

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 SX50's EDS, including the aperture switch (limiting the amount of X-rays that can hit the detector). Liquid nitrogen, rules for dry dewar.

2. Spectrum Acquisition: Setup EDS software for 60 sec acquisitions (Acquire→ Attributes). Set the pulse process time set to 51 microsec (under Acquire->Setup pulse processor), set accelerating voltage (E0) to a “normal” value of **20 kV** on the SX51. Adjust beam current to produce ~35-45% deadtime, which will probably be around 2500 cps. The EDS aperture (on SX51 column) should be in middle position (see appendix). Record your beam current (on SX51).
Current = _____ nA at _____ kV with _____ deadtime.

The instructor will position an unidentified sample under beam if not done so already.

Three major peaks should be visible (disregard any “zero” peak that is at the extreme far left; this is electric noise which should be ignored (should be below 0.5 keV). Make sure you count for at least 60 seconds (need good statistics!)

Can say immediately how many elements are present in the sample? ____
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 cursor position; read the x-ray peak position at top.

The software should also aid by telling you the peak identity (9th icon from left: page with **small ?** -NOT the big black ?, which is Help file.

Low energy Peak _____ keV	peak counts _____	->?? Element____	Line ____
Mid energy Peak _____ keV	peak counts _____	->?? Element____	Line ____
High energy Peak _____ keV	peak counts _____	->?? Element____	Line ____

Now repeat for 15 keV and 10 keV (trying to maintain Deadtime around same value you used before, 30-40% by changing beam current—DO NOT change the column aperture) and record the results. We are going to ignore the highest energy (highest keV—furthest to the right) peak. Record Faraday cup current for each measurement.

	15 keV curr=___nA	10 keV curr=___nA
	deadtime _____	deadtime _____
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 [E₀] can be an important number to monitor regularly, for indications that there is no degradation of the EDS system, particularly the low energy end.

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, while an overvoltage varying from 10 to 20 is not going to produce many additional x-rays (the cross section is slowly 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 14.3 p 747 in Goldstein et al (1992) or on the CD for the new edition. 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 _____.

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 kV		
15 kV		
20 kV		

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

3. Continuum: Set the E0 back to 15 kV. 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 useful check on an instrument that you might find yourself using some day, to verify that it is operating properly)

4. Dead time, pulse processing time, and FWHM: On Cu metal, at 15 keV. TC of 51.2 microsec [“Real time”=clock time; “Live time”=time detector is accessible to new counts] You will vary the current, to produce higher and higher count rates, that progressively yield higher and higher dead times. Go from low to high dead time. Collect data in the format of the following table (Display→Attributes for “Real Time” and Process→Peak Stats for other info). Set the live time to 20 sec (in Acquire). Read off Dead Time and “thruput” (cps) off the bottom of the screen display.

Beam current	Real Time	Dead Time (%)	Total count Rate (cps)	Cu Ka peak counts	FWHM
4 nA					
20 nA					
40 nA					
80 nA					

Note that the x-ray intensity (“Rate” in cps, counts/second) displayed is the TOTAL x-rays being detected (continuum plus characteristic).

With the conditions still set to 80 nA, change the pulse processing time constant to 1.6 usec (Acquire => Setup Pulse Processor). Now repeat the last measurement and record your results below.

Beam current	Real Time	Dead Time (%)	Total count Rate (cps)	Cu Ka peak counts	FWHM
80 nA					

What is different about the 80 nA results for 1.6 microsec vs 51.2 microsec?

When would you use longer, e.g. 51 usec? _____

When would you use 1.6 usec? _____

6. 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, which particular element(s)? Under what conditions (time constants, count rates, deadtimes)

Sum Peaks _____

Si-escape Peaks _____

Si-dead layer Peaks _____

7. Write a summary that includes the main points presented in this lab.

Include a paragraph that includes a plot of the data in part 2 (keV on x axis, La/Ka on y axis) and discusses the factors responsible for the observed trend. Also explain why monitoring the Cu La/Ka ratio can be of value.

Revisions:

9/29/03

10/4/04

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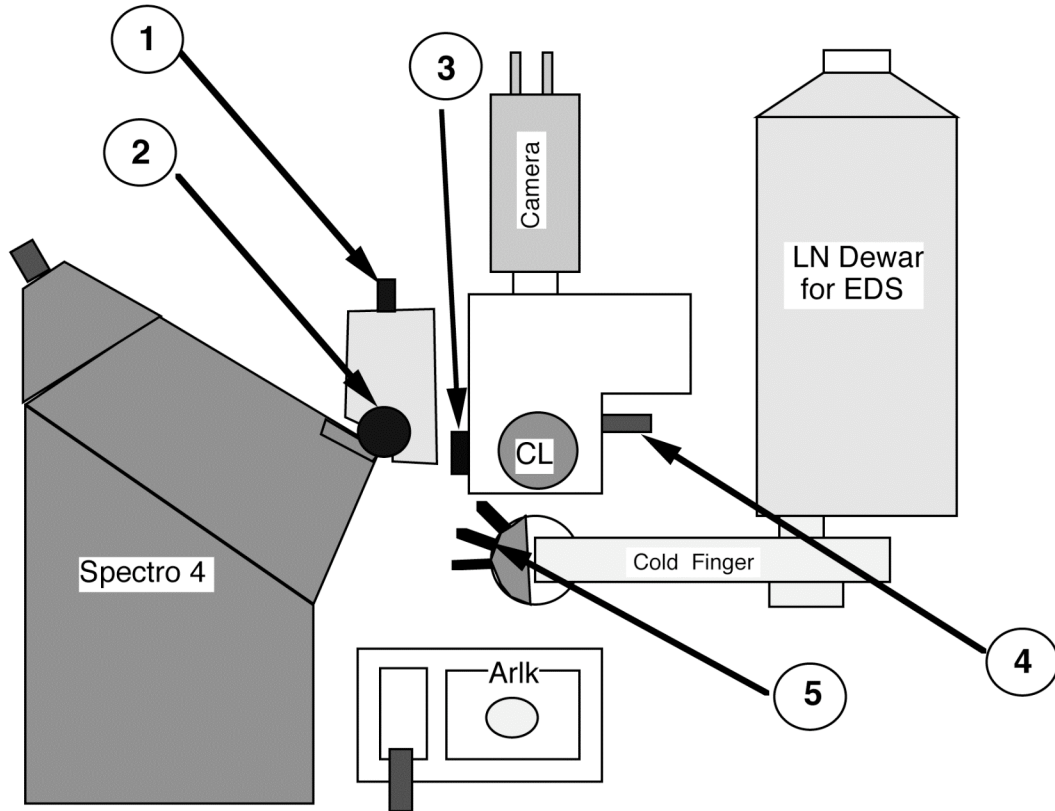
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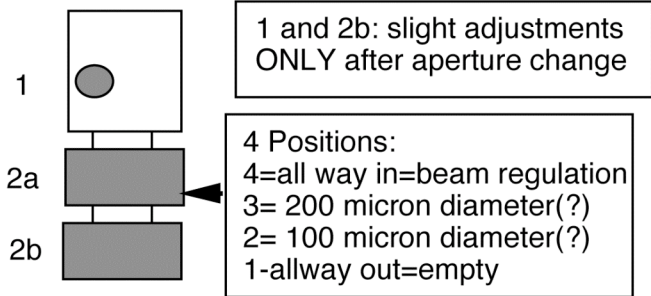
Front View of SX51 #485

Location of important knobs that might rarely require turning. Consult with Lab Director or experienced user before touching!



- 1 and 2: Aperture change knob (2a) plus 2 aperture adjustment knobs (1 and 2b)
- 3: Mirror flip knob
- 4: Reflected - transmitted light lever
- 5: EDS aperture lever (3 positions)

1 and 2, view from above



4: Lever in=transmitted light' lever out=reflected light

5: Aperture to limit xray counts collected by EDS detector
 Bottom position (~9:00) for high current operation (~20 na);
 Top position (~11:00) for low current operation (i.e., SEM work in pA range)

3: White arrow up= normal camera operation
 white arrow at 1:00 = CL operation