

Improved EPMA Trace Element Accuracy Using A Matrix Iterated Quantitative Blank Correction

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Modern EPMA instruments equipped with low noise detectors, counting electronics and large area analyzing crystals can now routinely achieve sensitivities for most elements in the 10 to 100 PPM levels. However, because of various sample and instrumental artifacts in the x-ray continuum, absolute accuracy is more often the limiting factor for ultra trace element quantification. These artifacts have various mechanisms, but are usually attributed to sample artifacts (e.g., sample matrix absorption edges), detector artifacts (e.g., Ar or Xe absorption edges) and analyzing crystal artifacts (extended peak tails preventing accurate determination of the true background and “negative peaks” or “holes” in the x-ray continuum). The latter being first described by Self, et al. and recently documented for the Ti α in quartz geo-thermometer. See Fig 1.

These x-ray continuum artifacts can produce systematic errors that are measurable at levels up to 100 PPM or more depending on the particular analytical situation. In order to correct for these inaccuracies, a “blank” correction has been developed that applies a quantitative correction to the measured x-ray intensities during the matrix iteration. Preliminary results from this new matrix iterated trace element blank correction demonstrate that systematic errors can be reduced to single digit PPM levels.

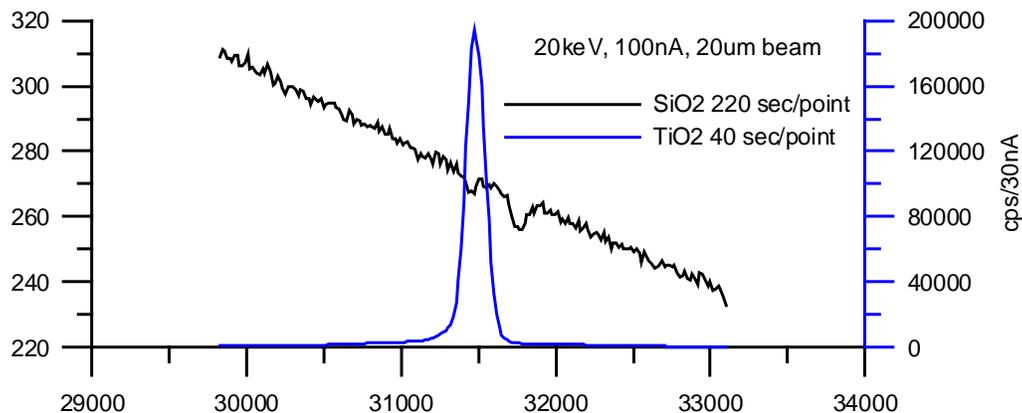


Fig 1. Wavelength dispersive scans on SiO₂ and TiO₂ at the position for Ti K α showing “holes” in the continuum both adjacent to and underneath the analytical peak.

Self, P.G., Norrish, K., Milnes, A.R., Graham, J. & Robinson, B.W. (1990): Holes in the Background in XRS. *X-ray Spectrom.* 19 (2), 59-61

Wark, DA, and Watson, EB, 2006, TitaniQ: A Titanium-in-Quartz geothermometer: Contributions to Mineralogy and Petrology, 152:743-754, doi: 10.1007/s00410-006-0132-308