

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

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. Physical layout - Pointing out the features on an old EDS detector, and some features of the Hitachi Thermo EDS, including the travel rail (set at 5 cm, **which you never should touch**). Liquid nitrogen, rules for dry dewar if you come in and hear the alarm. Also a brief discussion of LN safety issues (freezing skin; suffocation)

2. EDS Program: The program is “NORAN System Six” on the right monitor. If it is not already running (check bottom task bar), double click. The Project Explorer opens. You need to be in the E: drive, and in the EDS Users-2010-NEW folder. Navigate to the 777-2013 folder, and then to your group’s folder, only click the expand (+) button, do not double click on folders. Now create a **project** folder for today’s work. Left click the upper **green** folder under “File”, this creates a new “Project” (**NOT** a brown folder). Rename the folder with today’s date. Click OK in upper right corner.

3. Sample: Assumption: The SEM is setup (High Vac), $E_0=20$ kV, probe current=50, WD=10 mm. Image=BSE. The instructor will have moved an unidentified material under the beam. And turned off the autoID function for this first part (Right click white area where spectrum displayed, Spectrum Properties, Peak Page, Labels=None). Adjust image brightness and contrast on the Hitachi monitor.

4. Set Program Parameters: On one of the upper menu bars, find the blue-green icon that looks like a cactus, then go one to left, yellow icon for “Edit Acquisition Properties”. Click to open it. This is an important panel. EDS should be selected. Here is where you set
a) **Live Time Limit**: the maximum LIVE time (depends upon what you are doing; here, set to 60 seconds); this is NOT clock time.

b) **Pulse Processor: Time Constant:** for now, have it set for “Auto” (more on this later)

5. Spectrum Acquisition: Make sure “Point and Shoot” are selected on left menu bar. Acquire an image now in the NORAN program by clicking the icon to the left of the black arrow (looks like a strat column). The sample is homogeneous, so you will click anywhere. Assign one student to be time keeper, and record the time. Click to start the EDS acquisition. Notice in the bottom, there is a ‘count down’ green bar that tells how many more ‘clock time’ seconds are approximately left. When it clicks off, record the clock time. Record, the numbers on the bottom right bar:

DT (deadtime) = _____% and SR (stored count rate) = _____ (it changes, any number OK) Also, from Detector Status Window, record the Time Constant (TC) _____ microsec

How many seconds on the clock did it take to acquire “60 seconds of live EDS time”?
_____ Seconds

We will come back to this.

Three major peaks should be visible . (If necessary, click the Black X icon in upper menu bar to see the whole spectrum. Can say immediately how many elements are present in the sample? Yes or No?

Why or why not? _____

Determine the peak position (=energy) of each of these peaks by both expanding each peak (position red marker over it and the expanding with the black <> areas, and fine tuning the red cursor position; read the x-ray peak position at top. Also read the counts; fill in the first two 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 the NORAN) at your disposal (EDS slide rule, x-ray tables from Goldstein handed out in class, small orange X-ray book), determine what elements are producing the 3 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 “138 eV”.

6. Now rerun for 15 keV and 10 keV. Turn OFF the HV first, then change, then turn ON. Acquire a new image, point and shoot, and record the results. We are going to ignore the highest energy peak (highest keV—furthest to the right). Also record Clock Time and Time Constant TC

15 keV				10 keV				
DT ___%	SR _____	Clock_____sec	TC____		DT___%	SR _____	Clock____sec	TC_____
low energy element _____	line _____	pk counts _____			pk counts _____			
mid energy element _____	line _____	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. Dead time, TC, counts and spectral artifacts: Above, we used the nice “auto” Time Constant setting, which many people use without understanding what it is doing, and when it is useful, and when it can possibly get you into trouble. Here we turn off the auto TC and manually set the TC, which you may need to do at some point in your research.

On Cu metal, at 20 keV. Live time of 60 seconds. We will run 5 acquisitions, at 3 TC settings, and 2 Probe Current settings, to record Real time, Clock Time, Dead Time %, Store Rate, Cu Ka peak counts, and any spectral artifacts.

Probe Current	Clock Time (seconds)	Time Constant microsec	Dead Time %	Store Rate (cts)	Cu Ka Peak Counts	Spectral Artifacts?
“40”		64				
“40”		30				
“60”		30				
“40”		10				
“60”		10				

Comment:

- A. For “quick and dirty” element/phase ID, auto TC usually is fine if you keep the DT below 50%.
- B. If you have a real unknown and need to determine the elements present, you may want to turn off auto TC, set it to 30 or 40, and drop the Probe Current, and count for several hundred seconds.
- C. If you need to X-ray map at very short TC, run a test spectrum NOT in “point and shoot” mode but in Spectrum mode at a low enough Mag so that several phases are blanketed by the rectangular beam scan. Look for spectral artifacts and proceed accordingly .

10. EDS artifacts:

There are several EDS artifacts that appear under assorted circumstances; generally when you are using shorter pulse processing time constants, and higher deadtimes, as the the detector is more “stressed” plus higher counts yield greater statistics so something that may be infrequent will have more probability of showing up. Also, if you happen to be searching for a small amount of an element and blow up the scale, you see all kinds of things.

Have you observed any of these? ____If so, Under what conditions (time constants, count rates, deadtimes)

Sum Peaks

Si-escape Peaks

Si-dead layer Peaks

Apparent X-rays beyond Duane Hunt limit

11. 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?

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

Revisions:

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