

Can I Trust My Quantitative EDS Data?

NOTES ON STANDARDLESS QUANTITATIVE EDS IN SEM

Scanning electron microscopes (SEM) coupled with an energy dispersive X-ray detector (EDS) are used extensively to provide insight into a sample's chemical makeup. This SEM-EDS technique can provide information on the elements present, their relative concentrations and spatial distribution over very small volumes (micron and some instances nanometer scale).

EDS, in general, is considered a semi quantitative elemental analysis technique. We are often asked how reliable are the quantitative results using SEM-EDS. This is a pretty broad question as it is dependent on a variety of factors including the sample matrix and morphology in addition to instrument considerations.

So... what can be detected and how much? Modern systems are capable of detecting elements from Be to U. Detection limits are typically considered to be $\geq 1\%$ for low atomic number elements (F to Be) and $\geq 0.1\%$ (1000 ppm) for higher atomic number elements.

One of the most common techniques used for quantitative EDS analyses is a method often described as Standardless quantitative EDS. With this method, the user does not use physical standards but instead uses a ratio of peak intensities to determine the relative abundance of the elements detected. The peak intensities are corrected for background and matrix effects and the results are then normalized to 100% based on the elements detected. This normalization can hide errors in the analysis results. With that said, if all criteria are met, one can expect around $\pm 2\%$ to $\pm 5\%$ relative for major components. However, this error can increase significantly for particles or rough surfaces.

So, what are the criteria to consider when performing EDS quantitative analysis? Several assumptions are made with this technique regardless of whether the quantitative method is 'Standardless' or with physical standards. First, the sample is polished and flat. It is also homogeneous and infinitely thick relative to the beam interaction volume. If the sample is not homogeneous with respect to the beam interaction volume, the results may vary based on the contribution of neighboring components (Figure 1).

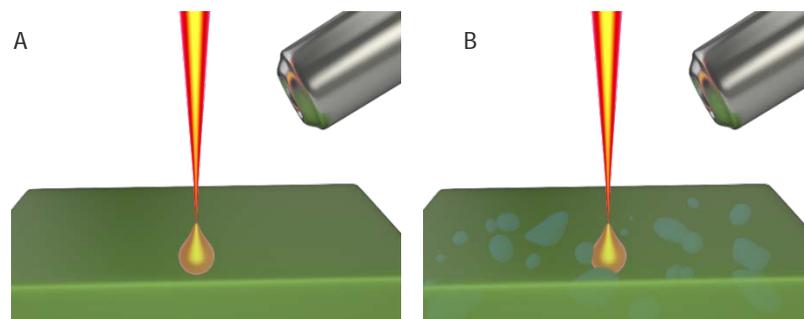
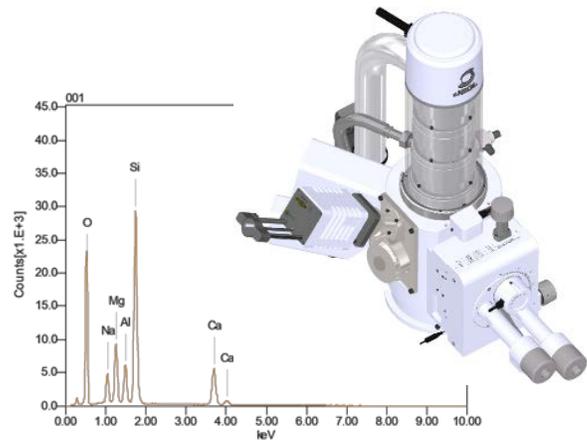


Figure 1: A: Homogeneous sample within the beam scattering volume. B: Heterogeneous sample, a particle within the scattering volume will contribute to EDS quantitative results.

On the other hand, it may be particles or inclusions that you are trying to identify and quantify. By placing the beam on the particle, you may get a contribution from the surrounding matrix if the scattering volume is larger than the particle itself. For non-uniform materials it is good practice to collect spectra from several different areas and average the results.

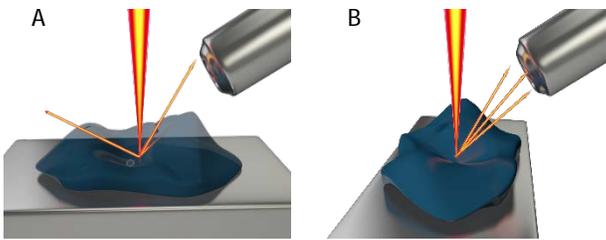


Figure 2: A: X-rays are blocked from reaching the detector by sample topography. B: Rotating the stage presents the region of interest in sight of the EDS detector allowing the X-rays to be detected.

Only those X-rays that are within line of sight to the EDS detector are collected. If the sample has significant topography, the X-rays can be blocked entirely and not reach the detector. Or, in some instances, low energy X-rays may be absorbed by the sample matrix more than higher energy X-rays contributing to error in the quantitative results.

When dealing with a topographic sample, it is important to understand the sample position with respect to the EDS detector position. It is often possible to position the region of interest so that it has direct line of sight to the detector (Figure 2).

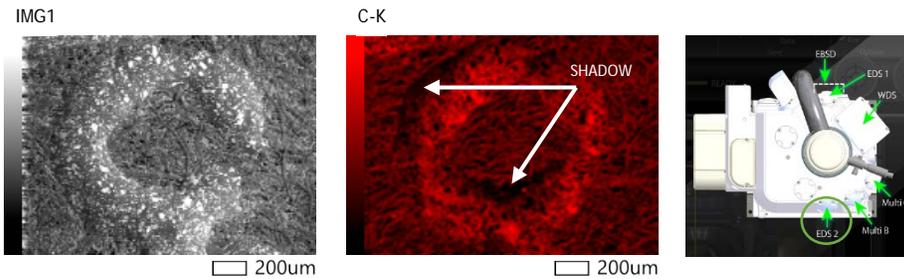


Figure 3: Example of C X-ray Intensity Map of Ink on Paper taken from EDS detector position 2. The ink is raised on surface of paper and the result is a shadow where C X-rays are blocked by the topography of the sample from reaching the detector.

Finally, the accelerating voltage must be high enough for efficient excitation of the X-ray lines for the elements present in the sample and there should be sufficient probe current to generate a statistically significant X-ray count rate. What is typical is to choose an accelerating voltage that is 1.5 to 2 times higher in energy than the energy of the X-Ray lines that is of interest. For an unknown sample, 15kV to 20kV is recommended. Deviation from any of these conditions will contribute to errors in the quantitative analysis results.

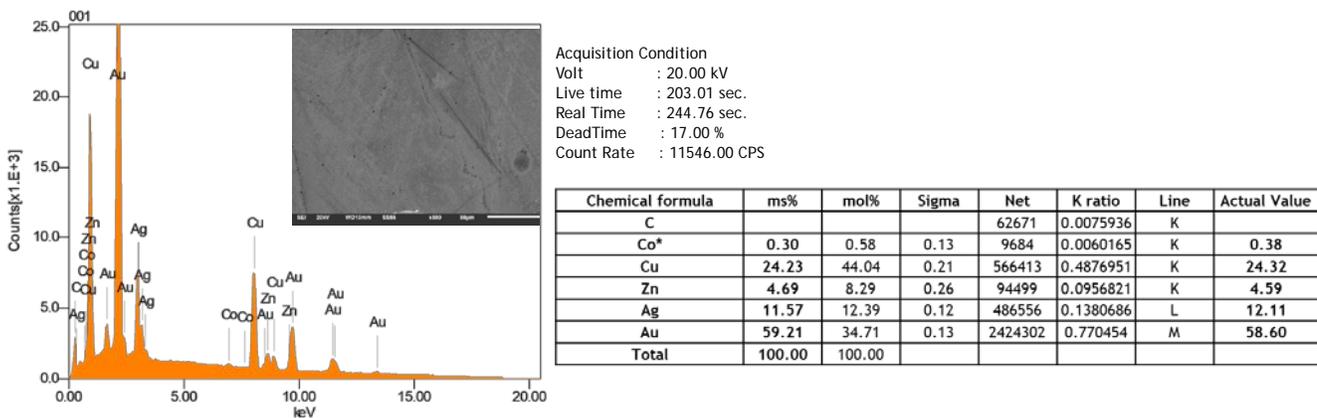


Figure 4: Example: EDS Standardless Quantitative Results - Gold Alloy