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# **16-BM-B Docs**

***Release 0.1***

**R. Hrubia**

**Jun 10, 2022**

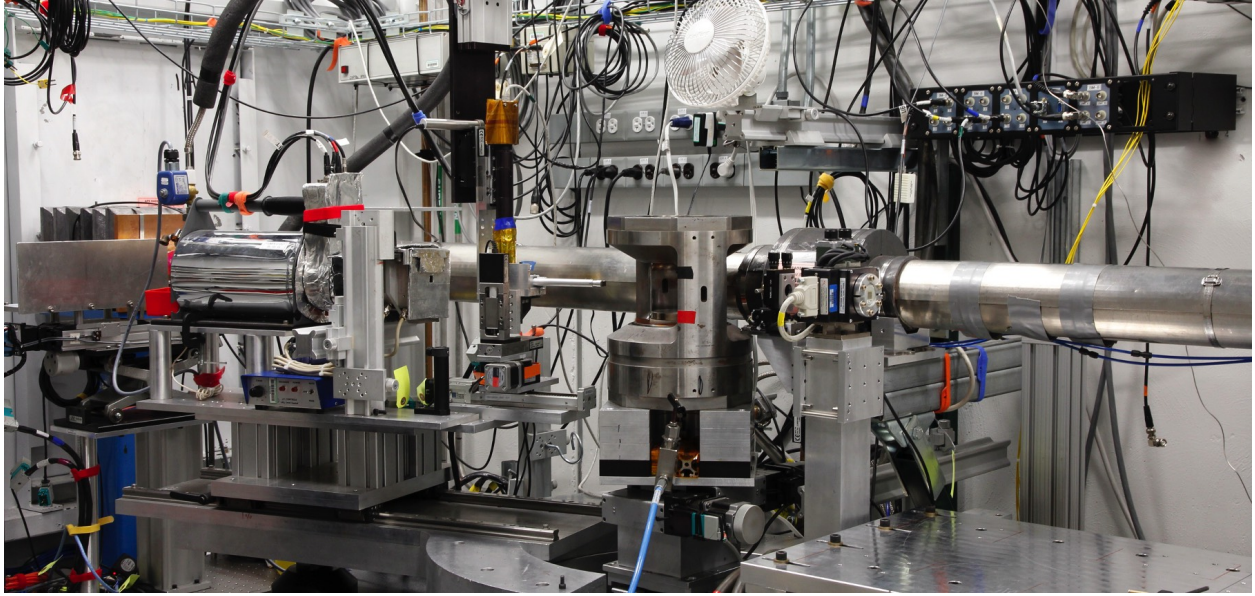


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## BEAMLINE DESCRIPTION

The HPCAT 16-BM-B beamline is dedicated to white beam x-ray diffraction and radiography research of materials under high pressure.

With the APS bending magnet white x-rays (5-120 keV), multi-angle energy dispersive x-ray diffraction (EDXD) technique is extensively utilized to obtain the structure factors up to  $Q \approx 20 \text{ \AA}^{-1}$ . A Paris-Edinburgh Cell (PEC) (250 ton capability) is equipped for samples requiring a large scattering volume, especially, for the high-temperature melt and amorphous structure. The range of pressure and temperature is up to 7 GPa and 2300K. The PEC setup includes white beam radiography system, which allows the structure measurement with EDXD to be combined with radiographic volumetry, ultrasonic sound velocity measurement and/or falling sphere viscosity measurement.

### 1.1 Summary of 16-BM-B features

Table 1: 16-BM-B features

Feature	Description
Source	bending magnet
Monochromator	N/A, white beam
Energy range	10-120 keV for parallel beam; 10-65 keV for focused beam
Beam size & focusing optics	50 m x 50 m to 2 mm x 2 mm for parallel beam (slit-limited) 10 micron (H) x 10 micron (V) FWHM with 200 mm Pt-coated Si KB- type mirror
Established techniques	Multi-Angle Energy Dispersive X-ray Diffraction, White Beam Radiography, Ultrasonic elastic wave velocity measurement, Falling sphere liquid viscosity measurement
Detectors	Ge Solid State Detector, CCD camera (Prosilica GC1380H), High-speed camera (Photron FASTCAM SA3)
Support equipment	Paris-Edinburgh type large volume press with resistive heating capacity (PE anvils, boron-epoxy gaskets, cylindrical graphite heater, 8V-220A power supplier; temperature and pressure range up to 2500 K under and 7 GPa)



## PEC SAMPLE ASSEMBILES

The HPCAT 16-BM-B beamline hosts a Paris-Edinburgh (PE) press that allows standardized sample assemblies to be pressurized and heated while conducting *in-situ* x-ray characterization. This section describes different types of PE experiments and optimized sample assemblies.

### 2.1 Types of cells

#### 2.1.1 Standard cell

Standard PEC

##### Cell materials

Cell materials

##### Cell assembly instructions

Cell assembly instructions

#### 2.1.2 CDT cell

CDT PEC

##### Cell materials

Cell materials

## **Cell assembly instructions**

Cell assembly instructions

### **2.1.3 Ultrasound cell**

Ultrasound PEC

## **Cell materials**

Cell materials

## **Cell assembly instructions**

Cell assembly instructions

### **2.1.4 Viscosity cell**

Viscosity PEC

## **Cell materials**

Cell materials

## **Cell assembly instructions**

Cell assembly instructions

### **2.1.5 Double-stage cell**

Double-stage PEC

## **Cell materials**

Cell materials

## **Cell assembly instructions**

Cell assembly instructions

### **2.1.6 Grooved cell**

Grooved PEC

#### **Cell materials**

Cell materials

#### **Cell assembly instructions**

Cell assembly instructions

## **2.2 Capsule materials**

Capsule materials





## BEAMLINE OPERATION

This section contains beamline operation instructions.

### 3.1 Login

This login id and password will be required when re-logging in the computers (e.g., after rebooting the operating system), and can be provided by the beamline staff.

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**Note:** The wireless mouse of the control PC has a battery saving feature and will turn off after prolonged idle period. The mouse has to be switched back on by pressing the power button on the bottom.

---

### 3.2 Start PEC EPICS/MEDM user interface

On the Windows desktop of the control computer, users will find a shortcut named 16bmb MEDM PEC.

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**Note:** MobaXterm program should be started or running in the background before opening the MEDM interface, otherwise the interface will not start.

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**Note:** If running in isolation mode, the EPICS/MEDM interface should be launched from MobaXterm. In the MobaXterm go to the tab duesenberg(s16bmbuser). Click Macros (located in either the top Menu bar or left-side tabs) and select Start MEDM for 16bmb PEC users.

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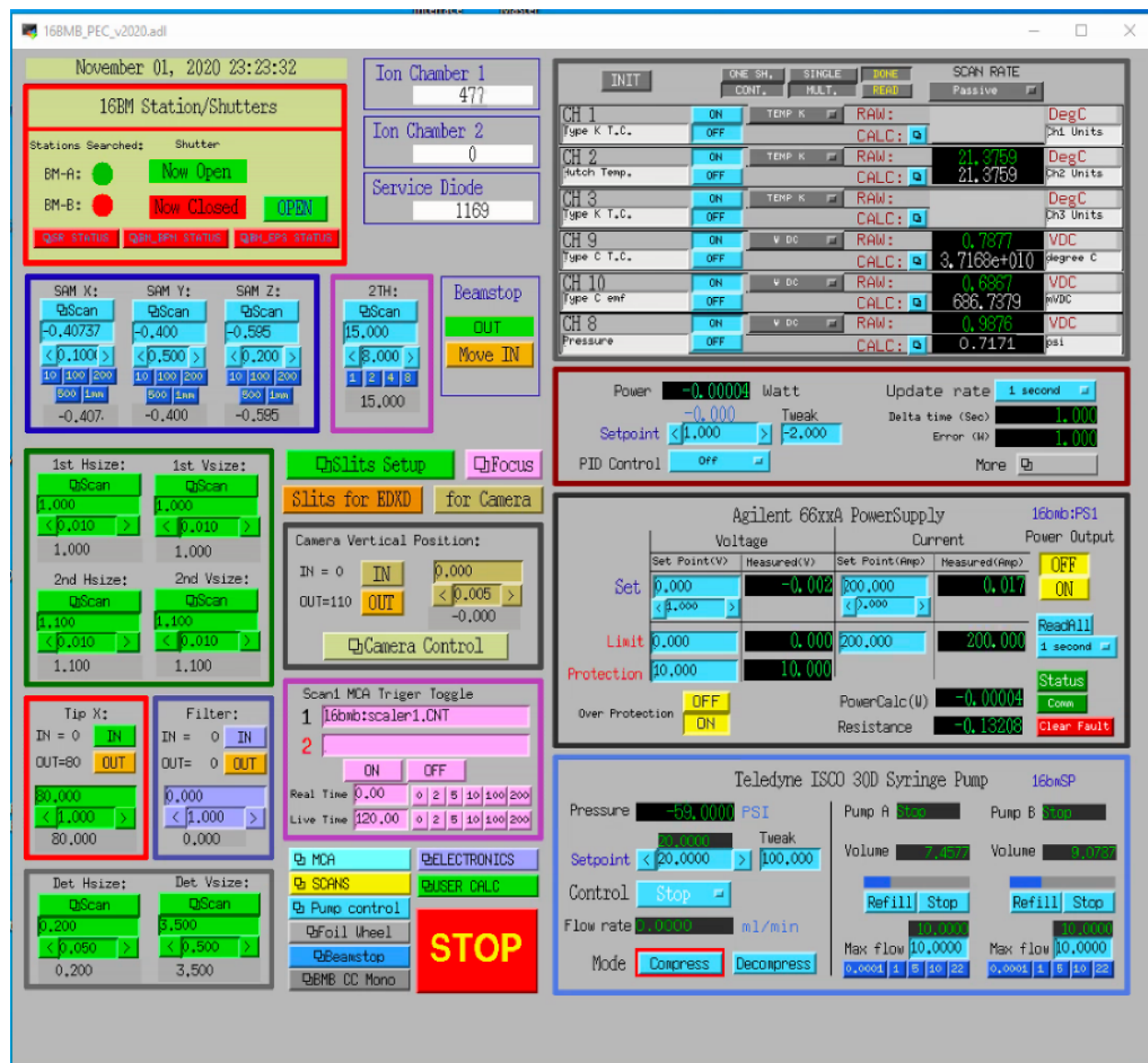
Attached below is a screen shot of the PEC User Interface.

It interfaces the control widgets with 16-BM-B station shutters, SR status, EPS status, most demanded motors for user experiments, intensity monitors with ion chambers and diode, and heater power supply controller and pressure control syringe pump.

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**Hint:** Please be aware that when entering a new value into a field in an MEDM interface, the mouse cursor must hover over the field. Then the Enter key on the keyboard needs to be pressed to accept the edited value. If the mouse cursor leaves the field before the Enter key is pressed the edited value will revert to the original value.

---



### 3.3 EDXD and radiography mode switching

Setup of slits and filter for EDXD and radiography measurement.

Open *Slits/Filter Setup* on the PEC user interface.



Input slit sizes and filter setup values in the 'Preset Position' window.

**Warning:** Please do not change other parameters (e.g., Tip X, Y, Z, and so on).

Setup 1 is for EDXD measurement and setup 2 is for radiography measurement. Filter value is typically 0 (no filter) for EDXD measurement and -45 (100 m molybdenum) for radiography measurement. Please close the window after completion. '1st Hsize', '1st Vsize', '2nd Hsize', '2nd Vsize', 'Filter' setups change simultaneously by clicking *Slit for EDXD* (EDXD) or *For Camera* (radiography).

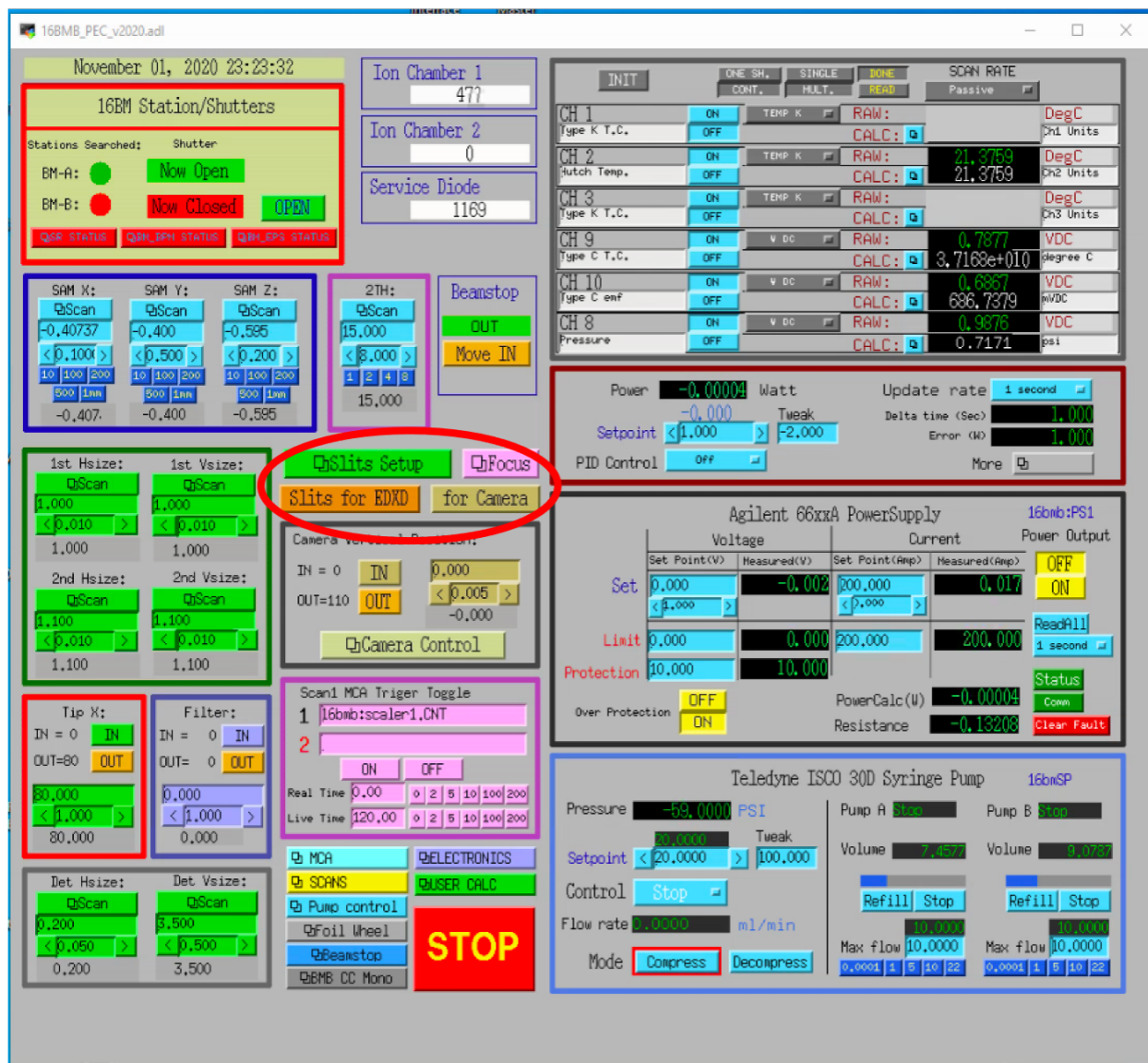
### 3.4 Radiography measurement

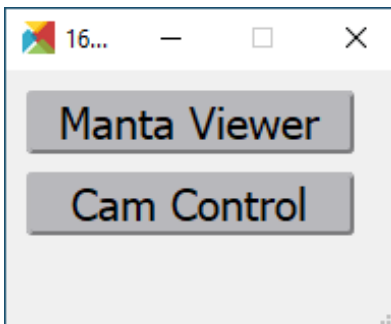
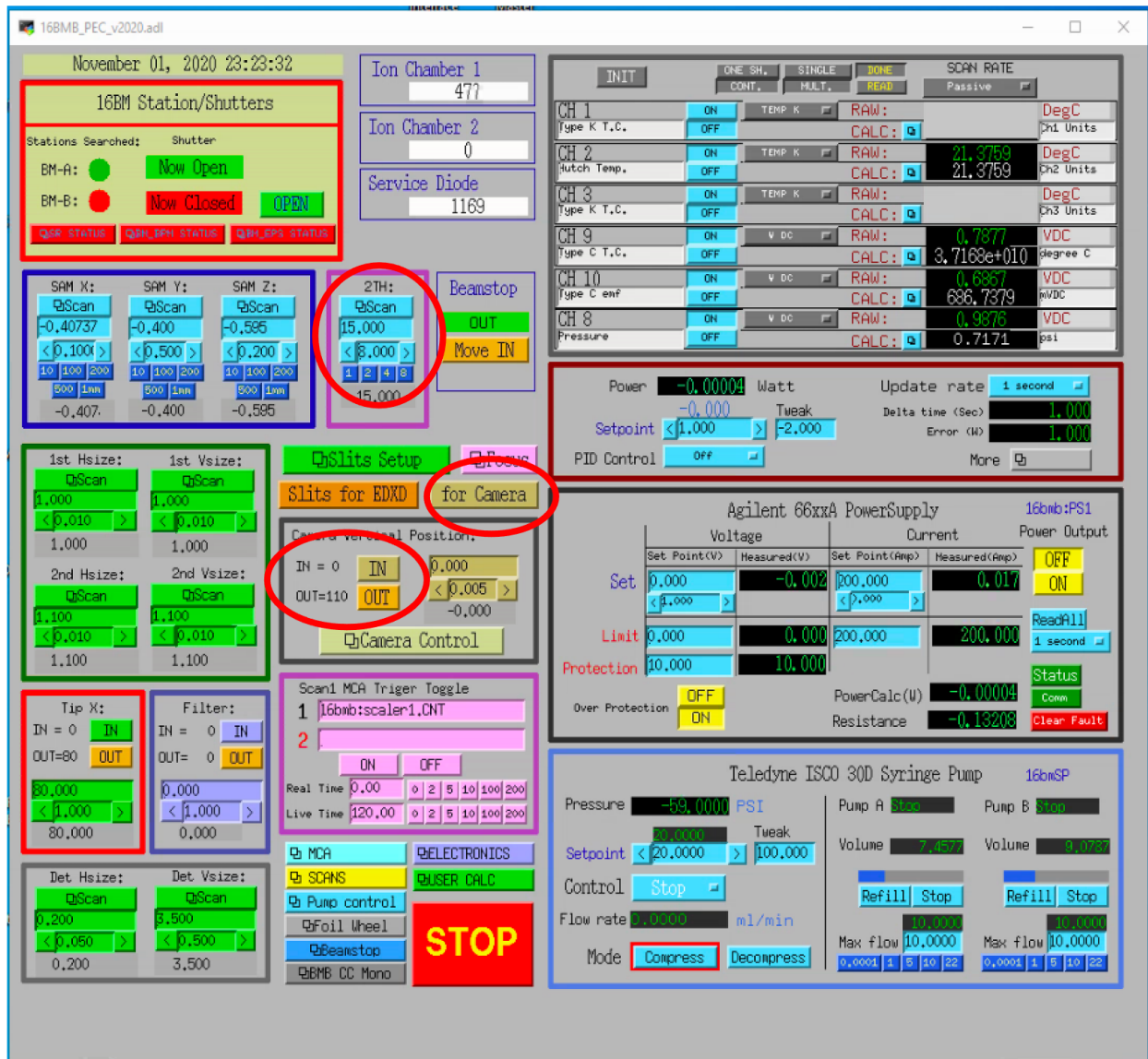
On the PEC user interface,

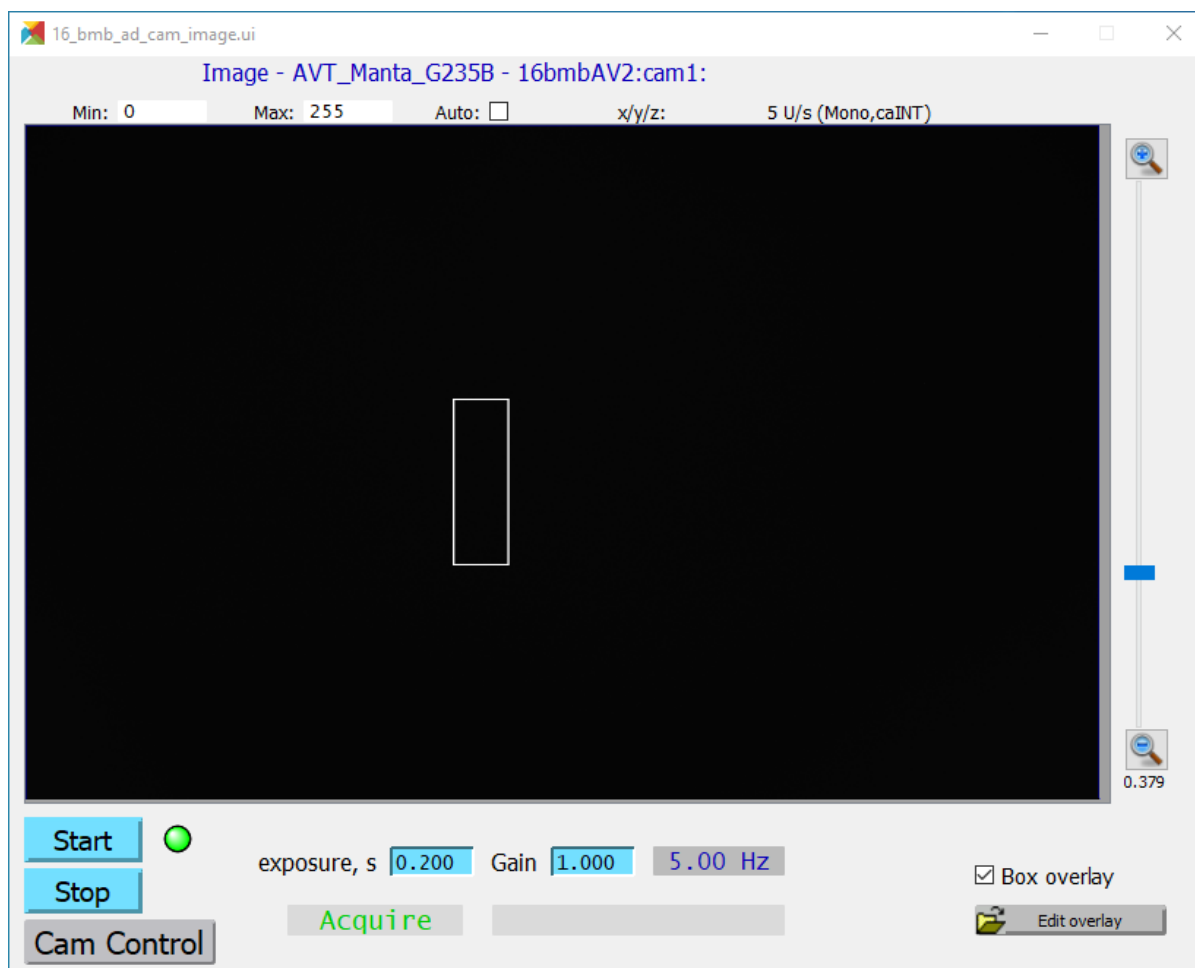
1. Click *for Camera* to open slits and to put filter.
2. Confirm and/or move '2TH' to > 11°.
3. Move camera IN

Open 'Manta Camera' shortcut on desktop. This will open a small launcher window with two buttons *Manta Viewer* and *Cam Control*:

Click *Manta Viewer* in the launcher window to launch the viewer window.

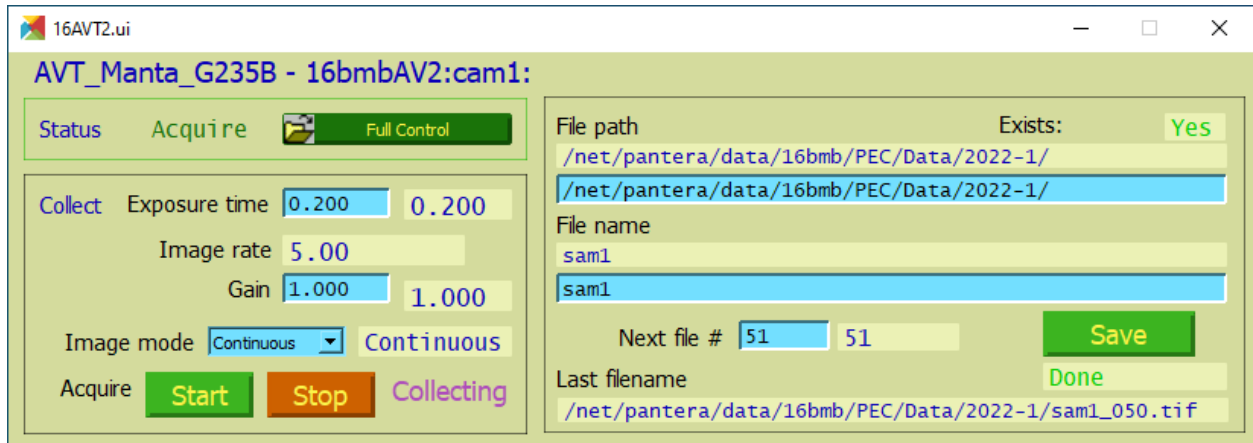






Click *Start* to start camera. Adjust the ‘exposure, s’ if needed.

Click *Cam Control* in the launcher window to launch camera image file saving control.



- Enter the ‘File path’ and the ‘File name’.
- To save the image, click *Save*.

**Note:** The File path for saving the data should be /net/pantera/data/16bmb/PEC/Data/, followed by the run number, eg. 2022-1, followed by beamtime category (GUP, etc.) and PI name.

**Note:** Be careful when copy-pasting the file path from the Windows Explorer into the file saving control. The file saving control uses forward-slashes ‘/’, while windows explorer uses back-slashes ‘\’.

## 3.5 EDXD measurement

On the PEC user interface,

1. Move camera OUT
2. Click *Slits for EDXD* to narrow slits and to remove filter.
3. Move Tip X to IN position

**Important:** Tip X must be moved to OUT position when putting in and removing PE cell to avoid accidentally bumping the collimator.

EDXD data collation and viewing is done using the *hpMCA* software:

1. Open ‘hpMCA’ from shortcut on desktop
2. *File* → *foreground* → *open detector*.
3. Click *OK*, keeping the default PV name.

**Note:** The EDXD detector PV name should be 16bmb:aim\_adc1.





4. Find sample  $Y$ ,  $Z$ , and  $X$  positions before starting EDXD data collection.
5. Start EDXD data acquisition (Refer to [hpMCA](#) section for EDXD data acquisition and viewing).

### 3.6 Bluediamond

The Java-based HPCAT Bluediamond software is a real-time scan viewer program. The user shortcut can be found on the Windows desktop. If the software is started fresh, go to *Configuration* → *open* to open the input configuration file named `16BMB.txt` in the `C:\HPCAT Software` directory. Note that this directory is local, but can be any directory in the network. The software is straightforward to use and most of the menu items are self-instructing.

Various detectors can be displayed in the scan:

- Beam intensity monitor by 'Ion Chamber 1' placed at the entrance of BMB hutch.
- Beam intensity monitor by 'Ion Chamber 2' (used only in absorption density measurement).
- Beam intensity monitor by 'IC2 or Diode' placed at the downstream of sample. This is mainly used for scanning sample  $Y$  and/or  $Z$  position by absorption contrast.
- Intensity of ROI in MCA software. This is mainly used for scanning sample  $X$  position in EDXD measurement.

**Note:** The labels for these detectors can change based on the names of the ROIs. Bluediamond only refreshes the names for the detectors when it is started. If you change the name of the ROI in hpMCA the names will not be updated in Bluediamond until it is restarted.

To use line cursors (two vertical and two horizontal), select menu *Util* → *Markers* → *Reset*. Left and right cursors can be dragged. The cursor feature is useful for graphical determination of the FWHM and peak center position. You can move sample position by clicking *Move* button at the 'Center' in the left column.





## 3.7 Sample Y and/or Z positions search

There are 2 ways to search sample Y and/or Z positions:

- (1) Search by radiography image
- (2) Scan absorption profile
- (3) Scan by EDXD or fluorescence profile (refer to *X position search*)

### 3.7.1 By radiography image

- Switch to *radiography measurement mode*.
- Narrow slit size to those of EDXD measurement.
- Mark the narrow slit size and position by tape or something on monitor.
- Open slit (click for *Camera*) for radiography measurement.
- Move sample position to x-ray beam position shown by a mark on monitor.

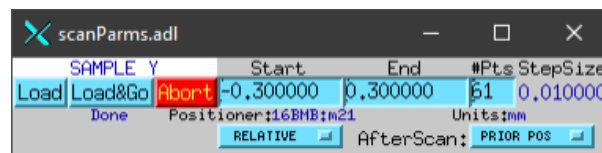
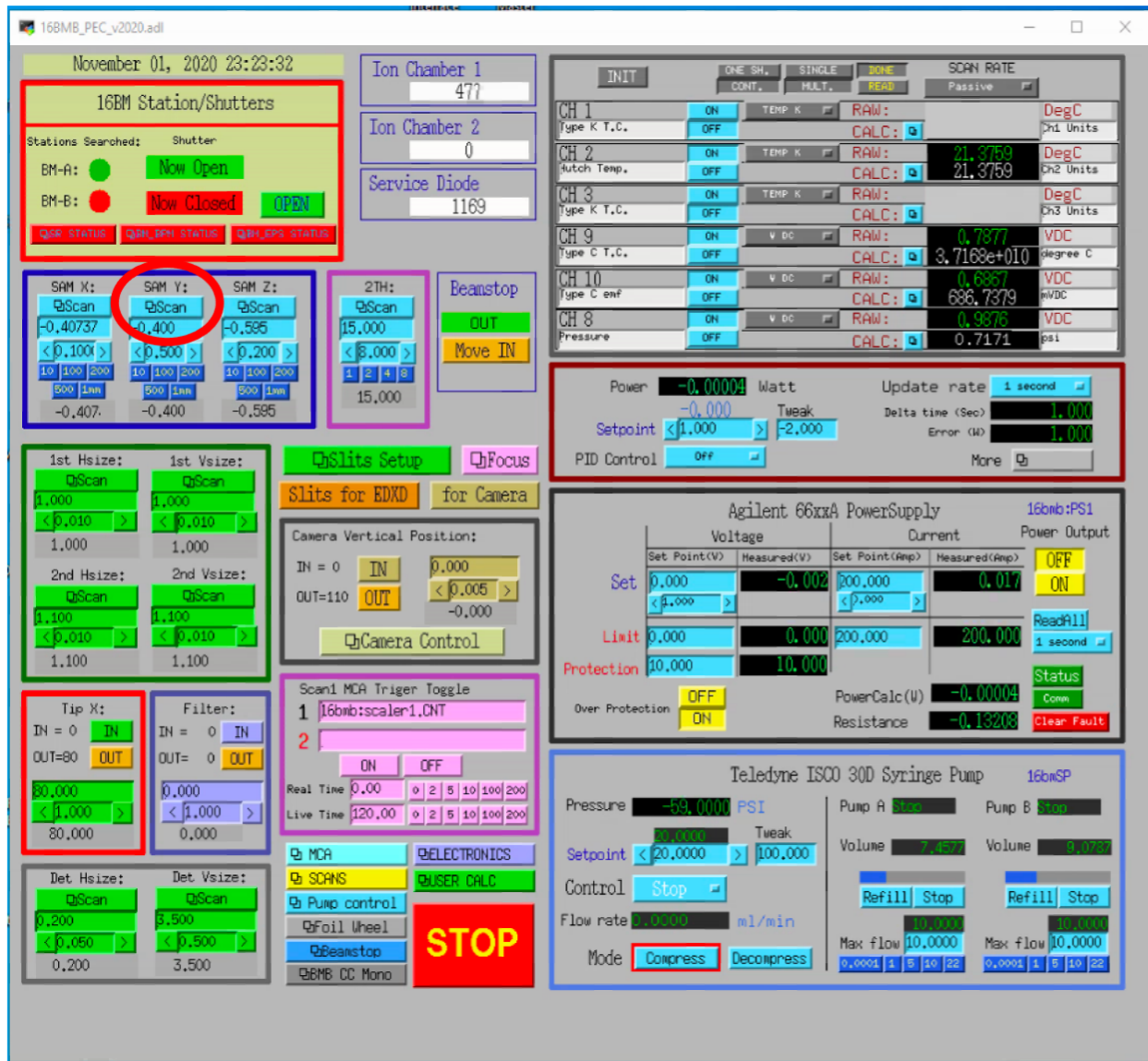
### 3.7.2 By absorption scan

- Move to *EDXD measurement mode*.
- Confirm 2TH is  $>10^\circ$ .
- Open 'Bluediamond' (*Bluediamond* software).
- Select 'IC2 or Diode' in Detector in Bluediamond, uncheck all others.
- Open 'scan' on SAM Y (or Z)
- Set scan parameters of 'Start', 'End' and '#Pts' (#Pts has to be odd number) (confirm 'Relative').
- Click *LoadGo* to start scan.
- Scan results will appear in the '*Bluediamond*' window.

## 3.8 Sample X position scan

Sample X position can be adjusted by using intensity of sample or diffraction pattern. However, it is difficult to scan sample X with diffraction intensity of amorphous material. We recommend scanning sample X by using diffraction intensity of MgO ring. Followings are procedures:

1. Move Y -1.5 mm from sample Y center to see diffraction patterns of MgO.
2. Add *ROIs* for MgO peaks.
3. Then, move Y -1.3 mm position from sample Y center (+0.2 mm Y from -1.5 mm position or move back to the sample Y center and move -1.3 mm Y).
4. In order to connect EPICS motor control and MCA software, please click *ON* in 'Scan1 MCA Trigger Toggle', and then input data acquisition time for each step in 'Preset Real Time' (typically, 2-5 second).
5. Open 'Scan' in 'SAM X', and input parameters (typically, Start=-1, End=1, #Pts=21).
6. Then, click *LoadGo* to start scan.

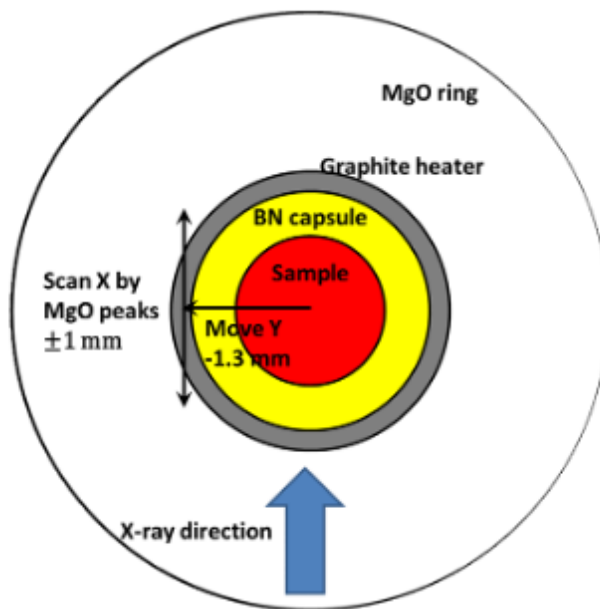


7. Sample X center is the location where MgO diffraction intensity is the minimum.

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**Note:** After the scan, please do not forget to 'OFF' 'Scan1 MCA Trigger Toggle', and input 0 in 'Preset Real Time'.

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## 3.9 Pressure control

The PEC oil pressure is controlled by the Teledyne ISCO 30D dual syringe pump system. The maximum pressure allowed is 14,000 psi (9,000 psi for ultrasound or grooved cells). Syringe pump is controlled through the MEDM interface

Basic pump operation Procedure for increasing, maintaining, and decreasing pressure.

**Warning:** Due to a problem with the pump controller, sometimes communication with the pump gets lost and the indicators colors will change to white. If this happens, please wait around 1-2 minutes, the communication should get re-established on its own. Afterwards, you may need to re-do the last command.

### 3.9.1 Compression

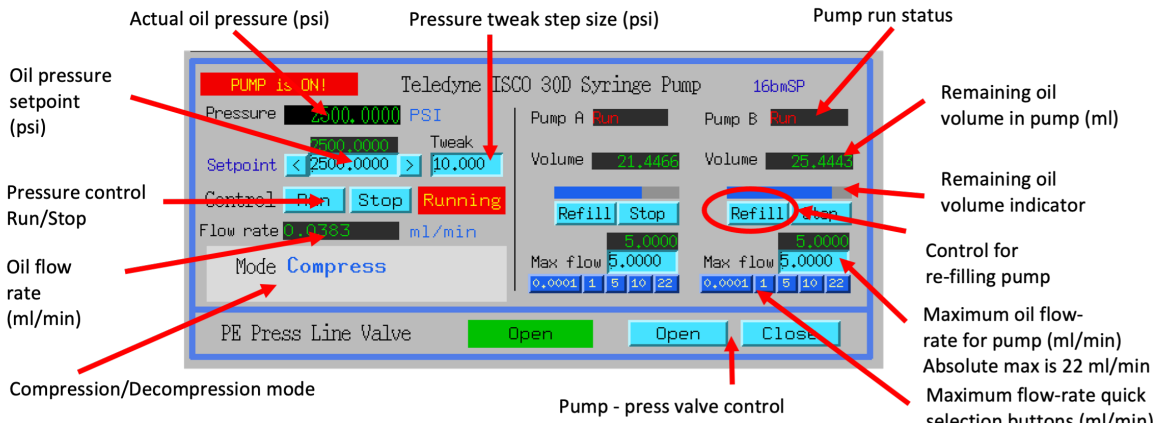
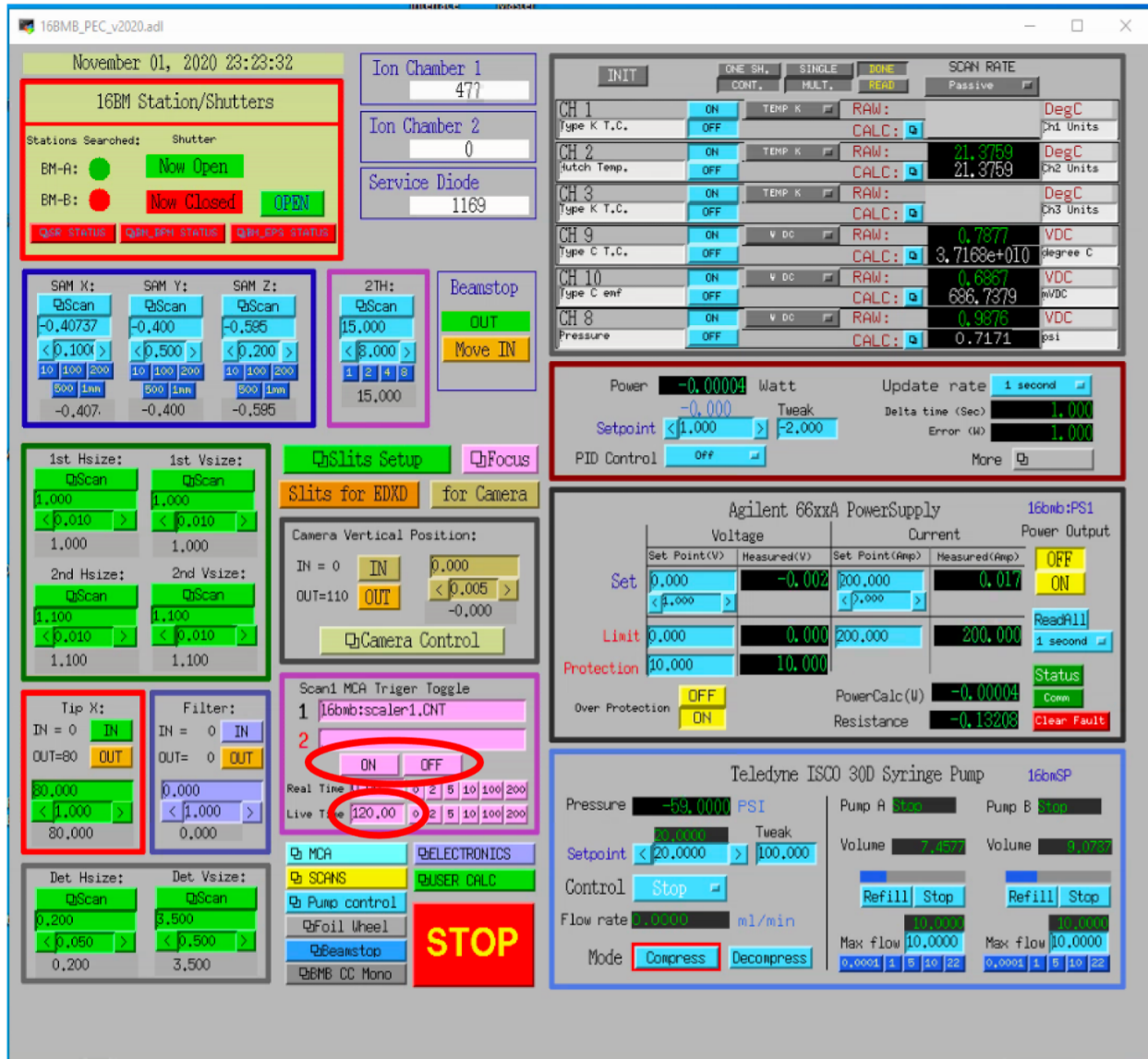
1. Make sure Mode is selected as "Compress".

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**Note:** Stop the Pressure Control before switching Mode. (Mode button is hidden while Pressure Control is in "Run" state).

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2. Refill pumps A and B (click the button *Refill* for each pump).



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**Note:** Wait until both pumps finish refilling.

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3. Set Max flow for both pumps to 5ml/min.
4. Set the Oil pressure setpoint to 20 psi.
5. Set Pressure control to Run. Pump will go through the initial equalization sequence; this will take around 30 seconds to one minute.

---

**Note:** Pressure may go up to ~80 psi and fluctuate somewhat during this process. Wait until the Actual oil pressure stabilizes at 20 psi.

---

6. Increase the Oil pressure setpoint to your required pressure (maximum allowed is 14,000psi). Pump will gradually reach the setpoint pressure and maintain the pressure continuously.
7. If you don't want the pump to maintain the pressure continuously after reaching the setpoint, set the Maximum oil flow-rates for pumps A and B to 0.0001 ml/min.

---

**Important:** DO NOT switch Pressure Control to Stop.

---

8. To reach the next oil pressure setpoint, re-enable pressure control by setting Max flow rates back to 5 ml/min.

### 3.9.2 Decompression

1. Set Pressure Control to Stop.
2. Set Mode to Decompress.
3. Set Pressure Control to Run.

---

**Important:** Wait around 1 minute before doing anything else. After around 30 seconds, one of the pumps (A or B) will start emptying out (there will be a valve opening/closing sound). Wait until the level in that pump reaches around 7.5 ml.

---

4. Set the setpoint pressure to 20 psi.
5. After the actual oil pressure is at 20 psi, switch pressure control to Stop.
6. Open the valves to vent the remaining oil pressure:
  1. Open valve control from the main PEC interface menu "Pump control menu"
  2. Toggle Valves 1-4 to Low.

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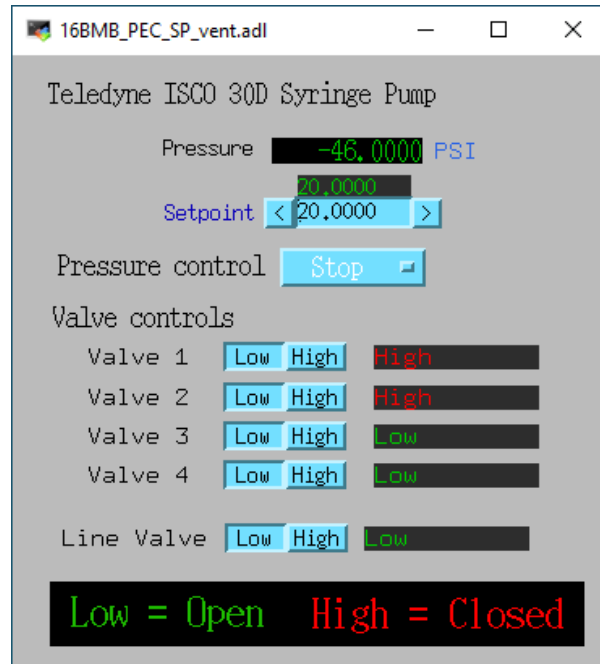
**Note:** If the readback text for a valve is high (red), and pressed button is low: click the *high* button and then the *low* button.

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**Note:** If the valve 1-4 buttons are hidden check the following conditions are met:

- Pressure :  $\leq 20$ psi



- Pressure setpoint: 20psi
- Pressure control: stopped

### 3.9.3 PE Press Live valve

The Live valve is an electronically actuated valve in-line between the Syringe pump and the PE press. The Line valve allows to keep the pressure in the PE press isolated from the Syringe pump when changing modes (Compression <-> Decompression), when heating, or when leaving the PE press unattended for an extended period of time. This subsection describes the operation of the Line valve.

## 3.10 Heating control

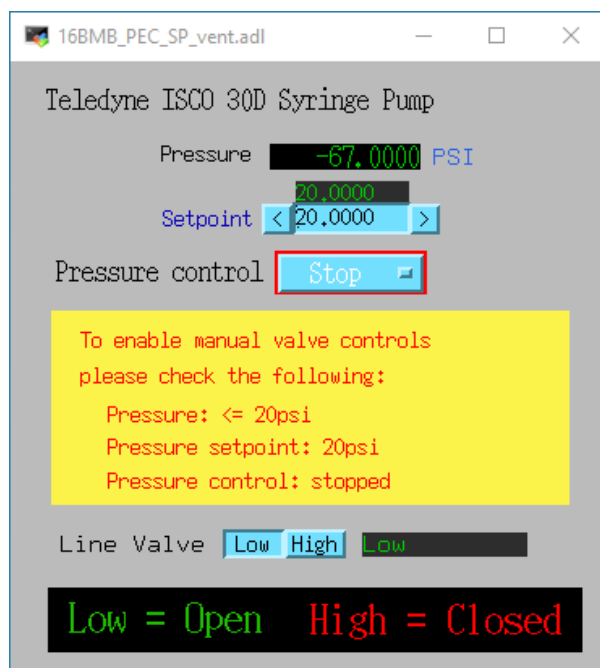
Before connection of cable, please confirm 'Power Output' in 'PEC User Interface' is 'OFF'.

In hutch, please confirm 'Heater Output Control Switch' is 'Disabled'.

1. See that the thick power cables are connected.
2. Turn On a fan on PE press for cooling of press body.
3. 'Enable' on the 'Heater Output Control Switch'.
4. Before starting heating, it is recommended to start '*LogBook*' to save log of heating.

On 'PEC User Interface',

1. At first, please confirm 'Voltage' 'Set Point (V)'=0, 'Setpoint (Watt) on PID control = 0, and 'Over Protection' is ON.
2. 'Power Output' ON
3. Input 200 in 'Limit' under 'Current'. Please input again even if the value is 200.



4. Click *Clear fault*.
5. 'PID ON/OFF' ON
6. Tweak 'Setpoint (Watt)' by 1 W to 3 W.
7. Check 'Readback (Watt)' is responding, and 'Resistance' is lower than 0.1 (typically, ~0.04-0.05 at ~1 W).

---

**Note:** Response of heater is slow particularly at <10W. Please wait a while.

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**Important:** Increase of 'Readback (Watt)' may stop at <3W. If so, please check 'Measured (Amp)' under 'Current'. If 'Measured (Amp)' value is 2.65, it is likely to forgot the procedure 3 (Input of 200 in 'Limit' of 'Current'). In this case, please lower 'Setpoint (Watt)' to 0, turn OFF the 'PID ON/OFF', input 0 in Set Point (V), and turn Off the 'Power Output'. Then, please restart the procedures.

---

8. If heater response and resistance is okay, increase 'Setpoint (Watt)' slowly (it is better to keep <5 difference between 'Readback (Watt)' and 'Setpoint (Watt)').

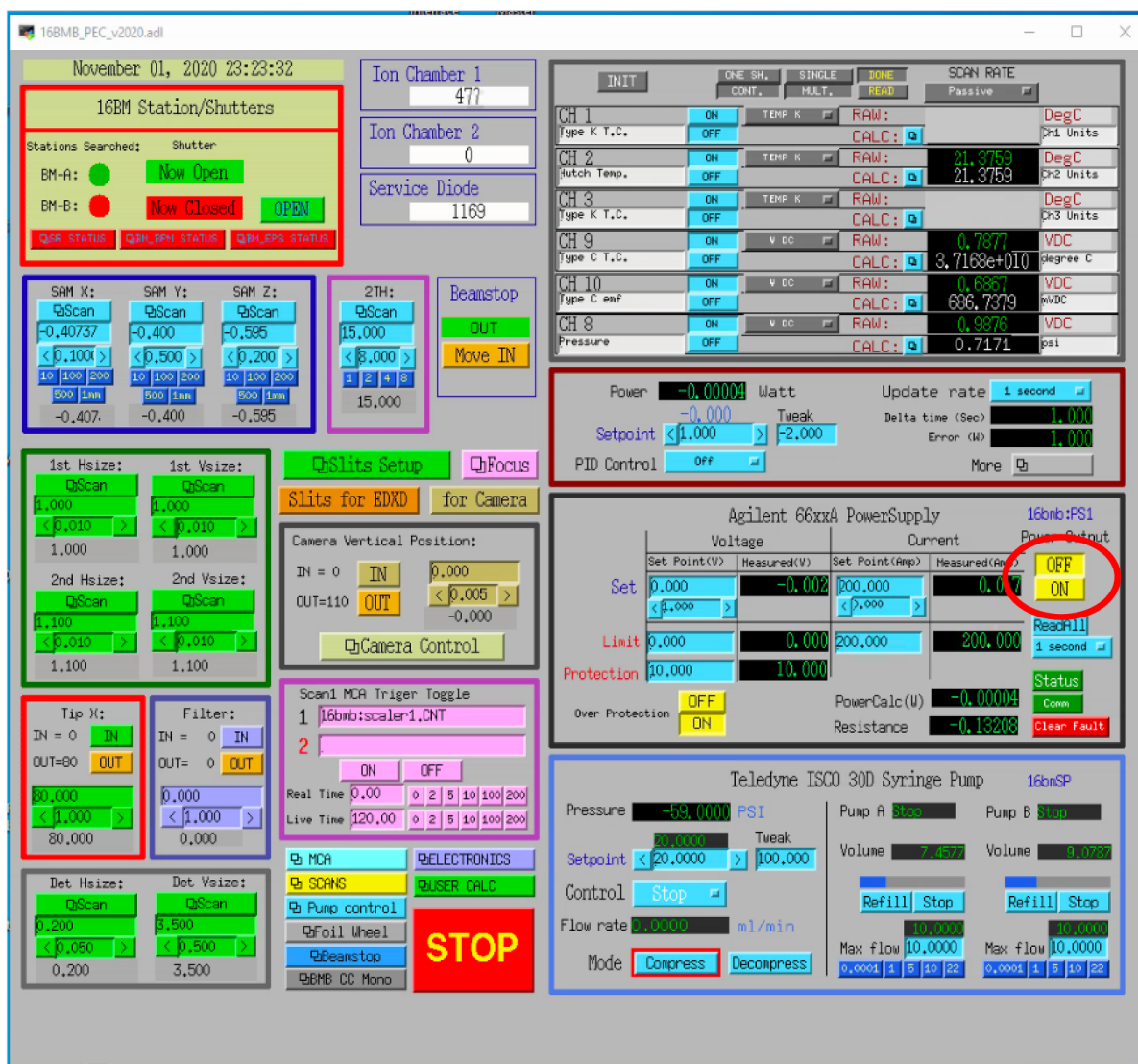
Cooling can be done by

- (1) slow cooling by gradually decreasing 'Setup (Watt)' to 0, or
- (2) Turn OFF 'Power Output' to quench sample.

In both cases, after cooling,

1. Input 0 in 'Setup (Watt)'.
2. 'PID On/OFF' OFF
3. 'Power Output' OFF
4. Input 0 in 'Set Point (V)' under 'Voltage'.







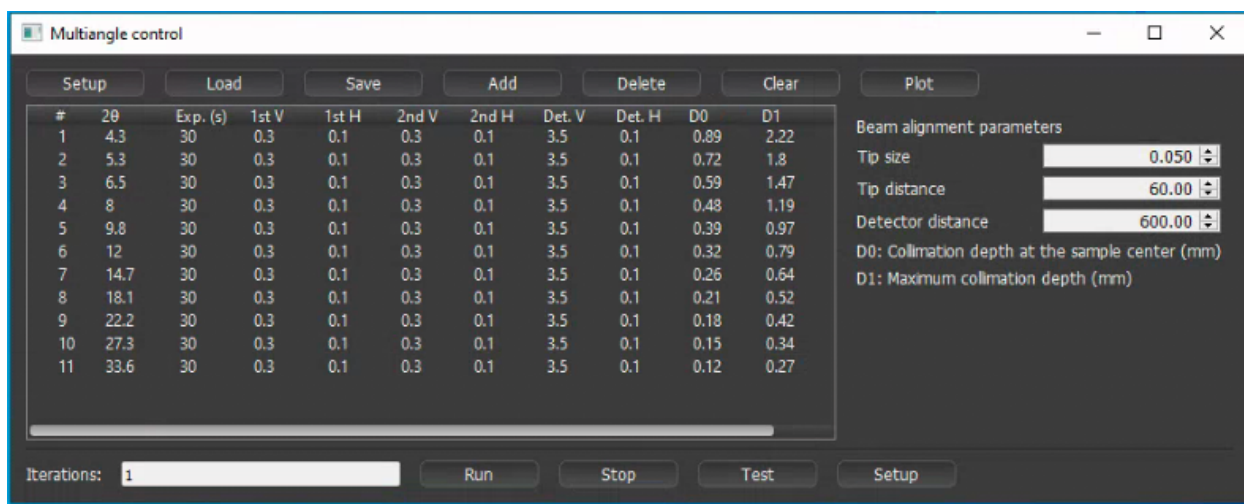
5. 'Disable' on the 'Heater Output Control Switch' in the hutch.

**Danger:** Do not touch on press until turning off the power of heater power supply. Even after the power off, please take care. If you heated more than 1000 °C for more than several hours, press body may be hot. Please wait until the press body is cool.

## 3.11 Liquid/amorphous structure measurement

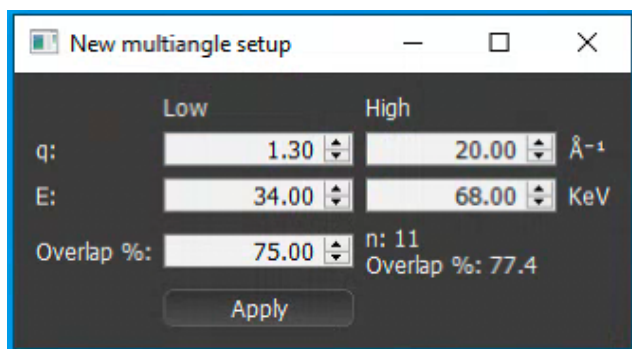
A python program 'multiangle.py' is available for automatic data acquisition of EDXD pattern with varying 2 angle.

- Open the python program by running 'multiangle.bat' from the desktop shortcut.



You have the following 3 options:

1. Create a new setup automatically by clicking Setup in main window. In the pop-up window enter desired q-range and usable E range, and % overlap for the measurements. The built in algorithm will calculate optimal 2theta angles and populate the main window.



2. Load previously saved setup, click *Load* in main window
3. Add 2-theta angles manually by clicking *Add* in the main window for each angle.

Adjust the slit sizes and exposure times for each 2-theta

Table 1: Input paramters description

Setting	Description
2	2theta angle
Exp. (s)	Data collection time in Live time (i.e. Actual data acquisition time is Live time + Dead time)
1stV	1st slit Vertical size
1stH	1st slit Horizontal size
2ndV	2nd slit Vertical size
2ndH	2nd slit Horizontal size
Det.V	Detector slit Vertical size
Det.H	Detector slit Horizontal size

If you want to repeat measurement, you can set 'Iterations' = 2 or higher.

---

**Important:** Confirm the following:

- 'Camera Vpos' = 110, 'Beamstop' = OUT, 'Tip X' = 0
  - 'Scan1 MCA Trigger Toggle' = OFF (nothing in line 2) Both 'Preset Real Time' and 'Preset Live Time' = 0
  - Slit and Filter setup is 'EDXD' condition ('Filter' = 0, slit size is small) 'position of sample is correct'.
- 

Then, in *hpMCA*:

1. Make sure file number is set to auto-increment
2. Open *File* → *Preferences*
3. In preferences, please check 'yes' for 'autosave when acquisition stopped'. (hpMCA will save file for each angle data with the name suffix of '\_001', '\_002'...).

Then, to start multiangle measurement,

- On Multiangle control window, click *Run*
- 

**Important:** After finishing the Multiangle collection, please do not forget to check 'no' for 'autosave when acquisition stopped'.

---

If you want to stop the Multiangle measurement, click *Stop*.

## 3.12 Ultrasound measurement

A GUI porgram sonicPy is used for the ultrasound measurement. SonicPy allows to automatically record ultrasound ultrasound waveforms from the oscilloscope with varying exitation wave frequencies.

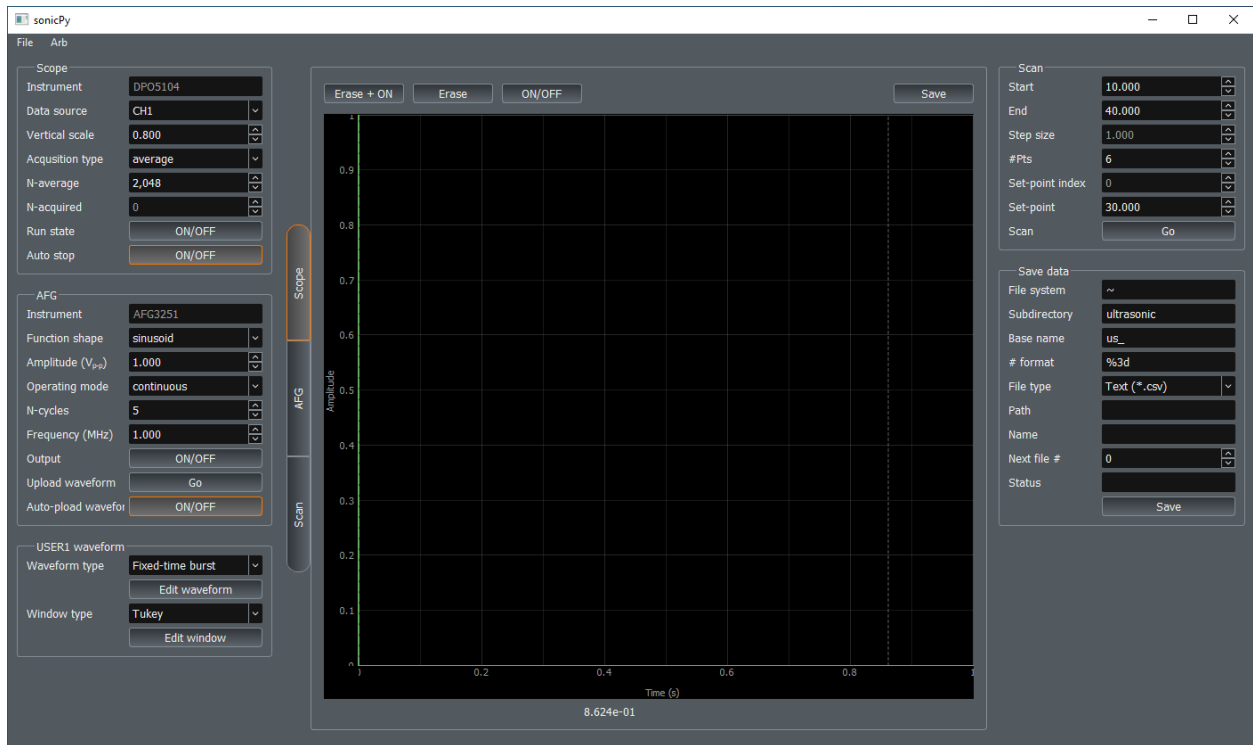
Before beginning, create a folder named **US** in your data folder where the ultrasound data will be saved to.

1. Open sonicPy by running the **ultrasound measurement** shortcut of the desktop.

---

**Important:** Check that the Scope-Instrument is DPO5104 and AFG-Instrument is AFG3251. If something else is displayed it means that the program could not communicate with the hardware. Check that the oscilloscope and the function generator are both powered on.

---



2. Check and update if needed the following settings values:

Table 2: Scope settings

Settings	Value
Data source	CH1
Vertical scale	~1
Acquisition type	avarage
N-average	1000-4000
Autostop	On

Table 3: AFG settings

Settings	Value
Function	user1
Amplitude	5
Operating mode	burst n-cycles
N-cycles	1
Output	On
Auto-upload waveform	On

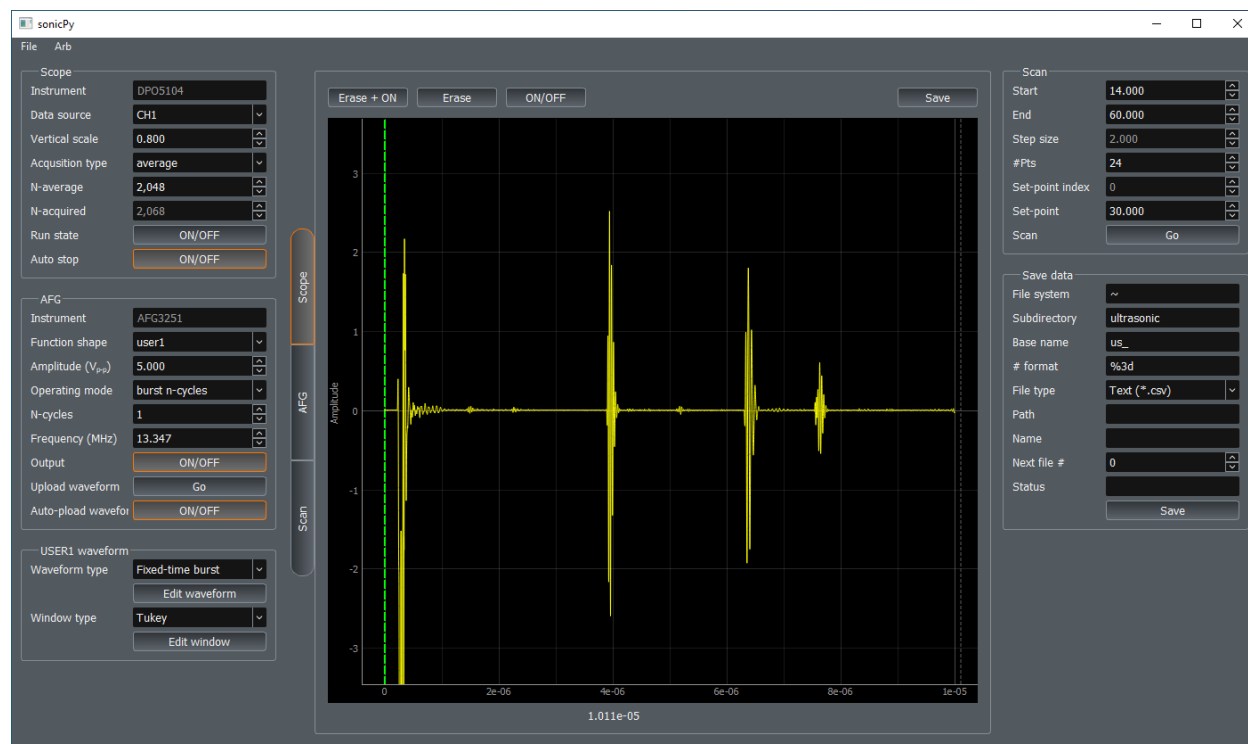
Table 4: Scan settings

Settings	Value
Start	14
End	60
#Pts	24

Table 5: Save data settings

Settings	Value
File system	file system path to the US data folder which you created
Subdirectory	P-T condition (eg. “1000psi” or “2000psi_500watt”)

3. To check the ultrasound signal level, clear the scope and acquire a new ultrasound waveform by clicking *Erase* + *ON* above the waveform plot.



**Note:** Scope Vertical scale may need to be adjusted depending on the signal level to avoid saturation and to optimize the oscilloscope’s dynamic range relative to the signal level.

4. Set the ‘Next file #’ zero, if needed; and click *Go* in the Scan panel. The the frequency sweep will start and conclude on its own. To interrupt the scan mid-way click *Go* again.

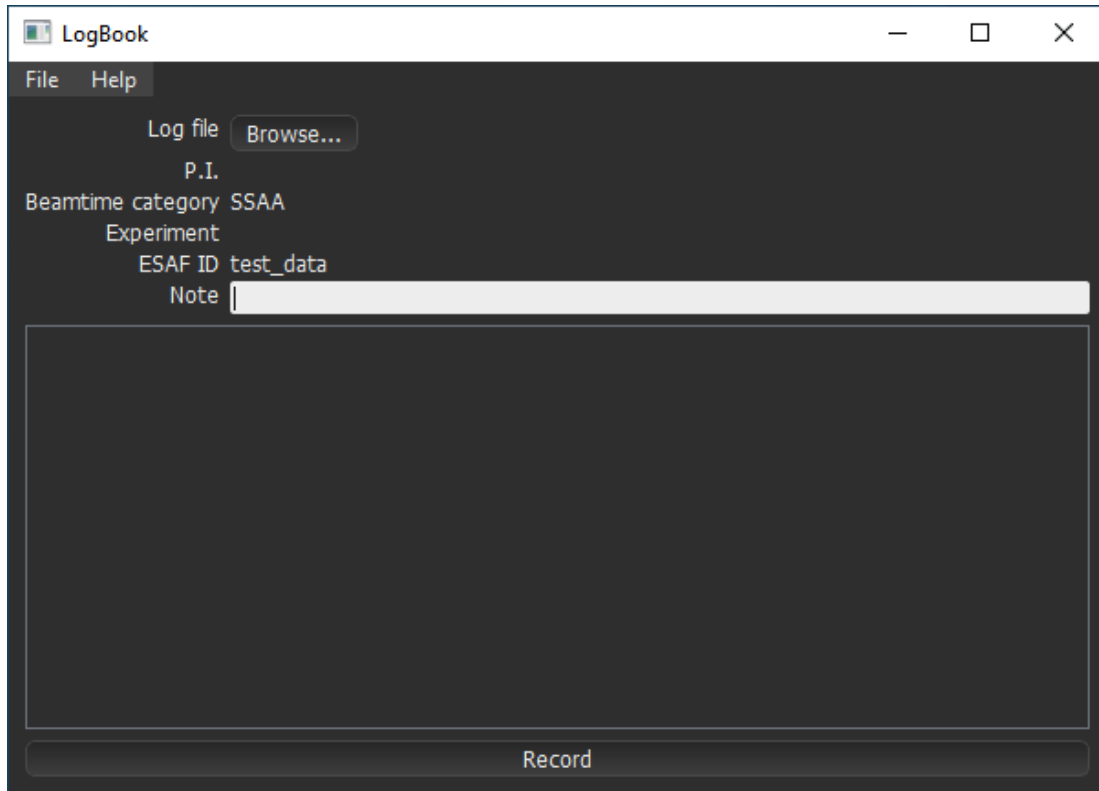
**Important:** Remember to reset Next file # back to 0 before each scan.



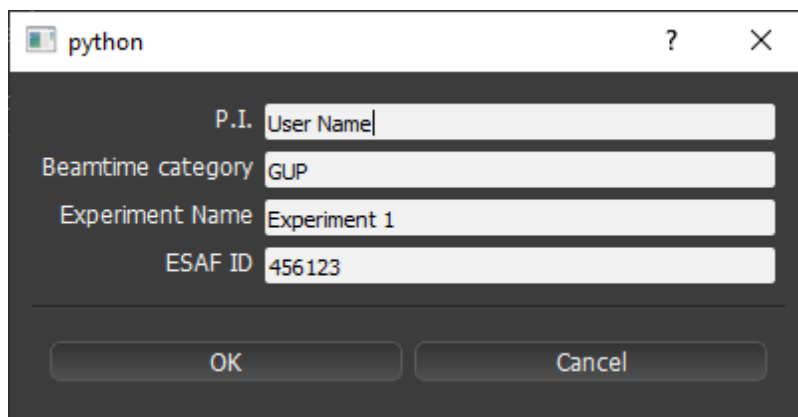
### 3.13 General data logging

A GUI program **Log book** can automatically log compression, heating, and other records whenever new EDXD and/or radiography files are saved.

1. Open 'Log book' from desktop shortcut.



2. *File* → *New log*



3. Next to 'Log file', click *Browse...* and choose the name for the log file, typically a new file in your own experiment data folder.

Logging can occur either automatically or manually. The program will automatically log a list of process variables (PV's) and data file names each time a new data file is saved (EDXD, or radiography). Alternatively, click *Record* at

any time to log the then-current experimental parameters. A time-stamped row of values will be appended to the log file each time.

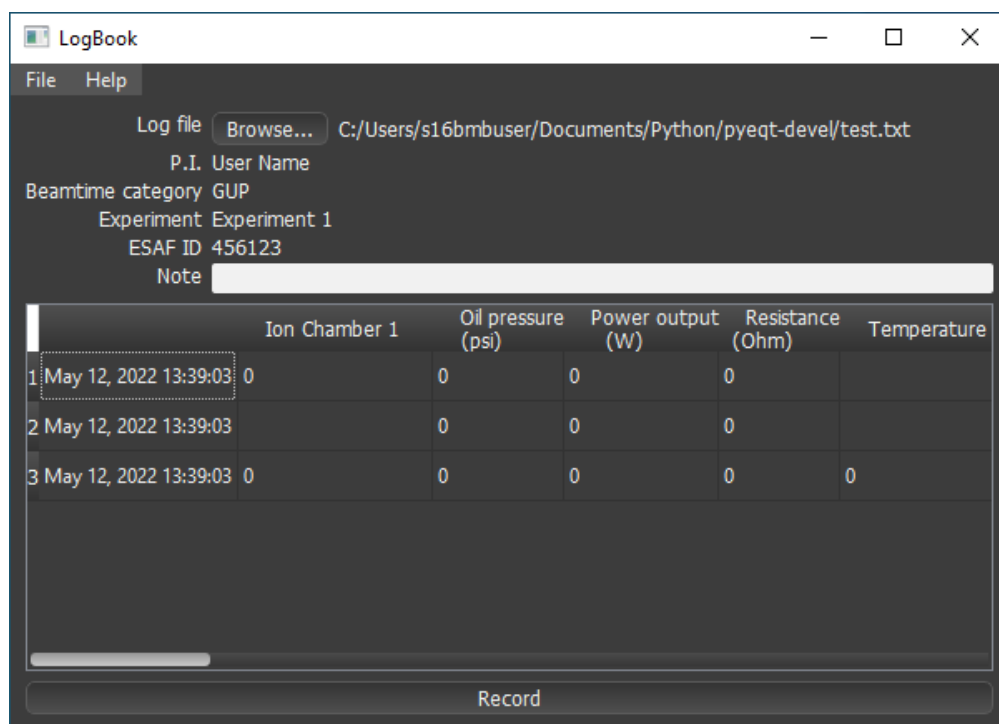
**LogBook** allows recording any process variable (PV); however a pre-defined list of PV's is stored in setup \*.env file in the program's resources directory.

The following default PV's will be logged:

Table 6: LogBook PV's

Description	PV
Ion Chamber 1	16bmb:scaler1.S2
Oil pressure (psi)	16bmb:DMM1Ch8_calc.VAL
Power output (W)	16bmb:PS66xxA:PS1:SR:powerOutPut
Resistance (Ohm)	16bmb:PS66xxA:PS1:SR:Resistance
SAM X (mm)	16bmb:m20.RBV
SAM Y (mm)	16bmb:m21.RBV
SAM Z (mm)	16bmb:m19.RBV
2-Theta motor (deg)	16bmb:m5.RBV
Tip X (mm)	16bmb:m53.RBV
Filter wheel (deg)	16bmb:m13.RBV
Experiment note	16bmb:exp_log:Note

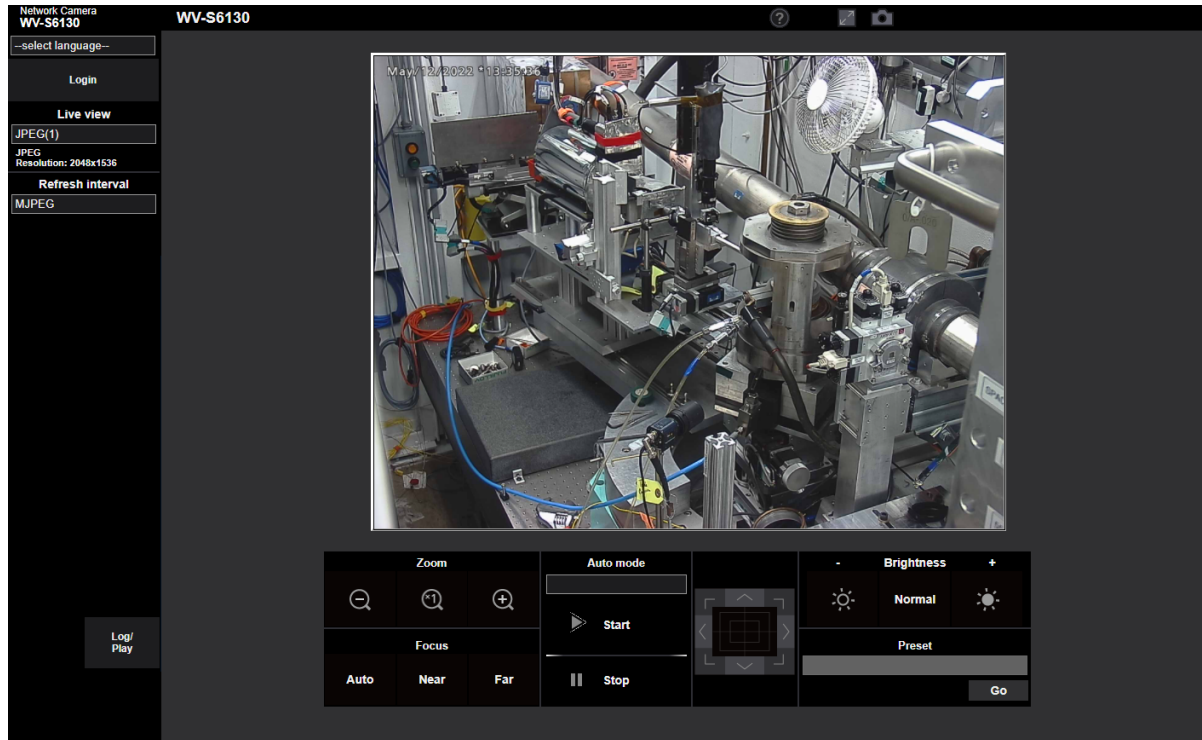
**Note:** Text entered into the 'Note' field will also be logged each time. The Note text can be changed by the user at any time and can be up to 80 characters long.





## 3.14 Hutch webcam

16-BM-B hutch is equipped with an overview camera with motion and zoom capabilities. To open the camera viewer use shortcut **WV-S6130 Network Camera** on the desktop.



Moving (re-centering the image) of the camera is possible by clicking on the cam view image, the clicked point will become the new center. Zooming-in is possible by drawing a rectangle over the region of interest in the cam view.

## 3.15 Data transfer

Data collected during the experiment can be transferred to users' personal computers using [Globus](#).

Please follow this user manual to transfer your experimental data: [GlobusPersonalConnect for Users](#).

### 3.15.1 Data transfer tips

Understand the data transfer and sync is one-way (source to target).

- Two options (ask your local contact to setup):
  - Upload the data all at once after completing the experiment
  - Real time data upload and sync
- During the real time upload and sync, creation of new folders and files, and modification of the existing ones at source will be synced real-time (with some latency depending on internet traffic).
- Deletion of existing ones, however, cannot be synced and may mess up the folder trees at Globus endpoint. Use real-time sync with a caution.



## **TROUBLESHOOT**

Troubleshooting section



## BEAMLINE SUPPORT

Call the floor coordinator: either call 2-0101 and leave the beamline phone extension or use this [link](#)  
16-BM-B page on APS website.

Please open a ticket using the github [Issue Tracker](#).

### 5.1 Beamline scientists contacts

Ross Hrubia (PEC)

Office: (630) 252-2264

email: [hrubia@anl.gov](mailto:hrubia@anl.gov)

Guoyin Shen (PEC)

Office: (630) 252-0429

email: [gyshen@anl.gov](mailto:gyshen@anl.gov)

Dmitry Popov (White Laue Diffraction)

Office: (630) 252-7106

email: [dpopov@anl.gov](mailto:dpopov@anl.gov)



## DOCUMENTS FOR BEAMLINE STAFF

### 6.1 Standard Operating Procedures

Globus dm-station-gui Staff's Guide

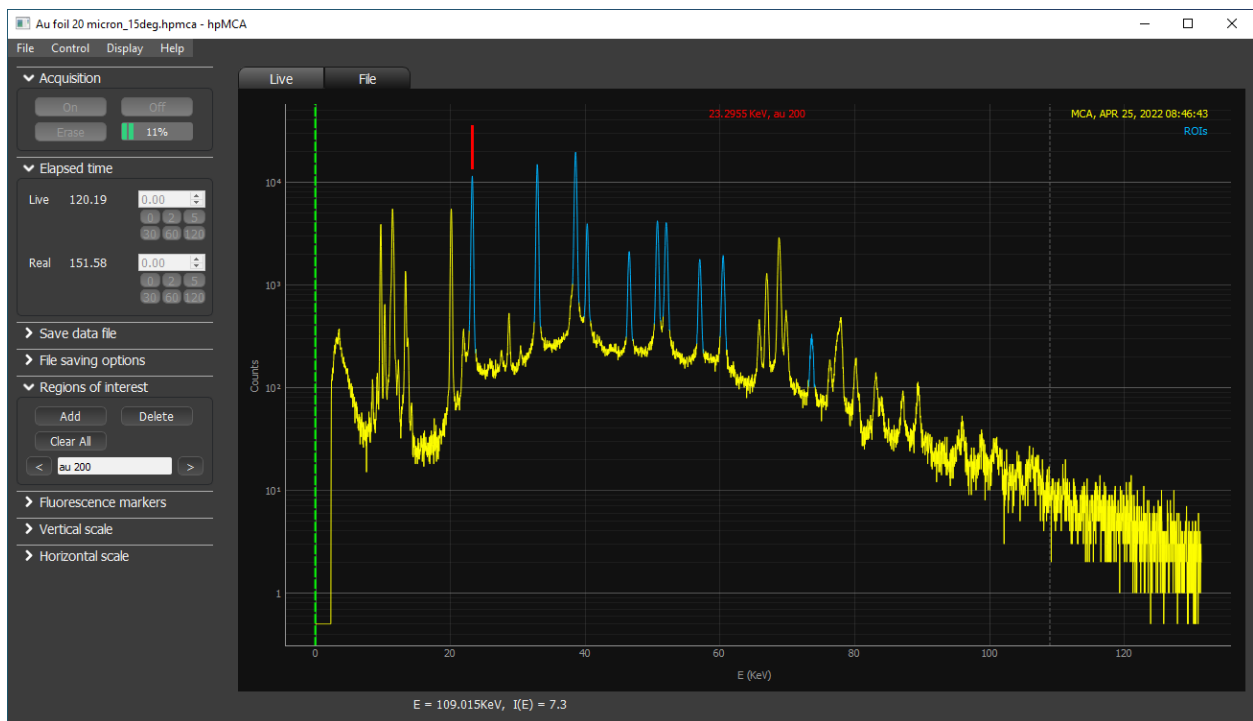
Moba MEDM shortcut instructions

Moba MEDM supplement





## 7.1 GUI basics



Left panel (from top to bottom):

- *Acquisition*
- *Elapsed time*
- *Save data file*
- *File saving options*
- *Regions of interest*
- *Fluorescence markers*
- *Vertical scale*
- *Horizontal scale*

The plot area supports the following mouse commands:

- **Left Click:**  
Move the vertical line cursor to the current mouse position.
- **Left Drag:**  
Zoom-in to the selected area.
- **Right Click:**  
Zoom out.
- **Mouse Wheel:**  
Zoom in and zoom out centered around the current mouse position.

Bottom panel: Energy and Counts information corresponding to location selected by the cursor

Menu bar:

- **File**
  - Open file
  - Open detector
  - Overlay
  - Save as
  - Export pattern
  - Environment
  - Multiple spectra
- **Control**
  - Calibrate Energy
  - Calibrate 2-theta
  - Set 2-theta
  - Set wavelength
  - Presets
- **Display**
  - Phase
  - Fluorescence
  - Regions of interest
  - Lattice refinement
  - Colors options
  - ROI options

## 7.2 Acquisition

- To start data collection, press *Erase* and then *On* in 'Acquisition' panel.
- To stop data collection and to save, press *Off*.

## 7.3 Elapsed time

Elapsed time section

## 7.4 Save data file

Save data file section

Data files are saved with a \*.hpmca extension.

## 7.5 File saving options

File saving options section

## 7.6 Regions of Interest (ROI)

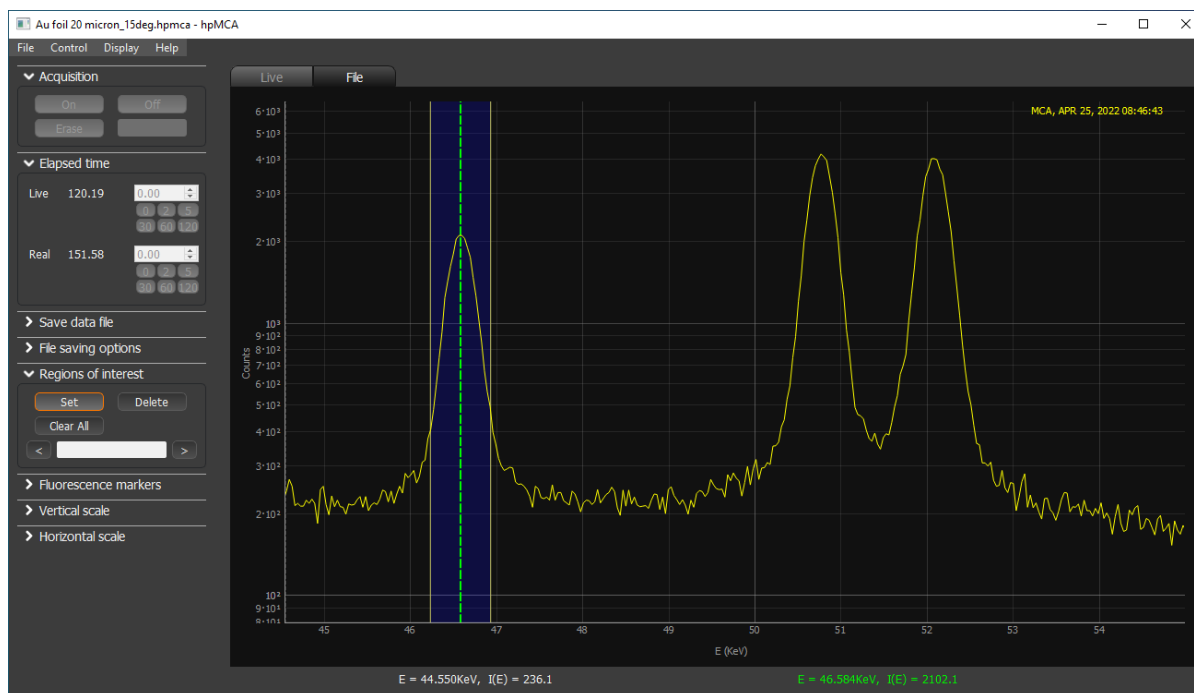
There are two methods to add an ROI.

- (1) *Manual selection of ROI.*
- (2) *Make ROI on all peaks for a crystal by using the JCPDS data.*

### (1) Manual selection of ROI

1. Select center of region of interest by moving cursor to that position (Left-click with mouse).
2. Click, *Add* button in 'ROIs' panel. The button will now read 'Set'.
3. Drag left and right extends of the ROI to appropriate positions.
4. Then, click *Set* button in 'ROIs' panel. The ROI area should now be a different color (default – blue).
5. Currently selected ROI is indicated by a red cursor above it.
6. The Centroid of the selected ROI is displayed in top-middle of the plot.
7. Different ROI can be selected by  $\leq$  and  $\geq$  buttons in the ROI panel.
8. More information about the ROIs can be displayed in the 'ROIs control' by selecting menu: Display/ROIs.
9. You can change the name of any ROI by double-clicking and typing a new name in the name column.
10. Peak fit can be displayed by clicking 'Show fit' button in ROIs control window.

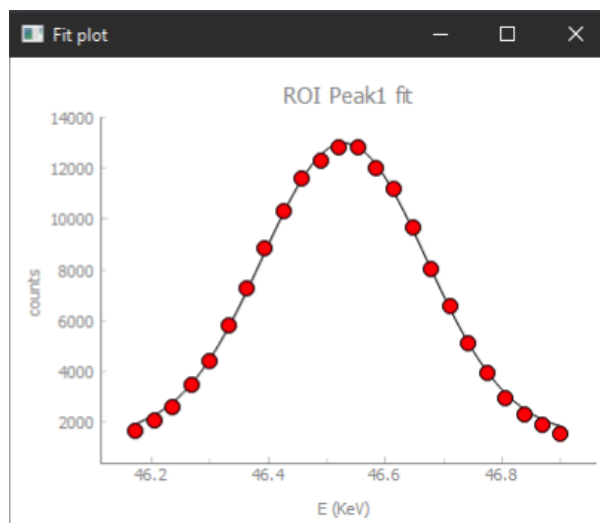
### (2) Make ROIs on all peaks for a crystal by using JCPDS phase



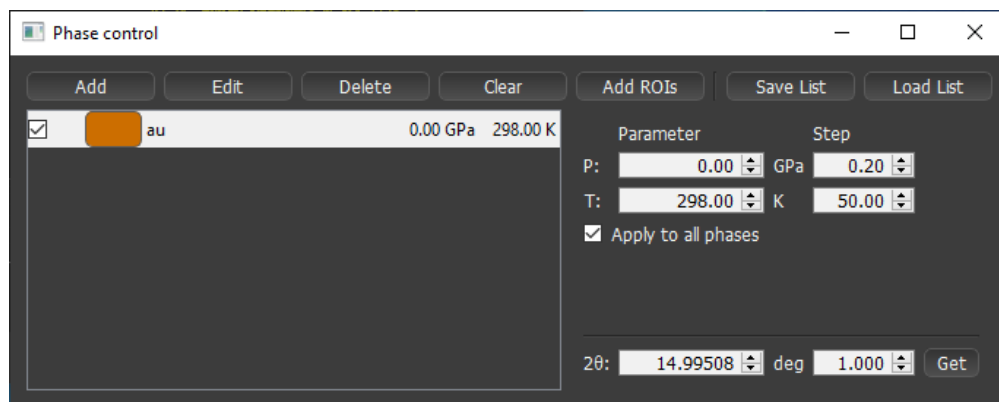
Regions of interest

Delete Clear Show fit Save List

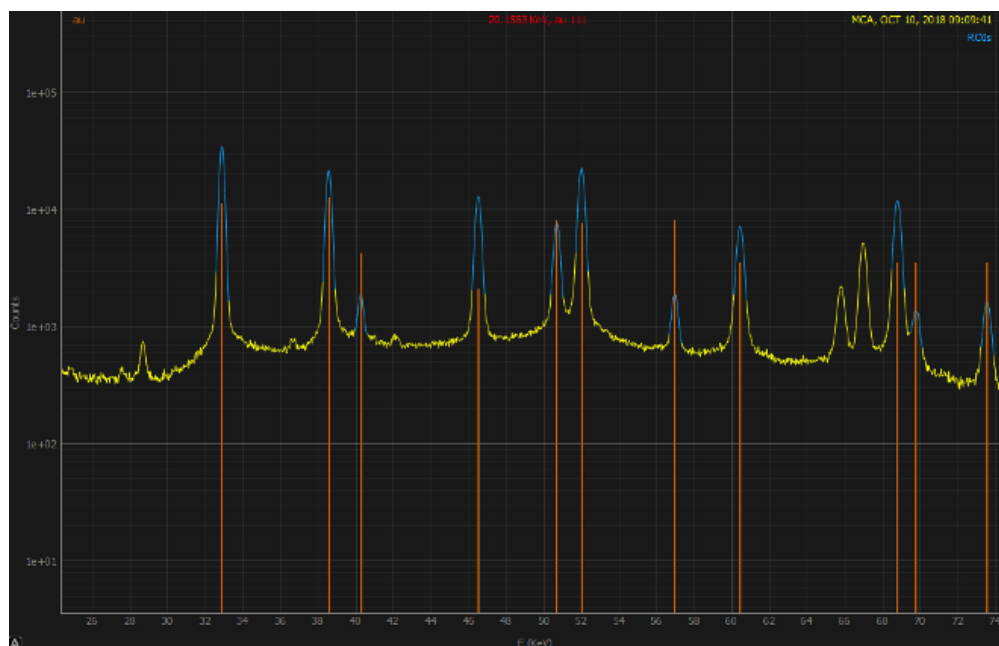
Use	Group	#	Use	Name	Center, E (KeV)	Counts	fwhm
<input checked="" type="checkbox"/>	au	0	<input checked="" type="checkbox"/>	au 111	20.179	5473	0.228
		1	<input checked="" type="checkbox"/>	au 200	23.295	11451	0.268
		2	<input checked="" type="checkbox"/>	au 220	32.948	14843	0.290
		3	<input checked="" type="checkbox"/>	au 311	38.631	19541	0.319
		4	<input checked="" type="checkbox"/>	au 222	40.351	3928	0.309
		5	<input checked="" type="checkbox"/>	au 400	46.589	2103	0.363



1. Open 'Phase control' window from menu *Display* → *Phase*



2. Select material by opening a jcpds file.
3. Check if  $2\theta$  angle is correct, adjust if needed.
4. Lines, which indicate positions of the peaks of the material, appear below EDXD data.



5. The positions of peaks lines can be shifted by changing 'P (GPa)', or 'T (K)'.
6. Then, click Add ROIS in 'Phase control' window to add ROIs for all peaks.

To erase a ROI, please click *Delete* after selection of the ROI. To erase all ROIs, please click *Clear All*.

## 7.7 Fluorescence markers

By typing in an element, hpMCA shows K and/or L shell emission lines positions.

## 7.8 Vertical scale

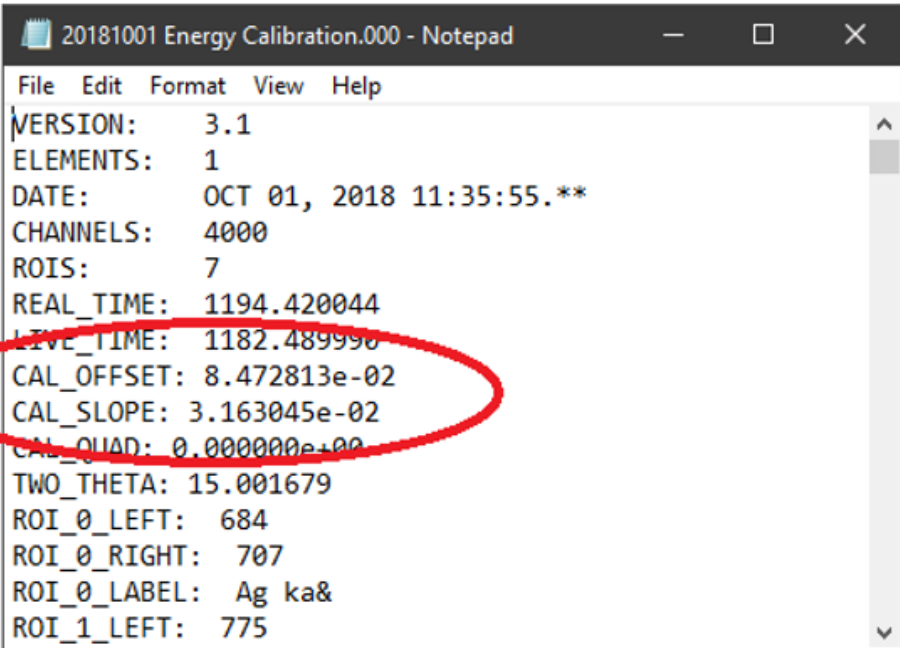
Vertical scale section

## 7.9 Horizontal scale

Horizontal scale section

## 7.10 Energy calibration

Beamline scientist does energy calibration of the germanium solid state detector by using Fluorescence lines of silver at 22.104 keV ( $K\alpha$ ) and 24.942 keV ( $K\alpha_1$ ), and gammas from  $^{109}\text{Cd}$  (88.04 keV) and  $^{57}\text{Co}$  (122.10 keV) at the beginning of each beamtime cycle. Parameters of energy calibration ( $Energy = CAL\_OFFSET + CAL\_SLOPE \cdot Channel$ ) can be found in the header of the EDXD data file.



```
20181001 Energy Calibration.000 - Notepad
File Edit Format View Help
VERSION: 3.1
ELEMENTS: 1
DATE: OCT 01, 2018 11:35:55.**
CHANNELS: 4000
ROIS: 7
REAL_TIME: 1194.420044
LIVE_TIME: 1182.489990
CAL_OFFSET: 8.472813e-02
CAL_SLOPE: 3.163045e-02
CAL_QUAD: 0.000000e+00
TWO_THETA: 15.001679
ROI_0_LEFT: 684
ROI_0_RIGHT: 707
ROI_0_LABEL: Ag ka&
ROI_1_LEFT: 775
```

## 7.11 $2\theta$ angle calibration

Beamline scientist does  $2\theta$  angle calibration at  $7^\circ$ ,  $15^\circ$ ,  $23^\circ$ , and  $31^\circ$  using unit-cell volume of Au, and make linear equation to calculate  $2\theta$  angle.

The following is the procedure for  $2\theta$  angle calibration:

- Collect Au EDXD pattern.
- *Make ROIs* for all Au peaks using JCPDS data at 0 GPa.
- Select *Control* → *Calibrate 2theta...* on Menu bar.
- Please remove weak or overlapping peaks by unchecking them in the column ‘Use?’.

---

**Note:** Because the MCA does not have background subtraction feature, background slope at low energy ( $< \sim 25$  keV) probably due to absorption influences on determining peak position. It is better not to use low energy data for  $2\theta$  angle calibration. Typically, at  $2\theta$  of  $\sim 15^\circ$ , the first and second peaks show marked deviation from other peaks.

---

- Click *Compute 2-theta*.
- $2\theta$  value appears in the ‘ $2\theta$ ’ box.
- Then, please click *OK* to apply the  $2\theta$  calibration.

---

**Note:** The  $2\theta$  calibration result is also saved in the header of the data file.

---

2θ calibration

Defined regions

ROI	Use?	Energy	D-spacing	HKL	2θ	2θ error
0	<input type="checkbox"/>	au 111	2.3490	20.155	15.04695	-0.00505
1	<input type="checkbox"/>	au 200	2.0343	23.268	15.05048	-0.00152
2	<input checked="" type="checkbox"/>	au 220	1.4385	32.904	15.05150	-0.00050
3	<input checked="" type="checkbox"/>	au 311	1.2267	38.586	15.05036	-0.00164
4	<input checked="" type="checkbox"/>	au 222	1.1745	40.289	15.05491	0.00291
5	<input checked="" type="checkbox"/>	au 400	1.0172	46.531	15.05218	0.00018
6	<input checked="" type="checkbox"/>	au 331	0.9334	50.709	15.05109	-0.00090
7	<input checked="" type="checkbox"/>	au 420	0.9098	52.021	15.05257	0.00057
8	<input checked="" type="checkbox"/>	au 422	0.8305	56.981	15.05417	0.00217
9	<input checked="" type="checkbox"/>	au 333	0.7830	60.451	15.05062	-0.00138
10	<input type="checkbox"/>	au 531	0.6877	68.817	15.05274	0.00074
11	<input type="checkbox"/>	au 442	0.6781	69.785	15.05452	0.00252
12	<input checked="" type="checkbox"/>	au 620	0.6433	73.579	15.05058	-0.00142

Compute 2θ Plot 2θ error 2θ 15.05200 +/- 0.00154

OK Cancel