Dickinson College Department of Geology

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

Equipment: OXFORD ENERGY-DISPERSIVE SYSTEM (EDS) OPERATING ON JEOL JSM-5900

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Running X-Ray (EDS) Analysis on the SEM: Basic Instructions

1. Check to make sure the water cooler is running at a temperature of 65 – 70°F. If it is not, contact Jeff Roth.
2. Check to see that the SEM is on and running. If it is not, contact Jeff Roth.
3. Check dewar connected to detector column to make sure it has liquid nitrogen in it. If it does not, contact Jeff Roth.
4. Check to see if the INCA computer is running (green light on front of case on). If not, contact Jeff Roth.
5. Turn on the SEM and X-Ray computers (and the printers if you wish).
6. Run JSM-5900 software and INCA software.
7. Insert your specimen and standards.
8. Set accelerating voltage to desired operating conditions (usually 15kV).
9. Turn on the filament.
10. Work on obtaining a sharp image using stage position (focus), stigmators, image brightness and contrast and final aperture alignment.
11. Saturate the filament.
   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).
12. Optimize the calibration on the Cobalt standard (Quantitative Optimization).
13. Return to your sample and begin collecting data (remember not to change working distance or spotsizes when collecting data, and always focus using stage Z-drive).
14. When done collecting data, exit INCA software.
15. Remove specimen from chamber, and return specimens to chamber before re-evacuation.
17. Turn off imaging and X-Ray computers.
Running X-Ray (EDS) Analysis on the SEM: Detailed Instructions

1. Starting up the Machine

Check to see that the water chiller is running at a temperature of 65-70 degrees. The chiller is the humming box next to the SEM—it circulates water to cool the diffusion pump. If it is not running, it has probably been shut down over a break. See instructions for system start-up after extended shutdown.

2. Check to see that the SEM is on and running

The SEM will most likely already be on (either the EVAC or VENT light on the front panel will be lit up). If it is not, either the system has been shut down over a break, or a power outage has knocked it off. Turn it on by turning the key on the front panel all the way to the right and then releasing it.

3. Check to see that there is liquid nitrogen in the detector dewar.

If the dewar is empty, you will need to run the cool-down procedure. See General Maintenance directions.

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

If not, turn it on using the rocker switch located on the back on the machine at the very bottom).
5. **Boot up the left and right computers.**

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 x-ray analyzer is on the right - this is computer #2. The red number on the gray box on the right-side table (below the printers) always indicates which computer the monitor and keyboard are working with. It is possible during start-up or shutdown, that the keyboard and mouse will be directed toward the wrong computer; to fix this, simply hit Ctrl-Alt-1, to switch the keyboard and mouse to computer #1 (the left computer), or Ctrl-Alt-2, to switch the keyboard and mouse to computer #2 (the right computer).

6. **Run JSM-5900 and INCA software**

Click on the JSM-5900 icon on the SEM computer desktop if it is not already running. This will bring you to the software interface for the SEM. Then start up INCA on the X-Ray computer, if it is not already running.
7. Loading Your Sample

The first thing you will want to do is to load your sample. Do this by clicking on the Stage button in the upper-right corner. Then select Initial Position at the bottom of the menu. When the sample stops moving, select OK, and then click on the icon in the upper-right corner. Click on Specimen Exchange, and then on Vent (see figure at left). This will vent the sample chamber, and allow you to open the sample chamber door.

Never reach into the sample chamber without gloves on! Use the sample exchange tool (if possible) to switch your sample with the one that is in the chamber. Put the sample you just removed into the metal desicator. If you are not familiar with the sample exchange tool or how the sample holders fit onto the stage, please be sure to ask for help. The small screw on the bottom of the sample holder should be facing you when you insert it into the sample chamber – this acts as a stop.

Once you have your sample on the stage, and before you close the sample chamber door, you should move the stage so that your sample is located directly under the pole piece, which is the place where the electron beam leaves the column. Use the joystick to move the sample stage in the X-Y direction (X is right-left; Y is backward-forward). Hit the 'X-Y' button (it will turn yellow) and the joy stick will move the stage in the 'X-Y' directions. You may also use the stage menu by hitting the blue Stage button in the upper-right corner of the screen.

The 'Z' direction is the vertical position of the stage. For a 10 mm working distance, and using the thin section holders, the 'Z' position should be about 8.0 mm. If you are using
other sample holders, the appropriate 'Z' position may vary significantly. Adjust the 'Z' position of the stage using the joystick and/or the 'Stage' menu.

Be sure that the 'R' (stage rotation) and 'T' (stage tilt) positions are at approximately zero (they often are off by only a few tenths of a degree - this is ok).

Once your sample is secure on the stage and positioned approximately where you want it, close the door, latch it, and click the Evac button on the computer. This will begin the sample chamber evacuation process. Notice that the EVAC button on the front of the SEM column table (left of the computers) is now flashing. After about 5-6 minutes it will stop flashing. At that point, the sample chamber is under vacuum, and you are ready to begin viewing your sample.

8. Set Your Operating Conditions

Accelerating voltage: 15 kV
Working Distance: 10 mm.
Signal: SEI (Secondary Electron Image)
Spot Size: 35.
Magnification: Click on 'View' (upper left) to set the system at the lowest magnification possible at 10mm working distance (this will be 90-100X).

Consult the literature to determine the specific appropriate operating conditions that may be required for the types of samples that you are working with and the kind of imaging that you are doing.

9. Turn on the Filament

Click on [H T] to start bombarding your sample with electrons. At this time, you should enter your information and the time-on into the SEM logbook.

10. Work on obtaining a sharp image using stage position (focus), stigmators, image brightness and contrast, and final aperature alignment.

If you are lucky, an image of your sample will already have appeared on the screen. More likely, you will have to adjust the focus, contrast, and brightness to get an image. Do this by adjusting the contrast and brightness up or down until you reach the crossover between a blank dark screen and a blank bright screen. Start by clicking on 'Contrast' and dragging the mouse upward (if you are trying to brighten the screen) while you hold the mouse button. A left-click is a fine adjustment, and a right-click is a coarse adjustment. After doing this once for 'Contrast', do it again for 'Brightness'. After several coarse adjustments (right-click-and-drag) to both 'Contrast' and 'Brightness', you will find the place where the image goes from blank and dark to blank and bright. Use the fine adjustments to get a fuzzy gray image that shows some contrast (i.e., is not totally flat gray).
If your image is not in focus, this is OK – DON’T FOCUS THE IMAGE USING THE ‘FOCUS’ KNOB. Doing so will change your working distance (WD), which MUST remain at 10mm if you wish to do X-Ray work. Instead, you must 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! If you engage the Z drive using coarse adjustments, you might inadvertently ram your sample into the pole piece. Therefore, you may want to turn on the infrared camera attached to the side of the sample chamber so that you can pay attention to how close your sample is from the pole piece. To make fine adjustments to the Z position, do the following:

Zoom in to increasingly higher magnifications and fine tune the Z position of your sample until you have a sharp image at a magnification significantly higher than what you will be using. Then, when you back off on your magnification, your image will be
very sharp. When you are done with this, turn off the infra-red camera, as it will interfere with your image in later steps.

**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.

### 11. Saturating the Filament

Once you have a reasonable image, you should take a minute to saturate the tungsten filament that produces the electrons that are bombarding your sample. It is important to operate the filament at saturation to ensure a stable beam current.

It may help to darken the room for this procedure.

Go to the button and select . You adjust the filament current using the first scroll bar labeled **Filament Heating**. You can make very coarse adjustments by dragging the sliding button left-and-right, but DON'T DO THIS because the rapid changes in filament current will shorten the life of your filament. You can make finer adjustments by clicking inside the scrollbar, between sliding button and the arrows, but DON'T DO THIS EITHER, again to prolong the life of your filament. To make fine adjustments, click on the 'right' and 'left' arrows themselves. The left arrow will slowly lower the filament current and the right arrow will slowly increase it.

Click the 'left' arrow several times and you will notice that your image begins to darken. Click the 'left' arrow several times more so that the image darkens significantly. Now switch to the 'right' arrow and increase the current. You will see the image get significantly brighter at first, but after several clicks on the right arrow, the increase in brightness will be much less noticeable. **After several clicks** on the right arrow with no increase in image brightness, switch back to the 'left' arrow and turn the filament down ONE CLICK AT A TIME until you see the image darken. At the point where the image first noticeably darkens, the filament is properly saturated.

After saturating the filament, it is a good idea to let the filament “warm-up” for at least 15 minutes or so – the longer the filament is on, the more stable the beam will become.

**Adjusting the Filament Tilts:** After changing filaments, and only occasion otherwise, it may be necessary to adjust the physical tilt of the filament in the gun assembly. The tilts are adjusted in the 'X' and 'Y' directions using the scroll bars immediately beneath the Filament Heating scroll bar under the Gun Alignment menu (see 'Saturating the Filament' above). To adjust the tilts, click on the sliding button and shift it slowly left or right, and
observe the brightness of your image. The idea is to adjust the 'X' and 'Y' tilts to maximize the image brightness (i.e., maximize the signal).

**Can't Get a Sharp Image?** Check the following in order:
- Go through the 'Final Focus' and 'Stigmation' procedures (see above)
- Align the final aperture (see separate instructions under 'General Maintenance')
- Repeat the 'Final Focus' and 'Stigmation' procedures (see above)

If all else fails, call the JEOL service guys in Landover, MD (570-490-2777).

**12. Setting up for Quantitative Analysis – Quant 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 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.**

Click on on the SEM side, select 'File', select 'cobalt', and then ‘Goto’ (if you were positioned over your sample, you may wish to write down the x, y and z coordinates of your position so that you can come back to this spot easily). Once you are positioned 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).

**13. Finding a location for analysis and Data Acquisition**

Back off to the lowest magnification you can, and return your signal to SEI. If you wrote down the x, y and z coordinates of your sample, enter those now by clicking on ‘Stage’ on the SEM side, clicking on one of the values listed, and entering your values into the boxes. When you click ‘OK’, this should take you right back to where you were over your sample. Once you’ve found a spot you would like to analyze, you are ready to send your image over to the X-Ray computer. Depending on your sample type, you may wish to view your sample in Backscatter (BEIW-compo). In this 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 (SEI). If you wish to view in Backscatter, set the following conditions on the SEM:

Signal: BEIW (compo)
Spotsize: ~ 45

You will have to adjust your contrast and brightness to get a good image. If the image goes white, and you can’t darken it, the infra-red camera may still be on. Turn it off.

Once you have an image you would like to analyze, move over to INCA on the X-Ray 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 is 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 spotsize 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 we 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. Typical conditions are:

- Accelerating Voltage: 15kV
- Working Distance: 10mm
- Spotsize: 60
- Signal: BEIW (compo)

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 we 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. If you were imaging in backscatter, these would typically be:

- Signal: BEIW (compo)
- Spotsize: 45mm

If you were imaging in Secondary Emission, these would typically be:

- Signal: SEI
- Spotsize: 35

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.

**14. Finishing Up**
Once you’ve collected all the data you want, you can shut down INCA. Make sure you’ve saved your project, and then exit the software. You may continue to do imaging on the SEM side if you wish.

15. Removing your Specimen

When you are done, remove your specimen by clicking on the Stage button in the upper-right corner. Then select Initial Position at the bottom of the menu. When the sample stops moving, select OK, and then click on the icon in the upper-right corner. Click on Specimen Exchange, and then on Vent (see figure at left). This will vent the sample chamber, and allow you to open the sample chamber door.

Remove the sample holder with the sample exchange tool or a gloved hand. Remove your specimen from the sample holder, and then return the standards to the chamber before re-evacuation.

16. Exiting the SEM Software

After you have returned the standards and evacuated the chamber, select ‘Menu’, and then ‘Exit Microscope’. This will turn off the high-tension to the filament, and close the software.

17. Turn off the SEM and X-Ray computers