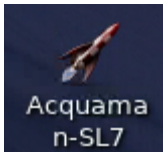
Acquaman User's Guide – Solid State Endstation

Table of Contents

September 18, 2023

Launching Acquaman

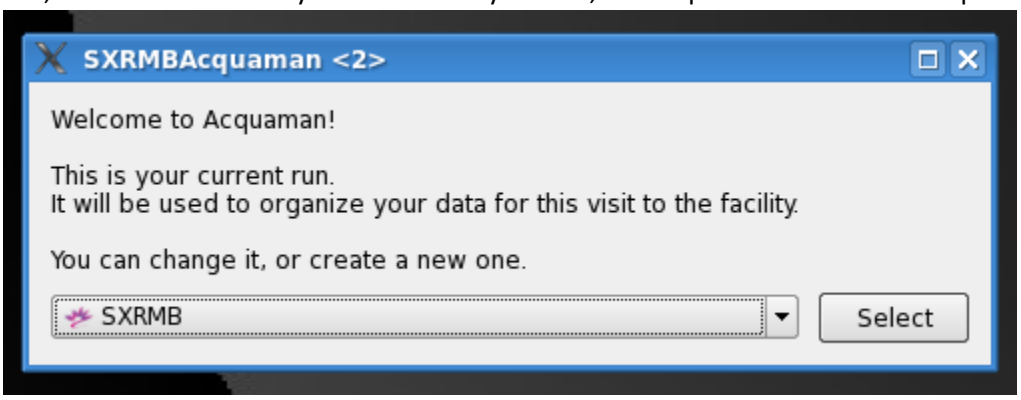
1. Open Acquaman by double clicking the icon on the desktop:



2. Put in the date in the proposal number box: YY-MMDD. This will be your folder for your whole experiment.

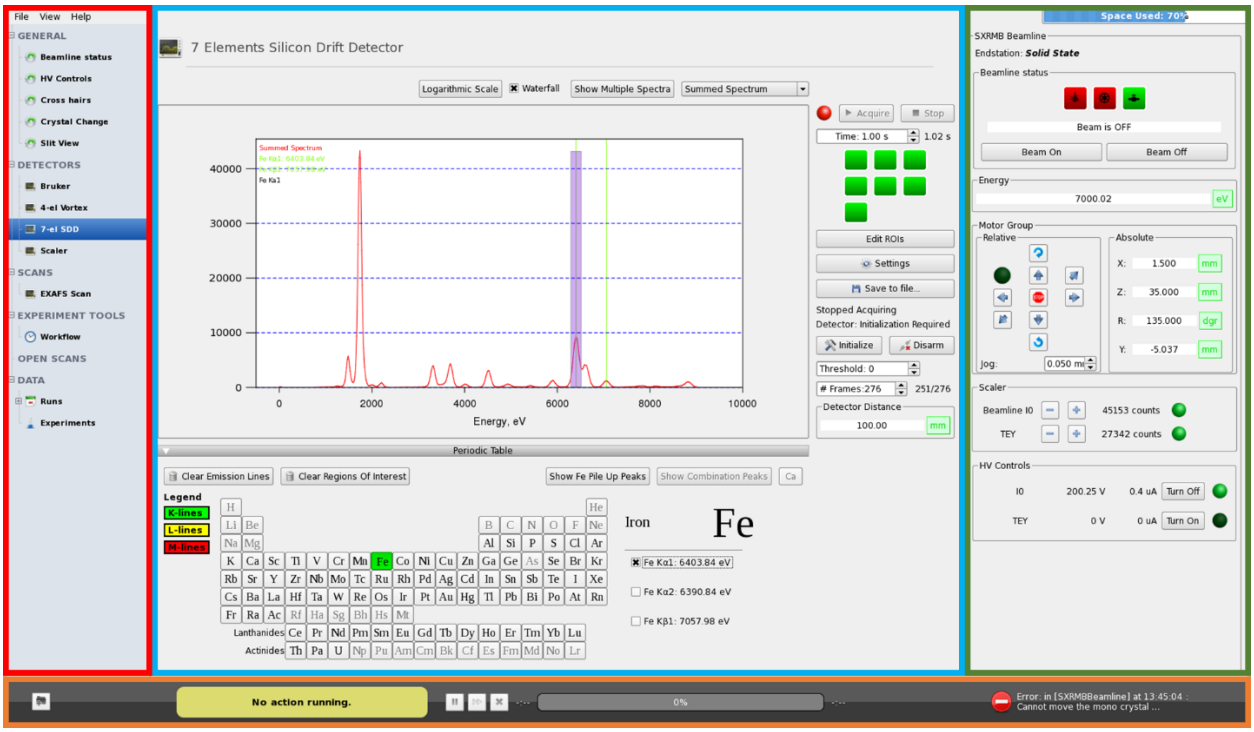
3. A window will open while the program is loading. If it is the first time you've used the "proposal number" it may take a few minutes to launch Acquaman. Please be patient.

4. Another window will open. Either select SXRMB or select the drop-down menu to make a new run, then select it. Once you've selected your run, the Acquaman interface will open.



Navigating Acquaman

Welcome to the main Acquaman interface. This is where all of your setup and acquisition parameters are selected:



Left Section: List of options for setup and acquisition parameters. You can also find your workflow information and data here.

Middle Section: The current selection from the left side is displayed here.

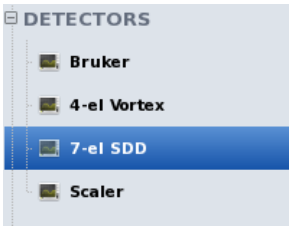
Right Section: These are the beamline settings. It shows the beamline status, manipulator motor positions, scaler output, and high voltage settings.

*****If you would like to collect TEY spectra, do not forget to turn on the TEY HV after you've turned on the beam*****

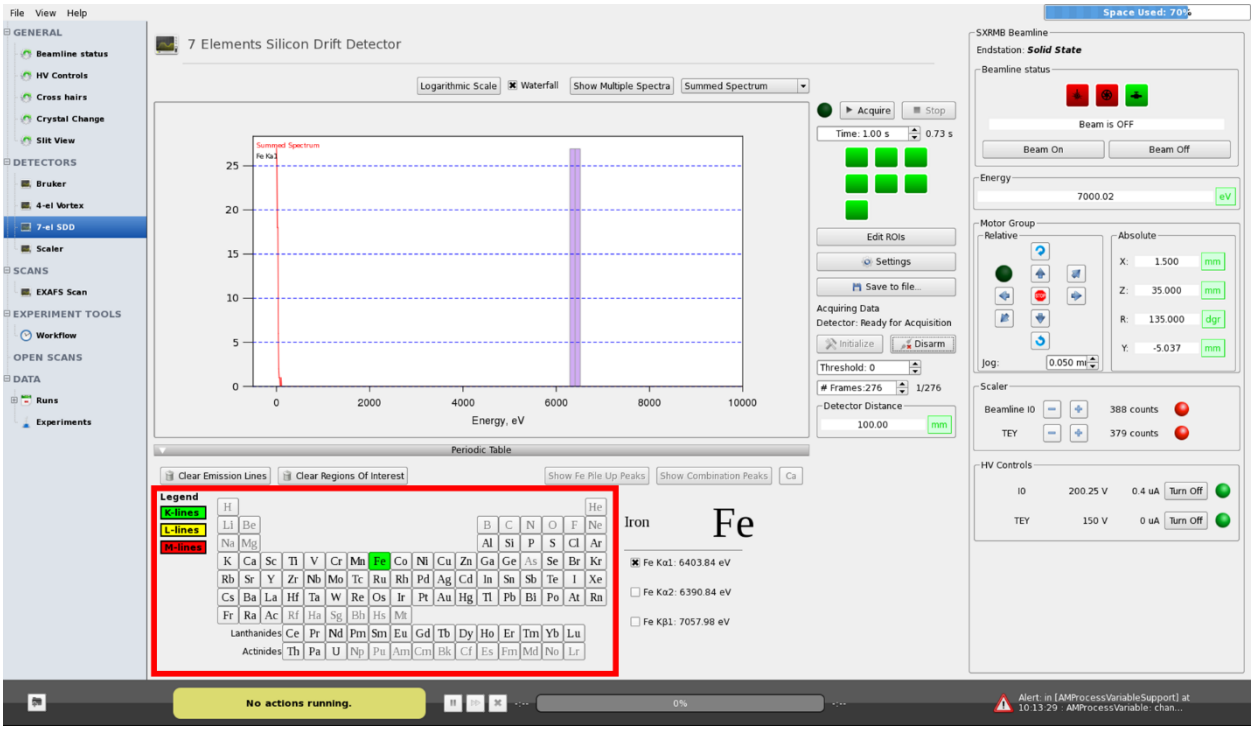
Bottom Bar: This is the status bar. When data is not being collected the "No actions running" status is displayed. If a scan is being collected, the name of the scan will be displayed, as well as [Running] with the number of scans beside it. An estimated percentage and time of completion are also displayed. You will also have the option to increase or decrease the number of scans using the up and down arrows, as well as the option to pause or cancel the current scan.

Setting up the 7-element silicon drift detector (SDD) for data acquisition

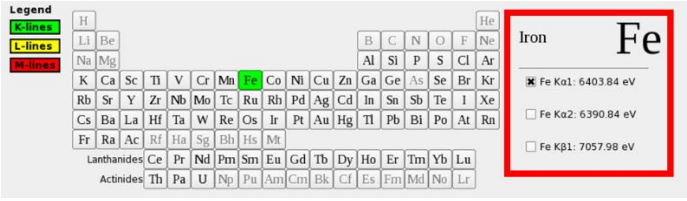
1. Select the '7-el SDD' tab on the left side of the Acquaman window



2. Select the element(s) you want to analyze from the periodic table

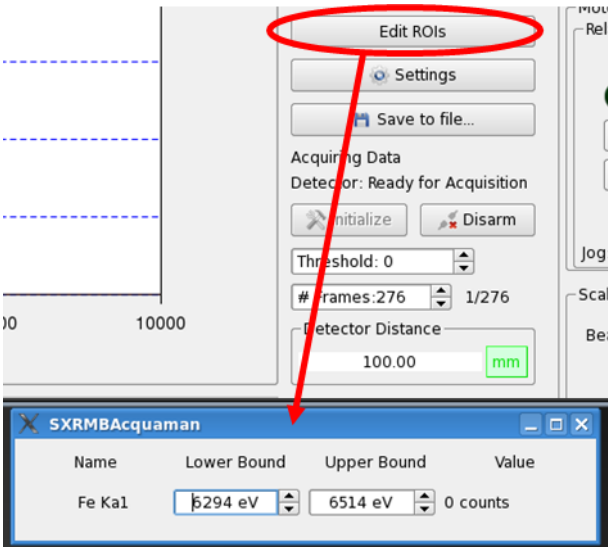


3. Select the emission line that you want to record during your scan



4. You will see the Region of Interest (ROI) highlighted on the spectrum above the periodic table.

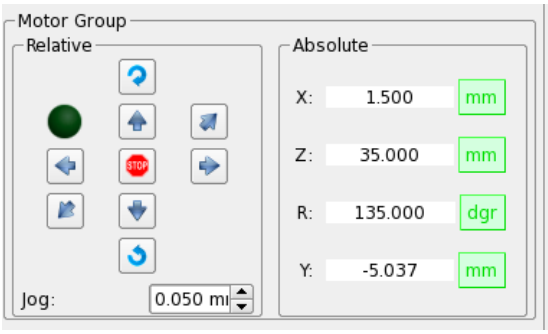
5. If you need to edit the region of interest:
    a. Click on the Edit ROI button to the right of the spectrum in the '7-el SDD' screen. A small window will pop up.
    b. Change the value of the lower or upper bound and adjust the window accordingly. In most cases pre-set bounds are good enough.
    c. The ROI should cover the emission line symmetrically in most cases.



The Edit ROI window will also display the counts for the selected emission line. This is useful for comparing relative concentration of each sample.

Motor Movements

On the right side of the Acquaman screen, you will see the Motor Group region. This controls the sample stage motors, enabling you to align your sample with the white box on the overhead monitor. In order to move your sample to the correct position, type in new values for X, Z, Y, and R. If you'd like to use the arrows that you see beside the input boxes, set the jog to a desired value then push an arrow to move the motors by the set jog value.



X = Horizontal. This motor moves from approximately -10 mm to +10 mm, the negative values moving to the left side of the plate.

Z = Vertical. The range of this motor is typically between 0 mm and 160 mm, the top being at 0 mm.

R = Rotation. For most analyses a rotation of 135 degrees is sufficient. Sometimes this value is changed for glancing angle studies (e.g. 110 degrees). If measuring samples on the back of the sample plate you may use -45 degrees or -70 degrees.

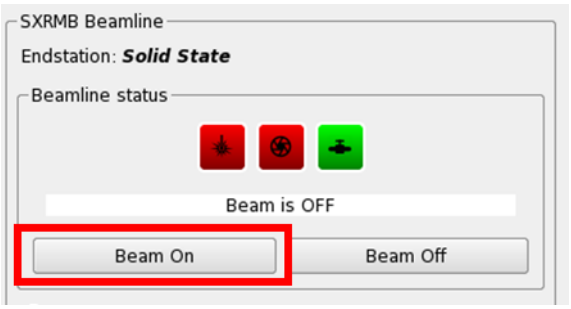
Y = Beam direction. It is common practice to leave this value at 0.000 mm, unless you are studying thick samples. For samples on the back of the plate use 3.5 mm.

Jog = The pre-set value for motor movements using the arrows to the left.

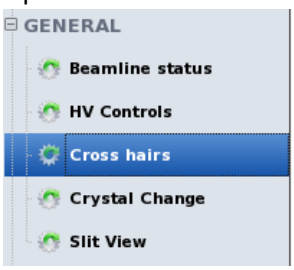
Locating Samples and Detector Positions for Measurement

1. Position the motors at X = 0 and Z = 0. This will position the X-ray beam on the YAG crystal above your sample plate.
2. Set the beam energy slightly above the edge of interest, at the white-line energy. Follow the calibration instructions (in a **separate** document) for changing energies from "low to high" or "high to low", or "low energy to another low energy" if applicable. **Note that the monochromator motors are not done moving until the "eV" button has turned green. You must wait until "eV" turns green.**

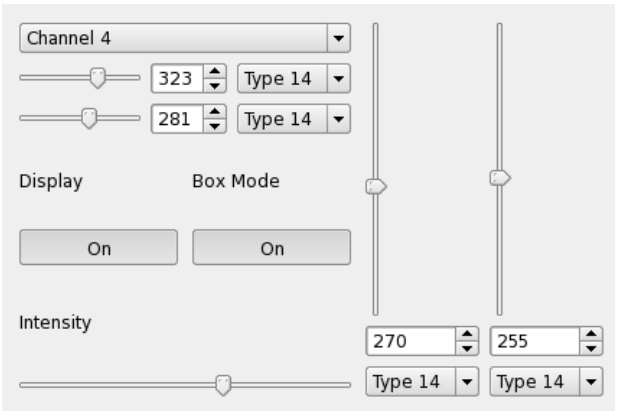
3. Turn the beam on using the button on the right side of the Acquaman window.



4. Open the Cross hairs window



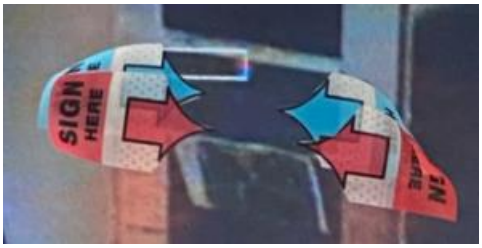
5. Adjust the sliders to position the white box on the overhead monitor around the X-ray beam. This will be one of your guidelines for determining sample positions.



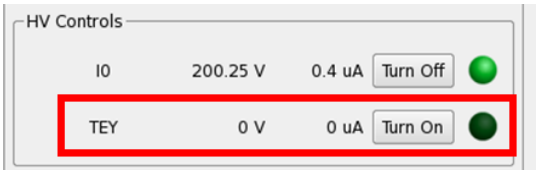
6. Move to the other YAG crystal(s) on your plate. Record the X and Z positions and mark the position on the overhead monitor using the arrow sticky notes. Now you have a visual of how the beam position changes as you move down the plate.

Manual Location of Samples

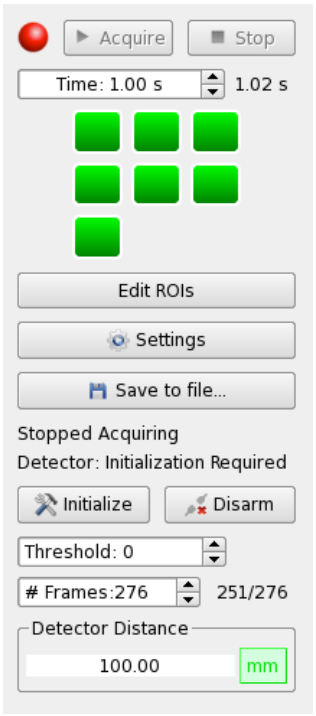
1. Use the white box and arrows as a guide and adjust the sample position (X and Z) so that the beam is centered on the sample of interest.



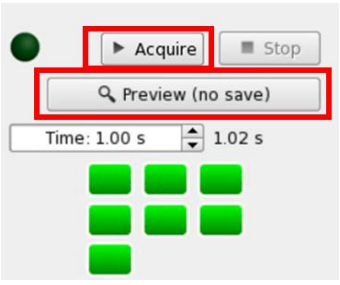
2. Return to the **7-el SDD** section of Acquaman and follow the directions in the *Setting up the 7-element silicon drift detector (SDD) for data acquisition* section of this document if you haven't done so already. Also turn on the TEY.



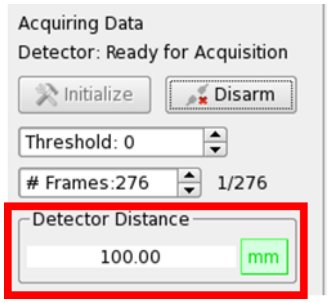
3. Initialize the detector by clicking "Initialize". The circle next to the acquire button will turn from red to green. If the "Initialize" button is greyed out, click "Disarm" and then "Initialize".



4. After detector set-up, click "Preview" or "Acquire" to obtain an XRF spectrum and observe the number of counts in the ROI window for the sample (If the window isn't visible, click "edit ROIs"). Jog by 0.5 mm in the Z direction on the sample to find the location where the number of counts for the sample is the highest and record the X, Z position.

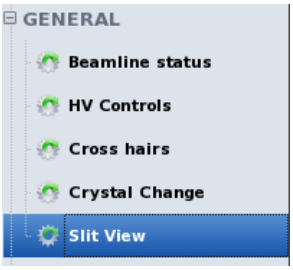


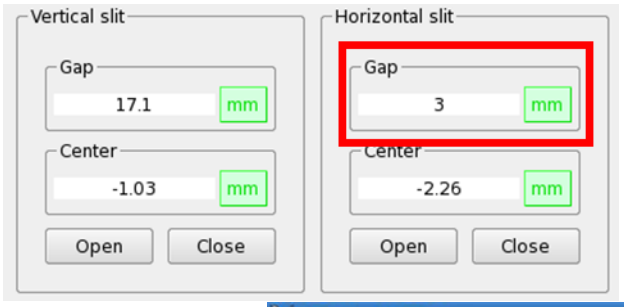
5. Typically, we set up the measurement conditions to obtain sample counts ***no more*** than 1 million. If the counts are too high or too low, the detector distance can be adjusted to a value from 5 – 200 mm in the 7-el SDD menu. 200,000 counts are enough to get a satisfactory spectrum. If the counts are still too high after moving the detector to 200 mm, the slits may be adjusted. Note that cutting the slits will affect the quality of your TEY signal.



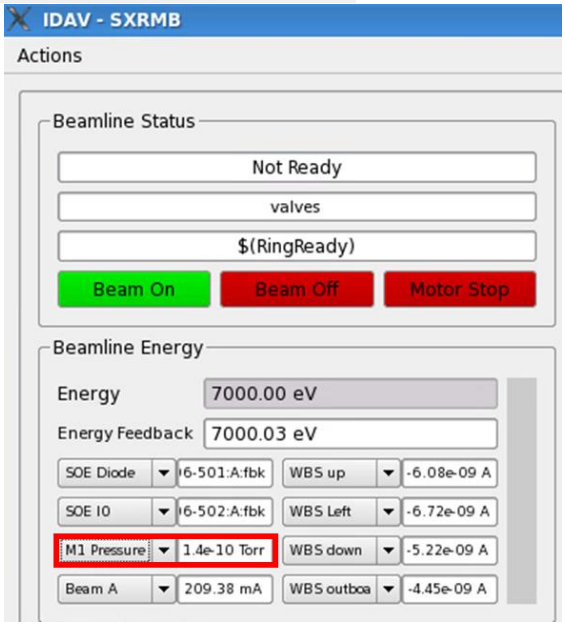
If counts are less than 20,000, you may consider measuring your sample for a longer time in your scan configuration (i.e. 4 seconds for each region instead of 1 second).

6. To adjust the slit size, click on the 'Slit View' menu, and adjust the horizontal slit gap. The maximum is 12.

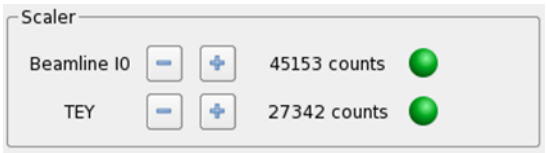




Keep your eye on the M1 Pressure value in the IDAV window. The beamline will trip off if the pressure reaches 5.5 × 10^{-7} torr. **If the pressure is at 5.3 × 10^{-7} torr or higher, the horizontal slit gap should be reduced.**



7. At your sample positions, also monitor the "Beamline I_0" and "TEY" counts. Ideally, they should be between 15,000 and 100,000 counts, with green circles next to them. The gain can be adjusted with the "+" and "-" buttons.

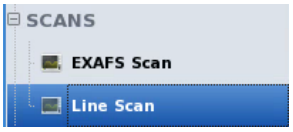


Line Scans for Determining Sample Positions

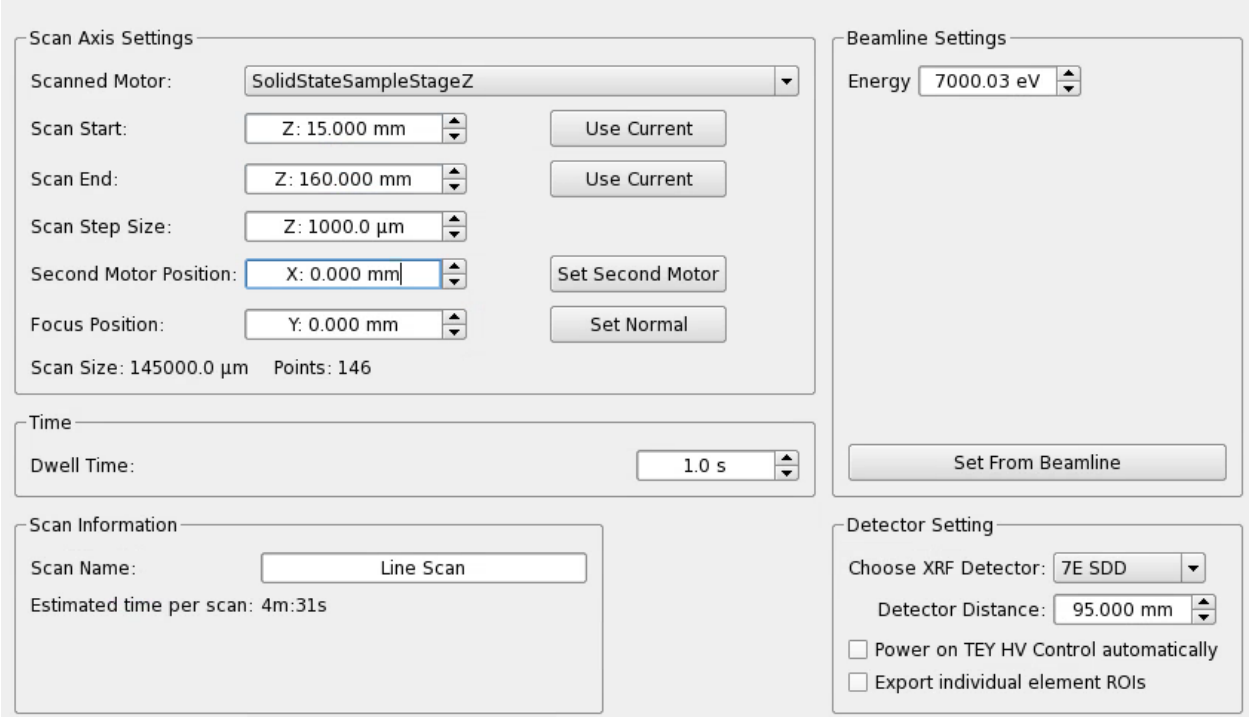
1. If the beam will hit all your samples adequately for the entire plate after testing beam position with YAG crystals, you can do a line scan at a single X position, and scan through Z vertically. If not, you can break your line scan up into multiple scans with different X values for each region.

2. You can manually look at the counts of one sample (see previous section) for insight into detector distance and slit sizes for your line scan if your samples have similar concentrations. Make sure the detector is set up for the element(s) of interest (see *Setting up the 7-element silicon drift detector (SDD) for data acquisition*).

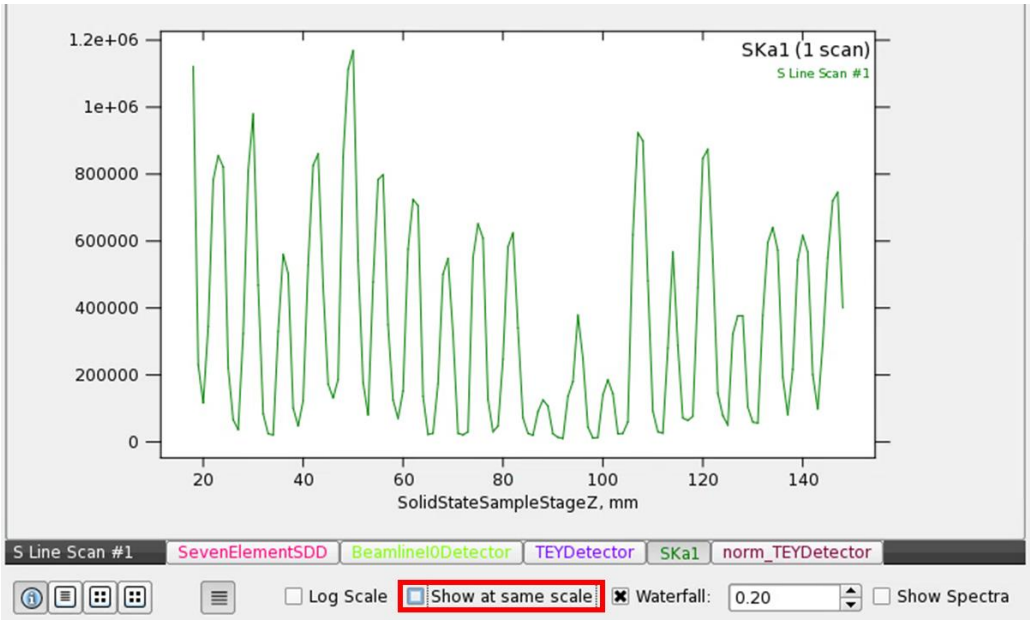
3. Select "Line Scan" under the SCANS menu.



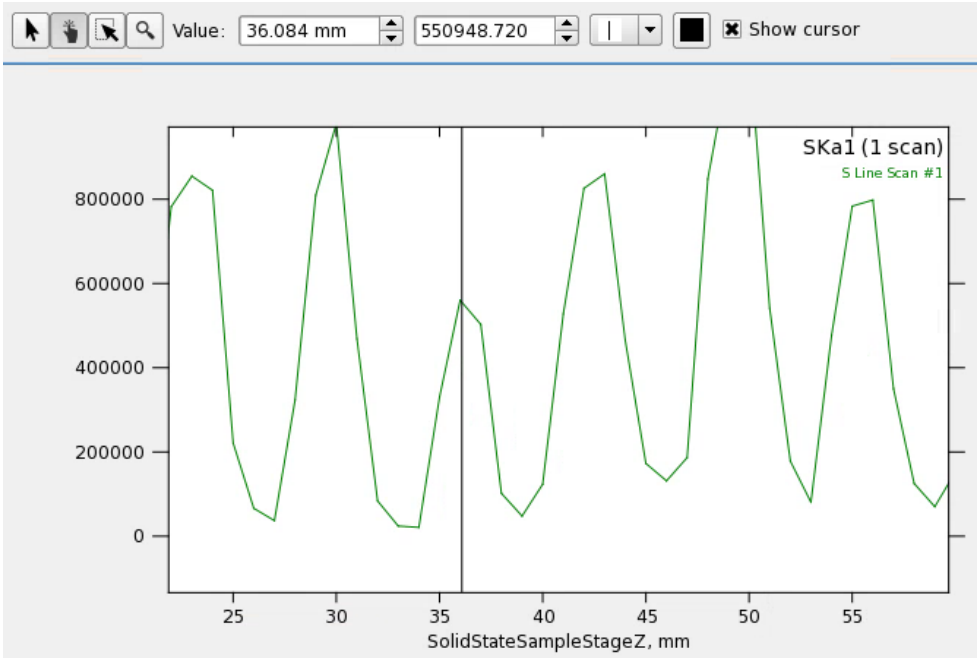
4. To do a vertical scan, select "SolidStateSampleStageZ" as the Scanned Motor. Select Scan Start and End Z-values. A scan step size of 1000 µm and dwell time of 1 s are adequate. If the samples are small you can reduce the scan step size to 500 µm or find your samples manually as described in the previous section. Set the energy to the white line energy, and set X and Y to the desired positions (most likely you can set these to the current value). Set your detector distance and note your current slit size (step 6 in *Manual Location of Samples*). Type a scan name and start the scan. (Note: the time estimate is not accurate. The scan will take 10-15 minutes.)



5. You will obtain a line scan with a peak at each sample position when the scan is complete. Uncheck the box “Show at same scale” to see the counts on the y-axis for the edge of interest. You can also look at the “TEYDetector” plot for TEY values. If your sample isn’t very concentrated you may not see it on the plot and may need to find the best measurement position as described in the previous section.



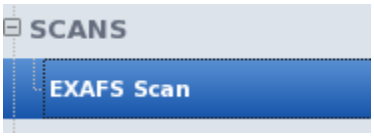
6. Above the plot there are buttons to Zoom in ( ) or select a point ( ). You can use these to read the Z values and counts from the plot. Right click to zoom back out. If your counts are too low (< 200,000) or too high (> 1 million), you can manually go to the sample Z-position (see *Motor Movements* section), change the detector distance and/or horizontal slit gap and preview the counts as described in the *Manual Location of Samples* section of the guide.



7. Record the X, Z, detector distance and slit values to be used for each sample.

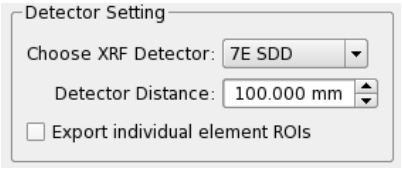
Setting up XANES or EXAFS Scan Regions

1. Select the EXAFS Scan tab from the left side of the Acquaman window

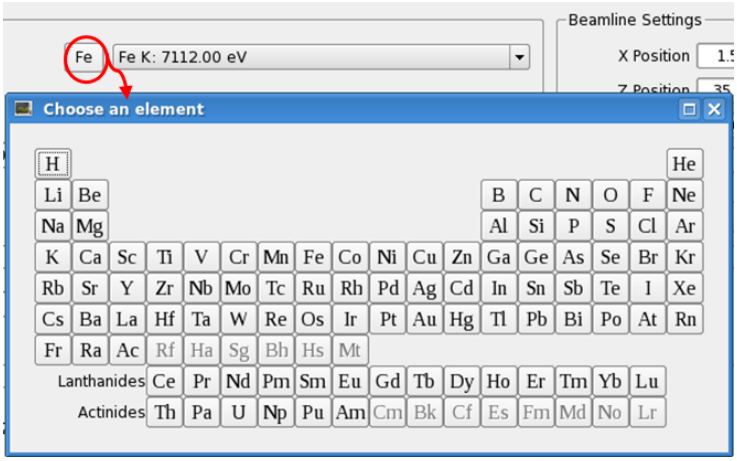


This is where the scan parameters are set up for the analysis you'd like to do.

2. Under the "Detector Setting" section, select "**7E SDD**" from the dropdown menu for the XRF detector.



3. Select the element and edge (K, L1, L2, L3) that you'd like to analyze. This will set the appropriate edge energy for the scan.



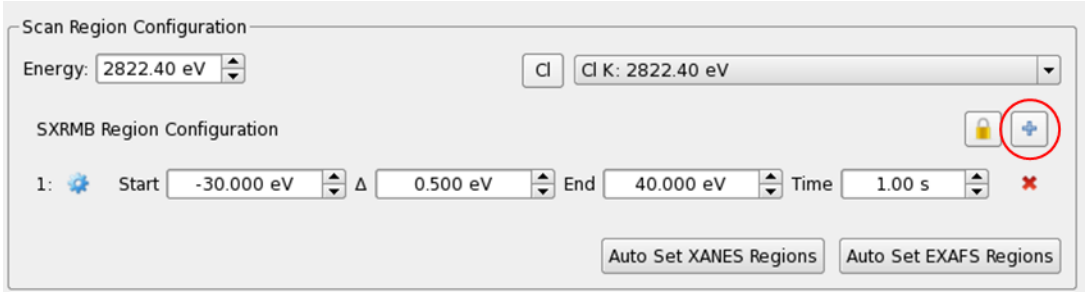
4. **XANES:** Generally has 3 regions; the pre-edge, the edge, and the post-edge. The pre-edge is generally flat and featureless. It is standard to use 2 eV steps up to 6 eV before the edge. The edge region is where a fine step should be employed. For low energy elements, a step size of 0.15 eV to 0.2 eV is used. Medium energy elements should have a step size of 0.2 to 0.3 and higher energy elements should have a step size of 0.3 to 0.4. To determine what step size you should use through the edge, see the table below:

Edge Energy (eV)	Suggested Step Size through the Edge (eV)
1700-2000	0.10
2000-4000	0.15
4000-5000	0.20
5000-6000	0.25
6000-7000	0.30
7000-8000	0.35
8000-9000	0.40
9000-9800	0.45

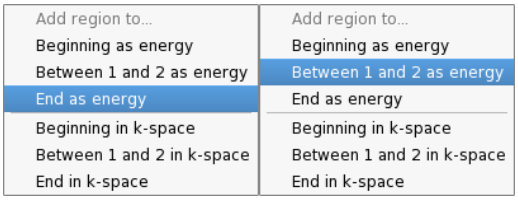
These step sizes are, however, a guide to get you started. You are free to increase or decrease the step sizes to achieve the data quality desired. The post edge generally has large, broad features so it is acceptable to increase your step size. For energies below 3000, a post edge step size of 0.75 eV is recommended. For energies above 3000, a 1 eV post edge step size should be sufficient.

To set up the configuration of the scan, you'll have to input energy ranges relative to the edge.

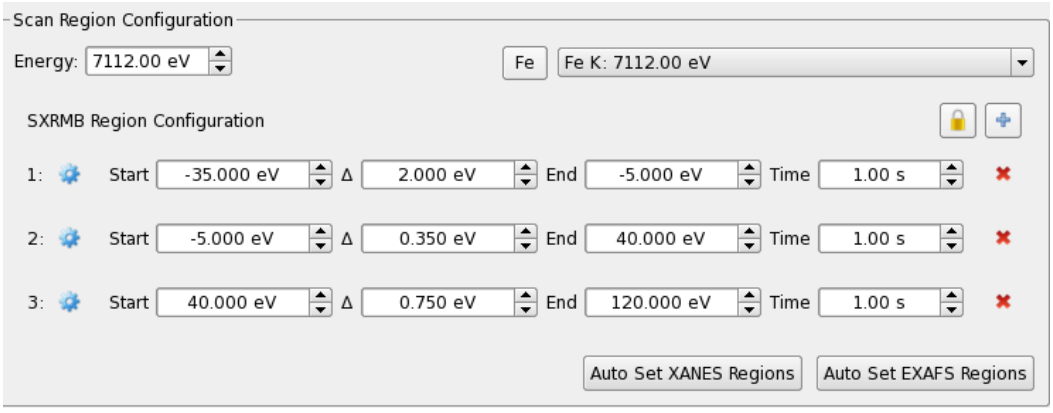
5. **XANES:** To add new regions, select the + button to bring up the region selection menu.



For XANES, select an energy option to add a row to your configuration. All of the step sizes and energy regions will be displayed in eV steps.

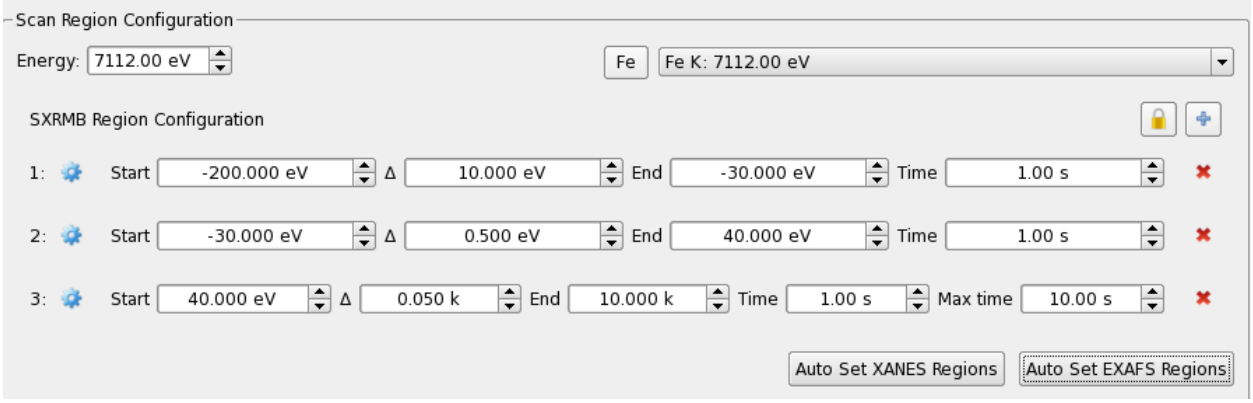


Adjust the start, end, step-size and time values to your desired measurement configuration.



6. **EXAFS**

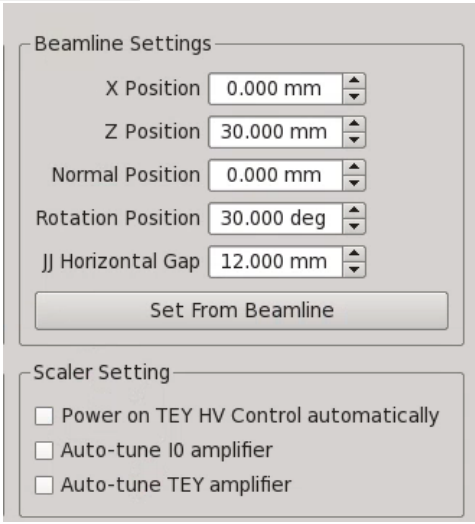
   Click on “Auto Set EXAFS Regions”. The steps and final energy will be displayed in units of k. The dwell time can also be chosen to increase with increasing k-space. Typically, a scan begins with 1 s dwell time and ends with 6 s.



Continued Scan Set-Up, Starting a Scan and Creating a Workflow

1. Make sure all the edges of interest for your workflow are checked off in the 7-el SDD tab.
2. In the “**EXAFS Scan**” menu, after setting up the scan region configuration, enter a “Scan Name” in the textbox below. Start the name with the element of interest, i.e. “Fe_Sample Name”. Note: you cannot use special characters like @, !, $, etc. in names.
3. Select the number of iterations in the lower right of the window.

4. Make sure the beamline and detector settings reflect the positions for the sample of interest, and then click “Start Scan”. You can also click “set from beamline” and the software will update the detector distance, positions, and slit size (JJ Horizontal Gap) for the scan to the *current* distance and positions.
5. You can select Auto-Tune I0 amplifier to auto-adjust the Beamline I0 counts into the appropriate range instead of adjusting the gain manually with the +/- buttons. This is particularly useful when your workflow has multiple elements or different slit sizes.
6. Select Auto-Tune TEY amplifier to auto-adjust TEY counts into the appropriate range. This is useful if samples vary in their TEY signals or you are using different slit sizes.



7. If checked, power on TEY HV Control automatically will Turn on the TEY HV if you forget to click “Turn On”.

8. Set up a workflow by entering the scan region configuration, sample name, sample position, and detector distance settings for subsequent samples, and clicking “Add to Workflow” after each sample. You can view your current workflow under the “Workflow” left-side menu. If you expand a Loop workflow item and double click on the sample name below, the “Show Configuration” button will appear, allowing you to edit your scan configuration.