Geoscience777 Lab 4 February 27, 2019 Name_____

Introduction to X-ray analysis: Energy Dispersive Spectrometry (EDS) and AZtec

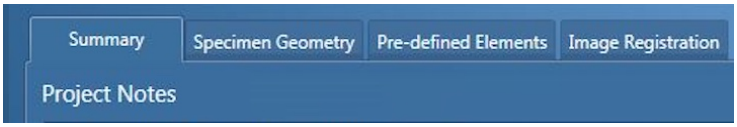
Purpose: The purpose of this lab is to introduce you to some basic concepts of X-ray analysis, via an introduction to EDS. These concepts include the generation of multiple lines for one element, overvoltage, X-rays as both characteristic peaks and continuum, deadtime, EDS spectral resolution, and EDS spectral artifacts.

1. SEM setup – The instructor has already inserted the mystery sample. Make sure the stage is set to the 10mm/"Analysis" height. Set kV to 20 and turn HV on. Set initial Probe Current to 60. Get an image, focus.

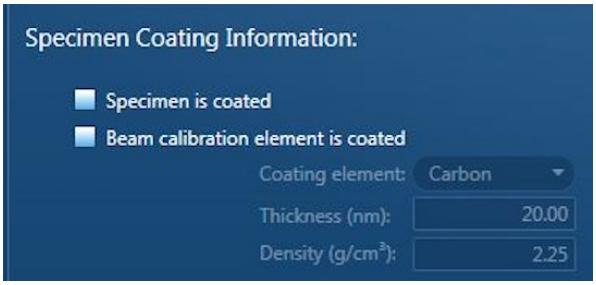
2. EDS Program: The program is "AZtec" on the right monitor. If is typically not running and is launched by double clicking on the Aztec icon (See right)

When Aztec launches, a "Welcome to Aztec" Window will appear, choose New Project, a window will appear that lets you name your project, but first find the correct folder on the computer. Hit the Browse button and find the EDS drive (NOT EBSD one), then data, then Geosciences, then open the G777 class folder, then your lab section, and hit "Select folder". Then create a new Project name appropriate to the work, e.g. Feb 22 lab. Then OK.

In upper left corner, select appropriate icon: here, we want **EDS-SEM**. If it says EBSD, you need to check some HITACHI settings[1].
Then select a specific option: Point & ID. This changes then other options for you.
Note: Users "work" from left to right in these windows, selecting options, mainly.

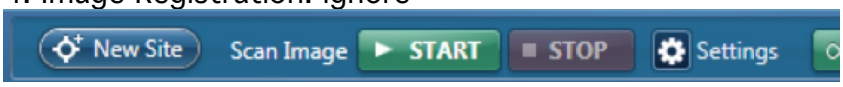


Describe Specimen button:
1. Summary: can ignore the project notes. But check carefully bottom Specimen Coating:
1a. Carbon Coating: If specimen is coated, e.g. carbon, you have option (recommended normally for non-carbonates) of checking the box "Specimen is coated". Then type the thickness, nominally 20 nm, density default.

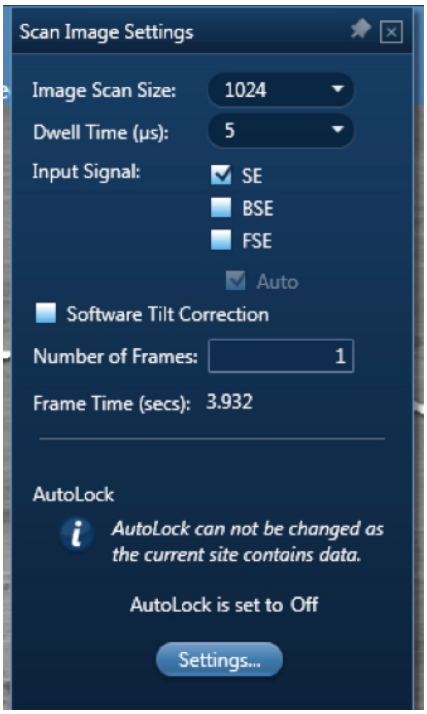


[1] Hitachi Top Menu: Operate→Image Adjustment, uncheck DynFocus and TiltComp. Raster rotation should be turned off.

2. Specimen Geometry: Be sure pretilted holder **not** checked (it will be many times if last user was doing EBSD)
3. **Pre-defined elements**. Check "Clear All" (if not, can create confusion later)
Then (for today's class teaching purposes!) **uncheck Perform AutoID During Acquistion**. We are doing this the first time, for this particular exercise, but in the future you will always want it on—but remember it is never 100% correct!
4. Image Registration: ignore

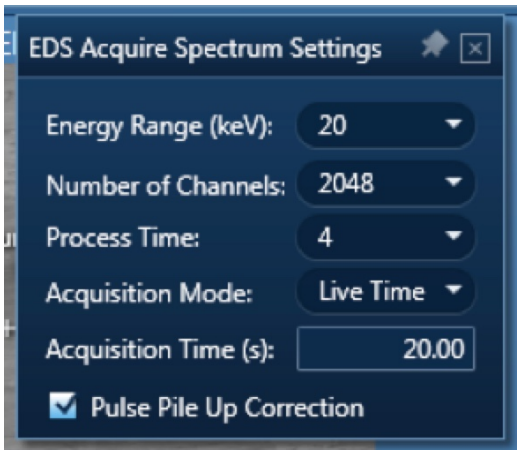


Click the Scan Image button (to right of Describe Specimen)
Assume image is focused on the SEM screen. To bring it over to Aztec, you will be automatically at "New Site" (for first time).
Next, check Settings on same line to right:
Settings:
Image Scan Size: 1024 is normal
Dwell time: how fast it scans. We typically use a fast, i.e. 5 microsec dwell.
Input signal: SE, or BSE, or both.
Autolock: do not touch. (leave it off)
Infinity symbol next to Settings: click this for scan to run continuously. Can leave on while focusing the SEM if you want/need to. Image loops until you stop it.,



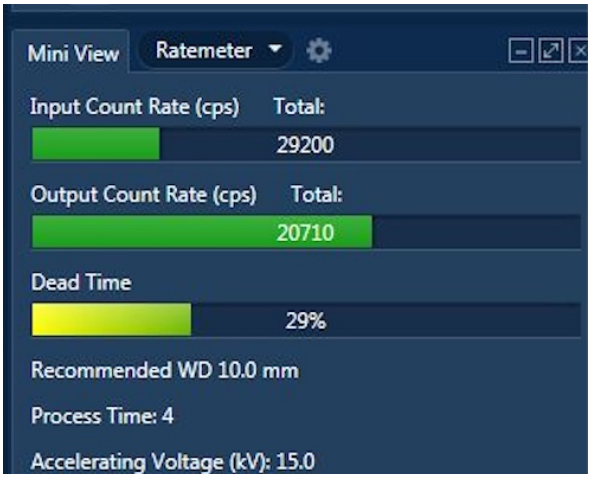
Assuming you have image on SEM, click the Green Start button to get an image. Wait a few seconds for it to appear.

Acquire Spectra button
First, click ***settings:***
Energy range: choices are 10 and 20. Typically use 20 if using 15 or 20 kV. But today, for teaching purposes, select 40
Number of channels: normally 2048; today set to 4096.
Process time: 4 (4 microsec time constant) (for x-ray mapping use 2 or 1; for critical peak separation/ID use 6 (17 microsec)
Acquisition Mode: choices Counts, Live Time, or Auto. Use Live Time for default
Acquisition Time: This is "LIVE" counting time, NOT clock time! Set today to 60 seconds, as we need to make some points. Can be more or less, depending upon your needs. 20 seconds is a good "normal" time.
Pulse Pile Up Correction: Turn off for now, but ultimately for real work, turn it on!



Settings completed, now move to far left of screen to the top green outlined toolbar with symbols. Here you select whether you want X-rays from a spot, or a region. Top cross is spot; below options: box, circle, freeform—these are pinned to the pixel you click on the image. For today, select the 3rd from top, the <u>circle</u> tool.

Bottom Right Side of screen: MiniView: To right is a dropdown menu. Make sure Ratemeter is selected: under it are important values
Input Count Rate (cps) Output Count Rate (cps) Dead Time (%)
These are ACTIVE all the time the beam is on anything and generating X-rays!



→ What is the current Dead Time?___% What is the Probe Current?___ Probe Curr___
If it is not in the 30-40% range, adjust the probe current so that the Dead Time is around 35%. What is the Probe Current now? ____

$$DT(\%) = [(ICR-OCR)/ICR] \times 100\%$$

3. <u>Deadtime calculation</u>: As the live numbers are constantly changing, you will do a deadtime calculation using the "frozen" numbers above and the DT equation. Do you get the "29%" the software is showing? ____

4. Acquire Spectra: The sample is homogeneous, so you can select any region.
→Assign one student to be time keeper, to record REAL (clock) time.
Using the circle tool, draw a circle about a centimeter in diameter. Do not "let go" until the time keeper is ready. Count down 3-2-1-go. Time keeper start counting.
Notice at top right side Under Site #, Electron Image #, there is a green bar counting down to completion of counting (the number of *live time* seconds you set). When it turns blue, time keeper stops and records the seconds. _____ seconds
→ Go to the Windows START menu and click "Snipping Tool" app. This freezes the data.

DT (deadtime) = _____% ; Input Count rate =_____Output count rate = _____

Also, record the Time Constant ("Process Time") _____ microsec

Do the math: DT = [(Clock time – Live time)/Clock time] x 100% = ____% Is that it?

Do a right click on the spectrum, select X axis, then Adjust, range 0 -20 keV. We can also use the mouse scroll wheel to expand/contract the spectrum.

There are 3 major peaks (one around 1 keV, 2 around 8-9 keV) and 3-4 smaller peaks. Can say immediately how many elements are present in the sample? Yes or No? ___

Why or why not?_____

Next we want to manually determine the peaks. Click the Confirm Elements Button at the top, which will bring up a new toolbar on the left. Click the *i* (info) button; this tool can then be positioned on the top of a peak to measure both keV and counts.

Determine the peak position (=energy) of each of these peaks by expanding the scale (mouse wheel) and using the "info" tool. Also read the counts; fill in the columns below.

Low energy Peak _____keV peak counts_____ ->?? Element_____ Line _____

Mid energy Peak _____ keV peak counts_____ ->?? Element_____ Line _____

High energy Peak _____ keV peak counts_____ ->?? Element_____ Line_____

Using all the tools (except Aztec) at your disposal (EDS slide rule, x-ray tables from Goldstein handed out in class, small orange X-ray book), determine what element/s are producing the 3 main x-ray lines. Fill in the 2 right columns above.

What is the full width at half maximum (FWHM) for the middle peak? _____ eV

The FWHM for "Mn Ka" is a figure of merit for an EDS detector. Ours is "129 eV".

6. Now rerun for 15 keV and then 10 keV. Turn OFF the HV first, then change, then turn ON. Adjust probe current to get ~35% DT. Acquire a new image, point and shoot, and record the counts for the La and the Ka peaks. We are going to ignore the Kb—furthest to the right). Label the New sites in the Data Tree with correct kV used.

		15 keV	10 keV
element ____	La	pk counts _____	pk counts _____
element ____	K	pk counts _____	pk counts _____

Calculate La/Ka count ratios: 10 keV _____ 15 keV=_____ 20 keV=_____

(The Cu La/Ka ratio at some commonly used accelerating voltage [E_0] can be an important number to monitor regularly, for indications that there is no degradation of the EDS system, particularly the low energy end (e.g. oil or ice buildup on detector window).

What happens to the La/Ka ratio with increasing accelerating voltage?_____

Let's try to understand why this trend occurs.

First, realize that we are looking at a ratio: Numerator = low energy peak; Denominator = high energy peak.

Second, in order to produce x-rays you should have your accelerating voltage at least ____ times the particular edge energy – this is known as the OVERVOLTAGE. An overvoltage of 1.1 would not yield many x-rays, an overvoltage of 2-3 is "optimal", while an overvoltage of 10-20 is not going to produce many additional x-rays (the cross section is decreasing).

Third, there is another factor that works AGAINST detecting x-rays....ABSORPTION. And low energy x-rays in many cases suffer high absorption, and the deeper the electrons penetrate, the greater the amount of absorption. So at 20 keV, Cu La x-rays will have maybe twice? the distance to travel to exit the sample (Monte Carlo simulation will give you the answer), and be absorbed that many times more.

Look up the mass absorption coefficient (MAC) for Cu Ka by Cu, Table 14.4 p 747 in Goldstein et al 1992. The x-ray is "emitter" and the material it is absorbed by is the absorber (here Cu, i.e. "self-absorption") It is _____.

Now look up the MAC for Cu La by Cu, Table 14.4, p.752. It is _____.

7. Overvoltage is the ratio of the accelerating voltage over the minimum excitation (or absorption edge) energy.

Calculate the overvoltages (U) for each line of the element used above (divide the E0 by the critical excitation energy, found in Goldstein et al (1992) Tables 14.6 and 14.7.

K edge energy_____ L edge energy_____

	Ka overvoltage	La overvoltage
10 keV		
15 keV		
20 keV		

In your final writeup, you will discuss these factors that contribute to the Cu La/Ka vs keV trend.

8. Continuum: Go back to the 15 keV data. Blow up the x-axis of the spectrum between 15 and 20 keV. Extrapolate a straight line thru the x-axis. Where does it intersect? ___ Why?

(this is called the **Duane-Hunt limit** and is a very useful check on an instrument that you WILL find yourself using some day, to verify that the SEM or microprobe or sample is acting properly)

9. Spectral Artifacts:
Now look past the 15 keV "cutoff". Do you see any peaks? Where? ____ and ____ keV. Why?

This are called _____

Now look around 1.8-1.9 keV. Anything there? What is it?_____

Depending upon the exact lab sample, you may see a peak at 1.49 keV. The instructor will explain its presence. It is possibly _____

Look at 0.525 keV; any ideas what it might be?_____

How about 0. 27 keV? _____

And how about the negative energy X-rays below zero keV! Are we slipping into a black hole? This is the "strobe zero peak".

10. A peak into the world of "Auto ID": Verify you are in the Confirm Elements Menu, and the bottom periodic table is empty (Clear all). Click AutoID. What unexplained element comes up? ___ What peak does it seem to be strongly "attached" to? ____

This points out one of the main possible problems with EDS as a black box: unwary users can be led to trust it 100%.

11. OK, let's let Aztec behave "normally", turning back on some features. (We are at 15 kV, right?)
Select the Scan Image chevron at the top. Click New Site, collect a new image (Start).
Select the Acquire Spectra chevron. Set energy range to 20, leave 4096 channels, live time, 60 sec acquisition time. Acquire a spectrum.
How is it different from the one you acquired before? (What artifacts are missing?)
_____

In Confirm Elements, Auto ID. In Settings click "No pulse pile up correction" and a green outline shows the "raw" spectrum like we created before. Notice where the sum peaks were. Also blow up the top of the Cu Ka peak; why is the yellow color higher than the green?
_____

What about the Cu La peak? _____

12. Two Gotchas:
a. Turn on the infrared chamber light while the HV is on and the beam is on a sample. What happens to the EDS system? (look at deadtime)

b. You are at a working distance of 10 mm (right?). Now move to a working distance of 5 mm. Acquire a spectrum? What do you see? (look at deadtime)
Why?

13. Write up a one page summary that includes the main points presented in this lab

Revisions:
9/29/03, 10/4/04, 10/3/05, 10/13/05, 10/2/06, 9/24/07, 9/28/09, 2/3/13
Aztec: 2/21/18 2/24/18