.

  Note that 1 mbar corresponds roughly to 1 l/min of Helium measured by the Hasting flowmeter.

Note that the time needed to cool down the insert from room temperature and 0.3 K is about 2.5 hours. Successive regeneration of the ^{3}He will be quicker.

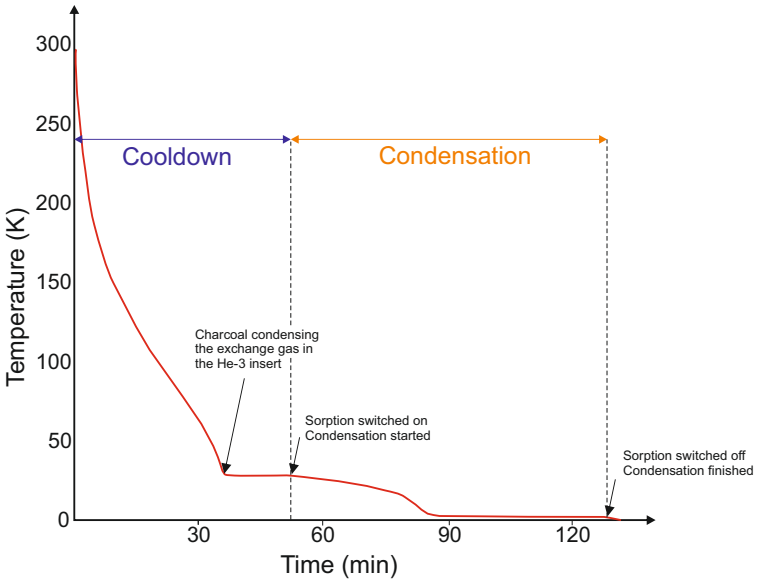


Figure 3: Typical time dependence of the `Heliox` temperature during an initial cooldown procedure.

3.2 Changing the temperature between base and 2 K

The temperature can be easily changed between the base temperature (about 0.25 K) and 2 K (parameter `CMODE_XOVER`).
In this temperature range, the `Mercury` will keep the status of the `Heliox` to `LOW TEMP`.

- Set a new setpoint for the `Heliox` rather in `Deltat` or in an autorun sequence (see section 5). The `Variox` temperature **DOES NOT** have to be changed.
- The `Mercury` will servo-control the temperature of the sorption pump (and therefore change its pumping power) in order to reach the desired setpoint temperature at the ^{3}He-pot.
- During this process the status of the `Heliox` will remain as `LOW TEMP`. The pressure setpoint for the VTI will still be 2 mbar (parameter `OPTIMAL_NV_LT`) and the `Variox` Heater will remain off.

In this temperature range, the setpoint can be changed back and forth as desired.

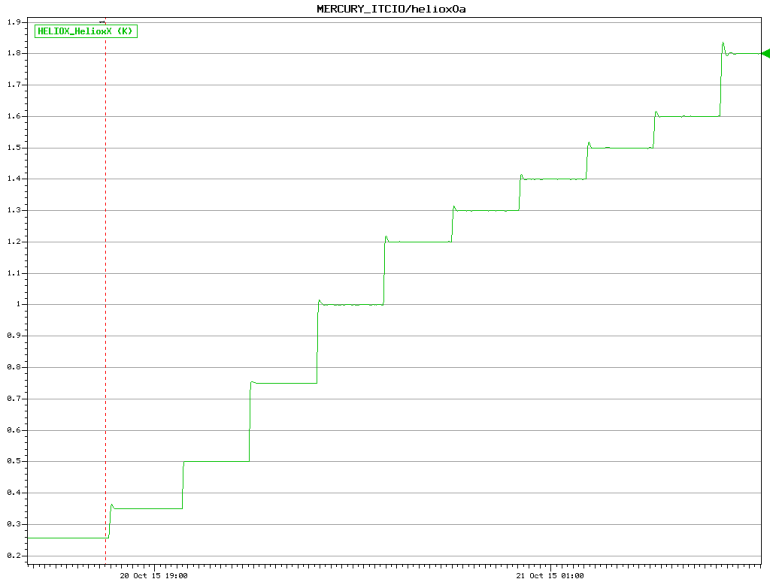


Figure 4: Typical temperature behaviour in the `LOW TEMP` regime.

3.3 Going above 2 K (and below 15 K)

When going above 2 K, the `Mercury` will change the status of the `Heliox` to `HIGH TEMP`.

- Set a new setpoint for the `Heliox` rather in `Deltat` or in an autorun sequence (see section 5). The `Variox` temperature **DOES NOT** have to be changed.
- The `Mercury` will automatically set the temperature of the sorption pump to 15 K (parameter `He3_SORB_HT_CONTR`). This is done to have some ^{3}He gas available for thermal link.
- The `Mercury` will servo-control the heater located at the ^{3}He-pot to reach the desired temperature.
- The pressure setpoint for the VTI will be set to 5 mbar (parameter `OPTIMAL_NV_HT`) and the `Variox` Heater will remain off.

Note that when crossing 2 K, the `Mercury` will automatically switch the main reference sensor (i.e. from RuO at low temperatures to Cernox for the high temperatures).

Note also that the switching from the `LOW TEMP` to the `HIGH TEMP` regime will result to a slight overshoot of the temperature.

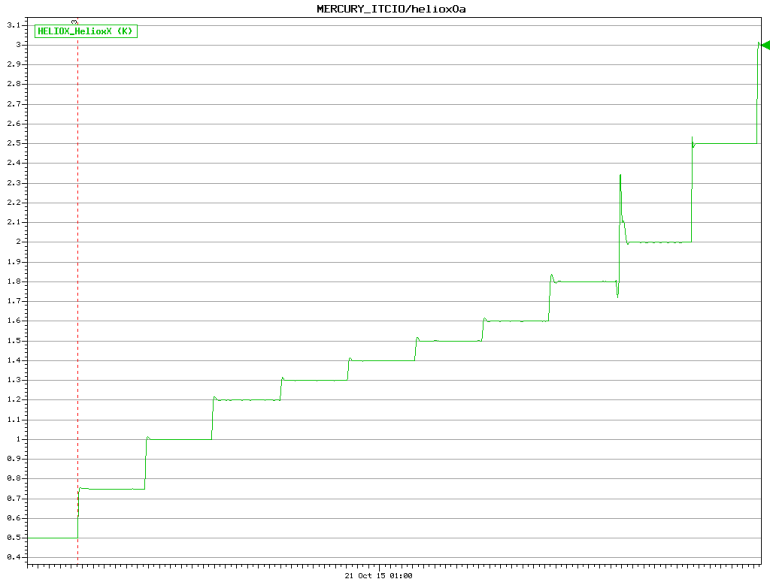


Figure 5: Typical temperature overshoot when going from the `LOW TEMP` to the `HIGH TEMP` regime.

In this temperature range, the setpoint can be changed back and forth as desired.

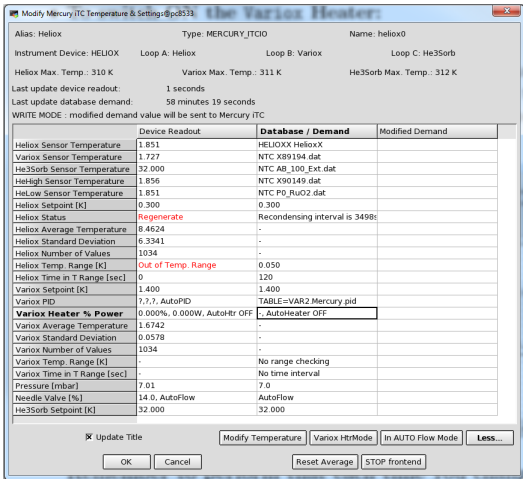
3.4 Going above 15 K

When going above 15 K, the `Mercury` will keep the status of the `Heliox` to `HIGH TEMP`, but the exchange gas trapped in the charcoal will slowly begin to be released. Therefore a temperature gradient can exist between the ^{3}He-pot and the sample which will also be cooled by the exchange gas.
So, one also has to change the temperature of the `Variox` (VTI) to minimize this gradient.

- Set a new setpoint for the `Heliox` and the `Variox` (use the same setpoints) rather in `Deltat` or in an autorun sequence (see section 5).
- The `Mercury` will automatically set the temperature of the sorption pump to 15 K (parameter `He3_SORB_HT_CONTR`). This is done to have some ^{3}He gas available for thermal link.
- The `Mercury` will servo-control the heater located at the^{3}He-pot to reach the desired temperature.
- The pressure setpoint for the VTI will be set to 5 mbar (parameter `OPTIMAL_NV_HT`) and the `Variox` **Heater will be at this point automatically turned off**.
- **To switch ON the `Variox` Heater:**

  Interactively:

  In the tab Modify Devices choose the `Heliox` entry and hit the buttons `Modify`. In the pop-up window hit the button `More...`. Be sure that the `Variox Heater % Power` reports `AuthoHeater`. If it reports `AuthoHeater OFF`, switch the heater ON with the buttons `Variox HtrMode`.



  Remember to perform that each time you change the temperature setpoint.

  In an Autorun sequence:

  Within an autorun sequence, a change of the setpoint in this regime will be given as:

  `SET HELIOX 20 1.0 120 20 0.5 120 1`

  - where the 3 first parameters correspond to the setpoint (20 K), temperature tolerance (+/-1 K) and waiting time (120 sec within tolerance) for the ^{3}He-insert,
  - the next 3 parameters are the the setpoint (20 K), temperature tolerance (+/-0.5 K) and waiting time (120 sec within tolerance) for the `Variox`,
  - and finally the 7th parameter is the control of the `Variox` Heater (1: Heater ON).

3.5 Coming back to below 2 K (parameter REGEN_ABOVE)

When the Heliox is set at a temperature above 2 K (i.e. in the HIGH TEMP Mode) and if a new setpoint below 2 K is given, then the system will automatically recondense and finally go to the desired temperature.

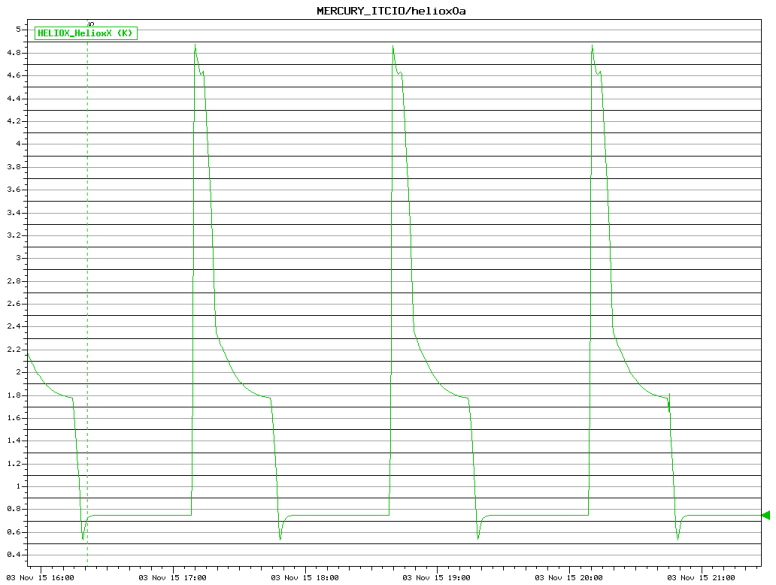


Figure 6: Cycles performed to obtain Field-Cooling conditions on a superconductor. The cycles were performed between 0.75 and 4.5 K. Note that when the setpoint was changed from 4.5 to 0.75, the system automatically recondensed and finally settle to the new stpoint were a run was performed.

4 SAMPLE CHANGE

As one should remove the `Heliox` Cryostat from the `Variox` Cryostat in order to change the sample, one has to note that the sample change can be painful and takes about 5 hours.

4.1 Removing the Heliox cryostat from the Variox cryostat

1. Put the `Variox` temperature higher than **80 K** (see Section 3.4) and wait until the `Variox` reaches this temperature.
2. Close the Dolly beamline valve either manually (by pressing a button on the touch screen located at the entrance of the area) or remotely from a computer (pc9573).
3. Go into the area and disconnect the `Heliox` insert cables and place the crane chop over the `Heliox`. The strap must be loosely tensioned.

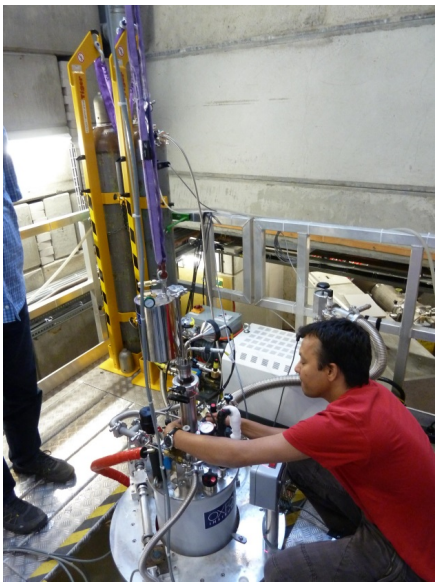


Figure 7

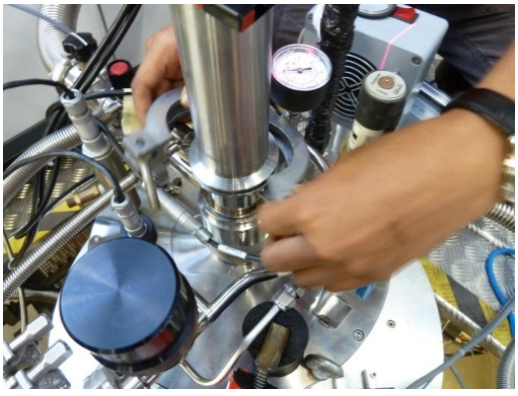
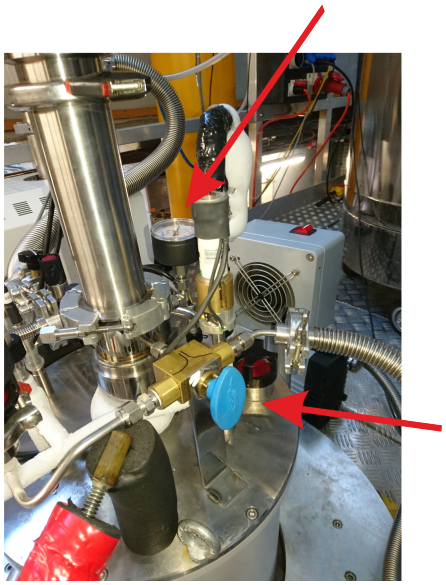


Figure 8

4. Disconnect the Helium balloon (small line) and replace it with the corrugated tube.

Figure 9

5. Open the lower clamp of the sample chamber.
6. Carefully fill the IVC with helium gas until ambient pressure. Be sure to monitor constantly the available manometer.



7. As soon as the ambient pressure is reached, slightly pull the `Heliox` out of the `Variox` with the crane. For this, center the `Heliox` with one hand using glove and operate the crane at slow speed with the other hand.
   Immediately interrupt the process in case the `Heliox` get stuck.

Figure 10

8. Close the opening of the `Variox` with the blind flange insert.
9. Stop immediately blowing He-gas in the `Variox`.
10. Slid a KF50 O-ring over over the `Heliox` before inserting it in the Service Frame. Then fix the `Heliox` with a KF50 clamp to the Service Frame.

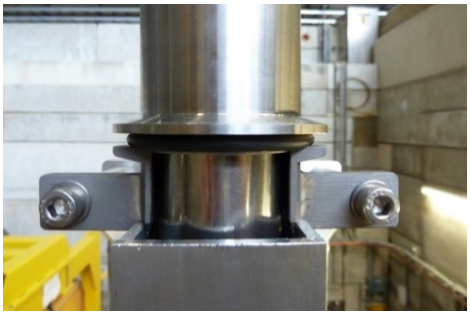


Figure 11

11. Connect the corrugated tube of the turbo pump unit. Pump the line before opening the vent valve to the `Heliox` sample chamber.
12. The ^{4}He gas contained into the balloon may now be transferred to the `Heliox` by opening the appropriate valve. This gas is used to impede water condensation when venting the `Heliox` sample space. Only after breaking the isolation vacuum a warm up of the `Heliox` is possible .

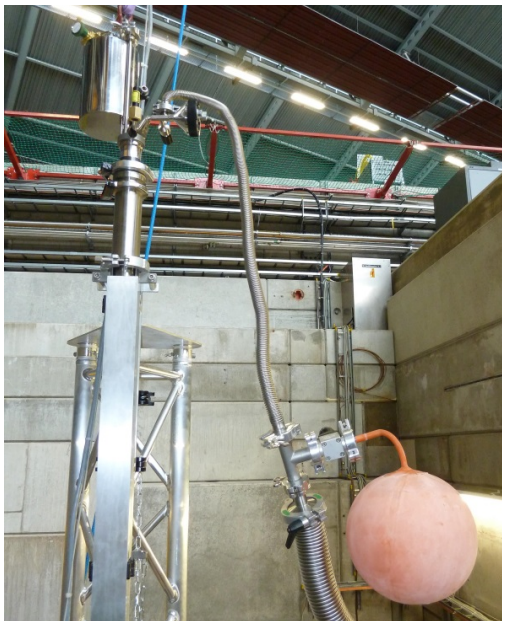


Figure 12

13. One can warm up the sample area with the hair dryer installed on the service frame.

4.2 Opening the Heliox sample space

One should use the special two part clamp.

1. Tighten the screws on the bracket.
   The tail should be held with one hand to prevent a bending of the `Heliox`.

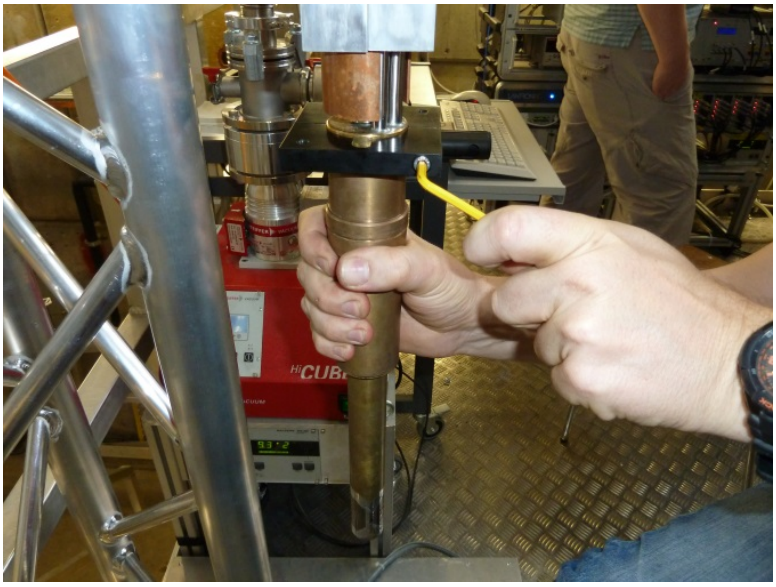


Figure 13

2. Spread the brackets using the bolts and pull off the tail. **The Tail should be held with one hand in order to prevent it to a fall down.**

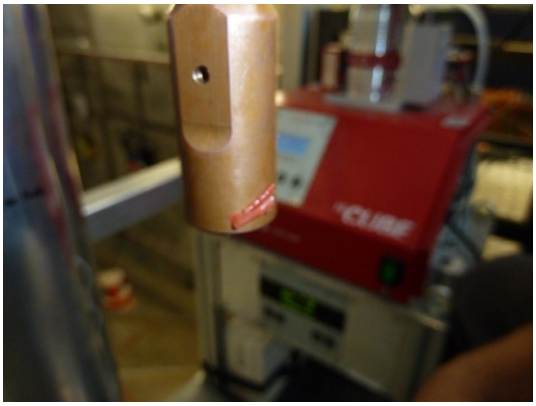
Figure 14

4.3 Cleaning the seal

1. Carefully remove the old still adherent red sealing paste.
   One can use his fingernails (this works very well) or, alternatively, a plastic part as a scraper (or with some ethanol).
   Sandpaper or harder object should never be used.
   Note that if you heat too much the sealing when removing the condensed ice, the cleaning process will be difficult.

Figure 15

2. Check the inner part, it sometimes happens that some sealing fall down and adhere there. If there are some, remove them as they may create a touch during the cooling and prevent a proper condensation of the ^{3}He.



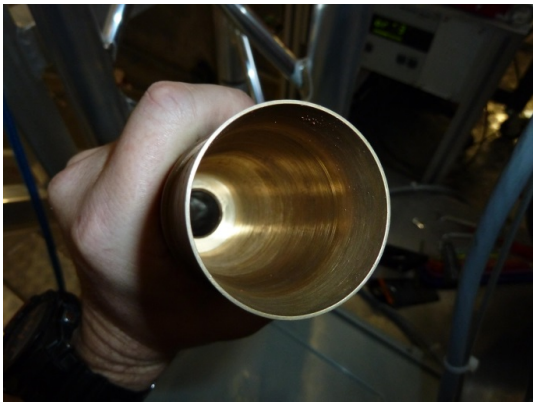


Figure 16

4.4 Sample change, check list

1. Change your sample
2. Check the alignment of the sample by using the laser at the top of the `Heliox`.

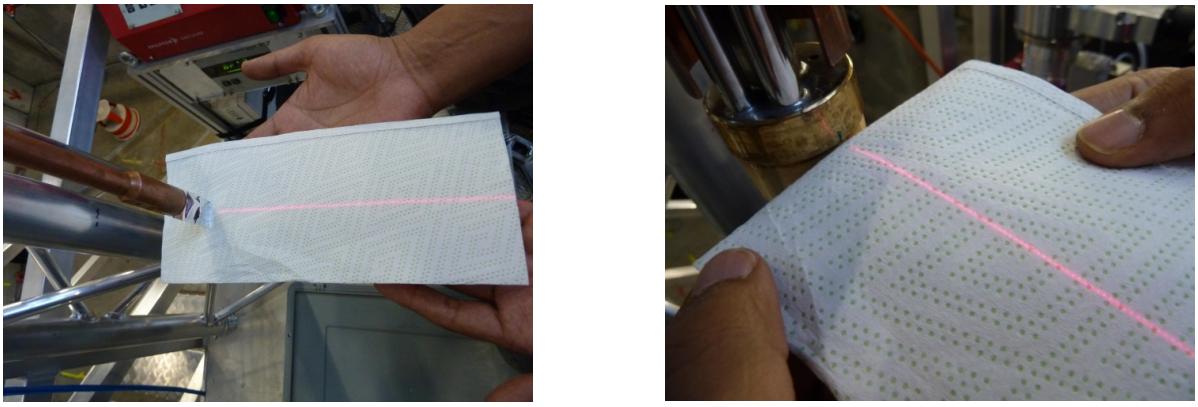


Figure 17

3. Check if the mark at the top of the sealing cone is clearly defined. If not renew it.
4. Carefully clean the opening of the red paste tube (CAF-paste) from any hard residue.

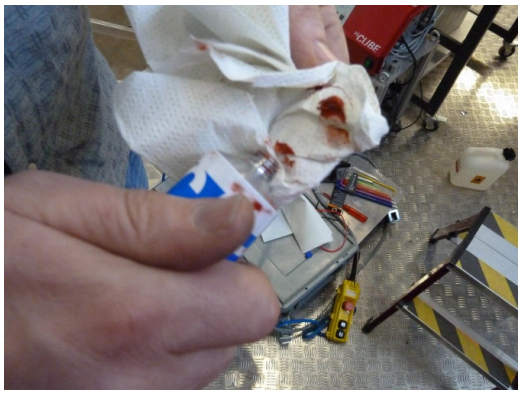


Figure 18

5. Coat the upper half of the sealing cone with a layer of approximately 3 mm thick CAF-paste.
6. Set up the tail by aligning the marks on the tail and at the top of the sealing.

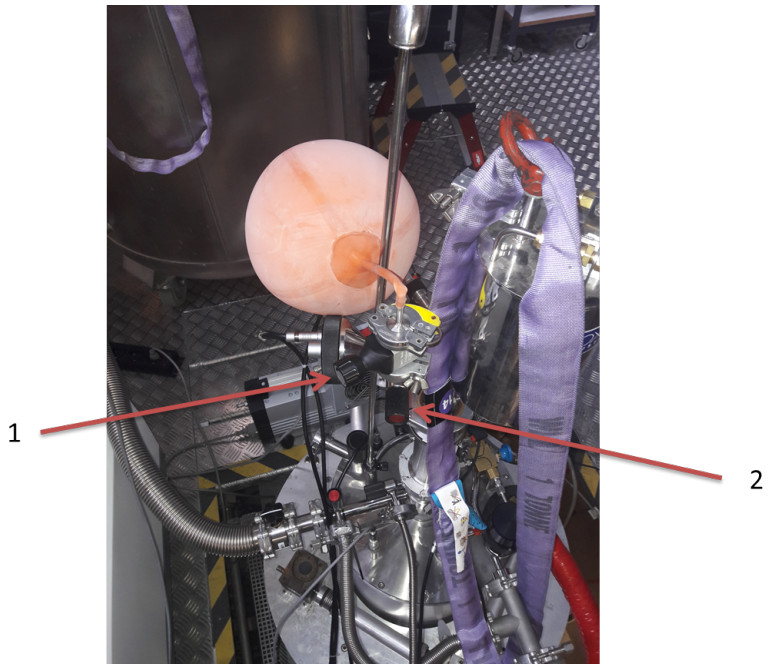
Figure 19

7. Be sure that the valve for the balloon is closed and turn the vacuum pump on.

8. Open the main vacuum valve and pump the ^{3}He-insert until the seal is completely dried (about 30 min).
   Note that the position 309 of the turbo pump unit indicates the speed of the pump rotator.

4.5 Inserting the Heliox into the Variox

1. Do not forget to close the `Heliox` valve and to disconnect the corrugated line of the turbo pump from the heliox. One can diconnect it at the T connection with the Helium balloon.
2. Bring the `Heliox` close to the sample space using the crane.
3. Open the lower clamp of the sample chamber.
4. Fill the `Variox` IVC carefully using He gas.
5. Once He-gas start blowing out of the O-ring, remove the blind flange insert and replace it with the `Heliox` carefully using the crane.
6. Immediately stop blowing He-gas and remove some of it with the avilable pump and the 3-way valve (leave about 100 mbar).
7. Reconnect the electrical cable.
8. Replace the remaining corrugated tube with the Helium balloon connected to the manometer.



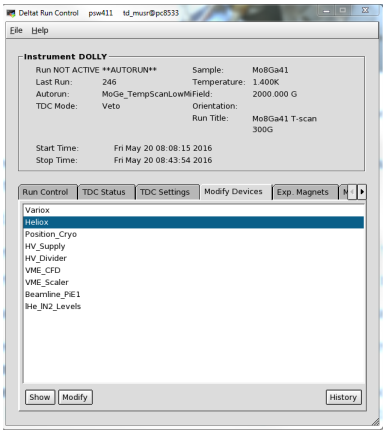
9. In order to have a thermal coupling at high temperature one has to introduce about 1 cm^3 of ^{4}He exchange gaz. One can try to blow a little bit of the ^{4}He contained in the balloon before opening the `Heliox` valve in order to to evacuate the air trapped inbetween the valves. Then, in order to add some exchange gaz, open the valve of the Helium balloon (labeled 1 in the above picture) and close it. Then open the `Heliox` valve (labeled 2) and close it. The ^{3}`He Pot` temperature should decrease faster.
10. Open the Dolly beamline and start the condensation process (Section 3.1).

5 HOW TO SET THE DESIRED TEMPERATURE FROM Deltat

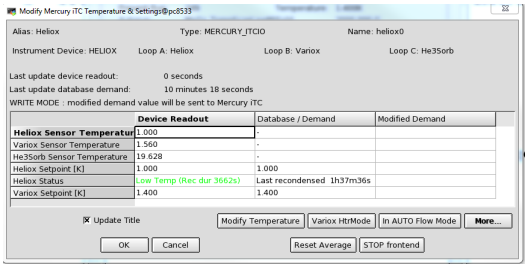
5.1 Interactively

5.1.1 Up to 15 K

Up to 15 K the Variox temperature does not have to change. In order to change the temperature one has to open Deltat and to go the the Modify Devices tab. Then choose Heliox and click on Modify.



Then one has simply to modify the Heliox Temperature at the desired setpoint.



The Mercury will then take care of the pressure parameters by itself as explained in section 3.

5.1.2 Above 15 K

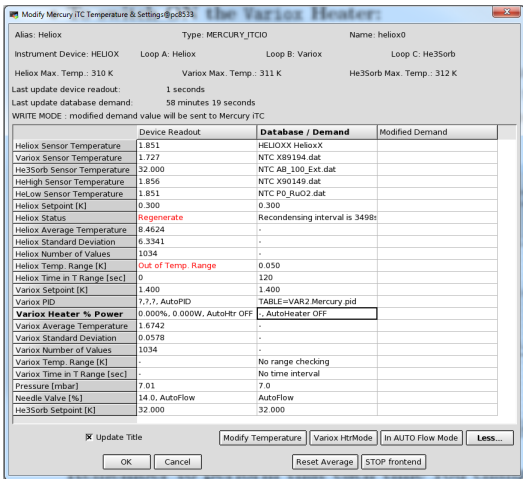
Above 15 K one has to heat the Variox in order to avoid a temperature gradient on the sample. First open Deltat and to go the the Modify Devices tab. Then choose Heliox and click on Modify. Put the same temperature for the Heliox and the Variox and click on the button OK. Then one has to click on the Variox HtrMode button to put the Variox heater in automode. One can check the status of the autoheater cliking on the More ... button.

Remember to perform that each time you change the temperature setpoint.

The temperature for the Heliox can go up to 50 K.

5.2 In an Autorun Sequence

In an Autorun Sequence, the parameters of the Heliox are changed with the command



`SET Heliox`

which can have up to 11 parameters... with the syntax:

`SET Heliox <ATeSP> <ATemRa> <ATimRa> <BTeSP> <BTemRa> <BTimRa> [<BAutoH> <PrSP> <AutoNV> <NVSP> <CTeSP> ]` **! -1 may be set when a value should not change

The first three parameters account for the `Heliox` temperature setpoint, range and waiting time. The next three parameters account for the `Variox` temperature setpoint, range and waiting time. The seventh parameter is the `Variox` autoheater mode. 0 means that the autoheater is off and 1 that it is on. The last four parameters account for the pressure and needle valve setpoint. Usually one does not have to change those parameters as the `Mercury` takes care of them.

5.2.1 Up to 15 K

As the `Variox` temperature does not have to change between base temperature and 15 K you only have to change the first 3 parameters. For example :

```
SET Heliox 3.0 0.2 120
WAIT Heliox inrange 1800
```

will set the `Heliox` temperature to 3.0 K within a range of 0.2 K with a waiting time of 120 s. The second line is here to ensure that the temperature reach 3.0 K within the asked range for at least 120 s. If this temperature is not reached before 1800 s the sequence will proceed anyway (one can note that this line is not compulsory).

5.2.2 Above 15 K

As the `Variox` temperature need to be changed above 15 K you have to change the first 7 parameters. For example :

```
SET Heliox 30.0 0.5 120 30.0 0.5 120 1
WAIT Heliox INRANGE 1800
```

will set the `Heliox` and `Variox` temperature to 30 K within a range of 0.5 K with a waiting time of 120 s. Further the `Variox` autoheater (parameter number 7) is turned on.

Remember to always set 1 for the 7th parameter at each temperature in this mode.

6 EXAMPLE AUTORUN COMMANDS

```
#   Set magnetic field in WEU to 1000 gauss and wait 120 seconds.
SET expmag  WEU 1000.0 120
#   Start data taking. Here 3000000 events are taken on the detector number 2 (Backward)
START Histogram 2 3000000

#   Set magnetic field in WEV to 100 gauss and wait 120 seconds.
SET expmag WEV 100 120
#   Set magnetic field in WEV to 0 gauss and wait 120 seconds.
#     - this procedure is required for the true zero field measurement
SET expmag WEV 0 120
START Histogram 2 3000000

#   Set position of the whole cryostat to 11 mm.
SET Position_Cryo HUB 11 120
#   Set temperature for the Variox and on the sample to 30 K
#       within a range of 0.5 K, and the minimum time in the range of 120 s.
#   One can note that in this case the command for a temperature above 15 K is used.
SET Heliox 30 0.5 120 30 0.5 120 1
#   To actually wait for the temperature to be in a range, WAIT command
#   has to be used.   If the requested temperature is not reached within
#   7200 seconds, the sequence will proceed anyway.
WAIT Heliox INRANGE 7200
START Histogram 2 3000000

#   Set temperature for T lower than 15 K.
#   Set temperature of the sample to 0.5 K within a range of 0.01 K
#       with a minimum time in the range of 120 s.
SET Heliox 0.5 0.01 120
#   To actually wait for the temperature to be in a range, WAIT command
#   has to be used.   If the requested temperature is not reached within
#   7200 seconds, the sequence will proceed anyway.
WAIT Heliox INRANGE 7200
START Histogram 2 3000000

#   Set the slits to 130 and wait 120 seconds
SET Beamline_PiE1 FS51-O 130
SET Beamline_PiE1 FS51-U 130
SET Beamline_PiE1 FS51-L 130
SET Beamline_PiE1 FS51-R 130 120
```

7 HOW TO CHECK THE PARAMETERS AND ANALYZE THE DATA

7.1 How to check the different parameters

There is three different possibility to check the stability of the different parameters (temperature, pressure, field ...).

i) Check directly in `Deltat` going to `Modify Devices` and click the `Show` button.

ii) On an internet browser go to the history of Dolly, http://psw411:8081/HS/All and check the desired parameter.

iii) For the temperature, one can also directly check the Mercury with the camera installed in the experimental area going to this address : http://musr-dolly-4/view/index.shtml

7.2 How to directly analyze the data

Due to trouble for maintaining the last version of the PSI software in every machine, no analyzing program is directly installed in pc8575. To analyze directly your data with this computer you have to be connected to the virtual machine `musruser` under the name `l_musr_tst`. To do so, open the console and write
`ssh l_musr_tst@musruser`
you can ask the password to a member of PSI staff.

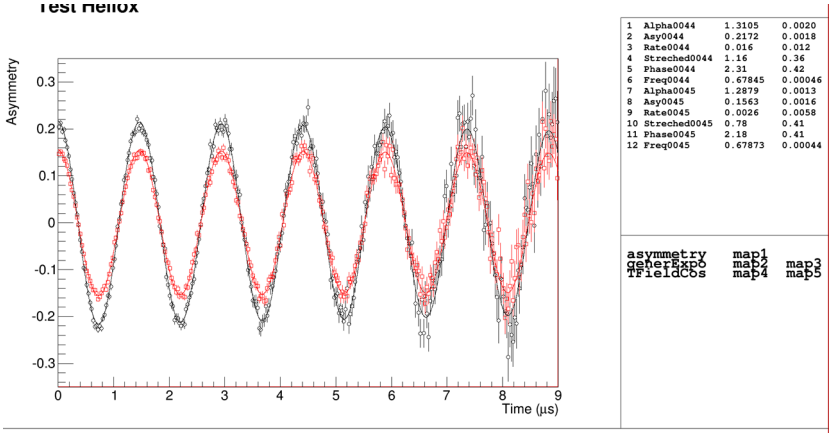
Once you are logged, you can create a folder at your name and work within it using `musredit` and `musrfit`. A manual for this program can be found on the internet : http://lmu.web.psi.ch/musrfit/user/MUSR/MusrFit.html

The `novellook` program can also be used with this virtual computer.

8 Heliox Characteristics

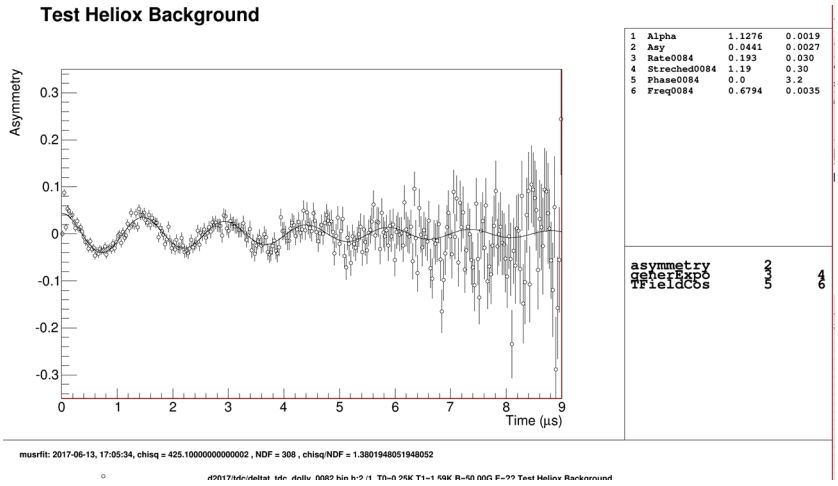
8.1 Initial Asymmetry

In this part we describe the expected initial asymmetry in the Heliox both in longitudinal (black) and transverse (red) polarization under a transverse field of 50 G. We found $A_0^{\text{long}} = 0.217(2)$ and $A_0^{\text{trans}} = 0.156(2)$. One has to keep in mind that as we had to apply a transverse field of 50 G the initial asymmetry in zero field can be slightly different.



8.2 Background

The background was estimated with a 4×4 mm Ni piece. We found for this sample a background of 0.044(3). If one assume a beam size of 8 mm diameter, therefore for a full coverage of the sample one can expect a background lower than 0.014(1).



8.3 WEU Field effect

The Initial asymmetry and alpha value can change when you apply a strong field. We put here an example of this effect in longitudinal and transverse polarization for a silver plate.