

Supplemental Material 1.

Procedures for preparing zircon solutions for CA-ID-TIMS-TEA

Zircon ages and geochemistry were determined using chemical abrasion-isotope dilution-thermal ionization mass spectrometry-trace element analysis (CA-ID-TIMS-TEA). Chemical abrasion methods were modified from Mattinson (2005). Individual annealed grains were removed from epoxy mounts and loaded into individual Savillex microcapsules with 100-125 μl of 29M HF and 25 μl of 30% HNO_3 . The microcapsules were then loaded into a Parr dissolution vessel and held at either 180 °C or 215 °C for 12-13 hours to leach out domains affected by Pb loss and melt or crystal inclusions. The resulting solutions were discarded, and each individual zircon crystal was repeatedly rinsed in its microcapsule with 29M HF, H_2O , 6N HCl and 30% HNO_3 before being spiked with EARTHTIME ^{235}U - ^{233}U - ^{205}Pb isotopic tracer (ET535; Condon et al. 2015). A final 75-100 μl of 29M HF were added to each microcapsule, which were re-loaded into the Parr dissolution vessel and held at 215°C for 48-60 hours for full dissolution. These solutions were dried down and dissolved in 6N HCl at 180°C for ~12 hours to convert the samples to a chloride form. The final solution was dried down once again and brought back up in 3N HCl for distillation of U, Pb, and trace elements by elution through AG-1 X8 200-400 mesh anion exchange resin using methods modified from Krogh (1973). Samples were loaded onto 50 μl anion exchange columns and rinsed dropwise to first elute trace elements. This solution was saved and dried down separately for trace element analysis (TEA) by solution ICPMS. The Pb and U were then eluted together into a separate Savillex beaker using 200 μl of 6N HCl and 250 μl of H_2O , respectively. Samples were dried down with a microdrop of 0.05M H_3PO_4 prior to analysis by TIMS.

Procedures for TIMS-TEA analyses and data reduction

The U-Pb aliquot from column chemistry was analyzed on the IsotopX Phoenix62 TIMS at Princeton University to obtain an age. Lead was run as a metal and measured by peak hopping on a Daly photomultiplier. Uranium was analyzed as UO_2 and measurements were made statically on a series of faraday cups.

Measured ratios were corrected assuming an $^{18}\text{O}/^{16}\text{O}$ of 0.00205 ± 0.00004 (2σ), corresponding to the modern atmospheric value (Nier 1950). Corrections for mass dependent fractionation were made using the known ratios of and $^{233}\text{U}/^{235}\text{U}$ in the ET535 spike and assuming a $^{238}\text{U}/^{235}\text{U}$ of 137.818 ± 0.045 (2σ), which represents the mean value of $^{238}\text{U}/^{235}\text{U}$ measured in natural zircon

(Hiess et al. 2012). Corrections for Pb fractionation occurred cycle-by-cycle, while the correction for U fractionation was done using the mean $^{233}\text{U}/^{235}\text{U}$ for the analysis. Repeat analyses of NBS981 and NBS982 Pb isotopic standards and the U500 uranium isotopic standard were run to monitor Pb and U deadtime on the Daly photomultiplier. Deadtime values changed after maintenance on the Princeton TIMS in 2016, and measurements were reduced with the appropriate Pb and U deadtimes for the period during which they were obtained. We followed the approach of Schoene et al. (2019) to correct for interferences by BaPO_4 , Tl, and unknown sources on Pb. A common Pb (Pb_c) correction was applied assuming all Pb_c was from laboratory contamination; we used the measured ^{204}Pb and a laboratory Pb_c isotopic composition, which we determined using procedural blanks. This value changed in Jan 2017 due to a change in our analytical procedure (implementation of side filament heating during sample warm up; see Schoene et al. 2019 for details).

For each analysis, we made a correction for initial secular disequilibrium in the ^{238}U - ^{206}Pb system due to the exclusion of Th during zircon crystallization (e.g., Schärer, 1984) using a zircon/melt partition coefficient of 0.29. This value was calculated using data from Claiborne et al. (2018) for a whole-rock composition comparable to the MSPT. Given that the MSPT is hypothesized to be a mixture of high-silica rhyolite and trachyte, we consider this to be a reasonable approximation of the parent melt. The effect of using this single value relative to other values (e.g., other single values based on other units or unit-specific values) is negligible, but a comparison can be seen in Supplementary File 2.

Data reduction was conducted using the Tripoli and ET_Redux software packages (Bowring et al. 2011), which uses data reduction and uncertainty propagations algorithms of McLean et al. (2011). We used U decay constants from Jaffey et al. (1971)

Zircon trace element compositions were determined using the TIMS-TEA method of Schoene et al. (2010), which capitalizes on the wash solutions from anion exchange column chemistry during U-Pb separations. Given that the process of column chemistry is intended to concentrate U and Pb, the wash solutions ultimately contain all other trace elements from the dissolved volume of zircon. If captured, these solutions (trace element aliquots) can be analyzed by solution inductively coupled plasma-mass spectrometry (ICP-MS).

Trace element aliquots were dried down and then re-dissolved in 3% HNO₃ + 0.2% HF + 1 ppb In. These solutions were analyzed on a Thermo-Fisher iCAP quadrupole ICP-MS using a Teledyne-Cetac ASX-100 autosampler at Princeton University. Uptake time was 20s and the line was washed with 3% HNO₃ + 0.2% HF for 60s between each analysis. A dilution series of a synthetic zircon solution was used to generate a concentration-intensity calibration curve over the range of concentrations observed in most zircon TIMS-TEA analyses. Reproducibility was assessed using a homogeneous solution of Plešovice zircon (Sláma et al. 2008) and a solution with a known Zr/Hf ratio of 50. Measurements of procedural blanks were monitored for laboratory trace element contamination. Sets of four unknowns were bracketed by individual measurements of the Zr/Hf and Plešovice standard, and a new calibration curve was made for every 20 unknowns. Solution measurements were converted to zircon concentrations by assuming that all of the measured trace elements substitute for Zr⁴⁺, such that $\Sigma = \text{Zr} + \text{Hf} + \text{Sc} + \text{Y} + \text{Nb} + \text{Ta} + \text{REE} = 497,646 \text{ ppm}$.

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