

Standards-Based Quantitative EDS Mapping.

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In many microanalytical software packages, standardless EDS (energy-dispersive spectroscopy) “semi-quantitative” analysis is typically done with a single mouse click and yields results that appear to be accurate to 0.001 wt% and always with analytical totals of exactly 100%. Although semi-quantitative analysis has proven to be getting progressively more accurate [e.g., 1], it is still unable to account for many factors that can greatly affect the accuracy of an analysis such as surface roughness, detector window contamination, and secondary fluorescence of a neighboring phase [2]. Recent discussion [e.g., 1,2,3] has highlighted the importance of standards-based EDS quantitative analysis. However, such discussion has not carried over to EDS mapping. EDS spectral imaging, in which a full EDS spectrum is acquired and stored at each pixel, allows for the peak deconvolution algorithms and matrix corrections involved with EDS quantitative analysis to be applied to EDS mapping. Here, I investigate the importance of the use of standards in EDS quantitative mapping.

EDS data were collected using a Thermo Scientific™ UltraDry™ SDD EDS detector mounted on a JEOL 7001F FE-SEM. EDS spectral images were processed using the Thermo Scientific NORAN System 7 X-ray microanalysis system. Quantitative mapping was done on an SPI Cu metal standard and lunar meteorite NWA 2727. Standards for EDS quantitative analysis were SPI metal and natural and synthetic mineral standards. Beam current was measured at the beginning of each mapping acquisition and was compared to an additional measurement after the mapping acquisition to ensure beam stability.

For mapping the Cu metal standard, eleven beam-rastered, 64×48 pixel EDS spectral images were acquired at 20 kV until the average X-ray counts/pixel (i.e., counts per spectrum) were 500; 750; 1,250; 2,500; 5,000; 10,000; 15,000; 20,000; 30,000; 40,000; and 50,000. Although mapping a single element standard produces uninteresting map images, it provides an excellent opportunity to investigate the reliance of EDS quantitative analysis on a sufficient number of counts without confusion added by topographic effects. By extracting so-called “quant maps” using a Cu standard, 3072 quantitative analyses were calculated from each of the eleven spectral images (Fig. 1). Because the spectral images were all acquired on a Cu standard, all quantitative analyses should yield 100 wt% Cu. However, accurate results were not achieved until ~50,000 counts/pixel were acquired. Using semi-quantitative analysis, every pixel of each of the eleven spectral images would have yielded exactly 100 wt% Cu, which is the correct answer in this example, but would yield incorrect results in samples with more than one element in spectral images with insufficient counts.

For mapping an area of NWA 2727, a 50,000 counts/pixel, 64×48 pixel (41.9×31.4 μm) EDS spectral image was acquired at 15 kV. Elemental quantitative maps were extracted. Additionally, analytical totals, pyroxene stoichiometry, and olivine stoichiometry maps were extracted (Fig. 2). The analytical total map is perhaps the most beneficial result of standard-based quantitative mapping in that it allows the user to quickly see which pixels yield poor analyses (i.e., high or low analytical totals). For example, in Fig. 1, there is a crack. One would expect that fewer X-rays reach the detector from a pixel representing the crack yielding a low total. However, with standardless analysis, this result is normalized to 100%, giving no indication of a pixel with a poor analysis.

References:

- [1] NWM Ritchie and DE Newbury, *Microsc. Microanal.* **20** (2014) p. 696.
- [2] JH Fournelle, S Kim, and JH Perepezko, *Surf. Interface Anal.* **37** (2005), p. 1012.
- [3] DE Newbury and NWM Ritchie, *Microsc. Microanal.* **18** (2012) p. 1004.

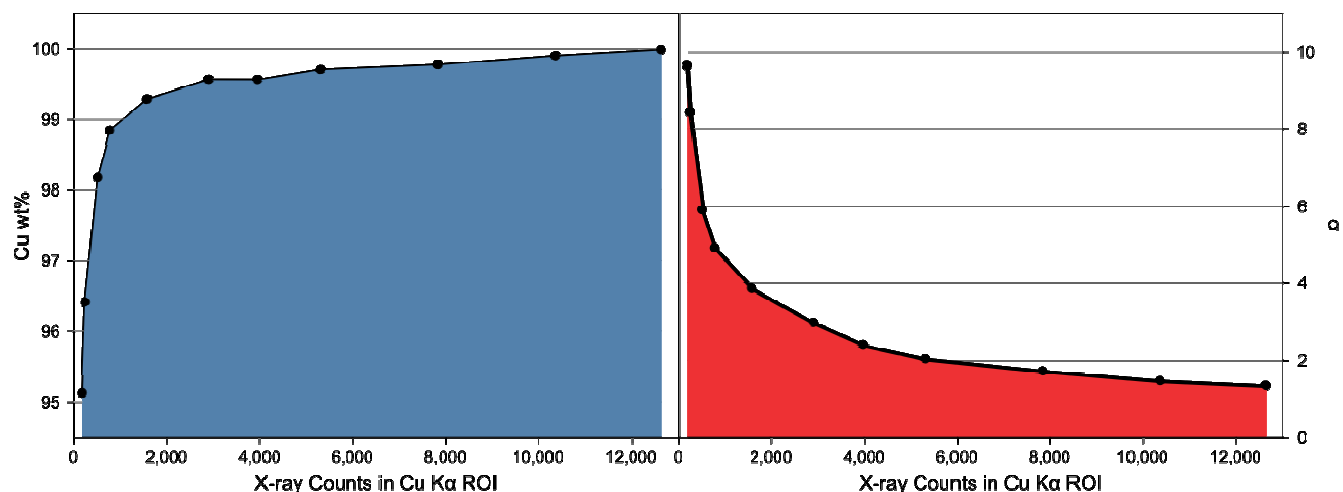


Figure 1. Average wt% Cu and standard deviation thereof as a function of X-ray counts in the Cu K α region of interest. Sample was 100 wt% Cu.

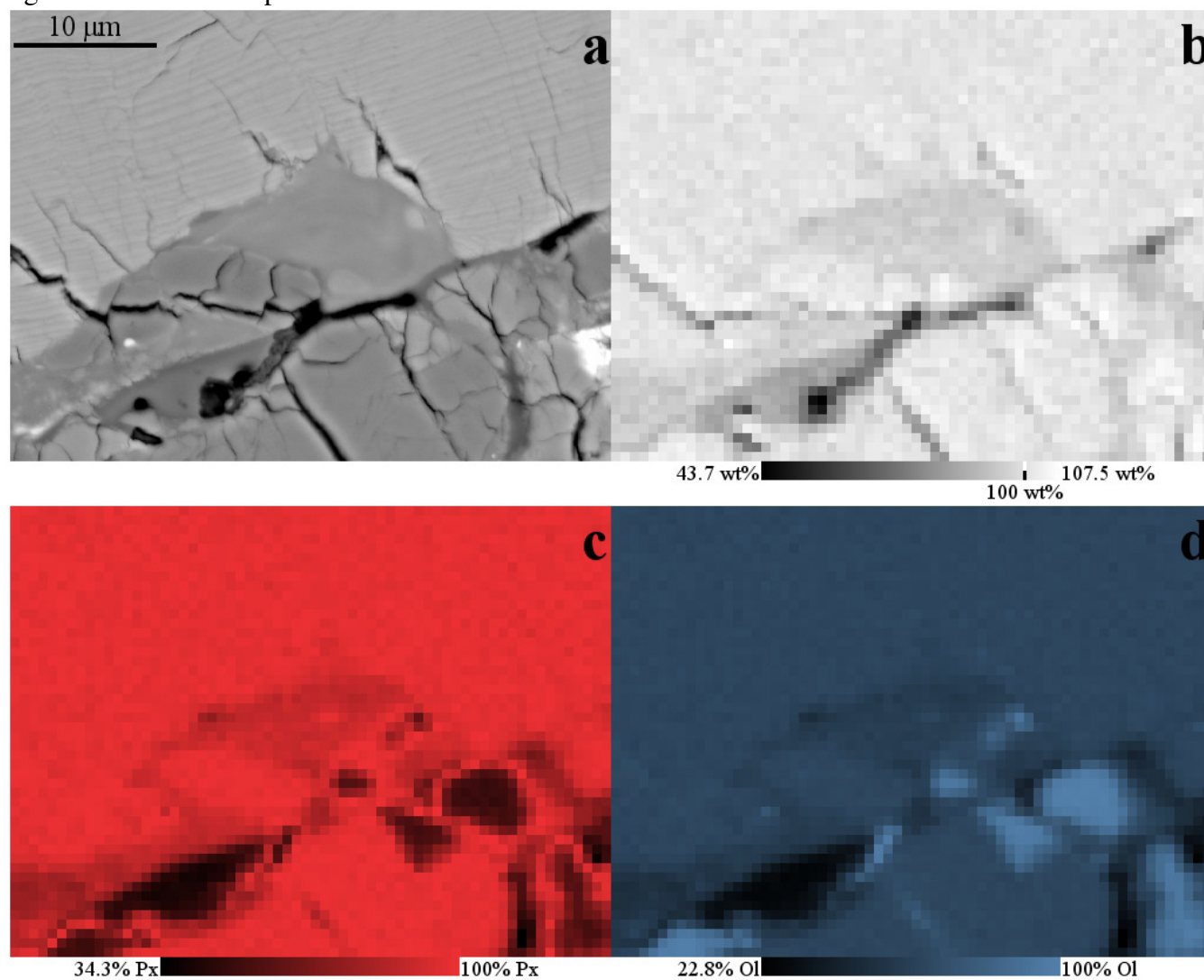


Figure 2. **a.** Backscattered electron image of the mapped area. **b.** Map of analytical totals; low totals are fractures. **c** and **d.** Maps of how well the analysis at each pixel matches pyroxene (**c**; $[\text{Ca,Mg,Fe}]_2\text{Si}_2\text{O}_6$) and olivine (**d**; $[\text{Mg,Fe}]_2\text{SiO}_4$) stoichiometries as calculated from standards-based quantitative analysis.