

Guidelines for Qualitative X-ray Analysis

The process of identifying the correct elemental composition of a sample, qualitative analysis, is the most important aspect of EDS. Without this accurate elemental identification quantification, standardless or full standards, is not worth much. The process of qualitative analysis is straight forward but detector artifacts, very small peaks, and peak overlaps can cause some frustrating challenges. Hopefully, this guide can shed some light on the process.

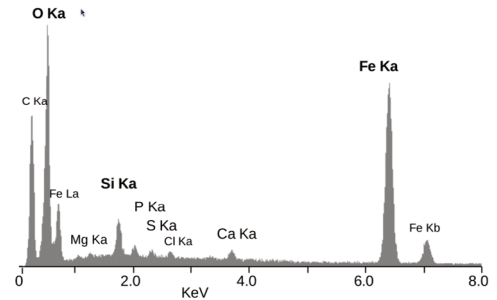
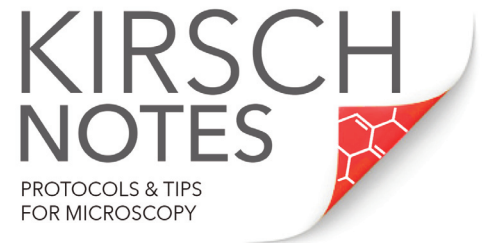
Directions

1. Look at the overall spectrum and take note of:
 - a. Peak locations on the horizontal Energy axis
 - b. Relative peak intensities on the vertical Counts axis
 - c. Shapes - Gaussian?
2. Start at the high-energy end (right) side of the spectrum since the alpha and beta peaks are separated.
3. Looking at the spectrum determine the first definite Alpha peak.
 - a. Always the highest peak of an element
 - b. There is no peak to the left (lower energy) of it, within 2 keV, which is more intense.
4. Place energy cursor in the center of the accepted alpha peak and read its energy.
5. Looking at the Energy Chart start with the Ka column and try to match the number within ± 20 -30eV. If not, go to the La and then the Ma lines for a match.

NOTE: If nothing is matching, stop and check the calibration of the system!

6. If a match is made place the energy cursor over the energy where the beta peak should be and check the β energy of that element to see if it matches as well. If the peak family ratio matches then write this down as a possible fit.
7. Using that same alpha energy check the La and Ma columns to make sure there is not another possible element that fits.
8. If an L or M alpha energy matches check the beta peak and its intensity ratio as above. If there is no other match and the beta fits, then that is the only possible fit and you have identification.
9. Once an element has been identified be sure to label it and check to see if its associated K, L or M lines are present. If so, label them as well.
10. Continue on with this system until all peaks are accounted for.
11. As you identify elements, check for possible overlaps by:
 - a. Checking peak family ratios:

K	$\alpha - 10$	$\beta - 2$		
L	$\alpha - 10$	$\beta 1 - 6-5$	$\beta 2 - 2$	$\lambda - 1$
M	$\alpha - 10$	$\beta - 5$		
 - b. Gaussian distribution
12. Other unidentifiable peaks, "rogue peaks", may be:
 - a. Escape peaks - 1.74 keV less than large intensity peak
 - b. Sum peak - 2X the energy of large intensity peak.
 - c. If there a significant L line, check the energy chart for the L_i line. (Lower energy than the α)



Elemental Energy dispersive X-Ray microanalyses of the mineral crust of Rimicaris exoculata, from a mineral particle $\sim 2 \mu\text{m}$ diameter. The peaks are labelled with the line of the corresponding element.

Obtained from Iron oxide deposits associated with the ectosymbiotic bacteria in the hydrothermal vent shrimp Rimicaris exoculata L. Corbari, M.-A. Cambon-Bonavita, G. J. Long, F. Grandjean, M. Zbinden, F. Gaill, and P. Compere, Biogeosciences, 5, 1295-1310, 2008

Image Credit: https://commons.wikimedia.org/wiki/File:EDS_-_Rimicaris_exoculata.png

Troubleshooting Tips

1. If nothing seems to be matching stop and check the calibration of the system!
2. Is it an elemental peak or is it noise?

Small peaks need to be statistically significant.

99% sure = $3\sqrt{\text{background intensity} + \text{background}}$

98% sure = $2\sqrt{\text{background} + \text{background}}$

Example:

peak intensity = 132 cts.

background intensity = 100 cts

$\sqrt{100} = 10$

$3(\sqrt{100}) = 30 + 100$ (background intensity)

= 130 cts – It is a peak & must be identified.
3. Use both the vertical (intensity) and horizontal (energy) expand and /or contract to get better views of the peaks/spectrum.
4. Be careful about labeling a peak as an element, which is rare or very unusual such as Noble gases, or man-made elements.
5. Take into account any coating, which may have taken place (such as C, Au, Pd, Pt).
6. For complicated spectra, print out a copy of the spectrum or the confusing area and write in the energy lines and intensities and work from that. Take your time and eliminate any possibilities.