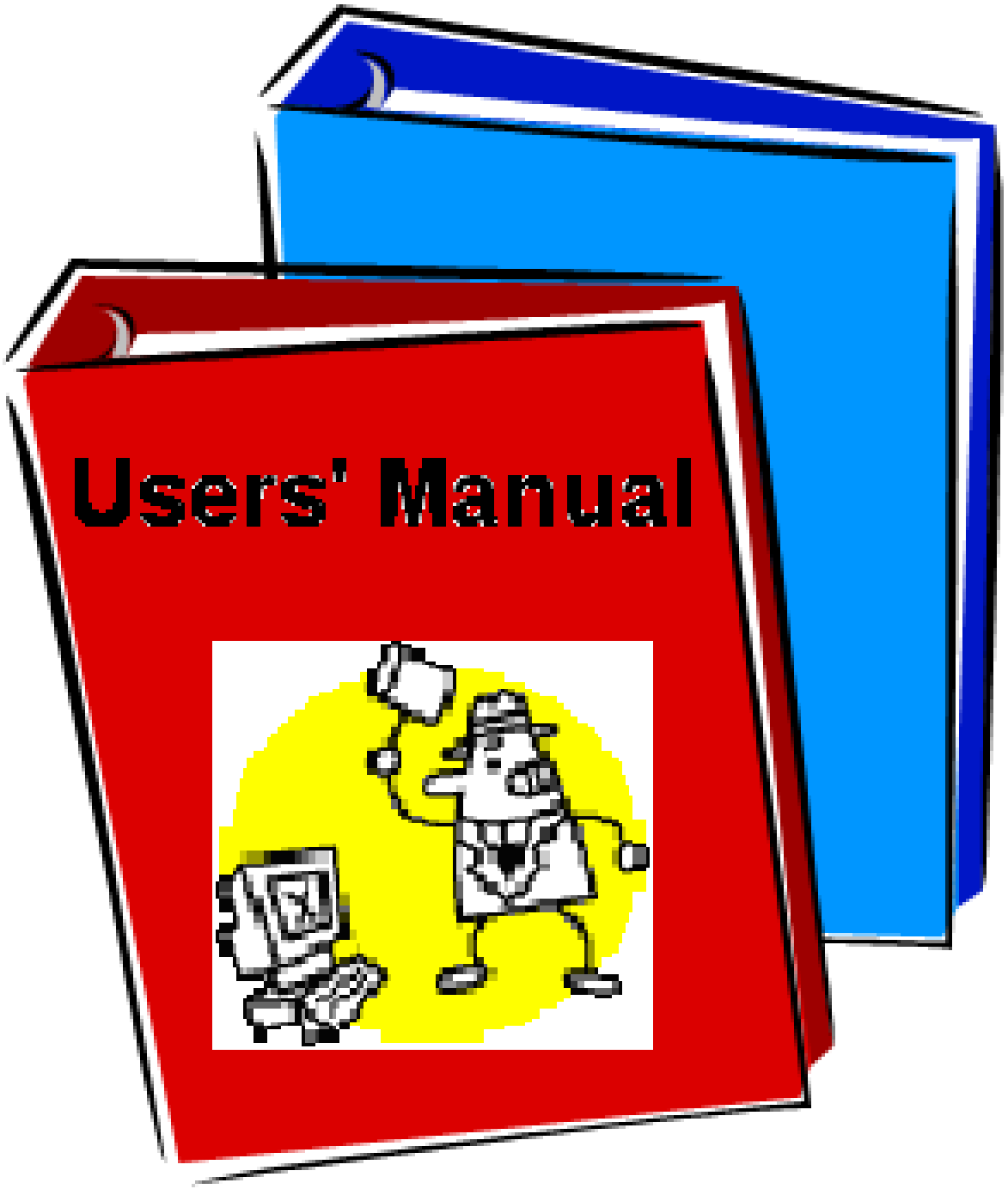
Please read carefully this manual!



Please read carefully this manual!

The instructions to independently run your experiment and to troubleshoot the most frequent problems have been exhaustively addressed.

During unsociable hours call the Floor Coordinator x3639 for assistance.

11ID-2 VLS-PGM Manual

Start the “PGM Control Panel” by double clicking on the icon:

On the left hand-side of the panel there are:

1. The switches of the High Voltages apply to the Branch A. Here you turn ON/OFF the HV FL before & after a scan when the Absorption Chamber is on Branch A.
2. The switches of the High Voltages apply to the Branch B.
3. Active Branch-line Selection (A or B).
   Before start the run make sure the right Branch-line is selected.
4. The state (Closed/Open) of the Photon Shutters along the beamline.
5. The state (Closed/Open) of the End-Station gate valve, for both branches.

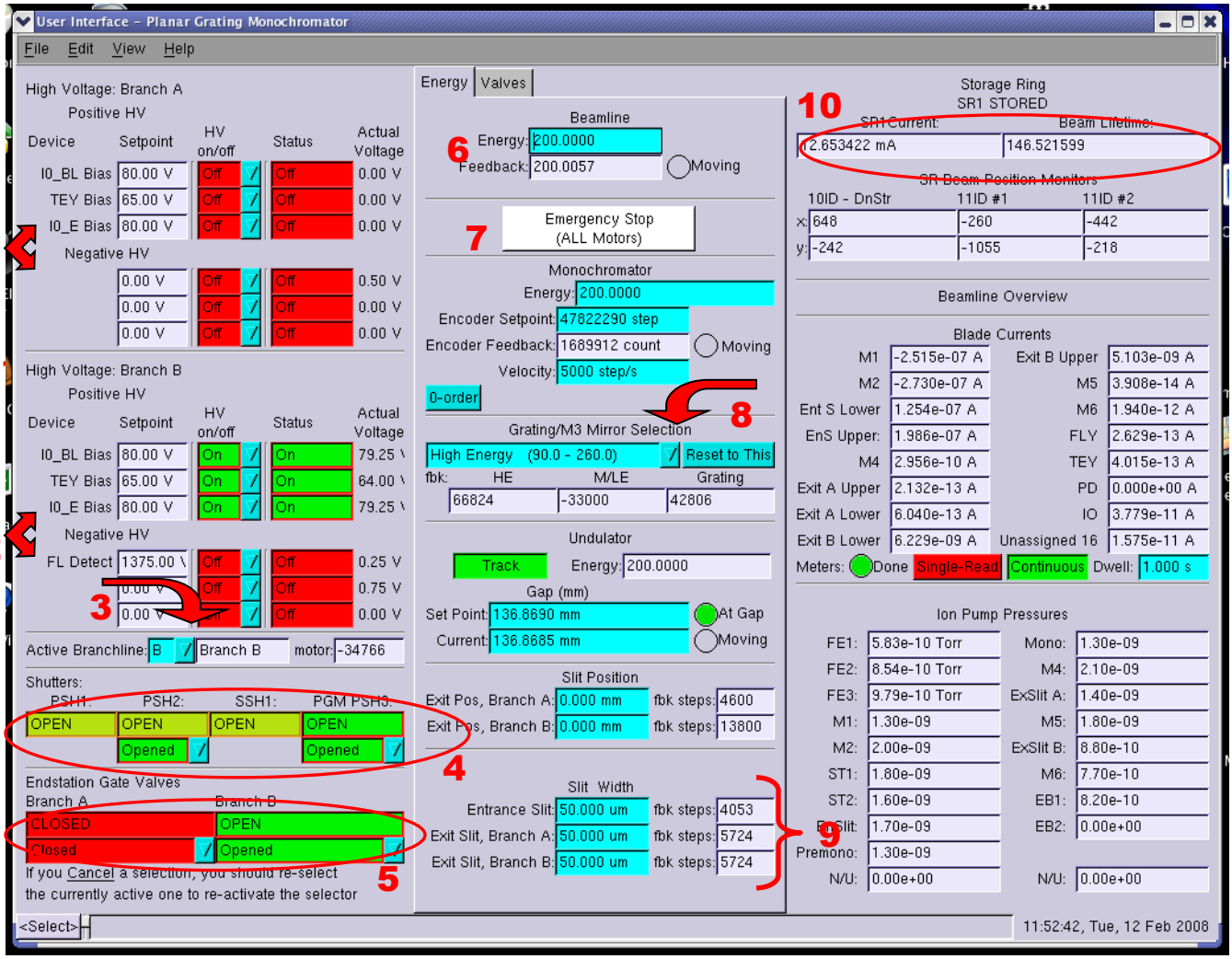
In the central part of the panel there are:

6. Energy Selection in eV, and related feedback. Once you start a scan the “User Data Acquisition Program” automatically sets this energy to the requested value.
7. Emergency Stop Button; it stops any moving motor in the “PGM Control Panel”
8. Grating Selection (High; Medium; Low).
   Before start the run make sure you are working with the right grating.
9. SLIT WIDTH selections in µm (5-250 µm). The Entrance Slit (common for both branches) and the Exit Slits Branch A or B.

The right hand-side of the panel gives indication about:

10. The Ring Current and Beam Lifetime

and a general Beamline Overview



To change the grating to the desired range

1. Select from the Grating/Mirror Selection box **8** the desired grating.
2. Wait till all the three numbers in the white boxes (HE; M/LE and Grating) have stopped! They represent the motor step positions for the optical components inside the monochromator.
3. Type in the turquoise box **6** an energy close the value you need for your scan, this will adjust the Undulator to work in the range defined by the grating.

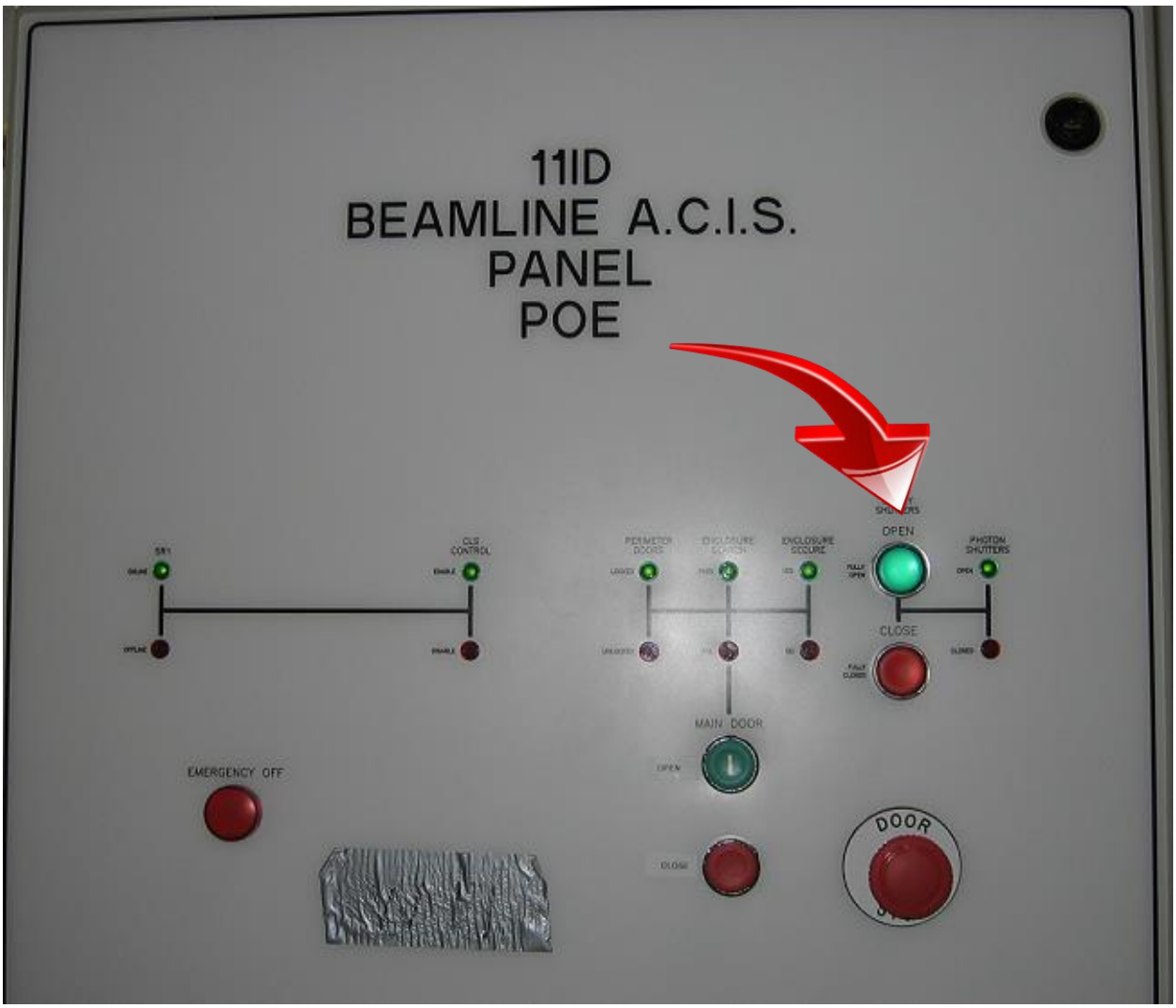
Always remember that this process is TIME CONSUMING ~5 minutes

Since December 2014 the Safety Photon Shutter (SSH1) can be open from the PGM control panel – User interface. If you do not succeed follow the following instructions:

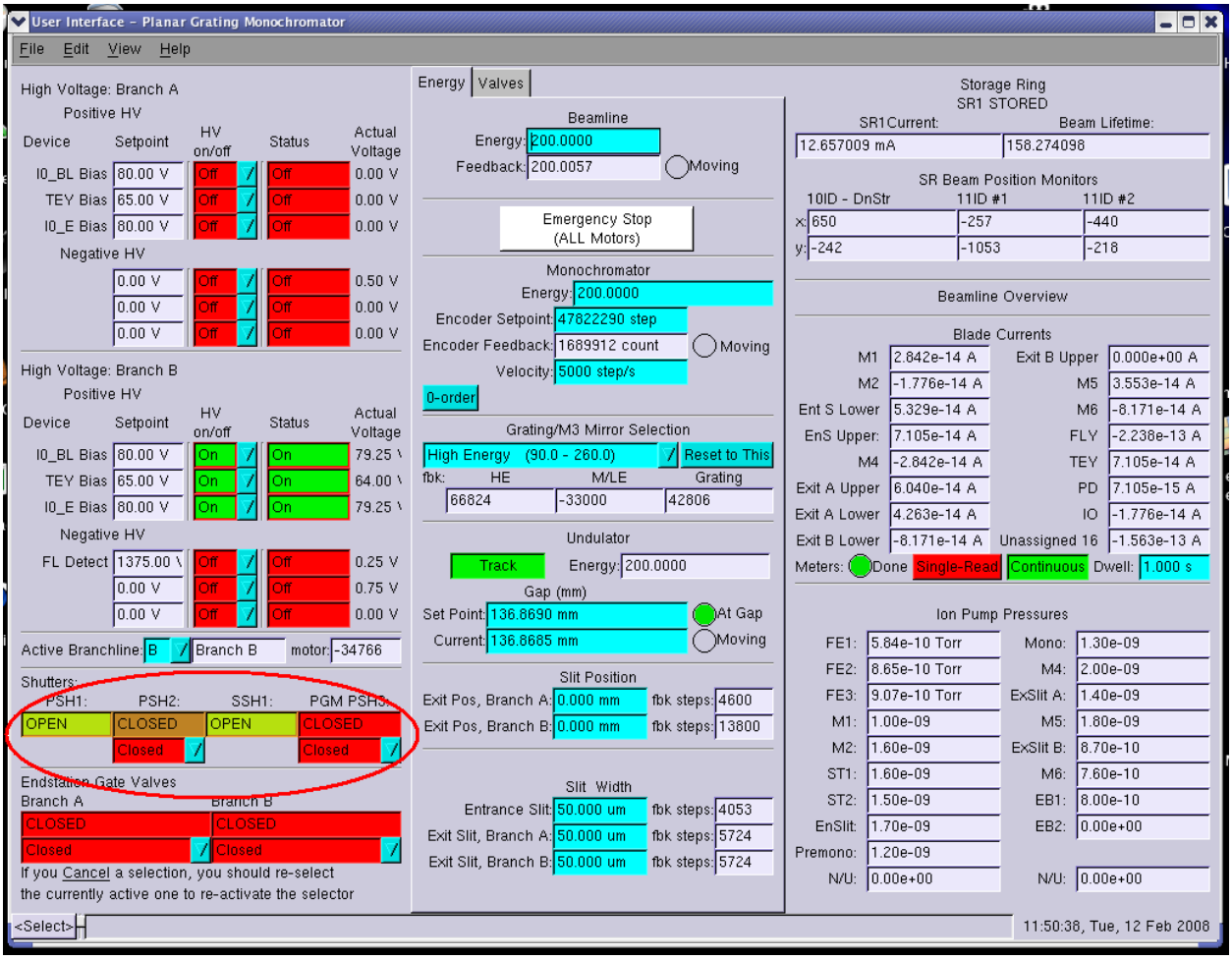
Things to do AFTER EVERY INJECTION

After every injection as soon as the control room has enabled the beamline and BEFORE start a new data acquisition you will have to **OPEN** few Photon Shutters.

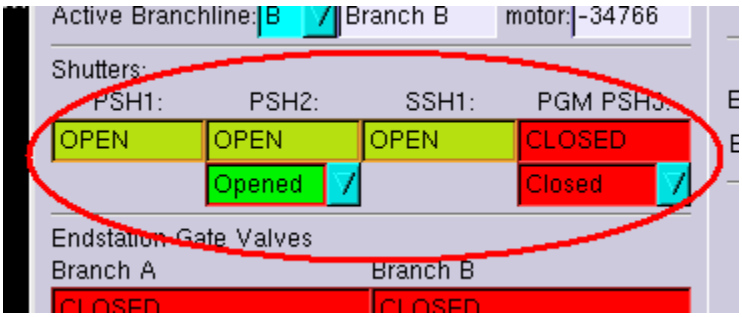
1. First thing to do is MANUALLY **OPEN** the Safety Photon Shutter (SSH1) by the panel outside the hutch pushing the green button.



It is possible check the state of the shutter from the PGM control panel – User interface



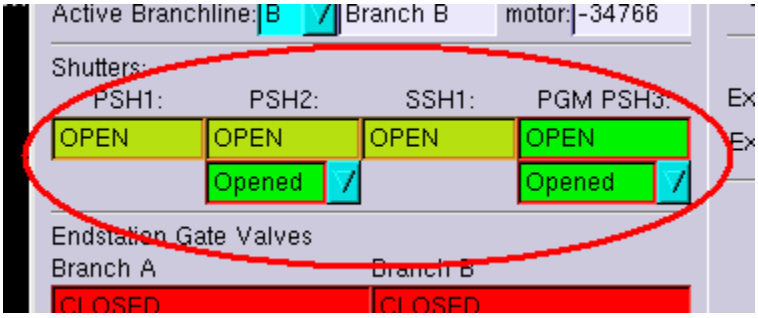
2. Next, **OPEN** the Shutter two (PSH2)



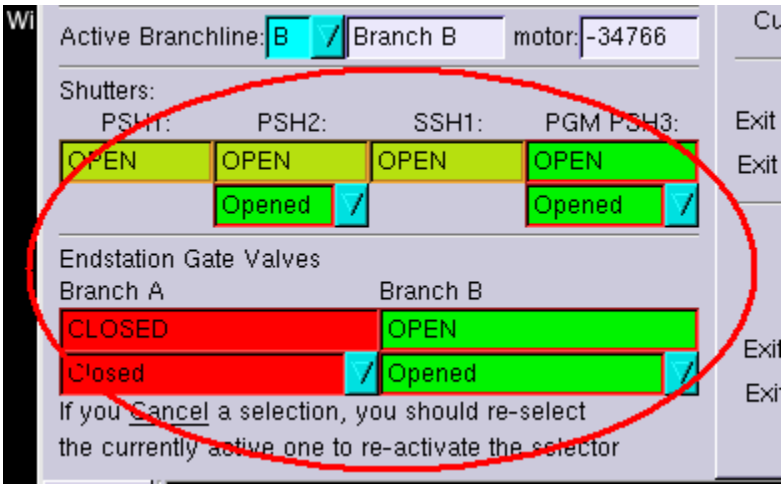
NB: this shutter (PSH2) is in COMMON with the SGM beamline.

Closing it could interrupt/jeopardize the SGM Users' experiment!!!

and the Shutter three (PGM-PSH3) from the PGM control panel – User interface.



3. Last, **OPEN** the End-Station gate valve of the Branch-line you are using. The light is now hitting your sample.



DATA ACQUISITION

VLS-PGM provide alternative forms of data acquisition scans for its standard XAS measurements. Users can choose the configuration best suited to their study.
The differences between Step and Fast scans are briefly outlined:

Step scan

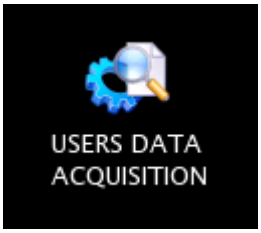
- Users decide the energy range, dwell time and step size for each scan.
- Each measurement occurs after the motors have stopped.
- The total duration of a typical 1sec dwell time scan is largely caused by the dead time required for starting and stopping the motors at each energy point.
- The points are equally energy spaced by the value input as Delta Value.
- Typically the duration of a 25eV scan is ~20minutes

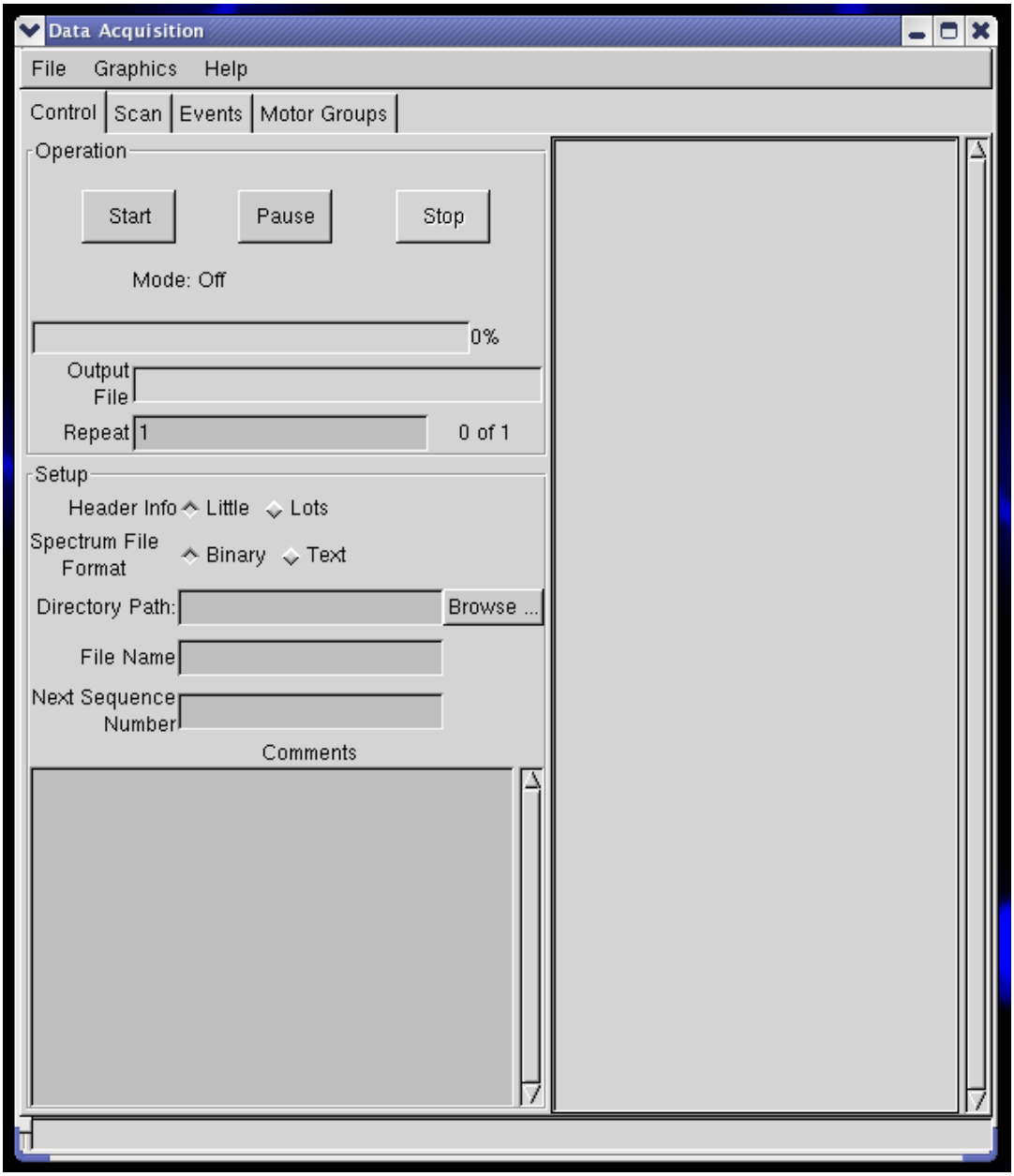
Fast (on-the-Fly) scan

- Energy range, dwell time and step size for each scan are pre-set and not changeable.
- The motors start running to the final point and the instrumentation recording I0, TEY and FLY are sampled along the motion at consistent measurement times (1sec).
- The provided “mean Energy fbk” value should be used to analyze and plot the resulting spectrum.
- The points within a scan are not equally energy spaced.
- Typically the duration of a 25eV scan is ~5minutes

USERS DATA ACQUISITION

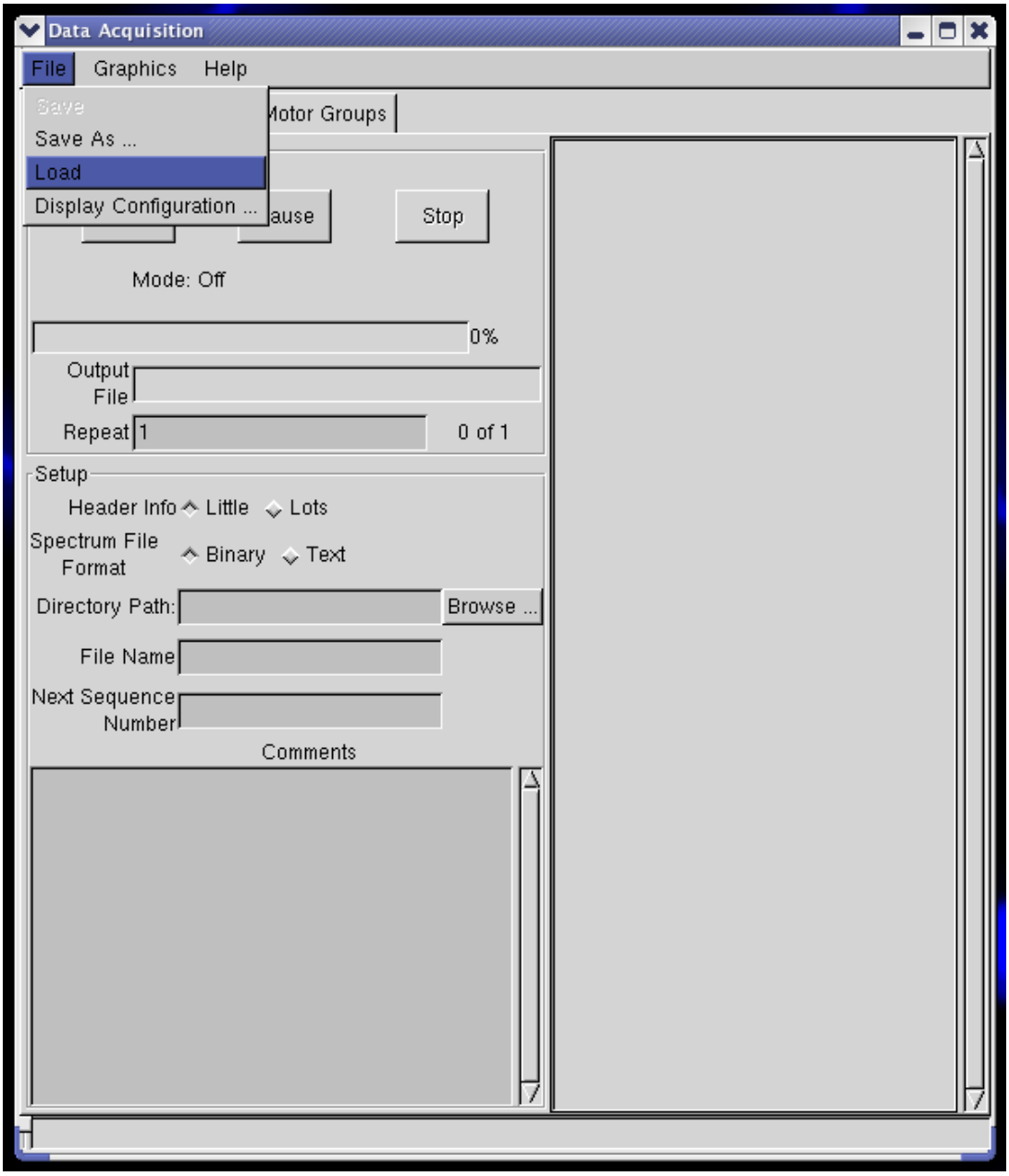
Start the “USERS DATA ACQUISITION” by double clicking on the icon:



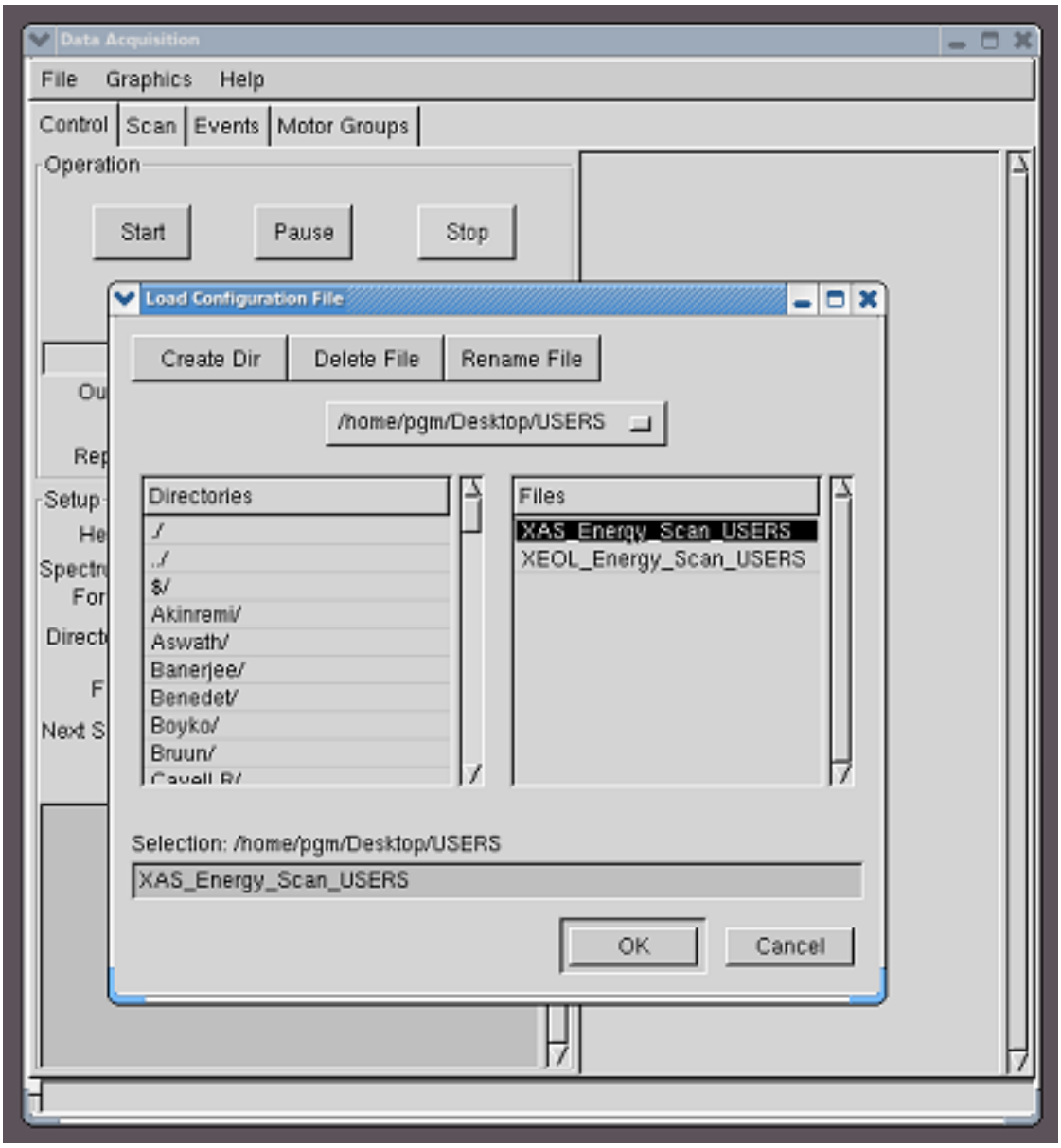


Step scan:

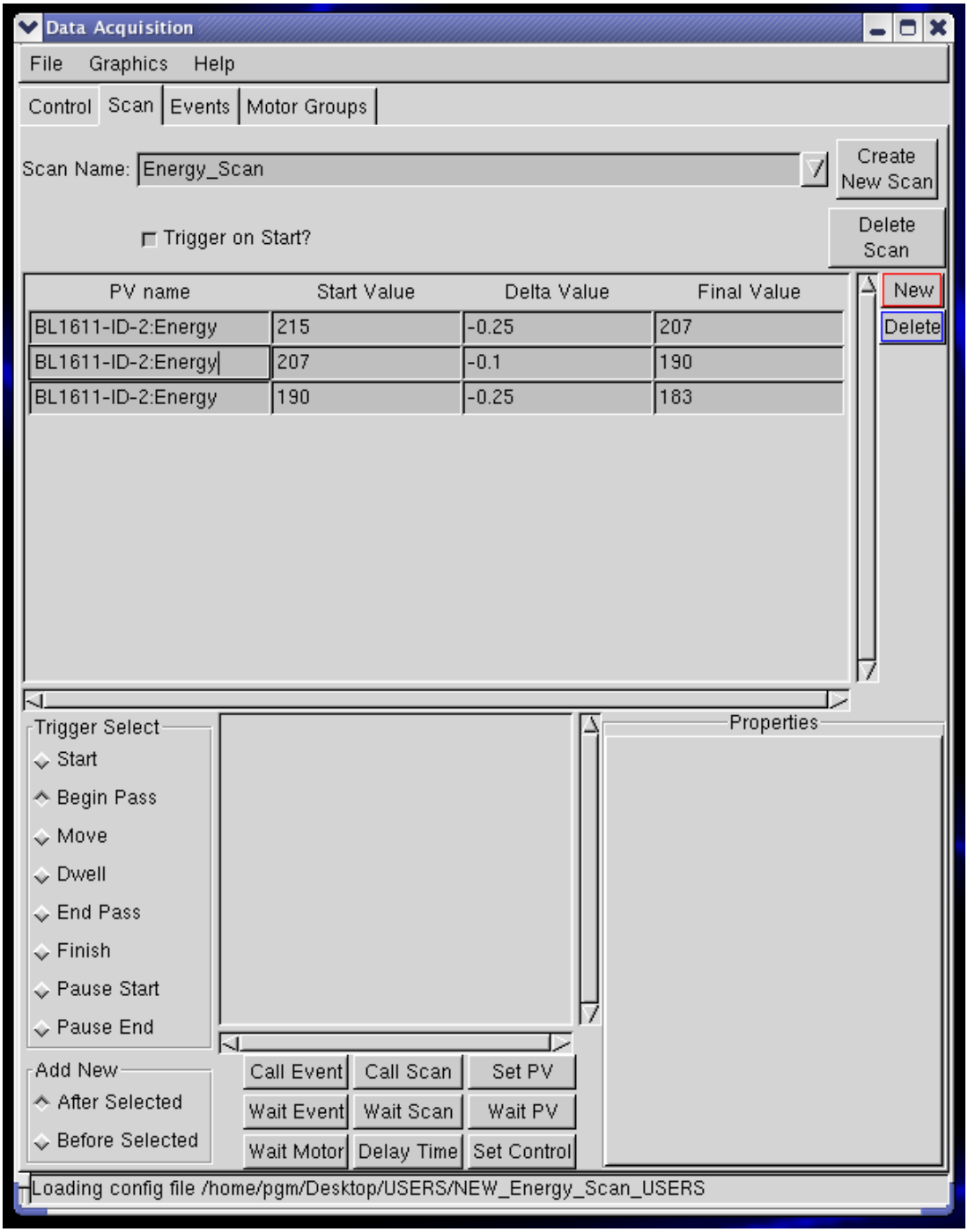
From the “File” menu Load the configuration file “XAS_Energy_scan_USER”



Work your way down the directory tree until you see the configuration file required. this file is in the ‘/home/pgm/Desktop/USERS directory (folder)

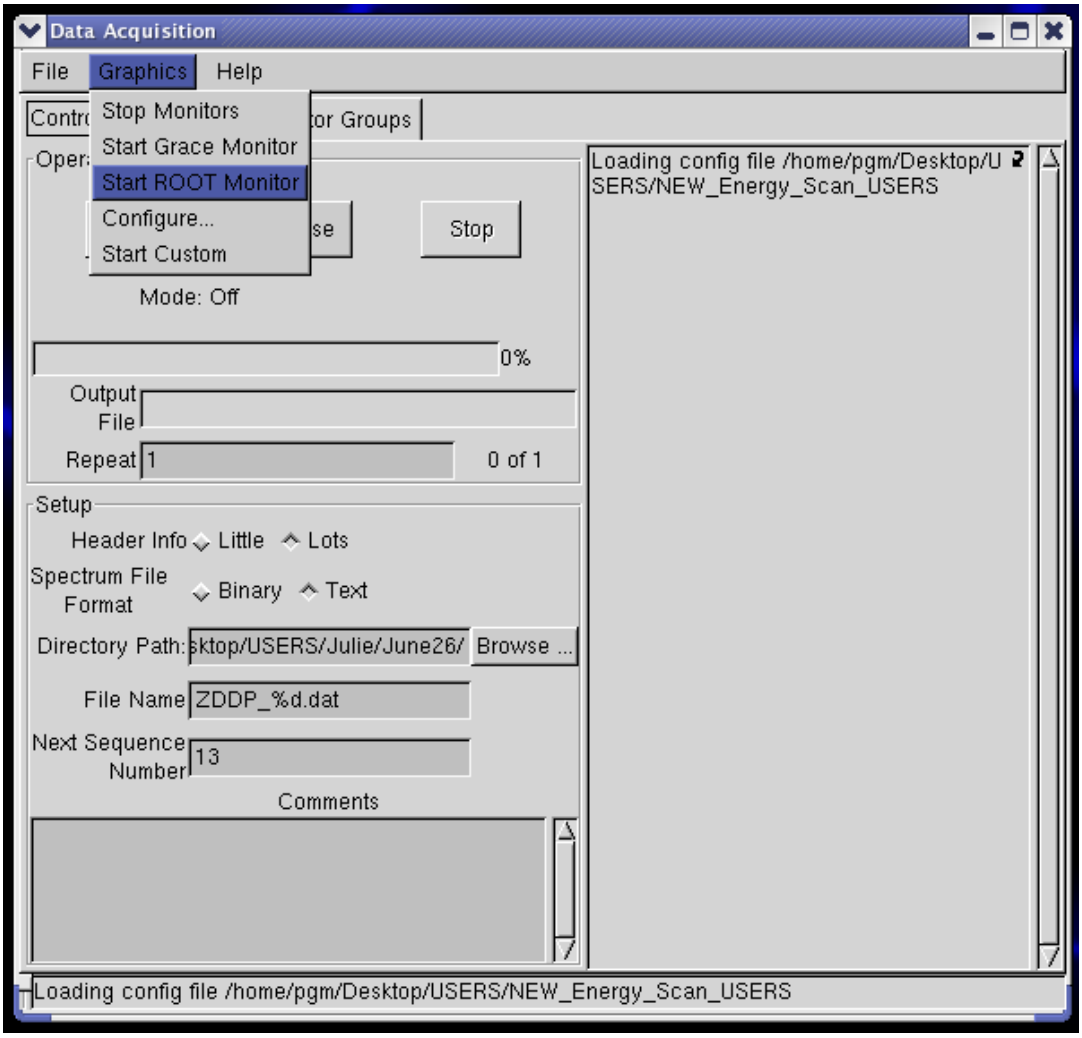


In the Scan tab make sure you are scanning over the right energetic range, from higher (Start Value) to lower (Final Value) energy, with a negative step (Delta Value).

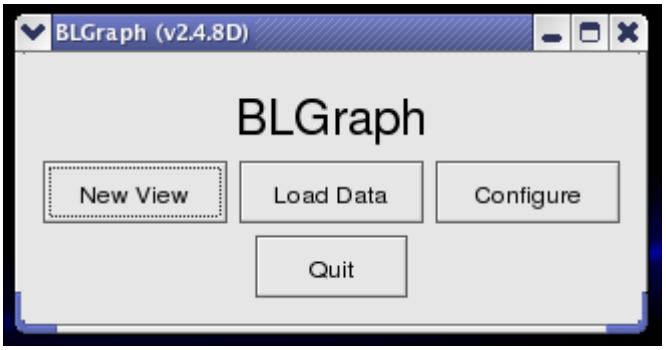


You can also scan over several consecutive regions with different steps (delta values).
To add a new region click the New button.
To delete a region click the Delete button.

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



This window will pop up:

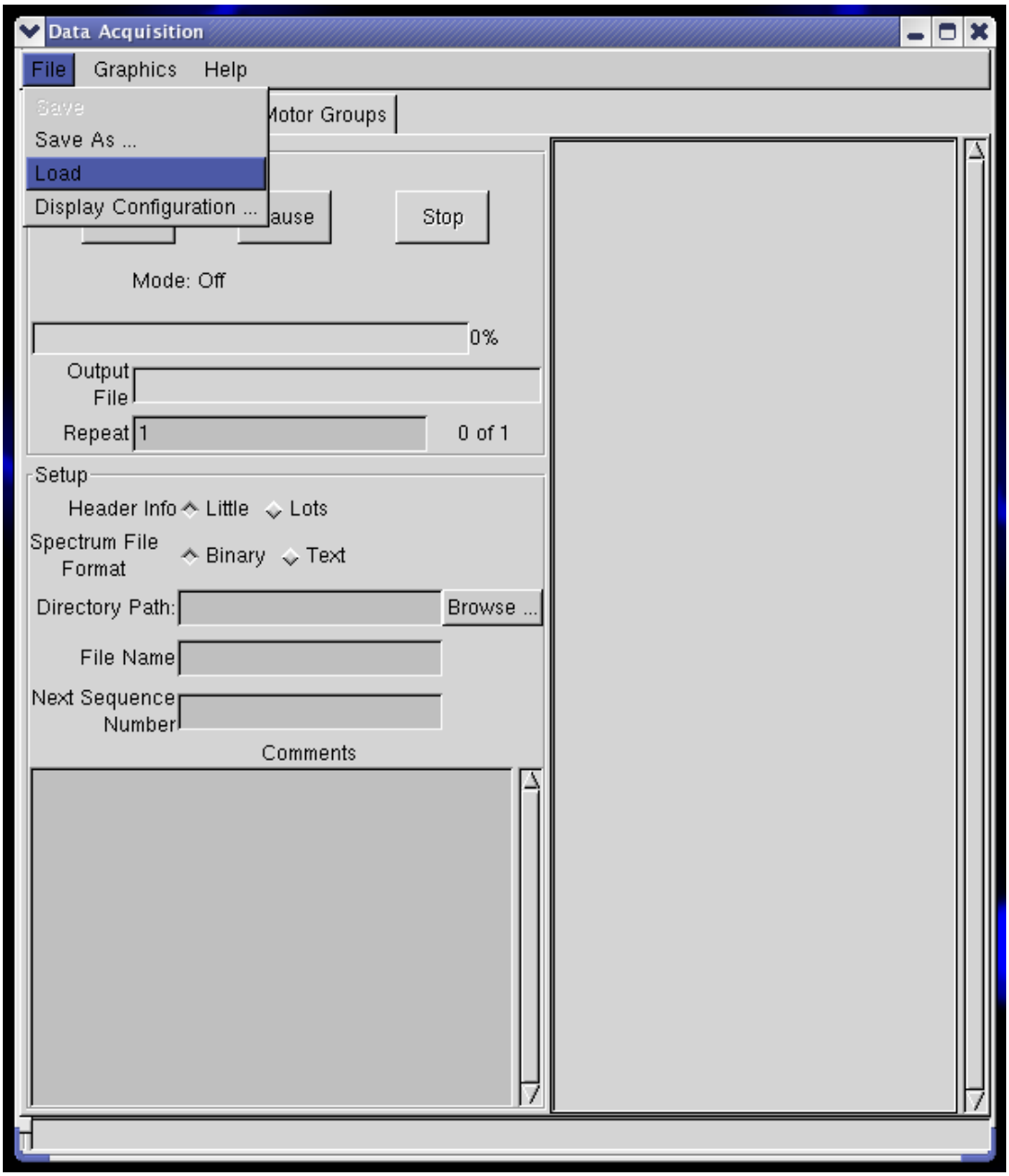


Keep the BLGraph window always open, DO NOT press the “Quit” button.
Automatically a new plot will start at the starting of each scan.

Next, check the settings in the Control Tab:
User's data are generally saved in '/home/pgm/Desktop/USERS' under your own directory.
The "Directory Path" shows where yours file will go
In the "File Name" the symbol "%d" will give you sequential file numbers for sequential runs.
Click "Start" in the Control page of the Data Acquisition when you are ready to scan.

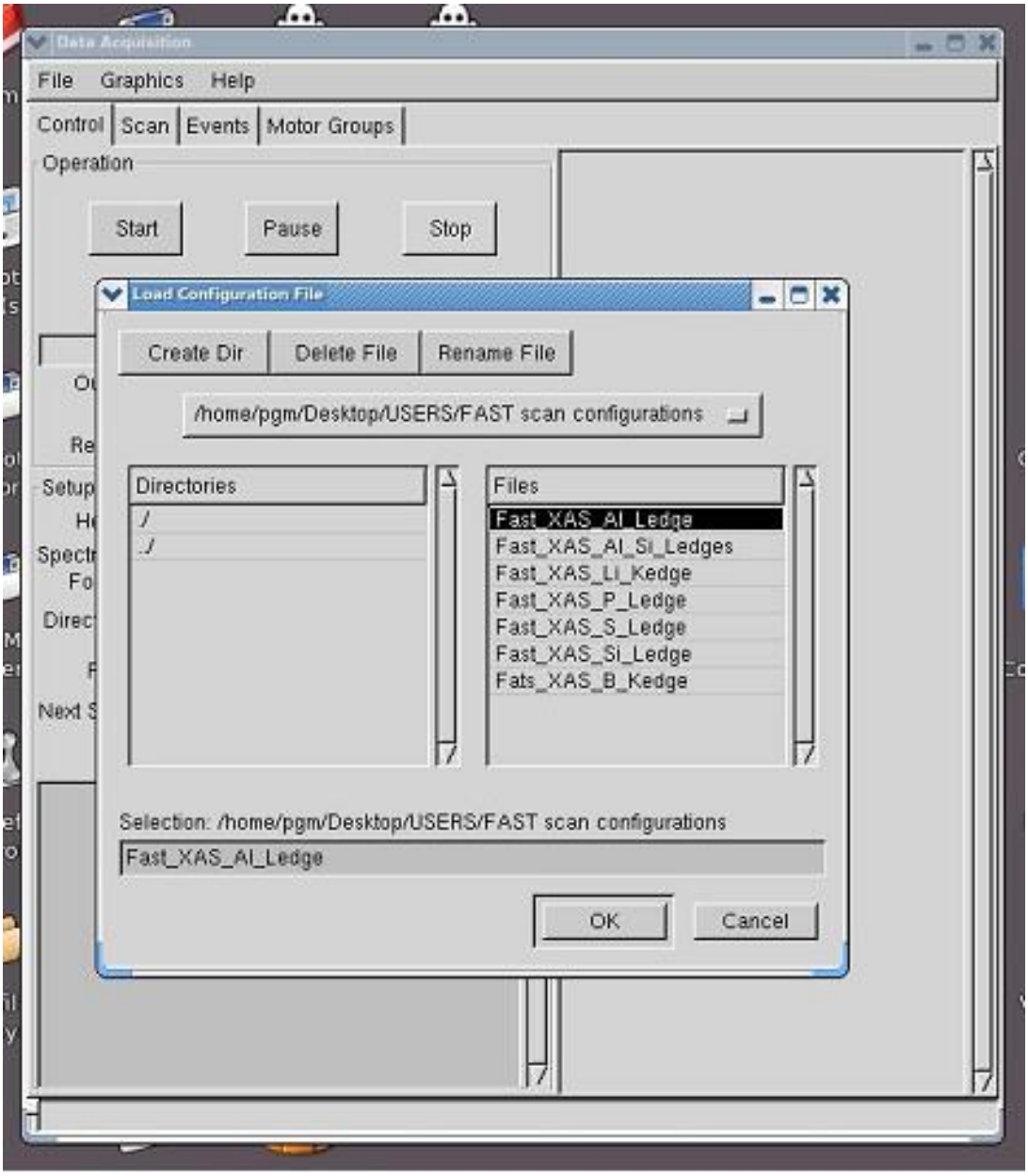
Fast (Fly) Scan:

From the “File” menu Load the configuration file of the region you are interested in:



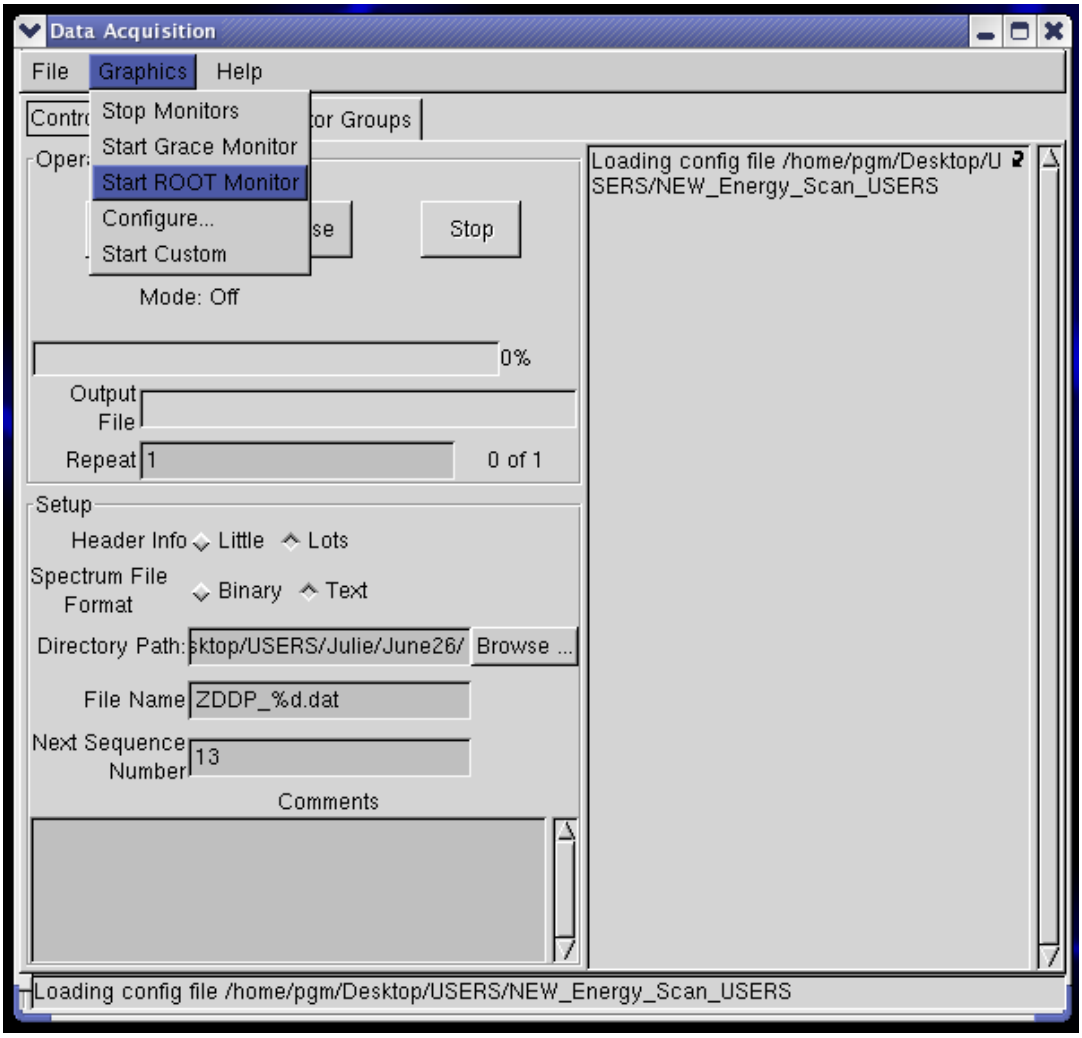
Work your way down the directory tree until you see the configuration file required.
this file is in the '/home/pgm/Desktop/USERS/FAST scan configuration' directory (folder)

"Fast_XAS_Li_Kedge"	[75-47.5eV]
"Fast_XAS_Al_Ledge"	[90-70eV]
"Fast_XAS_Al_Si_Ledges"	[120-70eV]
"Fast_XAS_Si_Ledge"	[121-95eV]
"Fast_XAS_P_Ledge"	[156-130eV]
"Fast_XAS_S_Ledge"	[193-158eV]
"Fast_XAS_B_Kedge"	[210-185eV]

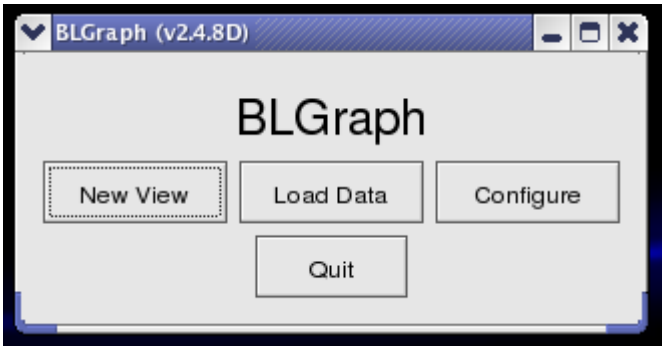


For this scan you cannot choose the energy range, as it is pre-fixed.

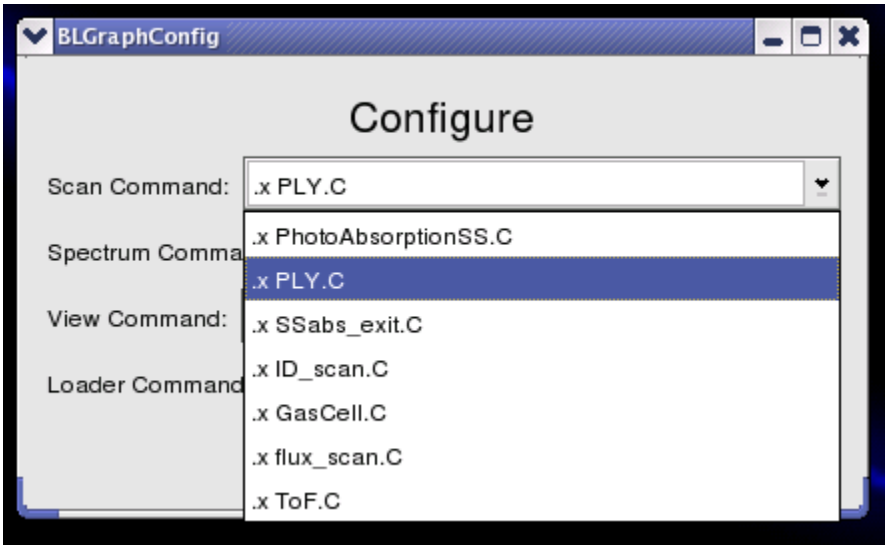
To visualize the data while acquiring, select “Start ROOT Monitor” from the “Graphics” menu



This window will pop up:



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



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

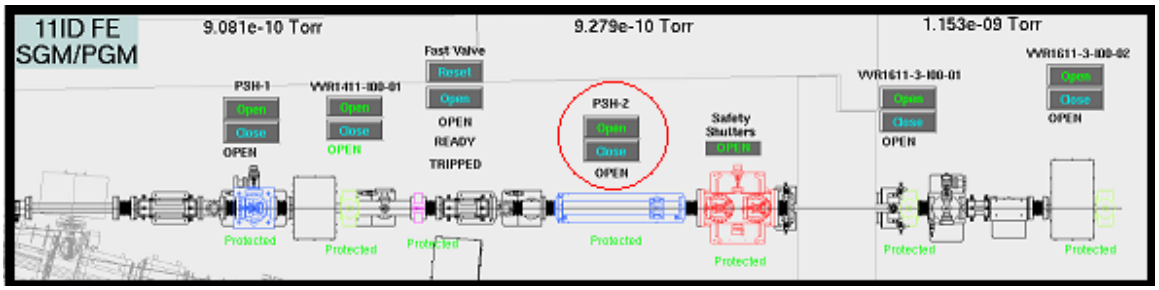
Next, check the settings in the Control Tab:
User’s data are generally saved in ‘/home/pgm/Desktop/USERS’ under your own directory.
The “Directory Path” shows where yours file will go
In the “File Name” the symbol “%d” will give you sequential file numbers for sequential runs.
Click “Start” in the Control page of the Data Acquisition when you are ready to scan.

BEAMLINE Troubleshooting

**** To be performed with the FC assistance ****

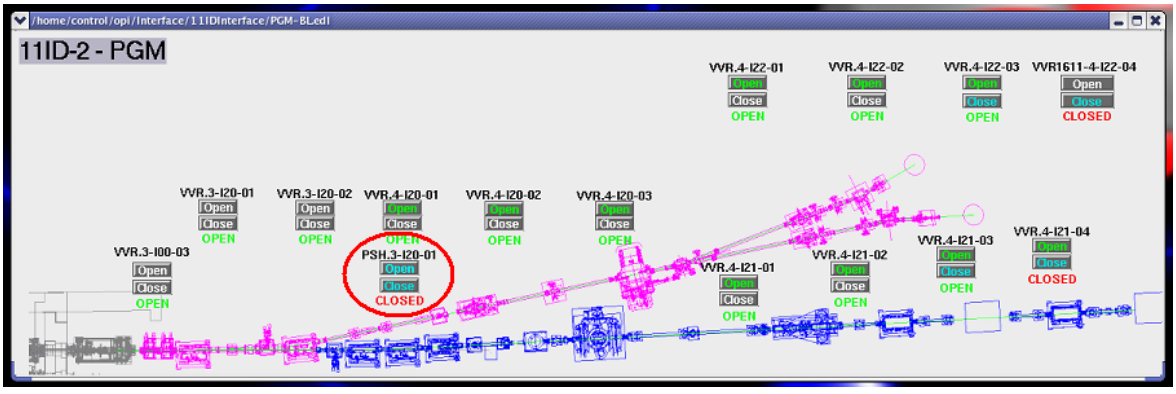
In the RARE event that you are not able to **OPEN** the Shutter two (PSH2), there are a number of things that should be check.
Start the "PGM FRONTEND" and "PGM BEAMLINE" by double clicking on the icons:

"PGM FRONTEND" panel:



Check that the beamline has been **ENABLED** by the control room and that the Safety shutter is opened by the panel outside the hutch. If closed, MANUALLY open the Safety shutters pushing the green button.
From the Computer screen **OPEN** the Shutter two (PSH-2) on the PGM frontend panel.

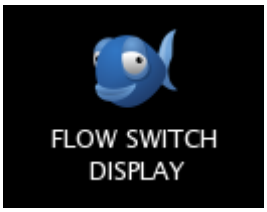
"PGM BEAMLINE" panel:



Check that all valves (VVR.*) are **OPEN** on the PGM frontend and PGM beamline panels.
If closed, **OPEN** the Shutter three (PSH.3-I20-01) on the PGM beamline panel.

NB: All the valves (VVR.*) of the active Branch have to be in the **OPEN** position, to be allowed to open the Shutter three (PSH.3-I20-01).
Keep these two panels open and running on one of the Desktops.

If all the valves (VVR.*) are **OPEN** but you still cannot **OPEN** the Shutter two (PSH2) start the SGM/PGM flow switches display by double clicking on the icon:



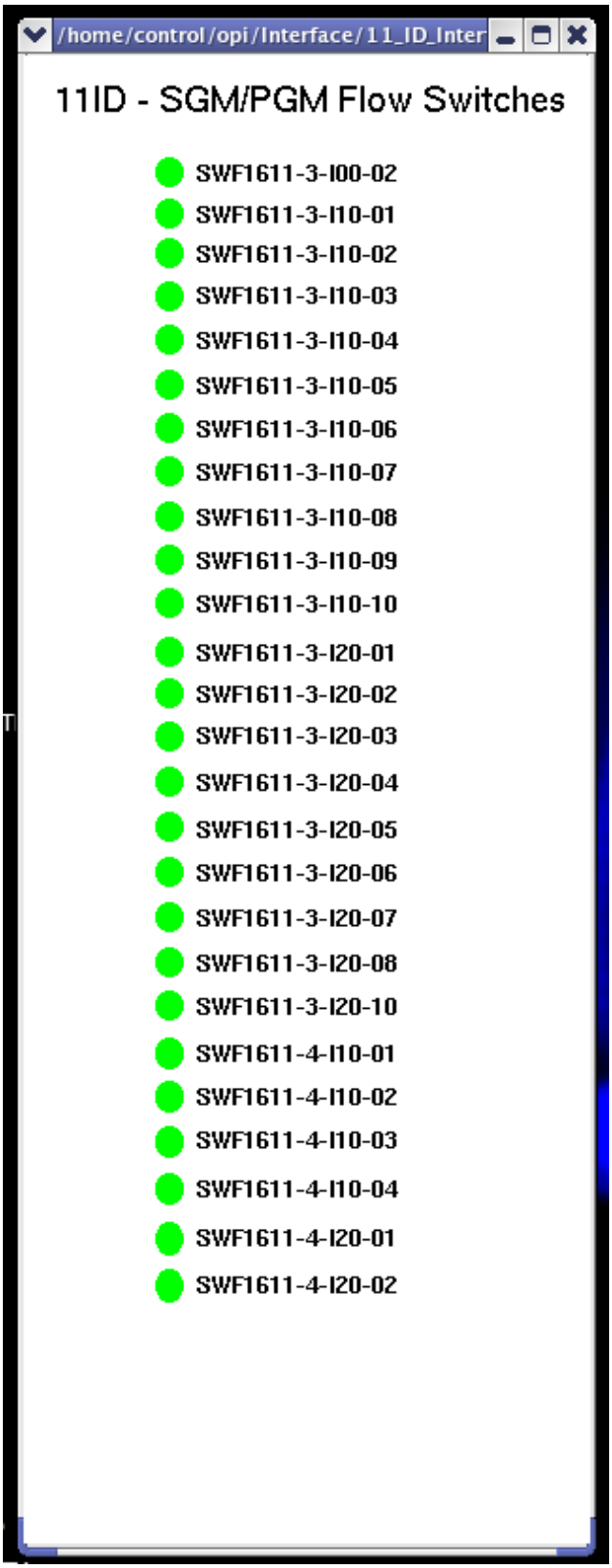
Check that ALL the Flow-Switch indicators ● are green.

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 are 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, the indicators should turn green ●, and you should be able to **OPEN** the Shutters and proceed as normal.



High Voltage Controller

**** To be performed with the FC assistance ****

If the HV controls **1** & **2** are not responding (i.e. you try to switch OFF or ON the 1450V of the FL detector as well any of the 80V, and nothing happen) and the pressure in the end-station chamber is better than 4.8×10^{-7} Torr; it means the HV control application has locked up.
The options are:

- During "office hour" contact the Beamline staff
- Otherwise, contact the Floor Coordinator (FC). The FC will call the on-call analyst from the Control and Instrumentation Development (CID) group.

Instruct the FC to ask the on-call CID to follow the instructions found in the CID Main Page http://wiki.clsi.ca/wiki/CID_Main_Page

- PGM section,
    - troubleshutting sub-section
        - "High Voltage Controller - Controls Unresponsive"

NB: Before the on-call CID leaves, double check that you can switched ON & OFF the High Voltages (try any of those set at 80V)

Multiple samples holder

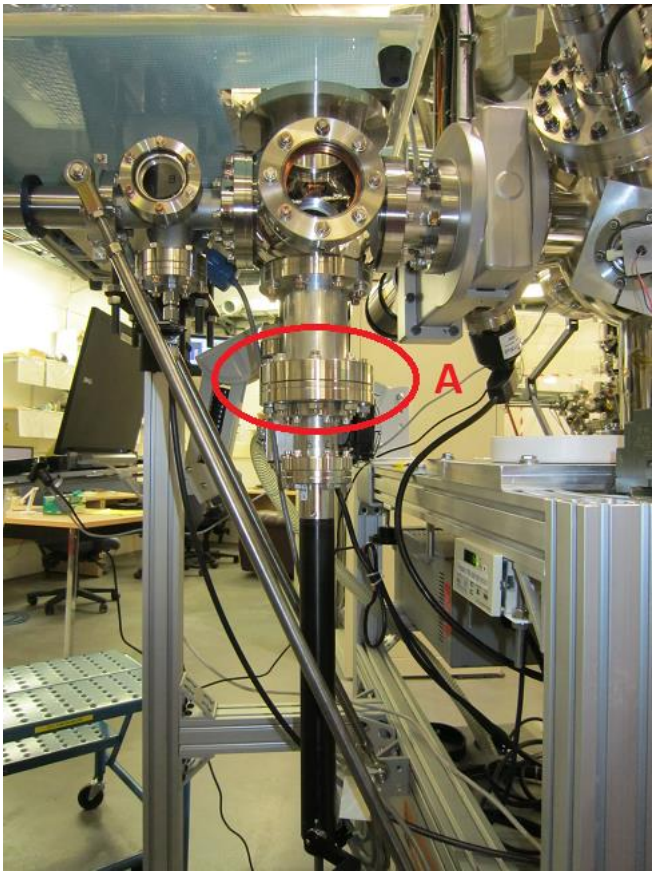
**** To be performed with the FC assistance ****

Have you dropped a sample in the loading chamber?

Is the XAS sample holding ladder jam?

In the unfortunate event the multiple samples holder is malfunctioning (usually dropped samples jamming into the bellow feed through), you will have to contact either

- 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) Bring the load-lock section up to air, as when you load a new sample
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 copper 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.
6) Replace dropped sample(s) or load new sample(s) onto the multiple sample holder.
7) Pump the load-lock section till it reaches a pressure better than 6.7 mTorr

At this point you should be able to continue your experiment.

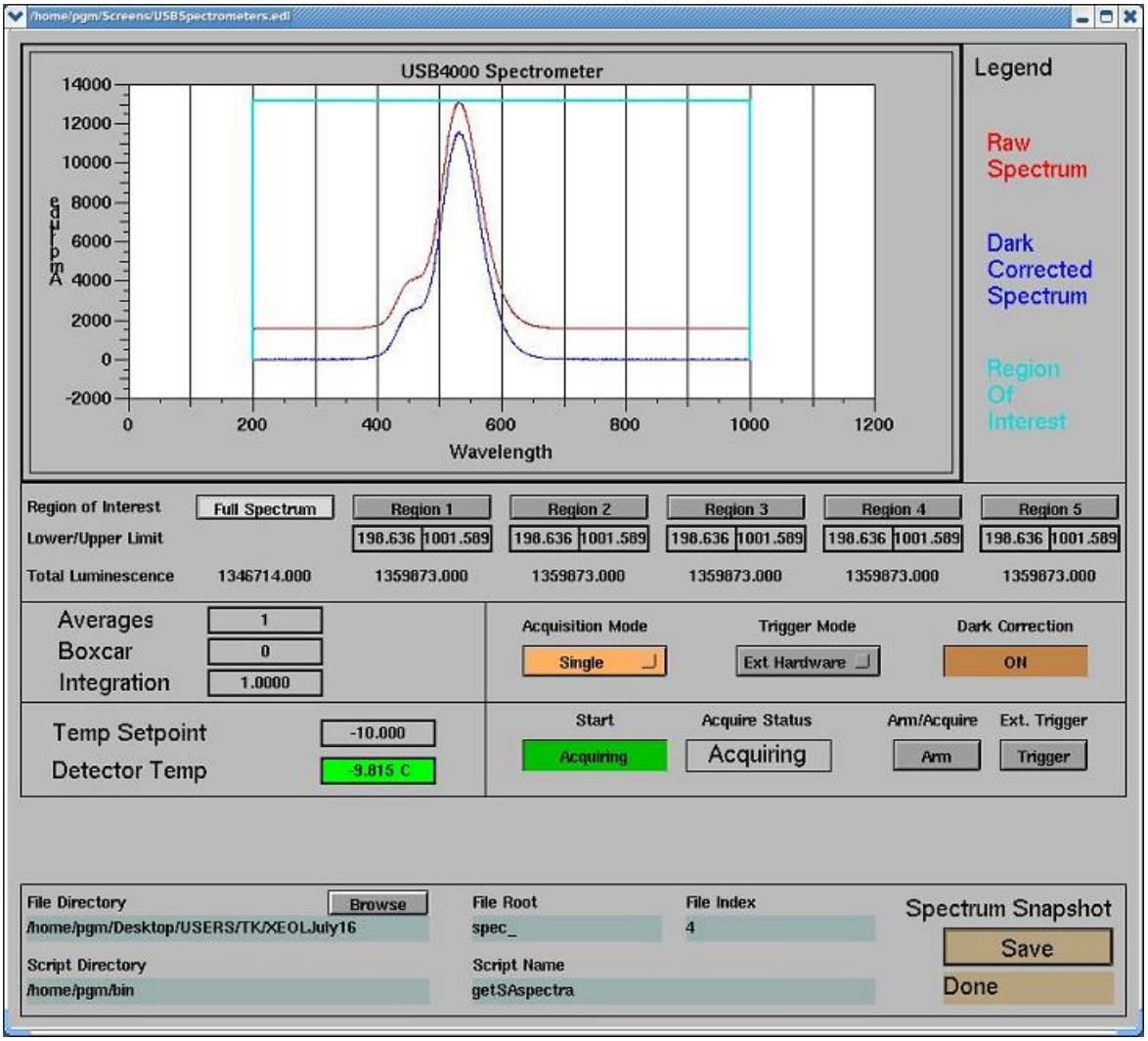
XEOL system

The XEOL spectrometer (QEPro) is connected to the laptop.
First start the spectrometer application “USBQEPro IOC” by double clicking on the icon:

Enter 1 in the terminal window asking “Which operation do you want to perform”

Two windows pop up, after 30 seconds they will disappear indicating that the startup sequence has completed.
Next start the “USBQEPro GUI” by double clicking on the icon:

The following screen will open.



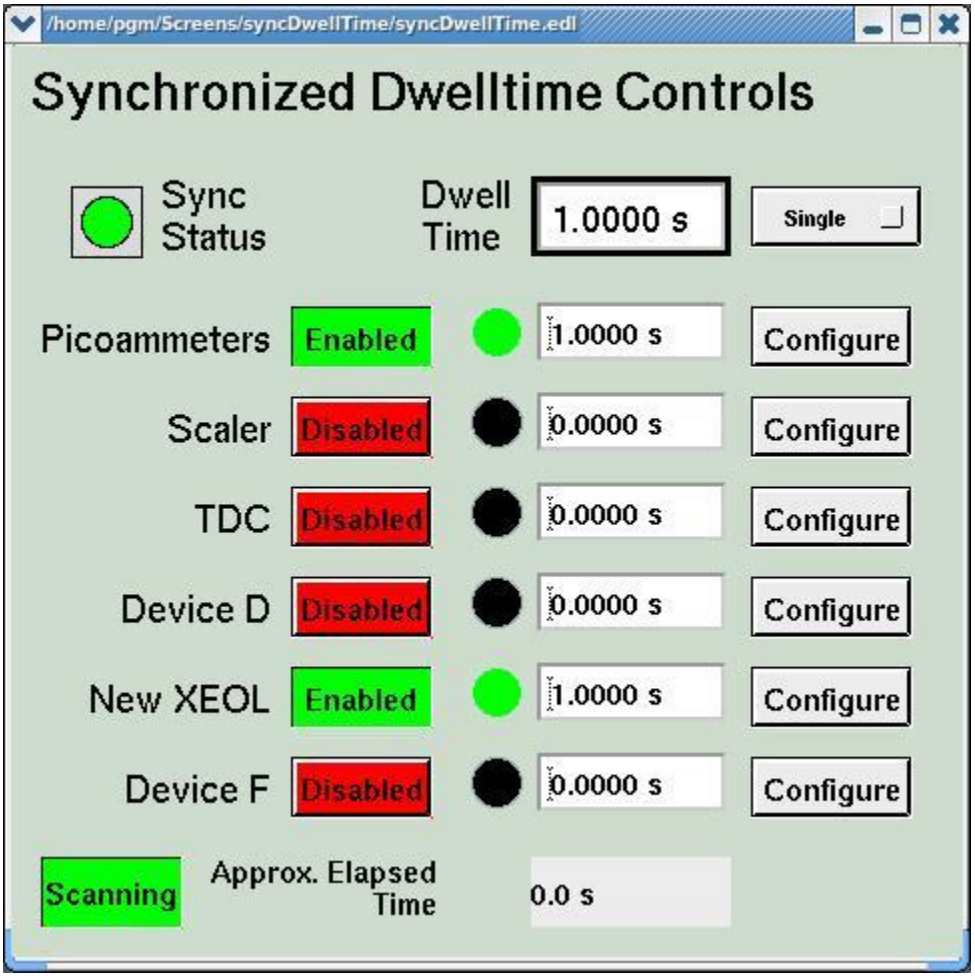
Set Trigger Mode to Ext Hardware.
Set the Integration (time) (if the control software has just been started, set this even if the integration time is what you want, just to make sure the hardware is synchronized). Do not set the Integration below 10 ms.
Typical values for **Averages** and **Boxcar** are 1 and 0, respectively.

Synchronization Software

A typical measurement involves multiple devices, and synchronization software needs to be informed that the XEOL spectrometer has been introduced.

Double-click the Beamline Dwell Time icon:

Enable the New XEOL by clicking on the “Disabled/Enabled” button.



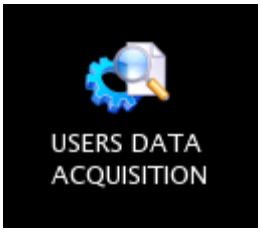
If you want to test the synchronization:

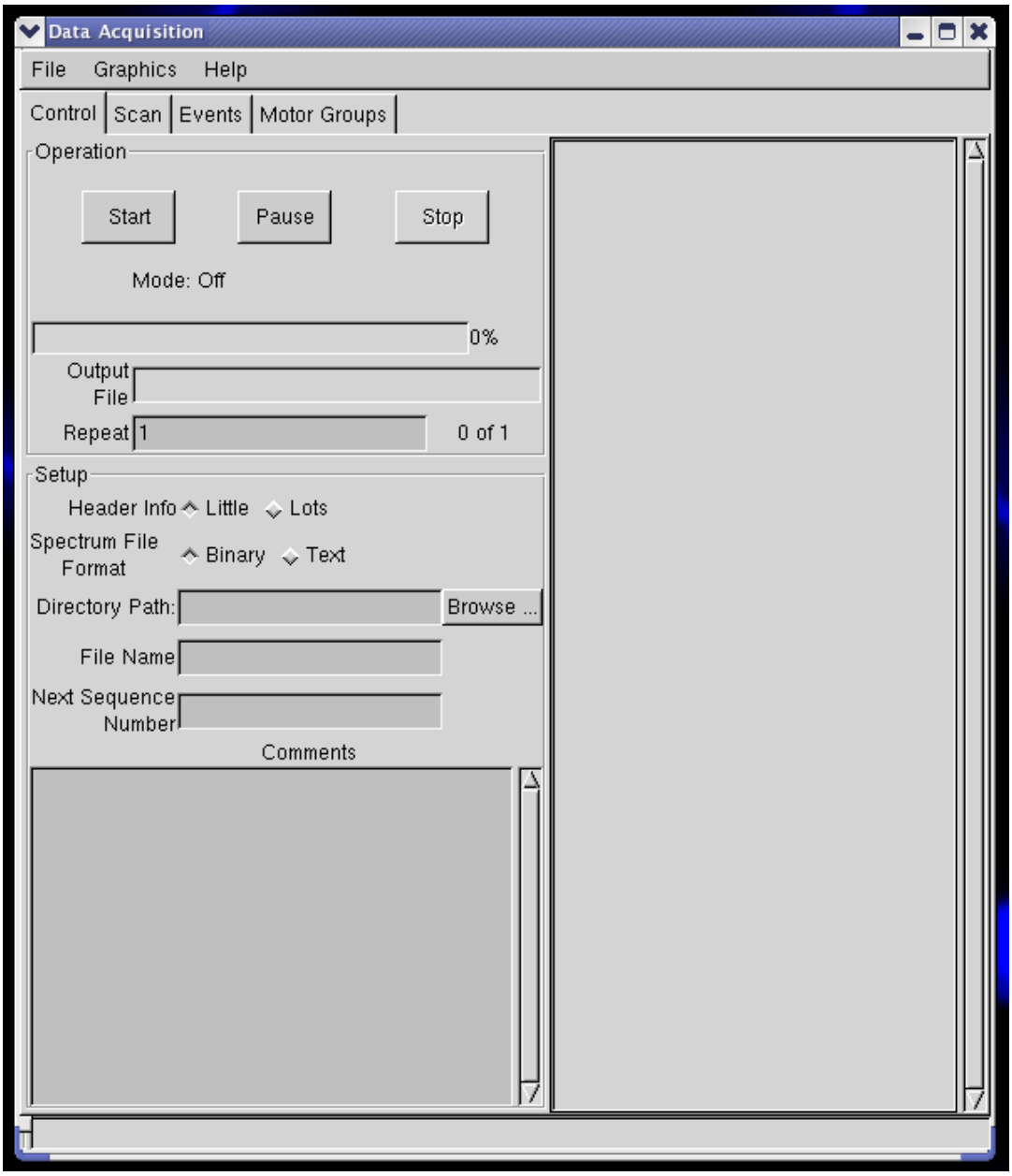
- enter a Dwell Time (near the top) and verify that the XEOL time entry matches
- set the Continuous/Single selector to "Single"
- press the Start button to make sure the spectrometer triggers (indicator should turn green at the same time as Picoammeters' indicator)

Most important

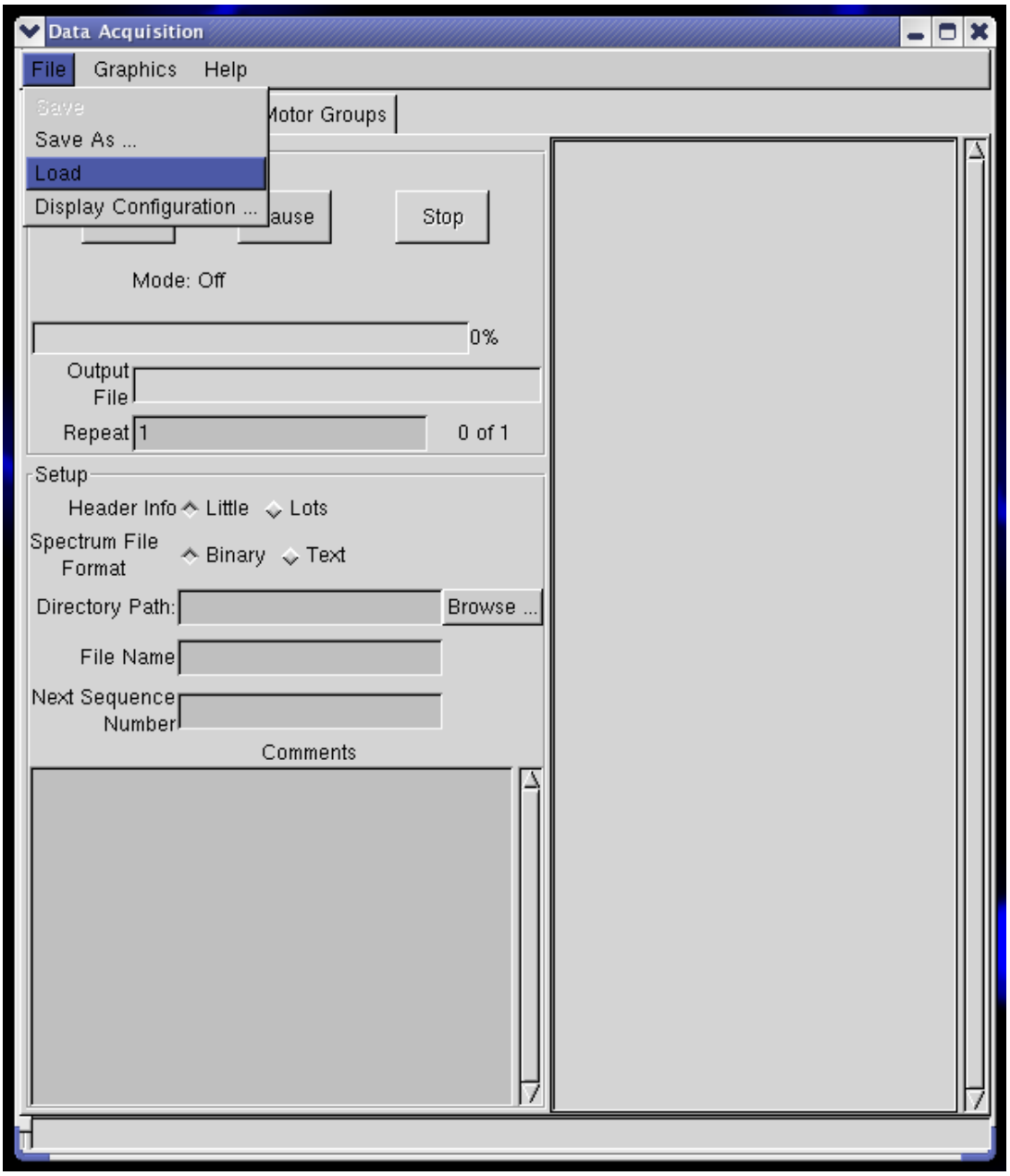
- set the Continuous/Single selector back to "Continuous"

To do energy scans with the XEOL system start the “USERS DATA ACQUISITION” by double clicking on the icon:

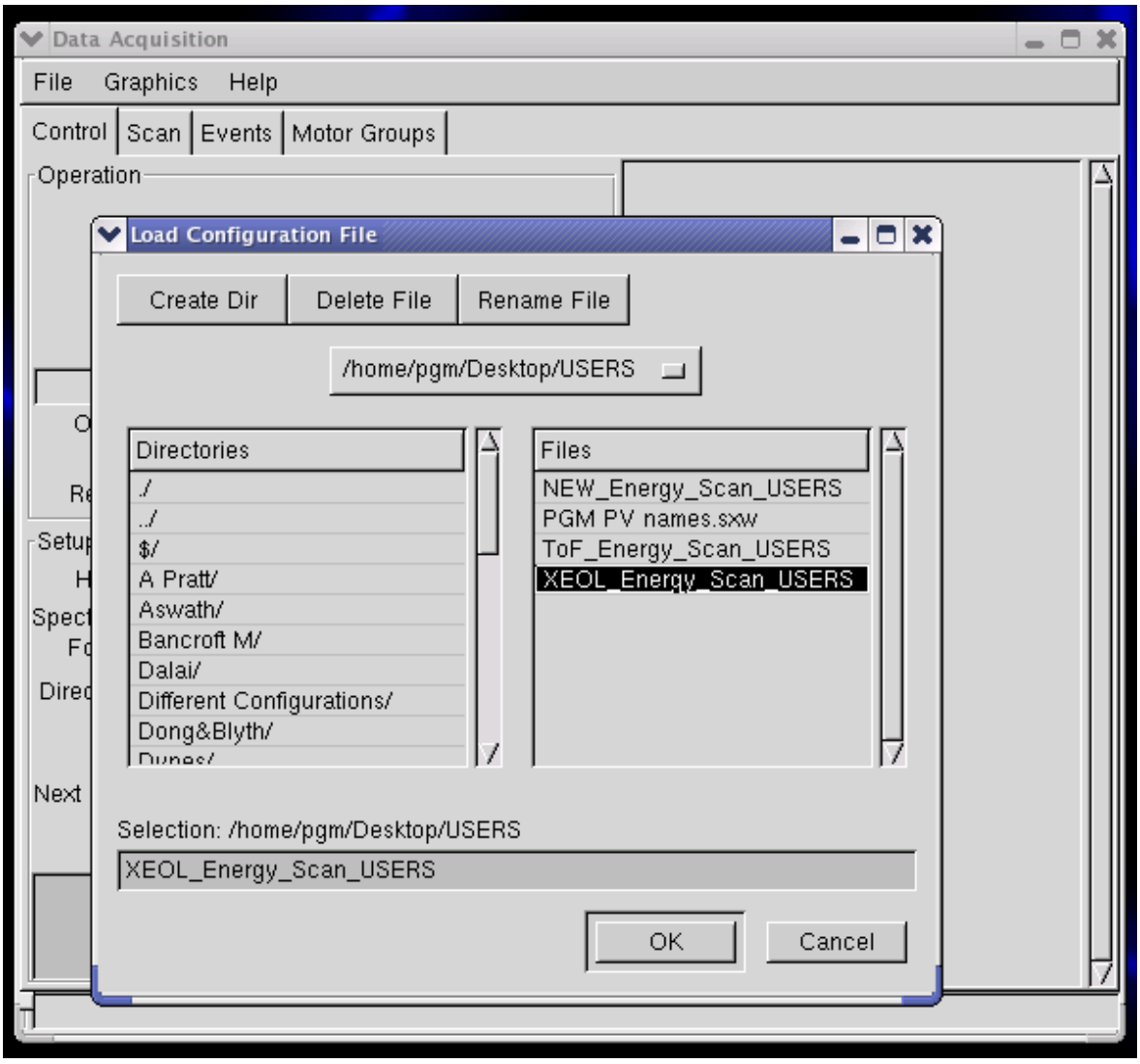




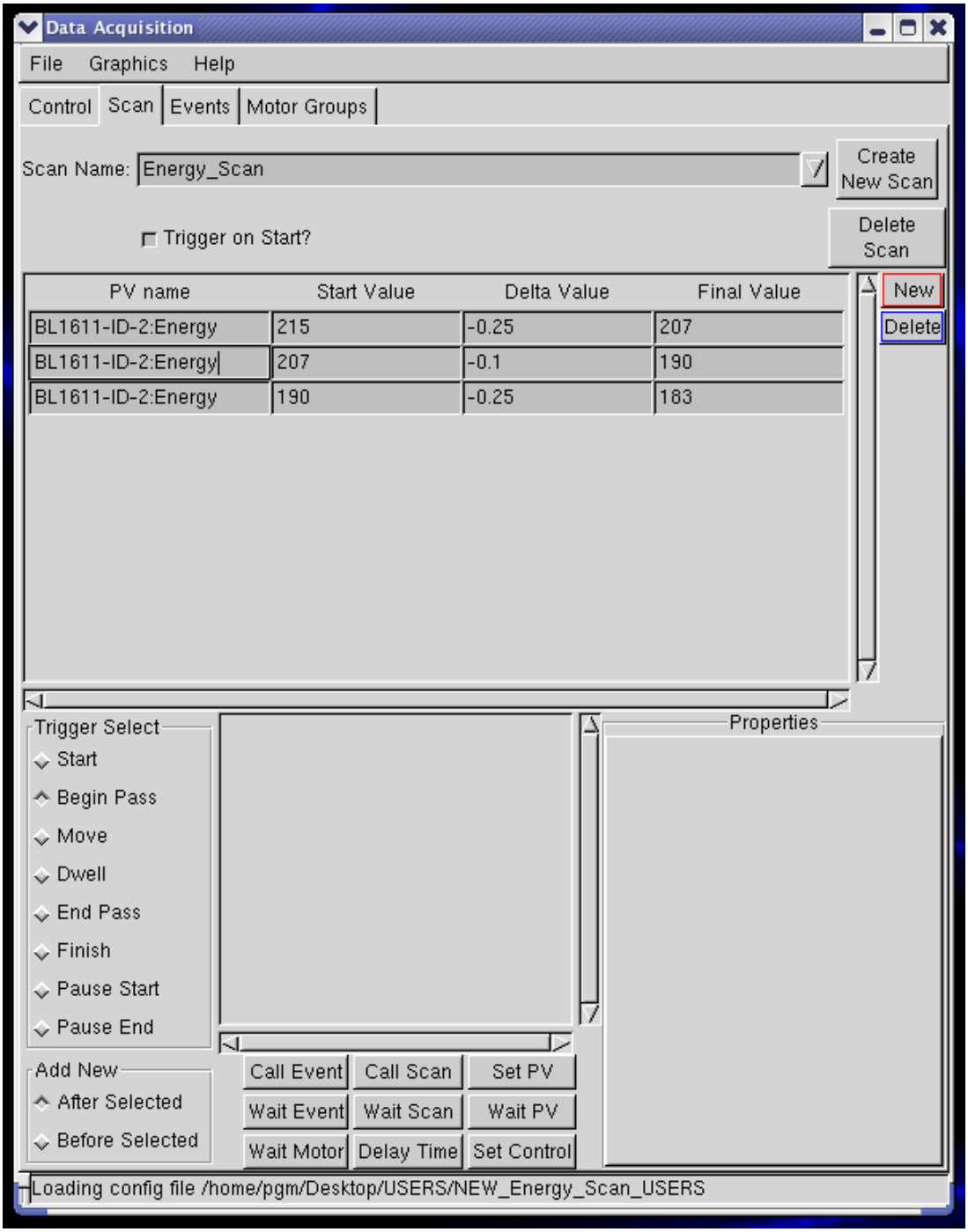
From the “File” menu Load the configuration file “XEOL_Energy_Scan_Users”



You will find this file in the ‘/home/pgm/Desktop/USERS’ directory

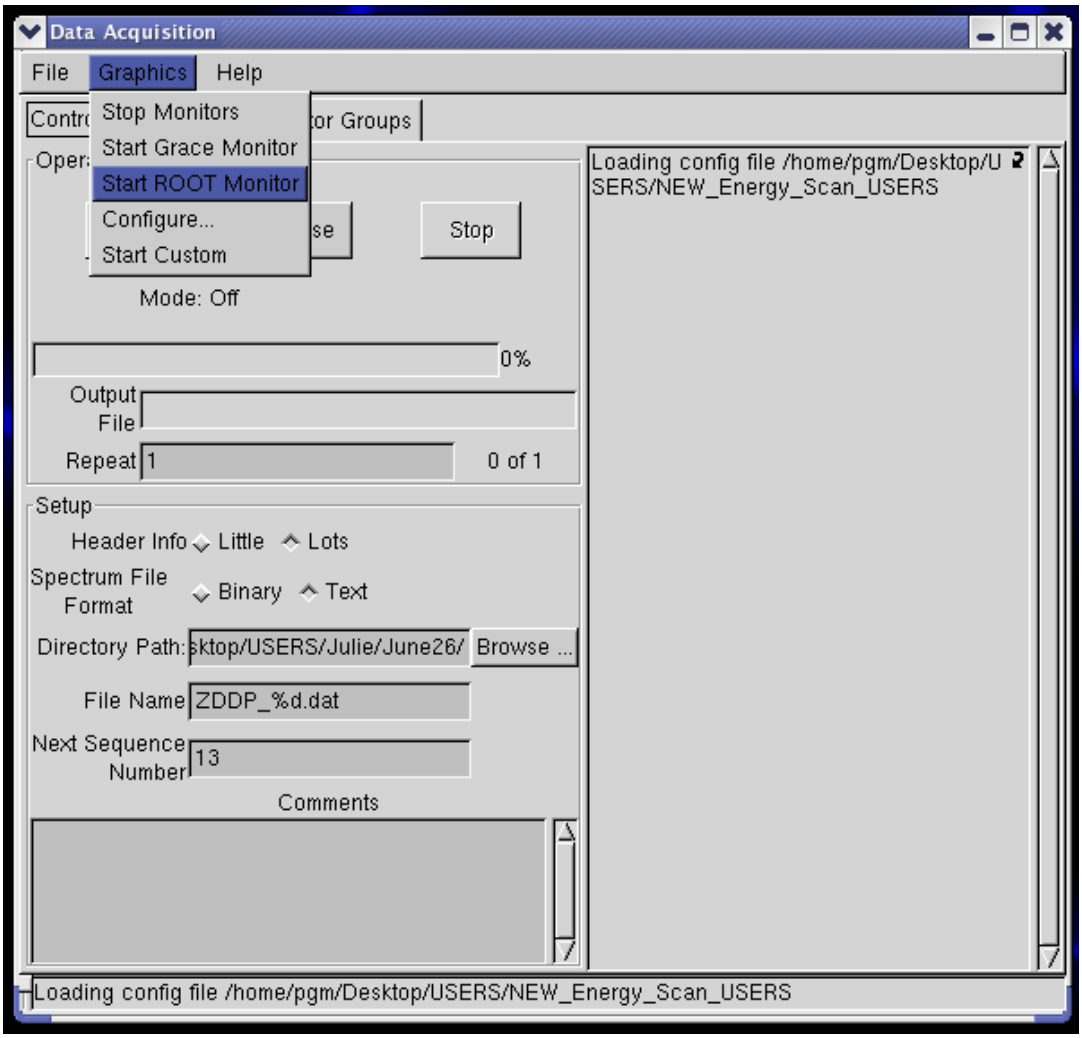


In the Scan tab make sure you are scanning over the right energetic range, from higher (Start Value) to lower (Final Value) energy, with a negative step (Delta Value).

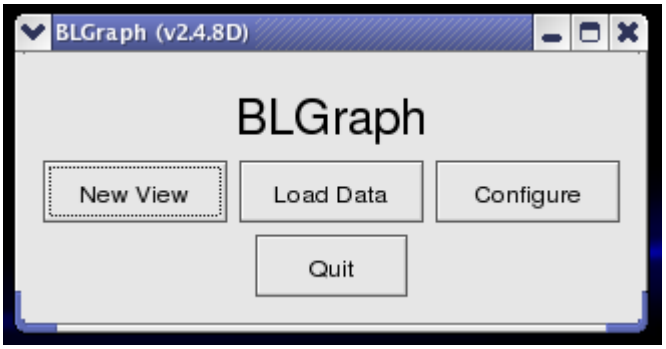


You can also scan over several consecutive regions with different steps (delta values).
To add a new region click the New button.
To delete a region click the Delete button.

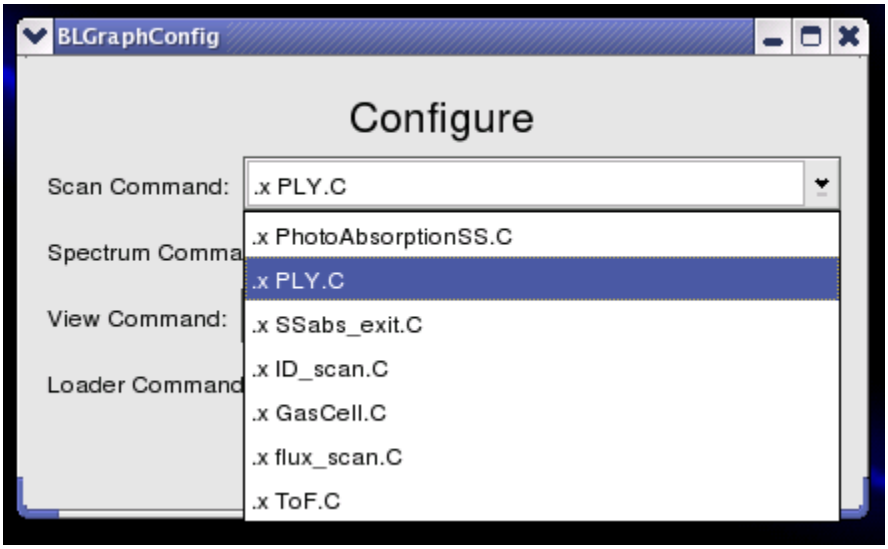
To visualize the data while acquiring, select “Start ROOT Monitor” from the “Graphics” menu



This window will pop up:



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



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

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

XEOL Troubleshooting

The laptop locked up / freeze:

Assuming the camera was connected, look at the LEDs at the top of the laptop keyboard (or below the screen). If two of them are flashing about once a second, the laptop had freeze. Your only resort is a hard shutdown, reboot, and start over:
- press the power button until the screen blanks,
- disconnect the power for the hub and disconnect the cable between the hub and the laptop (this power cycles the hub)
- disconnect all devices from the usb hub
- reboot the laptop
- reconnect the hub

do NOT reconnect anything until you are ready to start the camera software
- go through the camera startup sequence above
- plug in the other devices as needed

Note: no other sequence of actions seems to work well or at all: trying different hub ports, rebooting with camera plugged in, rebooting with camera disconnected but mouse plugged in, starting camera software at different times with respect to plugging it in, power cycling the hub,...

Integration no longer triggers:

This sometimes happens when the integration time is set very short and the mode is set to "Continuous". The software can't keep up, and it locks up.
- shut down the IOC application (exit)
- unplug the USB cable (doesn't matter which end)
- leave unplugged for 5 seconds, then plug USB cable back in
- restart the IOC application
- if that doesn't work, shut down the spectrometer in addition to the software, then restart both as if you are initially setting up.

XAS chamber: samples loading & unloading procedure

Loading:

1. With the load-lock section up-to-air, load the sample on the sample holding ladder. Lower the ladder, close the viewport door.
2. Pump down to vacuum the Load-lock chamber:
    a. SLOWLY open the manual "speedy" valve to the scroll pump monitoring at the same time the pressure in the load-lock section.
    b. Start the Turbo pump (pushing the start/stop button).
3. Wait till the Turbo pump reaches NORMAL OPERATION and the pressure in the load-lock chamber is better than 6.7 mTorr (i.e. 6.5 mTorr).
4. 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).
5. Using the transfer arm, grab the sample. Push the transfer arm into the main chamber and gently slide the sample into the holder. Use the in-vacuum screwdriver to secure the sample onto the holder.
6. Fully retract the transfer arm back into the load-lock.
   Close the manual gate valve.
7. Align the sample with the help of the two lasers.
8. If the pressure in the main chamber is reasonable (better than $5x10^{-7}$ Torr; i.e. $4.8x10^{-7}$ Torr) FROM THE PGM CONTROL PANEL switch **OPEN** the End-Station gate valve between the main chamber and the upstream of the beamline and turn ON the FL negative high voltage (-1450 V).

Unloading:

1. Make sure FROM THE PGM CONTROL PANEL (VLS-PGM Manual):

   - the negative high voltage (-1450 V) on the fluorescence (FL) detector is OFF (ramped down and off);
   - the End-Station gate valve (connecting to the upstream of the beamline) is **CLOSE**.

2. Position the manipulator on the marks (x~**10**, z~**32**, and θ~**19**°).
3. Open the manual gate valve connecting the load-lock to the main chamber.
4. Push the transfer arm into the main chamber. Lock onto your sample; use the in-vacuum screwdriver to unscrew the sample from the holder.
5. Smoothly fully retract the transfer arm back into the load-lock, CAREFUL not to open the jaws. Place the sample onto the sample holding ladder.
6. Close the manual gate valve.
7. Vent the load-lock chamber:
   a. Close the manual "speedy" valve to the scroll pump.
   b. Switch off the turbo pump of the load-lock chamber (pushing the start/stop button), bring the load-lock up-to-air (~700 Torr) using the Nitrogen gas-line.
8. Open the viewport door in the glove box. Raise the ladder and remove the sample.

XAS Process Variables

In every saved file the recorded process variables (Energy, TE yield, FL yield etc.) are listed as follow:

Step Scan:

BL1611-ID-2:Energy:fbk	☞	Beamline Energy feedback (eV)
A1611-4-11:nA:fbk	☞	EndStation Ni mesh Io current (nA)
A1611-4-09:nA:fbk	☞	TEY (nA)
A1611-4-08:nA:fbk	☞	FLY (nA)

A1611-4-10:nA:fbk	☞	Si Photodiode current (nA)
A1611-4-02:nA:fbk	☞	Branch A Exit Slit Lower Blade current (nA)
A1611-4-03:nA:fbk	☞	Branch A Exit Slit Upper Blade current (nA)
A1611-4-04:nA:fbk	☞	Branch B Exit Slit Lower Blade current (nA)
A1611-4-05:nA:fbk	☞	Branch B Exit Slit Upper Blade current (nA)
A1611-3-03:nA:fbk	☞	Entrance Slit Lower Blade current (nA)
A1611-3-04:nA:fbk	☞	Entrance Slit Upper Blade current (nA)
UND1411-02:gap:mm:fbk	☞	Undulator Gap (mm)
PCT1402-01:mA:fbk	☞	Ring current (mA)
SMTR16114I2004:enc:fbk	☞	Monochromator Encoder Feedback
BL1611-ID-2:Energy	☞	Beamline Energy

A1611-4-12:nA:fbk	☞	Beamline Ni mesh Io current (nA)

The green and blue ones are the very important ones. The others are an important source of information for the beamline staff in case anything goes wrong.

Fast (on the Fly) Scan:

Relative-Start-Time	☞	Relative Time
BL1611-ID-2:Energy:fbk:mean	☞	Mean value Beamline Energy feedback (eV)
A1611-4-11:nA:fbk	☞	EndStation Ni mesh Io current (nA)
A1611-4-09:nA:fbk	☞	TEY (nA)
A1611-4-08:nA:fbk	☞	FLY (nA)
A1611-4-10:nA:fbk	☞	Si Photodiode current (nA)
A1611-4-02:nA:fbk	☞	Branch A Exit Slit Lower Blade current (nA)
A1611-4-03:nA:fbk	☞	Branch A Exit Slit Upper Blade current (nA)
A1611-4-04:nA:fbk	☞	Branch B Exit Slit Lower Blade current (nA)
A1611-4-05:nA:fbk	☞	Branch B Exit Slit Upper Blade current (nA)
A1611-3-03:nA:fbk	☞	Entrance Slit Lower Blade current (nA)
A1611-3-04:nA:fbk	☞	Entrance Slit Upper Blade current (nA)
A1611-4-12:nA:fbk	☞	Beamline Ni mesh Io current (nA)
PCT1402-01:mA:fbk	☞	Ring current (mA)
UND1411-02:gap:mm:fbk	☞	Undulator Gap (mm)
SMTR16114I2004:enc:fbk	☞	Monochromator Encoder Feedback
BL1611-ID-2:Energy	☞	Beamline Energy
SMTR16114I2004:velo:fbk	☞	Monochromator Encoder Feedback
BL1611-ID-2:Energy:fbk	☞	Beamline Energy feedback
BL1611-ID-2:dwell:setTime	☞	Sampling dwell time

The green and blue ones are the very important ones. The others are an important source of information for the beamline staff in case anything goes wrong.