Analysis of trace elements in zircon at high mass resolving power using forward-geometry secondary ion mass spectrometry

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Trace elements in zircon form the basis for estimates of magmatic pressures, temperatures, and differentiation, providing tools to understand magma evolution throughout Earth history. Recently, empirical relations have led to the proposal that specific trace elements, including Sc-Y-Nb-Ce-Yb-Hf-U, are distinctive of different tectonic settings on the modern Earth [1]. Broader application of these relations has been tempered, in part, by the challenges associated with accurate and reproducible measurement of Sc and Nb; each has a single naturally occurring isotope, and require $m/\Delta m>12000$ and $m/\Delta m>14000$ to resolve interferences with ⁹⁰Zr⁺⁺ and ⁹²ZrH⁺, respectively.

We present analytical methods for maintaining accurate magnetic field calibration for days-to-weeks at a mass resolving power=12500 ($m/\Delta m$, full width at 10% peak height) on the large-radius, forward-geometry IMS-1280. Maintaining accurate positioning on peaks during magnetic field cycling requires ± 15 ppm precision ($\Delta m/m$, full width at 95% peak height) while accounting for drift and perturbations. To achieve this, we monitor count rates at the approximate half-height-width of specific major element and metal-oxide peaks; relative count rates are used to make small (ppm-level) adjustments to the magnetic field settings for these and 26 other interpolated trace element masses after each analysis. Regular analysis of reference materials over >10 days shows the majority of measured trace elements, including Sc and Nb, have relative analytical uncertainties at or below 10%. Heterogeneous and/or low concentration elements show variability comparable to the ranges cited in published literature. Detection limits and precision benefit from (1) an RF-plasma source with higher beam density $(5.5 \text{nA}^{16}\text{O}^{-}\text{primary beam}, \sim 13 \mu\text{m pit diameter}), (2) optimizing}$ secondary ion transmission (~25%), and (3) continuous use of a liquid nitrogen cold-trap. Average, estimated background levels for Nb and Sc at peak centers are $\leq 0.001 \mu g/g$. We have combined this method with custom AutoHotKey protocols which coordinate data recording, calibration reading/writing, and stage movement to enable continuous, automated analysis. These results represent a robust method for quantification of unique trace elements, and a means to evaluate their application throughout Earth history [2,3].

[1] Grimes et al. (2015), Contrib. Min. Pet. 170, 46.

[2] Shimizu et al. (this meeting).

[3] Valley et al. (this meeting).