Acquaman User's Guide – Ambient Table

Note: There will be some differences if the 7-element SDD is used instead of the Bruker detector.

- Wherever Bruker is selected, select 7-el SDD or 7E SDD instead
- Ensure counts for the 7-element detector are < 1 million. You may need longer dwell times if counts are low. Consider 4 second dwell times when counts are < 20,000.
- Detector distance will be fixed and the detector clamped into place according to the set up.

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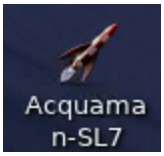
October 5, 2023

Launching Acquaman

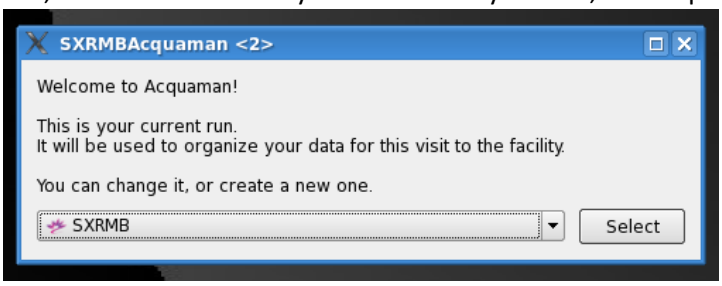
1. Open Acquaman by double clicking the icon on the desktop:



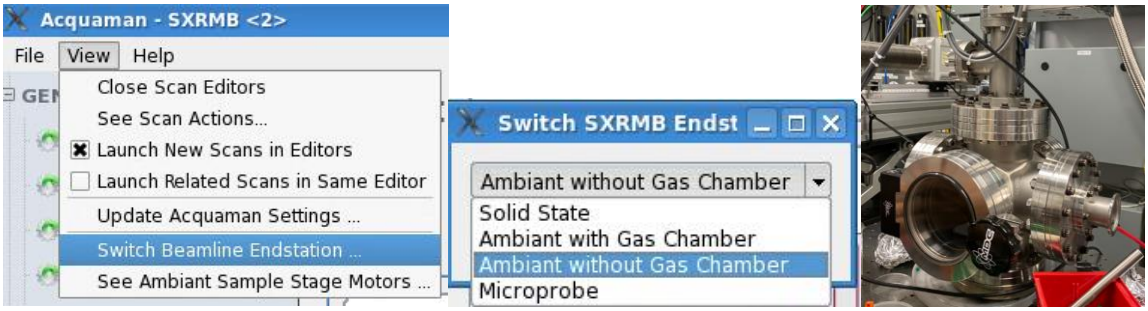
2. Put in the date in the proposal number box: YY-MMDD

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.

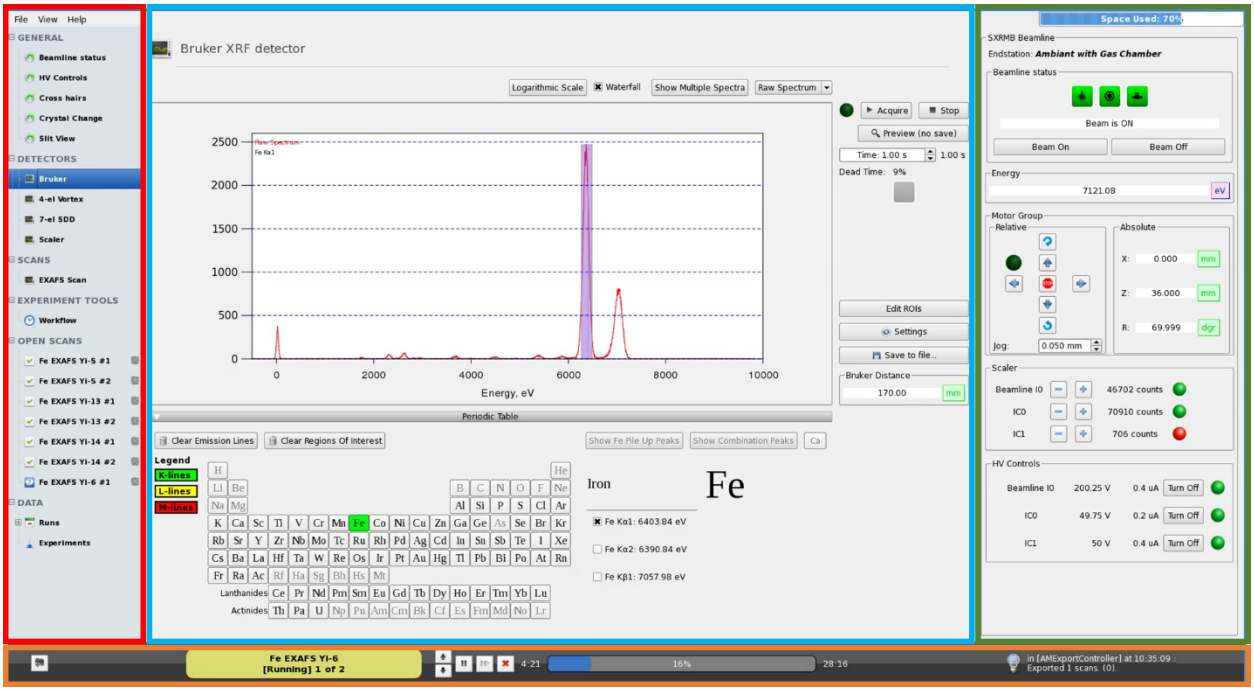


5. Confirm you have the correct beamline endstation selected by selecting "Switch Beamline Endstation …" in the "View" menu. If you are using the SXRMB gas chamber (shown on the right), make sure you select the "Ambient with Gas Chamber" option.



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.

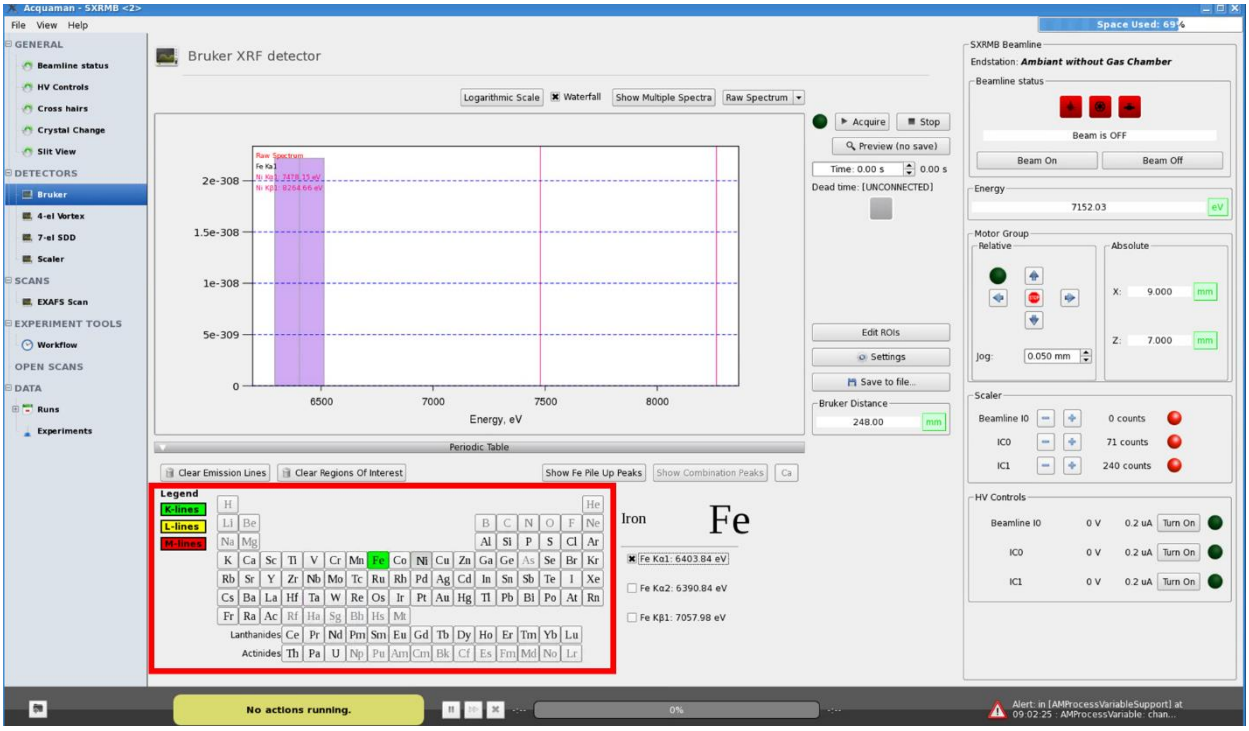
Right Section: These are the beamline settings. It shows the beamline status, manipulator motor positions, scaler output, and high voltage settings.

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.

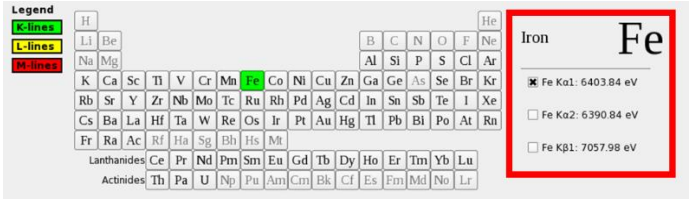
Setting up the Bruker detector for data acquisition

1. Select the ‘Bruker’ tab on the left side of the Acquaman window

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

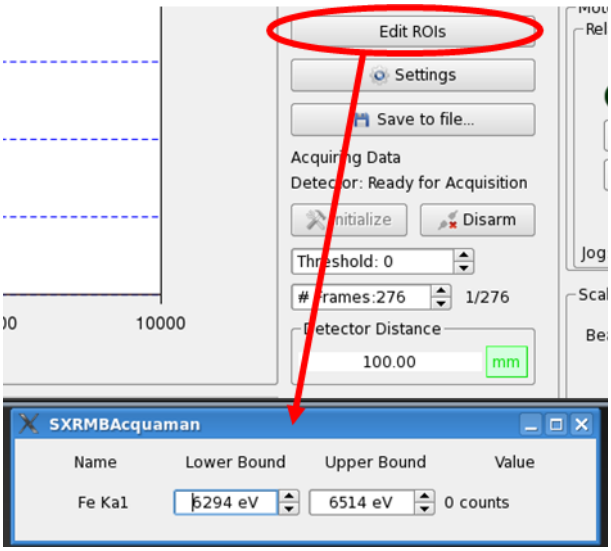


3. Select the emission line that you want to record during your scan



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

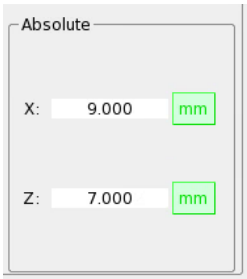
5. If you need to edit the region of interest:
    a. Click on the Edit ROI button to the right of the spectrum in the 'Bruker' screen. A small window will pop up.
    b. Change the value of the lower or upper bound and adjust the window accordingly. In most cases pre-set bounds are good enough.
    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 without Gas Chamber

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 beam. Without the gas chamber, the motors available are X and Z.



Beamline staff will help you locate the beam, and you should not touch these values. This may vary depending on your set-up, as the ambient table is versatile.

X = Horizontal. This motor moves from approximately -12.5 mm to +12.5 mm, if the centre position is set to zero.

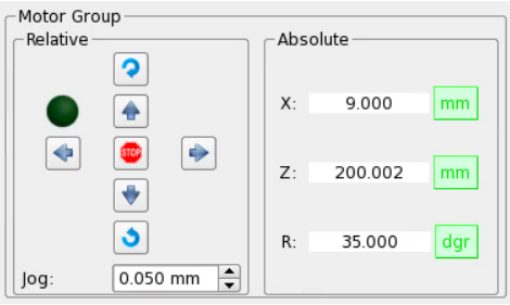
Z = Vertical. The range of this motor is typically from -13 mm to +13 mm, if the centre position is set to zero. **Caution: once the detector is mounted on the chamber, the sample stage vertical Z cannot be moved! Otherwise, the detector will be damaged!**

The sample's vertical height can be changed by changing whole table height (+/- 3.0 mm).

Motor Movements with Beamline Gas Chamber

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 beam. With the gas chamber, the motors available for movement are X, Z, and R.

In order to move your sample to the correct position, type in new values for X, Z, 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 (sample stage) = Horizontal. The range of this motor is +/- 10 mm.

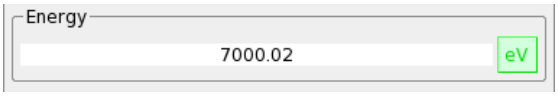
Z (sample holder) = Vertical. The range of this motor is -5 to 105 mm.

R (sample holder) = Rotation. For most analyses a rotation of 45 degrees is sufficient. Sometimes this value is changed for glancing angle studies. The range is from 0 to 360 degrees.

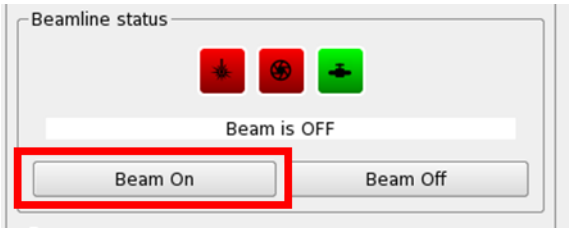
Once the beam has been located, in most cases you will only need to adjust the sample holder Z value. This will allow you to aim the beam at different samples on your sample plate.

Locating the Beam

1. Place a YAG crystal in your set-up, located where your samples will be loaded.
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.

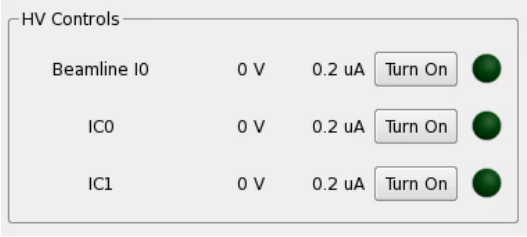


4. The beamline staff will help adjust the motors and set-up until the beam is on the YAG crystal. If the beam cannot be easily located, burn paper may be used.

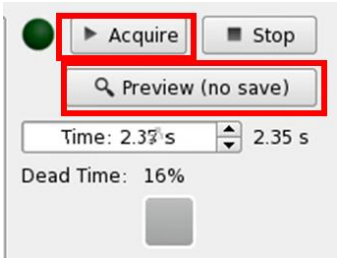
5. Once the beam has been located, it is likely for your set-up that you will not adjust the motor positions for the sample stage, but the Z and R positions for the sample holder may be adjusted if you are using the gas chamber.
6. Mark the beam position on the monitor using the arrow sticky notes. This will be your guideline for determining sample positions.

Location of Samples and Determination of Detector Distance

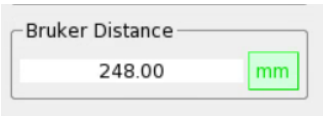
1. Use the arrow stickies as a guide and make sure they are pointing to your sample.
2. You may adjust the sample holder position (Z) so that the beam is centered on the sample of interest ***if you are using the gas chamber***.
3. Return to the **Bruker** section of Acquaman and follow the directions in the *Setting up the Bruker detector for data acquisition* section of this document if you haven't done so already. Turn on any HV controls that aren't on.



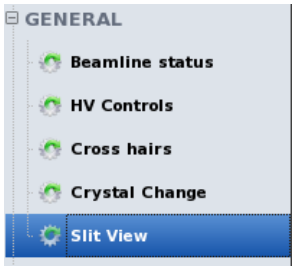
4. After detector set-up, click "Preview" or "Acquire" to obtain an XRF spectrum and observe the number of counts in the ROI window for the sample (If the window isn't visible, click "edit ROIs"). A percentage will also appear next to "Dead Time". The Time can be set to 1 second.

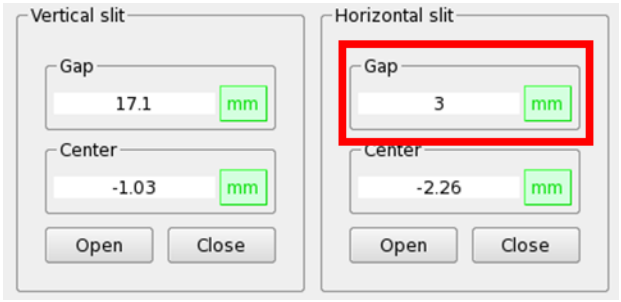


5. The detector begins saturating at a dead time of 20% and over 200,000 counts. Typically, we set up the measurement conditions to obtain a dead time of ~16-17% at the white line energy. If using the **gas chamber**, you can jog the Z value to find an appropriate measurement location (i.e. highest counts). Record the Z value.
6. If the dead time for a sample is too high or too low, the detector distance can be adjusted in the Bruker menu. **The detector distance limits will depend on your set-up.** If the dead time and counts are still too high after moving the detector to its maximum distance, the slits may be adjusted.
   If counts are lower than 2000, 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).



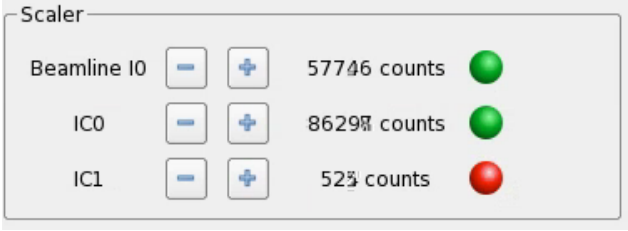
7. To adjust the slit size, click on the 'Slit View' menu, and adjust the horizontal slit gap. The minimum is 0 and the maximum is 12. **Do not** adjust the vertical slits.





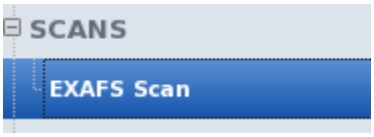
8. At your sample positions, also monitor the "Beamline I_0", "IC_0" and "IC_1" counts. Ideally, they should be between 15,000 and 100,000 counts, with green circles next to them.
If the energy of your scan is above 8000 eV, the IC_0 can be set around 100,000 counts at the white line energy, and if the energy is lower than 4000 eV, it can be set to around 50,000 counts.

   The gain can be adjusted with the "+" and "-" buttons. If you are not doing transmission measurements, you do not need to worry about the IC1 value.



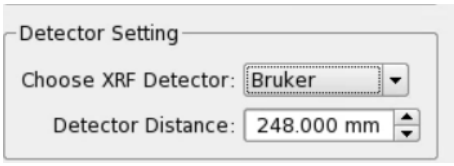
Setting up XANES or EXAFS Scan Regions

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

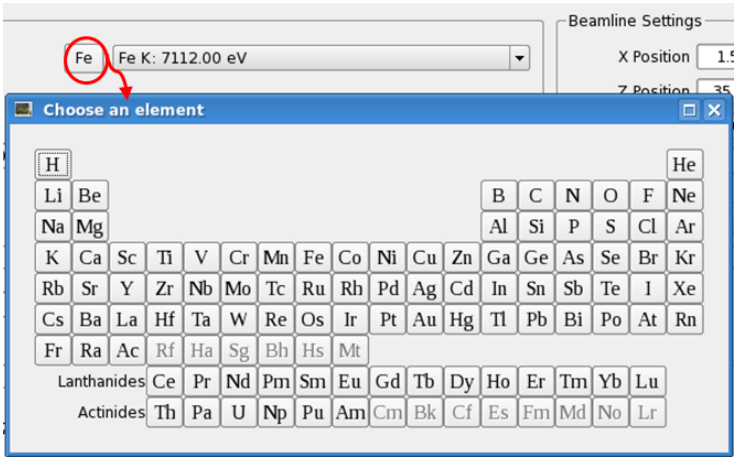


This is where the scan parameters are set up for the analysis you'd like to do.

2. Under the "Detector Setting" section, select "**Bruker**" 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.



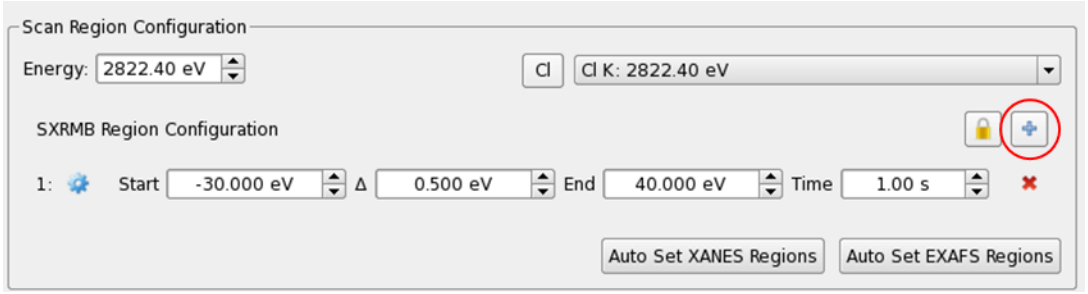
4. **XANES:** Generally has 3 regions; the pre-edge, the edge, and the post-edge. The pre-edge is generally flat and featureless. It is standard to use 2 eV steps up to 6 eV before the edge. The edge region is where a fine step should be employed. For low energy elements, a step size of 0.15 eV to 0.2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

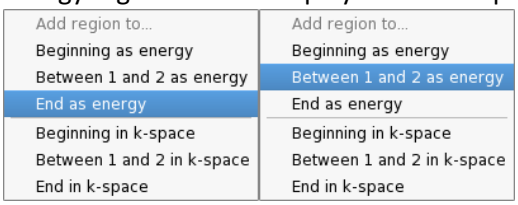
These step sizes are, however, a guide to get you started. You are free to increase or decrease the step sizes to achieve the data quality desired. The post edge generally has large, broad features so it is acceptable to increase your step size. For energies below 3000, a post edge step size of 0.75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

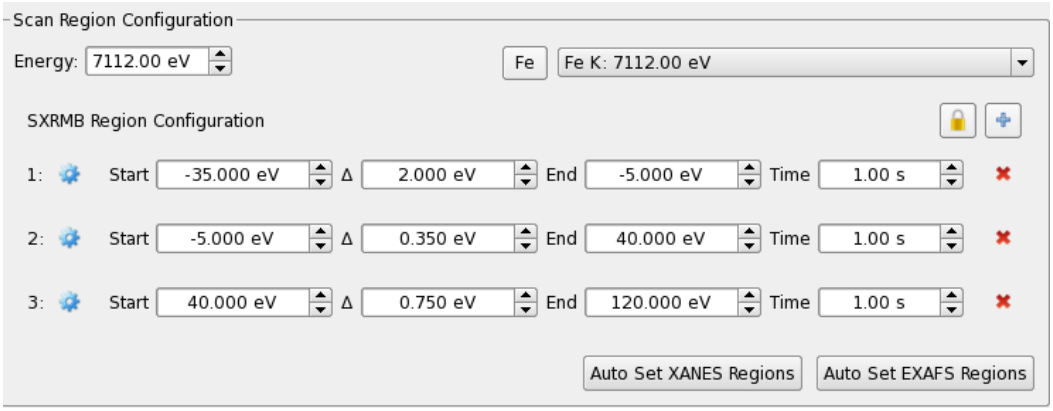
5. **XANES:** To add new regions, select the + button to bring up the region selection menu.



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.

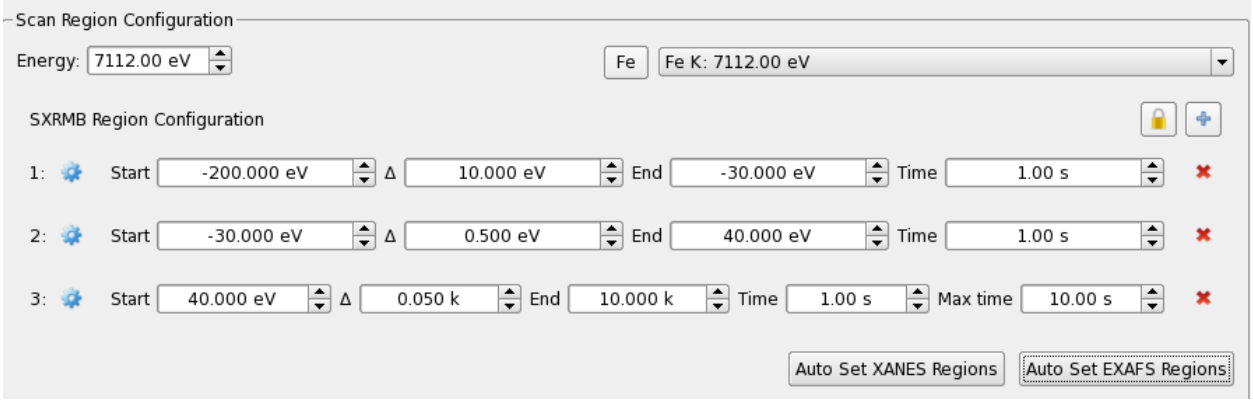


Adjust the start, end, step-size and time values to your desired measurement configuration.



6. **EXAFS**

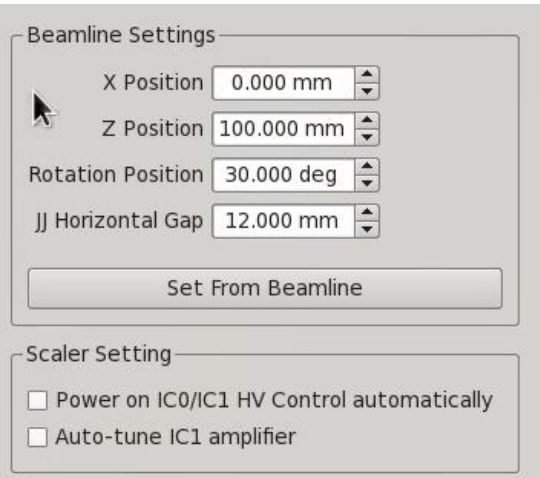
   Click on “Auto Set EXAFS Regions”. The steps and final energy will be displayed in units of k. The dwell time can also be chosen to increase with increasing k-space. Typically, a scan begins with 1 s dwell time and ends with 6 s.



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

1. 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. You can select Auto-Tune IC1 amplifier to auto-adjust the IC1 counts into the appropriate range instead of adjusting the gain manually with the +/- buttons.
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 as well as the positions for the scan to the current distance and positions.



5. 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.