

Acquaman User's Guide – Microprobe Endstation

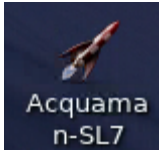
Table of Contents

1. Launching Acquaman.....	2
2. Navigating Acquaman.....	3
3. Setting up the 7-element silicon drift detector (SDD) for data acquisition.....	4
4. Motor Movements.....	6
5. Locating the Beam.....	6
6. Setting up a Coarse Scan for XRF Mapping.....	7
7. Setting up a Fine Scan for XRF Mapping.....	11
8. Preparing for a Micro-XAS Scan.....	11
9. Setting up the EXAFS Scan Configuration for μ -XAS.....	14
10. Continued Scan Set-Up, Starting a Scan and Creating a Workflow.....	17

October 4, 2023

1. Launching Acquaman

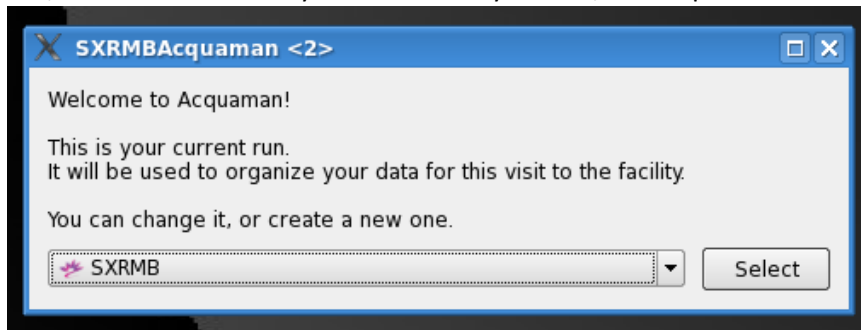
1. Open Acquaman by double clicking the icon on the desktop:



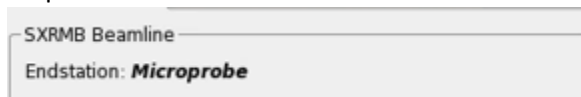
2. Put in the date in the proposal number box: YY-MMDD. This is used for your whole beamtime.



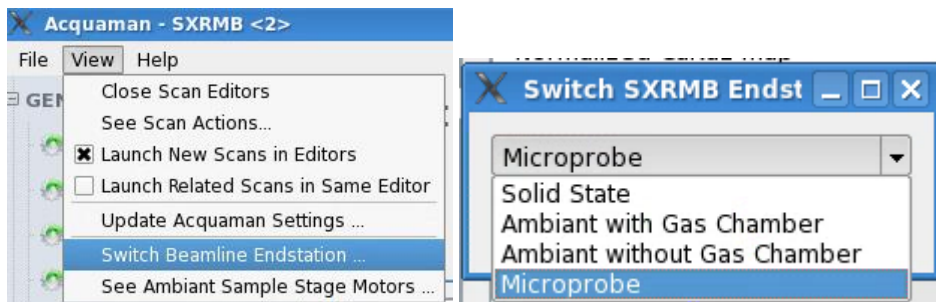
3. A window will open while the program is loading. If it is the first time you've used the "proposal number" it may take a few minutes to launch Acquaman. Please be patient.
4. Another window will open. Either select SXRMB or select the drop-down menu to make a new run, then select it. Once you've selected your run, the Acquaman interface will open.



5. Confirm you have the correct beamline endstation selected in the top-right corner of the Acquaman window

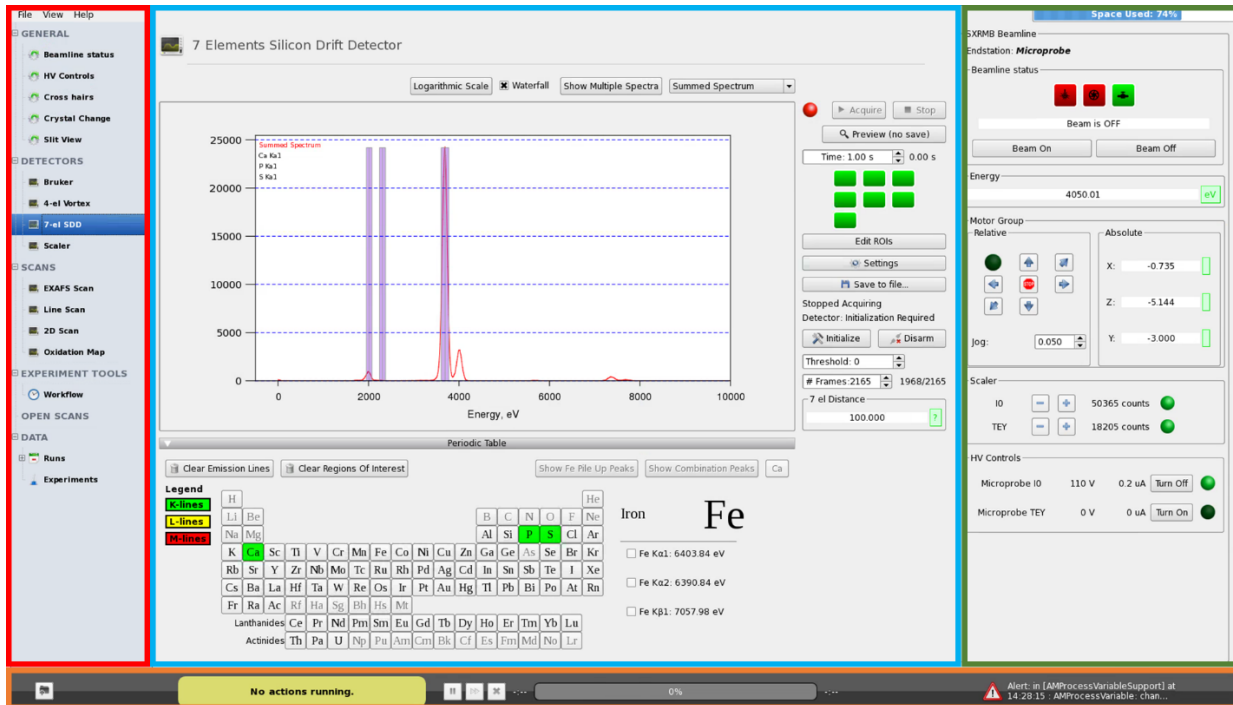


Switch the endstation if necessary by selecting "Switch Beamline Endstation ..." in the "View" menu.



2. 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.

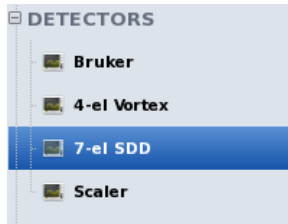
Right Section: These are the beamline settings. It shows the beamline status, manipulator motor positions, scaler output, and high voltage settings.

*****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.

3. 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

7 Elements Silicon Drift Detector

Logarithmic Scale Waterfall Show Multiple Spectra Summed Spectrum

Acquire Stop

Preview (no save)

Time: 1.00 s 0.00 s

Edit ROIs

Settings

Save to file...

Stopped Acquiring
Detector: Initialization Required

Initialize Disarm

Threshold: 0

Frames 2165 1968/2165

7 el Distance 100.000

SXRMB Beamline
Endstation: Microprobe

Beamline status

Beam is OFF

Beam On Beam Off

Energy 4050.01 eV

Motor Group

Relative Absolute

X: -0.735

Z: -5.144

Y: -3.000

Jog: 0.050

Scaler

IO 50365 counts

TEY 18205 counts

HV Controls

Microprobe ID 110 V 0.2 uA Turn Off

Microprobe TEY 0 V 0 uA Turn On

Legend

K-lines

L-lines

M-lines

Iron Fe

Fe Kα1: 6403.84 eV

Fe Kα2: 6390.84 eV

Fe Kβ1: 7057.98 eV

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

Legend

K-lines

L-lines

M-lines

Iron Fe

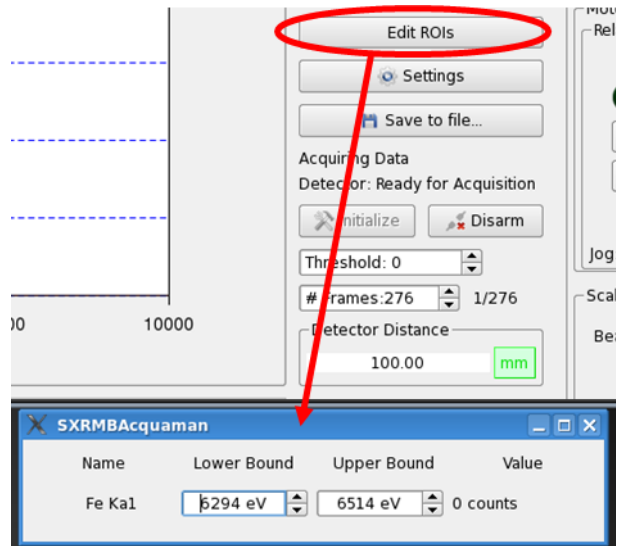
Fe Kα1: 6403.84 eV

Fe Kα2: 6390.84 eV

Fe Kβ1: 7057.98 eV

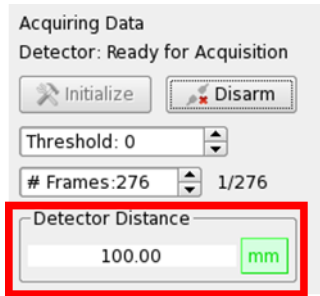
4. You will see the Region of Interest (ROI) highlighted on the spectrum above the periodic table.

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.



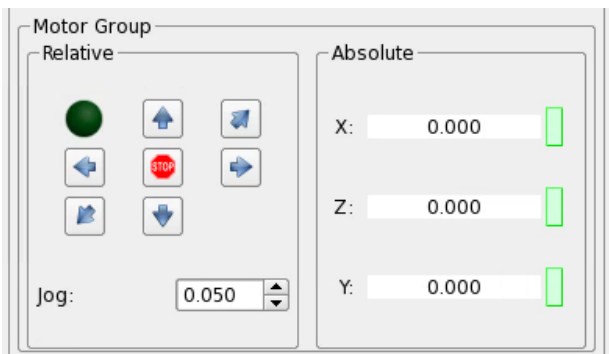
The Edit ROI window will also display the counts for the selected emission line. This is useful for comparing relative concentration of each sample.

6. The detector distance is typically set to 40 mm for XRF mapping experiments. However, this distance should be increased if the counts are greater than 1 million. The detector distance range is 40 to 195 mm.



4. Motor Movements

On the right side of the Acquaman screen, you will see the Motor Group region. This controls the sample stage motors. In order to move your sample to the correct position, type in new values for X, Y, or Z. 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.



For the microprobe endstation:

X = Horizontal. This motor moves from approximately -9 mm to +9.8 mm, the negative values moving to the left side of the plate.

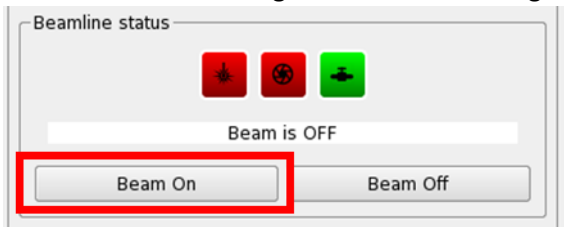
Z = Vertical. The range of this motor is typically between -12 mm and +12 mm.

Y = Beam direction/focus plane. It is common practice to leave this value at 0.000 mm, unless you are studying thick samples. Measure the thickness of your sample. The positive direction moves downstream. The range is -5 to +5.

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

5. Locating the Beam

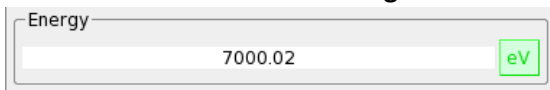
1. A YAG crystal is placed on your sample plate to help with locating the beam.
2. Set the beamline at a high energy (e.g. 7130 eV) initially to have a clearer view of the beamspot.
3. Turn the beam on using the button on the right side of the Acquaman window.



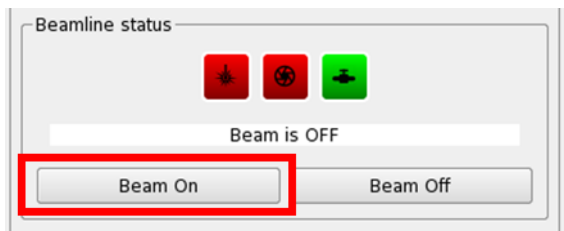
4. The beamline staff will help you adjust the motors and locate the beam on the YAG crystal. The beam will be marked on the monitor with sticky notes – at this point make sure **you do not** move the camera window on the computer.
5. After mapping scans, the sticky notes may be able to be fine-tuned to a more accurate position if there are distinct features on the sample that the motors can be moved to.

6. Setting up a Coarse Scan for XRF Mapping

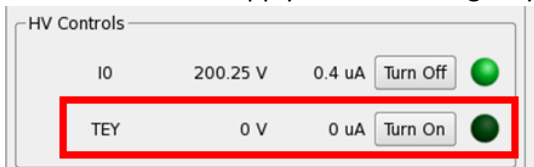
1. Prepare for XRF Mapping by following Section 3 and selecting the elements of interest that you'd like to map.
2. Turn the Beam OFF. Select an energy to use for mapping; generally, use the white-line energy for the element with the highest energy edge. If you plan on doing micro-XAS scans for a particular element later, you might choose to do the mapping at the white-line energy for that element. 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.**



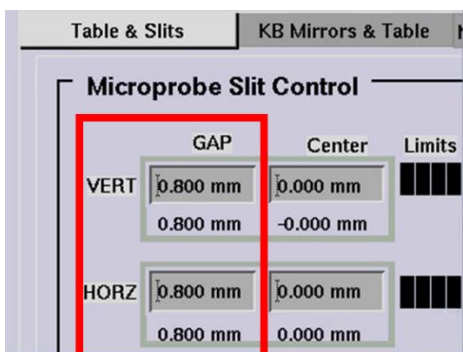
3. Turn on the beam.



4. Turn on the TEY to apply TEY bias voltage if you wish to collect the TEY signal.



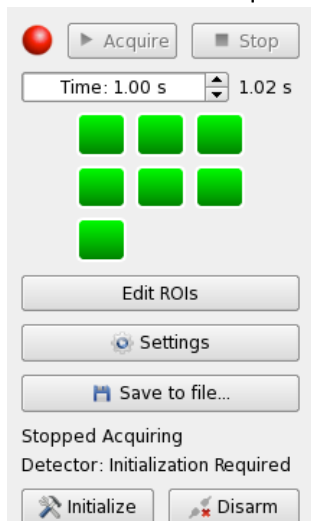
5. Set the horizontal and vertical slits to a gap of 0, if not already 0. Note: this is not done in Acquaman. There will be another window open on the right monitor with these controls.



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



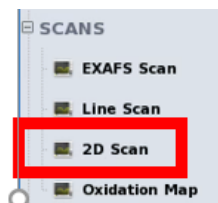
7. If the circle next to the Acquire button is red, initialize the detector by clicking "Initialize". The circle next to the acquire button will turn from red to green.



8. Position the beam on the sample, and 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").



9. If any counts are greater than 1 million, move the detector to a further distance and check counts again. The detector is typically set to a distance of 40 mm for XRF mapping, unless the sample is large enough to physically hit the detector at this distance, or the counts are too high.
10. Click on 2D scan from the menu on the left.



11. 2D scans are rectangular or square. The start point of a 2D scan must be the lower left corner and the end point must be the upper right. The camera view and the X and Z motors can be used to position the beam at the desired start point on the sample. You may also set a distance to jog and use the arrows to move the sample stage. Once at a desired point, you can click “Use Current” to set this point as the start or end of the scan.

Start:	H: -8.500 mm	V: -7.300 mm	Use Current
End:	H: -5.500 mm	V: -4.800 mm	Use Current

12. Select a step size for your coarse scan ($> 10 \mu\text{m}$).
13. Consider the “Estimated time per scan” when deciding on your step size and scan region in steps 8 and 9. Decide how much time you have available to dedicate to each scan. You will follow up this coarse scan with a fine scan of the areas of interests and hot spots identified from your coarse scan.

Scan Information

Scan Name:

Estimated time per scan: 1h:7m:33s

14. Set the dwell time to 0.5 s. This is typically enough time to get a good map.

Time

Dwell Time:

15. Give the scan a name. Include an identifier such as “CS” to indicate it is a coarse scan.

16. Click “Set From Beamline”. This will set your Beam Energy for your scan to the current energy, and the detector distance to the current distance (change this distance if necessary). The detector is typically set to a distance of 40 mm for XRF mapping, unless the sample is large enough to physically hit the detector at this distance, or the counts are greater than 1 million.

Beamline Settings

Energy:

Set From Beamline

Detector Setting

Choose XRF Detector:

Detector Distance:

Export individual element ROIs

17. Click either “Start Scan” or “Add to Workflow”, depending on if you are setting up multiple coarse scans of different areas.
18. You will get a map for each element you selected.

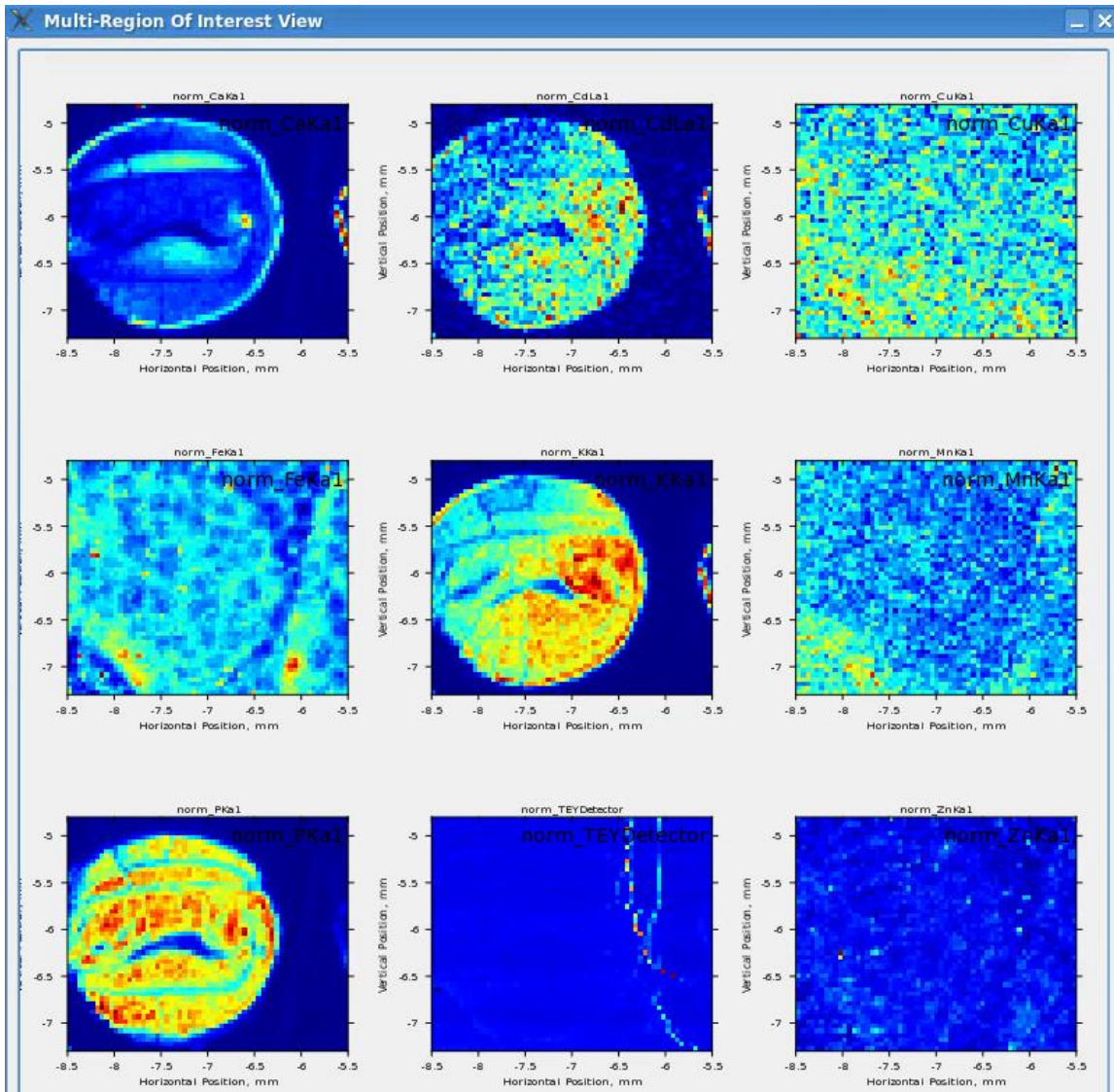
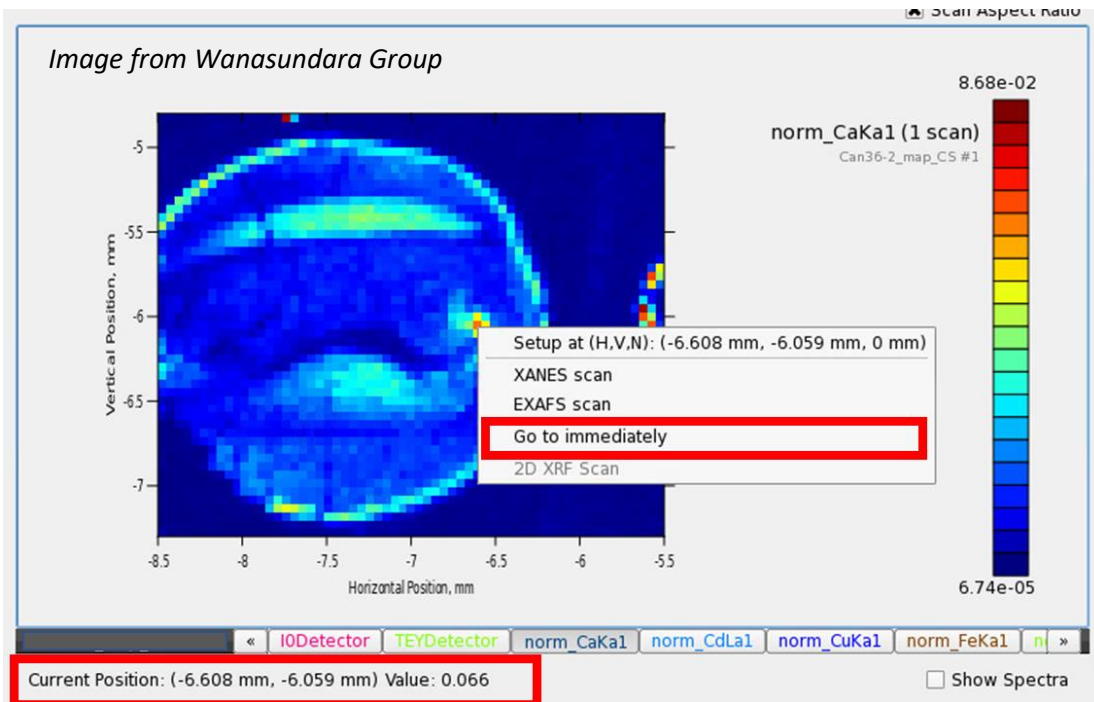


Image from Wanasundara Group

7. Setting up a Fine Scan for XRF Mapping

1. Examine your coarse maps and decide on areas of interest for fine maps. For example, you may want to fine map “hot spots” where the concentration of an element is the highest, or areas where one element is associated with other interesting elements for your research project.
2. To set up a fine map, *follow the same steps for the coarse map set up*. Instead of eyeballing your start/end points based on the sticky notes on the camera image, you may use the coarse map to move motors by doing the following:
 - a. Double click on the position you would like to move to on the image to update the “current position”.
 - b. Right click, and then select “go to immediately”.

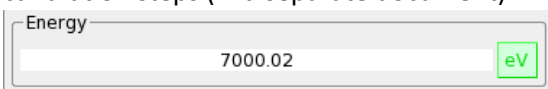


3. For a fine scan, use a Step Size of 10 x 10 μm

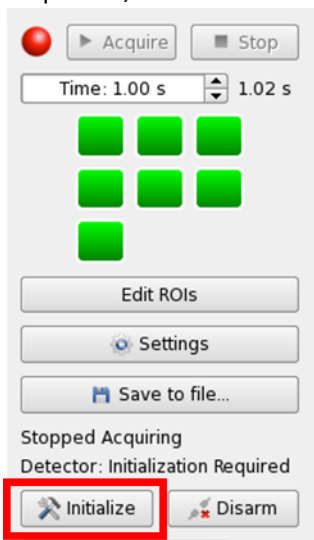
Step Size: H: 10.0 μm V: 10.0 μm
4. Use an identifier such as “FS” in the sample name to indicate it is a fine scan.

8. Preparing for a Micro-XAS Scan

1. Identify a sample location to collect micro-XAS on your fine scan map. Double click on it, then right click and select “Go to immediately”.
2. If necessary, change the energy to the white-line energy of the element of interest. Follow calibration steps (in a separate document) if needed.



3. If the XRF mapping was done at a high energy (>4000 eV), and the desired element edge for XAS measurements is low energy (< 4000 eV), there will be a small shift in the beam position. Consequently, a quick ~5 minute fine map of the area of interest is needed to accurately determine the measurement position. Using your last fine scan map, follow the directions in the last section to collect another fine map of a small region surrounding the point of interest.
 - a. Identify the new sample location for measurement using this fine map. Double click on the location, then right click and select “Go to immediately”.
4. 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 make sure the TEY HV control is on.
5. If the circle next to the “Acquire” button is red, and below it says “Detector: Initialization Required”, click the “Initialize” button.

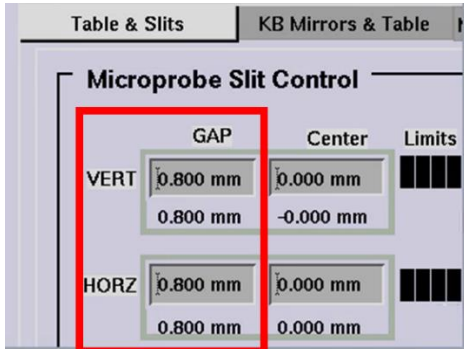


6. After detector set-up, click “Preview” or “Acquire” to obtain an XRF spectrum and observe the number of counts in the ROI window for the sample (If the window isn’t visible, click “edit ROIs”). Acquire saves the XRF spectrum, but it is not automatically exported to your data folder.
7. To protect the detector, set up the measurement conditions to obtain sample counts ***no more than 1 million***, and aim to have the counts be ~500,000. If the counts are too high or too low, the detector distance can be adjusted to a value from 40 – 195 mm in the 7-el SDD menu.



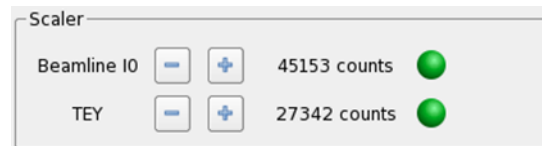
8. If the concentration is too low (e.g. less than 6000 counts) with the detector at 40 mm, the horizontal and vertical slit gaps can both be opened up to 0.5. However, this will increase the beam spot size to about $20 \times 20 \mu\text{m}$

Note: this is *not* done in Acquaman. There will be another window open on the right monitor with these controls.



If the counts are still low (< 2000 counts) after increasing the slit gaps, the scan times can be adjusted to 4 seconds in the next section.

9. At your sample positions, also monitor the “Beamline I₀” and “TEY” counts. Ideally, they should be between 10,000 and 200,000 counts/second, with green circles next to them. The gain can be adjusted with the “+” and “-” buttons.



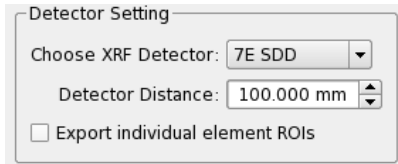
9. Setting up the EXAFS Scan Configuration for μ -XAS

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

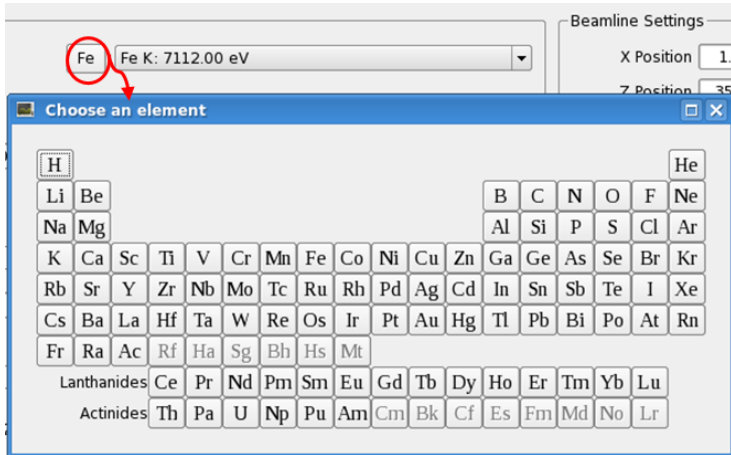


This is where the scan parameters are set up for the analysis you'd like to do.

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.



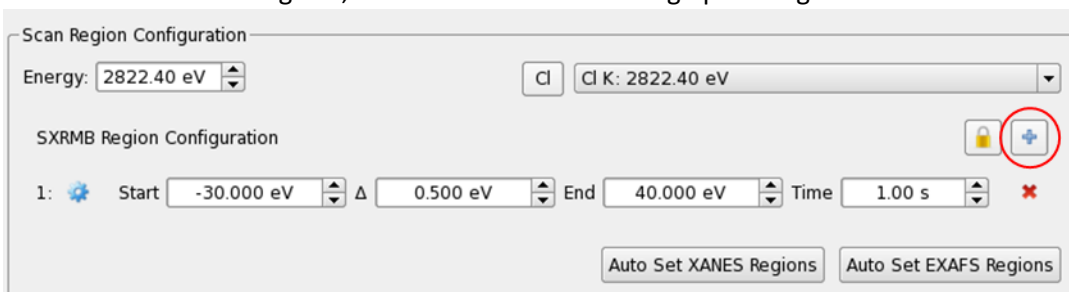
4. **XANES:** Generally has 3 regions; the pre-edge, the edge, and the post-edge. The pre-edge is generally flat and featureless. It is standard to use 2 eV steps up to 6 eV before the edge. The edge region is where a fine step should be employed. For low energy elements, a step size of 0.15 eV to 0.2 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

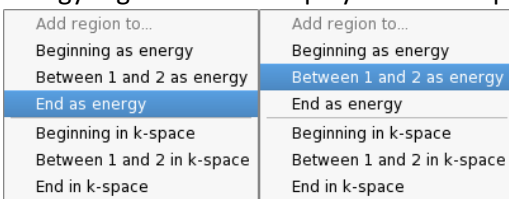
These step sizes are, however, a guide to get you started. You are free to increase or decrease the step sizes to achieve the data quality desired. The post edge generally has large, broad features so it is acceptable to increase your step size. For energies below 3000, a post edge step size of 0.75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

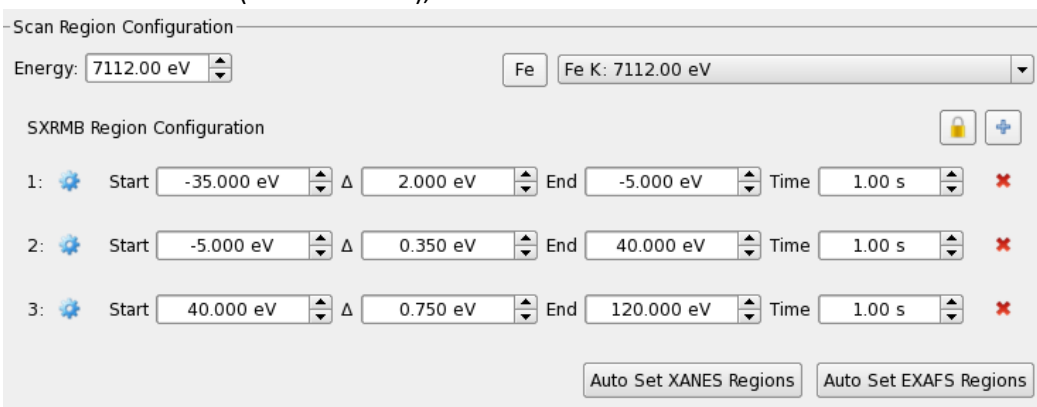
5. **XANES:** To add new regions, select the + button to bring up the region selection menu.



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.

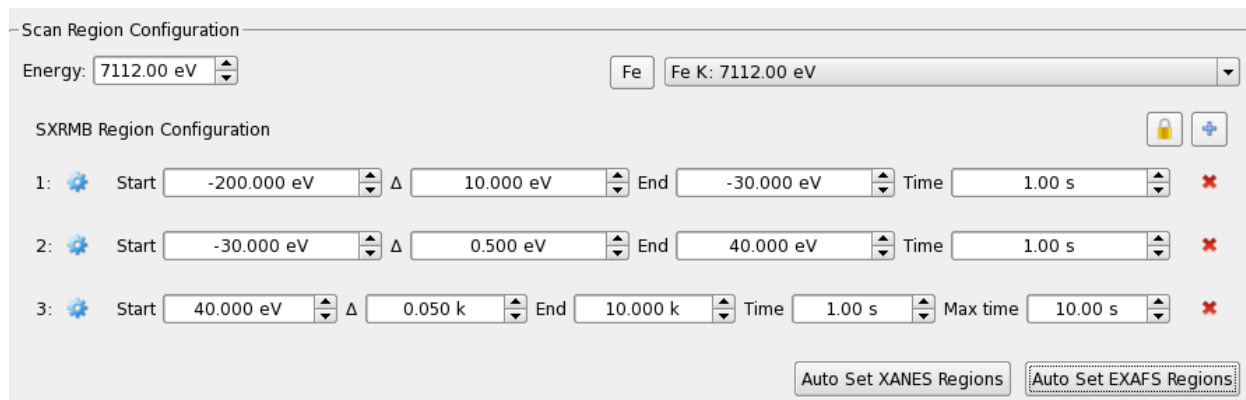


Adjust the start, end, step-size and time values to your desired measurement configuration. For low concentrations (< 2000 counts), 4 seconds is recommended.



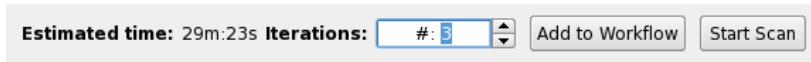
6. EXAFS

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.

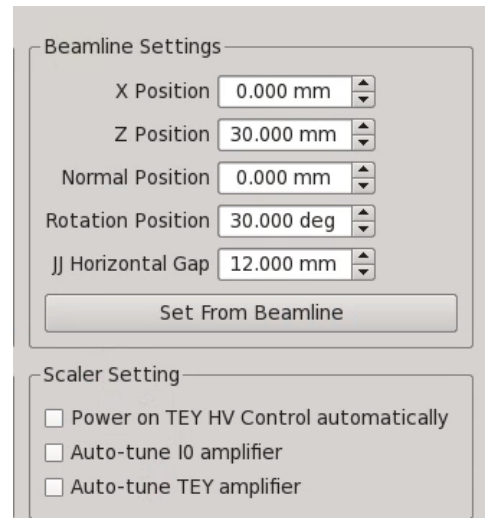


10. Continued Scan Set-Up, Starting a Scan and Creating a Workflow

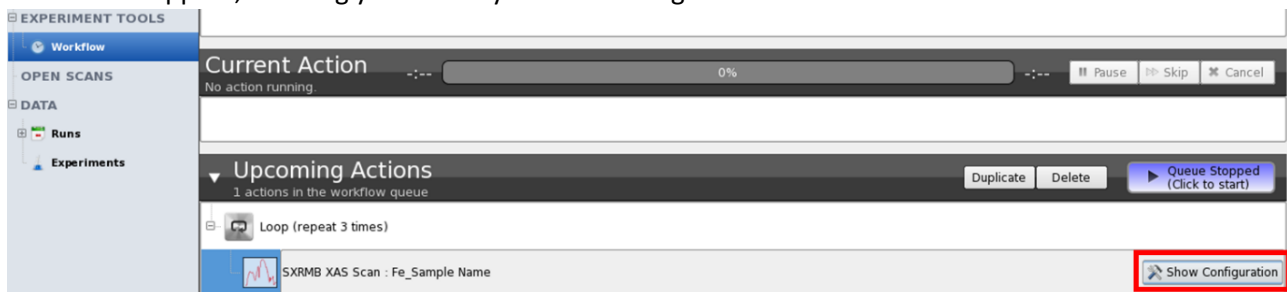
1. In the “EXAFS Scan” menu, after setting up the scan region configuration, enter a “Scan Name” in the textbox below. Start the name with the element of interest, i.e. “Fe_Sample Name”. Note: you cannot use special characters like @, !, \$, etc. in names.
2. Select the number of iterations in the lower right of the window.



3. Click “set from beamline” and the software will update the sample positions X, Y, Z, and the detector distance to the *current* positions.
You can select Auto-Tune IO amplifier to auto-adjust the Beamline IO counts into the appropriate range instead of adjusting the gain manually with the +/- buttons. This is particularly useful when your workflow has multiple elements. Select Auto-Tune TEY amplifier to auto-adjust TEY counts into the appropriate range. This is useful if samples vary in their TEY signals. Click “Add to Workflow”.



4. Move to the next measurement position using the “go to immediately” process, adjust detector distance if necessary based on counts, and then update the “Scan Name” and click “Set from Beamline” to update the samples positions and detector distance to the current positions. Click “Add to workflow”. Keep repeating to build your workflow.
5. 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.



6. Click the play button at the top of your workflow to start your workflow.



Workflows can also be created for 2D scans in a similar manner.