# **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:

Energy Valves		
	Beamline	
Energy: 80.00	00	~
Feedback: 79.99	96	OMoving
Eme (Al	rgency Stop LL Motors)	
Mor	nochromator	
Energy	80.0000	
Encoder Setpoint	4966999 ste	ab.
Encoder Feedback:	199416 cou	
Velocity.	5000 step/s	
Grating/M	3 Mirror Sele	ection
Medium Energy (25	0 - 125.0	Reset to This
fbk: HE	M/LE	Grating
1 2		0
1	Indulator	
Track	Enorgy 80.0	000
Gon (m	m)	
Set Point 71 443 m	n j	At Gap
Current 71 4432 w	-	- Anavina
Cutency 1.4402 II	ina i	Ownering
SI	it Position	
Exil Pos, Branch A:	145.0 mm	1bk: 145.0 T
Exit Pos, Branch B:	75.0 mm	fbk: 75.00 T
		<u> </u>
s	lit Width	
Entrance Slit	100.0 um	1bk 100.0 ur
Exit Slit, Branch A.	100.8 um	fbk. 100.0 ur
Exit Slit, Branch B	50.0 um	fbk: 50.0 um

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.

	Stora	ge Ring			
SR1Curren	t	Be	am L	ifetime:	
215.893440 mA		30.351547			
SI 10ID - DnStr	R Beam Po 11ID #	sition Monit	tors 1110	) #2	
x 118	124		-58	)	
y: -254	-453		-22	23	
	Beamline	Overview			
	Blade	Currents			
M1 0.000	e+00 A	Branch B	PD	7.974e-12 A	
M2 -1.27	9e-13 A	Branch B	3 IO	-1.545e-13 A	
Entr Lower -7.10	5e-15 A	minor k F	LY	-4.086e-14 A	
EnS Upper: 6.750	e-14 A	minor k T	ΈY	-2.593e-13 A	
M4 -1.31	5e-13 A	Branch A	PD	8.351e-12 A	
Exit A Upper 4.974	e-14 A	minor	k 10	7.816e-14 A	
Exit A Lower 7.372	e-14 A	Branch A	A 10	-3.553e-15 A	
Exit B Lower -2.45	e-13 A	Major K T	ΈY	1.545e-13 A	
Exit B Upper -8.88	2e-14 A	Major K F	LY	-1.439e-13 A	
Meters: ODone Si	ngle-Read	Continuou	s Di	well: 2.0000 s	Integration time (Dwell time)
	Ion Pump	Pressures			
FE1: 4.22e-10	Torr	Mono:	5.30	le-10	
FE2: 7.85e-10	Torr	M4:	7.00	le-10	
FE3: 7.29e-10	Torr	ExSlit A:	5.70	le-10	
M1: 8.50e-10		M5:	4.90	le-10	
M2: 9.20e-10		EXSIIT B:	5.40	le-10	
ST1: 1.10e-09		M6:	5:30	le-10	
ST2: 9.70e-10		EB1:	1.00	le-09	
EnSlit: 1.10e-09		EB2:	0.00	le+00	
Premono: 5.60e-10					
N/U: 0.00e+00		N/U:			
		08:35:24,	, Mo	n, 21 Feb 2022	

#### 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 PGM CONTROL PANEL check the following requirements:

- 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 **CLOSED**.

From the Endstation check that:

• The MANUAL gate valve between the main chamber and the load-lock chamber is **TIGHTLY CLOSED**.

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

- i. Close the manual "speedy" valve to the scroll pump.
- ii. Switch off the turbo pump of the load-lock chamber (pushing the start/stop button)
- iii. Slowly bring the load-lock up-to-air (~700 Torr) using the Nitrogen gas-line. Check the pressure in the main valve; it will raise however it should never go higher then 2x10<sup>-6</sup> Torr (i.e. 5x10<sup>-6</sup> Torr).
- iv. 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:

- i. SLOWLY open the manual "speedy" valve to the scroll pump monitoring at the same time the pressure in the load-lock section.
- ii. Start the Turbo pump (pushing the start/stop button).
- iii. Wait few minutes until the Turbo pump reaches NORMAL and the pressure in the load-lock is better than 6.7 mTorr (i.e. 6.5 mTorr).

Open the manual gate valve between the load-lock and the main chamber; the pressure in the main chamber should stay better than  $2 \times 10^{-6}$  Torr (i.e.  $1.8 \times 10^{-6}$  Torr).

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.

Align the sample with the help of the camera cross.

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).

#### Sample un-loading procedure

From the PGM CONTROL PANEL check the following requirements:

- 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 **CLOSED**.

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)

				_
Shutters: PSH1:	PSH2:	SSH1:	PGM PSH3	E>
OPEN	CLOSED	OPEN	CLOSED	E
	Closed 📝	Í	Closed	/-
Endstation Ga	te Valves			
Branch A		Branch B		
Active Branc	hline:  <mark>B</mark>	Branch B	motor: -34766	
Active Branc Shutters: PSH1:	hline: <mark>B /</mark> PSH2:	Branch B SSH1:	motor: -34766 PGM PSH3:	
Active Branc Shutters: PSH1: OPEN	hline: B	Branch B SSH1: OPEN	motor: -34766 PGM PSHJ. CLOSED	)
Active Branc Shutters: PSH1: OPEN	hline: B PSH2: OPEN Opened	Branch B SSH1: OPEN	motor: -34766 PGM PSH3: CLOSED Closed	D
Active Branc Shutters: PSH1: OPEN Endstation G	PSH2: OPEN Opened	Branch B SSH1: OPEN	motor: -34766 PGM PSH5 CLOSED Closed	D

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)

Active Bran	chline: <mark>B </mark> B	Branch B	motor: -34766	_
Shutters:				
PSH1:	PSH2:	SSH1:	PGM PSH3:	E
OPEN	OPEN	OPEN	OPEN	Y
	Opened 7		Opened 📝	Γ
Endstation Gate Valves				
Branch A		Branch B		

- Сι Active Branchline: B Branch B motor: -34766 Shutters: PGM PSH3: Exit PSHI: PSH2: SSH1: OPEN OPEN OPEN. OPEN. Exit Opened Opened Endstation Gate Valves Branch A Branch B OPEN Exit losed Opened Exit If you Cancel a selection, you should re-select the currently active one to re-activate the selector
- 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. IO, 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 xaxis 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**.

Data Acquisition <	5> <u>vo</u> x	
File Graphics Help		
Save fator Groups		
Save As	2	
Load		
Display Configuration ause Stop		
Mode: Off		
Mous. On		
0%		
File		Load Configuration File 🛛 😸 🔗 ⊗
Repeat 1 0 of 1		
Setup		Create Dir Delete File Rename File
Header Info 🛧 Little 🖕 Lots		/homo/hom/Dackton/0.11SERS
Spectrum File A Binary CText		Moneygin Desizopio 032KS
Directory Path: Browse		Directories
File Name		/ SDD_Energy_Scan_TopupVeb
Next Sequence		0 FAST scan configurations/ XEOL_Energy_Scan_TopupVet
Comments		A_Tristan special config/
		Addison/
		Akio/
		Antonio/
		Aswath/
		Ranarica/ I/ N /
		Colorian Assessment Devidence 10/105200
	1	Selection, monepginoeskopo useks
		lono_trueida_ocau_inhidhAeinen_noewo
H	Ļ	OK Cancel

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.

✓ Data Acquisition			,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	×
File Graphics Help				
Control Scan Events	Motor Groups			
				Create 1
Scan Name: Energy_Sca	an		$\overline{\Delta}$	New Scan
				Delete
F Trigger or	n Start?			Scan
PV name	Start Value	Delta Value	Final Value	ANew
BL1611-ID-2:Energy	215	-0.25	207	Delete
BL1611-ID-2:Energy	207	-0.1	190	
BL1611-ID-2:Energy	190	-0.25	183	
			r	
Trigger Select			Properties	
♦ Start				
A Begin Pass     ■				
⇔ Move				
↓ Dwell				
End Pass				
↓ Einich				
↓ Fimish				
Pause Start		H		
♦ Pause End				
Add New	Call Event Call Scan	Set PV		
	Wait Event   Wait Scan	Wait PV		
♦ Before Selected	Wait Motor Delay Time	e Set Control		
Loading config file /hom	e/pam/Desktop/USERS/	NEW Energy Scan	USERS	
J- taking tering ine thom				

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

✓ Data Acquisition	_ <b>D</b> ×
File Graphics Help	
Contro Stop Monitors for Groups	Loading config file /home/pgm/Desktop/U 2
Start ROOT Monitor	SERS/NEW_Energy_Scan_USERS
Configure se Stop	
Mode: Off	
0%	
Output File	
Repeat 1 0 of 1	
Setup	
Header Info 🕹 Little 🐟 Lots	
Spectrum File Format 💠 Binary 🐟 Text	
Directory Path: sktop/USERS/Julie/June26/ Browse	
File Name ZDDP_%d.dat	
Next Sequence Number 13	
Comments	
Loading config file /home/pgm/Desktop/USERS/NEW_I	Energy_Scan_USERS

♥ BLGraph (v2.4.8D	)	- • ×
l	BLGraph	
New View	Load Data	Configure
	Quit	

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.

Data	Acquisition 💿 💿 🛞
File Graphics Help	
Control Scan Events Motor Groups	
Operation Start Pause Stop	Loading config file /home/pgm/Desktop/USERS/0 FAST 2
Mode: Off	Output Directory 📀 🔿 🛞
0%	Create Dir Delete File Rename File
Output File	/home/pgm/Desktop/USERS
Repeat     1     0 of 1       Setup     Header Info ↓ Little ▲ Lots       Spectrum File     ♦ Binary ▲ Text       Directory Path:     USERS/Your name in here/       Browse        File Name     test_00%d.dat       Next Sequence     1       Comments     Comments	Directories       A         Variansi/       XAS_Energy_Scan_USERS         Vigy/       XAS_Energy_Scan_USERS         Vogel/       XEOL_Energy_Scan_USERS         Zuin_10305/       Zuin_10712/         deBoer/       J         xAimee/       J         xDifferent Configurations/       J
A coding config file /home/ngm/Deckton/USEBS/0_EAS	Selection: /home/pgm/Desktop/USERS

- 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

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_AI_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

The following are the available pre-set Fast Scan configurations

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.

	Data Acquisition <5>	$\odot$ $\odot$ $\otimes$
File Graphics Help		
Bave Notor Grou	adi	
Save As		
Load		
Display Configuration ause	Stop	
Mode: Off		
	0%	
Output	0.10	
File		
Repeat 1	D of 1	
Setup		
Header Info 🛧 Little 🖕 Lots		
Spectrum File A Pinary A Taxt		
Format Format		
Directory Path:	Browse	
File Name		
Next Sequence		
Number		
Comments		
	H	H
4	,	

### element and edge of interest

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

▶ Data Acquisition	
File Graphics Help	
Control         Stop Monitors         for Groups           Oper:         Start Grace Monitor            Start ROOT Monitor             Configure         se         Stop	Loading config file /home/pgm/Desktop/U ₽ △ SERS/NEW_Energy_Scan_USERS
Mode: Off	
0%	
Output File	
Repeat 1 0 of 1	
Setup Header Info 🔷 Little 🐟 Lots	
Spectrum File Format 💠 Binary 🐟 Text	
Directory Path:sktop/USERS/Julie/June26/ Browse	
File Name ZDDP_%d.dat	
Next Sequence 13	
Comments	
Loading config file /home/pgm/Desktop/USERS/NEW_E	Energy_Scan_USERS

✓ BLGraph (v2.4.8D)	The BLGraph GUI opens.
BLGraph	Click Configure.
New View Load Data Configure	
Quit	Never "Quit" this window

♥ BLGraphConfig	- 0	×
	Configure	
Scan Command:	.x PLY.C	
Spectrum Comma	.x PhotoAbsorptionSS.C	]
	.x PLY.C	
View Command:	.x SSabs_exit.C	
Loader Command	.x ID_scan.C	
Loudor oonmand	.x GasCell.C	
	.x flux_scan.C	
	.x ToF.C	_

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.

Data	Acquisition 📀 🔗 🛞
File Graphics Help	
Control Scan Events Motor Groups	
Operation	Loading config file /home/pgm/Desktop/USERS/0 FAST 2
Start Pause Stop	scan configurations/Fast_XAS_LI_Kedge
Mode: Off	Output Directory 📀 📀 😣
0%	Create Dir Delete File Rename File
Output	/home/pgm/Desktop/USERS
Repeat j1 U of 1	
Setup Header Info A Little A Lots	Var voge XAS_Energy_Scan_USERS
Spectrum File	Vijay/ Vijay/ Vogel/
Directory Path: USERS/Your name in here/ Browse	Your name in here/
File Name test_00%d.dat	Zuin_10712/
Next Sequence	Aimee/
Number <sup>j</sup> Comments	xDifferent Configurations/
	Selection: /home/pgm/Desktop/USERS
	OK
J H	]/
Ucading config file /home/pgm/Desktop/USERS/0 FAS	Fscan configurations/Fast_XAS_Li_Kedge

- 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 together with IO, TEY and FLY during an XAS scan involves multiple devices to 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.

Beamline Staff is responsible for 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 Beamline Dwell Time:



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

Simple XAS measurements have only the Picoammeters Enabled, responsible for collecting the TEY and FLY signals.

Check that the New XEOL, or SDD, 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)
- o set the Continuous/Single selector back to "Continuous"

# SDD instructions

#### Switch ON the system from the Detector Read-Out "The BOX"

Hold the button for 6sec. You can probably hear two very soft beeps. If not, due to background noise (e.g. pumps), hold and count 1001-1002-1003-1004-1005-1006. That will give enough time to the system to load the required parameters.

#### Start the detector control system

In the SDD *Endstation name*\* folder:

- open the SDD IOC

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

- open the SDD GUI.

- check the temperature reading, wait until it is ~225K and the box-line goes from RED to Yellow to clear.

Real time preset  $\neq 0$  (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.

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

### Collect a 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. Take a MCA and verify that:
  - a. the Average % dead is below 5% for any peaking time value, not just for the default
     4μs value
  - b. the *Rate* in the *Energy channel* do not exceed 8,000/sec (assuming you did not change the default peaking time of  $4\mu$ s)

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  $25\mu m$
- 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.)

## Setting up for XAS

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**.

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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.

File Graphics Help         Control Scan Events Motor Groups         Scan Name: Energy_Scan         If Trigger on Start?         Delete         Scan Name: Energy 215         PV name         Start Value         Delta Value         Final Value         BL1611-ID-2:Energy         215         -0.25         207         BL1611-ID-2:Energy         207         -0.1         190         BL1611-ID-2:Energy         190         -0.25         183	💙 Data Acquisition				- • ×
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To visualize the data while acquiring, select "Start ROOT Monitor" from the "Graphics" menu.

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Click on Configure, select SDD\_Endstation name.C, 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 150 $\mu$ m x 150 $\mu$ m, close the slits to 125 $\mu$ m x 125 $\mu$ m before the next scan).

### Switch off procedure

Every time a sample is changed, and at the end of the measuring session, this procedure has to be followed:

- close the PSH3 and Endstation Gate Valve from the BL User Interface

- open the SDD IOC
  - type 2, a second window will open and after few minutes will/should close.

- switched the system OFF from the detector read-out "The BOX"

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

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**.

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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.

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

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

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	.x GasCell.C	
	.x flux_scan.C	
	.x ToF.C	

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 ✔ /home/control/opi/Interface/11\_ID\_Inter 🕳 🚍 🗙 clicking on the icon: 11ID - SGM/PGM Flow Switches 😑 SWF1611-3-100-02 SWF1611-3-I10-01 SWF1611-3-I10-02 FLOW SWITCH SWF1611-3-I10-03 DISPLAY SWF1611-3-I10-04 SWF1611-3-I10-05 SWF1611-3-I10-06 Check that ALL the Flow-Switch indicators are SWF1611-3-I10-07 green. SWF1611-3-I10-08 SWF1611-3-I10-09 The indicators beginning with SWF1611-3-\* are water SWF1611-3-I10-10 cooling indicators inside the SGM/PGM hutch. SWF1611-3-I20-01 SWF1611-3-I20-02 The SWF1611-4-\* are water cooling indicators outside SWF1611-3-I20-03 the hutch. SWF1611-3-I20-04 SWF1611-3-I20-05 If any of the indicators is e red you will have to SWF1611-3-I20-06 contact either SWF1611-3-I20-07 SWF1611-3-I20-08 the beamline staff or SWF1611-3-I20-10 the Floor Coordinator (FC). • SWF1611-4-110-01 SWF1611-4-I10-02 The FC will contact the on-call mech-tech. SWF1611-4-I10-03 The on-call mech-tech will re-equilibrate the water SWF1611-4-110-04 flow along the beamlines. SWF1611-4-I20-01 SWF1611-4-I20-02 Once done, all the indicators are green e 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?

Is the XAS sample holding a ladder jam?

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

- the beamline staff, or
- during unsociable hours the Floor Coordinator (FC) and ask the FC to phone the oncall 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 loading 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 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.

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 until it reaches a pressure better than 6.7 mTorr.

# 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