Supplementary Material*

For

Precambrian olistoliths masquerading as sills from Death Valley, California

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Methods: Isotopic age analysis

Apatite U-Pb at Trinity College Dublin: Samples KR1, SH3B

Samples KR1 and SH3B were run on the 2nd August 2016 and SHSA to SH5E on the 5th May 2017, at the Department of Geology, Trinity College Dublin. U-Pb apatite ages were determined using a Photon Machines Analyte Exite 193 nm ArF Excimer laser-ablation system with a Helex 2-volume ablation cell coupled to an Agilent 7900 ICPMS. The ICPMS was tuned using NIST 612 standard glass to yield Th/U ratios of unity and low oxide production rates (ThO+/Th⁺ typically <0.15%). 0.5 l/min He carrier gas was fed into the cell body and 0.2 l/min He was fed into the cup, and the aerosol was subsequently mixed with 0.56 l/min Ar make-up gas and a small volume of N₂ (ca. 6 ml/min) in a c. 1.5 cm³ mixing bulb to enhance signal sensitivity and reduce oxide formation. The following 8 isotopes were measured (with their respective dwell times in milliseconds listed in parentheses): ⁴³Ca (5), ²⁰²Hg (5), ²⁰⁴Pb (5), ²⁰⁶Pb (40), ²⁰⁷Pb (60), ²⁰⁸Pb (5), ²³²Th (5) and ²³⁸U (20). For all analyses the laser fluence was 3.5 J/cm², with a repetition rate of 10 Hz, a 60 μm spot size and analysis time of 30 s, followed by a 10 s background measurement.

The raw isotope data were reduced using the “VizualAge UcomPbine” data reduction scheme (DRS) of Chew et al. (2014), a modification of the U-Pb geochronology “VizualAge” DRS of Petrus & Kamber (2012) that can account for the presence of variable common Pb in the primary standard materials. The DRS runs within the freeware IOLITE package of Paton et al. (2011). In IOLITE, user-defined time intervals are established for the baseline correction procedure to calculate session-wide baseline-corrected values for each isotope. The time-resolved fractionation response of individual standard analyses is then characterised using a user-specified down-hole correction model (such as an exponential curve, a linear fit or a smoothed cubic spline). The VizualAge data reduction scheme then fits this appropriate session-wide “model” U-Th-Pb fractionation curve to the time-resolved standard data and the unknowns. Sample-standard bracketing is applied after the correction of down-hole fractionation to account for long-term drift in isotopic or elemental ratios by normalizing all ratios to those of the U-Pb reference standards. Common Pb in the apatite standards was corrected using the ²⁰⁷Pb-based correction method.

Blocks of six standards and two NIST612 standard glass analyses were followed by 20 unknown samples. For apatite analyses, the Madagascar natural mineral standard (Thomson et al. 2012; weighted average ID-TIMS concordia age of this crystal of 473.5 ± 0.7 Ma for an in-house aliquot) was used as the primary standard. McClure Mountain syenite apatite (weighted mean 207Pb/235U age of 523.51 ± 2.09 Ma, Schoene & Bowring 2006) and Durango apatite (31.44 ± 0.18 Ma, McDowell et al. 2005) were used as secondary standards. McClure Mountain apatite yielded a U-Pb Tera-Wasserburg concordia lower
intercept age of 517.8 ± 4.6 Ma (n = 27), anchored using a $^{207}\text{Pb}/^{206}\text{Pb}$ value of value of 0.88198 derived from an apatite ID-TIMS total U-Pb isochron (Schoene & Bowring 2006). The Durango apatite standard yielded a U-Pb Tera-Wasserburg concordia lower intercept age of 31.81 ± 0.33 Ma (n = 28), anchored using a $^{207}\text{Pb}/^{206}\text{Pb}$ value of value of 0.838 derived from the Stacey and Kramers (1975) terrestrial Pb evolution model for an age of 31.44 Ma. See Table S1 for apatite data, including standards, and Table S4 for a summary and lower intercept ages of standards.

Zircon U-Pb at Trinity College Dublin: Samples DZ-SH1, DZ-SH2, SH5A to SH5C

The following description is adapted from Chew et al. (2016). Samples were run during 2017 at the Department of Geology, Trinity College Dublin on the following dates: DZ-SH1 and DZ-SH2 - 25th April; SH5B and SH5C - 26th April; SH5A - 5th May. U–Pb data were acquired using a Photon Machines Analyte Exite 193 nm ArF Excimer laser-ablation system with a Helex 2-volume ablation cell coupled to an Agilent 7900 ICPMS at Trinity College Dublin. 0.65 l/min He carrier gas was split evenly between the large outer sample chamber and the small inner volume (the “cup”) where ablation occurs. A small volume of N₂ (ca. 6 ml/min) to enhance signal sensitivity and reduce oxide formation and 0.7 l/min Ar nebulizer gas was then introduced to the sample-gas mixture via an in-house smoothing device. $^{202}\text{Hg}$, $^{204,206,207,208}\text{Pb}$, $^{232}\text{Th}$, and $^{238}\text{U}$ were acquired for all U–Pb analyses. $^{91}\text{Zr}$ was employed as the internal standard for zircon. Analyses employed a 30 μm laser spot, a 4 Hz laser repetition rate, and a fluence of 3.9 J/cm². The analytical procedure utilized repeated blocks of four zircon standards followed by 20 unknown samples. Multiple spots per grain were analysed for each metabasite zircon. The primary standard was 91500 Zircon (published age 1065.4 ± 0.6, Wiedenbeck et al. 1995) and the secondary standards were WRS-1348 (published age 526.26 ± 0.70, Pointon et al. 2012), Plešovice (published age 337.13 ± 0.37, Sláma et al. 2008) and Temora (published age 416.8 ± 1.3, Black et al. 2003). See Tables S2 and S3, for zircon data from igneous and metasedimentary rocks respectively, and Table S4 for a summary of concordia ages of standards.

The raw isotope data was reduced using the “Vizual Age” data reduction scheme of Petrus & Kamber (2012) within the freeware IOLITE package of Paton et al. (2011). User-defined time intervals are established for the baseline correction procedure to calculate session-wide baseline-corrected values for each isotope. The time-resolved fractionation response of individual standard analyses is then characterized using a user-specified down-hole correction model (such as an exponential curve, a linear fit, or a smoothed cubic spline). The data reduction scheme then fits this appropriate session-wide “model” U–Th–Pb fractionation curve to the time-resolved standard data and the unknowns. Sample-standard bracketing is applied after the correction of down-hole fractionation to account for long-term drift in isotopic or elemental ratios by normalizing all ratios to those of the U–Pb reference standards.
Zircon U-Pb at Arizona Geochronology Center: Samples DZ-SH3, DZ-BM1, aliquots of SH5B and SH5C.

Zircons separated from sedimentary samples DZ-BM1 and DZ-SH3 were analyzed by Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICPMS) at the University of Arizona LaserChron facility on the 30th April 2014 and 28th August 2014 respectively. Methods and procedures followed those described by Gehrels and Pecha (2014). Analyses were conducted using a New Wave DUV193 Excimer laser (193 nm) with a spot diameter of 35 µm coupled to an Element II HR-ICPMS. The raw isotope data was reduced using the software package Isoplot (Ludwig 2003).

Zircons separated from metabasite samples SH5B and SH5C were analyzed by laser ablation - multi collector - inductively coupled plasma mass spectrometry (LA-ICPMS) at the University of Arizona LaserChron facility on the 12th May 2017.

U-Pb geochronology of zircons is conducted by laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) (Gehrels et al., 2006, 2008; Gehrels and Pecha, 2014). The analyses involve ablation of zircon with a Photon Machines Analyte G2 excimer laser equipped with HelEx ablation cell using a spot diameter of 20 microns. The ablated material is carried in helium into the plasma source of an Element2 HR ICPMS, which sequences rapidly through U, Th, and Pb isotopes. Signal intensities are measured with an SEM that operates in pulse counting mode for signals less than 50K cps, in both pulse-counting and analog mode for signals between 50K and 5M cps, and in analog mode above 5M cps. The calibration between pulse-counting and analog signals is determined line-by-line for signals between 50K and 5M cps, and is applied to >5M cps signals. Four intensities are determined and averaged for each isotope, with dwell times of 0.0052 sec for 202, 0.0075 sec for 204, 0.0202 sec for 206, 0.0284 sec for 207, 0.0026 sec for 208, 0.0026 sec for 232, and 0.0104 sec for 238.

With the laser set an energy density of ~5 J/cm2, a repetition rate of 8 hz, and an ablation time of 10 seconds, ablation pits are ~12 microns in depth. Sensitivity with these settings is approximately ~5,000 cps/ppm. Each analysis consists of 5 sec on peaks with the laser off (for backgrounds), 10 sec with the laser firing (for peak intensities), and a 20 second delay to purge the previous sample and save files.

The primary standard was Sri Lanka zircon, 563.5 ± 2.3 Ma (Gehrels et al. 2008). Two secondary zircon standards were also used: Duluth Gabbro (published age 1099 ± 2 Ma, Paces & Miller 1993) and Braintree diorite (published age 419.26 ± 0.39 Ma, Black et al. 2004). See Tables S2 and S3, for zircon data from igneous and metasedimentary rocks respectively, and Table S4 for a summary of concordia ages of standards. Of the unknown zircon grains four were analysed with two spots whilst the remaining eight were analysed with only one spot. All spots were located near the rim of the grains and cathodoluminescence revealed zonation in most zircons (Fig. S1).
Prior to analysis, grains are imaged to provide a guide for locating analysis pits in optimal locations, and to assist in interpreting results. Images are made with a Hitachi 3400N SEM and a Gatan CL2 detector system (www.geoarizonasem.org). In general, BSE images are made for detrital mounts and CL images are made for igneous mounts.

Following analysis, data reduction is performed with an in-house Python decoding routine and an Excel spreadsheet (E2agecalc)

Methods: Whole Rock compositional analysis

Major and Trace Elements by X-ray fluorescence

Major element data were obtained on fusion beads by X-ray fluorescence (XRF) analysis, using a PANalytical Axios Advanced XRF spectrometer at the University of Leicester, UK. This spectrometer runs a 4Kw Rhodium anode end window ceramic technology X-ray tube. Control and processing software used were the PANalytical SuperQ system with IQ+, WROXI and ProTrace extensions, using the default PANalytical SuperQ conditions. Total loss on ignition (LOI) was measured on pre-dried powders after ignition at 950°C in air for 1 hour. Instrumental conditions have been selected to avoid any significant line overlaps within the usual compositional range of most geological materials. Stability of the current generation X-Ray Spectrometry systems is such that measurements are no longer ratioed to a monitor sample to minimise instrumental drift effects. Instead, selected suitable drift monitoring samples are analysed at the commencement of each analytical run. Calibrations were set using international rock reference material (Table S5), under the same conditions, and regressing the measured count ratios against the recommended concentrations from Govindaraju (1994), Imai et al. (1995, 1996, 1999), Jochum et al. (2016) and values published on the GeoREM reference site, utilising a Fundamental parameters correction technique. The basis for the method outlined above is consistent with Tarney and Marsh (1991). Standard deviation uncertainties stated in Table 2 were determined from 19 different runs of an in-house basaltic sample, WS-1 (Table S6), chosen for its compositional similarity to the metabasites of this study.

Trace element data were obtained using 10g pressed powder pellets, with a polyvinylpyrrolidone methylcellulose binder (Watson 1996), using a PANalytical Axios Advanced XRF spectrometer at the Earth Sciences department, Royal Holloway University of London. Trace elements are calibrated using around 36 international silicate rock standards, with regression concentrations derived from compilations, published work on reference samples, and 6-10 isotope dilution analyses of Zr, Ba and REE, carried out at Royal Holloway. Typical calibration graphs can be viewed from the links tab at https://www.royalholloway.ac.uk/earthsciences/labs/xrf.aspx. The calibration graphs
illustrate maximum concentrations at which the calibrations are valid. Line overlaps are minimised by choice of instrument conditions, and corrected using low concentration samples, doped with a limited range of interfering elements. Comparison between XRF data and Isotope Dilution data (uncertainty ~±0.4% or better), for a suite of basaltic samples comparable to the metabasites of this study, shows agreement between XRF and ID data close to the XRF analytical error (Fig. S2). A monitor sample is used to check instrumental drift every fourth sample, which has been 0.6-1.6% from heavy to light elements over a three-year period. Typical reproducibility, based on 2σ of replicate basalt pellets, is given in Table S7, but varies proportional to concentration above ~ 100 ppm. These data are used to indicate estimated 2σ uncertainty in Table 2. Mass absorption corrections are calculated from major element concentrations normalised to 100 wt%, modified iteratively to include the minor contributions from potentially high-concentration trace elements such as Ni, Cr, S, Ba, Sr, Cl, F. Processing is performed in excel using software written by Thirlwall.

Major and trace elements by Inductively Coupled Atomic Emission Spectrometry and Inductively Coupled Mass Spectrometry

Major and trace (Ba, Sr, Y, Zr, Co, Cr, Cu, Ni, Sc, V, Zn) element data were obtained by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), using a Perkin Elmer Optima 3300RL. Further trace element data, including rare earth elements, were obtained by Inductively Coupled Plasma Mass Spectrometry (ICP-MS), using an Agilent 7500cx. Preparation and analyses were carried out at the Earth Sciences Dept., Royal Holloway University of London. The trace elements Ba, Sr, Y, Zr were analysed both by ICP-MS and ICP-AES. The results for these duplicated analyses, at the level of precision reported in Table 2, were identical between ICP-MS and AES and are reported in the AES data only. Powdered samples were fused with the help of Lithium Metaborate flux at 950°C for 25 minutes in graphite crucibles. Loss on ignition was not directly measured, but was instead obtained for aliquots of the same samples during preparation of fusions for XRF. The resulting glass pearls were diluted in 5% nitric acid to be analysed by ICP-AES, then the same solution was diluted 1:19 for ICP-MS. An external drift monitor with a known value for each element was analysed between every eight samples in order to correct them for instrumental drift. Four working standards (KC 10, KC 11, KC 12 and KC 14) plus a blank have been used to calibrate the ICP-AES for each element, and four International standards (NIM G, NIM N, GBW 07403, GBW 07405) plus a blank for the ICP-MS. To determine analytical precision, one sample of this study, CSD5, was run three times during ICP-AES (Table S8) and twice during ICP-MS (Table S9). Standard deviation uncertainties stated in Table 3 were determined from multiple runs of an in-house basaltic sample, KC-10 (Table S10, S11), chosen for its compositional similarity to the metabasites of this study. Three in-house standards were
prepared and run during ICP-AES and ICP-MS of this study (KC-10, KC-11, KC-12), along with the international Hawaiian basalt standard, BHVO 1 (Govindaraju 1994) (Tables S8, 9).

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Figure S1: Cathodoluminescent images of metabasite zircons analysed at the Arizona LaserChron facility. Circles indicate ablation spots. Names are of the format [Sample - Grain - Spot] as is used in Table S2. Ages stated are $^{206}\text{Pb}/^{238}\text{U}$ when $^{206}\text{Pb}/^{238}\text{U} < 1$ Ga, other ages are $^{207}\text{Pb}/^{206}\text{Pb}$. 