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Zircon ablation volume measurements using high-resolution optical interferometry, stoichiometric proxies, and x-ray computed tomography: Uncertainties and implications for in-situ (U-Th)/He ages

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(U-Th)/He thermochronometry is seeing a rapid development towards methods with high spatial resolution down to the micron scale. However, these novel in-situ laser-ablation approaches have yet to become routine and remain underutilized as (U-Th)/He dating requires two separate analytical measurements for parent and daughter isotopes via different analytical techniques. This currently necessitates two separate laser-ablation pits and determinations of their exact volume for age dating. The accuracy and precision of these volume measurements are critical, but to date, no systematic studies have been published that quantify the accuracy and uncertainty of volume measurements and their impact on (U-Th)/He ages. We conducted systematic volume measurements of ablation-pits in polished, un-zoned, gem-quality zircon using (1) optical interferometry, (2) total signal intensity of the stoichiometric isotopes ^{29}Si and ^{96}Zr via LA-ICP-MS and (3) high-resolution x-ray computed tomography (CT). Common limitations in optical interferometry are the inability to image steep pit walls and interference from dust or ejecta in the pit. Stochiometric isotope proxies, commonly employed for internal ICP-MS elemental calibration, rely on a fixed elemental concentration and constant ablation characteristics across the grain. Further problems might arise from mass fractionation on the mass spectrometer over large signal intensities and temporal changes in laser energy and ablation characteristics. High-resolution x-ray CT likely yields more accurate volume markers and is used to assess uncertainties in the other two methods. While the reproducibility of volume measurements by optical interferometry was ~3.5%, the total uncertainties were 5-10% but dramatically increased (>>20%) for very small diameter pits (<10-µm). Volume measurements based on stochiometric intensities were surprisingly variable (10%) – a fact that is particularly significant as parent isotope concentrations cannot be measured by interferometry and are commonly determined by internal standardization. The standard error of in-situ He ages of Fish Canyon Tuff zircon confirm the magnitude of our new precision estimates and illustrate the critical need for developing improved volume measurement methods for better He ages.