BioXAS User Operation Manual for BioXAS-MAIN Beamline

Date: Tuesday, July 16, 2024
Date Amended: Thursday, January 16th 2025

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1.0 INTRODUCTION & TECHNIQUE DESCRIPTION

The purpose of this work is to outline best practices used at the Canadian Light Source (CLS) for X-ray Absorption Near Edge Spectroscopy (XANES) and Extended X-ray absorption Fine Structure (EXAFS) experiments at the BioXAS-Main beamline [1]. Several beamlines at CLS are also capable of this technique, with varying energy ranges that make the beamlines best suited to probing certain elements.

XANES experiments provide information on the oxidation state of the element of interest. With a good set of standards, Linear Combination Fitting (LCF) can be applied to determine the atomic proportions of various oxidation states of the element of interest within a sample. If there is a good understanding of the sample, LCF can also be used to estimate the atomic proportions of species within the sample, including proportions of standards of the same oxidation state.

EXAFS experiments also compliment XANES experiments can provide information on the local coordination environment. EXAFS analysis can also be used to extract static and dynamic disorder, bond lengths, and particle sizes.

BioXAS-Main is well suited for measuring various elements from 5-32 keV. Benefits of using BioXAS-Main for XANES/EXAFS measurements include cryogenic capabilities, highly sensitive detectors, and X-ray energies up to 32 keV.

2.0 EXPERIMENT DESIGN

2.1 BEAMLINE DESCRIPTION

The BioXAS-Main beamline uses a wiggler as the source of X-rays. The energy range of BioXAS-Main makes it well suited to measure the K-edges of elements in the 3d, 4d, select elements in the 4p and 5p blocks as well as L1,2,3- edges of elements in the 5d and 4f block. **Table 1** shows the specifications of the BioXAS-Main beamline [1].

Table 1: Beamline specifications for the BioXAS-Main beamline [1].

Beamline	BioXAS-Main
Energy Range, keV	5 – 32
Detectors	Ionization chambers; PIPS; and Canberra 2 x 32-element HPGe solid-state
Relative Strengths	-large energy range -cryogenic capabilities -highly sensitive Ge detectors -8-16 sample wheel
Relative Weaknesses	-Fewer samples can be loaded at a time (up to 5 in the cryostat)
Typical Samples	-Powdered, liquid or thin film samples

2.2 SAMPLE PREPARATION CONSIDERATIONS

Samples for XANES experiments are usually well ground with a mortar and pestle, or mechanically micronized if possible, to a particle size that is small compared to the wavelength of the incident beam, provided this grinding can be completed without inherently damaging the sample. Mineralogical and inorganic materials samples can generally (but not always) be grinded or

micronized to reduce crystallite size without significantly changing other properties of the sample. The sensitivity of the samples to grinding should always be discussed prior to shipping, whenever possible.

The samples are generally loaded onto cryo sample plates that can be attached to both the multi-stage and cryo vacuum stage. Samples are typically prepared for transmission or fluorescence measurements. Samples are prepared for transmission measurement by packing the powdered sample into cryo-sample holders and sealing with Kapton® tape, prepared with appropriate adsorption edge jump (ud~1). If the sample is too concentrated with the element of interest or another interfering element, sometimes the sample needs to either be diluted with boron nitride (BN) and packed into the sample holder, or it can be prepared for fluorescence measurement. Samples are prepared for fluorescence measurement by dusting a small amount of sample onto Kapton® tape and then sealing it with a second piece of Kapton® tape. When possible, all standards are prepared in transmission.

Transmission standards are prepared using calculations from the Hephaestus, CatMass or MAC3.0 software. An example calculation is shown in **Figure 1**. Using the "XAS Sample Mass (mg)" from MAC3.0, the amount of BN needed to dilute the sample is determined by the following equation:

$$BN_{mg} = 25\ mg - XAS\ Sample\ Mass\ mg$$

The amount of sample and BN can be increased by a common factor (usually 6x), in order to get a sufficiently large mixture to fill the holder and account for material lost upon grinding.

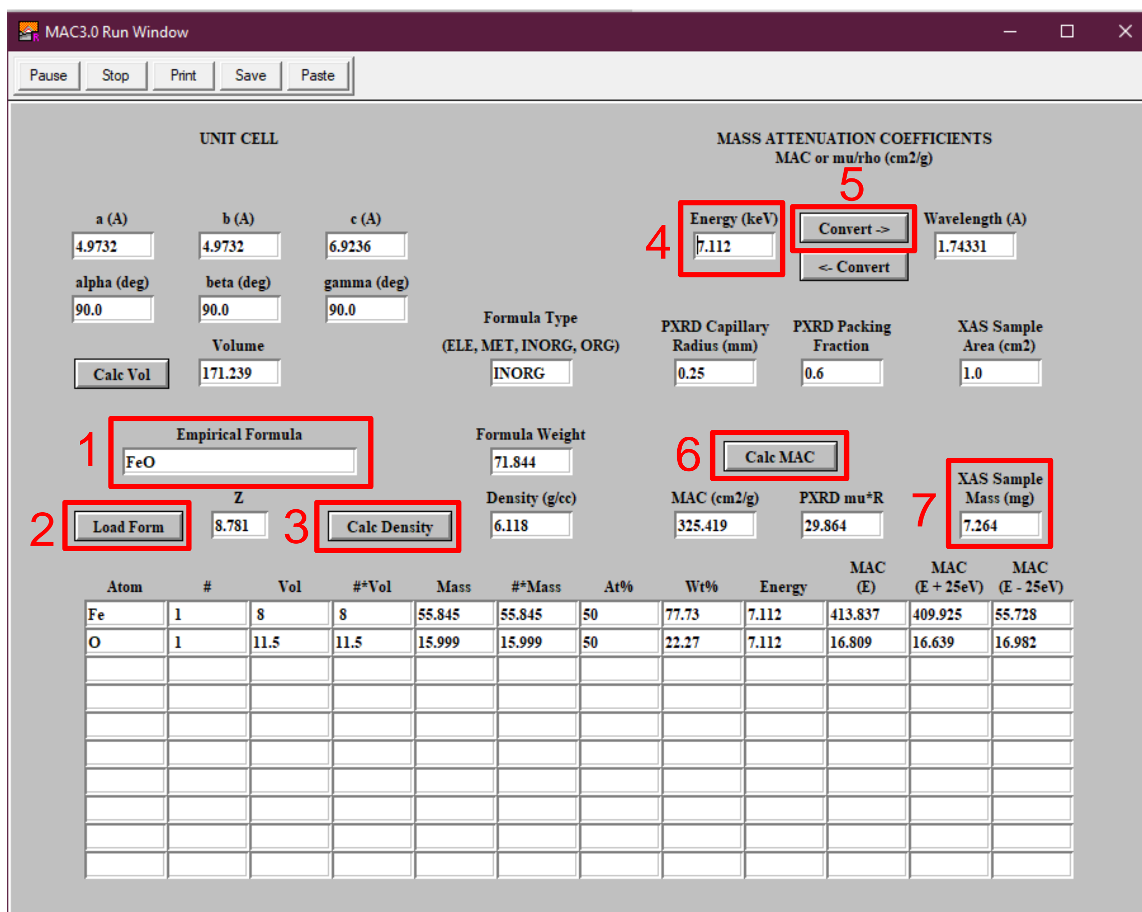


Figure 1: Example of a standard dilution calculation for iron oxide (FeO) using MAC3.0 using the following steps:

1. Enter the Empirical Formula, in this case, FeO.

2. Click “Load Form.”
3. Click “Calc Density.”
4. Enter the energy of the edge of interest. In this case, the Fe K-edge is measured at 7.112 keV.
5. Click “Convert ->.”
6. Click “Calc MAC.”
7. Read the “XAS Sample Mass (mg).”
8. Determine the amount of BN required.
9. Multiply required masses by a common factor before weighing.

User are also referred to read the FAQ at the end of the manual, or refer to this link to access how to prepare a Pellet.

2.3 OTHER PREPARATION BEFORE BEAMTIME

Aside from sample and standard preparations, there are other things that need to be done before beamtime.

- Create a notebook folder for you and your team
- Communicate with the Beamline Scientist in advance of the beamtime about the edges that will be measured, the number of shifts required, temperature requirements, and the project number.

3.0 GETTING FAMILIAR WITH ACQUAMAN

Acquaman **Figure 3** is the software used to run XAS at the BioXAS beamline. The software has different trees in order to (energy alignment, calibration, jj slit (defines beam size), check gains and offsets, move detectors in and out, move sample stage). The software is also used to prepare the beamline for sample data collection and setting up workflows.

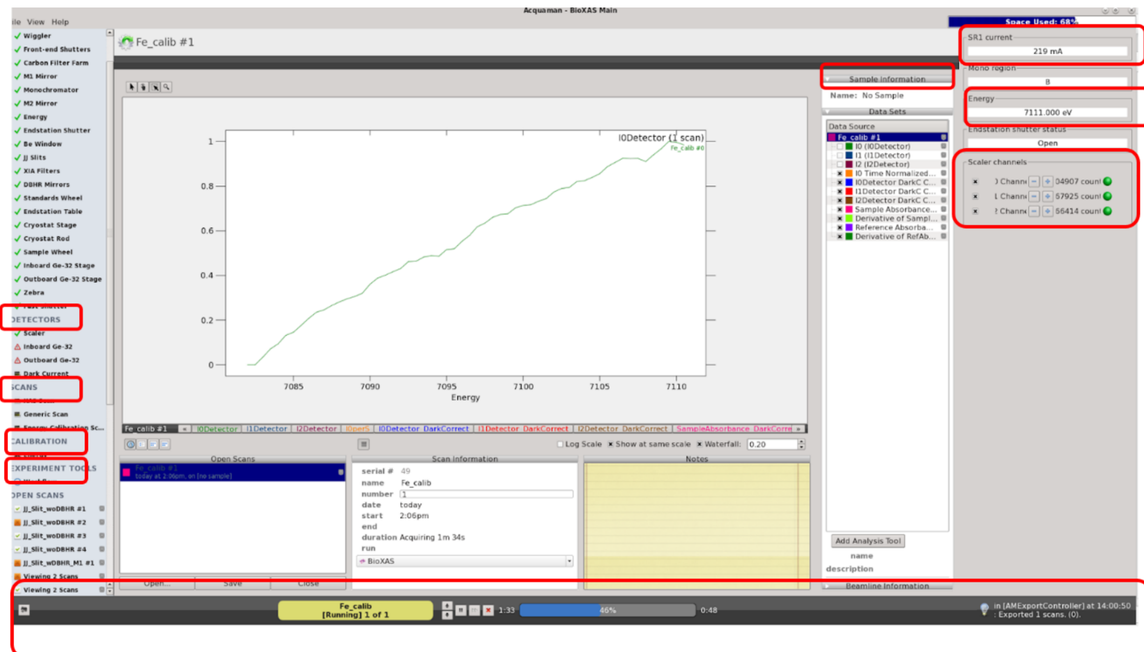


Figure 3: Diagram for the schematics of the setup for the data acquisition software Acquaman.

Acquaman has a multi tree setup.

- The first branch showcases the components of the beamline that Aquaman can control, and it is called COMPONENTS
- The second branch is the DETECTORS and it controls the inboard and outboard 32 element Ge detector.
- The third branch is the SCANS and allows one to scan motors, sample (or generic), collect an XAS.
- The fourth branch is CALIBRATION which allows you to perform energy alignment and calibration
- EXPERIMENT TOOLS
- OPEN SCANS: allows you to open and observe collected scans
- DATA (runs and experiments)- stores the data in a visual format

The righthand side shown in Figure 3 shows the sample information, the data being collected (I0, I1, I2, derivative and reference absorbance), ring current, energy of the monochromator, and counts on the scaler channels.

At the bottom, there is a status bar which helps indicate the status of the run being collected.

4.0 GETTING FAMILIAR WITH CSS PHEBOUS

Control System Studio (CSS) Phoebus **Figure 4** is the current control system studio toolset that allows for widgets and displays of BL parameters.

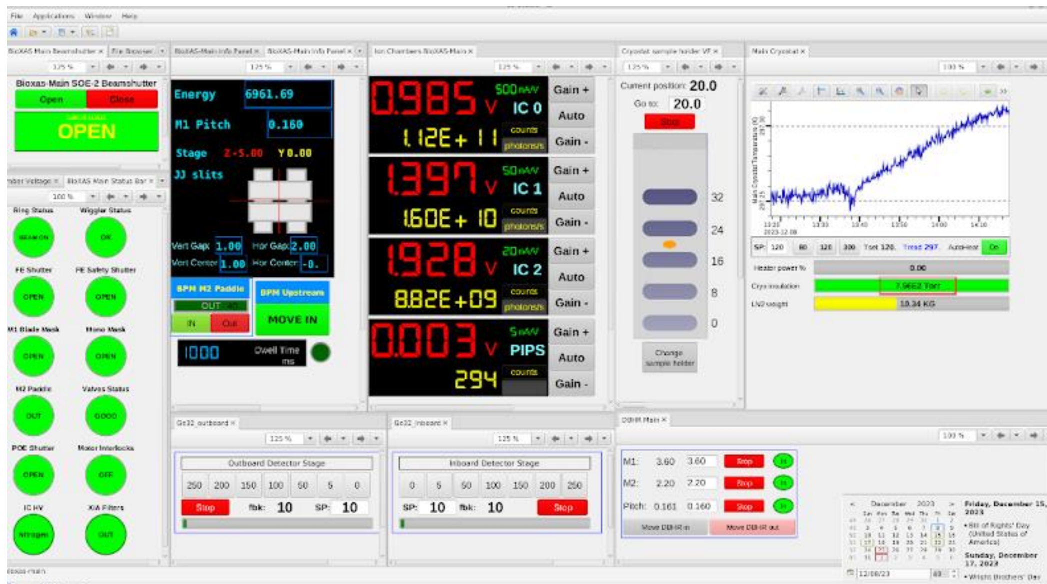


Figure 4: Diagram for the schematics of the setup of Beamline parameters using CSS Phoebus.

Within this interface (CS-Studio), there are 9 sub-panels.

- *SOE-2 Beam Shutter panel:* used to close and open photon shutter
- *BioXAS Main Status Bar:* Gives beam status, and overall should all be green if all is running well at the BL

- *BioXAS main Info panel:* Gives energy, M1 pitch position reading, stage (Z,Y) reading, JJ slit reading, Paddle (in or out of beam)
- *Ion Chambers BioXAS main:* Gives the photo counts, voltage reading and supplied rate to the different ionization chambers (I0, I1, I2) and the PIPS detector.
- *Cryostat Sample Holder:* gives position of the sample in the beam path
- *Main Cryostat:* Gives cryostat temperature reading, LN2 weight, pressure, and heating power status bar
- *Ge32-outboard:* Can move the detector closer or farther away from the sample
- *Ge32-Inboard:* Can move the detector closer or farther away from the sample
- *DBHR main:* changes the M1 and M2 and Pitch position and used at lower energies < 10 keV

5.0 BEAMLINE PREPERATION

In order to prepare the beamline for experiments, several key components needs to be ensured (voltages, gases in ionization chambers ICs, detector initialization, and energy calibration).

Voltages

Voltage readings for the ionization chambers should be as follows depending on the energy; this is important to enable a linear response.

<10 keV
nA /V with DBHR

>10 keV
nA/V-uA/V without DBHR

>> 15 keV
uA /V w DBHR

Ion Chamber Gases

1200 V Ar (50% flow)

2000 V N2 (50% flow)

Detector Configuration

Ion Chamber Initializing:

Go to Zebra on Acquaman configuration. You will find a button that says configuration on the top left corner (this will reset all values of gains and offsets on the ICs, you may also need to click on soft I1 or soft I2). Once that is done, go to scaler tab, and make sure that the current run configuration is set to continuous.

Initializing the In and Outboard Ge detectors:

To be performed if using Germanium detectors. In the DETECTOR branch in Acquaman, select the appropriate detector, ensure that the mouse is not in the vicinity of the white portion of the graph, select the desired element and edge of interest. Click Initialize and then acquire. Ensure that the channels are reading in the green region to make sure that the detector is not being saturated and the response remains in the linear response regime.

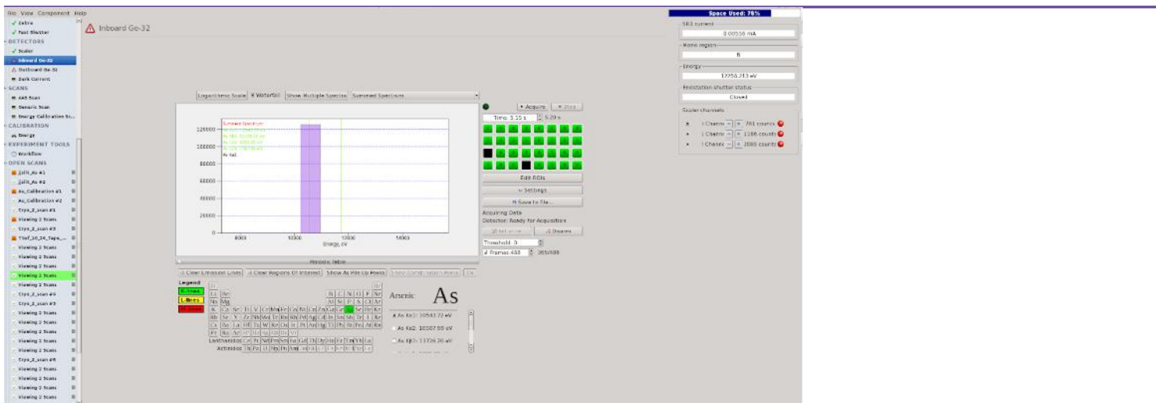


Figure 5: Diagram of the initialization process for the In and Outboard Ge detectors.

PIPS configuration:

If using the PIPS; go to the configuration tab in Acquaman and click on the PIPS detector.

Beamline Calibration

Communicate with the Beamline Scientist in advance about which edges will be measured. This communication will allow for proper planning of the experiment so that the correct monochromator and approximate calibration are ready ahead of time.

The beamline scientist will prepare the beamline for your experiments and will have already performed an energy calibration and alignment. If there is an energy change, additional beamline calibration could be possible, if energies are not far apart.

The calibration is set by placing the appropriate metal foil between the I1 and I2 ion chambers and collecting an edge scan in the XANES region. The foils at the BioXAS-MAIN beamline are kept in a rotating wheel. Ensure that the foil is either already in the wheel, or if not, add it to the wheel and note the position. Use the Acquaman GUI to select and move to the correct element and position on the wheel. Make sure that the wheel is seeing the exit window of the IC (I1). The standard wheel is shown in **Figure 6** and the part of the GUI for selecting the foil is shown in **Figure 7**. Run an XAS scan in the XANES region (under the SCANS→ XAS Scan tab). Relative to the edge, the scan should start at -20 eV and end at 40 eV with a 0.500 eV step size and a dwell time of 2.00 s. Once scan is collected, navigate to Calibration tab, and load the data that was being collected for the foil. Select the cursor to the maximum of the inflection point. Take note of the initial energy. Move the energy to the maximum of the first derivative that was denoted. Once at that energy, click “Calibrate” to change the set point to that energy. Run the foil scan again after it has been calibration to the correct energy grid. This spectrum will also be used for calibration in Athena or Larch. The areas of the Acquaman GUI used are shown in **Figure 8 and Figure 9**. If wanting to extract S02, an EXAFS foil scan must be run (check section on XAS data collection for more information).

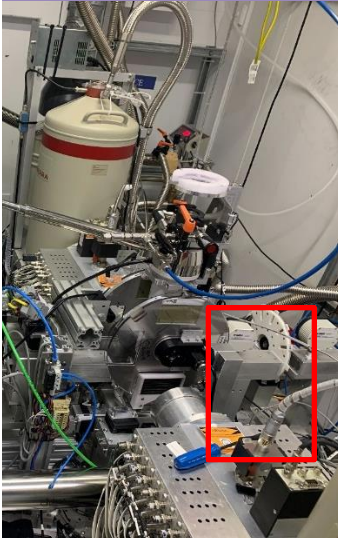


Figure 6: Photo of the standard wheel inside the hutch. The foils in the wheel can be removed and replaced if needed.

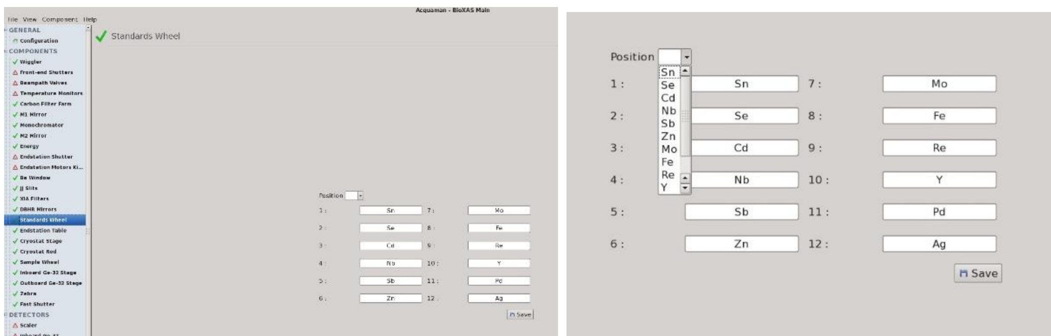


Figure 7: Acquaman GUI showing the foils and positions. The drop-down menu is used to select the foil required for the experiment.

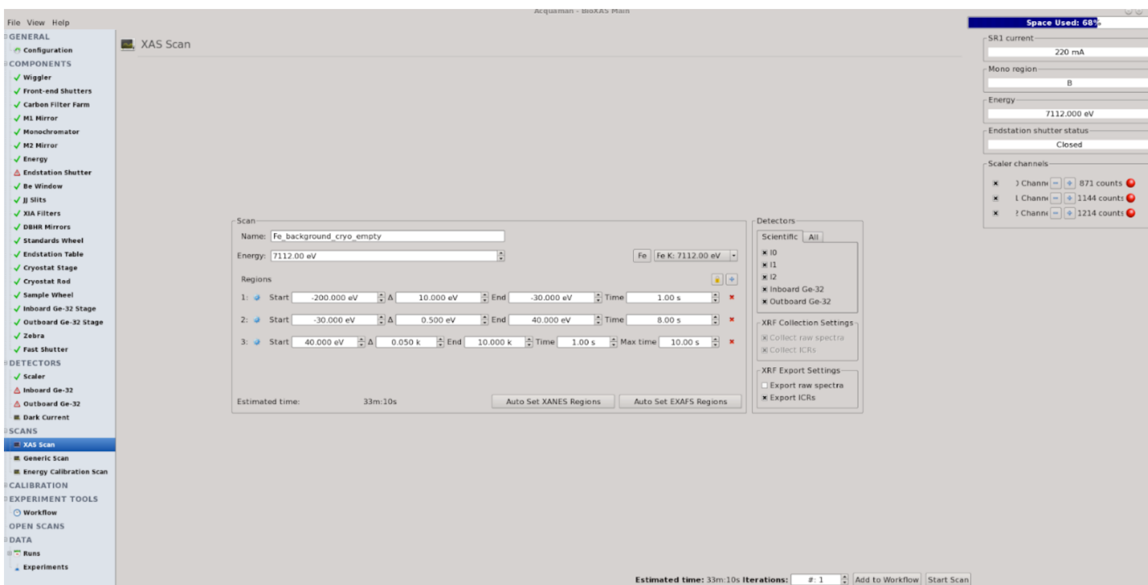


Figure 8: Image shows the maximum of the first derivative of the XANES scan of the foil.

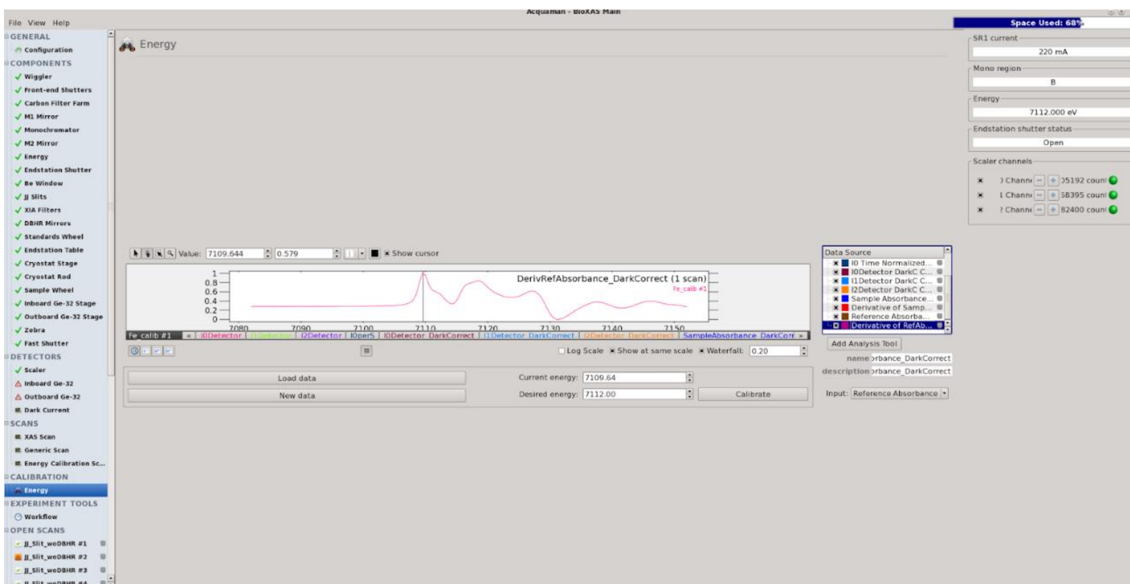


Figure 9: Image shows the energy calibration window used to load the foil data and calibrate the energy scale to the maximum of the first derivative.

6.0 XANES DATA COLLECTION USING BIOXAS-MAIN

This section will describe basic steps for XANES data collection at BioXAS-Main. To use BioXAS-Main, a user must use the computer at the beamline. Samples, standards, and foils need to be loaded while at the beamline.

The monochromator crystals on the beamline should generally be allowed to warm up for 20 minutes- 2h (in the event of beam-dump) before collecting data. If the monochromator is still changing temperature during data collection, changes in the reference line shape can occur. Inconsistent reference foil line shapes mean that comparisons across samples and proper LCF of samples cannot be performed.

Also, the lower the energy, the better the resolution. This means that we can resolve XANES better. Typically at higher energies the resolution of the DCM 220 at BioXAS is between a Si (220) and Si(111) with a resolution around 0.4eV, at lower energies the resolution is improved and the XANES can be collected with a resolution of 0.25eV.

6.1 LOADING SAMPLES

6.1.1 Cryogenic Sample Loading

The sample rod is removed from the cryogenic chamber and the metal cap is put over the chamber to minimize the warming of the chamber. Using Kapton® tape, secure the prepared samples to the sample holder that is compatible with the sample rod. If condensation interferes with the tape's adhesion, dry the rod and sample holders with a KimWipes® tissue before securing the samples to the rod. The sample holder, attached samples, and sample rod with samples are shown in **Figure 10**. Remove the cap to the cryo-chamber and replace and secure the sample rod with the clamp. Follow the lockup procedure and proceed toward the vacuum and gas control located next to the

back door of the hutch. Evacuate the chamber and refill it with helium. Repeat the evacuation and refilling procedure for a total of three rounds; these three rounds will ensure an effective flush of the lines which will remove enough air to improve scan quality. The evacuation pump and fill station is shown in **Figure 11**. Head back to the computers in the BioXAS-Main User area and ensure that the temperature drops to 80 K. The temperature monitoring window is shown in **Figure 12**.

Figure 10: From left to right: cryo-compatible sample holder, sample holder with attached samples, and samples locked into cryo-sample rod.

Figure 11: Evacuation pump and helium fill station behind the outboard hutch door at BioXAS-Main.

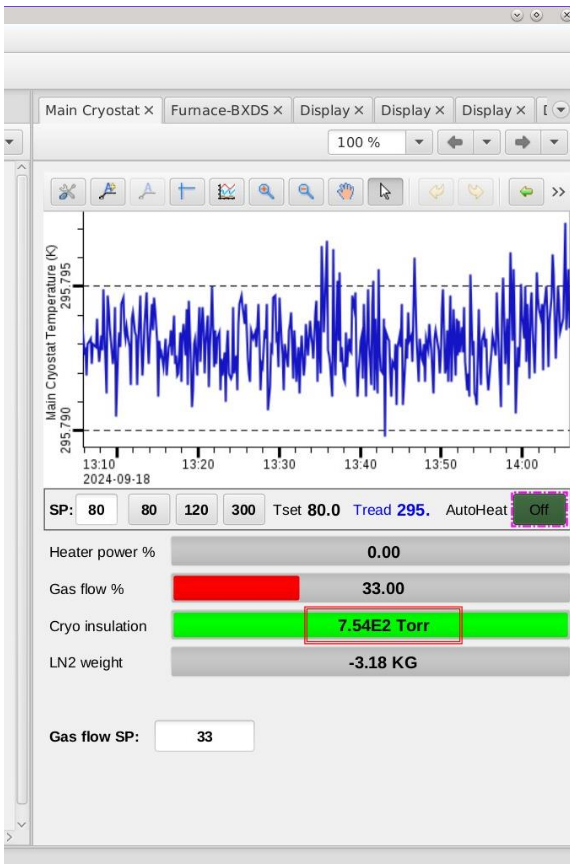


Figure 12: Temperature monitoring display within the CS-Studio GUI. The setpoint (SP) is 80 K.

6.1.2 Room Temperature Sample Loading

The sample wheel is removed from the experimental hutch and brought to the sample preparation bench. Prepared samples are secured on the sample wheel using double-sided Kapton® tape. The wheel is brought back into the hutch and secured onto the rotating wheel stage. Next, the lockup procedure is followed. The sample wheels are shown in **Figure 13**.

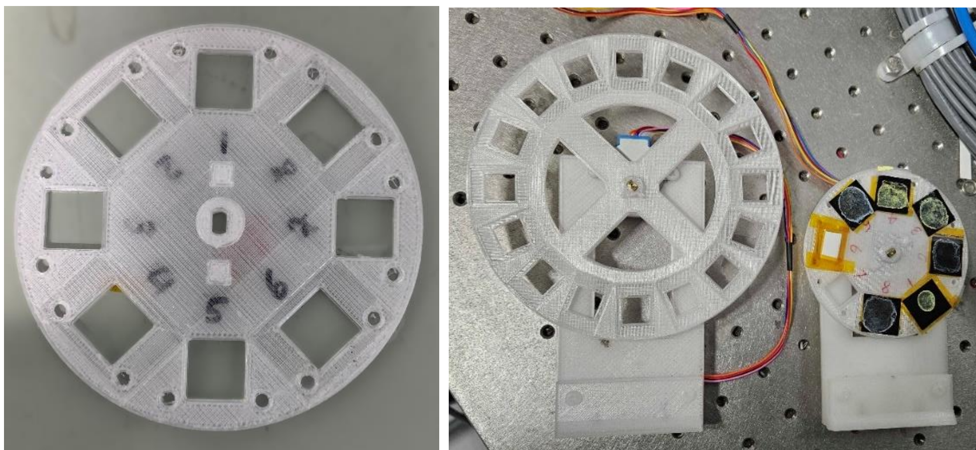


Figure 13: From left to right: 8-sample wheel detached from the wheel stage, 16-sample wheel attached to the wheel stage, and 8-sample wheel attached to stage with samples and phosphor card.

6.2 IDENTIFYING THE BEAM POSITION

At room temperature, click “Beam On” and use the GUI (i.e., Acquaman) to navigate to the phosphor card. Use the camera in the upper-right screen and a sticky note to mark the location of the beam. Under cryogenic conditions, a vertical scan is used to determine the relative location of the beam using the transmission signal.

6.3 PREPARING TO SCAN SAMPLES AND STANDARDS

On each sample and standard, check the signal below and above the edge to ensure the ion chambers are at acceptable values (between 0.2 V and 3 V). Ion chamber settings are located in the CS-Studio GUI, shown in **Figure 10**. Adjust the ion chamber gain settings to the appropriate setting. For transmission data, the sample may need to be re-made if the I1 ion chamber value drops below 0.2 eV. If X-ray fluorescence data is required, the Ge detectors dead times should be at acceptable values (showing green boxes in the detector tab after “acquiring”). The green boxes are shown in **Figure 11**. Adjust the sample-to-detector distance and/or slit size in the CS-Studio GUI to reduce the detector dead time. The CS-Studio GUI with detector distance and slit size adjustment is shown in **Figure 10**. If collecting fluorescence data, please record the deadtime above the edge.

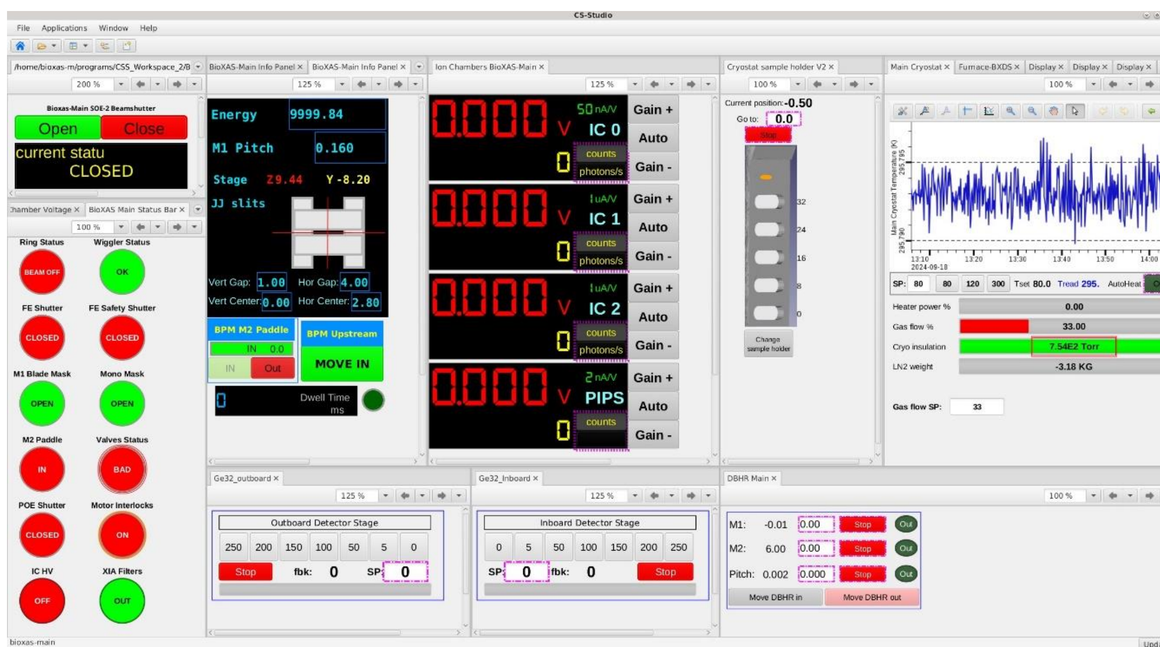


Figure 14: CS-Studio GUI showing ion chamber voltages and gains, detector distances, and slit sizes.

After checking that the dead time is okay and ion chamber gains are suitable for the sample, select the element of interest and its edge in the “Inboard Ge-32” and/or “Outboard Ge-32” tab(s). This menu is shown in **Figure 11**. Selecting the edge auto-loads the edge energy.

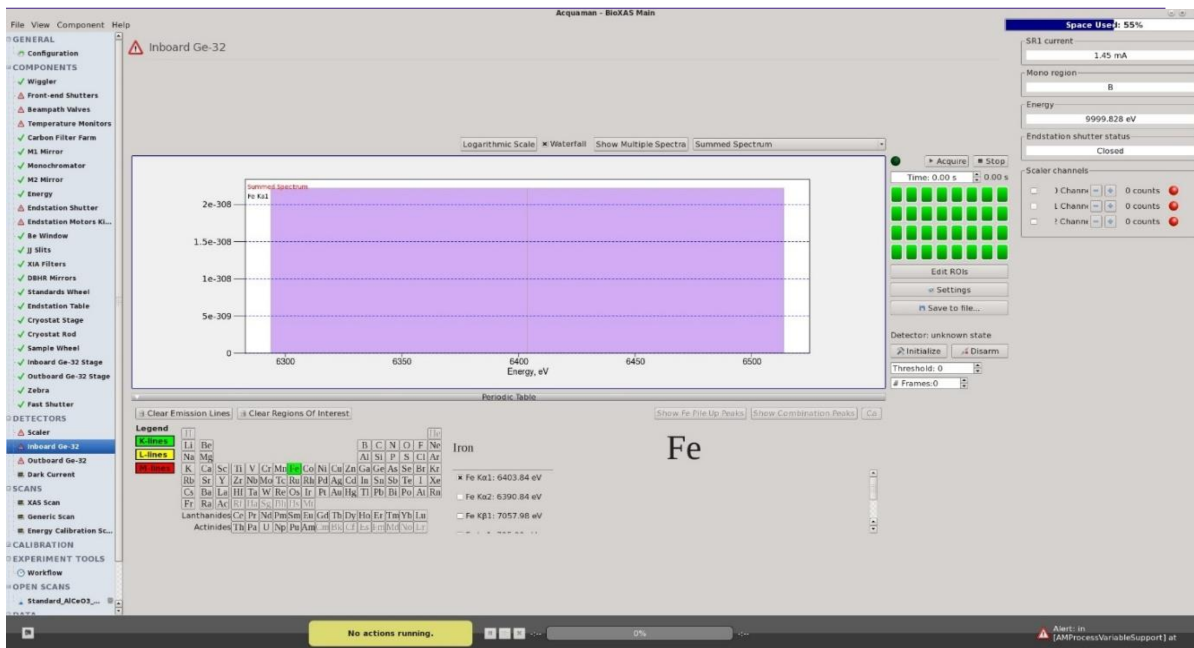


Figure 15: The green squares on the right side show the counts of each channel of the detector after acquiring; green values show acceptable dead times, whereas yellow or red show less than ideal dead times. The Inboard Ge-32 menu to select element and edge of interest is located at the bottom portion of the window.

In the “XAS Scan” tab, change the sample name. If collecting XRF data, click the boxes associated with the inboard and outboard detectors so that they are included. Select the element and edge of interest in this tab so that the energy will auto-load. Click “Auto Set EXAFS Regions” and adjust the parameters as needed. The typical scan configuration is as follows:

1. Start -200 eV ; Δ 10 eV ; End -30 eV ; Time 2 s
2. Start -30 eV ; Δ 0.5 eV ; End 40 eV ; Time 2 s
3. Start 40 eV ; Δ 0.05 k ; End 10 k (XANES scan) or 16 k (EXAFS scan) ; Time 2 s ; Max Time 10 s

For standards, click “Auto set EXAFS Region” and set end to 16 k (all standards should be collected to 16 k). Dwell time is 2 seconds and there should be 2 replicate scans. Follow the instructions above to confirm the standard has not been damaged by the X-ray beam. The scan configuration should be as follows:

1. Start -200 eV ; Δ 10 eV ; End -30 eV ; Time 2 s
2. Start -30 eV ; Δ 0.5 eV ; End 40 eV ; Time 2 s
3. Start 40 eV ; Δ 0.05 k ; End 16 k ; Time 2 s ; Max Time 10 s

The “XAS Scan” tab is shown in **Figure 16**. A minimum of two scans should be collected per sample. Two scans are required for determining if the sample is sensitive to beam damage and for getting a better signal-to-noise ratio. For low-signal samples, at least three scans should be collected.

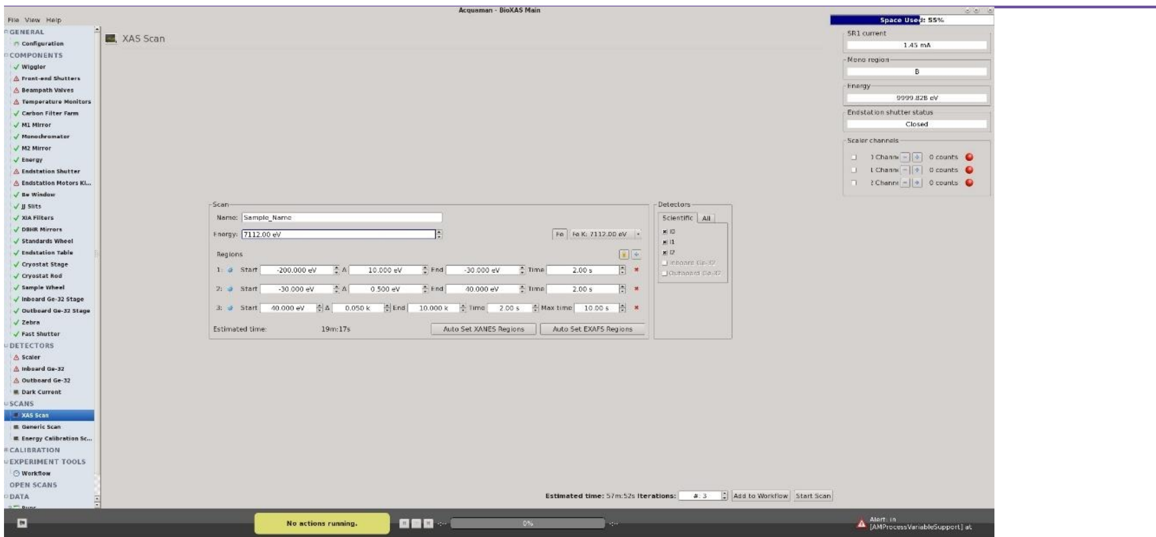


Figure 16: “XAS Scan” tab in the Acquaman GUI.

6.4 COLLECTING THE SCANS

Once the scan configuration is finalized for the sample or standard, click “Start Scan” or “Add to Workflow.” When running a workflow, ensure that the “control move” is set to move to the correct location before scanning each sample scan when adding to workflow. To start the multi-sample workflow, navigate to the “Workflow” tab and then click start. The “Workflow” window is shown in **Figure 17**.

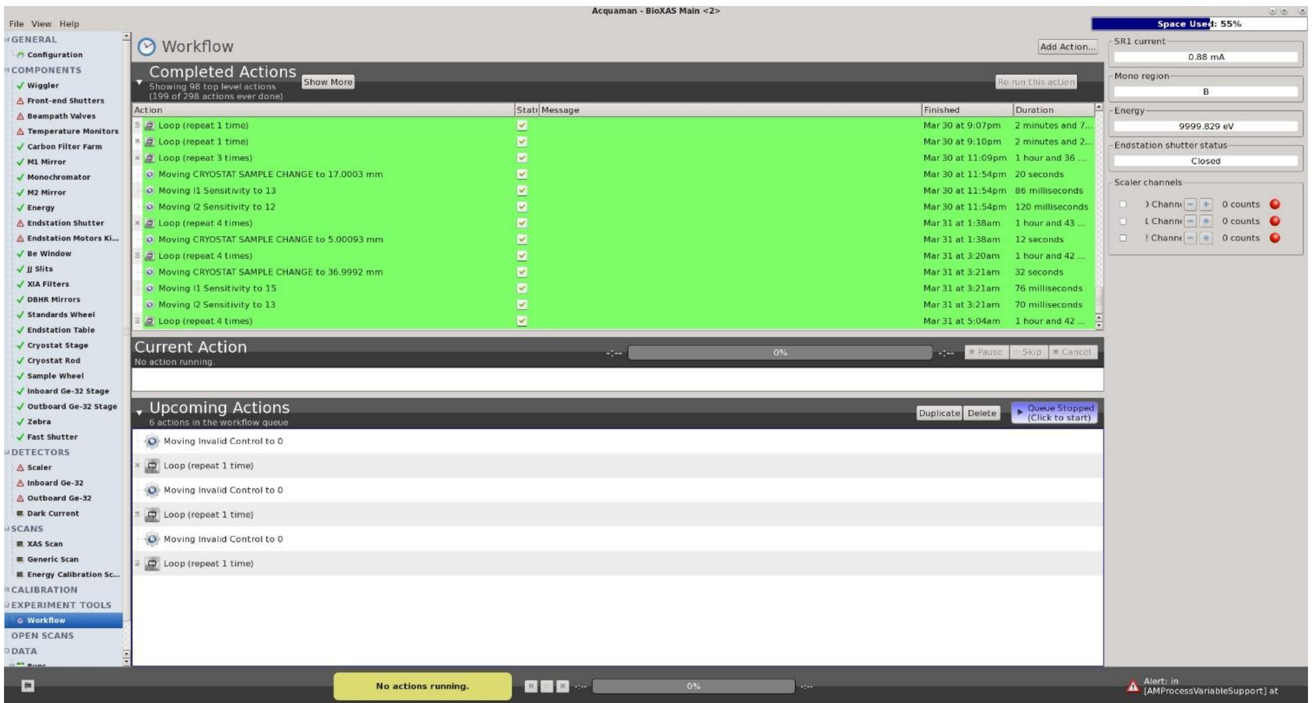


Figure 17: The “Workflow” window in the Acquaman GUI.

When the scan starts you will be brought to a new screen with a large window in the center where the data from the scan will appear. The tabs at the bottom of the plot correspond to active data channels. The “SampleAbsorbance_DarkCorrect” corresponds to the transmission XAS spectrum. A normalized transmission spectrum can be seen using the “Add Analysis Tool” button and using the Sample Absorbance and I0Detector selections. The “InB/I0” and “OutB/I0” tabs corresponds to the I0 normalized X-ray fluorescence XAS spectra. As the scans are collected, overlay the scans of the sample to ensure that there is no X-ray beam damage (i.e., scans are identical). If there is evidence of beam damage, slightly move the sample between scans to minimize the exposure of the sample to the X-ray beam. The data collection window is shown in **Figure 18**.

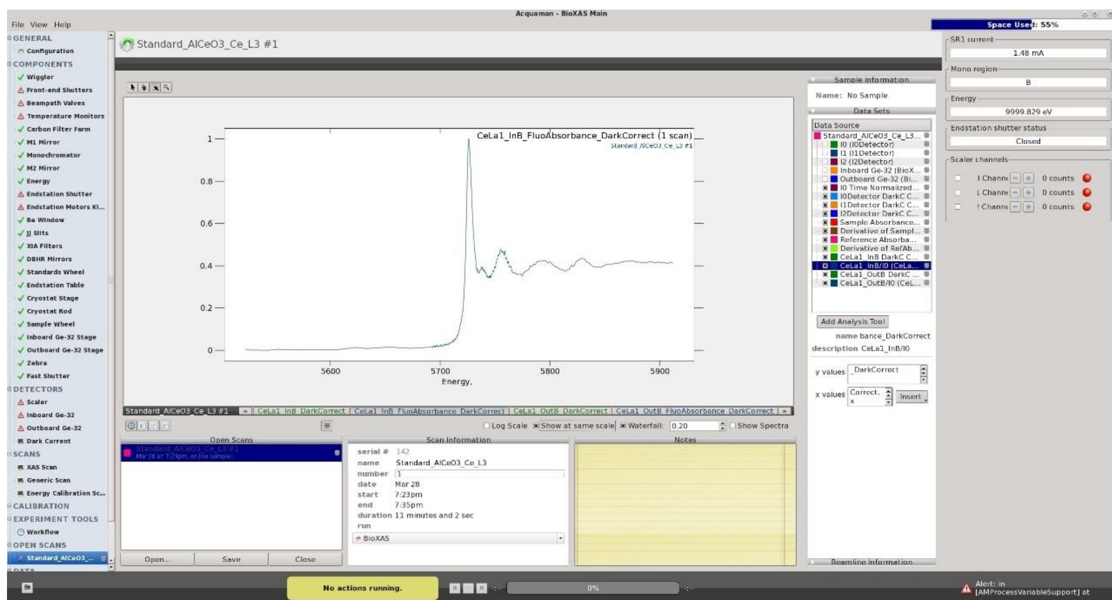
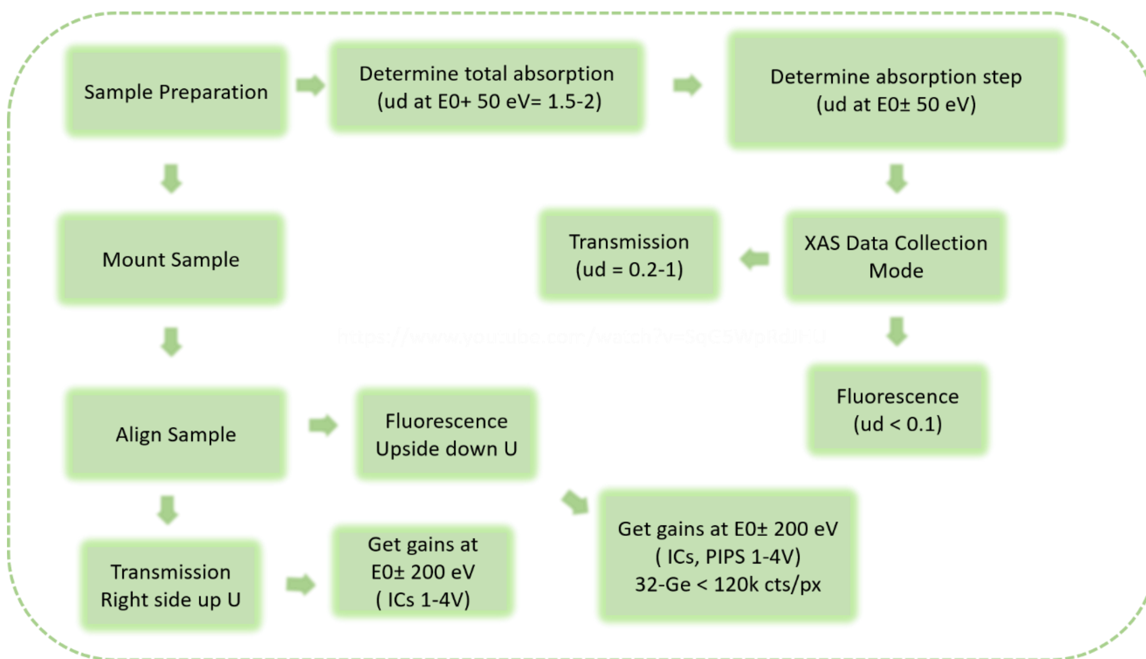


Figure 19: Data collection window in Acquaman for BioXAS-Main.

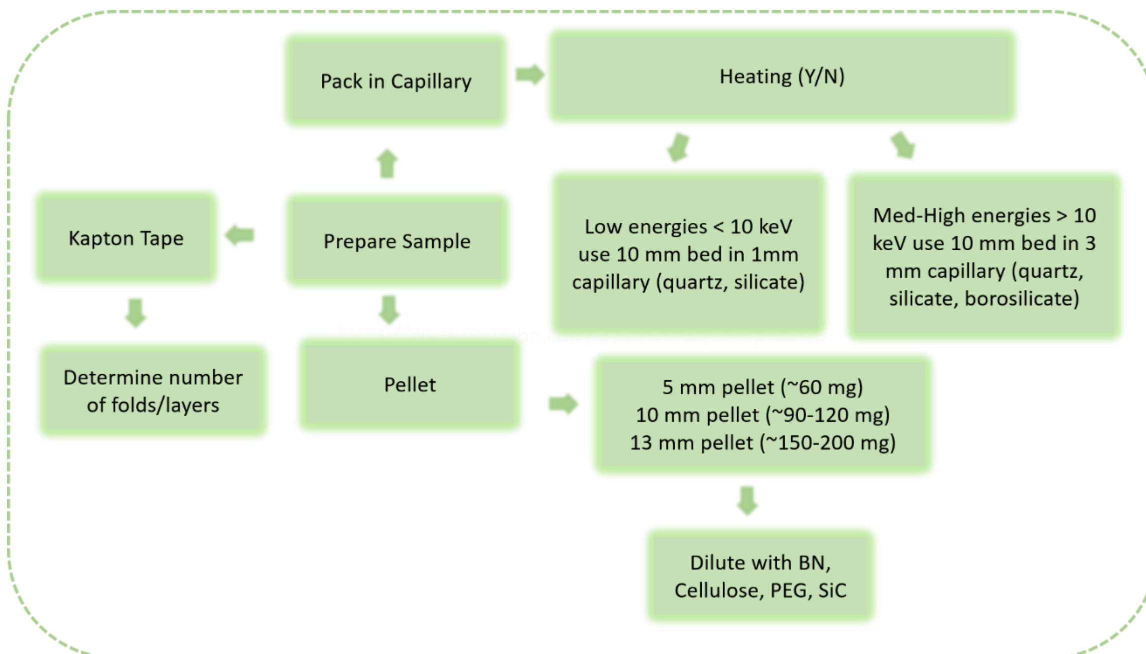
Record the start time of each scan, any notes such as IC gases, voltage, gains and offsets, beam size, sample motor positions, trajectory file used, number of scans, etc. When running multiple samples in a workflow, periodically check the scans to ensure high-quality data is being collected. Export the files as they are collected and create an Athena file with them.

7.0 FAQ

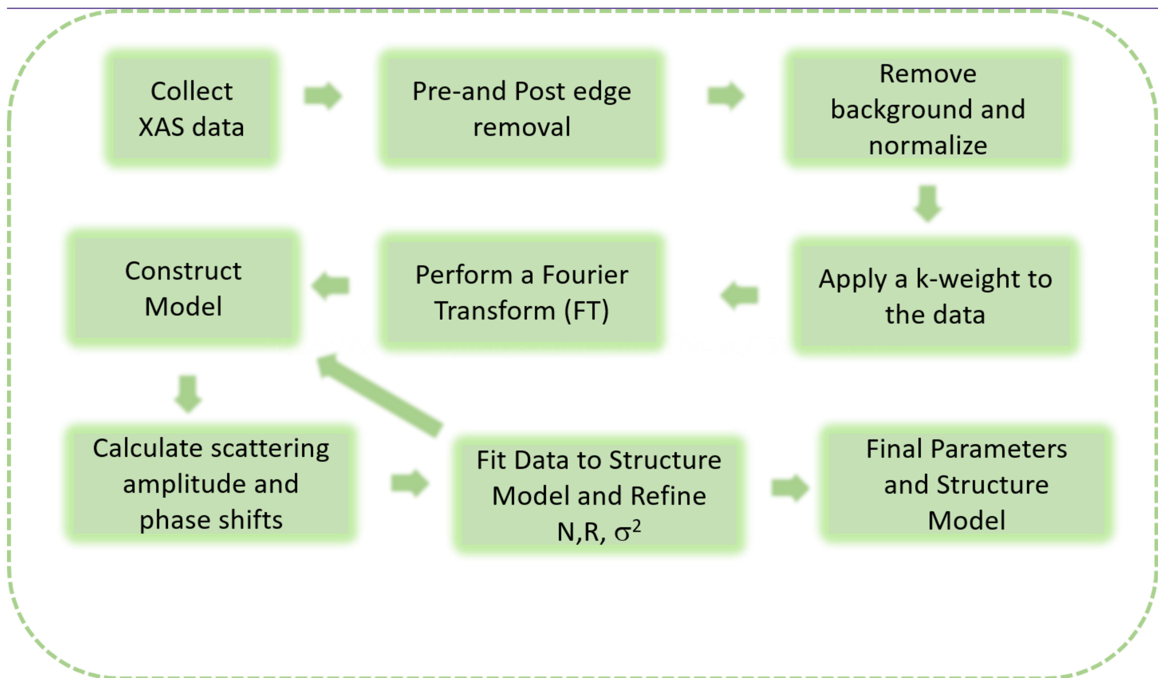
What is involved in XAS Experiment?



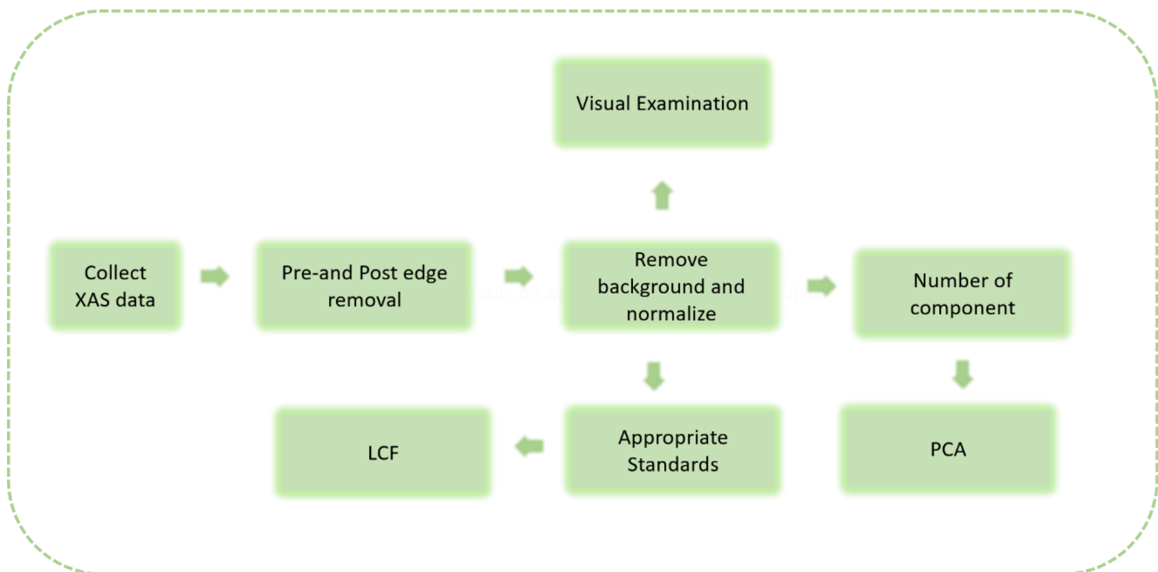
Can I get more insights on Sample Preparation?



What is a typical XAS workflow for EXAFS analysis?



What is a typical XAS workflow for XANES analysis?



How do I setup a XAS Scan?

- Acquaman does a good job in predetermining the regions for XANES and EXAFS. Generally as energy is scanned across, the absorption cross section decreases, and

EXAFS signal must be improved by longer count rates or by averaging across several scans. For XANES, the limitations in the step size (vs. oversampling) is the resolution of the crystal set used. Generally, a guide below can help provide information for setting up a step size in the XANES region. For EXAFS a typical spacing of 0.05 inverse angstrom (~ $0.2E^{1/2}$) is reasonable.

Edge Energy (eV)	Suggested Step Size (eV)
5000-6000	0.2
6000-7000	0.25
7000-8000	0.3
8000-9000	0.35
9000-10,000	0.45
10,000 and above	0.5

Start (eV)	ΔE	End	Time (s)
-200.0 eV	10.0 eV	-30.0 eV	1.0
-30.0 eV	0.5 eV	40.0 eV	1.0
40.0 eV	0.05 k	12 k	10.0

How far out in X(k) can I measure?

- The EXAFS oscillations die as 1/E. S/N is proportional to sqrt of the # of scans. Careful interplay of statistic and time should be considered.
- Generally, one can collect 1000eV above the edge, the uncertainty in will plateau, and are only able to radially resolve at most a difference of 0.09A

I have a sample that I am measuring in transmission mode, and I don't see the reference signal? What does this mean?

- This would mean that your sample is too thick, and is absorbing and reducing the amount of transmitted beam to the IC for the foil. The advice would be to reduce sample thickness

I have a sample that is supported sample, I know that it has adequate composition of the element of interest in transmission mode, but I don't see a good signal in the Foil IC or in transmission.

- This would mean that your support even at this energy is absorbing some of the beam, and reducing the beam going into the foil IC. Reduced sample thickness or number of tapes.

There a systematic glitch in my transmission data - what is it?

- It can either be due to glitches in I0 (check the signal in I0) or it can be what is referred to as a pinhole effect, where the beam sees no sample on the beam scale or it can be that the crystal size are too large that there is signal sample diffraction.

When I started my measurements, the shutters closed?

- This is normal, Acquaman measures dark currents before the start of the scan. The dark current scan takes as long as the maximum dwell time used in the region file for the XAS scan. For example, if you measure EXAFS up to 14 k, and the scan has a 10 s dwell time, then the dark current measurements will take 10 s to collect. If measuring XANES with a 1 s/point dwell time, then dark currents will measure for 1 s. In some occasions, the Phoebus panel update, the indicator for the photon shutter maybe be closed for longer, but the ACIS panel will automatically update in real time. As a precaution, look at the ACIS panel to ensure that after dark current measurements, the photon shutter is open

Why is Acquaman lagging?

- It is advisable to restart Acquaman program every day. This will hep reduced the software lag.

I am having trouble with energy calibration using a standard foil. When I hit the calibrate button, nothing seems to happen.

- Yes, this is normal, only click the calibrate button once and that will allow the software to more to the desired position. If the calibrate button is clicked more than once, it will shift the energy the same magnitude as the shift between the absolute energy and calibrant foil energy. Please only click once.

What is the correct way to calibrate?

  - Generally, use the first derivative peak.

My scan is running but the acquisition window stopped showing the spectra being collected. What should I do?

  - It is likely that there is a change in the scale that you are viewing. Re-adjust the scale and you should see your scan

I have low counts on my reference spectra when doing the calibration, why is that?

- Make sure that the reference foil on the reference foil wheel is properly aligned and in the beam path.

I am on my sample and ready to measure, but I don't see any counts on the Ge detector.

  - Make sure that the photon shutter is open, the Ge-detectors are initialized and that the caps are removed.

I have calibrated and I am ready to find my sample in the beam. I tried to find the sample but I could not, what do I do?

  - Use the fluorescent screen to verify that the beam is on your sample. If you see the beam on sample, then the sample concentration for the element of interest is low, or

- the sample is too thick (absorbing)-you can check if you are getting signal from your reference ionization chamber, or the sample is too thin and the amplifier is saturated (i.e. the voltage reading is above 5V). If you have a standard sample with known concentration it is advisable to measure the standard samples to see if you are getting expected signal and signal quality.

Acquaman just crashed and the software closed abruptly- How do I troubleshoot?

- Navigate to desktop screen and restart Acquaman

I restarted Acquaman but now I don't have counts in my ICs

- Navigate to Scaler, change the counts from single shot to continuous

There is a huge spike (downward spike) when measuring that is more intense than the counts/signal from the XAS data I collected, and I see it only in the Dark Corrected Spectra? What is this?

- It can be a few things (ice diffraction, contamination of a different edge, or incorrect dark current counts).
  - If ice diffraction, and measuring using Ge detector, not all pixels will capture the diffraction, and the signal will be more localized (i.e. some pixels will see it, other pixels will not see it). Check each channel,
  - If contamination of different edge, corroborate this with the edge finder on Hephaestus (typically for Cu, there is almost a Zn signal),
  - and finally, if in correct dark current, the spike will only be present in the dark corrected spectra, and you are probably getting negative counts because CSS was measuring dark currents with the beam accidently turned on- shutters were open (Acquaman automatically shuts the beam during this process), please make sure beam is off for dark current measurements). Change the gains, and the offsets will be adjusted accordingly.

I stopped the scan because I need to measure with Ge detector instead of PIPS. How can I restart collecting Fluorescence with the Ge detector?

- In the Data Collection Tab in Acquaman, make sure to tick the channels for In and Outboard Ge detector. If switching back to PIPS, navigate to configuration tab in Acquaman and click on PIPS to configure for PIPS.

Why are shutters not closing in the beginning of the scan?

- It maybe an Acquaman bug, please make sure to restart Acquaman and make sure only one instance is open The shutters close at the beginning of the scan to measure dark current. You will see that the POE shutter will be RED and turn GREEN after dark measurements are collected.

I am using the Ge detector to measure my sample, but I do not see any counts when running test scan?

- If you are on the sample, with correct position and beam slit size, make sure that the correct fluorescence line for the elements of interest is selected, otherwise it will read out no counts.

In the event of a beam dump

- Check Machine Status or login in to the user portal to follow updates on machine status.
- In the event of a trip, ring dump, etc., the machine status will change. With no beam, you can choose to pause or cancel your scans and wait for the machine status to show that the ring current is ~220 mA and that the ring is in top up mode.
- How long do I need to wait for the optics to warm up after beam-dump?
  - If it was an abrupt dump which was recovered quickly. You may wait the duration of the dump for warm-up. Generally, rule of thumb, wait the duration of the beam-dump for warm- up but no more than 2 hours.

Cryocooler and Cryostat

After a sample swap, the cryostat temperature started to decrease slowly, we tried to flush and opened the needle valve a little bit, but there was no detectable temperature change. How can we get the cryostat to cool down to 80K faster?

- First it is important to check that the needle valve on the cooling pump is open. The needle valve is fully open after 6 full rotations. In order for adequate cooling, 2-3 turns is sufficient. Please ensure you have enough LHe or LN2 when doing so. Monitor the cryo-insulation pressure, the reading should be around 6-5 E-5 torr. Please be careful when opening the needle valve and if uncertain if the valve is closed or open. Fully close the valve and then take note of the number of rotations.

My sample fell in the cryostat, what should I do?

- It may be a good idea to warm the cryostat to 300 K, disconnect the lines and flip the cryostat under a fume hood.

Detectors

- I am using the Ge detector, what is an acceptable count value?
  - Each of the green windows on the Ge detector software represent a pixel. The Ge detector we have has 32 pixels. Each pixel count rate should not exceed 150 (i.e. 150k counts).
- What does a filter do?
  - A filter will absorb the scattered peak, while transmitting most of the fluorescent line of interest. A filter can improve the S/N. For more information visit X-ray filter assembly for fluorescence measurements of x- ray absorption fine structure.

Where Can I find Useful XAS Data Bases?

- RefXAS
- CLS XAS
- Farrel Lytle Database
- IXAS XAFS database
- XASDataLibrary
- xaslib

8.0 REFERENCES

1. https://bioxas-spectroscopy.lightsource.ca/about-us/bioxas-main/
2. Standard Chemical and Methods document (CLSI doc. no. 27.1.1.2)