

## DATA REPOSITORY

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### APPENDIX 1: MINERAL SEPARATION AND ANALYTICAL TECHNIQUES USED FOR ZIRCON ANALYSIS AND INTERPRETATION

#### Mineral Separation Procedure

Zircons from sandstone samples were prepared for analysis at the University of Calgary using methods outlined by Matthews and Guest (2016). Sandstone samples (5–10 kg) were first rinsed with water and cleaned of soil and lichen to reduce the risk of surface contamination. Samples were then crushed using a Bico™ Chipmunk crusher and pulverized using a Bico™ disc mill. Pulverization took place over two steps, first using a gap of 800  $\mu\text{m}$  and finally a gap of 350  $\mu\text{m}$  to improve the uniformity of fine-grained material. The pulverized material was then processed using an MD Gemini Goldharvester™ shaking table (water table) to separate the high-density minerals ( $> 3.0 \text{ g/cm}^3$ ; e.g., zircon, apatite, rutile, sulphides) from the low-density minerals ( $< 3.0 \text{ g/cm}^3$ ; e.g., quartz, feldspars, calcite). Methylene iodide (density:  $3.33 \text{ g/cm}^3$ ) density separation was then employed to further concentrate the dense minerals. The remaining dense mineral fraction was then processed using a Frantz™ isodynamic separator over multiple trials up to maximum setting of 1.8 A with a side slope angle of  $5^\circ$ . An aliquot of grains ( $n = 1000\text{--}5000$ ) was then randomly selected from the remaining mineral population, and mounted in round epoxy pucks (diameter: 1"; thickness: 0.5"). Mounts were polished using 5 and 3  $\mu\text{m}$  silicon carbide polyester lapping films, and finished with a 1  $\mu\text{m}$  diamond lapping film. Mounts were cleaned and then loaded into an ablation cell for analysis via laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS).

Zircons from volcanic ash samples were separated at Zirchron Geoscience Services in Tucson, AZ. Because the ash samples were unconsolidated, no crushing was required. Zircons were separated from ash deposits using a Wilfley table, and then the quality of zircon separate was improved using a Frantz™ isodynamic separator (maximum setting: 1.8 A, side slope angle:  $20^\circ$ ). Methylene iodide density separation was then used to further concentrate the dense minerals. Zircons were then hand-picked to avoid inclusions, cores and other fractures that could confound the resulting age data. Zircons were dissolved in Teflon tubes inside Parr bombs and spiked with  $^{205}\text{Pb}$  and  $^{234}\text{U}$ . U and Pb were then separated using anion columns and loaded onto

filaments for measurement via thermal ionization mass spectrometry (TIMS; Saleeby et al., 1987).

### **LA-ICP-MS Isotope Ratio Measurement Procedure**

Zircons were ablated at the University of Calgary using an ASI Resochron™ 193 nm excimer laser ablation system that incorporated a Laurin Technic M-50™ dual volume cell. Laser fluence and repetition rate was set at 1.5 J/cm<sup>2</sup> and 10 Hz respectively for each measurement session. In all cases, a 33 µm beam diameter was used for laser ablation. Isotopic signal intensities were measured using an Agilent 7700 Q-ICP-MS; the instrument was tuned for maximum sensitivity and minimum oxide production using NIST SRM 610 glass prior to each session.

For the high throughput ( $n > 500$ ) measurements, ~800 measurements were conducted in a single ablation sequence (2.75 h). Each sequence included measurement of ~640 unknown zircons and ~160 measurements of five zircon reference materials. Reference materials included Temora-2 (Black et al., 2004), 91500 (Wiedenbeck et al., 1995, 2004), FC-1 (Paces and Miller, 1993), FCT (Kuiper et al., 2008), and TRD (Ganerød et al., 2011). Each measurement consisted of a 5 second ablation period and resulted in a pit ~3.5 µm deep. Twenty-second gas blanks were measured prior to each ablation of the calibration reference material (Temora-2).

For the low throughput ( $n \approx 30$ ) analyses, up to ~110 measurements were conducted in a single ablation sequence. Each sequence included measurement of up to ~70 analyses of unknown zircons and up to ~40 measurements of five zircon reference materials. The total number of analyses incorporated multiple measurements ( $n_{\text{max}} = 5$ ) of individual unknown zircon grains. Reference materials used were the same as those used in the high throughput analyses. Each measurement consisted of a 20 second gas blank, followed by a 20 second period of ablation, and a 4 second delay to purge the previous sample and prepare for the next analysis. The ablation period resulted in a pit ~12 µm deep.

### **TIMS Isotope Ratio Measurement Procedure**

U and Pb isotope measurements were performed in static mode for both U and Pb on a VG Sector multi-collector TIMS instrument at the University of Arizona (Otamendi et al., 2009). Total Pb blank during the course of the analyses was < 0.1 ng Pb. Uncertainties ( $\pm$ ) of radiogenic <sup>207</sup>Pb/<sup>206</sup>Pb ratios were based on agreement between spiked and unspiked aliquots, mass spectrometer statistics, and uncertainty in initial Pb isotopic composition. Common Pb compositions used for nonradiogenic correction were based on blank and initial Pb values, which were approximated using the Stacey and Kramers (1975) Pb evolution model. Mass spectrometric performance was monitored on a regular basis by runs of NIST NBS 981 and U500 zircon standards.

### **Data Reduction Procedure**

Data reduction and age calculation for LA-ICP-MS data was performed at the University of Calgary using the Iolite™ (V2.5) software package (Paton et al., 2010) with the VizualAge data reduction scheme (Petrus and Kamber, 2012). Temora-2 (age: 416.8 ± 0.33 Ma; Black et al., 2004) was used as the primary reference for data reduction. All subsequent data manipulation,

including the incorporation of excess variance for the  $^{206}\text{Pb}/^{238}\text{U}$  and  $^{207}\text{Pb}/^{206}\text{Pb}$  ratios ( $\varepsilon$  and  $\varepsilon'$ ) and propagation of systematic uncertainties ( $\sigma_{\text{sys}}$ ) was performed in Microsoft *Excel* using a custom VBA macro and the Isoplot add-in (Ludwig, 2012). Calculation of uncertainty was aligned with recommended best practices outlined by Horstwood et al. (2016). Calculation of  $\varepsilon$  incorporated the addition of uncertainty associated with replicate measurements of the  $^{206}\text{Pb}/^{238}\text{U}$  ratio of 91500 within a given analytical session (range of  $\varepsilon$  values: 1%–5%). Calculation of  $\varepsilon'$  incorporated a weighted average of measurements of 91500 across 39 analytical sessions; as a result, an  $\varepsilon'$  value of 1.2% was added in quadrature to each  $^{206}\text{Pb}/^{238}\text{U}$  age uncertainty, and an  $\varepsilon'$  value of 0.7% was added in quadrature to each  $^{207}\text{Pb}/^{206}\text{Pb}$  age. Other systematic uncertainties added included uncertainty in the  $^{206}\text{Pb}/^{238}\text{U}$  and  $^{207}\text{Pb}/^{206}\text{Pb}$  ratios of Temora-2 (Black et al., 2004) and uncertainties in the U-Pb decay constants (Jaffey et al., 1971, with modifications from Mattinson, 1987). Data reduction and age calculation for TIMS data was performed at the University of Arizona. The data incorporates an uncertainty of 0.6% based on long-term reproducibility of the  $^{206}\text{Pb}/^{238}\text{U}$  ratio.

## **APPENDIX 2—LA-ICP-MS and TIMS DATA (SUPPORTING DOCUMENTATION)**

### **Information for Probability Density Plot Construction**

Construction of probability density plots for provenance analysis employed dates from the Accepted Dates column of the high throughput (HT) LA-ICP-MS results. These dates are derived from  $^{207}\text{Pb}/^{206}\text{Pb}$  ratios for instances where dates > 1300 Ma, and from  $^{206}\text{Pb}/^{238}\text{U}$  ratios where dates < 1300 Ma (cf. Gehrels et al., 2008). All accepted dates have a probability of concordance > 1% (see column AI in the HT results spreadsheet; cf. Matthews and Guest, 2016); probability of concordance was determined using formulas outlined in Ludwig (1998). Scientists that wish to use these data to construct their own probability density plots should use uncertainty measurements reported in the  $2\sigma_{\text{total}}$  columns.

### **Calculation of Depositional Ages**

Maximum depositional ages (MDAs) were determined using dates from the Accepted Dates column for the low throughput (LT) LA-ICP-MS results only. All of these dates are derived from  $^{206}\text{Pb}/^{238}\text{U}$  ratios, as all dates are < 1300 Ma (cf. Gehrels et al., 2008). All accepted dates have a probability of concordance > 1% (see column AI in the LT results spreadsheet; cf. Matthews and Guest, 2016); probability of concordance was determined using formulas outlined in Ludwig (1998). Ages were calculated using the Weighted Average tool included with the Isoplot add-in for Microsoft *Excel* (Ludwig, 2012). In instances where multiple measurements were made on a single grain, a weighted mean age was calculated for that grain using the dates obtained from each measurement (see Note 1 in low throughput ages spreadsheet). MDA calculations incorporated these ages, as well as dates from grains that could only accommodate one measurement. To replicate the calculation of depositional ages presented in this study, scientists should first use the  $2\sigma_x$  level of uncertainty to determine a weighted mean age, and then add the systematic errors outlined in Appendix 1 in quadrature to the computed uncertainty from the weighted mean age calculation. Depositional ages from TIMS data were determined via calculation of a weighted mean age using  $^{206}\text{Pb}/^{238}\text{U}$  apparent age and uncertainty data.

## REFERENCES CITED

- Black, L.P., et al., 2004, Improved  $^{206}\text{Pb}/^{238}\text{U}$  microprobe geochronology by the monitoring of trace-element-related matrix effects: SHRIMP, ID-TIMS, ELA-ICP-MS and oxygen isotope documentation for a series of zircon standards: *Chemical Geology*, v. 205, p. 115–140, doi:10.1016/j.chemgeo.2004.01.003.
- Ganerød, M., Chew, D.M., Smethurst, M.A., Troll, V.R., Corfu, F., Meade, F., and Prestvik, T., 2011, Geochronology of the Tardree Rhyolite Complex, Northern Ireland: Implications for zircon fission track studies, the North Atlantic Igneous Province and the age of the Fish Canyon sanidine standard: *Chemical Geology*, v. 286, p. 222–228.
- Gehrels, G.E., Valencia, V.A., and Ruiz, J., 2008, Enhanced precision, accuracy, efficiency, and spatial resolution of U-Pb ages by laser ablation-multicollector-inductively coupled plasma-mass spectrometry: *Geochemistry Geophysics Geosystems*, Q03017, doi:10.1029/2007GC001805.
- Horstwood, M.S.A., Košler, J., Gehrels, G., et al., 2016, Community-derived standards for LA-ICP-MS U-(Th-) Pb geochronology – uncertainty propagation, age interpretation and data reporting: *Geostandards and Geoanalytical Research*, v. 40, p. 311–332, doi:10.1111/j.1751-908X.2016.00379.x.
- Jaffey, A.H., Flynn, K.F., Glendenin, L.E., Bentley, W.C., and Essling, A.M., 1971, Precision measurement of half-lives and specific activities of  $^{235}\text{U}$  and  $^{238}\text{U}$ : *Physical Review*, v. 4, p. 1889–1906.
- Kuiper, K.F., Deino, A., Hilgren, F.J., Krijgsman, W., Renne, P.R., and Wijbrans, J.R., 2008, Synchronizing rock clocks of Earth history: *Science*, v. 320, p. 500–504, doi:10.1126/science.1154339.
- Ludwig, K.R., 1998, On the treatment of concordant uranium-lead ages: *Geochimica et Cosmochimica Acta*, v. 62, p. 665–676, doi:10.1016/S0016-7037(98)00059-3.
- Ludwig, K.R., 2012, *Manual for Isoplot 3.75*: Berkeley, Berkeley Geochronology Center, Special Publication No. 5. Rev. January 30, 2012, 75 p.
- Matthews, W.A., and Guest, B., 2016, A practical approach for collecting large-n detrital zircon U-Pb data sets by quadrupole LA-ICP-MS: *Geostandards and Geoanalytical Research*, doi:10.1111/ggr.12146.
- Mattinson, J.M., 1987, Analysis of the relative decay constants of  $^{235}\text{U}$  and  $^{238}\text{U}$  by multi-step CA-TIMS measurements of closed-system natural zircon samples: *Chemical Geology*, v. 275, p. 186–198, doi:10.1016/j.chemgeo.2010.05.007.
- Otamendi, J.E., Ducea, M.N., Tibaldi, A.M., Bergantz, G.W., de la Rosa, J.D., and Vujovich, G.I., 2009, Generation of tonalitic and dioritic magmas by coupled partial melting of gabbroic and metasedimentary rocks within the deep crust of the Famatinian magmatic arc, Argentina: *Journal of Petrology*, v. 50, p. 841–873, doi:10.1093/petrology/egp022.
- Paces, J.B., and Miller, J.D., 1993, Precise U-Pb ages of Duluth Complex and related mafic intrusion, northeastern Minnesota: Geochronological insights to physical, petrogenetic, paleomagnetic, and tectonomagmatic processes associated with the 1.1 Ga Midcontinent Rift System: *Journal of Geophysical Research*, v. 98, p. 13997–14013, doi:10.1029/93JB01159.
- Paton, C., Woodhead, J.D., Hellstrom, J.C., Hergt, J.M., Greig, A., and Maas, R., 2010, Improved laser ablations U-Pb zircon geochronology through robust downhole fractionation

- correction: *Geochemistry Geophysics Geosystems*, v. 11, p. 1–36, doi:10.1029/2009GC002618.
- Petrus, J.A., and Kamber, B.S., 2012, VizualAge: A novel approach to laser ablation ICP-MS U-Pb geochronology data reduction: *Geostandards and Geoanalytical Research*, v. 36, p. 247–270, doi:10.1111/j.1751-908X.2012.00158.x.
- Saleeby, J.B., Sams, D.B., and Kistler, R.W., 1987, U/PB zircon, strontium, and oxygen isotopic and geochronological study of the southernmost Sierra Nevada Batholith, California: *Journal of Geophysical Research*, v. 92, p. 10443–10466, doi:10.1029/JB092iB10p10443.
- Stacey, J.S., and Kramers, J.D., 1975, Approximation of terrestrial lead isotope evolution by a two-stage model: *Earth and Planetary Science Letters*, v. 26, p. 207–221, doi:10.1016/0012-821X(75)90088-6.
- Wiedenbeck, M., Alle, P., Corfu, F., Griffin, W.L., Meier, M., Oberli, F., Von Quadt, A., Roddick, J.C., and Spiegel, W., 1995, Three natural zircon standards for U-Th-Pb, Lu-Hf, trace element and REE analysis: *Geostandards Newsletter*, v. 19, p. 1–23, doi:10.1111/j.1751-908X.1995.tb00147.x.
- Wiedenbeck, M., et al., 2004, Further characterization of the 91500 zircon crystal: *Geostandards and Geoanalytical Research*, v. 28, p. 9–39, doi:10.1111/j.1751-908X.2004.tb01041.x.