

Standard-less Quantification and Standard Customized Coefficients

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While the roots of EDS quantification trace back to pure standards-based methods, the vast majority of EDS quantification today is using standard-less methods. This is to a large extent driven by convenience since use of standard-less quantification does not require expensive standards, beam current measurements or a significant amount of time setting up an analysis of a given sample type. The drawback is that standard-less models are optimized for general conditions and do not have the same flexibility as standards-based methods, where precision and accuracy can be greatly increased by optimizing conditions for one specific experimental setup.

One way of bridging part of the gap between standards and standard-less methods is to apply Standard Customized Coefficients (SCC). Unlike quantification with standards, where the goal is to minimize model contributions to the quantification results, the goal of SCC factors is to optimize the model to a given experimental setup and set of conditions without forgoing the ease of use and speed of standard-less quantification. In general, the number of counts for element in channel can be described as [1]:

$$N_i^{ch} = c_i \varepsilon_i i t (\Omega/4\pi) \omega_i q_i S_i^{ch} (RAF)_i^{ch},$$

where c is the concentration of the element i , ε is the detector efficiency at the relevant energy, i is the beam current, t is the collection time, Ω is the solid angle of detection, ω is the fluorescence yield, q is the weight of the line, and S is the primary ionizations generated in a given shell, while RAF are the correction factors accounting for backscatter variations, sample self-absorption and fluorescence. Most standard-less results are normalized to 100%, but by measuring a reference spectrum (typically single element standard) it is possible to determine the term of the equation and calculate non-normalized results. By measuring either compound or single element standards using the non-normalized results, it is now possible to determine any deviations between model output and certified values as well as compensate for the deviations by applying a SCC factor/scaling factor. Figure 1 shows spectra from a selection of single element standards along with the calculated SCC numbers.

One of the advantages of using SCC factors is the ability to calibrate the system with a limited number of standards and to extrapolate to a wider set of experimental conditions. Figure 2 shows a selection of Al₂O₃ spectra and quantification results where SiO₂ and pure Al samples were used to measure the SCC factors for O and Al respectively. The quant results are non-normalized and in excellent agreement with the nominal values showing there is very good agreement between model and experiment for this sample. The SCC factors can be used to optimize results under numerous conditions such as low energy analysis where ionization cross sections and mass absorption coefficients are poorly characterized, but also where systematic errors influence the results such as an offset in detector elevation angle or a variation in detector window characteristics relative to nominal values.

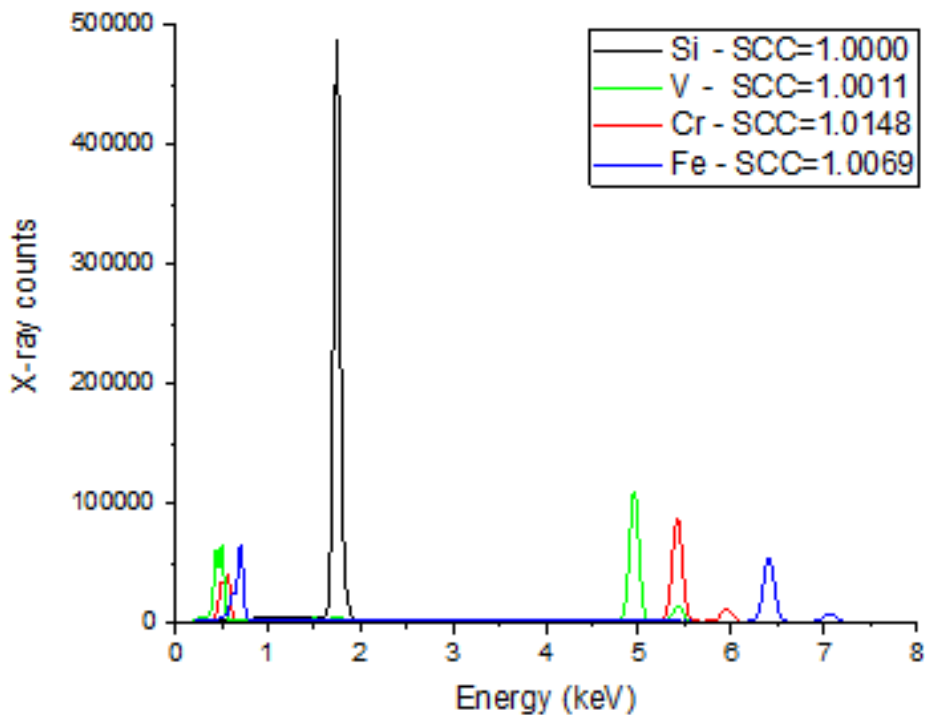


Figure 1. Spectra and corresponding SCC numbers for Si (reference), V, Cr, and Fe.

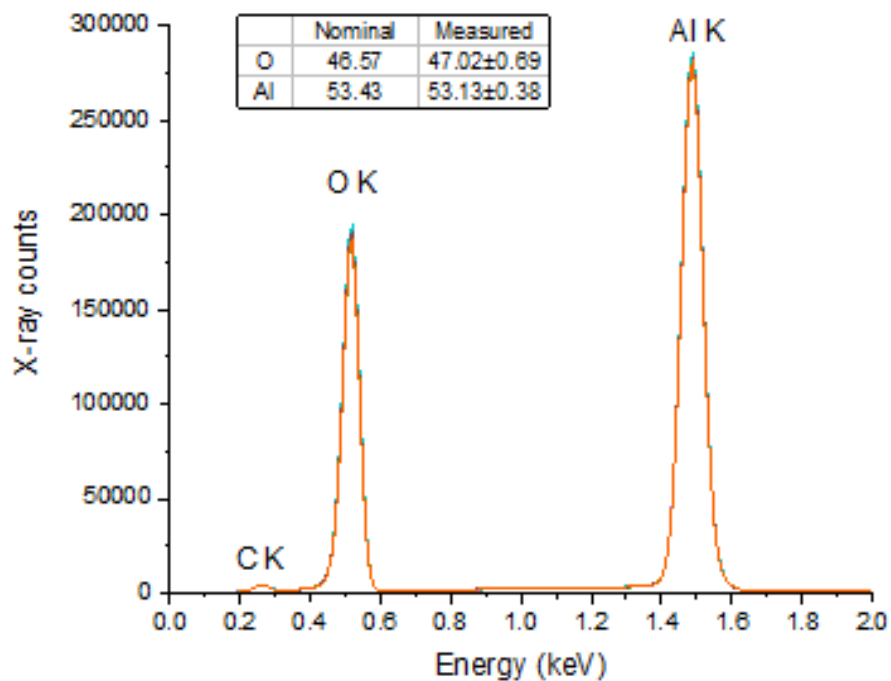


Figure 2. 5 Overlaid Al₂O₃ spectra with the corresponding nominal and calculated composition.

References

[1] J. Trincavelli et al, Spectrochimica Acta, Part B (101), pp. 76-85, 2014.