**Appendix A: Sample preparation and LA-ICP-MS analysis and session details (GeoSep methods only, samples Winkler-1, Franklin-1, SS-2, SS-4A, SS-5A, PR-1-5976m, PR-1-5977.9m, PR-1-5978.7m)**

Mineral grains were isolated and prepared for LA-ICP-MS analysis using standard procedures combined with specific customized procedures described by Donelick *et al.* (2010). Similar descriptions of the methods followed to produce and process zircon U/Pb data have been presented in Bradley *et al.* (2009), Hults *et al.* (2013), and Moore

*et al.* (2015). Zircons (both standards and unknowns) were mounted in epoxy wafers

and ground and polished to expose internal grain surfaces.

Isotopic analyses were performed with a New Wave UP-213 laser ablation

system in conjunction with an Agilent 7700x quadrapole LA-ICP mass spectrometer in

the GeoAnalytical Lab at Washington State University. For all laser analyses, the beam

diameter was 30 μm and the frequency was set at 5 Hz, yielding ablation pits ~12-15 μm deep. He and Ar gas were used to deliver the ablated material into the plasma source. Each analysis of 32 cycles took approximately 30 seconds to complete and consisted of a 6-second integration on peaks with the laser shutter closed (for background measurements) followed by a 24-second integration with the shutter open and the laser ablating zircon material. A 20-second delay occurred between analyses.

The isotopes measured included 202Hg, 204(Hg + Pb), 206Pb, 207Pb, 208Pb, 232Th, 235U, and 238U.

Previous LA-ICP-MS studies of U-Pb zircon dating used the ‘intercept’ method, which assumes that isotopic ratio varies linearly with scan number due solely to linearly varying isotopic fractionation (Chang *et al.* 2006; Gehrels *et al.* 2008). The data modeling approach favored here was the modeling of background-corrected signal intensities for each isotope at each scan. Background intensity for each isotope was calculated using a fitted line (for decreasing background intensity) or using the arithmetic mean (for non-decreasing background intensity) at the global minimum of selected isotopes (206Pb, 232Th, and 238U) for the spot. Background + signal intensity for each isotope at each scan was calculated using the median of fitted (2nd-order polynomial) intensity values for a moving window (7 scans wide here) that includes the

scan. The precision of each background-corrected signal intensity value was calculated

from the precision of background intensity value and the precision of the background +

signal intensity value.

A number of zircon U-Pb age standards were used during analysis for

calibration purposes. These included the 1099 ± 0.6 Ma FC zircon (FC-1 of Paces and

Miller 1993) as the primary age standard. The secondary age standard was the 61.2 ± 0.1 Ma Tardree Rhyolite zircon (Ganerød *et al.* 2011). Third-level age standards included the Fish Canyon Tuff with an age of 28.20 ± 0.1 Ma (Lanphere *et al.* 2001), the Mount Dromedary Syenite with an age of 99.1 ± 0.1 Ma (Renne *et al*. 1998), and the Temora2 diorite with an age of 416.8 ± 0.3 Ma (Black *et al.* 2004). At the beginning of the LA-ICP-MS session, zircon standards (TR and FC1) were analyzed until fractionation was stable and the variance in the measured 206Pb/238U and 207Pb/206Pb ratios was at or near 1 percent. In order to correct for inter-element fractionation during the session, these standards were generally reanalyzed after each 15-25 unknowns. Uranium decay constants and the 238U/235U isotopic ratio reported in Steiger and Yäger (1977) were used.

Uranium decay constants and the 238U/235U isotopic ratio reported in

Steiger and Yäger (1977) were used in this study. 207Pb/235Uc (235Uc = 137.88238U),

206Pb/238U, and 207Pb/206Pb ages were calculated for each data scan and checked for concordance; concordance here was defined as overlap of all three ages at the 1σ level (the use of 2σ level was found to skew the results to include scans with significant common Pb). The background-corrected isotopic sums of each isotope were calculated for all concordant scans. The precision of each isotopic ratio was calculated by using the background and signal errors for both isotopes. The fractionation factor for each data scan, corrected for the effect of accumulated α-damage, was weighted according to the 238U or 232Th signal value for that data scan; an overall weighted mean

fractionation factor for all concordant data scans was used for final age calculation.

If the number of concordant data scans for a spot was greater than zero, then either the

206Pb/238U (for ages <1.5 Ga) or 207Pb/206Pb (for ages >1.5 Ga) age was chosen as the preferred age. If zero concordant data scans were observed, then the analysis was deleted. Common Pb was subtracted out using the Stacey and Kramer (1975) common Pb model for Earth. Ages and common Pb ratio were determined iteratively using a pre-set, session-wide minimum common Pb age value (default for each session was the age of the oldest age standard which was 1099 Ma FC-1.

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