RESEARCH INSTRUMENTATION STANDARD OPERATING PROCEDURE FOR EASYXAFS 300+ X-RAY SPECTROMETER (OPXAFS-1) AT NUCS DODGEN

REVISION 0.2, 8/30/2022

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OPERATING PROCEDURE OPXAFS-1: OPERATION OF THE EASYXAFS 300+ X-RAY SPECTROMETER

1 Background

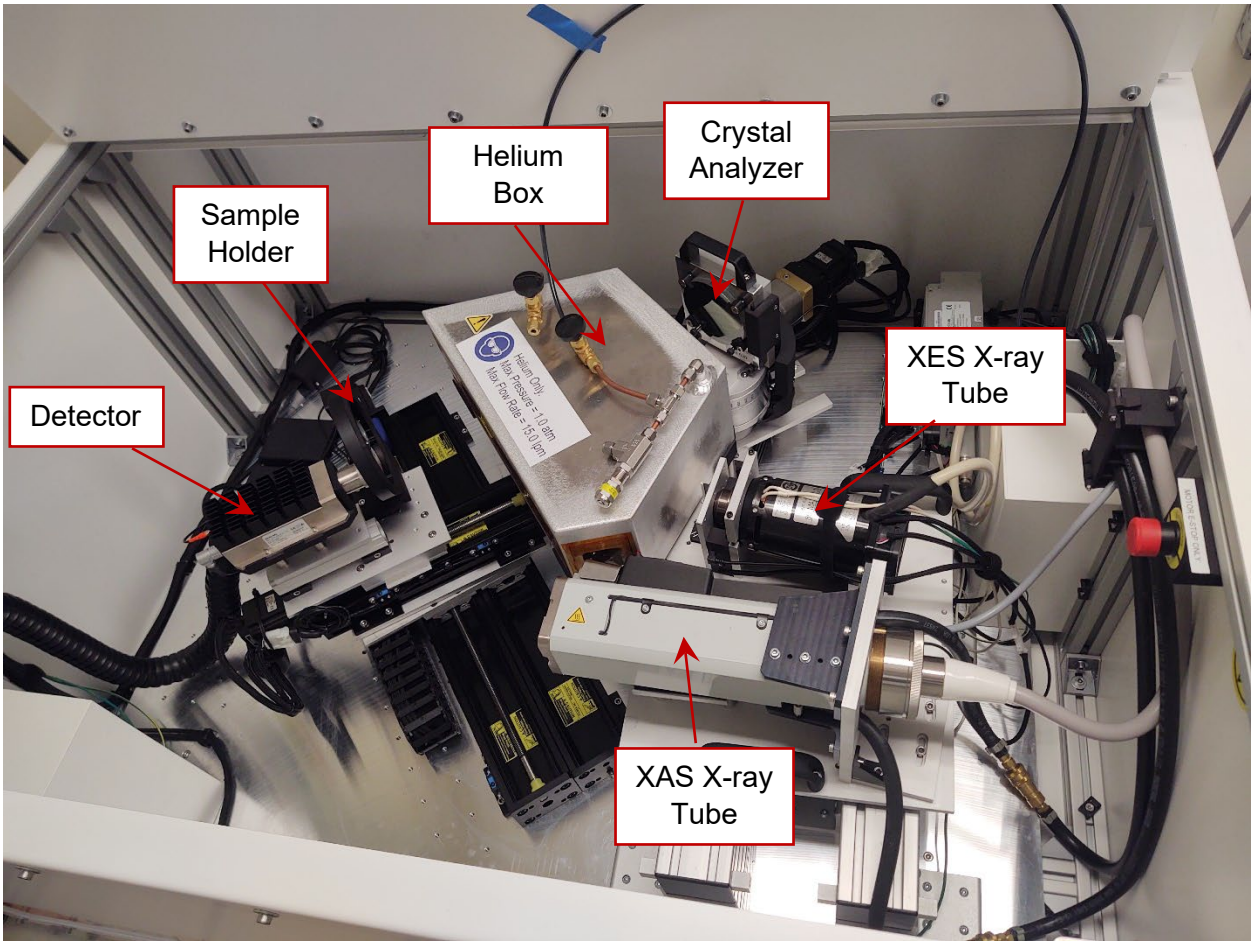
X-ray absorption fine structure (XAFS) refers to the details of how X-rays are absorbed by an atom at energies near and above the core-level binding energies of that atom. XAFS spectra are especially sensitive to the formal oxidation state, coordination chemistry, and the distances, coordination number and species of the atoms immediately surrounding the selected element. Because of this dependence, XAFS spectra provides a way to determine the chemical state and local atomic structure for a selected atomic species. XAFS is routinely used in a wide range of scientific fields, including biology, environmental science, catalysts research, and material science. XAFS data often complements other measures of structure such as microscopy and diffraction.

Since XAFS is an atomic probe, it places few constraints on the samples that can be studied. All atoms have core level electrons, and XAFS spectra can be measured for every element on the periodic table. Importantly, crystallinity is not required for XAFS measurements, making it one of the few structural probes available for noncrystalline and highly disordered materials, crystals, amorphous or glassy materials, macromolecules, surface sorbed species, and liquids. Because X-rays are fairly penetrating in matter, XAFS is not inherently surface-sensitive, though special measurement techniques can be applied to enhance its surface sensitivity. In many cases, XAFS measurements can be made on elements of minority and even trace abundance, giving a unique and direct measurement of chemical and physical state of dilute species in a variety of systems.

The XAS spectra are usually divided in two energy regions: 1) X-ray absorption near-edge spectroscopy (XANES) energy region which extends between the edge region and over a 50-100 eV energy range around the edge and 2) the extended X-ray absorption fine-structure (EXAFS) region which extends for hundreds of electron volts past the edge. XANES is strongly sensitive to formal oxidation state and coordination chemistry (e.g., octahedral, tetrahedral coordination) of the absorbing atom, while the EXAFS is used to determine the distances, coordination number, and species of the neighbors of the absorbing atom.

X-ray emission spectroscopy (XES) is a form of X-ray spectroscopy where core electrons are excited out of their shell and data is collected on the emitted photons from the return of these electrons from the excited states to their core state. XES is element-specific and site-specific, making it a powerful tool for determining detailed electronic properties of materials. XES measurements often investigate K_{β}-lines, which measurements of valence-to-core energy transitions.

A laboratory XAFS instrument contains a 1200 W X-ray absorption X-ray tube (either Mo or Ag X-ray tube is available), a 100 W X-ray emission tube (either W or Pd X-ray tube is available), a crystal analyzer (Ge 620, Ge 211, Si 100, Si 111, and Si 211 are available), a helium box to minimize the loss of X-rays as they travel to the sample and detector, a sample holder (a single holder or a sample changer with seven spots is available), and a detector.

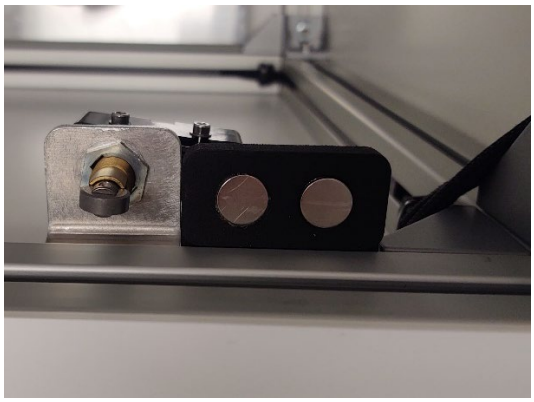


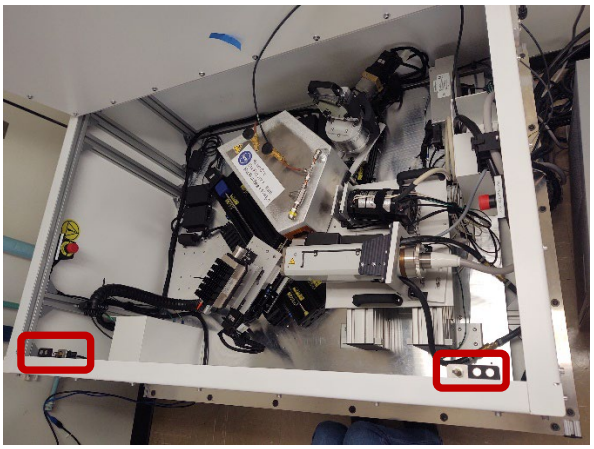
1.1 **Monthly Calibrations/QA/QC Checks**

Monthly calibrations are undertaken to verify the functionality of the instrument and to head off any problems or safety concerns before they become larger problems that could result in instrument failure. A calibration standard (iron foil) is used in the monthly calibration (see Section 3).

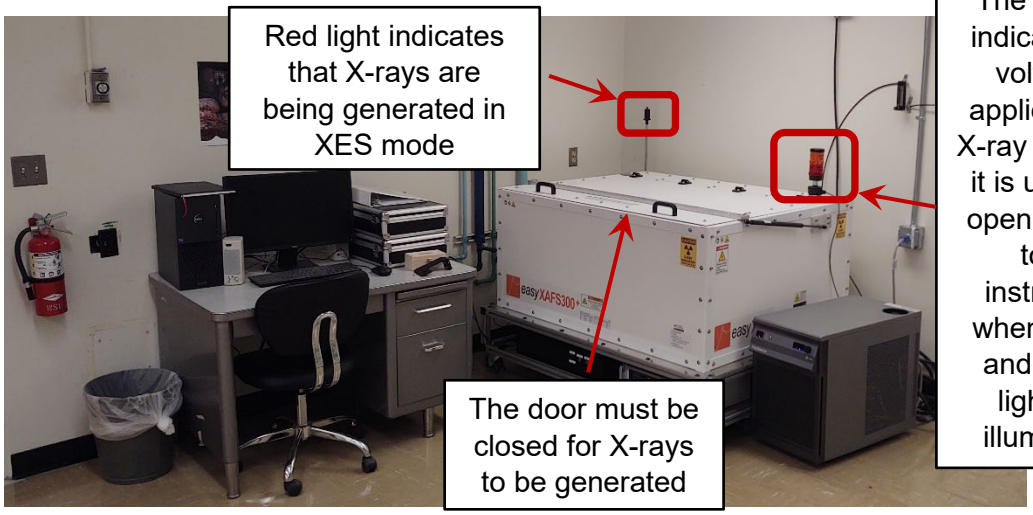
2 Safety Requirements

X-ray Absorption Fine Structure (XAFS) is a specific structure observed in X-ray absorption spectroscopy (XAS). This scientific technique utilizes X-rays to obtain geometry and local structural information on samples. X-rays are ionizing radiation and X-ray photons carry enough energy to ionize atoms and disrupt molecular bonds, which makes X-rays harmful to living tissue. A very high radiation dose over a short period of time will cause radiation sickness, while lower doses will give an increased risk of radiation-induced cancer. The easyXAFS 300+ contains radiation protective interlocks (the main door needs to be closed for operation) to prevent a user from being exposed to the X-ray radiation during the use of the X-ray spectrometer.

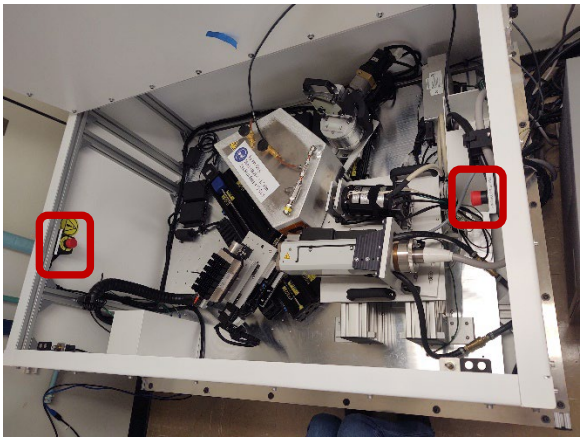




Opening the door to the instrument during operation will result in the X-ray tube being turned off. The small red light in the back (left rear) of the instrument (XES mode) or the large red light on the right rear of the instrument (XAS mode) is illuminated when voltage is applied to the X-ray tube and X-rays are being generated.



If something gets caught in the motors of the instrument, the instrument has two motor emergency stops, one on each end of the instrument (highlighted in red).



All users are required to provide the NUCS Core Facility Staff with records of completion of the WSU Radiation Safety Office Training Courses #1-7 & 10 (https://rso.wsu.edu/wsu-radiation-safety-training/), *prior* to being trained on the use of the easyXAFS 300+ X-ray spectrometer.

3 Calibration Procedure

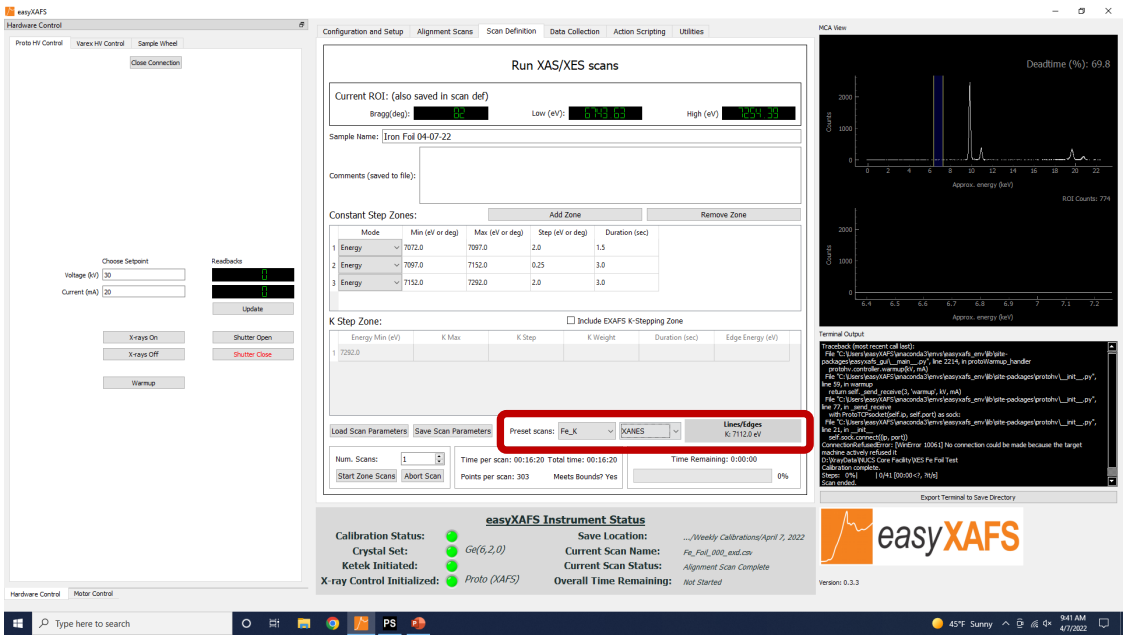
Materials: 7.5 µm Iron Foil, Ge 620 spherically-bent Bragg crystal analyzer (SBA), easyXAFS 300+, Mo or Ag XAFS X-ray tube

3.1 Turn on the instrument and calibrate the instrument according to section 6 (Sections 6.1 to 6.41).

3.2 During the calibrations, set the Directory (Steps 6.22 & 6.23) to D:\XrayData\NUCS Lab\Year\MonthlyCalibrations

3.3 Place the iron foil in the single sample holder as seen in Figure 6.49.

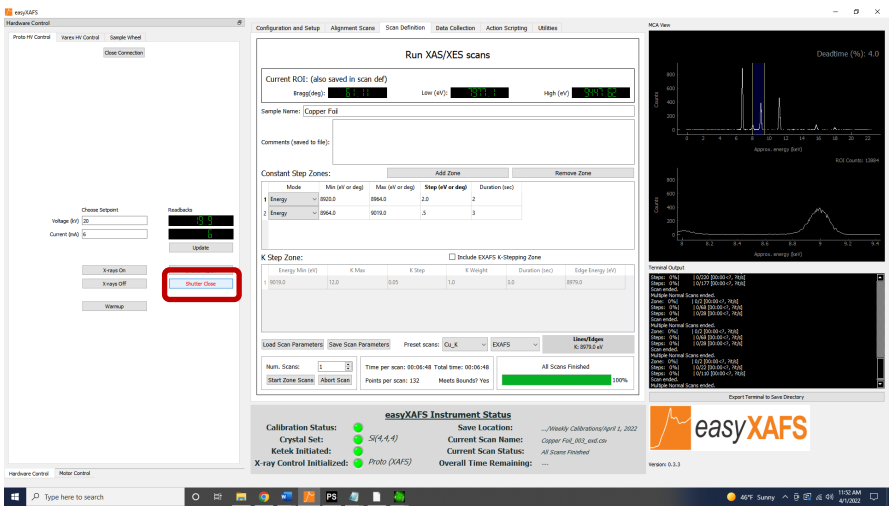
3.4 The Kα energy for Fe is 7112 eV. Set the Preset scans to Fe_K and XANES mode (highlighted in red).



3.5 Enter the Sample Name as: Iron Foil Calibration MM-DD-YYYY, where MM is the month, DD is the day of the month, and YYYY is the year.

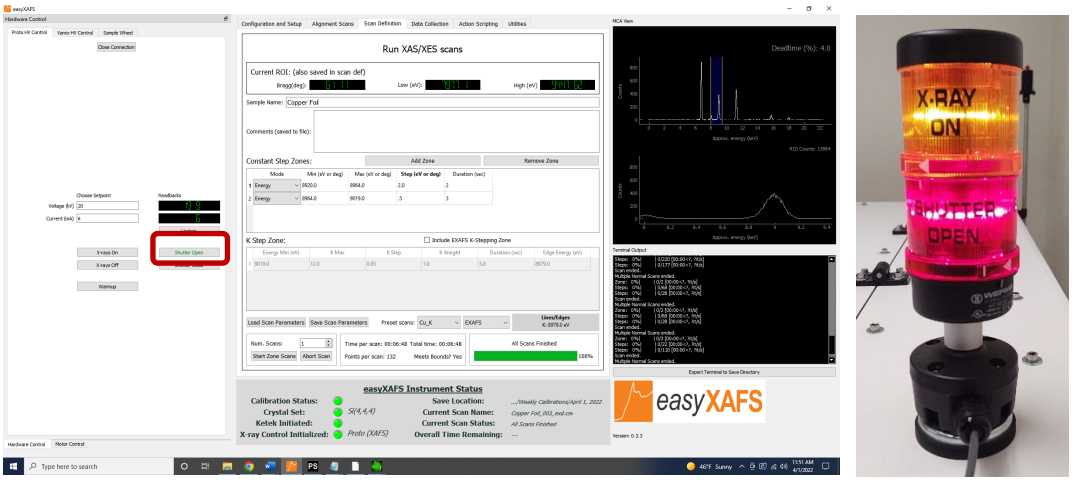
3.6 Click on the Start Zone Scans to begin the experiment. The experiment will take 16 minutes and a completed scan will look similar to the picture below.

3.7 After the data collection has completed, click on the Power Supply window and click on the Close Shutter button to close the shutter. The Shutter Open light should not be illuminated.

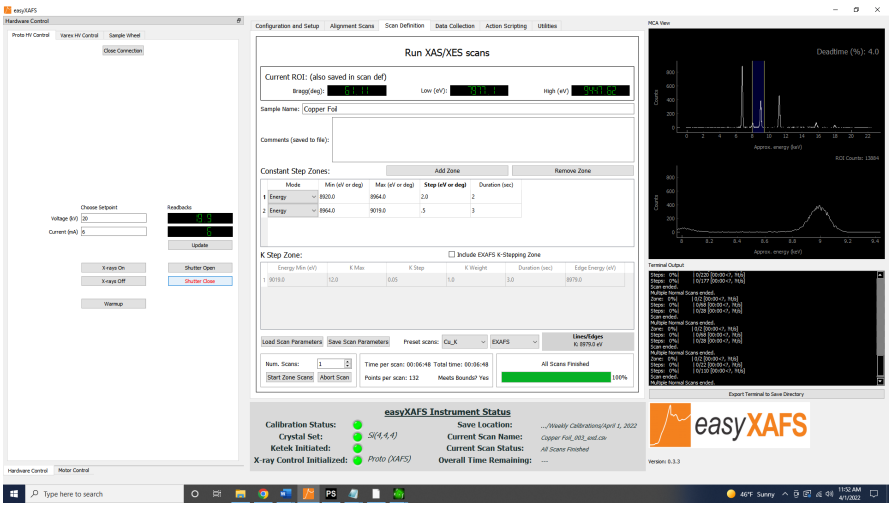


3.8 Remove the single sample holder and close the door to the easyXAFS.

3.9 Click on the Power Supply window and click on the Open Shutter button. The X-ray light and the shutter open light should now be on.



3.10 Change the Sample Name to: Iron Foil Izero Calibration MM-DD-YYYY, where MM is the month, DD is the day of the month, and YYYY is the year.



3.11 Collect data on an empty instrument to collect an I_0. The experiment will take 16 minutes.

3.12 After the data collection has completed, switch to XES following Steps 7.1 – 7.10.

3.13 Since the instrument is already on, skip to Step 7.21 to turn on the XES X-ray tube.

3.14 Complete Steps 7.21 – 7.34.

3.15 Go to Step 7.36 and enter a filename of Fe Foil XES Theta Scan MM-DD-YYYY, where MM is the month, DD is the day of the month, and YYYY is the year. Before pressing the Run Scan button, enter the values of -0.5 for Theta Offset Min (deg), 0.0 for Theta Offset Max (deg), and 0.10 Step Size (deg). The Theta scan will take 5-10 minutes.

3.16 Complete Steps 7.37 – 7.47.

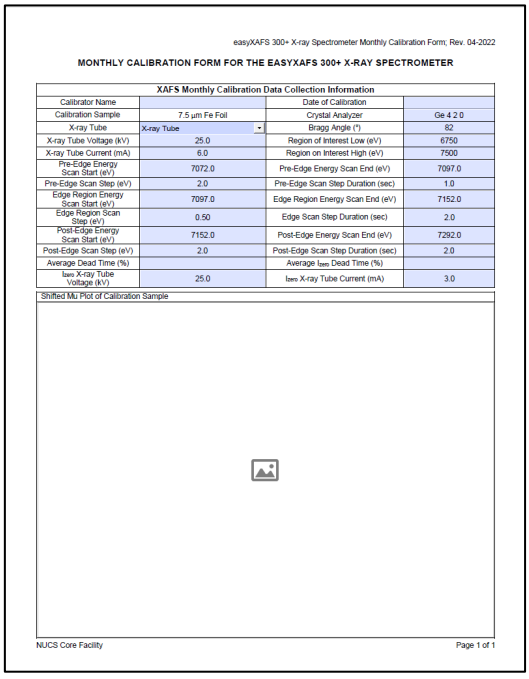
3.17 When the data collection has been completed, the instrument can be turned off as indicated in Section 9.

3.18 While you are waiting for the X-ray tube to cool down, a calibration report as indicated in Section 4 can be completed.

4 Reporting Weekly Calibration

4.1 Collect XAFS and XES data on the iron foil reference sample as indicated in Section 3.

4.2 A blank report for monthly calibrations can be found on the Research drive at the following file location: Z:\ easyXAFS\Weeky Calibration Data\



easyXAFS 300+ X-ray Spectrometer Monthly Calibration Form; Rev. 04-2022

MONTHLY CALIBRATION FORM FOR THE EASYXAFS 300+ X-RAY SPECTROMETER

XAFS Monthly Calibration Data Collection Information			
Calibrator Name		Date of Calibration	
Calibration Sample	7.5 µm Fe Foil	Crystal Analyzer	Ge 4 2 0
X-ray Tube	X-ray Tube	Bragg Angle (°)	82
X-ray Tube Voltage (kV)	25.0	Region of Interest Low (eV)	6750
X-ray Tube Current (mA)	6.0	Region on Interest High (eV)	7500
Pre-Edge Energy Scan Start (eV)	7072.0	Pre-Edge Energy Scan End (eV)	7097.0
Pre-Edge Scan Step (eV)	2.0	Pre-Edge Scan Step Duration (sec)	1.0
Edge Region Energy Scan Start (eV)	7097.0	Edge Region Energy Scan End (eV)	7152.0
Edge Region Scan Step (eV)	0.50	Edge Scan Step Duration (sec)	2.0
Post-Edge Energy Scan Start (eV)	7152.0	Post-Edge Energy Scan End (eV)	7292.0
Post-Edge Scan Step (eV)	2.0	Post-Edge Scan Step Duration (sec)	2.0
Average Dead Time (%)		Average I_{zero} Dead Time (%)	
I_{zero} X-ray Tube Voltage (kV)	25.0	I_{zero} X-ray Tube Current (mA)	3.0

Shifted Mu Plot of Calibration Sample

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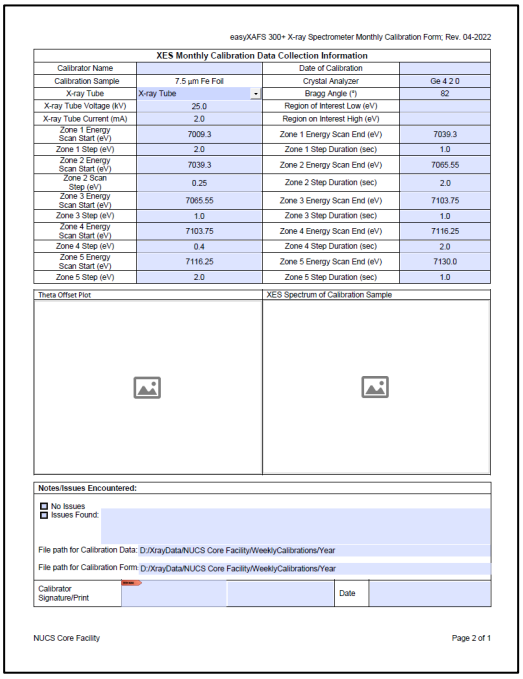

easyXAFS 300+ X-ray Spectrometer Monthly Calibration Form; Rev. 04-2022

XES Monthly Calibration Data Collection Information			
Calibrator Name		Date of Calibration	
Calibration Sample	7.5 µm Fe Foil	Crystal Analyzer	Ge 4 2 0
X-ray Tube	X-ray Tube	Bragg Angle (°)	82
X-ray Tube Voltage (kV)	25.0	Region of Interest Low (eV)	
X-ray Tube Current (mA)	2.0	Region on Interest High (eV)	
Zone 1 Energy Scan Start (eV)	7009.3	Zone 1 Energy Scan End (eV)	7039.3
Zone 1 Step (eV)	2.0	Zone 1 Step Duration (sec)	1.0
Zone 2 Energy Scan Start (eV)	7039.3	Zone 2 Energy Scan End (eV)	7065.55
Zone 2 Scan Step (eV)	0.25	Zone 2 Step Duration (sec)	2.0
Zone 3 Energy Scan Start (eV)	7065.55	Zone 3 Energy Scan End (eV)	7103.75
Zone 3 Step (eV)	1.0	Zone 3 Step Duration (sec)	1.0
Zone 4 Energy Scan Start (eV)	7103.75	Zone 4 Energy Scan End (eV)	7116.25
Zone 4 Step (eV)	0.4	Zone 4 Step Duration (sec)	2.0
Zone 5 Energy Scan Start (eV)	7116.25	Zone 5 Energy Scan End (eV)	7130.0
Zone 5 Step (eV)	2.0	Zone 5 Step Duration (sec)	1.0

Theta Offset Plot | XES Spectrum of Calibration Sample

Notes/Issues Encountered:

☐ No Issues
☐ Issues Found:

File path for Calibration Data: D:/XrayData/NUCS Core Facility/WeeklyCalibrations/Year

File path for Calibration Form: D:/XrayData/NUCS Core Facility/WeeklyCalibrations/Year

Calibrator Signature/Print | Date

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4.3 Enter the name of the NUCS Core Facility staff that collected the data and date of the data collection. Also, include the instrument parameters on the Monthly Calibration Form.

4.4 Use the easyXAFS software to analyze the collected data to generate a mu plot for XAS data and the XES spectrum which are added to the Monthly Calibration Form (see Section 8).

4.5 Please save the data and the report on the NUCS drive using the following file location: Z:\easyXAFS\Monthly Calibration Data\Year\Month and place in the respective year and folder for the data and report. Please ask the NUCS Core Facility staff if you have any questions.

5 Sample Preparation

5.1 Prepare 100-200 mg of sample.

5.2 Use a mortar and pestle to finely grind the solid.

5.3 Place the ground solid on a flat surface (e.g. clean tabletop or a glass slide).

5.4 Dab the area covered with solid with a piece of Scotch tape (~ 2-3” in length) to obtain an even layer of solid.

5.5 Brush off any excess solid with a paintbrush.

5.6 Repeat Steps 5.3 & 5.4 until have 5-10 pieces of Scotch tape with sample adhered to it.

5.7 Layer 5 pieces of tape (containing sample) on top of each other and tape together.

5.8 Trim the tape layers to have the sample about 1-2” in length, until the sample fits into the holder.

5.9 Tape the layered tape with sample to the sample holder and analyze with the easyXAFS.

5.10 If the sample is not concentrated enough, add more layers of tape.

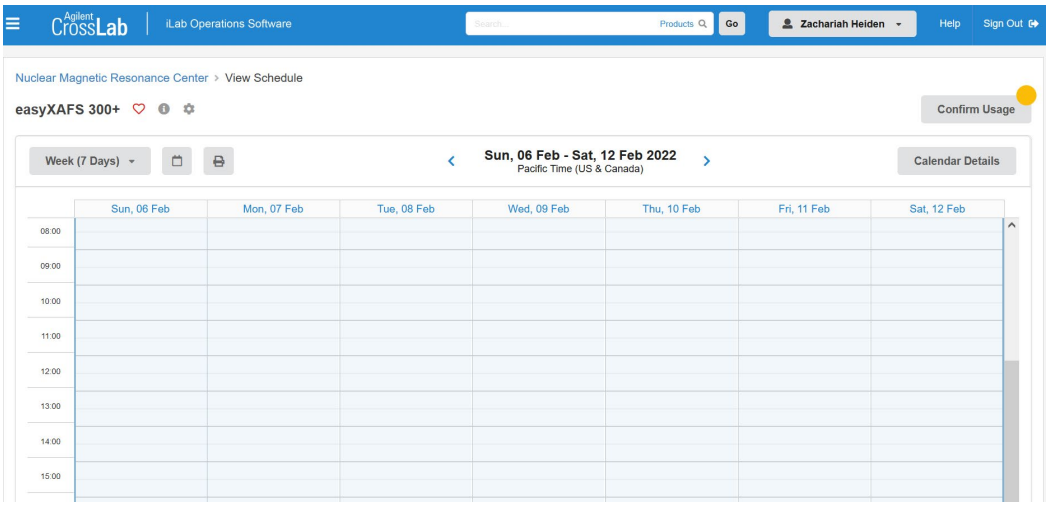
5.11 If the sample is too concentrated, remove some layers of tape.

6 XAFS Data Collection on the easyXAFS 300+ X-ray Spectrometer

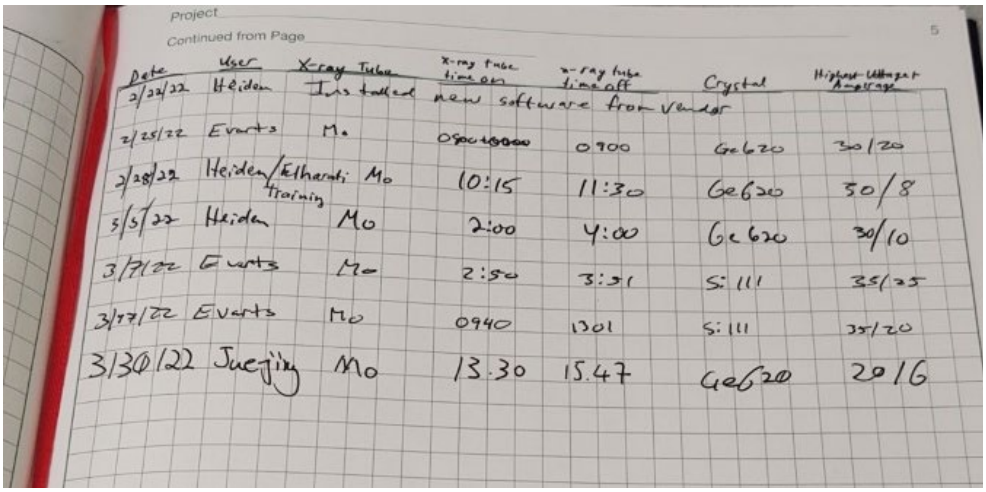
This section is to be completed after a weekly calibration (Sections 3 & 4) has been performed.

Materials: X-ray Sample, easyXAFS 300+ X-ray Spectrometer, Mo or Ag X-ray tube

6.1 Instrument time on the easyXAFS 300+ X-ray spectrometer are made on iLab (https://wsu.corefacilities.org/), at least one hour prior to the user's instrument time. Reservations are based on instrument time and are reserved in 30 minute increments, with a minimum reservation time of one hour. Please reserve the desired instrument time on iLab. If you have questions regarding booking instrument time, please ask the NUCS Core Facility Staff.



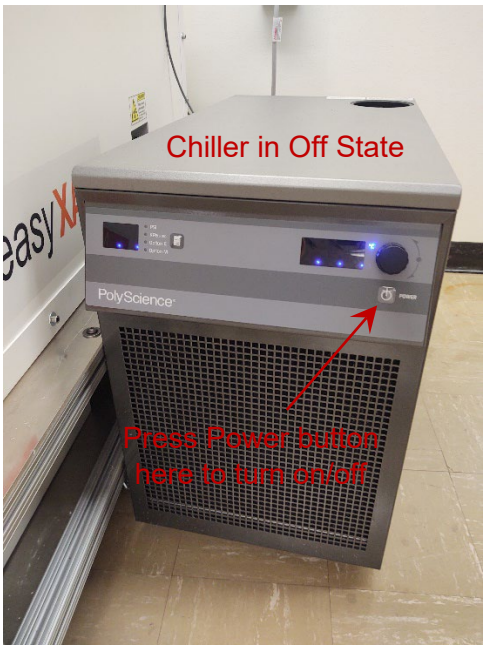
6.2 Start by writing the date, starting time, and your name in the instrument logbook.



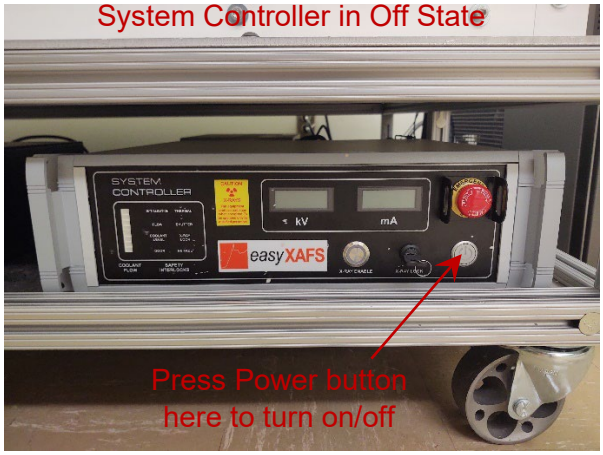
6.3 If the computer screen requires a password, the password is changed every two weeks and it is either easyXAFS or easyXAF$

6.4 Then, check to see if the easyXAFS 300+ X-ray spectrometer is on. The default state is to have the system powered down.

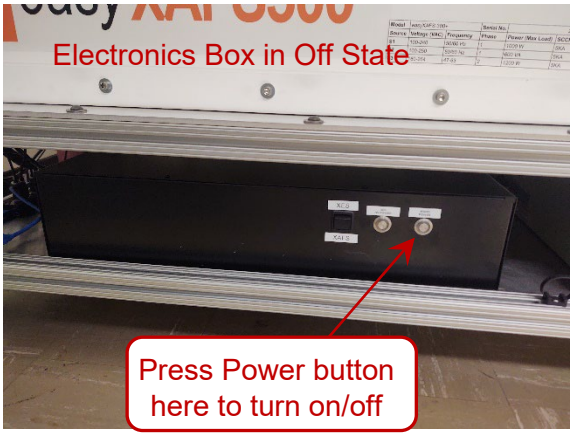
6.5 To start working with the easyXAFS 300+, you will want to start by turning on the chiller for the X-ray tube. This is done by pushing the on button on the chiller. This should be running for a few seconds before starting up the X-ray tube.



6.6 Press the power button on the system controller to turn on.

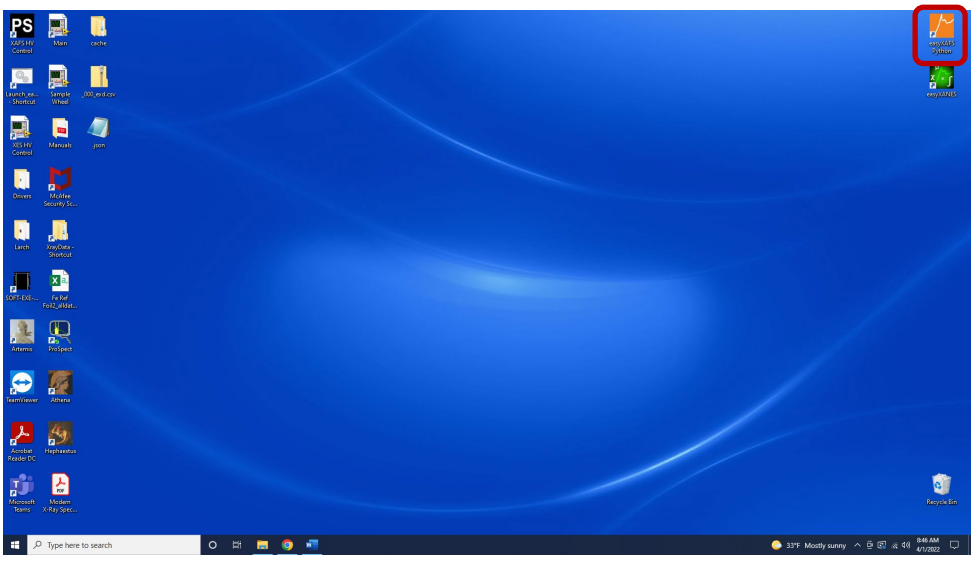


6.7 Power on the electronics box by pushing the Mono power button.

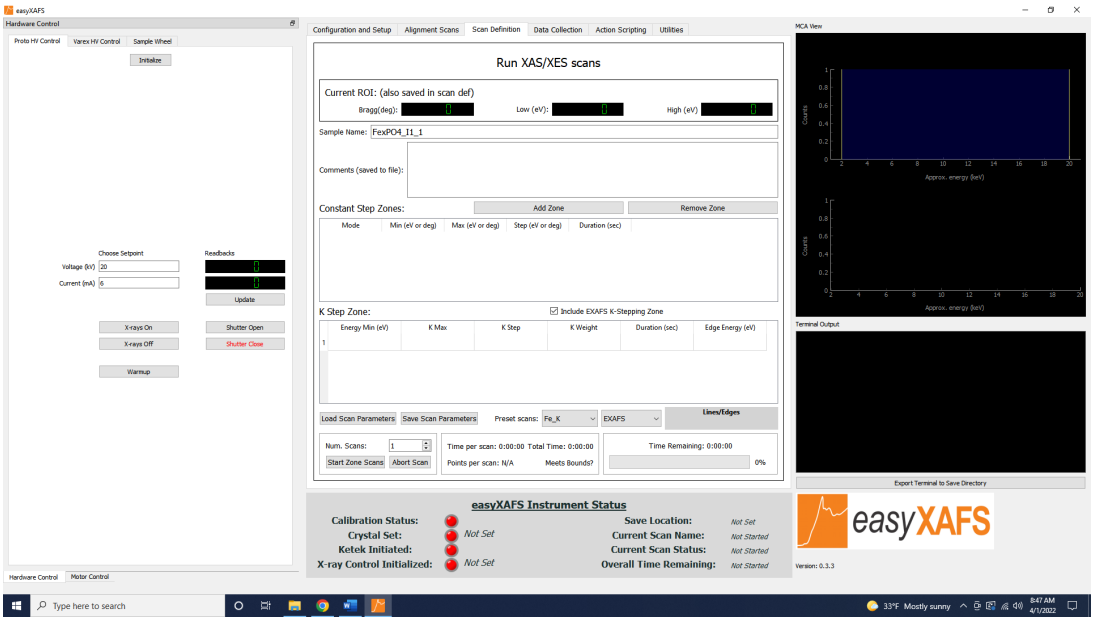


6.8 Close the door to the easyXAFS, if it is not already closed.

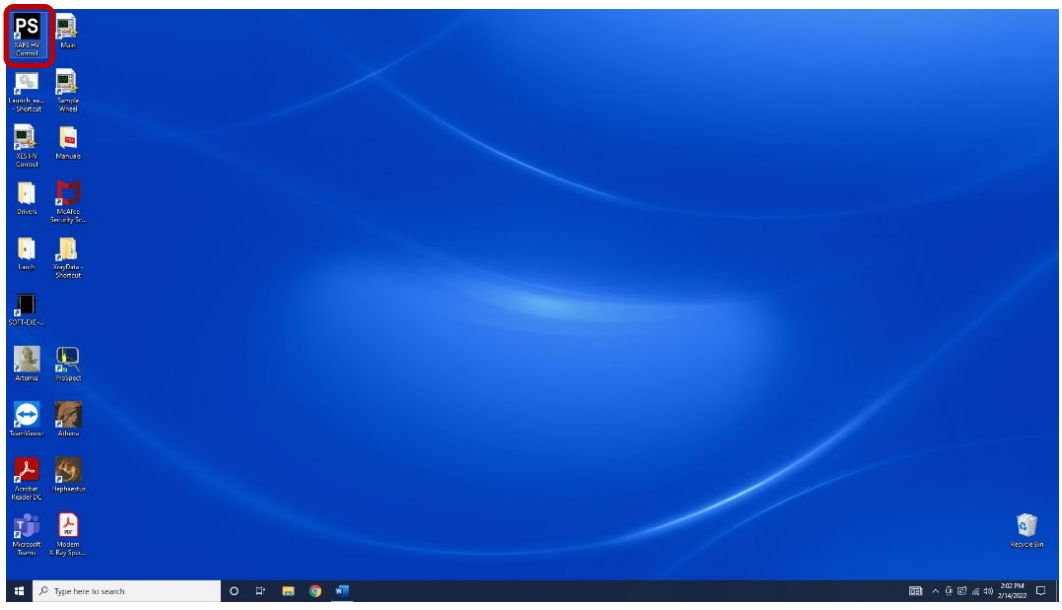
6.9 After the power has been turned on to the electronics box and the system controller, the software to control the instrument can be opened. To open the software, click on the easyXAFS Python icon in the top right of the computer screen.



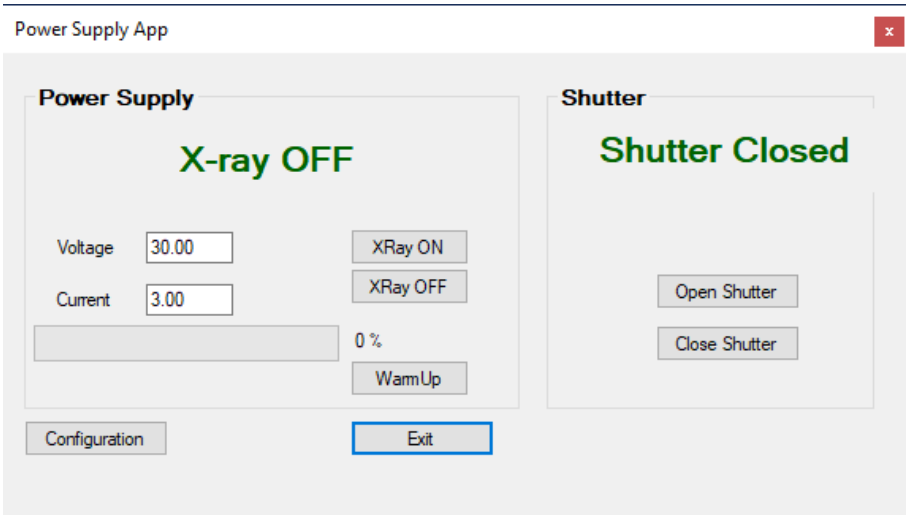
6.10 When the software opens, it will look like the following picture.



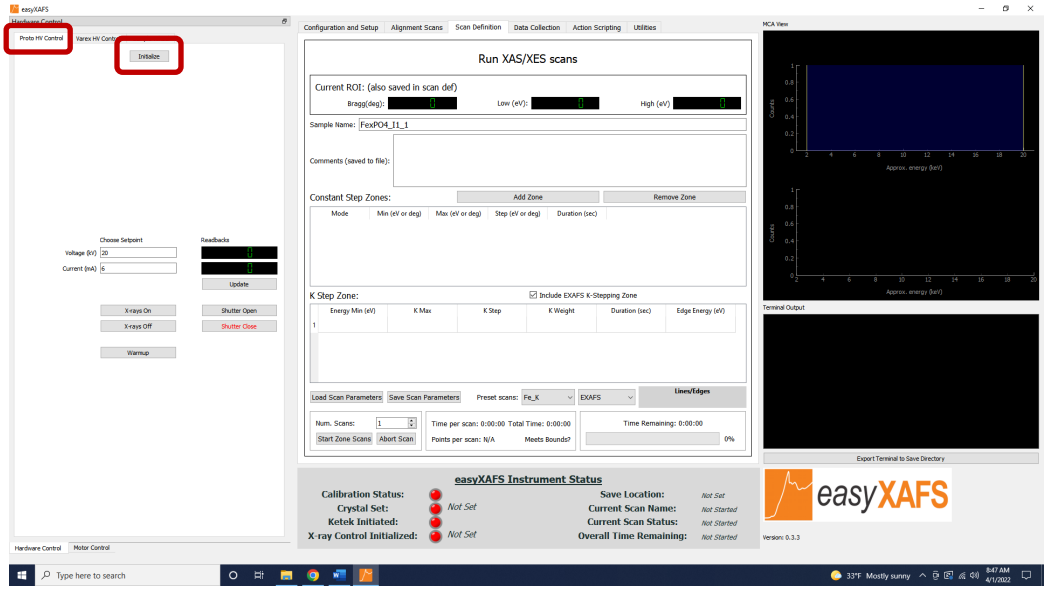
6.11 After the easyXAFS software opens, it will need to be minimized and the XAFS HV Control icon in the top left of the computer screen will need to be opened. Double clicking on this icon will open the Power Supply control, which needs to run in the background of the easyXAFS software. The XAFS HV Control needs to be opened after the easyXAFS software, otherwise an error message will result.



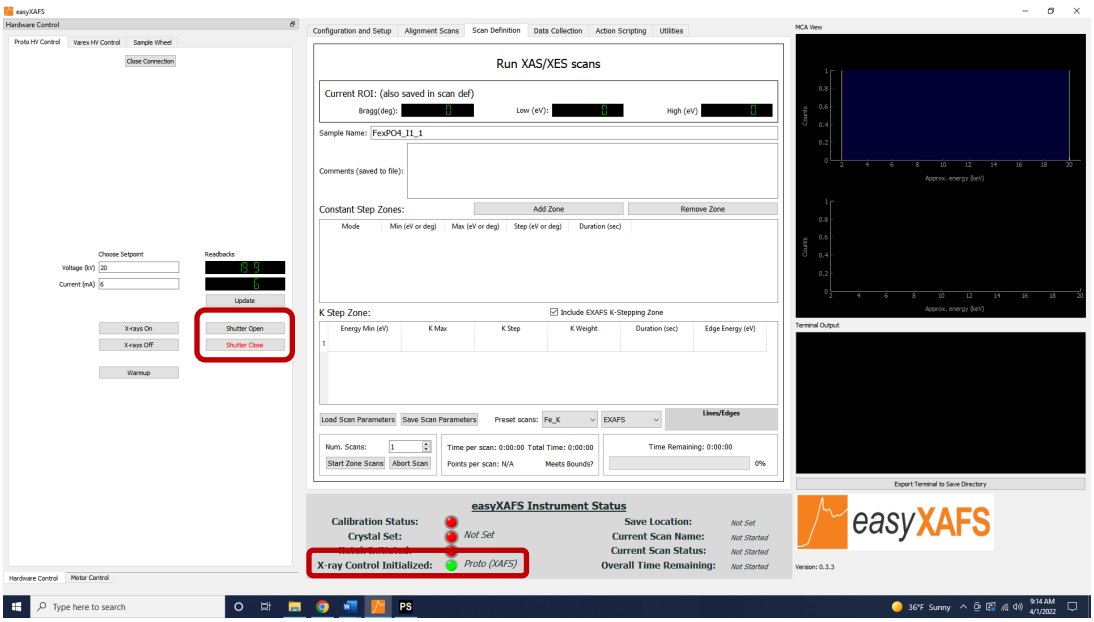
6.12 When the Power Supply control opens, the following window will appear. This program just needs to run in the background for XAFS measurements and will not be used with the new software.



6.13 After the Power Supply App opens, the easyXAFS software can be maximized and the inititalize button (highligted in red), under the ProtoHV Control tab, is clicked to connect to the XAS X-ray tube.



6.14 When the easyXAFS software properly conencts to the XAS X-ray tube, the shutter to the XAFS X-ray tube will need to be opened (click Shutter open button) and closed (click Shutter Close button) to initialize the system. The X-ray Control Initialized light will become green (highlighted in red).

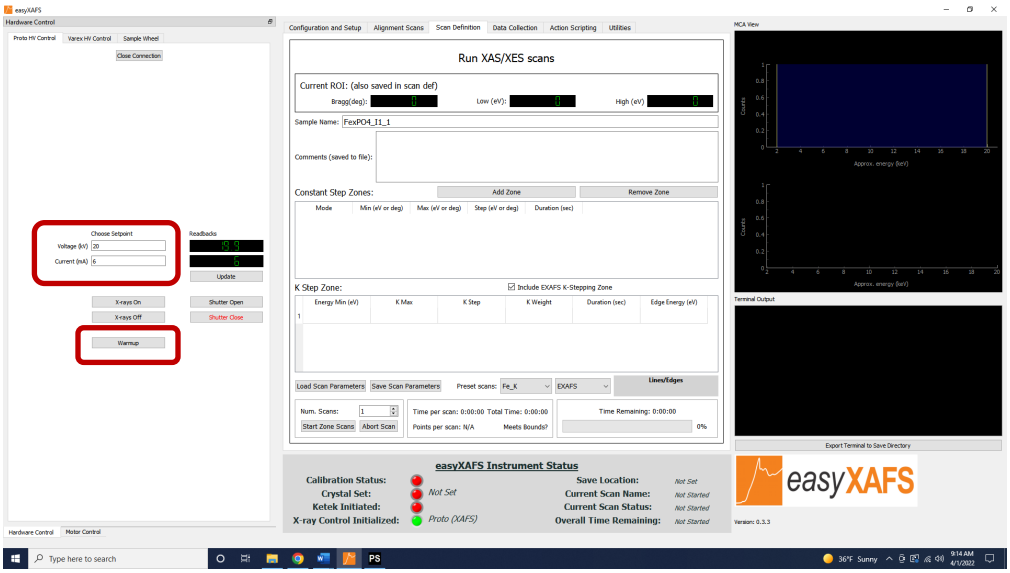


6.15 Before turning on the X-rays, we need to check to see that all of the lights on the system controller are off in the Safety Interlocks box. The vertical bar indicates the amount of coolant flow on the X-ray tube and should have four-five bars illuminated. If one of the safety interlocks is illuminated, there is a problem that needs to be dealt with prior to turning on the X-rays. Please alert NUCS Core Facility staff if you need help in diagnosing a problem.

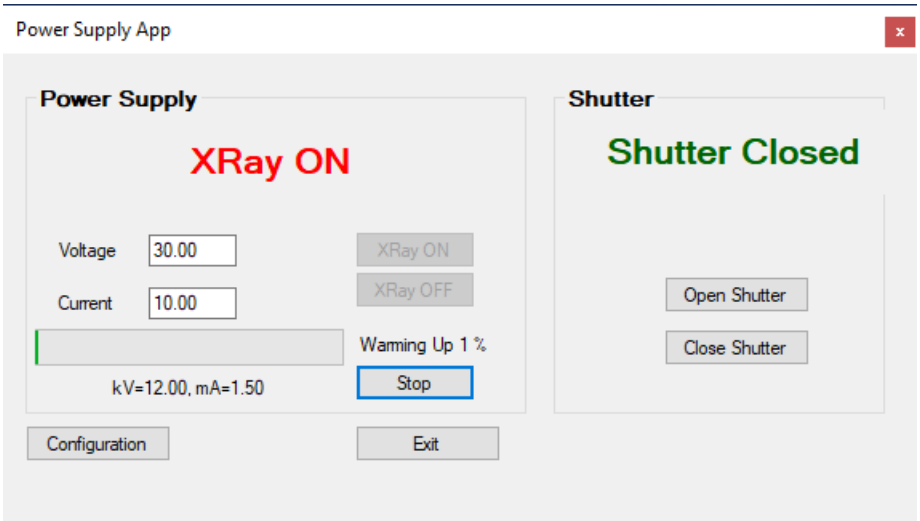
6.16 Then push the X-ray Enable button on the system controller to enable X-rays.

6.17 Once the X-rays are enabled, the X-ray tube will need to be warmed up. Move back to the computer and use the easyXAFS software to warm up the X-ray tube.

6.18 To warm up the X-ray tube select a Voltage and Current for the X-ray tube to be warmed up to. You usually want to pick the highest values you will need to measure your samples. For most cases a Voltage of 30 kV and a Current of 20 mA (setting of 600 Watts), will be fine. The maximum settings for this instrument is a Voltage of 40 kV and a Current of 30 mA (setting of 1200 Watts). After the desired voltage and current are entered click on the Warmup button. The X-ray light should turn on and the values on the system controller should be ramping up.

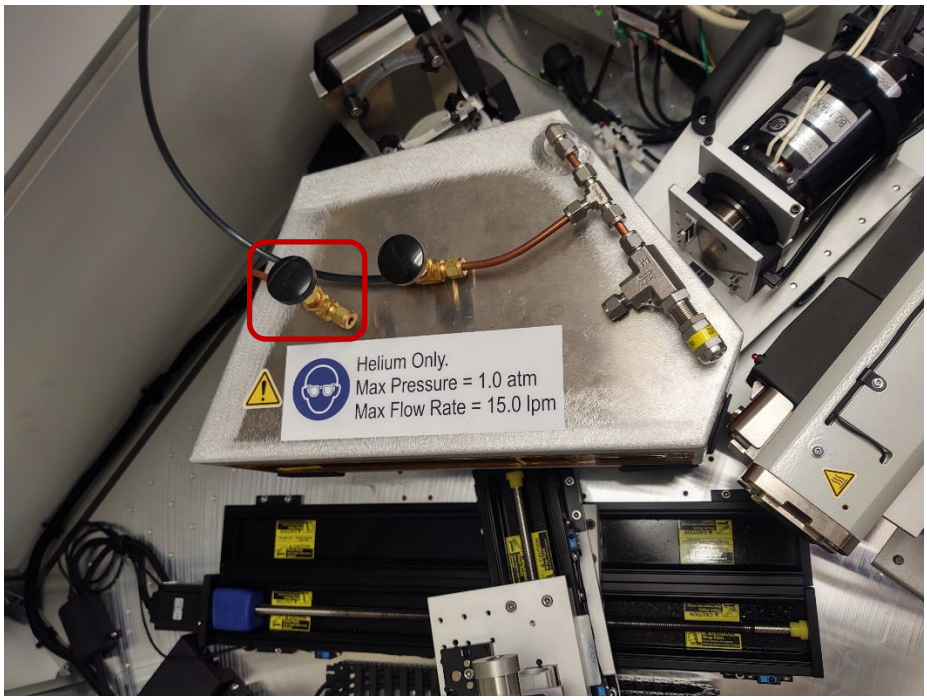


6.19 The X-ray tube will begin to warm up and will take about 15 minutes to warm up. You can monitor the progress by looking at the Power Supply App or clicking on the Update button on the Proto HV Control of the easyXAFS software.

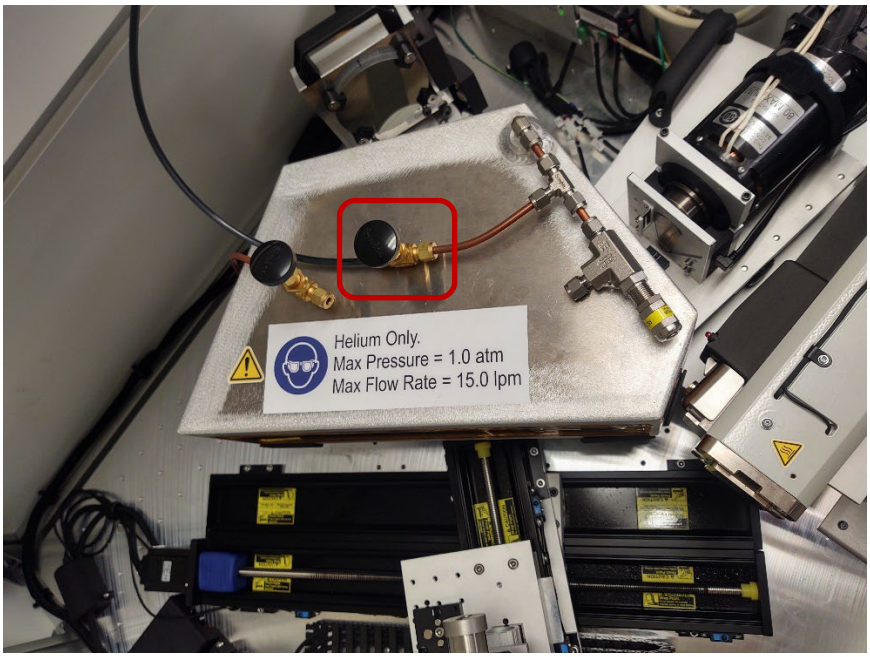


6.20 While the X-ray tube is warming up, the instrument can be filled with helium gas. The X-ray beam can be weakened by traveling through air, so instrument is filled with helium to provide the most intense X-ray beam possible. The helium box should be filled during the weekly calibration of the instrument by NUCS Core Facility staff and is only necessary if it has been more than a week since the helium box was last filled.

6.21 Open the door to the instrument and open the valve on the helium box (highlighted in red) by turning it counterclockwise about a full turn. The vent valve needs to be opened **first** to avoid over pressurizing the helium box.

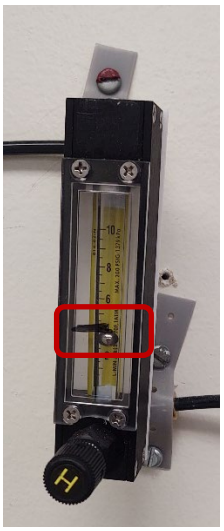


6.22 After opening the vent valve, slightly open the valve (highlighted in red) connected to the helium line by turning counterclockwise. You will hear a sound of the Kapton windows expanding as the box fills with helium.



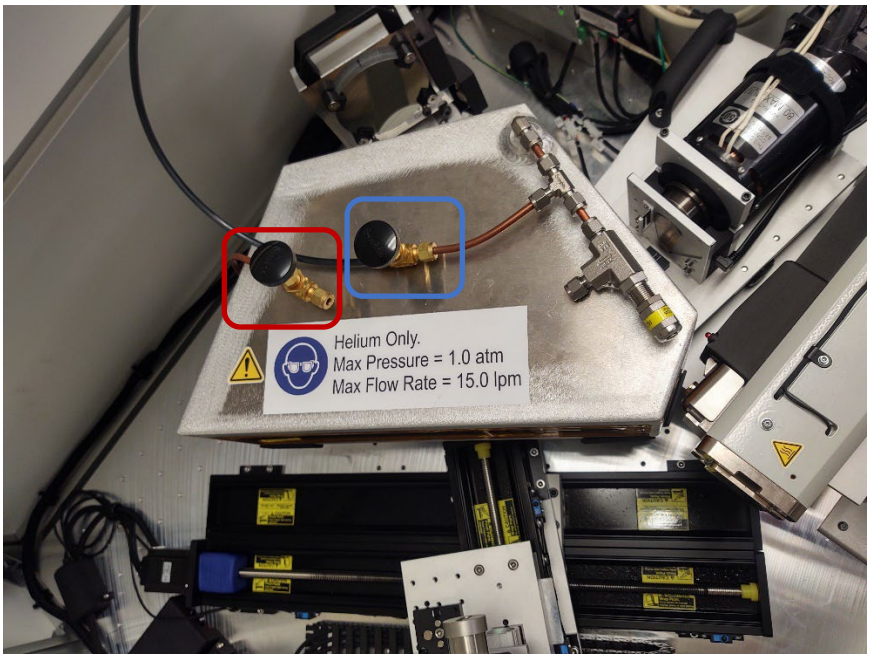
6.23 Open the helium tank by turning the tank valve (highlighted in red) counterclockwise until it stops, then turning it clockwise one-half turn. When the valve is open, the pressure inside the tank should read greater then 500 psi. If the pressure is 500 psi or below, please inform NUCS Core Facility staff.

6.24 Check the flow rate on the wall, the center of the float should be at (or below) the black mark on the flow meter to not exceed the max flow rate of 15 liters/minute. Adjustment of the flowmeter should only be completed by NUCS Core Facility staff.



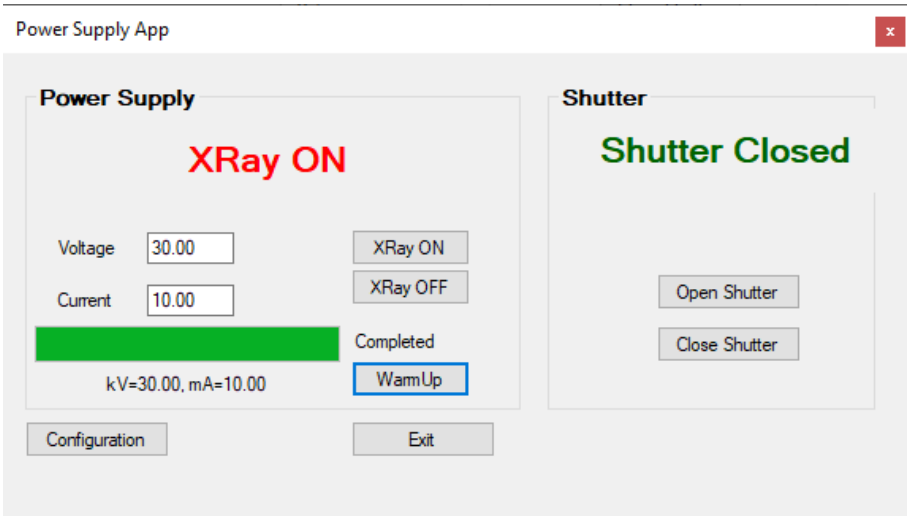
6.25 Let the helium box purge for 5-10 minutes.

6.26 After 5-10 minutes, close both valves, turning them clockwise, simultaneously to avoid a pressure buildup. If in doubt, close the valve to the helium line (highlighted in blue), immediately followed by vent valve (highlighted in red).

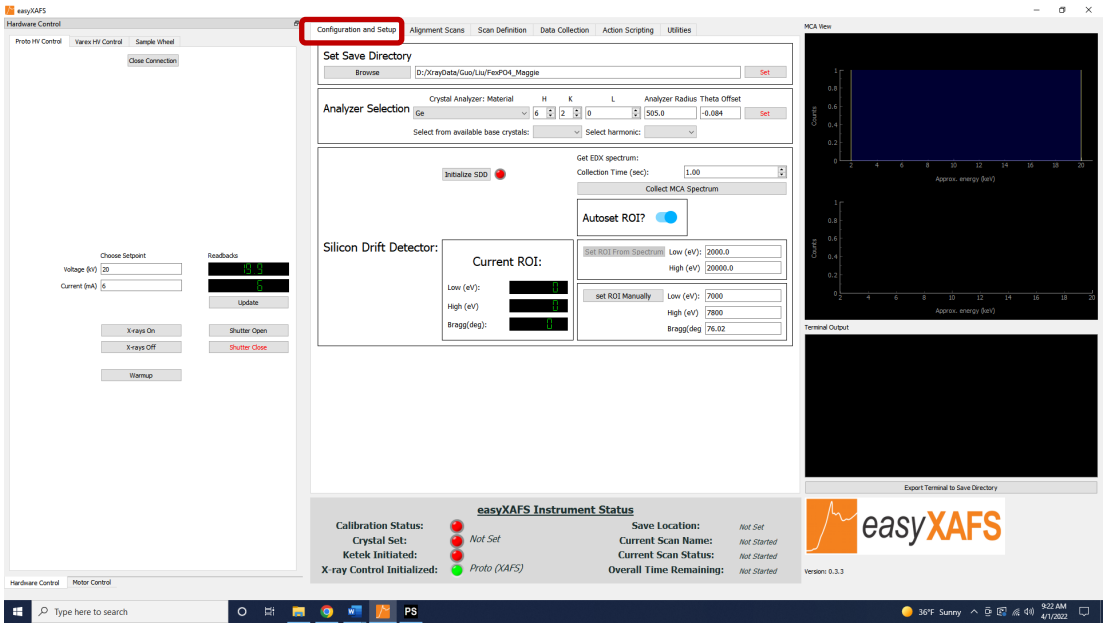


6.27 Once the helium box has been filled, close off the helium tank by turning the tank valve (highlighted in red) clockwise until it stops.

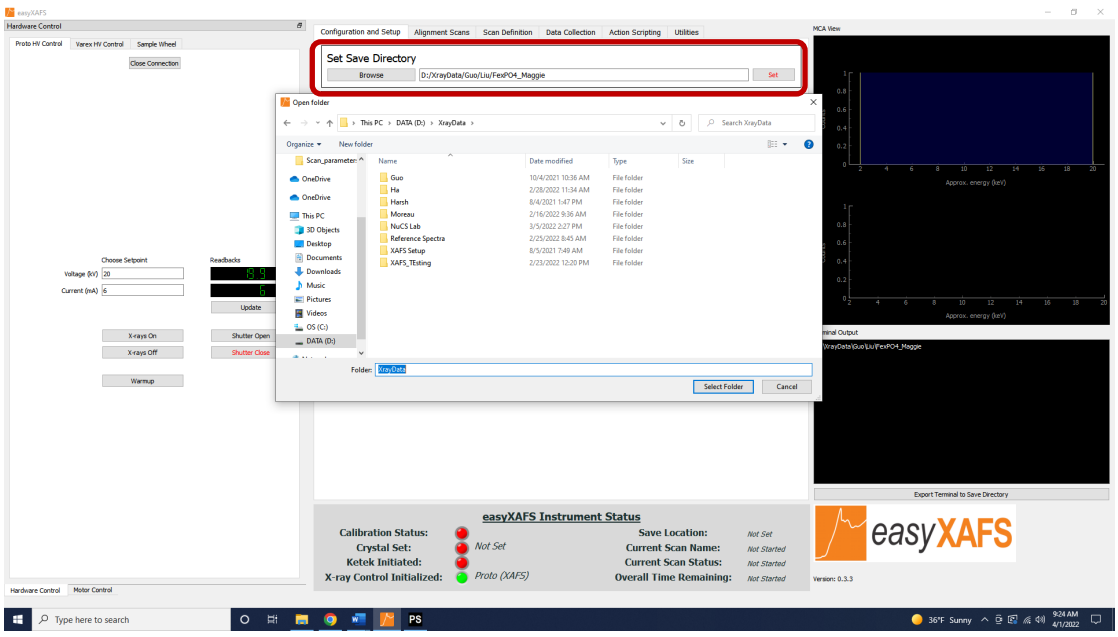
6.28 When the X-ray tube has warmed up the Power Supply App will look like the following:



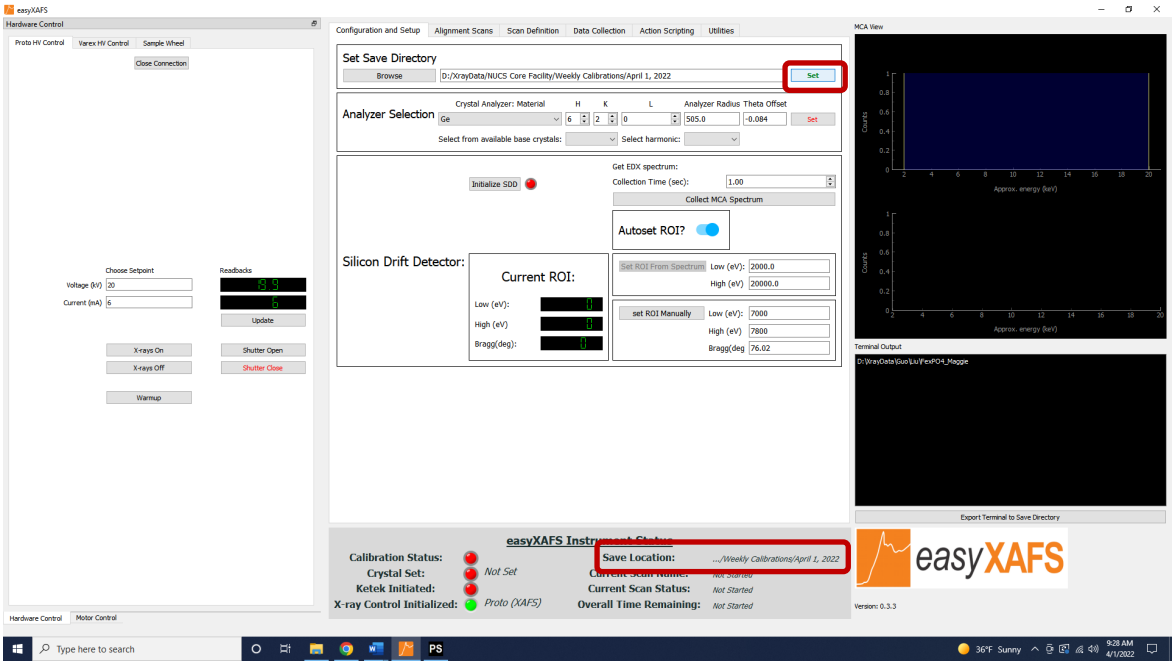
6.29 After the X-ray tube has warmed up, you will need to configure the instrument. Using the easyXAFS software, click on the Configuration and Setup tab.



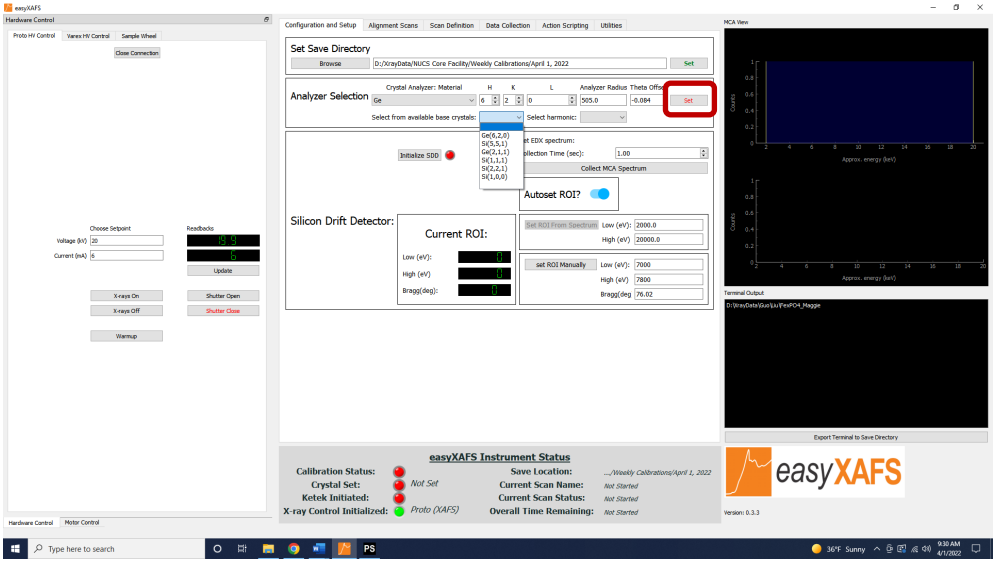
6.30 Start by setting the directory in the Set Save Directory section for the data collection. To select a folder, click on Browse. The data is stored in the D:\XrayData folder. Then, select the folder of the particular research group (i.e. NUCS Core Facility), followed by the folder designated by the user's last name.



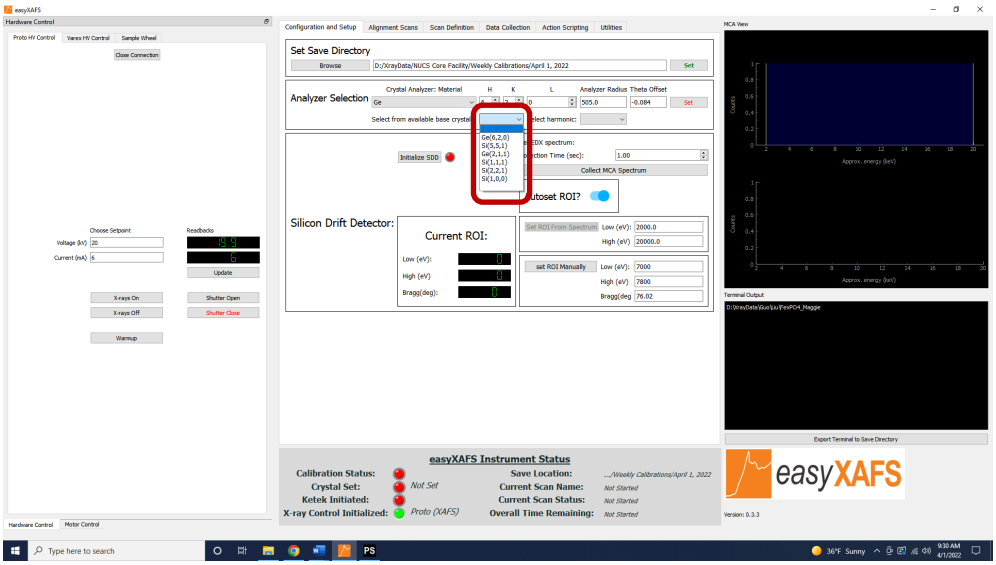
6.31 Once the directory has been chosen, click on Set (highlighted in red) to set the directory for data collection. If the directory has been set correctly, the Set button will become green and the Save Location will be indicated on the lower part of the screen (highlighted in red).



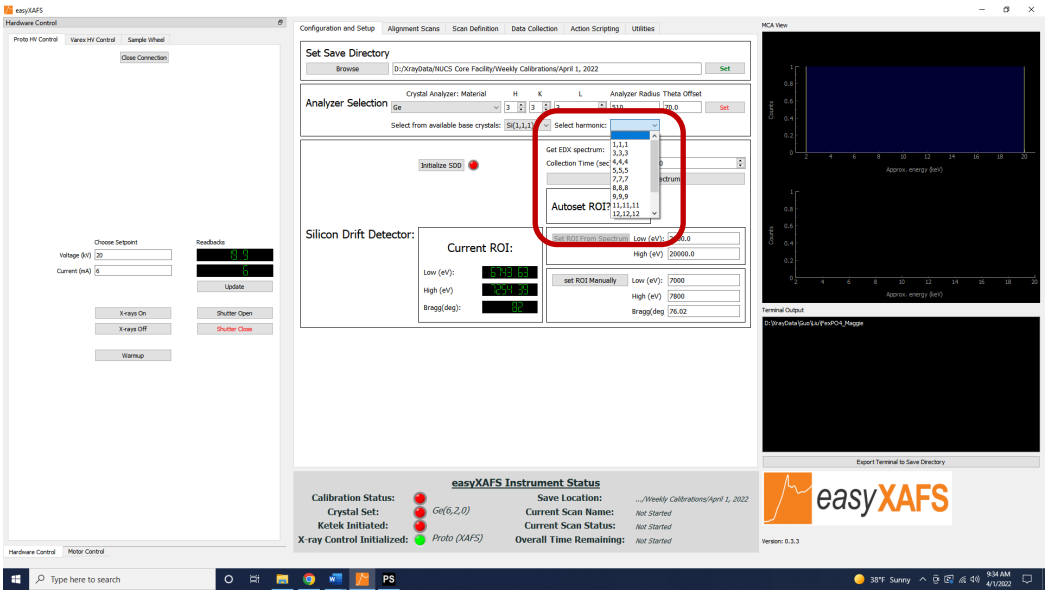
6.32 Next, the crystal analyzer identity will need to be specified. The default value is for a Ge 620 crystal, which is used to analyze iron samples. If this crystal is to be used, the set button can be clicked. See Steps 6.36-6.41 for directions on changing a crystal.



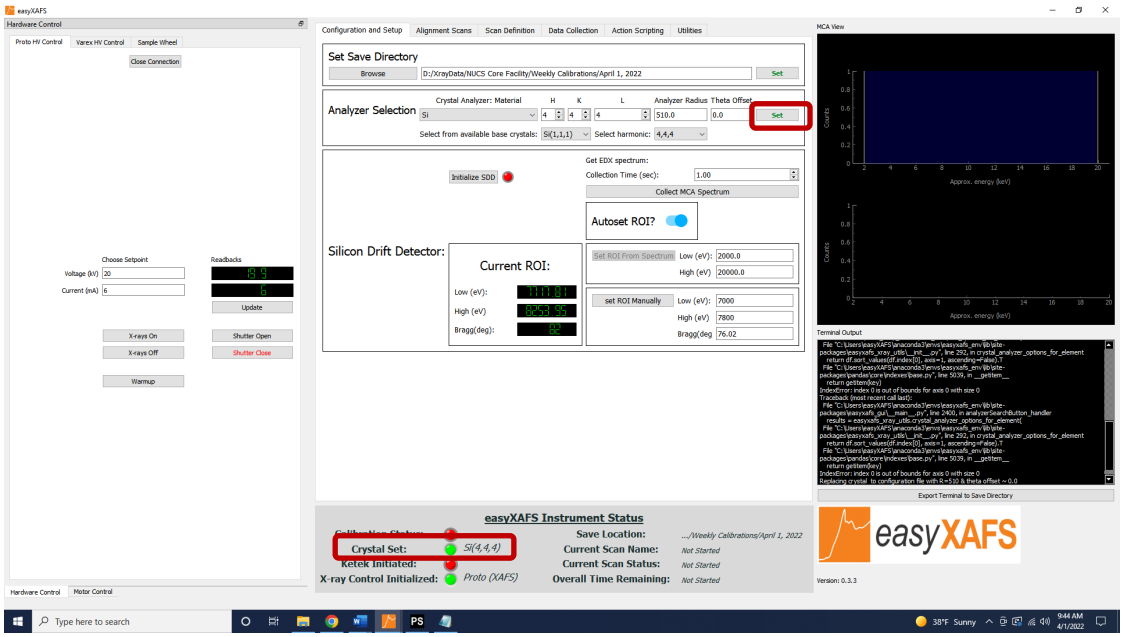
6.33 If another crystal is to be used, it can be selected from the drop-down menu, as seen below.



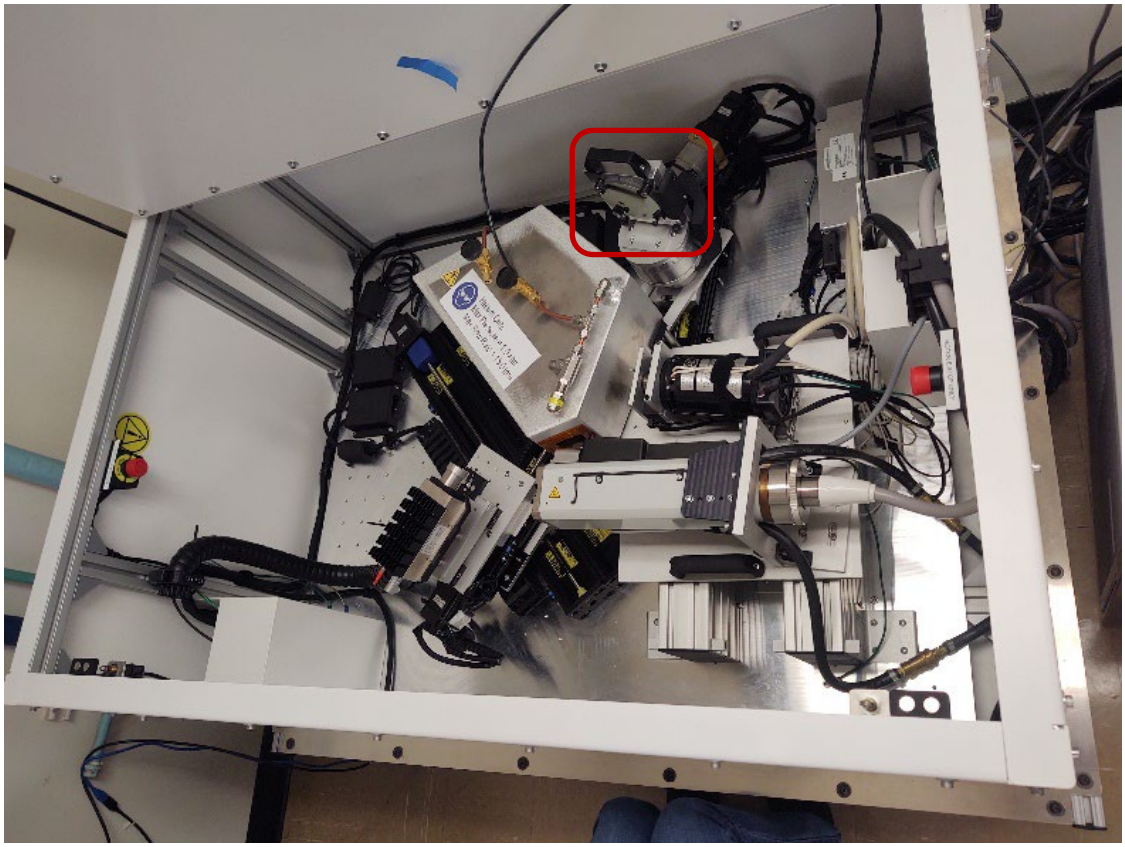
6.34 Upon selecting the crystal of choice, the harmonics will then need to be selected. A selection from the harmonics drop down will need to be chosen. After making a selection, click on the Set button to set the crystal analyzer.



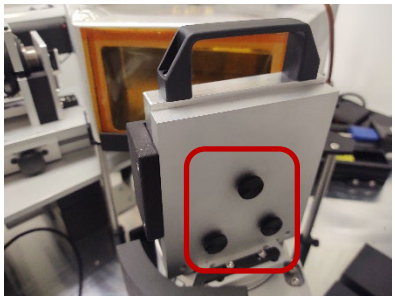
6.35 If the crystal analyzer has been chosen correctly, the Set button will become green and the Crystal Set will be indicated on the lower part of the screen (highlighted in red).



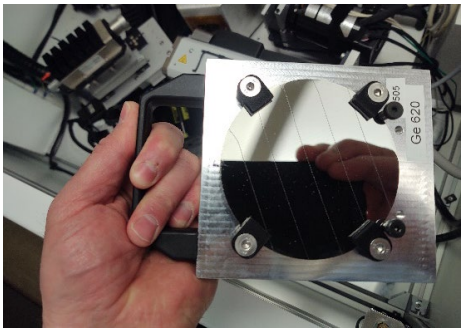
6.36 If the crystal analyzer needs to be changed, it can be replaced with the desired analyzer. The crystal analyzer can be found in the rear of the instrument (highlighted in red).



6.37 To remove the current crystal, reach behind the holder for the crystal analyzer and loosen the three mounting screws with your hand.



6.38 When the screws have been loosened, the crystal can be lifted out of the mount by pulling up on the handle at the top of the crystal. If the crystal does not lift out of the mount, then the mounting screws have not been completely loosened. Be careful handling the crystal analyzers, they cost ~ $8,000 each.

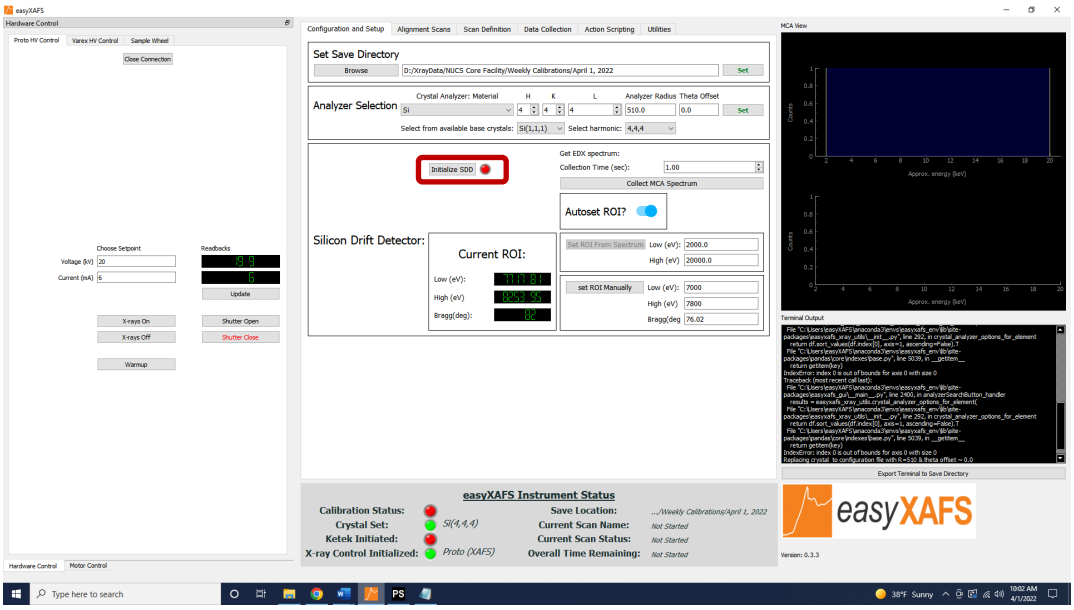


6.39 Place the crystal analyzer in one of the empty slots in the crystal cases.

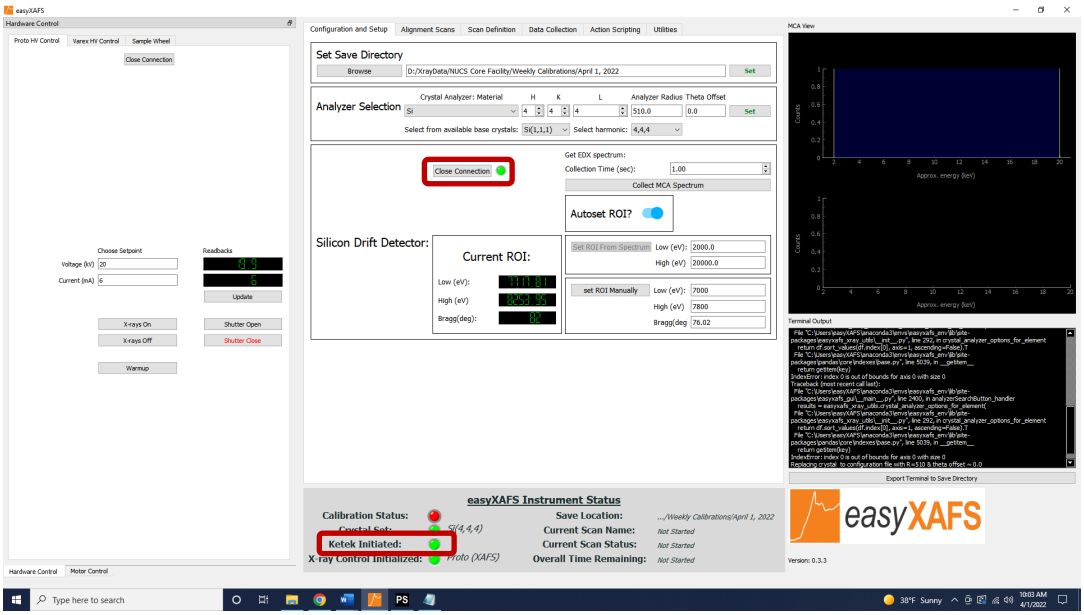
6.40 Remove the desired crystal from the crystal case and place in the crystal analyzer mount in the instrument. It helps to set the crystal on the bottom mounts (highlighted in red) and then to slide it to the side mount (highlighted in blue).

6.41 Tighten the mounting thumb screws, they should easily screw into the crystal analyzer. If the screws do not easily screw in, the crystal analyzer is not in the correct position. The thumb screws should be snug, but not overly tight. The thumb screws should be easily loosened by hand.

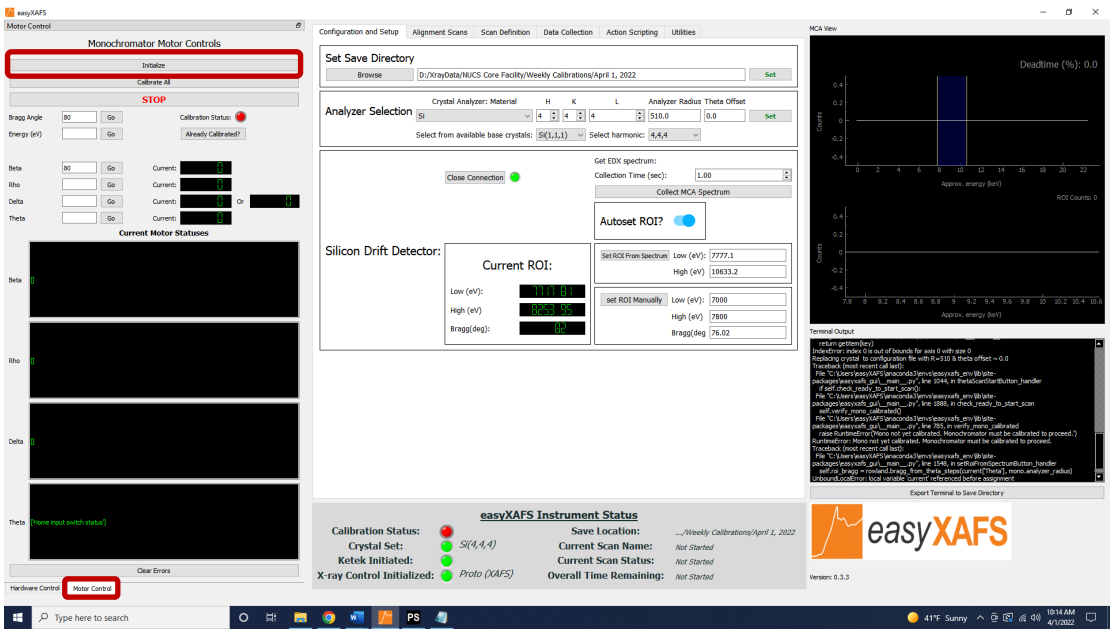
6.42 After setting the crystal analyzer, the detector needs to be initialized. Click on the Initialize SDD button.



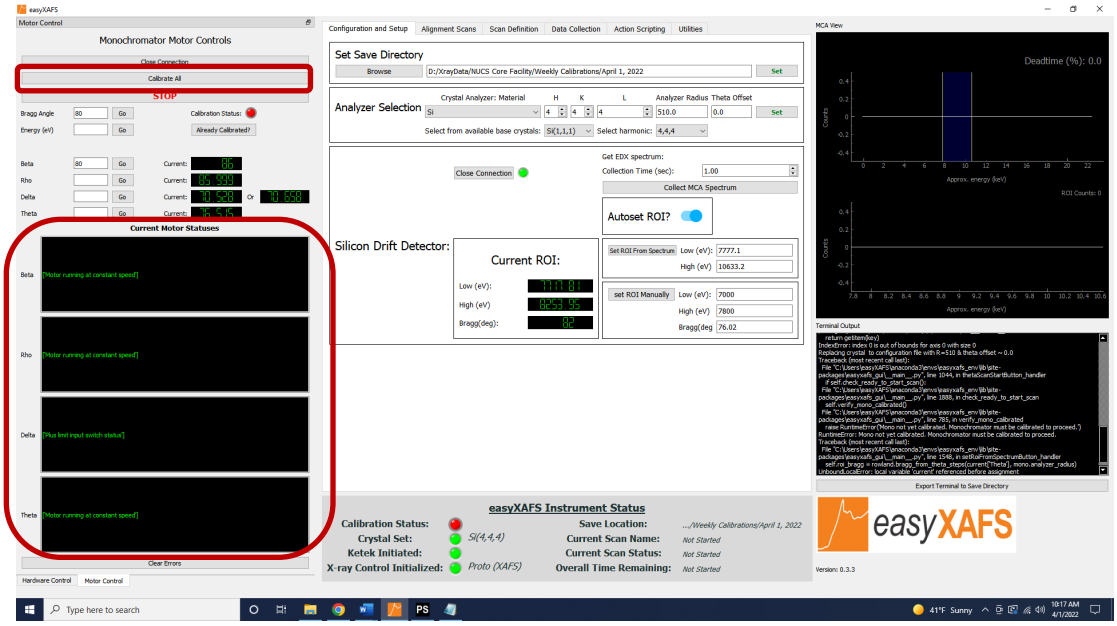
6.43 The light next to the Initialize SDD will become green and the light next to Ketek Initiated will become green (see below).

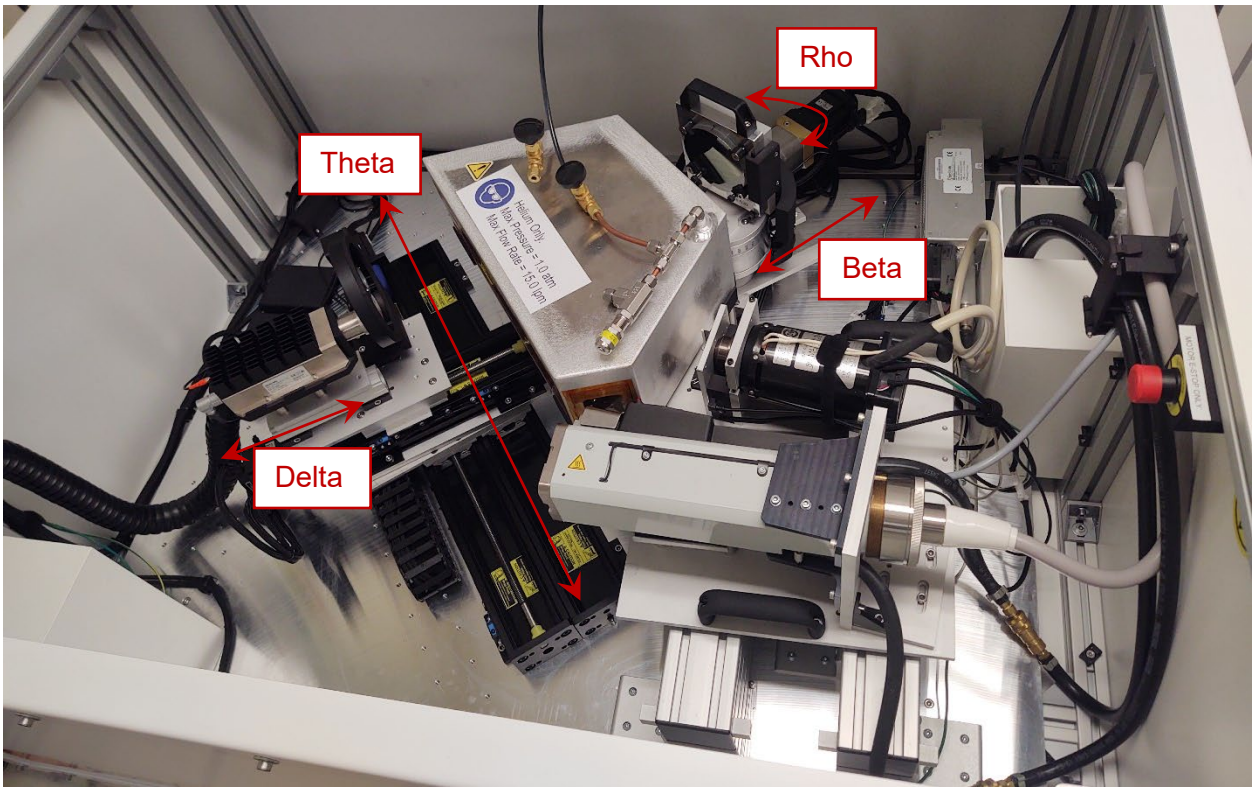


6.44 Before a spectrum can be collected the motors in the instrument need to be calibrated. This calibration process helps the motors find their stopping points. This calibration only needs to be completed if the instrument was turned off since its last use. To begin the calibration of the motors of the instrument, click on the Motor Control tab in the Hardware control window (if not already visible), followed by clicking on the Initialize button. Clicking of the initialize button will return the current positions of the motors.

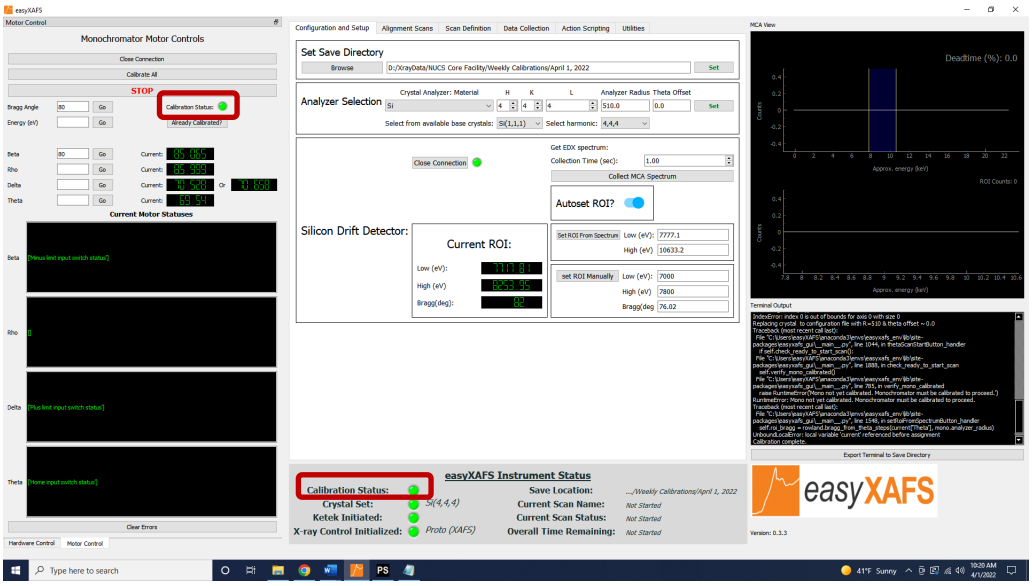


6.45 To calibrate the motors, click on the Calibrate All button, and you will hear the motors moving and the motor status window will indicate that the motors are moving to their starting positions. The motors and their respective names can be seen below.



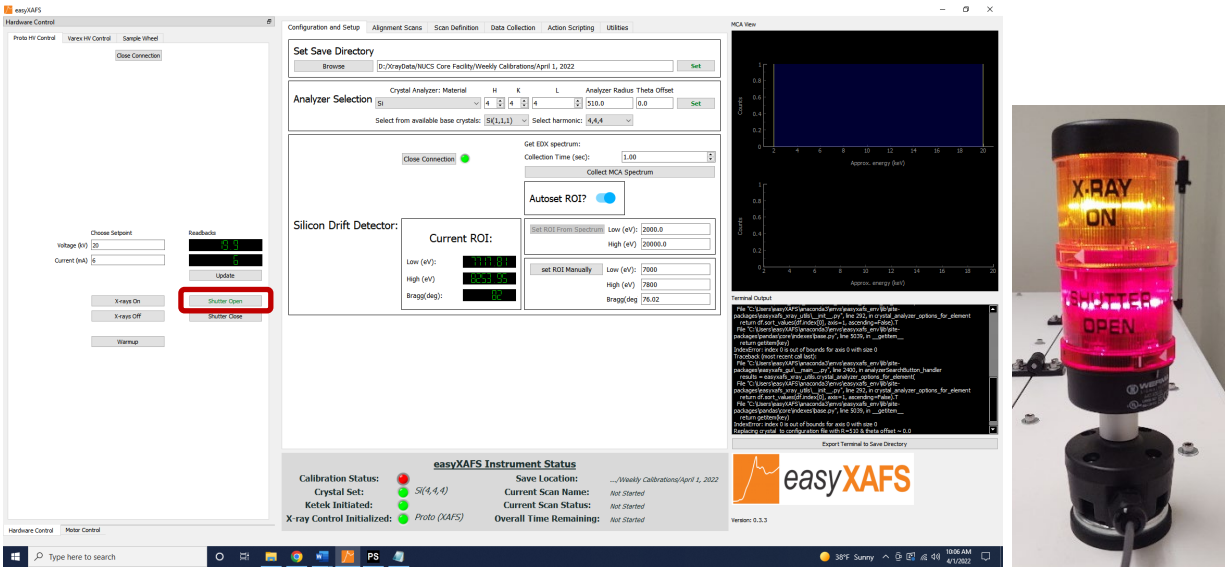


6.46 When the motors have been calibrated, the Calibration Status lights will be colored green.

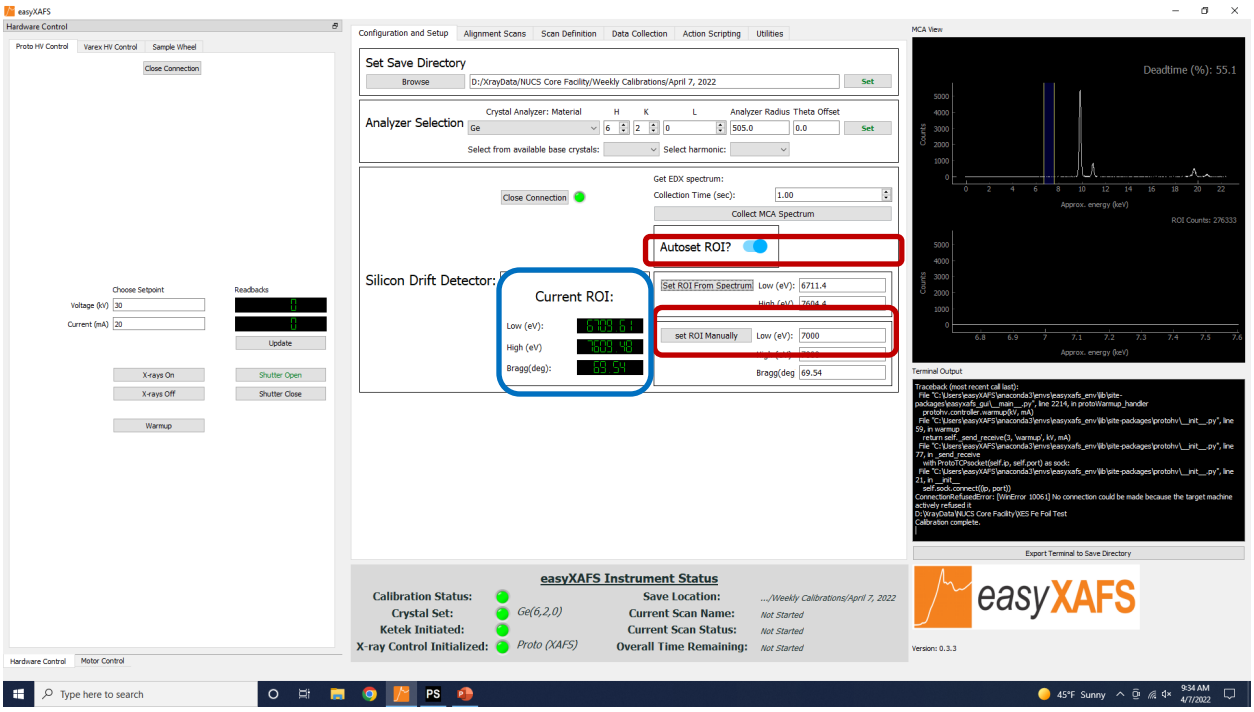


6.47 Close the door to the instrument if it is not already closed. Note: the door should only be completely open or completely closed. Partial opening and closing the door to the instrument reduces the lifetime of the gas shocks on the instrument door.

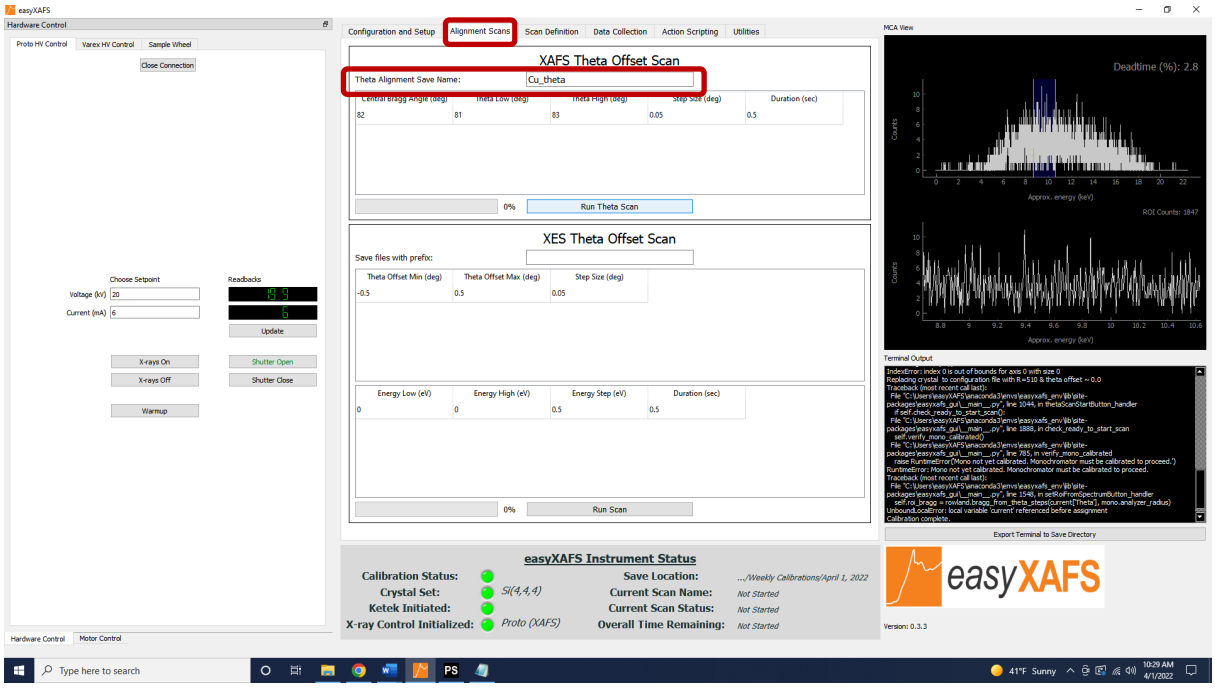
6.48 Then, to continue with the calibrations, the shutter to the X-ray tube needs to be opened (instrument door should be closed at this time). Click on the Shutter Open button in the Proto HV Control window. The button will become green when the shutter is open. The lights on both the X-ray On and Shutter Open indicators on the top of the easyXAFS should be illuminated. WARNING: Do not open the door to the instrument when the shutter is open and X-rays are on.



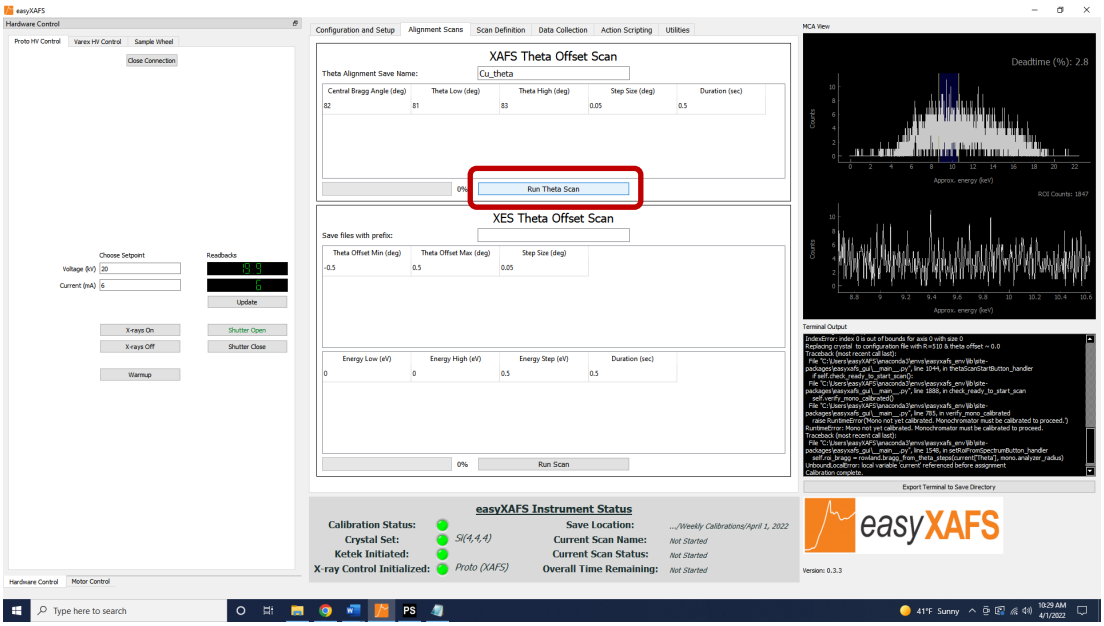
6.49 To continue calibrating the detector, click on the Collect MCA Spectrum button and adjust the Region of Interest by clicking and dragging the boxes in the top MCA spectrum to surround the energy of interest and clicking on the Set ROI from Spectrum button. For an iron foil, the K-edge is 7110 eV, so a range of about 6500 eV to 7500 eV is acceptable. The ROI needs to have the energy of interest (7110 eV for iron) in between the Low and High energies of the ROI. Current ROI is highlighted in blue.



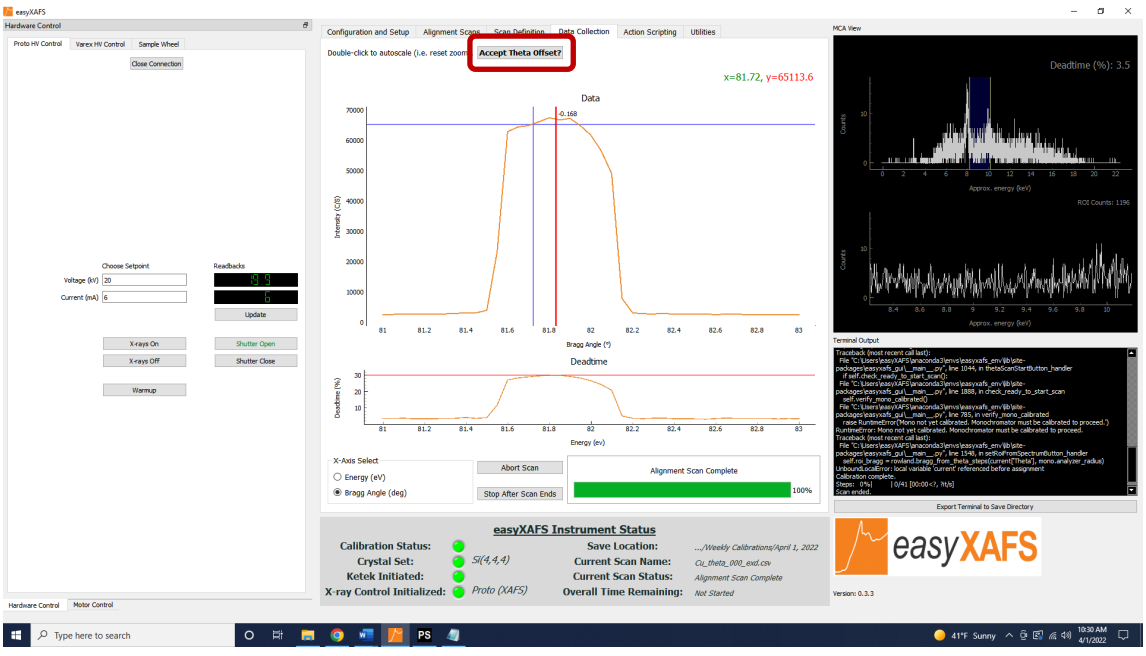
6.50 Then, select the Alignment Scan tab and enter the Theta Alignment Save Name.



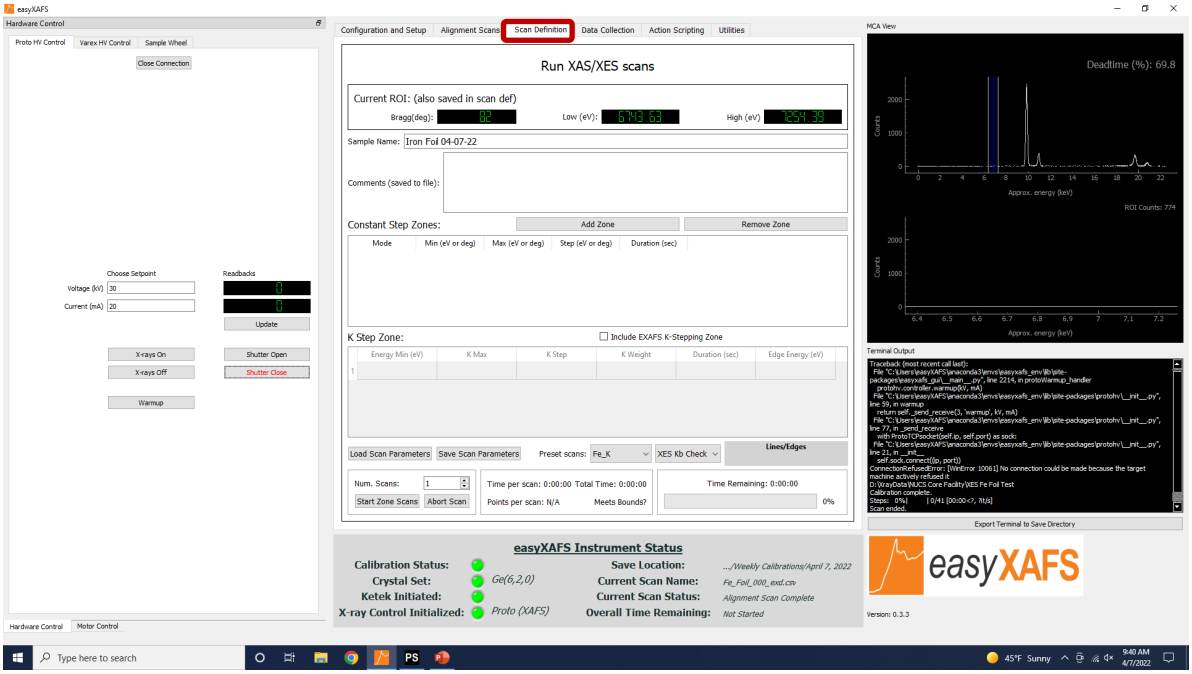
6.51 Then click on the Run Theta Scan button to start the Theta scan.



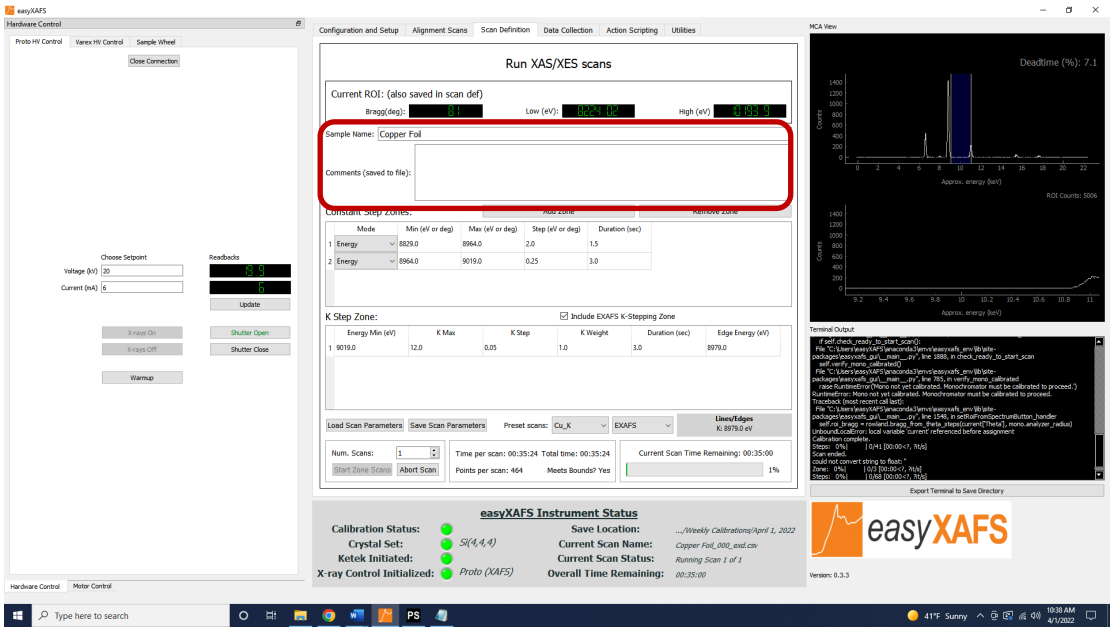
6.52 The screen will change to the Data Collection tab and the following plot will be observed when the Theta scan has completed. The program will place a cursor in the center of the peak and ask you to accept the Theta Offset. Click on the Accept Theta Offset button. In a normal scan, all four motors move. When aligning for the crystal, we want only the Theta motor to move.



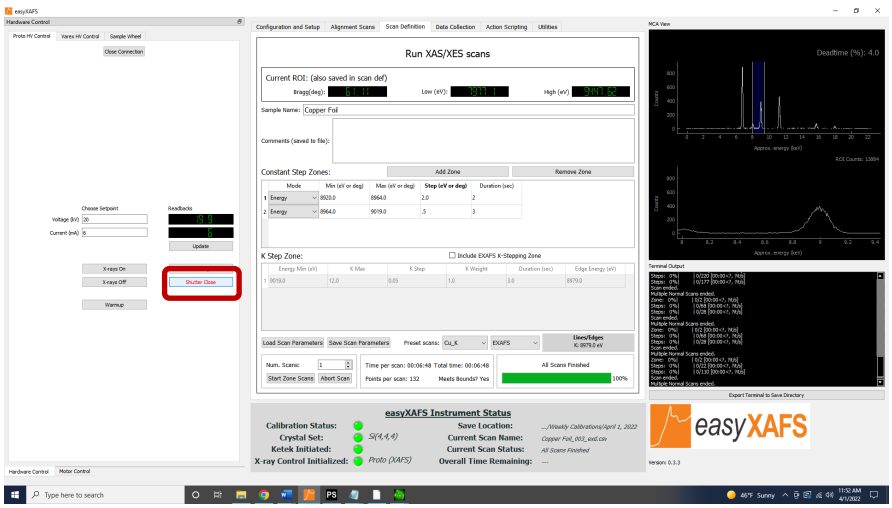
6.53 After setting the Theta offset, the scan definition tab is used to set the parameters for data collection.



6.54 Start by entering the Sample Name and any comments with regard to the experiment. It is good to include information about the sample, X-ray tube identity (Mo or Ag), X-ray tube settings, etc.



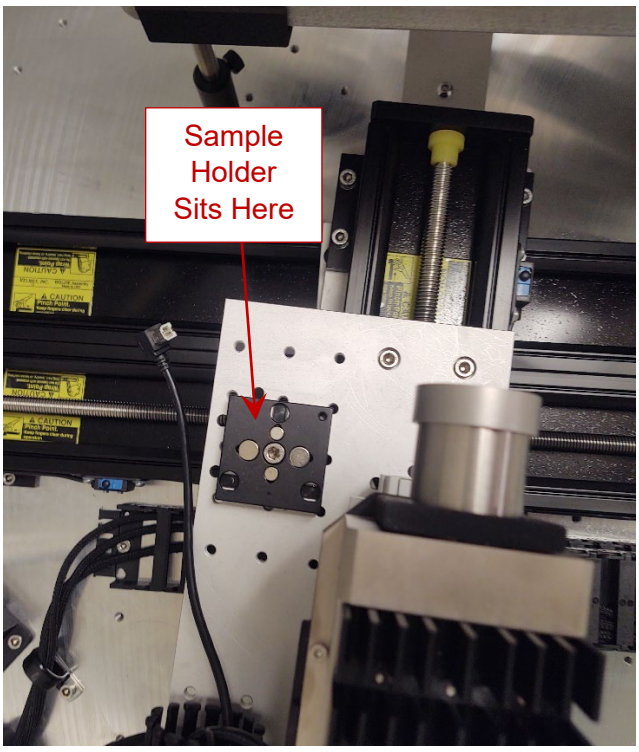
6.55 Click on the Power Supply window and click on the Close Shutter button to close the shutter. The Shutter Open light should not be illuminated.

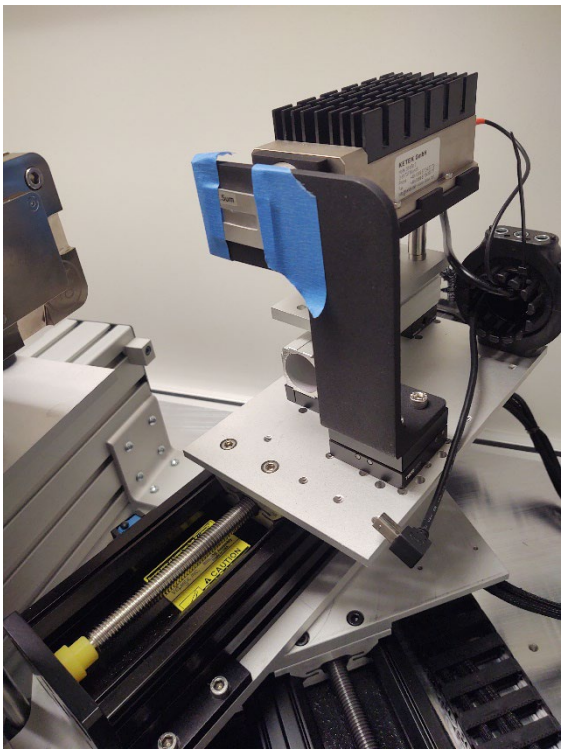


6.56 Select sample for analysis, this should be a reference foil for an initial run to calibrate the energy space. The NSC has acquired a set of 3d, 4d, and 5d metal foils from EXAFS Materials as standards for calibration of the easyXAFS 300+. The 7.5 µm iron foil is used for a training session.

6.57 Attach the sample with masking tape to the single sample holder. The sample changer can also be used, but it is often easier to have the reference foil o the single sample holder. Blue painter's tape is provided. Make sure that the metal foil can be seen through the hole in the sample holder. Also, make sure that the sample is on the opposite side of the magnetic mount.

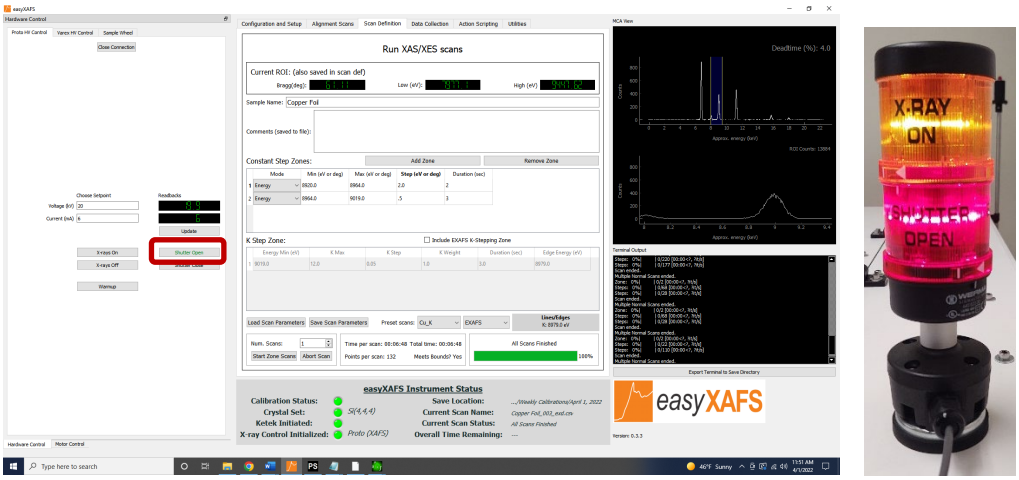
6.58 Place the sample holder into the instrument in front of the detector. The sample holder clicks into place with magnets.



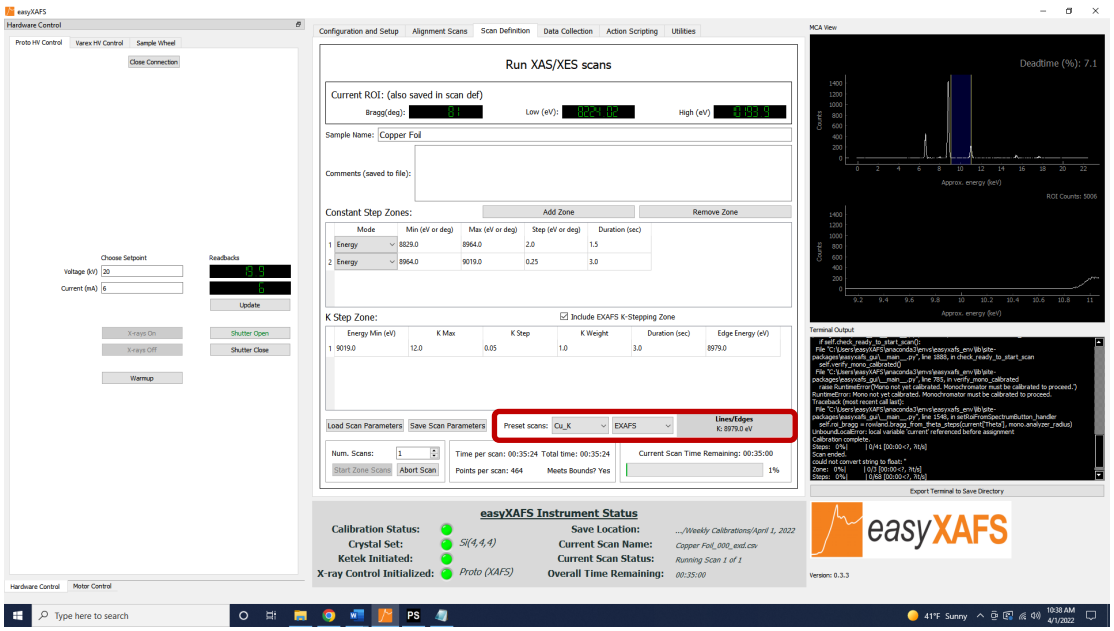


6.59 Close the door to the instrument.

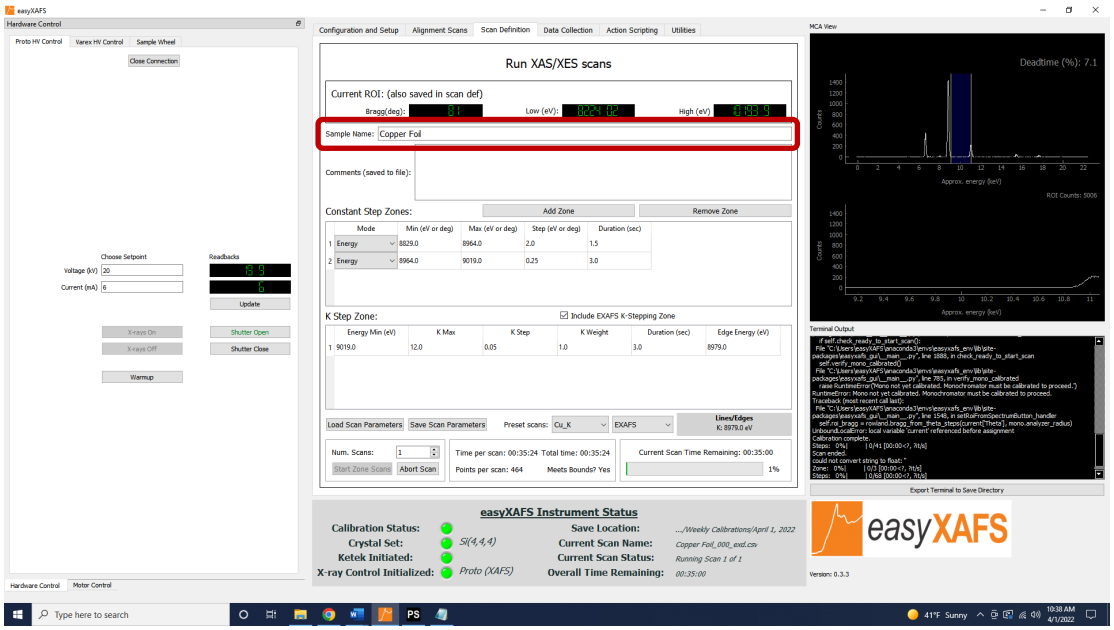
6.60 Click on the Power Supply window and click on the Open Shutter button to open the shutter. Both the Shutter Open and X-rays On lights should be illuminated at this time.



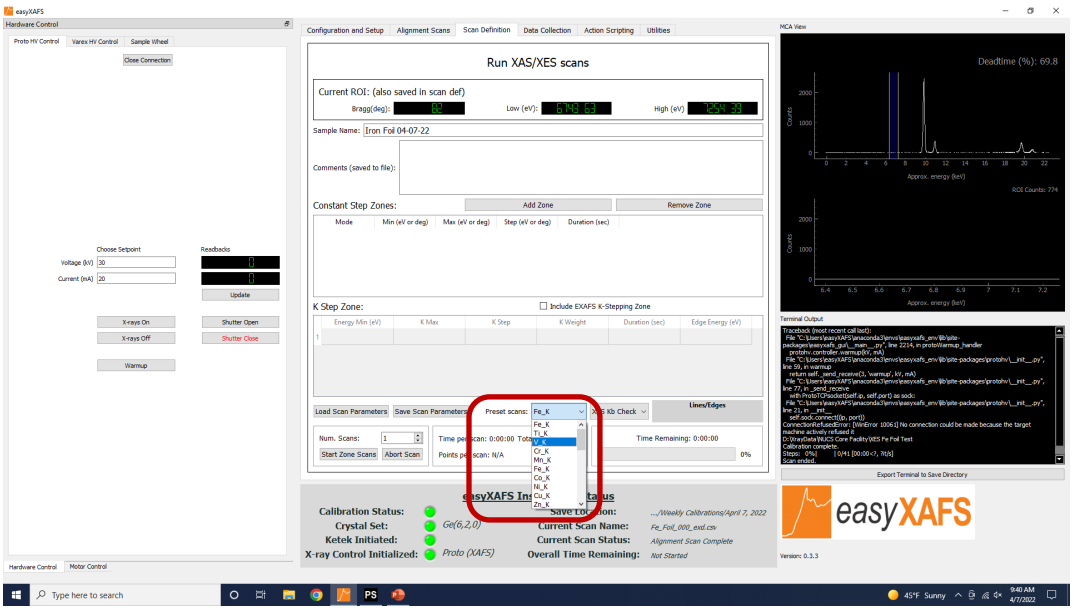
6.61 A good place to start in setting up your experiment is to use the preset parameters (highlighted in red).



6.62 Adjust the Sample Name and comments, if necessary.



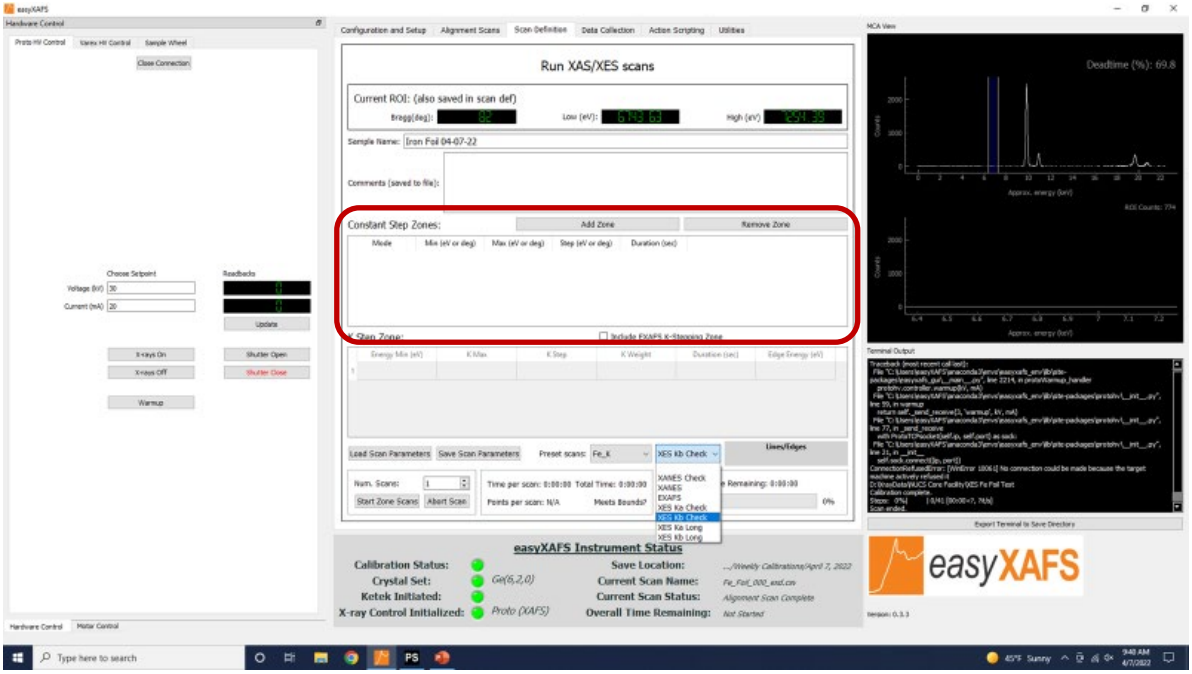
6.63 Clicking on the element drop down allows for the selection of a set of element edge energies to be selected. Select the element and edge to be examined. For example, the Fe_K option should be selected to measure the K-edge of iron.



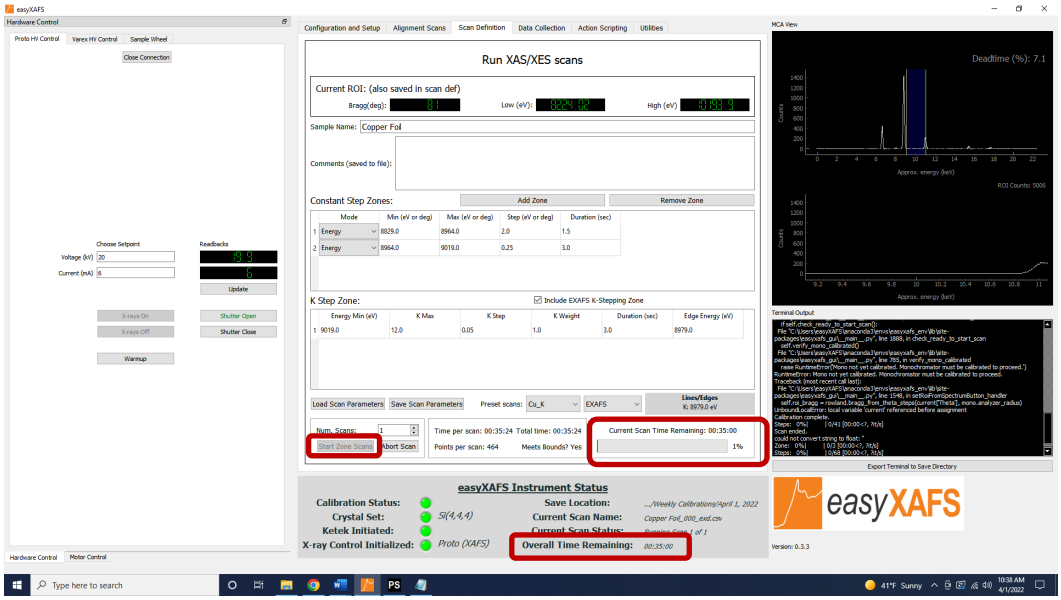
6.64 Clicking on the energy drop down allows for the selection of a vendor defined set of scan parameters that are based around the absorption or emission energies of a particular element. Select the desired experiment.

In the case of an XAS experiment, the XANES check experiment will last 18 seconds (Range of 30 eV above and below the edge) and provide a user with information on whether an absorption edge is present. The XANES option consists of three zones (pre-edge (40 eV before edge to 15 eV before the edge), edge region (15 eV before edge to 40 eV after the edge), and post-edge (40 eV after the edge to 180 eV after the edge),) and will take about 16 minutes for the data collection. The EXAFS option consists of two zones (pre-edge (150 eV before edge to 15 eV before the edge) and edge/post-edge region (15 eV before the edge to 40 eV after the edge),) and will take about 36 minutes for the data collection. For an initial scan, the XANES check should be selected.

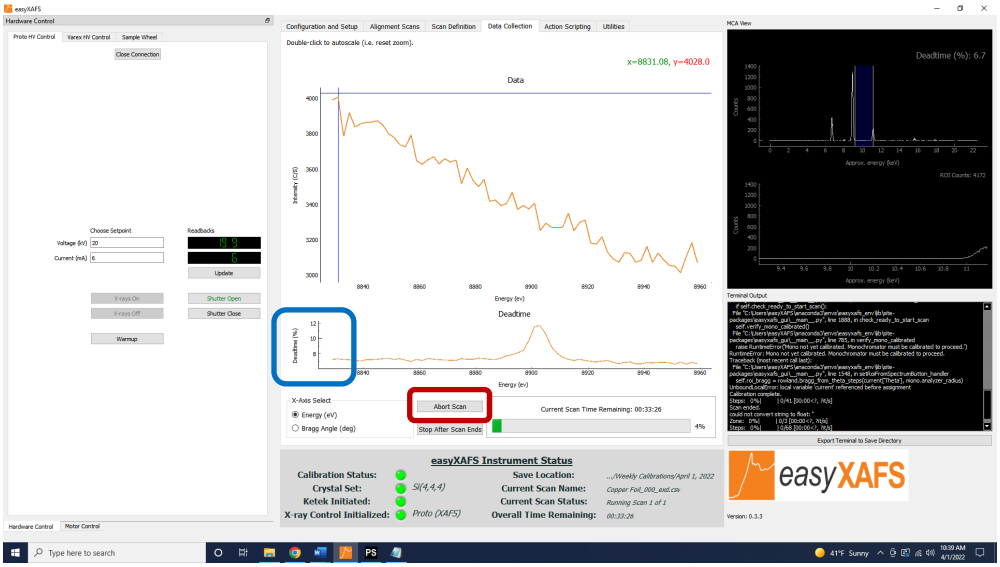
By selecting one of these preset scans, a user will have a good starting point for a data collection experiment, but these parameters may not be the best for all samples. If other parameters are desired, they can be changed in the Constant Step Zones section (highlighted in red). Increasing the Step size will result in lower resolution data but a faster data collection. Increasing the Duration will increase the resolution and result in a slower data collection.



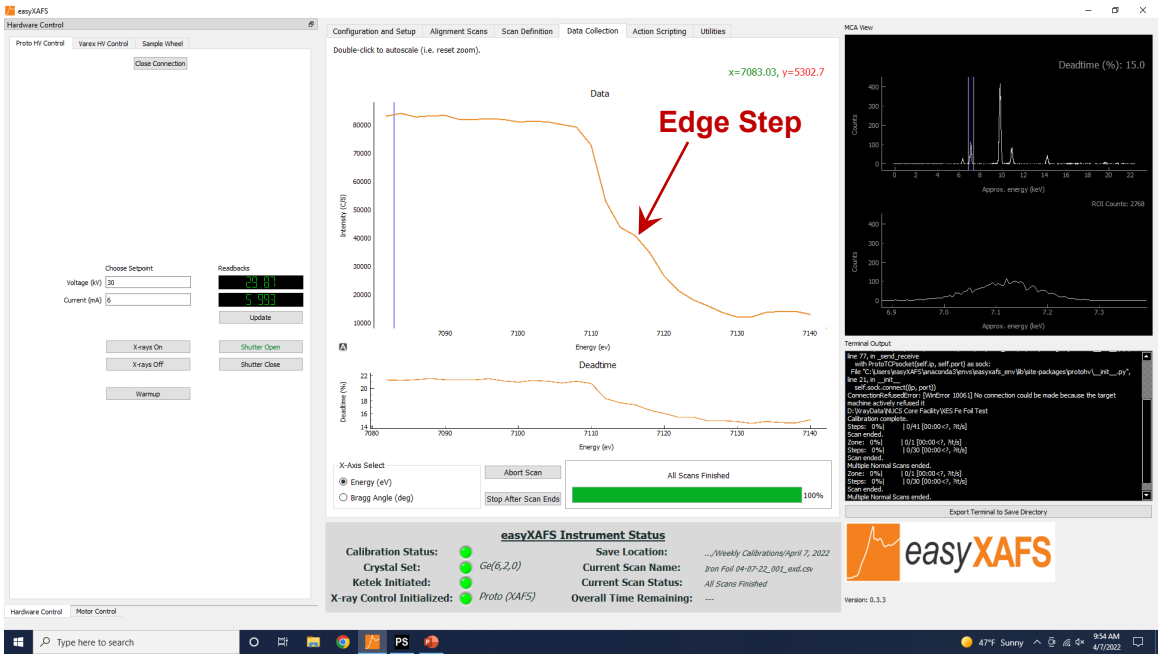
6.65 Once the desired zones for the experiment have been selected, click on the Start Zone Scans button to start the data collection. After the data collection has started, the time remining will be listed.



6.66 When data begins to be collected the following image will result. If a scan needs to be stopped, the Abort Scan button can be clicked on. The deadtime should be less than 30% (highlighted in blue).

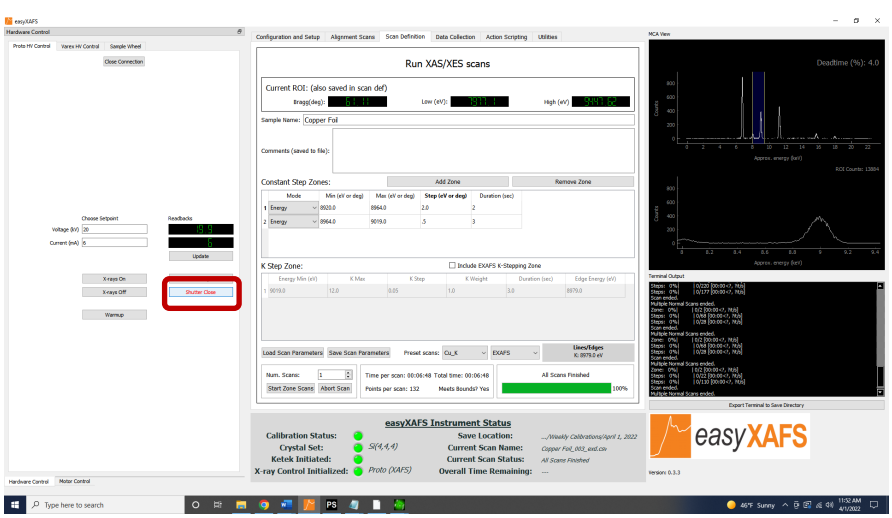


6.67 If an edge is not seen with a reference foil, then there is a problem with your Region of Interest. Try resetting it (Step 6.49) and try a data collection again.



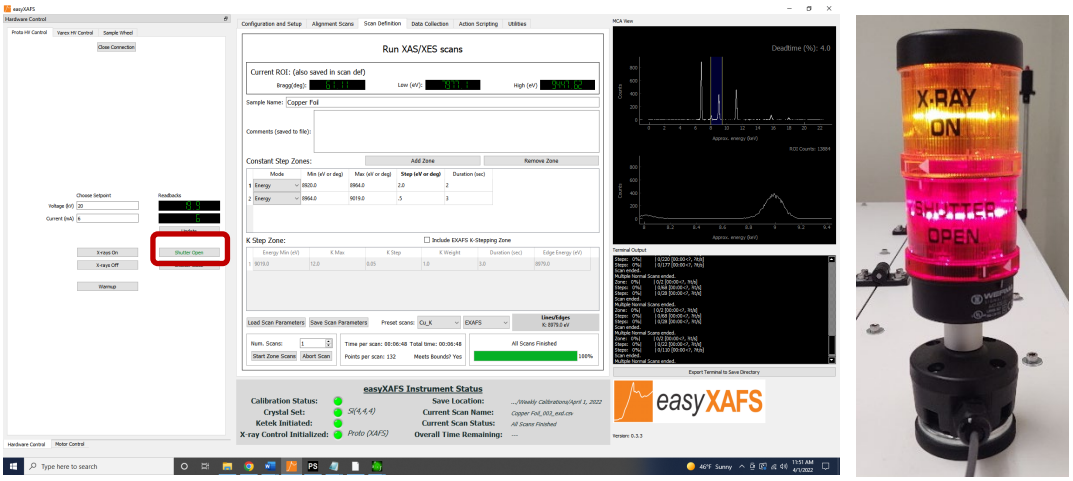
6.68 If an edge is seen in Step 6.67, then the instrument is ready to collect data on samples containing the element present in the reference foil and a user sample can be placed in the spectrometer.

6.69 Click on the Power Supply window and click on the Close Shutter button to close the shutter. The Shutter Open light should not be illuminated.

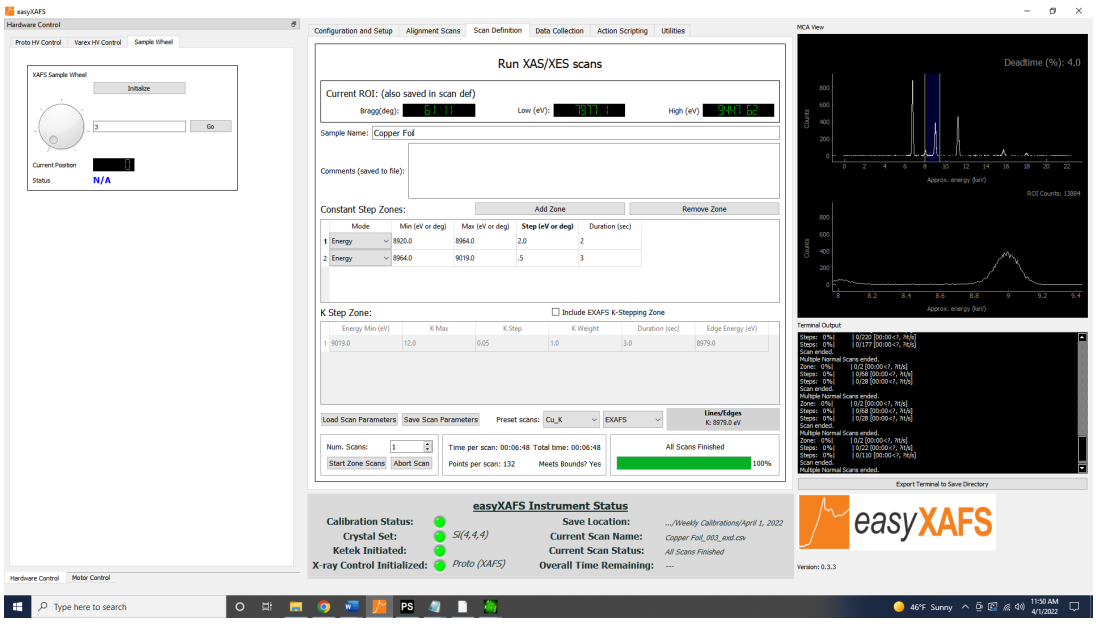


6.70 Attach the sample to the sample holder, place the sample in the spectrometer, and close the door to the spectrometer.

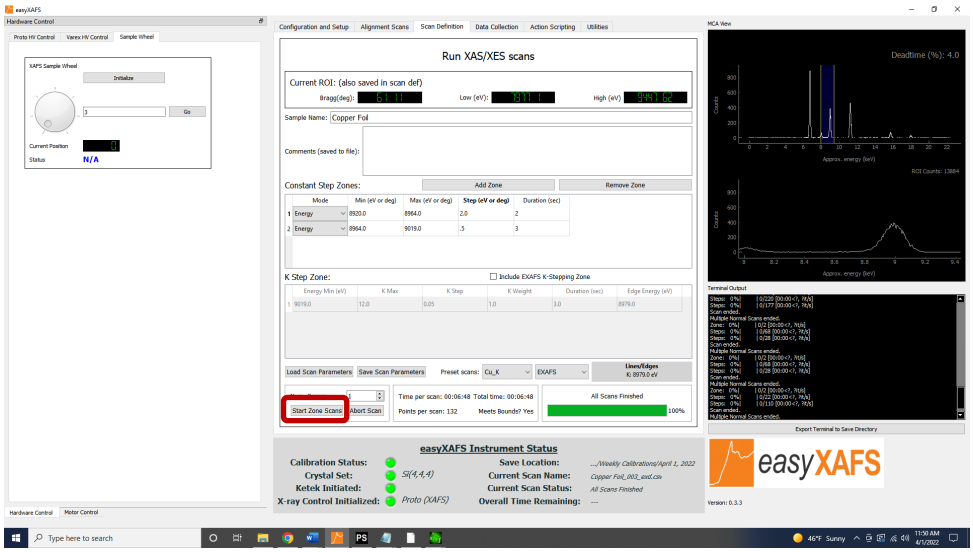
6.71 Click on the Power Supply window and click on the Open Shutter button. The X-ray light and the shutter open light should now be on.



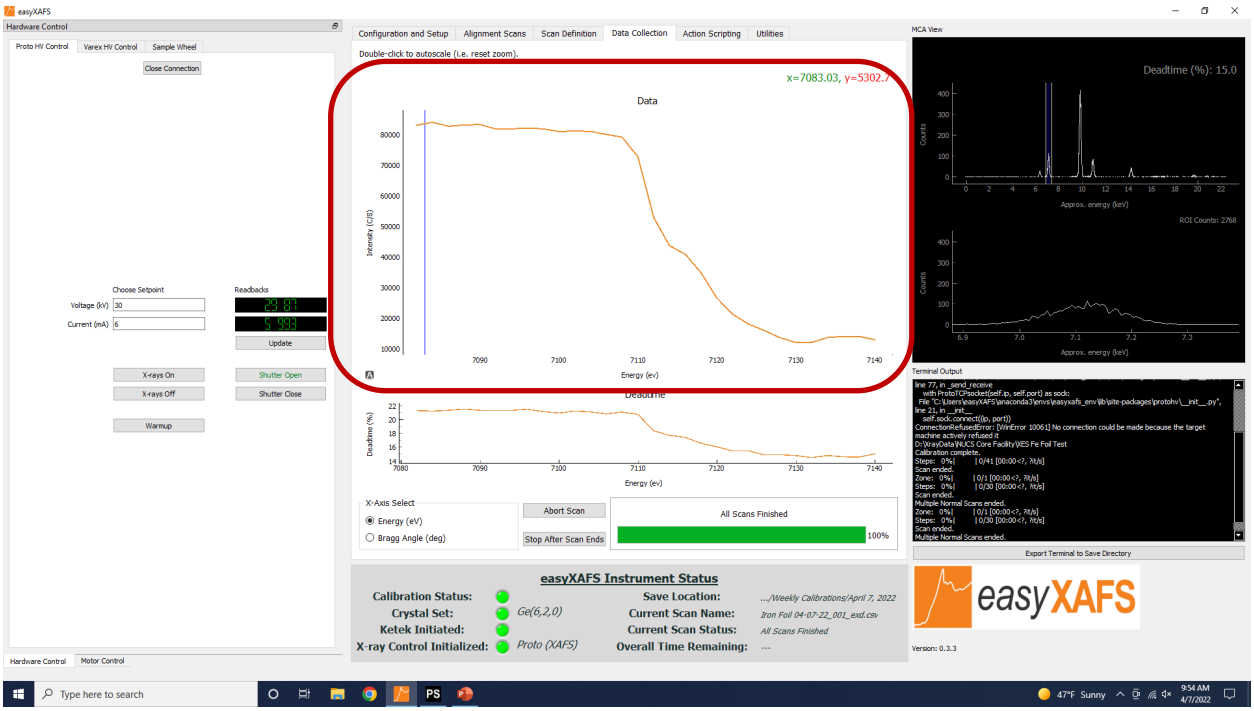
6.72 Click on the Scan Definition tab, adjust the zones for data collection and Sample Name as desired. Adding additional zones, if needed.



6.73 Then click on the Start Zone Scans button. Make sure that the shutter is open, if not, the Dead Time will read 255%. The Dead Time should be 30% or less. If the Dead Time is greater than 30%, decrease the current or the voltage of the X-ray tube to obtain a Dead Time less than 30%. If the scan needs to be stopped, the Abort Scan button can be clicked.



6.74 When the scan has completed, a XAFS spectrum will be displayed in the XES/XAFS Spectrum window. The data is automatically saved at the end of the data collection.



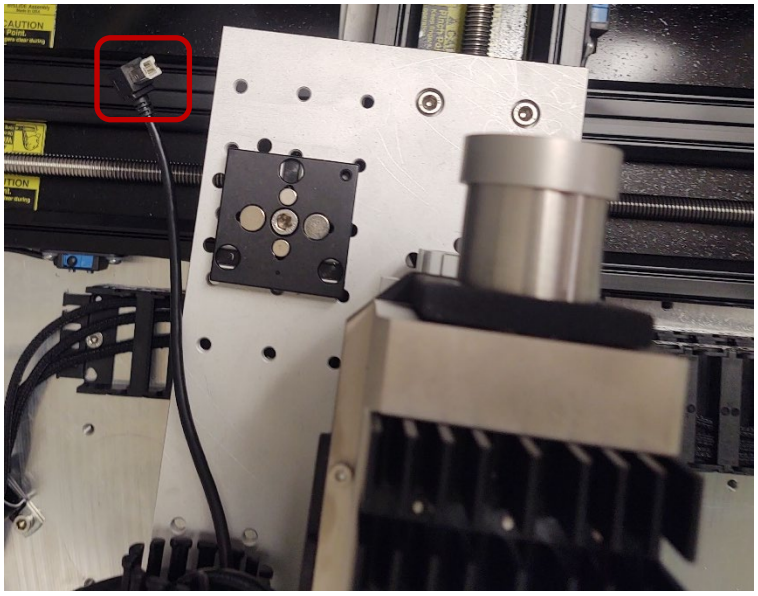
6.75 At this point you can collect the I_{zero} (background) data, by closing the shutter (Step 6.69) and repeating Steps 6.71 and 6.73 without a sample present in the spectrometer. This measurement provides a background spectrum. Be aware that Currents as low as 3 mA, when the Voltage is 30 kV, are needed to have a Dead Time lower than 30%. The I_{zero} needs to be collected at some point during the instrument time before changing the instrument settings. An Izero will need to be collected for each data collection parameters (i.e. changing the Zone Steps in Step 6.64).

6.76 After the I_{zero} has been collected, the sample holder can be inserted back into the spectrometer and data on samples are collected according to Steps 6.69-6.74. Please keep the data collection parameters (Step 6.64) the same for all of the samples collected to simplify the data analysis (Section 8).

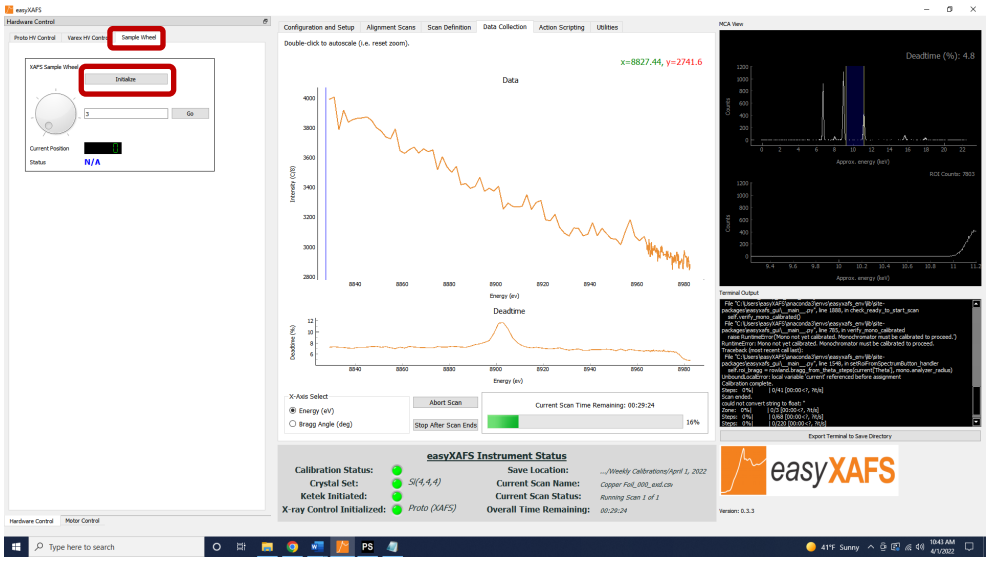
6.77 If desired, the Sample Wheel (pictured on the left), which can house seven samples, can be used in place of the single sample holder (pictured on the right).

6.78 If the sample wheel is used in the spectrometer, make sure that the masking tape that is holding the samples in the sample holder does not prevent the sample wheel from rotating.

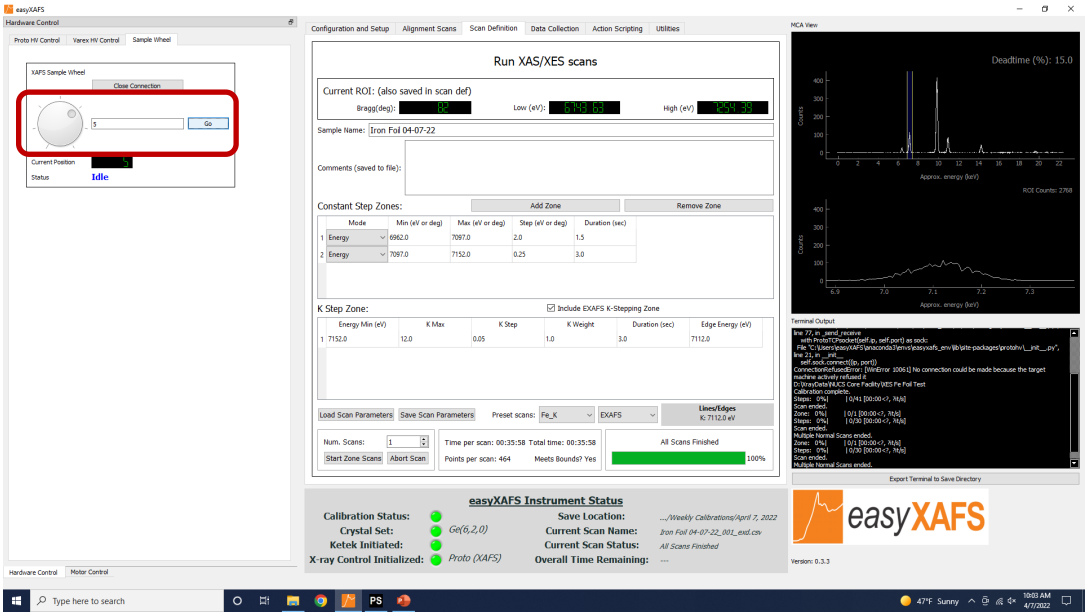
6.79 Once the sample wheel is loaded with samples, it is placed in the spectrometer and the USB cable is connected to the holder to control the sample selection.



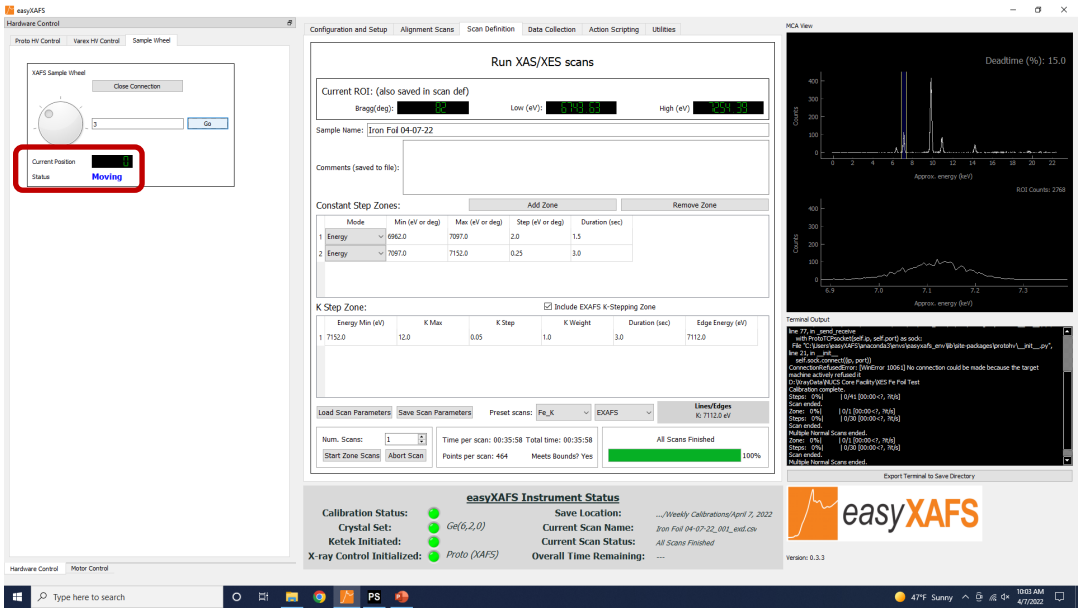
6.80 To use the sample wheel, click on the Sample Wheel tab and click on the Initialize button. Every time the sample wheel is removed from the instrument, the Initialize button will need to be clicked.



6.81 To advance the Sample Wheel, either enter a sample position in the text box and click on the Go button or use the wheel to adjust the sample location and click on the Go button to advance the Sample Wheel.



6.82 The sample wheel will take 1-2 seconds to move to the desired sample location. The indicator will say that the wheel is moving and the Current Position is 0, until it reaches the set position, which will have the Current Position listed as the desired sample position.

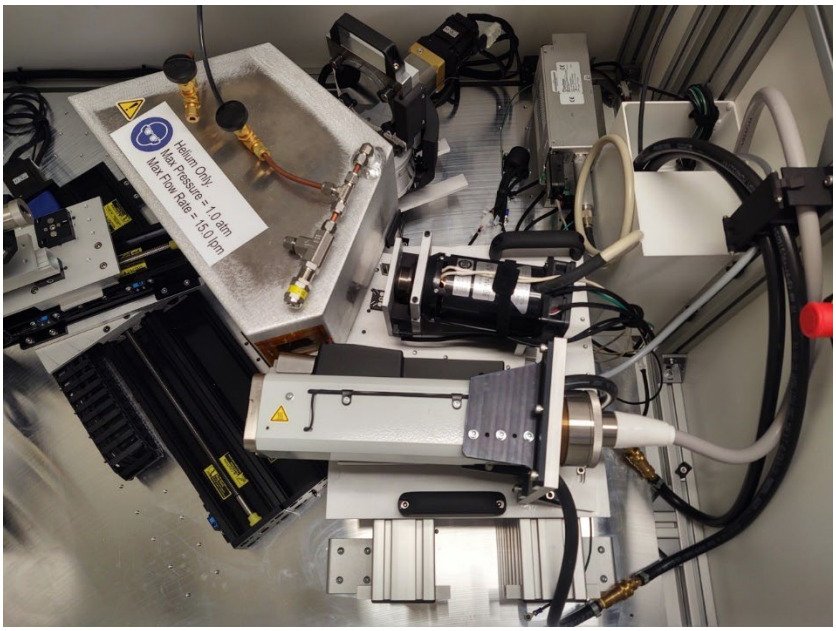


6.83 Upon completion of the data collection, please see Section 8 for Data Analysis and Section 9 to power down the spectrometer.

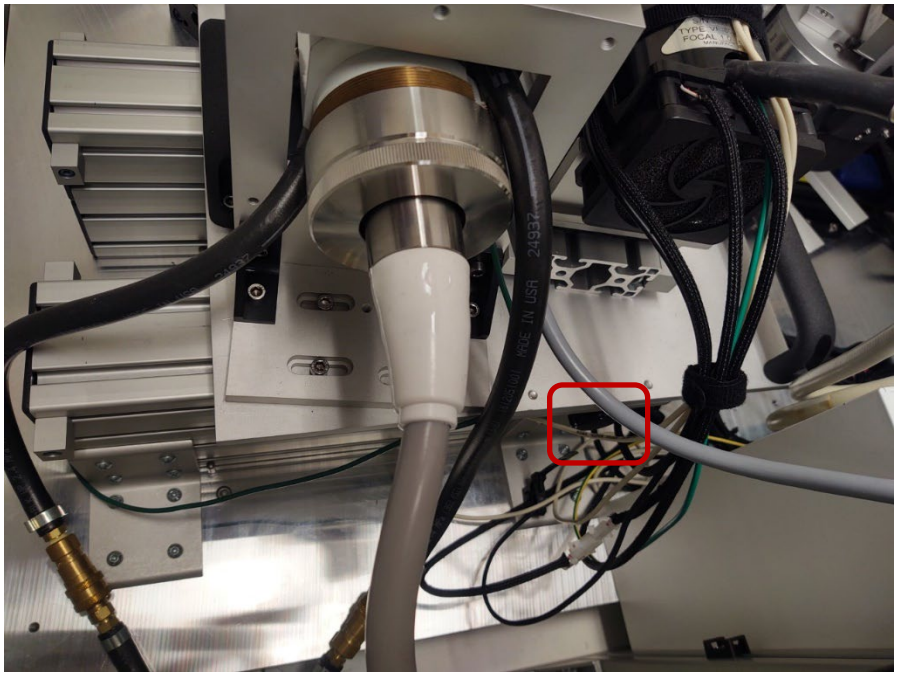
7 XES Data Collection on the easyXAFS 300+ X-ray Spectrometer

It will take at least 15-30 minutes to switch between XAFS and XES mode. The NUCS Core Facility has a Pd and a W X-ray tubes for XES. The default XES X-ray tube is W. Please contact the NUCS Core Facility staff if the Pd X-ray tube is desired for XES measurements.

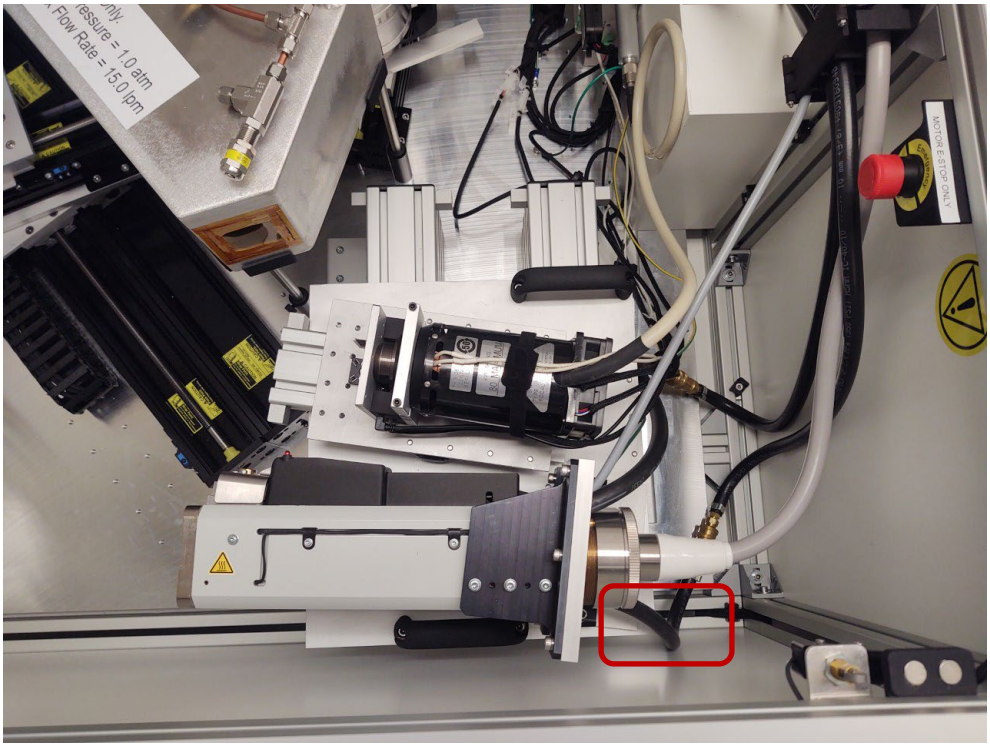
7.1 To set up the instrument for XES measurements, the XES X-ray tube needs to be moved into the source position, the original source position is seen below.



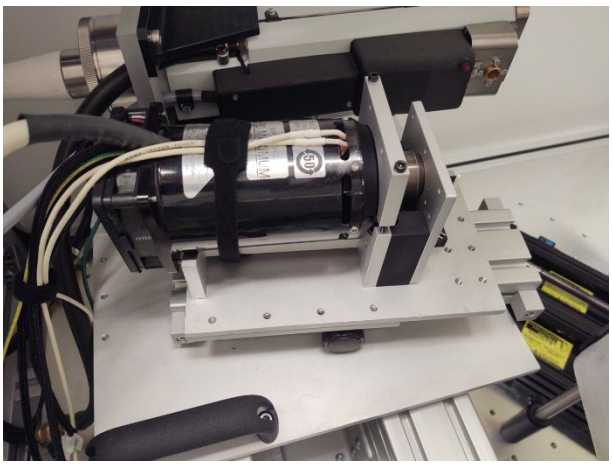
7.2 To move the X-ray tubes, unscrew knob underneath the X-ray tube platform.

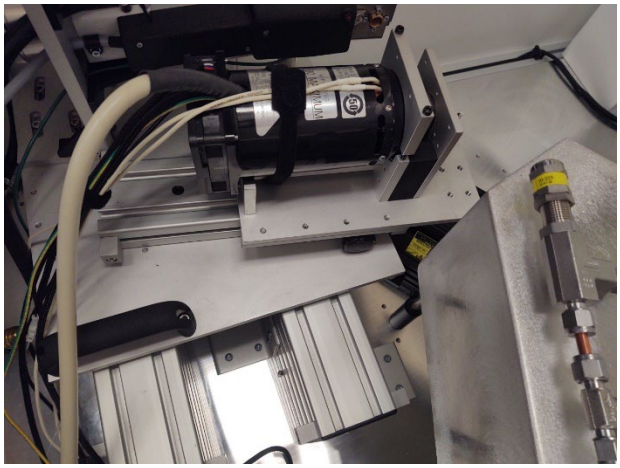


7.3 Slide X-ray tube platform back until it stops and retighten the knob to keep the tubes from moving. Make sure to not pinch any cables as the X-ray platform is moved.



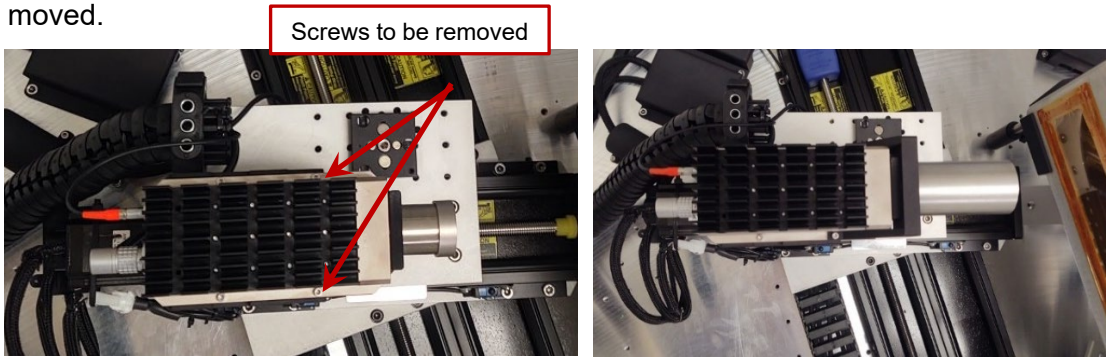
7.4 Unscrew knob underneath the XES tube and slide XES tube platform forward until it stops and retighten the knob.



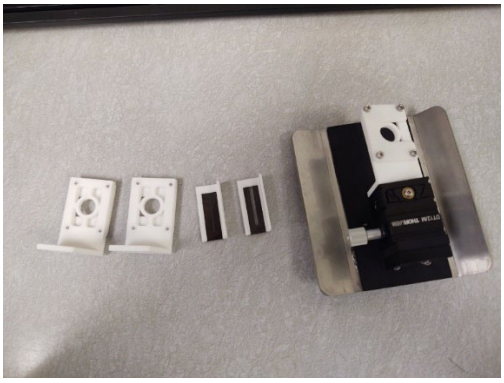


7.5 The sample holder, slits, and collimator can be found in a cardboard box in the bottom drawer of the desk at the easyXAFS.

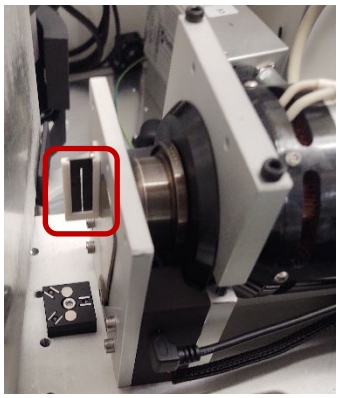
7.6 To minimize the counts that are not from the crystal a collimator is placed on the detector. Use a 2.5 mm hex key to remove the screws on the front of the detector and add the collimator and tighten the screws back down. If more space is needed, the motors can be moved.



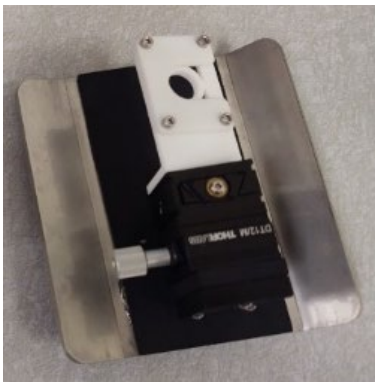
7.7 The instrument is equipped with three slits (0.5, 1.0, and 2.0 mm), where one is attached to the XES X-ray tube.



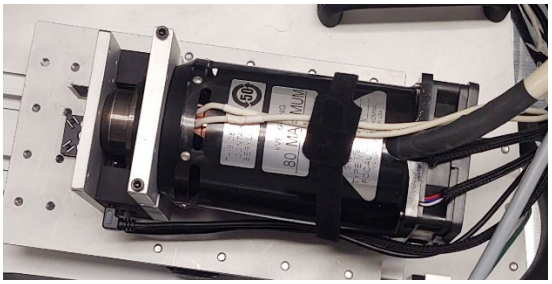
7.8 Will want to start with the 0.5 mm slit and move to a larger slit if your sample is too dilute. A 1.5 mm hex key will be needed to change the slit size. The slit will need to be adjusted on the XES tube (highlighted in red) before inserting your sample holder.



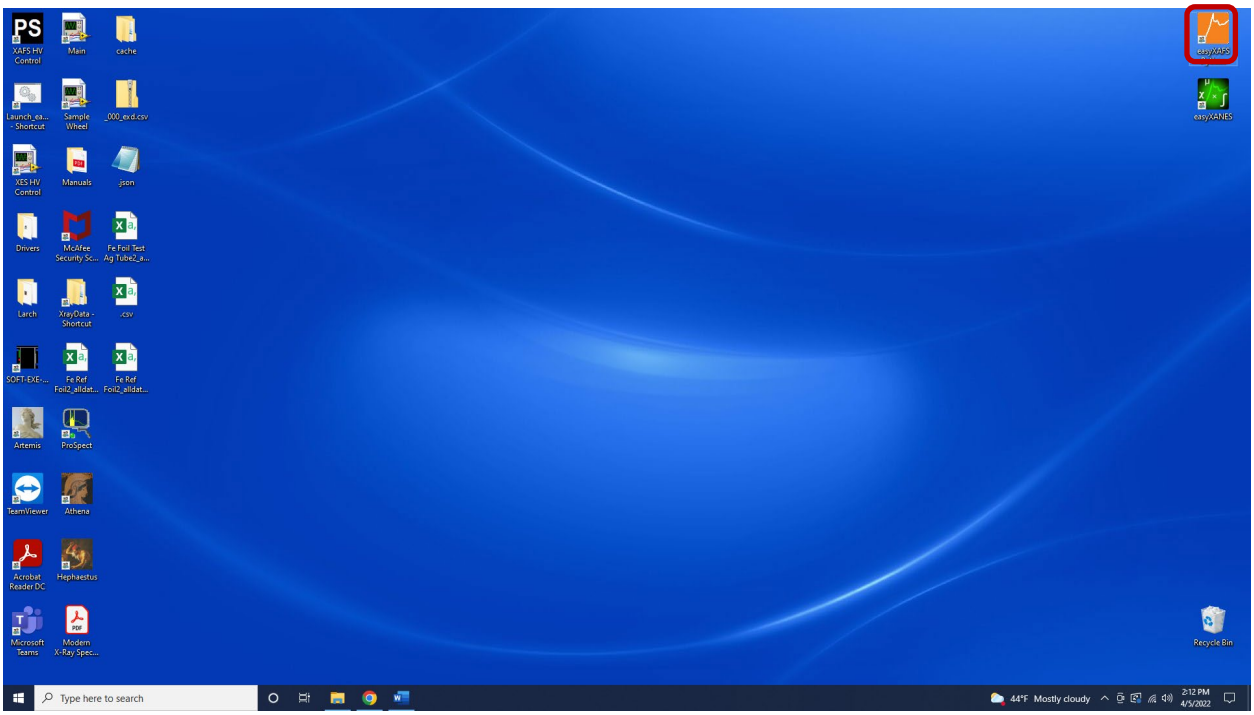
7.9 The sample holder uses a 1.5 mm hex key to hold the sample in place. The sample will need to be centered over the circle in the holder. The sample can also be taped onto the front of the sample holder.



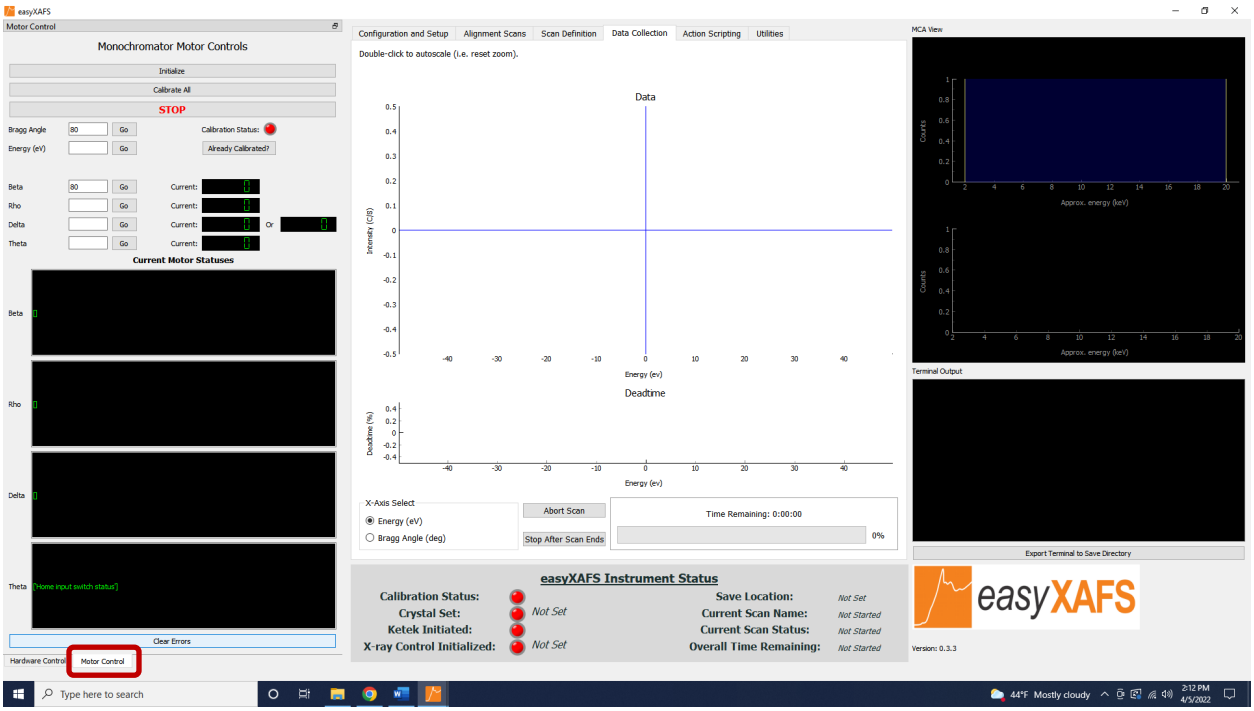
7.10 Then add the sample holder in the magnetic position in front of the XES tube. The picture on the right has the sample holder in place.



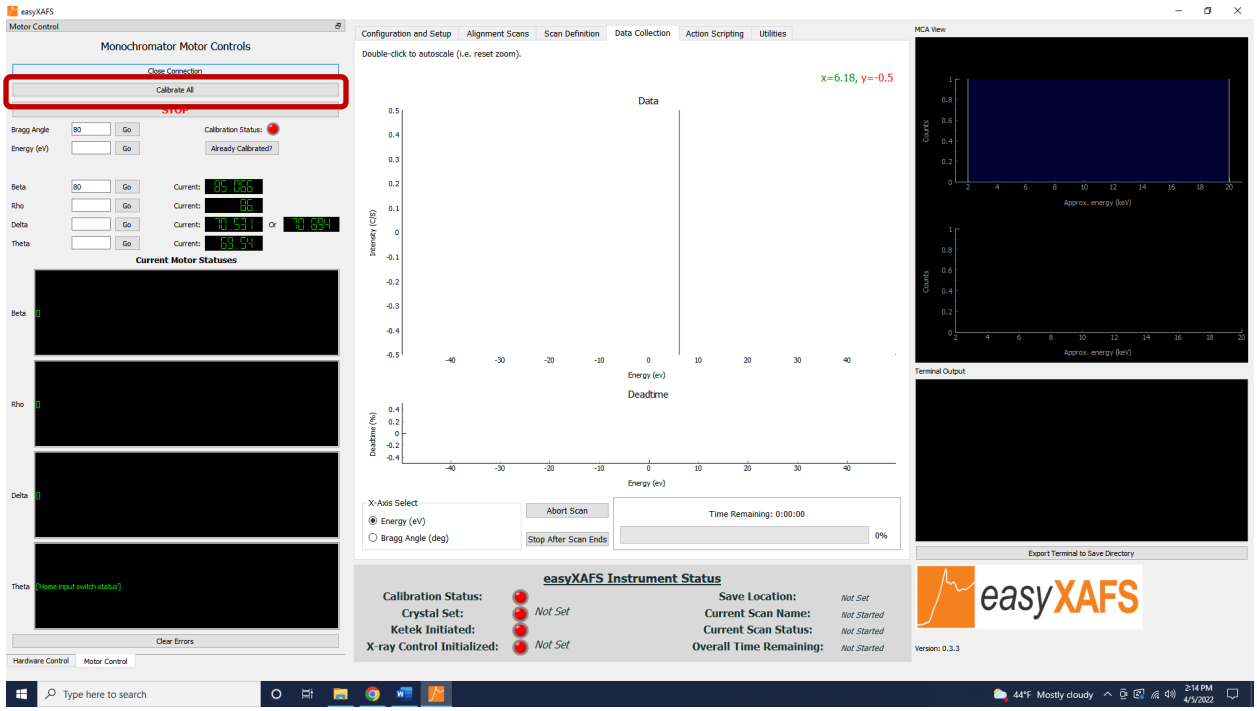
7.11 Open easyXAFS data collection software, if not already open, by double clicking on the icon in the top right of the Desktop.



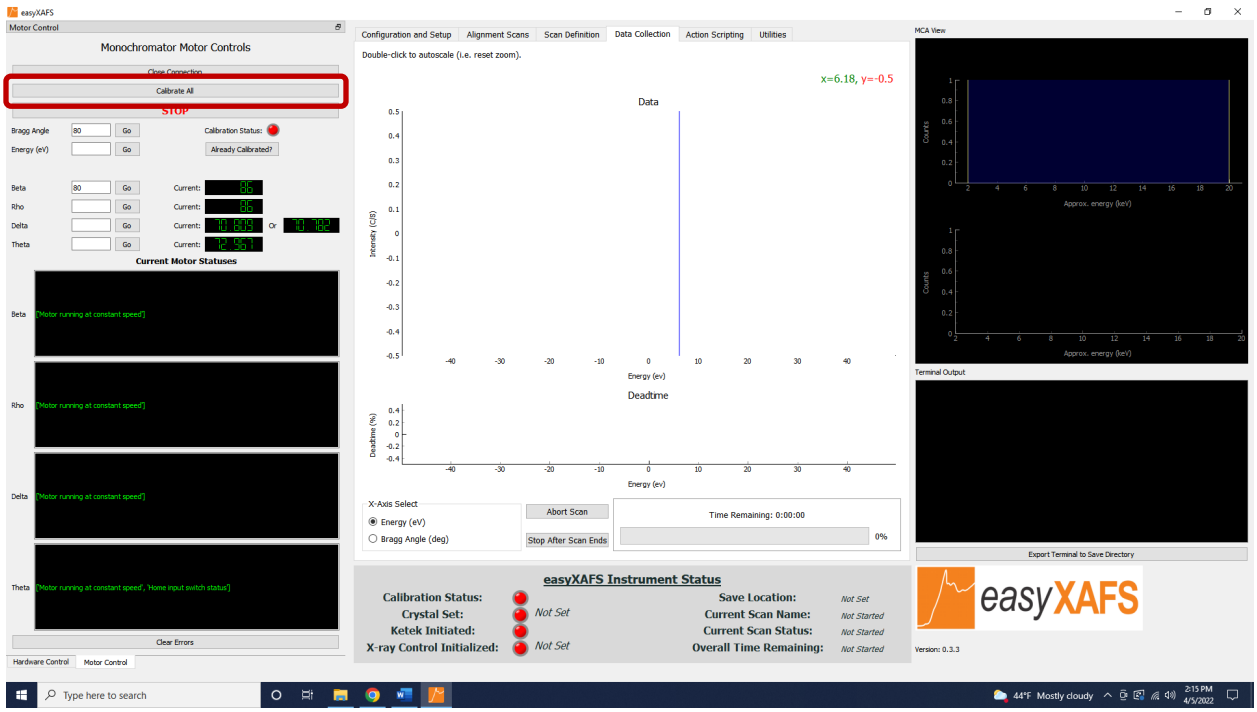
7.12 Switch to the Motor Control tab once the software is open.



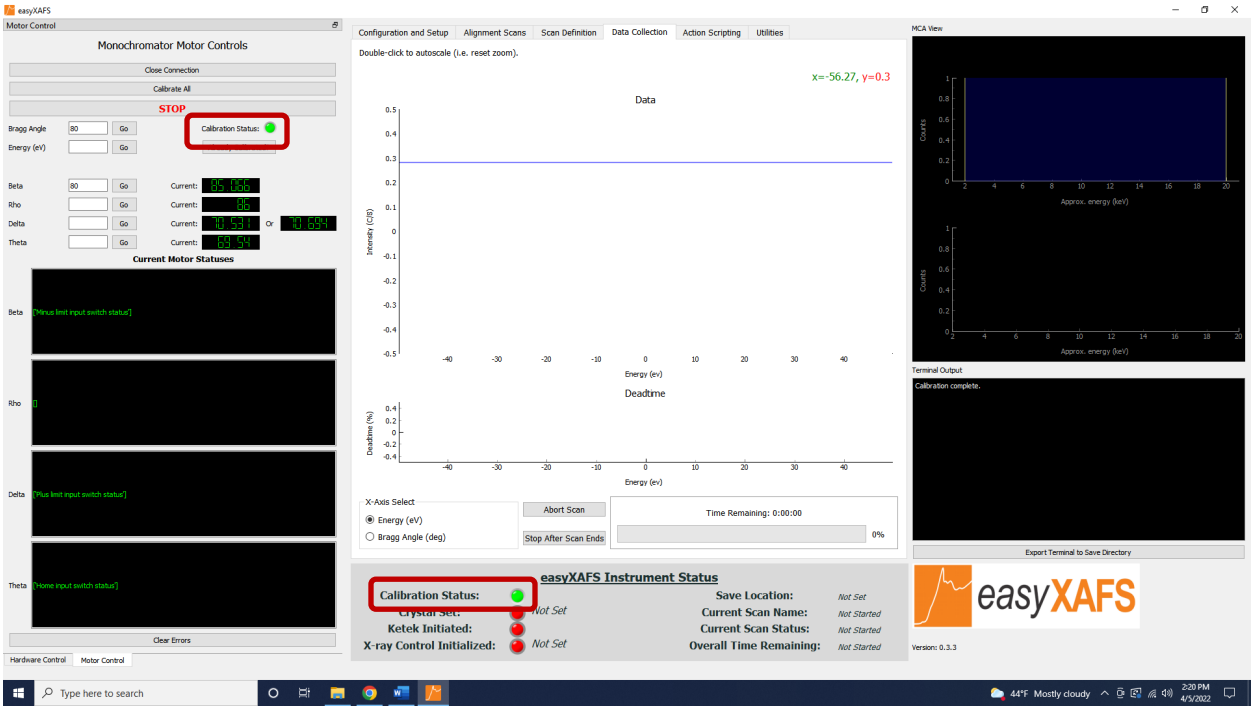
7.13 Initialize the Motors, the button should read Close Connection, when connected.



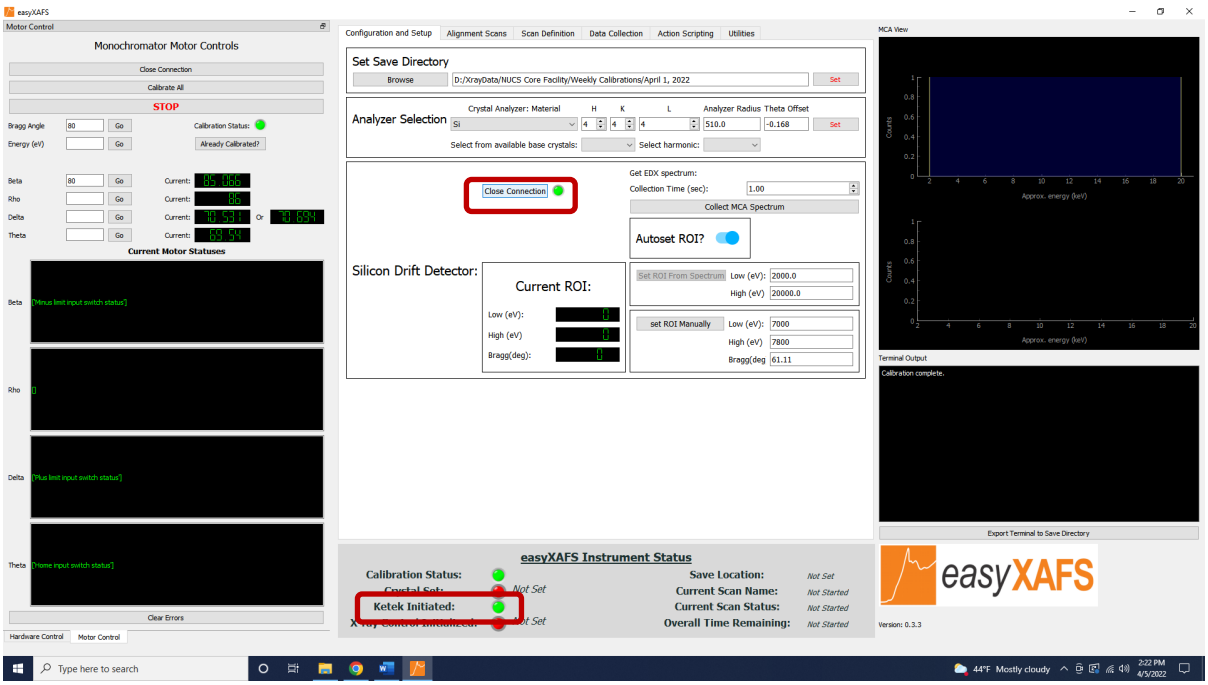
7.14 Then click on calibrate the motors if they have not been calibrated.



7.15 When the motors have been calibrated the Calibration status light will be green. The Calibration Status at the bottom of the screen will also have a light that will be green in color.



7.16 Then initialize the detector by clicking on the Configuration and Setup tab and click on Initialize button in the Silicon Drift Detector Section. The light will become green. The light next to the Ketek Initiated will also become green.

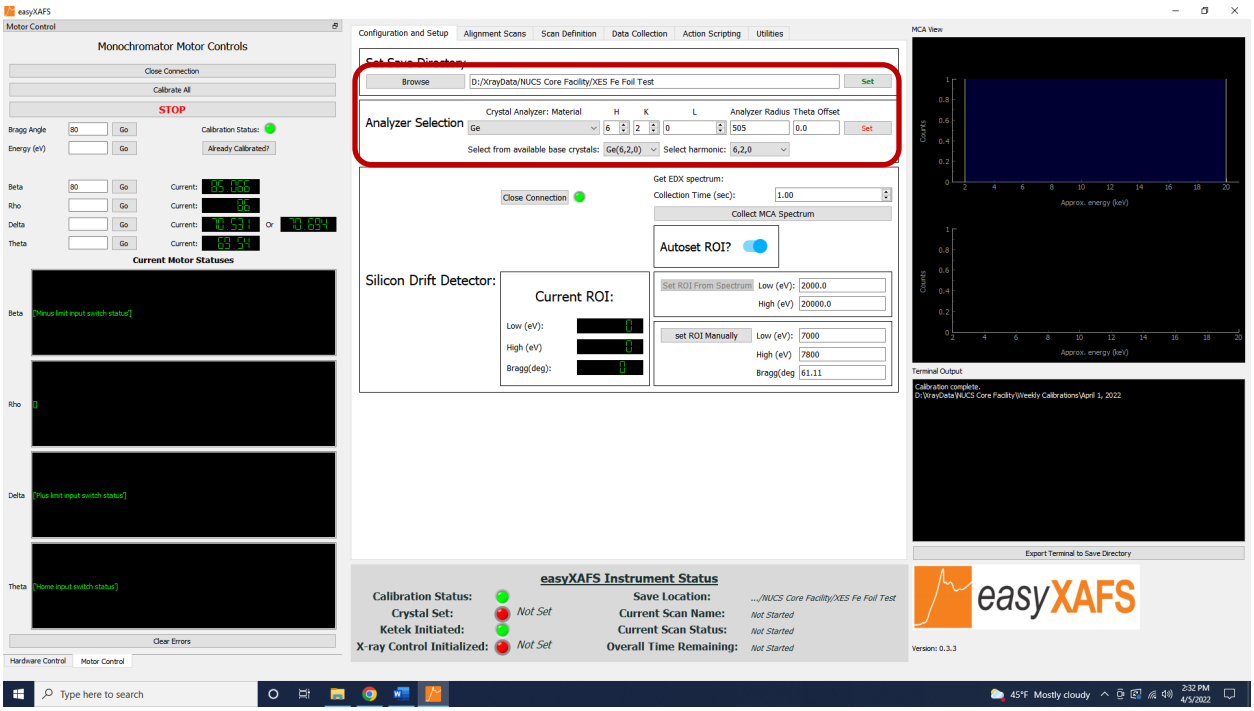


7.17 The sample location can be adjusted with the screw located on the back of the sample holder. Want the sample aligned behind the slit for the best counts and best resolution.

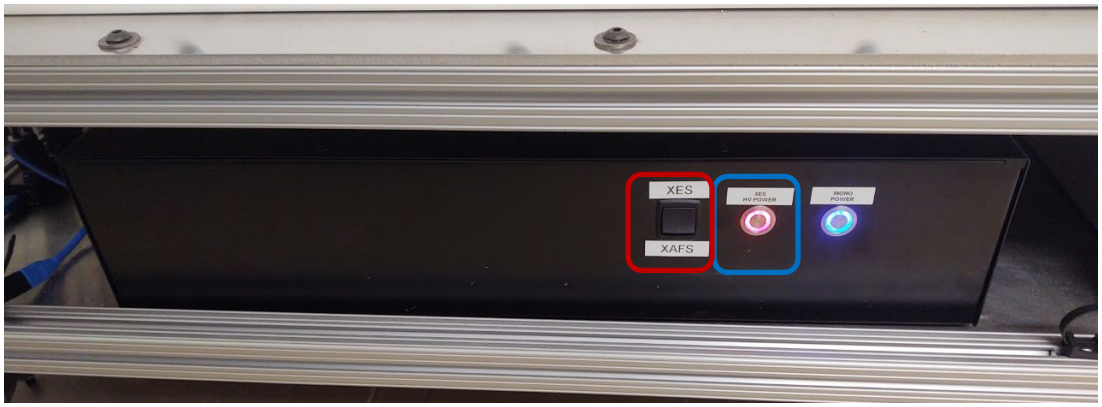
7.18 Will want to collect some data and look at the count rate, then do two ¼ turns and check again, want the highest counts.

7.19 If count rate goes up, then the sample is in a better position

7.20 Set the save Directory and the Crystal Analyzer

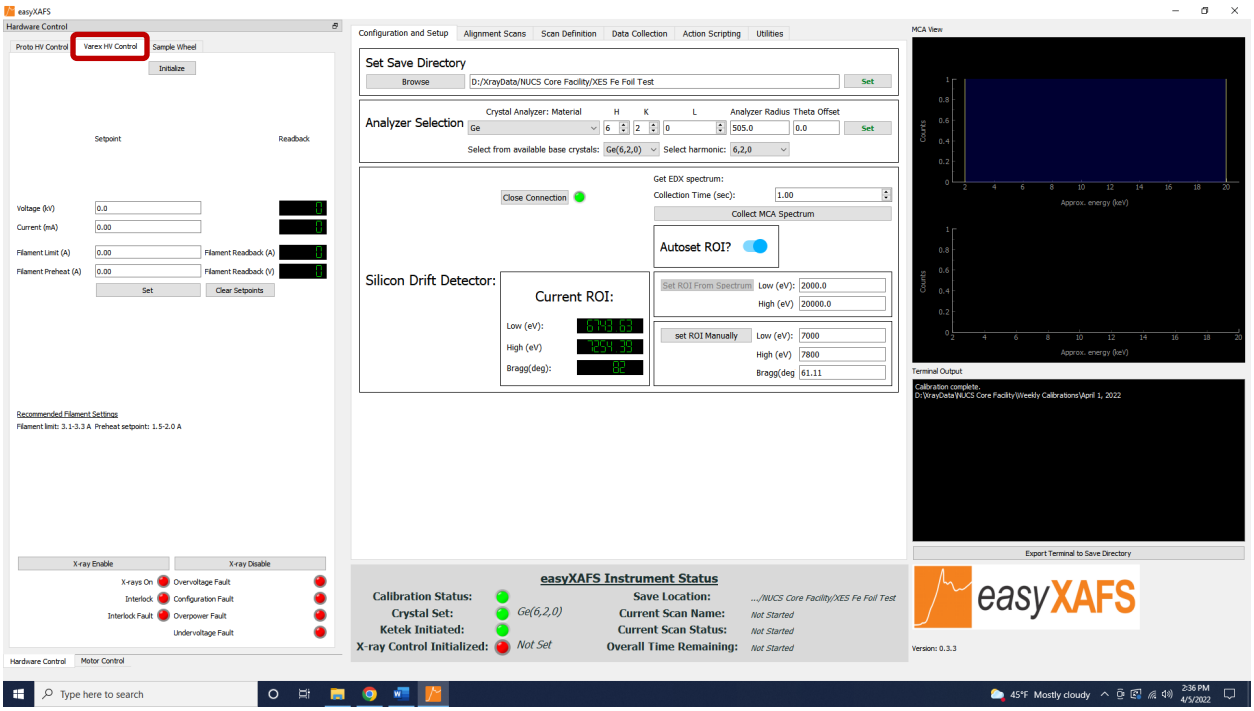


7.21 Before Collecting data, the door to the instrument needs to be closed and the power to the XES power needs to be turned on (highlighted in blue). A red light will turn on when the button is pushed.

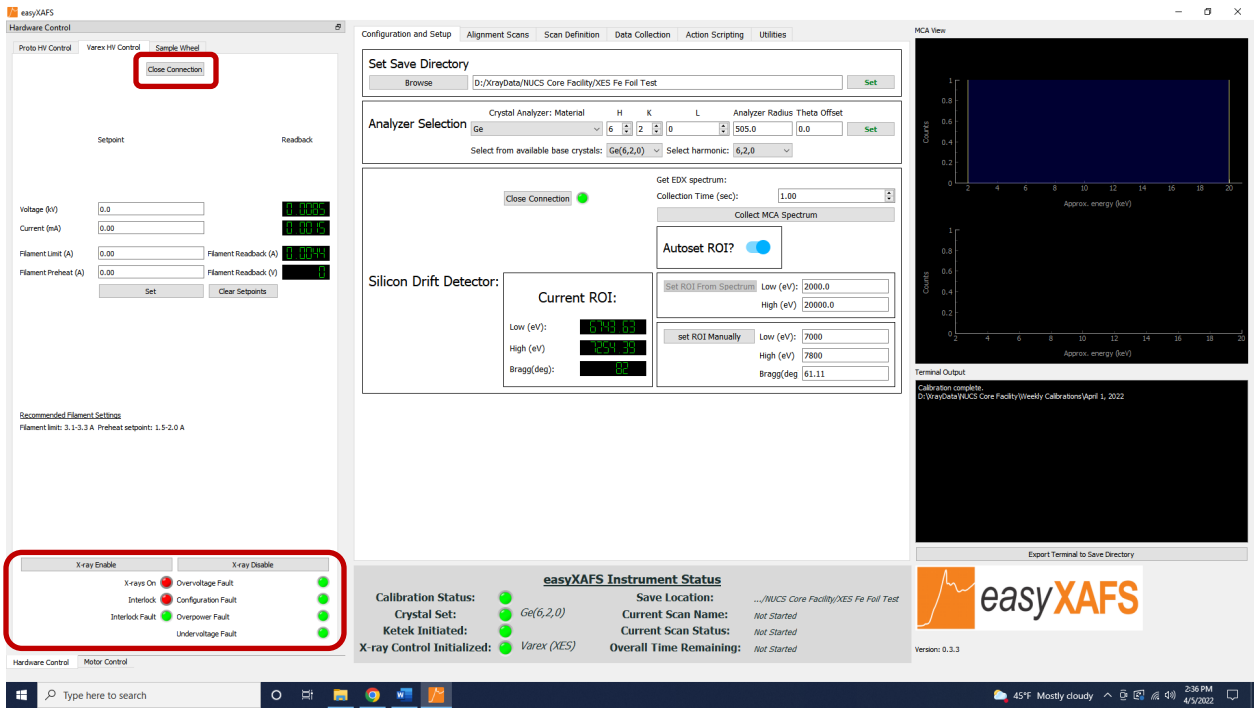


7.22 Then flick the switch on the electronics box from XAFS to XES (highlighted in red in above picture).

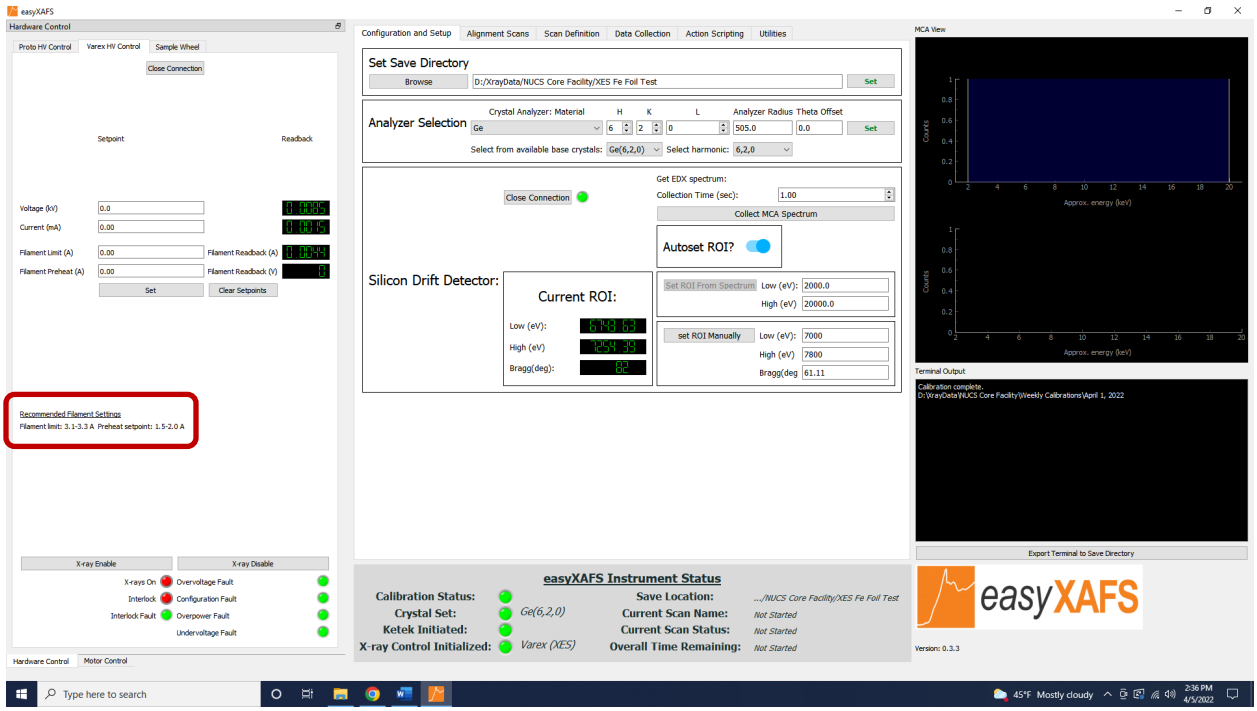
7.23 At the software, select Varex HV Control to work the XES Tube, under Hardware Control



7.24 Click on initialize to turn on the X-ray tube. The button will read Close Connection when activated. Several lights on the bottom of the screen will also be light up green in color.

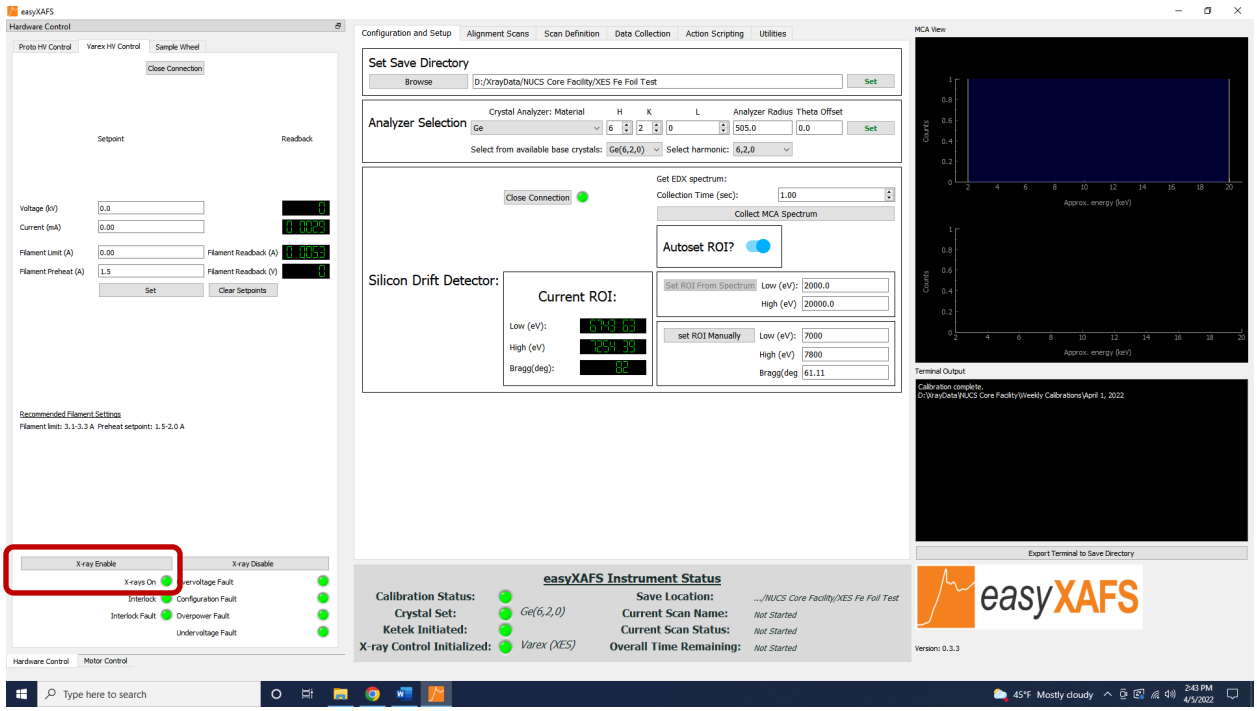


7.25 The preheat setpoints and filament limits are shown in the software.



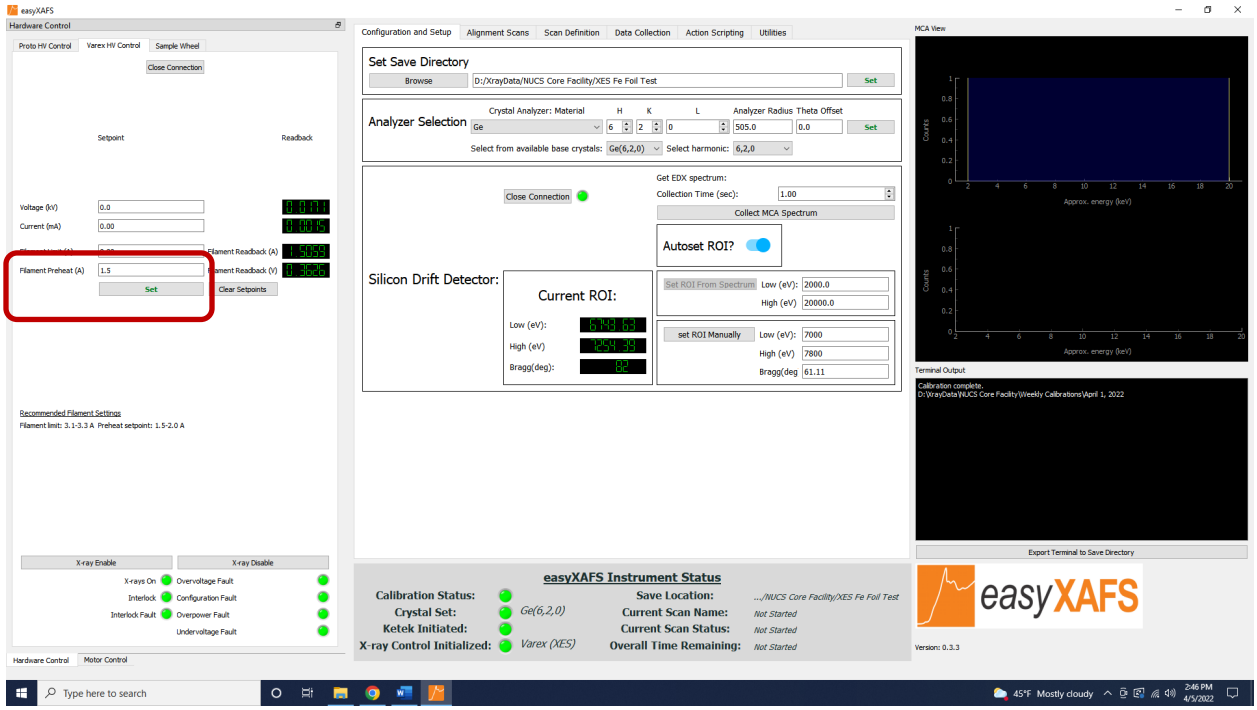
7.26 If the mode on the electronics box is not on XES mode, you will not be able to ramp up the power on the X-ray tube.

7.27 Then click on X-ray Enable, all of the lights should be green, and the X-rays On light should be green. Enabling the X-rays also opens the shutter to the XES tube.

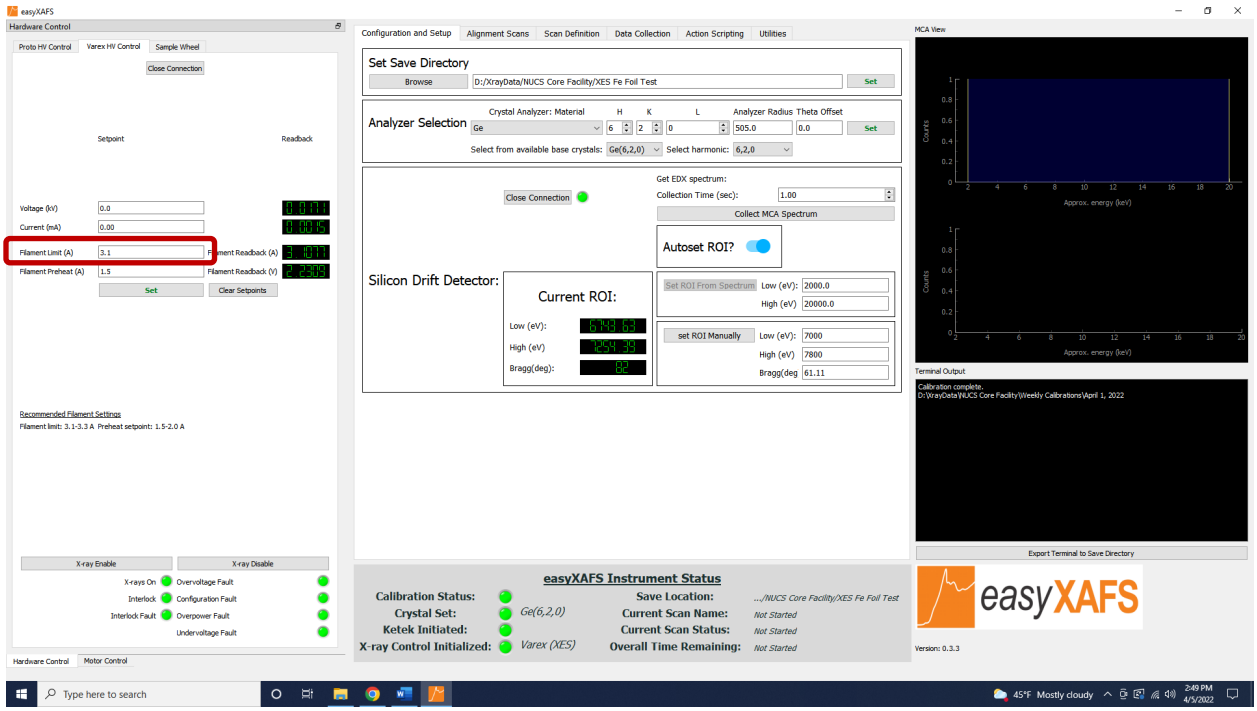


7.28 The XES light indicator for the instrument should be illuminated, indicating that the X-ray tube is powered up and the shutter is open.

7.29 Then enter a Filament Preheat of 1.5 A and click on Set. The Set button should turn green and the Preheat value should always be 1.5 A.



7.30 The Filament limit should be set to 3.1 A. Click on the Set button to accept the value.

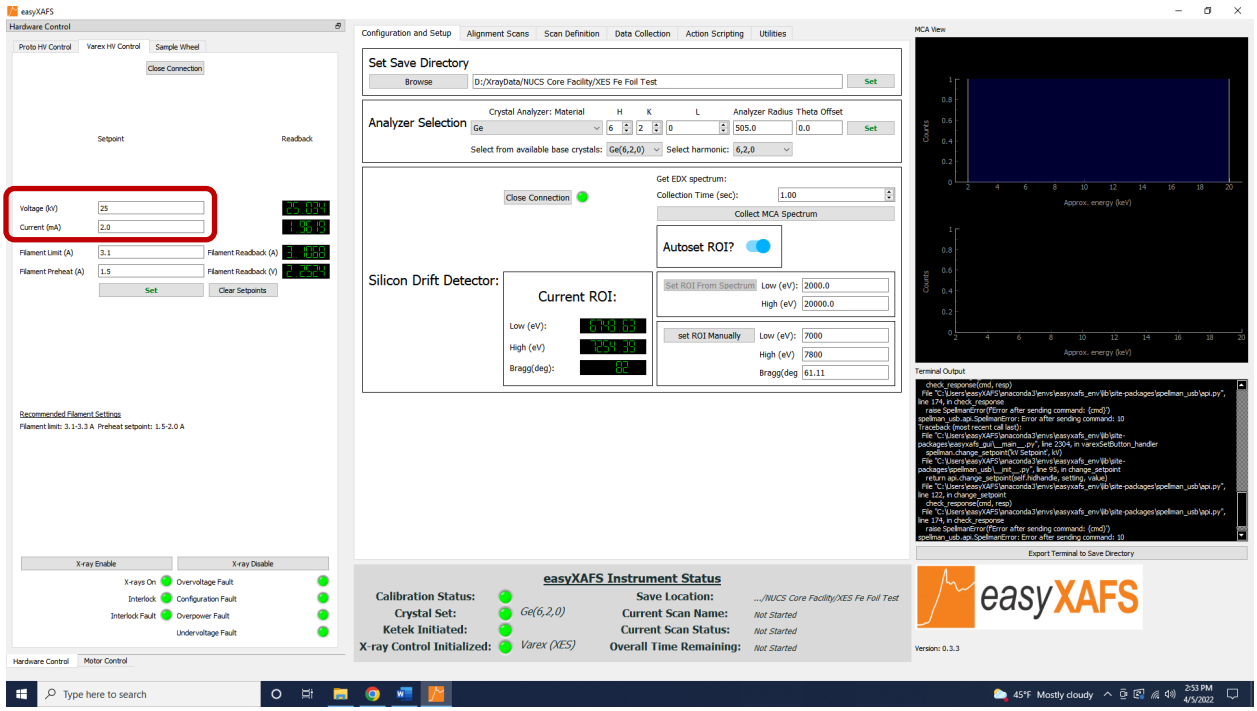


7.31 Max power on the XES tube is 100 W.

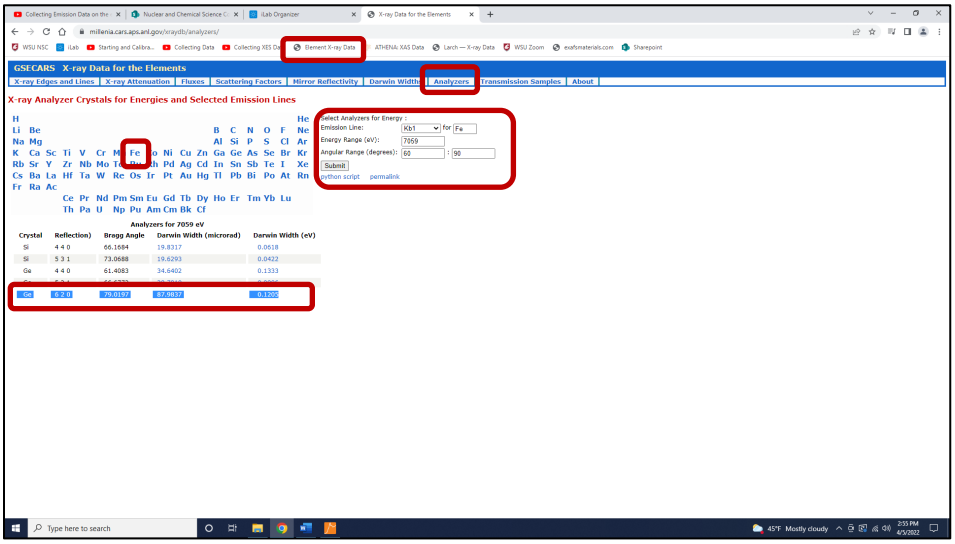
7.32 The XES tube does not have a warm up procedure.

7.33 The max voltage for the XES tube is 35 kV. The combination of voltage and current cannot exceed 100 W.

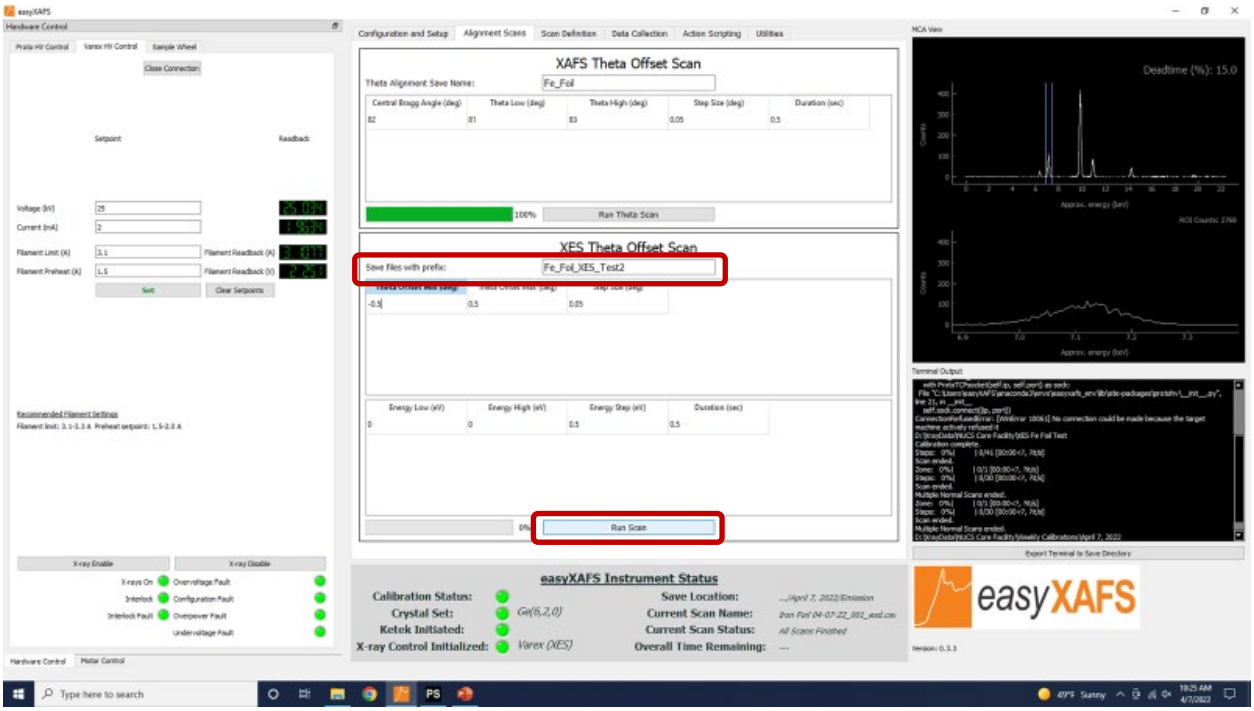
7.34 A value of 25 kV and 2.0 mA is a good place to start. Click on the Set button to accept the values.



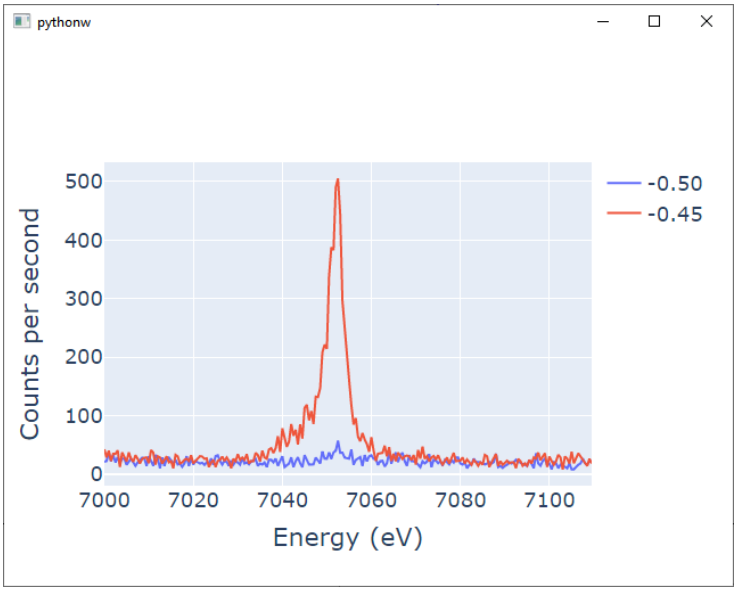
7.35 To determine the analyzer, go to X-ray data for elements and select analyzers and the element of choice: Fe in this case, then Kβ1 and see that the best choice is the Ge 620 crystal. Usually the higher Bragg angle is the best choice.



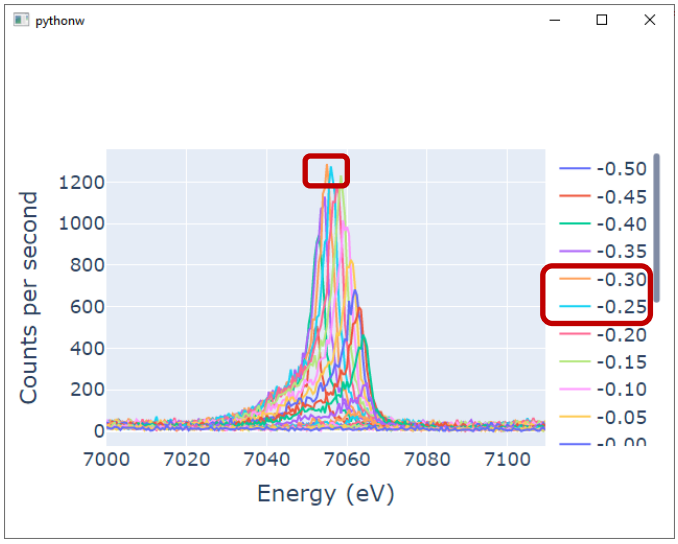
7.36 Click on an alignment scan. And then add a filename and then click on Run Scan under the XES section. A sample should not be in the instrument at this time.



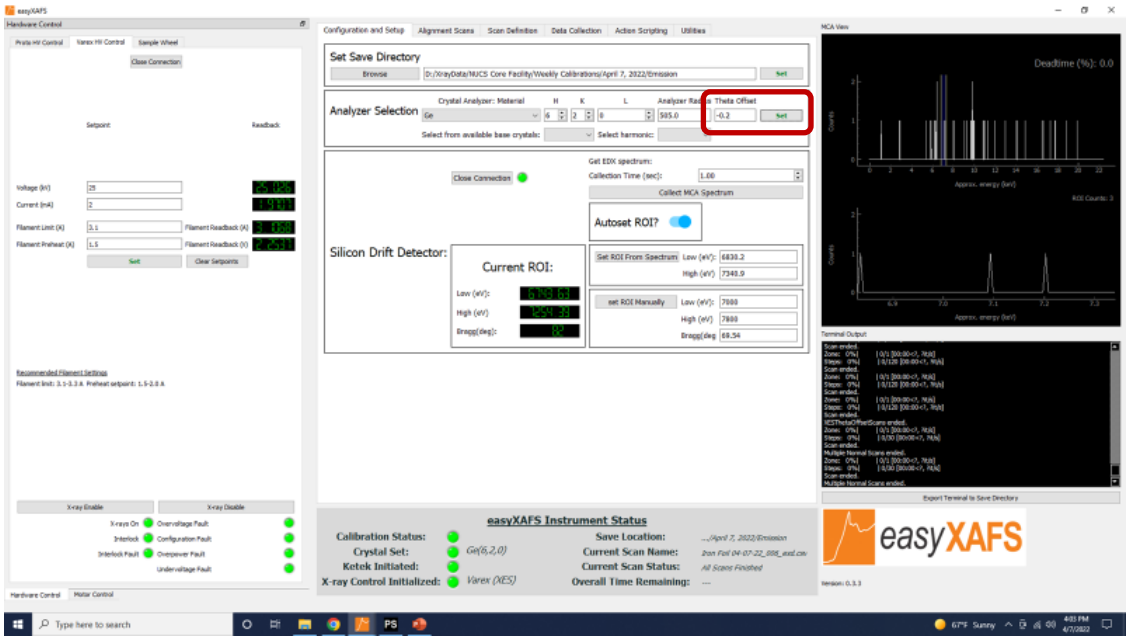
7.37 After running for a little bit (2-3 minutes), a window will pop up where the alignment scans will show up.



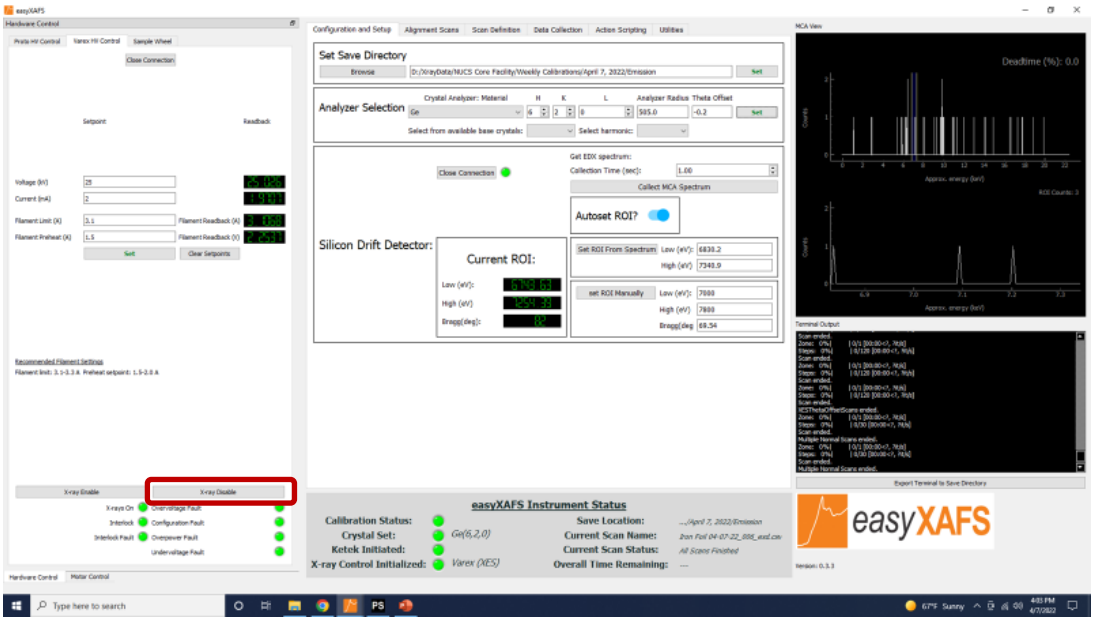
7.38 Using the default settings will take about 45 minutes to complete the alignment scans and a plot looking similar to the plot below will result. Changing the Step Size to 0.10 or 0.20 in Step 7.36 can greatly reduce the time for the Theta offset data collection. To determine the Theta offset, pick the peak that has the largest number of counts. A choice of -0.30 or -0.25 would be the best for the plot below. If a peak is not seen after a few scans, trying placing the reference foil into the sample holder in the spectrometer and repeating the theta scan. If peaks are still not seen, then the region of interest is incorrect or the best theta offset value is outside the default values and a new window or theta offset upper and lower bounds (try +1.5 to -1.5) needs to be chosen.



7.39 Enter the Theta Offset value in the Analyzer Section of the Configuration and Setup tab and click on the Set button.

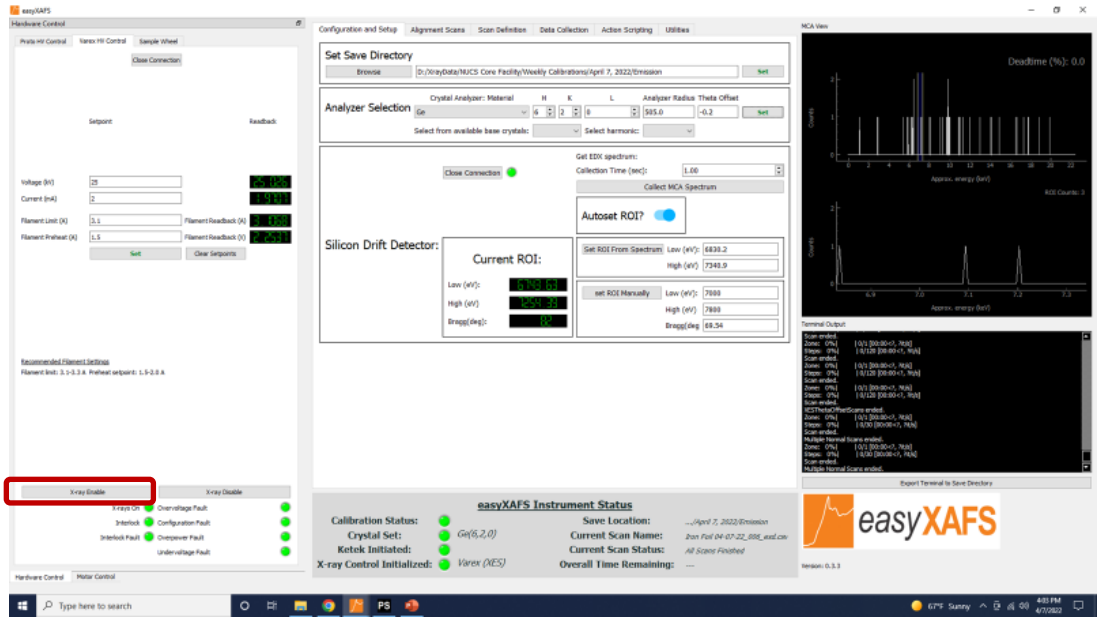


7.40 After setting the Theta Offset. Click on the X-ray Disable button to close the shutter and turn off the X-ray tube. The indicator for the X-ray emission X-ray tube should turn off.

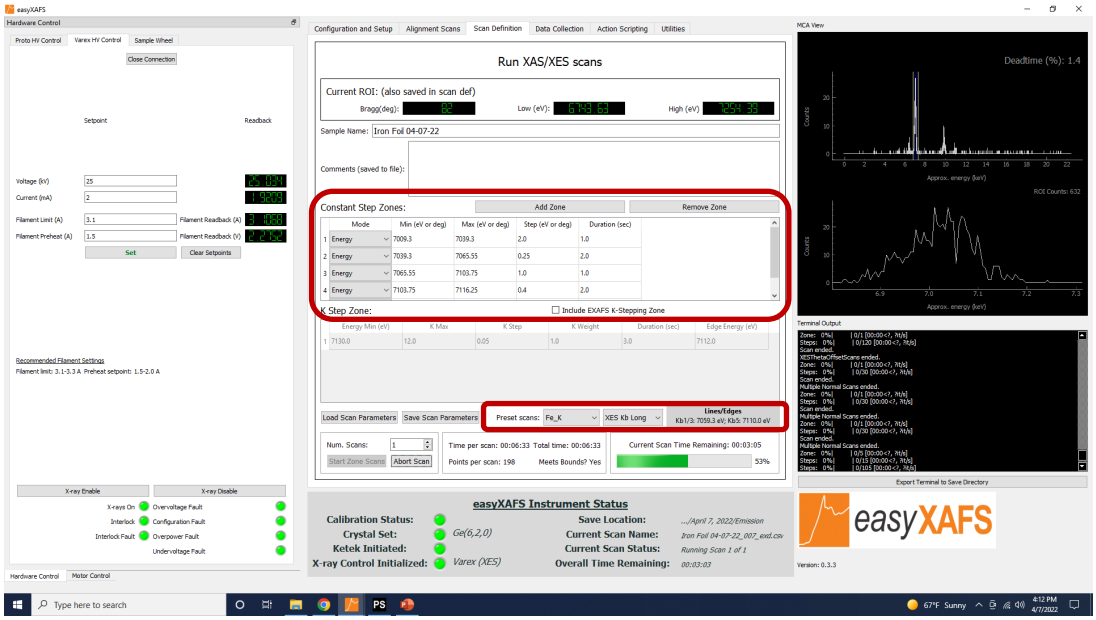


7.41 Insert the sample in the instrument as shown in Step 7.10.

7.42 Click on the X-ray Enable button to open the shutter and turn on the X-ray tube. The XES indicator light should turn red.



7.43 Then, click on the Scan Definition tab and select the zones to be analyzed. The Preset scan can be set to XES Kβ long, which is about a 7 minute scan. If other parameters are desired, they can be adjusted the parameters in the Constant Step Zone Section (highlighted in red).



7.44 Click on Start Zone Scan to begin the data collection.

7.45 When the scans have completed, an emission spectrum will be shown on the screen. The emission spectrum below is for an iron foil.

7.46 If the analysis of additional samples is desired, repeat Steps 7.40 – 7.45 as needed. Note: a new Theta scan is only needed if the crystal is changed.

7.47 Before shutting off the instrument, a background spectrum without the sample present will need to be collected.

7.48 To return to XAFS mode, click the switch on the Electronics box back to XAFS and turn off power to the XES tube by pressing the XES power button, which will turn it off.

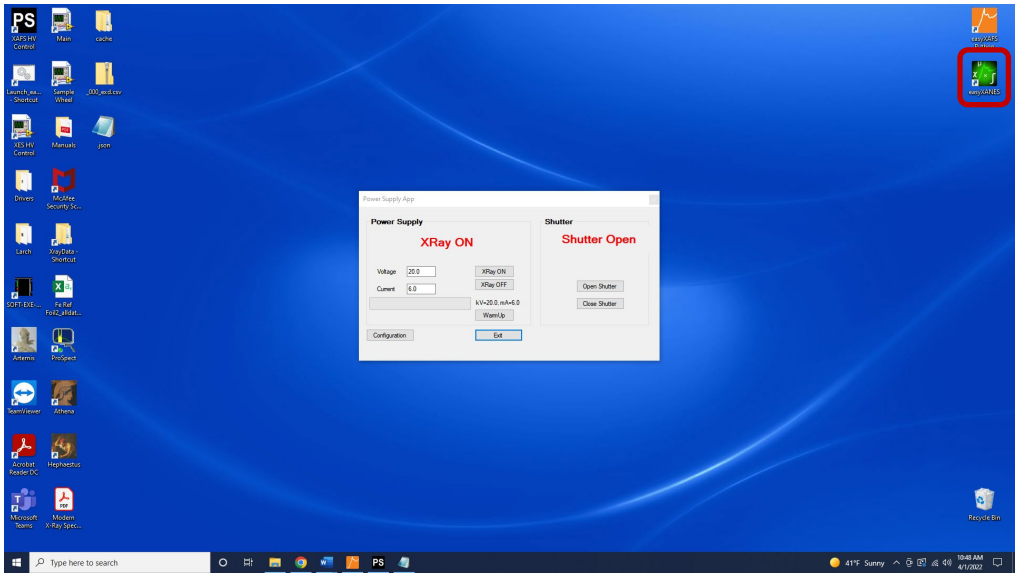
7.49 Remove the collimator as shown in Step 7.6 and move the X-ray tubes back to their original positions, following Steps 7.2 – 7.4, so that the instrument matches what is shown in Step 7.1.

7.50 See Section 9 for instructions on how to turn off the spectrometer.

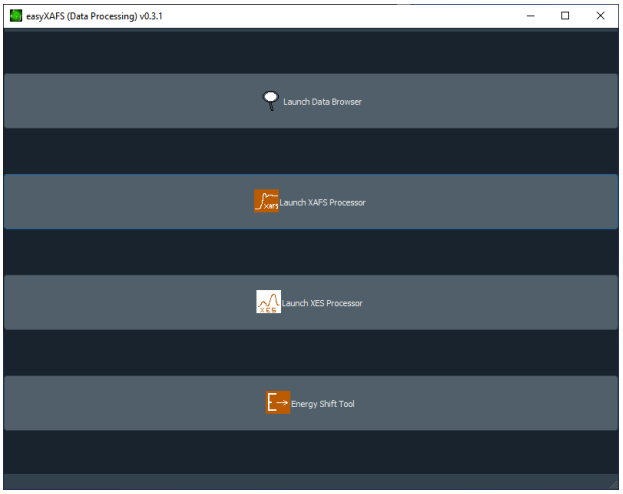
8 Data Analysis on the easyXAFS 300+ X-ray Spectrometer

8.1 To work up easyXAFS data, the instrument vendor provides a program to work up the collected data.

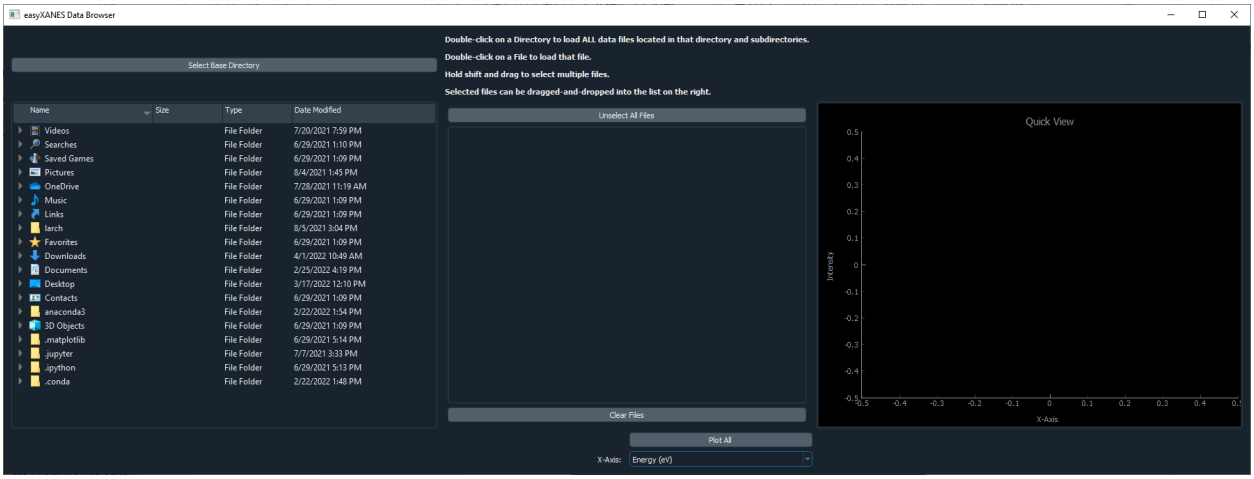
8.2 To open up the data processing software double click on the easyXANES icon on the top right of the Desktop.



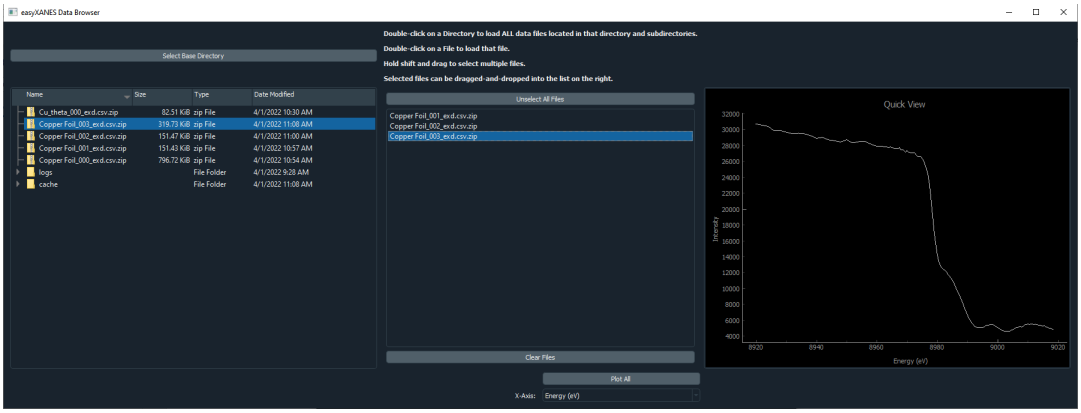
8.3 The following program will open.



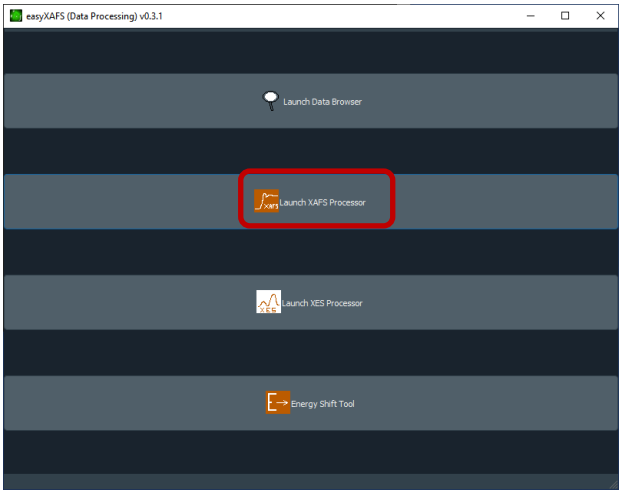
8.4 Clicking on the Launch Data Browser will cause the following window to open:



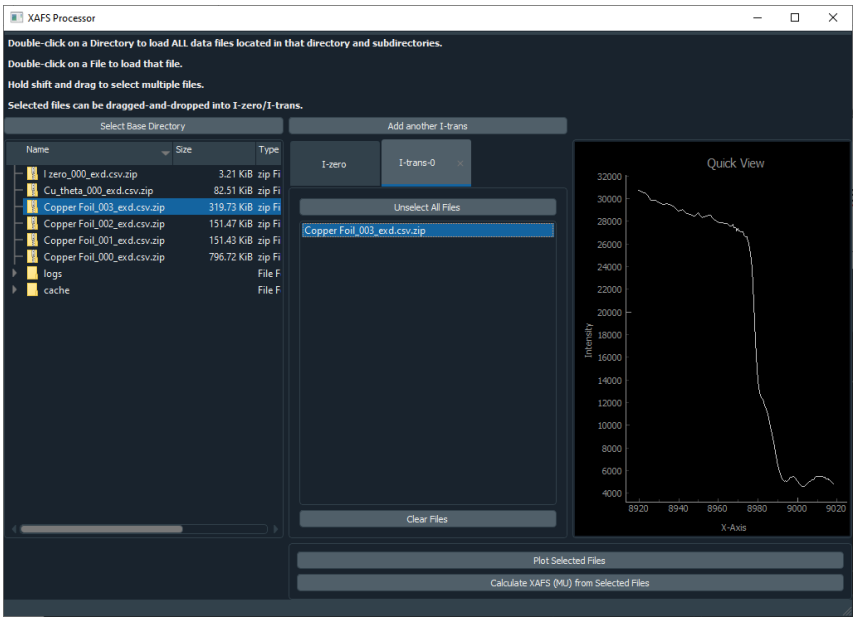
8.5 To visualize the raw data collected, click on the easyXANES DataViewer, which will open a new tab.



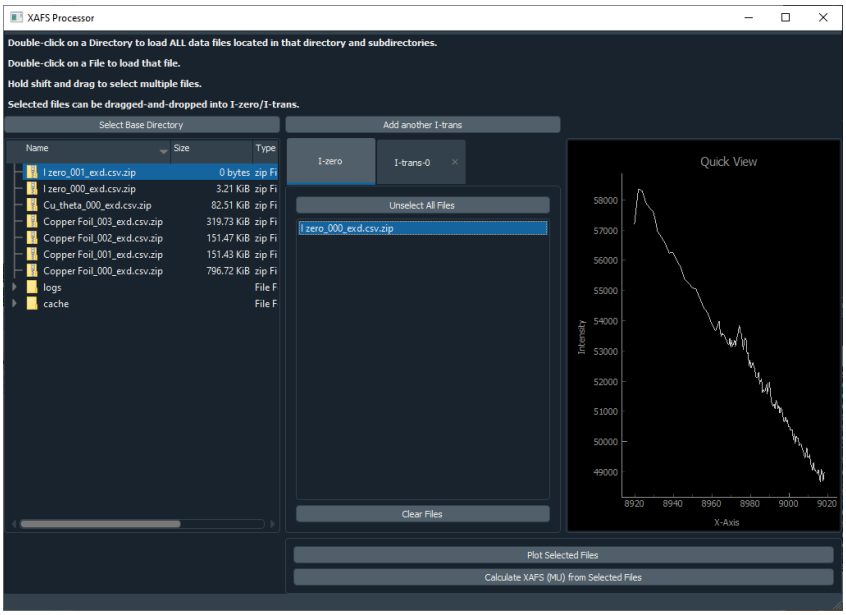
8.6 To process XAFS data, click on the Launch XAFS Processor icon (highlighted in red).



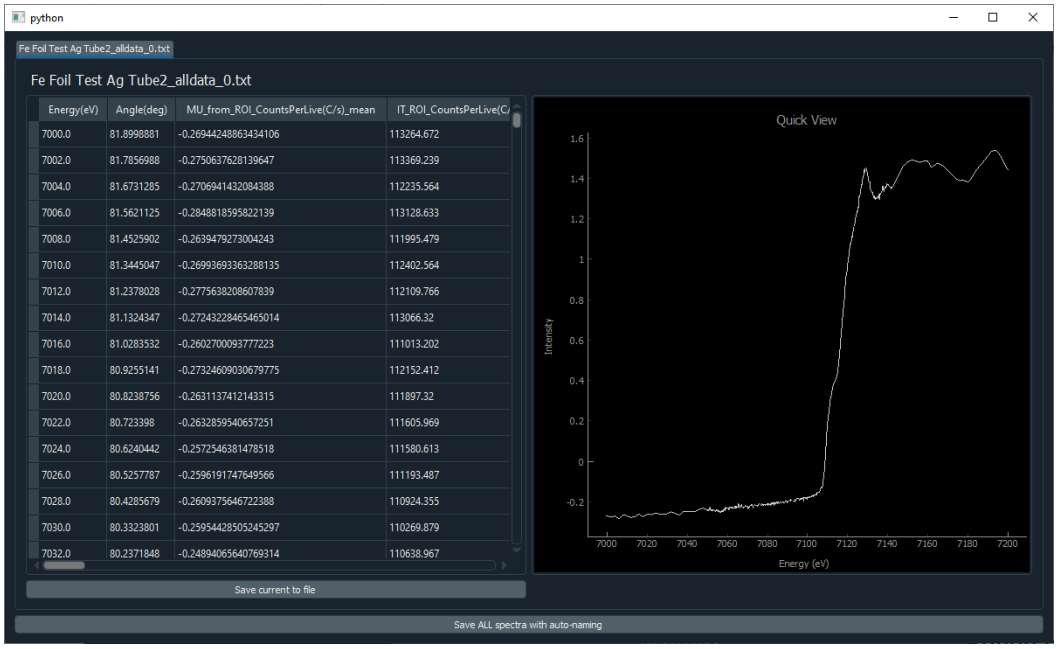
8.7 When the XAFS Processor is loaded, a data and an I_0 file will need to be selected prior to creating a mu plot. Start by selecting a trans file, click Plot selected files to get a quick view of the spectrum to make sure that the correct spectrum is plotted.



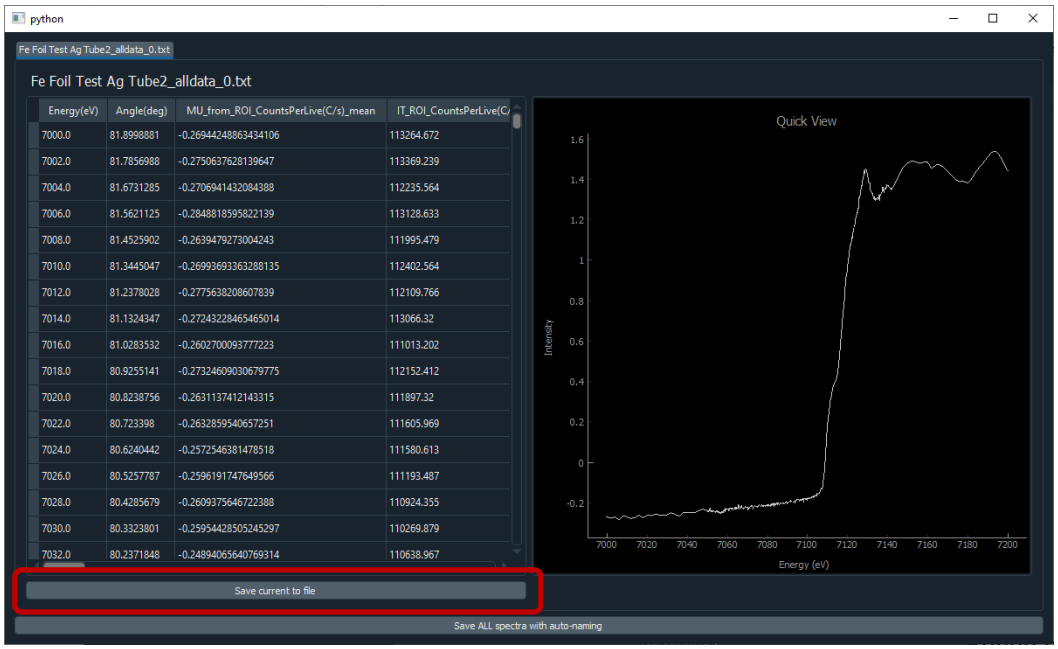
8.8 To view the data, click on Load Files button, select the files to be viewed and click on the Plot Selected Files button to view the data plots.



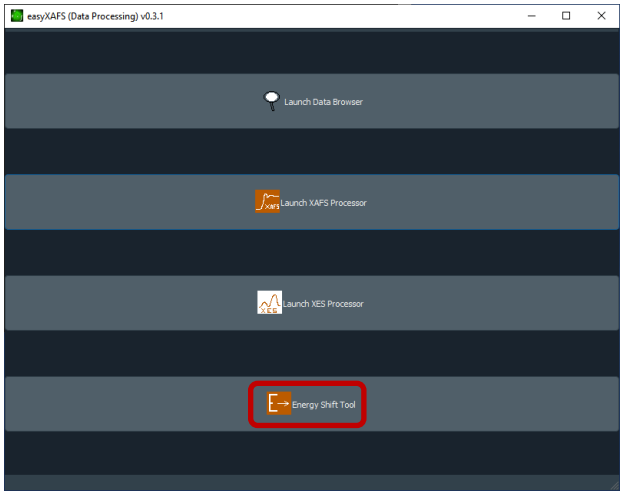
8.9 To view the mu Plot, click on the Calculate XAFS (MU) from Selected Files button, and a window showing the data and the mu plot will show up.



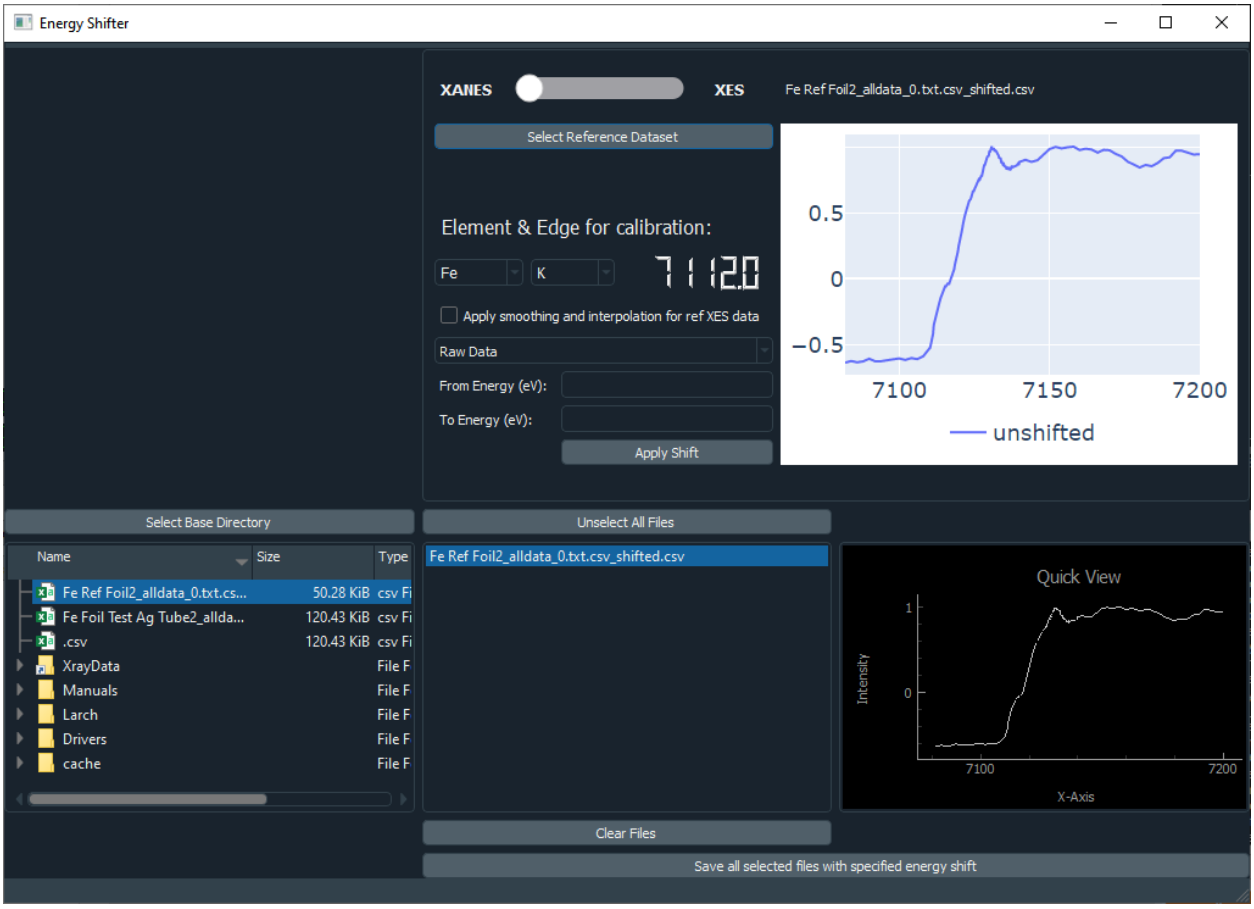
8.10 To save the mu plot, click on the Save current to file button (highlighted in red), and save the data as a .csv file. Right clicking on the plot will allow for the unshifted mu plot to be saved as an image (needed for weekly calibrations).



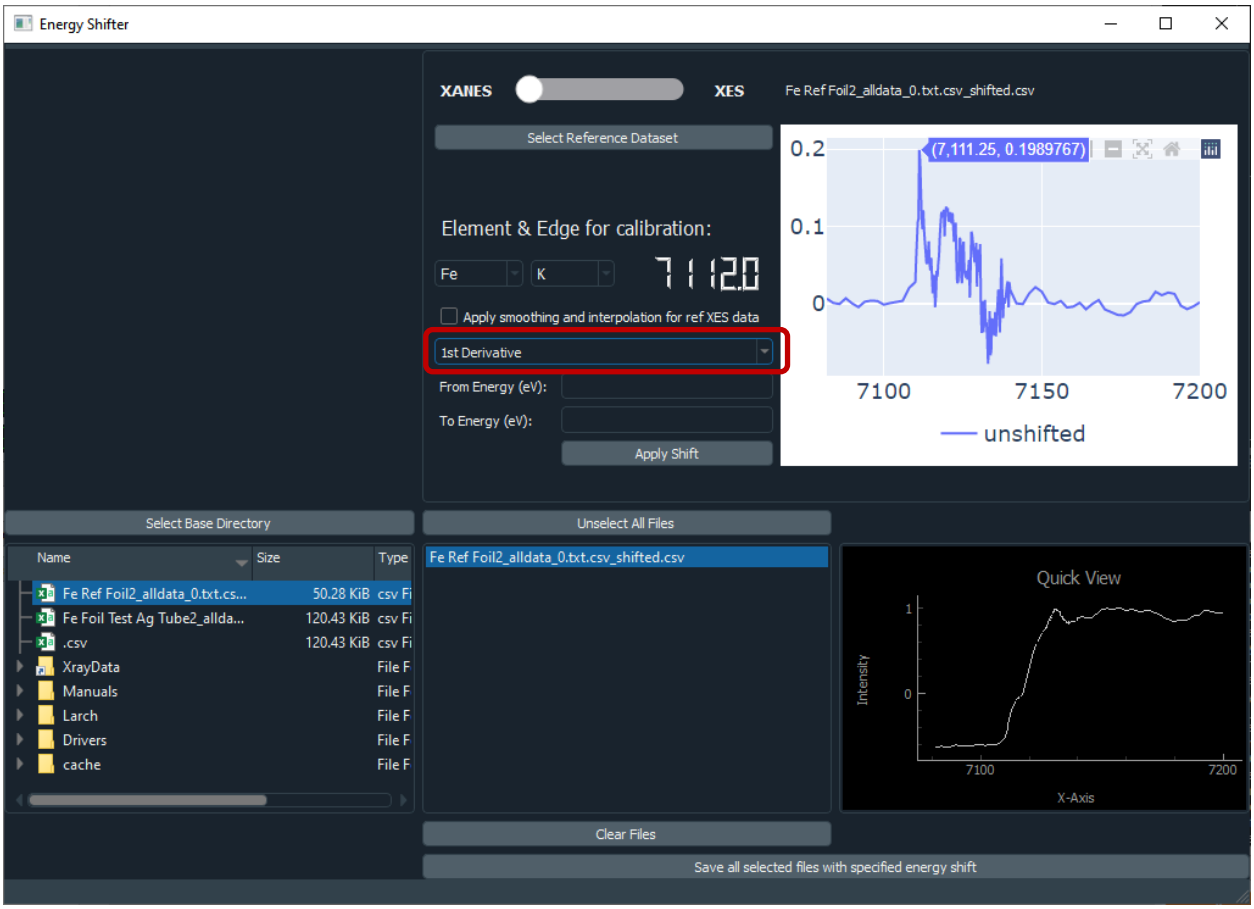
8.11 After saving the mu plot, the data in the file will need to be shifted in regards to a reference. To do this, click on the Energy Shift Tool (highlighted in red).



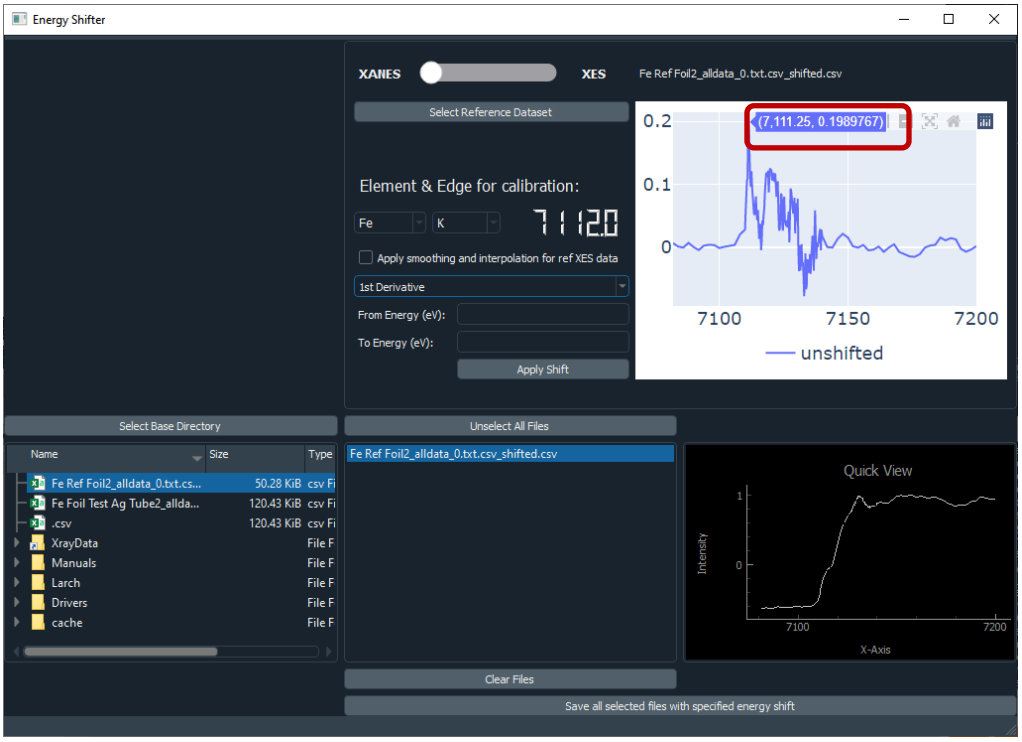
8.12 A window will then open and the .csv file generated in Step 8.11 will need to be selected. When selected, the mu plot will be seen in the upper right of the window.



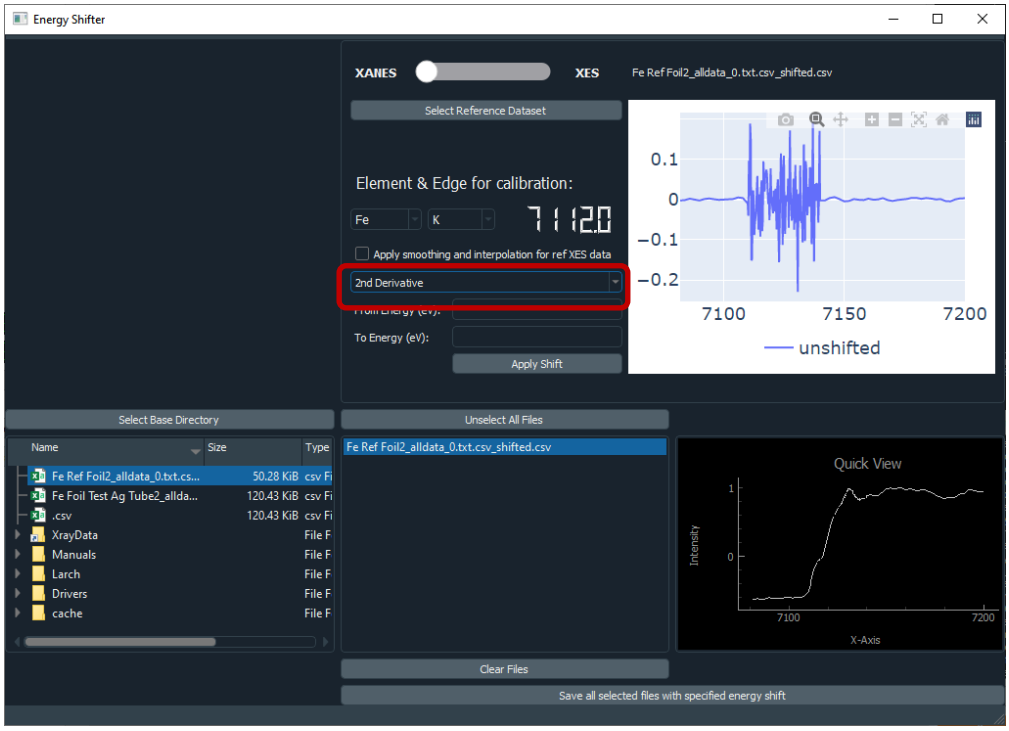
8.13 To determine the amount of energy to shift the spectrum, the first derivative of the mu plot can be taken by changing from Raw data to 1st Derivative (highlighted in red below).



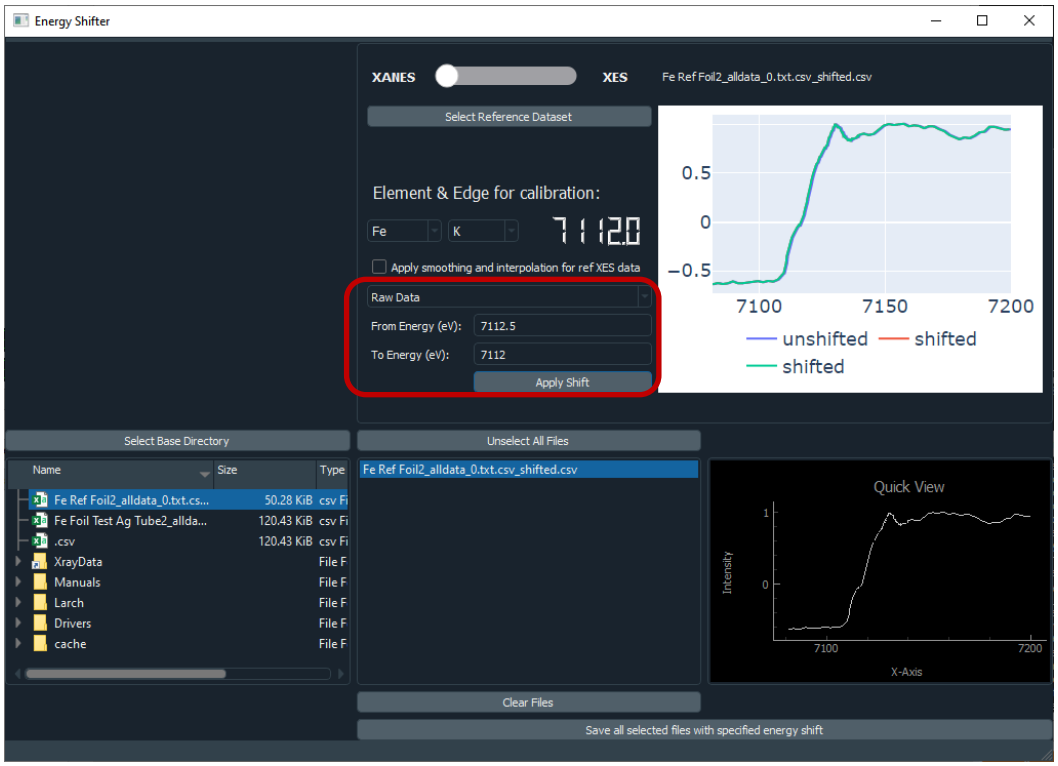
8.14 The maximum value can be determined by moving the cursor over the highest peak and the energy value is given as the x-value (highlighted in red).



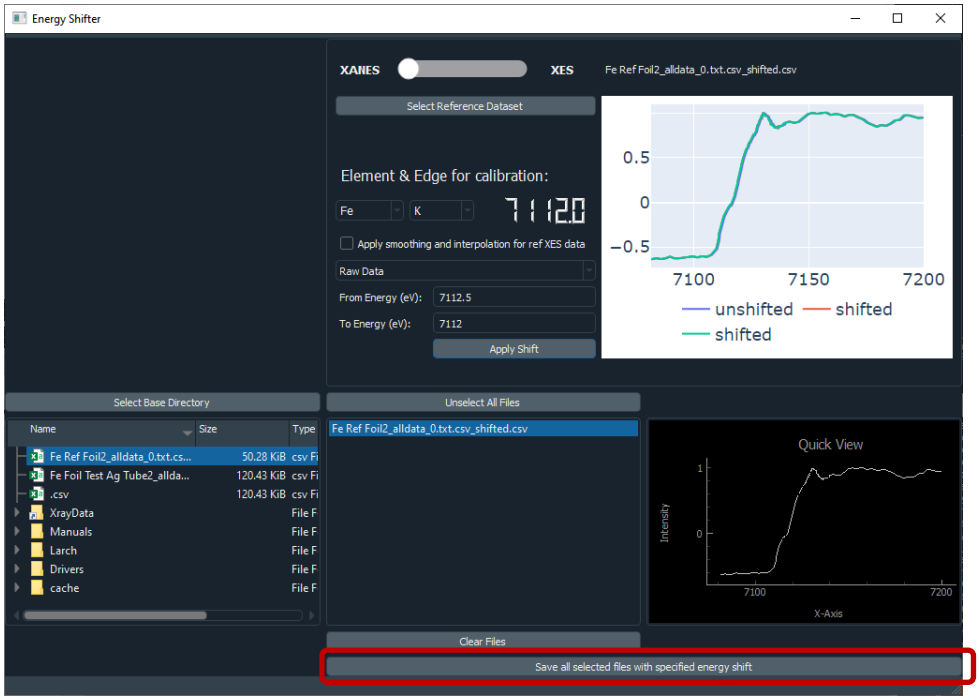
8.15 If desired, the 2nd Derivative can also be examined by changing the 1st Derivative option to 2nd Derivative (highlighted in red below).



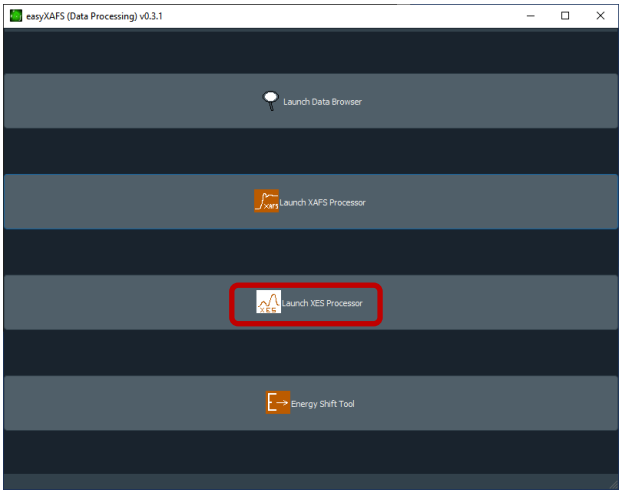
8.16 Using the value of the shift for the mu plot from the 1st derivative, the value can be entered as the from energy and the To Energy can be set to the calibration energy. Clicking on the button to Apply shift results in a mu plot at the shifted energy.



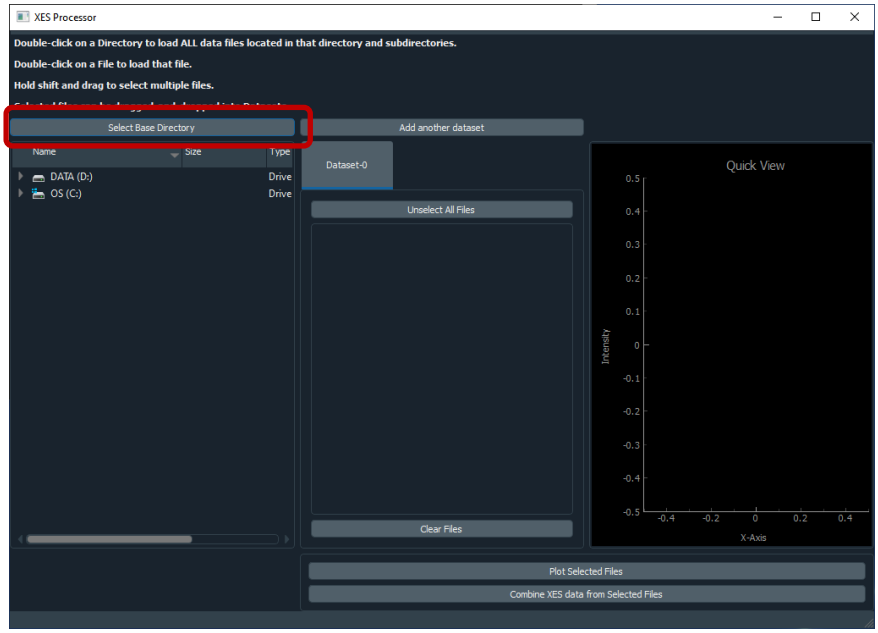
8.17 The shifted mu plot is saved by clicking on the Save all selected files with specified energy shift button (highlighted in red).



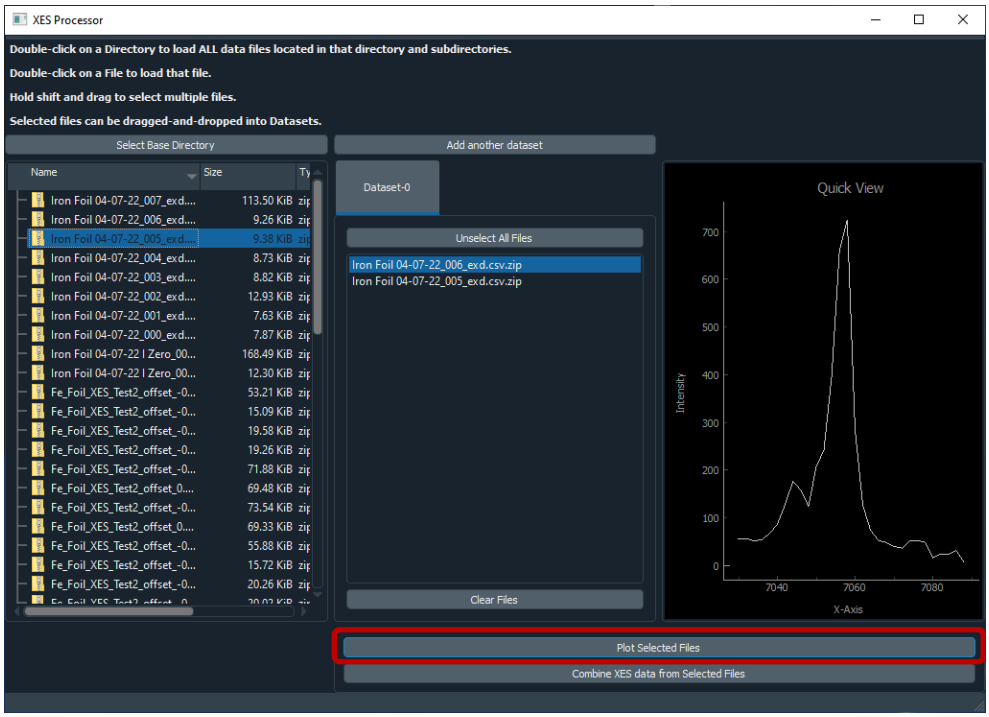
8.18 To analyze XES data, click on the Launch XES Processor option.



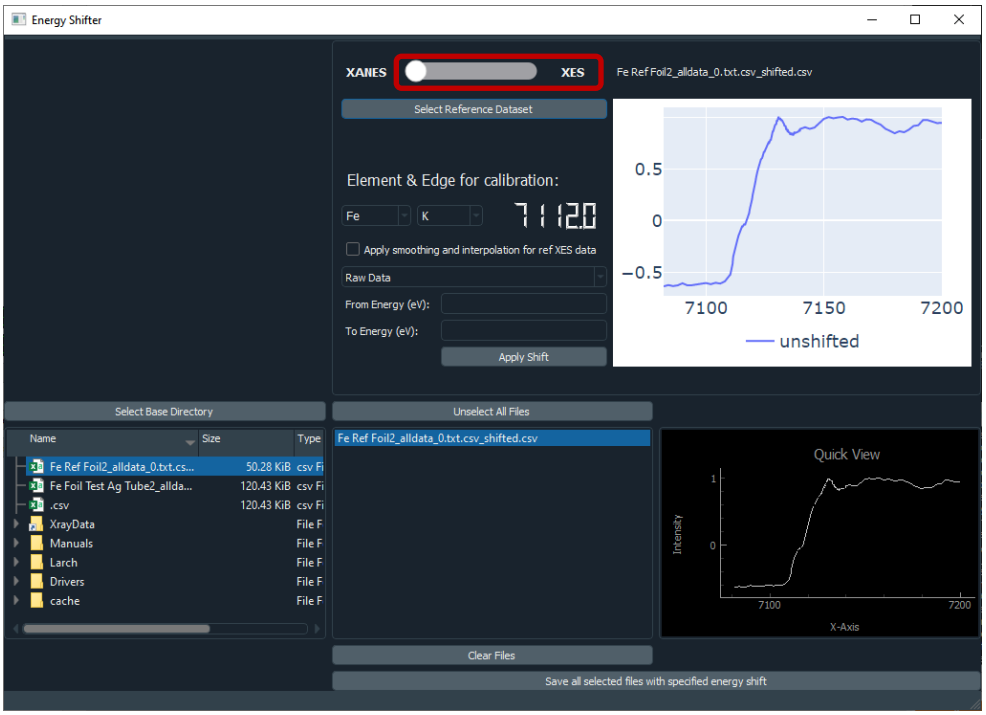
8.19 A window will open up. Start by selecting the base directory, by clicking on the Select Base Directory button.



8.20 Click on the zip file of a data set, to be added to the plotting window. Highlight the desired files and click on the Plot Selected Files button to view a plot of the emission data. Right clicking on the emission plot will allow for the plot to be saved as an image (needed for the weekly calibration report).

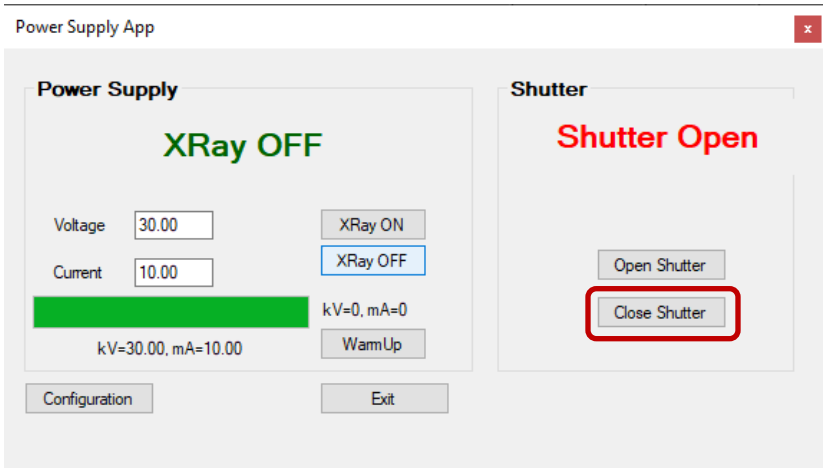


8.21 XES data can also be shifted similar to XAS data, by following Steps 8.11 – 8.17, but clicking on the XES option (highlighted in red).

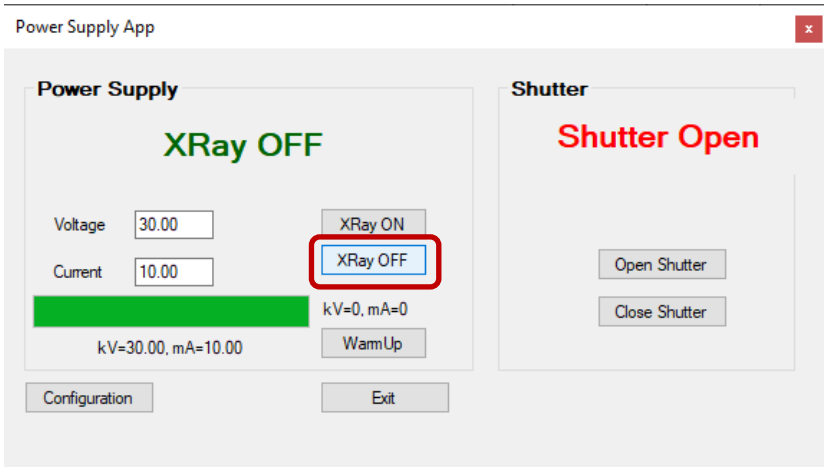


9 Shutting Down the easyXAFS 300+ X-ray Spectrometer

9.1 To shut down the easyXAFS spectrometer, click on the Close Shutter button on the Power Supply App, which will close the shutter on the X-ray tube. When the shutter is closed the Shutter Open light will no longer be illuminated.



9.2 Then click on XRay OFF button on the Power Supply App, which will turn off the X-ray tube. When the X-ray tube is off the X-ray On light will no longer be illuminated.



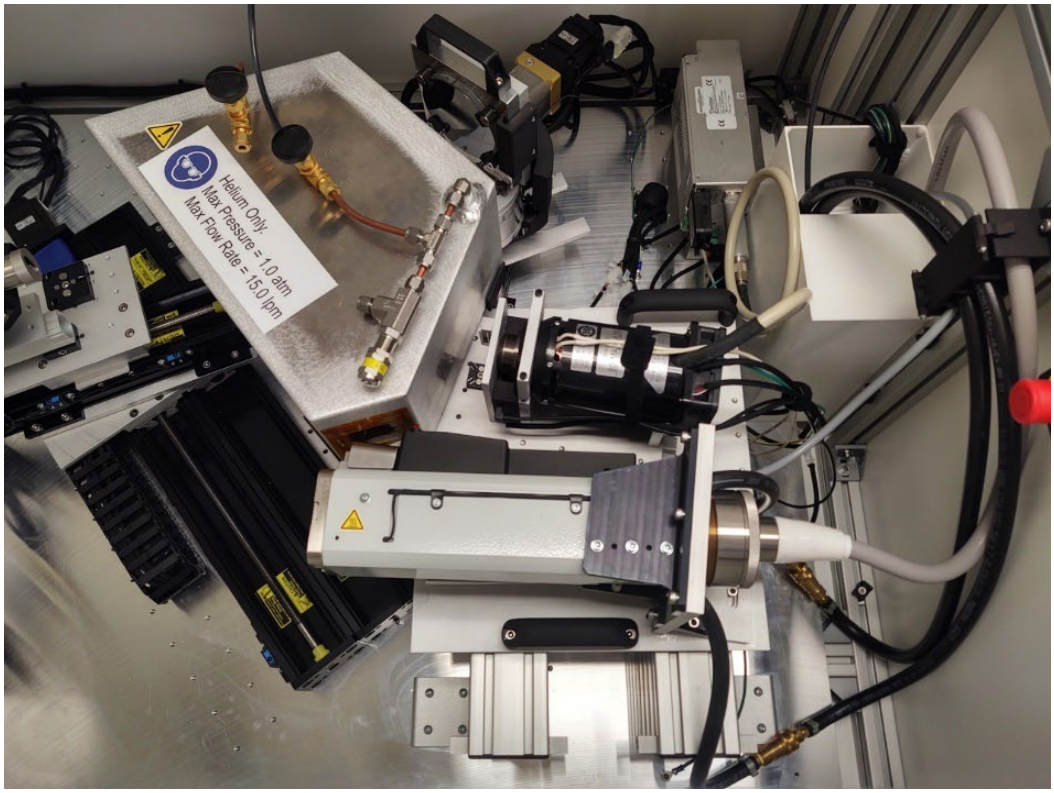
9.3 After the X-ray tube has been turned off, the electronics box can be turned off. The off state of the electronics box is shown below.

9.4 Then the system controller can be turned off by first pushing the X-ray enable button, followed by the power button. The off state of the system controller is shown below.

9.5 Let chiller run for about 15 minutes after the X-rays have been turned off to make sure the X-ray tube is cooled down before turning off the chiller.

9.6 While you are waiting to turn off the chiller, this is a good time to analyze your data (see Section 8) and to enter the ending time of your experiment in the instrument logbook.

9.7 Also, check to make sure the instrument is set up for XAFS measurements for the next user. This only needs to be done if samples were measured by XES. The instrument should look like the following picture when done.



9.8 Turn off the chiller by pushing the power button on the front of the chiller. The off state of the chiller is pictured.

9.9 Before leaving the room, make sure the door to the easyXAFS is closed and the instrument should look as seen in the picture below.

10 Training

All users are required to provide the NUCS Core Facility Staff with records of completion of the WSU Radiation Safety Office Training Courses #1-7 & 10 (https://rso.wsu.edu/wsu-radiation-safety-training/), *prior* to being trained on the use of the easyXAFS 300+ X-ray Spectrometer.

Instrument Trainers

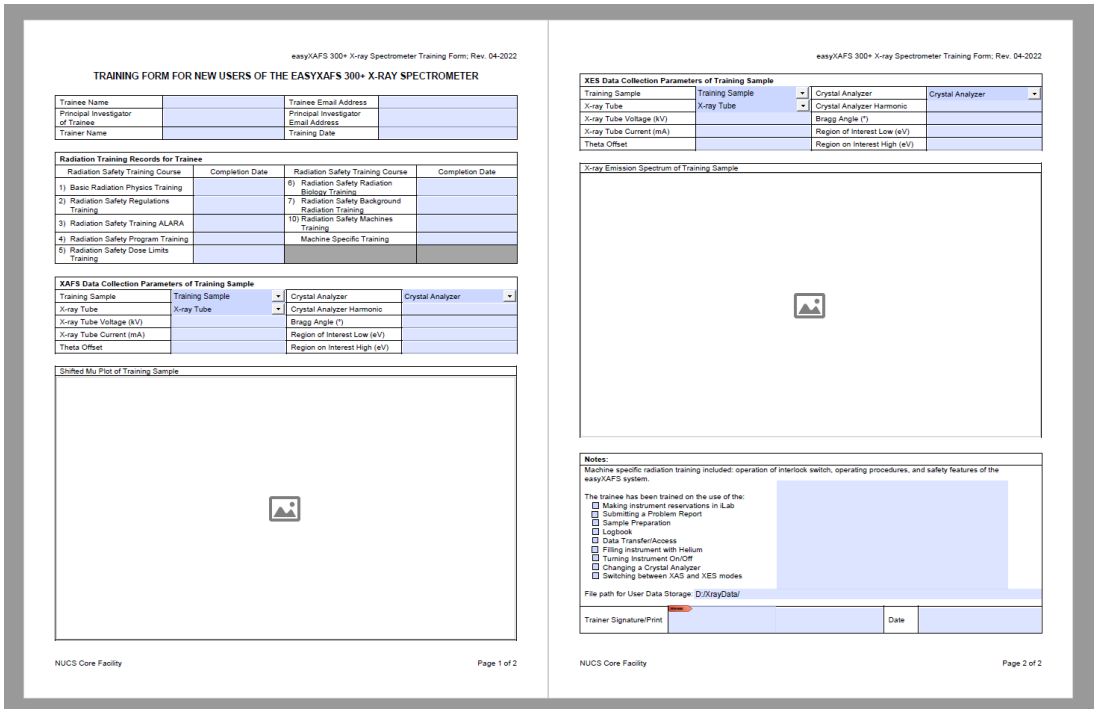
Zach Heiden, NUCS Core Facility, 509-335-0936

Bill Hiscox, NUCS Core Facility, 509-335-8259

Nuclear Science Center Emergency Line: 509-335-0004

Training on the easyXAFS X-ray Spectrometer consists of the safe use of the instrument, collection of a single XAFS and XES dataset on an iron foil, data analysis using the easyXAFS software, discussion of the interlock system and safety features of the easyXAFS 300+ X-ray Spectrometer, making instrument reservations in iLab, submitting an instrument problem report, powering the X-ray tube up and down, keeping records of use in the instrument logbook, and data transfer/access.

The form that is completed during a training session is seen below:



easyXAFS 300+ X-ray Spectrometer Training Form; Rev. 04-2022

TRAINING FORM FOR NEW USERS OF THE EASYXAFS 300+ X-RAY SPECTROMETER

Trainee Name		Trainee Email Address	
Principal Investigator of Trainee		Principal Investigator Email Address	
Trainer Name		Training Date	

Radiation Training Records for Trainee

Radiation Safety Training Course	Completion Date	Radiation Safety Training Course	Completion Date
1) Basic Radiation Physics Training		6) Radiation Safety Radiation Biology Training	
2) Radiation Safety Regulations Training		7) Radiation Safety Background Radiation Training	
3) Radiation Safety Training ALARA		10) Radiation Safety Machines Training	
4) Radiation Safety Program Training		Machine Specific Training	
5) Radiation Safety Dose Limits Training			

XAFS Data Collection Parameters of Training Sample

Training Sample	Training Sample	Crystal Analyzer	Crystal Analyzer
X-ray Tube	X-ray Tube	Crystal Analyzer Harmonic	
X-ray Tube Voltage (kV)		Bragg Angle (°)	
X-ray Tube Current (mA)		Region of Interest Low (eV)	
Theta Offset		Region on Interest High (eV)	

Shifted Mu Plot of Training Sample

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easyXAFS 300+ X-ray Spectrometer Training Form; Rev. 04-2022

XES Data Collection Parameters of Training Sample

Training Sample	Training Sample	Crystal Analyzer	Crystal Analyzer
X-ray Tube	X-ray Tube	Crystal Analyzer Harmonic	
X-ray Tube Voltage (kV)		Bragg Angle (°)	
X-ray Tube Current (mA)		Region of Interest Low (eV)	
Theta Offset		Region on Interest High (eV)	

X-ray Emission Spectrum of Training Sample

Notes:

Machine specific radiation training included: operation of interlock switch, operating procedures, and safety features of the easyXAFS system.

The trainee has been trained on the use of the:

- ☐ Making instrument reservations in iLab
- ☐ Submitting a Problem Report
- ☐ Sample Preparation
- ☐ Logbook
- ☐ Data Transfer/Access
- ☐ Filling instrument with Helium
- ☐ Turning Instrument On/Off
- ☐ Changing a Crystal Analyzer
- ☐ Switching between XAS and XES modes

File path for User Data Storage: D:/XrayData/

Trainer Signature/Print		Date	

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