



**Saint Mary's**  
**University**

## **Electron Microscopy Centre**

**Title:           RUNNING X-RAY (EDS) ANALYSIS ON THE SEM**

**Equipment: OXFORD ENERGY-DISPERSIVE SYSTEM (EDS) OPERATING ON LEO 1450VP**

**Revision:    1.0**

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## Running EDS on the SEM: Basic Instructions

1. Check dewar connected to detector column to make sure it has liquid nitrogen in it. If it does not, contact lab staff.
2. Check to see if the INCA computer is running (green light on front of the blue case on). If not, contact lab staff.
3. Logon LEO 1450VP user interface and run INCA software.
4. Load your specimen and standards (please wear provided powder free gloves only).
5. Turn on the filament and set accelerating voltage to desired operating conditions (if not sure, set 20kV).
6. Work on obtaining a sharp image using stage position (focus), stigmators, image brightness and contrast.
7. Leave the filament on for at least 15 minutes before optimizing (this is a good time to “survey” your specimen – do NOT leave the beam sitting unattended on your specimen as it will damage your specimen).
8. Set the working distance to **19 mm** (always focus using stage Z-drive) and adjust the spot size until the acquisition counts are between 2-3 k cps.
9. Optimize the calibration on the standard (Quantitative Optimization).
10. Return to your sample and begin collecting data (remember **not to** change working distance and spot size when collecting data).
11. When done collecting data, exit INCA software.
12. Turn off the beam and wait for 30 min so the filament is cooling down to room temp.
13. Remove specimen from chamber, close the door before pumping.
14. Log off the LEO user interface.
15. Remember to fill the log book.

## Running EDS on the SEM: Detailed Instructions

### 1. Check whether the dewar has liquid nitrogen in it.

If the liquid nitrogen level is low, the EDS system will make a buzz warning sound, contact the lab staff to refill the dewar.

If the dewar is empty and dried up, the standard LN<sub>2</sub> cool-down procedure should be followed, it will take 1.5 hours to proceed it. **DO NOT ATTEMPT TO ADD LIQUID NITROGEN ON YOUR OWN WITHOUT RUNNING THE SOFTWARE – YOU MAY EASILY DAMAGE THE EDS DETECTOR.**

### 2. Check to see if the INCA computer is running (green light on front of the blue case on).

If not, turn it on using the rocker switch located on the back on the machine at the very bottom.

Also check whether the EDS detector is located at its working location. If there is not previous usage, the detector should be retracted at resting location. Move the detector down to its working location by turning the knob until you cannot move it further.

### 3. Logon LEO 1450VP user interface and run INCA software.

There are two computers on the SEM, but only one keyboard and mouse. The monitor for the SEM computer is on the left - this is computer #1. The monitor for the EDS system is on the right - this is computer #2. The red number on the gray box in between the two monitors indicates which computer the monitor and keyboard are working with.

A quick way to switch the keyboard between two computer is simply hitting Ctrl-Alt-1, to switch the keyboard and mouse to computer #1 (the LEO system), or Ctrl-Alt-2, to switch the keyboard and mouse to computer #2 (the EDS system).

Click on the LEO icon on the SEM computer desktop and logon the software interface using the username and password assigned to you.


### 4. Load your specimen and standards (please wear provided powder free gloves only).

The first thing you will want to do is to load your sample. **Please make sure the filament has been turned off at least 30 minutes so to increase the lifetime the filament.** Do this by right clicking on the **Vac** button in the bottom-right corner and select **Vent** button at the popup window. This will vent the sample chamber, and

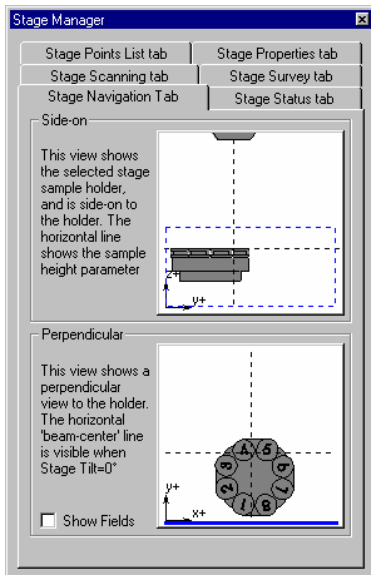
allow you to open the sample chamber door. It will take approximately 5 minutes to balance the pressure within the sample chamber with atmosphere. **Never pull the door during the 5 minutes venting period – the EDS window is quite fragile and expensive, a sudden pressure increase in the sample chamber would likely damage the EDS window.**



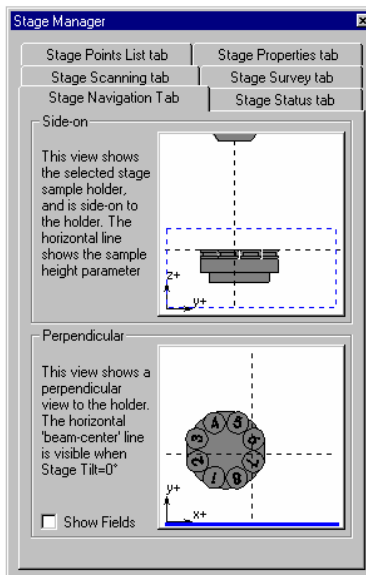
**Never reach into the sample chamber without gloves on!** Always use the sample exchange tool (tweezers and special designed screw-driver) to switch your sample with the one that is already in the chamber. Put the sample you just removed into covered box to prevent the dust. If you are not familiar with the sample exchange procedure or how the sample stubs fit onto the stage, please ask for help.

Once your sample is secure on the stage and positioned approximately where you want it, close the door, latch it, and **right click** the **Vac** button on the computer then select **Pump** to begin the pumping process. After about 5 minutes the **Vac** indicator  will show a ready sign. At that point, the sample chamber is under vacuum, and you are ready to begin viewing your sample. **It is always a good idea to wait a little longer until the vacuum reading is approaching  $10^{-6}$  torr.**

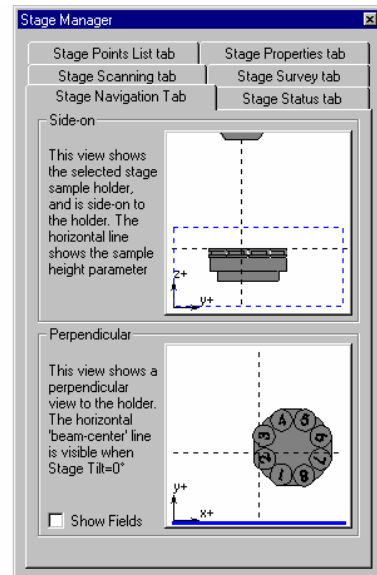
While waiting for the vacuum to be ready, adjust the sample stage and locate your first sample under the center of the column. In order not to hit on any detectors within the chamber, it is always a good practice to move the sample at front end of the stage under the column and rotate the stage to locate your interested one.



**Safe stage allocation**  
Simply turn the stage to reach next sample




**Unsafe stage allocation: sample 4/5 might hit detectors when moving along Z direction.**



Press Ctrl-Alt-2, to switch the keyboard and mouse to the EDS system. Start up INCA if it is not already running.

**5. Turn on the filament and set accelerating voltage to desired operating conditions (if not sure, set 20kV).**

Right-click on to **Fil** button (  ) and select **beam on** to turn on the beam. At this time, you should enter your information and the time-on into the SEM logbook.

Set your operating conditions: if your specimen is made of light elements, the EHT could be set between 5~10 kV; 10 ~20 kV is suitable for heavier elements. If you are not sure what number is good for you, 20 kV is a good start point

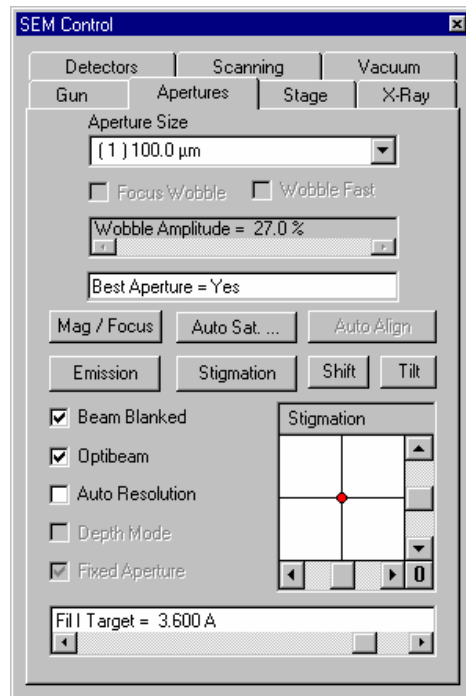
**6. Work on obtaining a sharp image using stage position (focus), stigmators, image brightness and contrast.**

In order to get an image, you will have to adjust the focus, contrast, and brightness. **It is always a good practice to start from a low magnification such 30x, 50x and switch to higher magnification.**

You could focus the image by selecting focus/mg mode and turning the '**FOCUS**' knob. In this case, the working distance (WD) physically remains but the electron beam focus on different level to match the WD. The WD value becomes REAL only when the beam is well focused on specimen.

You could also focus by changing the Z-position of your sample (this is the physical distance between the sample holder and the pole piece – the tapered column that hangs above your sample). **BE CAREFUL!** You might inadvertently ram your sample into the pole piece. Therefore, you may want to switch on the infrared camera (TV mode) so that you can pay attention to how close your sample is from the pole piece.

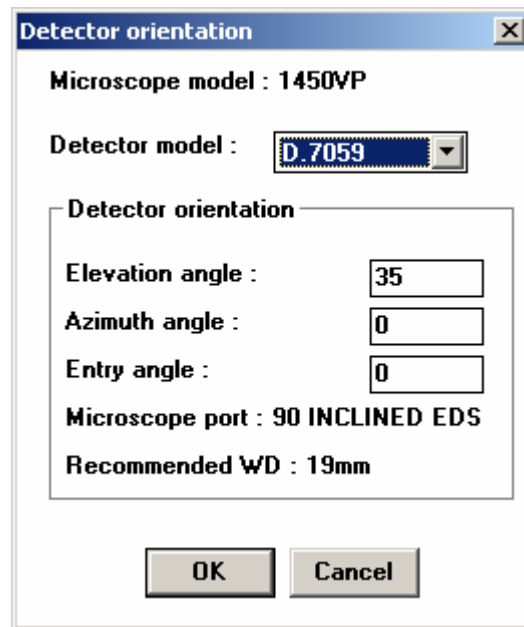
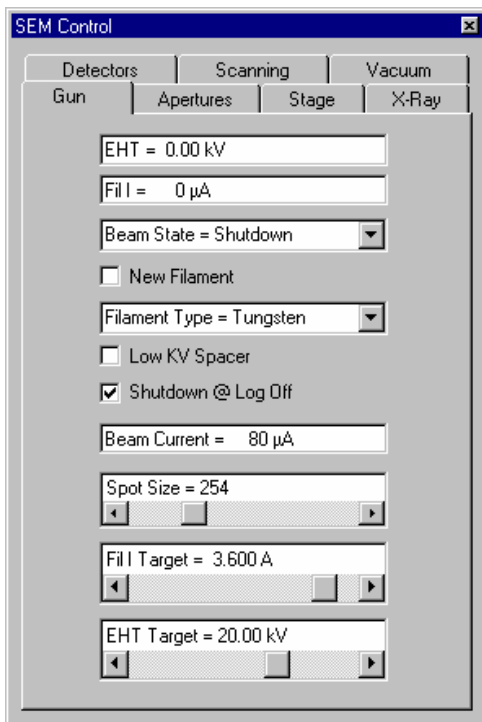
**Stigmation:** While you are still at high magnification, and once you have made your final focus adjustments, you should adjust the 'Stigma-X' and 'Stigma-Y' to try to improve the focus slightly. Moving between fine focus and Stigma adjustments at high magnification may provide small, but sometimes significant improvements to the quality of your image.



7. **Leave the filament on for at least 15 minutes before optimizing (this is a good time to “survey” your specimen – do NOT leave the beam sitting unattended on your specimen as it will damage your specimen).**

Please remember to check whether “Remcon 32” software is running. The software is designed to set up the communication between SEM control computer and EDS system. If the software is not running, you can still collect x-ray information but you would be unable to perform Point & ID and Mapping function within EDS. Run the software if you wish to have these functions on. The icon for the program is sitting below the LEO icon.

8. **Set the working distance to 19 mm (always focus using stage Z-drive) and adjust the spot size until the acquisition counts are between 2-3 k cps.**



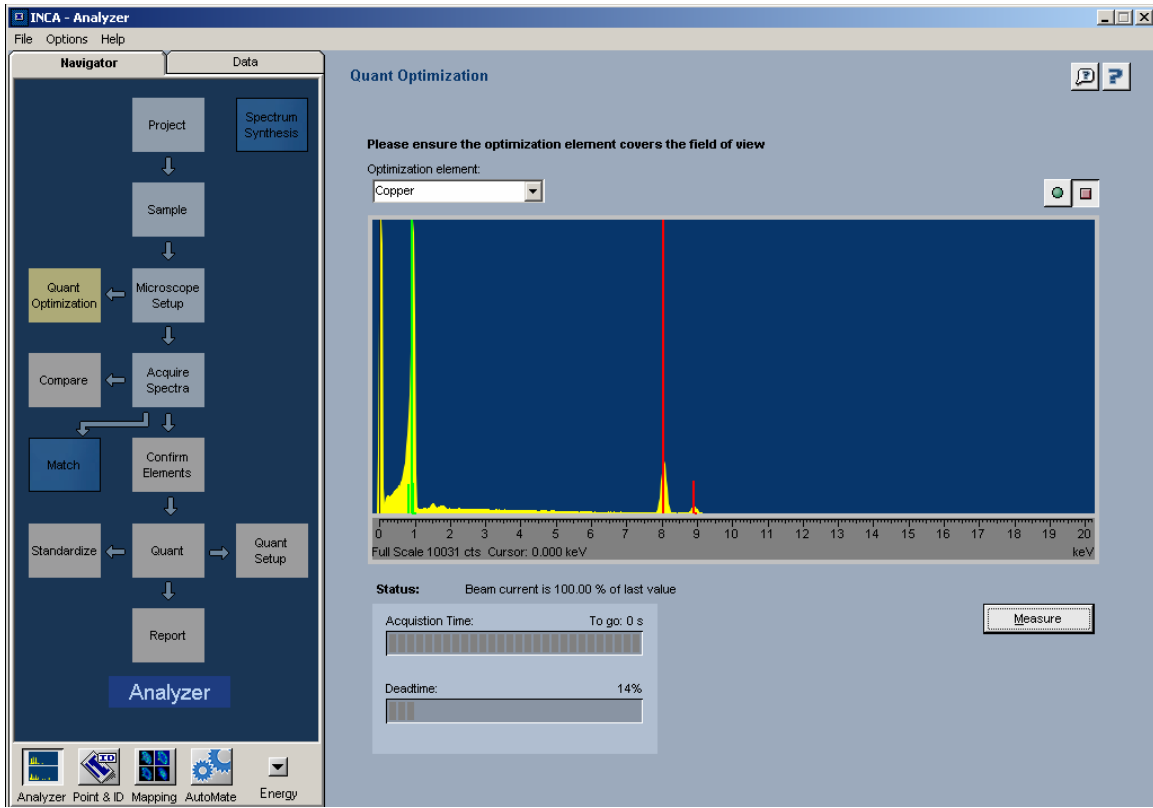
9. **Optimize the calibration on the standard (Quantitative Optimization).**

If you wish to work with un-normalized results, you will have to optimize the calibration before you begin your analysis. In order to obtain consistent quantitative results, the electron beam must be very stable. **It is inadvisable to begin doing quantitative x-ray work if the beam has not been on for AT LEAST 15 minutes.**

Position over the Cobalt standard, increase the magnification to 10,000X, and set the SEM to your optimal operating conditions (IMPORTANT: once you have optimized the detector, you must use the same operating conditions for all subsequent analysis

in order for your results to be quantitatively accurate. If you wish to change settings, you may need to go back and re-optimize with your new operating conditions).

Now, click on the box labeled 'Quant Optimization'. Press the green record button in the top right corner. After the detector is finished building a spectrum, click 'Measure'. The software will tell you how close this optimization is to the previous value, allowing you to monitor beam current stability (the closer to 100.00%, the more stable the beam current).



## 10. Return to your sample and begin collecting data (remember not to change working-distance and spot size when collecting data).






Back off to the lowest magnification you can so it is easier to switch your specimen back under the electron beam. Once you've found a spot you would like to analyze, you are ready to send your image over to the EDS computer. Depending on your sample type, you may wish to view your sample in secondary (SE) or Backscatter (BSD, if fitted) mode. In BSD mode, objects with a high atomic number will be brighter, where those with low atomic numbers will be darker. For geologic samples, this may help you to see minerals of different compositions that you may not have otherwise seen in Secondary (SE).

You will have to adjust your contrast and brightness to get a good image. Once you have an image you would like to analyze, move over to INCA on the EDS side. If you are beginning a new project, type in the name of your project, and any notes you

would like in the area provided. **Every project can have multiple samples (eg. thin sections), and each sample can have multiple sites of interest (areas of the sample that you would like to analyze).** Move down the flowchart to Sample, and enter your sample name, ID, and any notes you think appropriate. Also, select “this sample is polished” and “this sample is coated” if they apply. If your sample is coated, also select the medium it was coated with – this way INCA will not include it in its analysis.

If you wish to do spot analysis, select the ‘Point-and-ID’ tab at the bottom of the screen. If you wish to do Linescans or element maps, select the ‘SmartMap’ tab. Then select the box labeled ‘Site of Interest’. Click on the green record button. Your image will transfer over. You can adjust the brightness and contrast by clicking on the button, clicking and holding the left mouse button down in the middle of the cross-hair, and dragging it around until you get an image that you like. When you are satisfied with your image, select ‘OK’. Now go back to the SEM side and change your spot size back to your optimal operating condition. Your image will most likely disappear, but this doesn’t matter.

### **Point-and-ID**

In INCA, if you are working under ‘Point-and-ID’, move down the flowchart to ‘Analysis’. Here there are several tools you may utilize. When the small crosshair is selected , you may simply point-and-click anywhere on the image to get an elemental analysis at that spot. There are several area tools, which allow you to select boxes , freehand areas , or automatically selected areas  for analysis. The last tool, which looks like a bulls eye , works like the crosshair, except that it shows you exactly where the data is being produced, since the size of the area that can be analyzed is of a finite size. This is more useful at high magnification. You may select as many spots as you like – as soon as the software is done analyzing one spot, it will move to the next.

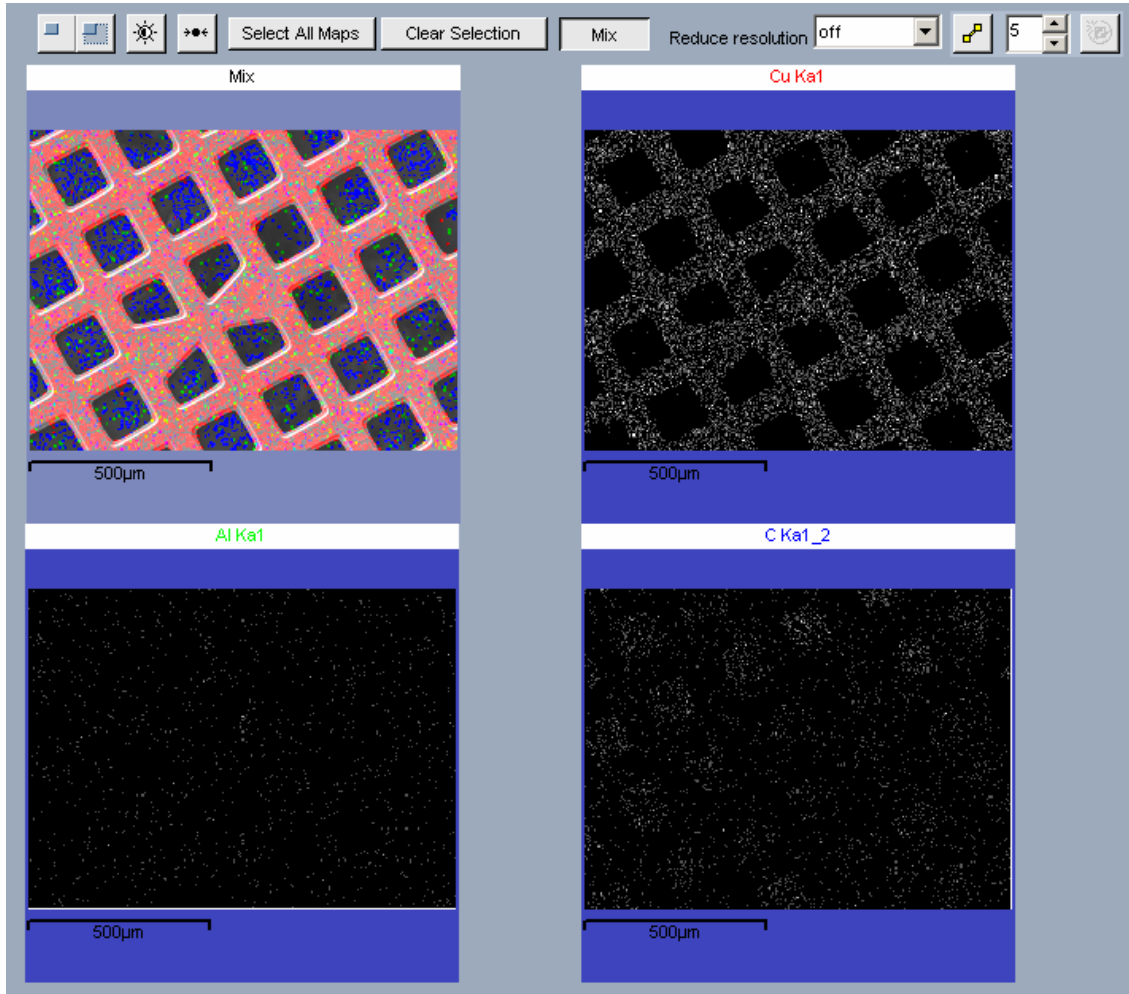
Look at your ‘deadtime’. It should be around 30%. When you are finished collecting your spectra, move down to ‘Confirm Elements’ on the flowchart. You may ‘get rid of’ any elements that you believe were identified in error, or select ones that weren’t identified by the computer that you believe are there. Now move down to ‘Quantitative Analysis’ on the flowchart to see your results.

### **Element Mapping and Linescans**

If you are working under ‘SmartMap’, move down the flowchart to ‘Analysis’. Here there are several tools you may utilize. When collecting maps, and linescans, you may need to use a larger spotsize.

When you are finished collecting your spectra, move down to ‘Confirm Elements’ on the flowchart. You may ‘get rid of’ any elements that you believe were identified in error, or select ones that weren’t identified by the computer that you believe are there.

Now move down to ‘Quantitative Analysis’ on the flowchart to see your results. The spectra for the entire map is labeled “Sum Spectrum”.



### Selecting a New Site of Interest

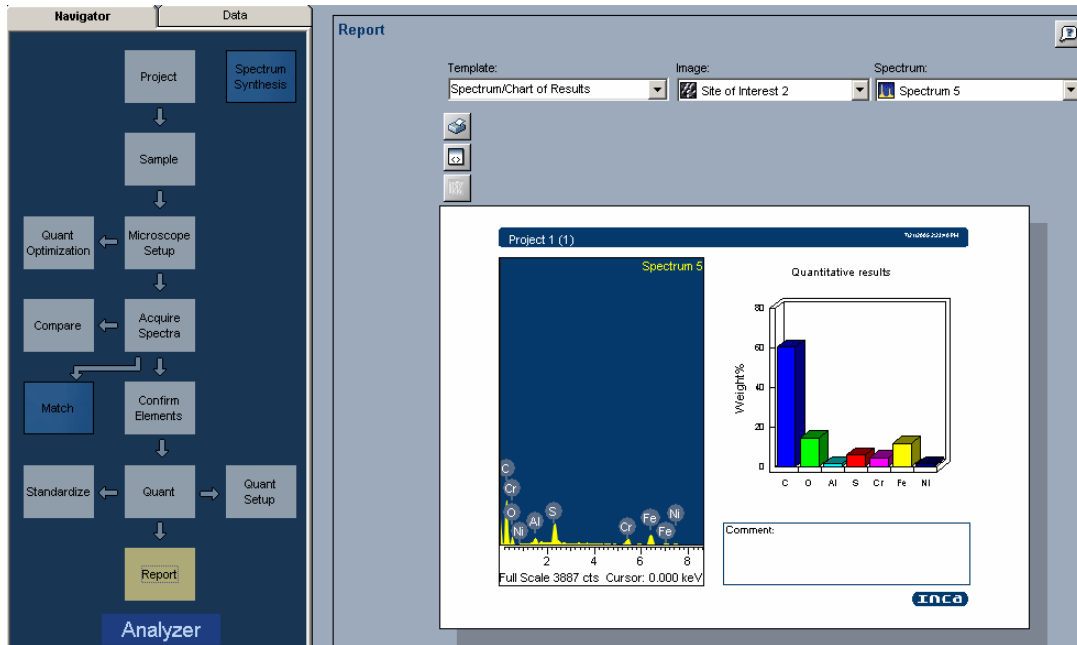
Once you are done acquiring data at your selected Site of Interest, go back to the SEM side, and change your settings back to where you can get an image. Move around your sample until you find a new site to analyze. Once you’ve found a new spot, transfer it to the X-Ray computer as described about in step 4.

### 11. When done collecting data, save your data and exit INCA software.

At the end of each project, you may want to create a report for your record. The INCA system provides several templates for you to use. You can also create your own template to meet your needs.

If there is not further usage of the detector, always retract the detector back to resting location. At this point, the detector will be protected to a maximum from any

potential damage (physically by users' false operations, e.g. moving the stage along Z-direction too fast and hit on the detector).



12. Turn off the beam and wait for 30 min so the filament is cooling down to room temp.
13. Remove specimen from chamber, close the door before pumping.
14. Log off the LEO user interface.
15. Remember to fill the log book.