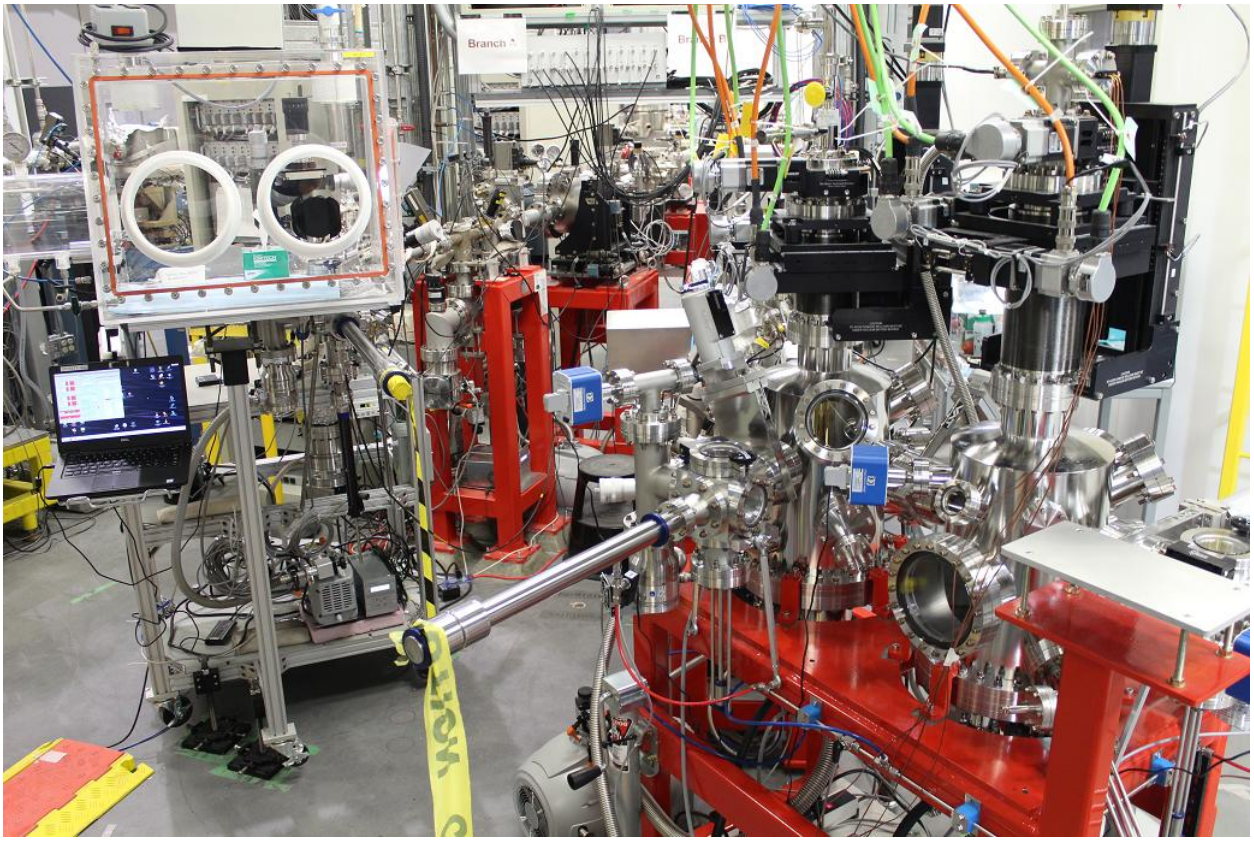
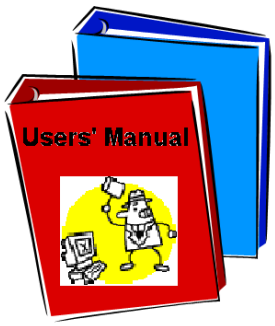
VLSPGM Beamline Manual



Please read carefully

The instructions **to independently run** your experiment, and to troubleshoot the most frequent problems.

During unsociable hours, for assistance, call the Floor Coordinator @ 3639.



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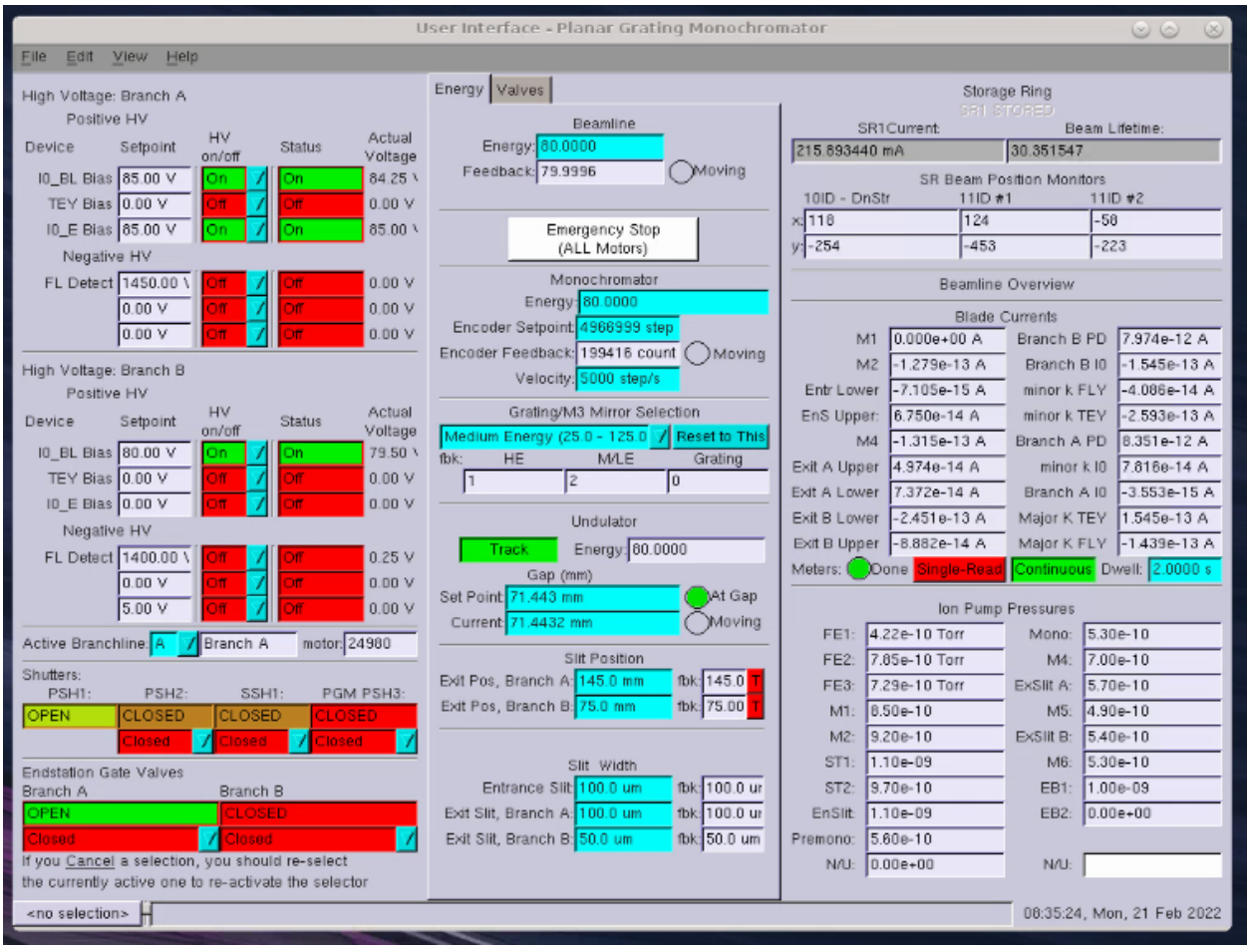
The VLSPGM Control Panel

The Beamline Control Panel GUI opens by double clicking on the “PGM Control Panel” icon:



This panel provides a full overview of the Beamline main components of User interest.

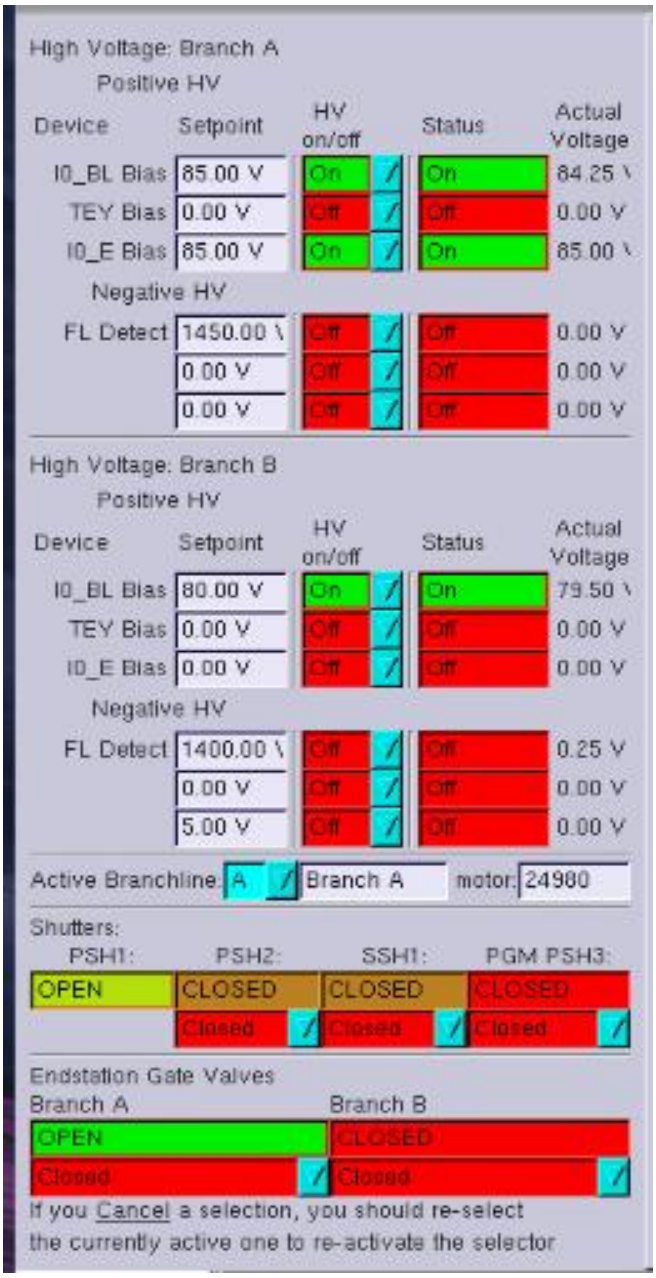
It shows the status of several components of the beamline (e.g. photon shutters, valves, detectors) and serves as the General User Interface to select different energies, gratings and to open/close valves.



A detailed “section by section” explanation is given in the following pages.

The left section

From Top to Bottom:



The High Voltages apply to Branch A:
To turn the HV FL ON (OFF) before (after) a scan when operating the endstation *minor k*

The High Voltages apply to Branch B:
To turn the HV FL ON (OFF) before (after) a scan when operating the endstation *Major K*

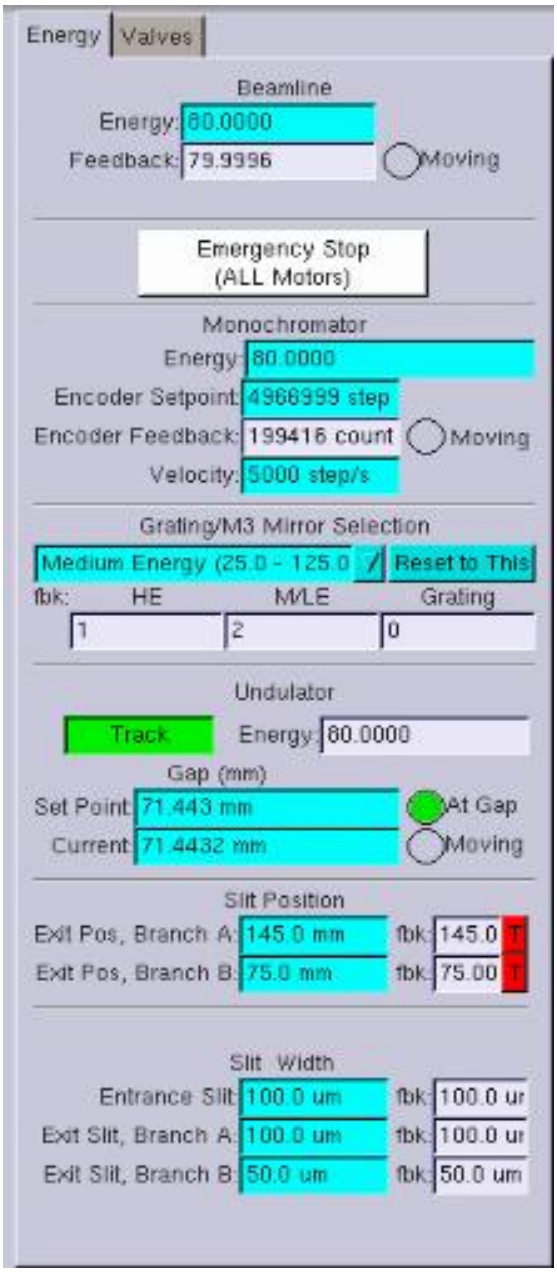
Active Branch-line Selection (A or B).

Photon Shutters (Closed/Open) along the beamline.

End-Station gate valve (Closed/Open), for both Branches A and B.

The central section

From Top to Bottom:



Photon Energy (eV) and relative feedback.
During a scan the “User Data Acquisition Program” automatically control this value.

Grating/M3 Mirror Selection (High; Medium; Low).
Before starting a scan, check that the correct grating is selected for the energy region you are interested in.

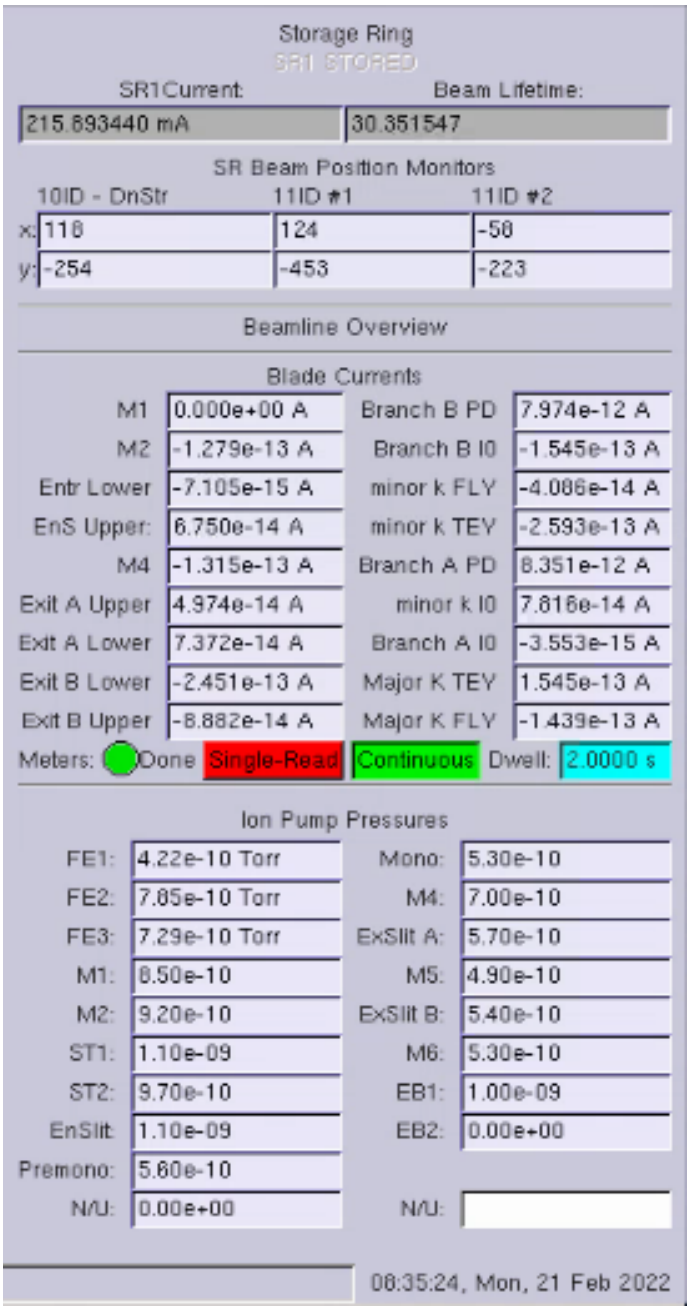
Slit width (5-250 µm) and relative feedback.

Entrance Slit, in common for both branches A and B.

Exit Slit for both branches A and B.

The right section

Storage ring, beamline alignment, and other general BL information, mainly useful for the BL Staff.



Integration time (Dwell time)

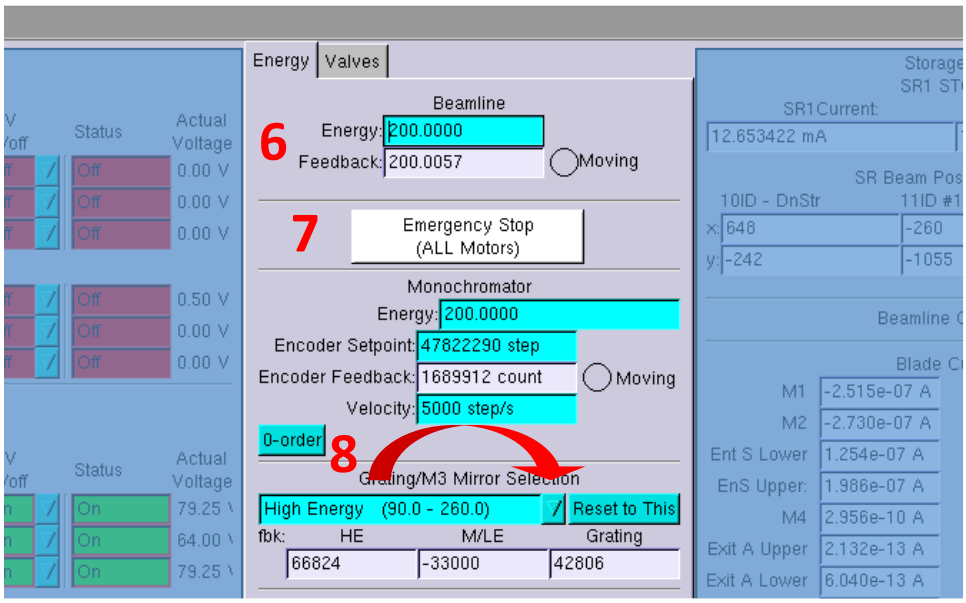
How to change the grating

1. From **8** Grating/M3 Mirror Selection, select the desired grating.
2. The panel will *freeze* until all the three numbers in the boxes (fbk: HE; M/LE; Grating) have stopped.
3. In **6** Beamline Energy, type an energy value close the starting point of your new scan.

NB: during some changes in gratings it can happen that the Energy Feedback reading **6** *indicates:*

- *negative numbers*
- *values not within the VLSPGM energy range*
- *values going in the wrong direction (increasing/decreasing)*

These readings are all consequences of calibration parameter adjustments, or motors repositioning, therefore they should be neglected.



Remember that this process is TIME CONSUMING ~5 minutes

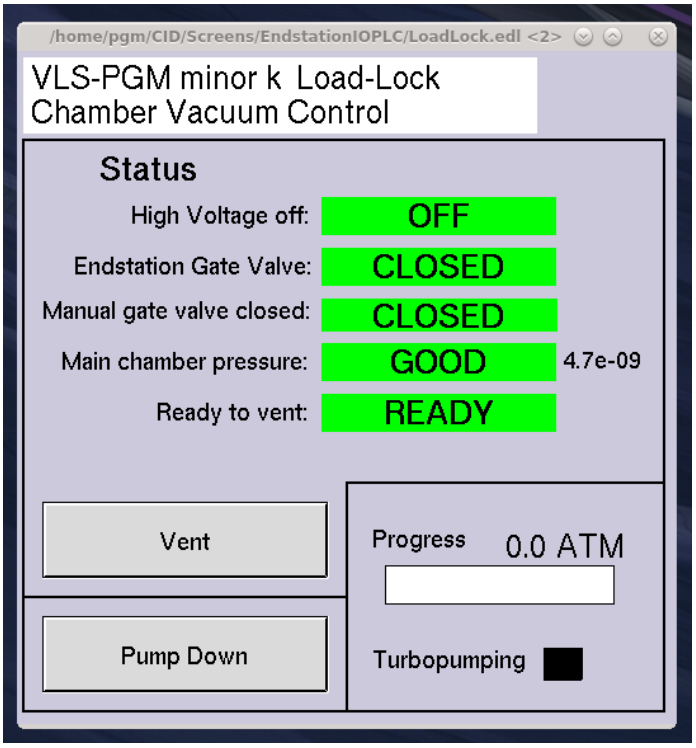
XAS chamber: sample loading & unloading procedure

The instructions on how to place the sample in the chamber are outlined in here.

Every time the Load-lock chamber is vented, 3 sample holders can be loaded on what is called the “ladder”. Only one sample holder can be introduced from the ladder to the main chamber when the load-lock chamber reaches the required vacuum.

Controlled vent of the Load-lock chamber

From the VLS-PGM Load-Lock Chamber Vacuum Control check the following requirements:



- the high voltage is OFF
- the End-Station gate valve is CLOSED
- the Manual gate valve between the main chamber and the load-lock chamber is CLOSED
- the system is READY to vent.

After all requirements are verified, you can start venting the load-lock chamber:

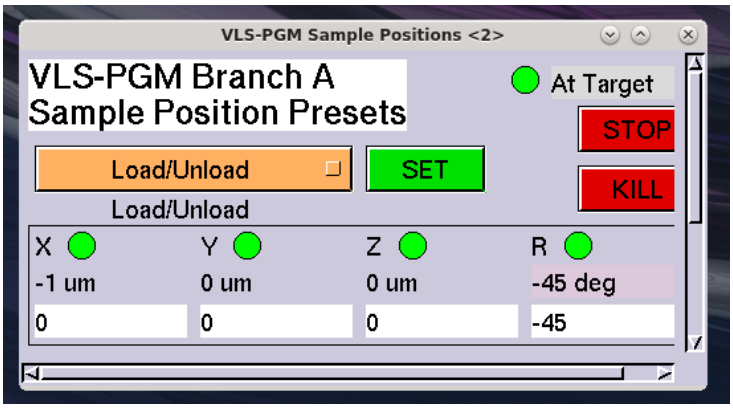
1. Push the VENT button
2. The Turbopumping square box will get **BLACK** in colour, indicating the pump has stopped
3. Wait the Progress bar is 100% **GREEN** in colour, and the pressure reads 1.0 ATM

Open the viewport door in the glove box and raise the ladder.

Sample loading procedure

With the load-lock section up-to-air, load the samples on the ladder. Lower the ladder, close and finger-tighten the viewport door.

Pump down the load-lock chamber to the required vacuum by pushing the PUMP DOWN button in the Load-Lock Vacuum Control GUI. Wait the Progress bar is white in colour, and the pressure reads 0.0 ATM. The Turbopumping square box will also get GREEN in colour, indicating the pump is ON.



Check the Sample Position Preset is set to “Load/Unload”.

If not, set the sample in Load/Unload, allow the motors to finish the movements, and the At Target is **GREEN** in colour.

Once the light on the box on top of the Manual Gate Valve has switched from **RED** to **GREEN**, open the manual gate valve between the load-lock and the main chamber; the pressure in the main chamber should stay better than $2x10^{-6}$ Torr (i.e. $1.8x10^{-6}$ Torr).

For pressure in the main chamber between $5x10^{-7}$ Torr and $2x10^{-6}$ Torr the message will appear on the VLS-PGM Load-Lock Chamber Vacuum Control. **CAUTION**

Proceed keeping an eye to the pressure value. If pressure in the main chamber exceeds $2x10^{-6}$ Torr and the message box turn **RED**, close the manual valve and wait a little longer even if the light on the box on top of the Manual Gate Valve is green.

Using the transfer arm, grab the sample. Push the transfer arm into the main chamber and gently slide the sample into the holder.

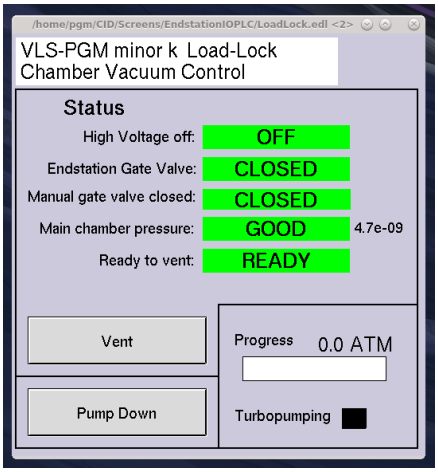
Fully retract the transfer arm back into the load-lock and close the manual gate valve.

Set the sample in the desired position (MCP; SDD) from the GUI and allow the motors to finish the movements.

If the pressure in the main chamber is reasonable (better than $5x10^{-7}$ Torr; i.e. $4.8x10^{-7}$ Torr) you can start to run your scan.

Sample un-loading procedure

From the VLS-PGM Load-Lock Chamber Vacuum Control check the following requirements:



- the high voltage is OFF
- the End-Station gate valve is CLOSED

Set the sample in Load/Unload position from the GUI and allow the motors to finish the movements.

Check that the light on the box on top of the Manual Gate Valve is **GREEN**

Open the manual gate valve connecting the load-lock to the main chamber.

Push the transfer arm into the main chamber. Lock onto your sample.

Smoothly fully retract the transfer arm back into the load-lock, CAREFUL not to open the jaws. Place the sample onto the ladder.

Close the manual gate valve.

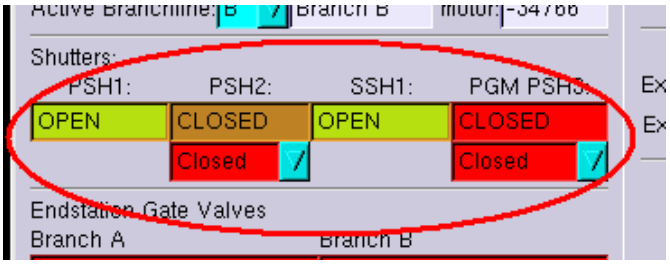
Do you have more samples on the ladder that need to be analyzed? Proceed with loading the next sample in the main chamber.

Have you analyzed all the samples on the ladder? Proceed with the controlled venting of the load-lock to remove or replace the samples.

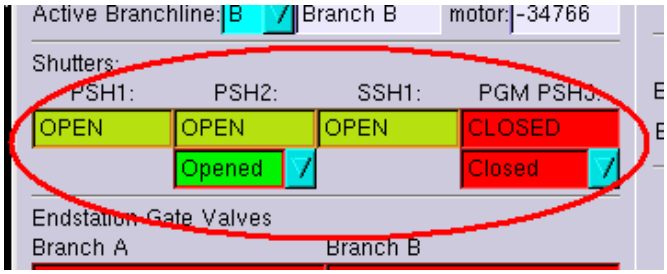
To get the light on the sample

After an injection with Shutters closed, as soon as the control room has enabled the beamlines, and BEFORE the start of a new data acquisition, a few Photon Shutters have to be opened in the correct order:

1. OPEN the Safety Photon Shutter (SSH1)



2. OPEN Shutter two (PSH2)

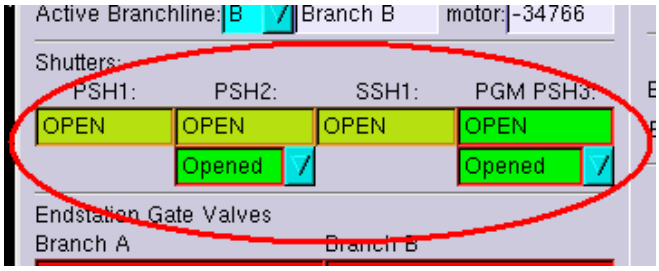


Never CLOSE (PSH2) and (SSH1)

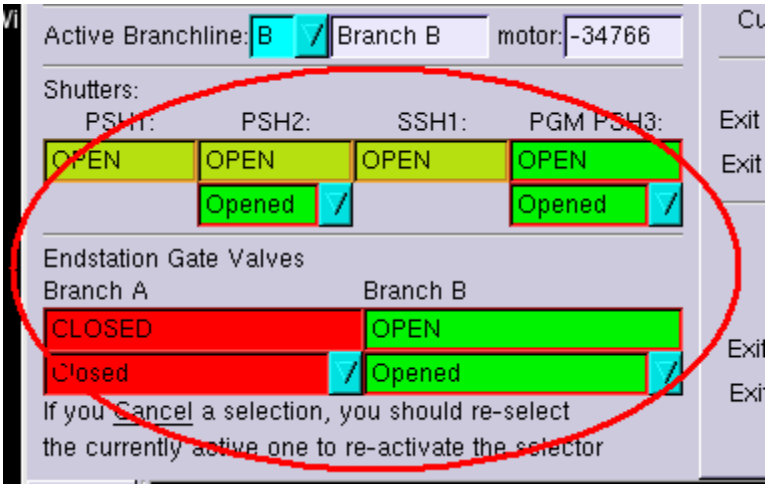
They are in COMMON with another beamline, therefore closing any of them jeopardizes the other Users' experiment.

---

3. OPEN Shutter three (PGM-PSH3)



4. OPEN the End-Station gate valve of the Branch-line (A or B) you are using.



The light is now at the sample position.

A couple of points need to be considered:

- The ring is operated in Top-Up mode and injections are performed with shutters open; therefore, most of the time, Users will not need to do steps 1. and 2.

- Often, after Injection-with-shutters-closed, SSH1 and PSH2 are opened by the neighboring SGM Beamline, with which VLSPGM shares those components.

Data Acquisition at VLSPGM

For the standard XAS measurements, where Total Electron Yield (TEY) and Total Fluorescence Yield (FLY) are simultaneously recorded, VLSPGM provides two data acquisition configurations: Step Scan and Fast Scan. Users can choose the configuration best suited to their study.

If the User's experiments require data collected with the XEOL QEPro spectrometer only Step Scan is available for Total Luminescence yield.

The differences between Step and Fast scans are briefly outlined:

Step scan characteristics

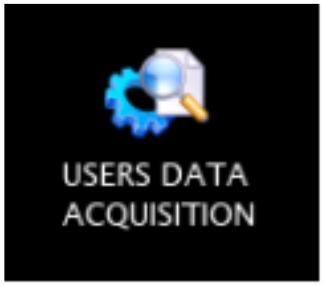
- Users decide the energy range (e.g. 158eV to 130eV), the dwell time (e.g. 1 second) and the step size (e.g. 0.1eV) for each scan.
- The measurement at each energy point (e.g. 158; 157.9; 157.8 etc.) occurs after the motors have stopped.
- The total duration of a typical 1sec-dwell-time scan is largely caused by the dead time required for starting and stopping the motors at each energy point.
- The points are equally energy spaced by the value input as step size (Delta Value).
- Typically, the duration of a 25eV scan is ~20 minutes

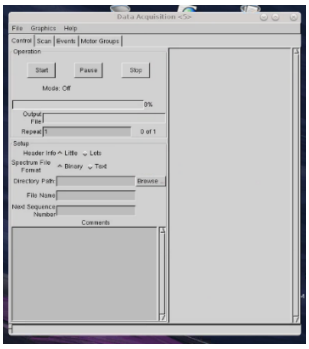
Fast (on-the-Fly) scan characteristics

- Energy range, dwell time and step size for each scan are pre-set and not changeable.
- At the start of the scan the motors go to the final point and the instrumentation recording the signals (e.g. I0, TEY and FLY) are sampled along the motion at consistent measurement times (1 second).
- The provided "mean Energy fbk" value should be used when analyzing the data and as x-axis when plotting the resulting spectrum.
- The points within a scan are not equally energy spaced.
- Typically the duration of a 25eV scan is ~5 minutes

XAS USERS DATA ACQUISITION in Step Scan configuration

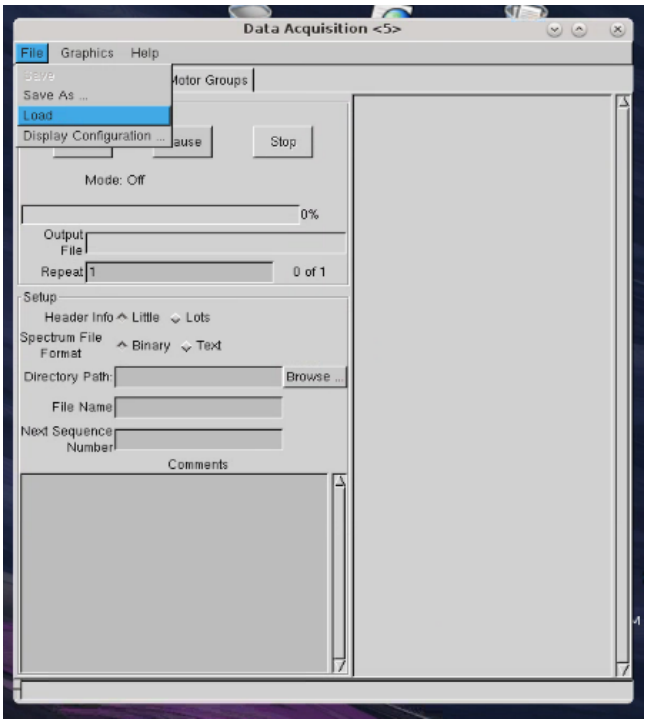
Open the “USERS DATA ACQUISITION” GUI by double clicking on the icon:

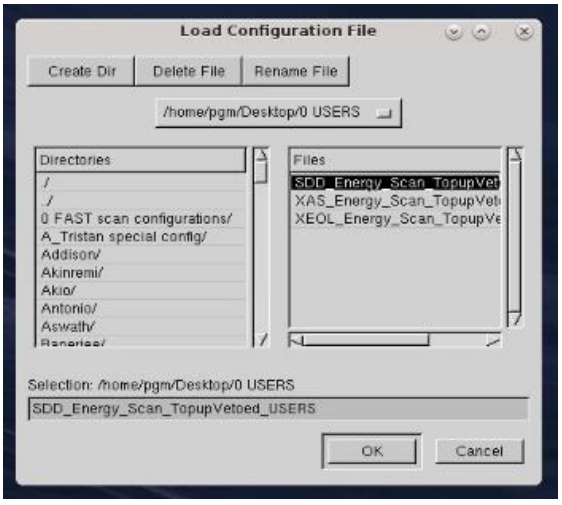




Step scan

From the “File” menu Load the configuration file “XAS_Energy_scan_TopupVetoed_USER”
Work your way down the directory tree to **/home/pgm/Desktop/0 USERS**.

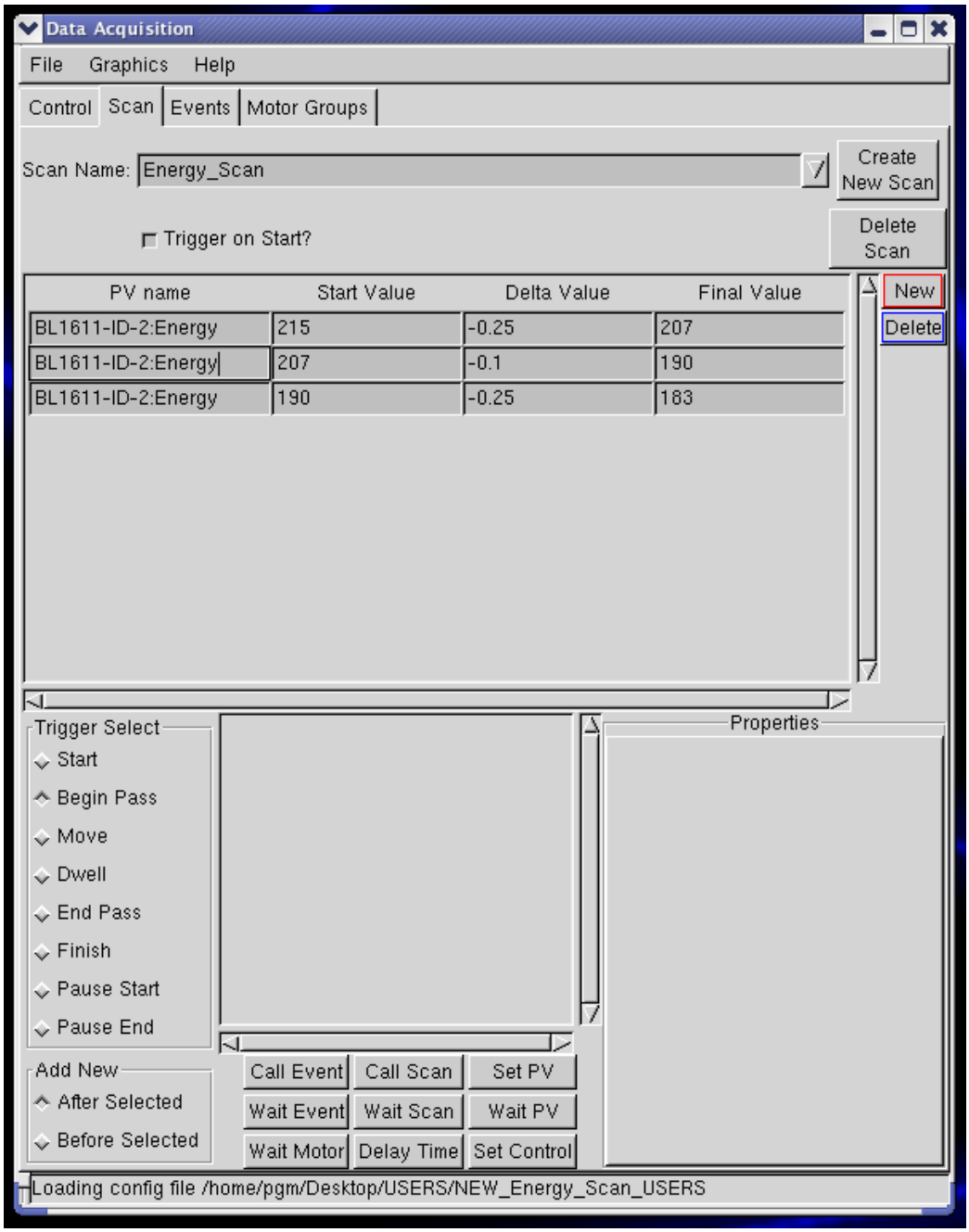




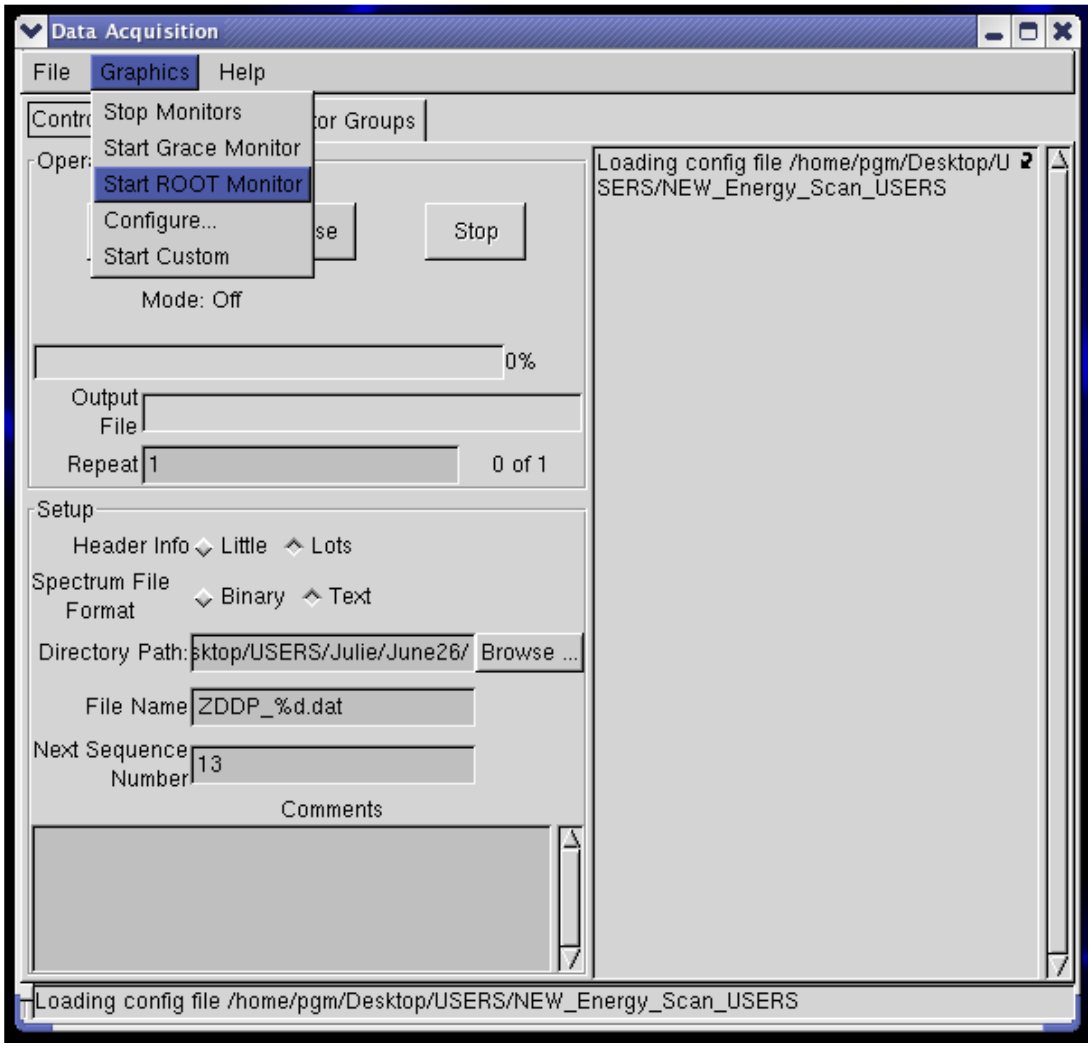
1. Setting the Scan Energy Range

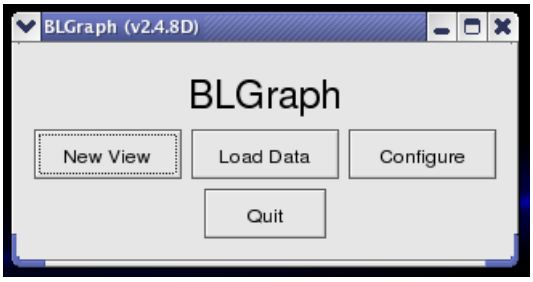
   In the *Scan* TAB make sure you are scanning over the correct energetic range, from high (Start Value) to low (Final Value) energy, with a negative step (Delta Value).

   You can also scan over several consecutive regions with different steps (delta values).
   To add a new region click the New button.
   To delete a region click the Delete button.



2. To visualize the data while acquiring, from the *Graphics* menu select “Start ROOT Monitor”

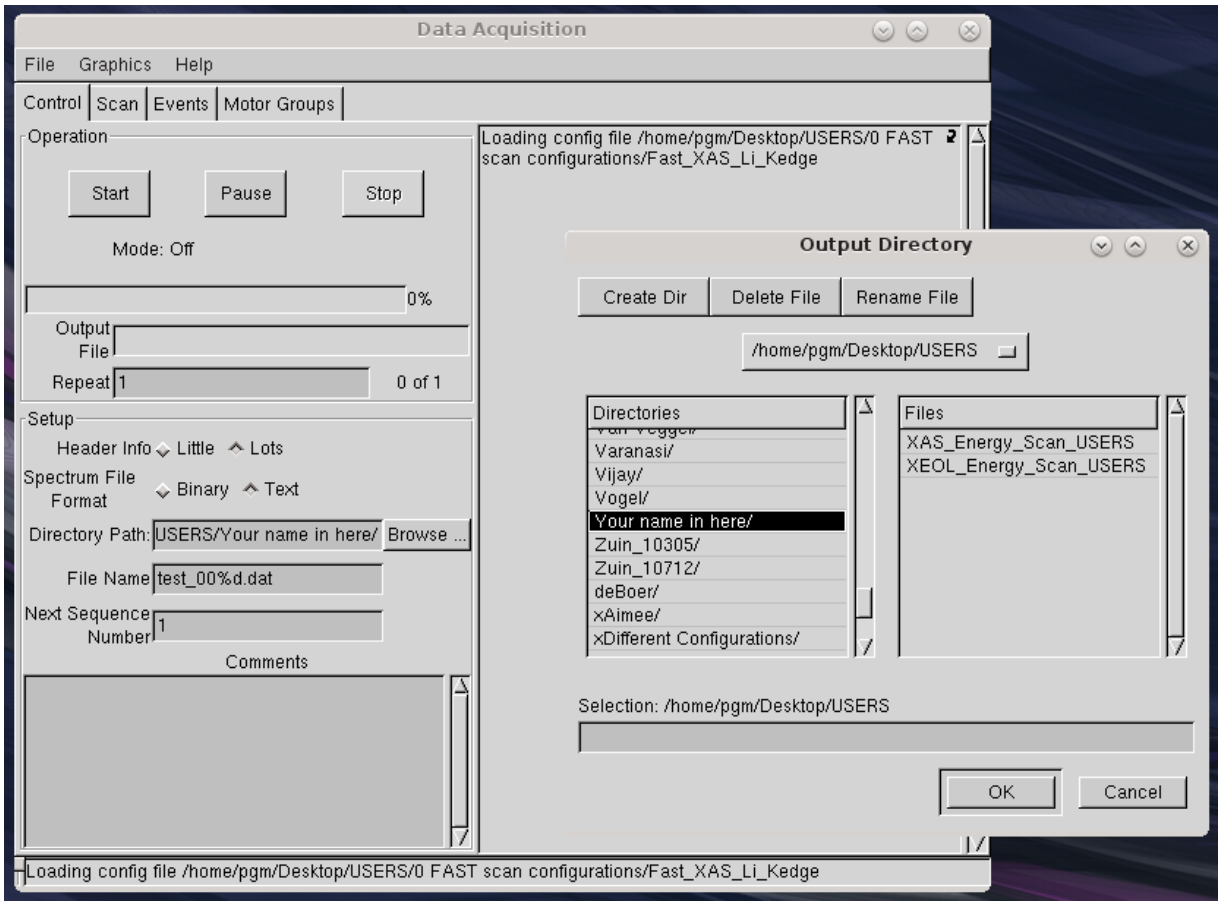




The BLGraph GUI opens.

Keep it always open, DO NOT “Quit”. Automatically a new plot will start at the beginning of each scan.

3. Check the settings in the *Control* tab
   User data are generally saved in '/home/pgm/Desktop/USERS' under your own directory.
   Directory Path shows where your file is saved.



4. In *File Name* the symbol "%00d" will give you sequential file numbers for sequential scans, do not delete it.

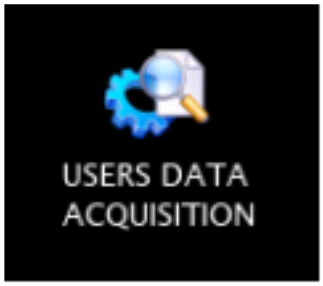
5. Click "Start" in the *Control* tab when you are ready to scan.

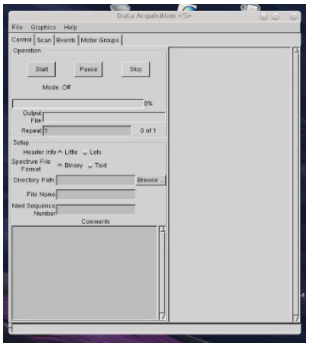
XAS USERS DATA ACQUISITION in Fast (Fly) Scan configuration

The following are the available pre-set Fast Scan configurations

Configuration file name	Pre-set Energy range	Grating
Fast_XAS_Mn_Medge	75-40eV	Medium
Fast_XAS_Li_Kedge	75-47.5eV	Medium
Fast_XAS_Al_Ledge	90-70eV	Medium
Fast_XAS_Al_Si_Ledges	120-70eV	Medium
Fast_XAS_Si_Ledge only HEG	121-95eV	High
Fast_XAS_P_125_Ledge	156-125eV	High
Fast_XAS_P_Ledge	156-130eV	High
Fast_XAS_S_Ledge	193-158eV	High
Fast_XAS_B_Kedge	210-185eV	High

Open the “USERS DATA ACQUISITION” GUI by double clicking on the icon:

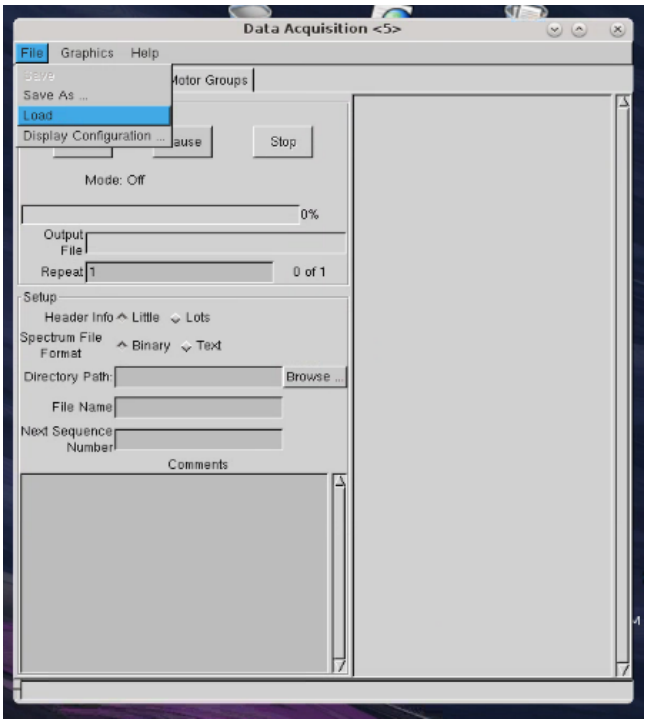




Fast Scan

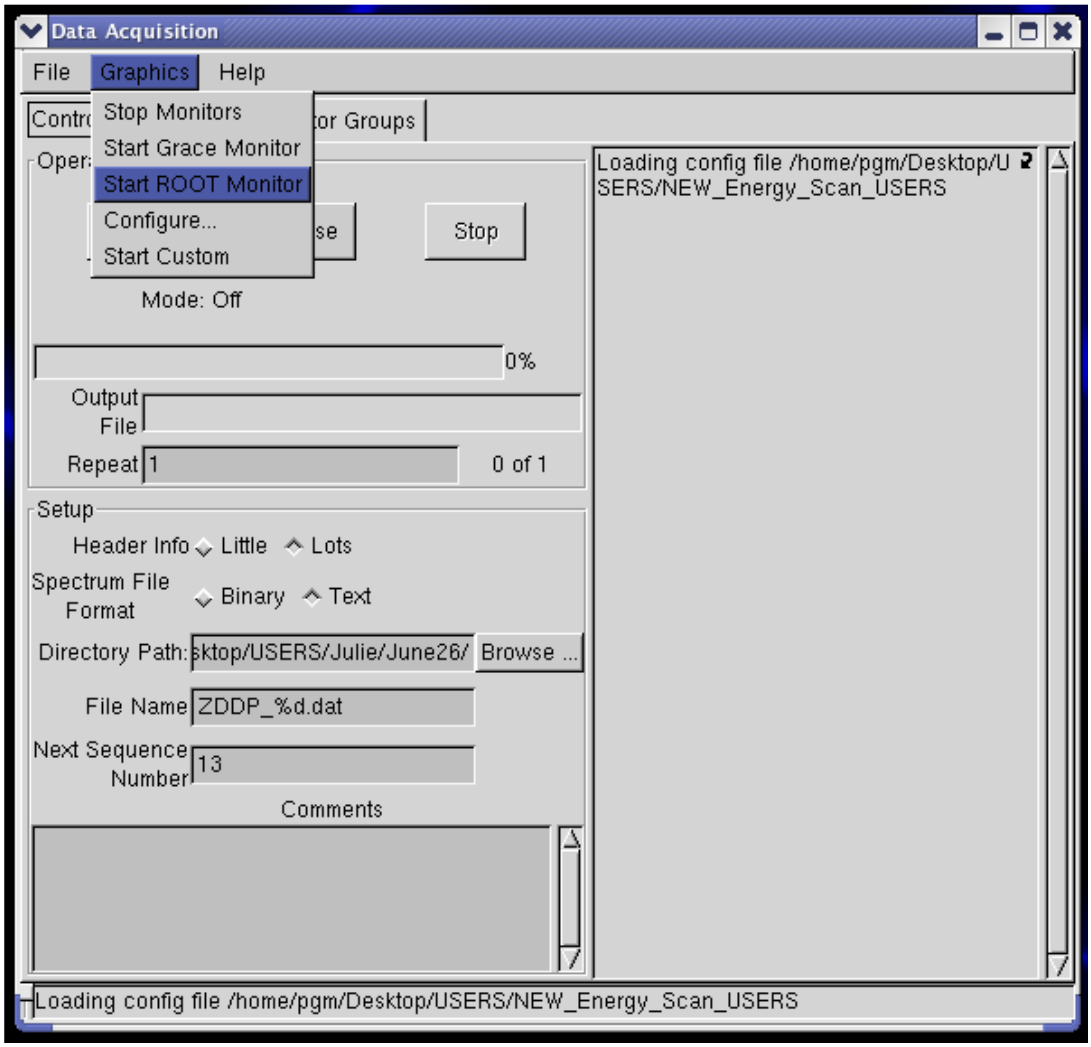
From the “File” menu Load the configuration file “Fast_XAS_**###**_scan_TopupVetoed_USER”

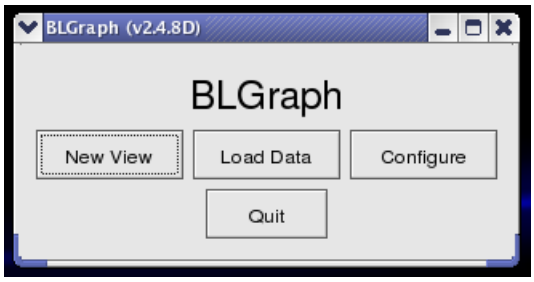
Work your way down the directory tree to **/home/pgm/Desktop/0 USERS/0 Fast scan configuration**.



element and edge of interest

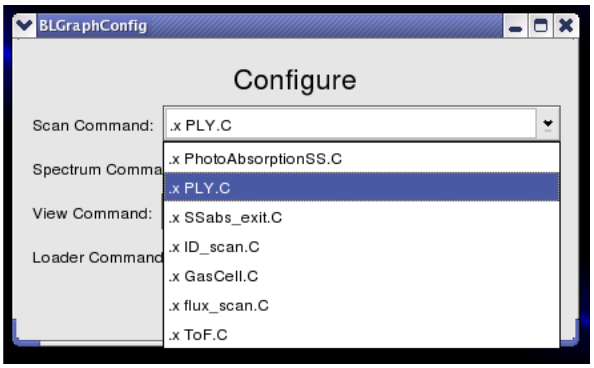
1. To visualize the data while acquiring, from the *Graphics* menu select “Start ROOT Monitor”





The BLGraph GUI opens.
Click Configure.

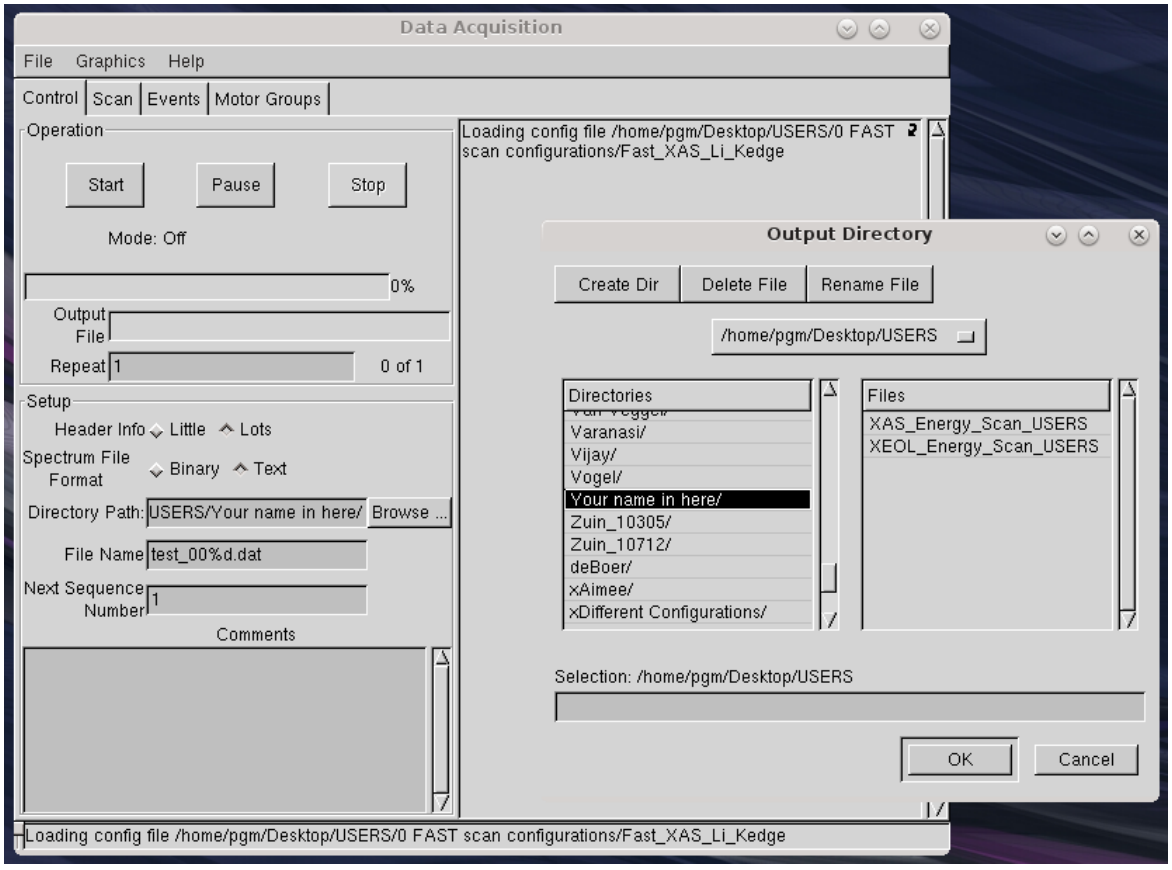
Never “Quit” this window



In Configure, select PhotoAbsSS_MeanEnergy.C, and then Close it.

Keep the BLGraph GUI always open, DO NOT “Quit”.
Automatically a new plot will start at the starting of each scan.

2. Check the settings in the *Control* tab
   User data are generally saved in ‘/home/pgm/Desktop/USERS’ under your own directory.
   Directory Path shows where your file is saved.



3. In *File Name* the symbol “%00d” will give you sequential file numbers for sequential scans, do not delete it.
4. Click “Start” in the *Control* tab when you are ready to scan.

XEOL, SDD and XAS Synchronization Software

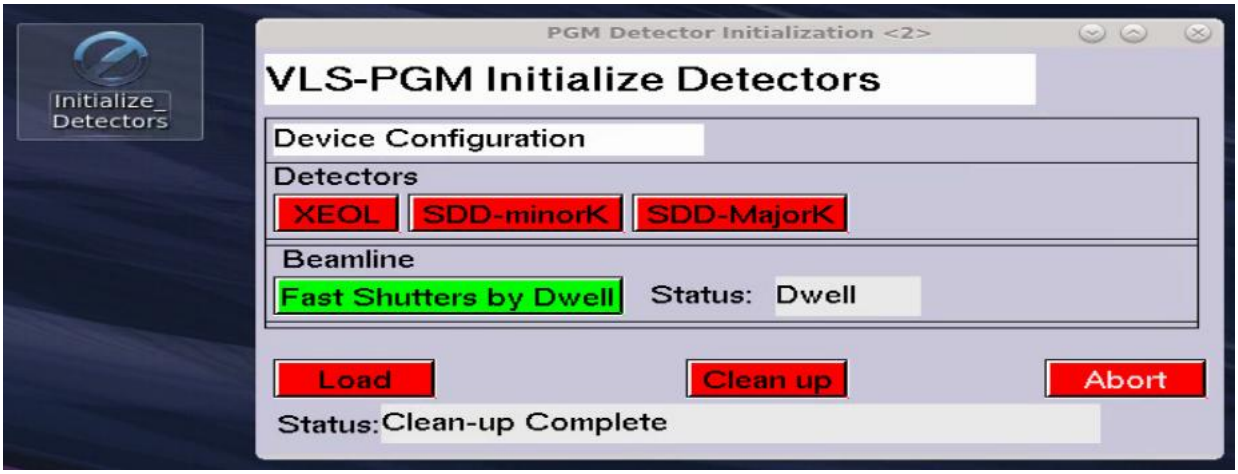
Collecting the Total Luminescence Yield (TLY) or the SDD FL from the min k or Maj K endstations together with I0, TEY and FLY during an XAS scan involves multiple devices to operate/read-out different signals in a synchronized mode.

The synchronization software needs to be informed that, together with the signals collected by Picoammeters, also the XEOL spectrometer or SDD has been introduced, and which Fast Shutter has to be operated.

Beamline Staff is responsible for checking this when preparing for the experiment.

The following instructions are as a reminder for the BL staff and shall not be performed by unexperienced Users.

Open the Initialize Detectors



After the IOC app of the required detector (e.g. XEOL) is launched:

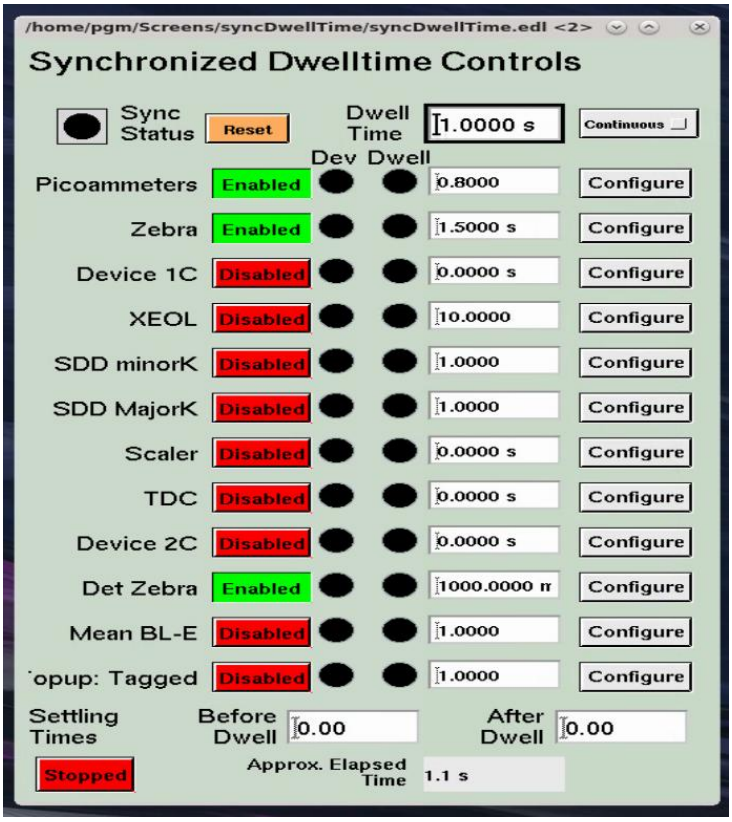
- Ensure the necessary detector (e.g. XEOL) is green, and unnecessary detectors are red
- Press "Fast Shutters by Dwell", status should say "Dwell"
- Press "Load"
- Wait until "Status" says "Load Complete"; this usually takes about 20-30 seconds
- Press "Clean up"
- Wait until "Status" says "Clean- up Complete"

This procedure could be required anytime a different Branch is selected and/or a different detector is required, especially if the latest used detector was not properly shut down.

This procedure is required after any power outage.

Open the Beamline Dwell Time:





All the BL available devices are listed in the Synchronized DwellTime Controls.

Simple XAS measurements have only the Picoammeters and two Zebras Enabled, responsible for collecting the TEY and FLY signals synchronized with the Fast Shutters.

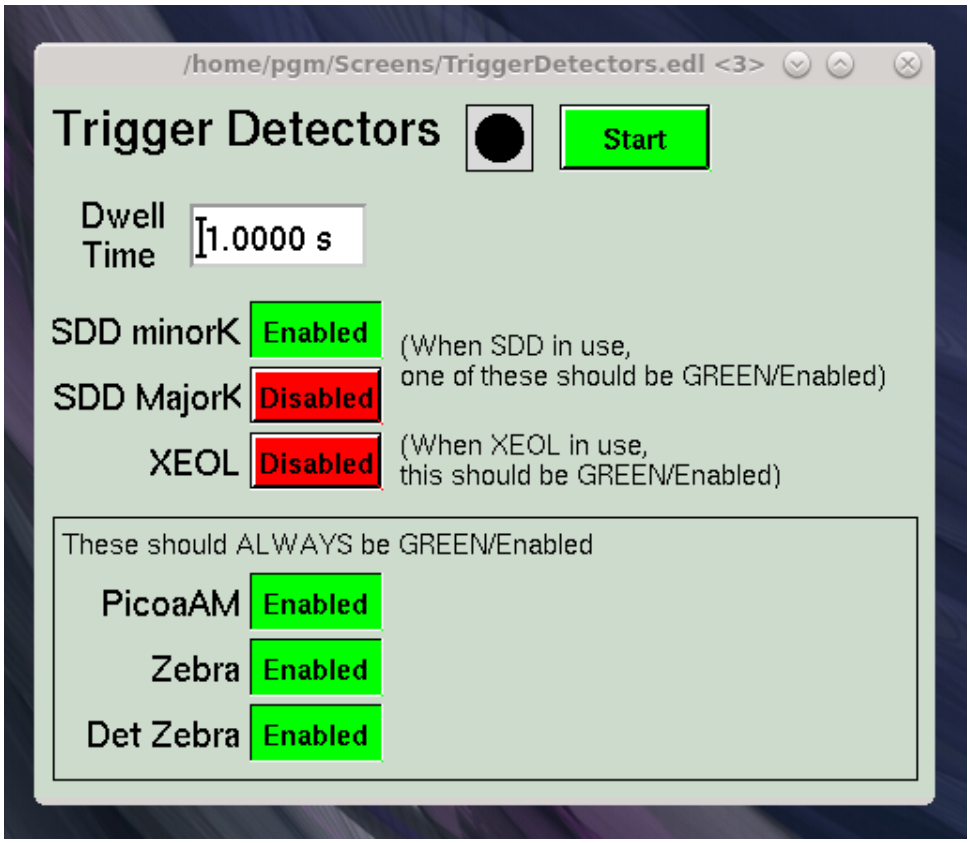
Check that the XEOL, or one of the two SDDs, is Enabled.

Test the synchronization

- enter a Dwell Time different from the one pre-set (e.g. 2 or 5 seconds) and verify that all devices time-entry match
- set the Continuous/Single selector to "Single"
- press the Start button to make sure all Enabled devices trigger (indicator should turn green at the same time as Picoammeters' indicator)
- set the Continuous/Single selector back to "Continuous"

Open the Trigger Detectors:





SDD instructions

Switch ON the system

Beamline Staff is responsible for switching on the detector read-out “The BOX” and the out-of-vacuum chiller for the SDD systems when preparing for the experiment. Press/release the button. It should light up. Do NOT HOLD the button down. You can probably hear ONE very soft beep.

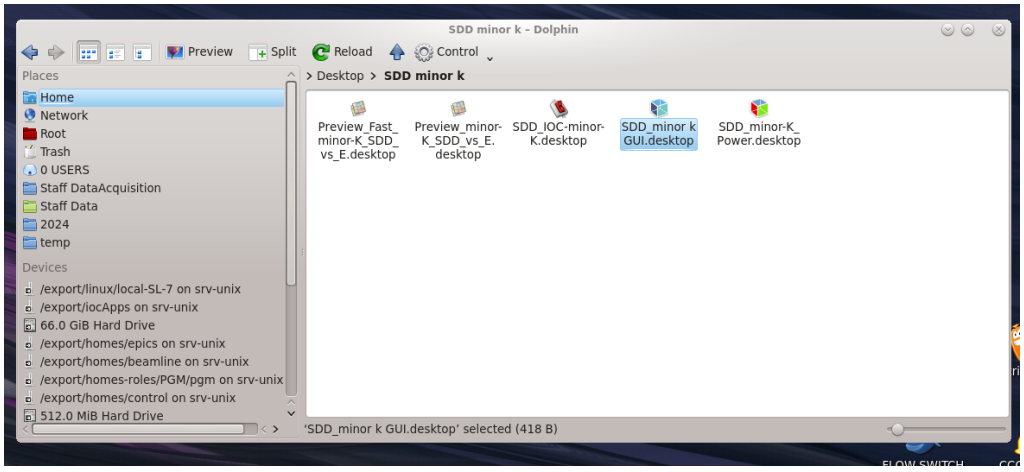
If you hear the second beep, you held the button too long. Therefore you MUST:

1. wait a full minute - use a clock to be sure you wait the correct amount of time,
2. switch off the box,
3. wait another full minute
4. switch ON “The BOX” paying more attention to the timing/beep requirements

Start the detector control system

Beamline Staff / Users: from the SDD *Endstation name** folder:



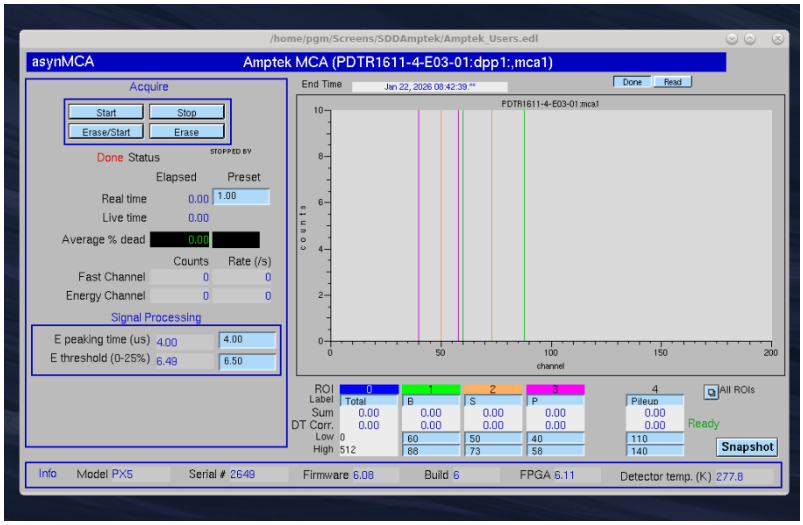


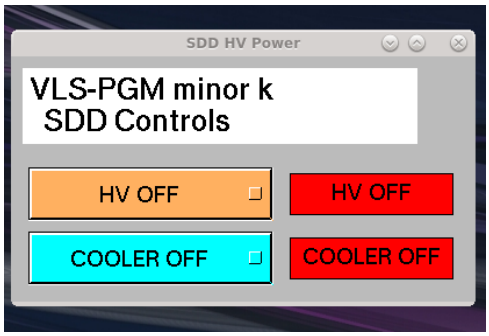
- double-click on the SDD IOC

- type 1, a second window will open and after few minutes will/should close.

- double-click on the SDD GUI

** minor k on Branch A*
Major K on Branch B





Two new windows/interfaces will open: the MCA GUI, and the "SDD temperature and HV control" to be used during the experiment when the sample is moved within the chamber and the chamber light needs to be lit; or samples are swap and the manual gate valve between the Main chamber and the Load-lock chamber needs to be open.

With the sample in position for starting the data acquisition, and the chamber light **OFF**, from the SDD Controls:

- Switch on the Cooler.
- On the MCA screen check the temperature reading, wait until it is ~225K and the box-line goes from RED to Yellow to clear.
- Switch on the HV

Other settings on the MCA screen include:

Real time preset ≠0 (e.g. 1sec)

E peaking time (µs)	4.00	1≤ t ≤8
E threshold (%)	6.5	0≤ % ≤25

Snapshot: allows saving the current MCA as a data file. Two columns: channels vs total counts

Erase/Start: clears the previous spectrum and take a new one for an integration time equal to the pre-set time.

Collect an MCA

In the following order:

1. Close the slits to 25µm x 25µm (Entrance Slit x Exit Slit)
2. Set the beamline energy to a value above the edge of interest, where the photon counts from sample should be highest
3. Open the Endstation Gate Valve
4. Open the PGM PSH3
5. Using the START button on the Trigger Detectors GUI (see picture on page 24 if you are not sure), take a MCA and verify that:
    a. the *Average % dead* is below 40% for any peaking time value, not just for the default 4µs value
    b. the *Rate* in the *Energy channel* do not exceed 30,000/sec (assuming you did not change the default peaking time of 4µs)

If you need to modify the slit's settings, for each new slits setting do the following:

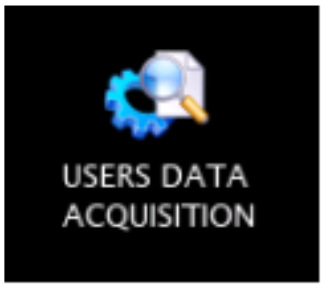
i. With the PSH3 closed, open/close the slits by 25µm
ii. Open the PSH3 and take a new MCA
iii. Verify the *Average % dead* and the *Energy Channel Rate* conditions (5.a. and 5.b.)

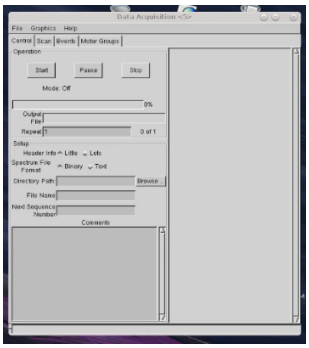
NB: Every time the chamber light needs to be switched ON, from the SDD Controls the HV MUST be switched OFF -- **Now the system is in safe mode from any light flood in the experimental chamber.**

NB: Every time the manual gate valve between the Main chamber and the Load-lock chamber needs to be opened, from the SDD Controls the HV MUST be switched OFF, and the COOLER MUST be switched OFF -- **Now the system is in safe mode from any light flood and pressure bumps in the experimental chamber.**

Setting up for XAS

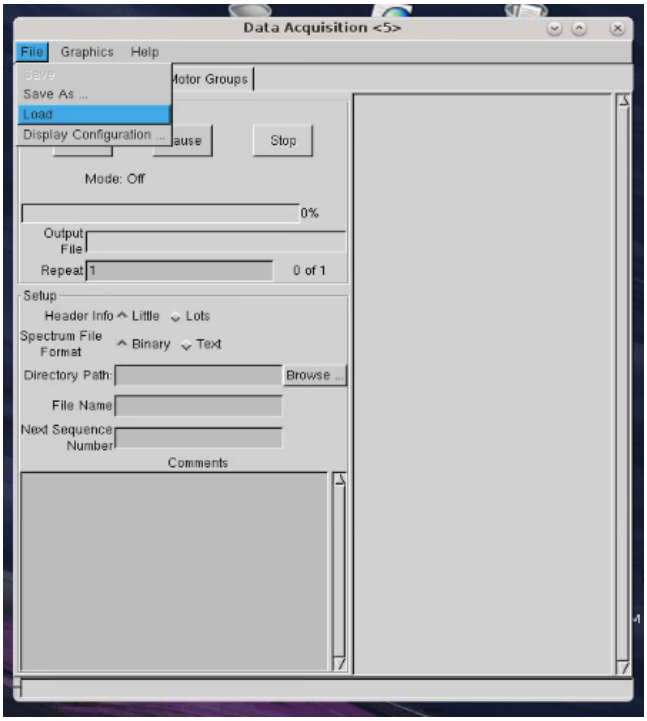
Beamline Staff / Users: Open the "USERS DATA ACQUISITION" GUI by double clicking on the icon:

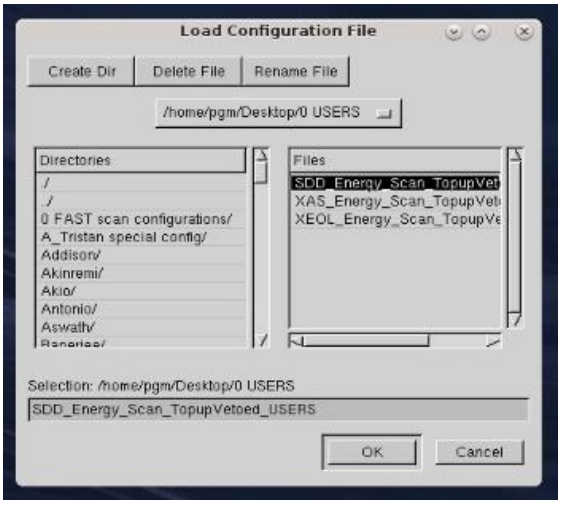




From the "File" menu Load the configuration file "SDD_Energy_scan_TopupVetoed_USER"

Work your way down the directory tree to **/home/pgm/Desktop/0 USERS**.

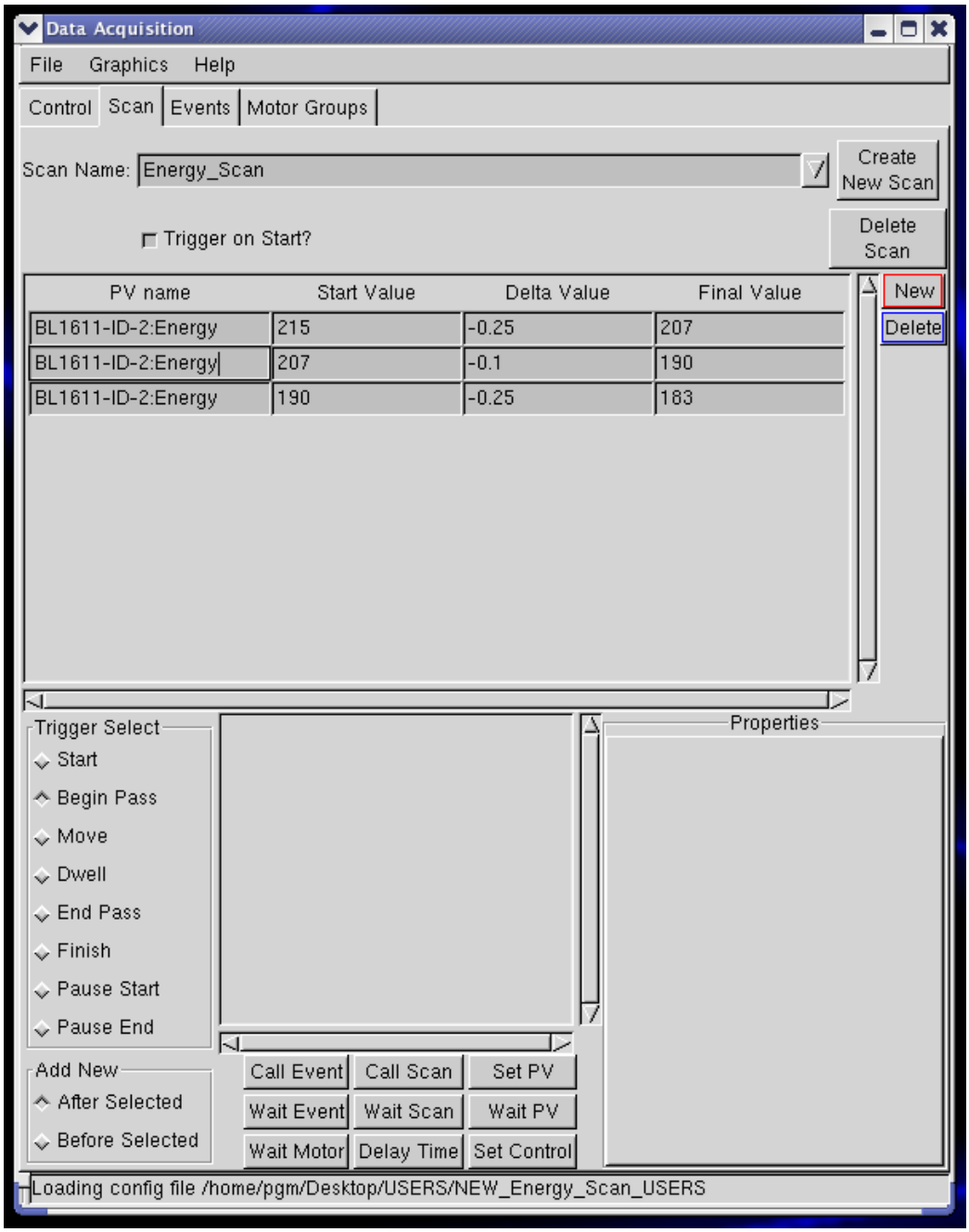




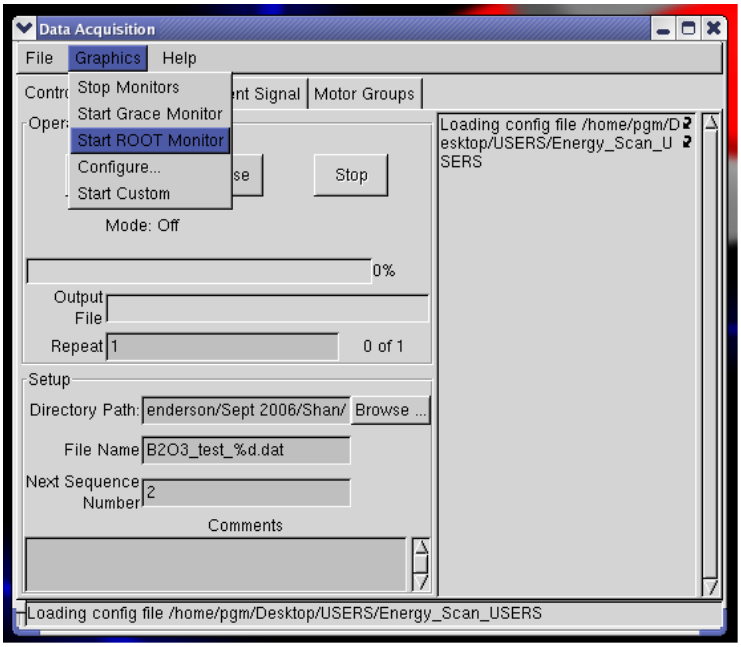
In the Scan tab make sure you are scanning over the right energetic range, from higher (Start Value) to lower (Final Value) energy, with a negative step (Delta Value).

The Dwell Time of the scan is the one set on the Trigger Detectors GUI (page 24)

You can also scan over several consecutive regions with different steps (delta values).
To add a new region click the New button.
To delete a region click the Delete button.

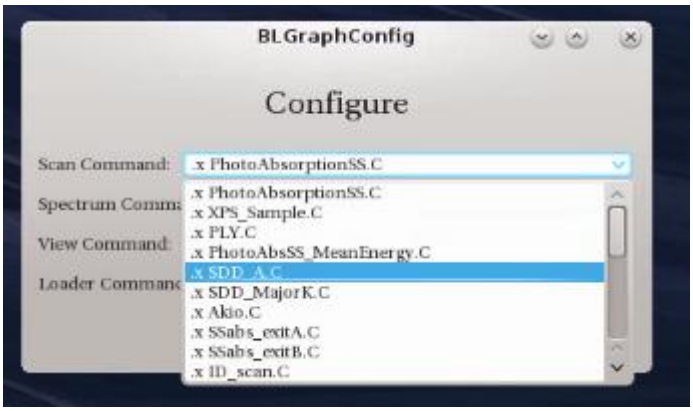


To visualize the data while acquiring, select “Start ROOT Monitor” from the “Graphics” menu.





Click on Configure, select SDD_A.C (or SDD_MajorK.C depending on which endstation you are using), and then Close.



Keep the BLGraph window always open, DO NOT press the “Quit” button. Automatically a new plot will start at the beginning of each scan.

Next, check the settings in the Control tab:
User data are generally saved in '/home/pgm/Desktop/0 USERS' under your own directory.
Click "Start" in the Control page of the Data Acquisition when you are ready to scan.

While collecting a XAS spectrum it is **good practice** to check the *Average % dead* just before the edge features and note whether it rises above 5%.

If it rises above 5%, before starting the next repetition on the same sample, you should readjust the slits to a smaller width (e.g. if the *Average % dead* rises above 5% at 150µm x 150µm, close the slits to 125µm x 125µm before the next scan).

Switch off procedure

Users: Every time a sample is changed, and at the end of the measuring session, from the SDD Controls the HV MUST be switched OFF, and the COOLER MUST be switched OFF

Now the system is in a safe mode from any light flood or pressure bumps in the experimental chamber.

XEOL instructions

The XEOL system allows Users to record:

- Photoluminescence data at a Single Energy value or
- Total Luminescence Yield (TLY) collected together with Total Electron Yield (TEY) and Total Fluorescence Yield (FLY) during a XAS scan.

In the latter case, the data acquisition is very similar to the one for general XAS spectra in step scan configuration mode.

The XEOL spectrometer (QEPro) is physically connected to and controlled by the IOC situated at the endstations.

The IOC start-up and GUI to operate the system are located in the folder “XEOL Optical Spectrometer”; this folder is available from any beamline computer.



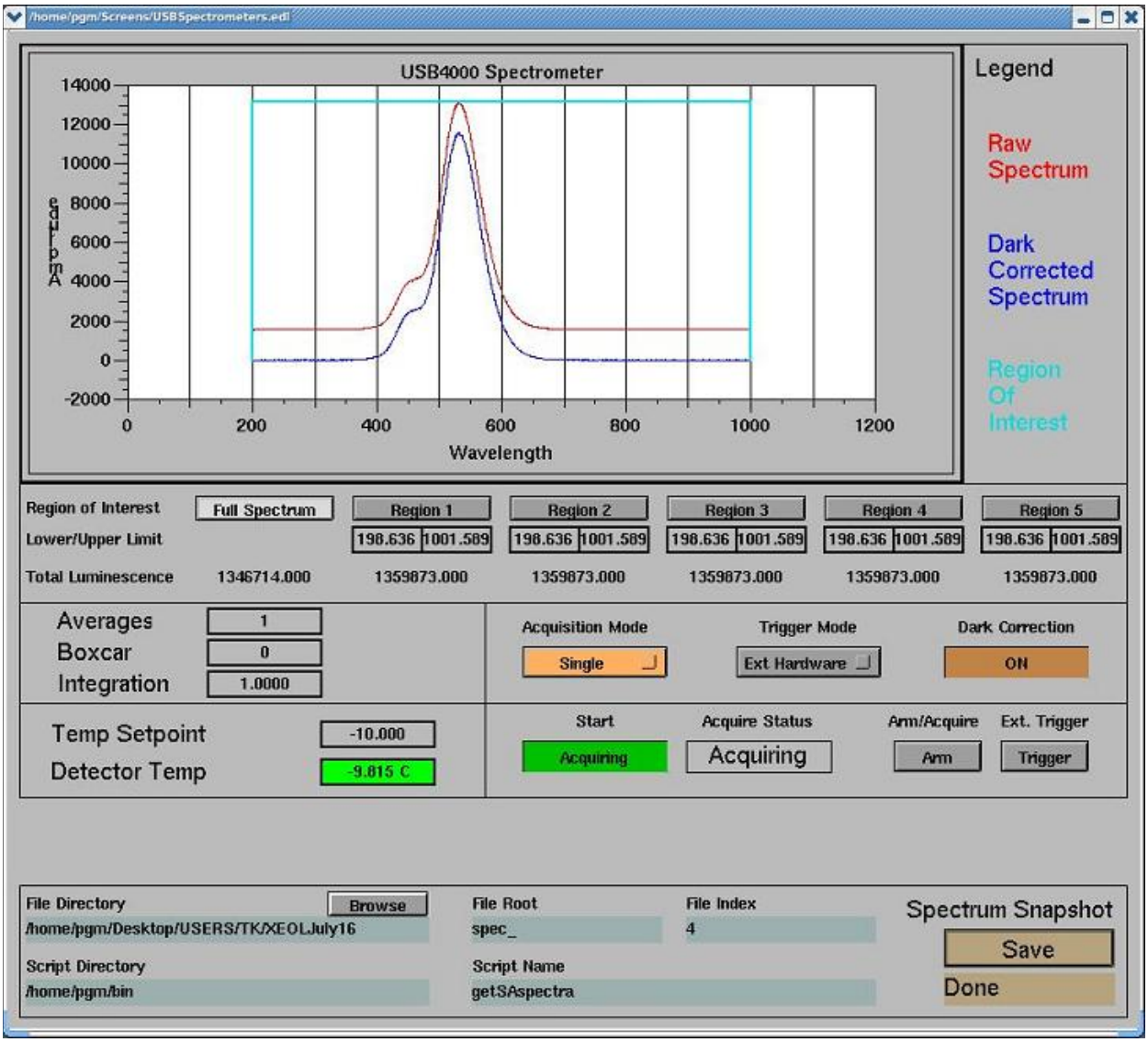


Start the XEOL detector control system

- Open the USBQEPro IOC
    - In the terminal window, type 1 if you are using the *minor k* endstation located on Branch A, or type 4 if you are using the Major K endstation located on Branch B.

      A second window will open and after few minutes will close indicating that the start-up sequence has completed.

- Open the USBQEPro GUI.



The Trigger Mode should be set to Ext Hardware.

The Integration (time) should never be set below 10 ms.

Typical values for **Averages** and **Boxcar** are 1 and 0, respectively.

Photoluminescence at single energy point

With the Endstation Gate Valve and PGM PSH3 closed:

- Set the required Integration time
- Check that the Dark correction is OFF
- Change the Acquisition mode from Single to Dark current
- Record the Dark Current

A new Dark Current should be recorded any time the scan conditions are changed (i.e. Integration time)

Change the Acquisition mode back to Single and the Dark correction to ON

Set the beamline energy

Open the Endstation Gate Valve

Open the PGM PSH3

Using the START button on the Trigger Detectors GUI (see picture on page 24) acquire a scan

Close the PGM PSH3

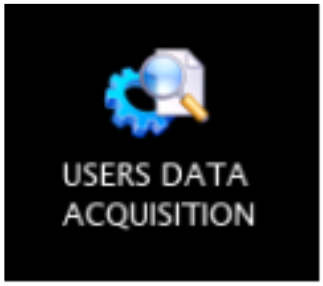
Check the File Directory: User data are generally saved in '/home/pgm/Desktop/USERS' under your own directory.

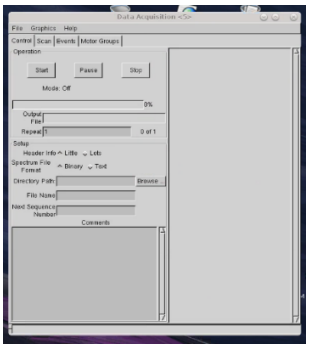
Set the File Root (name) and File Index

Save the scan

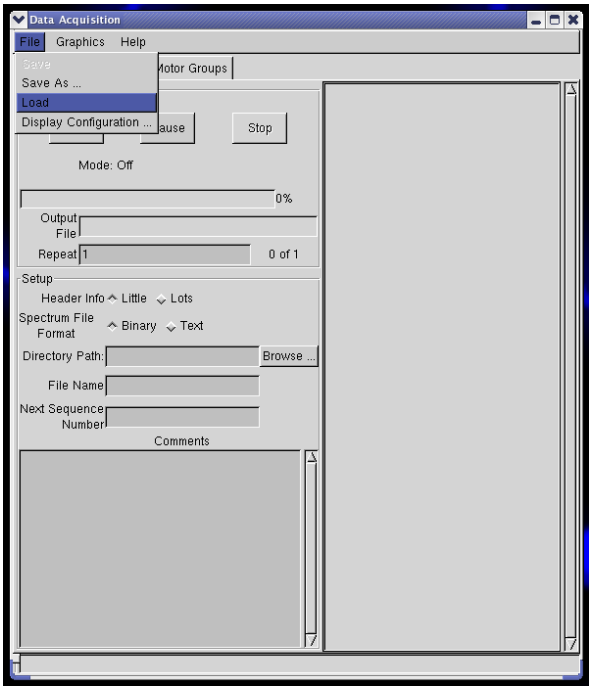
XEOL and XAS data collection in Step Scan configuration

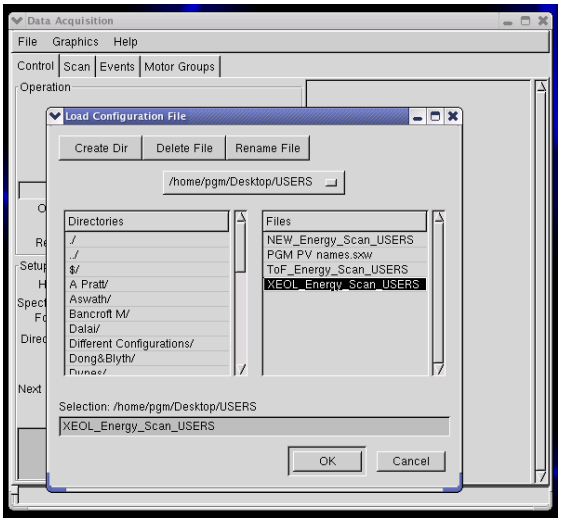
Open the “USERS DATA ACQUISITION” GUI by double clicking on the icon:





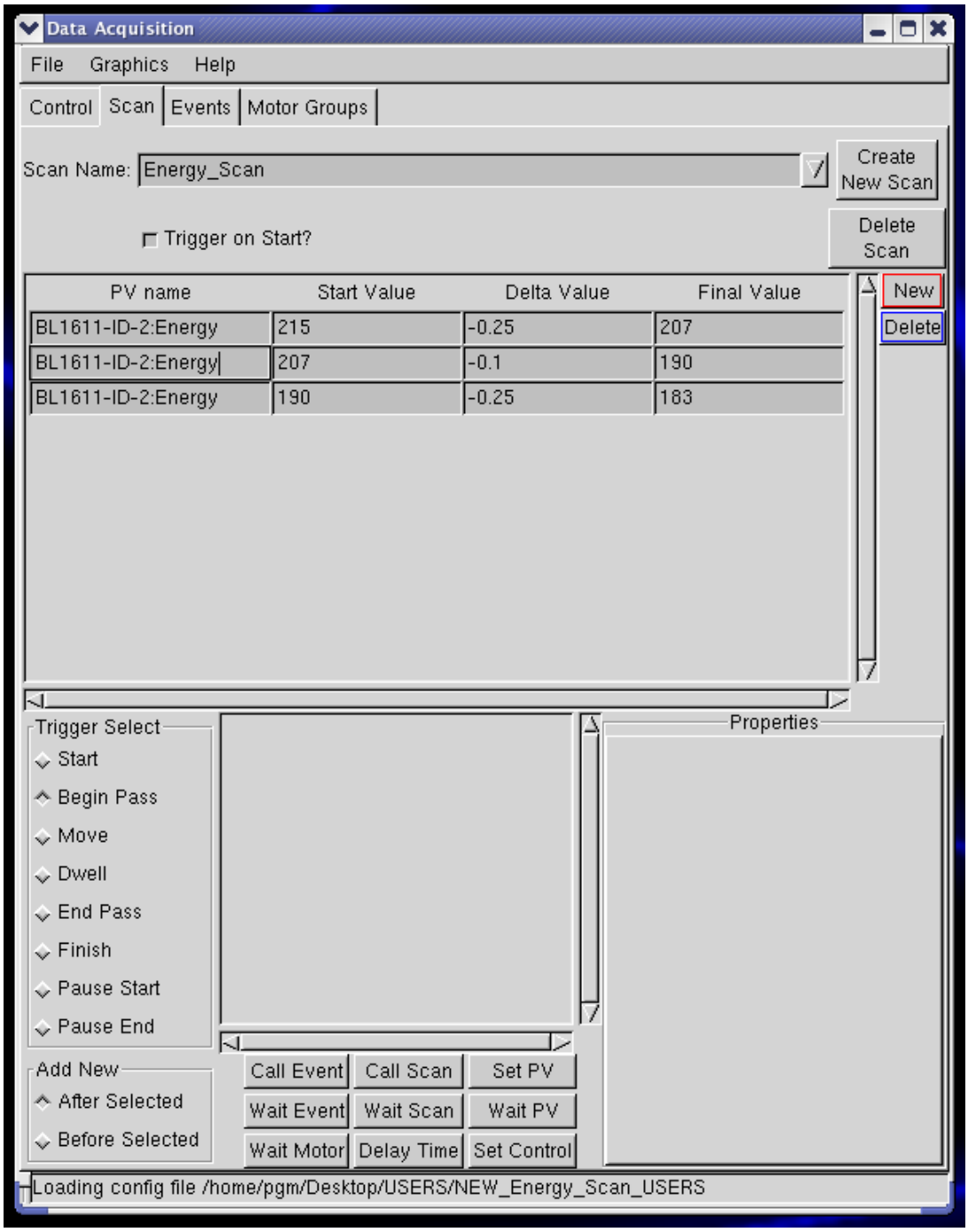
From the “File” menu Load the configuration file “XEOL_Energy_scan_TopupVetoed_USER”
Work your way down the directory tree to **/home/pgm/Desktop/0 USERS**.



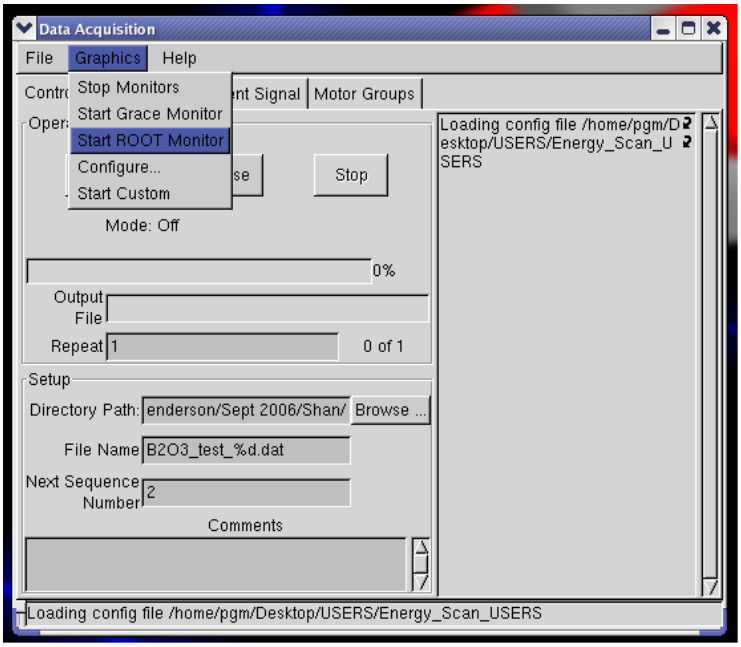


In the *Scan* tab make sure you are scanning over the right energetic range, from high (Start Value) to low (Final Value) energy, with a negative step (Delta Value).

You can also scan over several consecutive regions with different steps (delta values).
To add a new region click the New button.
To delete a region click the Delete button.

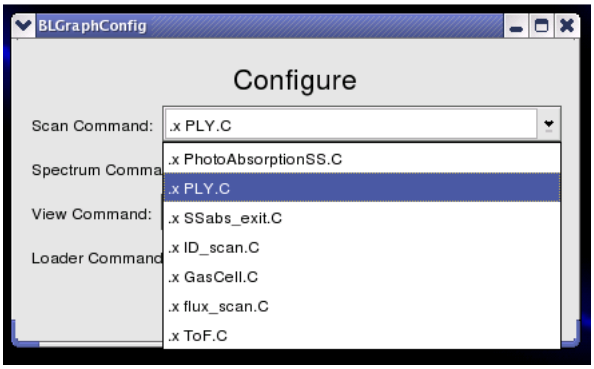


To visualize the data while acquiring, select “Start ROOT Monitor” from the “Graphics” menu





Click on Configure, select PLY.C, and then Close.



Keep the BLGraph GUI always open, DO NOT “Quit”. A new plot will start automatically at the beginning of each scan.

Next, check the settings in the Control Tab:
User data are generally saved in ‘/home/pgm/Desktop/0 USERS’ under your own directory.
Click “Start” in the Control page of the Data Acquisition when you are ready to scan.

Beamline Troubleshooting

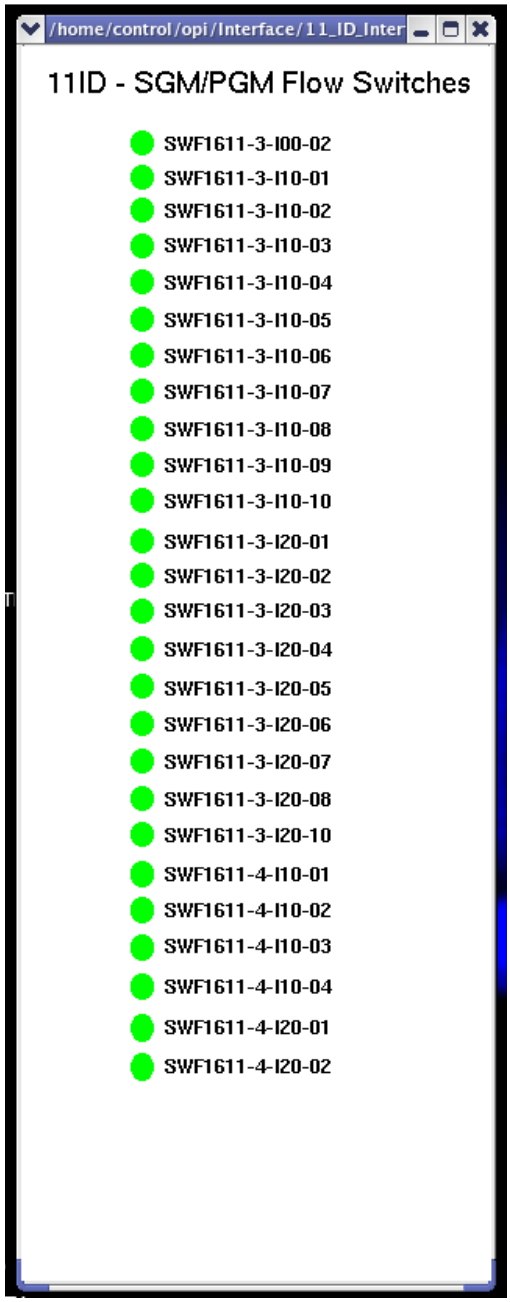
To be performed with Floor Coordinator assistance

ISSUE: The Photon Shutter 2 (PSH2) does not open -1-

The Beamline has been enabled by the Control Room but you cannot open PSH2

Open the SGM/PGM Flow Switch Display by double clicking on the icon:





Check that ALL the Flow-Switch indicators are green.

The indicators beginning with SWF1611-3-* are water cooling indicators inside the SGM/PGM hutch.

The SWF1611-4-* are water cooling indicators outside the hutch.

If any of the indicators is red you will have to contact either

- the beamline staff or
- the Floor Coordinator (FC).

The FC will contact the on-call mech-tech.

The on-call mech-tech will re-equilibrate the water flow along the beamlines.

Once done, all the indicators are green and you can open all the Shutters and proceed as normal.

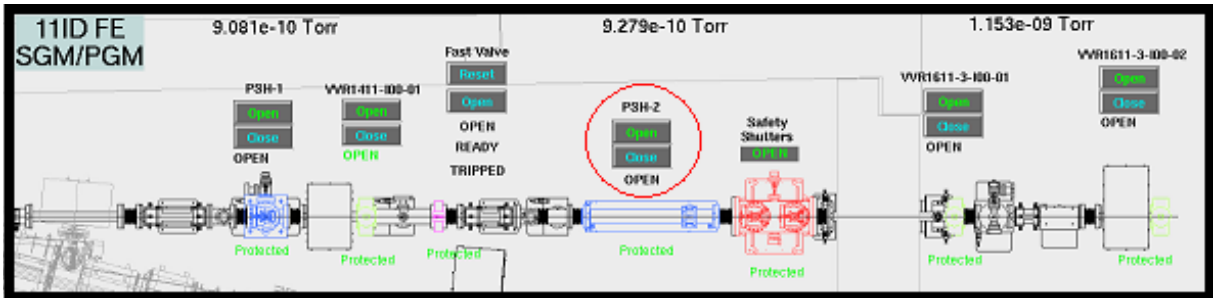
ISSUE: The Photon Shutter 2 (PSH2) does not open -2-

If after checking that all the water indicators are green you are still unable to open PSH2, open the “PGM FRONTEND” and “PGM BEAMLINE” by double clicking on the icons:



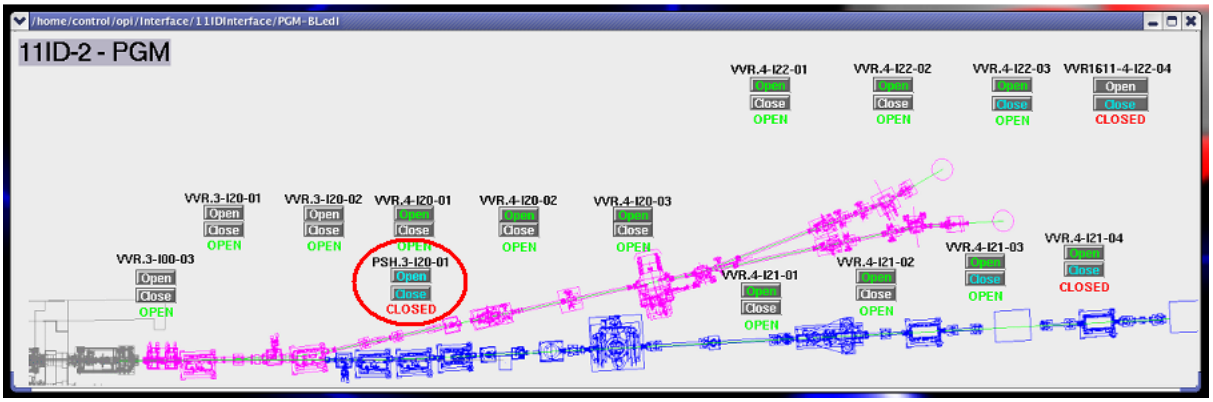


The “PGM FRONTEND” panel:



Check that the beamline has been **ENABLED** by the control room (ask the FC) and that the Safety shutter is **Open.** If **Closed**, open the Safety shutter and the others shutters as instructed in page 11 of this manual.

The “PGM BEAMLINE” panel:



Check that all valves (VVR.*), with the exception of VVR1611-4-I21-04 and VVR1611-4-I22-04, are **OPEN** on the PGM frontend and PGM beamline panels, as shown in the pictures.

If closed, **OPEN** the Shutter 3 (PSH.3-I20-01) on the PGM beamline panel, as described on page 11 of this manual.

ISSUE: XAS Sample dropped in the Loading Chamber

Has a sample dropped in the loading chamber?

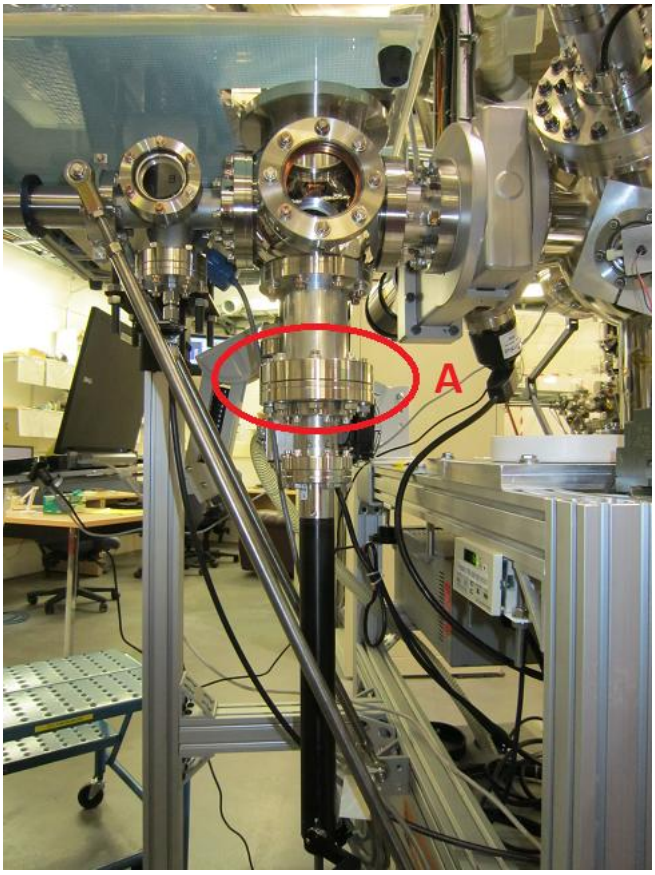
Stop your experiment!

In that unfortunate event you must controlled vent the Load-lock chamber following the instructions on page 9, and contact

- the beamline staff, or
- during unsociable hours the Floor Coordinator (FC) and ask the FC to phone the on-call mech-tech.

---

*Users **are not allowed** to perform any of the following!*



Procedure (for BL Staff and/or Techs):

1) Ask the users to bring the load-lock section up to air, if they haven't already done so.

2) Disconnect Flange **A** and lower the multiple sample ladder.

3) Retrieve the dropped sample from the bellow and/or load lock area.

4) Using a new gasket, reconnect Flange **A**

5) Once connected, ensure that the multiple sample ladder is properly aligned, such that the transfer manipulator is able to pick up samples from all the three ladder positions. Users should test this while Techs are still there.

At this point Users should be able to continue with the experiment by replacing the dropped sample(s), or loading new sample(s) onto the multiple sample holder, and pumping the load-lock section as instructed.

Sample Preparation procedure

The following is a guideline on how to prepare samples using the provided sample holders.

The BL staff at the beginning of your beamtime will give you:

- 6 sample holders that have been previously thoroughly cleaned using the procedure in Appendix A
- A roll of double sided carbon tape
- Clean lab tools (e.g. tweezer, spatula, scissors, mortar & pestle)

In the assigned lab, and workstation, you will find Kim-wipes, gloves, beakers, solvents (acetone and methanol) and a can of compress air.

If any of the material is missing, please ask.

Sample preparation

1. Take a clean sample holder and place it on a clean large Kim-wipe.

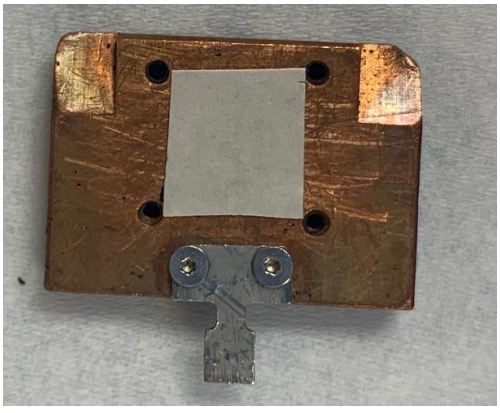
The Kim-wipe will be through in the garbage once the sample holder has been prepared, avoiding sample's spilling to contaminate subsequent sample holders.

2. Cut one piece of double sided carbon tape and tape it on the sample holder, Picture 1.

The size of the sample must not be bigger then the area between the 4 screw-holes on the holder.

Indicatively, if the sample is a solid piece or has been prepared in a pellet form, the size must not be larger then 0.8cmx0.8cm. If the sample is powder, it must not be spread anywhere outside the 4 screw-holes area.

If the sample is a solid piece (e.g. steel plate for tribology sample), it cannot be thicker the 4.5mm.

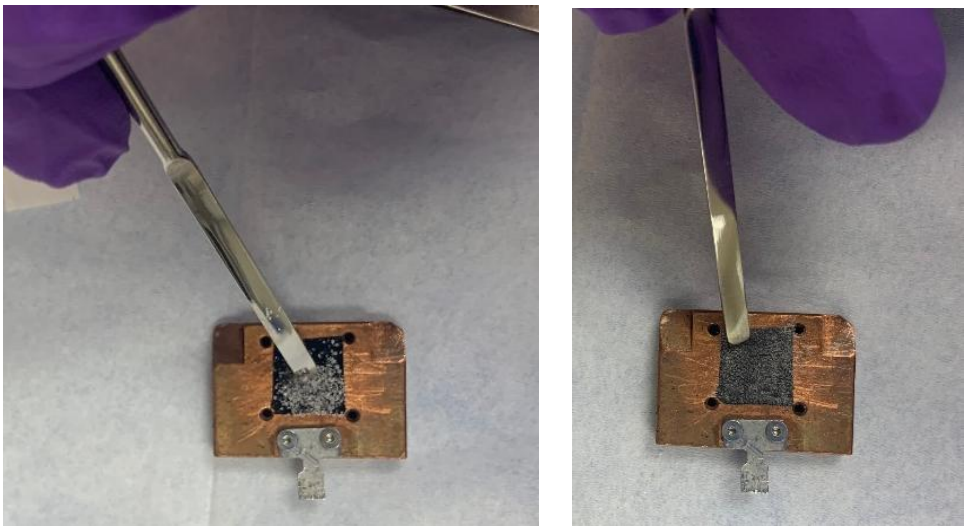


Picture 1: Sample older with attached the double-sided carbon tape, this is the maximum allowed size of C-tape. It can be smaller. It CANNOT be bigger.

3. Attach the sample onto the carbon tape. If it is powder, with a clean spatula place a small amount on the tape and smear it as uniform as possible to form a very thin layer covering the full tape, Picture 2.

If the powder sample is coarse, use clean mortar and pestle to grind it into a finest powder.

Remember to thoroughly clean the tools you used before moving to a new sample, wiping with several clean acetone soaked Kim-wipes and to replace the large Kim-wipe.



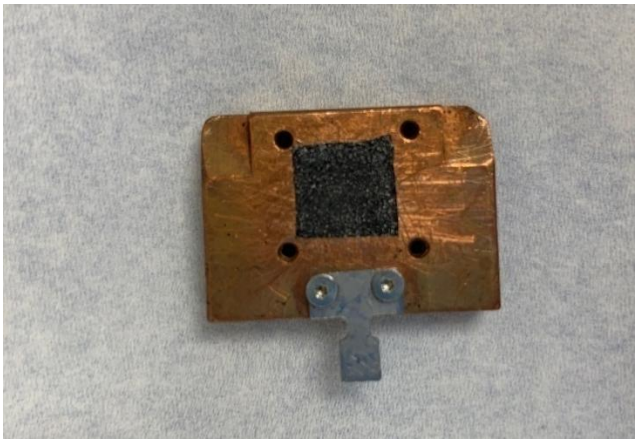
Picture 2: Powder samples on carbon tape

4. Use the compressed air can to gently remove the exceeding sample powder not glue to the tape.

This action prevents the loose powder to float in the experimental chamber, contaminating the chamber and detector's surfaces and worsening the vacuum. Do not old the sample above other samples when spraying the air to avoid powder to deposit on top of clean tools, clean sample holders or already prepared sample.

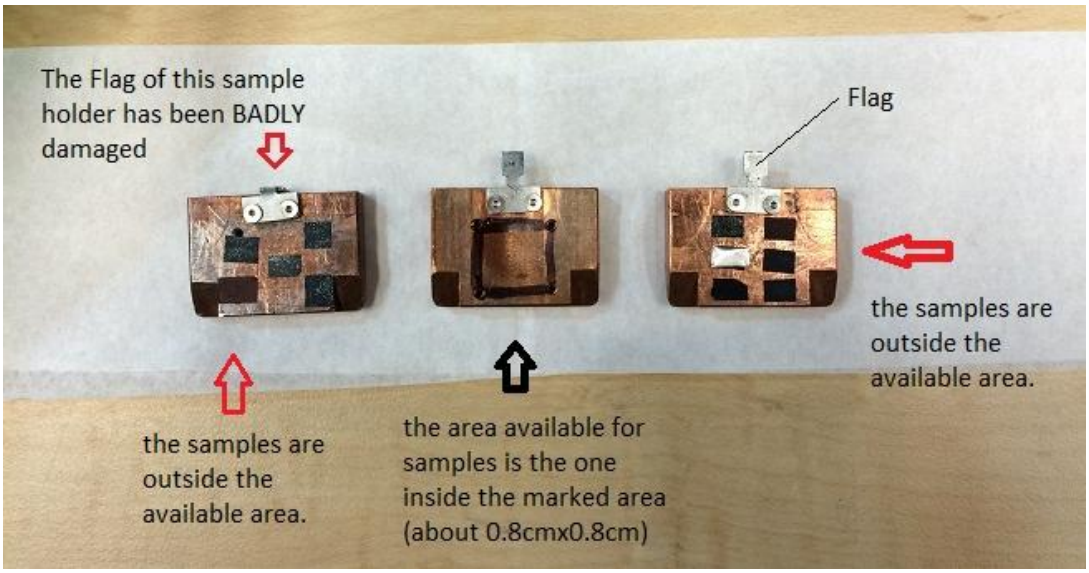
Be shrewd.

An example of how your sample should look like is shown in Picture 3.



Picture 3: Nicely prepared sample – how it should look like ☺

Picture 4 shows how your sample should NOT look like.

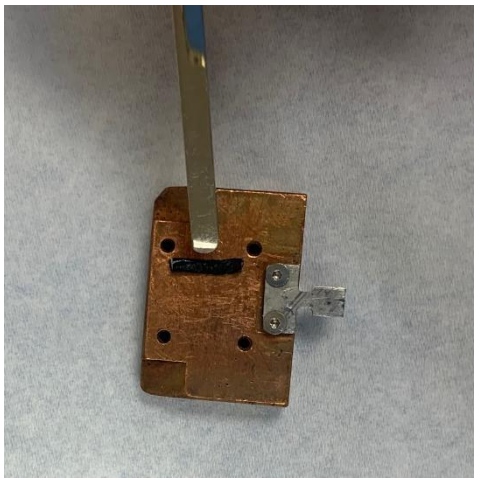


Picture 4: How NOT to mount samples ☹

Remember to always thoroughly clean the tools you used before moving to a new sample wiping them with several clean acetone soaked Kim-wipes and to replace the large Kim-wipe.

Cleaning the sample holder

a. To remove the “tape+sample” from the holder, after that particular sample has been analysed, use the spatula or the tweezer to first remove the carbon tape, Picture 5.



Picture 5: Removing of the tape

b. Wipe the sample holder with an acetone soaked Kim-wipe
c. Repeat (b)
d. Wipe the sample holder with a methanol soaked Kim-wipe
e. Repeat (d)

The procedure should completely remove any traces of carbon tape and glue from the holder.

Remember that the tape’s glue presents traces of chemicals (e.g. Boron) that could interfere with your results.

Occasionally during the beamtime is necessary to thoroughly clean the sample holder. This is the procedure followed by the BL staff between every Users group.

i. Place the sample holder in an acetone filled beaker, just enough to fully submerge the holder, Picture 6



Picture 6: Beaker with an acetone fully submerged sample holder

ii. Place the beaker in the ultrasonic cleaner, switched it on setting the timer to 10 minutes, Picture 7.

Remember the ultrasonic cleaner has to be fill with some water before switch it on. If you never used one, ask for help.



Picture 7: Ultrasonic cleaner with some water and the beaker containing the sample holder and the acetone

iii. Take the sample older out from the beaker, rinse with acetone, wipe it dry

The sample holder is now clean and ready to be used.

VLSPGM Staff availability

- **9am – 5pm, Mon to Fri excluding Holidays**

  Staff are on-site or able to respond at short notice.

- **10am – 10pm, Weekends and Holidays and**

  5pm - 10pm, Week days

  By email. A response is not guaranteed.



After 10pm, BL staff cannot be reached.

If you need assistance, it is strongly encourage you send an email to the Staff and to contact the Floor Coordinator (x3639), possibly after having familiarized yourself with the BL manual troubleshooting section.

In the event the Floor Coordinator (FC) is familiar with the BL, they may be able to offer assistance. However, the FC is not authorized to perform technical fixes, and Users should not expect the FC to solve technical problems.



Support Scientist
Daniel.Correia@lightsource.ca
x 3775



BL Responsible - Senior Scientist
Lucia.Zuin@lightsource.ca
x 3724